[redacted]

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES
3 4456 0352690 4

ORNL 885
Progress Report
Chemistry-Separation
Processes for Plutonium
and Uranium

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION

# CHEMICAL TECHNOLOGY DIVISION
# PILOT PLANTS SECTION REPORT
# FOR SEPTEMBER-OCTOBER, 1950

LABORATORY RECORDS

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION

LIBRARY LOAN COPY

DO NOT TRANSFER TO ANOTHER PERSON

If you wish someone else to see this document, send in name with document and the library will arrange a loan.

OAK RIDGE NATIONAL LABORATORY
OPERATED BY
CARBIDE AND CARBON CHEMICALS DIVISION
UNION CARBIDE AND CARBON CORPORATION
UCC
POST OFFICE BOX P
OAK RIDGE, TENNESSEE

[redacted]

[redacted]

ORNL-885
Chemistry Separation Processes for Plutonium and Uranium

Contract No. W-7405-eng-26

This document consists of 22 pages. Copy 10 of 136. Series A.

CHEMICAL TECHNOLOGY DIVISION

# PILOT PLANTS SECTION REPORT FOR SEPTEMBER-OCTOBER, 1950

D. G. Reid

DECLASSIFIED
CLASSIFICATION CHANGED TO:
BY AUTHORITY OF: TID-1151
BY: R. Morrison 7/10/57

DATE ISSUED
NOV 28 1950

OAK RIDGE NATIONAL LABORATORY
operated by
CARBIDE AND CARBON CHEMICALS DIVISION
Union Carbide and Carbon Corporation
Post Office Box P
Oak Ridge, Tennessee

[redacted]



[redacted]

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES
3 4456 0352690 4

TABLE OF CONTENTS

| | Page Number |
|---|---|
| 0.0 Abstract | 4 |
| 1.0 Purex Process | 5 |
| 2.0 Radiochemical Waste Processing | 13 |
| 3.0 ORNL Metal Recovery Unit | 16 |
| 4.0 Chalk River Separation | 18 |
| 5.0 Miscellaneous | 19 |
| Appendix I - Specifications for ORNL Bldg. 205 Pilot Plant First Cycle Pulse Columns | 20 |

## 0.0 ABSTRACT

Report for the Pilot Plants Section of the ORNL Chemical Technology Division for the period of September and October, 1950 is presented. The information contained in this report includes:

1. Decontamination and conversion progress in the 205 and 706-HB Pilot Plants and miscellaneous equipment development in preparation for engaging in the Purex process developmental work about December, 1950.
2. Development on the ORNL Chemical Waste Evaporator including vapor scrubber and results of previous improvements.
3. Progress in construction of the ORNL Metal Recovery Unit.

# PUREX PROCESS

Technical - Jackson, Harrington, Hylton, Landry, Lewis, Lindauer, Sadowski, Ullmann, Winget

Non-Technical - Benson, Davis, Grizzell, Groover, Jennings, Ledbetter, Lockmiller, McLellan, Purkey, Sexton, Shields, Shipwash, Spangler, Strader, Summers, Thompson, Tipton, Wiggins

## 1.0 PUREX PROCESS

### 1.1 Summary

The ORNL Pilot Plants in Buildings 205 and 706-HB will be used for Purex process development beginning in December, 1950. Equipment changes in Bldg. 706-HB have been completed, and the conversion in Bldg. 205 is 80% complete. The decontamination of first cycle equipment, removed from the cells to permit installation of stainless steel cell liners, is essentially complete. Pulse columns are being installed in parallel to permit pilot plant development of the process with both pulse and packed columns. Cold runs will start in December, and the basic features of the chemical and equipment flowsheets will be demonstrated during the period January to June, 1951.

### 1.2 Decontamination - G. S. Sadowski

The necessary revisions of the first cycle feed makeup equipment for the conversion of the Pilot Plant equipment to the Purex process were minor. It was, however, decided to line Cells I and IA with stainless steel, install spray nozzles inside and around the cell tanks and revise the piping layout to permit easier and quicker decontamination of the cell equipment for future equipment revisions. To accomplish this job, it was first necessary to remove all the Cell I

tanks, the IA extraction column, all iron supports, and replace the contaminated wall and floor areas with new concrete.

All first cycle feed makeup tanks except the filter tank have been removed from Cell I and are being decontaminated. All structural iron supports and a three inch layer of the concrete floor have also been removed. In general, the walls and floor varied from 300 mr/hr to 5 R/hr, while several small areas ranged from 5-40 R/hr. due to leaks that developed in the equipment over a three year operating period. Unsuccessful attempts were made to lower the general cell background by sandblasting, buffing with a power driven steel brush, and flaking with an oxygen-acetylene torch. Small areas of 5 to 40 R levels were removed with a jackhammer, while larger areas ranging from 1 to 7 R were covered with lead sheet prior to installing the stainless steel liner. It is estimated that the general working time, after the installation of the lead sheet and stainless steel liner, will increase from approximately 18 minutes to 3 hours.

