MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES
3 4456 0060437 1

[redacted]

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION

ORNL-1385

PROGRESS

Classification Cancelled
Or Changed To
By Authority of UCC 9/13/71
By C. Goldberg Date 11/10/71

AEC RESEARCH AND DEVELOPMENT REPORT

LABORATORY LIBRARY

CHEMICAL TECHNOLOGY DIVISION

QUARTERLY PROGRESS REPORT

FOR PERIOD ENDING AUGUST 20, 1952

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.

UNCLASSIFIED
This document contains restricted data as defined in the Atomic Energy Act of 1946. Its transmittal or the disclosure of its contents in any manner to an unauthorized person is prohibited.

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

[redacted]

[redacted]

ORNL-1385

This document consists of 28 pages.
Copy No. 5 of 142 copies. Series A.

Contract No. W-7405-eng-26

CHEMICAL TECHNOLOGY DIVISION

QUARTERLY PROGRESS REPORT

for Period Ending August 20, 1952

F. L. Steahly - Division Director
F. R. Bruce - Laboratory Chief
J. O. Davis - Unit Operations Chief
H. K. Jackson - Pilot Plant Chief
F. L. Culler - Process Design Chief

DATE ISSUED

OCT 22 1952

OAK RIDGE NATIONAL LABORATORY
Operated by
CARBIDE AND CARBON CHEMICALS COMPANY
A Division of Union Carbide and Carbon Corporation
Post Office Box P
Oak Ridge, Tennessee

[redacted]

[redacted]

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES
3 4456 0060437 1

[redacted]

ORNL-1385
Progress

INTERNAL DISTRIBUTION

1. G. T. Felbeck
2. Biology Library
3. Health Physics Library
4. Metallurgy Library
5-6. Training School Library
7. Reactor Experimental Engineering Library
8-17. Central Files
18. C. E. Center
19. C. E. Larson
20. W. B. Humes (K-25)
21. L. B. Emlet (Y-12)
22. A. M. Weinberg
23. E. H. Taylor
24. E. J. Murphy
25. E. D. Shipley
26. F. C. VonderLage
27-28. F. L. Steahly
29. R. C. Briant
30. J. A. Swartout
31. S. C. Lind
32. A. H. Snell
33. A. Hollaender
34. M. T. Kelley
35. G. H. Clewett
36. K. Z. Morgan
37. J. S. Felton
38. A. S. Householder
39. C. S. Harrill
40. C. E. Winters
41. D. W. Cardwell
42. E. M. King
43. W. K. Eister
44. F. R. Bruce
45. F. L. Culler
46. J. O. Davis
47. H. K. Jackson
48. R. S. Livingston
49. C. P. Keim
50. C. D. Susano
51. F. W. Hurd (K-25)
52. S. Cromer (K-25)
53. R. E. Engberg
54. R. N. Lyon
55. C. B. Graham
56. R. B. Briggs
57. D. D. Cowen
58. P. M. Reyling
59. E. P. Wigner (consultant)

EXTERNAL DISTRIBUTION

60-66. Argonne National Laboratory
67. Armed Forces Special Weapons Project (Sandia)
68. Armed Forces Special Weapons Project (Washington)
69-75. Atomic Energy Commission, Washington
76-78. Brookhaven National Laboratory
79. Carbide and Carbon Chemicals Company (C-31 Plant)
80-81. Carbide and Carbon Chemicals Company (K-25 Plant)
82-83. Carbide and Carbon Chemicals Company (Y-12 Area)

[redacted]

[redacted]

[redacted]

84. Chicago Patent Group
85-90. duPont Company
91-96. General Electric Company, Richland
(1 copy to G. F. Hill and 1 copy to F. W. Albaugh)
97. Hanford Operations Office
98-102. Idaho Operations Office (1 copy to Phillips Petroleum Co.)
103. Iowa State College
104-107. Knolls Atomic Power Laboratory
108-110. Los Alamos
111-113. Mound Laboratory
114. Naval Research Laboratory
115-118. New York Operations Office
119. Patent Branch, Washington
120. Savannah River Operations Office (Augusta)
121. Savannah River Operations Office (Wilmington)
122-125. University of California Radiation Laboratory
126-127. Vitro Corporation of America
128-142. Technical Information Service, Oak Ridge

[redacted]

[redacted]

Reports previously issued in this series are as follows:

ORNL-268	Period Ending May 31, 1949
ORNL-467	Period Ending August 31, 1949
ORNL-530	Period Ending November 30, 1949
ORNL-663	Period Ending February 28, 1950
ORNL-763	Period Ending May 31, 1950
ORNL-846	Period Ending August 31, 1950
ORNL-936	Period Ending November 20, 1950
ORNL-1000	Period Ending February 20, 1951
ORNL-1061	Period Ending May 20, 1951
ORNL-1141	Period Ending August 20, 1951
ORNL-1311	Period Ending February 10, 1952
ORNL-1328	Period Ending May 20, 1952

[redacted]

[redacted]

[redacted]

TABLE OF CONTENTS

	PAGE
ABSTRACT	1
INTRODUCTION	1
SUMMARY	2
1. PUREX PROCESS	2
Purex Process Demonstration	2
Laboratory Studies of Purex Conditions	3
Plutonium Recovery from Slag and Crucible Waste	3
2. SCRUP PROGRAM	4
3. HOMOGENEOUS REACTOR FUEL REPROCESSING	4
4. THOREX PROCESS	5
5. IDAHO CHEMICAL PROCESSING PLANT	6
6. ISOLATION PROCESSES FOR PLUTONIUM AND URANIUM	7
7. HOMOGENEOUS REACTOR SLURRIES	7
8. VOLATILITY	9
9. ELECTROCHEMISTRY	9
10. ION EXCHANGE	10
11. CHEMICAL ENGINEERING RESEARCH	10
Solvent-Extraction Studies	11
Equipment and Materials Evaluation	11
Cost Studies	12
Metal-Dissolution Studies	13
Equipment-Decontamination Studies	13
12. LIST OF REPORTS ISSUED	14

[redacted]

CHEMICAL TECHNOLOGY DIVISION

F. L. Steahly, Director
June Parrott, Secretary

W. K. Eister, Special Assignment
Martha Gerrard, Reports Editor
Bettye Burns, Typist

C. W. Schersten, Assistant to Director
Virginia Wells, Secretary
J. W. Clark, Procurement
Augusta Dobbin, Janitress
Aola Johnson, Janitress

LABORATORY SECTION[1]

F. R. Bruce,[2] Section Chief
W. B. Lanham, Special Assignment
Mary Stobbe, Secretary
J. E. Farmer, Technician
C. F. Keck, Technician
Bettye Large, Typist
C. J. Shipman, Technician

PUREX

J. R. Flanary, Problem Leader

TECHNICIANS
S. V. Castner[3]
R. L. Folger[3]
L. E. Line[3]
V. J. Reilly[3]
V. A. Ryan[3]
E. B. Sheldon[3]
L. A. Byrd
E. R. Johns

HOMOGENEOUS REACTOR CHEMICAL PROCESSING

D. E. Ferguson, Problem Leader

TECHNICIANS
G. I. Cathers
C. V. Ellison
R. G. Mansfield
R. A. McNees
W. E. Tomlin
V. L. Fowler
W. B. Howerton
R. C. Shipwash
O. K. Tallent

HOMOGENEOUS REACTOR FUEL SLURRY STUDIES

F. R. Bruce,[2] Problem Leader

TECHNICIAN
J. O. Blomeke
J. M. Dalla Valle[4]
J. M. Fulmer
J. P. McBride
L. E. Morse
J. L. Banberg

THOREX

A. T. Gresky, Problem Leader

TECHNICIANS
M. R. Bennett
S. S. Brandt[3]
K. L. Mattern[5]
W. T. McDuffee, Jr.
J. E. Savolainen
R. P. Wischow
R. C. Lovelace
J. F. Talley
C. T. Thompson

