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ORNL 715
Chemistry-Separation
Processes for Plutonium
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SUMMARY REPORT FOR THE 23 PILOT PLANT PROGRAM

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CHEMICAL TECHNOLOGY DIVISION

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0.0 Abstract

This report summarizes the results of the Pilot Plant Development at Oak Ridge National Laboratory of a continuous solvent extraction process for the isolation of U^{233} from irradiated thorium metal.

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1.0 Introduction

Uranium-233 is a fissionable material produced by the neutron irradiation of thorium metal. In an Uranium-233 (23) production cycle, the thorium after irradiation must be processed to separate chemically the 23 from thorium and fission products. Experimentation by the Chemistry Division of ANL and the Chemistry and Chemical Technology Divisions of ORNL showed solvent extraction to be the most promising method of effecting this separation. Subsequently, four solvent extraction processes were developed. These processes consisted of one solvent extraction cycle in large, moderately shielded equipment and a laboratory scale clean-up and concentration step with only light shielding. Continuous solvent extraction in packed columns was chosen for the large scale extraction because of operational simplicity and economy. The Chemical Technology Division of ORNL then tested and compared four processes on a semi-works scale. On the basis of this work, the 23 Pilot Plant was designed and constructed during 1948 and 1949.

The purpose of the 23 Pilot Plant was to obtain the engineering data necessary for the design and operation of a 23 plant, and to recover about 300 grams of 23 produced at Hanford for experimental purposes. The solvent extraction process that was demonstrated consists of the following steps: dissolution of irradiated thorium metal in nitric acid, continuous solvent extraction of 23 in a packed column with methyl isobutyl ketone, scrubbing the solvent containing the product with aluminum nitrate solution, and stripping the product with dilute nitric acid in a second column.

The general features of the 23 Pilot Plant are similar to the Pilot Plant used for the 25 and Redox investigations at ORNL. The average volume of hot process tanks is 40 gallons and the maximum capacity of the plant is about 35 kilograms of thorium per day. Shielding consists of two feet of concrete. Direct maintenance procedures were used. Twenty-one major equipment units including process tanks, condensers, filter and two 1 1/2 inch packed columns were used for hot feed preparation and solvent extraction. Facilities were also provided for cold feed preparation and solvent treatment.

2.0 Summary

Initial equipment operation and testing of the 23 Pilot Plant began in October 1949. Eight cold runs were made during December, and nineteen hot runs were made from January to April 1950. Sufficient engineering data were obtained during this program to design and operate a 23 separations plant. The 23 process flowsheet was tested and demonstrated, and 330 grams of 97% isotopically pure 23 were isolated from thorium metal which was irradiated 100 days at Hanford and cooled 13 to 32 months. The 23 process gave decontamination factors of 2×10^4 for gross beta and gamma fission product activities, 5×10^5 for protactinium, and 1×10^4 for thorium. The total, one-cycle 23 loss was 0.1%. Fission product and protactinium activity remaining with the 23 isolated under flowsheet conditions was less than the 23 background, and ruthenium activity was more than 50% of the fission product activity.

The fission product decontamination factor decreased to about 1×10^3 when the $Th(NO_3)_4$ concentration of the metal feed was increased from 1.6 M to 1.8 M and when the metal feed acidity was increased from 0.2 N acid deficient to 0.05 N acid deficient. Solvent treatment, other than a caustic wash for decontamination, was eliminated by demonstrating that gross amounts of solvent impurities, especially methyl isobutyl carbinol, did not affect process performance. An extraction length of 16 feet was sufficient to recover all but the inextractable 23 (0.05% of the total 23) and scrub section length in excess of eleven feet did not improve separation. The calculated HETS for 23 extraction was 1.7 feet at 15% of flooding and 1.5 feet or less at 50% of flooding.

The 23 solvent extraction process was found to have the same high degree of mechanical operability as was found for the 25 and Redox processes. Equipment operation during the program was, in general, smooth with performance of conventional items, such as column interface controllers, being satisfactory. No leaks were detected in process tubing or Parker Tube fittings. Tinned, screwed pipe joints on filter blister and hot pump piping leaked persistently and were replaced with welded joints. Direct pumping of active feeds by means of the Chicago bellows pump was unsatisfactory due to frequent bellows failure. A thermoelectric flowmeter was found to be a dependable and accurate hot feed flowmeter.

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3.0 Process Description

3.1 Process Flowsheet

Uranium-233 (23) is produced by the irradiation of thorium-232 with thermal neutrons as follows:

$$_{90}Th^{232} \xrightarrow{(n,\gamma)} {}_{90}Th^{233} \xrightarrow[23m]{\beta} {}_{91}Pa^{233} \xrightarrow[27.4d]{\beta} {}_{92}U^{233}$$

After the irradiated thorium is removed from the pile it must be cooled for about eleven months to reduce the amount of protactinium-233 to 0.01% the amount of 23; otherwise, a significant amount of potential 23 is lost as protactinium during chemical processing.

The chemical separation of 23 from thorium and fission products is made in two cycles of solvent extraction. The first cycle is carried out in large moderately shielded equipment, the second in laboratory equipment with only light shielding. Figure 3.0-1 gives a schematic flowsheet for the first cycle of solvent extraction which was demonstrated in the 23 Pilot Plant. In this cycle the irradiated thorium metal is dissolved in 65% HNO_3 using 0.1 M F^- as catalyst. A 0.3 N acid deficient solution is obtained by using a 150% excess of metal and by dissolving to a final specific gravity of 2.09 at a temperature 115°C - 120°C. This solution is then filtered and the filtrate adjusted to 1.6 M $Th(NO_3)_4$, 0.2 N acid deficient and pumped to the extraction column. The 23 is extracted from this solution with methyl isobutyl ketone (MIBK) leaving essentially all of the thorium, fission products and protactinium in the aqueous phase. $Th(NO_3)_4$ acts as the

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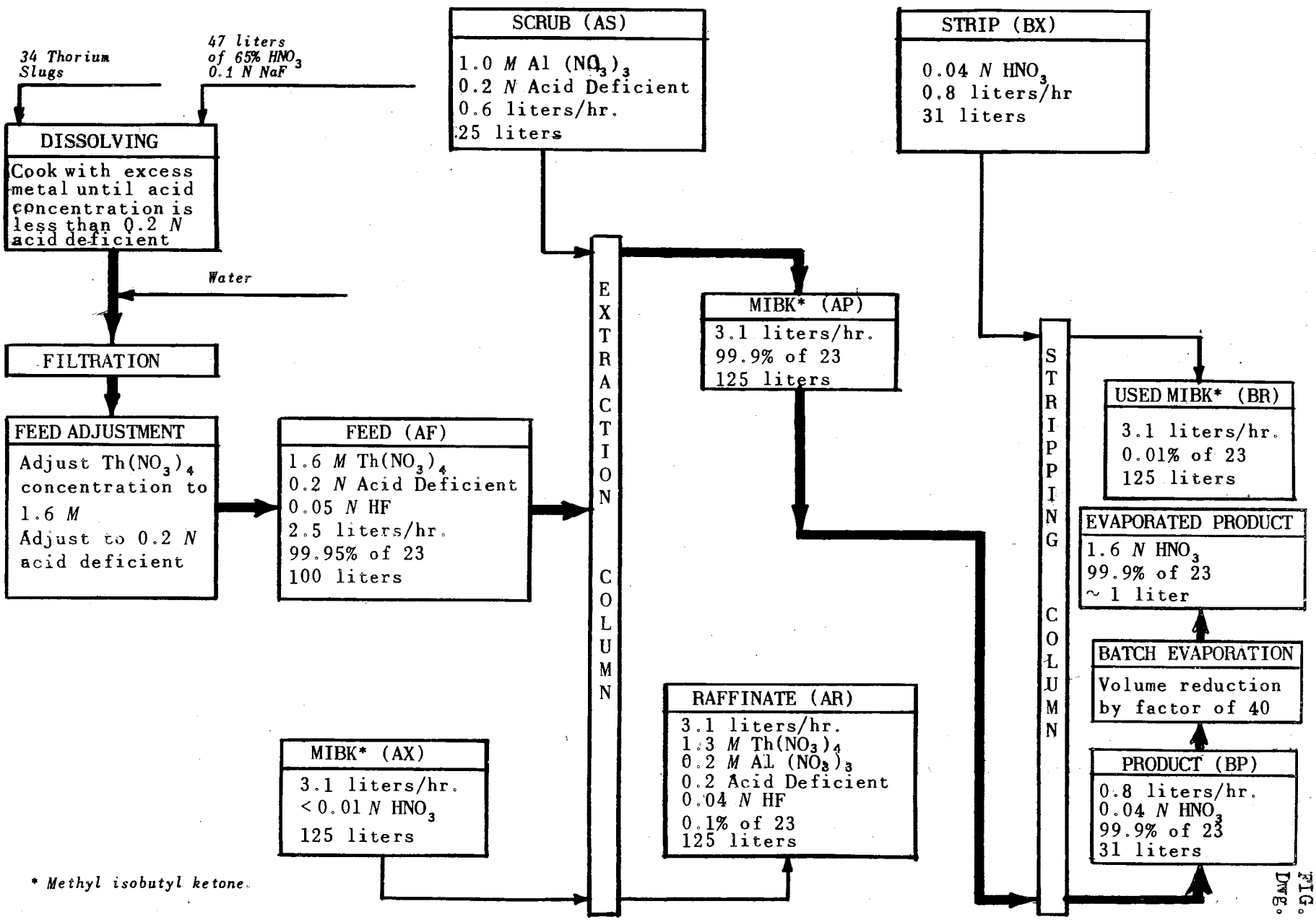

* *Methyl isobutyl ketone.*

23 PROCESS PILOT PLANT CHEMICAL FLOWSHEET

FIG. 3.0-1
Dwg. 8767

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salting agent. The MIBK solution of 23 is scrubbed in the upper section of the same column with 0.2 N acid deficient 1.0 M $Al(NO_3)_3$ to further the separation from thorium and fission products, and the 23 is stripped in a second column with 0.04 N HNO_3. The resulting aqueous solution of 23 is then reduced in volume by evaporation and transferred to laboratory equipment for further cleanup and concentration.

3.2 Process Requirements

3.21 Overall Process Requirements

Decontamination requirements of a 23 chemical separation process depend on both the flux level of the reactor in which the 23 is produced and irradiation time. Successful breeding will depend both on low 23 losses by fission in the pile and on chemical process economy. If 23 production is for experimental purposes, the 23 losses need only be consistant with economy. With the present solvent extraction method of separation, a maximum 23 loss of 0.1% per cycle can be obtained with reasonable operational care and present equipment design.

A thorium decontamination factor of 10^6 or greater is attained in the two cycles of solvent extraction. This insures a product containing less than 0.2% of thorium. This amount of thorium contamination will not interfere with the metallurgy of 23 or constitute a significant amount of poison, if the 23 is used for fuel.

After a cooling period of 11 months a Pa decontamination factor of 10^6 is required to reduce Pa gamma to 10% the gamma background of 23 (See Appendix III-B).

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The required fission product decontamination factor is directly proportional to the fraction of total 23 fissioned in the pile. This fraction is determined by flux and irradiation time (see Appendix III-C). The 23 lost by fission for 100 day irradiation at Hanford is approximately 1%, and a gamma fission product decontamination factor of 5×10^5 is required during chemical processing of this Hanford irradiated thorium to reduce fission product gamma to 10% of 23 background. The largest burnup of 23 during irradiation that has been suggested to date is 3% (for 23 production in the Materials Testing Reactor). A gamma fission product decontamination factor of 10^6 must be obtained during chemical processing of this material after 11 months cooling to reduce fission product gamma to 10% of 23 gamma background.

3.22 First Cycle Requirements

The requirements for the first cycle of solvent extraction are to provide a suitable feed for the laboratory cleanup cycle and to provide sufficient separation from fission products, protactinium and thorium to insure that the overall process requirements are met in two cycles. Reasonable requirements for the first cycle are as follows:

Maximum 23 loss	0.1%
Minimum Th decontamination factor	10^4
Minimum Pa decontamination	10^4
Minimum fission product decontamination factor (based on γ)	10^4

3.3 Process Equipment

The major items of process equipment and the flow of process streams

[redacted]

[redacted]

for the 23 Pilot Plant are shown schematically in Figure 3.3-1. A detailed equipment flowsheet, Figure 3.3-2 shows the process equipment, instrumentation, and auxiliary services to each equipment piece. The cell tanks have volumes ranging from 20 to 100 gallons and were fabricated of stainless steel to conform to the ASME code for Unfired Pressure Vessels. They were equipped with accessories for mixing, heating and cooling, liquid level and specific gravity determination, overflow alarms, and temperature measurement and control. Solutions were transferred by pumps, steam jets, pressure-vacuum, and gravity, and were sampled by recirculating air jet samplers. Makeup tanks outside the cells have volumes of approximately 50 gallons, were fabricated of stainless steel, were mounted on scales, and were equipped with mixers and hinged flap lids for chemicals addition. Further description of process equipment is given in Appendix II.

Detailed drawings for the equipment are listed by ORNL Drawing C-4391, and building drawings are listed by ORNL Drawing TD-1318. A set of typical process run sheets can be found in C.F.# 50-7-22, and a detailed description of the equipment will be presented in Design Report for the 23 Pilot Plant, to be issued by R. J. Klotzbach.

3.31 Building and Area Layout

The 23 Recovery Pilot Plant is housed in Building 706-HB. A plan of the building and cell block, Figure 3.3-3, shows the area occupied by the 23 process equipment. Shielded equipment is located in the east 18 by 18 by 30 ft. high cell block, which contains two 6 by 6 ft. cells and one

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FIG. 3.3-1
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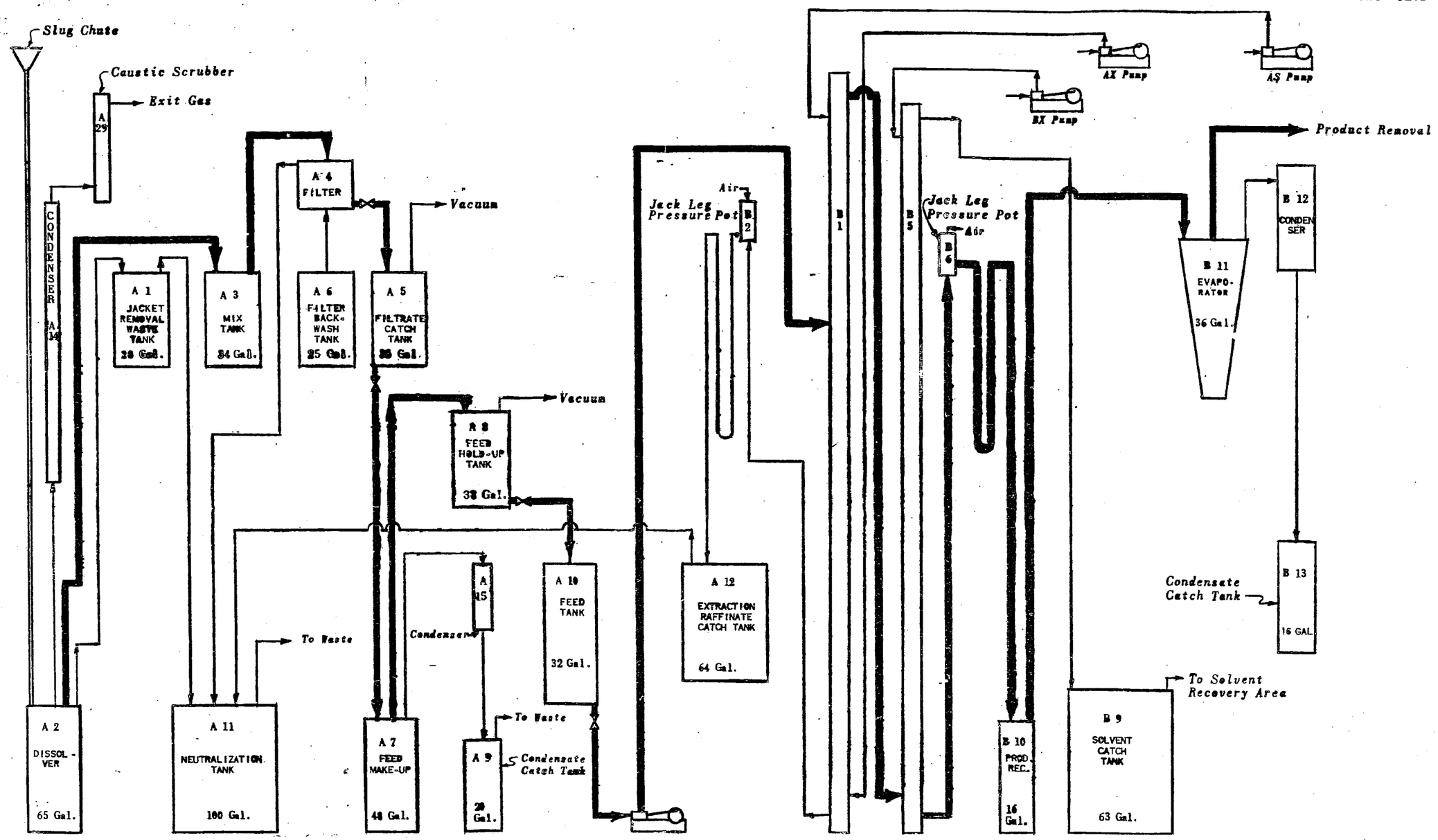

706 HB-1300 PRODUCT PILOT PLANT
CONDENSED SCHEMATIC EQUIPMENT FLOWSHEET

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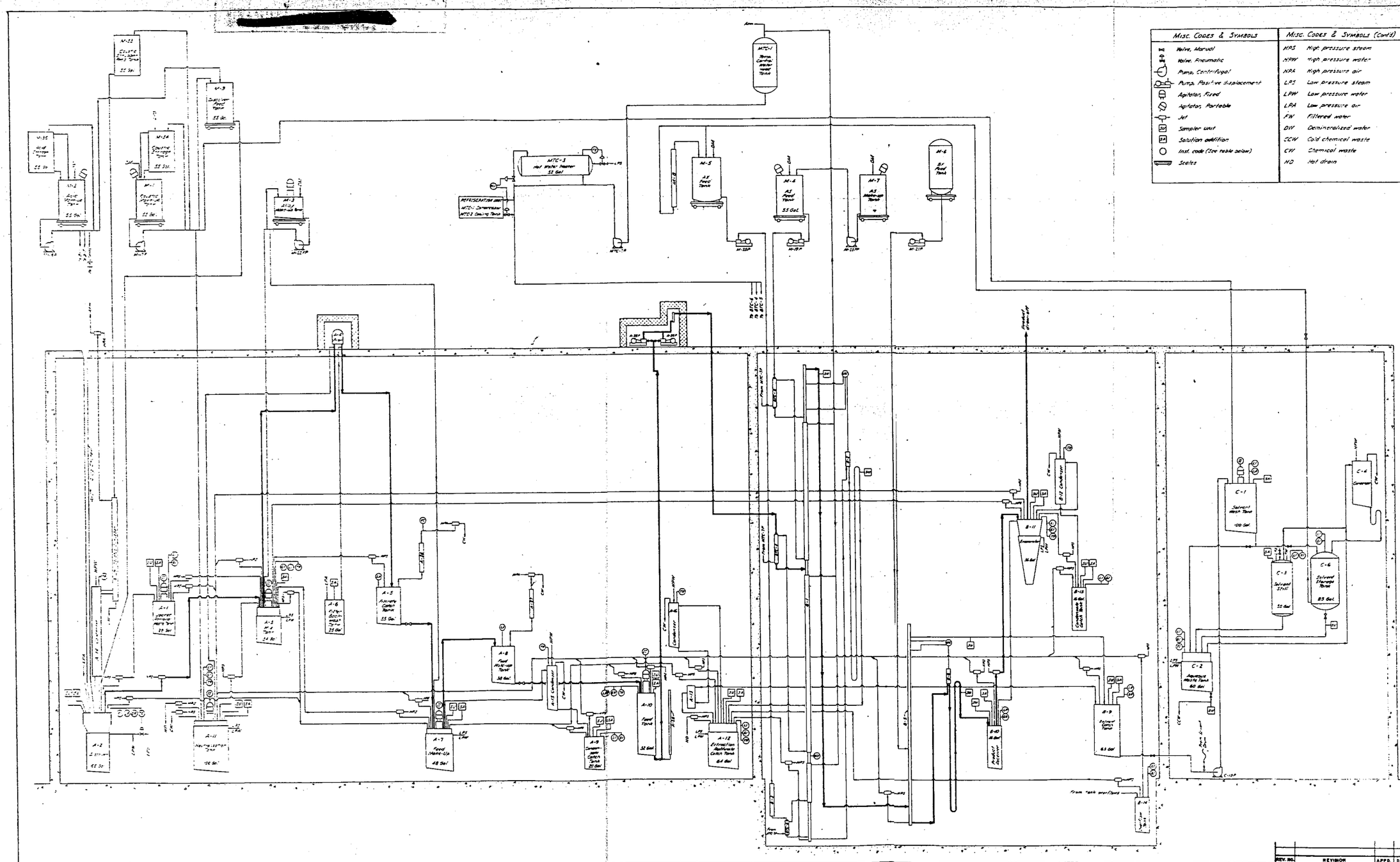

Misc. Codes & Symbols	Misc. Codes & Symbols (Cont'd)
Valve, Manual	HPS High pressure steam
Valve, Pneumatic	HPW High pressure water
Pump, Centrifugal	HPA High pressure air
Pump, Positive displacement	LPS Low pressure steam
Agitator, Fixed	LPW Low pressure water
Agitator, Portable	LPA Low pressure air
Jet	FW Filtered water
Sampler unit	DW Demineralized water
Solution addition	CCW Cold chemical waste
Inst. code (see table below)	CW Chemical waste
Scales	HD Hot drain

Equipment Code	
Equip. No.	Equip. Name
A-13	Separator
A-16	Sorderter
A-26	Vacuum spray trap
A-27	Vacuum spray trap
B-1	Extraction column
B-2	2A Jacketed pressure pot
B-3	2X Accum-Mixer
B-5	Stripping column
B-6	BP Jacketed pressure pot
B-14	Overflow tank
BTC-4	A5 heat exchanger
BTC-5	A5 heat exchanger
BTC-6	A5 heat exchanger
H-B	A5 Pressrubber

Instrumentation Code							
	Symbol	Indicator - I	Recorder - R	Controller - C	Pneumatic Controller - PC	Pneumatic Transmitter - PTC	Electric Transmitter - ETC
Flow rate	F	FI	FR	FC	FPC	FTC	FTC
Specific gravity	S	SI	SR	SC	SPC	STC	STC
Liquid level	L	LI	LR	LC	LPC	LTC	LTC
Pressure	P	PI	PR	PC	PPC	PTC	PTC
Temperature	T	TI	TR	TC	TPC	TTC	TTC
Current	I	II	IR	IC	IPC	ITC	ITC
A code to the symbol indicates an alarm							

General Notes:

1 - Tank and column vent lines are not indicated

2 - All air discharge lines indicated as common lines are actually individual.

