Atlantic Richfield Hanford Company
Richland, Washington 99352

ARH-2127

PUREX
PROCESS OPERATION AND PERFORMANCE
1970 THORIA CAMPAIGN

Editors:

RR Jackson
RL Walser

March 1977

PREPARED FOR THE U.S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION UNDER CONTRACT E (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.

MASTER

DISCLAIMER

ARH-2127

PUREX
PROCESS OPERATION AND PERFORMANCE
1970 THORIA CAMPAIGN

Editors:

R. L. Walser

Purex Process Engineering
Engineering
Production and Waste Management

R. R. Jackson

Safety Analysis and Engineering
Safety and Environmental Analysis
Research and Engineering

Operated for the Energy Research and Development Administration
by the Atlantic Richfield Hanford Company under Contract E(45-1)-2130

TABLE OF CONTENTS

ABSTRACT

The Hanford Purex Plant fulfilled a 1970 commitment to the Atomic Energy Commission to produce 360 kilograms of high purity ^{233}U as uranyl nitrate solution. Overall plant performance during both 1970 and 1966 confirmed the suitability of Purex for processing thorium on a campaign basis. The 1970 processing campaign, including flushing operations, is discussed in this report with particular emphasis on problem areas. Background information on the process and equipment used is also presented. The organizations and their designations described are those existing in 1970.

INTRODUCTION

The Purex Plant chemical separations facility at Hanford is operated by the Atlantic Richfield Hanford Company for the Atomic Energy Commission as part of the chemical processing complex associated with the Hanford Reactors. Its primary function is the aqueous reprocessing of irradiated uranium fuel elements for the separation and purification of uranium, plutonium and neptunium.

However, since plant startup (January 1956), a number of programs have been undertaken to achieve added plant versatility. The programs have been developed with three goals in mind: to achieve increased plant capacity; to develop the capability to safely reprocess fuel of higher ^{235}U enrichments, and to establish flowsheets and techniques for the reprocessing of a variety of different materials. One achievement resulting from increased plant flexibility has been the successful reprocessing of irradiated thoria (ThO_2) target elements on a campaign basis for thorium and ^{233}U separation and purification.

The first Purex production scale thoria campaign was conducted in 1966. Approximately 165 tons of thorium and 220 kilograms of ^{233}U were recovered. The thorium and ^{233}U product quality met all targeted specifications except for the fission product content of the thorium product. The 1966 campaign was the culmination of an extensive developmental program. Flowsheet development work was conducted both with the Chemical Processing Department programs and through programs contracted to Battelle Northwest Laboratories. A process test involving thorough plant flushing and plant operability testing was conducted in Purex in January 1965, to provide operating and process control experience. On the basis of information obtained from the development work and the process test, a series of engineering studies dealing with criticality prevention safety, flowsheet and equipment capabilities and requirements were prepared.(45, 46, 47, 48) After four weeks of extensive plant modifications and flushing, thoria target elements were charged to the dissolver in May 1966. The campaign was completed in July 1966, and turnaround to uranium-plutonium production was completed in August 1966.

The second thoria campaign, conducted in 1970, was targeted to produce a minimum of 360 kilograms of ^{233}U for use in the Light Water Breeder Reactor (LWBR) program (Division of Naval Reactors). Extensive plant flushing was again required as the allowed ^{238}U content of 0.5 weight percent was equivalent to only 6.6 pounds of ^{238}U for the entire campaign. The success of this second campaign was, to a large extent, attributable to the experience gained in the preceding thorium processing operations. The process flowsheet and operations were quite similar to those employed for the 1966 campaign. A major contribution to the success of the campaign was made by ARHCO technical, operating and administrative personnel. Also, each of the Hanford contractors ensured the safe and timely conclusion of the 1970 Thoria Campaign by efficiently and effectively performing their individual functions in close coordination.

SUMMARY

The primary goal of the 1970 Thoria Campaign was the recovery of 360 kilograms (kgs) of high purity ^{233}U (as uranyl nitrate hexahydrate solution) to fulfill a commitment to the Atomic Energy Commission. Only minor modifications of the dissolution and solvent extraction flowsheet used during the successful 1966 thoria processing campaign were required. The campaign was successfully conducted and resulted in the recovery of about 600 kgs of ^{233}U from approximately 470 tons of irradiated thoria target elements. The overall recoveries based on reactor input data were 95.2 percent for uranium and 94.9 percent for thorium. Approximately 490 kgs of ^{233}U exceeded all product specification requirements except for ^{232}U content, which was determined by reactor operation. Thus, the production goal was exceeded by about 35 percent. Approximately 40 percent of the thorium product met all specifications.

Preparations for the 1970 Thoria Campaign were begun immediately preceding the shutdown of uranium-plutonium processing on June 8. Plutonium and neptunium levels were reduced by reworking uranium product and reducing solvent extraction waste losses to minimum values. Following the shutdown, most of the uranium, plutonium, and neptunium was removed from the processing equipment with low volume dilute nitric acid flushes which were concentrated and stored for future processing. Additional decontamination flushes were begun on June 30, and continued until the uranium and plutonium removal rates reached satisfactory levels on August 10. A total of 360,000 gallons of waste were transferred to non-boiling underground tank storage during these operations.

Extensive equipment modifications, including approximately 275 remote piping jumper changes, were required to changeover to thoria processing. Major equipment changes made for the campaign included the installation of two new concentrators (feed concentration-denitration and first cycle uranium product), a new downdraft condenser tower on the TK-A3 dissolver, and a new uranium product receiver-sampling tank. Extensive calibration and operability testing of all systems was conducted between July 23 and August 10.

The initial charge of non-specification (high ^{238}U) thoria to the TK-A3 dissolver was made on August 5 and startup of the solvent extraction cycles was first attempted on August 20. The initial charge of specification-grade material was made on September 26 and solvent extraction processing of this material was started successfully on October 13. Solvent extraction processing was completed on December 16. Post-thorium flushing was started immediately following the shutdown and continued until January 16, 1971.

The campaign, including turnarounds and processing, required about two months more than had been originally scheduled. The actual processing time was extended to four and one-third months versus the three months originally scheduled. About one month was lost due to equipment problems

and the remaining time was required primarily for unscheduled flushes of the solvent extraction equipment. The primary equipment difficulties were: poor dissolver vacuum; decomposition of pulser gasket material; and hydraulic flow problems. Small leaks in all three dissolver coils were also discovered following the campaign. The unscheduled flushes were required to remove solids, primarily thorium dibutylphosphate, causing unstable column operation.

Overall plant performance again confirmed the suitability of Purex for processing thorium on a campaign basis. Major improvements over the 1966 campaign were the superior dissolution characteristics of wafers; the superior performance of the E-F11 Concentrator for feed concentration-denitration; and the reduction of solvent degradation by decreased residence time in high acid environs.

The aluminum cans containing the thoria powder or wafers were dissolved by the conventional sodium nitrate-sodium hydroxide process and the waste solution was centrifuged to recover thoria fines. These fines were dissolved in a nitric acid-aluminum nitrate-potassium fluoride solution. A similar solution was used in the dissolvers to dissolve the major portion of the thoria. The solutions were then combined for concentration and distillation to produce an acid-deficient thorium nitrate solution as feed for the first solvent extraction cycle. The maximum attainable thoria dissolution rate was about twenty percent lower than that achieved in 1966 due primarily to damaged air-lifter tubes which resulted in inefficient circulation. The performance of the new feed concentration-denitration (E-F11) concentrator was far superior to that of the pot-type dissolver used for this purpose in 1966. However, ruthenium volatilized during this operation resulted in low-level contamination of the ground near the plant stack.

The solvent extraction flowsheet was based on the Thorex II(54, 57, 59, 61, 68, 71) developed at the Oak Ridge National Laboratory and development work conducted at other sites.(60, 62, 70) The first solvent extraction cycle was used for co-decontamination and partitioning of the thorium and the ^{233}U. Further decontamination of the products was attained in one additional thorium and two additional uranium solvent extraction cycles. A solvent consisting of 30 volume percent tributyl phosphate diluted with normal paraffin hydrocarbons ($n-C_{10}$ to $n-C_{14}$) and pulse column contactors were used in all cycles. The Third Uranium Cycle aqueous product stream (3BU) was also passed through a fixed bed of cation resin for thorium absorption prior to final concentration.

The performance of the solvent extraction flowsheet was satisfactory. An overall thorium-uranium separation factor of 8.0×10^6 was obtained for the ^{233}U product. The overall separation factor for the thorium product was 1.8×10^2, which reflected partitioning difficulties experienced during transient conditions in the 1BX Column. Overall fission product arithmetic decontamination factors follow the same pattern. The protactinium, zirconium-niobium and ruthenium decontamination factors (df's) were 3.5 x 10^6, 3.3 x 10^7, and 6.1 x 10^5, respectively, for the uranium product. The respective factors for the thorium product were ^{233}Pa, 1.8 x 10^2; $^{95}ZrNb$, 5.3 x 10^3; and ^{106}Ru, 1.1 x 10^2. As in 1966, solvent extraction losses were generally low except for the ^{233}U loss to the organic waste stream (1CW) from the first cycle uranium stripping column, and the Second Thorium Cycle ^{106}Ru and ^{233}Pa decontamination performance was poor.

Several problems were encountered in the Waste Concentration-Acid Recovery and Solvent Treatment Systems. The acid loss to the vacuum fractionator overhead condensate was abnormally high until the operating procedures were revised; the acid waste disposal rate was marginal; solvent losses via the organic wash waste were high throughout the campaign; and aqueous waste volumes were 35 to 50 percent greater than predicted. The major sources of the excess aqueous waste volume were the organic wash wastes (due to excessive washing or "spinning" of the solvent during down periods) and laboratory and miscellaneous wastes which were not included in the predicted value.

BACKGROUND AND PREPARATIONS

PROCESS TECHNOLOGY

Flowsheet

The flowsheet was essentially the same as that used for the 1966 Thoria Campaign.(32, 48) The solvent extraction flowsheet was basically a modified Thorex II process. However, both the Head End and Solvent Extraction flowsheets were the end results of extensive pre-1966 campaign development work conducted both in the Chemical Processing Department and at Battelle-Pacific Northwest Laboratories (BNW), Savannah River, and the Oak Ridge National Laboratory (ORNL).(39, 42, 43, 50, 54, 57, 59, 60, 61, 68, 70, 71)

The Head End process, as shown in Figures 2 and 3, consisted of three batch operations. First the thoria target elements were dejacketed (Figure 2). The aluminum cladding was chemically removed with sodium nitrate-sodium hydroxide, and the resulting liquid decladding waste was treated for product recovery. Second, the thoria powder (or wafer) was dissolved in concentrated nitric acid and complexed fluoride (Figure 3). Third, the resulting thorium nitrate solution was concentrated and denitrated until acid-deficient in a specially modified concentrator (E-F11-1). After assurance that the specifications for thorium concentration and acid deficiency were met, the feed solution was transferred to the first solvent extraction cycle feed tank.

The solvent extraction flowsheet is shown in Figures 4 and 5. A four-cycle process was required. Initially the thorium and uranium were co-extracted into the organic phase in the HA Column to obtain primary separation from other radioisotopes. The thorium and uranium were then partitioned in the 1BX Column by selectively stripping out the thorium. The uranium exited the 1BX Column with the solvent and was, in turn, stripped out in the 1C Column and concentrated to Second Uranium Cycle feed requirements. The thorium exited the 1BX Column in the aqueous, was scrubbed with fresh solvent to remove uranium in the 1BS Column, and concentrated to Second Thorium Cycle feed conditions.

The Second Thorium Cycle is shown in Figure 4. It consisted of an extraction operation and a back-extraction (stripping) operation, followed by concentration of the thorium nitrate to product specifications.

The Second and Third Uranium Cycles shown in Figure 5 consisted of two extraction back-extraction sequences. The uranyl nitrate solution was then routed to N Cell for further thorium decontamination by passage through a cation exchange bed, and concentration to uranium product specifications.

In addition to the above cycles, two solvent treatment systems (Figure 6) were used to clean up the solvent prior to recycle. The No. 1 Solvent Treatment System serviced the Co-Decontamination and Partition, Second Uranium, and Third Uranium Cycles. The No. 2 Solvent Treatment System serviced the Second Thorium Cycle.

The aqueous waste streams from solvent extraction processing were collected, concentrated, and acid denitrated. The recovered nitric acid was concentrated for re-use in head end dissolution (Figure 7).

The notable features of this flowsheet sequence were as follows:

1. One solvent was used in the four cycles - 30 volume percent tributylphosphate (TBP) in a normal paraffin hydrocarbon (NPH) diluent.

2. Although the feed was acid-deficient, the HA Column was operated acidic.

3. The 1BSU solvent stream was recycled to the HA Column rather than to the 1BXF.

4. Phosphoric acid was added to the HA Column via the HAS, as well as to the 2D Column via the 2DIS (when required), to enhance protactinium decontamination.

5. Ferrous sulfamate was added to the HA Column via the HAS (as well as to the 2D and 2A Columns via the 2DIS and HAS streams, respectively) to enhance plutonium and chromium decontamination.

6. Partitioning of the thorium from the uranium in the 1BX Column resulted from the low relative solubility of thorium nitrate in a TBP solvent in an environment of low salting strength and moderate temperatures.

Nuclear and Chemical Safety - Documentation

In preparation for the thorium campaign, a number of documents were issued relating the conditions necessary for safe processing of thorium and ^{233}U in the plant. The safety analysis report(16) reviewed the entire process and discussed methods of maintaining or regaining control of the process. Potential problem areas were pointed out along with the immediate effects and subsequent or downstream effects of abnormal operation. Areas requiring additional instrumentation, alarms, blanks or other safeguards were described. The criticality prevention specifications(15) and the process specifications and standards(12) used the safety analysis report as a basis for creating limits within which the plant would routinely operate.

The criticality prevention specifications were based on two necessary conditions; the first condition was the safety of the individual vessels under the worst foreseeable situation, the second condition was the safety of the downstream vessels under the worst foreseeable situation. The vessel limits were based either on the maximum permissible mass in the vessel or the maximum permissible concentration. The concentration limit was given either in terms of volume (grams ^{233}U per gallon) or in terms of thorium (grams ^{233}U per ton of thorium). The vessel limits were derived from the following values:

1.	minimum critical mass	590 grams
2.	minimum critical mass for precipitation	330 grams/square foot
3.	minimum critical solution concentration	11 grams/liter
4.	always safe ratio	5,000 grams ^{233}U/ton of thorium

The process specifications and standards established the limits within which the processing operations could be safely conducted while producing products attaining specified quality. Specifications and standards for feed, essential materials, and chemical hazards were provided. Further, the targeted ^{233}U and thorium product qualities were given. With respect to the feed, the primary concern was to insure by visual inspection and shipping records that uranium-plutonium fuel elements were not charged to the dissolvers. The essential materials specifications(33) defined those chemicals permitted in Purex for use in thorium processing.

The primary chemical hazards during thorium operations were possible hydrogen, ammonia, nitrated solvent or vaporized solvent explosions, fluoride corrosion, uncontrolled sugar denitration of waste, and inadvertent routing of acidic wastes to carbon steel underground storage tanks. In addition, specifications and control methods were provided to limit radiolytic heat loads on vessels and dissolver shell erosion by air sparging.

PROCESS CONTROL

The process control programs for Purex thorium operations called for instituting a number of system controls preliminary to plant operation. These controls were either required by nuclear and chemical safety considerations or implicit in such requirements. The principle items in the program were the process routing controls, the process sample schedule, the instrumentation modifications, and the Standard Operating Procedures (SOP's). Each of these items also contained features aimed solely at providing best methods for accomplishing the various tasks.

Process Routing Controls

Routing controls instituted for thorium processing consisted of installing nuclear safety blanks or process control blanks, and the locking and tagging of valves, jet switches, and power supplies to pumps.

A nuclear safety blank consisted of the physical removal of a section of pipe in a route. The primary purpose was to prevent either the addition of a precipitating agent to a vessel containing ^{233}U or the transfer of solution containing ^{233}U to an environment in which precipitation could occur.

The nuclear blanking requirements were included in the Criticality Prevention Specifications.(15) Prior to each startup during the thorium campaign, the routes were audited to assure compliance.

A process control blanking and lock and tag schedule(13) was also prepared and similarly audited prior to each startup. A process control blank was a "pancake" blank inserted between two flanges in a route. This control removed unnecessary chemical addition and process solution routings from service.

Those routes to be locked out and tagged were included as part of the process control blanking schedule. The lock and tag procedure consisted of locking out the manual valves on jet steam supplies and the jet switches, and tagging the jet switches. The shift supervisor or specialist signed and dated the tag. The lock and tag control method prevented routine usage of a route only occasionally needed.

Instrumentation

Thorium processing in Purex necessitated major modifications in existing instrumentation and installation of new instruments. Instrument modifications consisted primarily of range changes to accommodate the different process flows and conditions, and of changes in alarm set points. New instruments were installed for new equipment systems and to provide additional monitoring capability.

The nuclear and chemical safety analysis report(16) listed the criticality prevention alarm systems necessary to reduce the processing risk to acceptable levels. The alarm systems were to ensure that proper aqueous to organic flow ratios were maintained in columns downstream of the partition column; that gross overconcentration of ^{233}U solutions was prevented and that proper nitric acid concentrations were maintained. An alarm system was required on the 1C Column (T-H3) organic detector to warn of solvent escaping to the concentrator. Further, a high volume alarm on the ^{233}U rework tank was required to warn of impending overflow (due to a sump limitation).

Aside from instrument changes required for criticality prevention control, a major instrument calibration effort was required both for the new equipment systems and as a result of the different process parameters in the other equipment systems. The effort was centered on flow measurement-control devices, and weight factor-specific gravity instruments.(10) Also, in-line gamma monitors were adjusted to permit monitoring ^{233}Pa and ^{232}U (rather than $^{95}ZrNb$).(8)

Standard Operating Procedures

The thorium campaign required the preparation and issuance of approximately 215 SOP's. Of this number, about 50 SOP's covered the plant flushing before and after the campaign. The complete set of operating procedures proved invaluable during plant operations as a source of general information and as operating guides, particularly in sequencing startup or shutdown activities. Appendix E lists the SOP's by number and title.

Sample Schedule

The thorium processing sample schedule(9) (Appendix F) was established to provide data for:

1. Process and Criticality Safety Control;

2. Input and Output Accountability;

3. Product Quality Control; and

4. Low-Level Liquid and Gaseous Effluent Control.

The sample schedule specified the individual sampling points required, the analyses for each point, and the sampling frequency. It was divided into four sections -- routine stream samples, routine batch samples, aqueous makeup samples, and waste effluent samples.

The entire sample schedule was programmed into the IBM-1800 Computer located in the Purex Laboratory. Sample requests were made by the shift dispatcher from the keyboard in his office. One entry on the dispatcher's keyboard would provide the complete list of routine samples to be taken on the shift, with the date, time, required analyses and a number assigned for each sample. In addition, a data card giving the analytical method, dilutions, sample size and calculation method would be printed at the laboratory.

Batch, AMU and waste effluent sample requests were handled similarly. When the laboratory completed the individual analyses, the variables were entered into the computer and the results were calculated and transmitted to the dispatcher.

Some modifications were made to the sample schedule after the document was issued. The major change was the deletion of the gamma scan analysis for a number of samples. Plutonium, neptunium, tungsten and mercury analyses on the L9 product sample were made on every batch, rather than on the five-batch composite, to meet customer specifications. The sulphate analysis on the L9 composite was eliminated.

A description of the primary analytical methods and the laboratory quality control program is included in Appendix G.(14)

PLANT PREPARATIONS

Preparation of the Purex Plant for the Thorium Run consisted of six major parts:

1. Pre-Shutdown Activities;

2. Plant Shutdown;

3. Product Recovery Flushes;

4. Equipment Change-Out;

5. Final Decontamination Flushes; and

6. Plant Operability Testing.

Pre-Shutdown Acitivites

The recovered acid system, which usually contains some uranium from nitric acid recovered at the Uranium Oxide (UO_3) Plant, was purged of uranium. The purge was accomplished by routing the UO_3 acid stream directly to the dissolvers, rather than "into" the Purex recovered acid system. This necessitated imposing special procedural controls to make sure the UO_3 acid always met tne dissolver acid specification. The purge started in mid-December 1969. As the recovered acid system inventory turned over, the uranium content dropped from a normal of 200-500 pounds to less than 20 grams at shutdown in June 1970.

Plans were made early to isolate the alternate plutonium concentration equipment (L Cell Package) from the system. The L Cell Package and the sump were flushed to remove most of the plutonium which was reworked into the current production. As soon as the plant was shut down, the normal 2BP connection to the L Cell Package was modified and blanked. This allowed the L Cell Package to be removed from the Thorium Flushing Plan.

Tank F8 (waste rework) was emptied, flushed, and was held empty so that it could be used to store the 3WB (backcycle waste) inventory and the plant product flushes.

Plant Shutdown

The plutonium and neptunium inventory in process was reduced to a minimum at shutdown. This was accomplished by feeding sufficient cold uranium to permit extensive heel cutting in the dissolvers and head end tankage to remove the majority of the uranium, plutonium, and neptunium. The processing of "cold" feed also reduced the plutonium content in the Partition and Second Plutonium Cycles. The ion exchange system also was partly stripped. Just as cold feed processing was started, a decontamination and concentration sequence (Phase II and III) was started in the Neptunium Cycle to remove the maximum amount of neptunium from the backcycle systems. When the HAF was shut off and the columns stripped, there was little uranium, plutonium or neptunium which ended up in the backcycle waste system due to operation with negligible losses in second cycle extraction columns after fission product activity was lowered by processing cold feed. The last full product batches were within specifications. The only out-of-specification plutonium loaded out resulted from dilute "heel" cleanouts. As soon as the HAF was shut off, the waste system inventory was denitrated and disposed to interim boiling waste storage (AR Vault) for B Plant processing.

Product Recovery Flushes

After the completion of normal waste processing, three low volume (1,000 gallon) water flushes were made from the HA Column through the high-level waste system (TK-F7, E-F6, TK-F26, TK-F15, TK-F16). The last of the water flushes was sampled and found to be low in cesium content. This was the last waste sent to boiling waste (AR Vault).

A caustic-tartrate flush and a water flush of the HA Column were sent through the high-level waste system to non-boiling waste. These flushes prepared the high-level waste system for the concentration and storage of product removal flushes.

The product removal flushes consisted of repeated dilute nitric acid flushes of equipment groupings (head end) and individual solvent extraction cycles until the product values were low. The flushes were all collected in TK-F10 or TK-F7, and then boiled down in the 1WW concentrator. This system was chosen for boil-down because much of the nitric acid could be boiled off and recovered in the acid absorber. The product flushes were boiled down to a volume which would fit in one tank, TK-F8. After the solution was moved to TK-F8, small water flushes followed to purge equipment of heels. The TK-F8 contents had to be boiled down to accommodate the last of the water flushes.

Equipment Change-Out

The Backcycle Waste Concentrator was known to contain silica solids which could not be dissolved with flush solutions compatible with stainless steel.(41) It had been decided in advance to replace this concentrator with a new sectionalized concentrator. The old concentrator was removed after extensive flushing with dilute nitric acid verified a minimal vessel inventory of plutonium.

The down draft condenser tower on dissolver A3 was replaced with a tower which had been cleaned up and modified. The vapor outlet was previously low on the dissolver and was known to have contaminated the tower which could not be decontaminated in place. When the tower was replaced, the normal A3 vapor outlet was blanked and a vapor outlet was installed from the dissolver lid to the down draft tower.

A new product loadout system was installed during the flushing period. The old product sample tank (TK-L9) was removed and a new three-barrel vertical sample tank was installed.

The movement of approximately 275 remote piping (Appendix C) jumpers was required to prepare the plant for Thoria processing. Also, numerous changes were required in the chemical addition systems.(16)

Final Decontamination and Cleanup Flushes

The final removal of uranium from the head end vessels was accomplished with potassium fluoride-aluminum nitrate in concentrated nitric acid. The flush approximated the starting chemical conditions of thoria dissolution. Vigorous sparging and long-time exposure were essential. The perforated cone sections of the A3 and B3 dissolvers were flushed with water near the end of the flush period. A special jumper which adapted to the holes near the top of the cone was used. No measurable quantity of uranium was found, indicating that the cones had been self-flushing during earlier boiling flushes or the uranium was in immobile solids. The low bleed-in rate during processing would indicate the former condition was true.

The strong nitric acid-fluoride flushes used in the dissolvers were also used to flush out the feed system tanks (Tanks D3, D4, D5, E6 and H1). Some of the solution was also sent through the coating waste treatment system (Tanks D2 and E3, and centrifuges G-E2 and G-E4).

The solvent extraction cycles were flushed individually using a sequence of flushes consisting of a five percent oxalic acid - three percent nitric acid solution, followed by a water flush, then a five percent caustic - two percent sodium tartrate solution, followed by a water flush. The acidic flushes and water flushes were all accumulated and processed before starting the caustic flushes. Samples of the individual flushes indicated that the 3WB Tank (TK-J1) and the neptunium cycle feed tank (TK-J21) required additional flushing. The uranium was attributed to siliceous solids known to accumulate in these tanks. A special metathesis-type flush was used on these vessels. The flush consisted of digesting a sodium hydroxide-sodium carbonate solution in the tank. Then, after jetting out the basic solution, a dilute nitric acid solution was added to dissolve the soluble compounds formed by the metathesis solution. The same solution was routed through TK-F12. This flush removed 1WW solids which accumulated when TK-F12 was previously used for 1WW rework storage.

The N Cell equipment was flushed solely with nitric acid. The N Cell flushing was discontinued after the plutonium level dropped to less than 0.1 gram in a flush.

Operability Testing

After the completion of chemical flushing and equipment/piping changes, the process equipment was operability tested. This shakedown run consisted of recalibrating all flow measurement devices, operating all equipment at thorium rates, and conducting a sampling program designed to ascertain the remaining ^{238}U levels. Most of the problems encountered were easily solved. However, a few required major engineering efforts to correct.

The A3 dissolver vacuum proved to be marginal due to the improper seating of the vent jumper when hot. Weights were used as a temporary means of seating the jumper. A new jumper solved the problem.

Trouble with the 1BXT air lifter was encountered. Initially, the system would not work at all. After repairing leaks and correcting a number of mistakes in the jumper itself, marginal 1BXT flowrates could be maintained.

The E-F11 concentrator system was initially incapable of achieving the necessary heat transfer rate. The problem was easily solved, however, by increasing the tube bundle submergence.

Throughout the operability test, the ^{238}U levels in the process samples were acceptably low.

PERSONNEL

Thorium processing in the Purex Plant required additional manpower, major changes in manpower utilization, and intensive personnel training programs. Increased process monitoring and additional operations external to Purex accounted for the increase in manpower requirements. The changes in manpower utilization were governed by the number of operations, the structure of the process, and the length of the campaign. The intensive training programs were necessary to acquaint personnel with the changes in nuclear and chemical safety requirements, modifications in equipment functions and relationships and the new operating and control techniques for thorium-^{233}U production.

Staffing Manpower Requirements and Manpower Utilization

Three groups were essential to the Purex operation; the Purex Operations, Purex Maintenance, and Technical Services' Purex Analytical Laboratory Subsections. The Separations Process Engineering Section provided technical support and assistance to these subsections. The Purex Operations Subsection consisted of four rotating shifts and a fifth straight-day shift. The Purex Maintenance Subsection included electricians, instrument technicians, pipefitters, millwrights and painters. Individuals from the first three crafts above were assigned to shifts. The Purex Analytical Laboratory was also organized to provide services on each of the rotating shifts. In addition, the fifth or day shift had several chemists responsible for development and review of analytical techniques and analysis of special samples. The Separations Process Engineering Section was organized into three teams for the process control, process technology, and new processes functions, respectively. In addition, other individuals in the group specialized in nuclear safety and process control instrumentation.

Staffing and manpower utilization adjustments were made in each of the above organizations. The ^{233}U product transloading operations at U Plant and partial coverage at the thorium product storage facilities (WR Vault at U Plant and the 204 Tank Farm at Redox) required five operators and a specialist on day shift. The number of operators assigned to the various control rooms was also increased to compensate for increased activity, notably in Feed Preparation and Waste Treatment. Further, specific operators were assigned to certain critical operations for the duration of the campaign. This contrasted to the normal practice of rotating the operators throughout the plant during uranium-plutonium processing to develop a diversity of skills and avoid job monotony. Due to the brevity of the thorium campaign and the complexity of the new process, operator specialization seemed to be the best way of rapidly achieving operator proficiency.

The Purex Analytical Laboratory added two analysts to each shift to handle the extra sample load. The chemists assigned to day shift provided supplemental shift coverage as necessary, as well as troubleshooting specific analytical problems. The Maintenance Subsection made no personnel adjustments specifically for thorium operations. The Separations Process Engineering Section did not require additional personnel above the normal requirements; however, the process control coverage on shift was augmented with four engineers from the other two teams. This additional shift coverage was discontinued in mid-November.

Training

Training programs were conducted within the Purex Operations and Technical Services' Purex Analytical Laboratory Subsections and the Separations Process Engineering (SPE) Section. The Operations training program for the thorium campaign supplemented the normal operator training status check sheets for recording progress in the thorium training program. As was the case in the training program for uranium-plutonium operations, the primary educational technique was the informal supervisor-operator contact. In addition, several lectures were prepared covering the campaign goals and purposes, the process, process control and troubleshooting, and chemical and nuclear safety. The lectures were prepared and delivered by Operations and SPE personnel. However, rather than repeat the lectures for each of the five shifts, the series was recorded on video-tape. The video-taped, two-hour program was then shown to the various shifts at the discretion of the shift supervisors. The program was an outstanding success by virtue of the general exposure. Further, the tapes permitted repeated operator exposure to the program and were re-shown several times on individual shifts.

The Purex Analytical Laboratory training program consisted of a series of lecture sessions over a one-week period (Appendix H). The lectures dealt primarily with the analytical tools and techniques required for analyzing thorium process samples. A portion of the program relating to the thorium flowsheet and sampling requirements was prepared and delivered by SPE engineers.

The Separations Process Engineering training program consisted of a series of weekly, informal seminars attended by the SPE engineers and representatives from the Operations and Laboratory Subsections. Fourteen sessions were held. The flowsheet, process operation and control, and chemical and nuclear safety were reviewed. Aside from the educational aspects, the discussions were invaluable as a final evaluation and critique of the proposed flowsheet and process.

RUN REPORT

THORIUM PROCESSING CHRONOLOGY

The following chronology lists the sequence of significant occurrences preceding, during, and following thorium processing in Purex. Preparations for the campaign required two months. Post-thorium turnaround required about one month, disregarding non-thorium related maintenance work. The campaign itself consisted of three segments; an initial ^{238}U purge with "non-specification" (high ^{238}U) feed, a middle "Division of Naval Reactor (DNR)" or "specification" run, and a terminal period of miscellaneous scrap processing.

Processing was interrupted on four occasions by the necessity to shut down and flush the solvent extraction system with concentrated nitric acid. The campaign lasted about four and one-third months, from first charge to final stripout and shutdown.

Chronology

6/5/70	Commenced solvent extraction shutdown of uranium-plutonium processing.
6/8/70	Plant shut down and stripout completed; began product flushing of entire plant.
6/30/70	Chemical flushing of solvent extraction systems started.
7/1/70	Product flushing completed; product flushes and backcycle waste inventory concentrated and stored in TK-F8.
7/23/70	Began plant operability testing.
8/5/70	Charged non-specification thoria powder to A3 Dissolver; had trouble maintaining A3 vacuum.
8/6/70	Charged non-specification thoria powder to B3 Dissolver.
8/7/70	Charged non-specification thoria powder to C3 Dissolver.
8/10/70	Completed concentrated nitric acid - ANN-KF flushing of feed tanks. Completed operability testing of processing systems.
8/12/70	First feed batch prepared in feed concentrator (E-F11); did not meet feed specifications due to poor concentrating efficiency.
8/15/70	Increased feed concentrator batch size to 2,500 gallons; no further problems.
8/20/70	Attempted to start up solvent extraction. Shut down due to 1BU organic siphoning via vessel vent header to a waste tank (TK-F18).
8/22/70	Solvent extraction processing recommenced; 1BXT flow limiting capacity factor (CF) 0.85 to 0.90.

8/24/70	Shut down due to major flood in 1C Column. Commenced solvent recovery and flushing of the 1CU Concentrator - Second Uranium Cycle feed system.
8/26/70	Loaded out first ^{233}U product solution. Resin in samples.
9/1/70	Began concentrated nitric acid flushing of Partition Cycle and Second Uranium Cycle.
9/6/70	Found faulty screens in uranium product cation exchange column (T-N50), commenced building a new column.
9/6/70	Solvent extraction processing re-started.
9/7/70	The 2D Column was unstable; gelatinous solids were observed in the 2DW. Flushed 2D and 2E Columns without shutting down the rest of the plant. Discontinued use of phosphoric acid in the 2DIS.
9/8/70	Started Second Thorium Cycle, no further problems.
9/15/70	Shortened dissolver cut time to 16 hours with some increase in dissolver heels.
9/22/70	The HAF stream was off for two and one-half hours to replace a failed feed pump.
9/24/70	A flood in the 1C Column was corrected by shutting down the HA Column for five hours. A partition failure resulted from the prolonged stripping of the 1BX Column.
9/26/70	Charged first specification grade ^{233}U-thoria powder targets to dissolvers. Began reworking TNT product solution high in ^{233}U.
9/27/70	Shut down HA Column at depletion of rework TNT solution. The 1C and 2A Columns flooded prior to shutdown.
9/29/70	Found two "E" metal slugs (irradiated ^{235}U-^{238}U fuel elements) on cask cars. Returned to reactor area.
10/1/70	Stripout and shutdown complete. Started concentrated nitric flushing of solvent extraction equipment. Also started flushing N Cell and solvent treatment systems.
10/7/70	Initiated startup activities. Terminated startup due to a failed 1BX Column pulser motor.
10/13/70	Recommenced startup on "specification" feed. Placed cation exchange column (T-N50) on-line.

10/14/70	The HAF was off for five hours to repair leak in 1BXF jumper. After 13 hours of operation, HAF shut off again to replace failed 1WF pump.
10/15/70	Turned HAF back on; lost partitioning in the 1BX Column due to the low thorium level.
10/16/70	Shut down Second Thorium Cycle to segregate TNT product solution high in ^{233}U. During re-startup, dropped solvent from 2E Column to TNT Concentrator.
10/17/70	Began reworking TNT product solution high in ^{233}U via feed makeup tank (TK-E6).
10/19/70	Recovered solvent from thorium product tank (TK-K6) for cleanup.
10/21/70	Shut off HAF due to loss of 1BXT flow. Began a concentrated nitric flush of the partition cycle.
10/23/70	Removed and unplugged 1BXT jumper, made two unsuccessful attempts to start up, but could not maintain 1BXT flow.
10/26/70	Pulled 1BX Column pulser and found that the Viton A* gasket was falling apart. Replaced with a Viton B* gasket sheathed in Teflon*. Installed a modified 1BXT jumper; with strainer and larger DOV trim.
10/29/70	Started back up, with minor stability problems in 1C and 2B Columns.
10/30/70	The 2A Column flooded.
11/1/70	Increased dissolution time to 18 hours.
11/3/70	First wafer charge to C3 Dissolver.
11/6/70	Shut down for a scheduled outage; began concentrated nitric acid flushing of solvent extraction equipment.
11/11/70	Began solvent extraction startup. 2BU jet plugged. Began flushing 2BU line.
11/12/70	Recommenced startup; airlock in 1BU line resulted in 1BU being routed to waste tank (TK-F18) via the vessel vent header.

---

*Trademark of E. I. DuPont Nemeurs and Company.

11/13/70	Stability problem with 2B Column. Instrument problems resulted in high 2D Column losses for 16 hours.
11/16/70	Reduced the L/V in the 1BX Column in an unsuccessful attempt to drive more ruthenium into the 1BU stream.
11/19/70	First C3 jetout jumper failure.
11/23/70	Organic-aqueous disengaging problems arose in #2 Solvent System. Shut down the Second Thorium Cycle for 13 hours and flushed R Cell.
12/2/70	Completed wafer processing in C3 Dissolver.
12/3/70	Began butting feed with TNT product solution to maintain solvent extraction rates.
12/6/70	Made first charge of "non-specification" thoria to dissolvers.
12/9/70	Commenced adding phosphoric acid to 2DIS as a result of the higher fission product activity.
12/12/70	Last feed solution taken from dissolvers.
12/16/70	Completed shutdown of plant. Began post-thorium flushing.
1/16/71	Post-thorium flushing of solvent extraction, solvent treatment, and product handling systems completed.
3/27/71	First uranium charge to dissolver.

TARGET HANDLING AND CHARGING SEQUENCE

In contrast to the 1966 Campaign, two types of targets were charged to the dissolvers. The bulk of the thoria was in a powder form, in elements identical in content and configuration to those processed in 1966. In addition, approximately 58 tons of "wafers" were processed to evaluate wafer dissolution characteristics. Thoria target shipments from reactor basins to Purex were handled in the same manner as in 1966 with the exception that buckets containing wafer material were specially marked(21) and charged to a specific dissolver (TK-C3). The charging sequence was established on the basis of ^{238}U content, ^{232}U content, length of time since discharge, and nature of the targets (i.e., whether powder or wafer material).(2, 6, 25)

Target Element Physical Description

The target element characteristics(30) are given in Table III in Appendix L. The thorium powder elements were aluminum cans filled with sand-like particles ranging in size from 325 mesh to six mesh. The bulk density of the vibratory compacted powder was about 7.5 grams per cubic centimeter. The thorium wafer elements were aluminum cans filled with three wafers. Each wafer was of right cylindrical geometry, approximately 1.5 inches in diameter by 2.8 inches long, with a bulk density of about 7.6 grams per cubic centimeter. The wafers were fabricated from thorium powder through a process of cold-pressing and sintering. The wafer targets were designed to hold their original geometry in the dissolver after the aluminum cans had been removed, and hence, dissolve faster.(31) Subsequent dissolver experience indicated that this indeed was the case.

Target Element Handling and Special Precautions

Though thoria target elements were delivered to the Purex Plant in the same general manner as uranium elements, a number of important differences existed to insure that uranium fuel elements were not included. The thoria elements were loaded into the shipping buckets vertically in two layers instead of horizontally as with uranium fuel elements. With the elements in a vertical position, the ends of each layer were inspected at the reactors, and the ends of the top layer were inspected prior to charging by the Purex crane operator. Since the thoria elements had solid ends, any uranium fuel element with a hole in the center of the ends would have been readily identifiable. Photographs of each layer were taken at the reactors during bucket loading. The top layer was photographed again immediately prior to shipment to Purex (Photo 1 and 2).

Several partially loaded buckets were processed during the campaign. These buckets had an incomplete layer of thoria elements which allowed the elements to fall from their verticle position. Prior to loading into cask cars for shipment, each bucket was inspected by an observer from Purex to determine that the partial layer contained only thoria fuel elements. This requirement resulted in occasional delays of up to two hours in dissolver charging due to scheduling problems.

The cask cars used for transporting the buckets to the Purex Plant were the same as the cars used for uranium fuel elements. However, the cars were designated as containing thoria elements by red "T" placards placed on the ends of the car and on the lids of the three wells. In addition, each bucket containing wafer elements was identified with a bright green tube placed diagonally across the top of the exposed ends of the top layer of wafer elements. The transfer papers for each thoria shipment were marked "Thoria". Responsible Purex personnel checked the key number listed against the stock report provided by ARHCO Production Planning, and the reactor basin station number against the Bucket Loading Summary to determine that the car contained thoria.

The Purex crane operator was required to inspect each bucket before charging it into the dissolvers. The crane operator was also instructed not to retrieve any elements accidentally dropped from the bucket onto the canyon deck or into the dissolver cell during charging due to the possibility of picking up an uranium fuel element.

The only significant occurrence during thoria target handling was the isolated case of finding two uranium fuel elements in one car well. The car was returned to the shipper who removed the uranium elements. The potential for product contamination in this case was low since the uranium elements could not have been charged.

Thoria Charging Schedule

Because of the wide variation in the ^{233}U, ^{238}U and protactinium contents of the irradiated thoria, a computer program was successfully developed and used to optimize the charging schedule of the thoria for both the "non-specification" (high ^{238}U) and "specification" (low ^{238}U) portions of the 1970 Thoria Campaign. A detailed listing of the planned charging sequence for both powder and wafer material is presented.(6) This schedule was followed with only minor deviations and resulted in progressively reducing the ^{238}U content of the input thoria during processing of high ^{238}U material from 0.7 to 0.3 percent (based on ^{233}U) while maintaining the ^{233}U content below criticality prevention specification limits. During processing of "specification" thoria, relatively constant levels of ^{232}U and ^{238}U contamination were maintained in the feed as shown below:

SPECIFICATION THORIA FEED

Contaminant	Overall Average	Charge		Composite*		Maximum Specification
		High	Low	High	Low	
**^{232}U (ppm)	7.78	8.08	7.23	7.93	7.54	8
^{238}U (% ^{233}U)	0.13	0.15	0.10	0.14	0.12	0.5

---

* 3 Charge Composite (overlapping)

** Based on predicted values from the reactor. Actual average in the product was 8.63 ppmp ^{233}U.

Thus, the charging schedule permitted the production of a uniform ^{233}U product with respect to ^{232}U and ^{238}U content. It also minimized the protactinium content of the thoria feed so as to limit the degree of solvent degradation experienced during the campaign.

Following the "specification" portion of the campaign, an additional eight tons of short cooled-high activity thoria powder elements were dissolved in three charges. The most recently discharged material contained 70 grams of ^{233}Pa in 0.8 tons of ThO_2. This material was processed to empty the reactor storage basins of this type material.

TARGET DECLADDING AND DISSOLUTION

Figures II and III in the Appendix represent the flowsheets for target decladding, decladding waste treatment, and thoria (ThO_2) dissolution. To increase plant capacity, three dissolvers were used versus the two used in the 1966 campaign. However, the targeted rate of 1.0 CF (10 tons per day) was not met. Further improvement in the Head End rate was achieved by processing approximately 58 tons of thoria (ThO_2) wafers.

Operating and Process Control Description - Thoria Powder

The thoria targets (approximately 3.2 tons ThO_2 per charge) were dumped into sufficient 1.9 $\underline{M}$ $NaNO_3$ to cover the charge, and the dissolver contents were heated to boiling. Sufficient 19.0 $\underline{M}$ caustic was added at a controlled rate to achieve a caustic-to-aluminum mole ratio of 2.25. The dissolver contents were digested at boiling temperature for two hours to complete the aluminum-cladding removal. The decladding solution was then cooled and jetted to the decladding waste treatment system where the solution was centrifuged to recover any thoria particles entrained from the dissolvers. The resulting thoria cake was washed with 8 $\underline{M}$ caustic and then with water. The cake was slurried from the centrifuge to a digest tank with four batcnes of concentrated nitric acid. The resulting slurry was butted with fluoride (KF) which was complexed with aluminum nitrate. A six-hour digest at 70 °C proved adequate to dissolve the thoria. The decladding solution which passed through the centrifuge and the cake washes was discarded to a non-boiling waste underground storage tank.

After aluminum de-jacketing, the thoria powder was dissolved with a nitric acid-potassium fluoride-aluminum nitrate solution. The potassium fluoride was added in two increments; the first at the beginning of the dissolution, the second after two hours of digestion. The aluminum nitrate used to complex the fluoride was also added in two increments; the first prior to starting the dissolution, the second at completion of the dissolution. After about 16 to 18 hours of digestion at boiling temperatures, the dissolver contents were cooled and jetted to lag storage tanks prior to concentration to meet feed requirements.

Head End Performance and Recovery Efficiencies - Thoria Powder

The length of time required to process a powder charge was critical to maintaining the plant rate. Caustic de-jacketing of aluminum-clad elements is a standard Purex operation, and the normal three and one-half hour digest period proved to be adequate. Powder dissolution, however, required anywhere from 16 to 48 hours. During the latter half of the campaign, the dissolution time was arbitrarily set at 18 hours. An extra dissolution step (1,000-gallon heel cut) then proved to be necessary every third or fourth charge to dissolve residual thoria. The shortest cycle time (time required for complete processing of one charge in a dissolver) for a powder charge was about 30 hours. Most charges required between 30 and 35 hours. The extra heel dissolution step added 12 hours to the dissolver cycle. The decladding waste treatment cycle, which required 12 hours, excluding the digestion step, did not affect the overall dissolver cycle. Two centrifuge cakes were generally combined for the six-hour digestion period.

Material balances were calculated for powder charges after establishing the arbitrary 18-hour dissolution period. During dissolution, approximately 75 to 80 percent of the thoria charged was dissolved. Another five to 10 percent was recovered during decladding waste solution centrifugation.

Problem Areas - Thoria Powder

1. Low Dissolution Rate

Based on reported dissolution rates for 1966, a 20 percent decrease in rate per dissolver occurred. As a result the targeted 1.0 CF overall rate was not achieved. The failure to attain the dissolver rate achieved in 1966 made the Head End operation rate limiting for the plant. During the latter half of the DNR run (in December), recycle of thorium product solution was necessary to maintain the solvent extraction rates.

Recent laboratory tests have shown that the dissolution rate of thoria powder from the 1970 Thoria Campaign was equivalent to that of thoria powder processed in 1966. Thus, the slower thoria powder dissolution rate observed in the 1970 plant campaign compared to 1966 was probably caused by another factor. Pictures of the air-lift circulators in the dissolvers confirmed our suspicions that the circulators in the dissolvers were not functioning properly during the 1970 campaign (Photo 3). Many of the circulator tubes were out of position and appeared to be inoperable.

2. TK-A3 Dissolver Vacuum

Tank A3, which had a new off-gas line installed from the dissolver lid to the cooling tower, experienced vacuum problems during boiling periods because the off-gas line from the dissolver had no expansion bellows. The distortion of the off-gas line caused leaks around the flanges and reduced vacuum in the dissolver. Weights on the off-gas line were used to produce a barely satisfactory vacuum in the dissolver. Near the end of Thorium Operations, a newly designed off-gas line was installed. With this new off-gas line, TK-A3 vacuum was more than adequate, even when TK-A3 contents were boiling with sparging.

3. Jetting Problems

Few problems arose during decladding waste jetting. Jetting the dissolved thorium was frequently hindered by plugged jets, however. The jet dipleg usually was unplugged either by back-flushing or with a hydraulic ram. Occasionally an alternate route had to be found and a heel cut was required to free the dipleg.

On four occasions, thorium solution jet-out jumpers failed. In all four instances, a hole was found in a 90° elbow immediately downstream of the jet. The first jumper to fail (TK-C3 jet-out) was replaced with a jumper of similar design, which eventually failed as well. Within two weeks, failures occurred at all three dissolvers. Tentatively, the failures were attributed to a combination of erosion and corrosion. The impinging thoria particles appeared to erode away the passified metal. The thorium solution remained corrosive, despite the three-or-four-to-one aluminum nitrate to fluoride mole ratio. The jet-out jumper was redesigned with a longer straight run of pipe between the jet discharge and a more gradual elbow curvature. The replacement jumpers of the new design performed satisfactorily for the remainder of the campaign.

Thoria Wafers

Approximately 58 tons of thoria wafers were processed through the C3 Dissolver during the "specification" portion of the campaign. The thoria wafers were processed through Head End using the same flowsheet and operating parameters used for powder except for changes made in the sparger operation during the dissolution step as described below. The wafers dissolved much more rapidly than the powder. The initial dissolution time of 18 hours was gradually decreased to between 10 and 12 hours without any significant heel buildup. With a dissolution time of eight hours an extra heel dissolution was necessary every fifth or sixth charge. For a 10 hour dissolution time, the overall cycle for thoria wafer processing was about 25 hours or equivalent to a 1.15 CF rate.

Sparger Operation

The mode of air lifter circulator operation was quite critical to the thoria dissolution efficiency. The dense particles had a pronounced tendency to pile around the bottom of the dissolver. The sparge rates given below were similar to the rates used in 1966. The maximum air velocity leaving the sparge ring was again limited to 50 feet per second. Control of the air flow was maintained by the use of an orifice and by monitoring the flow and back-pressure indicators for each dissolver.

SPARGER RATES

Step	Inner Sparger	Outer Sparger
Coating Removal		
Charging	OFF	OFF
Heating and Digestion	300 pph* (steam)	50 scfm** (air)
Cooling	50 scfm (air)	100 scfm (air)
Jetting	35 scfm (air)	35 scfm (air)
Powder Dissolution		
Heating or Cooling	200 scfm (air)	75 scfm (air)
Dissolving		
Normal - 60 minutes duration	240 scfm (air)	75 scfm (air)
Redistribution - 15 minutes	50 scfm (air)	200 scfm (air)
Holding, Sampling or SpG Check	50 scfm (air)	50 scfm (air)
Wafer Dissolution		
Heating or Cooling	100 scfm (air)	100 scfm (air)
Dissolving	50 scfm (air)	200 scfm (air)
Holding, Sampling or SpG Check	50 scfm (air)	50 scfm (air)

---

* pounds per hour

** standard cubic feet per minute

The sparge rate was lowered during wafer dissolution because the dissolution rate increase, due to reduced turbulence at the wafer surface, more than compensated for the rate decrease resulting from less efficient mixing.

Dissolver Off-Gas Treatment

During the decladding operation, the off-gas stream passed through a downdraft condenser, an ammonia scrubber tower, a heater, silver reactor, and a high efficiency filter to the 200-foot Purex stack. During the dissolution operation no water was added to the ammonia scrubber tower and the off-gas, after leaving the filter, was also padded through the two absorber columns located in the Purex Backup Facility. A continuous monitor normally used to detect iodine in the dissolver off-gas stream was adjusted to permit detection of ruthenium.

The absorber columns, normally used to recover nitric acid from the off-gas stream, were operated on reflux water alone during the thoria campaign due to the low nitric acid content of the off-gas. Operation in this manner resulted in some ruthenium decontamination as evidenced by analyses of reflux water samples. Fresh demineralized water additions were made when the tank contents were changed out or to replace evaporation losses.

FEED CONCENTRATION AND DENITRATION

The flow sketch and the detailed chemical flowsheet for the Feed Concentration and Denitration Operation are presented in Figures 1 and 3 in the Appendix. The major variance from the original flowsheet(8) was the increase in concentrator operating level from 2100 to 2500 gallons. This change was required to increase the heat transfer area utilized and thus obtain the desired concentration rate.

Operating and Process Control Description

Feed to the concentrator (E-F11) was composed of dissolver solution from the lag storage tanks (TK-D1 or TK-D3) and dissolved thoria recovered from the decladding waste. These solutions were transferred to TK-D4 by steam activated jets for blending and were subsequently pumped to the concentrator. The initial 2300-2400 gallons was transferred in one batch and the remaining solution was transferred continuously to maintain the concentrator operating level at 2500 gallons.

In the concentrator, the thorium concentration was increased from the initial value of 0.5 to 0.7 $\underline{M}$ to a final value of 3.0 $\underline{M}$. Thus, up to 15,000 gallons of blended solution were required to complete one concentrated feed batch. The amount of thorium accumulated per batch was controlled by material balance calculations based on sample analyses and volume measurements. As secondary controls, the endpoint temperature and specific gravity guideline values were established at 135 °C and 2.35, respectively.

At that point the solution still contained most of the input nitric acid. A continuous water addition was then made to the concentrator at boiling conditions to remove the nitric acid by steam stripping while maintaining a constant volume of solution. In this manner, the solution was made

0.20 M acid deficient. The amount of acid removed was primarily a function of the amount of water added which could be determined by multiplying the addition rate by time or from the dilution water add totalizer. As secondary controls, guideline values for the terminal temperature and specific gravity at boiling were established at 115 °C and 2.15, respectively.

The concentrated acid deficient feed solution was then transferred while still hot (about 75 °C) to the receiver tank with a submerged jet which used demineralized water to supply the motive force. The water also served to dilute the solution to the desired thorium concentration. The transfer had to be conducted at an elevated temperature to prevent solidification of the solution which had a freezing point of about 55 °C. For this reason, the approximate 120-gallon heel remaining in the concentrator after each transfer was immediately butted with dilute feed or water.

The receiver tank contents were transferred to the Accountability and Feed Adjustment Tank (TK-D5) for sampling and adjusting to the desired 1.57 M thorium, -0.105 M nitric acid concentrations. From there, the feed was pumped to the feed storage tank (TK-E6) and then transferred to the solvent extraction feed (HAF) tank (TK-H1) with a steam motivated jet. The desired HAF thorium and nitric acid concentrations were 1.5 M and -0.10 M, respectively. Small volumes of 19 M sodium hydroxide or 12.2 M nitric acid were added as required to adjust the acid concentration in TK-D5 or TK-E6, respectively.

The concentrator off-gas stream passed through a demister pad and a condenser to the vessel vent system. The demister pad reduced the radionuclide content of the off-gas and aluminum nitrate was added via the upper spray nozzle to complex any free fluoride on the pad. The condenser condensate was routed to the Acid Recovery System but an alternate route to the second cycle waste receiver tank (TK-F10) was provided for use if the radionuclide content became unacceptably high.

Normal Operating Performance and Capacity

The feed concentration and denitration time cycle was adequate to provide feed for sustained solvent extraction operation at a 1.5 CF. Typical heat transfer coefficients (U values) ranged from about 170 BTU/hour-ft^2-°F at low thorium concentrations to 115 BTU/hour-ft^2-°F during the denitration step. The typical TK-D5 thorium and nitric concentrations were 1.4 M and 0.1 to -0.1 M, respectively. As a result, a butt of ten-to-twenty gallons of 19 M sodium hydroxide to TK-D5 was usually required to adjust the nitric acid concentration.

Problem Areas

1. Low Concentration Rate

The initial concentration or boil-off rate attained was too low to support sustained operation at the desired processing rate. Investigations revealed that the heat transfer coefficients calculated during this period were very low. The problem was apparently caused by poor circulation of solution within the concentrator due to operation with a low percentage of the tube bundle covered by solution. The concentration-denitration rate increased to an equivalent CF of 1.5 after the concentrator operating volume was increased from 2100 to 2500 gallons. Operation of the concentrator at a 2.0 CF would have been possible if the blend tank (TK-D4) pump had not been limiting.

2. Ruthenium Volatilization

Ruthenium levels in the Purex recovered acid at the conclusion of the thorium campaign were a factor of 20 to 200 higher than normally encountered during uranium-plutonium processing. The only operation to which this difference could be attributed is the addition of the feed concentrator condensate to the recovered acid. The recovered acid was still useable and the condensate was, therefore, routed to the Acid Recovery System throughout the campaign.

However, increased levels of beta-gamma ground contamination were found around the Purex stack and along the path in the direction of the standard stack plume during a quarterly area survey in January 1971. A study of gamma energy analyses indicated the activity was primarily ^{106}Ru released during the thorium campaign.

The lack of other fission products supported the conclusion that ruthenium was released as the volatile RuO_4 compound which is formed in boiling solutions containing greater than 8 $\underline{M}$ nitric acid. These conditions were attained during thoria dissolution, feed concentration-denitration, waste concentration, and recovered acid concentration. Evidence, which included finding ruthenium in samples of reflux water from the dissolver off-gas treatment facility and the high ruthenium content of the recovered acid, indicated both of the first two operations, thoria dissolution and feed concentration-denitration, contributed to the release. However, based on available information, the feed concentration-denitration operation was the major contributor.

CO-DECONTAMINATION AND PARTITION CYCLES

The flow sketch and the detailed chemical flowsheet for the Co-Decontamination and Partition Cycles are presented in Figures 1 and 4 in Appendix J.(8) The major change from the 1966 flowsheet was a 45 percent increase in the 1CX flow which was made in an attempt to reduce ^{233}U losses to the 1CW stream.

Operating and Process Control Description

The prepared aqueous feed (HAF) was pumped to the HA (T-H2) Column where the thorium and uranium were co-extracted into the organic phase (HAX), leaving most of the other radio-isotopes in the aqueous phase. The organic 1BSU recycle stream was also pumped from TK-J2 to the HA Column as the HAO. The organic was scrubbed in the top of the HA Column by the aqueous HAS stream. This dilute nitric acid solution (made up in TK-209 and TK-210 in the AMU area and fed to the column by gravity flow) contained small amounts of phosphoric acid and ferrous sulfamate to improve the plutonium, chromium, protactinium and zirconium-niobium decontamination. A 13 M HNO_3 stream (HAX-HNO_3) from the recovered nitric acid header was added near the bottom of the column to control the thorium waste loss. The combined aqueous stream exited the bottom of the column as the HAW. The combined organic stream, bearing the thorium and uranium, exited the top of the column as the HAP.

