DIGESTION AND FILTRATION OF LEACHED ZONE IN PILOT PLANT

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DIGESTION AND FILTRATION OF LEACHED ZONE IN PILOT PLANT

February, 1955

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-3-
## TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>I. Introduction</td>
<td>5</td>
</tr>
<tr>
<td>II. Summary</td>
<td>6</td>
</tr>
<tr>
<td>III. Pilot Plant Digestion and Filtration of -200 Mesh Leached Zone.</td>
<td></td>
</tr>
<tr>
<td>A. Period May 5, 1952 through February 26, 1953. Pressure Digestion</td>
<td>9</td>
</tr>
<tr>
<td>B. Period February 27 through March 13, 1953. Batch Atmospheric Digestion</td>
<td>14</td>
</tr>
<tr>
<td>C. Periods March 16 - May 8, and May 27 - July 16, 1953. Continuous Atmospheric Digestion</td>
<td>16</td>
</tr>
<tr>
<td>D. Period May 12 through May 26, 1953. Fifty-Gallon Batch Atmospheric Digestions</td>
<td>23</td>
</tr>
<tr>
<td>E. Period July 20 through August 7, 1953. Atmospheric Digestion Followed by Pressure Digestion</td>
<td>30</td>
</tr>
</tbody>
</table>
I. INTRODUCTION

Following a mineral evaluation (RMO-2005) and laboratory tests (RMO-2006), a pilot plant was constructed to study the recovery of three products, $\text{U}_3\text{O}_8$, $\text{P}_2\text{O}_5$, and $\text{Al}_2\text{O}_3$, from the -200 mesh fraction of the leached zone in the Florida phosphate fields owned by the International Minerals and Chemical Corporation. The present report summarizes pilot plant operations on the sulfuric acid digestion of -200 mesh leached zone and filtration of the digested slurry for the period May 5, 1952 through August 7, 1953.

Work reported represents the combined efforts of the members of International Minerals & Chemical Corporation's AEC Research Section under Contract No. AT(49-1)-545 with the U. S. Atomic Energy Commission.
II. SUMMARY

The pilot plant operations of 1952 and 1953 on the sulfuric acid digestion of -200 mesh leached zone for recovery of $\text{U}_3\text{O}_8$, $\text{P}_2\text{O}_5$, and $\text{Al}_2\text{O}_3$ have been broken into five chronological periods according to digestion technique. The digested slurry was filtered with three or four stages of countercurrently operated, rotating-disc vacuum filters to produce a first-stage filtrate product of 1.3 specific gravity for further processing.

The first digestions were carried out in a 40-gallon, Carpenter 20 stainless steel autoclave at the optimum conditions found in earlier laboratory work: 160 psig, 360°F, and 70% to 100% acidulation. The digestion time was 1/2 to 2 hours. Severe corrosion of the autoclave and lines and plugging of the lines, particularly when continuous operation was attempted, led to the consideration of atmospheric digestion with longer retention times.

Twenty-four hour batch digestions were carried out in 1000-gallon stainless steel tanks at atmospheric pressure, 180°F, and 80% acidulation. Recoveries were good: $\text{U}_3\text{O}_8$, 95%; $\text{P}_2\text{O}_5$, 96%; and $\text{Al}_2\text{O}_3$, 90%. Filtering rates for the digested slurries ranged from 0.8 to 1.4 gal/(hr)(ft$^2$).

Continuous atmospheric digestion was next carried out in a series of three overflow-connected, 500-gallon
rubber-lined mild steel tanks. Comparisons of results obtained during thirteen sub-periods of operation at various conditions brought out the following points:

1. For all conditions studied, dissolutions of values compared favorably with laboratory results, falling in the following ranges: \( \text{U}_3\text{O}_8, \) 90-99%; \( \text{P}_2\text{O}_5, \) 88-98%; and \( \text{Al}_2\text{O}_3, \) 60-90%.

2. Within these ranges of dissolution, there were slight improvements as acidulation was increased from 66% to 118% and/or acid strength was increased from 21% to 30%.

3. Average filtering rates were 0.9 to 1.6 gal/(hr)(ft^2), representing filtering areas of 7 to 13 square feet per ton of dry leached zone ore processed per day.

