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Ore Dressing Investigations on  
Grey Rock Ore

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## INTRODUCTION.

This report is presented in fulfilment of the requirements of Ore Dressing 3 at the University of British Columbia. All flotation tests and assays were made in the University laboratories.

The writers wish to acknowledge the assistance and suggestions of Mr. G. A. Gillies, Professor of Metallurgy and Mr. W. R. Smith, Assistant in Ore Dressing, under whom this work was carried on. Thanks are also due to Dr. H. V. Warren, Department of Geology, for his assistance in the polished section work.

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The University of British Columbia,  
April, 1944.

Ore Dressing Investigations on  
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OBJECT OF INVESTIGATION.

The object of this investigation was to determine a method of producing a commercial antimony concentrate.

LOCATION OF PROPERTY.

The property is located in the Lillooet Mining Division of British Columbia at the head of Truax Creek, and is held by the Grey Rock Mining Syndicate. The area ranges in elevation from 6000 - 8000 ft. and is on steep, precipitous, rocky ground above timber line.

SUMMARY OF RESULTS.

Saleable products can be obtained from the ore in two ways:

(1) By producing a bulk antimony concentrate containing 56 per cent antimony and impurities up to 1 per cent total of arsenic, copper and silver. (See Test 0)

(2) By making a copper - silver concentrate and a high grade ( 60 per cent) antimony concentrate containing only 0.5 per cent impurities.

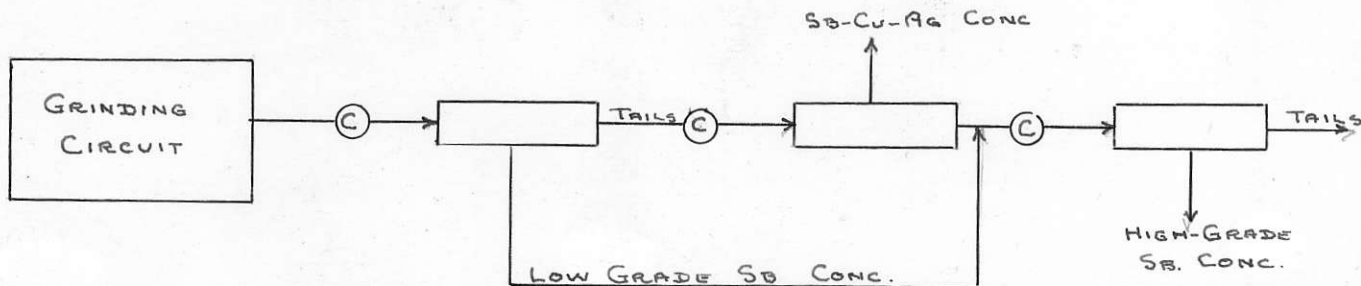
Summary of Conclusions and Recommendations

The best collector of stibnite was found to be Reagent #301 used at a low pH. At a high pH, the most selective collectors were found to be sulphonated Castor Oil and White Russian Mineral Oil but they pull a lower grade concentrate and only about half of the stibnite mineral.

In treating the ore on a commercial basis, the following procedure based on test 18c is recommended:

1. Grind the ore to 50 per cent -200 mesh .
2. Float a low-grade antimony concentrate at a pH of 8.5.
3. Float a silver-copper concentrate at a pH of 4.9.
4. Inject the low grade concentrate into the Ag-Cu tails and lower the pH to 2. Float a high grade antimony concentrate.

The following is a suggested flow sheet for the concentration.



Ⓒ = CONDITIONER

### CHARACTERISTICS of ORE.

Polished sections were made from eight representative samples of the ore and the following was observed:

1. The antimony occurs mainly as stibnite. A small amount of oxidized antimony (cervantite) was noticed, which could be expected to appear in the flotation tailings. This oxide should not be found at depth. The third source of antimony was tetrahedrite.
2. Copper occurs as tetrahedrite, covellite and chalcopyrite. The covellite is a supergene enrichment and should not be found in deeper mine workings. The amount of chalcopyrite is negligible but may be found in greater quantities at depth. Most of the coppers occur as tetrahedrite.
3. Silver is found in tetrahedrite and, to a lesser extent, in the stibnite.
4. Arsenic occurs in the stibnite; therefore, separation of the arsenic by flotation is not possible.
5. Iron occurs as pyrite and chalcopyrite.

