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Technology - Hanford Processes

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**HANFORD WORKS**

**SEPARATIONS PRODUCTION TESTS  
AND PROCESS CHEMISTRY REPORTS**

**A BIBLIOGRAPHY**

By

R. V. Winsor

Technical Information  
Technical Services Unit

November 24, 1952

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RICHLAND, WASHINGTON**

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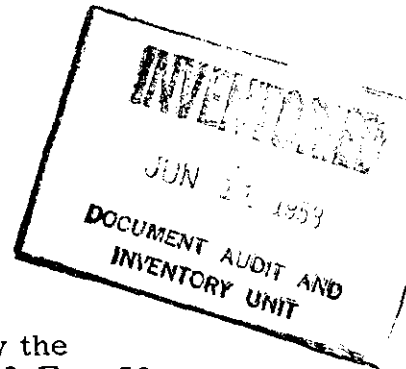
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HANFORD WORKS  
SEPARATIONS PRODUCTION TESTS  
AND PROCESS CHEMISTRY REPORTS  
A BIBLIOGRAPHY

INTRODUCTION

This bibliography is a compilation of reports on Hanford Works Separations Production Tests and Process Chemistry Reports (SE-PC Reports) issued prior to November 1, 1952. A few Process Chemistry Section reports issued prior to the assigning of SE-PC numbers have also been included.

The Production Tests have been arranged numerically and then alphabetically according to Building numbers. Extraneous letters in the designation of the early tests have been ignored. The Process Chemistry reports which have no SE-PC number are arranged according to date and the SE-PC Reports are arranged numerically.

An author index, a report number index, and a subject index are included.

*R. V. Winsor*

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R. V. Winsor

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SEPARATIONS PRODUCTION TESTS

PRODUCTION TEST SE-1

Test Proposal

HW-25033

Christianson, E. B.

"Production Test Request No. SE-1" (11-20-44)

Objective: To develop information which will permit the preparation of recommendations regarding standard procedures and conditions for normal and emergency operations for ventilating systems in 221-T, 271-T and 224-T Buildings.

PRODUCTION TEST SE-2

Test Proposal

HW-7-929

Evans, P. B.

"Production Test Request SE-2" (11-21-44)

Object: To develop a method for testing filter installations in 231 Building.

Reference

HW-7-989, P. B. Evans, "Production Test SE-2", (12-6-44).

PRODUCTION TEST 200-1

Test Proposal

HW-17469

Szulinski, M. J.

"Recovery of Plutonium Solutions" (4-7-50)

Objective: To recover approximately 121.6 grams of plutonium from 13 liters of solution, formerly used as a source of americium-241 at another site, and 30 grams of plutonium in 319 liters of aluminum bearing solution from which neptunium-237 had been extracted at another site.

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PRODUCTION TEST 221-B-1

Test Proposal

HW-3-2397

Acken, M. F.

"Alternate Coating Removal Procedure: Addition of Sodium Hydroxide at a Controlled Rate to Boiling Sodium Nitrate Solution" (5-14-45)

Objective: To demonstrate a coating removal procedure which offers more positive control of the aluminum-sodium hydroxide reaction rate and is independent of the size of the charge.

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

After performing seven coating removal operations in the B Canyon, the procedure was adopted as standard.

PRODUCTION TEST 221-T-1

Test Proposal

HW-3-1723

Apple, R. S.

"Elimination of Second Precipitation and Second Centrifugation in First Cycle By-Product Step" (3-1-45)

Objective: To determine the reduction, if any, in the process decontamination factors resulting from the elimination of the second scavenger precipitation and centrifugation in the first cycle by-product step. The purpose of eliminating this part of the step is to shorten the step time from approximately 25 hours to 20 hours.

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Production Test 221-T-1 (contd.)

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

The elimination of the second scavenger precipitation and centrifugation in the first decontamination cycle resulted in a reduction in processing time in Section 13 of approximately eight hours at practically no cost in over-all decontamination, but at a cost of approximately two in the decontamination obtained through the canyon. The greater portion of decontamination lost in the Canyon Building was regained in the Concentration Building, particularly in Cell A (bismuth phosphate by-product step). This small loss of decontamination was not considered serious (see P. T. 221-T-5) and the change was adopted as standard procedure.

PRODUCTION TEST 221-B-2

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

"Potassium Hydroxide Wash (Metathesis) of First and Second Cycle Product Cakes". This is a companion test to 221-T-8. This test was not performed in the B Canyon, but was performed in the T Canyon.

PRODUCTION TEST 221-T-2

Test Proposal

HW-3-2031

Apple, R. S.

"Reducing Time Cycles by Shortening Non-Process Times" (4-4-45)

Objective: To shorten all non-process times as much as possible in order to demonstrate a time cycle of under 24 hours in each section. Particular emphasis is being placed on sections 8, 14, and 17 which are the "bottleneck" sections in the Canyon Building.

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HW-26352 08L

Production Test 221-T-2 (contd.)

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

The changes enumerated, together with increased efficiency of the operating personnel resulted in a demonstrated (10 runs) average cycle time of under 19 hours for each section in the Canyon Building.

Reference

HW-3-2002, L. Squires, "Program for Increased Output of the Separation Plants", (4-3-45).

PRODUCTION TEST 221-B-3

Test Proposal

HW-3-3241

Apple, R. S.

"Operation of B Plant Using One Ventilating Stack Fan (291 Building)" (11-27-45)

Objective: To operate B Plant with one exhaust fan over an extended period for the purpose of obtaining additional plant data on: (1) the rate of activity build-up on the operating fan; (2) the rate of activity decay on the idle fan; (3) the effect of reduced stack dilution upon special hazards around the area; and (4) the effect of reduced ventilating air upon operations in the Canyon and Concentration Buildings.

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HW-26352 DEL

Production Test 221-B-3 (contd.)

Report

HW-3-3381

Apple, R. S.

"Operation of B Plant Using One Ventilating Stack Fan (291 Building)"  
(1-28-46)

Shutting down a running, contaminated ventilating fan permits the activity on the fan to decay with a half-life of approximately 30 days. At the same time, the activity build-up on the operating, contaminated ventilating fan approximates a doubling period of 100 days. Ventilation in the Canyon and Concentration Buildings has not been affected adversely by one fan operation.

PRODUCTION TEST 221-T-3

Test Proposal

HW-3-2029

Apple, R. S.

"Reduction in Process Volumes Transferred from the Canyon Building to the Concentration Building" (4-4-45)

Objective: To reduce the volume which must be processed in 224 Building by a factor of approximately two. This reduction in volume is necessary in order that 224 may realize a 24-hour time cycle.

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

Enumerates changes by which the process volume was reduced stepwise through the Canyon Building in a manner which resulted in the Concentration Building receiving charges which were 60 per cent of their former volume. This was accomplished without significant loss of yield in the Canyon Building and the procedure was adopted as standard.

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Production Test 221-T-3 (contd.)

Reference

HW-3-2002, L. Squires, "Program for Increased Output of the Separation Plants" (4-3-45).

PRODUCTION TEST 221-B-4

Test Proposal

HW-3-3345

Apple, R. S.

"Plant Test to Decrease Phosphoric Acid Consumption in the Canyon Building" (1-9-46)

Objective: To decrease the quantity (and concentration) of phosphoric acid used in each product precipitation step in the Canyon Building, for the purpose of reducing a principal item of cost in the canyon separation process.

Final Report

HW-7-4630

Apple, R. S.

"Plant Test to Decrease Phosphoric Acid Consumption in the Canyon Building" (8-22-46)

The results show that the extraction step can be performed at 0.5M phosphoric acid and the product precipitation step of the decontamination cycles made at 0.4M phosphoric acid with a saving of 1680 pounds of 100 per cent phosphoric acid per run. In addition, a reduction in plutonium losses has resulted at all three product precipitation steps amounting to greater than 0.2 per cent per run.

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PRODUCTION TEST 221-T-4

Test Proposal

HW-3-2028

Apple, R. S.

"Increasing the Extraction Charge Size by Increasing Both the Extraction Volume and the Extraction Concentration" (4-4-45)

Objective: To increase the quantity of material processed in the extraction step by 52 per cent. This increase is necessary if two canyons are to process 90 tons (short) of metal pushed each month and carry out operation on the basis of a 24 hour time cycle with a 10 per cent allowance for delays.

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

By means of increased strike and digestion temperature and time, and increased phosphoric acid concentration, it was possible to increase the extraction charge size 50 per cent by increasing the extraction volume 25 per cent and the UNH concentration 20 per cent without materially increasing the waste loss.

Reference

HW-3-2002, L. Squires, "Program for Increased Output of the Separation Plants", (4-3-45).

PRODUCTION TEST 221-B-5

Test Proposal

HW-7-6236

Reber, E. J.

"By-Product Cake Washing" (5-6-47)

Objective: To reduce plutonium losses in the first by-product precipitation without overloading the centrifuge.

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Production Test 221-B-5 (contd.)

Final Report

HW-18591

Szulinski, M. J. and B. E. Kirkendall

"By-Product Cake Washing" (8-15-50)

Washing of the first cycle by-product precipitate in the precipitator tank resulted in no significant change in waste losses but did result in an increase in the decontamination factor.

PRODUCTION TEST 221-T-5

Test Proposal

HW-3-2484

Acken, M. F.

"Plant Tests to Improve Decontamination and Decrease Solids Carry-Thru in First By-Product Step" (5-21-45)

Objective: To reduce the frequency of runs occurring with sub-normal decontamination through the Canyon Building, to reduce the amount of "fuzz" (cerium and zirconium phosphates) carried into the first and second cycle product precipitates and to increase the over-all decontamination obtained in the Canyon Building.

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

The centrifugation rate appeared to have no conclusive effect upon the subsequent decontamination factors. In view of these results and the adequacy of the decontamination obtained normally, the completion of this test was postponed indefinitely.

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PRODUCTION TEST 221-B-6

Test Proposal

HW-7106

Reber, E. J.

"Scavenger Concentration in First By-Product Precipitation"  
(7-15-47)

Objective: To reduce the plutonium losses in the first cycle by-product precipitation by decreasing the amount of scavengers.

Final Report

HW-18596

Szulinski, M. J. and B. E. Kirkendall

"Scavenger Concentration in First By-Product Precipitation" (8-15-50)

It was concluded that decreasing the amount of cerium and zirconium in the first cycle by-product precipitation did not significantly affect the plutonium loss or decontamination factor in this step.

PRODUCTION TEST 221-T-6

Test Proposal

HW-3-2525

Apple, R. S.

"Potassium Hydroxide Wash of Second Cycle Product Cake" (6-5-45)

Objective: To effect further reduction in the process volumes handled in the Concentration Building by reducing the amount of acid required for dissolving the second cycle product cake. Smaller process volume in the Concentration Building is an aid in reducing waste losses from the lanthanum fluoride product precipitation step.

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Production Test 221-T-6 (contd.)

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

The expected decrease in the lanthanum fluoride product step waste loss was less than had been anticipated and several disadvantages to the use of this procedure in routine operation were uncovered: the total waste losses were increased and the process cycle time was increased by approximately two hours at the point of performing the metathesis. For these reasons, the idea of using a potassium hydroxide metathesis step as a routine procedure was abandoned, but the feasibility of its use in special cases, where the quantity or concentration of the cake dissolving acid would be, through design or inadvertance, below the normal operating level, was established.

PRODUCTION TEST 221-B-7

Test Proposal

HW-10755

Szulinski, M. J.

"Centrifugation of Extraction  $\text{BiPO}_4$  Precipitate at 1720 G(1740 RPM)" (8-17-48)

Objective: To decrease the plutonium loss in the extraction waste by centrifuging at high bowl speeds (1740 RPM).

Final Report

HW-15258

Szulinski, M. J.

"Centrifugation of Extraction  $\text{BiPO}_4$  Precipitate of 1720 G(1740 RPM)" (9-27-49)

It was indicated by the test that extraction waste losses were not significantly lowered by centrifugation at the higher bowl speed and that mechanical overloading of the centrifuge at existing throughput rates, 140 pounds per minute, precluded time cycle reduction by means of increased rates. It was recommended that the standard condition of 140 pounds per minute throughput at the speed of 870 RPM be maintained.

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PRODUCTION TEST 221-T-7

Test Proposal

HW-3-3275

Acken, M. F.

"Coating Removal from Individual Irradiated Slugs" (12-12-45)

Objective: To remove the coating and bonding materials from individual irradiated slugs in such a manner as will permit inspection of the decoated slug.

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

Removal of the coating and bonding materials from irradiated slugs by the normal operating procedure is sufficiently complete to permit accurate observations of the slug surface. There are no mechanical or operational difficulties involved in this procedure which make it hazardous.

PRODUCTION TEST 221-B-8

Test Proposal

HW-18430

Szulinski, M. J.

"Coating Removal Waste Loss Reduction" (7-27-50)

Objective: To lower the amount of plutonium and uranium lost to first cycle waste storage from the coating removal operation by substituting the present standard 5 per cent nitric acid flush with one of water.

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Production Test 221-B-8 (contd.)

Final Report

HW-21442

Kirkendall, B. E.

"Coating Removal Waste Loss Reduction" (6-25-51)

Substitution of washes ranging from 515 gallons of water for the combined 100 gallon water and 500 gallon five per cent nitric acid wash resulted in a saving for each dissolver charge of from 0.54 to 0.45 per cent of the plutonium in an average run and from 1.46 to 1.37 pounds of uranium. The volume of the wash was reduced from 600 to 300 gallons for an additional saving in waste storage of approximately \$100.00 per dissolver charge in waste storage capacity. A saving of approximately \$7.50 per dissolver charge was realized in nitric acid consumption. Metal dissolution proceeded satisfactorily; plutonium losses in extraction and decontamination factors were essentially unchanged in all test runs.