The decontamination of the dissolver and the five other feed makeup tanks was begun on June 30 and ended on July 20. Although the tanks were only partially decontaminated, it was decided to remove them from the cell and complete this operation elsewhere so that the cell alterations could begin. By September 11, these tanks had been removed from Cell I to the Burial Ground where temporary facilities for decontamination were installed. These facilities consisted of a 2,000 gallon stainless steel tank serviced with solution addition and drain lines to provide a large bath tank for submerging the cell vessels, a set of rollers on which the tanks could be laid and rotated, and a concrete block shield. The general decontamination procedure was to lay the tank on the rollers and strip it

(remove all flanges, dip tubes and gaskets) with an 8 foot long socket wrench through a slot in the concrete shield. The vessel was then set in the bath tank and steamed for 8 to 12 hours in 15% nitric acid followed by 25% caustic and finally periodic acid. This procedure was effective in decreasing the bulk of the activity from greater than 10 R/hr. to 100-500 mr/hr on the inside of the tank and from 2-10 R/hr to 50-200 mr/hr on the outside of the tank. Provisions were also made to permit the use of a buffing wheel with an 8 foot shaft for removal of localized contamination. The tanks were then removed to the Decontamination Building, 903, for final cleansing. One tank, the waste neutralizer, A-8, remains to be stripped and decontaminated.

1.3 Conversion Progress and Status of Building 205 Pilot Plant - F.E. Harrington

A supplement to increase the Purex Construction Recommendation from $156,500 to $228,000 has been issued. The original CR did not provide for the following items of the expanded program:

1. Stainless steel liners for the cells.
2. Pulse columns and accessories.
3. New samplers for first cycle tanks and process lines.

Conversion of the pilot plant is 80 per cent complete. Changes in Cells II and III are essentially complete, and equipment testing and calibration are in progress. Cells IA, II, and III equipment installation and testing will be complete and ready for cold runs by December 1. Acid recovery equipment is scheduled for completion by December 15, and the first cycle feed makeup equipment by January 1, 1951.

## 1.4 Equipment Development

### 1.41 Pulse Columns - R. B. Lindauer

Pulse columns are being installed in parallel with packed columns in the first cycle of the pilot plant equipment to permit development and demonstration of the Purex Process with both types of columns. The use of pulse columns in a production plant might significantly reduce the building cost, at the expense of simplicity of equipment and operation. Sufficient data will be obtained in the pilot plant to permit the selection of the contactor. Detailed specifications for the pulse columns were furnished by the Separations Technology Division, Hanford Works, and are given in Appendix I. The 2" IA column, 2.5" IB column, and 4" IC column will increase the pilot plant processing rate to 150 kg. of uranium per day. The columns will be pulsed by Milton Roy pumps.

### 1.42 Samplers - J. W. Ullmann

#### 1.421 New First Cycle Samplers

Redesign of the air jet, recirculating-type samplers to provide greater shielding was discussed in the last progress report (ORNL-850). The construction of one unit is complete and testing has been started.

#### 1.422 Sampler for Evaporated IAW Stream

In the Purex process the volume of the IAW stream will be reduced by a factor of twenty in order to reduce waste volumes and to recover nitric acid. The high specific activity of the residual evaporator solution necessitates a mode of sampling different from that employed for sampling other process vessels. The sampler designed for the RaLa Process, suitably modified for the

present installation has been adopted because:

(1) The operation of the sampling cycle is completely remote.

(2) A small, fixed volume of sample is collected.

This main feature of the sampler is a four-port plug valve with a hole in the plug. In the "recirculating" position, solution is drawn through the valve plug and returned to the evaporator by means of an air jet. A small sample (0.1 ml) of the recirculating stream is obtained by turning the plug to the "drain" position. The solution trapped in the hole in the plug is drained into the sample receiver.

The sampler is to be mounted in Cell IV adjacent to the nitric acid still and controlled from the pipe tunnel. In operation, the sample carrier is set in front of a plug through the cell wall, a plug slide and carrier slide are opened, the sample receiver is pushed into the cell underneath the sampling valve by means of an extension rod, and a sample is taken.

