PROBLEMS OF REFINING URANIFEROUS RESIDUES
Technology Feed Materials Progress Report — June 1953

By
H. Fleck
J. E. Summers

July 15, 1953
Vitro Manufacturing Company
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AEC RESEARCH AND DEVELOPMENT REPORT

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A. SUMMARY

GENERAL

1. Scope of Program

As of June 1, 1953, the Research Program consisted of the following projects:

Digestion and Refining of C-Slags and Other Residues
(Project No. 1)

Investigate the applicability and refinement of various procedures for the treatment and digestion of C-Slags and other uraniferous residues for recovery of uranium values, placing emphasis on methods adaptable to the Canonsburg Plant with minimum change of equipment.

Refining of BFC-6
(Project No. 4)

Investigate the recovery of uranium from BFC-6 placing emphasis on procedures which will not chemically alter the copper and tin media.

Refining and Processing of Sodium Carbonate Residues
(Project No. 5)

Investigate methods of recovery of uranium from various residues, resulting from sodium carbonate digestion of
miscellaneous uranium bearing materials, to reduce the U₃O₈ content of the residues below 0.05%.

2. Previous Reports

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PROJECT STATUS

3. Research Project - Digestion and Refining of C-Slags and Other Residues

Experiments were conducted on C-Oxide using "Wilson Dam" P₂O₅ contaminated hydrochloric acid to determine if uranium precipitated from a clarified digest filtrate of this acid would constitute an acceptable end product containing less than 0.5% P₂O₅ and more than 50% U₃O₈.

4. Research Project - Digestion and Refining of BFC-6

Additional experiments were conducted using dilute hydrochloric and nitric acid to determine if these procedures would be effective in satisfactorily extracting uranium from either calcined or uncalcined material. A procedure was sought which would lead to a discard residue containing less than 0.05% U₃O₈.
Experiments were also conducted using concentrated hydrochloric acid, concentrated nitric acid, and concentrated sulfuric acid respectively as digest media. Digestion of both calcined and uncalcined BFC-6 was investigated using concentrated acid to determine if this would lead to acid insoluble residues containing less than 0.05% U₃O₈.

Preliminary experiments were initiated on uncalcined and calcined BFC-6 to determine if sodium hydroxide digestion for the removal of metallic tin followed by sulfuric acid digestion of the insoluble residue and removal of solublized copper by neutralization of the digest slurry with ammonium hydroxide would benefit the raw material to such an extend that uranium content would exceed the minimum specification of 50% U₃O₈ for an acceptable end product.

5. Research Project - Digestion and Refining of Sodium Carbonate Residues

Additional experiments were undertaken in the laboratory parallel with pilot level experiments to determine if adjustments in chemical or handling procedures would lead to more efficient extraction of uranium.
B. DIGESTION AND REFINING OF C-OXIDE AND OTHER RESIDUES

Research Project No: 1
Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

6. Digestion and Refining of C-Oxide using Wilson Dam P₂O₅ Contaminated Hydrochloric Acid

Experiments were initiated on C-Oxide using "Wilson Dam" P₂O₅ contaminated hydrochloric acid as a digest media. Experiments were conducted to determine if this acid would be a satisfactory digest reagent resulting in a clarified hydrochloric acid digest filtrate from which uranium could be precipitated using ammonium hydroxide and standard procedures leading to a satisfactory end product containing less than .5% P₂O₅ and more than 50% U₃O₈.

Five-hundred gram samples of C-Oxide were calcined at 1200° F for one hour and were pulverized to less than 100 mesh. Since the Wilson Dam hydrochloric acid contained less than 10% free HCl, experiments were conducted using H₂O to slurry at a ratio of 1:1. Other experiments were completed without the addition of slurry water. Approximately 4000 ml of the low grade hydrochloric acid was added before the pH of digestion was reduced to approximately 0.25. Sodium chlorate was added to oxidize. Digestion was carried out at 90° C for three hours. Acid insolubles were removed from the digest slurry by filtration and washed
with water until color free. Average uranium content of insoluble residues was less than .05% $\text{U}_3\text{O}_8$. The pregnant clarified filtrate obtained by this procedure was neutralized to pH 6.0 by the addition of dilute (6 molar) ammonium hydroxide. The resultant precipitate was separated by filtration and washed until calcium and magnesium free. Average uranium content of precipitates obtained in this manner was 4.5% $\text{U}_3\text{O}_8$. $\text{P}_2\text{O}_5$ content exceeded 6.5%. Representative data may be found in Table I.