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		TECHNICAL DIV. — P.O. BOX P — OAK RIDGE, TENN.
		BLDG. 706-HB
		1300 PRODUCT PILOT PLANT
		DETAILED SCHEMATIC EQUIPMENT FLOWSHEET
		DRAWN BY: [illegible] DATE 10-31-49 SCALE None
		CHECKED BY DATE DRAWING NO. TD-1369 REV.

REV. NO.	REVISION	APPD.	DATE

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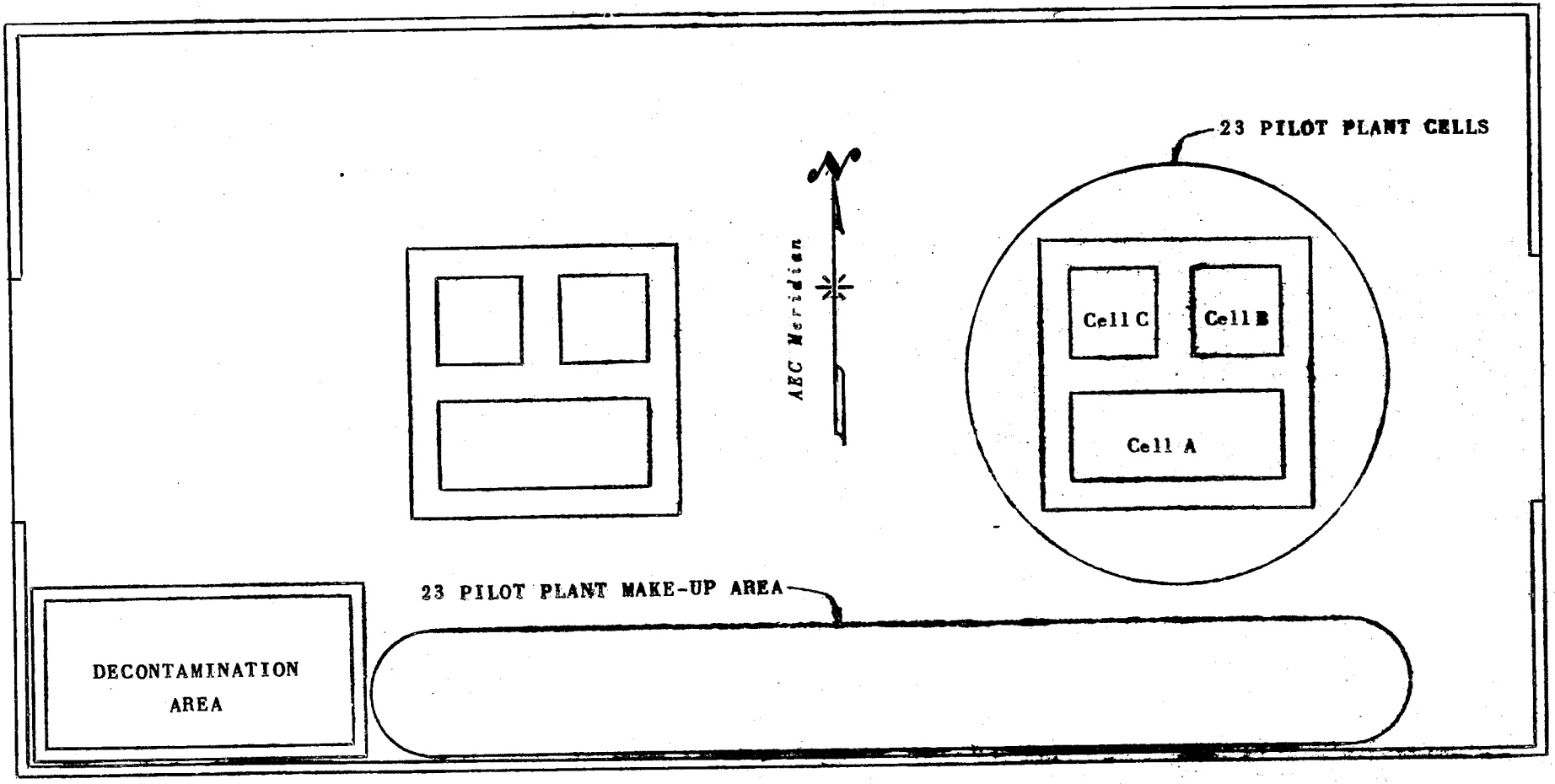

FIGURE 3.3-3

SCHEMATIC PLAN OF BLDG. 706 HB

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14 by 6 ft. cell. Cell B, a 6 by 6 ft. cell, has a pit 3 by 6 by 18 ft. deep to accommodate the columns and all cells have 2 ft. concrete walls. The operating and sampling areas are located on platforms which surround three faces of the cell block at the 10, 20 and 30 ft. elevations.

The reagent makeup area is at ground level along the south wall of the building. Electrical equipment conforms to NEMA Class VII specifications in the cell and to NEMA Class IV or better specifications elsewhere.

3.32 Materials of Construction

Most major tanks were constructed of AISI Type 347 stainless steel. The dissolver, feed adjustment tank, and product evaporator tank were constructed of heat treated AISI Type 309 SCb stainless steel and the caustic storage tank was constructed of mild steel.

Process piping is schedule 40, AISI Type 347 stainless steel. Threaded connections in the stainless steel piping were tinned to give leak free connections. Service piping to equipment jackets, condenser coils, and jet steam lines outside the cell is galvanized steel pipe. Thermocouple wiring and equipment power lines were installed in galvanized conduit. Caustic distribution piping is mild steel.

Most of the process lines in the cell were constructed of seamless, AISI Type 304 stainless steel tubing with 20-22 B and W gage wall thickness. Instrument lines were constructed of copper tubing outside the cell and of stainless steel tubing inside the cell.

All tube connectors are Parker Triple flared Type fittings, Aircraft 811 Type. Type 303 stainless steel fittings were used for stainless steel tubing

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[redacted]

and brass fittings were used for copper tubing. Stainless steel tubes to tanks were connected to fittings screwed and tinned into couplings welded directly into the tank cover.

Bellows sealed, Type 347 stainless steel, replaceable Teflon disk valves manufactured by the Crane Co., Fulton Sylphon Co., and Alloyco Corporation were used in the process equipment. Other types of process valves are Teflon packed and are either standard gate valves or globe valves with replaceable Teflon seats. Panel board and service line valves are brass gate or globe valves. Jet steam valves are globe valves with replaceable composition disks.

Flanged connections to tanks and process tank covers were sealed with Teflon or polythene gaskets. U.S. Gasket Co., Teflon jacketed, corrugated stainless steel core gaskets were used on some connections. Reagent and solvent recovery area pumps were either Teflon packed or equipped with mechanical shaft seals.

[redacted]

4.0 Process Chemistry

4.1 Summary

The 23 process flowsheet is given in Figure 3.0-1, page 16. On the basis of pilot plant runs made under the conditions of this flowsheet, the first cycle of the process is evaluated in the following table.

Table

Evaluation of 23 Process Flowsheet

Decontamination Factor	
Gross Beta	2×10^4
Gross Gamma	2×10^4
Ruthenium	2×10^3
Zirconium	1×10^5
Cerium	5×10^5
Protactinium	5×10^5
Thorium	1×10^4
Total 23 Loss,%	0.1

The 23 process was found to have the same high degree of mechanical operability and adaption to remote control as has been demonstrated for the 25 recovery and Redox processes. Sixteen feet of extraction section were adequate to recover all but the inextractable 23 (about 0.05% of total 23) and the HETS for 23 extraction was 1.7 feet at 15% of flooding and 1.5 feet or less at 50% flooding.

Decontamination was not significantly affected by the following changes.

1. Increasing scrub section length from 11 to 25 feet.
2. Lowering the extraction column temperature from 20° C to 10° C.
3. Increasing the flowrate from 15% to 50% of flooding.
4. Using untreated MIBK containing 0.5% methyl isobutyl carbinol, instead of treated MIBK containing less than 0.02% methyl isobutyl carbinol.

The fission product decontamination factor was decreased by decreasing the acid deficiency of the metal feed, and by increasing the $Th(NO_3)_4$ concentration of the metal feed. This decrease was due entirely to reduced ruthenium decontamination.

4.2 Feed Preparation

4.21 Slug History

The thorium metal used for the Pilot Plant runs was irradiated an average of 100 days at Hanford. The metal had cooled 24 to 32 months for Runs 1T - 12T, 18 months for Runs 13T - 16T, and 13 months for Runs 17T - 19T. A high Pa activity was obtained on Run 3T by spiking with three thorium slugs irradiated in the ORNL pile for six weeks and cooled six weeks. The hottest slugs processed read 10 R/hr at 6 inches.

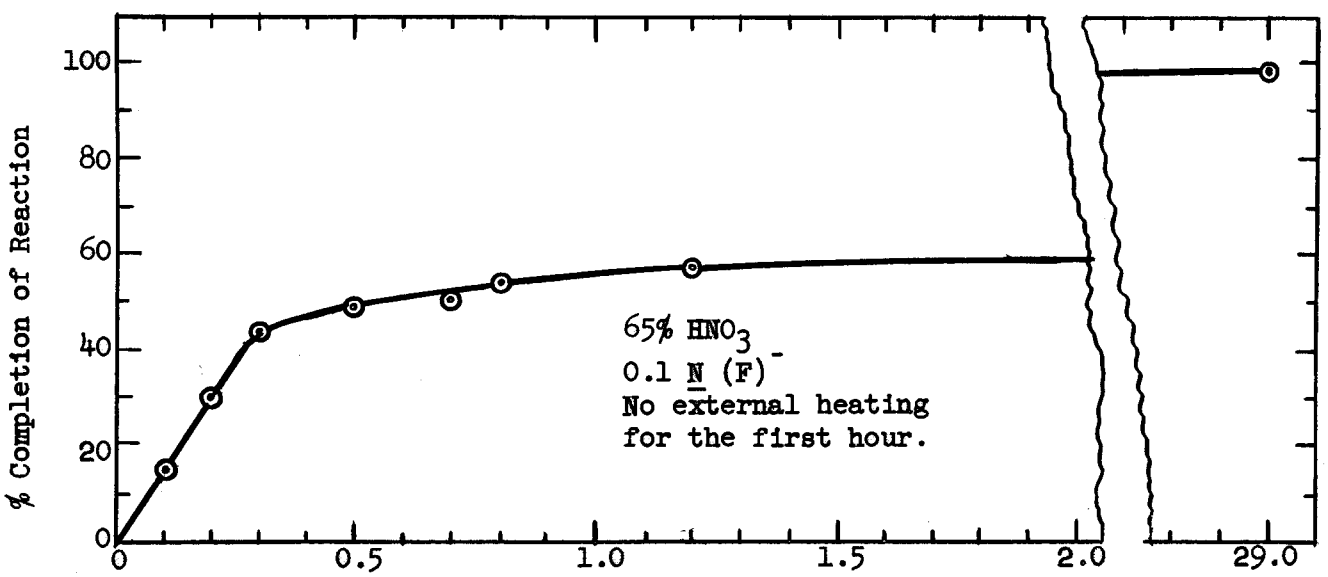
4.22 Coating Removal

Aluminum jackets were removed by the standard 10% NaOH - 20% $NaNO_3$ procedure. On the first run the 23 lost during this step was found to be less than 0.01%. Coating removal losses after the first run were determined by the amount of residual 23 in the dissolver after transfer of dissolver solution and washing of the dissolver (see Section 4.24).

4.23 Dissolving

At the beginning of the 23 Pilot Plant dissolving experiments, it was known that dissolver solutions 0.2 N to 0.5 N acid could be produced by using 60% nitric acid, 0.1 N fluoride and a dissolving time of 12 hours. The objective of the Pilot Plant dissolvings was to develop a method of consistently producing dissolver solution about 0.3 N acid deficient. Based on uranium dissolving experience, the point at which to stop the dissolving can be most accurately determined by specific gravity measurements. It was found that by using an initial HNO_3 concentration of 65%, 0.1 N fluoride as catalyst, a metal heel of about 150% and dissolving to a specific gravity of 2.09 at 115° C to 120° C, a dissolver solution of 0.2 - 0.4 N acid deficient could be produced.

Time of dissolving varied from about 20 to 30 hours, and it was found that the rate of reaction was such that about 45% of the charge was dissolved in the first 20 minutes with no external heating. The following curve gives the per cent completion of the reaction at various times as determined by thorium and HNO_3 analyses.



Time - Hours

Rate of Dissolution of Thorium Metal

[redacted]

Complete data on 18 dissolvings are presented in Table 1, page 110, and the following conclusions are based on these data.

1. Using 65% HNO_3, 0.1 N fluoride, 150% metal heel, and dissolving to a specific gravity of 2.09 at 115° C to 120° C will produce a 3 M $Th(NO_3)_4$ solution, 0.2 to 0.4 N acid deficient. The boiling point of this solution is 120° C.
2. The fluoride for catalyst can be supplied by adding either HF solution or NaF to the dissolver acid, but NaF is preferred because of the hazards of handling a concentrated solution of HF.
3. Some thorium hydroxide precipitation was observed when dissolver solutions reached 1 N acid deficient.
4. The ratio of moles of HNO_3 consumed to moles of thorium dissolved was 5.7.
5. The thorium dissolving data are equally as consistent as available uranium dissolving data.

The actual amount of metal remaining in the dissolver at the conclusion of each dissolving was determined by volume measurements. The procedure consisted of adding a measured amount of water to the dissolver, and carefully measuring the apparent volume of solution in the dissolver.

4.24 Recovery of Residual 23 in the Dissolver after Transfer of the Dissolver Solution

Any 23 in solution remaining in the dissolver after transfer of the dissolver solution was lost with the following coating removal solution. The dissolver jet heel was about 100 ml or 0.1% of the dissolver solution volume, and the procedure used for the first ten runs to recover the 23 in this heel from the sides of the tank, dip tubes and surface of residual metal was as follows:

1. Add 2 liters of water to dissolver, sparge, jet to mix tank.
2. Add 12 liters of water to dissolver, sparge, jet to mix tank.

Using this procedure 0.1 to 0.5% of the total 23 was lost during jacket removal step. In order to reduce this 23 loss, four 15 liter water washes were used on the last 9 runs which reduced the loss to ~ 0.03%; however, this gave an excessive dilution of the feed and it is suggested (see Section 5.512) that the design of the dissolver be changed to permit recovery of this 23 with an acceptable dilution.

4.25 Acid Adjustment

Acidity of the feed was adjusted by adding concentrated solutions of nitric acid or sodium hydroxide. No operational difficulties were encountered.

4.26 Filtration

After adjustment of the acidity, feed solutions were filtered by vacuum through a Micro-Metallic sintered stainless steel filter of 10 micron porosity. This removed all solids that might otherwise have interferred with solvent extraction, and no significant amount of 23 was lost during this step.

4.27 Adjustment of Thorium Concentration

Thorium nitrate concentrations were adjusted by evaporation or dilution on the basis of specific gravity.

4.3 Column Operation

4.31 23 Losses and HETS

The concentration of 23 in the aqueous raffinate (AR) was 0.00006 to 0.0001 mg/ml under normal flowsheet conditions (see Table 2, page 111). This was shown to be inextractable 23 by equilibrating an AR sample four times with an equal volume of MIBK with no detectable decrease in 23 concentration. This amount of 23 was $\sim$0.1% of the total on early, low concentration runs, but was 0.05% on the latter runs. The phenomenon of inextractable 23 was observed in previous laboratory extractions (see MonT-253). The amount found to be inextractable with diisopropyl ether was 0.0001 mg/ml, and 0.00005 mg/ml was inextractable with dibutyl cellosolve.

In two runs an extraction section length of 16 feet was used with no increased 23 loss over the inextractable amount. Two runs were also made using 9.5 feet of extraction length, one at 15% of the flooding rate and the other at 50% of flooding. At 15% of flooding, a loss of 0.05% of 23 in excess of the inextractable occurred, but at 50% of flooding no extractable 23 was lost with the 9.5 feet of extraction. From these two experiments it was concluded that the HETS for uranium extraction was about 1.7 feet at 15% of flooding and 1.5 feet or less at 50% of flooding. The 23 distribution coefficient (O/A) for 23 extraction was about 4.

The 23 loss in the solvent raffinate (BR) was less than 0.01% for all runs.

4.32 Decontamination

A summary of fission product, 13 and thorium decontamination factors are given in Table 3, page 112. The gross fission product decontamination factors have been corrected for 23 gamma and beta counting background. Tables 4 and 5, pages 113 and 114 present detailed data on gross fission product, individual fission product and 13 activities and decontamination factors for all 23 Pilot Plant runs.

4.321 Gross Fission Product Decontamination

The gross fission product decontamination factor was 2×10^4 for both beta and gamma under flowsheet conditions and 50% or more of the beta and gamma activity of the product was from 23 itself. These product solutions, containing about 20 grams of 23 in one to two liters, read ≤ 8 mr/hr at 2 inches with only the glass container as shielding. The gross fission product decontamination varied from 2×10^4 by less than a factor of two when the following changes were made: increasing the scrub section length from 11 to 25 feet, using untreated MIBK made 0.5% methyl isobutyl carbinol, lowering the column temperature from 20° C to 10° C and increasing the flowrate from 15% to 50% of flooding. Also, no change in gross decontamination occurred over the range of cooling time of the irradiated thorium metal from 12 to 32 months.

Gross decontamination was decreased by increasing the acid concentration of the metal feed and by increasing the $Th(NO_3)_4$ concentration of the feed. This was due entirely to decreased ruthenium decontamination (see Section 4.326).

4.322 Ce Decontamination

The decontamination factor for Ce beta was about 5×10^5 under flowsheet conditions. Ce decontamination did not change significantly when any of the variations listed in Section 4.321 were made. Ce beta analysis were made on the concentrated strip solution for Runs 10T - 18T. While this solution is a composite and not entirely representative of equilibrium conditions, the Ce decontamination factors reported for these runs are more accurate than those reported for 1T - 9T due to more precise determination of the higher Ce beta concentration in the concentrated strip solution.

4.323 Zr Decontamination

Only on Runs 3T (spiked with 3-six weeks cooled ORNL slugs) and Runs 17T and 18T (13 months cooled Hanford irradiated metal) was sufficient Zr present in the product to determine a decontamination factor. The decontamination factors for Zr beta were 2×10^4 for 3T and 1×10^5 for 17T and 18T.

4.324 Pa Decontamination

The Pa decontamination factor was about 5×10^5 when flowsheet conditions were used (see Table 3, page 112). As was the case with Zr, Pa concentrations on most runs were too low to determine the Pa decontamination factor. A spike of 3-six weeks cooled ORNL slugs supplied sufficient Pa to measure the decontamination factor on Runs 3T - 5T, and the 13 months cooled metal used for Runs 17T - 18T also contained sufficient Pa. However, no variations in flowsheet conditions were made on these runs, except for 5T, and the only conclusions that can be drawn are that increased flowrate did not affect Pa decontamination and increased salt concentration may have decreased the decontamination factor to 5×10^4 (Run 5T, Table 4, page 113).

4.325 Th Decontamination

The thorium decontamination factor was 1×10^4 or greater for all runs, and no correlation of thorium decontamination factor could be made with any of the variations made in process conditions during the Pilot Plant program.

5.326 Ru Decontamination

The Ru decontamination factor was 2×10^3 under 23 flowsheet conditions, and no change was noted in Ru decontamination when the scrub section length was increased from 11' to 25', solvent containing gross amounts of impurities was used, the column temperature was lowered from 20° C to 10° C and the flowrate increased from 15% to 50% of flooding capacity. The Ru decontamination factor varied from 5×10^3 at 1.3 M $Th(NO_3)_4$ in the metal feed to 110-600 at 1.8 M $Th(NO_3)_4$. Ru decontamination was also markedly affected by the acid concentration of the metal feed. The decontamination factor was 40 when a 0.05 N acid deficient, 1.6 M $Th(NO_3)_4$ feed was processed and about 2×10^3 for similar feeds 0.2 to 0.3 N acid deficient. See Figure 1, page 120 for a plot of Ru decontamination factor as a function of feed acidity.

Ru was the major fission product activity in all products. When flowsheet conditions were used, Ru beta contributed 50% or more of total fission product beta (see Table 8, page 119).

4.33 Material Balances

Thorium, 23 and solution weight material balances for Runs 1T - 18T are given in Table 4, page 113. These material balances were consistently within the probable error of the measurements from which they were calculated. Thorium concentration in the feed preparation tanks was determined from specific gravity and the consistency of this method is shown by the excellent agreement of the material balances (average deviation $\pm$ 2%) over these tanks. The aqueous raffinate (AR) was analyzed colorimetrically for thorium and the average variation in material balances for the AR was about 5%. The largest variations in the 23 material balances were observed for the product catch tank. This tank had only sampler exhaust air for agitation, and the wide variation in this material balance was attributed to non-representative samples.

The weight material balances for the aqueous streams over both columns were satisfactory. The average weight material balance of methyl isobutyl ketone over the system was only 90%, with an average deviation of less than 2%. This agreement was good, and the low material balance was due to evaporation of the solvent. When diisopropyl ether (boiling point 68° C), was used, essentially all of the solvent was evaporated in the process piping until the vent system vacuum was reduced from 4" to 1/4" of water.

4.34 Shutdown Procedure

The following column shutdown procedure was used to insure minimum 23 holdup and to prevent cross-contamination between runs:

(1) Four hours before the metal feed was exhausted, the scrub feed was changed to 1.4 M $Al(NO_3)_3$ - 0.2 N acid deficient.

(2) When the metal feed was exhausted, the scrub rate was increased by 250%, and the solvent feed increased 50%.

(3) Seven hours after the feed was exhausted, the solvent and scrub feeds were shut off, and at the same time, the strip (BX) feed rate was increased by a factor of 5.

(4) Thirteen hours after the feed was exhausted, the strip was shut off.

Figure 2, page 121 plots 23 concentration in the AP and BP streams versus hours after metal feed was off. The 23 concentration in the MIBK extract (AP) began to decrease in less than one hour and within six hours decreased by a factor of 10^3. The use of 1.4 M $Al(NO_3)_3$ scrub instead of the normal 1.0 M $Al(NO_3)_3$ scrub and the increased solvent feed rate made this rapid reduction possible.

The 23 content of the strip product (BP) did not begin to drop until six hours after the feed was off. The rate of decrease was much less than the AP, requiring 9-10 hours to decrease by a factor of 10^3 at 5 times the normal flow rate, making a total of 17 hours after the metal feed was exhausted.

4.4 Product Evaporation, Filtration and Purity

4.41 Product Evaporation

The average volume of strip product was about 50 liters. This aqueous solution of 23, containing only trace contaminants and saturated in MIBK, was transferred to a batch evaporator and reduced in volume to 0.5 liters. This concentrated 23 solution was then transferred to a glass bottle and the evaporator washed with a nimimum of water, giving a final volume of 1 to 2 liters of concentrated 23 solution. Losses of 23 for this step were less than 0.01% for all runs.

