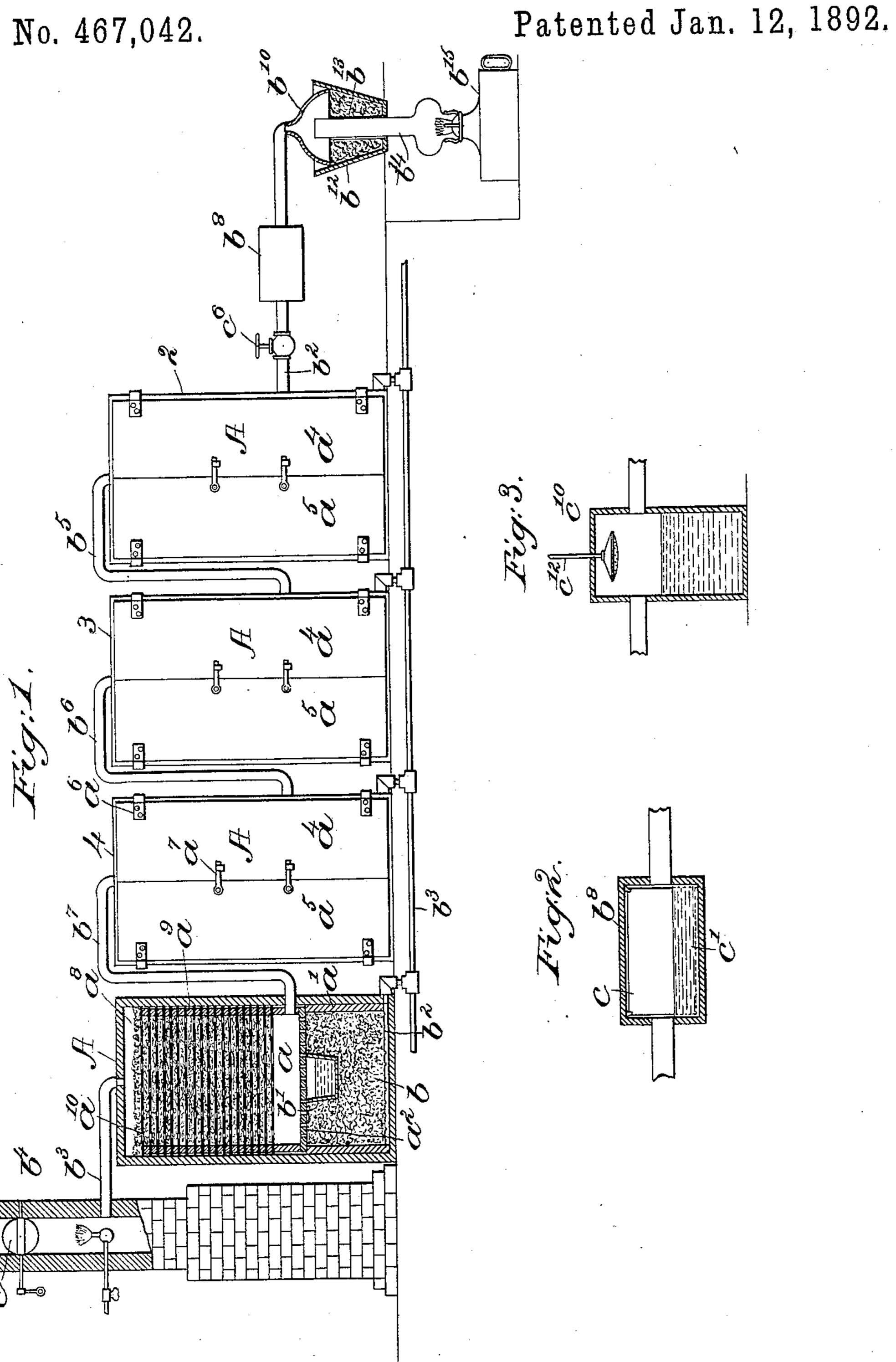
(No Model.)

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METHOD OF AND APPARATUS FOR PRODUCING LEAD CARBONATE.