An additional series of experiments of this nature was conducted to determine the optimum end product that could be obtained from C-Oxide using the low grade phosphate contaminated acid. Two-hundred-fifty gram samples of C-Oxide were calcined at 1200°F for one hour, slurried in 250 ml of water, and digested in approximately 2,150 ml of hydrochloric acid at pH 0.2 - 0.3, in the presence of 0.5% (w/v) sodium chlorate to oxidize, for three hours at 90°C. Acid insolubles were removed by filtration and washed with water until color free. Pregnant liquor obtained by this procedure was precipitated by the addition of dilute (6 molar) ammonium hydroxide over a pH range extending from 1.5 - 3.0. The precipitate was separated from the supernatant liquor by filtration and washed with water until calcium and magnesium free. Optimum precipitation was obtained at pH 1.5 with the resultant end product containing on the average 47.0% $\text{U}_3\text{O}_8$, 85% F and 11.5% $\text{P}_2\text{O}_5$. Specifications for a satisfactory end product are exceeded or not obtained in every instance. It is fairly obvious that it will not be possible to obtain a satisfactory end product using the Wilson Dam hydrochloric acid unless additional purification processes are utilized. Average data may be found in Table II.
C. DIGESTION AND REFINING OF BFC-6

Research Project No.: 4
Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

7. Extraction of Uranium from BFC-6 Using Mineral Acid Digestion

Additional experiments using dilute hydrochloric and dilute nitric acid respectively as a digest media for calcined and uncalcined BFC-6 were completed to confirm preliminary data. One-hundred gram samples of BFC-6 were calcined at 1000°F for one hour, slurried in 300 ml of H₂O, and digested in hydrochloric and nitric acid respectively at pH 0.0 for 6 hours at 95 - 100°C in the presence of sodium chlorate as an oxidizing agent. Experiments were also conducted in the same manner using uncalcined material. Acid insolubles were separated from the digest slurry by filtration and washed with water until color free. An investigation of the U content of insoluble residues resulting from this treatment indicated a uranium level averaging .1 - .3% U₃O₈. This is well above the maximum discard specification of .05% U₃O₈ for a discard residue. Representative data may be found in Table III.

Experiments on uncalcined material were conducted using concentrated hydrochloric and sulfuric acids. One-hundred gram samples of uncalcined material were slurried in concentrated sulfuric and hydrochloric acids respectively. Digestion in concentrated hydrochloric was carried out over a three hour period at
90 - 95° C using sodium chlorate to oxidize. Digestion in fuming H₂SO₄ was continued for three hours. An investigation of the uranium level of acid insolubles resulting from procedures using concentrated acids indicated no beneficiation of extraction. Uranium content continued to exceed maximum specifications for a discard residue. Acid insolubles averaged from 0.1 - 0.5% U₃O₈. Data may be found in Table IV.

8. Refining of BFC-6 Using Sodium Hydroxide Digestion

A series of experiments was conducted to determine if the uranium content of BFC-6 could be elevated above the minimum specifications of 50% U₃O₈ for an End-product by digestion of the material in sodium hydroxide to remove metallic tin, digestion of the insoluble digest residue in sulfuric acid followed by removal of solubilized copper by the addition of ammonium hydroxide. One-hundred gram samples of both calcined and uncalcined material were slurried in 300 grams of H₂O and digested in 100 grams of sodium hydroxide for three hours at 95 - 100° C. The supernate was removed by a process of decantation and filtration. The insoluble residue from the sodium hydroxide digestion was repulped in 300 grams of H₂O and digested in sulfuric acid at pH 0.0 in the presence of sodium chlorate for three hours at 95 - 100° C. The digest slurry was neutralized to pH 9.0 using 1:1 ammonium hydroxide. Solubilized copper was removed by filtration and the resultant residue investigated for uranium content. Average uranium content of the end product obtained by this procedure was 30 - 35% U₃O₈. Preliminary data may be found in Table V.
D. REFINING AND PROCESSING OF SODIUM CARBONATE RESIDUES

Research Project No: 5
Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

9. The Extraction of Uranium from Pitchblende Carbonate Residues by a Process of Sodium Carbonate and Hydrochloric Acid Digestion

