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THE DEPRESSION OF QUARTZ

IN SULPHIDE FLOTATION.

BY

W. M. BILLINGHURST.

A

THESIS

submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

in partial fulfillment of the work required for the

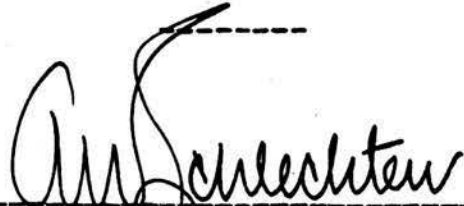
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Chairman, Department of Metallurgy.

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INTRODUCTION.

The flotation of sulphide minerals from siliceous and calcareous gangue minerals always gives a concentrate that is contaminated to a greater or lesser degree with the gangue minerals, even though the collectors used do not show any appreciable collecting properties for the gangue minerals when they are treated alone. In an effort to reduce the amount of undesirable gangue mineral floated with the concentrate it is the practice to add arbitrary amounts of various reagents such as quebracho, alkali hydroxides, carbonates, silicates, sulphides, phosphates, fluorides, and sulphuric acid which have been found to be advantageous. The mechanism of depression is in general not clear, although a number of possibilities are recognised⁽¹⁰⁾ such as, (i)

(10) Taggart, A.F. Handbook of Mineral Dressing. John Wiley and Sons Inc. (New York). 1948. p. 12-23.

prevention of activation resurfacing by soluble salts present in the pulp, (ii) closure of surfaces against collector reactions, (iii) destruction or nullification of collector coatings, (iv) dispersion, (v) resurfacing to produce water avidity. In any given case one or more of these possible mechanisms may be operative.

The use of sodium silicate as a conditioning agent in flotation pulps for the depression of gangue is widespread, and it is desirable that the mechanism of the depressant action be elucidated to allow close control of the flotation process to be obtained.

Two probable mechanisms for sodium silicate depression of gangue have been postulated⁽¹⁰⁾, viz. (i) surface closure

(10) Taggart, A.F. Op. Cit. p.12-35.

of gangue mineral surfaces by silicate ions by mass action effect, and (ii) dispersion of gangue minerals by adsorption of either silicate ions or sodium ions on the surface of the gangue minerals. An effort has been made in the work to be described to determine which of the ions, silicate or sodium, is the effective ion in producing dispersion and hence depression of the gangue minerals in the flotation of sulphides from an artificial pyrite-quartz mixture.

REVIEW OF LITERATURE.

Flotation of any given mineral is intimately bound up with the degree of dispersion or flocculation of that mineral in the pulp and it may be said⁽¹⁴⁾ that flotation is not

(14) Wark, I.W. Principles of Flotation. Australasian Institute of Mining and Metallurgy. (Melbourne). 1938. p.301.

possible in any instance when particles show Brownian movement. Flocculation ensues and Brownian movement ceases when a collector film has been adsorbed.

In considering soap flotation of non-sulphides⁽²⁾ the

(2) Clemmer, J.B. and O'Meara, R.G. U.S.B.M. Reports of Investigations, No. 3239. 1934. p.9.

degree of flocculation is very important since it may be so intense that the concentrates are contaminated with gangue or so moderate that valuable mineral is lost in the tailings. Since fatty acids are not good selective flotation reagents, a control is usually attempted by the addition of reagents to depress one or more of the gangue minerals.

Again high recoveries⁽⁴⁾ and flocculation of sulphides

(4) Fitt, T.C., Thomas, A.W. et al. Trans. A.I.M.E. vol. 153. 1943. p.493.

may be closely correlated due to the binding together of oiled sulphides by minute air bubbles, which is characteristic when at least one of the mineral species is coated with insoluble oil, indicating further that dispersion is a condition unfavourable to flotation.

It was postulated⁽¹¹⁾ that Brownian movement, dispersion,

(11) Taggart, A.F. et al. Trans. A.I.M.E. vol. 112. 1934. p.348.

and non-floatability are all dependent on the condition of the surface of the dispersed particles. It was also postulated that the surfaces of dispersed particles are ionised, the ionised compound being a product of reaction between a component of the particle and a component of the pulp. It was then further asserted, and has since been confirmed⁽¹⁾,

(1) Bankoff, S.G. Trans. A.I.M.E. vol. 153. 1943. p.473.

that sulphides so conditioned with soluble collectors as to be floatable flocculate without the intervention of air bubbles as binding media.

It has been shown⁽¹³⁾ that the floatability of silver

(13) Talmud, D. and Tubman, N.M. Koll. Zeitsch. vol. 50. 1930. p.159.

iodide, using oleic acid, was greatest at the isoelectric point and fell away sharply if either silver ions or iodide ions were in excess. Similar results were obtained for calcium carbonate and barium sulphate, and it has been demonstrated⁽⁵⁾ that organic molecules are adsorbed best by

(5) Frumkin, A. Zeitsch. Physik. vol. 35. 1926. p.792.

electrically neutral surfaces, hence it is concluded that adsorption - and therefore flotation - by collectors that are not in an ionised condition should be at its maximum at the isoelectric point. Flotation by ionised collectors cannot be governed by the same principles⁽¹⁴⁾, which may

(14) Wark, I.W. Op. Cit. p.310.

apply only to van der Waals' adsorption.

A further limitation to satisfactory recoveries of minerals by flotation arises from the hindrance offered to effective collector coating by the coating of the mineral to be floated by fine solid particles commonly designated as slimes⁽¹⁰⁾.

(10) Taggart, A.F. Op. Cit. p.12-20.

Evidence for the dependence of the existence of slime coatings on minerals on the nature of the charges on the various mineral particles is offered as follows⁽¹⁾:

(1) Bankoff, S.G. Op. Cit. p.477.

a. Slime coatings are inhibited when slime particle surfaces are ionised sufficiently for Brownian movement and are facilitated if that is not the case.

b. Both slime coating and flocculation are aided by water repellent surfaces, adhesion taking place when one surface is coated with a substance known to react with the other surface to make it water repellent or if both surfaces are water repellent.

c. Gelatin accelerates both slime coating and flocculation.

These conclusions were confirmed by the use of a cataphoretic cell using converging light and a microscope⁽⁹⁾

(9) Sun Shiou-Chuan. Trans. A.I.M.E. vol. 153. 1943. p.479.

by means of which the velocity of particles under a known potential gradient could be measured and the sign of the charge on the particle determined.

The nature of the charge on particles in suspension has been considered^(6,7), and it has been postulated by Helmholtz

(6) Hauser, E.A. Colloidal Phenomena. McGraw Hill Book Co. Inc. (New York). 1939. pp.91-94.

(7) Phelps, G.W. Ceramis Age. vol. 49. 1947. p.163.

that since the overall charge of a suspension is neutral, the charge on the particle surface is distributed as a double layer with a sudden potential drop at the interface, layers carrying opposite charges being separated by a monomolecular distance, one layer being firmly attached to the particle while the other is movable and may be stripped off during cataphoresis. This hypothesis entails the movement of a dry particle through the liquid dispersion medium.

The charge existing between two layers, which may be considered as oppositely charged plates of a condenser, is as follows:

$$Z = \frac{4\pi ed}{D}$$

where Z = electrokinetic potential.

e = density of charges on surface.

d = thickness of double layer.

D = dielectric constant of liquid medium.

In an effort to overcome the difficulties inherent in

the Helmholtz theory, Gouy formulated a modification in which the charge on the surface of the particle is considered to be distributed in the plane of the surface or imbedded therein, while counter-charges are located in the liquid in such a manner that the double layer extends over greater than monomolecular dimensions into the liquid. The portion in the liquid is considered to be of two parts, one a layer closely attached to the particle which during electrokinetic phenomena remains motionless with the particle and a second layer located in the freely movable liquid and extending to a point of zero charge.

The origin of the charge on the particle has been postulated as being due to several possible mechanisms as follows⁽¹⁵⁾:

(15) Weiser, H.B. Colloidal Chemistry. John Wiley and Sons. (New York). 1939. p.198.

- a. Preferential adsorption of one ion of the electrolyte
- b. Direct ionisation of surface molecules.
- c. Selective adsorption of hydrogen or hydroxyl ions from the water.

The stability of a given suspension is closely related to the magnitude of the zeta potentials of the individual particles, and it will be seen from Helmholtz' equation that the zeta potential is directly affected by changes in the density of charges on the surface and in the thickness of the double layer. These conclusions apply also to the modified theory of Gouy and Freundlich⁽⁶⁾.

(6) Hauser, E.A. Op. Cit. p.106.

An important corollary derived from these conclusions lies in the relation between the thickness of the double

layer and the size, as determined by the degree of hydration, of the ions making up the double layer. Thus ions that may take part in the formation of a double layer may be arranged in the order of their degree of hydration in aqueous solution this order being that of increasing zeta potential and hence stability of the suspension with which they are associated or alternatively that of decreasing flocculating power. Such a series is known as a Hofmeister series as for example as follows⁽⁷⁾:

(7) Phelps, G.T. Op. Cit. p.164.

Cations: H, Al, Ba, Si, NH₄, Na, Li.

Anions: SO₄, NO₃, Cl, OH.

More generally the stability of a suspension depends on the following factors⁽⁷⁾:

(7) Phelps, G.W. Op. Cit. p.163.

a. Particle size. b. Average distance between particles (dilution). c. Character of the liquid. d. Character of the solid. e. Type and amount of electrolyte (character of counterion layer).

A reversal of the sign of the charge on mineral surfaces may take place under the influence of chemical reagents⁽⁹⁾

(9) Sun Shiou-Chuan. Op. Cit. p.482.

as for example sodium silicate can induce a negative charge on galena while ferric chloride and aluminium nitrate can change the surface charge to positive. As regards the mechanism of such an alteration in charge with change in electrolyte it has been suggested⁽⁶⁾ that first the outermost

(6) Hauser, E.A. Op. Cit. pp.107-108.

ions are gradually replaced by new ions while at the same time new ions may also occur in the rigid inner layer by adsorption or ion exchange. The double layer will, at low concentrations of added electrolyte, consist of various positive and negative ions and in such a mixed double layer the possibility exists that charge and thickness of the layer change in different directions. With increasing concentration of added electrolyte, the double layer becomes more and more uniform until it is finally composed exclusively of ions of the added electrolyte.

On the addition of ions having a charge of opposite sign to that of the suspended particles, the diminution of zeta potential prior to a reversal of charge is accompanied by an increased tendency toward flocculation of the particles with associated decrease in Brownian movement, the relative flocculating powers being indicated by the Hofmeister series as previously mentioned.

