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T H E S I S

for the

D E G R E E

of

M A S T E R O F S C I E N C E

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in

M E T A L L U R G I C A L E N G I N E E R I N G

-SUBJECT-

THE HISTORY AND THE DEVELOPMENT OF THE  
CYANIDE INDUSTRY.

Horace Tharp Mann

1909.

Approved

*Donward Copeland*

8292

UNIVERSITY OF MISSOURI  
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DEPARTMENT OF METALLURGY  
AND ORE DRESSING

DURWARD COPELAND  
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BOYD DUDLEY, JR.

May 27, 1909.

To The Members Of The Theses Committee:

The accompanying thesis is a compilation from articles published in the magazines and from books. Where no references are given, the statements are the impressions gained from reading several articles on the same subject. None of the work originated with myself.

Respectfully,

A handwritten signature in cursive script, reading "H. T. Mann". The signature is written in dark ink and is positioned to the right of the typed name "H. T. Mann".

## THE HISTORY AND THE DEVELOPMENT OF THE CYANIDE INDUSTRY.

### HISTORY.

The history of the salts of cyanide covers about two hundred years and the solubility of gold and silver in certain of these salts has been known for approximately one hundred years.

The first cyanide salt, of which mention is made, was Prussian blue. This compound was discovered by Dusbach (1) in 1704, but he had no idea of its composition. About 1750 Macquer (2) found that it was ferrocyanide of potassium, which combined with iron salts to form Prussian blue.

The next step was the discovery of potassium cyanide by Scheele (3) but it was not until 1815 that Gay Lussac (4) succeeded in determining the composition of the radical (CN). He also discovered the acid HCN (5).

In 1806 Hagen (6) predicted that potassium cyanide would dissolve gold. He placed potassium cyanide in the group with chlorine, bromine, and iodine (7).

The knowledge of this fact was not made use of by any one for nearly thirty-five years, when in 1840 Elkington (8) discovered that potassium cyanide aided by electricity would dissolve gold. The same year he patented his discovery (9).

There is some question as to whom the credit is due for first putting the solution of gold in cyanide to commercial use. A Dr. Wright, of Birmingham, England, seems to have used it for electro-plating as early as 1840 (10).

While investigating the patented process of Elkington's the Russian Prince P. Bragration (11) published a paper in which he stated that:

- I. Gold is soluble in potassium cyanide.
- II. The finer the gold, the more rapid is its solution.
- III. The electric current did not aid the solution in any way.
- IV. Heat was of great benefit.
- V. The electric current was not necessary for the precipitation of gold on metallic surfaces.
- VI. The solution of gold in potassium cyanide is hastened by the presence of air.

The next year, 1844, Elsner (12) stated that gold was soluble in potassium cyanide and that the dissolution of the metal is due to the oxygen absorbed from the air. Philip Argall states (13) that Elsner discovered the solubility of gold in potassium cyanide, but that the solution was the result of oxygen was published in 1846 (14). Also that he did not give out his equation to that effect until 1866.

In 1857 Farraday (15) announced that gold leaf was one hundred times more soluble when placed on the surface of potassium cyanide solution than when immersed in it, the work having been done the previous year.

In 1866 Wurtz (16) found that gold was soluble in dilute solutions of potassium cyanide.

The next step was the granting of a patent (United States Patent No. 61866, February 5, 1867) to Julio F. Rae (17), of Syracuse, New York, for the treatment of auriferous and argentiferous ores by potassium cyanide. His method was to crush the ore and then treat it by potassium cyanide aided by electricity and agitation, precipitating the gold by electricity. This method was never used, although it was along the lines of present practice.

In 1875 William Skey (18), of New Zealand, called attention to the fact that the use of potassium cyanide on the plates caused a loss. He cautioned mill men as to its excessive use.

From this date until 1880 nothing seems to have been done with the process, when a patent was issued to Clark (United States Patent No. 229586, 1880) (19) for the use of potassium cyanide in metallurgical operations. The next year (1881) a patent was issued to Faucett (United States Patent No. 234424, 1881) (20). The same year a patent was issued to J. F. Sanders,

(United States Patent No. 244080, July 12, 1881) (21) for the use of potassium cyanide for the removal of the coating from gold preliminary to pan amalgamation.

The first patent of any importance was issued to Jerome W. Simpson (22) (United States Patent No. 323222, July 28, 1885) for the separation of gold and silver from their ores. His method was to grind the ore to a powder of greater or less fineness, and then treat the ore with a mixture of about the following composition:

KCN	1 pound
(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	1 ounce
NaCl	$\frac{1}{2}$ ounce
H <sub>2</sub> O	16 quarts

He agitated, settled, and then put in a sheet of zinc to precipitate the metals. If silver was not present he omitted the sodium chloride.

