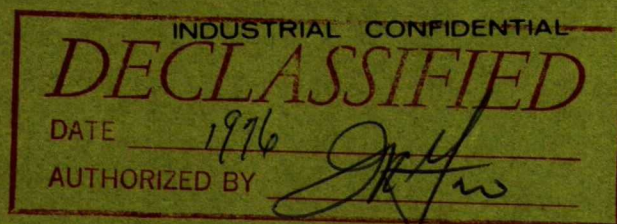


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CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 73-6

**FLOWSHEET DEVELOPMENT FOR PROCESSING
AN ANTIMONY ORE FROM
SOUTH HAM TOWNSHIP, QUEBEC**

by

G. I. MATHIEU AND R. W. BRUCE

MINERAL PROCESSING DIVISION

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G.I. Mathieu* and R.W. Bruce**

- - -

SUMMARY OF RESULTS

Head analysis of the sample received for investigation gave the following results:

<u>Sb</u>	<u>As</u>	<u>S</u>	<u>Fe</u>	<u>Insol</u>
1.25%	0.09%	0.66%	4.10%	79.7

Mineralogical examination indicated that the antimony occurs chiefly in the form of sulphides, with minor amounts in the form of oxides, and as traces of native metal. The main antimony sulphides are gudmundite and, to a much lesser extent, stibnite. Valentinite is the principal antimony oxide. The gudmundite occurs largely as coarse grains (plus 100 microns), but also as small inclusions in gangue minerals. A few inclusions are present in the gudmundite grains; these include valentinite, stibnite, native antimony, chalcopyrite, pyrite, pyrrhotite, arsenopyrite, and gangue minerals. They range in size from 2 to 200 microns. The remainder of the stibnite is largely intergrown with pyrrhotite, whereas practically all the valentinite is associated with the gudmundite as rims and inclusions.

Attempts were made to concentrate the antimony values (1) by tabling, (2) by flotation, and (3) by a combination of the two processes. The following results were obtained:

<u>Method</u>	<u>Concentrate Grade (% Sb)</u>	<u>Recovery (% Sb)</u>
Tabling	54.8	42.5
Flotation	49.6	65.1
Tabling and Flotation	41.4	76.3

Removal of stibnite-bearing pyrrhotite could be achieved by magnetic separation with minimal losses of antimony.

A flowsheet incorporating tabling, flotation, and magnetic separation under optimum conditions, produced a concentrate assaying 45.5% Sb with a 77.5% recovery. The impurities in this concentrate were low enough to be acceptable at 0.23% As, 0.15% Cu, 0.08% Pb, 0.09% Zn, 0.03% Ni, 15.9% Fe and 4.7% SiO₂. The antimony lost in the tailing from this flowsheet treatment was mostly in grains finer than 500 mesh (25 microns).

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Direction des Mines
Rapport d'Investigation IR 73-6

Développement d'une procédure pour le traitement
d'un minéral d'antimoine du Canton de Ham-Sud, Quebec

par

G.I. Mathieu* et R.W. Bruce**

- - -

Résumé

A l'analyse, l'échantillon soumis pour fin d'investigation donne les résultats suivants:

<u>Sb</u>	<u>As</u>	<u>S</u>	<u>Fe</u>	<u>Insol</u>
1.25%	0.09%	0.66%	4.10%	79.7%

L'examen au microscope montre l'antimoine généralement sous forme de sulfures et, à un degré moindre, sous forme d'oxides et de métal natif. La gudmundite est le sulfure majeur tandis que la stibnite est beaucoup plus rare. L'oxide principal est la valentinite. La gudmundite se présente en majeure partie en grains relativement grenus de plus de 100 microns; cependant, on en trouve également sous forme de fines inclusions dans la gangue. Quelques inclusions, notamment de valentinite, de stibnite, d'antimoine natif, de chalcopryrite, d'arsénopyrite, de pyrrhotine et de minéraux de gangue apparaissent dans la gudmundite. Celles-ci varient en grandeur de 2 à 200 microns. La plupart du reste de la stibnite apparaît dans le réseau cristallin de la pyrrhotine, alors que la presque totalité de la valentinite est intimement associée à la gudmundite.

Les auteurs ont tenté de concentrer les valeurs d'antimoine, d'abord sur table, puis par flottation, et enfin par une combinaison des deux procédés, et ce avec les résultats qui suivent:

<u>Méthode</u>	<u>Teneur du Concentré (% Sb)</u>	<u>Récupération (% Sb)</u>
Table	54.8	42.5
Flottation	49.6	65.1
Table et Flottation	41.4	76.3

La stibnite contaminée de pyrrhotine peut être enlevée des concentrés par séparation magnétique et sans perte importante d'antimoine.

Avec une procédure incorporant le traitement sur table, la flottation et la séparation magnétique, les auteurs ont produit un concentré de 45.5% en antimoine avec un recouvrement de 77.5% de ce métal. Les impuretés se sont maintenues à un niveau acceptable à 0.23% As, 0.15% Cu, 0.08% Pb, 0.09% Zn, 0.03% Ni, 15.9% Fe et 4.7% SiO₂. L'antimoine perdu au cours du traitement était en majorité dans les granulométries inférieures à 500 mailles (25 microns).

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INTRODUCTION

Property

A sample was submitted by Quebec Antimony Mines Ltd. from an antimony prospect located in South Ham Township, near S.S. Martyrs-Canadiens, Quebec. The latter is on Highway No. 34, about half-way between Victoriaville and Lac Megantic. The reserves of commercial-grade ore in the deposit were estimated at 500,000 tons.

Purpose of Investigation

The investigation was requested by Mr. Alain Matte, President, Quebec Antimony Mines Ltd., 1875 Théoret, Brossard, Quebec. He was interested in a feasibility study on the concentration of the antimony values and in the development of a suitable flowsheet for a 100 ton/day mill. The final concentrate was to contain about 50% Sb and a minimum of arsenic.

Shipment

On October 7, 1971, a 500-lb sample of lump ore was received from the company for investigation. The shipment was sent by Mr. Matte.

Sampling and Analysis

The whole shipment was crushed to minus one inch and a few representative fragments were selected for mineralogical examination. One half of the remainder was reduced to minus 10 mesh, from which a head sample was riffled out by conventional methods for chemical analysis.

TABLE 1

Chemical Analysis of Head Sample*

Antimony (Sb)	-	1.25%
Arsenic (As)	-	0.09%
Sulphur (S)	-	0.66%
Iron (Fe)	-	4.10%
Insoluble	-	79.7%

*From Internal Report MS-AC-71-482

MINERALOGICAL EXAMINATION

A portion of the head sample and several representative pieces of rock were sent to the Mineralogy Section of the Mineral Sciences Division for identification of the minerals and determination of their grain sizes and textural relationships. Excerpts from the mineralogical report* follows.