4.42 Filtration of Concentrated 23 Product Solution

To insure a clear feed for the 23 laboratory cleanup, and for accountability reasons, the concentrated 23 product solutions were filtered through a medium porosity, sintered glass filter in the laboratory. This yielded a solution containing no visible solids. Filtration was a simple operation to perform, except for two product solutions which contained large amounts of $Al(NO_3)_3$ carried over from the extraction column due to instrument failure.

The observed 23 loss for product filtration was less than 0.01% for all runs.

4.43 Product Purity

4.431 Isotopic Purity

Results of isotopic abundance analyses by the mass spectrographic method on the 23 produced during the program are given in Table 7, page 118. Of the 327 grams of uranium isolated, 318 grams (97.3%) were U^{233}, 7.9 grams (2.4%) were U^{238} and 0.95 grams (0.3%) were U-234. This will be split into two batches for laboratory processing. Composition of the two batches are given in the following table.

	% U^{233}	% U^{234}	% U^{235}	% U^{238}
Batch #1, 234 g of U	98.2	0.3	0.01	1.5
Batch #2, 93 g of U	95.0	0.3	0.04	4.7

The instrument used for the analyses on Runs 1T - 9T was also being used for other enriched uranium analyses and the unreasonable amounts of U-235 and U-236 reported in these products were due to contamination of the instrument. The 0.3% - U^{234} content was from (n ,γ) reactions on Th^{233}, Pa^{233} and U^{233}. This amount of U^{234} agrees well with the calculated production of this isotope. Five ppm of natural uranium in the original thorium metal accounts for the 1.5% of U^{238} in the 234 gram batch. The 4.7% of U^{238} content of the other batch was also attributed to contamination of the source material. The increased U^{238} content beginning on Run 9T coincided with a change in batches of thorium metal, and the decrease in U^{238} content beginning on 13T after this batch was exhausted substantiates this reasoning.

4.432 Fission Product, Th and Pa Contamination of Product

The 23 produced when the 23 flowsheet was used without variation contained about 0.02 mg of thorium per mg of 23. Fifty per cent or less of the beta and gamma activity of these products was from fission products and Pa, the remainder of the activity was 23 background (see Table 8, page 119.) All products except 3T - 6T, which contained excessive aluminum due to column control instrument failure, were suitably pure for feed to the laboratory clean-up cycle. The 3T - 6T products were reprocessed and met specifications for the laboratory cycle feed.

4.5 Diisopropyl Ether Run, 19T

One run, 19T, was made using diisopropyl ether as the 23 solvent. The diisopropyl ether flowsheet was not recommended for first cycle 23 because of the low flash point solvent and engineering difficulties of handling nearly saturated salt solutions. However, it seemed advisable to demonstrate the

advantages of this process at full Hanford level as this solvent could be successfully used for many special separation problems.

4.51 Flowsheet Description

The following table gives the pertinent features of the 23 diisopropyl ether flowsheet.

Solvent	AF Composition	AS Composition	BX Composition	Flow Ratio
DIPE	2.5 M $Th(NO_3)_4$	2.5 M $Al(NO_3)_3$	0.04 M	AS:AF:AX:BX
		0.7 M NaF		
	0.1 N HNO_3	0.4 N acid deficient	HNO_3	= 1:4:5:1

4.52 Results

Twenty-two grams of 23 were recovered from 13 months cooled, 100 day Hanford irradiated thorium with a gross fission product decontamination factor of 1×10^5 (based on gamma activity), a Pa decontamination factor of $> 1 \times 10^6$ and a Th decontamination of 5×10^6. This degree of separation was about 5 times greater than that obtained with MIBK. The total 23 lost during this run was less than 0.1%. The aqueous raffinate loss was 0.01%, the solvent raffinate loss 0.04% and the jacket removal loss was 0.02%. Detailed results of Run 19T are included in the Appendix with the MIBK runs. The Ru decontamination factor for Run 19T was 1×10^4, the Ce decontamination factor 6×10^6 and the Zr decontamination factor 1.6×10^5. The Ru and Ce decontamination factors were higher than for MIBK runs, but Zr decontamination was essentially the same as for MIBK.

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Some operational difficulties were encountered during the diisopropyl ether run. The scrub solution solidified when the building temperature dropped to 10° C at night and essentially all the solvent was evaporated as it passed through the columns and piping by the sweep of air through the vent system. However, when the building temperature was held above 20° C and the vacuum on the vent system reduced to 1/4" of water, the run was made without further difficulty.

4.6 Analytical

With the exception of 23 and Pa^{233} radiochemical analyses and thorium colorimetric analyses, all analytical procedures used in the 23 program were the same as those used in the Redox and 25 work and have been reported in ORNL-463. Detailed procedures for 23, Pa^{233} and thorium analyses are given in Appendix IV, page 102.

4.61 Individual Analyses

The following comments are made on the analyses used during the program.

Thorium Analyses - Thorium concentrations in feed preparation tanks was determined from specific gravity by the following relation:

$$\text{Molarity of } Th(NO_3)_4 = \frac{SpG-1.000-.0013(27-T^{\circ}C)-.03\ \underline{N}\ HNO_3-.04\ \underline{N}\ NaOH-.04\ \underline{N}\ NaNO_3}{0.395}$$

This method is accurate ± 3% in the molarity range 1.0 to 1.8.

HNO_3 (Feed Make-Up Samples) - Potentiometric titration was used.

Gross Beta and Gamma - Gross beta counts were made at 10% geometry with about 20 mg of solids on the counting plate. Gross gamma activity was determined by a high pressure ion chamber and reported in millivolts (mv). One mv is equivalent to 2.5 gamma counts/minute at an efficiency of 0.05% for 0.5 Mev gamma emitters.

Specific Fission Products - Conventional radiochemical analyses were used to determine Ru, Zr and Ce beta activity. Zr beta determinations gave erratic results due to interference from Pa and it was necessary to scavange the samples by precipitating Cb_2O_5 to remove Pa before analyzing for Zr.

4.62 Beta to Gamma Ratios for Ru and Pa

Factors for conversion between beta counts and ion chamber determinations were used for interpreting and evaluating data. Counts/mv values for Ru (from 60-100 day cooled metal), Zr and Cb decay can be found in ORNL-463. For the 23 program it was necessary to re-determine the ratio of Ru activities which were essentially due to Ru^{106} after the 1 to 2 year cooling period. The beta to gamma ratio for Pa^{233} was also determined. The experimental values were as follows:

$$\frac{Ru^{106}\ \beta\ c/m}{Ru^{106}\ mv} = 2.2 \times 10^3$$

$$\frac{Pa^{233}\ \beta\ c/m}{Pa^{233}\ mv} = 2.8 \times 10^3$$

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4.63 Gamma and Beta Counting Background from 23

Uranium-233 has a weak gamma radiation which produces X-rays and conversion electrons that count as beta. The gamma background from 23 was found to be 0.60 mv/mg as measured in a high pressure ion chamber. The beta counting background varied markedly with the amount of solids on the counting plate as this radiation is made up of three components, one of 102 mg of Al half-thickness, another of about 11 mg of Al half-thickness, and a weaker component of 1.5 mg of Al half-thickness. With no appreciable solids on the counting plate a sample of 23 counted 6×10^3 β C/m/mg, but with 30 mg of solids (reducing agents added to prevent loss of Ru in the conventional gross beta analyses), the sample counted 3×10^3 c/m/mg. The value of 3×10^3 was used in calculating decontamination data as 20 mg or more of solids were added to all beta counting plates for gross beta analysis.

Beta activity will grow into freshly extracted 23 at a maximum rate of 2 β c/m/mg of 23 per day (the first daughter, Th-229, has a half-life of 7×10^3 years). The beta counting background from 23 is a factor of 10^3 higher than that from freshly extracted natural uranium, and about a factor of 4 higher than natural uranium in equilibrium with its daughters. The gamma activity of freshly extracted 23 is 10^3 times greater than that of freshly extracted natural uranium and 100 times that of uranium in equilibrium with its daughters.

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5.0 Equipment Performance

Equipment evaluation is based primarily on the short term operation of the 23 Pilot Plant. Performance observations on the 205 Bldg. Hot Pilot Plant during 25 and Redox Process work are used to amplify the evaluation of similar equipment items in the 23 Pilot Plant. The activity level of the 23 Recovery Process is such that direct maintenance was performed on most hot equipment items without decontamination and on the few remaining pieces after several mild chemical rinses.

5.1 Building

5.11 Operating Areas

Equipment layout in most operating areas was satisfactory although the chemical makeup facilities were somewhat congested. Office space should have been provided with the building. Operating areas at the elevated levels were crowded; maximum utilization of potential operating space should have been achieved by extending the upper levels to the building wall. Steel grating floors for the elevated operating areas were completely unsatisfactory in that chemical handling and sampling operations were hazardous to personnel and equipment below. A stainless steel, raised edge floor covering in the sampling and reagent addition area partially remedied this situation.

Floors, which were loosely specified "pitch all floors to trench," do not drain adequately. A minimum floor pitch of 1/8"/ft. should be used to insure rapid and complete drainage of floors. Drain trenches and possibly the floors in the chemical makeup area should be surfaced with acid and solvent

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resistant material. Prufcoat painted floors have good abrasion resistance, and appear satisfactory for all areas that do not use concentrated acid or solvent.

The heating and ventilation system, which draws air into the building at roof height and exhausts it at ground floor level into the evacuated cells, does not give uniform temperatures at the upper and ground floor levels. An uncomfortable building noise level is maintained by the ventilation blowers in the cell exhaust ducts.

Slug charging operations require transporting the heavy carrier into and above a conjested operating area on the ground floor. This is unsatisfactory from the safety standpoint.

5.12 Cell Areas

Relative congestion in the cell areas has not hampered maintenance because of the low level of radiation from adjacent tanks. The hot feed pump and filter blisters are too small for efficient equipment maintenance. The stainless steel flooring in Cell A is very highly recommended, as it is superior, in all respects, to coated concrete for resisting activity adsorption. The strippable paint, G. E. Cocoon, appears satisfactory for resisting activity adsorption by the previously painted cell walls. Cell ventilation is excessive, thereby increasing building heating problems. Hood ventilation standards should not be used for cells. Drains, both for cold and hot wastes, should be positively connected to the equipment to prevent fume and active vapor escape.

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The two-foot thick concrete cell walls were adequate for shielding the low activity levels encountered.

5.2 Materials of Construction

5.21 Tanks and Tank Supports

Corrosion of stainless steel has caused the failure of one welded tip thermocouple well. In addition, a few leaks have occurred on process piping and about 10 leaks have occurred on A14 and B12 condenser coils. All leaks occurred in welds.

Corrosion penetration of the dissolver was found to be $<0.02''$/year for continuous operation of the metal solution step. Quantitative checks of corrosion of the feed preparation and product concentration equipment were not made. About 300 hours of operating time ($\sim$7 runs) were required, as evidenced by the quality of the concentrated product solution, to initially clean and pickle the equipment. Similar cleanup times were observed in the 205 Bldg. Hot Pilot Plant.

Attack of the painted, mild steel cell tank supports did not occur, primarily because intensive decontamination was not required. Experience indicates that major spills on the structural steel would be extremely difficult to decontaminate because of activity retention on the corrosion roughened surface of the steel.

5.22 Tubing, Pipe and Jets

5.221 Tubing and Tube Fittings

The Type 304 stainless steel tubing with Parker Triple Type flared fittings, Aircraft 811 Type, was highly satisfactory for

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process, service and instrument lines in the cell. Advantages of tubing over a welded and flanged piping system are:

1. No leaks were observed from corrosion failure or from couplings.
2. Initial tightness and maintenance of a leak free system required ~80% less effort than for a flanged piping system. Frequent retightening of joints to take up gasket extrusion was not necessary.
3. Frequent disassembly of joints for inspection and cleanouts of lines did not affect tightness.
4. Vibration from agitator operation did not loosen couplings.
5. Tube couplings are particularly suited for installations where space is at a premium.
6. Line changes were made with a minimum of lost time because of the simplified fabrication and installation proceedures for tubing.
7. No welding is required, therefore unstabilized stainless steel (type 304) is satisfactory. Appreciable savings of strategic stabilizing metals are effected.
8. The line cost of a tubing installation is ~60% less than for a similar pipe and flange installation because of material and installation time savings.

Tubing is highly recommended as the successor to pipe for radiochemical pilot plant and plant installations.

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5.222 Tinned Joints

All pipe thread joints were tinned with 50% lead, 50% tin solder to insure tightness. Tinning was not required on any of the Parker tube couplings.

Tinned joints on the hot feed pump and feed filter piping leaked consistently, even after repeated tinning. This may be attributed to any or all of the following:

1. Pulsing turbulent flow which may have accelerated corrosion of the tinning alloy. Transmitted vibration from the pump may have accelerated failure.
2. Insufficient tightening of the tinned joint before allowing the tinning alloy to harden. Rough, imperfect threads may have prevented proper tightening.
3. Strains in the piping system induced by piping misalignment and frequent operation of valves.

These tinned joints were replaced with welded joints wherever possible.

Tinned joints on tank covers, jets, and columns showed no sign of failure and were entirely satisfactory.

5.223 Pipe, Pipe Flanges, and Gaskets

Field welds made on the hot pump piping and in the filter blister were unsatisfactory with about 20% of the welds developing leaks. This is attributed to insufficient space to make adequate electrode passes and in certain cases to the emergency procedure of welding over

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defective tinned joints. Other welded joints were satisfactory except as noted in 5.21.

The special three bolt flanges on the A4 Filter were unsatisfactory as they could not be tightened sufficiently. They were replaced, wherever possible, with four bolt, 1/4" pipe flanges. Tube fittings are recommended over flanges for similar congested installations.

The Teflon tank cover gaskets on Tanks A2, A7, A14, A15, A16, B11, B12, C3 and C4 and the polythene gaskets on all other tank covers were satisfactory. Some gasket creep caused by thermoplasticity was observed on the polythene gasketed covers on tanks A3, A11, and A12. Polythene should not be used on lines or tanks that are heated. Full face Teflon and polythene gaskets were moderately satisfactory after intensive initial tightening on the few pipe flanges and on the column sections. A few leaks in the hot pump blister and on the column flanges were traced to gasket creep. Replacement of these gaskets with very thin, Teflon ring gaskets, or with U. S. Gasket Co., Fench type, corrugated stainless steel core, Teflon jacketed gaskets was satisfactory. The latter gasket has significant advantages over a solid Teflon gasket and is highly recommended.

The mild steel and galvanized steel piping was satisfactory for electrical and jacket services, but are not recommended for high level work where a spill would be difficult to remove from the corrosion roughened pipe.

5.224 Valves

About one-third of the 35 Alloyco #62 valves with Teflon

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packed stems failed. Failure occurred because the steam threads gall and freeze to the bonnet or the soft Teflon seat is damaged by closing the valves too tightly. Standard gate valves were satisfactory for similar service in the 205 Bldg. Hot Pilot Plant and were satisfactory as replacement valves in the A4 feed filter blister.

Four Allayco, four Fulton-Sylphon and four Crane bellows sealed valves were used on various process lines in the plant. Because of the small number of bellows valves and the short duration of the program, the only conclusion drawn concerning the performance of these valves is that the Alloyco and Crane valves were generally satisfactory and were superior to the Fulton-Sylphon valves.

One Fulton-Sylphon bellows sealed valve in a solvent line failed by a break in the stem and bellows connection and the valve could not be opened. Three other Fulton-Sylphon valves were in active feed lines and developed slow leaks. Inspection showed scoring from extreme compression of the Teflon stem disk and possible misalignment of the valve stem. The Alloyco and Crane bellows sealed valves with the Teflon disk on the stem did not develop noticeable leaks. However, these valves are not installed on main process lines, but on column jet lines and on solvent transfer lines. Inspection of four Alloyco, three Fulton-Sylphon, and three Crane bellows sealed valves at the conclusion of the hot runs disclosed that the seats of all valves were slightly deformed but all were operable. One Alloyco valve and one Fulton-Sylphon valve had the seats roughened by dirt particles pressed into the Teflon. The three Fulton-Sylphon valves showed a tendency for the stem threads to bind. One Crane valve had a disorted bellows, possibly caused by accidental twisting of the bellows. About 17 standard Crane gate valves used for similar service in the 205 Bldg. Pilot Plant were uniformly in good condition after 2 years service, while one of four Teflon disk Hammel- Dahl

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valves had failed from seat scoring by glass fragments and metal particles which had not damaged the gate valves. A packed stem gate valve, possibly bellows sealed and motor operated, is recommended for testing for remote hot process line valves.

The renewable composition disk and replaceable seat, brass body, jet steam valves in the operating area gave some trouble from steam leakage after extended use. A 300 lb. standard is recommended as a more rugged valve than the 150 lb. standard valves now used. The renewable, floating, composition disk and replaceable seat features must be retained to insure leak free cutoff and facilitate maintenance.

5.3 Chemical Makeup and Solvent Treatment Equipment

5.31 Tanks

5.311 Nitric Acid and Caustic Systems

The head tanks for storage of concentrated acid and caustic are too small and require refilling every second run. Pumping concentrated acid from Acid Makeup Tank M2 to Dissolver Feed Tank M9, instead of pumping acid directly from the Acid Storage Tank has resulted in accidental dilution in M2 of several batches of acid. A similar unsatisfactory arrangement is used for the caustic system. Head tanks for acid and caustic should be sufficiently elevated so that gravity drainage is obtained to critical feed and makeup tanks and to outlets in the reagent addition funnel and solution makeup areas.

Protected sight glasses and drip pans should be provided for concentrated reagent storage tanks.

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The decontamination system for pumping from Acid Makeup Tank M2 directly to all major equipment items was not tested. However, it is a recommended process adjunct, based on 25 and Redox process work.

5.312 Scrub

The system of using one makeup tank for standard scrub and filtering it to the pump feed tank was satisfactory. However, the use of Alloy Makeup Tank M3 to prepare shutdown scrub is unsatisfactory as the scrub cannot be thoroughly filtered before use and M3 was occasionally needed for thorium solution makeup. A system which had considerable merit utilizes a large stock tank for preparation of concentrated salt solution which is diluted as required.

Space must be provided in a plant for expansion of makeup and pumping facilities.

5.313 Solvent

5.3131 Solvent Feed Tank

The Solvent Feed Tank M5 was not large enough to hold all the solvent for a run. However, this is not a handicap, if refilling operations are adequately recorded.

5.3132 Solvent Prescrubber

The glass pipe, Raschig ring packed prescrubber, M8, used to scrub the hexone draining to M5, accumulated less than five grams of dirt during the entire program.

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5.3133 Solvent Recovery Tanks

The solvent recovery vessels C1, C3, C4, and C6 were satisfactory. However, tests indicated that gross amounts of impurities in the methyl isobutyl ketone flowsheet do not affect the extraction operation. Therefore, a solvent treatment system consisting of a collector-wash tank and a washed solvent storage tank are adequate for a 23 plant. Direct operation of the solvent treatment system can be used as only small amount of activity is retained by the solvent.

The aqueous waste tank C2, designed for flashing solvent from aqueous waste, was not used as the wash solutions were not stored in underground tanks. Even if underground storage were used, the distillation of the impure solvent in C2 into C6 would be unsatisfactory.

5.314 Tank Covers

The field fabricated tank covers for the equipment in the chemical makeup area were unsatisfactory in that the covers are loose and frequently bind against addition lines. Openings at the pipe entrances and flap hinges allow dirt from grating flooring to fall into the tanks. It is recommended that dirt tight covers, which must allow free play of entering pipes, be provided for all pump feed and makeup area tanks. Hinged flaps and in some cases, hoppers, should be provided on all tanks for inspection and chemical additions.

5.32 Pumps

5.321 Column Feed Pumps

Milton Roy positive displacement pumps for pumping scrub,

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strip and solvent to the columns gave excellent service and are highly recommended. Pump stroke can be set within 1% of the desired flow and average flowrate variation was 2%. Similar pumps in the 205 Bldg. Hot Pilot Plant have given excellent service for a 3 year period, although the micro-stroke adjustment mechanism was not sufficiently splash and corrosion proof and consequently a few pumps lost their adjustability.

The rotameters on the pump discharge lines were useful as a qualitative flow indicator only. Flowrate checks made by pumping from a calibrated burette were more accurate and reproducible. An alarm sounding upon stream flow failure would be desirable in conjunction with the burette. Recording rotameters and orifice meters in the 205 Bldg. Hot Pilot Plant for comparable low flowrates were unsatisfactory because of meter plugging and because of recording instrument defects. The accuracy and simplicity of the burette flowrate check, combined with the constant output of the Milton-Roy pumps, is such that the need for installation and maintenance of flow recorders for cold process streams appears questionable.

The pump discharge manometer was useful as a check on pump operation and as a dependable pressure relief valve. In general, the feed pump, manometer, and burette piping was not sufficiently accessable for ease of operation and maintenance.

5.322 Filter Pumps

The Alsop "Centripoise" pumps on the scrub and alloy makeup tank filters gave excellent service for all ordinary process

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solutions. The mechanical shaft seals did not leak.

5.323 Solvent Pump

The Eastern Industries Model F centrifugal pump was satisfactory for pumping solvent from the Catch Tank B9 to Solvent Wash Tank C1. The mechanical shaft seal showed no sign of leaking. However, the poor suction characteristics of this centrifugal pump made it impossible to completely empty solvent drums. A rotary positive displacement pump would be more satisfactory in this service.

5.324 Acid Pump

The Durco Model X3MDR-72 centrifugal acid pump was satisfactory for pumping from Acid Makeup Tank M2. However, to empty carboys the pump must be primed by inserting the suction hose into the carboy and applying pressure to the carboy with a hand bulb. This undesirable situation could be remedied by filling the head tank with a rotary, positive displacement pump or by using vacuum on the head tank to empty carboys. A glass wool packed suction line filter would keep abrasive particles from scoring the pump and would remove carboy gasket particles from the acid.

5.325 Caustic Pump

The Viking Model ZG2D, rotary, positive displacement pump was completely satisfactory.

5.33 Scales

The Howe beam type scales for all makeup and feed tanks in the chemical makeup area were extremely useful. Chemical makeup procedures

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and feed tank inventory checks during a run were considerably simplified by the use of scales. Scales could be improved by adequate corrosion protection for weights and beam parts and by protecting the beams under the platform from floor splatter.

5.34 Agitators

The Mixing Equipment Company Lightnin Mixers have been satisfactory. Shaft lengths were such that propellers were about 18" from the tank bottom and crystals could not be suspended. The shafts were lengthened so the propellers were 2 to 4" above the tank bottom wherever possible.

Small volumes of thorium or scrub solutions could not be agitated easily in M3 because of an excessive distance, 6", between the propeller and the tank bottom. A replacement shaft for this model VH-1 Eastern Industries agitator was not available.

The model VH-1, Eastern Industries agitator on Solvent Wash Tank C1 vibrated excessively from shaft whip. This could be remedied by a stabilizer ring, by a shorter or larger diameter shaft, or, preferably, by operating at a lower speed with a larger impeller. The agitator mixes the solvent-wash solutions satisfactorily.

