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OTTAWA July 20, 1945.

REPORT

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1902.

Retreatment of Molybdenite Concentrate from the La Corne Molybdenum Project of the Wartime Metals Corporation, Val d'Or, Quebec.

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(Report of Investigation No. 902)

FOREWORD

The reader of this report should understand that it is not confined to discussion of a single metallurgical problem. Originally these Laboratories were asked to work out a method for eliminating the bismuth contained in La Corne Molybdenum concentrates as an impurity. It was found that the bismuth content could be lowered to fit the required specifications by a process of flotation and fractional roasting. The success of this procedure immediately suggested the possibility of making a bismuth concentrate, and of devising a more efficient milling procedure.

The report covers, therefore, a discussion of these three related problems. They are:

1. A method for producing a clean molybdenite concentrate.
2. Production of a bismuth concentrate.
3. Improvements in milling practice that will result from use of a rougher-retreatment flow-sheet.

Production of a clean molybdenite concentrate is only feasible by a process of flotation and fractional roasting. Reasons for the failure of other methods (gravity concentration, leaching, screening, and selective flotation) are discussed on pages 3 to 9. This part of the report is included to show the inadvisability of further work on these lines.

Production of a bismuth concentrate is comparatively simple, since this by-product is collected into an acceptable concentrate in the tailings from the molybdenite cleaning process (pages 12 to 18). That the bismuth in this concentrate may be recovered by leaching is pointed out on pages.

10 to 12.

Improvements in milling that will result from use of the flow-sheet described here are discussed on pages 19 to 28. This section also includes an elaboration of some of the problems involved, such as fractional roasting, which are not apt to be immediately understood by the reader lacking experience with this milling scheme. On page 23 it is pointed out that, fortunately, the mill equipment now on hand is sufficient to make up the new flow-sheet, with the purchase of a small amount of new machinery.

Flow-Sheet No. 1 represents the best milling procedure for a retreatment plant. Three other flow-sheets are included, to show how certain refinements may be introduced, if found advantageous, at a later date.

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Ottawa, Ont.,
July 20, 1945.
FKMcK:LB.
(Foreword to Report of
Investigation No. 1902).

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Retreatment of Molybdenite Concentrate from the La Corne Molybdenum Project of the Wartime Metals Corporation, Val d'Or, Quebec.

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Shipments:

A shipment of molybdenite concentrate, weighing approximately 1,000 pounds, was received at these Laboratories on December 11, 1944, and a second one, of two tons, was received on March 23, 1945. These shipments were submitted by Mr. G. Shartner, resident manager of the La Corne Molybdenum Project, Val d'Or, Quebec, on the advice of Mr. H. P. Dickie, General Manager, Wartime Metals Corporation, 633 Craig St. West, Montreal, Quebec.

This material was reported to have been selected from regular mill-run concentrate. Representative samples from each lot gave the following analyses:

	Weight, pounds	MoS_2, per cent	Bismuth, per cent	Copper, per cent
Shipment No. 1 -	1,000	86.98	2.75	0.61
Shipment No. 2 -	4,000	88.07	2.54	0.58

Purpose of Investigation:

Tests were made on this material in order to develop a process for eliminating the impurities (e.g., bismuth) from the concentrate. A secondary consideration, though of equal importance if post-war operation of the mine is contemplated, was the necessity for raising the grade above that now produced at the mill.

The problem may be resolved into three requirements, which are noted here in the order of their importance. Study of several methods of refining the concentrate was discontinued after the work had progressed far enough to show that these three main points could not be satisfied. These points are:

1. The bismuth content of the molybdenite concentrate produced must be below 0.5 per cent, and the concentrate itself should be over 90 per cent MoS_2 in grade.
2. Good recoveries of molybdenite and bismuth are essential, as both products sell at a high price per pound.
3. Expenditures on equipment must be kept to a minimum, as good practice would dictate in a small operation like this.

SUMMARY OF INVESTIGATIONS:

A review of the chemical and physical properties of the two minerals to be separated, molybdenite and bismuthinite, discloses several differences in the characteristics of the two which offer theoretical possibilities of a solution for the problem. Some of these possibilities (gravity concentration, leaching out bismuth, etc.,) yield disappointing results upon actual trial, and a close study supplies the reasons for the failure. Earlier work on the La Corne concentrate, carried only to the point of theory, had indicated procedures which

(Summary of Investigations, cont'd) -

we now know do not apply.

Below will be found details of the investigations and a discussion of those methods which are satisfactory.

1. - Gravity Concentration.

There is a wide differential between the specific gravities of the two minerals--molybdenite, 4.7; bismuthinite, 6.4. Ordinarily this would be sufficient to make possible a highly satisfactory separation by gravity methods, but in the present case test work indicates that no satisfactory result can be obtained. Under the microscope, it is found that bismuthinite grinds into angular, chunky pieces that are easily pushed sidewise by the stream of wash water across the concentrating table. A portion of the molybdenite leaves the mill in flat plates, which, because of their shape, cling to the table and offer very little resistance to the cross-stream of the wash water. Thus the grains of molybdenite push forward into the concentrate band, lowering the grade of the bismuthinite, and disturb it so it is more easily washed down to the tailings side of the table.

Another point in the failure of gravity methods is the fact that bismuthinite is very friable in the grinding circuit. Concentrating tables do not work well on minus 100 mesh material, especially with unclassified feed. In this case, only 0.18 per cent bismuth reports in the plus 100 mesh portion of the table feed. A table will give results worth consideration if over-sliming of the sulphides can be avoided in grinding. Some suggestions along this line will be discussed later.

A concentrating table, when working under special conditions, will produce a satisfactory bismuth concentrate, as shown in Flow-Sheets Nos. 3 and 4.

(Continued on next page)

(Summary of Investigations, cont'd) -

2. - Selective Flotation of Molybdenite from Bismuthinite.

Investigations here and at Val d'Or plant show that, to an appreciable degree, molybdenite enters a flotation froth more readily than does bismuthinite. This difference is not sufficiently great, however, to warrant hope that acceptable concentrates and recoveries could be made in a milling process based on it alone.

Attempts at depressing bismuth with special reagents in the flotation circuit were likewise unsuccessful. The flotation characteristics of molybdenite and bismuthinite are so nearly identical that there is little hope of arriving at a solution in this manner. Taking a broad view of milling at La Corne, research along this line is seen to be quite useless. Depressing the bismuth into mill tailings would mean that it would be wasted; the possibilities of an income to be derived from sale of this as a by-product are too enticing to warrant elimination of the bismuth in such a way.

Table I shows results from tests using cleaner cells, depressing agents, and fractional roasting to depress bismuth.

- Discussion of Table I.

In Table I are reported results from some of our small-scale work on the problem. The first two tests, 8R and 9R, show that only a part of the bismuth may be depressed in four cleaner cells, with a low recovery of the MoS_2. Tests 10R, 11R, L30 and 2B show the ineffectiveness of depressing agents. Many other tests have been made, especially with cyanide, trying to depress bismuth

(Table I appears on next page. Text continues on Page 6.)