Summary of Results

Mineralogical studies have been made on a sample of antimony ore from a deposit located in South Ham Township, Quebec, on behalf of Quebec Antimony Mines Limited. The results show that the ore is composed of siliceous gangue minerals, with sparse and disseminated mineralization. Antimony occurs chiefly in the form of sulphides, with much less in the form of oxides, and as traces of native metal. The antimony sulphides include gudmundite, stibnite, and tetrahedrite; the antimony oxides include valentinite, senarmonite, kermesite,

*From Mines Branch Investigation Report IR 72-13 by D.R. Owens, March 27, 1972.

and an unidentified iron antimonide. Other minerals identified are pyrrhotite, pyrite, marcasite, chalcopyrite, galena, sphalerite, rutile, ilmenite, goethite, arsenopyrite, quartz, feldspar, mica, chlorite, zircon, and dolomite.

Method of Investigation

Polished sections and thin sections were prepared from a number of selected hand specimens, and examined microscopically to identify the minerals present and to determine their grain sizes and textural relationships. The 48- to 270-mesh fraction was screened from the head sample. This fraction was separated into an ore mineral concentrate and a gangue product by means of heavy liquids. Polished sections were prepared from the concentrate and examined microscopically to permit a comparison of the ore minerals* in the head sample with those in the hand specimens. X-ray diffractometer analyses were made on the gangue product of the head sample to substantiate the identities of the principal gangue minerals. The identities of some of the ore minerals could not be determined by ore microscopy alone but were established by X-ray powder diffraction analyses; other ore minerals were studied by the electron microprobe to determine significant trace elements.

Results of Investigation

General Mineralogy of the Ore

The hand specimens do not appear to be representative of the ore because they are characterized by a scarcity of pyrrhotite, whereas pyrrhotite is the second most prevalent ore mineral in the head sample. Nevertheless they had to be used for determining the grain sizes and the textural relationships.

*The term "ore minerals" as used in this report, does not necessarily have an economic connotation.

A microscopic examination (examination) of a portion of the head sample (deemed more representative of the ore than the hand specimen) reveals that the principal ore mineral is gudmundite. Smaller amounts of pyrrhotite and less stibnite and valentinite are also present. Pyrite, chalcopyrite, marcasite, sphalerite, galena, native antimony, kermesite, ilmenite, senarmontite, rutile, tetrahedrite, goethite, and an unidentified iron antimonide occur in small to trace amounts.

Detailed Mineralogy of the Ore

(a) Antimony-Bearing Minerals

The antimony-bearing minerals identified are gudmundite (FeSbS), stibnite (Sb_2S_3), valentinite [(Sb_2O_3) -orthorhombic], kermesite ($\text{Sb}_2\text{S}_2\text{O}$), native antimony, tetrahedrite [$(\text{Cu,Zn,Fe})_{12}\text{Sb}_4\text{S}_{13}$], senarmontite [(Sb_2O_3) -cubic], and an unidentified iron antimonide which may be schafarzikite ($\text{Fe}_5\text{Sb}_4\text{O}_{11}$). The amount of antimony as sulphides is greater than that as oxides and native metal combined.

Gudmundite is the dominant antimony sulphide. It occurs largely as coarse grains, disseminated in gangue, some of which are surrounded and penetrated by valentinite. These disseminated grains range from 10 microns to about six millimetres in size* and the majority are larger than 100 microns. Small amounts of gudmundite have been partly replaced by antimony oxides such as valentinite, kermesite, senarmontite, and the unidentified iron antimonide, and, in a few places, only remnants of gudmundite are left. In addition, some grains of gudmundite are present as inclusions in stibnite and native antimony and a few are intergrown with either pyrrhotite or native antimony in gangue. These grains range from about 5 to 300 microns in size. Only a few inclusions are

*The word "size", as used in this report, refers to the greatest dimension of the mineral grain being described.

present in the gudmundite. They include tetrahedrite, native antimony, stibnite, chalcopyrite, rutile, pyrite, pyrrhotite, arsenopyrite, and gangue. These inclusions range from 2 to about 300 microns in size.

Stibnite is present as laths, interconnected grains, thin discontinuous veinlets in gangue, large aggregates intergrown with pyrrhotite and marcasite, and inclusions in gudmundite. The laths range from 15 to 150 microns in size, and the veinlets are up to 15 microns wide. The large aggregates range from 0.5 to 3.0 millimetres in size. Some of the stibnite is closely associated with pyrrhotite and marcasite as inclusions in pyrrhotite, as intimate fine-grained intergrowths, and as grains with pyrrhotite cores and numerous minute inclusions of marcasite. In most of these associations, the stibnite grains rarely exceed 30 microns in size. In a few places, the stibnite is associated with rutile in gangue; in other places, it has been partly replaced by kermesite-senarmontite intergrowths. Some of the large stibnite grains contain inclusions of gudmundite, rutile, and gangue; these inclusions range in size from 15 to 300 microns.

The native antimony is present as small inclusions in gudmundite and as somewhat larger grains in gangue. Those in gudmundite are generally smaller than 35 microns in size but those in gangue range from about 15 to 500 microns. A few of the larger native antimony grains contain inclusions of gudmundite, chalcopyrite, and tetrahedrite; these are less than 25 microns in size.

Most of the tetrahedrite occurs as small inclusions in gudmundite but some is present as inclusions in gangue and as intergrowths with chalcopyrite, pyrite, and sphalerite. The inclusions in gudmundite range from 10 to 150 microns in size; the grains in gangue are somewhat larger with a maximum dimension of 250 microns.

Valentinite is the principal antimony oxide. It occurs largely in association with the gudmundite as rims and inclusions. The largest valentinite grains were found in this association and they range from 15 to 400 microns in size. The mineral also occurs in an intimate replacement texture with kermesite, where it contains remnants of gudmundite (Figure 3). It is also present as grains in gangue and, occasionally, as a replacement of stibnite. The grains in gangue range in size from 10 to 300 microns.

X-ray diffraction analyses show that the kermesite is present in both crystalline and amorphous states. It was, at first, felt that some of the amorphous grains might be metastibnite (an amorphous form of Sb_2S_3) because the optical and physical properties of metastibnite are very similar to those of kermesite. However, the replacement textures indicate kermesite. The kermesite occurs primarily in association with valentinite, replacing the antimony sulphides. Smaller amounts are also associated with senarmontite replacing both stibnite and gudmundite. Traces of kermesite are present as inclusions in gangue and gudmundite, and these range in size from 5 to 150 microns.

Senarmontite is present only in trace amounts. It was found only as an intergrowth with kermesite replacing gudmundite and stibnite.

The fourth antimony-bearing oxide is an iron antimonide which may be schafarzikite. X-ray diffraction analyses were inconclusive in establishing its identity as the patterns obtained were poor. However, electron microprobe studies show that it is an iron-antimony oxide, with a variable ratio of Fe to Sb, and a composition close to that of schafarzikite. This mineral occurs as a number of grains in gangue, most of which contain remnants of gudmundite. These grains range from 25 to about 300 microns in size.

(b) Other Ore Minerals

Pyrrhotite is the most significant of the metallic minerals. Most of it is associated with stibnite but some occurs as discrete grains, 15 to 325 microns in size, disseminated in gangue, and as small inclusions in gudmundite. Most of the pyrrhotite either contains inclusions of marcasite or is interlocked with grains of marcasite.