The importance of the zeta potential of suspended particles in the formation and inhibition of slime coatings has been demonstrated⁽⁹⁾, and it is found that slime coatings

(9) Sun Shiou-Chuan. Op. Cit. p.491.

on minerals are heavy when the zeta potentials of the mineral and slime particles are high and of opposite sign; coatings are light when the zeta potential of the slime is high and the zeta potential of the particle is low, while they are heavy when the zeta potential of the slime is low. Two different mechanisms are thus illustrated in the formation of heavy slimes viz. the mutual attraction of slime and mineral particles in the first case and the flocculation and subsequent deposition on the mineral surfaces of the slime particles when their zeta potential is low as in the second case.

The depression of gangue minerals in flotation requires the use of reagents that will effectively prevent the flocculation of these minerals by favouring the acquisition of a high zeta potential as a result of the adsorption of ions that will also inhibit the adsorption of collectors by these minerals. Various possible procedures that fulfil one or both of these requirements have been suggested as follows⁽¹⁰⁾:

(10) Taggart, A.F. Op. Cit. p.12-23.

a. Prevention of activation resurfacing by soluble salts in the pulp by the precipitation of activating ions; for example, the usual precipitants for heavy metal ions are hydroxyl, carbonate, silicate, and sulphide ions while the earth metal ions require high concentrations of hydroxyl for precipitation as hydroxides, but their basic carbonates, and their phosphates, silicates, and fluorides as well as their metal-acid salts (tungstates, molybdates, etc.) are relatively insoluble.

b. Closure of mineral surfaces as, for example, the addition of sulphide ion may lower the concentration of metal ion on the surface of a mineral until adsorption of collector is insufficient to allow flotation in the case of sulphide minerals. Again cyanide ion may lower the concentration of ferric ion on pyrite with a similar result, and silicate ion may prevent flotation of silicate minerals by both anionic and cationic collectors.

c. Destruction of collector coatings by the application of heat, by the adsorption of organic colloids giving water avid surfaces, or by the provision of acid or alkaline conditions which produce acid and basic soaps which ionise and may decrease collection.

d. Dispersion by mechanisms already discussed.

In the consideration of the effectiveness of a given salt in the dispersion of gangue minerals a further con-

sideration, besides the ability to provide the mineral with a high zeta potential and to inhibit the adsorption of collector, lies in the possibility of the formation of insoluble salts with the ions replaced from the mineral surface⁽⁷⁾.

(7) Phelps, G.W. Op. Cit. p.229.

Thus the soluble silicates are the principal siliceous gangue dispersing reagents used in soap flotation⁽²⁾, being superior

(2) Clemmer, A.B. and O'Meara, R.G. Op. Cit. p.10.

to sodium hydroxide or sodium carbonate used alone owing to their ability to form insoluble salts with many replaced ions.

The mechanism of the action of soluble silicates, notably sodium silicates, in the depression of gangue by dispersion that has been postulated⁽¹⁴⁾ relies on two possibilities

(14) Wark, I.W. Op. Cit. p.294.

as follows;

a. Repression of ionisation of the surface of the mineral particles by free silicate ions in the solution, thus preventing adsorption.

b. Covering of particle surfaces with a layer of charged colloidal silicate micelles, formed by the polymerisation of silicate ions in solution, with the resultant acquisition of a high zeta potential and water avidity by the mineral particle which favours a high degree of particle dispersion.

DISCUSSION.

I. Introduction.

The investigation carried out entailed the examination of the flotation of an artificial quartz-pyrite mixture under

various conditions of pH and reagent addition using a modified Dietrich type flotation cell⁽³⁾.

(3) Dietrich, W.F. et al. U.S.B.M. Reports of Investigations No. 3328. 1937. p.26.

Experiments were conducted to determine, (i) the importance of mechanical entrapment of quartz in a non-mineralised froth using terpeneol alone, (ii) the extent of flotation of quartz using a mineralised froth produced by the use of potassium ethyl xanthate and terpeneol as reagents with a quartz-pyrite pulp and, (iii) the effects of cupric and ferric ions on the recovery of quartz from a pulp using potassium ethyl xanthate and terpeneol as flotation reagents. Since recoveries of quartz proved to be low, the use of potassium ethyl xanthate and terpeneol as reagents did not offer sufficient scope for the examination of the depressant effects of various reagents on quartz, therefore flotation tests were made using sodium oleate as collector, with terpeneol to aid in the formation of a suitable froth.

Using these latter reagents, reproducibility of technique was examined and tests conducted to determine the effect of variation of pH, ferric ion concentration, sodium silicate concentration, sodium chloride concentration, and sodium sulphate concentration on the recovery of quartz from a known quartz-pyrite mixture.

Concentrates and tailings were analysed for insoluble, this being assumed to be identical with the quartz content of these products, and sodium silicate was analysed for sodium in order that the sodium content of the various solutions might be compared.

A comparison of the depressant effects of the various added salts was made with a view to evaluating the importance of the sodium and silicate ions respectively as the effective

depressants.

2. Preparation of Materials and Reagent Solutions.

A sample of Ottawa quartz sand was screened on the Rotap, fractions -65 + 100, -100 + 150, and -150 + 200 mesh being retained for further treatment.

On making up samples for flotation testing a number of 50 gram lots of the size fraction being used were weighed out and treated with hot concentrated hydrochloric acid to ensure the removal of any impurity associated with the quartz and at the same time to relace any adsorbed ions with hydrogen ions. Following several hours treatment the 50 gram samples were washed with distilled water until the pH of successive washes remained approximately constant indicating complete removal of hydrochloric acid. The samples were then stored under distilled water until required.

Pyrite consisting of flotation concentrates from the Tennessee Copper Company were screened for 15 minutes using the Rotap, fractions -65 + 100, -100 + 150, -150 + 200, and -200 mesh being retained. The various size fractions were washed with acetone followed by distilled water to ensure removal of any remaining flotation reagents. Samples for use in the series of flotation tests using potassium ethyl xanthate as collector were dried and 50 gram lots were weighed out and added to previously prepared quartz. Samples used in the tests using sodium oleate were elutriated in the apparatus illustrated in Figure 1 to ensure the removal of as much quartz as possible. The lack of any tendency toward the flotation of pyrite during elutriation indicated satisfactory removal of any flotation reagents remaining on the original concentrates by washing with acetone. Elutriated pyrite was dried and weighed out in 50 gram lots to be added to previously prepared quartz as required.

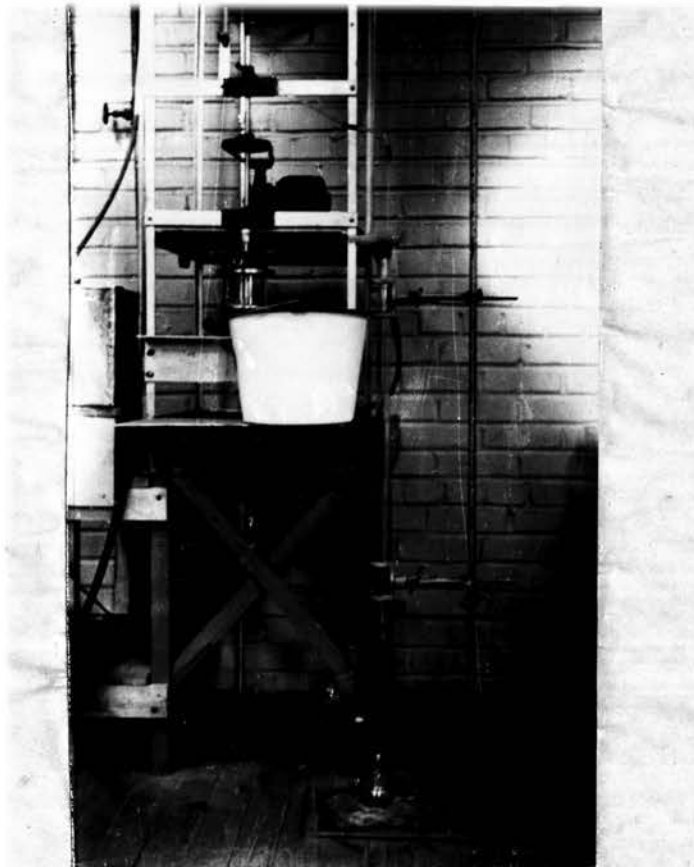


FIGURE 1. ELUTRIATION APPARATUS.

Purification of the available potassium ethyl xanthate was found necessary, the procedure adopted being as follows⁽¹⁴⁾;

(14) Wark, I.W. Op. Cit. p.84.

The sample of impure reagent was washed with ether and filtered to remove di-xanthogen and insoluble material; the remaining insoluble reagent was recrystallised from acetone. A pale cream salt was obtained which was filtered and dried in a desiccator. A solution of 1 gram per litre of purified potassium ethyl xanthate was made up from which additions to the flotation cell were made.

The sodium silicate used was Cyanamid Technical Grade, a solution containing 10 grams per litre being made up.

The sodium oleate used was pure reagent available in the laboratory, a solution of 1 gram per litre being made up, while the terpeneol was Kodak pure reagent, no dilution, being possible.

All the remaining reagents used, sodium chloride, sodium sulphate, sodium hydroxide, copper sulphate, and hydrochloric acid were reagent grade materials all being made up as 5 gram per litre solutions except the sodium hydroxide and hydrochloric acid which were made up as N/10 solutions for use as pH control agents in the flotation cell.

3. Apparatus.

The principal apparatus used during the work was a full size replica of the Dietrich type flotation cell⁽³⁾ constructed

(3) Dietrich, W.F. et al. Op. Cit. p.26.

of lucite as shown in Figure 2. The cell was mounted on a steel frame to allow easy mobility and was driven by a 1/6 H.P. 220 volt D.C. motor connected by single belt drive without reduction. The rated speed of the motor was 1750 r.p.m.