The sampler receiver is removed by pulling it back into the sample carrier, after which the plug and carrier slides are closed. The sampling lines will be fitted to the valve by a coupling which can be broken from the pipe tunnel, after which the entire sampler can be slid through the wall for any necessary decontamination or repair. This sampler will be fabricated during November.

1.43 Internal Spray System for Decontaminating Tanks - C. D. Hylton

Cell I tanks have been decontaminated previously by heating and agitating solutions within the tanks. This method does not effectively remove contamination which has adhered to the upper sections of the tanks, particularly

the tank cover and flanged nozzles. In order to increase the effectiveness of decontamination procedures, a Spraco Full Cone nozzle will be installed permanently in all of the Cell I tanks. The nozzle, pointed upwards, will spray the top and a portion of the sides of the tank. Decontaminating solutions will be pumped directly to individual nozzles from a steam jacketed head tank located on the cell roof. The spray solution will then be heated and agitated in the tank, after which it will be jetted to waste and the cycle repeated.

1.44 Decontamination Tools - C. D. Hylton

The first cycle feed makeup tanks to be returned to Cell I will be modified to permit essentially complete decontamination of the tanks within the cell. However, there will probably be a continued requirement in the pilot plant for tools to affect simple mechanical operations from the top of the cell block, a distance of ten to thirty feet from the various tanks. Simple mechanisms are now being designed to tighten or remove nuts and cut pipe. The cost of an instrument now being fabricated to remove and tighten nuts from the top of the cell block is expected to be about $500. The design of tools for other simple operations will be governed by the effectiveness of this instrument.

1.45 Stainless Steel Cell Liners - F. E. Harrington

During the past three years the concrete floors and walls of the 205 Bldg. cells have become porous and retain considerable radioactivity from repeated decontaminations. In order to facilitate future decontamination operations, a liner of 11 gauge stainless steel sheet is being installed on the floors and 16 gauge on the walls. In low level radiation areas, Cells II, III and IV, the stainless steel has been installed to a height of 6 feet on the walls. In the high

level radiation areas, Cells I and IA, the walls will be covered to a height of 18 feet. Type 347 stainless steel is being used, and each sheet is being arc welded to each adjacent sheet.

In Cells III and IV, the cost of installing the stainless steel was $6.40 per square foot of which $2.56 was charged for materials, $1.92 for labor and $1.92 for overhead. In these cells the work was experimental by nature, and one third of the work was done on an overtime basis. The sheets were anchored to the walls and floors by means of cinch anchors rather than by studs shot into the concrete by a "Drive-it" (trade name) power gun, as is being done in Cell I. In a new installation where the stainless sheets could be welded directly to angle or zee bars employed as anchors in the concrete, it is estimated the stainless could be installed for $5.00 per square foot including labor, material and overhead.

The combination of Type 304 e.l.c. stainless steel and heliarc welding is attractive because of the critical supply of columbium stainless steel and the absence of air pockets and welding slag. The estimated cost of this combination is $6.50 per square foot. Although 304 e.l.c. stainless steel is 25 per cent cheaper than type 347, heliarc welding is more time consuming than arc welding. The absence of columbium, which when present may exchange with radio columbium, may make type 304 e.l.c. easier to decontaminate.

1.46 Welding of Tank Covers - C. D. Hylton

Seven of the eight major vessels in Cell I are now equipped with gasketed tank covers, and it is known that much of the activity on the floor of Cell I can be attributed to leaky gaskets on these tanks. Since it has been found

unnecessary to have access to the interior of these tanks, each tank except the dissolver will have the cover welded to the tank body.

1.5 Operation of Bldg. 706-HB Pilot Plant - W. H. Lewis

Three runs under Purex First Cycle Flowsheet conditions have been made in the 706-HB Pilot Plant columns, and additional runs are planned for November and December. The columns are being operated as a service to the Laboratory Section, due to lack of necessary column height in the laboratory. Results of the runs will be reported by the Laboratory Section.

1.6 Program

Cold runs in the 205 Pilot Plant will begin in December and hot runs in January, 1951. A five months experimental program to develop and demonstrate the major features of the process will then be conducted. Starting about July, 1951 the pilot plant equipment will be modified as necessary to obtain all data required for the detailed design and operation of equipment for a Purex production plant.

# RADIOCHEMICAL WASTE PROCESSING

Technical - Shank

## 2.0 RADIOCHEMICAL WASTE PROCESSING

### 2.1 Evaporator Operating Summary

The Operations Division processed in the ORNL Radiochemical Waste Evaporator approximately 390,000 gallons of mixed wastes during September and October with an average overall beta decontamination factor of $6 \times 10^3$ and an average condensate discharge activity of $3 \times 10^{-10}$ curies/ml. Weekly decontamination factors of $1.5 \times 10^5$ were obtained while processing high level activity sludge wastes (approximately $1 \times 10^7$ beta cts/min/ml) from the feed surge tank, W-5.