Process control of the HA Column consisted of overall plant rate control, maximization of fission product and ^{233}Pa decontamination and minimization of thorium waste losses. The HAF flowrate was adjusted to provide the specified thorium throughput rate. The thorium losses were then minimized by adjusting the organic to feed ratio with the HAX and the HAW acidity with the HAX-HNO_3. Fission product decontamination was maximized by adjusting the HAS rate and, to some extent, the organic to feed ratio.

The 1BXF feed to the Partition Cycle was composed of the organic overflow from the HA Column (HAP) and the organic waste streams from the Second and Third Uranium Cycles (2BW and 3BW). The separation of thorium and uranium was accomplished by stripping (back-extracting) the thorium from the organic phase with 0.2 M HNO_3 (1BX), which was line blended in the pipe and operating (P & O) gallery.

The uranium-bearing organic stream (1BU) flowed by gravity to the bottom of the 1C Column (T-H3), where the uranium was stripped out of the organic with 0.01 M HNO_3 (1CX). The 1CX was also line blended. The product-bearing aqueous stream (1CU) then exited the bottom of the 1C Column and was routed via a steam operated jet to the 1CU concentrator (E-H4), where it was concentrated to Second Uranium Cycle feed requirements.

The thorium-bearing aqueous stream (1BXT) was routed via an air-lift to the top of 1BS Column (T-J7). Here, it was scrubbed with an organic stream (1BS) from the No. 1 Solvent header to re-extract any uranium which may have stripped out of the 1BX Column. The organic scrub (1BSU) was routed to TK-J2 and recycled to the HA Column as the HAO stream. The aqueous stream (1BT) was routed to the 1BT (E-J8) concentrator and boiled down to Final Thorium Cycle feed requirements.

Process control of the Partition Cycle was primarily a matter of maintaining the operating variables at the specified values. Adjustments to the 1BX acid concentration and to the 1BX-to-1BXF ratio were made, particularly during startups and shutdowns, to reduce the amount of ^{233}U stripped from the 1BX Column. [See section on Partitioning Problems (Page 39).] Control of the E-H4 Concentrator posed a problem sue to the dilute overflow stream requirement.

Normal Operating Performance and Capacity

The HA Column operation presented no unusual problems during the campaign. Performance was satisfactory at the rates attempted. The thorium losses in the HAW stream were typically 0.01 to 0.1 percent of the HAF input. On several occasions (primarily during startups) the HAW thoria loss was as high as 0.4 to 0.6 percent of the input. The ^{233}U losses were typically 0.02 to 0.08 percent of the input. However, during the period from November 25 to November 29, the ^{233}U losses increased to 0.1 to 0.2 percent. Difficulty experienced in controlling the HAF flow during this period resulted in minor over-saturation of the organic (the thorium losses were also up slightly).

The steady-state performance of the Partition Cycle was satisfactory. Thorium loss to the uranium cycles was typically less than 0.01 percent of the input rate and the uranium loss to the thorium cycle was typically 0.01 to 0.05 percent. Uranium recycle via the 1BSU stream was normally 10 to 12 percent; however, it reached 60 to 70 percent during partitioning failures. Uranium losses in the 1CW organic waste stream ranged from 0.5 to 0.05 percent of the feed input. These losses were incurred even though the 1CX flow was increased by approximately 45 percent early in the campaign.

Fission product decontamination performance across the Co-Decontamination and Partition Cycles is summarized in the table below. The data are presented as decontamination factors, ratios of the fission product concentrations (microcuries per pound thorium or gram ^{233}U) in the HAF versus the 2DF for thorium and the HAF versus 2AF for uranium.

CO-DECONTAMINATION AND PARTITION CYCLE DECONTAMINATION FACTORS

	Zr-Nb	Ru-Rh	Pa
Thorium	180	105	85
Uranium	3.2×10^4	115	5.0×10^4

Some minor flowsheet adjustments were made during the campaign in an effort to shift the ^{106}Ru split toward the uranium cycles where the ruthenium decontamination was more effective. These changes included a reduction in the 1BX Column aqueous-to-organic flow ratio (L/V), and increases in the 1BX HNO_3 concentration and the 1BS organic rate. No significant differences were noted.

The 1BXT flow rate proved to be rather limited with a maximum sustainable rate corresponding to a 1.05 capacity factor. However, since the head end rate (and also the Second Uranium Cycle rate on several occasions) was even more restrictive, the 1BXT flow limitation was not a problem.

Operating Problem Areas

1. 1BU Route Hydraulics and Airlocking

The initial solvent extraction startup resulted in the transfer of approximately 3,500 gallons of solvent to TK-F18 by way of the 1BU sample pot vent line and the vessel vent header. (See Figure 19 in Appendix). Review of the hydraulic characteristics of the 1BU route revealed that if the six-inch header between nozzle JT-16 and HT-21 were full of air, a 22-foot, four-inch head could be developed at the H3 Column versus an 18-foot, eight-inch organic head between the 1BU sample pot vent and the six-inch header. When the 1BU flow was started, the solution backed up into the 1BU sample pot vent and began overflowing to the vessel vent header. The problem was further compounded by a syphon effect between the 1BU pot and the vessel vent header. The normal flow was established by loosening the jumper at nozzle HT-21 and allowing all the air in the header to bleed out while overflowing aqueous from the 1BX Column. Also, the jumper at nozzle "8" on the 1BU vent line was loosened to prevent the future formation of the syphon route. The 1BU stream became airlocked once more during the November 12 startup, and was restarted by loosening the jumper nozzle HT-21.

2. 1BU Sampler Hydraulics

The sampler outlet for the 1BU stream was located about two feet below the actual liquid level in the 1BU sample pot. (See Figure 19 in Appendix.) A special sampling procedure was issued to reduce the possibility of leaking 1BU solution into the sampler hood. Problems encountered with the valving sequence prior to removal of the sample bottle resulted in several minor contamination incidents. This problem was eliminated as the operators became more familiar with the system.

3. 1CW Sampler Operation

As was expected, the operation of the 1C (T-H3) Column was a major problem during the campaign. The problem was compounded when it was discovered, shortly after startup, that the H3-1CW sampler was not working. (Samples taken during the operability testing evidently came from the H3-HAP sampler which is in the same hood.) In order to provide ^{233}U loss and criticality prevention data, the sampling schedule for the No. 1 Solvent Treatment System (G Cell) vessels was increased until the H3-1CW sampler was returned to service on August 26. The sampler plugged several more times during the run but was repaired within two to three shifts each time.

4. 1C Column Flooding

Within 36 hours after the August 22 startup, the ^{233}U analysis on the No. 1 solvent wash waste (TK-G8) sample indicated that high 1CW losses were being sustained. In an attempt to reduce these losses, the 1CX flow rate was increased by approximately 45 percent and then on day shift, August 24, the 1C Column pulse frequency was increased from approximately 55 to 70 counts per minute (cpm). At 4:00 PM, the 1C Column (T-H3) flooded, resulting in the transfer of approximately 3,500 gallons of solvent through the 1CU concentrator to the 1UC Receiver (TK-J1) and the 2AF (TK-J21) tanks. A shutdown of the solvent extraction system was initiated at 11:30 PM in order to evaluate the situation and recover the organic.

The organic was processed through the Second Uranium Cycle to the 1BXF tank (TK-J3). The organic was then transferred to a spare tank (TK-F13) located in the Waste Concentration and Acid Recovery System for cleanup and return to the solvent system as described in the Section on Batch Solvent Recovery Operations, Page 63.

The limit on the ^{233}U grams per ton thorium ratio in the 1BXF established by the Criticality Prevention Specifications(15) was exceeded during this operation as discussed in the section on Nuclear and Chemical Safety, Page 73.

After the organic was removed from the Partition Cycle and Second Uranium Cycle, the columns were flushed, first with hot 57 percent HNO_3 and then with washed organic to remove thorium-dibutylphosphate (Th-DBP) solids (see section on Interim Flushing, Page 39). (Solids formation was the major cause of flooding in the 1966 thorium run.)

Detection of flooding in the 1C Column from the static pressure readout was not possible, so an interface float was installed in the bottom of the column. Initially it was used to activate an organic alarm on the central alarm panel. After the first flooding problems, the interface position was also recorded on a strip chart near the H3 position to provide a more tangible check point.

The 1C Column was restarted on September 1 and operated well until September 24, when it flooded again. The 1BXF-1BP streams (and the HA Column streams) were shut off and the organic was displaced from the 1C Column with 1CX solution. The 1BP flow was restarted, only to have the column flood again. The displacement and startup was repeated three times before the column would operate properly. The column flooded once more on September 27, shortly before the scheduled shutdown and turnaround to the "specification" run. No organic was lost during either of these floods as corrective action was taken before the organic detector float dropped off scale.

The 1C Column performed satisfactorily throughout the remainder of the thorium campaign. There were some indications of minor flooding during startup on October 29, but a small pulse frequency adjustment stabilized the column.

5. Reduction of Thorium-DBP Generation

One of the major reasons for the improvement in the 1C Column operation was the apparent reduction in the rate of thorium-DBP generation. The operating volume in the 1BXF Tank (TK-J3) was dropped by 50 percent, resulting in a reduction in organic residence/exposure time from 50 minutes to 25 minutes. The degradation of TBP from exposure to gamma radiation and nitric acid, and hence the amount of thorium-DBP formed, was reduced. The operation of the Second Uranium Cycle also showed significant improvement as a result of this change.

6. 1BXT Jumper Problems

The 1BXT air-lift jumper from the 1BX to the 1BS Column (see Figure 19) was redesigned to improve the plant capacity over that of the 1966 campaign. After some initial problems with the orientation of air lines in the jumper, flow tests were made to establish the maximum throughput rate. The highest sustainable flow was equivalent to a 1.0 CF rate. The loading pressure to the 1BXT DOV had to be increased to approximately 25 pounds per square inch (psi) versus the normal 10 to 12 psi in order to maintain the flow.

During the time when problems were experienced with the 1BX Column (T-J6) pulser gasket, the 1BXT DOV became plugged with gasket material. A new jumper was build with a screen on the inlet end to keep the material out of the valve. It was subsequently discovered that the trim of the valves in both jumpers was smaller than specified (1 1/4-inch and 3/4-inch versus 2-inch specifications). The valve trims in the screened jumper were corrected and the jumper was reinstalled. Only slight improvement in the flow capacity was noted.

7. J6 Pulser Gasket Problems

Prior to the initial thorium startup, the J6 pulser developed a leak and was changed out. On October 7, 1970, the motor on the new pulser burned out and another pulser was installed. Several unsuccessful attempts to seat the pulser to the column flange using varying thicknesses of teflon gaskets were made. On October 12, the leak at the flange was finally sealed using a Viton A gasket. The plant was started on October 13, and ran until October 23, when the 1BXT jumper plugged with gasket material. The pulser was removed and the Viton A gasket, which was falling apart, was replaced with a Viton B gasket sheathed in teflon.

8. Partitioning Problems

On numerous occasions, the partitioning in the 1BX Column degenerated and a large percentage of the ^{233}U followed the thorium. The ^{233}U was generally re-extracted in the 1BS Column and recycled to the HA Column via the 1BSU-HAO stream. However, in the more severe cases, the ^{233}U continued on into the Final Thorium Cycle. A total of 20 to 25 tons of thorium product had to be reworked to lower the ^{233}U content as a result of these problems.

The partitioning problems were caused by two conditions: periodic high L/V ratios in the column; and loss of salting strength due to low thorium concentration in the 1BXF. Improved control of the 1BX L/V was gained in mid-October by installing a visual flowmeter and a pre-amplifier on the magnetic flowmeter signal output of the 1BX-H_2O system (demineralized H_2O).

The ^{233}U tended to strip out of the organic whenever the thorium nitrate concentration in the 1BXF dropped. Startups and shutdowns unavoidably resulted in the thorium concentration being a factor of 10 to 100 below flowsheet. Recycled ^{233}U from the Second and Third Cycles and any ^{233}U being refluxed in the 1BX and 1BS Columns would immediately start stripping out into the thorium streams (1BXT and 1BT). This problem was reduced to a satisfactory level by doubling the 1BX-HNO_3 flow when low thorium concentrations were expected. The criticality prevention specifications for the 1BXF Tank (3,000 grams/ton) and the Final Thorium Cycle (500 grams ^{233}U maximum) were violated on several occasions as a result of this problem. The specifications were subsequently changed as discussed in section on Nuclear and Chemical Safety, Page 74.

9. Interim Flushing

The Partition Cycle equipment was flushed four times during the campaign to remove thorium-DBP solids. Hot 57 weight percent HNO_3 was used to dissolve the solids and 500-gallon purges of organic were pulsed through the columns (with the HNO_3 still in) to "soak up" the dissolved thorium-DBP. The organic was then treated in TK-F13 to remove the contaminants before recycle to the solvent treatment system. The aqueous phase was routed to the Waste Concentration-Acid Recovery System for nitric acid recovery, neutralization and disposal. The flushing cleaned out the columns quite effectively.

FINAL THORIUM CYCLE

The flow sketch and the detailed chemical flowsheet for the Final Thorium Cycle are presented in Figures 1 and 5 in the Appendix. The major variance from the flowsheet was the elimination of the phosphoric acid from the 2D (T-K2) extraction column intermediate scrub stream (2DIS). This change, made early in the run, largely eliminated 2D-2E Column instability problems encountered during the 1966 Thoria Campaign.

Operating and Process Control Description

The concentrated thorium stream from the Partition Cycle (1TC) overflowed the 1BT Concentrator (E-J8) to the 2DF feed tank (TK-K1). A stream containing ferrous sulfamate was also added to the feed to reduce any residual plutonium to the nearly inextractable plus three valence. The combined streams were pumped as the feed to the midpoint of the dual-purpose, extraction-scrub, 2D Column where the thorium was extracted into the 30 volume percent TBP-NPH solvent (2DX) while the plutonium and most of the remaining fission products exited via the aqueous 2DW waste stream. The 2DW stream was routed to TK-F10 in the Waste Concentration and Recovery System for further processing and disposal.

An intermediate scrub stream (2DIS) containing concentrated nitric acid (12.2 M) and, originally, 0.66 M phosphoric acid was added by gravity flow from aqueous makeup tanks to an entry point midway between the feed and the top of the column. The primary purpose of the nitric acid was to provide additional salting strength in the column to prevent excessive thorium losses to the 2DW stream. The phosphoric acid was intended to increase the protactinium decontamination attained in the column. However, this constituent was eliminated during most of the run due to the low protactinium content of the feed and to precipitation problems discussed in the section on 2D Column Startup Problems - Phosphoric Acid Removal (Page 42). A water scrub stream (2DS) was added to the top of the column by gravity flow to reduce the nitric acid concentration in the organic product stream (2DT) and, thus, minimize the volume of the final thorium nitrate product solution.

The 2DT overflowed the top disengaging section of the 2D Column to the bottom of the single-purpose 2E stripping column (T-K3). A dilute nitric (0.01 M) 2EX stream (line-blended in the P & O Gallery) was added by gravity flow to the top of the column to strip (back-extract) the thorium from the organic. The organic waste stream (2EW) was routed to the No. 2 Solvent Treatment System. The thorium-bearing aqueous stream (2ET) was routed to the 2ET (E-K4) Concentrator by jet transfer (flow controlled by 2E Column (T-K3) interface recorder-controller). There, the 2ET was steam stripped and concentrated as required. The resulting bottoms solution overflowed the concentrator to TK-K5 where it was accumulated in batches for subsequent jet transfer to the Thorium Nitrate Product Tank (TK-K6). Here, the product was sampled and subsequently pumped to a tank trailer for transfer to the storage tank(s). The overhead condensate

(2TD) from the 2ET (E-K4) Concentrator was routed to the 216-A10 process condensate crib for disposal.

Process control of the Final Thorium Cycle was maintained by close control of the aqueous-to-organic (L/V) ratios in the columns and stream acidities. For example, although the continued use of phosphoric acid in the 2DIS would probably have resulted in improved protactinium decontamination, most of the decontamination achieved in the 2D Column is due to close control of the above parameters. Control of these parameters was maintained in the 2D Column by avoiding over-concentration of the 1BT in E-J8 and adherence to the flowsheet 2DF, 2DX, 2DIS and 2DS rates. Likewise, control of the 2E strip column operation was primarily a matter of maintaining flowsheet L/V, 2EX acidity, and 2DT acidity. The effectiveness of these controls in reducing the fission product content of the 2ET thorium stream was continuously measured by passing a small portion of the stream through a recording gamma monitor. Routine sampling and analysis of the 2DW and 2EW once per shift provided adequate information to maintain control of thorium losses via these routes.

Normal Operating Performance and Capacity

Under normal conditions, the operation and performance of the Final Thorium Cycle was satisfactory throughout the campaign at rates up to 1.0 CF. The typical thorium losses in the 2DW and 2EW streams were less than 0.007 percent of the input during the early or "non-specification" portion of the run. However, the typical 2DW thorium loss increased to a range of 0.1 - 1.0 percent during the "specification" portion of the campaign. Even higher 2DW losses were experienced on several occasions. Typical thorium losses during the 1966 Campaign were 0.2 and less than 0.05 percent in the 2DW and 2EW streams, respectively.(3, 4)

The higher 2DW losses resulted from a deliberate increase in organic saturation which was made in an effort to improve the fission product decontamination attained in the 2D Column. This change was required due to a reduction in cooling time of the feed as the campaign progressed, resulting in a corresponding increase in fission product content.

The average (and range of) arithmetic decontamination factors attained in the Final Thorium Cycle during the specification portion of the campaign were 150 (18-470), 12 (2.5-21), and five (1-15) for ^{95}Zr, ^{106}Ru, and ^{233}Pa. The protactinium decontamination during the processing of shorter cooled material at the end of the campaign increased to the 500 to 2,700 range. This increased DF was due partly to a factor of 100 increase in the protactinium content of the 2DF and to the addition of phosphoric acid via the 2DIS stream. These DF's compare favorably with the typical DF's reported for the 1966 campaign of 7-8, 4-5 and 10 for zirconium, ruthenium, and protactinium, respectively.(34)

Operating Problem Areas

1. 2D Column Startup Problems - Phosphoric Acid Removal

Two attempts to start the 2D-2E Columns failed on September 7 and 8 because of precipitation of a gelatinous material in the 2D Column. The precipitate was qualitatively identified as a thorium phosphate gel which would dissolve in 10 M nitric acid. The precipitate formed as a result of following the original startup procedure which called for establishing the column interface with the concentrated 2DF thorium (0.8 M) solution. The subsequent addition of phosphoric acid via the 2DIS stream resulted in the rapid formation of the thorium phosphate gel.

The Final Thorium Cycle columns were flushed with concentrated nitric acid following each of these startup attempts. The resulting flush solutions were jetted to the Waste Treatment and Acid Recovery System (TK-F10). Prior to the third startup attempt, the procedure was changed to call for the use of pulser water as the aqueous phase in establishing the interface and the phosphoric acid was removed from the 2DIS stream until increased protactinium decontamination was required. This early removal of phosphoric acid largely eliminated column instability problems in the Final Thorium Cycle experienced during the 1966 Thoria Campaign.(32, 34) In addition, the lower solids content of the 2EW organic waste stream contributed to improved operation of the No. 2 Solvent Treatment System.

Phosphoric acid was not added again until several tons of short cooled material were processed at the end of the campaign. At that time, the phosphoric acid content of successive 2DIS batch makeups was gradually increased until a concentration equal to 75 percent (0.50 M) of the flowsheet 0.66 M was reached. The use of phosphoric acid was largely responsible for the increased protactinium DF attained during this portion of the campaign.

2. Rework of Thorium Contaminated With ^{233}U

On two occasions, the ^{233}U content of the thorium product exceeded acceptable levels and required rework of the solution through the Co-Decontamination and Partitioning Cycle. The uranium followed the thorium through the Final Thorium Cycle as indicated by 2EW ^{233}U analyses during these periods. These analyses show no increased loss of ^{233}U to the No. 2 Solvent Treatment Cycle.

Both occasions were due to partitioning failure brought about by a reduction in the thorium content of the 1BXF. The 1BXF thorium concentration was reduced because the Co-Decontamination Column (HA) was stripped and the Partitioning Column continued to operate after shutting off the HAF stream. The loss of salting strength caused the uranium to be stripped or back-extracted from the organic and exit with the normally thorium-bearing aqueous stream. This problem was subsequently eliminated by modifying the operating procedures to use a higher nitric acid concentration in the 1BX stream during periods with low 1BXF thorium concentrations at startup and to shut down the Partition Cycle at equilibrium whenever the HA Column was shut down.

Rework of approximately 4.5 tons of thorium was required on the first occasion, and approximately 12 tons of thorium were reworked on the second occasion. In both cases, the rework operation proceeded satisfactorily and did not cause any unusual problems.

3. Organic to Final Thorium Cycle Concentrator (E-K4)

On two occasions, a significant quantity of plant solvent was routed to the Final Thorium Cycle product concentrator and was found as an organic layer in the product storage tanks (TK-K5 and TK-K6). Both instances occurred during a startup situation due to instability problems encountered in the 2E (T-K3) stripping column. Approximately 500 gallons of organic were involved the first time, and about 1,000 gallons were involved the second time. In each case, the organic was jet transferred from the K Cell tanks to a spare utility tank located in the Waste Concentration and Acid Recovery cell. There, the organic was batch washed and returned to the No. 1 Solvent Treatment System for final cleanup and reuse.

4. High Thorium Loss to Waste

On November 13, a very high thorium loss to the 2DW stream (35 percent) was experienced for a period following startup. Significant leaks in the air lines to the 2D Column (T-K2) extraction section differential pressure taps and the 2DF Tank (TK-K1) specific gravity dip tubes resulted in indicated values for these important operating parameters that were much lower than the actual values. Based on these data, the 2DX solvent flow was biased much lower than required and the high loss occurred because the solvent was saturated. About 2,000 to 3,000 pounds of thorium were lost to waste before analytical data became available and appropriate corrective measures could be taken.

5. HNO_3 Balance and Adjustments from Flowsheet

As indicated in the table below, the nitric acid concentration in thorium streams was, in some cases, almost a factor of two above the flowsheet value throughout the non-specification portion of the campaign. The high nitric acid concentrations were primarily caused by maintaining the 1BX stream at a 0.85 capacity factor flow rate as compared to a thorium throughput rate equivalent to a 0.7 CF. Flowsheet adjustments made during the specification portion of the campaign significantly reduced the acid content of the thorium product stream below the flowsheet value.

THORIUM STREAM NITRIC ACID CONCENTRATIONS

Stream	Flowsheet, M	Typical, M Non-Specification	Typical, M Specification
1BT	0.325	0.40	0.38
2DF	1.10	1.71	1.47
2ET	0.25	0.48	0.091
TNT	1.46	2.87	0.63

6. K2 Saturation Control

As described in section on Normal Operating Performance and Capacity, Page 41, the 2D Column organic saturation was increased during the specification portion of the campaign in an effort to increase the fission product decontamination. As expected, the increased organic saturation resulted in higher 2DW thorium losses. However, the losses appeared to be unusually sensitive to slight flowsheet changes and a long period of operation was required to reach steady-state operation. The average 2DW thorium loss during this portion of the campaign was 0.95 percent as compared to less than 0.01 percent during the non-specification portion of the campaign. The high average loss included many instantaneous losses of greater than two percent. A more typical loss for the last three weeks of the campaign was 0.3 percent.

7. Interim Flushing

The Second Thorium Cycle equipment was flushed four times during the campaign to remove thorium-DBP solids. The first two flushes were associated with the use of phosphoric acid in the 2DIS during startup as described in the section on 2D Column Startup Problems - Phosphoric Acid Removal, and the others were made as part of the scheduled flushing conducted between prolonged processing periods. In each case, the columns were filled with hot (58 to 63 °C) 57 weight percent nitric acid to dissolve the solids. After a two-hour digestive period, a 500-gallon organic purge was pulsed through the filled columns to remove the dissolved thorium-DBP solids. The organic was then routed to the No. 2 Solvent Treatment System for cleanup prior to reuse. The aqueous nitric acid phase was routed to the Waste Concentration Acid Recovery System.

THORIUM NITRATE PRODUCT SHIPMENT AND STORAGE

Thorium product solution shipment proved to be less of a problem than was the case during the 1966 Campaign. Due to much lower fission product levels, the truck drivers experienced little exposure to radiation. There was no need to install the cab protection shield. During one period of time, as described below in number 2 under Problem Areas, a timekeeper was required to measure driver exposure.

Thorium Product Solution Shipment

The thorium nitrate product batch was sampled in TK-K6 and pumped to a 2,800-gallon tank-trailer standing by at the 203-A trailer loading facility. The trailer was hauled to either 221-U (WR Vault) or 204-S Tank Farm and unloaded.

With shipments only on day shift and swing shift, no difficulties were encountered in maintaining product shipments commensurate with an 0.85 CF. At a 1.0 CF, however, careful scheduling of shipments was necessary to prevent exceeding the small amount of lag storage volume available (TK-K5 and TK-K6). For the most part, direct radiation readings ranged between 10 and 20 millirads per hour on the bottom of the trailer, and background readings in the trailer cab were negligible.

Problem Areas

1. The system was barely adequate for a 1.0 CF thorium rate. At processing rates greater than 1.0 CF, additional lag storage volume and round-the-clock shipping would have been required to maintain plant rates.

2. During one period of operation, high ^{144}CePr levels in the thorium product resulted in significant exposure levels for the truck drivers. The peak reading approached 250 millirads per hour, with eight shipments reading in excess of 30 millirads per hour at the bottom of the trailer. During three of these shipments, measurable exposure levels were found in the cab. However, the cab exposure level was less than 10 millirads per hour. Hence, though a timekeeper was required, the protective concrete shield (required in 1966) was not installed between the cab and the trailer.

SECOND AND THIRD URANIUM CYCLES

The flow sketch and the chemical flowsheet for the Second and Third Uranium Cycles are presented in Figures 1 and 5 in the Appendix. The operation of these cycles was essentially identical to the final flowsheet used in 1966.(32)

Operating and Process Control Description

The concentrated uranium stream from the Partition Cycle overflowed the 1CU (E-H4) Concentrator to Tank J1 where it was combined with the overhead condensate (3UD) from the uranium product concentrator. The solution was then pumped through a flow control system to the 2AF Feed Tank (TK-J21). From there, it was pumped through a second flow control system to the 2AF entry point about two-thirds of the way up the dual-purpose (extraction-scrub) 2A Column (T-J22).

The middle column differential pressure tap was utilized to add a concentrated nitric stream (12.2 M) near the feed entry point. The additional acid was required to prevent excessive uranium losses to the aqueous waste stream (2AW). This acid could not be added directly to the feed tank due to criticality prevention specifications which assumed an organic layer of 100 percent TBP in the tank. Minimizing the acid concentration also minimized the possibility of extracting excessive amounts of ^{233}U into such a TBP layer.

The 2AS scrub stream added to the top of the column contained ferrous sulfamate to reduce any residual plutonium to the nearly inextractable plus three valence. The uranium was extracted into the thirty volume percent TBP-NPH solvent (2AX) which entered the bottom of the column while the plutonium and most of the fission products exited via the aqueous 2AW waste stream. The 2AW stream was routed to TK-F10 in the Waste Concentration and Acid Recovery System for further processing and disposal.

The uranium-bearing organic stream overflowed the top disengaging section of the 2A Column to the bottom of the single purpose, stripping or back-extraction, 2B Column (T-J23). Here, a dilute nitric acid (0.01 M) stream (2BX) was used to remove the uranium from the organic. The resulting uranium-bearing aqueous stream (2BU) was jet transferred through a flow control system and sample pot to the Third Uranium Cycle feed tank. The stripped organic 2BW stream was recycled to the Partition (1BX) Column feed (1BSF) tank, TK-J3.

The flowsheet and operation of the Third Uranium Cycle was very similar to that of the Second Uranium Cycle. However, to maximize product purity, no ferrous sulfamate was added and distilled water was used in the preparation of the 3BX stream. In addition to providing decontamination from fission products, thorium, plutonium, and other metallic impurities, the Second and Third Uranium Cycle flowsheets resulted in a factor of 15 increase in the uranium concentration.

Process control of the Second and Third Uranium Cycles was maintained primarily by monitoring the ^{233}U throughput and adhering closely to the flowsheet. Alpha monitors were placed on the 1BU, 2BU and 3AW streams as process control aids. Operational control of the 2A and 3A columns was somewhat complicated by the lack of extraction and scrub differential pressures (dp's) but was generally satisfactory. Instability of the 2B Column was encountered on various occasions, but was usually alleviated by lowering the pulse frequency.

Normal Operating Performance and Capacity

Generally, the operation and performance of the Second and Third Uranium Cycles were satisfactory throughout the campaign at rates up to 1.0 CF. The typical uranium losses in the 2AW and 3AW streams were 0.2 percent and less than 0.1 percent, respectively. Reported 2BW recycle rates averaged 1.0 percent during the early or "non-specification" portion of the campaign and decreased to 0.5 percent during the "specification" portion of the campaign. However, these numbers are believed to be biased high because of cross-contamination resulting from the use of the same sampling equipment for both the 1BXF and 2BW streams. Based on the flowsheet, the 2BW and 3BW uranium recycle rates should have been approximately equal.

The average (and range of) arithmetic decontamination factors attained in the Second and Third Uranium Cycles during the 1970 Campaign are shown and compared with the typical DF's reported for the 1966 Campaign(34) in the table below. The comparison is favorable in all cases.

SECOND AND THIRD URANIUM CYCLE DECONTAMINATION FACTORS

	Second Cycle 1970	1966	Third Cycle 1970	1966
^{95}Zr-Nb	400 (5-2800)	20	45 (0.3-186)	20
^{106}Ru	1700 (13-5300)	200	11 (1.3-24)	20
^{233}Pa	100 (<1-550)	13-75	13 (1-168)	2-3

As was the case in 1966, accurate DF's could not be determined for thorium since the thorium concentrations in the uranium streams were nearly always reported as less than values. However, using these values would give indicated thorium DF's of 30, 45, and 1,350 for the Second Uranium Cycle, Third Uranium Cycle, and both systems, respectively.

Operating Problem Areas

1. Organic in Feed

On August 24, a flood of the 1C Column (T-H3) resulted in the transfer of about 3,000 gallons of organic with the aqueous ^{233}U stream to the 1CU (E-H4) concentrator and the Second Uranium Cycle feed tanks. The process was shut down and the decanted aqueous phase was successfully processed through the Second and Third Uranium Cycles to recover the ^{233}U using a modified flowsheet.

An unsuccessful attempt was also made to process the organic portion through the Second Uranium Cycle. The high ^{233}U content of the organic required an abnormally high aqueous to organic flow ratio in the 2B Column. An unacceptably high ^{233}U loss to the 2BW resulted when the flow ratio was not increased to the desired level and the operation was discontinued. The organic was then transferred to a spare utility tank in the Waste Concentration and Acid Recovery System. There, the organic was batch washed and returned to the No. 1 Solvent Treatment System for final cleanup and reuse.

2. Column Stability

The stability of both the 2A and 2B Columns decreased gradually, but steadily, during a prolonged operating period. Major flooding of the 2A and 2B Columns occurred on September 27, October 30, and November 13. The columns were unstable at other times during operation and gradual reductions in the pulse frequency were required.

For example, the stability of the 2B Column was reduced markedly by the end of the first three week operating period. The column contents frequently became emulsified, resulting in two-phase 2BU and 2BW streams. Toward the end of the run, the 2BW sample contained small amounts of white thorium-dibutylphosphate (Th-DBP) solids. This confirmed the belief that the buildup of these solids on the column cartridges was responsible for column instability. Reducing the pulse frequency was the only effective means of stabilizing the column. By the end of the run period, the pulse frequency had been reduced from 60 to 45 cycles per minute.

The 2B Column was very stable during the last four-week operating period, and a significant reduction in the amount and frequency of appearance of the Th-DBP solids in the samples was noted. The only change made in the system to reduce solids formation was decreasing the organic inventory in the HAO and 1BXF tanks. This reduced the organic exposure to nitric acid degradation by about 30 to 50 percent.

3. 2BU Jet Plugging

During startup preparations on November 11, the 2BU jet suddenly plugged and would not permit flow from the 2B Column to the 3AF Tank. This occurred even though the entire solvent extraction system had been flushed with concentrated nitric acid during the preceding down period and flow through the jet had been initially established. Extensive backflushing of the jet suction, discharge, and steam supply lines with a hydraulic ram followed by several concentrated nitric acid flushes was required to restore normal flow. These flushing operations delayed the plant startup by four shifts. Less serious plugging of this jet was also experienced during the early startups, but was generally prevented by establishing 2BX-2BU flow prior to turning on the jet. During the first startup on August 20, the jet would not turn off in the normal manner and had to be manually valved out.

4. 3A Column Interface Control

Control of the 3A Column (T-L1) interface became erratic on September 20. The erratic overflow also upset the 3B Column for a short period. This problem was corrected by placing the 3AX flow control on manual and maintaining a constant static pressure in the 3A Column. Operation continued in this manner for approximately ten days until the end of the operating period. At that time, the interface float was removed from the column and was found to be filled with liquid which had caused it to sink. The float was replaced with a new one prior to the next startup. Minor problems with the signal converter subsequently experienced on November 15 and December 4 were corrected by instrument adjustments.

5. 2A Column Pulser Control Problems

The regular 2A Column (T-J22) pulse generator failed on three occasions during the two-week period of attempting to start up in October which coincided with the 1BX Column pulser gasket difficulties. These failures resulted in minor upsets of both the 2A and 2B Columns which may have been more serious had they occurred during normal operation.

On each occasion, the motor could be restarted but the pulse frequency could not be maintained at the desired level. It was, therefore, necessary to switch to the spare unit. The cause of the problem could not be readily determined. However, following the second failure, the control transformer was replaced and several loose connections in the control circuit were tightened. Additional adjustments were made following the third failure and the unit operated satisfactorily for the remainder of the campaign.

6. Reduction of 3BU Organic Entrainment

Local analyses of the early ^{233}U product indicated the phosphorus content exceeded the specification limit of 150 parts phosphorus per million parts uranium dioxide (ppmp UO_2) by a factor of two to three. In addition, observations of the product loadout tank indicated the presence of a thin floating organic layer. Although these high values were not confirmed by the customer, whose analyses generally indicated a phosphorus content of less than 100 ppmp UO_2, corrective measures were taken in an effort to reduce the phosphorus contamination.

Since the soluble TBP content of the 3BU would have been reduced to only a few ppm by steam stripping during concentration, the source of organic was assigned to entrainment of excessive quantities of solvent in the aqueous 3BU stream. Corrective measures taken to reduce this problem consisted of decreasing the 3B Column pulse frequency and raising the column interface. In addition, the 3BU receiver tank (TK-N1) liquid level was maintained at a higher value to provide the maximum practical time for phase separation prior to concentration.

Although it could not be determined if these measures were solely responsible, the locally reported phosphorus content of subsequent product batches did decrease to below the specification limit in most cases. Only eight of the remaining 36 batches were reported to contain phosphorus at greater than 150 ppmp UO_2. Of these, two batches were produced immediately following a startup, four were produced immediately following shutdowns, and the other two were reported at only 153 ppmp UO_2.

7. Interim Flushing

The Second and Third Uranium Cycle equipment was flushed with hot (58 to 63 °C) 57 weight percent nitric acid on four occasions during the campaign. Two of the flushes were necessitated by process upsets and the other two were made as scheduled between prolonged processing periods. An organic purge was pulsed through the filled columns to remove dissolved thorium-DBP and was collected with the organic used in the Partition Cycle flushing operations. The nitric acid flush solution was processed through the Waste Concentration-Acid Recovery System.

URANIUM-233 PRODUCT CONCENTRATION, LOADOUT AND STORAGE

The ^{233}U product thorium decontamination and concentration flow sketch and the chemical flowsheet are shown in Figures 1 and 5 in the Appendix. An N Cell-PR Room flow schematic showing both product concentration and product loadout is also provided (Figure 20). The major equipment items are described in Appendix B.

Operation and Process Control Description

The 3BU stream from the Third Uranium Cycle flowed by gravity to the N Cell feed tank, TK-N1. From TK-N1, the 3BU was pumped up through a cation exchange column, (T-N50, Figure 21), where any thorium present in the feed solution was preferentially absorbed onto the cation resin. The thorium-decontaminated column effluent (3CU) overflowed to the product concentrator, E-N6, for final concentration. After reaching the proper concentration (a volume reduction of a factor of 50), the concentrated product overflowed to the product receiver tank, TK-N7. Thirty liter product batches were accumulated in TK-N7, then vacuum transferred to the product sampling tank (TK-L9, Figure 29) in the PR Room. In TK-L9, the solution was well mixed and then sampled. Samples were taken using an air jet to circulate solution past a four-way sample valve inside the TK-L9 hood. Upon receipt of sample results, the product solution was vacuum transferred in 2.65 liter volumes to the loadout head tank, TK-L13. From L13, the uranium nitrate product solution was gravity loaded into tared three liter polyethylene bottles. Loaded bottles were capped, weighed, and then placed in either storage or shipping containers. The containers were closed and removed from the PR Room to temporary storage areas in the Purex Plant. The loaded containers were moved, via the PR truck, to the pipe gallery of the 221-U Building to be stored and to be prepared for shipment offsite.

Primary differences between this process sequence and that used in 1966 were the elimination of the Vycor glass column used in 1966 for ^{233}Pa decontamination, installation of the L9 product sampling tank for the 1970 Campaign, and the use of a new product loadout system.

The final thorium removal step by ion exchange contacting required no activity on the part of operating personnel. However, final thorium levels in the ^{233}U product were monitored for bed breakthrough as well as for indications of overall thorium-uranium partitioning efficiency. The radiation level of the bed was monitored to determine fission product holdup.

The two primary operations, 3BU concentration and product batch loadout, were relatively simple to control. Based on product sample results, the ^{233}U product concentration was correlated with the E-N6 bottoms specific gravity. Adjustments in the degree of concentration were then made as required to meet the targeted ^{233}U product concentration of 350 ± 25 grams per liter.

Product solution transfer and loadout required close attention to proper batch volumes. Approximately 30 liters of solution were required to constitute a discrete L9 batch for sampling purposes. (To meet customer requirements, a sample of at least every 12 kilograms of product was necessary.) The 30-liter batch was transferred in 2.65 liter batches for loadout. The loadout volume was visually checked in the glass loadout tank (TK-L13) prior to gravity transfer to the bottle.

A log book was kept for maintaining the identity and history of each loaded bottle. Titled, U-233 Record Loadout Book, it was kept in the shift office at Purex and contained the bottle container number; loadout weights, dates and times; TK-L9 weight factor and specific gravity (SpG) data; and the operators' names.

Operating Performance and Problem Areas

1. T-N50 Cation Resin Column

The ion exchange column was bypassed during the high ^{238}U portion of the campaign. The high uranium holdup on the bed would have acted as a source of ^{238}U during subsequent specification grade ^{233}U production. During specification ^{233}U production the column provided backup thorium separation. There was no difference in the thorium levels in the high ^{238}U product and the specification grade product. At the conclusion of processing, approximately 2.8 kilograms of uranium were eluted from the bed, concentrated, and loaded out as product. This material met all product specifications except those for ^{232}U and phosphorous.

The only problem associated with the cation column arose due to incomplete sealing around the top and bottom resin screens. Resin was found in the first batch of product. After changing to resin with a larger bead size failed to eliminate the problem, a replacement column was fabricated and installed. During the course of the campaign, resin beads occasionally were observed in product samples. These were found to be residual beads left in the system from the original breakthrough.

2. E-N6 - Product Concentration

The concentrator capacity was not overtaxed at ^{233}U production rates. During transitions to rates near 1.0 CF, steam adjustments often required approaching maximum steam flow. Control of the N6 solution specific gravity was excellent. After an initial period of empirically determining the proper control point, product concentration was maintained at flowsheet with only minor difficulties.

3. Product Sampling and Loadout

The new L9 tank proved to provide excellent mixing and the L9 sampler was reliable, trouble-free, and no contamination problems were experienced with it. One problem with the sampler was that only two milliliters of sample could be obtained with each turn of the sampler valve. As the required sample size gradually grew to 150 milliliters, it required many turns of the sampler valve and about five minutes to take this sample.

The volume of product solution charged to a bottle had to be decreased to 2.65 liters, from the 2.9 liters used in 1966, due to smaller size bottles. The 2.9 liters filled the new bottles almost to the top of the neck. As described in the section on Shipment under Uranium-233 Storage, Transloading and Shipment, some spillage occurred which was attributed to jostled bottles. Decreasing the volume of solution per bottle was one remedy used.

In general, product sampling and loadout was trouble-free. On one occasion, about 200 milliliters of solution overflowed a bottle to the floor of the loadout hood. The operating error came about through faulty communications between the individual at the L13 Tank and the individual at the loadout hood. The spilled solution and excess solution in the loadout bottle were transferred to a rework tank by means of the loadout hood vacuum transfer line. The loadout hood required extensive decontamination work.

URANIUM-233 STORAGE, TRANSLOADING AND SHIPMENT

Facility Description

The northeast end of the Pipe Gallery in the 221-U Building (200 West Area) was set aside under a special Radiation Work Procedure (RWP) as a storage, transloading, and offsite shipping area for ^{233}U product.(5, 40) A small metal enclosure, approximately 8 feet 9 inches by 16 feet, was erected near the center of the storage and shipping area for use as a containment facility within which loaded uranium product bottles could be handled outside their containers. The structure was ventilated by a standard exhaust blower - filter arrangement.

The area northeast of the enclosure was used for storing loaded product containers received from Purex. The area southwest of the enclosure was used for preparing product containers for shipment.

The transloading facility consisted of two sections; one section for the actual bottle-to-bottle transfer, the other section for decontamination work, bottle weighing, and container testing.

Operations Conducted

1. Transloading

The transloading equipment consisted of a transfer system with positions for three bottles and an air jet for establisning a vacuum on the three bottles. The third bottle was included for deentrainment. The vacuum jet exhausted to the canyon of the 221-U Building via a filter and wall nozzle.

Transloading operations were conducted on 18 occasions. Ten bottles were transloaded to separate out gross quantities of cation resin associated with the first product loadouts (as discussed in the section on T-N50 Cation Resin Column). Five bottles had to be transloaded because of spillage resulting from misseated O-Rings. One bottle was transloaded to decant a layer of organic. Two bottles had to be transloaded after exceeding the thirty-day limit on the storage period allowed by the Division of Transportation (DOT) shipping permit. All transloading operations were conducted without significant trouble.

2. Preparations for Offsite Shipment

Preparations for shipment to the customer involved transferring bottles in storage containers to shipping containers, transloading all solution stored over 30 days from old bottles to new ones, and checking each shipping container vent valve and pressure chamber for leaks. Bottle transfer from storage containers to shipping containers also had to be conducted within the confines of the transloading facility.

Each shipping container was tested prior to shipment to assure the absence of gas leakage from the chamber. Two potential leak locations were the vent valve and the cover flange and gasket. The vent valve was checked on a test stand which was pressurized to five psi for five minutes while a manometer was monitored for indications of pressure drop. If there were no indications of leakage, the cover flange, with valve, was replaced on the shipping container pressure chamber and tightened to 40-foot pounds torque on each of the eight stud bolts. The shipping container was then moved to the pressure chamber test stand, located just outside the transloading facility. The cover flange and gasket system was tested by pressurizing the pressure chamber to two psi for five minutes while monitoring a manometer for pressure drop.

After passing these tests, the shipping carrier was deemed ready for shipment. The carriers were loaded onto the trailer, protective shoring was installed to hold the containers in place, and the trailer was closed. The trailer doors were sealed with tamper-proof seals.

3. Shipment

A total of 639 loaded bottles, plus three bottles containing samples were shipped during the 1970 Thorium-^{233}U Campaign. The shipments were made to Oak Ridge, Tennessee in eleven separate trailer loads; five by commercial truck and trailer, five by AEC-owned truck and trailer with couriers, and one by AEC-owned trailer and Tri-State truck tractor. The commercial truck and trailer loads were limited to 25-kilogram loads (safeguards limit for drivers without a security clearance), whereas the AEC-owned trailers with couriers and security cleared drivers were limited only by criticality prevention specifications. The largest load shipped during the campaign was 121 containers.

The only problem with uranium product transfer occurred while the first three shipments were in transit. The customer reported that several bottles had leaked. For the most part, the uranium product solution was contained within the plastic bag around the bottles; however, eight shipping carriers required internal decontamination. More importantly, there was no contamination release outside the shipping carriers.

An investigation found that two types of leaks had occurred. Some of the bottles had leaked from under the cap. The remainder had leaked through the cap vents. In the former case, the leaks were attributed to improper seating of the O-Ring gasket in the cap. Subsequently, inspections of all bottles prior to shipment were made to ensure that the gaskets were properly placed.

To prevent leaking through the cap vent, a number of changes were made. The amount of product solution loaded into a bottle was decreased from 2.9 liters to 2.65 liters. Rubber spacers were installed between the bottle tops and the shipping container closing flange to ensure bottle immobility within the carrier. Further, all subsequent shipments were made via exclusive-use vehicles. This eliminated in-transit handling of the shipping carrier.

Records

1. A log book was kept in the storage area for maintaining the identity and history of each loaded bottle from the time it was received into the storage area until it was shipped offsite. The log, <u>U^{233} Record of Storage and Shipping</u>, contained run and bottle number, container

numbers, venting data, transfer and transloading data, radiation levels on bottles and shipping dates.

2. An identification tag, showing run number, bottle number and date was attached to the container lid for each bottle at the time the bottle was loaded into the container at the Purex loadout station. This tag remained with the bottle, moving to other container lids, as the bottle was transferred between containers.

3. Shipper-Receiver reports were made out for each load of uranium product shipped. The report showed run and bottle numbers, container numbers, weights of bottles and materials shipped.

4. Certified analyses sheets were obtained from the analytical laboratories for each product batch shipped. Analytical data reports were included with the shipping data sheets and cover letter sent to Oak Ridge following each shipment.

5. A ^{233}U product loadout record was started for each bottle of ^{233}U at the time of loadout in Purex Plant. Upon receipt of certified analyses sheets, these records were completed and checked against Shipper-Receiver reports and then delivered to Nuclear Materials Accounting. The ^{233}U product loadout record showed loadout data, run and bottle number, container number, sample numbers, weights, uranium values, and names of loadout operators.

WASTE TREATMENT AND ACID RECOVERY

The waste treatment and acid recovery flow sketch and the chemical flowsheet are shown in Figures 1 and 7 in the Appendix. The flowsheet used was similar to that used in the 1966 Campaign.(32, 48) The main differences were:

1. The rerouting of the overheads during the feed concentration step from the waste concentrator feed tank (TK-F7) to the acid absorber bottoms (AAA) receiver tank (TK-F3); and

2. The utilization of TK-F18 for reverse strike waste neutralization and disposal to underground storage, rather than direct strike waste neutralization and disposal from TK-F16.

Rerouting of the feed concentrator overheads to the AAA receiver tank (TK-F3) permitted operation of the concentrator at optimum conditions for steam stripping of nitric acid without exceeding boiloff capacity. Despite the direct addition of the condensate into the 25 percent recovered acid, only a moderate rise in the recovered acid fission product content was noted. As previously stated, this was due to the long target cooling time prior to processing.

Operating and Process Control Description

The aqueous waste streams from the extraction columns were collected and fed to the high level waste (1WW) concentrator (E-F6). The HAW stream gravity flowed directly to the feed tank (TK-F7). The 2DW, 2AW and 3AW streams flowed by gravity to TK-F10, combined with the vent system condensate and were pumped to TK-F7. A small stream of sugar solution was added to TK-F7 to suppress ruthenium volatilization from the concentrator. The combined waste was pumped from TK-F7 to the waste concentrator (E-F6). The overheads from the concentrator passed through an auxiliary demister and an acid absorber (T-F5). The bottoms overflowed into the 1WW receiver tank (TK-F26) and was jetted in batches to the sugar denitration vessel (TK-F15). After sugar denitration, the waste batch was jetted to TK-F16 and digested at elevated temperatures to destroy the remaining sugar and denitration byproducts. From TK-F16 the waste was jetted to TK-F18 for reverse strike neutralization* with 19 M sodium hydroxide (NaOH). The neutralized waste was then disposed of to underground storage.

The 25 weight percent nitric acid recovered in the F5 absorber tower (AAA) overflowed to TK-F3, was combined with the feed concentrator condensate, and was pumped to the vacuum fractionator feed tank (TK-U5). The vacuum fractionator concentrated the dilute nitric to 60 weight percent (13 M) for reuse, primarily in the dissolvers, as Purex recovered nitric acid.

Process control focussed on four key parameters; the concentrated waste generation rate, the sugar denitration and waste neutralization cycle time, the nitric acid concentration of the acid absorber bottoms, and the recovered acid quality and percent recovery.

The waste generation rate was maintained by controlling the E-F6 bottoms specific gravity and monitoring the actual gallons of waste generated per ton of thorium fed to the HA Column. The cycle time for waste denitration was controlled by the sugar addition rate, the initial acidity, and the targeted terminal acid concentration. The neutralization portion of the cycle was primarily a function of lag storage space and availability of TK-F18. The caustic added for each waste batch was calculated from an empirically derived formula.

The absorber bottoms acidity was monitored with the solution specific gravity. The specific gravity was in turn controlled by adjusting the reflux water flow rate to the acid absorber.

The acid concentration operation was evaluated in terms of condensate acidity (losses) and bottoms acid concentration. Condensate acidity and and recovered acid strength were controlled by adjusting the bottoms

---

*Reverse strike caustic neutralization - addition of the acidic solution to the 19 M caustic. This method decreases the quantity of solids formed.

specific gravity and reflux water flow rate in accordance with a nomograph which provided optimum values of these two parameters as functions of feed quality and flowrate.

Normal Operating Performance and Capacity

The performance of the waste concentrator and associated off-gas system was satisfactory. The F6 concentrator contributed little activity to the Purex recovered acid. Typical ratios of the ^{95}ZrNb and ^{106}Ru content of the bottoms to that of the condensate ranged between 10^4 and 10^5.

The nitric acid boiloff in E-F6 was close to that predicted in the flowsheet.(8) The bottoms acid concentration ranged between 3.7 M and 4.1 M versus a flowsheet value of 4.0 M. The control of the F6 concentrator bottoms overflow rate was poor, initially. Approximately 350 gallons of waste were generated for every ton of thorium processed during the non-specification portion of the campaign, versus a flowsheet value of 300 gallons per ton. Most of the excess volume was generated during the first two weeks of operation. During subsequent processing, the overflow averaged about 10 percent above flowsheet. The concentrator boiloff capacity was more than adequate for a 1.0 CF rate. This was the primary benefit derived from routing the condensate from the feed concentrator to the recovered acid system instead of to the waste concentrator feed tank (TK-F7).

The waste denitration and neutralization processes proved adequate. Sugar efficiency ranged between 14 and 15 moles of nitric acid destroyed per mole of sugar consumed. These values were typical for 1WW with a high aluminum nitrate content. Batch turnaround times in TK-F15 were satisfactory at a 0.75 CF rate, but marginal at a 1.0 CF rate. Some improvement in batch turnover was achieved by lowering the reaction temperature to accommodate higher sugar addition rates. However, at too low a temperature, this technique resulted in increased waste volume because the byproduct water was not boiled off. During the digestion period, some volume reduction was obtained which compensated for part of the increase during sugar denitration.

Caustic consumption during waste neutralization by reverse strike in TK-F18 was high during the non-specification portion of the campaign. The reported caustic ratios (pounds of 50 percent caustic required to neutralize one gallon of the sampled solution) were inaccurate. Further, the reported pH values of neutralized waste were difficult to correlate due to the complexity of the solution. An empirical curve was established which provided caustic ratios based on known aluminum nitrate concentration in the waste and the acid concentration measured in the laboratory. Improved control of caustic additions resulted, although caustic consumption continued to be excessive.

Performance of the T-F5 acid absorber was difficult to assess. The recovery efficiency was indirectly determined to be about 80 ± 5 percent.

This recovery had been normal for the level of acid in the condensate samples during thorium operation. The efficiency and bottoms concentration could not be directly evaluated due to the influx of the feed concentrator (E-F11) condensate into TK-F3.

The acid vacuum fractionator operation was difficult to control at the required constant bottoms acid concentration and production rate with minimal overhead loss. The difficulty was due primarily to the nonuniformity of the feed acid concentration and the wide variation in feed rate. The feed fluctuations were due to the batchwise denitration operations conducted in the feed concentrator.(17, 55, 56) A nomograph was constructed which provided material balances around the fractionator for given operating conditions. Upon incorporation of the nomograph in the operating procedure and fractionator process control, the overall acid recovery rose from between 90 and 95 percent to 99.95 percent.(1)

Operating Problem Areas

1. Concentrator Overflow

The high waste generation rate during the first two weeks of solvent extraction processing resulted from a number of unsuccessful attempts to achieve continuous overflow out of the concentrator. Similar problems with high aluminum nitrate content 1WW had been encountered in the past, both during processing of Zirflex-clad fuels and during the 1966 Thorium Campaign. The overflow appeared to be restricted by solids. An increase in operating level improved the continuity of flow; but also interfered with liquid level and specific gravity bubbler dip tubes, hence affecting control of boiloff rate and bottoms concentration. A number of operating techniques were tried but proved of limited value. During the initial three weeks, the overflow rate gradually improved. However, the operation continued to be troublesome throughout the campaign, particularly after starting up the concentrator without a 1WW inventory.

2. Waste Batches High in Thorium

As discussed in the section on Rework of Thorium Contaminated With U-233, Page 42, high 2D Column losses resulted in a series of waste batches with relatively high thorium concentrations. During the latter stages of sugar denitration, a precipitate consisting of $Th(C_2O_4)_2$ and $Th(SO_4)_2 \cdot 4H_2O$ was formed. During the reverse strike, caustic neutralization precipitated most of the remaining thorium. The solids impeded jet transfer of the waste solution and also increased the possibility of plugging the waste transfer line out of the facility to underground storage tanks.

3. Waste Disposal Scheduling

The F18 tank served two waste disposal functions. The first was as a sump collection and sampling point prior to disposal to underground storage. The second function was as a caustic reverse strike neutralization tank for 1WW. The transfer route to underground storage had a low capacity steam jet to prevent overheating of the buried line. Consequently, batch turnover times in TK-F18 proved limiting at rates approaching 1.0 CF. During such periods a temporary route had to be installed from TK-F16 directly to underground storage. Even at reduced rates, however, the waste disposal system out of TK-F18 was marginal. Excessive sump volumes or poor scheduling frequently resulted in filling lag storage space in TK-F16 which then required double batching in TK-F18.

NO. 1 AND NO. 2 SOLVENT TREATMENT SYSTEMS

The flow sketch and the chemical flowsheet for the No. 1 and No. 2 Solvent Treatment are presented in Figures 1 and 6 in Appendix B.(8) The operation of these systems was essentially identical to the final flowsheet used in 1966(32) with the following exceptions: (i) the potassium permanganate concentration in the Na_2CO_3-$KMnO_4$ wash was reduced by one-third; (ii) the No. 2 Solvent (200) was not centrifuged; (iii) the acid content of the 200 was reduced during the 1970 Campaign; and (iv) the operating temperatures were reduced.

Operating and Process Control Description

The organic waste stream (1CW) from the uranium stripping (1C) Column of the Co-Decontamination and Partition Cycle was washed with a Na_2CO_3-$KMnO_4$ solution in TK-G1 as described in Appendix A, Carbonate-Permanganate Washers (TK-G1 and TK-R1). The aqueous stream was recycled by a pump at a high flow rate [200 gallons per minute (gpm)] for a period of eight to 12 hours and then changed out on a batch basis. The continuous organic stream was transferred to the bottom of the pulsed acid-wash (IO) Column via a pump with a short dipleg.

