

**Atlantic Richfield Hanford Company**  
Richland, Washington 99352

RL-SEP-352



OPERATIONAL REPORT ON THE PUREX PLANT  
THORIUM PROCESS TEST

JP Duckworth  
DG Harlow  
CC Herrington

March 15, 1965

PREPARED FOR THE U. S. ATOMIC ENERGY  
COMMISSION UNDER CONTRACT AT(45-1) 2130

PRELIMINARY REPORT

THIS REPORT CONTAINS INFORMATION OF A PRELIMINARY NATURE. IT IS SUBJECT TO REVISION OR CORRECTION AND THEREFORE DOES NOT REPRESENT A FINAL REPORT. IT WAS PREPARED PRIMARILY FOR INTERNAL USE WITHIN THE ATLANTIC RICHFIELD HANFORD COMPANY ANY EXPRESSED VIEWS AND OPINIONS ARE THOSE OF THE AUTHOR AND NOT NECESSARILY OF THE COMPANY.

NOTICE

THIS REPORT WAS PREPARED AS AN ACCOUNT OF WORK SPONSORED BY THE UNITED STATES GOVERNMENT. NEITHER THE UNITED STATES NOR THE UNITED STATES ATOMIC ENERGY COMMISSION, NOR ANY OF THEIR EMPLOYEES, NOR ANY OF THEIR CONTRACTORS, SUBCONTRACTORS, OR THEIR EMPLOYEES, MAKES ANY WARRANTY, EXPRESS OR IMPLIED, OR ASSUMES ANY LEGAL LIABILITY OR RESPONSIBILITY FOR THE ACCURACY, COMPLETENESS OR USEFULNESS OF ANY INFORMATION, APPARATUS, PRODUCT OR PROCESS DISCLOSED, OR REPRESENTS THAT ITS USE WOULD NOT INFRINGE PRIVATELY OWNED RIGHTS.

## **DISCLAIMER**

**This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.**

## **DISCLAIMER**

**Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.**

**DECLASSIFIED**

RL-SEP-352  
Page 1

This document consists of  
75 pages. No. 15 of  
35 copies.

OPERATIONAL REPORT ON THE PUREX PLANT  
THORIUM PROCESS TEST

By

J. P. Duckworth, D. G. Harlow and C. C. Herrington

Purex Operation  
CHEMICAL PROCESSING DEPARTMENT

March 15, 1965

Operated for the Atomic Energy Commission  
By the General Electric Company  
Under Contract # AT (45-1)-1350

HANFORD ATOMIC PRODUCTS OPERATION  
Richland, Washington

Restricted Data

This document contains restricted data  
as defined in the Atomic Energy Act of  
1954. Its transmittal or the disclosure  
of its content in any manner to an  
unauthorized person is prohibited.

GROUP 1

Excluded from automatic downgrading and declassification

**DECLASSIFIED**

DISTRIBUTION

1. O. F. Beaulieu
2. J. M. Blackburn
3. A. G. Blasewitz-W. H. Swift
4. R. E. Burns
5. R. A. Connell
6. J. P. Duckworth
7. J. B. Fecht
8. J. W. Fillmore
9. C. B. Foster
10. M. K. Harmon
11. W. M. Harty
12. R. E. Isaacson
13. J. B. Kendall
14. W. Koontz-P. S. Kingsley
15. C. W. Malody ✓
16. R. W. McCullugh
17. L. R. Michels
18. A. M. Platt
19. H. C. Rathvon
20. H. P. Shaw
21. O. V. Smiset
22. S. G. Smolen
23. R. E. Tomlinson
24. A. J. Waligura
25. M. T. Walling
26. J. H. Warren
27. W. K. Woods
28. L. L. Zahn
- 29-33. Extra
34. 700 Files
35. Extra

TABLE OF CONTENTS

- I. INTRODUCTION
- II. SUMMARY
- III. CONCLUSIONS
- IV. DISCUSSION
  - A. Thorium Run Plans
    - 1. Feasibility and Equipment Needs
    - 2. Planning and Scheduling
    - 3. Routing and Equipment Changes
    - 4. Procedure Preparation
    - 5. Hazard Review
  - B. Pre-Test Plant Clean Out
    - 1. Recovered Acid System
    - 2. Dissolver and E Cell Equipment
    - 3. Solvent Extraction Equipment
  - C. Processing Operations
    - 1. Coating Removal
    - 2. Thoria Dissolution and Denitration
    - 3. Protactinium Recovery and Load Out
    - 4. Uranium-233 Recovery and Load Out
    - 5. Thorium Recovery and Load Out
  - D. Post Thorium Clean Out and Turn-a-round
    - 1. Dissolver Clean Out
    - 2. Solvent Extraction Flushes
    - 3. Solvent Treatment
  - E. Associated Operations and Incidents
- V. REFERENCES
- VI. LIST OF FIGURES AND TABLES
- VII. ACKNOWLEDGEMENTS

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 4

### INTRODUCTION

Long-range planning for Hanford production is based on the efficient use of the Purex Plant as a multipurpose facility. One of the variety of products proposed is U-233, obtained from the irradiation of thorium oxide. Since there was a lack of sufficient data to completely define a processing run of thorium in the Purex Plant, a test run was proposed using five tons of thoria to better define general technology, develop an operable flowsheet, and demonstrate the flexibility of the various equipment systems.

To process such a small amount of feed and still produce a U-233 product with a usable specification, it is necessary to completely flush the plant of U-238 and alter the normal process routes so that the U-233 can be separated from thorium and both materials purified for shipment.

The operations and changes required for such a run were considered desirable on a test basis in order to obtain experience and information on the projected "campaign" mode of operation. Also, detailed evaluation of the efficiency of types and methods of flushing in all sections of the plant was considered necessary to aid future planning.

Combined, all of the information generated from such a test would enable a reasonable estimate of plant turn-a-round time to be made which would be invaluable in future planning and scheduling activities for the Purex Plant and Hanford.

~~SECRET~~  
**DECLASSIFIED**

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 5

SUMMARY

The Purex Plant Thorium Process Test was successfully completed February 10, 1965, after 25 days of processing. Three special products were loaded out: 2886 grams of U-233, 7660 pounds of thorium, and 4 grams of protactinium-233. The U-233 was 95.5% pure, which exceeded the expected purity of 90%.

Extensive flushing of the plant was required before and after the run to ensure product purity. A total of 450,000 gallons of solution was used to carry out 185 individual flushes described in 93 procedures. The presence of a small amount of uranium bearing siliceous deposits in the dissolver pot and auxiliary equipment extended the time estimated for flushing this system by approximately fifty-five days. The problem was resolved by replacing the dissolver pot and downdraft tower. Seventy days were devoted to flushing the head end section of the plant and 37 days were used for the solvent extraction section. Eight days of terminal flushing followed the test.

Thoria targets were charged to the dissolver on January 11, 1965, and the solvent extraction operation began January 25, 1965. Essential material costs for the test included \$35,800 for flushing and \$11,000 for processing with \$5,200 charged to solvent extraction processing. Six hundred and twenty thousand gallons of unconcentrated waste were generated by the run.

Since development flowsheets were used to process thorium and uranium-233, all existing operational and control specifications and procedures had to be reviewed

~~SECRET~~  
**DECLASSIFIED**



~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 6

SUMMARY (Con't)

and revised to ensure operating safety within the limits defined by Research and Engineering. Valuable process control experience was obtained from the run and associated procedure preparation. Considerable laboratory experience was also gained during the test with more than 15,000 analytical determinations being made on 4,000 samples.

An unusually heavy burden was imposed on the canyon crane operations in order to reroute, maintain and replace remote equipment and jumpers. A total of 230 jumper changes and nine major equipment lifts were necessary to complete the test.

The Thorium Test was completed with relatively few problems. Besides the detection of trapped solids in the dissolver off-gas system, the only other significant problem was the rapid decrease in the solvent quality during the thorium decontamination run.

CONCLUSIONS

The Thorium Process Test demonstrated that irradiated thoria fuel can be processed efficiently in the Purex Plant. All three products, U-233, thorium and protactinium were successfully recovered and loaded out. No serious problems were encountered that cannot be resolved by flowsheet, operating or equipment changes for future thorium processing. Except for the detection of solids in the dissolving equipment and the rapid solvent degradation during thorium processing, the various aspects of the test proceeded as originally predicted for the process test conditions.

~~SECRET~~  
**DECLASSIFIED**

CONCLUSIONS (Con't)

Estimates of plant turn-a-round time have been better defined as a result of evaluation of the equipment changes and flushing operations carried out for the thorium test. For future multipurpose operation, the following nominal turn-a-round estimates\* are made:

Plant turn-a-round from normal processing to thorium processing - 2 weeks.

Plant turn-a-round from thorium processing to normal processing - 1 week.

Plant turn-a-round from normal processing to E metal processing - 1 week.

Plant turn-a-round from E metal processing to normal processing - 1 week.

These values are based on average operating times for equipment changes and flushes. Where the crane operations are limiting, an average of eight remote jumpers can be installed per shift in a single canyon opening, or an average of four remote jumpers can be installed per shift in multiple canyon openings and the remote replacement of a major equipment piece averages 1 to 2 shifts after the jumpers are removed.

For empty-out operations, five days are required to purge the plant so that less than 150 units of each product are retained in either the head end or solvent extraction sections.

To further reduce the residual product hold up in the head end section to less than 50 grams, an estimate of eight days is required for the clean out of a standard Purex dissolver and four days for the new annular dissolver using a boiling 12M nitric acid, 0.025M fluoride, and 0.05M ANN solution. For tanks or equipment with efficient agitation such as in D and E cell, a maximum of four days is required for clean out.

\*Estimates can be very sensitive to product specifications.

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 8

CONCLUSIONS (Con't)

Complete clean out of the solvent extraction equipment to less than 50 grams can be accomplished with three cycles of flushes which can be completed in nine days. A cycle is composed of a flushing series of 20% nitric, 3% nitric acid, water, 5% caustic-5% tartrate, and water, which generates approximately 50,000 gallons of concentrated waste solutions per cycle for a total of 150,000 gallons for the clean out.

For future multipurpose operation, use of the canyon cranes will have to be phased directly into the processing sequence for canyon routing changes in addition to the normal crane function of remote maintenance and equipment replacement. Since processing steps will be dependent on crane work to this unusual degree, the ease of remote handling of canyon equipment should be included in the primary criteria for future design. Also, future equipment design should be reviewed to minimize traps or dead spots which could hold up product containing solution.

The successful application of closeup remote TV for the inspection of the bottom of the C3 dissolver proved its effectiveness as an inspection tool. The scrap detected on the bottom of the dissolver indicates that more positive control of metal transfers needs to be invoked when annular dissolvers are installed, to minimize the possibility of fuel jamming in the annular dissolver or sections of the bottom being plugged. In addition, such material is undesirable should a weight input measurement system be instituted in the Purex Plant.

~~SECRET~~  
**DECLASSIFIED**

DISCUSSION

Brief reports on the information gathered during the Thorium Process Test are presented to support the conclusions of the test.

Thorium Run Plans

The first document for a Purex Plant Thorium Process Test was issued in July, 1964. In the five months that followed, the plans for the plant scale process test took form.

Feasibility and Equipment Needs Thorium processing in the Purex Plant was judged to be feasible after a detailed study by the CPD Research and Engineering Operation. However, certain aspects of the flowsheet and the performance of plant scale equipment were not completely defined with the available data. To further evaluate the flowsheets and operational problems which might arise, a process test was approved using five to six tons of thorium.

Planning and Scheduling Preliminary flowsheets for the process test had to be defined early so that the following activities could commence:

1. Ordering remote canyon jumpers for new routings.
2. Revising critical mass control specifications to include Th and U-233.
3. Preparing flush specifications and procedures.
4. Issuing operating procedures.
5. Writing operational control specifications.
6. Developing analytical methods and sample schedules.

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 10

Thorium Run Plans (Con't)

When these items had been prepared in draft form, a Hazards Review was conducted by a CPD task force. Planning was, therefore, a joint effort of all CPD and other HAPO components, as several interrelated items were passed between groups until a satisfactory solution was found.

The original schedule called for the C3 dissolver to be removed from normal uranium processing in time to allow fifteen days for flushing prior to charging with the irradiated thoria target elements. This flush of the dissolver and protactinium recovery vessels was to remove residual normal uranium and plutonium. Dissolution and protactinium recovery would then take thirty-three days, during which time the normal uranium processing would be completed and the solvent extraction equipment stripped and flushed (see Figure II). The schedule allowed eleven days from the completion of the normal uranium run in which to flush the solvent extraction equipment and make the necessary routing changes preparatory to running thoria.

The total solvent extraction time, including partitioning, U-233 purification and thorium purification was scheduled to require twelve days.

Post-run flushes and restoration of plant equipment to normal was planned to take sixteen days.

This schedule was not met due to the difficulty in removing residual uranium and plutonium from the original dissolver and the necessity of developing equipment routings to avoid uranium contamination even after a new dissolver and repaired dissolver tower were installed. However, as can be seen from Figure II, once the

~~SECRET~~  
**DECLASSIFIED**

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 11

Thorium Run Plans (Con't)

thoria was charged the original schedule was either attained or surpassed in terms of elapsed time.

Routing and Equipment Changes The dissolution flowsheet, with centrifugation of the coating waste and protactinium recovery, required the installation of four new routes [dissolver (C3) to centrifuge feed tank (E3), centrifuge catch tank (E5) to coating waste tank (D2), centrifuge slurry tank (E1) to dissolver (C3) and centrifuge catch tank (E5) to concentrated backcycle waste tank (J1)] so that the installed centrifuges and storage vessels in E Cell could be used. A route from dissolver (C3) to the acid recovery system LWF tank (F12) (see Table I) was provided to transfer the reflux condensate from the dissolver off-gas tower to the acid recovery facilities. This route was designed to allow the reflux condensate to be directed either to the dissolver as is normal or to F12. A hold-up pot was built into the jumper connecting the dissolver off-gas knock-out pot and the dissolver. A jet built into the hold-up pot could be activated to direct the reflux condensate to F12. This route was intended for use only during the acid removal step but it was used during dissolution to avoid introducing contamination from the dissolver tower. A special monitor was installed on the dissolver coil to detect any coil failure resulting from the corrosive solutions used in the thorium dissolution. The off-gas monitors normally used to detect radioiodine emission from the C3 dissolver were specially calibrated to alarm should radoruthenium evolution occur and not be captured in the off-gas tower and filters.

~~SECRET~~  
**DECLASSIFIED**

Thorium Run Plans (Con't)

A shielded cask was provided and the fission product load-out facilities were used for the load-out of the protactinium recovery product.

The small amount of feed to be processed made it advisable to employ a 5% TBP solvent extraction flowsheet similar to ones previously developed at ORNL and SR for thorium - U-233 partitioning the U-233 purification in the Neptunium Recovery and Final Plutonium cycle equipment. However, the higher capacity flowsheet was also tested during the thorium purification run in the large solvent extraction equipment. The use of these two distinct flowsheets in the process test made extensive equipment and routing changes necessary (Table I).

The U-233 recovery required three routes -- solvent makeup tank (R1A) to 1BXF tank (J3), J3 to HAO tank (J2) and J2 to the No. 1 Solvent System (G Cell) header -- to modify the J2 and J3 tanks for makeup, storage, and feed of the 5% TBP solvent to the solvent extraction columns. Special routes were also required to transfer the thorium-bearing waste to storage vessels, J-22 aqueous waste (3AW) to 1WF tank (F12) and F12 to 1WW neutralization tank (F16). A route was required for the U-233 product from the partition cycle to the final uranium purification cycle, J-23 product (3BN) to 2AF tank (J5). The L Cell Package equipment (stripper T-L3, concentrator E-L4 and product receiver tank TK-L6), which was used to concentrate the U-233 product, was not geometrically favorable for all concentrations of U-233, so special safety circuits and instruments were provided as a backup for the batch size control system of critical mass control. Modifications were required in the Product Removal Room so that the U-233 load-out into the special M-102 "bird cage" carriers could be performed. The thorium purification run required canyon jumpers and cold side piping so that three non-routine

**DECLASSIFIED**

RL-SEP-352  
Page 13

Thorium Run Plans (Con't)

streams (HAX-HNO<sub>3</sub> to the HA (H2) column and HSIS-HNO<sub>3</sub> and HSS-Butt to the HS (H3) column) could be added to the solvent extraction columns. A special route, 2DF tank (K1) to concentrated backcycle waste or 3WB tank (J1), was provided so the thorium product could be "spun" back to the feed tank for additional solvent extraction treatment. Three jumpers were required to route the final thorium product to the load-out facilities (K1 to 203 load-out line). The product was loaded into a trailer and transported to the 200-W Area for storage in the specially re-activated WR vault.

