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JANUARY, 1951 - JUNE, 1959

AUTHOR

H. W. Crocker

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SUMMARY OF 234-5 DEVELOPMENT PROGRESS
JANUARY, 1951 - JUNE, 1959

By

H. W. Crocker

234-5 Development Operation
Research and Engineering Operation
Chemical Processing Department

July 29, 1959

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

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SUMMARY OF 234-5 DEVELOPMENT PROGRESS
JANUARY, 1951 - JUNE, 1959

INTRODUCTION

This summary of laboratory investigations has been assembled to provide a directory for source data of past laboratory activities. The dates listed in the outline are the operational periods covered by Separations Technology Section and Research and Engineering monthly activity reports. The monthly activity reports or specific program reports are to be consulted for valid detailed information on the indicated topics. A list of the program documents and activity reports is included. This document supersedes HW-49123 RD, "Summary Of Development Laboratory Progress, 1951 - 1954", by W. L. Lyon.

A. WET CHEMISTRY

1. Plutonium(III) Oxalate

1951

July	Study of process variables.
August	Study of process variables; coupling to Redox and Recuplex.

1957

March	Precipitation of continuous ion exchange product at 55 C. 1 M oxalic acid, filtrate 0.2 g/l Pu with solid formation.

2. Plutonium Peroxide

1951

August	Coupling studies.
September	Coupling studies - acid neutralization; plutonium peroxide from AT-solution - 0.5 M sulfate.

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October	Variations in bulk densities; purities from single peroxide strike on 3BP- for dry chemistry and reduction; effect of fluoride upon lanthanum separation.
November	Bulk density variations; disposal of methanol; Redox coupling.
December	Bulk density variations; reaction of supernatants with alcohols; lanthanum separation.
1952	
January	Neutralization of 3BP by various agents; precipitation from concentrated plutonium solutions.
February	Effect of total nitrate concentrations; temperature variations.
March	Purity data for peroxides.
April	Decontamination factors for peroxide precipitations.
September	Precipitation from cleanout solutions containing manganese ion.
1953	
February	Decontamination factors for peroxide precipitation. Work on high recycle.
March	High solubility in MRC's; effect of manganese ion.
1954	
February	Effect of fluoride and phosphate on formation of peroxy-complex.
August	Sulfate elimination: addition of phosphate, oxalate; temperature and acid effects.
1956	
March	Continuous precipitation from 1.3 M HNO_3, 0.05 M sulfate, 50 g/l Pu solution at 20 C feasible with 50 percent H_2O_2. Effects of sulfate concentration.

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3. AT-Solution Studies

1952	
June	Sulfate in AT still.
1953	
April	Plutonium sulfate in AT-solution (more work: brown vs. pink sulfates).
June	Gas evolution.
November	Gas evolution from AT solution.
December	Gas evolution from AT solution.
1954	
January	Plutonium(IV)-(VI) change in evaporation as a function of nitric acid concentration; sulfate studies - dissolution of red sulfate. Unreported - gas evolution studies.
1956	
May	Prevention of solid precipitation in solution shipment.

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4. Plutonium(IV) Oxalate

1951

September	Preliminary entries.
October	More preliminary work.
December	Preliminary work (supernatant destruction by evaporation).

1952

January	Precipitation from 3BP solution; also from concentrated plutonium solution.
February	Precipitation from synthetic 2BP; filter boat tests, cake-washing, filtration rates, etc.
April	Precipitation in 231 Bldg.; decontamination factors.
May	Coupling studies solubility vs acidity; plutonium-uranium separation; supernatant treatment - boiling, platinum, hydrogen peroxide.
June	Purity data, effect of filtration; UX1 separation; supernatant kill - platinum.
August	Filter tests; effect of impurities on waste losses.
September	Effect of oxalic acid concentration.
October	Rate of oxalic acid addition; separation from lanthanum, iron, nickel.
November	Orange oxalate(VI?).
December	Precipitation from concentrated solutions, greater than 100 grams per liter.

1953

January February	Mixed reagent addition.
March	Decontamination factors.
April	Valence adjustment.
June	Task I prototype studies.
August	Temperature effects; aluminum addition; 0.06 M oxalate prior to pre-reduction; (pre-reduction-precipitation

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effects, explanation?); Task I prototype, supernatant destruction - oxalate kill by permanganate as function as temperature, 15 to 65 C; use of ozone.

October	Effect of anions; effects of stirrer speed; precipitation of solution after partial neutralization; filter cloth tests.

1954

February	Glycol in the oxalate precipitation; dissolution of plutonium oxalate in aluminum and iron systems
June	Agitation studies including use of air lift; interference by manganese ion; filter cloth evaluation; and mercury separation (factors for dry chemistry, reduction).
November	Continuous filter - Task I; gas evolution from supernatants; supernatant treatment - kills with permanganate and dichromate.
December	Supernatant treatment: valence adjustment after kill. Unreported effect of ANN on plutonium oxalate solubility; mercury separation factors.

1955

February	Batch precipitation of concentrated Redox and AT solutions in Task I prototype.
March	Hg limits on Task I nitrate feed of 1,000 ppm, corrosion effect of Hg on gold components of filter boat.
May	DF's of 6.6 and 10 for Zr-Nb and Ru-Rh from Redox PR solution in oxalate precipitation. Stability of Dynel SD-9 cloth to oxalate precipitation chemicals.
June	Batch precipitation studies. Continuous filter and calciner fabrication.
August	Initial continuous oxalate precipitation studies.
September	No effect on batch precipitation characteristics by increasing Al to 100,000 ppm and Fe to 70,000 ppm in feed.
November	Continuous oxalate precipitation studies at ambient temperature process operation.
December	Continuous precipitation, filtration, calcination studies at 0.5 kg/hr rate, 4 percent loss to filtrate.

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1956

January	Continuous Task I runs, effect of process rates, slurry holdup on waste losses.
February	Continuous precipitation studies, separation factors equivalent to batch process, effects of washing on filter cake quality. Life test (500 hours) for Dynel SD-9 filter cloth in continuous oxalate processes. Dissolution of Pu(IV) oxalate by aqua regia (without metallic contamination) for preparation of spectrographic analysis samples.
March	Effect of Zr, Versene, fluoride on Zr-NB DF's across batch oxalate precipitation. Continuous Task I studies, 1 kg/hr rate with 3 percent filtrate loss. Effects of agitation speed, slurry residence, filter cloth deterioration. Capacity of 3" diameter by 36" long calciner. Continuous prereduction of plutonium nitrate feed. Continuous precipitation from dilute (10 g/l) solutions with low filtrate loss.
April	Calciner operating temperature limits. Effect of temperature on calciner capacity.
May	Degradation of Dynel SD-9 filter cloth in Task I process solutions.
November	Continuous Task I operation with 120 g/l Pu in 5 M HNO_3 containing 0.65 M $(NH_4)_2SO_4$ (negligible effect).
December	Effect of 0.65 M $(NH_4)_2SO_4$ on sulfate precipitation in Task I feed solutions on standing at room temperature.

1957

January	Purex product 146 g/l Pu 6 M HNO_3, 0.8 M $SO_4^=$ did not show precipitation after one-week storage. First cold runs in new continuous Task I unit used cerous nitrate (90 g/l Ce) feed, 1 M oxalic acid strike solution. Calcined at 300 C, with 15 minutes' residence. Throughput 250 g/hr.
February	Continuous Task I cold run was made under identical conditions of the first run. Rocker arm agitator modifications.
May	Operability and capacity of continuous Task I unit demonstrated in hot runs. Purex 2BP feed 75 - 100 g/l Pu in 4 M HNO_3, 500 g/hr, 1 M oxalic acid, loss to filtrate 1 - 3 percent. Precipitation directly in filter pan.

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June	Continuous Task I runs using Redox 3BP as feed 95 g/l Pu in 4.5 M HNO_3, 500 g/hr, 1 M oxalic acid, loss to filtrate $\overline{5}$ percent. Calciner residence time studies at 350 C. Fe impurities 300 ppm.
July	Continuous Task I runs. Continuous oxalate chemical kill of filtrates.
August	Operation of continuous Task I on extended period to supplement Building production. 25 kg Pu, 10 percent waste loss, Dynel SD-9 cloth superior to 904 in runs. Material acceptable for fluorination and reduction.
September	Spectrographic results of the oxide produced in the sustained runs of August demonstrate the higher quality of oxide produced in continuous processing equipment. Runs demonstrate use of continuous Task I precipitation reactor with overflow of slurry into filter pan. Normal flow sheet. Effects of flow control on cake quality.
October	Continuous Task I runs demonstrate suitability of simultaneous reduction-precipitation process. Calciner surge capacity tests.
November	Calciner surge tests were continued. Surge capacity 70 percent above normal steady state operating capacity. Test of cyclone separator and filter on calciner off-gas system.
1959	
March	Effect of titanium and vanadium on Z Plant processing. Aluminum oxide filter media tested with plutonium(IV) oxalate slurry. Study of plutonium oxalate and filtrate densities.
April	Particle size distribution in plutonium(IV) oxalate slurry. 10 - 100 micron size range. Water content of filtered plutonium oxalate cake.
May	Purification study of the continuous oxalate process flow sheet using 200 g/l Pu in feed.

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5. F-10-P And 3BP Studies

1952

April	Red precipitate in P-1 solution - analyses of solution and of solid; abnormal beta, gamma activities in sample cans and 3BP: solids in 3BP.
May	Solids in 3BP and P-1.

1953

March	Solids and Redox PR solution.
October	Sludge in Redox PR can - high radiation.

1954

February	Plutonium phosphate in F-10-P.
December	F-10-P filtration and stabilization.
Unreported	ANN in 3BP - concentration effects.

1955

May	Filtration studies on Redox PR solution - no decontamination from FP's across solution filter.

1956

February	Effects of filtration and precipitation on removal of Zr-Nb from Purex PR solution. DF of 1.2 obtained.
March	Studies on use of silica gel or Dowex-1 column for Zr-Nb removal from Purex PR solution (DF's of 3). Effect of versenes and fluoride on Zr-Nb DF across across oxalate precipitation.
May	Solids in concentrated Purex PR solution - Pu polymer, silica, iron, phosphorus. Dissolution procedure. Concentration of Purex 2BP.
July	Solids in Redox L-3 to L-4 valve. Ru, Zr, Nb, Al, Cr, Cu, Fe, Ni identified.
August	Study on solids in Purex tube bundle - primarily silica. Study on precipitation in Redox E-1, E-3 vessels.