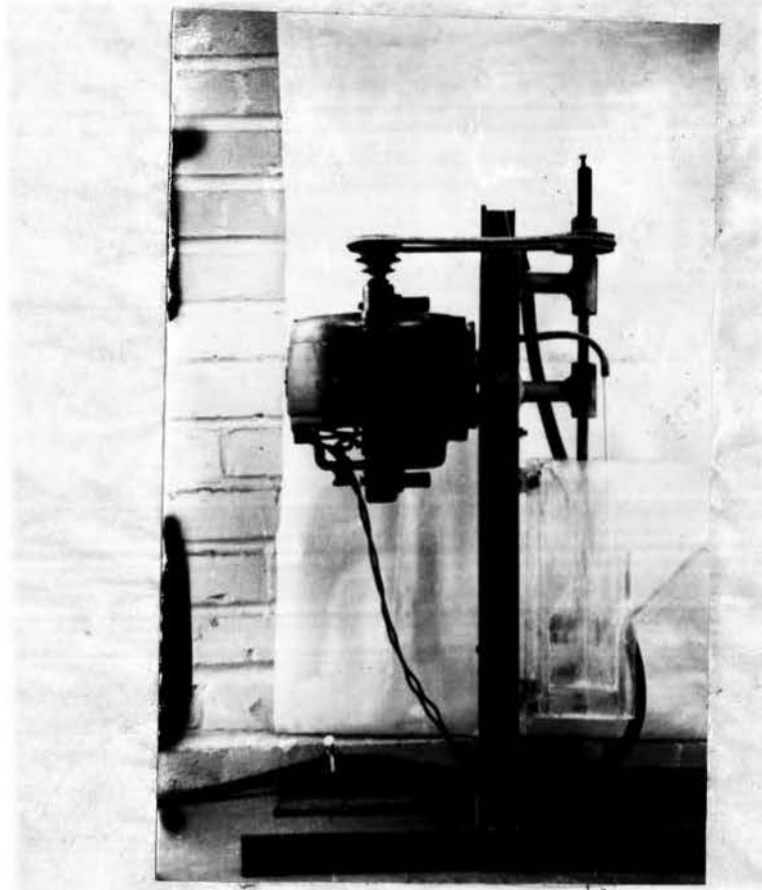


FIGURE 2. DIETRICH TYPE FLOTATION CELL

and a revolution counter showed that the speed of the cell impeller at 220 volt was 1745 r.p.m.

Difficulty was experienced initially due to the vigorous agitation of the pulp in the cell during operation, the agitation being so strong that only a very poor froth formed on the surface while there was a tendency for pulp to be expelled from the cell by splashing. It was found that the introduction of extra air to the impeller zone effectively prevented undue agitation and quiescent conditions on the surface of the pulp were attained. To allow of extra air being admitted during operation a glass tube was attached within the cell with the lower end below the impeller and the upper end connected to the pressure air system through a reduction valve.

The reduction valve inlet gauge was calibrated in pounds per square inch and the outlet gauge in litres per minute. A constant flow of two litres per minute was maintained to the cell during operation.

pH measurements were made with a Beckman Industrial Model pH meter.

4. Experimental Procedure.

Flotation cell experiments were carried out using as nearly identical technique as possible to reduce as far as possible the influence of variables that might affect reproducibility.

Material was weighed out as previously described giving 100 gram flotation samples of 50 grams quartz and 50 grams pyrite. These samples were kept under distilled water until required.

Prior to each test the flotation cell was cleaned using

sodium carbonate, the cell being filled with a concentrated solution and operated for several minutes. Following agitation several washings of tap water were made to ensure the removal of all sodium carbonate and the cell was ready for use in the test.

In conducting a test the mixed quartz-pyrite sample already prepared was washed into the cell using distilled water and the volume made up to within half an inch of the top of the cell with distilled water. The pulp was agitated by operating the cell for a few moments and the pH determined. Standard sodium hydroxide or hydrochloric acid was added to adjust the pH to within 0.3 of the value desired and the reagents added in the following order; salt solution a.g. sodium chloride, sodium silicate etc., collector e.g. sodium oleate or potassium ethyl xanthate, and terpineol, the various reagents being added by means of a pipette with the exception of terpineol which was added by means of a dropper. The pipette and dropper were cleaned with sulphuric acid-potassium dichromate mixture.

Following the addition of the reagents the cell was operated for a few moments to ensure uniform composition throughout. The pH of the pulp was then measured and adjusted as previously described to the required value. The pulp was then conditioned for two minutes by operating the cell with the air turned off. On the completion of conditioning, the pH of the pulp was again determined and, if necessary, adjustment was again made and the reading noted as initial pH.

In making a test the cell was operated for two minutes with the air turned on during which time the pulp level was maintained constant by the addition of distilled water, that previously been adjusted to the pH of the pulp, to the rear

of the cell. The addition of water served to force mineralised froth over the lip of the cell where it was collected as concentrate in an enameled pan. The addition of water during the flotation cell tests was constant, being maintained at 600 ccs. during each test.

After two minutes operation the impeller and air were turned off and the cell emptied; the pH of the tailing pulp was determined and recorded as final pH. It was found that two minutes was ample time to allow for the flotation of the mineral, the bulk of flotation having ceased after one minute.

Concentrates and tailings were filtered, using a vacuum filter to reduce the amount of water to be evaporated, and dried on a hot plate; the dry material was weighed and analysed for insoluble.

5. Analysis of Reagents and Flotation Products.

The analytical work required the determination of sodium in technical grade sodium silicate and the determination of insoluble in the flotation products.

The method used for the determination of sodium was that of Scott⁽⁸⁾, while the determination of insoluble in the flo-

(8) Scott, W.W. Standard Methods of Chemical Analysis. D. von Nostrand Company, Inc. (New York). 1939. p.804.

tation products involved treatment of a given sample with aqua regia followed by dilution and filtration, the insoluble material which was retained was ignited to remove filter paper and weighed; the insoluble was considered to be silica.

6. Flotation Tests and Results.

a. Examination of the importance of mechanical entrapment

on the flotation of quartz.

(1) 100 grams of -100 + 150 mesh quartz was added to the flotation cell with two drops (41.26 mg.) of terpineol and the extent of flotation was examined at various pH values, the pH of the pulp being varied with N/10 sodium hydroxide and N/10 hydrochloric acid as required, the technique being as described above. The results obtained are given in Table 1.

Table 1.

| <u>Flotation of Quartz with Variable pH using Terpineol alone.</u> | | |
|--|----------------------------|--------------------------|
| <u>pH</u> | <u>Extent of Flotation</u> | <u>Remarks</u> |
| 2.3 | No Flotation. | |
| 2.6 | No Flotation. | |
| 3.15 | No Flotation. | |
| 4.45 | No Flotation. | Appreciable flocculation |
| 4.9 | No Flotation. | around air bubbles was |
| 5.3 | No Flotation. | observed in the cell. |
| 5.6 | No Flotation. | |
| 6.0 | No Flotation. | |
| 6.6 | No Flotation. | Floccules remain at the |
| 8.2 | No Flotation. | same density. |
| 9.3 | No Flotation. | |
| 10.05 | No Flotation. | Froth oily and dirty. |
| 10.35 | No Flotation. | |
| 10.6 | No Flotation. | |
| 10.85 | No Flotation. | |
| 11.2 | No Flotation. | |

It will be seen from Table 1 that there is no evidence of any flotation of quartz, although between pH values of 4.5 and 9.0 appreciable flocculation of particles around air bubbles was noted. Mechanical entrapment of quartz

thus would appear to be insignificant when quartz is the only mineral present. However the evidence of strong flocculation in the middle pH range would suggest the possibility that quartz would tend to float in association with a readily floatable mineral.

(ii) 100 grams of pyrite and -100 + 150 mesh quartz were prepared as previously described, the pyrite not having been elutriated. Reagent addition, conditions of flotation, and results were as follows:

| | | pH | | Weight % | Weight % | Recovery % | | | |
|-----------------|-----------|---------|-------|----------|---------------|------------|---------------|------|------|
| KEtX | Terpineol | Initial | Final | Concs. | Insol. Tails. | Insol. | Pyrite Quartz | | |
| 10mg. | 41.26mg. | 7.0 | 6.55 | 63.0 | 3.16 | 68.6 | 86.5 | 86.9 | 3.28 |

From the previous test it would appear that appreciable flotation of quartz would take place under the influence of a heavily mineralised froth, however from the above it will be seen that this is not substantiated, particularly since the pyrite used was found to contain 7.5% silica. Hence it may be concluded that the flotation of quartz in association with pyrite is negligible under the conditions used.

(iii) 100 grams of mixed pyrite and -100 + 150 mesh quartz was prepared as in test (ii) above. It was proposed to conduct a test to indicate the effect of highly flocculating conditions, obtained by conducting the test under acid conditions, on the flotation of quartz in association with pyrite. Reagent addition, conditions of flotation, and results were as follows:

| | | pH | | Weight % | Weight % | Recovery % | | | |
|-----------------|-----------|---------|-------|----------|---------------|------------|---------------|------|------|
| KEtX | Terpineol | Initial | Final | Concs. | Insol. Tails. | Insol. | Pyrite Quartz | | |
| 10mg. | 41.26mg. | 4.8 | 4.94 | 70.0 | 2.52 | 58.5 | 94.1 | 95.2 | 2.83 |

Again it will be seen that recovery of quartz in the concentrate is negligible, indicating mechanical entrapment to

be a minor factor in the contamination of concentrates by gangue under the conditions used.

b. Examination of the effect of various ions on the flotation of quartz in a quartz-pyrite mixture.

(i) Cupric ion. 100 gram samples of a mixture of pyrite and -100 + 150 mesh quartz were prepared as in test 'a (ii)' above. The effect on the flotation of quartz of varying amounts of copper sulphate was examined. Reagent addition, conditions of flotation, and results are given in Table 2, potassium ethyl xanthate and terpineol being maintained constant at 10mg. and 41.26mg. respectively.

Table 2.

| Flotation of Quartz as affected by Copper Sulphate Addition. | | | | | | | | |
|--|---------|-------|----------|--------|----------|--------|--------------|--------|
| CuSO ₄ | pH | | Weight % | | Weight % | | Recoveries % | |
| | Initial | Final | Concs. | Insol. | Tails. | Insol. | Pyrite | Quartz |
| 20mg. | 7.0 | 10.3 | 41.7 | 6.6 | 81.4 | 61.0 | 55.1 | 5.16 |
| 20mg. | 7.0 | 5.9 | 51.2 | 1.73 | 70.0 | 85.2 | 83.0 | 1.32 |

Neither of these tests was carried out under ideal conditions as there was a considerable variation in pH during each test. However it will be seen that in neither case is there a significant recovery of quartz.

(ii) Ferric ion. 100 grams of a mixture of pyrite and -100 + 150 mesh quartz were prepared as in test 'a (ii)' above. The effect on the flotation of quartz of varying amounts of ferric chloride was examined. Reagent additions, conditions of flotation, and results are given in Table 3, potassium ethyl xanthate and terpineol being maintained constant at 10mg. and 41.26mg. respectively.

Table 3.

Flotation of Quartz as affected by Ferric Chloride.

| FeCl ₃ | pH | | Weight % Concs. | Weight % Insol. | Weight % Tails. | Weight % Insol. | Recoveries % | |
|-------------------|---------|-------|--------------------|--------------------|--------------------|--------------------|--------------|--------|
| | Initial | Final | | | | | Pyrite | Quartz |
| Nil | 7.0 | 6.55 | 63.0 | 3.16 | 68.6 | 86.5 | 86.9 | 3.28 |
| 40mg. | 7.0 | 6.6 | 36.3 | 3.7 | 63.1 | 85.4 | 79.2 | 2.14 |
| 80mg. | 7.0 | 6.1 | 67.0 | 2.7 | 55.0 | 92.9 | 94.4 | 3.41 |
| 120mg. | 7.4 | 6.8 | 39.0 | 3.05 | 67.0 | 81.5 | 75.4 | 2.0 |
| 160mg. | 6.9 | 6.4 | 65.0 | 2.65 | 57.0 | 92.9 | 94.0 | 3.11 |
| 200mg. | 7.0 | 6.7 | 63.0 | 4.73 | 51.0 | 96.15 | 96.8 | 4.67 |

It would be expected that a significant increase in recovery would result from the use of ferric chloride as an activating agent, but an examination of the above results shows only a negligible variation in quartz recovery.

