April 21, 1940.

Master's Thesis.
on Treatment of Cobalt Ore.

By

Raymond R. Taylor.

is acceptable to the Department
of Mining and Metallurgy.

Head of Dept.
An Investigation of Treatment Methods
of Cobalt Ore from the Gem Mine
Bridge River

by

Raymond Russell Taylor

A Thesis submitted in Partial Fulfilment of
the Requirements for the Degree of

MASTER OF APPLIED SCIENCE

in the Department
of

METALLURGY

The University of British Columbia
April, 1941
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</tr>
</tbody>
</table>
An Investigation of Treatment Methods of Cobalt Ore from the Gem Mine Bridge River

I. INTRODUCTION

The purpose of this investigation was to determine a suitable treatment process by which both the cobalt and gold in the ore of the Gem Mine, Bridge River, could be recovered separately, and as marketable products.

II. ACKNOWLEDGMENTS

The writer wishes to express his appreciation of the assistance received from Mr. Frank A. Forward, Professor of Metallurgy, University of British Columbia, in the preparation of this work.

Acknowledgment is also made to Mr. Alfred G. Lyle, for his useful suggestions concerning testing and assay work.
III. **SUMMARY OF TESTS**

A. **Roasting**

(a) **Primary Calcines**

**Characteristics of Primary Roasts**

<table>
<thead>
<tr>
<th>No.</th>
<th>Temperature Start</th>
<th>Temperature Finish</th>
<th>Time</th>
<th>% Reduction in weight</th>
<th>% charcoal added</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>300° C.</td>
<td>750° C.</td>
<td>2 - 40</td>
<td>33.8%</td>
<td>7%</td>
</tr>
<tr>
<td>2</td>
<td>400</td>
<td>800</td>
<td>3 - 00</td>
<td>37.7</td>
<td>3</td>
</tr>
<tr>
<td>3</td>
<td>550</td>
<td>800</td>
<td>2 - 45</td>
<td>38.0</td>
<td>-</td>
</tr>
<tr>
<td>4</td>
<td>550</td>
<td>800</td>
<td>2 - 45</td>
<td>38.3</td>
<td>-</td>
</tr>
<tr>
<td>5</td>
<td>550</td>
<td>700</td>
<td>1 - 30</td>
<td>24.4</td>
<td>-</td>
</tr>
<tr>
<td>6</td>
<td>550</td>
<td>800</td>
<td>1 - 30</td>
<td>31.5</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>5550</td>
<td>800</td>
<td>1 - 30</td>
<td>31.1</td>
<td>-</td>
</tr>
<tr>
<td>8</td>
<td>550</td>
<td>800</td>
<td>1 - 30</td>
<td>33.0</td>
<td>-</td>
</tr>
<tr>
<td>9</td>
<td>800</td>
<td>800</td>
<td>1 - 30</td>
<td>39.2</td>
<td>-</td>
</tr>
<tr>
<td>10</td>
<td>820</td>
<td>820</td>
<td>2 - 00</td>
<td>40.5</td>
<td>-</td>
</tr>
</tbody>
</table>

Calcines 3 & 4 were combined and assayed.

Calcines 6, 7 & 8 were combined and assayed.

**Primary Calcine Assays:**

<table>
<thead>
<tr>
<th>No.</th>
<th>Co %</th>
<th>Fe %</th>
<th>SiO₂ %</th>
<th>As %</th>
<th>S %</th>
<th>CaO %</th>
<th>Gold oz/ton</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.70</td>
<td>32.4</td>
<td>22.0</td>
<td>6.2</td>
<td>1.26</td>
<td>1.9</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>8.04</td>
<td>33.4</td>
<td>22.1</td>
<td>4.0</td>
<td>.55</td>
<td>1.9</td>
<td>-</td>
</tr>
<tr>
<td>3+4</td>
<td>8.56</td>
<td>35.5</td>
<td>22.2</td>
<td>5.68</td>
<td>0.20</td>
<td>1.9</td>
<td>1.80</td>
</tr>
<tr>
<td>5</td>
<td>6.88</td>
<td>29.1</td>
<td>18.1</td>
<td>21.1</td>
<td>7.06</td>
<td>1.6</td>
<td>1.47</td>
</tr>
<tr>
<td>6+7+8</td>
<td>7.76</td>
<td>32.3</td>
<td>20.1</td>
<td>14.1</td>
<td>4.28</td>
<td>1.7</td>
<td>1.62</td>
</tr>
<tr>
<td>9</td>
<td>8.61</td>
<td>36.0</td>
<td>22.5</td>
<td>4.82</td>
<td>0.05</td>
<td>2.0</td>
<td>1.82</td>
</tr>
<tr>
<td>10</td>
<td>8.90</td>
<td>36.6</td>
<td>22.8</td>
<td>3.46</td>
<td>0.10</td>
<td>2.0</td>
<td>1.87</td>
</tr>
</tbody>
</table>
Note:

The dusting loss of gold and cobalt amounted to, 1.5 - 4.0%. This dusting loss would be largely eliminated if the ore were treated in an Edwards Roaster.

A fast high temperature roast, such as Test 9 or 10, produces a calcine that has a maximum removal of arsenic, and a maximum reduction in weight. A calcine of this type is suitable for the preparation of a sulphated calcine.

(b) Sulphated Calcines

Characteristics of Sulphated Calcine Roasts

<table>
<thead>
<tr>
<th>No.</th>
<th>Temperature Start</th>
<th>Temperature Finish</th>
<th>Time hr. - min.</th>
<th>% Increase in weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>650°C.</td>
<td>650°C.</td>
<td>1 - 20</td>
<td>5.5%</td>
</tr>
<tr>
<td>2</td>
<td>650</td>
<td>650</td>
<td>1 - 40</td>
<td>19.0%</td>
</tr>
<tr>
<td>3</td>
<td>650</td>
<td>650</td>
<td>1 - 30</td>
<td>27.5%</td>
</tr>
<tr>
<td>4</td>
<td>350</td>
<td>650</td>
<td>1 - 35</td>
<td>32.0%</td>
</tr>
<tr>
<td>5</td>
<td>450</td>
<td>650</td>
<td>1 - 40</td>
<td>34.0%</td>
</tr>
<tr>
<td>6</td>
<td>450</td>
<td>650</td>
<td>1 - 30</td>
<td>26.2%</td>
</tr>
<tr>
<td>7A, B, C, D &amp; E</td>
<td>450</td>
<td>450</td>
<td>0 - 30</td>
<td>65.7%</td>
</tr>
<tr>
<td>7F</td>
<td>450</td>
<td>750</td>
<td>2 - 30</td>
<td>5.7%</td>
</tr>
<tr>
<td>7G</td>
<td>650</td>
<td>650</td>
<td>1 - 45</td>
<td>33.7%</td>
</tr>
<tr>
<td>8A</td>
<td>450</td>
<td>450</td>
<td>0 - 30</td>
<td>53.5%</td>
</tr>
<tr>
<td>8B</td>
<td>550</td>
<td>675</td>
<td>1 - 30</td>
<td>22.0%</td>
</tr>
<tr>
<td>9A</td>
<td>450</td>
<td>450</td>
<td>0 - 30</td>
<td>53.3%</td>
</tr>
<tr>
<td>9B</td>
<td>675</td>
<td>650</td>
<td>1 - 00</td>
<td>31.5%</td>
</tr>
</tbody>
</table>
### Sulphating Charges

<table>
<thead>
<tr>
<th>No.</th>
<th>Primary Calcine No.</th>
<th>Calcine</th>
<th>H$_2$SO$_4$</th>
<th>Charcoal</th>
<th>Salt</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>-100 mesh</td>
<td>50 c.c.</td>
<td>2 gm.</td>
<td>2 gm.</td>
</tr>
<tr>
<td>1</td>
<td>sulphatized calcine</td>
<td>100 gm.</td>
<td>50 c.c.</td>
<td>2 gm.</td>
<td>2 gm.</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>100 gm.</td>
<td>50 c.c.</td>
<td>2 gm.</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>100</td>
<td>50 c.c.</td>
<td>2 gm.</td>
<td>5</td>
</tr>
<tr>
<td>4</td>
<td>9</td>
<td>300</td>
<td>150</td>
<td>6</td>
<td>15</td>
</tr>
<tr>
<td>5</td>
<td>9</td>
<td>100</td>
<td>50</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>9</td>
<td>100</td>
<td>35</td>
<td>2</td>
<td>5</td>
</tr>
<tr>
<td>7a, b, c, d &amp; e</td>
<td>10</td>
<td>500</td>
<td>50</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>7F</td>
<td>7a, b, c, d, e</td>
<td>2000</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>7G</td>
<td>7a, b, c, d, e</td>
<td>985</td>
<td>-</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>8</td>
<td>9</td>
<td>300</td>
<td>150</td>
<td>6</td>
<td>9</td>
</tr>
<tr>
<td>9</td>
<td>9</td>
<td>300</td>
<td>150</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

### Sulphated Calcine Assays

<table>
<thead>
<tr>
<th>No.</th>
<th>Total Co %</th>
<th>Fe %</th>
<th>As %</th>
<th>SiO$_2$ %</th>
<th>Water Soluble Co %</th>
<th>Fe %</th>
<th>As %</th>
<th>Gold oz/ton</th>
<th>Recovery % Co</th>
<th>% Au</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.4</td>
<td>28.5</td>
<td>14.5</td>
<td>17.8</td>
<td>1.94</td>
<td>0.25</td>
<td>0.15</td>
<td></td>
<td></td>
<td>28.2</td>
</tr>
<tr>
<td>2</td>
<td>6.75</td>
<td>29.6</td>
<td>-</td>
<td>18.5</td>
<td>5.18</td>
<td>0.30</td>
<td>11</td>
<td></td>
<td></td>
<td>77.0</td>
</tr>
<tr>
<td>3</td>
<td>6.30</td>
<td>26.1</td>
<td>17.5</td>
<td>17.5</td>
<td>5.73</td>
<td>0.25</td>
<td>-</td>
<td></td>
<td></td>
<td>91.0</td>
</tr>
<tr>
<td>4</td>
<td>6.39</td>
<td>26.6</td>
<td>2.12</td>
<td>17.1</td>
<td>6.08</td>
<td>0.20</td>
<td>0.42</td>
<td>1.38</td>
<td>95.0</td>
<td>100</td>
</tr>
<tr>
<td>5</td>
<td>6.25</td>
<td>25.8</td>
<td>1.51</td>
<td>17.0</td>
<td>5.84</td>
<td>1.20</td>
<td>-</td>
<td>1.36</td>
<td>93.5</td>
<td>100</td>
</tr>
<tr>
<td>6</td>
<td>6.88</td>
<td>27.5</td>
<td>2.86</td>
<td>17.8</td>
<td>5.74</td>
<td>0.05</td>
<td>-</td>
<td>1.45</td>
<td>83.5</td>
<td>100</td>
</tr>
<tr>
<td>7F</td>
<td>5.44</td>
<td>21.8</td>
<td>-</td>
<td>13.6</td>
<td>4.04</td>
<td>2.80</td>
<td>-</td>
<td></td>
<td></td>
<td>74.5</td>
</tr>
<tr>
<td>7G</td>
<td>8.06</td>
<td>32.2</td>
<td>-</td>
<td>20.2</td>
<td>2.20</td>
<td>0.05</td>
<td>-</td>
<td></td>
<td></td>
<td>27.3</td>
</tr>
<tr>
<td>7A</td>
<td>7.05</td>
<td>28.8</td>
<td>17.9</td>
<td>17.9</td>
<td>6.10</td>
<td>0.50</td>
<td>-</td>
<td></td>
<td></td>
<td>78.0</td>
</tr>
<tr>
<td>7B</td>
<td>5.50</td>
<td>22.4</td>
<td>13.9</td>
<td>4.00</td>
<td>2.00</td>
<td>-</td>
<td>-</td>
<td></td>
<td></td>
<td>71.5</td>
</tr>
<tr>
<td>7C</td>
<td>6.55</td>
<td>26.5</td>
<td>16.5</td>
<td>5.24</td>
<td>0.90</td>
<td>-</td>
<td>-</td>
<td></td>
<td></td>
<td>80.0</td>
</tr>
</tbody>
</table>
Notes:

The addition of salt and charcoal to the sulphating charge increases the amount of water soluble cobalt.

It seems necessary to have the arsenic content in the calcine used in the sulphating charge quite low if you are going to get ideal sulphating conditions.

The addition of salt and charcoal, as in test 7G, to the sulphated calcine after it has been baked at 450° C. for 30 minutes to drive off the excess acid, produces a more desireous working condition than adding them together as in test No. 4 because the free chlorine vapor produced by the violent reaction of the salt and acid is largely eliminated.

(c) Chloridized Calcines:

### Characteristics of Chloridized Calcine Roasts

<table>
<thead>
<tr>
<th>No.</th>
<th>Temperature °C.</th>
<th>Time hr. - min.</th>
<th>% Increase in weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>200°C. to 650°C.</td>
<td>5 - 00</td>
<td>- 1.9%</td>
</tr>
<tr>
<td>2</td>
<td>350</td>
<td>0 - 40</td>
<td>5.0%</td>
</tr>
<tr>
<td>3</td>
<td>350</td>
<td>1 - 00</td>
<td>5.0%</td>
</tr>
<tr>
<td>4</td>
<td>350</td>
<td>1 - 30</td>
<td>11.5%</td>
</tr>
</tbody>
</table>

### Chloridizing Charges

<table>
<thead>
<tr>
<th>No.</th>
<th>Calcine No.</th>
<th>Calcine Parts of Charge</th>
<th>HCl</th>
<th>Charcoal</th>
<th>Salt</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>155.5 gm. plus 86.5 gm. ore</td>
<td>-</td>
<td>-</td>
<td>37.9 gm.</td>
</tr>
<tr>
<td>2</td>
<td>9</td>
<td>100 gm.</td>
<td>15 c.c.</td>
<td>15 c.c.</td>
<td>37.9 gm.</td>
</tr>
<tr>
<td>3</td>
<td>9</td>
<td>100 gm.</td>
<td>30 c.c.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>6 + 7 + 8</td>
<td>100 gm.</td>
<td>30 c.c.</td>
<td>3 gm.</td>
<td>3</td>
</tr>
</tbody>
</table>
Chloridized Calcine Assays:

<table>
<thead>
<tr>
<th>No.</th>
<th>%Co.</th>
<th>%Fe.</th>
<th>%SiO₂</th>
<th>%Co.</th>
<th>%Fe.</th>
<th>%Co.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.20</td>
<td>-</td>
<td>-</td>
<td>0.10</td>
<td>Nil</td>
<td>1.4%</td>
</tr>
<tr>
<td>2</td>
<td>8.20</td>
<td>34.3</td>
<td>21.4</td>
<td>0.70</td>
<td>0.20</td>
<td>8.7%</td>
</tr>
<tr>
<td>3</td>
<td>8.20</td>
<td>34.3</td>
<td>21.4</td>
<td>1.38</td>
<td>0.20</td>
<td>17.0%</td>
</tr>
<tr>
<td>4</td>
<td>6.95</td>
<td>28.9</td>
<td>18.0</td>
<td>1.60</td>
<td>1.80</td>
<td>23.1%</td>
</tr>
</tbody>
</table>

Note:

It does not seem possible to produce a chloridized calcine that will have a high water soluble cobalt content.

(d) Sulphatized Calcine

Characteristics of Sulphatizing Roast

<table>
<thead>
<tr>
<th>No.</th>
<th>Temperature°C</th>
<th>Time hr. - min.</th>
<th>%Decrease in weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>IA</td>
<td>250°C - 650°C</td>
<td>2 - 35</td>
<td>27.1%</td>
</tr>
<tr>
<td>IB</td>
<td>600 - 770</td>
<td>0 - 45</td>
<td>27.2%</td>
</tr>
</tbody>
</table>

Sulphatizing Charge

1000 gm. (-100 mesh) ore

Sulphatized Calcine Assays

<table>
<thead>
<tr>
<th>No.</th>
<th>%Co.</th>
<th>%Fe.</th>
<th>%SiO₂</th>
<th>%Co.</th>
<th>%Fe.</th>
</tr>
</thead>
<tbody>
<tr>
<td>IA</td>
<td>7.50</td>
<td>30.2</td>
<td>18.8</td>
<td>0.07</td>
<td>nil</td>
</tr>
<tr>
<td>IB</td>
<td>7.50</td>
<td>30.2</td>
<td>18.8</td>
<td>Trace</td>
<td>nil</td>
</tr>
</tbody>
</table>

Note:

Further work along this line might lead to encouraging results if a calcine could be made with a high sulphur low arsenic content. The temperature of the final sulphatizing roast should be kept below 450°C.
B. Leaching

(a) Sulphuric Acid Leaching

(i) Ore

**Assay**

Co. 5.45%

Fe. 22.0%

As. 36.1%

S. 11.1%

Co. 5.45% Hg S04, 500 c.c. H2SO4

Leach at 300°C.

for 40 min.

Cool to 230°C. &

filter

Dried Residue at

500 - 550°C. for 60 Min.

Divided Calcine into

two parts of 64.8 gm.

210 c.c. H2SO4

filtrate

Continued on

next page.

625°C. Roast for

2 hr. & 15 min.

54.0 gm. Calcine

Assay 625°C.

Watep Sol. Fe. 1.3%

" " " Co. 3.60%

" " " S. 10.35%

" 10% H2SO4 Sol. Fe. 20.75%

" " " Co. 4.15%

Total Fe. 20.9%

" " Co. 4.16%

" " S. 10.5%

" " As. 1.7%

Recovery Water Sol. Co. 86.5%

" Acid Sol. Co. 100.0%
8

AsgOg residue 16.8 gm.

Filtrate 210 c.c. H₄SO₄ Sat. CoSO₄ x H₂O

Assay 3.86 mg. Co per c.c.

Stand 9 days

Filtrate 210 c.c.

Assay 1.05 mg. Co per c.c.

ASgOg GoS0 4 xHg O x Xs

Leach, 250 c.c. water

As₂O₃ residue 16.8 gm.

Filtrate of Sol CoSO₄ x H₂O xls.

58.9 mg Co.

or 9.73% of Co.

in the ore used.

Note:

The recovery of water and acid soluble cobalt in this test is almost 100%.

The cost of such a leaching process would be too great because it would require three tons of acid per ton of ore.