In the IO Column, the organic was contacted with a recirculating dilute nitric acid stream which was also changed every eight to 12 hours. A small amount of concentrated (12.2 M) nitric acid was added continuously at the top of the column to maintain stability. The solvent overflowed the column to the TK-G7 turbomixer [described in Appendix A, Turbomixer (TK-G7)] where it was contacted with a continuous sodium carbonate stream. The aqueous waste was pumped to the first cycle combined solvent treatment (1OW) storage tank (TK-G8) via the decanter (TK-G6) for subsequent batch transfer to underground storage.

The treated solvent was pumped to the 100 pump tank (TK-G5) where nitric acid was added during agitation of the vessel contents to assure that entrained sodium carbonate was not pumped to the solvent extraction columns. The acidified solvent was then pumped to the 100 header which supplied the Co-Decontamination and Partition, Second Uranium, and Third Uranium Cycles.

In the No. 2 Solvent Treatment System, the organic waste stream (2EW) from the Second Thorium Cycle received a carbonate-permanganate wash followed by a nitric acid wash prior to returning to the process as the 2DX stream. The equipment, flowsheet, and waste handling procedures for this system were as described for these operations in the No. 1 System, except a different type cartridge was used in the acid wash (20) Column and more acid was added to the top of the column to provide the desired 200 acid concentration.

Process control of the Solvent Treatment Systems depended upon minimizing the aqueous carryover from the carbonate-permanganate wash tanks to the acid wash columns which would result in column instability. Another potential problem was the accumulation of excessive amounts of thorium dibutylphosphate solids with a resulting decline in solvent quality.

Normal Operating Performance and Capacity

Operation of the Solvent Treatment Systems was generally satisfactory at rates up to 1.0 CF. Solvent quality was high throughout the campaign except during recycle or "spinning" of the solvent within the system. A comparison of the typical 1970 values for solvent plutonium retention and radionuclide content with those of 1966(32) is shown in the table below.

NO. 1 AND NO. 2 SOLVENT QUALITY

	No. 1 Solvent		No. 2 Solvent	
	1966	1970	1966	1970
Pu Retention counts/min.-gal.	1.5×10^6	2×10^6	2.5×10^6	6×10^6
Ru, μc/gal.	50	200	40	13*
Zr, μc/gal.	3	0.5	5	0.3*
Pa, μc/gal	15	0.2	50	0.3*

---

* Only three analyses reported.

The reduction in the zirconium and protactinium content of the 1970 solvent was probably due to the longer cooling time of the target elements prior to processing. The increased ruthenium content of the 1970 No. 1 Solvent did not significantly affect solvent extraction performance. Fission product decontamination factors across the Solvent Treatment Systems could not be calculated as gamma scan analyses of the 1CW and 2EW streams were not reported.

The average overall solvent loss during the 1970 thorium campaign was 585 gallons per operating day (gpd). This value was about a factor of three above the normal 150 to 200 gpd loss during uranium-plutonium processing and ten percent above the 530 gpd loss experienced during the 1966 Thorium Campaign.(32) The solvent loss would have been even higher without the recovery of 20,000 gallons of solvent during the campaign (described in the section on Batch Solvent Recovery Operations, Page 63) which would have normally been discarded to waste.

Operating Problem Areas

1. TK-G1 Recirculation Pump

During the spinning of the No. 1 Solvent Treatment System prior to startup, the organic feed to the 10 Column (T-G2) contained an excessive amount of aqueous. Inspection from the crane revealed the TK-G1 aqueous recirculation pump was spraying liquid from the graphite bearing near the top of the pump shaft. Thus, the aqueous was falling on the organic layer and was pumped to the column with organic. The problem was corrected by placing the pump in a metal tube which was raised off the tank bottom and extended the full length of the pump shaft. The leaking solution then hit the side of the tube and returned to the aqueous phase at the bottom of the tank.

2. Solvent "Spinning"

Following a solvent extraction system shutdown for an extended period the organic was recycled within each solvent treatment system to continue removing radionuclides and solvent degradation products (dibutylphosphate, monobutylphosphate). This "spinning" operation was also conducted prior to starting solvent extraction operations. The performance of each system was monitored by obtaining plutonium retention analyses of the treated solvent. During these periods the plutonium retention values increased by factors up to 1,000 which indicated very unsatisfactory solvent quality. The cause of the problem was determined to be the reaction of nitric acid returned in the solvent from the acid wash column with the sodium carbonate in TK-G1 (TK-R1). This reaction resulted in a low pH and ineffective washing. The problem was corrected during the latter part of the campaign by eliminating the concentrated acid streams to the acid-wash columns during the "spinning" operation.

3. TK-R1 Circulation Pump

The reported plutonium retention values for the treated No. 2 Solvent exceeded normal values by a factor of 1,000 for several days while spinning the system prior to the scheduled solvent extraction system startup in late August (did not occur until September 7). Early analyses indicated the TK-R1 pH was low due to nitric acid recycle as described above. However, the problem persisted even after the concentrated nitric acid flow to the acid-wash column was stopped and the TK-R1 pH returned to greater than ten. Further investigation revealed the aqueous recirculation rate in TK-R1 was only 15 gallons per minute as compared to a normal 200 gpm. Installation of a new pump corrected the problem prior to the delayed startup.

4. Organic Transfer from TK-R1 to TK-R8

During the 1966 campaign, about 70 percent of the 530 gpd solvent loss (370 gpd) exited in the combined No. 2 Solvent Treatment System waste stream (20W) from TK-R8.(32) In an effort to prevent the recurrence of this problem, a new aqueous selective jet was installed to transfer the carbonate-permanganate solution from TK-R1 to TK-R8 during change-outs. However, excessive amounts of organic continued to be transferred with the aqueous as TK-R8 instruments indicated the presence of an organic layer. This layer was periodically returned to the No. 2 Solvent inventory for retreatment after the aqueous was transferred from TK-R8 to underground storage. The problem was reduced by using the TK-R1 specific gravity recorder as a guide for controlling the transfer to TK-R8. The steam supply to the jet was turned off as soon as the instrument indicated the presence of organic.

5. Acid-Wash Column Operation

The interface float in both acid-wash columns failed to respond during the latter portion of the campaign. Both columns were operated by controlling the static pressure for the remainder of the campaign. The 20 Column (T-R2) became so unstable that concentrated nitric acid and oxalic-nitric acid flushes of the column were made on November 23. The flushing operation required a thirteen-hour equilibrium shutdown of the Second Thorium Cycle to complete. These flushes resulted in improved 20 Column operation, but did not free the interface float which was apparently held up by solids in the float guide. The float was replaced following the campaign.

6. Excessive Solvent Losses

Action taken to minimize solvent losses during the 1970 thorium campaign included installing a new aqueous selective jet for the TK-R1 to TK-R8 transfer, returning solvent from TK-R8 to TK-R1 for retreatment, and treating approximately 20,000 gallons of organic that would have normally been discarded to waste. However, in spite of these measures, the average solvent loss per operating day exceeded the normal value of 150 to 200 gpd for uranium-plutonium processing by nearly a factor of three at 585 gpd.

Measurement of the organic level in the underground organic wash waste receiver tank confirmed that this was the major loss point. Part of the losses can be attributed to excessive spinning of the solvent treatment systems while attempting to start up the solvent extraction systems. Prolonged startup delays due to problems in other areas of the plant resulted in spinning both solvent systems for approximately 40 days each.

Batch Solvent Recovery Operations

A spare, unbaffled 10-foot diameter by 9-1/4 foot tall tank was used to successfully recover batches of badly degraded solvent accumulated during process upsets and flushing. Agitation was provided by an off-center 300 RPM, ten-blade, 4-inch by 22-inch diameter, turbine agitator. Phase separations were made with a steam jet without provision for efficient decanting. A total of about 20,000 gallons were processed. The two operations discussed below were significant since the solvent previously would not have been considered for recovery.

The first of these operations followed a flush of the solvent extraction equipment with hot 60 weight percent nitric acid solution to convert the accumulated thorium-dibutylphosphate (Th-DBP) solids to a form which is soluble in plant solvent. A 500-gallon solvent batch was contacted with the acid in the Partition, Second Uranium, and Third Uranium Cycles to remove the Th-DBP. This solvent was combined with another 500 gallons of solvent which had passed through the Final Thorium Cycle concentrator. The combined solvents received a long series of equal volume washes (water, 0.5 $\underline{M}$ $NaOH$-0.02 $\underline{M}$ $KMnO_4$, 1.0 $\underline{M}$ HNO_3, 0.6 $\underline{M}$ $NaOH$-0.25 $\underline{M}$ Na_2CO_3, 0.25 $\underline{M}$ Na_2CO_3-0.03 $\underline{M}$ $KMnO_4$, 0.55 $\underline{M}$ $H_2C_2O_4$-0.3 $\underline{M}$ HNO_3) before the solvent quality was sufficiently high to permit transfer of the solvent to the No. 1 Solvent Treatment System. Of the chemical washes used, caustic-permanganate was the most effective and was adopted for routine use in this operation. Due to the large number of washes and decanting operations involved, about 400 gallons of solvent were lost to the aqueous waste.

In the second operation, approximately 1,000 gallons of solvent which had passed through the Final Thorium Cycle concentrator on a second occasion were combined with 1,500 gallons of solvent from emptying the columns prior to acid flushing, and 1,000 gallons of solvent misrouted to a waste tank during the subsequent startup. After water washing to remove thorium, the quality of this solvent, as measured by the plutonium retention analysis, was a factor of more than 10,000 poorer than that of the normal washed solvent. This solvent was washed with three 1,250-gallon batches of 0.5 $\underline{M}$ $NaOH$-0.02 $\underline{M}$ $KMnO_4$ solution. Following this treatment, the solvent's plutonium retention analysis was only about fifteen times normal. The solvent was then transferred to the No. 1 Solvent Treatment System in small volume batches. Less than 100 gallons of solvent were lost to the aqueous waste.

The water wash of this batch, and of others which contained high thorium concentrations, was transferred in small increments to the waste receiving tank. There, the solution was diluted five to one with water to prevent thorium precipitation during the subsequent neutralization prior to transfer to underground storage.

PLANT TURNAROUND

The flushing and equipment changes required for returning the plant to uranium-plutonium processing were less extensive than those required prior to the thoria campaign. Flushing operations were completed on January 16, 1971 about 30 days following shutdown. Less time would have been required but the manpower available was significantly reduced for several days due to the holidays. Startup of the plant on uranium-plutonium processing was delayed until April due primarily to the time required for determining the condition of the dissolver coils and the eventual replacement of one of the dissolvers. The Third Plutonium Solvent Extraction Cycle was also installed and tested during this period.

POST THORIUM FLUSH

Flushing to remove thorium and ^{233}U from the Purex Plant following the campaign was conducted in a manner similar to the uranium-plutonium removal preceding the campaign. The general steps were as follows:

1. Dissolved the residual thoria (ThO_2) in the dissolvers and head end equipment.

2. Stripped the solvent extraction columns to remove as much thorium and ^{233}U as practical prior to shutdown.

3. Flushed the solvent extraction section with hot concentrated nitric acid to remove thorium-dibutylphosphate.

4. Flushed the uranium concentration and loadout facility to remove ^{233}U.

5. Conducted a series of chemical flushes of the solvent extraction equipment.

The flushing operations started on December 16, 1970, and continued to January 16, 1971.

Head End Flush

Heel dissolutions or "cuts" were conducted after the completion of the normal dissolution cycles to remove the majority of the thoria. However, additional flushing removed approximately 5,000 pounds of thorium and 9 kilograms of ^{233}U from the dissolvers and the dissolver solution storage tanks (TK-D1, D3 and D4). Most of this material was recovered from solids in the storage tanks. The failure of four dissolver jet jumpers during the campaign due to erosion indicated solids were being transferred with the dissolver solution.

The dissolver and storage tank flush solution consisted of 11 $\underline{M}$ HNO_3-0.04 $\underline{M}$ KF-0.20 $\underline{M}$ ANN which was boiled in the dissolvers for at least two 36-hour periods. Samples of the solution were obtained following each boiling period. This solution was sent to the storage tanks where it was held at 90 ± 5 °C for 16-to-24-hour periods. The lower temperatures were required here since the storage tanks were not equipped with condensers. Flushing was terminated when the thorium removal rate was reduced to less than 0.5 pounds per hour at temperature. The feed concentrator (E-F11) and concentrated feed storage tanks (Tanks F12, D5, E6, H1) were also flushed with nitric-fluoride solution but proved to be relatively free of thoria solids. The flush solutions from the dissolver solution storage tanks were routed through the coating waste system (Tanks D2, E1, E3 and Centrifuges E2 and E4) to TK-E5 for neutralization and transfer to an underground storage tank. Flush solutions from the feed concentrator-feed storage tank system were also disposed of via TK-E5. No further flushing of this system was required.

Solvent Extraction Flush

Immediately following shutdown on December 12, 1970, the entire solvent extraction system was flushed with hot 60 weight percent nitric acid to remove thorium-dibutylphosphate. The flushes were maintained at 58 to 63 °C to comply with the maximum solvent temperature limit of 65 °C. The HAF Tank (TK-H1) and HA Column (T-H2) were flushed separately. Any organic present overflowed to the 1BXF Tank (TK-J3) with a total of about 300 gallons of nitric acid solutions to flush the HAP line. Additional nitric acid flush solution was added to the 1BXF Tank and routed through the remainder of the Partition Cycle, Second Uranium Cycle, and the Third Uranium Cycle. The organic displaced during this operation was routed to the No. 1 Solvent Treatment System (TK-G1). The organic was transferred from TK-G1 to a spare tank in the Waste Concentration-Acid Recovery System (TK-F13) for batch washing prior to its re-use and the aqueous acid phase was neutralized for transfer to an underground storage tank.

A third batch of nitric acid flush solution was added to the 2DF Tank and was routed through the Second Thorium Cycle. In this case, the 2D and 2E Columns were filled with acid solution and then contacted with 500 gallons of 2DX solvent. The solvent was then displaced to the No. 2 Solvent Treatment System where it was washed with the standard 0.27 M sodium carbonate-0.024 M potassium permanganate solution.

All nitric acid flush solutions were collected in feed tanks (TK-F7, F10) for processing through the Waste Concentration-Acid Recovery System. Most of the nitric acid was removed from the solution by adding water while boiling the solution in the concentrator. The off-gas was processed through the Acid Absorber to remove nitric acid which was then concentrated in the Vacuum Acid Fractionator for re-use. The bottoms solution was then neutralized and transferred to an underground non-boiling waste storage tank. Analyses of this solution prior to neutralization, revealed that only about 200 pounds of thorium and 500 grams of ^{233}U were removed in these flushes. The low product content was indicative of the effectiveness of the pre-shutdown strip of the solvent extraction system.

The solvent extraction system was then flushed with a series of solutions consisting of (a) five weight percent oxalic acid-three weight percent nitric acid, (b) water, (c) five weight percent sodium hydroxide-two weight percent sodium tartrate, and (d) water. For these flushes, the solvent extraction system was divided into eight equipment groupings:

1. HAF Tank (TK-H1), HA Column (T-H2), 1C Column (T-H3), and 1CU Concentrator (E-H4);
2. IBXF Tank (TK-J3), 1BX Column (T-J6), 1BS Column (T-J7), and 1BSU Tank (TK-J2);
3. 1BT Concentrator (E-J8), 2DF Tank (TK-K1) and 2D Column (T-K2);
4. 2E Column (T-K3), 2ET Concentrator (E-K4), 2TC Tank (TK-K5), and TNT Product Tank (TK-K6);
5. Second Uranium Cycle;
6. Third Uranium Cycle (through the 3B Column);
7. No. 1 Solvent Treatment System; and
8. No. 2 Solvent Treatment System.

The ^{233}U contained on the cation exchange column used for removal of thorium from the 3BU stream was eluted with about 180 liters (five to seven column volumes) of 0.5 $\underline{M}$ HNO_3. The solution was concentrated in the 3CU Concentrator (E-N6) and loaded out as product. The resin was then washed with about 250 liters of 4 $\underline{M}$ HNO_3 which was routed to the Waste Concentration-Acid Recovery System for further processing and disposal. This flush was used to remove the very small quantity of ^{233}U from the column and lines. No attempt was made to elute thorium from the resin which was slurried with 0.5 $\underline{M}$ HNO_3 and transferred from the column by jet to the organic wash waste tank (TK-R8) in the No. 2 Solvent Treatment System. In TK-R8 the solution was sampled, neutralized, and resampled prior to transfer to an underground waste storage tank. Analysis indicated the resin contained less than 10 grams of ^{233}U. The 3CU Concentrator (E-N6), 3UC Receiver Tank (TK-N7), the ^{233}U Product Sampling Tank (TK-L9), and the ^{233}U Product Loadout Tank (TK-L13) were then flushed repeatedly with dilute nitric acid solutions until a negligible amount of ^{233}U was removed. These flush solutions were also routed to the Waste Concentration-Nitric Acid Recovery System.

Water flushes of the 211-A Thorium Loadout Facility was routed to the TNT Product Tank (TK-K6) during flushing of that system. Water flushes of the trailers used for transporting thorium nitrate solutions were made into the thorium nitrate storage tanks until the trailers contained less than 50 pounds of thorium. The trailers were then sent to the Equipment Decontamination Facility for final cleaning with commercial decontaminating solutions.

EQUIPMENT CHANGES

The major equipment changes required for returning the plant to uranium-plutonium processing involved replacing one dissolver and routing the coil discharge from a second dissolver through the concentrator installed for the thoria campaign (E-F11) prior to disposal. These changes were necessitated by the discovery of small leaks in the coils of all three disolvers during the flushing operations. Inspection of one coil indicated mechanical failure due to cyclic stressing. The plant was eventually started using only two dissolvers with a replacement for the third dissolver on order.

Other required work related to the thoria campaign included the removal of numerous process blanks or locks and tags from "cold" side piping and the return of processing systems to normal functions. The movement of about 140 remote piping jumpers (Appendix D) was required to complete this work.

A third major piece of work completed during this period did not result from the thoria campaign. This item was the installation of a Third Plutonium Solvent Extraction Cycle to functionally replace the ion exchange system previously used for final plutonium purification. Extensive operational testing of this equipment was conducted prior to startup.

ANALYSES AND CONCLUSIONS

PRODUCT QUALITY

The quality of the ^{233}U product was generally excellent throughout the campaign. However, three problem areas encountered were: the ^{238}U content of the first 118 kilograms produced; the ^{232}U content of the specification product; and phosphorous analyses throughout the campaign. These problems are discussed below. A comparison of the average analytical values with the specification limits and 1966 results is also included in Table IV of Appendix L.

The quality of the thorium product was in many respects lower than desired. Contributing to the problem was the delay in obtaining meaningful analyses which precluded rework that may otherwise have been considered. A discussion of the problem areas and a comparison of analytical values with the targeted goals are presented below and in Table V.

^{233}U Product

1. ^{238}U Content

Isotopic analyses of solvent extraction feed and ^{233}U product batches indicated the pre-thoria campaign flushing effectively removed ^{238}U from plant equipment. The data in the table below show that the ^{238}U contamination from the dissolvers and feed tanks had decreased to less than 0.1 percent (based on ^{233}U) after thoria containing only approximately 70 kilograms of ^{233}U had been dissolved. The ^{238}U content of the product equalled the specification limit of 0.5 percent after 118 kilograms had been produced. As a result, a blend of approximately the last 55 kilograms produced during the "non-specification" portion of the campaign satisfied all Division of Naval Reactor specifications if a low phosphorous value is assumed. The ^{238}U content of the 1966 product is included in the table for comparison.

PERCENT ^{238}U IN FEED AND PRODUCT VERSUS THROUGHPUT

Throughput, kgs ^{233}U	Thoria Feed	Dissolved Feed	Product 1970	Product 1966*
10		7.1	6.7	5.2
20	0.7	1.6	2.2	2.2
30	0.65	3.1	1.7	1.4
40		1.8	1.4	1.3
50	0.60	1.0	1.0	1.5
60		1.0	0.8	1.5
70	0.56	0.6	0.8	1.4
80			0.7	1.3
90	0.51		0.6	1.2
100	0.45		0.57	1.1
110	0.40		0.51	0.9

---

* Reached a minimum of 0.63 percent at 130 kilograms and then increased due to an increase in the ^{238}U content of the thoria feed.

Data obtained during the campaign indicated the minimum bleed-in rate of ^{238}U from plant equipment was 2.2 to 2.5 grams per day which occurred after processing about 210 tons of thorium. Most of the ^{238}U originated in the dissolvers and feed storage tanks.

2. ^{232}U Content

Based on calculations performed by reactor personnel, the overall average ^{232}U content of the specification product should have been 7.78 parts per million parts uranium (ppmpU). The actual average was 8.63 ppmpU versus a specification maximum limit of 8.0 ppmpU. This deviation was due to variance in reactor operating parameters and could not be corrected in the Purex Plant.

3. Phosphorous Analyses

The locally reported phosphorous content of some of the first product batches produced during the "non-specification" portion of the campaign exceeded the specification limit of 150 ppmpUO_2 by a factor of two to three. Corrective measures were taken to reduce the transfer of organic from the 3B Column to the product concentrator as described in the section on Reduction of 3BU Organic Entrainment. These measures were apparently beneficial as the locally reported phosphorous content of subsequent product batches averaged only 116 ppmpUO_2 if two batches produced immediately following a startup and four batches produced immediately preceding a shutdown are excluded. The high values were not confirmed by the customer.

4. Analytical Results

The averaged analytical results of the ^{233}U product produced during both the non-specification and specification (DNR) portions of the campaign are listed and compared with the specification limits and 1966 averages in Table IV. With the exception of phosphorous and ^{232}U, all requirements were exceeded by the DNR product.

Thorium Nitrate Product

The thorium nitrate product quality information is presented in Table V of Appendix L. For comparison purposes, the target specifications and 1966 campaign product analyses are included.

With a few exceptions, the thorium product met the targeted specifications. The most significant exception was the ^{106}Ru-Rh content which averaged about 180 percent of the specification limit. As reported in the section on Normal Operating Performance and Capacity of the Final Thorium Cycle, the average ruthenium decontamination factor obtained across the Second Thorium Cycle was only 12. The ^{233}U content averaged below the 20 parts per million (ppm) limit but exceeded this by 75 percent in the storage tank containing the thorium product batches produced immediately following the start of the DNR or specification portion of the campaign. Also, the total uranium (excluding ^{233}U) content of the first thorium product was 63 ppm versus the target limit of 10 ppm. The contamination resulted from residual uranium held up in the Second Thorium Cycle equipment which was used for the Second Uranium Cycle during previous uranium-plutonium processing.

As shown in Table V, the reported analyses for the 1970 campaign compare reasonably well with the results reported for the 1966 campaign product. The higher fission product content of the 1966 product was primarily due to processing "hotter" feed. The higher ^{233}U levels for the 1970 campaign product resulted from partitioning problems associated with low salting strength in the 1BX Column (T-J6) during startup and shutdown periods.

The listed Total Metallic Impurity (TMI) data are typical values based on results for individual product batches. A wide variation was noted in these results. For example, values exceeding 500 ppmp thorium were reported for several elements (calcium, chromium, iron, magnesium, silicon) in some batches. Samples of the thorium product solutions in the storage tanks were not analyzed for TMI. Therefore, it was tentatively concluded that the iron content exceeds the 100 ppm specification limit.

ACCOUNTABILITY AND MATERIAL BALANCES

As was the case in 1966, fairly close agreement was obtained between the measured product output (loadout plus waste losses) and the reactor input data for both thorium and ^{233}U. On this basis, overall material balance was minus 2.3 percent for thorium and plus 1.2 percent for ^{233}U.

Product Input

The input of thorium and uranium to the Purex Plant was measured with two systems. The reactor bucket loading summary listed the tons of thoria and grams of uranium in each bucket and was the official input measurement to the Purex Plant. As a secondary measurement the dissolved thorium nitrate solution was batched through a sample tank (TK-D5) where thorium and ^{233}U analyses were made for accountability. A comparison of reactor measurements and the sample tank measurements for each portion of the campaign, and for the total, indicated the thoria and uranium input measured in TK-D5 was low in each case. Some of this discrepancy could be accounted for by undissolved dissolver heels, solids carryover to feed solution storage tanks (TK-D3 and TK-D4), and the coating waste loss. However, the instrumentation on Tank D5 did not allow an accuracy greater than about ± two percent. Thus, if the material in the post-campaign head end flush and the coating waste were included in the TK-D5 measurements, the differences between the reactor measurement and the Purex measurement were within the capability of the feed measurement system, i.e., the Purex thorium measurement was 1.7 percent low and the uranium measurement was 0.3 percent high. The comparisons of the input measurements are presented in Tables VI through VIII.

Product Loadout

Both the thorium and uranium products were measured as loaded out from the Purex Plant. The thorium measurement was made in Tank K6 and the uranium measurement was made from the batch analysis of Tank L9 and the net weight of product solution in each product shipping container. The tank K6 measurement was not as accurate as those made in Tank D5 and, as a result, the product inventory in the thorium nitrate storage tanks did not agree with the combined TK-K6 loadout for the two portions of the 1970 Thoria Campaign. The thorium product loadout data indicated 119.8 tons and 274.9 tons, were produced during the non-specification and specification portions, respectively, for a total of 394.7 tons. The final inventory of the storage tanks indicated a revised total loadout of 397.4 tons.

The uranium loadout measurements were done as accurately as possible to form the basis for shipping the product to Oak Ridge National Laboratory. The loadout during the non-specification portion of the campaign was 161.3 kilograms and during the specification portion 436.2 kilograms for a total of 597.5 kilograms.

Waste Losses

The waste losses from the Purex Plant were measured at five points:

1. Tank F18, losses consisted of products contained in high-level waste from all solvent extraction cycles, sump waste, and solvent extraction cycle flushes.

2. Tank E5, losses consisted of products contained in coating waste and head end flushes.

3. Tank G8, losses consisted of products contained in waste and flushes from the number one solvent treatment system.

4. Tank R8, losses consisted of products contained in waste and flushes from the number two organic wash system.

5. Tanks U3 and U4, losses consisted of products contained in laboratory waste, flushes, and stack condensate drainage.

All waste losses from the non-specification and specification portions of the campaign are listed in Tables VI and VII. The major losses from Tank F18 during both portions of the campaign were caused by several process upsets which resulted in the gross loss of product. Tank G8 was another high loss point due to major process upsets in the uranium stripping (1C) Column in the first solvent extraction cycle.

Material Balance and Material Unaccounted For (MUF)

The overall material balance for the non-specification and specification portions and the complete thoria campaign are presented in Tables VI through VIII. The discrepancy in the material balance is referred to as MUF. The MUF is equal to the input minus the output and waste losses. The MUF is calculated on the basis of both the reactor input measurements and the Tank D5 measurement. Although the official MUF is measured by using the reactor shipping data, the TK-D5 MUF is a useful means of evaluating the plant measurement system. In calculating the TK-D5 MUF, the head end waste losses, which occur before the plant accountability point, were added to the TK-D5 input measurement.

The uranium MUF for the non-specification and specification portions of the campaign were minus 2.7 percent and plus 2.8 percent, respectively (based on reactor input measurements). Part of the MUF difference was caused by the undissolved heels in the three dissolvers and the feed storage tanks at the end of the non-specification portion of the campaign. This resulted in low measurements for the non-specification portion of the campaign when the material in the post-campaign flushes was included. The overall campaign uranium MUF was plus 1.2 percent. The overall campaign thorium MUF was minus 2.3 percent.

WASTE VOLUMES

The volume of liquid waste with its estimated salt content that was transferred to non-boiling underground storage tanks from each of the five disposal points listed in the Waste Losses section is presented in Table IX. A comparison of the actual waste volumes with those predicted in the flowsheet document(8) is presented in Table X.

The data show that the actual waste volume exceeded the predicted total by 64.4 percent. The major sources of the overage were the organic wash wastes and laboratory and miscellaneous wastes. Sump wastes, coating wastes, and post-campaign flushes also exceeded the predicted values by significant amounts. The increased coating waste volume was due partly to steam dilution that was not included in the predicted volume.

The organic wash wastes accounted for nearly half (31.2 percent) of the excess volume. The major cause of the excessive volume was prolonged "spinning" (described in the section on Solvent "Spinning") during down periods. The batch treatment of organic recovered from process upsets also contributed significant waste volumes.

Laboratory and miscellaneous wastes accounted for about one-quarter (16.5 percent) of the excess volume. This stream was not included in the predicted waste since it was previously routed to an underground trench or crib. A detailed review of the laboratory drain system plus an estimate of routine usage revealed that approximately 60 percent of this volume originated as cooling water to a reflux condenser which is a part of platinum recovery equipment used only during thoria processing.

ENVIRONMENTAL RELEASES

The amount of radioactivity released to the environment during the 1970 Thoria Campaign was, in general, higher in both the liquid and gaseous effluents than that normally released during a comparable uranium-plutonium processing period. The increase was due primarily to required changes in the processing operation and to an increase in volatile radionuclides.

Gaseous Effluents

Routine total alpha and total beta analyses of gaseous effluents were made during the thorium campaign. Both of these measurements increased above normal levels in the main building exhaust stream. Periodic checks disclosed that the majority of this abnormal activity had the short half-life characteristic of ^{212}Pb. The mechanism postulated involves the volatilization of ^{220}Rn (a daughter of ^{228}Th) from boiled or sparged process solutions. The 56-second half-life of the ^{220}Rn allowed only partial decomposition in the tank vapor spaces and off-gas lines. This permitted some of the ^{220}Rn to decompose downstream of the ventilation filters. Thus, the decomposition products were caught on the air samplers. Simple calculations based on plant inventory yielded approximate agreement with the measured releases. Calculations also indicated that the radionuclide concentrations at ground level did not exceed the limits for industrial exposure. However, some ^{103}Ru and ^{106}Ru-Rh were also emitted (probably from the acid boil-off system in the feed preparation cycle) and deposited on the ground which resulted in low-level ground contamination as described in the section on Ruthenium Volatilization, Page 33. The total alpha and total beta content of all gaseous effluents emitted during 1970 are shown in Table XI.

Liquid Effluents

The volume, uranium, plutonium, and certain radionuclides contained in Purex Plant liquid effluents discharged to the ground (surface or subsurface) during 1970 are listed in Table XII. The data shown that the reported values of many of these constituents were higher during the thoria campaign than for comparable uranium-plutonium processing periods. For example, the reported ^{233}U content of all streams was consistently higher than the normal plutonium content of the same streams. In all cases, the actual values released are probably lower than reported due to the relatively high detection limits and large volumes.

NUCLEAR AND CHEMICAL SAFETY

There was no occurrence during thorium processing that resulted in an approach to criticality. However, a number of violations of the specifications occurred and are discussed below. Two other incidents had serious chemical safety implications; one in terms of corrosion, the other in terms of combustible materials in a burial box.

Violations of the criticality specifications occurred in TK-J3 and in the Second Thorium Cycle feed. As mentioned previously (Nuclear and Chemical Safety section under Process Technology, Page 12), the specifications were related to safe limits in down stream vessels. Originally, TK-J3 had a 3,000-gram per ton thorium limit <u>and</u> a 0.09-gram per liter limit, imposed by the Safety Analysis Report.(16) During startup and shutdown, ^{233}U was recycled to TK-J3 without thorium. Hence, the limit of 3,000 grams per ton was exceeded on two occasions. Both the Safety Analysis Report and the criticality specifications were reworded to permit either the gram per ton limit <u>or</u> the concentration limit to apply.

The violations in the Second Thorium Cycle arose in a similar fashion and were similarly resolved. During transient conditions, partial partitioning failure in the 1BX Column (T-J6) occurred. In several instances the 30 grams ^{233}U per ton of thorium limit was exceeded in the 2DF. The specification limit was established based on the Safety Analysis Report assumption that the ^{233}U would be partitioned from the thorium in the 2E Column (T-K3). This limit would safeguard the organic treatment vessel (TK-R1) for several hours. An analysis of the system disclosed that the limit was unnecessary. The uranium was stripped out in the 2E Column with the thorium under flowsheet conditions. The specifications were revised to increase the limit to 3,000 grams ^{233}U per ton of thorium. Safety was assured in TK-R1 by maintaining its own batch limit of 500 grams ^{233}U.

During one limit violation in the Second Thorium Cycle (K Cell), however, a more serious violation of administrative procedure occurred. There was a failure to notify the engineering manager to obtain his concurrence with the remedial actions taken. This indicated a somewhat casual attitude toward the specification. In addition to receiving the revised specification, personnel were reminded of their responsibilities in the above regard.

The addition of combustibles to a burial box represented a violation of criticality prevention and of chemical safety specifications. The canyon burial box normally received failed motors, jumpers and miscellaneous items of a similar nature. Several broken cardboard boxes were deposited in a box on one occasion. Criticality safety required physical separation of hydrogenous material from fissile material with the greater-than-minimum critical mass limit permitted for the box. Chemical safety considerations required that hydrogenous material be segregated to prevent contact with nitric acid or other oxidants. Investigation of the box and its contents revealed that the fissile content was minimal and strong oxidants were absent. Burial of the box was permitted under alternate specifications permitting unsegregated hydrogenous material with a lower fissile material limit. The persons responsible were reminded of the purpose for the limits violated.

The other significant incident from the standpoint of chemical safety resulted in marked corrosion of the feed concentrator (E-F11-1). During an outage, a feed batch was prepared and stored in the feed concentrator at elevated temperatures. Over the eight-day period an estimated 2.5 mil uniform corrosion occurred. Processing of the feed batch resulted in high ^{144}Ce levels in the thorium product due to cerium extraction through oxidation by corrosion product chromate ion. The high ^{144}Ce product batches resulted in truck driver exposure to radiation levels of about 10 millirads per hour as measured by a Hanford-type "cutie pie" (CP) radiation instrument.

CORROSION AND FLUORIDE PATH

Potassium fluoride was added to the dissolver solution to facilitate thoria dissolution. To reduce corrosion rates, aluminum nitrate was also added as a fluoride complexant at a ratio of three moles of aluminum per mole of fluoride. Although this complexed most of the fluoride, some free fluoride was still present after the feed concentration-denitration operation as indicated by analyses of the feed adjustment tank (TK-D5) sample composites.

Since a very low fluoride concentration was measured in the recovered acid, most of the fluoride must have exited in the high-level waste (1WW). Data obtained during the 1966 campaign indicated the amount of fluoride remaining in the bottom liquid over-flow stream of both the feed concentration-denitration and high-level waste evaporators exceeded that in the condensate by a factor of 100 to 1000.(32) These results agreed with previously reported data.(62, 69)

During the 1970 campaign, iron analyses were reported for samples of the feed adjustment tank (TK-D5), the acid absorber product (AAA) receiver tank (TK-F3), and the Purex recovered acid tank (TK-U1, U2) contents. These results were averaged and converted to pounds of 304L stainless steel. Corrosion rates were then calculated for each of the three dissolver coils, the feed concentrator (E-F11) tube bundle, the acid absorber (T-F5), and acid fractionator (T-U6) reboiler. Assuming an equal contribution of stainless steel to the feed solution from each of the dissolver coils and the concentrator tube bundle, the calculated average uniform corrosion rate of these surfaces was approximately 2.5 mils per month. Based upon the AAA and recovered acid iron analyses, the uniform corrosion rates of the acid absorber and the acid fractionator were 7.1 and 17.5 mils per month, respectively. The latter value is believed to be considerably higher than the actual rate as only "less than" results were obtained for the recovered acid iron analyses. No data were accumulated with respect to vapor phase corrosion.

During 1966, iron analyses of both the terminal dissolver solutions and the feed solutions were available which permitted individual estimates of dissolver coil and feed concentration-denitration coil corrosion rates. These rates were reported as five to ten mils per month and ten to 20 mils per month, respectively. Also, the reported 1966 corrosion rate of the vacuum fractionator tube bundle was only 0.5 to 2.0 mils per month.(32)

Rust colored samples of two consecutive feed batches were obtained during the 1970 campaign. Based on an iron analysis, the first batch contained 156 pounds of iron which is equivalent to 220 pounds of stainless steel. Investigation revealed that this feed batch had been held in the feed concentrator for eight days at about 80 °C. The second rust colored sample was apparently due to residue or "heel" remaining from the first batch. The color was due to dichromate solids.

The calculated uniform corrosion rate of the feed concentrator (E-F11) during this eight-day period was 9.5 mils per month. The total surface area in contact with the 1700 gallons of solution was used in this calculation. The calculated corrosion rate using only the tube bundle surface was 16.8 mils per month. These values were not included when calculating the average tube bundle corrosion rate (2.5 mils per month) for the campaign.

Testing conducted following the thoria campaign indicated leaks in the coils of all three dissolvers. The holes were quite small as the leak rates at 90 psig were only 18, 7, and 16 gallons per hour for A3, B3 and C3 dissolvers. Although the increased corrosion due to the use of fluoride as required during the thoria and Zirflex campaigns may have contributed to these failures; inspection of one coil indicated mechanical failure due to thermal cycling stresses.

FUTURE PROCESSING RECOMMENDATIONS

During the course of the campaign, various opportunities for process improvement were noted. Very few of these could be implemented because the press of production precluded time to complete the required development work. In addition, some problems arose which were not completely solved. These various aspects are discussed in the following paragraphs with the intent that these items be given consideration in the event that another thoria processing campaign is undertaken.

Wafers

The superior dissolution characteristics of the wafer targets makes these an obvious choice for a future run. Jet dipleg plugging was observed, and it is possible that partially dissolved wafers were drawn into the dipleg during solution transfer. It is suggested that a screen or other guard system be developed to reduce the likelihood of dipleg pluggage.

1C Cartridge

Even with the 1C Column (T-H3) cartridge essentially free of thorium-DBP solids, some evidence of instability was noted at a 0.85 CF. A new cartridge design is needed to provide improved capacity. This would allow a closer match of dissolution and solvent extraction capabilities.

Solvent Washing

The carbonate-permanganate wash solutions were changed on a fixed time basis. Changeout based on solution pH would probably save materials and waste volumes. In addition, particularly during pre-startup solvent spinning, some evidence was noted of significant permanganate leaving the 10 or 20 Column in the organic. When this permanganate decomposed to manganese dioxide solids, pluggage problems were noted on downstream filters. It also is possible that this contributed to high plutonium retention values. In any event, the need for permanganate or at least as much as was used is subject to additional study.

Material Balances

The detailed material balances kept during the early portion of the run were of only minimal value, primarily because of the uncertainties in flowmeter calibration. It is suggested that the shift manpower would be better utilized by providing additional process analysis and support and that a daily plotting of stream concentrations would provide more meaningful information. Routine material balances on G Cell plus routine reviews of the F Cell waste system should be maintained.

Ruthenium

Additional studies should be made to improve ruthenium decontamination in the solvent extraction battery and to reduce ruthenium volatilization from the feed denitration equipment. An alternate would be to route the denitration equipment off-gas to the Backup Recovery Facility.

Protactinium

The need for a protactinium removal step would be increased with shorter cooling times and should be reviewed in light of the requirements of the specific campaign.

Solvent Degradation

A modest change in the organic residence time under high acid conditions in the Co-Decontamination and Partition Cycle made a significant change in the deposition rate of thorium-DBP solids in the 1C Column. It is possible that further benefits such as the capability for processing shorter cooled targets could be obtained if the effect of thorium concentration, time, and acid concentration upon the degradation rate of TBP were known.

Also, the degradation of solvent in the 2A and 3A Columns could be reduced if mixing pots were installed in the 2AF and 3AF lines. The pots would eliminate the need for adding concentrated acid solutions to organic continuous columns. It is felt that this practice contributed to the formation of thorium-DBP solids in the 2B and 3B Columns.

Third Plutonium Cycle

Use of the recently installed Third Plutonium Cycle Columns (T-L4 and L5) for the Third Uranium Cycle would eliminate the need to use J Cell Package columns (T-J22 and J23) for the Second Uranium Cycle. This change would be desirable as these columns are of marginal capacity for the flow required. In this scheme, the T-L1 and T-L2 Columns which were used for the Third Uranium Cycle in previous campaigns would be used for the Second Uranium Cycle. This change might result in fewer piping changes to conduct the campaign and some flowsheet changes to accomodate the equipment.

REFERENCES

1. ARH-[redacted] (Unclassified), January 7, 1971, R. A. Pease, "Improvement of the Vacuum Acid Fractionator Performance"

2. Letter, [redacted] 10, 1970, G. L. Ritter to M. K. Harmon, "Computer Optimized Dissolver Charge Schedule - 1970 Thorium Campaign"

3. ARH-1774 (Unclassified), August 10, 1970, G. A. Nicholson, "Quality Control Program Purex Thoria Processing"

4. ARH-1775 (Unclassified), July 29, 1970, R. R. Jackson, "Process Control Guidelines for CY 1970 Thorium Campaign"

[redacted]

[redacted]

7. [redacted]

8. ARH-1748 (Unclassified), July 10, 1970, G. F. Smith, "Purex Plant Chemical Flowsheet for the Thorium Campaign"

[redacted]

10. Letter, July 2, 1970, H. M. Jones to Distribution, "1970 Thorium Campaign Instrumentation Calibration List"

[redacted]

12. ARH-1757 (Unclassified), June 30, 1970, R. E. Van der Cook and G. L. Ritter, "Process Specifications and Standards for the 1970 Thorium Campaign in the Purex Plant"

13. Letter, June 22, 1970, G. L. Ritter to C. W. Malody, "Final Process Control Blanking Schedule - 1970 Thorium Campaign"

14. "Quality Control Plan for the Thorium Campaign - Technical Services Section - Purex Analytical Laboratory," June 5, 1970, J. C. Abercrombie, undocumented, unclassified.

15. ARH-1514 (Unclassified), June 1, 1970, W. E. Matheison, et al., "Criticality Prevention Specifications, Thorium-Uranium-233 Separations in the Purex Plant"

[redacted]

18. Letter, May 19, 1970, H. M. Jones to G. L. Ritter, "CY 1970 Thorium In-Line Monitors"

[redacted]

20 [redacted]

[redacted] 1970, J. P. Schmidt to G. C. Oberg, "[redacted]ion Markers for the Thoria Wafer Buckets (1970 Thorium Campaigh)"

22. Letter, April 9, 1970, T. F. Scanlon to [redacted] Harmon, "Minimum Technical Requirements of Uranium-233 for Light Water Breeder Reactor (LWBR) Demonstration Core"

[redacted]

24 [redacted]

25. Le[redacted]Oberg to R. W. McCullugh, "Blending of Wafer and Powder Material, 1970 Thorium Campaign"

26. Undocumented Report, November 21, 1969, M. K. Harmon and M. L. Madeen, "Trip Report - ORNL and Savannah River - DNR ^{233}U program - LWBR Program"

[redacted]

[redacted]

[redacted]

[redacted]

[redacted]

32. [redacted] of the First [redacted]

33. [redacted]

34. ISO-419 [redacted], December 1967, Purex Process Subsection, "Thorium Process Operations Report - Purex"

35. [redacted] J. P. Phillips and H. D. Huber, "Dissolution of Thorium Oxide"

36. Letter, July 16, 1966, H. C. Rathvon to L. L. Zahn, "Flushing of Thorium Solids"

37. Letter, July 7, 1966, M. H. Campbell to J. B. Kendall, "Dissolution of Thorium-DBP Solids from Purex"

38. ISO-SA-3 REV, May 1966, H. C. Rathvon, et al., "Recovery of Uranium-233 from Irradiated Thoria"

39. [redacted]

40. [redacted] "

41. Letter, March 7, 1966, J. B. Kendall to L. L. Zahn, "Purex Thorium Processing Fluoride Corrosion Equipment"

42. Letter, February 16, 1966, R. F. Maness to J. B. Kendall, "Corrosion During Thorium Processing - Purex"

43. [redacted] Dissolvent Solutions"

44. Letter, August 13, 1965, C. A. Colvin to J. B. Kendall, "Solid Material in Purex H4 Concentrator"

45. [redacted]

46. RL-SEP-267 Pt. 3 (Secret), July 3, 1965, J. B. Kendall, "Engineering Study Thorium Processing - Purex Part III Equipment Capacities"

47. RL-SEP-267 Pt. 4 (Unclassified), May 26, 1965, R. D. Ehrlich, et al., "Engineering Study Thorium Processing - Purex, Part IV Process Design"

48. RL-SEP-267 Pt. 1 (Unclassified), May 12, 1965, J. B. Kendall, "Engineering Study Thorium Processing - Purex I Flowsheet"

[redacted]

50. T. R. McKenzie, March 12, 1965, "Some Effects of Thorium Feed, Acid and Acid-Deficient on Solvent Degradation and FP Decontamination"

[redacted]

52. HW-80658 (Unclassified), January 31, 1964, R. F. Maness, "Corrosion of 304-L Stainless Steel and HAPO-20 in Acid Fluoride Systems"

53. HW-80407 Pt. 1 (Secret), January 17, 1964, R. D. Ehrlich, et al., "Design Criteria - CGC-124 - Increased Processing Flexibility - Purex"

54. ORNL-3418, June 7, 1963, R. E. Blanco, et al., "Aqueous Processing of Thorium Fuels"

[redacted]

56. HW-74000 Pt. 2 (Unclassified), July 16, 1962, L. R. Michels and W. R. Kendall, "New Sectionalized Purex Concentrators Part II - Detail Design Drawings"

57. ORNL-3155, May 11, 1962, R. H. Rainey and J. G. Moore, "Laboratory Development of the Acid Thorex Process for Recovery of Consolidated Edison Thorium Reactor Fuel"

[redacted]

59. Nuclear Science and Engineering, (VOL. 10), 1961, R. H. Rainey and J. G. Moore, "Solvent Extraction Separation of ^{233}U and Thorium from Fission Products by Means of Tributyl Phosphate," Pages 505-510.

60. Reactor Handbook, Vol. II, "Fuel Reprocessing", 1961; Interscience Publishers, Inc., New York

61. ORNL-3045, January 17, 1961, A. D. Ryan, "McCabe-Thiele Graphical Solution of Uranium-Thorium Partitioning from 30% TBP Amsco Solvent"

62. DP-491, October 1960, E. S. Occhipinti, "Reprocessing of Power Fuels, Tenth Quarterly Progress Report, January 1 to April 1, 1960"

63. ORNL-2844, Undated, J. L. English, "Thorex Pilot Plant Corrosion Studies: Corrosion of Types 304L, 309 SCB and 347 Stainless Steel During Development and Production-Development Periods"

64. ORNL-2673, Undated, J. L. English, "Thorex Plant Corrosion Studies: Corrosion of Types 304L, 309 SCB Stainless Steel During Production and Development Periods"

65. DP-250, November 1957, E. K. Dukes, "The Formation and Effects of Dibutyl Phosphate in Solvent Extraction"

66. HW-51386 (Unclassified), July 3, 1957, J. J. Schefcik, "Performance of a Turbomixer for Continuous Washing of Purex Organic"

67. HW-47813 (Unclassified), January 14, 1957, R. L. Moore, "Liquid Vapor Equilibrium in the System Nitric Acid-Water Trace Fluoride"

[redacted]

69. HW-47813 (Unclassified), January 14, 1957, R. L. Moore, "Liquid Vapor Equilibrium in the System Nitric Acid-Water Trace Fluoride"

70. KAPL-1306, May 1956, W. O. Haas, Jr. and D. J. Smith, "Thorex Process Development at KAPL"

71. 1955 Geneva Conference, Vol. IX (P/540), A. T. Gresky (USA), "Laboratory Development of the Acid Thorex Process for Recovery of Thorium Reactor Fuel," Pages 367-371.

72. The Reactor Handbook, Vol. 2 - Engineering, U. S. Atomic Energy Commission AEC-3646, May 1955, Pages 643-645.

APPENDIX A

EQUIPMENT DESCRIPTION

DISSOLVERS(53)

The three Purex multipurpose dissolvers are each approximately 24 feet tall (13.5 feet to overflow) by nine feet in diameter (Figure 8). Each vessel, annular in configuration, consists of an inner concrete moderator, a metal receiving annulus, and an outer solution annulus. The inner moderator is a hollow concrete cylinder encased in a 304-L stainless steel (SS) cone with a cadmium sheet liner. The 12-inch metal annulus receives the fuel elements as charged onto the grate. The outer 13-inch solution annulus contains the coils for heating and cooling dissolver solutions, chemical addition lines and jetout routes, and the instrumentation. Both annuli contain lifter-circulator type spargers, utilizing either steam or air for solution agitation (Figure 9).

Annular dissolvers are designed to be geometrically safe for uranium enriched to 0.94 percent ^{235}U. Fuel of greater enrichment requires administrative controls on amount of material charged to a dissolver and, for co-product processing, installation of an insert decreasing the width of the metal annulus.

[redacted]

To convert from uranium fuels processing to thoria target processi[illegible] in the dissolvers required only minor modifications. The decladding waste jetout jumper dipleg was shortened to about six inches off the bottom of the dissolver to diminish the amount of entrained thoria particles jetted to the decladding waste handling system. The weight factor dip tube was shortened eight inches to lessen dip tube pluggage by any thoria cake buildup in the solution annulus.

CENTRIFUGE

The Purex centrifuge used for thoria recovery from the dissolver decladding waste was manufactured by Bird and modified for remote installation and operation. It was a solid bowl type (48-inch bowl diameter) with a maximum volume of 90 gallons. The centrifuge held about 20 gallons of solids (skimmed). Operation was at 1,200 revolutions per minute (rpm).

SOLVENT EXTRACTION COLUMNS

Design parameters for the pulse columns used in Purex are presented in Table I. The columns vary from seven to 34 inches in diameter, with contacting heights between 18 and 32 feet. The column cartridges are described in Table II. The cartridges consist of fixed, spaced, horizontal, perforated plates. The various service requirements in Purex dictate a variety of plate spacings (1/2-inch to four-inch), plate materials, types of plates (sieve, nozzle, and louver), and plate materials (stainless steel or plastic). The cartridges are generally removable.

The pulsing is imparted to the solution in the column by a graphite ringed piston, reciprocated by a conventional crank arrangement. The crank is turned by a variable speed electrical motor with the speed controlled by varying the frequency of the alternating current. A typical column and pulser arrangement is shown in Figure 10.

CONCENTRATORS

The concentrators used in Purex are steam heated, vertical tube, thermal recirculation type evaporators. The boiler in each concentrator is composed of a draft tube or downcomer to which are attached two reboiler sections containing steam chests, and an overflow chamber containing most of the instrumentation as well as the overflow line. Steam is supplied to the chest side of the tube bundle which contains 687 tubes, (1-1/4 inch OD x 10 feet 10 inches) and is fabricated of stainless steel (as is the vessel itself). The bundle is expected to operate at an overall heat transfer coefficient (U) of 100 to 300 BTU/hour-foot^2-°F.

A tower is located on top of the boiler to remove entrained liquid from the off-gas stream. The tower was an integral part of the original concentrator, but the second generation or sectionalized concentrator is built with a detachable tower. The 1BT and 2ET concentrators were of the original type; the 1WW, Feed, and 1UC concentrators were sectionalized. The towers on the sectionalized concentrators can be used to remove entrained liquid from an off-gas stream and/or to remove solvent from an aqueous feed by steam stripping.

The 1BT and 2ET concentrators were identical. Each contained about 4,000 gallons and were steam heated with 29 psig steam. The towers were 14 feet by nine feet in diameter and contained seven bubble-cap trays (six operated wet) plus an upper packed section. The six "wet" trays were used in steam stripping the incoming feed to remove entrained solvent. The seventh tray and upper three-feet thick section packed with one-inch Raschig rings were relied on for de-entrainment.

The 1WW Concentrator was a typical acid service sectionalized concentrator and is shown in Figure 11. It had an operating volume of about 2,600 gallons and was heated with 39 psig steam. De-entrainment was provided by a complex arrangement of baffles in the enlarged settling chamber of the downcomer, coupled with an attached tower and auxiliary demister. The remotable tower was designed for acid service and contained a six-inch thick tantalum wire (five mil) mesh pad with upper and lower water sprays. Seated on top of the tower was an auxiliary demister containing a six-inch thick tantalum wire (six mil) mesh pad. Fission product (excluding ruthenium) concentrations in the overheads condensate were on the order of 10^5 to 10^6 lower than in the concentrator bottoms overflow stream.

Concentration and acid stripping of the feed solution was conducted in a concentrator similar to the 1WW Concentrator. The major modification consisted of installing a cooling coil and a submerged, high pressure, water-motivated transfer jet instead of a tube bundle in the west (left) reboiler well. The cooling coil was necessary for the high heat load encountered during cooling portions of the feed preparation cycle. The water motivated transfer jet was necessary for feed dilution. The steam supply to the remaining tube bundle was at 80 psig in order to achieve the necessary thorium concentrations for satisfactory nitric acid stripping. The tower was similar to the 1WW concentrator tower. However, the wire mesh pad was composed of Tophet M* (10 mil wire), which in laboratory tests proved to be superior to tantalum for acid-fluoride service in that it did not embrittle. Also, it was considerably cheaper.

The 1CU sectionalized concentrator boiler was modified by dropping the reboiler wells down to a "flat bottom" configuration.(27) This change was made for nuclear safety purposes, (specifically to increase the safe ^{233}U mass allowed for uniform precipitation). The tower differed from the 1WW Concentrator tower in that it was designed for uranium service. That is, it contained six bubble-cap trays in addition to the upper tantalum wire mesh demister pad. As was the case with the 1BT and 2ET Concentrators, the incoming feed was countercurrently steam-stripped for solvent removal.

ACID RECOVERY AND CONCENTRATION

An acid absorber (T-F5) and vacuum fractionator (T-U6) plus associated pump tanks comprised the nitric acid recovery and recycle system. No major equipment modifications were required for the thoria campaign.

The F5 acid absorber was a modified acid absorber and fractionator. The original design called for a 27 foot, seven inch high vessel, consisting of a bottom reboiler, a three foot six inch diameter by ten foot ten inch rectifying section in the middle, and a ten foot diameter by nine foot three inch high absorber section. In the original design, the reboiler proved to be subject to severe corrosion. The replacement tower, in use since plant startup, did not have a reboiler section. Consequently, for this service the middle section is superfluous.

The upper, enlarged section contained seven trays on 12-inch spacing. Each tray had 156 bubble caps (5 1/8 inch OD). The 16 inch ID vapor inlet line was located just below the lowest diameter tray. The off-gas exited the top of the tower via a 16 inch ID pipe to a condenser.

The U6 vacuum acid fractionator was an insulated eight foot ten inch OD by 32 foot three inch high tower. The upper 19 foot six inch section contained

---

* 65 nickel, 30 chromium, 5 molybdenum.

14 bubble-cap trays. Each tray had 86, 5 1/8 inch ID bubble caps. Heat was supplied to the fractionator bottoms by an external vertical tube reboiler. The two foot three inch ID by eight foot six inch reboiler contained 372 one-inch OD tubes. Saturated steam was supplied to the shell side. A two-stage jet system was used to maintain an absolute pressure of 100 millimeters mercury at the top of the tower.

N CELL AND PR ROOM EQUIPMENT

Equipment statistics for the ^{233}U product concentration and loadout systems are given in Appendix B. In preparation for ^{233}U product handling, the following new equipment was installed:

. T-N50, cation exchange column,

. TK-L9, product receiver tank, and

. TK-L13, product loadout tank.

In addition, the product routing was changed to bypass the N Cell continuous anion exchange purification unit. Based on operating experience during the 1966 Campaign,(32,34) a decision was made to eliminate the Vycor glass bed column, and the old loadout system. The plutonium PR Room loadout system was used.

URANIUM-233 PRODUCT CONTAINERS

Three-liter, polyethylene bottles (Figure 16) were used for UNH product solution loadout and shipment. The bottles were capped with either a "slit vent" or "duo valve" cap, sealed with an O-ring (see Figures 12 and 13). About 75 percent of the bottles shipped had the slit vent cap as the duo-valve cap was considerably more expensive. The use of the duo valve cap was instituted when leakage occurred during the early shipments. Use of the slit vent cap was resumed, however, when testing revealed other causes responsible for the leakage.

The bottles were pressure tested for leaks by pressurization to ten psig for five minutes. The bottles were then visually inspected for structural flaws. Less than 0.5 percent of the bottles failed the testing. The caps were tested to insure venting at five psig. About three percent of the caps failed to vent properly.

One hundred thirty-one shipping containers were available for bottle shipments. Of the containers, 59 were the Bettis Atomic Power Laboratory (BAPL) type (Figure 14), 57 were Oak Ridge National Laboratory (ORNL) type (Figure 15), and 15 belonged to ARHCO. The ARHCO containers were identical to the BAPL type. In addition, a number of storage containers (Figure 16) were available for temporary on-site bottle containment.

