C. M. T. du MOTAY.

Manufacture of Baryta.

No. 144,517. Patented Nov. 11, 1873.

fig. 1

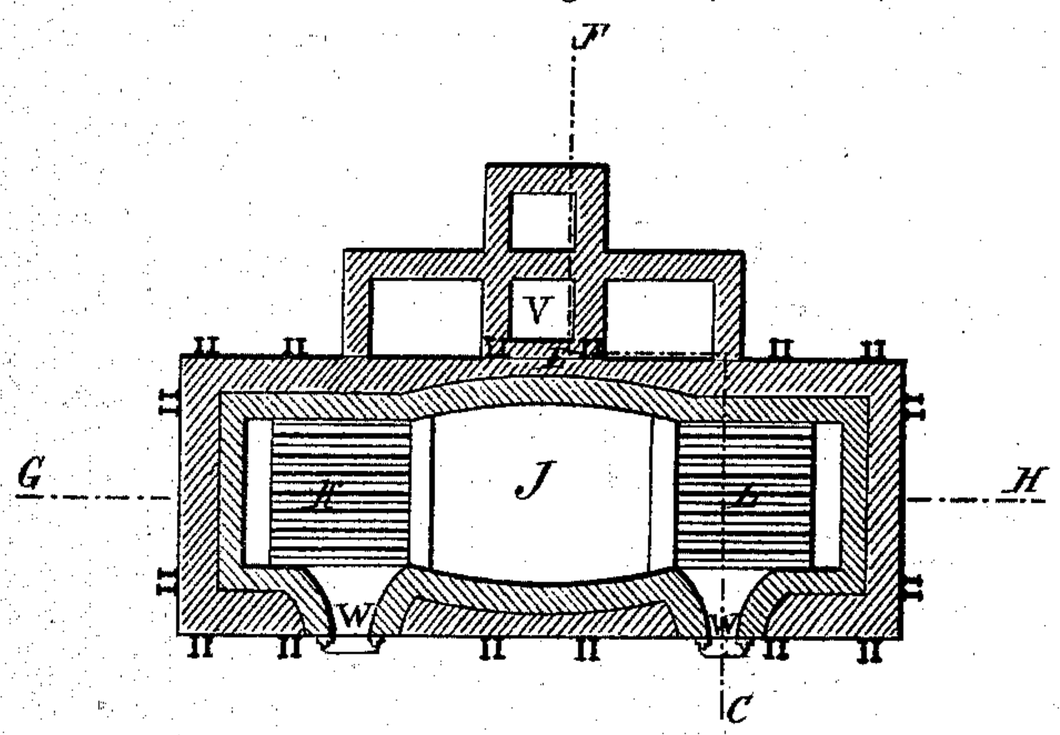


fig. 4

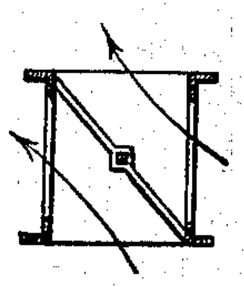


fig. 2.