Pyrite, chalcopyrite, sphalerite, and galena are present in successively smaller amounts. Both pyrite and chalcopyrite occur mainly as disseminations in gangue. Trace amounts of each form inclusions in gudmundite, native antimony, and kermesite and form interlocked grains with sphalerite and tetrahedrite in gangue. Both minerals are of the same grain size. The disseminated pyrite and chalcopyrite range from 5 to about 325 microns in size; and the inclusions are from 5 to 250 microns in size.

The sphalerite and galena are present in insignificant amounts. The sphalerite, from 10 to 150 microns in size, occurs as grains in gangue and interlocked with pyrite, tetrahedrite, and chalcopyrite. Galena occurs as a few minute inclusions, all smaller than 25 microns, in a valentinite-kermesite intergrowth.

The amount of arsenopyrite is insignificant. Only a few inclusions, up to 30 microns in size, were found in gudmundite.

Rutile is one of the most prevalent minerals. It occurs largely as disseminated grains and clusters of grains in gangue. These grains range in size from 5 to about 100 microns. A few grains also occur as inclusions in gudmundite and stibnite. Trace amounts of ilmenite occur in a manner similar to that of the rutile. All the ilmenite grains are smaller than 25 microns.

(c) Gangue Minerals

The gangue minerals, in their order of abundance, are quartz, feldspar, chlorite, mica, dolomite, and zircon.

Conclusions

The antimony is present largely as sulphides. Gudmundite is the most prevalent ore mineral, but the amount of stibnite is also significant. The gudmundite is quite coarse-grained and most of it should be liberated. Some of the stibnite is coarse-grained and free from intergrowths but much of it is fine-grained and locked in pyrrhotite, from which liberation will be difficult.

Some antimony occurs as oxides, and many of these replace and corrode the antimony sulphides. Valentinite is the principal antimony oxide. It is relatively coarse-grained and much of it can be liberated.

The other antimony minerals; native antimony, tetrahedrite, kermesite, senarmontite, and schafarzikite? are intergrown with gudmundite, stibnite, and valentinite; therefore, some of these will be recovered with them.

The amount of chalcopyrite, galena, and sphalerite, present in the ore, is considered too small to be of economic value.

OUTLINE OF INVESTIGATION

The initial testwork was oriented towards gravity concentration with the aim of recovering both the sulphide and oxide antimony-bearing minerals. Table concentration was investigated and the size of the feed was the main variable under study. Because this gravity treatment failed to recover the fine (friable) sulphides, flotation concentration was tested for recovering

the antimony values. However, as conventional flotation recovered little of the antimony oxides, a combination of tabling and flotation was investigated for a better over-all recovery, despite the company's indication that the antimony oxides in the surface ore submitted would not likely be representative of the main orebody. Finally, attempts were made to remove the stibnite-bearing pyrrhotite from both the gravity and flotation concentrates by magnetic separation.

Using the information gathered in this investigative testwork, a flowsheet that involved tabling, flotation, and magnetic separation was developed for the effective concentration of the Quebec Antimony ore.

DETAILS OF INVESTIGATION

Gravity Concentration by Tabling, Tests 1-3

Three tests were conducted on samples of ore ground to minus 48, 65 and 80 mesh, respectively. In each case, a 4,000-g sample was passed over a laboratory Deister table and four products were retained and analysed, namely, a first and a second table concentrate, a middling, and a tailing. The antimony analyses and the corresponding distribution of the metal are given in Table 2.

TABLE 2
Results of Tabling Tests

Test	Product	Weight %	Analysis % Sb	Distribution % Sb
1 (-48 m)	Sb conc #1	1.0	53.6	42.6
	Sb conc #2	1.0	29.9	21.4
	Table middling	23.6	0.65	11.0
	Tailing	74.4	0.41	25.0
	Feed (calcd)	100.0	1.26	100.0
2 (-65 m)	Sb conc #1	1.1	45.5	44.3
	Sb conc #2	0.8	19.0	13.4
	Table middling	16.7	0.72	10.6
	Tailing	81.4	0.44	31.7
	Feed (calcd)	100.0	1.13	100.0
3 (-80 m)	Sb conc #1	1.0	54.8	42.6
	Sb conc #2	0.9	22.6	15.8
	Table middling	21.4	1.14	19.0
	Tailing	76.7	0.38	22.6
	Feed (calcd)	100.0	1.29	100.0
	Combined conc (No. 1 + No. 2)	1.9	39.5	58.4

*From Internal Reports MS-AC-72-15, 22 and 28.

Mineralogical examination* of the table concentrates indicated that they consist essentially of gudmundite, stibnite, and valentinite, with minor amounts of native antimony (and schafarzikite?). The main metallic impurities identified were pyrite and pyrrhotite. Traces of marcasite, galena, chalcopyrite, magnetite, rutile and hematite were also observed. All the metallic impurities, and the pyrrhotite in particular, were more abundant in Sb concentrate No. 2.

*From Internal Report MS-72-4 by D.R. Owens.

Little gangue was found in any of the table concentrates, albeit a few grains of zircon were commonly present in the first concentrates and some quartz, in the second.

Removal of the pyrrhotite from the table concentrates would improve the grade of the final product. However, this will result in a significant decrease in the recovery of antimony because an appreciable proportion of the stibnite is intimately associated with the pyrrhotite.

Examination of the table tailings revealed important losses of antimony in the form of fine sulphide minerals (gudmundite and stibnite). This prompted investigation of flotation techniques for a better recovery of the antimony-bearing sulphides.

Flotation Concentration

The initial flotation tests were made directly on the ore. They consist of (i) rougher flotation, (ii) cleaner flotation, and (iii) integrated rougher and cleaner flotation under optimum conditions.

(i) Rougher Flotation, Tests 4 to 9

Preliminary rougher flotation tests were made at increasing degrees of fineness in order to determine the minimum grinding necessary for the liberation of the antimony sulphide minerals. The three grinds investigated were minus 80, 100, and 150 mesh, respectively. Two series of tests were conducted at each grind, the first using lead acetate as antimony activator (Series A) and second, using sulphuric acid* for the same purpose (Series B). All the tests were made

*This method has been developed recently by the authors and is described in Report MPI(O) 72-16, "Activation of Antimony Minerals for Flotation - An Empirical Study", August 1972.

on 2,000-g samples using a laboratory Denver cell. The flotation procedure is shown in detail in Table 3 and the results are given in Table 4.

TABLE 3
Reagents and Conditions of Rougher Flotation

Operation	Time min	Reagents*	lb/ton		pH
			Series A	Series B	
Conditioning	2	Lead acetate	0.30	-	6.9
		Sulphuric acid	-	2.5	5.8
Rougher flotation	18	Lead acetate	0.30	-	6.9
		Sulphuric acid	-	1.0	5.6
		Xanthate Z-6	0.20	0.20	
		Dowfroth 250	0.08	0.08	

*During rougher flotation, the reagents were added by stages at 6-minute intervals.