4. Process changes which were made to ensure the formation of \( \text{CaSO}_4 \cdot 2\text{H}_2\text{O} \) (gypsum) rather than \( \text{CaSO}_4 \cdot 1/2\text{H}_2\text{O} \) crystals in the digested slurry had little effect on filtering rates. Apparently the effect of the type of calcium sulfate crystal formed was overshadowed by the presence of insolubles in the 10 micron range which blinded filter cloths.

A series of 50-gallon batch atmospheric digestions were next carried out to study the effects of time (2 to 22 hours), temperature (no heat addition vs. 140°F), and percent acidulation (78% to 106%). None of these variables had much effect on \( \text{U}_3\text{O}_8 \) and \( \text{P}_2\text{O}_5 \) recoveries, which were 95% to 97%. \( \text{Al}_2\text{O}_3 \) recoveries, which were lower and quite erratic, appeared to be somewhat better at 140°F than with no heat addition.
In the final series of tests, atmospheric digestion for 20 hours at 160°F was followed by one hour of digestion in the autoclave at 10 to 50 psig and 220° to 290°F. The data obtained from a few short runs indicated that filtering rates were improved by 40% to 80%. The best average rate obtained was 2.4 gal/(hr)(ft²) for a four-hour filtering run after digestion at 20 psig. This rate represents a filtering area of 5 square feet per ton of dry leached zone processed per day.

Based on dissolution recoveries as noted heretofore and on recoveries from the ore dressing section (RMO-2045), the overall recoveries, from raw leached zone ore through filtration, during the last six months of operation (processing only the -200 mesh fraction) were approximately as follows:

<table>
<thead>
<tr>
<th>Pilot Plant Type</th>
<th>Overall Recovery, Lb/Ton dry leached zone</th>
<th>H₂SO₄ Used Ton/lb U₃O₈ recovered</th>
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<tr>
<td>Ore Sample Number</td>
<td>U₃O₈</td>
<td>P₂O₅</td>
</tr>
<tr>
<td>7, 8</td>
<td>0.19</td>
<td>100</td>
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<tr>
<td>9</td>
<td>0.15</td>
<td>90</td>
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</table>

Lower overall recoveries and higher acid consumption at 70% acidulation in the bottom line of the above tabulation resulted mainly from the poorer quality of ore sample 9 as compared to samples 7 and 8.
A. Period May 5, 1952 through February 26, 1953.

Pressure Digestion

Laboratory work (see Report RMO 2006) indicated that the optimum conditions for sulfuric acid digestion of the -200 mesh fraction of leached zone ore were approximately 15 minutes (or more) at 160 psig, with 80 percent acidulation and an acid strength of 35 percent. Consequently, during this first period of operations of the acidulation and filtration sections of the pilot plant, the digestions were carried out under conditions approximating these optimums.

During the period 5/5/52 through 1/30/53, all of the digestions were carried out batchwise. The digestion section was operated approximately 800 hours during this period, producing some 400 batches of 30 to 40 gallons each. During the period February 2 through 27, continuous pressure digestion was attempted. The flowsheet for this process is shown in figure 1. The autoclave was the same for either batch or continuous operation.

In a typical batch operation, a 30 percent solids feed slurry was first prepared in a slurry mix tank. The underflow from the thickener in the ore dressing section during this period contained approximately 20 to 25 percent solids. Part of this underflow was filtered on a rotary vacuum filter to give a 50 percent solids cake, which was then mixed with more underflow in the correct proportions to give a 30 percent solids slurry for digestion. Some of the earliest runs were made by charging the autoclave directly, first putting the required amount of 66°Be sulfuric acid into it (approximately five gallons), and then adding 30 percent slurry to make a total batch of 40 gallons.
Fig. 1
Flow Sheet for Continuous Pressure Digestion of -200 Mesh Leached Zone Slurry, Feb. 1953
Although the autoclave contained a 1/2 HP agitator, it was decided that mixing was unsatisfactory with this procedure, so that in later runs, the acid and slurry were mixed and brought up to approximately 250°F in a steam-jacketed tank before charging to the autoclave under 100 psig air pressure. The majority of the batches were digested for one-half to two hours at 360°F and 160 psig and an acidulation level of 70 to 100 percent.

