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THE CONCENTRATION OF FLAKE GRAPHITE ORES.

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Introductory

The recent revival of interest in graphite due to improved market conditions has prompted the writing of this article. The history of graphite mining in Canada should be carefully studied by any company contemplating entering this field. Many failures in the past have been due to inefficient methods of concentration, but a number have resulted also from the erection of extensive milling and plant equipment before the extent and grade of the ore bodies were determined; and from insufficient knowledge of the market and trade requirements.

Types of Canadian graphite ores: Two main classes of graphite ores are known to occur in Canada: disseminated crystalline flake graphite, and the more or less massive crystalline graphite which occurs in veins or pockets. The deposits of the latter variety which is commonly called "crystalline graphite", have never been considered commercially important, and for this reason their concentration will not be discussed in this article.

In Canada the average run-of-mine ore of the flake variety will contain 10 to 15 per cent graphite; the average American ore of this type contains only between 3 and 7 per cent.

Factors governing the value of a graphite ore: In determining the value of a graphite ore, granted that there is a sufficient tonnage available, the governing factor is the size of the flake. Since there is a ready market only for large flake, the value of the ore depends on the amount of No. 1 and No. 2 flake recoverable rather than on the total graphite content of the ore. It is possible that a 10 per cent ore may be more valuable than a 15 per cent ore if more No. 1 and No. 2 flake

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+ Engineer, Division of Ore Dressing and Metallurgy can be recovered from it. However, certain ores may contain more large flake than others but still be of less value, due to the hardness and nature of the associated gangue. A hard siliceous gangue is difficult to free, causing a large amount of flake to be ground up in the process of separation.

Concentration: Before the introduction of froth flotation, the concentration of graphite presented many difficulties. It may be conservatively stated that the losses in the tailings exceeded 50 per cent. The actual production of a high grade graphite concentrate by flotation would be a simple matter, as it is one of the most easily floated minerals, were it not for the fact that the trade demands certain qualities, and pays the highest price for No. 1 flake (which is flake remaining on a 90 mesh screen). The finer sizes ha ve a limited market and at most times it is impossible to dispose of them at any price. The main problem is, therefore, the design of a flotation plant that will destroy as little as possible of the large flake in the ore. The efficiency of the mill will depend more on the amount of large flake produced than on the total recovery of graphite

Refining: It can be readily understood that there is a point beyond which it would not be practical to raise the grade of a concentrate produced by continuous operation in a concentrating mill. Therefore, to determine when concentration should be stopped, and refining in batch lots resorted to, is essential. In the refinery each operation is under exact control and the size of the flake is more easily preserved

General principles in design of concentrating mill: There are a few general principles which should be kept in mind when designing a flow sheet for a mill to concentrate graphite ore. The ore should be crushed only to the point where it is possible to obtain a reasonably clean tailing. The concentrate produced by the primary flotation should contain a minimum of free gangue. In re-cleaning this concentrate the middling products will consist of true middlings and particles of gangue which have become mechanically entangled with the concentrate. The greater part of the entangled gangue can be eliminated by operating the first cleaner cells in closed circuit with the roughers. The flow sheet should be flexible enough to grade the true middlings into two general classes. These two classes are: first, particles of gangue to which are attached small pieces of graphite; second, particles of graphite to which are attached smaller pieces of gangue. These two classes naturally grade into each other. Just where the division between these two classes should be made will depend on final results in the refining department. After the division has been effected, the first class should be returned to a separate grinding circuit, and the second class treated as final concentrate and delivered to the refinery, where the work of freeing the attached pieces of graphite can be effected more efficiently

Flow sheet of an all-flotation plant: A flow sheet of a 200-ton mill which operated three years on a hard 7 per cent ore is shown as Figure I. This mill was one of the first flotation plants to treat a hard siliceous graphite ore. Flotation replaced a system of buddle concentration. A study of this flow sheet will show that the entire concentrate from the flotation cells is passed through two pebble mills for re-grinding

When the mill was first built only one re-grinding mill was used, but the additional recovery of graphite overloaded the capacity of the refining plant, and an agitation was immediately started by the refinery superintendent to have a higher grade product delivered to the refinery. A second re-grinding pebble mill was then installed. The graphite from this mine was used entirely for lubricating purposes, and even a grade of 88 per cent carbon had to be further refined.

