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FLOTATION OF SPECULAR HEMATITE FROM THE CAROL LAKE DEPOSITS OF THE IRON ORE CO. OF CANADA LTD., SCHEFFERVILLE, QUEBEC

by

D. E. PICKETT

MINERAL DRESSING AND PROCESS METALLURGY DIVISION

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Mines Branch Investigation Report IR 59-47

FLOTATION OF SPECULAR HEMATITE FROM THE CAROL LAKE DEPOSITS
OF THE IRON ORE COMPANY OF CANADA, SCHEFFERVILLE, P.Q.

by

D. E. Rickett^{*}

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SUMMARY OF RESULTS

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Flotation tests were made on samples of iron ore from the Carol Lake deposits of the Iron Ore Co. of Canada containing from 31 to 45% iron mainly as specular hematite in a quartz gangue.

Two methods were investigated:

- (a) Dry grinding with oleic acid in the mill followed by conventional flotation.
- (b) Grinding without reagent, desliming, high-density conditioning with oleic acid, and flotation.

Locked tests on a composite sample from three pits gave concentrates containing 66.5% iron with a recovery of 86.1% using method (a). A similar grade of concentrate was made with 78% recovery using method (b). Oleic acid consumptions for the two methods were 0.72 and 0.54 lb/ton, respectively.

Recoveries of 90% were made on a sample of Pit No. 5 ore with as little as 0.16 lb/ton oleic acid using method (b).

A pilot plant continuous test using method (a) gave a concentrate assaying 64.3% iron, at a recovery of 85% with oleic acid consumption of 0.8 lb/ton.

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CONTENTS

| | <u>Page</u> |
|---|-------------|
| Summary of Results | i |
| Introduction | 1 |
| Object of Investigation | 1 |
| Ore Samples | 2 |
| Characteristics of the Ore | 2 |
| Outline of Investigation | 3 |
| Results | 5 |
| Details of Investigation | 6 |
| A. Dry Grinding Tests - No. 5 Pit Sample | 6 |
| B. Continuous Pilot Plant Test - Carol Lake Composite Sample | 13 |
| C. Laboratory Tests on the Carol Lake Sample ... | 16 |
| D. Laboratory Tests with Conditioning after Grinding | 19 |

Tables

| | |
|---|----|
| Table 1 - Analysis of Pit Samples from Carol Lake Ore Body | 2 |
| 2 - Effect of Varying Oleic Acid Contact Time in Dry Grinding | 9 |
| 3 - Test Results Using Emulsified Oleic Acid. | 11 |
| 4 - Metallurgical Balance for Test SC 29 | 14 |
| 5 - Details of Laboratory Locked Cycle Tests on the Carol Lake Composite | 17 |
| 6 - Effect of Desliming Flotation Feed After Wet Grinding | 21 |
| 7 - Batch Tests on the Carol Lake Composite Sample | 23 |

Appendices

| | |
|--|----|
| 1 - Mineragraphic Report M-1555-E | 25 |
| 2 - Mineralogical Report M-1614-E | 30 |
| 3 - Test SC 2 - Locked Cycle Flotation of Pit No. 5 Ore Using Dry Grinding with Oleic Acid | 32 |
| 4 - Material Balance for Locked Cycle Test SC 28 and Metallurgical Balance | 34 |
| 5 - Covering letter, 2-ton shipment | 35 |
| 6 - Details of Continuous Flotation Test on Carol Lake 2-Ton Shipment | 36 |
| 7 - Test Details of Iron Ore Co. Test 0925 .. | 39 |
| 8 - Test Details of Batch Test 1203-1 | 41 |
| 9 - Test Details of Locked Test 1218 | 42 |

INTRODUCTION

The first flotation investigations at the Mines Branch on the specular hematite-quartz ore from the Iron Ore Co. of Canada operations in Quebec-Labrador were carried out by Mr. J. D. Johnston and reported in Investigation Report MD 3231, dated Nov. 28, 1957. On composite samples from several pits, flotation tests in which the quartz was floated using amine type collectors were very successful with concentrate grades of better than 61% iron containing less than 3.8% silica. Recoveries were from 76 to 85% not including 15 to 7% recoverable iron in cleaner tailings. Fatty acid flotation tests were not as successful due to lack of selectivity.

During a research investigation on fatty acid collector action, a sample of No. 5 pit ore from the Carol Lake deposits was used as a typical hematite-quartz ore. The results of a procedure in which the ore was ground dry with oleic acid added to the mill, followed by flotation in a laboratory cell, gave such encouraging results that the present investigation was started. Results of the preliminary tests were reported in Mineral Dressing and Process Metallurgy Internal Report MDI-58-4 dated May 20, 1958 by Mr. S. Chwastiak.

Object of Investigation

The object of the present investigation was to develop a suitable fatty-acid flotation procedure for hematite ores, in particular the quartz-hematite ore of the Carol Lake deposits of the Iron Ore Co. of Canada.

Initially, a dry grinding procedure was investigated. However, as the required conditions for flotation of hematite became

known an alternative wet grinding procedure which seemed promising was also investigated.

Ore Samples

The samples available for test work were sample rejects of Carol Lake pits Nos. 1, 3 and 5 ore from a shipment received at the Mines Branch Nov. 3, 1957. A representative composite of the three lots was used for pilot plant tests. The bulk of this shipment was reserved for magnetizing roast investigations. (Results of the magnetizing roasting preliminary tests were reported in Technical Memorandum MDT-58-4, "Magnetizing Roasting Tests on Hematite Ore from the Carol Lake Property of Iron Ore Company of Canada, Ltd." by S. Chwastiak, dated April 10, 1958.)

The composition of the ore shipment is given in the following table, taken from a letter of Nov. 4, 1957 from Dr. C. Dufresne of Iron Ore Co. of Canada:

TABLE 1

Analysis of Pit Samples from Carol Lake Ore Body

| | <u>Fe</u> | <u>Phos.</u> | <u>Mn</u> | <u>Insol</u> | <u>Tons</u> |
|--------------------------|--------------|--------------|------------|--------------|-------------|
| Pit No. 1 (Carol 1 West) | 45.46 | .004 | .06 | 34.65 | 7.0 |
| " " 3 (Carol 1 East) | 31.27 | .006 | .12 | 50.44 | 5.5 |
| " " 5 (Carol 1 East) | <u>38.69</u> | <u>.009</u> | <u>.16</u> | <u>39.42</u> | <u>8.5</u> |
| Total | 39.00 | .006 | .12 | 40.72 | 21.0 |

Characteristics of the Ore

Mineralogical Reports on the three pit samples in the 21 ton shipment (Report No. M-1555-E) and on the composite sample from the Carol Lake ore (Report No. M-1614-E) are attached as Appendices Nos. 1 and 2 to this report.

Both reports show that hematite is the principal ore mineral with minor amounts of magnetite and that the gangue mineral is mainly quartz. Figure 2 of report M-1555-E shows that the quartz gangue contains small inclusions of iron oxide. Subsequent magnetic separation tests have shown that these are mainly magnetite inclusions. Their presence should not affect flotation results to any appreciable degree. However, they do affect magnetic separation results very seriously.

From the reports, effective liberation should be obtainable at -48 mesh grind with the No. 5 pit sample, whereas a -65 mesh or finer grind is required for the No. 1 and No. 3 pit samples. Complete liberation of the inclusions in the gangue would not be practical. Therefore, the grade of the tailing produced would be determined by the amount of iron oxides in the quartz and this would be lowest in the No. 5 pit sample.

OUTLINE OF INVESTIGATION

Two methods of flotation of hematite from the Carol Lake ore samples were investigated and tested. These methods were:

- (a) Dry grinding with oleic acid in the mill followed by flotation with staged small additions of oleic acid to the cells.
- (b) Wet grinding followed by desliming, high-density conditioning with oleic acid, and flotation.

The test work is summarized below:

| <u>Test Series</u> | <u>Method</u> | <u>Ore Sample</u> | <u>1000 g</u> |
|--------------------|---------------|----------------------|-------------------------------|
| A-1 | (a) | Carol Lake Pit No. 5 | Batch tests. |
| A-2 | (a) | Carol Lake Pit No. 5 | Locked cycle tests. |
| B | (b) | Carol Lake Composite | Pilot plant continuous test. |
| C | (a) | Carol Lake Composite | Batch and locked cycle tests. |
| D-1 | (b) | Carol Lake Pit No. 5 | Batch tests. |
| D-2 | (b) | Carol Lake Composite | Locked cycle test. |
| D-3 | other methods | Carol Lake Composite | Batch tests. |

In all tests the oleic acid used was Armour's Neo-Fat 94.04. Fluosilicic acid, H_2SiF_6 , was used as a silica depressant in cleaning and recleaning stages. In some of the early tests, a petroleum sulphonate, Petronate L, was used to improve the froth as the froth tended to fall off rapidly. In subsequent tests it was found that better results were obtained if the oleic acid addition was regulated more carefully and no Petronate was used.

