

FLOTATION OF PUERTO RICO
CHALCOPYRITE

by

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A Thesis Submitted to the Faculty of the
DEPARTMENT OF METALLURGICAL ENGINEERING
In Partial Fulfillment of the Requirements
For the Degree of
MASTER OF SCIENCE
In the Graduate College
THE UNIVERSITY OF ARIZONA

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ACKNOWLEDGMENTS

I wish to express my gratitude to those who have helped in their many ways to make this thesis possible. Especial credit is due to:

The Puerto Rico Mining Commission for underwriting my studies at The University of Arizona and for providing manuscript typing service.

George Roosveare for his technical guidance in the various laboratories and for his help in proofreading the final draft of the thesis manuscript.

Fausto Muñoz for assistance in developing the format of this thesis.

Professors Morris and Smith for their patience and assistance, both as instructors and as manuscript proof-readers. Also to Professor Morris as my advisor.

Fernando L. Benitez for his efforts in preparing the final format draft of the thesis.

My wife, Wilma Nydia Torres (de Perez), who provided the assistance and confidence which made this thesis possible.

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ABSTRACT

This work deals with the technique known as froth flotation of minerals applied to low grade ores, less than 0.4 per cent copper.

Several different tests were conducted with the low grade copper ores of the Cala Abajo deposit at Utuado, Puerto Rico, to determine the most suitable particle size required in the process, pH, time of grinding and conditioning, and flotation. Type and quantity of reagent required to minimize the copper present in the tailings in order to obtain a concentrate of grades between 25 to 30 per cent copper were also established. The most suitable or best condition were determined and the results are discussed.

CHAPTER 1

INTRODUCTION

Flotation is a well known process used in the mining industry for upgrading of low grade ores. The basic difference in the use of this process depends on the particular type of ore to be treated for extraction. The type of ore determines the grinding operation, the conditioning reagents to be used, the number of flotation cells step, and finally the time required to obtain the upgrade concentrate (1, 2, 3).

1.1 Characteristics of Flotation Process

In the treatment of ores by flotation, it is first necessary to grind the ore to fine particles, until the desired locked particles are detached from each other. The pulp, a mixture of solid and liquid, is mixed with certain chemical compounds by agitation, and transferred to flotation machines. In the flotation machines, air is introduced by the impellers and dispersed throughout the pulp in the form of fine bubbles. The particles of the desired mineral to be recovered become attached to the air bubbles and rise to the surface of the pulp as a mineral froth. This is skimmed off and collected as the concentrate. This concentrate may be upgraded by refloating, in order to provide a

final concentrate of satisfactory purity for commercial usage. The gangue minerals in the rougher flotation stay behind as the tailing products.

Often it is better to float the gangue minerals and recover the valuable minerals from the tailing product.

Due to the fact that high grade ores are scarcer than low grade ores, the flotation process constitutes an economically feasible method to recover rich concentrates from the low-grade ores. Flotation is presently the principal process for the concentration of sulfide ores such as the concentration of chalcopyrite, sphalerite, galena, etc. (4).

1.2 Flotation Reagents

In the treatment of ore material using the flotation technique, one of the most important things to be considered is the chemical reagents to be used (5, 6). The use of these chemical reagents is to make possible or facilitate the flotation treatment by modifying the surface properties of the mineral surfaces. The function of the reagents is to produce hydrophobicity on the mineral surface, thus providing for the mineral to be floated from the pulp (1, 2, 3). There are many different products on the market whose chemical properties make them suitable to be used as flotation reagents (7, 8). According to its function these reagents are generally divided into three groups: frothers,

collectors, and modifiers. A brief description of each group is presented below.

1.2.1 Frothers

The function of frothers is to produce bubbles of such stability and peculiarities that could make possible the attachment of mineral particles in their interface. Frothers are heteropolar organic compounds (nonpolar hydrocarbon groups attached to polar groups). The nonpolar part is a water-repellent or hydrophobic, and the polar part is a water-like or hydrophilic. Frothers are characterized by being strongly adsorbed at the air-liquid interface. Therefore, this chemical tends to lower the water surface tension in the pulp. The froth produced should be strong enough to support the weight of the mineral which is attached to it, and permit separation from the pulp. Finally, the amount of frother used must produce sufficient and adequate froth for the operation. This is achieved by proper addition of the dosage required. Common frothers used are: pine oil, aliphatic alcohols, and cresylic acids. The generally used alcohols are those composed of five to eight carbon atoms such as mixed amyl alcohols, methyl isobutyl carbinol, certain heptanol, and octanols.

1.2.2 Collector

In the case of the collector, its function is to produce a hydrophobic mineral surface in order to make the

mineral float. As the frother, the collector has polar and nonpolar groups. The nonpolar part of the collector is oriented away from the mineral and provides the water repellency. The nonpolar part is absorbed on, or reacts with the mineral surface. The nonpolar part of the collector has an affinity for a mineral or class of minerals. Some types of collectors are: fatty acids, xanthates, dithiophosphate, sulfonated oils, and sulfonated fatty acids. For the flotation of sulfide and precious metal ores, the xanthates and dithiophosphate are commonly used.

1.2.3 Modifier

The modifier reagent has several different functions, which depend on their use. For this reason, the modifying agents may be classified according to their functions in one of the following groups: pH regulators, depressant, activator, sulfidizer, and dispersant.

In the case of pH regulators, sometimes it is desirable to carry out the flotation on the alkaline or acid side because it may change the relationship between the concentration of collector species and the concentration of heavy metal ions in solution. Wark and Cox (8) investigated the influence of these pH regulators in the flotation of sulfide minerals with xanthates and other sulfhydryl collectors. They found a critical pH value below which the

mineral will not float. This critical pH value is going to depend on the mineral, the collector, collector concentration, and the temperature. Some of the agents used are lime, soda ash, and sulfuric acid. Other types of modifiers are the depressants; their function is to suppress the flotation of one mineral species while floating another. Some of the depressants are: lime, sodium cyanide, sodium sulfide, and sodium silicate. The first two prevent the flotation of pyrite. In the case of sodium cyanide it is a depressant for most sulfide minerals to a less extent. An activator is used to reactivate or increase its susceptibility to flotation, if a mineral has been depressed, or is not normally floatable.

In the case of the gangue material, it is possible to have some material that tends to flocculate or hold together to the extent that it can interfere with the flotation of the desired mineral. Thus it becomes necessary to apply deflocculators or dispersants.

It is impossible to predetermine the exact amount of flotation reagents to be used on the basis of pure theoretical reasoning. This determination is on experimental determinations; the amount of flotation reagents to be used varies according to the ore to be beneficiated.

1.2.4 Other Parameters

Other aspects that must be considered in the flotation process are the particle size and the conditioning of the pulp. With respect to the ore particle size, the minerals desired to be liberated are found imbedded in the rock. To liberate the desired mineral particles it is necessary to crush and grind the ores. It is necessary to keep strict control of this operation because undergrinding can cause a loss of mineral in the tailing since the minerals would not be completely liberated. Therefore, the correct grinding size must be determined by laboratory experiments. Nevertheless too fine grinding is not advisable because the rate of flotation could be drastically reduced as the particle size decreases below 135 mesh (9). Conditioning may be defined as a process of coating or mixing the ore pulp with the reagents to create the necessary conditions required prior to the actual flotation step. The minerals in some ores may not respond in the normal way to the conditioning treatment, unless the interfering slimes and soluble salts have been removed. The advantages of desliming are reduction in reagent consumption, time of conditioning, number of flotation cells, and the improvement in metallurgical grade of the concentrate (1, 2, 3).

In the conditioning step, several variables should be taken into consideration, such as percentage solids in the pulp, time of treatment, temperature, intensity of

treatment, aeration, and the addition order of the flotation reagents.

Time of conditioning is important, since a short period may not yield the desired result and a long time will increase the reagents and power consumed (3). The time required is related to pulp density, temperature, intensity of agitation, and speed of reaction of the reagent with the mineral surface.

In the particular case of the flotation of copper sulfide ores, such as chalcopyrite, it is treated differently in each particular deposit due to the form of their association with other minerals, the grade, the occurrence in the deposit either disseminated or locked in the coarse, and finally other variables that are necessary to be considered. Therefore, the treatment of each ore is a different problem from deposit to deposit.

1.3 Previous Works

The existence of some low grade copper deposits in the central part of Puerto Rico (Utua-do-Adjuntas-Lares) attracted American Metal Climax, Inc. (AMAX) to perform studies of this area. AMAX sent ore samples of the Cala Abajo and Piedras Hueca deposit to Colorado School of Mines Research Foundation in order to conduct moderate amounts of laboratory test work to investigate possible applications of the flotation process for this material. The ore sample

treated by the Foundation had a grade that varied from about 0.2 to about 2.3 per cent for total copper and an oxide content that varied from about 0.04 to about 0.28 per cent. Three principal zones of the Cala Abajo ore were identified (details of these zones are discussed in the next chapter) as the "primary zone," the "transition zone," and the secondary "enrichment zone" (10).

The transition and secondary enrichment zones were initially treated differently with respect to the primary zone, due to the sticky clayey nature of their constituents. In these two zones the ore samples were initially treated for sand-slime separation. Each product of the separation was then floated separately. In the case of the primary zone, it was processed in a conventional manner without any prior sand-slime separation because this zone had no slime constituents. The average copper recovery from these zones was 90 per cent. Included in Appendix A are two flowsheets used for Piedra Hueca and Cala Abajo.

1.4 Goal and Scope of the Research

The Mining Commission, a governmental agency of the Commonwealth of Puerto Rico, started negotiations in 1965 for the extraction and development of copper deposits previously prospected by Ponce Mining (a subsidiary of AMAX) and Bear Creek Mining Company (a subsidiary of Kennecott Copper Corporation).

The mining companies based their technology of ore beneficiation on a cut-off grade of 0.4 per cent copper. Therefore, the technological conditions involved in the recovery of this type of low grade minerals must be established. Based on this assumption, the purpose of this investigation was to determine some of these conditions.

The ore samples used in this investigation were obtained from cores drilled by the mining companies at the Cala Abajo deposit. Since the remittance of ore samples by the Puerto Rico Mining Commission to the laboratories of The University of Arizona were only representatives of grade ores of less than 0.4 per cent copper, the investigation only applied to the low grade ore of less than 0.4 per cent copper.

The main objective of this thesis is to determine the most suitable treatment of low grade ore of less than 0.4 per cent copper. To this end, this investigation will try to minimize the copper present in the tailings following a suitable technology for obtaining a concentrate in the range of commercial usage.

CHAPTER 2

MATERIAL AND EXPERIMENTAL PROCEDURE FOR THE PRELIMINARY TEST

The description of the material, its location and characteristic as well as the methods employed in this investigation and data in copper recovery and tailing is presented below.

2.1 Location and Description of the Material

The material investigated in this thesis was obtained from the deposit designated as Cala Abajo, which is located in the central part of Puerto Rico (Utuado-Lares-Adjuntas) (see Figures 1 and 2).