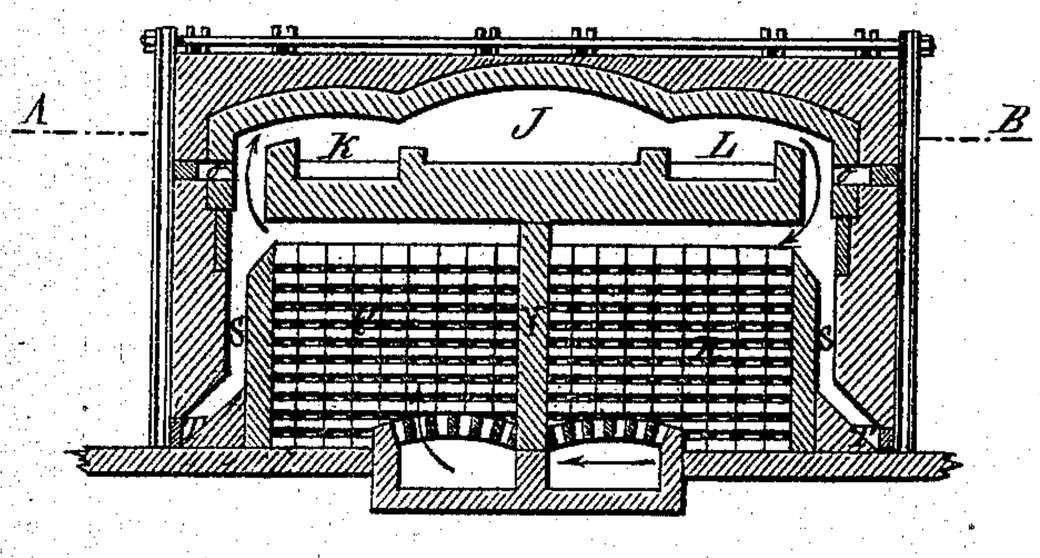
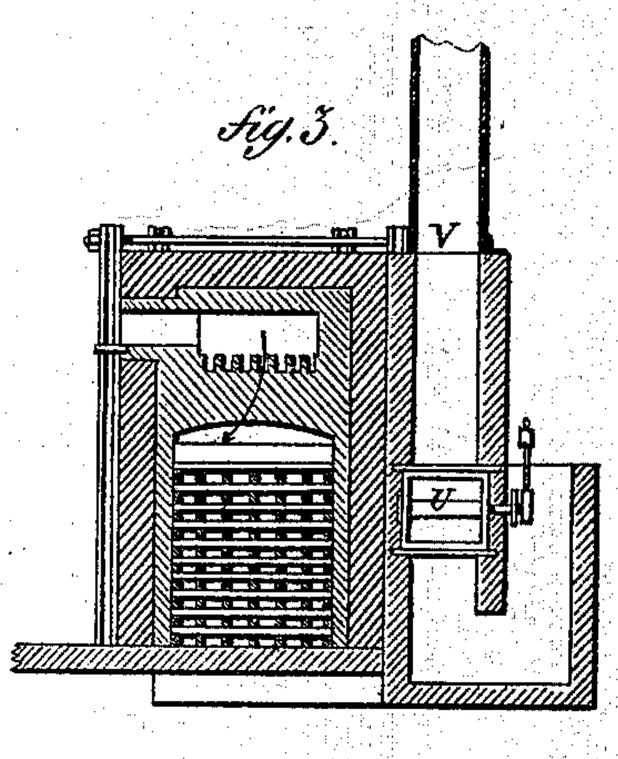


fig. 3.



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CYPRIEN M. T. DU MOTAY, OF PARIS, FRANCE.

IMPROVEMENT IN THE MANUFACTURE OF BARYTA.

Specification forming part of Letters Patent No. **144,517**, dated November 11, 1873; application filed April 10, 1873.

To all whom it may concern:

Be it known that I, CYPRIEN MARIE TESSIÉ DU MOTAY, of Paris, in the Republic of France, have invented a new and Improved Process of Manufacturing Baryta, and apparatus employed therein; and I do hereby declare the following, when taken in connection with the accompanying drawings and the letters of reference marked thereon, to be a full, clear, and exact description of the same, and which said drawings constitute part of this specification, and represent, in—

Figure 1, a plan of the furnace; Fig. 2, a section on line G H; Fig. 3, a section on line C D E F; and Fig. 4, a partial view of the valve.

The two natural minerals of baryta are the sulphate of baryta and the carbonate of baryta. The present invention relates to new economical and practical means of producing this substance. It is necessary previously to bring sulphate of baryta, which is the more abundant of these minerals, to the state of sulphuret of barium. For this purpose I have constructed a special furnace, the description of which is equally applicable to the other process of the transformation of the carbonate of baryta into baryta. In the second place, the different methods which I have discovered for the extraction of the baryta from the sulphuret of barium will be explained.

The reverberatory furnace, from its nature, is powerless to furnish at the same time an elevated temperature and a reducing-flame; but in order to bring about the complete reduction of the sulphates and carbonates of baryta, these two chemical and physical conditions must be absolutely fulfilled, for, under the action of oxidizing-flames and corresponding temperature, it is impossible to avoid inverse reactions, which retransform the sulphurets into sulphates and the baryta into carbonate. These inconveniences are suppressed in my system of furnace, which is founded upon the application of the principle of recurrence, in order to produce and store up very high temperatures, which permit the continuous employment of reducing-flames.

