

Declassified 4/3/81

Director, Mines Branch

~~CONFIDENTIAL~~

This document was produced by scanning the original publication.
Ce document est le produit d'une numérisation par balayage de la publication originale.

CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 59-17

PILOT PLANT TESTS ON A NIOBIUM ORE FROM NOVA BEAUCAGE MINES LIMITED, NORTH BAY, ONTARIO

by

D. E. PICKETT

MINERAL DRESSING AND PROCESS METALLURGY DIVISION

NOTE: THIS REPORT RELATES ESSENTIALLY TO THE SAMPLES AS RECEIVED. IT SHALL NOT, NOR ANY CORRESPONDENCE CONNECTED THEREWITH, BE USED IN PART OR IN FULL AS PUBLICITY OR ADVERTISING MATTER.

DECLASSIFIED
DATE *4/3/81*
AUTHORIZED BY *[Signature]*

01-2989730

IR 59-17

Declassified
Déclassifié

~~Confidential~~

Mines Branch Investigation Report IR 59-17

PILOT PLANT TESTS ON A NIOBIUM ORE FROM
NOVA BEAUCAGE MINES LIMITED, NORTH BAY, ONTARIO

by

D. E. Pickett^{*}

- - - -

SUMMARY OF RESULTS

Flotation, sink-float, tabling and magnetic separation tests were carried out on a niobium ore from Nova Beaucage Mines containing approximately 0.75% Nb₂O₅ as pyrochlore.

Flotation concentrates containing up to 18% Nb₂O₅ were produced at recoveries up to 60%. Observations indicate that better recovery is possible.

The flotation process was proven to be a practical method of recovering pyrochlore concentrate from the ore. The economic limitations of the overall niobium production from the Beaucage ore can only be determined after more test work and a study of chemical extraction problems.

Preliminary testing indicated that sink-float would be valuable as a pre-concentration process. Tabling and magnetic concentration appear to have doubtful value.

^{*}Senior Scientific Officer, Mineral Dressing and Process Metallurgy Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

CONTENTS

	<u>Page</u>
Summary of Results	i
Introduction	1
Purpose of Tests	3
Ore Shipments and Sampling	4
Assaying	4
Laboratory Tests	4
A - Sink-Float Tests	4
B - Laboratory Flotation	5
Pilot Plant Tests	6
A - Pilot Plant Table Concentration	6
B - Magnetic Separation Tests	9
C - Pilot Plant Flotation	10
Discussion of Results	15
Appendices	17

= =

(33 pages, 2 figures, 8 tables)

INTRODUCTION

The Beaucage niobium deposit on Lake Nipissing, near North Bay, Ontario, is one of the pyrochlore ores which form the niobium resources of Canada. The ore is a complex calcite-silicate type containing pyrochlore as well as sulphides, magnetite, mica, etc. The mineralization has been described in detail in Mines Branch Report of Investigation No. MD3126 by E. H. Nickel.

In 1955 Beaucage Mines Limited, then a subsidiary of Inspiration Mining and Development Company Limited, North Bay, built a 50 ton per day pilot plant at North Bay to produce niobium, based on a process developed by Battelle Memorial Institute, Columbus, Ohio. Mine development was carried out to explore the underground deposit and to obtain a stock pile of ore for the pilot plant. This plant was visited by Mines Branch officers in November, 1955, and the mine and plant are described in Visit Memorandum No. 631 by E. H. Nickel and D. E. Pickett.

Briefly, the process was an attempt to chlorinate the niobium after the interfering calcite and sulphides had been removed by fatty-acid flotation. The pilot plant was operated intermittently into 1957 and was finally closed down pending further research work. During these operations a good stockpile of ore was built up at the pilot plant.

In 1957 Consolidated Mining and Smelting Company of Canada (Cominco) acquired an interest in Beaucage Mines. The Cominco Research Department undertook the development of a new process for treatment of the ore and considerable test work was carried out at Trail and Kimberley, British Columbia. High tension,

electrostatic, and magnetic tests were conducted by Mr. C. H. Bushel of the Cominco research staff at the Mines Branch laboratories in Ottawa.

As a result of the flotation research at Trail and Kimberley, a new flotation process was developed for concentration of the pyrochlore from the Beaucage ore. This process gave good results in laboratory flotation. About the same time, Mr. H. W. Poole, consulting metallurgist for Beaucage Mines, working at the Cominco Kimberley laboratories, developed a pre-concentration gravity process which promised to improve the economics of the overall process. It was then decided to make a pilot plant run as the next stage in the process development. Since the equipment at the Beaucage plant was not suited to the scale of pilot run planned, a request was made to the Mines Branch for the use of the Mines Branch pilot plant facilities. Permission was granted for the Beaucage and Cominco staff to make pilot plant tests in Ottawa about October 1, 1958, and plans were made accordingly.

Detailed plans and preparation for the tests were completed by Mr. R. Woodford, Manager of Beaucage Mines, and Mr. L. E. Djinghezian of the Mines Branch in correspondence dated September 10 and September 17, 1958. The first shipment of ore arrived from North Bay on September 29. Upon the arrival of the Beaucage and Cominco personnel, work commenced on the project on October 2 and the first gravity tests were started on October 6. The final flotation test was made on November 24 with assaying completed on November 26. A 200 lb sample of crushed ore has been retained at the Mines Branch in case future work is requested by Beaucage Mines.