Three principal zones of the Cala Abajo ore identified by the American Metal Climax, Inc. are: primary, transition, and secondary enrichment zones. The upper part of the ore is highly altered by hydrothermal action and weathering, and the ore is overlain by a blanket of barren, leached material averaging 30 meters in depth. Beneath this zone, lies a zone of secondary enrichment which ranges in thickness from 5 to 30 meters. Underlying this zone is a transition zone which varies greatly in thickness up to a maximum of about 100 meters. This zone grades into the primary zone at varying depths. The ore in the secondary

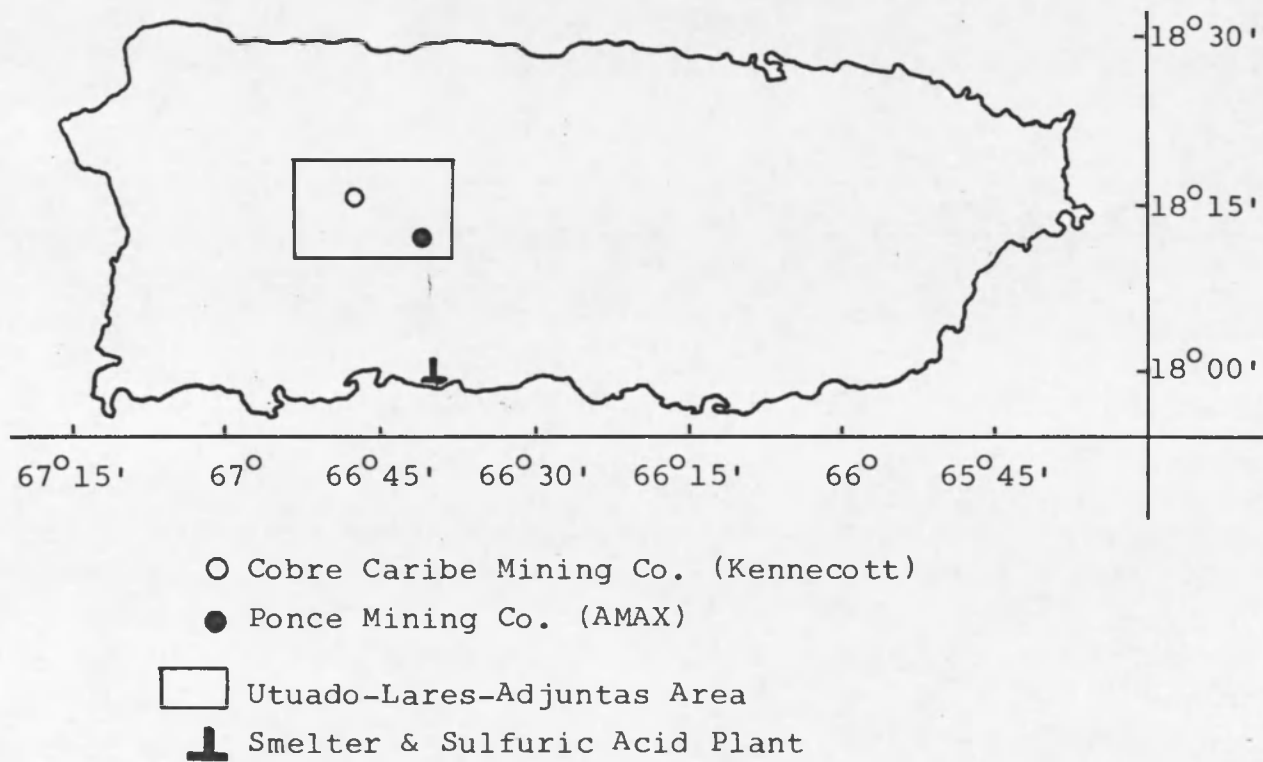


Figure 1. Open Pit, Milling Facility, Smelter, and Sulfuric Acid Plant Sites

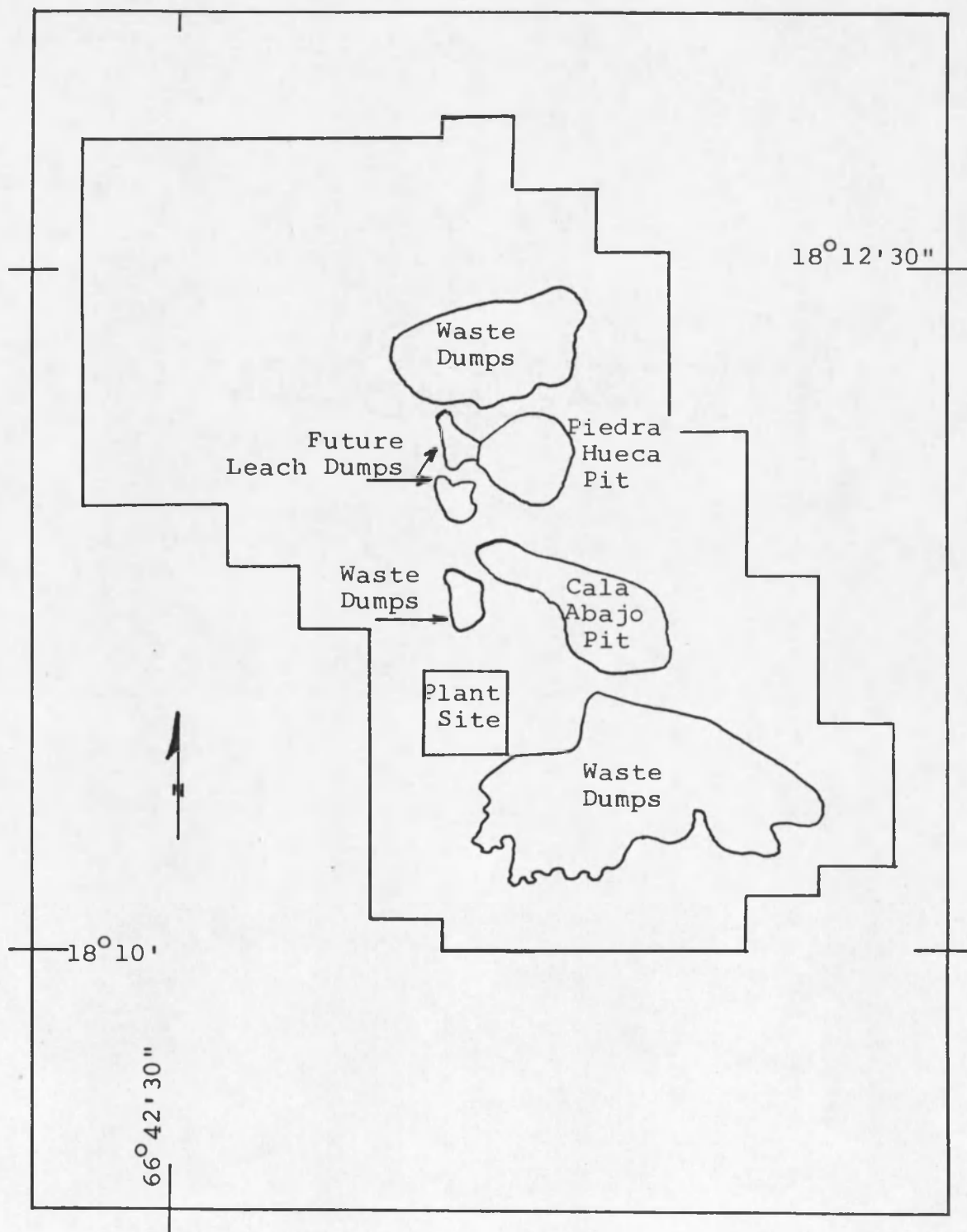


Figure 2. Mining Site, American Metal Climax Project in Puerto Rico

enriched and transition zones is composed of a poorly consolidated, sandy, clayey mass of material containing various copper minerals. Toward the bottom of the transitional zone, the material gradually becomes harder with sections of rock appearing, and grades into solid rock of the primary zone underlying it (for more detail see Figure 3).

The deposit of Cala Abajo is composed principally of chalcopyrite, covellite, chalcocite, talc, feldspar, quartz, and altered silicate minerals. The chalcopyrite is found free and incrustated in the rock. The average ore grade in this deposit is 0.819 per cent copper as determined by the American Metal Climax Inc. This deposit was divided into 37 sections, spaced at 50 meters in the majority of cases. From these sections, the Puerto Rico Mining Commission sent 29 samples representing 29 sections. Each one of these samples is composed of different depth levels including samples from the surface.

2.2 Analytical Methods Used in the Present Investigation

The atomic absorption technique was used as a tool to determine the copper content of the ore samples (11).

The procedure used in this technique is based on the vaporization of the sample and the dissociation of the constituents of the sample by the heat of a flame and

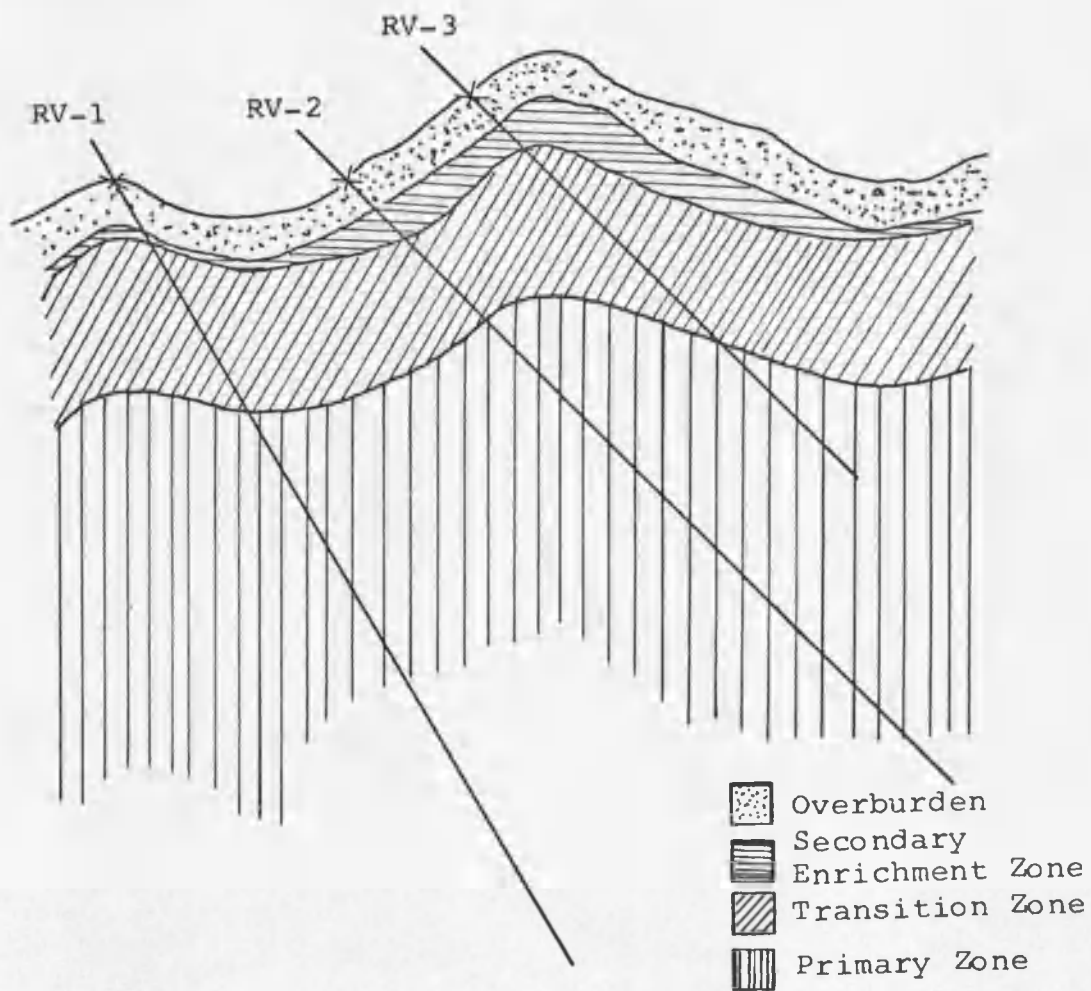


Figure 3. Schematic Diagram of the Cala Abajo Deposit Zones

depends that metal atoms absorb strongly at discrete characteristic lines of the particular metal.

The discrete wavelength is produced by the so-called Hollow Cathode lamp that precedes the atomizer; it emits the spectrum of the metal used to make the cathode. This beam traverses the flame and is focused on the entrance slit of a monochromator which is set to read the intensity of the chosen spectral line. Light with this wavelength is absorbed by the metal in the flame and the degree of absorption is a function of the concentration of the metal with the sample. Measurements are compared with a standard calibration curve and results calculated as ppm and transformed to percentages.

Another technique used was the iodide (12, 13) method and the procedure for this method is included in Appendix B.

The analysis of each sample which was sent from Puerto Rico, as well as the analysis of the composite sample made from the mixture of the 29 samples, are presented in Table 1. The composite sample gave an average of 0.255 per cent. This value compares favorably with the per cent obtained as an average of results obtained from the 29 samples: 0.259 ± 0.011 .

The ore samples also included some amount of oxidized copper material, which was separated by leaching the ore sample with 5 per cent sulfuric acid, saturated with SO_2 for

Table 1. Analysis of Copper Ore Sample from Cala Abajo
Utuaado, Puerto Rico

No.	Section	Per Cent Copper
1	0N	0.245
2	50N	0.285
3	50S	0.255
4	200S	0.290
5	300N	0.260
6	250N	0.175
7	100N	0.100
8	150N	0.125
9	150N	0.490
10	200S	0.370
11	300S	0.215
12	350S	0.235
13	100S	0.175
14	250S	0.530
15	1150S	0.245
16	1200S	0.2075
17	850S	0.410
18	1300S	0.145
19	2000S	0.145
20	950S	0.295
21	1100S	0.325
22	800S	0.325
23	900S	0.265
24	550S	0.255
25	400S	0.355
26	450S	0.115
27	650S	0.245
28	500S	0.095
29	725S	0.280

Other data:

Dilution: 1/2:250

Per cent copper (average) = 0.2597

Per cent standard deviation = \pm 0.011

<u>Composite sample (Head)</u>	<u>% Cu</u>
Atomic absorption spectrophotometer	0.255
Iodide method (this method was used only to verify the data obtained using the atomic absorption)	0.252
Copper oxide	0.028

a period of one hour (14). The mixture was located in a glass jar, covered with a rubber stopper. The sample was filtered and the liquid fraction was submitted to analysis by the atomic absorption. A result of 0.028 per cent of oxide copper was obtained.

