(No Model.) P. G. L. G. DESIGNOLLE. 2 Sheets—Sheet 1.

Method of Treating Copper Ores Containing Precious Metals.

No. 243,673. **Patented June 28, 1881.**

Fig. 7.

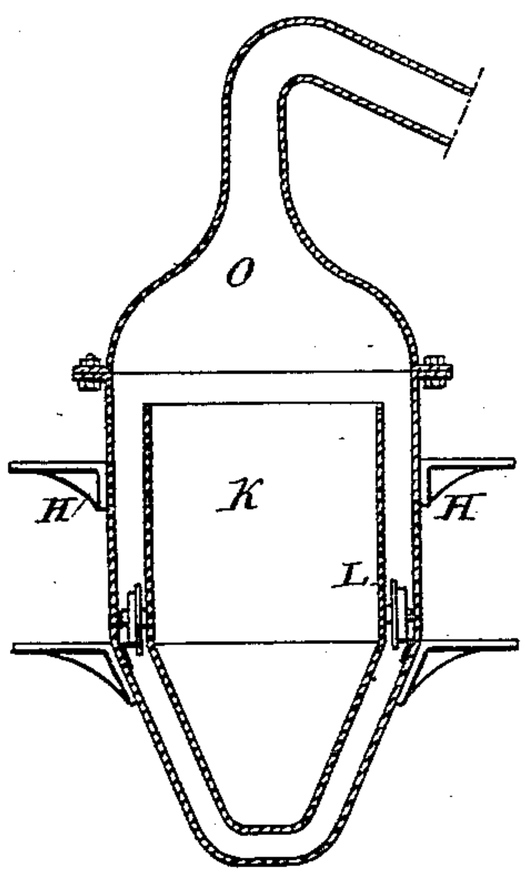


Fig. 8.

Fig. 2.

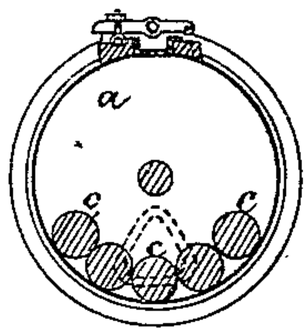


Fig. 3

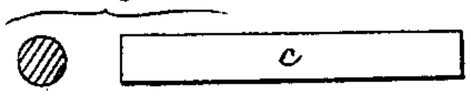


Fig. 1

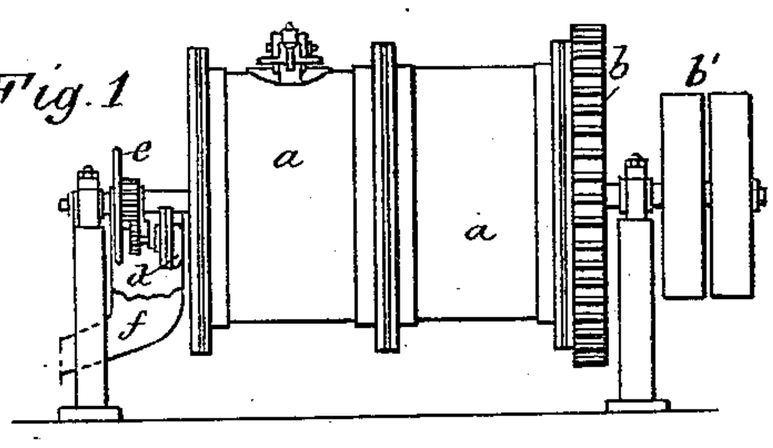
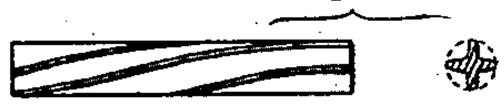


Fig. 4



Witnesses:

E. E. Masson

P. Maurø

Inventor

Paul G. L. G. Designolle by

Pollok his attorney

(No Model.) P. G. L. G. DESIGNOLLE. 2 Sheets—Sheet 2.

Method of Treating Copper Ores Containing Precious Metals.