PRODUCTION TEST 221-T-8

Test Proposal

HW-7-2147

Perry, L. C. and R. S. Apple

"Potassium Hydroxide Wash (Metathesis) of First and Second Cycle Product Cakes" (7-30-45)

Objective: To permit dilution of the product cake dissolving acid, in the precipitator, to sufficient volume so that it can be agitated by the agitator, and still obtain solution of the product cake in the centrifuge bowl. The objective in agitating the acid in the precipitator is to dissolve all bismuth phosphate and plutonium which may have accumulated therein.

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

The very small amount of plutonium found in the precipitator washes indicated that no accumulation of product was occurring

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Production Test 221-T-8 (contd.)

Summary Report (contd.)

in these vessels and therefore this procedure was not necessary to insure freedom from such accumulation. This conclusion has been substantiated by subsequent acid washes which also showed no significant accumulation of product in the precipitators.

PRODUCTION TEST 221-B-9

Test Proposal

HW-19609

Szulinski, M. J.

"Elimination of Iodine from Canyon Building Exhaust Air" (12-1-50)

Objective: To decrease the amount of radioiodine liberated to the atmosphere.

Final Report

HW-25150

Kirkendall, B. E.

"Elimination of Radioiodine from Canyon Building Exhaust Ventilation Air" (7-29-52)

The average improvement in iodine evolution resulting from sparging during the dissolution reaction was 6.4 per cent and ranged from 0 to 14 per cent. The degree of improvement in iodine evolution produced was considered to be an inadequate solution to the iodine problem.

References

HW-14349, J. B. Work, "Semi-monthly Report - 200 Area Technical Section", (9-1-49).

HW-20803, C. H. Holm, "Removal of Iodine from Aqueous Solutions by Sparging", (6-51).

HW-11528, M. Garbrecht, "Activity of Fission Products", (11-11-48).

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PRODUCTION TEST 221-T-9

Test Proposal

HW-3-3004

Acken, M. F.

"Plant Test to Decrease Process Time in the Decontamination Cycles by Eliminating the Reduction Period Prior to Product Precipitation" (8-8-45)

Objective: To reduce the processing time in the product half of the canyon decontamination cycles (Sections 14 and 17) by eliminating the reduction time prior to the bismuth phosphate product precipitation.

Summary Report

HW-7-4204

Apple, R. S.

"Summary of Canyon Building Production Tests Start-up through December 31, 1945" (Memorandum Report SE-PA-2) (6-6-46)

No adverse effects resulted from this change and it was adopted as standard procedure.

PRODUCTION TEST 221-B-10

Test Proposal

HW-19528

Kirkendall, B. E.

"Process Volume Reduction" (11-20-50)

Objective: To lower production cost by decreasing the size of the extraction precipitation cake and employing the inherent advantages of reduced chemical cost, reduced waste storage requirements, and slight time cycle reduction in the Canyon and Concentration Buildings.

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Production Test 221-B-10 (contd.)

Final Report

HW-24878

Kirkendall, B. E.

"Process Volume Reduction" (6-30-52)

The bismuth concentration in extraction was successfully reduced to 2.5 grams per liter compared to the previous standard of a bismuth to plutonium weight ratio of 95 to 1 (approximately 4.5 grams per liter at 400 megawatt days per ton). Based on the September 1, 1946 process volumes, volumes in the first decontamination cycle were reduced to 42 per cent; and second decontamination cycle and cross-over cycle were reduced to 49 per cent compared to the previous standard of 70 per cent volume for all three cycles. The level of the volumes adopted as the new standard was limited by the inability to obtain complete dissolution of the product cakes, particularly in the product section of the first decontamination cycle.

Reference

HW-21948, B. E. Kirkendall, "Dollar Savings Under Production Test 221-B-10", (8-10-51).

PRODUCTION TEST 221-T-10

Test Proposal

HW-3-3293

Acken, M. F.

"Extraction Conditions to Be Used with 'Class C' Metal (Approximately 400 gt)" (12-14-45)

Objective: To employ extraction conditions for the "Class C" metal which will permit (1) determination of the optimum conditions for processing metal of high plutonium concentration, and (2) savings in metal waste storage volume.

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Production Test 221-T-10 (contd.)

Final Report

HW-7-4604

Apple, R. S.

"Extraction Conditions to be Used with 'Class C' Metal (Approximately 400 g/t)" (8-29-46)

A significant increase in extraction waste loss as well as a slight decrease in the first cycle by-product loss and a slight increase in the first cycle product waste loss has resulted whenever "Class C" metal has been processed. The increased extraction loss was not affected by such process variables as bismuth concentration, bismuth to plutonium ratio, acidity of the extraction solution, UNH concentration, agitation, phosphoric acid concentration or type of strike. In addition the high loss values were shown not to result from iron interference, state of plutonium oxidation, or the presence of uncentrifuged solids. These results suggested the presence of a "foreign" alpha activity which incompletely followed plutonium.

PRODUCTION TEST 221-T-11

Test Proposal

HW-7-5214

Work, J. B.

"Plant Evaluation of Bismuth Subnitrate Solution Prepared from Metallic Bismuth" (9-20-46)

Objective: To establish conditions for dissolving bismuth metal in nitric acid, obtaining a solution that is  $24 \pm 0.5$  per cent bismuth subnitrate ( $\text{BiONO}_3$ ) and  $19 \pm 2$  per cent nitric acid, and to compare plant performance data obtained using this solution with those obtained using bismuth subnitrate solution prepared routinely by dissolving the solid salt.

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Production Test 221-T-11 (contd.)

Report

HW-7-5857

Work, J. B.

"Plant Evaluation of Bismuth Subnitrate Solution Prepared from Metallic Bismuth" (2-25-47)

The yield of synthesized bismuth subnitrate was quantitative in regard to the bismuth metal used. Disposal of the nitrogen dioxide off-gases through a roof vent was satisfactory for the tests, but would present a problem for routine operation since certain wind conditions would sweep the fumes into the ventilation system. Control of the reaction was not difficult with the large pieces of bismuth used. Examination of the waste loss data indicated the test bismuth solution to be (a) equivalent to or better than routinely prepared solution with respect to plutonium losses in the Canyon decontamination steps in which the plutonium was oxidized and (b) equivalent to routinely prepared solution in regard to losses in the Canyon decontamination steps in which plutonium was reduced. The decontamination data revealed no significant differences in the average logarithm decontamination values for the series of runs before, during, and after the use of synthesized material.

PRODUCTION TEST 221-T-12

Test Proposal

HW-7-6159

Reber, E. J.

"Extraction Waste Losses" (4-28-47)

Objective: To reduce the extraction waste losses.

Final Report

HW-18597

Szulinski, M. J. and B. E. Kirkendall

"Extraction Waste Losses" (8-15-50)

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It was concluded that washing the centrifuge between runs, a second reduction in the extraction precipitator tank, a slower

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Production Test 221-T-12 (contd.)

Final Report (contd.)

phosphoric acid addition rate for the second half of the acid, and a digestion period following the phosphoric acid addition all contributed to a lower extraction waste loss. It was also determined that extraction waste losses were not significantly affected by recycling the effluent if the centrifuge was water washed.

PRODUCTION TEST 221-T-13

Test Proposal

HW-9297

Szulinski, M. J.

"Process Volume Reduction" (3-31-48)

Objective: To shorten the time required to process a run in both the Canyon and Concentration Buildings through a reduction in process volumes and also effect reductions in the first and second decontamination cycles waste storage space requirements, and process essential material consumption.

Final Report

HW-18654

Szulinski, M. J. and B. E. Kirkendall

"Process Volume Reduction" (8-10-50)

Testing indicated that 70 per cent of standard was the optimum volume for routine operation.

PRODUCTION TEST 221-T-14

Test Proposal

HW-11367

Szulinski, M. J.

"Use of Ammonium Silico Fluoride Containing Insoluble Material in Excess of That Allowable Under HW Specifications" (10-27-48)

Objective: To determine if a lot of ammonium silico fluoride containing insoluble material in excess of HW specifications can be used in routine process operations.

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HW-26352 D&L

Production Test 221-T-14 (contd.)

Final Report

HW-14996

Szulinski, M. J.

"Use of Ammonium Silico Fluoride Containing Insoluble Material in Excess of that Allowable Under HW Specifications" (9-27-49)

It was concluded that the use of the off-standard ammonium silico fluoride presented no operating difficulties when clarified by decantation after settling for a period of fifteen hours. It was recommended, therefore, that the material be accepted under a waiver of specifications.

PRODUCTION TEST 221-T-15

Test Proposal

HW-21426

Kirkendall, B. E.

"Control of Radioiodine in Canyon Building Exhaust Ventilation Air" (6-13-51)

Objective: To reduce the radioiodine contamination of Canyon Building exhaust ventilation air by complexing with mercury, the iodine in metal solution.

Final Report

HW-25151

Kirkendall, B. E.

"Control of Radioiodine in Canyon Building Exhaust Ventilation Air" (7-24-52)

Radioiodine was successfully suppressed in metal solution by addition of mercuric nitrate to a concentration of  $5 \times 10^{-4}$  M in the dissolver after dilution water addition (when UNH is 1.55 M). The mercuric nitrate was added with the dilution water following dissolving. Concentrations of  $10^{-3}$  M,  $5 \times 10^{-4}$  M, and  $10^{-4}$  M were tested, the degree of suppression varying with concentration. Compared to evolution into ventilation air when no mercury was present,  $10^{-3}$  M mercury resulted in 90 per cent

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HW-26352 DEL

Production Test 221-T-15 (contd.)

Final Report (contd.)

suppression,  $5 \times 10^{-4}$  M in 80 per cent suppression, and  $10^{-4}$  M in no suppression. The concentration of  $5 \times 10^{-4}$  M mercury resulted in a degree of suppression considered to be adequate and is recommended for standard operating procedure. The presence of mercury in the concentrations added showed no effect on waste losses or decontamination in the Canyon or Concentration Buildings.

References

HW-25150, B. E. Kirkendall, "Final Report - Production Test 221-B-9 Elimination of Iodine from Canyon Building Exhaust Ventilation Air", (7-29-52).

HW-19572, O. F. Hill, Trip Report - "Solvent Extraction Studies at ORNL and KAPL", (11-27-50).

HW-21103, C. H. Holm, "Retention of Iodine in Process Solutions by Mercuric Salts", (6-15-51).

PRODUCTION TEST 221-T-16

Test Proposal

HW-24716

Szulinski, M. J. by B. E. Kirkendall

"Four Hour Time Cycle in the Canyon and Concentration Buildings"  
(6-10-52)

Objective: To determine by test the changes needed in the Bismuth Phosphate Process (Buildings 221 and 224) to permit a four hour time cycle and to determine the effect of these changes on the product waste losses and decontamination.

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HW-26352 DEL

PRODUCTION TEST 224-B-1

Test Proposal

HW-3-2507

Beaton, R. H.

"Lanthanum Increase in  $\text{LaF}_3$  Product Precipitation" (5-29-45)

Objective: To lower lanthanum fluoride product waste losses (E-3-WS) to assure a charge-per-day operation with a two centrifugation process. To obtain this objective it is proposed to increase the lanthanum carrier concentration 50 per cent per strike.

Summary Report

HW-3-3408

Beaton, R. H.

"Summary of Concentration (224) Building Production Tests"  
(Memorandum Report SE-PA-5) (1-10-46)

The results and conclusions are summarized. The use of 2.0 pounds of lanthanum in each of the two strikes (125 milligrams per liter lanthanum) in the Cell E lanthanum fluoride product precipitation became standard practice with Runs B-5-07-B-6 and T-5-07-D-6 and is being used as such at present. Continued use has supported the operational data obtained during the test series.

PRODUCTION TEST 224-T-1

Test Proposal

HW-3-1546

Beaton, R. H.

"Recycling of Combined Isolation Supernatants" (2-3-45)

Objective: To substitute a single recycling of six combined Building 231 isolation supernatants for the present separate charge recycling procedure.

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HW-26352 DEL

Production Test 224-T-1 (contd.)

Summary Report

HW-3-3408

Beaton, R. H.

"Summary of Concentration(224) Building Production Tests" (Memorandum Report SE-PA-5) (1-10-46)

The results indicate that 231 Building isolation supernatants can be combined and processed as a separate charge in the Concentration (224) Building. This master recycle procedure has been adopted as one method of reworking recycle from the Isolation Building.

PRODUCTION TEST 224-B-2

Test Proposal

HW-3-3097

Beaton, R. H.

"Acid Slurry Removal of Lanthanum Fluoride Product Cake from Centrifuge" (9-12-45)

Objective: To improve the completeness of removal of the lanthanum fluoride product cake from the 40-inch Bird Centrifuge, B-E.

Summary Report

HW-3-3408

Beaton, R. H.

"Summary of Concentration (224) Building Production Tests " (Memorandum Report SE-PA-5) (1-10-46)

The use of a nitric acid slurry intermediate between successive uses of the bowl spray for the removal of the lanthanum fluoride product cake from centrifuge E-2 did not succeed in lowering the Beckman readings appreciably, either following the nitric acid slurring itself or the successive water spraying. During the test series of five runs, it was observed that the cake hold-up became worse and culminated in an estimated accumulation hold-up of approximately 25 per cent of a normal product charge.

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HW-26352 DEL

Production Test 224-B-2 (contd. )

Summary Report (contd. )

The use of nitric acid slurring technique was abandoned as unsatisfactory. Shortly thereafter, use of the hot potassium hydroxide slurring method was initiated and this method has been used successfully at both T and B-Areas since that time.

PRODUCTION TEST 224-T-2

Test Proposal

HW-7-1238

Acken, M. F.

"On Use of Potassium Hydroxide Metathesis" (1-31-45)

Objective: To replace present potassium hydroxide-potassium carbonate type of metathesis with the simpler straight potassium hydroxide metathesis.

Summary Report

HW-3-3408

Beaton, R. H.