TABLE I. - RESULTS FROM TESTS USING CLEANER CELLS, DEPRESSING AGENTS AND ROASTING TO DEPRESS BISMUTH IN THE LA CORNE MOLYBDENITE CONCENTRATE.

Test No.	Depressing Agent	FEED: Roasting Temp., ° C.	FEED: Assays, per cent MoS_2	FEED: Assays, per cent Bi	MoS_2 CONCENTRATE: Weight, grams	MoS_2 CONCENTRATE: Bismuth, per cent	TAILING: Weight, grams	TAILING: Bismuth, per cent	Remarks
8R	Lime.	No roast,	85.66	2.89	319.5	0.93	174.6	3.37	Reagents: kerosene and pine oil. Cleaned 4 times in flotation cell.
9R	"	" "	85.66	2.89	441.3	1.74	537.7	2.95	" " " "
10R	Nacconol SP.	" "	69.86	2.45	424.7	1.44	559.3		" " " "
11R	Nacconol NRSF.	" "	69.86	2.45	397.7	1.83	576.7		" " " "
L30	Sodium cyanide.	450° C., 45 mins.	85.66	2.89	1,348.0	0.50	528.0	13.12	Cleaned 3 times. Kerosene and pine oil.
2B	" "	300°, 25 mins.	90.65	2.25	373.0	1.24	29.2	10.71	Cleaned twice. Kerosene and pine oil.
1R	Lime.	300°, 25 mins.	85.66	2.89	366.3	0.50	126.9	7.90	Cleaned 3 times. Kerosene and pine oil.
2R	"	325°, 25 mins.	85.66	2.89	380.4	0.69	107.9	7.86	" " "
3R	"	375°, 25 mins.	85.66	2.89	402.4	1.44	88.5	7.22	" " "
4R	"	345°, from Herreschoff.	69.86	2.45	1,380.0	1.14	587.5	5.92	" " "
5R	"	" " "	69.86	2.45	1,544.0	0.71	416.5	8.58	" " "
6R	"	300°, " "	69.86	2.45	1,301.0	0.24	676.0	7.44	Cleaned 3 times. Kerosene and ~~B23~~. pine oil.
13R	"	270°, " "	69.86	2.45	453.8	Trace.	563.3		Cleaned 4 times. Kerosene and B23. ~~pine oil~~.

(Summary of Investigations, cont'd) -

both from the concentrate and from the mill discharge. These have been uniformly unsuccessful, except that lime apparently has a depressing action. This, it is believed, is due more to the fact that lime depresses the slime fraction of the pulp, which contains most of the bismuth, than to any selective depressing action on bismuthinite.

Tests 1R to 6R show flotation results after roasting to various temperatures. Test 13R indicates that B23 frother produces a cleaner MoS_2 concentrate than does pine oil.

3. - Fine Size of Bismuthinite in MoS_2 Concentrate.

Since 70 per cent of the bismuthinite in this material reports with the minus 200 mesh fraction, it was considered worth while to try to isolate this part of the concentrate by screening through 200 mesh. The result was discouraging: 46.8 per cent of the concentrate reported in the minus 200 mesh portion. These fines assayed 86 per cent MoS_2 and 5 per cent bismuth. It was concluded that the only possible application of a screening process might be to separate the plus 100 mesh portion of the material for regrinding.

4. - Leaching.

Since bismuthinite is readily soluble in a weak solution of hydrochloric acid, while molybdenite is not soluble to any large degree, a leaching process will lower the bismuth content to fit the desired specifications. The concentrate must be roasted before leaching.

There are many objections to this process that raise grave doubts as to its suitability. However, it has been studied, and will be discussed at greater length in a subsequent section of this report.

5. - Fractional Roasting.

It was found that a satisfactory separation by flotation could be made, provided the concentrate is first subjected to a low-temperature roast. Roasting has the effect of slightly oxidizing or sulphatizing the surface of the bismuthinite, while the molybdenite is unchanged. This alteration of the surface virtually destroys the floatability of the bismuthinite, making it possible to float the molybdenite into a clean concentrate, while the bismuthinite goes into the flotation tailing.

Working on a feed such as is provided by the mill concentrate from La Corne, by this process a concentrate containing 92 to 95 per cent MoS_2 with less than 0.3 per cent bismuth, can be made, while the tailings contain from 15 to 22 per cent bismuth. Around 98 per cent of the molybdenite in the feed to this retreatment process is recovered, and about 90 per cent of the bismuth is gathered in the tailing.

This is the only process which we can recommend as a solution for the problem, after considering the technical and economical aspects.

DETAILS OF INVESTIGATIONS:OF INVESTIGATIONS

A. - LEACHING.

Leaching bismuth out of this concentrate is not effective unless the material has been roasted. The resistance to leach solutions in the unroasted material seems to be caused by the flotation reagents, which form a protective coating over the particles. Roasting also probably makes the bismuthinite more easily soluble because it is thus oxidized to some extent. This latter point seems to be rather important, for the performance of the leaching process is only slightly improved if the reagents are first driven off by steaming the concentrate.

The most effective leaching solution, so far as our experiments have been able to discover, is a mixture of hydrochloric acid and ferric chloride, 10 per cent HCl and 8 per cent Fe_2Cl_3. After having been roasted to 400° C., the bismuth content may be lowered from 2.92 per cent to 0.48 per cent by two leaches, 40 hours and 16 hours respectively, changing the solution each time.

From an operational point of view, leaching is an undesirable process. A special acid-resistant filter would be required if countercurrent decantation were contemplated. The slime fraction of this material is difficult to settle in an acid solution, a fact that practically eliminates consideration of anything but a batch leaching process. This would require a very large amount of equipment to recover a small amount of valuable material. It is doubtful that the costs could be kept low enough to make a profit on the leaching.

The strongest objection to a leaching process is that by this means the grade of the MoS_2 concentrate would not be raised materially, and the appreciable percentage of copper in the material would not be removed. If pre-war marketing conditions are resumed, it will be found that the copper content of an MoS_2 concentrate, as well as the bismuth, must be below 0.5 per cent. Leaching out the two or three per cent

(Leaching, cont'd) -

bismuth would have the effect of slightly improving the grade of MoS_2 but would at the same time increase the percentage of copper to a point that would make for difficulty in marketing. At present there is from 0.5 to 0.6 per cent copper in the La Corne concentrate, which is already too high according to peacetime specifications.

In view of these objections, leaching as a possible solution is pretty well eliminated. However, it might be useful as a process for treating the bismuth concentrate from the flow-sheets we are about to deal with.

TABLE II. - Leaching MoS_2 Concentrate.

No.	Sample	Assays Before Leaching, per cent		Assays After Leaching, per cent		Extraction of Bismuth, per cent
		MoS_2	Bi	MoS_2	Bi	
1.	As received	86.80	2.80	87.50	1.00	64.3
2.	As received	87.70	2.75	88.65	0.95	65.46
3.	Steamed	86.60	2.92	88.34	0.80	69.18
4.	Roasted, 400° C.	88.60	2.92	89.65	0.85	70.89
5.	Roasted, 400° C.	88.60	2.92	89.33	0.48	83.56

- Discussion of Table II.