VOLATILITY

R. E. Leuze, Problem Leader

TECHNICIAN
C. E. Schilling
H. B. Graham

SPECIAL PROBLEMS

R. E. Blanco, Problem Leader

TECHNICIANS
M. D. Barringer
G. A. Eaton
A. H. Kibbey
L. D. Lynch
R. F. Sessions[6]
D. M. Helton
J. F. Land

ELECTROCHEMISTRY

J. A. McLaren, Problem Leader
J. H. Goode
C. W. Keenan[6]
H. L. Peeler

ISOLATION

D. C. Overholt, Problem Leader

TECHNICIANS
D. G. Karraker[3]
D. A. Orth[3]
R. H. Rainey
F. W. Tober[3]
J. M. Delozier
C. W. Greene
D. R. Hendrix
R. B. Quincy

ION EXCHANGE STUDIES

I. R. Higgins, Problem Leader

TECHNICIAN
J. T. Roberts
W. E. Shockley

PROCESS DESIGN SECTION

F. L. Culler, Section Chief
Lucille Kuykendall, Secretary
May Pepper, Secretary

IDAHO CHEMICAL PLANT

H. E. Goeller, Problem Leader

STENOGRAPHIC STAFF
F. N. Browder (Idaho)
E. J. Frederick[7] (Idaho)
F. E. Harrington (Idaho)
R. J. Klotzbach (Idaho)
F. C. McCullough
E. L. Nicholson (Idaho)
P. L. Robertson[8] (Idaho)
A. M. Rom (Idaho)
J. B. Ruch (Idaho)
Julia Hoppe
Kay Dixon

INTERIM-23, THOREX

W. E. Unger, Problem Leader

TYPIST
G. B. Berry[8]
B. F. Bottenfield[8]
R. V. Foltz[8]
A. R. Irvine[8]
K. G. Steyer[5]
W. R. Winsbro
Helen Hamrick

SPECIAL PROBLEMS

R. P. Milford, Problem Leader
W. L. Carter,[8] Assistant Problem Leader
B. Lieberman[9]
H. H. Messenheimer[8]
L. W. Michael[8]
R. W. Naylor[10]
W. F. Schaffer
D. B. Stewart[10]
H. O. Weeren[8]

ECONOMIC STUDIES

W. G. Stockdale, Problem Leader
G. W. T. Kearsley[8]
H. E. Schweyer[6]

PUREX ECONOMIC STUDY
H. L. Hull,[3] Problem Leader
H. R. Zeitlin[8]

UNIT OPERATIONS SECTION

J. O. Davis, Section Chief
Ruth Pennington, Secretary
Marion Hoppe, Secretary

SOLVENT EXTRACTION

A. C. Jealous,[11] Problem Leader

TECHNICIANS
R. A. Fisher[6]
R. W. Horton
H. F. Johnson[4]
E. Lieberman
H. C. Osment[3]
G. Jones
J. C. Rose
H. F. Soard

SPECIAL EQUIPMENT

T. A. Arehart,[12] Problem Leader

TECHNICIANS
S. H. Jury[4]
J. R. Parrott
G. A. West
C. L. Williamson[3]
E. D. York
T. D. Napier
R. O. Payne

RADIATION ENGINEERING

C. D. Watson,[13] Problem Leader

TECHNICIANS
J. L. Alston[3]
M. L. Feldman[5]
R. J. McNamee
J. C. Suddath
G. R. Quinn
F. L. Rogers

FEED PREPARATION

E. O. Nurmi,[14] Problem Leader

TECHNICIANS
W. E. Covery[3]
E. C. Elener[3]
J. E. Ericson[3]
D. L. Foster
G. B. Dinsmore
D. B. Masters

DU PONT COMPANY REPRESENTATIVES

R. I. Martens, Liaison Office Manager
Ellen Tober, Secretary
Alice Duff, Clerk-Typist (ORNL)
A. E. Wible, Assistant to the Liaison Office Manager
A. L. Denny, Administrative Assistant

PILOT PLANT SECTION

H. K. Jackson, Section Chief
Agnes Hair, Secretary
Mary Lynn Cameron, Typist
R. B. Waters, Draftsman
E. Wright, Clerk
W. J. Greter,[9] Instrumentation
G. S. Sadowski, Special Assignment

PUREX

D. O. Darby, Problem Leader

ENGINEERING
R. B. Lindauer[2]
G. R. Tully[5]

ACID RECOVERY
J. W. Landry

DATA AND TRAINING
J. M. Chandler
J. E. Bigelow
B. W. Cetnar[3]
A. J. Pecquet
C. E. Pickett[3]

OPERATIONS
R. H. Winget

OPERATORS
W. D. Burch, Shift Supervisor
R. B. Keely, Shift Supervisor
T. H. Spencer,[3] Shift Supervisor
T. T. Thompson,[3] Shift Supervisor
R. H. Vaughan, Shift Supervisor
W. T. Bostic
J. H. Brock
E. W. Brown
J. H. Groover
J. E. Higgins
R. E. Purkey
W. A. Shannon
D. H. Summers
H. C. Thompson
J. R. Vest
J. H. Walker

SPECIAL PROBLEMS

R. R. Lindauer,[2] Problem Leader

SCRUP

W. H. Lewis, Problem Leader

OPERATORS
R. E. Brooksbank,[7] Shift Supervisor
W. D. Daniel[3]
J. L. Matherne, Shift Supervisor
P. H. Porter,[3] Data
J. W. Schauer[3]
D. J. Skovholt[3]
R. W. Yancey,[3] Shift Supervisor
C. W. Boatman
W. E. Ledbetter
J. F. Lockmiller
R. J. Shannon
D. E. Spangler
S. E. Spencer
G. R. Thompson
C. H. Tipton

INTERIM-23

C. D. Hylton, Problem Leader

OPERATORS
S. Mann,[15] Shift Supervisor
M. A. Turner, Shift Supervisor
H. P. Wood,[3] Shift Supervisor
W. J. Bryan
H. S. Caldwell
G. D. Davis
R. L. Hickey
W. Jennings
C. H. Jones
S. A. McCosh
C. E. Waddell

THOREX

E. M. Shank, Problem Leader
J. W. Ullmann

RAGS

W. B. Watkins,[3] Problem Leader
D. R. Lindow,[3] Data
T. S. McMillan[3]

---

(1) C. P. Johnston, on loan to Materials Chemistry Division
(2) Dual capacity
(3) Du Pont personnel
(4) Consultant
(5) California Research and Development Company personnel
(6) Research participant
(7) On loan from Analytical Chemistry Division
(8) On loan from Engineering Department
(9) On loan from Instrument Department
(10) Vitro Corporation personnel
(11) Responsible in this section for Purex and Thorex
(12) Responsible in this section for RaLa, metal recovery, and special problems.
(13) Responsible in this section for waste disposal and slurry studies.
(14) Responsible in this section for "25" Process and homogeneous fual reprocessing
(15) Atlantic Refining Company personnel

CHEMICAL TECHNOLOGY DIVISION QUARTERLY PROGRESS REPORT

ABSTRACT

Work done on the following problems is reported: Purex, Scrup, and Thorex processes; homogeneous reactor fuel preparation and product recovery; Idaho chemical plant development; isolation, volatility, electrochemistry, ion-exchange, solvent-extraction, metal-dissolution, and cost studies; equipment and materials evaluation; and equipment decontamination.

INTRODUCTION

The major programs in the Chemical Technology Division during the quarter ending August 20, 1952 were Purex process development, Thorex process development, and development of processes for homogeneous reactor fuel preparation and product recovery.

The major effort on the Purex program in the Laboratory Section was expended on uranium purification and plutonium recovery from metallurgical wastes. Purex runs in the Pilot Plant were made on a 75-kg/day scale, and the plutonium was processed through both solvent-extraction and ion-exchange cycles. In the Design Section, preparation of material-balance and equipment flow sheets for use in an economic development study of a Purex plant was begun.