No. 243,673. Patented June 28, 1881.

Fig. 5.

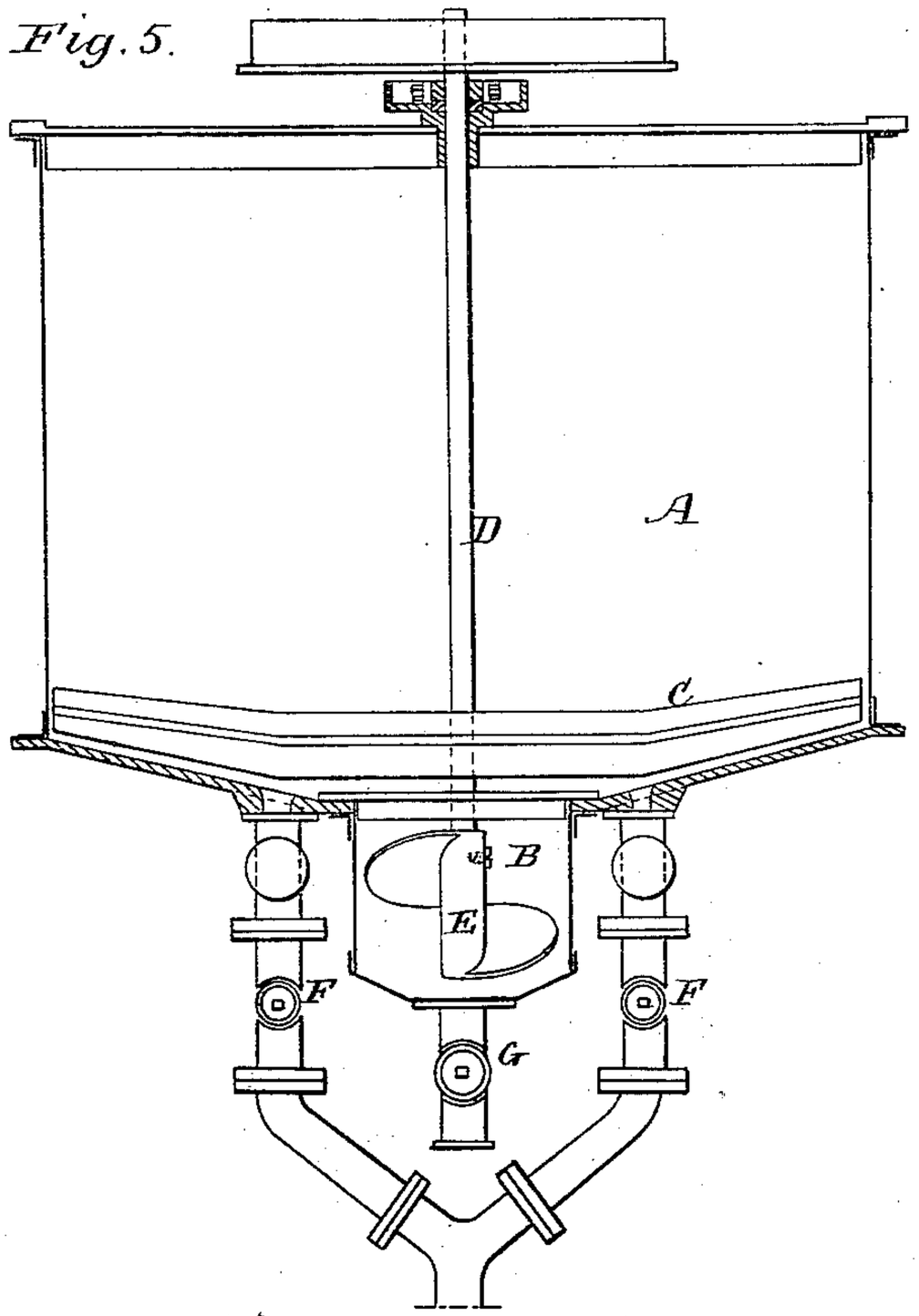
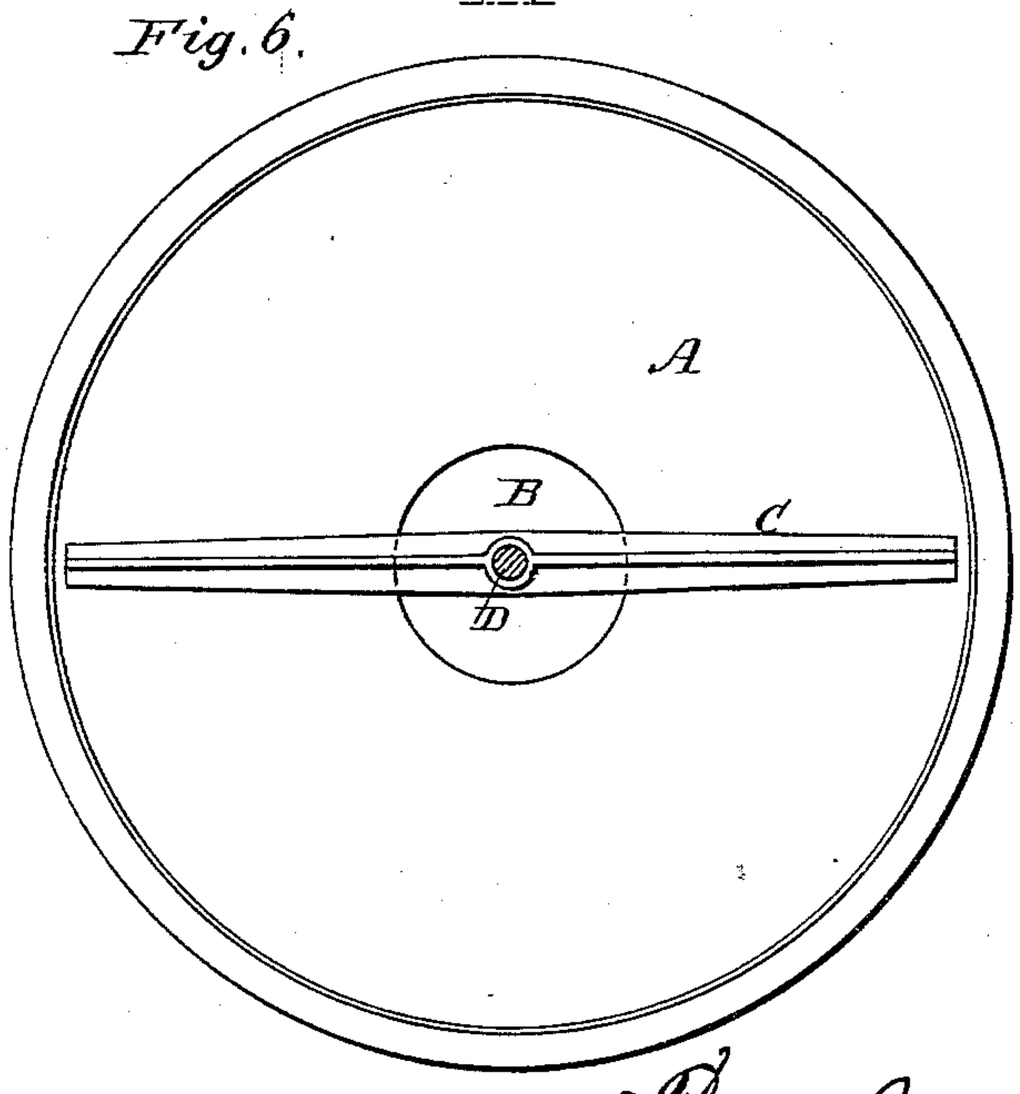