The furnace consists of a chamber, J, heated by two hearths, K L, the grates of which are formed from bars of refractory earth, forming between them a receptacle, into which the cinders fall. These cinders are withdrawn by scrapers, which are introduced through openings O O, and finally fall into the cinder-bed S, from which they are removed through an opening, T. The hearths are each in communication with recurrences or superposed chambers Q R, which are separated by a wall, Y. The recurrences are, in their turn, in communication with a valve, U, and the chimney V, which is provided with a register. The fuel is introduced upon the hearths through doors W W. Air enters through the valve U, and, after having traversed one recurrence, Q, it arrives hot in one of the hearths, K. The combustion proceeds and the flame produced passes across the top surface of the furnace J, then above the second hearth, L, and goes to reheat the other recurrence, R, from thence proceeding away through the chimney V. At the end of about an hour's work the position of the valve U is reversed and fuel is fed into the other hearth, L, which, in its turn, receives the heated air proceeding in an opposite direction. Instead and in place of solid combustibles burning in the heaters of the furnace just described, the gases generated in gasogenes may be advantageously substituted. This furnace is applied to the manufacture of sulphuret of barium by the reduction of the sulphate of baryta, and to the manufacture of baryta by the reduction of carbonate of baryta.

The description of these operations successively is as follows:

Sulphuret of barium.—The reduction taking place at red heat, it is necessary, in order to obtain the reducing temperature, to heat the furnace, before charging it, for at least an hour. It is then charged with sulphate mixed with from thirty to forty per cent. of coal, the recurrences are put in action and are charged every two hours, the matters being stirred at each charge. At each stirring it is necessary to take care to charge the grate with coal, in order to obtain a great excess of carbureted hydrogen and oxide of carbon in the furnace. The reduction is complete at the end of seven or eight hours, so that three operations may be made in twenty-four hours. The product is cooled in water, or, preferably, is placed in closed boxes, in order to avoid the production, through contact with the air, of a certain

quantity of hyposulphite of baryta and other analogous products. The reduction of the precipitated sulphates is complete; that of natural sulphates is from eighty-seven to ninety per cent. With an ordinary reverberatory furnace more than sixty-five per cent. is never obtained. The consumption of coal is also considerably reduced.

Caustic baryta.—The operation is effected in nearly the same conditions as the preceding. As the temperature of the recurrential furnace must be raised, it is necessary before the charging to heat the furnace with the cinder-box open, and to a white heat, for from one and one-half to two hours. The carbonate is then changed mixed with thirty per cent. of coal, and the recurrences are brought into play, and they must be charged once an hour, or, at least, once every hour and a half. The atmosphere consists almost entirely of carbureted hydrogen and oxide of carbon, which will take fire at every opening of the furnace; a little excess of coal is therefore necessary. At each change the furnace is stirred, and the operation lasts eight hours, or three operations to twenty-four hours.

Transformation of the sulphuret of barium into carbonate of barium and baryta.—The sulphuret of barium obtained can, in its turn, be converted either into carbonate of baryta, or directly into baryta.

Carbonate of baryta.—In order to transform the sulphuret of barium into carbonate of baryta, a current of carbonic acid may be simply caused to pass through the sulphuret in solution; but in this case, during the decomposition, hyposulphite of barium is produced, which, mixed with the carbonate and acted upon by the carbon, reproduces sulphuret of barium in sufficient quantity to insure the purity of the baryta to the extent of ninety-seven per cent. of baryta for three per cent. of sulphuret. In order to obtain an absolutely-pure carbonate, I commence by boiling in equal weights sulphuret of barium with chloride of magnesium. Chloride of barium and hyposulphuret of magnesium are produced in equal quantities, which decompose during the boiling into magnesia and sulphydric acid. The chloride of barium thus formed remaining in the presence of the magnesia, is treated with water, and it is into the bath thus formed that I pass a current of carbonic acid, until the complete transformation of the magnesia into bicarbonate of magnesia, which, in reacting while hot upon the chloride of barium, produces carbonate of baryta and chloride of magnesium. This chloride serves again to convert into chloride of barium another equivalent quantity of sulphuret of barium. The carbonate of baryta being insoluble is easy to separate. It is found in an absolutely-pure state, and is suitable not only to producing baryta, but also for employment in the manufacture of crystal instead of oxide of lead.