On November 18, 1958 the company was reorganized under the new name of Nova Beaucage Mines.

PURPOSE OF TESTS

The pilot plant tests were designed to obtain information about the process which could not be obtained in laboratory work.

(1) Sink-Float Tests: Although it is generally agreed that laboratory sink-float tests can be scaled up to a production size plant with very little change in metallurgical results, it was felt that pilot plant tests should be made at the Mines Branch on a larger sample than was available at Kimberley, particularly as the sample, cut from a large shipment, would be more representative of the Beaucage ore. However, as facilities for carrying out continuous pilot plant tests were not available, these tests were limited to one series of laboratory tests to check the reliability of the sample.

(2) Tabling Tests: Laboratory tabling tests carried out at Kimberley had indicated that this method might give the same pre-concentration as sink-float. It was decided that tests should be run on the full scale equipment at the Mines Branch to confirm this result, and to obtain concentrates which might be used for flotation tests.

(3) Flotation Tests: Some doubt existed as to the behaviour of one of the reagents which was suspected to be unstable chemically under the oxidizing conditions existing in the flotation circuit. Therefore, it was not known whether the cleaner tailings (and solutions) could be recycled. Also information was desired about circuit control, flotation rates, grades of concentrate obtainable, and the effect of a continuous closed circuit grinding stage.

ORE SHIPMENTS AND SAMPLING

Four shipments of ore were received by truck from North Bay during the test. The head assays are given below:

<u>Shipment No.</u>	<u>Nb₂O₅ %</u>	<u>U₃O₈ %</u>
1	0.73	0.05
2	0.69	--
3	0.81	--
4	0.62	--

The first shipment was crushed to minus 1 in. and sampled to get a feed sample for sink-float tests. This sample was further reduced by Jones splitter and crushed to minus 4 mesh to obtain a standard assay head sample. The other shipments were crushed to minus 4 mesh before sampling. All the ore except that used for the sink-float tests was reduced to minus 4 mesh for ball mill or rod mill feed.

ASSAYING

Assaying for Nb₂O₅ was carried out in the Mines Branch chemical laboratories by the calorimetric method as described by G. H. Faye, in Chemistry in Canada, Vol. 10, No. 4, p. 90, April, 1958.

LABORATORY TESTS

A. - Sink-Float Tests

These tests were carried out by Mines Branch personnel under the direction of Mr. H. W. Poole of Beaucage. A sample of minus 1 in. Symons crusher product from No. 1 shipment was wet screened at 10 mesh to remove the fines and then tested by standard "bucket test" using galena medium with a specific gravity of 2.95. The float product from this test was retreated at a medium specific

gravity of 2.85. This procedure gave four products:- sink products at s.g. 2.95 and 2.85, a float product at s.g. 2.85, and the minus 10 mesh fines. Each product was dried and separated into four screen fractions. The results are tabulated in Tables 1 and 2 attached as appendices to this report which show the distribution of niobium. At the medium specific gravity of 2.85 approximately 30% of the weight was discarded with a loss of only 8.2% of the niobium. The 15% of the feed which floated at s.g. 2.95 and sank at 2.85 was essentially of feed grade so the lower medium density would be preferable.

Table 2 which gives the distributions of niobium in the screen fractions shows poor recovery from the minus 6 plus 10 mesh fraction. It seems that this screen fraction is much lower in niobium content than either the coarser or finer material. This may be due to the nature of the ore or to the crushing method. Fine preparation screens may be warranted but this can only be decided in full scale continuous operation where maintenance costs can be evaluated.

B. - Laboratory Flotation at Mines Branch

Several laboratory tests were carried out on samples of mill feed to compare the behaviour of the pilot plant sample with the sample used at Trail and Kimberley. These tests were performed by Mr. G. Moody, Cominco technician.

The standard laboratory test procedure is as follows:-
a 500 g sample of feed is ground to 90% minus 200 mesh and conditioned in a 500 g cell for 5 min with 2 g NaOH at a pH of 12 or higher. For flotation 0.25 g Duomac T (a tallow fatty diamine diacetate) is added along with 3 drops of methyl isobutyl carbinol. The froth is bulky and well laden with slime sized calcite, mica and sulphides. After

15 min flotation, an additional 0.12 g Duomac T is added and after another 10 min, 0.10 g Duomac T is added. The froth carries coarser calcite and mica toward the end of the float and this "calcite float" is terminated at 30 min. The conditioning and flotation timing appears to be critical if the next step of the float is to be controlled. For the next step, the "pyrochlore float", 2.5 g of $MgSiF_6$ is added which reduces the pH to about 5. After 1 min conditioning, 0.5 g "catechol" is added and the pulp is conditioned for 5 min. A rougher concentrate is then floated and cleaned once with an additional 0.5 g catechol. The pH of the rougher tailing is about 7.0. Typical results follow:

<u>Product</u>	Assay Nb_2O_5 %	
	<u>Test (1)</u>	<u>Test (3)</u>
"Calcite" float	0.33	0.31
Pyrochlore rougher tailing	0.18	0.13
Pyrochlore cleaner tailing	1.44	0.40
Final concentrate	23.30	13.55

Comparing these results with those of the pilot run continuous tests show that recoveries and concentrate grades were lower in the pilot plant. Some of this difference can be attributed to the much better control of froth and reagent concentration in the laboratory batch cell. Some of the poorer results obtained in the pilot plant may be due to the recirculation of the middling products which eventually dropped the concentrate grade or increased the tailing losses.