He stated that the idea of the use of zinc and potassium cyanide did not originate with him.

In 1884, A. P. Price (23) applied for and was granted a patent (English Patent No. 5125) for the precipitation of gold and silver, by zinc in a fine state of division, from cyanide solutions.

During the next two years, two caveats were filed covering the use of potassium cyanide for the extraction of gold and silver from their ores, one in the names of

F. M. Endlich and N. H. Mühlenberg, in the spring of 1885, and the other in the name of Louis Janin, Jr., in 1886, but no patents were issued (24).

On October 19, 1887, McArthur and Forrest were granted their English Patent (English Patent No. 14174). Their application for a patent in the United States was dated November, 1887, but the patent was not issued until May 14, 1889 (United States Patent, No. 403302) (25). Their claim, or patent, as finally granted by the United States, was for the extraction of gold and silver from their ores by a solution of potassium cyanide which contained not over eight parts of cyanogen (CN) per thousand.

Sometime later they were granted a patent on the precipitation of gold and silver from cyanide solutions by zinc, preferably in a filiform, or threadlike, condition and on the use of caustic alkali to neutralize the acid in the ores (26).

In 1887 the first plant was installed at the New Zealand Crown Mines, Karangahake District, by the Cassel Gold Extraction Co. They used agitation for a time, giving it up in favor of percolation (27).

The first plant in South Africa was erected by Butters to treat the tailing from the Robinson Mine at Johannesburg. The production of South Africa by cyanide was less than six thousand dollars for 1890 (28). This plant was started December 23, 1890 (29).



At about this time, or a year later, (1891) the first plant was erected in the United States but where or by whom I do not know. Matt W. Alderson (30) states that one of the first successful plants in the United States was the Revenue Mill in Montana.

After this date the process seems to have come into general favor and its development was very rapid. This is shown by the output of South Africa (31), which for 1890 was less than six thousand dollars, for 1891 was sixty thousand dollars, and for 1892 was three hundred thousand dollars.

The next point of interest was the introduction of crushing in a dilute solution of cyanide. This, according to Philip Argall (32), was introduced by Almarin B. Paul at the Calumet Mill, Shasta County, California, in 1891 (33).

In 1892 Gaze (34) discovered the benefits of using the Halogen salts of cyanide to aid the solution of gold.

The next year Argall claims the credit of introducing the separation of the dust and slime from the sand and the separate treatment of each (35).

According to Tomas N. Smith (36) agitation and filter pressing were introduced at the mill of the Attica mine which commenced operation July 23, 1893. At this date there were only five or six cyanide mills in the United States. The production of South Africa for this year by cyanide was six million

dollars. Also McArthur patented the idea of adding soluble lead salts to the cyanide solution to prevent the formation of sulpho-cyanides (39). Argall (40) states that this modification was patented by McArthur and Ellis in 1896 (United States Patent No. 555463, February 25, 1896).

In 1895 Argall claims to have introduced the roasting of sulpho-telluride ores (41).

In 1896 the decantation process was introduced by J. R. Williams (42) at the Crown Reef Gold Mining Company's mill, which began operation August 1896. In the same year Diehl (44) claims to have used a filter-press.

The estimated production of gold by the cyanide process for 1897 was (45):

United States	190,000 fine ounces
World	1,215,000 fine ounces

At this time there were forty cyanide mills in the United States (46).

The next year, 1898, J. W. Southerland (47) introduced tube-milling and filter-pressing into the Australian gold fields.

The world's production of gold by cyanide for 1900 was (48):

United States	497,820 ounces bullion
Transvaal	85,000 ounces bullion
Other countries	<u>1,243,233 ounces bullion</u>
Total	1,826,053 ounces bullion

During 1903 Moore was granted a patent on a suction or vacuum filter (United States Patent No. 748088, December 23, 1903) but this filter was not a success. In 1904 a second patent was issued to him, (United States Patent No. 764486, July 5, 1904) (49).

The next year (1904) Cassel was granted a patent on a vacuum filter (United States Patent No. 773473, December 25, 1904) (50). This filter differs from the Moore in that the filter is fixed, the solutions being changed instead of the filter being moved from one tank to another.

In 1905 Ridgway (51) introduced his revolving automatic suction filter. During 1906 the consumption of potassium cyanide was ten thousand tons, of which South Africa used about one-third. (52).

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### CRUSHING.