Batch tests were all made on 1000 g charges ground in a porcelain mill with steel balls, either wet or dry as required by the conditioning method. Locked cycle tests were made using successive 1000 g batches. The best grind for the ore samples was not fully investigated. In the tests, fine grinding resulted in excessive reagent consumption without any increase in recovery. A 65 mesh grind (80% passing) seemed to give good recovery although the Carol Lake composite required a little finer grind than the No. 5 pit sample to give a good concentrate grade.

Details of the test procedure are given in "Details of

Investigation", below.

RESULTS

From the tests to date it appears that hematite can be floated with reasonable recovery and low reagent cost from the Carol Lake ores. This may be done either (a) by dry grinding of the ore with oleic acid in the mill followed by a short conditioning period and flotation or (b) by grinding without reagent followed by desliming, high-density conditioning, and flotation of the hematite. In method (b) most of the test work was done on wet ground batches but a test on a dry ground batch indicated that desliming (or de-dusting) a dry ground ore followed by high-density conditioning might give good results.

The ore taken as representative of probable Carol Lake mill feed was a composite of pits 1, 3 and 5. On this sample, in a locked cycle test using the dry grinding-conditioning method (a), a concentrate assaying 66.5% iron, was produced at 86.1% recovery. Using method (b) a concentrate of the same grade was made at 78% recovery. Oleic acid consumptions in these two tests were 0.72 and 0.54 lb/ton, respectively.

The sample from pit No. 5, when treated alone, was much more amenable to concentration by flotation and oleic acid consumption was very low. Using the dry grinding method (a), a concentrate assaying 64.7% iron was produced at a recovery of 92.5% in a locked test. Oleic acid consumption was 0.4 lb/ton, and 0.2 lb/ton fluosilicic acid was used. The locked test reached equilibrium quickly indicating good control of an operating plant. With the wet grinding method (b) the concentrate assayed 65.1% iron with 90% recovery. Oleic acid consumption in this latter test was approximately 0.16 lb/ton. Desliming was required.

The reason for the difference in results, when the composite sample is compared with the No. 5 pit sample, may be due to the much greater content of red ferruginous cementing material in the No. 1 pit sample which forms part of the composite. In method (b), if this slime material is not removed prior to flotation, reagent consumption is higher and the grade of concentrate is lower. Desliming is not necessary in method (a).

The continuous pilot plant test using the composite sample did not give good results due to the difficulty of getting complete conditioning in the grinding equipment employed. No continuous tests were made using the wet conditioning method.

More test work is required to improve the details of the processes. Variations in mineralogy, as indicated above for No. 1 and No. 5 pits, may be important with other pit samples. No attempt was made to differentiate between the magnetite and hematite behaviour. This would require investigation if some portions of the ore body are high in magnetite. The slime content of the ore is also important because of its effects on recovery and reagent consumption. A research investigation is being carried out on the oleic acid adsorption in the conditioning stage and this should give useful information on the quartz depression problem.

DETAILS OF INVESTIGATION

A. Dry Grinding Tests - No. 5 Pit Sample

This series of tests was made to determine the essential steps in procedure for the dry grinding method. For this purpose the No. 5 pit sample from the Carol Lake ore was chosen as it was a simple two-mineral mixture with a good possibility of control by binocular

microscope observation in the laboratory since the minerals were readily recognizable.

The tests in this series were conducted by Mr. S. Chwastiak of the Division staff. In all, 19 tests were made including 17 dry grinding batch tests and 2 locked cycle dry grinding tests. In several tests the results were not assayed as optical methods indicated very inferior results.

As a result of preliminary tests a standard laboratory procedure was adopted. Subsequent testwork showed that some changes could be made in the procedure with better control and more informative results.

The standard procedure developed for the dry grinding batch tests was as follows:

Grinding - 1000 g of -14 mesh crushed ore was ground dry in the laboratory porcelain mill for 15 min. To the grind 5 drops of oleic acid (Armour Neo-Fat 94.04) was added. The screen analysis of the product was approximately:

| | | |
|---------|---|---------------|
| + 65 m | - | 4.0 % |
| + 100 m | - | 13.6 " |
| + 150 m | - | 24.0 " |
| + 200 m | - | 20.6 " |
| - 200 m | - | <u>37.8 "</u> |
| | | 100.0 " |

Conditioning - The ore was transferred to a 1000 g Agitair cell and agitated with 1000 ml of water for 2 min. An additional 2 drops of oleic acid was added to this step.

Flotation of 1st Concentrate - At the end of the conditioning step, water was added to the cell to give the operating volume (approx. 25% solids) and the air was regulated to float a concentrate at a controlled rate.

Flotation of 2nd Concentrate - When the froth had subsided an additional 1 drop of oleic acid was added and flotation was continued until the froth again subsided.

The reagent (oleic acid) addition in the standard procedure was checked and the result with 5 drops in the dry grind was obviously better than with either 4 or 6 drops.

The results of varying the time of contact with oleic acid during the grinding step are shown in Table 2.

TABLE 2

Effect of Varying Oleic Acid Contact Time in Dry Grinding

| Test | Contact Time (min) | 1st Concentrate | | | 2nd Concentrate | | | Tailing | |
|-------|--------------------|-----------------|-------------|--------------------|-----------------|-------------|--------------------|-----------|-------------|
| | | Weight, % | Assay, % Fe | Distribution, % Fe | Weight, % | Assay, % Fe | Distribution, % Fe | Weight, % | Assay, % Fe |
| SC 13 | 15 | 35.2 | 60.1 | 68.6 | 51.6 | 54.3 | 90.7 | 48.4 | 5.9 |
| SC 14 | 10 | 48.7 | 55.9 | 87.1 | 56.4 | 51.3 | 92.6 | 43.6 | 5.3 |
| SC 15 | 5 | 46.3 | 55.7 | 82.7 | 55.1 | 50.9 | 89.9 | 44.9 | 7.0 |
| SC 18 | 2 | 33.4 | 60.5 | 65.1 | 40.0 | 60.1 | 77.4 | 60.0 | 11.7 |

* Cumulative

In these tests the grinding time was uniformly 15 min but the oleic acid was introduced to the mill at different times. The calculated feed varied from 31 to 32% iron. It is apparent that conditioning was not complete with less than 10 min contact under the conditions in the 1000 g grinding mill.

A series of tests was conducted to determine the effect of adding the oleic acid to the dry grind as a water emulsion. Neo-Fat 94.04 was emulsified in water using approximately 2% petroleum sulphonate (Petronate N) to produce a stable emulsion containing approximately 75% oleic acid. This was added to the grinding mill for the 15 min grinding period in amounts varying from 3 to 7 drops. One drop of emulsion was added to obtain the second concentrate. The best results were obtained with 4 or 5 drops as shown in Table 3.

The floated product was much lower in grade than with standard oleic acid. Recovery was high and it is possible that cleaning several times would produce a good concentrate. Circulating loads would be high.

Two locked cycle tests were made using modifications of the dry grinding and conditioning procedure. In test SC 2, six 1000 g batches of pit No. 5 ore were ground for 15 min with 5 drops of oleic acid. Each dry ground charge was transferred to a 1000 g Agitair flotation cell. Distilled water was added to obtain a pulp density of 55 to 60% solids, and the pulp was agitated with additional collector. At the end of the conditioning period, the scavenger concentrate and the cleaner tailing from the flotation of the previous batch of ore were added to the cell. Some solution had to be decanted from the cleaner tailing for this step, but it was set aside and used

TABLE 3

Test Results Using Emulsified Oleic Acid

| Test No. | Emulsion to grind, drops | 1st Concentrate | | | 2nd Concentrate | | |
|----------|--------------------------|-----------------|-------------|--------------------|-----------------|-------------|--------------------|
| | | Weight, % | Assay, % Fe | Distribution, % Fe | Weight, % | Assay, % Fe | Distribution, % Fe |
| SC 24 | 5 | 53.2 | 60.2 | 85.6 | 70.2 | 49.4 | 97.2 |
| SC 27 | 4 | 55.1 | 59.5 | 83.0 | 72.8 | 52.8 | 97.3 |
| SC 23 | 7 | 57.7 | not assayed | | all floated | | |
| SC 25 | 3 | 35.0 | not assayed | | high Fe tailing | | |

(Feed calculated 35.7 to 39.5% Fe)

to maintain a constant pulp level in the cell during the cleaner, rougher and scavenger flotation stages. (Thus, only a small amount of solution remained at the end of the test.) Rougher flotation was then begun. Some Petronate N was added during flotation to sustain the froth which fell off rapidly toward the end of the flotation period. More collector was added and a scavenger concentrate was floated in the same cell.

