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ORNL 655
Chemistry-Separation Processes for Plutonium and Uranium

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# CHEMICAL TECHNOLOGY DIVISION

## PILOT PLANTS SECTION
## REPORT FOR FEBRUARY, 1950

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ORNL-655*
Chemistry-Separation
Processes for Plutonium
and Uranium

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CHEMICAL TECHNOLOGY DIVISION

# PILOT PLANTS SECTION REPORT FOR FEBRUARY, 1950

D. G. Reid

DATE ISSUED
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## 0.0 ABSTRACT

The monthly report for the Pilot Plants Section of the ORNL Chemical Technology Division for the month of February, 1950 is presented. The data and progress reported include:

1. Six additional U-233 separation runs (7-12) representing an overall problem completion of 60%. During this problem investigation, a total of 185 grams of U-233 has been separated and adequately decontaminated.
2. The 205 Pilot Plant processing of the IBP solution from the Redox process investigations for recovery of the Pu which is now essentially complete.
3. Data obtained during evaluation of the ORNL Radiochemical Waste Evaporator.
4. Program for separation of the highly irradiated uranium that is to be started in the coming period.
5. Preliminary design basis for the ORNL Waste Uranium Recovery Unit.
6. Status of the 25 process report and miscellaneous analytical statistical data.

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## "23" PILOT PLANT DEVELOPMENT

Technical - Jackson, Ferguson, Hylton, Landry, Lewis, Nicholson, Winget

Operators - Davis, Gifford, Caldwell, Summers, Jones, Shipwash, Grizzell, Lockmiller, Spangler, Jennings, Thompson, Thomas, Purkey

### 1.0 23 PROCESS DEVELOPMENT

Six $U^{233}$-Th separations runs (7T-12T) processing Hanford irradiated thorium were made during February. The results of the twelve hot runs completed to date indicate that the near-optimum flowsheet conditions for the methyl isobutyl ketone system have been established, and the one-cycle process yields decontamination factors are $2 \times 10^4$ for fission products, $1 \times 10^5$ for Pa, and $1 \times 10^4$ for Th. The fission product activity in the product is about equal to the 23 background. Total 23 losses have been less than 0.2%, and 185 grams of 98.5% isotopic purity U-233 have been separated. Equipment operation continued to be smooth.

Eight additional hot runs will be made with the available irradiated thorium metal. The effect of acidity and solvent impurities will be studied, and stage height will be investigated by decreasing the extraction height until the losses can be measured with some degree of accuracy. One run at increased flowrates will also be made.

A detailed progress report of the first ten runs has been presented in ORNL-651.

[redacted]

1.1 Feed Preparation

The general features of feed makeup are very similar to feed make-up for the Redox process. The thorium metal used during February was irradiated at Hanford for 100 days and cooled 24 months. Aluminum jackets were removed by the standard 10% NaOH - 20% $NaNO_3$ procedure, and dissolver solutions about 0.2 N acid deficient were produced by using 65% nitric acid and dissolving to a specific gravity of 2.09. A summary of dissolving data is given in Table I-1, Appendix I. Necessary minor acidity adjustments are made by the addition of concentrated nitric acid or sodium hydroxide. Feed solutions are filtered by vacuum through a sintered stainless steel filter of 10 micron porosity.

1.2 Column Operation

1.21 Objectives of Runs

The present 23 process flowsheet, shown in Figure 3, Appendix I, was used for all runs, except that variations were made in feed thorium concentrations, as noted below. The major objectives of runs completed during February were as follows:

7T-10T - To investigate the effect of feed thorium concentration on decontamination. Runs were made at 1.8, 1.4, 1.2, and 1.6 M $Th(NO_3)_4$ in the metal feed.

11T - To investigate the effect of low temperature on decontamination. The extraction column was maintained at 50° F during this run.

12T - To determine the effect of increasing the scrub height from 11' to 25' and decreasing the extraction height from 30' to 16'.

1.22 Results of Column Runs

A summary of loss and decontamination data for Runs 1T to 12T is given in Tables I-2, I-3 and I-4, Appendix I. The following conclusions are drawn from these data.

1. The present 23 process flowsheet (1.6 M thorium in the feed) yields decontamination factors of $2 \times 10^4$ for gross fission products, $10^5$ for Pa, and $10^4$ for Th.
2. Gross fission product decontamination varies by less than a factor of two when the feed thorium concentration is reduced to 1.2 M or increased to 1.8 M.
3. The decontamination factor for ruthenium increased from 100-600 at 1.8 M thorium nitrate to $5 \times 10^3$ at 1.2 M thorium nitrate. The decontamination factors for Th and Ce appeared to be independent of metal feed concentration, and were $1 \times 10^4$ to $2 \times 10^5$ for Th, and $1 \times 10^5$ to $7 \times 10^5$ for Ce.
4. The 23 loss was consistent and averaged 0.10% in the aqueous raffinate (AR) and less than 0.01% in the solvent raffinate. The AR loss was due to an inextractable form of 23, and no variation was noted when the extraction was done at $50^\circ$ F or when the extraction height was decreased from 25' to 16' or thorium concentration varied.
5. Increasing the scrub height from 11' to 25' resulted in only slightly increased decontamination - less than a factor of two.

1.3 Product Evaporation, Filtration

The strip product from each run is evaporated from 50 liters to 1.0 liter, and is then filtered in the laboratory to yield a sparkling clear solution. Total 23 loss for evaporation and filtration is less than 0.01%.

1.4 Product Purity

The results of an investigation of the beta and gamma activity associated with 23, Ru and Pa are given in Appendix I. On the basis of these data it is concluded that about half of the beta and gamma activity of the product is due to 23 background.

The results of mass spectrographic analyses on the 23 product from Runs 1T, 5T, 8T and 9T are given in Table I-5, Appendix I. The isotopic purity of the product is about 98.5%, with the major contaminants being U-238 and U-234.

1.5 Equipment Performance

A detailed summary of 23 Pilot Plant equipment performance has been presented in the report referred to in Section 1.0. The major equipment investigation conducted during February was in connection with the low temperature run. It was found that the extraction column temperature control unit could maintain $\pm 1^{\circ}$ F temperature control between $50^{\circ}$ F and $100^{\circ}$ F with no difficulty other than that caused by significant cell temperature changes. Cell temperature changes of $10^{\circ}$ F require manual adjustment of the heating and refrigeration units.

1.6 Program

Tentative major objectives of the final ten runs are given below. Unless otherwise noted, the flowsheet for these runs will be that given in Figure I, Appendix I.

14T,15T - To determine the effect of feed acidity on decontamination and loss. The present specification on feed acidity is 0.20 ± 0.05 N acid deficient. Wider specifications would be desirable for plant operation, and it is hoped to demonstrate that feed specifications can be changed to 0.20 ± 0.15 N acid deficient.

16T - To determine the effect of gross amounts of solvent impurities. The solvent used for this run will contain 0.5% methyl isobutyl carbinol. If the results of this run are satisfactory, subsequent solvent treatment will consist of a simple caustic wash.

17T - To determine the effect of flowrates on decontamination, losses and column operability. This run will be made at 50% of flooding, three times the normal flowrates.

18T,19T - These runs will be reserved for additional special studies, or for demonstration of the flowsheet at the highest available activity.

20T - To determine the losses and decontamination obtainable using diisopropyl ether as solvent. This ether was tested during the laboratory investigation of the 23 Process, and a comparison of this system with the methyl isobutyl ketone system is desired.

## PLUTONIUM RECOVERY

Technical - Rigstad, Harrington

Operators - Benson, Burnett, Groover, Jennings, Ledbetter, McLellan, Sexton, Shields, Spangler, Strader, Thompson, Wiggins

### 2.0 PLUTONIUM RECOVERY

Pilot Plant processing of the IBP solution which was separated during the Pilot Plant Redox investigation has been essentially completed. The material was processed in a series of eight runs, followed by two equipment cleanout runs. The concentrated plutonium product solution will be transferred to the laboratory when facilities are available for further purification.

No additional modifications in flowsheet conditions for processing the non-representative material were found necessary other than as outlined in the January 1950 report. These modifications were:

(1) Reduction of the IBX from 1.3 to 0.9 M $Al(NO_3)_3$ in order to reduce IBU losses.