The method of crushing the ore previous to its treatment by the cyanide process is important. If the ore is to be treated by percolation, machines should be chosen which will crush the ore to the requisite fineness and produce the minimum of fine material which cannot be treated by percolation. If the ore is to be ground fine and treated by some method of agitation, the production of fine material will be a benefit. If the ore must be roasted, some method of dry crushing must be used. If it is to be amalgamated, some wet method will be used.

Then, the important item of cost must be considered. Especially is this true at the present time when so much attention is being given to the working of low-grade deposits and to the perfection of the mechanical details of construction.

WET CRUSHING:--There is probably more ore crushed wet in stamps than by all other methods combined. This is probably due to the fact that cyanide is used as a secondary treatment after amalgamation and it seems to be almost universal practice to crush by stamps for that purpose.

The most note-worthy point in connection with the crushing by stamps has been the great increase in the weight of the stamps used in South Africa. A few years ago there were few, if any, stamps used in Africa which were over one thousand pounds in weight. In 1907 they were installing stamps

which weighed from sixteen hundred fifty to eighteen hundred pounds, and at the present time are considering stamps up to twenty-five hundred or three thousand pounds in weight. It seems rather peculiar that instead of using such heavy gravity stamps they do not install some form of steam stamp, especially when there is no longer any objection to the production of fine sand and slime.

Other methods of wet crushing are used, such as breakers followed by rolls, and Chillian mills or rolls alone.

DRY CRUSHING:--While wet crushing seems to lead, the output of dry crushing plants is by no means small.

Alfred James (1901) (1) states that the output of gold from dry crushing plants is very large but at that time the African mines were shut down on account of the war. Also, it appears that dry crushing is cheaper.(2).

Dry crushing is used extensively in Australia where a large amount of the ore is a sulpho-telluride which must be roasted. This is also true of the Cripple Creek ores from below the oxidized zone which have not been weathered on dumps.

There are two general methods of dry crushing:

I. By breakers and rolls.

II. By breakers and ball-mills.

Which of these is the better is difficult to state. Alfred James (3) prefers the ball-mill. Philip Argall, at the Metallic Extraction Works, Florence, Colorado, used rolls.



For the fine grinding of sands for treatment by agitation or filter-pressing, the tube-mill has been <sup>adapted</sup> practically the world over. There are a few mills in Australia and a few in other parts of the world where pans are used.

This machine, <sup>the tube-mill,</sup> will grind either wet or dry material and at the present time it is the most efficient machine for this class of work. When working wet, Bosqui (4) states that the best mixture is one part of sand to one part of water.

Almost every form of crushing device has been used in some plant. Some were successful, others failures. Whether it was the fault of the machine or of the man in charge, it is difficult to say. As to which method (wet or dry) or what machine to use is one of the questions the engineer must decide for each plant and he must be guided to some extent by local conditions.

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ROASTING.

When it was attempted to treat the unoxidized sulpho-telluride ores, metallurgists found that it was necessary to roast the ore and that it should be roasted "sweet". Almost every furnace on the market has been tried. The majority of mill men seem to prefer a form of roasting-furnace having one roasting hearth and mechanically rabbled, or a revolving furnace. James (1) states that the "Argall", "Edwards", and the "Ropp" appear to be the most successful. The requirements for roasting this class of material are:

- I. The heat must be applied gradually.
- II. The finishing heat must be fairly high in order to break up the sulphates if possible and to drive off all the sulphur.
- III. The rabbling must not be violent as the ore is usually in a fine condition and loss by dust would be great.

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### CLASSIFICATION.

This is usually done by Spitzkasten and Spitzlütten or by cone classifiers. At Hanans Brownhill Mine (Mineral Industry, Vol. 7, Page 326) the dry material is classified by air.

### SOLUTION.

The next step is the solution of the gold. There are three general methods of doing this:

I. Percolation.

II. Agitation.

III. Filter-Pressing.

besides combinations of these.

PERCOLATION:-- This is usually done in vats. In the old plants they were of almost any size and shape. In the modern plants they are of great size holding from four hundred to six hundred tons or more.

In most of the modern plants the tanks are of steel, circular, and varying in size according to the capacity of the plant. Butters (1) states that about the largest tanks in the world are at the Rand Central Ore Reduction Co. and are fifty feet by sixteen feet. At one time rectangular tanks were used but experience has shown that they are not as satisfactory as the round tanks.

One trouble with the rectangular tanks was the difficulty of keeping the solution from cutting chambers in the corners of the tank.

