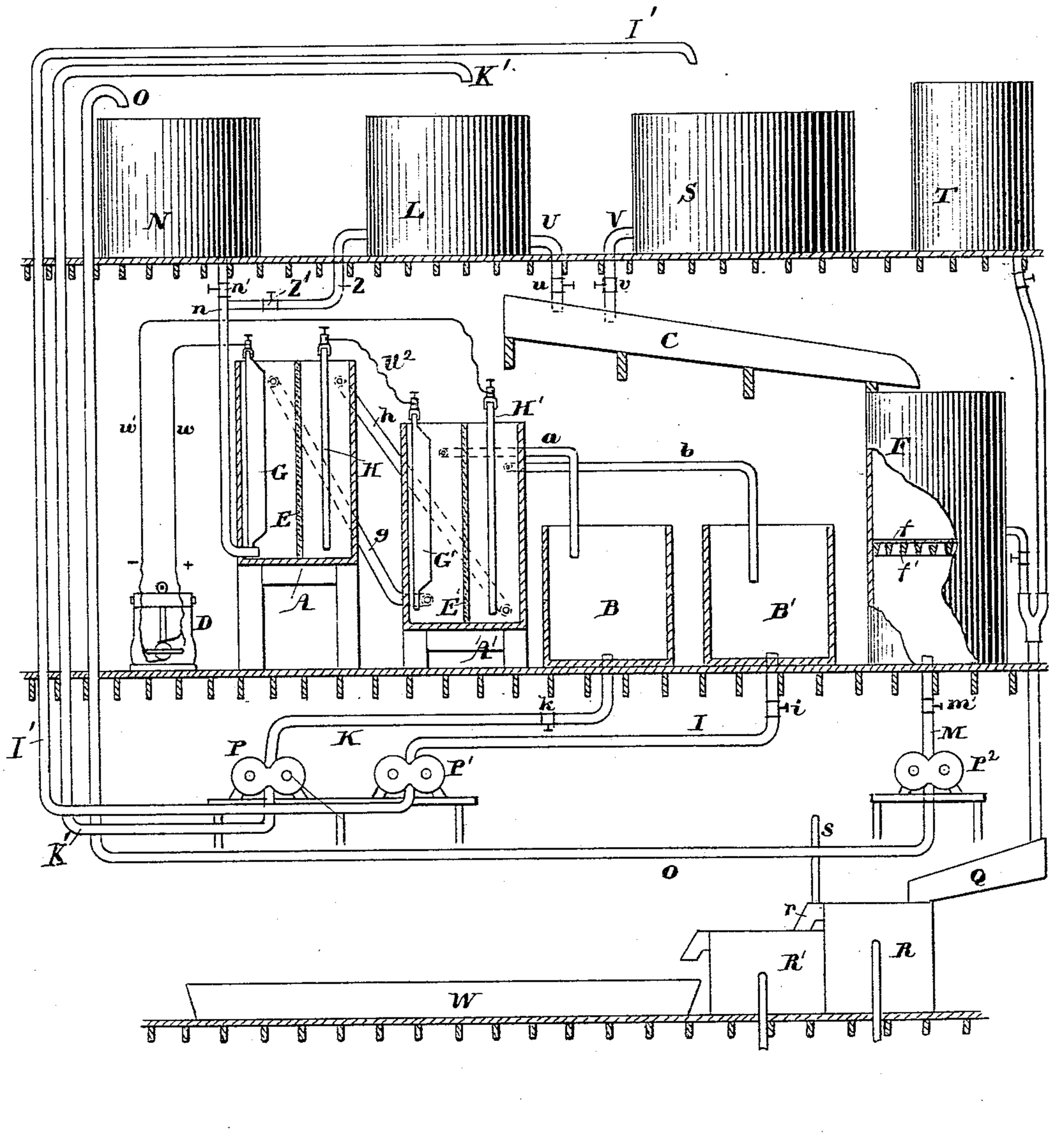
(No Model.)

A. B. BROWNE & E. D. CHAPLIN.

PROCESS OF MANUFACTURING WHITE LEAD BY ELECTROLYSIS.

No. 555,232. Patented Feb. 25, 1896.



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UNITED STATES PATENT OFFICE.