2.3 Preparation of the Copper Ore Sample for Flotation

The samples were prepared for flotation following the procedure (15) described below.

2.3.1 Grinding and Mixing

After finishing the assays of the sections, the sample of each section was reduced to minus 10-mesh on a jaw crusher; all these sections were mixed until a composite sample of the material received was obtained. This sample was divided in groups of 600 grams each.

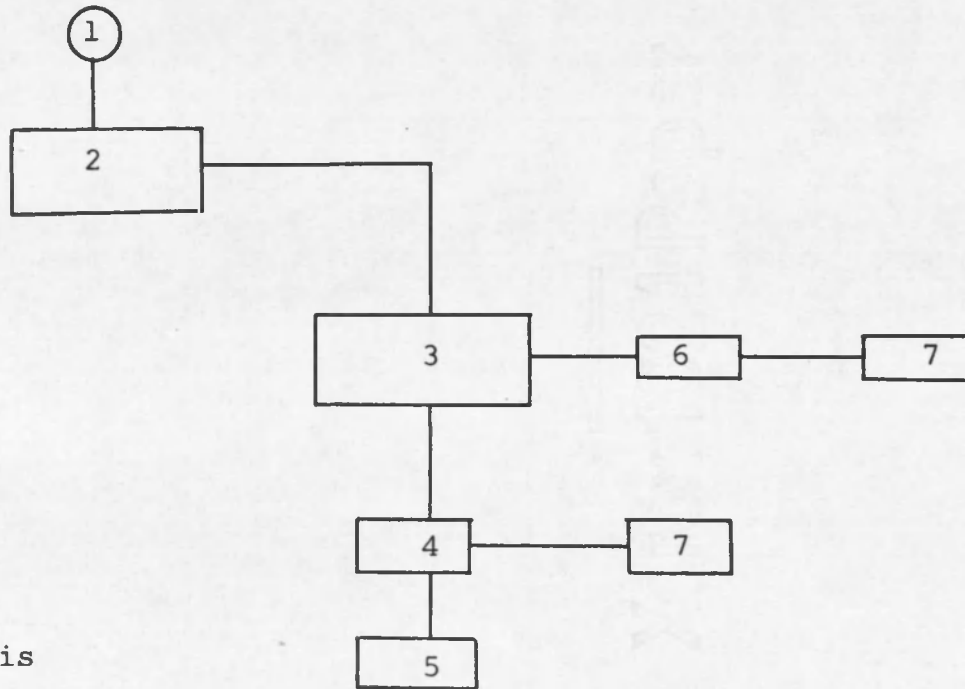
2.3.2 Conditioning of the Samples for Flotation

Six hundred grams of the composite sample were introduced in a ball mill that had a diameter of 8 inches and a critical velocity of 162.5 rpm and an actual operation speed of 110 rpm. To this material was added 400 grams of water, the collector, and calcium hydroxide to condition the material. The sample was ground for six minutes in a ball mill. The product obtained from the ball mill was transferred to a flotation cell (Fagergreen Laboratory Test Machine, serial No. 173 ST 115), and diluted with water to

20 per cent solids. After this, the material was conditioned again with collector and frother for a period of two minutes, adding at the same time sufficient calcium hydroxide until the medium ore was made alkaline. The material was floated for six minutes. The pulp in the cell was conditioned for one minute and floated again for six minutes, except in Test 1 this last part was not performed. The corresponding flowsheet is presented in Figure 4.

2.3.3 Other Studies

Screen analyses of the tailing were also carried out and are described in the corresponding chapter. Analyses of the copper content of concentrates and tailings were performed by the atomic absorption method to study the yield of the process.



Legend:

- 1--Feed
- 2--Ball mill
- 3--Flotation
- 4--Tailing
- 5--Screen Analysis
- 6--Concentrate
- 7--Chemical Analysis

Figure 4. Flowsheet for the Preliminary Experiment Test

CHAPTER 3

PRELIMINARY TESTS

A series of preliminary tests were performed to obtain the necessary data for the design of the treatment for the material received from Puerto Rico in order to obtain a tailing with a low copper content and at the same time that could be exploited commercially.

The variables considered were type and quantities of reagents, pH, time of flotation, grinding, and conditioning. The reagents used in these tests are used commercially in similar processes. A total of 5 tests were performed.

3.1 Test 1

Six hundred grams of the composite sample were introduced into a ball mill with 400 grams of water, 10 pounds of calcium hydroxide, and 0.05 pounds of sodium secondary Butyl Xanthates (collector) per ton of ore. The ball mill ran for six minutes. The product from the ball mill was transferred to a flotation cell, and sufficient water was added to make 20 per cent solids. The material was conditioned with 0.06 pounds of methyl isobutyl carbinol per ton of ore for about 1 minute. Finally, the material was floated for about six minutes at an initial pH of 11.1.

The froth obtained was excellent and the final pH was 10.7. The results obtained are shown in Table 2.

Table 2. Results of Test 1

Product	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
Head	100.0	0.255	---
Concentrate	6.8	2.6	72.5
Tailing	93.2	0.072	27.5

It can be observed from this table that the concentrate assays 2.6 per cent copper and contains 72.5 per cent of the total copper. The tailing assayed had 0.072 per cent copper and contained 27.5 per cent of the total copper. The concentrate was essentially a mixture of copper and iron sulfides.

3.2 Test 2

This test followed the same procedure as the first test, except that the ball mill was conditioned with 0.05 pounds of potassium amyl xanthate instead of the 0.05 pounds of sodium secondary butyl xanthate per ton of ore. The material was floated for six minutes having an initial pH

of 11. The same frother was used as in test 1. The concentrate obtained in this part of test 2 will be called concentrate 1.

By means of microscopic analysis of the tailing of test 1, it was found that the tailing contained free particles of chalcopyrite which had not been floated. Thus, the residue in the flotation cell from test 2 was conditioned with 0.03 pounds of potassium amyl xanthate and 0.066 pounds of methyl isobutyl carbinol per ton of ore for about 2 minutes, and proceeding to float the material for six minutes more. The initial pH for this flotation was 10.85. The concentrate of this second part of test 2 will be called concentrate 2 (see Table 3).

Table 3. Results of Test 2

Product	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
Head	100.00	0.255	--
Concentrate 1	7.51	2.300	71.72
Concentrate 2	3.04	0.310	3.78
Tailing	89.45	0.066	24.50

From this table it can be noticed that the distribution of copper is 71.72 per cent of the total copper concentrate 1, and assays 2.3 per cent of copper. For the second concentrate, the distribution is 3.78 per cent of the total copper with a content of 0.31 per cent copper.

If this result is compared with the one obtained on test 1, it can be noted that the per cent of copper in the tailing has diminished. The presence of iron sulfide in this test increased in the second concentrate perhaps due to the slight variation of pH.

3.3 Test 3

This test followed the same procedure as the test before, except that the ball mill was conditioned with 0.05 pounds of sodium di-secondary butyl dithiophosphate per ton of ore instead of 0.05 pounds of potassium amyl xanthates per ton of ore. The initial pH for the flotation was 11.1.

In the conditioning of the residue in the cell, 0.03 pounds of potassium amyl xanthates and 0.066 pounds of methyl isobutyl carbinol per ton of ore were used. The initial pH for this flotation was 10.9 and the time of flotation was 6 minutes.

Results are tabulated in Table 4.

The per cent of distribution of copper for concentrate 1 in this test was 64.0 per cent with percentage of copper of 3.75; the second concentrate of this test obtained

Table 4. Results of Test 3

Product	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
Head	100.00	0.255	--
Concentrate 1	4.41	3.750	64.00
Concentrate 2	4.10	0.350	5.59
Tailing	91.49	0.084	29.71

5.59 per cent of distribution with a percentage of 0.35 of copper.

It can be noticed that the per cent of copper in the tailing increased in comparison to the other tailings. Concentrate 1 had an increase in the per cent of copper, but the per cent of distribution decreased at the same time.

3.4 Test 4

This test followed the same procedure as before, except that the ball mill ore was conditioned with 0.02 pounds of potassium amyl xanthate per ton of ore. Before undertaking the flotation for concentrate 1, the contents of the ball mill were conditioned with 0.05 pounds of aeropromoter 3501 and 0.044 pounds of methyl isobutyl carbinol per ton of ore for about 1 minute. The acropromoter 3501 has the property of being collector and froth-forming (6); for

this reason the quantity of methyl isobutyl carbinol used on this test was reduced in comparison with the ores used before. The flotation proceeded with an initial pH of 11 for about 6 minutes.

The rough tailing obtained was conditioned with 0.01 pounds of potassium amyl xanthate and 0.022 pounds of methyl isobutyl carbinol per ton of ore and was floated at an initial pH of 10.8 during six minutes.

The results obtained are shown in Table 5.

Table 5. Results of Test 4

Product	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
Head	100.00	0.255	--
Concentrate 1	8.00	2.280	71.53
Concentrate 2	1.20	0.500	2.82
Tailing	90.80	0.070	25.65

This table shows a distribution of 71.53 per cent in the first concentrate and 2.82 per cent in the second, with 2.28 and 0.60 per cent of copper, respectively.

We can notice the results obtained in the concentrate increased with respect to the first two concentrates

obtained in tests 1 and 2, but diminished in comparison with the concentrate in test 3.

The per cent of copper in the tailing diminished with respect to test 3, but did not vary considerably with the other two tests. The progress to date has been little, but we conclude that the per cent of copper in the tailing will be about 0.06 to 0.07 per cent.

3.5 Test 5

The procedure used in this test was identical to the last one, except that in the ball mill the material was conditioned with 0.01 pound of potassium amyl xanthate per ton of ore. The product of the ball mill was conditioned with 0.05 pounds of aeropromoter 3477 and 0.022 pounds of methyl isobutyl carbinol per ton of ore for about one minute. The flotation took place with an initial pH of 11, for six minutes.

The rough tailing obtained was conditioned with 0.01 pounds of potassium amyl xanthate and 0.022 pounds of methyl isobutyl carbinol per ton of ore for one minute. This flotation ran for six minutes at initial pH of 10.8. The results are shown in Table 6.

The per cent of distribution of copper in concentrates 1 and 2 was 68.6 and 7.06 with a per cent of copper of 4.7 and 0.32, respectively. The per cent of copper in

Table 6. Results of Test 5

Product	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
Head	100.00	0.255	--
Concentrate 1	3.70	4.700	68.60
Concentrate 2	5.60	0.320	7.06
Tailing	90.70	0.068	24.34

this test was high comparable with the other test, also the concentrate was clean.

It can be noticed the per cent of copper in the tailing was kept approximately steady which makes us choose this test to continue the final tests where we will try to obtain a concentrate of 25 per cent copper, which would be considered of commercial usage.

3.6 Discussion of the Results

In Table 7 a resume of the experimental reagent, as well as data in percentage by weight, concentration, and distribution obtained from the preliminary flotation test are presented. This shows us that some discrepancy exists between the five tests performed, probably due to the selectivity of each reagent. From this observation it can be concluded that tests 2 and 4 show no selectivity, the

Table 7. Resume of Data Obtained from the Preliminary Experiments

Test	Reagents	Quantity (lbs/Ton. Ore)	% of Weight	% of Copper	Distribution of Copper (%)
1	Sodium Secondary Butyl Xanthates (G)	0.05	Concentrate #1 (6.8)	Concentrate #1 (2.6)	Total Concentrate (75.5)
			Tailing (93.2)	Tailing (0.072)	Tailing (27.5)
	Methyl Isobatyl Carbinol (F)	0.066	Concentrate #2 ()	Concentrate #2 ()	
2	Potassium Amyl Xanthates (C)	0.05	Concentrate #1 (7.51)	Concentrate #1 (2.3)	Total Concentrate (75.5)
			Tailing (89.45)	Tailing (0.060)	Tailing (24.50)
	Methyl Isobatyl Carbinol (F)	0.066	Concentrate #2 (3.04)	Concentrate #2 (0.31)	
	Potassium Amyl Xanthates (C)	0.03			
	Methyl Isobatyl Carbinol (F)	0.066			

Table 7.--Continued Resume of Data Obtained from the Preliminary Experiments

Test	Reagents	Quantity (lbs/Ton. Ore)	% of Weight	% of Copper	Distribution of Copper (%)
3	Sodium di-secondary Butyl de- phiophosphate (C)	0.05	Concentrate #1 (4.41)	Concentrate #1 (3.75)	Total Concentrate (69.59)
			Tailing (91.49)	Tailing (0.084)	
	Methyl Isobatyl Carbinol (F)	0.066	Concentrate #2 (4.10)	Concentrate #2 (0.35)	Tailing (29.71)
	Potassium Amyl Xanthates (C)	0.03			
	Methyl Isobatyl Carbinol (F)	0.066			
4	Potassium Amyl Xanthates (C)	0.02	Concentrate #1 (8.0)	Concentrate #1 (2.28)	Total Concentrate (74.35)
	Aero-promotes 3501	0.05	Tailing (90.8)	Tailing (0.07)	Tailing (25.65)
	Methyl Isobatyl Xanthates (C)	0.044	Concentrate #2 (1.2)	Concentrate #2 (0.60)	

Table 7.--Continued Resume of Data Obtained from the Preliminary Experiments

Test	Reagents	Quantity (lbs/Ton. Ore)	% of Weight	% of Copper	Distribution of Copper (%)
	Potassium Amyl Xanthates (C)	0.01			
	Methyl Isobatyl Xanthates (C)	0.022			
5	Potassium Amyl Xanthates (C)	0.01	Concentrate #1 (3.7)	Concentrate #1 (4.7)	Total Concentrate (75.6)
	Aero-promoter 3477	0.05	Tailing (90.7)	Tailing (0.068)	Tailing (24.34)
	Methyl Isobatyl Carbinol (F)	0.022	Concentrate #2 (5.6)	Concentrate #2 (0.32)	
	Potassium Amyl Xanthates (C)	0.01			
	Methyl Isobatyl Carbinol (F)	0.022			

remaining tests showed some selectivity, and test 5 showed a high degree of selectivity as is illustrated by the results.