The rougher concentrate was combined with the recleaner tailing from the treatment of the previous batch of ore, and then cleaned in a 1000 g Agitair cell. The cleaned concentrate was repulped in a 500 g Agitair cell with distilled water, and recleaned using hydrofluosilicic acid as a silica depressant.

Slight changes in procedure were necessary in the flotation of the first batches of ore to compensate for the effect of recycling the middlings. Details of this test are given in Appendix 3. The reagents and conditions are given for the equilibrium conditions at the end of the test.

The composite concentrate grade (calculated) was 65.5% iron with a recovery of 88.7%. Approximately 3.7% of the iron remained in the combined middling products from the last cycle, assaying 26.8% iron. Assuming a tailing of 6.5% iron from retreatment of these middlings, overall recovery in continuous operation would be nearer 90%.

In test SC 28 a locked cycle test was made using a modified method developed as a result of the batch testing program. The procedure

varied from that used in test SC 2 in that no Petronate was used. As in test SC 2, 5 drops of oleic acid was added to each batch grind, with two drops to the conditioning stage. The scavenger float was made with 1 drop of oleic acid. In the first cycle only, 1 drop of oleic acid was required as frother in the cleaner stage. For all cycles 5 ml of 2% fluosilicic acid was added to the recleaner stage. All products were weighed to obtain information on the equilibrium in the locked test. As the test seemed to be in balance after two cycles, the products from the last three cycles were combined and assayed to calculate the probable recovery and grade from a continuous test. The composite concentrate assayed 64.7% iron with a very high recovery of 92.5% from a calculated feed of 32.4% iron. Details of the test results are given in Appendix 4. The stability of the system after two cycles is shown in the material balance. The reagent consumption was: oleic acid 0.44 lb/ton and fluosilicic acid 0.8 lb/ton.

B. Continuous Pilot Plant Test - Carol Lake Composite Sample

As the result of the preceding tests on No. 5 pit ore, plans were made to make a continuous test in the pilot plant. Iron Ore Co. arranged to ship a 2 ton composite sample from Carol Lake with the composition as given in the letter from Mr. J. C. Caron to Mr. L. E. Djingheuzian dated Aug. 18, 1958 (copy of this letter is attached as Appendix 5).

As this sample did not arrive in time a 2 ton composite sample was made up from the Nov. 4, 1957 shipment with the same weight distribution, i.e. 37% from Pit No. 1, 55.6% from Pit No. 3, and 7.4% from Pit No. 5. This composite was used for the pilot plant test.

Preliminary mineralogical examination (see Appendix 2, Mineralogical Report M-1614-E, attached) indicated that grinding for this sample should be finer than for the No. 5 pit sample.

One laboratory locked cycle test, SC 29, was made on a sample of the Carol Lake composite to determine a reagent balance for the pilot test. The procedure was as used in test SC 28 with some changes in reagent quantities. Depression of hematite by slime was noticeable when the middling products were recycled. Therefore, these were dewatered. Although the test seemed to be in balance after three cycles, the product weights indicated a build up in the amount of middling, mainly in the scavenger concentrate. The concentrates and tailings from the last two cycles were combined, assayed, and the metallurgical balance in Table 4 was calculated.

TABLE 4
Metallurgical Balance for Test SC 29

| Product | Weight, % | Assay, % Fe | Distribution of Iron, % |
|-----------------|--------------|----------------|----------------------------|
| Concentrate | 49.0 | 68.8 | 83 |
| Tailing | 51.0 | 13.1 | 17 |
| Calculated Feed | 100.0 | 40.4 | 100 |

These results indicated that recovery would be somewhat lower from the Carol Lake composite than from the pit No. 5 ore alone. Also it was apparent that additional oleic acid should be added to the grind and that the middling returns might be a problem.

To prepare the feed for the grinding unit the 2 ton sample was passed through the impact crusher to make a nominal -8 mesh product. This was then ground in a 6 in. x 20 in. Hardinge mill in closed circuit

with a 48 mesh screen. Oleic acid was added to the mill feed at the rate of 0.4 lb/ton. When approximately half of the ore had been ground in this way it was fed at the rate of 100 lb/hr to the flotation unit which consisted of nine 8 in. x 8 in. Agitair cells arranged to give a conditioning cell, 3 roughers, 2 cleaners, 2 recleaners, and a scavenger cell.

Details of this test are given in Appendix 6 attached to this report. As expected from the preliminary test, recovery was not as good as from the tests on pit No. 5 ore alone. The grinding circuit did not perform satisfactorily as the type of mill used would not discharge the dry ground product. This was the first time the Agitair cells had been used and considerable ore and time were wasted before the circuit was made to function properly. As a result both ore and time ran out before the test conditions were fully investigated.

The best results were obtained during a 1 hr period on Sept. 5 when the composite concentrate sample assayed 64.28% iron at a recovery of 85% from a feed assaying 34.7% iron. This feed had been ground with approximately 0.4 lb oleic acid per ton and an additional 0.4 lb of oleic acid was added to the flotation circuit. About 0.5 lb of 30% fluosilicic acid per ton of feed was added to the cleaner circuits.

The following observations were made as a guide for future work:

- (1) The Carol Lake three pit sample contained much more slime sized material and in addition to being more refractory metallurgically required about twice as much oleic acid as the pit No. 5 sample.

(2) From the amount of reagent required in the rougher circuit (0.2 lb/ton), it would seem that more reagent should have been added to the grind.

(3) It was necessary to dewater, and thereby partially deslime the cleaner and recleaner tailings before returning them to the circuit.

(4) The ore should be floated within a day of being ground with the reagent, or selectivity will deteriorate. It is possible that best results would be obtained if the ore were floated immediately after grinding.

C. Laboratory Tests on the Carol Lake Sample

The object of these tests was to find some method of improving on the results of the pilot plant test either by better grinding or better reagent control.

Three problems appeared to require investigation; (1) the grinding method required to give thorough dispersal of the oleic acid and adequate conditioning of the minerals; (2) the correct reagent addition to the mill and (3) the effect of a long delay between grinding and flotation. Locked tests were used since the results of batch testing did not give grade or recovery data which could be correlated to production.

Six locked tests were made using the Carol Lake composite ore remaining from the continuous testing. Details of the reagents used and the results are shown in Table 5.

TABLE 5Details of Laboratory Locked Cycle Tests on the Carol Lake Composite

| <u>Test No.</u> | 0915 | 0925 | 1014 | 1016 | 1027 | 1024 |
|--|-------------------------------|-------------|-------------|-------------|------------|------------|
| <u>Mill Size</u> | 1000 g | 1000 g | 20 lb | 20 lb | 20 lb | 20 lb |
| <u>Reagents lb/ton</u> | | | | | | |
| <u>Oleic Acid</u> | | | | | | |
| to grind | 0.48 | 0.60 | 0.80 | 0.80 | 0.90 | 1.0 |
| to conditioning | 0.12 | 0.12 | 0.12 | 0.18 | nil | nil |
| to scavenger | 0.06 | 0.06 | 0.12 | 0.12 | 0.06 | 0.06 |
| to cleaner | <u>0.06</u> | <u>0.06</u> | <u>0.06</u> | <u>0.06</u> | <u>nil</u> | <u>nil</u> |
| Total | 0.72 | 0.84 | 1.10 | 1.16 | 0.96 | 1.06 |
| <u>Fluosilicic Acid</u> | 0.1 lb/ton to recleaners only | | | | | |
| <u>Metallurgy</u> | | | | | | |
| <u>Final Concentrates Analysis, % Fe</u> | | | | | | |
| Cycle A | 66 | 66 | 61 | 59 | 64.8 | 60.5 |
| B | 67.4 | 67.5 | 62 | 59.4 | 61.6 | 57.3 |
| C | 65.5 | 66.5 | 60.6 | 62.2 | 58.6 | 61.5 |
| D | 67.4 | 67.2 | 60.6 | 61.8 | 59.2 | 60.4 |
| <u>Final Tailing Analysis, % Fe</u> | | | | | | |
| Cycle A | 18.6 | 13.0 | 14.3 | 19.9 | 16.2 | 13.0 |
| B | 11.0 | 8.6 | 21.2 | 21.8 | 15.9 | 11.5 |
| C | 9.2 | 8.5 | 17.4 | 22.2 | 19.5 | 13.9 |
| D | 14.6 | 9.3 | 19.0 | 25.4 | 17.6 | 15.6 |
| <u>Concentrate Recovery, % Fe</u> | | | | | | |
| | 80.8 | 86.1 | 68 | 58 | 82 | 78 |
| <u>Concentrate Weight, % of Feed</u> | | | | | | |
| | 42.2 | 47.0 | 40 | 36 | 53 | 45 |
| <u>Average Circulating Load, % of New Feed</u> | | | | | | |
| | 33 | 39 | 45 | 49 | 76 | 73 |