In the bottom of the tank is placed a filter. These filters are usually of cloth resting on a lattice work of wood or on boards full of auger holes, supported on stringers. The filter used at the Homestake Mill is ten-ounce-duck over cocoa matting(2). At the Metallic Extraction Company's Plant at Florence, Colorado, the filter was ten-ounce-duck over cane matting on a board frame (3). Sand is sometimes used as a filter and where used is usually covered with burlap or canvas to protect it.

The vats are filled in various ways. If the ore is crushed dry, the more common way of filling is by hand. The ore is loaded into cars, trammed to the tanks and dumped. When the tank is filled it is leveled off. If the ore is crushed wet it is, after the removal of the slime, run into the tanks, usually some form of mechanical distributor is used.

When the ore contains a large amount of free acid or soluble acid salts, the usual practice is to wash the ore either with water or with an alkaline solution, caustic soda, or lime being the alkalies ordinarily employed.

If the ore contains only a small amount of these salts or acids enough lime ( $\text{CaO}$ ) is usually added to the ore to neutralize the acid and acid salts.

Lime is not the only alkali used. Some ores have a tendency to "set" (like cement) in the tanks, in which case lime makes this condition worse. Sodium and potassium hydroxide are used but they are usually more expensive than lime and if the ore contains soluble sulphides they form the alkali sulphides. This can be remedied by the addition of soluble lead salts which form lead sulphide ( $\text{PbS}$ ) which is not acted on by the cyanide solution to any extent.

SOLUTION:--The strength of the solution must be determined for each case. The extremes of general practice seem to be between .01 of one per cent or 0.2 pounds of potassium cyanide per ton (4) and .5 of one per cent or ten pounds per ton (5), although weaker and stronger solutions are probably used.

It is aimed to keep the protective alkalinity high enough so that there will always be at least one-half pound of free alkali per ton of solution.

The rate of percolation is determined from a commercial stand-point. It is a question as to whether it is better to percolate slower and treat more of the fine material, or to maintain a higher rate and treat more slime by agitation.

It is seldom that the rate of percolation is less than one inch per hour, but this rate is used(6). The amount of solution used per ton of ore and the time of leaching must be determined by experiment. In South Africa they use 1.3 to 1.5 tons of solution per ton of ore and leach for from four to seven days (7). At another plant they leach the ore sixteen days (8). After the gold has been dissolved and the solution drained off, the residue is washed, sometimes first with dilute solution and then with water, or with successive water washes until the solution coming from the vat is too low-grade to treat. The washing is then discontinued and the tailing is discharged. This is done in several ways, some of which are as follows:

- I. It may be sluiced out through doors in the side and in the bottom of the vat.
- II. It may be shoveled into cars or belt conveyors through bottom doors.
- III. It may be discharged by means of some of the mechanical appliances designed for this purpose.

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### AGITATION.

Today this method of treatment is superseding the old method of percolation all over the world. The oldest patents granted used agitation but this was discarded in favor of percolation. This was probably due to the lack of the proper machinery and knowledge of the subject.

As time went on the hundreds of thousands of tons of slime increased, which could not be treated by percolation. Engineers turned their attention to methods of handling this material. Argall (1) claims the credit for first separating the fines and slimes from the sand and treating them separately. Williams (2) introduced the decantation process into South Africa while to Southerland (3) probably belongs the credit of introducing slime treatment into Australia.

The material to be treated by agitation should be in a fine condition. If not it settles rapidly and packs. This requires that the agitation be violent and is a needless waste of power. If the material is coarse, the solution of the gold is slow and it is not practical from a financial stand-point to agitate one charge several days.

The methods of agitation are numerous. In South Africa the methods which seem to be most in use are two in number. One is allowing the pulp to flow from the bottom of the tank into a centrifugal pump and pumping the material to the top of the same tank, or to the top of another tank, where it is discharged.



The other method employs a series of radical arms attached to a central shaft and this is rotated by a bevel gearing. These arms travel just fast enough to keep the mass in motion. In Australia pans were used but were found to be too small and the agitator having radical arms seems to be the favorite. The United States does not seem to have any standard practice. There are numerous other forms of agitators, some of which are as follows:

The Brown (4) is a tank from twelve to fifteen feet in diameter by from forty to fifty-five feet deep, having a conical bottom. In the center of this tank is a pipe having a diameter of about one-twelfth that of the tank. Into the bottom of this pipe compressed air is introduced which causes the pulp to flow upwards through the pipe, aerating as well as agitating the pulp.

The Hendrix (5) is about the same as the Brown except that instead of air a screw propeller is used to raise the pulp. At the top of the central tube is a plate to carry the pulp to the edge of the tank. This plate is often amalgamated, and is the cathode for precipitating by electricity.