Fig. 6.



Witnesses:
E. E. Masson
P. Thauro

Inventor
Paul G. L. G. Designolle by
Pollok his attorney

UNITED STATES PATENT OFFICE.

PAUL G. L. G. DESIGNOLLE, OF PARIS, FRANCE.

METHOD OF TREATING COPPER ORES CONTAINING PRECIOUS METALS.

SPECIFICATION forming part of Letters Patent No. 243,673, dated June 28, 1881.

Application filed January 19, 1881. (No model.)

To all whom it may concern:

Be it known that I, PAUL GUSTAVE LOUIS GABRIEL DESIGNOLLE, of Paris, in the Republic of France, have invented a new and useful Improvement in Treating Copper Ores Containing Precious Metals, which improvement is fully set forth in the following specification.

This invention relates to the treatment of copper ores which contain gold and silver or other precious metals, and has for its object to extract the metals from the ores by the aid of amalgamation, and to separate the copper from the precious metals contained in the compound amalgam. It comprises as well the apparatus hereinafter described, whereby the treatment is or may be carried on, as the methods or processes constituting said treatment.

I have ascertained that in treating by the electro-chemical amalgamating process described in Letters Patent No. 228,970, granted to me June 15, 1880—that is to say, with a solution of mercuric chloride (corrosive sublimate) to which a small quantity of sodium chloride (common salt) has been added in presence of iron—copper matts or sulphureted ores containing copper and gold or silver, or both, an amalgam containing the copper and precious metals is obtained; but this amalgam is hard, pulverulent, and set in the mass or gangue if only the proportion required to produce the theoretic copper amalgam (CuHg) is employed. The amalgam, however, becomes pasty and susceptible of collecting upon amalgamated plates, provided a sufficient quantity of metallic mercury is added. I have also ascertained that the same results are obtained in treating certain other compounds of copper containing the precious metals—such as the subsulphide of copper, (Cu_2S,) the antimoniuret, the arseniuret, the sulphantimoniuret, and sulpharseniuret of copper, and even the copper oxides, (Cu_2O, CuO.) It must also be understood that the process is applicable where the ore contains only copper in one or the other of the forms mentioned without precious metal.

The first part of the invention, with relation to the amalgamation, therefore, has reference particularly to the following points, to wit: the treatment of auriferous and argentiferous copper matts with or without lead; the direct treatment of gray copper, antimoniated and arsenated, auriferous and argentiferous, after a simple roasting, without being converted into matts; the direct treatment, for copper, gold, and silver, of the material resulting from roasting certain kinds of pyrites in order to extract the sulphur for use in the manufacture of chemical products—notably the pyrites of San Domingo, Tarcis, Rio Tinto, and others of the same class; the suppression of the chlorination by salt (sodium chloride) of the above-mentioned materials, as heretofore practiced, and of the treatment with iodides to extract the silver.

The simplicity of the improved process permits the same to be applied not only to rich minerals, but also to those too poor for exportation.

As already pointed out, I have ascertained that copper pyrites containing gold or silver in quantities more or less important are amalgamable by the electro-chemical process without requiring to be roasted; but the amalgamation takes place, if possible, still more readily when almost all of the sulphur has been removed by roasting. In fact, the subsulphides of copper (Cu_2S) and the oxide as well are amalgamable electro-chemically, and yield up all the copper to mercury. It is of little consequence, therefore, if the copper sulphide be converted by thorough roasting into oxide, (Cu_2O or CuO,) since, as experience has proved, in that condition the copper is also amalgamable by my electro-chemical process. I have also ascertained that the copper can be separated from the gold, silver, or precious metals in compound amalgams obtained by treatment of auriferous or argentiferous ore by the electro-chemical process by an action of liquation, which takes place in distilling the compound amalgam. This portion of the invention is, however, also applicable to the treatment of compound amalgams obtained in any suitable way, as by trituration with metallic mercury.