Tests 0915 and 0925 were made to confirm the results of test SC 29 and to establish the reagent requirements more accurately. In test 0915 the 1000 g batches of ore were ground dry with 8 drops (0.48 lb/ton) of oleic acid. In test 0925, 10 drops of oleic acid were added. The latter addition seemed to be the better as the concentrate composite assayed 66.5% iron with 86.1% recovery. Details of this test are given in Appendix 7. The circulating load stabilized at approximately 400 g. The scavenger concentrate and cleaner tailing were returned to the rougher float feed without dewatering. Some of the recleaner tailing solution was discarded; otherwise, cell capacity was exceeded. The recycling of the slimes in these solutions did not seem to have an unfavourable effect.

To investigate the effect of a different batch grinding method, 20 lb batches were ground with the reagent in a large laboratory batch mill. The product from each grind was split into approximately 1000 g lots and these were used for a locked cycle test employing basically the same procedure as in test 0925. Tests were made at three levels of oleic acid addition to the mill. Test 1016 was a locked test made on the same batch grind as test 1014 but after storage for 48 hr.

In spite of the high consumption of oleic acid, recovery and concentrate grade were poor in all tests on the 20 min batch grinds. The large circulating loads are an indication of inadequate conditioning in the mill and resulting poor selectivity. Since the collector is not adsorbed strongly, both hematite and quartz drop out in the first cleaning stage. In test 1016, after the ground material was aged, flotation was slower and an additional drop of oleic acid was required in the rougher stage. Recovery was poorer than with freshly ground

ore in test 1014.

In a seventh locked test a 20 lb batch was ground without reagent and floated. In this test more than 2.6 lb/ton of oleic acid was required in the cell before a concentrate could be floated. The selectivity was very poor and the samples were not assayed.

From these laboratory tests the following conclusions can be drawn:

(1) From the results of tests 0915 and 0925 it appears that good results should be possible in continuous flotation if equivalent conditions can be developed in a continuous dry grinding circuit.

(2) The 20 lb batch grinding tests demonstrated that a change in grinding method can give very poor results although reagent additions and time are not changed. This can explain why the grinding in the pilot plant test gave inferior results.

D. Laboratory Tests with Conditioning after Grinding

To compare the method in which the collector was added to the dry grinding charge with more conventional methods, tests were made in which the ore was ground without reagent, and then conditioned with oleic acid before flotation.

(1) Tests on Carol Lake Pit No. 5 Sample

The first tests were made on the Carol Lake Pit No. 5 sample. In test SC 3 a wet ground 1000 g batch was conditioned at high density (50% solids) with oleic acid. In test SC 4 the pulp, after wet grinding, was deslimed by decantation before conditioning. The results were normal for both tests, with rougher concentrate grades of approximately 55% and 90% recovery, but the reagent requirement

in the deslimed test was less than half of the requirement when the pulp was not deslimed.

To check more accurately on recovery, grade of concentrate, and reagent consumption, tests SC 5 and SC 12 were made in which locked cycle tests were employed. In test SC 5 the pulp was not deslimed while in test SC 12 the pulp batches were deslimed by double decantation before the recycled middling products were added for conditioning.

In test SC 5 six 1000 g batches were ground wet and conditioned at 50% solids without desliming. To obtain this density, wash water was kept to a minimum and some solution was decanted before conditioning. This solution was returned to the cell to make up cell volume and to reduce the density to 25% solids for flotation. For each cycle, after the first, the cleaner tailings and scavenger concentrate from the previous cycle were added to the pulp for the conditioning step. Conditioning was for 2 min. Rougher, scavenger and cleaner flotation steps were then carried out. Fluosilicic acid was used in the recleaner stage and Petronate L was used to assist frothing.

The overall grade of the concentrate composite was 63.3% iron but the circulating load was variable and the test never appeared to be in balance, with the recovery falling off with each cycle.

In test SC 12 the wet ground feed was deslimed before conditioning. Sodium silicate was used for desliming so an extra washing step was required. The reduction in oleic acid consumption, as compared to test SC 5, was not expected as the recycled products varied considerably in the first three cycles. However, the four cycle concentrate composite assayed 65.1% iron with a recovery of 90%.

Comparative data for these two tests are given in Table 6.

TABLE 6

Effect of Desliming Flotation Feed After Wet Grinding

| Ore | Test SC 5 | Test SC 12 |
|---|---------------------|-------------------|
| | <u>not deslimed</u> | <u>deslimed</u> |
| | Carol Lake Pit #5 | Carol Lake Pit #5 |
| No. of cycles | 6 | 4 |
| Grinding time, min | 10 | 10 |
| <u>Reagents, lb/ton</u> | | |
| Sodium silicate | nil | 2.0 |
| Oleic acid | 0.31 | 0.16 |
| Petronate L | 0.06 | 0.025 |
| Fluosilicic acid (30% grade) | 0.40 | 0.40 |
| <u>Concentrate (Overall)</u> | | |
| Weight, % | 52.7 | 51.4 |
| Assay, Fe % | 63.3 | 65.1 |
| Distribution, Fe % | 89.5 | 90.0 |
| <u>Tailing (Overall)</u> | | |
| Weight, % | 47.3 | 46.4 |
| Assay, Fe % | 8.3 | 7.0 |
| Distribution, Fe % | 10.5 | 8.7 |
| <u>Slimes (Overall)</u> | | |
| Weight, % | nil | 2.2 |
| Assay, Fe % | nil | 22.3 |
| Distribution, Fe % | nil | 1.3 |
| <u>Circulating Load, based on feed to one cycle</u> | | |
| Weight, % | 7.1 | 13.1 |
| Distribution, Fe % | 6.9 | 7.7 |

The circulating load being variable, the recovery figures in the table are approximate only. The effect of desliming of this ore sample before flotation seems to be mainly a reagent saving. In both tests oleic acid consumption was less than in dry grinding tests.

Wet grinding tests were also carried out in which the oleic acid was added to the grinding mill. In three tests, at three levels of oleic acid addition corresponding to 0.3, 0.6 and 0.9 lb/ton, results were very poor with rougher recoveries about 70% and concentrate grades of 48.7, 54.1 and 57.7% iron, respectively.

(2) Tests on Carol Lake Composite Sample

The pilot plant dry grinding test and subsequent laboratory tests on the Carol Lake composite sample indicated that proper conditions might become a problem in the dry grinding method. The success of the wet grinding tests on the pit No. 5 sample suggested that an alternate wet grinding procedure might be useful. Also the proposed flowsheet of the Iron Ore Co. required a method of treatment for preground ore.

Therefore the following methods were investigated by batch testing:

- (a) Dry grinding with no reagent followed by conditioning at high density.
- (b) As (a) but deslimed before conditioning.
- (c) Wet grinding with no reagent followed by desliming and conditioning at high density.
- (d) Wet grinding with reagent in the mill.

Of these, methods (b) and (c) in which the ore was deslimed before flotation gave the best results with low reagent consumption. Table 7 gives the details of the batch tests. Conditioning was carried out at 50% solids after desliming by decantation.