A series of curves is given in Figure II, showing a comparison between the buddle system, flotation using one re-grinding mill, and flotation using two re-grinding mills. These curves give the number of pounds of pure graphite (carbon) recovered from a ton of ore; the weight of concentrate recovered from a ton of ore; the grade of concentrate and percentage of carbon contained; and the unit cost per pound of concentrate produced. A comparison between the use of buddles and flotation with reference to the amount of +60 mesh pure graphite (carbon) is given in Table I.

TABLE I

A comparison of average results obtained on a 6 to 7 per cent hard graphite ore

	Buddles	Flotation - one re-grinding mill	Flotation - two re-grinding mills
Pounds of concentrate recovered from one ton of ore	45	120	116
Per cent recovery on 60 mesh screen	60	43	25
Per cent carbon content of +60 mesh product.	60	73	88
Pounds of pure graphite recovered, +60 mesh	16	35	25.5

The flow sheet used in this mill (Figure I) is not in accordance with the principles previously given. The clean coarse flake should have been separated from the concentrate before passing to pebble mills for re-grinding. One of the most practical methods of doing this is to table the concentrate first. This flow sheet is given to illustrate how readily any additional grinding will destroy the coarse flake, and to draw attention to the remarkable improvement in recovery and lowering of costs by flotation.

Flow sheet of plant using flotation & tabling: A flow sheet, Figure III, is given of a mill in which tables were used on the flotation concentrate. In this case the sands and part of the middlings were run to waste in the table tailing, instead of being re-ground and returned to the circuit. The grade of concentrate delivered to the refinery depended largely on the operation of the tables, so that in order to raise the grade it was necessary to turn more graphite into the table tailing, thus causing a loss of considerable large flake. The recovery was low, probably not exceeding 75 per cent, but this was offset by the large proportion of No. 1 crucible flake recovered.

It will be observed from the flow sheet that the flotation concentrate was not re-ground as in the previous one, so that although the recovery was low, the proportion of No. 1 crucible flake produced was large and the refinery actually produced 70 per cent No.1 flake of a grade exceeding 92 per cent carbon.

Theoretical <u>flow sheet:</u> A flow sheet, Figure IV, has been worked out according to the general principles of design given. This flow sheet starts with grinding in closed circuit with either an Akins or Dorr type classifier, the grinding being carried only to the point where clean tailings can be made from the rougher cells. There is a possibility that it would be practical at this stage to table the classifier returns to prevent the coarse flake contained in the oversize again passing through the ball or rod mill. The rougher concentrate is cleaned once in a cleaner cell and the cleaner tailing returned to the rougher feed. The cleaner cell should be operated to drop only entangled sands. The concentrate from the cleaner cell goes to tables which as far as possible should be operated to separate only the sand particles containing relatively small flakes of graphite. The concentrate from the tables is dewatered and sent to a second cleaner cell, while the table tailing goes to a pebble mill for re-grinding. The second cleaner cell will drop some coarse free flake as well as flake of various sizes with attached pieces of gangue. The second cleaner tailing is then tabled and a finished concentrate produced. By this tabling operation the clean coarse and fine flake dropped from the flotation cell is recovered and not returned for further grinding in the pebble mill, whereas the flake of various sizes containing attached gangue is sent to the pebble mill for re-grinding. The second cleaner flotation concentrate is again tabled; this tabling may not be actually necessary, but it acts as a safeguard against the inefficient operation of the flotation cells. The discharge of the re-grinding pebble mill is tabled, and a finished concentrate made, and the sands and entangled particles of graphite and gangue are returned to the head of the rougher cells.

It is believed that a flow sheet designed along these general lines will give the most efficient results on flake graphite ores. On soft and easily disintegrated ores it will not be necessary to carry the flotation and tabling so far. The particular advantage claimed for this flow sheet is that besides eliminating the coarse flake from the circuit as soon as it is freed, the fine flake produced in the refinery will be of a much higher grade than that obtained from the two flow sheets previously given.