Procedure Preparation A great deal of time and effort was spent in preparing specifications due to the magnitude and complexity of the Thorium Process Test. Separations Process Engineering with the assistance of Purex Process Control performed the bulk of this "paperwork." The specifications and procedures required the review and approval of several organizations to assure safe operation.

The desired product purity, coupled with the purity of the thoria target elements before processing, dictated the solvent extraction cleanup necessary and the amount of contamination from normal processing that could be allowed to remain within the processing equipment. When the processing flowsheets were finalized, the flushing and operating procedures were written.

A sample schedule was prepared by Separations Process Engineering, after which Purex Analytical Control, assisted by the Process Chemistry group developed and

**DECLASSIFIED**



~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 14

Thorium Run Plans (Con't)

adapted analytical procedures with capabilities for process control.

The processing of a fissionable isotope other than Pu-239 and natural uranium in the Purex Plant made revision of the critical mass control specifications necessary.

New essential material and chemical hazards specifications were required, since the Thorium Process Test used some chemicals not previously used at Purex, e.g., phosphoric acid, manganous nitrate and potassium fluoride.

Flushing requirements were particularly stringent to assure product purity for the relatively small amount of material to be processed. Forty-five separate flush procedures were written and used for the dissolver and related equipment before the vessels were judged clean enough to charge the target elements. In many cases, new routings had to be provided so the flushing could proceed with a minimum of time and material expended.

A typical listing of the magnitude and variety of specifications and procedures follows:

~~SECRET~~  
**DECLASSIFIED**

~~SECRET~~  
**DECLASSIFIED**

A. Specifications

1. Product Specifications
2. Flushing Specifications
3. Feed Specifications
4. Essential Material Specifications
5. Critical Mass Control Specifications
6. Chemical Hazards Control Specifications
7. Chemical Flowsheet Specifications
8. Operational Control Specifications
9. Sample Schedule

B. Procedures

1. Flushing Procedures
  - a. Dissolver and Pa Recovery Equipment
  - b. Partitioning and U-233 Purification Equipment
  - c. U-233 Concentration and Load-out Facilities
  - d. Thorium Purification Equipment
  - e. Thorium Load-out Facilities
  - f. Miscellaneous Routings
  - g. Post Thorium Run Flushes of All the Above Equipment
2. Operating Procedures
  - a. Use of  $UO_3$  Acid During Thorium Run
  - b. Aqueous Makeup - Flushes and Processing
  - c. Coating Removal and Centrifugation
  - d. Thoria Dissolution and Denitration

~~SECRET~~  
**DECLASSIFIED**

Thorium Run Plans (Con't)

- e. Protactinium Recovery and Solvent Extraction Feed Adjustment
- f. Solvent Extraction - U-233
- g. U-233 Load-out
- h. Solvent Extraction - Thorium
- i. Thorium Load-out
- j. Solvent Treatment
- k. Waste Concentration and Nitric Acid Recovery

Hazards Review A team was appointed from within CPD to review the plans for processing the thoria in order to evaluate the hazards involved in the proposed methods of operation.

The task force concluded that the greatest areas for concern were in avoiding radio-ruthenium volatilization with possible environmental contamination and critical mass control, especially in the non-geometrically safe U-233 concentration equipment.

Special procedures and controls were deemed adequate for the original test run but the processing of large quantities of thoria would require redesign of some systems and reevaluation of the hazards involved.

For further details, see Reference 12.

Pre-Test Plant Clean Out

The target isotopic purity of the U-233 was set at 90%. Because the total U-233 to

**DECLASSIFIED**

RL-SEP-352

Page 17

Pre-Test Plant Clean Out (Con't)

be processed was so small (~3000 grams) the estimated allowable U-238 to be left in the plant equipment was 150 grams. The Plant was divided into three sections for flushing purposes, the recovered acid system, the dissolver and E Cell equipment, and the solvent extraction system. The target for each of the sections was a retention of 50 grams of U-238. These goals were met, as the isotopic purity of the U-233 product was 95.5% (see flush summary below).

Recovered Acid System Flushing of the recovered acid system was relatively easy. The source of uranium in the recovered acid ( $UO_3$  recovered acid at 0.07 pounds uranium per gallon of acid) was valved off on October 22. Dilution by process turnover reduced the Purex recovered acid system uranium content to between one and four pounds of uranium per million gallons by November 18. This was well within the specification for use in the thorium processing.

$UO_3$  recovered acid was used during the month and a half of normal processing after the above line was blanked, because neither Purex nor the  $UO_3$  Plant has capacity to store the amount of acid generated with the uranium product (0.5 pounds  $HNO_3$  per pound of U). Purex used the  $UO_3$  acid directly in the dissolvers after a program was started to insure a uniform acidity. The  $UO_3$  Plant set up special procedures to assure a constant  $UO_3$  recovered acid concentration and shipped only from a tank which was known to meet a specification of 49 to 52.5%  $HNO_3$ . As a double check, the  $UO_3$  recovered acid was sampled and checked at Purex before use. The acid used in the dissolver must be within the limits of 49 to 52.5 percent to make sure the dissolving solution will not become acid deficient under normal operating control limits.

**DECLASSIFIED**

**DECLASSIFIED**

RL-SEP-352  
Page 18

Pre-Test Plant Clean Out (Con't)

Dissolver and E Cell Equipment Dissolver C3 was chosen as the dissolver to be used for thoria dissolving. The last charge of uranium was made on 4 - 12 shift, October 31. Following two normal cuts, the heel was removed by two full cuts and a heel cut. The actual flushing started on day shift November 3, when 1600 gallons of 57 percent nitric acid were added to a 400-gallon heel cut. This flush, No. 1 on Table III, was boiled for 24 hours under reflux. The flush removed 26 pounds of uranium, indicating that the heel cut procedure does a good job of removing the readily-dissolvable uranium. To keep from diluting the dissolved feed in the feed storage tank (D4), the acid from C3 was jetted to dissolver B3 by special jumpers at the dissolver rinse tank (D1). The acid was used in B3 for a normal dissolution. Two more 57 percent acid flushes were made on C3 until it was found that the heel had been sufficiently removed so that acid flush solutions could be sent to the acid recovery system without excessive loss of products. A route was then established to the acid recovery system feed tank, LWF tank (F12).

Operation of E Cell for fission product recovery was suspended on November 12. The first flush sent from dissolver (C3) to centrifuge feed tank (E3) to start flushing the E Cell equipment was flush No. 4, a 20 percent  $\text{HNO}_3$  flush (see Table III). The flushing of E Cell continued with the dissolver flushing until the task was completed. The head end flushes are summarised in Table II and shown individually in Table III. Until the rest of the plant was shut down, all acid flushes went to the LWF tank (F12) and about 75 percent of the acid was recovered.

**DECLASSIFIED**

Pre-Test Plant Clean-Out (Con't)

After 27 flushes totaling 64,630 gallons, it was found that the amount of uranium removed from dissolver C3 had leveled off at a value greater than 10 grams per hour, too great to accept as a final clean-out. At this point, the flushing conditions were intensified, the acid - ANN/fluoride (2:1) solutions were boiled and in some cases the fluoride concentrations were raised. The intensified flushes took three general courses: high nitric (57%) low fluoride (.025 M) with 2:1 ANN at boiling; low nitric (28%) medium fluoride (.5 M) with 2:1 ANN added after heating at 80° C; and low nitric (20%), high fluoride (1.0 M) with 2:1 ANN added after heating at 90° C. Silica-base solids were suspected to be causing the high dissolution rates because the plutonium to uranium ratio was about four times the normal ratio. It was assumed that the dissolved iron in the flush solutions came from the dissolver coils because the coil surface was at a higher temperature than other surfaces. The average corrosion rate for mild flushes was 8.5 mils per month. For more intense flushes the rate was 15 mils per month. At the maximum concentration of 2.0 M fluoride the corrosion rate was 70 mils per month for the four-hour flush period (ANN was added after the four-hour heating period on a 2:1 mole ratio). An estimated 3.5 mils were removed from the coil of the original dissolver. The original wall thickness of the coil was 150 mils.

On December 17, a TV inspection of the dissolver pot showed the dissolver was extremely clean of product solids except for miscellaneous scrap from a broken charging bucket and 100 Area experimental equipment (See Photos 1 and 2). At this point, a new dissolver was installed on the basis of the uncertainty of the dissolver content. The new dissolver was in place on December 21.

**DECLASSIFIED**

RL-SEP-352  
Page 20

Pre-Test Plant Clean-Out (Con't)

After the new dissolver was installed, flushes intended to remove welding and fabricating debris showed small amounts of uranium in each flush (See Table III). It appeared to be coming from the dissolver tower via the reflux return line and possibly from the expansion bellows in the vapor line from the dissolver to the tower. Seventeen flushes of the new dissolver and old tower removed about eight pounds of uranium. The Pu/U ratio of the product removed from the tower was still in the range of 2200 grams/ton. The rate of dissolution was still too high, at 10 to 100 grams per hour, so the tower was removed on January 3.

A 10-inch pipe jumper was fabricated to by-pass the tower knock-out pot and ammonia scrubber. Three boilups with flush solutions were made. The uranium dissolution rate decreased but ruthenium contamination appeared to be a problem. The off-gas monitors which had been modified to measure Ru-103 and -106, alarmed during all of the boilups. The vapor-handling capacity of the off-gas system was also a problem. A dissolver tower which had been removed from dissolver B3, in February, 1964, was repaired in T Plant on an accelerated work schedule and installed on C3. The tower had been scheduled to be used when the new annular dissolvers were installed. An estimated 3 to 4 weeks was required to repair the leaking tubes and mock it up on a normal schedule; however by working around the clock on an "all-out" schedule, it was completed in four days. After installation, this tower was also found to have some uranium contamination. The uranium/plutonium ratio was the same as the original tower. It was decided to install a plug-blank in the reflux return line and jet all condensed reflux to the LWF tank (F12) through a jumper route already installed for use during acid removal.

**DECLASSIFIED**

Pre-Test Plant Clean Out (Con't)

When the plant completed normal processing in early December, all the flushing was routed from E Cell to coating waste tank (D2). After neutralizing, this material was jetted to non-boiling waste storage.

Flushing of E Cell equipment was continued in conjunction with dissolver flushing. All the flushes were sent through E Cell, whichever route of disposal was taken: to acid recovery system, 1WF tank (F12) or to coating removal waste tank (D2). G-E4, a B Plant-type centrifuge, was new and unused but was included in the flush schedule as a backup for centrifuge G-E2. This proved to be fortunate as the diverter valve on G-E2 failed during the last portion of the protactinium run and G-E4 was used to complete the run.

Solvent Extraction Equipment The solvent extraction system shutdown was started on December 5. Cold feed was processed to remove gamma activity from the system so the final stripping would not produce a gamma burst. The cold feed also displaced plutonium from the system so the backcycle system contents could be reduced in volume after shutdown and stored in the HAF tank (H1).

The final strip and displacement of columns was the normal procedure used for an inventory clean out. After the plant was shut down, a 10 percent nitric acid and water flush (pre-flush) was put through all solvent extraction systems which were to be used in the thorium - U-233 run (See Figure III and Table IV). This flush was intended to remove the gross quantities of uranium, plutonium, and neptunium so that subsequent decontamination flushes could be thrown away. The pre-flush was collected in 3WF tank (F10) and boiled down batchwise in the backcycle concentrator (H4). A



**DECLASSIFIED**

RL-SEP-352

Page 22

Pre-Test Plant Clean Out (Con't)

sample of each batch was used to monitor product content. This pre-flush solution was combined with the backcycle waste, the complete batch was reduced in volume by boiling off nitric acid, then stored in HAF tank (H1). The pre-flush product content was very low at 52 pounds uranium and 136 grams plutonium, indicating the normal shutdown clean-out is very effective. The flushing with 10 percent nitric acid by cascading forward consumed a large volume of flush and was slow because the routes available were limited by the flush requirements and other shutdown activities. For instance, early flushes from the J Cell Package could not be routed through L Cell because their high uranium concentration would unnecessarily contaminate the uranium-free equipment. Therefore, L Cell and J Cell Package were flushed separately requiring extra coordination to schedule both flushes through the 2AF tank (J5). Another delaying circumstance was that the LBX column (J6) empty-out route shared a nozzle with the 2DF tank (K1) to 3WF tank (F10) route, and therefore, required extra coordination to schedule the right route to be in place.

The decontamination flushes following the pre-flush consisted of a series: 20 percent nitric acid, water, 10 percent caustic-2 percent tartaric acid, water, 30 percent nitric acid-5 percent oxalic acid. Two cycles of these flushes were generally used. A summary of the flushes is shown on Table IV. The total product removed by the decontamination flushes was 48 pounds of uranium, 31 grams plutonium and 60 grams of neptunium. The points of greatest hold up and resistance to flushing were found to be as predicted: 3AF tank (J21), the ICU concentrator (EJ8), and the 2DF tank (K1). Additional flushing was required to reduce these vessels to desired uranium limits.

~~SECRET~~  
**DECLASSIFIED**

Pre-Test Plant Clean Out (Con't)

The following flush summary outlines the flush limits by processing groups and compares the limits with actual results:

PRE-THORIUM FLUSHING SUMMARY

First Cycle System

Limits

0.0012 grams plutonium per gallon in J6, J4, J5

0.0001 pounds uranium per gallon in J1, H2, H3, J3, J6, J7, J8, K1

Level Attained

0.0007 grams plutonium per gallon in J6, J4, J5

0.00004 pounds uranium per gallon in J1 through K1

Material Removed

Plutonium: 10 grams, Uranium: 20 pounds

Neptunium Recovery System and Second Plutonium System

Limits

Less than 60 grams uranium in J21, J22 and J23

Less than 100 grams plutonium in J21, J22, J23 J5, L1 and L2

Level Attained

Approximately 40 grams uranium in J21, J22 and J23

Less than 1 gram plutonium in J21 through L2

Material Removed

Plutonium: less than 25 grams, Uranium: 20 pounds

Neptunium: 25 grams.

Pre-Test Plant Clean Out (Con't)

L Cell Package and Product Removal Vessels (L3, L4, L6, L9, L13)

Limits

Less than 0.12 grams plutonium per gallon

Level Attained

0.075 to 0.12 grams plutonium per gallon

Material Removed

Approximately 50 grams plutonium

Processing Operation

The experience of processing thoria in the Purex Plant was entirely new, and as such it was given close attention in order to gain the maximum value from the process test.

Coating Removal On January 11, 1965, 9489 pounds of thoria were charged to the dissolver. The physical equipment comprising the Purex dissolving system was in its normal configuration with the exception of some jet routes.

The thoria was charged in the normal manner and the aluminum jackets were removed following the Standard Operating Procedures. Before removing the coating waste, the dissolver was digested for six hours with sparging to maximize removal of aluminum which may have been covered or coated with the thoria fines. The off-gas from the coating removal step was scrubbed for ammonia removal and routed to the stack, bypassing the backup facility.

The coating waste solution plus two 1000-gallon rinses were transferred to E Cell

Processing Operation (Con't)

for recovery of entrained thoria. In the E Cell the coating waste and rinses were centrifuged and the resulting cake washed with caustic to remove the aluminum solids. Then the cake was slurried out of the centrifuge bowl to slurry tank E1 where it was digested in a nitric acid, fluoride, ANN solution to dissolve the thoria. Samples of this solution indicated a pickup of two percent of the thorium charge. It was planned to recycle this solution to the dissolver, but because of fifty percent contamination of the U-233 with U-238, the solution was discarded to underground storage.

Before the thoria dissolution was begun, the acid reflux recycle route to the dissolver from the dissolver tower and knock-out pot was blanked. This was done to prevent the residual U-238, which was trapped in the tower and bellows connectors, from contaminating the dissolver cuts.

Thoria Dissolution and Denitration Dissolving was started on January 12, 1965, using 12.2M HNO<sub>3</sub>, 0.025M KF and 0.05M ANN solution. The off-gas from the dissolving operation was handled in the normal manner. The reflux from the tower and knock-out pot was continually jetted to the LWF tank (F12) for acid recovery. Batch additions of fresh 57% HNO<sub>3</sub> acid were made to the dissolver to maintain a volume of 1700 to 2200 gallons.