PILOT PLANT TESTS

A. - Pilot Plant Table Concentration

Since sink-float equipment was not available, gravity

pre-concentration was carried out on the Wilfley and Deister tables, with grinding in the rod mill in closed circuit with screens.

In the basic flowsheet, ore at minus 4 mesh was fed by constant-weight feeder at the rate of 800 to 900 lb per hr to the 36 in. by 72 in. rod mill. The rod mill discharge was elevated to a 4 by 4 ft model V7 Tyler Hummer unit with a 50 mesh screen. The screen oversize was returned to the mill. The minus 50 mesh screen undersize was fed to a V16 Tyler Hummer unit with an 80 mesh screen. The screen oversize was treated on a Deister diagonal deck table. The screen undersize was pumped to a hydro-cyclone where it was deslimed before treatment on a full-size Wilfley table.

Screen analyses of the rod mill and screen products are given below:

Tyler mesh	Rod Mill feed	V7 Screen product	V16 Screen	
	%		O'size %	U'size %
+14	0.2	-	-	-
- 14+20	0.3	-	-	-
- 20+48	12.6	0.8	0.9	-
- 48+65	13.3	10.7	18.7	-
- 65+100	13.8	16.2	30.6	1.0
-100+150	11.2	13.6	18.2	8.7
-150+200	9.0	11.2	8.2	13.7
-200+325	39.6	47.5	5.3	19.2
-325			17.6	57.4
	100.0	100.0	100.0	100.0

The 50 mm ceramic Dorr hydro-cyclone produced a slime product (overflow) which was 99.6% minus 325 mesh, at the rate of approximately 105 lb/hr. The overflow of the Type M Dorrclone was 87.9% minus 325 mesh.

To investigate a range of operating conditions this basic flowsheet was varied as shown below:

<u>Tabling Test No.</u>	<u>Conditions of Test</u>
T1	Not deslimed; +80 Hummer product to coarse (Deister) table; -80 m to fine (Wilfley) table.
T2	Deslimed in M30 Dorrclone, otherwise as T1.
T3	Deslimed in 50 mm ceramic hydro-cyclone; coarse table concentrate and middling reground to -80 m and recycled to fine table.
T4	Deslimed as in T3. All +80 m Hummer product reground to -80 m before treatment on the Deister table. Table middlings recycled. Concentrate bands cut at 6 in. and 4½ in. on Wilfley and Deister table respectively.
T5	As T4 but 12 in. concentrate bands cut on both tables.

The regrind unit used in Tests T3 to T5 was a small ball mill in closed circuit with a Sweco 80 mesh screen.

A series of tests was made on the Wilfley table treating the classifier overflow product from the flotation pilot plant grinding unit. This feed was almost all minus 100 mesh. The best concentrate contained 2.2% Nb₂O₅ but the recovery was only 55%. The cleanest tailing contained 0.45% Nb₂O₅, so losses would be too high in this treatment. The results obtained are shown in Tables 3 and 4 attached as appendices to this report.

From the tables it is apparent that tabling would not give as good pre-concentration as sink-float. The best tailing assays

were greater than one-half of the feed assay so recovery would not be acceptable. The slime losses were high when desliming was used, as this product usually assayed almost as high as the feed and amounted to more than 10% of the weight. Concentrate grades were about 2% Nb₂O₅, slightly better than the sink-float concentrates, but at a lower recovery. Any table concentrate would require flotation treatment or other method of beneficiation. Magnetic beneficiation was tried on these concentrates and is described below.

B. - Magnetic Separation Tests

High intensity magnetic separation tests were carried out on the coarse and fine table concentrates from the gravity pilot plant tests. The results on a plus 80 mesh table concentrate using a Dings three-roll separator are given below. The "magnetite" concentrate was obtained by a preliminary pass through a low-intensity belt-type separator. The "middling" product is the non-magnetic tailing obtained by retreatment of the combined concentrates from the first pass through the Dings separator.

Magnetic Concentration of Table Concentrate

Product	Weight, %	Assay, % Nb ₂ O ₅	Distribution, % Nb ₂ O ₅
Magnetite conc.	4.6	0.33	1.0
Top roll conc.	20.8	0.90	12.7
Middle roll conc.	36.7	0.94	23.5
Bottom roll conc.	11.9	1.31	10.6
Middling	6.7	3.00	13.6
Non-magnetics	19.3	2.95	38.6
Calculated feed	100.0	1.41	100.0

A similar test procedure on the minus 80 mesh table concentrate, gave the results shown in the following table.

Magnetic Treatment of Fine Table Concentrate

Product	Weight, %	Assay, % Nb ₂ O ₅	Distribution, % Nb ₂ O ₅
Magnetite conc.	1.5	0.57	0.3
Top roll conc.	28.6	2.15	17.8
Middle roll conc.	42.8	2.68	33.2
Bottom roll conc.	6.4	6.15	11.4
Middling	1.7	7.55	3.8
Non-magnetics	19.0	6.10	33.5
Calculated feed	100.0	3.45	100.0

It appears that some of the pyrochlore is more magnetic than the rest, possibly due to small inclusions of magnetite in the grains. Therefore, although the non-magnetic products are of good grade, no magnetic products are made which can be discarded and the recovery of niobium is low. Most of the pyroxene reported in the top and middle roll concentrates. The calcite is mainly in the non-magnetic tailing.