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	Studies on solids in Redox L-3 to L-4 valve.
October	Study of solids found in Purex concentrator. Contained high silica and iron. Si/Pu ratio of 1/5 in solids.

1957

March	Purex concentrator solids. Black flint-like material from Purex product concentrator contained mostly Si and Fe. Cr. Ni also detected, Pu was 1%.

1958

April	Solids in Purex ion exchange concentrator. Pu(IV) polymer and resin fines identified.

6. 224 Building Studies

1951

September	Barium sulfate scavenging.
October	Scavenging of barium.

1953

May	Calcium separation; tolerance to aluminum.
July	Tolerance to aluminum.

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1954	
April	Decontamination factors for various steps - omission of lanthanum fluoride by-product.

7. Double Salt Studies

1953	
November	Precipitation studies; large-scale strike.
December	Precipitation studies.
1954	
January	Double salt precipitation; 300 gram reduction.
February	Calcium-thorium fluoride precipitation.
March	Separation factors for the calcium double salt.
April May	Large-scale operations.
June	Plant scale operations.
Unreported	Decontamination factors for zirconium, niobium, and separation from sulfate.

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8. Miscellaneous

1951	
October	Disposal of methanol.
1952	
August	Evaluation of filter aid for N-1 filter.
1953	
August	Stability of hydrogen peroxide; spectrophotometric determination of plutonium valence.
October	Plutonium(III) oxalate precipitation - hydrogen peroxide, sulfamic acid reduction of Redox product.
1954	
February	Plutonium fluoro oxalate.
October	Specific gravity measurements for plutonium nitrate - nitric acid solutions.
Unreported	Specific gravities for plutonium polymer. Effect of fluoride on plutonium for light absorption.
1956	
September	Titanium corrosion in 8 M HNO_3, 130 g/l Pu(IV), at B.P. 0.0003 ipm.
October	Corrosion of tantalum, titanium, 304L stainless steel in HNO_3-Pu solutions. Effect of sulfate ion. Ta and Ti superior.
December	Recovery of Pu from sandpaper and sweeping by ashing at low temperature followed by 6 M HNO_3 - 0.25 M HF leaching at B.P. Pu recovery of 90%. 304-L corrosion rate in boiling solution of 0.003 M oxalic acid - 5 M HNO_3 containing 1.5 g/l Cr^{+3}, 0.00018 ipm.

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1958

January	Spectrophotometric method for nitrate to determine small amounts of nitric acid in presence of acids or salts yielding hydrogen ions.
August	Neptunium oxide (< 200 ppm metallic impurities) produced by oxalate precipitation and calcination. Bulk density was 2.7 g/cc. Effect of radiation buildup noted.

1959

June	Initial studies of direct calcination of plutonium nitrate (275 g/l) to plutonium oxide and subsequent chlorination to $PuCl_3$.

9. Plutonium Trifluoride Precipitation

1956

April	Continuous precipitation and filtration of PuF_3 from HNO_3 solution using HF as precipitant. Effects of alcohol wash and filtrate loss. Sulfamic reductant.
	Attempted precipitation of PuF_3 from HNO_3 solution using fluosilicic acid. No precipitation noted. Effect of complexing strengths of Pu(III) and Pu(IV) on fluoride.
June	Studies on PuF_3 precipitation from high purity plutonium nitrate using HF precipitant.

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September	Precipitation of PuF_3 by HF from an HNO_3 solution. Effects of reducing agents, and precipitation data included.
October	Continuous precipitation from nitrate solution using 3 M HF. Feed reduced with SO_2 + ascorbic acid. Filtrate losses 8 - 17%. Separation factors 10, 20, 10, 4 for Fe, Ni, Cr, Mg.
December	PuF_3 dried at 600 C showed no weight change on standing 17 days.

1957

March	Continuous ion exchange product subjected to PuF_3 precipitation using 1 M excess fluoride. First strike slow filtering, 0.2 g/l Pu in filtrate. Second strike, spiked 1000 ppm Dow Separan 2610, fast filtration, 0.05 g/l Pu in filtrate.
April	Small-scale continuous precipitation runs, feed 50 g/l Pu, 5 M HNO_3, precipitant 3 M HF. Valence adjustment with semi-carbozide. Separation factors 125, 147 for Al and Fe. Slurry degrades upon standing overnight.

10. Plutonium Tetrafluoride Precipitation

1955

October	Aqueous precipitation of PuF_4 from Pu(IV) nitrate solution using HF and ammonium fluosilicate.
November	Precipitaion of PuF_4 from Pu(IV) nitrate solution using hydrofluosilic acid and HF.

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1956

January	Precipitation of PuF_4 from 40 g/l Pu and 5 M HNO_3 with 4 M HF. Fe and Al separation factors of 10 - 20 at initial impurities of 10,000 and 100,000 ppm respectively. Effects of washing on cake quality.
February	Studies on drying of the PuF_4 precipitate.

11. Oxalate Kill

1955

September	Chemical kill of plutonium oxalate demonstrated with $KMnO_4$ and H_2O_2.

1956

May	Investigation of conditions for batch and continuous killing of oxalate filtrate. Studies on stability of Dynel SD-9 filter cloth in oxalate precipitation chemicals.
June	Studies on continuous evaporation of killed oxalate filtrates. Studies on evaporative heat kill of oxalate filtrates.
July	Prototype continuous oxalate filtrate kill equipment installed. Studies on continuous oxalate kill. Studies on evaporative heat kill of oxalate filtrate.

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November — Adaptability of Recuplex dissolvers for evaporative oxalate kill process.

1957

January — Pu nitrate solutions containing sulfamic acid or semi-carbazide, reoxidized and concentrated by a factor of five in a continuous kill. Feed solution was 6 M HNO_3, 0.05 M semi-carbazide, 11-50 g/l Pu. Feed solution slowly added to boiling solution to safely accomplish the kill.

July — Filtrate from continuous oxalate precipitations treated by continuous chemical kill using $KMnO_4$ at 45 C. Permanganate leg of the "U" column operated satisfactory. Peroxide leg (1" diameter) reaction too vigorous at 10% or 2-1/2% peroxide concentration. Increased diameter of peroxide leg is required.

October — Evaporative kill experiments indicate manganous ion addition may be needed as catalyst, due to low amount of iron in filtrate.

November — Evaporative kill studies indicate a 21-minute decomposition half-time. Small amount of solids can be tolerated in the evaporative kill process.

12. Plutonium Polymer

1956

August — Limiting conditions for formation of plutonium polymer in nitric acid solution.

November — Formation of plutonium polymer in nitric acid solutions. Absorption peaks of polymeric solutions. Effect of nitric acid concentration and HNO_3/Pu ratio on polymer formation. Temperature effects.

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December	Study of polymer formation in Pu(IV) nitrate solutions of low acidity. Effects of nitric acid concentration, and temperature. Absorption spectra of Pu(IV) from 0.02 M HNO_3 to 14 M HNO_3.
1957	
February	An E-7 sample from Redox studied spectrophotometrically, found to have a spectrum characteristic of polymeric plutonium. Depolymerization in 4 M HNO_3 produced plutonium(IV) spectrum.
1958	
February	Study of polymer formation as a purification process. Separation factors of 3300, 40, 1000, 5, 100, 100 for Al, Cr, Cu, Fe, Mg, Ni based on initial impurity concentrations of 10,000 ppm. Effect of precipitation on fine particle formation.

13. Ion Exchange

1956	
November	Initial testing of semi-continuous Higgins-type columns.
December	Equipment testing results. Flow control problems on continuous equipment.
1957	
January	Mechanical testing of hydraulic ram, conductivity probe, and pumps.
February	First cation hot run completed. Steady state not achieved because of excessive gassing. Run conditions:

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XAF	35 ml/min	Dowex 50W-X8 resin
XCS	2.1	
XAS	2.0	10-minute cycle
XCIS	5.0	Feed concentration 8.3 g/l Pu
XAX	2.0	Product concentration 25 - 70 g/l Pu
		Waste losses ~1%

Effects of feed reductants discussed.
Elutant was 6.4 M HNO_3, 0.3 M sulfamic or semicarbazide.

March

Workable flow sheet was demonstrated in a two-week-long "hot" run of cation columns. Run conditions:

XAF	39.5 ml/min	10-minute cycle
XCIS	5.0	Dowex 50W-X8 resin
XAS	2.0	Feed 2.5 - 7 g/l Pu
XCS	2.1	Product 35 - 75 g/l Pu
XAX	2.0	Waste loss 0.1%

Most promising feed reductant was 0.1 M NH_4HSO_3.

Effects of plutonium polymer formation during feed preparation.

Most promising elutant 5 M HNO_3-0.33 M sulfamic acid.

Higher product concentration may require cooling of XC column.

Stability of cation exchange product: oxidation of XCP dependent on solution acidity. Oxidation rate 0.2 - 5. %/day. Effects of other reagents discussed.

Adjustment of Purex 2BP to 7 M HNO_3 resulted in oxidation to Pu(IV) within two minutes, for anion exchange feed.

April

Tentative Purex flow sheet (HW-49524) was demonstrated in a hot anion exchange run.

Run conditions:

Resin Dowex 1-X4 (nitrate form) 5-minute cycle
Feed concentration 6 g/l
Product concentration 45 g/l Pu
Column temperature 50 C

Effect of column temperature, feed flow rates, etc., discussed.

May

Continuous runs with Purex 2BP as feed solution showed anion exchange to be superior to cation process for

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purification and decontamination.

1. Anion Zr-Nb DF of 28, Ru-Rh DF of 2.0
   Cation Zr-Nb DF of 3, Ru-Rh DF of 3.0
2. Total gamma DF of 3 for both anion and cation.
3. Anion reduces silicon 10 - 20 fold, silicon not removed in cation process.
4. Anion: separation factors for (Cr, Cu, Fe, Mn, Ni, Pb) of 5 - 20 and (Al, Ca, K, Mg, Na) of 100.

Cation removed only slight amounts of light metals, none of the heavy metals.