Interpretation of <u>small scale tests:</u> The correct interpretation of the results of small scale laboratory test work requires explanation. The scope of this paper does not allow a lengthy discussion on this subject, but it has been thought advisable to draw attention to the great difference in results obtained by crushing in small laboratory ball mills and crushing in large standard sized mills. One example will be given to illustrate this difference. An exhaustive number of laboratory tests were run on a certain ore to ascertain the quantity of large flake that could be recovered. These tests showed a recovery of more than 50 per cent of the flake on 60 mesh screen, the product assaying 85 per cent carbon. The results from the mill designed from these tests showed a recovery of only 30 per cent on 60 mesh, assaying 85 per cent carbon. This large difference was due to the action of grinding in large ball mills.

Some notes on the types and operation of machinery <u>in most general use:</u>

Grinding: The ball mill is in general use, and there is little doubt that this is the most practical and satisfactory machine for the final reduction of the ore. The conical type of mill is most favoured because it is claimed that the flake when freed floats out of the mill more quickly than with the cylindrical types. However, the writer has seen equally as good results obtained in the ordinary cylindrical mill when used with either a diaphragm or open trunnion discharge. The lubricating nature of graphite has been found to reduce the rated capacity of a ball mill 25 per cent, a factor that should be taken into consideration when determining the size of mill to install. From extensive tests carried out by the writer it was found that less flake was destroyed by grinding in a dilute pulp of from 45 to 55 per cent solids (as much as 10 per cent additional flake reporting on a 60 mesh screen) The rod mill is suggested as possibly giving more efficient grinding than the ball mill. The Ore Dressing and Metallurgical Division of the Mines Branch contemplates carrying out a number of comparative tests with the rod mill and ball mill.

Closed circuit grinding with classifier and ball mill: An excess of water must be avoided in order to obtain efficient results in a flotation plant. Hydraulic classifiers should not be used in the circuit. It has been claimed that they are better than the Dorr or Akins types for preventing the return of the large flake with the oversize to the ball mill, but it has been found in three different mills that any advantage gained in this way is offset by the increased loss of coarse flake in the flotation tailing, due to high dilution of the pulp in the cells.

Flotation machines: The Callow pneumatic cell has been more extensively used than any other for the flotation of graphite, but there is no reason why just as satisfactory results cannot be obtained from any other standard type of machine. The type of Callow cell found to be the most satisfactory for graphite is the short 8 by 2 feet cell with sloping bottom. A short narrow flat bottom cell of the same size should also work well. The long 19 by 3 feet flat bottom cell or the 40 by 3 feet cell, are not recommended for the average Canadian flake ore.

In the operation of a pneumatic type of cell it has been found advantageous to use a system of baffling in the cell. The sides of the cell are built up with boards to a height of 12 to 14 inches, and a series of three vertical cross baffles of different heights are built in the cells so that the froth decends in cascades and flows through an outlet. In the rougher cell, the outlet is at the feed end, the side boards being cut away 12 to 20 inches from that end, allowing the froth to discharge over the sides. In the cleaner the froth is carried direct through an opening cut in the front or discharge end of the cell. These baffles are carried down to within 6 inches of the blankets and are arranged on top so that there is approximately a 3 to 4 inch drop between each baffle. The use of the baffles makes the operation of the rougher cell much simpler, and by their use in the cleaner cell, the grade of the concentrate can be raised 5 to 10 per cent.

Flotation Reagents: Kerosene and steam distilled pine oil are the two oils which should be used. The use of kerosene, however, requires careful manipulation, as the least excess will tend to kill the froth in the cells. For this reason some operators have had trouble using it, but the writer has operated with it for a number of years and never experienced trouble. Hardwood creosote oil has been used along with the kerosene to overcome the raw oil effect of the kerosene, but its use is not to be recommended, as it is accompanied by a greater loss of graphite in the tailing. Additional reagents such as lime and soda ash are seldom necessary.