The dissolution rate of the thorium was monitored by observing the buildup of the dissolution solution specific gravity. The first cut was cooled for sampling after 25 hours of boiling. At this time, a new sampler jumper was installed in the

Processing Operation (Con't)

dissolver. The jumper dip leg was shortened by seven inches to allow sampling of solution without plugging the intake leg.

The dissolver solution was then heated back to boiling and acid boil-off begun. This was accomplished by adding 500 gallons of water, concentrating to 1700 gallons and repeating this for a total of 21 hours. The first cut was cooled, allowed to settle with minimum air on the sparger for one hour and then jetted to centrifuge feed tank (E3). The total time required to dissolve and adjust the acid for this cut was 55 hours. At the end of the first cut a new thermohm, 13 inches off the bottom of the tank, was installed to give better control of solution temperature. The second cut was begun immediately following the completion of crane work and continued until January 17, 1965. At this time acid adjustment began. Samples of the first cut indicated a  $\text{HNO}_3/\text{Th}$  ratio of 1.95 rather than the desired 1.5 so the first cut was brought back to the dissolver and both the first and second cuts were acid adjusted simultaneously. The feed acid adjustment procedure was altered by using steam through the sparger lines and in the dissolver heating coil instead of through the coil alone. With this additional heating capacity and with the combined volume of two cuts, the thorium concentration was increased more easily without compromising the minimum volume limitation. In approximately 24 hours, the acid adjustment of the combined cuts was completed and they were moved to Tank E3. Then a third cut was begun. The third cut continued for a total boiling time of 47 hours ending January 21. The fourth cut continued for a total boiling time of 35 hours ending January 23. The fifth cut continued for a total boiling time of  $36\frac{1}{2}$  hours ending January 24 (see Figure I and Table V).

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 27

Processing Operation (Con't)

Starting January 25, cuts 3, 4, and 5 were combined in C3 dissolver and acid adjusted in the same manner as cuts 1 and 2. At this time jumpers were changed to route the reflux from the tower and knock-out pot to 1WW tank (F7) rather than the 1WF tank (F12) to avoid diluting the 3AF from the U-233 run. Acid adjustment was completed on January 26. The combination of cuts 3, 4, and 5 was transferred to E Cell and C dissolver was held pending return of routings available to dispose of final flushing. The complete cleanout of the dissolver is described in the following section.

Protactinium Recovery and Loadout - Following acid boil-off from the combination of the first and second cuts, the dissolver solution was transferred to E Cell for protactinium recovery. The protactinium run began January 19 and continued until January 21, for a total of 47 hours of processing time.

The protactinium run began by adjusting the dissolver feed solution to 1.0 M  $\text{HNO}_3$  and 0.1 M sulfamic acid. Then manganous nitrate and potassium permanganate were added to the solution to form manganese dioxide to scavenge the protactinium 233. All butt solutions were added to centrifuge catch tank (E5) and the cake carrying the Pa-233 was slurried to slurry tank (E1). In Tank E1 the cake was dissolved in a nitric acid sugar solution and the resulting solution was recentrifuged to remove the highly radioactive barium sulfate. During centrifuging the diverter, which directs the centrifugate to either Tank E3 or Tank E5, malfunctioned, allowing approximately 50 percent of the Pa solution to mix with the solvent extraction feed in Tank E5.

A second  $\text{MnO}_2$  strike was made to remove the thorium and U-233 from the remaining Pa-233. For this strike Centrifuge G-E4 was used at 1750 rpm. The final product

~~SECRET~~  
**DECLASSIFIED**

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 28

Processing Operation (Con't)

was then loaded out into a 400 gallon cask in the Fission Product Loadout Building (Table VI). Radiation levels during loadout were 50 mr/hr exposure to personnel. No difficulty was encountered in the loadout.

A second protactinium run was made on the accumulated thorium purification cycle waste (HAW). The volume of this waste was 6000 gallons, three times greater than in the first Pa strike. Pre-strike samples showed 28 units of protactinium were available but after the strike less than ten units could be detected in the slurry catch tank (E1). Besides this discrepancy in Pa units, the feed scavenging step concentrated the fission products in the precipitate, making the probability of meeting the product contamination specifications remote. So, rather than chance contaminating the first strike product, the second strike was discarded to boiling waste storage.

Uranium-233 Recovery and Loadout - The U-233 recovery portion of the thorium run utilized the following equipment and routes: Centrifuge catch tank (E5) to 3WB tank (J1) to 3AF tank (J21) to 3A column (J22) to 3B column (J23) to 2AF tank (J5) to 2A column (L1) to 2B column (L2) to 2BP stripper (L3) to 2BP concentrator (L4) to product receiver tank (L6) to sampler tank (L9) to loadout (equipment nomenclature joined by to indicates a route prepared especially for this run).

The feed solution was adjusted to -0.2 pounds/gallon  $\text{HNO}_3$  (acid deficient) in 3WB tank (J1) (this signifies that the solution lacks two tenths pound of nitric acid per gallon of solution from being in stoichiometric balance). On January 25,

~~SECRET~~  
**DECLASSIFIED**

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 29

Processing Operation (Con't)

J Cell Package and L Cell were started up. In the 3A column (J22), the feed was contacted with a five percent solution of TBP in diluent, the 3AX extractant stream. The scrub stream, 3AS, contained iron, ANN, and phosphoric acid. The iron (ferrous ion) was to insure U/Pu decontamination. The ANN was for salting the U and the phosphoric acid to insure U/protactinium decontamination.

In the 3A (J22) column the U-233 favored the organic phase and the thorium remained in the aqueous. The aqueous waste (3AT) containing the thorium was collected in F Cell (Tank F12 and Tank F16). The U-233 was stripped from the organic with 0.05 M  $\text{HNO}_3$  in the 3B (J23) column. The aqueous was then continuously jetted via the 3BN jet system to the 2AF (J5) tank. The stripped organic was returned to the organic storage tank J3.

The U-233 in tank J5 was continuously butted with  $\text{HNO}_3$  and  $\text{Fe}^{++}$  and contacted in the 2A (L1) column with 5 percent TBP, stripped in the 2B (L2) column with 0.01 M  $\text{HNO}_3$  and concentrated in the L Cell Package. Little difficulty was encountered with equipment operation (see Figure IV).

With the critical mass limit placed on the U-233 content in the L Cell Package (1300 units maximum), it was necessary to control the buildup of product in the "package" by specific gravity instrumentation and material balance calculations. There were instances of conflicting information between the indicated and calculated accumulation. During one "package" empty-out, the steam jet malfunctioned, causing concern for the mass limit. Because of these circumstances, the product

~~SECRET~~  
**DECLASSIFIED**



~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 30

Processing Operation (Con't)

was transferred to the load-out tanks before it reached the desired concentration, never exceeding sixty-nine percent of the limit. The first batch from the L Cell Package was moved to the sampling tank (L9) on January 26, after 20 hours of accumulation, at a concentration of 13.6 g/l. This transfer and subsequent transfers were accomplished by cutting off the stripping stream (2BX) and product stream (2BP) to the 2B (L2) column and moving the 2BP stripper (L3) and concentrator (L4) contents to the product receiver tank (L6) and then recharging L3 and L4 with fresh 1 M HNO<sub>3</sub>. The L3 and L4 contents were then brought to boiling and the 2BX and 2BP returned to normal rates.

The first and second load-out batches were very dilute (see Table VII) because of extra caution used in the critical limit control point. For these two batches, 2BP flow was used for control (see Sketch I). The analyses necessary to operate solvent extraction and control critical mass were often delayed. This was due to the overload of analyses requested from the Laboratory with routines, re-runs and re-samples, and the length of time required per sample, such as 105 minutes for thorium by the ion exchange method.

The second U-233 batch was transferred to sample tank (L9) at 0030 on January 27 (after 10 hours of collection) at 10.8 g/l total U.

The third U-233 batch was transferred to Tank L9 at 1330 on January 27 (after 13 hours of accumulation) at a concentration of 31.7 g/l total U.

~~SECRET~~  
**DECLASSIFIED**

Processing Operation (Con't)

The fourth U-233 batch was transferred to Tank L6 at end of stripping L Cell after shutting down the solvent extraction system. Its concentration was 30 g/l. The product was loaded out via load-out tank L13 into one-gallon plastic jugs. A total of 48 jugs were loaded out, including the four product batches and 1½ flush batches. The jugs were contained in M-102 "bird cage" containers.

Final flushes containing  $\leq 5$  g/l U-233 and  $\sim 0.04$  g/l Pu were loaded into 5-gallon plastic jugs for storage.

Thorium Recovery and Loadout - Following the U-233 recovery run, the 5 percent TBP solution in Tanks J2 and J3 were butted to 30 percent TBP. The organic header system was restored to normal the LBXF jumper installed. The organic in J2 and J3 was transferred to the No. 1 organic treatment system (G Cell) through the LBX (J6) column and 1C (J7) column. G Cell was "spun" to clean up the solvent.

From January 27, the end of the U-233 run, until January 29, the beginning of the thorium run, routings were restored to conditions necessary for thorium processing; eleven jumpers were involved. Also, during this period, the feed for the thorium decontamination run was transferred from F Cell to E Cell from LWF tank (F12) to LWW neutralization tank (F16) to centrifuge feed tank (E3) and through the centrifuge to remove any traces of solids picked up from the F Cell vessels, then adjusted to feed specifications in the centrifuge catch tank (E5) and the 3WB tank (J1). During this period, a leak was discovered in the left hand removable tube bundle of the LWW concentrator (E-F6). The tube bundle was not changed at this time, but merely isolated, requiring eight hours of crane time.

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352  
Page 32

Processing Operation (Con't)

Difficulty was encountered in the makeup and analysis of HSS butt solution. This solution was initially planned to be 1.76 M Fe<sup>++</sup> and 1.75 M PO<sub>4</sub>-3. Due to the low solubility of the PO<sub>4</sub>-3, the makeup was changed to one-half strength.

The thorium run utilized the following equipment and routes: 3WB tank (J1) to HA column (H2) to HS column (H3) to LBXF tank (J3) to LBX column (J6) to LC column (J7) where partitioning took place. The thorium went to the LCU concentrator (J8) to 2DF tank (K1) to 3WB tank (J1) for Phase II, and to 203-A loading station for load-out to a trailer. (Equipment nomenclature joined by to indicates a route prepared especially for this run.)

The HA-HS column battery was used for decontamination from fission products. These columns were operated as normal HA-HS columns except that an additional scrub stream, the HSIS (fresh HNO<sub>3</sub>) was added to the HSIS distributor and (recovered HNO<sub>3</sub>) -HAX-HNO<sub>3</sub> was added to the normal HA nitrite distributor at the bottom of the HA column. The thorium and U-233 in the organic phase overflowed the HS column to the LBXF tank (J3), was pumped through the LBX column, and the thorium was partitioned by stripping out of the organic in the LC column. The thorium was then concentrated in the LCU (J8) concentrator which overflowed to the 2DF (K1) tank. Trace amounts of uranium remained with the organic and flowed to G Cell (see Figure V). The initial throughput of the acid-deficient thorium feed began on January 29, at 1530 and ended January 30, on 4-12 shift. It was difficult to establish equilibrium operating conditions because of the erratic feed control in the 3WB system and the small amount of feed. Higher-than-expected losses were experienced in the HAW stream.

~~SECRET~~  
**DECLASSIFIED**

**DECLASSIFIED**

RL-SEP-352  
Page 33

Processing Operation (Con't)

Phase II of the thorium decontamination run consisted of recycling the thorium product from the 2DF (K1) tank to the 3WB tank (J1) and running this as HAF. This phase began on January 30, on 4-12 shift. The difference between Phase I and Phase II was that the HAF was acidic in Phase II. After approximately 25 hours of running on Phase II, the HA column (H2) pulser failed. The process was shut down and the HA column contents displaced and the pulser changed out. During shutting down, replacing the pulser and starting up there was considerable loss of thorium. This loss was both to the cell sumps due to equipment maintenance and to the HAW waste due to the upsetting of the process by stopping and starting with such a small volume of feed. The 1CU (J8) concentrator was emptied to the 2DF (K1) tank and the 1WW (E-F6) concentrator was emptied to 1WW tank (F7).

While shut down, it was decided to switch back to an acid deficient flow sheet for startup, in an effort to obtain better decontamination of the thorium. The process was restarted up on February 2, on 8-4 shift after 64 hour shutdown to replace the HA pulser. Also, the flow control problem on the HAF (3WB) system was corrected by blanking off the pump recycle line. This was a prototype pump recycle jumper with a magnetic flowmeter. The run was terminated on February 4, on 4-12 shift after approximately 50 hours of Phase II operation for a total operation time Phase I and II of 98 hours.

At shutdown, the columns were stripped. The total thorium load was accumulated in the 1CU (J8) concentrator and then transferred as one 2000-gallon batch to the 2DF (K1) tank. The concentrator was then rinsed with one water flush to tank K1

**DECLASSIFIED**

Processing Operation (Con't)

ending up with a total volume of 2500 gallons. This material was then loaded out to a tank truck via the 203-A loading station and transferred to 200-W Area for storage in the WR vault. Five subsequent flushes of the 1CU (J8) concentrator and 2DF (K1) tank were also loaded out to a truck and moved to 200-W. A total of 7600 gallons were loaded out (see Table VIII).

Near the end of the thorium recovery cycle the No. 1 Solvent System quality dropped to well below normal. The organic Pu retention (measure of quality) increased up to 100 times its normal value (see Figure VI): After attempting to clean up the solvent by recycling through G Cell, with no apparent improvement, G Cell was shut down and the vessels flushed. Solid manganese dioxide was found in samples throughout the system, indicating that the acid recycle rate was not great enough to kill the entrained solids. All of G Cell was flushed with oxalic-nitric solution to dissolve the manganese dioxide. This procedure required five days.

Post-Thorium Cleanout and Turn-Around

The specifications for thorium and uranium-233 impurity in normal uranium and plutonium products required that the dissolver and the solvent extraction equipment be thoroughly flushed (see flush summary below).

Dissolver Cleanout - A series of dissolver flushes were made immediately following the four product cuts and the fifth heel cut (see Table V). Number 6 and 7 (flushes) utilized nitric acid-fluoride solution identical to that used during earlier cuts.

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352

Page 35

Post Thorium Clean Out and Turn-A-Round (Con't)

Flushes 8, 9 and 10 comprised a sequence of 57% nitric acid flushes, the last of which digested for approximately 50 hours. A final water rinse followed the above flushes. Normal dissolving operations began on February 5.

Total material removed from the dissolver amounted to approximately 250 pounds of thorium and 100 grams U-233.

Solvent Extraction Flushes In general, flushing of the extraction equipment required much less time following the run than before the run. This was primarily due to the thoroughness of the initial flushes, the relatively short period between flushes, and the higher limit of contamination allowed after the run.

The flushing goals and actual results are presented in the flush summary below. Product levels were calculated from vessel volumes and sample data taken during flushing. It is important to note that the absolute quantity of product in a given piece of equipment could be more than the figures given. The product levels given are derived from a sizeable tabulation of sample data and are considered to be the best available estimates (see Table IX).

First cycle columns HA (H2) and HS (H3) were flushed separately from the remainder of the first cycle equipment, to avoid fission product contamination. Earlier equipment flushing results indicated that fission products were retained longer by the columns than the product materials. The product levels attained in columns H2 and H3 after three 10% nitric acid flushes were low enough to begin normal uranium processing.

~~SECRET~~  
**DECLASSIFIED**

Post Thorium Clean Out and Turn-A-Round (Con't)

First cycle equipment, columns 1BX (J6) and 1BS (J4), the 1BXF tank (J3) and 2AF tank (J5), was given a separate flush. Samples taken in J5 (the last vessel in the flow scheme of the flush) reached low product levels rapidly. First cycle vessels, column 1C (J7), 1CU concentrator (J8) and 2DF tank (K1), were also flushed as a unit. U-233 levels in the terminal vessel of this flush were very low (see the flush summary below).

Five 10% nitric flushes were applied to the J Cell Package neptunium equipment. Sample data taken during the flush indicated a greater than normal product holdup in the J Cell Package feed tank. Product levels attained are shown in the summary below.

The second cycle plutonium equipment was decontaminated quite thoroughly after four 10% nitric flushes. Product levels dropped to less than one gram of U-233 and less than 13 grams of thorium per thousand gallons.