June	Changes made in flow, and temperature control to permit sustained anion exchange runs at high throughput with Dowex 1-X4 anion resin.
July	Operation of the anion exchange system on a Purex flow sheet in the temperature range of 50 - 70 C and feed acidity range of 5 - 7.2 $\underline{M}$ HNO_3 was satisfactory. Flow rates up to 30 percent greater than the 54 mg Pu/min/cm^2 required by flow sheet were used. Product concentration was near the 58 g/l equilibrium value. The ratio of XSW to XAX appears to depend on the volume of resin pushed. Satisfactory resin push was attained with 10 psi pressure. Zr-Nb DF = 250, Ru-Rh DF = 30 when adequate scrub flows are used. Appreciable FP's accumulate on the resin and are difficult to remove. Metallic separation factors varied from 5 - 100. Effects of temperature control, acidity, and column dimensions are recorded.

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14. Metal Dissolution

1957	
September	Plutonium dissolved in 15 weight percent sulfamic acid solution. Dissolution not as rapid as HNO_3-HF, no residue left as in HNO_3 - sulfamic mixtures. Concentrations 100 g/l Pu, in 0.3 - 1 M acidity, stable for three weeks.
1958 March	Initial tests of continuous dissolution of plutonium metal (10 g scale) in HNO_3. Effect of PuO_2 formation.
1959	
April	Study of massive plutonium metal dissolution using sulfamic acid. Effects of final oxidation adjustment, plutonium sulfate and plutonium polymer formation.
	Study of reactive alpha Pu skull passivation by caustic solution.
May	Study of reactivity of Pu-Al skulls during caustic passivation.
June	Problem of unusual reactivity of temperature cycled alpha Pu skulls.

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15. Solution Concentration

1955

September	Concentration limits of oxalate filtrates containing 200,000 ppm Fe, 10,000 each Al, Cr, and Ni was a factor of three. Oxalate destroyed by concentration.
October	Concentration studies for Redox 3BP containing 100,000 ppm Al. ANN precipitation noted at 130 g/l Pu concentration.

1957

March	Concentration of cation exchange product, XCP (0.33 M sulfamic) forms sulfate precipitation after concentration by a factor of two.
April	Cation exchange product: Solution of 65 - 80 g/l Pu, 5.4 - 6.8 M HNO_3, 0.5 M $SO_4^=$ stable at room temperature.

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Solutions up to 150 g/l Pu, 7 M HNO_3, 0.8 M $SO_4^=$ stable at boiling temperature.

16. Valence Adjustment

1956

April	Pu(IV) to Pu(III) reduction studies using H_2O_2, hydroxylamine hydrochloride, with sulfamic acid as holding reductant. Solutions were 17 - 70 g/l Pu and 0.8 - 2.5 M HNO_3.
June	Studies of Pu(IV) to Pu(III) reduction in HNO_3 solution using sulfamic acid and ascorbic acid. Effects of solution concentration.
September	Ascorbic acid stability tests. Effect of sulfamic acid, nitric acid, and plutonium concentration.
October	Ascorbic acid stability Use of ascorbic acid as Pu(IV) to Pu(III) reductant for HNO_3 solution feed, ion exchange process. Effect of ascorbic and sulfamic acid. Adaptability of ascorbic-sulfamic bearing filtrate solutions to batch and continuous kills temperature effects.
November	Effect of semi-carbazide and aminoguanidine as reducing agents for ion exchange process. Effects of sulfamic acid on reduction stability.

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Preliminary study on reduction of Purex 2B stream with sulfurous acid, ascorbic acid, and hydroxylamine sulfate. Initial studies with sulfurous acid fail to reduce the Pu in the 2B stream.

December Effectiveness of hydroxylamine, ascorbic acid, aminoguanidine, and semi-carbazide as reductants in high acid solutions. Effects of time and oxidation. Semicarbazide is slower, but more stable than others.

TBP strip experiments on Purex 2BP show ascorbic acid and hydroxylamine as effective reductants. Sulfurous acid ineffective.

1957

March Study of reduction of cation exchange feed to Pu(III). 0.05 M hydroxylamine sulfate, 0.05 M sodium bisulfite, and 0.1 M semicarbazide required 24 hours, 2.5 minutes and 0.5 minute,respectively, for reduction. Hydroxylamine sulfate reduced solution stable for two weeks, others for four days.

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17. Plutonium Trichloride

1958

March	Synthetic ("cold") chloride slag and crucible recovery studies. Effect of chloride boil-off with nitric acid as part of the dissolution process.
April	Dissolution, I_2 removal and chloride removal, were effected by 13 $\underline{M}$ HNO_3 at B.P. on synthetic slag and crucible. High degree of chloride removal accomplished. Degree of silica coagulation not evaluated.
August	Flow sheet for chloride removal and slag and crucible recovery determined from "hot" runs. Pu recovery of 89 percent. Elimination of separate iodine removal period. Precipitation and redissolution of plutonium polymer from slag and crucible solution to perform purification and volume reduction.
September	Little effect noticed on aluminum slag and crucible cans due to long-term storage of chloride reduction slag. Description of an alternate recovery process involving hydrolysis and precipitation of plutonium. Low acidity for precipitation is obtained by distillation of acid and water until a molten salt residue remains. Material then diluted, filtered, cake dissolved for processing, and filtrate discarded. Volume reduction advantages.
October	Recovery of chloride slag and crucible was tested on the hydrolysis-precipitation flow sheet. Plutonium recovery of 99 percent. Document HW-58021, "Precipitation Method For The Recovery Of Plutonium From Chloride Slag And Crucible", issued.
December	Nickel-coated crucible dissolutions were made with the standard flow sheet, chloride boiloff, followed by HNO_3-HF dissolution of residue, without difficulty. Test of hydrolysis-precipitation flow sheet revealed difficulties caused by nickel in slag and crucible. Coprecipitation of Mg, Ca, and Ni, along with the Pu, and high waste losses to the filtrate occurred.

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1959

January	Test of reducing agents for Pu reduction to (IV) valence in hydrolysis-precipitation recovery method for nickel-coated MgO crucibles. Large-scale (one crucible and slag) dissolver has been installed for chloride slag and crucible recovery development.
February	Study of valence adjustment and precipitation parameters for hydrolysis-precipitation method of reprocessing nickel-coated MgO crucibles.

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B. DRY CHEMISTRY

1. Processing Of Metal Turnings

1951

August September	Hydrofluorination.

2. Hydrogen Fluoride Absorption

1951

September October	Miscellaneous.

1952

February	Dorex cannister capacity.
June July	Caustic scrubber for HF absorption.
September	Calcium carbonate.

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3. Hydrofluorination Of Plutonium Peroxide

1951

October	Effect of air leakage.

4. Fluoride Analysis

1952

January February	Analysis by conversion to oxide.
November	Analysis by conversion to oxide.

5. Filter Boats

1952

May	Losses in drying - filter boat.
June	Filter boat leak tests.
July	Filter paper liners.
October	Filter boat cleanout - use of oxalic acid or hydrogen peroxide.

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6. Freonation

1952

July	First work.
December	Additional results.

1953

February	Two deep cake runs.
April	Work abandoned: corrosion; polymerization.

7. Calcination

1952

September	Drying of plutonium(IV) oxalate: 150 C, lumps; 300 C for calcination; thermal decomposition of plutonium(III) and (IV) oxalate.
October	Plutonium(IV) oxalate drying procedure - glowing boat; temperature measurements.
December	Drying of plutonium(IV) oxalate (130 C - hexahydrate).

1953

February	Plutonium(IV) oxalate decomposition - exothermic reaction.
May June	Calcination of plutonium(IV) oxalate; reactivities of oxides (HCl-KI).
July	PuO_2 reactivities (wet).
September	Calcination of plutonium oxalate.
October	Thermal decomposition of plutonium oxalate.

1954

February	Infra-red drying and calcining of oxalate.

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March	Decomposition of plutonium(IV) oxalate; reactivity of PuO_2.

1956

April	Operating temperature range of 300 - 400 C for continuous Task I calciner determined from laboratory tests.

8. Hydrofluorination

1952

July	Summary of dry chemistry and reduction data.
September	Cake depth vs. hydrofluorination rates.
October	Tolerance to laboratory air - 37 percent.
November	Hydrofluorination of erupted boat load; tolerance to laboratory air.

1953

January February	Preparation of reduction of low temperature fluorides.
September	Direct hydrofluorination.
October	Direct hydrofluorination of oxalate - PuF_3 production.

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1954

November	CeF_3 production; conversion of blue powder to PuF_4.
December	Conversion of blue powder to PuF_4.

1955

February	Continuous PuO_2 hydrofluorination studies with 97 percent conversion to fluoride; metal reduction yields were 98 percent.
May	Continuous PuO_2 hydrofluorination studies.
June	Continuous PuO_2 hydrofluorination studies.
August	Study on SiC filters as replacement of platinum frits for Task II.
December	Continuous PuO_2 hydrofluorination studies, processing rate 0.4 kg/hr, $PuF_{3.72}$, reduction yield of 98.6 percent on PuF_4 product.

1956

March	Continuous PuO_2 hydrofluorination studies, 100 percent conversion to PuF_4.
May	Operating temperature range for continuous hydrofluorinator determined to be 450 - 550 C.
September	Simulated operation of plant reactor tested by conveying powder in a vibrating oval tray.

1957

January	A plastic (Teflon and fluorothene) rotary feed valve replaced inconel feed screw to eliminate corrosion in continuous hydrofluorinator.

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9. Chlorination

1958

February	Initial $PuCl_3$ preparation by CCl_4 (in argon carrier) at 450 C in Pyrex.
	$PuCl_3$ hydration path and rate study. Effect of closed container storage on hydration.
March	Plutonium trichloride hydration study resumé. Effects of atmosphere and powder storage.
	$PuCl_3$ preparation (600 g) by CCl_4 treatment at 450 C resulted in Cl/Pu molar ratio of 2.94.
	Study on drying of $PuCl_3 \cdot 6H_2O$ by heating.
	$PuCl_3$ process off-gas line plugging problems.
	Neutron count study of $PuCl_3$ shows greater reduction in neutrons from $PuCl_3$ than PuF_4.
April	Chlorination rate study for conversion of PuO_2 to $PuCl_3$ with CCl_4. Effect of temperature on rate and powder density.
May	$PuCl_3$ prepared in large-scale batches by CCl_4 in argon carrier at 500 C.
	Batch tests of PuO_2 chlorination by $COCl_2$ gave 97 percent conversion, decreased reagent consumption, more desirable off-gas by-products.
	Small Pyrex vibratory tube chlorinator assembled to permit continuous process study.
June	Chlorination rate study for conversion of PuO_2 to $PuCl_3$ with CCl_4. Reaction products identified as C_6Cl_6, C_2Cl_4, C_2Cl_6, CO_2, CO, Cl_2, $COCl_2$.