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## United States Patent Office.

NORMAN K. MORRIS AND JOHN W. BAILEY, OF DENVER, COLORADO.

METHOD OF AND APPARATUS FOR PRODUCING LEAD CARBONATE.

SPECIFICATION forming part of Letters Patent No. 467,042, dated January 12, 1892.

Application filed March 31, 1891. Serial No. 387,105. (No model.)

To all whom it may concern:

Be it known that we, NORMAN K. MORRIS and JOHN W. BAILEY, both of Denver, county of Arapahoe, State of Colorado, have invented an Improvement in Methods of and Apparatus for Producing Lead Carbonates, of which the following description, in connection with the accompanying drawings, is a specification, like letters on the drawings representing like parts.

This invention relates to a novel method of and apparatus for the manufacture of metallic carbonates, especially lead carbonate

or white lead.

Prior to our invention we are aware that lead carbonate or white lead has been made by means of carbonic-acid gas acting upon the lead in the presence of acetic-acid vapor.

Lead carbonate manufactured in a stack 20 by what is known as the "Dutch" method, in which the carbonic-acid gas is made from tan-bark, &c., possesses superior qualities; but the objection to this method is the length of time required to convert the metallic lead 25 into the form of the carbonate. Metallic lead has also been converted into the carbonate in a much quicker time than what is required by the Dutch method by means of carbonicacid gas generated by the combustion of pe-30 troleum. This method, although much quicker than the Dutch method, does not ordinarily produce a superior quality of lead carbonate owing to the drying out of the metallic lead.

This invention has for its object to produce a superior quality of lead carbonate in a substantially short time, whereby the advantages of the old Dutch method and of the petroleum method are combined.

In accordance with this invention, the lead to be converted into the carbonate is placed in a corroding-chamber in a finely-divided condition, preferably in the form of thin strips or fibers, and is therein acted upon by a current of cooled hydrous carbonic-acid gas with an oxidizing agent, which for the best results consists of vapors of acetic acid, preferably commingled with the hydrous carbonic-acid gas before being brought into contact with the lead, the said gas and vapor before entering the corroding-chamber being cooled to about 95° Fahrenheit, and the tem-

perature of the corroding-chamber is maintained at about 95° to 105° Fahrenheit, so that the danger of drying out the metallic lead or 55 stock is prevented, for we have ascertained by a series of long-continued experiments that if the metallic lead is subjected to a too high temperature spots are formed on the surface of the lead which resist the action of the 60 carbonic-acid gas and acetic-acid vapor and cannot be subsequently corroded. These spots give rise to lumps of metallic lead in the carbonate and deteriorate the quality of the carbonate produced. The formation 65 of these spots is not, so far as our experiments show, prevented by the presence of an excess of watery vapor generated by heating water or even acidulated water; but we are led to believe by the results of our experi- 70 ments that water formed by the combustion of refined petroleum-oil or other unsaturated or saturated hydrocarbon—such, for instance, as gasoline, kerosene, natural gas, naphtha, alcohol, &c.—has a peculiar property itself 75 or imparts to the carbonic-acid gas produced by the combustion of the above-mentioned bodies, the property of more energetically acting upon the metallic lead, for the lead carbonate produced in accordance with this in-80 vention is of a beautiful white carbonate of creamy consistency and free from particles of metallic lead, so that subsequent treatments, such as grinding, are entirely dispensed with and the carbonate may be taken 85 from the corroding-chamber and used without any preparatory treatment.

The corroding-chamber referred to will preferably be made in the form of a stack and will preferably contain in its lower porgotion tan-bark, cow-dung, or like material now commonly employed in the Dutch method, and preferably a series of such stacks will be connected together for a circulation of gas and vapor from one to another, as will be described, the circulation being effected by means of the draft in a chimney, thereby dispensing with a circulating-pump and cheapening the cost of the apparatus, the said draft being controlled by a suitable damper.

The bottom or lower chamber of each stack may and preferably will have located in it a steam-pipe, by which the tan-bark or like material may be heated.

The particular features of our invention will be pointed out in the claim at the end of

this specification.