The plutonium stripper-concentrator pair (L3 and L4) was extensively flushed along with product removal equipment to insure a low degree of contamination from U-233 and thorium.

POST THORIUM FLUSHING SUMMARY  
SOLVENT EXTRACTION

First Decontamination Cycle (T-H2 through E-J8)

Limits

Less than 20 grams thorium in J3 and J6

~~SECRET~~  
**DECLASSIFIED**

RL-SEP-352

Page 37

Post Thorium Clean Out and Turn-A-Round (Con't)

Less than 300 grams thorium in H2, H3, J7, J8

Less than 20 grams U-233 in H2, H3, J3, J6, J7, J8

Level Attained

Approximately 130 grams thorium in H2 and H3 after flushing

Approximately 250 grams thorium in H2 through J8 during cold operability run

Less than 20 grams U-233 in H2, H3, J3, J6, J7, J8

Material Removed

Thorium: 14 pounds, U-233: less than 5 grams

J Cell Package - Neptunium Recovery Equipment

Limits

Less than 300 grams thorium

Less than 20 grams U-233

Level Attained

Approximately 500 grams thorium

Approximately 1 gram U-233

Material Removed

Thorium: 1 pound, U-233: 5 grams

Second Cycle Plutonium System

Limits

Less than 10 grams thorium

Less than 10 grams U-233

Level Attained

Less than 1 gram thorium in J5, L1, L2, L3, L4

Less than 1 gram U-233 in J5, L1, L2, L3, L4

~~SECRET~~  
**DECLASSIFIED**



**DECLASSIFIED**

RL-SEP-352

Page 38

Post Thorium Clean Out and Turn-A-Round (Con't)

Material Removed

Thorium: Less than 25 grams, U-233: Less than 2 grams (J5, L1, L2)  
75-100 grams (L3, L4)

Solvent Treatment The No. 1 Solvent System furnished solvent for the first cycle thorium decontamination. The solvent and solvent equipment were essentially unchanged for the thorium decontamination phase of the run.

Analytical data relating solvent quality were obtained from the usual solvent plutonium retention analysis (Figure VI). It was noted during the thorium decontamination run that the solvent quality decreased (high plutonium retention) during the time when acid deficient thorium feed was used and increased during the time when acid feed was used. Examination of the system indicated an entrainment problem in the solvent purification equipment. A solid formed and normally confined to the solvent system feed tank (G1), was found to be distributed throughout all of the solvent system equipment.

At the end of the thorium run, the No. 1 Solvent System (G cell) equipment was flushed with 18,500 gallons of 5% oxalic acid, 3% nitric acid flush solution in seven flush throughputs. The oxalic-nitric flush dissolved the manganese dioxide solids. The system was returned to spinning status and the solvent was found not to be degraded.

**DECLASSIFIED**

**DECLASSIFIED**

RL-SEP-352  
Page 39

Associated Operations and Incidents

Canyon equipment changes during the thorium test run required concentrated activity by the canyon cranes to meet the processing schedule (Figure I). Nine major lifts of dissolver equipment were required during replacement of the C3 dissolver and tower in preparation for thoria charging. Some twenty-six new jumpers were installed, which in turn required removal of fifty-three existing jumpers.

Twelve temporary flex routings were installed during the run, for flushing and waste processing. Equipment failures such as concentrator tube bundle leaks, and HA column (H2) pulser failure, contributed emergency crane work and some delay in processing.

Timely maintenance on the cranes made it possible at times to utilize both cranes effectively, doing cell work at opposite ends of the canyon. The most critical and closely scheduled period for crane work was in early February when the test run was completed and it was necessary to restore the plant for normal processing. Three shifts of the jumper work, needed to restore fission product processing capability, were delayed until after startup to make time for higher priority work and are not shown on the schedule (Figure VII). In general, the jobs completed which were not directly related to the test run are typical of a Purex shutdown although the number of equipment failures was above average. Three concentrator tube bundles failed and were replaced, in addition to the HA column pulser, LWF pump and a dozen failed jumpers.

**DECLASSIFIED**

**DECLASSIFIED**

RL-SEP-352  
Page 40

Associated Operations and Incidents (Con't)

Efforts were successful in removing failed canyon equipment such as the F6 concentrator, F15 tank and assorted failed jumpers during the period. This is normal canyon activity during a shutdown.

While not apparent from the schedule, the crane work performed in support of the test run exceeded reasonable expectations in every phase of the run. The skill and experience of the crane operators was one of the intangible but very heavy plus factors in successful completion of the run.

During the dissolver flushing stage of the test, it became necessary to view the inside of the dissolver. This was undertaken with a remote television camera in a stainless steel carrier which was lowered into the dissolver to determine if undissolved material remained. By remote cable connection, the picture was relayed to the Operating Gallery for general viewing (see Photos I and II). Some undissolved material was in evidence under the grating. An unexpected collection of stainless steel scrap, observed in the bottom of the dissolver, was presumed to have collected over the years from foreign material in slug buckets dumped during charging.

**DECLASSIFIED**

DECLASSIFIED

RL-SEP-352  
Page 41

THIS PAGE DELETED

DECLASSIFIED

LIST OF FIGURES AND TABLES

Figure I Composite Operating Schedule  
Figure II Original Operating Forecast vs. Actual Performance  
Figure III Solvent Extraction Flush Details  
Figure IV Sample Analysis of U-233 Product Streams  
Figure V Sample Analysis of Thorium Product Streams  
Figure VI G Cell Solvent Pu Retention  
Figure VII Crane Schedule

Table I Thorium Major Routing and Equipment Changes  
Table II Dissolver Clean-Out Summary  
Table III Pre-Test Dissolver Flushing Details  
Table IV Summary of Solvent Extraction Flushes  
Table V Thorium Dissolving and Denitration  
Table VI Protactinium Recovery  
Table VII U-233 Material Balance  
Table VIII Thorium Material Balance  
Table IX Solvent Extraction Thorium Removal Flushes  
Table X Thorium Run Waste Routes and Volumes

Sketch I Thorium Process Flow Sketch

Photo I TV View of C3 Dissolver  
Photo II Scrap on Dissolver Grid  
Photo III Reclaimed Tower at T Plant

Jan 11 13 15 17 21 23 25 27 29 31 Feb 3 5 7 9 11 13

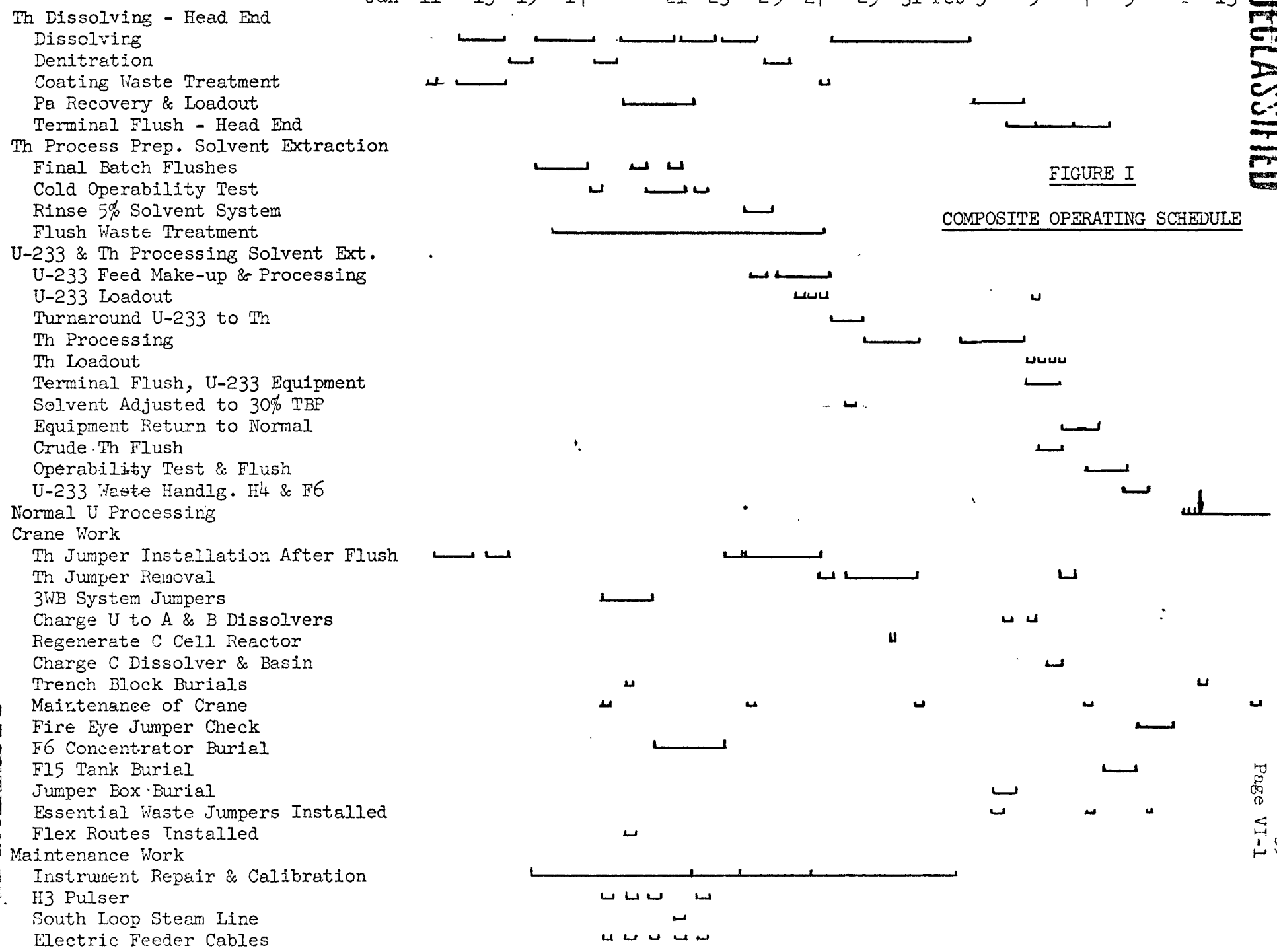


FIGURE I  
COMPOSITE OPERATING SCHEDULE

FIGURE II  
ORIGINAL OPERATING FORECAST  
VERSUS  
ACTUAL PERFORMANCE

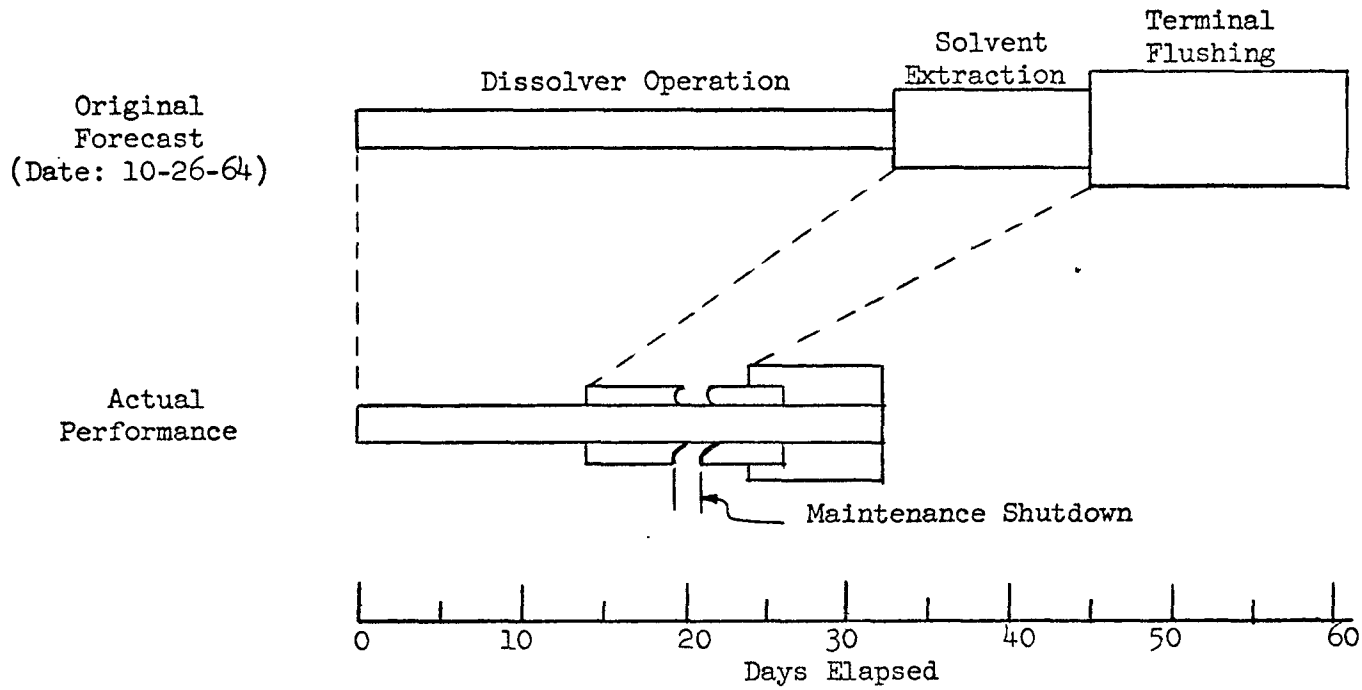


FIGURE III

SOLVENT EXTRACTION FLUSH DETAILS

Equipment Systems Flushed	12-7	12-8	12-9	12-10	12-11	12-12	12-13	12-14	12-15	12-16	12-17	12-18	12-19	12-20	12-21
L Cell Package Deep Flush															6
J5-L1-L2			3			3	3	3					1	5	
J Cell Package		3	3		3						3	3	1		5
J1-H2-H3-J3 J6-J7-J8-K1											2	2	2	2	1 5 5
K1-J1 K1-F10			1			1							1	1	1 1 1
J7-J8-K1 K1-J1		1				1									
J6-J4-J2-J5			3		3		3								
Backcycle Spin H4 MT out															
G1 Flush G7 Flush	4	4	4	1				4	5						
R1 Flush	4		4					4	5						

- |                         |                            |
|-------------------------|----------------------------|
| 1. Water                | 4. 5% Oxalic-30% Nitric    |
| 2. 20% HNO <sub>3</sub> | 5. 10% Caustic-2% Tartaric |
| 3. 10% HNO <sub>3</sub> | 6. 30% HNO <sub>3</sub>    |



FIGURE III (Con't)

SOLVENT EXTRACTION FLUSH DETAILS

Equipment Systems Flushed	12-22	12-23	12-24	12-25	12-26	12-27	12-28	12-29	12-30	12-31	1-1	1-2	1-3	1-4	1-5	
L Cell Package Deep Flush	6	6									6	6	6			
J5-L1-I2	5	1	4					1								
J Cell Package	5	1	4				1									
J1-H2-H3-J3 J6-J7-J8-K1	5	1								4		4	4	4	1	5
K1-J1 K1-F10														4	4	4

- |                         |                            |
|-------------------------|----------------------------|
| 1. Water                | 4. 5% Oxalic-30% Nitric    |
| 2. 20% HNO <sub>3</sub> | 5. 10% Caustic-2% Tartaric |
| 3. 10% HNO <sub>3</sub> | 6. 30% HNO <sub>3</sub>    |

FIGURE III (Con't)

SOLVENT EXTRACTION FLUSH DETAILS

Equipment Systems Flushed	1-6	1-7	1-8	1-9	1-10	1-11	1-12
L Cell Package Deep Flush							
J5-I1-I2		5	1	4	1		
J Cell Package	5	1	4		1		
J1-H2-H3-J3 J6-J7-J8-K1	5,5	1	4	4	4	1	
K1-J1 K1-F10			1	4	4	1,1,1,1,1,1,1,1	

- |                         |                            |
|-------------------------|----------------------------|
| 1. Water                | 4. 5% Oxalic-30% Nitric    |
| 2. 20% HNO <sub>3</sub> | 5. 10% Caustic-2% Tartaric |
| 3. 10% HNO <sub>3</sub> | 6. 30% Nitric              |