In view of the lack of promise of results using potassium ethyl xanthate as collector under various conditions of flocculation and activation, the experimental conditions in later tests were altered by the replacement of potassium ethyl xanthate as collector by sodium oleate.

c. Tests to indicate the degree of reproducibility attainable.

50 grams each of -100 + 150 mesh pyrite and -65 + 100 mesh quartz were prepared as previously; the pyrite was cleaned by elutriation and hence contained only 1.76% insoluble. Reagent addition, conditions of flotation, and results are given in Table 4, sodium oleate and terpeneol being maintained constant at 10mg. and 41.26mg. respectively.

Table 4.

Examination of Reproducibility Attainable.

| Initial | pH | | Weight % Concs. | Weight % Insol. | Weight % Tails. | Weight % Insol. | Recoveries % | |
|---------|-------|------|--------------------|--------------------|--------------------|--------------------|--------------|--------|
| | Final | | | | | | Pyrite | Quartz |
| 8.2 | 8.2 | 21.5 | 43.8 | 78.0 | 53.0 | 24.8 | 18.4 | |
| 7.8 | 8.0 | 25.5 | 45.7 | 74.0 | 52.0 | 28.1 | 23.3 | |

A later examination of the effects of pH variation on the recovery of pyrite and quartz shows that a significant decrease in recovery may be expected on changing the pH from 7.8 to 8.2, hence it would appear that agreement between the two results is even closer than would be apparent from cursory examination.

Further results showing the technique to be capable of producing results agreeing within reasonably close limits is shown in Table 5; the collector and frother additions were the same as in the tests shown in Table 4, but the material used was -100 + 150 mesh quartz and -65 + 100 mesh pyrite.

Table 5.

Examination of Reproducibility Attainable.

| pH | | Weight | % | Weight | % | Recoveries % | |
|---------|-------|--------|--------|--------|--------|--------------|--------|
| Initial | Final | Concs. | Insol. | Tails. | Insol. | Pyrite | Quartz |
| 8.6 | 8.8 | 15.5 | 67.3 | 80.5 | 47.0 | 10.65 | 21.7 |
| 8.4 | 8.5 | 14.0 | 59.73 | 84.5 | 48.76 | 11.5 | 16.9 |
| 8.5 | 8.4 | 10.8 | 58.14 | 89.5 | 51.9 | 9.47 | 11.2 |

d. Examination of flotation with variable pH.

50 grams each of -100 + 150 mesh pyrite and -65 + 100 mesh quartz were prepared as in tests 'c'. pH control was obtained by the use of N/10 sodium hydroxide and N/10 hydrochloric acid. Reagent addition, conditions of flotation, and results are given in Table 6, sodium oleate and terpineol being maintained constant at 10mg. and 41.26mg. respectively.

Table 6.

Quartz and Pyrite Recovery with Variable pH.

| pH | | Weight Concs. | % Insol. | Weight Tails. | % Insol. | Recoveries % | |
|---------|-------|------------------|-------------|------------------|-------------|--------------|--------|
| Initial | Final | | | | | Pyrite | Quartz |
| 6.4 | 7.4 | 99.0 | 50.79 | 1.0 | 37.3 | 98.6 | 99.2 |
| 7.3 | 8.0 | 95.5 | 51.32 | 3.0 | 33.5 | 96.0 | 98.0 |
| 8.4 | 8.7 | 77.0 | 51.24 | 23.0 | 47.62 | 84.0 | 78.4 |
| 8.9 | 9.1 | 4.5 | 50.34 | 97.0 | 48.41 | 2.92 | 4.60 |
| 8.9 | 9.6 | 10.5 | 51.46 | 90.5 | 49.16 | 10.0 | 10.8 |
| 9.3 | 9.4 | 17.0 | 40.0 | 83.0 | 52.8 | 20.7 | 13.4 |

Plots of the recoveries of pyrite and quartz respectively against pH value are shown in Figures 3 and 4.

It will be seen that a considerable decrease in the recovery of both pyrite and quartz accompanies even a small increase in the pH value of the pulp and hence difficulty may be expected in obtaining reproducible recoveries owing to the difficulty of maintaining an absolutely constant pH value during any one experiment.

e. Examination of flotation as affected by varying concentrations of added salts.

(i) Variable ferric chloride. 50 grams each of -100 + 150 mesh pyrite and -65 + 100 mesh quartz was prepared as in test 'c' above. Reagent addition, conditions of flotation and results are given in Table 7, sodium oleate and terpeneol being maintained constant at 10mg. and 41.26 mg. respectively.

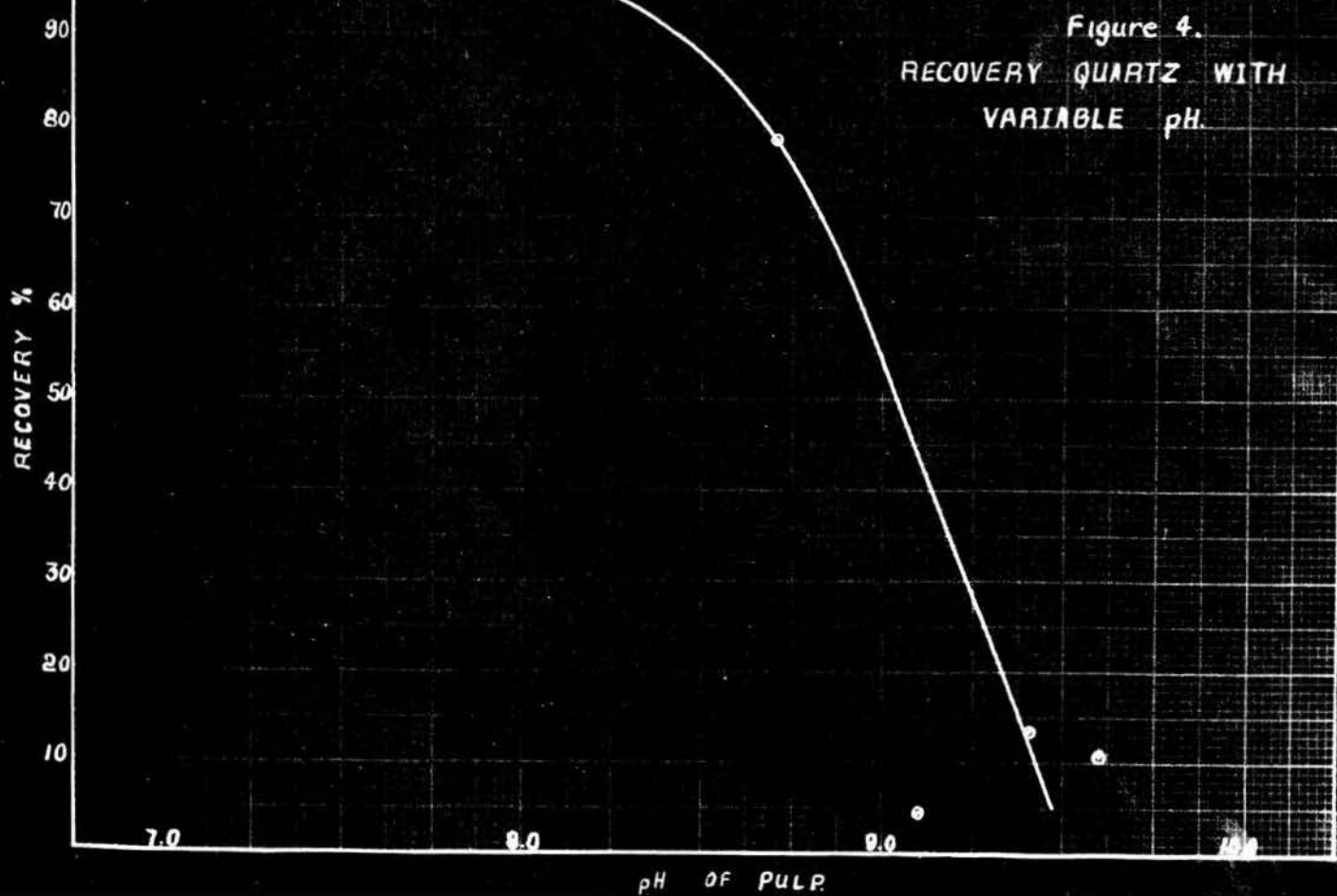
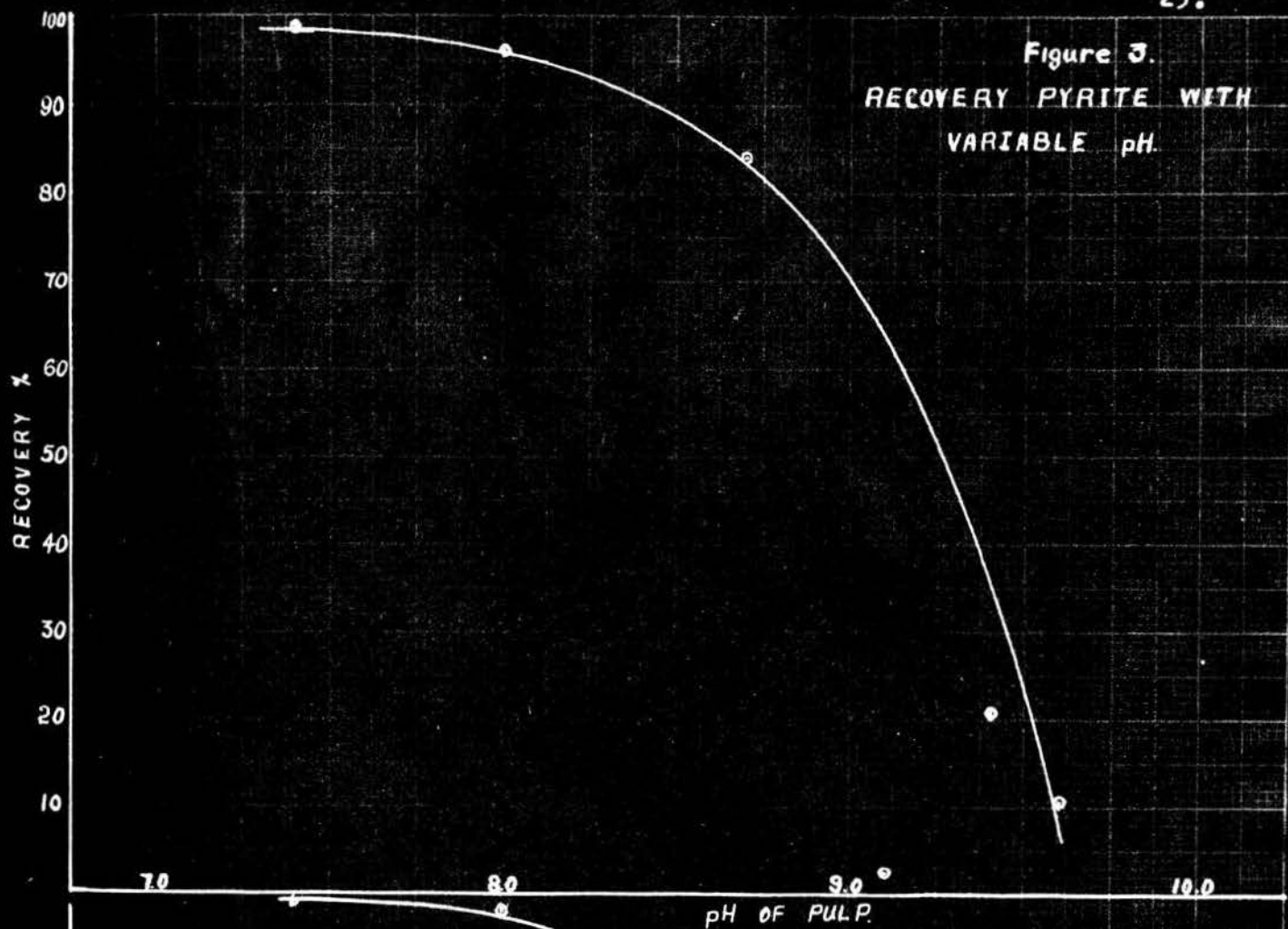