The first phase of the Scrup program, which was concerned with isolation of plutonium with various histories from Chalk River reactor-irradiated metal, was completed in the Pilot Plant Section, and the recovery of the bulk of the plutonium was begun.

Homogeneous reactor fuel reprocessing by solvent-extraction methods was emphasized in the Laboratory Section.

In the Thorex program, laboratory-scale studies were carried out on alternate solvent compositions and solvent recovery. In the Unit Operations Section, studies were made on the Thorex solvent-extraction process, and the Thorex flow sheet No. 2 was tested on the concatenated pulse column. In the Design Section, design studies for a Thorex pilot plant were begun.

In an attempt to improve the isolation processes for plutonium and uranium, means of increasing the uranium concentration in the uranium product solution were emphasized.

The properties of uranium trioxide slurries, for use in the homogeneous reactor, were studied in the Laboratory Section.

Chemical engineering research in the Unit Operations Section included work on pulse generators. Various pieces of equipment were tested, and studies were continued on radiation- and reagent-resistant plastics and coatings, equipment decontamination, and continuous metal dissolving. In the Design Section, cost studies of the Idaho plant were continued.

CHEMICAL TECHNOLOGY DIVISION QUARTERLY PROGRESS REPORT

SUMMARY

Purex Process. Flow sheet conditions for dissolution of slag and crucible waste were fixed so that 99.5% plutonium recovery is possible. Material-balance and equipment flow sheets for the Purex plant design study were approximately 75% completed.

Homogeneous Reactor Fuel Reprocessing. Tributyl phosphate extraction of plutonium and uranium from 45-day-cooled fuels was shown to decontaminate the product satisfactorily from neptunium.

Thorex Process. The Thorex pilot-plant design is proceeding on schedule. Detailed design of some equipment components has been started, and materials of construction are on order. A suitable Thorex feed was obtained in a continuous-dissolving process for thorium slugs.

Isolation Processes for Plutonium and Uranium. In the uranium-isolation (ion-exchange) process studies, a method was found for increasing the uranium concentration of the eluate and the speed of the elution.

Homogeneous Reactor Slurries. Mild dynamic studies on type 347 stainless steel showed that this material is resistant to the effects of uranium trioxide slurries at 250°C. Static corrosion tests on pretreated samples showed no indication of an advantage of one kind of pretreatment over another.

Chemical Engineering Research. Power requirements and pressure conditions for large (up to 50 in. in diameter) pulse columns were determined.

---

1. PUREX PROCESS

The purpose of the Purex project is to develop and demonstrate a continuous solvent-extraction process that will be more efficient and economical than other processes for the separation and decontamination of plutonium and uranium from irradiated uranium metal. The scope of the project includes chemical and equipment development and the design, construction, and operation of a hot pilot plant.

Present plans are to use the Purex flow sheet No. 1 at the Savannah River plant, with an ion-exchange process to couple the solvent extraction with the peroxide precipitation.

Purex Process Demonstration. The Purex solvent-extraction process was satisfactorily demonstrated in the pilot plant pulse-column equipment at a uranium throughput of 75 kg/day on Hanford material that had been irradiated to 550 Mwd/ton and cooled 120 days (runs HCP-6 through 10). Plutonium recoveries greater than 99.8% were obtained without nitrite treatment of the process feed. Nitrogen dioxide gas was found suitable for plutonium valence adjustment.

The plutonium from the solvent extraction was concentrated by an ion-exchange (Dowex-50 resin) process, and the product met specifications. An over-all gross beta decontamination factor of 5×10^7 was achieved through the solvent extraction and ion exchange. The resin used in the ion-exchange process was successfully decontaminated from large amounts of beta and gamma activity with oxalic acid and ammonium citrate.

A detailed report[1] on plutonium isolation by ion exchange in runs HP-6 through 14, SP-20, and HCP-1 through 5 was prepared.

Satisfactory ruthenium decontamination across the second uranium cycle was achieved by the use of sodium nitrite in the ID feed. Nitrogen dioxide was found to be equally as satisfactory as sodium nitrite for adjusting the valence of the ruthenium. An over-all gross beta decontamination factor of 1.4×10^7 was achieved for the uranium through the solvent-extraction process and the silica-gel treatment. The uranium recovery was 99.99%.

Laboratory Studies of Purex Conditions. Laboratory-scale studies on the stability of the Purex solvent to nitric acid were continued during the quarter, and results indicate that very little nitrogen is fixed by the solvent. As solvent nitration increases, however, more trouble is experienced with such effects as emulsification and crud formation, and these may be the limiting factors in the useful life of the solvent rather than significant changes in such physical properties as viscosity, density, or flash point.

Studies on the distribution of uranium, plutonium(IV), and specific fission products between 30% tributyl phosphate-Amsco solvent and nitric acid at several temperatures indicate that operation of the extraction column at a higher temperature is undesirable. The extraction of both plutonium and fission products is increased as the temperature is raised and that of uranium is decreased.

Plutonium Recovery from Slag and Crucible Waste. During the quarter, seven pilot-plant-scale slag and crucible dissolution runs were made with an average plutonium loss of 0.43% to the solid residue. The flow sheet conditions required to give 99.5% plutonium recovery were fixed at (1) controlled dissolution with nitric acid in the presence of free fluoride and (2) a dissolution time of 70 min or longer at temperatures above 100°C. The use of dilute nitric acid (6 to 9 *N*) in the initial dissolution eliminates the need for using sodium nitrite to reduce iodate to iodine. The iodine volatilizes from the dissolver; only about 0.1 g/liter is left in the solution. This concentration is not considered harmful to the process.

The rate of corrosion of type 309Cb stainless steel by synthetic slag and crucible solution was sufficiently low to indicate that this material might be suitable for use in equipment for slag and crucible dissolution.

Twelve grams of purified plutonium was recovered from 120 liters of slag and crucible solution by solvent extraction and Dowex-50 resin ion exchange. An over-all decontamination from aluminum of the order of 10^5 and from calcium and magnesium of the order of 10^4 was achieved. The purity of the plutonium was comparable with that of the Purex product before ion-exchange isolation. As much as 18% of the plutonium in the dissolver solution is plutonium(VI), but, on a laboratory scale, this is satisfactorily reduced to plutonium(III) with ferrous sulfamate and oxidized to plutonium(IV) with sodium nitrite.

---

[1] D. C. Overholt, F. W. Tober, D. A. Orth, *An Ion-Exchange Process for Plutonium Isolation and Purification*, ORNL-1357 (Sept. 16, 1952).

2. SCRUP PROGRAM

The objective of the Scrup program is the recovery of plutonium from Chalk River reactor-irradiated uranium, which differs significantly from Hanford-produced plutonium in isotopic content and therefore was not processed in the Hanford plant. The program includes chemical and engineering development, modification of the existing plant, and operation of the modified plant. The Scrup process is a further development of the Purex process, which is being used for the recovery of plutonium and uranium. The Scrup plant constitutes, in effect, a demonstration of the natural-uranium type of radiochemical processing plant.

The 3505 Pilot Plant, which was modified for use in the Scrup process, is a direct-maintenance plant. The columns have a uranium capacity of 500 kg/day, but the plant capacity is limited at present by the dissolver capacity, which is 100 kg/day.

The first phase of the Scrup program, which was designed to recover plutonium with various radiation histories from Chalk River irradiated metal, was completed during the quarter. Since the primary interest was plutonium, two plutonium cycles and one uranium cycle were provided and the uranium was stored for future processing. Final concentration of the plutonium was by ion exchange, and the over-all loss of plutonium in the process was 0.08%. The plutonium was transferred to Los Alamos and Y-12.

The second phase of the program – the recovery of all plutonium from the bulk of the Chalk River material – is in progress and is scheduled for completion in October.