Leaching of mill concentrate as received here was uniformly unsuccessful unless the material was subjected to roasting before leaching. Even after being roasted (as in Test No. 4) the bismuth was not lowered to below 0.5 per cent. In Test No. 5,

(Leaching, cont'd) -

a larger amount of reagents was used, and a longer time was allowed, than is consistent with economical practice. We offer this result as evidence that, if so desired, bismuth may be eliminated by leaching the mill concentrate. However, we do not suggest that it can be done economically.

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Leaching Bismuth Concentrate.

A leaching method is quite satisfactory when applied to the bismuth concentrates from the flow-sheets described herein. None of the objections made to leaching as applied to the removal of the bismuth from the MoS_2 concentrate is of weight in this case, for:

1. The bismuth concentrate from flotation gives no trouble in leaching, since it is in reality a tailing and thus is free from the flotation reagents that cause trouble in treating the mill concentrate.
2. The bismuth leaches readily, since the minerals have already been roasted.
3. The amount produced is small enough so that batch leaching is feasible. And the percentage of valuable material removed is much higher than in the case of the mill concentrate, making for higher efficiency, since a comparatively small volume of solution will recover the same amount of bismuth as would the much larger volume of solution needed to treat the whole MoS_2 concentrate.

(Continued on next page)

(Leaching, cont'd) -

4. In leaching molybdenite concentrate, an appreciable percentage of MoS_2 dissolves along with the bismuth. This loss would be reduced in leaching the bismuth concentrate, for the amount of MoS_2 present is much smaller.

One of the strongest points in favour of this method is that it would make for almost 100 per cent recovery of the MoS_2 that appears in the rougher concentrates. A portion of the residue from the leaching tanks could be added directly to the mill concentrate, for though it would be low in grade, the mill concentrate would be above the required 90 per cent MoS_2 and a small amount of this residue would not lower the grade too much. In some quarters the opinion is that an 85 per cent to 88 per cent MoS_2 concentrate is the most desirable product; if this is true, then the residue from the leach would form ideal material to lower the mill concentrate in grade. The portion of the residue that was not added to the mill concentrate could be returned to the head of the circuit. Tests show that 60 per cent of the MoS_2 from this residue may be re-floated, after further grinding.

In Table III, results of leaching bismuth concentrate are recorded.

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(page.)

(Leaching, cont'd) -

TABLE III. - Leaching Bismuth Concentrate from Flotation.

SAMPLE	Bismuth Content Before Leaching, per cent	Bismuth Content After Leaching, per cent	Extraction of Bismuth, per cent
Concentrate from Lab. Test	21.80	0.16	99.37
Concentrate from Mill Run No. 4	15.90	1.07	93.28
Concentrate from Mill Run No. 12	8.87	1.49	83.20

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B. - FLOTATION TESTS.

The concentrate described at the beginning of this report was mixed together and then divided into lots of from 600 to 900 pounds for flotation tests. Head samples were cut from each lot, as the bismuth content varies widely throughout the material.

Performance of several different flow-sheets was investigated. There was no difficulty in finding a scheme to eliminate the bismuth from the molybdenite concentrate, although only moderate success was achieved in making a high-grade, molybdenite-free bismuth concentrate. Various changes in the flow-sheets were tried, in order to improve the bismuth concentrate. These included:

(1) The flotation feed was classified, inorder to send slimes and coarse material to separate flotation circuits. A screen was also tried, with the same object.

(Continued on next page)

(Flotation Tests, cont'd) -

There was nothing found in this line of investigation that would warrant its use, considering the good results and the simplicity of Flow-Sheet No. 1.

(2) Trial of a concentrating table, working on MoS_2 concentrate received, after classifying, and after screen sizing. Nothing impressive was noted.

(3) Use of a regrind mill, as shown in Flow-Sheets Nos. 2 and 3. There are possible benefits to be derived from these schemes, which will be apparent upon further study of this report.

Flow-Sheets Nos. 1 and 2 will now be discussed in detail and two other variations will be shown, in part, with some mention of their points of interest.

Flow-Sheet No. 1.

This represents the basic design of a flow-sheet for treating molybdenite ores by flotation and fractional roasting. It gives satisfactory results, simplicity of operation, and incorporates most of the mill equipment now in use at the property.

The attached plans (Figure 1) are drawn to represent an estimation of what would be required in a mill operating under the conditions at the La Corne project. The tests reported in the tables below were run in our pilot plant machines, at a rate varying from 50 per cent to 100 per cent of the rate of production of molybdenite concentrate at La Corne. With a regrind mill in the circuit, it was possible to feed four pounds per minute in the pilot plant (using No. 7 and No. 5 flotation cells), which is almost the same as the rate of production at the mill. This means that the results may be easily correlated with what may be expected from a plant-size opera-

(Flotation Tests, cont'd) -

tion.

Considering it as installed in a mill, Flow-Sheet No. 1 would involve six steps in the milling process:

1. A rougher concentrate will be made in eight Denver No. 18 cells.

2. This concentrate will be dewatered on one leaf of an American filter.

3. The filter cake will be sent to a roaster-dryer.

4. The discharge from the roaster will be repulped and sent to four Denver No. 12 cells. There it will be refloated and cleaned three times.

5. Tailings from the cleaner cells will go to a dewatering cone or thickener, where the pulp will be thickened to from 25 to 30 per cent solids.

6. The underflow from the cone will go to six Denver No. 8 cells, where the remaining molybdenite that will float will be scavenged off and returned to the cleaner circuit. Tailings from this bank will be the bismuth concentrate.

The advantages of this flow-sheet are that it is simple and extremely easy to operate. Its disadvantage is that it produces a bulky bismuth concentrate, unless the middlings are reground to liberate the MoS_2. In this flow-sheet, which is drawn in elevation to show the circulation of the products through the mill, the scavenger froth is shown returning to the ball mill for further grinding. Unfortunately, in our tests, we could not duplicate this condition, though results from small-scale tests indicate that it is quite satisfactory. The flow-sheet, if installed in a mill, should embody the principles shown here, for this scheme would perform the task required of it, and changes could be introduced at a

(Flotation Tests, cont'd) -

later date if refinements are desired.

In the diagram, the molybdenite floated in the scavenger circuit is shown returning to the cleaner cells. This material is sufficiently pure to go directly into finished MoS_2 concentrate (typical assay: 95.64 per cent MoS_2, 0.038 per cent bismuth). To send it there either a small pump would be required, pumping the scavenger concentrate to the filter, or else the scavenger cells could be placed at a high enough elevation in the mill to allow this material to flow by gravity to the filter.

The best combinations of reagents will be:

(1) Kerosene and pine oil to the rougher cells;

(2) lime, kerosene, Reagent B23, and sodium silicate to the cleaner circuit; and

(3) kerosene and Reagent B23 to the feed of the fourth cell in the scavenger bank.

The kerosene-pine oil combination seems to give the best recovery of molybdenite and bismuth, so its use is recommended in the rougher cells. Lime is added to the cleaner circuit, for roasting the concentrate makes it slightly acid upon being repulped and the lime is added to correct this condition. It has a depressing action on the bismuth and helps to settle the pulp in the dewatering cone.

