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RECOVERY OF GOLD FROM ARSENICAL

GOLD ORES.

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in part fulfillment of the requirements  
of the Degree of Master of Engineering.

- Department of Mining and Metallurgy  
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RECOVERY OF GOLD  
FROM ARSENICAL GOLD  
ORES

FOREWORD

The experimental work which comprises the subject matter of this thesis was carried out in the Metallurgical and Ore Dressing Laboratories at McGill University. A flotation concentrate from the Beattie Mine, received at the University in the fall of 1936 was used. At the time the treatment of the ore at this property consisted of direct cyanidation followed by flotation, the flotation concentrate being shipped by rail and water to the Tacoma Smelter of the American Refining and Smelting Co. Ltd.

Since the investigation concerns itself with the extraction of gold from arsenical ores by the cyanide process, a short history of the development of the cyanide process is included, as well as a summary of previous work treating arsenic ores. A review of the modern concept of the mechanism of gold solution is also included.

The work was under the direction of Professor J.U. MacEwan, Birks Professor of Metallurgy to whom thanks are due for his advice and criticism in the experimental work, and in the preparation of this thesis. The writer also wishes to acknowledge his indebtedness to Professor J.W. Bell for his many suggestions and permission to use the equipment of the Ore Dressing Laboratory.

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DEVELOPMENT OF CYANIDE PROCESS.

The extraction of gold and silver by the use of cyanide solutions began from suggestions and laboratory experiments which conveyed to various minds the possibility of devising a process for dissolving precious metals from ores. That gold was soluble in cyanide solutions to which air was accessible was known for many years before any practical process was developed. Hagen (1) in 1805 is reported to have made the statement that gold is dissolved, not only by free chlorine and aqua regia, but by a solution of prussiate of potash. Glennell in his Cyanide Handbook (2) attributes to Hagen the earliest statement of the solubility of metallic gold in cyanide solutions, although he admits that no confirmation of the reference is available.

No practical use however seems to have been made from this discovery until the middle of the century, although some interesting investigations were made. In 1843, Prince Pierre Bagration (3) published the first scientific literature on the subject. At the close of his paper he

states that "it is certain that in future cyanides of potassium must be reckoned among the number of solvents of gold." Glassford and Napier (4) in 1844 published a paper giving considerable information about the preparation and properties of the cyanides of gold and silver. The paper also contains the earliest reference to the volumetric method of estimating the free cyanide present in solutions, by means of silver nitrate. Elsner (5) in 1846 showed that oxygen played an important part in the solution of gold, silver and other metals in cyanide solutions. He formulated no equation although the accepted expression of this reaction goes by his name. Faraday (6) in 1856 made use of a cyanide solution to produce thin films of gold.

The earliest reference to the effectiveness of cyanide solutions in dissolving gold from ores to be found in a paragraph of a letter written by Wurtz (7) in 1866. This reference cannot however be considered as an anticipation of the cyanide process, since Wurtz only mentioned the phenomenon as a drawback to the use of potassium cyanide as an aid to amalgamation. The following year, J.H. Rae (8) obtained



a patent in the U. S. A. for his process entitled an "Improved Method of Treating Auriferous and Argentiferous Ores." The process made use of an electric current. The current was passed through a solution of potassium cyanide in which the crushed ore was being agitated. This current was believed to facilitate the solution of the gold and silver and at the same time to precipitate the dissolved metals on cathodic surfaces of copper. Between the granting of this patent in 1867 and 1885, many other patents were applied for. Clark(9), Faucett (10) and Sanders (11) obtained patents in the United States all of them involving the use of cyanide or some cyanogen compound in the treatment of gold and silver ores. It is of interest to note that in these patents the use of the chemical is suggested as an aid to amalgamation and not as a solvent for gold. Also during this period many investigators such as Hahn (12) in 1870, and Skey (3) in 1875 published their findings on the dissolution of gold in ores by the use of cyanide. Hahn describes experiments showing the solubility of silver sulphide in cyanide solutions, and pointed out that gold present in sulphides in ores was soluble in cyanide solution. He mentions the Union Mine in California where this knowledge was being used to advantage. Skey made the observation that the action of potassium cyanide as used at the time in connection with amalgamation occasioned a loss of gold and silver

as these metals were both soluble in the solution.

In 1885 Simpson (14) applied for a patent in the United States for a process of separating gold, silver and copper from their ores. He made use of a solution of potassium cyanide and carbonate of ammonia. Almost at the same time Endlich, Muhlenberger (15) and Louis Janin Jr. (16), metallurgists of the time, began to interest themselves in the cyanide treatment of gold ores. In their writings however they speak in a disparaging way of the prospects of solutions of cyanide as a commercially successful solvent of gold and silver. It may be concluded from the remarks of Janin that the practical difficulties in the way of the application of solutions of cyanide salts as a solvent had not been overcome by 1888, when MacArthur and Forest (17) obtained their patent.

The essential points in which this patent differed from those of previous patents are enumerated in Clennell's Cyanide Handbook (2) as follows,

"1. The solutions of the gold and silver is to be effected by means of a liquid to which a cyanide alone is added, without the aid of an electric current or other chemicals.

2. The patentess emphasize the use of dilute in preference to strong solutions for accomplishing their purpose.

3. Certain definite relations are specified between the quantities of ore values in gold and silver, and strength and quantity of solution, which, however, were not adhered to in practice and cannot be regarded as being essential to the process.

4. Cyanogen gas is mentioned as one of the solvents claimed."

To MacArthur and Forrest is due not only the distinction of being the first to prove the worth of the use of cyanide solutions by a practical demonstration, but it is also to their credit that they were the first to recommend the use of alkalis, such as potash or lime, for neutralizing the ore previous to cyanide treatment. They also recognized the value of zinc as a precipitant, and were pioneers in its use in a finely divided state. The modern cyanide process dates from the successful operation on a commercial scale of a cyanide mill at Karangahake, New Zealand in 1889.

Arrangements were now made in the chief mining countries to introduce the process on a practical basis, and many mills were erected in South Africa, New Zealand and the United States. But the chemistry of the process was not well understood and as a result many of these attempts were unsuccessful. It was not until investigators the world over turned



their attention to a study of <sup>the</sup> process that the early difficulties were overcome. In 1896 George A. Packard (18) wrote of the application of the process in the United States. "In 1892 the Livingston Mill, Colorado was running a few small agitators; the Mercur Mill in Utah was said to be a success, but was 'closed down to increase capacity.' Mills had been or were being erected in Arizona; on the Comstock lode; and in South Dakota. Some of these never started, and several were shut down or remodelled for other processes. Altogether the outlook was anything but favourable." But he goes on to say, "Since then the improvements of the process both chemically and mechanically has placed it among the recognized successful methods of ore treatment."

And in the same year H.F. Furman (19) described a series of laboratory tests to be used in connection with the extraction of gold from ores by the cyanide process. In his article he declares that, "It is the opinion of the writer that, had the following simple tests been better understood many of the failures would not have occurred, and we should probably have a larger number of successful plants in operation."

By this time so much interest had been aroused throughout the mining world in the cyanide process that many chemists and metallurgists turned their attention to a study of the difficulties encountered. In a very few years, by 1910

or thereabouts most of the mechanical difficulties had been solved and the process took its place as the most important of all known methods of extracting gold.

TREATMENT OF ARSENICAL GOLD ORES.

As far as the treatment of arsenical gold ores is concerned one of the first properties to attempt the application of the cyanide process was the Ouro Prito Gold Mine in Brazil (20). At this property the chlorination process was used successfully until 1907, when it was replaced by the cyanide process. In 1910 Kendall (21) reports their treatment as consisting of concentration, washing with lime and followed by the use of cyanide.

A pamphlet by Leaver and Woolf (22) published in 1926 by the United States Bureau of Mines lists four other properties which at the time were successfully treating arsenical gold ores by the cyanide process. These were,

The Jardine Mining Co. at Jardine, Montana, U.S.A., (23) where 83 per cent of the gold was extracted by a process which consisted of amalgamation, gravity concentration, fine grinding and flotation. The flotation concentrate was then roasted; the calcine reground and then cyanided.

The Hedley Gold Mining Company at Hedley, B.C. (24) Canada where 88 per cent of the gold in an arsenical gold ore was recovered by amalgamation, cyanidation, and the sale of an arsenic-gold concentrate.



At La Belliere, France in 1910 an ore containing auriferous mispickel was treated by tube milling followed by concentration, the concentrate being roasted and cyanided (24).

The Cam and Motor Mine in Rhodesia (25) after a year of experimentation evolved a treatment which recovered 85 per cent of the gold in an arsenical gold ore containing 1.19 per cent antimony.

The pamphlet by Leaver and Woolf gives the results of a study of the cyanide extraction of gold and silver associated with arsenic and antimony in ores. This publication together with a later report by Woolf and Jackson (26) are the only published investigations on the subject that the writer has been able to find. Leaver and Woolf note that,

(a) Roasting at a high temperature seemed to lock up more gold than a low temperature roast. They studied the extraction of gold in an artificially prepared iron-arsenic sulphide and found that at a higher temperature of  $650^{\circ}\text{C}$  22 per cent of the gold was locked up. A low temperature roast, about  $450^{\circ}\text{C}$ , resulted in a loss of only six per cent of the gold.

(2) They also found that the addition of lime before roasting seemed favorable for the extraction of gold by

cyanide solution, that is, the calcium arsenate retains less gold than the ferric arsenate.

The later work by Woolf and Jackson was published in 1938 as a section of Report of Investigation No. 3425 of the U. S. Bureau of Mines. It deals with the treatment of arsenical gold ores only. These ores are divided into two main divisions, those that require roasting, and those that do not require roasting. The second group is further subdivided into four.

(1) Ores amenable to cyanidation after a low temperature roast, that is, a temperature not exceeding 550° C.

(2) Ores that require roasting at a temperature above 550° C to produce a calcine suitable for cyanidation.

(3) Ores the calcines from which require an acid treatment before cyaniding.

(4) Ores which because of their high lime content cannot be given a sulphating type of roast and cannot be acid treated after roasting. In case such ores do not respond to a straight oxidizing roast they may be treated by flotation to remove parts of the refractory material, after which the flotation tailing may be roasted and cyanided.

In general Woolf and Jackson continue the work begun in 1926 by Woolf and Leaver. They advance a considerable amount of interesting information, noting that ores which are

apparently similar require very different treatment during roasting. They suggest that this phenomenon may be due to the gold bearing arsenical sulphides having different crystalline structures. A structure which is exceedingly dense would require a higher roasting temperature to decompose the sulphides and liberate the gold.

In addition to the above there are available the reports of investigation of the Canadian Department of Mines. From 1926 to 1937 twenty-five arsenical gold ores were studied at the ore dressing laboratories of the Department. Various treatments were recommended and recoveries ranged from 55 per cent to 95 per cent of the gold content. In connection with these investigations two points are worthy of mention. One is that in studying the ore of Whitewater Mine at Taku River, B.C. (27), it was noticed that roasting at a comparatively high temperature of  $750^{\circ}$  C locked up more gold than a low temperature roast. This is in agreement with the findings of Leaver and Woolf as mentioned earlier in this section.

Secondly in investigating the roasting of a flotation concentrate from the Beattie Mine, Duparquet, Quebec, (28) the report states that after roasting, a long period of grinding with lime appeared to improve the extraction.

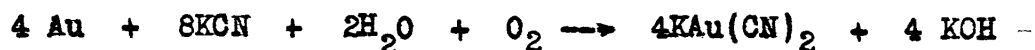


The only additional articles which have appeared on the treatment of refractory gold ores are papers which were presented before the various mining and metallurgical Societies by the executives of various companies, describing the method of ore treatment at their plant.

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THE MECHANISM OF GOLD SOLUTION.

There is no doubt in anyone's mind to-day as to the validity of Elsner's equation



in representing the reaction of the solution of gold in cyanide. But when the equation first appeared in Watt's Dictionary of Chemistry soon after 1846, and for many years after, many chemists and metallurgists believed it to be incorrect.