3. HOMOGENEOUS REACTOR FUEL REPROCESSING

The purpose of the homogeneous reactor fuel reprocessing program is to develop a chemical process for recovery of the products from a homogeneous plutonium-producing reactor. The work will include chemical, equipment, and pilot-plant development, and design, construction, and operation of a plant to process the fuel from the intermediate-scale homogeneous reactor. Studies have been confined to a heavy water solution of uranyl sulfate type of fuel, but, in general, the results apply equally well to a uranium oxide slurry fuel. Details of the studies are reported in the homogeneous reactor project quarterly reports.[2]

Previous studies[3] had shown that, owing to the extraction of Np^{239}, tributyl phosphate extraction did not adequately decontaminate uranium and plutonium from fuels that had cooled for 15 to 20 days. Further studies have now shown that, if the fuel is is allowed to cool for 45 days (the time expected to be used in the process) a one-cycle tributyl phosphate extraction decontaminates the plutonium and uranium from neptunium in excess of the required factors of 10^4 and 10, respectively.

As far as is now known, the radiation damage to the solvent will not significantly affect the uranium and plutonium decontamination. Mixtures of tributyl phosphate in Amsco diluent irradiated

(2) See, for example, *Homogeneous Reactor Project Quarterly Progress Report for Period Ending July 1, 1952*, ORNL-1318.

(3) *Chemical Technology Division Quarterly Progress Report for Period Ending May 20, 1952*, ORNL-1328.

for five days in the 6000-r/min Co^{60} source, which is roughly 300 times the maximum radiation dosage expected in the process (i.e., 300 times the radiation from 15-day-cooled fuel, the minimum cooling expected), showed only a hundredfold increase in the extraction of fission products and retention of plutonium under simulated process conditions. Since the amount of the damage was also shown to be roughly proportional to the square of the dosage, about 30 times the process dosage would be required to produce appreciable damage to the solvent.

Results of studies of plutonium solubility in 1 *M* uranyl sulfate at 100 and 250°C showed the solubility of tetravalent plutonium at 250°C to be about one-third of that at the lower temperature (0.003 and 0.010 mg/ml, respectively). Any excess plutonium hydrolyzed and precipitated as the dioxide; the reaction was more rapid at the higher temperature.

No differences were apparent in the oxidation-reduction behavior of plutonium at 100 and 250°C, but the results of experiments that were conducted are difficult to interpret in terms of the precipitation of plutonium to be expected in a homogeneous reactor operating at either of the two temperatures. At initial hexavalent plutonium concentrations above 0.1 mg/ml, significant reduction to tetravalent plutonium occurred; at initial plutonium concentrations below 0.05 mg/ml, and in the presence of some tetravalent plutonium, measurable oxidation of the tetravalent form to the hexavalent form occurred. At all concentrations the ratio of hexavalent to tetravalent plutonium remaining in solution after heating under 200 psi of oxygen was 35:1. However, in these experiments no hydrogen was present, and the introduction of this element would probably favor the precipitation of plutonium.

4. THOREX PROCESS

The purpose of the Thorex program is to develop a process for the separation of U^{233}, Pa^{233}, and Th. The 23 Process, previously developed through the pilot-plant stage, recovered only U^{233}. The scope of the project includes chemical, equipment, and pilot-plant development and plant conception studies.

A report[4] was prepared summarizing the laboratory phase of the Thorex development work through June 1, 1952, and giving details of the Thorex flow sheet No. 2, which was published in the last quarterly report.[3] Continuous dissolving, concatenated pulse columns, 41% tributyl phosphate (TBP) solvent, and a highly acid-deficient aluminum nitrate aqueous scrub solution are basic features of the process.

Results of batch countercurrent tests with preconditioned Amsco 125-90W, which is being evaluated as a diluent for TBP, indicate that its phase-mixing properties and chemical behavior are satisfactory under flow sheet No. 2 conditions. Its physical properties, low aromatic content, and stability to nitric acid make it attractive as a possible diluent.

A continuous solvent-recovery method that uses a calcium hydroxide slurry in contact with used solvent for removal of impurities and decomposition products of TBP was found to be more efficient and economical than sodium hydroxide–sodium carbonate procedures. Settling and filtration properties of the precipitates formed are good, and the solid and aqueous waste volumes are 100 times less than

[4] A. T. Gresky, J. E. Savolainen, W. T. McDuffee, S. S. Brandt, M. R. Bennett, *Progress Report: Laboratory Development of the Thorex Process*, ORNL-1367 (to be published).

those in the sodium hydroxide-sodium carbonate procedure.

Physical data of all process solutions - densities, viscosities, freezing points, conductivities, and pH - are being determined to provide detailed information for engineering and design work.

The Thorex pilot-plant design is proceeding approximately on schedule. Detailed design of some equipment components has been started, and chemical flow sheets and schematic drawings of equipment are being revised. Most of the materials of construction have been ordered, but promised deliveries are not satisfactory and may result in construction delays.

A Thorex preliminary plant study is under way. The results of this study are intended to dictate the pilot-plant design and allied development programs to ensure that items potentially of primary interest to a production facility are not overlooked in the development stage.

Pulse-column studies on the Thorex solvent-extraction process have been limited by silica impurity in the thorium nitrate and by tributyl phosphate hydrolysis products in the solvent, and laboratory work is being done on the removal of these impurities. However, preliminary indications are that the pilot-plant A column will probably be approximately 3.5 in. in diameter for operation at 60% of flooding and will have 12 ft each of extraction and scrubbing sections. The dimensions for the B column may be as great as 5 in. in diameter and 50 ft in height. A recommendation was made to confine all studies to the 41% TBP flow sheet because the 54% TBP flow sheet necessitated a column startup procedure involving initial operation at one-half flow rate to avoid flooding.

Work was continued on the concatenated pulse column.[3] A unit with four 4-ft sections was tested with the Thorex preferential-strip column flow sheet and was found to give satisfactory performance. Extraction efficiency of this unit was similar to that of the vertical column, flooding rates were 10 to 15% higher, and temporary cessation of the operation did not result in complete top-to-bottom separation of the two liquid phases, as happens in the vertical column.

5. IDAHO CHEMICAL PROCESSING PLANT

The first, and most important, phase of the Idaho Chemical Processing Plant program is concerned with satisfactory operation of the plant, which includes assistance in the design, field engineering, and plant-acceptance tests. The second phase is concerned with solving the problems that inevitably arise in starting the plant. ORNL engineers and technicians will remain at Arco until operation of the plant at full activity levels starts, which should be in November 1952.

The laboratory building, including the works laboratory, has been turned over to the operator, American Cyanamid Co., and only a few areas in the building, such as the basement machine shop, remain to be completed. Necessary changes in the service building have been made, the boilers are being tested, and the building is to be accepted by American Cyanamid Co. as soon as several items now on order are delivered and installed. This is scheduled tentatively for September 1, 1952.

In the waste-disposal building, construction and inspection are almost

complete, and during the quarter most of the instrumentation was completed. The liquid-waste-handling area was turned over to the operator for vessel calibration, water runs, and detergent cleaning. The tank farm area is still incomplete. Work on the dissolver off-gas system is continuing.

In the process building, construction is complete except for a few items, such as samplers. Water and detergent runs have been made, and the plant hydrostatic and Freon test is under way. The heating, ventilating, and air-conditioning systems in both the laboratory and process buildings have been balanced.

Operating manuals are being prepared, and analytical methods for use in the control laboratory are being tested. Minor revisions and adjustments are being made in the sample-handling facilities and in devices for use in the analysis of radioactive samples.

6. ISOLATION PROCESSES FOR PLUTONIUM AND URANIUM

The purpose of the isolation program is the development of processes for purification of plutonium and uranium that will be more economical and that will give a purer product than existing processes. The scope of the program includes chemical, equipment, and pilot-plant development and design studies.

Evaporation techniques were formerly used to concentrate solutions of Pu, U^{233}, and U^{235} isotopes prior to their precipitation as the peroxides. A satisfactory ion-exchange method of concentrating these solutions has been developed which gives, in addition, some separation from the ionic impurities present. Direct application of the ion-exchange method is being made in the Purex, U^{233}, and Thorex processes.

