STARTUP OPERATION OF A PRODUCTION FACILITY
FOR SEPARATING BARIUM-140 FROM MTR FUEL

B. M. Legler      P. N. Kelly
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by

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R. A. Robinson

ABSTRACT

Described is the startup operation of a batch production facility for separating and purifying barium-140 from short-cooled MTR fuel. Extensive operation involving unirradiated material and low activity level material was conducted prior to high activity level operation.

Heart of processing equipment is a special suspended type, batch, solid basket centrifuge which is used for liquid-solid separation.

The process is based on the solubility characteristics of barium and barium salts in selected basic and acidic media.

Startup operation demonstrated adequacy of flowsheet under irradiation in plant equipment. Recovery of 25,000-curie batches of barium-140 was shown to be possible with adequate reproducibility at an approximate cost of $3.00 per curie. Operating time per batch is 30 hours.

The problem of iodine removal from process off-gas remains unsolved because inadequate provision was made for iodine removal in the original facility.

Work done under contract AT(10-1)-205 to the U. S. Atomic Energy Commission
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Description</th>
<th>Page No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>I.</td>
<td>INTRODUCTION</td>
<td>9</td>
</tr>
<tr>
<td>II.</td>
<td>SUMMARY AND CONCLUSIONS</td>
<td>9</td>
</tr>
<tr>
<td>III.</td>
<td>PROCESS HISTORY</td>
<td>12</td>
</tr>
<tr>
<td>A.</td>
<td>ORNL Experience</td>
<td>12</td>
</tr>
<tr>
<td>B.</td>
<td>Hanford Experience</td>
<td>12</td>
</tr>
<tr>
<td>C.</td>
<td>American Cyanamid Company</td>
<td>12</td>
</tr>
<tr>
<td>D.</td>
<td>Phillips Petroleum Company</td>
<td>12</td>
</tr>
<tr>
<td>E.</td>
<td>Blaw-Knox Company</td>
<td>12</td>
</tr>
<tr>
<td>F.</td>
<td>J. F. Pritchard Company</td>
<td>12</td>
</tr>
<tr>
<td>IV.</td>
<td>PROCESS DESCRIPTION</td>
<td>13</td>
</tr>
<tr>
<td>A.</td>
<td>Chemical Flowsheet</td>
<td>13</td>
</tr>
<tr>
<td>1.</td>
<td>Starting Material</td>
<td>13</td>
</tr>
<tr>
<td>2.</td>
<td>Desired Product</td>
<td>13</td>
</tr>
<tr>
<td>3.</td>
<td>Chemical Flowsheet Description</td>
<td>13</td>
</tr>
<tr>
<td>B.</td>
<td>Engineering Flowsheet</td>
<td>16</td>
</tr>
<tr>
<td>1.</td>
<td>Major Processing Equipment</td>
<td>16</td>
</tr>
<tr>
<td>2.</td>
<td>Design Philosophy</td>
<td>16</td>
</tr>
<tr>
<td>V.</td>
<td>STARTUP PROGRAM</td>
<td>19</td>
</tr>
<tr>
<td>A.</td>
<td>Line Checking</td>
<td>19</td>
</tr>
<tr>
<td>B.</td>
<td>Vessel Calibration</td>
<td>19</td>
</tr>
<tr>
<td>1.</td>
<td>Instrument Checking</td>
<td>19</td>
</tr>
<tr>
<td>2.</td>
<td>Vessel Flush</td>
<td>19</td>
</tr>
<tr>
<td>3.</td>
<td>Vessel Calibration Using Water</td>
<td>19</td>
</tr>
<tr>
<td>C.</td>
<td>Correction of Deficiencies</td>
<td>20</td>
</tr>
<tr>
<td>1.</td>
<td>Vessels and Other Processing Equipment</td>
<td>20</td>
</tr>
<tr>
<td>2.</td>
<td>Electrical and Instrumentation Equipment</td>
<td>22</td>
</tr>
<tr>
<td>3.</td>
<td>Mechanical Equipment</td>
<td>24</td>
</tr>
<tr>
<td>4.</td>
<td>Centrifuge Equipment</td>
<td>26</td>
</tr>
<tr>
<td>5.</td>
<td>Off-Gas System</td>
<td>29</td>
</tr>
<tr>
<td>6.</td>
<td>Miscellaneous</td>
<td>30</td>
</tr>
<tr>
<td>D.</td>
<td>Operation</td>
<td>31</td>
</tr>
<tr>
<td>E.</td>
<td>Cost</td>
<td>31</td>
</tr>
</tbody>
</table>
# TABLE OF CONTENTS (Continued)

## VI. DISCUSSION OF RESULTS
- Operation Involving Non-Radioactive Material
  1. General
  2. Sampling
  3. Operation
  4. Material Balances
  5. Product

- Operation Involving Irradiated Material
  1. General
  2. Sampling
  3. Material Balances
  4. Product
  5. Operating Manpower
  6. Off-Gas Handling
  7. Liquid Waste Handling
  8. Radiation and Contamination
  9. Decontamination

- Operational Difficulties
  1. Dissolution
  2. Centrifugation
  3. Evaporation
  4. Drying
  5. Pressure Pot Operation

## VII. CONCLUSIONS AND RECOMMENDATIONS
- Construction Considerations
- Process Operations

## VIII. PROCESSING EQUIPMENT AND MATERIALS
- Vessels
- Centrifuge Equipment
- Off-Gas Handling Equipment
- Mechanical Equipment
- Shielded Carriers
- Instrumentation
- Shielding
- Decontamination Equipment
- Chemicals Required
TABLE OF CONTENTS (Continued)

IX. APPENDICES ........................................................................................................... 117

A. Detailed Flowsheets ................................................................................................ 118
B. Run Descriptions ...................................................................................................... 129
C. Off-Gas Handling ..................................................................................................... 170
D. Decontamination Experience .................................................................................... 177
E. Detailed Run Sheets .................................................................................................. 184

LIST OF TABLES

<table>
<thead>
<tr>
<th>Table No.</th>
<th>Title</th>
<th>Page No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Estimated RaLa Production Cost</td>
<td>31</td>
</tr>
<tr>
<td>2</td>
<td>Data for Runs Involving Unirradiated Material</td>
<td>33</td>
</tr>
<tr>
<td>3</td>
<td>Major Component Material Balances for Runs Involving Unirradiated Material</td>
<td>35</td>
</tr>
<tr>
<td>4</td>
<td>Product Composition for Runs Involving Unirradiated Material</td>
<td>36</td>
</tr>
<tr>
<td>5</td>
<td>Contaminants in 75 ml. of 70 percent HNO₃ Leachate from Run 15 Product</td>
<td>37</td>
</tr>
<tr>
<td>6</td>
<td>Data for Runs Involving Irradiated Material</td>
<td>38</td>
</tr>
<tr>
<td>7</td>
<td>Major Component Material Balances for Runs Involving Irradiated Material</td>
<td>40</td>
</tr>
<tr>
<td>8</td>
<td>Basic Chemical List</td>
<td>72</td>
</tr>
<tr>
<td>9</td>
<td>Iodine-131 Discharge to Atmosphere</td>
<td>172</td>
</tr>
<tr>
<td>10</td>
<td>Comparison of Iodine Adsorbers</td>
<td>174</td>
</tr>
<tr>
<td>11</td>
<td>Iodine Adsorption on Various Depths of 1-Inch Diameter Carbon Beds</td>
<td>175</td>
</tr>
<tr>
<td>12</td>
<td>Decontamination Results</td>
<td>183</td>
</tr>
<tr>
<td>Figure No.</td>
<td>Title</td>
<td>Page No.</td>
</tr>
<tr>
<td>-----------</td>
<td>-------------------------------------------------</td>
<td>----------</td>
</tr>
<tr>
<td>1</td>
<td>Simplified Chemical Flowsheet</td>
<td>17</td>
</tr>
<tr>
<td>2</td>
<td>Simplified Flow Diagram</td>
<td>18</td>
</tr>
<tr>
<td>3</td>
<td>Process Makeup Area</td>
<td>49</td>
</tr>
<tr>
<td>4</td>
<td>Operating Corridor Control Area</td>
<td>50</td>
</tr>
<tr>
<td>5</td>
<td>Main Control Panel, Access Corridor Area</td>
<td>51</td>
</tr>
<tr>
<td>6</td>
<td>South Face of Cell L, Access Corridor Level</td>
<td>52</td>
</tr>
<tr>
<td>7</td>
<td>RaLa Cell Model</td>
<td>53</td>
</tr>
<tr>
<td>8</td>
<td>Equipment Layout - Plan</td>
<td>75</td>
</tr>
<tr>
<td>9</td>
<td>Equipment Layout - Section AA</td>
<td>77</td>
</tr>
<tr>
<td>10</td>
<td>Equipment Layout - Section BB</td>
<td>79</td>
</tr>
<tr>
<td>11</td>
<td>Dissolver, L-101</td>
<td>81</td>
</tr>
<tr>
<td>12</td>
<td>Feed Pot, L-104 and L-154</td>
<td>83</td>
</tr>
<tr>
<td>13</td>
<td>Supernate Tank, L-105</td>
<td>85</td>
</tr>
<tr>
<td>14</td>
<td>Centrifuge Feed Tank, L-106 and L-156</td>
<td>87</td>
</tr>
<tr>
<td>15</td>
<td>Product Cup</td>
<td>89</td>
</tr>
<tr>
<td>16</td>
<td>Centrifuge Plan, L-400 and L-450</td>
<td>91</td>
</tr>
<tr>
<td>17</td>
<td>Centrifuge Section L-400 and L-450</td>
<td>93</td>
</tr>
<tr>
<td>18</td>
<td>Off-Gas Flowsheet</td>
<td>95</td>
</tr>
<tr>
<td>19</td>
<td>Mechanical Manipulator</td>
<td>97</td>
</tr>
<tr>
<td>20</td>
<td>Manipulator Sub-Assembly</td>
<td>99</td>
</tr>
<tr>
<td>21</td>
<td>Platform Elevator</td>
<td>101</td>
</tr>
<tr>
<td>22</td>
<td>Dissolver Slide Valve</td>
<td>103</td>
</tr>
<tr>
<td>23</td>
<td>Drying Station Positioner</td>
<td>105</td>
</tr>
<tr>
<td>24</td>
<td>Carrier-Charger Plan</td>
<td>107</td>
</tr>
<tr>
<td>25</td>
<td>Carrier-Charger Sections</td>
<td>109</td>
</tr>
<tr>
<td>26</td>
<td>Shipping Cask</td>
<td>111</td>
</tr>
<tr>
<td>27</td>
<td>Shipping Cask Enclosure</td>
<td>113</td>
</tr>
<tr>
<td>28</td>
<td>Photoneutron Counter Block</td>
<td>115</td>
</tr>
<tr>
<td>29</td>
<td>Chemical Flowsheet</td>
<td>119</td>
</tr>
<tr>
<td>30</td>
<td>Flowsheet - Dissolution</td>
<td>121</td>
</tr>
<tr>
<td>31</td>
<td>Flowsheet - Centrifugation</td>
<td>123</td>
</tr>
<tr>
<td>32</td>
<td>Flowsheet - Product Drying</td>
<td>125</td>
</tr>
<tr>
<td>33</td>
<td>Flowsheet - Waste Handling</td>
<td>127</td>
</tr>
</tbody>
</table>
I. INTRODUCTION

This report presents data obtained during the successful startup operation of a new RaLa facility at the Idaho Chemical Processing Plant designed to separate from short-cooled MTR fuel approximately 25,000-curie batches of barium-140. The startup program included use of both unirradiated and irradiated MTR fuel. Major effort in the startup program was devoted to locating and correcting equipment deficiencies.

The RaLa facility described herein was designed and built to supplant an outmoded facility at Oak Ridge National Laboratory which was operated from 1945 to 1956 for the production of barium-140. Facility design was performed by Blaw Knox Company and construction by J. F. Pritchard Company.

This report covers the period November 1, 1955, to June 30, 1957, which includes the entire startup program from plant takeover to successful high activity level production.

The term "RaLa" is an abbreviation for Radioactive Lanthanum-140 which is the decay product of barium-140. RaLa as used in this report refers to all phases of barium-140 production from development to actual production facility operation.

The startup program reported herein was considered to be the final development stage of the RaLa process with the ICPP Technical Branch being directly responsible for the program.

RaLa equipment, except for off-gas handling, is installed in cell L of Idaho Chemical Processing Plant. An independent dissolver off-gas system was provided for the RaLa plant after the program described herein was completed.

II. SUMMARY AND CONCLUSIONS

Operation involving both active and inactive material was conducted in production equipment operating at full scale. Successful operating techniques developed during operation involving only inactive material were applied and refined during the subsequent operation involving irradiated material. Production of kilocurie quantities of barium-140 was successfully accomplished although product recovery efficiency was generally not as good as that obtained during the pilot plant development program\(^1\). Average product recovery efficiency during irradiated material operation was 62 percent compared to 62 percent during unirradiated material operation and 74 percent during pilot plant operation.

A brief process description follows:

1. One MTR assembly is dissolved in five molar sodium hydroxide under temperature-controlled conditions. Aluminum, cesium, and tellurium are selectively dissolved leaving uranium and other fission products as insoluble particulates in suspension.

2. Dissolver solution is centrifuged to separate aluminate supernate from particulates.

3. Particulates, except silica, are dissolved in dilute nitric acid. Centrifugation is employed to separate dissolved uranium, barium, and fission products from silica and other insoluble particulates.

4. Silica is slurried with water and transferred to a waste tank.

5. After addition of inactive barium carrier, the supernate from step (3) is concentrated by evaporation to initiate barium precipitation which is completed by the addition of fuming nitric acid.

6. Nitrate solution is centrifuged to separate uranium and fission product supernate from barium and strontium precipitates.

7. Barium and strontium precipitates are dissolved in water and converted to chromates upon addition of sodium dichromate. The solution is buffered to a pH of 4.25±0.25 to precipitate selectively barium chromate.

8. Chromate solution is centrifuged to remove strontium solution from barium precipitate.

9. Barium is dissolves in dilute nitric acid; inactive strontium is added to the acidified barium solution to improve active strontium decontamination. After addition of sodium dichromate the solution again is buffered to precipitate selectively barium chromate.

10. Chromate solution is centrifuged to remove strontium solution from barium precipitate.

11. Metathesis of barium to the nitrate form is effected by addition of fuming nitric acid. Excess acid is removed by centrifugation.

12. Barium nitrate is dissolved in water and re-precipitated upon addition of fuming nitric acid to remove impurities.

13. Nitrate solution is centrifuged to remove final impurities from barium nitrate precipitate.
Barium nitrate is dissolved in water and transferred to a drying cup where water is evaporated, leaving dry barium nitrate product.

The startup program demonstrated that the processing of short-cooled MTR fuel to produce 25,000-curie batches of barium-140 can be conducted adequately with no deleterious effects of radiation on the chemical flow-sheet. Exposure of operating personnel to radiation did not exceed the maximum prescribed limit. Product of acceptable activity and purity was produced.

Development of the RaLa process is considered complete. However, process improvements are expected to be made as long as the process operates.

The startup program was aimed principally at making equipment operable and at developing operating techniques compatible with the equipment and radiation levels involved. The major findings as a result of this program are:

1. Plant turnover date is not necessarily indicative of plant startup date.

2. Complete checkout of all equipment is absolutely necessary prior to startup operation.

3. Equipment cleanout following construction is essential to remove foreign matter which otherwise would plug equipment.

4. It is impractical to measure the progress of evaporation and drying operations by collecting and measuring condensate where small volumes are concerned. Vapor loss in the condenser vent line prevents good volume balances.

5. Probe type instruments are satisfactory for determining volumes in vessels where evaporation is being conducted.

6. Centrifuge cake cutting was performed satisfactorily using a high pressure solution spray.

7. Unskimmable residual holdup volume in the centrifuge was less than 30 milliliters.

8. The remote centrifuge skimmers operated satisfactorily when due care was exercised in operation.

9. Control of pH during the chromate precipitation steps is reproducible within necessary limits.

10. The multitude of sequential operating steps accentuates the critical need for constant operator alertness and attention.
III. PROCESS HISTORY

A. ORNL Experience

Kilocurie amounts of barium-140 were produced in the Oak Ridge National Laboratory RaLa plant between 1945 and 1956. Unreliable equipment made exigent the construction of a new RaLa facility(3).

B. Hanford Experience

A RaLa plant was designed and construction started in 1949. The project was terminated early in 1950 when construction was only 5 percent complete.

C. American Cyanamid Company

In October, 1952, American Cyanamid began development studies for a RaLa facility to be located at the Idaho Chemical Processing Plant.

D. Phillips Petroleum Company

Upon termination of American Cyanamid's contract with the AEC, Phillips Petroleum continued the studies instituted by Cyanamid. These studies resulted in a "chromate" flowsheet(2) which was demonstrated to be adequate in pilot plant operation(1). Following construction of the new RaLa facility, Phillips took over the facility on November 1, 1955, to initiate the startup program which is described in this report.

E. Blaw Knox Company

Design of the RaLa facility described herein was initiated in July, 1954, by Blaw Knox Company, AEC architect-engineer contractor.

F. J. F. Pritchard Company

Construction of the RaLa facility was begun in November, 1954, and was essentially complete by November, 1955.

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(3) R. E. Blanco; Laboratory Development of the MTR-RaLa Process for the Production of Barium-140, ORNL-1148, August 19, 1952.
IV. PROCESS DESCRIPTION

A. Chemical Flowsheet

1. Starting Material

A single spent MTR fuel assembly constitutes the charge material for each RaLa run. It was originally estimated that each assembly would be irradiated only twenty days and cooled two days prior to processing. From a practical standpoint it is not advisable to locate a new fuel element in the position of highest neutron flux in the MTR due to adverse effects on flux distribution. It is therefore necessary to use elements which have been through several MTR operating cycles. In this manner it is possible to obtain elements containing 50,000 to 60,000 curies of barium-140 at final reactor discharge without adversely disturbing the reactor flux distribution. This procedure provides an element with more uranium burnup (and thus more fission products) than the theoretical one-cycle element originally envisioned. However, no deleterious processing effects due to the increased fission products were detectable during the startup program. See Table 6 for a description of the actual elements used in startup operation.

2. Desired Product

Product specifications were rather arbitrarily set by the customer. Following the RaLa development program, customer requirements for barium-140 activity per batch of delivered product were reduced from the original 30,000 - 50,000 curie range to approximately 25,000 curies. The original specification of five grams total barium was relaxed to allow up to eight grams to provide better recovery efficiency by the addition of more inactive barium carrier. Specifications for maximum allowable contaminants remained unchanged. Product specifications per batch are summarized as follows:

a. Barium

(1) Barium-140: 25,000 curies
(2) Total: up to 8.0 grams total barium

b. Contaminants

(1) Strontium: 10 curies maximum
(2) Total metallic impurities: maximum of 50 milligrams soluble in 75 milliliters of 70 percent nitric acid (lanthanum-140 and cerium-140 excepted).

3. Chemical Flowsheet Description

Only minor changes were made in the chemical flowsheet following the pilot plant program. Sodium bromide was eliminated completely in the chromate
steps to minimize corrosion. The final nitrate boildown volume was changed from 0.9 liter to 1.5 liter to allow better operational control. The decreased nitrate concentration caused by this increased volume was offset by increasing the volume of fuming nitric acid precipitant added from 0.5 liter to 1.0 liter. Other minor flowsheet changes involved reagent and wash volumes. A detailed flowsheet is given in Figure 29, Appendix A.

A simplified chemical flowsheet is given in Figure 1. Processing may be thought of as occurring in several definite steps as follows:

a. **Step No. 1 - Fuel Assembly Dissolution**

   The RaLa process begins with batch dissolution of an MTR assembly in aqueous 5M sodium hydroxide. Dissolution control is attempted by controlling the dissolution temperature in order to provide a fairly even evolution of hydrogen off-gas. This off-gas passes through a caustic scrubber for iodine removal. Aluminum, cesium, and tellurium are actually dissolved in the caustic solution while silica, uranium, barium and other fission products remain undissolved in the form of suspended particles. Radioactive xenon is liberated as a gas and passes to the dissolver off-gas system.

b. **Step No. 2 - Dissolver Solution Centrifugation**

   Dissolver solution is jetted through a flow control device (constant head orifice) to the centrifuge which effects separation of solution from solids. Aluminate supernate containing cesium, tellurium and excess caustic overflows the centrifuge bowl to a waste tank. Particulates remain as a cake in the centrifuge bowl where they are washed with additional caustic solution and water.

c. **Step No. 3 - Fission Product Dissolution**

   The cake is dissolved in 6M nitric acid to remove uranium, barium and other fission products from insoluble silica which remains in the centrifuge bowl. Silica is subsequently slurried with water and transferred to a waste tank.

d. **Step No. 4. - Nitrate Precipitation**

   The nitric acid solution obtained as supernate in the previous step is transferred to a boildown tank. Inactive barium carrier is added to minimize loss of active barium due to solubility. Evaporation initiates the precipitation of barium and strontium nitrate due to increased concentration of these salts. Concentrate resulting from evaporation is transferred back to the
centrifuge where fuming nitric acid is added to complete the precipitation of barium and strontium nitrate. Centrifugation is then employed to separate uranium supernate from barium and strontium nitrate cake which remains in the centrifuge bowl. Uranium supernate is transferred to a storage tank for subsequent charging to the ICPP uranium recovery process.

e. Step No. 5 - First Chromate Precipitation

Nitrate cake from the previous step is dissolved in water. Sodium dichromate is added to effect conversion to barium and strontium chromate. Sodium acetate buffer is added to adjust the acidity to pH 4.0 - 4.5. At this condition barium chromate precipitates but strontium nitrate does not. Centrifugation is then employed to remove strontium supernate from barium chromate cake which remains in the centrifuge bowl. Strontium supernate is transferred to storage for future strontium recovery.

f. Step No. 6 - Second Chromate Precipitation

Chromate cake from the previous step is dissolved in 0.2M nitric acid. Sodium dichromate is again added to convert nitrate salts to chromates. Inactive strontium is added along with the dichromate to improve the active strontium decontamination factor. Sodium acetate buffer is added to adjust the acidity as before so that barium chromate will be selectively precipitated. Centrifugation is employed to separate strontium supernate from barium chromate cake which remains in the centrifuge bowl. Strontium supernate is transferred to waste.

g. Step No. 7 - Metathesis

Barium chromate precipitate is converted to barium nitrate by the addition of fuming nitric acid. Excess acid is transferred to waste following centrifugation. The cake is not washed as it is following other centrifugation steps.

h. Step No. 8 - Final Nitrate Precipitation

Barium nitrate is dissolved in water and re-precipitated upon the addition of fuming nitric acid. Centrifugation removes excess acid which is transferred to waste while barium nitrate cake remains in the centrifuge bowl.

i. Step No. 9 - Product Drying

Barium nitrate is dissolved in water and transferred to a product cup where evaporation to dryness is performed. The final product is dried barium nitrate which is shipped to the customer in a special shipping cask.
B. Engineering Flowsheet

1. Major Processing Equipment

A simplified schematic sketch of major processing equipment is given in Figure 2. Processing begins in the dissolver. Off-gas from the dissolver flows through a packed column where it is scrubbed countercurrently with 1M sodium hydroxide solution containing 0.10M sodium thiosulfate. Dissolver solution is transferred to a feed pot by means of a high pressure air jet. The feed pot contains an internal constant head orifice through which dissolver solution flows by gravity to the centrifuge which is equipped with two skimmers for supernate removal. One skimmer routes skimmed solution to the drying station and the other to the supernate tank. The supernate tank is provided with jets for transferring solutions to waste, storage, or back to the process via the centrifuge feed tank.

A special carrier-charger is used to transport and charge the MTR assembly used as charging material. A special shipping cask is used to transport the product cup. The shipping cask is handled inside the cell by a cable operated platform elevator and outside the cell by a 15-ton crane. The product cup is handled in the cell by a manually operated mechanical manipulator which operates in a limited vertical plane.

Detailed engineering flowsheets are given in Figures 30, 31, 32, and 33, Appendix A.

2. Design Philosophy

a. Direct Maintenance

As in the ICPP proper there is no provision in the RaLa cell for remote maintenance. Two lines of equipment (feed pot, centrifuge feed tank, and centrifuge) provide for maximum operability and minimum maintenance.

b. Minimum Mechanical Equipment

(1) Pumps. Mechanical pumping is avoided completely within the processing cell. Solution transfer is accomplished by air or steam jetting, air lifting, or by gravity flow.

(2) Valves. Except for the centrifuge feed tanks L-106 and L-156, which contain internal solenoid operated valves, there are no valves in process solution lines. Diaphragm operated valves are used in vapor service.

(3) Agitation. Agitation in cell vessels is accomplished by air sparging. Except in the Process Makeup Area mechanical agitators are avoided completely.
FIGURE 1

SIMPLIFIED CHEMICAL FLOWSHEET
FIGURE 2

SIMPLIFIED FLOW DIAGRAM

SCRUBBER

DISSOLVER

OFF-GAS

VENT

Solenoid

CENTRIFUGE

FEED TANK

AIR

JET

FEED POT

DRIED STATION

CENTRIFUGE

Uranium Storage

Strontium Storage

Waste Solutions

Supernate Tank
c. **Chemical Addition**

The addition of chemicals to the process is accomplished by the use of pressurized addition funnels (pressure pots) to provide positive flow from control to processing areas.

d. **Visual Observation**

A lead glass window is provided to facilitate manipulation of the product cup which must be moved from shipping cask to drying station to product testing station and back to the shipping cask.

e. **Separation of Liquids from Solids**

Two centrifuges (one of which is an installed spare) are provided for separating liquids from solids in the process.

V. **STARTUP PROGRAM**

A. **Line Checking**

After takeover, the piping was checked against Blaw-Knox drawings for routing accuracy and for material of construction. Each section of pipe and each weld was checked with a magnet and with nitric acid to insure the presence of stainless steel. No deficiencies were found in material of construction. This phase of the startup program was complete by November 10, 1955.

B. **Vessel Calibration**

1. **Instrument Checking**

All instruments were checked for operability. In several cases it was found that instrument probes to certain vessels were improperly connected. Corrections were made where necessary and all instruments were adjusted for optimum operability.

2. **Vessel Flush**

Prior to calibration vessels generally were flushed with nitric acid to remove scale and other foreign matter which might have caused jets and lines to become plugged during subsequent calibration.

3. **Vessel Calibration Using Water**

Volume calibration data were obtained for all tanks essential to the process. Where possible, tanks were first completely emptied by use of
a vacuum pump. On accessible tanks, at least one nozzle had been left open by the construction contractor. This nozzle was used for insertion of a suction line for the vacuum pump. Following tank cleanout, incremental additions of water were made to obtain calibration curve data. A micromanometer attached to vessel level and gravity probes was used for optimum accuracy in the calibrations. Raw calibration data were converted into finished calibration charts and nomographs by the CPP Operations Evaluation Section.

Calibration was complete by December 31, 1955.

C. Correction of Deficiencies

A major portion of the time required for the startup program was devoted to location and correction of equipment deficiencies. Over one hundred separate items required revision or replacement. These ranged from crossed vessel probes to replacement of the platform elevator actuating mechanism. Deficiency correction was completed by December 5, 1956. Details of the major deficiencies follow.

1. Vessels and Other Processing Equipment

a. Caustic Makeup Tank, PM-120-0

The mechanical agitator shaft on this vessel was not long enough to reach makeup solutions for the dissolver, L-101. A new shaft was installed.

b. Dissolver, L-101

As originally installed, the dissolver sparger caused dissolver solution to be air lifted out of the dissolver through a feed pot return line. The condition was corrected by installing a separate sparge line such that sparge nitrogen could not become trapped in other dissolver probes.

c. Centrifuge Feed Tank No. 2, L-156

The solenoid operated valve in this tank became stuck in the open position. Disassembly indicated inadequate clearance between valve stem and guide hole. The tank had also been installed one degree out of the true vertical alignment which further contributed to the sticking valve. The tank was realigned and additional guide hole clearance was provided.

d. Feed Tank Condenser No. 2, L-256

A leak in the low pressure density line at the instrument transmitter caused faulty gravity readings. The leak was repaired.
e. **Centrifuge Condenser No. 1, L-300**

A leak in the common high pressure instrument probe line caused faulty level and gravity readings. The leak was determined to be in the wall sleeve section of the line. A spare wall sleeve was used to correct the condition.

f. **PEW Sampling Tank, L-110**

Originally, water discharging from the hydraulic cylinders at the drying and recycle stations was piped directly to PEW sampling tank, L-110. This would have kept the tank full and would have nullified its intended use of collecting condensates for sampling. The hydraulic cylinder operating water was re-routed to discharge directly into the cell wall drain.

g. **Air Jets, L-514, L-515 and L-545**

These jets were undersized to the extent that flow was extremely slow and frequent plugging occurred. Throats were enlarged to double the capacity and minimize plugging due to particulates.

h. **Strontium Batch Tank, L-111**

Sparging this tank caused solution loss out the overflow line to L-113. No corrective measures were taken other than to avoid use of the sparger.

i. **Overhead Receiver, L-320**

This vessel is a collecting condenser attached to the dryer head. Originally, inadequate condensing capacity caused excessive vapor loss out the vent line. An additional condensing section was added.


Instrument probes on these vessels were improperly connected as originally installed. Corrections were made.

k. **Pressurized Addition Funnels (Pressure Pots)**

On each of four pressure pots in the Process Makeup Area no valve had been included on the service nozzle. The required valves were added.
2. Electrical and Instrumentation Equipment

a. Cell Lighting

The sodium vapor lights installed in cell L required frequent replacement during the startup program due to high burnout rate. The original lighting consisted of two banks of six sodium vapor lamps each (all on one circuit) plus two banks of three sodium vapor lamps each (all on one circuit). The latter two banks are theoretically replaceable from the vent tunnel. However, this replaceable feature is essentially negated by high vent tunnel contamination and by the extremely tight fit of the lighting fixtures in the wall access hole. To improve the cell lighting system, four banks of white lights containing three lamps each were installed. Each bank was provided with a separate switch. Further, the sodium vapor lamp circuits were revised to provide a switch for each three sodium vapor lamps. It is intended that only a minimum of lighting be used. The white lamps will be used first until burnout. Then the sodium vapor lights will be used until burnout, at which time complete lamp replacement will be required.

b. Intercommunication System

As originally installed the intercommunication system was unsatisfactory due to feedback. The system was replaced.

c. Hydrogen Analyzer

The hydrogen analyzer required frequent filament replacement. Erroneous analyzer readings prevailed during the initial cold runs.

d. Condensate Collection Tank, WF-100, Alarm

As originally installed, the lower float switch actuated an annunciator alarm on WF-100 indicating that one discharge pump was working. The alarm was changed to the upper float switch which starts a second discharge pump.

e. Dissolver Slide Valve Interlock System

The electrical interlocks on the dissolver slide valve actuating mechanism were improperly adjusted as orginally installed. Also, limit switches on the slide valve itself were unsatisfactory and required replacement.

f. Centrifuge Electrical Connection Amphenols

Amphenols installed in the centrifuge electrical leads had mismatched male and female securing devices. The units were replaced with standard amphenols.
g. Centrifuge Jet Position Indicator Lights

Jet "up" and "down" lights were reversed as originally installed. The necessary correction was made.

h. Drying Station Electrical Leads

Originally, the electric lines to the drying station were not encased in conduit but instead were wrapped and painted. Flexible conduit was installed and the conduit was encased in tygon tubing to minimize contamination.

i. Timer on Centrifuge Motor Circuit

Each centrifuge was provided with a timer to allow adequate time for centrifuge oiling before DC power could be applied to the centrifuge motor. Any power outage thus caused the timer to be actuated. To prevent unnecessary delays in centrifuge operation, an electrical bypass and switch was installed around each timer.

j. Dissolver Instrumentation

Originally, no vacuum gauge was provided for the dissolver, L-101. A compound gauge was installed as a further check on dissolver operation.

k. Control Panel

Originally, the control panel did not include a clock. To aid in operation of the facility a clock was installed.

l. Centrifuge Periscope Light

Originally, the light on centrifuge L-450 periscope did not operate. Corrective action was taken.

m. Photoneutron Counter

Originally, there was no provision for inserting a calibration source in the photoneutron counter block. New concrete shielding blocks were fabricated to provide a calibration source access hole.