FIGURE IV

SAMPLE ANALYSES OF U<sup>233</sup> PRODUCT STREAMS

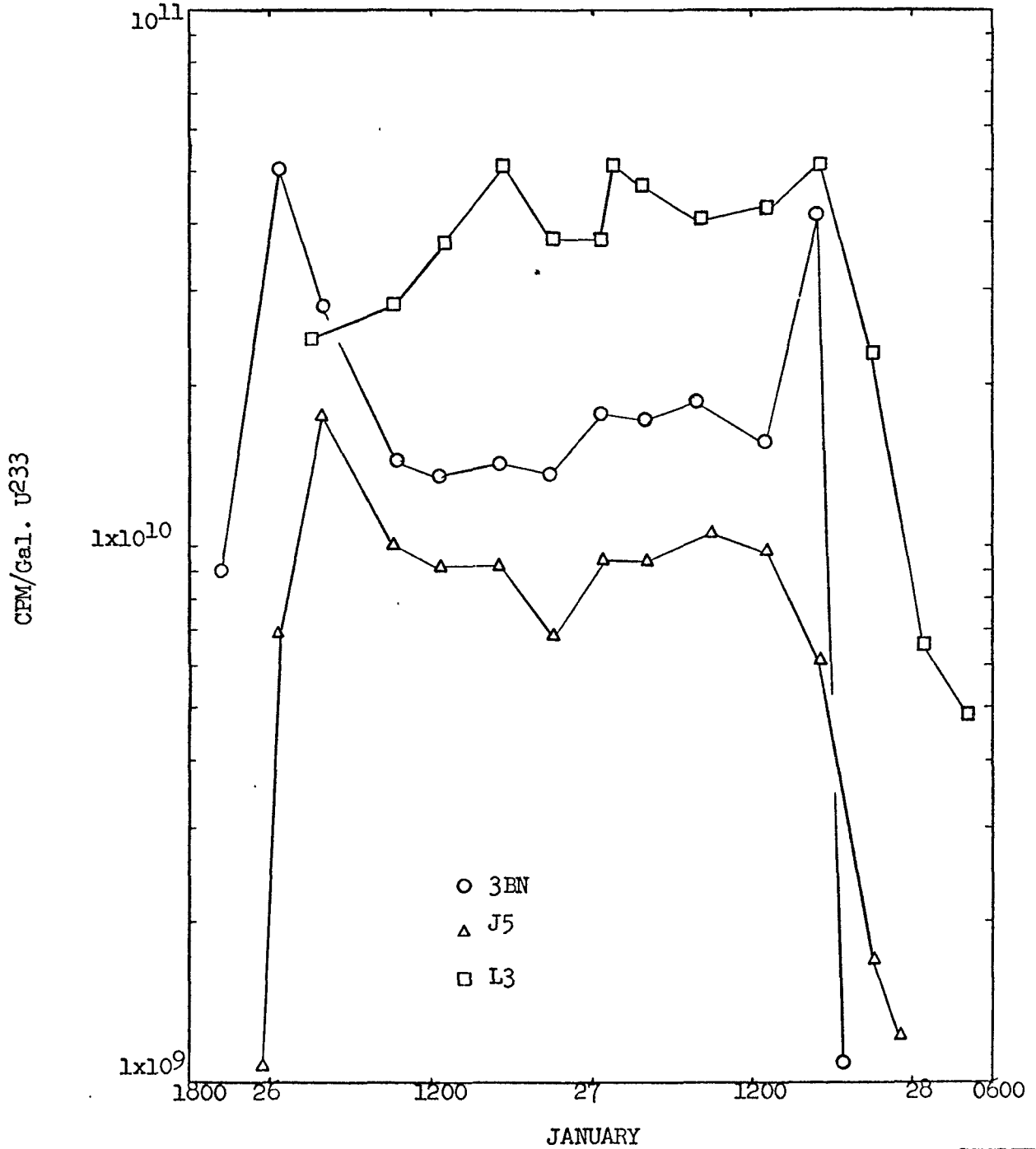


FIGURE V

SAMPLE ANALYSES OF THORIUM PRODUCT STREAMS

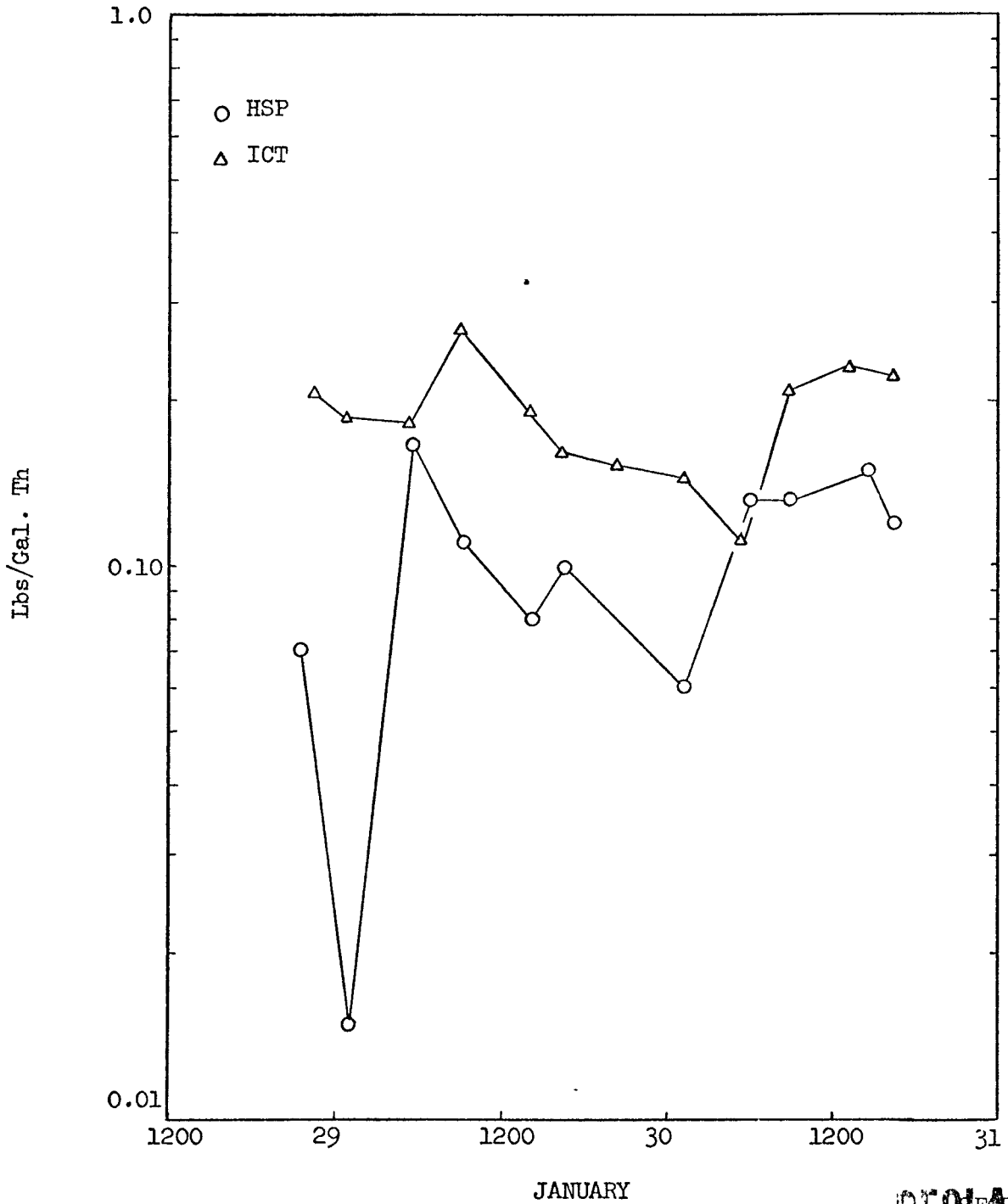


FIGURE V (Con't)

SAMPLE ANALYSES OF THORIUM PRODUCT STREAMS

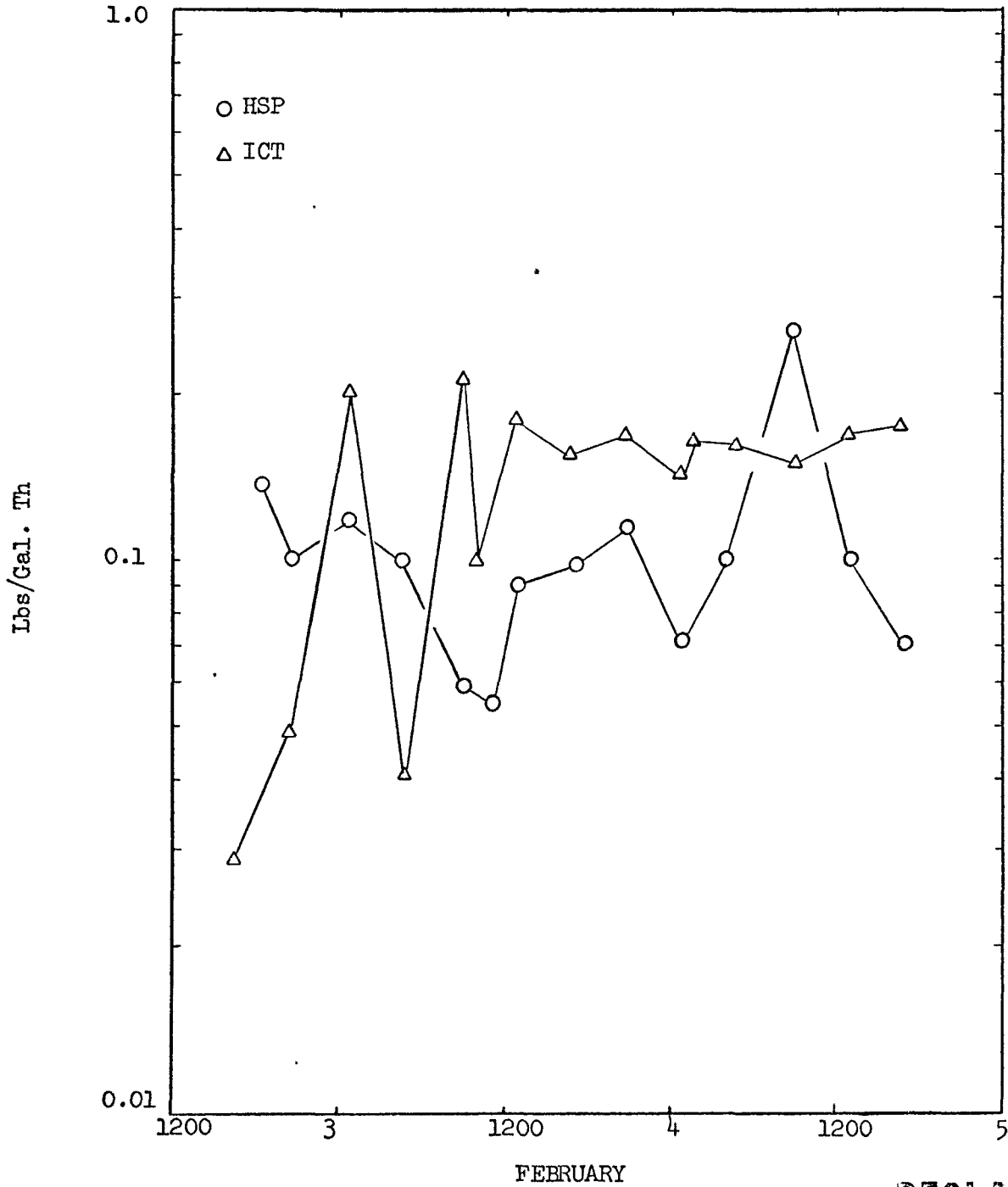
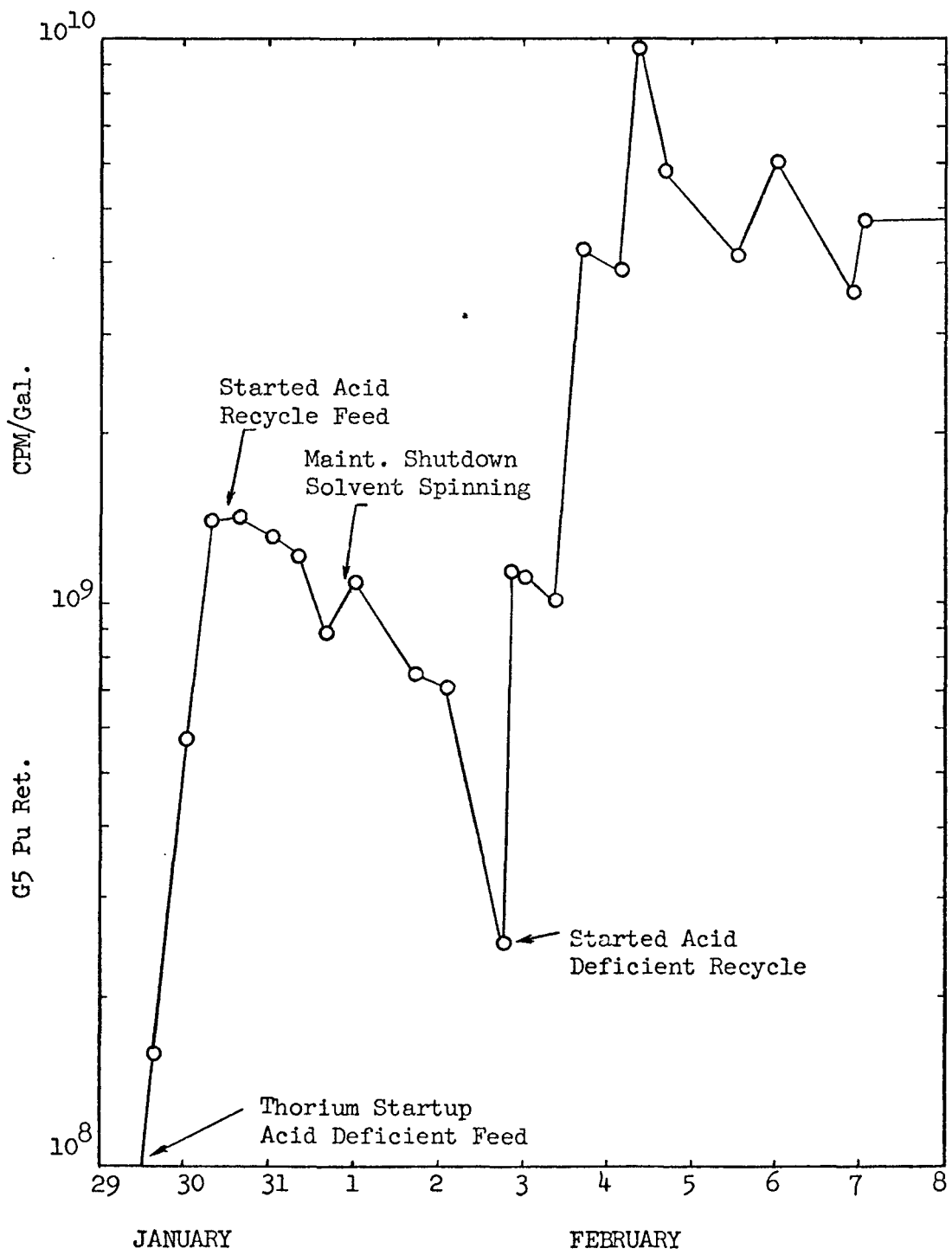


FIGURE VI

G CELL SOLVENT Pu RETENTION

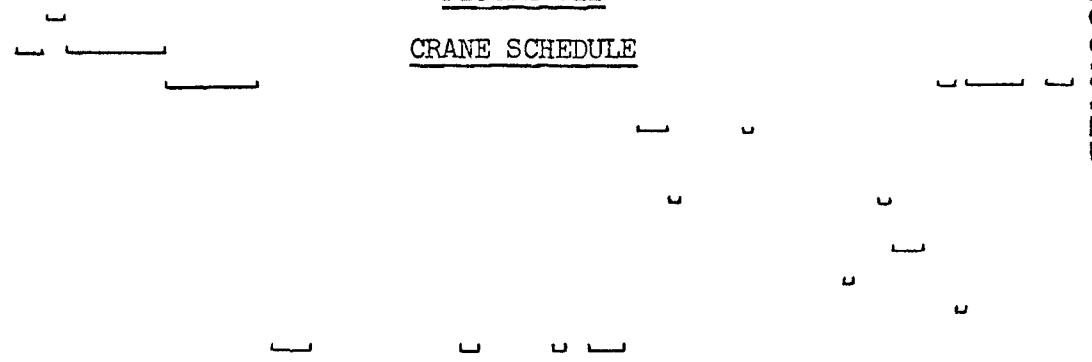


DEC. 15 17 19 21 23 25 27 29 31 JAN. 4 6 8 10 12 14

THORIUM RUN

- TV Viewing of C3 Dissolver Interior
- Replacement of C3 Dissolver
- Thoria Dissolution Step Jumper Installation
- Removal of C3 Dissolver Tower and Ammonia Scrubber
- Installation and Removal of New By-Pass Vapor Jumper
- Installation of Repaired Dissolver Tower
- Crane Operator to T-Plant for Tower
- Thoria Charge to C3 Dissolver
- Flex Jumper Flush Routings