### PREPARATION OF THE HEAD SAMPLE.

Representative samples were taken from the eight bags of ore received. These samples were crushed, cut and sampled by standard methods, and a representative sample



obtained for assaying. The results of the head assay were as follows:-

Sb	-	17.1%
Ag	-	9.0 oz/ton
Cu	-	0.16 %
As	-	0.11 %
Fe	-	0.7 %
S	-	7.5 %
Insol	-	68 - 71 %

#### GRINDING TESTS.

##### Object

To find the length of grind required for substantial unlocking.

##### Procedure

All grinding was done in a rod mill containing 23 rods,  $\frac{7}{8}$ " in. diameter and  $11\frac{1}{2}$ " long. The rod mill was 12" in diameter and 12" long and was rotated at a speed of 46 revolutions per min. Tests were made on charges of 1000 grams of ore and 1000 grams of water for 10, 15 and 20 minutes respectively. The products were dried and a 200 gram sample was screened for 20 minutes on the Ro - Tap. The ore used had previously been crushed to - 10 mesh.



Results.

Mesh.	10 min. grind %	15 min. grind %	20 min. grind %
+ 65	3.7	1.7	0.8
- 65 + 100	16.7	8.1	3.15
- 100 + 150	21.35	16.5	6.2
- 150 + 200	17.3	23.4	21.2
- 200	40.8	50.25	68.5
Total	99.85 %	99.95 %	99.85 %

Microscopic examination revealed that the 10 minute grind did not unlock all the mineral particles. The 15 minute grind did unlock all the minerals, so this length of grind was used through out the investigations

#### PROCEDURE of FLOTATION TESTS.

##### Grinding.

All tests were run on 1000 grams of ore. Grinding was done in the rod mill with 1000 grams of water for 15 minutes. Some reagents were added to the grind when deemed necessary and are shown in the individual tests.

##### Flotation.

The rod mill product was transferred to a Ruth Flotation Machine and made up to a 3.5 : 1 pulp dilution. The remaining reagents were added and the pulp conditioned for 5 minutes.

The machine was then reversed and the froth skimmed by hand. Skimming was continued until the froth became barren or dirty. The flotation products were then dried and weighed.

Assays.

All silver assays were run by the Løtharge Cupellation Method.

All antimony assays were run by the Permanganate - Oxidation Method.

Arsenic assays were run by a modified distillation method and copper assays by the colorimetric method.

See Appendix for detailed description of assays.

NOTES on TESTS.

Flotation Test No. 00

To test reagents used at Yellow Pine.

Reagents.

To cell:	0.08 #/ton	oresylic acid.
	0.05 #/ton	of Reagent #301
	1.0 #/ton	of Na OH

Notes.

The froth was too brittle, so 1 drop of frother 52 & 60 was added. This killed the froth and the test was a failure.

Flotation Test No. 0

To test flotation of stibnite in acid pH.

Reagents

To cell	0.02 #/ton	pine oil
	0.01 #/ton	reagent #301

Notes.

A very good stibnite concentrate was obtained.

Results.

Product.	Wt.	% Sb	Rec Sb.	Oz/ton Ag.	Rec. Ag.	% Cu	Re Cu
Conc.	304.9	56.0	97.4	23.0	74.6	0.4	76.
Tails	694.8	Tr	-	-	-	-	-

Stibnite floats easily at low pH but copper and silver minerals float with stibnite.

Flotation Test No. 1

Duplication of Test 0. The assays and recoveries for test 0 were checked.

Flotation Test No. 2

It was decided to run a series of tests at high pH to see if we couldn't hold down the copper and silver and take off a stibnite concentrate.

