Dr. Convey

[redacted]

CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 61-107

CONCENTRATION OF SCHEELITE FROM ORE OF THE CANADA TUNGSTEN MINING CORPORATION LIMITED, FLAT RIVER, N.W.T.

by

T. F. BERRY

MINERAL PROCESSING DIVISION

NOTE: THIS REPORT RELATES ESSENTIALLY TO THE SAMPLES AS RECEIVED. THE REPORT AND ANY CORRESPONDENCE CONNECTED THEREWITH SHALL NOT BE USED IN FULL OR IN PART AS PUBLICITY OR ADVERTISING MATTER.

COPY NO. 5

SEPTEMBER 26, 1961

Industrial Confidential

Mines Branch Investigation Report IR 61-107

CONCENTRATION OF SCHEELITE FROM ORE OF THE CANADA TUNGSTEN MINING CORPORATION LIMITED, FLAT RIVER, N.W.T.

by

T. F. Berry*

SUMMARY OF RESULTS

Neither flotation in a brine solution as in Test 1, nor the use of Orso and Emcol X-25 as in Test 2, was successful in producing a scheelite concentrate of sufficiently high grade to make the use of these reagents attractive.

In Test 3 a gravity cleaner concentrate assaying 76.96% WO_3 and a flotation concentrate assaying 60.00% WO_3 were produced.

In Test 6 in which an SO_2 leach of an impure scheelite rougher flotation concentrate was employed, followed by three stages of cleaning of the leached concentrate, a high grade final conconcentrate assaying 73.08% WO_3 was obtained.

---

* Technical Officer, Mineral Processing Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

INTRODUCTION

Following the original work done on ore shipments from Canada Tungsten Mining Corporation Ltd., Suite 502 - 1200 West Pender Street, Vancouver 1, B.C., the results of which were reported in Mines Branch Investigation Report IR 60-80, a series of metallurgical tests of an experimental nature was done on several different shipments in an endeavour to outline some new or simplified reagent combination which would float the scheelite.

In addition, the Extraction Metallurgy Division undertook an investigation into the possibilities of leaching impure scheelite flotation concentrates to be followed by the re-flotation of the leached concentrates. The leaching phase of this work has been reported in a separate Mines Branch Investigation Report IR 61-108.

Shipments

The following shipments were received:

October 3, 1960 - approximately 100 lb $\frac{1}{4}$ in. mill feed from Lakefield Research of Canada Limited, Lakefield, Ontario.

October 3, 1960 - one sample bag designated diamond drill hole No. 17, weighing 7.5 lb and representative of 74 ft of core from Canada Tungsten Mining Corporation Ltd.

October 4, 1960 - 125 lb of diamond drill core comprising 17 separate samples from Canada Tungsten Mining Corporation Ltd.

March 27, 1961 - 500 lb of lump ore from Canada Tungsten Mining Corporation Ltd.

Location of the Property

All the above ore shipments were said to have come originally from Canada Tungsten's Flat River property in the Northwest Territories.

Sampling and Analysis*

The company requested that the 18 diamond drill core samples be assayed separately for WO_3, $CaCO_3$, soluble Fe and total S, and that a composite sample for test work be prepared proportional to the amount of each drill core, omitting only holes numbered 26, 37, 38 and 40. The results of the analyses of the drill cores may be seen in the following table.