TABLE 4
Results of Rougher Flotation

Test	Product	Series A (Lead Acetate)			Series B (H ₂ SO ₄)		
		Weight %	Analysis % Sb	Dist'n % Sb	Weight %	Analysis % Sb	Dist'n % Sb
4 (-80 m)	Sb conc	11.6	10.1	77.7	9.5	13.7	80.4
	Flot tailing	88.4	0.38	22.3	90.5	0.35	19.6
	Feed (calcd)	100.0	1.51	100.0	100.0	1.62	100.0
5 (-100 m)	Sb conc	10.9	11.1	84.0	10.7	11.5	83.1
	Flot tailing	89.1	0.26	16.0	89.3	0.28	16.9
	Feed (calcd)	100.0	14.5	100.0	100.0	1.48	100.0
6 (-150 m)	Sb conc	11.9	8.83	74.9	12.4	10.3	78.8
	Flot tailing	88.1	0.40	25.1	87.6	0.38	21.2
	Feed (calcd)	100.0	1.48	100.0	100.0	1.62	100.0

*From Internal Reports MS-AC-72-13, 137, 239 and 293.

Microscopic examination of the flotation tailings indicated the presence of appreciable antimony oxide minerals listed below in their order of decreasing abundance: valentinite, kermesite, senarmontite, and an unknown iron-antimony oxide. Very little antimony sulphide (gudmundite and stibnite) was observed in the flotation tailings. Most of it was present as very fine inclusions in gangue minerals, although a few free grains of gudmundite and stibnite could be found in the tailing samples from Tests 4 and 6. This limits the maximum antimony, recoverable by flotation, to about 85%; the remaining values (0.25% Sb) being in the form of either oxides or sulphide inclusions, both unrecoverable by conventional flotation under normal conditions. Primary grinding to minus 100 mesh appeared necessary to achieve the maximum recovery.

Mineralogical examination of the flotation concentrates was done after the cleaning stages.

(ii) Cleaner Flotation, Tests 7-8

The rougher flotation concentrates from each series of the preceding tests were combined for the cleaning stages. During this operation, the concentrates were filtered and washed by a water spray, to remove excess reagents, and briefly reground to refresh mineral surfaces. The conditions of cleaner flotation and its results are shown in Tables 5 and 6, respectively.

TABLE 5

Reagents and Conditions of Cleaner Flotation

Operation	Reagents and Conditions	Test 7	Test 8
		(PbAc)*	(H ₂ SO ₄)
1st Cleaning	Reagents (lb/ton of ore):		
	H ₂ SO ₄	nil	0.05
	Pb Acetate	0.05	nil
	Z-6	0.02	0.02
	Dowfroth 250	0.02	0.02
	Time (min)	7	8
	pH	6.8 - 7.0	5.1 - 5.5
Washing/Filtering	Hot and cold water		
2nd Cleaning	Reagents (lb/ton of ore):		
	H ₂ SO ₄	nil	0.06
	Pb Acetate	0.05	nil
	Z-6	0.02	0.03
	Dowfroth 250	0.02	0.02
	Time (min)	5	7
	pH	6.9 - 7.0	5.0 - 5.4
3rd Cleaning	Reagents (lb/ton of ore):		
	H ₂ SO ₄	nil	0.03
	Pb Acetate	0.03	nil
	Z-6	0.02	0.02
	Dowfroth 250	0.01	0.01
	Time (min)	4	5
	pH	6.9 - 7.1	4.7 - 5.3
Regrinding	3 min (87% - 200 m)		
4th Cleaning	Reagents (lb/ton of ore):		
	H ₂ SO ₄	nil	0.05
	Pb Acetate	0.05	nil
	Z-6	0.02	0.03
	Dowfroth 250	0.02	0.02
	Time (min)	5	6
	pH	7.0 - 7.3	4.5 - 5.4

*Lead acetate

TABLE 6

Results of Cleaner Flotation

Test	Products	Weight %	Analysis*		Distribution % Sb	
			Sb	Insol	In fraction	Overall
7 (Pb Acetate)	Sb cl conc	31.4	29.0	21.6	89.1	70.1
	Cleaner tailings	68.6	1.63	66.0	10.9	8.6
	Feed (calcd)	100.0	10.2	52.1	100.0	78.1
8 (H ₂ SO ₄)	Sb cl conc	31.0	31.5	15.8	89.0	71.7
	Cleaner tailings	69.0	1.76	67.0	11.0	8.9
	Feed (calcd)	100.0	11.0	51.1	100.0	80.6

*From Internal Report MS-AC-72-245.

Microscopic examination of representative samples (and sized fractions) of each flotation concentrate showed the main gangue impurities to be mostly quartz and a little feldspar. Although some small free grains of these minerals could be observed, the majority appears as relatively coarse particles (larger than 75 micromillimeters) with attachments of pyrite, arsenopyrite, and antimony sulphide minerals, i.e., gudmundite and stibnite. These attached minerals would be liberated by selective grinding of the plus 325-mesh fraction of the flotation concentrates.

The main metallic impurity in the concentrates is pyrrhotite, but pyrite is also abundant. In addition to these, both types of concentrates contain arsenopyrite, sphalerite, and chalcopyrite, in small but significant amounts, and traces of other metal sulphides and oxides. Most of the sulphide impurities appears as discrete grains free of inclusions; but pyrrhotite contains many inclusions of stibnite.

During the course of the mineralogical examination, it was observed that metallic minerals were the dominant impurities in the concentrates from tests in which sulphuric acid was used, while the gangue contaminants were more abundant when lead acetate served as activator*. From this, it was inferred that both types of impurities could be minimized by activating the antimony minerals in the rougher flotation with lead acetate and in the cleaning steps with sulphuric acid.

Obviously, the pyrrhotite in the flotation concentrates can be separated magnetically, albeit with some losses** of stibnite associated with that mineral.

(iii) Integrated Flotation, Test 9

The ore sample was treated by a procedure incorporating the best features of previous flotation testwork and observations, including:

- (1) grinding 4,000 grams in stages to minus 100 mesh;
- (2) rougher flotation using lead acetate to activate antimony minerals;
cleaner flotation using sulphuric acid as activators;
- (3) regrinding of the plus 325-mesh fraction before the final cleaning;
- (4) magnetic separation of the stibnite-bearing pyrrhotite from the final flotation concentrate.

Flotation data for rougher, scavenger, and five cleaning stages are outlined in Table 7. Pertinent analytical results are given in Table 8.

*This observation is supported by the "acid insoluble" analyses of the concentrates: 21.6% (with Pb acetate) versus 15.8 (with H₂SO₄).

**To assess these losses, concentrates were passed through a Jones high-intensity magnetic separator set at 5 amperes with the following results:

Concentrate grade, % Sb		Sb loss, %
Before	After	(in magnetic pyrrhotite)
31.5	37.6	2.9
29.0	36.9	3.2

TABLE 7

Reagents and Conditions of Integrated Flotation

Operation	Time Min.	Reagents	lb/ton	pH
Rougher Flotation	20	Pb acetate Z-6 Dowfroth 250	0.50 0.15 0.07	6.8
Scavenger Flotation	10	Pb acetate Z-6 Dowfroth 250	0.20 0.05 0.03	6.9
Washing/Filtering	-	Water	-	-
1st Cleaning	9	H ₂ SO ₄ Z-6 Dowfroth 250 pH	0.10 0.03 0.02	5.4 - 5.6
2nd Cleaning	7	H ₂ SO ₄ Z-6 Dowfroth 250	0.03 0.02 0.01	5.3 - 5.4
Regrinding	2	nil	(52.5% - 325 m)	(49.1% - 325 m)
3rd Cleaning	6	H ₂ SO ₄ Z-6 Dowfroth 250	0.04 0.02 0.02	5.1 - 5.4
4th Cleaning	5	H ₂ SO ₄ Z-6 Dowfroth 250	0.02 0.01 0.01	4.3 - 4.7
Regrinding +325 m Fraction	10	nil	(98.6% - 325 m)	(96.2% - 325 m)
5th Cleaning	5	H ₂ SO ₄ Z-6 Dowfroth 250	0.02 0.01 0.01	4.1 - 4.3

Note: The 5th cleaner flotation concentrate was passed through a Jones magnetic separator set at 5 amperes.