Table 7.

Flotation of Quartz and Pyrite with Variable Ferric Chloride.

| FeCl ₃ | pH | | Weight % Concs. | % Insol. | Weight % Tails. | % Insol. | Recoveries % | |
|-------------------|---------|-------|--------------------|----------|--------------------|----------|--------------|--------|
| | Initial | Final | | | | | Pyrite | Quartz |
| 5mg. | 8.6 | -- | 21.0 | 35.13 | 76.5 | 55.32 | 28.6 | 14.85 |
| 10mg. | 8.6 | 8.2 | 15.0 | 38.44 | 80.0 | 53.76 | 20.0 | 11.18 |
| 50mg. | 8.4 | 8.0 | 2.5 | 18.6 | 96.2 | 51.0 | 4.15 | 0.93 |
| 100mg. | 8.2 | 7.9 | 4.0 | 50.8 | 94.2 | 49.7 | 4.0 | 4.15 |
| 150mg. | 8.0 | 7.8 | 5.7 | 50.33 | 92.4 | 49.95 | 5.75 | 5.88 |

Plots of the recoveries of pyrite and quartz respectively against ferric chloride addition are shown in figures 5 and 6.

It will be seen that ferric chloride acts as a depressant for both pyrite and quartz possibly due to consumption of available sodium oleate. It is to be anticipated⁽¹²⁾

(12) Taggart, A.F. et al. Trans. A.I.M.E. vol. 134. 1939. p.195.

that in the presence of sufficient collector ferric chloride would act as a strong activator for quartz and hence it had been hoped to study the depressant effect of various salts on quartz sufficiently activated as to give a high recovery in the absence of depressants. However in view of the depressant effect of ferric chloride indicated in tests 'c (i)' and since a study of recoveries obtained in test 'd' show a high quartz recovery below pH 8.0 it was decided to conduct the investigation of the depressant effect of various salts at pH 8.0 without the prior activation of quartz.

(ii) Variable sodium silicate. Samples of 50 grams each of -100 + 150 mesh quartz and -150 + 200 mesh pyrite were prepared as in test 'c' above. Reagent addition, conditions of flotation, and results are given in Table 8, sodium oleate and terpeneol being maintained constant at 10mg. and 41.26mg. respectively.

Figure 5.
RECOVERY PYRITE WITH
VARIABLE FERRIC
CHLORIDE.

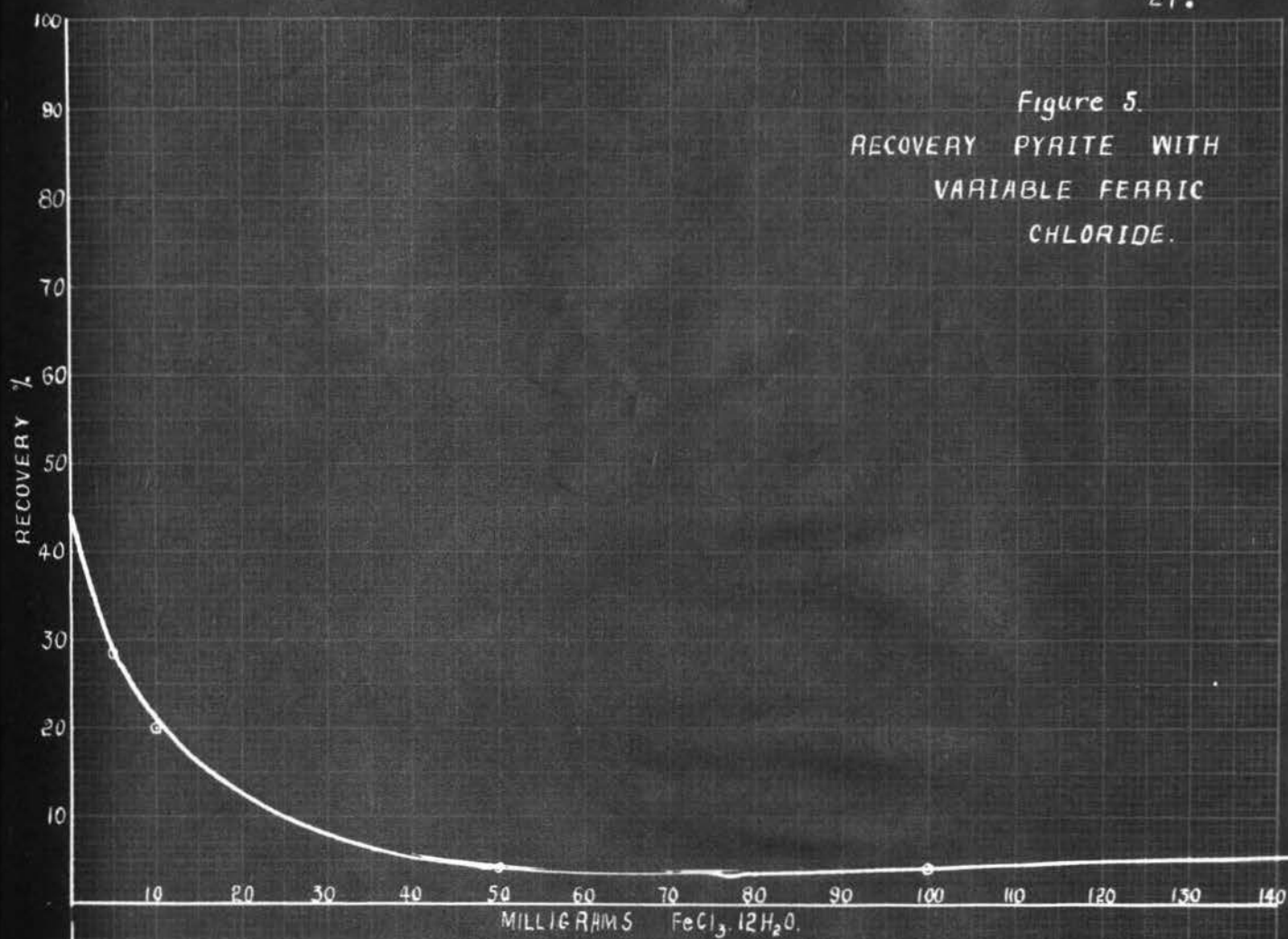


Figure 6.
RECOVERY QUARTZ WITH
VARIABLE FERRIC
CHLORIDE.

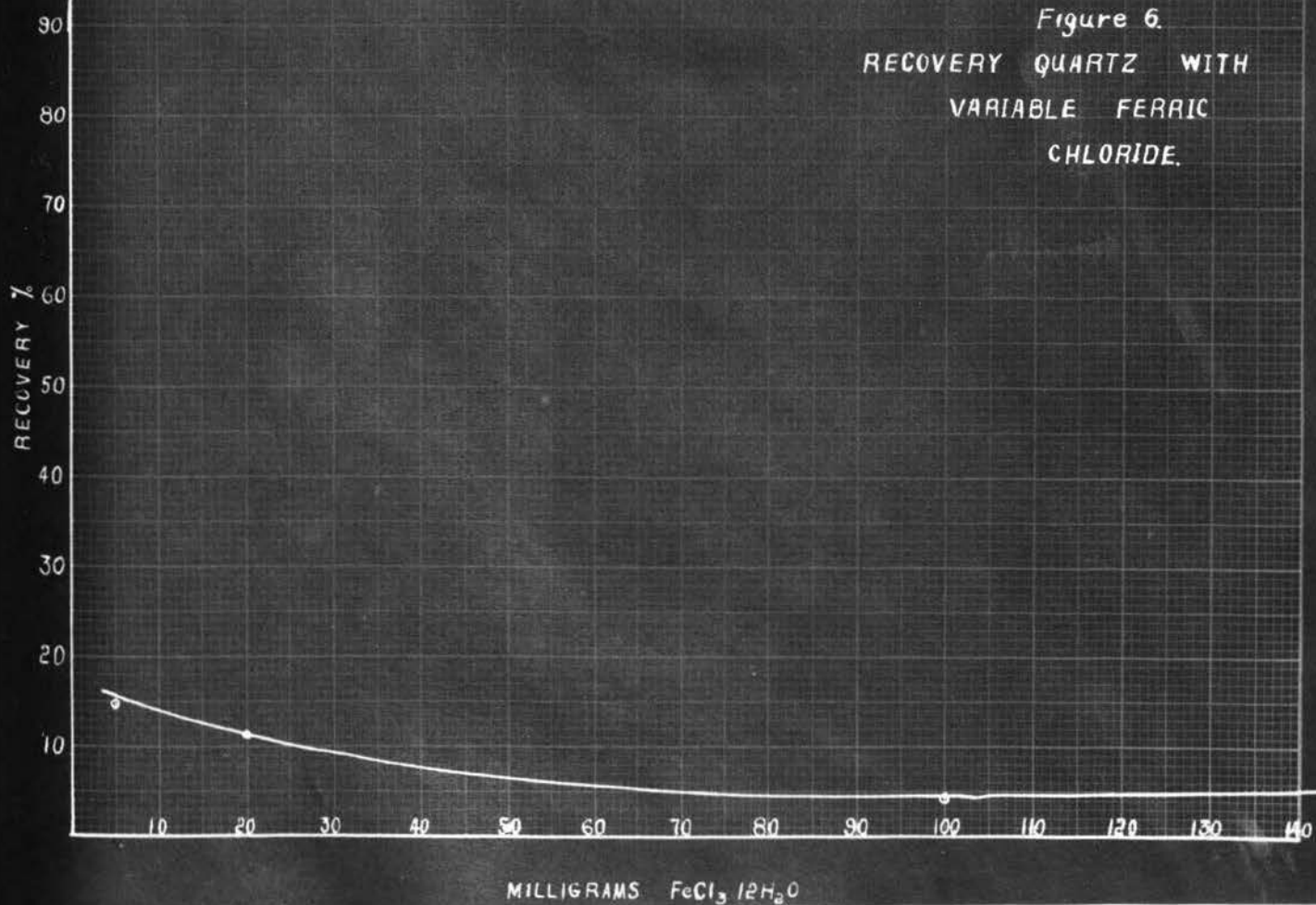


Table 8.