TABLE 7

Batch Tests on the Carol Lake Composite Sample

| <u>Test No.</u> | <u>1112</u> | <u>1203-2</u> | <u>1203-1</u> | <u>1204-1</u> | <u>1120-1</u> |
|--|-------------|---------------|---------------|---------------|---------------|
| Grinding time (min) | 15 | 20 | 20 | 20 | 10 (dry) |
| <u>Slime</u> | | | | | |
| Weight, % | 6.2 | -- | 8.4 | 7.2 | 4.1 |
| Assay, Fe % | 21.7 | -- | 21.8 | 21.3 | 19.4 |
| Distribution, Fe % | 3.7 | -- | 5.3 | 4.5 | 2.3 |
| <u>Reagents</u> | | | | | |
| <u>Oleic Acid (drops)</u> | | | | | |
| Conditioning | 8 | 8 | 10 | 12 | 5 |
| Rougher Float | nil | 3 | nil | nil | nil |
| Scavenger Float | 1 | 2 | 1 | 1 | 1 |
| <u>H₂SiF₆ (2%, ml)</u> | | | | | |
| Cleaner Float | 10 | 10 | 10 | 10 | nil |
| Recleaner Float | 5 | 10 | 10 | 10 | not recleaned |
| <u>Products</u> | | | | | |
| <u>Final Tailing</u> | | | | | |
| Weight, % | 27.3 | -- | 26.9 | 21.3 | 18.7 |
| Assay, Fe % | 9.1 | 20 est. | 6.8 | 6.2 | 7.7 |
| Distribution, Fe % | 7.2 | -- | 5.2 | 3.9 | 4.0 |
| <u>Concentrate</u> | | | | | |
| Weight, % | 34.4 | 35.7 | 34.5 | 37.9 | 49.4 |
| Assay, Fe % | 66.2 | 62.9 | 66.0 | 64.2 | 57.1 |
| Distribution, Fe % | 66.1 | 66 est. | 65.6 | 70.9 | 81.0 |
| <u>Middling Products (scavenger concentrates and cleaner tailings)</u> | | | | | |
| Weight, % | 32.1 | -- | 30.2 | 33.6 | 27.8 |
| Distribution, Fe % | 23.0 | -- | 23.9 | 20.7 | 13.7 |

Test 1120-1 was a dry grind with no reagent in the mill. The grade of the one stage cleaned concentrate is normal and should come up to nearly 65% iron on recleaning with fluosilicic acid. Test 1203-1 was the most successful of these batch tests and the detailed procedure and results for this test are given in Appendix 8.

To investigate the reagent requirements and recovery, a locked test, No. 1218, was made in which 4 batches were ground wet and floated after desliming and high density conditioning. The indicated recovery of iron was 78% at a grade of 66-67% Fe. Total oleic acid consumption was 0.54 lb/ton. The circulating load was building up during the test but the conditioning period was increased from 3 to 5 min in the last cycle and more concentrate floated so a longer conditioning time may reduce the weight floating in the scavenger concentrate and bring the circuit to equilibrium. The details of procedure and the results are given in Appendix 9.

• • •

DEP/DV

APPENDIX 1

MINERAGRAPHIC REPORTM - 1555 - EMINERALOGICAL INVESTIGATION OF IRON ORE FROM THE CAROL LAKE
DEPOSIT OF THE IRON ORE COMPANY OF CANADA, SCHEFFERVILLE, QUE.Introduction:

The sample of ore described in this report was obtained from a 21-ton bulk sample from three open pits (No. 1, 3 and 5) of the Carol Lake deposit.

Mineralogical Description:

The ore consists largely of hematite and quartz in a rather loosely bound granular aggregate. Magnetite and a manganeseiferous ankerite are present in relatively small amounts.

The hematite has two chief modes of occurrence: as dense, specularite-like grains, and as porous particles. The porous particles frequently contain inclusions of magnetite (Figure 1), which suggests that the porous hematite, at least, may have been formed from the oxidation of magnetite. The dense hematite generally contains no visible magnetite, but much of it has a higher-than-normal magnetic susceptibility, and is therefore weakly attracted by hand magnet. The proportion of porous to dense hematite is highest in the sample from Pit No. 1, where it constitutes about a third of the hematite, whereas, the samples from Pit Nos. 3 and 5 contain only a small proportion of porous hematite.

The magnetite, in addition to its occurrence as remnants in the hematite, is also present as large discrete grains. The hematite-magnetite ratios in the three samples were determined by Davis Tube tests on -100 mesh ore. The results are as follows:

TABLE IHematite-Magnetite Ratios from Davis Tube Tests

| <u>Pit No.</u> | <u>Hematite-Magnetite Ratio</u> |
|----------------|---------------------------------|
| 1 | 55:1 |
| 3 | 14:1 |
| 5 | 8:1 |

The hematite-magnetite ratio is particularly high in the sample from Pit No. 1. Since this sample also contains the highest

proportion of porous hematite with magnetite remnants, it is possible that the original hematite-magnetite ratio in the primary rock was fairly uniform, but that secondary oxidation of the magnetite in the ore from Pit No. 1 has increased this ratio.

Samples 3 and 5 contain about five percent of a manganese-ferrous ankerite, while in sample No. 1, this mineral is in the order of one percent. Table II shows a spectrographic analysis of a small sample of the manganese-ferrous ankerite concentrated from the Pit No. 3 sample by heavy liquids and magnetic separator.

TABLE II

Spectrographic Analysis of Manganese-ferrous Ankerite

| | |
|-----------|-----|
| Calcium | 20% |
| Iron | 12 |
| Magnesium | 7.5 |
| Manganese | 2.5 |

The grain size of both gangue and ore minerals varies from about 1 mm. in diameter (14 to 20 mesh) down to about 10 microns. A measure of the degree of liberation of ore from gangue minerals can be obtained by determining the specific gravities of sink products resulting from the heavy liquid separation of screened samples of ore. Complete liberation, for example, is achieved when the specific gravity of the sink product equals that of the pure ore minerals. Lower gravities result if combined ore-gangue particles are present in the sink. Heavy liquid separations at a density of 2.96 were carried out on three sizes of the crushed ore (-10+28 mesh, -35+48 mesh, and -65+100 mesh), and the specific gravities of the sink products determined by pycnometer. The results are shown in Table III, together with the estimated iron contents calculated from the specific gravities determined, assuming only quartz and hematite to be present. The small amount of magnetite and ankerite will not seriously affect these estimates.

TABLE III

Measured Specific Gravity and Calculated Iron Content
of Screened Sink Products

| Mesh Size | Pit No. 1 | | Pit No. 3 | | Pit No. 5 | |
|--------------|-----------|------------|-----------|------------|-----------|------------|
| | S.G. | Calc. % Fe | S.G. | Calc. % Fe | S.G. | Calc. % Fe |
| -10+28 | 4.12 | 50 | 3.99 | 47 | 4.04 | 48 |
| -35+48 | 4.38 | 56 | 4.23 | 53 | 4.65 | 61 |
| -65+100 | 4.30 | 63 | 4.67 | 61 | 4.86 | 64 |

Table III shows that a 60% iron concentrate is achieved only at 48 or 65 mesh. The small grain size of some of the iron oxides is illustrated by Figure 2, an oil immersion mount of the float from the Pit No. 5 sample. This photograph shows -65 mesh quartz grains,

the majority of which contain small inclusions of the iron minerals.
This condition is also typical of the other samples.

E. H. Nickel,
Ore Mineralogy Laboratory.

Mines Branch,
January 30, 1958.

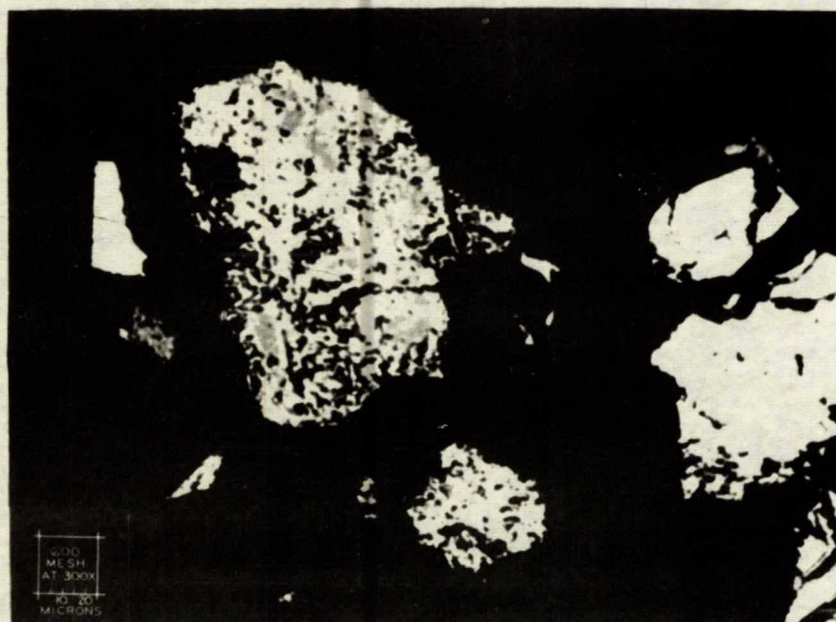


Figure 1. - Polished section of iron ore particles from the Pit No. 3 sample, showing dense and porous hematite grains (white). The porous grains contain magnetite inclusions (grey).