(ii) Calcine

100 gm. No. 10 Primary Calcine were leached for one hour at 70°C. with 15 c.c. (conc) H₂SO₄ and 235 c.c. water.
Assays: Acid Filtrate

<table>
<thead>
<tr>
<th></th>
<th>Cold</th>
<th>Hot</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble Co.</td>
<td>1.23%</td>
<td></td>
</tr>
<tr>
<td>&quot; &quot; Fe.</td>
<td>1.5%</td>
<td></td>
</tr>
<tr>
<td>Total Co.</td>
<td>8.90%</td>
<td></td>
</tr>
<tr>
<td>Recovery of Co. = ( \frac{1.23 \times 100}{8.90} )</td>
<td>13.8%</td>
<td></td>
</tr>
</tbody>
</table>

(iii) Sulphatized Calcine

Cold Treatment:
20 gm. Sulphatized Calcine were leached for 30 min. at 20\(^\circ\)C. with 10 c.c. (conc.) \( \text{H}_2\text{SO}_4 \) and 100 c.c. water.

Hot Treatment:
20 gm. Sulphatized Calcine were leached for 30 min. at 65\(^\circ\)C. with 10 c.c. (conc.) \( \text{H}_2\text{SO}_4 \) and 100 c.c. water.

Assays: Acid Filtrates

<table>
<thead>
<tr>
<th></th>
<th>Cold</th>
<th>Hot</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water Soluble Co.</td>
<td>0.63%</td>
<td>2.07%</td>
</tr>
<tr>
<td>&quot; &quot; Fe.</td>
<td>0.92%</td>
<td>4.05%</td>
</tr>
<tr>
<td>Total Co.</td>
<td>7.50%</td>
<td>7.50%</td>
</tr>
<tr>
<td>Recovery of Water Sol Co.</td>
<td>8.40%</td>
<td>26.8%</td>
</tr>
</tbody>
</table>
(b) Water Leaching

(i) No.4 Sulphated Calcine

100 gms. of No.4 Sulphated Calcine were water leached with 250 c.c. water at 80°C for 30 min. and filtered.

Assay of Filtrate

Water soluble Co.  5.66%
"   " Fe.  2.6%
"   " As.  0.42%
Total   " Co.  8.50%

Recovery of Water Sol. Co.  90.0%

The residue from the above leaching was leached again with 250 c.c. water at 80°C for 90 min. and filtered.

Assay of Second Filtrate

Water soluble Co.  0.09%
"   " Fe.  Trace

Recovery of Water Sol. Co.  1.43%

Total " " " Co.  91.43%

Weight of Residue from 100 gm. No.4 Sulphated Calcine  62.0 gm.

Assay Residue:

Total As.  2.73%
"   Fe.  39.0%
"   SiO₂  27.9%
"   Co.  0.89%

Gold  2.22 oz/Ton

Gold Recovery  99.5%
(ii) **No. 7G₂ Sulphated Calcine**

305 gm. of No. 7G₂ Sulphated Calcine were water leached with 750 c.c. water at 80°C. for one hour and filtered.

**Assays of filtrate**

<table>
<thead>
<tr>
<th>Component</th>
<th>Water soluble</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co.</td>
<td>6.30%</td>
<td>6.65%</td>
</tr>
<tr>
<td>Fe.</td>
<td>0.62%</td>
<td></td>
</tr>
<tr>
<td>As.</td>
<td>0.43%</td>
<td></td>
</tr>
</tbody>
</table>

**Recovery of water sol. Co.** 95.0%

**Weight of Residue, from 305 gm. No. 7G₂ Sulphated Calcine**

= 218 gm.

**Assay: Residue**

<table>
<thead>
<tr>
<th>Component</th>
<th>Total</th>
<th>As.</th>
<th>3.64%</th>
</tr>
</thead>
<tbody>
<tr>
<td>&quot;</td>
<td></td>
<td>Co.</td>
<td>0.49%</td>
</tr>
<tr>
<td>&quot;</td>
<td></td>
<td>Fe.</td>
<td>37.50%</td>
</tr>
<tr>
<td>&quot;</td>
<td></td>
<td>SiO₂</td>
<td>23.80%</td>
</tr>
</tbody>
</table>
C. Precipitation

Test No. I.
The Precipitation of As., Fe., and Co. and the Roasting of Cobalt Mud to Cobalt Oxide.

100 c.c. of leach solution from No. 4 Sulphated Calcine Water Leach was treated as follows:

Precipitating Fe. and As.

The Fe. and As, were precipitated with 1.7 gm. Na\textsubscript{2}CO\textsubscript{3} and 3 c.c. of 5% H\textsubscript{2}O\textsubscript{2}. The solution was agitated (cold) for 15 min. and filtered.

Cobalt Precipitation

The cobalt solution was agitated with 3.6 gm. Na\textsubscript{2}CO\textsubscript{3} at 80°C for 30 minutes to precipitate the cobalt mud. Filtered off cobalt precipitate.

Roasting Cobalt Mud.

The purple cobalt mud was dried and ignited at 800°C for 45 minutes.

Weight of Cobalt Oxide 0.9975 Gm.

Cobalt contained in iron precipitate 0.2166 gm.

Cobalt content in Cobalt Oxide 73.1% Co.

Test No. II.

100 c.c. of leach solution from No. 4 Sulphated Calcine Water Leach was treated as follows.

Precipitating Fe. and As.

First Iron Precipitate

The cobalt solution was agitated with 1.25 gm. CaCO\textsubscript{3}
at 80°C. for 30 min. and then filtered.

**Second Iron Precipitate**

The above filtrate was divided into two parts. One portion was agitated with 0.5 gm. CaCO₃ at 80°C. for 15 min. and then filtered.

**Third Iron Precipitate**

The filtrate from the second iron precipitation was agitated with 0.5 gm. CaCO₃ and 5 c.c. of 3% H₂O₂ at 80°C. for 30 min. and then filtered.

**First Cobalt Precipitation**

The filtrate from the third iron precipitation was agitated with 1 gm. Na₂CO₃ at 80°C. for 20 min. to precipitate the cobalt. The solution was filtered and the CoCO₃ precipitate washed.

**Second Cobalt Precipitate**

The filtrate from the first cobalt precipitation was agitated with 0.5 gm. Na₂CO₃ at 80°C. for 15 minutes to precipitate the remainder of the cobalt.

The precipitates of cobalt carbonate were combined and dried.

**Roasting Cobalt mud.**

The cobalt mud was ignited at 800°C. for two hours.

**Weight of Cobalt oxide** 0.5380 gm.
Assays on Iron Precipitates

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td>0.0025</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td>0.0051</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td></td>
<td>0.0180</td>
</tr>
</tbody>
</table>

% Co. in Cobalt Oxide 74.5%

Tests No. III, IV, V and VI

These tests were run similar to Test No. II on various Water Leach Solutions from Sulphated Calcines. Lack of time prevented the assaying of the precipitated products.

The approximate composition of the partly ignited cobalt precipitates are as follows:

<table>
<thead>
<tr>
<th>Test</th>
<th>Co. in gm. Cobalt Oxide</th>
</tr>
</thead>
<tbody>
<tr>
<td>III</td>
<td>49.5% 22.0545</td>
</tr>
<tr>
<td>IV</td>
<td>67.5% 27.0315</td>
</tr>
<tr>
<td>V</td>
<td>72.1% 4.5729</td>
</tr>
<tr>
<td>VI</td>
<td>66.5% 17.879</td>
</tr>
</tbody>
</table>
D. Smelting

Characteristics of Smelting Tests

<table>
<thead>
<tr>
<th>No. of Test</th>
<th>Time of fusion in min.</th>
<th>Weight in gm.</th>
<th>No. of Calcine Used</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Speiss</td>
<td>Slag</td>
</tr>
<tr>
<td>1</td>
<td>20</td>
<td>21.0</td>
<td>320.1</td>
</tr>
<tr>
<td>2</td>
<td>15</td>
<td>87.2</td>
<td>237.1</td>
</tr>
<tr>
<td>3</td>
<td>15</td>
<td>143.8</td>
<td>144.2</td>
</tr>
<tr>
<td>4</td>
<td>20</td>
<td>123.0</td>
<td>241.3</td>
</tr>
<tr>
<td>5</td>
<td>25</td>
<td>123.0</td>
<td>176.5</td>
</tr>
<tr>
<td>6</td>
<td>20</td>
<td>103.5</td>
<td>172.0</td>
</tr>
<tr>
<td>7</td>
<td>20</td>
<td>121.5</td>
<td>183.0</td>
</tr>
<tr>
<td>8</td>
<td>20</td>
<td>44.0</td>
<td>304.8</td>
</tr>
<tr>
<td>9</td>
<td>20</td>
<td>117.0</td>
<td>--</td>
</tr>
<tr>
<td>10</td>
<td>20</td>
<td>219.5</td>
<td>360.8</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>No. of Test</th>
<th>Charge in grams</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Calcine</td>
</tr>
<tr>
<td>1</td>
<td>300</td>
</tr>
<tr>
<td>2</td>
<td>300</td>
</tr>
<tr>
<td>3</td>
<td>300</td>
</tr>
<tr>
<td>4</td>
<td>600</td>
</tr>
<tr>
<td>5</td>
<td>300</td>
</tr>
<tr>
<td>6</td>
<td>300</td>
</tr>
<tr>
<td>7</td>
<td>300</td>
</tr>
<tr>
<td>8</td>
<td>300</td>
</tr>
<tr>
<td>9</td>
<td>600</td>
</tr>
<tr>
<td>10</td>
<td>600</td>
</tr>
<tr>
<td>No. of Speiss</td>
<td>% Co. Recovery</td>
</tr>
<tr>
<td>--------------</td>
<td>----------------</td>
</tr>
<tr>
<td>1</td>
<td>45.1</td>
</tr>
<tr>
<td>2</td>
<td>24.3</td>
</tr>
<tr>
<td>3</td>
<td>12.5</td>
</tr>
<tr>
<td>4</td>
<td>25.1</td>
</tr>
<tr>
<td>5</td>
<td>20.3</td>
</tr>
<tr>
<td>6</td>
<td>21.2</td>
</tr>
<tr>
<td>7</td>
<td>16.6</td>
</tr>
<tr>
<td>8</td>
<td>35.5</td>
</tr>
<tr>
<td>9</td>
<td>20.4</td>
</tr>
<tr>
<td>10</td>
<td>17.7</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>No. of Slag</th>
<th>% Co. Recovery</th>
<th>% Fe</th>
<th>% Insol.</th>
<th>% As.</th>
<th>% CaO</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.4</td>
<td>55.0</td>
<td>28.3</td>
<td>37.16</td>
<td>1.52</td>
</tr>
<tr>
<td>2</td>
<td>1.89</td>
<td>17.5</td>
<td>22.5</td>
<td>41.46</td>
<td>0.77</td>
</tr>
<tr>
<td>3</td>
<td>1.90</td>
<td>13.5</td>
<td>14.3</td>
<td>40.18</td>
<td>1.67</td>
</tr>
<tr>
<td>4</td>
<td>0.90</td>
<td>4.0</td>
<td>10.3</td>
<td>48.12</td>
<td>0.61</td>
</tr>
<tr>
<td>5</td>
<td>0.30</td>
<td>2.0</td>
<td>11.8</td>
<td>55.8</td>
<td>-</td>
</tr>
<tr>
<td>6</td>
<td>0.61</td>
<td>4.0</td>
<td>9.1</td>
<td>58.8</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>0.70</td>
<td>5.5</td>
<td>20.3</td>
<td>48.5</td>
<td>-</td>
</tr>
<tr>
<td>8</td>
<td>2.36</td>
<td>29.5</td>
<td>26.2</td>
<td>43.0</td>
<td>-</td>
</tr>
<tr>
<td>9</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>10</td>
<td>0.92</td>
<td>7.1</td>
<td>23.7</td>
<td>44.5</td>
<td>-</td>
</tr>
</tbody>
</table>

The dusting loss varies from 2 - 7% as shown by the sum of the cobalt recoveries in the above table. Test No. 5 shows a dusting loss of 1.8% and No. 8 a dusting loss of 6.2%.

Some slag was lost from tests 1, 4, 8 and 9.
IV. CONCLUSIONS

General Conclusion

It is possible to recover separately 90--95 per cent. of both the cobalt and gold in the ore of the Gem Mine, Bridge River, and as marketable products.

The experimental Flow Sheet as outlined in the Appendix consists of a combination of the most suitable tests obtained. These tests are: (1) Primary Calcine No. 10; (2) Sulphating Roast 7G; (3) Water Leaching Sulphated Calcine 7G; (4) Precipitation Test No. 2.

A. Roasting

1. Primary Calcines:

(a) A fast high temperature roast such as test 10 at 800°C. for two hours produced a Primary Calcine low in arsenic.

(b) The low arsenic calcine gives a higher water soluble cobalt content when sulphated.

(c) Efficient rabbling is necessary for a maximum removal of arsenic.

(d) The addition of charcoal gives a slightly greater removal of arsenic.

(e) The removal of sulphur was almost 100 percent. when the calcine was roasted at a high temperature. (See test 10.)

(f) Sulphur came off with the arsenic in all cases and it does not seem possible that a low arsenic high sulphur calcine can be made directly from the ore. A high sulphur content
would increase the efficiency of a sulphating roast.

(g) The calcines showed no sign of sintering at 820°C.

2. Sulphated Calcines

(a) Tests No. 4 and 7G gave the highest recovery of water soluble cobalt.

(b) The presence of arsenic in large amounts prevents ideal sulphating conditions and lowers the amount of water soluble cobalt. (See test No. 1.)

(c) Decreasing the amount of acid used in the sulphating charge lowers the recovery of water soluble cobalt considerably. (See Test No. 6.)

(d) Considerable sulphating action takes place when the sulphated calcine is roasted at 650° C. for one hour. (See Tests (9A and 9B.)

(e) The addition of salt and charcoal as in test 7G to the sulphated calcine and then roasted at 650° C. for one hour and forty-five minutes helps to convert a large portion of the cobalt into water soluble cobalt.

(f) Efficient rabbling is necessary for the oxidation of the iron.

(g) The amount of water soluble cobalt is decreased by heating sulphated calcine above the critical range at which soluble cobalt compounds are decomposed. The critical range lies between 650 and 675° C. Test No. 7F proves this effect.

(h) The addition of either charcoal or salt to the sulphating charge helps to increase the amount of water soluble cobalt.
3. Chloridized and Sulphatized Calcines.

No suitable procedure was determined in either case to recover the cobalt as water soluble cobalt.

B. Leaching.

(a) The feasibility of leaching raw ore with concentrated sulphuric acid is not advisable because of the high consumption of acid. The acid and water soluble cobalt however was almost 100 per cent.

(b) Recovery of gold from the leached residue by cyanidation seems possible; however, no tests were run to confirm this idea.

(c) There are no apparent water soluble gold compounds in the sulphated calcines.

C. Precipitation

(a) The first iron precipitate contains only a small amount of cobalt and it can be rejected.

(b) The final iron precipitate contains considerable cobalt. The cobalt contained in this precipitate can be resulphated to recover the cobalt.

(c) The cobalt mud produced by precipitating the cobalt with sodium carbonate can be ignited at 800°C to produce a marketable cobalt oxide containing 70 - 73% cobalt.
D. Smelting

(a) No test attempted combines the properties of high recovery and high concentration of the cobalt in the form of a speiss.

(b) The highest recovery of cobalt was 96.2% for No.5 speiss with a concentration ratio of 2.4:1 based on the cobalt content contained in the calcine used in the charge.

(c) The addition of silica to the charge caused the slag to foam.

(d) Foaming can be eliminated by changing from a reducing to an oxidizing condition inside the furnace.

(e) Shots of speiss in the acid slag caused a large drop in the cobalt recovery.

(f) Iron reduced from the slag by excessive digesting in a molten state combines with the speiss already formed to produce a very hard speiss (Test No. 5).

(g) Only a small amount of cobalt combines with the slag. (See Test No.5) The slag contained only 0.3 per cent cobalt.
V. DETAILS OF TESTS

A. Preparation of Ore

About 200 pounds of hand-picked ore were put through a small Dodge jaw-crusher set at \( \frac{1}{2} \)". The crushed product was then fed to a pair of rolls and by gradual reduction was sized to -20 mesh. The fines were screened out between each reduction. The ore had the appearance of a massive sulphide and was very friable, producing a large amount of fines when crushed.

A head sample of about 500 gms. was taken with a Jones splitter and ground to -100 mesh.

The head sample was assayed according to assay procedures as outlined in the appendix.

Head Sample

<table>
<thead>
<tr>
<th>Element</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co.</td>
<td>5.46%</td>
</tr>
<tr>
<td>Fe.</td>
<td>22.00%</td>
</tr>
<tr>
<td>As.</td>
<td>36.10%</td>
</tr>
<tr>
<td>S.</td>
<td>11.10%</td>
</tr>
<tr>
<td>CaO.</td>
<td>1.20%</td>
</tr>
<tr>
<td>Insoluble(SiO₂)</td>
<td>13.70%</td>
</tr>
<tr>
<td>Undetermined</td>
<td>10.44%</td>
</tr>
<tr>
<td>Ni.</td>
<td>Nil</td>
</tr>
<tr>
<td>Gold</td>
<td>1.15 oz/Ton</td>
</tr>
<tr>
<td>Silver</td>
<td>0.20 oz/Ton</td>
</tr>
</tbody>
</table>
Bi Roasting

(a) Primary Calcines

No. I. Calcine

A charge consisting of 1000 grams of -20 mesh ore and 3% charcoal (30 grams) was roasted in a fused quartz dish 37 cm. long, 23 cm. wide and 2 cm. deep. The charge was well mixed before it was charged into the large electric furnace at 300° C. The temperature was raised gradually to 800° C. The first fumes of As₂O₃ were observed 20 minutes after starting to roast at 440° C. The arsenious oxide fumes came off very heavy between 530° and 600° C. The last fumes came off at 700° C., reaching this temperature 2 hours and 10 minutes after the start of the roast. The calcine was rabbled continuously while the As₂O₃ was coming off. The calcine was finally heated to 800° C. and minor amounts of As₂O₃ came off. The calcine had no tendency to sinter even at 800° C.

Removed and cooled calcine before adding 2% (20 grams) more charcoal. The charcoal was mixed in with the calcine at a temperature just below where the charcoal would burn. The calcine was recharged to the furnace at 600° C. and rabbled continuously till it reached 780° C. just 15 minutes after recharging. The arsenious oxide fumes came off heavy at 700° C. but little or no fumes at 780° C.