Flotation of Quartz and Pyrite with Variable Sodium Silicate.

| Sodium Silicate | pH | | Weight % Concs. | Weight % Insol. | Weight % Tails. | Weight % Insol. | Recoveries % | |
|-----------------|---------|-------|--------------------|--------------------|--------------------|--------------------|--------------|--------|
| | Initial | Final | | | | | Pyrite | Quartz |
| Nil | 8.0 | 8.2 | 51.8 | 34.2 | 45.8 | 69.3 | 70.8 | 35.7 |
| 20mg. | 8.0 | 8.3 | 43.7 | 20.4 | 54.7 | 74.8 | 71.8 | 17.8 |
| 50mg. | 8.0 | 8.1 | 39.0 | 15.2 | 59.5 | 73.6 | 67.9 | 11.9 |
| 100mg. | 8.0 | 7.8 | 16.8 | 14.7 | 81.4 | 58.0 | 29.5 | 4.97 |

Plots of the recoveries of pyrite and quartz respectively are shown in Figures 7 and 8.

It will be noted that significant differences exist between the recoveries obtained in comparable tests in series 'e (ii)' and series 'c'. A difference in particle size exists between the two series however, limitations in the supply of material not allowing one particle size to be used throughout, and it is probable that this difference in particle size is reflected in different recoveries.

(iii) Variable sodium chloride. Samples of 50 grams each of -100 + 150 mesh quartz and -150 + 200 mesh pyrite were prepared as in test 'c' above. Reagent addition, conditions of flotation, and results are given in Table 9, sodium oleate and terpeneol being maintained constant at 10 mg. and 41.26mg. respectively.

Figure 7.
RECOVERY PYRITE WITH
VARIABLE SODIUM
SILICATE.

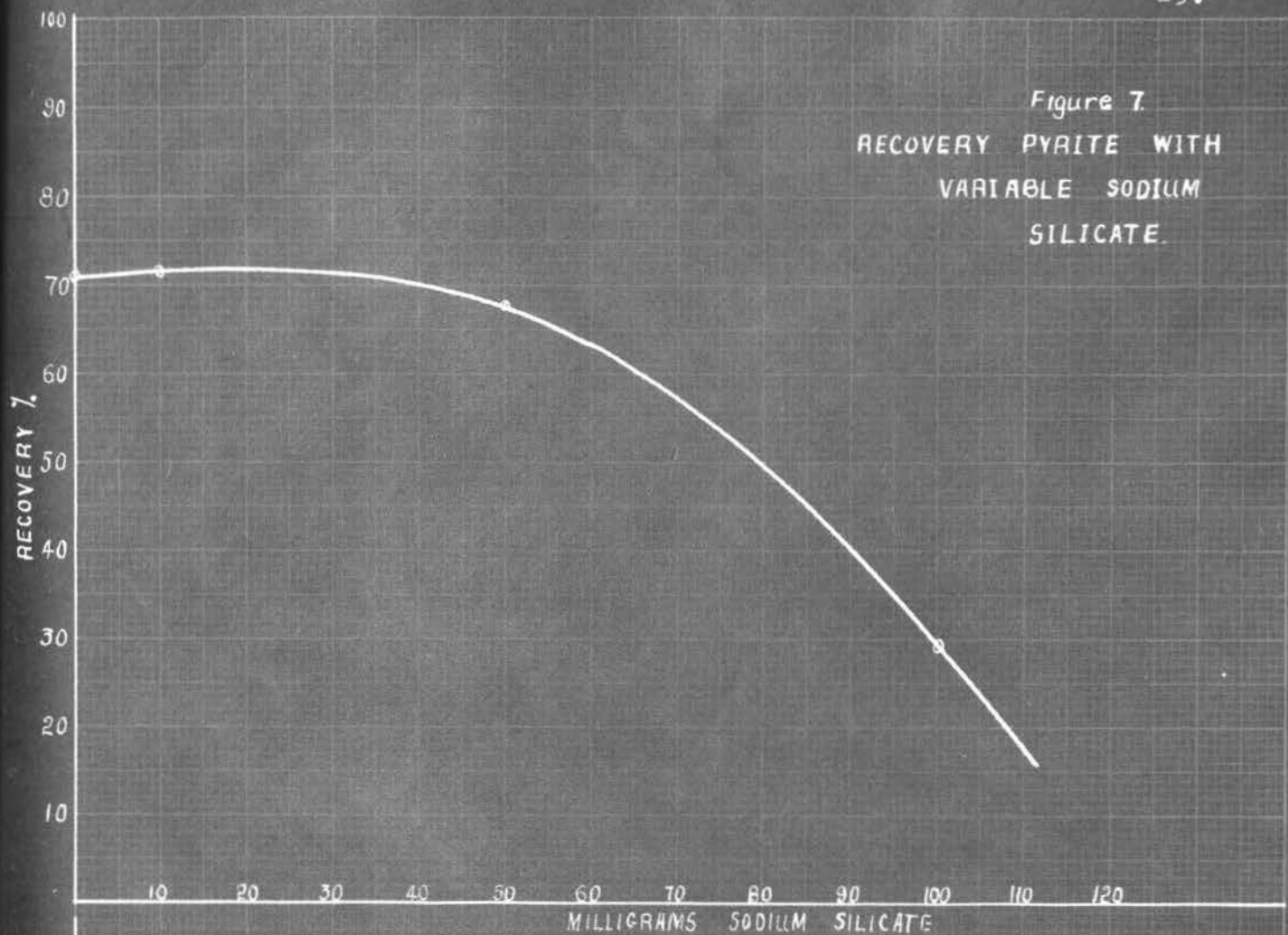


Figure 8.
RECOVERY QUARTZ WITH
VARIABLE SODIUM
SILICATE.

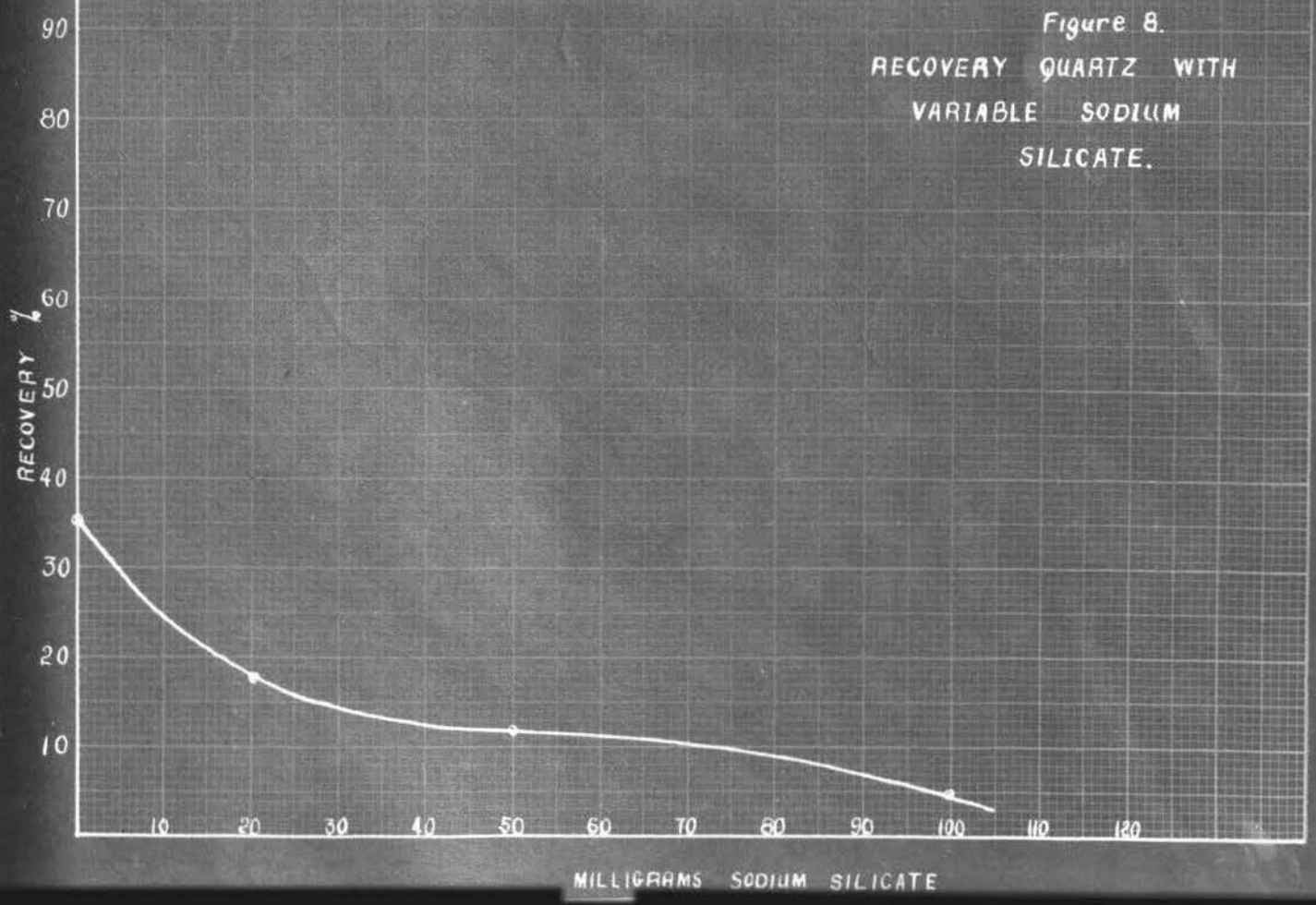


Table 9.