C. - Pilot Plant Flotation

Ore

The pilot plant flotation tests were carried out on ore which had not been pre-concentrated. The feed varied considerably since four lots of ore were mixed at some stages of the test.

Crushing

On arrival, each shipment was crushed to minus 4 mesh in the jaw and gyratory crushers with the fine crusher in closed circuit with a screen.

Grinding

The grinding circuit, fed by a constant-weight feeder at approximately 420 lb/hr, consisted of a Denver 30" x 36" ball mill in closed circuit with a Dorr Duplex 8 ft classifier. With the classifier overflow density of 25% solids, the screen analyses of the flotation feeds in five typical test runs were as shown in the following table:

Test Run	#117	169	220	337	349
Tyler Screen mesh	Percent weight				
+65	0.4	0.2	0.2	0.3	0.3
- 65+100	0.8	0.6	0.4	0.7	1.2
-100+150	3.2	3.1	2.0	2.8	4.7
-150+200	9.3	9.8	7.2	7.9	10.1
-200+325	18.8	20.3	17.3	18.3	19.0
-325	67.5	66.0	72.9	70.0	64.7
	100.0	100.0	100.0	100.0	100.0

Conditioning

Sodium hydroxide was fed to the classifier overflow which was then conditioned in a Denver conditioner for approximately 5 min. Collector (Duomac T) was added to the first flotation cell for a short conditioning period. The pH of the pulp at this stage was approximately 12.

Flotation Procedure

The flotation circuit as finally developed after many changes is shown in Figure 1. Initially only the first 10 cell bank of No. 7 Denver cells was used for the first stage or calcite float with the floated calcite product discarded to tailings. Frother and Duomac were staged to this circuit. The "calcite flotation" tailing was

conditioned with magnesium fluosilicate at a pH of about 5 in the pump and in the first cell of another 10 cell bank. The modifying agent, catechol, was added to the second cell and a rougher pyrochlore float was made in the next six cells. The rougher tailing was discarded as "final pyrochlore tailing" and the rougher concentrate was cleaned twice with additional catechol in No. 5 Denver cells, with cleaner tailings going to waste.

This process was modified progressively as experience was obtained. The final flowsheet in Figure 1 included an enlarged rougher circuit, a calcite float retreatment stage, four stages of pyrochlore concentrate cleaning and a scavenger float on the final tailing, with all middling products recycled.

Reagent concentration control was very important both to control the selectivity and to control the froth. Duomac was staged to the calcite float at the 1st, 6th, 10th and 14th cells. Duomac was also added to the scavenger and pyrochlore circuits. After the initial conditioning with magnesium fluosilicate, catechol was staged to the pyrochlore rougher cell feed, and to the recleaner stages. Although the Duomac staging controlled the froth to some extent, methyl isobutyl carbinol frother was staged to the circuits. The final froth control was obtained by throttling the inter-cell flow passages to reduce the air supply. Lack of a positive method for controlling the air was the cause of much operating difficulty and erratic results.

During the testing program, 41 test runs were made in the pilot flotation circuit. The reagent additions and operating conditions for each test are shown in Table 5 attached as an Appendix to this report. For each test the desired operating and reagent changes were made and

the circuit was operated until conditions were stable and recirculating loads reached an apparent equilibrium. This normally took from two to four hours depending on the point of circuit change and the difficulties met in readjusting the air supply. Samples of the products were then taken at 5 min intervals for one-half hour or more. In addition to samples of the three final products, other samples of stage products were taken as desired. A classifier overflow sample was taken for each test. This did not always agree with the calculated feed for a test run indicating imbalance in the circuit.

Controls

Reagents were fed in water solution with Clarkson feeders;- the Duomac T at 2%, and the other reagents at 10% by weight. An attempt was made to feed the magnesium fluosilicate by automatic pH recorder to maintain a constant pH in the pyrochlore float circuit but this was abandoned due to mechanical difficulties. Otherwise, reagent control was manual. Pulp density was established at 25% solids in the classifier overflow and no attempt was made to control densities in the pyrochlore float circuit.

During the tests grab samples were taken of the stage products for control purposes. These were dried and rapidly checked for radioactivity by Dr. B. Ositis, an analyst of Cominco. A direct relationship had been established between the radioactivity of a sample of standard size and its pyrochlore content. By this method a rapid check was obtained on flotation circuit performance.

Flotation Results

The results of the 41 tests are summarized graphically in Figure 2 appended to this report. Details of three typical tests

are given in Tables 6, 7 and 8. Table 6 gives the details of Test F11 during which no products were recycled. Table 7 gives details of Test F28 in which the pyrochlore concentrate was cleaned four times and the middlings were recycled. Table 8 gives the details of Test F41, the last of the series, for which Figure 1 is the flowsheet.

Discussion of Flotation Results

The results in Figure 2 appear to be very erratic. There are three main causes for this:

1) A great deal of the variability, particularly in the earlier tests, was caused by the difficulty of reproducing operating conditions in successive tests. As the collector in the calcite float was also a frother, control of air volume was the only way to control the froth.

2) Each lot of feed was different and this affected the uniformity of results. From the graph it is apparent that the good concentrate grades often accompanied increases in feed grade.