"Summary of Concentration (224) Building Production Tests"  
(Memorandum Report SE-PA-5) (1-10-46)

Product waste losses for the test runs averaged 0.5 per cent for F-7-WS and 0.3 per cent for F-9-WS, as compared 0.90 per cent and 0.29 per cent respectively for the former potassium hydroxide-potassium carbonate method. Time cycles for the test runs averaged 21 hours, 20 minutes as compared to 26 hours, 18 minutes for the potassium hydroxide-potassium carbonate method. The straight potassium hydroxide method of metathesis was adopted as standard practice at this time and has been carried out on all charges since then. Further development work has been carried out on its process simplification, resulting in an over-all time cycle of approximately 15 hours.

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HW-26352 DEL

PRODUCTION TEST 224-B-3

Test Proposal

HW-3-3321

Gilbert, E. R.

"Evaluation of Crystalline  $\text{LaF}_3$  in Dummy Runs" (12-31-45)

Objective: To make dummy runs in Cell B equipment evaluating an improved crystalline lanthanum fluoride carrier. It is expected that this information will provide the basis for a subsequent production test evaluating the use of crystalline lanthanum fluoride in production runs as a tool for reducing plutonium waste losses in the product lanthanum fluoride precipitation (E-3-W).

PRODUCTION TEST 224-T-3

Test Proposal

HW-7-1394

Kay, W. C.

"On Lanthanum Fluoride Precipitations" (3-8-45)

Objective: To decrease lanthanum fluoride product precipitation losses (E-3-WS wastes).

Summary Report

HW-3-3408

Beaton, R. H.

"Summary of Concentration (224) Building Production Tests"  
(Memorandum Report SE-PA-5) (1-10-46)

The results and conclusions of this study are given. The results were applied to the activation of further test work (P. T. 224-T-4) and no direct application to plant process was made.

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HW-26352 D&L

PRODUCTION TEST 224-B-4

Test Proposal

HW-7-4629

Apple, R. S.

"Plant Evaluation of Alternate Source (Penn. Salt) Anhydrous HF"  
(8-21-46)

Objective: To compare plant performance data obtained using present source (duPont) double distilled anhydrous hydrogen fluoride with those obtained using similar material supplied by Pennsylvania Salt Co., with the ultimate objective of acquiring a new source of hydrogen fluoride.

Report

HW-7330

Work, J. B.

"Plant Evaluation of Alternate Source (Penn. Salt) Anhydrous HF"  
(8-15-47)

Gives comparison of double-distilled hydrogen fluoride from Pennsylvania Salt Company with that from duPont and a comparison of double and single distilled hydrogen fluoride. The double-distilled hydrogen fluoride supplied by the Pennsylvania Salt Company was adjudged satisfactory by the tests.

PRODUCTION TEST 224-B-4, SUPPLEMENT A

Test Proposal

HW-7768

Szulinski, M. J.

"Plant Evaluation of Alternate Source (Harshaw) Anhydrous HF"  
(10-10-47)

Objective: To compare plant performance data using double distilled anhydrous hydrogen fluoride proven satisfactory under P. T. 224-B-4 (Penn. Salt) with that obtained using similar material supplied by the Harshaw Chemical Company, with the ultimate objective of acquiring an alternate source of hydrogen fluoride.

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HW-26352 OSL

Production Test 224-B-4, Supplement A (contd.)

Final Report

HW-9132

Browne, W. G.

"Plant Evaluation of Alternate Source (Harshaw) Anhydrous HF"  
(2-23-48)

The double-distilled hydrogen fluoride supplied by the Harshaw Chemical Company was adjudged satisfactory by the tests. The data taken for the test runs are given in tabular form.

PRODUCTION TEST 224-T-4

Test Proposal

HW-3-2030

Beaton, R. H.

"Lanthanum Fluoride Precipitation" (4-5-45)

Objective: To shorten lanthanum fluoride by-product and product operations in order to attain practical time cycles of 24 hours or less in Cells D and E.

Summary Report

HW-3-3408

Beaton, R. H.

"Summary of Concentration (224) Building Production Tests"  
(Memorandum Report SE-PA-5) (1-10-46)

The results and conclusions of this study are given. A single-strike flocculent lanthanum fluoride by-product and a 2-strike, 2-centrifugation flocculent lanthanum fluoride product precipitation process was adopted as standard practice for a short time. Further experimental work on increasing the lanthanum concentration in the product strike (to 125 milligrams per liter) through a volume reduction to 40 per cent start-up by potassium hydroxide metathesis of the bismuth phosphate product cakes in the Canyon Buildings (see P. T. 221-T-6) was attempted later. The results were promising in the Concentration Building but additional waste losses encountered in the Canyon Building in

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HW-26352 DEL

Production Test 224-T-4 (contd.)

Summary Report (contd.)

using this process made the net gain nearly zero so it was not adopted. Further, a 50 per cent increase in the absolute weight of flocculent lanthanum used in each of the 2-strikes in the 60 per cent volume process (P. T. 224-B-1) reduces E-3-WS waste losses even more and has now become standard practice.

Reference

HW-3-2002, L. Squires, "Program for Increased Output of the Separation Plants", (4-3-45).

PRODUCTION TEST 224-B-5

Test Proposal

HW-17512

Szulinski, M. J.

"Lanthanum Fluoride By-Product Precipitation Time Cycle Reduction"  
(4-13-50)

Objective: To reduce the lanthanum fluoride by-product precipitation time cycle through process changes.

PRODUCTION TEST 224-T-5

Test Proposal

HW-3-2032

Beaton, R. H.

"Reduction of Concentration Building Time Cycle Through Non-Process Changes" (4-5-45)

Objective: To shorten and simplify the cross-over process in the Concentration Building (224) by moderate "streamlining" of procedures, restricted to non-process changes.

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HW-26352 DEL

Production Test 224-T-5 (contd.)

Summary Report

HW-3-3408

Beaton, R. H.

"Summary of Concentration (224) Building Production Tests"  
(Memorandum Report SE-PA-5) (1-10-46)

The conclusions and disposition of this production test are covered under the following headings: (1) cooling of C-4-0 solution; (2) hot bismuth phosphate dilution strike; (3) order of by-product cake removal; (4) hot D-1-0 sampling; (5) displacement cake washes; (6) 35 C centrifugation in fluoride cells; (7) limited sparging of E-3 before sampling; (8) higher metathesis centrifugation rates; (9) high-pressure spray metathesis cake removal; (10) F-9-WS sampling.

Reference

HW-3-2002, L. Squires, "Program for Increased Output of the Separation Plants", (4-3-45).

PRODUCTION TEST 224-B-6

Test Proposal

HW-22302

Bailor, W. C.

"Decontamination Improvement by Means of Barium Sulfate Precipitations" (10-1-51)

Objective: To determine if decontamination in the Concentration Building can be improved by means of solid barium sulfate additions during the bismuth phosphate by-product precipitation.

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HW-26352 D 26

PRODUCTION TEST 224-T-6

Test Proposal

HW-3-2532

Beaton, R. H.

"Recycling of Isolation Supernatants to Production Runs" (6-1-45)

Objective: To recycle Isolation supernatants to the Concentration Process without delaying production by blending recycles from two charges with each production run in Cell D and T-Area.

Summary Report

HW-3-3408

Beaton, R. H.

"Summary of Concentration (224) Building Production Tests"  
(Memorandum Report SE-PA-5) (1-10-46)

No impairment of the lanthanum fluoride by-product decontamination factor or significant increase in the D-4-BP waste loss was observed. The method of handling isolation supernatants outlined in this production test was adopted as the standard procedure. Although recycling of isolation supernatants to production runs is still standard practice, certain changes have been made in the procedure.

PRODUCTION TEST 224-T-7

Test Proposal

HW-3-3135

Gilbert, E. R.

"Potassium Hydroxide Removal of Lanthanum Fluoride Product Cake from the Centrifuge" (9-24-45)

Objective: To improve the completeness of removal of the lanthanum fluoride product cake from the 40 inch Bird centrifuge (E-2).

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HW-26352 DEL

Production Test 224-T-7 (contd.)

Summary Report

HW-3-3408

Beaton, R. H.

"Summary of Concentration (224) Building Production Tests"  
(Memorandum Report SE-PA-5) (1-10-46)

The use of a 50 per cent potassium hydroxide slurry treatment has resulted in complete cake removal from the E-2 centrifuge. It has not been necessary to use an acid flush as an aid in dissolving residual metathesized cake. The procedure as originally outlined has been adopted as the standard E-2 cake removal procedure.

PRODUCTION TEST 224-T-8

Test Proposal

HW-3-3344

Gilbert, E. R.

"Elimination of Digestion Time in  $\text{LaF}_3$  Product Precipitations"  
(1-14-46)

Objective: To eliminate the digestion time after the lanthanum is added for the lanthanum fluoride product precipitations.

Final Report

HW-3-3607

Beaton, R. H.

"Elimination of Digestion Time in  $\text{LaF}_3$  Product Precipitations"  
(7-29-46)

Conclusions: (1) Elimination of the second strike digestion had no effect on the final waste loss. (2) Elimination of both digestions produced a 50 per cent increase in final waste loss. (3) Additional tests with a 30-minute digestion after the second strike produced occasionally higher E-3-WS losses. In view of the above, it was decided not to eliminate digestion and a 30 minute digestion after each strike was adopted as standard practice.

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HW-26352 D&C

PRODUCTION TEST 224-T-9

Test Proposal

HW-7-3831

Lehman, P. H.

"Volume Reduction in Potassium Hydroxide Metathesis" (4-1-46)

Objective: To obtain more efficient separation of lanthanum hydroxide precipitate (metathesis) during centrifugation and thereby reduce metathesis waste losses (F-7-WS). An attendant objective is to decrease potassium hydroxide requirements.

Final Report

HW-7-4631

Beaton, R. H.

"Volume Reduction in Potassium Hydroxide Metathesis" (8-1-46)

The test data are summarized and tabulated. The 30 per cent reduced metathesis volume process has been recommended for adoption as standard practice at both B and T Plants. The 30 per cent reduction in metathesis volume reduces F-7-WS waste losses by approximately 0.2 per cent.

PRODUCTION TEST 224-T-10

Test Proposal

HW-7-5478

Work, J. B.

"Recirculation of Metathesis Wash Waste Solution" (12-2-46)

Objective: To reduce the plutonium loss in the waste solutions in Cell F of the Concentration Buildings.

Reference

HW-25034, J. B. Work, "Re: Production Test 224-T-10", (2-10-47).

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HW-26352 DEL

PRODUCTION TEST 224-T-11

Test Proposal

HW-7767

Szulinski, M. J.

"Bismuth Phosphate By-Product Cake (A-4BP) Removal with Water or Water-Nitric Acid Combination" (10-10-47)

Objective: To reduce consumption of nitric acid and sodium hydroxide used to dissolve and neutralize the bismuth phosphate product cake in A Cell and thereby effect a reduction in process costs.

Final Report

HW-15001

Browne, W. G.

"Bismuth Phosphate By-Product Cake (A-4BP) - Removal with Water or Water-Nitric Acid Combination" (9-27-49)

Water was used successfully to remove the bismuth phosphate by-product cake from the A-2 centrifuge; the resulting slurry could not be satisfactorily sampled and analyzed, so the use of water for cake removal is not recommended unless sampling and analytical difficulties are overcome.

PRODUCTION TEST 224-T-12

Test Proposal

HW-11940

Work, J. B.

"Plant Evaluation of Harshaw Specification No. 102 Single Distilled Anhydrous HF" (12-23-48)

Objective: To compare plant performance data using Harshaw double-distilled anhydrous hydrogen fluoride proven satisfactory under Production Test 224-B-4, Supplement A, with that obtained by using Commercial grade, Specification No. 102, single-distilled Harshaw anhydrous hydrogen fluoride, as suggested by the "S" Division.

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HW-26352 D/L

Production Test 224-T-12 (contd.)

Final Report

HW-18563

Kirkendall, B. E.

"Plant Evaluation of Harshaw Specification 102 - Single Distilled Anhydrous HF" (8-10-50)

The Harshaw Chemical Company single distilled hydrogen fluoride, Specification No. 102, was adjudged satisfactory for process use. As a supplement to the production test an improved grade of Penn Salt Company single distilled hydrogen fluoride was tested. This was adjudged satisfactory for process use.

Reference

HW-7330, W. G. Browne, "Report on Production Test SE-224-B -PA-4, Plant Evaluation of Alternate Source (Harshaw) Anhydrous HF", (2-23-48).

PRODUCTION TEST 224-T-13

Test Proposal

HW-14495

Browne, W. G.

"Metathesis (F Cell) Time Cycle Reduction" (10-7-49)

Objective: To obtain a time saving of one to two hours in the metathesis cell by decreasing the metathesis centrifugation volume.

PRODUCTION TEST 224-T-14

Test Proposal

HW-18080

Szulinski, M. J.

"Lanthanum Fluoride Product Precipitation Time Cycle Reduction" (6-14-50)

Objective: To reduce the lanthanum fluoride product precipitation time cycle to nine hours with a minimum increase in plutonium losses.

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HW-26352 D&L

PRODUCTION TEST 231-1

Test Proposal

HW-3-1325

Squires, L.

"Improvement of Isolation Process" (12-28-44)

Objective: To simplify operational procedures by (a) elimination of the necessity for certain operations, (b) decrease in the time required, and (c) provision for less hazardous operating conditions.

Memorandum Report

HW-3-1912

Watt, G. W.

"Memorandum Report on P. T. 231-1-SE" (SE-PC -10) (3-21-45)

P. T. 231-1 has been deferred except for two rather limited applications. The decision to defer these tests was based upon laboratory demonstration of a rather marked and unanticipated difference in the behavior of actual F-10-P solutions in comparison with that of synthetic solutions.

PRODUCTION TEST 231-2

Test Proposal

HW-3-1575

Squires, L.

"Improvement in Isolation Process" (2-8-45)

Objectives: (1) To improve the purity of plutonium; (2) to decrease cycle time; and (3) to reduce the volume of solution recycled.

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HW-26352 DEL

Production Test 231-2 (contd.)

Memorandum Report

HW-3-1911

Watt, G. W.