TABLE 8

Results of Integrated Flotation

Products	Weight %	Analysis %		Distribution %	
		Sb	Insol	Sb	Insol
Sb final conc	2.0	49.6	7.0	65.0	0.1
Pyrrhotite conc	0.3	14.4	3.1	2.8	0.0
5th cl tailing	0.3	24.1	40.3	4.8	0.1
1st - 4th cl tailings	7.6	1.44	63.0	7.1	5.2
Scavenger conc	2.2	1.41	81.5	2.0	2.0
Flotation tailing	87.6	0.32	95.3	18.3	92.6
Feed (calcd)	100.0	1.52	90.3	100.0	100.0

The final antimony concentrate, the pyrrhotite concentrate, and the flotation tailing were examined microscopically with the following results.* The principal ore mineral in the final concentrate is gudmundite. Small amounts of stibnite, pyrite, quartz, zircon and feldspar accounts for most of the remainder. Most of the above minerals are present as free grains. Other minerals present in trace amounts in the concentrate include chalcopyrite, marcasite, pyrrhotite, goethite, sphalerite, galena, tetrahedrite, valentinite, kermesite, senarmonite, and rutile. The latter five minerals occur largely as small inclusions in the gudmundite, while the others are present essentially as free grains. Therefore, it is believed that further grinding will not appreciably increase mineral liberation but that additional cleaning of the final concentrate might slightly upgrade it by removing the free impurities. As gudmundite, which only contains 58.1% Sb, is the dominant antimony mineral in the flotation concentrate, the maximum possible grade of the concentrate will not exceed this figure.

*From private communication with D.R. Owens of the Mineralogy Section, and from Internal Reports MS-AC-72-4 and 12.

The pyrrhotite (magnetic) concentrate consists largely of grains of pyrrhotite, many of which contain small inclusions and/or adhering particles of stibnite. Some marcasite and gudmundite grains are also locked in the pyrrhotite. The balance of the concentrate is made up of tramp iron and free grains of magnetite, zircon, gudmundite, and quartz. It is suggested that the quartz, zircon, and gudmundite grains were entrapped during concentration. These should be removed by a cleaning stage with a magnetic separator.

The antimony content of the flotation tailing consists essentially of the oxides present in the ore, valentinite in particular. This suggested that such minerals are resistant to conventional flotation and should be recovered by another means, possibly by gravity concentration prior to flotation.

Flowsheet Development, Tests 10 and 11

An initial flowsheet was tested with two objectives, namely,

- 1) to integrate the findings from previous gravity concentration, flotation, and mineralogical examination;
- 2) to gather information for the development of a final proposed procedure for effective concentration of the antimony minerals in the ore, i.e., a flowsheet aiming at optimum grade and recovery.

The latter objective explains the separate retention and analysis of all the intermediate products in the first test.

The treatment pattern was devised to minimize formation of slimes which resist both flotation and gravity concentration and to recover the antimony values as soon as liberated. The features of the procedure can be summarized as follows:

- (1) stage-grinding;
- (2) primary concentration by tabling at minus 48 mesh;

- (3) recovery of the residual antimony by re-tabling at minus 100 mesh and by flotation of the table tailing; and
- (4) up-grading of the concentrates produced in (3) by magnetic separation of the pyrrhotite and cleaner flotation in presence of sulphuric acid.

The flowsheet is illustrated in Figure 1; the reagents and conditions of flotation are listed in Table 9; and the results are given in Table 10.

TABLE 9

Reagents and Conditions of Flotation with Flowsheet I

Feed	Operation	Time min	Reagent	lb/ton of ore	pH
Table Concentrate	Flotation	5	Sulphuric acid	0.05	4.8
			Xanthate Z-6	0.003	
			Dowfroth 250	0.003	
Table Tailing	Rougher flotation	20	Lead acetate	0.40	6.9
	1st cleaner flotation	8	Xanthate Z-6	0.12	5.5
			Dowfroth 250	0.06	
			Sulphuric acid	0.10	
	2nd cleaner "	6	Xanthate Z-6	0.02	5.1
			Dowfroth 250	0.02	
			Sulphuric acid	0.04	
	3rd cleaner "	5	Xanthate Z-6	0.01	4.9
			Dowfroth 250	0.01	
			Sulphuric acid	0.02	