3) In many cases, however, particularly in the last tests the erratic results were the result of deliberate changes in reagents or operating conditions. The points at which the collectors were staged were very critical and many of the erratic results were caused by experiments to determine the right points for Duomac or catechol addition. As an example of the result of changing reagent concentration, the drop in recovery from 54.4% in Test F25 to 34.4% in Test F26 was caused by a change in total catechol to the cleaner circuit from 3.53 to 2.27 lb/ton. Similarly the increase in recovery and drop in concentrate grade from Test F18 to Test F19 can be attributed to an increase in Duomac at the last addition point from

0.24 to 0.43 lb/ton.

Control of the process was eventually established by experience in operation and by changes in the circuit. Retreatment of the calcite float product starting with Test F16 helped to stabilize the calcite float weight and grade. Recycling of the pyrochlore cleaning stage tailings starting with Test F18 helped to control the pyrochlore recovery, and concentrate grade. The effect of addition of two extra cleaning stages in Tests F23 and F27 is shown by the uniform recovery (at good grade) from Test F28 to Test F41. During these tests the final tailing grade was steadily lowered and this improvement mainly resulted from the addition of the scavenger circuit. This also provided a point of addition for extra Duomac to control the flotation rate in the rougher cells. Also effective was the change in catechol addition to the final cleaner stage. In addition to better products and recovery, these changes allowed a big reduction in reagent consumption from approximately 5 lb/ton of catechol in Test F28 to 2.4 lb/ton in the last tests.

DISCUSSION OF RESULTS

Although preliminary testing indicated that pre-concentration by sink-float would be valuable, its economic importance can be better evaluated after pilot plant tests. It appears doubtful if tabling or magnetic concentration can make much improvement in the process. Magnetic tests on a high intensity pilot plant separator gave little concentration of niobium. Tabling tests on full size tables gave low grade concentrates and results indicated that pre-concentration would not be as good as with sink-float.

A great deal of useful information and experience was

obtained from the flotation tests. The pilot run proved that the laboratory batch process could be carried out successfully in a continuous process. The catechol reagent was not affected adversely by decomposition in the recycled solutions and, if anything the selectivity was improved. The process could be varied with good control to give a range of concentrate grades up to 18% Nb₂O₅. Changes in the tenor of the ore did not affect operation other than to affect the concentrate grade slightly. Additional cleaning stages might be necessary for lower grade ore.

While the recovery of pyrochlore obtained was low (about 60%), it was estimated that with refinement of operating controls and more stable conditions in a large mill the expected recovery would be close to 70%. More testing could have been carried out in the small pilot plant to obtain a better recovery. However, it was decided that operating conditions, equipment design, and operating technique had such a large effect on the results that further test work should be carried out in plant size equipment. Froth control and reagent control would be more effective.

Some of the results indicated the necessity of doing more laboratory testing. In particular, some effort should be made to reduce the amount and cost of reagents.

- - -

DEP/DV

APPENDICES

Tables

1. Sink-Float -- Distribution of Niobium in Products
2. Sink-Float -- Distribution of Niobium in Screen Fractions
3. Tabling
Tests -- Coarse and Fine Tables, Tests T1 and T2
4. Tabling
Tests -- with Grinding to -80 mesh, Tests T3, T4 and T5
5. Flotation -- Log of Reagent Additions and Operating Changes
6. Flotation -- Details of Test F11
7. Flotation -- Details of Test F28
8. Flotation -- Details of Test F41

Figures

Figure 1 - Flotation Pilot Plant Flowsheet

Figure 2 - Results of Pilot Plant Flotation

TABLE 1

Sink-Float Test - Distribution of Niobium in Products

Medium Specific Gravity	Product	Screen Fraction, Tyler	Weight, %	Assay, % Nb ₂ O ₅	Distribution, % Nb ₂ O ₅
2.95	Sink	+ $\frac{1}{2}$ in.	12.1	1.30	18.8
		- $\frac{1}{2}$ + $\frac{1}{4}$ in.	18.9	1.45	32.7
		- $\frac{1}{4}$ in.+6 m	2.7	1.43	4.6
		-6+10 m	<u>0.4</u>	<u>1.38</u>	<u>0.7</u>
			34.1	1.40	56.8
2.85	Sink	+ $\frac{1}{2}$ in.	2.9	0.44	1.6
		- $\frac{1}{2}$ + $\frac{1}{4}$ in.	8.2	0.72	7.0
		- $\frac{1}{4}$ in.+6 m	3.1	1.11	3.9
		-6+10 m	<u>0.7</u>	<u>0.93</u>	<u>0.7</u>
			14.9	0.75	13.2
2.85	Float	+ $\frac{1}{2}$ in.	4.5	0.13	0.7
		- $\frac{1}{2}$ + $\frac{1}{4}$ in.	11.1	0.11	1.4
		- $\frac{1}{4}$ in.+6 m	8.1	0.37	3.6
		-6+10 m	<u>6.2</u>	<u>0.34</u>	<u>2.5</u>
			29.9	0.23	8.2
	Fines	-10 m	21.1	0.87	21.8
	Feed (calc.)		100.0	0.84	100.0