(2) Elimination of dichromate from both first and second cycle scrub streams IAS and IIAS in order to reduce dichromate carry-over in the organic product streams.

(3) Reduction of $HNO_3$ in IAS and IIAS scrub from 0.30 N to 0.05 N $HNO_3$ and addition of 0.05 N $HNO_3$ to the IAX and IIAX organic streams in order to enhance decontamination without excessive losses.

Overall operation of the equipment under these conditions was satisfactory. Overall column losses range from 0.4 - 1.7% and decontamination factors of $2 \times 10^2 - 2 \times 10^4$ and $7 \times 10^2 - 5 \times 10^3$ for $\beta$ and $\gamma$ respectively were obtained dependent on the activity in the feed material. Detailed data is given in

Plutonium Recovery (Continued)

Tables I, II, and III of Appendix II. The activity of the final concentrated (2 liters) product solution is 2-4 x $10^8$ d/min/gm Pu for beta and 3 x $10^4$ mv/gm Pu.

Conditions were altered in the second plutonium cycle of Run No. 8RBP-II to test the modified (hybrid) flowsheet as developed by KAPL, namely, operation of the IIA columns with an acidic extraction section in conjunction with a basic scrub section. Results indicate an improvement by a factor of 3 in both β and γ decontamination and no appreciable change in losses. After operation for 16 hours difficulty occurred in column operations due to formation of ferric hydroxide precipitate at the interface, probably due to insufficient acid at the feed point. Additional experimental tests will be conducted with this type flowsheet prior to start of processing of Chalk River material. Results are given in Appendix II of this report.

2.1 First Cycle Feed Preparation

Feed preparation consisting of adjustment to 1.3 M $Al(NO_3)_3$, 0.3 N $HNO_3$, and 0.10 M $Na_2Cr_2O_7$, followed by filtration, was satisfactory and without incident. Filtration losses were of the order of 0.05 - 0.1%.

2.2 First Cycle Column Operations

Extraction losses in the 1A column continued to be low, averaging 0.01%. As reported last period, high plutonium losses were obtained in the 1B U-Pu partitioning column due to the lack of the back salting effect of uranium in the non-representative feed material. After the first run this loss was reduced to about 0.2% by reducing the concentration of the IBX stream from 1.3 M to 0.9 M $Al(NO_3)_3$. No further difficulties were encountered.

Plutonium Recovery (Continued)

2.3 Second Cycle Feed Preparation

Second cycle feed preparation consisting of adjustment of the IBP to 1.3 M $Al(NO_3)_3$, 0.30 N $HNO_3$, and 0.10 M oxidizing was satisfactory. For this type feed the IIAX was acidified to 0.05 N $HNO_3$ and the IIAS was adjusted to 1.3 M $Al(NO_3)_3$ and 0.05 N $HNO_3$ for optimum decontamination. However, flowsheet conditions were altered in Run 8RBP-II in order to test the effect on decontamination of operation of the extraction column (IIA) under conditions as developed at KAPL, i.e., with an acid extraction section and an acid deficient scrub section. For this run the feed was adjusted to 0.05 N $HNO_3$, 0.10 M oxidizing, and 1.3 M $Al(NO_3)_3$. The IIAS scrub was adjusted to 0.20 N $HNO_3$-deficient, and the organic (IIAX) stream was made 0.35 N $HNO_3$.

2.4 Second Cycle Column Operation

Column operations under acid scrub and extraction conditions were satisfactory with losses of the order of 0.5%. Beta decontamination varied from 6-200 dependent on the activity of the feed material.

An improvement in decontamination by a factor of 3 was observed for $\beta$ and $\gamma$ and extraction losses were 0.2% for the modified flowsheet. After 16 hours of operation under the modified KAPL flowsheet conditions (8RBP-II) column difficulties developed which it is felt were due to the build-up of the ferric hydroxide at the interface of the column. A sample of the organic (IIAP) stream taken at this time showed the presence of the ferric precipitate. It is felt that formation of the ferric ion was caused by insufficient acid in the IIAX stream since the $HNO_3$ equilibrium is very delicate and HETS values

Plutonium Recovery (Continued)

for the IIA column were not accurately known. An assumed HETS value of 2 feet was made for calculation of the acid in the IIAX such that acid conditions would be maintained at the feed point. However small errors in this HETS value would make large differences in the acidity at the feed plate because of the long 205 Pilot Plant columns.

Further tests are planned to establish equilibrium $HNO_3$ conditions for this flowsheet prior to processing of the Chalk River material.

2.5 Product Evaporator

The product evaporator has continued to operate satisfactorily. However, it is planned to alter the evaporator equipment to provide a two stage evaporation in order to flash off the solvent prior to concentration since it has been found that considerable solvent decomposition products form during the concentration of the product.

2.6 Program

Additional equipment cleanouts are in progress to remove residual plutonium from equipment before going into the Chalk River Separations program.

The concentrated product solution from each run will be processed and further purified in the alpha laboratory which is being prepared in the 204 Building.

RADIOCHEMICAL WASTE PROCESSING

Technical - Shank

## 3.0 RADIOCHEMICAL WASTE PROCESSING

### 3.1 Summary

The ORNL Radiochemical Waste Evaporator processing radioactive wastes averaged a 18:1 volume reduction and an overall beta decontamination factor of $2.4 \times 10^3$ during the past period. Laboratory work on a foam level indicator is approximately 50% complete with installation scheduled for April. Bids on the proposed evaporator condensate resin columns have been received but final decisions and awarding of contract are yet to be made. A laboratory test vapor scrubber is scheduled to be installed by April.

### 3.2 Evaporator Operation

Two hundred and seventy-three thousand gallons of mixed waste were processed by the ORNL Radiochemical Waste Evaporator during the period January 22 to February 25, 1950, with an average volume reduction of 18:1. The feed contained 2900 curies of beta activity and discharged 1.2 curies to the Settling Basin to give $2.4 \times 10^3$ decontamination factor for the period. For more details on the evaporator operations, see Appendix III.

### 3.3 Evaporator Studies

The laboratory evaluation of anti-foam agents by T. C. Runion is essentially complete. Larger quantities of the agents which indicated better foam control than the one presently being used have been ordered for field testing. A detailed report of the laboratory evaluation is to be published later.

Radiochemical Waste Processing (Continued)

An attempt to corrolate free $OH^-$ concentrations in the evaporator feed with evaporator operability is being investigated. In conjunction, an analytical procedure has been found which will permit operation determination of the free $OH^-$ concentration in the feed prior to its entry into the evaporator. The procedure and results of the analytical investigation are given in Appendix III.

3.4 Equipment Development

Several laboratory tests are to be conducted on a mock-up of the proposed foam level indicator prior to actual installation. Description and operation of the indicator and proposed laboratory tests are given in Appendix III.

Modifications of the existing low-level activity monitor are waiting further development of the foam level indicator. Control of foam may permit satisfactory monitoring operation without modifications to the present equipment.

3.5 Equipment

Major activities of the radioactive waste processing program are listed below:

1. Prime emphasis will be placed on development and installation of the foam level indicator.
2. A 2" diameter by 6'-0" high cation-anion resin column test system will be installed on the evaporator condensate stream.

Radiochemical Waste Processing (Continued)

3. Fabrication of a Pyrex vapor scrubber containing interchangeable scrubbing sections is expected to be completed and installed the Unit Operations Section by April. Deentrainment efficiencies of various types of scrubbing media will be investigated.
4. Volume reduction vs solids concentration and activity decontamination comparison studies will be started in April.
5. Average evaporator operating cost/pound of condensate will be compiled.

CHALK RIVER SEPARATION

Technical - Sadowski

## 4.0 CHALK RIVER SEPARATION

Approximately 775 grams of Pu will be separated and decontaminated from various levels of neutron irradiated, natural uranium metal. Three shipments of Chalk River fuel material have been received; the remaining two are to be received during March. The additional equipment to be installed to this program, as requested in CR-103, is approximately 50% complete and is scheduled to be completed by March 15. The Pilot Plant start-up date has been set as March 20.

The chemical conditions for Pilot Plant separation have been tentatively set as an ORNL #1 flowsheet for the first cycle and a KAPL flowsheet for the second cycle. The present cell III evaporator is being modified to provide a two stage evaporation; the first stage will flash off the solvent carried in the strip product solution, and the second stage will concentrate the solution to approximately 2 liters. The final product will be further purified in a laboratory cycle.