Flotation of Quartz and Pyrite with Variable Sodium Chloride.

| Sodium Chloride | pH Initial | pH Final | Weight Concns. | % Insol. | Weight Tails. | % Insol. | Recoveries % Pyrite | Recoveries % Quartz |
|-----------------|------------|----------|----------------|----------|---------------|----------|---------------------|---------------------|
| Nil | 8.0 | 8.2 | 51.8 | 34.2 | 45.8 | 69.3 | 70.8 | 35.7 |
| 100mg, | 8.1 | 7.5 | 41.0 | 27.2 | 52.5 | 71.1 | 66.2 | 23.0 |
| 200mg. | 8.0 | 8.0 | 44.3 | 25.7 | 52.6 | 73.0 | 70.0 | 22.9 |
| 400mg. | 8.0 | 8.0 | 30.7 | 20.4 | 67.4 | 66.4 | 52.0 | 12.3 |
| 800mg. | 8.0 | 8.1 | 9.7 | 18.4 | 79.5 | 58.5 | 19.4 | 3.69 |
| 1000mg. | 8.2 | 8.4 | 7.5 | 39.8 | 91.0 | 51.0 | 10.1 | 5.06 |

Plots of the recoveries of pyrite and quartz respectively are shown in figures 9 and 10.

It will be seen that there is considerable depression, particularly of quartz, at high concentrations of sodium chloride.

(iv) Variable sodium sulphate. Samples of 50 grams each of -100 + 150 mesh quartz and -150 + 200 mesh pyrite were prepared as in tests 'c' above. Reagent addition, conditions of flotation, and results are given in Table 10, sodium oleate and terpineol being maintained constant at 10mg. and 41.26mg. respectively.

Table 10.

Flotation of Quartz and Pyrite with Variable Sodium Sulphate.

| Sodium Sulphate | pH Initial | pH Final | Weight Concns. | % Insol. | Weight Tails. | % Insol. | Recoveries % Pyrite | Recoveries % Quartz |
|-----------------|------------|----------|----------------|----------|---------------|----------|---------------------|---------------------|
| Nil | 8.0 | 8.2 | 51.8 | 34.2 | 45.8 | 69.3 | 70.8 | 35.7 |
| 100mg. | 8.0 | 8.5 | 13.5 | 43.65 | 86.5 | 52.24 | 15.55 | 11.6 |
| 500mg. | 7.9 | 8.0 | 13.5 | 25.66 | 74.4 | 59.04 | 24.9 | 7.29 |
| 1000mg. | 8.0 | 8.5 | 7.3 | 42.75 | 91.4 | 52.0 | 8.69 | 5.35 |

Plots of the recoveries of quartz and pyrite respectively

Figure 9.
RECOVERY PYRITE WITH
VARIABLE SODIUM
CHLORIDE.

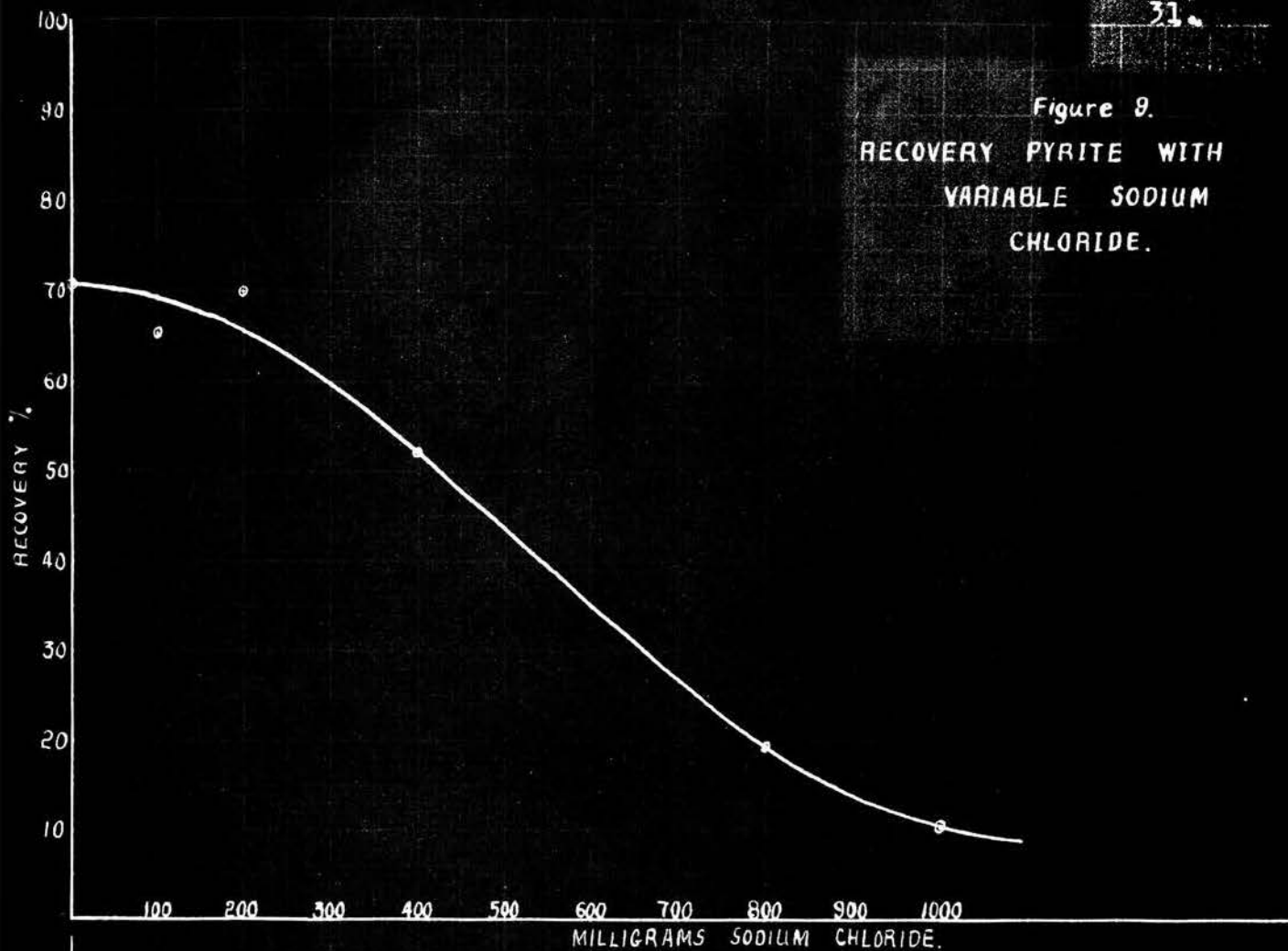
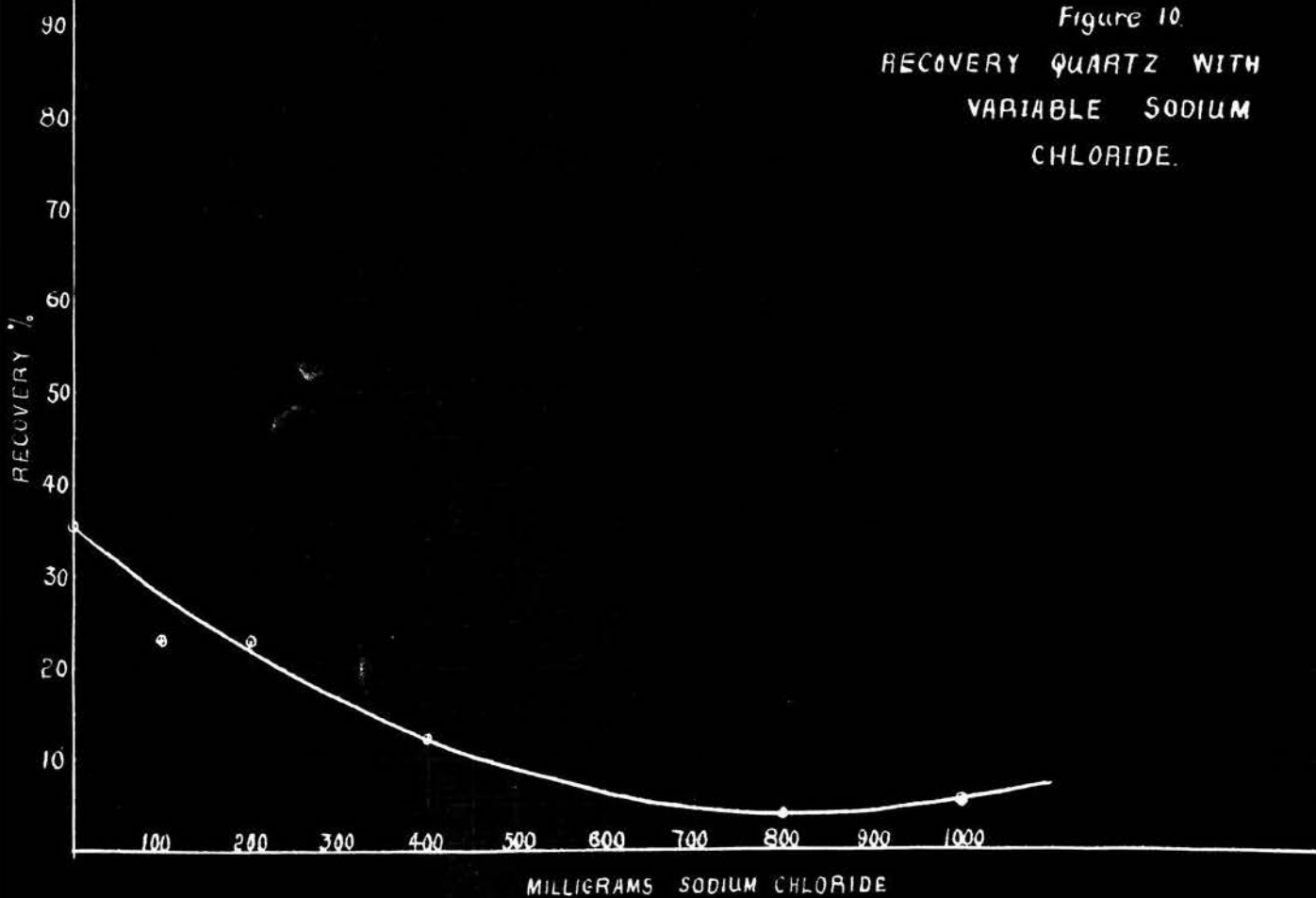


Figure 10.
RECOVERY QUARTZ WITH
VARIABLE SODIUM
CHLORIDE.



are shown in Figures 12 and 11.