To assure the best action in the scavenger circuit, the reagents are fed to the last cells in the bank. This lifts the last of the MoS_2 that will float, and returns it to the head of the circuit where the cleaner cells eliminate any bismuth that floats with it. A slight excess of reagents is fed here, so that when a surge comes in the feed these cells pick up the molybdenite and return it along with enough extra reagents needed to handle the increased feed at the head of the circuit. This automatically adjusts for any fluctuations,

(Flotation Tests, cont'd) -

reducing to a minimum the care needed on the part of the operators.

The use of Reagent B23 (or another of the alcohol frothers) is recommended for the cleaner and scavenger circuits, as it produces a less persistent froth than pine oil. This type of froth breaks up more quickly, making for better action in the cleaner cells and making it easier to overflow a clean discharge at the dewatering cone. Substitution of Reagent B23 for pine oil, which was tried also, made it possible to drop the bismuth content of the MoS_2 concentrate from 0.35 per cent to 0.16 per cent, the conditions of operation remaining the same.

Sodium silicate is fed to the retreatment circuit, to break up the flocs of gangue, bismuth and Molybdenite. Our observation indicates that it is helpful, although further work must be done to give definite assurance of this.

TABLE IV. - Results, Flow-Sheet No. 1.

Mill Run No.	FEED (per cent) Assays, per cent				MOLYBDENITE CONCENTRATE Assays, per cent				Recovery of MoS_2, per cent	BISMUTH CONCENTRATE Assays, per cent		Recovery of Bismuth, per cent
	MoS_2	Bi	Cu	Impurities (Cu+Bi)	MoS_2	Bi	Cu	Impurities, (Cu+Bi)		Bi	MoS_2	
4	88.07	2.54	0.65	3.19	93.89	0.27	0.20	0.47	97.57	17.43	25.22	90.77
7	84.00	3.20	0.42	3.62	95.06	0.16	0.23	0.39	98.43	22.50	10.10	95.69

- Discussion of Table IV.

Results from the two mill runs reported here were selected for the report, as it is believed mill operation

(Flotation Tests, cont'd) -

results will be somewhere between these two, according to the most conservative estimate. Using this flow-sheet in a mill, where scavenger float could be returned to be reground, there is little doubt that higher recovery of MoS_2 will result, while the Bismuth concentrate will be higher grade than in Mill Run No. 7. The Bismuth recovery would be slightly lower.

Flow-Sheet No. 2.

The main features of this flow-sheet are the same as in No. 1. A regrind mill is included in the circuit, making it possible to liberate the molybdenite in the middlings, which would otherwise report in the Bismuth concentrate. This cuts down the bulk of the Bismuth concentrate, reduces the MoS_2 content of that product, and also increases the capacity of the flotation cells. In Flow-Sheet No. 1 the feed rate was 1½ pounds per minute. Using the regrind mill, the feed rate was increased to four pounds a minute, with a consequent raising of the efficiency of the process, since the circulating load of middlings that had loaded the circuit before was now eliminated.

This type of flow-sheet is perhaps not shown at its best when working on a finished concentrate such as was used in these tests, but if a more bulky concentrate is fed to the retreatment circuit, as will be suggested under the heading 'General Discussion', this scheme would give much better results.

(Continued on next page)

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(Flotation Tests, cont'd) -

TABLE V. - Results, Flow-Sheet No. 2.

Mill Run No.	FEED - Assays, per cent: MoS_2	Bi	Cu	Impurities (Cu+Bi)	MOLYBDENITE CONCENTRATE - Assays, per cent: MoS_2	Bi	Cu	Impurities, (Cu+Bi)	Recovery of MoS_2, per cent
12	86.52	2.10	0.51	2.61	96.00	0.36	0.10	0.46	87.36

BISMUTH CONCENTRATE - Assays, per cent: Bi	MoS_2	Recovery of Bismuth, per cent
8.34	51.41	86.62

- Discussion of Table V.

The feed to this mill run consisted of molybdenite concentrate that had been used for filter tests, screening tests and gravity concentration. Due to the small amount of material on hand, this was used to make the test using the regrind mill, as shown in Flow-Sheet No. 2. The concentrate was roasted twice. This depressed a large part of the molybdenite, making a low-grade bismuth concentrate. And again, feed to the circuit was doubled over that fed in Mill Runs Nos. 4 and 7, with the result that the scavenger circuit did not clean the remaining MoS_2 as well as in other tests. However, though this test leaves much to be desired, it does show that a greater feed may be sent to the retreatment circuit when regrinding of middlings is provided for and it shows that a slightly higher-grade MoS_2 concentrate results when middlings are reground.

GENERAL DISCUSSION:

Advantages of New Milling Procedure

The problem as presented to these Laboratories was simply to find a suitable method for eliminating the bismuth from the molybdenite concentrate. In the foregoing the only apparent solution has been discussed and the difficulties involved in trying to use other methods at La Corne have been shown.

However, in developing the scheme of fractional roasting and retreatment for this concentrate, it has occurred to us that its adoption would make possible other improvements beyond the original scope of the problem of getting rid of the bismuth. The flow-sheets illustrated in this report are designed to make provision for these advantages, if the mill is operated properly. In order that the operator shall understand what is required, a discussion of the recommended changes and of the basis of our reasoning in suggesting them follows:

In the early stages of the present period of activity at the La Corne mine, it was found that a 60 per cent MoS_2 concentrate could be made, with tailings as low as 0.02 per cent. This meant a recovery of 96 per cent. This concentrate was, of course, too low in grade for the market. With alterations in the milling scheme, a concentrate grading 85 per cent MoS_2 was made, with slightly lower recovery. Later, the present flow-sheet was devised, in order to raise the grade of the concentrate still more. It is comparatively inefficient, for it is not making as high a recovery as is possible considering the reagents and the power consumed. This inefficiency is due to the heavy load of circulating middlings in the flotation circuit, and to overgrinding of the molybdenite. However, efficiency had to be sacrificed for expediency in making a suitable product.

(Continued on next page)

(General Discussion, cont'd) -

Now that a retreatment process is indicated in order to recover the bismuth, it is possible to attain the high recovery that goes with the making of a low-grade rougher concentrate such as that mentioned above, using the retreatment plant flow-sheet. In other words, the flow-sheets designed for making a bismuth-free molybdenite concentrate will also raise a rougher concentrate to the required grade (90 per cent MoS_2) and would at the same time increase mill recovery of molybdenite to an estimated 93 to 94 per cent as compared with the present recovery of 88 to 89 per cent. A process for making a rougher concentrate, with later retreatment to separate the different products, usually represents the most efficient procedure for a flotation plant producing more than one concentrate.

However, it should be noted here that milling results would not be the same, in a plant working on a low-grade rougher concentrate, as are those reported in these tables. In the first place, the molybdenite concentrate would be slightly lower in grade, from 89 per cent to 95 per cent, depending on the operation of the cleaner cells producing this concentrate. Estimating from small-scale tests, best results could be obtained by making a 60 per cent rougher concentrate, followed by retreatment and the final production of material grading 92 per cent MoS_2 containing less than 0.2 per cent bismuth.

