Econ. Division
(Miss Stewart)

OTTAWA October 15, 1945.

REPORT

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1932.

Disposal of Bismuth Concentrate from the La Corne Molybdenum Project, Val d'Or, Quebec.

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Ore Dressing
and Metallurgical
Laboratories

CANADA
DEPARTMENT
OF
MINES AND RESOURCES

Mines and Geology Branch

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Source of Material and Purpose of Investigation:

Within the past year two shipments of molybdenite concentrate have been forwarded to these Laboratories from the La Corne Molybdenum Project of the Wartime Metals Corporation at Val d'Or, Quebec. This material was used in investigations to work out a method for eliminating the bismuth contained in this concentrate as an impurity.

By a process of fractional roasting and flotation, described in Report of Investigation No. 1902, dated July 20, 1945, it was found that a clean molybdenite concentrate could be made, and a bismuth concentrate of the following qualities resulted as a tailing from the process:

Bismuth Concentrate		Bi	MoS_2
		- Per Cent -	
A	-	15.40	33.00
B	-	22.50	10.10

(Source of Material and Purpose of Investigation, cont'd) -

The first of these, Bismuth Concentrate A, was selected for further investigation to determine the best procedure for refining or marketing the concentrate. Bismuth Concentrate A was not the highest grade of material resulting from our mill tests, but was selected so that results from the work would represent a safe appraisal of what could be expected from an operating plant. As pointed out in Report No. 1902, the probability is that an operation at La Corne could put out a much better grade of bismuth concentrate.

General Discussion:

The opinion is that the bismuth concentrate should be disposed of by sale to a refinery, if satisfactory marketing conditions can be arranged. However, before such a procedure is adopted, there are certain aspects which should be discussed. The most important of these is the possibility that a large part of the molybdenite contained in the bismuth concentrate may be recovered.

If the flotation bismuth concentrate is passed over a concentrating table, as in Flow-Sheet No. 4 (see Figure 1 herein) from Report No. 1902, about 50 per cent of the bismuth contained is taken off in the table concentrate, while the greater proportion of the molybdenite goes into table tailings. The suggested procedure in refining the concentrate would be, then:

1. To table the flotation bismuth concentrate, as in Flow-Sheet No. 4.
2. The table concentrate, high in bismuth and low in molybdenite, would be smelted or sent to a refinery, depending on the decision of the company officials.
3. The table tailing, high in molybdenite and low in bismuth, would be leached to remove the bismuth, and

(General Discussion, cont'd) -

the residue from leaching would be added to the molybdenite concentrate put out by the main part of the mill.

4. From leaching, bismuth oxychloride would result. This could be added to the table bismuth concentrate and smelted, to reduce the contained bismuth to metal.

Both the smelting and leaching steps are extremely easy to perform, so that no difficulty is expected if they are carried on at the plant. Moreover, the bismuth concentrate resulting from table concentration is so high in grade and so relatively free from impurities that it should find a ready sale at a refinery. It should be possible to sell the bismuth oxychloride from leaching to the same buyer.

This procedure is recommended because:

(1) It is easier and cheaper to smelt the coarse fraction of the bismuth (which reports in the table concentrate) than to leach such material.

(2) The tailings from the table contain only the extremely fine bismuth, which is readily leached out by dilute hydrochloric acid.

(3) After the bismuth has been leached from these tailings, the residue may be added to the molybdenite concentrate, thus saving virtually all the molybdenite sent to the retreatment plant.

In Table No. 1, below, an analysis is made of the results obtained by concentrating the flotation bismuth concentrate on a table. It will be noted that a high-grade bismuth concentrate is made, small in bulk, and relatively low in molybdenite.

(General Discussion, cont'd) -

The table tailing is low in bismuth and high in molybdenite. After leaching, it is still higher grade in molybdenite, while the fact that it contains only the fine bismuth makes it possible to leach out this impurity, so that no contamination of the molybdenite concentrate would result if the leach residue were added to it before shipping.

TABLE I. - Results of Table Concentration of Bismuth Concentrate.

FLOTATION BISMUTH CONCENTRATE FROM FLOTATION		TABLE PRODUCTS					
		Concentrate			Tailing		
Bi, per cent	MoS_2, per cent	Bi, per cent	MoS_2, per cent	Recovery of Bi, per cent	MoS_2, per cent	Bi, per cent	Recovery of MoS_2, per cent
15.40	33.00	26.48	9.00	62.95	46.20	9.08	90.32

INVESTIGATIONS OF LEACHING AND SMELTING PROCEDURES:

A. - Leaching.

A flow-sheet for leaching the table tailings from the concentrating table mentioned above is included in this report (see Figure 2).