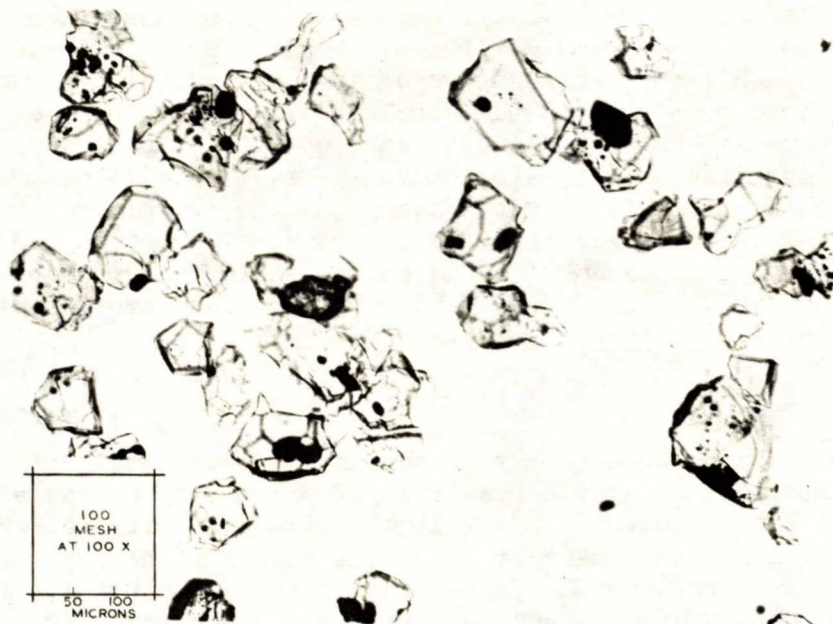


Figure 2. - Oil immersion mount of float product of -65 mesh Pit No. 1 sample, showing quartz grains (white), most of which contains small black inclusions of iron oxides.

APPENDIX 2

MINERALOGICAL REPORTM - 1614 - ETHE HEAVY LIQUID SEPARATION OF SCREENED FRACTIONS
OF COMPOSITE HEAD SAMPLE "C" OF THE IRON ORE COMPANY OF CANADA(cut from 2 ton sample used for mill feed
in pilot run Sept. 1-15, 1958)Introduction:

The purpose of the heavy liquid separations described here is to determine the size at which maximum liberation of the ore and gangue minerals is achieved. The density of the heavy liquid is chosen so that the grains of pure gangue will barely float, while the ore minerals, both those liberated and those combined with gangue minerals, will sink. If liberation is complete, only ore minerals will be present in the sink while, if liberation is not complete, the ore mineral content in the sink fraction will be lower, because of the combined gangue. If a series of screened sizes is separated in this fashion, then the size with the maximum degree of liberation will have the highest ore mineral content.

Procedure:

The ore under consideration consists predominantly of hematite and quartz, with specific gravities of 5.2 and 2.65, respectively. For the reason mentioned above, a heavy liquid with a density of 2.68 (tetrabromoethane and nitrobenzene mixture) was used for the separations. The screened samples were stirred in a separatory funnel containing the heavy liquid, and permitted to stand for at least 20 minutes. The sink and float fractions were then drawn off, washed, dried, weighed, and submitted for iron analyses. The results are shown in Table 1.

Discussion of Results:

TABLE 1

The Iron Content of Sink and Float Portions of Screened Iron Ore

| <u>Tyler Mesh Size</u> | <u>Percentage of Total Weight</u> | | <u>Iron Content (% Fe)</u> | |
|------------------------|-----------------------------------|--------------|----------------------------|--------------|
| | <u>Sink</u> | <u>Float</u> | <u>Sink</u> | <u>Float</u> |
| +35 | 11.9 | 0.3 | 46.7 | 1.55 |
| -35 +48 | 8.3 | 0.4 | 43.0 | 1.74 |
| -48 +65 | 12.1 | 1.7 | 38.9 | 1.06 |
| -65 +100 | 19.7 | 8.1 | 44.7 | 0.77 |
| -100+150 | 5.4 | 5.3 | 64.5 | 1.16 |
| -150+200 | 7.9 | 5.9 | 66.8 | 1.06 |
| -200+325 | 3.5 | 2.3 | 68.0 | 0.82 |
| -325 | 7.2 | | | |
| Total | 100.0 | | | |

It can be seen from Table 1 that the iron content of the sink portions rises sharply to 64.5% iron in the -100+150 mesh fraction, and increases to 68.0% iron in the -200+325 mesh fraction. This indicates that the point of significant liberation is 100 mesh, and that liberation is essentially complete at 325 mesh. This is corroborated by a microscopic examination of the products.

E. H. Nickel,
Ore Mineralogy Laboratory.

Mines Branch,
September 18, 1959.

APPENDIX 3

(Extracted from MDI-58-4)

Test SC 2 - Locked Cycle Flotation of Pit No. 5 Ore
Using Dry Grinding with Oleic Acid

Details of Test Conditions:Reagents and Conditions, 1000 g Ore Charge

| <u>Operation</u> | <u>Reagent</u> | <u>Time, min</u> | <u>pH, tailing</u> |
|------------------|------------------------------|------------------|--------------------|
| Grind | Reagent 94.04 - 5 drops | 15 | |
| Conditioning | Reagent 94.04 - 2 drops | 2½ | |
| Rougher flot. | Petronate - 5 drops | 2½ | 8.1 |
| Scav. flot. | Reagent 94.04 - 1 drop | ½ | 7.9 |
| | Petronate - 5 drops | | |
| Cleaner flot. | | 3 | 7.9 |
| Recleaner flot. | Hydrofluosilicic acid - 2 ml | 2½ | 7.4 |

Screen Analysis, 15 min Dry Grind

| <u>Screen Size</u> | <u>Retained, %</u> |
|--------------------|--------------------|
| 65 mesh | 4.0 |
| 100 " | 13.6 |
| 150 " | 24.0 |
| 200 " | 20.6 |
| minus 200 " | 37.8 |

A drop of Armour Neo-Fat 94.04 was found to weight between 0.025 and 0.3 g with 0.028 g/drop normally used for calculation of consumption. Five drops of Petronate solution are estimated to contain 0.01 g of Petronate N. The hydrofluosilicic acid solution was 10%.

Results of Flotation, Test No. 2

| Product | Weight, % | Assay, % | | Distribution, % | |
|-----------------|--------------|----------|------------------|-----------------|------------------|
| | | Fe | SiO ₂ | Fe | SiO ₂ |
| Conc. A | 8.00 | 59.0 | 12.4 | 12.6 | 2.3 |
| " B | 6.85 | 65.9 | 3.7 | 12.0 | 0.6 |
| " C | 6.85 | 66.8 | 2.4 | 12.2 | 0.4 |
| " D | 7.45 | 67.0 | 2.6 | 13.3 | 0.5 |
| " E | 6.87 | 67.0 | 2.7 | 12.2 | 0.4 |
| " F | 7.44 | 66.8 | 2.9 | 13.2 | 0.5 |
| " G | 7.40 | 67.0 | 2.3 | 13.2 | 0.4 |
| Recl. tailing G | 1.09 | 41.8 | 24.8 | 1.2 | 0.6 |
| Cl. tailing G | 2.58 | 20.4 | 55.0 | 1.4 | 3.4 |
| Scav. conc. G | 1.54 | 27.1 | 43.1 | 1.1 | 1.6 |
| Tailing A | 2.88 | 4.6 | 85.8 | 0.4 | 5.8 |
| B | 6.22 | 7.4 | 85.0 | 1.2 | 12.5 |
| C | 6.85 | 4.7 | 90.5 | 0.9 | 14.6 |
| D | 7.17 | 7.0 | 86.2 | 1.3 | 14.6 |
| E | 7.04 | 7.1 | 86.3 | 1.3 | 14.3 |
| F | 6.05 | 6.2 | 86.9 | 1.0 | 12.4 |
| G | 7.72 | 7.5 | 82.9 | 1.5 | 15.1 |
| Head* | 100.00 | 37.6 | 42.4 | 100.0 | 100.0 |
| O'all conc.* | 50.86 | 65.5 | 4.3 | 88.7 | 5.1 |
| Midds.* | 5.21 | 26.8 | 45.2 | 3.7 | 5.6 |
| O'all tailing* | 43.93 | 6.5 | 86.2 | 7.6 | 89.3 |

*Calculated

Based on a calculated head of 37.6% iron, a concentrate assay of 66.5% iron, and a tailing assay of 7.5% iron, the following metallurgical balance has been calculated.