For the continuous operation, as shown in figure 1, a concentric tube mixer was used in which the acid was continuously fed into the slurry stream through a small orifice. The resulting mixture was fed to the bottom of the autoclave and the acidulated slurry was discharged near the top of the autoclave, whence it passed to a receiver and on to the filtration section. The autoclave temperature leveled out at 310°F at 160 psig. Considerable experimentation with mixing tube arrangements was carried out. A small acid orifice was best while running, but a small orifice would plug up during down periods.

With either batch or continuous operation, corrosion of the autoclave and lines, which were all made of Carpenter 20 stainless steel, was severe, whether the acid was pre-heated or not. Lines had to be replaced after two or three shifts of continuous operation. Plugging of the mixing tube, lines, and valves was also a continual problem. The digestion section could not keep up with the ore dressing and filtration sections. For these reasons, the next period was devoted to atmospheric digestion.

The filtration setup, which was relatively constant throughout all pilot plant operations, is shown in figure 2. It consisted of four rotating-disc vacuum filters operated countercurrently. These were 3-foot diameter, two-disc Eimco filters. Each complete filter disc contained eight
FIG 2
FLOW SHEET FOR FILTRATION OF DIGESTED SLURRY,
FOUR-STAGE COUNTERCURRENT VACUUM FILTRATION

Digested Slurry

F-1

F-2

No. 2 Filterate

No. 1 Filterate Prod.

To further processing

O.F.

To digestors if desired

No. 3 Filterate

F-3

F-4

No. 4 Cake to waste

F - Filter, vacuum, Eimco rotating disc.
M - Mixer for feed to F-1
V - Vacuum receivers for filtrate
R - Repulper steam jacketed & equipped with 2 H.P. agitator
RP - Repulped slurry pump, roller tube type
OF - Overflow return line

MP - Mixed slurry pump, roller-tube type
FP - Filtrate pump, roller-tube type

-12-
sectors of 1.5 square foot filtering area each, so that the maximum area per filter was 24 square feet. The filters were operated with 50% submergence, so that the form time was one-half the total cycle time. The filtrate from the first filter passed on to the alum, uranium, and phosphate recovery sections which are described in separate reports. The cake from the fourth filter was discarded as waste.

During this period of pressure digestions, there was insufficient feed to the filters to permit any evaluation of continuous performance. In many instances, only one or two of the four filters were operated, and those only intermittently. In general, feed slurry pan temperatures were held at 140° to 160°F, and a number one filtrate of approximately 1.3 specific gravity was produced as desired. Filtration rates were usually in the 0.4 to 1.5 gal/(hr)(ft²) range.
B. Period February 27 through March 13, 1953.

Batch Atmospheric Digestion

After the pressure digestion system was temporarily abandoned for the reasons listed heretofore, a system was set up for batch digestions at atmospheric pressure. The nominal conditions for most batches were 80 percent acidulation at 180°F with a retention time of 24 hours. Feed slurry, acid, and heating steam were adjusted to obtain these conditions and to produce a 30% solids acidulated slurry feed to the filtration section. The system was operated approximately 300 hours, producing 14 batches of acidulated slurry which averaged 850 to 900 gallons per batch. Percentage extractions of values were approximately: U₃O₈, 95%; P₂O₅, 96%; Al₂O₃, 90%.

The slurry for digestion was prepared in a 1000-gallon stainless steel tank by mixing stored slurry from the ore dressing section with filtered solids as required to give a 30 to 34 percent solids slurry. Acidulation was carried out in either of two 1000-gallon stainless steel tanks set up in parallel. (In a typical batch, 112 gallons of 66°Be sulfuric acid was added to 757 gallons of a 30% solids slurry.) A 500-gallon rubber-lined mild steel tank was used to evaporate excess water from the acidulated slurry and to serve as a surge tank for feeding the filtration section. Recycle lines were installed so that the latter tank could also be used for heating during the digestion period if desired.

Three-stage countercurrent filtration was used during this period. The pan temperatures were held at approximately 140°F. Disc speeds in the range 1.0 to 1.5 RPM appeared to give the best filtering rate. Water was fed to the second repulper (which fed the third filter) at approximately 0.15 gallons per minute. Under these conditions, filtering rates of 0.8 to 1.4 gallons per hour per square foot were obtained. Attempts
were made to balance the three filters by plugging off sectors in the second and third filters, but these attempts were only partially successful. The main mechanical difficulties encountered were (1) failures of roller-tube pumps, (2) plugging of repulpers, and (3) torn filter bags.