Experiments were conducted on Pitchblende carbonate residues in parallel with a pilot operation to determine if adjustments in procedure could possibly lead to more efficient extraction of uranium from this material with minimum handling. One hundred gram samples of Pitchblende carbonate residues assaying 1.6% U₃O₈ and 0.69% U₃O₈ respectively were slurried in 200 ml of water and digested in 5% (w/v) sodium carbonate for three hours at 90° C. The digest slurry was filtered and the insoluble residue washed with hot 5% sodium carbonate solution until colorless. This procedure was successful in removing 50 - 55% of the contained uranium. The resultant residues were slurried in 200 ml of water and digested in hydrochloric acid at pH 0.25 for one hour at 90° C. The acid slurry was neutralized with sodium carbonate and digested for three hours at 90° C. After filtration the insoluble residue was washed until color free with hot 5% sodium carbonate solution. Insoluble residues resulting from this treatment averaged 0.1 - 0.3% U₃O₈. The residues were repulped in 200 ml of water and redigested in 5% (w/v) sodium
carbonate for three hours at 90° C. The supernate was removed by filtration and the residue washed until color free using hot 5% sodium carbonate solution. Insoluble sodium carbonate residues obtained by this procedure averaged between .03 - .06% U₃O₈. Representative data may be found in Table VI.

EXHIBITS

TABLE I

PURIFICATION OF C-OXIDE USING P₂O₅ CONTAMINATED HCl

Reagent: "Wilson Dam" Hydrochloric Acid

Assay: 0.5% P₂O₅; 9.0% HCl

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<tbody>
<tr>
<td></td>
<td>1</td>
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</tr>
<tr>
<td>Weight of Sample (gm)</td>
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<td>500</td>
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<tr>
<td>Weight of H₂O (gm.)</td>
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<tr>
<td>Weight of HCl (gm.)</td>
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<td>4,200</td>
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<tr>
<td>Digest Time (hr.)</td>
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<tr>
<td>Digest pH</td>
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<td>0.25</td>
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<tr>
<td>Digest Temperature (°C)</td>
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<td>95</td>
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<td>Acid Insoluble (gm.)</td>
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<td>Acid Insolubles (% U₃O₈)</td>
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<td>.05</td>
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<td>pH of Precipitation</td>
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<td>Residue (gm.)</td>
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<td>Residue (% U₃O₈)</td>
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<td>Residue (% P₂O₅)</td>
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<td>Residue (% F)</td>
<td>1.12</td>
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# TABLE II

## PURIFICATION OF C-OXIDE USING P$_2$O$_5$ CONTAMINATED HCl

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<td>Digest Time (hr.)</td>
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<td>3</td>
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<tr>
<td>Digest pH</td>
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<td>Digest Temperature (°C)</td>
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<tr>
<td>pH of Precipitation</td>
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<td>Residue (% U$_3$O$_8$)</td>
<td>47.04</td>
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<td>Residue (% P$_2$O$_5$)</td>
<td>10.90</td>
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<td>11.68</td>
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<td>Residue (% F)</td>
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### TABLE III

**EXTRACTION OF URANIUM USING DILUTE HYDROCHLORIC AND NITRIC ACIDS**

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<th>Weight of Sample (gm.)</th>
<th>Calcination Temperature (°F)</th>
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<th>Weight of H₂O (gm.)</th>
<th>Reagent</th>
<th>Weight of Acid (gm.)</th>
<th>Digest pH</th>
<th>Digest Temperature (°C)</th>
<th>Digest Time (hr.)</th>
<th>Acid Insolubles (gm.)</th>
<th>Acid Insolubles (% U₃O₈)</th>
<th>Recovery (%)</th>
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<td>-</td>
<td>300</td>
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### TABLE IV

**EXTRACTION OF URANIUM USING CONCENTRATED HCl, HNO₃ AND H₂SO₄**

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<th>EXPERIMENT NUMBER</th>
<th>Weight of Sample (gm.)</th>
<th>Reagent</th>
<th>Weight of Reagent (gm.)</th>
<th>Digest Time (hr.)</th>
<th>Digest Temperature (°C)</th>
<th>Acid Residue (gm.)</th>
<th>Acid Residue (% U₃O₈)</th>
<th>Recovery (%)</th>
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<td>1</td>
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<td>.19</td>
<td>99.65</td>
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<tr>
<td>2</td>
<td>100</td>
<td>HNO₃</td>
<td>280</td>
<td>6.0</td>
<td>95</td>
<td>54</td>
<td>.23</td>
<td>99.36</td>
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<tr>
<td>3</td>
<td>100</td>
<td>H₂SO₄</td>
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<td>3.0</td>
<td>120</td>
<td>37.2</td>
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<td>99.60</td>
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TABLE V