During the past quarter, in the application of this procedure to the U^{233} separation problem, studies were carried out specifically on increasing the uranium concentration of the U^{233} product eluate. Changing the acetic acid concentration of the 2.0 *M* ammonium acetate elutriant from 0.2 to 0.8 *M* was shown to give greater than 99.8% elution, with a uranium concentration in the product of 120 g/liter, in contrast to the 75 g/liter formerly obtained. Raising the elution temperature from 50 to 70°C increased the uranium concentration of the eluate about 10%. Increasing the elutriant flow rate from 0.25 to 0.5 $ml/min/cm^2$, which decreases the elution time to about 2.5 hr, had no deleterious effect on the uranium concentration of the eluate.

Uranyl absorption by the resin was shown to be satisfactory when the flow rate of the feed to the column was increased from the 1 $ml/min/cm^2$ formerly used to as much as 15 $ml/min/cm^2$.

A detailed report on uranium isolation by ion exchange was prepared.[5]

7. HOMOGENEOUS REACTOR SLURRIES

The purpose of the homogeneous reactor slurry program is to develop a method for preparing uranium and thorium systems to be used as fuel media in homogeneous reactors. The development of fuel systems other than aqueous solutions is important because the use of aqueous fuels is limited by solubility considerations, corrosion

[5] D. C. Overholt, *An Ion-Exchange Process for Uranium-233 Isolation and Purification*, ORNL-1364 (to be published).

problems, and the cross sections of added anions. The program has thus far been concerned primarily with the development of uranium slurries, although a limited amount of work has been done on fused fluoride salt systems. Studies will include an investigation of methods of preparing slurries and the determination of their radiation stabilities. Details of the studies are reported in the homogeneous reactor project quarterly reports.[2]

A literature search was made for available information on corrosion of stainless steel by water and uranium oxide at high temperatures, but very few data were found. The results of mild dynamic corrosion tests on samples of type 347 stainless steel showed that water and uranium trioxide slurries attack this material only slightly in 20 hr at 250°C, that is, at a corrosion rate of less than 1 mil/year. During this slight attack, superficial films which protect the surface are formed; therefore the rate of corrosion decreases as the length of exposure increases. Dissolved salts slightly accelerate the corrosion and increase the soluble uranium by a small amount. In static corrosion tests, type 347 stainless steel bombs for use in the radiation stability studies were pretreated with hydrofluoric acid–nitric acid and then with nitric acid, dry oxygen, or oxygen plus steam. Corrosion rates of less than 1 mil/year were obtained in all three cases. There was no indication of an advantage of one pretreatment over another.

Previous studies had shown that even small amounts of impurities, especially uranyl and nitrate ions, influence markedly the characteristics of UO_3 slurries that are heated at 250°C. Two methods of purifying these slurries were studied and found useful: (1) repeated hydration and dehydration and (2) grinding, washing, and digesting in water at 250°C. The second was the better method, but results were not entirely reproducible on a laboratory scale.

The possibility was considered that the reduction of UO_3 slurries that occurs under irradiation is brought about by nitrite ion formed from nitrate impurity by irradiation. However, in experiments in which UO_3 and nitrite ion were kept at 250°C for as long as 112 hr, there was no evidence of reduction of the UO_3.

Hole 11 of the ORNL graphite reactor has been modified and instruments have been installed for recording pressure and for controlling and recording temperature for work especially on reactor irradiation of enriched-uranium slurries in steel containers. In a 21-day irradiation of a natural-uranium $UO_3 \cdot H_2O$-platelet slurry containing 0.01% nitrate, the gas production was insignificant.

Single-crystal x-ray-diffraction studies made in the Chemistry Division with a precession camera indicated that the cell sizes and intensities of reflections of the $UO_3 \cdot H_2O$ bipyramid crystals are those of a β-$UO_3 \cdot H_2O$.[6] This establishes the fact that the bipyramids, originally thought to be triclinic, are orthorhombic in structure.

Several methods of determining particle size were investigated. Comparison of the results obtained in preliminary experiments indicated that at a uranium concentration of 100 g/liter in the slurry, the sedimentation-rate method gives results that are in fair agreement with the standard nitrogen-adsorption method. The work is not complete.

---

[6] W. H. Zachariasen, *Report from July 1 to December 31, 1946, of Mass Spectroscopy and Crystal Structure Division*, CP-3774, p. 20.

A spray dryer similar to the Harvard unit[7] was tested for spray-calcining of uranyl nitrate hexahydrate to uranium hydroxide. Because the product contained soluble uranyl nitrate and U_3O_8, it was unsatisfactory, and development work on this dryer was discontinued. A 2-in. Micronizer has been used satisfactorily for reducing the amount of soluble uranyl nitrate in Mallinckrodt uranium trioxide along with reducing the particle size. The Micronizer has been suggested for calcining uranyl nitrate hexahydrate by using superheated steam and recycling the UO_3. Tests on this equipment have been delayed pending development of a more satisfactory method for analyzing the product for nitrate.

8. VOLATILITY

The purpose of the volatility program is to study the application of volatility techniques to the solution of AEC separation problems. The study will include consideration of volatile compounds of uranium, plutonium, and fission products and of structural materials of interest in the reactor program. Both chemical and equipment development studies will be made.

As a part of this program an attempt is to be made to prepare uranium hexafluoride directly from uranium oxides and from uranyl sulfate. Antimony pentafluoride, which is being considered for the fluorinating medium, is not commercially available in sufficient purity, and attempts to make this product have so far been unsuccessful. It is expected that the work will be continued.

A small distillation system, which consists of two columns with the necessary auxiliaries for completely decontaminating UF_6, is being designed. Efforts are being made to reduce the operating holdup to a minimum so that the equipment can be operated with a total of 500 ml of liquid UF_6, the amount obtained by fluorinating one X-slug. Some of the proposed auxiliary equipment has been tested.

9. ELECTROCHEMISTRY

The successful application of electrochemical techniques to separation processes may significantly reduce chemical and waste storage costs. The purpose of the electrochemistry program is to study the electrochemical properties of source and fissionable materials and of fission products.

During the quarter, studies on the electrochemical production of deuterium were continued, with the object of combining high separation factors for hydrogen and deuterium with low energy requirements in a cell of high current capacity. A survey of electrolytic methods has indicated that (1) heavy water may be separated as a by-product in either the electrolytic production of hydrogen or the electrolytic consumption of hydrogen in a fuel cell and (2) production and consumption of hydrogen may be combined to give a cell with a low power requirement.

In the cathodic reaction of hydrogen the usual separation factor is of the order of 3 to 6. In laboratory experiments, factors in the range from 10 to 20 have been obtained. Further study of cell reactions is needed to determine the conditions that bring about a high separation factor.

The capacity and energy requirement of a cell are dependent on the electrode spacing and the development of electrode surface. After an electrolytic process of surface development, a fine nickel-screen electrode (0.003 in. thick) was

[7] M. Eisenbud, *Monthly Report of Laboratory Activities, July, 1950*, NYO-1529.

found, over a range of 0.05 to 0.2 v, to carry from 100 to 1000 times the amount of current that was carried by a smooth-surface nickel electrode. If such developed-surface electrodes were installed with 0.02- to 0.04-in. spacing, that is, 25 to 50 electrodes per foot (which has been done in a small section of a cell), the total electrode area would be 2,500 to 50,000 ft^2 per cubic foot of cell.

A study of the anodic separation factor has been started with palladium electrodes. The investigation will include studies on surface development, catalytic structure, rate and depth of hydrogen penetration, and exchange currents.

In order to include gases as a source of deuterium, samples of fine nickel screen, porous carbon, and porous nickel have been obtained for the preparation of electrodes to be used in the study of surface development in connection with gas-diffusion electrodes.

10. ION EXCHANGE

The purpose of the ion-exchange program is to investigate the application of ion exchange to AEC separation problems. The problems that will be investigated include evaluation and development of continuous ion exchange as a separation technique and the use of ion exchange for uranium, plutonium, and fission-product separations and for waste processing. The scope of the program will be primarily chemical and equipment development.