FIGURE VII  
CRANE SCHEDULE



SHUTDOWN WORK

- Filter Trouble (no crane work)
- H2 Jetout Jumper
- F6 RHTB
- B2 Silver Reactor Regeneration
- 8842 Condensate Header Tie-In from Tunnel
- Trench Block Burial
- L Cell Repair
- F8 Agitator



JAN. 15 17 19 21 23 25 27 29 31 FEB 3 5 7 9 11 13

THORIUM RUN

Extraction Battery Flush Jumper Installation \_\_\_\_\_

U-233 Processing Jumper Installation \_\_\_\_\_

Thorium Processing Jumper Installation \_\_\_\_\_

Thoria Dissolution Step Jumper Removal \_\_\_\_\_

U-233 Processing Jumper Removal \_\_\_\_\_

Thorium Processing Jumper Removal \_\_\_\_\_

HA Column Pulser Replacement \_\_\_\_\_

Thief Sample - Thorium Product \_\_\_\_\_

IWF Pump Replacement \_\_\_\_\_

Normal Processing \_\_\_\_\_

FIGURE VII (Con't)

CRANE SCHEDULE

SHUTDOWN WORK

Electrical Head Recovery \_\_\_\_\_

3WB Jumper Installation \_\_\_\_\_

Trench Block Burial \_\_\_\_\_

Normal Charging \_\_\_\_\_

F6 Burial \_\_\_\_\_

HSR Jumper \_\_\_\_\_

F6 LHTB Replacement \_\_\_\_\_

C2 Silver Reactor Regeneration \_\_\_\_\_

H2 Pulser \_\_\_\_\_

F12 Pump and Rotometer Jumper \_\_\_\_\_

EW Routing Jumpers \_\_\_\_\_

Coating Waste Test \_\_\_\_\_

F15 Tank Replacement \_\_\_\_\_

H4 RHTB Replacement \_\_\_\_\_



TABLE I

THORIUM MAJOR ROUTING & EQUIPMENT CHANGES

CANYON

<u>Routes</u>	<u>Service</u>	<u>No. of Jumpers Required</u>
C3-E3	Dissolver to Centrifuge Feed Tank	2
E1-C3	Centrifuge Slurry Tank to Dissolver	3
E5-D2	Centrifuge Catch Tank to Coating Waste Tank	2
C3-F12	Dissolver to IWF Tank	4
E5-J1	Centrifuge Catch Tank to 3WB Tank	3
3A Col-F12	3AW to IWF Tank	1
J2-7713 HDR	HAO Tank to No. 1 Organic HDR	1
3B Col-J5	3BN to 2AF Tank	1
R1A-J3	Organic Makeup Tank to 1BXF Tank	3
J3-J2	1BXF Tank to HAO Tank	1
K1-J1	2DF Tank to 3WF Tank	2
K1-Trailer Loadout	2DF Tank to UNH HDR	2
HS1S-HNO <sub>3</sub> -HS Col	Utility Spare to HA Column	1
HAX-HNO <sub>3</sub> -HA Col	HSR Nitric to HA Column	1
K1-F10	2DF Tank to 3WF Tank	3 Flex
IF Jetout Hdr-F10	Interface Jetout Header to 3WF Tank	1 Flex
HA Col-F12	HAW to IWF Tank	1 Flex
F12-F16	IWF Tank to PAW Tank	1 Flex
E5-F12	Centrifuge Catch Tank to IWF Tank	1 Flex

Equipment

New C3 dissolver installed.

Repaired dissolver tower installed.

Temporary tower drain blank installed.

Temporary C3 dissolver vapor by-pass jumper installed.

PIPE & OPERATING GALLERY

<u>Routes</u>	<u>Service</u>
3AF Acid-J5	3AF acid routed to 2AF tank at JG95
7007 HDR-K1	AMU utility header to 2DF tank at KG127

TABLE I (Con't)

PIPE & OPERATING GALLERY (Con't)

<u>Routes</u>	<u>Service</u>
Chem Add Dolly-L3A	Chem add dolly fitting installed on 2BP stripper acid system.
Chem Add Dolly-C3	Chem add dolly fitting installed on dissolver nitrate add system.
Jet Steam-3BN Jet	3WB tank to 3WB concentrator jet steam supply used for 3BN jet.
7007 HDR-HS Col	Concentrated nitric header to HS Col at HG37
217TK-J5	2DF iron feed tank routed to 2AF tank at JG94
219 TK-HS Col	Auxiliary nitrite feed tank routed to HSS-H <sub>2</sub> O line
Steam- C3 Sparger	Steam supply routed to C3 dissolver
UO <sub>3</sub> HDR-U5 (Blanked)	UO <sub>3</sub> recovered acid header to AFF tank blanked in pipe chase.

INSTRUMENT

- 3B and HA column Dp recalibrated.
- 293-A dissolver off-gas I<sub>131</sub> monitor recalibrated for Ru.
- C3 dissolver knock-out pot liquid level alarm installed.
- C3 dissolver off-gas Ru monitor installed.
- C3 dissolver coil discharge monitor and alarm installed.
- C3 dissolver temperature probe and Sp.Gr. Wt.Ft. dip tubes shortened.
- PSC concentrator (L4) high Sp.Gr. alarm installed.
- 3AF acid flow meter repositioned.
- HSS-Butt rotometer installed.
- HSIS-Nitric rotometer installed.
- Pa load-out cask liquid level probe installed.

TABLE II  
DISSOLVER CLEAN-OUT SUMMARY

	Old C3 Dissolver		New C3 Dissolver			Total
	Original Flushes	Modified Flushes	With Original Tower	Tower Removed	With Re-Used Tower	
Number of Flushes	27	21	17	3	2	70
Time (Days)	29	18	14	4	2	69
Total Volume (Gal)	64,630	27,520	35,630	5,000	3,285	136,630
Chemical Cost	\$6,034	\$2,853	\$3,321	\$379	\$367	\$12,954
Uranium Removed (lbs)	79.8	7.8	8.2	0.14	0.87	96.8
Plutonium Removed (gm)	91.2	11.6	6.2	0.35	1.07	110.4
Pu/U (gm/ton)	2,286	2,974	1,512	5,000	2,459	2,280
Cs/U (curies/ton)		2.75x10 <sup>3</sup>	3.54x10 <sup>3</sup>	3.50x10 <sup>3</sup>	2.58x10 <sup>3</sup>	3.09x10 <sup>3</sup>
Avg. Uranium Dissolving Rate (gm/hr)	153.7	26.9	32.6	1.5	31.4	:
Avg. Corrosion Rate mils/mo.	8.5	14.9	4.6	1	5.6	

DECLASSIFIED

TABLE III  
PRE-TEST DISSOLVER FLUSHING DETAILS

<u>Flush</u>	<u>Type</u>	<u>Vol. (Gal.)</u>	<u>Date</u>	<u>Time (Hrs)</u>	<u>Temp. For Time Period (°C)</u>	<u>Disposed To</u>	<u>Chem Cost (\$)</u>	<u>U Picked Up For Time Period (lb)</u>	<u>Pu (gm)</u>	<u>Pu/U (gm/ton)</u>	<u>U Pickup Rate gm/hr</u>	<u>Corrosion Rate mil/mo.</u>
1	57% HNO <sub>3</sub>	2000	11-3	2 <sup>1</sup>	Boiling	B-3	335	26	24	1846	491	
2	57% HNO <sub>3</sub>	400	11-6	20	Boiling	F-12	67		6.4		4.5	
3	57% HNO <sub>3</sub>	400	11-7	20	Boiling	F-12	67	0.80	1.4	3500	18	
4	20% HNO <sub>3</sub>	2800	11-9	4	95°	F-12	136	1.40	1.2	1714	159	
5	10% NaOH-2% Tartaric	2800	11-10	4	80°	D-2	254	4.7	3.3	1404	533	
6	30% HNO <sub>3</sub> -0.03M KF 0.15M ANN	2800	11-13	1	80°	F-12	261	25	39	2720	11340	<25
7	Water	2800	11-15	1	80°	F-12		0.90	1.3	2889	408	
8	20% HNO <sub>3</sub>	2800	11-16	4	80°	F-12	136	0.59	1.0	3390	67	
9	10% NaOH-2% Tartaric	2800	11-18	4	80°	D-2	254	3.6	5.9	3278	408	
10	30% HNO <sub>3</sub> -0.03M KF 0.15M ANN	2800	11-19	1	80°	F-12	261	8.7	2.7	620	3946	~53
11	Water	2800	11-19	1	80°	F-12		0.9	1.3	2890	408	
12	30% HNO <sub>3</sub> -0.03M KF 0.15M ANN	2800	11-20	4	Boiling	F-12	261	1.8	1.2	1330	204	<33
13	10% HNO <sub>3</sub>	2800	11-21	1	50°	F-12	68	0.28	0.10	714	127	
14	30% HNO <sub>3</sub> -0.03M KF 0.15M ANN	2800	11-21	4	Boiling	F-12	261	0.95	0.45	947	108	

DECLASSIFIED

TABLE 1 (Con't)

Flush	Type	Vol. (Gal.)	Date	Time (Hrs)	Temp. For Time Period (°C)	Disposed To	Chem Cost (\$)	U Picked Up For Time		U Pickup Rate gm/hr	Corrosion Rate mil/mo.	
								Period (lb)	Pu (gm)			
15	Water	2800	11-22	1	80°	F-12		0.21	<0.1	<952	95	
16	30% HNO <sub>3</sub> -0.03M NaF 0.15M ANN	2800	11-23	15	Boiling	F-12	261	0.89	0.48	1079	27	5
17	10% NaOH-2% Tartaric	2800	11-24	4	80°	D-2	254	1.02	0.11	216	116	
18	57% HNO <sub>3</sub> -0.025M NaF 0.125M ANN	1400	11-25	12	Boiling	F-12	257	1.57	0.34	433	59	4
19	57% HNO <sub>3</sub> -0.025M NaF 0.125M ANN	1515	11-26	12	Boiling	F-12	257	0.34	0.35	2058	13	4.4
20	57% HNO <sub>3</sub> -0.025M NaF 0.125M ANN	1580	11-28	8	Boiling	F-12	257	0.16	0.01	125	9	3.3
21	57% HNO <sub>3</sub> -0.025M NaF 0.125M ANN	2192	11-28	8	Boiling	F-12	355	0.12	0.19	3167	7	3.1
22	57% HNO <sub>3</sub> -0.025M NaF 0.125M ANN	1927	11-29	8	Boiling	F-12	323	0.15	0.04	533	9	4.3
23	25% Caustic	1980	11-29	4	Boiling	D-2	205	0.55	0.21	764	63	
24	57% HNO <sub>3</sub> -0.025M NaF 0.125M ANN	2390	11-29	8	Boiling	F-12	376	0.11	0.14	2546	6	4.9
25	57% HNO <sub>3</sub> -0.025M NaF 0.125M ANN	2185	11-30	8	Boiling	F-12	376	0.094			5	4.0
26	57% HNO <sub>3</sub> -0.025M NaF 0.125M ANN	2360	12-1	8	Boiling	F-12	376	0.09	0.085	1890	5	3.0
27	57% HNO <sub>3</sub> -0.025M NaF 0.125M ANN	2280	12-1	8	Boiling	F-12	376	0.14	0.13	1860	8	5.7
28	28% HNO <sub>3</sub> -0.05M NaF 0.05M ANN	500	12-1	8	Boiling	F-12	37	0.14	0.014	2000	8	5.8

DECLASSIFIED

DECLASSIFIED

DECLASSIFIED

TABL (Con't)

Flush	Type	Vol. (Gal.)	Date	Time (Hrs)	Temp. For Time Period (°C)	Disposed To	Chem Cost (\$)	U Picked Up For Time Period (lb)	Pu (gm)	Pu/U (gm/ton)	U Pickup Rate gm/hr	Corrosion Rate mil/mo.
29	28% HNO <sub>3</sub> -0.05M NaF 0.05M ANN	1000	12-2	24	Boiling	F-12	74	0.82	1.10	2683	15	8.1
30-A	20% HNO <sub>3</sub> -0.025M NaF 0.025M Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	450	12-5	8	45°			0.147	0.14	1905	6	
30-B	20% HNO <sub>3</sub> -0.76M NaF 0.08M Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	1400		4	60°			0.453	0.21	927	51	
	20% HNO <sub>3</sub> -0.76M NaF 0.08M Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>			5	30°	D-2	73	<0	<0		<0	1.8
31	Water	1000	12-6	0	<50°	D-2						
32	15% NaOH	500	12-6	1	Boiling	D-2	23					
33	12% HNO <sub>3</sub>	320	12-6	1	Boiling	D-2	9					
34	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2160	12-7	0	35°			0.07	0.037	1057		
		2153		4	Boiling			0.60	0.057	190	60	
		2166		~4	Boiling							
	57% HNO <sub>3</sub> -0.033M NaF 0.05M ANN	2205		4	Boiling			1.01	1.18	2340	51	4.8
		2170		4	Boiling			1.78	1.64	1840	57	8.8
		2135		4	Boiling			1.35	1.86	2760	25	30.0
	57% HNO <sub>3</sub> -0.04M NaF 0.05M ANN	2150		4	Boiling			1.57	1.61	2050		24.0
		2170		4	Boiling			1.69	2.42	2860	32	
	57% HNO <sub>3</sub> -0.049M NaF 0.05M ANN	2170		4	Boiling			1.82			15	48.0

DECLASSIFIED

DECLASSIFIED

TAE (Con't)

<u>Flush</u>	<u>Type</u>	<u>Vol. (Gal.)</u>	<u>Date</u>	<u>Time (Hrs)</u>	<u>Temp. For Time Period (°C)</u>	<u>Disposed To</u>	<u>Chem Cost (\$)</u>	<u>U Picked Up For Time Period (lb)</u>	<u>Pu (gm)</u>	<u>Pu/U (gm/ton)</u>	<u>U Pickup Rate gm/hr</u>	<u>Corrosion Rate mil/mo.</u>
	57% HNO <sub>3</sub> -0.049 NaF 0.05M ANN	2170		4	Boiling			2.28			52	
	57% HNO <sub>3</sub> -0.049 NaF 0.061M ANN	2213		6	Boiling			2.21	2.96	2680	<0	26.0
	57% HNO <sub>3</sub> -0.059M NaF 0.061M ANN	2221		4	Boiling			2.04	3.33	3260	<0	26.0
	57% HNO <sub>3</sub> -0.059M NaF 0.067M ANN	2283		4	Boiling	D-2	356	1.60	3.71	4640	<0	31.2
35	Water	300	12-9	0	35°	D-2						
36	1.2% HNO <sub>3</sub>	1543	12-10	1	Boiling	D-2	5	0.123	0.154	2570	56	
37	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2168		0	35°			0.028	0.035	2500		
		2168		4	Boiling			0.607	0.473	1560	66	7.0
		2115		6	Boiling			0.466	0.782	3360		3.5
		2115		6	Boiling			0.634	0.910	2870	1	10.1
		2115		4	Boiling	D-2	348	1.04	1.270	2440	46	20.7
38 <sup>(2)</sup>	20% HNO <sub>3</sub> -0.5M NaF	80	12-11	4	~80°	D-2	15	0.032	0.034	2125	3.6	9.8
39 <sup>(2)</sup>	20% HNO <sub>3</sub> -0.5M NaF	80	12-12	4	~80°	D-2	15	0.043	0.049	2279	5	18.0
40 <sup>(2)</sup>	20% HNO <sub>3</sub> -0.5M NaF	80	12-12	4 2/3	~80°	D-2	15	0.170	0.099	1176	16.3	31.0
41 <sup>(2)</sup>	20% HNO <sub>3</sub> -0.5M NaF	80	12-13	4 2/3	~80°	D-2	15	0.073	0.238	6520	7	4.9
42	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2338	12-14	0	35°			0.010	0.002	400		

DECLASSIFIED

TABLE III (Con't)