Another form employs compressed air. It has arms, arranged like a revolving sprinkler, placed close to the bottom of the vat. Through these arms compressed air is forced.

The solution used in agitation, as a general rule, is not as strong as that used for percolation. Lime is the general alkali used. There are two reasons for this: first, that it furnishes the necessary alkali; and second, that it helps to coagulate the slime, giving a greater settling rate.

There are three general methods of slime treatment:

- I. Agitation and Decantation.
- II. Agitation and Filter-Pressing.
- III. Agitation and Filtering with a Vacuum Filter.

Another method used is to carry out all the work in the filter-press.

THE DECANTATION PROCESS:--This method is used principally in Africa where it originated. In Africa the principal methods of agitation are either circulating the pulp by means of a centrifugal pump, from the bottom of one tank, through the pump, to the top of the same tank or another tank, or by mechanical agitation, the agitator having radial arms being preferred. At some properties they use a combination of these methods.

After the gold is in solution, which takes from four to eight hours, the agitation is stopped and the pulp, to which lime has been added at the time of crushing, is allowed to settle. The clear solution is siphoned off, more solution added, agitated just enough to mix thoroughly, settled, and the clear solution again siphoned. This dilution, agitation,

settling, and siphoning is continued until the value of the pulp is low enough to throw away. It is then discharged. When discharged the pulp is usually about fifty per cent moisture. One objection to this method is the large amount of solution which must be handled. This necessitates an expensive plant.

AGITATION AND FILTER-PRESSING:--In Australia the dewatering of the slime is accomplished by the use of the filter-press, the decantation process never having met with much favor. This is probably due to two causes:

- I. The scarcity of water.
- II. The high value of the ore treated would take too much dilution before the tailing could be discharged.

There are two methods of pressing used:

- I. Single Pressing.
- II. Double Pressing.

In the single pressing method the slime is dewatered in settlers and then run to agitators, usually of the radial arm, mechanical driven type, agitated with solution until the gold is dissolved. It is then run to filter presses, washed in the press and discharged.

In the double pressing method the slime is dewatered by filter-presses before going to the agitators. This enables them to treat a pulp in the agitator containing less water than is possible by the single pressing method. After

agitation it is pressed, washed, and discharged as in single pressings.

The cake or tailing when discharged contains fifteen to twenty-five per cent moisture and they use about one ton of water per ton of ore (6).

#### FILTRATION BY VACUUM:--

This method seems to be the favorite in the United States at the present time. The solution of the gold in the slime is carried out by various schemes of agitation. The solution is then withdrawn, the tailing washed and discharged by means of a vacuum filter such as the Moore, Butters, Ridgway, and others.

Another method of treating slime is to carry out the entire operation in the filter-press.

This method is used at the Homestake Slime Plant where the Merrill Press is used. It presents the advantages of using pressure during the solution of the gold, the use of a minimum quantity of solution and wash water.

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OTHER METHODS OF TREATMENT--BROMO CYANIDE:--This method was used for years in Australia for the treatment of sulpho-telluride ores without roasting. It has also been used at some other places. It depended on the use of bromo cyanide mixed with potassium cyanide to dissolve the gold.

The method was used for years, but was finally replaced at most places by roasting and fine grinding. It is, however, still used at a few mills. The worst feature of the process was the cost of the bromine or the bromo cyanide used.

CYANIDE OF MERCURY:--Dr. Keith (Mining and Scientific Press, Vol. 75, Page 119, 1897 Edition) claims to have invented the use of cyanide of mercury.

He used a solution of 0.05 per cent potassium cyanide and 0.025 per cent  $HgCN$ . The gold and mercury were precipitated on amalgamated copper plates by electricity.

SCHILZ PROCESS (Mining and Scientific Press, Vol. 84, Page 192):--This process was tried in South Africa and depended on the use of a strong solution of potassium cyanide, leaching for a long time and the use of barium peroxide.

BERGER CYANIDE PROCESS (Mining and Scientific Press, Vol. 84, Page 299):--This process depended on the use of centrifugal pumps to aerate the solution, thereby supplying the necessary oxygen to effect the solution of the gold. This method was used at the Dorcas Mill, Florence, Colorado.

There are three general methods of precipitation in use at the present time. They are:

- I. Precipitation on filiform zinc.
- II. Precipitation by electricity or the Siemens-Halske process.
- III. Precipitation by zinc dust.

There are other methods of precipitation in use but they have not received general application.

**I. FILIFORM ZINC:--**This method of precipitation is the oldest of the methods in use at the present time. It is also used to a greater extent than all other methods combined.