Work to date has been concerned principally with development of a continuous ion-exchange column and with radiation stability of ion-exchange resins.

Two models of the Higgins continuous ion-exchange column[8] have been operated successfully, and a third model, which features standard equipment, has been nearly completed by the Unit Operations Section. The new model is motivated with a 7-in. in diameter bellows equipped with an air motor from Bellows, Inc. A water bleed line, which permits the water to flow with the stroke of the bellows, makes it possible to use one bellows unit instead of two working simultaneously, as in the previous models. It is planned to use a standard timer in this model for automatic control.

A detailed report[9] was prepared covering the work on capacity loss of anion- and cation-exchange resins under irradiation.

Results of experiments on separating uranium and thorium in concentrated chloride solution by an anion-exchange resin indicated that a practical process for separating U^{233} from irradiated thorium might be worked out.

11. CHEMICAL ENGINEERING RESEARCH

The chemical engineering research program is a long-range program with the objective of reducing radiochemical processing costs. The high cost of radiochemical process plants emphasizes the need for applied research studies of the chemical engineering aspects of radiochemical processes. The program involves radiochemical unit process and unit operation evaluation studies supported by experimental research effort. Typical fields of study may include solvent extraction,

(8) I. R. Higgins, *A Countercurrent Solid-Liquid Contactor for Continuous Ion Exchange*, ORNL-1310 (Sept. 4, 1952).

(9) I. R. Higgins, *Radiation Damage to Organic Ion Exchange Materials*, ORNL-1325 (to be published).

metal dissolution, liquid-solid separations, ion exchange, evaporation, precipitation, equipment evaluation, materials evaluation, surface decontamination, radiation stability, and plant economic studies.

Solvent-Extraction Studies. The objective of the solvent-extraction work is to evaluate different types of contactors, including packed, pulsed, and Scheibel columns, mixer-settlers, and the Podbielniak centrifugal contactor. At present, emphasis is being placed on the application of pulse columns to various processes and the development of pulse generators.

Pulse-generator development work during the quarter included determination of power requirements and pressure conditions for large pulse columns. Columns 24 to 50 in. in diameter and 50 ft in height will require pulse-generator drives in the range 50 to 150 hp if liquid-liquid systems with high average specific gravity (3.0 to 4.0) and pulse frequencies up to 200 cycles/min are incorporated in the application. Cyclic varying of the pressure conditions from 20 to 170 psi in each cycle will necessitate special pumping considerations. Reciprocating pumps may have to be used instead of centrifugal pumps for the influent and effluent streams at the base of the column.

The Vickers hydraulic-drive pulser system installed last quarter was operated continuously for approximately 80 days without mishap. All parts of this unit are commercially available, and it can be used to drive several adjacent piston pulsers. Both the amplitude and frequency of the pulse can be varied.

A special 5-in. mechanical-drive pulser (Ultramotor) with variable stroke pattern was found satisfactory in a 30-day test. This unit is being prepared for "semisquare" pulse-wave tests.

Four mechanical-drive piston units, providing a simple harmonic pulse wave, for the 6- and 9-in.-dia pulse columns in the Metal Recovery Building were designed and constructed. Testing and installation of these units are expected to be completed by September 1.

Two Graphitar-piston pulse generators driven by double-acting pneumatic piston motors, which were controlled by a microswitch-solenoid system, were provided for use on the Purex pilot-plant IC and IE columns. These installations have reduced the uranium losses from 0.2 to 0.005% in each column.

A study was begun on a new method, which has been studied[10] at the University of Texas, of dispersing the discontinuous phase in liquid-liquid extractions. In this system the dispersed phase is comprised of composite bubbles of uniform size, each of which consists of a gas bubble enclosed in a sheath of the dispersed liquid phase. This type of dispersion may have the advantage of relatively high throughput rates that may be preserved at high surface-to-volume ratios for the dispersed liquid phase. A modified form of the Texas equipment has been designed, and it is planned to determine data of a fundamental nature, such as the pressure drop through the column and mass-transfer information.

Equipment and Materials Evaluation. Studies on the resistance to radiation of various plastics were continued in two new Co^{60} sources of approximately

(10) N. S. Rao, *An Improved Method of Dispersing the Discontinuous Phase in Liquid-Liquid Extraction Spray Columns*, M. S. Thesis, University of Texas (May 1952).

2.24×10^6 and 1.92×10^6 r/hr intensity. Carbon-filled polyethylene (1% carbon) was not superior to natural polyethylene in a gamma-radiation field. Homalite CR-39, a thermosetting resin, was found to deteriorate at 10^9 r but was considered satisfactory for use in a viewing window up to 10^8 r. Irradiation of unplasticized, rigid polyvinyl chloride, carbon tetrachloride, and plasticized and unplasticized Fluorothene resulted in evolution of halogen and deterioration of the plastic. Results of these experiments have been reported.[11] Various service irradiations were conducted during the quarter.

A satisfactory and practicable-to-apply protective coating for Azphex asphalt tile has not been found, but a Fiberglas membrane secured to the tile with a pressure-sensitive adhesive looks promising. Trial quantities of Fiberglas and of PS-1 adhesive (Minnesota Mining and Manufacturing Co.) have been received and will be applied to a test area.

At the request of Du Pont, coatings from various manufacturers were tested for ease of decontamination and resistance to the acid and organic solutions used in chemical processing of radioactive materials. The following were satisfactory enough to warrant further consideration: Del Medium Gray No. 78 (David E. Long Corp.), Sealcoat No. 15 and 18 (Carboline Co.), Nukemite No. 40 and 77 (Nukem Products Co.), Corrosite No. 22 (Corrosite Corp.), and Ucelon, System E (United Chromium, Inc.).

A four-component coating system of flame-spraying polyethylene, devised at Powder Weld Process Laboratory, was found to be fairly easy to decontaminate. It is considered to be useful in protecting both metallic and concrete surfaces in radioactive environments.

Cost Studies. An economic study program has been undertaken to obtain cost data on design engineering, construction, and operation of radiochemical plants so that present philosophies and design practices may be reviewed for the ultimate purpose of reducing costs. There are three phases of this program: (1) accumulation of data; (2) determination of the effects on plant costs of various factors such as criticality, radiation, waste disposal, and feed preparation; and (3) preliminary plant economic studies.

All major cost items of design, construction, and preoperation have been received from the Bechtel Corp., including charges of the Idaho Falls Operations Office, of American Cyanamid Co., and of the design and construction forces. Of the total costs, 11.6% went for design, 78.7% for construction, and 9.7% for preoperation. A breakdown of the construction costs is as follows:

	PER CENT OF CONSTRUCTION COSTS
Site development	7.1
Storage	5.5
Processing	46.5
Waste handling	20.0
Laboratory	13.7
Services	7.2

Analysis of the various costs incurred during construction will be continued in order to obtain cost factors that will be useful in predicting the costs of new plants and in highlighting steps in the process where new development work would possibly show major cost reduction.

(11) *The Effect of Gamma Radiation on Polyvinyl Chloride and Fluorothene*, MIT Practice School Report EPS-X-172 (to be published).

Material and equipment flow sheets, which are being prepared as the first phase of a Purex plant design study, are approximately 75% complete.

Metal-Dissolution Studies. The objective of the metal-dissolution studies is the conversion from a batch to a continuous dissolution process in the preparation of solid reactor fuels for feeding to recovery plants. In small-scale studies, continuous dissolving has been shown to be feasible for uranium, thorium, and uranium-aluminum alloys. A large-scale continuous dissolver of the "trickle" type, in which no liquid level is maintained in the column, is being constructed and will be installed shortly.

During the past quarter, small-scale studies were concentrated on continuous dissolving of unjacketed thorium slugs in the trickle type of dissolver. With a dissolver feed 7.33 *N* in nitric acid, continuous dissolution was maintained, and a product that was suitable for the Thorex flow sheet No. 2 feed was obtained without adjustment of solution concentrations. The dissolving rate in the trickle dissolver could not be measured when 13 *N* nitric acid, which was used in earlier studies[12] in one- and two-section semiflood models, was used. The dissolving rate was so high that below 25 ml/min the solution was too concentrated and solidified in the product line, and above this rate the reaction was so vigorous that foam passed into the off-gas line and condenser.