CHAPTER 4

MICROSCOPIC STUDY OF THE TAILING

The particle size of ore to be treated is an important part of any hydrometallurgical process. The mineral must be liberated from the gangue material with the minimum amount of overgrinding.

To control the overgrinding and the oversize, the ball mill must be operated in a closed circuit with a classifier or a screen. The classifier is a device that separates the ball mill discharge into two portions: the finished product, which is ground as fine as desired, and oversize material.

The particle size of the tailings was determined by means of screen analyses. To perform these analyses a sample of the tailing material was shaken on a series of screens according to a certain prescribed technique (16). The amount of material remaining on each screen was weighed and calculated in percentage of the total sample. The results (either tabulated or plotted) represent the screen analysis of that particular sample, and they showed the size distribution of the particles within the sample. When this was performed, each portion on the screen was submitted

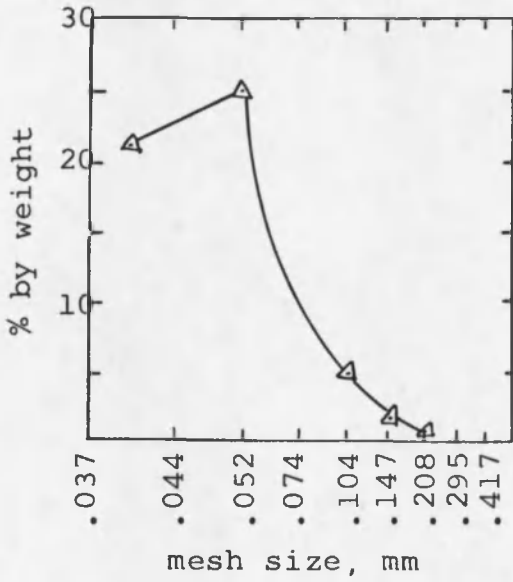
to a microscopic analysis that determined which sizes were present and their percentages (17, 18).

Following the technique described above, these analyses were performed to determine the distribution of the particles and the desired degree of grinding to which the ore must be submitted. The results obtained are expressed on Figures 5, 6, and 7 with the exception of test 1, which was not performed. The data used in the tabulation of these graphs are in Appendix C.

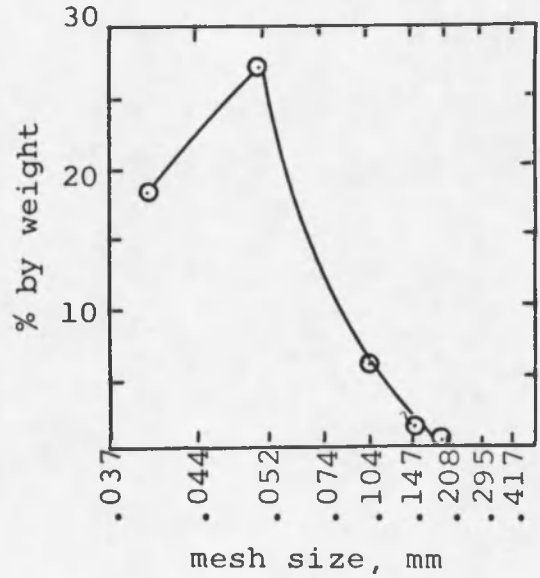
4.1 Discussion of the Results

From Figure 7 we can conclude that the distribution of the particles in the screen analysis of the preliminary tests was approximately the same with small differences. This could be caused by the differences in hardness that exist in the material used. Pictures were taken of each mesh size and the concentrate for test 5 because this was the one that satisfied the objectives of the preliminary test (Figure 8). These pictures illustrate the degree of liberation of the chalcopyrite particles, that for this test was fairly high.

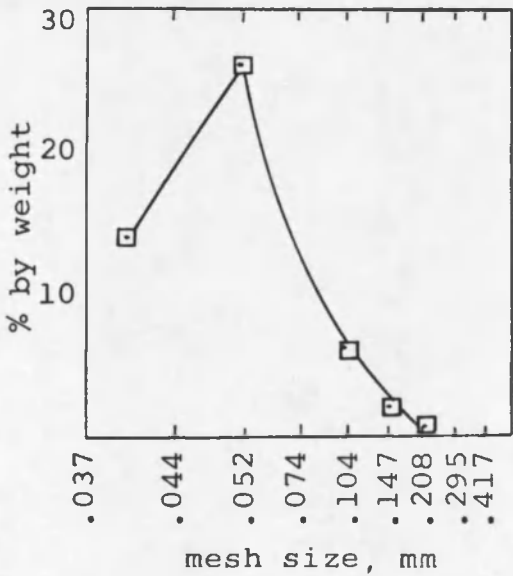
From this microscopic study of test 5 it was determined that the mesh size required for the liberation of chalcopyrite was 49.3 per cent minus 325 mesh for the feed material to the rougher flotation. This conclusion was reached taking into consideration the three prototypes of



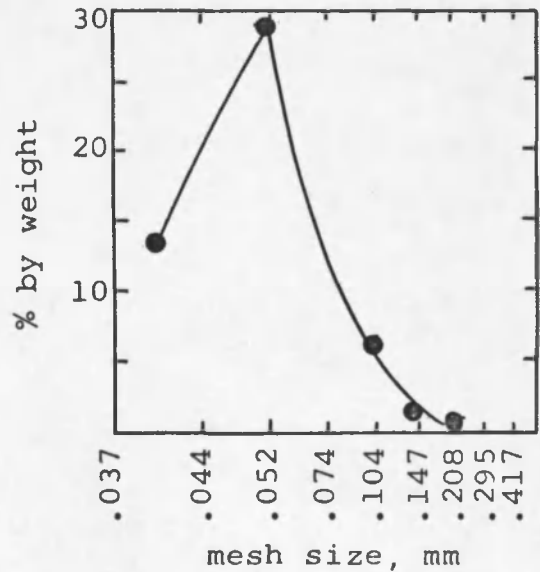
(a) test 2



(b) test 3



(c) test 4



(d) test 5

Figure 5. Direct Charting of a Sizing Analysis (Tailing) -- Data from Appendix C.

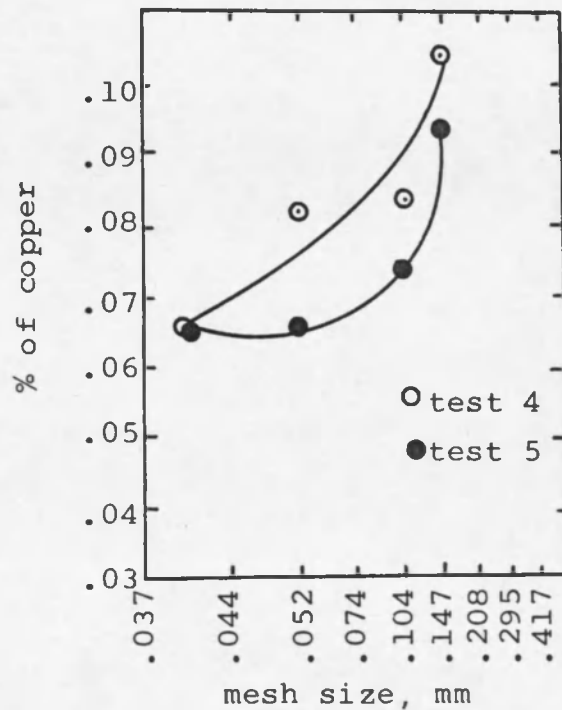
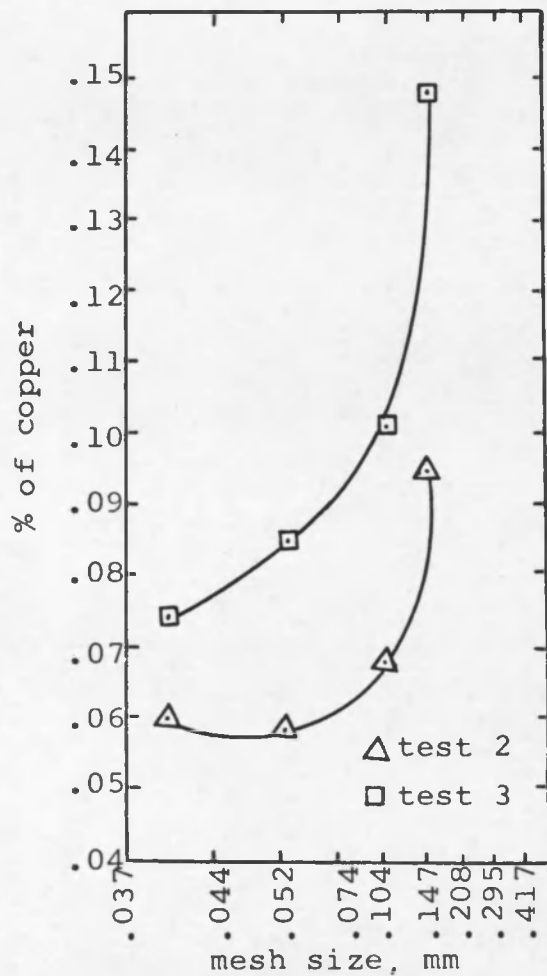


Figure 6. Direct Charting of a Chemical Analysis of Each Screen (Tailing) -- Data from Appendix C.