One of the first men to criticise the equation was J.S. MacArthur. In 1890 (29) he said that he "never could find that the presence of oxygen was necessary either to dissolve gold by itself or from ores by cyanide." MacArthur thought it more probable that hydrogen was evolved according to an equation later expressed in an independent investigation by L. Janin (30) as



MacArthur gave no proof of his contentions.

Five years after this announcement J.S. MacLaurin (31) published the results of a careful investigation into the role of oxygen in dissolving pure gold foil in a solution of

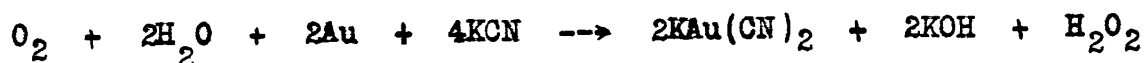
potassium cyanide and concluded that oxygen was necessary and that no gold dissolved in its absence. At the same time he advanced the first discussion of a possible mechanism for the role of oxygen in the reaction. He suggested three hypotheses giving the following as the most probable; that the oxygen and the cyanide ion act simultaneously on the gold in bringing about its solution. His explanation was that the affinity of gold for cyanide ion, and oxygen for potassium ion, was greater than that of potassium ion for cyanide ion. In essence this is the modern concept, although the modern electrochemical explanation expresses it in a simpler and clearer form.

The following year, 1896, S.B. Christie (32) criticised MacArthur very sharply for his assertions. Christie quoted from MacLaurin to prove the necessity of oxygen to bring about the solution of gold. He also declared that a personal intensive investigation had convinced him of the role played by oxygen and completely confirmed Elsner's equation. He expressed the belief that gold will go into solution only if free cyanogen is produced. The mechanism of solution is described as follows, "The affinity of the potassium for oxygen and water, combined with the affinity of the cyanogen for gold and cyanide of potassium, leads to the formation of potassium aurocyanide and caustic potash."

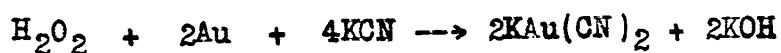


Clennel and Butters (33) in 1892 accepted Elsner's equation. They showed that the amount of cyanide used in plants operating at the time was considerably in excess of the amount required, and explained the additional consumption as being due to the destructive action of acidic material present.

Bodlaender (34) carried out experiments which indicated that Elsner's equation was correct. He dissolved gold readily in aerated solutions and showed that hydrogen peroxide is formed when gold dissolves in cyanide. He represents the reaction as follows,



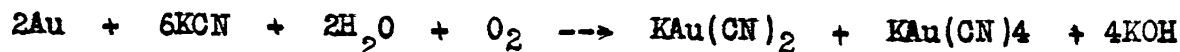
The hydrogen peroxide then reacts with more gold causing the solution of two more atoms according to the following equation



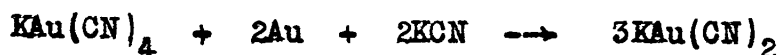
The two equations combined give Elsner's equation. Bodlaender carried out quantitative experiments and was able to detect 72.3 per cent of the hydrogen peroxide theoretically required by the first reaction.

In 1897 Bethel floated gold leaves on a cyanide solution and excluded oxygen by a continual passage of a stream

of hydrogen, when no gold dissolved. The following year writing in the South African Mining Journal (35) he suggested that  $\text{KAu}(\text{CN})_4$  was formed as an intermediate product. The equations would then be



this is followed by



The combination of the two equations giving the Elsner equation.

In 1900 S.B. Christie (36) presented the electrochemical theory of the solution of gold. The theory is based on Nernst's Theory of Solution Pressure and the Dissociation Theory as advanced by Arrhenius. Until the work on solutions by these men was generally accepted, and the theories advanced by them to explain observed phenomena were recognized, no satisfactory explanation of the solution of gold in cyanide solution could be advanced. Christie's article is the first attempt at a correlation of these theories and the first presentation of the modern concept of the problem. His explanation is similar to the one given at the close of this section.

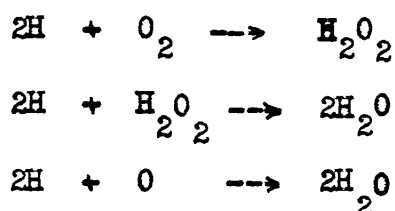
About this time, 1903, Julian and Smart's book Cyaniding Gold and Silver Ores (37) first appeared. In it they advanced the theory that the solution of gold was due to the action of a galvanic couple. To bring about solution of the metal there was needed a difference of potential between two points on its surface, and secondly, these two points had to be in electrical contact in the same solution. A similar couple would be set up if the gold was placed in the solution in contact with a metal, electro-negative to it in the solvent.

Julian and Smart declared that "For the continued dissolution of gold and silver at ordinary temperature and pressures, oxygen or an oxidizing agent is desirable if not absolutely essential." But they continue the oxygen, "has no direct action in the dissolution of gold."

They give the mechanism as being similar to the following. When the gold is present in a cyanide solution in contact with a metal which is electro-negative to it in the solvent, an electric current is set up, gold dissolves and hydrogen is deposited on the cathodic metal. From this we may surmise that Julian and Smart probably agreed with the following as an expression of the reaction.



The hydrogen generated tends to polarize the cathode and results in a diminution in the current strength. When the cathode is completely polarized, the current stops and solution stops. The effect of air or oxygen, or an oxidizing agent is to saturate the solution surrounding the cathode and induces the occluded hydrogen to become ionized forming hydrogen peroxide and water, as well as water directly

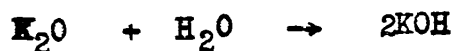
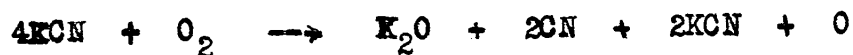


The above mechanism is plausible, but it only permits the solution of gold in the presence of, and in contact with a metal which is electro-negative to it. It has been shown by many investigators that in the presence of oxygen, pure gold foil will dissolve in cyanide solutions. The Julian and Smart mechanism offers no explanation for this phenomenon.

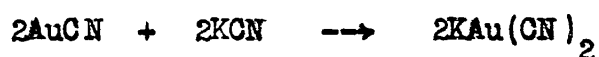
From 1903 to 1918 the literature contains three references to the solution of gold. In 1904 Engler and Weissberg(38)

and in 1913 Reichenstein (39) expressed the belief that an intermediate product, gold peroxide, is formed. In 1917 Watts and Whipple (40) declared that the access of air is essential to the solution of gold in the potassium cyanide process, being attended by the evolution of hydrogen.

In 1918 T.H. Crowe (41) delivered a paper at the Colorado meeting of the A.I.M.E. He described qualitative experiments under vacuum which showed that if the oxygen is removed no gold will dissolve. The following year Keith (42) published an article in the Engineering and Mining Journal entitled Effect of Air in Cyanidation. He states, "In analysing the reactions one finds that gold in cyanide solutions has a higher affinity for cyanogen than has potassium but it cannot exercise that affinity unless some element is present in the solution to combine with the potassium to form its oxide  $K_2O$ . To complete the reaction and fulfill the equation the oxide combines with water to form  $2KOH$ . The gold cyanide, insoluble in water alone, dissolves in the solution of  $2KCN$  to form  $2KAu(CN)_2$ ." The reactions involved would then be as follows.







From 1919 to 1934 papers appeared by White (43) and by Hay (44) but no new ideas were advanced. As late as 1934 then, although it was generally agreed that oxygen was essential for the solution of gold and the Elsner equation was generally accepted. No one had disproven the theory that hydrogen was evolved during the solution. In 1934 Barsky, Swainson and Hedley (45) calculated the free energy changes for various hypothetical reactions and showed that the reaction involving hydrogen as first proposed by MacArthur and first expressed by Jenin was impossible from the point of view of the energy changes involved. They also showed that from the free energy changes involved the two stages involving the formation and decomposition as first postulated by Bodlaender could occur readily. In 1935 Holmes (46) carried out a very careful study of the reactions involved in the solution of gold and silver in cyanide. His results showed that Elsner's equation is correct, and he suggested that the reaction proceeds in two steps as follows:



which summarized, gives



Holmes also proved that  $\text{KAu}(\text{CN})_4$  does not form as Bettel suggested in 1897. In his work he gives the electro-chemical explanation as first advanced by Christie in 1900. The theory of the solution of gold is concerned with Nernst's Theory of Solution Pressure and the Arrhenius Dissociation Theory.

#### Dissociation Theory.

The Dissociation Theory is so well known and so widely accepted to-day that no elaborate account of it will be given here. Stated simply it says that all substances will, in solution, dissociate into electrically charged particles called ions. The amount of dissociation varies with different substances. Considering a solution of potassium cyanide we can think of it as consisting of potassium and cyanide ions as well as undissociated ions of potassium cyanide. We can also think of oxygen dissolved in the solution as being present in the ionic state.

Nernst Theory of Solution Pressure.

Nernst (47) postulated a property for all metals called solution pressure, or 'solution tension' in virtue of which they tend to pass into solution as positive ions. When a metal is dipped into pure water a number of positive ions pass into the water under the stress of the solution pressure of the metal, leaving the metal with a negative charge. A difference of potential therefore exists between the metal and the liquid. Since the ions carry a comparatively large charge they do not move far away from the oppositely charged metal. The attraction of the opposite charge prevents the further expulsion of positive ions from the metal and a state is established, with a very definite potential difference, when only a very few ions of the metal have entered the liquid.

In developing the theory Nernst assumed that the tendency for the metals to ionize was opposed by the reverse tendency of the ions in the solution, as a consequence of their osmotic pressure, to leave the solution and deposit on the solid metal. Considering a metal dipping into a solution of its ions, the relative magnitudes of the solution pressure and the osmotic pressure will determine which tendency is to predominate. Three cases are possible.

(1) The solution pressure of the metal is greater than the osmotic pressure of the ions of the metal. The tendency for ions to leave the metal will predominate and as a result the metal is negatively charged.

(2) The solution pressure of the metal just equals the osmotic pressure. In this case no tendency for metal ions to leave the solution exists and no difference of potential exists between metal and solution.

(3) The solution pressure of the metal is less than the osmotic pressure. The tendency for ions to leave the solution now predominates. The metal acquires a positive charge and the solution is left negatively charged.

According to this view there exists a difference between the tendency of the metal to assume the ionic state and the tendency of the ions to assume the metallic state, and this difference can be expressed on a potential scale. The difference of potential is called the single potential of the metal and is a function of the effective concentration of the metal ion. Nernst showed that the potential difference between a metal and its ions depended on the logarithm of the concentration of the metal ions in the solution. He formulated the mathematical expression which is called the Nernst Equation, that,

$$E = E_0 - \frac{R T}{n F} \log_e c$$

where  $E$  is the potential of the metal, when  $C$  is the concentration in gram ions per litre,  $R$  is expressed in joules,  $T$  is degrees Kelvin and  $E_0$  is the potential difference between the metal and the solution when the concentration of the metal ions in the solution is one gram per litre.

The new concept of ionization has shown that the degree of ionization is much greater than was thought previously. Due to the work of Ghosh, Debye, Huckel (47) and others, Nernst's equation can be modified by the substitution of the logarithm of the product of the concentration and the activity coefficient of the system, for  $\log_e C$ . The product is known as the activity.