Equipment-Decontamination Studies. The purpose of the equipment-decontamination program is to conduct both fundamental and applied studies aimed at developing improved methods for decontamination of chemical process equipment. Application of these methods to the decontamination of homogeneous reactor components is being studied. Because of the ease of application of reagent decontamination, this type is favored over electrolytic or mechanical decontamination.

A commercial product, Copper-Brite, was found effective in decontaminating copper, brass, and types 304ELC, 316, 309Cb, and 347 stainless steel, but not in decontaminating aluminum. This material should prove useful in decontaminating instruments, tools, and working surfaces.

Studies on decontamination of No. 7 (mirror) finish stainless steel with 3-20 reagent (3 wt % HF-20 wt % HNO_3) did not indicate any superiority of this finish over the other seven commercially available finishes. The less expensive finishes may therefore be used in work with radioactive materials.

Attempts to find a process for recovering contaminated 3-20 reagent for reuse were continued. Preliminary studies of activity removal by adsorption and precipitation methods indicate that a decontamination factor of 6 to 8 can be obtained by adsorption on activated alumina, and that a decontamination factor of about 13 can be obtained by a lanthanum trifluoride precipitation. Removal of 80% of the fluoride ion was obtained with calcium hydroxide and aluminum sulfate. These studies will be continued.

Batch distillation studies on 3-20 reagent indicate that although the hydrogen fluoride concentration in the

(12) *Chemical Technology Division Progress Report for Period August 20, 1951 to February 10, 1952*, ORNL-1311.

still bottoms is lower than was first expected, it is still prohibitive as far as finding a material of construction for an evaporator is concerned.

Calculations based on data obtained in a water-HF-HNO_3-fission product-50% tributyl phosphate system indicate that a maximum acid recovery of 72% could be obtained by a solvent-extraction procedure with an infinite number of stages.

During the past quarter, studies were begun on removing scale from the homogeneous reactor piping system. The most effective reagent tried was a mixture of 1 volume of 37% hydrochloric acid, 1 volume of 95% sulfuric acid, and 8 volumes of water at 150°F. The solution caused severe corrosion of the base metal, but there was evidence that the attack could be controlled by the corrosion inhibitor Rodine 41. Since radioactive sections of the loop are not available and analytical methods are not sensitive enough to determine the dead fission products that were added to the uranyl sulfate solution in the mockup, scale removal is being used as the criterion of decontamination. It is, however, possible that complete decontamination may be achieved without complete scale removal.

12. LIST OF REPORTS ISSUED

Following is a list of the reports issued by the Chemical Technology Division during the period May 21 through August 20, 1952:

DOCUMENT NO.	TITLE	AUTHOR	DATE
	Purex Process		
CF-52-6-1	Trip Report, KAPL, February 29 to March 3, 1952	V. A. Ryan	6-3-52
CF-52-6-153	Purex Third-Cycle Decontamination Data	J. R. Flanary	6-25-52
CF-52-6-171	Plutonium Recovered from Reduction Crucibles and Slag	E. B. Sheldon R. L. Folger	6-9-52
CF-52-7-113	Gamma Activity of the Purex IIBP Solution	D. C. Overholt F. W. Tober	7-21-52
CF-52-7-161	Purex Pilot-Plant Uranium Product for Denitration by Harshaw Chemical Co.	W. B. Watkins	7-30-52
CF-52-7-163	Progress Report - Recovery of Uranium from Purex IEU	E. M. Shank	7-29-52
CF-52-8-44	Proposal for Study of a High-Saturation Purex Flowsheet	V. J. Reilly	8-5-52
CF-52-6-121	Pilot Plant Runs HCP-1 through HCP-5 Operational Summary	R. A. Lewis	6-10-52
CF-52-7-66	Pilot Plant Run HCP-6 Summary	J. M. Chandler A. J. Pecquet	7-15-52
CF-52-7-154	Pilot Plant Run HCP-7 Summary	J. M. Chandler A. J. Pecquet	7-21-52
CF-52-5-174	Pilot Plant Status Report	H. K. Jackson	5-23-52

DOCUMENT NO.	TITLE	AUTHOR	DATE
CF-52-6-124	Pilot Plant Status Report	H. K. Jackson	6-23-52
CF-52-7-61	Pilot Plant Status Report	H. K. Jackson	7-10-52
CF-52-7-110	Pilot Plant Status Report	H. K. Jackson	7-24-52
CF-52-8-24	Pilot Plant Status Report	H. K. Jackson	8-5-52
CF-52-6-106	Discussion of Portions of the Savannah River Plant	M. L. Feldman	6-18-52
CF-52-7-153	General Information Pertaining to Savannah River Plant	M. L. Feldman	7-29-52
	Scrup Program		
CF-52-5-172	Estimate of Sr^{90} in Waste from the Pilot Plant and Scrup Programs	H. K. Jackson	5-21-52
CF-52-5-225	SCRUP Run UR-1 Summary	P. H. Porter	5-27-52
CF-52-6-23	SCRUP Run SB-2 Summary	P. H. Porter	6-5-52
CF-52-6-89	SCRUP Run SB-3 Summary	P. H. Porter	6-13-52
CF-52-6-91	SCRUP Run SB-4 Summary	P. H. Porter	6-13-52
CF-52-6-90	SCRUP Run SB-5 Summary	P. H. Porter	6-13-52
CF-52-6-135	SCRUP Run SB-6 Summary	P. H. Porter	6-23-52
CF-52-7-94	SCRUP Run SB-7 Summary	P. H. Porter	7-21-52
CF-52-7-47	Monthly Report for Period 7-10-52	K. L. Mattern	7-10-52
CF-52-8-58	Chalk River Dissolving Samples	W. H. Lewis	8-12-52
	Waste Metal Recovery		
CF-52-6-162	Resolution of the Sodium Diuranate Precipitation in W-7 Metal Waste Tank	J. E. Savolainen	6-26-52
	Homogeneous Reactor Fuel Reprocessing		
CF-52-6-149	ThO_2 Slurries	F. R. Bruce	6-26-52
CF-52-7-111	HRE Chemical Processing Quarterly	D. E. Ferguson	7-1-52
	Thorex Process		
CF-52-5-194	Letter: Thorex	K. L. Mattern	5-27-52
CF-52-6-60	Thorex Process Discussion	K. L. Mattern	6-11-52
CF-52-6-88	Non-radive Thorium Nitrate for Use in Thorex Pilot Plant	A. C. Jealous	6-13-52
CF-52-7-89	Measurement of Gamma Radiation from Irradiated Thorium	W. R. Winsbro	7-18-52
CF-52-7-155	Measurement of Gamma Radiation from Irradiated Thorium	H. K. Jackson	7-31-52
CF-52-8-22	Thorex Development Required Data for Pilot Plant Start-up	K. G. Steyer J. W. Ullmann	8-6-52