Reagents

To mill	0.6 #/ton Cu SO <sub>4</sub>
	2.0 #/ton Na OH
	0.1 #/ton Reagent #301
To cell	0.04 #/ton cresylic acid.
	10.0 #/ton Na OH

Notes.

A dirty concentrate was pulled but copper did not come off, so it was decided to regulate pH and float off stibnite until copper started to come off.

Flotation Test No. 3

This test was run at a pH of 9. The same reagents were used as in Test 2. No froth was obtained.

Flotation Test No. 4

To see if  $\text{Fe SO}_4$  would depress copper and silver and stibnite float at a low pH. The test was carried out at a PH of 4. A good stibnite concentrate was obtained but the copper and silver were with the stibnite. This would indicate that the use of  $\text{Fe SO}_4$  in an acid solution would not depress the copper and silver minerals.

Flotation Test No. 5.

Na CN was added to the mill to try and depress the copper and silver. The test was run at a pH of 11. A poor froth was obtained and no concentrate was taken off.

Flotation Test No. 6

$\text{Fe SO}_4$  was added to mill to see if it would not depress copper and silver in basic pH. The test was run at a pH of 8. Poor froth - test a failure. It was decided to add reagents to cell instead of mill.

Flotation Test No. 7

In this test it was decided to add  $\text{Na}_2 \text{S}$  to try to depress the copper and silver. The test was run at a pH of 10. A good froth was obtained but it quickly became barren. The pH was lowered to 2 and a good froth was obtained.

Flotation Test No. 8

Test 7 was repeated using less  $\text{Na}_2\text{S}$  and at a pH of 6. Cresylic acid was added to improve froth but it killed the froth.

Flotation Test No. 9

Due to the difficulty of obtaining a froth in basic pH, it was decided to try a different frother. Barrett No. 4 was used first in acid pH. The pH was then gradually raised by the addition of Na OH to the cell. As the pH was increased the froth and collecting power decreased. More Barrett No. 4 was added but no change took place.

Flotation Test No. 10.

It was desired to determine the effect of  $\text{Na}_2\text{S}$  variation and pH variation on the froth and collection. At a pH of 9 and 0.12 #/ton of  $\text{Na}_2\text{S}$ , the froth and collection was good. At a pH of 11 the froth was fairly good and collection not so good. Then 0.06 #/ton of  $\text{Na}_2\text{S}$  was added. This gave a fair froth but poor collection. Copper samples were taken at a pH of 9 and 11. The copper decreased as the pH was increased.

Flotation Test No. 11

It was observed in test No. 2 that the stibnite concentrate, although dirty, contained little copper. This observation was made by taking grab samples during the flotation test and assaying them colorimetrically. The possibility of producing a concentrate low in copper was therefore the object of Test No. 11. In this series of tests lime was added in increments of 5 #/ton and its effect upon copper content of the



concentrate was observed. In all these tests the lime was added to the ball mill along with 0.2 #/ton of Na CN which was added as a possible depressant of copper.

Reagents added to cell were pine oil (.04 #/ton) cresylic acid (.04 #/ton) and Reagent #301 (.03 #/ton).

The froth in every instance was fair but became quickly barren and none of the tests yielded more than 100 grams of concentrate. It was observed that none of these concentrates contained more than 0.25 per cent copper. This showed definitely that copper could be depressed at pH above 7.

#### Flotation Test No. 12

In this test an attempt was made to observe whether the copper was being floated early or late as compared to the stibnite. The pH was to be basic, 15 #/ton of lime being added and the sodium cyanide increased to 2.5 #/ton. The reagents added to the cell were the same as those in Test No. 11 and conditioning time was 5 minutes.

#### Results.

Concentrate #	Skimming time.	Weight.	% Sb.	Ag oz/ton	% Cu.
1	10	170	44.5	19.3	0.55
2	10	53.7	45.5	42.0	1.0
3	10	12.6	51.6	68.0	1.3
Tails	-	775	7.1	-	-

This test showed that there was a close relationship between the copper and silver in the ore. A briquette was made of concentrate #3 to determine the form in which the silver and copper were being recovered. It turned out to be tetrahedrite.