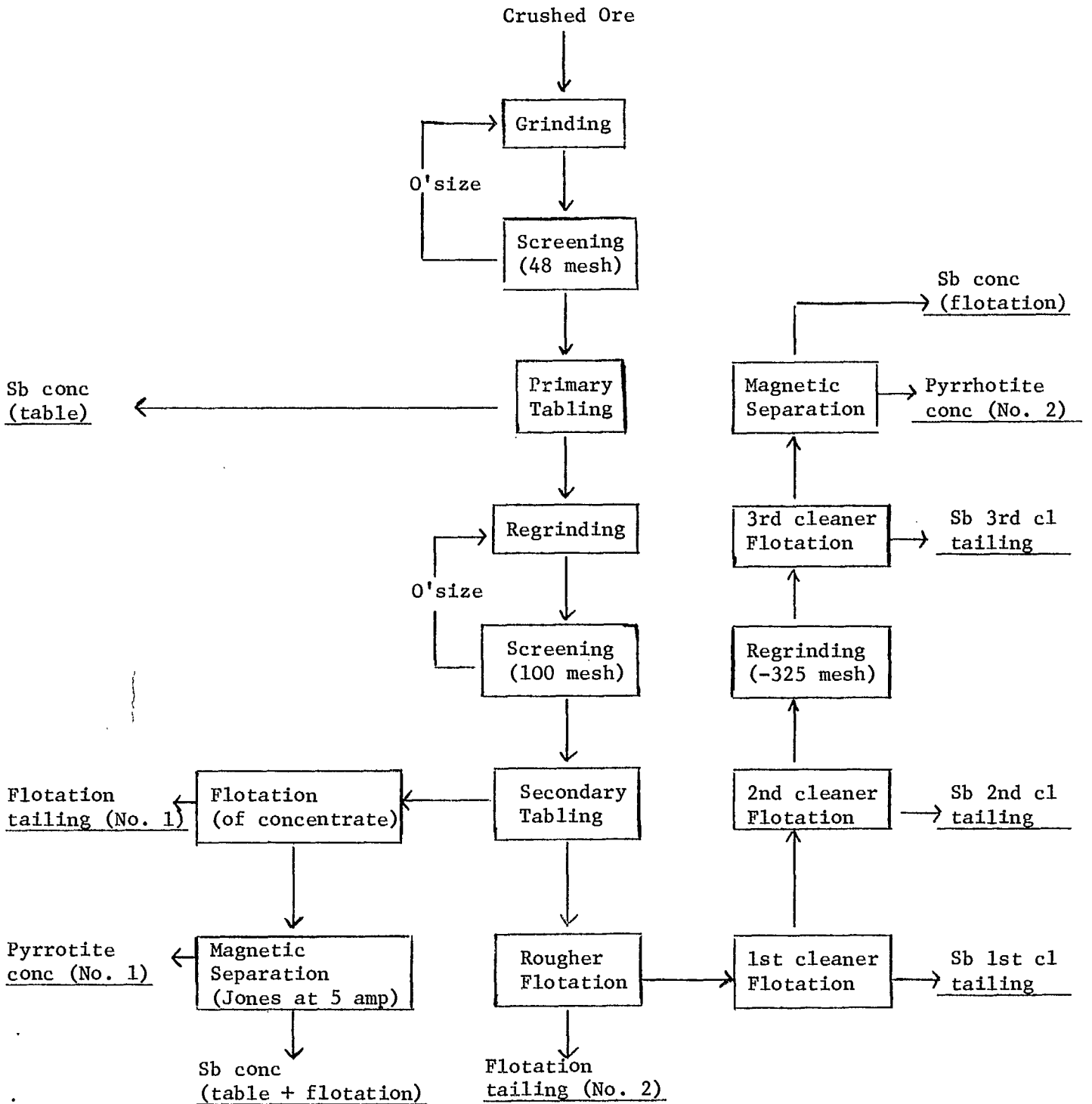


Figure 1 - Flowsheet I

TABLE 10

Results of Antimony Concentration by Flowsheet I⁽¹⁾

Operation	Product	Weight %	Analysis* % Sb	Distribution % Sb
Primary concentration (Tabling)	Sb conc (table)	1.1	53.1	44.5
Secondary concentration (Tabling, flotation and magnetic separation)	Sb conc (table + flotation)	0.6	42.1	21.2
	Flotation tailing (No. 1)	0.2	12.4	2.1
	Pyrrhotite conc (No. 1)	0.1	9.2	0.8
Tertiary concentration (Flotation and magnetic separation)	Sb conc (flotation)	0.5	25.4	10.6
	Pyrrhotite conc (No. 2)	0.1	20.3	1.7
	Sb 3rd cl tailing	0.2	9.2	1.5
	Sb 2nd cl tailing	2.2	3.6	6.6
	Sb 1st cl tailing	4.3	0.29	1.1
	Flotation tailing (No. 2)	90.7	0.13	9.9
Feed (calculated)		100.0	1.19	100.0
Sb concentrates (combined)		2.2	41.4	76.3

*From Internal Reports MS-AC-72-28, 72 and 107.

(1) Note: Further tests were made with this basic flowsheet, in which the primary tabling was conducted at minus 65 mesh and the secondary tabling and flotation at minus either 80 or 150 mesh. Analyses of a few selected products from these tests indicated no significant improvement over the above results.

Mineralogical studies* were conducted on the various concentrates produced in this test, the results of which are summarized below.

(a) Table concentrate

This concentrate contains approximately 90 per cent sulphides, 8 per cent silicates, and 2 per cent oxides and metals. The sulphides are gudmundite, small amounts of pyrite, and trace amounts of pyrrhotite, marcasite, stibnite, galena, and chalcopyrite. The sulphides that are present in trace amounts occur mainly as inclusions in the gudmundite and pyrite. The only significant silicate in the concentrate is zircon which occurs as well-rounded free grains. The oxides are valentinite, magnetite, hematite, rutile, and an unidentified iron-antimony oxide. A few grains of native antimony occur in the concentrate. A visual estimate was made of the quantities of minerals in the table concentrate by examining a polished section under the ore microscope. The results are given in Table 11.

TABLE 11

Estimated Quantities of the Minerals in the Table Concentrate

<u>Minerals</u>	<u>Per Cent</u>
Gudmundite	85
Zircon	7
Pyrite	5
Valentinite	1
Other Sulphides	1
Other Oxides	1

*From Internal Report MS-72-4 by D.R. Owens.

(b) Concentrate (Table + Flotation)

Estimates of the approximate percentages of the individual minerals in this concentrate are very difficult to establish because of the diversity of the minerals present, their large variations in grain size (5 to 200 microns), and the optical similarity of many of the non-metallic minerals. A rough estimate shows that the flotation concentrate is composed of about 80 per cent sulphides, 20 per cent oxides and silicates, and trace amounts of native antimony. The gudmundite content is between 50 and 60 per cent, pyrite between 10 and 15 per cent, stibnite about 5 per cent, and the remaining sulphides, pyrrhotite, chalcopyrite, marcasite, galena and tetrahedrite, about one per cent.

Slightly more than one half of the remainder of the flotation concentrate is composed of silicates which include zircon, quartz, and feldspar. The principal oxide is valentinite. Some rutile, as well as traces of magnetite, goethite, senarmontite, and the unknown iron-antimony oxide are also present.

(c) Pyrrhotite concentrates

Examination of these concentrates confirmed the close association of stibnite and pyrrhotite as well as the presence of free grains of gudmundite, zircon, and quartz.

(d) Flotation Concentrate

This mill product is extremely fine-grained, with particles varying from 1 to about 30 microns in size. The estimated sulphide content is 70 per cent, and the oxides plus silicates is 30 per cent. Due to the diversity of minerals that occur in this concentrate and to their small grain size, it was not possible to quantitatively assess the proportions of the various minerals. However, it can be stated that the principal sulphide is gudmundite and that

the other main sulphides are pyrite and stibnite. Other sulphides include chalcopyrite, pyrrhotite, galena and sphalerite. The principal silicates are quartz, feldspar, and zircon; the oxides are valentinite, rutile, and goethite.

From a mineral processing point of view, the general conclusions of the mineralogical study on the various antimony concentrates are that all these could be upgraded by further cleaning. The use of selective depressants for pyrite and for gangue minerals during the flotation should also increase the grade of the final products. For instance, addition of sodium cyanide (pyrite depressant) and of sodium silicate (dispersant of siliceous gangue) in the rougher and primary cleaner flotation stage might be beneficial in this respect, albeit their effect would be lost when the subsequent addition of sulphuric acid is made. However, sulphuric acid is also a gangue depressant.

The second flowsheet retained the main features of the first, but also aimed at the simplification of the treatment procedure by eliminating some unnecessary (or redundant) steps in the operation. For instance, the cleaner flotation and the magnetic separation stages of the secondary and tertiary concentration were combined. The other features of the flowsheet were as follows:

- 1) production of only one final concentrate;
- 2) addition of pyrite and gangue minerals depressants in the rougher and primary cleaner flotation circuits;
- 3) expansion of the rougher flotation, cleaner flotation and magnetic separation circuits.

The resulting procedure is illustrated in Figure 2. The flotation reagents and conditions and the results obtained are shown in Tables 12 and 13, respectively.