There are various opinions as to what should be the specifications for a molybdenite concentrate. During the war there has been a market for low-grade and impure material. However, it is known that before the war competitors with Climax sold a 95 per cent MoS_2 concentrate and that they had no market for material of less than 90 per cent MoS_2. Regulations governing impurities said that the sum of these

(General Discussion, cont'd) -

must not be above 0.5 per cent (copper, bismuth, and antimony). It would therefore be dangerous to plan on the production of anything that could not meet these specifications.

The bismuth concentrate produced under the conditions described above would be much larger in bulk than that produced in our experimental runs. The gangue and middlings from the rougher concentrate would report in the tailings with the bismuth. The regrind mill shown in Flow-Sheet No. 2 would be of assistance in reducing the volume of this concentrate, as it would make possible the returning of much of the MoS_2 in the middlings to the rougher cells where it would be floated off with the finished concentrate. This would be essential if direct sale of the bismuth concentrate is contemplated, as it would be wasteful to ship the contained MoS_2 with this product.

In attempting to show how greater efficiency of operation would result from the use of this flow-sheet, mention should be made of the fact that it would prevent a great deal of the overgrinding of the molybdenite that is undoubtedly causing much of the tailings loss in the present operation. Slime molybdenite is extremely difficult to float, and, as mentioned above, the material received here is ground to 46 per cent minus 200 mesh. This contrasts with the ball mill discharge, which is only 16 per cent minus 200 mesh. It is evident that selective overgrinding of the molybdenite is taking place, for this sulphide is much more friable than the hard gangue materials. Contrasting a screening test of the bismuthinite in the concentrate with the screen size of the ball mill discharge, we note that 70 per cent of the bismuthinite is minus 200 mesh, as compared to 16 per cent of the ball mill discharge. This shows that the bismuthinite, which is very heavy and not inclined to overflow the classifier

(General Discussion, cont'd) -

discharge weir, is being held in the circuit much longer than should be the case.

Making a low-grade rougher concentrate, as suggested above, would reduce this sliming action in the grinding circuit, as there would be no necessity for grinding to completion in one stage. At Climax Molybdenum Co., the practice is to float a rougher concentrate after crushing to 28 mesh. This is not practical in a small plant such as the La Corne, for it involves four regrinding stages later in the circuit. However, it points the way for more efficient practice, and to all the advantages to be obtained by avoiding overgrinding, which is very important in milling molybdenite.

In this regard, also, we recommend the trial of a unit cell in the ball mill discharge circuit. This would remove the sulphides as soon as they are freed from the gangue. With a unit cell, it is possible that the number of cells in the rougher bank could be reduced, resulting in a substantial saving in power.

If the full import of the benefits to be derived from these recommendations is realized, the reader will see why retreatment by fractional roasting and flotation is recommended, rather than a solution of the problem based on leaching, or some other method merely aimed at eliminating the bismuth from the concentrate. The improvements thus made possible will reduce operational expenses and increase the capacity of the grinding unit. They are always as important, economically, as is the income to be derived from saving the bismuth.

Further Considerations:

A great deal of study has been given to the problem of making a table concentrate of the bismuth; as explained earlier, this is not possible as applied directly to the mill concentrate. However, when tried on the tailings from the cleaner cells, as shown in Figure 3, Flow-Sheet No. 3, encouraging results are possible, since in this feed most of the molybdenite has already been floated off. A table working in this manner performs the triple operation of a concentrating bismuth, desliming, and separating a middling product for regrinding. Such a table will recover 12 per cent of the bismuth in the feed, in a concentrate grading as high as 68 per cent bismuth, and as low as 2 per cent MoS_2.

The table shown in Figure 4, Flow-Sheet No. 4, will recover 53 per cent of the bismuth in the feed, in a concentrate grading 20 per cent bismuth and 12 per cent MoS_2. It has an opportunity to work very effectively on the tailings from the flow-sheet, which are high in bismuth and comparatively free from the interference of the MoS_2.

The use of a table should be given more serious consideration if direct sale of the bismuth concentrate is contemplated. It also is helpful before a leaching process, as removal of the coarse bismuth greatly facilitates the task of leaching bismuth from this material and it is to be expected that if overgrinding of the sulphides is avoided, as set down in the above discussion, a larger amount of coarse bismuthinite would appear in the table feed. Under those conditions it is expected that the effectiveness of a concentrating table would be much greater, since bismuthinite behaves very well on a table when operating conditions are at all favourable.

<u>Mill Equipment</u>:

A large part of the mill equipment now in use could be included in these flow-sheets. The following list shows what would be needed for Flow-Sheet No. 11. The additional equipment in the other schemes may be seen in the diagrams. The eight No. 18 cells needed in the rougher circuit are

(General Discussion, cont'd) -

already on hand, as are the four No. 12 cells in the cleaner circuit. Beyond this, the following new equipment would be required.

1. A two-leaf American filter, 4-5 feet in diameter.
2. A roaster.
3. One 1-inch Wilfley pump.
4. Six No. 8 flotation cells.
5. A desliming cone, 6 to 8 feet in diameter, or a small thickener.

Equipment needed for leaching bismuth concentrate, in case this is decided upon, would consist of three wooden tanks and an acid pump. The three filter boxes now on hand at the mill would serve, though they would have to be treated with acid-resistant material.

Roaster

The reaction to our previous report (Report of Investigation No. 1808, March 1945) was to the effect that a roaster and a roasting process were too expensive to warrant consideration in the solution of this problem. Perhaps a better understanding of the true picture may be obtained after some explanation of the use of a roaster here.

Use of the term 'roaster' is perhaps confusing; the costs of this machine and of operation are more closely comparable with those of a dryer; actually, that is all that is required. The concentrate from the rougher cells is dried, then the kerosene and pine oil reagents are ignited to complete the tarnishing of the bismuthinite particles. In these tests, the hearth temperature of the roaster was held around 280° Centigrade, which is only a fraction of that of an ordinary roaster.

Since care must be taken not to reach too high a temperature, the use of a conventional roaster is not recommended for this work. In a multiple-hearth roaster, it is

(General Discussion, cont'd) -

difficult to avoid high temperatures at the points where the material drops from one hearth to another. This concentrate must not be heated to above 300° C., or a part of the molybdenite will be depressed, in flotation, along with the bismuth. We have sketched a machine (see Roaster Diagram*) that should give the proper conditions of operation for fractional roasting. Exposure of the roaster feed to too high a temperature is avoided in the machine shown.

The cake from the first leaf of the filter (the rougher concentrate) drops into a spiral conveyor (1). There it is dried as it passes down the conveyor, both by hot gases passing up and by heat applied directly to the casing of the conveyor from the flame in the firebox. It is discharged onto a sloping plate, where it is rabbled forward while the flame has an opportunity to beat directly upon it (2). This provides for ignition of the reagents. From the hearth it drops onto a discharge conveyor (3), where air entering the furnace will cool the material before it is repulped.

This system assures optimum control with the least possible amount of attention. The air admitted to the cooling conveyor (3) is preheated while cooling the discharged concentrate, and is thus ready to perform the oxidizing process on the roasting hearth. This air, along with hot gases from the burner flame (4), is then passed out of the machine by the three dampered outlets (5, 6, 7), so arranged as to allow application of heat where it is needed in the system.