This test provided the first tangible proof that a separation of copper and silver from the stibnite was possible. The recovery of silver as a silver concentrate was 34.5 per cent. The 7.1 per cent Sb in the tails was unsatisfactory.

#### Flotation Test No. 13

An attempt was made to improve the antimony in the tails of Test No. 12. Barrett #4 was substituted for pine oil as a frother, otherwise the test was similar to test No. 12.

The froth and collection were poor and only 75 grams of concentrate were removed.

It was ~~was~~ apparent that Barrett #4 was not suitable as a frothing agent for stibnite. It seems to hinder the action of the collector.

#### Flotation Test No. 14

In this test the following reagents were added to the cell -

20 #/ton lime	0.1 #/ton Reagent #301.
0.6 #/ton Cu SO <sub>4</sub>	0.04 #/ton Cresylic acid.

The froth was poor and only 100 grams of concentrate were floated off. Subsequent small additions of reagent #301 progressively killed the froth.

#### Flotation Test No. 15

The object of this test was to observe qualitatively the effects on the stibnite of sulphonated castor oil and a



solution of mercurous chloride. Pine oil was used as a frother and a good froth was obtained in which stibnite was noticed.

One gram of starch was added as a disperser but had no effect on the quality of the froth. Ten c.c. of zirconium nitrate were added and everything was depressed.

No samples were taken.

#### Flotation Test No. 16.

The effects of sulphonated castor oil as a collector were observed at a pH of 6.7. Gelatin was added (0.5 #/ton) as a disperser and 0.02 #/ton of pine oil were added for frothing action.

Everything was depressed so the test was rejected.

#### Flotation Test No. 17

This test was commenced with the object of floating as much copper as possible while depressing the stibnite with mercurous chloride. Having collected this copper concentrate, a stibnite concentrate was to be obtained by lowering the pH until a good froth was obtained.

The reagents throughout this series of tests were to be -

0.0 g #/ton Sulphonated castor oil.

0.1 #/ton mercurous chloride

0.02 #/ton pine oil.

Lime or sulphuric acid was added in order to produce the required pH.

Flotation Test 17 (a).

In this test no lime was added. The pH was 7.2. The other reagents were added to the cell and conditioned for 10 minutes. The following results were obtained:

<u>Concentrate #.</u>	<u>Weight.</u>	<u>Skimming time.</u>	<u>Ag(oz/T)</u>	<u>%Sb</u>	<u>%Cu</u>
1	117.4	5	16	48.6	0.4
2	69.8	5	25.7	43.9	0.38

(3.5 #/ton of sulphuric acid was added and the pH was lowered to 2.5. Then 0.22 #/ton of reagent 301 and 0.02 #/ton of pine oil were added and pulp conditioned for 10 minutes. A good stibnite froth was obtained and the following results fit into the above table)-

3	132.7		21.9	50.2	0.4
Tails	652.6		2.6	3.2	-

No separation of copper and silver was accomplished by this test.

Flotation Test No. 17 (b)

As a previous test had shown that copper could be depressed at high pH, this test was planned to be carried on at a basic pH. The reagents were added as per schedule after 5 #/ton of lime had been added to the cell. The pH at this stage was 8.6.

After pulling 2 concentrates, each of which had been skimmed 5 minutes, sulphuric acid was gradually added with the object of bringing the pH down to about 2.0. However, when a pH of 4.9 was reached, 0.22 #/ton of reagent with 0.02 #/ton of pine oil were added with the object of examining the froth

at this pH. The froth was poor and upon close examination by panning, much pyrite was observed. ~~As~~ A concentrate was pulled until the pyrite became exhausted from the concentrate. Simultaneous with this depletion, the froth became barren. The pH was then lowered to 2.3 and 0.04 #/ton of pine oil were added. A fine stibnite concentrate was obtained.