Tables: Any table made by a reputable manufacturer should be satisfactory. Some operators use fine silk botling cloth over the launder along the low sides of the table, for dewatering the graphite concentrate. The frame holding the silk is attached to the side of the table so that the bumping action of the table keeps it free. In most cases the riffling of the table will have to be altered to take care of the type of feed to the table.

Filters and dewaterers for the concentrate: Continuous suction filters have not as a rule been satisfactory, due to the physical character of the graphite. In most cases it has been found impossible to reduce the moisture content of the concentrates below 30 per cent. The lubricating qualities of graphite, together with the tendency of the thin flat flakes to seal the pores of the filter cloth prevent a cake from being built up and held in the filter until the proper time for discharging. A number of more recent tests made by the United Filters Corporation on a sample of concentrate from one of the Buckingham mills have shown more favourable results. The results of these tests are as follows:

Test No.	Pulp ratios by weight.	Vacuum on cake inches	Vacuum on dry inches	Moisture in cake per cent	Cake thickness inches	Capacity, sq.ft.per hour dry graphite
1	3 to 1	18	18	20.9	2	216
2	8 to 1	11	16	20.5	$1\frac{1}{4}$	36
3	20 to 1	15	18	20.5	5/8	24
4	16 to 1	8	17	20.1	9/16	16
5	5 to 1	5	18	20.6	7/8	42.5
6	10 to 1	21	20	22.5	7/8	60
7	20 to 1	21	21	24.5	3.8	23.8

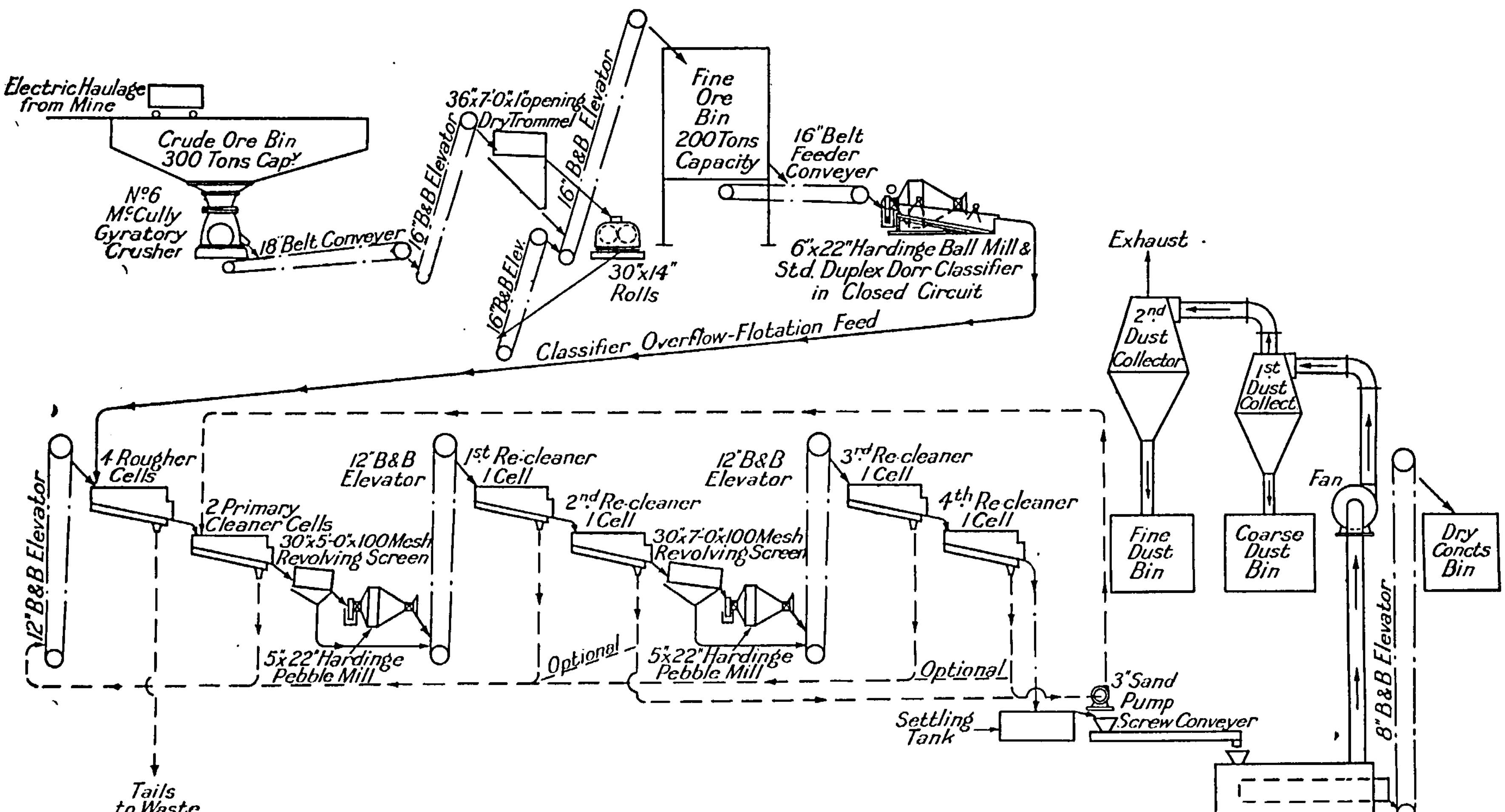
In each test, with the exception of No. 4, the pulp in the filter was air agitated. The square feet per hour capacity of the filter varies, due to the speed at which the filter is revolved, the vacuum at which the cake is built, and the pulp ratio. It has been suggested that if the +90 flake were screened out before filtering, much better results could be obtained. The suggestion is practical, and it would appear that better results can be expected.