Further, only one counter instrument had been provided. A second counter was subsequently installed to provide more reliable counting data.
3. **Mechanical Equipment**

a. **Platform Elevator**

The original platform elevator was operated by a 3-inch diameter stainless steel screw shaft working in a manganese bronze nut. Excessive nut wear was experienced. Elevator travel time was excessive, requiring two hours and forty minutes for a round trip. Further, the shaft seized frequently. To provide dependable operation and to eliminate a known maintenance item the shaft and nut were removed and replaced with a nylon-encased cable and stainless steel drum. A special shielded cubicle was installed in the Process Makeup Area to house the cable drum and idler pulley. The original elevator platform and guide beam required no revisions except for alignment.

This elevator revision program required considerable effort for resolution. The revised design was provided by the original architect-engineer and construction work was performed by Arrington Construction Company. Elevator revisions were completed October 8, 1956.

Another elevator problem was caused by an oversized platform centering pin which prevented the shipping pot from resting on the elevator platform. The centering pin was filed to proper size.

b. **Mechanical Manipulator**

The mechanical manipulator as originally installed was unsatisfactory in the following respects:

1. Pins in the connectors on the operating rods were inadequately secured.
2. The spring-loaded lid in the cup tool pickup did not contact the cup with the cup in the secured position.
3. Set screws in the operating mechanism were not countersunk and consequently allowed slippage.
4. Manual operation was extremely difficult due to binding and excessive friction.
5. Gears on cantilever operating rods did not mesh properly resulting in considerable wear and difficult operation.
6. A sliding bearing on the horizontal guide rod caused excessive friction during horizontal movement of the manipulator.
(7) Alignment of the manipulator, guide rods, and operating rods was unsatisfactory.

(8) No lubrication provision was incorporated in the original design.

A considerable amount of maintenance time was spent in making manipulator operation dependable. Major overhauls proved short lived until a system was installed to provide intermittent lubrication to all working parts. Correction of the deficiencies listed above was completed by Phillips Maintenance on November 23, 1956.

c. Drying Station Positioner

The drying station positioner suffered a parted connection because set screws were not countersunk at connector locations. Corrective action was taken.

d. Product Testing Station

The hole in the product testing station was originally too small to receive a product cup. The hole was enlarged.

e. Cup Tool Adapter Station

As built, there was no provision for storing the cup tool adapter which is used for handling the cup plug. A special station was installed to correct this omission.

f. All Product Cup Stations

At none of the product cup stations was any provision made for securing the cup against rotation during manipulation. Consequently, each station was provided with a locking ear to match the slot in the cup rim.

g. Drying and Recycle Station

The bottom stop on both drying and recycle station vertical guide posts caused the product cup to contact probes extending down from dryer and recycle head. The situation was corrected by removing the upper portion of the mechanical stop bar on both drying and recycle station vertical guide posts.

Additional trouble was caused by the drying station binding on its vertical guide post. Binding was eliminated by installing roller bearings in place of a sliding friction contact.
h. **Dissolver Slide Valve**

A mechanical interlock on the slide valve did not operate satisfactorily. Part replacement and realignment was necessary.

i. **Carrier - Charger**

A locating hole in the carrier-charger frame was too small to receive the locating dowel pin at the slide valve station. The hole was enlarged.

Hose adapter fittings had to be provided on the carrier-charger coolant connections.

No special lifting yoke had been provided for the carrier-charger. The required yoke was fabricated.

j. **Shipping Pot Handling**

Adequate lifting bails had not been provided for handling the shipping pot. A new yoke was fabricated containing removable cables and hooks for maximum operating flexibility.

4. **Centrifuge Equipment**

a. **Lubrication**

The original lubrication system consisted of an oil feed tank in the Process Makeup Area connected to Clarkson feeders which transferred oil to funnels. The oil funnels were connected to oil lines leading to the centrifuges. It was found that in cold weather the oil could not be transferred by the Clarkson feeders due to excessive oil viscosity. Further, when oil did flow, excessive time was required for it to reach the centrifuges due to the long length of small (1/8-inch pipe) feed line becoming air locked. To provide adequate lubrication, the Clarkson feeders in the Process Makeup Area were replaced with sight feeders at the access corridor level. This eliminated air locking because a constant head of oil was maintained from the PM area oil feed tank to the access corridor sight feeders.

b. **Drive Buttons**

A modified universal joint transfers power from the direct current motor shaft to the centrifuge bowl shaft. The bowl is of the suspended type to provide for variable center of rotation under variable loading conditions. In the universal joint assembly contact between drive and driven parts was originally made by a rounded drive button which contacted a flat driven surface.
Original drive buttons were constructed of stainless steel. It was established that excessive friction developed between button head and driven surface. The problem was solved, after a great deal of experimenting with various button materials, by using hardened steel buttons on both driver and driven parts. Furthermore, it was necessary to provide lubrication for the buttons to minimize friction and eliminate undesirable bowl sway.

c. **Skimmer Kidneys**

The original skimmer kidneys were undersized to the extent that skimming caused flooding of the kidneys.Flooding resulted in solution loss due to leakage onto the centrifuge top plate and also due to solution being forced into the kidney vent line. This problem, probably the most serious of all the centrifuge problems, was referred to Bird Machine Company, fabricator of the centrifuges. New and larger kidneys were built and installed. The new kidneys were of sufficient volume to hold all solution from any skimming operation. The problem thus was solved by installing a larger surge volume in the skimmer discharge system. This revision required six months to complete.

d. **Skimmer Operating Levers**

Operating levers were originally inadequately attached with set screws. It was necessary to spot weld connections. Furthermore, considerable friction in the entire skimmer lever mechanism precluded "feel" in the skimming operation. Additional bearings were installed to minimize friction and provide more operating "feel". A scale and pointer arrangement was installed on each skimmer operating lever to provide a means of locating the skimmer pickup nozzle with respect to the centrifuge bowl wall.

e. **Centrifuge Feed Manifolds**

As originally installed the centrifuge feed manifold piping contained two 1/8-inch and one 1/4-inch pipe inlets and one outlet of 1/8-inch pipe. The 1/4-inch inlet handled dissolver solution and provided a 20-foot head of solution on the manifold. The small manifold outlet caused solution to back up into the other two manifold inlet lines. To correct this situation the 1/4-inch line was disconnected from the manifold and routed separately through the centrifuge top plate. This required drilling an extra hole in each top plate and welding a new nozzle in place.
f. **Centrifuge Shaft Oil Seal**

As originally installed, the lower shaft bearing oil seal on centrifuge L-450 was not leak tight. Thus it was possible for oil to leak down the shaft and eventually contaminate product solution in the centrifuge bowl. The situation was corrected by refacing metal surfaces where necessary to provide better mating of seal components. As further protection against product contamination a slinger ring was installed on the shaft just below the lower shaft bearing housing. A stationary collector trough was built around the slinger ring for oil collection. These revisions were made on both centrifuges.

g. **Centrifuge Shaft Seals**

Floating carbon ring seals were originally provided to effect a seal between the centrifuge shaft and the shaft hole in the centrifuge top plate. It was found that use of these seals caused carbon to be ground from the seals due to intimate contact with the rotating shaft. The carbon particles so produced fell into the centrifuge bowl and resulted in product contamination. Replacement of carbon with tygon ring seals did not eliminate product contamination so the shaft seals were completely removed. Absence of seals resulted in considerable external cell contamination but this was selected as the lesser of two evils since the seals were rather inefficient at best.

h. **Flexible Oil Lines**

The flexible oil discharge line from each centrifuge was considered to be a potential decontamination problem. Consequently, these lines were encased in tygon tubing to minimize exposed surface area.

i. **Shaft Sway**

The suspended type centrifuge is quite sensitive to any unbalancing forces. During the startup program considerable shaft sway was experienced at times. A Bird Machine Company representative visited the plant several times in connection with the problem of shaft sway. In January, 1956, a bent shaft resulted from excessive sway which caused the rotating bowl in centrifuge L-450 to contact the stationary case. Exact cause of the excessive sway is unknown. It was necessary to send the bent shaft to the Bird Machine Company plant for straightening. The bowl and shaft from the spare centrifuge unit was installed in centrifuge L-450. Installation of lubricated drive buttons as described in section 4b minimized shaft sway.
j. **Centrifuge Motor Can**

Four-inch air lines to and from each centrifuge motor enclosure were originally welded without provision for rapid disassembly. It thus was necessary to use a hack saw on two four-inch pipes before motor cans could be removed. To minimize disassembly time, each air line was provided with flanges near the motor can. A large rag was found stuffed in the four-inch air outlet line from centrifuge L-450 motor can. Apparently the rag had been used to seal off the pipe when a final weld was made by construction forces.

k. **Centrifuge Microphones**

A microphone attached to each centrifuge was expected to be an aid in skimming. However, in practice, the skimming noise picked up by the microphone was practically nil. Instead, mechanical noises were transmitted. No corrections were attempted. The microphones are useful to some extent in determining the mechanical condition of bearings and other working parts.

5. **Off-Gas System**

a. **Off-Gas Processing**

A caustic scrubber was the only equipment originally installed in the RaLa cell for processing radioactive gas. The assumption was made by the architect-engineer that any active gases passing through the scrubber could either be stored in existing gas holders or vented directly to the stack. Later developments indicated that this assumption was not good. Hydrogen-containing gas cannot be handled in existing gas holders due to the attendant explosion hazard and iodine cannot be released indiscriminately to the stack. A special study conducted during the startup program resulted in the recommendation that a gas holder be installed to store diluted RaLa gas for release under favorable meteorological conditions. AEC approval was granted for a 10,000-cubic foot capacity gas holder. Installation was completed in July, 1957.

Actual processing during the startup program indicated that the gas holder would not be adequate for containing all active gases released as a result of processing because iodine was released over a period of days following each run rather than over a period of hours during each run. Experiments conducted on actual off-gas samples indicated the desirability of installing carbon beds in the off-gas line between the existing
scrubber and the new off-gas holder. A proposal to the AEC for such beds is pending. The off-gas system was thus incomplete during the startup program.

b. Off-Gas Rotameter

As originally installed, the off-gas rotameter was not provided with a bypass. Any plugging within the rotameter thus restricted the off-gas flow and caused the dissolver to become pressurized. Just prior to Run No. 001-RP the cell was decontaminated sufficiently for installation of a rotameter bypass containing automatic valves. The bypass valves open when dissolver pressure exceeds two inches of water vacuum.

c. Off-Gas Monitoring

The Raka facility originally contained no provision for monitoring radioactive gases being discharged to the stack. Two monitors were subsequently installed. One monitor is a scintillation type counter attached to the hydrogen analyzer sample line. The other monitor system consists of a monitored caustic scrubber through which a very small fraction of the stack gas passes. These monitors provided information on the time rate of activity release during and after processing.

6. Miscellaneous

a. Cell Window

Oil in the cell window became cloudy during the startup program. The oil was drained, dried, and recharged to the window. Original clarity was restored.

Originally there was no provision for cleaning the inside surface of the window. To alleviate this condition a water spray pipe and windshield wiper were installed for remote operation.

b. Decontamination System

The original decontamination system was inadequate in that no sprays were located in the vicinity of the drying station-centrifuge area. Additional sprays were installed to cover that area.

c. Remote Service Valves

Service valves which are operated remotely from the control panel were found to leak in varying degrees. This was especially
true of those in jet air service. Replacement of valve seats did not increase reliability significantly. It was necessary to use hand valves in conjunction with the automatic valves. This, of course, negated the remote operation feature of the automatic valves, but the additional insurance against leakage and operational errors was considered necessary.

d. **Cell Ceiling Hatch Cover**

A shielding block for the cell ceiling hatch was found to be too large for the hatchway. It was necessary to remove the metal lining on two sides of the block before proper fit was obtained.

D. **Operation**

Operation involving unirradiated material was conducted during the period April 25 to November 23, 1956. Operation involving irradiated material covered the period November 24, 1956 to June 30, 1957, at which time the process development was considered complete.

Operation is discussed in more detail under Discussion of Results. Detailed operating Instructions are given in Appendix E.

E. **Cost**

Startup operation by Phillips involved considerable cost due to the large number of equipment deficiencies which required correction. This cost was about $400,000.

A summary of some of the costs estimated for the facility and its operation is given in Table 1. These costs are only approximate and do not include all factors which may be involved in recovery costs.

**Table 1**

<table>
<thead>
<tr>
<th>Description</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell L (allocated portion of original ICPF cost)</td>
<td>$2,000,000</td>
</tr>
<tr>
<td>Design</td>
<td>$366,000</td>
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<tr>
<td>Construction</td>
<td>$1,621,000</td>
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<tr>
<td>Startup Tests and Modifications</td>
<td>$400,000</td>
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<tr>
<td>Maintenance per Year (estimated)</td>
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<tr>
<td>Utilities, per batch processed</td>
<td>$500</td>
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<tr>
<td>Waste Storage, per batch processed</td>
<td>$1,500</td>
</tr>
</tbody>
</table>
VI. DISCUSSION OF RESULTS

A. Operation Involving Non-Radioactive Material

1. General

The initial startup operation prior to the processing of active material involved fifteen separate runs using unirradiated MTR fuel. In each run a tracer spike solution of barium and strontium was added to the process just after the dissolver solution centrifugation step. Operation was conducted concurrently with maintenance work in the cell.

These runs accomplished several objectives as follows:

a. Locate equipment deficiencies.

b. Demonstrate operability of equipment.

c. Demonstrate compatibility of flowsheet and plant equipment.

d. Develop necessary operating techniques for reliable operation with minimum chance for errors.

2. Sampling

A fairly complete sampling program was followed in Runs 1-9 since open centrifuge skimmer discharge lines permitted sampling of individual skimmed supernates. Following Run 9, extensive sampling was not possible because all centrifuge piping connections were welded. Barium and strontium tracer level was insufficient to provide meaningful radiochemical analyses of composite waste streams.

Except for aluminate supernate, all supernates in Runs 1-9 were collected in graduated cylinders for volume measurement and sampling. In subsequent runs sampling was performed using the plant remote samplers.

3. Operation

Remote operation was followed in Runs 10-15 except for manipulation of the product container. The mechanical manipulator was not made operable until just after Run 15.

Pertinent operational data are given in Table 2 and in Appendix B.

Process operation is rather tedious and requires close attention to details. Operating time in these runs averaged 24 hours which includes neither assembly nor product handling. Considerable operating technique
TABLE 2

DATA FOR RUNS INVOLVING UNIRRADIATED MATERIAL

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Type Element* Charged</th>
<th>Run Date</th>
<th>Centrifuge Used</th>
</tr>
</thead>
<tbody>
<tr>
<td>001-RC</td>
<td>A</td>
<td>Apr. 25-27, 1956</td>
<td>L-450</td>
</tr>
<tr>
<td>002-RC</td>
<td>A</td>
<td>May 9-11, 1956</td>
<td>L-450</td>
</tr>
<tr>
<td>003-RC</td>
<td>A</td>
<td>June 6-8, 1956</td>
<td>L-450</td>
</tr>
<tr>
<td>004-RC</td>
<td>A</td>
<td>June 20-22, 1956</td>
<td>L-450</td>
</tr>
<tr>
<td>005-RC</td>
<td>A</td>
<td>July 5-6, 1956</td>
<td>L-450</td>
</tr>
<tr>
<td>006-RC</td>
<td>A</td>
<td>July 11-13, 1956</td>
<td>L-450</td>
</tr>
<tr>
<td>007-RC</td>
<td>A</td>
<td>Sept. 5-7, 1956</td>
<td>L-400</td>
</tr>
<tr>
<td>008-RC</td>
<td>B</td>
<td>Sept. 11-13, 1956</td>
<td>L-400</td>
</tr>
<tr>
<td>009-RC</td>
<td>B</td>
<td>Oct. 3-5, 1956</td>
<td>L-400</td>
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<tr>
<td>010-RC</td>
<td>C</td>
<td>Nov. 5-6, 1956</td>
<td>L-450</td>
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<tr>
<td>011-RC</td>
<td>C</td>
<td>Nov. 8-9, 1956</td>
<td>L-400</td>
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<tr>
<td>012-RC</td>
<td>C</td>
<td>Nov. 11-12, 1956</td>
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<tr>
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<td>C</td>
<td>Nov. 13-14, 1956</td>
<td>L-450</td>
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<tr>
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<td>C</td>
<td>Nov. 15-16, 1956</td>
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</tr>
<tr>
<td>015-RC</td>
<td>C</td>
<td>Nov. 21-22, 1956</td>
<td>L-450</td>
</tr>
</tbody>
</table>

*Element Type

A - Containing 140 g. normal uranium.
B - Containing 215 g. depleted uranium.
C - Containing 200 g. enriched uranium.
development was required for proper control during nitrate boildown and product drying. Operation of the pressurized addition funnels requires close attention to valve operating sequence. Centrifuge skimming was performed adequately.

4. Material Balances

Major component material balances for these runs are given in Table 3. Barium balances average well under 100 percent while balances for both strontium and uranium average over 100 percent. Barium and strontium analyses were performed radiochemically and were based on known quantities of tracer added in each run. The average barium balance is 85.7 percent which is exactly the same average balance obtained in pilot plant runs.

5. Product

a. Recovery

To produce the required quantities of active barium for customer consumption, recovery of barium in RaLa processing must usually exceed 50 percent. Recovery averaged 61.5 percent in these runs and ranged from a low of 18.8 percent in the first run to a high of 95.2 percent in a later run.

b. Purity

Product specifications were rather arbitrarily set by the customer. Up to 50 mg. of total metallic contaminants soluble in 75 ml. of 70 percent nitric acid and up to 10 curies of total strontium are allowed in each product batch. This specification generally was not met although it was approached in several runs. A test batch of product from Run 12 was sent to the customer. This batch was considerably off-color and contained particulates which were insoluble in both nitric and hydrofluoric acid but soluble in hot concentrated hydrochloric acid. The customer expressed limited satisfaction with this test batch so no extensive program was developed to improve product purity. However, purity probably could be improved by increasing the number of barium nitrate re-precipitations prior to product drying.

Product composition is summarized in Table 4.

Dried product from Run 15 was washed with 75 ml of 70 percent nitric acid. Contaminants analyzed in the acid leachate are given in Table 5.
<table>
<thead>
<tr>
<th>Run No.</th>
<th>Aluminate Supernate</th>
<th>Uranium Supernate</th>
<th>First Chromate Supernate</th>
<th>Second Chromate Supernate</th>
<th>Waste Nitrate Supernate</th>
<th>Product</th>
<th>Condensate Losses</th>
<th>Total Accounted for, Percent</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>U</td>
<td>Ba</td>
<td>Sr</td>
<td>U</td>
<td>Ba</td>
<td>Sr</td>
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<td>1</td>
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<td>42.8</td>
<td>236.0</td>
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<td>17.8</td>
<td>2.1</td>
<td>2.3</td>
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<td>4</td>
<td>6.7</td>
<td>26.6</td>
<td>26.8</td>
<td>89.8</td>
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<td>55.3</td>
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<td>5</td>
<td>12.0</td>
<td>9.0</td>
<td>23.9</td>
<td>113.0</td>
<td>4.1</td>
<td>96.6</td>
<td>0.5</td>
<td>1.7</td>
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<tr>
<td>6</td>
<td>3.5</td>
<td>2.9</td>
<td>24.9</td>
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<tr>
<td>Run No.</td>
<td>Barium, Grams</td>
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<td>Cr</td>
<td>Cu</td>
<td>Fe</td>
<td>La</td>
<td>Na</td>
<td>Ni</td>
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<td>23</td>
<td>&lt; 10</td>
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<td>9</td>
<td>&lt; 3</td>
<td>39</td>
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</table>
TABLE 5
CONTAMINANTS IN 75 ml. OF 70 PERCENT HNO₃ LEACHATE FROM RUN 15 PRODUCT

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight, mg.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>&lt;2</td>
</tr>
<tr>
<td>Ba</td>
<td>2</td>
</tr>
<tr>
<td>Cr</td>
<td>12</td>
</tr>
<tr>
<td>Cu</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Fe</td>
<td>4</td>
</tr>
<tr>
<td>Na</td>
<td>&lt;2</td>
</tr>
<tr>
<td>Ni</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Sr</td>
<td>&lt;1</td>
</tr>
</tbody>
</table>

c. Losses

Product loss occurs in each waste supernate and in each cake wash stream. Highest average single loss (15.4 percent) occurred in the uranium supernate. In other waste streams total losses averaged under 12 percent. Unaccountable losses averaging 14.3 percent are attributed to spattering during skimming, entrainment, and deposition on pipe and vessel walls. This unaccountable loss is actually not excessive when the small amount of barium involved is considered in the light of the relatively large volume of process vessels and piping through which it passes.

B. Operation Involving Irradiated Material

1. General

The final stage of startup operation involved eight runs using irradiated fuel as charging material. Pertinent fuel element data are given in Table 6. These runs were the first to demonstrate adequacy of the chemical flowsheet under radiation conditions. Centrifuge L-450 was used in all of these runs. Operationally these runs were no different from the final runs involving unirradiated material except for liberation of radioactive gases, heating effects due to fission products, and special handling precautions due to radioactive product. Detailed operating instructions are given in Appendix E.

The objectives accomplished in these runs were:

a. Demonstrate adequacy of chemical flowsheet under radiation conditions.

b. Refine operating techniques to allow for radiation effects.

c. Produce acceptable product for delivery to the customer.
### TABLE 6

DATA FOR RUNS INVOLVING IRRADIATED MATERIAL

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Element No.</th>
<th>Run Dates</th>
<th>Pre-Processing Cooling Time, Days</th>
<th>Total Charged Uranium, grams</th>
<th>Calculated Barium-140 Content @ LST, Curies</th>
<th>Barium-140 Recovered as Product, Curies*</th>
</tr>
</thead>
<tbody>
<tr>
<td>001-RH</td>
<td>I-461</td>
<td>Nov. 24-25, 1956</td>
<td>1138</td>
<td>131</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>002-RH</td>
<td>P-137</td>
<td>Nov. 30-Dec. 1, 1956</td>
<td>27</td>
<td>191</td>
<td>3,033</td>
<td>833</td>
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<tr>
<td>003-RH</td>
<td>M-238</td>
<td>Dec. 6-7, 1956</td>
<td>11</td>
<td>158</td>
<td>16,250</td>
<td>8,081</td>
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<tr>
<td>001-RP</td>
<td>P-126</td>
<td>Feb. 1-3, 1957</td>
<td>5</td>
<td>143</td>
<td>38,800</td>
<td>18,000</td>
</tr>
<tr>
<td>002-RP</td>
<td>P-146</td>
<td>Feb. 20-21, 1957</td>
<td>2</td>
<td>158</td>
<td>43,700</td>
<td>49,000</td>
</tr>
<tr>
<td>003-RP</td>
<td>P-227</td>
<td>Apr. 5-6, 1957</td>
<td>5</td>
<td>192</td>
<td>23,800</td>
<td>18,600</td>
</tr>
<tr>
<td>004-RP</td>
<td>P-237</td>
<td>May 19-20, 1957</td>
<td>7</td>
<td>172</td>
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<td>21,600</td>
</tr>
<tr>
<td>005-RP</td>
<td>P-285</td>
<td>June 24-25, 1957</td>
<td>6</td>
<td>163</td>
<td>32,000</td>
<td>14,500</td>
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</table>

* Reported by customer @ LST (Last Separation Time)
2. **Sampling**

It was originally planned that no samples be taken during the course of a run. This philosophy was followed in the first two of this series of runs. High product losses in these runs made exigent more complete sampling as performed in subsequent runs. Sample results of a particular run have no bearing on the operation of that run because product is obtained before samples can be analyzed. Operating time would be increased several fold if each waste supernate had to be analyzed before proceeding with the next operating step. Sample results are used to detect high barium losses if and when they occur so that future operation at the high loss point may be revised or corrected as necessary.

Since all waste solutions are collected in caustic waste tank No. 2, L-109, this tank is sampled after each separate waste solution is added. Contribution of each waste stream is determined by difference.

Sampling technique for iodine in solution has not been developed to the desired state. In acid solution iodine tends to be stripped by the lift air introduced to the sample suction line which makes sample results erratic and unreliable. This tendency was exhibited to a much lesser degree in caustic solutions.

Sampling for barium required minor development in technique. General ICPP sampling practice requires that vessels be sparged and samplers recirculated for fifteen minutes prior to removal of samples. In Run CO2-RP this period was shown to be inadequate in sampling the uranium supernate. A sample taken after a 15 minute sparging and recirculation period contained barium activity of $3.55 \times 10^{14}$ disintegrations per minute per milliliter. Another sample taken after a one hour period contained barium activity of $3.52 \times 10^{10}$ disintegrations per minute per milliliter (corrected for elapsed time between samples for accurate comparison). As a result, samples for barium analyses are recirculated for one hour where possible. This applies specifically to the final composite aqueous waste, the by-product strontium solution, and the by-product uranium solution.

3. **Material Balances**

Major component material balances for these runs are given in Table 7. Balances for both barium and iodine-131 are more erratic than those for uranium. Barium input is determined from theoretical burnup in the MTR. Barium product activity is used as reported by the customer. Accuracy of the latter is unknown while accuracy of calculated input is of the order of $\pm 15$ percent. In the final analysis, customer satisfaction with delivered product is tantamount to successful operation, regardless of what the theoretical material balance might be. The extremely high barium balance in Run CO2-RP was caused by the high customer reported product activity. The high balance in Run CO5-RP is caused by the high unexplained percentage found in the first decontamination flush. Barium balances differing from 100 percent by 15 to 20 percent are to be expected because of input and product measurement inaccuracies.
### Table 7
MAJOR COMPONENT MATERIAL BALANCES FOR RUNS INVOLVING IRRADIATED MATERIAL

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Aluminate Supernate</th>
<th>Uranium Supernate</th>
<th>First Chromate Supernate</th>
<th>Other Waste Supernates</th>
<th>Scrubber Solution</th>
<th>Condensates</th>
<th>Product</th>
<th>Decontamination Solutions</th>
<th>Balance</th>
</tr>
</thead>
<tbody>
<tr>
<td>001-RH</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>6.4</td>
<td>---</td>
<td>1.0</td>
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<td>002-RH</td>
<td>---</td>
<td>---</td>
<td>4.1</td>
<td>80.8</td>
<td>---</td>
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<td>0.4</td>
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<td>---</td>
<td>3.4</td>
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<td>0.5</td>
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<td>70.8</td>
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<td>0.2</td>
<td>3.6</td>
<td>0.3</td>
<td>162.4</td>
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</table>

*iodine-131
Iodine-131 balances are quite erratic due to inaccuracies in calculated input and uncertainties in sampling. Iodine behaved quite different from expectations prior to processing irradiated material. The tendency of iodine to be stripped from acidic solutions by air resulted in questionable samples. Further, the tendency of iodine to plate out on vessel and pipe walls confounded iodine accountability. Iodine-131 discharge to the atmosphere is the most important aspect of iodine accountability. This phase of iodine detection is described in the "Off-Gas Handling" section of this report.

Uranium accountability typically averaged within ± 10 percent of 100 percent based on the theoretically calculated charge which is subject to error by at least that same margin. Since uranium recovery, per se, is not the goal of the RaLa process, relatively low recoveries (< 95 percent) cause no alarm when the small amount of material and the extremely low production rate are considered.

Strontium balances are not given because of analytical complications in distinguishing strontium-90 in the presence of all the other strontium isotopes.

4. **Product**
   a. **Recovery**

   Barium recovery in these runs averaged 62 percent and ranged from a low of 27.5 percent to a high of 112.0 percent. Recovery is based on the customer reported barium-140 activity compared to calculated barium-140 content of the charged fuel element. Either of these quantities is accurate only to ± 15 percent. Recovery efficiency is given as the "Product" column of Table 7.

   b. **Purity**

   No measure of product purity is possible prior to shipment. The customer reports a relative purity based on the number of product washes required to reduce contaminants to an acceptable level. Contaminants have not been excessive in shipments made to date. Iodine-132 was detected in two batches following the customer's filtration operation. This indicates that tellurium-132 was one of the contaminants present in product. In Run 004-RP product strontium activity was 78 millicuries of which only 4 percent was strontium-90.

   c. **Losses**

   In Runs 001-RH and 002-RH high losses were experienced in waste streams. However, the individual waste stream contributing high barium loss could not be detected because of incomplete sampling. In subsequent runs it was possible to
pinpoint losses. In Run 002-RP a 33.9 percent barium loss in the uranium supernate was determined to be attributable to dilution of fuming nitric acid used to precipitate barium from the uranium solution.

5. **Operating Manpower**

Operation of the RaLa facility during the report period was accomplished by four men on each twelve-hour shift as follows:

1. Technical employee from Operations Branch.
2. Technical employee from Technical Branch.

Maintenance and Health and Safety personnel requirements were in addition to the above requirements.

6. **Off-Gas Handling**

Iodine in the process off-gas proved to be the most troublesome aspect of processing. A caustic scrubber was provided in the original design for iodine removal. However, actual operation demonstrated that excessive activity was associated with the scrubber effluent gas even at scrubber efficiencies of 95-98 percent. Iodine was evolved in curie quantities during processing and over a period of several days following each production run. Because of this fact the operation was weather dependent throughout the startup period involving radioactive material. This usually resulted in processing delays and consequent reduced product activity.