A tabulation of the results follows:

<u>Concentrate #.</u>	<u>Weight.</u>	<u>Time.</u>	<u>Ag.</u>	<u>Cu.</u>	<u>Sb.</u>	<u>As.</u>
1	122.3	5	15.8	0.25	51.3	
2	22.7	5	13.2	0.18	34.8	
3	33.6	-	112.3	2.0	38.9	
4	136.0	-	14.0		57.8	0.35
Tails	670	-	2.0		2.8	

**Conclusions:**

(1) A briquette ~~of concentrate~~ of concentrate #3 was made and microscopic examination showed that the copper and silver were occurring as tetrahedrite.

(2) A copper recovery of 53.3 per cent and a silver recovery of 51.6 per cent were obtained in concentrates #3 and #4.

(3) The tetrahedrite was successfully depressed by the reagents at a pH of 8.6.

(4) A better grade of stibnite concentrate was obtained at a pH of 2.3 than was obtained at 8.6. The stibnite was almost completely depressed at a pH of 4.9.

Flotation Test No. 17 (c)

This test was a similar test to test 17 (a), except

that 10 #/ton of lime was added and a pH of 10.9 was maintained during the first stage. The froth was skimmed for 5 minutes and concentrate #1 was taken off. Sulphuric acid was added until the pH reached 2.4. Reagent #301 (.3 #/ton) and pine oil were added and the pulp was conditioned for 10 minutes.

Results:

<u>Concentrate #</u>	<u>Weight.</u>	<u>%Sb.</u>	<u>%Cu.</u>	<u>Ag.</u>
1	78.1	36.3	-	12.98
2	161.3	55.6	-	28.08
Tails	734.8	-	-	3.2

This test was not successful in separating the silver from the antimony.

Flotation Test No. 17 (d)

This test was similar to test 17 (c). Fifteen #/ton of lime were used and a pH of 12.4 was maintained during the first stage. The second stage was carried on at a pH of 2.1.

<u>Concentrate #.</u>	<u>Weight.</u>	<u>% Sb.</u>	<u>% Cu</u>	<u>Ag</u>
1	83.1	26.1	0.17	11.3
2	203.7	52.9	0.42	27.9
Tails	709.9	-	-	-

The results obtained in this test were very similar to those in 17 (c).

Flotation Test No. 17 (e)

This test was made under conditions similar to those in 17 (b). Instead of adding lime, the pH was immediately lowered to 4.9 to see if copper and silver could be removed directly from the stibnite without any previous flotation.



Heavy stibnite concentrate was obtained with little visible pyrite. This was unsatisfactory, so the pH was raised by lime in small steps until the froth became almost barren; the concentrate was panned. The concentrate was still mostly stibnite so the test was rejected. This test showed that silver and copper cannot be removed in a concentrate relatively free from stibnite by direct adjustment of pH to 4.9.

#### Flotation Test No. 17 (f)

This test was also made under conditions similar to those in 17 (b). This time the pH was raised to 11.5 and prescribed reagents were added and the pulp allowed to condition for 12 minutes to allow the lime to react. When the pH was lowered to 6.5 the froth turned black and a pan sample showed some pyrite. The pH was then lowered to 5.5 and the froth became brown and very brittle. Two concentrates were pulled under these conditions and copper assays showed them to be 0.36 AND 0.32 per cent. This test was rejected.

#### Flotation Test No. 17 (g)

This test was an attempted duplication of 17 (f) in which the reagent 301 and pine oil were to have been added, but when the pH reached 6.0 there was no change in the froth which contained too much stibnite. It was not a successful test. The pH was lowered to 2.1 and 53.6 per cent antimony concentrate was floated giving a recovery of 94.7 per cent.

Flotation Test No. 17 (h)

To prevent accidental activation of the stibnite by allowing it to have too low a pH at any one time, the lime was added to the grind, otherwise this test was carried on similarly to 17 (f). When the pH was 4.9 the stibnite was too strong to provide a good silver - copper concentrate so the test was rejected.