TABLE 1

Chemical Analysis of Diamond Drill Core Samples

D. D. Hole No.	Intersect Footage	Assays %				Ratio WO_3 : $CaCO_3$
		WO_3	$CaCO_3$	Fe	S	
12	127.9-162.9	1.50	2.50	18.65	9.06	1 : 1.67
13	6.0- 76.0	4.40	1.73	29.13	14.50	1 : 0.39
14	8.5- 50.0	1.08	3.28	23.68	13.66	1 : 3.04
16	35.0-112.0	3.86	2.03	27.45	16.20	1 : 0.52
17	7.0- 81.0	2.83	3.45	10.66	18.86	1 : 1.21
18	86.0-117.0	1.67	4.11	22.42	13.70	1 : 2.46
19	84.0-161.0	2.30	3.10	17.82	9.56	1 : 1.35
20	40.0-120.0	3.50	2.50	25.15	9.90	1 : 0.71
21	7.0- 62.0	2.91	1.67	31.23	16.32	1 : 0.57
22	49.0-145.0	2.29	4.65	25.13	12.57	1 : 2.03
23	85.0-140.0	1.88	3.10	22.21	12.25	1 : 1.65
25	33.0-73.0	2.60	4.65	18.23	9.45	1 : 1.79
26	127.0-153.0	1.87	9.65	19.70	10.66	1 : 5.16
27	128.0-178.0	3.58	1.79	26.20	13.80	1 : 0.50
28	115.5-201.0	2.10	1.55	25.98	12.75	1 : 0.74
37	116.0-166.0	1.59	10.73	14.25	6.32	1 : 6.76
38	171.0-243.0	2.50	7.39	19.07	9.84	1 : 2.96
40	138.0-163.0	1.50	5.36	20.75	9.84	1 : 3.57

---

* The chemical analyses were done by the Analytical Section, Mineral Sciences Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

The composite sample which was prepared had the following calculated analysis:

Tungsten Trioxide (WO_3)	-	2.73	per cent
Calcium Carbonate ($CaCO_3$)	-	2.81	" "
Soluble Iron (Fe)	-	23.39	" "
Total Sulphur (S)	-	13.18	" "

DETAILS OF INVESTIGATION

Preliminary Tests

A series of 8 flotation tests was carried out on ground samples of the composite diamond drill core to determine the amenability of the scheelite to flotation in a brine solution. In these tests an increasing amount of Quebracho was used as a calcite depressant, with distilled oleic acid as the collector and frother. The best result obtained was a scheelite rougher concentrate assaying 35.46% WO_3 representing a recovery of 82.8%.

Test 1

With the same flotation scheme that was used to obtain the above results, the following test was done.

A 14,000 g sample of the composite drill core was stage ground through an 80 M screen. The flotation of the sulphides was carried out on the ground pulp on 7 lots of 2000 g each. Each sulphide rougher concentrate was cleaned once with the cleaner tailing being returned to the succeeding cycle. This recycling applied only to the sulphide flotation section.

The sulphide flotation tailing was tabled to remove a coarse rougher scheelite concentrate which was cleaned once in a Jones high intensity wet magnetic separator operating at 25 amperes.

The table tailing was evenly divided into 4 fractions and the scheelite was floated in a 4% brine solution, made by dissolving rock salt in water. The scheelite rougher concentrates were combined for a cleaning and a recleaning step.

The flotation scheme used and the results which were obtained may be seen in the following tables.

TABLE 2

Sulphide Flotation

Reagents	lb/ton Feed		Time, Min
	Conditioning	Flotation	
$CuSO_4$	0.25		4
Xanthate Z-5	0.10		2
Dowfroth 250		0.04	4
$CuSO_4$	0.15		2
Xanthate Z-5	0.10		1
Dowfroth 250		0.04	4
$CuSO_4$	0.10		2
Xanthate Z-5	0.05		1
Dowfroth 250		0.04	4

Scheelite Flotation

4% brine sol			5
Quebracho	0.5		2
Oleic Acid	0.16	1	2
" "	0.06	1	2
" "	0.08	1	2
" "	0.06	1	2

To the cleaner and recleaner circuits were added 0.05 lb Quebracho per ton and 0.02 lb oleic acid per ton. Flotation time was 3 min in each circuit.

TABLE 3

Results of Test 1

Product	Weight %	Assays % WO_3	Distn % WO_3
Sulphide cl concentrate	39.3	0.34	5.1
" " tailing	0.6	3.18	0.7
Table cl concentrate	1.9	67.28	47.2
" magnetics	1.0	9.07	3.4
WO_3 recl concentrate	1.2	33.81	15.0
" " tailing	0.2	9.87	0.7
" cl "	0.6	2.19	0.5
Flotation "	55.2	1.34	27.4
Head (calcd)	100.0	2.70	100.0

In this test and in others in which a brine solution was used, there was very poor collection (as evidenced by the high tailing) and poor frothing. For these reasons the use of brine was discontinued.