The shipping containers were 55-gallon drums containing stainless steel pressure chambers of 4.81 inches internal diameter. The annular space the BAPL and ARHCO containers was filled with vermiculite, and that of the ORNL containers was filled with foamed glass. The drum acted as a spacer for criticality prevention and the filler as a heat barrier. Before using, each empty container was inspected for mechanical integrity and lack of smearable contamination. The storage containers were 30-gallon steel drums, painted black and had a 4.87-inch diameter, stainless steel, cylindrical inner pressure chamber. The annular space between the chamber and drum was filled with concrete.

SOLVENT TREATMENT CONTACTORS

Carbonate-Permanganate Washers (TK-G1 and TK-R1)

The sodium carbonate-potassium permanganate washers (Figure 17) are similar to the standard 5,000-gallon, ten foot OD x nine foot three inches high, tanks used at Purex. However, each contains a central cylindrical chimney four foot seven and three-quarters inches ID, surrounded by an annular four foot deep section packed with one-inch stainless steel Raschig rings which is supported two feet nine inches above the tank bottom.

The solvent extraction cycle organic waste stream (1CW or 2EW) enters the tank via a distributor located above the packing. A high capacity pump recirculates the aqueous wash stream (200 gpm) from the central chimney, which has a perforated lower section, through a separate distributor over the packing. The two streams are contacted while flowing concurrently through the packing into the disengaging section below. After disengaging, the organic is pumped from the central chimney to the acid washer (10 or 20 Column) which is a standard Purex pulse column.

Turbomixer (T-G7)

The solvent from the 10 Column was continuously mixed with a dilute sodium carbonate solution in a 15,000 gallon tank. The special pump (turbomixer-Figure 18) consists of a paddle rotating within a diffuser attached to a draft tube. The rotating paddle acts as the impeller of a double suction pump.

Organic from above and aqueous from below are drawn into the turbomixer, intimately mixed, and discharged through the diffuser. The diffuser directs the aqueous-organic mixture into a radial flow pattern which prevents the tank contents from swirling. Orifices located above and below the diffuser minimize any change in the organic-to-aqueous mix ratio due to a change in the volume of aqueous wash solution.(66)

APPENDIX B

^{233}U PRODUCT CONCENTRATION AND LOADOUT EQUIPMENT

A. TK-N1 - 3BU Receiver/Concentrator Feed Tank

8 feet long, 2.625 inches wide, 5 feet high, 64 gallons maximum volume, (20 gallons operating volume).

B. E-N6 - Product Concentrator and Cooler

1. Thermal Syphon Recirculated Evaporator

Fabricated of 4-3/4" ID tantalum-lined stainless steel pipe, 8 feet high.

Tube Bundle

12-3/4" inch tantalum tubes, 6 feet long
11.2 square feet heat transfer surface
29 psig steam to shell side

Volume: 8.5 gallons

2. Cooler

Vertical, up-flow, 2.4 inches ID, 5 feet high, cooling water shell side.

C. TK-N7 - Product Receiver Tank

Two-barrel titanium tank, fabricated of 4-3/4" OD pipe, 6 feet high.

Barrels connected with 2-inch pipe crossovers at top and bottom.

Volume: 10 gallons

D. T-N50 - Cation Column

5 inch, Sch. 40, 304L stainless steel pipe
7 feet, 3 inch in height
6 foot resin bed
Dowex-50 W-X8 resin (acid form)
50 to 100 mesh
Volume: 8.5 gallons

E. TK-L9 - Product Sampling Tank

3 barrel, 304L stainless steel; fabricated of 5-inch schedule 40 pipe.
3 feet, 4 inches high
Barrels connected with top 2½ inch schedule 10 pipe, middle two 1" schedule 10 pipes, bottom, 3 inch schedule 10 pipe volume - 11 gallons nominal.

APPENDIX B (continued)

F. TK-L13 - Product Loadout Tank

3 inch diameter - glass
30 inches high
Volume: 0.8 gallons.

APPENDIX C

PRE-THORIA CAMPAIGN - CANYON EQUIPMENT CHANGES

A CELL

1. Remove A-G13-A3, H-2-60014 (save)
2. Install A-G13-A3, H-2-61853
3. Remove A-G2-AH, H-2-60002 (save)
4. Remove A-T2-A3, H-2-60003 (save)
5. Install A-T2-A3, H-2-60003 (modified, 8" short)
6. Install A-G2-AH, H-2-60002

B CELL

1. Remove B-G13-B3, H-2-60014 (save)
2. Install B-G13-B3, H-2-61853
3. Remove B-G2-AH, H-2-60002 (save)
4. Remove B-T2-B3, H-2-60003 (save)
5. Install B-T2-B3, H-2-60003 (modified, 8" short)
6. Install B-G2-AH, H-2-60002

C CELL

1. Remove C-G13, H-2-60014 (save)
2. Install C-G13-C3, H-2-61853
3. Remove C-G2-AH, H-2-60002 (save)
4. Remove C-T2-C3, H-2-60003 (save)
5. Install C-T2-C3, H-2-60003 (modified, 8" short)
6. Install C-G2-AH, H-2-60002
7. Remove C-G1-AG, H-2-60000 (save)
8. Remove C-T1-C3, H-2-60001 (save)
9. Remove C-C3-Y-AK, H-2-59116 (save)
10. Remove C-T8-C3, H-2-60035 (save)
11. Install C-T1-T8-L3, H-2-61854
12. Install C-G1-AG, H-2-6000
13. Blank nozzle Z on C3

D CELL

1. Remove D-T7-T8, H-2-57199 (save)
2. Install D-T7-D4, H-2-64714
3. Install D-T8-D5, H-2-64741
4. Install a Group III pump in TK-D4, West Manhole
5. Install D-G19-P-D4, H-2-53701
6. Install D-T10-D4, H-2-64739
7. Install D-G16-T10, H-2-64740
8. Remove D-T18-D4, H-2-53805 (save)
9. Install D-T18-D3, H-2-59189
10. Install D-T38-D2, H-2-53550
11. Remove D-T27-D2, H-2-63475 (save)
12. Remove D-T30-D2, H-2-63474 (save)
13. Install D-T-27-T30, H-2-64748
14. Install 2" blank on D-T41
15. Install 2" blank on D-T24

E CELL

1. Remove E-(TE3-1)F-E5, H-2-60078 (save)
2. Install E-T18-E5, H-2-53550
3. Remove E-(E3-1)-E3, H-2-60093 (save)
4. Install E-T38-E3, H-2-59923

F CELL

1. Remove F-G244, H-2-53602 (save)
2. Install standard drop leg on F-T161
3. Install sectionalized concentrator, 1-TB-F6, reboiler section in the F11 position
4. Install stainless tube bundle No. 65 H-2-58755 in the F-11 east position (1-1/4" tubes, remote gasket)

F CELL (continued)

5. Install a cooling coil, H-2-64705, in F11 west position (remote gasket)
6. Install submerged jet, H-2-64706, in the cooling coil flange (remote gasket)
7. Install F-G100-F11-1, H-2-64728
8. Install F-G99-A-F11-1, H-2-64731
9. Install F-(F11-1)-F12, H-2-64727
10. Install F-G103-F11-1, H-2-64726
11. Install 2" blank on F11-1 nozzle GA
12. Install 2" blank on F11-1 nozzle GG
13. Install F-(F11-1)F12, H-2-53999
14. Install F-G95-F11-1, H-2-64735
15. Install F-G97-8, H-2-54001
16. Install 2" instrument blank on F11-1 nozzle NN
17. Install F-F11-1-F12, H-2-53999
18. Install 3" blank on F11-1 nozzle FF
19. Install 3" blank on F11-1 nozzle HH
20. Install 3" blank on F11-1 nozzle A
21. Install F-G89-F11-1, H-2-64743
22. Install F-G98-A-F11-1, H-2-53950
23. Install F-T97-T92-A, H-2-64708
24. Install F-92B-F11-1(B), H-2-64729
25. Install F-T86-F11-1, H-2-54017
26. Install F-(F11-1)J-M, H-2-58239
27. Install F-G110A-F11-1, H-2-58242
28. Install F-G113-F11-1, H-2-64711
29. Install F-G114-F11-1, H-2-64711
30. Install F-G115-F11-1, H-2-64711
31. Install F-G117-F11-1, H-2-64711
32. Install acid tower T-F11-1(2-TT-F6), H-2-58104, with "Tophet M" demister pad
33. Install F-G109-7, H-2-59652
34. Install condenser, 0-E-F11-2, H-2-52551
35. Remove F-T107-F10, H-2-53560 (save)
36. Install 4" Blank on F10 nozzle L

F CELL (continued)

37. Install F-T107-(F11-2)-C, H-2-64709
38. Install F-G111-5, H-2-64718
39. Install F-4(F11-1)K, H-2-64719
40. Install F-G112-F11-1, H-2-58236
41. Install F-(F11-1)-JJ-LL, H-2-58098
42. Install F-G101-RR, H-2-58246
43. Install F-(F11-1)TT-UU, H-2-58235
44. Install F-(F11-1)S-WW, H-2-58240
45. Install F-3-2, H-2-64717
46. Install F-1-(F10)-A, H-2-64710
47. Install F-T93-F11-2, H-2-53688
48. Install F-T92-F11-2, H-2-53667
49. Install F-T91-F11-2, H-2-53667
50. Install F-T90-F11-2, H-2-53667
51. Install F-G105-F11-2, H-2-53921
52. Install F-G106-F11-2, H-2-53921
53. Install F-G107-F11-2, H-2-53921
54. Install F-G108-F11-2, H-2-54044
55. Install F-T121-T122, H-2-57199
56. Install F-T120-T127, H-2-64737
57. Install F-T138-T141, H-2-64736
58. Install F-TK16(M)-10, H-2-58671

G CELL

1. Determine that G-T110 is blanked
2. Blank G-T1

H CELL

1. Remove H-G15-H4-2, H-2-54044 (save)
2. Remove H-G14-H4-2, H-2-53921 (save)
3. Remove H-G13-H4-2, H-2-53921 (save)
4. Remove H-G12-H4-2, H-2-53921 (save)
5. Remove H-T13-H4-2, H-2-53688 (save)
6. Remove H-T11-H4-2, H-2-53677 (save)
7. Remove H-T10-H4-2, H-2-53677 (save)

H CELL (continued)

8. Remove H-T9-H4-2, H-2-53677 (save)
9. Remove H-G27-T15, H-2-53875 (save)
10. Remove H-G26-T15, H-2-53853 (save)
11. Remove H-T14-T15, H-2-53710 (save)
12. Remove H-T15-H4-2, H-2-53561 (save)
13. Remove H-G20-H4-1, H-2-53686 (scrap)
14. Remove condenser H4-2(0-E-F1), H-2-52480 (store on deck)
15. Remove H-(G8)-H4-FF, H-2-61886 (scrap)
16. Remove H-G8-DOV, H-2-61859 (scrap)
17. Remove H-G1-H4-1, H-2-57791 (save)
18. Remove H-G4-H4-1, H-2-53950 (scrap)
19. Remove H-T1-H4-1, H-2-59653 (save)
20. Remove H-T8-D5, H-2-59606 (save)
21. Remove H-T2-H4-1, H-2-54017 (save)
22. Remove J-G184-H4-J1, H-2-54011 (save)
23. Remove H-G2-H4-1, H-2-54001 (save)
24. Remove H-H4-1-J1, H-2-56519 (save)
25. Remove H-H4-1-J1, H-2-53999 (save)
26. Remove J-J1-H4-1, H-2-54011 (save)
27. Remove H-T4-T6, H-2-57199 (save)
28. Remove H-T7-H4-1, H-2-57209 (save)
29. Remove H-G3-H4-1, H-2-53698 (save)
30. Remove H-G5-H4-1, H-2-53698 (save)
31. Remove H-G7-H4-1, H-2-53698 (save)
32. Remove H-G9-H4-1, H-2-53698 (save)
33. Remove H-G16-(H4)JM, H-2-59658 (save)
34. Remove H-H4-J-M, H-2-59654 (save)
35. Remove H-G21-H4-1, H-2-53708 (save)
36. Remove H-G22-H4-1, H-2-53708 (save)
37. Remove H-G23-H4-1, H-2-53708 (save)
38. Remove H-G25-H4-1, H-2-53708 (save)
39. Remove reboiler tube bundles to temporary storage on the deck
40. Remove H4 concentrator (4-E-F11) to the G3-G4 storage position

H CELL (continued)

41. Move the reboiler tube bundles from the deck to normal positions in the concentrator. Do not make up nuts.
42. Install sectionalized concentrator reboiler section, (5-TB-F6), H-2-66104, in the H4 position
43. Install tube bundles, (67 + 68), H-2-58755, in the reboiler canisters (1-1/4" tubes, remote gaskets)
44. Install H-T2-H4-1, H-2-54017
45. Install H-G3-H4-1, H-2-53698
46. Install H-G5-H4-1, H-2-53698
47. Install H-G7-H4-1, H-2-53698
48. Install H-G9-H4-1, H-2-53698
49. Install H-G21-H4-1, H-2-53708
50. Install H-G22-H4-1, H-2-53708
51. Install H-G23-H4-1, H-2-53708
52. Install H-G25-H4-1, H-2-58233 (new jumper)
53. Install a 2" blank on H4 nozzle GA
54. Install H-H4-J1 (as revised), H-2-53999
55. Install H-H4-J1, H-2-56519
56. Install H-G2-H4-J1, H-2-54001
57. Install H-J1-H4, H-2-54011
58. Install a 2" blank on H4 nozzle CC
59. Install a 2" blank on H4 nozzle EE
60. Install a 3" blank on H4 nozzle HH
61. Install H-T1-H4, H-2-59653
62. Install H-G1-H4, H-2-57791 Assembly 2 (new jumper)
63. Install H-G4-H4, H-2-58230 (new jumper)
64. Install H-H4-J-M, H-2-58239 (new jumper)
65. Install H-G18-H4, H-2-58242 (new jumper)
66. Install H-G16-(M-J), H-2-57724

H CELL (continued)

67. Install concentrator tower T-H4, (1-TT-H4) H-2-58102, with demister pad
68. Install H-H4-JJ, H-2-58248 (new jumper)
69. Install H-G8-H4, H-2-58244 (new jumper)
70. Install H-H4-(T1-G8), H-2-58245 (new jumper)
71 Install H-H4-W-KK, H-2-58237 (new jumper)
72. Install H-H4-5-Y, H-2-58238 (new jumper)
73. Install a 2" blank on H4 nozzle VV
74. Install H-T7-H4, H-2-58229 (new jumper)
75. Install a 2" blank on H4 nozzle U
76. Install H-G17-H4, H-2-58231 (new jumper)
77. Install H-G20-H4, H-2-58236 (new jumper)
78. Install condenser H4-2, (0-E-F1), H-2-52480
79. Install H-T15-H4-2, H-2-53561
80. Install H-T14-(T15), H-2-53710
81. Install H-H4-PP-RR, H-2-58234 (new jumper)
82. Install H-G26-(T15), H-2-53853
83. Install H-G27-(T15), H-2-53875
84. Install H-T13-H4-2, H-2-53688
85. Install H-T4-T5, H-2-57245
86. Install H-T9-H4-2, H-2-53667
87. Install H-T10-H4-2, H-2-53667
88. Install H-T11-H4-2, H-2-53667
89. Install a 4" blank on H-T12
90. Install H-G12-H4-2, H-2-53921
91. Install H-G13-H4-2, H-2-53921
92. Install H-G14-H4-2, H-2-53921
93. Install H-G15-H4-2, H-2-54044
94. Install a 3" blank on H-T6
95. Remove H-T40-H2(U), H-2-62848, (save)
96. Remove H-G61-H2 (CC), H-2-57729 (save)
97. Install H-T44-H2(U), H-2-61863
98. Install H-G63-H2(CC), H-2-58621
99. Remove H-G34-H3, H-2-57964 (save)
100. Remove H3 cartridge, RC-4 and store in pool cell.
101. Complete item 8 of the J Cell changes
102. Install H-G34-H3, H-2-57964
103. Install H-G37-H3, H-2-61878
104. Remove H-T17-H3, H-2-57698 (save)
105. Install H-T21-H3, H-2-61879
106. Install H-T20-H3, H-2-61880
107. Remove H-G30-H3, H-2-63417 (save)
108. Remove H-T16-H3, H-2-63416 (save)
109. Install H-(H3)F-9, H-2-61883
110. Install H-9-(H4)V, H-2-61881
111. Install H-G28-2, H-2-57207
112. Install H-G30-3, H-2-57206
113. Blank H-G61

J CELL

1. Remove J-G31-P-J8, H-2-63465 (save)
2. Remove J-(G33)-J8-10, H-2-63467 (save)
3. Remove J-SPLP-J8-E-J7, H-2-63464 (save)
4. Remove J-G37-J7, H-2-63463 (save)
5. Install a 2" blank on J8 sample pot, nozzle E
6. Remove J-G34-J7, H-2-57686 (save)
7. Remove probe end of J-G43-J7, H-2-58433
8. Remove J7 cartridge, RC-10, H-2-58900 and install in H3 column
9. Move cartridge RC-1, H-2-57684, from pool cell and install in J7 column
10. Install J-G34-J7, H-2-57686
11. Remove J-T14, J7, H-2-53774 (save)
12. Remove J-T15-J7, H-2-54022 (save)
13. Install a 3" blank on nozzle J-T15
14. Install J-T14-J7, H-2-64746 (new jumper)
15. Remove J-G52-(T22), H-2-53904 (save)

J CELL (continued)

16. Remove J-G53-(T23), H-2-53899 (save)
17. Remove J-G55-(T23), H-2-57463 (save)
18. Remove J-G57-J6, H-2-53903 (save)
19. Remove J-T23-J6, H-2-57243 (save)
20. Remove J-T22-J6, H-2-53902 (save)
21. Install a 2" blank on J6 nozzle A
22. Remove J-T20-J7, H-2-53912 (save)
23. Remove 2" blank from nozzle J-T21
24. Install 2" blank on nozzle J-T20
25. Remove J-T31-PG-J7, H-2-53912 (save)
26. Install J-T31-PG-J7, H-2-61876
27. Install J-PG-J6-12, H-2-61857
28. Remove J-T25-J6, H-2-53914 (save)
29. Remove J-G40-J7, H-2-53836 (save)
30. Install 2" blank on J7 sample pot nozzle D
31. Install J-T23-J7, H-2-61868
32. Install J-T23-J7, H-2-61868
33. Install J-T16-J6, H-2-61865
34. Install J-T21-2, H-2-61856
35. Install J-T23-7-8, H-2-61884
36. Install J-G40-J6, H-2-61852
37. Install J-J6(F)-J7(G), H-2-64744 (new jumper)
38. Install J-G53-(J6-J7), H-2-64745 (new jumper)
39. Install J-10-11, H-2-64747 (new jumper)
40. Remove J-G112-T50, H-2-53910 (save)
41. Remove J-G114-T50, H-2-57463 (save)
42. Remove J-T50-J4, H-2-53909 (save)
43. Install J-T45-T40, H-2-61873
44. Install J-G112-T45-T50, H-2-61871
45. Install J-G114-T45-T50, H-2-61872
46. Remove J-13-14, H-2-62679 (save)
47. Remove J-G89-12, H-2-62631 (save)
48. Remove J-G91-CC-T46, H-2-62788 (save)

J CELL (continued)

49. Remove J-T46-J4, H-2-62651 (save)
50. Remove J-T52-PG-J4, H-2-53912 (save)
51. Remove J-T56-T62, H-2-59425 (save)
52. Install J-T52-T62, H-2-58601
53. Install a 3" blank on nozzle J-T56
54. Install J-T46-AN, H-2-61870
55. Install a 2" blank on J4 nozzle 14
56. Remove J-T48-J4, H-2-58938 (save)
57. Install a 2" blank on J4 nozzle D
58. Install a 2" blank on J4 nozzle B

K CELL

1. Remove hairpin, H-2-57199 between (K-G2) and (K-T6)
2. Remove blank from jet nozzle on H-2-63484
3. Install blank on outlet nozzle on H-2-63485
4. Install hairpin, H-2-57199 between jet nozzle on H-2-63484 and nozzle going to K-T6

APPENDIX D

POST THORIA CAMPAIGN
CANYON PIPING AND EQUIPMENT CHANGES

A Cell

1. Remove AG-13-H-2-61853 and Junk.
2. Install AG-13-H-2-60014.
3. Remove AT-2-H-2-60003 and save - It is 8" short - AG-2 - steam to AT2 is a flex.
4. Install AT2-H-2-60003 - was on F1 Block.
5. Install Flex AG2 to Jet AT2.

B Cell

1. Remove B-G-13-H-2-61853 and Junk.
2. Install B-G-13-H-2-60014 - its in the cell.
3. Remove B-G-2-H-2-60002 - save.
4. Remove B-T2-H-2-60003 - save.
5. Install B-T2-H-2-60003.
6. Reinstall B-G-2-H-2-60002.

C Cell

1. Remove C-G13-H-2-61853 - Junk.
2. Install C-G13-H-2-60014.
3. Remove GG-2-H-2-60002 - Save.
4. Remove CT-2-H-2-60003 - Save.
5. Remove C-G1-H-2-60000 - Save.
6. Remove CT-1-CT8-H-2-61854 - Junk.
7. Install CT-1-H-2-60001.
8. Install CT-2-H-2-60003.
9. Reinstall CG-1-H-2-60000.
10. Reinstall CG-2-H-2-60002.
11. Install C8-H-2-60035.
12. Install AK-Y-H-2-59116 - Piggy-back steam.
13. Remove corrosion probe from R Nozzle and Blank - Save.

D Cell

1. Install Jet DT51-H-2-53683 - I think it is on FT73.
2. Install DG72-H-2-53685 - in the cell.
3. Remove hairpin DT31-DT33 - Save.
4. Install Jet DT33.
5. Install drop DT31.
6. Move lower end of DG51 to DT33 jet.
7. Remove hairpin DT27-DT30 - save.
8. Blank DT27.
9. Remove DG-16-H-2-64740 - Junk.
10. Remove DT10-H-2-64739 - Junk.
11. Remove DT7-H-2-64714 - Junk.

D Cell (continued)

12. Remove DT8. Reinstall anything else that is off here to make it look like the book.
13. Install HP-DT7-DT8.

E Cell

1. Remove EG101 - Save.
2. Remove ET55 - Save.
3. Install Hair Pin ET54 - ET55.

F Cell

1. Remove F-3-2-H-2-64717 - Junk.
2. Remove Electric Head F4 to K nozzle, to clear FG111 - Junk.
3. Remove FG-111-H-2-64718 - Junk.
4. Remove FT107 - F11-C-H-2-64709 - Junk.
5. Remove FT93 - H-2-53688 - Save.
6. Blank FT93.
7. Remove Flex Steam from FT36 and install on T-22B.

G Cell

Leave as it is.

H Cell

1. Remove HT44-H-2-61863 - Junk.
2. Install HT40-H-2-62848.
3. Remove H-G-63 - H-2-58621 - Junk.
4. Install HG-61-H-2-57729.
5. Remove HG-37-H-2-61878 - Junk.
6. Remove HG-28-H-2-57207 - Junk.
7. Remove HG-30-H-2-57206 - Junk.
8. Remove H-T21-H-2-61879 - Junk.
9. Remove H-T20 - H-2-61880 - Junk.
10. Remove H-9-H4V - H-2-61881 - Junk.
11. Remove H-H3-F9 - H-2-61883 - Junk.
12. Blank V Nozzle-H4 - 2"V.
13. Remove H-G34-H-2-57964 - Save.
14. Loosen H3 Cartridge Anchor nuts for move to J7.
15. Remove hairpin HT4-HT5-H-2-57245 - Save.
16. Install hairpin HT4 to HT6 - H-2-57199.
17. Install HT8 - H-2-59604.
18. Remove HT19 - H-2-53774 and blank T Wall Nozzle.

J Cell (J1-2-3-4 and 5)

1. Remove hairpin JT-52-JT-62 - H-2-58601 - Junk.
2. Remove lower end of JG114 flex - Save.
3. Remove JG-112 - H-2-61871 - Junk.

J Cell (J1-2-3-4 and 5) (continued)

4. Remove JT46 - H-2-61870 - Junk.
5. Remove H-P-JT-45-JT50 - H-2-61873 - Junk.
6. Remove flex JG-89 to J3 Tank - Save.
7. Remove JG97 - H-2-66155 - Junk.
8. Blank AN nozzle on J2 - 2" Vertical.
9. Install flex JT60 to A Nozzle - J3.
10. Install H.P. JT56 to JT62 - H-2-59425.
11. Install JT52 - pulse leakage, H-2-57457, 53912, either one.
12. Install JT50 - H-2-53909.
13. Install JT48 - H-2-58938.
14. Install J-13-14 - J4.
15. Install JT46-H-2-62651.
16. Install JG97 - H-2-60166.
17. Install Neutron Probe, H-2-62788 to CC at JG91.
18. Install JT40 - H-2-60154.
19. Install JG-85 - H-2-60158.

J Cell (J6-7 and 8)

1. Remove JG65 - H-2-53927 - Save.
2. Remove JG66 - H-2-54285 - Save.
3. Remove JG68 - H-2-54283 - Save.
4. Remove JG73-H-2-53896 - Save.
5. Remove JG75-H-2-53862 - Save.
6. Remove JT29-H-2-63060 - Junk.
7. Remove JT30-H-2-54281 - Save.
8. Remove Pulse Leakage - H-2-61857 - Junk.
9. Remove J6 pulse generator and regasket pulse leg.
10. Reinstall J6 pulse generator.
11. Remove 1BXF Vent Line - H-2-66174 - Junk.
12. Remove JG53 - H-2-64745 - Junk.
13. Remove J6-J7-1BXF - H-2-66173 - Junk.
14. Remove JG40 - H-2-61852 - Junk.
15. Remove JT16-H-2-61865 - Junk.
16. Remove JT21 - H-2-61856 - Junk.
17. Remove JT23 - H-2-61868 - Junk.
18. Remove H-P-JT24-JT25 - H-2-61869 - Junk.
19. Remove JG-34-H-2-57686 - Save.
20. Remove Tower End of JG43 - Save.
21. Loosen tower cartridge for removal.
22. Move cartridge RC-1 from J7 to H-3 Cell.
23. Move cartridge RC10 from H3 to J7.
24. Install JT31 - H-2-53912.
25. Install JT30 - H-2-54218.
26. Install JT29 - New from Shop - H-2-63060.
27. Install JT25 - H-2-53914.
28. Install JT23 - H-2-57243.
29. Install JT20 - H-2-53912.
30. Install JT15 - H-2-54022.
31. Install JG37 - H-2-63463.

J Cell (J6-7 and 8) (continued)

32. Install jumper H-2-63464 from JG37 to E Nozzle at JT10.
33. Install JG31 - flex to H-2-63464.
34. Install JG40 - H-2-53836.
35. Check JG-42-43 and 44.
36. Install JG53 - H-2-53899.
37. Install JG55 flex to HT-23.
38. Install JG-60 - H-2-64537.
39. Install JG61 - H-2-53850.
40. Install JG65 - H-2-53927.
41. Install JG66 - H-2-54285.
42. Install JG68 - H-2-54283.
43. Install JG73 - H-2-53896 - or flex.
44. Install JG75 - H-2-53862.

K Cell

1. Move hairpin on KT6 set up to the two west nozzles.
2. Blank the east nozzle.
3. Install a 2" H x 3" V flex on KG18 to H Nozzle at K6.

APPENDIX E

THORIUM PROCESSING STANDARD OPERATING PROCEDURE (SOP) INDEX

General Outline

101 thru 124	Pre-Thorium Run Flushing Operation (Pink Paper)
125 thru 150	Thorium Run Operation (Green Paper)
151 thru 174	Post Thorium Run Flushing Operation (Yellow Paper)

INDEX

(Procedures 101.00 → 124.99 on Pink Paper)

101.00	General - Pre-Thorium Flushing
102.00	Special Procedures - Pre-Thorium Flushing
102.01	UO_3 Acid Routing in Preparations for Thorium Run
102.02	Flush Handling in F Cell - Pre-Thorium Flushing
103.00	Dissolver Flushing - Pre-Thorium Flushing
103.01	Product Flush of Headend - Pre-Thorium
103.02	HNO_3 - KF Flush of Dissolvers - No. 1
103.03	Bellows and Cone Flush
104.00	D Cell Flushing - Pre-Thorium Flushing
104.01	HNO_3 - KF Flushes, D Cell
105.00	E Cell Flushing - Pre-Thorium Flushing
105.01	Flush Procedures for E Cell Vessels
105.02	D5-E6-H1 Flush
106.00	Solvent Extraction Flushing - Pre-Thorium Flushing
106.01	HA Column Flush
106.02	J Cell Package Flush
106.03	H4-J4 Nitric Product Flush
106.04	J5, L1, L2 Flush
106.05	J2, J3, J6, J4, J5 Product Flush
106.06	J2, J3, J6 Chemical Flush
106.07	J7, J8, and K Cell Product Flushes
106.08	J7, J8, K1 Chemical Flush
106.09	Deep Flush of L Cell Package
106.10	G Cell
106.11	J1, F12 Metathesis and Flush
106.12	Chemical Flush K4, K5, K6
106.13	J1, J21 Chemical Flush
106.14	J1, J21 Caustic Tartaric Flush
106.15	J1, J21 KF Flush
107.00	N Cell Flushes - Pre-Thorium
108.00	PR Room Flushes - Pre-Thorium
109.00	Outside Area Flushing - Pre-Thorium Flushing
109.01	203A Area Piping Flush - Pre-Thorium

INDEX

(Procedures 125.00 → 150.00 on Green Paper)

131.03	Special Procedure - Synthetic 1BXF to J3 - Thorium Operation
131.04	Pre-startup - Thorium Operation
131.05	Startup from Extended Shutdown - Thorium Operation
131.06	Startup from Equilibrium Shutdown - Thorium Operation
131.07	Equilibrium Shutdown - Thorium Operation
131.08	Extended Shutdown - Thorium Operation
131.09	Air or Water to HA (H2) Pulsers
132.00	Partition Cycle - Thorium Operation
132.01	General Description - Thorium Operation
132.02	Emergency Shutdown - Thorium Operation
132.03	Special Procedure - Filling 1BU Line - Thorium Operation
132.04	Pre-startup - Thorium Operation
132.05	Startup from Extended Shutdown - Thorium Operation
132.06	Startup from Equilibrium Shutdown - Thorium Operation
132.07	Equilibrium Shutdown - Thorium Operation
132.08	Extended Shutdown - Thorium Operation
132.09	Shutdown for Flush - Thorium Operation
132.10	Air On and Off the J6, J7 and H3 Column Pulsers
132.11	H4 Concentrator Startup and Shutdown
132.12	J8 Concentrator Startup and Shutdown
132.13	Conc. HNO_3 Flush of Partition Cycle
133.00	Final Thorium Cycle - Thorium Operation
133.01	General Description - Thorium Operation
133.02	Emergency Shutdown - Thorium Operation
133.03	Pre-startup - Thorium Operation
133.04	Startup - Thorium Operation
133.05	Equilibrium Shutdown - Thorium Operation
133.06	Stripping Shutdown - Thorium Operation
133.07	Air or Water on 2D (K2), 2E (K3) Pulsers
133.08	K4 Concentrator Startup and Shutdown
134.00	Second Uranium Cycle - Thorium Operation
134.01	General Description - Thorium Operation
134.02	Emergency Shutdown - Thorium Operation
134.03	Pre-startup - Thorium Operation
134.04	Startup - Thorium Operation
134.05	Equilibrium Shutdown - Thorium Operation
134.06	Stripping Columns - Thorium Operation
134.07	Displacing Columns - Thorium Operation
134.08	Air or Water On or Off, 2A/2N(J22), 2B/2P(J23) Pulsers
134.12	Conc. HNO_3 Flush of J Cell Package
135.00	Third Uranium Cycle - Thorium Operation
135.01	General Description - Thorium Operation
135.02	Emergency Shutdown - Thorium Operation
135.03	Pre-Startup - Thorium Operation
135.04	Startup - Thorium Operation
135.05	Equilibrium Shutdown - Thorium Operation
135.06	Stripping Columns - Thorium Operation

135.07	Displacing Columns - Thorium Operation
135.08	Air or Water On or Off - 3A/2A(L1), 3B/2B(L2) Pulsers
136.00	G Cell Solvent System - Thorium Operation
136.01	General Description - Thorium Operation
136.02	Emergency Shutdown - Thorium Operation
136.03	Pre-Startup - Thorium Operation
136.04	Startup - Thorium Operation
136.05	Fresh Carbonate Change - Thorium Operation
136.06	Acid Change TK-G2 - Thorium Operation
136.07	TK-G7 Solution Change - Thorium Operation
136.08	Purging Column Interface - Thorium Operation
136.09	TK-G8 - Disposal - Thorium Operation
136.10	Shutdown - Thorium Operation
136.11	Air or Water on G2 Pulser
136.12	G Cell Flush - Thorium
136.13	G1 Acid Flush
137.00	R Cell Solvent System - Thorium Operation
137.01	General Description - Thorium Operation
137.02	Emergency Shutdown - Thorium Operation
137.03	Pre-Startup - Thorium Operation
137.04	Startup - Thorium Operation
137.05	Fresh Carbonate Change - Thorium Operation
137.06	Acid Change TK-R2 - Thorium Operation
137.07	Purging Column Interface - Thorium Operation
137.08	TK-R8 Disposal - Thorium Operation
137.09	Shutdown - Thorium Operation
137.10	R8 Flush
138.00	Acid Recovery and Waste Treatment - Thorium Operation
138.01	General Description - Thorium Operation
138.02	Startup-Operating-Shutdown-Waste Concentration and Acid Recovery-Thorium Operation (F7, F6, F26, F5)
138.03	Sugar Denitration - Thorium Operation
138.04	Digestion and Waste Neutralization - Thorium Operation
138.05	Vacuum Fractionator Operation - Thorium Operation
138.06	Organic Disposal - F7 - Thorium Operation
138.07	Anti-Foam Makeup and Addition
138.08	Emergency F Cell Shutdown
138.09	Disposal of Conc. Nitric Flush
138.10	Fresh Acid Makeup in U1 and U2
138.11	U3 & U4 Waste Disposal
138.12	Sump Waste Disposal - Thorium Operation
139.00	U-233 Ion Exchange and Concentration - Thorium Operation
139.01	General Description - Thorium Operation
139.02	U-233 Ion Exchange and Concentration - Pre-Startup
139.03	U-233 Ion Exchange and Concentration - Startup and Normal Operation
139.04	U-233 Ion Exchange and Concentration - TK-N7 to TK-L9 Batch Transfer
139.05	U-233 Ion Exchange and Concentration - Shutdown, Short and Long Duration

139.06	U-233 Ion Exchange and Concentration - Emergency Shutdown
139.07	U-233 Ion Exchange and Concentration - TK-N1 Recycle to TK-L11
139.09	U-233 Ion Exchange and Concentration - Cation Column Elution
139.10	U-233 Ion Exchange and Concentration - Resin Pretreatment
139.11	U-233 Ion Exchange and Concentration - Cation Column - Resin Changeout
139.12	TK-N1 - Organic Detection and Handling
140.00	Product Handling - Thorium Operation
140.01	General Description in U-233 Product Handling
140.02	Thorium Product Sampling and Loadout
140.03	Thorium Product Return Route (Rework)
140.04	U-233 Sampling and Loadout
140.05	U-233 Rework from TK-L11 to TK-E6
140.06	U-233 On-Site Transport and Storage
140.07	Preparation of U-233 Containers for Shipment Off-Site
140.08	U-233 Transloading
140.09	Weighing - Dynamic Balance
140.10	Weighing - Hadley Balance
140.11	PR Emergency
140.12	U-233 - L3 Shipping Bottle - Inspection and Testing
140.13	U-233 Shipping Container Inspection
140.14	Operability Test of Pressure - Test Stand
140.15	Container and Pressure Testing
140.16	Unloading Thorium Nitrate
140.17	U-233 Cleanup and Waste Disposal
140.18	Bottle Transfer Between Containers
140.19	Hydrostatic Test of Bettis Pressure Vessels
140.20	Venting Pressure Vessel of Loaded U-233 Containers
140.21	TK-L11 Sampling
141.00	Special Shutdown Procedures
141.01	Head End September, 1970
141.02	Solvent Extraction Shutdown
141.03	Solvent Extraction Shutdown, November 6, 1970
141.04	Solvent Extraction Shutdown, November/December
141.05	Solvent Extraction Shutdown, December 12, 1970
142.00	Special Startup Procedures
142.01	Startup September 1970
142.02	Head End DNR Startup
142.03	Solvent Extraction Startup, October 1970
142.04	Solvent Extraction Startup, November 11, 1970
142.05	Solvent Extraction Startup, December 1970
143.00	Special Flushes
143.01	Conc. HNO_3 Flush of Solvent Extraction
143.02	Conc. HNO_3 Flush of Solvent Extraction, November
143.03	F13 Organic Handling
143.04	Conc. HNO_3 Flush of Solvent Extraction

POST THORIUM SOP INDEX

(Procedures 151.01 → 174.00 on Yellow Paper)

- 150.00 General - Post Thorium Flushing
  - 150.01 General Description - Post Thorium Flushing
- 151.00 Special Procedures - Post Thorium
  - 151.01 Waste Handling F Cell
  - 151.02 Waste Handling E Cell
- 152.00 Dissolver Flushes - Post Thorium Flushing
  - 152.01 Low Volume - HNO_3 - KF Flush of Dissolvers
  - 152.02 High Volume Dissolver Flushes
  - 152.03 Nitric Flush of Headend
- 153.00 D Cell Flushes - Post Thorium
  - 153.01 D Cell Flush
- 154.00 E Cell Flushes - Post Thorium
  - 154.01 E Cell Flush
- 155.00 Solvent Extraction Flushes - Post Thorium
  - 155.01 Strong Nitric Flush - H1 and H2
  - 155.02 Strong Nitric Flush - Solvent Extraction System
  - 155.03 Chemical Flush H3, H4, J1, H1, H2
  - 155.04 Chemical Flush J3, J6, J7, H3
  - 155.05 Chemical Flush J7, J8, K1, K2
  - 155.06 Chemical Flush K3, K4, K5, K6
  - 155.07 Chemical Flush J Cell Package
  - 155.08 Chemical Flush L Cell Package
  - 155.09 Chemical Flush G Cell
  - 155.10 Chemical Flush R Cell
  - 155.11 F13 Organic Wash
- 156.00 N Cell Flushes - Post Thorium
  - 156.01 Cation Column Elution
  - 156.02 N1, N50, N6, N7, N9 Product Flush
  - 156.03 U-233 Hood, N Cell Vent Header Flush
  - 156.04 N1, N50, N6, N7, L9 Final Flush
- 157.00 Miscellaneous Flushes - Post Thorium
  - 157.01 203-A Piping Flush
  - 157.02 TK-206 (KF) Disposal Flush

APPENDIX F

THORIUM PROCESSING SAMPLE SCHEDULE

PUREX PLANT
ROUTINE STREAM SAMPLE SCHEDULE
THORIUM OPERATION

SAMPLE	GRAVEYARD	DAYS	SWING
F3	HNO_3, Gamma Scan, Comp. (1)		
F5	HNO_3, Total β, Gamma Scan		
F10-2WF	AT	AT	AT
G5-100		Tues. & Thurs. → Daily →	% TBP & Pu Ret Gamma Scan
G7-10D	pH	pH	pH
H2-HAW	Th, HNO_3, U^{233}	Th, HNO_3, U^{233}	Th, HNO_3, U^{233}
HAP	Gamma Scan		
*H3-1CW	*AT, Gamma Scan	*AT	AT*
*J1-1UC	Gamma Scan (once/shift), *AT, *HNO_3, (twice/shift) (2)	*AT, *HNO_3 (twice/shift) (2)	Th (once/shift), *AT, *HNO_3, (twice/shift) (2)
*J2-1BSU	AT	AT	AT
*J3-1BXF	*AT, Th	*AT, Th	*AT, Th
*J7-1BU	*AT, Th	*AT, Th	*AT, Th
*J8-1BT	*U^{233}, Th, HNO_3, Visual	*U^{233}, Th, Visual	*U^{233}, Th, Visual
*J21-2AF/2NF	*AT, HNO_3, Comp. (3)	*AT, HNO_3, Comp. (3)	*AT, HNO_3, Gamma Scan, Comp (3)
*J22-2AW/2NW	*AT, HNO_3, (twice/shift) (2)	*AT, HNO_3, (twice/shift) (2)	*AT, HNO_3, (twice/shift) (2)
*J23-2BW/2PW	AT	AT	AT

**These analyses are required for critical mass control and are not to be omitted without supervisor's approval.*

Gamma Scans: ZrNb, Ru^{103}, $RuRh^{106}$, Pa

PUREX PLANT
ROUTINE STREAM SAMPLE SCHEDULE
THORIUM OPERATION

SAMPLE	GRAVEYARD	DAYS	SWING
*J5A-3AF/2AF	*AT, HNO_3, Comp. (3)	*AT, HNO_3, Comp. (3)	*AT, HNO_3, Gamma Scan, Comp. (3)
*L1-3AW/2AW	*AT, HNO_3, (twice/shift) (2)	*AT, HNO_3, (twice/shift) (2)	*AT, HNO_3, (twice/shift) (2)
*L2-3BW/2BW	AT	AT	AT
*L3-3BU/2BP	*AT, HNO_3, Th	*AT, HNO_3, Th	*AT, HNO_3, Th, Gamma Scan
*K1-2DF	*U^{233}, HNO_3, Th, Gamma Scan	*U^{233}, HNO_3, Th	*U^{233}, HNO_3, Th
K2-2DW	HNO_3, Th	HNO_3, Th	HNO_3, Th
K3-2EW	AT, Th	AT, Th	AT, Th
K4-2ET/2EU	U^{233}, Th, Visual Gamma Scan	Visual	Visual
R7-200	HNO_3	Tues. & Thurs. → HNO_3	% TBP α Pu Ret HNO_3
1BX*	*HNO_3	*HNO_3	*HNO_3
1CX		HNO_3	
2EX		HNO_3	
E71A	Deposition	Deposition	Deposition
E Cell		Deposition	
1CC		I^{131}	
1CS		I^{131}	

**These analyses are required for critical mass control and are not to be omitted without the supervisor's approval.*

Gamma Scan, ZrNb, Ru^{103}, RuRh^{106}, Pa

PUREX PLANT
ROUTINE BATCH SAMPLE SCHEDULE
THORIUM OPERATION

Sample	Description	Batch Code(6)	Analyses and Computer Output	Frequency
A3,B3, C3	Dissolver Solution	1	HNO_3, AT, Th, Gamma Scan	Every Batch
D5	Feed Adjustment (Pre-Butt)	2	HNO_3, Th	Every Batch
D5	Feed (Post-Butt) Accountability	1	HNO_3, AT, Th, Gamma Scan, SpG, U^{233}, Pu Composite (5 batch)	Every Batch
D5	Composite	3	U-Isotopic, Fe, F^-, Al	Lab. Request
E1	Centrifuge Product	1	HNO_3, AT, Th, Gamma Scan	Every Batch
E5	Coating Waste	1	Visual, pH, AT, Th, Gamma Scan, Comp. (5 batch)	Every Batch
E5	Composite	3	Cs	Lab Request
E6	Feed Acidity	1	HNO_3	On Request
*E6	Rework Control	2	HNO_3, Th, *AT, Pu	Every Batch Containing Rework
E6	Alternate Acc't. Sample	3	HNO_3, AT, Th, Gamma Scan, SpG, U^{233}, Pu, U-Total, Composite (5) (5 batch)	Alternate to D5 Sample
F3	Composite	3	Fe, Al, F^-	Lab. Request
*F26 or F15	Presugar 1WW	1	Th, Al, *U^{233}, *Pu, U-Total, Gamma Scan, Cs, HNO_3	Every Batch
F16	Post Sugar 1WW	2	CR	Every Batch
*(Alt.) F15	Post Sugar 1WW	3	Th, Al, *U^{233}, *Pu, U-Total, Gamma Scan, Cs, CR	To be taken if Presugar Sample not good
(Alt.) F16	Neutralized Waste	1	pH	Every Batch

This page uses bold and italic as printed: **These samples are required for critical mass control and are not to be omitted without the supervisor's approval.*

Gamma Scan ZrNb, Ru^{103}, $RuRh^{106}$, Pa

PUREX PLANT
ROUTINE BATCH SAMPLE SCHEDULE
THORIUM OPERATION

Sample	Description	Batch(6) Code	Analyses and Computer Output	Frequency
*F18	Sump Waste	1	Pu, *U^{233}, U-Total, Th, Gamma Scan, Cs, CR	Every Batch
F18	Neutralized Waste	3	pH	Every Batch
G8	OWW	1	pH, Th, U^{233}, Gamma Scan, Composite (5 batch)	Every Batch
G8	Composite	3	Cs	Lab Request
J21	Composite	3	U-Isotopic, Th	Lab Request
J5A	Composite	3	U-Isotopic, Th	Lab Request
K6	Thorium Prod	1	HNO_3, Th, U-Total SpG, Gamma Scan Comp. (10 batch)	Every Batch
K6	Composite	3	Pu, U^{233}, TMI, SO_4^{--} Cl^-, UX_1	Lab Request
*L11	U^{233} Rework	1	HNO_3, *U^{233}, *Pu	Every Batch
*L9	U^{233} Product	1	*U^{233}, HNO_3, Gamma Scan, TMI, *AT, Th, SpG, Composite (5 Batch), UMPIRE(4), U-Isotopic	Every Batch
L9	Bottle Sample	2	U^{233}, SpG	On Request
L9	Composite	3	Fe, Np, Pu, SO_4^{--}, F^-, Na Cl^-, W, Hg, UMPIRE(4)	Lab. Request
R8	OWW	1	pH, AT, Th, Gamma Scan, Comp. (5 batch)	Every Batch
R8	Composite	3	Cs	Lab Request
SA, SB, SC, SD, SE, SFA, SFB, SG, SJ, SK	Sumps to F18	1	Pu, U^{233}, U-Total, HNO_3 on Request	Every Batch
*SLA, SLD, SN	Sumps to E6	1	*Pu, *U^{233}, U-Total, HNO_3	Every Batch

---

**These analyses are required for critical mass control and are not to be omitted without the supervisor's approval.*

Gamma Scans, ZrNb, Ru^{103}, $RuRh^{106}$, Pa

PURKEX PLANT
ROUTINE BATCH SAMPLE SCHEDULE
THORIUM OPERATION

Sample	Description	Batch Code (6)	Analyses and Computer Output	Frequency
U1/U2	Recovered Acid	1	HNO_3, Gamma Scan, Composite (5 batch)	Every Batch
U1/U2	Composite	3	Fe, F^-, Al	Lab. Request

---

(1) *3 batch composite. See routine batch list for required analyses.*

(2) *Samples twice per shift - two numbers will be generated by the computer for J1, J22 and L1 when routine list is prepared, the second number will be held for the mid-shift sample.*

(3) *10 batch composite. See routine batch list for required analyses.*

(4) *Lab Quality Control Program Card - no results returned.*

(5) *The E6 alternate accountability sample composite is to be combined with the D5 composite.*

(6) *Enter the sample point and the batch code number on the dispatcher's keyboard to request the analyses listed for each.*

Gamma Scan ZrNb, Ru^{103}, $RuRh^{106}$, *Pa.*

PURKEX PLANT
ROUTINE BATCH SAMPLE
AQUEOUS MAKEUP
THORIUM OPERATION

Makeup	Tank	Analyses	Computer Code
$Fe(SA)_2$	TK-101	Fe^{++}, pH	1
2DIS-H_3PO_4	TK-103/ 218/219	HNO_3 H_3PO_4	1 3
$NaNO_3$	TK-104	$NaNO_3$**, SpG**	-
Syn 2AF	TK-105	HNO_3	1
$Na_2CO_3 \circ KMnO_4$	TK-107	Na_2CO_3**, SpG**	-
XCX	TK-151	HNO_3	1
$Cd(NO_3)_2$	TK-151	$Cd(NO_3)_2$*	1
KF	TK-206	KF	1
HAS	TK-209/210	HNO_3 H_3PO_4, Fe^{++}	1 3
*2AS	TK-220/221	Fe^{++}, *HNO_3	1
2BX	TK-224/225	HNO_3	1
*3AS	TK-211/212	*HNO_3	1
3BX	TK-213/214	HNO_3	1

---

*These analyses are required for critical mass control and are not to be omitted without the supervisor's approval.

**Qualitative test run by AMU operator.

PUREX PLANT
202-A BUILDING CRIB AND EFFLUENT SAMPLE SCHEDULE
THORIUM OPERATION

Sample (Computer Design.)	Stream	Sample Point	Batch(6) Code	Analyses and Computer Output	Frequency
216-A10C	Process Condensate	A5 Proportional Sampler	1	U, pH, U^{233}, Th, Total β, Pu, Comp.	One/Week
			3*	Dissolved Salts, U, U^{233}, Th, Pu, Total β, pH, NO_3^-, Cs^{137}, Ru^{106}, Co^{60}	2 Mo. Comp.
216-A30C	Steam Condensate	Japanese Proportional Sampler Near Crib	1	U, U^{233}, Pu, Th, Total β, Comp.	One/Week
			3*	Dissolved Salts, U, U^{233}, Pu, Th, TB, pH, NO_3^-, Cs^{137}, Ru^{106}, Co^{60}	2 Mo. Comp.
216-A36C	Ammonia Scrubber Waste	Proportional Sampler Station So. of 202-A	1	Total β, Comp.,U,Pu,pH, U^{233}, Th, Dissolved Salts	One/Day During Operation
			3*	Dissolved Salts, U, U^{233}, Pu, Th, Total β, pH, NO_3^-, Cs^{137}, Ru^{106}, Co^{60}, Ce^{144}, Sb^{125}, Pm^{147}	
TK-U3 & TK-U4	Lab Waste (to 216-A27)	Sample Gallery	1	U, U^{233}, Pu, Th, Total β, pH, Comp.	Every Batch
			3*	Dissolved Salts, U, U^{233}, Pu, Th, Total β, pH, NO_3^-, Cs^{137}, Ru^{106}, Co^{60}, Sb^{125}	2 Mo. Comp.

**On Lab Request Only.* (Performed by BNW).

<u>NOTE</u>: *Two samples required for all crib and effluent samples. (One for Lab. Sample and one for composite.)*

PUREX PLANT
202-A BUILDING CRIB AND EFFLUENT SAMPLE SCHEDULE
THORIUM OPERATION

Sample (Computer Design.)	Stream	Sample Point	Batch(6) Code	Analyses and Computer Output	Frequency
TK-216-A2	Misc. Wastes	Vacuum Sampler Above Tank	1	Total β, pH, Comp.	Every Batch
			3*	Dissolved Salts, U, U^{233}, Pu, Th, Total β, pH, NO_3^-, Cs^{137}, Ru^{106}, Co^{60}	2 Mo. Comp.
C-Sew	Chem Sewer	Japanese Proportional Sampler NE - 202-A	1	U, U^{233}, Pu, Th, Dissolved Salts, pH, Cd^{++}, Total β, Comp.	One/Week
			3*	Dissolved Salts, U, U^{233}, Pu, Th, Total β, pH, NO_3^-, Cs^{137}, Ru^{106}, Co^{60}, Cd^{++}	2 Mo. Comp.
Pump Pit	Cooling Water	241A-201 Pump Pit	1	Total β, U, U^{233}, Pu, Th, pH	One/Week
			3*	Dissolved Salts, U, U^{233}, Pu, Th, Total β, pH, NO_3^-, Cs^{137}, Ru^{106}, Co^{60}	
S-Flu	Stack Flush	TK-216-A2	1	Total β, U, U^{233}, Pu, Th, pH, Dissolved Salts, Comp. (*NOTE: Comp. Sample to be added to TK-216-A2 Comp.*)	Every Batch

**On Lab Request Only.*

NOTE: *Two samples required for all crib and effluent samples. (One for Lab. Sample and one for composite.)*

PUREX PLANT
202-A BUILDING CRIB AND EFFLUENT SAMPLE SCHEDULE
THORIUM OPERATION

Sample (Computer Design.)	Stream	Sample Point	Batch(6) Code	Analyses and Computer Output	Frequency
U8	Fractionator Overheads (To A9)		1	U, U^{233}, Pu, Th, Total β, pH	One/Week
			3*	Dissolved Salts, U, U^{233}, Pu, Th, Total β, pH, NO_3^-, Cs^{137}, Ru^{106}, Sr^{90}, I^{131}, Ca, Co^{60}	2 Mo. Comp.
P-Cond.	P Tank Coil Condensate	203 Area	1	U	One/Week
203-AS	203 Area Sumps	203 Area	1	U, Th, Nitric	Every Batch

**On Lab Request Only.*

NOTE: *Two samples required for all crib and effluent samples. (One for Lab. Sample and one for composite.)*

PUREX PLANT
STARTUP SAMPLE SCHEDULE
THORIUM OPERATION

The following samples and frequencies are recommended for plant startup control, in addition to the routine sample schedule. Sampling of each point should be started as the product level starts to increase at this point and should be continued until the shift supervisor and process control engineer are satisfied with the results. (NOTE: *These sample points are "batched" in the computer under Code 2).*

1. To be sampled once per hour:
    - a. J8-1BT - Visual
    - b. K4-2ET - Visual
2. To be sampled every two hours:
    - a. H2-HAW - HNO_3, Th, U^{233}
    - b. J22-2AW/2NW - HNO_3, AT , visual
    - c. L1-3AW/2AW - HNO_3, AT , visual
    - d. K2 - 2DW - HNO_3, Th , visual
3. To be sampled every four hours:
    - a. 1BX/1CX - HNO_3
    - b. 1CX/HCX - HNO_3
    - c. 2EX - HNO_3
    - d. H3/1CW -AT, Visual
    - e. K3/2EW -Th, Visual

ROUTINE STREAM SAMPLE RESULT PRIORITIES

Priority I

The following sample analyses should be handled on a rush basis. The results are essential for critical mass and product loss control. Note to Samplers: These samples should be taken first and delivered to the lab immediately.

H2-HAW	Th	
K2-2DW	Th	
J1-1UC	AT	(twice/shift)
J22-2AW/2NW	AT	(twice/shift)
L1-3AW/2AW	AT	(twice/shift)

Priority II

H2-HAW	HNO_3, U-233
J1-1UC	HNO_3
J2-1BSU	AT
J3-1BXF	AT, Th
J7-1BU	AT, Th
J8-1BT	AT, Th
J22-2AW/2NW	HNO_3
J23-2BW/2PW	AT
L1-3AW/2AW	HNO_3
L2-3BW/2BW	AT
K2-2DW	HNO_3
1BX/1CX	HNO_3

Priority III

All other analyses.

APPENDIX G

PUREX ANALYTICAL LABORATORY QUALITY CONTROL PLAN FOR 1970 THORIA CAMPAIGN

SECTION A

THORIA QUALITY CONTROL

This report will describe and detail steps taken to provide accurate knowledge of input and product solutions during thoria processing. This plan will: (1) establish or identify those procedures and controls to be used in the analysis of specified samples; (2) establish and define the means to determine and maintain the accuracy, precision and reliability of the specified measurements; and (3) establish and define responsibilities for the various quality control functions. The plan is based on the policies and definition of responsibilities as stated in Atlantic Richfield Hanford Company Operating Instruction 4.7.1.1, dated May 28, 1968.

INPUT SAMPLE SUMMARY

After dissolution of the dejacketed slugs has taken place, the solution is moved forward batchwise to TK-D5 before entering the solvent extraction system. Samples of the D5 material are analyzed to determine the amount of uranium and thorium put into the plant and to determine the quality of that material. The official amount of material charged into the plant is determined by Douglas United Nuclear, Inc. calculations.

INPUT ACCOUNTABILITY

Input accountability will be determined using the following analytical procedures:

Thorium by PAL AP Th-001 - ThXR-1A

X-ray emission spectrography is used for qualitative and quantitative analysis of thorium. High energy primary X-rays are emitted from a target tube and strike the sample causing an electron to be ejected as a quanta of energy, which is characteristic for each element. This primary energy and secondary radiation characteristic of the elemental composition of the sample is measured.