It will be seen that depression of both quartz and pyrite follows the same general trend as that shown by sodium chloride indicating that the common sodium ion has a depressant effect on these minerals.

f, Calculation of available sodium ion in solutions of sodium chloride, sodium silicate, and sodium sulphate.

Sodium Silicate:

From analysis sodium content = 46.6%.

Sodium chloride:

From molecular weight sodium content = $\frac{23}{58.5} \times \frac{100}{1}$
= 39.3%.

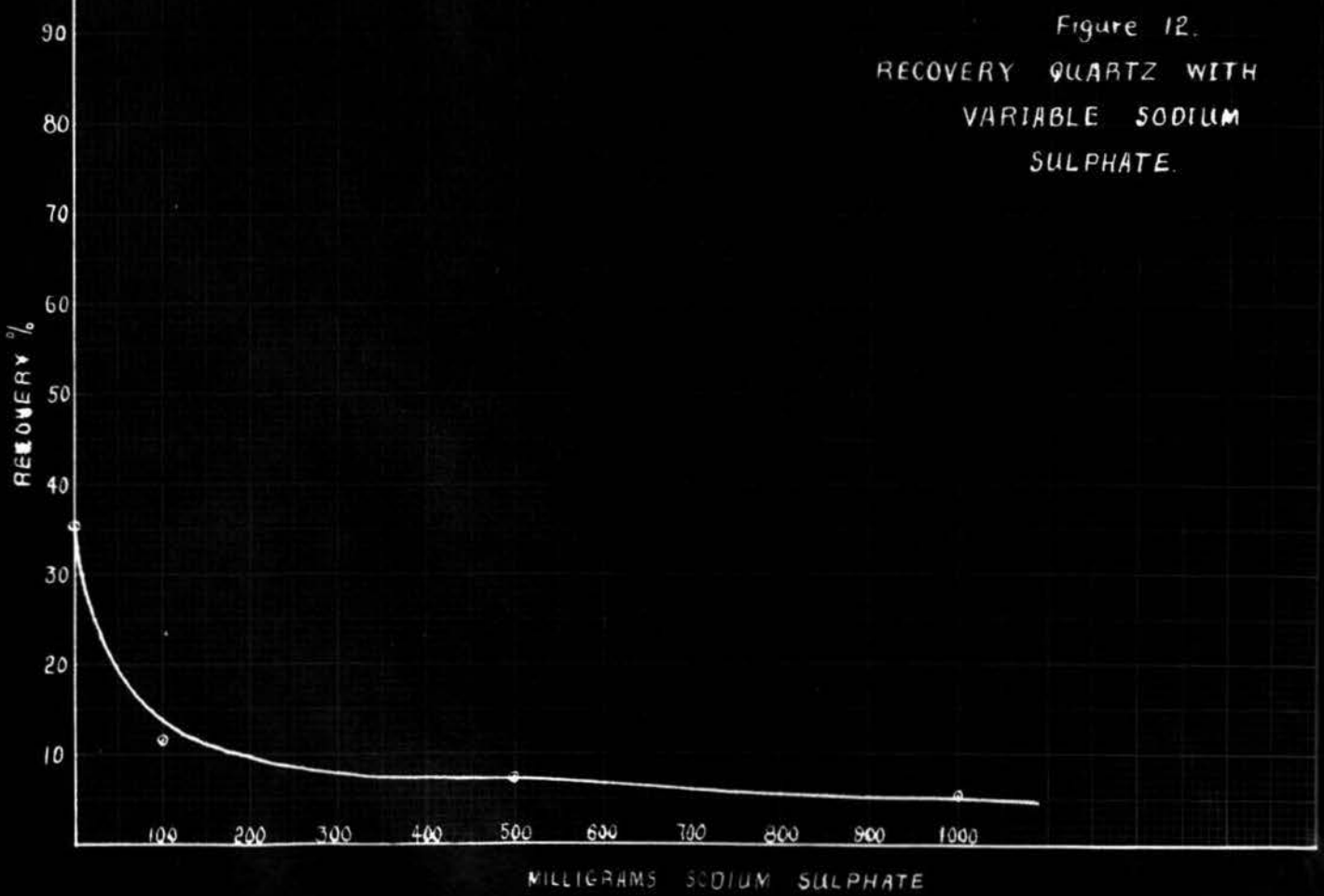
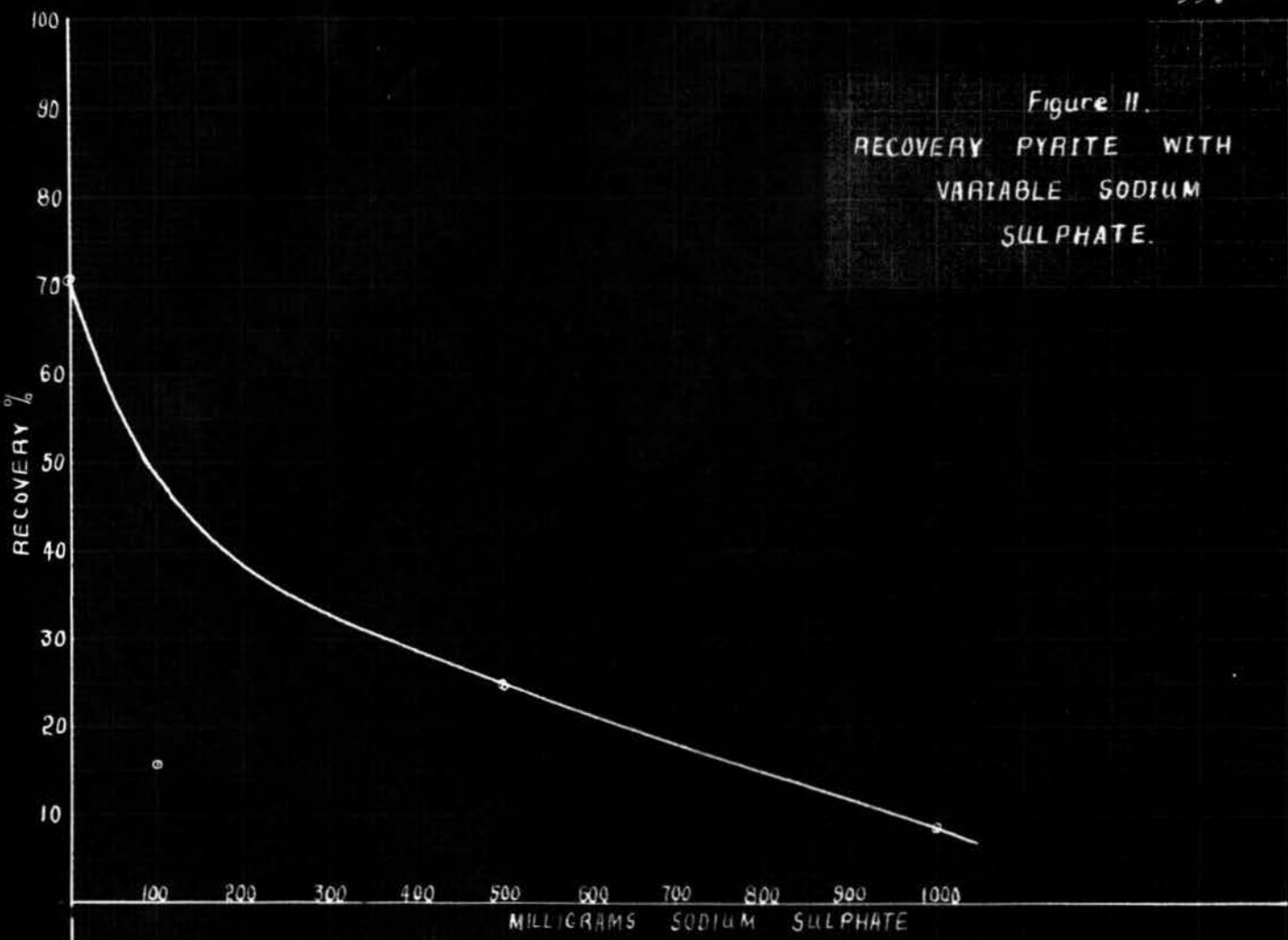
Sodium sulphate:

From molecular weight sodium content = $\frac{46}{142} \times \frac{100}{1}$
= 37.4%.

A comparison of figures 8, 10, and 12 shows that the amount of sodium silicate, sodium chloride or sodium sulphate required to lower the recovery of quartz to 5% is 100, 650, and 700mg. respectively. It will be seen that the amount of sodium ion present in each of these cases is 46.6, 256, and 263mg. respectively. Thus it will be seen that to obtain an equivalent depression using sodium chloride or sodium sulphate it is necessary to add sufficient of these salts to give a sodium ion concentration approximately six times that necessary using sodium silicate.

CONCLUSIONS.

From the experiments conducted using terpeneol both



alone and with potassium ethyl xanthate as collector with quartz it would appear:

a. That contamination of concentrates as a result of the mechanical entrapment of gangue in either a mineralised or a non-mineralised froth is not important since very little contamination resulted even though the flotation conditions were such as would be expected to produce a maximum of mechanical entrapment, the experiments being conducted under conditions producing a high degree of flocculation in the pulp.

b. that the influence of ferric chloride on the flotation of quartz from a quartz-pyrite mixture using potassium ethyl xanthate as collector is negligible.

From the experiments conducted using sodium oleate as collector it would appear:

a. That the influence of the pH of the pulp on the flotation of both quartz and pyrite is considerable, making reproducible recoveries difficult to obtain owing to the difficulty of preventing small variations in the pH of the pulp during flotation.

b. That the addition of ferric chloride to the pulp produces considerable depression of both pyrite and quartz probably due to the consumption of the available sodium oleate by the ferric chloride.

c. That the addition of sodium silicate, sodium chloride, or sodium sulphate to the pulp serves to depress both pyrite and quartz, the degree of depression being greater in the case of quartz. It is probable that the common sodium ion of these reagents plays a significant part in the depression of these minerals.

d. That the concentration of sodium ion required to effect a given depression of quartz is considerably greater when added as sodium chloride or sodium sulphate than when added as sodium silicate, being approximately six times

greater in the former case than in the latter to lower the recovery of quartz to 5%.

e. That in the case of sodium silicate sodium ion is not the only factor controlling depression, the effect of silicate ion in repressing ionisation of the quartz surface and of producing a high zeta potential on the quartz particles by the absorption of silicate micelles probably also being appreciable.

f. That the depressant effect of sodium chloride and sodium sulphate is not due to the chloride and sulphate ions respectively since an almost identical concentration of sodium ion is required to produce the same decrease in quartz recovery in both cases. The possibility of chloride and sulphate having almost identical depressant effects should not be overlooked however.

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VITA.

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