### 4.1 Schedule and Chemical Conditions

Material will be processed in seven separate batches, of which six will be segregated sections of twenty Chalk River fuel rods, and one will be from specially irradiated Hanford material.

#### 4.11 Pilot Plant Separations Schedule

The startup date for the ORNL 205 Bldg. Pilot Plant has been set as March 20. The history of the feed material and the estimated completion dates for each separation are given in the following table:

Chalk River Separations (Continued)

TABLE 4.1-1

CR Separations Feed Material History

And ORNL Processing Schedule

| Batch | Source | Irradiation in MWD/T Maximum | Irradiation in MWD/T Average | No. of 8" Pieces | Total gm. Pu (Approx.) | Estimated Completion Date |
|---|---|---|---|---|---|---|
| 1 | NRX | 360 | 220 | 40 | 30 | 5/1 |
| 2 | NRX | 610 | 490 | 40 | 70 | 5/10 |
| 3 | NRX | 830 | 720 | 40 | 100 | 5/20 |
| 4 | NRX | 1010 | 920 | 40 | 120 | 5/30 |
| 5 | NRX | 1130 | 1080 | 40 | 140 | 6/10 |
| 6 | NRX | 1205 | 1180 | 60 | 220 | 6/20 |
| 7 | HEW | 800 | 750 | 35* | 95 | 7/1 |

* Actually 70 four-inch slugs.

4.12 Metal Dissolving

Forty slugs, each eight inches in length and weighing eight pounds, will be charged to the dissolver for each of the first five runs. The sixth dissolving will be sixty slugs, and the seventh will be 70 pieces of special Hanford material. The aluminum jackets will be removed by the standard 10% NaOH - 20% $NaNO_3$ method. Sufficient nitric acid will be added to dissolve the metal to give a final dissolver acidity of 0 - 0.1 N. This uranium solution will then be adjusted to feed specifications and will yield sufficient material for a 27 hour column run. Again, nitric acid will be added to the dissolver to dissolve the residual metal heel, together with sufficient chemical UNH to provide material for a 13 hour cleanup run.

Chalk River Separations (Continued)

4.13 First and Second Cycle Flowsheet Conditions

The first cycle U-Pu separation will be made using the ORNL #1 acid deficient flowsheet described in detail in ORNL-463. The second cycle plutonium decontamination combining both portions of the first cycle run will be made using the KAPL flowsheet described in KAPL-244. The conditions for the third cycle laboratory purification have not been established.

4.14 Service Samples and Analyses

Twenty ml. of dissolver solution will be sampled from each batch for storage at ORNL. A 5 ml. dissolver sample of batch 6 for B. B. Cunningham at Berkley, and a 20 ml. dissolver sample of batch 6 for W. M. Manning at ANL will also be taken.

Isotopic analyses of the separated uranium (ICU stream) will be made for each run.

4.15 $Np^{237}$ and F.P. Separations

The first cycle aqueous raffinate (IAW) from each of the seven batches will be stored in new tanks being installed in the North Tank Farm area. This material will be processed for the recovery of $Np^{237}$ and some of the fission products by the ORNL Chemistry Division in Bldg. 706-C (Memo G. W. Parker to J. A. Swartout, C.F. #50-2-27), and other fission products will be separated by the ORNL Operations Division.

Chalk River Separation (Continued)

4.2 Equipment Installation and Modifications

The additional equipment necessary to complete the CR Separations Program includes a modification of the existing product catch tank in Cell III of the Pilot Plant, storage and Transfer facilities for the first cycle aqueous raffinate in the North Tank Farm area, separations facilities in Bldg. 706-C for $Np^{237}$ and fission products, and product purification facilities in Bldg. 204.

Details of the equipment modifications are given in Appendix IV.

4.3 Program

The final two shipments of the CR fuel material will be received during March. The Hanford material will be obtained later. The ORNL Maintenance group is making an intensive effort to complete the installation during the coming period.

The KAPL second cycle Pu flowsheet and the laboratory purification cycle will be investigated further to establish the final flowsheet conditions. The Pilot Plant equipment will be cleansed of residual Pu from the Redox IBP cleanup runs and given a preventative maintenance check.

## ORNL WASTE URANIUM RECOVERY UNIT

Technical - Stewart

### 5.0 ORNL WASTE URANIUM RECOVERY UNIT

The program for the construction and operation of the ORNL Metal Recovery Plant has progressed through the stages of cost evaluation and preliminary equipment requirements. The tentative building layout and design criteria have been prepared for the Engineering Department.

#### 5.1 Design Status - Building

The preliminary layout of the building has been prepared for the Engineering Department, and the design criteria will be submitted with the Work Order to start engineering studies the first week in March. The building structure will consist of a bank of five cells, along one side of the building, two of which will be the hot cells requiring 2 feet of concrete shielding. The remaining cells will serve as isolated areas, and will be constructed of concrete block. Three other areas adjacent to the cells will be for the make-up area, change room and control room. The overall dimensions of the building will be 57 ft. long by 50 ft. wide. The building will be a single story structure having 16' head room in the make-up areas and a 10' ceiling in the control room and change room. General building design is to conform with the criteria established for ORNL Plan H construction and renovation.

ORNL Waste Uranium Recovery Unit (Continued)

5.11 Design Status - Equipment

The majority of the equipment pieces have been sized for capacity for the tentative flowsheet conditions, including a IB column for the recovery of the plutonium.

The design provides for 48 hour column runs at the rate of 750 lbs. of uranium metal to be recovered per day as 2 M UNH. Nitric acid recovery is considered for the AW stream, and the equipment is to be sized to provide for the handling of the fumes from the boildown of the CU stream. Solvent treatment will consist of continuous washing of the solvent with 2 M caustic in a packed column. A solvent inventory representing only three hours of operation will be adequate.

5.2 Development - Chemical and Mechanical

A chemical flowsheet, sufficiently firm for preliminary design, has been developed by the Laboratory Section of the Chemical Technology Division.

The major equipment problem lies with the study of equipment to give the volume reduction of the column streams so that the recovery of reasonably activity-free nitric acid is feasible. Studies concerning the recovery of nitric acid are now under way by the Unit Operations Section.

The sludge removal problem is being attacked by the construction of a traverse mechanism, and the types of pumps which may best remove the sludge from the tanks are being investigated.

ORNL Waste Uranium Recovery Unit (Continued)

5.3 Program

The program consists of the following basic points:

1. Establishing a flowsheet about which equipment may be frozen.
2. Investigation as to the availability, cost and delivery of equipment requirements.
3. Continuing of the work on sludge removal and transfer.
4. Following building design by the Engineering Dept.
5. Adoption of a system for nitric acid recovery.
6. Establishing design criteria for instruments and piping.

## 25 RECOVERY

Technical - Rom

### 6.0 25 RECOVERY

The terminal 25 Recovery Process Report is approximately 50% complete.

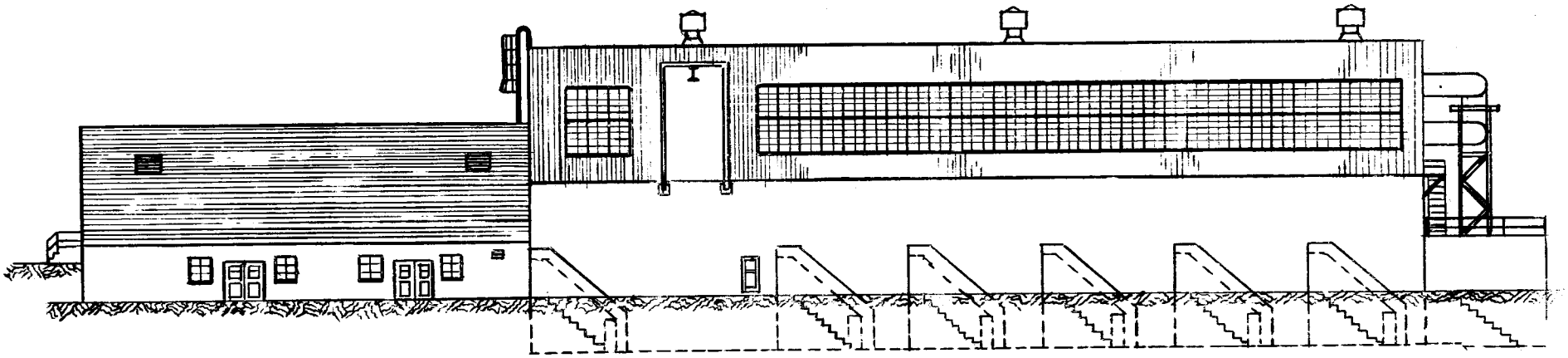
Messrs O. Carpenter and A. de la Garza of the K-25 Statistical Section continued to render valuable assistance in the effort to determine the limits of error to be used in reporting the enriched uranium material balance for the 25 process verification runs. This work is now essentially complete and is scheduled to be issued as a separate document in the coming period.