The precipitation of the gold and silver is accomplished by passing the gold-bearing solution slowly through a box, or some other form of container, filled with zinc shavings or thread.

The precipitation is a replacement, the zinc replacing the gold in solution.

The precipitation, extractor, or zinc box is usually built of wood or enameled iron and varies in size, depending upon the amount of solution to be precipitated. It is considered best to have a separate box for each class of solution which is to be precipitated; thus, a mill in which the solutions used are a strong solution, a weak solution, and wash water, should have three boxes.

The boxes are usually from fourteen to twenty feet long, from two to four feet wide, and from two to three feet deep. Each box is divided into from five to ten or more compartments. These compartments should be arranged so that the depth is greater than any other dimension (1). In order to prevent the clogging of the box by the precipitated slime and the material which is carried there mechanically, baffle-boards are placed at the head of each compartment. These force the solution to the bottom of the box and it then rises through the zinc. In each compartment and about four inches from the bottom is placed a screen tray of about one-eighth-inch mesh<sup>screen</sup> (2) on which the zinc rests.

Usually the first and last compartments are used as settlers, the balance being packed with freshly-cut zinc. The shavings should be packed in the box evenly, the corners being well-filled. Care should be taken that there are no bunches of zinc which will not allow the solution to pass and that the entire mass is loose enough to insure a free passage of the solution. The zinc is cut or turned on a lathe, the turning being about  $1/1260$  inches thick and  $1/32$  inches wide (3). One pound of shavings has about fifteen hundred thirty square feet of surface (4) and when packed in the box weighs about six and one-quarter pounds per cubic foot (5).

The length of time that the solution should be in contact with the zinc is dependent on the strength and purity of the solution. James (6) states that for weak, foul solutions the time of contact should be from one and one-half to two hours. Stronger solutions would not take as long a time of contact.

The rate of flow through the box is dependent on the time of contact necessary to precipitate the gold and silver. The rate varies from about one-half ton solution per cubic foot of zinc per twenty-four hours for weak solutions, to about two tons per cubic foot of zinc per twenty-four hours for strong solutions. General practice seems to be about one ton per cubic foot of zinc per twenty-four hours for ordinary strong solutions and about one-half this rate for weak solutions.

It has been found that pure zinc is not as good as ordinary commercial zinc which usually contains lead, but it should not contain over 1.5 per cent lead (8).

In the early days of the process, trouble was experienced in precipitating the gold from weak solutions. This led to the introduction of the zinc-lead couple and the zinc-mercury couple. The former, which consists of placing the shavings in a ten per cent solution of lead-acetate for a few minutes, was patented by McArthur and Ellis (9) (United States Patent No. 592153, October 19, 1897) and was



introduced at the Crown Deep Slime Plant by T. L. Carter and  
L. H. Betty (10).

The zinc-mercury couple was patented by Caldecott (11)  
(United States Patent No. 620100, February 28, 1899). The  
amount of zinc is approximately one-half to one pound of zinc  
per ton of ore treated. James (12) states that it is about  
one-quarter pound per ton of ore treated. The following are  
some of the statements as to the consumption of zinc:

Bodie, California (13) 0.1839 pounds of zinc per ton  
of ore. Metallic Extraction Company, Florence, Colorado (14)  
5.31 ounces troy of zinc precipitated, one ounce of gold, and  
one-tenth ounce of silver.

Horseshoe Mill, Dakota, (15), one pound of zinc  
per ton of ore.

Dakota Mill, Dakota, (16) 0.58 pounds of zinc per  
ton of ore.

As fast as the zinc in the box is consumed it must  
be replaced. This is usually done every twenty-four hours  
or oftener if necessary. The fresh zinc is always added to  
the last or lowest compartment, the old zinc being moved up to  
the compartment above it. When it is desired to "clean up"  
the box the solution is turned off and water is run through  
the box until most of the cyanide is washed out. Then the  
zinc is stirred and the zinc taken out.

The short zinc on the screen may be sent to the refinery as also may part of the coated zinc thread. The amount of zinc taken from the box will depend upon how close it is desired to "clean up". Sometimes it is better to lose zinc in refining than to have the gold coated zinc in the box and pay interest on the money invested.

The slime which has settled to the bottom of the box is discharged through valves or gates in the bottom of the box. The box is then repacked, the old zinc being used and fresh added to make up that lost by removal.