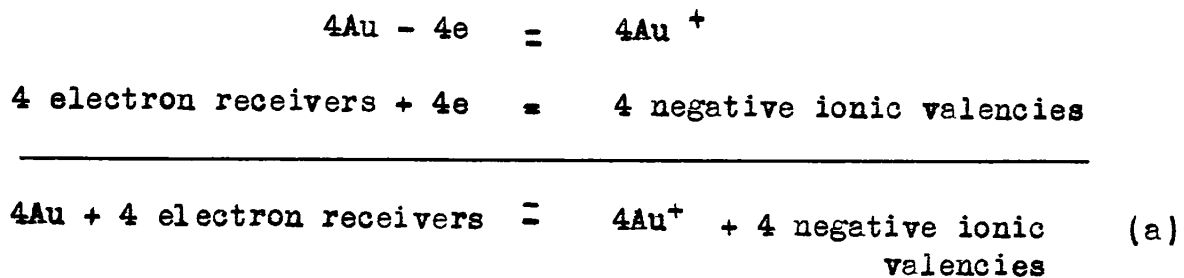
The position of gold in the electromotive series shows that it has a low electrolytic solution pressure, that is, there is only a slight tendency for gold atoms to lose their valence electron and form gold ions. The osmotic pressure due to the gold ions in the solution must therefore be maintained at a low value if solution is to be effected.

Nernst's theory, although it successfully explains many observed phenomena is but a theory, and therefore one cannot suggest a mechanism of gold solution, and state with absolute certainty that the solution of gold is according to that explanation and cannot take place according to any other. One can only advance



what seems to be a plausible explanation of known facts.

A mechanism which explains all phenomena that are known to take place and is simple in concept, is as follows. It has been firmly established that an oxidizing agent is essential for the solution of gold, and that sodium or potassium aurocyanide is formed. It is assumed that the oxidizing agent is dissolved oxygen since oxygen is the most economical and most widely used oxidizing agent in the cyanide process. The gold is oxidized by the dissolved oxygen and in this oxidized form it reacts with the alkali or alkaline cyanides to form the aurocyanide. The probable equations are,



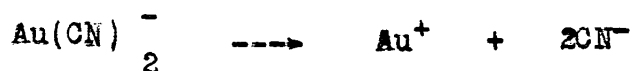
There are many investigators who have suggested that the oxidization of the gold, and the formation of gold ions takes place in more than one step. There is no intention here of dismissing this possibility as being entirely implausible. All that concerns this explanation is that gold atoms give up electrons and form gold ions, and that an oxidizing agent must be present to receive these electrons. The gold ion reacts

with cyanide in the following manner.



Equation (a) is an expression in the form of a chemical equation of the equilibrium between gold in the metallic and the ionic states. As long as the activity of the gold ions is below the value required to satisfy the equilibrium the reaction will move to the right and the solution of the gold will continue.

It has been shown that the complex aurocyanide ion is very stable (48). The ion dissociates in the following manner



and the value of the dissociation constant for the equilibrium is,  $K = \frac{[\text{Au}^+] \times [\text{CN}^-]^2}{[\text{Au}(\text{CN})_2^-]} = 10^{-28}$ . This means that the auro-

cyanide is only slightly dissociated and that the activity of gold ions is very small. The value may be further depressed by maintaining a high concentration of the cyanide ion. In modern milling practice the activity of gold ions is maintained below the value required to satisfy the equilibrium constant for equation for equation (a). The reaction proceeds to the right and goes to completion.

The very basis of the solution of gold in cyanide solution is therefore the fact that a gold-cyanide complex ion which is slightly dissociated is formed, and as a result the gold ion activity is very low. Therefore, the osmotic pressure due to the presence of the gold ions in the solution is very small. It is less than the electrolytic solution pressure of gold, and therefore the solution of gold takes place.

PURPOSE OF THE INVESTIGATOR.

All studies of arsenical gold ores, notably those carried out by Leaver, Woolf and Jackson, have been confined to the problems encountered in the roasting of these ores. Previous experience had shown that the eliminations of the arsenic left the ore more amenable to treatment by cyanidation, and as a result a complete elimination of the arsenic was aimed at. The idea was to drive the arsenic off at a low temperature and thereby prevent the formation of arsenic-iron compounds which might retain some of the gold.

In these studies however no thought was given to the effect of sending large volumes of toxic arsenic fumes off to the atmosphere. In certain regions of Canada, Quebec for example, there is legislation limiting the quantity of arsenic fumes which may be given off per day. This means that efficient collecting apparatus must be installed to condense and collect the arsenic trioxide. And having eliminated the arsenic from the stack fumes the question of its disposal must be considered. Arsenic is to-day a drug on the world market. It cannot be given away. It cannot be dumped into the streams or disposed of in any way; it can only be stored in the hope that the world demand will increase, and it can then be marketed.

It was therefore decided to first investigate the possibility of roasting the arsenical ore in such a manner as to fix the arsenic in the form of non-volatile compounds. A low temperature roast was tried. In an oxidizing atmosphere a reaction occurs between calcium and arsenic and a compound, believed to be calcium arsenate is formed. In the spring of 1937, Ramsay (49) carried out a series of experiments along these lines. He managed to retain 70 per cent of the arsenic in the calcine, and by fine grinding and treatment by cyanide, obtained an economic extraction of the gold. The experimental work of the first three months therefore had as its aim the thorough investigation of the possibilities of this process.

The second half of this work is concerned with the treatment of the raw concentrate. An effort was made to evolve a process of recovering the gold value economically without roasting, particular attention being paid to tests which would indicate the mode of occurrence of the highly refractory gold. For the greater part of this work the same concentrate was used.

In the last month of the term a shipment of fresh untreated concentrate was received and several of the later experiments were carried out on this material.



DESCRIPTION OF EQUIPMENT USED.

The equipment used may be divided into three parts, the Grinding, Roasting and Agitation Divisions. Each of these will be dealt with separately.

(a) Grinding Equipment. Grinding was carried out in the following apparatus.

(1) Porcelain Pebble Mill. The mill was used for the dry grinding of small charges of ore and calcine, and for mixing charges of lime and concentrate. A charge of four pounds of silica pebbles was used. (Fig. 1)

(2) Two Iron Pebble Mills. These mills were made in the Metallurgy laboratory and were used for grinding and mixing of small charges. They were each charged with two pounds of silica pebbles and were rotated by the use of wooden rollers. The mill dimensions are 12 inches by 4 inches diameter.

(3) Ball Mill. This mill was used for large charges up to 2000 grams in weight. The ball charge was 45 pounds of one inch forged iron balls. The shell of the mill is from a special casting free from blow holes. Its dimensions are 3 feet by 18 inches diameter. (Fig. 2).

(b) Roasting Equipment. Small scale roasts were carried out in 4 inch Battersea clay crucibles heated in a small gas fired, muffle furnace. (Fig. 3). Charges of 1,000 grams or over were roasted in a single hearth roaster heated by means of a resistance element in the cover. (Fig. 4). The charge was constantly stirred by a mechanical rabble, belt driven by a motor through a set of reduction gears. Speed of the rabbles was about five revolutions per minute. The roaster is about 16 inches in diameter and lined with alundum cement.

The temperature of the furnace was raised by connecting the resistance element in the cover to a power source of 220 volts, through an automatic cut out circuit. A nichrome-chromel thermocouple was inserted in one side of the furnace so that it rested just above the rabble arm. The thermocouple was connected to an automatic temperature recording device. The temperature recorded was that of the gases as they left the charge.

(c) Agitation Equipment. Three types of Agitators were used. These will be designated by the names Roller, Rotating and Pressure types. In connection with the first two, glass "Winchester" bottles were used. In the Roller Agitator the bottles were placed on a series of wooden Rollers which were belt driven by a motor. The bottles were not stoppered. In the Rotating type

the bottles were held in compartments carried on a shaft connected to a motor. Each compartment had a spring lid, and a rubber pad at the bottom; a pad of sponge rubber was fitted around the neck of the bottle and it was held securely radially to the shaft, by the spring lid. The shaft carried eight compartments, in four pairs, enclosed in a box, 4 feet long, 2 feet wide and  $3\frac{1}{2}$  feet deep. Speed of rotation of the shaft was 40 revolutions per minute.

The Pressure apparatus (Fig. 5) consisted of a small agitating shell similar in design to a Pachuca tank. The apparatus is completely enclosed. Air at 30 pounds per square inch gauge pressure, is fed into a centre tube which leads it down to the base of the shell. It bubbles up through the charge leaving the apparatus at the blow-off valve. A continual supply of air is therefore provided in addition to very intensive agitation.

EXPERIMENTAL WORK

PART 1

TESTS ON ARSENIC

FIXATION

A total of thirty-six tests were carried out to determine the possibility of fixing the arsenic by a low temperature roast with lime. Of these, thirty-two were on a small scale in the gas fired muffle using a thirty to fifty gram charge in a four inch crucible. The results are somewhat erratic because the temperatures were judged entirely by eye, and some difficulty was encountered in reproducing similar temperatures with different charges. For the low temperature roast an attempt was made to keep the temperature high enough to just oxidize the sulphur to sulphur dioxide. Such a roast on a small scale usually lasted from twenty to thirty minutes.

The high temperature roast was of approximately the same duration but the temperature was raised quickly to approximately 700° C. In many of the tests air was blown over the charge.

The lime used in all the tests had a CaO content of 83 per cent available lime. It was finely ground, the degree of fineness being such that 50 per cent could pass through the openings of a 200-mesh screen. The impure lime was added in amounts to give the desired CaO content, that is, a 100-gram charge containing ten per cent CaO, consisted of 12 grams of impure lime and 90 grams of concentrate.

The results of representative tests are given at the close of this section. They indicate that between 60 and 70 per cent of the arsenic can be retained in the calcine provided the concentrate is ground with about ten per cent by weight of lime, to approximately 100 per cent minus 200 mesh. Three tests in which the concentrate was mixed as received, that is, at 35 per cent minus 200 mesh, with varying amounts of lime, gave arsenic fixations from 35.3 to 42.5 per cent of the original arsenic content. Grinding the charge to about 100 per cent mesh 200 mesh, as in test No. 2, raised the percentage of arsenic retained to about 60 per cent. In several tests 70 per cent was obtained. The effect of high or low temperature roasts, or

of having an air current blowing over the charge cannot be accurately ascertained, since the estimation of the temperature of the charge was quite indefinite, and it was difficult to determine whether varying results were due to the higher roasting temperature or the air current. Addition of manganese dioxide to the concentrate, with, and without lime, and the substitution of pure limestone in the place of the lime, do not give any higher fixation of the arsenic.

The results of the large scale tests do not differ any marked manner from the tests in the small crucibles. The procedure was to grind the concentrate and the lime together in the pebble mill or in the ball mill and then charge the mixture into the roaster, cold. The current was then turned on and the charge brought up to temperature. The temperature used varied with different tests but the average was about 450° C. After two or three hours roasting the current was shut off; the mechanical rabbler was stopped; and the charge was well mixed with a spatula. The furnace was then brought up to heat again, and the roast continued until the smell of sulphur fumes was no longer obtained at the chimney.

The purpose of these large scale tests was to determine the amenability of the calcines to treatment by cyanidation. A gold extraction of 80 per cent was obtained from the



calcine of test No. 5. This was the highest value obtained, and is probably due to the fact that this roast was finished at a temperature considerably above the roasting temperatures of the other three tests. The arsenic fixation in this test however was only 59.5 per cent as compared with 68 per cent obtained in test No. 6.

Under the best of conditions therefore only about 70 per cent of the arsenic can be retained in the calcine. The elimination of the remaining 30 per cent from the furnace gases would still necessitate the use of expensive electrical precipitators, and the problem of disposing of this precipitated arsenic trioxide would still have to be faced. Also the most favourable conditions for arsenic fixation does not seem to leave the calcine in a condition in which an economic extraction of the gold is possible. It was, therefore, thought inadvisable to continue the study further.

It is quite possible that in a multiple hearth roaster operated under highly oxidizing conditions, the oxidizing effect of the furnace gases from the lower hearths would be strong enough to oxidize the volatile arsenic trioxide to the more stable arsenic pentoxide. Then with the greater part of the arsenic in a stable form as the arsenate or the pentoxide, the roast might be finished at a temperature high enough to produce a calcine which is amenable to treatment by cyanidation. This idea could not be

investigated because there was no equipment available which would serve as a multihearth roaster.

EXPERIMENTAL RESULTS.