CHEMICAL TECHNOLOGY DIVISION QUARTERLY PROGRESS REPORT

DOCUMENT NO.	TITLE	AUTHOR	DATE
CF-52-8-25	Status of Dissolver for Thorex Pilot Plant	A. R. Irvine	8-5-52
CF-52-5-233	Thorex Committee Meeting No. 15, 5-22-52	E. M. Shank	5-23-52
CF-52-7-26	Thorex Information Meeting No. 16, 6-26-52	E. M. Shank	7-7-52
CF-52-7-139	Thorex Corrosion Studies Meeting	A. C. Jealous	7-30-52
CF-52-7-34	Thorex Program	Thorex Committee	7-7-52
CF-52-6-70	Thorex Committee Meeting No. 2, June 10, 1952	E. M. Shank	6-13-52
CF-52-6-133	Thorex Committee Meeting No. 3, June 19, 1952	E. M. Shank	6-23-52
CF-52-7-141	Thorex Committee Meeting No. 4, July 17, 1952, Continuous Dissolving	Thorex Committee	7-30-52
	Interim-23 Process (also see Thorex Process)		
CF-52-5-223	INT-23 Quarterly for Period 2-10-52 to 5-10-52	C. D. Hylton K. G. Steyer	5-29-52
CF-52-6-93	Shipment of Irradiated Thorium (1066 Shipment)	H. K. Jackson	6-17-52
	RaLa Process		
CF-52-7-16	RaLa Project Committee Meeting No. 1	E. J. Witkowski	7-1-52
	Waste Treatment Studies		
CF-52-7-90	Absorption of Waste Furnace Gas	V. J. Reilly	7-17-52
	Isolation Processes for Plutonium and Uranium		
CF-52-6-51	Plutonium Isolation	D. C. Overholt	6-6-52
CF-52-8-67	Dowex-50 Resin Clean-up and Grading Procedure, Reagent Specifications	D. C. Overholt D. G. Karraker D. A. Orth	8-11-52
	Homogeneous Reactor Slurries		
CF-52-5-228	Slurry Fuel Studies Quarterly, 7-1-52	J. O. Blomeke J. P. McBride L. E. Morse F. R. Bruce	5-29-52
	Electrochemistry		
CF-52-5-166	Quarterly Report for Period 2-10-52 to 5-10-52: A Survey of the Electrolytic Method for the Production of Deuterium	J. A. McLaren	5-21-52
	Ion Exchange		
CF-52-6-35	Equipment Development - Small-Scale Continuous Resin Column	J. R. Parrott	6-6-52

DOCUMENT NO.	TITLE	AUTHOR	DATE
CF-52-6-107	An Experimental Comparison of the Higgins Continuous Ion Exchanger with a Conventional Fixed-Bed Ion-Exchange Column	J. T. Roberts	6-18-52
CF-52-8-51	Equipment Development for "Higgins" No. 3 Continuous Ion-Exchange Column, Quarterly, 8-1-52	J. R. Parrott	8-11-52
CF-52-7-132	Equations Useful in Determining Separation Factors by Repeated Batch Extraction and Equilibration		7-14-52
ORSORT Lecture	Principles of Sorption Processes, Lecture No. 27 in Atomic Energy Separations Technology Course		

Solvent Extraction

CF-52-5-186	Concatenated Pulse-Column Quarterly for Period 2-10-52 to 5-10-52	E. Lieberman A. C. Jealous	5-24-52
CF-52-5-202	Pulse-Generator Development Quarterly, 5-10-52	J. E. Conaway A. C. Jealous	5-26-52
CF-52-5-204	Thorex Process Solvent-Extraction Pulse-Column Studies, Quarterly Report for Period 2-10-52 to 5-10-52	J. E. Conaway A. C. Jealous	5-28-52
CF-52-6-22	Solvent Entrainment	V. J. Reilly	6-3-52
CF-52-6-68	Thirty-Inch Diameter Pulse Column	A. C. Jealous	6-12-52
CF-52-8-8	Conference on Solvent Degradation	L. E. Line	8-4-52

Equipment and Materials Evaluation

CF-52-6-87	Sampler Evaluation and Development Quarterly, 2-10-52 to 5-10-52	J. L. Cripple	6-12-52

Metal Dissolution

ORNL-1195	Dissolution of Uranium-Aluminum Alloy Slugs	D. L. Foster E. O. Nurmi	7-7-52

Equipment Decontamination

CF-52-8-33	Decontamination of HRE Loop, Report for July 1952	M. L. Feldman	8-10-52
CF-52-8-42	Recovery of Used 3-20 Decontamination Reagent, Quarterly, 8-1-52	M. L. Feldman	8-8-52

25 Process

CF-52-8-50	Letter: 25 Process	W. B. Lanham	8-7-52
CF-52-6-29	Letter: 25 Process	W. B. Lanham	6-9-52

DOCUMENT NO.	TITLE	AUTHOR	DATE
	Miscellaneous		
CF-52-6-30	Monthly Report, 5-10-52 to 6-10-52	M. L. Feldman	6-5-52
CF-52-6-123	An Outline of Chemical Processing in the Atomic Energy Program	W. B. Lanham	6-23-52
CF-52-7-84	Forwarding of Classified Documents	M. L. Feldman	7-15-52
CF-52-8-47	Process Design Section Status Report, 8-1-52	F. L. Culler R. P. Milford W. G. Stockdale W. E. Unger	8-7-52
CF-52-6-143	Report of Meeting with KAPL Cost Study Engineers	F. L. Culler	6-24-52
CF-52-6-152	Report of Meeting with Catalytic Construction Co. on Fernald Cost Studies	F. L. Culler	6-25-52
CF-52-7-48	Monthly Report 7-10-52	M. L. Feldman	7-10-52
CF-52-7-63	Monthly Report for July 1952	K. G. Steyer	7-8-52
CF-52-6-36	Monthly Report for June 1952	K. G. Steyer	6-9-52
CF-52-5-196	Laboratory Weekly Status Report, Week Ending 5-23-52	F. R. Bruce	5-27-52
CF-52-6-12	Laboratory Weekly Status Report, Week Ending 5-29-52	F. R. Bruce	6-3-52
CF-52-6-45	Laboratory Weekly Status Report, Week Ending 6-6-52	F. R. Bruce	6-10-52
CF-52-6-94	Laboratory Weekly Status Report, Week Ending 6-13-52	F. R. Bruce	6-16-52
CF-52-6-138	Laboratory Weekly Status Report, Week Ending 6-20-52	F. R. Bruce	6-23-52
CF-52-6-174	Laboratory Weekly Status Report, Week Ending 6-27-52	F. R. Bruce	6-30-52
CF-52-7-5	Laboratory Section Summary for June 1952	F. R. Bruce	7-1-52
CF-52-7-139	Laboratory Status Report, 7-3-52	F. R. Bruce	7-7-52
CF-52-7-107	Laboratory Status Report, 7-18-52	F. R. Bruce	7-23-52
CF-52-7-146	Laboratory Weekly Status Report, Week Ending 7-25-52	F. R. Bruce	7-30-52
CF-52-8-52	Laboratory Weekly Status Report, Week Ending 8-1-52	F. R. Bruce	8-11-52
CF-52-8-62	Unit Operations Status Report - ADP Program	J. O. Davis	8-1-52
CF-52-5-224	Unit Operations Status Report	J. O. Davis	5-23-52
CF-52-5-231	Unit Operations Status Report	J. O. Davis	5-30-52
CF-52-6-63	Unit Operations Status Report	J. O. Davis	6-6-52
CF-52-6-111	Unit Operations Status Report	J. O. Davis	6-13-52
CF-52-6-147	Unit Operations Status Report	J. O. Davis	6-20-52
CF-52-6-178	Unit Operations Status Report	J. O. Davis	6-27-52
CF-52-7-36	Unit Operations Status Report	J. O. Davis	7-3-52

FOR PERIOD ENDING AUGUST 20, 1952

DOCUMENT NO.	TITLE	AUTHOR	DATE
CF-52-7-152	Unit Operations Status Report	J. O. Davis	7-25-52
CF-52-7-160	Unit Operations Status Report - ADP Program	J. O. Davis	7-25-52
CF-52-8-154	Unit Operations Status Report	J. O. Davis	8-1-52
CF-52-8-73	Progress Report for Period 7-21-52 to 8-10-52	G. R. Tulley	8-11-52
CF-52-7-135	Report on the Dissolution of the First Batch of Single-Channel-Experiment Slugs	W. H. Lewis	7-28-52
CF-52-7-145	Criticality Information Requirements for Chemical Process Plant: Review of Experimental Program for ORNL Criticality	W. K. Eister	7-29-52
CF-52-7-53	Criticality Experiments Program: Program Recommendation by the Chemical Technology Division	F. L. Steahly	7-1-52