Laboratory filtrations of feed slurries for the first filter indicated that the following percentage recoveries of values in the product were possible: $U_3O_8$, 99%; $P_2O_5$, 95%; $Al_2O_3$, 85%. However, no recoveries could be calculated on pilot plant filtration because of intermittent, erratic operations due to mechanical difficulties and erratic feed from the digestion section.
C. Periods March 16 through May 8, and May 27 through July 16, 1953

Continuous Atmospheric Digestion

The acidulation section was rearranged for continuous operation by placing three 500-gallon rubber-lined mild steel tanks in series so that the first overflowed to the second and the second overflowed to the third, as shown in Figure 3. Each was supplied with a 2-HP agitator. The capacity to the overflow was approximately 420 gallons per tank, or 1260 gallons for the system. Retention times for the system were based on this volume divided by the withdrawal rate of the digested slurry (except for short periods when only two of the tanks were used).

Several changes in conditions were made during these periods, so that typical operating conditions and results for thirteen sub-periods have been summarized in Table I. During the start-up operations of sub-period 1, the data obtained were sketchy and erratic, so that the comparatively high filtering rate of 1.6 gal/(hr)(ft²) cannot be attributed to any one variable. Some points of interest among the other twelve sub-periods are noted in the following paragraphs.

Sub-periods 2 through 4 showed slight increases in extractions (except for Al₂O₃ in sub-period 3) as percent acidulation increased from 66 percent to 118 percent. Note, however, that acid strength also increased from 21 percent to 30 percent, and that an increase in acid strength would be expected to contribute to an increase in extractions (RMO's 2006 and 2031). Moreover, it must be pointed out that in controlled repeat analyses on a single sample (RMO 2031), it was found that the reproducibility of these analyses was no better than ±2%, so that the small variations in extractions shown in Table I are insignificant. It is perhaps more interesting to note that for all of the samples taken during periods of continuous atmospheric
Fig 3
Flow Sheet for Atmospheric Digestion Eqpt.,
Added Pressure Digestion Option & Alternate
Feeding Arrangements Indicated by Dashed Lines

No. 2 Filtrate

-200 mesh slurry
From OEE dressing

STEAM

66° Be (93%) H₂SO₄

STEAM

No. 1 Digester

No. 2 Digester

No. 3 Digester

STEAM

ADDITIVES

STEAM

AUTOClave

Digested slurry → Filtration

Aroclor IN

Aroclor OUT

M.R. Pump

Surge Tank

Digested slurry → Filtration
digestion, the percent extractions fell in the following ranges: \( \text{U}_3\text{O}_8, \ 90-99\%; \ \text{P}_2\text{O}_5, \ 88-98\%; \ \text{Al}_2\text{O}_3, \ 60-90\% \). These recoveries compare favorably with those obtained in the laboratory for similar feeds and conditions.

For all continuous atmospheric digestion periods, the total retention time in the digestors was at least ten hours, and it was usually nearer 24 hours. As expected from laboratory tests and confirmed by the 50-gallon batch atmospheric digestions reported hereinafter, no effect of time can be detected in this range.