Uranium-233 by a composite of modified procedures. Uranium-233 will be analyzed using the theory and name of PAL AP Pu-001, PuA-6b + AEA. Differences between procedure and practice:

1. Theory-^{233}U is determined rather than plutonium.

2. The interference of thorium rather than uranium is taken care of by applying a correction for its alpha counts.

3. Collodion will not be used on the discs.

4. The addition of an alpha energy analysis will be required.

Alpha Energy Analysis

Alpha energy analysis will be based in part on theory found in PAL AP Np-007 and PAL AP AE-001. Calculations are based in part on theory of PAL SOP 5.16. Calculations for ^{233}U will be in the computer and at the clerk's desk.

Specific Gravity by PAL AP SpGr-003

A drop of the sample of accurate volume is allowed to fall through an accurately measured distance in a column of a calibrated immiscible organic liquid medium of slightly lower specific gravity than the drop.

Sample Validity by PAL SOP 6.2

A specific gravity determination (as above) is made on each sample received and compared with Purex Operations board specific gravity. The range between the two is used to determine the validity of the sample.

Accountability Analysis Frequency

Thoria and ^{233}U accountability analyses will be performed in duplicate with standards in duplicate. Range between duplicate thorium analyses should be $\leq$ 2.5 percent. Range between ^{233}U duplicate analyses should be $\leq$ 5.0 percent. Specific gravity analyses will be made single determination and compared with Purex Operations Board SpGr. Range between PAL and PO specific gravity determinations are set forth in PAL SOP 6.2. All accountability analyses (Th, ^{233}U and SpGr) will be refereed (run as the original by a second person). Thoria and ^{233}U referee limits are 2.5 and 5.0 percent respectively of the original average. Laboratory specific gravity referee limits are .003 specific gravity units. All accountability analyses are corrected for standard recovery.

Input Material Quality

Input material quality will be determined as follows:

Nitric Acid by PAL AP H-008, HVC-2A

The sample is placed in solution of complexing agents, which prevent metal ions present from hydrolyzing; this makes it possible to titrate only the free acid. This analysis may be required on each valid D5 sample to determine conformity with process specifications before other analyses are performed.

Alpha Total by PAL AP Pu-001 PuA-6b

Total alpha emission is determined radiochemically by transferring an aliquot of the sample directly onto a 22 mm stainless steel disc, drying, flaming and measuring on a standard Alpha Simpson Proportional Counter.

Gamma Energy Analysis by PAL AP G-001, GE-1A

Gamma photons are absorbed in a Lithium-Drifted Germanium Detector and produces a voltage pulse proportional to the energy of the photon. This voltage pulse is analyzed and stored according to its energy. Quantitative statements are based on the number of counts in a given energy range.

Plutonium by PAL AP Pu-003, PuA-20A

Acidity and heat are used to destroy colloidal and polymeric forms of plutonium. The plutonium valence is adjusted, plutonium is extracted into TTA, mounted on stainless steel discs and counted on an ASP counter.

Iron by PAL AP Fe-002, FeS-1b

Iron is reduced to the ferrous state. Ortho-phenanthroline is then added and forms a colored complex, the sample is then read spectrophotometrically.

Fluoride by PAL AP F-003, FSE-1b

The sample is placed in a high concentration of phosphate ion to complex the metal ions and release the fluoride. The potential of the fluoride specific ion electrode versus a reference electrode is used to calculate fluoride concentration.

Aluminum by PAL AP Al-002, AlS-1b

Oxine reacts with aluminum to form a yellow complex which is quantitatively extracted into xylene and read spectrographically.

Uranium Total by PAL AP 14.90-1, U-1X

Uranium is quantitatively retained on an anion exchange column, separating it from thorium and fission products. The uranium is eluted, mounted on a platinum dish fused with a Na-LiF flux and read on a fluorimeter.

Uranium-Isotopic Analysis

The elutant from PAL AP 14.90-1 is analyzed for isotopic content by Plutonium Finishing Analytical Laboratory.

Quality Analyses Frequency

Each batch of D5 material is analyzed, as above, to determine nitric acid concentration, alpha total, gamma emitting isotopes, plutonium concentration and total uranium concentration. A five batch composite is made using 1 ml from each D5 batch during that time. The five batch composite is analyzed, as above, for iron, fluoride, aluminum and uranium isotopic concentration.

SECTION B

THORIUM PRODUCT

SUMMARY

After decontamination and final concentration, the thorium product is moved batchwise to TK-K6. The solution is analyzed to determine the quantity and quality of thorium before the solution is removed from the plant.

THORIUM PRODUCT

The thorium product will be analyzed to determine the amount and quality of the thorium being recovered. Thorium and specific gravity analyses are used for accountability measurement. Other analyses noted in this section are used to determine product quality.

ACCOUNTABILITY MEASUREMENT

Thorium by PAL AP Th-001, ThXR-1A

X-ray emission spectrography is used for qualitative and quantitative analysis of thorium. High energy primary X-rays are emitted from a target tube and strike the sample causing an electron to be ejected as a quanta of energy. This primary energy and secondary radiation characteristic of the elemental composition of the sample is measured.

Specific Gravity by PAL AP SpGr-003

A drop of sample of accurate volume is allowed to fall through an accurately measured distance in a column of a calibrated immiscible organic liquid medium of slightly lower specific gravity than the drop.

Accountability Measurement Frequency

Thorium accountability analyses (Th by ThXR-1A and SpGr by DZ-1a) are done in duplicate with standards also in duplicate. The results are corrected for standard recovery. All product accountability analyses are refereed. The referee program will cover only the product accountability analyses and will be performed as the original by a second person.

Duplicate thorium analyses (ThXR-1a) should have a range of $\leq$ 2.5 percent. Referee results should be within 2.5 percent of the original $\overline{X}$. Specific gravity duplicate analyses should have a range of $\leq$ .003 specific gravity units. Referee results should be within 2.5 percent of the original $\overline{X}$.

All accountability analyses are corrected for standard recovery.

Thorium product quality will be determined as follows:

Nitric acid by PAL AP H-008, HVC-2A.

Uranium-233 by PAL AP 14.90-1, U-1X (AT mount).

Uranium Total by PAL AP 14.90-1, U-1X (UF mount).

Alpha Total by PAL AP Pu-001, PuA-6b.

Plutonium by PAL AP Pu-003, PuA-20A.

Gamma Energy Analyses by PAL AP G-001, GE-1A.

Thorium-234 by PAL AP G-001, GE-1A.

Summaries of the above methods may be found under input analyses.

Chloride by PAL AP.

Sulfate by PAL AP S04-002, S04-1A

Interfering ions are removed from the sample using appropriate organics. Sulfate is complexed by adding $BaCl_2$ in excess. The excess is extracted with TTA in hexone, stripped into 1 M HNO_3 and measured using the flame spectrophotometer.

Total Metal Impurities

Total metal impurity (TMI) analysis is performed by PFAL. Probable results will cover aluminum, boron, beryllium, bismuth, calcium, cadmium, cobalt, chromium, copper, iron, potassium, lithium, magnesium, manganese, sodium, nickel, phosphorus, lead, silicon, tin and zinc.

This list does not cover all analyses that may be done, nor is it intended that all elements will be reported.

Quality Measurement Frequency

Each batch of K-6 is analyzed to determine nitric acid, ^{233}U, total uranium, alpha total, plutonium and gamma emitting isotope concentrations.

A five batch composite is used for chloride, sulfate, total metal impurities and ^{234}Th determinations.

SECTION C

^{233}U PRODUCT

SUMMARY

After decontamination and final concentration, the ^{233}U product is moved batchwise to TK-L9. The solution is analyzed to determine the quantity and quality of uranium before the solution is removed from the plant.

ACCOUNTABILITY MEASUREMENT

Uranium-233 product accountability measurement will be determined as follows:

Uranium-233 by PAL AP U-007, UC-2A

Uranium is determined by the exchange of electrons between a mercury pool and an electrolyte which contains the sample. The method is based on the exact measurement of the quantity of electricity passed through the solution during the reaction.

Specific Gravity by PAL AP SpG-003, DZ-1A

A drop of the sample of accurate volume is allowed to fall through an accurately measured distance in a column of a calibrated immiscible organic liquid medium of a slightly lower specific gravity than the drop.

Accountability Measurement Frequency

Uranium-233 accountability analyses (UC-2A and DZ-1A) are done in duplicate with standards also done in duplicate. All product accountability analyses are refereed. The referee portion of the program will cover only the product accountability analyses and will be performed as the original by a second person. Duplicate uranium analyses should have a range of 1.8 percent, referee range should be 2.0 percent of the original average. Duplicate specific gravity analyses should have a range of .002 specific gravity units, referee range should be .003 SpGr units.

All accountability analyses are corrected for standard recovery.

Uranium-233 Quality Measurement

Material quality will be determined as follows:

Alpha Total by PAL AP Pu-001 (modified) PuA-6b.

Nitric acid by PAL AP H-008, HVC-2A.

Gamma Energy Analysis by PAL AP G-001, GE-1A.

Protactinium by PAL AP 14.90-1 followed by PAL AP G-001, GE-1A.

Uranium-232 by PAL AP's Pu-001 and AE-001 (both modified).

PuA-6b and AEA-1A.

Thorium by PAL AP 14.90-1, U-1X.

Total metal impurities* by Plutonium Finishing Analytical Laboratory.

Isotopic Analysis by Plutonium Finishing Analytical Laboratory.

Summaries of the above methods may be found under Input Analyses.

*Under Thoria QC.

Sodium by PAL AP Na-001, NaFS-1A

The sample is aspirated into an O_2-H_2 flame. Spectral lines (characteristic of elements present) of the emitted light are separated by an optical system. Intensity of certain spectral lines is measured with a photodetector to determine concentrations.

Iron by PAL AP Fe-003, FES-1e

All iron in the sample is reduced to the ferrous state, which forms a colored complex with ortho-phenanthroline. Uranium interference is corrected for by preparing a "blank" containing an amount of uranium equal to the sample. Iron concentration is determined spectrophotometrically by measuring absorption at 510 mu.

Neptunium by PAL AP Np-015, NpA-6c

Neptunium-237 is separated from other alpha emitters using thenoyltrifluoroacetone (TTA) and tri-iso-octylamine (TIOA), mounted and alpha counted.

Mercury will be determined using a spectrophotometer method.

Tungsten will be determined using a spectrophotometer method.

Fluoride by PAL AP F-003, FSE-1A.

Chloride and bromide will also be determined. The method of determination is not final.

Quality Measurement Frequency

Each batch of L9 solution is analyzed to determine total alpha emitter, ^{232}U, nitric acid, gamma emitting isotope, protactinium, thorium, sodium and iron concentrations. Total Metal Impurity and Isotopic Analyses are performed by Plutonium Finishing Analytical Laboratory on each batch of L9 material.

SECTION D

GENERAL LABORATORY QUALITY CONTROL

STANDARDS

Analytical standards, used for this plan are prepared by the Technical Services Section Standards Laboratory. The standards used for this plan, with the exception of the specific gravity standard, are synthetic solutions simulating plant solutions.

The specific gravity standard is obtained from TSS Standards Laboratory and is similar to plant material only in specific gravity characteristics. It does not simulate a plant solution.

For additional information, see ARH-1494, "Standards Used In the LSR Program".

EQUIPMENT CONTROLS

Diluent Dispensing Burets

Dispensing equipment, which is used to provide known volumes of sample diluents, will be calibrated every six months or as necessary due to equipment failure. The calibration is performed according to Purex Analytical Laboratory SOP No. 4.62 as described below.

Dispensing equipment is calibrated by weighing a liquid dispensed from the equipment at a given temperature. The volume dispensed by the equipment is calculated from the weight and specific gravity of the liquid.

Coulometer

At least once each six months, the coulometers in service are calibrated over the analytical ranges used and calibration factors calculated for the isotope concentrations in the material being processed. These factors are posted on the instrument and at the calculator's desk.

Calibration is made by connecting 100.1^5 ohm test cell to the rear of the coulometer in place of the sample cell lead. The potential across the test cell is measured with a millivolt box. If the potential varies beyond one percent of previous calibration, equipment repair is performed.

Specific Gravity Equipment

1. Drop size of sample is measured with a manostat.

   The manostat uses an accurate gear drive to expel liquid in a hydraulic system.

2. Temperature of the system is measured to 0.1 °C by a calibrated thermometer.

3. Standardized falling drop media obtained from TSS Standards Laboratory is used in the specific gravity equipment.

pH Meter

Commercial buffers pH 7 and 4 are used to calibrate and determine linearity of the pH meter. The pH meter is checked once/shift.

Pipets

A random sampling of all pipets used by Purex Analytical Laboratory is made to assure that pipets are within established tolerances. For 50 and 100 lambda pipets, the tolerance is ± 0.3 percent. Calibrated pipets are within .005 lambda of their stated value.

Alpha Counting Instruments

1. ASP Counters are operated according to PAL SOP 5.04. The procedure lists steps required for proper performance of the instrument, including positioning of sample and counting times.

2. Instrument control is maintained according to PAL 5.03, 5.05 and 5.08. AP 5.03 describes the checks required by each shift (three per day) before the instrument is used and further actions to be taken if the instrument fails a check. AP 5.05 describes correlation and control tests to be made when an instrument is placed into service and at scheduled intervals during its use. AP 5.08 - Shift Standard Tests and $\overline{X}$ - this test is designed to indicate any change in counting efficiency or detect erratic instrument fluctuations.

Alpha Energy Analysis Equipment

Alpha Energy Analysis Equipment is operated according to the theory of PAL SOP's 5.20, 5.21 and 5.27. These SOP's give a general description of the physical instrument, functional components, operational procedure and method of calculating results on a prototype instrument.

Spectrophotometric Instruments

DK-2 - This instrument is operated according to PAL SOP 5.18 and 5.19. These procedures cover the physical description, functions, set-up and operation of the instrument covering both samples and standards.

DU Flame Spectrophotometer - This instrument is operated according to PAL SOP 5.28. The procedure covers operation of the instrument and covers standards which are run with each sample.

X-ray Fluorescence Equipment - XRD-6. SOP 5.34 covers the component functions of the instrument and explanations, both theoretical and actual, of its uses.

Control Plan

The validity of each sample will be assured by using PAL SOP 6.2. See IV A-2.

Analytical method precision and accuracy will be determined using LSR Program document ARH-9. The minimum required standard frequency is determined from ARH-242. The laboratory Standards and Referee Program report is issued monthly, quarterly and (on request) yearly. These reports are described in ARH-242, section 3.1 and ARH-9. ARH-9 describes methods used to evaluate the precision and accuracy of the measurements produced by Purex Analytical Laboratory. ARH-242 defines the minimum requirements of the Laboratory Measurements Quality Control Program and assigns component responsibilities for performing the various parts of the program. Reports issued by TSS will provide the demonstration of the reliability of the Chemical Processing Division's analytical measurements.

Numbers generated by the laboratory will be governed by PAL SOP's 8.3 - Calculating and Reporting Limits and 4.2 - Rounding Numbers.

Limits for laboratory analyses are established using a yearly LSR report. Results failing to meet requirements are checked as stated in letter 3-21-69, "Out of Limits Standards" to all PAL Supervisory personnel.

In addition to established limits, noted analyses are governed by the following actions.

See Accountability and Quality Measurement Frequencies under individual sample point Q.C. Plans covered earlier. Also see Thoria Standard Frequency below.

Action in Event of Loss of Control

Specific Gravity

1. Check pipeter - This can be done using PAL SOP's 6.1 and/or 4.62.
2. Check falling drop equipment - check manostat, temperature and falling drop media.
3. Establish that the analytical system is in control - The system is considered in control if three determinations by one person or single determinations by two people are within specified limits. (Established through LSR). The percent precision of the average determined from 12 months data is used to establish limits for laboratory performance.

Sample Validity

1. Rerun or resample - A resample is in order after it has been determined that a valid sample was not received. (See SOP 6.2 for D5).
2. Check pipeter - According to PAL SOP's 6.1 and 4.62.

Individual Analyses

1. Rerun analysis - Results of a valid sample that fall out of established limits demand a rerun.
2. If analysis remains out of control, corrective action in the form of method or equipment investigation or process variations are required.
3. Establish that system is in control.

Modifications to the Plan

Changes will be necessary to this plan as new data process requirements or method developments occur. These changes will be initiated with the approval of the Purex Analytical Laboratory Manager. Addendums to this plan will be used to document such changes but need not necessarily precede implementation of the change.

Responsibilities

The Purex Analytical Laboratory Manager will have overall responsibility for the operation of the analytical portion of this plan. Any delegation of specific responsibilities for the application of this plan shall be by written communication.

The Purex Analytical Laboratory Manager, or his appointed delegate, will have the responsibility for reviewing this plan at a minimum frequency of once per quarter.

The Purex Analytical Laboratory shift supervisors will be responsible for the collection and analysis of data to assure proper plan control and will take corrective action as specified in the plan.

The Quality Control Chemist is responsible for monitoring laboratory quality control performance by use of the LSR and the inclusion of charts, tests, etc., as deemed necessary. He is responsible for actions to correct long term out of control conditions and to maintain control conditions.

The Process and Methods Chemist is responsible for providing the technologies required to measure required material analyses and coordinate technological changes with SCL.

APPENDIX H

PURREX ANALYTICAL LABORATORY THORIUM SCHOOL OUTLINE

Monday - June 8, 1970

8-9	Introduction
9-1030	Process Flowsheet - Van der Cook
Break	
11-12	LSR Program (PAL QC Guide) - Abercrombie
Lunch	
1245-1415	Interpretation of Data - Don Shepard
Break	
1430-1600	Standard Make-up - Don Rochon

Tuesday

8-9	IBM 1800 - Atterberry
9-1030	Process Flowsheet (Cont'd.) - Van der Cook
Break	
11-12	LSR and Supervisor's Roll - Abercrombie
Lunch	
1245-1415	F^- Analyses and Coulometric Analyses - Weiss
Break	
1430-1600	Critical Mass - JD Anderson

Wednesday

8-9	U-1X Procedure - Anderson
9-1030	Process Flowsheet (Cont'd.) - Van der Cook
Break	
11-12	D5 Th Feed QC - Abercrombie
Lunch	
1245-1415	Cl, Br Analyses - ML Oliver
Break	
1430-1600	Gamma Scan Analyses - GT Furner

Thursday

8-9	TOPO Procedures - Anderson
9-1030	Process Flowsheet (Cont'd.) - Van der Cook
Break	
11-12	K6 Th Product QC - Abercrombie
Lunch	
1245-1415	Gamma Scan Lab - GT Furner
Break	
1430-1600	Alpha Energy Analyses - Anderson

Friday

8-9	ThS-1x Procedure - Anderson
9-1030	Process Flowsheet (Cont'd.) - Van der Cook
Break	
11-12	L9 U-233 Product QC - Abercrombie
Lunch	
1245-1415	Hg, W, Al - ML Oliver
Break	
1430-1600	XRD-Training - MJ Kupfer

APPENDIX I

THORIUM DRAWING INDEX (Ref. H-2-61850)

PIPING

Jumper Assemblies (New)

H-2-64707	F Cell Arrangement
H-2-64708	F-T97-(E-F11-1)W & F-T92A- T92B
H-2-64709	F-T107-(E-F11-2)C
H-2-64710	F-1-(TKF10)A (Alt)
H-2-64711	Steam to Tube Bundle
H-2-64712	F-G108-(E-F11-1)-J & J-G1E-(E-J8-1)-J
H-2-64713	D-T27-T30
H-2-64714	D-T7-(TK-D4)-T
H-2-64717	F-3-2
H-2-64718	F-G111-5
H-2-64719	F-4-(E-F11-1)K
H-2-64726	F-G103-(E-F11-1)D
H-2-64727	F-(E-F11-1)F-(TKF12)H
H-2-64728	F-G100-(E-F11-1)C
H-2-64729	F-T92B-(E-F11-1)B
H-2-64731	F-G99A-(E-F11-1)H
H-2-64735	F-G95-(E-F11-1)BB
H-2-64736	F-T138-T141
H-2-64737	F-T120-T127
H-2-64739	D-T10-(TKD4)-PD4
H-2-64740	D-G16-2
H-2-64741	D-T8-(TKD5)R
H-2-64742	D-T8-T3(Alt)
H-2-64743	F-G89-(E-F11-1)CC
H-2-64744	J-(TJ6)F-(TJ7)G
H-2-64745	J-G53-3
H-2-64746	J-T14-(TJ7)J
H-2-64747	J-10-11
H-2-64748	E-T47-T36
H-2-64749	F-T137-(TKF7)L
H-2-64750	F-G32-(TKF16)A
H-2-66034	Flushing Head Jumper for Dissolver Cone
H-2-66155	J-G97-(TK-J5)-E

Jumper Assemblies (Existing)

H-2-53550	E-T18-(TKE5)M (Alt)
H-2-53604	F-T161-(TKF3)P & F-T79-(TKF12)R
H-2-53622	E-T50(TK-E1)P
H-2-53667	Cooling Water Out (F-T92,T91,T90)
H-2-53688	F-T93-(E-F11-2)B
H-2-53701	D-G19-(TK-D4)-PD4
H-2-53921	Cooling Water in (F-G107,G106,G105)
H-2-53950	F-G98A-(E-F11-1)EE
H-2-53999	F-(E-F11-1)DD-(TKF12)J
H-2-54001	F-G97-8
H-2-54017	F-T86-(E-F11-1)AA
H-2-57199	F-T121-T122
H-2-57206	H-G30-3
H-2-57207	H-G28-2
H-2-57463	D-G19-D4
H-2-64519	H-G59-(TH2)A
H-2-57245	H-T4-T5
H-2-58235	F-(E-F11-1)TT-UU
H-2-58236	F-G112-(E-F11-1)X
H-2-58239	F-(E-F11-1)M-J
H-2-58240	F-(E-F11-1)S-WW
H-2-58242	F-G110A-(E-F11-1)SS
H-2-58246	F-G101-(E-F11-1)RR
H-2-58249	F-(E-F11-1)JJ-LL
H-2-58601	J-T62-T52

H-2-58621	H-G63-(TH2)CC
H-2-58671	F-(TKF16)M-10 (Alt)
H-2-59121	A-G6-(A3-1)BR, B-G6(B3-1) BR & C-G6-(C3-1)BR
H-2-59189	D-T18-(TK-D3)-U (Alt)
H-2-59652	F-G109-7 & FG194-J-M
H-2-59928	E-T38-(TKE3)M (Alt)
H-2-60003	A-T2(TKA3)A, B-T2-(TKB3)A, & C-T2-(TKC3)A
H-2-60122	D-T13-(TK-D4)R (Alt)
H-2-61852	J-G40-1
H-2-61853	Wt. Factor, Sp. Gravity & Diff. Press (Dissolvers)
H-2-61854	C-T8(TK-C3)AF
H-2-61856	J-T21-2
H-2-61857	J-T31-(PGJ6)A
H-2-61863	H-T44-(TH2)U
H-2-61865	J-T16-(TJ6)E
H-2-61868	J-T23-(TJ7)K
H-2-61869	J-T24-T25
H-2-61870	J-T46-(TJ2)AN
H-2-61871	J-G112-5
H-2-61873	J-T45-T50
H-2-61876	J-T31-(PGJ7)A
H-2-61878	H-G37-(TH3)G
H-2-61879	H-T21-(TH3)E
H-2-61880	H-T20-(TH3)K
H-2-61881	H-9-(TH4)V
H-2-61883	H-(TH3)F-9
H-2-61884	J-T23-7-8

Piping General

H-2-35743	U-233 Transloading Station - Over Center Latch
H-2-61888	"N" Cell Loadout Hood Arrangement
H-2-61891	P&O Gallery Piping Modification Plan
H-2-61892	P&O Gallery Piping Isometric Details
H-2-61898	"N" Cell Loadout Hood (Sheet 1)
H-2-61898	"N" Cell Loadout Hood (Sheet 2)
H-2-61900	Loadout Head Tank
H-2-61902	Cation Exchange Column
H-2-63451	U-233 Loadout Vessel - L13
H-2-64701	Piping Modification P&O Gallery and AMU Plan
H-2-64702	P&O Gallery Piping Isometric Details
H-2-64704	"N" Cell Loadout Hood Piping Arrangement
H-2-64705	Sectionalized Concentrator Remote Coil
H-2-64706	Sectionalized Concentrator Remote Jet & Flange
H-2-64723	U-233 Transloading Station-Plans & Details
H-2-64724	U-233 Transloading Station-Bottle Transfer Arrangement & Details
H-2-64725	"N" Cell Piping Modifications

Instrumentation & Engineering Flow Diagrams

H-2-61885	U-233 Concentration & Load Out
H-2-61903	First Decontamination and Partition Cycle
H-2-61904	Second Thorium Cycle

H-2-61905	Second Uranium-233 Cycle
H-2-61906	Third Uranium-233 Cycle
H-2-61907	Waste Processing & Acid Recycle (Sheet 1)
H-2-61907	Waste Processing & Acid Recycle (Sheet 2)
H-2-61908	Caustic Dejacketing
H-2-61909	Thoria Dissolution
H-2-64700	Sectionalized Concentrator (E-F11-1) & (TK-F12)

Instrument

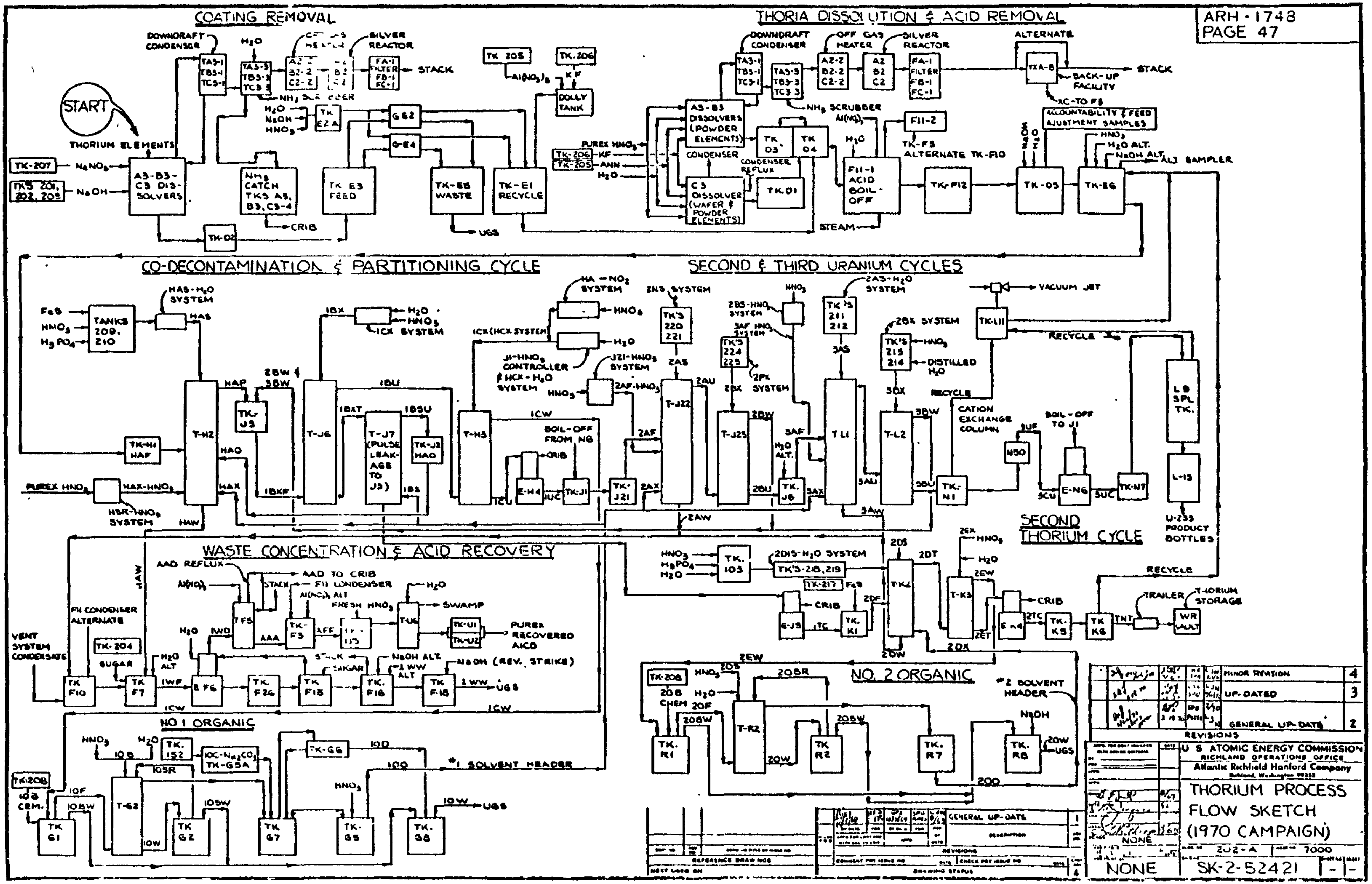
H-2-61916	Graphic Panels B1, B2, & B3 Piping & Wiring Modifications (Sheet 1)
H-2-61916	Graphic Panels B4, B5, B6, B8 and B17 Piping & Wiring Modifications (Sheet 2)
H-2-61916	Panel A3, N-Panel Board & G1 Piping & Wiring Modifications (Sheet 3)
H-2-61917	Cells J,H,N, & PR Room Impulse Piping Arrangement & Details (Sheet 1)
H-2-61917	Cells B,C,D, & F Impulse Piping Arrangement & Details (Sheet 2)
H-2-64756	Instrument Rear Panel Wiring Panel B9 & Elementary Diagrams
H-2-64757	Central Control Room Graphic Panel B9 Modifications
H-2-64758	Graphic Panel B9 Piping Modifications
H-2-64759	Cells D & F Impulse Piping Arrangement & Details
H-2-64760	Transmitter Racks 32, 15 & 32A - Converter Rack B9 Modifications

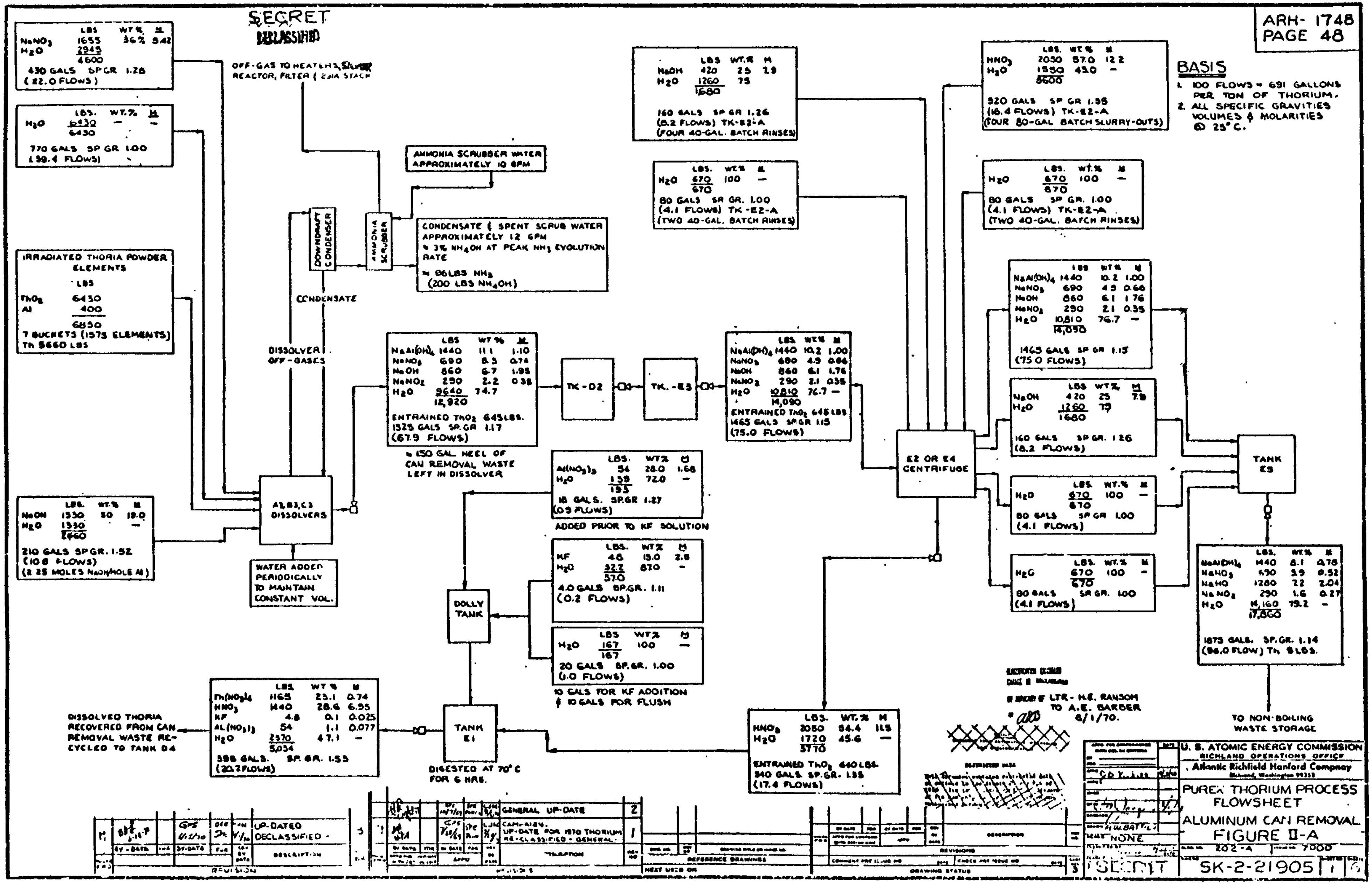
Electrical

H-2-64761	Electrical Plan & Diagrams

APPENDIX J

FIGURES

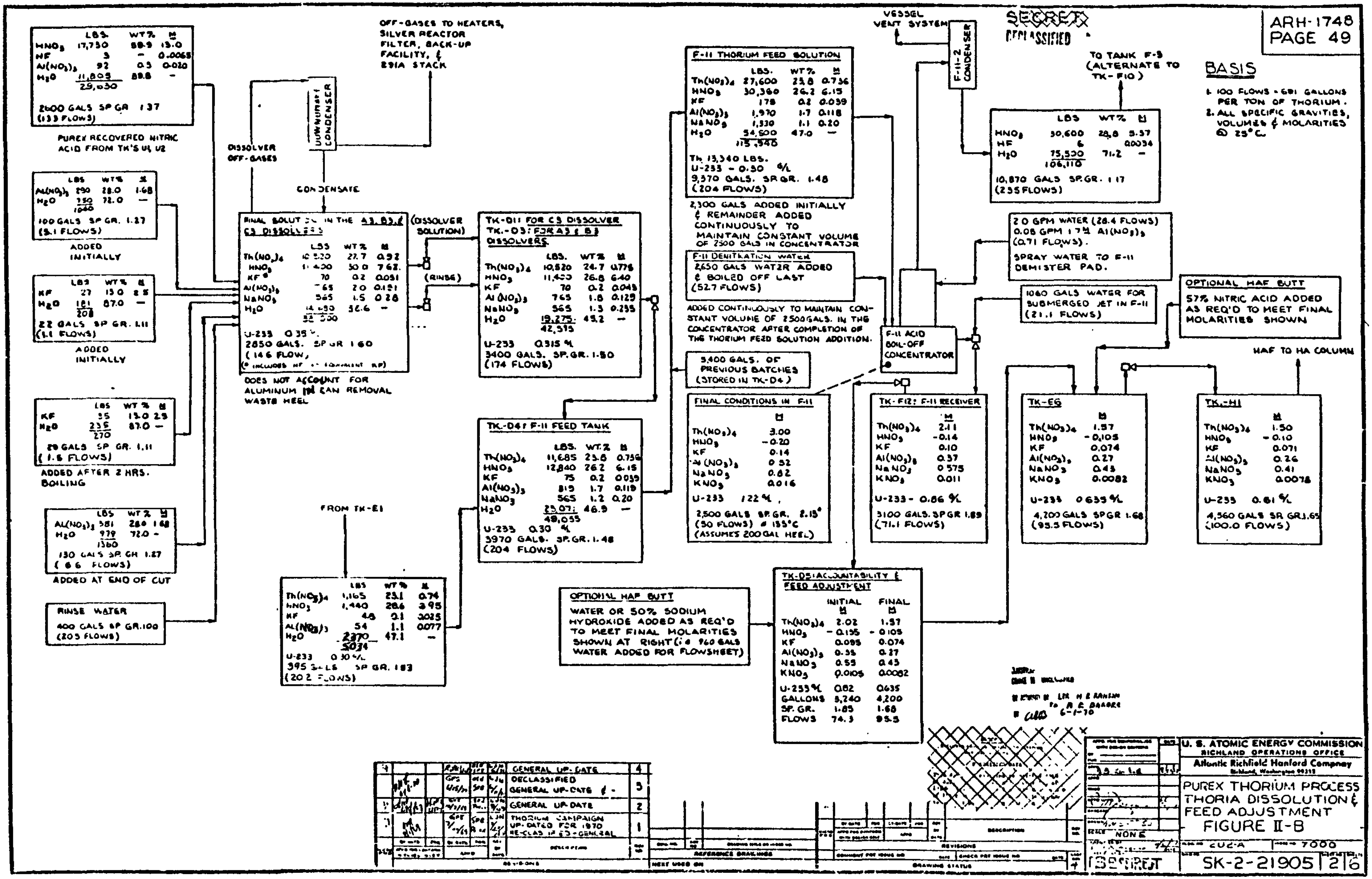

ARH-1748
PAGE 47
COATING REMOVAL
THORIA DISSOLUTION & ACID REMOVAL
START
THORIUM ELEMENTS
TK-207
$NaNO_3$
TKS 201, 202, 203
NaOH
A3-B3-C3 DISSOLVERS
DOWNDRAFT CONDENSER
TA3-1 TB3-1 TC3-1
TA3-3 TB3-3 TC3-3
NH_3 SCRUBBER
OFF GAS HEATER
A2-2 B2-2 C2-2
SILVER REACTOR
A2 B2 C2
FA-1 FILTER FB-1 FC-1
STACK
NH_3 CATCH TKS A3, B3, C3-4
CRIB
TK-D2
TK E2A
G E2
G E4
TK E3 FEED
TK-E5 WASTE
TK-E1 RECYCLE
UGS
TK 205
$Al(NO_3)_3$
TK-206
KF
DOLLY TANK
PUREX HNO_3
TK-206 KF
TK-205 ANN
H_2O
A3-B3 DISSOLVERS (POWDER ELEMENTS)
C3 DISSOLVER (WAFER & POWDER ELEMENTS)
CONDENSER
CONDENSER REFLUX
TK D3
TK D4
TK D1
F11-1 ACID BOIL-OFF
F11-2
TK-F5 ALTERNATE TK-F10
TK-F12
STEAM
ALTERNATE
TXA-B
BACK-UP FACILITY
AC-TO F3
ACCOUNTABILITY & FEED ADJUSTMENT SAMPLES
TK-D5
TK-E6
H_2O ALT.
NaOH ALT.
ALT. SAMPLER
CO-DECONTAMINATION & PARTITIONING CYCLE
SECOND & THIRD URANIUM CYCLES
FeS
HNO_3
H_3PO_4
TANKS 209, 210
HAS-H_2O SYSTEM
HAS
TK-H1 HAF
T-H2
HAP
HAO
HAX
HAW
PUREX HNO_3
HAX-HNO_3
HSR-HNO_3 SYSTEM
TK-J3
2BW & 3BW
1BX
ICX SYSTEM
T-J6
1BXF
1BU
1BXT
T-J7 (PULSE LEAKAGE TO J3)
1BSU
TK-J2 HAO
1BS
ICX (HCX SYSTEM)
HA-NO_2 SYSTEM
J1-HNO_3 CONTROLLER & HCX-H_2O SYSTEM
T-H3
ICW
BOIL-OFF FROM N6
E-H4
ICU
IUC
TK-J1
J21-HNO_3 SYSTEM
2AF-HNO_3
TK-J21
2AF
2AX
2NS SYSTEM
TK'S 220 221
2AS
T-J22
2AU
2AW
2BS-HNO_3 SYSTEM
3AF HNO_3 SYSTEM
TK'S 224 225
2BX
2PX SYSTEM
T-J25
2BW
2BU
H_2O ALT.
3AF
TK-J8
3AX
2AS-H_2O SYSTEM
TK'S 211 212
3AS
T-L1
3AU
3AW
2BX SYSTEM
TK'S 215 214
DISTILLED H_2O
3BX
T-L2
3BW
3BU
RECYCLE
CATION EXCHANGE COLUMN
TK-N1
N50
3UF
BOIL-OFF TO J1
E-N6
3CU
3UC
TK-N7
VACUUM JET
TK-L11
L8 SPL TK.
L-13
U-233 PRODUCT BOTTLES
SECOND THORIUM CYCLE
WASTE CONCENTRATION & ACID RECOVERY
AAD REFLUX
AAD TO CRIB
F11 CONDENSER
$Al(NO_3)_3$ ALT
FRESH HNO_3
SWAMP
T-F5
TK-F3
TK-U1
TK-U2
PUREX RECOVERED ACID
F11 CONDENSER ALTERNATE
VENT SYSTEM CONDENSATE
TK-204
SUGAR
TK F10
TK F7
IWF
IWD
E F6
TK F26
TK F15
TK F16
TK F18
NaOH (REV. STRIKE)
NaOH ALT
1WW ALT
ICW
NO 1 ORGANIC
TK-208
10B CHEM.
10F
10BW
TK G1
T-G2
10S
10SR
10SW
10W
TK 152
10C-Na_2CO_3 TK-G5A
TK-G6
10D
10O
#1 SOLVENT HEADER
TK G7
TK G5
TK G8
UGS
TK. 103
2DIS-H_2O SYSTEM
TK'S-218, 219
TK-217
FeS
E-J8
1TC
TK K1
2DF
2DS
2DT
T-K2
T-K3
2DW
2DX
2EX
2EW
2ET
E K4
2TC
TK K5
TK K6
TNT
TRAILER
THORIUM STORAGE
WR VAULT
RECYCLE
NO. 2 ORGANIC
TK-208
20B CHEM
20S
20F
20BW
20SR
T-R2
20W
20SW
TK R1
TK R2
TK R7
TK R8
20O
#2 SOLVENT HEADER
20W
MINOR REVISION
UP-DATED
GENERAL UP-DATE
REVISIONS
U S ATOMIC ENERGY COMMISSION
RICHLAND OPERATIONS OFFICE
Atlantic Richfield Hanford Company
THORIUM PROCESS FLOW SKETCH (1970 CAMPAIGN)
NONE
202-A
7000
SK-2-52421
GENERAL UP-DATE
REVISIONS
REFERENCE DRAWINGS
DRAWING STATUS
NEXT USED ON

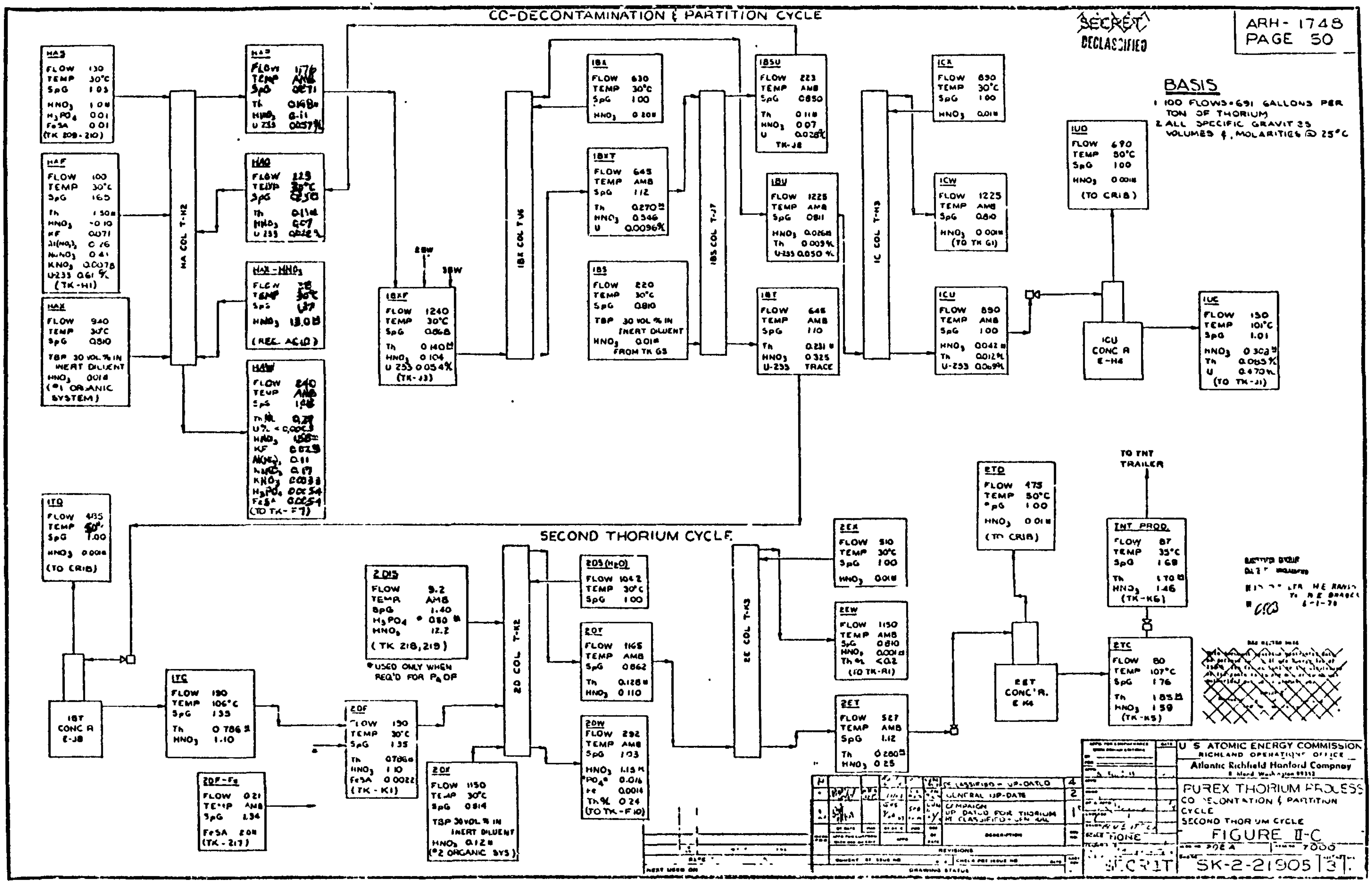


PUREX THORIUM PROCESS FLOWSHEET

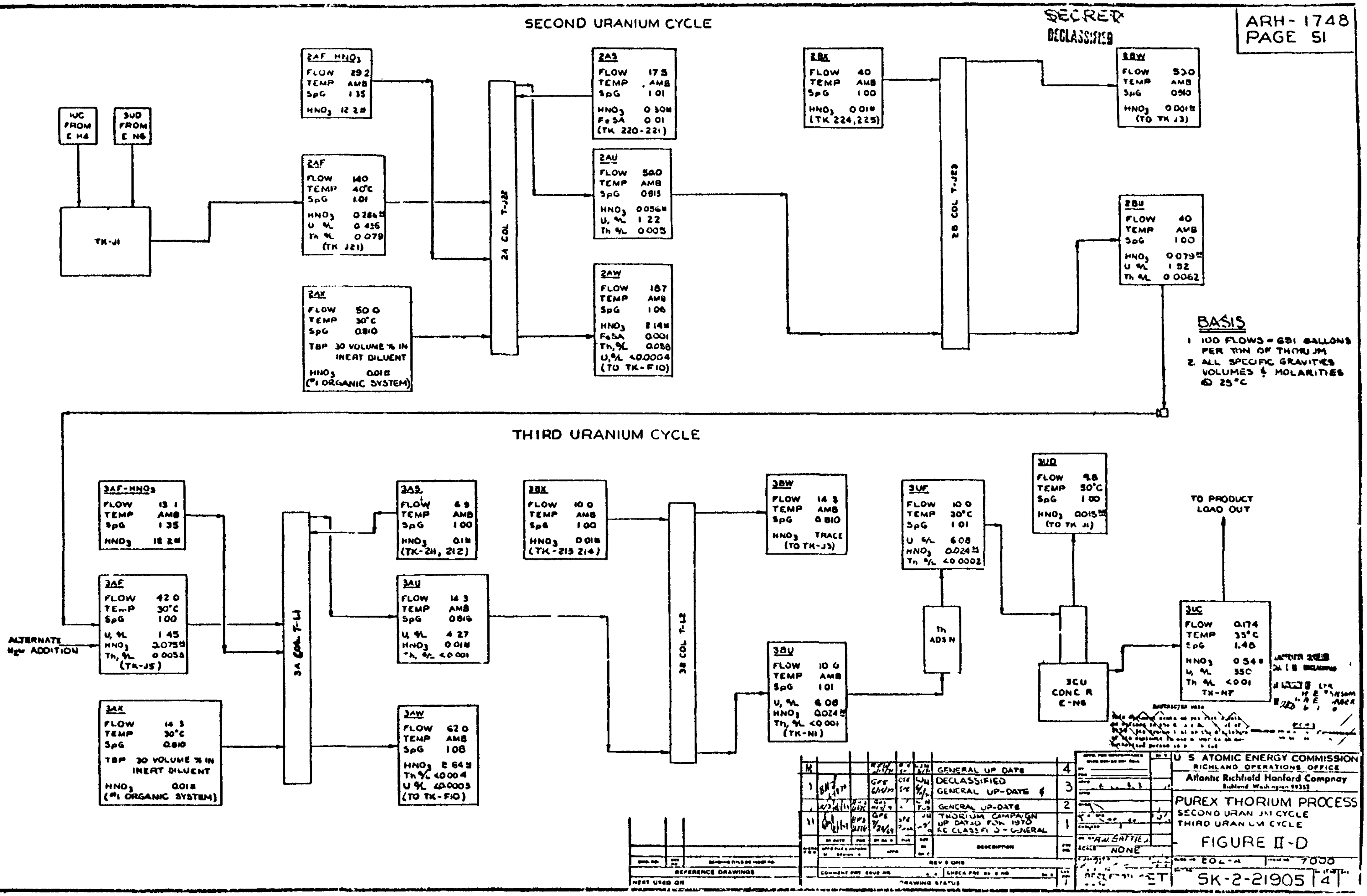
ALUMINUM CAN REMOVAL

FIGURE II-A


ARH-1748
PAGE 49
SECRET
DECLASSIFIED
BASIS
1. 100 FLOWS = 681 GALLONS PER TON OF THORIUM.
2. ALL SPECIFIC GRAVITIES, VOLUMES & MOLARITIES @ 25°C.
LBS. WT% M
HNO3 17,750 58.9 13.0
HF 3 – 0.0065
Al(NO3)3 92 0.3 0.020
H2O 11,805 38.8 –
29,030
2600 GALS SP GR 1.37
(133 FLOWS)
PUREX RECOVERED NITRIC ACID FROM TK'S U1 U2
LBS WT% M
Al(NO3)3 290 28.0 1.68
H2O 750 72.0 —
1040
100 GALS SP GR. 1.27
(5.1 FLOWS)
ADDED INITIALLY
LBS WT% M
KF 27 13.0 2.5
H2O 181 87.0 —
208
22 GALS SP GR. 1.11
(1.1 FLOWS)
ADDED INITIALLY
LBS WT% M
KF 35 13.0 2.5
H2O 235 87.0 —
270
29 GALS SP GR. 1.11
(1.5 FLOWS)
ADDED AFTER 2 HRS. BOILING
LBS WT% M
Al(NO3)3 381 28.0 1.68
H2O 979 72.0 –
1360
130 GALS SP GR 1.27
(6.6 FLOWS)
ADDED AT END OF CUT
RINSE WATER
400 GALS SP GR.1.00
(20.5 FLOWS)
OFF-GASES TO HEATERS, SILVER REACTOR FILTER, BACK-UP FACILITY, & 291A STACK
DOWNDRAFT CONDENSER
DISSOLVER OFF-GASES
CONDENSATE
FINAL SOLUTION IN THE A3, B3, & C3 DISSOLVERS
LBS WT% M
Th(NO3)4 10,520 27.7 0.92
HNO3 11,400 30.0 7.62
KF* 70 0.2 0.051
Al(NO3)3 765 2.0 0.151
NaNO3 565 1.5 0.28
H2O 14,450 38.6 –
38,000
U-233 0.35 %.
2850 GALS. SP GR 1.60
(14.6 FLOW,
(* INCLUDES HF AS EQUIVALENT KF)
DOES NOT ACCOUNT FOR ALUMINUM CAN REMOVAL WASTE HEEL
(DISSOLVER SOLUTION)
(RINSE)
TK-D11 FOR C3 DISSOLVER
TK.-D3: FOR A3 & B3 DISSOLVERS.
LBS. WT% M
Th(NO3)4 10,520 24.7 0.776
HNO3 11,400 26.8 6.40
KF 70 0.2 0.045
Al(NO3)3 765 1.8 0.129
NaNO3 565 1.3 0.255
H2O 19,275 45.2 —
42,595
U-233 0.315 %
3400 GALS. SP.GR. 1.50
(174 FLOWS)
TK.-D4: F-11 FEED TANK
LBS. WT.% M
Th(NO3)4 11,685 23.8 0.756
HNO3 12,840 26.2 6.15
KF 75 0.2 0.039
Al(NO3)3 815 1.7 0.119
NaNO3 565 1.2 0.20
H2O 23,071 46.9 —
49,055
U-233 0.30 %
3970 GALS. SP.GR. 1.48
(204 FLOWS)
FROM TK-E1
LBS WT% M
Th(NO3)4 1,165 23.1 0.74
HNO3 1,440 28.6 3.95
KF 48 0.1 .0025
Al(NO3)3 54 1.1 0.077
H2O 2370 47.1 –
5034
U-233 0.30 %
395 GALS SP GR. 1.83
(20.2 FLOWS)
VESSEL VENT SYSTEM
F-11-2 CONDENSER
F-11 THORIUM FEED SOLUTION
LBS. WT% M
Th(NO3)4 27,600 23.8 0.736
HNO3 30,360 26.2 6.15
KF 178 0.2 0.039
Al(NO3)3 1,970 1.7 0.118
NaNO3 1,330 1.1 0.20
H2O 54,500 47.0 —
115,940
Th 13,340 LBS.
U-233 – 0.30 %
9,370 GALS. SP GR. 1.48
(204 FLOWS)
2,300 GALS ADDED INITIALLY & REMAINDER ADDED CONTINUOUSLY TO MAINTAIN CONSTANT VOLUME OF 2500 GALS IN CONCENTRATOR
F-11 DENITRATION WATER
2,650 GALS WATER ADDED & BOILED OFF LAST
(52.7 FLOWS)
ADDED CONTINUOUSLY TO MAINTAIN CONSTANT VOLUME OF 2500 GALS. IN THE CONCENTRATOR AFTER COMPLETION OF THE THORIUM FEED SOLUTION ADDITION.
5,400 GALS. OF PREVIOUS BATCHES (STORED IN TK-D4)
F-11 ACID BOIL-OFF CONCENTRATOR
FINAL CONDITIONS IN F-11
M
Th(NO3)4 3.00
HNO3 –0.20
KF 0.14
Al(NO3)3 0.52
NaNO3 0.82
KNO3 0.016
U-233 1.22 %,
2,500 GALS SP GR. 2.15
(50 FLOWS) @ 135°C
(ASSUMES 200 GAL HEEL)
TK-F12: F-11 RECEIVER
M
Th(NO3)4 2.11
HNO3 –0.14
KF 0.10
Al(NO3)3 0.37
NaNO3 0.575
KNO3 0.011
U-233 – 0.86 %
3100 GALS. SP GR 1.89
(71.1 FLOWS)
TO TANK F-3 (ALTERNATE TO TK-F10)
LBS WT% M
HNO3 30,600 28.8 5.37
HF 6 0.0034
H2O 75,500 71.2 –
106,110
10,870 GALS SP.GR. 1.17
(235 FLOWS)
2.0 GPM WATER (28.4 FLOWS)
0.08 GPM 1.7 M Al(NO3)3 (0.71 FLOWS).
SPRAY WATER TO F-11 DEMISTER PAD.
1060 GALS WATER FOR SUBMERGED JET IN F-11
(21.1 FLOWS)
OPTIONAL HAF BUTT
57% NITRIC ACID ADDED AS REQ'D TO MEET FINAL MOLARITIES SHOWN
HAF TO HA COLUMN
TK-E6
M
Th(NO3)4 1.57
HNO3 –0.105
KF 0.074
Al(NO3)3 0.27
NaNO3 0.43
KNO3 0.0082
U-233 0.635 %
4,200 GALS SPGR 1.68
(95.5 FLOWS)
TK.-H1
M
Th(NO3)4 1.50
HNO3 –0.10
KF 0.071
Al(NO3)3 0.26
NaNO3 0.41
KNO3 0.0078
U-233 0.61 %
4,560 GALS SP GR 1.65
(100.0 FLOWS)
OPTIONAL HAF BUTT
WATER OR 50% SODIUM HYDROXIDE ADDED AS REQ'D TO MEET FINAL MOLARITIES SHOWN AT RIGHT (i.e. 960 GALS WATER ADDED FOR FLOWSHEET)
TK-D5: ACCOUNTABILITY & FEED ADJUSTMENT
INITIAL M FINAL M
Th(NO3)4 2.02 1.57
HNO3 – 0.135 – 0.105
KF 0.095 0.074
Al(NO3)3 0.35 0.27
NaNO3 0.55 0.43
KNO3 0.0105 0.0082
U-233 % 0.82 0.635
GALLONS 5,240 4,200
SP. GR. 1.85 1.68
FLOWS 74.3 95.5
LTR H E RANSON
To R E DAABERG
6-1-70
GENERAL UP-DATE 4
DECLASSIFIED
GENERAL UP-DATE & - 3
GENERAL UP-DATE 2
THORIUM CAMPAIGN UP-DATED FOR 1970 RE-CLASS IF ED - GENERAL 1
REVISIONS
REFERENCE DRAWINGS
NEXT USED ON
REVISIONS
DRAWING STATUS
DESCRIPTION
SCALE NONE
SECRET
U. S. ATOMIC ENERGY COMMISSION
RICHLAND OPERATIONS OFFICE
Atlantic Richfield Hanford Company
Richland, Washington 99352
PUREX THORIUM PROCESS
THORIA DISSOLUTION & FEED ADJUSTMENT
FIGURE II-B
202-A
7000
SK-2-21905
2
6