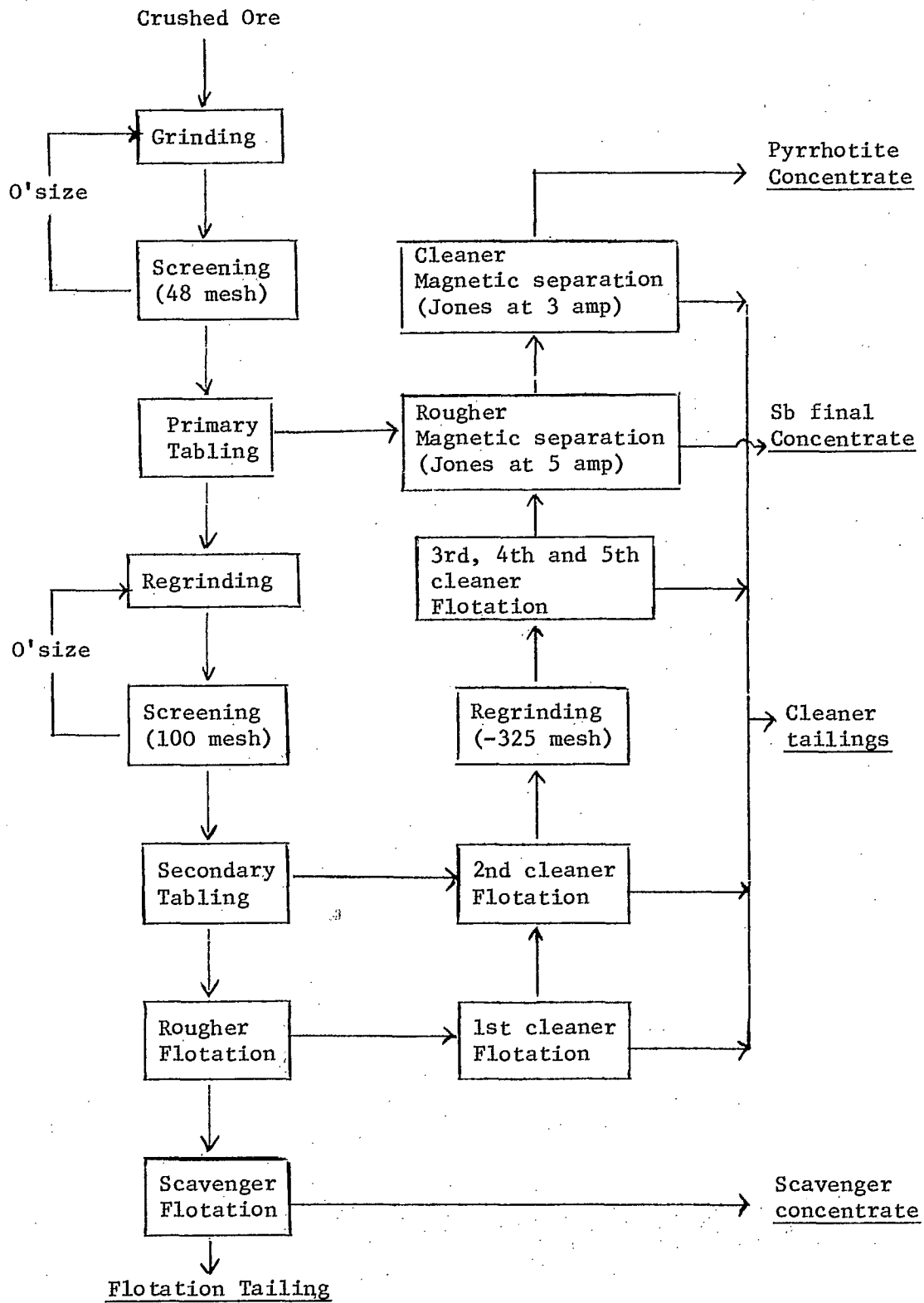


Figure 2 - Flowsheet II

TABLE 12

Reagents and Conditions of Flotation

Operation	Time min	Reagents	lb/ton	pH
Rougher flotation	18	Sodium silicate Sodium cyanide Lead acetate Xanthate Z-6 Dowfroth 250	0.15 0.15 0.40 0.12 0.06	8.5
Scavenger "	6	Lead acetate Xanthate Z-6 Dowfroth 250	0.15 0.04 0.02	8.4
1st cleaner flotation	8	Sodium silicate Sodium cyanide Lead acetate Xanthate Z-6 Dowfroth 250	0.04 0.04 0.04 0.02 0.02	8.7
Washing/Filtering	-	Water		
2nd cleaner flotation	7	Lead acetate Xanthate Z-6 Dowfroth 250	0.04 0.02 0.02	8.3
Regrinding	6			
3rd cleaner flotation		Sulphuric acid Xanthate Z-6 Dowfroth 250	0.10 0.03 0.02	5.5
4th and 5th cleaner flotation	5	Sulphuric acid Xanthate Z-6 Dowfroth 250	0.03 0.01 0.01	5.1

TABLE 13

Results of Antimony Concentration by Flowsheet II

Product	Weight %	Analysis* % Sb	Distribution % Sb
Sb final conc	2.1	45.5	77.5
Pyrrhotite conc	0.2	15.8	2.6
Cleaner tailings	7.4	1.58	9.5
Scavenger conc	1.1	1.89	1.7
Flotation tailing	90.2	0.12	8.7
Feed (calcd)	100.0	1.23	100.0

Additional analyses on the Sb final concentrate:

<u>S</u>	<u>As</u>	<u>Cu</u>	<u>Pb</u>	<u>Zn</u>	<u>Ni</u>	<u>Fe</u>	<u>Insol</u>
15.1%	0.23%	0.15%	0.08%	0.09%	0.03%	15.9%	4.7%

*From Internal Reports MS-AC-72-22, 59 and 508.

Mineralogical studies on a representative portion of the antimony concentrate indicated that most of these impurities occurred as inclusions in the gudmundite, with the exception of some grains of pyrite and quartz. However, since sodium cyanide and silicate were used in this test and as the cleaner flotation circuit was expanded to practically a maximum, further attempts to upgrade the concentrate did not appear worthwhile.

A screen test was made on the flotation tailing to determine the size of the antimony losses. Weighing and analyses of Tyler screen fractions from plus 150 mesh up to minus 500 mesh led to the following distribution.

TABLE 14

Screen Analysis of Flotation Tailing

Size mesh	Weight %	Analysis* % Sb	Distribution % Sb
+150	15.0	0.09	8.9
-150+200	20.0	0.10	13.2
-200+270	15.4	0.11	11.2
-270+325	10.3	0.14	8.8
-325+500	18.3	0.13	15.7
-500	20.0	0.32	42.2
Total	100.0	0.15	100.0

*From Internal Report 72-22.

The high concentration of antimony in the minus 500 mesh fraction (i.e. a size which resists both gravity and flotation concentration) indicated that a practical maximum recovery was attained.

Other screen analyses were made on a several intermediate products to determine if some of these could be directly discardable. Only the plus 200 mesh fractions of the secondary table middling and tailing proved to be relatively low in antimony content with assays of 0.27% and 0.07% Sb, respectively. In plant operation, a careful control of this point might be worthwhile, as the rejection of the plus 200 fraction of the secondary table tailing could very well reduce the flotation feed by a third without an appreciable loss of antimony.