TEST NO. 1

A 450 gram charge of the concentrate was carefully rolled and sampled. A chemical analysis showed that there was 4.56 per cent arsenic and 19.5 per cent iron. A screen analysis of the sample was as follows:

TABLE NO. 1

Screen Analysis of Ore.

<u>Mesh</u>	<u>Weight Grms.</u>	<u>Distribution.</u>
		<u>Per cent</u>
+ 100	4.46	11.30
+ 150	7.43	18.60
+ 200	14.71	36.80
- 200	13.28	<u>33.30</u>
		<u>100.00</u>

Three 100 grams charges were then mixed with varying amounts of lime and roasted at a low temperature.

TABLE NO. 2                      Arsenic Fixation in Test No. 1

<u>Test</u>	<u>Charge</u>	<u>Fe</u>	<u>As.</u>	<u>Fixation.</u>
<u>No.</u>		<u>Per Cent</u>	<u>Per cent</u>	<u>Per cent</u>
A	Ore + 5 per cent CaO	20.8	1.51	35.3
B	Ore + 10 per cent Cao	18.8	1.62	34.2
C	Ore + 15 per cent CaO	17.1	2.21	42.5
	Head	19.5	4.56	

Sample calculation of the per cent fixation is

as follows:

Per cent Fixation is

$$\frac{\text{Per cent Fe in Calcine}}{\text{Per cent Fe in Concentrate}} \times \frac{\text{Per cent As in Calcine} \times 100}{\text{Per cent As in Concentrate}}$$

$$= \frac{20.8}{19.5} \times \frac{1.51}{4.56} \times 100 = 35.3 \text{ Per cent}$$

TEST NO. 2

A 1250 gram charge of ore was mixed well, sampled, and analysed. The arsenic present was 4.75 per cent; and the iron 19.4 per cent. The screen analysis was the same as in test No. 1.

Part E of this test is as follows. A weight of ore plus ten per cent CaO were mixed to give a total charge of 200 grams, and were charged dry, into the porcelain pebble mill. Two and a half hours of grinding reduced the charge to 97.25 per cent minus 200 mesh. The ground material was divided into five samples and each was roasted in a different manner.

TABLE NO. 3      Arsenic Fixation in Part E of Test No. 2

<u>Sample No.</u>	<u>Type of Roast</u>	<u>Fe Per cent</u>	<u>As Per cent</u>	<u>Fixation Per cent</u>
Head	--	19.40	4.75	--
1	Low Temp.	18.65	2.98	60.4
2	High Temp.	18.80	2.87	58.4
3	High Temp. with air	18.55	2.99	60.1
4	High Temp. No air	18.30	2.86	56.8
5	High Temp. with air	18.15	3.64	71.5

Part G of this test was to determine the effect of adding  $\text{MnO}_2$  to the charge. A 100 gram charge of ore plus ten per cent CaO and two per cent  $\text{MnO}_2$  was ground to 99 per cent minus 200 mesh. Two-fifty gram samples were roasted as follows:

TABLE NO. 4      Arsenic Fixation in Part G of Test No. 2

<u>Sample No.</u>	<u>Type of Roast</u>	<u>Fe Per cent</u>	<u>As Per cent</u>	<u>Fixation Per cent</u>
1	Low Temp.	19.00	2.61	54.0
2	High Temp.	18.25	3.08	61.0

Part H of this test was to determine the effect of  $\text{MnO}_2$  on the roast of the concentrate without the addition of lime. The charge was ground fine and roasted. Two per cent by weight of  $\text{MnO}_2$  was added.

TABLE NO. 5      Arsenic Fixation in Part H of Test No. 2

<u>Sample No.</u>	<u>Type of Roast</u>	<u>Fe Per cent</u>	<u>As Per cent</u>	<u>Fixation Per cent</u>
1	Low Temp.	22.4	1.55	37.6
2	High Temp.	22.4	1.46	35.4

TEST NO. 3

TEST NO. 3    A one thousand gram charge of concentrate was rolled and sampled. The analysis was 4.85 per cent arsenic and 19.7 per cent iron. Individual one hundred gram samples were ground to approximately one hundred per cent minus 200 mesh and roasted in various ways. Part A, E, F and G are given here to show the effect of replacing the lime with limestone. In all these tests the limestone was added to the con-

centrate in the pebble mill and ground dry.

TABLE NO. 6                      Arsenic Fixation in Test No. 3

<u>Sample No.</u>	<u>Charge</u>	<u>Type of Roast</u>	<u>Fe Per cent</u>	<u>As Per cent</u>	<u>Fixation Per cent</u>
Head	---		19.7	4.85	
A 1	Ore	Low Temp.	23.4	0.75	18.4
A 2		High "	23.7	0.96	23.7
E 1	Ore + 5 per cent CaO +	Low "	19.4	2.04	41.4
E 2	5 per cent CaCO <sub>3</sub>	High "	19.3	2.32	46.8
F 1	Ore + 3 per cent CaO +	Low "	19.6	1.83	37.6
F 2	7 per cent CaCO <sub>3</sub>	High "	19.7	2.09	43.0
G 1	Ore + 10 per cent	Low "	19.3	1.08	21.3
G 2	CaCO <sub>3</sub>	High "	19.6	1.25	25.0

TEST NO. 5

A charge of 1350 grams concentrate and 150 grams CaO was ground for four hours in the pebble mill. It was then carefully rolled and sampled, and roasted at a temperature between 450 and 480° C. for three hours. The top of the furnace

was then lifted off and the charge well mixed with a spatula. The roasting was then continued for two hours. During this period the temperature was raised to 580° C and the roasting was finished at that temperature.

TABLE NO. 7                      Arsenic Fixation in Test No. 5

<u>Material</u>	<u>Fe</u> <u>Per cent</u>	<u>As</u> <u>Per cent</u>	<u>Fixation</u> <u>Per cent</u>
Head	16.3	4.30	
Calcine	18.7	2.24	59.5

Cyanide Test on Calcine from Roast No. 5

Two-five assay ton charges of the calcine were placed in "Winchester" bottles with cyanide solution. Bottle No. 1 was agitated on the roller agitator; bottle No.2 was stoppered securely and placed in the rotating agitator. Air was blown into the latter bottle every two hours for the first eight hours of the test.

TABLE NO. 8                      Gold Extraction and Cyanide Consumption in

Test No. 5.

Time of Agitation - - 24 hours.

Gold Content of Calcine -- 1.02 oz. per ton.

<u>Bottle No.</u>	<u>NaCN at Start lb.per ton Sol.</u>	<u>Dilution</u>	<u>NaCN Consumed lb.per ton Calc.</u>	<u>Extraction Per Cent</u>
1	1.65	2.5:1	3.92	81.5
2	2.70	4:1	4.00	83.0

TEST NO. 6

A 2000 gram charge of concentrate was ground for four hours in the ball mill with 45 lbs. of one inch iron balls. The charge was then washed through a 200-mesh screen. The oversize, about two grams of material did not carry any gold. The undersize was mixed in the porcelain mill for one hour with ten per cent of its weight of lime. The roaster was then charged, cold, with 1470 grams of this mixture and the charge was roasted for five hours at a temperature between 395 and 425° C. The charge was then well mixed with a spatula and the roasting continued for another hour at a temperature between 430 and 450° C.

When cool the charge was screened dry through a 120 mesh screen. The material passed through the openings of the screen very easily and no metallics were retained.



TABLE NO. 9                      Arsenic Fixation in Test No. 6

<u>Material</u>	<u>Fe</u> <u>Per cent</u>	<u>As</u> <u>Per cent</u>	<u>Fixation</u> <u>Per cent</u>
Charge to roaster	19.7	4.54	---
Calcine	19.7	3.02	66.5

Cyanide Tests on Calcine from Roast No. 6

The calcine was divided into two parts B-1 and B-2. Part B-1 was cyanided directly and B-2 was ground for  $1\frac{1}{2}$  hours in the pebble mill and then cyanided.

TABLE NO. 10                      Gold Extraction and Cyanide Consumption  
in Test No. 6.

Pulp dilution --- 2.5:1

Time of agitation, --- 24 hours

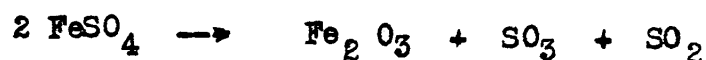
Strength of cyanide solution --- 2.75 lb. per ton of solution

Gold content of the calcine --- 1.12 oz. per ton.

<u>Material</u>	<u>Bottle</u>	<u>NaCN Consumed</u> <u>lb. per ton Calcine</u>	<u>Extraction</u> <u>Per cent</u>
B-1	1	5.72	68.9
	2	6.10	69.2
B-2	3	6.42	63.0
	4	6.26	68.0

In tests No. 5 and No. 6 the extraction was determined by assaying a known volume of solution for gold content. The assay of the solution of Bottle No. 3 is doubtful and the result should be neglected.

The cyanide consumption in Test No. 6 is about 50 per cent greater than in Test No. 5. This is probably due to the fact that the  $\text{Fe SO}_4$  formed was decomposed to a much greater extent in Test No. 5 because the roast was finished at a much higher temperature. Ferrous sulphate is known to decompose at a temperature above  $600^\circ \text{C}$  according to the following reaction.



If not decomposed it will react with sodium cyanide in the following manner.



Grinding the calcine apparently does not effect the extraction.

The results of Tests No. 4 and No. 7 will not be given in detail. They do not differ greatly from Test No. 6 except that both the arsenic fixation and the cyanide extractions are somewhat lower.

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P A R T 11

S E C T I O N A   T E S T S   O N   T H E   C O N C E N T R A T E  
R E C E I V E D   I N   1 9 3 6 .

The ore from the Beattie Property in the raw state, is highly refractory to treatment by the cyanide process. Samples of the ore and of a flotation concentrate were tested at the Ore Dressing Laboratories of the Canadian Department of Mines, Ottawa, Canada, and at the Sullivan Concentrator of the Consolidated Mining and Smelting Co. Limited at Chapman Camp, B.C. Both laboratories reported low extractions ranging from 55 to 78 per cent for various treatments. The highest extraction obtained at the Sullivan Concentrator was 78 per cent, and this value was arrived at only after subjecting the concentrate to a long grinding period and cyaniding for twenty-four hours. The particle size of the final product is estimated at 2000 mesh.

From the above it may be surmised that the gold that is readily dissolved is present as minute particles, intimately associated with the sulphides in the ore. This opinion is born out by the results of the experimental work which follows. The extractions obtained checked very closely with those obtained at the Sullivan Concentrator and at the Canadian Department of Mines Laboratory.

In the first few tests given here either the solution was saved and assayed or the final tailing was washed with hot water, dried, and then assayed. In this way the extractions were computed, and it was not possible to check with the analysis of the head sample. Starting with Test No. 15 however both the solution and pulp were saved. The solution and pulp washings were well mixed and by measuring the total volume, and assaying a known volume, the total amount of gold dissolved was calculated. Adding this value to the amount of gold left in the pulp gave the total gold content of the ore. In this way a check on the accuracy of the analytical work was obtained. Extractions as given however are based only on the tailing assay and the head assay. They differ from the extractions obtained by dividing the total gold in the solution by total gold in the ore, by the amount that the total gold content of the ore as obtained by the analysis of the solutions

and tailing, differs from the value obtained by multiplying the weight used by the analysis of the head. Extractions were expressed on this basis because it was felt that this value is more accurate and obviates any error due to mechanical loss of solution or final pulp.

In many tests the dilution is given as being approximate. This means that after grinding, the sample was charged into the bottle for agitation in cyanide solution without being dried. The moisture content of the pulp was neglected and the dilutions therefore are only approximate.

Only a few determinations of the lime or sodium hydroxide consumed are given. In most tests the effect of these alkalis was sought in a qualitative manner and therefore no quantitative determinations were made.