ARTHUR BENJ. BROWNE, OF CAMBRIDGE, AND EDWIN D. CHAPLIN, OF NATICK, MASSACHUSETTS, ASSIGNORS TO THE AMERICAN LEAD COMPANY, OF KITTERY, MAINE.

PROCESS OF MANUFACTURING WHITE LEAD BY ELECTROLYSIS.

SPECIFICATION forming part of Letters Patent No. 555,232, dated February 25, 1896.

Application filed July 2, 1894. Serial No. 516,384. (No specimens.)

To all whom it may concern:

Be it known that we, ARTHUR BENJ. BROWNE, of Cambridge, and EDWIN D. CHAPLIN, of Natick, in the county of Middlesex and State of Massachusetts, have invented certain new and useful Improvements in Processes of Manufacturing White Lead by Electrolysis, of which the following, taken in connection with the accompanying drawings, is a specification.

Our invention relates to the manufacture of white lead by electrolysis; and it consists in certain novel methods or processes of operation, which will be readily understood by reference to the description hereinafter given and illustrated by the accompanying drawings, and to the claims hereto appended and in which our invention is clearly pointed out.

It is well known that when lead is dissolved in nitric acid nitrous gas is liberated during the decomposition of the nitric acid. Now in the electrolytic process described in the Letters Patent No. 496,109, granted to Arthur Benj. Browne April 25, 1893, it is obvious that owing to the partial decomposition of the nitric acid a portion of it would escape from the solution, leaving the sodium with which it was combined (as nitrate) in the state of hydrate. It is also apparent that the solution would very soon become alkaline, owing to the absence of the portion of the nitric acid thus lost.

It is further found in practice that the resistance of the solution (with its consequent loss of electric energy) is very largely decreased by performing only a partial precipitation, thus retaining a certain quantity of lead nitrate in solution.

In order to accomplish this and other hereinafter-described results we make use of any form of apparatus suitable for the purpose. One form of such an apparatus is shown in the accompanying drawing in sectional elevation, in which A and A′ represent a plurality of electrolyzing-tanks set upon different levels and provided with the porous diaphragms E and E′, of clay or other suitable material. These diaphragms divide the electrolyzers into two compartments each.

The two compartments of the electrolyzer A are connected to the compartments of the electrolyzer A′ by the pipes *g* and *h*, as shown.

B and B′ are tanks or reservoirs into which the solutions flow from the two compartments of the electrolyzer A′ by means of the pipes *a* and *b*.

N, L, and S are the supply-tanks.

C is a chute.

F is a filter.

P, P′, and P^2 are pumps, and D is a dynamo or other source of electric energy, and *u*, *u′*, and u^2 are the conducting-wires connecting the electrolyzers with said dynamo.

G and G′ are anodes of metallic lead to be acted upon, while H and H′ are the cathodes and may be of any conducting material which is insoluble in the solutions used.

The apparatus shown in the drawing and partially described, as above, illustrates a convenient means of carrying out our improved process, but is not essential thereto, as our process may be performed by other arrangements of apparatus—such, for instance, as is shown in part in the Letters Patent No. 415,576, of November 19, 1889, or more completely in another application of ours filed at the same time with this application, Serial No. 516,385—or the process may be conducted in several simple vessels, the manipulation being performed by hand.

In working our invention the electrolyzers A and A′ and the supply-tank N are filled with a solution of nitrate of soda, and then a current of electricity is passed through each of the electrolyzers and the solution of nitrate of soda is allowed to flow from said supply-tank N to and through the anode-compartment of the several electrolyzers and into the tank B.

The solution of nitrate of soda during its passage through the electrolyzer A has, it is obvious, become partially decomposed, nitrate of lead being formed in the anode-compartment and sodium hydrate in the cathode-compartment. As the nitrate of soda, now mixed with nitrate of lead, passes through the pipe *g* into and through the anode-compartment of the electrolyzer A′, it is still further decomposed and an additional quantity of lead nitrate is formed.

The diaphragms E and E′ being of a porous nature the cathode-compartment is filled and caused to overflow through the pipe *h* by the percolation of the solution through said

diaphragms. Thus the mixture of nitrate of sodium and sodium hydrate in the cathode-compartment of electrolyzer A overflows into and through the cathode-compartment of the electrolyzer A′, and thence through the pipe *b* into the tank B′.