CC-DECONTAMINATION & PARTITION CYCLE
SECRET
DECLASSIFIED
ARH-1748
PAGE 50
BASIS
1 100 FLOWS = 691 GALLONS PER TON OF THORIUM
2 ALL SPECIFIC GRAVITIES VOLUMES & MOLARITIES @ 25°C
HAS
FLOW 130
TEMP 30°C
SpG 1.05
HNO_3 1.0M
H_3PO_4 0.01
FeSA 0.01
(TK 209-210)
HAF
FLOW 100
TEMP 30°C
SpG 1.65
Th 1.50M
HNO_3 -0.10
KF 0.071
$Al(NO_3)_3$ 0.76
$NaNO_3$ 0.41
KNO_3 0.0078
U235 0.61 %
(TK-H1)
HAX
FLOW 940
TEMP 30°C
SpG 0.810
TBP 30 VOL % IN INERT DILUENT
HNO_3 0.01M
(#1 ORGANIC SYSTEM)
HA COL T-H2
HAP
FLOW 1176
TEMP AMB
SpG 0.871
Th 0.168M
HNO_3 0.11
U 235 0.057%
HAO
FLOW 123
TEMP 30°C
SpG 0.850
Th 0.114
HNO_3 0.07
U 235 0.028%
HAX-HNO_3
FLOW 26
TEMP 30°C
SpG 1.37
HNO_3 13.0M
(REC. ACID)
HAW
FLOW 840
TEMP AMB
SpG 1.08
Th % 0.28
U% < 0.0003
HNO_3 1.58
KF 0.025
$Al(NO_3)_3$ 0.11
$NaNO_3$ 0.17
KNO_3 0.0033
H_3PO_4 0.0054
FeSA 0.0054
(TO TK-F7)
2BW
3BW
1BXF
FLOW 1240
TEMP 30°C
SpG 0.868
Th 0.140M
HNO_3 0.104
U 235 0.054%
(TK-J3)
1BX COL T-J6
1BX
FLOW 630
TEMP 30°C
SpG 1.00
HNO_3 0.20M
1BXT
FLOW 645
TEMP AMB
SpG 1.12
Th 0.270M
HNO_3 0.546
U 0.0096%
1BS
FLOW 220
TEMP 30°C
SpG 0.810
TBP 30 VOL % IN INERT DILUENT
HNO_3 0.01M
FROM TK G5
1BS COL T-J7
1BSU
FLOW 223
TEMP AMB
SpG 0.850
Th 0.11M
HNO_3 0.07
U 0.028%
TK-J8
1BU
FLOW 1225
TEMP AMB
SpG 0.811
HNO_3 0.026M
Th 0.009%
U-235 0.050 %
1BT
FLOW 645
TEMP AMB
SpG 1.10
Th 0.231 M
HNO_3 0.325
U-235 TRACE
IC COL T-H3
ICX
FLOW 890
TEMP 30°C
SpG 1.00
HNO_3 0.01M
ICW
FLOW 1225
TEMP AMB
SpG 0.810
HNO_3 0.001M
(TO TK G1)
ICU
FLOW 890
TEMP AMB
SpG 1.00
HNO_3 0.042 M
Th 0.012%
U-233 0.069%
1UD
FLOW 690
TEMP 50°C
SpG 1.00
HNO_3 0.001M
(TO CRIB)
ICU CONC R E-H4
1UC
FLOW 150
TEMP 101°C
SpG 1.01
HNO_3 0.302M
Th 0.065%
U 0.470%
(TO TK-J1)
1TD
FLOW 485
TEMP 50°C
SpG 1.00
HNO_3 0.001M
(TO CRIB)
1BT CONC R E-J8
1TC
FLOW 190
TEMP 106°C
SpG 1.35
Th 0.786 M
HNO_3 1.10
2DF-Fe
FLOW 0.21
TEMP AMB
SpG 1.34
FeSA 2.0M
(TK-217)
2DF
FLOW 190
TEMP 30°C
SpG 1.35
Th 0.786M
HNO_3 1.10
FeSA 0.0022
(TK-K1)
2DIS
FLOW 9.2
TEMP AMB
SpG 1.40
H_3PO_4 * 0.50 M
HNO_3 12.2
(TK 218, 219)
* USED ONLY WHEN REQ'D FOR P_a DF
SECOND THORIUM CYCLE
2D COL T-K2
2DX
FLOW 1150
TEMP 30°C
SpG 0.814
TBP 30 VOL % IN INERT DILUENT
HNO_3 0.12M
(#2 ORGANIC SYS)
2DS(H_2O)
FLOW 1042
TEMP 30°C
SpG 1.00
2DT
FLOW 1165
TEMP AMB
SpG 0.862
Th 0.128M
HNO_3 0.110
2DW
FLOW 292
TEMP AMB
SpG 1.03
HNO_3 1.15M
PO_4* 0.016
Fe 0.0014
Th% 0.24
(TO TK-F10)
2E COL T-K3
2EX
FLOW 510
TEMP 30°C
SpG 1.00
HNO_3 0.01M
2EW
FLOW 1150
TEMP AMB
SpG 0.810
HNO_3 0.001M
Th % <0.2
(TO TK-R1)
2ET
FLOW 527
TEMP AMB
SpG 1.12
Th 0.280M
HNO_3 0.25
2TD
FLOW 475
TEMP 50°C
SpG 1.00
HNO_3 0.01M
(TO CRIB)
2ET CONC'R. E-K4
TO TNT TRAILER
TNT PROD.
FLOW 87
TEMP 35°C
SpG 1.68
Th 1.70M
HNO_3 1.46
(TK-K6)
2TC
FLOW 80
TEMP 107°C
SpG 1.76
Th 1.85M
HNO_3 1.59
(TK-K5)
C-LASSIFIED - UP-DATED 4
GENERAL UP-DATE 2
CAMPAIGN UP DATED FOR THORIUM HT CLASSIFIED - GENERAL 1
DESCRIPTION
REVISIONS
DRAWING STATUS
SCALE NONE
U S ATOMIC ENERGY COMMISSION
RICHLAND OPERATIONS OFFICE
Atlantic Richfield Hanford Company
Richland Washington 99352
PUREX THORIUM PROCESS
CO-DECONTAMINATION & PARTITION CYCLE
SECOND THORIUM CYCLE
FIGURE II-C
202A
7000
SECRET
SK-2-21905 3

SECRET
DECLASSIFIED



2AF HNO₃
FLOW 29 2
TEMP AMB
SpG 1 35
HNO₃ 12 2M

2AF
FLOW 140
TEMP 40°C
SpG 1.01
HNO₃ 0 284M
U %L 0 436
Th %L 0 079
(TK J21)

2AX
FLOW 50 0
TEMP 30°C
SpG 0.810
TBP 30 VOLUME % IN INERT DILUENT
HNO₃ 0.01M
(#1 ORGANIC SYSTEM)

2AS
FLOW 17 5
TEMP AMB
SpG 1 01
HNO₃ 0 30M
FeSA 0 01
(TK 220-221)

2AU
FLOW 50.0
TEMP AMB
SpG 0.815
HNO₃ 0.056M
U, %L 1 22
Th %L 0.005

2AW
FLOW 187
TEMP AMB
SpG 1 06
HNO₃ 2 14M
FeSA 0.001
Th, %L 0.058
U, %L <0.0004
(TO TK-F10)

2BX
FLOW 40
TEMP AMB
SpG 1 00
HNO₃ 0 01M
(TK 224,225)

2BW
FLOW 53.0
TEMP AMB
SpG 0.810
HNO₃ 0 001M
(TO TK J3)

2BU
FLOW 40
TEMP AMB
SpG 1 00
HNO₃ 0 079M
U %L 1 52
Th %L 0 0062

3AF-HNO₃
FLOW 13 1
TEMP AMB
SpG 1 35
HNO₃ 12 2M

3AF
FLOW 42 0
TEMP 30°C
SpG 1 00
U, %L 1 45
HNO₃ 0.075M
Th, %L 0 0058
(TK-J5)

3AX
FLOW 14 3
TEMP 30°C
SpG 0.810
TBP 30 VOLUME % IN INERT DILUENT
HNO₃ 0.01M
(#1 ORGANIC SYSTEM)

3AS
FLOW 6 9
TEMP AMB
SpG 1 00
HNO₃ 0.1M
(TK-211, 212)

3AU
FLOW 14 3
TEMP AMB
SpG 0.816
U, %L 4 27
HNO₃ 0 01M
Th, %L <0 001

3AW
FLOW 62 0
TEMP AMB
SpG 1 08
HNO₃ 2 64M
Th %L <0 004
U %L <0.0003
(TO TK-F10)

3BX
FLOW 10 0
TEMP AMB
SpG 1 00
HNO₃ 0 01M
(TK-213 214)

3BW
FLOW 14 3
TEMP AMB
SpG 0 810
HNO₃ TRACE
(TO TK-J3)

3BU
FLOW 10 6
TEMP AMB
SpG 1 01
U, %L 6 08
HNO₃ 0.024M
Th, %L <0 001
(TK-N1)

3UF
FLOW 10 0
TEMP 30°C
SpG 1 01
U %L 6 08
HNO₃ 0.024M
Th %L <0 0002

3UD
FLOW 9.8
TEMP 50°C
SpG 1 00
HNO₃ 0.015M
(TO TK J1)

3UC
FLOW 0.174
TEMP 35°C
SpG 1.46
HNO₃ 0 54M
U, %L 350
Th %L <0 01
TK-N7

RESTRICTED DATA

M							GENERAL UP DATE	4
1			GPS				DECLASSIFIED GENERAL UP-DATE	3
							GENERAL UP-DATE	2
1			GPS 7/24/69				THORIUM CAMPAIGN UP DATED FOR 1970 RE CLASSIFIED - GENERAL	1

REVISIONS
DRAWING STATUS
REFERENCE DRAWINGS

R.W. BATTIE
SCALE NONE

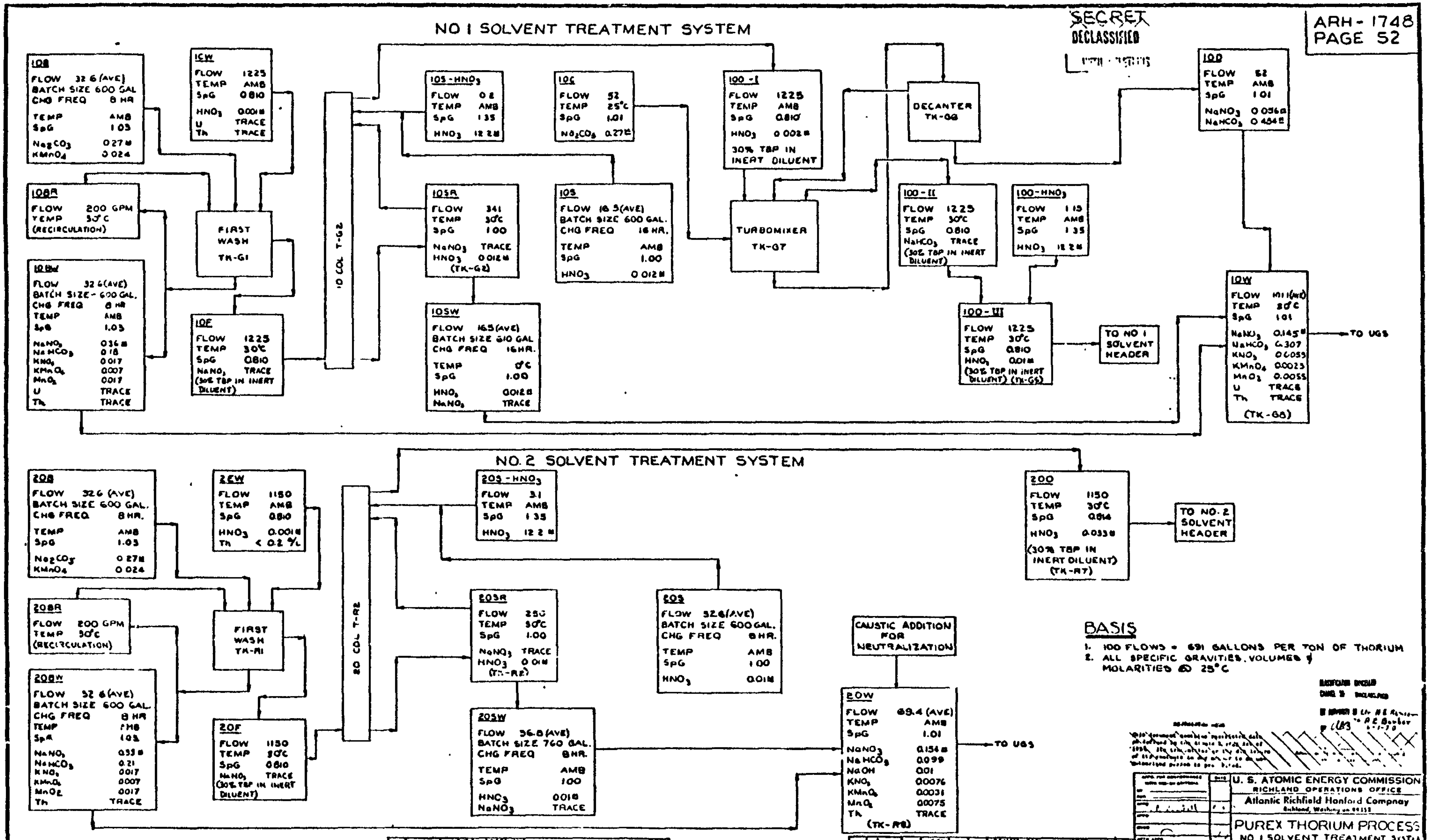
U S ATOMIC ENERGY COMMISSION
RICHLAND OPERATIONS OFFICE
Atlantic Richfield Hanford Company
Richland Washington 99352

PURSEX THORIUM PROCESS
SECOND URANIUM CYCLE
THIRD URANIUM CYCLE

FIGURE II-D

EOL-A 7000
SK-2-21905 4

SECRET
DECLASSIFIED



BASIS

1. 100 FLOWS = 691 GALLONS PER TON OF THORIUM
2. ALL SPECIFIC GRAVITIES, VOLUMES & MOLARITIES @ 25°C

					GENERAL UP-DATE	4
1					GENERAL UP DATE DECLASSIFIED	3
					GENERAL UP-DATE	2
			7/15/69		CAMPAIGN UP-DATED FOR 1970 THORIUM RE-CLASSIFIED - GENERAL	1

REVISIONS DESCRIPTION
REFERENCE DRAWINGS
REVISIONS
DRAWING STATUS

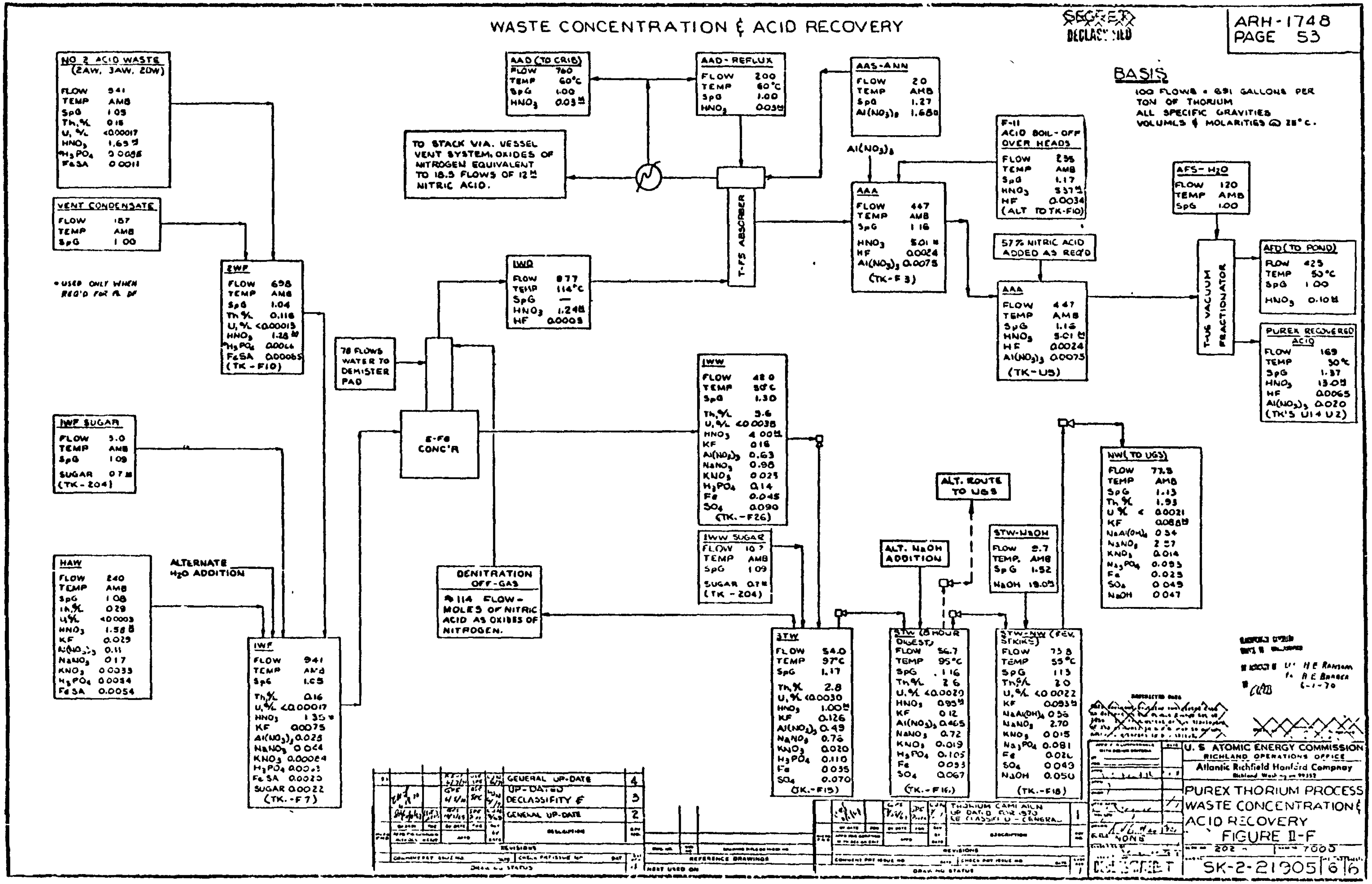
U. S. ATOMIC ENERGY COMMISSION
RICHLAND OPERATIONS OFFICE
Atlantic Richfield Hanford Company
Richland, Washington 99352

PUREX THORIUM PROCESS
NO 1 SOLVENT TREATMENT SYSTEM
NO 2 SOLVENT TREATMENT SYSTEM

FIGURE II-E

SCALE NONE
202-A 7000
SK-2-21905 5 6

WASTE CONCENTRATION & ACID RECOVERY



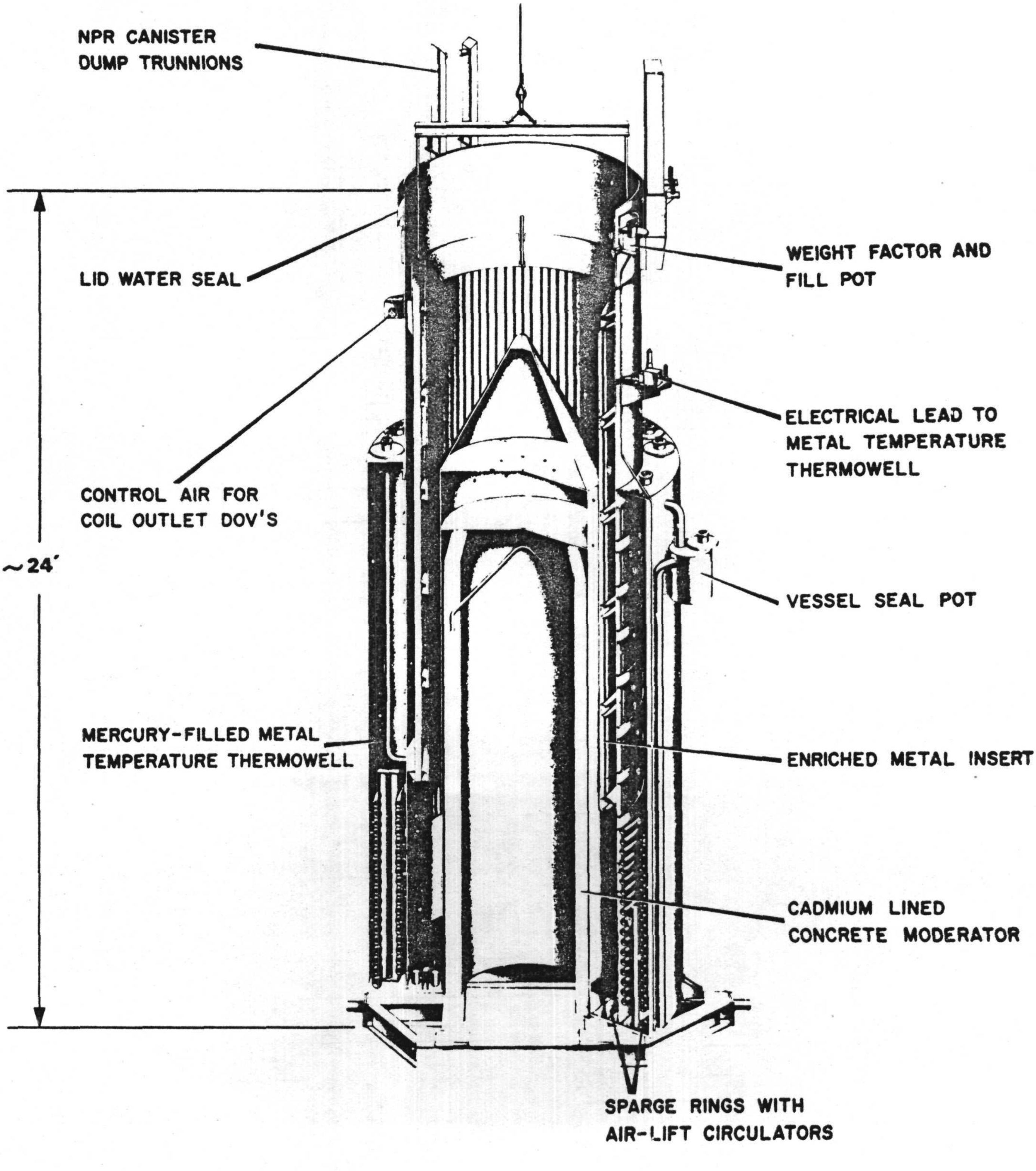
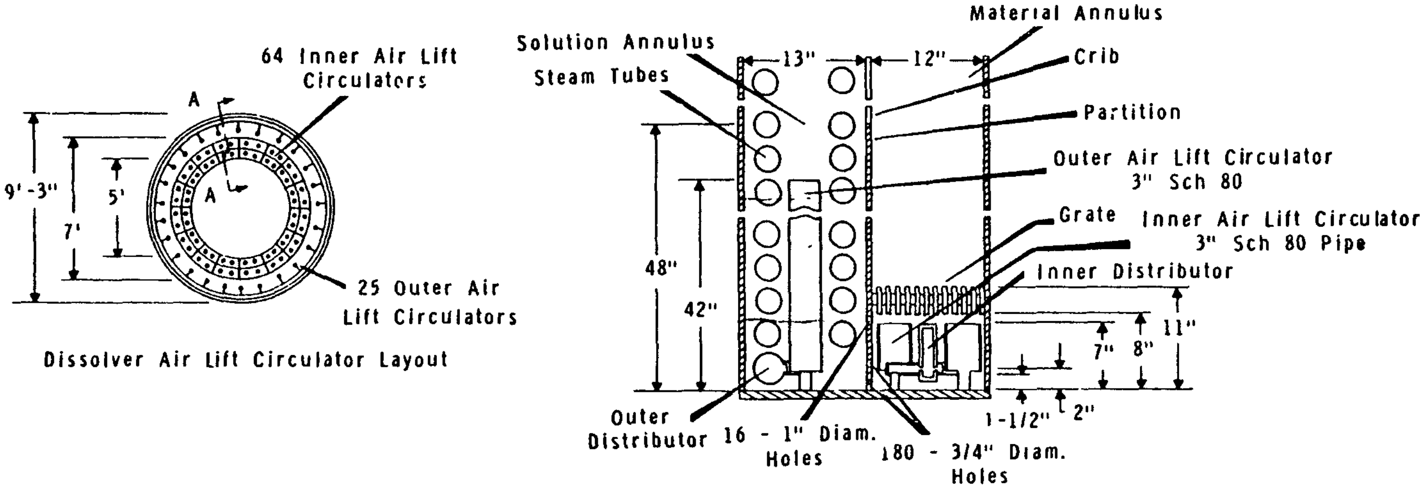


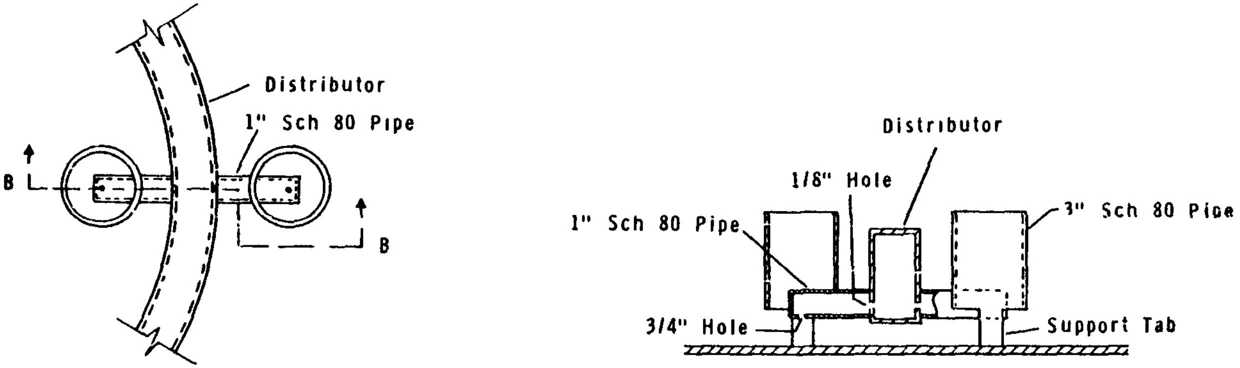
FIGURE 8

PUREX ANNULAR DISSOLVER



Dissolver Air Lift Circulator Layout

Section A-A



Section B-B

FIGURE 9

DISSOLVER AIR LIFT CIRCULATOR

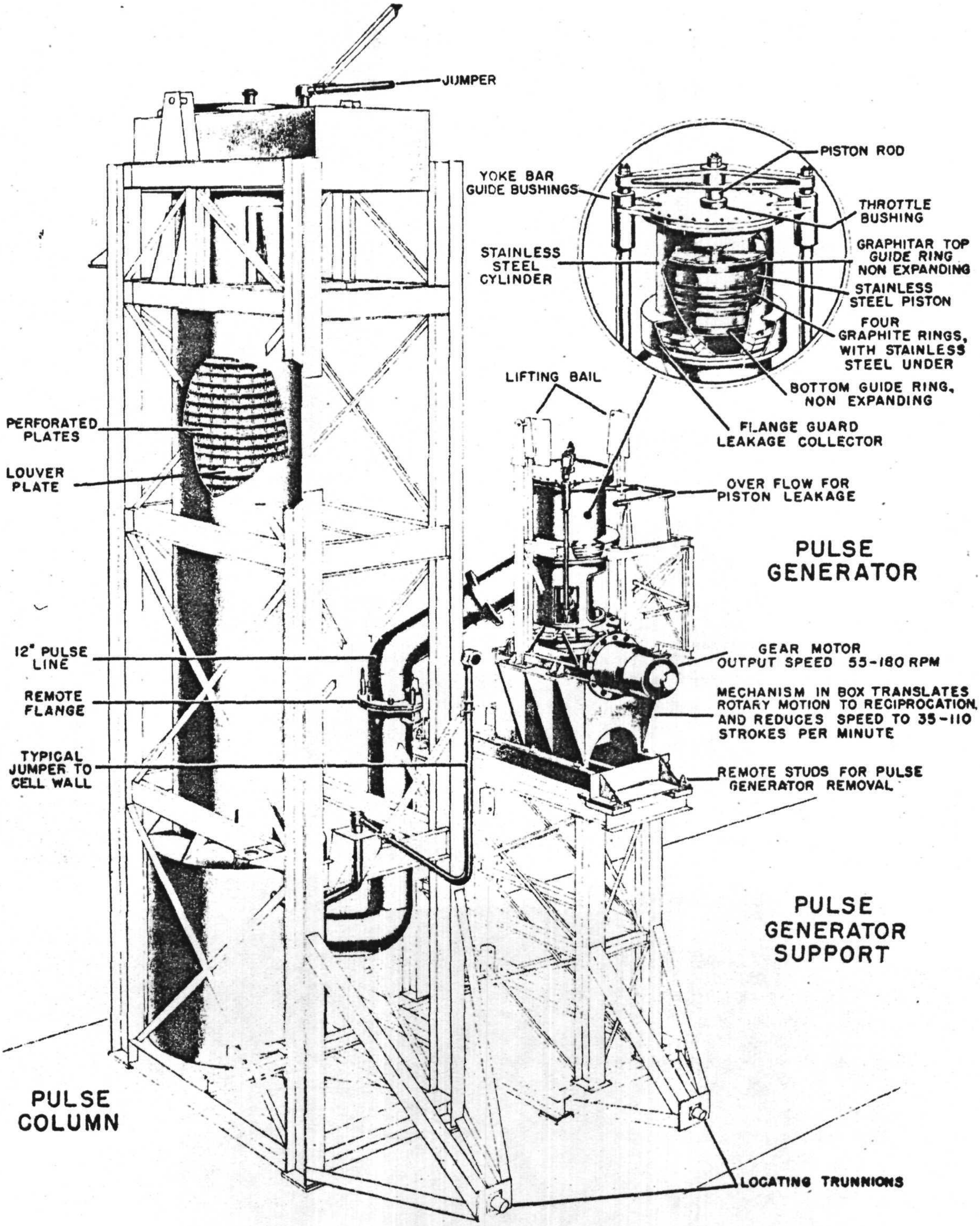


FIGURE 10

HANFORD PUREX PULSE COLUMN

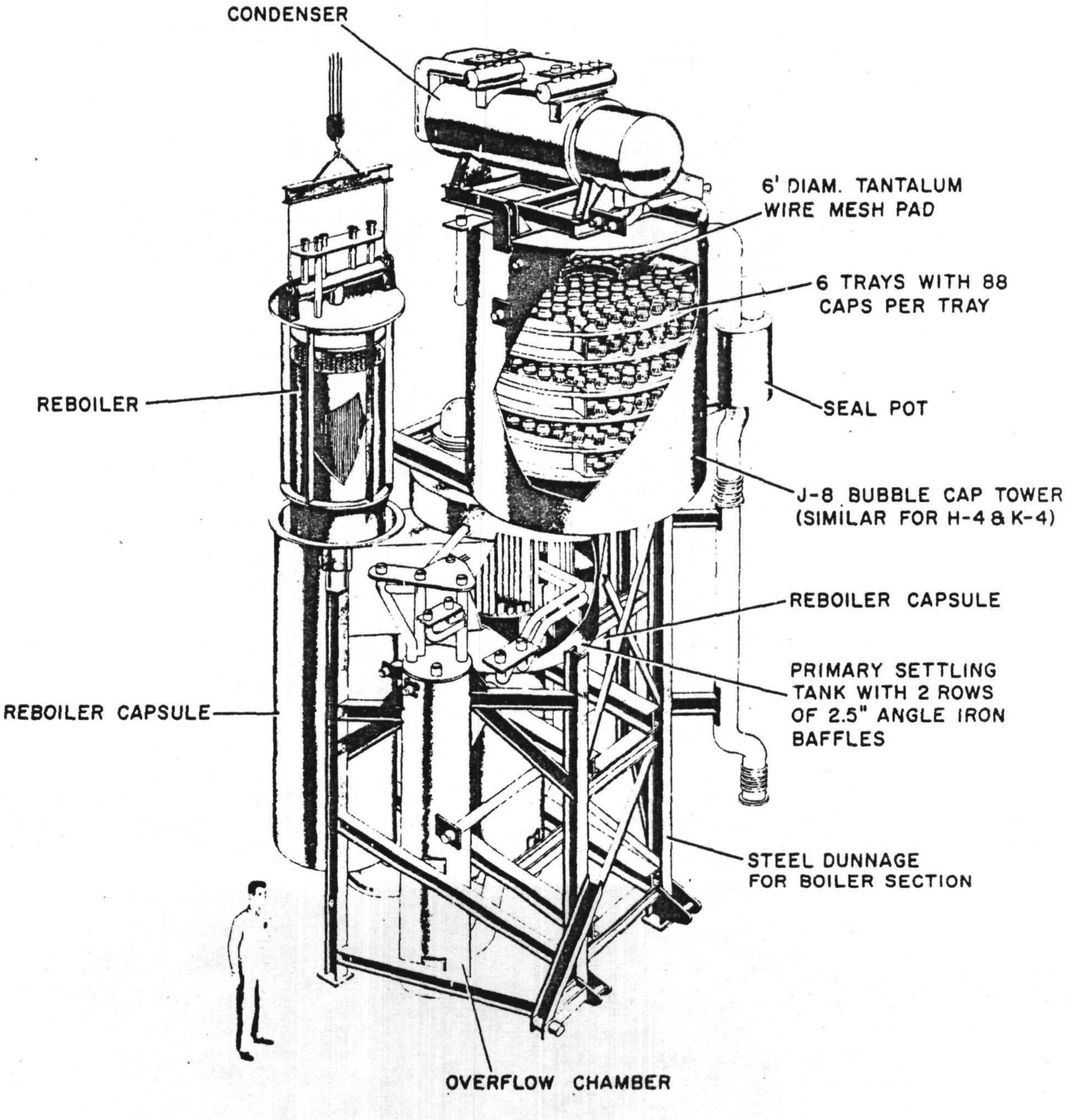
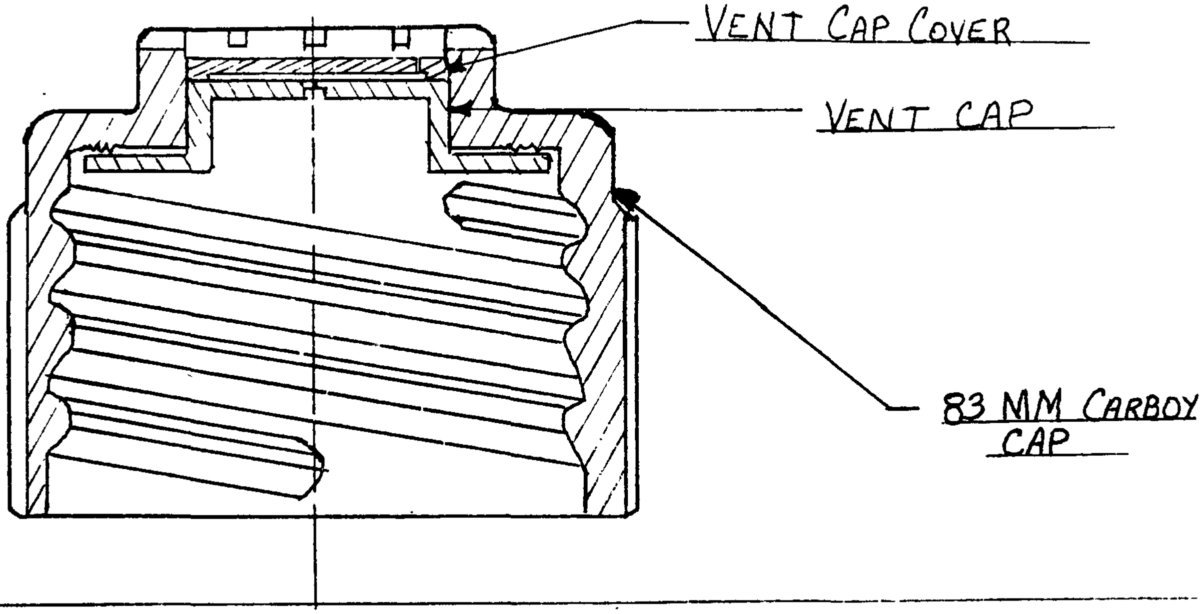
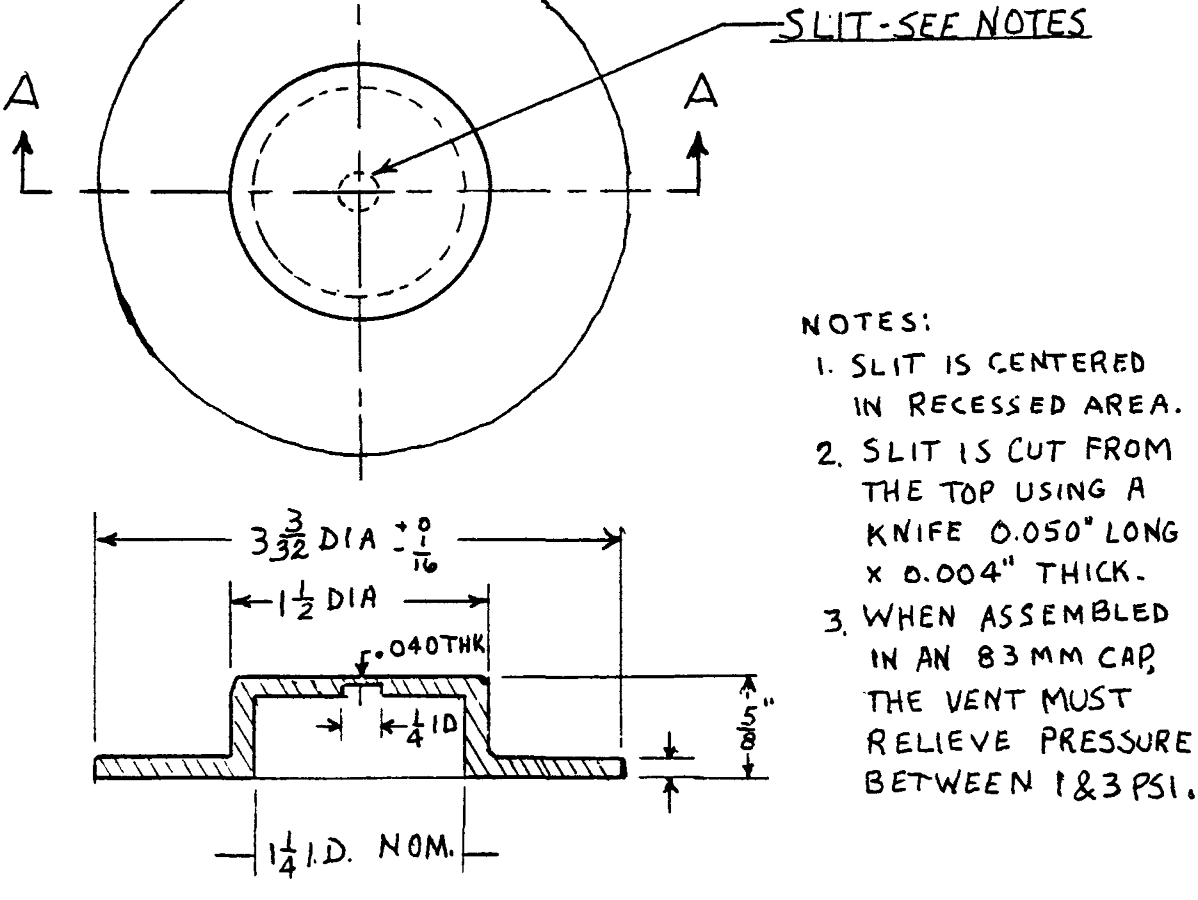


FIGURE 11

SECTIONALIZED CONCENTRATOR ASSEMBLY



VENT CAP DETAIL



NOTES:

1. SLIT IS CENTERED IN RECESSED AREA.
2. SLIT IS CUT FROM THE TOP USING A KNIFE 0.050" LONG x 0.004" THICK.
3. WHEN ASSEMBLED IN AN 83 MM CAP, THE VENT MUST RELIEVE PRESSURE BETWEEN 1 & 3 PSI.