The system most generally used is a series of settling tanks which are allowed to fill alternately. After settling, the water is decanted and the graphite allowed to drain through a diaphragm of burlap, or other porous material. There is no reason why a Dorr thickener cannot be used. The only objection to the Dorr tank that the writer has heard, is that a heavy froth forms on the top of the tank, but this can be taken care of by building a high retaining ring inside the tank and by occasionally skimming off this froth, which in some cases consists of very high grade fine flake.

Driers: The type of drier commonly used is the direct fired rotary. There are two disadvantages to a drier of this type: contamination by ash from the fuel, and the dust loss. The dust loss can be avoided by the use of dust collectors. In one instance, the installation of a dust collector saved 1,200 pounds of graphite a day. Indirect types of driers, such as the Lowden, or Ruggles-Cole, although the first cost is high, are probably the more satisfactory.

Finishing plant: The process of refining must be developed for the particular concentrate to be treated. The most important factors which will affect the treatment scheme to be used are such physical characteristics as, diameter, thickness, hardness, and toughness of the flake, and the nature of the impurities to be removed, whether soft or hard. Coarse, hard impurities are more difficult to handle and the amount of work the refinery should do on this class of product should be balanced against the amount of work which can be done effectively in the concentrating mill. A good deal of information has come to the writer's attention pertaining to different methods of refining, but the information obtained is not considered reliable enough to draw definite comparative conclusions. One large American company which has been in the graphite industry from the beginning, uses a system of batch ball mills and dry screening. A large successful Canadian company uses burr stone mills and dry screening. Both claim they have tried other methods, and that their present practice has proven the best. The writer favours the use of the burr stone mill. Both the operation of the ball mill and the burr stones must be in the hands of experienced and capable men, as the personal element governs the efficiency of the operation in both cases. An experienced operator will recover 50 per cent more No. 1 flake than an inexperienced one.

The writer recommends that when considering a new installation for a finishing plant, a simple system of burr stones and screens of the Hummer type be tried at first. Any elaborations on this system can be developed gradually as experience with the particular ore shows the way. It will be found advisable to house the finishing plant and drier in a separate building from the concentrating mill, on account of the dust from the dry process of finishing.