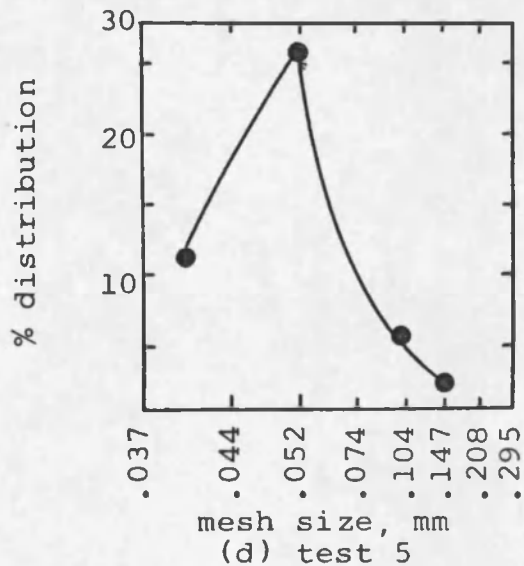
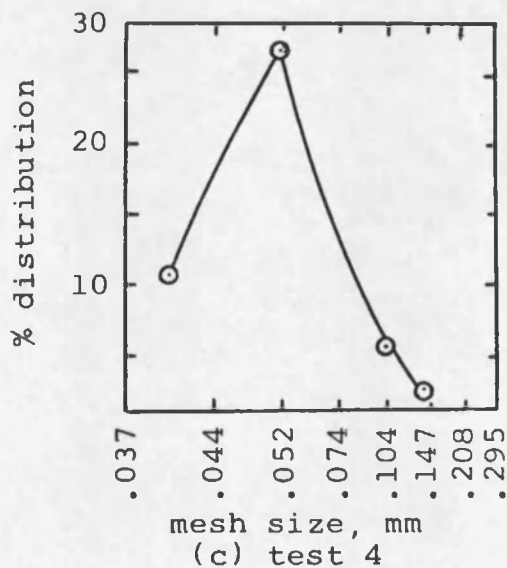
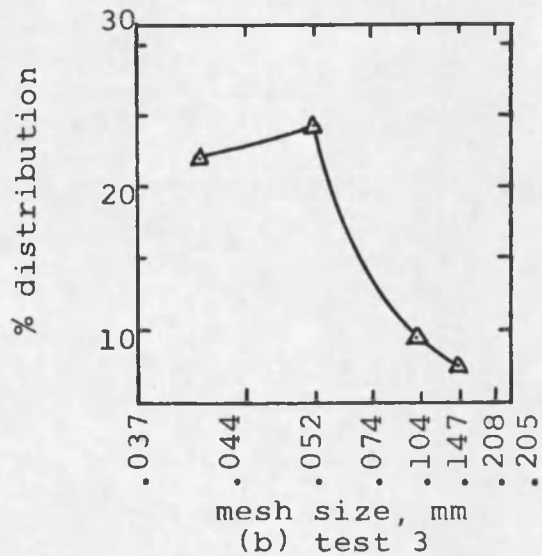
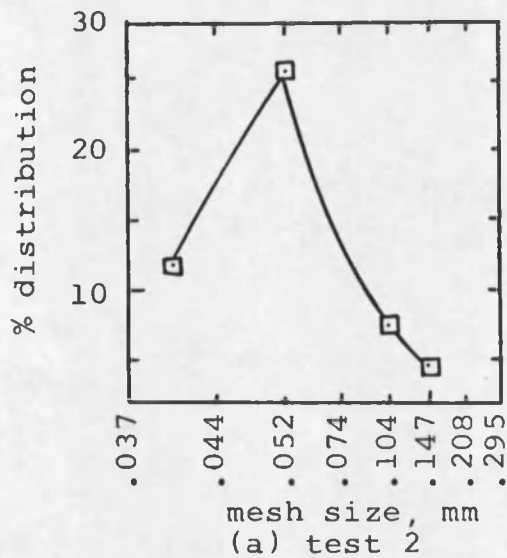
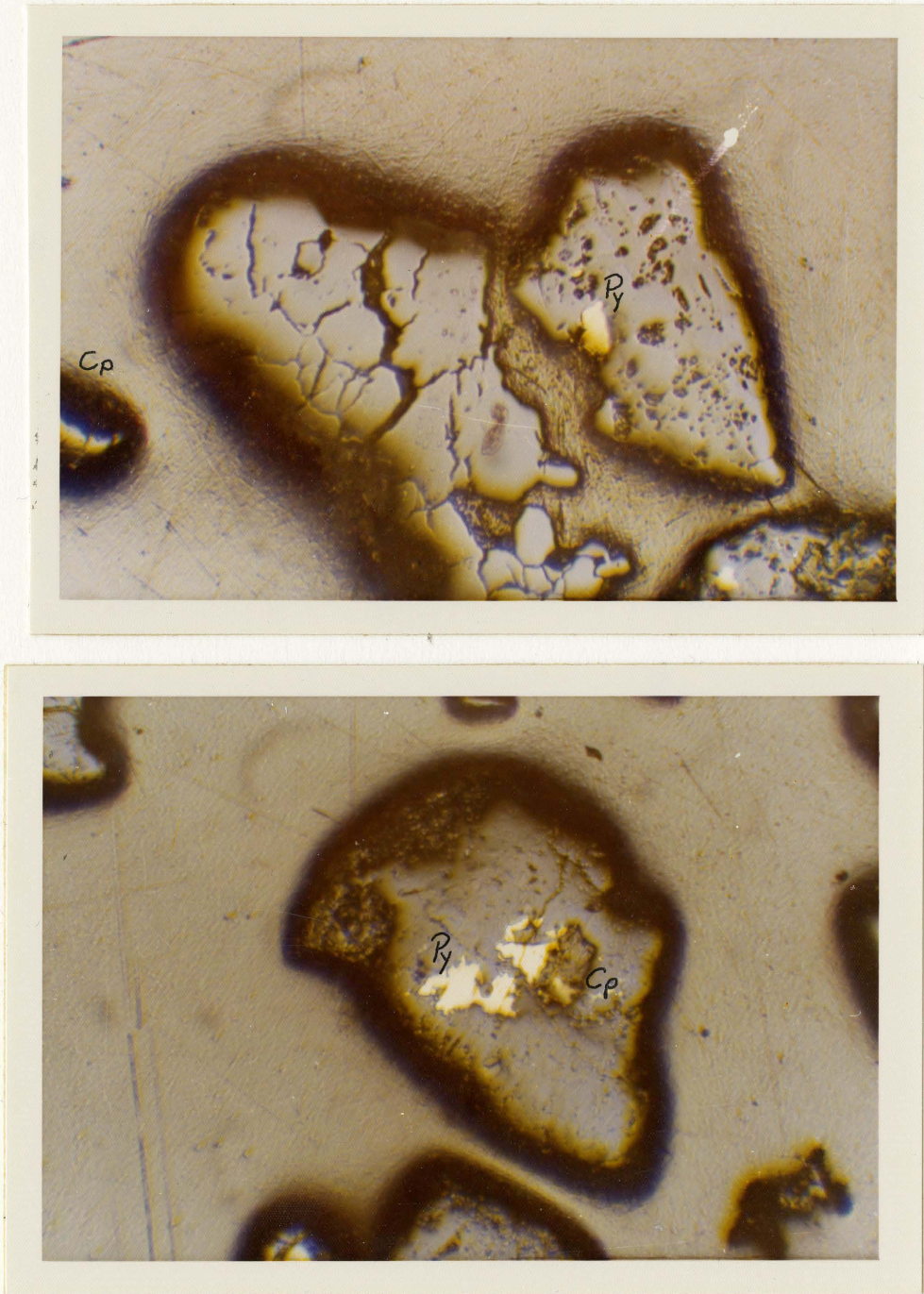
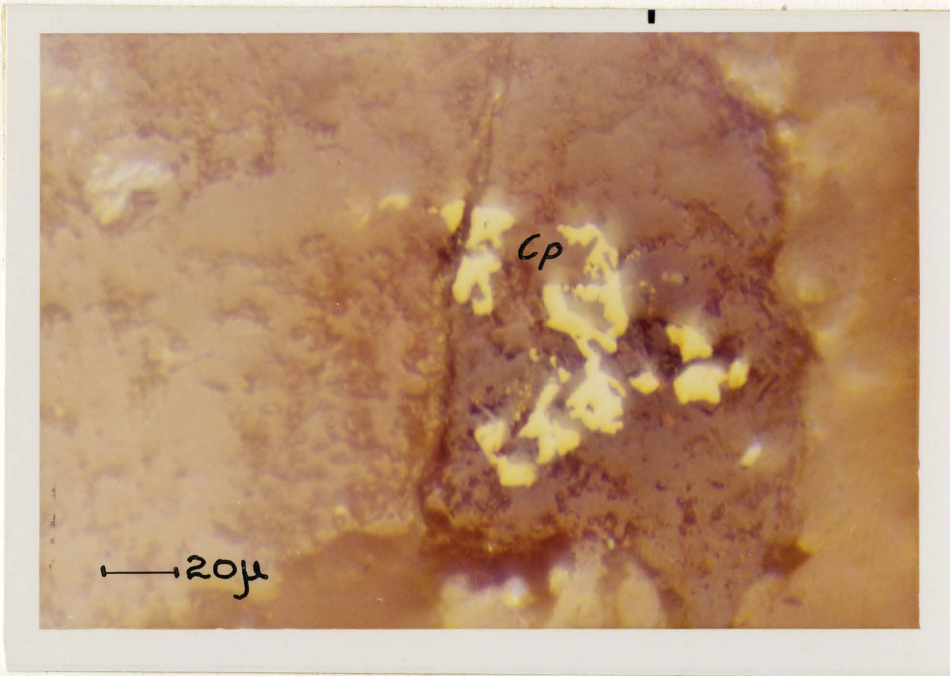


Figure 7. Direct Charting of Per Cent Distribution of Screen Analysis (Tailing) -- Data from Appendix C.

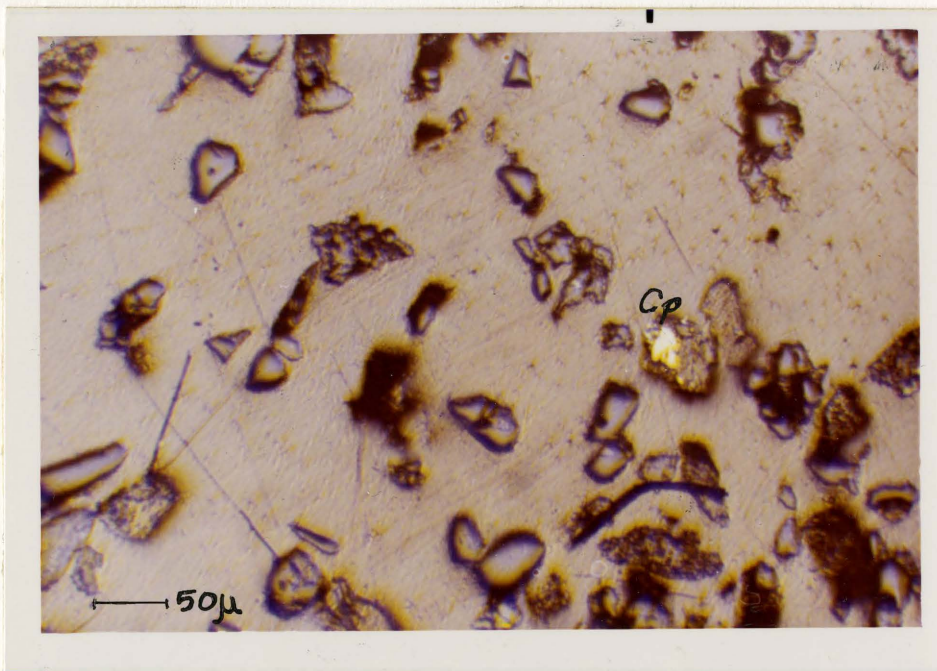


(a) Test 5, minus 48 to plus 65 mesh, shows chalcopyrite (Cp) and pyrite (Py) locked in gangue mineral.

Figure 8. Pictures of Screen Analysis of Tailing of Test 5

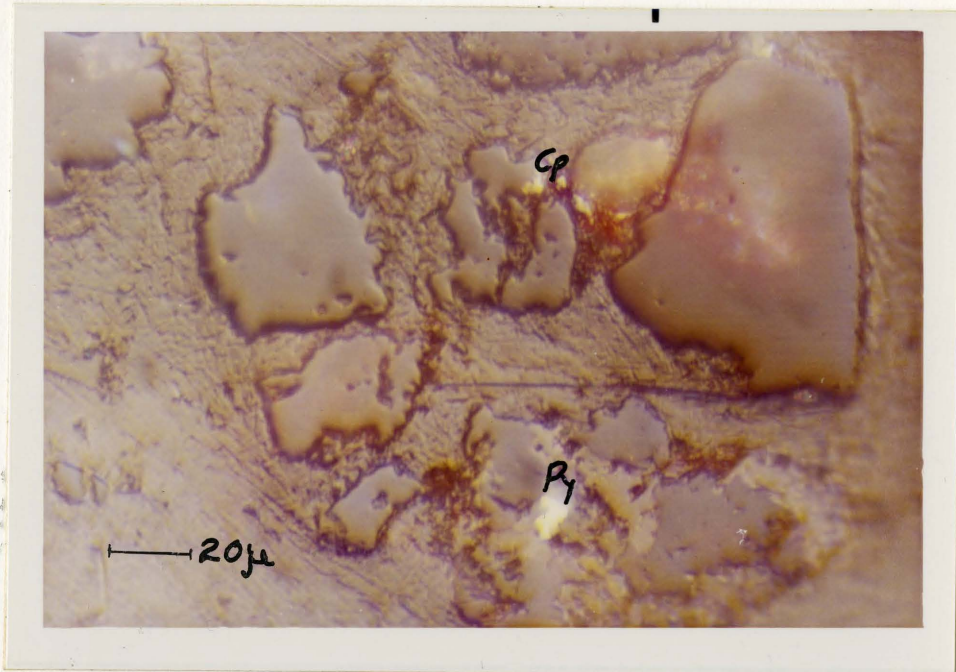


(b) Test 5, tailing, minus 65 to plus 100 mesh, shows chalcopyrite (Cp) locked in gangue mineral.