Before any operating experience was gained, iodine was expected to be evolved over a period of minutes. Instead, evolution over a period of days proved to be the case. During startup it became obvious that a gas holder, then under construction, would not be adequate for gas storage over the entire processing and post-processing period when iodine evolution prevailed. Experiments with a side stream of radioactive gas from actual production runs indicated that satisfactory iodine removal could be effected by installation of carbon beds downstream from the scrubber. Authorization for carbon bed installation is being requested of the Atomic Energy Commission so that future operation can be weather independent.

Details of the experimental off-gas testing program are given in Appendix C.
7. Liquid Waste Handling

a. By-Product Wastes

(1) Uranium-containing supernate. Uranium supernate and washes resulting from the barium nitrate precipitation step are collected and stored in uranium storage tank, L-107. Dilution with aluminum nitrate solution provides coarse solution adjustment as well as reduction in specific activity for safe sampling. The solution is transferred by steam jet to a storage tank in N cell of IGPP to await uranium recovery in plant facilities.

(2) Strontium-containing supernate. Strontium supernate resulting from the first chromate precipitation step is collected in strontium batch tank, L-111. Long term storage is provided by strontium collection tank, L-112, and strontium storage tank, L-113. Strontium produced thus far is being stored in L-112 until a demand for radioactive strontium arises. Should no demand for strontium-90 arise, the solution will eventually be transferred to permanent underground waste storage facilities along with IGPP first cycle raffinates.

b. Disposable Wastes

(1) Waste supernates and washes. All waste supernates, except those described in the preceding two paragraphs, are collected in caustic waste tank No. 2, L-109. An initial charge of acid to this tank provides for neutralizing the aluminate supernate as well as slightly acidifying the entire waste batch. In normal operation the activity in this waste averages 75,000 curies in 700 liters of solution. Because of this activity and the contained iodine-131, no attempt is made to concentrate the solution prior to transfer through U cell to permanent underground waste storage facilities.

Waste solution can be routed to the IGPP Process Equipment Waste (PEW) system. However, maximum allowable activity of 250 curies per 3,500 gallon batch limits the amount which may be disposed in this manner.

During part of the startup program when IGPP uranium recovery facilities were idle, special procedures were arranged to dispose of all RaLa wastes to the PEW system. Concentration was effected in the PEW evaporator. Volatile fission products, principally iodine-131, caused
condensate to be recycled through the evaporator in many cases. Furthermore, vessel off-gas system lines and fans became contaminated. Thus this disposal alternative is practical only for low activity wastes.

(2) **Condensates.** Condensates resulting from the nitrate boildown and product drying operations are collected in caustic waste tank No. 2, L-109, along with waste supernates and thus are not handled separately. However, separate disposal can be effected by transfer to PEW through PEW sampling tank, L-110. This is not done because the relatively small volume (<5L) adds little to the 700-liter supernate waste. Furthermore, solution handling is minimized.

(3) **Scrubber solution.** Solution from the RaLa off-gas scrubber contains 1M sodium hydroxide and 0.1M sodium thiosulfate. The solution removes radioactive iodine from off-gases and thus contains considerable activity in its 100-liter volume. From the off-gas scrubber, L-102, the solution is transferred to one of two scrubber waste hold tanks, L-103 and L-153, for neutralization with nitric acid. Final disposition is accomplished when the solution is transferred through U cell to permanent underground waste storage facilities.

8. **Radiation and Contamination**

Heat from fission products contributes to heat in the centrifuge bowl during processing. It is necessary to keep cooling water circulating in the centrifuge case jacket in order to maintain temperatures of 30-40°C.

Effects of radioactive gases are discussed in item 6 of this section.

During operation of the RaLa facility a representative of Health and Safety is present at all times. Hourly surveys are made to detect any radiation in operating areas. A radiation survey is made, also, during each sampling operation.

Exposure of operating personnel to radiation has not been excessive and is well within the maximum permissible limits. Decontamination between Runs 003-RH and 001-RP resulted in more exposure than actual operation during both runs combined.

The radiation level at the cell door has exceeded 350 R/hr during operation. This door is located in a well below and away from the main operating control area in the Access Corridor.

On several occasions, radioactive solution has backed up into lines and equipment in operating areas. In one such instance the dissolver, L-101,
recirculating steam jet was turned off without proper venting during decontamination. Condensing steam caused radioactive solution to fill the steam line to the jet. Immediate flushing with water reduced radiation in the steam line from 50 R/hr to 30 mR/hr. Another incident occurred during Runs 002-RP and 003-RP when condensate resulting from heating centrifuge L-450 case jacket became contaminated and caused condensate collection tank WF-100 to reach a contamination level of 300 mR/hr. Emptying and flushing the tank reduced radiation to a tolerable level. Source of the contaminated condensate was suspected to be a leak in the centrifuge case jacket.

Removal of the shipping pot from the cell during Run 002-RH resulted in a short burst of air-borne activity in the Process Makeup Area. The shipping pot itself is normally contaminated upon removal from the cell. A disposable plastic covering is provided to protect the shipping pot from direct contamination.

9. Decontamination

Extensive decontamination of processing equipment was attempted after Run 003-RH only. Four hundred man-hours were expended in reducing the general background radiation in L cell from an estimated 1000 R/hr to 0.3-1 R/hr. Sixteen complete equipment flushes with various decontaminants and three partial flushes were required. The rate of decontamination was slower than anticipated on the basis of previous experience with other plant equipment. This may have been caused by delays in disposing of flushes resulting from inadequate disposal facilities.

Considerable external contamination on cell vessels was detected. Highest external contamination occurred on cell ventilation filters and highest internal contamination occurred in centrifuge kidneys.

A detailed account of decontamination following Run No. 003-RH is given in Appendix D.

C. Operational Difficulties

1. Dissolution

Precise control of the dissolution reaction has not been obtained because the automatic dissolver temperature control system contains too much time lag. Since reaction rate is temperature-dependent it is thus not possible to control the off-gas evolution rate precisely at 3 cfm during dissolution. Off-gas rate varies normally from one to ten cfm but this is not considered serious. Purpose of the desired even evolution rate is to insure proper action of the automatic nitrogen dilution system used to dilute hydrogen in the off-gas below the lower explosive limit.
2. **Centrifugation**

Heating the centrifuge bowl directly with live steam results in condensate dilution of bowl contents. This fact was discovered early in the first phase of startup operation after volume increases were observed in the centrifuge bowl.

3. **Evaporation**

Evaporation of the nitrate solution is the most delicate operation in the process because of the relatively large heat transfer area to volume ratio in the slender evaporation vessel. Boilover of solution was experienced several times during the startup program. This tendency was accentuated by fission product heating. It was found that the most likely time for boilover to occur was just as the nitrate solution reached boiling temperature. Heat reduction as the boiling point is reached usually prevents this undesirable boilover.

4. **Drying**

Proper operating technique in drying is necessary to prevent either overfilling the product cup or evaporating to dryness between product wash additions. It was found that the volume of collected condensate could not be used as a precise indicator of the drying status due to solution loss during skimming and vapor loss out the condenser vent line. Drying station temperature was found to be a good indicator of drying progress. As long as product solution surrounds the drying station thermocouple, the solution boiling point is registered. As solution volume decreases below that required to cover the thermocouple, a fairly rapid temperature rise is registered due to radiant heat from the product cup walls.

5. **Pressure Pot Operation**

Operation of the pressure pots proved to be the most troublesome aspect of the entire operation. Due to the large number of times the pressure pots are used and the number of valve operations necessary with each use, there is considerable chance for operator error. Errors were minimized by establishing a standard operating procedure.
VII. CONCLUSIONS AND RECOMMENDATIONS

The startup program reported herein demonstrated that processing two-day-old MTR fuel can be conducted routinely for recovery of kilocurie quantities of barium-140. Remote operation was shown to be practical. Processing each MTR fuel element requires 24-36 hours.

Centrifugation proved to be a practical means of separating liquid from solids under radioactive conditions. Remote operation of centrifuge skimmers was developed to the desired degree to remove down to 30 milliliters of supernatant solution from gram quantities of solids.

Unrecovered uranium during processing amounts to 10 percent or less of that charged. The major uranium-bearing waste solution is processed without difficulty after necessary feed adjustment in the uranium recovery portion of the Idaho Chemical Processing Plant.

Strontium-bearing supernate from all runs involving active material was collected for storage. This solution is available for radioactive strontium recovery although none was processed during this startup program. Extensive development work for purifying this by-product strontium-bearing waste has not been conducted.

The following conclusions and recommendations are made as a result of startup experience:

A. Construction Considerations:
   (1) Plant turnover date from construction contractor is not necessarily indicative of plant startup date.
   (2) Complete equipment cleanout between construction and operation is absolutely necessary to minimize possibility of plugging small lines, jets, orifices, and other small equipment.
   (3) Complete checkout of all equipment is necessary in locating and correcting deficiencies prior to operation.

B. Process Operations
   (1) Control of dissolution reaction by automatic dissolver temperature control was not precise in providing a steady off-gas evolution rate. Nevertheless, adequate dissolution was performed.
   (2) Centrifuge cake cutting was performed satisfactorily using a high pressure solution spray. Centrifuge braking alone was not adequate in providing extreme shear action between solution and separated solids due to rather poor braking characteristics.
(3) Centrifuge bowl heating and cooling was performed adequately by using the centrifuge case jacket while rotating the bowl at 1000 rpm.

(4) Progress of the nitrate boildown operation in the Centrifuge Feed Tank (L-106 or L-156) was indicated satisfactorily by the purged probe-type level instrument in the tank itself. Attempts to follow boildown progress by observing volume of collected condensate were misleading due to loss of saturated vapor in the condenser vent system.

(5) Progress of the product drying operation was indicated satisfactorily by the product cup vapor space thermocouple temperature. Attempts to follow drying progress by observing volume of collected condensate were negated by unknown solution losses during centrifuge skimming and vapor losses in the condenser vent system. However, the collected condensate volume was useful in determining completion of drying as indicated by cessation of volume increase.

(6) Progress of centrifuge skimming was indicated satisfactorily by use of a stationary pointer and moving scale on the skimmer operating rod. It is necessary to reset the scale with respect to the pointer from time to time because of the variable centrifuge center of rotation.

(7) Unskimmable residual holdup volume in the centrifuge bowl amounted to less than 30 milliliters in spite of the relatively large bowl size. This accomplishment is facilitated by the 1/8-inch deep dimple into which the skimmer pickup nozzle works.

(8) Regulation of pH during the chromate precipitation steps is feasible even though no actual pH measurements can be made.

(9) The multitude of sequential operation steps accentuates the necessity for constant operator alertness and attention.

VIII. PROCESSING EQUIPMENT AND MATERIALS

Photographs of operating areas are given in Figures 3-6. A model of the RaLa cell is shown in Figure 7. Cell equipment layout is shown in Figures 8-10 which appear at the end of this section.

A. Vessels

1. Caustic Feed Tank, PM-120-C

This tank is located in the Process Makeup Area and is used for preparing sodium hydroxide solutions for the dissolver and off-gas scrubber. The tank is constructed of Type 347 stainless steel. Capacity is 126 liters in the
FIGURE 3. PROCESS MAKEUP AREA
(57-3450)
FIGURE 4. OPERATING CORRIDOR CONTROL AREA
(57-3332)
FIGURE 5. MAIN CONTROL PANEL, ACCESS CORRIDOR AREA

(57-1925)
FIGURE 6.
SOUTH FACE OF CELL L,
ACCESS CORRIDOR LEVEL
FIGURE 7.
RALA CELL MODEL
3-foot straight side by 1-foot 6-inch outside diameter vertical vessel. A sight glass and a mechanical agitator are provided on the vessel. A cartridge-type filter is provided on the tank discharge line.

2. **Water Feed Tank, PM-121-0**

This tank is located in the Process Makeup Area and is used for charging water to the dissolver. The 48 liter capacity vertical vessel is constructed of Type 347 stainless steel of 10-3/4-inch outside diameter by 3-foot straight side. A liquid level recorder is connected to the vessel.

3. **Nitric Acid Feed Tank, PM-122-0**

This tank is located in the Process Makeup Area and is used for charging nitric acid to the process for neutralizing caustic waste. It is also used for adding aluminum nitrate solution to the uranium storage tank for coarse feed adjustment of uranium solution. The Type 347 stainless steel vertical vessel has a capacity of 146 liters provided by 1-foot 6-inch outside diameter and 3-foot straight side. A gauge glass is provided for volume determinations.

4. **Dissolver, L-101**

The dissolver, Figure 11, is used in dissolving the spent fuel assembly for each batch. The vessel has an overall height of 11 feet 6 inches excluding the slug chute. The lower 7-foot by 8-inch inside diameter section is jacketed and serves as the dissolution section. A vapor head at the top of the dissolver measures 3 foot 11 inches high by 6-inch inside diameter by 16-inch outside diameter. The vapor head is equipped with coils of 1-inch Schedule 40 pipe and serves as a reflux condenser. A slug chute 6-feet by 6-inch diameter is welded to the top of the vapor head and extends through the cell ceiling. Material of construction is Type 347 stainless steel. Agitation of dissolver contents is provided by a nitrogen sparger. Instrumentation consists of level and density recorders plus a recording temperature controller. A crash plate rests on a special casting in the bottom of the dissolver. Air jets provide for discharging vessel contents to feed pots, L-104 and L-154.

5. **Off-Gas Scrubber, L-102**

The off-gas scrubber provides countercurrent contact of dissolver off-gas and sodium hydroxide-sodium thiosulfate scrubber solution. The vessel is 10 feet 5 inches high overall and consists of a 2-foot by 2-foot outside diameter bottom reservoir surmounted by a 7-foot by 8-inch inside diameter packed section. Packing consists of Raschig rings made of 3/4-inch long sections of 1/2-inch Schedule 40 stainless steel pipe. The bottom reservoir is jacketed for cooling. An integral air lift is provided for recirculating solution over the packing. Construction is entirely of Type 347 stainless steel. Instrumentation consists of level and density recorders plus a temperature indicator. Steam jets provide for discharging scrubber solution to the scrubber waste hold tanks L-103 and L-153.
6. **Scrubber Waste Hold-Tanks, L-103 and L-153**

These tanks serve to collect spent scrubber solution for decay and neutralization prior to disposal as waste. Each Type 347 stainless steel vertical jacketed vessel is 2-feet long by 2-feet outside diameter with flanged and dished heads. Maximum volume is 176 liters. An air sparger provides agitation. Instrumentation consists of level and density recorders plus a temperature indicator. Steam jets provide transfer to the plant waste system. A remote sampler is connected to each vessel.

7. **Feed Pots, L-104 and L-154**

The feed pots, Figure 12, serve to meter dissolver solution to each centrifuge at a constant rate. The vertical vessels are 2-feet 9-inches by 6-5/8-inch outside diameter and contain an internal constant head orifice resembling a funnel. Overflow from the funnel returns to the dissolver to be recirculated by an air operated jet. Type 347 stainless steel is the material of construction.

8. **Supernate Tank, L-105**

The supernate tank, Figure 13, collects solution from the centrifuge skimmer used for handling waste supernates. Air jets are used to transfer skimmed supernates from the supernate tank to waste, storage, or process. The vessel is constructed of a 6-foot length of 4-inch Schedule 80 stainless steel pipe and contains an internal annular jacket as well as an external jacket. Maximum volume is 15 liters. Instrumentation consists of level and gravity recorders plus a temperature indicator. An air sparger provides agitation.

9. **Centrifuge Feed Tanks, L-106 and L-156**

The centrifuge feed tanks, Figure 14, serve as the evaporation vessels for concentrating uranium nitrate solution in the process. Each vertical vessel is constructed of a 6-foot 2-inch length of 4-inch Schedule 80 pipe and contains an internal annular jacket as well as an external jacket. Maximum vessel volume is 10 liters. The vessels contain an internal valve at the bottom outlet which is operated by a solenoid at the top of the vessel. A valve stem extends through the entire length of the vessel. Energizing the solenoid causes the valve to be lifted off the valve seat and allows tank contents to flow out of the tank under the force of gravity. All tank components are constructed of Type 347 stainless steel. Instrumentation consists of level and density recorders plus a temperature indicator. An air sparger provides agitation.

10. **Uranium Storage Tank, L-107**

This vessel serves as a storage facility for by-product uranium solution from the RaLa process. The vertical jacketed 3-foot by 2-foot diameter
vessel is constructed of Type 347 stainless steel and has a capacity of 236 liters. An air sparger provides agitation. Air and steam jets provide transfer to waste, process, or to ICOPF uranium recovery facilities. Instrumentation consists of liquid and density recorders plus a temperature indicator. A remote sampler is connected to the vessel.

11. **Caustic Waste Tanks, L-108 and L-109**

The caustic waste tanks collect overflow supernate from the centrifuge bowl. Each horizontal, cylindrical, jacketed vessel is 6-feet long by 4-feet diameter. Maximum volume is 1900 liters. Type 347 stainless steel is the material of construction. An air sparger provides agitation. Instrumentation consists of level and density recorders plus a temperature indicator. Steam jets provide transfer to the plant waste system. A remote sampler is connected to the vessel.

12. **PEW (Process Equipment Waste) Sampling Tank, L-110**

This tank serves to collect waste condensates for sampling prior to disposal as waste. The Type 347 vertical cylindrical tank is 3 feet by 6-5/8 inches outside diameter. Maximum volume is 13.9 liters. An air sparger provides agitation. The tank overflows to Process Equipment Waste. Steam and air jets provide for discharging the tank contents to waste or process. Instrumentation consists of level and density recorders plus a temperature indicator. A remote sampler is connected to the vessel.

13. **Strontium Batch Tank, L-111**

This tank serves to collect the by-product active strontium-bearing supernate for sampling prior to storage. The vertical vessel is fabricated of 2-foot by 4-inch diameter Schedule 80 Type 347 stainless steel pipe fitted with dished heads. Maximum volume is 1.9 liter. An air sparger provides agitation. A remote sampler is provided. Instrumentation consists of level and gravity recorders plus a temperature indicator. The tank is also used to measure batches of strontium solution to be processed for strontium recovery. Air jets provide for transferring contained solution to storage or process. An overflow nozzle discharges to strontium storage tank, L-113.

14. **Strontium Collection Tank, L-112**

This tank serves as one of two vessels provided for long-term active strontium storage. The vertical, jacketed 3-foot by 10-inch inside diameter vessel is constructed of Type 347 stainless steel. An air sparger provides agitation. Maximum volume is 43 liters. Instrumentation consists of level and density recorders plus a temperature indicator. Air jets provide transfer to companion storage, to waste or to strontium batch tank, L-111. A remote sampler is connected to the vessel.
15. **Strontium Storage Tank, L-112**

This vessel is similar to strontium collection tank, L-112.

16. **Centrifuge Condensers, L-300 and L-350**

These condensers serve to condense and collect vapors in the centrifuge vent system. Each vessel consists of an upper 5 foot jacketed section of 1-1/2-inch schedule 40 stainless steel pipe and a lower 4 foot jacketed section of 3-inch schedule 80 stainless steel pipe. Maximum volume is 5.3 liters. Vapors enter the top of the top section, condense, and collect as condensate in the bottom section. The top of the bottom section is vented. An air jet provides for transfer of condensate to the PEW Sampling Tank, L-110. Instrumentation consists of level and gravity recorders plus a temperature indicator. A steam sparger is provided for agitation and decontamination.

17. **Feed Tank Condensers, L-306 and L-356**

The feed tank condensers serve to condense and collect vapors from the feed tank during nitrate boildown. These condensers are identical to the centrifuge condensers previously described.

18. **Overhead Receiver, L-320**

This vessel serves as a collecting condenser for the product dryer head. The vertical, jacketed, stainless steel vessel is constructed of a 5-foot length of 1-1/2-inch schedule 40 pipe. Maximum volume is 2.3 liters. An air jet provides for transfer of condensate to the PEW Sampling Tank, L-110. Instrumentation consists of a level recorder and temperature indicator. An air sparger provides agitation.

19. **Condensate Collection Tank, WF-100**

This vessel serves to collect steam condensate from process vessel jackets. The horizontal, cylindrical Type 347 stainless steel vessel with dished heads has a maximum volume of 100 gallons. Two 15 gpm centrifugal pumps are provided for emptying the vessel to waste. A level indicator controller starts one pump when the vessel is half full. Should the level continue to rise, the second pump starts automatically. Water to a condenser on the vessel inlet line is controlled by a temperature actuated valve.

20. **Oil Feed Pot, PM-123-0**

The vessel serves as an oil supply tank for the centrifuges. The two-gallon carbon steel vessel is provided with a removable lid for manual filling.
21. **Waste Oil Tank, WF-101**

This vessel serves to collect waste oil from the centrifuge. The carbon steel vessel of 6-gallon capacity is constructed for manual dumping. The vessel is equipped with handles, a pour spout, and an inlet funnel. When full, the tank is physically carried to an oil disposal facility.

22. **Pressure Pots, PA-801, PA-851**

These vessels serve as pressurized addition funnels for adding solutions to process equipment. Stainless steel 2-inch schedule 40 pipe is used to provide 1 liter capacity. A conical bottom provides minimum holdup. Two flanged inlet nozzles at the top of the vessel provide for solution charging and air line connection. The 1/4-inch pipe bottom outlet is provided with a plug valve.

23. **Pressure Pots, PA-800, PA-850, PA-805, PM-800, PM-850, PM-806, PM-856**

These vessels serve as pressurized addition funnels for adding solutions to process equipment. Stainless steel 4-inch schedule 40 pipe is used to provide 4 liter capacity. A conical bottom provides minimum holdup. Two flanged inlet nozzles at the top of the vessel provide for solution charging and air line connection. The 1/4-inch pipe bottom outlet is provided with a plug valve.

24. **Product Cup**

The product cup, Figure 15, is used to evaporate product solution to dryness. It also serves as a shipping container for dried product. The 3-inch diameter vessel is platinum lined over its entire 10-inch length including the cone bottom. The cup is handled in the cell by a remotely operated mechanical manipulator.

B. **Centrifuge Equipment**

Centrifuge plan and section drawings are shown in Figures 16 and 17.

1. **Motor**

The five horsepower direct-current shuntwound centrifuge motor has three speed ranges:

\[\begin{align*}
(1) & \text{ Jog } - 25 \text{ rpm}., \\
(2) & \text{ Low } - 100 \text{ to } 1000 \text{ rpm}, \\
(3) & \text{ High } - 1000 \text{ to } 2600 \text{ rpm}. \\
\end{align*}\]

On the centrifuge control panel a push-button is provided for each speed range and rheostats are provided for low and high speed ranges. Dynamic...
braking permits stopping the centrifuge from maximum speed of 2600 rpm in 60 seconds. Braking is used during cake cutting and washing operations to provide shear action between cake and supernatant liquor.

A stainless steel can encloses the centrifuge motor. Two 4-inch nozzles on the can provide inlet and outlet for a forced air cooling system for the motor. The can is flanged and is bolted to the motor base flange. An electric driven blower provides 25.0 cfm of air for cooling the motor.

A frictionless tachometer provides indication of speed on a dial mounted on the centrifuge control panel.

A motor-generator set supplies direct-current power to the centrifuge motor.

Continuous, once-through oiling to the centrifuge motor bearings is supplied through a sight feeder located in the control area. Oiling rate is ten drops per minute. Used oil is collected in a small hand-dump can and is not re-used.

The centrifuge motor is mounted on a frame above the centrifuge.

2. Universal Joint

A universal joint is located in a housing just below the motor. This housing is actually part of the motor support framework. A two-prong drive fork pinned to the motor shaft is the driver portion of the universal joint. Two drive buttons are installed at the end of each prong of the drive fork. These buttons are installed stem to stem, the leading button for driving contact and the trailing button for braking contact. These buttons face against similar buttons installed in a web of the driven drum which is rigidly attached to the centrifuge shaft. The outer surface of the driven drum normally would be used in conjunction with brake shoes for braking. However, in this particular machine there is no provision for mechanical braking. There is no mechanical connection between drive fork and driven drum. The fork is merely inserted in the drum so that buttons in the drive fork contact buttons in the driven drum. The buttons are constructed of carbon steel and are case hardened after fabrication.

The buttons are lubricated from an oil collector ring attached to the drive fork hub. Capillary tubes carry oil from the collector ring directly to the button contact surfaces. A special oil nozzle feeds the collector ring. Oil dripping from the buttons is drained from the bottom of the universal joint housing.
3. **Shaft Bearings**

A shaft bearing housing just below the universal joint carries the entire suspended weight of centrifuge bowl, shaft, bearings, and driven drum of the universal joint. The bearing housing is supported by stainless steel mesh rings to allow for oscillation of the centrifuge bowl shaft. The mesh rings are supported by the universal joint housing which is a part of the centrifuge motor support frame. The shaft bearing housing is about 6 inches long and contains two roller bearing assemblies spaced 4 inches apart. One oil connection to the bearing housing serves both bearings. Oil overflows an inlet reservoir to the top bearing from which it flows by gravity to the bottom bearing and on to the oil outlet connection. Oil flow rate is regulated at ten drops per minute through a sight feeder. Discharge oil joins oil from the motor bearing and drive buttons. Oil lines to and from the bearing housing are provided with flexible lines to allow for shaft oscillation.

4. **Centrifuge Shaft**

The shaft is welded to the centrifuge bowl at the lower end and is bolted to the universal joint driven drum at the upper end. The shaft is 2-feet 4-inches long and is 1.062-inch diameter over most of its length except for 0.781-inch diameter in the bearing housing and a 0.9375-inch diameter intermediate section. An oil slinger ring is attached to the shaft at the junction of the 0.9375-inch and 1.062-inch diameters. The slinger ring serves to prevent bearing oil from reaching the centrifuge bowl where product contamination would result. A floating carbon ring seal is provided where the shaft passes through the top plate assembly. However, the seal is not air-tight and becomes less effective as wear occurs. The centrifuge was operated during the startup program without the shaft seal in order to prevent carbon contamination of product.

5. **Centrifuge Bowl**

The centrifuge bowl is 6-3/4 inches deep by 16-3/4 inches I.D., and 18-15/16 inches O.D. It contains two horizontal ring baffles attached to the inner bowl wall. These baffles stabilize operation when operating at maximum volume and also prevent short circuiting of solution over the surface of supernate under conditions of continuous feeding and supernate overflow. An overflow lip at the top of the bowl is drilled with eight 3/16-inch diameter holes to provide a weir for supernate overflow when operating at the maximum bowl capacity of 5 liters. Extension of the bowl lip for 1 inch toward the center beyond the weir holes provides a splash baffle to minimize spray losses. The inner bowl wall contains a continuous horizontal depression 1/8-inch deep which is located 1-1/4 inches down from the underneath side of the bowl lip. This depression, or dimple, provides a low spot into which the skimmer pickup nozzle works for supernate removal. Solution volume remaining after maximum skimming is 30 milliliters or less. The bowl outer wall is provided with a jacket for heating and cooling. The annular area between jacket and bowl wall is provided with a spiral baffle such that coolant makes about thirteen complete circuits around the bowl prior to
discharging near the top of the bowl. Coolant is supplied to the jacket from a stationary nozzle directed to discharge into the jacket lip which overhangs the bottom of the bowl proper.

The inner bowl wall is not tapered but is straight except for the small dimple area mentioned previously. The bowl bottom is not level but contains a double taper draining to a depression halfway between bowl center and wall. A jet suction probe can be lowered into this depression within 1/16 inch of the bowl bottom for nearly complete removal of the solution from the bowl. A hub 2-1/4-inches high by 2-inches O.D. in the center of the bowl bottom is provided for attachment to the shaft which is bolted and seal welded in place.

6. **Skimmers**

The centrifuge is provided with two independent skimmers. One of these is used for waste supernate removal and the other is used solely for removal of product solution to minimize cross contamination of product. The skimmers are identical except for location and size of discharge bowls or kidneys. Both skimmers are operated remotely through direct mechanical levers.

The skimmer pickup nozzle consists of a curved 1/8-inch pipe with an open end such that it can be introduced into the rotating supernate. The skimmer pivots in a plane approximately 3-1/2 inches away from and parallel to the bowl wall. When fully engaged the skimmer touches the bowl wall tangentially with the pickup nozzle pointing opposite to the direction of bowl rotation. When fully disengaged the skimmer pickup nozzle is rotated clockwise to a point which is clear of the overflow lip. Maximum operating angle of the skimmer from fully in to fully out is ninety degrees. A splash shield near the skimmer pickup nozzle minimizes spray during the skimming operation. A special metal bushing provides a limited seal where the skimmer extends through the centrifuge top plate. The moving (inner) portion of this bushing is welded to the straight vertical section of the skimmer pipe while the stationary (outer) bushing surface is attached to the top plate. An operating arm is attached by set screw to the inner bushing for motion transmission to the skimmer. The skimmer works into the 1/8-inch deep dimple in the bowl wall. Flow of supernate out the skimmer is induced as in a pump by the conversion of velocity head to pressure head. Each skimmer discharges into a vented reservoir called a kidney. One kidney is sized to hold 5 liters which is the maximum operating volume of the centrifuge bowl. The second kidney handles product solution only and is sized for only one liter because product solution does not exceed that volume. Each kidney contains two compartments. The smaller compartment surrounds the skimmer uptake pipe and contains a limited seal provided by a metal bushing. From this compartment a return drain line connects to the centrifuge bowl through the top plate. A weir separates the two compartments. The skimmer discharge nozzle extends over the weir into the larger kidney compartment. It is this compartment which provides the volumes mentioned previously. A drain from a low point in the large compartment carries skimmed supernate from the kidney by gravity flow.
In case the large compartment becomes flooded due to a plugged outlet or excessive skimming rate, solution overflows the weir into the small compartment and returns to the bowl through the drain line. The kidney compartments have separate bolted lids, both of which are provided with decontamination spray nozzles and one of which contains a vent line.

7. **Unloading Jet**

An air jet suction nozzle of 1/8-inch pipe extends through the top plate of the centrifuge. The position of this nozzle is adjustable over a vertical distance of 1/4 inch. In normal centrifuge operation the nozzle remains in its maximum vertical up position. For unloading, the nozzle is lowered 1/4 inch to within 1/16 inch of the bowl bottom while the centrifuge is operated at jog speed. A pair of bellows, one above and one below the top plate, provide a seal between jet suction nozzle and centrifuge top plate. Solution does not contact the inner surface of either bellows but spatter from the bowl can contact the outer surface of the lower bellows. The jet raising and lowering mechanism is operated remotely through direct mechanical levers.

8. **Cake Cutter Nozzle**

A cake cutter nozzle of 1/8-inch pipe extends down through the top plate and terminates just above the bowl bottom. Six holes of approximately 1/32-inch diameter are drilled in the cutter nozzle, the lower end of which is closed. Two sprays impinge against the bowl wall in each of the three wall sections divided by the bowl baffles. The sprays are directed so that spray solution opposes the bowl rotation and strikes the bowl wall at minimum angle. Solution used for cake cutting is introduced to the cutter nozzle at 40 psig to provide high velocity flow through the nozzle spray holes.

The cake cutter nozzle assembly is bolted to the top plate to facilitate disassembly for physically unplugging spray holes when necessary.