5.35 Ventilation

The solvent recovery system tanks are adequately vented by the process vessel off gas system.

The only positively ventilated tanks in the makeup area are the Solvent Feed Tank M5, which has an air jet discharging to atmosphere outside the

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building, and the Alloy Makeup Tank M3 which is equipped with a hood and a fan for contamination control. Fumes from the Acid Makeup Tank M2 and the Dissolver Feed Tank M9 have corroded adjacent mild steel equipment and were unpleasant to personnel.

It is recommended that off gas facilities be provided for all makeup area and pump feed tanks. This could be conveniently done with one small fan for ventilating all the cold makeup tanks.

5.36 Filters

The Alsop Sealed Disk filters for scrub and thorium nitrate solutions were satisfactory. Filtration rates were about 2.0 gpm for M7 to M4 and only 0.5 gpm for M3 because of the considerably smaller area in the M3 filter.

The effect of filtration was nullified by dirt falling into the tanks from the over-head grating flooring. Pump stoppages and dirt accumulation in the columns from this source might be eliminated by installation of a very fine mesh stainless steel screen filter element on the pump suction line. Sintered stainless steel previously tested for this service in the 205 Bldg. was unsatisfactory.

5.4 Generalized Cell Equipment Evaluation

The following information applies generally to equipment items in the feed preparation, the column and column accessories, and the product concentration equipment groupings.

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5.41 Process Vessels

Tank volumes were adequate and jet and vacuum transfer line heels were low, generally less than 100 cc. The experimental design of the tank covers with dip pipes screwed and tinned into couplings welded in the tank cover was satisfactory and, with tubing and tube fittings, was a major contributor to a leak free system. Decontamination is simplified by this elimination of gaskets and inaccessible surfaces on the tank cover. An improvement in tank design would feature elimination of the large gasketed lid in favor of a small, more readily tightened manhole for inspection and dip tube installation. Use of couplings welded in the tank top appears advisable wherever possible to eliminate flanges and inacessable surfaces on the tank cover; however, this practice complicates maintenance on dip tubes and thermocouple wells.

5.42 Tank Jackets and Condensers

Tank jackets were satisfactory in that tanks can be heated and cooled in a minimum of time. Jacket heat transfer coefficients were measured on a typical tank, Waste Neutralizer All, and are applicable to tanks of similar design. The data is summarized in the following table:

Tank Conditions	U, Btu/hr/sq.ft/°F	Rate Tests	
		Temperature Range	Time
No agitation, heating, 10 # steam	330	70°F to 210°F	50 min.
Agitated, heating, 10 # steam	400	70°F to 210°F	40 min.
No agitation, cooling, 25 # water	80	195°F to 110°F	54 min.
Agitated, cooling, 25 # water	140	195°F to 110°F	48 min.

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The tank was filled with water and the bare jacket was exposed to cell ventilation drafts. Heating coefficients were obtained with essentially all the jacket area heated by condensing steam.

All condensers were satisfactory from the heat transfer standpoint. It is estimated that the dissolver Reflux Condenser A14 has a safety factor of about two, and that all other condensers have safety factors of about two to three. The overall heat transfer coefficient was found to be U = >550 BTU/hr/sq.ft/^{o}F for Condenser B12, which is a typical coiled tube, single tube, condenser. This is an approximate coefficient as the vapor condensing area was estimated because marked sub-cooling of the condensate occurred at maximum evaporation rates and with minimum cooling water flow. Solvent condenser coefficients were not measured.

Thermocouples in the condensate outlet line instead of the vapor inlet line would be more useful from the operating standpoint, except for the solvent condensers which should have thermocouples in both locations.

5.43 Tank Agitation

Cell tank agitation by the Eastern Industries Company low speed mixers was satisfactory with uniform blending being attained in a few minutes. The low speed of this unit is deemed a desirable feature for long agitator unit life.

Air spargers were improvised for unagitated raffinate, product, and condensate catch tanks in an attempt to get reproducible sampling. The spargers have been satisfactory for mixing in four tanks but erratic results

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were obtained in the sparger equipped Product Catch Tank, B10. Dissolver instrument pen fluctuation, caused by the sparger, was eliminated by installing snubbers on instrument lines.

A suitable method of agitation should be provided for all tanks that are to be sampled.

5.44 Jets

The Schutte and Koerting 3/4" process transfer steam jets transferred solutions with a specific gravity of 1.8, suction head of five feet, and a discharge head of 15 feet. These jets had satisfactory capacity (about 4 gpm with 1.8 M Th), were little affected by wide variations in operating steam pressure, and performed satisfactorily with bends on steam, suction, and discharge lines.

The Schutte and Koerting steam jets operated with air were excellent for vacuum transfers and maintained a vacuum of 22"-24" of mercury on the Filter A4. Use of these jets prevents possible contamination of plant vacuum systems.

Schutte and Koerting steam jets are recommended for solution transfer and vacuum supply.

5.45 Overflow Catch Tank

The Overflow Catch Tank B14 was useful in collecting overflow and flush liquids from tanks, columns, and hot pump blisters. Such a feature is highly recommended. The utility of B14 could be increased by providing a sampler, a sparger, and a jet directly to the feed mix tank.

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Tank overflow lines, although normally never used, should be arranged so that tank cover under surfaces could be decontaminated by flooding. Overflow lines are undesirable on evaporators and other tanks used for boiling liquids.

5.46 Waste Neutralizer

The waste neutralizer functioned as a holdup tank for all wastes except thorium waste, rather than as a neutralizer. This tank was particularly useful for collection and sampling overflow liquid transferred from Overflow Catch Tank B14, as B14 has no sampling facilities.

5.47 Process Vessel Off Gas

Excessive air leakage through the slug chute cover seal reduced the dissolver off gas vacuum to less than 1/2" of water. This was remedied by sealing the slug chute cover with a plastic compound. However, the resulting high off gas vacuum drew large volumes of air into the system through the hot feed pump blister drain and through waste jet discharge lines. Normally high solvent losses (10% for methyl isobutylketone) from evaporation increased to 100% loss for diisopropyl ether. Also, steam condensate from a boil-down in A7 flowed into A2 via the A2 to A7 jet line. When the high off gas vacuum was throttled to prevent excessive solvent losses, adequate dissolver vacuum could not be maintained. Recommendations for a similar installation are as follows:

1. Provide liquid seal loops or jackleg seals in lines to prevent vapor flow to the wrong tank.

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2. Provide liquid seals on unvalved drains into the systems and on jet discharges from the system so air leakage into the system will be kept to an absolute minimum.
3. Arrange the off gas system so the dissolver is provided with a high vacuum (∿ 10" of water) large capacity source. This system should be capable of being readily throttled by manual means and could be used to discharge vacuum transfer and filter air jet discharges.
4. Arrange the off gas system so the remaining process vessels are provided with a low vacuum (1" - 2" of water), moderate capacity source for disposing of sparger, sampler, instrument, and leakage air.
5. Orifice plates or valves for throttling purposes should be installed on all vessel off gas lines to provide maximum vacuum on evaporator condensers and minimum air flow through columns and solvent catch tanks.

5.48 Shielding

The concrete cell walls with the stacked concrete block entry way closures and the stacked block entrance labryniths were satisfactory for shielding this process. Maximum radiation of 17 mr/hr occurred at the surface of the 16 inch thick, stacked block wall opposite the bottom of the

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dissolver loaded with 96 slugs. This was the only place that appreciable radiation was detected through lead or concrete shielding.

5.5 Feed Preparation Equipment

5.51 Slug Dissolving Equipment

5.511 Slug Chute A17

Occasional jamming of slugs side by side in the chute was caused by the practice of loading the charging machine in such a manner that slugs dropped two at a time. The defective seal between the slug chute and chute cover mentioned in Section 5.47 could be corrected by providing a narrow, raised collar on the bottom of the cover. Unit pressure on the chute gasket would be high enough to cause the gasket to yield and give a tight seal. A standard sound detection, recording, and amplifying device for slug counting would be useful in a production plant.

5.512 Dissolver A2

The dissolver instrument dip tubes were adjacent to the sparger dip pipe; consequently sparger operation caused violent fluctuation of the liquid level and specific gravity pens. This fluctuation was eliminated by instrument line snubbers as the sparger dip pipe could not be relocated.

The procedure used for recovering small amounts of 23 from the dissolver walls and metal heel prior to coating removal was multiple, small-volume rinses that were boiled to reflux liquid and remove 23 from the exposed surfaces. To increase the efficiency of 23 removal and reduce the

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volume of dissolver rinse liquid that must be evaporated for incorporation with the feed, it is recommended that the bottom of the dissolver be provided with a separate jacket. Rinse volumes of about five liters could be efficiently refluxed from the cold tank walls, dip pipes, and the metal heel. Also, the tank wall should be jacketed only to the level of the normal dissolving volume to prevent baking solids on the tank walls above the liquid surface. Consideration should be given to the feasibility of providing a jacket on the upper tank wall. Such a jacket would serve as an emergency condenser or to assist in larger than design volume operations.

Dissolver jacket temperature control is most important during the first two hours of metal solution. Water cooling of the jacket was necessary in some dissolvings to prevent flooding of the off gas scrubber. Manual jacket temperature control is satisfactory to reduce the initial violence of reaction, but requires close attention to the jacket cooling water flow. A jacket cooling water rotameter and a steam-water mixing device for use during the first two hours of dissolving would be sufficient for close manual control.

5.513 Condenser A14

Capacity of the Condenser A14 was adequate (estimated 100% excess capacity) as off gas temperatures were maintained at 25°C during all stages of dissolving. A leak was detected in this condenser at the conclusion of Run 17T and A14 was removed for inspection. Preliminary decontamination was not required and maximum radiation readings of only 15 mr/hr were obtained on A14. Numerous leaks at spacer bar welds and tube butt welds were

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found on the two inaccessible, inner cooling coils. To avoid delay for fabrication of a new coil assembly, the leaking coils were blanked off and the condenser reassembled the same day. Additional cooling supplied by directing a stream of water on the condenser shell was adequate to keep the off-gas temperature to a maximum of 60°C during initial stages of a dissolving.

A condenser fabricated so that individual coils can be removed for repair or replacement is recommended. A minimum of welding should be done on condenser tubes to avoid tube wall damage. Also, it is practical to design coils for individual replacement or repair in both high and low activity level plants. If repair is required, working time will be limited by radiation from adjacent tanks in the cell rather than from the condenser. Finally, unheatreated type 347 stainless steel coils will give as good service as unheatreated type 309 SCb stainless steel coils. These conclusions were confirmed in the 205 Bldg. Hot Pilot Plant when the dissolver condenser was inspected at the conclusion of the Redox program.

5.514 Off Gas Scrubber A29

Undesirable amounts of NO_2 escaped from the Off Gas Scrubber during initial stages of the dissolving reaction. Increasing caustic flow and adding excess air to insure complete oxidation of NO to NO_2 had no appreciable effect on scrubber operation. It was concluded that the rate of gas throughout during initial stages of the reaction was too high to get complete adsorption, since 45% of the metal dissolves in the first 20

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minutes with no dissolver jacket heating or cooling beyond the initial heating to start the reaction. This can be remedied by temperature control to slow down the initial stages of the dissolving, by increasing the scrubber height by about 50%, and by eliminating air leaks into the dissolver via the slug chute and transfer lines.

5.52 Mix Tank and Filtration Equipment

5.521 Mix Tank A3

The volume of the Mix Tank A3 was too small to hold a batch of dissolved metal and the dissolver rinse liquid. Operational complexity was increased by having to filter part of the metal solution before the last portions of dissolver rinse were transferred to A3. It would be desirable to concentrate the combined metal solution and dissolver rinse in A3 before filtration with only a dilution to specifications being required in Tank A7 after filtration. Both A3 and A7 should be equipped to evaporate feed solutions. A3 would then be very useful as a concentrator before filtration for reclaimed liquids from tank washes, hot pump cleanups, or overflow from tanks or column feed line vents.

5.522 Filter A4

As noted previously, the original piping in the filter blister valve compartment was unsatisfactory. Tinned joints, three bolt flanges, and needle valves were replaced by welded joints, standard four bolt flanges, and flanged gate valves. This assembly, although very difficult to repair because of confinement by the shield, was satisfactory after repairing several defective field welds.

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The filter element was removed at the end of Run 10T to determine if holes in the filter element were responsible for large amounts of silica in Makeup Tank A7. The complete filter unit had to be removed, with considerable overexposure risk to personnel, because space was not available in the blister to dismantle the shell and remove the star element in place. Connections could not be made by remote or semi-remote means and shielded facilities were not available for handling the highly active element. The cause of the high filter activity level was a heavy deposit of silica from slug jacket bonding alloy. The silica could not be washed off of the filter and out of the filter shell by the low liquid rates attainable by backwashing. After removal, the star filter unit was easily decontaminated to 40 mr/hr and was in excellent condition. It is believed that the silica in A7 came from inadvertent operation of the filter bypass valve.

With the above exceptions, the filter was entirely satisfactory. Filtration rates of 1.5 gal/hr sq. ft. were maintained with 1.6 M $Th(NO_3)_4$ and 23" Hg vacuum. No washing procedure was done, other than a small volume water rinse after filtration.

Recommendations for future hot feed filtration equipment are as follows:

1. Provide sufficient space in the filter and filter piping shields for repair and replacement operations.
2. Arrange filter so it can be replaced by remote means. Transfer shields should be provided for disposal of radioactive elements, particularly for high radiation level processes.

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3. Jacket the filter in order that hot filtrations may be done and for more efficient decontamination in place.
4. Eliminate horizontal surfaces and corners in the bottom of the filter shell or provide high pressure spray nozzles so that silica deposits of the consistency of a dense mixture of clay and fine sea sand can be slurried up and washed from the shell. Backwashing through the filter element is not adequate as sufficiently high flow-rates cannot be obtained without risking damage to the star filter.

5.523 Filtrate Catch Tank A5

The Filtrate Catch Tank A5 was satisfactory and the Moore liquid level alarm was useful in preventing overflows. Capacity of this tank, like that of A3, was not sufficient for a combined batch of metal solution, dissolver rinses, and filter water rinses. This slightly increased operational complexity. If off gas capacity were available, the air discharge from the vacuum jet on both this tank and on A8 should be put into the dissolver off gas system. Experience in the 205 Bldg. Hot Pilot Plant indicated that very little activity escapes from vacuum filtrate catch tanks, and as a result the glass wool packed line filter remains relatively cold.

5.53 Makeup Tank A7 and Auxiliary Equipment

The capacity of Makeup Tank A7 was too small to evaporate the combined dilute batch of metal solution, dissolver rinse liquid, and

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filter wash liquid in one batch. This slightly increased operational complexity. Evaporation losses were less than 0.01%. Final adjustments of feed thorium concentration were easily made on the basis of specific gravity. The Condenser A15, Condensate Catch Tank A9 and Feed Holdup Tank A8 were satisfactory.

5.6 Solvent Extraction and Stripping Equipment

A schematic drawing for the extraction and stripping equipment, with elevations of important equipment items, is given in the Appendix, Figure II-I, page .

.61 Hot Feed System

5.611 Feed Tank A10

Feed Tank A10 was satisfactory. The only trouble experienced was a small leak in a weld on the A10 outlet flange.

5.612 Electronic Burette

The electronic burette was a reliable and accurate method for checking the feed pump rate. The constant error of this instrument was less than 2%, which was comparable to the error in checking the flows of inactive streams. Occasional erratic operation of the burette was traced to formation of a grease film on the platinium electrode insulators. Routine cleaning operations eliminated any further trouble.

5.613 Feed Rotameter

The feed rotameter was used as a qualitative visual check on the flowrate. It was particularly useful during feed pump startup

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and shutdown when the thermoelectric meter was not at equilibrium or when there was insufficient feed in A10 to fill the electronic burette.

5.614 Thermoelectric Flowmeter

The thermoelectric flowmeter was stable with less than 2% constant error. Grease and solids which make the electronic burette and the rotameter inoperable had detectable effect on this meter. Two thermocouple failures caused by moisture in the instrument housing, rather than by corrosion failure of the thermocouple wells, occurred during the program.

Suggested changes would be to improve the design of the instrument case and to incorporate a recorder, rather than an indicator on the unit. Based on 23 Pilot Plant experience, this meter is recommended for plant operation for metering hot feed and should be considered for inactive process streams.

A typical Thermoelectric Flowmeter calibration curve for these feed solutions is given in the Appendix, Figure 3, page 112 .

5.615 Chicago Bellows Pumps

Fatigue failure of bellows, after an average life of about 100 hours or 300,000 flex cycles, occurred during the first half of the program. Replacement of the bellows was simple but involved shutting down both pumps to flush active solution from the piping and bellows. Liquid from bellows failure drained into, corroded and contaminated the rocker and rocker adjustment mechanism in the pump base.

The pumps maintained constant flow, ± 2%, at a constant suction head but exhibited a 10% decrease in pumping rate at constant stroke when the

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suction head was decreased from 20" to 6". Pump stroke was adjusted during a run to compensate for this.

It is concluded that the bellows pump would be satisfactory for low activity level, direct feed pumping if bellows life were 1000 hours or more. However, it is believed that this pump, in spite of the bellows seal, offers no advantages over the conventional Milton Roy pump for direct feed pumping. The Milton Roy is reliable, maintains constant flows, and can be packed and maintained so that essentially no leakage occurs from the shaft gland.

5.62 Extraction Column B1

5.621 General Operability

The Extraction Column B1 operated smoothly over a wide range of feed concentrations and with two solvents for a total operating time of about 1600 hours. Throughputs ranged from 100 gal/hr/sq.ft. (15% of flooding) to 400 gal/hr/sq.ft. (50% of flooding) at maximum pump output. No sign of column plugging was observed although feed solutions containing appreciable amounts of solids were inadvertently used.

5.622 Feed Points

Additional feed points for feed and for solvent were installed on the column in order that extraction column variables could be studied. Such extreme flexibility is not needed in a plant, although a spare feed point should be provided to lengthen the scrub section. Also, to eliminate uncertain time lags in changing from one scrub to another, dual

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scrub lines, one for regular and one for shutdown scrub, should go to the scrub feed point from the respective pumps.

An air vent line should be installed on the solvent accumulator to insure complete air displacement from the system.

5.623 Column Interface Control

Column interface control by the Taylor Fulscope Controller was smooth and positive over a wide range of flows and concentrations. Initial difficulties caused by the large density changes in the column and jackleg line during a run were eliminated by lowering the jackleg loop and jackleg pot and operating the pressure pot with air directly from the controller to get the necessary wide air pressure range.

A typical plot of interface position and observed jackleg air pressure is given in the Appendix, Figure 4, page 123 .

Recommendations for a plant installation are:

1. Provide the interface control unit with an additional pressure recording pen for recording the jackleg control air pressure. This was found to be useful for detecting conditions that may cause jackleg blowing or jackleg seal.
2. Provide the interface control unit with a manual-automatic transfer switch in order that the interface can be maintained by manual control when repair or testing is required on the automatic control unit. This need was demonstrated when repair of a loose part in the automatic control unit was required.

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3. Have trained instrument men check the assembly of all internal parts of the controller prior to installation. This recommendation is based on 23 Pilot Plant and Tank Farm Evaporator experience.

4. Locate the jackleg pressure pot and jackleg loop low point at elevations such that all conditions during a run and run shutdown can be controlled.

5.624 Temperature Control Unit

The temperature control unit can maintain $\pm$ 1°F temperature control between 50°F and 100°F with no difficulty other than those caused by significant cell temperature changes. Cell temperature changes of $\pm$ 10°F require readjustment of the water heating and cooling units because of uninsulated column surfaces in the cell. Heat exchangers on the feed lines are located several feet from the feed points and exit feed streams attain cell temperature before they enter the column.

Temperature was shown to have very little effect on the methyl isobutylketone extraction process. Hence, incorporation of a temperature control unit is not recommended for a plant installation.

5.63 Stripping Column B5

The Stripping Column, B5, operated satisfactorily. The jackleg seal overflow should be located at least 12 inches (instead of 3") lower than the pressure pot in order to provide a greater margin of safety for the necessary hydraulic heads.

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5.64 Raffinate and Product Catch Tanks

Capacities of the Extraction Raffinate Catch Tank A12, Solvent Catch Tank B9, and Product Catch Tank B10 were adequate, although B10 had to be sampled and emptied at the midpoint of a run. The system for flashing dissolved solvent from the extraction column aqueous raffinate in A12 was satisfactory. Mechanical agitation rather than sparging should be provided on these tanks.

5.7 Product Concentration

Evaporation of the product solution in Evaporator B11 was entirely satisfactory with loss of $< 0.01\%$ at an evaporation rate of 15 liters/hour. Product drawoff into unprotected glass bottles, although satisfactory for short term pilot plant operation, is not recommended. A suitably ventilated and enclosed hood, with protected glass product containers and stainless steel transfer lines should be provided.

Rinsing operations to remove residual 23 from B11 to segregate product batches could be facilitated by an additional jacket for the lower portion of the tank. Small rinse volumes ($\sim$100-250 cc) could be boiled and efficiently refluxed off the cold piping and upper tank walls. This is the same problem that occurs in removal of trace amounts of 23 from the dissolver.

5.8 Sampling

The recirculating air jet samplers were satisfactory for sampling all process solutions, and were not a source of excessive contamination.

Solutions containing greater than 2.0 M thorium were difficult to sample

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because of crystallization in the jet throat. Similar troubles were experienced with concentrated uranium solutions in the 205 Bldg. Hot Pilot Plant and can be solved by an arrangement in which the air jet serves only as a vacuum source for a sampler liquid collector. The pot may be continuously drained back to the tank through a separate barometric leg sealed line or an electronic liquid level alarm probe, with a manually operated drain valve may be used.

More space should be provided in the blister and the sampler tips must be designed to handle standard conical bottom, centrifuge type, sample tubes, as well as small bottles. Cell wall space limitations required the use of a gravity drain valve sampler for the extraction column product stream (AP) and required the valving of Product Catch Tank B10 sample blister to use the blister for sampling either the tank or the flowing stream. Both arrangements were unsatisfactory. The extraction column product line outside the cell was unduly hazardous from the standpoint of possible leaks, splattering and contamination of sample tube surfaces during the sampling operation, and the return of sampler flush liquid to the system. Doubling up on the functions of a sampler blister increases possibility of operational error and complicates the equipment. Organic stream samplers for the columns should be provided with a separator to return entrained aqueous to the column. Aqueous entrainment is particularly noticeable in a small column where instrument probe air agitates the interface.

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5.9 Instrumentation

Instrument maintenance in this plant required about 80% less effort than in the 205 Bldg. Hot Pilot Plant. The use of Fisher-Porter "C" Clamp" rotameters instead of sight feed bubblers for dip tube air metering was responsible for most of the reduction in maintenance. Manometer check valves should be used on all manometers.

The utility of Moore constant differential relays on instrument probe lines, particularly on small tanks, is highly questionable.

No instrument troubles or air failures were caused by the use of filtered plant air for instrument air.