No change in extractions or filtering rate due to digestion temperature can be isolated from the data in Table I. It will be noted that two average temperature levels were generally used: 170°F, wherein the individual digestor temperatures were usually staggered in the decreasing order 190°, 170°, and 150°F; and 127°F, wherein the individual digestor temperatures were 130°, 130°, and 120°F, respectively. The shift to the lower temperatures was made after microscopic examination of the solids in the extracted slurry that revealed the poor-filtering hemihydrate of calcium sulfate \((\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O})\) was being formed at the higher temperatures in preference to the desired gypsum \((\text{CaSO}_4 \cdot 2\text{H}_2\text{O})\). It was found that neither diluting the acid by returning the No. 2 filtrate to the acid in order to prevent local over-temperatures, nor adding wet process phosphoric acid gypsum cake to seed out gypsum crystals, prevented the formation of the hemihydrate at the higher temperature level. At the lower temperature level, 98% of the calcium sulfate was in the form of needle-shaped gypsum crystals, 65 to 75 microns in length. However, comparison of the filtering rates for sub-periods 6 to 13 with the earlier rates shows that no improvement was effected. The type of calcium sulfate crystal formed was apparently unimportant compared to the fact that a large portion of the -200 mesh fraction of leached zone ore reports to the -10 micron size (of which 30 to 40 percent is insolubles) which blinds filter cloths.
For the entire period, filtering rates fell in the range 0.9 to 1.6 gal/(hr)(ft$^2$), representing filtering areas of 7 to 13 square feet per ton of leached zone processed per day. Most of the variations in filtering conditions and in filtering rates were due to mechanical difficulties leading to poor physical control, such as mentioned for previous periods. In general, pan temperatures were held near 140°F, pickup vacuums were 18-22 in. Hg, and a disc speed of 1.43 rpm was maintained. Feeding conditions, including the rate of water addition to the last repulper, were adjusted in an attempt to obtain a specific gravity of 1.30 in the No. 1 filtrate, which was believed to be the optimum for succeeding operations. In sub-periods 3 and 4, where higher pan temperatures were used, slightly higher filtering rates were obtained; however, the low rates in sub-period 2 were believed to be due to the tendency of the pan slurry to set up like a gel, which in turn was attributed to low acidulation. Thus there is no clear-cut evidence of the effect of pan temperature. The ratios of filtering areas necessary to keep the four filters in balance varied considerably, but the most frequently used ratios were perhaps 8:7:5:3.

Comparison of sub-periods 4 and 5 shows that returning the filtrate from the second filter to the first digestor instead of to the feed mixer for the first filter had no effect on extractions. As mentioned heretofore in connection with digestion temperatures, this practice did not improve filtering rate by preventing the formation of calcium sulfate hemihydrate crystals. In fact, there was a slight decrease in filtering rate (compare sub-periods 4 and 5) which may have been caused by decreased pan temperatures. It appears that splitting the total feed of slurry, acid, and No. 2 filtrate in 20-80 proportions between the first and second digestors, respectively, may have been responsible for again raising the filtering rate slightly in sub-period 6. However, splitting the
slurry in 1/3 - 2/3 proportions in sub-period 13 had no beneficial effect (as compared to the regular procedure in 12). Both of these feed-splitting techniques allow part of the slurry to have a much greater digestion time without changing the overall average digestion time, but the latter technique of splitting only the slurry feed increases the acid strength in the first digestor, whereas splitting the total feed does not.

The various materials added to the third digestor in sub-periods 8 to 10 in attempts to increase the calcium sulfate content of the filter feed had no appreciable effect on filtering rates as compared to the previous sub-periods 6 and 7. The effect of these additions was to cut back on the overall acidulation for succeeding operations after digesting at approximately 100 percent acidulation.

The overall recoveries from raw ore through filtration were approximately 0.19, 100, and 130 pounds per ton of dry ore for U₃O₈, P₂O₅, and Al₂O₃, respectively, when good -200 mesh recoveries were obtained in the ore dressing section with either ore sample 7 or 8. The consumption of 66°Be sulfuric acid per pound of U₃O₈ recovered varied from approximately 1.3 tons at 70 percent acidulation to 2 tons at 120 percent acidulation.


<table>
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<th>Dates and Special Features</th>
<th>DIVIDED ONE</th>
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<th>DIGESTION</th>
<th>DIGESTION</th>
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<td>Average Digestion Conditions</td>
<td>Percent Extracted &amp; Calculated by Laboratory Filtration</td>
<td>No. 1 Filter Conditions</td>
<td>Filtration Rate, Gal./Hr. (eqP/L)</td>
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<tr>
<td>11</td>
<td>6/20/53 - 7/5/53</td>
<td>8</td>
<td>Sodium Silicate</td>
<td>0.34</td>
<td>30.8</td>
<td>102</td>
<td>72</td>
<td>0.23</td>
<td>22</td>
<td>61</td>
<td>16</td>
<td>131</td>
<td>95</td>
<td>0.8</td>
</tr>
<tr>
<td>12</td>
<td>7/4/53 - 7/11/53</td>
<td>8</td>
<td>Sodium Silicate</td>
<td>0.34</td>
<td>31.0</td>
<td>112</td>
<td>76</td>
<td>0.22</td>
<td>22</td>
<td>71</td>
<td>22</td>
<td>140</td>
<td>135</td>
<td>0.9</td>
</tr>
<tr>
<td>13</td>
<td>7/11/53 - 7/18/53</td>
<td>8</td>
<td>Sodium Silicate</td>
<td>0.34</td>
<td>29.2</td>
<td>124</td>
<td>105</td>
<td>0.26</td>
<td>21</td>
<td>(73)</td>
<td>17</td>
<td>174</td>
<td>157</td>
<td>0.9</td>
</tr>
</tbody>
</table>