9. **Thermocouple**

A thermowell of 1/2-inch pipe extends down through and is welded to the top plate terminating just above the bowl bottom. An iron-constantan thermocouple is located at the bottom of the thermowell.

10. **Feed Nozzles**

Two separate feed nozzles, 1/8 inch and 1/4 inch, are welded to the top plate and extend down into the bowl.

11. **Top Plate**

The centrifuge top plate serves several purposes as follows:

a. Provides top for open centrifuge bowl.
b. Provides support for stationary case bolted to the under side of the top plate.

c. Provides support for bowl auxiliaries such as skimmers, jet suction nozzle, thermowell, and feed nozzles which extend down into the bowl.

d. Provides base for motor mounting frame.

e. Provides foundation support for entire centrifuge and motor assembly.

The top plate is constructed of 5/8-inch stainless steel plate 26 inches by 42 inches. The shaft opening in the center of the top plate is provided with a 3-1/2-inch O.D. hollow vertical cylinder projecting 3-1/2 inches above the top plate. The floating carbon shaft seal assembly rides on the top of this cylinder. Because of this shaft opening the bowl is not sealed from the external environs. On one side of the cylinder is connected the 2-inch centrifuge vent line which is connected through a condenser to a vacuum vent system. A 360° decontamination spray ring is located at the bottom of the cylinder underneath the top plate. This spray ring facilitates flushing internal equipment. A series of three concentric deflector baffles welded underneath the top plate minimizes bowl spray loss and also deflects decontamination spray for maximum coverage.

The top plate contains a 4-inch diameter access hole for insertion of a periscope. The access hole is fitted with a remotely operated plug.

12. Case

A cylindrical centrifuge case surrounds the bowl and is bolted to the under side of the top plate. The case bottom is sloped to a two-inch drain nozzle to facilitate solution removal. Sides and bottom of the case are jacketed for heating and cooling by steam and water, respectively. A bowl bumper ring is welded inside the case. Six decontamination sprays extend through the case side for washing the outside bowl wall. Two spray nozzles extend up through the case bottom to supply steam or water for heating or cooling the bowl by direct contact. One of these nozzles is directed so as to fill the bowl jacket when the bowl is operating. The other nozzle terminates in a horizontal spray pipe containing fourteen spray holes along its top for heating or cooling the bowl while at rest or operating at low speed. The case drain handles overflow supernate from the bowl as well as spray water used to cool the bowl by direct contact.

13. Recirculating Air System

A recirculating air system takes suction from the top of the case drain line and discharges to the top plate shaft hole cylinder mentioned in previous sections. A stainless steel mesh filter and a condenser are
provided in the recirculating air line. The line is also connected to a vacuum vent system through another collecting condenser. Operation of the centrifuge induces an air flow through the recirculation system. Purpose of the recirculation system is to reduce the volume of air to be handled by the vacuum vent system.

14. Oil Collection Trough

A sheet metal oil collection trough surrounds the shaft oil slinger ring. Any oil leaking down the shaft is slung off the slinger ring into the collection trough which drains to the floor. The collection trough is attached by straps bolted to the kidney lids.

15. Periscope

A remotely-operated periscope is inserted through a special access hole in the centrifuge top plate for viewing the inner bowl wall. The access hole plug is operated remotely from the operating area through direct mechanical levers. The periscope is extended and retracted by use of an operating handwheel. Two integral lamps near the periscope lens automatically turn on when the periscope is fully extended to provide illumination. The periscope field of vision is not adjustable and is limited to a 2-1/2-inch diameter spot in the middle third of the bowl wall. The periscope extends 55 inches from fully retracted to fully extended. The periscope is used to determine status of bowl cleaning operations.

16. Microphone

A microphone is attached to the centrifuge top plate for transmission of sounds to a speaker located in the operating area. Principally, bearing and skimming noises are transmitted.

17. Instrumentation

Instrumentation consists of speed indicator and thermocouple, both of which are described in preceding sections. Instrumentation is mentioned to point out that neither level nor specific gravity instruments are provided on the centrifuge. These are unnecessary in this application but, nevertheless, are adaptable.

18. Decontamination Sprays

Internal decontamination sprays are provided in the bowl and skimmer kidneys. External sprays are directed toward the top plate for decontamination.

19. Lubrication

Three sight feed oilers provide lubrication to motor bearings, universal joint drive buttons, and shaft bearings. A 30-foot head of oil
is maintained on the sight feeder inlet valves. The sight feeders are half
filled with water to facilitate regulating oiling rate and to provide a
liquid-full system without air traps.

20. Unit Shielding

Centrifuge unit shielding consists of 4 inches of lead surrounding
the case. The lead shielding lies between stainless steel forms which also
act as a foundation. The top plate rests on top of the unit shielding and
supports the entire centrifuge and motor assembly.

21. Interlocks

Several interlocks are provided in the centrifuge system. One such
interlock is provided on the centrifuge jet lowering lever. When the jet
suction probe is lowered to jetting position in the centrifuge bowl, a switch
is energized causing a "jet down" light to glow on the centrifuge control
panel. Simultaneously, the low and high speed electrical circuits are de-
energized so that only jog speed can be obtained when the jet is down. Other
interlocks are provided on the skimmer operating levers such that a warning
horn sounds if the skimmers are left in operating position and the centrifuge
is operating below safe skimming speed (500 rpm). Solenoid-operated locks
prevent skimmer levers from being moved from the out position when the centri-
fuge is operating below safe skimming speed. These interlocks are provided
to prevent damage to centrifuge equipment and to prevent solution loss due to
accidental skimming.

C. Off-Gas Handling Equipment

1. General

Newly constructed off-gas handling equipment outside the RaLa cell
consists of a blower room (containing jets, blower, hydrogen analyzer), valve
box, gas-holder, and stack. The off-gas scrubber, L-102, is described in
the vessel section of this report. See Figure 18 for the off-gas flowsheet.
The external equipment mentioned here was not actually used in any of these
runs because it was not included in the original construction. Instead, the
regular ICPP dissolver off-gas system was used to discharge RaLa gases to the
stack.

2. Blower Room Equipment

A concrete blower room just outside the processing building con-
tains two jets and an electrically driven blower for transferring dissolver
off-gas from the RaLa cell to either the gas-holder or stack. The jets
are provided with air, steam, and nitrogen. Remote valves provide operating
flexibility. Gas from the blower room is discharged through a 3-inch
stainless steel pipe to a valve box.
3. Valve Box

A concrete valve box houses piping and remote valves for transferring gas from blower room to gas holder or from gas holder to stack.

4. Gas Holder

A Wiggins gas holder provides gas storage capacity of 10,000 cubic feet. The gas holder is used to store radioactive gas during processing periods when meteorological conditions preclude direct venting of gas to the stack. An earth berm around the gas holder provides shielding.

5. Stack

The ICPE stack is used for discharging gas to the atmosphere.

D. Mechanical Equipment

1. Manipulator

A hand-operated remote mechanical manipulator, Figures 19 and 20, is provided for handling the product cup. The manipulator operates in one limited vertical plane. Cup stations served by the manipulator are: elevator, spare cup station, drying station, recycle station, and product testing station. Three handwheels provide for vertical, horizontal, and rotation movement. Each wheel is provided with a Feeder Counter to aid in positioning the manipulator.

The manipulator sub-assembly travels horizontally on two guide rods. The handwheel which controls the horizontal movement turns a lead screw through a universal joint. The lead screw works in a nut on the sub-assembly to produce horizontal movement.

The handwheel controlling vertical movement turns a square bar through a universal joint. This turns a horizontal shaft on the sub-assembly through a gear assembly. Another gear assembly on the opposite end of the shaft turns the vertical lead screw on which the cup tool rides. A keyway prevents the cup tool assembly from turning.

The handwheel controlling rotation of the cup tool also turns a square bar through a universal joint. This turns a geared horizontal shaft which imparts motion to the sub-assembly arm.

The cup tool is designed to engage the lifting trunions on the product cup and provides a cover for the cup when it is being handled by the manipulator. A cup tool adapter which also is handled by the cup tool is provided for handling the cup plug.
2. Elevator

An electrically driven cable operated mechanical elevator, Figure 21, is provided for transferring the product cask (shipping pot) in and out of the cell. The elevator platform rides on a vertical track assembly which allows only vertical movement. A shielded box in the Process Makeup Area contains the cable drum and idler pulley. Outside the box is located the electric motor and gear reducer which is connected to the cable drum shaft. Limit switches automatically stop elevator movement at either extremity of travel.

An integral part of the elevator platform is a 12-inch thick lead plug which provides shielding when the elevator is in its maximum up position with a 13-inch thick shielding plug removed from the top of the shipping pot opening in the cell ceiling.

3. Dissolver Slide Valve

The dissolver slide valve, Figure 22, serves as lid and charging port for the dissolver. It is located in the floor of the Process Makeup Area directly above the dissolver. The slide valve consists of two pneumatically-operated lead plugs. The charging plug contains a vertical hole for the fuel assembly to pass through and the solid shielding plug contains a gasket at the bottom to seal off the dissolver from the Process Makeup Area. Pneumatic cylinders provide motivation for both vertical and horizontal movement of the slide valve. A mechanical interlock prevents the carrier-charger drawer from being operated before the slide valve is opened.

4. Drying Station

a. Positioner

The manually-operated drying station positioner, Figure 23, is designed to position the product cup under the dryer head or recycle head for product drying or recycling to the process. The drying station positioner is provided with two positioning arms to serve both heads. The product cup fits into the dryer which is attached to one of the positioning arms. This arm operates over a ninety-degree arc to accommodate cup loading, dryer head, and recycle head. A Veeda Counter geared to the handwheel operating shaft facilitates precise positioning of product dryer and recycle station.

b. Product Dryer

An elevation of the product dryer is shown in Figure 23. The product cup rests in a well in the center of the dryer. Surrounding the well is an electric heating coil encased in Nichrome steel tubing. Flexible electrical leads to the
dryer permit movement. The dryer can be raised or lowered on a vertical guide post by use of a hydraulic lifter in the floor underneath the dryer head.

c. **Dryer Head**

The dryer head is shown in Figure 23. It serves as a lid for the product cup during the drying operation. The dryer head contains a thermocouple and feed lines from the centrifuge product skimmer kidneys. A vent line from the dryer head connects to a collecting condenser for measuring condensate volume.

d. **Hydraulic Lifter**

A hydraulically-operated piston underneath the dryer head serves to raise the product dryer so that contact is made between product cup and dryer head.

5. **Recycle Station**

a. **Product Cup Holder**

The cup holder consists of a water bath mounted on a vertical guide post. An elevation of water bath and recycle head is shown in Figure 23. The product cup is inserted in the water bath at loading position No. 2. The water bath can be raised or lowered on its vertical guide bar by use of a hydraulic lifter in the floor underneath the dryer head.

b. **Recycle Head**

The recycle head contains an addition line and a jet suction line. By use of these, product may be dissolved in water and recycled to process. A vent line connected to the dryer head is provided with a remote controlled valve.

c. **Hydraulic Lifter**

A hydraulically-operated piston underneath the dryer head serves to raise the water bath so that contact is made between product cup and recycle head.

6. **Viewing Window Wiper**

A pneumatically-operated blade wiper is installed on the cell side of the viewing window. A water spray is provided for use in conjunction with the wiper.
7. Caustic Metering Pump, PM-220-1

The caustic metering pump is located in the Process Makeup Area and serves to transfer sodium hydroxide solution from caustic tank PM-120-0 to dissolver L-101. The pump is driven by an electric motor and contains a reciprocating piston with variable stroke. Maximum pumping rate is 1.5 liters per minute.

8. Samplers

The standard sampler used throughout the ICPP is utilized to sample solutions in the RaLa process. A manually-operated tong is used to handle sample bottles remotely. Sample bottles contain Neoprene gaskets to permit insertion of hypodermic needles to complete sample flow induced by an air jet.

9. Overhead Crane, PM-10-1

A 15-ton overhead crane is located on a track over the RaLa cell in the Process Makeup Area. The crane is used to handle carrier-charger, shipping pot, and elevator access hole plug.

E. Shielded Carriers

1. Carrier-Charger

The carrier-charger, Figures 24 and 25, serves to carry an irradiated fuel assembly from the Materials Testing Reactor to the RaLa slug chute in the Chemical Processing Plant. The carrier-charger is designed to contain water for fuel assembly cooling. External finned cooling coils provide for water cooling by natural convection recirculation. A drawer for unloading the assembly is operated by a pneumatic wrench through a screw shaft. A top plug can be removed for fuel element loading. Lead shielding reduces radiation to the following calculated values at contact:

Top = 1.1 mr/hr
Sides = 5.2 mr/hr
Bottom = 144 mr/hr

2. Shipping Cask

The shipping cask is shown in Figure 26. Shielding is provided by a uranium sphere and lid. A once-through cooling system provides for heat removal from the cask caused by gamma irradiation from product. The shipping cask is shipped inside a carbon steel enclosure, Figure 27, to meet ICC regulations.
F. **Instrumentation**

Level and density instruments are the purged probe type. All cell vessels except feed pots are provided with these instruments.

Iron-constantan thermocouples are connected to point recorders for temperature indication.

A rotameter in the dissolver off-gas line records gas flow rate (through an induction coil and electric transmitter) on a chart.

A hydrogen analyzer in the dissolver off-gas system operates on the thermal conductivity principle. Sample flow through the analyzer is induced by a nitrogen operated stainless steel ejector. This instrument is used to determine completion of the fuel assembly dissolution reaction.

Magnehelic vacuum gauges are used to determine cell and cell vent system vacuum. A compound gage is used on the dissolver high level instrument probe to indicate dissolver vacuum. This compound gage actuates automatic valves in the off-gas rotameter by-pass to relieve pressure surges in the dissolver.

Frictionless tachometers provide centrifuge speed indication.

Bourdon tube-type pressure gages are used to indicate steam and air pressure.

A photoneutron radiation counter, Figure 28, is used for product quantity determination. Gamma rays from lanthanum-140 in the product produce neutrons in a beryllium block. Emitted neutrons are moderated by paraffin before striking a boron counting tube where alpha particles are produced to ionize gas in the tube. The tube is electrically connected to a counter register.

G. **Shielding**

1. **Cell Walls**

   Shielding was designed to reduce radiation at the outer wall surface to 1 mr/hr. Cell walls are constructed of ordinary concrete 6 feet thick. Where this thickness could not be maintained because of equipment, lead or barytes concrete was used to maintain uniform shielding.

2. **Pipe Sleeves**

   Pipe sleeves in walls and ceiling are offset at least six pipe diameters. A bend is made in each sleeve at one-third and two-thirds the distance through the wall, thus retaining two-thirds of the wall shielding.
3. **Viewing Window**

The window consists of seven layers of Type B shielding glass \( (\mathcal{Q} = 3.27 + 0.08 - 0.07) \). Each layer is 7-1/4 inches thick. Space around the glass is filled with steel shot. Space between glass layers is filled with a special oil. The cell wall underneath the window is constructed of high density concrete.

4. **Product Testing Station**

The testing station is shielded on the bottom and around part of the sides by 5 inches of lead to prevent cell background activity from affecting product counting.

5. **Centrifuge Unit Shielding**

The centrifuges are shielded on sides and bottom by 4 inches of lead to provide limited cell access before complete decontamination is effected.

6. **Dissolver Slide Valve**

The valve box containing the slide valve is shielded on the bottom by 5 inches of lead. When closed the slide valve shielding plug provides 12-1/4 inches of lead over the dissolver slug chute.

7. **Platform Elevator**

A 13-inch lead plug rests in the top of the elevator access hole in the cell ceiling. When this is removed with the elevator in the up position, shielding is provided by a 12-inch lead plug mounted on the elevator platform.

8. **Shielded Process Storage Vessels**

To provide for both long-term storage and cell accessibility, several vessels are locally shielded. The strontium tanks, L-112 and L-113, are shielded separately. A second shielded compartment contains caustic waste tank No. 2, L-109; scrubber waste hold tanks, L-103 and L-153; and uranium storage tank, L-107.

H. **Decontamination Equipment**

1. **Internal**

Nearly all process vessels contain internal decontamination spray nozzles connected to addition lines in operating areas. Steam, water, or chemicals may be added through these addition lines.
2. **External**

Six spray heads in the top of the cell, two spray heads near the cell ventilation filters, and ten spray heads in the centrifuge-drying station area provide for equipment external decontamination. Water and steam connections are provided for these spray heads.

I. **Chemicals Required**

1. **Process**

Basic chemicals used in the RaLa process are listed in Table 8.

**TABLE 8**

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Use</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium hydroxide, pellet, grade, 98.8 percent</td>
<td>Dissolver solution</td>
</tr>
<tr>
<td>Sodium hydroxide, granulated flakes, technical grade, 96-98 percent</td>
<td>Off-gas scrubber solution</td>
</tr>
<tr>
<td>Sodium thiosulfate, pentahydrate, crystal</td>
<td>Off-gas scrubber solution</td>
</tr>
<tr>
<td>Barium nitrate, crystal</td>
<td>Barium carrier</td>
</tr>
<tr>
<td>Strontium nitrate, crystal</td>
<td>Strontium carrier</td>
</tr>
<tr>
<td>Aluminum nitrate nonahydrate, crystal</td>
<td>Coarse feed adjustment of uranium solution</td>
</tr>
<tr>
<td>Sodium acetate, crystal</td>
<td>Buffer</td>
</tr>
<tr>
<td>Sodium dichromate dihydrate, crystal</td>
<td>Convert NO$_3^-$ salts to CrO$_4^-$</td>
</tr>
<tr>
<td>Nitric acid, fuming, 90 percent</td>
<td>Precipitate Ba(NO$_3$)$_2$</td>
</tr>
<tr>
<td>Nitric acid, reagent, 69-71 percent</td>
<td>Ba(NO$_3$)$_2$ cake wash and dilute HNO$_3$ makeup</td>
</tr>
<tr>
<td>Nitric acid, 60 percent</td>
<td>Caustic waste neutralization</td>
</tr>
</tbody>
</table>
2. **Decontamination**

   Basic chemicals used for decontamination are as follows:

   a. Nitric acid, 60 percent.

   b. Sodium hydroxide, granulated flakes, technical grade, 96-98 percent.

   c. Sodium fluoride, crystal.

   d. Phosphoric acid, research grade, 99 percent.

   e. Tartaric acid, crystal.

   f. Sodium hydroxide, pellet, grade, 98.8 percent (used for dissolver L-101 only).
TYPICAL FOR FOLLOWING CONNECTIONS:
1. INLET FROM 422/148
2. OUTLET TO 357/145
3. OUTLET TO 357/176
4. OUTLET TO 357/176
5. OUTLET TO 357/176
6. TANGENT LINE
7. TANGENT LINE
8. JACKET OUTLET
9. JACKET INLET
10. ANNULAR INLET
11. SUPPORT
PT. LINING SPIN INTO GEODES

4 x SIZE

1/4 DIA. SS. ROD
(TYPE-3/4). PRESS
FIT TO CUP

PLATINUM LINING:

LOAD THICK

GENERAL NOTES:
1. COPPER MATERIAL - STAINLESS STEEL.
2. DIMENSIONS GIVEN APPLY TO STAINLESS STEEL CUP ONLY. PT. LINING TO BE SPUN ON AS FINAL OPERATION.

TOLERANCES UNLESS OTHERWISE SPECIFIED:

FRACTIONS ±
DECIMALS ±
BREAK ALL DIAMETERS UNLESS OTHERWISE SPECIFIED.
FINISH MACHINES AS STANDARD.
DO NOT SCALE THIS DRAWING.

PHILLIPS PETROLEUM COMPANY
ATOMIC ENERGY DIVISION
IDARO FALLS, IDAHO

FIG. 15
PRODUCT CUP

A B C D E F
MANIPULATOR SUB-ASSEMBLY

WITH SHIPPING CONTAINER COVER ENGAGED BY CUP TOOL

SECTION A

WITH PRODUCT CUP ENGAGED BY CUP TOOL
PLAN

BOTTOM RESERVOIR DRAIN

TUBE SUPPORT

FINNED TUBING

TUBE SUPPORT

FINNED TUBING

TOP RESERVOIR

TOP RESERVOIR DRAIN (BOTTOM RESERVOIR DRAIN DIRECTLY BELOW)

HANDLE

SPRING

INTERLOCK PIN

INTERLOCK PIN RELEASE ARM

DISSOLVER SLIDE VALVE

CHARGER DRAWER INTERLOCK

FIG. 24

CARRIER-CHARGER PLAN
## APPENDICES

<table>
<thead>
<tr>
<th>Appendix</th>
<th>Title</th>
<th>Page No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appendix A</td>
<td>Detailed Flowsheets</td>
<td>118</td>
</tr>
<tr>
<td>Appendix B</td>
<td>Run Descriptions</td>
<td>129</td>
</tr>
<tr>
<td>Appendix C</td>
<td>Off-Gas Handling</td>
<td>170</td>
</tr>
<tr>
<td>Appendix D</td>
<td>Decontamination Experience</td>
<td>177</td>
</tr>
<tr>
<td>Appendix E</td>
<td>Detailed Run Sheets</td>
<td>184</td>
</tr>
</tbody>
</table>
APPENDIX A

DETAILED FLOWSHEETS

1. Chemical - The RaLa Chemical Flowsheet is given in Figure 29

2. Engineering - Flow diagrams are given in Figures 30, 31, 32, and 33
FIG. 29
RALA CHEMICAL FLOWSHEET - DWG. No. CPP-C-1544-RI

NOTES:
(1) CIRCLED NUMBERS INDICATE OPERATING SEQUENCE.
(2) HEAVY LINES INDICATE PATH OF BARIUM.
(3) PERCENTAGES REPRESENT PERCENT OF COMPONENT INITIALLY CHARGED.
APPENDIX B

RUN DESCRIPTIONS

A. Runs Involving Unirradiated Material

1. General

a. Run Nos. 1, 2

Centrifuge skimming was performed at the machines with the remote operating levers disconnected. A tygon tube was attached to the skimmer outlet for physically routing skimmed supernates to a graduated cylinder for accurate volume determinations and sampling. Waste supernates thus collected generally were not re-introduced to the system but were held for observation of clarity and precipitate formation tendency. Product solution was not dried but was collected in the same manner. A spike solution containing active barium and strontium tracer was added to the caustic residue following dissolver solution centrifugation.

b. Run No. 3

Same as Run Nos. 1, 2 except that product solution was dried after being collected in a graduated cylinder.

c. Run No. 4

Same as Run No. 3 except that centrifuge skimming was performed using the remote operating levers.

d. Run No. 5

Same as Run No. 4 except for a flowsheet change in the first nitrate precipitation step.

e. Run No. 6

Same as Run No. 5 except for additional washing of dissolver cake to remove excess aluminum and except for planned large skimmer heels to determine process effects.

f. Run Nos. 7, 8

Skimming was performed using the remote skimmer levers which were modified to include a scale to facilitate leaving pre-determined heel volumes in the centrifuge bowl. The original
small skimmer kidneys had been removed and a tygon tube had been attached to each skimmer outlet for physically routing skimmed supernates to a graduated cylinder. Barium and strontium tracers were used to facilitate analytical requirements.

g. **Run No. 9**

Same as Run Nos. 7, 8 except that use of pH 4.0 water for washing chromate precipitates was initiated. Also in this run, two boildown steps were used in an attempt to remove silica more effectively. However, silica removal was not significantly improved.

h. **Run No. 10, 11, 12, 13, 14, 15**

For these runs the revised skimmer kidneys had been permanently installed on the centrifuges. All centrifuge piping connections had been welded. A lubrication system had been installed for the centrifuge drive buttons to minimize friction and attendant bowl sway. Extensive sampling was not possible due to permanently welded skimmer discharge lines.

2. **Assembly Charging**

a. **Run No. 1**

Unfortunately the mock assembly was not weighed before charging due to an erroneous assumption that mock assemblies duplicated actual enriched assemblies. The assumed weight was about 1.0 kg too low and resulted in insufficient caustic solution being charged to provide the proper NaOH; Al mole ratio. The assembly was charged by hand to a dry carrier-charger placed over the dissolver slug chute. The assembly was dropped from the carrier-charger into 23 liters of water in the dissolver. This is the calculated minimum amount of water required to cover the assembly. The carrier-charger and dissolver slide valve operated satisfactorily.

b. **Run Nos. 2, 3, 4, 5, 6, 7, 8, 9**

A weighed mock assembly was charged by hand to the dissolver which contained 23 liters of water.

c. **Run Nos. 10, 11, 12, 13, 14, 15**

A weighed mock assembly was charged by hand to the water filled carrier-charger. Water was drained to waste just prior to charging the assembly to the dissolver.
3. **Caustic Addition**

a. **Run No. 1**

An attempt made to drain the caustic batch by gravity from the caustic tank, PM-120-0, to the dissolver, L-101, resulted in a flow rate of only 0.5 liter per minute. Because of this low flow rate, the final portion of caustic solution was pumped into the dissolver by caustic metering pump, PM-220-1, at a maximum rate of 1.5 liters per minute. Unfortunately, the dissolution reaction was allowed to start before all the caustic had been added to the dissolver. This resulted in a caustic deficiency which produced an insoluble aluminate formation which caused trouble throughout the remainder of the run.

b. **Run Nos. 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15**

The dissolver temperature was held below 20°C while caustic solution was added by the caustic metering pump at the maximum rate of 1.5 liters per minute. During caustic addition the dissolver was nitrogen purged to expel air. In some runs the off-gas hydrogen analyzer indicated that the dissolution reaction began as soon as caustic solution reached the dissolver. However, the hydrogen concentration in the off-gas did not exceed 2 percent during the addition period.

4. **Dissolution and Digestion**

a. **Run No. 1**

An attempt to control the dissolver temperature by hand in order to provide an even hydrogen evolution rate was not successful. The hydrogen analyzer in the off-gas line was inoperable during the main dissolution period. The off-gas rotameter was somewhat unreliable also. After the off-gas rotameter indicated that no gas was being evolved and after a suitable digestion period, the transfer of dissolver solution to the centrifuge feed pot was started.

b. **Run No. 2**

Hand temperature control of the dissolution reaction was again unsuccessful. However satisfactory dissolution was accomplished. During the main reaction period off-gas flow varied from 30 to 60 percent of off-gas rotameter scale (20 cfm maximum) and hydrogen concentration varied from 35 to 65 percent. The off-gas rotameter indicated that the dissolution reaction was complete long before the hydrogen concentration dropped to zero. This situation was caused by a pocket of hydrogen which had collected
in the slug chute above the off-gas outlet line. When nitrogen purge was admitted to the top of the slug chute, the hydrogen concentration in the off-gas dropped rapidly. Digestion of the dissolver contents at 100°C was maintained until the off-gas hydrogen concentration fell to zero. This insured complete fuel element dissolution.

c. Run No. 3

Hand temperature control was not precise but dissolution was adequate. The hydrogen analyzer was inoperative during the entire run. Nitrogen purge of the slug chute was maintained to prevent a hydrogen pocket. A digestion period longer than usual was necessitated by a centrifuge power failure.

d. Run No. 4

Same as Run No. 3 except that the hydrogen analyzer was made operable during the digestion period and no centrifuge power failure was experienced.

e. Run No. 5

No attempt was made to control the dissolution reaction which is exothermic. Even though the reaction was allowed to run "wild" no serious consequences resulted. The bulk of the reaction occurred within a 15-minute period. The hydrogen analyzer operated satisfactorily.

f. Run No. 6

Successful attempts were made, after an initial off-gas surge of 8 cfm, to control hydrogen evolution at 3 cfm during dissolution. Steam, rather than water, was required in the dissolver jacket to maintain the desired rate. The hydrogen analyzer operated satisfactorily.

g. Run No. 7

After all caustic had been charged, the dissolver was steam heated to speed dissolution. Precise off-gas evolution rate control was not attempted. The bulk of the dissolution reaction had occurred by the time dissolver temperature reached 80°C. Thereafter, steam was used to raise and maintain the temperature at 100°C. The hydrogen analyzer gave a faulty reading (1 percent hydrogen) after the reaction was complete. This was determined by introducing reference gas to the sample gas filament.
h. **Run No. 8**

This run produced the most peculiar dissolution results obtained to date. The dummy assembly dissolved much more slowly than previous assemblies. Off-gas evolution was very erratic as was hydrogen concentration in the off-gas. The exact reason for this is unknown. A reasonable explanation may lie in the fact that the aluminum in the assembly was perhaps purer than before. Certain impurities in aluminum do tend to increase the dissolution reaction with sodium hydroxide.

The hydrogen analyzer was found to give false readings as in Run No. 7. The dissolution and digestion period required an hour longer than usual due to slow reaction and false hydrogen analyzer readings.

i. **Run Nos. 9, 10, 11, 12, 13, 14, 15**

Dissolution was adequate in all runs. The hydrogen analyzer functioned during all runs. An even evolution of off-gas was not obtained in any of these runs. Furthermore the off-gas rotameter had a tendency to stick, thereby providing erroneous flow indication.

5. **Dissolver Solution Transfer**

a. **Run No. 1**

Considerable plugging of the transfer jet and/or feed pot orifice occurred. This was due to metal heel and/or aluminum precipitate in the dissolver. Plugs could usually be removed by applying air pressure downstream from the feed pot orifice. This, however, resulted in one undesirable feature. Considerable air blew out the other end of the feed line which terminates in the centrifuge bowl. This air escapes to the cell around the shaft opening in the centrifuge top plate. Centrifuge venting was thus demonstrated to be inadequate.

Some plugs could not be removed by air pressure. At times it was necessary physically to tap the feed pot and jet. One plug required disconnection of temporary tygon tubing in the jet line for plug removal. All of these plugs were caused by poor dissolution conditions.

During transfer of dissolver solution, the hydrogen analyzer was made operable and indicated that a small percentage of hydrogen still remained in the dissolver. This could have been
caused by a hydrogen pocket in the slug chute or by incomplete assembly dissolution. At any rate the particulates of excessive size slowed the dissolver solution transfer rate to about half design value (500 ml/min).

b. Run No. 2

Dissolver solution temperature was allowed to fall from 100°C to 75°C during transfer to the centrifuge. No feed pot orifice or jet plugging occurred during transfer of dissolver solution proper. An apparent plug during the first wash was removed by applying air pressure on the downstream side of the feed pot orifice. The orifice flow rate was determined to be 590 ml/min.

c. Run No. 3

Same as Run No. 2 except that the dissolver solution temperature was maintained near 100°C during transfer.

d. Run Nos. 4, 5

One feed pot plug occurred during dissolver solution transfer. It was cleared by back-flushing the feed pot orifice with high pressure air. No plugs occurred during dissolver wash transfers.

e. Run No. 6

A feed pot plug occurred during transfer of dissolver wash. Air back-flushing did not clear the plug and it was necessary to tap the feed pot to clear the plug.

f. Run No. 7

No feed pot plugging occurred during transfer of dissolver solution proper. The transfer of caustic dissolver wash solution, however, was too slow, indicating a partial feed pot plug. Air pressure applied to the feed pot orifice restored flow to normal rate.

g. Run No. 8

No plugs occurred during transfer operations. The extra long dissolution and digestion period insured the absence of undissolved assembly particles.

h. Run Nos. 9, 10, 11, 12, 13, 14, 15

In Run No. 10 the dissolver jet and/or feed pot was partially plugged. Following this run the dissolver jet was removed
for cleaning and was re-installed prior to Run No. 11. In some remaining runs partial plugging occurred but all plugs were cleared by the application of air pressure to the feed pot discharge line.