Continuous temperature indication, in addition to recording, should be provided on tanks such as the dissolver, dissolver condenser, and solvent condensers in which there are rapid, critical temperature changes.

Thermocouples on condensers should be put in the condenser off gas line rather than the vapor inlet line; except in the case of solvent distillation, it is more important to know that all vapors are being condensed than to know the inlet vapor temperature.

Thermocouple wells should be fabricated by first plugging the end with a drive fit, taper plug and then welding. If weld failure does occur, the well will still be leak free.

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(13) ORNL-703 The Pilot Plant Demonstration of a Batch Solvent Extraction Process for the Recovery of U^{233} by R. E. Leuze, 1950.

(14) ORNL-676 Terminal Report of ORNL Pilot Plant Development of the Materials Testing Reactor 25 Recovery Process by A. M. Rom, May 1950.

(15) ORNL-343 The Laboratory Development of a Continuous Solvent Extraction Process for the Recovery of U-235 by F. R. Bruce, March 1950.

(16) ORNL-466 Recommendations for Design of Radioactive Solvent Extraction Units by D. G. Reid, September 1949

(17) ORNL-109 First Progress Report of Pilot Plant Test of 25 Decontamination and Recovery by H. M. Feder, E. L. Nicholson, and D. G. Reid

(18) ORNL-463 Summary Report for the ORNL Pilot Plant Investigation of the Redox Process by H. K. Jackson and G. S. Sadowski.

 [redacted]

APPENDIX I

Cost of the 23 Pilot Plant Program

A. Building Cost

The cost of 706-HB Building was $303,000. This building is of permanent construction and was designed to house two pilot plants in addition to a wide variety of unit operations experimental equipment. A reasonable building charge for the 23 Pilot Plant Program is assumed to be $50,000.

B. Equipment Cost

The cost of the 23 Pilot Plant equipment was $255,000, distributed approximately as follows:

Engineering	$28,000
Labor	70,000
Materials	87,000
Overhead	70,000
Total	$255,000

The 23 Pilot Plant is a soundly designed and constructed unit, and will be used on a routine basis for further process development work. It is assumed that an amortization of $70,000 for the 23 program is reasonable.

C. Program Cost

The program cost for the 23 Pilot Plant Program was $170,000, including labor, materials, chemical supplies, analytical services, and laboratory overhead.

D. Total Cost

On the basis of the pro-rated building and equipment cost given above, the total cost for the 23 Pilot Plant Program was $290,000.

APPENDIX II

Details of Process Equipment and Operations

A. Reagent Makeup and Solvent Treatment

1. Nitric Acid and Sodium Hydroxide Systems

Seventy per cent C.P. nitric acid was unloaded from carboys and pumped to the gravity head tank, M35, by a Durco Co. Model X 3 MDR-72 Centrifugal Durichlor pump. Acid was pumped from the Acid Makeup Tank M2 to the Dissolver Scale Tank M9 on the 20 ft. level for slug jacket removal and metal dissolution. Acid for decontamination operations was pumped to various equipment items by lines to each reagent addition funnel line.

Commercial 50% sodium hydroxide was unloaded from drums and pumped to the heated gravity head tank M34 by a Viking Co. Model ZG2D iron pump. Caustic was pumped from the Caustic Makeup Tank M1 to the Dissolver Scale Tank M9 for slug jacket removal and to Head Tank M33 for feed to the Off-Gas Scrubber, A29.

2. Thorium Nitrate Solutions

Thorium nitrate solution was prepared as required in the hood equipped, Alloy Makeup Tank M3 and pumped to the process through an Alsop Filter equipped with a stainless steel "Centripoise" pump.

3. Scrub Solutions

Standard scrub solution was prepared in Scrub Makeup Tank M7 and pumped to the Scrub Feed Tank M4 through an Alsop Filter equipped with a stainless steel "Centripoise" pump. Scrub solution was pumped to the extraction column through a surge pot and rotameter by a Milton Roy Model MD1-13-24 pump, equipped with an

indicating, micro stroke adjustment. Flow rates were checked by pumping from a calibrated glass standpipe and by weighing M4 at regular intervals.

The Shutdown Scrub solution, prepared in Alloy Makeup Tank M3, was recirculated through the filter, and pumped to the extraction column by the M4 Milton Roy pump during shutdown of a run. Flowrates were checked by pumping from a calibrated glass standpipe and by weighing M3 at regular intervals.

4. Strip Solution

Strip solution prepared in Strip Makeup-Feed Tank M6 was pumped to the stripping column through a surge pot and rotameter by a Milton Roy Model MD1-13-24 pump equipped with an indicating, mocro stroke adjustment. Flowrates were checked by pumping from a calibrated glass standpipe and by weighing M6 at regular intervals.

5. Solvent Treatment

Solvent treatment equipment is located in Cell C and the Solvent Feed Tank, M5, is in the reagent makeup area.

(a) Solvent Pretreatment

Raw solvent was pumped from drums into the Solvent Wash Tank, C1, by an Eastern Industries Model F centrifugal pump. Solvent was washed in C1 by agitation with aqueous solutions. The phases were allowed to separate and the waste wash solution drained to the Aqueous Waste Tank, C2. An electronic probe in the C1 drain actuates an alarm when the aqueous-solvent interface moves past the probe and aqueous heel removal is complete. Washed solvent can be drained to either the Solvent Storage Tank C6 for immediate use or to the Solvent Still C3 for steam distillation from a caustic heel. Vapors from the distillation go to Condenser C4 and condensate is collected in C6. Water heel in C6 from distillation is drained to C2 and purified solvent is drained to the Makeup Area. Solvent in C6 may be pumped back to C2 for additional treatment.

(b) Solvent Recovery

Stripped solvent from Solvent Catch Tank B9 was pumped to C2 where it was washed with water and either drained to C6 or steam distilled to C6 from C3.

(c) Solvent Feed Tank

Purified solvent from the Solvent Storage Tank C6 was drained by gravity to the Solvent Feed Tank M5 in the makeup area through the prescrubber column M8 where it was equilibrated with aluminum nitrate solution. Solvent was pumped to the extraction column through a surge pot and rotameter by a Milton Roy Model MD1-24-48 pump, equipped with an indicating, micro stroke adjustment. Flowrates were checked by pumping from a calibrated glass standpipe and by weighing M5 at regular intervals.

6. Demineralized Water

Demineralized water for process use was supplied to the makeup area and the solution addition funnel area by a building head tank replenished by a small Barnstead demineralizer unit.

B. Feed Preparation

1. Slug Charging and Jacket Removal

Irradiated thorium slugs were weighed to prevent inclusion of a uranium slug and then loaded into a drawer type charging machine. Slugs dropped from the charging machine into the Slug Chute A17 fell into a previously prepared sodium nitrate solution in the Dissolver A2. The dissolver was heated and the aluminum slug jackets removed by adding an excess of caustic from the M9 Dissolver Scale Tank. Vapors were condensed by the Reflux Condenser A14 and fumes removed from the fixed gases by Caustic Scrubber A29. The fixed gases were discharged to

the atmosphere by the building off gas fan. Spent jacket removal solution was steam jetted to Jacket Removal Waste Tank A1, sampled, and jetted to Waste Neutralizer, A11, and then to Chemical Waste storage. Traces of caustic were removed from A2 Dissolver by repeated water rinses.

2. Metal Solution

Sixty-five per cent nitric acid, catalyzed with hydrofluoric acid or sodium fluoride, was added to the Dissolver A2 and the thorium metal dissolved by prolonged heating. Acid vapors were condensed and returned to A2 by Reflux Condenser A14. Off gases were passed through Caustic Scrubber A29 and discharged to the atmosphere by the off gas fan. The metal solution was diluted while hot, then cooled, sampled, and steam jetted to Mix Tank A3. Residual metal heel in A2 was determined by comparing the apparent volume with the known volume of a dissolver rinse.

3. Feed Preparation

(a) Filtration

Metal solution in Mix Tank A3 was combined with A2 Dissolver rinse solutions and acidity adjustments were made by adding concentrated nitric acid or C.P. sodium hydroxide. Metal solution was transferred from A3, by vacuum, to the Filter A4, mounted in a lead blister on the outside cell face. Solids were collected on a sintered stainless steel, 1.33 sq. ft., star shaped, Grade G porosity, Micro-Metallic Corporation filter element. Filtrate was collected in Filtrate Catch Tank A5 which was evacuated by a Schutte and Koerting air jet exhauster and provided with a Moore Electric Liquid Level overflow alarm. Solutions for filter element backwashing to Waste Neutralizer A11 or air for sparging to Mix Tank A3 were forced from Filter Backwash Tank A6.

(b) Feed Adjustment

Filtrate was drained from Filtrate Catch Tank A5 through a bellows sealed valve to Feed Makeup Tank A7. Final acidity adjustments were made and the thorium concentration adjusted by dilution or evaporation. Vapors were condensed by Condenser A15 and collected in Condensate Catch Tank A9 for sampling and disposal to Waste Neutralizer A11.

Adjusted feed solution was transferred by vacuum to Feed Holdup Tank A8 and dropped by gravity to Extraction Column Feed Tank, A10, through a bellows sealed valve. A8 is provided with an Schutte and Koerting air jet exhauster for vacuum and a Moore Electric Liquid Level overflow alarm.

C. Column Operation

1. Feed Pumping

The feed solution drains from A10 by gravity flow through a bellows sealed valve to the check valves of one of a pair of parallel connected, individually shielded Chicago Bellows Pumps, M23P and M24P. E ach pump consists of a Chicago Metal Hose Co. stainless steel bellows which is flexed by an adjustable, cam-driven rocker arm. The bellows actuate a Hills-McCanna, double ball suction and discharge, check valve. Pumping rate is controlled by the adjustable rocker arm pivot and is checked by a shielded rotameter, an electrode type burette located in Standpipe A28, and by an ORNL Thermoelectric Flowmeter. The Thermoelectric Flowmeter indicated and integrated the wattage required to maintain a constant temperature differential between two thermocouples in the feed line, and flowrate was determined by referring to previously prepared calibration curves.

[redacted]

2. Extraction Column

Feed solution was pumped through a heat exchanger into the Extraction Column B1. The column is a vertical 1-1/2 inch stainless steel pipe packed with 1/4 x 3/8 inch stainless steel Raschig raings. Packed height is 41.5 ft. with a 30.75 ft. extraction section, a 10.75 ft. scrub section,and an overall height of 46.5 ft. Alternate feed points permit wide variation of the scrub to extraction height ratio. Approximately 35 ft. of the column is jacketed for temperature control. A schematic sketch of the columns and tabulated elevations is given in Figure 5, page 124.

3. Extraction Operations

The feed stream enters Extraction Column, B1, approximately 13.75 ft. below the solvent overflow point. Scrub was pumped from Scrub Feed Tank M4 through a heat exchanger to a point on B1 10.8 ft. above the feed point. Solvent was pumped from Solvent Feed Tank M5 through the Accumulator B3 and a heat exchanger into the bottom of the column countercurrent to the aqueous stream. The combined aqueous streams flow out the bottom of the column through the Jackleg Pressure Pot B2 to the Aqueous Waste Tank A12 in Cell A. Tank A12 is equipped with a Condenser A16 and a solvent-aqueous Separator A13 for flashing off solvent before transferring waste to the underground Thorium Storage Tank.

4. Extraction Column Temperature Control

Extraction column temperature was controlled by recirculating water through a closed system temperature control unit outside the cell and through column jacket and feed stream heat exchangers. The temperature control unit consists of a recirculating pump, a constant head tank, a hot water reservoir and a chilled water reservoir. Control valves actuated by the B1 Column Temperature Recorder Controller permit recycling of water through the appropriate reservoir to maintain constant column temperature.

5. Stripping Column

The solvent containing essentially all of the 23 overflows from Column B1 and entered the bottom of the Stripping Column, B5. The column is a vertical 1 1/2 inch stainless steel pipe packed with 1/4 x 3/8 inch Raschig rings. The packed height is 18 ft. and the overall length is approximately 23 ft.

6. Stripping Operations

Strip solution, pumped from Strip Feed Tank M6, enters Column B5 approximately 3.8 ft. below the organic overflow. This aqueous stream flows down the column removing essentially all the 23 from the solvent, leaves the bottom of the column and flows to the product Catch Tank B10 via the Jackleg Pressure Pot B6. The stripped solvent overflows from the column and was collected in the Solvent Catch Tank B9. Solvent was pumped from B9 to the solvent recovery equipment in Cell C for purification before reuse.

7. Column Interface Control

The position of the interface in the upper unpacked sections of both the extraction and stripping column was detected by a set of bubbler probes positioned approximately 2 ft. apart. The differential pressure was proportional to interface position, and was recorded by a Taylor Fulscope Recorder-Controller which supplied controlled air directly to the extraction column jackleg pressure pot and to the stripping column jackleg pressure pot via a Moore Products Co. 5:1 reducing relay. As the interface position varied from the set point, the controller varied the pressure head on the exit aqueous stream controlling its flow from the column and returning the interface to normal.

D. Product Concentration

The aqueous product solution in Product Catch Tank B10 was steam jetted to Evaporator B11, which is a conical jacketed tank equipped with a Condenser B12. Condensate was collected in Condensate Catch Tank B13 for sampling and disposal and the concentrated product solution, having a volume of one liter or less, was transferred by vacuum to a glass bottle.

Concentrated product solution was clarified by laboratory filtration through a medium porosity sintered glass filter. The filtrate was carefully weighed, sampled, and stored pending further concentration and cleanup in the laboratory.

E. Waste Disposal

1. Gaseous Waste

Gaseous wastes, consisting of instrument, sampler, sparger, and leakage air and dissolver fumes were dispersed to the atmosphere through a building height 4" pipe stack. The off gas line was provided with a heater to prevent condensation and a blower to maintain a reduced pressure in the cell vessels. Fumes from the dissolver condenser passed through a caustic scrubber, an air jet, and then into the off gas system. The jet supplied vacuum during dissolver sparging or in case of blower failure. Air discharge from the A5 and A8 vacuum tank jets is piped directly into the cell ventilation air exhaust ducts. Glass wool packed filters on these two suction jet lines remove entrained activity.

2. Liquid Wastes

Chemical Waste, consisting of coating removal solution, low 23 content filter washes, and evaporation wastes were steam jetted to Waste Neutralizer A11 and then to a stainless steel, underground waste storage tank. No neutralization was done as the dissolver off gas scrubber liquid, which drains directly to the

storage tank, contained an excess of caustic. The underground storage tank was periodically pumped to the tank farm for disposal.

Thorium waste, after being flashed to remove dissolved solvent, was steam jetted to a stainless steel, underground storage tank where it will be held for thorium recovery.

Cell floor drains and the solvent pretreatment recovery system lines drain to the settling pond because of the low activity content. Jacket and condenser discharge lines go to the sanitary waste system.

F. Sampling

Tanks and all column flowing streams, except the product bearing solvent stream (AP) were sampled by recirculating CL #5 air jet samplers mounted in lead shields on the cell faces accessible from the 20 ft. operating level. The AP stream was sampled by a valved drain tap on the external AP line. The other flowing stream samplers recirculate from small holdup pots in the raffinate and product lines.

G. Instrumentation

1. Panel Boards

Panel boards, which combine both instrumentation and services for process control, are located on the ground floor and the 10 ft. level. The ground level board controls the extraction column temperature control unit and the other two boards, located on the 10 ft. level, control operations in Cell A and in Cells B and C.

2. Pressure Gages

Pressure gages for panel board headers, process services, and jets are flush mounted, bourdon tube type Ashcroft Gages.

3. Liquid Level and Specific Gravity

Liquid level and specific gravity instruments of both recording and indicating types were used. Two pen, aneroid bellows, Taylor Instrument Company recorders were used for recording liquid level and specific gravity for the Dissolver, the Extraction Column Feed Tank, and the three Column Catch Tanks. All other liquid level and specific gravity instruments are well type Meriam Manometers. Air for instrument probes was supplied through Moore Constant Differential Relays to Fisher-Porter C clamp rotameters for metering to the tank probes. Meriam Check valves are provided the low pressure side of all manometers.

4. Temperature

Temperatures, determined by I. C. thermocouples and resistance elements, were indicated and recorded by two Leeds and Northrup Co. Micromax recorders, and one Brown Inst. Co. Recording Resistance thermometer. A Brown Inst. Co. Multipoint Electronik Temperature Indicator indicated temperatures of the extraction column temperature control system and cell C items. The extraction column temperature was controlled and recorded by a Brown Electronik temperature controller.

5. Interface Control

Taylor Fulscope aneroid bellows recording controllers were used for column interface control. The bubbler lines have the same accessories as the liquid level and specific gravity instruments. Extraction column control air came directly from the instrument and stripping column air went to a Moore Products Co. 5:1 reducing relay which fed air to the pressure pot.

6. Overflow Alarms

Moore Products Co. electric liquid level controllers were mounted on tanks A5 and A8 and operate warning lights and a howler.

APPENDIX III

23 Process Calculations

A. Calculation of the Amount of 23 and Pa^{233} Produced by the Neutron Irradiated of Thorium Metal

The following equations are developed in detail using these symbols:

N_{02} - Amount of thorium metal (grams)

N_{13} - Amount of Pa^{233} (grams)

N_{23} - Amount of U^{233} (grams)

$\sigma_c(Th)$ - Capture cross section of Th^{232} (7 barns $\pm$ 10%)

$\sigma_a(23)$ - Fission cross section plus capture cross section of U^{233} (560 barns)

λ_{13} - Decay constant of Pa^{233} = $\ln 2/T_{1/2}$

t - Exposure time in pile (days)

t_c - Cooling time (days)

nv - neutron flux

(1) $N_{23} = N_{02}\ \sigma_c(Th)\ nvt \pm 14\%$ (for $t \geq 400$ days, $t_c = 0$ or $t \cong 100$ days, $t_c > 45$ days)

(2) $N_{23} = N_{02}\ \sigma_c(Th)\ nvt - \dfrac{N_{02}\ \sigma_c(Th)\ nv}{} \left[F\left(\dfrac{t}{T_{1/2}}\right) \right]^{*} e^{-\lambda_{13} t_c}$ **

(for $t < 400$ day, $t_c = 0$) or ($t \cong 100$ days, $t_c < 45$ days)

(3) $N_{13} = \dfrac{N_{02}\ \sigma_c(Th)\ nv}{\lambda_{13}} \left[F\left(\dfrac{t}{T_{1/2}}\right) \right]^{*}$ (no cooling)

(4) $N_{13} = \dfrac{N_{02}\ \sigma_c(Th)\ nv}{\lambda_{13}} \left[F\left(\dfrac{t}{T_{1/2}}\right) \right]^{*} e^{-\lambda_{13} t_c}$ ** (cooled for t_c days)

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* F is given in Table III-1 (page) as a function of $t/T_{1/2}$

** $e^{-\lambda_{13} t_c}$ is given in Table III-2 (page) as a function of $t/T_{1/2}$

The reaction is as follows:

$$_{90}Th^{232} \xrightarrow{(n,\gamma)\ \sigma c = 7\ \text{barns}} {}_{90}Th^{232} \xrightarrow[23.5m]{\beta} {}_{91}Pa^{233} \xrightarrow[27.4d]{\beta} {}_{92}U^{233}$$

Since the half-life of Th^{233} is small compared to that of Pa^{233} and can be neglected, the rate of Pa^{233} production is $N_{02}\ \sigma c(Th)\ nv$ and the rate Pa^{233} decay is given by $\lambda_{13}N_{13}$, thus the rate of Pa^{233} accumulation is

$$\frac{dN_{13}}{dt} = N_{02}\,\sigma_c(Th)\ nv - \lambda_{13}N_{13}.$$

By inspection a particular integral of this equation is a constant and by substitution this constant is found to be

$$\frac{N_{02}\,\sigma_c(Th)nv}{\lambda_{13}}$$

The solution of the homogeneous part of the equation is found to be $c\ e^{-\lambda_{13}t}$. Thus,

$$N_{13} = \frac{N_{02}\,\sigma_c(Th)nv}{\lambda_{13}} + C\ e^{-\lambda_{13}t}$$ is a solution of the differential equation.

When

$$t = 0,\ N_{13} = 0 \text{ and } C = -\frac{N_{02}\,\sigma_c(Th)nv}{\lambda_{13}}$$

and the desired solution is:

$$N_{13} = \frac{N_{02}\,\sigma_c\ (Th)nv}{\lambda_{13}}\ (1 - e^{-\lambda_{13}t}),$$

the amount of Pa^{233} present at the end of any bombardment time t. The factor $(1 - e^{-\lambda_{13}t})$ is given in Table III-1 as a function of $t/T_{1/2}$, so the equation can be written:

$$(3) \qquad N_{13} = \frac{N_{02}\,\sigma_c(Th)nv}{\lambda_{13}}\ F(t/T_{1/2})$$

where F is determined from Table III-1.

To determine the amount of "23" present at the end of a given bombardment time, it is only necessary to calculate the total "13" produced and subtract from it the amount of "13" present, thus:

$$N_{23} = N_{02}\sigma_c(Th)(nvt) - N_{13}$$

$$N_{23} = N_{02}\sigma_c(Th)(nvt) - \frac{N_{02}\sigma_c(Th)nv}{\lambda_{13}}(1-e^{-\lambda_{13}t})$$

If we wish to calculate the amount of Pa^{233} present after a given bombardment and a given cooling time t_c, we have only to apply $e^{-\lambda_{13}t_c}$ to N_{13} getting

$$N_{13} = \frac{N_{02}\sigma_c(Th)\ nv}{\lambda_{13}}(1-e^{-\lambda_{13}t})\ e^{-\lambda_{13}t_c}$$

and

$$N_{23} = N_{02}\sigma_c(Th)nvt - \frac{N_{02}\sigma_c(Th)nv}{\lambda_{13}}(1 - e^{-\lambda_{13}t})\ e^{-\lambda_{13}t_c}$$

using Table III-1 to determine $(1-e^{-\lambda_{13}t})$ and Table III-2 to determine $e^{-\lambda_{13}t_c}$. This equation becomes:

$$(2)\quad N_{23} = N_{02}\sigma_c(Th)\ nvt - \frac{N_{02}\sigma_c(Th)\ nv}{\lambda_{13}}\ F(t/T_{1/2})\ e^{-\lambda_{13}t_c}$$

Thus for one Kg. of thorium metal exposed to an average (nvt) of 6.2×10^{19} (exposure time 98.5 days)

$$(\lambda_{13} = \frac{.69}{27.4} = 2.52 \times 10^{-2})$$

$$N_{13} = \frac{10^3 \cdot 7 \times 10^{-24} \cdot 6.2 \times 10^{19}}{(2.52 \times 10^{-2}) \times 98.5}\quad 1 - e^{(-2.52 \times 10^{-2})\ 98.5}$$

$$= 1.7 \times 10^{-1}\ (1 - e^{-2.48})$$

$$= .16 \text{ grams of } Pa^{233}$$

and the amount of U^{233}

$$N_{23} = 10^3 \cdot 7 \times 10^{-24} \cdot 6.2 \times 10^{19} - .16$$

$$= .43 - .16 = 0.27 \text{ grams of "23"}$$

It should be emphasized that this calculation is correct to only ± 14%. The capture cross section of thorium is probably accurate ± 10% and the error in determining (nvt) is accurate ± 10%, and the probable error for the calculation is 14%.