The final purification of the 2400 grams of product from the "25 Verification Runs" has been essentially completed by the Laboratory Section.

### 7.0 MISCELLANEOUS ADMINISTRATIVE

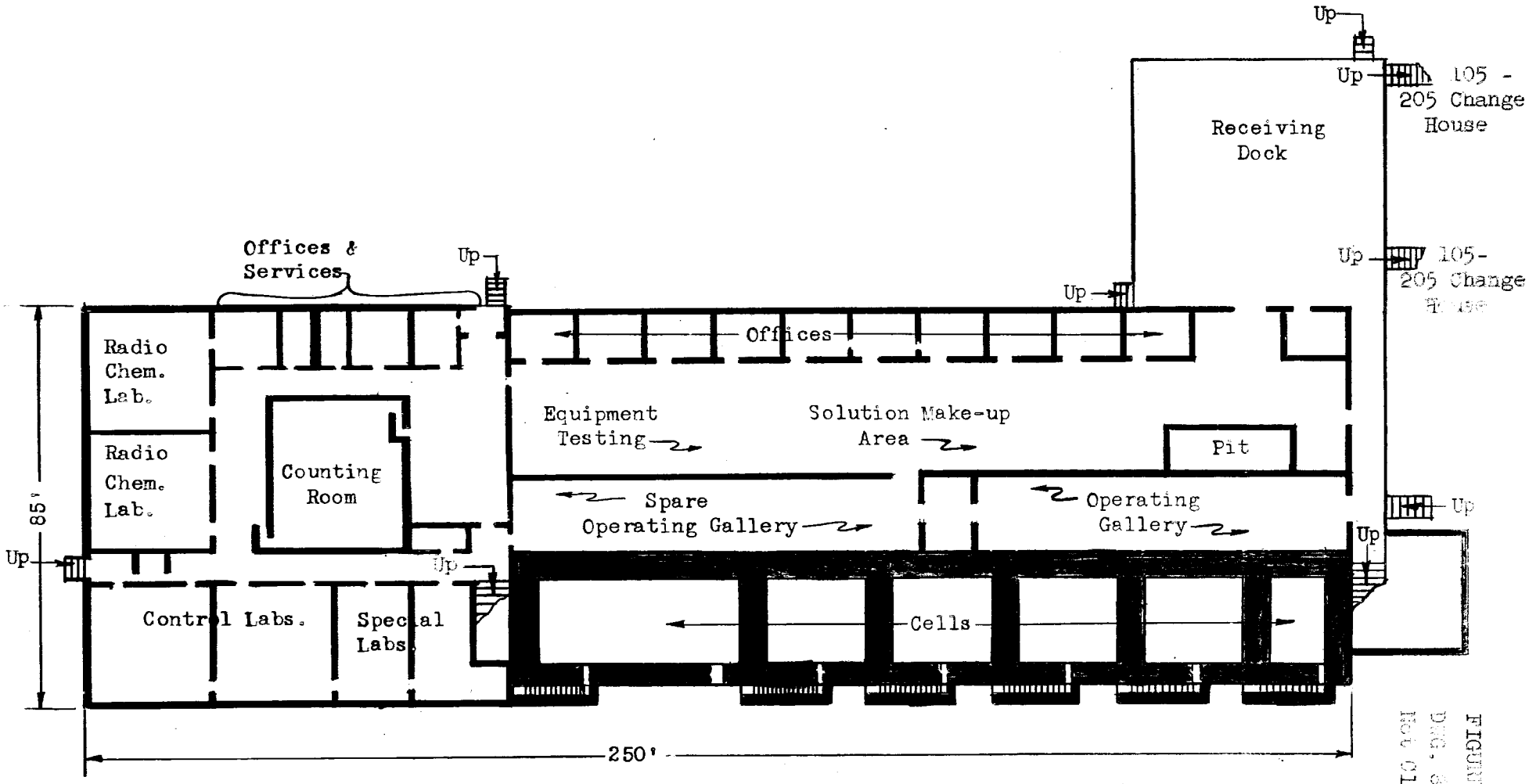
Ground was broken by J. A. Jones on February 13, 1950, for permanentization of the 205 Building. This permanentization includes renovation, expansion and supplementing of the existing facilities to permit the building to be used exclusively by the Pilot Plants Section and related service groups. An architectural sketch showing the elevation and first floor plan of the building is given in Figures 1 and 2. The structure, made up of two floors and a basement, is scheduled for completion July 1, 1950, and will provide 6,800 sq. ft. of laboratory analytical space, 11,300 sq. ft. of operating space, 5,500 sq. ft. of service area, and 5,800 sq. ft. of equipment testing and office space.

R. M. Burnett, Research Operator B, was transferred to the Reactor Technology Division effective February 13, 1950.



SOUTH ELEVATION OF 205
PILOT PLANT BLDG.

FIGURE I
DWG. 8783
Not Classified




PLAN OF 205 PILOT PLANT BLDG.

FIGURE II
DWG. 6784
Not Classified

## APPENDIX I

## 23 PROCESS DEVELOPMENT

### A. Beta and Gamma Background of $U^{233}$

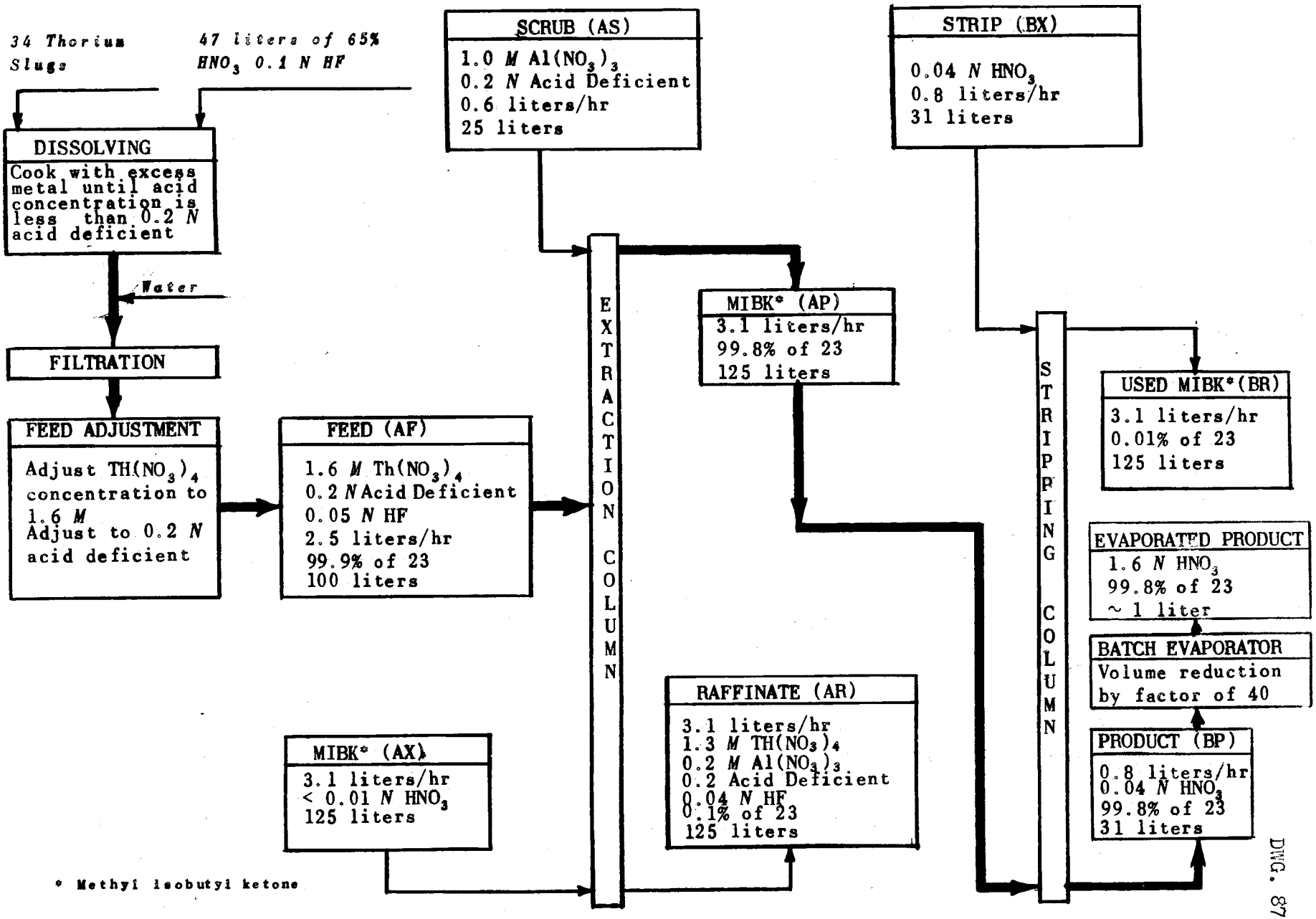
The activity of 23 was found to be $3 \times 10^3$ to $6 \times 10^3$ conversion electron and X-ray counts (apparent beta counts) per minute per mg of 23 at 10% geometry, depending on the amount of solid on the plate, and 0.6 mv of gamma per mg of 23 measured in an ion chamber that was used for all 23 Pilot Plant gamma determinations. These values were determined by measurements on a sample of 23 that was essentially free of fission products and Pa activity as determined by Ru, Zr, Ce and Pa analyses. This sample of 23 was obtained by re-extracting one gram of 23 from Pilot Plant product three times with diethyl ether and once with diisopropyl ether. The background activities given above are considered preliminary values, but are sufficiently accurate to be used in calculating decontamination data. A more precise determination of 23 background is now being made by the Chemistry Division. Additional data concerning 23 background are as follows:

(1) Beta and gamma activity will grow into freshly extracted 23 at a maximum rate of 2 $\beta$ c/m per mg of 23 per day because the first daughter, $Th^{229}$, has a half-life of $7 \times 10^3$ years.

(2) The beta counting background from freshly extracted 23 is a factor of $10^3$ higher than from freshly extracted natural uranium, and about a factor of 4 higher than uranium in equilibrium with its daughters.

(3) The gamma activity of freshly extracted 23 is $10^3$ times that of freshly extracted natural uranium, and 100 times that of uranium in equilibrium with its daughters.

[redacted]




* Methyl isobutyl ketone

**23 PROCESS PILOT PLANT CHEMICAL FLOWSHEET**

DWG. 8785

[redacted]

TABLE I-1

ORNL 23 Pilot Plant

Summary of Dissolving Data

| Dissolving Number | % $HNO_3$ | Dissolving Time, Hrs. | Moles $HNO_3$/ Moles Th | Final Sp.Gr. | Final[1] Acidity, N | Th Metal Heel, Kg. |
|---|---|---|---|---|---|---|
| 1T | 65 | 26 | 5.9 | 2.03 | 0.14 b | 39 |
| 2T | 65 | 30 | 5.8 | 2.04 | 0.02 b | 44 |
| 3T-1 | 65 | 33 | 7.3 | -- | 0.10 b | 47 |
| 3T-2 | 65 | 23 | 6.5 | 2.10 | 0.20 b | 37 |
| 4T | 65 | 31 | 5.7 | 2.01 | 0.25 b | 30 |
| 5T | 65 | 28 | 5.7 | 2.06 | 0.06 b | 75 |
| 6T | 65 | 33 | 6.3 | 2.15 | 0.40 b | 114 |
| 7T | 65 | 30 | 5.5 | 2.01 | 0.38 b | 64 |
| 8T | 66 | 29 | 5.0 | 2.12 | 1.1 b | 65 |
| 9T | 65 | 29 | 5.7 | 2.08 | 0.42 b | 70 |
| 10T | 66 | 28 | 5.6 | 2.05 | 0.44 b | 57 |

(1) b denotes acid deficiency



[redacted]

[redacted]

TABLE I-2

ORNL 23 Pilot Plant

Gross Fission Product Activity and Decontamination Factor

| Run No. | Feed $Th(NO_3)_4$ Conc. Molar | Gross Activity of the feed | | | Sp. Fission Product Activity of Feed(2) | | Sp. Activity of BP β cts. or γ mv per mg of 23 | | | | Gross Fission Product Decont. Factor | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Beta x $10^{-7}$ | mv γ x $10^{-3}$ | 23α x $10^{-6}$ | β cts/mg 23 x $10^{-7}$ | γ mv/mg 23 x $10^{-4}$ | Gross β x $10^{-3}$ | FP β x $10^{-3}$ (3) | Gross Gamma | F.P.(3) Gamma | Beta | Gamma |
| 9T | 1.26 | 1.1 | 2 | 1.5 | 8 | 1.5 | 4 | 1 | 0.7 | 0.4 | $6 \times 10^4$ | $4 \times 10^4$ |
| 8T | 1.46 | 1.4 | 2 | 1.8 | 8 | 1.5 | 5 | 2 | 1.2 | 0.6 | $4 \times 10^4$ | $3 \times 10^4$ |
| 3T | 1.52 | 30 | 15 | 1.0 | 8 | (1) | 5 | 2 | 1.1 | 0.6 | $4 \times 10^4$ | (1) |
| 1T | 1.60 | 0.3 | 0.6 | 0.92 | 3 | 0.7 | 5 | 2 | 1.0 | 0.5 | $1.4 \times 10^4$ | $1.4 \times 10^4$ |
| 2T | 1.60 | 0.4 | 1 | 0.97 | 4 | 1.1 | 5 | 2 | 1.1 | 0.5 | $1.9 \times 10^4$ | $2 \times 10^4$ |
| 4T | 1.60 | 2.0 | 8 | 1.7 | 9 | 2.5 | 5 | 2 | 1.9 | 1.3 | $4 \times 10^4$ | $2 \times 10^4$ |
| 10T | 1.60 | 1.4 | 2 | 2.1 | 7 | 1.2 | 5 | 2 | 1.0 | 0.5 | $3 \times 10^4$ | $3 \times 10^4$ |
| 11T | 1.6 | 1.4 | 2 | 2.1 | 7 | 1.3 | 5 | 2 | 0.9 | 0.5 | $3 \times 10^4$ | $3 \times 10^4$ |
| 12T | 1.6 | 1.5 | 3 | 2.2 | 8 | 1.4 | 4 | 1 | 0.9 | 0.4 | $8 \times 10^4$ | $4 \times 10^4$ |
| 5T | 1.80 | 2.0 | 4 | 2.0 | 8 | 1.0 | 34 | 31 | 7.9 | 7.3 | $2 \times 10^3$ | $1.3 \times 10^3$ |
| 7T | 1.86 | 1.8 | 4 | 2.1 | 9 | 1.9 | 7 | 4 | 2.2 | 1.6 | $2 \times 10^4$ | $1.2 \times 10^4$ |

(1) Insufficient Data

(2) Obtained by subtracting Pa activity from gross activity and dividing by 23 concentration

(3) Obtained by subtracting 23 and Pa activity from gross activity and dividing by 23 concentration.