6. **Centrifugation of Dissolver Solution**

   a. **Run No. 1**

   The centrifuge was maintained hot (80 - 100°C) during dissolver solution centrifugation. Steam on the stationary case jacket was found to cause vaporization of aluminate supernate solution from the rotating bowl which collected in centrifuge condenser L-350. When steam and water were mixed to provide hot water to the case jacket, no vaporization occurred. Use of steam only in the rotating bowl jacket caused a volume increase in the centrifuge condenser.

   When all dissolver solution had been processed and the 5-liter residual centrifuge holdup volume had been skimmed out, the centrifuge was stopped for inspection of the cake. The major portion of the cake was found at the outer lower corner of the centrifuge bowl. A thin cake film was in evidence over the remainder of the bowl wall.

   Three water washes were routed through the dissolver, feed pot, and centrifuge. Each was skimmed off separately. The centrifuge was stopped after each wash was skimmed. This proved to be poor procedure because the cake tended to migrate toward the top of the bowl wall. This caused a small amount of cake to be lost during the skimming operation because no heel volume was left in the bowl.

   Because of the slow feed rate during centrifugation, an excessive amount of bowl heating water was collected with the aluminate supernate. This caused the caustic waste tank to overflow. Some waste solution was thus lost before sampling.

   b. **Run No. 2**

   Hot water was supplied the centrifuge case jacket but none was supplied the rotating bowl jacket. This represented an attempt to reduce the aluminate waste volume since bowl jacket water joins the aluminate supernate in the centrifuge case. As a result the centrifugation temperature was not maintained at the recommended 100°C but instead, varied from 35 - 77°C.

   Three water washes were used following dissolver solution transfer. The centrifuge was not stopped until the final wash
had been skimmed. This precaution minimized cake migration and cake loss out the skimmer line. As before, the major portion of the cake was found at the outer lower corner of the centrifuge bowl. In cross section the cake is triangular, extending up the wall about 1/2 inch and back from the wall on the bowl bottom about 3/4 inch. A thin film of solids was distributed over the remainder of the bowl wall.

The relatively low centrifugation temperature apparently caused no serious consequences. Analytical results showed a uranium loss of only 1.44 percent in the aluminate supernate.

c. **Run No. 3**

Hot water was supplied to the rotating bowl jacket as well as to the centrifuge case jacket. The centrifuge was not stopped until the final wash was skimmed. The cake collected principally at the lower outer corner of the bowl. Analytical results indicated a 3.5 percent uranium loss in the aluminate supernate resulting from this centrifugation step.

d. **Run No. 4**

Same as previous run except that uranium loss was 6.7 percent.

e. **Run No. 5**

Same as previous run except that uranium loss was 12 percent.

f. **Run No. 6**

Same as previous run except that uranium loss was 3.5 percent. Additional water washing of cake was performed. Centrifuge bowl was stopped after each skimming operation. Large skimmer heels were left to minimize cake loss.

g. **Run No. 7**

During centrifugation, the temperature was maintained at 70°C by admitting steam to the rotating bowl jacket. Higher temperatures cannot be maintained because the additional steam required results in excessive condensate dilution of centrifuge bowl contents.

One caustic wash and five water washes were used on the caustic residue. In each skimming operation a 300 ml liquid heel was left to preclude cake removal. The bowl was stopped and agitated each time a new wash solution was added.

Uranium loss to the aluminate supernate amounted to 1/2 percent of the uranium charged.
h. **Run No. 8**

Too much hot water was used in heating the centrifuge bowl. This resulted in excessive volume in the caustic waste tank which collects aluminate supernate as well as heating water. Washes and skimmer heels were used as in the previous run.

Uranium loss to the aluminate supernate amounted to 7.3 percent of the uranium charged.

i. **Run Nos. 9, 10, 11, 12, 13, 14, 15**

Adequate centrifugation was obtained in all these runs except Run No. 12 in which 22 percent of the charged uranium was lost to the sodium aluminate supernate. No explanation is known for this high loss. In other runs the uranium loss in this step ranged from 2.8 to 8.9 percent.

7. **Neutralization of Aluminate Supernate**

a. **Run No. 1**

A quantity of nitric acid was added to caustic waste tank L-108 before the run was started. Insoluble aluminum precipitate was found in the neutralized aluminate supernate. This was due to poor assembly dissolution conditions and insufficient acid. Proper technique requires 0.5N excess acid after neutralization. This was not obtained because of the large volumes of centrifuge bowl heating water used in the extra-long centrifugation period which resulted from feed pot plugging.

b. **Run No. 2**

Neutralizing acid was not introduced until the aluminate supernate had been collected and diluted twenty-fold in caustic waste tank L-108. The resulting neutralized solution was clear and gave no evidence of precipitate formation.

Through an oversight cooling water was not supplied to the caustic tank jacket. The resulting solution temperature rise caused excessive solution pickup by the sparge air. The hot moisture-laden sparge air was cooled in the vent piping causing solution buildup in several vessels connected to the vent header. This situation was not discovered until after the sparger was left on all night. Neutralization was the last step performed before stopping operations for the day.
c. **Run Nos. 3, 4, 5, 6, 7**

In each run a calculated quantity of 60 percent HNO₃ was added to the aluminate supernate to acidify the solution to 0.5N acid. No known precipitation problems were encountered in the neutralized aluminate solution.

d. **Run No. 8**

Neutralization was performed as in Run No. 7. When this neutralized solution was transferred by steam jet to the process equipment waste system, it was not known that a new valve had been installed (at CPP operations instigation) in the jet vent header to block off cell L jets from the header. It was discovered too late that this valve was closed. Thus, the jet vent header could vent only to other connected jet lines. This resulted in gradual volume increases in nearly all cell L vessels. As indicated, the cause at the time of occurrence was unknown. However, no serious consequences resulted. The main inconvenience experienced was erratic behavior of the nitrate boildown operation which was being conducted at the time.

e. **Run Nos. 9, 10, 11, 12, 13, 14, 15**

During these runs the neutralization procedure was changed from adding neutralizing acid to caustic waste to introducing caustic waste to a waste tank containing neutralizing acid. This procedure permits charging of neutralizing acid to an empty vessel (L-109) before a run is started. Caustic waste collected in L-108 can then be jetted to L-109 at any convenient time during the run.

8. **Silica Separation**

a. **Run No. 1**

The acid dissolution of the caustic residue was performed satisfactorily except for reaction gases escaping to the cell. No shaft seals were installed on the centrifuge due to the seals being reworked.

Skimming of the combined supernate to leave silica in the bowl was not entirely satisfactory. A flocculent precipitate was carried along with the supernate. This probably was a combination of silica and aluminum compound particulates. The silica remaining in the bowl was flushed out with water. Inspection showed a scale (similar to boiler scale) to be clinging to the bowl wall following silica removal. It was impossible
to remove this scale which apparently was aluminum oxide resulting from the poor assembly dissolution conditions. Because of the scale, aluminum appeared in every subsequent supernate including product solution. After the run was completed, the scale was removed by treatment with hot caustic solution. This caused flaking of the scale which was then removed by hand.

b. Run No. 2

Acid dissolution of the caustic residue was performed satisfactorily. A flocculent silica-type precipitate was skimmed off with the combined supernate even though a ten-minute centrifugation period was allowed for silica separation.

The silica remaining in the bowl was flushed out satisfactorily. The operating technique employed continuous water flush plus air sparging through the cake cutter nozzle. Flush water overflowed the bowl into the centrifuge case and flowed to caustic waste tank L-108. This technique increased waste volume somewhat but provided good silica removal from the centrifuge bowl. It was originally intended that the centrifuge jet be used to facilitate silica removal. However, this jet is so small (0.2 liters/min) it is not effective in this operation.

Following silica removal the bowl was clean and no scale remained as in the first run.

c. Run Nos. 3, 4

Acid dissolution of the caustic residue was performed satisfactorily. The resulting supernate was not collected in a graduate but was skimmed directly to the supernate tank, L-105. A clean separation between silica and supernate was not obtained due to the flocculent nature of silica and possibly due to skimming too near the bowl wall. Silica remaining in the bowl was flushed out satisfactorily with water.

c. Run Nos. 5, 6

An attempt was made to leave up to 200 ml heel when skimming supernate and washes. This should have caused less silica to follow the product-containing solution. However, due to centrifuge shaft movement and lack of a skimmer position indicator, it was impossible to determine just what heel volume remained after skimming.
e. **Run No. 7**

Acid dissolution of the caustic residue was performed satisfactorily. The resulting supernate was skimmed to the supernate tank. No solids appeared in the skimmed supernate and washes because a known 300 ml liquid heel was left in the bowl at the end of each skimming operation. The resulting higher barium loss was partially offset by an additional wash operation.

f. **Run No. 8**

Through an oversight, the spike solution containing tracer was not added to the caustic residue. Tracer was added later along with the barium carrier solution prior to boildown.

Due to insufficient time to complete the first day's activities, washing of the silica cake was not completed. A wash solution was left in the bowl over night along with the silica cake. This proved to be poor procedure because the gummy cake adhered tenaciously to the bowl wall and bottom and was very difficult to remove on the following day. Normal technique for silica removal was inadequate. Manual cleaning was necessary. Wash solution left in the bowl apparently evaporated over night allowing the cake to dry.

g. **Run No. 9**

A special procedure was used in this run in an attempt to remove silica more effectively. In run No. 8 it was established that considerable silica is soluble in the 6M nitric acid used to dissolve uranium and fission products away from the silica cake. The silica thus following the nitric acid supernate subsequently precipitates as silica gel during the nitrate boildown step. This silica gel is picked up by the skimmer during the uranium supernate removal.

In view of the fact that silica gel forms during the nitrate boildown, a special boildown was added in this run in an attempt to precipitate silica completely prior to subsequent processing. The silica cake thus formed was leached with water to remove barium and strontium. Apparently considerable silica carried along with barium and strontium in the leach water because in the subsequent boildown, silica gel again was formed. Volume of gel in the uranium supernate was 175 ml. In previous runs with only one boildown step the volume of gel ranged from 200 to 300 ml. It was therefore concluded that the extra boildown did not accomplish the desired effect.
h. **Run Nos. 10, 11, 12, 13, 14, 15**

A new procedure was established in Run No. 13 for removing silica and insoluble particulates from the centrifuge bowl. This new procedure consists of intermittent cake cutting using high pressure water followed by skimming at low speed to remove slurry. The former procedure which was inadequate involved flooding the bowl with water while sparging with air through the cake cutter nozzle.

9. **Nitrate Precipitation**

a. **Run No. 1**

The combined supernate from the silica separation step was diluted to 5 liters and introduced to centrifuge feed tank L-156 for evaporation to a 0.7 ± 0.1 liter. This evaporation step proved to be the most troublesome part of the process. The feed tank is a slender vessel (4-inch Schedule 80 pipe) containing both inner and outer jackets. This large heating area per unit volume makes the evaporation operation very delicate, especially as the boiling point of the solution is reached. A too rapid steam increase here caused an estimated 35 percent of the solution to be carried over into feed tank condenser, L-356. Normally, solution so carried over could be re-routed to the feed tank by way of PEW sampling tank L-110 and supernate tank L-105. In this run, however, the PEW sampling tank contained another waste solution and could not be used. Thus, the 35 percent carry-over loss could not be recovered.

Because of operation inexperience the evaporation step was not carried far enough and the resulting concentrate was about 2.4 times the proper volume. Because of this the subsequent addition of fuming nitric acid did not effect complete precipitation of barium and strontium nitrate. Consequently, considerable barium was lost in the uranium supernate.

The supernate contained considerable flocculent precipitates of aluminum and silica. It was later determined that another contaminant was stop-cock grease from the addition burettes used for nitric acid addition. Use of these burettes was abandoned in later runs.

b. **Run No. 2**

Evaporation of the diluted combined supernate from the previous centrifugation again caused considerable operating difficulty.
Before boildown the solution volume was 9 liters instead of 5 liters. It was determined that the extra 4 liters came from water in pressure pot, FM-856. This pressure pot had been used the day before for water addition to feed tank L-156. Through an oversight the feed pot was not drained before its required use in the process. This demonstrates the necessity for careful pre-operational checking.

The large volume in feed tank L-156, where evaporation is performed, caused some solution to be carried over to feed tank condenser L-356 when steam heat was applied. The solution thus lost was subsequently recovered and re-introduced to the feed tank after partial evaporation of the original solution.

Because of the large solution volume the feed tank condenser could not hold all the condensate. Thus, it was necessary to empty the condenser near the middle of the evaporation operation. This led to difficulties in determining completion of the evaporation step since concentrate volume was determined by subtracting condensate volume from original volume. The feed tank gravity and level chart was finally relied upon for evaluation of concentration volume. As in Run No. 1 the evaporation step was stopped too soon resulting in excessive concentrate volume. However, this fact could not be determined until the subsequent centrifuging and skimming operation. In this run the concentrate volume was about one liter instead of the required 0.7 + 0.1 liter. High barium and strontium losses in the skimmed nitrate supernate resulted.

c. Run No. 3

This step caused difficulties which resulted in high barium loss. The boildown operation was carried too far but this was not discovered until later. No carryover of solution to the condenser was experienced. The concentrate plugged the line to the centrifuge due to being too concentrated and thus extremely viscous. Jarring the line finally resulted in starting the flow.

In heating the centrifuge bowl, too much live steam was used resulting in condensate diluting the bowl contents. The diluted nitrate solution caused a barium loss of 43 percent in the subsequent supernate removal step.

d. Run No. 4

Again the boildown operation was carried too far due to false instrument readings. Centrifuge heating was accomplished by admitting steam to the case jacket only. This prevented any condensate buildup in the centrifuge bowl. A large amount of fluffy precipitate was in the uranium supernate.
e. **Run No. 5**

A new flowsheet change was instituted. This change involved boiling nitrate solution down to 1.5 liter rather than 0.7 liter followed by addition of 1.0 liter fuming nitric acid rather than 0.5 liter. Also, the precipitation was performed at room temperature rather than at 50°C. However, skimming the supernate revealed a fluffy precipitate near the bowl wall. Due to the possibility of barium being occluded in this precipitate, the entire supernate was re-introduced to the centrifuge. The temperature was raised to 50°C and the solution centrifuged and skimmed again. The same precipitate was scooped up again by the skimmer but the quantity was slightly less.

f. **Run No. 6**

The flowsheet change of the previous run was followed again. The nitrate precipitation was conducted at room temperature. In the subsequent centrifugation considerably less of the fluffy precipitate appeared in the skimmed supernate.

g. **Run No. 7**

The nitrate boildown required longer than usual because the concentrate was cooled for accurate volume determination before the flowsheet volume of concentrate was reached. Additional time thus was required in reheating the solution to boiling.

The skimmed supernate again contained the usual fluffy precipitate which was scooped up near the centrifuge bowl wall. About 300 ml of extra solution volume appeared in the supernate. Source of this dilution is unknown. This dilution caused a barium loss of 13 percent in the uranium supernate.

h. **Run No. 8**

The nitrate boildown step was conducted without incident. Barium in the condensate was negligible.

The uranium supernate from this step again contained a characteristic fluffy precipitate which was analyzed as silica. A sample of shaken supernate contained 0.99 g/l SiO₂. Filtered supernate contained 0.026 g/l of SiO₂. The difference, 0.964 g/l, thus represents precipitated SiO₂ concentration. Later runs demonstrated that the fluffy precipitate of SiO₂ could be tolerated without adversely affecting uranium solution handling.
i. **Run No. 9**

As indicated in the previous section, two boildown steps were conducted in this run. Nitrates precipitated in the first boildown were dissolved away from insolubles to provide solution for the second boildown. The extra boildown increased processing time by four hours. Barium loss was not excessive.

h. **Run Nos. 10, 11, 12, 13, 14, 15**

In these runs it was found that the silica gel formed during the nitrate boildown step did not plug the skimmer kidney outlet line. Thus, presence of silica gel in the uranium supernate is not objectionable from the standpoint of fluid flow.

Another fear due to the presence of silica gel involved the tendency of silica gel to collect on the skimmer pickup nozzle and absorb elements which could contaminate product solution. This probability was lessened by using the centrifuge de-contamination spray for two of the final nitrate washes. This spray washes down all probes and should remove any silica gel from the skimmer.

Another unknown was the effect of silica on uranium recovery in ICPP. No deleterious effects were noted in connection with processing cold enriched uranium from the startup program. Thus it appears that even though silica gel forms during the nitrate boildown step, no particular problems are involved.

In Run No. 10 high barium losses were experienced in the nitrate boildown condensate and in uranium supernate for no apparent reason.

10. **Barium Chromate Precipitation**

a. **Run No. 1**

The most important operating requirement in this step is completion within ten minutes to prevent excessive chromate ion decomposition. This requirement was not met in this run because samples were physically taken from the centrifuge bowl for pH measurement which consumed extra time. The original flowsheet quantity of sodium acetate buffer was not adequate to bring the chromate solution up to the proper pH range (4.25\textasciitilde0.25). Therefore, the buffer volume was increased to provide the proper pH in order to precipitate barium and leave strontium in solution. Barium loss in the chromate supernate was quite low. The strontium concentration was quite low also, due to high strontium
loss in the previous uranium supernate. This first chromate supernate is the one which is to be held for later strontium-90 recovery.

In the second chromate precipitation step the original flowsheet quantity of buffer caused a high pH (5.2). However, this was not considered serious. Buffer volume for subsequent runs was reduced.

Supernates from both chromate precipitation steps contained a flocculent precipitate.

b. **Run No. 2**

Adjusted buffer volume based on the previous run findings did not result in the proper pH. Additional buffer was required. No attempt was made to complete the chromate precipitation within the prescribed ten minutes because of the necessary pH checking and adjustment.

The chromate supernate was clear but a precipitate formed upon standing. Excessive supernate volume was experienced. It was discovered that water had been left in a pressure pot used during the silica removal operation. This demonstrates the necessity for continual alertness throughout the process. Barium loss in the first chromate supernate was slightly high (3.6 percent).

In the second chromate precipitation the adjusted buffer volume based on the previous run findings was adequate. The supernate from the second chromate precipitation step was clear but a precipitate formed upon standing. The precipitate so formed was probably barium and strontium since it dissolved when acidified. Barium loss was not excessive.

c. **Run Nos. 3, 4**

Adjusted buffer volume based on the previous run findings resulted in the proper pH during the first chromate precipitation step. However, additional buffer was required during the second chromate step. The reason for this inadequacy is unknown.

d. **Run No. 5**

No pH adjustments were necessary beyond normal flowsheet buffer quantities.
e. **Run No. 6**

It was necessary to add additional sodium acetate buffer during the first chromate precipitation step in order to bring the pH to the proper range. This was due to excessive skimmer acid heel left from the previous uranium supernate separation.

g. **Run No. 9**

This run was the last in which chromate solution samples were taken directly from the centrifuge bowl for pH measurements. The first chromate solution was pH 4.7 and the second was 4.9. These are slightly higher than the 4.5 specified but not excessively so.

**11. Metathesis and Final Nitrate Precipitation**

a. **Run No. 1**

No difficulties were encountered here except that supernates contained a flocculent precipitate as before. Barium loss in the nitrate supernates was not excessive.

b. **Run No. 2**

In these steps no heating was used. This fact plus too much haste to complete processing resulted in incomplete conversion of barium chromate to barium nitrate. Barium loss in the nitrate supernate was not excessive.

Heating was shown to be necessary during metathesis as well as in cake dissolution prior to final nitrate precipitation.
c. **Run No. 3**

No known difficulties were encountered in the conversion of barium chromate to barium nitrate.

d. **Run Nos. 4, 5**

After metathesis, the nitrate cake was dissolved in water and skimmed to the supernate tank for storage over night. The following day this solution was boiled down to normal volume before the final nitrate precipitation. This procedure represented a departure from previous operation but was planned to determine feasibility in case processing is to be limited to day shift only.

e. **Run No. 6**

The skimmer heel left in the bowl was too large. This allowed excessive contaminants to follow barium into product solution.

f. **Run No. 7**

Through some operational error, the fuming nitric acid used for metathesis became diluted with approximately 350 ml water in the centrifuge bowl. This dilution caused a barium loss of 16 percent in the nitrate supernate.

Two extra nitric acid washes were used to improve product purity.

g. **Run No. 8**

No known difficulties were encountered in the conversion of barium chromate to barium nitrate. Extra nitric acid washes were used to improve product purity as in Run No. 7.

h. **Run Nos. 9, 10, 11, 12, 13, 14, 15**

No known difficulties were encountered in any run. Beginning with Run No. 9, two of the final nitrate washes were added via the centrifuge bowl decontamination spray to wash probes and skimmers.

12. **Product**

a. **Run No. 1**

Barium nitrate product solution was not dried due to the drying station being disassembled for modifications. Instead, product solution and washes were skimmed to a graduated cylinder for volume measurement and sampling. The product skimmer left an
unskimmable heel of 350 ml. This heel was removed by the regular skimmer and collected and sampled separately.

Barium recovery in the product was extremely low due to previous high losses.

b. **Run No. 2**

As in the previous run, product solution was not dried but was skimmed off in two portions to graduated cylinders. The product solution had a distinct orange color characteristic of chromate solutions. This resulted from the incomplete conversion mentioned in the previous section.

Barium recovery in the product was fair (64.5 percent) but contamination was high due to chromium and sodium.

c. **Run No. 3**

Product solution was skimmed to a graduate for sampling. The product was not clear but contained a light colloidal precipitate which settled after standing over a weekend. This precipitate apparently was silica. Before being dried the product was filtered to remove this precipitate and a clear supernate resulted.

A tygon gasket was tried on the product cup to provide a seal between cup and dryer head. The gasket was softened by heat to the point that it was not serviceable.

It was determined that inadequate condensing surface was provided in the original design as indicated by vapor loss out the condenser vent line.

The product cup is too small to receive all the aqueous product solution at one time. It is therefore necessary to add the solution in increments. The condenser level was used to determine the proper time to add each successive increment. Because of errors caused by vapor loss, the additions in this run were not always made properly. As final dryness was approached, full heat was maintained on the drying station coil. This proved to be poor procedure since considerable crystal popping and attendant dusting occurred. Additional condensing area was subsequently provided.

d. **Run No. 4**

All product solution was skimmed to a graduate for sampling. As before, the product was not completely clear but contained a
light flocculent precipitate of silica. After sampling, the product solution was dried in the product cup. Maximum heat on the drying station coils caused a 34.8 percent barium loss to the drying station condenser.

As final dryness was approached, the heat was turned down from maximum to half that value to eliminate crystal popping and dusting.

The dried product was generally white but contained some colored matter. Distribution of product in the cup was fairly good. Some dried product remained on the dryer head thermocouple probe after the cup was lowered away from the head. This was probably caused by the excessive heat applied during the drying operation.

e. **Run No. 5**

Less heat was applied to the drying station coils than in Run No. 4. Barium loss to the condensate was only 1 percent. Dried product was not found on the thermocouple probe.

f. **Run No. 6**

Too much product solution was introduced to the product cup. Even though low heat was used in drying, a carryover condition resulted in 4 percent barium loss to the condensate.

g. **Run No. 7**

Product solution was collected in a graduate for sampling. A 100 ml liquid heel was left in the centrifuge at the end of each product skimming operation. This solution was the clearest obtained to date. It was not filtered prior to analysis.

Dried product contained a small amount of brown residue on top of the white crystalline barium nitrate. In spite of this, the product met purity specifications but recovery was fairly low (52 percent).

Drying with 60 percent of full power on the drying station coils caused considerable spattering within the product cup. However, only 0.4 percent of charged barium was found in the condensate produced in the drying operation.

A technique was developed, based on dryer head temperature, for determining the proper time for successive batch additions of product solution to the drying station.
A small amount of dried product was found on the dryer head thermocouple probe.

h. Run No. 8

As in the previous run, a 100 ml liquid heel was left in the centrifuge at the end of each product skimming operation. This insured absence of particulates in product. Product solution was pale yellow in color indicating chromate contamination. Analysis, however, revealed only 12 mg. chromium. Product recovery was excellent (94 percent) and purity was good.

Drying with 50 percent of full power on the drying station coils caused some spattering within the product cup. Small particles of product were apparent all over the inside surface of the cup. The bulk of the product was concentrated in the cone tip of the product cup.

Dried product contained a small amount of brown residue on the cup sides immediately above the white product. A small amount of dried product was again found on the dryer head thermocouple probe.

i. Run No. 9

The first portion of product solution was accidentally skimmed to the cell floor due to a tygon tube on the skimmer outlet becoming disengaged. Exact product loss is unknown. Remaining product solution was collected in a graduated cylinder for sampling before drying.

j. Run No. 10

Product solution was not sampled prior to drying. Dried product which was brown in color was dissolved in water for sampling. Recovery was 56.2 percent due to high losses in the uranium supernate and in the nitrate boildown condensate.

k. Run No. 11

Product solution was dried and then dissolved in water for sampling. A brown residue was again apparent in the product. Recovery was extremely low (19.6 percent) due presumably to a high loss in one of the waste supernates. Qualitative analysis of the undesirable residue indicated the following elements were present in the order of decreasing concentrations:

Al, Si, Fe, Ti, Cr, Ni, U.
l. Run No. 12

Product solution was collected in a graduated cylinder before drying for sampling. Brown particulates were evident in the product solution. After completion of the run the centrifuge bowl was found to contain several grams of black sticky cake, presumably left from poor silica removal procedure. Dried product was sent to the customer for testing. It contained a reddish-brown residue which was insoluble in both nitric and hydrofluoric acids but soluble in hot concentrated hydrochloric acid. Product recovery was 90 percent.

m. Run No. 13

Product solution was not dried but was collected in a graduated cylinder for sampling. Product solution was yellow in color and contained some particulates. Recovery was 79 percent.

n. Run No. 14

Product solution was dried and then dissolved in water for sampling. The re-dissolved product solution was not clear because of particulates and contained a faint chromate color. After filtration the supernate was quite clear.

o. Run No. 15

Product was dried before sampling. After drying, the product was washed with 75 ml of 70 percent nitric acid. This acid leach was analyzed for contaminants which are listed in Table 5.

13. Run Deficiencies

a. Run No. 1

(1) Inadequate NaOH : Al ratio

   (a) Insufficient caustic added
   (b) Inadequate caustic addition rate

(2) Carry-over loss during nitrate boildown.

(3) High nitrate concentrate volume.

(4) Low pH - first chromate precipitation.

(5) High pH - second chromate precipitation.
(6) Dissolved stopcock grease in nitric acid.
(7) Waste solution loss due to overflowing tanks.

b. Run No. 2
(1) Water in two pressure pots before chemical solution addition.
(2) Carry-over during nitrate boildown.
(3) High nitrate concentrate volume.
(4) Inadequate time and temperature during chromate conversion to nitrate.
(5) Low pH - first chromate precipitation.

c. Run No. 3
(1) Steam condensation in centrifuge vent line and subsequent dilution of nitrate concentrate in centrifuge bowl.
(2) Poor nitrate boildown technique.
(3) Low pH - second chromate precipitation.
(4) Tygon gasket decomposition during product drying.
(5) Poor product drying technique.

d. Run No. 4
(1) Poor nitrate boildown technique.
(2) Low pH - second chromate precipitation.

e. Run No. 5
(1) Excessive flocculent precipitate in uranium supernate.
(2) Rather poor barium recovery and barium balance.

f. Run No. 6
(1) Excessive skimmer heels caused additional pH adjustment as well as excessive contaminants in the product.
(2) Carry-over of product solution to condenser during drying caused by over-filled product cup.
g.  **Run No. 7**

1. Extra volume in uranium supernate caused excessive barium loss.

2. Extra volume in methathesis supernate caused excessive barium loss.

h.  **Run No. 8**

1. Silica cake dried on centrifuge bowl wall overnight.

2. Unvented steam jet vent header caused condensate buildup in cell vessels.

3. A solenoid on one centrifuge skimmer operating rod interlock became overheated and had to be disconnected.

4. Tracer spike was added too late in the process to provide typical barium losses during the silica removal step.

i.  **Run No. 9**

1. High barium loss (13.3 percent) in uranium supernate; reason unknown.

2. Temporary tubing failure caused high product loss (40-50) percent.

j.  **Run No. 10**

1. Partial plug in dissolver jet and/or dissolver solution feed pot.

2. Through an oversight, the Ba-Sr-La spike solution was added just prior to the nitrate boildown step instead of prior to acid dissolution of caustic cake.

3. High barium loss in the nitrate boildown condensate; reason unknown.

4. High barium loss in the uranium supernate; reason unknown.

5. Dried product contained considerable discoloration.

k.  **Run No. 11**

1. Extremely low barium recovery and balance; reason unknown.
(2) Dried product contained considerable discoloration.

(3) Inadequate silica removal procedure.

1. Run No. 12
   (1) High uranium loss in aluminate supernate; reason unknown.
   (2) Inadequate silica removal procedure.

m. Run No. 13
   (1) Off-gas system developed a leak discovered later to be in M cell.
   (2) Operational error at shift change resulted in the first chromate supernate being jetted to the uranium storage tank rather than to the strontium batch tank.
   (3) Operational error resulted in first and second water washes being added together during the first chromate precipitation step.
   (4) Product contained excessive chromate contamination.

n. Run No. 14
   (1) Operational error resulted in extra water being added to the first dissolver caustic wash. No serious consequences resulted except for the additional jetting time required.

   (2) The off-gas rotameter did not function properly during assembly dissolution. Apparently the rotameter contained deposited solids which caused the float to stick.

o. Run No. 15
   (1) Low product recovery; reason unknown.

B. Runs Involving Irradiated Material

1. General

a. Run No. 001-RH

   This run was made to refine remote operating techniques and to detect possible radiation effects. Product contained negligible active barium due to the long assembly cooling time and was not
shipped to the customer. Radioactive iodine and xenon were also negligible. Minimum sampling was performed.