Since the probable error is so large there is no objection to dropping the term for residual Pa^{233} from the calculation of "23" if the exposure time is of the order of 400 days on longer, or cooling time > 45 days.

$N_{23} = N_{02}\,\sigma_c(Th)$ nvt ± 14% for exposure of > 400 days or cooling time of 45 days for 100 day exposure.

Using the results of the foregoing method another, more elementary calculation can be made. Starting with the differential equation.

$$\frac{dN_{13}}{dt} = N_{02}\,\sigma_c(Th)nv - \lambda_{13}\,N_{13}$$ and realizing that at equilibrium

$$\frac{dN_{13}}{dt} = 0$$ we can solve for the equilibrium value of N_{13}.

$$N_{13}\ (\text{equilibrium}) = \frac{N_{02}\,\sigma_c(Th)(nv)}{\lambda_{13}}$$

Since the production of Pa^{233} is assumed constant we can determine from a table the percent equilibrium reached at the given exposure time and this percent of the equilibrium value is N_{13}. Then as the total amount of Pa^{233} produced, $N_{13}(\text{total}) = N_{02}\,\sigma_c(Th)nvt$, we can get N_{23} by difference.

Using the same example as before (1 Kg thorium, nvt = 6.19×10^{19} and exposure time = 98.5 days).

The equilibrium value of N_{13} is:

$$N_{13}\ (\text{equilibrium}) = \frac{10^3 \cdot 7 \times 10^{-24} \times 6.2 \times 10^{19}}{(2.5 \times 10^{-2})\ (98.5)}$$

$$= 0.17$$

[redacted]

and from the table for $\frac{98.5}{27.4} = 3.6$ half-lives the percent saturation is 92%.

Then:

$$N_{13} = .92 \times 0.17 \text{ grams}$$

$$\text{and } N_{23} = N_{02}\,\sigma_c(\text{Th})nvt - 0.16$$

$$= 0.43 - 0.16 = 0.27 \text{ grams.}$$

[redacted]

B. Calculation of Required Cooling Time and Required Pa Decontamination Factor

The required cooling time to reduce the 23 loss as undecayed Pa^{233} to .01% is a function irradiation time only. We want

$$\frac{N_{13}}{N_{23}} = .0001,$$

then from equations (1) and (4) of Appendix III-A.

$$\frac{N_{13}}{N_{23}} = \frac{N_{02} \quad \sigma_c(Th)\ nv \quad F\ (t/T_{1/2})}{\lambda_{13}\ N_{02} \quad c(Th)\ nvt} \cdot \frac{e^{-\lambda_{13}t_c}}{} = .0001$$

and $e^{-\lambda_{13}t_c} = \frac{.0001 \quad \lambda_{13}t}{F\ (t/T_{1/2})}$, this can be solved for t_c as a function of irradiation time, t. Values of t_c required for $\frac{N_{13}}{N_{23}} = .0001$ for various irradiation times are given below in tabular form.

Irradiation Time - Days	Cooling Time Required for $N_{13}/N_{23} = .0001$, days
1	360
27	350
55	340
110	320
1100	230

[redacted]

If we assume that the metal is cooled for the time required to reduce the 23 lost as undecayed 13 to .01%, and that the Pa gamma activity of the product 23 after chemical processing must not exceed 10% the gamma background from 23, the required Pa decontamination factor can be calculated.

$$\text{Pa Decontamination Factor} = \frac{\text{Pa } \gamma/\text{gram of 23 in the feed}}{\text{Pa } \gamma/\text{gram of 23 in the product.}}$$

$$= \frac{\dfrac{2.5 \times 10^{12} \text{ (specific beta activity of Pa)}}{2.8 \times 10^{3} \text{ (ratio of Pa beta to gamma)}} \times .0001\ (N_{13}/N_{23})}{.6\ (23\ \alpha \text{ background}) \times 10\%} = 1 \times 10^{6}$$

C. Calculation of Fission Product Decontamination Requirements

This calculation is an extension of the calculations of C. H. Hanson published in ORNL-34, and is based on the assumptions made by Hanson covering the similarity of the 23 fission spectrum to that of 25 which is well known.

To calculate the production of 23 fission products on a weight basis, one can start with equation (2) Appendix III-A, in the form

$$N_{23} = N_{02}\,\sigma_c\,(Th)nvt - \frac{N_{02}\,\sigma_c(Th)nv}{\lambda_{13}} + \frac{N_{02}\,\sigma_c\,(Th)nv\,e^{-\lambda_{13}t}}{\lambda_{13}}$$

which is an expression for the amount of 23 present at the end of a given irradiation time, t, and the amount of fission products produced is

$$N(F.P.'s) = \int_0^t \left[N_{02}\,\sigma_c\,(Th)\,nv\right]\left[\sigma_f\,(23)\,nv\right]\left[t - 1/\lambda_{13} + \frac{e^{-\lambda_{13}t}}{\lambda_{13}}\right] dt$$

$$= N_{02}\sigma_c\,(Th)\,\sigma_f\,(23)\,(nv)^2\left[t^2/2 - t/\lambda_{13} + \frac{(1 - e^{-\lambda_{13}t}}{\lambda_{13}^2}\right]$$

[redacted]

From this expression one can obtain the ratio of fission products to 23 (on a weight basis) by dividing by the amount of 23 produced.

$$\frac{N(F.P.'s)}{N_{23}} = \frac{N_{02}\sigma c(Th)\ \sigma_f(23)\ (nv)^2 \left[t^2/2 - t/\lambda_{13} + \left(\frac{1-e^{-\lambda_{13}t}}{\lambda_{13}^2}\right)\right]}{N_{02}\ \sigma c(Th)\ nvt}$$

$$= \sigma_f(23)\ nv \left[t/2 - 1/\lambda_{13} + \left(\frac{1-e^{-\lambda_{13}t}}{t\lambda_{13}^2}\right)\right]$$

which is the ratio of fission products to total 23.

Now by making the following assumption the required Ru^{106} decontamination factor can be determined.

(1) Due to the long half life of Ru^{106} (1 year) decay during irradiation can be neglected for irradiation times up to one year, and the error made will be less than a factor of 2.

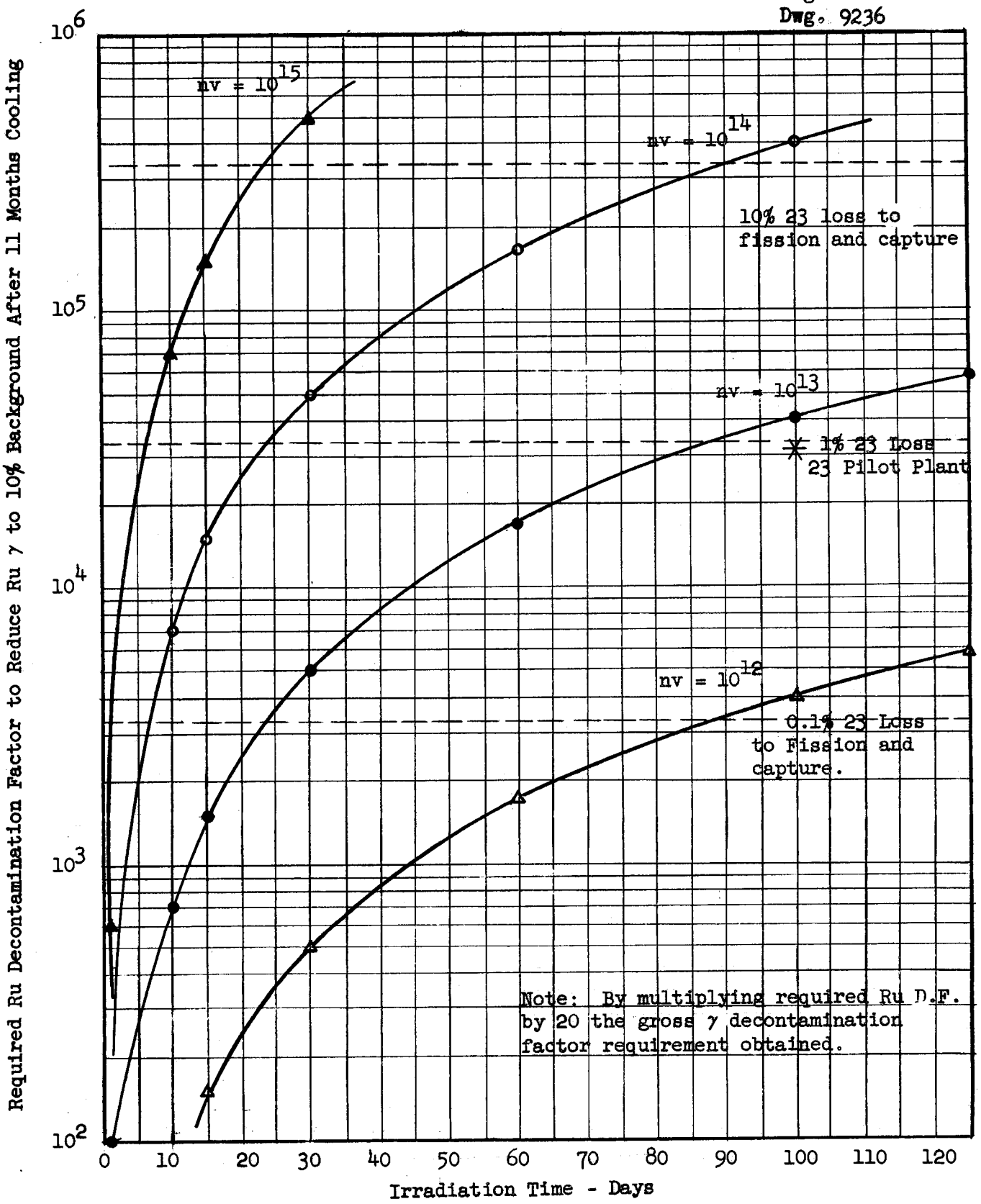
(2) The chemical processing will take place after eleven months cooling. By determining the Ru^{106} activity of metal with a known irradiation time and flux and correcting this to eleven months cooling, the specific activity (of eleven months cooled metal) can be obtained and the required Ru^{106} decontamination factor calculated. This was found to be 3×10^4 for 23 produced by 100 days irradiation at a flux of 8×10^{12} neutrons per cm^2 per second. (After 13 months cooling the metal contained 1.7×10^3 $n\gamma$ of Ru gamma/mg of 23). By combining this result with the equation for N_{FPs}/N_{23} one can determine a constant that will convert N_{FP}/N_{23} to decontamination factor required to reduce Ru^{106} gamma to 10% of the 23 gamma background after eleven months cooling. This conversion factor was found to be 3.7×10^6 and the expression

for Ru decontamination factor becomes,

$$\text{Required Ru D.F.} = \sigma_f\ (23)\ nv \left[t/2 - 1/\lambda_{13} + \frac{1-e^{-\lambda_{13}t}}{t\lambda_{13}^2} \right] 3.7 \times 10^6$$

This equation is represented graphically in Figure III-1 (page) as a function of irradiation time and at four different flux levels. It should be noted that the ordinate of this graph is also per cent of total 23 that is fissioned in the pile. Ru^{106} gamma makes up more than 5% of the total gamma from fission products after eleven months cooling and by multiplying the required Ru decontamination factor by 20, one arrives at a gross gamma fission product factor requirement. The reasons for choosing Ru as a basis for this calculation are that Ru is the limiting activity of the product for separations by MIBK extraction and Ru^{106} has a comparatively long half life.

Fig. III-1
Dwg. 9236



DECONTAMINATION REQUIREMENTS AS A FUNCTION
of
FLUX AND IRRADIATION TIME

TABLE III-1

Growth of a Radicelement
(Produced at constant rate)
F = Fraction of saturation value = $(1 - e^{-\lambda_{13} t})$

$t/T_{1/2}$	F	$t/T_{1/2}$	F	$t/T_{1/2}$	F	$t/T_{1/2}$	F
0	0	.78	.418	1.56	.661	2.85	.861
0.02	0.0138	.80	.426	1.58	.666	2.90	.866
.04	.0273	.82	.434	1.60	.670	2.95	.871
.06	.0407	.84	.441	1.62	.675	3.00	.875
.08	.0539	.86	.449	1.64	.679	3.10	.883
.10	.0670	.88	.457	1.66	.684	3.20	.891
.12	.0798	.90	.464	1.68	.688	3.30	.898
.14	.0925	.92	.471	1.70	.692	3.40	.905
.16	.105	.94	.479	1.72	.696	3.50	.912
.18	.117	.96	.486	1.74	.701	3.60	.918
.20	.129	.98	.493	1.76	.705	3.70	.923
.22	.141	1.00	.500	1.78	.709	3.80	.928
.24	.153	1.02	.507	1.80	.713	3.90	.933
.26	.165	1.04	.514	1.82	.717	4.00	.938
.28	.76	1.06	.520	1.84	.721	4.25	.947
.30	.188	1.08	.527	1.86	.725	4.50	.956
.32	.199	1.10	.533	1.88	.728	4.75	.963
.34	.210	1.12	.540	1.90	.732	5.00	.969
.36	.221	1.14	.546	1.92	.736	5.25	.974
.38	.232	1.16	.553	1.94	.739	5.50	.978
.40	.242	1.18	.559	1.96	.743	5.75	.981
.42	.253	1.20	.565	1.98	.747	6.00	.984
.44	.263	1.22	.571	2.00	.750	6.50	.989
.46	.273	1.24	.577	2.05	.759	7.00	.992
.48	.283	1.26	.582	2.10	.767	7.50	.994
.50	.293	1.28	.588	2.15	.775	8.00	.996
.52	.303	1.30	.594	2.20	.782	9.00	.998
.54	.312	1.32	.559	2.25	.790	10.00	.999
.56	.322	1.34	.605	2.30	.797		
.58	.331	1.36	.610	2.35	.804		
.60	.340	1.38	.616	2.40	.881		
.62	.349	1.40	.621	2.45	.817		
.64	.358	1.42	.626	2.50	.823		
.66	.367	1.44	.631	2.55	.829		
.68	.376	1.46	.636	2.60	.835		
.70	.384	1.48	.641	2.65	.841		
.72	.393	1.50	.646	2.70	.846		
.74	.401	1.52	.651	2.75	.851		
.76	.410	1.54	.656	2.80	.856		

TABLE III-2

TABLE OF RADIOACTIVE DECAY

$t/T_{1/2}$	$e^{-\lambda t}$	$t/T_{1/2}$	$e^{-\lambda t}$	$t/T_{1/2}$	$e^{-\lambda t}$	$t/T_{1/2}$	$e^{-\lambda t}$
0	1.0000	0.52	0.6974	1.54	0.3439	3.80	0.0718
0.01	0.9931	0.54	0.6878	1.56	0.3391	3.85	0.0693
0.02	0.9862	0.56	0.6783	1.58	0.3345	3.90	0.0670
0.03	0.9794	0.58	0.6690	1.60	0.3299	3.95	0.0647
0.04	0.9726	0.60	0.6597	1.62	0.3253	4.00	0.0625
0.05	0.9659	0.62	0.6507	1.64	0.3209	4.10	0.0583
0.06	0.9593	0.64	0.6417	1.66	0.3164	4.20	0.0544
0.07	0.9526	0.66	0.6329	1.68	0.3121	4.30	0.0508
0.08	0.9461	0.68	0.6242	1.70	0.3078	4.40	0.0474
0.09	0.9395	0.70	0.6156	1.75	0.2973	4.50	0.0442
0.10	0.9330	0.72	0.6071	1.80	0.2872	4.60	0.0412
0.11	0.9266	0.74	0.5987	1.85	0.2774	4.70	0.0385
0.12	0.9202	0.76	0.5905	1.90	0.2679	4.80	0.0359
0.13	0.9138	0.78	0.5824	1.95	0.2588	4.90	0.0335
0.14	0.9075	0.80	0.5744	3.00	0.2500	5.00	0.0312
0.15	0.9013	0.82	0.5664	2.05	0.2415	5.10	0.0292
0.16	0.8950	0.84	0.5586	2.10	0.2333	5.20	0.0272
0.17	0.8888	0.86	0.5509	2.15	0.2253	5.30	0.0254
0.18	0.8827	0.88	0.5434	2.20	0.2176	5.40	0.0237
0.19	0.8766	0.90	0.5359	2.25	0.2102	5.50	0.0221
0.20	0.8705	0.92	0.5285	2.30	0.2031	5.60	0.0206
0.21	0.8645	0.94	0.5212	2.35	0.1961	5.70	0.0192
0.22	0.9596	0.96	0.5141	2.40	0.1895	5.80	0.0179
0.23	0.8526	0.98	0.5070	2.45	0.1830	5.90	0.0167
0.24	0.8467	1.00	0.5000	2.50	0.1768	6.00	0.0156
0.25	0.8409	1.02	0.4931	2.55	0.1708	6.20	0.0136
0.26	0.8351	1.04	0.4863	2.60	0.1649	6.40	0.0118
0.27	0.8293	1.06	0.4796	2.65	0.1593	6.60	0.0103
0.28	0.8236	1.08	0.4730	2.70	0.1539	6.80	0.0090
0.29	0.8179	1.10	0.4665	2.75	0.1487	7.00	0.0078
0.30	0.8122	1.12	0.4601	2.80	0.1436	7.20	0.0068
0.31	0.8066	1.14	0.4538	2.85	0.1387	7.40	0.0059
0.32	0.8011	1.16	0.4475	2.90	0.1340	7.60	0.0052
0.33	0.7955	1.18	0.4413	2.95	0.1294	7.80	0.0045
0.34	0.7900	1.20	0.4353	3.00	0.1250	8.00	0.0039
0.35	0.7846	1.22	0.4293	3.05	0.1207	8.20	0.0034
0.36	0.7792	1.24	0.4234	3.10	0.1166	8.40	0.0030
0.37	0.7738	1.26	0.4175	3.15	0.1127	8.60	0.0026
0.38	0.7684	1.28	0.4118	3.20	0.1088	8.80	0.0022
0.39	0.7631	1.30	0.4061	3.25	0.1051	9.00	0.0020
0.40	0.7579	1.32	0.4005	3.30	0.1015	9.20	0.0017
0.41	0.7526	1.34	0.3950	3.35	0.0981	9.40	0.0015
0.42	0.7474	1.36	0.3896	3.40	0.0948	9.60	0.0013
0.43	0.7423	1.38	0.3842	3.45	0.0915	9.80	0.0011
0.44	0.7371	1.40	0.3789	3.50	0.0884	10.00	0.0010
0.45	0.7320	1.42	0.3737	3.55	0.0854	10.50	0.0007
0.46	0.7270	1.44	0.3685	3.60	0.0825	11.00	0.0005
0.47	0.7220	1.46	0.3635	3.65	0.0797	11.50	0.0004
0.48	0.7170	1.48	0.3585	3.70	0.0770	12.00	0.0002
0.49	0.7120	1.50	0.3536	3.75	0.0743	13.00	0.0001
0.50	0.7071	1.52	0.3487				

APPENDIX IV

Analytical Procedures

A. 23 Acridine Procedure

Reagents

Acridine - 7.5 gms/375 ml. conc. HCl - Dilute to liter

NH_4SCN 1M - 76 gms/liter

3N HCl - 256.5 ml. conc. HCl/liter

1N HNO_3 - 67 ml. conc. HNO_3/liter

Uranium tracer - 128 mg. of depleted U/100 ml

Procedure

1. Pipette an aliquot of the sample (containing about 5000 α c/m) not greater than 1 ml. in a 3 ml. glass centrifuge conc. (If micro pipette is used wash well with 3N HCl.)
2. Add 100 γ of uranium carrier. Agitate thoroughly with platinium rod.
3. Add 10 drops of acridine, agitate and cool in ice bath for 3 minutes.
4. Add 4 drops of 1M NH_4SCH, agitate and cool in ice bath for 5 minutes. Centrifuge for 5 minutes and draw off supernate.
5. Add 3 drops 3N HCl, agitate and place in hot water bath until ppt. dissolves. Remove from water bath and add 3 drops acridine, 4 drops NH_4SCN and cool in ice bath for 2 minutes. Centrifuge

5 minutes. Draw off supernatant.

6. Dissolve precipitate in 3 drops of 1N HNO_3. Warm in hot water batch until ppt. is dissolved.

7. Transfer to clean platinum plate. Wash cone with 3-5 drops of 1N HNO_3, scrubbing sides of cone thoroughly with platinum rod. Rinse transfer pipette with 1N HNO_3.

8. Evaporate to dryness beneath lamp. Burn off plate and determine 23 by alpha counting. (See note at bottom of page).

B. 23 Extraction Procedure

Reagents:

1.6 M $Al(NO_3)_3$ made to a pH of 1.9 with NH_4OH

Methyl isobutyl ketone

Procedure

1. Place an aliquot of the sample (containing about 5000 α c/m, but not more than 2 ml) in a 50 ml separatory funnel.

2. Add 5 ml of $Al(NO_3)_3$ solution.

3. Extract for 10 minutes with 10 ml of methyl isobutyl ketone.

4. Evaporate an aliquot of the extract of dryness beneath lamp, and determine 23 by alpha counting.

C. Colorimetric Thorium

Reagents:

R_2 arsonic acid (2-hydroxyl-3,6-sulfonaphthaleneazobenzene-2-arsonic acid) 0.15 grams/100 ml distilled water.

NOTE: Results should be corrected for the alpha activity introduced as carrier.

Conc. HCl

Conc. HNO_3

Conc. $HClO_4$

Procedure:

1. Pipette an aliquot of sample containing 5-85 micrograms of thorium into a 10 ml. glass-stoppered volumetric flask.
2. Add 5 drops of conc. HCl and shake thoroughly.
3. Add (volumetric pipette) 1.0 ml. of R_2-arsonic acid, wash sides of flask with distilled water and shake thoroughly.
4. Dilute to mark with distilled water and shake thoroughly.
5. Read on Beckman Model DU Quartz Spectrophotometer. Measure at 545 mμ against a reference solution. (This reference solution or Blank is made by adding 5 drops of conc. HCl and 1.0 ml. of the R_2 arsonic acid to a 10 ml. glass-stoppered volumetric flask and making to volume with distilled water.)
6. Thorium concentration is determined from a standard curve.

Interferences:

1. Sodium fluoride - the minimum amount of fluoride which can be tolerated is 1 part of fluoride to 300 parts of thorium. If the amount of fluoride is greater, the following procedure must be followed:
   (a) Take to dryness on a hot plate and make one or more evaporations with conc. HNO_3.

(b) Add 0.5 ml. of conc. HNO_3 and 1 ml. of 70% perchloric acid and evaporate to dryness.

(c) Add 1 ml. of 70% perchloric acid and again evaporate to dryness.

(d) Take up in 5 drops conc. HCl. Allow to stand for several minutes. If an excessive amount of aluminum is present, it is advisable to add a small amount of water and allow to digest on a hot plate until the aluminum goes into solution.