Flotation Test No. 17 (j)

This test was run under conditions duplicating those in 17 (b). Here the first concentrate was pulled at a pH of 10.2 and set aside. The second concentrate was pulled at a pH of 5.1 and saved, and concentrate #1 was dumped back into #2 tails. The pH was lowered to 2 and a stibnite concentrate floated.

The purpose of this test was to determine the overall recovery of antimony and silver.

## Results:

<u>Concentrate #.</u>	<u>Weight.</u>	<u>% Cu.</u>	<u>% Sb.</u>	<u>Ag.</u>	<u>As</u>
1	-	-	-	-	-
2	57.7	0.76	52.5	75.64	-
3	35.0	0.34	54.7	28.96	-
4	196	0.17	58.7	14.2	0.36
Tails	671	-	1.7		

The overall recovery of silver in concentrate #2 and #3 was 59.7 per cent and the recovery of antimony in concentrate #4 was 67.6 per cent.

Flotation Test No. 18.

This test was run to observe the effects of Russian Mineral Oil as a collector of stibnite using Arctic Syntex M as a frother at a high pH, preferably at 8.5, and to observe the effect of the addition of a small amount of pine oil to assist the frothing. This procedure has been successfully used in the flotation of molybdenite.

Test 18 (a) - Reagents added:

To grind: 5 #/ton lime.

To cell: 1 #/ton white Russian oil.

0.05 #/ton Syntex M.

Condition time 5 minutes at a pH of 9.2.

The froth was weak and barren so an additional 0.14 #/ton of Syntex M was added. This gave an improved froth structure but no collection was evidenced. Another 3.5 #/ton of Russian oil were added gradually and a very slight improvement in the froth was noticed.

The pH was dropped in small step by adding 10 per cent sulphuric acid until a pH of 2.1 was reached. Still very little collection was noticed.

Then, 0.025 #/ton of pine oil was added and stibnite was floated quite strongly. However, this did not persist and the froth soon became barren. Subsequent additions of the reagents used did not bring up any more stibnite.

The tails were very dirty and were rejected. An overall recovery of antimony of 43.5 per cent was obtained.

This test showed that, as a collector of stibnite, white Russian Oil is unsatisfactory when used with <sup>only</sup> Syntex "M"



as a frother. Pine oil improved this collection at a low pH but not to a sufficient degree to warrant the use of these reagents.

Test 18 (b). This test was commenced with the intention of observing the effect of the addition of 0.028 #/ton of pine oil while the pH was 8.5. All other conditions were similar to those in test 18 (a).

Reagents added to the mill:	4 #/ton of lime.
" " " cell:	1 #/ton Russian oil.
	0.028 #/ton Pine oil.
	0.025 #/ton Syntex M.

Conditioning time was 5 minutes at a pH of 8.6.

With the exception of the use of lime as a pH controller, the use of these reagents is analogous to that in a successful molybdenite flotation. It was discovered later that the flotation of molybdenite, the pH is controlled with Na OH.

A fairly brittle froth with moderate collection resulted. This froth became barren after about 75 grams of stibnite had been floated. The quantities of reagents other than lime were doubled, but only a small additional amount of stibnite was floated. This procedure seemed to be meeting with little success, although an attempt to separate copper and silver from the charge was made in a manner similar to Test 17 (j) and is outlined as Test 18 (c).

Test 18 (c). The concentrate from Test 18 (b) which had been floated at a pH of 8.5 was set aside, and the tails in the cell were lowered to a pH of 5.1 by 10 per cent sulphuric acid. The froth was still barren so 0.03 #/ton of reagent 301 was

added. The light pyritic froth that had been observed in Tests 17 (b) and 17 (j) was noticed and concentrate #1 was floated until the froth became barren.

Another 0.03 #/ton of reagent 301 was added along with 0.02 #/ton of pine oil. After conditioning for 5 minutes, concentrate #2 was floated. The pyritic froth persisted to a somewhat lesser extent than in concentrate #1.