"Memorandum Report on P. T. 231-2-SE" (SE-PC -11 ) (3-22-45)

Using the procedures outlined in detail in HW-3-1575, uniformly satisfactory results were obtained in the plant. The process modifications which have been incorporated in the operating logs are given.

PRODUCTION TEST 231-3

Test Proposal

HW-3-1901

Watt, G. W.

"Improvement of Isolation Process" (3-19-45)

Objectives: To decrease the time required for operation of the first cycle and to decrease the quantity of plutonium recycled as a result of plutonium removed from P-1 mechanically following washing operations.

Memorandum Report

HW-3-2703

Watt, G. W.

"Memorandum Report on Production Tests 231-3-SE, 231-4-SE, and 231-5-SE" (SE-PC - 67)

Changes of 0.25 M ammonium sulfate for 0.20 M sulfuric acid in first cycle and of reduction settling time from nine to six hours were made. The use of ammonium sulfate resulted in improvement in the physical characteristics of the first cycle precipitate to the extent that six hour settling period was actually longer than necessary. Substitution of 0.2N sulfuric acid for 1 N nitric acid as the first cycle wash solution improved rates of settling after each washing operation.

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HW-26352 DEL

PRODUCTION TEST 231-4

Test Proposal

HW-3-2057

Watt, G. W.

"Improvement of Isolation Process" (4-6-45)

Objective: The primary objective of the tests outlined is to eliminate a potentially hazardous pre-reduction step and to place the nitrite method (for the destruction of hydrogen peroxide) on a more routinely reproducible basis. It is also desired to decrease the processing time in the first cycle.

Memorandum Report

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HW-3-2703

Watt, G. W.

"Memorandum Report on Production Tests 231-3-SE, 231-4-SE and 231-5-SE" (SE-PC-67)

The use of 0.5M ammonium sulfite at 25 C in place of 1 per cent hydrogen peroxide by weight was initiated and the measured plutonium (IV) peroxide "solubilities" indicated complete reduction of plutonium (VI). This change eliminated the hazard of heating hydrogen peroxide and made possible a reduction in cycle time amounting to one to two hours. Circa one and one half hours per plant run has been saved by scheduling hydrogen peroxide pre-reductions prior to the first cycle acidity adjustment. Potentiometric determination of the end-point in the reaction between hydrogen peroxide and sodium nitrite is used satisfactorily (HW-3-2533). Improved rates of filtration through N-2 were had by (a) elimination of the N-2 washes; (b) increase in the N-2 pre-coat from 50 to 200 grams of filter aid. The reducing of the first cycle settling time from six to four hours did not lead to any increase in the percentage of plutonium recycled. Formation of first cycle precipitates from P-1 solutions 2.0N in nitric acid and 0.25M in ammonium sulfate rather than 1.0N nitric acid-0.25M ammonium sulfate or 1.0N nitric acid -0.2N sulfuric acid is considered desirable. Second cycle initial acidity range was broadened from  $2.0 \pm 0.1N$  to 1.8-2.2N nitric acid and led to no disagreeable differences in process performance.

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HW-26352 *ASC*

PRODUCTION TEST 231-5

Test Proposal

HW-3-2510

Watt, G. W.

"Decrease in the Ammonium Ion Content of the Final Product" (5-10-45)

Objective: In connection with the general subject of final plutonium purity, it is desired to institute a process modification which will essentially eliminate ammonium ion from the plutonium nitrate solutions which constitute the starting materials for the final evaporation step.

Memorandum Report

HW-3-2703

Watt, G. W.

"Memorandum Report on Production Tests 231-3-SE, 231-4-SE and 231-5-SE" (SE-PC-67)

Making the second cycle precipitation from 1.5N nitric acid -0.5N sulfuric acid rather than 2.0N nitric acid -0.25N ammonium sulfate resulted in an ammonium ion content of AT (plutonium nitrate) solution too low to permit quantitative determination by existing analytical methods.

PRODUCTION TEST 231-6

Test Proposal

HW-7-2146

Lindner, M. and J. B. Work, J. J. Urban

"Improvement of Isolation Process" (7-27-45)

Objective: To reduce the volume of solution recycled to the Concentration Building (224) from about 150 liters to 40 liters or less, by applying an "oxalate" method of treating the recycle.

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HW-26352 *DSL*

Production Test 231-6 (contd.)

Memorandum Report

HW-3-3271

Work, J. B.

"Memorandum Report on P. T. SE-231-PA-6" (SE-PA - 4) (11-30-45)

The information developed during the work is compiled and correlated. The advantages and disadvantages which are now apparent in the oxalate method of handling recycled material are summarized.

PRODUCTION TEST 231-7

Test Proposal

HW-7-5713

Haught, D. W.

"Pre-Reduction Conditions" (1-24-47)

Objective: To reduce the weight of plutonium recycled from Isolation to the Concentration Buildings.

PRODUCTION TEST 231-8

Test Proposal

HW-11446

Work, J. B.

"Isolation Process Minimum Time Requirements" (10-29-48)

Objective: To determine the minimum time required for the successful operation of each purification cycle in the Isolation Process.

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HW-26352 DEL

Production Test 231-8 (contd.)

Final Report

HW-15002

Browne, W. G.

"Isolation Process Minimum Time Requirements" (9-27-49)

A time saving of 120 minutes in both the first and second purification cycles of the Isolation Building process was effected by decreasing the peroxide addition time from 60 to 30 minutes, by changing the settling time of the plutonium precipitate prior to decanting from 120 to 60 minutes, and by decreasing the digestion period following product precipitation from 60 to 30 minutes.

PRODUCTION TEST 231-9

Test Proposal

HW-13028

Szulinski, M. J.

"Use of 50 Per Cent Hydrogen Peroxide in the Isolation Process"  
(4-4-49)

Objective: To decrease the amount of plutonium recycled from the Isolation Building to the Concentration Building and to reduce the cost of essential materials by replacing the 27.5 per cent hydrogen peroxide, currently in use, with 50 per cent peroxide.

Final Report

HW-24770

Packer, G. V.

"Use of 50 Per Cent Hydrogen Peroxide in the Isolation Process"

Analyses of the data disclose that substitution of 50 per cent hydrogen peroxide for 27.5 per cent hydrogen peroxide results in a minor decrease on yield of approximately 1.4 per cent and leaves isolated plutonium purity essentially unaffected. Data indicate that the yield will be essentially unaffected by peroxide reagent concentration providing the acidity is adjusted to the same value for the final strike volume. Purity of the isolated plutonium nitrate (AT solution) was 98.6 per cent for 50 per cent

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HW-26352 D&L

Production Test 231-9 (contd.)

Final Report (contd.)

reagent and 98.4 per cent with 27.5 per cent reagent. Gives the advantages obtained by using 50 per cent hydrogen peroxide and recommends that it be used routinely in the Isolation Process.

PRODUCTION TEST 231-10

Test Proposal

HW-15907

Kerr, W. B.

"Determination of Optimum Sample Can Load Weight to be Delivered to the 234 Building" (2-7-50)

Objective: To determine the optimum weight of material to be left in sample cans. Off-site shipments may require more complete evaporation than material for the 234-5 Building; evaporation in the latter case may have a minimum limit, however, in order to avoid interference with processing through purification and dry chemistry.

Interim Report

HW-18617

Kerr, W. B.

"Optimum Sample Can Load Factor" (8-18-50)

A study of the inspection notes on the runs processed in this test, indicated that the material on normal loads does not become hard within two weeks. Those cans near the upper load factor limit of 231 Building normal operation contained some liquid, and as the load factor increased, the material became more liquid. At no point did a viscous liquid develop. Recommends that the load factor for those sample cans not scheduled for off-site shipment should be increased to  $3.03 \pm 0.15$  grams per gram of product.

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HW-26352 086

PRODUCTION TEST 231-10, SUPPLEMENT A

Test Proposal

HW-18659

Kerr, W. B.

"Increase in Sample Can Load Factor" (8-22-50)

Objective: To establish a new load factor for the dried sample can material scheduled for on-site shipment only.

Interim Report

HW-18877

Kerr, W. B.

"Optimum Sample Can Load Factor" (9-20-50)

In forty-eight batches, the average supernate plutonium concentration was 0.0182 grams per liter with approximately 14.5 per cent of those batches outside the allowable limits of 0.01 to 0.03 grams per liter. On forty batches under this production test, the average supernate plutonium concentration was 0.0213 grams per liter with 15 per cent of these outside the allowable limits. The slightly higher figures are not considered significant. It is recommended that the load factor of  $3.03 \pm 0.15$  grams per liter be established as the standard for all sample cans scheduled for on-site shipment.

PRODUCTION TEST 231-10, SUPPLEMENT B

Test Proposal

HW-19254

Kerr, W. B.

"Purification of Undried AT Solution" (10-27-50)

Objective: To test in plant equipment a process for the purification of AT (plutonium nitrate) solution by the present oxalate precipitation process without first removing the excess nitric acid.

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HW-26352 026

Production Test 231-10, Supplement B (contd.)

Final Report

HW-22333

Kerr, W. B.

"Process Evaluation - Production of Concentrated Plutonium Nitrate Solution in Building 231-Transfer to and Processing in Building 234-5" (10-3-51)

It was demonstrated that satisfactory plutonium III oxalate precipitate can be obtained from 231 Building AT solutions containing  $350 \pm 50$  grams per liter plutonium in 8.8 to 10.7 M nitric acid solutions. Processes established by this test permit the elimination of the final concentration in the 231 Building and subsequent dissolution in the 234 Building for all internal processing at Hanford Works. This results in the saving of approximately 37.5 man hours operating time per kilogram of plutonium processed.

References

HW-15907, W. B. Kerr, (P. T. 231-10) "Determination of Optimum Sample Can Load Weight to be Delivered to the 234 Building", (2-7-50).

HW-18659, W. B. Kerr, (P. T. 231-10, Suppl. A) "Increase in Sample Can Load Factor", (8-22-50).

HW-18617, W. B. Kerr, (P. T. 231-10 Interim Report) "Optimum Sample Can Load Factor", (8-18-50).

HW-18877, W. B. Kerr, (P. T. 231-10, Suppl. A Interim Report) "Optimum Sample Can Load Factor", (9-20-50).

HW-18738, W. L. Lyon and J. F. Facer, (Summary Report) "Use of AT Solution with Evaporation, 234-5 Development Group", (9-1-50).

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PRODUCTION TEST 231-11

Test Proposal

HW-17546

Packer, G. V.

"Use of Heat for Destroying Excess Hydrogen Peroxide in Isolation Supernatant Solutions" (4-17-50)

Objective: To substitute heat for sodium nitrite in the destruction of the excess hydrogen peroxide in Isolation supernatant solutions, thereby decreasing the volume of solutions recycled to the Concentration Building from an average of 108 liters per run to approximately 90 liters per run.

PRODUCTION TEST 231-12

Test Proposal

HW-17990

Szulinski, M. J.

"Recovery of Americium" (6-2-50)

Objective: To establish a process for americium recovery, and to use this process upon AEC request, to obtain 50 milligrams of americium in 25 liters or less volume with less than 50 grams of lanthanum.

Reference

HW-16038, O. F. Hill, "Americium Separation from Plant Solutions", (2-15-50).

PRODUCTION TEST 231-13

Test Proposal

HW-22653

Szulinski, M. J. by G. V. Packer

"Time Cycle Reduction: Elimination of Peroxide Precipitation Digestion and Shortened Settling Periods" (P. T. 231-13) (11-8-51)

Objective: To demonstrate the feasibility of eliminating the digestion period following the plutonium peroxide precipitation,

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HW-26352 026

Production Test 231-13 (contd.)

Test Proposal (contd.)

and of shortening the periods designated for settling the precipitate prior to separation of the supernatant solutions from the precipitate by decantation.

Final Report

HW-25178

Schmidt, W. C.

"Elimination of Peroxide Precipitation Digestion and Shortened Settling Periods" (7-30-52)

It is recommended that the test conditions, resulting in a 6.25 hour cycle, be adopted as a standard. Specifically the process changes recommended are: A. Eliminate the digestion period of thirty minutes just after the peroxide addition in both the first and second cycle. B. Reduce the settling time from one hour to thirty minutes in both the first and second cycle. C. Reduce the settling time after each of the three washes in the first cycle by fifteen minutes. D. Reduce the settling time after each of the first two washes by fifteen minutes and after the third wash by thirty minutes in the second cycle.

PRODUCTION TEST 231-14

Test Proposal

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HW-23416

Szulinski, M. J. by G. V. Packer

"Demonstration of 231 Building Filter Boat Station with Plutonium IV Oxalate and Plutonium Peroxide Flowsheets" (1-30-52)

Objectives: The general objective: to evaluate the process equipment provided for transferring a plutonium compound from the Isolation Process Building (231) to the Purification and Fabrication Building (234-5) when operating on either a plutonium IV oxalate flowsheet or a plutonium peroxide flowsheet. The specific objectives to determine: (1) the flow characteristics of the slurries of (a) the IV oxalate or (b) the peroxide precipitates;



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Production Test 231-14 (contd.)

Test Proposal (contd.)

(2) the filter cake densities of the plutonium compounds under process conditions; (3) the 231 Building filter boat station equipment deficiencies, if any; (4) the hydrofluorination process behavior of the compounds; (5) the physical characteristics (particle size, apparent density, adherence to filter boat) of the plutonium IV fluorides from the hydrofluorination operation; (6) the chemical and physical behavior (yield, purity, button formation) of the plutonium IV fluorides in the reduction operation.

References

HW-21026, W. B. Kerr, (P. T. 234-3) "Direct Hydrofluorination of Plutonium Peroxide", (5-23-51).

HW-22340, V. R. Cooper, "Progress Report for August 1951", (10-4-51).

HW-3409-T, J. R. Facer, Jr., Personal Notebook.

HW-23015, V. R. Cooper, "Separations Technology Unit - Process Progress Report for November, 1951", (12-12-51).

HW-23075, M. J. Szulinski/W. C. S., "Handling of Methanol in the 231 Building Filter Boat Process", (12-19-51).