The tailings from the table (No. 1 in Figure 2) will be collected in a filter box (No. 2). A batch of this material will then be transferred to a leaching tank (No. 3). Leaching solution will be added to this, and the whole will be agitated for eight hours, then allowed to settle in the tank. The clear solution from the top of the tank will be decanted off by means of an acid pump and pipe line, and sent to a second leaching tank (No. 4), in which a second batch of ore has been placed. This also will be agitated for eight hours. Then, when the leaching solution has become saturated with dissolved bismuth, (Leaching, cont'd) -

the tank will be allowed to settle and the solution will be decanted off and sent to a precipitation tank (No. 5). There, water will be added, which will precipitate the bismuth as bismuth oxychloride by a process of hydrolysis. The oxychloride will be filtered out.

A part of the barren solution from the filter will be used to make up new leaching solution. For each 100 pounds of ore in the leaching tank, four hundred pounds of barren solution together with 20 pounds of ferric chloride and 50 pounds of HCl will be used, Less dilute or more concentrated leaching solutions may be used, but this is a satisfactory proportion of acid to old solution, and only practice will make it possible to decide upon the best mixture. This new leaching solution will be pumped into leaching tank No. 4, which contains partially leached ore. The new solution will absorb the remaining bismuth in this material, then the tank will be allowed to settle and the solution will be decanted off and sent to leaching tank No. 3, in which a fresh batch of ore from the filter box has been placed. When the solution is exposed to this new ore, rich in bismuth, it will become saturated with dissolved bismuth, and then may be decanted off and sent to the precipitation tank (No. 5).

The ore remaining in tank No. 4, which will be completely leached, will then be removed and added to the main molybdenite concentrate in another part of the mill. Fresh ore from the filter box will be placed in this tank. Then, after the saturated leaching solution has been removed from tank No. 3, fresh acid will be added to this tank, the solution will be decanted off after four hours, and sent to the fresh ore in tank No. 4.

In this way partially leached ore will be exposed

(Leaching, cont'd) -

to fresh leaching solution, and partially saturated leaching solution will be exposed to fresh ore. Thus the advantages of counter-current leaching will be obtained, by a simple process of pumping the solution from one tank to another. This results in complete utilization of the acid employed in leaching, and almost complete extraction of the bismuth in the ore.

A process in which the ore is simply placed in a leaching tank, exposed to the acid, and then filtered, will serve to remove most of the bismuth, but it will result in a lower extraction of bismuth and a greater consumption of acid.

In Table II, results from leaching tests are summarized.

TABLE II. - Leaching Table Tailing.

TABLE TAILING		AFTER TWO 8-HOUR LEACHES		
MoS_2, per cent	Bi, per cent	MoS_2, per cent	Bi, per cent	Extraction of Bismuth, per cent
46.20	9.08	53.00	0.32	96.47

B. - Smelting.

Bismuth smelting is carried on either in crucibles or in small reverberatory furnaces. If a furnace is built at La Corne, smelting in crucibles is recommended as this procedure requires an inexpensive furnace, and the pot method is best for small lots of concentrate.

Smelting procedure would be:

1. 100 pounds of bismuth concentrate from the table will be a suitable charge for each pot.
2. The bismuth oxychloride resulting from a leaching batch will be dried, mixed with sodium carbonate, and added to the bismuth concentrate.
3. This charge will be mixed with suitable fluxes and

(Smelting, cont'd) -

placed in the smelting crucibles.

4. The pots will be placed in the furnace and held at 1050° C. until smelting is complete.
5. The melt will be poured from the crucibles into conical moulds.
6. The products from smelting will be slag, iron matte and metallic bismuth. The slag will break away easily, but the matte and metal will not separate at once. If left exposed to the air for a short time the matte spalls off, leaving a clean metal button.

A typical charge for a smelting pot will be:

100	pounds	bismuth concentrate.
20	"	bismuth oxychloride.
30	"	sodium carbonate.
5	"	lime.
10	"	sodium sulphate.
7	"	sodium hydroxide.
5	"	fluorite.
6	"	charcoal.
15	"	metallic iron.