(1) Periods for acidulation, extracted slurry analysis, and filtration chosen to allow for time lag between steps. (2) Values in parentheses were estimated from partial information. (3) All values represent average conditions over one eight-hour shift for that particular operation. (4) Values, except for the second column under filtering rate, do not necessarily represent the average conditions for the entire sub-period. (5) Slurry, acid, and No. 2 filtrate split 20-40 between first and second digestors. (6) Material indicated added to third digestor in attempt to improve filtering. (7) Either Calgon solution, sodium silicate solution, or both used. (8) All acid added to first digestor, but slurry split 1/3-1/3 between first and second digestors. (9) Extracted slurry sample was vacuum filtered, cake was repulped with water and refiltered. Percent extraction was calculated by dividing weight recovered in total filtrate by sum of weight in total filtrate and dried cake. (10) Percent recovery calculated by dividing average wt. rate in filtrate by sum of average weight rates in filtrate and cake discarded from last filter.
D. Period May 12 through May 26, 1953.

Fifty-Gallon Batch Atmospheric Digestions.

In order to study the effects of digestion time, temperature, and percent acidulation on the recoveries of $U_3O_8$, $P_2O_5$, and $Al_2O_3$ under more carefully controlled conditions, it was decided to make 50-gallon batch digestions at atmospheric pressure. These batches were made by adding either 6, 7.25, or 8.5 gallons of 66°Be sulfuric acid to 138 pounds of -200 mesh solids and approximately 322 pounds of water (30 percent slurry). The above quantities of acid were expected to give acidulations of 70, 85, and 100 percent and acid strengths of 21, 24, and 27 percent, respectively. Any water lost by evaporation was replaced periodically. For batches to which no heat was added (figures 4 and 5), the temperature first rose to 140° to 160°F in the first few hours after mixing, fell to 100°F in approximately 12 hours, and asymptotically approached room temperature of 85°F thereafter. Other batches were kept at 140°F in a steam-jacketed stainless steel pot (figures 6 to 8). Samples were taken two hours after mixing and every four hours thereafter up to 22 hours or longer. These samples were filtered immediately by the repulping procedure described in RMO 2031.

Figures 4 to 8 show that none of these variables had much effect on the $U_3O_8$ and $P_2O_5$ recoveries, which were generally in the 95 to 97 percent range. The alumina recoveries were lower and quite erratic; about all that might be said in this regard is the broad generalization that alumina recovery was somewhat better at 140°F than when no heat was added.
As seen by the acidulation values (mean value from all samples and probable error of mean value) given on the figures, the actual acidulations as determined by laboratory analysis varied considerably from the 85, 100, 70, 85, and 100 percent, respectively, which were expected for these five batches. These discrepancies may be ascribed to: (1) variations in slurry composition, even though the dressed ore came from ore sample number 8 in all cases; (2) errors in mixing, (3) difficulty in obtaining representative samples for analysis; and (4) errors in analysis. With regard to item (4), it might be pointed out that percent acidulation is calculated from the total Al₂O₃, CaO, Fe₂O₃, and SO₄ contents of the samples, hence the probable error of the mean value (which is only 1/2 to 1/3 of the probable error for a single sample) reflects the variations in these totals, particularly those for Al₂O₃ and SO₄. Therefore, the erratic variations in alumina recoveries may be attributed largely to experimental errors.

Since laboratory data (RMO 2031) indicated that little change in recoveries with time would be expected for times greater than 10 hours, the following summary values with regard to temperature and acidulation have been calculated by averaging all samples taken from a given batch after ten hours or longer. These values demonstrate the points made above that U₃O₈ and P₂O₅ recoveries were not affected by temperature or acidulation but that Al₂O₃ recoveries were somewhat better at 140°F.