Baryta.—Two methods are used for the conversion of the sulphuret of barium directly into baryta, which possess the advantage of expelling all the sulphur contained in the sulphurets in the form of sulphhydric acid, which can be employed for the production of sulphur or sulphurous acid.

First method.—One equivalent of sulphuret of barium being given as an example, the sulphuret is dissolved in water, and boiled with one equivalent or more of oxide of lead, until when, by double exchange, all sulphuret of the alkaline sulphuret is carried to the lead, while the oxygen, on the other hand, is combined with the barium, in order to form caustic baryta. The barytic solution is put on one side and collected. The insoluble residue of the sulphuret of lead is then treated with one equivalent of hydrochloric acid, which transforms the lead into chloride of lead. This chloride is mixed with one equivalent of magnesia or lime, and the necessary quantity of water, for the transformation of the chloride of lead into oxide of lead, and the magnesia or the lime into chloride of magnesium or calcium. The oxide of lead regenerated is suitable to decompose a fresh equivalent of sulphuret of barium. The chloride of magnesium or calcium is put on one side in order to reproduce the failing case of hydrochloric acid by one of the methods devised by me for that purpose. The sulphhydric acid is wholly or partly burnt in special chambers, either in order to generate sulphur or to produce sulphurous acid. In putting this method into practice it is preferred, instead of the oxygen, to employ oxide of zinc or the hydrate of the oxide of zinc, because the sulphur of zinc is more easily attackable by the hydrochloric acid than it is by the oxide of lead. Instead of the oxide of lead as the intermediate agent it is preferred to use oxide of zinc. In this case, in order to cause the sulphuret of zinc to return to the state of oxide, recourse is had to the combined and new employment of the following reactions. A current of air in excess, or oxygen, or, better still, of air in excess and sulphurous acid and steam, with or without the intervention of nitrous vapors, is made to pass upon the sulphuret of zinc at a dull-red heat. The sulphur is partly or wholly transformed into sulphate, which, boiled with chloride of potassium or sodium, gives sulphate of potash or soda and chloride of zinc. This chloride is then decomposed by magnesia or lime and the oxide of zinc is revivified. When the whole of the heated sulphuret of zinc has not been transformed into sulphate the oxygen remaining after the washing of the sulphate is suitable for the direct transformation of the sulphuret of barium into baryta, as explained above. Whence it will be perceived that whatever may be the result of the treating of the sulphurets of zinc at a low temperature the oxides of this metal are ceaselessly regenerated, and are suitable for use again in the transformation of the sulphuret of barium into hydrate of baryta.

Second method.—Dry plan.—In the recur-

rential furnaces, with blown gas at the temperature of fusion of the steel or nickel, the sulphurets of baryta and the carbonates are decomposed and caustic soda is produced. But this phenomenon of decomposition can only be industrially employed upon foundations of magnesia, dolomite, or lime completely sheltered from contact with silica or alumina, for the baryta at the temperature at which it is produced combines with these two bodies so as to form insoluble compounds. The sulphuret of barium, mixed with one or two equivalents of sulphate of baryta or sulphate of lime, is also decomposed in recurrential gas-furnaces and brought to the state of caustic baryta. The sulphites, hyposulphites of baryta, and other salts of the series, undergo in the same circumstances the same transformation.

I claim as my invention—

1. The combination of the chamber J, the two hearths K L, chambers Q R, valve U, and chimney V, all substantially as and for the purpose described.

2. The methods herein described of revivifying and utilizing indefinitely compounds and intermediate re-agents, to transform the sulphuret of barium into hydrated baryta or into carbonate of baryta.

In testimony whereof I have signed my name to this specification before two subscribing witnesses.

C. M. TESSIÉ DU MOTAY.

Witnesses:

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ALBERT CAHEN.