**II. THE ELECTRICAL METHOD OF PRECIPITATION OR THE SIEMEN'S—HALSKE PROCESS:--**This process was first introduced on a practical basis in 1894 at the Worchester Mill in the Transvaal (17). It has been and is extensively used in South Africa but at the present time it seems to be on the decline. This, no doubt, is due to the fact that it is now possible to precipitate weak solutions by zinc, its chief claim being that it was possible to use weaker solutions when the electrical method of precipitation was employed, than with zinc precipitation. It has never been used to any extent in America. In 1904 there were two plants in the United States and one in Mexico (18).

The process as carried on for years used vats, anodes, and cathodes of various sizes. In 1897 they were making standard size vats (19). The vats intended for treatment of the solution from percolation plants were as follows:

Thirty-two feet long by five feet wide and five feet deep, containing one hundred fifty-six anodes.

Those intended for use in slime plants were:

Thirty-two feet long by ten feet wide and five feet deep, containing two hundred eighty-eight anodes four and one-half inches apart.

The anodes are of iron and are usually about three-sixteenths inches thick.

The cathodes are of sheet lead which weighs about three ounces per square foot. The current density employed is about 0.06 ampere per square foot of anode surface. The voltage is just sufficient to overcome the resistance and will vary from two to five or six volts, depending upon conditions of the solution and the construction of the vat.

The following is a description of a vat in use at the Croesus Works (20):

"Boxes thirty feet long by four feet, seven and one-half inches wide, by nine feet deep,

Each contain

One hundred twenty-one anodes of iron four and one-half feet wide, three feet high by three-sixteenths inches thick,

One hundred twenty cathode frames each holding four sheets of lead four feet by two feet or, seventy-six hundred eighty square feet of cathode surface, figuring both sides.

Each box will precipitate fifty tons of solution from one-tenth ounce to a trace per twenty-four hours. The solution travels one hundred twenty by eight feet or nine hundred sixty feet in fourteen and one-half hours or one foot per minute".

III. PRECIPITATION BY ZINC DUST:--This method of precipitation has been used for years but has not met with very great success. Its use has been confined mostly to a few of the larger plants. Whether this has been due to the fact that the process is not suited to the use of small plants or that men do not care to change from a process with which they are familiar to an uncertain one, is impossible to state.

The method as used consists of spraying the zinc dust or "fume" into the solution to be precipitated and agitating for a short time to insure a complete mixture, then putting the solution through a filter-press to collect the precipitate.

OTHER METHODS OF PRECIPITATION:--There are several other methods of precipitation, the most important of which is the precipitation by charcoal. This, the Johnson process (21) of precipitation is to pass the solution slowly through crushed charcoal. There are several points in its favor, some of which are as follows:

The fineness of the bullion is high and the extraction is high regardless of the strength of the solution (22). The

principal objections to it are:

The large bulk of charcoal required for a given amount of solution. It takes forty filters, each holding five hundred sixty pounds of charcoal to precipitate four hundred tons of solution per week (23). The large consumption of cyanide and the danger of loss when burning the charcoal after precipitation (24). This method has not met with general usage but it has been used at several mills.

The Pelatan-Clerici process was patented December 17, 1895 (United States Patent No. 551684) (25) and has been used in a few places in the United States but it was practically a failure.

The process consisted of agitating the ore with cyanide. The bottom of the agitator was an amalgamated copper plate which served as a cathode, the arms of the agitator being the anode (26).

The Malloy Process was the precipitation by means of sodium or potassium amalgam, which was formed in the precipitator as fast as required by electricity.

The Bettle Process (28) was the precipitation of gold and silver by electricity. It is practically the same as the Siemens-Halske except that Bettle used a cathode of zinc instead of lead.

The Moldenhauer Process (29) uses aluminum in place of zinc.

The Andrelai's Method is the same as the Siemens-Halske except that he uses an anode of lead coated with lead peroxide (30).

Sherard Cowper-Coles uses a cathode of aluminum instead of lead as in the Siemens-Halske (31).

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## REFINING OF THE PRECIPITATE.

The refining of the precipitate obtained in the cyanide process especially that obtained by precipitation by zinc is necessarily a troublesome operation. This is due to these facts; , first, the precipitate is in a fine condition and there is danger of loss from dust; second, that the precipitate is high in zinc and in all smelting operations a high percentage of zinc causes trouble; third, the high value of material makes a high percentage extraction necessary.

There have been many methods used for the refining of this precipitate the most important of which are as follows (1):

- I. Direct Fusion with Fluxes.
- II. Roasting and Fusion.
- III. Treatment with Acid and then Fusion of the Residue.
- IV. Tavner's, or Smelting with Lead Salts and Cupellation of the Resultant Lead Bullion.
- V. Volatilization of the Zinc and Fusing the Residue.