The following description will enable those skilled in the art to which the invention appertains to make and use the same. The process will be explained in connection with the apparatus preferably employed in carrying it into effect and illustrated in the accompanying drawings. These drawings form a part of this specification, and in them—

Figure 1 represents, in side elevation, the

apparatus for disintegrating the ore; Fig. 2, the same in vertical section; Fig. 3, one of the crushing-rollers in elevation and cross-section; Fig. 4, one of the rollers used in the amalgamating apparatus, also in elevation and cross-section; Figs. 5 and 6, an apparatus for separating the amalgam from the gangue by difference in the specific gravity; and Figs. 7 and 8, the apparatus for distilling the amalgam, Fig. 7 being a view, in cross-section, of the crucible for containing the amalgam and chamber for receiving the crucible; and Fig. 8, a view of the crucible, receiving-chamber, and furnace in longitudinal section, and of a carriage for transporting the crucible in elevation.

The disintegrating apparatus comprises a cast-iron drum, *a*, which is supported by a shaft turning in bearings, and is provided with a gear-wheel, *b*, and a number of metallic (cast-iron) crushing-rollers, *c*, of a length nearly equal to that of the drum and of a diameter varying according to the hardness of the ore and the interior dimensions of the drum. As shown, there are five of these rollers. They are loose within the drum. A pinion on a counter-shaft, driven by the aid of suitable belt-pulleys, *b'*, engages with the gear *b* and revolves the drum *a*. The latter is provided with an opening on the periphery to admit the ore, and also with an outlet, *d*, in one end for permitting the crushed or pulverized ore to run out. The opening on the periphery is provided with suitable devices for tightly closing the same, and the outlet *d* has a gate, which is controlled by the hand-wheel *e* by means of a wheel and pinion. The outlet discharges into a funnel, *f*, which carries off the ore.

The amalgamator differs from the disintegrator mainly in the form of the crushing-rollers, which, as shown in Fig. 4, have helicoidal ribs. The object of this is to allow all the amalgam or mass containing the same to be readily drawn off from suitable openings in the lower part of the drum. The contact of the ribs with each other and with the interior of the drum being only at isolated points, spaces are left for the free passage of the material.

The separating apparatus, Figs. 5 and 6, consists of a large vessel, A, and a smaller vessel, B, placed under the larger vessel and communicating with it at the bottom, as shown in Fig. 5. In the center of both vessels is a revolving shaft, D, to which is attached, just above the bottom of the large vessel, a scraper, C, and to the lower end, which extends into the vessel B, a screw, E, of cast-iron. The large vessel is provided with outlet-pipes F, and the smaller vessel with an outlet, G. Suitable cocks control the outlets.

The distilling apparatus, Figs. 7 and 8, comprises a receiving-chamber, H, which is or may be of cast-iron, supported in a furnace, I, so as to leave free space around it for the passage of the product of combustion. The receiving-chamber has a cap or hood, O, for carrying off the products of distillation which are to be conveyed to a condenser; and both said chambers and the furnace I have doors J J' to give access to their interior and to allow the crucible K to be introduced and removed as required. This crucible is rectangular, and is provided with a hopper-shaped bottom. It has rollers L, which run upon rails M, part of which are secured to the interior of the chamber H. A carriage, N, which runs upon a track in front of the furnace, is adapted to receive the crucible, to convey it from place to place.

Having now explained the apparatus, I will proceed to describe the process.

The copper ore containing precious metals, together with a sufficient quantity of water, is introduced into the drum *a* of the disintegrating apparatus, which is then put in motion. After a suitable time the drum is stopped, and the more fluid portions in the upper part of the drum are drawn off through the outlet *d*, the large uncrushed portions of the ore remaining in the bottom of the drum for further disintegration with a new charge of ore and water. The portion drawn off is passed through several decantation or settling tanks, provided or not, as may be desired, with agitators. In these the finer particles are separated from those which have not been sufficiently reduced and the latter are returned to the drum *a*. The last tank is provided with a double perforated partition or bottom, and a layer of sponge inclosed between the two plates, whereby the solid matters, even the smallest particles, are retained, and only the clear liquid is allowed to flow off. The pulverized ore is collected, and, with a sufficient quantity of water to render the mass semi-fluid, and a suitable weight of mercuric chloride (corrosive sublimate) to which common salt has been added, is placed in the iron drum of the amalgamator.