[redacted]

[redacted]

TABLE I-3

ORNL 23 Pilot Plant

Specific Fission Product, Pa and Th

Decontamination Factors

| Run Number | 9T | 8T | 1T | 2T | 3T | 4T |
|---|---|---|---|---|---|---|
| **Ru Beta** | | | | | | |
| $Th(NO_3)_4$ Conc. in Metal Feed - M | 1.26 | 1.46 | 1.60 | 1.60 | 1.52 | 1.60 |
| AF cts/min/mg of 23 | $2 \times 10^6$ | $2 \times 10^6$ | $8 \times 10^5$ | $1.3 \times 10^6$ | $1.6 \times 10^6$ | $2 \times 10^6$ |
| BP cts/min/mg of 23 | 500 | 500 | 900 | 900 | 300 | $1.1 \times 10^3$ |
| Decont. Factor | $4 \times 10^3$ | $4 \times 10^3$ | 900 | $1.4 \times 10^3$ | $5 \times 10^3$ | $2 \times 10^3$ |
| **Zr Beta** | | | | | | |
| AF cts/min/mg of 23 | $1.1 \times 10^5$ | $1.3 \times 10^5$ | $8 \times 10^4$ | $4 \times 10^4$ | $8 \times 10^6$ | $5 \times 10^5$ |
| BP cts/min/mg of 23 | 30 | 50 | 70 | 60 | 500 | 20 |
| Decont. Factor | (1) | (1) | (1) | (1) | $1.7 \times 10^4$ | $2 \times 10^4$ |
| **Ce Beta** | | | | | | |
| AF cts/min/mg of 23 | $5 \times 10^5$ | $5 \times 10^7$ | $1.6 \times 10^7$ | $3 \times 10^7$ | $4 \times 10^7$ | $5 \times 10^7$ |
| BP cts/min/mg of 23 | 80 | 70 | 150 | 400 | 130 | 200 |
| Decont. Factor | $5 \times 10^5$ | $5 \times 10^5$ | $1 \times 10^5$ | $1 \times 10^5$ | $3 \times 10^5$ | $2 \times 10^5$ |
| **Pa Beta** | | | | | | |
| AF cts/min/mg of 23 | $1.1 \times 10^6$ | $3 \times 10^6$ | $<10^4$ | $<10^4$ | $4 \times 10^8$ | $6 \times 10^7$ |
| BP cts/min/mg of 23 | 40 | 200 | – | – | 400 | 500 |
| Decont. Factor | (1) | (1) | (1) | (1) | $1 \times 10^6$ | $1 \times 10^5$ |
| **Thorium** | | | | | | |
| Decont. Factor | $2 \times 10^5$ | $2 \times 10^4$ | $1 \times 10^4$ | $1 \times 10^4$ | $3 \times 10^4$ | $7 \times 10^4$ |

(1) Decontamination factor not reliable due to low activity. Except for 3 ORNL Th metal slugs (cooled 6 weeks) all metal used had cooled 24 months or longer.

[redacted]

[redacted]

TABLE I-3 (Continued)

| Run Number | 10T | 11T | 12T | 5T | 7T |
|---|---|---|---|---|---|
| Ru Beta | | | | | |
| $Th(NO_3)_4$ Conc. in Metal Feed - M | 1.60 | 1.6 | 1.6 | 1.80 | 1.86 |
| AF cts/min/mg of 23 | $2 \times 10^6$ | $2 \times 10^6$ | $2 \times 10^6$ | $3 \times 10^6$ | $3 \times 10^6$ |
| BP cts/min/mg of 23 | $1.1 \times 10^3$ | $1.5 \times 10^3$ | 800 | $3 \times 10^4$ | $4 \times 10^3$ |
| Decont. Factor | $2 \times 10^3$ | $1.4 \times 10^3$ | $3 \times 10^3$ | 100 | 600 |
| Zr Beta | | | | | |
| AF cts/min/mg of 23 | $1.2 \times 10^5$ | $9 \times 10^4$ | $1.7 \times 10^5$ | $9 \times 10^5$ | $2 \times 10^5$ |
| BP cts/min/mg of 23 | 6 (2) | 3 (2) | 8(2) | 50 | 30 |
| Decont. Factor | $2 \times 10^4$ | $3 \times 10^4$ | $2 \times 10^4$ | $2 \times 10^4$ | (1) |
| Ce Beta | | | | | |
| AF cts/min/mg of 23 | $5 \times 10^7$ | $5 \times 10^7$ | $6 \times 10^7$ | $5 \times 10^7$ | $5 \times 10^7$ |
| BP cts/min/mg of 23 | 70 | 140 | 70 | 400 | 100 |
| Decont. Factor | $5 \times 10^5$ | $3 \times 10^5$ | $8 \times 10^5$ | $1 \times 10^5$ | $5 \times 10^5$ |
| Pa Beta | | | | | |
| AF cts/min/mg of 23 | $7 \times 10^5$ | $7 \times 10^5$ | $1.6 \times 10^5$ | $3 \times 10^7$ | $8 \times 10^5$ |
| BP cts/min/mg of 23 | 30 | 50 | 6 (2) | 600 | 200 |
| Decont. Factor | (1) | (1) | $3 \times 10^4$ | $5 \times 10^4$ | (1) |
| Thorium | | | | | |
| Decont. Factor | $2 \times 10^5$ | $3 \times 10^5$ | $2 \times 10^5$ | $1 \times 10^5$ | $3 \times 10^4$ |

(1) Decontamination factor not reliable due to low activity in the feed. Except for 3 ORNL Th metal slugs (cooled 6 weeks) all metal used had cooled 24 months or longer.

(2) Analysis made on concentrated BP.



[redacted]

[redacted]

TABLE I-4

ORNL 23 Pilot Plant

23 Losses

| Run Number | $Th(NO_3)_4$ Conc. in AF (Molar) | 23 Concen. in AR mg/liter | 23 Loss % | | |
|---|---|---|---|---|---|
| | | | AR | BR | Dissolver Residual (1) |
| 1T | 1.6 | 0.093 | 0.14 | .002 | <0.001 |
| 2T | 1.5 | 0.10 | 0.18 | .004 | 0.05 |
| 3T | 1.6 | 0.075 | 0.12 | .009 | 0.06 |
| 4T | 1.6 | 0.093 | 0.08 | .002 | 0.02 |
| 5T | 1.8 | 0.12 | 0.10 | .001 | 0.07 |
| 6T | 1.9 | 0.13 | 0.06 | .001 | 0.10 |
| 7T | 1.8 | 0.14 | 0.10 | .003 | 0.09 |
| 8T | 1.4 | 0.057 | 0.08 | .002 | 0.06 |
| 9T | 1.2 | 0.066 | 0.06 | 0.002 | 0.24 |
| 10T | 1.6 | 0.083 | 0.05 | 0.001 | 0.08 |
| 11T | 1.6 | 0.097 | 0.07 | 0.006 | 0.05 |
| 12T | 1.6 | 0.088 | 0.06 | 0.003 | 0.02 |

(1) This is the fraction of 23 lost with the coating removal solution and is due entirely to insufficient washing of the dissolver after dissolving.



[redacted]

[redacted]

TABLE I-5

ORNL 23 Pilot Plant

Isotopic Purity of Product

| Run Number | 23% of Total Uranium | | | % of Other Isotopes | | | |
|---|---|---|---|---|---|---|---|
| | Y-12 Mass Spectrograph | By comparison of 23 Radiochemical Analysis with | | | | | |
| | | Colorimetric | Potentiometric | U-234 | U-235 | U-236 | U-238 |
| 1T | 98.35 | 95.9 | 94.5 | 0.20 | 0.29 | 0.04 | 1.11 |
| 2T | | 95.2 | - | | | | |
| 3T | | 87.1 | - | | | | |
| 4T | | 87.0 | - | | | | |
| 5T | 98.73 | 88.9 | 91.4 | 0.30 | 0.14 | 0.07 | 0.76 |
| 6T | | 94.3 | - | | | | |
| 7T | | 94.4 | - | | | | |
| 8T | 98.36 | 94.3 | - | 0.29 | 0.66 | 0.00 | 0.68 |
| 9T | 98.27 | 87.6 | 90.2 | 0.30 | 0.68 | 0.00 | 0.81 |
| 10T | | 87.7 | | | | | |

[redacted]

[redacted]

## APPENDIX II

### PLUTONIUM RECOVERY FROM REDOX SOLUTIONS

TABLE II-1

**ORNL Pilot Plant Plutonium Recovery**

**Two Cycle Decontamination and Loss Data**

| Run No. | Decontamination | | Column Losses-% | Overall Losses-% | Product Activity | |
|---|---|---|---|---|---|---|
| | Gross β | Gross γ | | | β dis/min/gm Pu | γ Mv/gm Pu |
| 1RBP | $1.8 \times 10^2$ | $6.5 \times 10^2$ | 9.5 (1) | 9.8 (1) | $4.0 \times 10^8$ | $6.1 \times 10^4$ |
| 2RBP | $2.5 \times 10^2$ | $3.7 \times 10^3$ | 14.5 (2) | 14.5 (2) | $2.1 \times 10^8$ | $3.3 \times 10^3$ (4) |
| 3RBP | $4.1 \times 10^2$ | $2.2 \times 10^3$ | 0.7 | 0.8 | $4.2 \times 10^8$ | $3.4 \times 10^4$ |
| 4RBP | | No Second Cycle Run Made | | | | |
| 5RBP | $6.6 \times 10^2$ | $3.8 \times 10^3$ | 1.7 | 2.0 | $3.3 \times 10^8$ | $2.1 \times 10^4$ |
| 6RBP | | No Second Cycle Run Made | | | | |
| 7RBP | $6.7 \times 10^3$ | $4.9 \times 10^3$ | 0.4 | 0.5 | $4.5 \times 10^8$ | $8.8 \times 10^4$ |
| 8RBP | $1.7 \times 10^4$ | $9.7 \times 10^3$ | 0.4 | 24.4 (3) | $1.3 \times 10^8$ | $1.5 \times 10^4$ |

(1) Essentially entire amount due to high IBU (partitioning) loss which is not a true loss since most (8.7%) was returned to process.