Continuing the search for a reagent combination which might be used to float the scheelite, several tests were carried out using a combination of Orso, a blown neutral soap of vegetable origin, which acts as a promotor and collector for scheelite, and a cationic collector, a salt of a sulfated complex alcohol called Emcol X-25.

The use of these reagents did not produce results which were substantially better than those tests in which the brine was used.

Results typical of those obtained with this reagent combination may be seen in Test 2.

Test 2

In this test flotation of the sulphides proceeded as in Test 1. The sulphide flotation tailing was conditioned with 0.25 lb of Quebracho per ton of original feed for 5 minutes.

Orso and Emcol X-25 were each stage added in the amount of 0.25 lb per ton. Flotation proceeded for 8 minutes. To the cleaner and recleaner circuit 0.1 lb NaCN per ton of feed was added, followed by 2 minutes of flotation. The results obtained were as follows:

TABLE 4

Results of Test 2

Product	Weight %	Assays % WO_3	Distn % WO_3
Sulphide cl concentrate	40.6	0.26	4.1
" " tailing	3.9	3.91	5.9
WO_3 recl concentrate	3.8	43.60	63.7
" " tailing	0.7	12.47	3.4
" cl "	2.9	4.54	5.1
Flotation "	48.1	0.97	17.8
Head (calcd)	100.0	2.60	100.0

Test 3

A 16,000 g sample of mill feed obtained from Lakefield Research of Canada Limited, Lakefield, Ontario, was stage ground to -80 M in lots of 2000 g each and the sulphides were floated using the same procedure outlined in Test 1.

The sulphide flotation tailing was jigged and the jig tailing was tabled. The table concentrate was cleaned in a Jones high intensity wet magnetic separator operated at 25 amperes. The table tailing was wet split into 8 equal fractions which were floated according to the following scheme. The 8 rougher flotation concentrates were combined for cleaning.

In Table 6 in which the results of this test are summarized, the jig concentrate, which assayed 76.36% WO_3 and represented only 3.60% of the total scheelite, was combined with the cleaned table concentrate.

TABLE 5

Scheelite Flotation

Distilled water was used throughout the entire flotation.

Conditioning - 4 min

Na_2CO_3 - to pH 9.6
NaCN - 0.5 lb/ton
Na_2SiO_3 - 0.3 " "
Quebracho - 0.4 " "

Rougher Flotation

Sodium Oleate - 0.3 lb/ton stage added
Distilled oleic acid - 0.1 " " " "
Methyl alcohol - 0.06 " " " " as auxiliary frother

Conditioning time - 4 min
Flotation time - 8 "

1st and 2nd Cleaner Flotation

NaCN - 0.1 lb/ton
Na_2SiO_3 - 0.06 " "
Quebracho- 0.1 " "
Conditioning time - 2 min
Flotation time - 4 "

3rd and 4th Cleaner Flotation

NaCN	-	0.1 lb/ton
Na_2SiO_3	-	0.06 " "
Conditioning time	-	2 min
Flotation time	-	3 "

The results of this test were as follows:

TABLE 6

Results of Test 3

Product	Weight %	Assays % WO_3	Distn % WO_3
Sulphide cl concentrate	30.4	0.41	5.8
" " tailing	1.1	1.46	0.8
Table + jig concentrate (non-magnetics)	1.6	76.96	57.9
Table + jig concentrate (magnetics)	2.0	6.77	6.4
Final WO_3 Flotation concentrate	0.6	60.00	16.9
Flotation cl tailing No.4	0.05	18.14	0.4
" " " " 3	0.05	7.80	0.2
" " " " 2	0.2	1.01	0.1
" " " " 1	0.9	1.96	0.8
Flotation rougher tailing	63.1	0.36	10.7
Head (calcd)	100.0	2.13	100.0

Test 4

An 8000 g sample, from the shipment of lump ore received from the property on March 27, was stage ground to -80 M. The sulphides were floated as in Test 1 and the entire flotation tailing was sent to the Extraction Metallurgy Division for leaching with sulphur dioxide. The leached sulphide flotation tailing was returned to the Mineral Processing Division for scheelite flotation.