FIGURE 12

"SLIT-VENT" CAP FOR ^{238}U PRODUCT BOTTLE

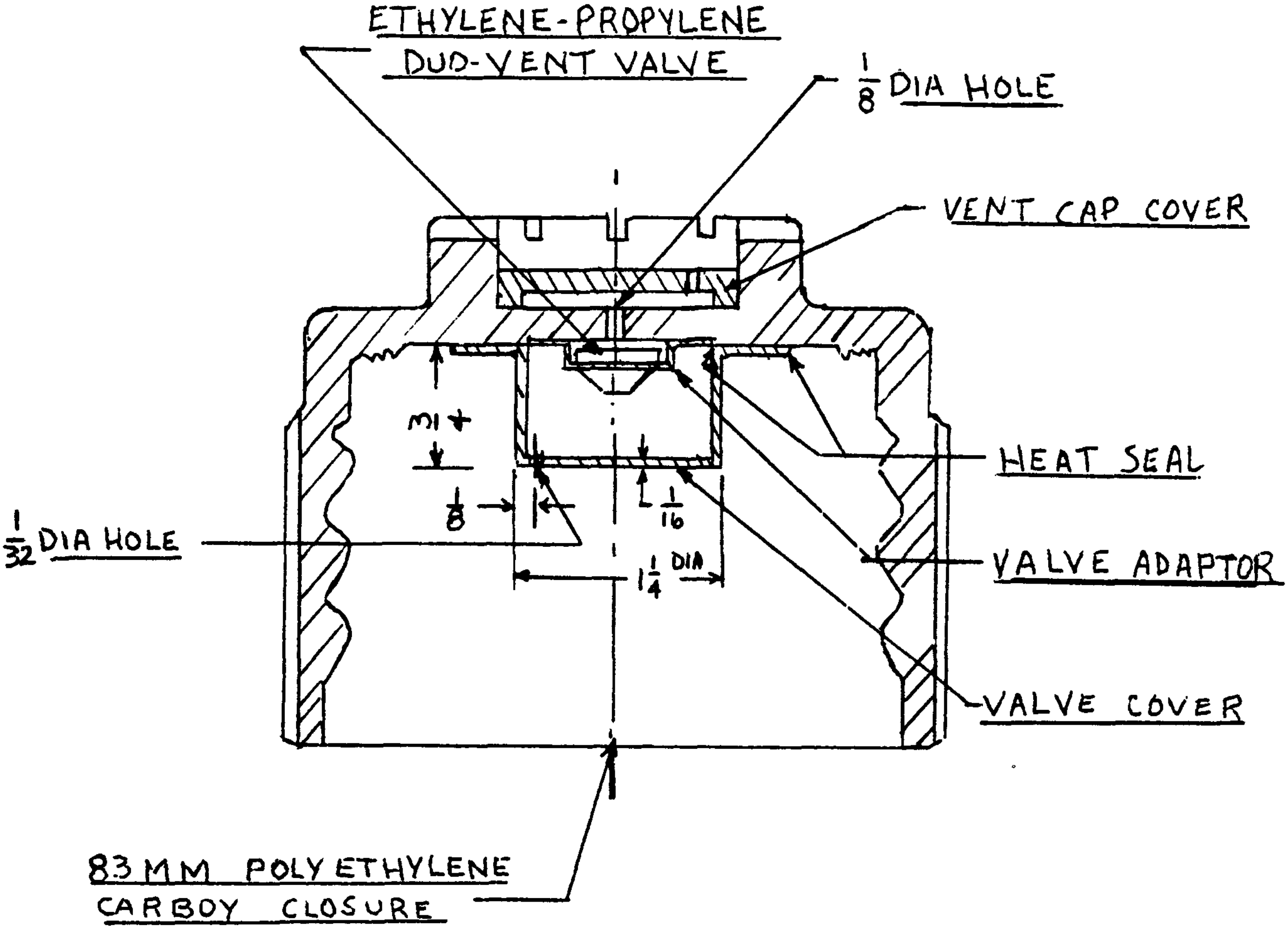


FIGURE 13

"DUO-VALVE" CAP FOR ^{233}U PRODUCT BOTTLE

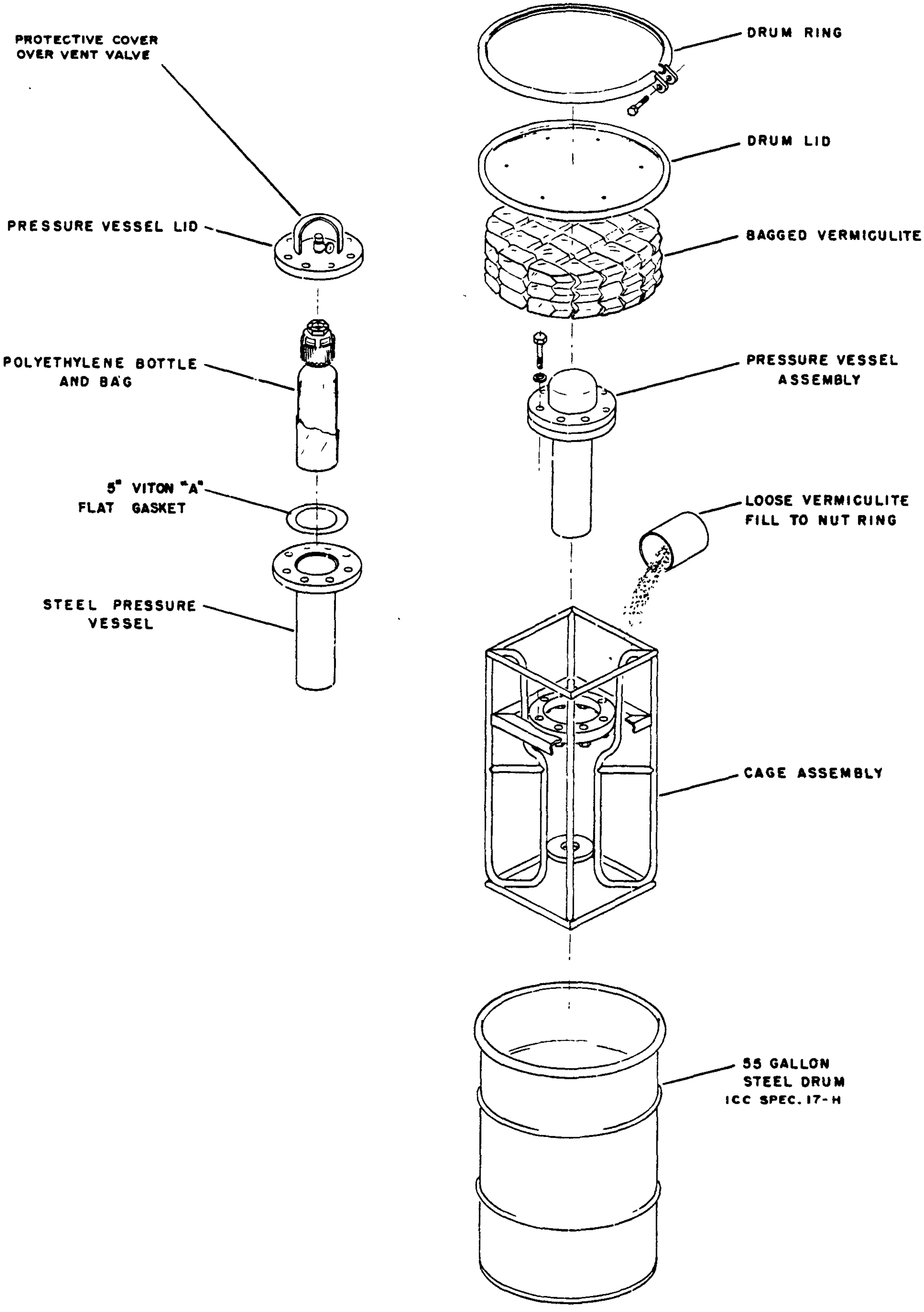


FIGURE 14

ARHCO - BAPL ^{233}U SHIPPING CONTAINER

ATLANTIC RICHFIELD HANFORD CO.
NOVEMBER 1970

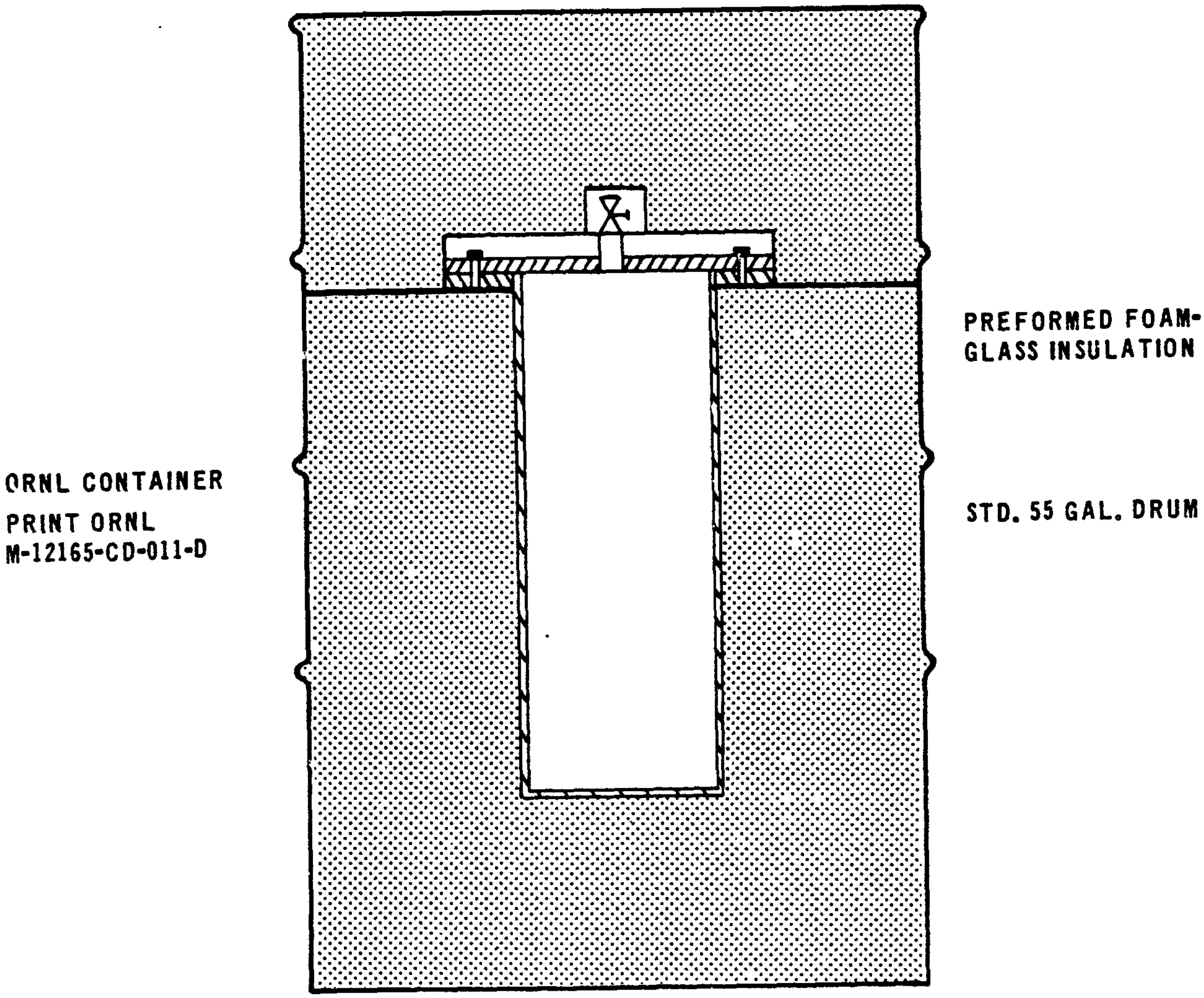


FIGURE 15

ORNL ^{233}U SHIPPING CONTAINER

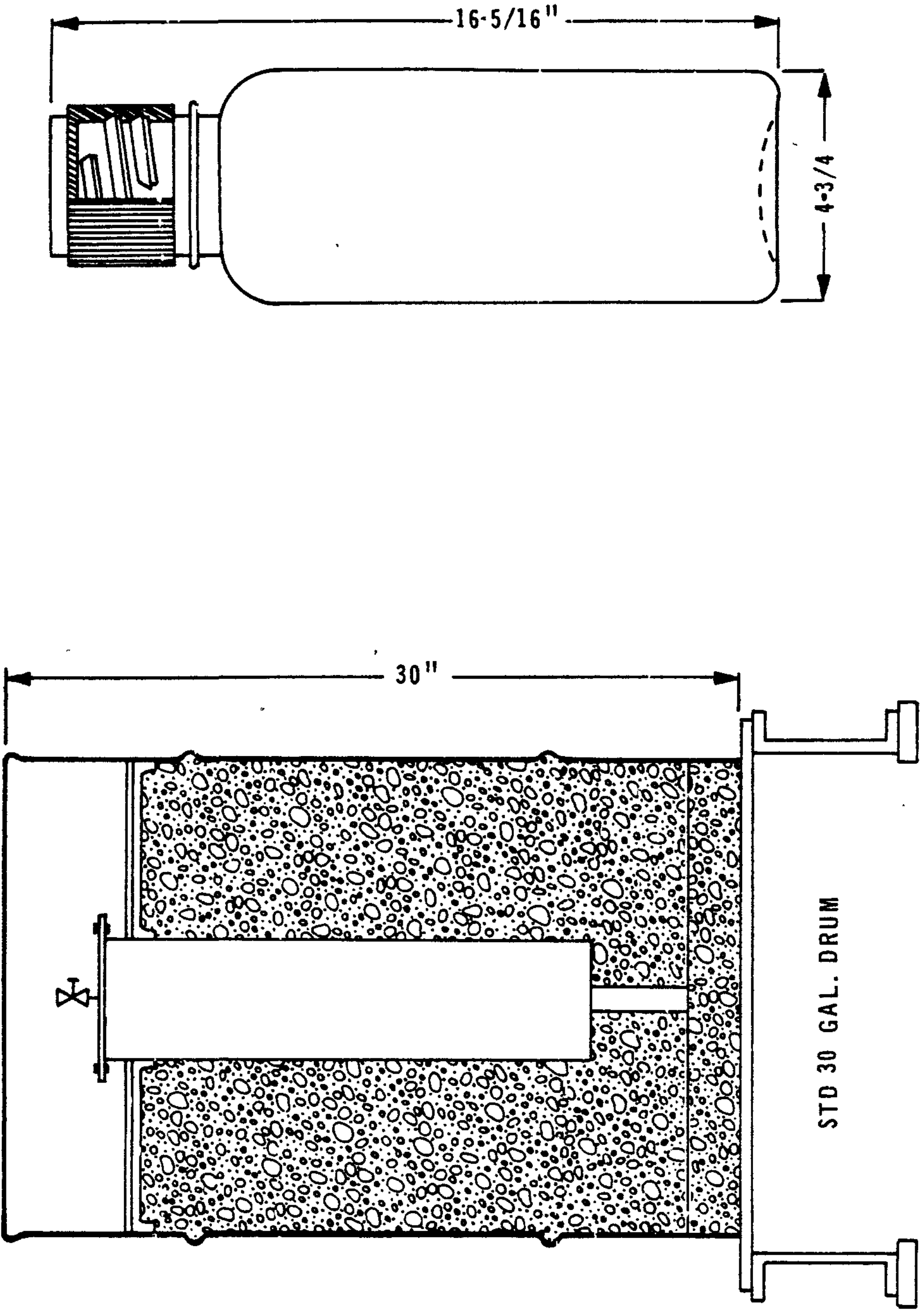


FIGURE 16

^{233}U PRODUCT BOTTLE AND STORAGE CONTAINER

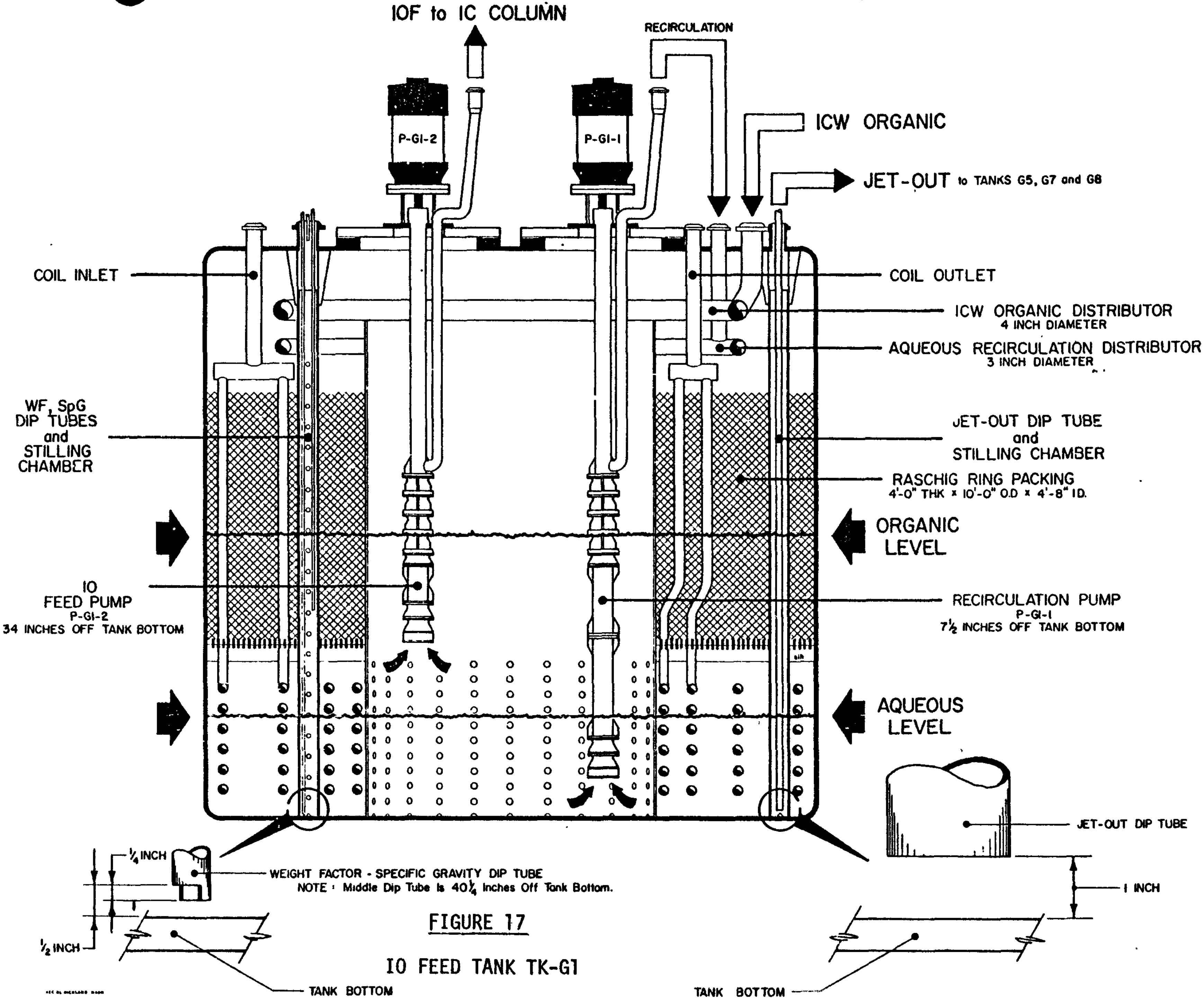
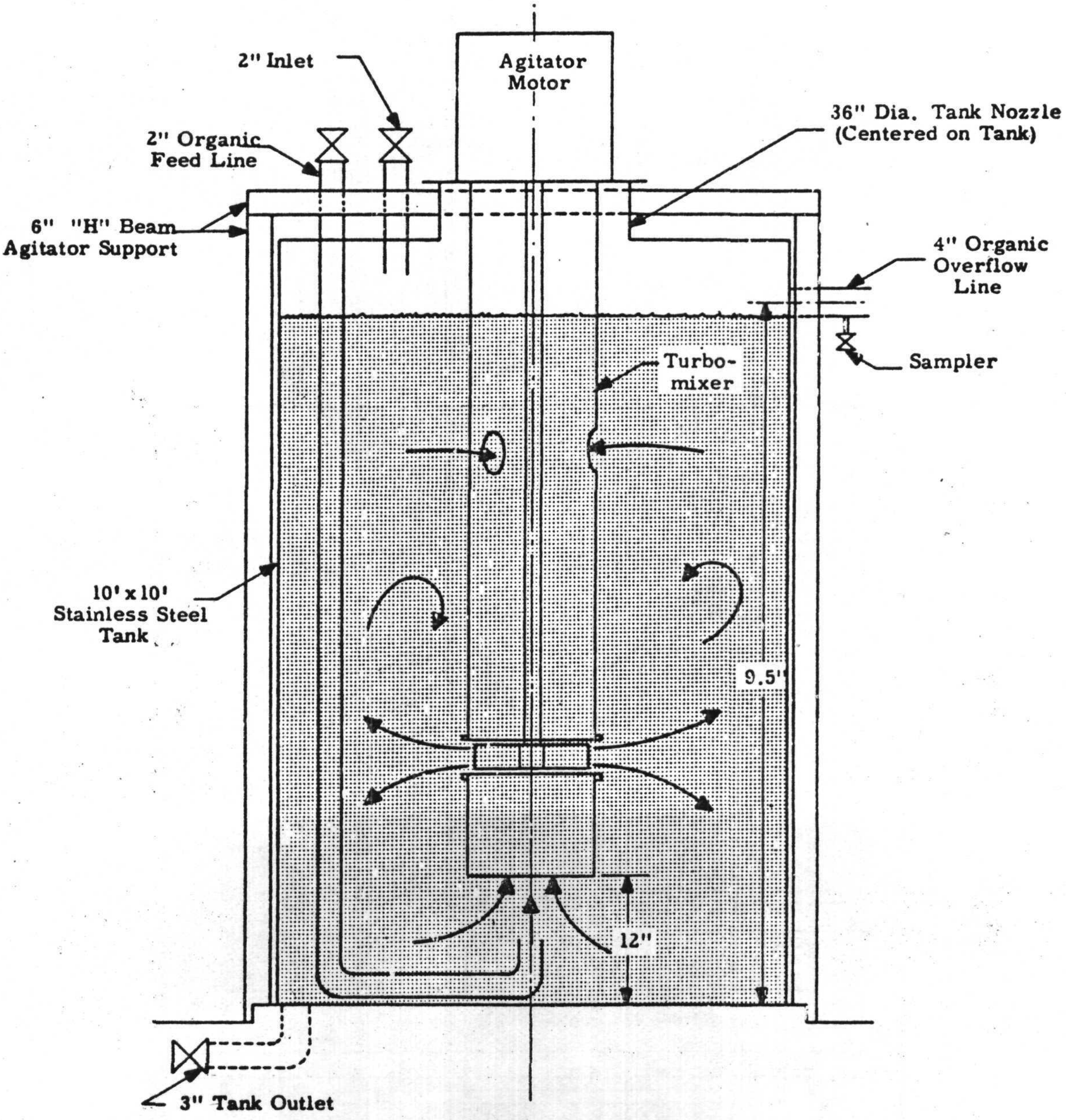


FIGURE 17

IO FEED TANK TK-G1



Arrows Indicate Flow Pattern

FIGURE 18

TURBOMIXER INSTALLATION

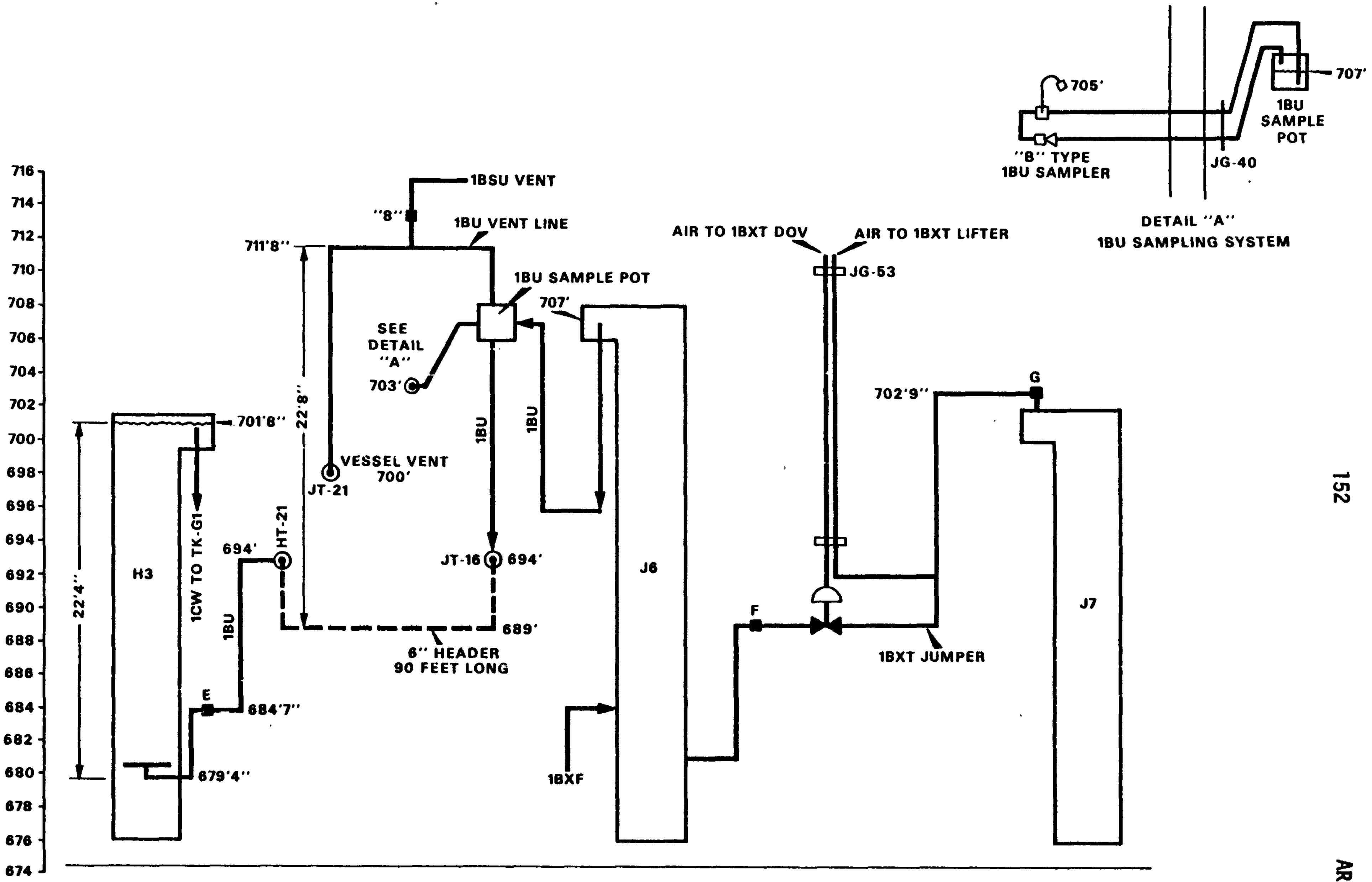


FIGURE 19

PARTITION CYCLE HYDRAULICS

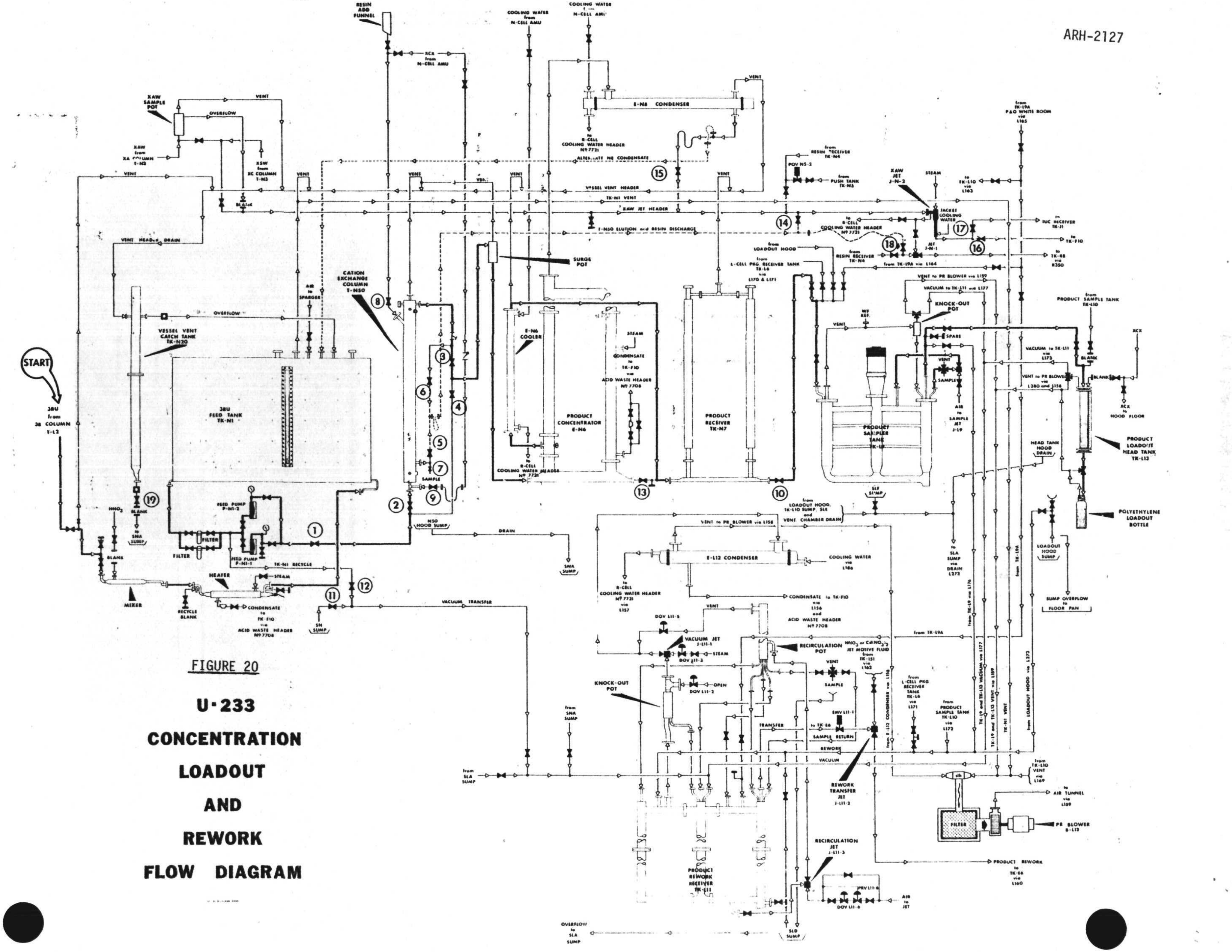


FIGURE 20

U-233

CONCENTRATION

LOADOUT

AND

REWORK

FLOW DIAGRAM

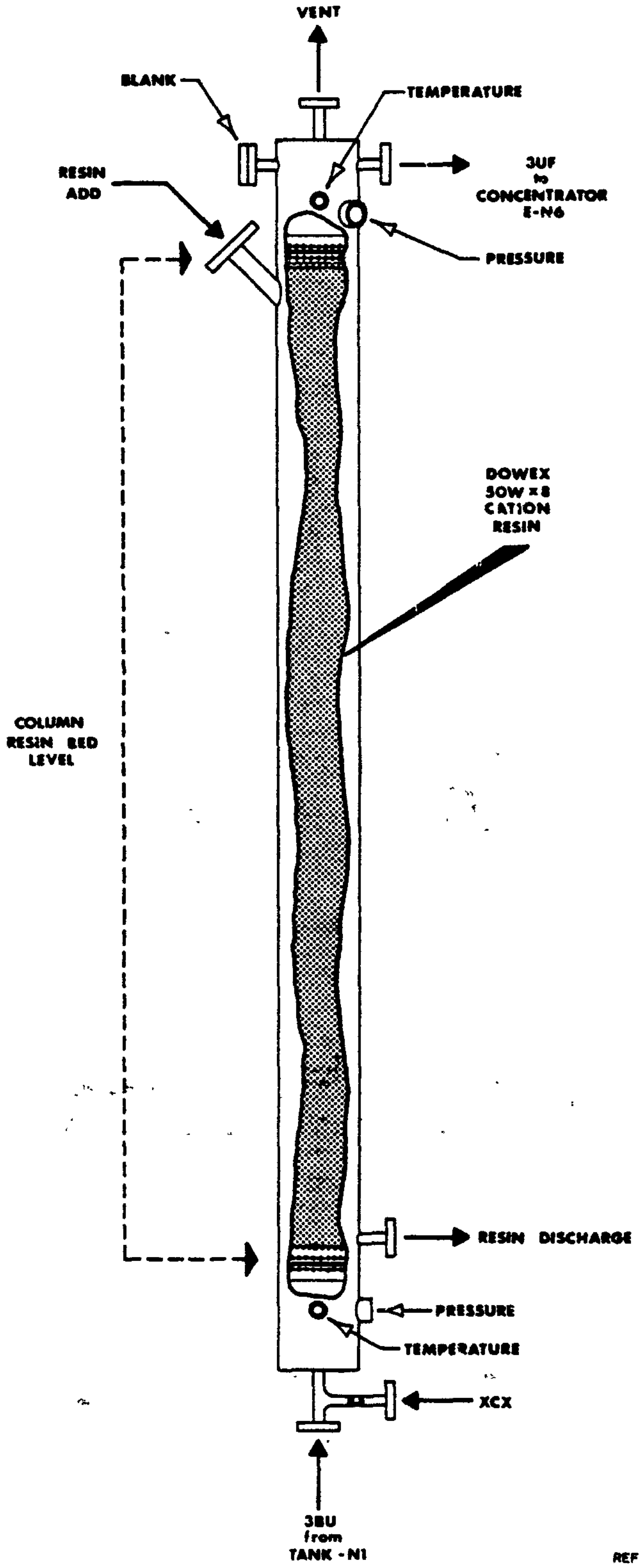


FIGURE 21

CATION EXCHANGE COLUMN

T-N50

REF DWG. CATION EXCHANGE COLUMN.... H-2-61902.....2500

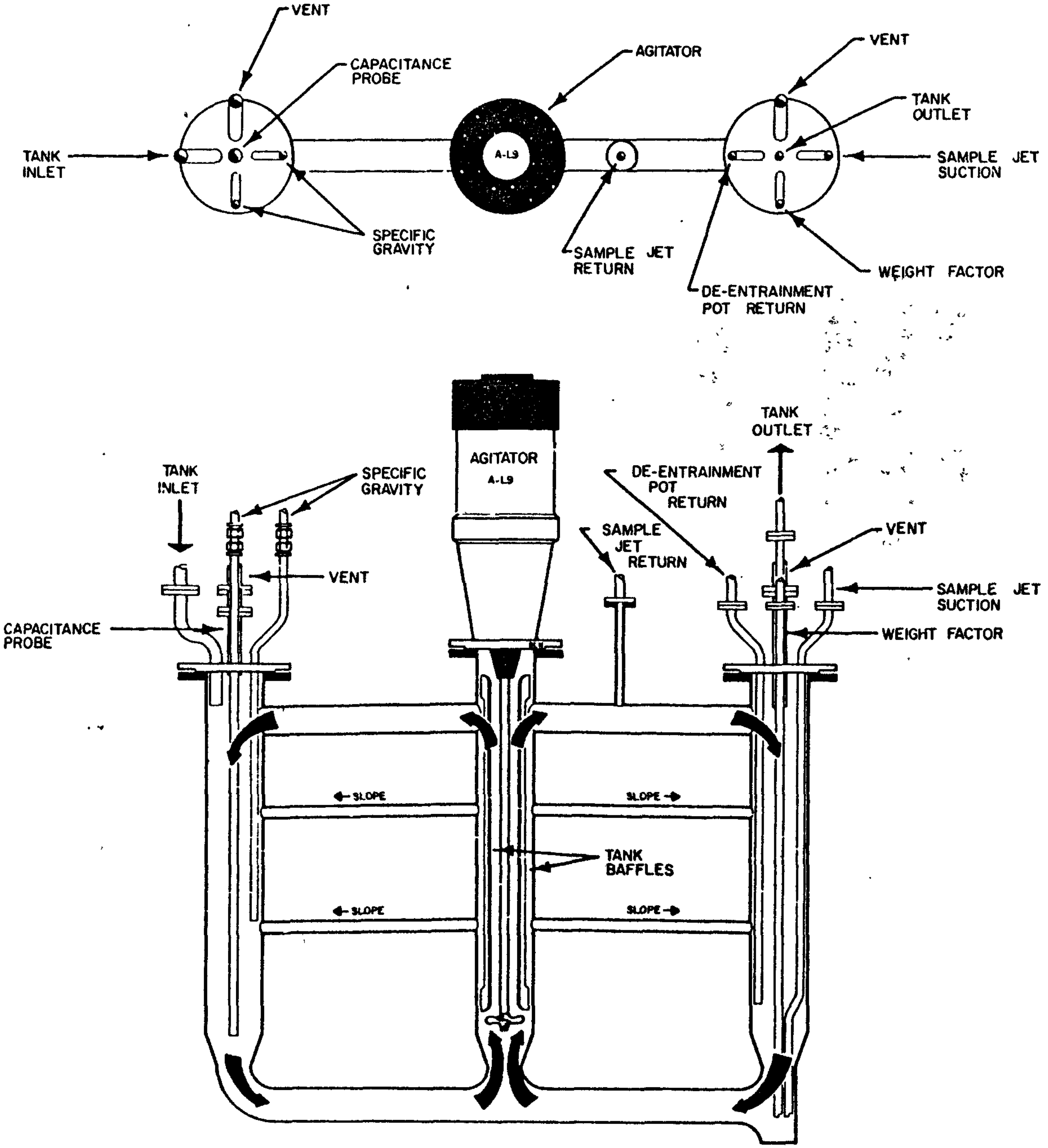


FIGURE 22

PRODUCT SAMPLE TANK

TK-L9

REF DWG			
	FLOW DIAGRAM	H-2-64695	7001
	SAMPLE TANK	H-2-64696	2500
	TANK FLANGE DETAILS	H-2-66117	2500
	PIPING ARRANGEMENT	H-2-66116	8407
	AGITATOR DETAIL	H-2-66119	2600
	CAPACITANCE LIQUID-LEVEL SYSTEM	H-2-66052	5901

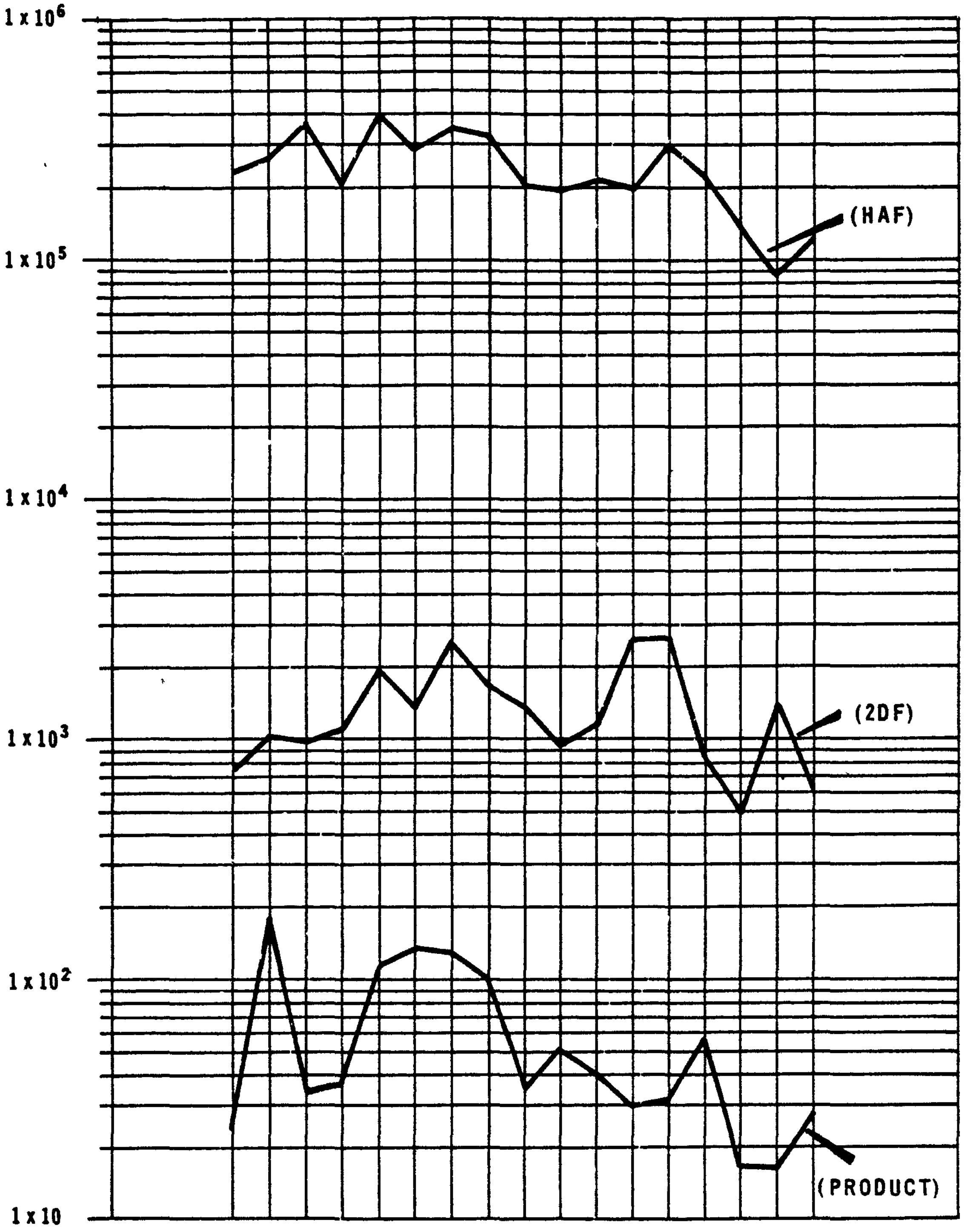


FIGURE 23

TYPICAL ^{95}Zr-$^{95}Nb/Th$ RATIOS IN PROCESS STREAMS (HAF,2DF, PRODUCT)

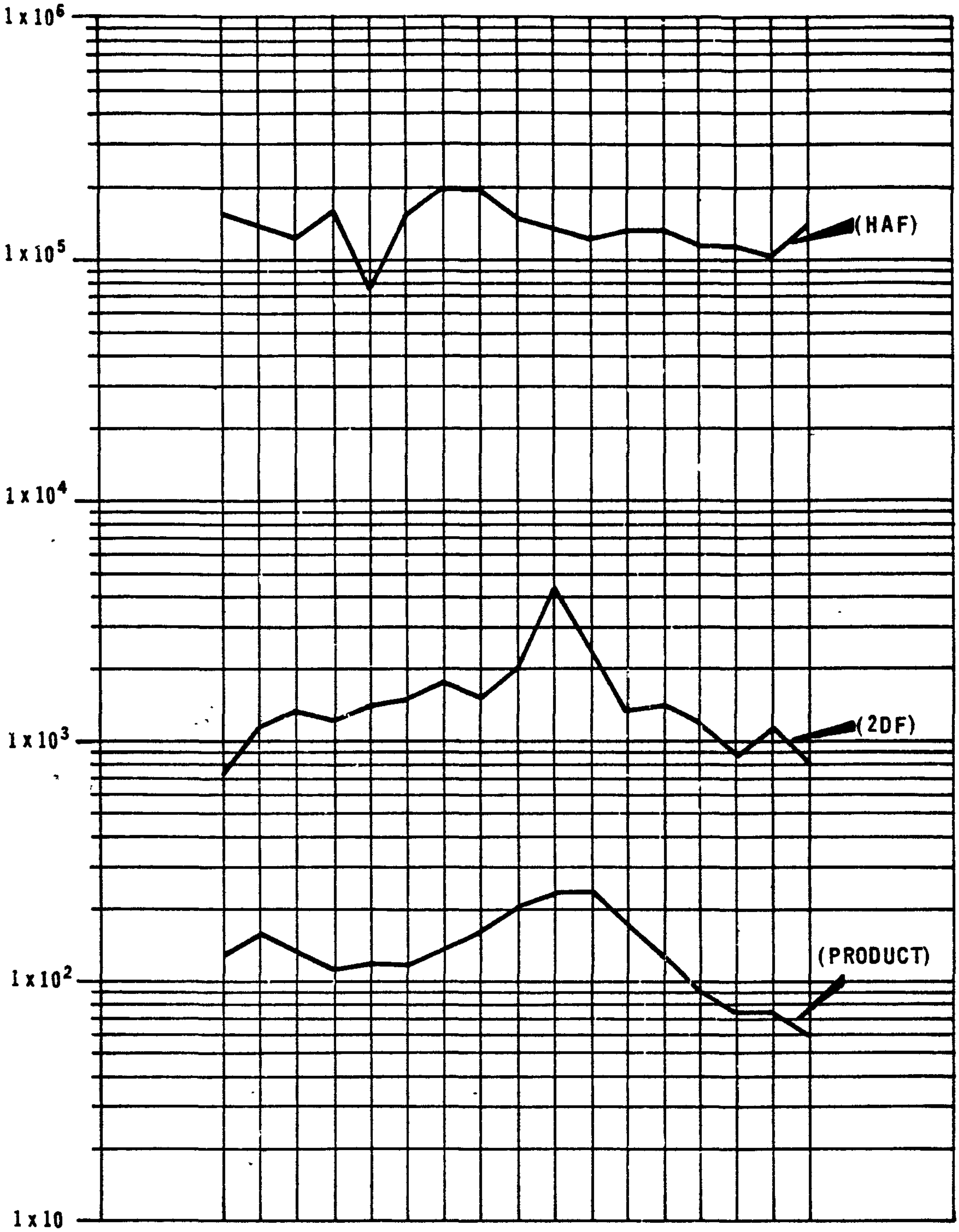


FIGURE 24

TYPICAL Ru-Rh/Th RATIOS IN PROCESS STREAMS (HAF,2DF, PRODUCT)

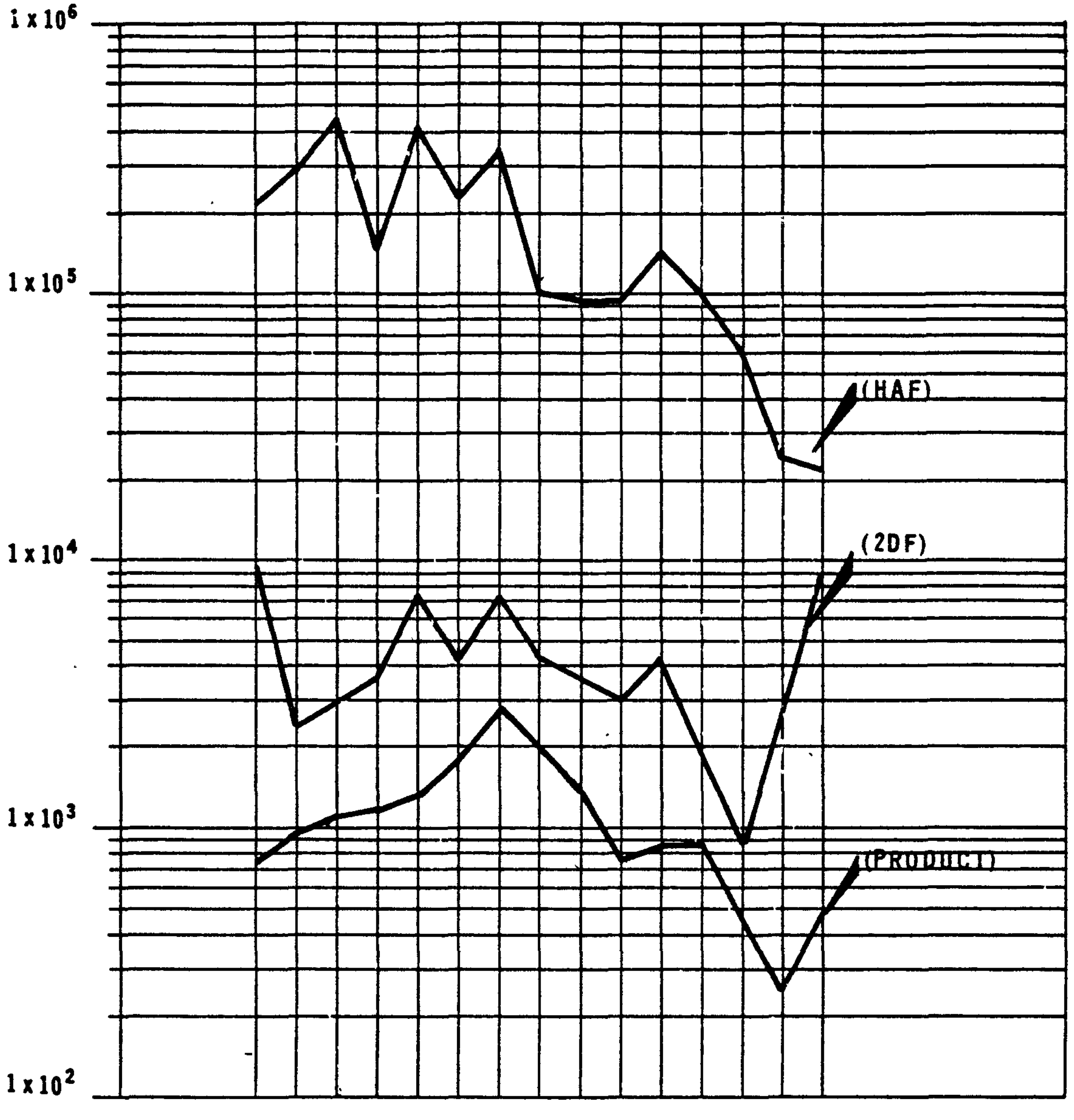


FIGURE 25

TYPICAL Pa/Th RATIOS IN PROCESS STREAMS (HAF,2DF, PRODUCT)

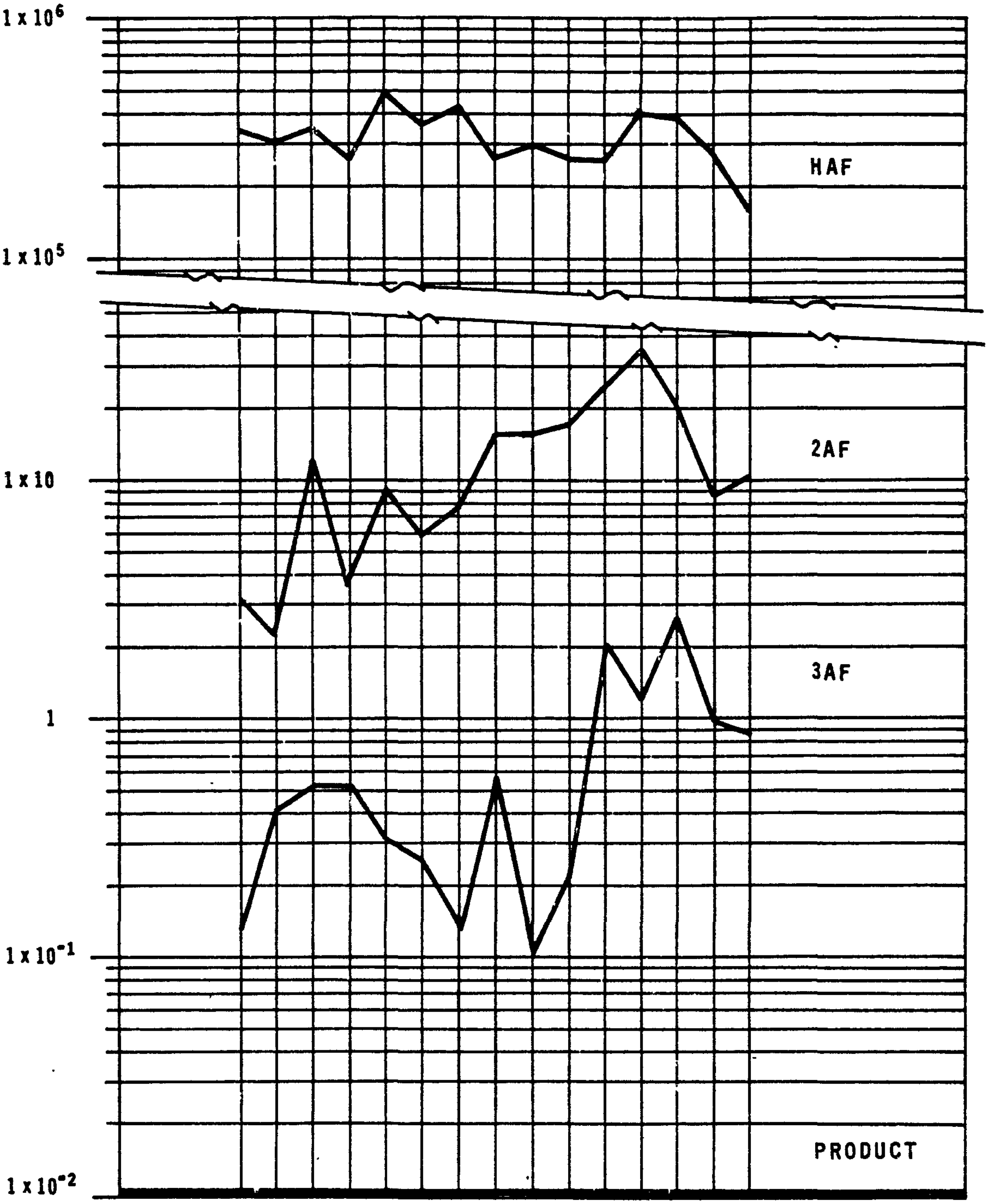


FIGURE 26

TYPICAL Zr-Nb/^{233}U RATIOS IN PROCESS STREAMS (HAF,2AF,3AF, PRODUCT)

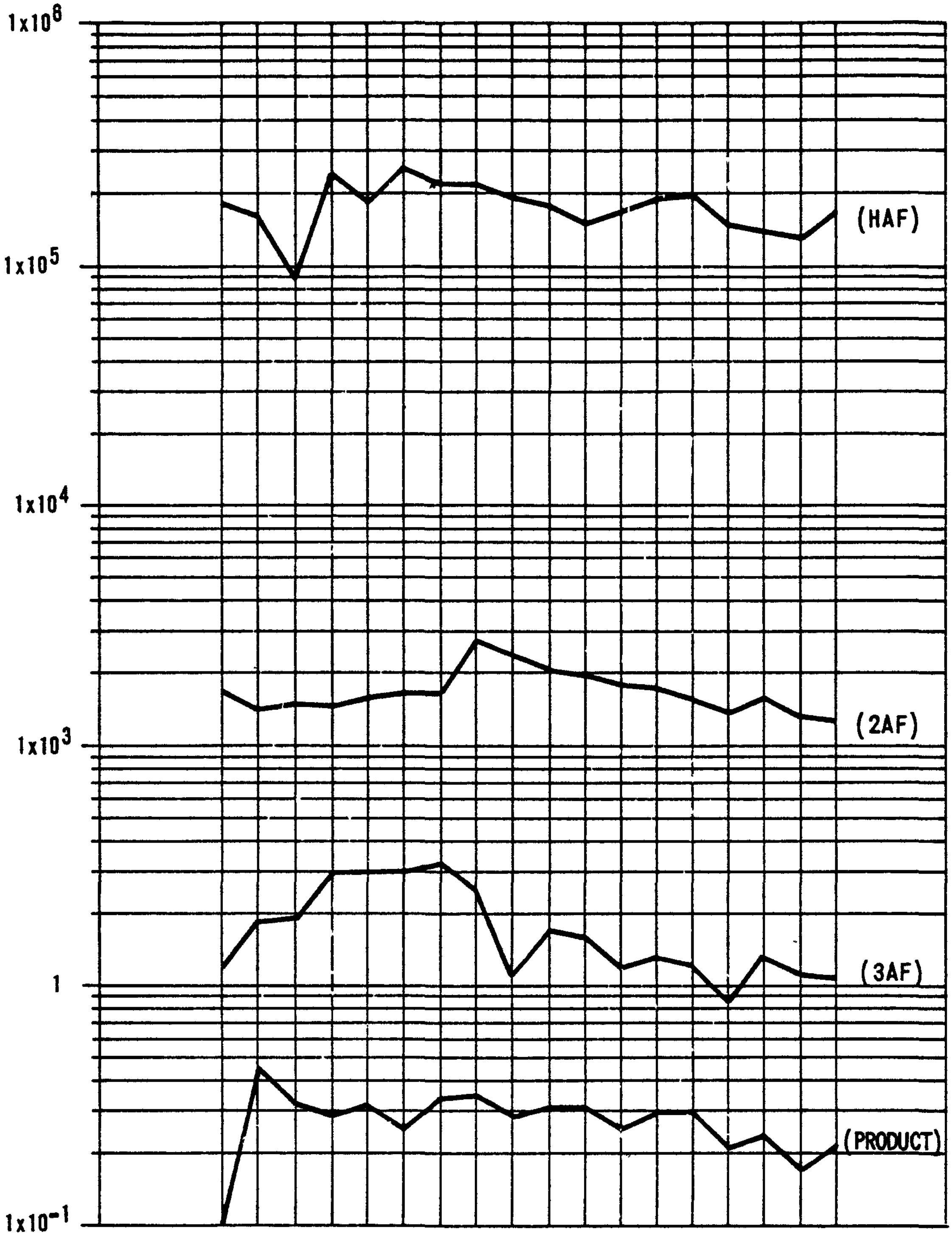


FIGURE 27

TYPICAL Ru-Rh/^{233}U RATIOS IN PROCESS STREAMS (HAF, 2AF, 3AF, PRODUCT)

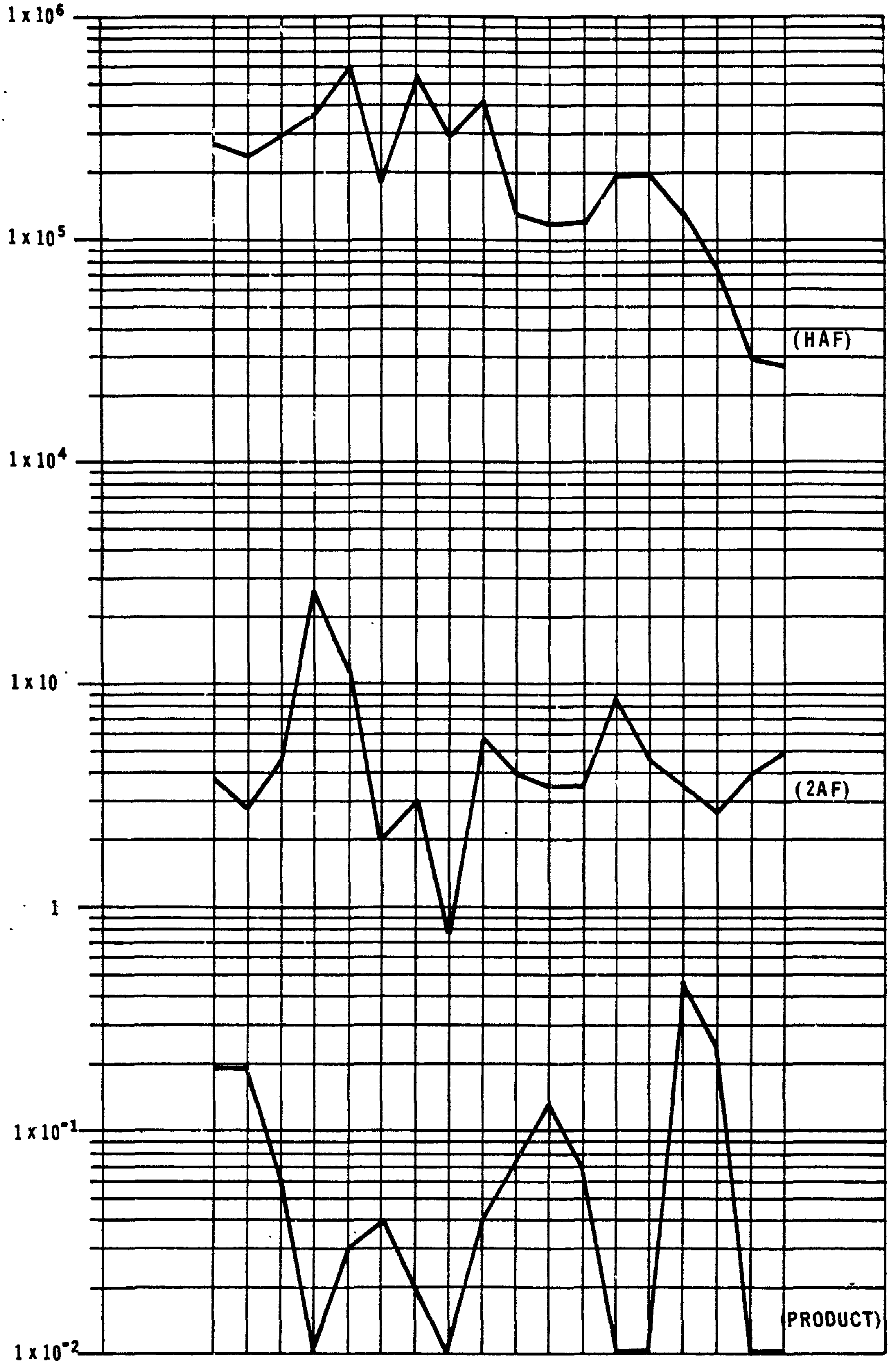


FIGURE 28

TYPICAL Pa/^{233}U RATIOS IN PROCESS STREAMS (HAF, 2AF, PRODUCT)

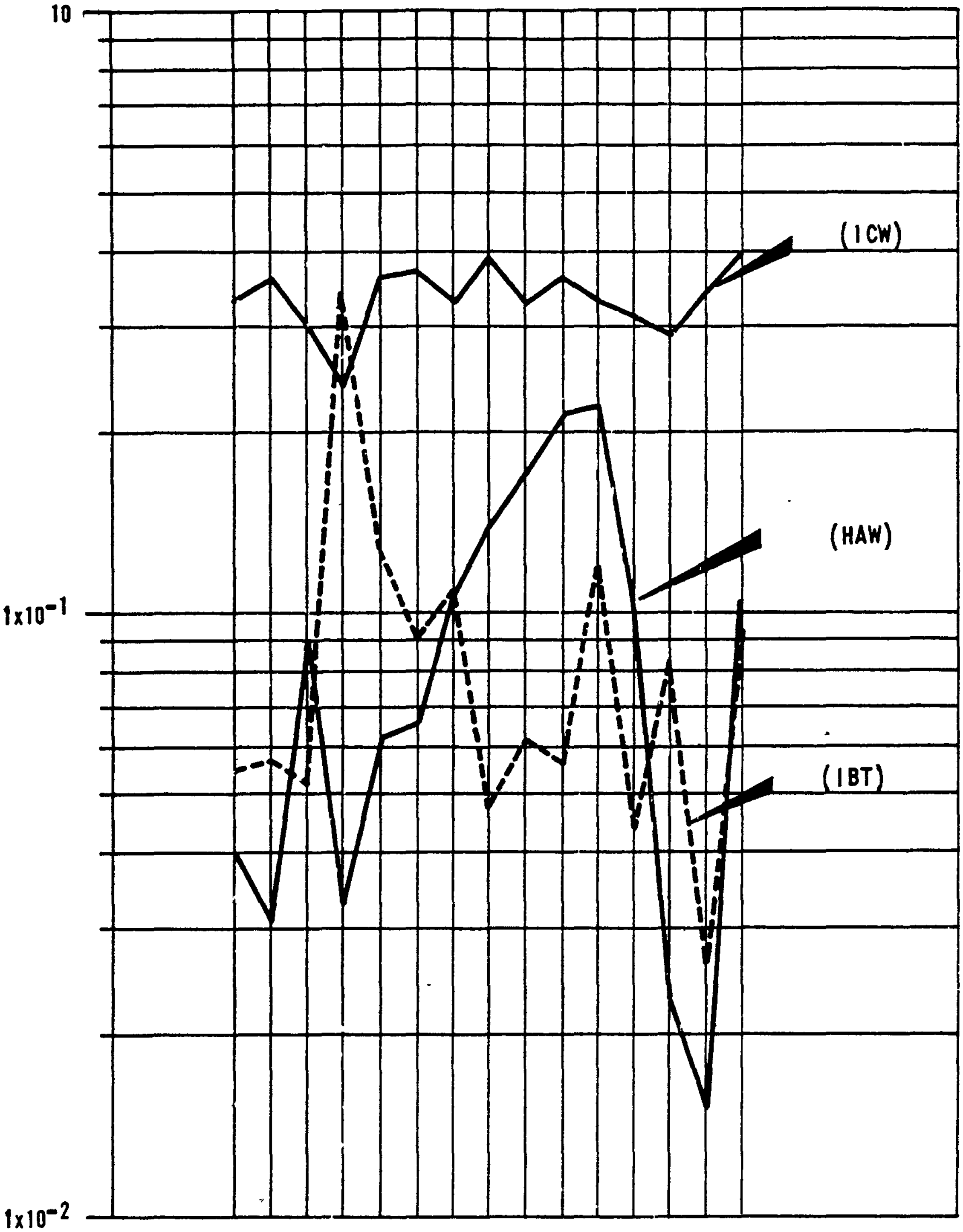


FIGURE 29

TYPICAL ^{233}U FIRST CYCLE SOLVENT EXTRACTION LOSSES (HAW, IBT, ICW)

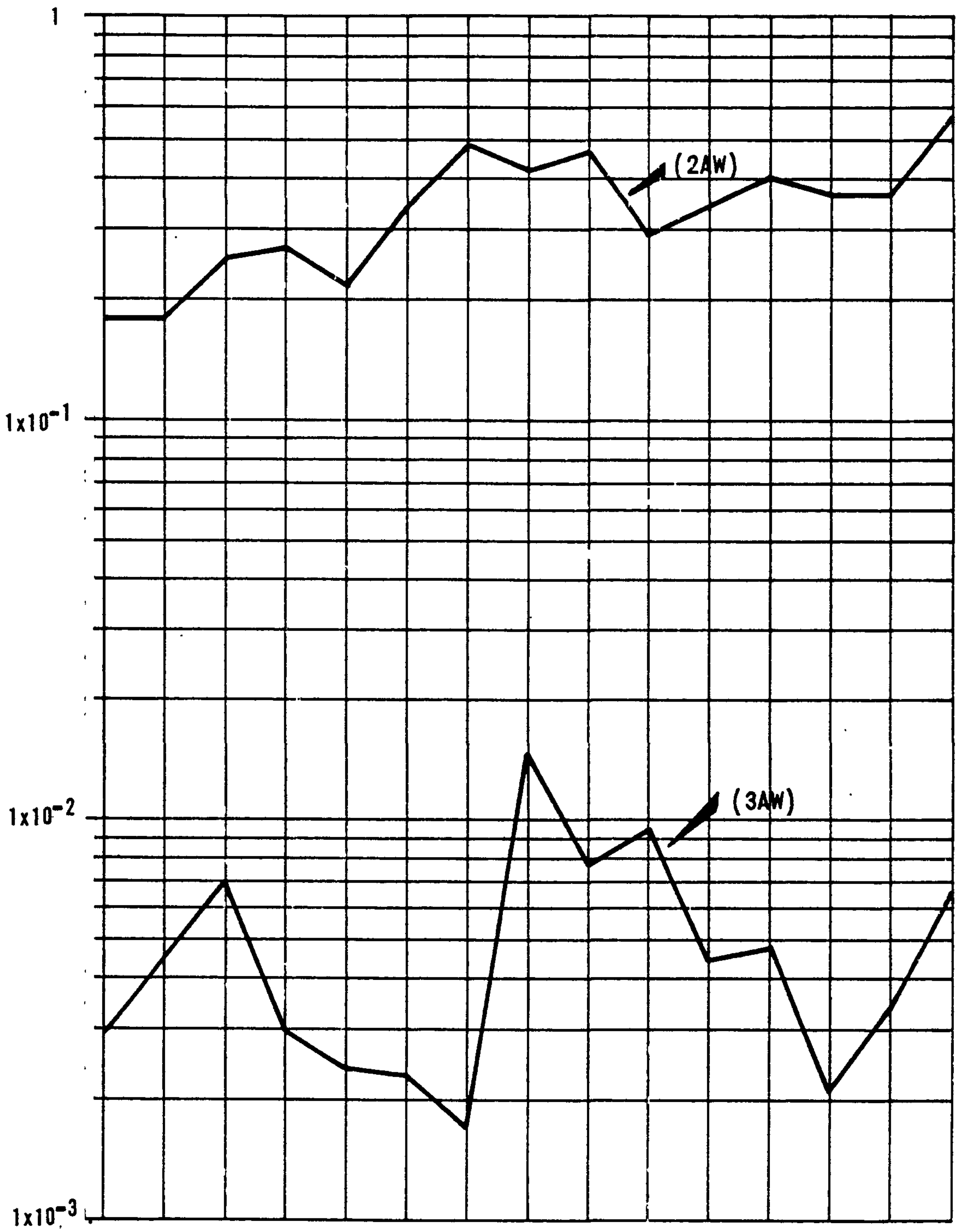


FIGURE 30

TYPICAL ^{233}U SECOND AND THIRD CYCLE SOLVENT EXTRACTION LOSSES (2AW,3AW)