### 2.2 Evaporator Vapor Scrubber

Substitution of the previously proposed 5 plate bubble cap vapor scrubber (ORNL-850) is to be made with a three stage Peabody Impingement Baffle Plate Scrubber. The request to purchase has been sent to the vendor with delivery expected in 6 to 8 weeks. The advantages of the Peabody Scrubber are lower initial cost, smaller size (2-1/2 feet diameter by 12 feet instead of 5 feet diameter by 15 feet), and anticipated greater vapor scrubbing efficiencies. The general fabrication requirements have not been altered.

### 2.3 Evaporator Study Results

#### 2.31 Condensate Conductivity Cell

The condensate conductivity cell installed for indication of foam-overs and high entrainment (ORNL-702) has continued to give satisfactory service. Increased experience of the Operating Division's personnel with this instrument is

permitting greater ease in control of foam-overs by means of controlled anti-foam agent additions and evaporation rate adjustments.

2.32 Analytical Development

A previous study (ORNL-655) of the effect of pH and free $OH^-$ concentration in various types feeds on decontamination factors indicated the following:

(1) pH is not a true indication of the free $OH^-$ concentration.

(2) Decontamination factors of Ru and Cs in metal waste supernate feeds were greater where the free $OH^-$ concentration was shown to be greater than 2.0 N whereas the pH determination did not indicate a change in free $OH^-$ concentration in these feeds.

As a result of the above, an analytical procedure was developed for feed determination of the free $OH^-$ concentration which could enable operations to adjust feeds prior to entering the evaporator. This procedure has not been incorporated into the evaporator procedure because:

(1) The installation of the condensate conductivity cell has permitted a $10^3$ decontamination factor to be obtained consistently.

(2) The installation of a vapor scrubber should further enhance the decontamination factor of the evaporator.

(3) An operating procedure requiring no feed adjustment is desired if sufficient decontamination can be obtained by other means.

## 2.4 Program

The following program for the evaporator is scheduled for completion by March 1951.

(1) Installation of an eight-point electronic probe in the evaporator for foam level indication, automatic steam control and anti-foam injections, and investigations of feed characteristics and anti-foam agents.

(2) Cost and efficiency evaluation of the evaporator.

(3) Installation and evaluation of the vapor scrubber.

## ORNL METAL RECOVERY UNIT

Technical - Stewart, J. P. Jackson

### 3.0 ORNL METAL RECOVERY UNIT

#### 3.1 Summary

The construction of the ORNL Waste Metal Recovery Building has progressed rapidly and is expected to be completed November 10, 1950. Process and design alterations have permitted the installation to be completely continuous rather than a semi-continuous operation. Purchase orders or inquiries have been made covering all of the process equipment. The responsibility for the project was transferred October 1, 1950 from the Design Section to the Pilot Plants Section.

#### 3.2 Progress

The progress of the overall project is summarized in Table I, which shows the percentage complete of various phases.

Table I

| | |
|---|---|
| Engineering | 85% |
| Process Equipment Folio | 60% |
| Building - (Contractor) | 85% |
| Equipment Purchased and Delivered | 5% |
| Equipment Ordered | 75% |
| Overall Project | 60% |

3.3 Process and Design Changes

The fabrication of a traverse mechanism to remove metal waste from the Tank Farm has been postponed until a further study can be made of an alternate method for this operation.

The use of 0.005 M hydrofluoric acid for the "B" column extractant scrub permits the elimination of the cold makeup area. Flowsheet Number 3 provided for mix and feed tanks for the extraction and partitioning column scrub streams. The respective streams were pumped by a Milton Roy duplex pump and required a total of three tanks for the mixing and storing of the scrub streams. Flowsheet No. 4 permits all streams entering the columns (except the feed) to be measured by a rotameter. The extraction column scrub stream is a blend of water and acid streams, and the scrub to the partitioning column is water into which hydrofluoric acid is injected on the "down-side" of the rotameter.

3.4 Program

The program for the coming period is as follows:

1. Completion of the building by the contractor.
2. Completion of engineering drawings for the equipment.
3. Initiation of the construction report.
4. Begin miscellaneous work by ORNL forces to rehabilitate old equipment for reuse and fabricate auxiliary pieces.
5. Continuation of work covering cost studies and the operating manual.

## CHALK RIVER SEPARATION

Technical - Rigstad

### 4.0 CHALK RIVER SEPARATION

The final rough draft of the terminal report covering the Pilot Plant processing of the Chalk River and Hanford fuel rods has been completed. This report, ORNL-880, is scheduled to be issued in November.