#### TEST NO. 8

A 200 gram charge of concentrate, in the condition in which it was received, that is, at 35 per cent minus 200 mesh was carefully rolled and sampled. Then five assay tons were agitated for twenty-four hours in cyanide solution using the roller agitator.

TABLE NO. 11      Gold Extraction in Test No. 8

<u>Head Assay</u>	<u>Dilution</u>	<u>Time of Agit.</u> <u>Hr.</u>	<u>Extraction</u> <u>Per cent</u>
1.08	2.5:1	24	58.0

TABLE NO. 12      Reagent Consumption in Test No. 8

<u>NaCN Added</u> <u>lb. per ton Sol.</u>	<u>NaCN Used</u> <u>lb. per ton Ore</u>	<u>CaO Added</u> <u>Lb. per ton Ore</u>	<u>CaO Used</u> <u>lb. per ton Ore</u>
1.94	4.14	4.93	4.93

TEST NO. 9

A charge of 1200 grams was ground to 100 per cent minus 200 mesh; filtered; dried and then sampled. Four charges of five assay tons each were then weighed up, and treated in the following way. The roller agitator was used.

TABLE NO. 13      Table Giving Treatment and Extractions  
in Test No. 9.

Pulp dilution was 2.5:1

Time of agitation was twenty-four hours.

Gold contents of concentrate was 1.03 oz. per ton.

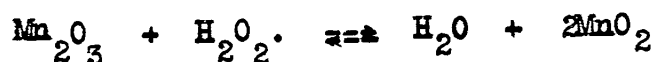
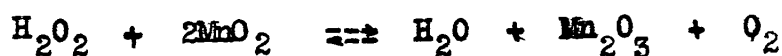
<u>Sample No.</u>	<u>Treatment</u>	<u>Extraction Per cent</u>
A	Cyanided for twenty-four hours with addition of 10 pounds of CaO per ton of ore.	68.5
B	Cyanided for twenty-four hours without addition of lime. Additional cyanide added after 12 hours of agitation.	58.4
C	Cyanided for twenty-four hours without lime. $H_2O_2$ and $MnO_2$ added every hour for first twelve hours then additional cyanide added and agitation continued.	68.7
D	Given a twenty-four hour preliminary treatment at a 2.5:1 dilution with solution containing fifty pounds of CaO per ton of ore. Cyanide then added and agitation continued for twenty-four hours.	71.9

TABLE NO. 14

Reagent Consumption in Test No. 9

<u>Bottle No.</u>	<u>NaCN Added Lb. per ton of Ore</u>	<u>NaCN Used Lb. per ton of Ore</u>	<u>CaO Added Lb. per ton of Ore</u>	<u>CaO Used Lb. per ton of Ore.</u>
A	7.07	6.75	10.0	10.00
B	11.08	9.72	--	--
C	9.33	8.62	--	--
D	7.85	2.48	50.0	--

In Test 9B and 9C the cyanide left after twelve hours agitation was 0.11 pounds NaCN per ton of solution. The  $H_2O_2$  was added as a three per cent solution by weight, the total amount added being 1.62 grams or 21.2 pounds per ton of ore. The  $MnO_2$  added was in the proportions required by the equation.



The increase in cyanide consumed in Test 9A over the amount used in Test No. 8 was probably due to the fact that additional oxidation of the minerals took place during the grinding and drying of the concentrate.

The gold extractions in Tests No. 8 and No. 9 were determined by assaying a known volume of solution.

### TEST NO. 13.

The purpose of this test was to determine the effect of grinding with lime, and also the time of agitation required for the best extraction. A charge of 960 grams of ore was mixed with enough lime of 83 per cent CaO content to provide 20.8 pounds of CaO per ton of ore, and ground to 100 per cent minus 200 mesh at 60 per cent solids.



The sample was filtered, cut into eight parts with a spatula, and each part was agitated with cyanide solutions for varying periods of time. The rotating type of agitator was used.

TABLE NO. 15      Gold Extraction and Cyanide Consumption.

in Test No. 13.

Gold Content of head, 1.03 oz. per ton

Pulp dilution, 4:1

Cyanide Strength, 3.20 per ton of solution.

<u>Sample No.</u>	<u>Time of Agit. Hours</u>	<u>NaCN Consumed lb. per Ton of Ore</u>	<u>Tailing Assay Oz. Au per ton</u>	<u>Extraction Per cent</u>
A	2	4.24	0.34	67.0
B	4	5.12	0.34	67.0
C	6	5.76	0.32	68.9
D	8	6.12	0.32	68.9
E	12	6.32	0.32	68.9
F	16	6.48	0.32	68.9
G	24	7.32	0.32	68.9
H	24	7.36	0.32	68.9

TEST NO. 14

This test was a repetition of Test No. 13 as far as the grinding and subdivision of the sample is concerned. The eight parts were charged into eight "Winchester" bottles and were treated as given in the following table, the rotating agitator being used.

TABLE NO. 16      Various Treatments in Test No. 14.

Pulp dilution, 4:1

Time of Agitation, 4 hours.

Strength of cyanide solution, 3.24 lb. per ton  
of solution.

<u>Sample No.</u>	<u>Treatment.</u>
A	Agitated for four hours
B	Agitated for two hours; then filtered and re-pulped with cyanide solution of strength 3.03 pounds per ton, and at approximately the same dilution. Agitation then continued for an additional two hours.
C	Same as B except that lime in amount to give ten pounds of CaO per ton of ore was added to the re-pulping solution.
D	H <sub>2</sub> O <sub>2</sub> was added to the solution as a 25 per cent solution in an amount equivalent to 128 pounds per ton of ore, and then charge was agitated for four hours.
E	Same as D. The filter cake was then

re-pulped with cyanide solution, of strength 3.03 pounds per ton and agitation continued for additional two hours.

- F Sample lost.
- G Identical with E except that the same amount of  $H_2O_2$  was added to the repulping solution.
- H Same as E except that  $H_2O_2$  and lime were added to the re-pulping solution in amounts equal to 128 pounds and 10 pounds per ton of ore respectively.

In the above test and in test No. 13 the final tailing was washed four times with hot water, then dried, and broken up by passing through a pulverizer with the plates wide open, and assayed for gold. The extraction was calculated from this tailing assay.

TABLE NO. 17                      Gold Extraction in Test No. 14

Gold Content of concentrate was 1.03 oz. per ton.

<u>Sample No.</u>	<u>Tailing Assay Ozs. of Au</u>	<u>Extraction Per cent</u>
A	0.34	67.0
B	0.32	68.9
C	0.32	68.9
D	0.44	57.3
E	0.42	59.3

<u>Sample No.</u>	<u>Tailing Assay Ozs. of Au</u>	<u>Extraction Per cent</u>
F	—	--
G	0.32	68.7
H	0.24	76.7

TABLE NO. 18                      Reagent Consumption in Test No. 14

<u>Sample No.</u>	<u>NaCN Used</u>			<u>CaO Used</u>		
	<u>Lb. per Ton Ore.</u>			<u>Lb. per Ton Ore.</u>		
	<u>2 hrs.</u>	<u>4 hrs.</u>	<u>Total</u>	<u>2 hrs.</u>	<u>4 hrs.</u>	<u>Total</u>
A	--	6.96	6.96	--	20.8	20.8
B	6.01	2.24	8.25	20.8	—	20.8
C	5.60	2.24	7.84	20.8	7.44	27.44
D	--	11.84	11.84	—	20.8	20.8
E	10.00	2.52	12.52	20.8	--	20.8
G	8.80	8.56	17.36	20.8	--	20.8
H	8.80	5.92	14.72	20.8	9.24	29.24

The purpose of this test was to determine whether the refractory nature of the residual gold was due to a lack of oxygen. The fact that the extraction in Tests D and E are so low almost renders the results meaningless. However, neglecting these tests or attributing their high tailing to salting or insufficient washing of the filter cake and considering

tests G and H, we see the re-pulping with added  $H_2O_2$  has little effect. The addition of lime, however raises the extraction by an appreciable amount.

The amount of reagents consumed is very high. This is due to the destructive effect of the oxidizer,  $H_2O_2$ . The action of strong oxidizers in decomposing cyanide solutions was recognized very early in the development of the cyanide process. As early as 1896 S.B. Christie (50) wrote that, "There is danger in using too strong an oxidizer or too much of it. In such a case,-----other secondary reactions likely to ensue with ores containing metallic sulphides, arsenides, and antimonides, which lead to the destruction of the cyanide,-----" There is little doubt that in this test not only was the oxidizer too strong but there definitely was too much of it. It is interesting to compare the cyanide consumption in this test with test No. 9 where a 3 per cent  $H_2O_2$  solution was used. From 50 to 100 per cent more NaCN was lost in this test.

#### TEST NO. 15

This test was a study of the effect of regrinding the cyanide tailing with additional lime. The charge to the ball mill was 480 grams of ore plus a weight of impure lime to give 20.8 pounds of CaO per ton of ore, and this charge

was ground at 60 per cent dilution to 100 per cent minus 200 mesh. The pulp was then filtered, cut into four parts, and each part was agitated in a separate bottle with cyanide solution for two hours. Then each bottle was filtered and the cake put back into the ball mill; another charge of lime equivalent to 20.8 lbs. of CaO per ton of ore was added, and grinding continued for 45 minutes at 60 per cent solid. The charge was then filtered and cyanided again in four bottles for two hours. All solutions were saved and assayed. The final pulps were washed four times with hot water, the washings being added to the solution. The pulps were then dried, broken up and well mixed.

TABLE NO. 19

Gold Extraction in Test No. 15

Pulp dilution, 4:1

Strength of cyanide solution, 2.70 and  
2.51 lb. NaCN per ton of solution.

<u>Au in pulp</u>	<u>Total Au</u>	<u>Total Au</u>	<u>Extraction</u>
<u>Oz. per ton</u>	<u>in Pulp</u>	<u>in Solution</u>	<u>Per cent</u>
	<u>Mgs.</u>	<u>Mgs.</u>	
0.23	3.79	15.80	77.7

On the basis of the above assays the calculated head is 1.14 oz. per ton and extraction 83.9 per cent.

This is not in agreement with the value of 1.03 oz. obtained by direct assay. The solution assay in the above table is however doubtful since the solution may not have been mixed well enough. On the basis of the tailing assay and the head of 1.03 oz. the extraction is 77.7 per cent. This value is probably correct. Cyanide consumption was 4.91 pounds per ton of ore.

TEST NO. 17

It was suggested that the lime be replaced with sodium hydroxide. There is the possibility that in solutions made alkaline with lime the arsenopyrite oxidizes and combines with the lime to form calcium arsenate. This compound is very insoluble and as it formed, it would tend to precipitate out of solution, and might very well coat gold particles with an insoluble film. The use of sodium hydroxide in the place of lime would bring about the formation of sodium arsenate which is soluble in water and hence no coating of the gold particles should be obtained.

The test is similar to test No. 16 except that NaOH in an amount equivalent to 18.75 pounds per ton of ore was used in place of the lime and after a two hour agitation the pulp from each of the four bottles was treated differently.

The procedure was as follows. The discharge from the ball mill was filtered and placed into four bottles. Bottles A and B were agitated for two hours and five hours respectively. Bottles C and D were agitated for five hours, the pulps were filtered and the filter cakes were then reground for one hour at 50 per cent dilution. Ten pounds of NaOH and CaO per ton of dry pulp were added to the regrind in tests C and D respectively. Agitation was then continued for another five hours.

TABLE NO. 20                      Gold Extraction in Test No. 17.

Pulp dilution, 4:1

Strength of cyanide solution, 2.92 and 3.19  
lb. NaCN per ton of solution.