REFINING OF BFC-6 USING NaOH DIGESTION, H$_2$SO$_4$ DIGESTION AND NH$_4$OH PRECIPITATION

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<th>Material:</th>
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<td>Assay:</td>
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<tr>
<td>Weight of Sample (gm.)</td>
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<td>100</td>
</tr>
<tr>
<td>Calcination Temperature (°F)</td>
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</tr>
<tr>
<td>Weight of H$_2$O (gm.)</td>
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<tr>
<td>Weight of NaOH (gm.)</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Digest Time (hr.)</td>
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<td>3</td>
</tr>
<tr>
<td>Digest Temperature (°C)</td>
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<td>95</td>
</tr>
<tr>
<td>Residue (gm.)</td>
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<td>92.3</td>
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<td>Residue (% U$_3$O$_8$)</td>
<td>20.3</td>
<td>21.7</td>
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<tr>
<td>Weight of H$_2$O to Slurry (gm.)</td>
<td>300</td>
<td>300</td>
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<tr>
<td>Weight of H$_2$SO$_4$ (gm.)</td>
<td>68.1</td>
<td>64.4</td>
</tr>
<tr>
<td>Weight of NaClO$_3$ (gm.)</td>
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<td>10.0</td>
</tr>
<tr>
<td>Digest pH</td>
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<td>0.0</td>
</tr>
<tr>
<td>Digest Temperature (°C)</td>
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<td>95</td>
</tr>
<tr>
<td>Digest Time (hr.)</td>
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<td>3.0</td>
</tr>
<tr>
<td>pH of Precipitation</td>
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<td>9.0</td>
</tr>
<tr>
<td>Precipitant</td>
<td>NH$_4$OH</td>
<td>NH$_4$OH</td>
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<tr>
<td>Residue (gm.)</td>
<td>63.0</td>
<td>58.2</td>
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<tr>
<td>Residue (% U$_3$O$_8$)</td>
<td>30.9</td>
<td>34.8</td>
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TABLE VI

EXTRACTION OF URANIUM FROM SODIUM CARBONATE RESIDUES

Material: Pitchblende Carbonate Residues

Assay: \(0.79 - 1.6\% \text{ U}_3\text{O}_8\)

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<tr>
<th>EXPERIMENT NUMBER</th>
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<td>Assay of Sample (% \text{ U}_3\text{O}_8)</td>
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<td>0.79</td>
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<tr>
<td>Weight of Sample (gm.)</td>
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<td>100</td>
</tr>
<tr>
<td>Weight of \text{H}_2\text{O} (gm.)</td>
<td>400</td>
<td>400</td>
</tr>
<tr>
<td>Weight of \text{Na}_2\text{CO}_3 (gm.)</td>
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<td>20</td>
</tr>
<tr>
<td>Digest Time (hr.)</td>
<td>3.0</td>
<td>3.0</td>
</tr>
<tr>
<td>Digest Temperature (°C)</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>Residue (% \text{ U}_3\text{O}_8)</td>
<td>0.59</td>
<td>0.43</td>
</tr>
<tr>
<td>Weight of \text{H}_2\text{O} to Slurry (gm.)</td>
<td>200</td>
<td>200</td>
</tr>
<tr>
<td>Weight of \text{HCl} (gm.)</td>
<td>155</td>
<td>205</td>
</tr>
<tr>
<td>\text{pH} of Digestion</td>
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<td>1.0</td>
</tr>
<tr>
<td>Digest Time (hr.)</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>Digest Temperature (°C)</td>
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<td>90</td>
</tr>
<tr>
<td>Weight of \text{Na}_2\text{CO}_3 Added (gm.)</td>
<td>125</td>
<td>120</td>
</tr>
<tr>
<td>Digest Time (hr.)</td>
<td>3.0</td>
<td>3.0</td>
</tr>
<tr>
<td>Digest Temperature (°C)</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>Residue (% \text{ U}_3\text{O}_8)</td>
<td>0.25</td>
<td>0.11</td>
</tr>
<tr>
<td>Weight of \text{H}_2\text{O} to Slurry (gm.)</td>
<td>200</td>
<td>200</td>
</tr>
<tr>
<td>Weight of \text{Na}_2\text{CO}_3 (gm.)</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Digest Time (hr.)</td>
<td>3.0</td>
<td>3.0</td>
</tr>
<tr>
<td>Digest Temperature (°C)</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>Residue (% \text{ U}_3\text{O}_8)</td>
<td>0.06</td>
<td>0.035</td>
</tr>
</tbody>
</table>
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