TABLE 2

Sink-Float Test - Distribution of Niobium in Screen Fractions

Screen Fraction Tyler	Product, Specific Gravity	Weight, % of feed	Assay, % Nb ₂ O ₅	Distribution, % of Feed Nb ₂ O ₅	Distribution, % of Fraction Nb ₂ O ₅
-1+ $\frac{1}{2}$ in.	Sink at 2.95	12.1	1.30	18.8	89.3
	" " 2.85	2.9	0.44	1.6	7.3
	Float at 2.85	<u>4.5</u>	<u>0.13</u>	<u>0.7</u>	<u>3.4</u>
		19.5	0.91	21.1	100.0
- $\frac{1}{2}$ + $\frac{1}{4}$ in.	Sink at 2.95	18.9	1.45	32.7	79.6
	" " 2.85	8.2	0.72	7.0	17.0
	Float at 2.85	<u>11.1</u>	<u>0.11</u>	<u>1.4</u>	<u>3.4</u>
		38.2	0.91	41.1	100.0
- $\frac{1}{4}$ in. +6 m	Sink at 2.95	2.7	1.43	4.6	38.2
	" " 2.85	3.1	1.11	3.9	32.3
	Float at 2.85	<u>8.1</u>	<u>0.37</u>	<u>3.6</u>	<u>29.5</u>
		13.9	0.73	12.1	100.0
-6+10 m	Sink at 2.95	0.4	1.38	0.7	18.2
	" " 2.85	0.7	0.93	0.7	18.2
	Float at 2.85	<u>6.2</u>	<u>0.34</u>	<u>2.5</u>	<u>63.6</u>
		7.3	0.45	3.9	100.0
	Fines	21.1	0.87	21.8	
	Feed (calc.)	100.0	0.84	100.0	

TABLE 3Tabling Tests, Coarse and Fine TablesTests T1 and T2

Product	Product Assay, % Nb ₂ O ₅			
	Test T1		Test T2	
	Fine Table	Coarse Table	Fine Table	Coarse Table
Feed	0.70	0.65	0.85	0.63
Concentrate	2.68	1.30	2.32	2.35
Middling	0.65	0.50	0.60	0.70
Tailing	0.38	0.27	0.35	0.34
Cyclone overflow	-	-	0.64	-
Conc. weight, %	8.2	15.7	-	-
Estimated recovery, Nb ₂ O ₅ %	34.	32.	-	-

TABLE 4

Tabling Tests with Grinding to -80 Mesh

Tests T3, T4 and T5

Table Product	Product Assay, % Nb ₂ O ₅					
	Test T3		Test T4		Test T5	
	Deister Table	Wilfley Table	Deister Table	Wilfley Table	Deister Table	Wilfley Table
Feed (overall)	0.75		0.75		0.79	
Cyclone overflow	0.54		0.50		0.54	
Table feed	0.69	0.68	0.62	0.85	0.70	0.98
" conc.	1.23	2.10	1.95	3.75	1.85	1.95
" midd.	-	0.80	1.10	1.13	0.36	0.59
" tailing	0.38	0.45	0.36	0.45	0.37	0.36

Note 1. Test T3 - Deister table concentrate and middlings reground.

2. Test T5 - Two stage grinding to minus 80 mesh

Total Nb₂O₅ recovery in concentrates approximately 69.%

Ratio of concentration - 3.5:1

Cyclone slime losses - weight 11.8%

Nb₂O₅ 8. %

TABLE 5
Flotation

Test Run No.	Log of Reagent Additions and Operating Changes				Operating Conditions
	Total Reagent Additions lb/ton of initial feed				
	Duomac T (DT)	Methyl Isobutyl Carbinol (MIC)	MgSiF ₆	Catechol	
F5	.94		5.7	1.25	16 cells on Calcite Float DT to Pyro Float .05
F6	"		"	"	Corks inserted for froth control. No DT to Pyro Float
F7	"		"	1.50	
F8	1.03	0.38	6.0	1.58	MIC to 1st cell only
F9	1.01	0.35	8.2	3.3	
F10	1.19	0.38	7.0	3.65	
F11	1.13	0.38	7.0	3.65	
F12	2.12	0.78	8.2	3.77	One-half of MIC to Pyro Roughers
F13	1.82	"	"	3.65	One-half of MIC to Pyro Roughers
F14	1.41	0.38	"	4.88	MIC to 1st cell only
F15	"	"	"	3.90	Catechol reduced to cleaner cells
F16	1.58	"	"	3.90	6 Fagergren cells on Calcite Float cleaners
F17	"	"	"	"	Better operating control
F18	1.42	"	"	4.6	Recycled pyrochlore cleaner and re-cleaner middlings
F19	1.62	0.68	8.2	4.6	MIC at Calcite Float Feed and Pyrochlore Float Feed
F20	1.47	"	9.45	4.5	MIC at Calcite Float Feed and Pyrochlore Float Feed
F21	"	"	"	5.8	Catechol only changed

(cont'd)

Table 5 (cont'd)