The calcine was removed again and a similar portion of charcoal, 2% or 20 gm., added to the calcine. The charge was put back in the furnace at 600° C. and rabbled continuous-
ly for 15 minutes till it reached 750° C. and no more arsenious oxide fumes came off. The charge was removed and cooled. The calcine weighed 662 grams showing a reduction of 33.8% by weight. A 30 gram portion was crushed to pass through a 100 mesh screen.

**Assays**

<table>
<thead>
<tr>
<th>Total Fe. in Calcine</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Co.</td>
<td>7.70%</td>
</tr>
<tr>
<td>S.</td>
<td>1.26%</td>
</tr>
<tr>
<td>As.</td>
<td>6.20%</td>
</tr>
<tr>
<td>SiO₂</td>
<td>22.00%</td>
</tr>
<tr>
<td>CaO.</td>
<td>1.9%</td>
</tr>
</tbody>
</table>

**No. II Calcine**

A dead roast of 1500 grams of -20 mesh ore was made in the electric furnace in a large fused quartz dish. The initial temperature was 400° C. The first fumes of arsenious oxide came off at 510° C. A flame ½ inch high and having a blue color indicated the sulphur burning off at 650° C. The temperature was gradually raised to 800° C. while the charge was rabbled almost continuously for three hours till the As₂O₃ fumes ceased to come off. While the arsenic was burning off, a one-inch white flame indicated the arsenic being burned to As₂O₃. The charge was removed and cooled. The calcine weighed 945 grams showing a reduction of 37%.

The dead roast was then treated with 3% (45 grams) of charcoal at 500° C. in the electric furnace. The furnace
reached a temperature of 570° C. ten minutes after charging but no arsenious exide fumes came off. The first fumes of As$_2$O$_3$ came off at 600° C. when the charge was rabbled vigorously, and fairly heavy fumes at 620° C. (for 5 minutes). The temperature reached 630° C., 20 minutes after charging at 500° C. and no more fumes came off. The calcine was now heated to 700° C. but with no signs of fumes. The charge was removed and cooled. The calcine weighed 934 grams showing a net loss of eleven grams by the reduction of the charcoal roast. The total reduction in weight was 37.7%. A 30 gram portion of the calcine was crushed to -100 mesh.

Assays

<table>
<thead>
<tr>
<th></th>
<th>Total</th>
<th>Fe.</th>
<th>33.4%</th>
</tr>
</thead>
<tbody>
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<td></td>
<td></td>
<td>Co.</td>
<td>8.04%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>S.</td>
<td>.55%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>As.</td>
<td>4.00%</td>
</tr>
<tr>
<td>Water soluble Co.</td>
<td>0.09%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total CoO.</td>
<td>1.9%</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

No. 3 Calcine

A charge of 1500 gm. of -20 mesh ore was placed in a large fused quartz dish and charged to the electric furnace at 550° C. The As$_2$O$_3$ fumes came off shortly after charging. A temperature of 700° C. was reached in 15 minutes. The arsenic fumes continued to come off for 45 minutes as a heavy white cloud. The arsenic burnt with a one-inch white flame on top of the charge. The charge was rabbled almost con-
tinuously while the As$_2$O$_3$ came off.

The temperature was maintained for one hour and fifteen minutes at 700° C. The temperature was then raised to 800° C. in fifteen minutes. The increase in temperature caused the arsenic to burn off quicker. The temperature was maintained at 800° C. for one hour, till no more As$_2$O$_3$ fumes were formed and a dead roast was made.

The calcine was removed from the furnace and allowed to cool.

**Weight of calcine** 930 gm.

**Reduction in weight by roasting** 38%

**No. 4 Calcine**

A similar charge of 1500 gm of #20 mesh ore was treated as in the preparation of No. 3 Calcine.

**Weight of calcine** 926 gm.

**Reduction in weight by roasting** 38.2%

Calcines 3 and 4 were combined together and a sample taken for assaying.

**Assays for Calcine (3+4)**

<table>
<thead>
<tr>
<th></th>
<th>Total</th>
<th>Co</th>
<th>8.56%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe</td>
<td>35.50%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SiO$_2$</td>
<td>22.20%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>As</td>
<td>5.68%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>0.20%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>CaO</td>
<td>1.90%</td>
<td>Gold 1.80 oz/Ton</td>
</tr>
</tbody>
</table>
**No. 5 Calcine**

A charge consisting of 1500 gm. of -20 mesh ore was charged to the electric furnace at 550° C. The temperature reached 700° C. in 15 minutes. Heavy As$_2$O$_3$ fumes came off as the charge was rabbled almost continuously. The temperature was maintained at 700° C. for 75 minutes. The charge was removed at 700° C., while the fumes of As$_2$O$_3$ were still coming off, and allowed to cool.

Weight of calcine 1135 gm.

Reduction in weight by roasting 24.4%

Assays: No. 5 Calcine

<table>
<thead>
<tr>
<th>Total</th>
<th>Co.</th>
<th>6.88%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe.</td>
<td>29.1%</td>
<td></td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>18.1%</td>
<td></td>
</tr>
<tr>
<td>As.</td>
<td>21.1%</td>
<td></td>
</tr>
<tr>
<td>S.</td>
<td>7.06%</td>
<td></td>
</tr>
<tr>
<td>CaO.</td>
<td>1.6%</td>
<td></td>
</tr>
</tbody>
</table>

Gold 1.47 oz/Ton.

**No. 6, 7 and 8 Calcines**

In each case 1500 gm. of -20 mesh ore was charged to the electric furnace at 550° C. in a large fused quartz dish. The temperature was raised to 700° C. in 15 minutes and maintained at that temperature for 15 minutes. The charge was rabbled almost continuously while heavy As$_2$O$_3$ fumes came off. The temperature was raised to 800° C. in
15 minutes and held at 800° C. for 45 minutes. The charge was removed while dense fumes of As$_2$O$_3$ were still coming off, and allowed to cool.

By increasing the temperature to 800° C. early in the roast the As$_2$O$_3$ seemed to come off heavier and required less rabbling for the same volume of arsenic fumes. A long white flame covered the roast while the arsenic was burning to As$_2$O$_3$.

<table>
<thead>
<tr>
<th>Weight of Calcine No. 6</th>
<th>1,028 gm.</th>
</tr>
</thead>
<tbody>
<tr>
<td>No. 7</td>
<td>1,034 gm.</td>
</tr>
<tr>
<td>No. 8</td>
<td>1,028 gm.</td>
</tr>
</tbody>
</table>

Reduction in weight by roasting

<table>
<thead>
<tr>
<th>No. 6</th>
<th>31.5%</th>
</tr>
</thead>
<tbody>
<tr>
<td>No. 7</td>
<td>31.0%</td>
</tr>
<tr>
<td>No. 8</td>
<td>31.5%</td>
</tr>
</tbody>
</table>

Calcines 6, 7 and 8 were combined together and a sample taken for assaying.

Assays on Calcine (6+7+8)

<table>
<thead>
<tr>
<th>Total</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Co</td>
<td>7.76%</td>
</tr>
<tr>
<td>Fe</td>
<td>32.3%</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>20.1%</td>
</tr>
<tr>
<td>As</td>
<td>14.1%</td>
</tr>
<tr>
<td>S</td>
<td>4.28%</td>
</tr>
<tr>
<td>CaO</td>
<td>1.7%</td>
</tr>
</tbody>
</table>

Gold 1.62 oz/Ton
No. 9 Calcine

A charge of 3000 gm. of -20 mesh ore was placed on the muffle floor of the electric furnace at 800° C. The temperature dropped to 700° C. in five minutes, but rose back to 800° C. in 35 minutes after charging.

Heavy arsenic fumes came off as soon as the ore was charged and when the charge reached a dull red heat white flames four inches high could be seen burning on top of the charge. The flame was dead in 45 minutes except when the charge was rabbled. A dead roast was obtained in one hour and twenty minutes and further rabbling did not produce any more arsenious oxide fumes. No SO₂ fumes could be detected after all the arsenious oxide fumes were off.

The furnace was shut off when the dead roast stage was reached and the calcine was allowed to cool in the furnace.

Weight of calcine No. 9. 1823 gm.
Reduction in weight by roasting 39.3%

The calcine was then pulverized to pass through a -100 mesh screen. A 80 gm. sample was taken for assaying.

Assays for No. 9 Calcine.

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Fe.</td>
<td>36.00%</td>
</tr>
<tr>
<td>Co.</td>
<td>8.61%</td>
</tr>
<tr>
<td>SiO₂</td>
<td>22.50%</td>
</tr>
<tr>
<td>As</td>
<td>4.82%</td>
</tr>
<tr>
<td>S</td>
<td>0.05%</td>
</tr>
<tr>
<td>CaO</td>
<td>2.00%</td>
</tr>
<tr>
<td>Gold</td>
<td>1.82 oz/Ton</td>
</tr>
</tbody>
</table>

Gold 1.82 oz/Ton
Reducing Roast

A charge consisting of:

300 gm. No. 9 Calcine (-100)

3% charcoal or 9 gm.

was mixed on a rubber mat and then placed in a fused quartz dish. The charge was then placed in the electric furnace at 500° C. and the temperature of the furnace was raised to 775°C in one hour. No fumes of arsenic were seen to come off during any stage of the reducing roast.

The charge was removed and allowed to cool. The charge was weighed and it showed only a loss of two grams. The removal of arsenic by the reducing roast was very small since no fumes of white As₂O₃ were produced between the temperatures of 500° C. and 775°C. with fairly constant rabbling.

No. 10 Calcine

A charge of 5000 gm. of -20 mesh ore was placed on the muffle floor of the electric furnace at 8200°C. The temperature dropped to 710°C in five minutes after charging but the temperature was back to 820°C in 30 minutes after charging.

Heavy arsenic fumes came off as soon as the ore was charged. A blue white flame two inches high was burning over the charge at 710°C just 10 minutes after charging. A three inch white flame was burning over the charge in 20 minutes after charging to the furnace and at 820°C.

The flame was dead in one hour except when the
charge was rabbled.

The charge was rabbled almost continuously from this point till a dead roast was produced and no more As₂O₃ came off. All the arsenic was off in one hour and fifty-five minutes after charging. During the stage when the last traces of arsenic were coming off a black coating formed on top of the charge if it was not rabbled continuously. When the "Dead Roast" stage was reached the hot calcine took on a dark color and the furnace was shut off.

The calcine was left in the furnace to cool. No sulphur fumes could be smelt coming off the cooling charge.

Weight of Calcine No. 10 2977 gm.

Reduction in weight by roasting 40.5%

The capacity per square foot of hearth area was found to be eleven pounds per square foot for the two hour roast. The depth of charge varied from one and one-half inches to one inch.

The calcine was ground to -100 mesh in the pulverizer and a sample taken for assaying with the Jones splitter.

Assays on No. 10 Calcine

<table>
<thead>
<tr>
<th>Component</th>
<th>Assay</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total As.</td>
<td>3.46%</td>
</tr>
<tr>
<td>&quot; Co.</td>
<td>8.90%</td>
</tr>
<tr>
<td>&quot; Fe.</td>
<td>36.60%</td>
</tr>
<tr>
<td>&quot; Insol.</td>
<td>22.80%</td>
</tr>
<tr>
<td>&quot; CaO</td>
<td>2.00%</td>
</tr>
<tr>
<td>&quot; S</td>
<td>0.10%</td>
</tr>
<tr>
<td>Gold</td>
<td>1.87oz/Ton</td>
</tr>
</tbody>
</table>
(b) Sulphated Calcines

No. 1 Sulphating Roast

One hundred grams of sulphatized calcine was mixed with 2 gm. NaCl and 2 gm. Charcoal. This product was then mixed with .50 c.c. of conc. H₂SO₄ to form a paste and then charged to the electric furnace at 650°C. The furnace door was kept closed for 2 minutes so as to get a strong sulphating atmosphere. Strong fumes of SO₃ came off for 5 min. and then stopped. The charge was removed from the furnace and the crust on top of the calcine showing signs of cobalt was recharged and heated at 650°C for 18 min. The charge was removed and cooled somewhat. The color of the calcine had changed from a yellow brown to a dark brown calcine. The calcine was fine and powdery and had no tendency to sinter or cake. The charge was rabbled continuously to speed up the oxidation.

The charge was roasted for an additional 50 min. at 650°C, with frequent rabbling. The calcine was removed and was found to be fairly red in color after cooling. The calcine weighed 105.5 gm.

Assay

<table>
<thead>
<tr>
<th></th>
<th>Co</th>
<th>1.94%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble</td>
<td>Fe</td>
<td>0.25%</td>
</tr>
<tr>
<td></td>
<td>Total Co</td>
<td>7.4%</td>
</tr>
<tr>
<td>Recovery of Co= $\frac{1.94 \times 100}{7.4}$ =</td>
<td>26.2%</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>Fe</td>
<td>28.6%</td>
</tr>
<tr>
<td></td>
<td>SiO₂</td>
<td>17.8%</td>
</tr>
</tbody>
</table>
Total CaO 1.5%
As 14.5%

No. 2 Sulphating Roast at 650°C.

A charge consisting of
100 gm. #2 calcine of -20 mesh
2 gm. Charcoal
5 gm. NaCl

was mixed on a rubber mat and then placed in a small fused quartz dish. The charge was then mixed with 50 c.c. of conc. H₂SO₄. Strong HCl fumes came off as the H₂SO₄ reacted with the NaCl. This mixture was then put in the electric furnace at 650°C. Strong SO₃ fumes came off for 6 minutes. The calcine was broken up and rabbled every few minutes for 90 minutes. The calcine weighed 119.0 gm. A 30 gm. portion was reduced to -100 mesh for assaying.

Three different coloured particles were noticed in the calcine. The first were white resembling quartz in the ore, second a bright red indicating sulphated iron converted to ferric iron, and thirdly dark brown red particles likely indicating the sulphated cobalt mineral dainite.

The roasted calcine had no tendency to sinter but formed a soft cake after the SO₃ was driven off. This cake was easily broken up by a scraper used as a rabbler.

### Assays

<table>
<thead>
<tr>
<th>Assay</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water Soluble</td>
<td></td>
</tr>
<tr>
<td>Co</td>
<td>5.18%</td>
</tr>
<tr>
<td>Fe</td>
<td>0.30%</td>
</tr>
<tr>
<td>As</td>
<td>Nil</td>
</tr>
<tr>
<td>Total</td>
<td></td>
</tr>
<tr>
<td>Co</td>
<td>6.75%</td>
</tr>
<tr>
<td>Recovery of</td>
<td></td>
</tr>
<tr>
<td>Co</td>
<td>5.18 / 6.75 = 77.0%</td>
</tr>
<tr>
<td>Fe</td>
<td>29.8%</td>
</tr>
<tr>
<td>SiO₂</td>
<td>18.5%</td>
</tr>
</tbody>
</table>

#### No. 3 Sulphating Roast at 650°C.

A similar charge to that of No. 2 Sulphating Roast was made at 650°C, except that the #2 calcine was ground to -100 mesh. The charge was treated in the electric furnace for 90 minutes at 650°C. Strong SO₃ fumes came off for the first 5 minutes. The calcine turned into a porous cake after the SO₃ was driven off. This porous cake was broken up by continuous rabbling and the increase in temperature of the sulphated calcine.

The calcine weighed 127.5 grams, and showed an increase of 27.5% over the charged calcine. The calcine looked bright brick red on cooling resembling the 650°C Calcine produced by heating the residue produced by leaching ore in hot conc. H₂SO₄.

### Assays

<table>
<thead>
<tr>
<th>Assay</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble</td>
<td></td>
</tr>
<tr>
<td>Co</td>
<td>5.73%</td>
</tr>
<tr>
<td>Fe</td>
<td>0.25%</td>
</tr>
</tbody>
</table>
A charge consisting of

300 gm. No. 9 Calcine
15 gm. Salt
6 gm. Charcoal
150 c.c. H₂SO₄

was prepared as follows:

The calcine (-100 mesh), salt and charcoal were mixed on a rubber mat and then spread out in a large fused quartz dish. The sulphuric acid was then poured on top of the charge and mixed in with a spatula into a smooth creamy product. When the H₂SO₄ was added strong HCl fumes came off due to the violent reaction of the salt and H₂SO₄.

The sulphated product was charged to the electric furnace at 350°C. and kept at that temperature for 30 minutes while heavy SO₃ fumes came off for 20 minutes. The temperature was then set at 710°C.

On trying to rabble the charge it was found to be very hard. The charge was removed and the sulphated product, which was yellow green in colour, broken up and recharged to the furnace at 710°C. in a fused quartz dish. The temperature was maintained at 710°C. for 45 minutes while
fairly heavy $\text{As}_2\text{O}_3$ fumes came off. The temperature was then dropped to 650° C. and the $\text{As}_2\text{O}_3$ fumes stopped to a great extent. The temperature was maintained at 650° C. for 30 minutes.

The charge was removed and allowed to cool. The sulphated calcine was red in color similar to No. 3.

The calcine weighed 396 grams showing an increase in weight of 32%.

**Assays**

<table>
<thead>
<tr>
<th>Component</th>
<th>Assay (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Co.</td>
<td>6.39%</td>
</tr>
<tr>
<td>Water soluble Co.</td>
<td>6.08%</td>
</tr>
<tr>
<td>Recovery</td>
<td>6.08 x 100 = 95.0%</td>
</tr>
<tr>
<td>Total Fe</td>
<td>26.6%</td>
</tr>
<tr>
<td>Water Soluble Fe</td>
<td>0.20%</td>
</tr>
<tr>
<td>Total As</td>
<td>2.12%</td>
</tr>
<tr>
<td>Insol.</td>
<td>17.1%</td>
</tr>
<tr>
<td>Water soluble As</td>
<td>0.42%</td>
</tr>
<tr>
<td>Gold</td>
<td>1.38 oz/Ton</td>
</tr>
</tbody>
</table>

**Recovery of Gold** = $\frac{1.38 \times 132 \times 100}{1.82 \times 100} = 100%$

**No. 5 Sulphating Roast**

A charge consisting of

100 gm. No. 9 Calcine

2 gm. Charcoal

was mixed on a rubber mat and then placed in a small fused quartz dish. The charge was then mixed with 50 c.c. of conc. $\text{H}_2\text{SO}_4$. Strong $\text{SO}_3$ fumes were made as the $\text{H}_2\text{SO}_4$ reacted with
the cold calcine and charcoal.