As a matter of record, in case the company undertakes smelting at some future date, a discussion of this smelting charge is set down herein. This is not pretended to be the ideal smelting mixture, but variations in the constituents, tried in our experiments, always resulted in unsatisfactory melts. The reasons for using more or less the same charge as that outlined above are:

Sodium carbonate is used to obtain a low melting point. This is essential, as bismuth begins to volatilize at 1100° C. and thus smelting should be done at a temperature below that point. Also, when sodium carbonate and bismuth oxychloride are smelted together, the sodium combines with the chlorine. This reduces volatilization losses of bismuth chloride.

(Smelting, cont'd) -

The use of lime makes for a highly fluid slag. Too much should not be used, as the silicates formed by this flux are fusible only at a high temperature.

Sodium sulphate melts at a low temperature and produces a slag low in specific gravity. Too much should not be used, or the slag will be sticky.

Sodium hydroxide melts at a very low temperature and thus hastens the reaction between the other constituents of the melt. No more than needed should be used, as it is hard on crucibles.

Fluorite is added to the melt to take care of any irregularities, as it will dissolve anything.

Charcoal is added to the melt to provide a reducing atmosphere. This reduces the bismuth oxychloride, and prevents oxidizing of the metal as it is liberated from the bismuthinite.

Metallic iron, in finely divided form such as scrap, sheet or lathe turnings, serves to liberate the metal from the sulphide. Iron has greater affinity for sulphur than has bismuth; hence when they are melted together an iron matte is formed, while the bismuth goes into the metallic state.

In Table III, below, is a summary of results obtained by smelting bismuth oxychloride and table bismuth concentrate. Recovery of the bismuth contained in the concentrate from flotation is shown, and also the purity of the metal produced, so that an estimate may be made of the results obtained by processing the mill concentrate in this way.

(Table III follows,)
(on next page.)

(Smelting, cont'd) -

TABLE III. - Smelting Bismuth Oxychloride and Table Bismuth Concentrate.

	Table Concentrate	BiOCl from Leaching	Bismuth Button from Crucible	Recovery of Bismuth in Smelting, per cent
Weight of product, gms.	100.0	5.6	35.7	
Contained Bi, per cent	26.48	69.88	81.84	96.11
Weight of Bi contained, gms.	26.48	3.91	29.21	

SUMMARY AND CONCLUSION:

This report outlines a rough approach to the problem of putting the bismuth concentrate from the La Corne Mill into marketable form. As there is no immediate need for a solution to the problem, pending the complete installation of the fractional roasting equipment, it is hoped that it will be sufficient for the present.

The general idea in any solution would appear to be that the concentrate should, as much as possible, be prepared for smelting, as this method of treatment is apparently cheaper than leaching. There seems to be little doubt that, if necessary, the whole of the bismuth concentrate could be leached. This method could be employed in case it were found to be difficult to smelt the table concentrate to a sufficient degree of purity. In any leaching method it seems advisable to use a process based on dilute leaching solutions, such as in the process outlined above, for this approach seems to assure lower expenses for equipment and for reagents consumed.

As refinements of the leaching process outlined above, the following possibilities might well be considered:

1. Perhaps the easily soluble molybdenite from the bismuth table tailing could be dissolved with ammonia.

(Continued on next page)

(Summary and Conclusion, cont'd) -

This would ensure that little of the MoS_2 would be taken up by the bismuth leaching solutions, making for both a cleaner bismuth oxychloride and a smaller loss of MoS_2. The molybdenum could be precipitated out of this leach solution by adding acid, and the precipitate could then be filtered out.

2. It might be that the bismuth and part of the molybdenite in the concentrate could be dissolved in HCl, after which the bismuth could be precipitated by a metal, such as iron or aluminium scrap. The residual solution, containing dissolved molybdenite, could then be treated to recover the molybdenum. This would result in the formation of a clean bismuth precipitate, and would make possible the recovery of any molybdenum lost in the leaching process.

3. The use of blanket tables should be tried, working on the table tailings from the bismuth table. It might be that such a piece of equipment would make possible the rejection of a molybdenite tailing from these two gravity concentrating steps which would be very low in bismuth.