(2) Caused by lack of oxidant in feed to second cycle extraction column.

(3) Large portion (22.7%) due to flooding of evaporator as a result of column difficulties due to ferric build-up in column. This is not a true loss since it was re-evaporated with cleanout run #2 (CO#2).

(4) Large amount of purified plutonium added to Run 2.



[redacted]

[redacted]

TABLE II-2

ORNL Pilot Plant Plutonium Recovery

**First Cycle Decontamination and Loss Data**

| Run No. | Gross β | Gross γ | Filtration Loss - % | IAW Loss - % | IBU Loss - % | ICW Loss - % | Material Balance |
|---|---|---|---|---|---|---|---|
| 1RBP-I | 16 | 21 | 0.12 | 0.006 | 8.70 (1) | 0.800 | 98 |
| 2RBP-I | 44 | 87 | 0.01 | 0.003 | 1.95 | 0.035 | 98 |
| 3RBP-I | 17 | 49 | 0.01 | 0.017 | 0.18 | 0.066 | 95 |
| 4RBP-I | 78 | 150 | 0.07 | 0.010 | 0.160 | 0.009 | ---(2) |
| 5RBP-I | 56 | 110 | 0.02 | 0.009 | 0.230 | 0.009 | 97 |
| 6RBP-I | 5 | 18 | 0.04 | 0.004 | 0.34 (3) | 0.002 | 99 |
| 7RBP-I | 370 | 400 | 0.01 | 0.014 | 0.34 (3) | 0.005 | 130 |
| 8RBP-I | 350 | 290 | 0.12 | 0.032 | 0.18 | 0.011 | 77 |
| CO #1 | - | - | 0.07 | 0.023 | 0.03 | 0.006 | 124 |
| CO #2 | - | - | - | 0.250 | 1.430 | 0.063 | 570 |

(1) Recycled thru first cycle. No loss to process.

(2) IBP contaminated with U and recycled thru first cycle with 6RBP-I.

(3) IBP from 6RBP-I and 7RBP-I combined and processed together in second cycle.



[redacted]

[redacted]

TABLE II-3

ORNL Pilot Plant Plutonium Recovery

Second Cycle Decontamination and Loss Data

| Run No. | Gross β | Gross γ | IIAW Loss-% | IIBW Loss-% | Prod. Evap. Loss - % | Material Balance-% |
|---|---|---|---|---|---|---|
| 1RBP-II | 12 | 30 | 0.002 | 0.001 | 0.14 | 101 |
| 2RBP-II | 6.4 | 42 | 12.50 | 0.001 | 0.02 | 102 |
| 3RBP-II | 240 | 45 | 0.44 | 0.002 | 0.27 | 98 |
| 4RBP-II | - | - | - | - | - | - |
| 5RBP-II | 120 | 33 | 1.4 | 0.001 | 0.14 | 103 |
| 6RBP-II | - | - | - | - | - | - |
| 7RBP-II | 18 | 12 | 0.06 | 0.001 | 0.06 | 105 |
| 8RBP-II | 47 | 34 | 0.21 | 0.002 | 22.7(1) | 91 |
| CO #1 | - | - | 0.09 | 0.001 | 2.7 | 280 (3) |
| CO #2 | - | - | 0.19 | 0.044 | 34.7(2) | 190 (4) |

(1) High loss due to flooding of evaporator which was caused by column operation difficulties resulting from formation of ferric precipitate in column. No loss to process. Recycled and re-evaporated with CO #2.

(2) Re-evaporated - no process loss.

(3 & 4) High material balance due to cleanout of heels in tanks and columns.



[redacted]

## APPENDIX III

## RADIOCHEMICAL WASTE PROCESSING

### A. Evaporator Operation

The installation and operation of the ORNL Radiochemical Waste Evaporator has permitted doubling the discharge volume of the Settling Basin while decreasing the β curies of activity discharged by a factor of 15-20. The increased Settling Basin discharge has increased storage space for current wastes, thus permitting greater control on the amount of activity discharged. Table III-1 lists the results of Tank Farm Waste handling during the past eleven months. A portion of Table III-1 is represented graphically in Fig. III-1.

### B. Evaporator Studies

Feed samples from six evaporator runs were submitted for base analysis, by four methods, in an attempt to develop a field analysis procedure which will permit an operator to determine the free $OH^-$ concentration in the feed. Table III-2 gives the results of these analyses with the procedures. The data given indicate that the $BaCl_2$ precipitation followed by the potentiometric titration with HCl (procedure 3) is the more accurate for free $OH^-$ determination. Direct titration without a prior precipipitation of the sulfates, phosphates, carbonates, etc., will give a total base analysis. A comparison of procedures 3 and 4, Table III-2, indicates that the volumetric procedure (4) is possibly within analytical accuracy and may therefore enable the volumetric procedure to be used in the field. Further determinations will be made to ascertain if the variation between procedures 3 and 4 remain within the limits $\pm$ 0.1 N.

C. Equipment Development

Foam Level Indicator

The proposed evaporator foam level indicator consists essentially of two co-axial sets of probes inserted into the vapor space in the evaporator with leads connecting to a recording instrument. Each co-axial probe consists of a perforated pipe surrounding a small rod. The size ratio of pipe to rod is determined by the physical properties of the fluid to be measured, i.e., sufficient size to permit the fluid to rise and fall inside the perforated pipe in a manner similar to the main vessel. The maximum length of the probe appears to be approximately 5'-0".