The charge was then heated in the electric furnace at 450° C. to drive off the excess \( \text{H}_2\text{SO}_4 \). Fairly heavy \( \text{SO}_3 \) fumes came off for 20 minutes. The calcine was removed and broken up and recharged to the furnace at 450° C. The temperature was raised to 650° C. in 25 minutes and maintained at 650° C. for 55 minutes. No more \( \text{SO}_3 \) could be seen coming off after recharging or when heated up to 650° C. The charge was removed and cooled and the final weight of calcine was 134 gm., showing an increase in weight of 34%.

**Assays**

<table>
<thead>
<tr>
<th></th>
<th>Co</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Total</td>
<td>6.25%</td>
<td></td>
</tr>
<tr>
<td>Water sol.</td>
<td>5.84%</td>
<td></td>
</tr>
</tbody>
</table>

\[
\text{Recovery} = \frac{5.84}{6.25} \times 100 = 93.5\%
\]

<table>
<thead>
<tr>
<th></th>
<th>Fe</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Total</td>
<td>25.8%</td>
<td></td>
</tr>
<tr>
<td>Water sol.</td>
<td>1.20%</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>As</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Total</td>
<td>1.51%</td>
<td></td>
</tr>
<tr>
<td>Insol.</td>
<td>17.0%</td>
<td></td>
</tr>
</tbody>
</table>

Gold | 1.36 oz/Ton

\[
\text{Recovery of Gold} = \frac{1.36}{1.82} \times \frac{134}{100} = 100\%
\]
No. 6 Sulphating Roast

A charge consisting of

100 gm. No. 9 Calcine (-100)
5 gm. Salt
2 gm. Charcoal
35 c.c. conc. H₂SO₄

was prepared as follows.

The calcine (-100 mesh), salt and charcoal were mixed on a rubber mat and then spread out in a small fused quartz dish. The sulphuric acid was then poured on top of the charge and mixed in with a spatula into a smooth product. When the conc. H₂SO₄ was added strong HCl fumes came off due to the violent reaction of the salt and the H₂SO₄. There was enough acid to make a fairly pasty product but not nearly so soft as test No. 4.

The charge was placed in the electric furnace at 450° C. The amount of fumes produced on heating was small compared to test No. 4.

The sulphated product was removed in twenty minutes. The charge was porous and pink in color. Small white specks were left in the vesicular openings in the calcine which were probably sodium sulphide or sulphate.

The calcine was broken up and recharged to the furnace at 650° C. The calcine was not so hard as No. 4 and was quite easily broken up.

No more fumes of SO₃ came off while the charge was roasted and rabbled for seventy minutes at 650° C.
The charge was removed and allowed to cool. The sulphated calcine was red in color similar to tests No. 3 and 4.

The calcine weighed 126.2 gm. and thus showing an increase in weight of 26.2%.

**Assays on Sulphated Calcine at 650° C.**

<table>
<thead>
<tr>
<th></th>
<th>Total</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Co.</td>
<td>6.88%</td>
<td></td>
</tr>
<tr>
<td>Water sol. Co.</td>
<td>5.74%</td>
<td></td>
</tr>
</tbody>
</table>

Recovery of Co. = \( \frac{5.74}{6.88} \times 100 = 83.5\% \)

<table>
<thead>
<tr>
<th></th>
<th>Total</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>27.5%</td>
<td></td>
</tr>
<tr>
<td>Water Sol. Fe</td>
<td>0.05%</td>
<td></td>
</tr>
<tr>
<td>Insol.</td>
<td>17.8%</td>
<td></td>
</tr>
<tr>
<td>Total As.</td>
<td>2.86%</td>
<td></td>
</tr>
<tr>
<td>Gold</td>
<td>1.45 oz/Ton.</td>
<td></td>
</tr>
</tbody>
</table>

**No. 7 Sulphating Roast**

A charge consisting of:

500 gm. No. 10 Calcine (-100)

250 c.c. conc. \( \text{H}_2\text{SO}_4 \)

was prepared as follows.

The acid and calcine were mixed together in a steel pan with a spatula, till the product formed a smooth brown pastel. The charge was placed in the electric furnace at 450°.

Strong \( \text{SO}_3 \) fumes came off as soon as the charge became warm or shortly after it was charged to the furnace. The \( \text{SO}_3 \) fumes died down in 10 minutes.
The charge expanded rapidly as the H\textsubscript{2}SO\textsubscript{4} was driven off. The charge had a green color while the acid was being driven off.

The fumes were dead in 20 minutes after charging and the temperature was maintained for another 10 minutes at 450\textdegree\,C. before the charge was removed and pulverized. The sulphated product changed from a yellow green to a dirty pink on cooling.

The calcine weighed 829 gm.

Four other charges were run as the charge above for 30 minutes at 450\textdegree\,C.

Weight of Calcines

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>7(a)</td>
<td>829.0 Gm.</td>
</tr>
<tr>
<td>7(b)</td>
<td>829.0 gm.</td>
</tr>
<tr>
<td>7(c)</td>
<td>829.5 gm.</td>
</tr>
<tr>
<td>7(d)</td>
<td>831.0 gm.</td>
</tr>
<tr>
<td>7(e)</td>
<td>830.5 gm.</td>
</tr>
<tr>
<td>Total</td>
<td>4149.0 gm.</td>
</tr>
</tbody>
</table>

The 4149 grams of calcine were ground to -100 mesh and a sample taken for assaying.

No. 7F Sulphating Roast

A charge consisting of 2000 grams of the above calcine was charged to the muffle floor of the electric furnace at 450\textdegree\,C. Strong fumes of SO\textsubscript{3} started to come off shortly after charging. The charge was rabbled every few minutes to help the removal of the SO\textsubscript{3} and the oxidation of the iron.
The temperature was raised to 650° C. in one hour after charging and maintained at 650° C. for one hour and a half.

Only slight fumes of SO\textsubscript{3} came off near the end of the roast.

The calcine was removed and cooled. Weight of final calcine 7F 1273.5 grams. The increase in weight was 5.7%.

The calcine was screened through a 100 mesh screen before assaying.

**Assays Calcine 7a+7b+7c+7d+7e at 450°C.**

<table>
<thead>
<tr>
<th>Component</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble Co.</td>
<td>4.04%</td>
</tr>
<tr>
<td>Total Co.</td>
<td>5.44</td>
</tr>
<tr>
<td>Recovery</td>
<td>$\frac{4.04}{5.44} \times 100 = 74.5%$</td>
</tr>
<tr>
<td>Water soluble Fe</td>
<td>2.8%</td>
</tr>
<tr>
<td>Total Fe</td>
<td>21.8%</td>
</tr>
<tr>
<td>Insoluble</td>
<td>13.6%</td>
</tr>
</tbody>
</table>

**Assays Calcine 7F (650° - 750° C.).**

<table>
<thead>
<tr>
<th>Component</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble Co.</td>
<td>2.20%</td>
</tr>
<tr>
<td>Total Co.</td>
<td>8.06%</td>
</tr>
<tr>
<td>Recovery</td>
<td>$\frac{2.20}{8.06} \times 100 = 27.3%$</td>
</tr>
<tr>
<td>Water soluble Fe</td>
<td>0.05%</td>
</tr>
<tr>
<td>Total Fe</td>
<td>32.2%</td>
</tr>
<tr>
<td>Insoluble</td>
<td>20.2%</td>
</tr>
</tbody>
</table>

**Note:**

The low recovery of cobalt was due to over heating
on the muffle floor above 650° C. The pyrometer showed 650° C. but the actual temperature must have been about 750° C.

**No. 7G Sulphating Roast**

A charge consisting of

- 985 grams of Calcine 7(a·b·c·d·e)
- 30 grams salt
- 30 grams charcoal

was mixed on a rubber mat and placed in a large fused quartz tray. The charge was placed in the electric furnace at 650° C. The temperature dropped to 575° C. in 5 minutes and was back to 650° C. in 15 minutes. The SO₃ fumes came off shortly after charging and continued quite strong for 20 minutes.

The charge was rabbled every few minutes to aid the oxidation of the iron.

The charge was removed in one hour after charging with still faint fumes of SO₃ coming off.

The charge was cooled and weighed.

**Weight of Calcine 7G, 827.0 gm.**

\[
\% \text{ Increase in weight over original Calcine No. 10} = \frac{827 - 595}{595} \times 100 = 39\%
\]

Calcine 7G was recharged to the electric furnace in a fused quartz tray at 650° C. The charge was roasted for 45 minutes at 650° C. and was rabbled every few minutes.

The charge was removed and cooled.

Only slight traces of SO₃ fumes came off during this final 45 minute roast.
Weight of Calcine $7G_2$ 795.0 gm.

% Increase in weight over original Calcine No. 10
$$= \frac{795 - 595}{595} \times 100 = 35.7\%$$

Calcine $7G_2$ was screened through an 80 mesh screen and a sample taken for assaying.

**Assays on Calcine $7G_2$**

- Total Co 6.65%
- Water soluble Co. 6.30%
- Recovery of Co $= \frac{6.30}{6.65} \times 100 = 95.0\%$

- Total Fe 27.3%
- Water soluble Fe 0.62%
- Total Insol. 17.0%
- As. 2.56%

**No. 8 Sulphating Roast**

A charge consisting of

- 300 gm. No. 9 Calcine (-100)
- 150 c.c. $H_2SO_4$ (conc.)
- 6 gm. charcoal

was prepared as follows.

The calcine and charcoal were mixed together on a rubber mat. The calcine and charcoal were then mixed with 150 c.c. of conc. $H_2SO_4$ in a steel pan.

The charge was placed inside the electric furnace at 450° C. Strong fumes of $SO_3$ came off during the first ten minutes.
The charge was removed after a 30-minute treatment at 450° C. The sulphated calcine was fairly hard and pink in color.

The calcine was broken up and weighed.

Weight of Calcine = 460.5 gm.

A portion of the above calcine (425 gm.) was recharged to the electric furnace at 550° C. in a large fused quartz tray. The charge was rabbled and the temperature was raised to 675° C. in 30 minutes. The temperature was maintained at 675° C. for one hour and the charge was then removed.

Only slight traces of SO₃ were noticed coming off the calcine when it was removed from the furnace.

The calcine was red in color and weighed 338 grams. The sulphated calcine showed an increase in weight of 22% over the No. 9 calcine used.

**Assays of Sulphated Calcine 8A at 450° C.**

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble Co</td>
<td>4.14%</td>
</tr>
<tr>
<td>Total Co</td>
<td>5.8%</td>
</tr>
<tr>
<td>Recovery of Co</td>
<td>( \frac{4.14 \times 100}{5.80} = 74.2% )</td>
</tr>
<tr>
<td>Total Fe</td>
<td>22.5%</td>
</tr>
<tr>
<td>Insoluble</td>
<td>14.0%</td>
</tr>
<tr>
<td>Water soluble Fe</td>
<td>2.0%</td>
</tr>
</tbody>
</table>

**Assays of No 8B Sulphated Calcine at 675° C.**

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water Soluble Co</td>
<td>8.10%</td>
</tr>
<tr>
<td>Total Co</td>
<td>7.05%</td>
</tr>
</tbody>
</table>
Recovery of Co. = $\frac{6.10 \times 100}{7.05} = 78.0\%$

Total Fe 28.8\%
Insoluble 17.9\%
Water soluble Fe 0.5\%

**No. 9 Sulphating Roast**

A charge consisting of

300 gm. No. 9 calcine (-100)
150 c.c. H$_2$SO$_4$

was prepared as follows:

The acid and calcine were mixed together in a steel pan with a spatula till the product formed a smooth brown paste.

The charge was placed in the electric furnace at 450°C. Strong SO$_3$ fumes came off as soon as the charge became warm. The fumes decreased rapidly after 10 minutes of roasting.

The charge was removed after being roasted for 30 minutes at 450°C.

**Weight of Calcine 9A** 460.0 gm.

The calcine was broken up and pulverized. A 400 gram portion was charged, in a large fused quartz tray, to the electric furnace at 675°C. The charge was rabbled every few minutes.

The temperature was lowered to 650°C. after heating at 675°C for 25 minutes. The temperature was maintained
at 650° C. for 35 minutes before the charge was removed.

The Calcine 9B weighed 343.0 grams.

The sulphated calcine showed an increase in weight of 31.5% over the No. 9 calcine used. The calcine, which was red in color, was pulverized and screened through a -100 mesh screen.

Assays of Sulphated Calcine at 450° C.

Water Soluble Co. 4.00%
Total Co. 5.60%
Recovery of Co. = \(\frac{4.00 \times 100}{5.60} = 71.5\%\)
Total Fe 22.4%
Insoluble 13.9%
Water Soluble Fe 2.0%

Assays of Sulphated Calcine at 650° C.

Water soluble Co 5.24%
Total Co 6.55%
Recovery of Co = \(\frac{5.24 \times 100}{6.55} = 80.0\%\)
Total Fe 26.5%
Insoluble 16.5%
Water soluble Fe 0.9%
(c) Chloridized Calcines

No. 1 Chloridizing Roast

The amount of raw ore, containing 11.1% S., required to mix with 100 gm. of No.2 Calcine, containing 1.0% S, to make a product having a total sulphur content of 4.5%, was calculated.

\[
\begin{align*}
100 \text{ gm. No.2 Calcine of 1.0\% S} & = 1 \text{ gm. S.} \\
x \text{ gm. ore of 11.1\% S} & = 0.111x \text{ gm S} \\
\text{To make a 4.5\% Total S content} \\
& (100 x)(.045) = 1 + .11x \\
\text{The weight of ore required} & = x = 53 \text{ gm.} \\
\text{Total sulphur content in 153 gm. of 4.5\% S} & = 6.88 \text{ gm. S} \\
\text{If 92\% of the sulphur or 6.55 gm. S. reacts with salt, then the amount of salt required according to the equation,} \\
2 \text{NaCl} + S + 2 O_2 & = \text{Na}_2\text{SO}_4 + \text{Cl}_2 \\
\text{can be calculated.} \\
\text{The weight of salt required to react with 6.35 gm. S is} \\
& (6.35 x 116.92) = 23.2 \text{ gm. NaCl} \\
23.2 \text{ gm. NaCl contain } & 23.2 x 35.46 = 14.1 \text{ gm. Cl.} \\
\text{Cobalt contained in} \\
100 \text{ gm. Calcine of 8.04\% Co.} & = 8.04 \text{ gm. Co.} \\
53 \text{ gm. Ore of 5.46\% Co.} & = 2.89 \text{ gm. Co.} \\
\text{Total Co} & = 10.93 \text{ gm. Co.} \\
58.97 \text{ gm. Co. combine with } & 70.92 \text{ gm. Cl.} \\
& 10.93 \text{ gm. Co. combine with (10.93 x 70.92)} = 13.15 \text{ gm. Cl.} \\
\therefore \text{there is enough Cl. to combine with all the cobalt.} \\
\end{align*}
\]

A charge consisting of 189.5 gm. No. 2 Primary Calcine and 100.5 gm. raw ore (-20 Mesh) were mixed on a rubber mat. Forty grams of the above product was ground to -100 mesh for assaying. The remainder of the charge was mixed with 37.9 gm. of salt and then placed in a fused quartz tray and charged to the electric furnace at 200° C. The temperature

reached 500° C. in 45 minutes and slight traces of As$_2$O$_3$ started to come off. Ten minutes later at 525° C. heavy fumes of As$_2$O$_3$ came off. The removal of the arsenic seemed complete at 565° C. just 20 minutes after the first fumes of arsenic came off. The temperature of the roast was raised to 600° C. in one hour and fifteen minutes, and maintained at this temperature for 30 minutes. Further treatment of 30 minutes at 625° C. and one hour and 30 minutes at 650° C. at a dull red heat completed the roasting.

The charge was rabbled every 5 minutes during the five hour roasting period.

The charge was removed and cooled.

Weight of Calcine 282.5 gm.

Loss in weight 1.9%

Assays on No. 1 Chloridized Calcine

<table>
<thead>
<tr>
<th>Substance</th>
<th>Assay</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble Co.</td>
<td>0.10%</td>
</tr>
<tr>
<td>Fe.</td>
<td>Nil</td>
</tr>
<tr>
<td>Total Co.</td>
<td>7.20</td>
</tr>
</tbody>
</table>

Recovery of Co. = \( \frac{0.10 \times 100}{7.20} = 1.4\% \)

No. 2 Chloridizing Roast

A charge consisting of

100 gm. No. 9 Calcine
15 c.c. conc. HCl
15 c.c. water

was prepared as follows.

The 100 gm. of calcine and 30 c.c. of 1:1 HCl were
mixed into a paste with a spatula in a small fused quartz tray. The charge was baked for 40 minutes on top of a hot plate, at about 350°C. Traces of CoCl₂ were seen on heating. Fumes of HCl came off at first and only a faint trace of Cl₂ could be recognized after 30 minutes of heating.

The chloridized calcine weighed 105 gm.

The calcine was pulverized and screened through a -100 mesh screen.

**Assays on Chloridized calcine**

<table>
<thead>
<tr>
<th>Component</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble Co.</td>
<td>.70%</td>
</tr>
<tr>
<td>Total Co.</td>
<td>8.20%</td>
</tr>
<tr>
<td>Recovery of Co. = ( \frac{.70}{8.20} \times 100 )</td>
<td>8.7%</td>
</tr>
<tr>
<td>Total Fe</td>
<td>34.3%</td>
</tr>
<tr>
<td>Insoluble</td>
<td>21.4%</td>
</tr>
<tr>
<td>Water Soluble Fe</td>
<td>0.20%</td>
</tr>
</tbody>
</table>

**No. 3 Chloridizing Roast**

A charge consisting of

100 gm. No. 9 Calcine

30 c.c. conc. HCl

was prepared as follows:

The calcine and acid were mixed into a paste with a spatula. The charge was heated in a small fused quartz tray on top of a hot plate for one hour at 350°C.

The chloridized calcine weighed 105 gm.

The calcine was pulverized and screened through a -100 mesh screen.
Assays on Chloridized calcine

Water soluble Co. 1.38%
Total Co. 8.20%
Recovery of Co. = \(\frac{1.38}{8.20} \times 100 = 17.0\%\)
Total Fe 34.3%
Insoluble 21.4%
Water Soluble Fe 0.20%

No. 4 Chloridizing Roast

A charge consisting of
100 gm. Na\((6 + 7 + 8)\) Calcine
3 gm. Charcoal
3 gm. Salt
30 c.c. conc. HCl.

was prepared as follows:

The calcine, salt and charcoal were mixed on a rubber mat and spread out in a small fused quartz tray. The conc. HCl was added and mixed into a paste with the calcine. The charge was baked for 90 minutes on a hot plate at 350° C.