PRODUCTION TEST 234-1

Test Proposal

HW-14215

Roth, O. T.

"Oxalate Supernatant Treatment" (8-19-49)

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Objective: To remove hydriodic and oxalic acids from 234 Building oxalate supernatant solutions for recycling of product to 224 Building.

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Production Test 234-1 (contd.)

Test Proposal Revision

HW-14526

Work, J. B.

"Revision of Production Test 234-1" (9-22-49)

It has been found that iron sulfate precipitates during the extended evaporation of SN-3 solution. Since it may be desirable to avoid the precipitation of this material on dip tubes and the wall of the evaporator in Hoods 29 and 30, it is proposed to stop the evaporations at slightly larger volumes.

PRODUCTION TEST 234-1, SUPPLEMENT A

Test Proposal

HW-21104

Blasewitz, A. G.

"Treatment of Oxalate Supernatant to Permit Product Recycling"  
(5-11-51)

Objective: To provide a plant scale evaluation of the laboratory-demonstrated process for the destruction of hydriodic and oxalic acids.

Final Report

HW-21902

Blasewitz, A. G.

"Treatment of the Oxalate Supernatant to Permit Product Recycling"  
(8-13-51)

The six lots of oxalate supernatants which were treated with hydrogen peroxide have substantiated the laboratory-demonstrated process for the destruction of hydriodic and oxalic acids. The amount of oxalate and total iodine (as iodide and/or iodate) remaining after treatment with a five per cent excess of the stoichiometrical requirement of hydrogen peroxide was 0.11 to 0.80 per cent and 0.10 to 0.11 per cent, respectively.

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Production Test 234-1, Supplement A (contd.)

Reference

HW-21082, R. E. Falkoski and R. A. Pugh, "Treatment of 234 Building Wet Chemistry Waste Solutions for Recycle", (5-23-51).

PRODUCTION TEST 234-1, SUPPLEMENT B

Test Proposal

HW-21901

Blasewitz, A. G.

"Combined Treatment of Concentrated and Current Oxalate Supernatants to Permit Product Recycling" (8-13-51)

Objective: To provide a plant-scale evaluation of the laboratory-demonstrated process employing hydrogen peroxide for the destruction of hydriodic and oxalic acids by the simultaneous processing of stored concentrated oxalate supernatants (SN-3) and current oxalate process supernates (SN-1).

Final Report

HW-23218

Blasewitz, A. G.

"Treatment of the Oxalate Supernatant to Permit Product Recycling" (1-9-52)

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The investigation has established that the concentrated and stored oxalate supernatants (SN-3) can be simultaneously treated with the current supernatants (SN-2) in the manner that will permit recycling of the material to the Concentration Building. There has been no harmful effect upon waste losses or corrosion of the E-4 Tank in the Concentration Building as a result of the recycled supernatant solutions.

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Production Test 234-1, Supplement B (contd.)

References

HW-21082, R. E. Falkoski and R. A. Pugh, "Treatment of 234 Building Wet Chemistry Waste Solutions for Recycle", (5-23-51).

HW-21104, A. G. Blasewitz, (P. T. 234-1, Supplement A) "Treatment of the Oxalate Supernatants to Permit Product Recycling", (5-11-51).

HW-21902, A. G. Blasewitz, (Final Rpt. P. T. 234-1, Supplement A) "Treatment of the Oxalate Supernatant to Permit Recycling", (8-13-51).

HW-21616, R. A. Pugh, "Treatment of 234 Building Concentrated Oxalate Waste Solutions for Recycle", (7-6-51).

PRODUCTION TEST 234-1, SUPPLEMENT C

Test Proposal

HW-24030

Blasewitz, A. G.

"Treatment of Concentrated Oxalate Supernatant to Permit Recycling" (4-7-52)

Objective: To put the present accumulation of stored concentrated plutonium III oxalate supernatants into suitable condition to recycle to the Concentration Building using a modification of a procedure found satisfactory in Supplements A and B of the Production Test 234-1.

Final Report

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PRODUCTION TEST 234-2

Test Proposal

HW-19005

Kerr, W. B.

"Recycle of Skull Solution to the Isolation Process" (10-2-50)

Objective: To evaluate a process for skull recovery. Skulls dissolved in a 16M nitric - 0.04M hydrofluoric acid solutions will be processed in a manner similar to regular production material in the Isolation and 234-5 Buildings.

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Production Test 234-2 (contd.)

Final Report

HW-19991

Kerr, W. B.

"Recycle of Skull Solution to the Isolation Process" (1-20-51)

Data are presented on waste losses, impurities and process yields obtained in the test runs. At all stages of the process, waste losses were normal, yields were satisfactory and impurities in the metal were well within acceptable limits. It is concluded that this process is satisfactory for routine operation in the 231 and 234 Buildings.

Reference

HW-19122, W. L. Lyon and J. F. Facer, "234-5 Development Summary Report - Recovery of Plutonium from Skulls" (10-12-50).

PRODUCTION TEST 234-3

Test Proposal

HW-21026

Kerr, W. B.

"Direct Hydrofluorination of Plutonium Peroxide" (5-23-51)

Objectives: (1) To determine the adaptability, under production plant conditions, of the process for direct hydrofluorination of plutonium peroxide previously demonstrated on a laboratory scale. (2) To incorporate modifications to the process as required from results observed in production plant equipment. (3) To evaluate yields, purity, and time cycle requirements obtained under production plant conditions. (4) To evaluate Inconel boats as a replacement for platinum-rhodium as construction material for boats to be used in the hydrofluorination of plutonium peroxide. In addition to the foregoing specific objectives, this test will serve as a forerunner for direct hydrofluorination of plutonium peroxide processing to be employed in the RM Line.

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Production Test 234-3 (contd.)

Final Report

HW-22802

Kerr, W. B.

"Process Evaluation - Hydrofluorination of Plutonium Peroxide"  
(11-15-51)

Thirteen batches of plutonium tetrafluoride were made by hydrofluorinating 231 Building filter cakes of plutonium peroxide. Characteristics of the 231 Building filter cakes are given. Fluorides containing hard lumps were produced when peroxides, which had been washed with five per cent hydrogen peroxide, were hydrofluorinated. Peroxides washed with methyl alcohol were easily dried and hydrofluorinated to a free flowing tetrafluoride powder. Metal purities for materials processed in platinum boats were satisfactory and comparable to those obtained from standard material. Impurities of nickel and chromium were higher than normal in the buttons made from the tetrafluorides produced in the Inconel boats. Plastic bag techniques were used successfully in removing filters from the 231 Building cells, transporting the filters to the 234 Building and placing them in the hood.

References

HW-18092, K. K. Kennedy and W. L. Lyon, "A Plutonium Peroxide Process for the Purification Building", (6-15-50).

HW-21691, W. L. Lyon, "Laboratory Evaluation of One and Two Cycle Peroxide Processing of F-10-P Solutions on the Basis of Metal Purities Obtained", (7-19-51).

HW-21751, W. L. Lyon and B. Weidenbaum, "The Use of Plutonium Peroxide for the Preparation of Plutonium Tetrafluoride", (7-26-51).

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PRODUCTION TEST 234-4

Test Proposal

HW-19651

Quinn, F. J., Jr.

"Direct Hydrofluorination of Plutonium (III) Oxalate" (12-7-50)

Objective: To evaluate a process as described in Reference 1, for converting the plutonium (III) oxalate directly to the tetra-fluoride without a planned intermediate oxidation step.

Final Report

HW-20916

Quinn, F. J., Jr.

"Production of Plutonium IV Fluoride by Direct Hydrofluorination of Plutonium III Oxalate" (3-30-51)

The thirty-seven runs which were processed in 234 Building equipment utilizing the direct hydrofluorination method have substantiated the performance and feasibility demonstrated by the laboratory work. Plutonium IV fluoride produced by the new method has been converted to metal with quality and purity comparable to that obtained by the present production process. The advantages to be realized are summarized. The possible disadvantage of the process is the lower bulk density of the plutonium IV fluoride powder (circa one half that of present fluorides). It is recommended that the present dry chemistry operation be replaced by the method of direct hydrofluorination of the plutonium III oxalate.

References

HW-18796, J. F. Facer and W. L. Lyon, "234-5 Development Group - Summary Report, Direct Hydrofluorination of Pu(III) Oxalate", (9-11-50).

HW-20330, F. W. Albaugh, "A Revised Method for 200 Area Accountability and Material Balance Control", (2-19-51).

HW-23345, W. J. Ozeroff, "Critical Mass Hazards in Production Test 234-4" (1-24-52).

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PRODUCTION TEST 234-5

Test Proposal

HW-23203

Kerr, W. B. and J. M. Hay

"Plant Process Evaluation - Precipitation of Plutonium IV Oxalate"  
(1-8-52)

Objectives: (1) To evaluate the laboratory - tested procedures for producing plutonium (IV) oxalate precipitates from standard 231 Building effluent solutions in plant equipment. (2) To evaluate procedures for recycling the supernatant solutions from these precipitations directly to the 224 Building. (3) To evaluate procedures for the hydrofluorination of the plutonium (IV) oxalate in RG Line equipment. (4) To determine and incorporate any minor modifications in the procedures outlined below to adapt the process to the existing plant equipment. (5) To evaluate yields, purity and time cycles when acceptable procedures have been established. (6) To evaluate slightly altered procedures to allow plutonium (IV) oxalate precipitations containing 350 g. of plutonium.

References

HW-18092, K. K. Kennedy and W. L. Lyon, "A Plutonium Peroxide Process for the Purification Building", (6-15-50).

HW-21691, W. L. Lyon, "Laboratory Evaluation of One and Two Cycle Peroxide Processing of F-10-P Solutions on the Basis of Metal Purities Obtained", (7-19-51).

Production Test 234-3.

PRODUCTION TEST 234-5, SUPPLEMENT A

Test Proposal

HW-24898

Collins, P. E. by R. E. Isaacson

"Plant Process Evaluation Precipitation of Plutonium (IV) Oxalate"

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This supplement proposes that 50 runs be processed to include: (1) omission of one acid wash; (2) combining three batches of supernates in one Recycle Can; (3) addition of potassium permanganate or other oxidizing reagents to dissolve solids.

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PRODUCTION TEST 234-6

Test Proposal

HW-23937

Benoliel, R. W.

"Plant Process Evaluation - Use of Inconel Filter Boat" (3-27-52)

Objective: To demonstrate that an Inconel filter boat can be used satisfactorily in the RM Line - in particular that it will not be attacked by the hydrofluorination reaction and contaminate material handled in an Inconel boat.

References

Production Test 234-3.

MCW-150, C. F. Ritchie, "Corrosion Resistance of Several Nickel Alloys in HF-H<sub>2</sub>O Atmosphere at High Temperature", (6-19-51).

PRODUCTION TEST 234-7

Test Proposal

HW-23956

Benoliel, R. W.

"Plant Process Evaluation - Hydrofluorination of Product Turnings" (3-31-52)

Objective: To demonstrate that the procedure described, or with slight modification, is satisfactory for recycling plutonium turnings in the RG and RM Lines, i. e., satisfactory physical handling of turnings, hydrofluorination, physical properties of resulting fluoride, reduction to buttons and production of castings and finished shapes.

References

HW-21134, P. E. Collins, "Trip Report", (6-1-51).

HW-21219, G. A. Halseth, "Los Alamos Trip Report", (6-6-51).

HW-22232, W. L. Lyon, "Preliminary Memorandum on Processing Plutonium Metal Turnings", (9-25-51).

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PRODUCTION TEST 235-3

Test Proposal

HW-19614

Quinn, F. J., Jr.

"Use Test for Chemical 60-48 Received on OGT-10971-1 Container  
#H-11" (12-1-50)

Objective: to evaluate the use of chemical 60-48 (calcium)  
Grade P (as described in Reference 2 and received on OGT-  
10971-1) in the 235 Building.

References

GEH-17576, I. L. Lind, "Chemical 60-48 Purification Program",  
(11-3-50).

GEH-17577, N. Williams, "Calcium Purification Program",  
(10-26-50).

Letter, R. S. Bell to I. L. Lind, "Chemical 60-48 Purification  
Program", (11-8-50).

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PRODUCTION TEST 235-4

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PROCESS CHEMISTRY REPORTS

HW-3-706

Willard, J. E.

"(Separations Program)" (9-14-44)

Possible process difficulties as the result of the production of new and heavy isotopes in the Hanford Pile and an experimental program to assess these difficulties are outlined.

HW-3-811

Dreher, J. L.

"Re: Wind Dilution Required to Reduce to Tolerance Levels the Activity Due to Xenon and Iodine in H. E. W. Dissolver Off-Gases" (9-29-44)

Calculations are given for the wind dilution required to reduce activities of xenon and iodine to tolerance levels. These estimates are based on evolution rate data obtained from Site X.

DuH-4718\*

Kohman, T. P.

"Heavy Isotope Problems at Hanford" (10-5-44)

A listing of all the isotopes in the heavy element region which can conceivably be formed at Hanford in appreciable yields by known types of reactions, together with a discussion of their possible importance in connection with the Hanford process. Calculations are made for metal run for 100 days at a power intensity of 2.5 megawatts per ton, and such metal is referred to as "typical" pile metal. The concentration of plutonium 239 in "typical" metal will be 250 grams per ton of uranium.

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\* This is a Metallurgical Laboratory memorandum. It is included because the survey was made specifically to outline potential problems at Hanford.

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HW-3-1276

Kohman, T. P.

"Gamma Radiation and Recontamination of Product by Fissions"  
(10-23-44)

The amount of fission product activity resulting from fissions in plutonium solutions are calculated. Gamma radiation resulting from such fissions are also calculated.

HW-3-1424

Perkins, B. H.

"Influence of Hydrazine on Carrying of Product by Bismuth Phosphate in the Extraction Step (A literature survey covering information from sites C, X, and W.)" (11-20-44)

A compilation of the data available on the influence of hydrazine on the extraction step in the separations process.