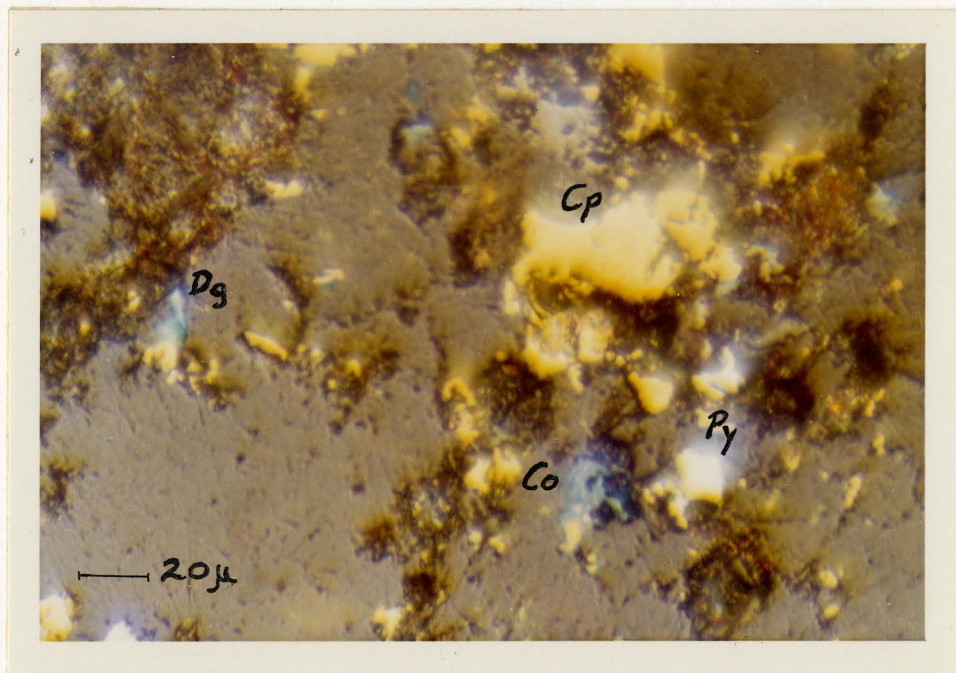


(c) Test 5, minus 100 to plus 200 mesh, shows chalcopyrite (Cp) locked in gangue mineral.

Figure 8.--Continued Pictures of Screen Analysis of Tailing of Test 5



(d) Test 5, minus 200 to plus 325 mesh, shows chalcopyrite (Cp) and pyrite (Py) in gangue mineral.



(e) Test 5, concentrate, shows chalcopyrite (Cp), pyrite (Py), covellite (Co), and digenite (Dg).

ores present in the deposit. For the cleaning step, it was judged that regrinding was required to 100 per cent minus 325 mesh.

CHAPTER 5

FINAL TESTS

After reducing the content of copper in the tailing in the preliminary test, the step to be followed is to try to reach a concentrate that can be used commercially (between 25 to 30 per cent), in other words, that we can use on future extractions of low grade ore (less than 0.4 per cent copper).

If we tried to increase the concentrate grade of the preliminary test 5 (4.7 per cent Cu in 22 grams), we could not reach sufficient quantity to upgrade the rougher concentrate. Therefore it was necessary to quadruplicate the feed of the operation in order to obtain sufficient amounts of concentrate to perform the required test. Also, due to the very low base metal content it probably would be necessary to use two or three stages of cleaners to upgrade the rougher concentrate.

The concentrate of the preliminary test 5 was submitted to X-ray diffraction (19); a certain quantity of talc was found which could not be determined.

The talc in the ore is naturally floatable; this will float with the chalcopryrite. In general, in the industry this is depressed before it is fed to the flotation

cell or in the cleaner section. The talc in this work was depressed in the cleaner section (3).

The first test in this chapter was made without depressing the talc, to compare the effect of the depression of talc with the rest of the tests.

Only the procedure for test 6 will be presented, since there is no difference among the tests except the addition of the depressant reagent, change in time of flotation, and conditioning. The quantity and type of reagent, pH, time of flotation, time of conditioning for these tests will be shown in Appendix D.

5.1 Test 6

Four samples of 600 grams each were floated individually in the same way as on preliminary test 5. The concentrate obtained from each sample was mixed using a screen of 325-mesh, the material classified in two portions of plus 325 and minus 325-mesh size respectively. The portion of plus 325 mesh was introduced in the ball mill with calcium hydroxide and P-5 3477. This was left running for twelve minutes. The product that was obtained from the ball mill was joined with the portion of minus 325 mesh and conditioned again with calcium hydroxide for about one minute, then floated (cleaner flotation) for eight minutes at an initial pH of 12.

The residue in the above flotation cell (cleaner tailing) was conditioned with potassium amyl xanthate and methyl isobutyl carbinol for about two minutes. The flotation (scavenger flotation) took three minutes at an initial pH of 11.5.

The cleaner concentrate obtained in the cleaner flotation was floated (re-cleaner flotation) again for six minutes at an initial pH of 12. For the other tests the time of pH will vary. A complete flowsheet is presented in Figure 9. The results obtained on this test are shown in Table 8.

From Table 8 we notice that the per cent of distribution was 58.1 with a per cent of copper of 16.55. As we can see the result was poor, compared to the purpose of this test, probably due to a bad control of the flotation and conditioning; also the pH was too high.

5.2 Test 7

As we can notice from Table 9 the results obtained were depressive compared with test 6 because the concentrate obtained was too low. This probably was due to the use of the depressant agent.

5.3 Test 8

In Table 10 the results were better but still are low as it keeps feeling the effect of the depressant agent.

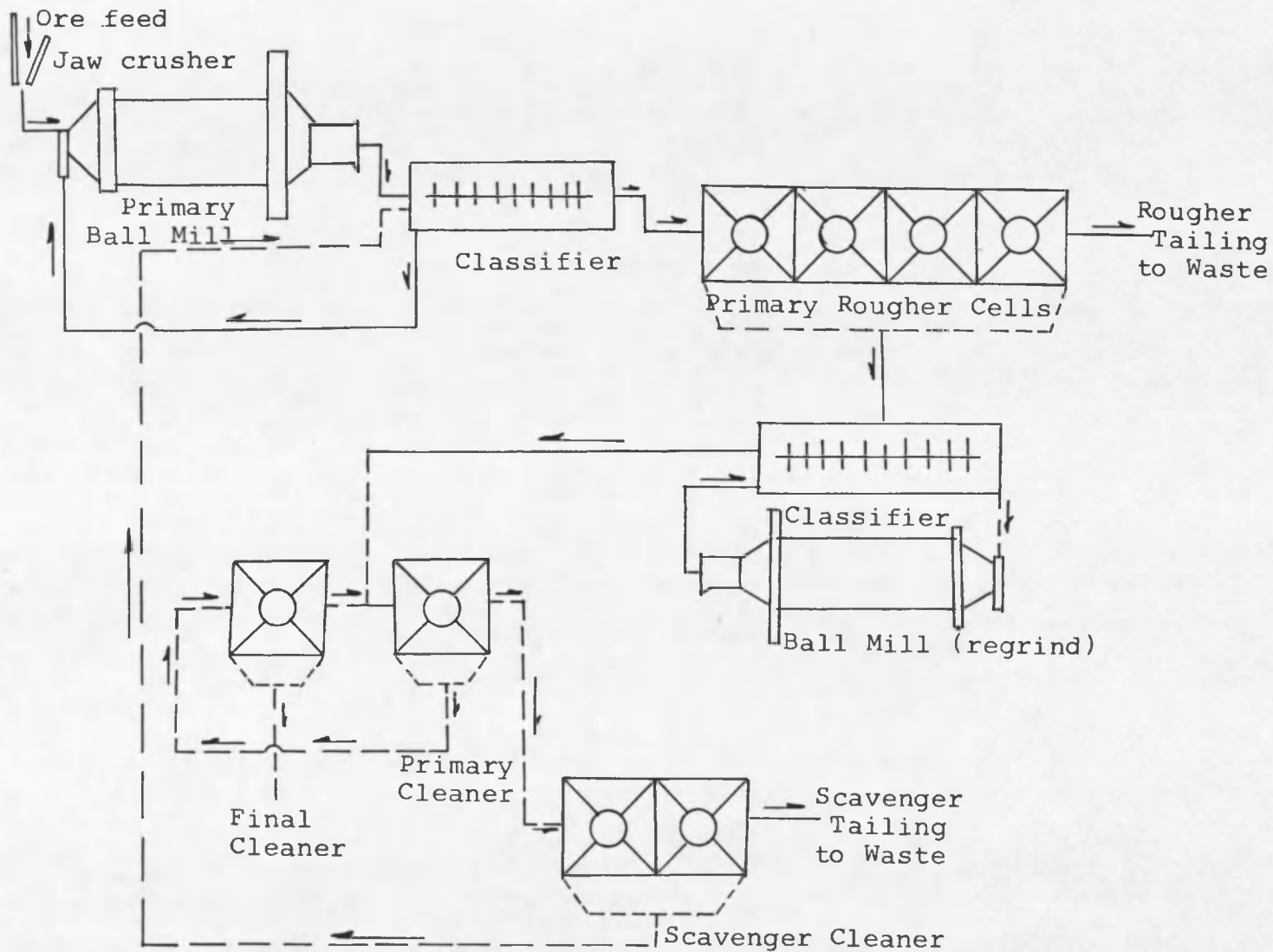


Figure 9. Flowsheet of Experimental Procedure for the Final Test

Table 8. Results of Test 6

Product	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
Head	100.00	0.255	--
Concentrate	0.88	16.550	58.1
Recleaner Tailing	1.33	1.560	8.2
Scavenger Concentrate	0.20	2.760	2.2
Scavenger Tailing	5.47	0.200	4.3
Rougher Tailing	92.12	0.074	27.2

Table 9. Results of Test 7

Product	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
Head	100.00	0.255	--
Concentrate	2.30	5.200	60.8
Recleaner Tailing	2.60	0.680	7.4
Scavenger Concentrate	0.50	2.180	4.6
Scavenger Tailing	3.10	0.168	2.2
Rougher Tailing	91.00	0.066	26.0

Table 10. Results of Test 8

Product	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
Head	100.00	0.255	--
Concentrate	1.90	9.550	70.4
Recleaner Tailing	1.60	0.330	2.0
Scavenger Concentrate	0.30	0.980	1.1
Scavenger Tailing	5.30	0.104	2.1
Rougher Tailing	90.90	2.069	24.3

5.4 Test 9

Test 9 (Table 11) satisfies the objective of the final tests as the per cent of copper in the concentrate is 25.7. The analysis of the concentrate gives results also of iron of 21.6 per cent and 18.2 per cent insoluble.

It was not possible to proceed with more tests because all the material received from Puerto Rico was exhausted.

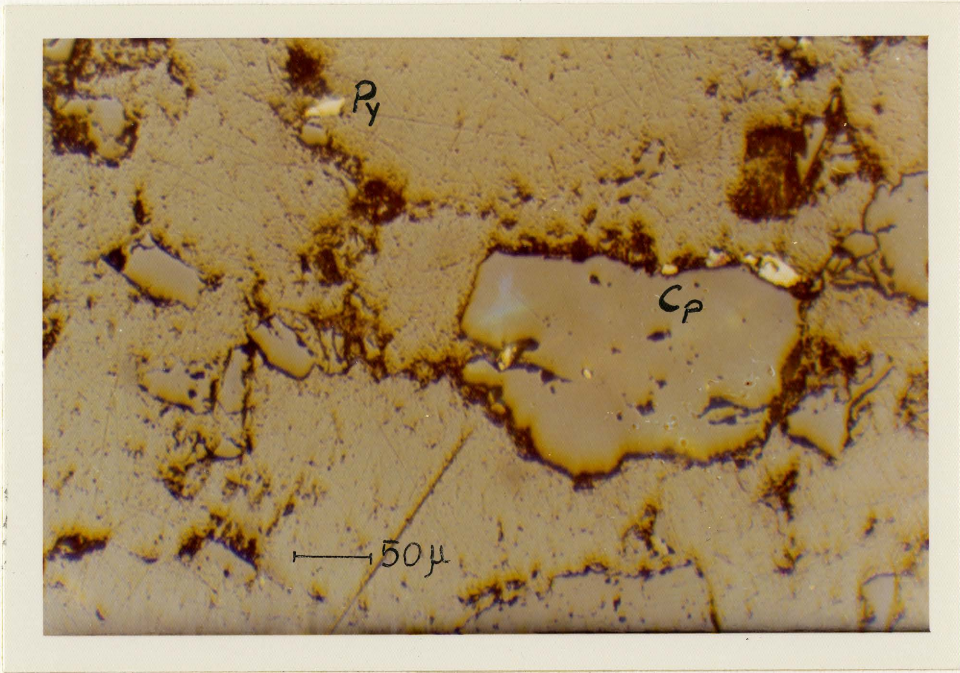
A series of pictures (Figure 10) is presented that shows how the product of each flotation step appeared.