The chloridized calcine weighed 111.5 gm.

The calcine was pulverized and screened through a -100 mesh screen.

Assays on Chloridized calcine

Water soluble Co. 1.60%
Total Co. 6.95%
Recovery of Co. = \(\frac{1.60}{6.95} \times 100 = 23.1\%\)
Total Fe 28.9%
Insoluble 18.0%
Water soluble Fe 1.8%

(d) Sulphatized Calcine

No. 1 Sulphatizing Roast

One thousand grams of (-100 mesh) ore were roasted in a confined oxidizing condition, so that any $\text{SO}_2$ gases produced might react with the cobalt present to produce cobalt sulphate by further oxidation with air. The ore, 1000 gm., was placed in a large fused quartz dish and another dish of similar size with the end broken out wired on top. The open end was to serve as a vent, by which air could enter and the gases produced by roasting escape.

The ore was charged to the electric furnace at 250° C. and heated to 400° C. in 15 min. The temperature was maintained at 400° C. for 15 minutes. The temperature was then raised, reaching 460° C. in 10 minutes, and the first fumes of $\text{As}_2\text{O}_3$ came off. The temperature was then raised to 500° C. and maintained at 500° C. for 15 minutes, while light fumes of $\text{As}_2\text{O}_3$ came off. The temperature was then raised to 550° C. for 15 min. and heavy fumes of $\text{As}_2\text{O}_3$ came off, then for 30 min. at 575° C. while more $\text{As}_2\text{O}_3$ came off. The temperature was raised to 600° C. for 20 minutes with $\text{As}_2\text{O}_3$ fumes turning lighter. The temperature was then raised to 650° C. in 20 min. and maintained at 650° C. for 15 minutes.
The charge was removed and cooled. The calcine weighed 729 gm. This showed a reduction of 27.1% by weight. A 30 gram sample of the calcine was taken for assaying.

**Assay**

Water soluble Co 0.07%.

The calcine, 699 gm., was recharged to the electric furnace in an open dish at 600° C. The temperature was raised to 770° C. in 45 minutes. No As$_2$O$_3$ fumes came off while heating to 770° C.

The calcine weighed 696.5 gm. Decrease in weight 27.2%

In both operations the charge was rabbled every few minutes to aid the removal of As$_2$O$_3$. The charge had no tendency to sinter or cake at any temperature.

**Assays 1B.**

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Co</td>
<td>7.5%</td>
<td></td>
</tr>
<tr>
<td>Total Fe</td>
<td>30.2%</td>
<td></td>
</tr>
<tr>
<td>Insoluble</td>
<td>18.8%</td>
<td></td>
</tr>
<tr>
<td>Water sol. Co</td>
<td>trace</td>
<td></td>
</tr>
<tr>
<td>Water col. Fe</td>
<td>nil</td>
<td></td>
</tr>
</tbody>
</table>

**Assays 1A**

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Water sol. Co</td>
<td>0.07%</td>
<td></td>
</tr>
<tr>
<td>Total Co</td>
<td>7.50%</td>
<td></td>
</tr>
<tr>
<td>Total Fe</td>
<td>30.2%</td>
<td></td>
</tr>
<tr>
<td>Insol.</td>
<td>18.8%</td>
<td></td>
</tr>
<tr>
<td>Water sol. Fe</td>
<td>nil</td>
<td></td>
</tr>
</tbody>
</table>
C. Leaching

(a) Sulphuric Acid Leaching

(1) Ore

One hundred and eleven grams of ore (-100 mesh) were added to a 1000 c.c. beaker containing 500 c.c. of conc. \( \text{H}_2\text{SO}_4 \) in 40 minutes. The ore was added slowly at first to the hot acid at \( 306^\circ \text{C} \). The temperature fell to \( 295^\circ \text{C} \) in 10 minutes and no more ore was added for two minutes allowing the solution to heat up to \( 305^\circ \text{C} \) with a cover-glass on top of the beaker to prevent fuming. A small electric heater provided the heat and the temperature was indicated with a mercury thermometer reading up to \( 500^\circ \text{C} \). About 25 grams of ore were added in the first 10 minutes. Added about 26 grams more ore in 4 minutes with the temperature falling from \( 305^\circ \text{C} \) to \( 295^\circ \text{C} \). A faint pink color of cobalt sulphate could be seen as the ore was leached in the acid. Heated solution back to \( 305^\circ \text{C} \) with cover glass on and then added 20 gm. of ore in four minutes. Repeated with two more portions of ore, as above, bringing temperature up to \( 306^\circ \text{C} \) between each addition and not allowing the temperature to drop below \( 295^\circ \text{C} \). The pulp density got so high that the speed of the reaction slowed down. The loss of acid was considerable, being 300 c.c. due to fuming. As the pulp settled in the acid and the acid became clear near the top, a faint pink color of \( \text{CoSO}_4 \cdot x\text{H}_2\text{O} \) could be seen in the acid. The solution was cooled to \( 230^\circ \text{C} \) and filtered through a large suction filter. The residue which was pale pink in
color was washed twice with two portions of H$_2$SO$_4$ of 30 c.c. each. The filtrate was red in color, with As$_2$O$_3$ coming down quite rapidly as the filtrate cooled. The volume of filtrate was 210 c.c.

Transferred the residue to a fused quartz dish 23 cm. long, 15 cm. wide and 1 cm. deep. A small amount of CoSO$_4$ was seen in the bottom of the filter next to the asbestos. Put residue in furnace to dry at 300° C. Copious fumes of SO$_3$ came off at 480° C. The residue was rabbled slightly to help the drying of the residue. The temperature had reached 550° C. in 55 minutes with the door partly open on the furnace so that the gases could escape from the furnace. The dish was taken from the furnace 60 minutes after charging to rabbble the calcine and turn the dish end for end. A small amount of pink CoSO$_4$.xH$_2$O was noted on top of the calcine before rabbling. The calcine was charged back into the electric furnace and left overnight with the door partly open and the current off. This calcine weighed 129.6 grams. The calcine was divided into two parts of 64.8 grams each. The first part was treated as a 625° C. calcine product and the second as a 650° C. calcine product.

625° C. Sulfate Residue Calcine

Half of the dried sulfate residue weighing 64.8 grams at the start was roasted for one and one-half hours at 600° C. At this temperature the reaction was very slow and only the calcine nearest the source of heat turned from a pinkish color to a reddish brown. Only a slight amount of
SO₂ came off while the temperature was held at 600°C. The temperature was then raised to 625°C. for 90 minutes and almost all the FeSO₄ was oxidized to Fe₂O₃. The calcine was rabbled every 20 minutes, while roasting. The calcine was removed from the small furnace and the calcine transferred to a larger fused quartz dish, 37 cm. long, 23 cm. wide and 2 cm. deep. This calcine was then charged to the large electric furnace at 625°C. and rabbled from time to time. The calcine was removed 45 minutes after charging and was found to be darker red in color indicating that more iron had been oxidized during the last 45 minute roast. The calcine was cooled and screened through a -100 mesh screen to remove any asbestos picked up by the sulphated residue when filtering. The calcine weighed 54 grams.

650°C. Sulfate Roast Calcine

Put remainder of dried sulfate residue in small fused quartz dish and charged to small electric furnace. Roasted calcine at 650°C. for two hours. A small amount of SO₃ fumes came off first, followed by SO₂ fumes. The calcine was turned end for end several times during the roast period, and rabbled every ten minutes. The residue turned from a yellow to a reddish brown color in two hours. The product was allowed to cool in the furnace. The cooled product weighed 44 grams. The calcine was then transferred to a large fused quartz dish and charged to the large electric furnace at 650°C. The calcine was roasted at 650°C. for 45 minutes, and rabbled from time to time. There was little
change in color of the calcine during this final roast. The cooled calcine was screened through a 100 mesh screen to remove any asbestos. The product weighed 44 grams.

Reduction in weight from the original ore = \(100 - \left( \frac{44}{55.5} \times 100 \right) = 20.7\%\).

### Assays for 625° and 650° C. Calcines

<table>
<thead>
<tr>
<th>Assay Type</th>
<th>625° C. Calcine</th>
<th>650° C. Calcine</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water sol. Fe</td>
<td>1.3%</td>
<td>0.47%</td>
</tr>
<tr>
<td>&quot;</td>
<td>Co</td>
<td>3.60</td>
</tr>
<tr>
<td>&quot;</td>
<td>S</td>
<td>10.35</td>
</tr>
<tr>
<td>10% H₂SO₄ Sol. Fe</td>
<td>20.75</td>
<td>26.9</td>
</tr>
<tr>
<td>&quot;</td>
<td>Co</td>
<td>4.15</td>
</tr>
<tr>
<td>Total</td>
<td>Fe</td>
<td>20.9</td>
</tr>
<tr>
<td>&quot;</td>
<td>Co</td>
<td>4.16</td>
</tr>
<tr>
<td>&quot;</td>
<td>S</td>
<td>10.5</td>
</tr>
<tr>
<td>&quot;</td>
<td>As</td>
<td>1.7</td>
</tr>
<tr>
<td>Recovery Water sol.</td>
<td>Co</td>
<td>86.5%</td>
</tr>
<tr>
<td>&quot;</td>
<td>Acid</td>
<td>100%</td>
</tr>
</tbody>
</table>

### Treatment of the (conc.) H₂SO₄ Filtrate From the Acid Leach

The hot acid filtrate (210 c.c.) was decanted from the suction flask into a 400 c.c. beaker and allowed to stand for 9 days.

The As₂O₅ that adhered to the suction flask was dried and weighed (19.8 gm.)
Considerable amount of $\text{As}_2\text{O}_3$ had deposited on the inside of the beaker, and crystals of pink $\text{CoSO}_4 \cdot x\text{H}_2\text{O}$ had started to grow on the surface of the $\text{As}_2\text{O}_3$.

The $\text{H}_2\text{SO}_4$ was decanted off and about 250 c.c. of water added to leach the $\text{CoSO}_4 \cdot x\text{H}_2\text{O}$.

The leach solution was filtered off and assayed. The $\text{As}_2\text{O}_3$ was dried and weighed (16.6 gm.).

**Assays** $\text{H}_2\text{SO}_4$ Filtrate

Total amount of Co. in $\text{CoSO}_4 \cdot x\text{H}_2\text{O}$ crystals that separated out of the Saturated $\text{H}_2\text{SO}_4$ filtrate when cooling from 230° C. to room temperature, and standing for 9 days, was 0.1589 gm. or 9.73% of total cobalt in 111 gm. of ore.

210 c.c. of conc. $\text{H}_2\text{SO}_4$ saturated with $\text{CoSO}_4 \cdot x\text{H}_2\text{O}$ at 20° C.; contained 0.221 gm. Co. or 1.05 gm. per litre of conc. $\text{H}_2\text{SO}_4$.

This amounted to 3.65% of the Co in the ore.

The total amount of $\text{As}_2\text{O}_3 = 36.6$ gms.

**(ii) $\text{H}_2\text{SO}_4$ Leaching of No. 10 Calcine**

A charge consisting of

100 gm. No. 10 Calcine (-100)

15 c.c. (conc.) $\text{H}_2\text{SO}_4$

235 c.c. water

was agitated in a 1000 c.c. beaker for one hour at 70° C.

The charge was removed and filtered.

The volume of filtrate was made up to 400 c.c.
Assays

Water soluble Co. 1.23%
Total Co. 8.9%
Water soluble Fe 1.5%
Recovery of Co. = \( \frac{1.23}{8.9} \times 100 = 13.8\% \)

(iii) \( \text{H}_2\text{SO}_4 \) Leaching of Sulphatized Calcine

A. Cold Treatment

A charge consisting of
10 c.c. conc. \( \text{H}_2\text{SO}_4 \)
100 c.c. water
20 gm. sulphatized calcine.

was agitated in a 400 c.c. beaker for 30 minutes at room temperature. The charge was then filtered and washed. The filtrate was diluted to 200 c.c. and a 50 c.c. portion representing 5 gm. of calcine taken for a water soluble Co. assay, and a similar portion for a water soluble Fe assay. A large amount of water soluble As was found when assaying for Co.

Assays

Water soluble Co. 0.63%
" Fe. 0.92%
Total Co. 7.50%
Recovery of Co. = \( \frac{0.63 \times 100}{7.50} = 8.4\% \)
B. Hot Treatment

A similar charge under similar conditions was run, only the temperature was raised to 65° C.

Assays

<table>
<thead>
<tr>
<th></th>
<th>Co.</th>
<th>Fe.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble</td>
<td>2.07%</td>
<td>4.5%</td>
</tr>
<tr>
<td>Total Co.</td>
<td>7.5%</td>
<td></td>
</tr>
</tbody>
</table>

Recovery of Co. = \( \frac{2.07 \times 100}{7.5} = 26.8\% \)

(b) Water Leaching

No. 4 Sulphated Calcine

No. 1 Water leach

One hundred grams of No. 4 Sulphated Calcine were water leached with 250 c.c. of water at 80° C. for 30 minutes. The solution was mixed with a mechanical mixer during the leaching process.

The pulp was filtered through a suction filter and washed with water. Traces of pink could be seen in the final wash water.

The volume was brought up to 600 c.c. The solution was quite/dark pink color but the color indicated some iron in solution.

Assays

<table>
<thead>
<tr>
<th></th>
<th>Co.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble</td>
<td>5.66%</td>
</tr>
<tr>
<td>Total Co.</td>
<td>6.30%</td>
</tr>
</tbody>
</table>

Recovery = \( \frac{5.66 \times 100}{6.30} = 90\% \)
Water soluble Fe 2.6%
  " " As 0.42%

No. 2 Water Leach
The residue from the first water leach was water leached again with 250 c.c. of water at 80° C. for one hour and a half. The solution was mixed with a mechanical mixer during the leaching process.

The pulp was filtered as before and washed with hot water.

The solution was only faint pink in color. The filtrate was brought up to 400 c.c.

Assays
Water soluble Co 0.09%
Recovery of Co = \frac{0.09 \times 100}{6.30} = 1.43%

Assays on No. 2 Water Leach
Water soluble Fe Trace

Total Recovery of Water Soluble Co from No. 1 and No. 2 Leaching = \frac{5.75 \times 100}{6.30} = 91.5%

Residue from No. 1 and No. 2 water leaching.
The residue was dried and weighed.
Weight of residue 62.0 gm.
Reduction in weight of No. 4 Sulphated calcine by water leaching 38.0%

Assays on No. 4 Residue
Total As. 2.73%
" Fe. 39.0%
Total SiO₂ 27.9%

" Co 0.89%

Gold 2.22 oz/Ton

Recovery of gold in residue from No. 4 sulphated calcine

Leach = $\frac{2.22 \times 0.62 \times 100}{1.38} = 99.5\%$

No. 7G₂ Sulphated Calcine

No. 1 Water Leach

Three hundred and five grams of No. 7G₂ sulphated calcine were water leached with 750 c.c. of water at 80° C. for one hour. The solution was agitated during the leaching process.

The pulp was filtered through a suction filter and the residue washed with warm water. Traces of pink could be seen in the final wash water.

The filtrate was dark red in color and did not show much soluble iron.

The volume of filtrate was 1,070 c.c.

Assays on above Filtrate

<table>
<thead>
<tr>
<th>Component</th>
<th>Assay</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble Co.</td>
<td>6.30%</td>
</tr>
<tr>
<td>Total Co.</td>
<td>6.65%</td>
</tr>
<tr>
<td>Recovery of Co.</td>
<td>$\frac{6.30}{6.65} \times 100 = 95.00%$</td>
</tr>
<tr>
<td>Water Soluble Fe.</td>
<td>0.62%</td>
</tr>
<tr>
<td>&quot; As.</td>
<td>0.43%</td>
</tr>
</tbody>
</table>
Residue from water leach

Weight of residue 218 gm.
Total As. in residue 3.64%
Total Co. 0.49%
" Fe. 37.30%
" Insol. 23.80%
D. Precipitation of Cobalt from Leach Solutions

Test No. I

One hundred c.c. of the 600 c.c. of filtrate produced by leaching 100 gm. of No. 4 Sulphated Calcine was treated as follows.

Iron Arsenic Precipitation

Sodium carbonate was added to precipitate the iron and arsenic.

The solution was agitated at 20° C. and 3 c.c. of 5% H₂O₂ were added to oxidize the ferrous iron; 1.7 gm. of Na₂CO₃ to precipitate the As and Fe as ferric arsenate (FeAsO₄) and the remainder of the iron that was in excess of the soluble arsenic as Fe(OH)₃.

A light-brown, flocculent to granular precipitate was produced by digesting at room temperature for 15 minutes.

The precipitate of iron and arsenic was filtered off and the filtrate treated for the cobalt precipitation.

Cobalt Precipitation

The cobalt was precipitated by adding 3.6 gms. of Na₂CO₃ to the filtrate at 80° C. The solution was agitated for 30 minutes and then filtered.

The precipitate of cobalt formed almost instantaneous with the addition of Na₂CO₃, and it was mauve to purple in color.

The cobalt precipitate was ignited for 45 minutes at 800° C.

Weight of cobalt oxide produced 1.006 gm.

The cobalt oxide was re-heated at 800° C. for one hour.

Weight of final cobalt oxide 0.9975 gm.

Water soluble Co in 100 c.c. of cobalt filtrate 0.9450 gm.

The iron precipitate contained 0.2166 gm. Co.

The per cent. of cobalt in the cobalt oxide made

\[ \frac{0.9450 - 0.2166}{0.9450} \times 100 = 73.1\% \text{ Co.} \]

The filtrate from the cobalt precipitation contained only a trace of water soluble cobalt.

Test No. II.

100 c.c. of the 600 c.c. of filtrate produced by leaching 100 grams of No. 4 Sulphated Calcine was treated as follows:

Precipitating Fe and As

The cobalt solution was heated to 80° C. and 1.25 grams of CaCO₃ were added. The solution was agitated for 30 minutes at 80° C. A light-brown, flocculent to granular precipitate was formed.

The precipitate was filtered off and the filtrate saved for further precipitation.