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	Unsuccessful attempts to produce PuOCl at 225 - 400 C. By-products from the reaction are Cl_2, HCl, CO_2.
	Chlorination of PuO_2 by $COCl_2$ at 350 - 550 C; Cl_2, HCl, CO_2 off-gas products.
	Direct chlorination of plutonium(IV) oxalate by $COCl_2$ studied; 175 - 450 C on 100 g scale. Reaction rate and powder density comparison.
	Preliminary testing of continuous 1" diameter chlorination reaction used cerium oxide.
	Corrosion tests by D&CCO show Hastelloy B and C to be suitable for $COCl_2$ system at 400 - 600 C. Effect on other materials recorded.
July	Large-scale batch chlorinations of PuO_2 by $COCl_2$ continued. Effect of calcination temperature of PuO_2 on chlorination rate. High temperature chlorination (600 C) introduced unknown residues in off-gas.
	Plastic materials' resistance tested in phosgene atmosphere.
August	$PuCl_3$ production by $COCl_2$ chlorinating agent.
	Conversion of PuO_2 to $PuCl_3$ by H_2 and HCl. Effect of temperature on chlorination rate. Formation of $PuOCl_2$.
September	PuO_2, previously heated to 900 C, was successfully chlorinated with $COCl_2$ at 500 C.
	Report, HW-57498, was issued to cover the status of the $PuCl_3$ program. Flow sheets for preparation of the compound, reduction to metal, and recovery schemes are included.
October	$PuCl_3$, prepared at 600 C, was a dark-green crystalline material in contrast to the blue-green powder produced at 500 C.
	Continuous 1" diameter chlorinator operated with PuO_2 and $COCl_2$ for $PuCl_3$ conversion at 500 C.
November	PuO_2, previously heated to 1800 C, was chlorinated to $PuCl_3$ in five hours using $COCl_2$ at 450 C.
	Study of hydration rates of $PuCl_3$ completed over range 2 - 75 percent relative humidity. A discussion and graph are included.
December	Description of 1" diameter continuous chlorinator system.
	Continuous 1" diameter chlorinator operated at 34 g/hr on extended run conditions. Chlorination of PuO_2 by $COCl_2$.

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was complete.

Samples of $PuCl_3$ from batch and continuous processing were found to have four and six micron particle size respectively. PuO_2 powders for the conversion were 0.2 micron size.

Large-scale batch conversions of PuO_2 to $PuCl_3$ by $COCl_2$ were made for reduction studies.

Off-gas studies for $COCl_2$ chlorination completed over 275 - 600 C range. Products are $COCl_2$, CO, HCl, and Cl_2.

1959

January

Continuous chlorination of PuO_2 by $COCl_2$ in the 1" diameter tube operated at 70 g/hr on an extended run.

Run conditions:

Powder bed depth: 3/8"
Residence time in furnace: 45 minutes
$COCl_2$ flow: 200 percent excess
Temperature: 500 C

Effects of temperature and flow rates on the conversion are included.

Pu/Cl ratio = 2.7
$COCl_2$ retention in cake: $0.02 \frac{\text{mols } COCl_2}{\text{mol } PuCl_3}$

Corrosion studies in $COCl_2$ system over 100 - 500 C range. Hastelloy B and C most promising materials.

Off-gas analysis study indicates gas composition to be dependent on temperature, phosgene flow, and powder bed composition. Elimination of off-gas solid deposits of metal chlorides discussed.

February

Lumping problems of $PuCl_3$ in the 1" diameter continuous chlorinator.

Study of brown solid deposits on chlorination tube.

March

Initial runs in the large Hastelloy continuous chlorinator at 90 g/hr Pu rate. Mechanical difficulties encountered.

Off-gas filter problems.

Document HW-59759, "The Analysis Of Exhaust Products In

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The Chlorination Of Plutonium Oxide With Phosgene", was issued to summarize off-gas studies.

April	Large continuous chlorinator operated at 295 g/hr Pu rate. Phosgene flow, cake lumping problems.
May	Off-gas filtration development.
June	Chlorination of PuO_2 from direct calcination of plutonium nitrate solution.
	Elimination of lump formation in the large continuous chlorinator.
	Chlorination of PuO_2 by CO plus Cl_2.
	Test of Davis phosgene detector.

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C. METAL REDUCTION

1. Reduction Of Off-Standard Fluorides (Blue And Low Temperature)

1951

July	Reduction of PuF_3.

1952

July	Sintered PuF_4 reduction.
October	Reduction of PuF_3 from freon halogenation.

1953

April	Reduction of low temperature fluorides.
May	Blue fluoride reductions.
July August	Blue fluoride reductions - plant scale.

1957

July	Nine hundred grams of "blue fluoride" was received from the processing operation. The material was divided into two batches and reduced. I_2/Pu was 0.8, excess Ca was 0.6. Yields were 98 percent, metallic impurities < 550 ppm. This indicates poor reduction from wet precipitated fluorides are probably due to oxide formation on the powder surface.

2. Sulfur Booster

1952

April	Melting points of slags; addition of calcium iodide; uranium reductions; use of mixed magnesium and calcium; addition of I_2.

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May	Reduction of PuF_4; addition of sodium fluoride; calcium-magnesium, calcium-stronium alloys.

3. Button Reactivities

1954

August September November	Miscellaneous entries.

4. Miscellaneous

1951

September	Bomb fusion of turnings.

1952

April	Use of gallium oxide.
July	Air reductions.
August	Air reductions; preparation and use of gallium oxide.
September	Use of gallium oxide.

1953

June	Use of single crucible for different batch sizes.

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November	Large-scale reduction of calcium plutonium fluoride; small reductions; reduction of calcium fluoride, plutonium fluoride mixtures.
1954	
January	Test of new Task III pressure vessel.
Unreported	Spectrochemical analysis of metal from double salt reduction.
1955	
July	Studies on hot water pickling of reduction buttons.
August	Evaluation of Nelco Calcium without redistillation or grinding for Task III.
December	Oxidation of calcium metal in unreacted PuF_4-Ca mixtures to eliminate H_2 evolution during powder dissolution.
1956	
January	Reduction of CeO to metal using Ca plus H_2 booster showed cerium formation. Effects of slagging agents noted.
May	Reduction of PuO_2 by Ca resulted in no segregation or coalescence of metal. Cerium oxide reduction by Ca resulted in metal beads (cerium) up to 1/4" diameter.
June	Attempt to reduce 40-gram PuO_2 resulted in no visible metal formation.
1958	
January	Study of calcium reactivity and pressure buildup associated with reduction in Task III. Effect of moisture and H_2 on PuF_4 and Ca mixtures at room temperature.

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5. Metal Recovery

1951

September	Bomb fusion of turnings.

1955

September	
October	Controlled oxidation of metal turnings in argon and oxygen, subsequent fluorination in Freon-12. Direct conversion of metal turnings to PuF_3 with argon and Freon-12.
December	

1956

January	
March	

1957

November	Recovery of plutonium from skulls by vacuum melting - impractical on small or badly-oxidized fragments (10 percent yields). Vapor phase reduction with calcium was unsuccessful. Experiments started on melting skulls in $CaCl_2$ - CaF_2 eutectic.
December	Glascast crucibles fired at 23[illegible] F will contain the $CaCl_2$ - CaF_2 melt at 1000 C. Magnesia, CaF_2, quartz, sodium silicate impregnated magnesia were ineffective. Study of Ca reduction of PuO_2 in a $CaCl_2$ - CaF_2 melt.

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1959

January	Test or recovering Pu from skulls was successfully made by melting skulls with Ca-$CaCl_2$ at 1050 C. Recovery was >90 percent.
February	Recovery of skulls by calcium chloride salt bath.

6. High Purity Plutonium Metal

1955

September	0.5 kg high-purity Pu prepared.

1957

January	900-gram pure metal has been made. Total impurities of most recent batch is < 500 ppm.

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7. Plutonium Trichloride

1958

March	Reduction of $PuCl_3$ by Ca. Effects of moisture concentration.
	Study of chloride slag soaking into MgO crucibles. Use of Glascast reduction liners, and effect of calcium and plutonium on Glascast.
	Test of Al slag and crucible cans exposed to $CaCl_2$, CaI_2, and CaF_2 at room temperature. Physical strength of cans adequate after 30-day exposure.
April	Small-scale (30-g) $PuCl_3$ batch reduction. Effects of water, iodine, and calcium concentrations were investigated. No serious crucible (MgO) sticking problem on 30-g scale.
	Effect of Si and B pickup by Pu from Glascast crucibles.
May	Large reductions (0.6 - 1.0 kg) of $PuCl_3$ investigated. Button quality acceptable. Slag soaking problem in large reduction. Effect of dual crucibles as slag container.
June	Nickel-coated MgO crucibles prevent slag soaking and crucible sticking in reduction of $PuCl_3$ to Pu.
	Acceptable reductions made with $PuCl_3$ containing up to one percent water.
	$PuCl_3$ reduction yields are >96 percent.
	Effect of $PuCl_3$ storage on reduction chemistry.

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July

Effect of batch reduction compounds on various crucible materials studied.

Study initiated to determine optimum nickel-coat thickness for MgO reduction crucibles.

A phase diagram for $CaCl_2$ - CaI_2 system was determined.

Effect of plutonium trichloride produced from plutonium oxalate on the batch reduction characteristics.

August

Large-scale batch reductions of $PuCl_3$ to Pu in nickel-coated MgO crucibles. Yields and purity satisfactory when crucible does not break.

Coating thickness study continued.

$PuCl_3$ stored 260 hours at 8.5 percent relative humidity was successfully reduced to metal.

September

$PuCl_3$ stored 1300 hours at 8.5 percent relative humidity was reduced to metal with a 72 percent yield.

October

$PuCl_3$ formed at 600 C required additional Ca-I_2 booster for good reduction to metal.

A mixture of 55 percent $PuCl_3$ - 45 percent PuOCl was reduced to metal by calcium, with a 95 percent yield.

Study of optimum amount of water tolerated in $PuCl_3$ to give 96 percent reduction to metal. 0.6 percent water can be tolerated..