<table>
<thead>
<tr>
<th>Temperature Condition</th>
<th>Percent Acidulation</th>
<th>Percent Extraction after 10 hours or longer</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>U₃O₈</td>
</tr>
<tr>
<td>No heat added</td>
<td>85</td>
<td>98</td>
</tr>
<tr>
<td></td>
<td>94</td>
<td>97</td>
</tr>
<tr>
<td>140°F</td>
<td>79</td>
<td>97</td>
</tr>
<tr>
<td></td>
<td>97</td>
<td>96</td>
</tr>
<tr>
<td></td>
<td>106</td>
<td>98</td>
</tr>
</tbody>
</table>
**Effect of Digestion Time on Recoveries**

**H₂SO₄ Digestion of -200 Mesh Leached Zone at Atmospheric Pressure**

- Digestion Temperature: No heat added.
- Acidulation: 85.0 ± 1.7%

![Graph showing percent extraction vs. digestion time for U₃O₈, P₂O₅, and Al₂O₃.](image)

**Percent Extraction vs. Digestion Time, Hrs.**
FIG. 5

EFFECT OF DIGESTION TIME OF RECOVERIES

H₂SO₄ DIGESTION OF 200 MESH LEACHED ZONE AT ATMOSPHERIC PRESSURE

DIGESTION TEMPERATURE: NO HEAT ADDED
ACIDULATION: 93.8 ± 0.8%

- - - U₃O₈  - - - P₂O₅  - - - Al₂O₃

PERCENT EXTRACTION

DIGESTION TIME, HRS.

-26-
FIG. 6

EFFECT OF DIGESTION TIME ON RECOVERIES

$\text{H}_2\text{SO}_4$ DIGESTION OF -200 MESH LEACHED ZONE AT ATMOSPHERIC PRESSURE

DIGESTION TEMPERATURE: 140°F
ACIDULATION: 78.5 ± 2.4 %

- - - U$_3$O$_8$    -- P$_4$O$_5$    - - - Al$_2$O$_3$

PERCENT EXTRATION

0  5  10  15  20  25

DIGESTION TIME, HRS.
Fig. 7

Effect of Digestion Time on Recoveries

H₂SO₄ Digestion of -200 mesh leached zone at atmospheric pressure

Digestion Temperature: 140°F
Acidulation: 97.4 ± 3.3%

- U₂O₈
- - - P₂O₅
△ Al₂O₃

Percent Extraction

Digestion Time, Hrs.
FIG. 8

EFFECT OF DIGESTION TIME ON RECOVERIES

H₂SO₄ DIGESTION OF -200 MESH LEACHED ZONE AT ATMOSPHERIC PRESSURE

DIGESTION TEMPERATURE: 140°F
ACIDULATION: 106.3 ± 1.5%

- - - U₃O₈  - - - - P₂O₅  - - - - - - - - Al₂O₃

PERCENT EXTRACTION

DIGESTION TIME, HRS.

-29-
E. Period July 20 through August 7, 1953

Atmospheric Digestion Followed by Pressure Digestion

The objective of this period was to determine whether better filtering rates could be obtained with slurry which was first digested at atmospheric pressure and then pumped through the autoclave at pressures in the range 10 to 50 psig. The major operating conditions and results for two series of runs are shown in table II. For both series, the atmospheric digestors were run continuously and the pressure changes between runs were made without emptying the autoclave. The extracted slurry from the autoclave was filtered on a single filter. Countercurrent filtration was simulated by returning 0.25 gal/min. of diluted filtrate (specific gravity 1.2) to the filter feed mix tank, where it was mixed with the extracted slurry. In the first series, the filtration was carried out continuously except for a shutdown between the 35 and 50 psig runs to clean the filters and change bags. In the second series, all filtration runs were of four-hour duration, and the filter bags were inspected between runs and replaced as needed.

The feeding conditions to the digestion section were similar to those of period C-13; the slurry was split 1/3-2/3 between the first two digestors, and approximately 70 percent acidulation was used. It should be noted, however, that a different ore sample, No. 9, was used. The 30 percent solids feed slurry prepared from this ore by the ore dressing section contained a considerable amount of +200 mesh solids; in fact, it must have contained essentially all of the -100 mesh solids. Since the analyses of these ore samples (RMO 2045) show that the -100+200 mesh fraction of ore 9 was high in unavailable (kaolinitic) alumina, it might be expected that alumina recoveries as determined by the repulped sample technique would be poorer with ore 9 than with ore 8. Such was the case,
as seen by comparing the recoveries in Table II with those in Table I for similar operating conditions.