The waste heat from the roaster could be passed to a horizontal spiral conveyor above the roaster. The filter cake off the second leaf, which would be the finished concentrate, would pass through this conveyor and be dried before reaching the shipping bin. It is probable that a small burner would be required here, to boost the heat from the furnace,

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* Figure 5, on Page 34.

(General Discussion, cont'd) -

in order to accomplish complete drying of the concentrate. Some such arrangement as this should be installed if sale of the material is to be in distant markets, such as England. Shipment over long distances implies almost complete removal of the moisture content; the system outlined in the drawing will accomplish this and at the same time provide for collecting the dust that is lost when such a concentrate is dried to 5 per cent moisture.

Depressing the bismuthinite in this concentrate by means of such a roaster would cost from thirty cents to a dollar per ton of material handled. The cost of building this roaster would be well under one thousand dollars.

Filtering Tests:

In order to prove definitely that there would be no trouble in filtering the La Corne molybdenite concentrate, tests were run on this material on a small Oliver filter, both before and after roasting. The result shows that molybdenite is one of the very best of mill products in its behaviour on a filter. This corroborates our previous experience.

TABLE VI. - Results of Filtering

Concentrate	Moisture, per cent	Dry weight of concentrate per sq. ft. of filter in 24 hours
As received	16	~~19 lb.~~ 1,106
Roasted	14	~~22 lb.~~ 1,280

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Correlation of Laboratory Results With Mill Runs:

There are points of variation between the results obtained in the mill run tests and those from laboratory-scale tests.

For one thing, our estimation of the size of flotation cells that would be needed in a mill, (No. 18 cells in the

(General Discussion, cont'd) -

cleaner circuit) called for cells much larger than is indicated by actual tests. The reason for this is that it is not necessary to carry so large a load of circulating middlings as was at first thought. No. 12 and No. 8 cells are now indicated for the retreatment plant. These are larger than is necessary but will do the work efficiently, and if a larger retreatment plant feed is taken, as discussed above, the extra capacity will be useful.

In the preliminary report of these Laboratories (Investigation No. 1808) it was said that a temperature of 400° C. was called for in the roasting process. We know now that best results are achieved at 300° C.

The recoveries and grades of the products are substantially the same in mill run tests as in small-scale work. In the mill runs, it was not found possible to make as high a grade of bismuth concentrate as in the laboratory tests. In the mill tests it was not possible to return the scavenger cell float to the roaster, as is essential for best operation of the circuit.

We believe that in a mill, where conditions may be regulated to follow more exactly the procedure used in making the small-scale tests, it will be possible to attain results similar to those of our preliminary report, that is, production of a bismuth concentrate grading 18 per cent MoS_2 and 36 per cent bismuth.

In the preliminary report it was indicated that the roaster discharge should be repulped, filtered and washed to cut down the acidity. This is necessary if the concentrate is roasted to a high temperature. When roasted to 300° C. which is now considered to be the best practice, the acidity resulting from roasting is so low that it may be easily corrected by the addition of a small amount of lime, and it is thus not necessary to filter the roaster discharge before it goes to the cleaner cells.

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SUMMARY AND CONCLUSIONS:

The results of the test work on La Corne molybdenite definitely indicate the advisability of adopting a rougher-retreatment method of flotation based on the fact that bismuthinite may be depressed, while floating molybdenite, by a process of fractional roasting. The process outlined here gives the only solution which will provide efficient operation of the mill, highest recovery of the products, and still meet the standards set for competitors with Climax Molybdenum Co. before the war. These specifications were, again, a plus 90 per cent MoS_2 concentrate with the sum of the impurities (copper, bismuth, antimony) below 0.5 per cent.

We have taken the liberty of outlining a new concept of milling for La Corne, aimed at more efficient milling procedure, the production of a cleaner MoS_2 concentrate and higher recovery of MoS_2, the production of bismuth as a by-product, and a more satisfactory method for handling finished concentrate in the bagging and drying stages. Our test work and previous experience provide sufficient background to enable us to say that these recommendations are the best procedure, although more work is necessary to decide upon the most efficient approach to some of the phases. These are:

1. Further study on the leaching of bismuth. We are continuing with these investigations, using the products from mill tests.
2. Search for possible improvements in the reagents used. This may wait until a mill is in operation, for the present reagents give entirely satisfactory results.
3. Investigation of results obtained using minor alterations in the flow-sheet described here. These alterations would be:

(Continued on next page)

(Summary and Conclusions, cont'd) -

(a) Inclusion of a unit cell at the ball mill discharge.

(b) A trial of results achieved by returning the scavenger-circuit froth to the ball mill, or to the roaster, or maintaining it as a circulating load within the scavenger circuit.

(c) Trial of five No. 12 cells in the cleaner circuit. We use five cells part of the time in the mill tests, with two units floating molybdenite and the last three recleaning this float. Results indicated that with this scheme operational control was slightly easier.

In view of these unsettled points it is definitely suggested that if fractional roasting is decided upon, mill design should be according to Flow-Sheet No. 1. Any of the variations suggested might then be included at a later date, if found advisable. This flow-sheet has given excellent results in mill tests, though we were at the disadvantage here that we could not return middlings to the ball mill, or to the roaster, as best operation calls for. The suggestion is that better results than those reported in the tables will be forthcoming when this flow-sheet is used in a mill.

A separate report will be issued shortly, covering the problem of refining bismuth concentrate.