November

Study to determine feasibility of eliminating the chemical mixing step in $PuCl_3$ batch reductions. Effects of chemical segregation are discussed.

Use of salt-impregnated MgO crucibles was tested in 30-g scale $PuCl_3$ batch reductions.

Effect of substitution of CaF_2 for MgO sand in $PuCl_3$ reductions.

December

Study of $PuCl_3$ reduction with elimination of the mixing step

1959

January

Reduction of $PuCl_3$ from continuous chlorinator was success-

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ful. Powders used had bulk densities of 2.0 - 2.9 g/cc, no differences in reduction characteristics noted.

Tests of reduction with all of Ca and I_2 on top of $PuCl_3$ have resulted in 75 percent yields. Method was successful when all of I_2 and 2/3 Ca were on top and remainder of Ca below the $PuCl_3$.

A copper-coated MgO crucible was used successfully in a 700-gram $PuCl_3$ reduction.

A 30-g button was recovered when 50 g of Pu as $PuCl_3$ were dropped with calcium into a crucible at 800 C. The semi-continuous reduction atmosphere was argon.

February

Batch studies of $PuCl_3$ reductions without mixing of chemicals.

Reaction bomb loading safe temperature limits.

Reduction of lumps chlorides.

Tests of dual reduction liner technique in $PuCl_3$ reduction.

March

Study of steel-coated MgO crucible in $PuCl_3$ batch reductions.

Batch studies of $PuCl_3$ reduction without mixing chemicals, 98 percent reduction yield.

April

Reduction of $PuCl_3$ from large continuous chlorinator runs were successful.

Study of tantalum liners for batch reductions.

Phase diagram study for CaI_2 - $CaCl_2$ slag summarized in HW-60151, "Calcium Iodide - Calcium Chloride System".

May

Test of reusable NaCl-KCl impregnated MgO crucible for $PuCl_3$ batch reductions 20 - 200 gram scale.

Test of reusable nickel-coated MgO crucible on 700-gram $PuCl_3$ batch reductions.

June

Reusable crucible - studies.

$PuCl_3$ reduction studies in tantalum crucibles.

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D. RECUPLEX

1. Slag And Crucible Dissolution

1951

August	Odd experiments.
October	Comparison of Los Alamos and Hanford procedures.
November	Flow Sheet No. 1.
December	Additional dissolvings.

1952

January	Dissolution of sulfur slags.
May	Corrosion rates for dissolution.
June	Analysis of filter cakes; removal of iodate-sodium nitrite addition.
July	Silica analysis.
August	Plutonium holdup from dissolution.
September	Slag and crucible dissolution.

1953

May June	Filtration of slag crucible solution - alundum (Al_2O_3) plate.
November	Dissolution of calcium fluoride - calcium iodide - calcium sulfide slag.

1954

January	Silica in off-gas.
March	Aluminum-to-fluoride studies; plutonium(IV) in SCF.
April	Iodate formation.
June	Silica in off-gas.
October	Slag and crucible dissolution - SCX-1 corrosion rates, Haynes 25 and stainless steel in dissolution; iodate method.

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1955

May	Studies on dissolution of reduction slags, effects of CaI_2 content.
July	Studies on hydrogen formation in dissolver reactions.
August	Studies on dissolver off-gas. Analysis of silica cake slurry.

1958

February	Study of steel slag and crucible can dissolution in 8 M HNO_3 - 0.3 M HF.

2. Miscellaneous Feed Preparation

1951

November December	Clarification of F-10-P filtration; use of carbon tetrachloride.

1952

September	Use of nitrite for feed adjustment (3BP, F-10-P).

1954

August	Precipitation in F-10-P solution.
September	Valence adjustment.

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October	F-10-P and solvent extraction - greases; valence adjustment and F-10-P, 3BP.

1955

March	Valence adjustment studies on oxalate filtrates.
May	Valence adjustment studies on oxalate filtrates.
June	Effects of ferric ion on valence adjustment studies for oxalate filtrates.
August	Studies on reactivity of H_2O_2 as associated with Recuplex feed makeup.

3. Extraction Studies

1952

February	Disengaging times for CAF-CAX as a function of specific gravities.
June	Extraction coefficient values.
July	Extraction coefficient values.
August	Effect of iodate, sulfate, hydrogen peroxide - adjustment on extraction coefficients.
September	Extraction coefficients.

1953

August	Life test of coated plates; coefficients for HW-10.
October	Plate testing solvent stability.
November	Plate testing.

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1954

January	Flow Sheet EX-1: effect of oxalate; solvent decompositions.
April	EX-1 batch, counter-current runs; effect of oxalate - function of plutonium concentration.
July	Coefficients for Recuplex system; total reflux.
August	Coefficients for plutonium(VI).
September	Coefficients for 20 percent TBP; organic phase saturation; small column studies.
December	Coefficients for CC column - function of time.

1955

January	Study of decrease of E_a^o in the Recuplex stripping column when CCX is heated to 50 C.
	Proposed uranium-plutonium partition flow sheet for Recuplex. H-1 and H-2 columns or batch processing in the solvent treatment tank.
February	Solvent decomposition studies, effect of plutonium concentration and solvent age.
March	Solvent damage studies, effects of CCl_4, formation of Cl^- corrodant.
	Plutonium distribution studies and an equation for scrub section of H-1 column.
May	Recuplex flow sheets C-9 and C-11 tested in laboratory columns.
June	Recuplex flow sheet C-11 studies. Solvent damage studies.
August	Valence studies on Recuplex concentrated CCP stream.

1957

August	Dibutyl butylphosphonate (DBBP) results in more favorable extraction of the plutonium into the organic phase than TBP. However, stripping from DBBP has been ineffective. DBBP is more stable than TBP in aqueous HNO_3 solutions. Tetrabromoethane (TBE) will be tested as a density-increasing agent. Charts relating density to composition for the TBP-TBE-CCl_4 system have been prepared.

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Solvent density increase will permit plant capacity increase.

September

Further work confirms more favorable distribution coefficients for DBBP over TBP. Satisfactory stripping could not be attained by acid adjustment. Hydroxylamine sulfate (0.1 M) in CCX resulted in satisfactory stripping.

October

Satisfactory extraction and stripping was observed in batch tests using DBBP-TBE-CCl_4 solvent. CCX stream was 0.05 M HNO_3 - 0.05 M $(NH_2OH)_2SO_4$.

December

Further extraction tests of the DBBP-TBE-CCl_4 solvent shows a decrease in distribution coefficient due to presence of TBE.

Pentachloroethane, a high-density solvent (possible replacement for CCl_4) exhibited much lower extraction coefficients for Pu(IV) with TBP and with DBBP than found with CCl_4.

Trimethoxyboroxine (TBM) was subjected to Recuplex-type extraction and stripping. 90 percent of TBM went to raffinate. TBM is a possible extinguishing agent for Pu fires.

1958

January

Extraction study shows TBE in the DBBP-CCl_4 system decreases distribution coefficient of unsalted feeds and in stripping. No material effect on highly-salted feeds. Pentachloroethane behaves similarly when in place of CCl_4.

Effect of TBE and pentachloroethane on solvent degradation.

Corrosion study of stainless steel extraction plate attack by TBP-DBBP-CCl_4, TBE, CCl_4, and pentachloroethane.

February

Investigation of Recuplex extraction column emulsification, stripping column precipitation. Effect of dispersed silica and TBP degradation products on extraction. Effect of fluoride on stripping.

Extraction coefficient studies on solutions from dissolution of steel slag and crucible cans.

June

Stripping from Recuplex DBBP solvent resulted in much poorer results than previous work showed. Degradation

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products from DBBP act similar to DBP in TBP system.

August Addition of "Mistron 28" to Recuplex sump cleanout solutions eliminated column emulsification problems during cleanout runs. Solvent was TBP-CCl_4.

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4. Product Concentration

1953

August	Plutonium - red oil reactions.
October	Evaporation of CCP.

1954

January	Red oil and precipitation of peroxide or oxalate.

5. CAW Treatment

1952

April	Neutralization of CAW; viscosities; setting characteristics.
May	Viscosity of neutralized CAW.
November	Neutralization of CAW.

1954

October	Protective coatings for CAW crib.

6. Miscellaneous

1951

December	First cycle waste evaporation; corrosion and material testing.

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1952

January February	Caustic scrubber solutions; cannisters.
April	First cycle waste evaporation; corrosion and material testing.
July	Physical properties of process streams.
August	Solvent treatment; first cycle waste evaporation; corrosion and material testing.
September	Solvent treatment.
October	Sintered stainless steel - porosities; resistance of rubber to nitric acid, hydrogen peroxide.
	Corrosion of various materials in hydrofluorination off-gases.
November	Dissolution of fluorides in ANN-nitric acid.
	Porosities of platinum discs.
December	Corrosion of alloys in $HF-HNO_3$ mixes; G-9 asbestos gaskets in iodine.

1953

February	Skull dissolution; platinum liner destroyed.
May June	Dissolution of outstanding reduction charges (caustic dissolution tried).
August	Chemical resistance of Homalite.
November	Corrosion tests - metals in nitric - ANN solutions; corrosion in freon.
December	Corrosion in freon.

1954

January March November	Filtration of aluminum nitrate.
February	Fluorothene stability in process streams.
September	Use of Haynes 25 for HNO_3 - HF systems; Elgiloy in

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HNO_3 - HF vapors.

October	Corrosion of Haynes 25 in HNO_3 - HF solutions.
December	Filter cloth or aluminum nitrate filtration.
	Stainless steel corrosion in slag and crucible solutions.
1958	
March	Scavenging of Recuplex solvent by precipitation of PuF_4 from CAX (15% TBP in CCl_4) by HF or HF and NaF. Residual Pu in organic ~ 0.01 g/l.

E. MISCELLANEOUS

1952	
November December	Nickel carbonyl analysis.
1953	
December	Reaction of plutonium metal in nitrogen or in CO_2; ferrous sulfamate preparation by ion exchange.
1955	
February	Nickel carbonyl coating studies. Eleven uranium pieces coated to one mil thickness.
March	Nickel carbonyl coating studies.

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April	Nickel carbonyl coating studies, experimental data submitted for statistical analysis.
June	Use of ultrasonics in nickel dissolution.
December	Dissolution of nickel coatings from rejected shapes using Enstrip "S". Cost is 1/6 that of HNO_3 treatment and Pu waste loss is 0.6 g per piece treated.