The iron precipitate was assayed for Co. and was found to contain 0.0025 gm. Co.
The filtrate from the first iron precipitation was divided into two equal portions of 100 c.c. each. One portion was treated with 0.5 gm. of CaCO$_3$ to precipitate the remaining iron. The solution was agitated at 80° C. for 15 minutes. The iron precipitate was filtered out and the filtrate saved for further precipitation.

The second iron precipitate was assayed for Co and was found to contain 0.0051 gm. Co.

The filtrate from the second iron precipitation was treated as follows. The solution was heated to 80° C. and 0.5 gm. CaCO$_3$ was added. The solution was agitated for 10 minutes at 80° C. but no brown ppt. of iron was formed. The solution remained pink. The ferrous iron was then oxidized with 5 c.c. of 5% H$_2$O$_2$ and a brown precipitate of ferric hydrate formed. The solution was agitated for 30 minutes at 80° C. and then filtered.

The filtrate was made up to 200 c.c. and a twenty c.c. portion treated as follows. The 20 c.c. of filtrate was made basic with NH$_4$OH and a dark blue ppt. formed. No precipitate of iron was noticed when the NH$_4$OH was added.

The third iron precipitate was assayed for Co and was found to contain 0.0180 gm. Co.

**Cobalt Precipitation**

The remainder of the filtrate (180 c.c.) from the third iron precipitation was warmed to 80° C. and 1 gram of Na$_2$CO$_3$ in 10 c.c. of water was added to precipitate the cobalt.
The solution was agitated for 20 minutes before filtering off the cobalt precipitate. The cobalt precipitate was purple in color and quite flocculent. The filtrate was still pink in color indicating that there was not enough Na₂CO₃ added to precipitate all the cobalt.
The filtrate from the first cobalt precipitation was heated to 80° C. and 0.5 gm. Na₂CO₃ was added to precipitate the remaining cobalt. The solution was agitated at 80° C. for 15 minutes and then filtered. The cobalt precipitate was washed with warm water and then combined with the first cobalt precipitate. The filtrate from the second cobalt precipitation was free of cobalt.

**Roasting Cobalt Precipitate to Cobalt Oxide**

The cobalt precipitate was dried on top of a low plate and then heated at 800° C. for two hours.

Weight of Cobalt oxide produced = 0.5380 gm.

Weight of Co in Cobalt Oxide = \( \frac{5.66 \times 100 \times 1 \times 160}{600 \times 2 \times 200} \)

\( = 0.025 \text{ gm.} = 0.4000 \text{ gm. Co} \)

\% Co in Cobalt Oxide \( \frac{0.4000 \times 100}{0.5380} = 74.5\% \text{ Co} \)

**Test No. III**

**Water leaching**

200 gm. of No. 4 Sulphated Calcine was water leached with 500 c.c. of water at 80° C. for one hour. The solution was filtered and the residue washed. The filtrate was made up to 1000 c.c. This cobalt solution contained 0.0111 gm. Co per c.c. of solution and 0.0062 gm. Fe per c.c.

**Precipitation of the Fe and As**

The cobalt solution (996 c.c.) was heated to 50° C. and 21 gm. of CaCO₃ were added to precipitate the As and Fe. The solution was agitated for 30 minutes at 50° C. The
light-brown precipitate of iron was filtered off and the filtrate saved for further precipitation.

The filtrate from the first iron precipitation was treated with 12 gm. CaCO₃ and 40 c.c. of 3% H₂O₂ at 50° C. for 15 minutes. The solution was agitated during the precipitation. The solution was filtered and the filtrate saved for the cobalt precipitation. The second iron precipitate was darker brown than the first.

**Cobalt Precipitation**

The filtrate from the second iron precipitation was treated with 35 gm. Na₂CO₃ at 80° C. for 15 minutes. The cobalt precipitate was purple in color and of a flocculent nature. The solution was agitated during the precipitation of the cobalt. The solution was filtered and the precipitate washed with warm water. The filtrate was free of cobalt.

The precipitate was dried on a low plate and the volume of the precipitate decreased to about 1/30 of the original cobalt precipitate. The precipitate was dark purple in color and was fairly hard and brittle.

The dry precipitate weighed 30.233 gm. The cobalt precipitate was broken up and ignited at 800° C. for one hour.

**Weight of calcined product** 22.0545.

The cobalt oxide produced still looked glassy and black.

The calculated cobalt content of this cobalt oxide was 49.5% Co.
Test No. IV.

The cobalt filtrate (1066 c.c.) from the water leaching of 305 gm. of No. 7G2 Sulphated Calcine was treated as follows for the precipitation of As., Fe., and Co.

Precipitation of the Fe and As

The cobalt solution containing about 18 mg. Co. per c.c. was heated to 60° C. and 20 gm. of CaCO₃ were added to precipitate the As and Fe. The solution was agitated for 30 minutes at 60° C. and then filtered.

The solution did not turn brown indicating that no iron was precipitated.

A white precipitate was collected on the filter paper, which was likely CaSO₄ caused by the neutralization of the acid solution.

The above filtrate was treated with 15 c.c. 5% H₂O₂ to oxidize the iron and 6 gm. of CaCO₃ added to precipitate the iron. The solution was agitated at 60° C. for 30 minutes and the iron precipitate filtered off.

The filtrate was treated again with 10 c.c. of 5% H₂O₂ and 5 gm. CaCO₃ and the remainder of the iron filtered off in 15 minutes.

The filtrate from the last iron precipitation was further treated with 5 c.c. of 5% H₂O₂ and 3 gm CaCO₃. The solution was agitated at 70° C. for 15 minutes. No precipitate was formed by this treatment of the filtrate. The excess CaCO₃ was filtered out and the filtrate treated to precipitate the cobalt.
Cobalt Precipitation

The filtrate from the final iron precipitation was treated with 30 gm. Na₂CO₃ at 80° C. for 20 minutes. The cobalt precipitate was filtered and washed with warm water.

The small amount of cobalt remaining in the filtrate was precipitated with 5 gm. Na₂CO₃. The second cobalt precipitate was filtered off and the clear filtrate rejected.

The cobalt precipitates were dried and ignited at 800° C. for 90 minutes.

Weight of cobalt oxide 27.0315 gm.
Calculated cobalt content of Cobalt oxide 67.5%.

Test No. V.

Three hundred and seventy c.c. of the 600 c.c. of filtrate produced by leaching 100 grams of No. 4 Sulphated Calcine was treated as follows.

Precipitating Fe and As

The filtrate containing 0.00945 gm. Co. per c.c.; 0.00434 gm. Fe per c.c.; 0.000068 gm. As per c.c., was agitated for 30 minutes at 60° C. with 8 gm. CaCO₃. The solution was filtered to get rid of the iron precipitate.

The filtrate from the first iron precipitate was treated with 4 gm. CaCO₃ and 10 c.c. of 5% H₂O₂ to precipitate the remaining iron. The iron precipitate was filtered off, after the solution was agitated for 15 minutes at 70° C.
Cobalt Precipitation

The filtrate from the Fe - As precipitation was heated to 80° C. and 8 gm. Na$_2$CO$_3$ added to precipitate the cobalt. The solution was agitated for 30 minutes and then the purple precipitate filtered off. The filtrate did not show any traces of cobalt.

Cobalt Oxide

The cobalt precipitate was dried and ignited at 800° C. for 2 hours.

The cobalt oxide produced contained 72.1% Co.

Weight of Cobalt Oxide 4.5729 gm.

Test No. VI.

Two hundred grams of No. 8 Sulphated calcine were water leached with 500 c.c. water for one hour at 70° C. The solution was filtered through a suction filter and the leached residue washed with warm water.

The filtrate was made up to 600 c.c. and contained 0.0204 gm. Co. per c.c., 0.00167 gm. Fe per c.c.

Precipitating Fe and As

The above filtrate was agitated for 20 minutes at 70° C. with 10 gm. CaCO$_3$. The solution was filtered to remove the iron precipitate.

The filtrate from the first iron precipitate was warmed to 70° C. and 5 gm. CaCO$_3$ added and the solution agitated. No precipitate came down indicating that all the ferric iron was removed in the first iron precipitation. The
remaining ferrous iron was oxidized with 15 c.c. of 5% \( \text{H}_2\text{O}_2 \) and a light brown iron precipitate was filtered off and the filtrate treated with 3 gm. CaCO\(_3\) and 5 c.c. of 5% \( \text{H}_2\text{O}_2 \). No precipitate formed and the excess CaCO\(_3\) was then filtered off.

**Cobalt precipitation**

The filtrate free of iron and arsenic was warmed to 80° C. and 21 gm. \( \text{Na}_2\text{CO}_3 \) were added to precipitate the cobalt. The purple precipitate of cobalt formed at once and the solution was agitated for 20 minutes before filtering.

The filtrate was still slightly pink. Four grams of \( \text{Na}_2\text{CO}_3 \) were added to precipitate the remaining cobalt.

**Cobalt Oxide.**

The cobalt precipitates were dried and ignited at 800° C. for 2 hours.

- Weight of cobalt oxide: 17.8790 gm.
- % Co in Cobalt oxide: 66.5%

![Fig. 1. The small annealing furnace used to ignite cobalt precipitate and the apparatus used to agitate the solutions.](image-url)
E. Smelting

Test No. I.

A No. 6 Morgan graphite crucible was heated to 900° C. in the electric furnace and allowed to anneal for 12 hours, in the furnace as it cooled down, with the door shut.

A charge consisting of:

- Calcine (3+4) (100 parts) 300 grams
- Limestone (9") 27"
- Silica (18") 54"
- Charcoal (5") 15"

was thoroughly mixed on a rubber mat and then charged to a No. 6 Morgan graphite crucible. A borax glass cover was added (10 gm) before placing inside the gas-fired furnace which had been warmed up to a white-heat. The amount of gas and air was so regulated that a reducing atmosphere was maintained. The charge fused quite easily at about 1350° C.

The molten charge, at first quite tranquil, started to foam as smelting proceeded. The foaming caused the slag to fill the crucible and to allow some of the slag to run over the top and lost inside the furnace. When an oxidizing atmosphere was maintained the foaming stopped, but since the oxidizing flame would burn a hole through the crucible a reducing atmosphere was maintained during the smelting operations.

The molten charge was poured in 20 minutes at about 1450° C. into a small conical-shaped iron mold lined with fire clay.
Weight of products

Speiss - 21.0 gm.
Slag - 320.1 gm.

Characteristics of Products

Speiss:
The speiss was very friable and appeared quite pebbly on the surface.

Slag:
The slag was a black porous frothy slag with small shots of speiss in it.

Assays

<table>
<thead>
<tr>
<th></th>
<th>Weight</th>
<th>% Co</th>
<th>% Co. Rec'y</th>
<th>Fe %</th>
<th>As %</th>
<th>Insol.</th>
<th>CaO %</th>
<th>S %</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1 Speiss</td>
<td>21.0</td>
<td>45.1</td>
<td>36.9</td>
<td>14.7</td>
<td>37.4</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>97.2%</td>
</tr>
<tr>
<td>#1 Slag</td>
<td>320.1</td>
<td>4.4</td>
<td>55.0</td>
<td>28.3</td>
<td>1.52</td>
<td>37.16</td>
<td>6.16</td>
<td>-</td>
<td>77.6%</td>
</tr>
</tbody>
</table>

Conclusions

1. The charge was calculated for a bisilicate slag and a bi-silicate slag was produced.

2. The poor recovery was due to shots of speiss in the slag, and to some extent to the loss in foaming and dusting. Some cobalt combines with the acid slag.

Test No. 2

A charge consisting of

Calcine (3+4) (100 parts) 300 gm.
Limestone (5 parts) 15 "
Charcoal (5 parts) 15 "
was thoroughly mixed as in Test No. 1 and charged to the same crucible and a 10 gm. borax cover added. The crucible was charged to the furnace as before. The charged melted more rapidly than No. 1 and appeared to be more fluid. The charge melted without foaming and no slag was lost in the furnace. A small amount of arsenic could be noticed escaping from the charge during the fusion period.

The molten charge was poured in 15 minutes into the iron mold.

Weight of Products

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Speiss</td>
<td>87.2 gm.</td>
</tr>
<tr>
<td>Slag</td>
<td>237.1 gm.</td>
</tr>
</tbody>
</table>

Characteristics of Products

**Speiss:**

The speiss produced was hard and dense. Considerable difficulty was experienced when preparing the assay sample to get it fine enough.

**Slag:**

The slag was fairly dense near the speiss. There were less air holes than No. 1. The slag contained a few specks of speiss but not so many as slag no. 1.

Assays

<table>
<thead>
<tr>
<th></th>
<th>Weight gm.</th>
<th>% Co.</th>
<th>% Co Recovery</th>
<th>Fe %</th>
<th>As %</th>
<th>% Insol.</th>
<th>CaO</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>#2 Speiss</td>
<td>87.2</td>
<td>24.3</td>
<td>82.5</td>
<td>60.9</td>
<td>14.62</td>
<td>-</td>
<td>-</td>
<td>99.8</td>
</tr>
<tr>
<td>#2 Slag</td>
<td>237.1</td>
<td>1.89</td>
<td>17.5</td>
<td>22.5</td>
<td>0.77</td>
<td>41.46</td>
<td>6.48</td>
<td>73.1</td>
</tr>
</tbody>
</table>
Conclusions

1. The foaming is caused by the addition of silica to the charge and the reducing atmosphere.
2. The slag produced was almost a bisilicate slag.

Test No. 3

A charge consisting of

<table>
<thead>
<tr>
<th>Component</th>
<th>Quantity</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcine</td>
<td>(5) (100 parts)</td>
<td>300 gm.</td>
</tr>
<tr>
<td>Limestone</td>
<td>(4.5 %)</td>
<td>15 &quot;</td>
</tr>
<tr>
<td>Charcoal</td>
<td>(5 &quot; )</td>
<td>15 &quot;</td>
</tr>
</tbody>
</table>

was thoroughly mixed as in Test No. 1 and charged to the same crucible, and a 10 gm. borax cover added. The crucible was charged to the furnace as before. The charge melted without foaming. Bright spots of speiss could be seen on the surface of the slag just before it was poured. Copious fumes of arsenious oxide came off during the fusion period. The molten charge was poured in 15 minutes into the iron mold.

Weight of Products

<table>
<thead>
<tr>
<th>Product</th>
<th>Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Speiss</td>
<td>143.8 grams</td>
</tr>
<tr>
<td>Slag</td>
<td>144.2 &quot;</td>
</tr>
</tbody>
</table>

Characteristics of Products

Speiss:

The speiss produced was quite friable, and was easily reduced to -100 mesh. There was no distinction between speiss and matte in this product.

Slag:

A very acid slag was produced and it contained very
few gas holes. The slag was dense and brittle.

**Assays**

<table>
<thead>
<tr>
<th>Weight (gm)</th>
<th>Co.%</th>
<th>% Co Recovery</th>
<th>Fe.%</th>
<th>As.%</th>
<th>Insol.</th>
<th>CaO%</th>
<th>S%</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>#3 Speiss</td>
<td>143.8</td>
<td>12.5</td>
<td>86.5</td>
<td>47.2</td>
<td>23.55</td>
<td>-</td>
<td>-</td>
<td>12.1</td>
</tr>
<tr>
<td>#5 Slag</td>
<td>144.2</td>
<td>1.90</td>
<td>13.5</td>
<td>14.3</td>
<td>1.67</td>
<td>40.18</td>
<td>9.10</td>
<td>67.2</td>
</tr>
</tbody>
</table>

**Conclusions:**

1. An excess of As. does not cause foaming.
2. The presence of S in the charge helps to collect the Co. but brings down iron as well.
3. A trisilicate slag was produced.

**Test No. 4**

A charge consisting of:

- Calcine (3+4) (100 parts) 600 gm.
- Limestone (9 parts) 54 "
- Silica (5 parts) 30 "
- Charcoal (5 parts) 30 "
- #2 Slag Cover (25 parts) 150 "

was thoroughly mixed and placed in a graphite crucible as before and the slag cover added to the charge. The crucible was charged to the furnace. As fusion took place the charge began to foam and some slag was lost. The charge was removed in 15 minutes and poured. Fusion was not complete so the slag was broken up and the whole charge remelted with 25 grams of charcoal.

The charge was poured in 20 minutes into the iron mould. The charge poured quite free but left shots of slag
in the crucible.

Weight of Products

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Speiss</td>
<td>123.0 gm.</td>
</tr>
<tr>
<td>Slag</td>
<td>241.3 gm.</td>
</tr>
</tbody>
</table>

Characteristics of Products

Speiss

The speiss produced was hard and dense. Considerable difficulty was experienced when preparing the assay sample to get it fine enough.

Slag

The slag was fairly dense and hard. There were small holes in slag but not as large as No. 3 slag. There were shots of speiss in the slag.

Assays

<table>
<thead>
<tr>
<th></th>
<th>Weight gm.</th>
<th>% Co</th>
<th>% Co Recovery</th>
<th>Fe.%</th>
<th>As.%</th>
<th>Insol.</th>
<th>CaO.</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>#2 Speiss</td>
<td>123.0</td>
<td>25.1</td>
<td>57.0</td>
<td>56.8</td>
<td>17.2</td>
<td>-</td>
<td>-</td>
<td>99.1</td>
</tr>
<tr>
<td>#2 Slag</td>
<td>241.3</td>
<td>0.90</td>
<td>4.0</td>
<td>18.3</td>
<td>0.61</td>
<td>48.12</td>
<td>3.61</td>
<td>76.5</td>
</tr>
</tbody>
</table>

Conclusions

1. The addition of silica caused foaming.
2. A large loss of slag resulted in a large loss of Co.
3. A sesquisilicate slag was produced.
Test No. 5

A charge consisting of

Calcine (3+4) (100 Parts) 500 gm.
Limestone (10 " ) 30 "
Charcoal (10 " ) 30 "

was thoroughly mixed as in Test No. 1 and charged to a No. 6 Morgan graphite crucible and 20 gm. of No.2 slag added as a cover. The crucible was charged to the furnace as before. The charge fused without foaming. The molten charge appeared to be quite fluid and shots of speiss could be seen digesting in the slag while the charge was in the furnace.

The charge was poured in 25 minutes into the iron mold. The slag seemed quite viscous on pouring.

Weight of Products

Speiss 123.0 gm.
Slag 176.5 gm.

Characteristics of Products

Speiss

The speiss produced was hard and dense. Considerable difficulty was experienced when preparing the assay sample to get it fine enough.