Flush	Type	Vol. (Gal.)	Date	Time (Hrs)	Temp. For Time Period (°C)	Disposed To	Chem Cost (\$)	U Picked Up For Time Period (lb)	Pu (gm)	Pu/U (gm/ton)	U Pickup Rate gm/hr	Corrosi Rate mil/mo
	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN			4½	Boiling			0.350	0.599	3420	35	< 3
				6	Boiling			0.620	0.852	2750	47	< 0
				6	Boiling	D-2	348	0.966	1.380	2860	27	< 0
43(2)	20% HNO <sub>3</sub> -1.0M NaF	80	12-15	4	80° → 35°	D-2	16	0.019	0.023	2868	2.1	61.5
44(2)	20% HNO <sub>3</sub> -1.0M NaF	80	12-15	8½	70° → 95° → 60°	D-2	16	0.131	0.312	4760	7	70.0
45	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2260	12-16	0	35°			0.017	0.073	8589		
		2260		4	Boiling			1.107	0.998	1800	125	17.0
		2260		4	Boiling	D-2	348	1.197	1.081	1815	11	10.0
46	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2237	12-16	0	35°			0.057	0.073	2560		
		2183		4	Boiling	D-2	350	0.221	0.319	2890	41	< 6
47	57% HNO <sub>3</sub> -0.025M NaF 0.10M ANN	2315	12-17	0	~35°			0.057	0.0732	2580		
				4	Boiling	D-2	350	0.221	0.319	2890	20	
48(2)	20% HNO <sub>3</sub> -2.0M NaF	60	12-18	4	60° → 95°			0.0450	0.115	5111	5	< 8.6
	53% HNO <sub>3</sub> -0.05M NaF 0.10M ANN	2360		4	Boiling	D-2	344	0.330	0.540	3270	31	2.8



TABLE I (Con't)

NEW DISSOLVER INSTALLED

Flush	Type	Vol. (Gal.)	Date	Time (Hrs)	Temp. For Time Period (°C)	Disposed To	Chem Cost (\$)	U Picked Up For Time Period (gm)	Pu (gm)	Pu/U (gm/ton)	U Pickup Rate gm/hr	Corrosion Rate mil/mo
1	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2360	12-21	2½	Boiling			281	0.753	2428	112.0	
		2390		3	Boiling			400	0.999	2763	39.7	16.1
		2390		20	50°	D-2	348	541	1.51	2529	7.1	1.0
2	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2362	12-22	0	35°			8	0.0236	2676		
		2002		2	Boiling			30	0.070	2116	11.0	
		1944		2	Boiling	D-2	348	123	0.263	1937	46.5	1.3
3	30% HNO <sub>3</sub> -Tower Flush	500	12-23	0	50°			91				
		2000		1 3/4	Boiling			236	0.377	1450	82.9	
		2200		1	50°	D-2	168	100	0.192	1739	<0	
4	30% HNO <sub>3</sub> -Tower Flush	530	12-24	0	50°			0.1				
		2053		3	Boiling	D-2	157	410	0.975	2155	137.0	
5	30% HNO <sub>3</sub> -Tower Flush	2129	12-24	2	Boiling	D-2	163	58	0.144	2243	29.0	
6	30% HNO <sub>3</sub> -Tower Flush	1767	12-24	3½	Boiling			55.4	0.015	246	15.8	
		1698		3	Boiling	D-2	136	123.3	0.265	1947	22.6	
7	30% HNO <sub>3</sub> -Tower Flush	1842	12-25	1 3/4	Boiling	D-2	147	82.8	0.190	2076	47.3	
8	30% HNO <sub>3</sub> -Tower Flush	1861	12-25	2	Boiling	D-2	142	57.5	0.150	2363	28.8	
9	30% HNO <sub>3</sub> -Knockout Pot Flush	2108	12-26	5 3/4	Boiling	D-2	162	110.1	0.290	2386	19.1	

DECLASSIFIED

DECLASSIFIED

TABLE III (Con't)

Flush	Type	Vol. (Gal.)	Date	Time (Hrs)	Temp. For Time Period (°C)	Disposed To	Chem Cost (\$)	U Picked Up For Time (gm)	Pu (gm)	Pu/U (gm/ton)	U Pickup Rate gm/hr	Corrosion Rate mil/mo.
10	30% HNO <sub>3</sub> -Knock-out Pot Flush	1751	12-26	2	Boiling	D-2	135	95.4	0.168	1595	47.7	
11	30% HNO <sub>3</sub> -Knock-out Pot Flush	1951	12-26	3½	Boiling	D-2	150	124	0.247	2004	35.4	
12	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2385	12-26	0	50°			27	0.061	2049		
		2300		4	100°			87.5	0.239	2477	15.0	
		2300		1 2/3	Boiling	D-2	348	146	0.340	2105	34.4	
13	30% HNO <sub>3</sub> -Knock-out Pot Flush	2260	12-27	4½	Boiling	D-2	174	84	0.020	205	18.7	
14	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2345	12-27	0	50°			11.7	0.025	194		
		2345		2	Boiling			127.5	0.213	1515	58.0	
		2345		6 3/4	Boiling	D-2	348	148.7	0.427	2604	2.9	
15	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2192	12-29	8½	90°			59.0			6.9	3.1
		2192		6	90°			218.9	0.291	907	15.1	7.3
		2115		5	Boiling	D-2	348	364.1	0.792	1973	29.0	7.9
16	Water + 1500 gal. 57% HNO <sub>3</sub> through Tower	2315		2				305	0.689	2049	152.5	
					6	Boiling	D-2	27	406	1.162	2861	16.8
17	Water + 850 gal. 57% HNO <sub>3</sub> through Tower	1750		2	Boiling			159	0.512	3223	78.5	

TABLE III (Con't)

<u>Flush</u>	<u>Type</u>	<u>Vol. (Gal.)</u>	<u>Date</u>	<u>Time (Hrs)</u>	<u>Temp. For Time Period (°C)</u>	<u>Disposed To</u>	<u>Chem Cost (\$)</u>	<u>U Picked Up For Time Period (gm)</u>	<u>Pu (gm)</u>	<u>Pu/U (gm/ton)</u>	<u>U Pickup Rate gm/hr</u>	<u>Corrosion Rate mil/mo.</u>
5	Cycles-Boiling-Cool	1628		~3	Boiling			339	0.797	2352	60.0	
5	Cycles-Boiling-Cool	1628		~3	Boiling			458	1.137	2482	39.7	
5	Cycles-Boiling-Cool	1600		~3	Boiling	D-2	20	679	1.385	2040	73.7	
18(3)	5% HNO <sub>3</sub>	1410	1-3	0	35°			14.7	0.052	3568		
		1065		3	95°	D-2	15	19.3	0.065	3353	1.5	
19	5% HNO <sub>3</sub>	1500	1-4	0	35°			1.8	<0.002	<987		
		1200		3	95°	D-2	16	3.6	0.012	3000	0.6	
20	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2100	1-5	28	Boiling	D-2	348	43.7	0.275	5708	1.6	<1.7
21(4)	57% HNO <sub>3</sub> -0.025M NaF 0.05M ANN	2178	1-9	0	35°			50				
		2178		2	Boiling			267	0.756	2832	108.5	
		2150		5	Boiling	D-2	348	322	0.813	2525	31.0	5.6
22	57% HNO <sub>3</sub> through Tower	1676	1-10	2	Boiling			47	0.069	1459	23.5	
		1360		2	Boiling	D-2	19	74	0.266	3600	13.5	

- (1) Corrosion rate based on surface area of coil exposed to boiling conditions. Where coil is not affected such as flushes 43 and 44 rate is based on surface area exposed to the solution.
- (2) ANN added on a 2-1 mole ratio to NaF after heating period, also 500 gallons of water was added to get a volume to sample.
- (3) Dissolver tower removed.
- (4) Re-used tower installed.

TABLE IV

SUMMARY OF SOLVENT EXTRACTION FLUSHES

<u>Equipment Systems Flushed</u>	<u>Number of Flushes</u>	<u>Total Vol (Gal)</u>	<u>Chem Cost (\$)</u>	<u>Type<sup>(2)</sup> of Flush</u>
L Cell Package	5	400	31	30% HNO <sub>3</sub>
J5-L1-L2	5	25,000		Water
	4	2,000	46	10% HNO <sub>3</sub>
	2	1,000	91	Caustic Tartaric
	2	1,000	151	Oxalic Nitric
J Cell Package	5	3,500		Water
	5	3,500	78	10% HNO <sub>3</sub>
	2	1,400	129	Caustic Tartaric
	2	1,400	208	Oxalic Nitric
J1-H2-H3-J3-J6-J7-J8-K1	5	25,000		Water
	4	20,000	666	20% HNO <sub>3</sub>
	6	30,000	3,750	Caustic Tartaric
	6	30,000	5,698	Oxalic Nitric
K1-J1 K1-F10	12	49,000		Water
J7-J8-K1-J1	2	13,200		Water
J6-J4-J2-J5	3	9,600	220	10% HNO <sub>3</sub>
G&R Cell	2	1,000		Water
	2	9,000	540	Caustic Tartaric
	7	31,500	5,746	Oxalic Nitric
TOTAL(1)	81	257,100	17,354	

(1) Total includes 31 water flushes with a total volume of 116,700 gallons.  
 (2) Concentration 10% Caustic-2% Tartaric and 5% Oxalic-30% Nitric

TABLE V

THORIUM DISSOLVING AND DENITRATION

<u>Cut No.</u>	<u>Dissolving Time Hrs</u>	<u>Denitration Time Hrs</u>	<u>Thorium Dissolved #</u>	<u>Thorium Dissolving Avg. Rate (lb/hr)</u>	<u>Acid<sup>(1)</sup> Used Gals</u>	<u>Cost Per Batch \$</u>
1	33	22 <sup>(2)</sup>	3000	91	4500	770
2	60	23	2500	42	6000	1020
3	46		2050	44	5000	850
4	34		1080	31	4500	770
5	36	36 <sup>(3)</sup>	470	13	5000	850
Sub-Total	209	81	9100		25000	4260
6	23		170	7.4	2000	350
7	8		50	6.3	2000	350
8	9		17	1.9	2100	350
9	20		8	0.4	1500	250
10	55		8	0.15	2300	390
Total	324	81	9353		34900	5950

(1)Initial volume 2000 gallons, 57% HNO<sub>3</sub>, 0.025M NaF, 0.05M ANN for cuts 1-7.  
Additional acid added in 500 gallon increments. Flushes 8-10 57% HNO<sub>3</sub>.

(2)Completed first cut denitration with second cut.

(3)Third, fourth and fifth cuts combined for denitration.

TABLE VI  
PROTACTINIUM RECOVERY

		<u>Time</u> <u>(Hrs)</u>	<u>Vol.</u> <u>(Gal)</u>	<u>Chem</u> <u>Cost</u> <u>(\$)</u>	<u>Pa</u> <u>Recovered</u> <u>(gm)</u>
Pa Run on U <sup>233</sup> Feed Solution (~21 gms)	1st Pa Recovery	26	2,000	215	17
	Ba SO <sub>4</sub> PPT. Removal	13		0	~5(1)
	Final Pa Recovery	10		35	4 ± 2
	Sub-Total	49		250	4 ± 2
Pa Run on Th Waste (20 gms ± 5 gms)	Two Attempted Pa Recovery Runs	48	6,000	655	<10
Total Pa Recovery		97		905	4 ± 2

(1)A portion of Pa lost to solvent extraction feed due to equipment failure.

TABLE VII  
U<sup>233</sup> MATERIAL BALANCE

Dissolver Input		3276 gm
U <sup>233</sup> Run Feed	3090 gm	
Loadout		2886 gm
Batch 1	324 gm	
Batch 2	428 gm	
Batch 3	852 gm	
Batch 4	689 gm	
Batch 5	413 gm	
Flush 6	59 gm	
Flush 7	31 gm	
Flush 8	46 gm	
Flush 9	18 gm	
Flush 10	21 gm	
Flush 11	6 gm	
Losses		390 gm
Coats	66 gm	
Dissolver Heel (6th & 7th cuts)	70 gm <sup>(1)</sup>	
Waste (Total)	126 gm	
Sumps (Total)	128 gm <sup>(2)</sup>	

(1) Lab Analysis 115 gm  
(2) Lab Analysis 377 gm

TABLE VIII  
THORIUM MATERIAL BALANCE

Input		9489 lbs
Th Run Feed	9100 lbs	
Loadout		7660 lbs
Losses		1829 lbs
Coats	200 lbs	
Dissolver Heel (6th & 7th Cuts)	220 lbs	
Waste (Total)	975 lbs <sup>(1)</sup>	
Sumps	434 lbs <sup>(2)</sup>	

(1) Lab Analysis 802 lbs  
(2) Lab Analysis 350 lbs



TABLE IX

SOLVENT EXTRACTION THORIUM REMOVAL FLUSHES

<u>Equipment Systems Flushed</u>	<u>No. of Flushes</u>	<u>Type</u>	<u>Total Vol (Gal)</u>	<u>Chem Cost (\$)</u>
H2-H3	3	Oxalic Nitric <sup>(1)</sup>	9,000	1,360
J3-J6-J4-J5	2	10% HNO <sub>3</sub>	8,000	184
J7-J8-K1	4	Water	7,000	
J21-J22-J23	5	10% HNO <sub>3</sub>	2,500	58
J5-L1-L2	5	10% HNO <sub>3</sub>	6,000	138
L3-L4-L6	5	10% HNO <sub>3</sub>	150	4
G Cell	7	Oxalic Nitric <sup>(1)</sup>	18,500	2,794
TOTAL	31		51,150	4,538

(1) Concentration 5% Oxalic-30% Nitric.

TABLE X  
THORIUM RUN WASTE ROUTES AND VOLUMES (1)

<u>Originating Tanks</u>	<u>Nov Vol (Gal)</u>	<u>Dec Vol (Gal)</u>	<u>Jan Vol (Gal)</u>	<u>Feb Vol (Gal)</u>	<u>Total</u>	<u>Receiving UGS Tank</u>
D2	11,000	119,700	24,100	22,200	177,000	102C
F16-F18-E5	15,000	98,800	409,100	42,300	565,200	105A
G8-R8		62,100	58,300	210,000	330,400	(101-103-104-106)A
TOTAL	26,000	280,600	491,500	274,500	1,072,600	

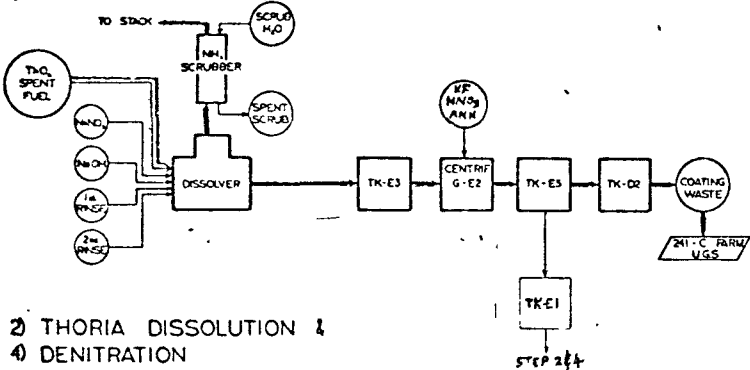
(1) These volumes represent neutralized waste transferred to UGS originating from plant flushing and thorium processing. They do not include wastes generated from normal processing in Nov., Dec., and Feb.