1956

January	Removal of nickel coating from reject shapes by using Enstrip "S", including waste losses, time cycle, solvent disposal.
	Absorption spectra of chromic, nickelous, ferrous, ferric, permanganate, calcium, lanthanum, aluminum, and their interaction in nitrate solution investigated.
	Extinction coefficients for Pu in various valence states have been determined.

1957

May	Optimum nickel coating removal was accomplished with 6.5 M HNO_3 at 70 C in 15 minutes. Additional effects of acid concentration, time, and temperature are included.
April	A two-gram sample of PuO_2-UO_2 mixed crystal prepared by H_2 reduction at 900 C of ammonium diuranate - plutonium hydroxide formed by an ammonium hydroxide precipitation from a mixed nitrate solution. Pu/U = 1/5.

1958

March	Test of Eastman Adhesive 910 for attaching Plexiglas port rings to Homalite CR-39 plastic.
	New dry chemistry hoods installed in Rooms 185, 186, and 187.
May	Eastman 910 adhesive in use for attaching Plexiglas to Homalite CR-39 (Hood 9).
August	Shipment of PuO_2, <1400 ppm impurities, to Monsanto Co.
December	Test of radiation dose increase with plutonium concentration for solutions in polyethylene bottles. Effect of lead shields recorded.

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1959

March	Study of equipment and technology for critical mass preparations, PuO_2 suspensions in hydrogeneous materials.
May	Preparation of CeO - paraffin test cylinders, H/Pu - 15, .by atmosphere and pressure casting.

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F. CERAMIC DEVELOPMENT

1955

April	Test of MgO - 5% CaF_2 crucible. Slip casting studies.
June	Graphite mold preparations. MgO - 5% CaF_2 preparations.
July	Coated graphite molds prepared by spraying of MgO slurry.
August	Preparation of Al_2O_3, MgO, zirconia slip cast crucibles.

1956

November	Fabrication study on straight thermocouple wells produced from MgO. Essential dimensions and technique.
December	Flow sheet for improved method of preparing magnesium oxide slip.

1957

February	Nine-inch, coarse-mix, MgO chimneys made without use of hardened dies or other costly equipment. CaF_2 slip casting.
May	Development of routine method of alkaline slip casting of CaF_2 crucibles was demonstrated. Crucibles were smooth, and free of blisters. Effect of calcining temperature on slurry pH was investigated. Low CaO impurities did not affect stability of crucible.
June	Doughnut-shaped setter plates show promise in reducing CaF_2. Crucible distortion experienced with circular setter plates.
July	CaF_2 (-48 mesh, calcined at 1200 C) required 12-hour ball milling to attain best slip casting properties. Patterns for ceramic reduction cells are being prepared in support of continuous Task III studies. A guillotine button cutter has been tested using aluminum buttons 1-1/8" thick. Twenty tons' force is required to cut the button.
August	Work is in progress on a four-compartment vibration-

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cast crucible. Cracks formed during firing at 1760 C. Slip cast shells (from the I-4 pattern) appear to be more promising and will be tested.

The guillotine button cutter was put into use. Cutter was operated by a one-ton press, and worked well until damaged by maloperation. A new model was built, but does not have suitable springs or button holder.

September

Acceptable four-compartment I-4 vibration-cast crucibles were produced by eliminating Carbowax from the formulation and substituting magnesium sulfate solution.

Blister-free slip cast CaF_2 crucibles were achieved by de-airing the slip by means of an evacuated desiccator and lowering the sintering temperature from 1100 C to 1000 C.

Magnesia crucibles were impregnated with molten CaF_2 at 1400 C. The crucibles thus treated were imprevious to water, while ordinary crucibles are not.

October

Difficulties in fabricating four-place magnesia casting mold (CS-201) were overcome.

A magnesia pouring crucible was successfully coated with 1/32" nickel.

Glascast was used successfully for slip casting tensile specimen molds.

November

Plutonium tensile specimens cast in Glascast molds are fine-grained, superior strength. Surface impurities of B, 4 ppm and Si, 200 indicate promise of Glascast in plutonium casting.

December

Studies initiated to form Glascast shell mold for Pit 65.

Glascast crucibles fired at 2300 F developed for skull recovery employing $CaCl_2$-CaF_2 melt at 1000 C.

1958

January

Problems of removing Glascast Pit 65 casting molds from plaster form overcome.

CaF_2 crucibles fired using green MgO setter plates to prevent cracking.

March

MgO crucibles nickel-coated externally for batch and

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continuous Task III.

April	Method for slip cast MgO continuous reduction cell with integral drain tube and riser.
June	
July	Tests of impregnating MgO crucibles with salts to reduce porosity. Initial fabrication of MgO liners for Rokide Z-coated pressure vessel. Nickel-coated MgO crucible study.
September	A paper, "Slip Casting Of High Purity Calcium Fluoride Crucibles", was prepared.
October	Test of impregnating MgO crucibles with salts to reduce porosity.
1959	
May	Test of H-7 pressed sintered high-silica and I-10 MgO-CaF_2 investment-cast pouring crucibles.
June	Results of the H-7 and I-10 crucible tests in pouring applications. H-7 most promising.

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G. PYROCHEMICAL PROCESSING

1. Continuous Task III

1958

March	Preparation of document on continuous electrodeposition of Pu metal--development equipment.
April May	Container studies started to determine suitable material for the NaCl-KCl-$BaCl_2$ melt at 800 C. Zircon, stabilized zirconia, high-density alumina, nickel-coated MgO show promise. Studies used argon and H_2-HCl atmospheres.
June	Initial solubility study for $PuCl_3$ in NaCl-KCl-$BaCl_2$ melt.
	Container study continued using system of $CeCl_3$, NaCl-KCl-$BaCl_2$ melt, and Ce in argon at 800 C. Effect of melt penetration and sticking noted.
	Installation of continuous reduction cell started in Hood 31.
July	Thoria, pyroceram, and molybdenum crucible tests in the NaCl-KCl-$BaCl_2$ melt, argon, H_2, HCl system at 800 C.

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August	Description of newly-installed continuous Task III equipment. Tests of crucibles in a mixture of plutonium metal, $BaCl_2$-KCl-NaCl melt, and $PuCl_3$ in argon at 800 C. Alumina crucible was most promising. Effect on other materials are recorded.

2. Miscellaneous

1958

August	Reduction of CeO to Ce with calcium in CaF_2-$CaCl_2$ eutectic.

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H. CASTING

1956

January

September

November

1957

January

February

May

June

November

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I. HIGH-PURITY PLUTONIUM

1956

February	First reduction of high-purity plutonium fluoride, small button, 10 percent yield; effect of water in reaction vessel.

1957

May	Two reductions to metal of purified PuF_3 were performed in CaO and CaF_2 crucibles. Button can be easily removed from CaF_2 crucible. High-purity CaF_2 crucibles are now available. Main impurities were: Run #6 Ca 20 - 40 ppm; Ni 10 - 100; Fe 150; density 19.54. Run #7 Al 8 - 20; Fe 50 - 200; Mg 10; density 19.36.
June	High-purity reduction (Run #8) with CaF_2 crucible using calcium with 60 ppm C resulted in 25 percent yield. Major impurities: Fe 69 ppm; Mg 100; Mn 10; Na 10; C 80.

1958

February	Metal button dissolved, purified by peroxide precipitation and hydrofluorinated to PuF_4 for special radiation studies. Total metallic impurities 100 ppm.

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J. HIGH-EXPOSURE PLUTONIUM

1958

February	Radiation dosage study on high-exposure plutonium. Effect of increase in hard gamma radiation on dosage rate.
March	Documents on capability and program of high-exposure plutonium button line.
June	Design of loading areas, powder trap, calciner-chlorinator combination, and product hood are completed. Chemical flow sheet and flow diagram have been issued.

1959

February	High exposure button line program cancelled. Details in notes of H. W. Crocker.
March	Initial design of laboratory equipment for processing PRTR spike fuel was started.
May	High-exposure plutonium processing summarized in HW-60297, "234-5 Development Processing Facility For PRTR Plutonium".

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Z. DOCUMENTS ISSUED

PERIODICAL SEPARATIONS TECHNOLOGY AND RESEARCH AND ENGINEERING REPORTS

1951

January	HW-20161 H
February	HW-20438 H
March	HW-20671 H
April	HW-20991 H
May	HW-21260 H
June	HW-21506 H
July	HW-21802 H
August	HW-22075 H
September	HW-22304 H
October	HW-22610 H
November	HW-22875 H
December	HW-23140 H

1952

January	HW-23437 H
February	HW-23698 H
March	HW-23982 H
April	HW-24337 H
May	HW-24605 H
June	HW-24928 H
July	HW-25227 H
August	HW-25533 H
September	HW-25781 H
October	HW-26047 H
November	HW-26376 H
December	HW-26720 H

1953

January	HW-27522
February	HW-27739
March	HW-27624 H
April	HW-28355
May - June	HW-28695
July	HW-29035
August	HW-29504
September	HW-29692
October	HW-30158
November	HW-30486
December	HW-30677

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1954

January	HW-31078	
February	HW-31336	
March	HW-31732	
April	HW-31956	
May	HW-32070	
June	HW-32189	
July	HW-32818	
August	HW-33150	
September	HW-33750	
October	HW-34072	
November	HW-34161	
December	HW-34147	H

1955

January	HW-34631	H
February	HW-35530	H
March	HW-35891	H
April	HW-36440	H
May	HW-36928	H
June	HW-37658	H
July	HW-38375	H
August	HW-38828	H
September	HW-39260	H
October	HW-39751	H
November	HW-40182	H
December	HW-40692	H

1956

January	HW-41205	H
February	HW-41702	H
March	HW-42219	H
April	HW-42626	H
May	HW-43137	H
June	HW-43938	H
July	HW-44580	H
August	HW-45115	H
September	HW-45707	J
October	HW-46432	J
November	HW-47056	J
December	HW-47675	J

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1957

January	HW-48132 J
February	HW-48835 J
March	HW-49503 J
April	HW-50089 J
May	HW-50584 J
June	HW-51211 J
July	HW-51802 J
August	HW-52353 J
September	HW-52864 J
October	HW-53449 J
November	HW-53967 J
December	HW-54319 J