After washing the pulp to reach a pH of 6.8, Na_2CO_3 was added until the pH was 9.6. Conditioning was carried out in distilled water at 50% solids in a 500 gram flotation cell for 10 min with the following reagents added:

NaCN	-	0.5 lb/ton feed
Na_2SiO_3	-	1.0 " " "
Sodium Oleate	-	0.2 " " "
Oleic acid (Hardesty No.4)	-	0.5 " " "

The pulp was transferred to a 2000 g cell and floated for 6 min at approximately 25% solids in a 1:1 distilled-tap water mixture.

The scheelite rougher concentrate was cleaned for 2 min with the following reagents added:

NaCN	-	0.3 lb/ton feed
Na_2SiO_3	-	0.2 " " "

Additional cleaning steps were not possible because of the small amount of material available.

The results of this test were as follows:

TABLE 7

Results of Test 4

Product	Weight %	Assays % WO_3	Distn % WO_3
Sulphide concentrate	28.9	0.28	5.8
WO_3 cl "	1.5	58.84	63.4
" " tailing	4.6	4.23	14.0
Flotation "	65.0	0.36	16.8
Head (calcd)	100.0	1.39	100.0

The leaching phase of Test 4 may be seen in Mines Branch Investigation Report IR 61-108 under Test 809.

Test 5

This test was similar to Test 4 with the exception that instead of using sulphide flotation, the ground pulp was passed through a Jones high intensity wet magnetic separator operated at 25 amperes to eliminate the sulphides and other weakly magnetic minerals.

Leaching of the non-magnetic fraction from the separator with SO_2 was identical to the procedure followed in Test 4 as was the subsequent washing of the leached pulp and the scheelite flotation. One extra cleaning stage was, however, added in this test. The same reagents were used in this cleaning stage as in the first stage.

The results of this test were as follows:

TABLE 8

Results of Test 5

Product	Weight %	Assays % WO_3	Distn % WO_3
Jones magnetics	65.5	0.36	12.9
WO_3 recl concentrate	2.4	59.10	77.4
" " tailing	0.1	27.00	1.5
" cl "	0.6	44.00	1.4
Flotation "	31.4	0.40	6.8
Head (calcd)	100.0	1.83	100.0

Test 6

A 12,000 g sample of ore, from the March 27 shipment, was ground in lots of 2000 g each to all -100 M. The sulphides were floated with no cleaning stage using the flotation scheme outlined in Test 1.

Scheelite flotation was then carried out on each of the 6 sulphide flotation tailing fractions. Each fraction was conditioned for 10 min at 50% solids in a 1:1 distilled-tap water mixture with the following reagents:

Na_2CO_3 - to pH 9.7
NaCN - 0.5 lb/ton feed
Na_2SiO_3 - 1.0 " " "

To the conditioned pulp was added 0.22 lb sodium oleate/ton and 0.06 lb oleic acid (Hardesty No. 4)/ton and conditioning was continued for 2 min. The pulp was then diluted to approximately 25% solids using the same distilled-tap water mixture used in conditioning and the scheelite was floated for 6 min. A small amount of methyl alcohol was added as an auxiliary frother to produce a fine lacy froth.

The 6 scheelite rougher flotation concentrates were combined and cleaned once for 4 min with 0.5 lb Na_2SiO_3/ton added.

The results of this stage of Test 6 were as follows:

TABLE 9

Results of Test 6 - Stage 1

Product	Weight %	Assays % WO_3	Distn % WO_3
Sulphide concentrate	36.9	0.43	9.9
WO_3 cl "	5.5	23.31	80.2
" " tailing	4.4	0.47	1.3
Flotation "	53.2	0.26	8.6
Head (calcd)	100.0	1.60	100.0

The WO_3 cleaner concentrate weighing 656.0 g was sent to the Extraction Metallurgy Division for calcite elimination by SO_2 leaching. This work may be seen in Mines Branch Investigation Report IR 61-108 under Test 812.