# THORIUM PROCESS FLOW SKETCH

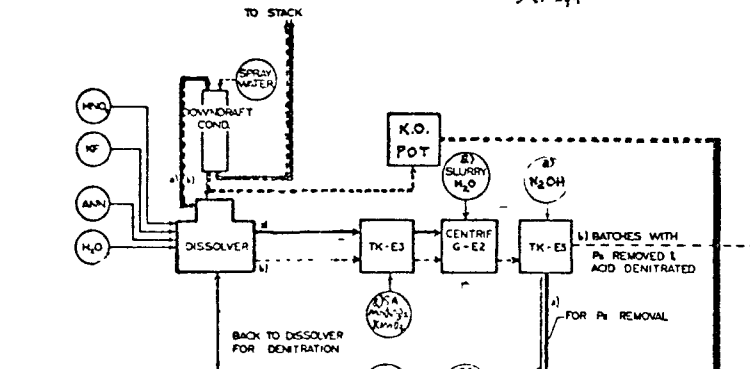
UNCLASSIFIED

- THORIUM 232
- URANIUM 233
- PROTACTINIUM
- ORGANIC
- WASTE

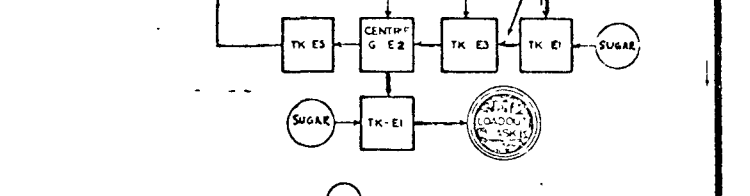
### 1) AI CAN DISSOLUTION



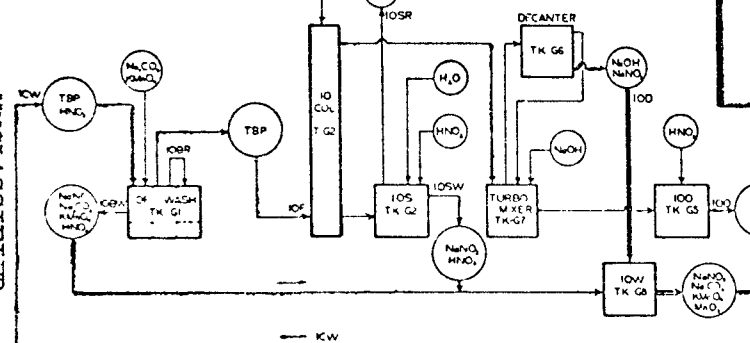
### 2) THORIA DISSOLUTION & DENITRATION



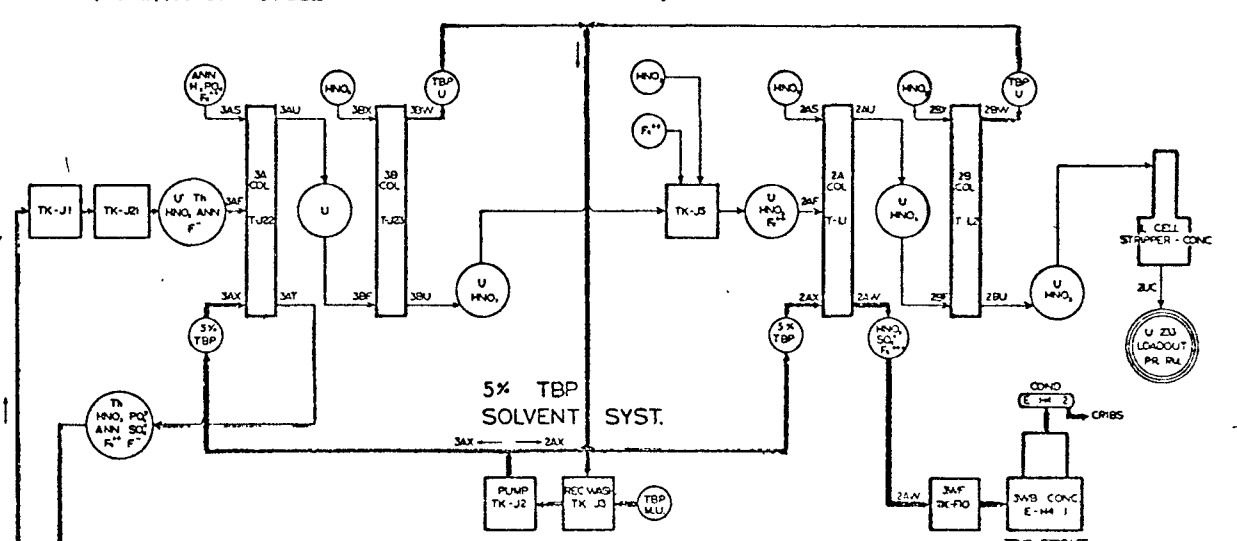
### 3) P2 RECOVERY



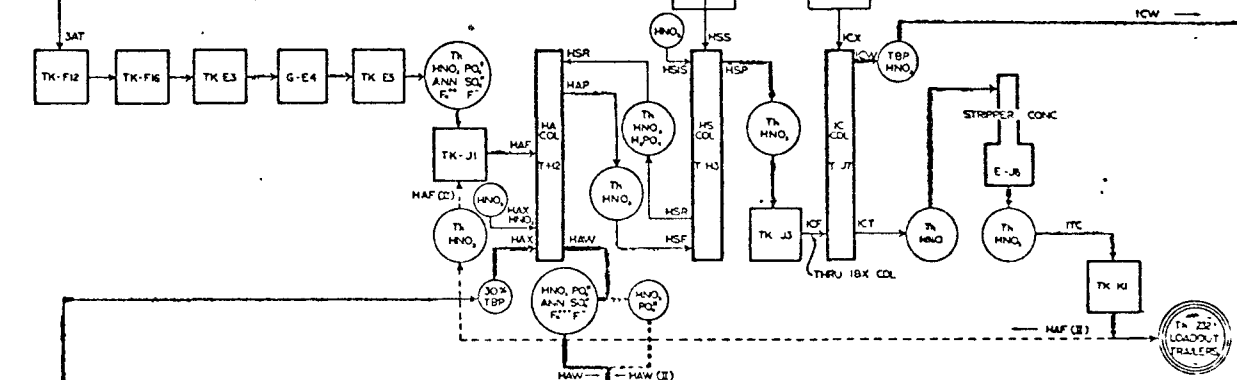
### 30x TBP SOLVENT SYST.



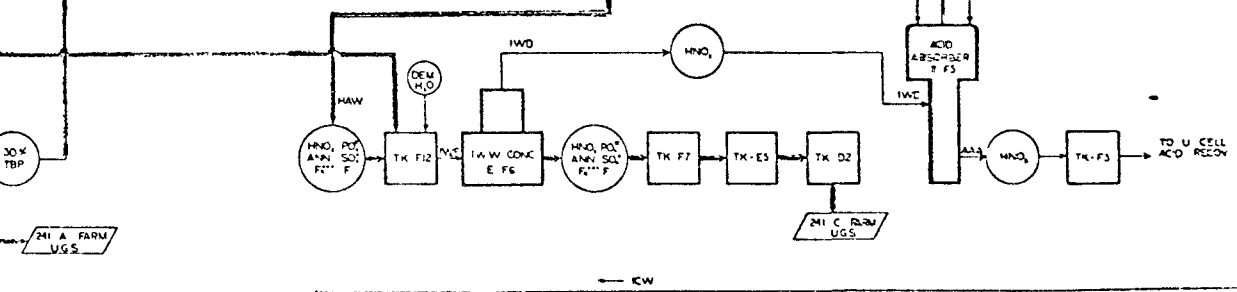
### 5) PARTITION CYCLE



### 7) Th DECONTAMINATION



### WASTE TREATMENT



UNCLASSIFIED

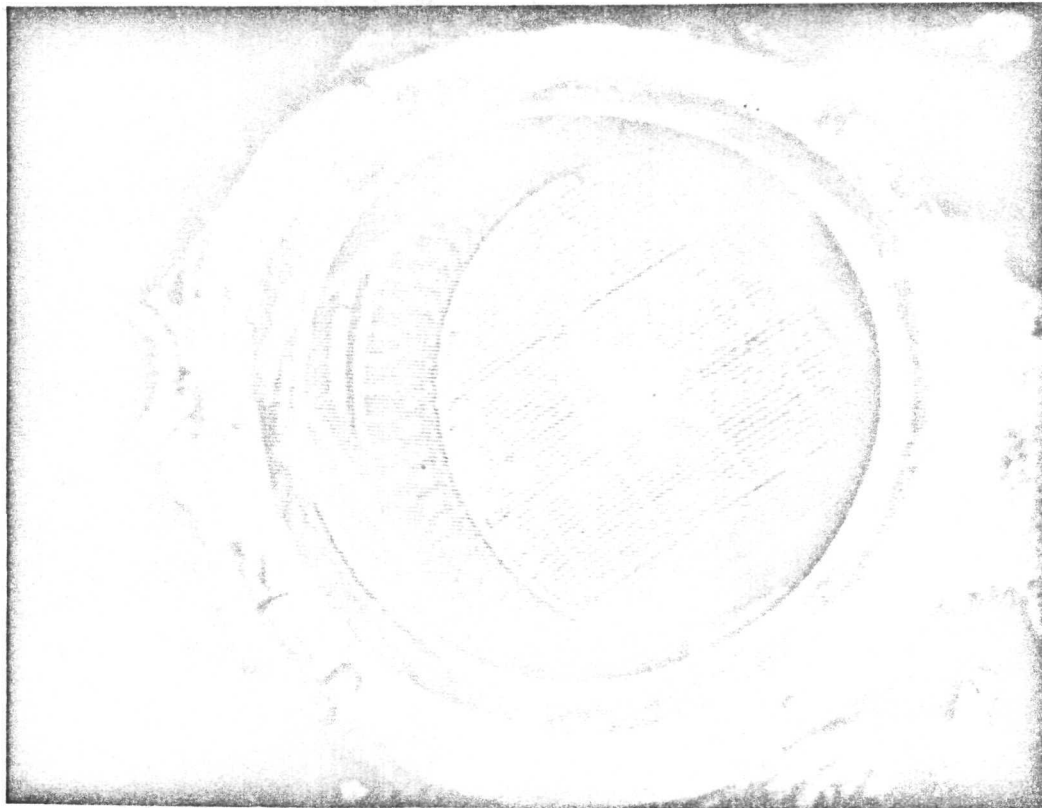
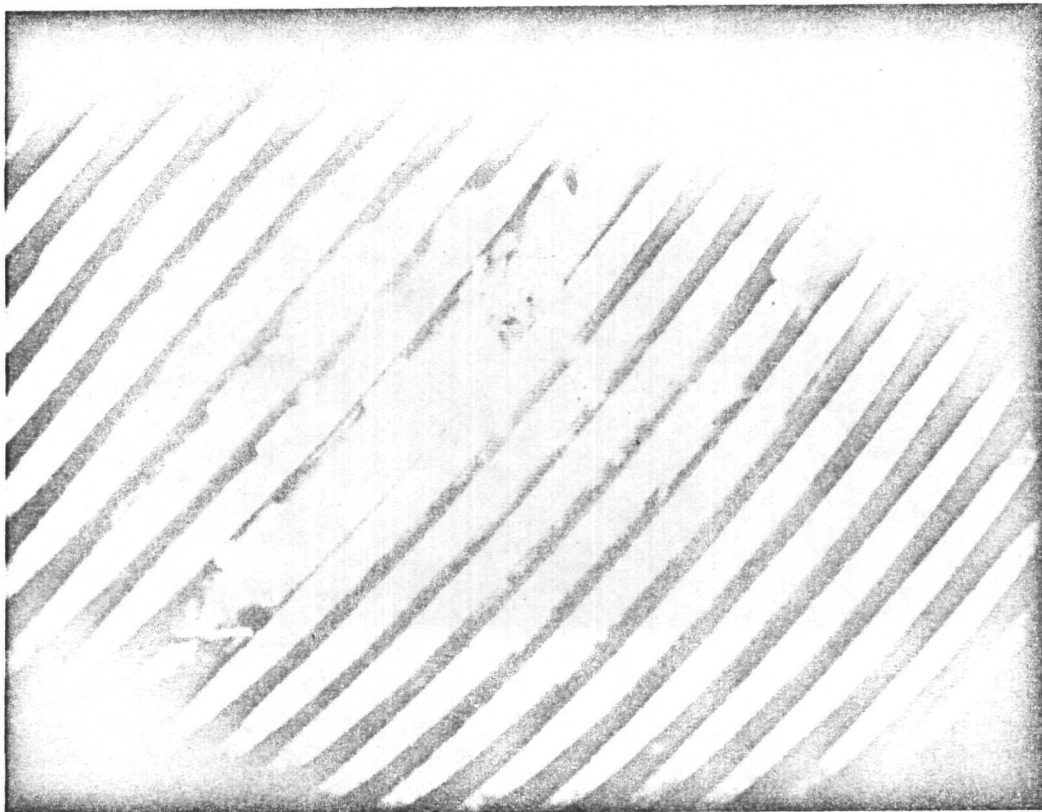


PHOTO 1 - TV VIEWING OF C3 DISSOLVER

PHOTO 2 - SCRAP ON DISSOLVER GRID



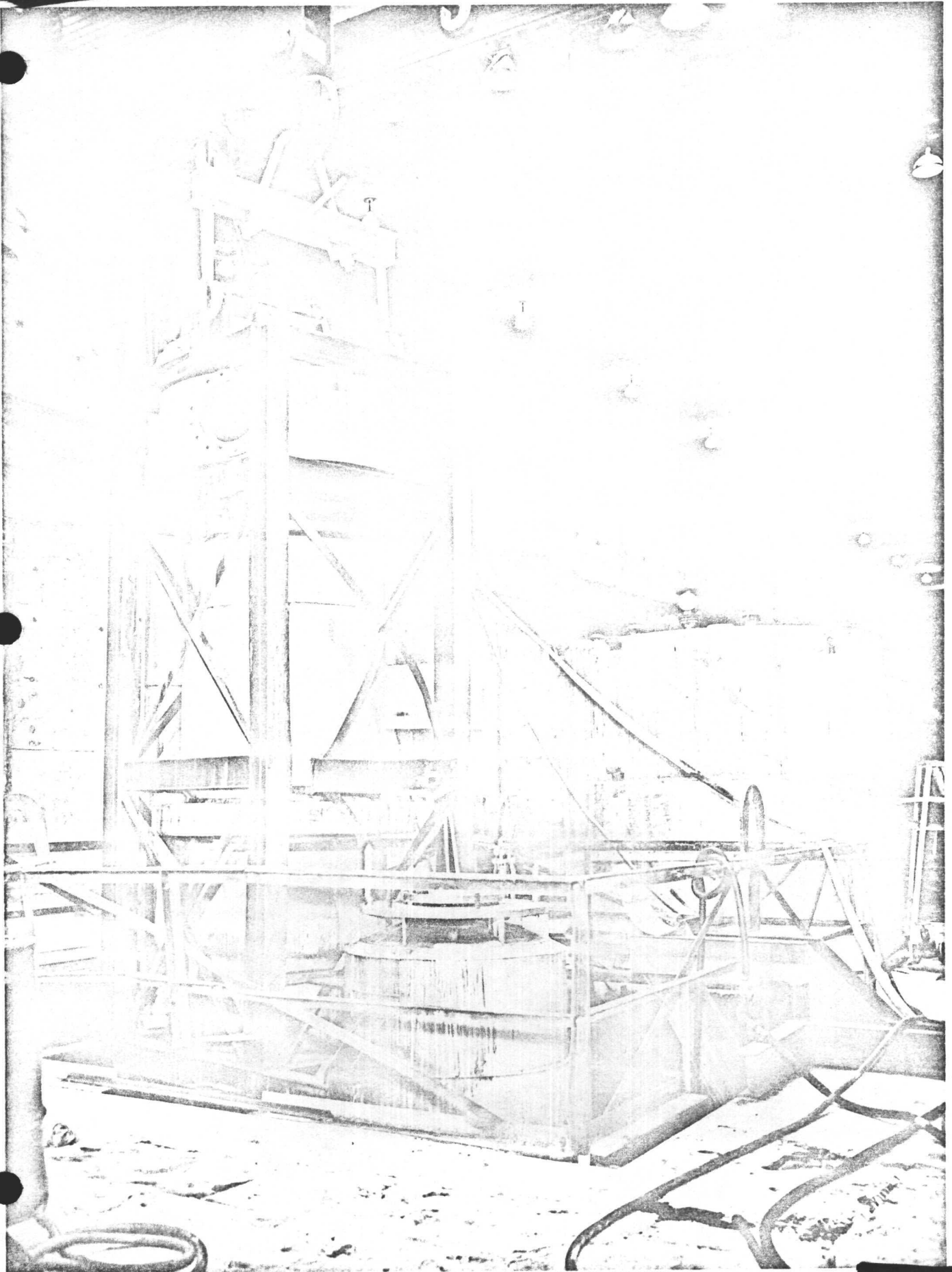


PHOTO 3 - RECLAIMED TOWER AT T-PLANT

ACKNOWLEDGEMENT

Contributions to this document were made by:

G. L. Borsheim  
J. P. Duckworth  
D. G. Harlow  
C. C. Herrington  
R. L. Hobart  
J. D. McIntosh  
G. J. Raab  
G. N. Ward