1958

January	HW-54821 J
February	HW-55215 J
March	HW-55571 J
April	HW-55914 J
May	HW-56218 J
June	HW-56602 J
July	HW-56972 J
August	HW-57328 J
September	HW-57640 J
October	HW-58051 J
November	HW-58305 J
December	HW-58711 J

1959

January	HW-59079 J
February	HW-59434 J
March	HW-59849 J
April	HW-60236 J
May	HW-60559 J
June	HW-60915 J
July	HW-61366 J
August	
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PROGRAM DOCUMENTS

DOCUMENT NUMBER	TITLE	AUTHOR	ISSUE DATE	CLASSIFICATION
HW-18738	Use Of AT Solutions Without Evaporation	JF Facer WL Lyon	9/1/50	Secret
HW-18796	Direct Hydrofluorination Of Plutonium(III) Oxalate	JF Facer WL Lyon	9/11/50	Secret
HW-19122	Recovery Of Plutonium From Skulls	JF Facer WL Lyon	10/12/50	Secret
HW-19771	Gas Composition For The Direct Hydrofluorination Of Pu(III) Oxalate	JF Facer WL Lyon	12/26/50	Secret
HW-19882	Filtration Of Plutonium Peroxide Slurry	WL Lyon	1/5/51	Secret
HW-20164	The Preparation Of Plutonium Tetrafluoride By Direct Hydrofluorination Of Pu(III) Oxalate	JF Facer WL Lyon	2/1/51	Secret
HW-20229	Report Of Invention - Direct Hydrofluorination Of Pu(III) Oxalate or Pu(IV) Oxalate	JF Facer WL Lyon	2/9/51	Secret
HW-20621	Report Of Invention - Oxidation Of Plutonium Metal Using Water At 140 C To Form A Soluble Powder	JF Facer WL Lyon	3/26/51	Secret
HW-20622	Report Of Invention - Dissolution Of Plutonium Metal Using 16 $\underline{M}$ HNO_3 - 0.04 $\underline{M}$ HF	JF Facer WL Lyon	3/26/51	Secret
HW-21082	Treatment Of 234-5 Building Wet Chemistry Waste Solutions For R Recycle	RE Falkowski RA Pugh	5/23/51	Secret
HW-21616	Treatment Of 234-5 Building Concentrated Oxalate Waste Solutions For Recycle	RA Pugh	7/6/51	Secret
HW-21691	Laboratory Evaluation Of One And Two Cycle Peroxide Processing Of F-10-P Solutions On The Basis Of Metal Purity Obtained	WL Lyon	7/19/51	Secret

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DOCUMENT NUMBER	TITLE	AUTHOR	ISSUE DATE	CLASSIFICATION
HW-21751	The Use Of Plutonium Peroxide For The Precipitation Of Plutonium Tetrafluoride	WL Lyon B Weidenbaum	7/26/51	Secret
HW-21897	Production Of Plutonium Tetrafluoride To Metal In Charges Containing Calcium And Sulfur	WL Lyon	7/26/51	Secret
HW-22136	The Recovery Of Plutonium From Metal Wastes	JF Facer B Weidenbaum	9/12/51	Secret
HW-22232	Preliminary Memorandum On Processing Of Plutonium Metal Turnings	WL Lyon	9/24/51	Secret
HW-22262	Report Of Invention - The Conversion Of Plutonium Metal Turnings To Massive Metal By A Bomb Fusion Process	WL Lyon	9/26/51	Secret
HW-22394	Report Of Invention - The Dissolution Of Plutonium From Metal Processing Wastes	JF Facer	9/18/51	Secret
HW-23019	Removal Of Hydrogen Fluoride At Low Concentrations In Air Streams By Activated Charcoal And Sodium Fluoride	GE Benedict KM Harmon	12/5/57	Restricted
HW-23132	234-5 Building Program Committee Meeting Minutes	KM Harmon	12/29/51	Secret
HW-24495	Removal Of Hydrogen Fluoride From An Air Stream By Adsorption In A Dorex Charcoal Filter	KM Harmon	5/16/52	Secret
HW-25401	Filter Aids For Isolation Building Plant Use	JF Facer KM Harmon	8/15/52	Secret
HW-25402	Addition Of 70-58 Oxide In Task III	KM Harmon WL Lyon MN Myers	8/15/52	Secret
HW-25468	Further Evaporation Of Residual Solution From The First Cycle Waste Evaporator	GE Benedict	8/28/52	Secret

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DOCUMENT NUMBER	TITLE	AUTHOR	ISSUE DATE	CLASSIFICATION
HW-25562	Processing Of Isolation Building Cleanout Solution	JF Facer KM Harmon	9/16/52	Secret
HW-25771	Capacity Of Marble Chips To Absorb HF Gas	KM Harmon	9/29/52	Unclassified
HW-25779	Basis And Program For Development Work On Plutonium Reduction-Casting	WL Lyon	9/26/52	Secret
HW-25844	Filter Media For Plutonium(IV) Oxalate	JF Facer MN Myers	9/11/52	Secret
HW-25955	Valve Stem Cones For Motor-Operated Throttling Valves To Give Close Remote Control Of Task II Furnace Gases	GE Benedict	10/17/52	Secret
HW-25986	Recovery Of Plutonium From Bismuth Phosphate Decontamination Runs	KM Harmon RA Pugh	10/21/52	Secret
HW-26092	Use of Counter-Current Caustic Scrubber Column For Removal Of HF From Hydrofluorination Off-Gases	GE Benedict	11/3/52	Secret
HW-26758	Observations On Mixing Of Redox And Uranium Recovery Plant Neutralized Aqueous Wastes	KM Harmon RA Pugh	1/6/53	Secret
HW-27488	Laboratory Evaluation Of Double Batch Size Reductions	KM Harmon WL Lyon	3/27/53	Secret
HW-27562	Laboratory Batch Evaporation Of Redox PR Solution	RL Beede JF Facer RA Pugh	4/3/53	Secret
HW-27563	The Precipitation Of Plutonium(IV) Oxalate In Task I	JF Facer KM Harmon	4/3/53	Secret
HW-27837	Procedures For Hydrogen Peroxide Addition In Valence Adjustment For Plutonium(IV) Oxalate Precipitation	JF Facer	7/29/53	Secret
HW-27881	Laboratory Evaluation Of The Use of Sulfur In Reduction	WL Lyon	5/1/53	Secret

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DOCUMENT NUMBER	TITLE	AUTHOR	ISSUE DATE	CLASSIFICATION
HW-28168	The Precipitation Of Plutonium(IV) Oxalate In Task I, Nitric Acid Tolerances, Reactor Dimensions	KM Harmon	5/26/53	Secret
HW-28177	Preliminary Menorandum On Combined Reduction And Casting Of Plutonium	WL Lyon	6/1/53	Secret
HW-28357	The Preparation Of Plutonium Alloy Castings In The Reduction Crucible	WL Lyon	10/1/53	Secret
HW-28789	Recovery Of Plutonium From Off-Standard Fluorides	RL Beede KM Harmon	6/29/53	Secret
HW-28838	Report Of Invention - Reduction Casting Development	WL Lyon	7/27/53	Secret
HW-29200	Plutonium Purification And Fabrication Manual (Chemical Technology Sections)	WL Lyon	4/1/54	Secret
HW-30015	Dissolution Of Slag And Crucible Residues For Plutonium Recovery	RA Pugh WL Lyon	11/1/53	Secret
HW-30036	Shipment Of Laboratory Slag And Crucible Solution	KM Harmon	11/18/53	Secret
HW-30188	Trip Report: Iowa State College	WS Figg	11/20/53	Secret
HW-30558	Laboratory Study Of Causes For High Solubilities Of Plutonium Peroxide	RA Pugh	1/6/54	Secret
HW-31186	Precipitation Pu(IV) Oxalate	JF Facer KM Harmon	3/30/54	Secret
HW-31628	Progress Report: Continuous Task II Program	WS Figg	4/13/54	Secret
HW-32100	Notes Pertaining To Recuplex Product Evaporation	RA Pugh	4/28/54	Secret
HW-33640 RD	Plutonium Requirements For 234-5 Development Program	KM Harmon	11/3/54	Secret - Rough Draft

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DOCUMENT NUMBER	TITLE	AUTHOR	ISSUE DATE	CLASSIFICATION
HW-33666 RD	Supernatant Kill In Task I	KM Harmon	11/4/54	Secret - RD
HW-34385	Recycle Solution Composition - Z Plant To Redox Plant	RA Pugh	1/10/55	Secret
HW-34540	Chemical Reactivity Of Plutonium Metal	KM Harmon RC Smith	1/24/55	Secret
HW-35727	Plutonium(IV) Oxalate Precipitation In The Task I Prototype	JF Facer	3/7/55	Secret
HW-35767	F-10-P Solution Studies	GE Benedict MN Myers RA Pugh	3/14/55	Secret
HW-36173	Continuous Task II Program - Plutonium(IV) Oxalate Calcination And Hydrofluorination Studies	WS Figg	4/12/55	Secret
HW-37114	Trip Report: Los Alamos Scientific Laboratory	RC Smith	6/7/55	Secret
HW-37115	Trip Report: Los Alamos Scientific Laboratory	RC Smith	6/7/55	Secret
HW-38555	Direct Concentration Of Redox Product Solution	RA Pugh	10/19/55	Secret
HW-38637	Media For Filtration Of Aluminum Nitrate Solution	RL Beede	8/12/55	Secret
HW-38753	Hydrogen Production In The Calcium And Magnesium - Nitric Acid Reactions	MN Myers	8/19/55	Official Use Only
HW-39012	Laboratory Evaluation Of Nelco Calcium For Plutonium Tetrafluoride Reduction	RL Beede	9/15/55	Confidential
HW-39080	Effect Of Ultrasonics On Dissolution Of Nickel In Nitric Acid	RC Smith R Wirta	9/20/55	Unclassified
HW-39328	Preliminary Flowsheet - Continuous Tasks I and II	HH Hopkins, Jr.	10/5/55	Secret