It is difficult to estimate costs of any method suggested, for they would depend on the procedure adopted. To show that smelting is cheaper than leaching, it might be pointed out that estimates based on the experiments described above show that 62 per cent of the bismuth from a day's run, or 550 pounds of the concentrate, may be smelted with the use of $4.70 worth of fluxing material, while the acid and ferric chloride needed to leach out the remaining bismuth in the table tailings from a day's operation would cost about $12.00.

In conclusion, there seems to be little doubt that good recovery may be made from the bismuth concentrate at

(Summary and Conclusion, cont'd) -

La Corne, by any of several methods which suggest themselves. The procedures outlined here suit the conditions expected when the mill begins operation. Refinements or changes will have to await more complete examination of the problem, which may best be approached after ascertaining what grade of bismuth concentrate the mill will produce.

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(Figures 1 and 2)
(follow, on Pages)
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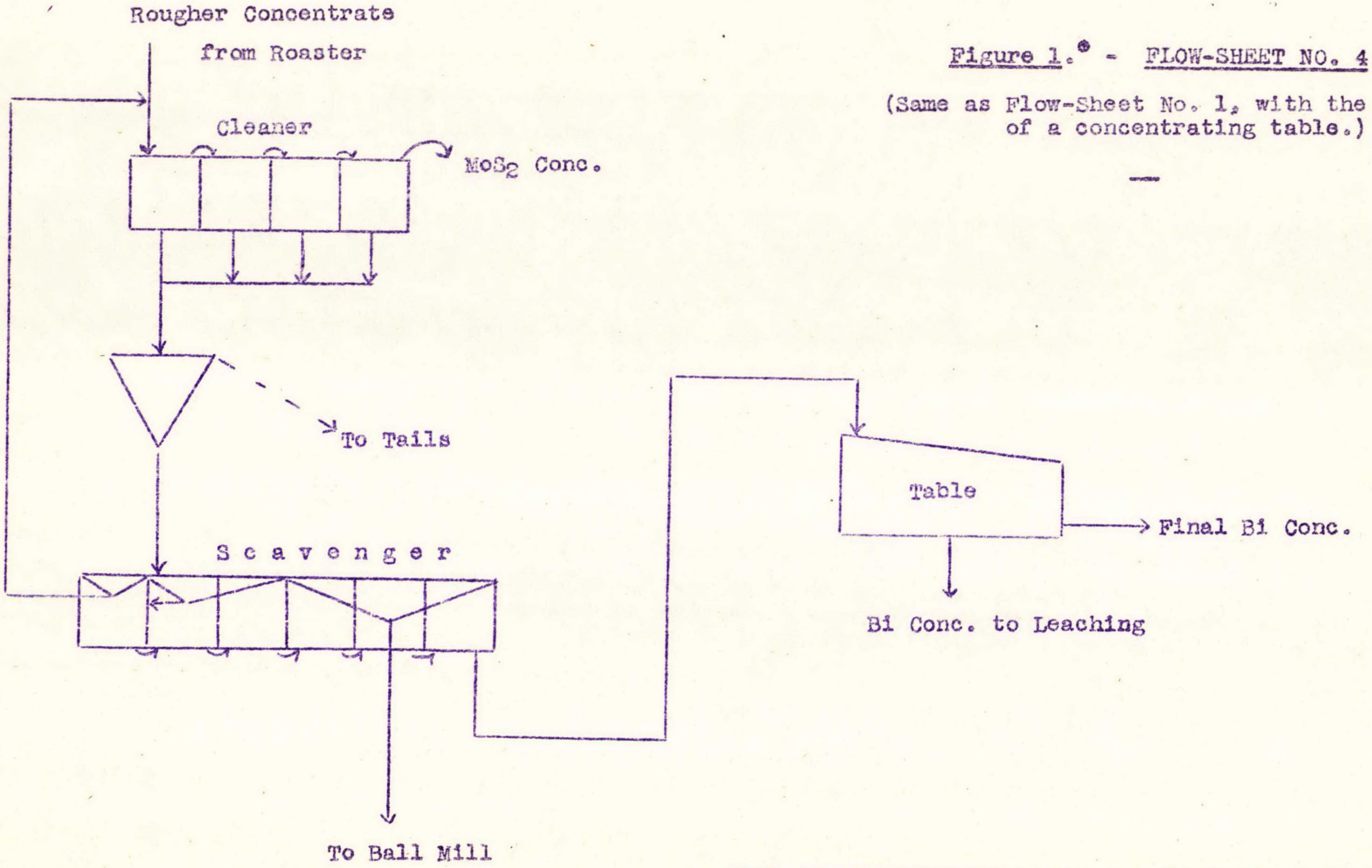

<u>Figure 1.</u>[*] - <u>FLOW-SHEET NO. 4.</u>

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

([*] This was Figure 4, Flow-Sheet No. 4, in Report of Investigation No. 1902, July 20, 1945, p. 35.)