Calculated Metallurgical Balance at Equilibrium
Test No. 2

| Product | Weight,* % | Assay, % | Distribution, %* |
|-------------|---------------|----------|------------------|
| | | Fe | Fe |
| Head | 100 | 37.6 | 100 |
| Concentrate | 51 | 66.5 | 90 |
| Tailing | 49 | 7.5 | 10 |

*Calculated

APPENDIX 4

Material Balance for Locked Cycle Test SC 28

| | <u>Weight, g</u> | | | | | <u>Total Weight, g</u> |
|-----------------|--------------------|--------------------|--------------------|--------------------|--------------------|----------------------------|
| | <u>Cycle A</u> | <u>Cycle B</u> | <u>Cycle C</u> | <u>Cycle D</u> | <u>Cycle E</u> | |
| New feed | 984 | 984 | 984 | 984 | 984 | 4920 |
| In Cycle | 984 | 1176 | 1238 | 1236 | 1252 | |
| Concentrate | 379 | 470 | 437 | 438 | 452 | 2176 |
| Tailing | 413 | 452 | 549 | 530 | 499 | 2443 |
| Scav. Conc. | | | | | 97 | 97 |
| Cleaner ton/g | | | | | 169 | 169 |
| Recleaner ton/g | | | | | 34 | 34 |
| To next cycle | 192 | 254 | 252 | 268 | 302 | |

Metallurgical Balance, Cycles C, D and E

| <u>Product</u> | <u>Weight, %</u> | <u>Assay, %</u> | | <u>Distribution, % Fe</u> |
|-------------------------|----------------------|-----------------|------------------------|-------------------------------|
| | | <u>Fe</u> | <u>SiO₂</u> | |
| Concentrate (composite) | 46.3 | 64.7 | 4.0 | 92.5 |
| Tailing (composite) | <u>53.7</u> | <u>4.5</u> | <u>-</u> | <u>7.5</u> |
| Calc. Feed | 100.0 | 32.4 | | 100.0 |

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APPENDIX 5

IRON ORE COMPANY OF CANADA

Schefferville, Que.,
August 18, 1958.

Mr. L. E. Djingheuzian,
Senior Engineer,
Division of Mineral Dressing
and Process Metallurgy,
552 Booth Street,
Ottawa, Ont.

Dear Mr. Djingheuzian:

Your 2-ton bulk sample of Wabush ore from Carol Lake deposit was to arrive in Montreal August 21st. It consists of samples from Pit Nos. 1, 3 and 5. Each bag can be identified as to its pit origin by the orange number painted on it.

Please note that the number of bags from Pit No. 5 is relatively low, our reserve of this pit sample being expired.

However, the number of bags from each Pit was carefully chosen to obtain a bulk sample which should approximate the overall grade of the Carol Lake Deposit, namely, 37.5% Fe.

Following are the analyses results from the blast hole cores of each Pit:

| | <u>% Fe</u> | <u>% Phos</u> | <u>% Mn</u> | <u>% Insol</u> | <u>Bags</u> |
|--------------------------|--------------|---------------|-------------|----------------|-------------|
| Pit No. 1 (Carol 1 West) | 45.46 | .004 | .06 | 34.65 | 30 |
| Pit No. 3 (Carol 1 East) | 31.27 | .006 | .12 | 50.44 | 45 |
| Pit No. 5 (Carol 1 East) | <u>38.69</u> | <u>.009</u> | <u>.16</u> | <u>39.42</u> | <u>6</u> |
| Bulk Sample | 37.08 | .006 | .10 | 43.78 | 81 |

We hope that the above will meet your requirements.

With our sincere appreciation for the work which your section will do on our samples,

Yours truly,

IRON ORE COMPANY OF CANADA

J. C. Caron, P. Eng.

APPENDIX 6

Details of Continuous Flotation Test on Carol Lake 2-Ton Shipment

Ore - 2 ton Carol Lake composite sample from Pits Nos. 1, 3 and 5.

Crushing - Single pass through Hazemag crusher.

Screen Analysis of Product -

| | |
|---------------|-----------------|
| +8 m - 1.2% | -28+35 m - 5.1% |
| -8+14m - 4.6 | -35+48 m - 9.3 |
| -14+20m - 3.3 | -48+65 m - 18.6 |
| -20+28m - 3.2 | -65 m - 54.7 |

Grinding - A small scale closed circuit grinding unit was used consisting of a small Hardinge mill and a 48 mesh screen. The feed rate was approximately 275 lb of new feed per hour with oleic acid (Armour NF 94.04) added at the rate of 0.4 lb per ton. This was not a satisfactory arrangement as the Hardinge mill would not discharge the dry product at this rate and the feed had to be cut off frequently with the result that some overgrinding probably occurred. The screen analysis of a composite product sample was:

| | |
|---------------|----------------|
| + 48 m - 0.2% | +150 m - 26.4% |
| + 65 m - 4.7 | +200 m - 18.3 |
| +100 m - 29.1 | -200 m - 21.3 |

Flotation - The flotation circuit was made up of 1 conditioner cell, 3 rougher cells, 1 scavenger cell, 2 cleaner cells and 2 recleaner cells. These were all Agitair cells.

The ground ore was fed at the rate of 100 lb per hour to a Denver vertical pump with water to give a density of 30% solids. The scavenger cell produced a final tailing and a scavenger float product, the latter being returned to the rougher circuit feed pump. The cleaner tailing was partially dewatered (and deslimed) in a small hydroseparator, and returned to the rougher circuit feed pump. The recleaner tailing was recycled to the cleaner circuit. Oleic acid was added to the rougher circuit feed pump, to the scavenger cell, and to the recleaner cells as required. Fluosilicic acid was added to the recleaner circuit, to the cleaner circuit and at times to the rougher feed in an attempt to reduce circulating loads and improve grade.

Grinding was carried out on Sept. 2 and Sept. 3. Due to mechanical difficulties with the new circuit, poor results were obtained on Sept. 4 in continuous flotation.

On Sept. 5, the best results were obtained (on ore ground Sept. 3). For this run operating conditions were:

Rate of new feed - 100 lb per hour
Pulp Density to rougher cells - 25% solids

all dilution water coming with cleaner returns after partial dewatering.

Reagents - Oleic acid - to rougher feed, 0.14 lb/ton
- to scavenger cell, 0.04 lb/ton
- recleaner cell, 0.04 lb/ton
Fluosilicic acid - to recleaner cell, 20 ml per ton

pH - Rougher feed - pH 7
Cleaner cells - pH 6.6
Recleaner cells - pH 6.6

Results of this test were:

| | <u>Assay, % Fe</u> | <u>Weight, % (calculated)</u> | <u>Distribution, Fe %</u> |
|-----------------------|------------------------|-----------------------------------|-------------------------------|
| Feed | 34.70 | 100 | |
| Recleaned concentrate | 64.28 | 46.2 | 85. |
| Tailing | 9.46 | 53.8 | 15. |

The rougher concentrate assayed 42% Fe.

On Sept. 9th, flotation was again tried under the same conditions and with the same feed sample as used on Sept. 5th. Results were much poorer in spite of better mechanical controls. Results were:

| | <u>Assay, % Fe</u> | <u>Weight, % (calculated)</u> | <u>Distribution, Fe, %</u> |
|-----------------------|------------------------|-----------------------------------|--------------------------------|
| Feed | 36.52 | 100 | |
| Recleaned concentrate | 58.16 | 50 | 79 |
| Tailing | 15.30 | 50 | 21 |

It was suspected that the floatability of the ore must have changed during the additional three days it was stored and attempts were made to get better results in the afternoon of Sept. 9. Changes were made in the oleic acid and fluosilicic acid additions. Also the cleaner tailing was completely dewatered and fresh water was used to make up the rougher density. Selectivity remained poor and the best results (maintained for one-half hour) were:

| | <u>Assay, % Fe</u> | <u>Weight, % (calculated)</u> | <u>Distribution, Fe, %</u> |
|--------------------------|------------------------|-----------------------------------|--------------------------------|
| Feed | 35.42 | | |
| Recleaned concentrate | 64.80 | 39 | 71.5 |
| Tailing | 16.50 | 61 | 28.5 |

APPENDIX 7

Test Details of Iron Ore Co. Test 0925Five Cycle Test on Carol Lake Composite Sample -
Dry grind with oleic acid followed by flotation.