Sample	Procedure	Tailing Assay <u>Oz. per ton</u>	Au in Tailing <u>Mg.</u>	Au in Solution <u>Mg.</u>	Extraction Per cent
A	2 Hours Agitation	0.23	0.945	3.070	77.7
B	5 Hours Agitation	0.23	0.945	3.520	77.7
C	5 Hours Agitation Re-grind with NaOH	0.23	0.945	2.99	77.7
D	5 Hours Agitation Re-grind with CaO	0.23	0.945	3.76	77.7



In the above test after drying the pulps, they should have been weighed. Since this weighing was omitted a weight of 120 gm. was used in computing the total gold in the tailing. To check with the head assay the total gold values obtained in all four tests was added and from this value the gold content in oz. per ton was calculated. It comes to 1.04 oz. per ton which checks with 1.03 oz. per ton, the value determined by direct assay.

TABLE NO. 21.                      Reagent Consumption in Test No. 17.

<u>Sample No.</u>	<u>NaCN Added Gm.</u>	<u>Amount Left Gm.</u>	<u>Amount Used Gm.</u>	<u>Amount Used Lb. per Ton Ore.</u>
A	0.71	0.44	0.27	4.50
B	0.71	0.27	0.44	7.34
C	1.48	0.54	0.94	15.70
D	1.48	0.47	1.01	16.85

ARSENIC REDUCTION TESTS.

The following four tests are intended to show whether the highly refractory gold is present in chemical combination with the arsenic. Gold arsenic compounds have been prepared synthetically. Alloys containing up to 25 atomic per cent

of arsenic have been prepared by fusing together gold and an alloy rich in arsenic (51, 52, 53). The freezing point curve reveals a eutectic point at 665 degrees centigrade (54) with about 46 atomic per cent of arsenic. It was therefore not entirely unreasonable, to suppose that arsenic gold compounds exist in the natural state and that the highly refractory nature of a definite percentage of the gold in some arsenical ores is due to its presence as an arsenic compound.

Many investigators have effected the reduction of arsenic compounds by nascent hydrogen in an alkaline medium. The Nipissing Mill at Cobalt (55, 56) is a very notable example. Here the nascent hydrogen generated by the action of a caustic solution on aluminum ingots was used to reduce mineral containing arsenic, antimony and silver which formed part of the ore. It was therefore decided to try this aluminum-caustic reduction treatment on the Beattie ore.

#### TEST NO. 18.

The tailing from test No. 15, which assayed 0.24 oz. of gold per ton was used in this test. A 300 gram charge was placed in the ball mill with 300 c.c. of a one per cent solution of caustic soda and 20 grams of granulated aluminum. The ball charge consisted of 21 -  $1\frac{1}{2}$  inch iron balls. The purpose of the balls was to keep the charge well agitated. The treatment

was continued for six hours; then the pulp was filtered, washed well with hot water, and agitated in a cyanide solution of 4.28 pounds NaCN per ton at a three to one dilution for twelve hours. The rotating agitator was used.

An additional five per cent of the total gold content of the concentrate was extracted, raising the overall extraction to 81.5 per cent. Cyanide consumed was 7.89 pounds per ton of ore.

TEST NO. 19.

This test was similar to the preceding one except that the reduction treatment was carried out in an open mouthed bottle on the roller agitator using three short lengths of 1/2 inch aluminum rod instead of granulated aluminum. Two bottles were used each containing 100 grams of the charge in a one per cent caustic solution at a three to one dilution. The aluminum was added to one bottle only, the second bottle being used for comparison purposes. After twelve hours treatment the pulps were filtered, washed and cyanided for twenty-four hours using the roller agitator and a cyanide solution of strength 3.33 pounds NaCN per ton, at a three to one dilution. The ore used was the composite tailing of Test No. 13, which assayed 0.32 oz. per ton .

No appreciable difference of extraction was obtained between the two bottles. An additional 4 per cent of the gold was dissolved but this was probably due to the action of the fresh cyanide solution. Cyanide consumption was 1.30 and 3.10 pounds NaCN per ton ore. The higher value was obtained in the bottle containing the aluminum.

There is little doubt that no reduction was effected in the above two tests. The gold dissolved in Test No. 18 undoubtedly is due to the additional grinding during the six hour reduction treatment and to the dissolving action of the fresh cyanide solution added. In these two tests however, relatively small amounts of aluminum were used, and there was the possibility that not enough nascent hydrogen was evolved. In the following two tests therefore twenty-one aluminum ingots of a total weight of 1770 grams were used, and the tests were carried out on the concentrate itself.

TEST NO. 20.

A charge of 575 grams of concentrate was ground to 100 per cent minus 200 mesh, and divided into four parts. Parts A and B were charged into two bottles with a one per cent caustic solution at approximately three to one dilution. To B was also added three short lengths of 1/2 inch aluminum rod.

The bottles were rotated on the roller agitator for twelve hours. Parts C and D were charged into the ball mill with a one percent caustic solution and the aluminum ingots mentioned above. The reduction treatment was continued for twelve hours.

At the completion of the reduction treatment, the pulps were filtered, washed and cyanided for twenty-four hours. Samples A and B were agitated in the roller agitator; C and D in the rotating type.

TABLE NO. 22      Gold Extraction in Test No. 20.

Gold content of head, 1.03 oz. per ton.

Pulp dilution, 3:1

Sample	Wt. of Ore Gm.	Tailing Assay Oz./Ton	Total Au in Solution Mg.	Calculated Head Oz. per Ton	Extraction Per cent
A	96	0.24	2.69	1.06	76.8
B	108	0.24	2.69	0.97	76.8
C	189	0.92	0.49	0.99	11.8
D	181	0.90	0.52	0.98	12.5

TABLE NO. 23

Reagent Consumption in Test No. 20

<u>Sample No.</u>	<u>NaCN Added Gm.</u>	<u>Amount Left Gm.</u>	<u>Amount Used Gm.</u>	<u>Amount Used Lb. per ton Ore</u>
A	0.50	0.21	0.29	6.03
B	0.50	0.15	0.35	6.49
C	0.75	0.36	0.39	4.12
D	0.75	0.36	0.39	4.30

TEST NO. 21

A charge of 700 grams of concentrate was ground for two hours in the ball mill. It was then dried, broken up and divided into parts A and B, each weighing 300 grams; 100 grams was retained as a head sample. Part A was treated with a one per cent solution of caustic soda in the ball mill for twelve hours. The mill was charged with twenty-one  $1\frac{1}{8}$ -inch iron balls. The sample was then filtered and cut into three parts. Each part was agitated in cyanide solution of strength three pounds NaCN per ton for six hours at a dilution of three to one. The roller rotating and pressure type agitators was used for parts one, two and three respectively. The blow off value of the pressure apparatus was set at 30 pounds per square inch gauge.

Sample B was treated in an identical manner except that twenty-one aluminum ingots of total weight 1770 gm.

replaced the iron balls in the ball mill.

TABLE NO. 24.                    Gold Extraction in Test No. 21.

Pulp dilution, 3:1

Time of agitation, six hours

Gold analysis of concentrate, 1.03 oz. per ton.

<u>Sample No.</u>	<u>Wt. of Ore Gm.</u>	<u>Tailing Assay Oz. per Ton</u>	<u>Total Au in Sol. Mg.</u>	<u>Calculated Head Oz. per Ton</u>	<u>Extraction Per cent</u>
A-1	104	0.30	2.57	1.02	70.9
A-2	98	0.22	2.78	1.05	78.6
A-3	98	0.22	2.67	1.05	78.6
B-1	94	0.34	2.11	1.00	67.0
B-2	121	0.55	1.85	1.00	46.0
B-3	91	0.55	1.53	1.01	46.0

TABLE NO. 25.                    Reagent Consumption in Test No. 21

<u>Sample No.</u>	<u>NaCN Added Gm.</u>	<u>Amount Left Gm.</u>	<u>Amount Used Gm.</u>	<u>Amount Used Lb. per ton</u>
A-1	0.45	0.35	0.10	1.92
A-2	0.45	0.31	0.14	2.86
A-3	0.45	0.16	0.29	5.82
B-1	0.45	0.16	0.29	6.16
B-2	0.45	0.15	0.30	4.95
B-3	0.45	0.15	0.30	6.06

TEST NO. 22

Since the above tests indicate that it is the degree of comminution which will determine the gold extraction, it was decided to run a test in which the charge was ground for varying lengths of time and then cyanided. A 1500 gram charge was ground in the ball mill for two hours at 60 per cent solid with 18 grams of impure lime; it was then filtered and  $1/4$  of it, Sample A, was cut out. The remainder of the charge was put back into the mill and ground for another two hours, when it was again filtered, and again  $1/4$  of it, Sample B, was cut out. The procedure was repeated for a third time, the final grinding period being an additional four hours. Sample C, therefore had received eight hours of grinding before it was cyanided. One half of each sample was dried and served as a head sample, the other half was agitated in the pressure apparatus.

TABLE NO. 26

Gold Extraction in Test No. 22

Pulp dilution, 2.5:1

Time of agitation, six hours

Gold content of head, 1.03 oz. per ton



Sample No.	Wt of Ore Gm.	Tailing Assay Oz. per ton	Total Au in Sol. Mg.	Calculated Head Oz. per Ton	Extraction Per cent
A	198	0.27	5.39	1.05	73.8
B	136	0.23	3.84	1.05	77.7
C	118	0.23	2.92	0.96	77.7

TABLE NO. 27.

Reagent Consumption in Test No. 22

Sample No.	NaCN Added %Gm.	Amount Left Gm.	Amount Used Gm.	Amount Used Lb. per Ton Ore
A	0.75	0.46	0.29	2.93
B	0.75	0.38	0.37	5.60
C	0.50	0.15	0.35	5.92

The fact that no additional gold was dissolved after grinding for eight hours suggests that either the last four hours of grinding did not bring about any further comminution or that the gold which was not dissolved, is present in a state in which it is insoluble in cyanide solution.

SUMMARY OF THE TESTS OF SECTION A.

In the above tests when a sample is said to be 100 per cent minus 200 mesh the degree of fineness was determined by washing the sample through a 200 mesh screen. In several

of the tests in which a small charge was used most of the sample was much finer than this screen size. This is definitely true of Test No. 17 (replacing lime with caustic in the ball mill) and explains the fact that additional grinding yielded no additional gold.

There is little doubt that in all the above tests it is the degree of comminution which determined the gold extraction. A sample ground to a degree of fineness such that 100 per cent will just pass the openings of a 200 mesh screen will yield 67 per cent of the gold. Additional grinding to an indefinite degree of fineness will raise the extraction to 78 per cent of the gold value and no higher value can be obtained no matter how prolonged the period of grinding.

The arsenic reduction tests carried out on the tailing of previous tests showed that small additional extractions of gold can be obtained by repulping with fresh cyanide solution, but otherwise the nascent hydrogen has no effect. The reduction tests on the ore itself only served to bring out this fact more clearly, and to confirm the statement that in the tests of this section it was the degree of comminution which determined the extraction. Thus the additional grinding of the iron balls in Test 20A was enough to lower the tailing to 0.22 Oz. per ton. The aluminum ingots, being much lighter could hardly have the same effect.

The very low extractions of Test Nos. 19 and 20, when the reduction treatment was tried may be explained in the following manner. It is possible that the amount of hydrogen evolved was so great that even the subsequent washing was not enough to wash it out of the ore. This nascent hydrogen probably combined with the oxygen available for gold solution and when the supply was used, the dissolving action stopped. This explanation is suggested by the fact that the roller type of agitator in which the bottle was rotated without a stopper gave improved recoveries over the revolving type.

A second possible explanation is that due to the attrition of the aluminum ingots, against the shell of the ball mill, small particles of aluminum were scraped off and mixed with the charge. The alkaline cyanide solution would react with these particles to give a constant evolution of hydrogen which again would affect the oxygen supply. In the pressure apparatus however where a continual supply of air was available it is hardly conceivable that the oxygen could all be taken out of solution. This fact should have been investigated further but due to the limited time available it was found impossible to do so.

Cyanide consumptions are somewhat erratic. The ore oxidizes very easily and the results indicate that the more handling it receives the greater is the cyanide consumed.

PART 11

SECTION B      TESTS ON THE CONCENTRATE  
RECEIVED IN 1939.