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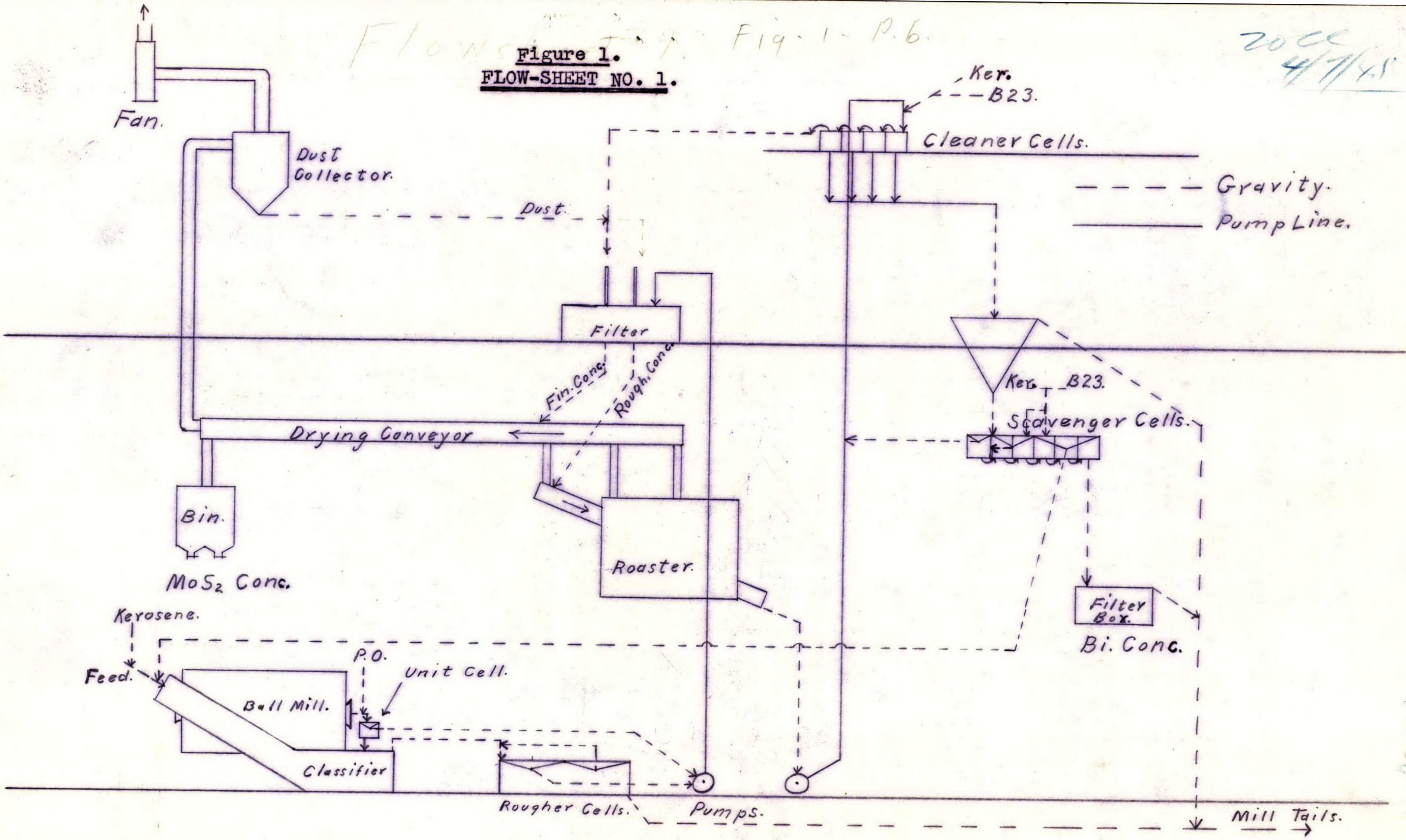
Figure 1.

FLOW-SHEET NO. 1.



Flowsheet #2 Fig 2 P. 9.

Figure 2.

FLOW-SHEET NO. 2.

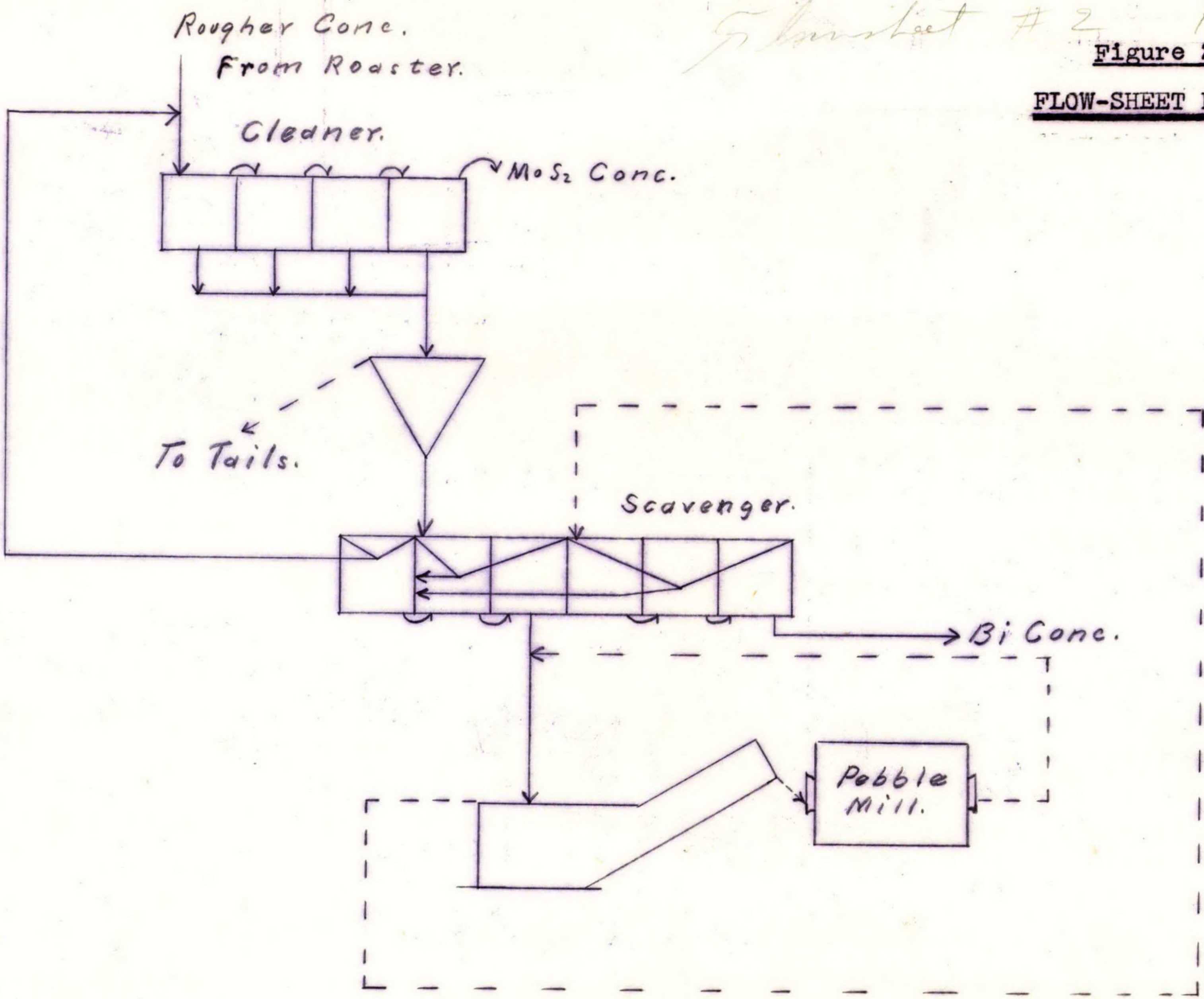
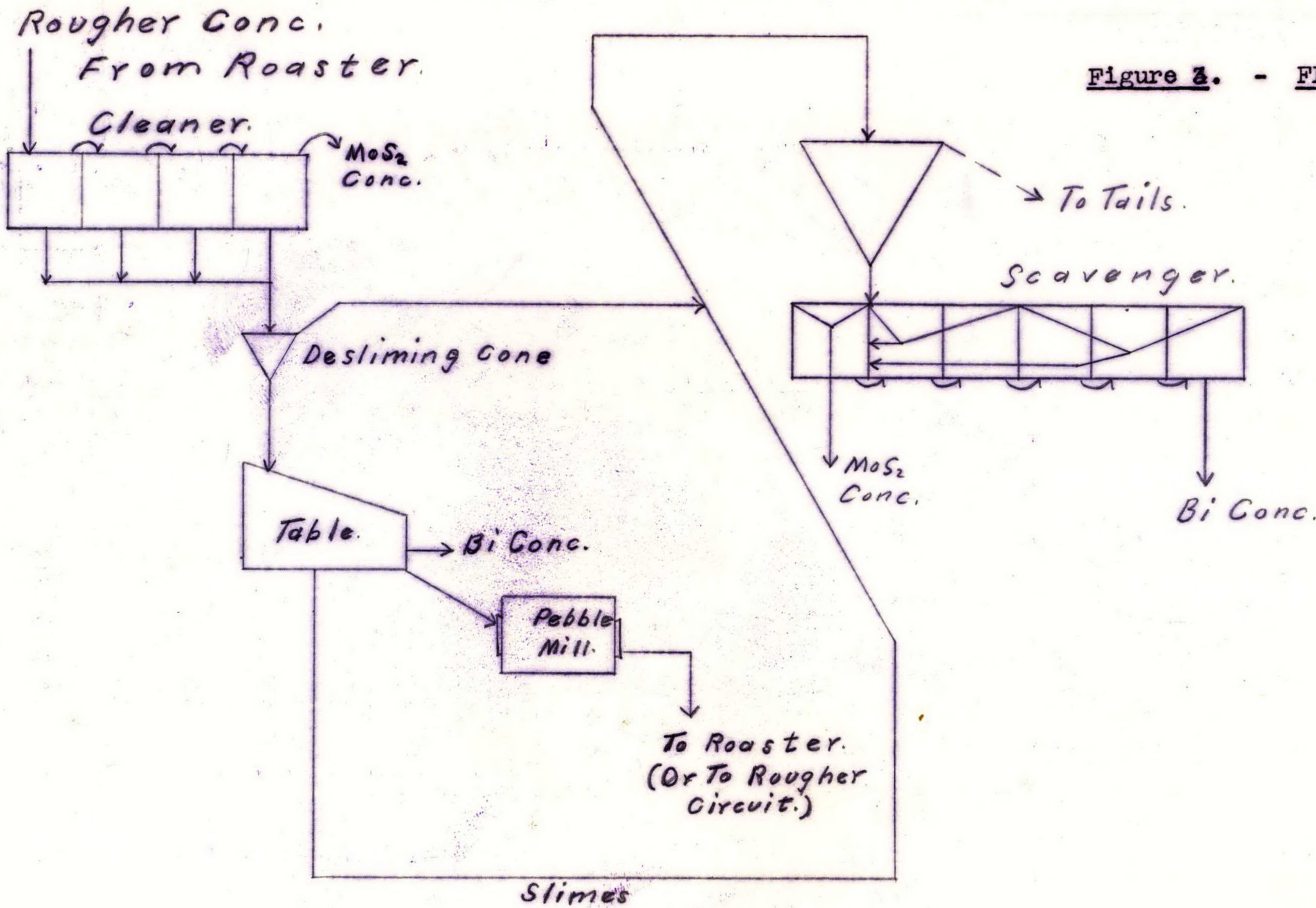