The final processing of the plutonium by the Laboratory Section of the Chemical Technology Division has been slow because of hexone decomposition products formed during pilot plant processing. The first Pilot Plant run (LAMS-995, Batch 6) was completed, as well as a 40 gm. portion of the most highly irradiated Chalk River material (LAMS-995, Batch 1). It is expected that this material plus another 60 gram portion of Batch 1 will be transferred to the customer early in the next period.

## 5.0 MISCELLANEOUS

Permanentization of the 205 Bldg., as far as the J. A. Jones Construction Company is concerned, is essentially complete. Occupancy of the building started the middle of October and acceptance of the building from the contractor will be made early next period. A number of miscellaneous items will be completed by ORNL maintenance forces.

F. M. Grizzell, Research Operator, was transferred to the Reactor Technology Division effective 10/16/50.

C. H. Tipton, Research Operator Helper, entered the Pilot Plants Section from the Security Division effective 10/16/50.

E. M. Shank has been assigned part-time to the Laboratory Section to assist in developing a RaLa process using Materials Testing Reactor fuel rods as feed material.

[redacted]

APPENDIX I

Specifications for ORNL 205 Pilot Plant
First Cycle Pulse Columns

| Sections | Length (Feet) | Diam. (Ins.) | Plates[d] No. | Spacing (Ins.) | Thickness (Ins.) | Hole Diam. (Ins.) | % Open Area |
|---|---|---|---|---|---|---|---|
| 1A | | | | | | | |
| Top[a] | 3 | 4 | - | - | - | - | - |
| Scrub Section[b] | 8 | 2 | 47 | 2 | 0.05 | 0.125 | 23 |
| Mid-Feed Point | 0.5 | 2 | - | - | - | - | - |
| Extr. Section | 8 | 2 | 47 | 2 | 0.05 | 0.125 | 23 |
| Bottom Sect.[c] | 1.5 | 2 | - | - | - | - | - |
| Over-all | 21.0 | | 94 | | | | |
| 1B | | | | | | | |
| Top[a] | 3 | 5 | - | - | - | - | - |
| Extr. Section[b] | 8 | 2.5 | 47 | 2 | 0.05 | 0.125 | 23 |
| Mid-Feed Point | 0.5 | 2.5 | - | - | - | - | - |
| Scrub Section | 8 | 2 | 47 | 2 | 0.05 | 0.125 | 23 |
| Bottom Sect.[c] | 1.5 | 2 | - | - | - | - | - |
| Over-all | 21.0 | | 94 | | | | |
| 1C | | | | | | | |
| Top[a] | 3 | 8 | - | - | - | - | - |
| Strip Section | 12 | 4 | 71 | 2 | 0.05 | 0.125 | 23 |
| Bottom Sect.[c] | 1.5 | 4 | - | - | - | - | - |
| Over-all | 16.5 | | 71 | | | | |

[redacted]

[redacted]

## Notes to Table

(a) Top sections of approximately 4 times column cross section and of the specified length are provided to permit phase disengagement and reasonable sensitivity to interface level variations in the top sections.

(b) These sections are spool pieces and are unfilled except for the single feed line entering from the side and pointing up.

(c) The bottom section is part of the section above it but is not filled with packing.

(d) The number of plates is arbitrarily determined by assuming plates are 0.05 inch thick. Thus, for an 8 foot section 47 plates x 0.05 in./plate + 46 spaces x 2 in./space = 94.4 inches.

## General Notes

Support Rods - Single, 1/4 in. diameter, stainless steel rods of proper length to allow for plates, spacers, rod support and locknuts.

Spacers - One-eight inch IPS stainless steel pipe.

Rod Support - Welded to the top flange.

Middle Feed - A single pipe introduces the 1AF or 1BF upward along the column axis at approximately the center of the "unpacked" middle feed section.

Bottom Organic Feed - A single pipe discharges up approximately 6 inches below the bottom plate.

Top Aqueous Feed - A single pipe discharges down approximately 4 inches above the top plate.

Interface Location - 6 to 10 inches above the top plate.

### Pulse Conditions

| Column | Amplitude (inches) | Frequency cycles/min) |
|---|---|---|
| 1A | 1 | 58 |
| 1B (Scrub) | 1 | 58 |
| (Extr.) | 0.6 | 58 |
| 1C | 0.5 | 74 |



[redacted]

[redacted]

Overflow Location - 12 inches below column top.

Instrumentation - Interface control by air on a jack leg pressure pot is used with a decreased sensitivity setting on the controller.



[redacted]