Test Run No.	Log of Reagent Additions and Operating Changes				Operating Conditions
	Total Reagent Additions lb/ton of initial feed				
	Duomac T (DT)	Methyl Isobutyl Carbinol (MIC)	HgSiF ₆	Catechol	
F22	1.29	0.34	7.94	5.6	MIC to Calcite Float only
F23	"	"	"	"	Recleaner added to circuit
F24	1.36	0.27	8.45	5.5	
F25	"	"	7.88	3.53	Catechol off Pyrochlore Roughers
F26	"	"	"	2.27	Catechol reduced in cleaners by 2/3
F27	1.40	0.57	9.1	5.10	MIC and DT to Pyrochlore Roughers. Fourth stage cleaner added. All cleaner middlings recycled
F28	1.42	"	"	5.4	A little DT to 4th stage cleaner
F29	1.38	"	"	"	DT off Pyrochlore Roughers
F30	-	-	"	"	Three hr test
F31	1.78	0.76	10.2	2.42	0.3 lb DT to Pyrochlore Roughers. MIC to Pyrochlore Roughers
F32	1.92	0.90	"	"	2 cell Fagergren added as scavenger unit. 0.14 DT + 0.27 MIC to this
F33	1.71	0.61	9.95	2.39	MIC at two points. Erratic feed
F34	1.80	-	"	"	
F35	1.71	0.61	"	"	Check on F33
F36	1.77	0.58	10.0	4.35	Catechol increased in 4th stage cleaner
F37	"	"	6.3	2.4	Almost all catechol to 4th stage cleaner
F38	1.8	"	"	"	More DT to end of calcite float
F39	"	0.80	"	"	Duomac increased in Pyrochlore Rougher feed
F40			Test cancelled		
F41	1.8	0.80	6.3	2.4	As Test F39

TABLE 6

Flotation - Details of Test F11

<u>Reagent</u>	<u>cc/min</u>	<u>lb/ton</u>	<u>Point of Addition</u>
NaOH		6.55	Flotation feed
Duomac T	50		Cell # 1
(DT)	13		6
2% solution	6		9
	5		11
	16		14
Total		<u>1.13</u>	
Methyl Isobutyl Carbinol (MIC)		0.38	Cell # 2
MgSiF ₆		7.0	Calcite float tailing pump
Catechol	31		Pyrochlore rougher
10% solution	13		" cleaner
	14		" recleaner
Total		<u>3.65</u>	

Feed rate 420 lb/hr

pH of pulp - Calcite Float - 12.2

Pyrochlore Rougher - 5.2

Circuit - Calcite Float - 16 cells # 7 Denver

Pyrochlore Rougher - 6 " # 7 "

" Cleaner - 2 " # 5 "

" Recleaner - 2 " # 5 "

(cont'd)

Table 6 (cont'd)

<u>Metallurgy</u>			
<u>Product</u>	<u>Weight, % Calc.</u>	<u>Assay, % Nb₂O₅</u>	<u>Distribution, % Nb₂O₅ *</u>
Classifier Overflow (Feed)		0.64	
Calcite Float	17.6	0.38	9.6
" Tailing		0.80	
Pyrochlore Rougher Conc.		4.00	
Pyrochlore Rougher Tailing	72.0	0.25	25.8
Pyrochlore Cleaner Conc.		9.70	59.1
Pyrochlore Cleaner Tailing	5.5	0.67	5.3
Pyrochlore Recleaner Conc.		27.10	28.0
Pyrochlore Recleaner Tailing	4.1	5.63	30.2

* A satisfactory material balance was not obtained in this test so distribution is approximate only.

TABLE 7

Details of Test F28

Circuit - Calcite Float 16 cells (Denver #7)
 Calcite Float retreatment 6 cells (Fagergren)
 Pyrochlore Rougher - 7 cells #7 Denver
 " 1st cleaner - 4 cells #5 "
 " 2nd " - 4 cells #5 "
 " 3rd " - 2 cells #5 "
 " 4th " - 2 cells #5 "

Pyrochlore scavenger (1 cell #7 Denver
 (2 cells Fagergren)

Feed rate 420 lb/hr

Reagents

<u>Reagent</u>	<u>cc/min</u>	<u>lb/ton</u>	<u>Point of Addition</u>
Duomac T	65		Cell 1 Rougher
(DT)	18		6 "
2% solution	9		9 "
	5		11 "
	12		15 "
	5		Pyrochlore rougher feed
	3		4th stage cleaners
Total		1.42	
NaOH		6.55	
Methyl Isobutyl Carbinol	14 drops	0.27	Calcite float feed
	16 drops	0.30	Pyrochlore rougher
MgSiF ₆		9.1	Calcite float tailing pump
Catechol	30		Pyrochlore roughers
	30		Cleaners
	15		Recleaners
	10		4th stage cleaners
Total		5.4	

(cont'd)

Table 7 (cont'd)

<u>Metallurgy</u>			
<u>Product</u>	<u>Weight, % Calc.</u>	<u>Assay, % Nb₂O₅</u>	<u>Distribution, % Nb₂O₅</u>
Classifier Overflow		0.73	
Cleaned Calcite Float	11.8	0.53	7.7
Rougher Tailing	84.4	0.27	28.0
Final Pyrochlore Conc.	<u>3.76</u>	<u>13.90</u>	<u>64.3</u>
Calculated Feed	100.00	0.81	100.0