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DOCUMENT NUMBER	TITLE	AUTHOR	ISSUE DATE	CLASSIFICATION
HW-39513	Concentration Of Redox Product With Increased Aluminum Concentration	MN Myers	10/12/55	Secret
HW-39581	Preliminary Coupling Flowsheet, Redox And Purex, For Task I Replacement	RA Pugh	12/28/55	Secret
HW-39880	Controlled Oxidation Of Plutonium Metal Turnings	RC Smith DR Doman	11/10/55	Secret
HW-40178	Plutonium Content Of Recuplex Slag And Crucible Filter Slurries	MN Myers	11/29/55	Confidential
HW-40274	Separan 2610 As Task I Flocculant	RL Beede	12/5/55	Confidential
HW-40275	Redox Product Coupling To Task I and 231 Building Processes	RL Beede	12/5/55	Confidential
HW-40781	Permanent Mold Casting Program	RC Smith	12/21/55	Secret
HW-40996	Report Of Invention: Use of Fluosilicate For The Precipitation Of Plutonium Fluoride	RL Beede	1/19/56	Secret
HW-41100	Recommendation For Inclusion Of Continuous Task I In Scoping Of Continuous Task II	HH Hopkins, Jr.	2/22/56	Confidential
HW-41286	Recommendations For Study Of Reflux And Modified Plutonium Concentrator Operation	HH Hopkins, Jr.	2/2/56	Secret
HW-41464	Evaluation Of Enstrip "S" For Stripping Nickel	RL Beede CC Wheeler, Jr.	2/17/56	Secret
HW-41635	Technical Specification Letter No. 2 - Continuous Task II - Calcination Reactor Substitution	WS Figg HH Hopkins, Jr.	2/23/56	Secret
HW-42660	Technocal Specification Letter #1 Permanent Mold Casting	HH Hopkins, Jr.	4/25/56	Secret

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DOCUMENT NUMBER	TITLE	AUTHOR	ISSUE DATE	CLASSIFICATION
HW-43953	The Use Of Freon-12 In the Conversion Of Plutonium(IV) Oxalate To Plutonium Trifluoride	MN Myers	6/27/56	Confidential
HW-44258	Technical Specification Letter No. 2 Continuous Task I	HH Hopkins, Jr. RL Beede	7/11/56	Confidential
HW-44744	Absorption Spectra Of Plutonium And Impurity Ions In Nitric Acid Solution	MN Myers	7/31/56	Declassified
HW-44987	Reduction Of Plutonium(VI) With Hydrogen Peroxide	MN Myers	8/17/56	Confidential
HW-45700	Continuous Plutonium(IV) Oxalate Precipitation, Filtration, And Calcination Process	RL Beede	9/27/56	Secret
HW-45917	Conversion Of Plutonium Trifluoride To Plutonium Tetrafluoride With Oxygen-Hydrogen Fluoride Mixtures	RL Beede	10/4/56	Secret
HW-46711	Evaporative Kill Of Oxalate Supernatants	CJ Berglund	11/30/56	Confidential
HW-46779	Corrosion Of Stainless Steel, Titanium, And Tantalum In Plutonium-Nitric Acid Solutions	A Brunstad	11/27/56	Unclassified
HW-48914	Continuous Plutonium Reduction--Processes Recommended For Development	MH Curtis	1/16/57	Secret
HW-49121 RD	Plutonium Distribution In Recuplex Systems	WL Lyon	Undated	Secret - Rough Draft
HW-49122 RD	Annual Report - CY 1956, 234-5 Development	HH Hopkins, Jr.	1/1/57	Secret - Rough Draft
HW-49124-RD	Thermometallic Reduction	MH Curtis	10/29/56	Confidential - RD
HW-49125 RD	Possible Continuous Plutonium Reduction Methods	MH Curtis	10/29/56	Confidential - Rough Draft
HW-49126 RD	Thermo-Decomposition Of Plutonium Iodide	MH Curtis	10/19/56	Confidential - Rough Draft

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DOCUMENT NUMBER	Title	AUTHOR	ISSUE DATE	CLASSIFICATION
HW-49138 RD	Research And Development Study TS-R, 234-5 Development	HH Hopkins, Jr.	6/8/56	Secret - Rough Draft
HW-49147	Continuous Plutonium Trifluoride Precipitation Progress Report	CJ Berglund	3/18/57	Confidential
HW-48900	Casting Of Plutonium In Copper Molds - Interim Report	RC Smith	1/17/57	Secret
HW-49464 RD	Research And Development Study TS-5, 234-5 Development FY-58	HH Hopkins, Jr.	4/1/57	Confidential - Rough Draft
HW-49699	Laboratory Plutonium Purity	HH Hopkins, Jr.	4/17/57	Secret
HW-49740	Trip Report - Knolls Atomic Power Laboratory, Rocky Flats Plant, Savannah River Plant	HW Crocker MH Curtis	4/19/57	Secret
HW-50280	Continuous Concentration Of Cation Exchange Product	RE Latta HH Hopkins, Jr.	5/20/57	Secret
HW-50597	Coating Removal With Dilute Nitric Acid	A Brunstad	7/31/57	Secret
HW-51276	Trip Report - Rocky Flats Plant, Savannah River Laboratory, Oak Ridge National Laboratory, And Argonne National Laboratory	HH Hopkins, Jr.	7/8/57	Secret
HW-51655	Oxidation Of Plutonium(III) By Sodium Nitrite	A Brunstad	7/19/57	Confidential
HW-52796	Reduction And Stabilization Of Plutonium Nitrate In Cation Exchange Feed And Product Solutions	A Brunstad RC Smith	9/24/57	Confidential
HW-53339	Trip Report, Argonne National Laboratory and Rocky Flats Plant, 9/2/57 - 9/5/57	RC Smith	10/24/57	Secret
HW-54203	Polymerization And Precipitation Of Plutonium(IV) In Nitric Acid	A Brunstad	12/17/57	Unclassified

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DOCUMENT NUMBER	TITLE	AUTHOR	ISSUE DATE	CLASSIFICATIO
HW-54405	The Stability Of Anhydrous Plutonium Trifluoride To Saturated Water Vapor At Room Temperature	RE Latta	1/7/58	Confidential
HW-54578	Laboratory Production Of Pure Plutonium	RE Latta	1/15/58	Secret
HW-54610	Annual Report 234-5 Development Operation - CY 1957	HH Hopkins, Jr.	1/20/58	Confidential
HW-54613	R&E Study 234-5 Development - CY-58 (July 1957 - December 1958)	HH Hopkins, Jr.	1/20/58	Confidential
HW-54874	A Plan For Plutonium Trichloride Process Development	HH Hopkins, Jr. by HW Crocker	2/6/58	Secret
HW-55072	Continuous Electrodeposition Of Plutonium Metal Developmental Equipment Considerations	MH Curtis	2/21/58	Confidential
HW-55349	Turbidimetric Microdetermination Of Sulfate In Plutonium Solution	HD Warren A Brunstad	3/14/58	Unclassified
HW-55357	Solubility Of Plutonium(IV) Sulfate In Nitric Acid Solutions	HD Warren A Brunstad	3/14/58	Unclassified
HW-55369	Capability Of Proposed Development Button Line For High-Exposure Plutonium	HH Hopkins, Jr.	3/18/58	Confidential
HW-55434	Program - Development Laboratory High-Exposure Button Line	HH Hopkins, Jr. by Members of 234-5 Development Laboratory	3/21/58	Confidential
HW-55674	Emulsification And Precipitation in the Recuplex Columns	A Brunstad	4/9/58	Confidential
HW-55713	Trip Report - Los Alamos Scientific Laboratory and Rocky Flats Plant, April 7 - 9, 1958	HW Crocker HH Hopkins, Jr.	4/14/58	Secret - Atomic Weapon Date
HW-56606 RD	Laboratory Evaluation Of Dibutyl Butylphosphonate	A Brunstad	7/1/58	Confidential - Rough Draft

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DOCUMENT NUMBER	TITLE	AUTHOR	ISSUE DATE	CLASSIFICATION
	And Tetrabromoethane For The Recuplex Solvent System			
HW-57362	Neptunium Oxide Shipment	HH Hopkins, Jr.	9/5/58	Secret
HW-57498	Status Of Plutonium Trichloride Process Development	HH Hopkins, Jr.	9/17/58	Secret
HW-58021	Precipitation Method For The Recovery Of Plutonium From Chloride Slag And Crucible - Progress Report	RW Henkens	10/30/58	Confidential
HW-58095	Recovery Of Plutonium From Chloride Slag And Crucible Solutions	RW Henkens	11/6/58	Confidential
HW-58763	Annual Summary And Program Review, 234-5 Development Operation	HH Hopkins, Jr.	1/6/59	Confidential
HW-59065	234-5 Development Support Of Prototype Button Line - Summary For January	HH Hopkins, Jr.	1/30/59	Confidential
HW-59749	The Analysis Of Exhaust Products In The Chlorination Of Plutonium Oxide With Phosgene	LF Lust	3/24/59	Confidential
Undocumented	Trip Report - Technical Meeting Sessions Of The Metallurgical Society, San Francisco, 2/15 - 19, 1959	RC Smith	3/25/59	Unclassified
HW-59796	Effect Of Titanium In Z-Plant Process	HH Hopkins, Jr. by HW Crocker	3/27/59	Confidential Undocumented
HW-60151	Calcium Iodide - Calcium Chloride System	TS Soine	4/24/59	Unclassified
HW-60153	Trip Report - Ames Laboratory, Rocky Flats Plant, And Los Alamos Scientific Laboratory	HH Hopkins, Jr.	4/24/59	Confidential
HW-60297	234-5 Development Processing Facility For PRTR Plutonium	HH Hopkins, Jr. by HW Crocker	5/4/59	Confidential
HW-60353	Trip Report - Rocky Flats	RS Rosenfels	5/13/59	Secret

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DOCUMENT NUMBER	TITLE	AUTHOR	ISSUE DATE	CLASSIFICATION
	Plant And Los Alamos Scientific Laboratory			
HW-60492	234-5 Development Processing Facility For Critical Mass Fuel Preparation	HH Hopkins, Jr. by HW Crocker	5/27/69	Unclassified
HW-61564 RD	Batch Reduction Of Plutonium Trichloride	TS Soine	8/14/59	Secret - Rough Draft
HW-61565 RD	The Continuous Chlorination Of Plutonium Dioxide	MJ Rasmussen	8/14/59	Confidential - Rough Draft
HW-61570	Status Of Plutonium Trichloride Process Development (II)	HH Hopkins, Jr.	8/14/59	Secret
HW-61577	Recovery Of Plutonium From Chloride Slag And Crucible	RC Smith	8/14/59	Confidential

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