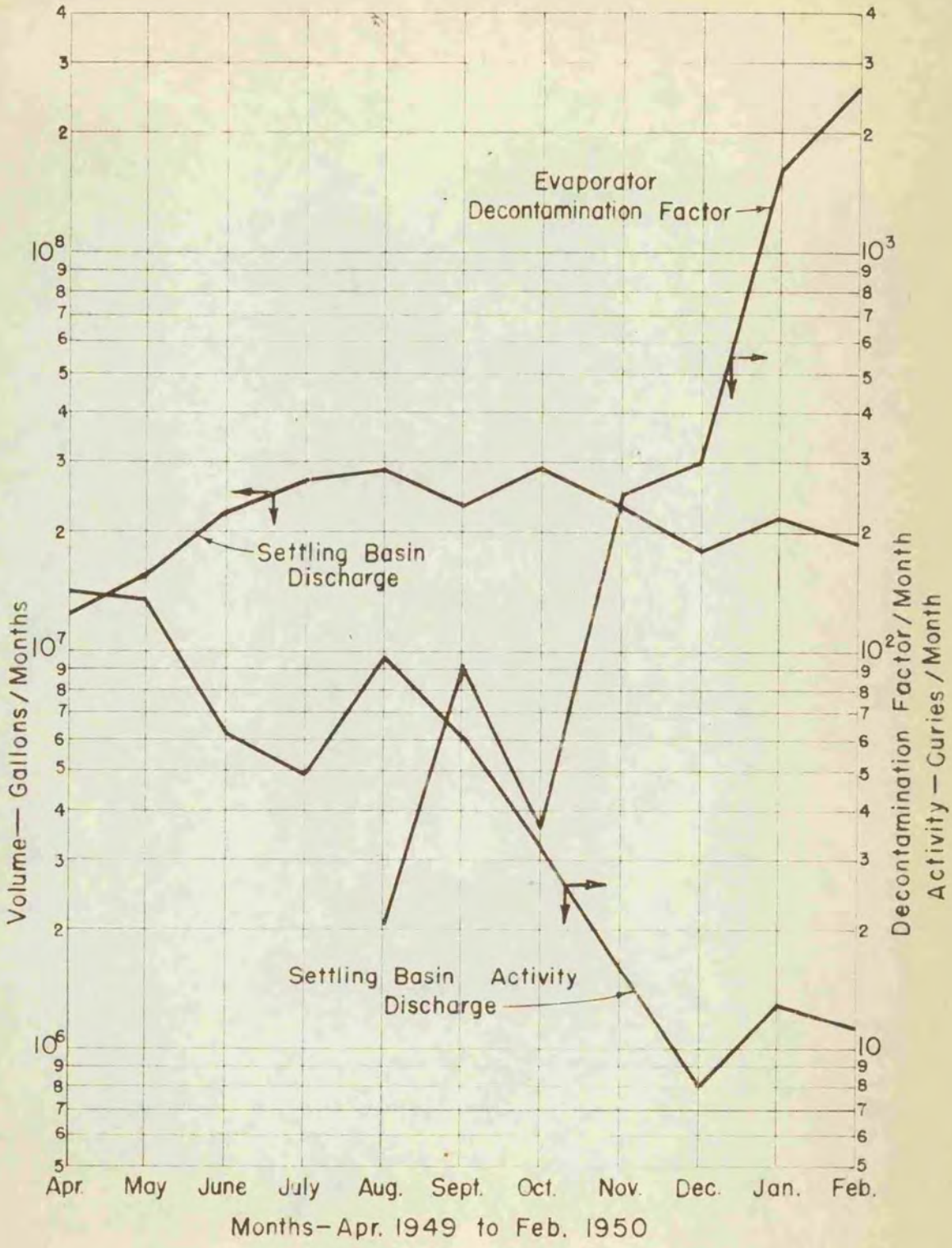
A variable frequency current is induced through one lead at a reasonant frequency which is automatically set by the sending device. As a conducting fluid rises or falls between the pipe and rod, shorting the covered portion of the probe, a change in the length of current travel and a change in the frequency results. The sending device automatically adjusts the induced frequency to reasonance and records the change as inches fluid level.

The development work by K. H. Kline, Instrument Development Group, has shown that there is a harmonic effect while using one co-axial probe. Two probes, operating at slightly different frequency, eliminates this effect. It has also been shown that the measured fluid must be equal to or greater than 0.05 N $H_2SO_4$ solution in conductivity to permit reproducible operation.

Laboratory tests, prior to the installation of the indicator, will consist of conductivity and physical property investigations. The test installation will consist of a 3'-0" diameter by 3'-0" height tank, located under a hood, in which evaporator feed solutions may be boiled to give representative foams. Tests will be made on this foam to ascertain if the proposed indicator will operate.

[redacted]

FIGURE IV




O.R.N.L. RADIOCHEMICAL WASTE EVAPORATOR

[redacted]

TABLE III-1

ORNL Radiochemical Waste Evaporator and Settling Basin Operation

| Date | Evaporator Feed | | Evaporator Cond. | | Curie Decont. Factor | Settling Basin | | |
|---|---|---|---|---|---|---|---|---|
| | Volume Gallons | Curies Gross β | Volume Gallons | Curies Gross β | | Volume Gallons | Curies Gross β | % From Evap. |
| Apr.-49 | (1) | (1) | (1) | (1) | (1) | $12.5 \times 10^6$ | 146 | (1) |
| May-49 | (1) | (1) | (1) | (1) | (1) | $16.7 \times 10^6$ | 137 | (1) |
| June-49 | 130,000 | (2) | 127,000 | (2) | (2) | $22.6 \times 10^6$ | 63 | (2) |
| July-49 | 230,000 | (2) | 206,000 | (2) | (2) | $27 \times 10^6$ | 50 | (2) |
| Aug.-49 | 210,000 | 730 | 190,000 | 38.5 | 21 | $28.8 \times 10^6$ | 98 | 39.3 |
| Sept.-49 | 260,000 | 935 | 220,000 | 12.5 | 92 | $23.4 \times 10^6$ | 62 | 20.2 |
| Oct.-49 | 250,000 | 718 | 240,000 | 20.9 | 36 | $29 \times 10^6$ | 32 | 65.3 |
| Nov.-49 | 290,000 | 1800 | 270,000 | 7.8 | 250 | $23 \times 10^6$ | 16 | 48.7 |
| Dec.-49 | 250,000 | 578 | 230,000 | 2.1 | 300 | $18 \times 10^6$ | 8 | 26.3 |
| Jan.-50 | 157,000 | 1346 | 151,000 | .88 | $1.6 \times 10^3$ | $22 \times 10^6$ | 13 | 6.8 |
| Feb.-50 | 247,000 | 2705 | 229,000 | 1.05 | $2.6 \times 10^3$ | $19 \times 10^6$ | 11.2 | 9.4 |

(1) Evaporator operation started June 1949.

(2) Curies in feed and condensate unavailable for June and July



[redacted]

[redacted]

TABLE III-2

Base Analyses of Six Evaporator Feed Samples

Four Methods

| Run | Type Feed (5) | Oxalate (1) | Normality Base | | |
|---|---|---|---|---|---|
| | | | Potentio-metric (2) | Pot. $BaCl_2$ (3) | Volume $BaCl_2$ (4) |
| 179 | MW | .59 | .42 | .15 | .20 |
| 180 | MW | .60 | .39 | .12 | .19 |
| 181 | MW | .52 | .35 | .12 | .18 |
| 191 | N | .21 | .22 | .01 | .03 |
| 192 | N | .45 | .30 | .09 | .13 |
| 193 | N | .47 | .34 | .05 | - |

(1) Base neutralized with an excess of HCl. Potassium Oxalate added to a pH of 4.5 and solution titrated to a polentiometric end-point with NaOH.

(2) Solution titrated directly to a potentiometric end-point with HCl.

(3) Sulfates, phosphate, carbonates precipitated with a 10% $BaCl_2$ solution and titrated to a potentiometric end-point with HCl.

(4) Same as 4 except solution titrated to a phenophathalien end-point with HCl.

(5) MW-Metal Waste supernate
N-Normal ORNL waste

[redacted]

[redacted]



## APPENDIX IV

## CR SEPARATIONS

### A. 205 Pilot Plant Equipment Modifications

#### Modifications to the Cell III Product Catch Tank

The present Cell III catch tank for the IE column strip solution is being modified to permit flashing off the dissolved hexone in the strip product (IIBP) solution. This tank will be provided with a steam heated jacket and a condenser; the condensate will drain to a condensate catch tank, P3. The collected product solution will then drain by gravity to the evaporator, P2, and will be concentrated to approximately 2 liters.

#### Storage and Transfer Facilities for the First Cycle Aqueous Raffinate

Three, 2000 gallon stainless steel tanks are being installed in the North Tank Farm area to permit storage of the aqueous raffinate from the Pilot Plant and also temporary storage of 706-C raffinate after removal of $Np^{237}$ and fission products. An additional tank is being installed for the ORNL Operations Division to permit independent collection of Pilot Plant raffinate. These tanks are provided with transfer lines to the Operations Division Separation facilities in the South Tank Farm area, and also to W-5 tank.

The 706-C facilities will include processing equipment in a cell bank, and product storage tanks located outside the building in a concrete pit.

[redacted]

APPENDIX IV (Continued)

Product Purification Facilities in the 204 Bldg.

The 204 Bldg. equipment, which formerly was used for plutonium isolation, has been removed, and the building has been decontaminated. The contaminated wall and ceiling panels were removed and replaced by new panels. The walls and ceiling will be finished with white enamel covered by clear cocoon. The building will consist of three rooms, two containing hoods and dry boxes, and the third room containing alpha counters.

The north room will have two 8 ft. long stainless steel hoods, of the latest ORNL design, and two dry boxes, one 8 ft. long and one 32 inches long. Each dry box will be interconnected with a hood by an air lock. The south room will contain a 32 inch stainless steel dry box interconnected to an 8 foot hood and also a wooden dry box purchased from an outside vendor. The building will be air conditioned, and the exhaust air from the hoods and dry boxes will be filtered through CWS filter paper before being discharged to the 205 Bldg. stack.