Fuel element data and run dates for these runs are given in Table 6.

b. Run No. 002-RH

This run was made to supply the customer with a requested 5000 curie "trial" batch of barium-140. Of the 3000 curies charged only 833 were recovered as product due to high processing losses. Minimum sampling was performed.

c. Run No. 003-RH

This run was made at customer request to produce up to 10,000 curies of barium-140. The charged element contained 16,250 curies of which 8000 were recovered as product. Extensive sampling was performed.

d. Run No. 001-RP

This run was the first actual production commitment. The 18,000 curies produced was short of the 25,000 curies requested by the customer. Extensive sampling was performed.

e. Run No. 002-RP

This run produced the highest activity product in the startup program. The customer reported receiving 49,000 curies of barium-140. Extensive sampling was performed.

f. Run No. 003-RP

This "routine" production run produced 18,600 curies of barium-140 which was less than the requested 25,000-curie batch. Theoretical assembly burnup was found to be low which accounts for the low activity shipment. Extensive sampling was performed.

g. Run No. 004-RP

A customer request for a barium batch of at least 10,000 curies prompted this run. Actual production was 21,350 curies. A processing delay of ten days was caused by adverse weather conditions. Because of the delay an alternate assembly was used to take advantage of less decay time.
h. **Run No. 005-RP**

A six-day-cooled MTR assembly was charged after a four day delay due to adverse weather conditions. Production was 14,500 curies of barium-140.

2. **Assembly Charging**

a. **Run No. 001-RH**

Assembly No. P-461 containing an estimated 131 grams of uranium and 1 gram of barium-138 was charged to the carrier-charger on November 21, 1956. Water was kept in the carrier-charger for assembly cooling until the assembly was charged to the dissolver on November 24, 1956. There were no difficulties connected with charging, except that the air supply to the slide valve did not cut off automatically when the slide valve was closed after charging. Manual closing was necessary.

b. **Run No. 002-RH**

Assembly No. P-137 containing an estimated 191 grams uranium, 3,033 curies barium-140, and 1,260 curies iodine-131 was charged to the carrier-charger on November 24, 1956. Maximum water level was maintained in the carrier-charger for assembly cooling until the assembly was charged to the dissolver on November 30, 1956. Charging had been tentatively scheduled for November 26, but was delayed four days due to weather conditions being too adverse to permit gas release to the stack.

c. **Run No. 003-RH**

Assembly No. M-238 containing an estimated 158 grams uranium, 16,250 curies barium, and 6,580 curies iodine-131 was charged to the carrier-charger on December 5, 1956. The assembly was charged to the dissolver on the same date. Processing started December 6, 1956. An extra ten-liter volume of water was added to the dissolver to insure complete submergence of the assembly. Radiation level just outside the cell door was 1.05 R/hr. with the assembly in the dissolver.

d. **Run No. 001-RP**

Assembly No. P-126 containing an estimated 143 grams uranium, 38,800 curies barium and 70,000 curies iodine-131 was charged to the carrier-charger on January 29, 1957. Because of poor weather conditions the assembly was not charged to the dissolver until February 1, 1957. While in the carrier-charger, the assembly heated the stagnant cooling water to 70°C before fresh cooling water was added.
e. **Run No. 002-RP**

Assembly No. P-146, containing an estimated 152 grams uranium, 43,700 curies barium and 166,000 curies iodine-131, was charged to the carrier-charger on February 20, 1957. It was charged to the dissolver on the same date. Failure of the carrier-charger drawer operating screw caused a slight delay in the charging operation.

f. **Run No. 003-RP**

Assembly No. P-227 containing an estimated 192 grams uranium, 23,800 curies barium-140 and 11,000 curies iodine-131 was charged to the carrier-charger on April 5, 1957, and was charged to the dissolver on the same date.

g. **Run No. 004-RP**

Assembly No. P-237 containing an estimated 172 grams uranium, 29,150 curies barium-140 and 14,500 curies iodine-131 was charged to the carrier-charger and to the dissolver on May 19, 1957.

h. **Run No. 005-RP**

Assembly No. P-285 containing an estimated 163 grams uranium, 32,000 curies barium-140 and 14,900 curies iodine-131 was charged to the carrier-charger and to the dissolver on June 24, 1957.

3. **Caustic Addition**

In each run the dissolver temperature was held below 20°C during caustic addition. In most runs an indication of slight reaction was observed as soon as caustic addition was begun. This probably was due to localized surface heating of the assembly.

4. **Dissolution and Digestion**

a. **Run No. 001-RH**

While the assembly was in the dissolver, the cell was entered for a limited radiation survey. A radiation level of 80 mR/hr was observed about eight feet above the cell floor. This increased to 450 mR/hr about sixteen feet above the cell floor. An electrician entered the cell to replace new lamps in centrifuge L-450 periscope. The dissolution and digestion operation was conducted without incident.

b. **Run No. 002-RH**

The radiation level just outside the cell door was 100 mR/hr with the assembly in the dissolver. During dissolution, several
300 ml samples of dissolver off-gas were taken. The most radioactive of these read 500 mr/hr at contact. Area A. E. C. radiation surveys indicated that activity in the dissolver off-gas discharging from the stack persisted for about five minutes. During that period the A. E. C. sky scanner instruments read maximum and then dropped to zero.

During digestion the dissolver liquid level dropped from 70.5 to 66 percent of full scale. Simultaneously the scrubber level increased from 51 to 52.7 percent. This would indicate that boiling occurred in the dissolver. However, the dissolver reflux condenser coil outlet temperature did not exceed 18.3°C during digestion, so any vaporized solution should have been condensed.

Dissolution and digestion required three hours.

c. **Run No. 003-RH**

The dissolution reaction started very slowly. The temperature controller was initially operated on manual control rather than automatic. It was later discovered that cooling water was leaking to the dissolver jacket with the controller on manual which accounted for the very slow initial reaction.

The off-gas rotameter was partially plugged during the entire dissolution. Toward the end of dissolution a burst of gas nearly caused the dissolver to pressurize. This burst finally freed the rotameter float as evidenced by a sudden increase in dissolver vacuum.

Off-gas activity during dissolution was practically nil. A counter on the off-gas line read only 0.31 mr/hr.

Approximately 7 percent of charged iodine was found in the scrubber solution after the run was completed.

d. **Run No. 001-RP**

Dissolution was performed without incident. No off-gas rotameter plugging occurred. Dissolver off-gas contained considerable iodine as well as active noble gases as indicated by the stack gas monitor. Limited sampling of dissolver off-gas was performed during dissolution.

Just prior to this run a pressure actuated bypass was installed around the off-gas rotameter.
e. **Run No. 002-RP**

Dissolution was performed rapidly without pressurizing the dissolver. Xenon activity persisted in the dissolver off-gas for about ten minutes after dissolution started. No gas samples were taken during dissolution. The radiation level just outside the cell door was 5 R/hr with the assembly in the dissolver.

f. **Run No. 003-RP**

Dissolution and digestion were performed without incident. Radiation levels were lower than in the previous run.

g. **Run No. 004-RP**

Neither iodine nor xenon activity was detected in the dissolver off-gas. In previous runs xenon always appeared in the dissolver off-gas. Reason for this anomaly is unknown since the gas monitoring system was the same as in previous runs.

h. **Run No. 005-RP**

The hydrogen analyzer did not function properly during the initial dissolution reaction. Xenon activity appeared in evolved hydrogen.

5. **Dissolver Solution Transfer**

a. **Run No. 001-RH**

The dissolver jet and/or feed pot was plugged initially and had to be backflushed with high pressure air before dissolver solution would flow. Three other plugs occurred later during dissolver solution transfer, sodium hydroxide wash transfer, and water wash transfer. All plugs were cleared satisfactorily by high pressure air backflushing.

b. **Run No. 002-RH**

Transfer of dissolver solution was accomplished much faster than previously experienced. Only one feed pot plug was experienced and that occurred during transfer of sodium hydroxide wash solution. The usual backflushing procedure restored flow to normal.
c. Run Nos. 003-RH, 001-RP, 002-RP

Transfer of dissolver solution and all wash solutions was conducted satisfactorily. No plugging was experienced. An off-gas activity increase was detected during jetting in each run. Dissolver solution was jetted at 80°C with 100 psig air.

d. Run No. 003-RP

Same as Run No. 002-RP except that off-gas activity due to iodine did not increase significantly during jetting. Jet air pressure was reduced from 100 psig to 60 psig just prior to this run. Dissolver solution was jetted at 30-60°C. The combination of these two changes is believed to have minimized iodine being stripped from dissolver solution by jet air.

e. Run No. 004-RP

A plugged jet and/or feed pot caused a one hour delay in starting transfer of dissolver solution to the centrifuge. Heating the dissolver to 70°C and backflushing the feed pot orifice with high pressure air finally resulted in normal flow conditions. It is believed that the plug was caused by not decontaminating the system immediately after the previous run. Both outlets from the feed pot apparently were plugged. This allowed the feed pot to become partially filled. Backflush air then caused dissolver solution to be air lifted into the vessel vent system. This was evidenced by a sudden volume increase in PEW sampling tank, L-110, which is the lowest point in the vessel vent system. Maximum volume of dissolver solution thus lost was only 1.2 liter. Barium loss was negligible. No plugs occurred during any of the dissolver wash operations.

f. Run No. 005-RP

At the outset of transfer operations the dissolver temperature was lowered to 40°C. Sluggish jetting resulted until the dissolver temperature was increased to 60°C after backflushing the feed pot orifice with high pressure air. It was also found necessary to heat dissolver washes to prevent feed pot orifice plugging.

6. Centrifugation of Dissolver Solution

a. Run No. 001-RH

The radiation level just outside the cell door was 60 mr during centrifugation. No known difficulties were encountered during centrifugation. Since no active barium was present in the
assembly it was not possible to determine barium loss in the aluminate supernate resulting from dissolver solution centrifugation.

b. **Run No. 002-RH**

The radiation level just outside the cell door was 400 mr during centrifugation. High barium loss may have occurred during dissolver solution centrifugation. Approximately 63 percent of charged barium was found in combined waste streams at the end of the run. Since no intermediate samples were taken it is not known which waste supernate contributed the high barium.

c. **Run No. 003-RH**

Radiation level at the cell door increased from 2 R/hr to 16 R/hr during dissolver solution centrifugation. A sample of the aluminate supernate indicated a barium loss of only 0.26 percent during centrifugation. This was the first barium loss determination in this supernate.

Approximately 70 percent of iodine originally present stayed with the aluminate supernate.

d. **Run No. 001-RP**

Radiation at the cell door increased from 2 R/hr to 150 R/hr during dissolver solution centrifugation. A sample of the aluminate supernate indicated a barium loss of only 0.4 percent during centrifugation.

e. **Run No. 002-RP**

Radiation at the cell door increased from 5 R/hr to 325 R/hr during dissolver solution centrifugation. Barium loss was 0.6 percent during centrifugation.

Condensate from the centrifuge case jacket became contaminated during the centrifugation operation. This would indicate a leak in the case since the condensate system itself is closed.

f. **Run No. 003-RP**

Radiation at the cell door increased from 50 mr/hr to 50 R/hr during dissolver solution centrifugation. Barium loss was 0.1 percent.
As in the previous run, condensate from the centrifuge case jacket became contaminated during centrifugation.

**g. Run No. 004-RP**

Radiation level at the cell door did not exceed 12 R/hr for some unknown reason. A change in operating technique prevented centrifuge case jacket condensate contamination. This change involved avoiding turning the steam off during centrifugation to keep the case jacket pressurized. Any leakage thus was from jacket to centrifuge case rather than in the opposite direction.

Barium loss in the aluminate supernate and washes was 1.3 percent.

**h. Run No. 005-RP**

Radiation at the cell door did not exceed 3 R/hr. However, it is possible that the radiation detection instrument was not operating properly. Steam supplied to the centrifuge case jacket caused some vaporization of aluminate supernate which collected in centrifuge condenser, L-350, and in FEW sampling tank, L-110.

Barium loss in the aluminate supernate and washes was 0.7 percent.

7. **Neutralization of Aluminate Supernate**

**a. Run No. 001-RH**

Neutralization was conducted in an excess acid medium by jetting supernate to Caustic Waste Tank No. 2, L-109, containing the entire batch of neutralizing acid. No difficulties were encountered.

**b. Run No. 002-RH**

Neutralization was conducted simultaneously with acid dissolution of the dissolver cake. Off-gas from both these operations was collected and held for controlled release under favorable weather conditions.

**c. Run No. 003-RH**

Neutralization was conducted simultaneously with acid dissolution of the dissolver cake. Off-gas activity reached a maximum of 320 mr/hr during this operation. Favorable weather conditions permitted venting off-gas directly to the stack.

Iodine was not liberated upon neutralization.
d. **Run Nos. 001-RP, 002-RP, 003-RP, 004-RP, 005-RP**

Neutralization was conducted separately from acid dissolution. Off-gas activity did not increase significantly during neutralization.

8. **Silica Separation**

   a. **Run No. 001-RH**

   Acid dissolution of the caustic residue was performed satisfactorily.

   The centrifuge periscope light was inoperable making inspection of the bowl impossible following silica removal. However, the washing procedure developed during the final cold runs was followed.

   Radiation level at the cell door was 90 mR/hr during this step.

   b. **Run No. 002-RH**

   Acid dissolution of the caustic residue was performed satisfactorily.

   The centrifuge periscope light had been repaired following the previous run and was used to determine extent of silica removal from the centrifuge. Traces of silica cake remained after extended washing but this was not considered serious.

   c. **Run No. 003-RH**

   The effect of fission product heating was noticed during the acid dissolution operation. For the first time the centrifuge required cooling rather than heating.

   Silica removal was satisfactory as determined by inspection using the centrifuge periscope.

   d. **Run No. 001-RP**

   Acid dissolution of the caustic residue was delayed 17 hours due to adverse weather conditions. During this delay the centrifuge was cooled by adding water to the centrifuge bowl occasionally and by using cooling water in the centrifuge case jacket while operating the centrifuge at 1000 rpm.
e. **Run No. 002-RP**

Acid dissolution of the caustic residue caused a 10°C temperature rise in the centrifuge. Off-gas activity increased slowly after acid addition. After the acid solution was skimmed to the supernate tank, the radiation level just outside the cell door increased to 350 R/hr.

f. **Run Nos. 003-RP, 004-RP, 005-RP**

Iodine activity in RaLa off-gas first appeared when the caustic residue resulting from dissolver slurry centrifugation was dissolved in 6M nitric acid. In Run 005-RP extensive testing of carbon beds in a sample off-gas stream was conducted. Results are reported in Appendix C.

9. **Nitrate Precipitation**

a. **Run No. 001-RH**

The nitrate boildown operation was performed without incident. Uranium supernate did not plug the centrifuge kidney outlet line even though silica gel was present.

b. **Run No. 002-RH**

The nitrate boildown operation was performed without incident. Radiation level at the cell door was 7 R/hr after uranium supernate was skimmed to the supernate tank, L-105. After uranium supernate was jetted to the uranium storage tank, L-107, the cell door radiation level dropped to 750 mr/hr.

c. **Run No. 003-RH**

The usual steam control setting caused carry-over of solution to the condenser during the nitrate boildown operation. It thus was necessary to stop the evaporation and transfer solution from the condenser back to the boildown tank. The transfer lines were not flushed so a small quantity of barium may not have been recovered.

The boildown was begun while venting to the vessel off-gas system. Activity release caused the VOG fans to read 500 mr. To prevent further fan contamination, boiling was stopped and cell vessels were switched to the dissolver off-gas system so that the off-gas scrubber would be used.

After uranium supernate was skimmed to the supernate tank the radiation field at the cell door increased to 100 R/hr.
d. **Run No. 001-RP**

The nitrate boildown operation was conducted without incident. Fuming nitric acid used to precipitate barium following the boildown operation became diluted with water which had been left in an addition pressure pot. However, this fact was not discovered until the run was over. A high barium loss of 34 percent was experienced because of this dilution.

e. **Run No. 002-RP**

During nitrate boildown a carry-over of solution to the condenser was experienced. This caused some delay because the condensate was returned to the boildown tank for re-evaporation which then was conducted without incident.

After uranium supernate was transferred from the supernate tank to the shielded uranium storage tank the radiation level just outside the cell door dropped from 350 to 30 R/hr.

f. **Run No. 003-RP**

A rapid decrease in the concentrate volume occurred near the end of the boildown operation. Simultaneously, an increase appeared in the collected condensate. This phenomenon occurred at the same steam setting as that used throughout the boildown period. However, no significant barium loss was experienced. A faulty steam trap may have caused the described phenomenon which had not occurred in previous runs.

g. **Run No. 004-RP**

The boildown operation required two hours. When air jets (used during the centrifuge silica cleanout period) were turned off, a significant increase in evaporation rate resulted.

h. **Run No. 005-RP**

Boildown was conducted slowly without incident.

10. **Barium Chromate Precipitation**

No pH measurements were possible during these runs. All chromate steps were conducted within the prescribed ten-minute period except the first chromate step in Run No. 003-RH which required twelve minutes. Even so, only 5.3 percent barium loss was experienced. In the first two runs no samples were taken for barium loss determination. In other runs where samples were taken no unusually high barium losses occurred (except for a 10.7 percent loss in Run No. 004-RP) which indicates that satisfactory chromate precipitation
prevailed. It may also be assumed that chromate decomposition under radiation is not excessive.

11. **Metathesis and Final Nitrate Precipitation**

   No known difficulties were encountered in any of these runs. The time at which the final nitrate precipitation is performed is called the last separation time (LST). At this time the growth of lanthanum-140 begins in the product.

12. **Product**

   a. **Run No. CO1-RH**

   Product was dried and brought out of the cell by use of the mechanical manipulator and elevator. Product contained considerable discoloration. Even though no active barium was present, the product contained too much activity for direct sampling. A radiation reading of 35 mr/hr was obtained at the cup plug. With the plug removed, a reading of 5 R/hr was obtained at one foot. Shielding out beta radiation gave 1 R/hr at one foot. The shipping pot itself was not contaminated after being in the cell.

   The product was returned to the cell and dissolved in water at the recycle station. The solution then was jetted to supernate tank, L-105, and from there to PEW sampling tank, L-110, for remote sampling. Barium recovery was 62.7 percent. Spectrochemical analysis indicated high contamination with aluminum and sodium. However, this contamination probably came from tanks and lines through which product solution was transferred prior to being sampled.

   Product solution was transferred to PEW for disposal.

   b. **Run No. CO2-RH**

   Product solution was dried satisfactorily. No radiation heating effects were noted. Power to the drying station coils was the same as used in the runs involving unirradiated material. The white crystalline product contained very little discoloration. Product recovery was only 27.5 percent (833 curies) although this was not known until delivery was made to the customer. Nine washing operations on the product were performed by the customer before contaminants were reduced to an acceptable level. However, this is normal procedure since ORNL product usually required from seven to fourteen washings.
c. Run No. 003-RH

When the shipping pot was initially lowered into the cell it was discovered that no product cup was in the pot. It was thus necessary to remove the pot from the cell for insertion of a cup. During the removal operation a short burst of air-borne activity escaped to the Process Makeup area. The source of this activity is undetermined. No such activity was experienced when the shipping pot was later removed from the cell after completion of the drying operation.

As in the previous run, no radiation heating effects were detected during the product drying operation. Drying required more time than usual due to a cautious approach to minimize the possibility of boilover.

Dried product was slightly off-white in color and gave off a faint glow in the dark. Some discoloration was visible on the cup walls. The customer reported barium-140 activity was 8,081 curies at LST which represents 51.6 percent recovery.

After the shipping pot was removed from the cell and stripped of its protective plastic bag a smear showed 5 mr/hr external contamination. Dry wiping with rags provided adequate decontamination.

The customer reported that seven washings were adequate in reducing product contaminant concentration to an acceptable level.

d. Run No. 001-RP

Product solution volume was reduced too much between product wash additions. This caused considerable spattering which was accompanied by an apparent barium loss of 18.5 percent in the drying condensate.

Dried product was reddish-brown in color. The customer reported 18,000 curies received at LST which represents 46.5 percent recovery. Five washings were adequate in purifying the product at the customers' site.

Upon removal of the shipping pot from cell L, considerable contamination was found on the uncovered lifting ears. These ears were removed for decontamination.

e. Run No. 002-RP

An extra product wash operation was added because of an uncertain product skimmer heel mark. The drying operation
proceeded faster than usual, presumably due to activity heating effects.

Dried product was off-white in color. The customer reported barium-140 activity of 49,000 curies which is 112 percent of the theoretical charge. Four washes were required for product cleanup.

Radiation level at the cell door was 10 R/hr with product cup in the shielded product testing station.

f. Run No. 003-RP

The drying operation was conducted satisfactorily. Dried product contained brownish deposits. Product activity corrected to LST was 18,600 curies as reported by the customer which represents a recovery efficiency of 78.2 percent. Three washes were required for product cleanup.

g. Run No. 004-RP

Product contained 21,350 curies of barium-140 at LST as reported by the customer. This represents 73.3 percent recovery. The product quality was the best ever produced, requiring only two washes on the part of the customer. Strontium contamination amounted to 78 millicuries of which only four percent was strontium-90.

h. Run No. 005-RP

Dried product contained 14,500 curies of barium-140 representing 45.3 percent recovery. Two washes were required for product cleanup.

13. Run Deficiencies

a. Run No. 001-RH

(1) Centrifuge feed pot plugged four times.

(2) Low product recovery (62.7 percent).

b. Run No. 002-RH

(1) Low product recovery (27.5 percent).

(2) High barium loss in waste.
c. Run No. 003-RH
   (1) Boilover during nitrate boildown operation.
   (2) Low product recovery (51.6 percent).

d. Run No. 001-RP
   (1) High barium loss (34 percent) in uranium supernate caused by fuming nitric acid dilution.
   (2) High barium loss (18.5 percent) during product drying operation caused by poor operational techniques.
   (3) Low product recovery (46.5 percent).

e. Run No. 002-RP
   (1) Boilover during nitrate boildown operation.
   (2) Contamination appeared in closed condensate system.

f. Run No. 003-RP
   (1) Contamination appeared in closed condensate system.
   (2) No known operational errors.

g. Run No. 004-RP
   (1) Centrifuge feed pot plug caused one hour processing delay.

h. Run No. 005-RP
   (1) Centrifuge feed pot plugging occurred when 40° C dissolver solution was jetted.
   (2) Low product recovery (45.3 percent)
APPENDIX C

OFF-GAS HANDLING

A. Introduction

A caustic scrubber was the only equipment originally installed in the RaLa cell for processing radioactive gas. The assumption was made by the architect-engineer that any active gases passing through the scrubber could either be briefly stored for decay or could be vented directly to the stack under favorable weather conditions. However, there was no equipment available for storage of this gas in the volume and composition anticipated and consequently operation was necessarily limited to periods when the weather was favorable for stack disposal.

RaLa off-gas involves a two-fold problem; namely, activity hazard due to contained active iodine and xenon, and explosion hazard due to contained hydrogen. The off-gas activity is too great to permit indiscriminate venting to the atmosphere and the hydrogen concentration is in the explosive region making mechanical compression and storage hazardous.

RaLa gaseous activity is evolved in two stages. The first stage involves the caustic dissolution of an MTR fuel assembly during which approximately 200 SCF of hydrogen and 60,000 curies of xenon are evolved. The second stage of active gas evolution begins when particulates and insolubles separated from dissolver solution are dissolved in dilute nitric acid. Before any active material was processed it was assumed that radioactive iodine would be evolved in this stage within a period of one hour. Total iodine present in an irradiated MTR fuel assembly after two days cooling approaches 76,000 curies with iodine-131 accounting for 28,000 curies of the total. Approximately 80 percent of this iodine was expected to reach the off-gas scrubber which was estimated to be 95 percent efficient in removing iodine. Thus about 3,000 curies of total iodine activity were expected to pass through the scrubber within a one-hour period. On this basis a storage facility was recommended in March, 1956, in order to contain both xenon and iodine activity. Subsequent AEC approval resulted in a 10,000-cubic foot gas holder installation being completed in July, 1957. This was too late to be of use during the RaLa startup program which was considered complete in June, 1957.

During the startup program it became obvious that the gas holder then being installed would not represent the final solution to the RaLa off-gas problem. This was due to iodine being released over a period of days rather than minutes or hours and also due to radiiodine escaping from centrifuges to cell off-gas which does not pass through the scrubber. The gas holder will contain process off-gas from 10 hours of operation. Thus it will still be useful for holding radioactive xenon until favorable weather conditions allow venting to the atmosphere. The tank is large enough so that hydrogen can be diluted below its lower explosive limit.
It is assumed that iodine-131 release as a result of each production run must not exceed 10 curies for the operation to be considered weather independent. A carbon bed installation is being recommended to the AEC for the required additional iodine removal capacity. Therefore, off-gas handling for the RaLa process is necessarily incomplete until such time as additional equipment is installed and demonstrated to be adequate for iodine removal.

The off-gas scrubber, L-102, efficiency has been determined to be 95-98 percent based on scrubber solution analyses and stack monitor results. Iodine reaching the scrubber from process vessels has varied from 0.8 to 48.8 percent of that theoretically present in an irradiated fuel element, with the average being 15 percent. Thus, on the average, only 0.75 percent of the initial iodine activity actually passes through the scrubber. This represents the order of 150 to 200 curies of iodine-131 per run which is considerably less than the 1100 curies of iodine-131 (or 3000 curies of total iodine) originally expected. If the iodine release could be effected within a few hours' time no further modifications or additions would be necessary except for sealing the centrifuges.

B. Off-Gas System

Three different systems handle off-gas from the RaLa process. A cell off-gas system provides ventilation for the process cell; a vessel off-gas system provides common vacuum venting for all process vessels except the dissolver, L-101, and off-gas scrubber, L-102; and a dissolver off-gas system provides vacuum venting for the dissolver, L-101 and off-gas scrubber, L-102. All three of these systems exhaust to a common stack. Remote valving permits venting all RaLa vessels to the dissolver off-gas system. In this manner process off-gas is scrubbed in the off-gas scrubber, L-102. However, because the centrifuges are open to the cell, it is also possible for active gases to pass from process equipment to the cell off-gas system. The centrifuges are to be sealed as soon as possible in order to confine gaseous activity to process equipment. All of these off-gas systems are part of the original ICPP and were not installed specifically for the RaLa process.

As mentioned previously, a separate RaLa dissolver off-gas system was being installed during the startup program. Operation of this new system will be reported separately after successful demonstration. The new system includes a blower and two jets for providing necessary vacuum venting, a 10,000-cubic foot gas holder for gas storage, and necessary valving and controls. It will also include carbon beds for iodine removal.

C. Stack Monitor Results

Just prior to RaLa Run No. 001-RP a stack monitor was installed to determine the iodine-131 discharge rate to the atmosphere. A continuous, metered sample of stack gas is bubbled through a stagnant NaOH-Na2S2O3 scrub solution which is
replaced daily and analyzed for iodine-131 and other beta emitters. Sample flow is induced by an air pump. Assuming a monitor scrubber efficiency of 50 percent and knowing gas flow rates in the stack and in the sample circuit, it is possible to calculate the theoretical iodine-131 discharge rate to the atmosphere. Iodine-131 discharge associated with each RaLa production run so calculated is listed in Table 9.

### Table 9

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Iodine-131, Curies</th>
<th>Discharge Period, Days</th>
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<tbody>
<tr>
<td>001-RP</td>
<td>230</td>
<td>19</td>
</tr>
<tr>
<td>002-RP</td>
<td>351</td>
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<tr>
<td>005-RP</td>
<td>159</td>
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</table>

Evolution of iodine-131 did not occur evenly over the discharge periods indicated. A significant fraction is evolved during the run proper and also during sampling and decontamination following each run. Any disturbance (i.e., sparging, sampling, jetting) of iodine-containing solutions tends to liberate iodine-131. Reduction of iodine-131 evolution subsequent to Run No. 002-RP was effected by lowering jet air pressure and by minimizing solution disturbance. Obviously, this limited decontamination efforts between runs. Extensive decontamination following Run 005-RP caused the comparatively high discharge rate.

Xenon activity was detected in all runs in the dissolver off-gas during caustic dissolution of the fuel assembly. Unlike iodine, xenon is evolved quickly over a period of minutes. It will thus be possible to contain essentially all of the xenon activity in the newly constructed 10,000-cubic foot gas holder. Without the gas holder it would not be possible to conduct weather independent runs because of xenon activity even if there were no active iodine present.

Results from the stack monitor indicated that more iodine-131 is evolved over a period of days following each run than is evolved during the several hours of operation when the run is actually in progress. This led to the obvious conclusion that additional iodine removal capacity is necessary in order to make RaLa operation safe and weather independent. A literature survey indicated that no likely reagent was available which would adequately complex or tie up iodine in solution to prevent its evolution as a gas. Consequently, an experimental program was initiated in conjunction with Run 003-RP to determine an applicable method for iodine removal from process off-gas.
In addition to stack gas monitoring, sample streams from the cell off-gas and dissolver off-gas systems were monitored separately over relatively short periods of time as a check on the accuracy of the stack monitor and to determine the relative amount of activity in each stream. Results from these sample streams corroborated stack monitor results. Also it was found that each stream had contributed approximately one-half of the iodine activity to the stack gas. The iodine in the cell off-gas system can be attributed to the fact that the centrifuges are open to the cell, and that the off-gas from this system is not scrubbed.

D. Experimental Program

1. Apparatus

The experimental system provided for drawing continuously four parallel sample streams from the off-gas scrubber effluent. Flow in these parallel lines was controlled between 1 and 5 SCFM compared to 15 SCFM in the main off-gas line. Sample flow was induced by a nitrogen jet downstream from experimental apparatus. Gas drawn through the system was discharged by the jet back to the off-gas line. Gas scrubbing bottles of 500 ml capacity with gas entry through fritted glass discs were used as contactors in scrubber solution evaluation studies. Various lengths of standard 1-inch pipe were used as contactor vessels in solid bed experiments. Piping to the experimental apparatus was 1/8-inch tubing. Gas flow was regulated by rotameters.

2. Comparative Studies

Initial experiments conducted during Run 003-RP used 0.1M Na₂S₂O₃ in 1.0M NaOH to determine the effectiveness of successive scrubbers using that media. Further experiments using scrubber off-gas in the same RaLa run were conducted to compare the thiosulfate-caustic medium with other solutions and solid beds. Data obtained in these experiments are found in Table 10.