The leached WO_3 cleaner concentrate weighing 492.2 g was returned to the Mineral Processing Division for reflotation of the scheelite. The pulp was washed to a pH of 6.2.

During the three recleaner stages the same mixture of distilled-tap water was used as in Stage 1 of this test.

The pulp was conditioned in the 1st recleaner stage for 10 min with the following reagents:

Na_2CO_3	-	to pH 9.5
NaCN	-	0.5 lb/ton leached concentrate
Na_2SiO_3	-	1.0 " " " "

The flotation time was 5 min. The 2nd and 3rd recleaner stages were as follows:

2nd recleaner stage	-	NaCN	-	0.1 lb/ton original leached concentrate
3rd " "	-	"	-	0.05 lb/ton original leached concentrate

The flotation time in each of these recleaner stages was 3 min.

The results of this stage may be seen as follows:

TABLE 10

Results of Test 6 - Stage 2

Product	Weight %	Assays % WO_3	Distn % WO_3
Final WO_3 concentrate	33.8	73.08	77.1
3rd recl tailing	4.1	66.91	8.6
2nd " "	6.9	36.79	7.9
1st " "	55.2	3.72	6.4
Head (calcd)	100.0	32.04	100.0

A complete summary of Test 6, taking into consideration the weight loss due to the SO_2 leaching may be seen in Table 11.

TABLE 11

Summary of Results of Test 6

Product	Weight %	Assays % WO_3	Distn % WO_3
Sulphide concentrate	36.9	0.43	7.8
Final WO_3 "	1.8	73.08	64.9
3rd recl tailing	0.2	66.91	6.6
2nd " "	0.4	36.79	7.3
1st " "	3.0	3.72	5.5
Cleaner "	4.4	0.47	1.0
Flotation "	53.3	0.26	6.9
Head (calcd)	100.0	2.03	100.0

Combining the final WO_3 concentrate and the 3rd recleaner tailing results in a concentrate assaying 72.46% WO_3 with a recovery of 71.5%. The addition of the 2nd recleaner tailing to this combined concentrate gives a concentrate assaying 66.5% WO_3 with a recovery of 78.8%.

CONCLUSIONS

Jigging of the ground pulp for the recovery of the coarse scheelite is not a satisfactory substitute for tabling. For example, in Test 3 a jig concentrate after cleaning with a magnetic separator assayed 76.36% WO_3 but contained only 3.6% of the total WO_3 content. In contrast when the jig tailing was tabled and the rougher table concentrate was cleaned by magnetic separation, a cleaner concentrate assaying 76.96% WO_3 was obtained representing an additional recovery of 54.3% of the WO_3.

Of the many tests which were done in which NaCl was used as a conditioning reagent for the scheelite only one is reported. In Test 1 there was very poor collection as evidenced by the high tailing. The maintenance of a stable froth was difficult particularly in the cleaning of the rougher scheelite concentrate. The use of brine in the flotation of the scheelite does not appear feasible.

In Test 2 in which Orso and Emcol X-25 were used slightly better results were obtained than in Test 1. These results were not sufficiently satisfactory to warrant further work, particularly in view of the much better results obtained in Test 3 with the use of sodium oleate and distilled oleic acid and in those tests in which a leach of SO_2 was employed.

The SO_2 leaching of pulps and rougher flotation concentrates has been discussed in a separate Mines Branch report. As far as the flotation of the scheelite is concerned, the use of SO_2 appears to offer some advantages. Less care need be taken with regard to the depression of gangue minerals, particularly calcite, since this mineral can be completely eliminated during leaching, thus eliminating the use of a calcite depressant such as Quebracho. Sodium silicate and cyanide can be used to good effect to depress silica and the iron minerals during the cleaning of the leached scheelite rougher concentrate.

With the large amount of pyrrhotite available in the ore, the installation of a sulphur burner to generate SO_2 for leaching could be augmented by a waste heat exchanger to aid in the heating of mill buildings.

TFB:EBM