HW-3-1123

Willard, J. E. by W. H. Sullivan

"Positioning of a Mica Window Counter Tube in Its Support" (11-24-44)

Recommendations are made for uniform positioning of counter tubes in all counters used for standard beta and gamma counting.

HW-3-1167

Sears, G. W. and H. T. Siefen

"Salicylaldehyde Method for the Analysis of Hydrazine" (12-4-44)

A method is described for the determination of hydrazine in process solutions.

HW-3-1198

Miller, D. R.

"Solubilities of Product Compounds under the Various Process Conditions" (12-8-44)

Solubilities of various plutonium compounds are compiled from Project literature. Emphasis is placed on the compounds of tetravalent plutonium but trivalent and hexavalent plutonium are considered. Plutonium compounds of main interest are the phosphate and fluorides.

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HW-3-1266

Thompson, S. G.

"Stability of Aluminate Solutions" (12-13-44)

Gives data concerning the stability of aluminate solutions formed during coating removal. Tests made on control of coating removal reaction rate by gradual addition of caustic are covered.

HW-3-1241

Sullivan, W. H.

"Health Hazard from a Long-Lived Rare Gas Activity" (12-16-44)

Calculations are given concerning the possible health hazard involved due to a rare gas activity appearing among the fission products.

HW-3-1300

Thompson, S. G.

"Optimum NaOH/NaNO<sub>3</sub> Ratio for Dissolving Tin" (12-27-44)

The rates of dissolution of the residual scale from decoated slugs in NaOH/NaNO<sub>3</sub> solutions are given. Data on the composition of the scale are included.

HW-3-1493

Swartout, J. A.

"Decontamination of Fission Elements in the Separation Process" (1-22-45)

This report is a survey of all data available at the time on decontamination of specific fission elements in the process. A critical appraisal of the data is included.

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SE-PC-1

HW-3-1331

Watt, G. W.

"Evaluation of Sample Can" (12-19-44)

The results of tests on the standard closure plug and modified plugs are given. The evaporation procedure for plutonium solution to dryness or to a non-fluid mass in the shipping container is described. Solidification and melting temperatures of plutonium nitrate solutions are given.

SE-PC-2

HW-3-1666

Miller, D. R.

"Purity of Product Isolated in 231 Building During January 1945"  
(2-19-45)

The analytical data obtained are presented along with a description of the methods used for making the analyses.

SE-PC-3

HW-3-1891

Perkins, B. H. and R. P. S. Black

"Recovery of Product from Lanthanum Fluoride By-Product  
Precipitates" (2-15-45)

Two processes are described for recovery of plutonium from lanthanum fluoride by-product precipitates and recommendations are made for carrying out the procedures in plant equipment.

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SE-PC-4

HW-3-1876

Watt, G. W.

"Summary of Plant and Laboratory Data Relating to January 1945 Runs in 231 Building (Runs T-4-12-B-5 through T-5-01-B-3)" (2-28-45)

This report covers in detail the data obtained in the laboratory and plant on the start-up series of runs through the Isolation Building. Recommendations for process modifications are included.

SE-PC-5

HW-3-1761

Swartout, J. A.

"Limiting Decontamination Factors Determined by Pu-239 Radiations" (3-3-45)

The maximum decontamination factors which can be achieved before reaching the limit of plutonium background for various plutonium concentrations have been calculated and tabulated.

SE-PC-6

HW-3-1846

Sullivan, W. H. and J. A. Swartout

"Identification of Fission Species Remaining at End of Concentration (224 Building) Process" (3-8-45)

The beta activity in the plutonium solutions is due primarily to praseodymium. The gamma activity is distributed between lanthanum, zirconium, niobium and ruthenium with the major portion in at least some of the runs being due to "orphan" lanthanum.

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SE-PC-7

HW-3-1814

Sullivan, W. H.

"Uniformity in Counting Samples for Beta and Gamma Radiation"  
(3-8-45)

Recommendations are given for standardization of counting procedures to maintain uniformity and thus permit plant wide comparisons of data.

SE-PC-8

HW-3-1943

Leader, G. R.

"Fission Product and Product Contamination in the T Area Process Cells and Ventilation System" (3-14-45)

Since it was desirable to know the nature of the contamination found deposited on the surfaces of some of the cells in the 221-T Building and in the fan-housing in the 291-T Building, relative to its origin as spray or volatile material, test "swipe" samples were taken and analyzed for ruthenium, iodine, plutonium, and several non-volatile fission products. A summary of the data is presented. From this it is concluded that, during succeeding production runs involving increasingly higher levels of activity, sufficient non-volatile fission products and plutonium will accumulate in the ventilation system to render normal servicing almost impossible.

SE-PC-9

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HW-3-1882

Sullivan, W. H.

"Instructions for Standardizing G-M Testers" (3-19-45)

Procedures are given for: (1) normalizing testing data, so that results obtained on any instrument are comparable; (2) determining geometry factors of the various shelves of the instrument; and (3) measuring coincidence losses for the G-M tube and scalar assembly.

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SE-PC-10

HW-3-1912

Watt, G. W.

"Memorandum Report on Production Test 231-1-SE" (3-21-45)

See Production Test 231-1, Memorandum Report, p. 38.

SE-PC-11

HW-3-1911

Watt, G. W.

"Memorandum Report on Production Test 231-2-SE" (3-22-45)

See Production Test 231-2, Memorandum Report, p. 39.

SE-PC-12

HW-3-1951

Siefen, H. T. and O. F. Hill

"Recovery of Product from First Cycle Ce-Zr Scavenger By-Product Precipitates" (2-24-45)

Procedures for the recovery of plutonium based on laboratory runs are described and recommendations are made for carrying out the process in plant equipment.

SE-PC-13

HW-3-1970

Swartout, J. A.

"Correction in B Decontamination Factor" (3-29-46)

Corrections are given for decontamination factors reported in HW-3-1910. The discrepancy was due to counting under non-standard conditions.

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HW-26352 026

SE-PC-14

HW-3-2278

Turk, E. H.

"Some Density Values and Crystallization Temperatures of UNH -  $\text{HNO}_3$  -  $\text{H}_2\text{SO}_4$  Mixtures" (4-10-45)

The following information is reported: (1) density of 40 per cent and 50 per cent UNH solutions at 40 C containing 0 to 4 per cent free nitric acid; (2) crystallization temperatures of 50, 60, 70, and 80 per cent UNH solutions containing free nitric acid ranging from 0 to 12 per cent; and (3) density of UNH- $\text{H}_2\text{SO}_4$  -  $\text{HNO}_3$  solutions at 20, 35, and 50 C in which the UNH concentration ranges from 16.5 per cent to 48 per cent and the weight ratio of sulfuric acid and free nitric acid to UNH remains constant.

SE-PC-15

HW-3-2106

Swartout, J. A.

"Correction of Beta Decontamination Factors Reported for March Runs" (4-11-45)

This document is a continuation and expansion of HW-3-1970. Sources of errors are pointed out. Suggestions are made for reporting data to clarify results and to place all data on a comparable basis.

SE-PC-16

HW-3-2183

Black, R. P. S. and B. H. Perkins

"Influence of Potassium Sulfate on the Decontamination of Zr-Nb in the  $\text{BiPO}_4$  By-Product Precipitation Step" (4-12-45)

Although some improvement in the decontamination of Zr-Nb activities in a bismuth phosphate by-product precipitation step was obtained by the addition of sulfate, the behavior was generally quite erratic and the degree of improvement realized was not sufficient to justify further exploratory work on a process as a replacement for the present scavenger procedure now used in the first cycle by-product precipitation step.

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HW-26352 022

SE-PC-17

HW-3-2131

Werner, L. B.

"Method for Analysis of  $\text{NH}_4^+$  in Solutions of High Product and High  $\text{HNO}_3$  Concentrations" (4-14-45)

The method described has been used to determine the ammonium ion concentration in AT solutions from the Isolation Building.

SE-PC-18

HW-3-2209

Swartout, J. A.

"Identification of the Radioactive Fission Elements Associated with Product at Various Stages Throughout the Separations Process Runs Made in February and March" (4-20-45)

The material in this document is presented under the following headings: I Effect of omitting one or both scavenger strikes in the first cycle by-product step on fission element distribution and decontamination; II Means of characterizing radiations from P-I (starting solution, Isolation Building) solutions - determination of half-life, determination of half-thickness; III Identification of radioactive contaminants present after the Concentration Process - significance of half-lives and half-thickness of radiation - evidence from chemical analyses; IV Completeness of decontamination following Isolation Process; V Evaluation of decontamination factors at the end of the Concentration and Isolation Processes.

SE-PC-19

HW-3-2211

Miller, D. R.

"Purity of Product Solutions from T-5-02 and T-5-03 Runs in the Isolation Building (231)" (4-23-45)

Extensive data are presented covering spectrographic and gravimetric determinations giving an indication of step-wise purification through the Isolation Process.

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HW-26352 026

SE-PC-20

HW-3-2530

Watt, G. W.

"Physical Properties of Solutions of Plutonium Nitrate in Nitric Acid"  
(4-27-45)

Data are given on physical properties of product solutions encountered in the later stages of the Isolation Process. Solidification temperatures, boiling points, and densities are included.

SE-PC-21

HW-3-2296

Faris, B. F.

"Use of Potassium Permanganate as the Only Oxidant in the Cross-Over" (5-2-45)

The laboratory results are tabulated and show that potassium permanganate is at least as effective an oxidizing agent as is sodium bismuthate.

SE-PC-22

HW-3-2387

Swartout, J. A.

"Methods for the Determination of Decontamination Factors by Measurement of the Ratio of Beta or Gamma Counts to Alpha Counts in a Single Solution" (5-4-45)

An independent method is described for determining overall beta or gamma decontamination factors through any step in the separation process. A series of four nomographs is included. Each nomograph covers a different level of activity and range of decontamination factors.

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HW-26352 D&L

SE-PC-23

HW-3-2467

Watt, G. W.

"Diagrams of Process Equipment (Isolation Building)" (5-4-45)

The first of these diagrams shows the sequence of transfer of product solution, and the second is a similar diagram relating to the treatment of solutions recycled to the Concentration Buildings. The third and fourth diagrams are section drawings of a precipitator and vacuum receiver (head tank), respectively, while the fifth is a complete piping diagram for the cell equipment in each 26 foot hood.

SE-PC-24

HW-3-2516

Sears, W. G.

"Effect of Lead upon the Bismuth Phosphate Separation Process" (5-7-45)

This study was undertaken to determine what difficulties in chemical processing, if any, would be occasioned by the accidental introduction of lead (from a dummy slug) into the uranyl nitrate solution.

SE-PC-25

HW-3-2529

Kay, W. L.

"Bowl Metathesis of Bismuth Phosphate" (5-7-45)

Procedures are given for the metathesis of bismuth phosphate to bismuth hydroxide by a potassium hydroxide solution wash of the extraction precipitate at volume ratios permitting treatment in a centrifuge bowl. Process applications are discussed. A summary of results obtained from larger scale tests in the Semi-Works is included.

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HW-26352 DEL

SE-PC-26

HW-3-2410

Werner, L. B. and D. R. Miller

"Solubility of Ammonium Plutonium Nitrate in Constant-Boiling  $\text{HNO}_3$ "  
(5-10-45)

Ammonium plutonium nitrate,  $(\text{NH}_4)_2\text{Pu}(\text{NO}_3)_6$ , is identified as the compound precipitating from AT solutions. Solubility values are given and Isolation Process implications are indicated.

SE-PC-27

HW-3-2531

Turk, E. H.

"Rates of Solution and Solubility of Plant Bismuth Phosphate Cakes in Nitric Acid" (5-10-45)

The rates of dissolution and the solubilities of bismuth phosphate by-product and product cakes are shown for acidities ranging from 2 to 10 M nitric acid.

SE-PC-28

HW-3-2371

Faris, B. F.

"The Use of Ammonium Fluosilicate Analogs for Improving Decontamination in Product Precipitation Steps" (5-10-45)

A large number of inorganic and organic compounds were evaluated as solubilizing agents for colloidal fission products, and their effectiveness was compared with that of fluosilicic acid. Of those tested, only the metal fluo-complexes showed promise.

SE-PC-29

HW-3-2411

Sullivan, W. H.

"The Specific Activity of Plutonium" (5-14-45)

The specific activity values of plutonium, based on the most recent experimental data are given. A bibliography is included.

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HW-26352 D&L

SE-PC-30

HW-3-2533

Turk, E. H.

"Investigation of Quantity of Sodium Carbonate Required for Neutralization of Extracted Metal Waste Solution" (5-14-45)

Laboratory tests showed that greater than 90 per cent of the flowsheet requirements of sodium carbonate were necessary to reduce the solids content (volume basis) to less than 0.2 per cent. Use of 60 per cent of flowsheet amount of sodium carbonate gave a slurry that was of low viscosity but which contained 46 per cent solids on a volume basis. Since laboratory tests have shown some past precipitation of solids even from completely neutralized metal waste solutions, it appears advisable from the viewpoint of solution stability to use the present flowsheet quantities of sodium carbonate.

SE-PC-31

HW-3-2415

Watt, G. W.

"Pre-Isolation Reduction of Plutonium (VI) by Means of Ammonium Sulfite" (5-16-45)

The experimental work done on the development of the ammonium sulfite procedure for the reduction of plutonium in Isolation Process starting solutions is reported.

SE-PC-32

HW-3-2535

Miller, D. R.

"Solubilities of Plutonium Compounds in Some Process Solutions" (6-1-45)

Experimental data are given on solubility of plutonium compounds in various process solutions, supplementing data in HW-3-1198.

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HW-26352 D & C

SE-PC-33

HW-3-2511

Stahl, G. W. and W. F. Schneller

"Decrease in Reduction Time Prior to Bismuth Phosphate Product Precipitation" (5-18-45)

In the effort to decrease the time cycle in the various process steps, the bismuth phosphate product precipitations have been critically examined. This report describes the laboratory results and the evidence in the literature that suggests the one-hour digestion at 75 C prior to the bismuth strike can be eliminated.