I. DIRECT FUSION OF THE PRECIPITATE:--This is the oldest method of refining the precipitate and at the present time it has been discarded. The objections to it were the difficulty of eliminating the zinc and other impurities from the bullion, of obtaining a clean liquid slag, the excessive cost of fluxes, the large volume of precipitate to be handled, and the correspondingly large amount of flux.



The method of procedure was to dry the washed precipitate and then charge it, with suitable fluxes, into graphite crucibles. Nitre was used extensively to oxidize the zinc.

**II. ROASTING OF THE PRECIPITATE PRELIMINARY TO FUSION:--**This method was used extensively in South Africa (2). Whether it is still in use or has been superseded by other methods, I cannot state.

This method is to roast the precipitate at a dull red heat until the zinc is oxidized.

The roasting is usually carried on in a cast-iron muffle, the precipitate being placed in shallow iron pans for the purpose.

Care must be used in this operation to prevent loss of gold. This is due to two causes: the first, is by dust; the second, if the heat is too high the fumes of zinc oxide will carry away gold mechanically. After the roasting is complete the precipitate is cooled, broken up, and charged with the proper fluxes to crucibles and melted.

**III. TREATMENT WITH ACID PRELIMINARY TO FUSION:--**This method of treatment has been and is being used in most of the plants in the United States. The object of treating with acid is to remove the zinc before fusion, thereby saving the large quantity of flux required to slag the zinc.

The objections to this method are the extra equipment required to treat the precipitate by acid, the loss due

to solution of the precious metals and the formation of the extremely poisonous gas, hydrocyanic acid (HCN), and arsine (AsH<sub>3</sub>) is also formed if arsenic is present in the ore treated which is as dangerous as the hydrocyanic acid.

The method presents the advantage of effectively removing the greater portion of the zinc and other impurities present which are soluble in the acid used. The removal of the zinc and other soluble salts enables them to treat a much higher grade precipitate in the furnace and saves the cost of the fluxes required to slag the zinc. The method as used is as follows:

The precipitate as it comes from the box is de-watered to a greater or less extent and then charged to the acid or "cutting down" tank. This tank should always be mechanically agitated and have a close-fitting hood with a very strong draft. This is to protect the men attending to this work from the hydrocyanic acid, arsine, and other poisonous gases which are given off during the acid treatment. The acid, which is usually sulphuric acid (H<sub>2</sub>SO<sub>4</sub>), on account of its low cost, is added, and the mixture agitated until the solution of the zinc is completed. Heat may or may not be used to aid the solution of the zinc.

The material is then filtered, the residue dried, and is then charged to the crucible with suitable fluxes and melted. This is usually done in a graphite crucible having a

capacity of from two to three gallons or larger. This fusion is poured and when cold the resultant button is broken off. The buttons thus obtained are again melted and cast into bars. The slag is usually crushed and concentrated; the concentrate, which is mostly gold-shot, is saved and mixed with the next melt. The slag, if of any value, is sold to some smelter.

**IV: SMELTING WITH SALTS OF LEAD AND CUPELLING THE RESULTANT LEAD, OR TAVNER'S METHOD;--**This method, or some modification of it, is used in South Africa and at some of the larger plants elsewhere. It seems to be by far the best method in use, if there is a large amount of precipitate to be re-fined, but for use in small plants I do not think it will ever come into favor.

The method is to mix the dry or partially dry precipitate with suitable fluxes. In one case, the mixture to be smelted was as follows:

Slimes	30.0 per cent
Litharge	32.48 per cent, ten per cent of which is saved for a cover.
Coal	4.1
Iron	4.2 in the form of tin plate scrap.
Assay Slag	16.0
Silica	<u>8.3</u> in the form of clean quartz 95.0 per cent

This is then charged to a reverberatory furnace with the lead from the last by-product smelt and fused. As the slag

forms it is run off and saved. When the entire charge is melted, and liquid, the balance of the slag is run off and the lead is withdrawn and cast. The resultant lead is then cupelled in a cupellation furnace. The impure slag is smelted in a small blast furnace, the lead bullion obtained from this being the lead added to the reverberatory furnace at the time of smelting the precipitate. The litharge obtained from the cupellation of the lead bullion is saved and used with the next charge.

A method somewhat similar to this is in use at the Homestake Mill.

V. VOLATILIZATION OF THE ZINC AND FUSION OF THE RESIDUE:--This method consists of volatilizing the zinc and then smelting the residue. Rose (4) states that Sulman and Picard at Deloro found a great loss of gold due to its being mechanically carried off by the zinc.

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