TABLE 8

Details of Test F41

Circuit - See flowsheet, Figure 1

Feed rate 420 lb/hr

Reagents

<u>Reagents</u>	<u>cc/min</u>	<u>lb/ton</u>	<u>Point of Addition</u>
NaOH		3.8	Classifier O'flow
Duomac T	64		Cell No. 1
2% solution	22		" " 6
(DT)	16		" " 22
	14		" " 14
	30		Pyrochlore rougher
	8		Scavenger cells
Total		1.8	
Methyl Isobutyl Carbinol (MIC)	21 drops		Cell No. 2
	11 drops		Pyrochlore roughers
	11 drops		Scavenger cells
Total		0.8	
MgSiF ₆		6.3	Calcite float tailing pump
Catechol	3		Pyrochlore roughers
	10		Recleaner
	25		4th cleaner
Total		2.4	

(cont'd)

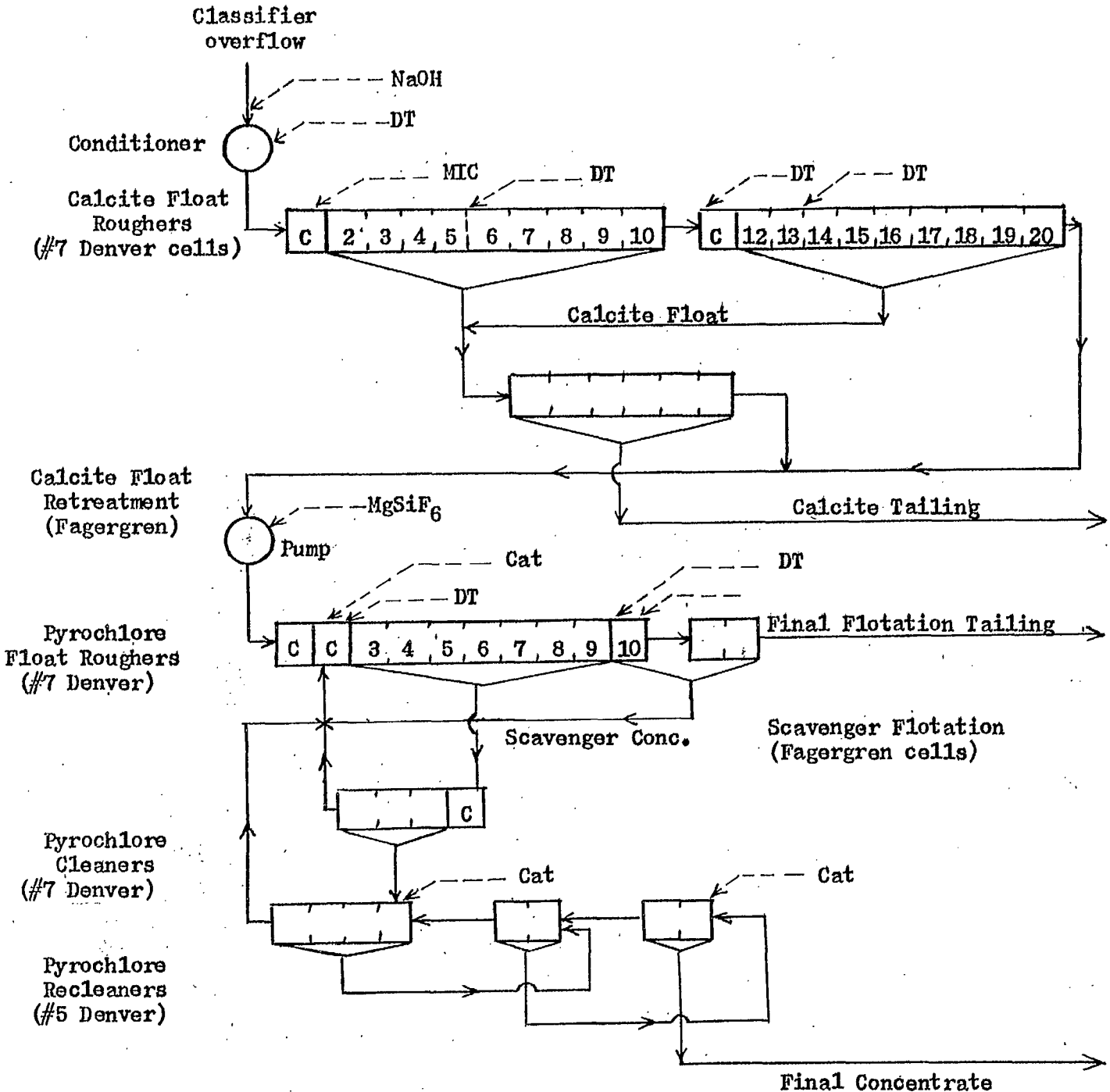
Table 8 (cont'd)

Metallurgy

<u>Product</u>	<u>Weight, %</u>	<u>Assay, % Nb₂O₅</u>	<u>Distribution, % Nb₂O₅</u>
Classifier Overflow	100	0.75	100
Calcite Cleaner Conc.	10.5	0.47	6.5
Final Tailing	81.0	0.27	29.1
Final Concentrate	5.5 (calc.)	8.80	64.4*
<hr/>			
3rd Stage Cleaner Conc.		7.80	
2nd Stage Cleaner Conc.		5.70	
1st Stage Cleaner Conc.		4.50	
Rougher Conc.		2.28	
Cleaner Tailing		0.77	

* This distribution is based on the classifier overflow feed grade with final concentrate weight calculated.

Figure 1 - Flotation Pilot Plant Flowsheet



Reagents

- DT - Duomac T
- MIC - Methyl Isobutyl Carbinol
- Cat - Catechol