SE-PC-34

HW-3-2513

Kay, W. L.

"Specific Gravities of Certain  $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  Solutions" (5-18-45)

Specific gravities of UNH solutions over specified limited concentration ranges and temperatures are reported. These data are supplemental to those reported in SE-PC-14.

SE-PC-35

HW-3-2496

Watt, G. W.

"Completeness of Precipitation of Plutonium (IV) Peroxide as a Function of Concentration of Hydrogen Peroxide" (5-22-45)

The feasibility of precipitating plutonium (IV) peroxide using concentrations of hydrogen peroxide less than 10 per cent by weight has been explored using both simulated and real process solutions. Although the use of as little as 4 per cent hydrogen peroxide by weight gave entirely satisfactory results when simulated process solutions were employed, those obtained using process solutions from the plant were so much less favorable that further consideration has not been given to use of less than 10 per cent hydrogen peroxide in the plant. Some, if not all, of the advantages inherent in such a process change have been realized by other means.

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HW-26352 D&L

SE-PC-36

HW-3-2537

Dreher, J. L.

"Cause and Prevention of Uranium Precipitates in the Extraction of Neptunium" (5-23-45)

This report covers the laboratory investigations of the difficulties encountered in the plant in the run made to recover neptunium from the waste metal solution of a standard run. Recommendations are made as to process changes necessary to avoid the difficulty.

SE-PC-37

HW-3-2477

Hill, O. F.

"A Method for Decontamination and Extraction of Plutonium from Carbonate Complexed Uranyl Solutions" (5-26-45)

Preliminary experiments on an alternate procedure, based on extraction of plutonium from an alkaline uranyl solution, are described. Because of more pressing work, further work was discontinued.

SE-PC-38

HW-3-2541

Knox, W. J.

"Search for a Pre-Extraction Scavenging Method for Rare Earth Fission Product Activities" (5-26-45)

Preliminary investigation of methods for separating lanthanum activity from UNH solutions prior to extraction failed to reveal any promising procedures. All of the compounds tried were unsatisfactory for one of the following reasons: (1) compound was too soluble to precipitate in slightly acid UNH solutions, (2) the anion used to precipitate the compound resulted in precipitation of uranium, or (3) the compound did not carry lanthanum activity.

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SE-PC-39

HW-3-2542

Kay, W. L.

"Literature Survey of Reducing Agents for Plutonium for Possible Plant Use" (5-28-45)

Since some difficulty has been experienced in obtaining a sufficient supply of ferrous ammonium sulfate to meet the anticipated needs of the plant in the near future, a search of the project literature dealing with alternate reducing agents has been made. The advantages and disadvantages of each that was considered are recorded.

SE-PC-40

HW-3-2543

Werner, L. B.

"Calibration of Quartz Microbalance" (6-1-45)

Details are given for the procedures which were used in calibration of a Kirk-Craig quartz microbalance.

SE-PC-41

HW-3-2558

Watt, G. W.

"The Isolation of Plutonium by a Process Involving Only One Precipitation of Plutonium (IV) Peroxide" (5-28-45)

A procedure is described, based on laboratory tests on plant solutions, for obtaining adequate final purity by an isolation procedure involving a single peroxide precipitation step. Recommendations are made for further study either in the laboratory or on a plant scale.

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HW-26352 DEL

SE-PC-42

HW-3-2583

Watt, G. W.

"The Elimination of Hydrogen Peroxide from Solutions Remaining After Isolation of Plutonium as Plutonium (IV) Peroxide" (6-4-45)

The various procedures used in the Isolation Building for killing peroxide are reviewed and evaluated. The development and application of an electrometric method for detecting the end point in the reaction between hydrogen peroxide and sodium nitrite are described in detail.

SE-PC-43

HW-3-2596

Watt, G. W.

"The Rate of Decomposition of Hydrogen Peroxide in Isolation Process Solutions" (6-4-45)

The rate of decomposition of hydrogen peroxide in simulated Isolation Process waste solutions has been found to be dependent upon the concentration of hydrogen peroxide and iron over the concentration ranges 2.5 - 10 per cent by weight and 0.001 - 0.003 M, respectively. The rate of reaction shows a marked dependence upon temperature over the range 25-45 C (77-113 F) and the energy of activation was calculated to be 22.6 kilocalories per mole. Data are given on the rate of decomposition of hydrogen peroxide as a function of temperature, iron content and hydrogen peroxide concentration.

SE-PC-44

HW-3-2597

Angerman, A. H.

"Crystalline Lanthanum Fluoride" (6-11-44)

Methods are described for preparing crystalline lanthanum fluoride. The results of evaluation runs on both a laboratory and a semi-works scale are given.

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SE-PC-45

HW-3-2609

Miller, D. R.

"Summary of Results of Investigations on the Influence of Hydrazine in the Bismuth Phosphate Extraction Step" (6-11-45)

A summary is given of experiments carried out relative to (1) formation of hydrazine during metal dissolution, (2) stability of hydrazine in extraction solutions, (3) destruction of hydrazine with sodium nitrite, and (4) mechanism of hydrazine interference with carrying.

SE-PC-46

HW-3-2646

Kohman, T. P.

"A General Method for Determining Coincidence Corrections of Counting Instruments" (6-13-45)

The theory of the method is discussed and details are given for application of the method including procedures for taking the data, examination of the data, making computations and in plotting of the data.

SE-PC-47

HW-3-2610

Johnson, W. C. and D. R. Miller

"Some Possible Effects of Sulfate and Pyrophosphate on the Isolation Process (231 Building)" (6-14-45)

Conditions are described under which plutonium sulfate precipitation may occur. Data are given for the solubility of plutonium sulfate in constant boiling nitric acid. Experiments on the possible effects of phosphate and pyrophosphate in the Isolation Process are described, particularly as related to the use of pyrophosphate stabilized hydrogen peroxide.

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HW-26352 DEL

SE-PC-48

HW-3-2694

Watt, G. W.

"An Alternate Procedure for the Recovery of Plutonium from Isolation Process Wastes" (6-4-45)

Two procedures are described: one based on destruction of hydrogen peroxide in wastes by increasing the acidity and by heating of the solutions (with or without catalyst), the other based on recycling of waste solutions to the 10N nitric acid solution of bismuth phosphate at the beginning of crossover and destroying hydrogen peroxide by heating this solution.

SE-PC-49

HW-3-2705

Watt, G. W.

"Properties of Plutonium in Solutions Containing Ammonium Sulfite" (6-13-45)

Procedures are described for preparing fairly concentrated, yet substantially neutral solutions of plutonium by dissolution of the peroxide in ammonium sulfite solutions. The properties of such solutions are described. The absorption spectra of various solutions are included.

SE-PC-50

HW-3-2621

Kohman, T. P.

"I Calculation of Changes in Specific Activity of Plutonium as a Function of Neutron Exposure of Pile Uranium. II The Half Life of Pu-240" (6-18-48)

Calculations for the change of specific activity as related to the concentration per ton of uranium. Calculations of the half-life of plutonium-240 are based on the experimentally determined specific activity of plutonium (150 g/t) given in HW-3-2622.

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HW-26352 DEL

SE-PC-51

HW-3-2622

Hoekstra, H. R.

"Determination of the Ratio of Specific Activity of 150 g/T Hanford Plutonium to Site X Plutonium" (6-18-45)

In addition to the determination of the ratio of the specific activity of HW plutonium to that of Site X plutonium, the data obtained indicated corrections to be made in counter geometry factors and comparisons of counting rates on platinum and glass.

SE-PC-52

HW-3-2675

Knox, W. J.

"Studies on the Centrifugibility of  $\text{LaF}_3$ " (6-19-45)

No very pronounced chemical effects on the centrifugibility of lanthanum fluoride were found in any case. Several variations (increased plutonium concentration, increased lanthanum concentration, increased hydrogen fluoride concentration, higher digestion temperatures, decreased agitation, decreased nitric acid concentration, use of cerous fluoride carrier in place of lanthanum fluoride and decreased concentrations of the constituents of the solution from previous steps) from the standard procedure produced an improvement in the separation of lanthanum fluoride as judged by controlled slow speed laboratory centrifugation.

SE-PC-53

HW-3-2708

Stahl, G. W. and W. F. Schneller

"Method for Obtaining a Decontamination Factor of  $10^5$  at the End of One Decontamination Cycle" (6-19-45)

A low acidity process has been devised that effects  $10^5$  gamma decontamination in a single cycle, but its use results in product losses ranging from 5 to 9 per cent.

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HW-26352 DEL

SE-PC-54

HW-3-2709

Siefen, H. T.

"The Use of KOH-Glycerol as a Reagent for Dissolving  $\text{BiPO}_4$ .  
Substitution of a Third Bismuth Phosphate Decontamination Cycle for  
the Lanthanum Fluoride Concentration Cycle" (6-20-45)

The promising laboratory results obtained on an alternate concentration process are described and discussed. Data are included on the properties of potassium hydroxide-glycerol solutions of bismuth phosphate, on tests evaluating process applications and on a preliminary survey of other reagents as substitutes for glycerol.

SE-PC-55

HW-3-2823

Stahl, G. W. and W. F. Schneller

"The Effect of Ammonium Fluosilicate on Decontamination in the  
Second Cycle Product Precipitation Step" (6-21-45)

Comparative second cycle product precipitations made in the laboratory from plant solutions showed that the decontamination was increased in this step by the presence of fluosilicic acid. On this basis it is recommended that the use of fluosilicate in the second decontamination cycle product precipitation step be continued.

SE-PC-56

HW-3-2667

Watt, G. W.

"The Use of Low Concentrations of Lanthanum and Hydrofluoric Acid  
in the Lanthanum Fluoride Product Precipitation" (6-19-45)

The extent by which lanthanum fluoride carrier can be reduced and still give adequate carrying is indicated. The efficiency of lanthanum fluoride removal by the plant centrifuge is the factor which determines the concentration of lanthanum fluoride used as carrier.

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HW-26352 DEL

SE-PC-57

HW-3-2825

Dreher, J. L.

"Decomposition of  $\text{NaBiO}_3$  and Oxidation of Product by  $\text{NaBiO}_3$  and by  $\text{Pb}_3\text{O}_4$ " (6-27-45)

The rates of oxidation of plutonium and decomposition of sodium bismuthate as related to acidity and temperature were studied and the data recorded. Data on the use of red lead oxide as an alternate oxidant are included.

SE-PC-58

HW-3-2706

Dreher, J. L.

"Studies on the Improvement of Decontamination in the By-Product Step of the First Decontamination Cycle (With and Without Scavengers)" (6-28-45)

A summary is given of various experiments directed toward simplification and improvement of the first cycle by-product precipitation step. Experiments are included on the preparation and evaluation of "crystalline" zirconium and ceric phosphates as scavengers.

SE-PC-59

HW-3-2707

Dreher, J. L.

"Investigation of Partial Neutralization of the Uranyl Nitrate Solution Prior to Extraction and of the Complete Neutralization of the Extraction Waste Solution" (6-29-45)

Limits are established for the extent to which free acids (sulfuric and nitric) in the UNH solution can be neutralized prior to extraction without inducing uranyl phosphate precipitation. Recommendations are made for complete neutralization of extraction wastes of variable composition.

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HW-3-2857

Peterson, J. H.

"Removal of Zirconium-Niobium Activities from Extraction Step  $\text{BiPO}_4$  Precipitates by Various Washing Procedures" (6-29-45)

Data are summarized on the use of various wash solutions, including: ammonium acid fluoride and other fluorides, phosphoric acid, and various organic acids (oxalic, acetic, tartaric, citric and benzoic). A 10 per cent solution of ammonium acid fluoride was found to be most effective. Data on the corrosion of 25-12 stainless steel by ammonium acid fluoride solutions are included.

SE-PC-61

HW-3-2824

Hill, O. F.

"Recovery of Product from  $\text{BiPO}_4$  By-Product Precipitates" (7-2-45)

A summary of: recovery of plutonium from second cycle bismuth phosphate by-product precipitates; recovery from cross-over cycle bismuth phosphate by-product precipitates; recovery of first cycle scavenger precipitates; and experimental details.

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HW-3-2736

Kohman, T. P. and J. A. Swartout

"The Alpha Disintegration of Pu-241 in HEW Product" (7-3-45)

A method of separating tracer quantities of uranium from macro quantities of plutonium has been developed. From 525 milligrams of 60 gt Hanford plutonium, beta activity assignable to U-237 has been isolated. The yield indicates that the ratio of Pu-240 capture cross section (in barns) to Pu-241 alpha half-life (in years) is circa  $1.5 \times 10^{-3}$ , uncertain by a factor of two. Assuming the most probable value for the half-life, circa  $5 \times 10^5$  years, the corresponding cross section is circa 800 barns. The effect of Pu-241 on the specific alpha activity of Hanford plutonium and of Pu-240 on the reactivity of Hanford piles will be small. High-power Hanford plutonium will be a convenient source of U-237 of high specific activity.



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SE-PC-63

HW-3-2767

Stahl, G. W. and W. F. Schneller

"Decontamination Obtained by the H. E. W. Separations Process if Scavengers are Omitted" (7-4-45)

Gross gamma decontamination factors of  $10^8$  were obtained at the end of the Concentration Cycle of the Bismuth Phosphate Process run without scavengers but using ammonium fluosilicate in each product precipitation step of the two decontamination cycles. It is suggested that if the plant should desire to make use of the advantages of the fluosilicate process, it might be approached gradually through a reduction in the amount of and finally the elimination of the zirconium scavenger, followed by the reduction and elimination of the ceric scavenger.

SE-PC-64

HW-3-2801

Swartout, J. A.

"Source of High Activity Levels Recently Observed from Shipping Containers and Transfer Cans" (7-5-45)

The radioactive contaminant present in the final product has been identified rather definitely as neptunium-239. The increase was due primarily to the increase in power levels and batch size and the trend toward shorter cooling times. Roughly two thirds of the gamma activity was due to neptunium and most of the remaining third to lanthanum. Because of the rapid decay of each of these isotopes and because of the efficient removal of lanthanum in the Isolation Process, the hazard is not considered to be serious.