Since the tests described in Section A were all carried out on a sample of concentrate which had already been treated by cyanidation, and the studies at the other two laboratories referred to (Page 48) was done either on fresh ore, or on fresh concentrate, it was felt that the three investigations were hardly comparable. In March of 1939, therefore, a fresh shipment of concentrate was requested from the management of the Beattie Gold Mines, and several tests were repeated using this material.

A detailed account of these tests is not included since no improvement in the gold extraction was obtained and they do not advance any additional information. An accurate comparison of the screen analyses of the two concentrates is given in the following table. The analyses were

obtained by washing the material successively over a 240 mesh and a 400 mesh screen.

TABLE NO. 28.      Wet Screen Analysis of Concentrates.

Screen	Weight Per cent	
	Concentrate received in 1936	Concentrate received in 1939
+ 240	27.1	50.5
+ 400	8.6	9.5
- 400	<u>64.3</u>	<u>40.0</u>
	100.0	100.0

The procedure and gold extraction of a test which is comparable to Test No. 15 is as follows.

TEST NO. 27.

A charge of 800 grams of concentrate was ground for two hours in the ball mill at 60 per cent dilution. A quantity of lime to give 20 pounds of CaO per ton was added. The sample was then filtered, split into four parts, and each part was agitated with cyanide solution for varying periods of time. The rotating type of agitator was used.

TABLE NO. 29.                    Treatment and Extraction in Test No. 27.

Pulp dilution, 3:1

Strength of cyanide solution, 3 lbs.per ton

Gold content of head sample, 0.96 oz.per ton.

<u>Sample</u>	<u>Treatment</u>	<u>Extractions</u>
A	One hour agitation.	77.1
B	Two hours agitation.	79.2
C	Three hours agitation.	79.2
D	Three hours agitation, followed by a regrind for one hour in the ball mill and addition- al two hours agitation with fresh cyanide solution.	81.3

P A R T 1 1

S E C T I O N C.

STUDY OF THE CONCENTRATE BY  
GRAVITY SEPARATION OF THE MINERALS.

A report of the Canadian Department of Mines (57) describes the Beattie ore as being extremely fine textured and consisting of a quartz-carbonate gangue through which pyrite and arsenopyrite are disseminated in varying proportions. No native gold was observed. A later report (58) showed the presence of small amounts of gold occurring as a portion of the filling of skelton crystals of arsenopyrite, and also associated with small amounts of chalcopyrite contained in a mass of fine crystals of arsenopyrite. The fact that the gold was always in contact with arsenopyrite is emphasized, and it is noted that apparently the metal is later than the mineral. On the other hand the micrographs presented by Leblanc (59) indicate that the gold is for the most part associated with the pyrite and is present as minute grains locked up in crystals of pyrite.

TEST NO. 28.

The concentrate received in the fall of 1936 was used in this test. A large sample, weighing 4320 grams was split into three products by washing successively over a 240 mesh and a 400 mesh screen. The plus-240 and plus-400 mesh material were independently treated on a Wilfley table and a separation effected between the pyrite and the arsenopyrite. Three products were obtained from each sample, a concentrate which was high in pyrite, a middling which contained a fair proportion of arsenopyrite and a table tailing. A very small weight of table tailing was obtained with the plus 400 mesh material. It was therefore mixed with the middling, the two being considered as one sample. The approximate mineral distribution is given in Table No. 30. The per cent of each mineral present was calculated from the iron and arsenic analysis of the samples on the assumption that other minerals containing iron or arsenic were present in negligible amounts.

Including the minus 400 mesh material therefore, six products were obtained. Each of these was treated with cyanide solution, using "Winchester" bottles and the rotating type of agitator. The results of the cyanide tests are given in Table No. 31. The details of the cyanide tests are as follows. The pulp dilution was 3:1. The strength of the cyanide



TABLE NO. 30

MINERAL DISTRIBUTION IN THE VARIOUS SCREEN  
SIZES AND TABLE PRODUCTS

Sample	Weight Percent of Head	Sample	Weight Percent of Head	PYRITE		ARSENOPYRITE			
				Percent	Per Unit of Head	Percent of Total Present	Percent	Per Unit of Head	Percent of Total Present
Head	100.0			35.2			10.9		
+ 240	27.1			38.3			6.0		
		Table Conc.	8.47	80.7	6.8	18.2	8.3	0.7	6.0
		Middling	16.70	19.5	3.3	8.8	5.5	0.9	7.7
		Table Tail.	1.93	11.1	0.2	0.5	3.1	0.1	0.9
			27.10						
+ 400	8.6			44.1			8.1		
		Table Conc.	5.30	65.7	3.5	9.3	10.0	0.5	4.3
		Middling	3.30	9.0	0.3	0.8	5.0	0.2	1.7
			8.60						
- 400	64.3			36.4	23.4	62.4	14.6	9.4	80.0
					37.5	100.0		11.8	100.6

TABLE NO. 31.

GOLD DISTRIBUTION AND EXTRACTION IN THE VARIOUS  
SCREEN SIZES AND TABLE PRODUCTS

Sample	Weight Percent of Head	Sample	Weight Percent	Mineral Present		Gold Content Oz. per Ton	Gold Content Per unit of Head	Percent of Total Gold Present in each sample		Au Ext. percent of Gold	Au Ext. Percent Gold in Head
				Pyrite Percent	Arsenopyrite Percent						
Head	100.0			35.2	10.9						
+ 240	27.1										
		Table Conc.	8.47	38.3	6.0	1.03	0.28	25.6	14.6	55.2'	8.06
		Middling	16.70	80.7	8.3	1.85			10.0	46.2"	4.62
		Table Tail.	1.93	19.5	5.5	0.65			1.0	81.1	.81
			27.10	11.1	3.1	0.58			25.6		
+ 400	8.6										
		Table Conc.	5.30	65.7	10.0	1.40	0.12	11.0	9.0	52.2	4.70
		Middling	3.30	9.0	5.0	1.84			2.0	70.4	1.40
			8.60			0.58			11.0		
- 400	64.3		64.30	36.4	14.60	1.08	0.69	63.4	63.4	62.6	39.70
	100.0						1.09	100.0			59.29
					Head by Analysis	1.08					
								Extraction in Test No. 8			58.00
								Extraction after grinding for one hour		21.6'	
										80.4"	

solution was three pounds of NaCN per ton. The time of agitation was one hour with a lime solution containing ten pounds CaO per ton of ore, then the cyanide was added and the agitation was continued for five hours.

The results indicate that of the gold present in each of the screen sizes the greater amount was associated with the minerals of the table concentrate that is with the highly pyritic material, but no definite statement can be made whether it is with the pyrite or arsenopyrite. In the plus 240 mesh material the concentrate contained 66.1 per cent of the total pyrite and 41.2 per cent of the total arsenopyrite and included in this material was 57.0 per cent of the gold. These figures are all on the basis of the total amounts present in the plus 240 mesh material. The remaining 43.0 per cent of the gold in this screen size was present in the middling and the table tailing which together contained 33.9 per cent of the pyrite and 58.8 per cent of the arsenopyrite. The plus 400 mesh material gives comparable results from which no definite deductions can be made as to whether the gold is associated with the pyrite or the arsenopyrite. The table products should have been examined under the microscope but unfortunately the test was done at the close of the year and there was not enough time left to carry out a thorough microscopic study.

The test does indicate however that the reason for more than 20 per cent of the gold being refractory to solution in cyanide is more complicated than was previously thought, and that it is connected in some way with minerals present in the table concentrate. All accounts of previous work done on the Beattie ore conclude that the problem is purely one of unlocking the gold; the gold is present as very minute particles and the unlocking of these particles is beyond the limits of economic grinding. Section A of this study to a certain extent supports this contention.

A communication from the management of the Beattie Mine, received in April 1939, reports that the refractory portions of the concentrate are higher in silver and contain more arsenic than could be present in arsenopyrite. This suggests an association between the arsenic and the silver in some form, perhaps as a mineral not identified as yet in this ore. It is possible that the mineral came off the table at the concentrate end, that is, with the pyritic material, and is responsible for the unusual results obtained when the table concentrate was treated with cyanide. The middling or highly arsenical table products of the plus 400 mesh material yielded more gold than the similar coarser product. The additional unlocking of the gold in the middling of the plus 240 mesh material, obtained by grinding for one hour in the ball mill, raised

the extraction from 46.2 per cent to 80.4 per cent (See Table 31, Page 79). But with the pyritic material not only was there no additional yield of gold in the finer screen size but additional grinding actually resulted in less gold being dissolved (See Table 31, Page 79). Only 21.6 per cent was dissolved as compared with 55.2 per cent on the unground material. No explanation can be advanced for this unusual result. It must be realized that this is the result of only one test. There was not enough time to repeat the test and verify the results. All analyses for gold content however checked very well, and definitely no error was introduced through faulty analytical procedure.

## CONCLUSIONS.

The published reports of studies carried out on the Beattie ore indicates that all previous investigators have approached the problem with the idea that the gold is present as very small particles. The aim of the investigators has been the unlocking of the gold particles to aid the dissolving action of cyanide solutions. To a certain extent in this study, the problem was approached from the same viewpoint. The final test as given in Part 11 Section C of the experimental work indicates, however that the problem is more complicated, and should be approached from the point of view of a complete understanding of the gold association in the table concentrate. The work done at the Beattie Mine suggesting the possibility of a complex arsenic silver mineral as the reason for the refractory nature of a portion of the concentrate, should certainly be continued. The existence of such a complex mineral with dissolved gold may be the reason for the difficulty encountered in treating refractory arsenical gold ores. In view of this possibility the reduction tests (No. 20 and No. 21) should be repeated and the explanations advanced for the low extractions (Page 72) more thoroughly investigated.

## A P P E N D I X.

### METHODS OF ANALYSES USED IN THIS INVESTIGATION(60,61)

#### Arsenic Determination.

The following method was used to determine arsenic in both the concentrate and calcine.

Place a one gram sample of the material in a 500 c.c. Erlenmeyer flask. Add 2 grams of anhydrous sodium sulphate, 5 - 6 c.c. of concentrated sulphuric acid, and 1/4 of a 9 cm. filter paper. Heat gently until decomposition of the material occurs and then more strongly until all the sulphur fumes have been driven off and the carbon has all been oxidized. When clear allow the flask to cool on its side in order to avoid breakage. Add 90 cc. concentrated hydrochloric acid, 3 grams of cuprous chloride, 2 grams ferric chloride and a few glass beads to prevent bumping. Distill the liquid and receive the distillate in 100 c.c. of water in a 400 cc. beaker. Neutralize the distillate with 15 N ammonium hydroxide using litmus paper as an indicator. Make just acid with concentrated hydrochloric acid and then add 3-4 grams, or an excess, of sodium acid carbonate. Cool to room temperature, add a few c.c. of freshly prepared starch solution and titrate with a standard iodine solution until a permanent blue color appears.

### Iron Determination.

The method used in the Dichromate Method for iron and the procedure is as follows. Treat a one gram sample of the material in a 600 c.c. pyrex beaker with 10 c.c. of aqua regia. Take to dryness at a low heat and bake gently for about ten minutes. Add 15 cc. of concentrated hydrochloric acid and heat until the solution takes on a uniform yellow color and the residue in the beaker consists only of clean scilica. While hot, add stannous chloride drop by drop until the solution turns colourless, that is, until all the iron has been reduced to the ferrous state. Then add two or three drops in excess, wash the sides of the beaker with distilled water and make up to about 400 cc. Then add 10 cc. of a saturated solution of mercuric chloride. A white gelatinous precipitate of mercurous chloride comes down. Titrate with a standard solution of potassium dichromate using a solution of potassium ferricyanide as an outside indicator.

The ferricyanide should be made up in small quantities when required and should be dilute; about 0.1 grams in 15 c.c. of water. The ferrous iron changes the color of the ferricyanide to a deep blue, but it is not affected by ferric iron. At



the end point, when the ferrous iron has all been oxidized to the ferric state the color therefore remains unchanged.