Flow-Sheet - Fig. 1

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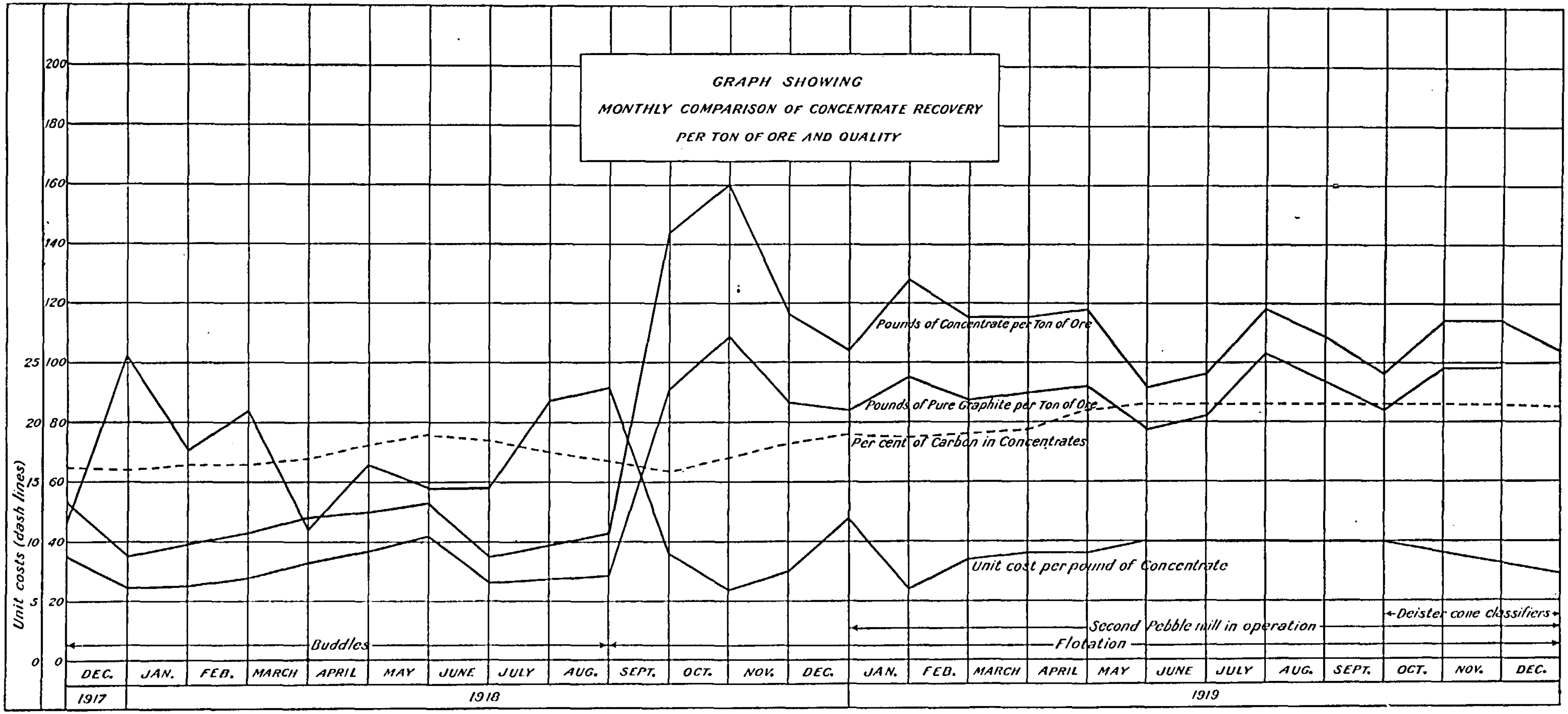