(e) Transfer to a 10 ml. glass-stoppered volumetric flask.

2. Organic Material

(a) Diethyl ether-evaporate on hot water bath for 5 minutes or until any trace of the ether has been removed.

(b) Dibutyl cellosolve (or hexone) - use procedure outlined for removing the fluoride interference.

3. Ferric Iron - boil sample (free from perchloric acid) for two or three minutes with 1 ml. of 10% hydroxylamine.

D. Pa^{233} Radiochemical Analysis

Reagents

Conc. HCl

6 N HCl

6 N HNO_3

DIPC, diisopropyl carbinol, equilibrated with equal volume of 6 N HCl.

Procedure

1. Pipette into a 125 ml separatory funnel a sample of appropriate size. Dilute to 2 ml with water and add 2 ml of conc. HCl.
2. Add 4.0 ml of equilibrated DIPC and shake 10 minutes.
3. Draw off and discard the lower (aqueous) phase taking care not to discard any DIPC.
4. Add ~4 ml of 6 N HCl and shake for 2 minutes. Draw off and discard aqueous phase.
5. Add ~4 ml of 6 N HNO_3 and shake for 2 minutes. Draw off and discard aqueous phase.
6. Repeat 5.
7. Support a watch glass on a large washer on a hot plate so that it is heated mostly near the edge. The temperature of the hot plate must be maintained low enough to avoid boiling the DIPC. Pipette 500 λ of the DIPC (from step 6) on the watch glass.
8. Allow the DIPC to evaporate to a small volume, then rinse the pipette with DIPC and add the rinse to the watch glass.

9. Mount watch glass and determine Pa^{233} concentration by beta counting.

Notes:

The above procedure was developed by S. A. Reynolds of the ORNL Chemistry Division and was used to obtain the Pa results given in this report. This procedure is applicable to solutions in which Pa contributes 1% or more of the gross beta activity. Principal interferences were due to U^{233}, Nb and Ru. Decontamination from a two year old fission product mixture was about 10^3, but was somewhat less for Nb and Ru. The decontamination from U^{233} was about 10^4.

A procedure is now being developed which will give better separation of Pa^{233} from other beta activities.

APPENDIX V

Process Raw Materials

S.S. Solvent This is a specially purified methyl isobutyl ketone made by the Shell Chemical Company. This solvent was used on runs 1T-15T.

Methyl Isobutyl Ketone (MIBK) This material was used for runs 16T - 18T without pretreatment and with 0.5% methyl isobutyl carbinol added.

Diisopropyl Ether This solvent was obtained in 55 gallon drums and was pretreated by steam distillation from a caustic heel.

Aluminum Nitrate Aluminum Nitrate of C.P. quality was used in the 23 Pilot Plant. Solutions of this material were filtered before being used for scrub solutions.

Caustic Two grades of sodium hydroxide were used in the 23 Pilot Plant. Technical grade, 50% liquid caustic was used for MIBK distillation, jacket removal, dissolver off gas scrubbing, and decontamination. Sodium hydroxide pellets of C.P. quality were used for acid adjustment of process solutions.

Fluoride The catalyst for metal dissolution was supplied by adding either concentrated hydrofluoric acid or C.P. sodium fluoride to the 65% HNO_3 used for dissolving.

Other Raw Materials Demineralized water, C.P. nitric acid, and C.P. sodium dichromate were used for process requirements and solvent treatment.

APPENDIX VI

Objectives of Runs and Run Conditions

Except for the variations noted below, the 23 flowsheet, shown in Figure 3.0-1 was used for all runs. The column consisted of a 30 ft. extraction section and 11 ft. of scrub section. The major objectivies of individual runs were as follows:

1T-2T - To make a preliminary process evaluation.

3T - To determine protactinium decontamination. Three ORNL slugs irradiated six weeks and cooled six weeks were used as a Pa spike.

4T-10T - To investigate the effect of feed thorium concentration on decontamination. Runs were made at 1.3 M, 1.4 M, 1.6 M and 1.8 M $Th(NO_3)_4$.

11T - To investigate the effect of low temperature on decontamination. The extraction column was maintained at 10°C during this run.

12T-13T - To determine the effect of increasing the scrub height from 11' to 25' and decreasing the extraction section length from 30' to 16'.

14T-15T - To investigate the effect of varying the acid deficiency of the metal feed on decontamination. Run 14T was made with a metal feed 0.05 N acid deficient, and Run 15T with a feed 0.20 N acid deficient.

16T - To investigate the effect of gross amounts of solvent impurities on decontamination. The solvent used was untreated methyl isobutyl ketone to which 0.5% methyl isobutyl carbinol was added.

17T - To determine the HETS for 23 extraction at 15% of flooding capacity. The column consisted of 9.5 ft. of extraction section with 18 ft. of scrub section.

18T - To determine the HETS for the 23 extraction at 50% of flooding capacity and the effect on decontamination of increased flowrate, and to test equipment performance at 50% of the flooding rate (flooding occurs at about 800 gal per hour per ft^2.).

19T - To evaluate the diisopropyl ether flowsheet.

APPENDIX VII

[redacted]

TABLE I

ORNL-715

Summary of Dissolving Data

Dissolving No.	% HNO_3	Dissolving Time-Hrs.	$\frac{M\ HNO_3}{M\ Th}$	Final Sp.G. at ~110°C	Final (1) Acidity, N	Th Metal Heel, Kg
1T	65	26	5.9	2.03	0.14b	40
2T	65	30	5.8	2.04	0.02b	45
3T-1	65	33	7.3	-	0.10b	45
3T-2	65	25	6.5	2.10	0.20b	40
4T	65	31	5.7	2.01	0.25b	30
5T	65	28	5.7	2.06	0.06b	75
6T	65	33	6.5	2.15	0.40b	115
7T	65	30	5.5	2.07	0.38b	65
8T	65	29	5.0	2.12	1.1 b	65
9T	65	29	5.7	2.07	0.42b	70
10T	65	28	5.6	2.05	0.44b	60
11T	65	29	5.7	2.06	0.44b	50
12T	65	19	5.7	2.05	0.18b	60
13T	65	23	5.8	2.09	0.31b	55
14T	65	29	6.2	2.08	0.20b	50
15T	65	30	5.9	2.09	0.28b	60
16T	65	42	5.4	2.05	0.30b	70
17T	65	13	5.7	2.09	0.00b	45
18T	65	12	5.7	2.09	0.24a	58

(1) b denotes acid deficient.

(2) Moles of HNO_3 consumed per mole of thorium dissolved.

[redacted]

[redacted]

TABLE II

23 Losses

Run No.	Conc. of $Th(NO_3)_4$ in AF, Molar	Extraction Length, Feet	23 Conc. in AR mg/liter	% 23 Loss		
				AR	BR	Dissolver Residue(1)
1T	1.6	30	0.09	0.14	0.002	0.0
2T	1.5	30	0.10	0.18	0.004	0.05
3T	1.6	30	0.08	0.12	0.009	0.06
4T	1.6	30	0.09	0.08	0.002	0.02
5T	1.8	30	0.12	0.10	0.001	0.07
6T	1.9	30	0.13	0.06	0.001	0.10
7T	1.8	30	0.14	0.10	0.003	0.09
8T	1.4	30	0.06	0.08	0.002	0.06
9T	1.2	30	0.07	0.06	0.002	0.24
10T	1.6	30	0.08	0.05	0.001	0.08
11T	1.6	30	0.10	0.07	0.006	0.05
12T	1.6	16	0.09	0.06	0.003	0.02
13T	1.6	16	0.09	0.06	0.008	0.03
14T	1.6	30	0.07	0.04	0.006	0.01
15T	1.6	30	0.07	0.05	0.003	0.01
16T	1.6	30	0.06	0.04	0.003	0.01
17T	1.6	9.5	0.03	0.10	0.002	0.02
18T (2)	1.6	9.5	0.08	0.05	0.005	0.02
19T	2.5 (3)	30	0.03	0.01	0.04	0.02

(1) This was the fraction of 23 lost with the coating removal solution and was due to insufficient washing of the dissolver after the dissolver solution was transferred to the mix tank.

(2) Run 18T was made at 50% of flooding, all other runs made at 15% of flooding.

(3) Run 19T was made with diisopropyl ether as solvent.

[redacted]

TABLE III

Summary of Decontamination Date From Pilot Plant Runs 1T - 19T

(See Tables IV and V Appendix For Complete Decontamination Data) Run Conditions - 23 Flowsheet (Figure 3.0-1) with exceptions given in Table. All runs at 15% of flooding except 18T made at 50% of flooding.

Run No.	Feed Acid Deficiency Normal	Feed $Th(NO_3)_4$ Conc. Molar	Length of Scrub Section Feet	Decontamination Factors						
				Gross Beta $x10^{-4}$	Gross Gamma $x10^{-4}$	Ru Beta $x10^{-3}$	Cr Beta $x10^{-5}$	Zr Beta $x10^{-4}$	Pa Beta $x10^{-5}$	Thorium $x10^{-4}$
1T	0.12	1.6	11	1.4	1.4	0.9	1	(b)	(b)	1
2T	0.14	1.6	11	1.9	2	1.4	1	(b)	(b)	1
3T	0.24	1.5	11	4	(a)	5	3	1.7	10	3
4T	0.21	1.6	11	4	2	2	2	(b)	1	7
5T	0.19	1.8	11	0.2	0.13	0.1	1	(b)	0.5	10
7T	0.22	1.9	11	2	1.2	0.6	5	(b)	(b)	3
8T	0.22	1.5	11	4	3	4	5	(b)	(b)	2
9T	0.19	1.3	11	6	5	4	5	(b)	(b)	20
10T	0.20	1.6	11	3	3	2	5	(b)	(b)	20
11T	0.20	1.6	11	2	3	1.4	3	(b)	(b)	30
12T	0.20	1.6	25	8	4	3	8	(b)	(b)	20
13T	0.22	1.6	25	7	4	6	8	(b)	(b)	20
14T	0.05	1.6	11	0.13	0.12	0.04	8	(b)	(b)	12
15T	0.30	1.6	11	5	2	1.5	5	(b)	(b)	17
16T	0.18	1.6	11	3	3	1.5	6	(b)	(1)	13
17T	0.15	1.6	18	7	4	1.0	1	20	4	10
18T	0.22	1.6	18	5	5	2	2	14	6	4
19T	Diisopropyl Ether Flowsheet		11	17(c)	13(c)	10	60	16	13	500

(a) Insufficient data.
(b) Activity of product solution not significantly different from background.
(c) The beta and gamma activity of the product from Run 19T was not significantly different from 23 background, and the decontamination factors reported are minimum values.

[redacted]

[redacted]

TABLE IV

Gross Fission Product Activities And Decontamination Factors

Conditions of runs - 23 Flowsheet with exceptions given in table. All runs at 15% of flooding except 18T made at 50% of flooding.

Run No.	Feed Acid Defic. Normal	Feed $Th(NO_3)_4$ Conc. Molar	Length of Scrub Section Feet	Gross Activity of the Metal Feed Counts/m/ml or mv/ml			Specific Fission Product Activity of Feed (b)		Specific Activity of BP β c/m/mg of 23 or γ mv/mg of 23				Gross Fission Product Decontamination Factor	
				Beta $x10^{-7}$	Gamma 23 mv $x10^{-3}$	Alpha $x10^{-6}$	Beta c/m/mg of 23 $x10^{-4}$	Gamma mv/mg of 23 $x10^{-4}$	Gross β $x10^{-3}$	F.P. Beta (c) $x10^{-3}$	Gross γ	F.P.(c) Gamma	Beta $x10^{-4}$	Gamma $x10^{-4}$
1T	0.12	1.6	11	0.3	0.6	0.92	3	0.7	5	2	1.0	0.5	1.4	1.4
2T	0.14	1.6	11	0.4	1	0.97	4	1.1	5	2	1.1	0.5	1.9	2
3T	0.24	1.5	11	3	15	1.0	8	(a)	5	2	1.1	0.5	4	(a)
4T	0.21	1.6	11	2	8	1.7	9	2.5	5	2	1.9	1.3	4	2
5T	0.19	1.8	11	2	4	2.0	8	1.0	34	31	8	7	0.2	0.13
7T	0.22	1.9	11	1.8	4	2.1	9	1.9	7	4	2.2	1.6	2	1.2
8T	0.22	1.5	11	1.4	2	1.8	8	1.5	5	2	1.2	0.6	4	3
9T	0.19	1.3	11	1.1	2	1.5	8	1.5	4	1	0.7	0.3	6	5
10T	0.20	1.6	11	1.4	2	2.1	7	1.2	5	2	1.0	0.5	3	3
11T	0.20	1.6	11	1.4	2	2.1	7	1.3	6	3	1.0	0.5	2	3
12T	0.20	1.6	25	1.5	3	2.2	8	1.4	4	1	0.9	0.4	8	4
13T	0.22	1.6	25	2	3	2.3	10	1.6	4	1	0.8	0.4	7	4
14T	0.05	1.6	11	2	3	2.4	10	1.4	80	80	12	11	0.13	0.12
15T	0.30	1.6	11	2	3	2.1	11	1.4	5	2	1.1	0.5	5	2
16T	0.18	1.6	11	2	3	2.3	11	1.6	6	3	1.0	0.5	3	3
17T	0.15	1.6	18	3	5	2.2	13	1.9	5	2	1.0	0.5	7	4
18T	0.22	1.6	18	3	6	2.0	13	2.6	6	3	1.1	0.5	5	5
19T	Diisopropyl Ether			4	11	2.8	17	4	3	1	0.7	0.3	17(d)	13(d)

(a) Insufficient data.
(b) Obtained by subtracting Pa activity from gross activity and dividing by 23 concentration.
(c) Obtained by subtracting Pa and 23 β (or γ) activity from gross activity and dividing by 23 concentration.
(d) The beta and gamma activity of the product from Run 19T (DIPE used as solvent) was not significantly different from 23 background, and the decontamination factors reported are minimum values.

[redacted]

TABLE V

Specific Fission Product, Pa and Thorium Decontamination Factors

Run Number	1T	2T	3T	4T	5T	7T
Ru Beta						
AF cts/min/mg of 23	$8x10^5$	$1.3x10^6$	$1.6x10^6$	$2x10^6$	$3x10^6$	$3x10^6$
BP cts/min/mg of 23	900	900	300	$1:1x10^3$	$3x10^4$	$4x10^3$
Decontamination Factor	900	$1.4x10^3$	$5x10^3$	$2x10^3$	100	600
Ce Beta						
AF cts/min/mg of 23	$1.6x10^7$	$3x10^7$	$4x10^7$	$5x10^7$	$5x10^7$	$5x10^7$
BP cts/min/mg of 23	150	400	130	200	400	100
Decontamination Factor	$1x10^5$	$1x10^5$	$3x10^5$	$2x10^5$	$1x10^5$	$5x10^5$
Zr Beta						
AF cts/min/mg of 23	$8x10^4$	$4x10^4$	$8x10^6$	$5x10^5$	$9x10^5$	$2x10^5$
BP cts/min/mg of 23	(1)	(1)	500	(1)	(1)	(1)
Decontamination Factor	-	-	$1.7x10^4$	-	-	-
Pa Beta						
AF cts/min/mg of 23	10^4	10^4	$4x10^8$	$6x10^7$	$3x10^7$	$8x10^5$
BP cts/min/mg of 23	(1)	(1)	400	500	600	(1)
Decontamination Factor	-	-	$1x10^6$	$1x10^5$	$5x10^4$	-
Thorium						
Decontamination Factor	$1x10^4$	$1x10^4$	$3x10^4$	$7x10^4$	$1x10^5$	$3x10^4$

(1) Activity not significantly different from background.

[redacted]

Table continued on next page.

Table V (continued)

Run Number	8T	9T	10T	11T	12T	13T
Run Beta						
AF cts/min/mg of 23	$2x10^6$	$2x10^6$	$2x10^6$	$2x10^6$	$2x10^6$	$3x10^6$
BP cts/min/mg of 23	500	500	$1.1x10^3$	$1.5x10^3$	800	500
Decontamination Factor	$4x10^3$	$4x10^3$	$2x10^3$	$1.4x10^3$	$3x10^3$	$6x10^3$
Ce Beta						
AF cts/min/mg of 23	$5x10^7$	$5x10^7$	$5x10^7$	$5x10^7$	$6x10^7$	$7x10^7$
BP cts/min/mg of 23	70	80	70	140	70	80
Decontamination Factor	$5x10^5$	$5x10^5$	$5x10^5$	$3x10^5$	$8x10^5$	$8x10^5$
Zr Beta						
AF cts/min/mg of 23	$1.3x10^5$	$1.1x10^5$	$1.2x10^5$	$9x10^4$	$1.7x10^5$	$3x10^5$
BP cts/min/mg of 23	(1)	(1)	(1)	(1)	(1)	(1)
Decontamination Factor	-	-	-	-	-	-
Pa Beta						
AF cts/min/mg of 23	$3x10^6$	$1.1x10^6$	$7x10^5$	$7x10^5$	$1.6x10^5$	$8x10^4$
BP cts/min/mg of 23	(1)	(1)	(1)	(1)	(1)	(1)
Decontamination Factor	-	-	-	-	-	-
Thorium						
Decontamination Factor	$2x10^4$	$2x10^5$	$2x10^5$	$3x10^5$	$2x10^5$	$2x10^5$

[redacted]

Table continued on next page.

Table V (continued)

Run Number	14T	15T	16T	17T	18T	19T(2)
Run Beta						
AF cts/min/mg of 23	$3x10^6$	$3x10^6$	$3x10^6$	$3x10^6$	$4x10^6$	$4x10^6$
BP cts/min/mg of 23	$8x10^4$	$2x10^3$	$2x10^3$	$3x10^3$	$1.7x10^3$	400
Decontamination Factor	40	$1.5x10^3$	$1.5x10^3$	$1x10^3$	$2x10^3$	$1x10^4$
Ce Beta						
AF cts/min/mg of 23	$7x10^7$	$8x10^7$	$8x10^7$	$8x10^7$	$9x10^7$	$1.0x10^8$
BP cts/min/mg of 23	80	160	130	800	400	17
Decontamination Factor	$8x10^5$	$5x10^5$	$6x10^5$	$1x10^5$	$2x10^5$	$6x10^6$
Zr Beta						
AF cts/min/mg of 23	$4x10^5$	$4x10^5$	$8x10^5$	$3x10^6$	$5x10^6$	$5x10^6$
BP cts/min/mg of 23	(1)	(1)	(1)	15	40	30
Decontamination Factor	-	-	-	$2x10^5$	$1.4x10^5$	$1.6x10^5$
Pa Beta						
AF cts/min/mg of 23	$1.1x10^5$	$9x10^4$	$3x10^6$	$1.3x10^7$	$2x10^7$	$2.4x10^7$
BP cts/min/mg of 23	(1)	(1)	(1)	30	32	19
Decontamination Factor	-	-	-	$4x10^5$	$6x10^5$	$1.3x10^6$
Thorium						
Decontamination Factor	$1.2x10^5$	$1.7x10^5$	$1.3x10^5$	$1.0x10^5$	$4x10^4$	$5x10^6$

(2) Diisopropyl Ether Flowsheet.

[redacted]

[redacted]

TABLE VI

Material Balances

Material balances are based on composite samples.

Run No.	Thorium Material Balance - %				23 Material Balance %					Weight Balance of Input Solution to Catch Tank - %		
	Dissolver	Mix Tank	Feed Tank	Raff. Catch Tank	Dissolver	Mix Tank	Feed Tank	Product Catch Tank	Concent-rated Prod. Sol.	B1 Column Aqueous	Solvent	B5 Column Aqueous
1T	101	100	100	105	100	110	100	93	94	103	(1)	91
2T	100	100	98	102	100	105	91	108	104	104	91	99
3T	(1)	(1)	100	105	100	100	93	98	110	103	92	96
4T	100	102	104	98	100	112	100	100	94	102	88	99
5T	100	103	100	90	100	104	98	112	108	98	92	89
6T	100	99	102	94	100	104	101	107	110	105	88	(1)
7T	100	99	98	89	100	99	99	(1)	100	103	89	99
8T	100	105	105	101	100	110	102	111	108	104	88	112
9T	100	102	105	99	100	104	103	112	110	103	91	106
10T	100	100	101	97	100	101	101	104	101	98	87	100
11T	100	99	98	96	100	111	96	101	99	99	89	99
12T	100	100	100	96	100	99	101	(1)	96	98	91	101
13T	100	102	100	108	100	102	99	102	101	100	86	100
14T	100	102	100	110	100	101	101	105	99	99	88	99
15T	100	104	105	104	100	112	102	103	102	100	88	100
16T	100	103	102	103	100	93	98	102	97	97	93	101
17T	100	102	100	103	100	104	104	106	102	101	90	97
18T	100	104	100		100	99	99	105	100			
Average	100	102	101	99	100	103	99	105	102	101	90	99

(1) Data not complete.

[redacted]

[redacted]

TABLE VII

Isotopic Purity of Product

Analyses by Mass Spectrograph at Y-12

Run No.	Grams of 23	Isotopic Analyses of Product Expressed as % of Abundance				
		U^{233}	U^{234}	U^{235}	U^{236}	U^{238}
1T	5.66	98.4	0.20	0.3 (b)	0.04(b)	1.1
2T	8.60	98.3	0.25	0.03(b)	0.26(b)	1.1
7T	18.65	98.8	0.32	0.3 (b)	0.07(b)	0.6
8T	21.49	98.4	0.29	0.7 (b)	0.04(b)	0.7
9T	18.08	98.3	0.30	0.7 (b)	0.00(b)	0.8
10T	17.87	94.6	0.34	0.05	0.00	5.0(d)
11T	17.24	93.6	0.30	0.05	0.02	6.1
12T	18.86	94.6	0.32	0.04	0.00	5.1
13T	17.30	95.7	0.34	0.03	0.02	3.9(d)
14T	17.00	96.6	0.33	0.02	0.03	3.1
15T	15.97	97.0	0.33	0.02	0.02	2.6
16T	17.32	97.4	0.32	0.02	0.01	2.2
17T	15.04	97.6	0.30	0.02	0.00	2.1
18T	15.27	97.6	0.30	0.02	0.01	2.1
19T	22.32	97.8	0.29	0.01	0.00	1.9
20T(a)	27.82	98.9	0.26	0.01	0.01	0.8
21T(a)	43.98	98.4	0.28	0.01	0.01	1.3
Average	318.5 (c)	97.3	0.30			2.4

(a) The products from runs 3T, 4T, 5T, 6T were reprocessed in runs 20T and 21T.

(b) The instrument used for 1T-9T analyses had been previously used for other uranium isotopic analyses, and these unreasonable values are due to contamination present in the instrument.

(c) Another 15 grams of 23 was salvaged from samples, analytical wastes etc. which will be kept separate from this material until an isotopic analysis can be made.

(d) The increased U^{238} content beginning on Run 10T coincided with a change in buckets of thorium, metal and the decrease beginning on Run 13T coincided with the depletion of this bucket.