5.5 Discussion of Results

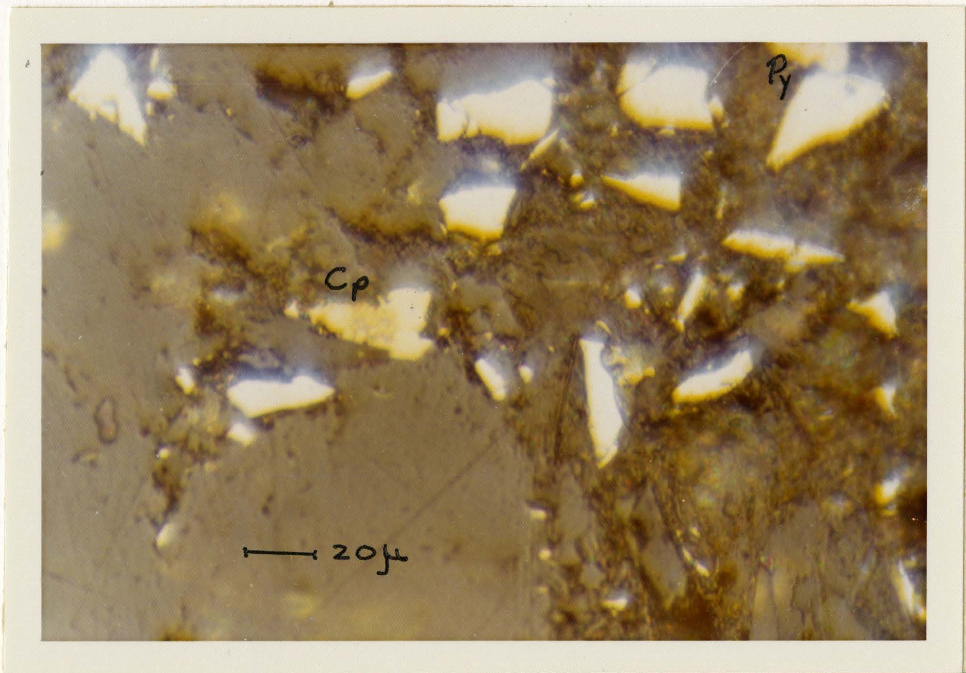
The lower recovery levels reported in tests 7 and 8 could probably be attributed to the use of depressants for

Table 11. Results of Test 9

Product	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
Head	100.00	0.255	--
Concentrate	6.60	25.700	63.1
Recleaner Tailing	0.66	2.000	5.2
Scavenger Concentrate	0.42	3.000	5.0
Scavenger Tailing	8.52	0.174	5.8
Rougher Tailing	89.80	0.059	20.9

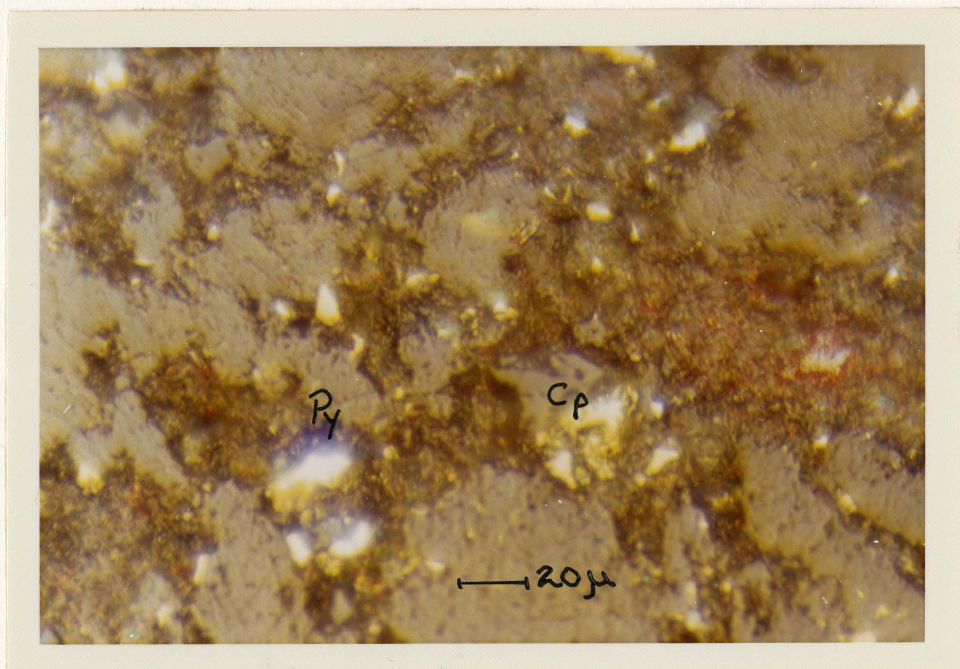


(a) Test 9, rough tailing, intermedia power (3200 M), shows small particle of chalcopyrite (Cp) and pyrite (Py).

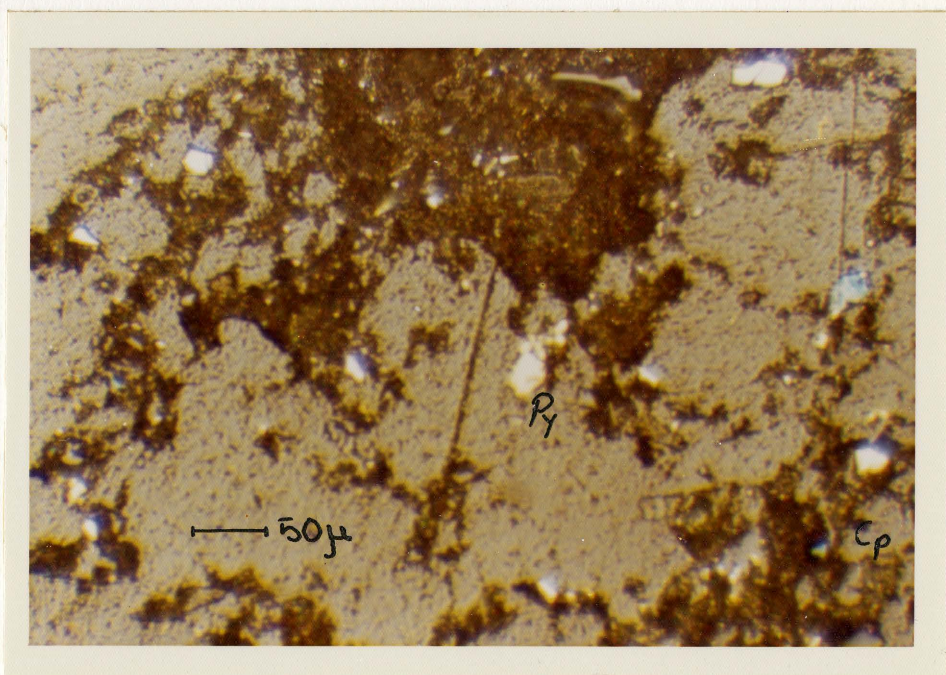


(b) Test 9, scavenger tailing, high power, shows particles of pyrite (Py) with small particles of chalcopyrite (Cp).

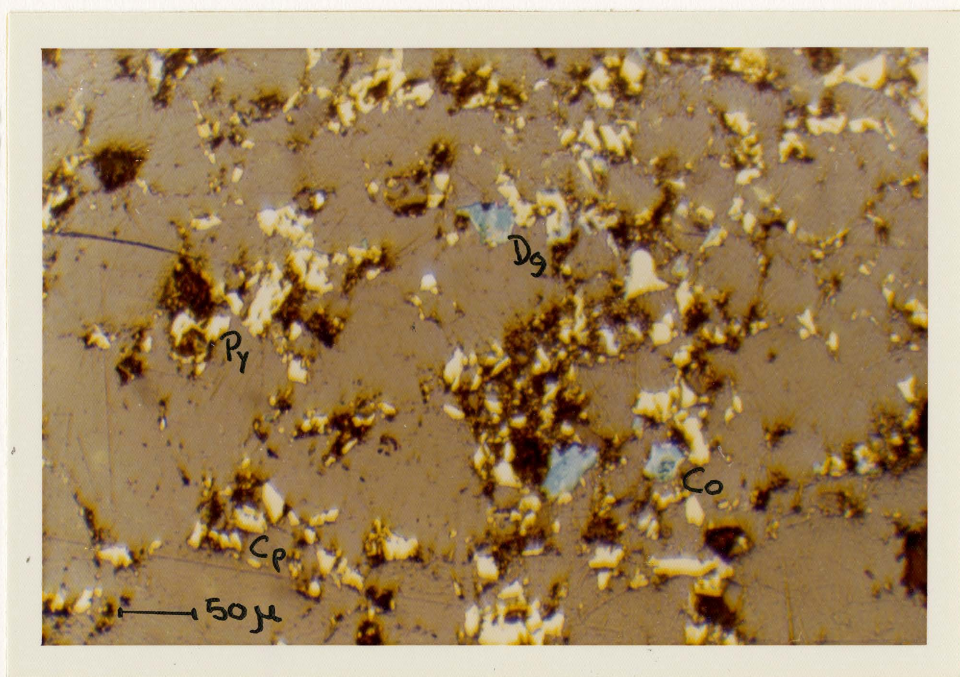
Figure 10. Pictures of the Products of Test 9



(c) Test 9, recleaner tailing, shows particle of chalcopyrite (Cp) and pyrite (Py).



(d) Test 9, scavenger concentrate, shows particle of chalcopyrite (Cp) and pyrite (Py).



(e) Test 9, final concentrate, shows particle of chalcopyrite (Cp), covelite (Co), digenite (Dg), and pyrite (Py).

Figure 10.--Continued Pictures of the Products of Test 9

tale and the control of the pH. This observation is obtained when the results of the above tests are compared with test 6.

One desirable feature of test 9 was the short detection time and the high quality exhibited by the concentrate. The effect of the other variables was minor.

CHAPTER 6

CONCLUSIONS

The following set of conclusions can be drawn:

1. The major losses of sulfide copper mineral values are generally represented by the coarse fraction of the tailing as is shown in Figure 8. This is proven by the fact that when the slimes are removed from these tailings and the sands subjected to flotation treatment a very substantial recovery of copper is registered in a very short period of flotation time.
2. The adsorption of collector on the slimy gangue causes the slimes to float along with the concentrate.
3. The main effect of these slimes is to decrease the flotation rate of the sulfide minerals and reduction of the copper recovery as is mentioned on page 6.
4. The concentrate had a high content of insoluble material due to the initial slime concentration present in the ore.
5. A high copper recovery from the Cala Abajo deposit is obtained if the ore is ground to 49 per cent minus 325-mesh for the feed material to the rougher

flotation and 100 per cent minus 325 to the cleaner section.

6. The principal effect on the flotation step was that of pH and the results clearly indicated that a high pH, about 11, was essential for satisfactory flotation of the copper sulfide minerals and rejection of the pyrite.
7. The use of only one stage of cleaning would probably result in the production of a concentrate of less than the desired copper content and more insoluble than the amount that could be tolerated.
8. It would appear that, considering all the variables, it would be prudent to use a two stage cleaning system. In this manner, a greater probability of consistently maintaining a concentrate of a grade between 25 and 30 per cent copper would be reached.
9. The copper grade produced in the concentrate of test 9 is of acceptable smelter grade.
10. It is concluded from all the information available, that an ore containing a fairly low copper content (0.255 per cent copper) could be concentrated to smelter grade.