GRAPH SHOWING
MONTHLY COMPARISON OF CONCENTRATE RECOVERY
PER TON OF ORE AND QUALITY
Pounds of Concentrate per Ton of Ore
Pounds of Pure Graphite per Ton of Ore
Per cent of Carbon in Concentrates
Unit cost per pound of Concentrate
Unit costs (dash lines)
Buddles
Flotation
Second Pebble mill in operation
Deister cone classifiers
0
20
40
60
80
100
120
140
160
180
200
5
10
15
20
25
DEC.
JAN.
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MARCH
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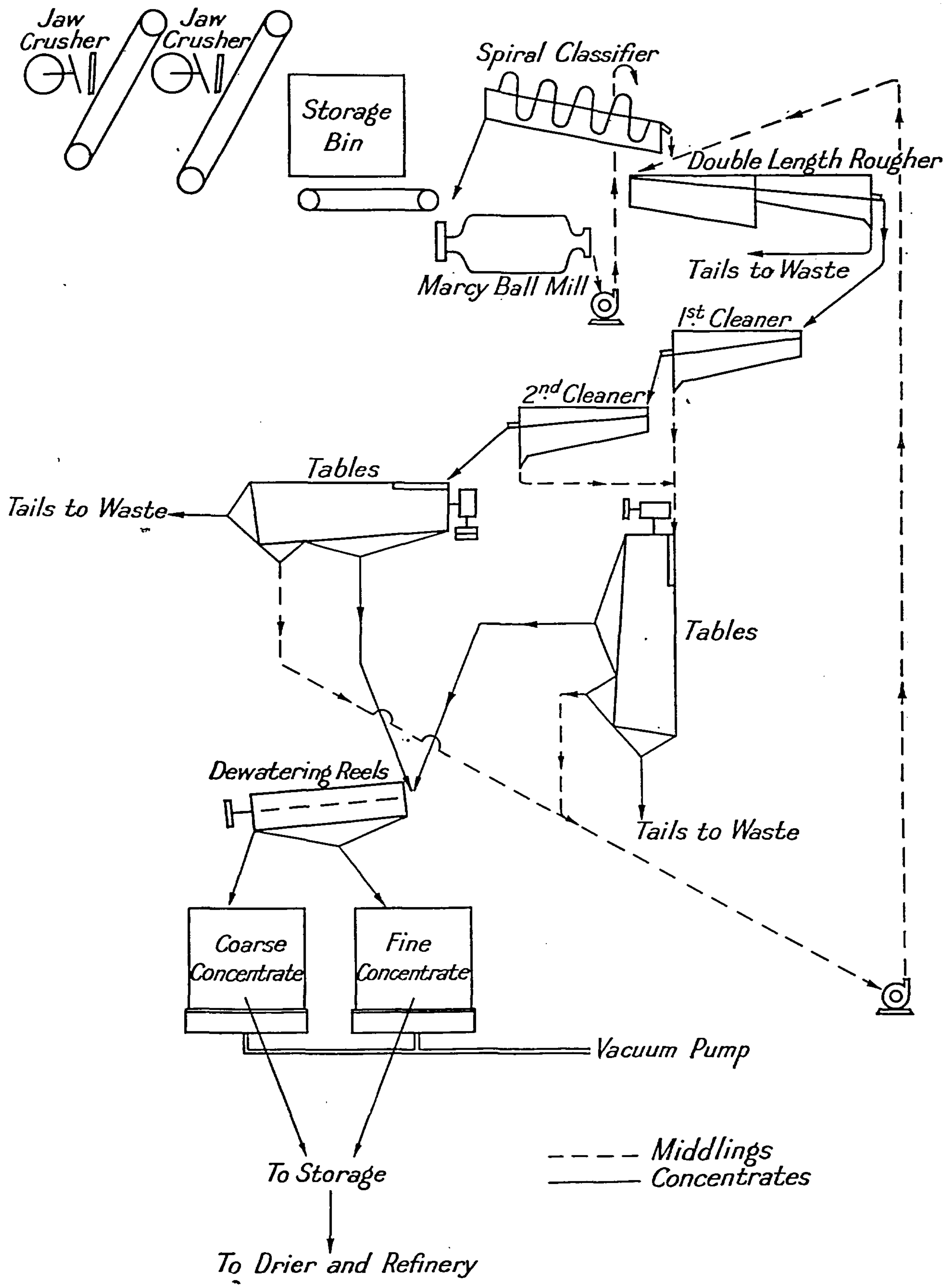
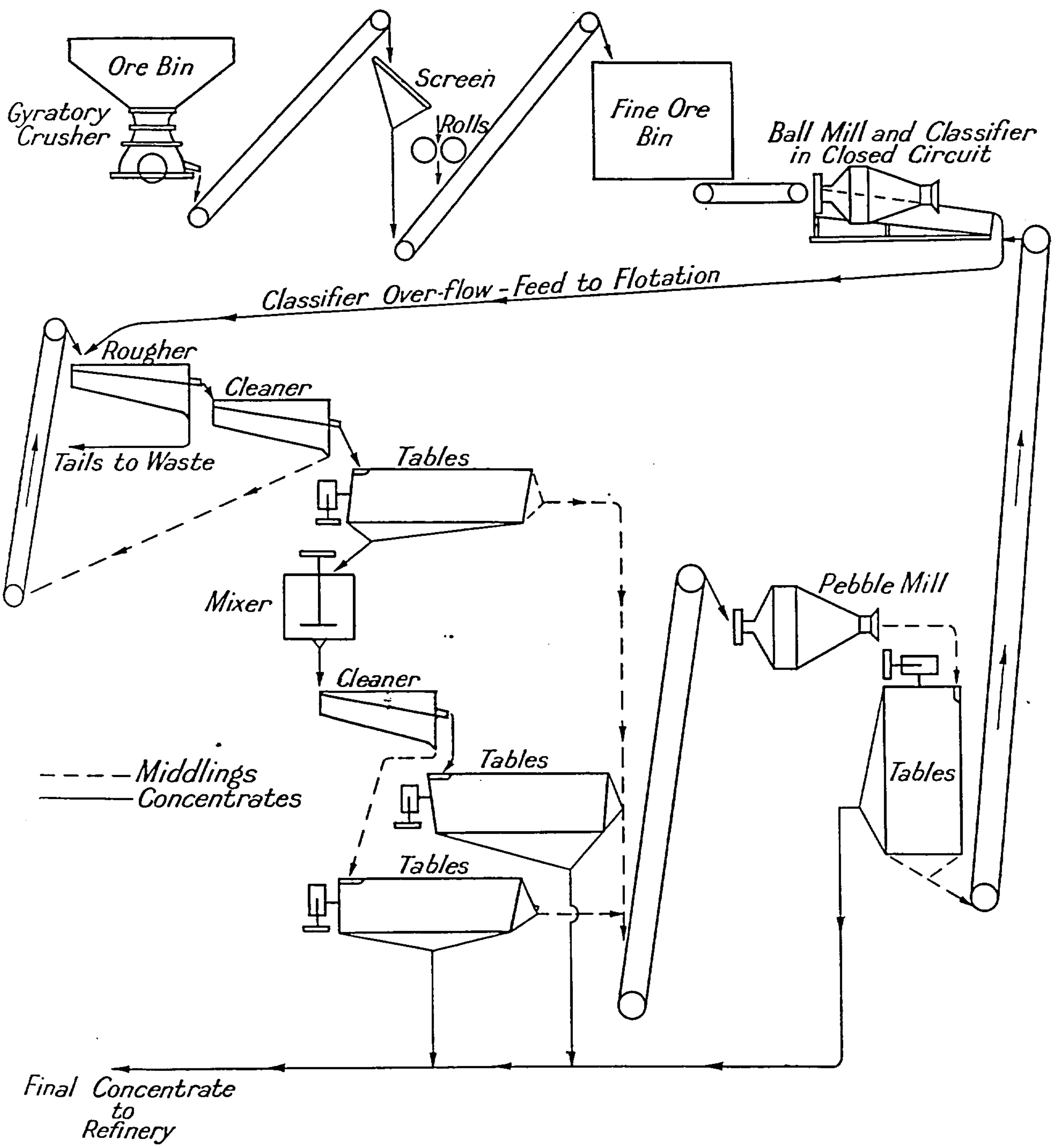
Figure 2



Flow-sheet - Fig. 3



Flow-sheet - Fig. 4