The last three columns of Table II show that some improvement in filtering rates did result from the added pressure digestion. For the first series, the improvement is best seen by comparing the leaf tests before and after pressure digestion; an increase of approximately 40 percent in the filtering rate was obtained at 20, 35, and 50 psig. For the second series, the pilot plant filtering data suggest that there is an optimum pressure for maximum filtering rate in the neighborhood of 20 to 30 psig. The average filtering rate of 2.4 gal/(hr)(sq. ft.) at 20 psig (which was also obtained by leaf tests) represents an 80 percent improvement over the 1.3 gal/(hr)(sq. ft.) obtained with no pressure digestion. These results are by no means conclusive, however, because the four-hour test runs were too short to obtain steady rates.

The average overall recoveries obtained during these operations with ore sample 9 were approximately 0.15, 90, and 92 pounds per ton of dry ore for U₃O₈, P₂O₅, and Al₂O₃, respectively. The consumption of 66 Be sulfuric acid was therefore approximately 1.7 tons per pound of U₃O₈ in the filtrate.

The pressure digestion operations were again shut down as a result of difficulties to those described in period A.
<table>
<thead>
<tr>
<th>RUN</th>
<th>Auto-</th>
<th>Date and</th>
<th>Sample</th>
<th>Disper-</th>
<th>Times</th>
<th>Slurry</th>
<th>Average Digestor Conditions</th>
<th>Percent Extraction</th>
<th>FILTERING</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>clave</td>
<td>Shift of</td>
<td>No.</td>
<td>sants</td>
<td>Lar</td>
<td>Feed</td>
<td>Valor</td>
<td>Consumption</td>
<td>Laboratory Filtration</td>
</tr>
<tr>
<td></td>
<td>PNSD</td>
<td>Filtration</td>
<td></td>
<td></td>
<td>used</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>Continuous filtration except for 3-hour shutdown 7/31/53 to clean filters and change bags.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>7/30/53 - 2</td>
<td>9</td>
<td>18</td>
<td>0.37</td>
<td>31.1</td>
<td>109</td>
<td>71</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>35</td>
<td>7/30/53 - 3</td>
<td>9</td>
<td>18</td>
<td>0.37</td>
<td>27.7</td>
<td>106</td>
<td>71</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>7/31/53 - 1</td>
<td>9</td>
<td>18</td>
<td>0.37</td>
<td>28.0</td>
<td>104</td>
<td>73</td>
<td>0.26</td>
</tr>
<tr>
<td>2</td>
<td>Filtration in 4-hour periods only. All bags inspected between runs.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>none</td>
<td>8/4/53 - 1</td>
<td>9</td>
<td>18</td>
<td>0.40</td>
<td>28.0</td>
<td>111</td>
<td>80</td>
<td>0.29</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>8/4/53 - 3</td>
<td>9</td>
<td>24</td>
<td>0.31</td>
<td>27.7</td>
<td>110</td>
<td>74</td>
<td>0.22</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>8/5/53 - 1</td>
<td>9</td>
<td>(20)</td>
<td>0.37</td>
<td>27.6</td>
<td>104</td>
<td>74</td>
<td>0.26</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>8/6/53 - 1</td>
<td>9</td>
<td>19</td>
<td>0.36</td>
<td>30.3</td>
<td>105</td>
<td>71</td>
<td>0.24</td>
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<tr>
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<td>40</td>
<td>8/6/53 - 2</td>
<td>9</td>
<td>20</td>
<td>0.36</td>
<td>30.2</td>
<td>106</td>
<td>74</td>
<td>0.27</td>
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<tr>
<td></td>
<td>50</td>
<td>8/6/53 - 3</td>
<td>9</td>
<td>19</td>
<td>0.36</td>
<td>31.2</td>
<td>98</td>
<td>73</td>
<td>0.28</td>
</tr>
</tbody>
</table>

(1) The feed slurry was split 1/3 - 2/3 between the first two digestors, as in period C-13.
(2) Single-stage filtration, simulating counter-current filtration.
(3) Standard test leaf filter, 0.1 sq. ft., with 20-second pick-up and 20-second dry times at 15 in. Hg vacuum.
(4) Ditto above, except 30-second times at 20 in. Hg vacuum.
REFERENCES