No shots of speiss were noticed in the slag.

Slag

The slag produced was dense and heavy. Only a few gas holes were noticed in the glassy acid slag.
Assays

<table>
<thead>
<tr>
<th>Weight gm</th>
<th>Co.%</th>
<th>% Co. Recovery</th>
<th>Fe.%</th>
<th>Insol.</th>
<th>As.</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>#5 Speiss</td>
<td>123.0</td>
<td>20.3</td>
<td>96.2</td>
<td>-</td>
<td>13.5</td>
<td>98.0</td>
</tr>
<tr>
<td>#5 Slag</td>
<td>176.5</td>
<td>0.30</td>
<td>2.0</td>
<td>11.8</td>
<td>55.8</td>
<td></td>
</tr>
</tbody>
</table>

Conclusions

1. About 2% of the cobalt was lost by dusting in the furnace.
2. Iron reduced from the slag by excessive digesting in a molten state combines with the speiss to make a very hard speiss.
3. A bisilicate slag was produced.

Test No. 6

A charge consisting of

Calcium (3+4) (100 parts) 300 gm.
Limestone (5") 15 "
Pumice (6") 18 "
Charcoal (10") 30 "

was thoroughly mixed as in previous tests and charged to a graphite crucible. A 20 gm. portion of No. 2 slag was added as a cover.

The crucible was charged to the furnace as before. The charge fused readily but the volume of molten slag seemed to increase due to some foaming. The foaming slag did not fill the crucible so no slag was lost. As the charge was fusing considerable dusting resulted.

The charge was poured in 20 minutes into the iron mold lined with fire clay. The slag seemed to be quite
viscous and dense on pouring.

Note: 1

Pumice was used instead of silica because a large supply of pumice of the following composition is available at the mine:

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>63.94%</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>16.34%</td>
</tr>
<tr>
<td>MgO</td>
<td>1.58%</td>
</tr>
<tr>
<td>CaO</td>
<td>3.18%</td>
</tr>
<tr>
<td>Na₂O K₂O</td>
<td>8.39%</td>
</tr>
<tr>
<td>H₂O</td>
<td>2.75%</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.45%</td>
</tr>
</tbody>
</table>

The pumice was ground to -65 mesh before using.

Weight of Products

<table>
<thead>
<tr>
<th>Type</th>
<th>Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Speiss</td>
<td>103.5 gm.</td>
</tr>
<tr>
<td>Slag</td>
<td>172.0 gm.</td>
</tr>
</tbody>
</table>

Characteristics of Products

Speiss

The speiss produced was hard and dense. Considerable difficulty was experienced when preparing the assay sample to get it fine enough.

Slag

The acid slag was more porous than No. 5 slag.

Assays

<table>
<thead>
<tr>
<th></th>
<th>Weight</th>
<th>Co %</th>
<th>% Co Recovery</th>
<th>Fe</th>
<th>Insol.</th>
</tr>
</thead>
<tbody>
<tr>
<td>#6 Speiss</td>
<td>103.5</td>
<td>21.2</td>
<td>84.5</td>
<td>58.8</td>
<td>-</td>
</tr>
<tr>
<td>#6 Slag</td>
<td>172.0</td>
<td>0.61</td>
<td>4.0</td>
<td>9.15</td>
<td>58.8</td>
</tr>
</tbody>
</table>

Conclusions:
1. The pumice reacted as a flux much similar to silica.
2. The pumice made the slag quite viscous.
3. A bisilicate slag was produced.

Test No. 7
A charge consisting of
Calcine \((6+7+8)\) (100 parts) 300 gm.
Limestone (5") 15"
Silica (5") 15"
Charcoal (10") 30"
was thoroughly mixed as in previous tests and charged to a graphite crucible. A 20 gm. portion of No. 4 slag was added as a cover.

The crucible was charged to the furnace as before. The charge fused without foaming. The slag appeared quite fluid when digesting.

The charge was poured in 20 minutes into the iron mold lined with fire clay.

Weight of Products
Speiss 121.5 gm.
Slag 183.0 gm.

Characteristics of Products
Speiss

The speiss was very friable and little difficulty was experienced in crushing it to -65 mesh.

Slag

The acid slag produced was quite porous and no shots
of speiss were noticed in the slag.

**Assays**

<table>
<thead>
<tr>
<th></th>
<th>Weight</th>
<th>Co.%</th>
<th>% Co. Recovery</th>
<th>Fe.%</th>
<th>Insol.</th>
</tr>
</thead>
<tbody>
<tr>
<td>#7 Speiss</td>
<td>121.5</td>
<td>16.6</td>
<td>85.9</td>
<td>44.4</td>
<td>-</td>
</tr>
<tr>
<td>#7 Slag</td>
<td>183.0</td>
<td>0.70</td>
<td>5.5</td>
<td>20.3</td>
<td>48.5</td>
</tr>
</tbody>
</table>

**Conclusions**

1. The high sulphur content in the calcine used helps to bring iron down into the speiss.
2. Continued digesting of the speiss in the slag causes more iron to go into the speiss.
3. A bisilicate slag was produced.

**Test No. 6**

A charge consisting of

- Calcine 2 (100 parts) 300 gm.
- Limestone (9 " ) 27 "
- Silica (12 " ) 36 "
- Charcoal (10 " ) 30 "

was thoroughly mixed as in previous tests and charged to a graphite crucible. A 20 gm. portion of No. 4 slag was added as a cover.

The crucible was charged to the furnace as before. The charge fused and started to foam. The foaming slag filled the crucible but only a small amount of slag was lost.

The charge was poured in 20 minutes into the iron mold lined with fire clay. The slag was very viscous when poured.
Weight of Products

Speiss  44 gm.
Slag  304.8 gm.

Characteristics of Products

Speiss

The speiss was very friable and the slag contained a large amount of speiss.

Slag

The acid slag contained a large number of shots of speiss. A magnet picked up a large percentage of the speiss contained in the crushed slag.

Assays

<table>
<thead>
<tr>
<th></th>
<th>Weight</th>
<th>Co.%</th>
<th>% Co Recovery</th>
<th>Fe</th>
<th>Insol.</th>
</tr>
</thead>
<tbody>
<tr>
<td>No.8 Speiss</td>
<td>44.0</td>
<td>35.5</td>
<td>64.3</td>
<td>32.4</td>
<td>-</td>
</tr>
<tr>
<td>No.8 Slag</td>
<td>304.8</td>
<td>2.36</td>
<td>29.5</td>
<td>26.2</td>
<td>43.0</td>
</tr>
</tbody>
</table>

Conclusions

1. The addition of silica causes foaming.
2. The more acid slag is more viscous and holds up shots of speiss which lowers the cobalt recovery.
3. The more acid slag holds up the iron and prevents iron going into the speiss. This causes a higher grade speiss.
4. A bisilicate slag was produced.
Test No. 9

A charge consisting of

Calcine \( (6 + 7 + 8) \) (100 parts) 600 gm.
Limestone \( (5 \text{ parts}) \) 30 "
Silica \( (5 \text{ parts}) \) 30 "
Charcoal \( (7.5 \text{ parts}) \) 45 "

which was similar to Test No. 7 charge was thoroughly mixed as in previous tests and charged to a graphite crucible. A 20 gm. portion of No. 4 slag was added as a cover.

The crucible was charged to the furnace as before. The charge fused without foaming. A hole was burnt through the side of the crucible allowing all the slag and part of the speiss to run out.

The remaining speiss was poured in 20 minutes into the iron mold lined with fire clay.

Weight of Products

<table>
<thead>
<tr>
<th></th>
<th>Speiss</th>
<th>Slag</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight</td>
<td>117.0 gm.</td>
<td>None recovered.</td>
</tr>
</tbody>
</table>

Characteristics of Products

Speiss

The speiss was very friable and little difficulty was experienced in crushing it to -65 mesh.

Assays

<table>
<thead>
<tr>
<th></th>
<th>Weight</th>
<th>Co.%</th>
<th>% Co. Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>#9 Speiss</td>
<td>117.0</td>
<td>20.4</td>
<td>51.0</td>
</tr>
<tr>
<td>#9 Slag</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Fe %</th>
</tr>
</thead>
<tbody>
<tr>
<td>#9 Speiss</td>
<td>38.1</td>
</tr>
<tr>
<td>#9 Slag</td>
<td></td>
</tr>
</tbody>
</table>
Conclusions.

1. Not so much iron is taken up by speiss if it is not allowed to digest very long in the molten slag.
2. The first speiss produced from the molten charge likely contains more cobalt.

Test No. 10

A charge consisting of

<table>
<thead>
<tr>
<th>Material</th>
<th>Parts</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcine (5+7+8)</td>
<td>100 parts</td>
<td>600 gm.</td>
</tr>
<tr>
<td>Limestone</td>
<td>5 &quot;</td>
<td>30 &quot;</td>
</tr>
<tr>
<td>Silica</td>
<td>5 &quot;</td>
<td>30 &quot;</td>
</tr>
<tr>
<td>Charcoal</td>
<td>7.5 &quot;</td>
<td>45 &quot;</td>
</tr>
</tbody>
</table>

which was similar to Tests No. 7 and 9 was thoroughly mixed as in previous tests and charged to a graphite crucible. A 20 gm. portion of No. 4 slag was added as a cover.

The crucible was charged to the furnace as before. The charge fused without foaming, and no slag was lost.

The charge was poured in 20 minutes into the iron mold lined with fire clay. The slag was quite fluid on pouring.

Weight of Products

- Speiss 219.5 gm.
- Slag 360.8 gm.

Characteristics of Products

The speiss was very friable and little difficulty was experienced in crushing it to -65 mesh.
Slag:

The slag was very dense. The acid slag contained no air holes. There were a few shots of speiss in the slag.

### Assays

<table>
<thead>
<tr>
<th></th>
<th>Weight</th>
<th>% Co.</th>
<th>% Co. Recovery</th>
<th>Fe.</th>
<th>Insol.</th>
</tr>
</thead>
<tbody>
<tr>
<td>#10 Speiss</td>
<td>219.5</td>
<td>17.75</td>
<td>83.2</td>
<td>14.4</td>
<td>-</td>
</tr>
<tr>
<td>#10 Slag</td>
<td>360.8</td>
<td>0.92</td>
<td>7.1</td>
<td>23.7</td>
<td>44.5</td>
</tr>
</tbody>
</table>

Conclusions:

1. The cobalt content was lowered somewhat by the speiss taking up more iron from the slag when digesting.
2. About 10% of the cobalt in the charge was lost in dusting.
3. A bisilicate slag was produced.
VI. APPENDIX
VI. APPENDIX

A. Cobalt Statistics

Reference is given to the writer's Metallurgy 7 Essay, "Cobalt", which deals with the properties, uses, production, imports, exports, prices and markets of cobalt.

B. Previous Testing

A brief investigation was carried out by P. Leckie-Ewing, Met.'39, and Percy A. Adams, Met. '39, to determine the feasibility of an all-electric process for smelting and refining the cobalt-gold ore from the Gem Mine. This research was carried out under the direction of Mr. Frank A. Forward, Professor of Metallurgy, University of British Columbia.

The results of the above research, as outlined in the Metallurgy 8 Research report titled "Experimental Reduction and Electrolysis of Cobalt Ore from the Little Gem Mine", indicate that an all-electric process for the smelting and refining of the ore is quite feasible.

C. Proposed Tests for Future Experimental Work

I. To study the possibility of driving off the greater part of the arsenic at 800 to 850° C. and to retain as much sulphur as possible in the calcine by removing the charge as soon as the last traces of Arsenic come off, and cool the calcine in a reducing atmosphere to prevent further oxidation of any remaining sulphur.

The above calcine can be further treated in several
(a) If the sulphur to cobalt ratio is greater than 32:59 a sulphating roast at 450° C. for two hours with constant rabbling might produce encouraging water soluble cobalt results.

(b) If the sulphur to cobalt ratio is less than 32:59, sulphur could be added in the form of pyrite and treated as above. The addition of 10 - 20% salt to the sulphating charge might increase the water soluble cobalt considerably. The addition of 2 - 3% charcoal to the charge in the case where 4 - 5% arsenic is left in the primary calcine will likely give increased amounts of water soluble cobalt.

II. The possibility of producing water soluble compounds by the addition of ferrous sulphate or sodium sulphate to the sulphating charge, could be experimented with.

III. To try various sulphating tests on low arsenic and roasted speiss.

IV. Further smelting tests in a suitable electric furnace similar to the ones outlined in this report are required to determine the possibility of producing a cobalt speiss that combines high recovery and high concentration of both the gold and cobalt contained in the ore.

V. To determine the feasibility of extracting a high-grade electrolytic cobalt from the leach solution after the first iron precipitate containing most of the water soluble iron and
arsenic has been filtered off.
D. Assay Procedures

ARSENIC

Distillation Method

Weigh out 1.0 gm. of sample in 250 c.c. beaker, add 10 c.c. chlorate mixture and take to dryness. Add 10 c.c. HCl and take to dryness again. Add 20 c.c. 1:1 HCl, and when in solution transfer to a 275 c.c. distillation flask containing a few grape-nuts. Add 0.5 gm. sodium bromide and 0.5 gm. hydrazine sulphate, and 75 c.c. HCl. Receive in a 500 c.c. beaker containing about 100 c.c. water with tip of condenser just under surface. Distill till volume in flask is down to about 20 c.c. then add 15 c.c. HCl and take down to 20 c.c. again. This should take about 40 minutes. Wash out condenser tube into beaker.

Add a few drops of methyl orange to the distillate in the beaker and add NH₄OH till solution becomes basic, i.e., solution changes from red to yellow. Make acid with HCl (red). Cool solution and make it alkaline with NaHCO₃ (add carefully to avoid foaming over), add 10 c.c. starch solution indicator and titrate with standard iodine solution.

The distillation method is used when the sample contains antimony. Antimony will titrate with the arsenic and hence give an incorrect assay if the fusion method is used.
Standard Iodine Solution:

Dissolve 50 gm. KI in 75 c.c. water in a 500 c.c. beaker. Add 25.5 gm. iodine, and leave in a warm place till in solution. Take bulk to 2 litres.

Take up .099 gm. As₂O₅ in a 400 c.c. beaker with ½ stick of NaOH and water. Make just acid with HCl, and take bulk to 300 c.c., and cool. Make alkaline with NaHCO₃ as noted before, add starch indicator and titrate till stirring rod is invisible.

Starch Indicator

Mix 2 gm. of prepared starch with 30 c.c. water. Pour into 100 c.c. boiling water and boil for about 4 minutes. This solution should be alkaline, hence add some KOH to the solution before using. A few drops of chloroform added to the starch solution will act as a preservative.

Fusion Method

Weigh 0.5 gm. ore in 8 oz. flask, add 2 gm. anhydrous sodium sulphate, 5 - 6 c.c. of conc. H₂SO₄ and 1/8 of a 9-cm. filter paper. Heat to fusion over hot element. Cool flask on its side or keep contents rotating till it solidifies.

To cold solid in flask add 25 c.c. of hot water and warm without boiling to disintegrate completely. Remove from heat, add 25 c.c. conc. HCl and agitate gently to dissolve anhydrous ferric sulphate. Dilute to 100 c.c. and pass in HgS for 10 min. while hot. The arsenic is ppted in the arsénious condition.
Filter off the $\text{As}_2\text{S}_3$, wash the delivery tube with
$\text{H}_2\text{S}$ water. Wash flask seven times with $\text{H}_2\text{S}$ water and pour
over ppt to remove HCl.

Place flask under funnel and cleanse delivery tube
with a few drops of warm 5% NaOH. Dissolve ppt with 10 c.c.
of 5% NaOH run slowly over ppt

Use 5 c.c. NaOH in 50 c.c. hot water for washing.

Wash seven times and keep filtrate small for boiling.

Add 2 gm. anhydrous sodium sulfate and 6 c.c. conc.
$\text{H}_2\text{SO}_4$. Boil down to small bulk and then to a melt.

Cool flask on side or rotate it till it solidifies.

Add 50 c.c. hot water and heat till in solution and
boil to expel $\text{SO}_2$. Add 50 c.c. cold water and cool under tap
to room temp. Add a few drops of phenolphthalein as indicator
and make alkaline with 20% NaOH soln.

Acidify with HCl and cool.

Add 3 - 4 gm. sodium acid carbonate slowly till it
no longer effervesces. Add 10 c.c. starch solution.

Titrate with standard iodine solution till blue.

Notes

If the ore contains antimony wash the $\text{As}_2\text{S}_3$ ppt.
with (25 c.c. water and 50 c.c. HCl) to remove the antimony.

Tartaric acid can be used instead of the filter
duster to reduce the arsenic.

Only a small amount of tartaric acid should be used
or else the solution will tend to boil over.
CALCIUM

Weigh 0.5 gm. ore into a 250 c.c. beaker, add 10 c.c. HCl, cover with a watch-glass and place on a hot plate. When violent action ceases add 5 c.c. HNO₃ and boil till all brown fumes are gone. (HCl and HNO₃ can be added in reverse order if desired.) Then place on low plate, rinse off cover glass and take to dryness. Bake at 120° C. for ½ hour, cool, add 10 c.c. HCl and 40 c.c. water, boil till clear and filter through a 12½ cm. rapid filter. Wash with boiling water then 1:1 HCl in succession till all yellow stains of ferric chloride are out of the filter paper, then wash with water 4 times.

To filtrate add 5 gm. NH₄Cl and NH₄OH till alkaline, and then add 2 c.c. H₂O₂. Boil for 3 minutes, then filter into a 400 c.c. beaker and wash twice with boiling water. Wash hydroxides off paper into original beaker, dissolve in 5 c.c. HCl, add 5 gm. NH₄Cl, add NH₄OH till alkaline and 2 c.c. H₂O₂. Boil 3 minutes and filter through original paper, then wash 4 times with boiling water. (Be sure to precipitate, as Fe. and Al. hydroxides hold up lime and Cu.)

To combined filtrates add HCl till acid then 1 c.c. in excess. Add 3 gm. ammonium oxalate, bring to boil and add NH₄OH drop by drop till alkaline. (Calcium oxalate is coarse and easily washed.) Allow ppt. to settle for ½ hour, decant soln. through filter, wash ppt. into filter and rinse beaker out to get rid of ammonium oxalate. Wash ppt. off into original beaker, fold filter paper over edge of beaker, add 100 c.c. hot water and 5 c.c. 1:1 H₂SO₄. Heat to about 60° C.
and titrate to pink color with standard KMnO₄ solution. Add filter paper and finish titration.