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HW-3-2900

Kay, W. L. and A. H. Angerman

"Volume Reduction Effected by Partial Neutralization of the Oxidized Solution Prior to the  $\text{BiPO}_4$  By-Product in the Cross-Over Cycle" (7-6-45)

Partial neutralization of process solutions was studied at Site C in connection with the low acidity by-product procedure where it

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SE-PC-65 (contd.)

was found that the use of certain salts did not increase by-product losses. No difficulty was encountered in carrying of reduced plutonium by bismuth phosphate following this partial neutralization procedure.

SE-PC-66

HW-3-2901

Werner, L. B.

"Specific Activity of  $95^{241}$  from H.E.W. Product" (7-9-45)

By isolation and weighing of a sample containing approximately  $1.2 \times 10^7$  disintegrations per minute of americium-241, a minimum value for the specific activity of  $1.44 \times 10^6$  disintegrations per minute per microgram has been determined, yielding a maximum value for the half-life of 2,280 years.

SE-PC-67

HW-3-2703

Watt, G. W.

"Memorandum Reports on Production Tests 231-3-SE, 231-4-SE, and 231-5-SE" (6-21-45)

Nine modifications in the details of the Isolation Process have been tested in the plant during the past three months. These changes have resulted in (a) marked decreases in cycle time, (b) improvement in the purity of the final product, and (c) increased safety of operation of the process. Upon plant demonstration of the anticipated process improvements, each of the experimental conditions or procedures was adopted as a permanent feature of the process unless superseded by changes which resulted in still further improvement.

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SE-PC-68

HW-3-3048

Sullivan, W. H. and T. P. Kohman

"Corrections and Predictions Concerning the Radioactive Properties of Heavy Isotopes" (8-4-45)

Data concerning the known heavy isotopes are correlated and predictions are made relative to the formation of other isotopes. Radioactive properties of these isotopes are also predicted.

SE-PC-69

HW-3-2921

Hoekstra, H. R.

"Some Phases of the Alkaline Chemistry of Plutonium" (7-20-45)

Results of experiments on: oxidation and polymerization of plutonium during metathesis; prevention of plutonium oxidation during methathesis; reduction of plutonium (IV) to plutonium (III) in an alkaline medium; and preparation and properties of plutonium (V) are given in this report.

SE-PC-70

HW-3-2898

Turk, E. H.

"Corrosion Studies on 25-12 and 18-8 Stainless Steel" (7-21-45)

The corrosion rate data are tabulated along with reference to Clinton Laboratory reports containing complementary data.

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HW-3-2905

Stahl, G. W. and W. F. Schneller

"Dissolution of  $\text{BiPO}_4$  by Complexing Agents in Acid Media" (7-24-45)

Most of the experimental work covered applies to the dissolution of bismuth phosphate in media ranging from 0.75M ammonium chloride - 1.75M nitric acid to 1.75M ammonium chloride - 0.75M nitric acid. Process applications are indicated. Extensive data on corrosion of stainless steel by such solutions are given.

SE-PC-72

HW-3-2910

Angerman, A. H.

"Crystalline Lanthanum Fluoride" (7-26-45)

This report covers the continuation of experimental work reported in SE-PC-44 (HW-3-2597) on the evaluation of crystalline lanthanum fluoride. Data obtained from plant evaluation test runs are also included.

SE-PC-73

HW-3-2996

Sears, G. W.

"Development of Methods for the Preparation of Sodium Lanthanum Sulfate" (7-30-45)

Describes methods developed for preparing sodium lanthanum sulfate which results in a minimum of fines on subsequent metathesis to crystalline lanthanum fluoride.

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SE-PC-74

HW-3-3003

Dreher, J. L.

"The Evolution of Iodine During Metal Dissolution" (8-2-45)

This report covers laboratory studies made to establish factors influencing the evolution of iodine during the metal dissolution step. Data are also given pertaining to the decontamination of iodine through the dissolution, solution storage, and extraction steps in the process.

SE-PC-75

HW-3-3221

Ballou, N. E.

"Fission Product Studies with Plant Dissolver Solutions" (10-18-45)

In the initial work to isolate, identify, and characterize some of the less well-known fission product activities appearing in plant dissolver solutions, a systematic analytical scheme was employed for separating the fission products into several fractions prior to isolation operations so that a maximum amount of information could be obtained with the limited volume of solution available. This report deals specifically with the daughters of active cerium. The isolation procedures and physical measurements for a number of other fission products appear in reports SE-PC-77 to 81.

In general, the work was directed to establish the existence or prove the absence of certain long-lived activities which have been reported in previous project literature or which theoretically could be present, according to the Bohr-Wheeler calculations for unstable nuclei. Most of the activities which were sought occur in elements whose isotopes lay on the extremities of the fission yield versus mass number curves and which could be studied most easily in solutions of high specific activity. Therefore tank dissolver solutions were used, since they were available and were representative of material being processed in the canyons.

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SE-PC-76

HW-3-3070

Peterson, J. H.

"Listing of Memorandum Reports Issued by the Process Chemistry Section" (8-23-45)

This report contains a list of all reports prepared and issued by the Process Chemistry Section of the Separations Engineering Division. A brief statement is given indicating the scope of each report. It is planned that a supplementary list will be prepared later covering additional reports which are being or will be prepared.

SE-PC-77

HW-3-3338

Swartout, J. A.

"Fission Product Studies with Plant Dissolver Solutions - III - Radium, IV - Molybdenum" (1-8-46)

Continuing the studies (SE-PC-75 and 77 - 81) to characterize more completely the fission product activities found in the piles, this memorandum reports that according to present experimental data, no long-lived rhodium or molybdenum activities are produced in pile fission.

SE-PC-78

HW-3-3208

Lincoln, D. C.

"Fission Product Studies with Plant Dissolver Solutions - V - Element 43, VI - Palladium" (10-24-45)

Of the studies (SE-PC-75 and 77 - 81), this report covers the elements technetium and palladium. In the examination for a long-lived technetium activity, an activity has been found which has decay and radiation characteristics unlike any fission product activity thus far identified. Attempts to isolate a long-lived palladium activity have only demonstrated that if isotope palladium-107 is long-lived, its half-life must be exceedingly long (of the order of  $10^8$  years).

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SE-PC-79

HW-3-3330

Leader, G. R.

"Fission Product Studies with Plant Dissolver Solutions - VII - Tin"  
(1-9-46)

In a series of studies (SE-PC-75 and 77 - 81), this report covers tin. Although previous work on tin had shown that at least three fission product activities, with half lives of 80 minutes, 62 hours, and 10 days, existed, the possibility that much longer-lived activities were present was not excluded. A long lived tin activity, with a half-life of 130 days, was identified in the plant dissolver solution.

SE-PC-80

HW-3-3370

Leader, G. R.

"Fission Product Studies with Dissolver Solutions - VIII - Antimony"  
(1-17-46)

In a series of studies (SE-PC-75 and 77 - 81) to identify and characterize some of the less well known fission product activities as outlined in SE-PC-75, attention was directed to the controversial approximately 400 day antimony activity. This report confirms the existence of this isotope in plant dissolver solutions.

SE-PC-81

HW-3-3366

Leader, G. R.

"Fission Product Studies with Plant Dissolver Solutions - IX - Iodine, X - Arsenic" (1-8-46)

In continuing studies (SE-PC-75 and 77 - 81) to characterize and identify fission product activities formed in piles, work was done to determine whether or not long-lived isotopes of certain elements were present. This report describes the work done on iodine and arsenic.

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HW-26352 O&L

SE-PC-82

HW-3-3220

Leader, G. R. and B. F. Faris

"A Study of Decontamination Cycle Waste Solutions and Methods of Preparing Them for Disposal" (10-29-45)

The initial purpose of this study was to determine how much plutonium and activity remain in solution after the precipitate has settled. Later, attention was directed to finding means for making the plutonium and activity precipitate more completely.

SE-PC-83

HW-3-3226

Hoekstra, H. R.

"The Determination of the Specific Activity of 265 gt Plutonium" (11-12-45)

The experimental work described in this report may be considered a continuation of that reported previously (SE-PC-51 - HW-3-2622 "The Determination of the Specific Activity of 150 gt Plutonium"). The over-all problem is to determine the rate of increase in specific activity of plutonium with increasing gt (grams of plutonium per ton of uranium) level. Such determinations are valuable in giving information concerning plutonium isotopes other than plutonium-239, which are formed in the piles, and also, to permit correlation of the radioassay and chemical methods of analysis for plant solutions.

SE-PC-84

HW-3-3332

Kamack, H. J.

"Fission Product Activities in Irradiated Uranium Cooled for Long Periods" (1-4-46)

Curves are presented to show the activities due to the individual fission products in natural uranium which has been uniformly irradiated with slow neutrons. These curves supplement those of the Project Handbook, CL-697, by extending to longer irradiation and longer cooling times.

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SE-PC-85

HW-3-3337

Lindner, M.

"Development of the Oxalate Method for Concentration of Isolation Building Wastes" (1-14-46)

A method has been developed for concentrating the Isolation Building waste liquors into a volume of 45 liters or less. This involves the carrying of plutonium, in slightly alkaline solution, on lanthanum oxalate initially precipitated from a solution 0.5N or less in acid. The lanthanum oxalate is dissolved in a relatively small volume of nitric acid and potassium permanganate. The resultant solution is then shipped to the Concentration Building in one recycle can.

SE-PC-86

HW-3-3336

Faris, B. F.

"The Use of Fluomolybdic Acid for Improving Decontamination in the Product Precipitation Step" (1-9-46)

Laboratory data indicate that fluomolybdic acid is several times as efficient as fluosilicic acid in effecting decontamination in the bismuth phosphate product precipitation step. A no-scavenger process would effect a saving of 1.0 per cent of plutonium. The fluomolybdic acid is recommended for plant evaluation.

SE-PC-87

HW-3-3367

Kamack, H. J.

"Calculations Concerning the Formation of Isotope  $95^{241}$  in a Chain-Reacting Uranium Pile" (1-18-46)

An equation for calculating the rate of formation of americium-241 in a chain-reacting uranium pile was developed. Calculations indicate that the amounts of americium-241 are not sufficient to cause a noticeable change in  $k$ , the pile reproduction factor.

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HW-26352 022

SE-PC-88

HW-3-3347

Sullivan, W. H.

"Hydraulically-Operated Tongs for Remote Control Hot Laboratory Operations" (1-16-46)

With the construction of a hot laboratory at HEW for handling activities at a level of one curie or less, the problem of designing tongs suitable for the manipulation of equipment behind the lead barricade was given consideration. The present report describes the development of the tongs currently in use.

SE-PC-89

HW-3-3409

Martens, R. I.

"Radiochemical Components of Films Deposited from 100 Area Cooling Water" (2-14-46)

Four film samples from 100-D, B, and F areas have been examined for alpha, beta, and gamma activities. It was not possible to identify activities with much certainty since the decay curves were complex and difficult to resolve; with the aid, however, of spectrochemical analyses some tentative assumptions of identity were made. On one of the samples it was possible to establish the presence of 2.6h manganese, 12.8h copper, 26.5d chromium, 42d tellurium, 14.3d phosphorus and probably 14.8h sodium, and the absence of 2.5h calcium and certain fission products in the film, but the total beta or gamma activity identified was only approximately 20 per cent of the total shortly after the end of bombardment. Analyses for plutonium were made on each of the samples.

SE-PC-90

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HW-3-3410

Pye, D. G. and J. B. Work

"Insoluble Solids Associated with Final Product from the Isolation Process" (2-18-46)

Experiments determined that the material troubling the customer was stannic oxide and was present in the 30 per cent plutonium nitrate solution before final evaporation in the sample cans. It

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SE-PC-90 (contd.)

could be removed by filtration through sintered glass. Corrective measures were started in two directions: one involving modification of the Isolation Process to include filtering and the other, the use of "Becco" instead of "Albone" hydrogen peroxide.

SE-PC-91

HW-3-3427

Kay, W. L.

"Retention Characteristics of 200 Area Soil for Product" (2-22-46)

The conclusions drawn from this study must be somewhat qualitative in nature as the soil was not completely saturated with plutonium (100 per cent break-through of plutonium with effluent) and time was not available for a study of the leaching characteristics of the soil. The soil has a marked capacity for accepting plutonium but the pick-up from an alkaline solution is not complete unless the contact time between soil and solution is rather long. This is borne out by the appearance of plutonium in the column effluent long before the soil is completely saturated. Also, the plutonium content of the effluent increases with the rate of flow of the waste.

SE-PC-92

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HW-3-3428

Kay, W. L.

"Decontamination of Product and Fission Product Activity from Various Surfaces" (2-26-46)

The relative ease of contaminating surfaces was determined, the order from most active to least active of those tested being: brass, Bakelite, Tulox HR, Koroseal, aluminum, varnished steel, mild steel, polystyrene, stainless steel, and black Duco on brass. Satisfactory procedures for decontaminating plastics, glassware, and porcelain are known. Decontamination of mild steel, stainless steel, uncoated brass, painted surfaces, painted and unpainted concrete, and linoleum is discussed. Rubber and unpainted wood should be removed from service when contaminated. Hand and laundry decontamination methods are given. A bibliography is included.

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SE-PC-93

HW-3-3639

Johnston, W. H.

"An Investigation of the Stability of Neutralized Extracted Metal Waste"  
(8-30-46)

The plant practice in metal waste disposal is to neutralize the solution partially with 50 per cent sodium hydroxide, then add an established quantity of 30 per cent sodium carbonate to keep the uranium in solution as a soluble complex carbonate prior to transfer to the storage tanks. On standing at elevated temperatures, a precipitate containing uranium is formed. This investigation studies this precipitation and attempts to improve the stability of these neutralized metal wastes. An improvement in stability was accomplished on a laboratory scale and is described in this report.

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