It was determined from the initial tests using successive caustic-thiosulfate scrubbers that a high rate of efficiency is not continuous in this medium from stage to stage. By comparison of the data obtained from parallel scrubber experiments the following relative decreasing efficiency of contacting media was indicated:

a. *Dry silver nitrate film on ceramic packing (heated)
b. *Carbon
c. Carbon tetrachloride
d. 0.1M sodium thiosulfate - 1.0M sodium hydroxide
e. 0.1M sodium thiosulfate
f. Sodium hypochlorite, 2 percent
g. 1.0M sodium hydroxide
h. *Tygon (polyvinyl chloride) packing
i. *Copper packing

*Solid packing
<table>
<thead>
<tr>
<th>Test No.</th>
<th>Test Time, Hours</th>
<th>Adsorber Description</th>
<th>Iodine Adsorbed, Millicuries per hour, Unit Position In Series</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>1</td>
<td>9.67</td>
<td>0.1M Na₂S₂O₃ - 1.0M NaOH</td>
<td>0.366</td>
</tr>
<tr>
<td>2</td>
<td>14.67</td>
<td>0.1M Na₂S₂O₃ - 1.0M NaOH</td>
<td>0.577</td>
</tr>
<tr>
<td>3</td>
<td>1.0</td>
<td>0.1M Na₂S₂O₃ - 1.0M NaOH</td>
<td>0.0873</td>
</tr>
<tr>
<td>4 A</td>
<td>4.83</td>
<td>0.1M Na₂S₂O₃ - 1.0M NaOH</td>
<td>0.0667</td>
</tr>
<tr>
<td>B</td>
<td>4.83</td>
<td>1.0M NaOH</td>
<td>0.0125</td>
</tr>
<tr>
<td>C</td>
<td>4.83</td>
<td>2% NaCl</td>
<td>0.0323</td>
</tr>
<tr>
<td>5 A</td>
<td>5.0</td>
<td>0.1M Na₂S₂O₃ - 1.0M NaOH</td>
<td>0.0874</td>
</tr>
<tr>
<td>B</td>
<td>5.0</td>
<td>0.1M Na₂S₂O₃</td>
<td>0.0419</td>
</tr>
<tr>
<td>C</td>
<td>5.0</td>
<td>CCl₄</td>
<td>0.147</td>
</tr>
<tr>
<td>6 A</td>
<td>3.75</td>
<td>0.1M Na₂S₂O₃ - 1.0M NaOH</td>
<td>0.411</td>
</tr>
<tr>
<td>B</td>
<td>3.75</td>
<td>1st Unit-12&quot; Carbon Bed Others-1.0M Na₂S₂O₃-1.0M NaOH</td>
<td>0.00166</td>
</tr>
<tr>
<td>C</td>
<td>3.75</td>
<td>1st Unit-12&quot; AgNO₃ Reactor Others-0.1M Na₂S₂O₃-1.0M NaOH</td>
<td>0.00142</td>
</tr>
<tr>
<td>7 A</td>
<td>4.25</td>
<td>0.1M Na₂S₂O₃ - 1.0M NaOH</td>
<td>0.819</td>
</tr>
<tr>
<td>B</td>
<td>4.25</td>
<td>1st Unit-Carrier Iodine added Others-0.1M Na₂S₂O₃-1.0M NaOH</td>
<td>0.690</td>
</tr>
<tr>
<td>C</td>
<td>4.25</td>
<td>1st Unit-18&quot; Bed of Copper Turnings Others-0.1M Na₂S₂O₃-1.0M NaOH</td>
<td>0.655</td>
</tr>
<tr>
<td>D</td>
<td>4.25</td>
<td>1st Unit-12&quot; Bed of Tygon Raschig Rings Others-0.1M Na₂S₂O₃-1.0M NaOH</td>
<td>0.280</td>
</tr>
<tr>
<td>8</td>
<td></td>
<td>Blank for lab analysis background check</td>
<td>0.000111</td>
</tr>
</tbody>
</table>

Note: Alphabetical series denote parallel tests. Flow in all lines was 5.0 SCFH.
These comparisons were all made relative to the Na$_2$S$_2$O$_3$-NaOH scrubber. Reference scrubbers containing thiosulfate-caustic solution were installed downstream from solid beds to determine solid bed effluent activity.

The conclusions drawn from experimental work were: (1) to use Na$_2$S$_2$O$_3$-NaOH scrub solution in the RaLa off-gas scrubber, and (2) to install a cleanup carbon bed downstream from the scrubber. The use of carbon tetrachloride was eliminated on the basis of its volatility, toxicity, and disposal problems. Since the silver nitrate reactor requires heating, it was eliminated from consideration because of the attendant hydrogen explosion hazard involved.

3. Carbon Studies

Final experiments were conducted using essentially the same experimental apparatus during Runs 004-RP and 005-RP to determine design data for the proposed carbon bed. One-inch diameter carbon beds of various heights were used to determine adsorptive capacity and efficiency. A reference carbon tetrachloride scrubber in parallel with the carbon beds provided iodine activity data for the inlet gas to the beds. Carbon tetrachloride scrubbers were also located on the carbon bed effluent lines to determine bed retention efficiency. In addition, 3/8-inch and 1/2-inch diameter beds were tested. The 1/2-inch diameter bed was constructed of polyethylene so that it could be dissected into 1/2-inch length sections for relative activity measurements. Two types of carbon, coconut derived and nutshell derived, were used in the experiments. Results of the experiments, which are listed in Table 11, were obtained during a 24-hour period of Run 005-RP. The flow rate through each line was 5 SCFH.

<table>
<thead>
<tr>
<th>Experimental Line</th>
<th>$^{131}$I in Scrubber Solution, mc</th>
<th>$^{131}$I Retained on Bed, mc</th>
<th>Bed Efficiency, Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Reference CCl$_4$</td>
<td>40.0</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>2. 1-Inch Deep Bed</td>
<td>3.1</td>
<td>36.9</td>
<td>92.3</td>
</tr>
<tr>
<td>3. 2-Inch Deep Bed</td>
<td>2.05</td>
<td>37.95</td>
<td>94.9</td>
</tr>
<tr>
<td>4. 4-Inch Deep Bed</td>
<td>1.50</td>
<td>38.50</td>
<td>96.2</td>
</tr>
<tr>
<td>5. 11-Inch Deep Bed</td>
<td>0.97</td>
<td>39.03</td>
<td>97.6</td>
</tr>
</tbody>
</table>

The first inch of the 1-inch diameter beds adsorbed an average of 74 curies of iodine-131 per cubic foot of carbon with evidence that saturation had not been reached. Upon dissection, the leading edges of individual beds were found to contain from three to fifteen times the average specific activity.
of iodine on the 1-inch deep bed. The amount of activity which passed through the beds decreased from 7 percent to 2 percent of that entering as the contact time, based on the velocity of the gas through empty pipe, increased from 0.3 to 3.3 seconds. There was no apparent difference in the adsorptive capacity of the two types of carbon used. These tests confirmed the earlier findings that charcoal is the most suitable iodine adsorbent in this particular application. Regeneration studies were not conducted but it is known that steam regeneration of charcoal is adequate for removing nitrogen oxide and water. Even if iodine is not removed by steam regeneration, the natural decay of iodine to xenon will provide time regeneration. Acidic gases are not expected to reach the carbon beds because of prior removal in the caustic scrubber.
APPENDIX D

DECONTAMINATION EXPERIENCE

A. General

Four hundred man-hours were expended in reducing the general background radiation in L cell from an estimated 1000 R/hr to 0.3-1 R/hr following Run No. 003-RH. Sixteen complete equipment flushes with various decontaminants and three partial flushes were required. The rate of decontamination was much slower than anticipated on the basis of other plant equipment. This was probably in part due to the smallness and batch-type nature of the equipment plus the delays resulting from the inadequate disposal facilities for the flushes due to their high activity.

B. Decontamination Procedure

In general, each batch of decontaminating solution was routed from vessel to vessel in the same sequence as process solutions during a production run. At the same time vessels that were "dead ends" in the process sequence, such as storage vessels and condensers, were treated separately with the decontaminating solution. The decontaminant was kept in contact with each vessel from 4 to 6 hours for each flush. Where possible, the solutions were maintained at a temperature of 80 to 90°C except when using NaF. Spargers and recirculating jets were used where available.

C. Manpower Requirements

Decontamination of L cell was carried out after Run No. 003-RH in order to install a by-pass around the off-gas rotometer. After the previous two runs, during which 1138-day and 27-day cooled MTR elements were processed, the equipment was flushed once with water and once with 10 percent HNO₃. The element used for the third run was an 11-day cooled MTR element. Decontamination started on December 10, 1956, and was finished on January 18, 1957. During this interval all the work was performed on a one shift, five-day week basis, except for one five-day period when two shifts were employed. Decontamination required approximately 400 man-hours including the time for preparing the solutions and feeding them into the vessels. Most of the remaining time was spent waiting for analyses of the decontaminating flushes and waiting for use of disposal facilities for the flushes. Due to the high activity of the early flushes it was sometimes necessary to wait 3 or 4 days before disposal could be accomplished through equipment that had to serve other plant facilities in addition to L cell.
D. Decontamination Effectiveness

1. Decontaminating solutions

Following is a list of the decontaminants employed and the number of times each was used:

<table>
<thead>
<tr>
<th>Decontaminant</th>
<th>No. of Times Used</th>
</tr>
</thead>
<tbody>
<tr>
<td>10% HNO₃</td>
<td>5</td>
</tr>
<tr>
<td>10% NaOH–2-1/2% C₆H₄O₆</td>
<td>5</td>
</tr>
<tr>
<td>3% NaF – 20% HNO₃</td>
<td>2</td>
</tr>
<tr>
<td>20% HNO₃</td>
<td>2</td>
</tr>
<tr>
<td>30% HNO₃</td>
<td>2</td>
</tr>
<tr>
<td>0.25M H₃PO₄</td>
<td>1 (Partial flush)</td>
</tr>
<tr>
<td>Steam</td>
<td>1 (Partial flush)</td>
</tr>
<tr>
<td>60% HNO₃</td>
<td>1 (Partial flush)</td>
</tr>
</tbody>
</table>

Each of the 10 percent sodium hydroxide–2-1/2 percent tartaric acid flushes was immediately followed by one of the 10 percent nitric acid flushes. Each of the 3 percent sodium fluoride–20 percent nitric acid flushes was followed by a flush of 1.1M aluminum nitrate in order to complex any fluoride ions left on the walls of the vessels and pipes. The 0.25M phosphoric acid flush was restricted to L-156, L-450, centrifuge kidneys and the DOG line up through the rotameter. The 60 percent nitric acid flush included the centrifuge and centrifuge kidneys only.

2. Equipment Decontamination

a. L-101 Dissolver

The dissolver decontaminated faster than any other vessel that held process solution. The first direct reading on the dissolver was taken after the eleventh flush at which time it read 500 mR/hr. The other cell vessels at this time were reading from 3 R/hr to 25 R/hr. The dissolver is well equipped for decontamination as it has a steam jacket for heating, a recirculating jet for washing down the top of the vessel, a sparger and a refluxing condensing coil which aids in maintaining a constant decontaminant concentration.

b. L-102, Off-Gas Scrubber

The scrubber decontaminated faster than the dissolver. However, its initial radiation level was probably not as high as that of the dissolver. Eight flushes were required plus an eight-hour steaming to reduce the radiation level to 500 mR/hr. The packed section was the most difficult part to decontaminate.
Decontaminating solution was recirculated over the packing by means of the nitrogen lift but could not be kept hot because of the rapid rate of dilution when using the steam sparger. The steam treatment seemed to be more effective than the other decontaminants with the exception of 3 percent NaF - 20 percent HNO3.

c. **Off-Gas Rotameter**

The rotameter was quite difficult to decontaminate because there was no provision incorporated in the design for flushing solution through the unit. Therefore, all of the in-cell decontamination had to be done with steam. After 1/4 hours of steaming, the radiation reading at contact on the rotameter dropped from 10 R/hr to 2-1/2 R/hr. Eight more hours of steaming reduced the contact reading to 0.5 - 1 R/hr. At this point, the rotameter was cut out of the cell because it was necessary to make alterations on the unit. It was submersed in hot "Turco" solution and periodically scrubbed by hand for four days. At the end of this period the external contact reading on the rotameter was 20 mr/hr while the inside varied from 100 to 300 mr/hr.

d. **L-154, Feed Pot**

This unit has no provision for holding solution and was fairly difficult to decontaminate. The procedure used was to jet solution from the dissolver to the feed pot from where it continuously drained to the centrifuge. Solution drains to the lower section of the pot by gravity through a 1/8-inch orifice which provides very little washing action for the lower section. The upper and lower sections are equipped with decontamination solution inlet lines. However, use of these lines did not seem any more effective than when using the normal process inlet line. After 16 flushes and 8 hours of steaming the L-154 contact reading was 1 R/hr.

e. **L-105, Supernate Tank**

This tank was quite representative of the rate of decontamination of most of the L cell vessels. The tank could not be completely flooded as it overflows into numerous other vessels among which are storage tanks which contained solutions that could not be mixed with the decontaminants. The vessel was filled to about 3/4 of overflow volume and then good washing action without dilution was provided by means of an air sparger in the tank. Even though the tank could not be directly heated it was possible to maintain a temperature of 60°C by
continuously jetting solution up to the boildown tank where it was heated, and continuously draining hot solution from the boildown tank to the centrifuge from which it was skimmed back to L-105. This procedure was more effective than just sparging a room temperature solution in L-105. It has the additional advantage of simultaneously decontaminating the supernate tank, boildown tank and centrifuge. After three flushes L-105 had a contact reading of 500 R/hr. After 14 more flushes the contact reading dropped to 1-1/2 R/hr.

f. **L-156, Boildown Tank**

This tank decontaminated at approximately the same rate as L-105. The procedure of recirculating solution from L-105 to L-156, down to the centrifuge and back to L-105 was the most effective procedure for L-156. After 16 flushes, L-156 read 1-1/2 R/hr at contact.

g. **L-450, Centrifuge**

The decontaminating solutions and procedure used on the centrifuge were only partially effective. After 19 flushes the interior of the centrifuge was still in excess of 25 R/hr and the kidneys were 15 R/hr. Fortunately, the centrifuges were designed with a 4-inch lead shield around each unit and therefore the background radiation near the centrifuges and away from the unshielded top was only 1 - 2 R/hr. Most of this background was contributed by the centrifuge kidneys which are not behind the unit shield. After 18 flushes, the interior of the centrifuge as seen through the periscope still had several small patches of material clinging to the sides which was believed to be silicon and perhaps ruthenium. When it becomes necessary to decontaminate the centrifuge bowl down to the mr level undoubtedly considerable time will be required plus the use of harsh fluoride-type decontaminants.

The procedure for decontaminating the kidneys involved continual skimming of decontaminating solution from the centrifuge bowl to the kidney. This produces a high velocity liquid stream into the kidney and floods the kidney. As can be seen, the procedure is not very effective. It is probable that there are deposits of silica on the kidney surfaces similar to those in the centrifuge bowl. The kidneys are equipped with decontaminating solution inlet sprays to wash down the inner surfaces, but they are designed in such a way that only 20 percent of the solution added goes to the kidney and the rest goes through a parallel line to the centrifuge case. This results in insufficient pressure to cause the kidney spray to operate as intended.
h. **L-110. Sampling Tank**

This vessel, a standard type tank, decontaminated slightly faster than L-105. After 13 flushes the tank read from 1 to 1-1/2 R/hr at contact.

i. **L-111. Strontium Collection Tank**

This tank was quite difficult to decontaminate and after 16 flushes still had a contact reading of 5 R/hr. Part of the difficulty was due to the small size of the vessel (2.0 liters) which made the sparger useless as all of the solution in the vessel was thrown out the overflow nozzle with sparge pressures as low as 5 psig. In addition, this tank holds strontium chromate solution which probably is quite corrosive and results in the deposition of activity within the granular structure of the stainless steel.

j. **L-320, -350, -356. Condensers**

The condensers decontaminated rapidly as expected. Five flushes reduced the contact readings to a maximum of 500 mr/hr.

k. **L-108. Waste Collection Tank**

This tank decontaminated at about the same rate as L-105. After 16 flushes, the contact reading had been reduced from greater than 500 R/hr to 1 R/hr. L-108 is the largest vessel in L cell (1900 l). However, it was satisfactorily decontaminated with 100 to 200 liters per flush by using the internal decontaminating sprays and recirculating jet. The recirculating jet is steam operated and consequently solutions at temperatures in excess of 70°C could not be recirculated. In addition, the decontaminant was diluted by steam from the jet.

l. **L-103, -153, -107, -109, -112, -113. Storage Vessels**

These vessels are located behind and under an 8-inch to 12-inch lead shield and consequently it was not necessary to decontaminate them.

3. **External Decontamination**

There was considerable external contamination in L cell because the centrifuges are open to the cell and the product cup is open to the cell during part of the operation. In the case of one vessel near the top of the cell (L-154), the contact reading was reduced from 2 R/hr to 1 R/hr by washing the outside of the vessel with "Turco" solution and then rinsing with water.
The exit cell air filters read 40 R/hr. After hosing the filters with water for 30 minutes the reading dropped to 10 R/hr. The cell sprays were used with water 4 times and with steam twice for approximately 5 minutes each time. In addition, the cell was hosed down with 900 liters of "Turco" solution. This treatment was sufficient to allow men to work in the cell without becoming unduly contaminated.

There was also considerable contamination in the pit just outside the cell door. This contamination probably resulted from back eddies of air when the cell door was opened after the run. The contaminated area was quickly and easily cleaned by wiping with damp rags.

Table 12 summarizes vessel decontamination with respect to the number, sequence and composition of the flushes each vessel received plus the beginning and ending radiation levels.
### Table 12

**Decontamination Results**

| Vessel | Est. R/hr at Contact Before Decon. | 1  | 2  | 3  | 4  | 5  | 6  | 7  | 8  | 9  | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | R/hr at Contact After Decontam. |
|--------|-----------------------------------|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|---------------------------------|
| L-101  | 2000                              | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | 0.3                           |
| L-102  | 300                               | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  |     |     |     |     |     |     |     |     | 0.5                           |
| L-105  | 2000                              | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  |     |     | 1.5                           |
| L-108  | 1000                              | x  | x  | x  | x  | x  | x  | x  | x  | x  |     | x  | x  | x  | x  |     |     |     |     | 1.0                           |
| L-110  | 1000                              | x  | x  | x  | x  | x  | x  | x  |     |     |     |     |     |     |     |     |     |     |     | 1.0-1.5                       |
| L-111  | 1000                              | x  | x  | x  | x  | x  | x  | x  |     |     |     |     |     |     |     |     |     |     |     |     | 5.0                           |
| L-154  | 1000                              | x  | x  | x  | x  | x  | x  | x  | x  |     | x  | x  | x  | x  | x  |     |     |     |     |     | 1.0                           |
| L-156  | 1000                              | x  | x  | x  | x  | x  | x  | x  | x  |     |     |     |     |     |     |     | x  |     |     |     |     | 1.5                           |
| L-320  | 300                               | x  | x  | x  | x  | x  | x  | x  | x  | x  |     |     |     |     |     |     |     |     |     |     |     | 0.5                           |
| L-350  | 100                               | x  | x  | x  | x  | x  | x  | x  | x  | x  |     |     |     |     |     |     |     |     |     |     |     | 0.3                           |
| L-356  | 300                               | x  | x  | x  | x  | x  | x  | x  | x  | x  |     |     |     |     |     |     |     |     |     |     |     | 0.5                           |
| L-450  | 3000                              | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  | x  |     | 15 > 25                       |

*Flushes: 1, 3, 5, 7, 11 - 10% HNO₃
2, 4, 6, 10, 14 - 2-1/2% C₆H₅NO₆ - 10% NaOH
8, 9 - 3% NaF - 20% HNO₃ followed by 1.1M Al(NO₃)₃
12 - 0.25M H₃PO₄
13, 15 - 20% HNO₃
16 - steam
17, 18 - 30% HNO₃
19 - 60% HNO₃
APPENDIX E
DETAILED RUN SHEETS

I. PRE-RUN PREPARATION

1. Reagent Supply Check

<table>
<thead>
<tr>
<th>Reagent</th>
<th>Vol/Run, L</th>
<th>Ample Supply</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. 90% HNO₃</td>
<td>1.75</td>
<td></td>
</tr>
<tr>
<td>b. 70% HNO₃</td>
<td>2.70</td>
<td></td>
</tr>
<tr>
<td>c. 50% HNO₃</td>
<td>0.3</td>
<td></td>
</tr>
<tr>
<td>d. 6M HNO₃</td>
<td>2.7</td>
<td></td>
</tr>
<tr>
<td>e. 0.2M HNO₃</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>f. NaOH</td>
<td>30 lbs. pellet - 15 lbs. Flake</td>
<td></td>
</tr>
<tr>
<td>g. CH₃COONa, 4M</td>
<td>0.675</td>
<td></td>
</tr>
<tr>
<td>h. Na₂Cr₂O₇ No. 1</td>
<td>0.065</td>
<td></td>
</tr>
<tr>
<td>i. Na₂Cr₂O₇ No. 2</td>
<td>(Contains Sr(NO₃)₂ carrier)</td>
<td>0.050</td>
</tr>
<tr>
<td>j. Ba(NO₃)₂ (Carrier)</td>
<td>0.172</td>
<td></td>
</tr>
<tr>
<td>k. pH 4 H₂O</td>
<td>1.0</td>
<td></td>
</tr>
</tbody>
</table>

List Checked by______________

2. Request Photo Neutron Counter Check  Date__________  By__________

3. Mechanical Check PA Area  By__________

   a. Oil manipulator
   
   b. Transport cup to each station and pick up cup at each station.
   
   c. Empty oil collection cup if necessary.
   
   d. Move heating coil to drying station.
   
   e. Raise heating coil via hydraulic lift.
   
   f. Turn power to heating coil just long enough to observe temperature increase.
g. Check hydraulic lift at recyle station.

h. Flush and operate centrifuge. Check heel settings on both skimmers. Adjust both to bowl wall setting at skimming speed.

i. Check centrifuge oil receiver.

j. Check traps - WF area by passing steam through them.

k. Check cell lighting. Replace burned out bulbs.

4. Make sure the following vessels are empty. Vessels should be on VOG.

   a. PA area pressure pots
   b. L 101
   c. L 106
   d. L 156
   e. L 300
   f. L 350
   g. L 320
   h. L 306
   i. L 356
   j. L 105
   k. L 110
   l. L 108
   m. L 109
   n. PM area pressure pots

5. Turn on H2 analyzer 48 hours before run starts.

6. Tie water and steam lines to pressure pots together out of the way making it impossible to erroneously use them in place of air.
6. Make out sample sheets in advance.

7. Cover shipping cask and lifting lugs with plastic.

8. Cover PM area floor with blotting paper and buff paper as on previous runs.

9. Check antifreeze supply and add required amount to the reservoir in advance. Be sure outlets do not leak.

10. Inspect cup and plug in cell and check cup tool adapter in cup plug. Cup No. ____________

II. SOLUTION MAKE-UP

1. Caustic for Off-Gas Scrubber (L-102).

   Done by ________________  Date ______________

   a. Rinse and empty PM 120-0.

   b. Close the outlet valves on PM 120-0.

   c. Add 113 liters H2O to PM 120-0.

   d. Turn on agitator.

   e. Slowly add 10 lbs. flake caustic and slowly add 6 lbs. of sodium thiosulphate.

   f. Agitate solution until all caustic is dissolved.

   g. Add caustic to scrubber through caustic filter.

   h. Close outlet valve and block valve.

   i. Record level on L-102.

   LL ________________

   DR ________________

   Volume ________________
2. **Caustic for Dissolver L 101**
   a. Rinse and empty PM 120-0.
   b. Close all outlet valves on PM 120-0.
   c. Add ______ liters H₂O to PM 120-0.
   d. Turn on agitator.
   e. Slowly add ______ Kg's (_______ pounds) pellet caustic to PM 120-0 based on Al ratio of assembly.
   f. Agitate until pellets are all dissolved.
      Done by ________________
      Date ________________

3. **Acid for Caustic Neutralization**
   a. Add water to L 109 through the PM area addition funnel to a liquid level reading of 15.
   b. Add 100 liters of 60% HNO₃ to L 109 from PM 122-0.
      Done by ________________
      Date ________________

4. **Dissolver (L 101) Charge Water**
   a. Close all outlet valves on PM 121-0.
   b. Charge ______ liters H₂O to L 101 through PM 121-0.
   c. Check that H₂O is going to L 101 condenser coils and record L 101 liquid level.
      L 101 Liquid Level
      Done by ________________
      Date ________________

III. **PM AREA EQUIPMENT CHECKS**

   1. Check air wrench to make sure it is operable. ______
   2. Check that elevator is operable. ______
   3. Check slide valve to see that it is operable. ______
   4. Check centrifuge oil supply. ______
   5. Place carrier-charger ready for straddle carrier pickup. ______
   6. Check operability of caustic pump. ______
IV. **FUEL ELEMENT DATA**

1. SS Material Requisition No. __________

2. Fuel Element No. __________

3. Reactor Shutdown Time __________ Date __________

4. Burnup Data:

<table>
<thead>
<tr>
<th>Grams</th>
<th>Curies</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ba$^{138}$</td>
<td>Ba$^{140}$</td>
</tr>
<tr>
<td>Ba$^{140}$</td>
<td>I$^{131}$</td>
</tr>
<tr>
<td>Total</td>
<td>I$^{132}$</td>
</tr>
<tr>
<td></td>
<td>I$^{133}$</td>
</tr>
<tr>
<td></td>
<td>Sr</td>
</tr>
</tbody>
</table>

Original U __________

Burnup __________

Accountable U __________

Time __________ Date __________

Run Number __________

Date __________

Initial

V. **PRE-DISSOLUTION CHECK**

1. Close all sleeve block valves in PO Area. To be checked independently by two (2) men.

   Checked by __________

2. Blow down headers - PO Area.

   __________
3. Check that all locks and seals are secured on jet valves.

<table>
<thead>
<tr>
<th>Valves Locked</th>
<th>Valves Sealed</th>
</tr>
</thead>
<tbody>
<tr>
<td>L 516 105-109</td>
<td>L 559 105-110</td>
</tr>
<tr>
<td>L 517 105-107</td>
<td>L 536 111-156</td>
</tr>
<tr>
<td>L 518 105-111</td>
<td>L 527 109-FEW</td>
</tr>
<tr>
<td>L 520 105-106</td>
<td>L 545 107-103</td>
</tr>
<tr>
<td>L 521 105-156</td>
<td>L 537 111-113</td>
</tr>
<tr>
<td>L 525 109-U130</td>
<td>L 544 113-112</td>
</tr>
<tr>
<td>L 526 109-U130</td>
<td>L 530 107-FEW</td>
</tr>
<tr>
<td>L 528 107-N150</td>
<td>L 535 111-106</td>
</tr>
<tr>
<td>L 529 107-N150</td>
<td>L 549 110-105</td>
</tr>
<tr>
<td>L 531 112-FEW</td>
<td>L 534 113-FEW</td>
</tr>
<tr>
<td>L 541 110-FEW</td>
<td></td>
</tr>
<tr>
<td>L 543 112-113</td>
<td></td>
</tr>
<tr>
<td>L 555 108-FEW</td>
<td></td>
</tr>
<tr>
<td>L 556 108-FEW</td>
<td></td>
</tr>
<tr>
<td>L 560 110-FEW</td>
<td></td>
</tr>
</tbody>
</table>

4. Check purge to instrument probes.
   a. Operating Corridor
   b. Access Corridor

5. Nitrogen header pressure ____________ psig.

6. Check dissolver liquid level. It should read approximately 33 inches.

7. Check each recorder for operability, inking, etc.

8. Set time and mark time, date, and run number for each recorder.

9. Set the off-gas valves so that only L 101 off-gas goes through L 102.
10. Check off-gas flow. It should be zero. If not, check for leaks. Possible leakage points are:
   a. N₂ sparge
   b. N₂ purge
   c. Addition lines to L 101.

11. Record flow meter reading _______ cfm.

12. Turn on cooling H₂O to L 101 jacket.

13. Blow down all headers in FM area. Air, steam and water.

VI. CAUSTIC DISSOLUTION

1. Check that cover plate is removed from charger chute.

2. Before the carrier is set in place, visually check that slide drawer opens.
   NOTE: If the loaded charger was procured previously, and is found in the charging position, raise it to visually inspect slide valve operation.

3. Leave the slide drawer open and in raised position.

4. Set charger over slug chute.

5. Drain cooling H₂O from charger to FEW.

6. Open the carrier drawer lock.

7. Attach wrench to charger drawer and drop element to dissolver and close slide valve. Time _________

8. Check off-gas flow to make sure slide valve closed. _______ CF

9. Turn on N₂ purge to L 101 at 4 cfm.

10. Start N₂ analyzer jet and adjust instrument.

11. Check that L 101 jacket and condenser water is on.

12. Turn on N₂ sparge to L 101.

13. Turn on N₂ lift to L 102 to 1.5 cfm.

14. Turn on NaOH metering pump and open sleeve block valve to L 101. Time _________
15. Transfer all the NaOH from FM 120-0 to L 101.

16. After all the NaOH from FM 120-0 is in L 101, close hand sleeve valve in caustic addition line and turn off pump. Time __________

17. Turn N2 purge down to 2 cfm and leave sparge on.

18. Make up caustic wash for dissolver.
   a. Close outlet valve from FM 120-0.
   b. Add 7.0 liters H2O to FM 120-0.
   c. Turn on agitator.
   d. Add 3.0 lbs. pellet caustic slowly.
   e. Agitate until pellets are dissolved. By __________

19. Add 3 liters H2O to L 106 or L 156 and check for solenoid leak.

20. Record off-gas rate. Initial Reading __________

21. Turn off the cooling H2O to L 101 jacket and bring L 101 temperature slowly up to 60° C. Time __________

22. Make sure that a negative pressure is always maintained in L 101. Excessive hydrogen evolution rate can cause pressure buildup. The reaction is quite temperature sensitive. Control reaction by varying H2O or steam to dissolver jacket. One man full time.

23. As the off-gas rate drops, heat the dissolver to 95° C for digestion. Leave the N2 sparge and purge on. Decrease temperature if boiling is rapid enough to cause build-up in L 102.

   Time 95° C reached __________

24. One-half hour before digestion is complete, turn on steam to case jacket (RSV-21-PM for L 400, RSV-29-PM for L 450). Digestion is complete one hour after the H2 analyzer recorder levels out at a constant reading of less than 10 on the low scale.

   Time digestion complete __________
25. When digestion is complete, turn off purge. Leave sparge on and keep sample jet on.

VII. PREPARATION FOR CENTRIFUGATION

1. Open RSV-8-PM and preheat feed line from L 104 to L 400 to 80 - 105°C or RSV-9-PM and preheat feed line from L 154 to L 450. (Mixing tee No. 2)

2. Turn on the power to a.c. motors. A time limit of one minute is required before centrifuge can be started. Centrifuge operates on d.c. power.

3. Turn on oil and regulate bubblers to ca. 1 drop per minute.

4. Turn on centrifuge microphone.

5. Open RVC-6-L vent to L 400 feed line or RVC-7-L vent to L 450 feed line.

6. Open discharge valve and turn on air blower to centrifuge motor.

7. Turn on cooling water to the following vessels: L 300 or L 350, and L 108.

8. Cool L 101 to 50°C for jetting.

9. Leave steam on case jacket and maintain bowl temperature of 80°C.

10. Start centrifuge, increase speed to 2600 rpm.

11. Skim any solution collected in the centrifuge to L 105.

12. Put vessels on DOG.

13. Reading at cell door __________ R/hr.

VIII. CENTRIFUGATION OF DISSOLVER SOLUTION L-400 OR L 450

1. Turn on jet L 501 or 502 from L 101 and jet entire solution to L 104 or L 154. Try to use a minimum amount of air to the jet. Time jet on __________
2. Manipulate RSV-8-PM or RSV-9-PM for L 400 or 450, respectively, so that the centrifugation proceeds as near 100° C as practicable. Watch for heating due to radiation.