**Standard KMnO₄ Solution**

Weigh out 5.66 gm. of KMnO₄, dissolve in water and make solution up to one liter. Weigh out 0.45 gm. of oxalic acid and dissolve in 100 c.c. hot water. To hot soln. add 5 c.c. H₂SO₄ and titrate at about 60 degrees C. to a pink color with KMnO₄ soln.

.00630 gm. oxalic acid is equivalent to .00280 gm. CaO.

CaO equivalent per c.c. of KMnO₄ = \(\frac{.45 \times .00280}{(\text{No. c.c. KMnO₄} \times .00630)}\)
COBALT ANALYSIS

Weigh out 1 gm. Ore (0.2 - 2.0%).

Moisten with 2 - 3 c.c. Water - Add 10 - 15 c.c. HCl and digest until all oxides are in solution.

Add 3.5 c.c. chlorate mixture. Digest.

Add 15 c.c. 1:1 H₂SO₄. Take to copious fumes on hot plate.

Cool. Add a few drops of water and then 15 c.c. HCl and 7 c.c. HBr or 2.0 gm. NaBr.

Digest at boiling point for a few minutes and then evaporate to fumes again, leaving 1 - 2 c.c. free H₂SO₄ after fuming.

Add 50 c.c. water. Digest until all sulphates are in solution.

Dilute to 100 - 125 c.c. and cool to room temperature.

Gas with HgS for 10 min. Place on warm plate and bring just to boil. Gass again for 10 min. Cool to room temperature.

Filter into 300 c.c. electrolytic beaker washing two or three times with cool distilled water.

Note: The ppt may now be run for Cu. by dissolving in nitric acid and Bromine water, evap. to syrup.

Neutralizing with Sod. Carbonate, making acid with acetic, adding KI after cooling and titrate with thiosulphate.

From Filtration above: Boil off the H₂S, cool somewhat and add 10 c.c. H₂O₂.

Evap. to about 100 c.c. and cool again.

Add 2 gm. Sod. Acetate and, when the acetate is in solution,
neutralize with ammonia and add 40 c.c. excess ammonia.

Cool to room temperature.

Weigh up cathode and place the beaker on the machine. If
the level of the electrolyte is too low add 1:1 ammonia to
bring it about \( \frac{1}{4} \)" above the top of the electrode.
Plate for 1½ hours at 4 amperes.

Remove from machine, wash thoroughly in at least two waters
draining between washes on soft paper and then wash in
alcohol.

Drain, ignite the excess alcohol, dry over low flame and weigh.
The difference in weight is cobalt plus nickel.

Possibly Fe in cathode so redissolve in hot 1:1
\( \text{HNO}_3 \), take to fumes with 10 c.c. of 1:1 \( \text{H}_2\text{SO}_4 \). Cool, dilute,
neutralize with ammonia (vol. now 100 c.c.)

Add 40 - 50 c.c. ammonia, cool, replate as above.

---

Fig. 2 Electrolytic machine showing
position of electrodes and
electrolytic beaker.
COBALT ANALYSIS

Ores by Nitrosobetanaphthol
Weigh out 0.5 - 2 gm. ore in 250 c.c. beaker.
Add 10 - 15 c.c. HCl, digest till oxides in solution.
" 3 - 5 c.c. Chlorate mixture
" 15 c.c. 1:1 H₂SO₄ and take to fumes (1 - 2 c.c. free H₂SO₄
" 50 c.c. H₂O. Digest until sulphates in solution.
Dilute to 100 or 125 c.c.
Gas with H₂S for 5 - 10 minutes.
Bring just to boil.
Gas again with H₂S for 3 - 5 minutes.
Filter - Ppte. to Cu assay.
Boil filtrate to eliminate H₂S.
Add 10 - 15 c.c. 5% H₂O₂.
Pour into 500 c.c. Volumetric Flask.
Add ZnO emulsion to ppt. All iron - Cool. Filter,
pour into 400 c.c. beaker.
Add 6 c.c. HCl.
" 25 - 50 c.c. HAc.
Boil till all O₂ expelled.
Add Nitro. (10 c.c. of 10% Soln. for every 25 mg. Co present)
Filter, Ignite, and weigh as Co₃O₄.
Weight x .734 Weight of Co.
**INSOLUBLE**

Weigh 0.5 gm. ore into 150 c.c. beaker, add 15 c.c. HCl, cover till violent ceases, add 10 c.c. HNO₃ and heat till brown fumes disappear (the acids may be added in the reverse order, if desired). Place on a low plate, rinse off cover and take to dryness. Cool, add 10 c.c. HCl and again take to dryness, on a low plate. Bake for 30 minutes at 120° C. Cool, add 10 c.c. HCl, 15 c.c. water and 2 gm. NH₄Cl. Cover and boil till clear. Filter through rapid filter and wash 6 times with boiling water. Ignite in a clay cup till the residue is white in color and weigh.

**Notes:**

If the residue is white and does not show gray color on ignition, the assay may be considered an accurate silica assay.
IRON

Dichromate Method

For Oxides

Weigh 0.5 gm. into 250 c.c. beaker, add 10 c.c. water and 10 c.c. HCl. Warm soln. till clear, add 10 c.c. nitric chlorate mixture, then add 10 c.c. 1:1 H₂SO₄ and fume nearly to dryness.

For Sulphides

Dissolve 0.5 gm. ore in 250 c.c. beaker with 3 c.c. water and 10 c.c. HNO₃ and warm till fumes (red) come off. Add 10 c.c. HCl and proceed as above.

In either case, cool, add 25 c.c. water, 5 c.c. HCl and boil. While hot add SnCl₂, drop by drop till yellow color (Fe₂Cl₆) goes and add one drop in excess. Cool (to room temperature) and add mercuric chloride, about 20 c.c., to neutralize the excess SnCl₂ and titrate with potassium dichromate using K₄Fe(CN)₆ as indicator.

Preparation of Dichromate

335.05 gm. Fe react with 294.2 gm of potassium dichromate; \( \cdot \cdot \cdot \) .00459 gm. dichromate react with .005 gm. Fe. For 0.5 gm. sample the no. of c.c. dichromate would represent the percent of iron.

Dissolve 8.78 gm. of K₂Cr₂O₇ in two litres of water.

Standardization

Weigh up 0.7 gm. of ferrous ammonium sulphate, acidify with HCl, add one drop SnCl₂; excess of mercuric
chloride and titrate. Iron in 0.7 gm. of ferrous ammonium sulphate divided by the no. of c.c. soln. used gives the standard of the soln. in gm. per c.c.

SnCl₂

60 gm. SnCl₂ in 600 c.c. of HCl and make up to 1 litre. Add a few pieces of mossy tin.

HgCl₂

60 gm. HgCl₂ in 1000 c.c. water.

Iron in Cobalt Electrolyte

Take soln after electrolysis for cobalt is complete and treat as follows. Filter through No.1 Watman and wash carefully, then wash ppt into a beaker with water. Clean the filter paper with hot 1:1 HCl into same beaker. Wash filter paper with water. Heat to boiling and treat with SnCl₂ as before. If the iron is very low, it is more accurately assayed by filtering through a weightless filter paper, washed and ignited. The iron is weighed as Fe₂O₃.
NICKEL

Weigh .5 gm. ore into 150 c.c. beaker, add 15 c.c. HCl, cover till violent ceases, add 10 c.c. HNO₃ and heat till brown fumes disappear (the acids may be added in the reverse order, if desired). Place on low plate, rinse off cover and take to dryness. Cool, add 10 c.c. HCl and again take to dryness, on a low plate. Bake for 30 minutes at 120 degrees C. Cool, add 10 c.c. HCl, 15 c.c. water and 2 gm. NH₄Cl. Cover and boil till clear. Filter through rapid filter and wash 6 times with boiling water. Add 2 crystals of tartaric acid and 2 - 3 c.c. HNO₃ to the filtrate and warm. Add an excess of NH₄OH till ammoniacal, and solution becomes clear. (Ni is slightly soluble in excess NH₄OH.) Dilute to 250 c.c. with hot H₂O. Put on cover and warm just to the boiling point or the first bubble. Add 25 c.c. of Dimethyl Glyoxime (CH₃)₂C₂(NOH)₂, solution. A red-brown ppt of Co(dmg.) indicates high Co, with much Ni is present a pink ppt of Ni(dmg.) will be formed, with some of the Ni(dmg.) floating on the surface. Allow dmg. soln. to stand for two hours on a low plate. Filter through a rapid filter. Retreat ppt for Ni by washing ppt into 400 c.c. beaker with water and wash filter paper with 20 c.c. of 1:1 HCl. Evaporate soln. to dryness, cool and add 100 c.c. water. Filter out any filter paper and wash with hot water. Make soln ammonical with NH₄OH and heat to the first bubble as before and add 25 c.c. of dmg. If the pink
ppte of Ni(dmg) cannot be seen due to too much Co(dmg), retreat ppte as before, repeating till Ni.ppt is free of Co(dmg). Place on low plate and leave over night. Filter through a tared Gooch filter crucible, wash 5 times with hot water, dry and weigh. Multiply difference by 0.2032 to find weight of nickel.

**Dimethyl Glyoxime**

Dissolve 1 gram dimethyl Glyoxime in 100 c.c. alcohol. 2 1/2 c.c. of 1% dmg. is required for each % of Co+Ni in .5 gm. sample taken.

**Notes:**

Where the amount of Ni. is small and the amount of Co large, it will be hard to get a ppt of Ni(dmg) free of Co(dmg). If the dmg. ppt is retreated several times the ppt can be made fairly free of Co(dmg.)
**SULPHUR**

Ores, Slags, and Roasted Material

Weigh .5 gm. of sample into 150 c.c. beaker, add 40 c.c. water and 30 c.c. nitric chlorate mixture and take to dryness on medium plate. Bake for 10 minutes on hot plate, cool, add 5 c.c. H₂O₂, 2 c.c. HCl and digest. Add excess Na₂CO₃ free from sulphate (0.6 gm. for calcine; 2 gm. for ore) Boil for 10 min. with coverglass on. Filter, after washing off cover into 400 c.c. beaker. Wash ppta once with hot water. Wash ppta into 150 c.c. beaker with water and cleanse filter paper with 1:1 HCl (20 c.c.). Bring to boil with cover on, and add excess of Na₂CO₃ as before. Boil for 10 min. and filter into same 400 c.c. beaker. Wash twice with hot water. Make the combined filtrates acid with HCl and add 2 c.c. excess HCl. Boil solution till all CO₂ is removed. Add hot BaCl₂ slowly (about 15 c.c. of 10% BaCl₂) to the boiling solution, and continue boiling till all the BaSO₄ has settled out. Take beaker containing BaSO₄ from hot plate and allow all the BaSO₄ to settle to the bottom for 2 hours. Filter through #2 Whatman filter and wash 4 times with hot water. Ignite and weigh as BaSO₄. The weight obtained multiplied by .1373 gives the weight of sulphur in the sample.

**10% BaCl₂**

10 gm. BaCl₂ in 100 c.c. of distilled water.
Notes

The filtrate from the pptn. with Na$_2$CO$_3$ must be acid, and should be tested for acidity with litmus paper. If the filtrate is not acid all the CO$_2$ will not be removed. by boiling and an addition of the BaCl$_2$ the solution is liable to foam over and BaCO$_3$ may be formed.

Ignition must be carried out slowly to avoid the reduction of the BaSO$_4$ to sulfide or sulphite by the carbon.
Slag Analysis for SiO₂, Fe and CaO

Weigh out 0.5 gm. into 250 c.c. beaker, moisten with a few drops of water. Add 10 c.c. HCl and warm till clear. Add 10 c.c. chlorate mixture and 10 c.c. 1:1 H₂SO₄ and fume nearly to dryness. Cool and dilute with 25 c.c. water and 5 c.c. HCl and boil.

Filter off the insoluble SiO₂ and wash with hot water. Ignite and weigh.

Dilute the filtrate and wash water to 150 c.c. Make solution basic with NH₄OH and boil for 10 minutes. Filter out the iron precipitate and wash three times with hot water. The filtrate and wash water is combined for the CaO determination. Wash the iron precipitate into a 250 c.c. beaker and cleanse the filter paper with a few c.c. of hot 1:1 HCl. Make the solution acid with HCl and add 2 c.c. in excess. Boil the solution for a minute. While hot add SnCl₂, drop by drop till yellow color (Fe₂Cl₆) goes and add one drop in excess. Cool (to room temperature) and add mercuric chloride, about 20 c.c., to neutralize the excess SnCl₂ and titrate with standard potassium dichromate using K₄Fe(CN)₆ as indicator.

The filtrate from the iron precipitation containing the calcium is boiled and 2 - 3 gm. of ammonium oxalate is added to precipitate the Ca. as calcium oxalate. Boil for 10 minutes and then let it stand for one hour. Filter, wash with water, ignite, and weigh as CaO.
Preparation of Dichromate

335.05 gm. Fe react with 294.2 gm. potassium dichromate. 0.00439 gm. dichromate react with .005 gm. Fe. For 0.5 gm. samples the no. of c.c. dichromate would represent the percent of iron. Dissolve 8.78 gm. $K_2Cr_2O_7$ in two litres of water.

Standardization

Weigh up 0.7 gm. of ferrous ammonium sulphate, acidify with HCl, add one drop SnCl₂, excess mercuric chloride and titrate. Iron in 0.7 gm. of Ferrous ammonium sulphate divided by no. of c.c. of solution used gives the standard of the solution in gm. per c.c.

SnCl₂

60 gm. SnCl₂ in 600 c.c. of HCl and make up to 1 litre. Add a few pieces of mossy tin.

HgCl₂

60 gm. HgCl₂ in 1000 c.c. water.
**Acid (H$_2$SO$_4$) Soluble Iron**

Weigh one gram of calcine into 250 c.c. beaker.

Add 100 c.c. 10% H$_2$SO$_4$ and boil 30 minutes.

Filter and Wash.

Add 5 c.c. HCl to filtrate.

Bring filtrate to a boil and add one small spoonful of granulated lead slowly.

Boil for 30 minutes, and filter into beaker containing 4 drops of stanous chloride.

Add 20 c.c. mercuric chloride when cold.

Titrate with K$_2$Cr$_2$O$_7$ and K$_4$Fe(CN)$_6$ as an indicator.

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**Acid (H$_2$SO$_4$) Soluble Cobalt**

Weigh one gram of calcine into 250 c.c. beaker, add 100 c.c. 10% H$_2$SO$_4$ and boil 30 min. Filter and wash.

Add 5 c.c. 1:1 H$_2$SO$_4$.

Gas for ten minutes cold, bring just to a boil and gas for ten minutes hot with H$_2$S. Cool and filter, wash pptd with water. Boil, H$_2$S off the filtrate and cool.

Add 10 c.c. H$_2$O$_2$ and boil down to a small volume (100 c.c.). Cool and add 2 gm. sodium acetate and when in solution neutralize with ammonia and add 40 c.c. excess NH$_4$OH. Cool.

Plate for 1$\frac{1}{2}$ hr. at 2 amps.

Remove cathode, wash in water and ignite excess alcohol as before. Weigh. Difference in weight is weight of acid soluble cobalt.
Water Soluble Iron

Take 1.0 gm. calcine.
Add 100 c.c. water to calcine in 250 c.c. beaker.
Boil 30 minutes, filter and wash.
Add 5 c.c. HCl to filtrate and bring to a boil, add one small spoonful of granulated lead slowly and boil 30 minutes.
Filter into beaker containing 4 drops of stannous chloride.
Add 20 c.c. mercuric chloride.
Titrate with $K_2Cr_2O_7$ and $K_4Fe(CN)_6$ as indicator.

Water Soluble Cobalt

One gm. calcine in 250 c.c. beaker.
Add 100 c.c. water and boil 30 min.
Filter and wash residue with warm water.
Make filtrate acid with 5 c.c. 1:1 H$_2$SO$_4$.
Gas for ten minutes cold, bring just to a boil and gas 10 minutes hot. Cool and filter, wash ppt$\alpha$ with water. Boil off the H$_2$S from the filtrate and cool. Add 10 c.c. 3% H$_2$O$_2$ and boil off the excess, evaporate to 100 c.c. and cool again.
Add 2 gm. sodium acetate and when in solution neutralize with ammonia. Add 40 c.c. excess NH$_4$OH and cool. Weigh up cathode.
Bring volume of electrolyte up to cover electrode with 30% NH$_4$OH. Plate at 2 amps. for 1½ hr. Remove cathode, wash in water and ignite excess alcohol as before.
Weigh, difference in weight is weight of soluble cobalt.
Fire Assay Charge for Gold

For calcines, sulphated calcines and leached residues.

Charge:

30 gm. Sodium carbonate
60 gm. Litharge
7 gm. Silica
5 gm. Borax
2.3 gm. Flour
.25 A.T. Calcine, etc.
and Silver.
E. Experimental Flow Sheet

1. Ore - 20 Mesh
2. Dead Roast 820°C
3. Agitator
4. Bake 450°C.
5. Salt
6. Pebble Mill
7. Roast 650°C.
8. Water Leach 80°C
9. Filter
10. Filtrate
11. First Iron Precipitation
12. Filter
13. Second Iron Precipitation
14. Filter
15. Filter
16. Filtrate
17. Cobalt Precipitation
18. Filter
19. Cobalt Precipitate
20. Roast 800°C.
21. Cobalt Oxide 70-73% Co.
F. Roasting Furnace

The furnace used was a small industrial annealing furnace made by the Walker Metal Products Co., Canada.

Type F.H. 206
K.W. 13
Volts 220

The tray dimensions are approximately twelve inches wide by eighteen inches long.

The following illustrations indicate the type of furnace and the position of the charge for roasting.

Fig. 3. The furnace door raised, showing the position of the tray and pyrometer.
Fig. 4. The furnace door lowered into position for roasting and rabbling.

Fig. 5. A side view of the furnace showing the relative position of the tray and pyrometer for roasting.
G. Smelting Furnace

The furnace used and illustrated here is a gas fired smelting furnace. The reducing atmosphere required was maintained by using an excess of gas and adjusting the low and high pressure gas valves to give the proper combustion conditions inside the furnace.

Fig. 6. Showing arrangement of preheater and valve controls.

Fig. 7. Showing arrangement of injector and pot.
VII. BIBLIOGRAPHY