APPENDIX A

FLWSHEETS OF CALA ABAJO AND PIEDRA HUECA

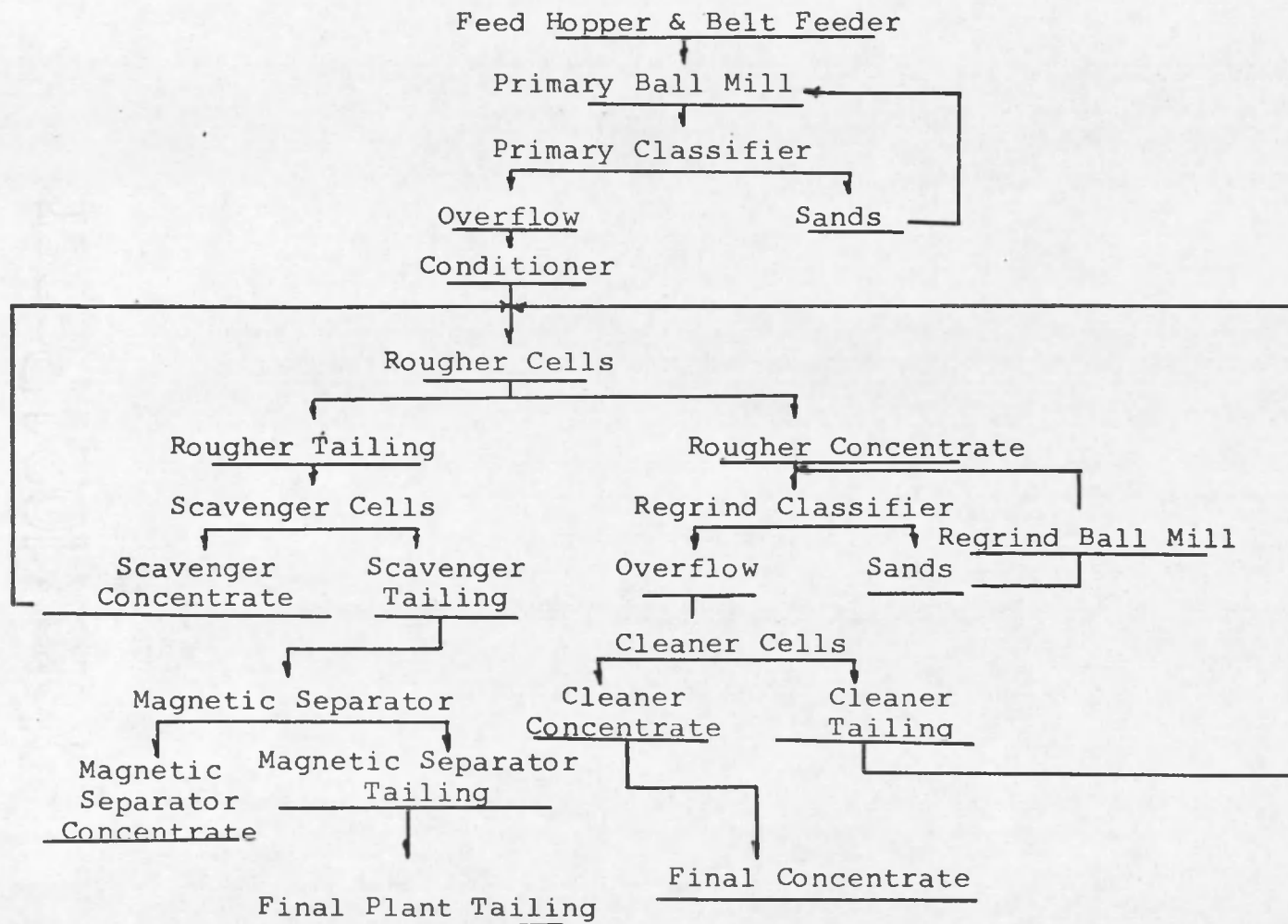


Figure 11. Pilot Plant Scheme for Treating Piedra Hueca Ore

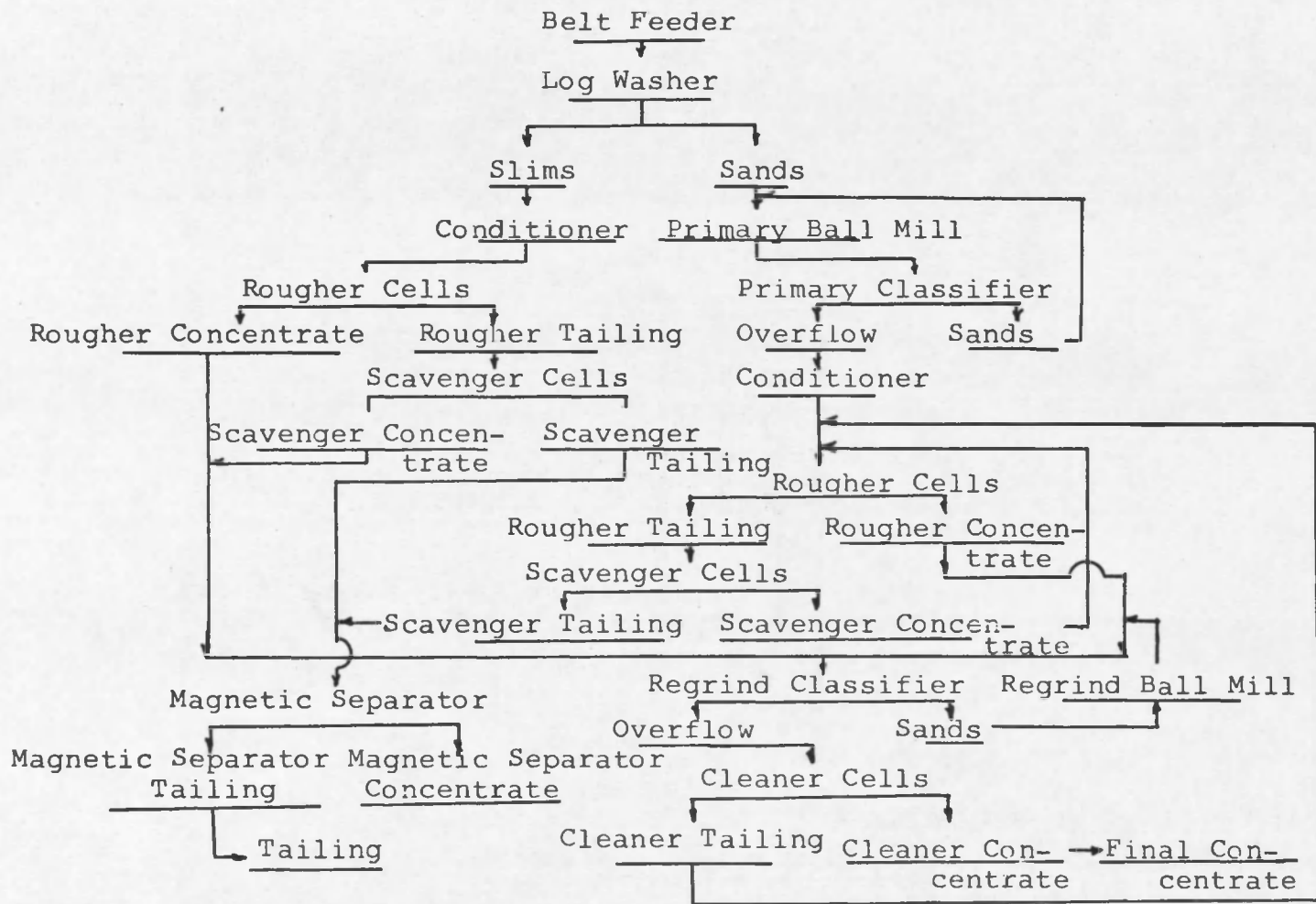


Figure 12. Pilot Plant Scheme for Treating Cala Abajo Ore

APPENDIX B

DESCRIPTION OF THE METHOD AND THE PROCEDURE FOR THE SOLUTION OF THE ATOMIC ABSORPTION SPECTROPHOTOMETER INSTRUMENT

This method depends on the formation of cuprous iodide and iodine when acidified solutions of cupric salts are treated with potassium iodide. This method possesses the advantages of being less subject to interfering elements, a consideration that makes it extremely useful for the analysis of complete materials like copper ores. The most important interfering substances are oxides of nitrogen, arsenic, and antimony that react with iodine. The reaction is usually carried out in an acetate acid solution, of which as much as 50 per cent by volume can be used. An excess of potassium iodide, usually 5 g per 100 ml of solution, is necessary. The titration of the liberated iodine is carried out with a thiosulfate solution which has been standardized by titration of a known amount of copper carried through the iodine method, and starch is not added until most of the iodure has been used up in order to avoid the separation of a difficulty soluble starch-iodine compound.

Procedure

One-half gram of sample was diluted with 10 ml of nitric acid and heated gently until the one sample was decomposed. After this, 3 ml of chloric acid and 1/2 ml of sulfuric acid were added and boiled until the sample dried totally, in other words, that it does not draw out any vapor of fume. Then 30 ml of water were added and heated gurtly. Sufficient amount of ammonium hydroxide was added until heavy precipitate was formed. Then acidify with 10 ml of acetic acid and boil for one minute. Finally, amonium bifluoride was added until the solution changed to blue; cool, and titrate. When the brown tints had nearly disappeared, 5 ml of starch solution were added and the titration was continued until the color changed from blue to yellowish-white.

Analysis for Copper Using the Atomic Absorption Spectrophotometer Instrument

One-half gram of the sample is dissolved in 5 cc of nitric acid (HNO_3) and boiled for approximately five minutes without letting it dry. Then add 5 cc of hydrochloric acid (Hcl) and 1 cc of sulfuric acid and boil again until the sample dries totally; in other words, it does not draw out any vapor or fume. Then 10 cc of hydrochloric acid (Hcl) is added to the sample previously obtained and boiled for approximately 5 minutes without letting it dry. Finally, the solution obtained is read in the Atomic Absorption

spectrophotometer instrument. This equipment was calibrated using a standard assay by Duval Corporation, Jacobs Assay, and Pima Mining Co.

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APPENDIX C

RESULTS OF SCREEN ANALYSIS OF THE TAILING
FOR THE PRELIMINARY TESTS

Table 12. Test 2

Mesh Size	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
+48	0.60	--	--
+65	1.60	0.095	2.8
+100	5.00	0.068	4.6
+200	25.00	0.058	19.7
+325	21.05	0.060	17.2
-325	26.75	0.909	57.2

Table 13. Test 3

Mesh Size	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
+48	0.5	--	--
+65	2.0	0.147	4.19
+100	6.0	0.105	7.18
+200	26.2	0.085	25.38
+325	14.0	0.740	11.81
-325	51.3	0.088	51.44

Table 14. Test 4

Mesh Size	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
+48	0.4	--	--
+65	1.6	0.094	2.56
+100	5.8	0.074	5.84
+200	27.3	0.072	26.77
+325	13.8	0.056	10.53
-325	51.1	0.078	54.30

Table 15. Test 5

Mesh Size	Per Cent By Weight	Per Cent Copper	Distribution Per Cent Copper
+48	0.4	--	--
+65	1.8	0.084	2.86
+100	6.1	0.064	6.05
+200	30.0	0.056	26.02
+325	13.4	0.056	11.62
-325	49.3	0.070	53.45

APPENDIX D

DESCRIPTION OF THE AMOUNT OF REAGENT AND POINT OF
ADDITION FOR FINAL TESTS

Table 16. Test 6

Pulp Conditions				Reagents: lb per ton of ore				
Place	% Solid	Time (Min.)	pH	Ca (OH) ₂	X - 350	P-S-3477	MIBC	A-B-633
Ball Mill	60	6	--	10.0	0.01	--	--	--
Conditioner	30	2	--	--	--	0.050	0.022	--
Primary Rougher Flotation	30	12	11.1	--	0.005	--	0.022	--
Conditioner	20	1	--	11.0	--	0.005	--	--
Cleaner Flotation	20	8	12	--	--	--	--	--
Conditioner	20	2	--	--	0.005	--	0.022	--
Scavenger Flotation	20	3	--	11.5	--	--	--	--
Conditioner	20	1	--	30.0	--	--	0.022	--
Re-Cleaner	20	6	12	--	--	--	--	--

Table 17. Test 7

Pulp Conditions				Reagents: lb per ton of ore				
Place	% Solid	Time (Min.)	pH	Ca (OH) ₂	X - 350	P-S-3477	MIBC	A-B-633
Ball Mill	60	6	--	10	0.01	--	--	0.03
Conditioner	30	2	--	--	--	0.05	0.044	--
Primary Rougher Flotation	30	12	11.1	--	0.005	--	0.022	--
Conditioner	20	2	--	12.5	--	0.005	0.044	--
Cleaner Flotation	20	5.5	11.4	--	--	--	0.022	--
Conditioner	20	2	--	--	0.005	--	0.022	--
Scavenger Flotation	20	3	11.2	--	--	--	--	--
Conditioner	20	1	--	--	--	--	0.044	--
Re-Cleaner Flotation	20	7	11	--	--	--	--	--

Table 18. Test 8

Pulp Conditions				Reagents: lb per ton of ore				
Place	% Solid	Time (Min.)	pH	Ca (OH) ₂	X - 350	P-S-3477	MIBC	A-B-633
Ball Mill	60	6	---	10	0.01	--	--	0.01
Conditioner	30	2	---	---	--	0.05	0.044	--
Primary Rougher Flotation	30	12	11	--	0.005	--	0.022	--
Conditioner	20	2	--	12.5	--	0.005	0.004	0.02
Cleaner Flotation	20	6	12	--	--	--	--	--
Conditioner	20	2	--	--	0.005	--	0.022	--
Scavenger Flotation	20	3	11.8	--	--	--	--	--
Conditioner	20	1	--	--	--	--	0.022	--
Re-Cleaner Flotation	20	3	11.7	--	--	--	--	--

Table 19. Test 9

Pulp Conditions				Reagents: lb per ton of ore				
Place	% Solid	Time (Min.)	pH	Ca (OH) ₂	X - 350	P-S-3477	MIBC	A-B-633
Ball Mill	60	6	--	10	0.01	--	--	0.01
Conditioner	30	2	--	--	--	0.05	0.044	--
Primary Rougher Flotation	30	12	11.1	--	0.005	--	0.022	--
Conditioner	20	2	--	12.5	--	0.005	0.044	0.02
Cleaner Flotation	20	6	11.4	--	--	--	--	--
Conditioner	20	1	--	--	0.005	--	0.022	--
Scavenger Flotation	20	5	11.3	--	--	--	--	--
Conditioner	20	2	--	--	--	--	0.022	--
Re-Cleaner Flotation	20	3	11	--	--	--	--	--

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