Then the concentrate from Test 18 (b) was poured back into the cell and sulphuric acid was added until the pH was 2.0. An excellent stibnite froth resulted and concentrate #3 was floated. The tails appeared quite clean. The results of this test are as follows:

<u>Concentrate #.</u>	<u>Weight.</u>	<u>% Sb.</u>	<u>% Cu</u>	<u>Ag.</u>	<u>% As</u>
1	40.2	45.6	1.9	102.0	-
2	38.8	33.0	0.3	29.8	-
3	230.1	60.6	0.15	14.0	0.4
Tails	670	0.9	-	1.2	-

	<u>Rec Cu.</u>	<u>Rec Ag.</u>	<u>Rec. Sb.</u>	<u>Rec. As.</u>
1	47.7	45.4	10.1	} Over- all 96.6
2	<u>7.3</u>	<u>12.7</u>	7.1	
3	55.0 %	58.1 %	79.4	
				83.6

This test confirmed the assumption made previously during the microscopic work that the antimony and the bulk of the arsenic were inseparable by flotation methods. It also shows that the value of the products can be enhanced by the methods of separating copper and silver from antimony, as carried on in this test. The reagents for this test provided the best control

of the quality of the silver - copper concentrate. The test also confirmed the previous assumption that the presence of pyrite in the silver - copper concentrate is indicative of its quality.

### GENERAL CONCLUSIONS.

#### Collectors

Reagent #301 was the best collector of stibnite and of tetrahedrite and the selectivity is almost solely a function of the pH of the pulp. At a high pH, reagent #301 has little collecting power for stibnite and tetrahedrite. At a pH between 4.9 and 6.0 reagent #301 will selectively collect pyrite and tetrahedrite. As a collector of stibnite the efficiency of reagent #301 increases as the pH is lowered and the most successful results were obtained at a pH close to 2.0.

No commercial stibnite concentrate was obtained at a basic pH by any of the reagents used. Both sulphonated Castor Oil and White Russian Mineral Oil gave fairly selective stibnite flotation at a pH of 8.5 - 9.0 but the grade and recovery do not warrant their use solely as collectors of stibnite.

#### Frothers.

Of the frothers used, pine oil was the most successful under practically all conditions. Syntex M does not give a good froth when used with White Russian Mineral Oil unless it is reenforced with pine oil.

#### Recovery

### Recovery

The best recovery of antimony was 97.4 % in Test No. 0. It was a rough concentrate high in silver, arsenic, and copper. These impurities were nearly 1 per cent, which would subject the concentrate to smelter penalties, but on the other hand the grade was 56 per cent antimony which would be subject to smelter premiums.

Test 18 (c) showed that it was possible to produce a clean, high grade antimony concentrate containing only 0.55 % impurities and also a second product high in silver and high in copper. The recovery of antimony in the concentrate # 3 was 79.4 %, most of the loss occurring in the silver-copper concentrate. This antimony concentrate (grade 60.6 % Sb.) would be subject to smelter premiums and no penalties.

The overall recovery of antimony by this method of concentrating depends largely upon successful marketing of the silver - copper concentrate.

The Engineering and Mining Journal (March 1944) reports a process whereby an argentiferous tetrahedrite may be treated and the silver, copper and antimony recovered. Under proper conditions of temperature and concentration the ore may be decomposed by an alkaline sulphide solution, dissolving the antimony and leaving the silver and copper in the residue as insoluble sulphides. The pregnant solution is electrolyzed in banks of diaphragm cells, recovering the antimony on mild steel cathodes. A more complete discussion of the chemistry of the process is given in U.S. patent No. 2,331,395 assigned to the Sunshine Mining Company.

If such a treatment is possible for the silver-copper concentrate, which is comparable to that used by the Sunshine Mining Company, the recovery of antimony will be increased by 17.2 per cent, giving an overall recovery of 96.6 per cent antimony, 55.0 per cent copper, and 58.1 per cent silver.

No attempts were made to clean any of the concentrates as time did not permit.



APPENDIX.A. Assay Procedures.

Antimony. The sample containing not more than 200 mg of Sb was placed in a copper flask and fused to a clear melt with sulphuric acid and potassium bisulphate. The ore was reduced with tartaric acid crystals to the antimonous state.