3. When L 101 is empty turn off jet L 501 or L 502.

   Time jet off _______

4. Turn off steam to L 400 or L 450 case jacket and fill case jacket with water.

5. Reading at cell door _______ R/hr.

6. Turn on caustic metering pump, open sleeve block valve to L 101 and transfer caustic wash to L 101.

7. Skim dissolver solution to L 105 through regular skimmer (300 ml heel).

8. Close sleeve block valve.

9. Turn off H₂ analyzer.

10. Add 15 liters H₂O to PM 120-0.

11. Jet dissolver solution in L 105 to L 109 (jet L 516). Key must be checked out by supervisor.

12. Jet caustic wash from L 101 to L 104 or L 154. (Jets L 501 or 502, respectively.)

13. Open sleeve block valve and RSV-2-PM and drain the 15 liters of H₂O from PM 120-0 to L 101.

14. Close sleeve block valve and RSV-2-PM. (From PM 120-0 to L 101.)

15. Turn off mixing tee No. 2 and close RSV-8-PM or RSV-9-PM. Whichever was in use.

16. Skim caustic wash to L 105 (300 ml heel).


18. Sparge and sample L 108 WAu 1 & 2 for Ba and I₂.

19. Jet approximately 3 liters of H₂O from L 101 to L 104 or L 154. (Jets L 501 or L 502.)
20. Skim wash to L 105, (leave 300 ml heel), and jet to L 109.


22. Check that L 106 or L 156 LL remains constant.

23. Open solenoid valve on L 106 or L 156 and drop contents to centrifuge bowl. Close solenoid valve.

24. Skim wash to L 105, (leave 300 ml heel), and jet to L 109.

25. Push stop button on L 101 condenser coil cooling water.


27. Start jetting L 108 to L 109. (Jets L 552 or L 553.)

    Time jet on

28. Turn on L 109 sparger. ≃ 8 lbs.

29. Turn off cooling H2O to L 108.

IX. DISSOLVER CAKE WASH

1. Wash cake through cake cutter PA 800 or PA 850 three times with 2 liters of H2O each wash. Add each wash when centrifuge stop button is pushed.

   Pressure Pot              Bowl
   a. 1st water wash         ______    ______
   2nd water wash            ______    ______
   3rd water wash            ______    ______

b. After each wash addition, the board operator will do the following:

   Washes
   1. Agitate solution.   1    2    3
   2. Centrifuge one minute.
   3. Skim to L 105 (300 ml heel).

   Time Complete
   Board Operator
2. Flush L 105 three times with two liters of water each through PA 805 with L 105 sparger on, and jet to L 109.

<table>
<thead>
<tr>
<th>PA 805</th>
<th>L 105</th>
<th>L 109</th>
</tr>
</thead>
<tbody>
<tr>
<td>_____</td>
<td>_____</td>
<td>_____</td>
</tr>
<tr>
<td>2nd water wash</td>
<td>_____</td>
<td>_____</td>
</tr>
<tr>
<td>3rd water wash</td>
<td>_____</td>
<td>_____</td>
</tr>
</tbody>
</table>

3. Turn off L 105 sparge, close sleeve block valve.

4. When L 108 is empty shut off sparger sleeve valve, sparger and jet valves. Time jet off _____

   Caution: If both jets were used, let one cool before shutting off the other.

5. Sparge and sample L 109 WAn 1 & 2 for Ba and I₂.

6. Reading at cell door: _____ R/hr.

X. PREPARATION FOR SILICA SEPARATION AND ACID DISSOLUTION

1. Turn on cooling water to solenoid head on L 106 or L 156.

2. Turn on cooling water to L 306 or L 356 condenser.

3. Turn on cooling water to line condensers from L 105 to L 106 or L 105 to L 156. (Includes line condenser from L 111 to L 106, and L 111 to L 156.)

4. Be sure solenoid valve on L 106 or L 156 is closed.

5. Make the following addition in the PM Area through PM 806 or 856 to L 106 or L 156 and flush with two water rinses. Check off each step as it is completed. Jets 520 or 521.

   Additions:          Pressure Pot | Feed Tank
   a. 172 ml Ba(NO₃) Carrier | _____ | _____
   b. 250 ml water | _____ | _____
   c. 250 ml water | _____ | _____
XI. ACID DISSOLUTION AND WASH

1. Check that vessels are on DOG. 

2. Put centrifuge on low low. 

3. Charge 1500 ml 6M HNO₃ to PA 800 or PA 850 pressure pot and charge to L 400 or L 450. Time __________

4. Regulate bowl temperature between 40-50° C by the following:
   
   NOTE: Whenever steam service is discontinued to the case jacket, fill jacket IMMEDIATELY with H₂O.

   a. Steam or H₂O to case jacket.

   b. If more cooling is required, turn on H₂O to centrifuge vent line condenser (RSV-16-PM or RSV-25-PM).

   c. Use bottom bowl spray (mixing tee No. 1) for cooling, only and as last resort. __________

5. Agitate solution in bowl with intermittent stops and starts up to 1000 rpm. Allow 15 minutes for dissolution. __________

6. Centrifuge at full speed for five minutes. __________

7. Turn on cooling water to L 105 jacket. __________

8. Turn trickle of cold water to line jacket on line leading from centrifuge to L 105 (mixing tee No. 2), valves RSV-(12 or 13)-PM. __________

9. Turn on jet L 520 or 521; L 105 to L 106 or L 156. __________

10. Skim bowl liquid to L 105, leave 300 ml heel. Time __________

11. Turn off jet L 520 or 521 from L 105 to L 106 or 156. __________

12. Wash dissolver cake 4 times with 300 ml portions of 6M HNO₃ per wash according to the following procedure:
(Make check mark for each step.) Washes

   a. Charge the 300 ml to PA 800 or PA 850. No. 1 No. 2 No. 3 No. 4

   b. Pressurize PA 800 or 850, and admit wash to bowl at low low speed. __________
c. Mix five minutes by rotating the bowl intermittently.

<table>
<thead>
<tr>
<th>Washes</th>
<th>No. 1</th>
<th>No. 2</th>
<th>No. 3</th>
<th>No. 4</th>
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d. Centrifuge full speed for 3 minutes.

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e. Skim to L 105, leave 300 ml heel. Reduce speed to low low.

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f. Jet contents of L 105 to L 106 or L 156 - jets 520 or 521.

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</table>

Time completed ______________  Initial __________

13. Add 1.0 liter water flush to L 105 through PA 805. Jet flush from L 105 to L 106 or L 156 (jets 520 or 521).

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14. Turn off jet 520 or 521 and close wall sleeve valve.

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15. Turn off any services used to:

a. Case jacket.

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b. Centrifuge vent condenser.

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c. Bowl spray.

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d. Line jacket on centrifuge to L 105 line.

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17. Record LL, sp. gr., and temperature on L 106 or L 156 before heating.

<table>
<thead>
<tr>
<th>LL</th>
<th></th>
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<table>
<thead>
<tr>
<th>Sp. Gr.</th>
<th></th>
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<table>
<thead>
<tr>
<th>Volume</th>
<th></th>
<th></th>
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<table>
<thead>
<tr>
<th>Temperature</th>
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</table>

XII. **COMBINED SUPERNATE BOILDOWN**

1. Turn on air sparge to L 106 or L 156.

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</table>

2. Turn on steam to L 106 or L 156 jacket. Use trap. **DO NOT EXCEED VALVE CONTROL PRESSURE OF 5 LBS.** at the start. Do not allow the temperature to rise too rapidly. Watch for heating due to radiation.  Time steam on ____________

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</table>

3. Turn off air sparge after boiling has begun. Time ____________
4. While volume is being reduced in L 106 or 156 to 1.5 liters, complete the silica removal.

XIII. SILICA REMOVAL

1. Flush silica from the centrifuge bowl as follows:

   a. Attach a treated water line to PA 800 or PA 850 and open the valve. (Hot water preferred)

   b. Turn the centrifuge to low low.

   c. Build up pressure in PA 800 or PA 850 and open the plug valve to the centrifuge in use.

   d. Allow the bowl to overflow for 10 minutes. (Do not use skimmer as kidney will be flooded.)

   e. Close plug valve to PA 800 or 850.

   f. Increase centrifuge speed to 500 rpm and gouge the regular skimmer in as rapidly as possible without causing undue bowl sway. Do not hit bowl wall. (Minimum heel)

   g. Reduce centrifuge speed again to low low.

   h. Open the plug valve from PA 800 or PA 850 to the centrifuge for 5 - 10 seconds.

   i. Bring the centrifuge up to 500 rpm and skim the water immediately to L 105. (Minimum heel)

   j. Reduce centrifuge speed to low low.

   k. Repeat steps h through j continually for 30 minutes.

   l. Jet L 105 to L 109 as necessary. (It is permissible to leave the jet on continuously but close the sleeve block valve first before closing the RSV.)

2. Inspect bowl.

3. Repeat steps g through l if necessary until bowl is clean.

4. When bowl is clean do the following:

   a. Blow H2O remaining in PA 800 or 850 to the centrifuge.

   b. Skim to L 105 (Minimum heel).
c. Wash bowl 4 times through PA 800 or 850 using one liter demineralized water per wash. Skim each wash to L 105 (Minimum heel).

<table>
<thead>
<tr>
<th>Wash</th>
<th>Pot</th>
<th>Centrifuge</th>
<th>L 105</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wash 1</td>
<td>1000 ml H2O</td>
<td>_________</td>
<td>_________</td>
</tr>
<tr>
<td>Wash 2</td>
<td>1000 ml H2O</td>
<td>_________</td>
<td>_________</td>
</tr>
<tr>
<td>Wash 3</td>
<td>1000 ml H2O</td>
<td>_________</td>
<td>_________</td>
</tr>
<tr>
<td>Wash 4</td>
<td>1000 ml H2O</td>
<td>_________</td>
<td>_________</td>
</tr>
</tbody>
</table>

**NOTE:** Adjust both skimmers to bowl wall setting at skimming speed.

d. Add 300 ml H2O to L 400 or L 450 via PA 800 or 850.

e. Run centrifuge at 2600 rpm, engage the REGULAR skimmer with the liquid in the bowl without skimming any solution out, and mark the masking tape on the skimmer control rod in a clear manner.

f. Skim the bowl contents to L 105.

g. Stop the centrifuge. Add 100 ml H2O to L 400 or L 450 via, PA 800 or 850.

h. Run centrifuge at 2600 rpm and engage the PRODUCT skimmer with the liquid in the bowl without skimming any solution out, and mark the masking tape on the skimmer control rod in a clear manner.
i. Turn on centrifuge jet L 514 or 515 to L 105.

j. Jog the centrifuge intermittently and remove all the water with jet.

k. Dry the bowl by rotating at high speed while admitting air through PA 800 or 850 for 5 minutes.

l. Turn off air, close pressure pot block valve, and stop the centrifuge.

5. Jet L 105 to L 109; jet 516. Turn off wall sleeve valve. __________


7. Check to be sure sleeve valve on L 516 is closed. __________

8. Shut off L 109 sparger. __________

XIV. COMBINED SUPERNATE BOILDOWN, Continued

1. Reduce volume in L 106 or 156 to 1.5 liters and cool to 30° C. Time __________

2. Record the following L 106 or 156 data after cooling to Ca. 30° C. __________

   LL __________

   Sp. Gr. __________

   Volume __________

   Temperature __________

3. Record LL and volume in L 306 and 356 when boildown is completed. __________

   LL __________

   Sp. Gr. __________
Volume  
Temperature  

XV. URANIUM SEPARATION (FIRST NITRATE PRECIPITATION) AND WASHES

1. Check L 105 to insure vessel is empty. 

2. Turn on cooling water to centrifuge case jacket (RSV 21 or 28) and vent line condenser (RSV 16 or 25). 

3. Adjust centrifuge speed to low low. 

4. Open solenoid valve and drop contents of feed tank to centrifuge. 

    Time  

5. Turn on cooling H 2 O to L 105 jacket (RSV-31 PM). 

6. Prepare to add the following nitric acid washes separately to the feed tank L 106 or 156 from PM area through PM 806 or 856 for L 400 or 450 upon orders from the board operator. 

7. After each addition the board operator will do the following steps and check off each wash. 

<table>
<thead>
<tr>
<th></th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1000</td>
<td>300</td>
<td>300</td>
<td>300</td>
</tr>
<tr>
<td></td>
<td>90%</td>
<td>70%</td>
<td>70%</td>
<td>50%</td>
</tr>
</tbody>
</table>

a. Have the addition made to pressure pot PM 806 or 856. 

b. Pressurize pot and admit contents to L 106 or L 156. 

c. Open solenoid valve and drop contents of L 106 and L 156 while jogging centrifuge. 

d. Agitate solution in bowl. 

e. Centrifuge for 3 minutes. (Watch for shaft wobble. Oil if necessary).
f. Skim with regular skimmer and leave minimum heel.

g. Stop centrifuge, call PM area operator for next addition and repeat (a) through (e) for remaining washes.

Time Complete ____________________

Initial

XVI. Ba(NO₃)₂ DISSOLUTION

1. Add 500 ml H₂O to PM 806 or 856.

2. Run centrifuge on low low.

3. Pressurize PM 806 or 856 and admit contents to L 106 or L 156.

4. Open solenoid and drain L 106 or L 156 to centrifuge.

5. Agitate solution in the bowl. Allow 10 minutes. Do the remaining steps in this section during the dissolution.

6. Turn on cooling H₂O to L 111 jacket.

7. Turn off H₂O to solenoid head L 106 or L 156 and L 306 or 356 and turn off H₂O to line condenser.

8. Turn off H₂O to case jacket.

9. Record the following L 105 data:

   LL
   DR
   Temperature
   Volume
10. Jet L 105 to L 107 (jet L 517), and turn on cooling water to L 107.

11. Rinse L 105 with water as shown below.

<table>
<thead>
<tr>
<th>Wash No. 1</th>
<th>Wash No. 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Add 4000 ml H_2O to PA 805 pressure pot.</td>
<td></td>
</tr>
<tr>
<td>b. Pressurize pot and admit contents to L 105.</td>
<td></td>
</tr>
<tr>
<td>c. Sparge L 105 and jet contents to L 107 (jet 517).</td>
<td></td>
</tr>
<tr>
<td>d. Close sleeve block valves on jet and sparger and turn them off.</td>
<td></td>
</tr>
</tbody>
</table>

Time

12. Put centrifuge on low low.

XVII. FIRST CHROMATE PRECIPITATION AND WASHES

1. Prepare the following solutions:

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>a.</td>
<td>65 ml Na_2Cr_2O_7 Precipitant No. 1.</td>
</tr>
<tr>
<td>b.</td>
<td>550 ml NaAc buffer.</td>
</tr>
</tbody>
</table>

NOTE: THE TOTAL LAPSED TIME BETWEEN THE Na_2Cr_2O_7 ADDITION AND COMPLETION OF THE SUBSEQUENT SKIMMING MUST NOT EXCEED 10 MINUTES. MAKE IT SHORT AS POSSIBLE, YET COMPATIBLE WITH GOOD OPERATING TECHNIQUE.

2. Have the centrifuge running on low low.
3. Add the 65 ml Na₂Cr₂O₇ to PA 801 or 851.  

4. Pressurize PA 801 or 851 and transfer contents to centrifuge.  
   Time ________  

5. Rinse the chromate container with part of the NaAc buffer and add the rinse to PA 801 or 851. ________  

6. Add remainder of the 550 ml of NaAc to PA 801 or 851. ________  

7. Pressurize pot and send contents to centrifuge. ________  

8. Agitate solution in the bowl. ________  

9. Get ready for cake wash, step 15. ________  

10. Centrifuge for 1 minute. ________  

11. Skim to L 105 (Regular Skimmer). (Minimum Heel)  
   Time completed ________  
   Total lapsed time (4-11) ________  

12. Cell door reading ________ R/hr. ________  

13. Jet L 105 to L 111 via jet L 518. ________  

14. Record the following L 111 data:  
   LL ____________  
   DR ____________  
   Volume ____________  
   Temperature ____________  

15. Turn off H₂O to L 105 jacket. ________
16. Wash the chromate precipitate with two 200 ml washes of pH 4.0 H₂O.

<table>
<thead>
<tr>
<th>Wash No. 1</th>
<th>Wash No. 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Add 200 ml of pH 4.0 H₂O to PA 800 or 850.</td>
<td></td>
</tr>
<tr>
<td>b. Pressurize pot and transfer to centrifuge.</td>
<td></td>
</tr>
<tr>
<td>c. Agitate solution in bowl.</td>
<td></td>
</tr>
<tr>
<td>d. Centrifuge for 1 minute.</td>
<td></td>
</tr>
<tr>
<td>e. Skim carefully to L 105 (minimum heel).</td>
<td></td>
</tr>
</tbody>
</table>

Time

17. Sparge and sample L 109 for Ba, U, I₂, (WAwn 5 & 6).

XVIII. BaCrO₄ DISSOLUTION

1. Add 1000 ml 0.2M HNO₃ to PA 800 or 850. Time

2. Operate the centrifuge on low low.

3. Pressurize PA 800 or 850 and admit contents to centrifuge.

4. Agitate solution in the bowl. Three minutes.

5. Put centrifuge on low low.

XIX. SECOND CHROMATE PRECIPITATION AND WASHES

1. Prepare the following solutions:
   a. 50 ml Na₂Cr₂O₇ (Precipitant No. 2)
   b. 125 ml NaAc buffer solution.

NOTE: AGAIN MINIMIZE THE TIME INTERVAL BETWEEN ADDITION OF THE CHROMATE AND COMPLETION OF THE SKIMMING OPERATION.
2. Have the centrifuge operating on low low.

3. Add the 50 ml of Na₂Cr₂O₇ No. 2 to PA 801 or 851.

4. Pressurize PA 801 or 851 and transfer contents to centrifuge. Time __________

5. Rinse the chromate container with part of the NaAc buffer solution and add the rinse to PA 801 or 851.

6. Add remainder of the 125 ml of NaAc to PA 801-851 pressure pot.

7. Pressurize pot and transfer contents to centrifuge.

8. Agitate solution in the bowl.


10. Centrifuge 1 minute.

11. Skim to L 105. Regular skimmer. (Minimum heel)

   Time completed __________

   Lapsed time (4-11) __________

12. Wash the chromate precipitate three times with 200 ml of pH 4 water each wash. The first wash goes through PA 801 or 851 to rinse out chromate. The other two go through the cutter PA 800 or 850. Check each step.

   | PA 801 or 851 | PA 800 or 850 | PA 800 or 850 |
   | Wash No. 1 | Wash No. 2 | Wash No. 3 |

   a. Add 200 ml pH 4 H₂O to pot.

   b. Transfer contents of pot to centrifuge.

   c. Agitate solution in bowl.

   d. Centrifuge 1 minute.

   e. Skim carefully to L 105 with regular skimmer (min. heel).

   f. Put centrifuge on low low.
13. Record L 105 data.
   LL
   DR
   Temperature
   Volume

14. Turn on steam to case jacket if necessary and bring centrifuge temperature up to 35°C for the Metathesis RSV-(21 or 29)-FM. When steam is turned off, fill the jacket immediately with water.


XX. METATHESIS

1. Add 250 ml 90% HNO₃ to PA 800 or 850. Time

2. Pressurize pot and send wash to L 400 or 450.

3. Agitate solution in bowl.

4. Cool centrifuge to 30°C with case jacket and vent condenser. Use bottom spray only if additional cooling is required.

5. Centrifuge for 3 minutes.

6. Skim to L 105 (Minimum heel).


8. Rinse L 105 through PA 805 with 400 ml H₂O.


XXI. DISSOLUTION OF METATHESIS CAKE

1. Add 250 ml H₂O to the centrifuge through PA 800 or 850.
   Time

2. Agitate solution in the bowl.
XXII. **FINAL Ba(NO₃)₂ PRECIPITATION AND CAKE WASH**

1. Prepare the following solutions for addition to L 400 or L 450 via pressure pots indicated.
   a. Precipitant 500 ml 90% HNO₃ - PA 801 or 851.
   b. 1st wash 300 ml 70% HNO₃ - PM 800 or 850.
   c. 2nd wash 300 ml 70% HNO₃ - PM 800 or 850.
   d. 3rd wash 300 ml 70% HNO₃ - PA 801 or 851.
   e. 4th wash 300 ml 70% HNO₃ - PA 801 or 851.
   f. 5th wash 300 ml 70% HNO₃ - PA 801 or 851.
   g. 6th wash 300 ml 70% HNO₃ - PA 801 or 851.
   h. 7th wash 300 ml 70% HNO₃ - PA 801 or 851.

2. Operate centrifuge on low low.

3. Add 500 ml 90% to PA 801 or 851.

4. Pressurize pot and add solution to centrifuge.

   **THIS IS THE L.S.T. RECORD EXACT TIME.**

   Time __________________
   Date __________________

5. Agitate solution in the bowl. Curies at LST________

6. Cool to 30°C using case jacket cooling if necessary.

7. Centrifuge for one minute.

8. Skim to L 105 (Minimum heel).
9. Wash the cake seven times using 300 ml of 70% HNO₃ for each wash.

<table>
<thead>
<tr>
<th>Wash No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure Pot No.</td>
<td>PM800,850</td>
<td>PM800,850</td>
<td>PA801,851</td>
<td>PA801,851</td>
<td>PA801,851</td>
<td>PA801,851</td>
<td>PA801,851</td>
</tr>
</tbody>
</table>

a. Add solution to pot.

b. Send solution to centrifuge

c. Agitate soln. in the bowl

d. Centrifuge for one minute

e. Skim to L 105 (Min. heel)

10. Turn off H₂O to case jacket.


12. Sparge and sample L 109 for Ba and I₂ (WAwn-9 and 10).

13. Check all sparge and jet sleeve valves in PO area to make sure they had been closed. Make note of any left open.

XXIII. **Ba(NO₃)₂** DISSOLUTION TRANSFER AND DRYING

1. Turn on steam to case jacket, if necessary, and heat bowl to 30° C (RSV-21, 29-PM). Be sure to fill case jacket with water after turning off steam.

2. Turn on cooling water to L 320.

3. Turn off cooling water to L 300 or 350.

4. Turn off cooling water to L 109 if temperature remains 30° C.
5. Check position of manipulator and send down elevator with product cup.

6. Remove cup plug and store in spare cup station.

7. Place product cup in drying station.

8. Turn on cable H_2O wash to 1.2 + on rotameter and raise elevator to cell ceiling and turn off cable H_2O when elevator reaches the top.


10. Move drying station under dryer head.

11. Carefully raise drying station to engage cup lip with dryer head.

12. The following demineralized H_2O additions will be made to the centrifuge through PA 800 or 850 as indicated.
   a. 1st addition 500 ml H_2O
   b. 2nd addition 300 ml H_2O
   c. 3rd addition 300 ml H_2O
   d. 4th addition 300 ml H_2O

13. After each addition do the following steps and check them as they are completed.

<table>
<thead>
<tr>
<th>H_2O Additions (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
</tr>
</tbody>
</table>

   a. Add volume of H_2O specified to PA 800-850.
   b. Operate centrifuge on low low.
   c. Pressurize pot and add solution to centrifuge.
   d. Agitate solution in bowl.
   e. Centrifuge 5 minutes.

NOTE: USE PRODUCT SKIMMER
   f. Skim through product skimmer; leave 100 ml heel.
g. Turn off steam to case jacket (last wash).

h. Turn on variac, watch for heating due to radiation, set variac according to rate of temperature increase. Boildown should proceed at setting of Ca 5.

Time __________

i. Proceed with (a) of next step only when TR-120 reaches 1250 C.

14. After last addition has been made to product cup, allow temperature on L 120 to reach 1250 C and remain for Ca 10 minutes at the variac setting in use.


16. Add 1000 ml H2O to L 400 or L 450 via PA 800 or 850.

17. Skim to L 105 and jet to L 109.

18. Fill bowl with H2O.


20. Reduce the variac setting to avoid spattering. Leave the cup against the dryer head for Ca 20 minutes.

21. Observe level increase in L 320 as indication of drying completion.

22. When drying is complete, record the following data for L 320.

   LL __________

   Temperature __________

   Volume __________

   Time __________ Date __________


24. Leave N2 lift on.
25. Turn off sampler jet. 

26. Turn off centrifuge motor, oilers and mikes. 

27. Turn off water to L320. 

28. Turn off centrifuge blower. 

XXIV. PRODUCT TESTING 

1. Scaler Test 
   a. Check scalers on test position. A two minute count should read 7200 ± 50. Record results below. 

<table>
<thead>
<tr>
<th>Count</th>
<th>Count</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scaler No. 1</td>
<td>Scaler No. 2</td>
<td></td>
</tr>
</tbody>
</table>

b. Scaler No. 1 | Scaler No. 2 |  |

c. Scaler No. 1 | Scaler No. 2 |  |

2. Product Counting 
   a. Move product cup to testing station. 

   b. Record two counts on each scaler. It is important to note time. Record results below: 

<table>
<thead>
<tr>
<th>1st Count</th>
<th>2nd Count</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scaler No. 1</td>
<td></td>
</tr>
<tr>
<td>Scaler No. 2</td>
<td></td>
</tr>
<tr>
<td>Average Count</td>
<td></td>
</tr>
<tr>
<td>Time at start of count</td>
<td></td>
</tr>
<tr>
<td>LST</td>
<td></td>
</tr>
<tr>
<td>Lapsed time</td>
<td></td>
</tr>
</tbody>
</table>

Date ___________ Recorded by ______________ 

3. Reading at cell door _______ R/hr. 

4. Visual Check 
   a. Focus viewer on product.
b. Record radiation reading at eye piece. ________

c. Describe product.

________________________

________________________

d. Turn off all cell lights and check for glow. Describe.

________________________

________________________

5. Bring down elevator in preparation of product removal. (Check manipulator position.) ________

XXV. PRODUCT REMOVAL

1. Transfer cup to shipping pot, use binocular while connecting lifting lugs to insure against dropping cup. ________

2. Place plug in cup. ________

3. Turn on trickle water to cable sprays. ________

4. Check that manipulator is clear of the elevator. ________

5. Turn on the elevator and bring the shipping pot to the top of the cell. Turn off the water to the cable sprays when the elevator reaches the top. ________

XXVI. CELL BACKGROUND CHECK

1. Run a count on each scaler to determine amount of Lanthanum left in cell and previously counted as product shipped. 1st Count 2nd Count ________

Scaler No. 1 ________ ________

Scaler No. 2 ________ ________

Average Count ________ ________

Time at start of count ________ ________

LST ________ ________
XXVII. PRODUCT REMOVAL AND SHIPPING

1. Call HP and have a health physicist present during the following steps. Request that he bring HP shipping form.

2. Wear protective clothing consisting of overalls taped at legs and sleeves, rubber boots, rubber gloves, surgical cap, safety goggles, hard hat, and respirator recommended.

3. Remove the elevator hatch cover and take a radiation reading at the opening.

4. Remove the shipping pot and spider from the elevator and lower them into a cardboard box.

5. Screw down the shipping pot cover and remove the spider. Place the spider back in the elevator hatch and replace the hatch cover.

6. Strip the covering from shipping pot into the cardboard box. Have HP take radiation readings and smears. Clean the pot with damp rags if necessary while it is still in the cardboard box.

7. When the shipping pot is clean, place it in the carrier and connect the outlet water line. Bring the carrier cover into position and connect the inlet water line.

8. Check that solution from water tank flows through carrier.

9. Bolt the carrier cover into place and fill it with water. (Use antifreeze solution in place of water in the winter time.)

10. Adjust the flow of coolant to the shipping pot.

11. Bolt the carrier to the pallet and carry it to CPP 603 by means of the straddle carrier.

12. Remove the carrier from the pallet and bolt it to the shipping plate.
13. Place the carrier and shipping plate in the truck.

Time

14. Be sure to write the LST on the IHP-30 shipping form.

XXVIII. POST RUN CHECK

1. Check that all cooling water is off, except what is needed to keep L 107, L 111 and L 109 cool.

2. Sparge and sample L 109 (WAwn 11 & 12) Ba, I2. (Waste supernates and product skim heel.)

   LL
   DR
   Temperature
   Volume

3. Jet L 306 or L 356 condensate to L 110 and sample (Ba) (Wwp 1 & 2).

   LL
   DR
   Volume


5. Jet L 320 to L 110 and sample. (Wwp 3 & 4) (Ba).

   LL
   DR
   Volume


   Record L 109 data:

   LL
   DR
   Temperature
   Volume

8. Add 150 ml of 70% HNO₃ to L 111 through decontamination line and chase with 100 ml H₂O.

9. Sample L 111 (without sparging). Ask for Ba, I₂, U and Sr. (PSa-1 and 2)

   LL __________________
   DR __________________
   Temperature ____________
   Volume ________________

10. Hold first chromate supernate in L 111 until results are back.

11. Jet L 102 to L 103 (jet L 504). Record L 103 data:

   LL __________________
   DR __________________
   Temperature ____________
   Volume ________________

12. Sample L 103. Ask for Ba, I₂, and U (WCu-1 and 2).

13. Dilute L 107 with 70 liters ANN before sampling. Check with Coordinations Supervisor for desired feed specifications in order to calculate amount of ANN to be added. Sparge and sample L 107 PUa 1 and 2, ask for U, Ba and I₂, also, N⁴ if a neutralization was made.

14. Begin decontamination procedure as soon as possible, especially the initial flushing of all "hot" lines and the filling of all vessels with the first solution.

15. Take care not to get additional solution in L 107 or L 111.
XXIX. **POST RUN PROCEDURE**

1. Check that all vessels are on DOG.

2. Check that L 102 scrubber is operating and that the lift is operating on air instead of nitrogen.

3. Allow solutions in L 102, L 109, L 111 and L 107 to set until word is received from supervision to move same.

4. Before any transfer, sparging or sampling operation is conducted, obtain formal approval from the Weather Bureau.

5. Alert CPP Health Physics and have them take a background check on their stack monitors.

6. If during any of the subsequent operations, the background increases by a factor of (2) as reported from CPP-HP, notify site survey.

7. Fill out the following sheet as operations are made:

<table>
<thead>
<tr>
<th></th>
<th>Check One</th>
<th>Date</th>
<th>Time</th>
<th>Initial</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Transferred L 102 to L 103</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>L 153</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>b. Sparged and sampled L 103</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>L 153</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>c. Sent L 111 to</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>L 112</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>L 113</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>d. Sent L 109 to</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>U Cell</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>PEW</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>e. Sent L 107 to</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>N Cell</td>
<td></td>
<td></td>
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<tr>
<td>f. Sent L 112 to</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>L 113</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>