When the tanks B and B′ hold a sufficient amount of their respective solutions, the valves *i* and *k* are opened and the lead-nitrate mixture is pumped into tank L by means of the pump P and pipes K and K′, while the sodium-hydrate mixture is pumped into the tank S by means of the pump P′ and the pipes I and I′.

Portions of the lead-nitrate mixture and the sodium-hydrate mixture are then withdrawn from their respective supply-tanks L and S by opening the valves *u* and *v* in the pipes U and V, respectively, and mixed in the trough or chute C, resulting in a precipitation of lead hydrate and the formation of a solution of nitrate of soda. This precipitate is filtered out on the filter *f*, supported by the perforated false bottom *f′* in the tank F. The filtrate is then pumped back into the tank N by means of the pump P^2 and the pipes M and O, to be used over again in the manner herein described, the excess of the sodium hydrate being retained in the sodium-hydrate tank S, and may be disposed as the operator desires.

If it is desired, for the purpose set forth, to maintain a certain amount of lead in solution in the electrolyzers, this additional amount is supplied by opening the valve Z′ in the pipe Z, thereby permitting a quantity of the lead nitrate in the tank L to flow into the pipe *n* and thence into the anode-compartment of the electrolyzer A in mixture with the nitrate-of-soda solution.

When the lead is precipitated, separated, and filtered from the resulting solution, it is in the form of lead hydrate and is then treated with a solution of a carbonate of soda, which may be allowed to flow from the tank T and mix with said lead hydrate as it is drawn from the filter-tank F and discharged into the spout Q, from which the hydrated lead carbonate is discharged into the filter R and separated from the resulting solution of sodium hydrate. The hydrated carbonate of lead is discharged from the filter R upon the spout *r*, and it is washed with water from any suitable source of supply, as the pipe *s*, and discharged into the filter R′, where it is filtered from the water and discharged upon the pan W to be dried.

It will be seen from the foregoing that a continuous circulation of the solution is maintained, the metallic lead of the anodes is constantly being dissolved by the action of the electric current and held in solution, a portion of it is precipitated and drawn off, and another portion is reconducted back into the electrolyzers, thereby constantly maintaining a certain quantity in solution. By this means the solution of nitrate-of-lead mixture may be maintained at definite densities in the several electrolyzers, though of different densities in the different cells. We have herein described the carrying out of our process as performed through the medium of the apparatus shown in the accompanying drawing as a convenient and perhaps the best means for the purpose; but the successful operation of our process does not depend upon the employment of the apparatus as shown, as the same results might be successfully obtained, though perhaps not so economically, if the pumps, pipes and valves were dispensed with and the solution were transferred from one tank or receptacle to another throughout the operations, in the order described, by hand by the use of buckets or other suitable vessels.

What we claim as new, and desire to secure by Letters Patent of the United States, is—

1. The process of manufacturing white lead by electrolysis, which consists in flowing a solution of nitrate of soda through a plurality of vessels; subjecting said solution in each of said vessels to the action of an electric current passing from an anode of lead to a suitable cathode, whereby a quantity of lead nitrate is diffused from the metallic-lead anode and held in solution in each of said vessels thereby forming a mixture of nitrate of soda and nitrate of lead; mixing a portion of said mixed nitrates of lead and soda with sufficient sodium hydrate, in a separate vessel, to cause precipitation and form lead hydrate, and then filtering and carbonating said lead hydrate.

2. The process of manufacturing white lead by electrolysis, which consists in flowing a solution of nitrate of soda through a plurality of vessels; subjecting said solution in each of said vessels to the action of an electric current passing from a metallic-lead anode to a suitable cathode, whereby a quantity of lead nitrate is diffused from the lead anode and held in solution in each of said vessels thereby forming a mixture of nitrate of soda and nitrate of lead; mixing a portion of said mixed nitrates of lead and soda with sufficient sodium hydrate, in a separate vessel, to cause precipitation and form lead hydrate; filtering, carbonating, and washing the same; mixing a portion of said mixed nitrates of soda and lead with a fresh quantity of nitrate of soda, and returning said mixture to the electrolyzers, thereby maintaining the electrical resistance of the solution substantially uniform.

In testimony whereof we have signed our names to this specification, in the presence of two subscribing witnesses, on this 29th day of June, A. D. 1894.

ARTHUR BENJ. BROWNE.
EDWIN D. CHAPLIN.

Witnesses:
N. C. LOMBARD,
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