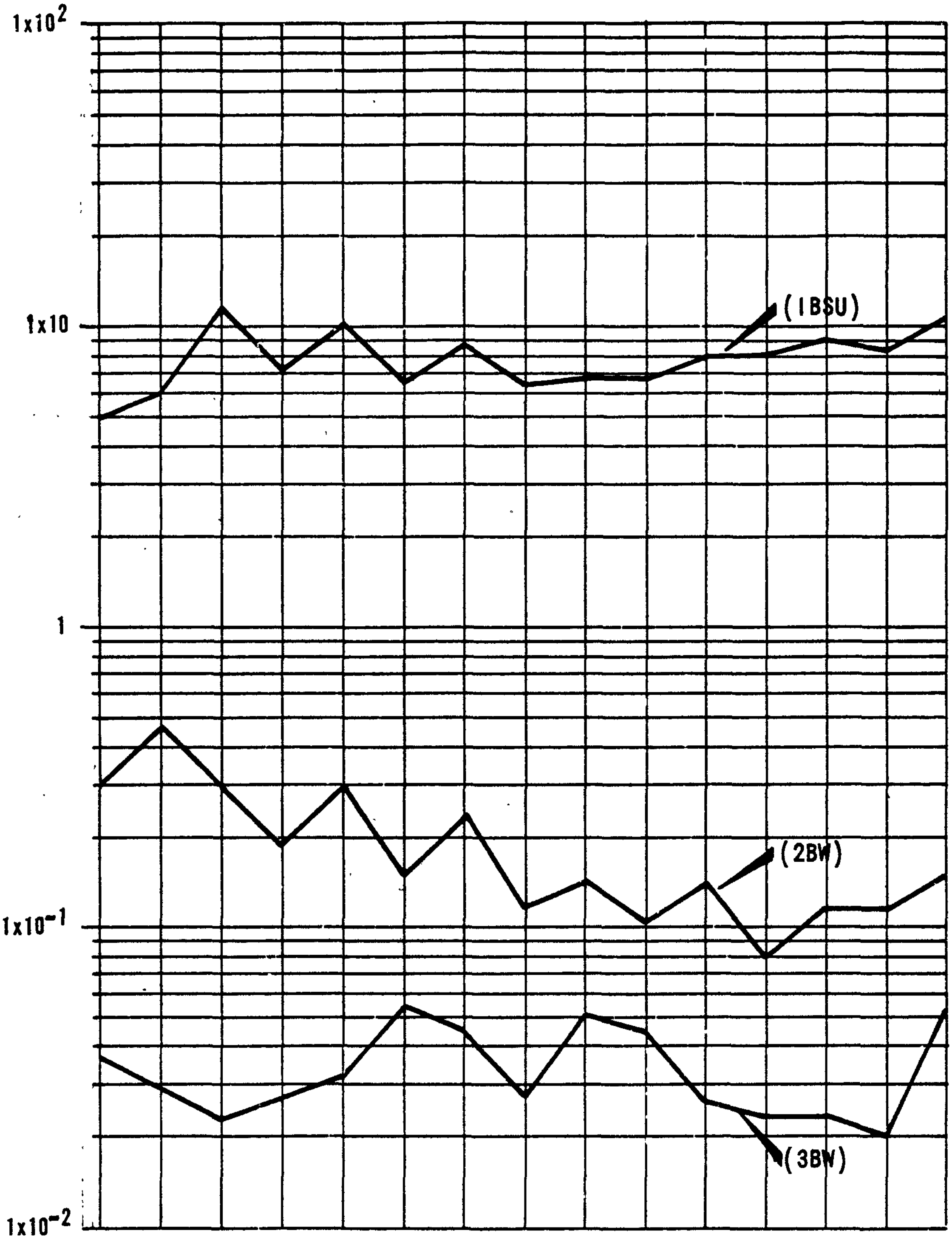


FIGURE 31

TYPICAL ^{233}U SOLVENT EXTRACTION RECYCLE (1BSU,2BW,3BW)

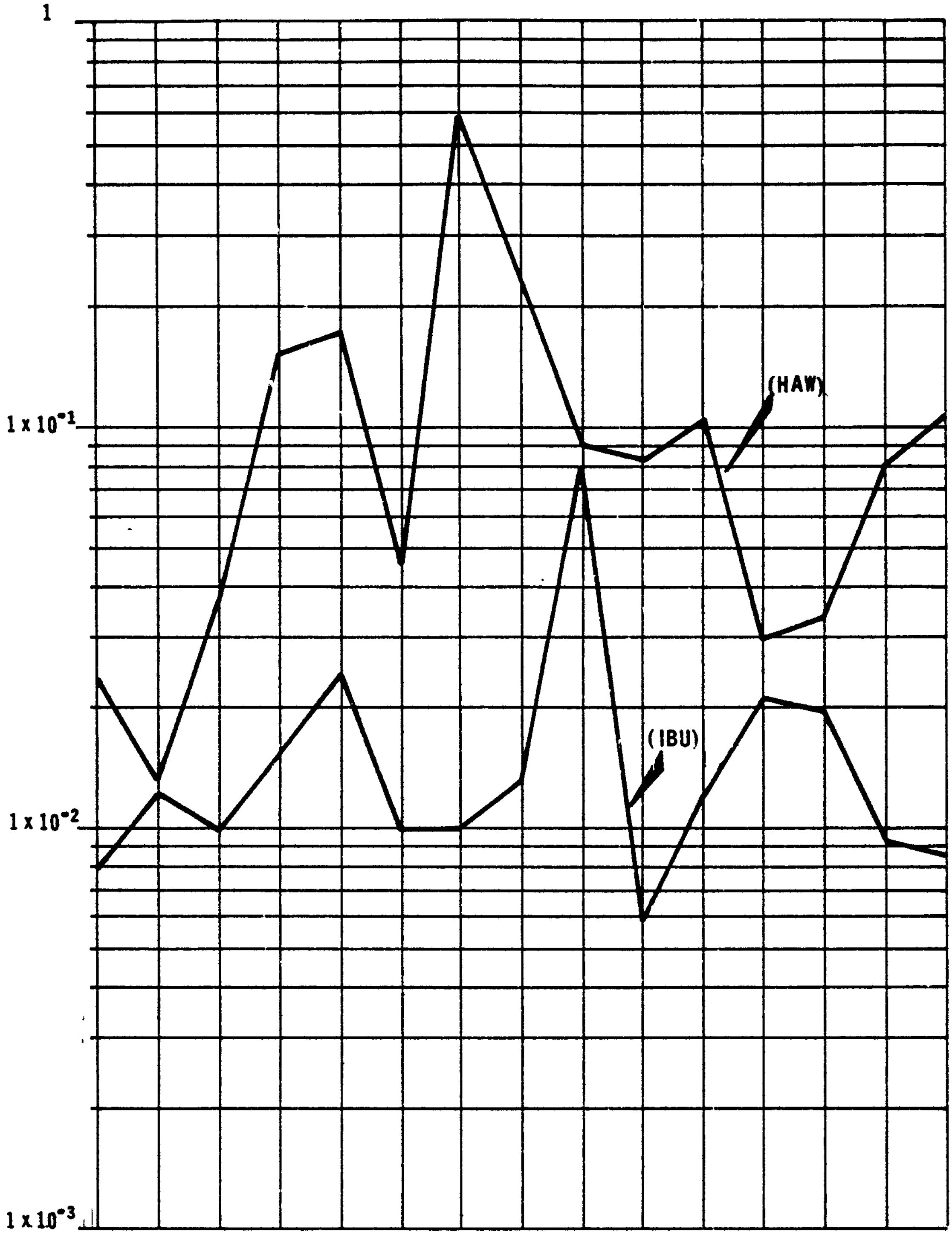


FIGURE 32

TYPICAL THORIUM FIRST CYCLE SOLVENT EXTRACTION LOSSES (HAW,IBU)

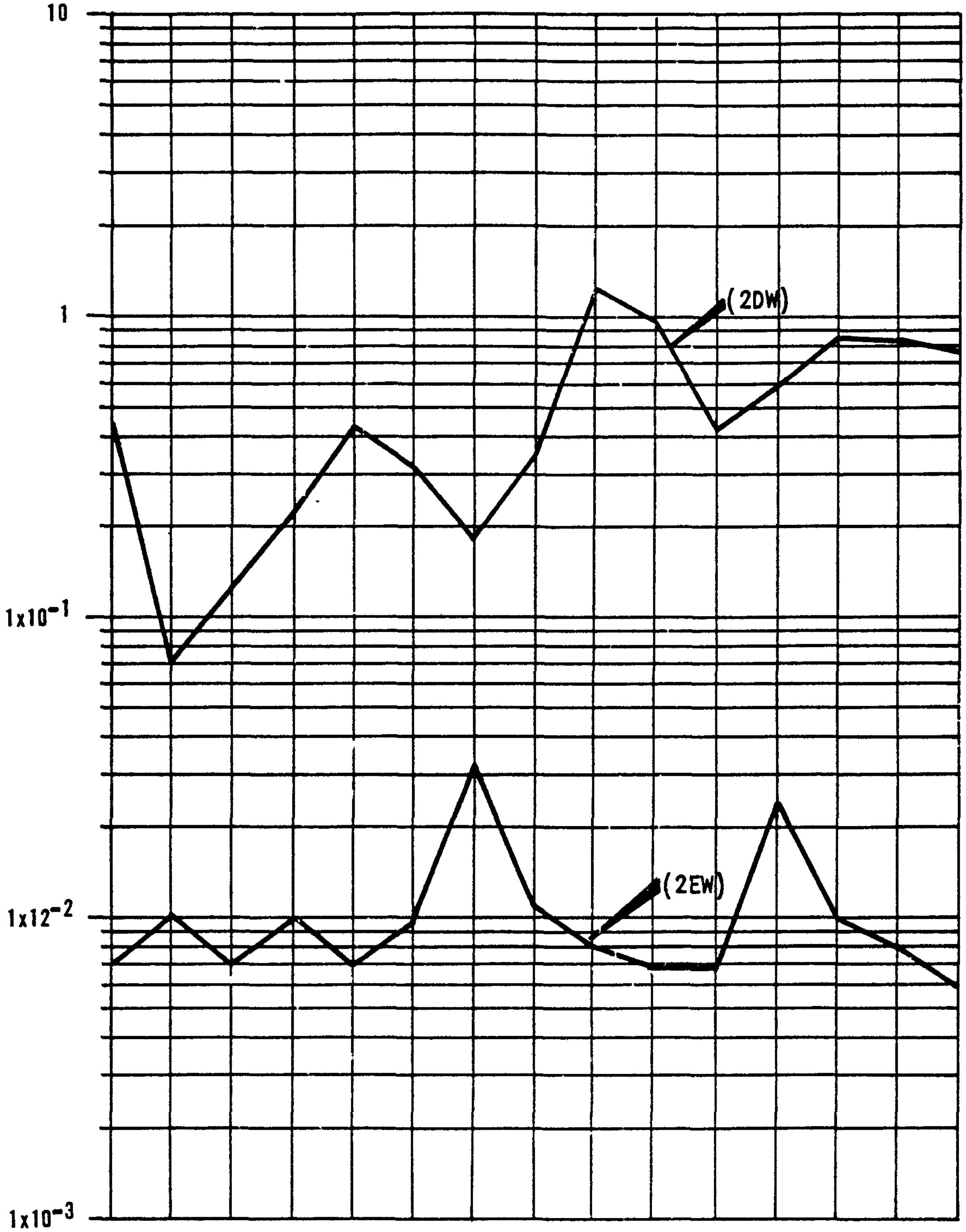


FIGURE 33

TYPICAL THORIUM SECOND CYCLE SOLVENT EXTRACTION LOSSES
(2DW, 2EW)

APPENDIX K

PHOTOS

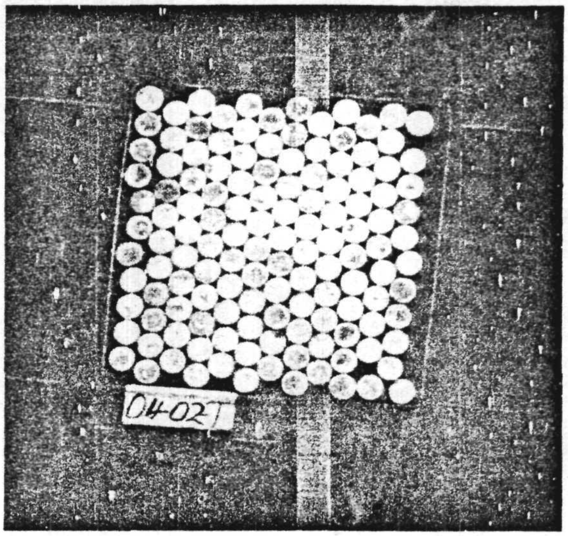


PHOTO 1

- TOP VIEW -

BUCKET CONTAINING THORIA "POWDER" ELEMENTS

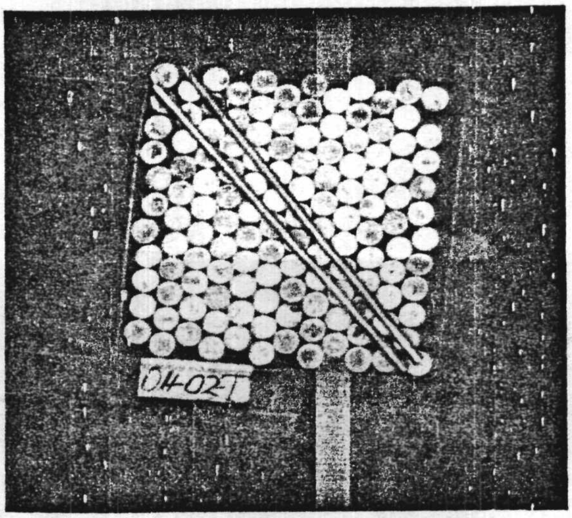


PHOTO 2

- TOP VIEW -

BUCKET CONTAINING THORIUM "WAFER" ELEMENTS WITH
DIAGONAL ALUMINUM IDENTIFICATION MARKERS

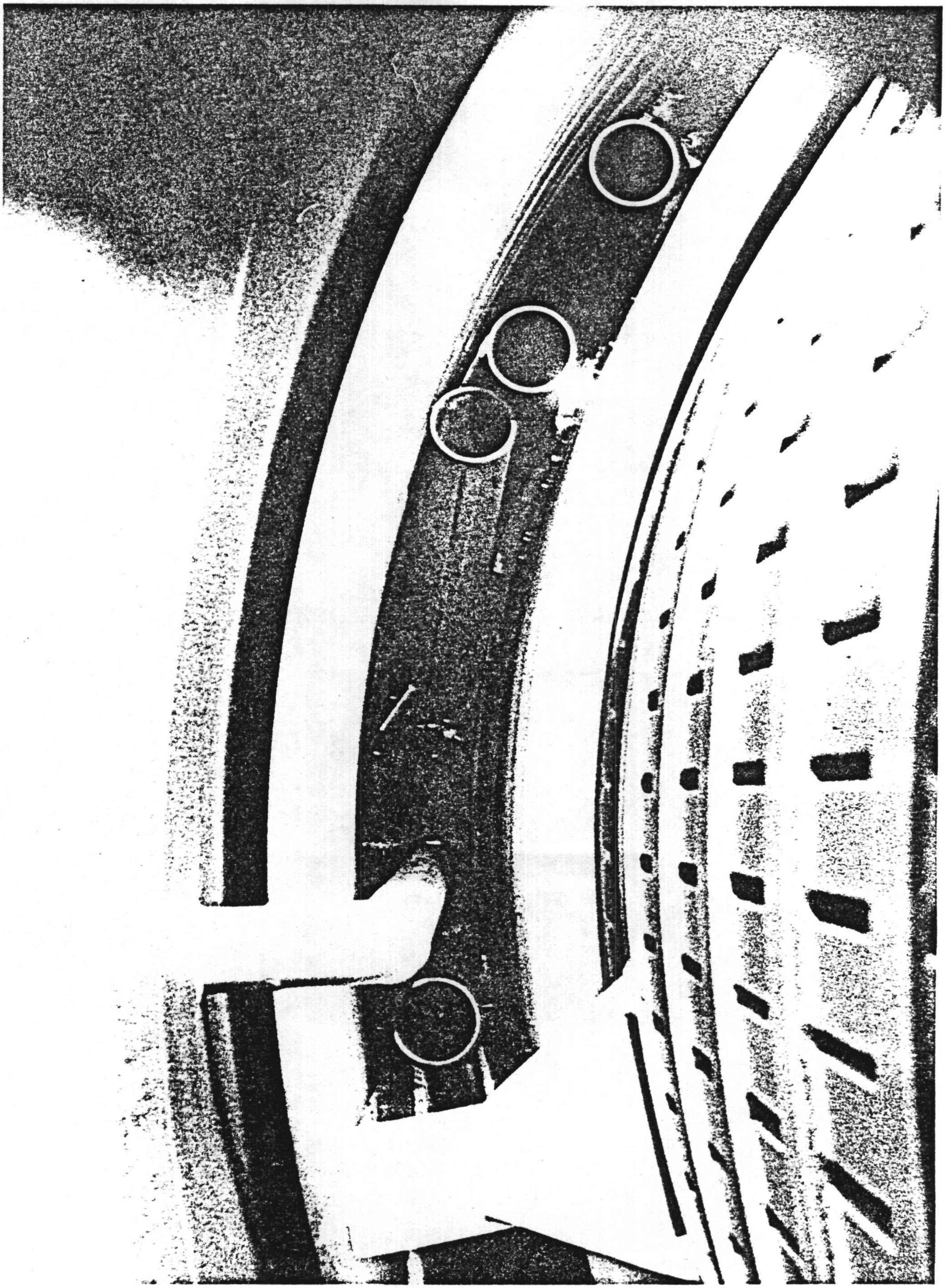


PHOTO 3

DISSOLVER AIR LIFT CIRCULATOR

APPENDIX L

TABLES

TABLE I

PULSE COLUMN DESCRIPTION

Column	Diameter (Inches)	Cartridge* Height (Feet)	Operating Volume Velocity (Gal/hr-ft^2)	Pulse Amplitude	Operating Pulse Frequency	Interface Instrument	Continuous Phase
HA Ext (T-H2)	26	13.2	620	1.86	50	Float	Organic
HA Scrub (T-H2)	26	18.1	685	1.86	50	---	Organic
1BX (T-J6)	32	28	650	0.61	50	Float	Aqueous
1BS (T-J7)	34	18	235	1.06	60	Float	Aqueous
1C (T-H3)	34	18	490	1.06	52	Dip Tubes	Aqueous
2A Ext (T-J22)	7	20	1,735	1.1	50	Float	Organic
2A Scrub (T-J22)	7	11	495	1.1	50	--	Organic
2B (T-J23)	7	19.3	660	0.75	40	Float	Aqueous
3A Ext (T-L1)	7	20.9	560	1.1	80	Float	Organic
3A Scrub (T-L1)	7	9.8	155	1.1	80	--	Organic
3B (T-L2)	7	23	180	1.1	50	Float	Aqueous
2D Ext (T-K2)	24	13.9	895	1.1	82	Float	Organic
2D Scrub (T-K2)	32	13.2	435	0.6	82	--	Organic
2E (T-K3)	34	18	505	1.06	58	Dip Tubes	Aqueous
10 (T-G2)	34	26.3	485	0.53	50	Float	Organic
20 (T-R2)	34	26.3	440	0.53	50	Float	Organic

* See Table V for Cartridge Descriptions

TABLE II

PULSE COLUMN CARTRIDGE DESCRIPTION

Column Cartridge	Plate Spacing, In.	Plate Material	Plate Type	No. of Plates	Free Area Percent	Nozzle Orientation	Hole Dia, in.
HA Ext(T-H2)	2	SS	Nozzle	58	23	Down	3/16
	(a)	SS	Louver	4	23		
HA Scrub(T-H2)	3	SS	Nozzle	61	23	Down	3/16
	(a)	SS	Louver	4	23		
1BX (T-J6)	2	SS	Sieve	138	23		3/16
	(a)	SS	Louver	6	23		
1BS (T-J7)	(d)	SS	Nozzle	74	23	Up	3/16
	(c)	SS	Louver	2	23		
1C (T-H3)	(b)	SS	Nozzle	74	23	Up	3/16
		Fluorothene*	Sieve	28	23		3/16
2A Ext(T-J22)	2	SS	Nozzle	109	23	Down	3/16
		SS	Louver	2	23		
2A Scrub (T-J22)	3	SS	Nozzle	38	23	Down	3/16
		SS	Louver	2	23		
2B (T-J23)	4(e)	SS	Nozzle	18	10	Up	1/8
	4(e)	SS	Nozzle	39	23	Up	3/16
	12(e)	Fluorothene*	Sieve	16	23		3/16
3A Ext(T-L1)	2	SS	Nozzle	128	23	Down	3/16
3A Scrub(T-L1)	3	SS	Nozzle	40	23	Down	3/16
3B (T-L2)	2	SS	Sieve	134	23		1/8
2D Ext(T-K2)	2	SS	Nozzle	51	23	Down	3/16
	(a)	SS	Louver	4	23		
2D Scrub (T-K2)	1(f)	SS	Sieve	64	21		0.085
	1(f)	Fluorothene*	Sieve	64	23		3/16
	(a)	SS	Louver	3	16		
2E (T-K3)	(b)	SS	Nozzle	74	23	Up	3/16
	(b)	Fluorothene*	Sieve	238	23		3/16
10 (T-G2)	4	SS	Nozzle	80	10	Down	1/8
20 (T-R2)	4	Fluorothene*	Sieve	80	23		3/16

* Trademark of E. I. DuPont De Nemours & Co. for poly-trifluoromonochloroethylene.

TABLE II (continued)

Notes:

(a) Louver plates are generally located adjacent to the feed distributors and at approximately four-foot intervals between.

(b) The 1BS Column and the 2E Column cartridges are composed of four different configurations as follows (from bottom up):

1. Alternate SS nozzle and fluorothene plates; spacing; nozzle plate, 3-inch space, fluorothene plate, 1/2-inch space, nozzle plate, etc. - total height = 30 inches.

2. Alternately, two nozzle plates and one fluorothene plate; spacing; nozzle plate, 3-inch space, nozzle plate, 3-inch space, fluorothene plate, 1/2-inch space, nozzle plate, etc. - total height = 46 inches.

3. Similar to Item 2, but three nozzle plates and one fluorothene plate in each group. Total height = 77 inches.
4. Alternately, six nozzle plates, and one fluorothene plate in each group. Two-inch spacing except 1/2-inch between fluorothene plate and next higher nozzle plate, - total height - 50 inches.

(c) The louver plates are spaced to divide the cartridge into three sections of approximately equal length.

(d) The nozzle plates in the lower half of the cartridge are spaced 4 inches on centers and in the upper half are spaced 2 inches on centers.

(e) The 2B Column cartridge is composed of three different configurations as follows (from bottom up):

1. A group of 11 SS nozzle plates, 1/8-inch diameter holes, 10 percent free area, with 3-inch spacing. Total height - 3 feet, 4 inches.

2. Two groups of one fluorothene sieve plate and three SS nozzle plates, 1/8-inch diameter holes, 10 percent free area. Spacing; flourothene plate, 2-inch space, nozzle plate, 4-inch space, nozzle plate, 4-inch space, nozzle plate, 2-inch space, fluorothene plate, etc. Total height = 24 inches.

3. Groups of one fluorothene sieve plate and three SS nozzle plates, 3/16-inch diameter holes, 23 percent free area. Spacing is similar to spacing in the center configuration of plates. Total height approximately 13 feet, 4 inches.

(f) The 2D scrub section cartridge configuration consists of alternate pairs of SS sieve and fluorothene sieve plates.

TABLE III

DESCRIPTION OF THORIA TARGET ELEMENTS

	Element Length (In.)	Element Diameter (In.)	Aluminum per Elem. (lb.)	Thorium per Elem. (lb.)
B Reactor				
C10T Powder Element	8.885	1.445	0.22	2.90
C Reactor				
C4T Powder Element	8.885	1.472	0.22	3.00
K Reactor Core				
K3T Powder Element	8.885	1.600	0.26	3.60
K9T Wafer Element	8.885	1.600	0.26	3.60
K Reactor Fringe				
K4T Powder Element	8.885	1.480	0.22	3.04

TABLE IV

URANIUM-233 PRODUCT QUALITY

	Specification Limit	Non-Spec Run (Batches 1-1 to 1-15)	DNR Run (Batches 2-1 to 2-44)	1966
^{232}U, ppmpU (maximum)	8.0	7.13	8.63	5.5
^{233}U, Wt. % (minimum)	97.0	97.848	98.487	97.8
^{234}U, Wt. % (maximum)	2.5	1.039	1.239	1.0
^{235}U, Wt. % (maximum)	0.1	.0225	.0207	.02
^{238}U, Wt. % (maximum)	0.5	1.090	.253	1.2
Total Alpha, d/m/gU (maximum)	2.6×10^{10}	2.5×10^{10}	2.20×10^{10}	---
Total Gamma, µc/gU (maximum)	22	.57	.35	---
^{228}Th, µc/gU (maximum)	.84	.84	.15	---
Al, ppmpUO$_2$ (maximum)	300	3	12	5
B	3	1	1	1
Ca	100	7	2	5
Cd	5	1	1	1
Cl + Br	40	19	22	---
Co	25	1	1	---
Cr	100	2	1	2
Cu	150	10	4	5
Hg	50	34	20	---
Fe	1000	12	15	25
K	200	8	3	5
Mg	150	5	1	1
Mn	50	1	2	1
Mo	100	1	1	5
Na	300	22	1	110/20*
Ni	200	2	1	2
Np	100	1	2	---
P	150	290	155	100
Pu	150	1	1	---
Si	300	16	7	30
Th	1000	57	60	---
Ti	75	3	1	---
	100	1	1	10
W	20	5	6	---
Zn	100	9	1	---

---

* 110 - Emission Spectrograph

20 - Flame Spectrophotometer

The high 1966 value for Si as associated with the use of a Vycor glass bed for ^{233}Pa decontamination that was not used in 1970.

TABLE V

TNT PRODUCT QUALITY
1970 THORIA CAMPAIGN

ANALYSES and SPECIFICATION LIMITS		TANK 006	008	007	009	204-3
Specific Gravity		1.739	1.750	1.736	1.713	1.713
Thorium	3.5 ± 0.5 lb/gal	3.40	3.44	3.52	3.50	3.53
HNO_3	0.5 ± 0.3 lb/gal	1.2	1.4	0.90	0.27	0.23
Pu	≤10 ppb	1.2	<0.16	0.71	0.24	<0.39
^{233}U	≤20 ppm	3.9	7.8	35	10	0.34
Total U (Less ^{233}U)	≤10 ppm	63	<1	<1	<1	<1
GAMMA SCAN						
ZrNb	≤50 µc/lb.Th	1.4	8.5	17	22	20
^{103}Ru and ^{106}RuRH	≤50 µc/lb.Th	22	67	80	110	145
^{233}Pa	≤300 µc/lb.Th	11	24	23	93	235
TMI (ppmTh)						
Al						
B						
Be						
Bi						
Ca						
Cd						
Co						
Cr						
Cu						
Fe	≤100					
K						
Li						
Mg						
Mn						
Na						
Ni						
P						
Pb						
Si	≤50					
Sn						
Zn						
TOTAL TMI (ppmTh)	≤1,000					
Cl (ppmTh)	≤ 50	<21	<21	<21	<21	<21
SO_4 (ppmTh)	≤ 200					
Batch Numbers		1-1 to 1-14	1-15to 1-28	2-1 to 2-15	2-16to 2-32	2-33to 2-44

TABLE V (continued)

TNT PRODUCT QUALITY
1970 THORIA CAMPAIGN

ANALYSES and SPECIFICATION LIMITS				Average		
	TANK	204-2	204-1	1970*	1966	
Specific Gravity		1.702	1.652	1.721	NA	
Thorium	3.5 ± 0.5 lb/gal	3.45	3.26	3.46	3.30	
HNO_3	0.5 ± 0.3 lb/gal	0.28	0.31	0.70	0.75	
Pu	$\leq$10 ppb	0.16	0.17	0.46	NA	
^{233}U	$\leq$20 ppm	0.25	0.23	9.3	5.0	
Total U (Less ^{233}U)	$\leq$10 ppm	<1	<1	<10	9.0	
GAMMA SCAN						
ZrNb	$\leq$50 µc/lb.Th	23	19	15	<100	
^{103}Ru and ^{106}RuRH	$\leq$50 µc/lb.Th	90	185	90	<500	
^{233}Pa	$\leq$300 µc/lb.Th	230	320	110	1500	
TMI (ppmTh)						
Al				40	50	
B				<1	1	
Be				<1	NA	
Bi				5	NA	
Ca				<1	2	
Cd				<1	5	
Co				20	NA	
Cr				70	50	
Cu				5	2	
Fe	$\leq$100			150	60	
K				<1	5	
Li				2	NA	
Mg				5	<1	
Mn				20	<2	
Na				200	3	
Ni				200	6	
P				100	<100	
Pb				<1	<2	
Si	$\leq$50			40	NA	
Sn				2	NA	
Zn				<1	<50	
TOTAL TMI (ppmTh)	$\leq$1,000			<700	<340	
Cl (ppmTh)	$\leq$ 50	<21	<21	<21	5	
SO_4 (ppmTh)	$\leq$ 200				50	
Batch Numbers		2-45to 2-61	2-62to 2-65			

*The TMI data listed are typical values.

TABLE VI

MATERIAL BALANCE FOR NON-SPECIFICATION PORTION

Waste Loss	Th, Tons	% R.R.*	U, Kgs	% R.R.*
F18				
1WW	.3	.2	1.4	.8
Sumps	.5	.4	1.7	1.0
Flushes	.1	.07	1.8	1.0
E5	.1	.07	1.0	.6
G8	.3	.2	2.0	1.1
R8	.1	.07	.1	.06
U3 & U4	0	0	0	
U in Th Product	___	____	.9	.5
TOTAL	1.4	1.0	8.9	5.1
Product Input				
Reactor Receipts	130.4	100.0	174.9	100.0
TK-D5	125.2	96.0	167.9	96.0
Product	119.8	91.9	161.3	92.2
Product & Waste Loss	121.2	92.9	170.2	97.3
Reactor MUF	-9.2	7.0	-4.7	2.7
TK-D5 MUF	-4.1	3.1	+1.3	.7

* % R.R. = percent of reactor receipts

TABLE VII

MATERIAL BALANCE FOR SPECIFICATION PORTION

Waste Loss	Th, Tons	% R.R.*	U, Kgs	% R.R.*
F18				
1WW	3.7	1.3	2.9	.6
Sumps	2.0	.7	5.5	1.2
Int. Flushes	.1	.04	.6	.1
Post Flush.	.6	.2	.7	.1
E5				
Coating Waste	.3	.1	2.7	.6
Post Flush	2.4	.8	8.7	1.9
G8				
OWW	.9	.3	3.0	.7
Post Flush	0	0	.4	.09
R8				
OWW	.1	.04	.3	.07
Post Flush	0	0	~0	
U3 & U4				
Lab Waste	0	0	.2	.04
U in K6 Product	——	——	3.9	.9
TOTAL	10.1	3.5	28.9	6.4
Product Input				
Reactor Receipts	285.5	100.0	452.6	100.0
TK-D5	280.6	98.3	449.3	99.3
Product	274.9	96.3	436.2	96.4
Product & Waste Loss	285.0	99.8	465.1	103.8
Reactor MUF	-.5	.2	+12.5	2.8
TK-D5 MUF	+1.7	.6	+4.4	1.0

* % R.R. = percent of reactor receipts

TABLE VIII

MATERIAL BALANCE FOR TOTAL CAMPAIGN

Waste Loss	Th, Tons	% R.R.	U, Kgs	% R.R.
Campaign 1	1.4		8.9	
Campaign 2	10.1		28.9	
TOTAL	11.5	2.8		6.0
Product Input #1				
Reactor Receipts	130.4		174.9	
TK-D5	125.2		167.9	
Product Input #2				
RR	285.5		452.6	
TK-D5	280.6		449.3	
TOTAL				
Reactor	415.9	100.0	627.5	100.0
TK-D5	405.8	97.6	617.2	98.4
Products				
Non-Specification	119.8		161.3	
Specification	274.9		436.2	
TOTAL	394.7	94.9	597.5	95.2
Thorium in Storage Tanks	397.4	95.6		
Product & Loss	406.2 408.9	97.7	635.3	101.2
MUF				
Reactor	-9.7	2.3	+7.8	1.2
TK-D5	-2.4	.6	+5.7	.9
Storage Tanks	-7.0	1.7		
	+ .3	.07		

TOTAL E5 Th = 2.8

TABLE IX

LIQUID WASTE VOLUMES AND ESTIMATED SALT MASS TO NON-BOILING UNDERGROUND TANK STORAGE 1970 THORIA CAMPAIGN

	Non-Specification Portion		Specification Portion		Total	
Source	Gallons	Tons of Salt	Gallons	Tons of Salt	Gallons	Tons of Salt
Pre-Campaign Flushes	360,000	150			360,000	150
TK-F18						
1WW	85,800	70	141,000	120	226,800	190
Sumps	91,800	50	213,000	120	304,800	170
Interim Flushes	43,900	40	25,000	30	68,900	70
Post Campaign Flushes			45,000	20	45,000	20
TK-E5						
Coating Waste	140,900	40	229,000	160	369,900	200
Post-Campaign Flushes			65,000	60	65,000	60
TK-G8						
OWW	278,400	20	314,000	30	592,400	50
Post-Campaign Flushes			80,000	6	80,000	6
TK-R8						
OWW	195,100	5	193,000	10	398,100	15
Post-Campaign Flushes			14,000	1	14,000	1
TK-U3 & U4 Lab. Waste plus Stack drainage, etc.	30,100	1	250,000	6	280,100	7
Total	1,226,000	380	1,569,000	560	2,795,000	940

TABLE X

COMPARISON OF ACTUAL WITH PREDICTED
WASTE VOLUMES
1970 THORIA CAMPAIGN

Source	Flowsheet Predicted, Gallons	Actual Gallons	% Increase	Increase as % of Total Predicted
Pre-Campaign Flush	350,000	360,000	2.9	0.6
Mid-Campaign Flush	*	69,000		
Post-Campaign Flush	150,000	204,000	36.0	3.2
Headend Coating Waste	270,000	370,000	37.0	5.9
Organic Wash Waste	450,000	980,000	117.8	31.2
Neutralized 1WW	220,000	227,000	3.2	0.4
Sumps	*	305,000		
Lab and Misc. Wastes	**Not Predicted	280,000		16.5
Contingency	260,000		43.8	6.7
Total	1,700,000	2,795,000	64.4	

* Sumps and Mid-Campaign Flush included in contingency
** Routed to an underground trench or crib until September, 1970.

TABLE XI

1970 PUREX PLANT GASEOUS EFFLUENTS

STACK NO. (Plant Code)	291-A-1 (291-A)			296-A-1 (E-1)		296-A-2 (E-2)	
FACILITY	Purex			Purex		Purex	
FUNCTION	Air from 291-A filters. Includes canyon ventilation air, vessel, and condenser vents, treated dissolver off-gas			Filtered air from N and Q Cells and PR Room		Hood exhaust from west sample gallery	
Radioactivity (curies)	Alpha	Beta	^{131}I	Alpha	Beta	Alpha	Beta
January	2.0x10^{-4}	0.017	0.041	5.5x10^{-5}	4.6x10^{-6}	2x10^{-7}	4.2x10^{-6}
February	7.1x10^{-5}	0.027	0.026	1.2x10^{-5}	1.7x10^{-5}	2x10^{-7}	2.0x10^{-6}
March	4x10^{-8}	0.100	0.012	4.0x10^{-6}	2.7x10^{-4}	1.10^{-7}	4.1x10^{-6}
April	1.8x10^{-4}	0.052	0.015	1.1x10^{-4}	6.9x10^{-5}	5x10^{-7}	1.4x10^{-6}
May	3.9x10^{-4}	0.092	0.049	2.1x10^{-4}	1.6x10^{-5}	4.6x10^{-6}	4.6x10^{-6}
June	3.1x10^{-4}	0.040	0.088	2.6x10^{-6}	1.6x10^{-6}	2.5x10^{-6}	3.9x10^{-6}
July	1.7x10^{-4}	0.094	0.019	6.2x10^{-6}	5.4x10^{-6}	1.9x10^{-7}	2.5x10^{-6}
August	2.9x10^{-3}	0.191	0.030	3.4x10^{-6}	2.4x10^{-6}	1x10^{-7}	1.5x10^{-6}
September	4.0x10^{-4}	0.088	0.015	4.4x10^{-5}	3.0x10^{-5}	2x10^{-7}	8x10^{-7}
October	3.26x10^{-2}	0.282	0.020	3.4x10^{-7}	2.2x10^{-6}	1.4x10^{-7}	7x10^{-7}
November	2.68x10^{-2}	0.307	0.021	1.4x10^{-5}	3.1x10^{-6}	1x10^{-7}	3x10^{-7}
December	9.31x10^{-2}	0.140	0.156	3.5x10^{-6}	1.0x10^{-5}	2x10^{-8}	5.6x10^{-6}
Total	0.157	1.430	0.492	4.7x10^{-4}	4.3x10^{-4}	8.9x10^{-6}	3.2x10^{-5}

GASEOUS EFFLUENTS (Continued)

STACK NO. (Plant Code)	296-A-3 (E-3)		296-A-5* (E-5-A)		296-A-5* (E-5-B)		296-A-6 (E-6)	
FACILITY	Purex (202-A)		Purex		Purex		Purex	
FUNCTION	Hood exhaust air from east sample gallery		Filtered air from 202-A laboratory hoods and glove boxes--West duct		Filtered air from 202-A laboratory hoods and glove boxes--East duct		Unfiltered air from east sample gallery and U Cell	
Radioactivity (curies)	Alpha	Beta	Alpha	Beta	Alpha	Beta	Alpha	Beta
January	3×10^{-7}	3.2×10^{-6}	2.4×10^{-6}	4.7×10^{-5}	Stack not in use		1.7×10^{-6}	2.7×10^{-4}
February	1×10^{-7}	2.3×10^{-6}	6×10^{-7}	2.3×10^{-6}	5×10^{-7}	4.5×10^{-6}	4×10^{-7}	5.3×10^{-5}
March	2×10^{-7}	1.6×10^{-6}	1.2×10^{-6}	8.4×10^{-5}	2.7×10^{-6}	9.1×10^{-4}	1.0×10^{-6}	7.5×10^{-4}
April	2×10^{-7}	4.9×10^{-6}	5×10^{-7}	1.9×10^{-6}	6×10^{-7}	1.5×10^{-6}	5×10^{-7}	1.1×10^{-4}
May	1×10^{-7}	4.9×10^{-6}	5×10^{-7}	3.6×10^{-6}	5×10^{-7}	8.3×10^{-5}	3×10^{-7}	5.5×10^{-5}
June	1×10^{-7}	1.8×10^{-6}	5×10^{-7}	2.9×10^{-6}	5×10^{-7}	1.5×10^{-6}	8×10^{-7}	4.3×10^{-4}
July	1.5×10^{-7}	9.1×10^{-7}	5.8×10^{-7}	1.5×10^{-5}	6.0×10^{-7}	1.9×10^{-5}	4.1×10^{-7}	3.0×10^{-5}
August	1×10^{-7}	1.9×10^{-6}	5×10^{-7}	2.2×10^{-6}	5×10^{-7}	2.7×10^{-6}	2×10^{-7}	1.2×10^{-4}
September	1×10^{-7}	2×10^{-7}	5×10^{-7}	1.3×10^{-5}	5×10^{-7}	8×10^{-7}	1.0×10^{-6}	2.0×10^{-5}
October	1.5×10^{-7}	4×10^{-7}	7×10^{-7}	1.6×10^{-6}	6×10^{-7}	2.7×10^{-6}	6.4×10^{-6}	6.9×10^{-5}
November	1×10^{-7}	1×10^{-7}	5×10^{-7}	1.4×10^{-6}	5×10^{-7}	2.9×10^{-6}	5×10^{-7}	2.7×10^{-5}
December	1.5×10^{-7}	4.6×10^{-6}	5.8×10^{-7}	2.6×10^{-5}	6×10^{-7}	1.6×10^{-5}	3.5×10^{-7}	2.5×10^{-4}
Total	1.8×10^{-6}	2.7×10^{-5}	9.1×10^{-6}	2.0×10^{-4}	8.1×10^{-6}	1.0×10^{-3}	1.4×10^{-5}	2.2×10^{-3}

* Single stack servicing two systems

GASEOUS EFFLUENTS - 1970 (Continued)

STACK NO. (Plant Code)	296-A-7 (E-7)		296-A-8 (Rack 73) (E-8)		296-A-10 (BT-2)		296-A-12 (150 ft.) (ARVVS)		
FACILITY	Purex		Purex		Purex		244-AR Vault		
FUNCTION	Unfiltered air from west sample gallery and R Cell		Unfiltered air from west end of P&O Gallery. (White Room exhaust)		Filtered air from burial tunnel No.2		Filtered air from vessel vent system		
Radioactivity (curies)	Alpha	Beta	Alpha	Beta	Alpha	Beta	Alpha	Beta	^{131}I
January	$8.1x10^{-6}$	$5.9x10^{-3}$	$2x10^{-7}$	$9.1x10^{-5}$	$2x10^{-7}$	$5.0x10^{-2}$	$8.6x10^{-7}$	$6.8x10^{-3}$	$3x10^{-7}$
February	$4.4x10^{-6}$	$1.9x10^{-4}$	$1x10^{-7}$	$9x10^{-7}$	$4x10^{-8}$	$2.0x10^{-3}$	$9.5x10^{-8}$	$3.1x10^{-5}$	$1.1x10^{-4}$
March	$3.2x10^{-5}$	$2.2x10^{-2}$	$2.1x10^{-6}$	$2.9x10^{-4}$	$1x10^{-7}$	$1.6x10^{-3}$	$2.1x10^{-7}$	$9.6x10^{-5}$	$1.6x10^{-6}$
April	$9.5x10^{-6}$	$1.8x10^{-3}$	$8x10^{-7}$	$1x10^{-7}$	$3x10^{-7}$	$6.0x10^{-4}$	$8.7x10^{-7}$	$3.5x10^{-4}$	$1.1x10^{-4}$
May	$5.7x10^{-5}$	$4.9x10^{-4}$	$5x10^{-7}$	$2.3x10^{-5}$	$2x10^{-7}$	$3.7x10^{-5}$	$<7.4x10^{-7}$	$6.7x10^{-4}$	$3.8x10^{-6}$
June	$3.0x10^{-5}$	$3.5x10^{-4}$	$5x10^{-7}$	$2.6x10^{-5}$	$2x10^{-7}$	$1.4x10^{-5}$	$<1.1x10^{-6}$	$1.3x10^{-4}$	$3.3x10^{-6}$
July	$3.8x10^{-6}$	$3.8x10^{-4}$	$6.8x10^{-7}$	$4.5x10^{-5}$	$2.0x10^{-7}$	$1.5x10^{-5}$	$<4.3x10^{-7}$	$2.2x10^{-6}$	$<2.0x10^{-7}$
August	$6x10^{-7}$	$3.2x10^{-4}$	$7x10^{-7}$	$1.8x10^{-5}$	$2x10^{-7}$	$6x10^{-7}$	$<6.5x10^{-7}$	$2.4x10^{-5}$	$<1.1x10^{-7}$
September	$3.7x10^{-6}$	$2.6x10^{-4}$	$2.2x10^{-6}$	$1.1x10^{-4}$	$1.0x10^{-6}$	$3.2x10^{-5}$	$<1.6x10^{-6}$	$1.2x10^{-5}$	$<1.3x10^{-7}$
October	$2.7x10^{-5}$	$3.5x10^{-4}$	$7x10^{-7}$	$1.1x10^{-5}$	$2.5x10^{-7}$	$6.3x10^{-6}$	$<6.0x10^{-7}$	$3.5x10^{-7}$	$2.9x10^{-7}$
November	$2.8x10^{-6}$	$3.0x10^{-4}$	$5x10^{-7}$	$4.7x10^{-6}$	$2x10^{-7}$	$7.6x10^{-6}$	$<1.1x10^{-6}$	$3.0x10^{-4}$	$<1.9x10^{-7}$
December	$2.1x10^{-6}$	$5.8x10^{-4}$	$7x10^{-7}$	$1.7x10^{-5}$	$1x10^{-7}$	$6.0x10^{-6}$	$<3.3x10^{-7}$	$3.9x10^{-6}$	$2.0x10^{-7}$
Total	$1.8x10^{-4}$	$3.3x10^{-2}$	$9.7x10^{-6}$	$6.4x10^{-4}$	$3.0x10^{-6}$	$5.4x10^{-2}$	$<8.6x10^{-6}$	$8.4x10^{-3}$	$<2.3x10^{-4}$

GASEOUS EFFLUENTS - 1970 (Continued)

STACK NO. (Plant Code)	296-A-13 (ARCS)			296-A-14 (E-4)		296-A-17 (AXFS)	
FACILITY	244-AR Vault			Purex		241-A Tank Farm	
FUNCTION	Filtered air from canyon and cell ventilation			Filtered air from 293-A Building		Filtered noncondensible vapors from waste storage tanks in 241-A and AX Tank Farms	
Radioactivity (curies)	Alpha	Beta	^{131}I	Alpha	Beta	Alpha	Beta
January	$1.5x10^{-6}$	$7.4x10^{-6}$	$1.4x10^{-5}$	Sampler activated February 1970		$7.6x10^{-7}$	$2.9x10^{-6}$
February	$6.3x10^{-7}$	$9.5x10^{-6}$	$8.9x10^{-6}$	$1x10^{-7}$	$5x10^{-7}$	$8.6x10^{-7}$	$2.4x10^{-6}$
March	$7.8x10^{-6}$	$1.7x10^{-5}$	$<5x10^{-6}$	$2x10^{-7}$	$4.7x10^{-6}$	$1.8x10^{-7}$	$6.1x10^{-5}$
April	$2.5x10^{-5}$	$2.4x10^{-5}$	$3.0x10^{-5}$	$3x10^{-7}$	$8x10^{-7}$	$3.8x10^{-6}$	$3.8x10^{-5}$
May	$<1.5x10^{-5}$	$2.1x10^{-5}$	$<6.0x10^{-6}$	$3x10^{-7}$	$8.1x10^{-6}$	$<3.5x10^{-6}$	$8.7x10^{-5}$
June	$<1.6x10^{-5}$	$1.5x10^{-5}$	$<2.2x10^{-6}$	$2x10^{-7}$	$3x10^{-7}$	$<3.8x10^{-6}$	$1.0x10^{-4}$
July	$<1.8x10^{-5}$	$<1.2x10^{-5}$	$7.0x10^{-6}$	$2.5x10^{-7}$	$5.5x10^{-7}$	$<3.0x10^{-6}$	$2.6x10^{-6}$
August	$<1.7x10^{-5}$	$<1.6x10^{-5}$	$<6.0x10^{-6}$	$2x10^{-7}$	$1.7x10^{-6}$	$<4.1x10^{-6}$	$<1.6x10^{-6}$
September	$<1.7x10^{-5}$	$1.9x10^{-5}$	$6.8x10^{-6}$	$2x10^{-7}$	$1x10^{-7}$	$<6.1x10^{-6}$	$<3.6x10^{-6}$
October	$<1.8x10^{-5}$	$1.3x10^{-5}$	$<7.0x10^{-6}$	$4x10^{-7}$	$2.6x10^{-6}$	$<3.7x10^{-6}$	$<2.0x10^{-7}$
November	$<1.5x10^{-5}$	$2.5x10^{-5}$	$<6.0x10^{-6}$	$2x10^{-7}$	$2.7x10^{-6}$	$<1.8x10^{-6}$	$<6.5x10^{-6}$
December	$<1.2x10^{-5}$	$3.8x10^{-5}$	$5.0x10^{-6}$	$2.5x10^{-7}$	$2.2x10^{-5}$	$<3.8x10^{-6}$	$2.8x10^{-5}$
Total	$<1.6x10^{-4}$	$<2.2x10^{-4}$	$<1.0x10^{-4}$	$2.6x10^{-6}$*	$4.4x10^{-5}$*	$<3.5x10^{-5}$	$<3.3x10^{-4}$

* Estimate for year

TABLE XII

LIQUID EFFLUENTS
1970

Disposal Site: 216-A-10 Crib
Waste Stream Description: Purex Process Condensates

1970	Volume (nx10^6 gal)	U (lbs)	Pu (gm)	Beta (Ci)	^{60}Co (Ci)	^{90}Sr (Ci)	^{137}Cs (Ci)	^{106}Ru (Ci)	^{233}U* (gm)
January	.907	.4	.9	35.2	.06	--	.47	.015	
February	.907	.4	.8	35.2	.05	--	.46	.015	
March	.351	< .1	1.5	2.4	<.001	.05	<.03	.31	
April	1.72	.9	9.0	25.8	<.01	.48	<.34	3.95	
May	.738	.7	6.4	26.6	.01	.5	.33	2.66	
June	.83	1.0	7.6	40.8	<.01	.59	.68	3.66	
July	.05	.06	.48	2.15	<.001	.03	.04	.22	
August	.251	< .27	--*	12.3	<.008	.14	.2	1.11	15.3*
September	2.08	1.89	--*	62.6	<.06	1.02	1.23	6.68	155. *
October	1.02	< .21	--*	7.22	<.004	.13	.14	.73	72.6*
November	2.57	.13	--*	3.45	<.002	.02	.06	.29	29.4*
December	1.43	< .06	--*	.29	<.0002	--	.007	.04	4.54*
12-Month Total	12.9	6.12	26.7	254.	<.216	2.96	3.99	19.7	277. *

* Thorium Campaign

LIQUID EFFLUENTS - 1970 (Continued)

Disposal Site: 216-A-25 Gable Mountain Pond
Waste Stream Description: Purex Process Cooling Water

1970	Volume (nx10^6 gal)	U (lbs)	Pu (gm)	Beta (Ci)	^{60}Co (Ci)	^{90}Sr (Ci)	^{137}Cs (Ci)	^{106}Ru (Ci)	^{233}U* (gm)
January	96.6	.4	.3	1.3	< .79	--	< .95	< 7.0	
February	96.5	.3	.2	1.3	< .79	--	< .94	< 6.9	
March	104.	5.3	< 4.2	.22	< .03	< .01	< .08	1.84	
April	142.	6.9	< 5.6	< .35	< .05	< .03	< .05	< .29	
May	196.	< 19.1	< 1.9	1.43	< .05	--	1.68	< .89	
June	204.	< 11.	< 1.7	1.31	< .03	--	< .06	< .36	
July	170.	2.32	< 1.83	.28	< .03	.08	< .03	< .41	
August	300.	< 26.4	--*	.16	< .07	.03	< .1	< .63	< 20.4*
September	317.	< 15.4	--*	< 6.6	.08	< .18	< .06	< 3.34	< 85.8*
October	383.	< 18.6	--*	< 7.97	.1	< .22	< .07	< 3.79	<104. *
November	299.	< 14.5	--*	< 6.22	.08	< .17	< .05	< 2.96	< 81. *
December	277.	< 13.4	--*	< 5.76	.07	< .16	< .05	< 2.74	< 74.9*
12-Month Total	2,585.	<133.6	<15.7	<32.9	<2.17	< .88	<4.12	<31.15	<366.1*

* Thorium Campaign

LIQUID EFFLUENTS - 1970 (Continued)

Disposal Site: 216-A-27
Waste Stream Description: Purex Laboratory waste and 291-A and 293-A Building waste

1970	Volume (nx10^6 gal)	U (lbs)	Pu (gm)	Beta (Ci)	^{60}Co (Ci)	^{90}Sr (Ci)	^{137}Cs (Ci)	^{106}Ru (Ci)	^{233}U* (gm)
January	.025	.025	< .00005	< .0025	< .0015	--	< .01	< .005	
February	.025	.025	< .00005	< .0025	< .0015	--	< .01	< .005	
March	.029	< .01	.2	2.25	< .001	.05	.06	.33	
April	.021	3.	.2	2.12	.0003	.0005	.001	.46	
May	.024	1.8	.1	5.56	< .009	.48	.49	.19	
June	.033	2.3	.9	29.5	< .04	2.66	2.68	.38	
July	.036	.55	7.12	195.	< .51	8.69	12.	3.26	
August	.056	.59	--*	165.	< .25	3.97	10.7	.09	17.2*
September	.028	.18	--*	241.	< .802	12.1	12.2	3.05	31.1*
October	No additions			Streams going to 216-A-27 crib were routed to underground storage starting in September, 1970.					
November	No additions								
December	No additions								
12-Month Total	.277	8.48	8.52	640.4	<1.62	27.95	38.15	7.77	48.3

* Thorium Campaign - No Pu in process

LIQUID EFFLUENTS - 1970 (Continued)

Disposal Site: 216-A-30 Crib
Waste Stream Description: Purex coil and steam condensate(1)

1970	Volume (nx10^6 gal)	U (lbs)	Pu (gm)	Beta (Ci)	^{60}Co (Ci)	^{90}Sr (Ci)	^{137}Cs (Ci)	^{106}Ru (Ci)	^{233}U* (gm)
January	3.1	.08	.08	.32	<.024	--	< .032	2.67	
February	3.88	.1	.1	.4	<.03	--	< .04	3.34	
March	1.72	< .1	< .1	35.7	<.08	2.12	2.83	2.	
April	5.25	1.7	< .3	10.	.005	.89	.56	.57	
May	4.18	.2	< .2	2.34	<.002	.15	.13	.15	
June	3.95	< .2	< .2	.64	<.001	.05	.05	.07	
July	.598	.03	< .02	.05	<.0001	.004	.002	.004	
August	1.59	< .08	--*	.09	<.0003	< .01	.005	.009	< .4 *
September	5.5	.27	--*	.54	<.002	.04	.03	.06	<1.37*
October	4.6	< .22	--*	.49	<.001	.03	.02	.05	<1.15*
November	7.36	.33	--*	2.55	.003	.11	.07	.1	<1.73*
December	6.13	< .28	--*	30.	<.02	--	.39	< .09	2.25*
12-Month Total	47.9	<3.59	<1.	83.1	<.168	3.4	4.16	9.11	<6.9

* Thorium Campaign

(1) In January, 1970, the 216-A-6 crib was removed from service and that portion of the condensate stream was also routed to the 216-A-30 crib.

LIQUID EFFLUENTS - 1970 (Continued)

Disposal Site: 216-A-36-B Crib
Waste Stream Description: Purex dissolver and E Cell ammonia scrubber waste

1970	Volume ($n \times 10^6$ gal)	U (lbs)	Pu (gm)	Beta (Ci)	^{60}Co (Ci)	^{90}Sr (Ci)	^{137}Cs (Ci)	^{106}Ru (Ci)	^{233}U* (gm)
January	.083	.1	< .0002	2.5	.015	--	< .001	4.9	
February	.082	.1	< .0002	2.4	.015	--	< .001	4.9	
March	.493	2.3	1.7	99.6	.26	2.62	< 2.3	3.7	
April	.136	.9	< .35	17.8	< .05	.73	< .7	7.82	
May	1.11	48.9	1.31	10,840.	4.85	218.	77.2	1,413.	
June	.016	.1	< .13	11.4	.02	< .19	< .24	3.91	
July	No addition	--	--	--	--	--	--	--	
August	.117	.11	--*	9.54	.05	6.25	25.2	10.4	< .8 *
September	.173	1.62	--*	92.9	< .09	.69	24.	79.9	9.97*
October	.083	< .02	--*	49.2	< .007	< .06	< .04	10.7	.99*
November	.228	1.69	--*	80.2	.1	.26	.68	55.4	3.43*
December	.069	1.84	--*	124.	.839	.08	< .68	73.9	.41*
12-Month Total	2.59	57.7	3.49	11,330.	6.3	228.9	131.	1,669.	15.6

* Thorium Campaign

LIQUID EFFLUENTS - 1970 (Continued)

Disposal Site: 216-B-3 Pond
Waste Stream Description: Purex Chemical Sewer and Acid Fractionator Condensate

1970	Volume (nx10^6 gal)	U (lbs)	Pu (gm)	Beta (Ci)	^{60}Co (Ci)	^{90}Sr (Ci)	^{137}Cs (Ci)	^{106}Ru (Ci)	^{233}U* (gm)
January	8.	.15	.05	.25	<.07	--	<.08	< .58	
February	8.	.15	.05	.25	<.06	--	<.08	< .57	
March	3.15	< .2	<.02	.03	<.001	<.0003	<.002	< .01	
April	13.8	.7	<.6	.07	<.006	<.002	<.008	< .07	
May	6.52	< .4	<.1	.06	<.003	<.003	<.003	< .02	
June	7.12	< .4	<.1	.04	<.001	--	<.003	< .01	
July	.141	< .01	<.01	.004	<.00002	<.0001	<.0001	< .0006	
August	1.01	< .05	--*	.15	<.0002	<.0006	<.0005	.08	< .07*
September	6.54	< .32	--*	.41	<.003	.004	<.003	.2	<1.84*
October	4.14	< .22	--*	.03	<.001	.001	<.001	< .01	<1.2*
November	9.39	< .43	--*	.75	<.002	.002	<.002	.22	<2.18*
December	5.71	.54	--*	.71	.003	--	<.0003	.47	<1.38*
12-Month Total	73.5	<3.57	<.93	2.75	<.15	<.013	<.183	<2.24	<6.67*

* Thorium Campaign - no Pu in process