#### Sulphur Determination.

Weigh one gram of ore into a dry 250 cc. Erlenmeyer flask. Add 3 grams of potassium chlorate and 10 to 15 cc. of nitric acid. Boil gently and take nearly to dryness, to a pasty condition, but do not bake. Neutralize with a saturated solution of sodium carbonate. Add 200 c.c. of the hot saturated solution of sodium carbonate and boil at constant volume for thirty minutes. Add 50 c.c. of cold distilled water. Filter and wash thoroughly. Cool the filtrate to room temperature, cover with a watch glass and cautiously neutralize with concentrated hydrochloric acid using 5 c.c. in excess. Boil to drive off the  $\text{CO}_2$ . Keeping the solution hot add 50 c.c. of a dilute solution of barium chloride, about 20 grams  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  to the litre. Add the barium chloride drop by drop stirring the solution constantly. Let the precipitate settle and add another 10 c.c. of the barium chloride solution. If more precipitate forms continue the addition until the precipitation is complete. Cover with a watch glass and continue heating below the boiling point for one hour or longer. Let it stand overnight. Then filter using filter paper of close texture. Wash the precipitate two or three times with hot water, then test the washings for presence of chloride ion. When the chloride test

is negative remove the filter from the funnel. Place in a porcelain crucible and burn off the paper at the lowest possible temperature. Complete the ignition by heating for 15 or 20 minutes at the full heat of a Tirril burner. Cool in a dessicator and weigh the precipitate. Calculate the weight of sulphur present.

If it is suspected that some reduction of the barium sulphate to the sulphide may have occurred, the contents of the crucible, after cooling, may be moistened with a few drops of concentrated sulphuric acid. The acid may then be driven off by heating carefully and then the precipitate weighed as above.

#### Determination of Free Cyanide.

The method used was identical with the practice at all mills, which treat gold ores by the cyanide process. A 10 cc. sample of the solution was diluted to 100 c.c. and then titrated with a standard silver nitrate solution using potassium iodide as an internal indicator. The end point is reached when the solution turns cloudy due to the formation of  $\text{Ag}(\text{CN})_2$ , a light grey precipitate. The solution of silver nitrate used was of a strength such that 1 c.c. was equivalent to 0.1 pounds KCN per ton of solution when titrating against a 10 c.c. sample.

#### Determination of CaO in Solutions.

Here again the method was one which has become standard

practice in most gold mills. A 10 cc. sample of the solution was titrated against a standard oxalic acid solution using phenol phthalin as internal indicator. The end point is reached when the characteristic pink of the phthalein disappears and the solution turns colorless. The strength of the oxalic acid was such that 1 cc. is equivalent to 0.1 pounds of CaO per ton of solution when a 10 cc. sample is used.

#### Determination of Available CaO in Lime.

The method is based on the fact that "free" or "available" CaO is easily soluble in a sugar solution. Procedure is to dissolve 20 gm. of cane sugar in distilled water and add one gram of the impure lime. Make up the volume to exactly two litres and agitate the solution well. The bottle may be shaken up at intervals for two days or placed in an agitating device for a few hours. When solution of the CaO is complete, allow the undissolved material to settle, withdraw an aliquot portion and titrate with standard sulphuric acid solution using phenol phthalein as an internal indicator.

#### Analysis for Gold in Solutions.

Procedure was to evaporate a known volume of the solution in a boat made of lead foil. A few grams of borax were then added, the lead scorified to a button of 25 grams, and cupelled.

Analysis of Gold in Ore and Calcine.

Gold was determined in the concentrate and calcine by the fire assay procedure. The flux charges were as follows.

(1) For 1/2 Assay Ton of Calcine.

1/2 Assay Ton	Scilica
2 $\frac{1}{2}$ "	" Litharge
1/2 "	" Soda
10 Grams	Borax
2 $\frac{1}{2}$ "	Flour

(2) For 1/2 Assay Ton of Concentrate.

1/2 Assay Ton	Scilica
2 $\frac{1}{2}$ "	" Litharge
1/2 "	" Soda
10 Grams	Borax
4 "	Nitre

(3) For one Assay Ton of Concentrate.

1 Assay Ton	Scilica
3 "	" Litharge
1 "	" Soda
20 Grams	Borax
14 "	Nitre

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PHOTOGRAPHS

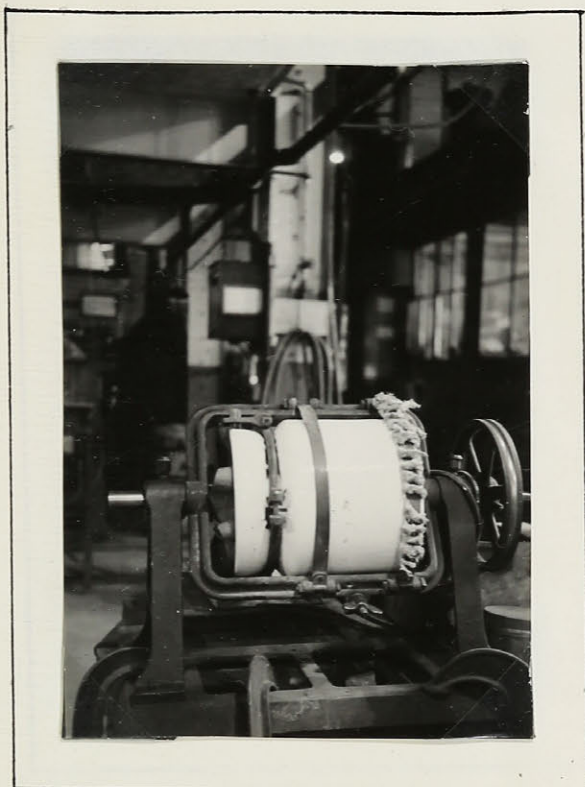


Fig. 1

Porcelain Pebble Mill.

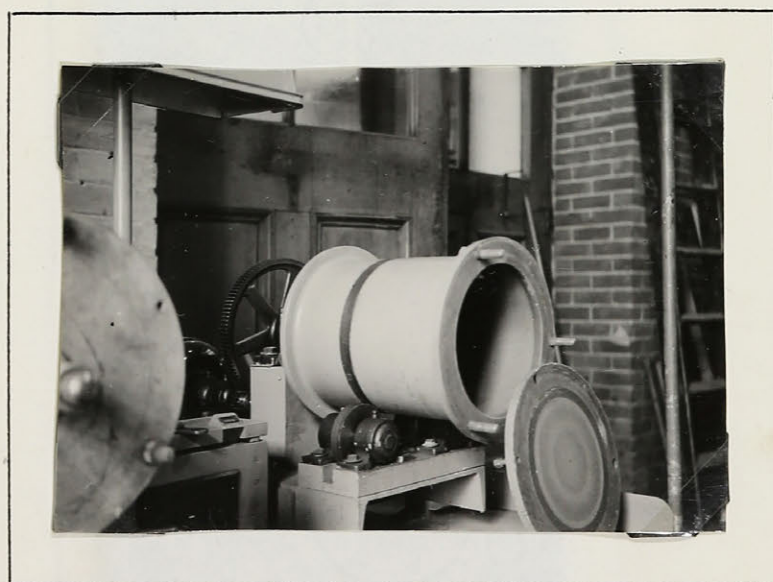


Fig. 2

Ball Mill



Fig. 3

Gas Fired Muffle Furnace.

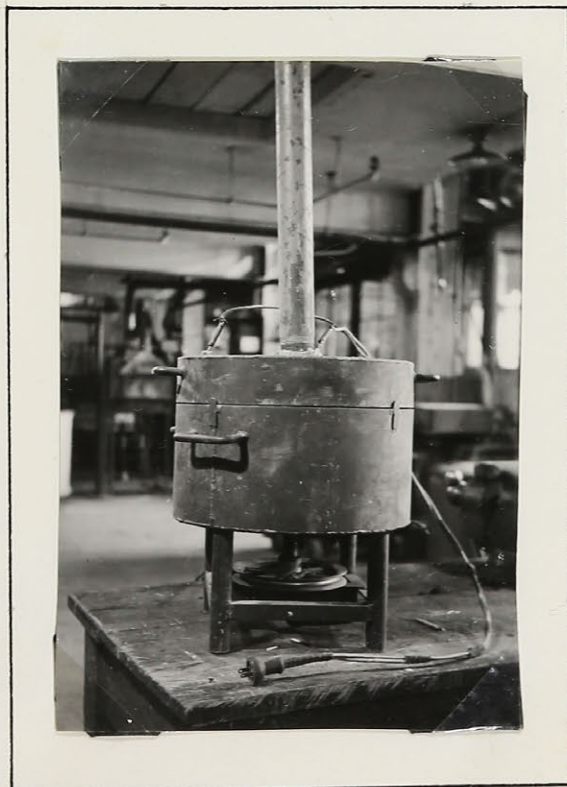


Fig. 4

Single Hearth Roaster



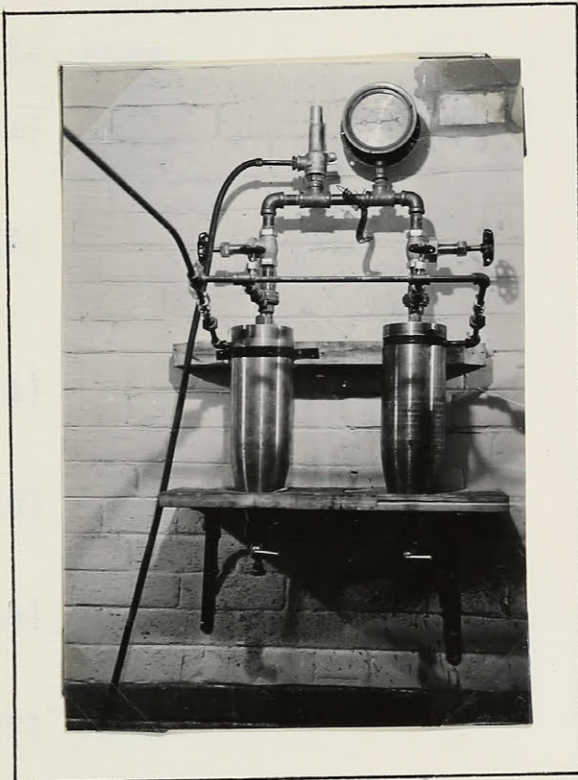


Fig. 5

Pressure Agitation Apparatus

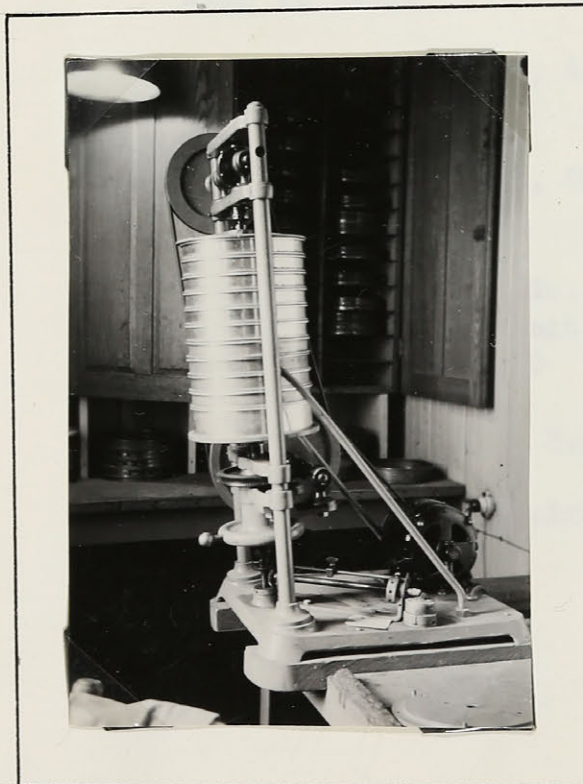


Fig. 6

Bell Screening Machine



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