In order to use the minimum quantity of mercuric chloride this should be in the proportions required to give one equivalent of mercury to two equivalents of copper, and, in addition, the proper amount for the precious metals. It is well to introduce into the mass in the amalgamator, in order to diminish the consumption of the metal of the drum, iron in the form of scales, or simply in the spongy condition in which it is obtained by calcining in presence of carbon ferric oxide (peroxide or sesquioxide of iron, Fe_2O_3) the residue from roasting pyrites. The amalgamator is set in operation and is revolved until the mercuric chloride has been decomposed. It will be understood that under the conditions named the resulting amalgam has not the characteristic buttery consistence of the theoretical amalgam, since the mercury in the chloride corresponds with half only of the weight of copper to be dissolved. It may be said that the amalgamation is simply begun. To complete it a quantity of metallic mercury equal to that contained in the first charge of mercuric chloride must be added. After the addition of the mercury the

rotation of the amalgamating-drum is continued until amalgamation is complete. The total weight of the metallic mercury to be added, in order to insure the good execution of subsequent operations, should be about four times greater than that necessary to obtain the theoretic amalgam, (CuHg.) The addition of this supplementary quantity of mercury has for its object to render the amalgam very fluid, to allow it to run together in the mass, and consequently to effect an almost complete separation of it from the gangue by displacement, leaving but a small portion of it to be removed from the gangue by amalgamated plates.

The treatment of copper ores gives rise to a considerable quantity of amalgam, which it would be impossible to recover upon the receiving-plates without liability to great loss, and without interfering with the good working of the apparatus, and therefore, after leaving the amalgamating apparatus the ore, instead of passing directly onto amalgamated plates for collection, is run into the separating apparatus A B, Figs. 5 and 6, already described. The materials from one or more amalgamators is introduced and a quantity of water—say about four times its volume—is added to the mass, so as to render it very fluid. The center shaft, D, is revolved, and the scraper C and screw E stir up the material. The globules of amalgam run together and fall into the lower vessel, B, where they accumulate, by reason of their greater density. The movement of the screw E in this vessel aids in washing and separating the amalgam from impurities. As the operation proceeds the amalgam continues to collect until only a few globules remain suspended in the gangue. The fluid mud is then drawn off from the upper vessel through the pipes F into a small mixing apparatus of ordinary construction placed above the collector with amalgamated plates, and in this apparatus a fresh supply of water is added. On the amalgamated plates the small remaining portions of the amalgam are collected. The collector preferably employed is one composed of a series of rotary plates alternating with a series of fixed plates, as described in Letters Patent No. 228,806, granted to me on the 15th day of June, 1880. As for the liquid amalgam which collects in the vessel B, fresh water is added and churned with it by the action of the screw E. The water removes the last impurities from the amalgam, which is, after a sufficient washing, ready to receive the final treatment in connection with that collected from the gangue by the amalgamating-plates. The amalgam thus obtained, being well washed and cleansed from impurities, is strongly compressed in a chamois-skin, so as to express as much as posssible of the mercury, and is then subjected to distillation to drive off the rest of this metal associated with the copper, gold, or silver. It is in this distilling operation that the separation of the copper and precious metals is effected—a separation which is based upon a scientific principle, so far as I am aware entirely new, and of which the explanation is as follows:

Whenever a compound amalgam containing copper in connection with gold, silver, platinum, and other simple bodies of the series is subjected to distillation in a suitable vessel, the precious metals become separated and occupy the bottom of the vessel, while the copper in the form of a spongy mass occupies the upper part. During the distillation then a true phenomenon of liquation of the amalgam takes place, a phenomenon not heretofore observed or usefully applied. Copper amalgam, when heated to about 300° centigrade, possesses the property of being swelled or puffed out as the small bubbles of mercury escape, while the amalgams of gold, silver, platinum, &c., abandon the mercury associated with them without swelling. When, therefore, a compound amalgam, such as indicated above, in which copper predominates is subjected to distillation, the mass liquefies a little under 100° centigrade, and the amalgams place themselves one above another in the order of their density, the copper amalgam being at the top, for there is no chemical combination of the different amalgams, but a simple mixture. When the temperature reaches 300° centigrade the copper amalgam is swelled considerably by the drops or bubbles of escaping mercury, which begin to volatilize; but the amalgams of precious metals remain liquid and occupy the bottom of the vessel. If the distillation be then pushed, the swelled amalgam does not change its state. It loses its mercury, leaving at last a spongy body, clearly separated from the precious metals, which, also deprived of the mercury previously associated with them, occupy the bottom of the vessel. The spongy copper being easily separated by a knife, it only remains to cut through the mass, following the clear line of demarkation between the copper and precious metals. Being chemically pure, the copper is then melted and run into bars ready for sale; or, if it be desired to avoid melting the copper, it can be agglomerated into ingots by strong pressure. The precious metals containing yet a small proportion of copper are in that state best adapted for refining. For the complete success of the operation the copper amalgam should predominate, should indeed be largely more (one hundred times greater) than the amalgam of gold and silver. The compound amalgam, before being distilled, should be strongly compressed, and the distillation should from the beginning be conducted slowly and with great care, the temperature during the whole operation not passing 400° to 500° centigrade.

In carrying on the distillation with the apparatus described (see Figs. 7 and 8) the amalgam is placed in crucible K, and this is run into the receiving-chamber H, wherein the distillation takes place. The interior of the crucible is lined with ferric oxide, in order to prevent adherence between the cake of copper

and the walls of the crucible. The doors being closed, the operation begins. The mercury vapors as they distill are carried off through the cap O to the condenser. When the distillation is ended the precious metals will be found to have accumulated in the hopper-shaped bottom, while the copper in a spongy state rests in the upper part. The cake of copper is removed entire by cutting it from the inverted truncated pyramid composed of precious metals.

It is obvious that other forms of apparatus than those described can be used for carrying out the process. For example, the pulverization of the ore can be effected in the dry or wet state by crushing machinery of any ordinary or suitable construction that will reduce the ore to the desired degree of fineness, and other amalgamating apparatus containing iron can be employed. The crushing and amalgamating apparatus described are considered best adapted to the purpose. They are not claimed herein, but are reserved for separate application.

Having thus fully described the said invention and the manner of carrying the same into effect, what I claim, and desire to secure by Letters Patent, is—

1. The method of amalgamating ores or other materials containing copper, copper oxides, copper salts or sulphides, antimoniurets or arseniurets of copper, by applying thereto the electro-chemical amalgamating process—that is to say, by triturating the pulverized copper ore with mercuric chloride with addition of chloride of sodium in presence of metallic iron—and employing in said process a proportion of mercury, such as indicated, so as to obtain the copper amalgam in condition for collection, as hereinbefore described.

2. The method of amalgamating copper ores by beginning the amalgamation by the electro-chemical process, and then adding metallic mercury in the proportion substantially as described, so as to obtain a pasty amalgam capable of agglomerating and collecting, as set forth.

3. In the amalgamation of copper ores, the method of effecting the separation of the amalgam from the gangue by rendering the amalgam pasty or fluid with mercury, separating the larger part by displacement in a suitable apparatus and recovering the remainder upon amalgamated plates, substantially as described.

4. The method of separating copper from precious metals contained in a compound amalgam by simple distillation, giving rise to the phenomenon of liquation, substantially as described.

5. The method of recovering copper and precious metals from copper ores by forming a pasty or fluid compound amalgam of the metals, as described, separating the greater part of said amalgam by displacement, and collecting the rest upon amalgamated plates, distilling the amalgam, as explained, and dividing the spongy copper from the precious metals.

6. The distilling apparatus comprising a receiving-chamber with cap for carrying off the products of distillation, crucible with hopper-shaped bottom, rollers supporting said crucible, and track on the interior of the receiving-chamber, substantially as described.

In testimony whereof I have signed this specification in the presence of two subscribing witnesses.

PAUL GUSTAVE LOUIS GABRIEL DESIGNOLLE.

Witnesses:
EUG. DUBOIS,
R. CHAPITAT.