The melt, when cool, was taken up in 10 c.c. of hydrochloric acid and 100 c.c. of water. The solution was saturated with  $H_2 S$  gas until all the antimony was precipitated. The sulphides were filtered out and washed with  $H_2 S$  water.

The antimonous sulphide was dissolved in hot 5 per cent Na OH and the solution washed through the filter paper. Copper, lead and manganese are left insoluble.

The solution of  $Sb_2 S_3$  in Na OH is treated with potassium bisulphate and sulphuric acid and fused again. The melt is taken up with 75 c.c. water and 10 c.c. HCl and bailed to expell sulphur dioxide.

The solution is then diluted to 150 c.c., 15 c.c. of hydrochloric acid added and titrated to a permanent pink tinge with standard potassium permanganate.

SilverAg on Antimony concentrates.

The fusion charge was made up as follows:-

Conc.	-	$\frac{1}{4}$ A.T.
Litharge		60 gr.
Soda Ash		30 gr.

Nitre - 1.5 gr.  
Cover

The charge was fused 20 minutes and poured. The resulting lead button was cupelled and the silver button weighed.

Ag on Tailings.

The fusion charge was made up as follows:-

Tailings -  $\frac{1}{2}$  A.T.  
Litharge - 60 gr.  
Soda Ash - 30 gr.  
Nitre - -  
Cover

The charge was fused 20 minutes and poured and the resulting lead button cupelled and the silver button weighed.

Arsenic

Modified Distillation Method.

The apparatus consists of two 100 c.c. Erlenmeyer flasks. One flask is used for decomposition of the ore and distillation of the product and the other for absorption of the volatilized  $\text{Ag Cl}_3$ . The flasks are connected by an air condenser made of  $\frac{3}{8}$ " pyrex tubing. The tube rises about 4 or 5 inches above the flask and is then bent for a length of about 5" on a slope to a second bend which dips down into the absorption flask. The ascending portion of the tube is made longer than usual in order to create a fractioning effect on traces of  $\text{Sb Cl}_3$  which tend to volatilize together with the  $\text{Ag Cl}_3$ ,  $\text{H Cl}$ ,  $\text{Ag Cl}_3$  and steam are distilled directly into a solution



of sodium bicarbonate containing considerable excess. In this manner, excessive heat during neutralization is avoided and the contents of the receiver are ready for titration N/100 iodine soon after distillation is complete.

#### Procedure.

- (1) 200 mg of sample are weighed into first Erlenmeyer.
- (2) A&S 7 c.c. of 10 N  $\text{HNO}_3$  and 5 c.c. 12 N  $\text{H}_2\text{SO}_4$  and heat until decomposition is complete (25 - 30 minutes)
- (3) Cool, add 5 c.c.  $\text{H}_2\text{O}$  and to cooled liquid add 1 gr of hydrozine chloride, 1 gr K Br and 10 c.c. H Cl.
- (4) Wash the neck of Erlenmeyer and place the condenser tube in the mouth of the flask with the other end dipping into the other Erlenmeyer in which has been placed 12 gr of sodium bicarbonate dissolved in 50 c.c. water.
- (5) The distillation flask is heated until the original volume is reduced by one half.
- (6) The tube is removed, the flask containing the sodium bicarbonate and volatilized  $\text{AsCl}_3$  is cooled and then titrated with N/100 iodine solution to a permanent blue colour. 5 c.c. of starch solution are added as an indicator.

#### Copper.

##### Colorimetric Method.

- (1) Weigh out a 1 gr sample and place in a copper flask.
- (2) A&S 10 c c  $\text{HNO}_3$  and boil until brown fumes disappear.
- (3) Add 7 c.c. H Cl and boil until digestion is complete.
- (4) Add 90 c.c.  $\text{H}_2\text{O}$  and then 15 c.c.  $\text{NH}_4\text{OH}$ .
- (5) Bring just to a boil.

- (6) Cool thoroughly.
- (7) Filter and compare to copper standards.