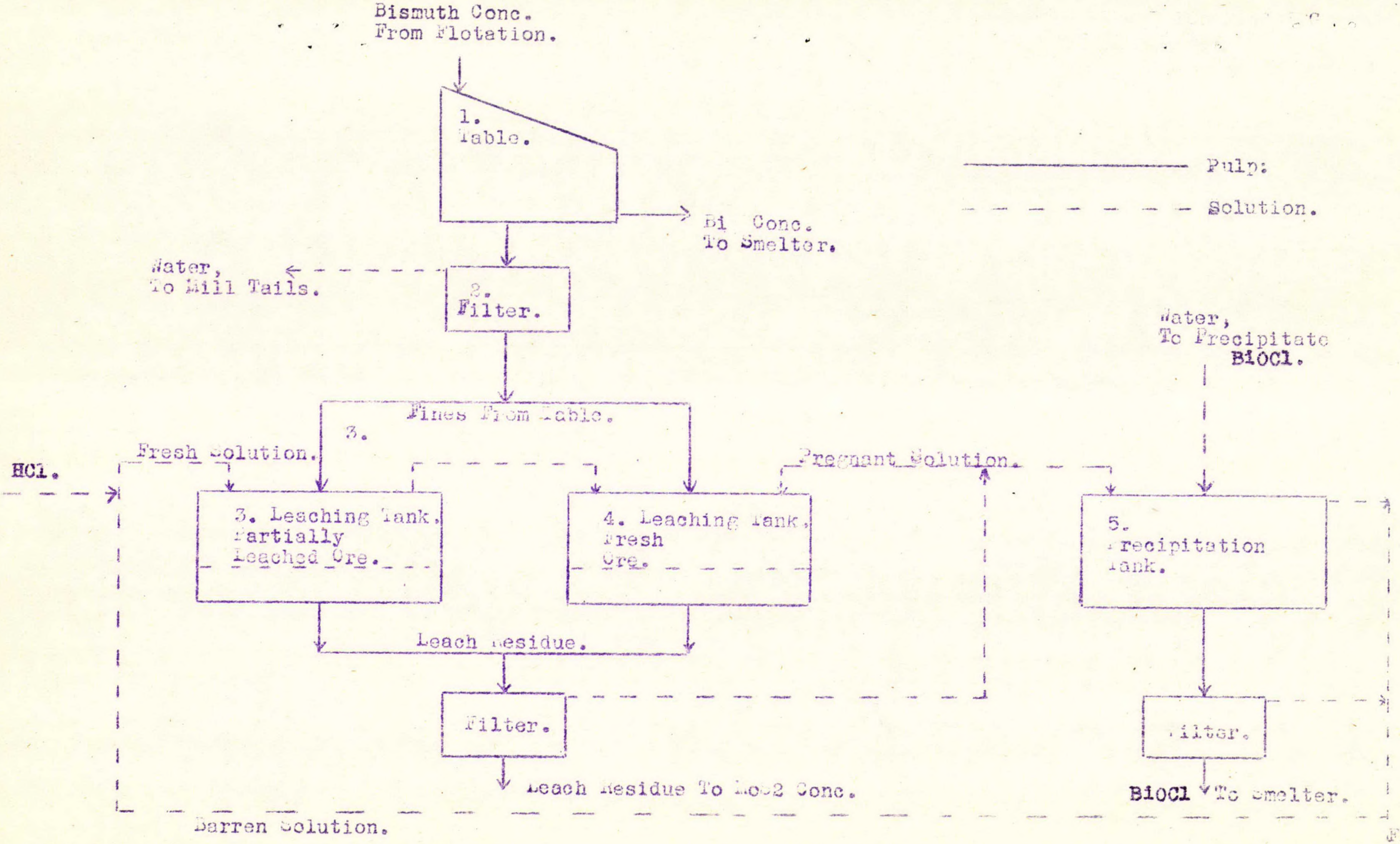

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Figure 2.

FLOW-SHEET FOR LEACHING TABLE TAILINGS.