Figure 3. - **FLOW-SHEET NO. 3.**



Flowsheet #4 Fig 5, P 17

Figure 4. - FLOW-SHEET NO. 4.

(Same as Flow-Sheet No. 1, with the addition of a concentrating table.)

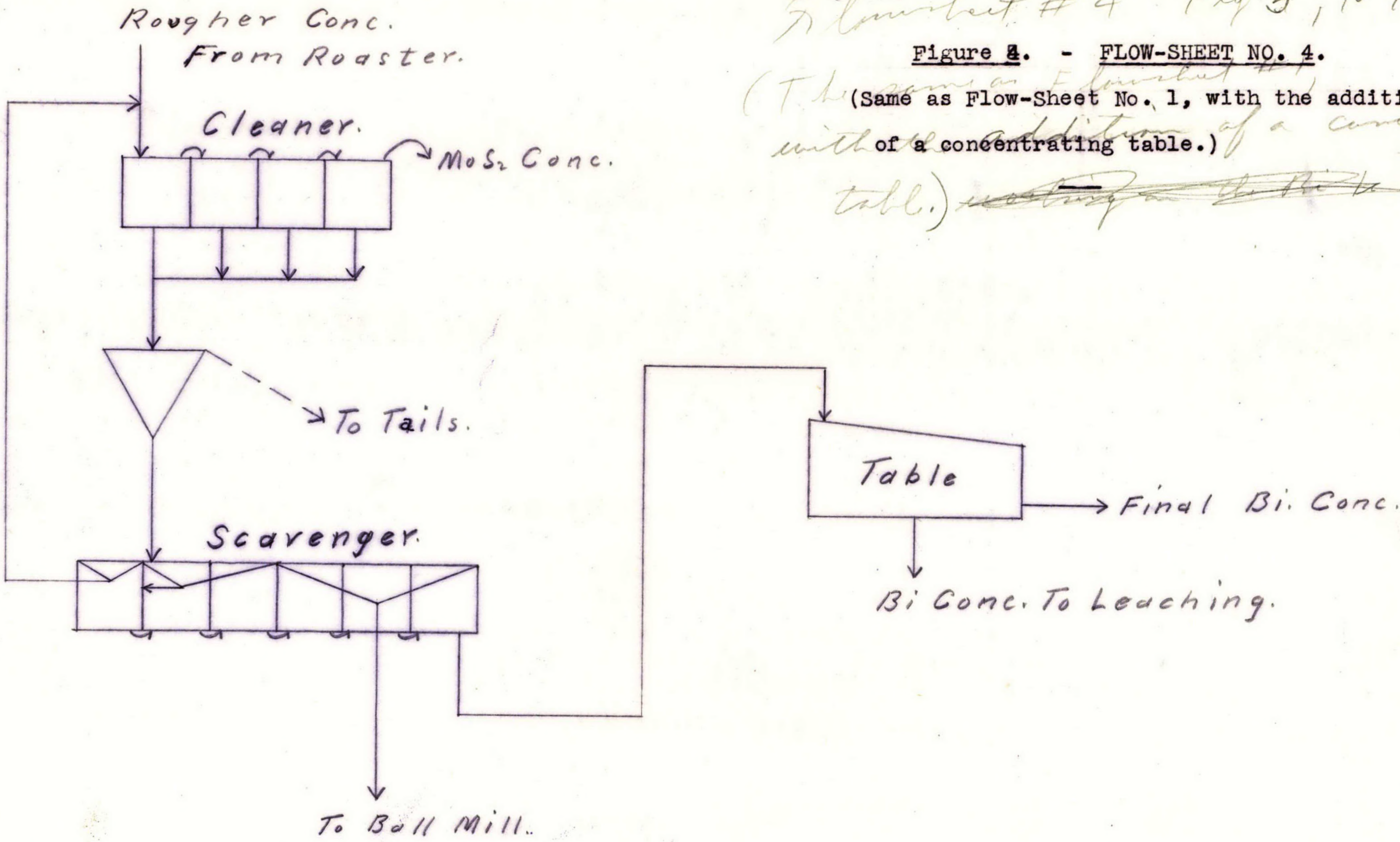


Figure 6.

ROASTER-DRYER.