[redacted]

[redacted]

TABLE VIII

Fission Product, Protactinium and Thorium Contamination

of Product Runs Made With 23 Flowsheet

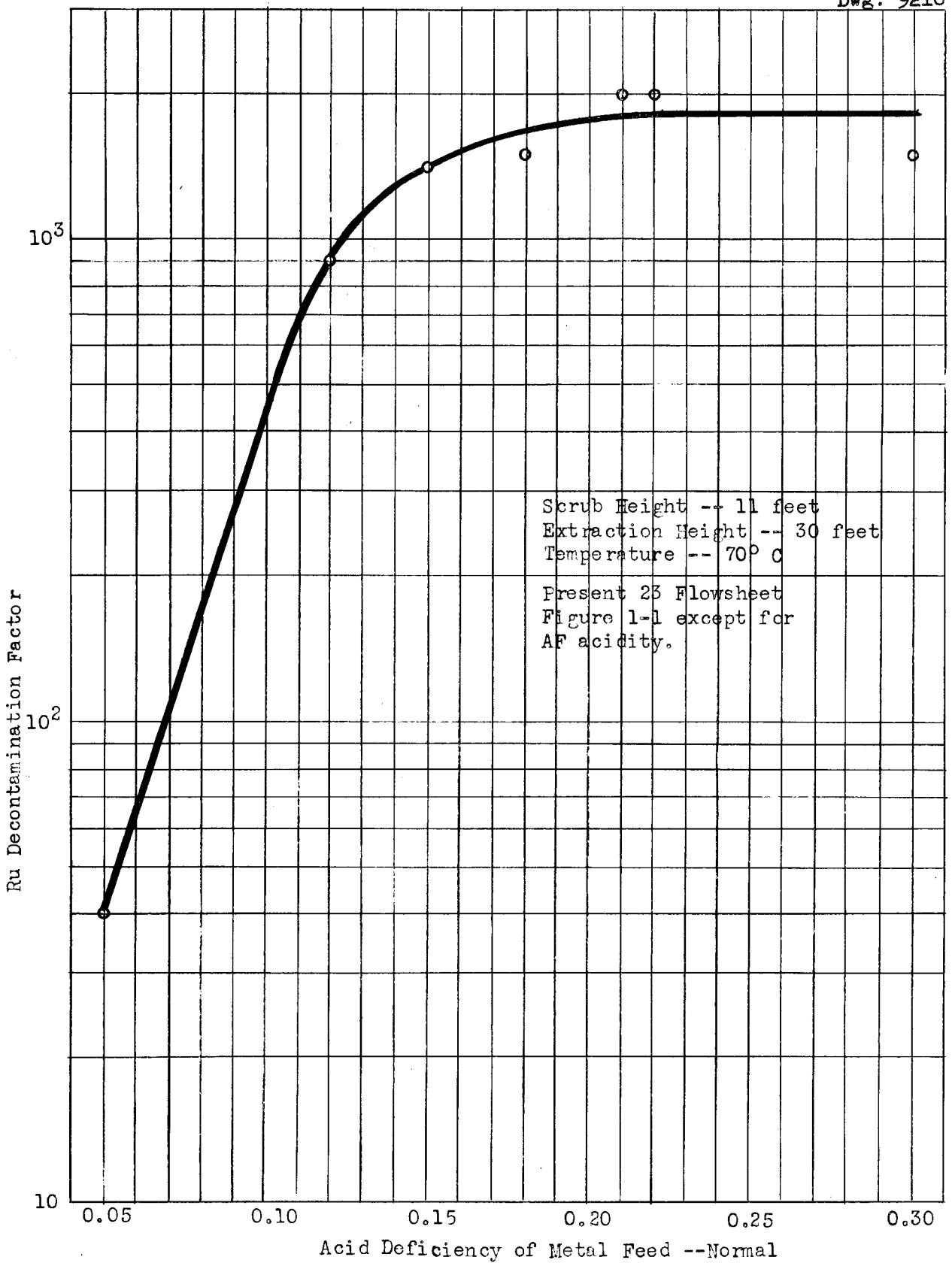
Run No.	Activity in Product as % of the 23 Background						Thorium mg/mg of 23
	Gross Fission Product Beta	Gross Fission Product Gamma	Ru Beta	Ce Beta	Zr Beta	Pa Beta	
4T	70	200	30	10	< 10	15	0.03
10T	70	80	40	< 10	< 10	< 10	0.01
11T	100	80	50	< 10	< 10	< 10	0.01
12T	30	70	30	< 10	< 10	< 10	0.01
13T	30	70	20	< 10	< 10	< 10	0.01
15T	70	80	60	< 10	< 10	< 10	0.01
16T	100	80	60	< 10	< 10	< 10	0.02
17T	70	80	70	20	< 10	< 10	0.02
18T	100	80	60	10	< 10	< 10	0.05

(1) The background from 23 was found to be about 3×10^3 β c/m/mg of 23 and 0.6 gamma mv/mg of 23.

[redacted]

[redacted]

FIGURE 1
Dwg. 9216



RUTHENIUM DECONTAMINATION AS A
FUNCTION OF FEED ACIDITY

[redacted]

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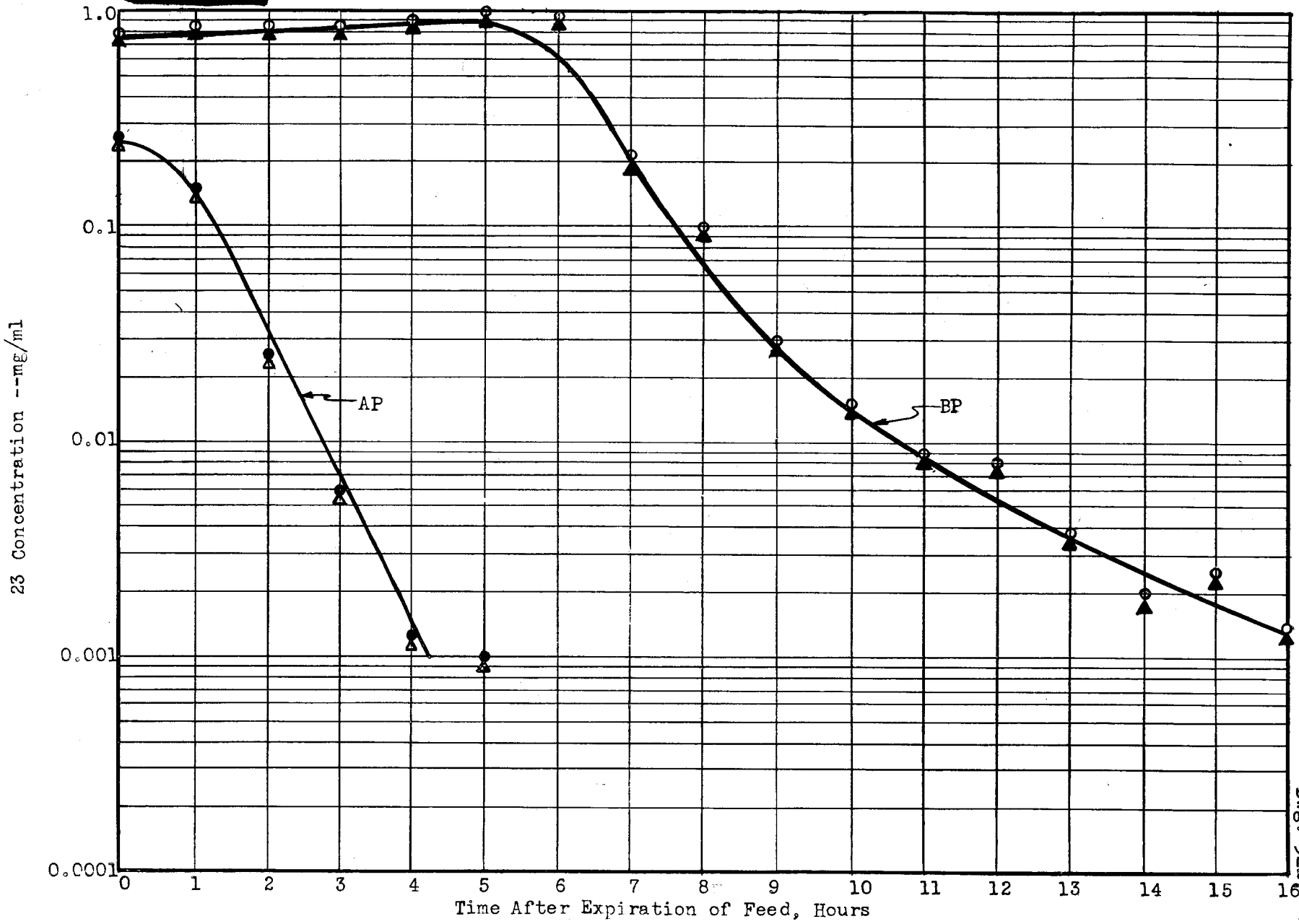


Figure 2
Dwg. 9217

CHANGE IN 23 CONCENTRATION IN HEXONE & STRIP DURING SHUTDOWN OF RUN

[redacted]

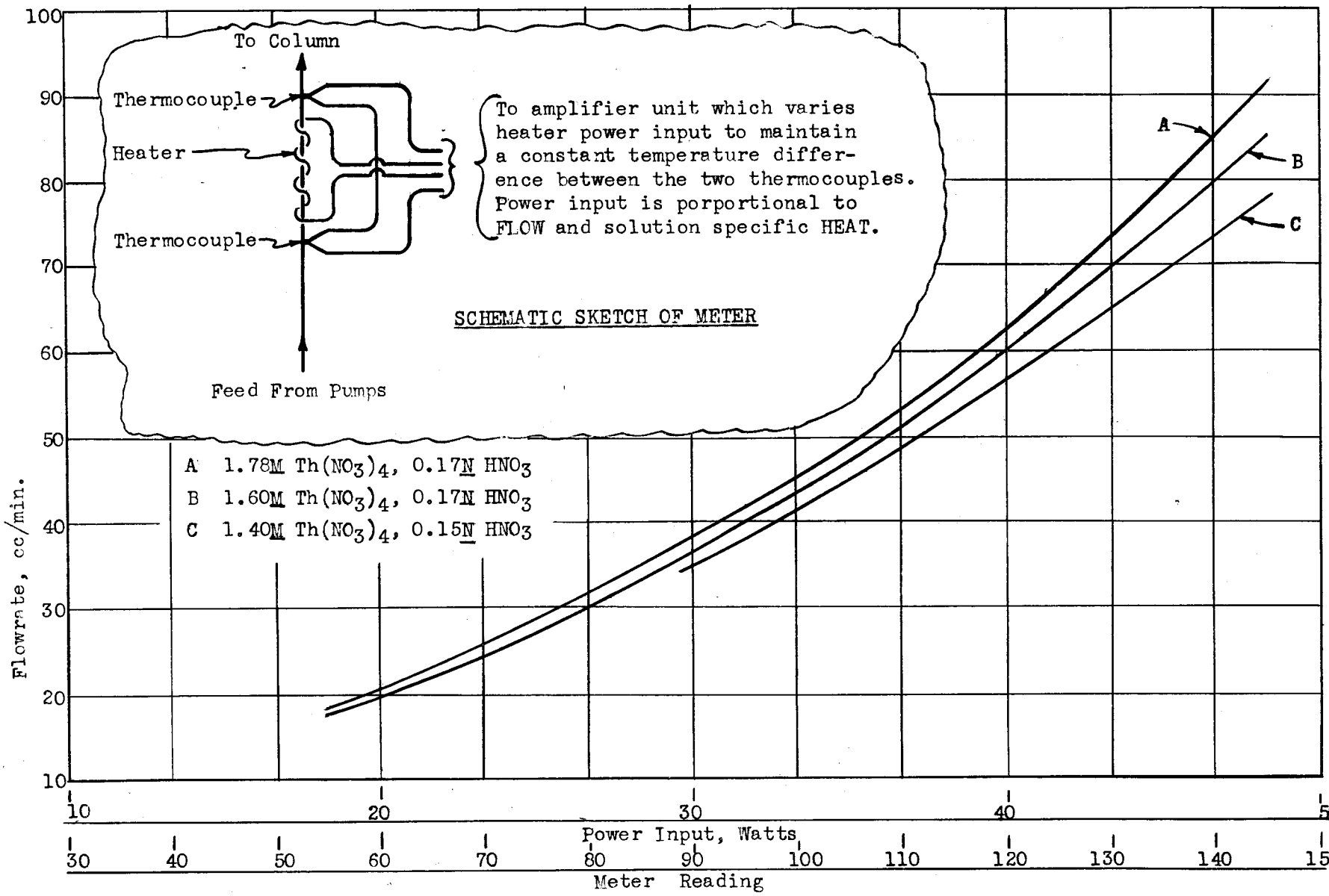


FIGURE 3
Dwg. 9218

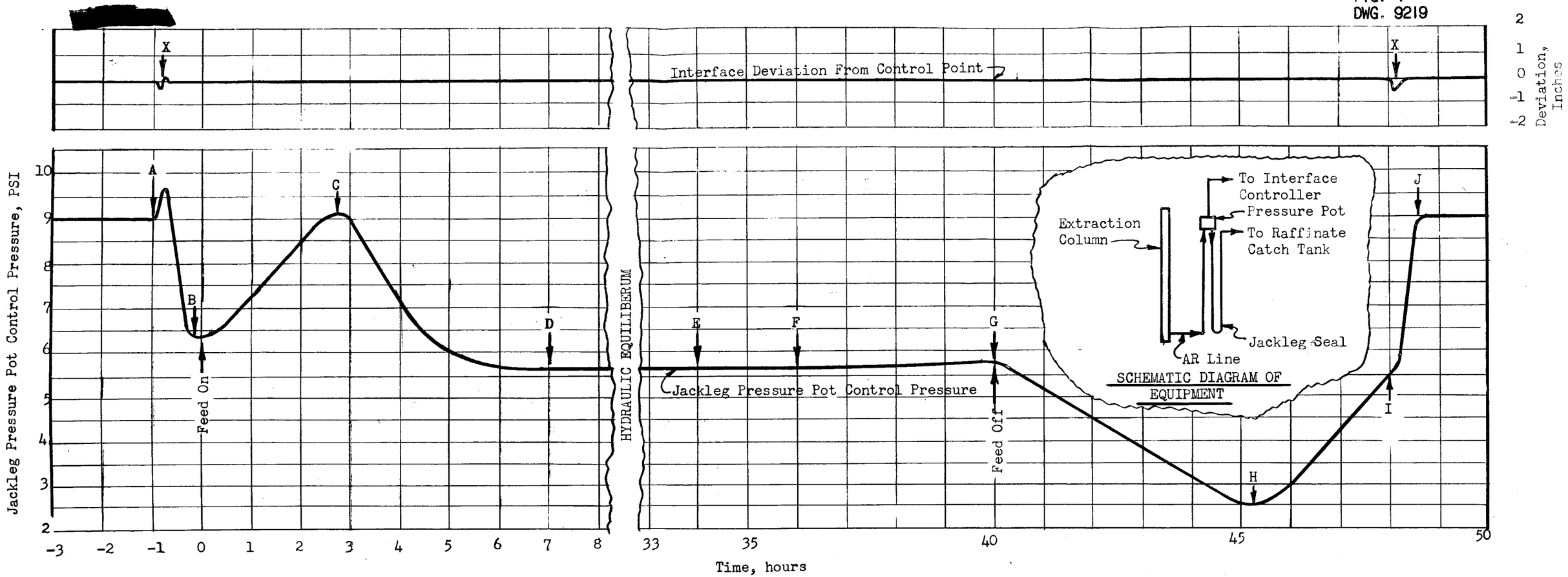
23 PILOT PLANT

THERMOELECTRIC FLOWMETER CALIBRATION CURVE

#1 Detector Unit, Low Scale, 25 Volts Input

[redacted]

FIG. 4
DWG. 9219



EXTRACTION COLUMN INTERFACE CONTROL STUDY

A. Solvent and scrub pump started. The pressure peak immediately after A is caused by a slight backflow of aqueous into the solvent accumulator when valves are set just prior to starting the solvent pump. Control pressure drops as jackleg fills with aqueous.

B. Hydraulic equilibrium. Feed is started and enters column a few minutes later.

B-C. Dense mixed feed and scrub displaces less dense scrub from column.

C. Mixed feed and scrub reaches bottom of column and starts out raffinate line to jackleg pressure pot.

C-D. Scrub is displaced from raffinate line to jackleg pressure pot by dense mixed feed and scrub.

D. Hydraulic equilibrium.

E. Valves are set to pump "shutdown" scrub to column.

F. Shutdown scrub enters column. Increase of pot pressure due to increased density of scrub.

G. Feed pump off, shutdown scrub and solvent rates are increased to clear 23 from the extraction column.

G-H. Control pressure decreases as shutdown scrub displaces mixed feed and scrub from column.

H. Mixed feed and scrub is cleared from column.

H-I. Mixed feed and scrub is displaced from line to jackleg pressure pot by less dense scrub.

I. Solvent and scrub flows are stopped.

I-J. Control pressure rises rapidly as aqueous is forced from the jackleg pressure pot line to replace the solvent that has been flowing up the column. If raffinate line volume isn't sufficiently large, as compared to the volume of flowing solvent in the column, there is danger of blowing the column or the jackleg seal; this is especially important at higher flowrates when the solvent holdup is greater. The pressure increase from I to J may be decreased to a final pressure slightly above that observed at B by gradually stopping the solvent pump while the scrub pump continues to operate, thereby effectively replacing flowing solvent with scrub.

X. Slight deviations from interface set point are attributed to the low sensitivity and reset rates, 15 and 0.5 respectively. These low response values were selected to minimize the possibility of blowing the column or jackleg seal.

[redacted]

FIG. 5
DWG. 8982
NOT CLASSIFIED

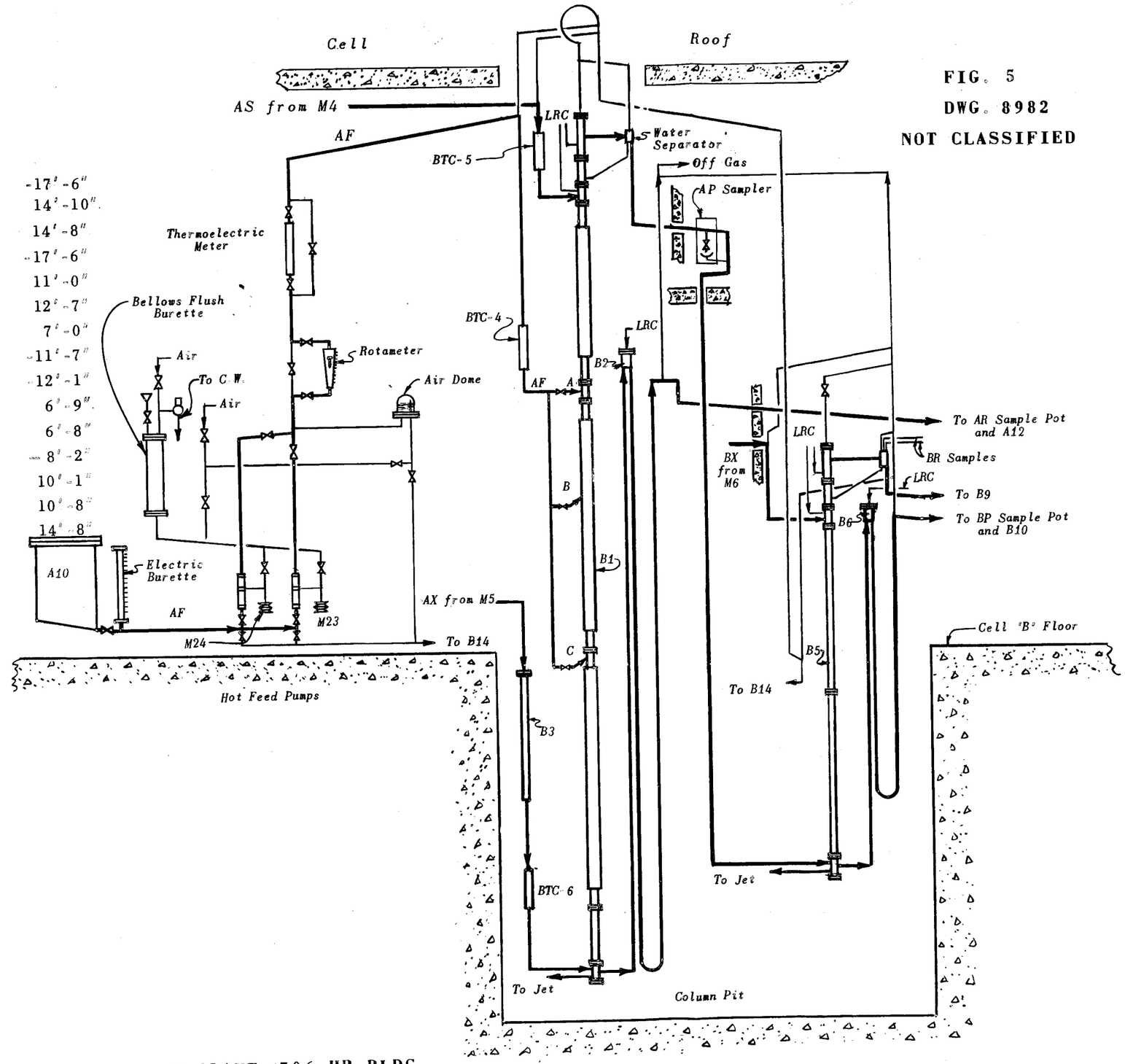
ELEVATIONS

Reference: 'B' Cell Floor = 0'-0"

AF High Point	28'-2"	AR out of BI	-17'-6"
AF Vent	33'-10"	B2 Bottom	14'-10"
Feed Point 'A'	13'-9"	B2 Jackleg High Point	14'-8"
Feed Point 'B'	9'-0"	B2 Jackleg Low Point	-17'-6"
Feed Point 'C'	-6"	BX High Point	11'-0"
AS High Point	29'-1"	BX Vent	12'-7"
AS Vent	33'-6"	BX Feed Point	7'-0"
AS Feed Point	24'-6"	AP into B5	-11'-7"
AX Feed Point	-17'-0"	BP out of B5	-12'-1"
B3 Top	-1'-0"	B6 Bottom	6'-9"
AP out of BI	27'-6"	B6 Jackleg High Point	6'-8"
AP Vent	31'-6"	B6 Jackleg Low Point	-8'-2"
BI Top	28'-6"	BR out of B5	10'-1"
BI Vent	34'-10"	Top of B5	10'-8"
		B5 Vent	14'-8"

SYMBOLS

LRC	Interface Recorder Controller
▬▬	Main Flows
▭	Vents & Secondary Lines
⧗	Bellows Pump
AF	Extraction Feed
AS	Extraction Scrub
AX	Extractant
AP	Extractant Product
AR	Extractant Raffinate
BX	Strip Extractant
BR	Strip Raffinate
BP	Strip Product



23 PILOT PLANT-706 HB BLDG

SCHEMATIC FEED, COLUMN AND VENTILATION DETAILS