Further simplification of the flowsheet was attempted by the elimination of all the secondary tabling treatment (i.e., using only primary tabling and flotation); however, this approach resulted in a final concentrate of lower grade and recovery. Therefore, it is recommended that essentially the procedure of Flowsheet II be used for the treatment of the Quebec Antimony ore. The latter

is reproduced below with provisions for recirculation of intermediate products, as commonly used in plant practice.

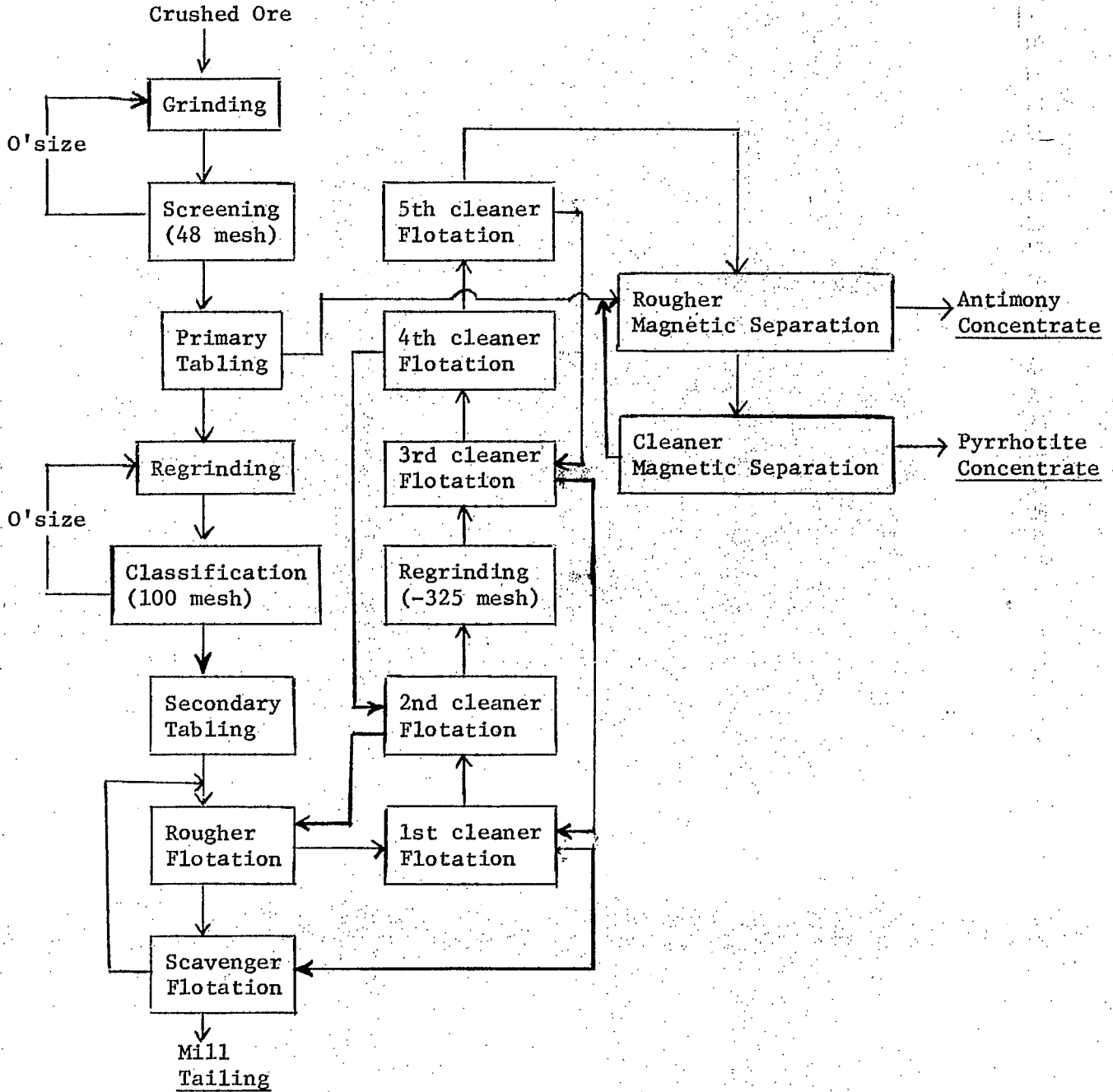


Figure 3 - Proposed Flowsheet for Plant Operation.

DISCUSSION AND CONCLUSION

Gravity concentration by tabling recovered up to 77% of the antimony in the ore in the form of a rougher concentrate containing 43% Sb (calculated). The high losses in this procedure were attributed to the presence of fine antimony sulphides not suitable for gravity treatment. Rougher flotation resulted in an 82% recovery with the main losses occurring as unfloatable antimony oxides as well as five inclusions of gudmundite (the dominant antimony sulphide) in gangue minerals. A combination of tabling and flotation increased the overall antimony recovery to 90%. Most of the antimony content in the gravity-flotation tailing (0.13% Sb) was present as extremely fine grains of both antimony sulphides and oxides, partially free and partially attached to gangue minerals.

Smaller cuts over the table, cleaner flotation stages and removal of the stibnite-bearing pyrrhotite improved the grades of the rougher concentrate from 4.3% (tabling), 10.1% (flotation) and 11.5% (tabling and flotation), to 39.5, 49.6 and 41.4% Sb, respectively. Some 15 to 20% of the antimony reported in the middling products of the cleaning operation. However, most of this should be recovered in plant operation by recirculation of these products.

As the mineralogical studies indicated that the major antimony losses in the gravity-flotation treatment occurred in the extreme fines, the development of the flowsheet for treating the Quebec Antimony ore placed emphasis on careful grinding and classification to minimize the formation of slimes. The flowsheet (see Figure 2, page 36) also involved a two-stage gravity concentration, flotation and magnetic separation. Its trial on laboratory scale gave an overall antimony recovery of 77.5% at a grade of 45.5% Sb, with 11.2% of the antimony reporting in the intermediate products and 2.6% of the stibnite-bearing pyrrhotite (15.8% Sb). The final tailing contained less than 8% of the antimony,

nearly half of it being less than 25 microns.

As the stibnite in the pyrrhotite concentrate occurs largely as inclusions constituting the core of the pyrrhotite grains, its separation by physical means is practically impossible. Therefore, this product should be either sold as is (if marketable) or treated by pyro- and/or hydro-metallurgy for extracting its antimony content. The addition of the stibnite-pyrrhotite concentrate to the final antimony concentrate might unduly dilute its grade and increase its iron content, particularly if the stibnite-bearing pyrrhotite is more abundant in some of the ore zones.

Briefly, the use of tabling (two stages), flotation and magnetic separation as illustrated in the Proposed Flowsheet (Figure 3, page 40) is recommended for processing the ore, as received. If the deep ore proved to be free of antimony oxides, straight flotation should give adequate recovery. In this case, the use of sulphuric acid, instead of lead acetate as antimony activator for flotation, should not be overlooked as it gave comparable results with a greater rejection of gangue minerals.

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