Crushing - Hazemag crusher (98.8% passing 8 mesh).
Crushed in laboratory gyratory to pass 10 mesh.

Grinding - Approximately 1000 gram batches, ground dry in
laboratory mill. To each batch grind 10 drops
(0.3 gram) Armour NF 94.04 oleic acid.
Time - 20 min

Screen Analysis - 1.6% on 65 mesh, 51.7% on 100 mesh, 69%
passing 200 mesh.

Flotation:

| <u>Cycle</u> | <u>Stage</u> | <u>Time</u> | <u>Reagents</u> | <u>Remarks</u> |
|--------------|--|-------------|--------------------|--|
| A | Condition | 2 min | 0.02 ml H_2SiF_6 | 40% solids |
| | Rougher float | 2 min | nil | 25% solids in 1000 g Agitair cell |
| | Scavenger float | 1 min | 1 drop oleic | Float product to next cycle rougher stage |
| | Cleaner float | 1 min | 1 drop oleic | In 500 g Agitair cell: Tailing to next cycle rougher stage |
| | Recleaner float | 1½ min | 0.1 ml H_2SiF_6 | Tailing to next cycle cleaner stage |
| B | - as Cycle A but no fluosilicic acid to rougher conditioning. All solutions recycled with scavenger float and cleaner tailing. Only part of solution from recleaner float recycled. | | | |
| C | - as Cycle B. | | | |
| D | - as Cycle C except 0.1 ml fluosilicic acid to cleaner stage. | | | |
| E | - as Cycle B. Scavenger and cleaner products retained for assay. | | | |

Material Balance

| <u>Weight, g</u> | <u>Cycle A</u> | <u>B</u> | <u>C</u> | <u>D</u> | <u>E</u> | <u>Totals</u> |
|--------------------------|----------------|----------|----------|----------|----------|---------------|
| Calc. feed to cycles | 979 | 1179 | 1311 | 1410 | 1376 | |
| Concentrate | 344 | 391 | 447 | 434 | 476 | 2092 |
| Tailing (Scav.) | 435 | 456 | 433 | 577 | 533 | 2434 |
| Scav. Float | | | | | 194 | |
| Cleaner Tlg. | | | | | 65 | |
| Recleaner Tlg. | | | | | 108 | |
| To next Cycle (calc.) | 200 | 332 | 431 | 399 | | (367) |
| | | | | | | <u>4893</u> |

Metallurgy

| <u>Product</u> | <u>Weight, g</u> | <u>Assay, Fe, %</u> | <u>Units, Fe, g</u> | <u>Combined Products</u> | | |
|------------------------|----------------------|-------------------------|-------------------------|--------------------------|-------------------------|---------------------|
| | | | | <u>Wt., g</u> | <u>Units, Fe, g</u> | <u>Assay, %</u> |
| Concentrate A | 344 | 66.0 | 227 | | | |
| B | 391 | 67.5 | 264 | | | |
| C | 447 | 66.5 | 297) | | | |
| D | 434 | 67.2 | 292) | 1357 | 903 | 66.5 |
| E | 476 | 66.0 | 314) | | | |
| Tailing A | 435 | 13.0 | 57 | | | |
| B | 456 | 8.6 | 39 | | | |
| C | 433 | 8.5 | 37) | | | |
| D | 577 | 9.8 | 57) | 1543 | 146 | 9.5 |
| E | 533 | 9.7 | 52) | | | |
| Cleaner tailing E | 194 | 25.6 | 50 | | | |
| Recleaner tailing E | 65 | 36.6 | 24 | | | |

Recovery from combined C, D & E Cycles = 86.1%
 " " " B, C, D & E " = 86.3% at 66.7% Fe grade

Calculated feed to cycles C, D, E = 36.3% Fe

APPENDIX 8

Test Details of Batch Test 1203-1Wet grinding - desliming - high density conditioning and flotation

Sample - minus 14 mesh screened from Hazemag product, Carol Lake composite sample.

Grind - 1000 gram batch ground wet for 20 minutes with tap water only.

Deslimed- by decantation.

Conditioning - 5 minutes with 0.3 g oleic acid, (Armour NF 94.04) at 50% solids.

Rougher flotation - no additional reagent required.

Cleaner flotation - add 0.2 ml fluosilicic acid (2% soln.)

Recleaner flotation - add 0.2 ml fluosilicic acid.

Scavenger float on rougher tailing - 0.03 g oleic acid

All flotation carried out in 1000 g Agitair cell.

Results

| <u>Product</u> | <u>Weight, %</u> | <u>Assay, Fe, %</u> | <u>Distribution, Fe, %</u> |
|-------------------|----------------------|-------------------------|--------------------------------|
| Slimes | 8.4 | 21.8 | 5.3 |
| Final tailing | 26.9 | 6.76 | 5.2 |
| Scav. Conc. | 15.4 | 23.9 | 10.6 |
| Cleaner tailing | 8.8 | 21.6 | 5.5 |
| Recleaner tailing | 6.0 | 45.2 | 7.8 |
| Cleaned Conc. | <u>34.5</u> | <u>66.0</u> | <u>65.6</u> |
| Feed (calculated) | 100.0 | 34.7 | 100.0 |

APPENDIX 9

Test Details of Locked Test 1218Test Procedure - 4 cycle locked test

Feed - Minus 14 mesh screened Hazemag product from Carol Lake composite.

Grind - Approximately 1000 g batches ground wet for 15 minutes with no reagent.

Desliming - by decantation repeated twice.

Conditioning - At 50% solids in Agitair 1000 g cell at 1000 rpm, 3 minutes.

Reagent: 0.48 lb/ton oleic acid (Armour NF 94.04)

Rougher flotation - at 25% solids, no additional reagents other than reagents returned to subsequent cycles in middling products.

Scavenger flotation - refloating rougher tailing with an additional 1 drop oleic acid (0.06 lb/ton). Scavenger concentrate returned to next rougher cycle.

Concentrate cleaning - rougher concentrate cleaned twice with 10 ml fluosilicic acid* to each stage. Cleaner and recleaner tailings returned to rougher and cleaner float, respectively.

* fluosilicic acid fed as 2% solution of reagent grade H_2SiF_6 , 10 ml approx. equals 0.5 lb/ton of the 30% commercial soln.

APPENDIX 9 (cont'd)

Results

| | <u>Cycle A</u> | | <u>Cycle B</u> | | <u>Cycle C</u> | | <u>Cycle D</u> | |
|-------------------------------------|----------------------------|-------------------------------|----------------------------|-------------------------------|----------------------------|-------------------------------|----------------------------|-------------------------------|
| | <u>Weight,</u> <u>g</u> | <u>Assay,</u> <u>Fe, %</u> | <u>Weight,</u> <u>g</u> | <u>Assay,</u> <u>Fe, %</u> | <u>Weight,</u> <u>g</u> | <u>Assay,</u> <u>Fe, %</u> | <u>Weight,</u> <u>g</u> | <u>Assay,</u> <u>Fe, %</u> |
| New feed to cycle | 1060 | | 1060 | | 1060 | | 1060 | |
| In cycle | 1060 | | 1358 | | 1421 | | 1521 | |
| Concentrate | 335 | 67.6 | 274 | 68.1 | 336 | 67.8 | 550 | 66.0 |
| Tailing | 367 | 9.9 | 663 | 12.1 | 564 | 14.5 | 374 | 8.56 |
| Slimes | 60 | 22.0 | 60 | 22.0 | 60 | 22.0 | 60 | 22.0 |
| To next cycle (circulating load) | 298 | | 361 | | 461 | | 537 | |
| Scavenger conc. | | | | | | | 248 | 29.28 |
| Cleaner tailing | | | | | | | 166 | 27.36 |
| Recleaner tailing | | | | | | | 125 | 46.52 |
| <u>Iron Units, g</u> | | | | | | | | |
| In concentrate | 226 | | 187 | | 228 | | 363 | |
| In tailing incl. slimes | 49 | | 93 | | 95 | | 45 | |
| | <u>275</u> | | <u>280</u> | | <u>323</u> | | <u>408</u> | |

Total recovery in 4 cycles (disregarding circulating load remaining in cycle D)

= 78%