Figure 1 represents in section and elevation 5 an apparatus embodying this invention and with which the improved method may be carried into effect; Fig. 2, a sectional detail of the gas-cooling apparatus shown in Fig. 1, and Fig. 3 a vertical section of a modified 10 form of cooling apparatus.

Referring to Fig. 1, four stacks or corroding-chambers A are shown and numbered 2 3 4 5, and the said stacks being alike we will

specifically describe but one.

Each stack A, made of wood or other suitable material, is divided into an upper compartment  $\alpha$  and a lower compartment  $\alpha'$ , as herein shown, by a perforated wall or partition  $a^2$ , resting, as shown, upon supports  $a^3$ , 20 which may be bars, rods, posts, or of any

other suitable construction.

The stack A is provided with, preferably, side doors  $a^4 a^5$ , hinged, as at  $a^6$ , to the main frame of the stack and secured together by one or 25 more latches  $a^7$  or by any other suitable form of locking device. The compartment a of each stack contains within it the metallic lead to be converted into the carbonate, which metallic lead is in a finely-divided form, prefer-30 ably in strips or fibers represented by  $a^8$ , the said finely divided lead being supported upon a series of trays, preferably composed of a wooden frame  $a^9$  and a body  $a^{10}$  of fibrous material, such as cords or strings, but which may 35 be made of any material not effected by the corroding agents, and which will not discolor or otherwise injure the carbonate. The compartment a' of the stack is partially or wholly filled with tan-bark, cow-dung, or like material 40 b, in which is placed a vessel b', to contain acetic acid or water acidulated with acetic acid, and the tan-bark or like material is heated, as herein represented, by a steam-pipe  $b^2$ , preferably provided with perforations and 45 connected to a supply-pipe  $b^3$ , which commu-

nicates with a boiler or other source of supply. The compartment a of the first stack (marked 2) has an inlet-pipe  $b^2$ , and the compartment a for the last stack has an outlet-50 pipe  $b^3$ , communicating with a chimney  $b^4$ , and the compartments a of the adjacent stacks are connected together by pipes  $b^5$ 

 $b^6 b^7$ .

The inlet-pipe  $b^2$  for the stack marked 2 is 55 connected to or communicates with the source of supply for carbonic-acid gas, it being herein shown as connected directly to a cooling apparatus or chamber  $b^8$ , placed in the inletpipe  $b^2$ , the latter being provided with a bell-60 shaped mouth  $b^{10}$ , projecting down within a vessel or chamber  $b^{12}$ , having a central pipe or tube  $b^{13}$ , up into and preferably beyond or above which is projected the chimney  $b^{14}$  of a lamp  $b^{15}$ . The vessel  $b^{12}$  contains water, or 65 it may be dilute acetic acid, which is vaporized by the heat from the products of com-

bustion passing up through the chimney  $b^{14}$ , and the vapor thus formed commingles with the products of combustion and passes into the corroding-compartment of the stack A, 70 (marked 2,) the said vapors and products of combustion being cooled on their passage through the cooling apparatus  $b^{8}$ , the latter, when made, as shown in Figs. 1 and 2, containing suspended therein a number or se-75 ries of strips or sheets c of flannel or other absorbent material which dip into a bath c'of water, or it may be dilute acetic acid. The lamp or burner  $b^{15}$  preferably contains refined petroleum or other hydrocarbon, or it may be 80 alcohol or equivalent body, which is decomposed by combustion into a hydrous carbonicacid gas—that is, carbonic-acid gas and a plurality of molecules of water, which reaction may be best represented as follows, viz: 85  $2C_6H_6+(3O)O=12CO_2+6H_2O$ .

The hydrous carbonic-acid gas formed as above represented may and preferably will be augmented with watery vapor arising from the vessel  $b^{12}$ , and the temperature of the gas 90 and vapors is lowered in the cooling apparatus, so that the said gas and vapor enter the corroding-compartment of the stack marked 2 at a substantially low temperature—that is, at about 95° Fahrenheit—and 95 the lead fiber contained therein is energetically acted upon by the said gas and vapor and converted into the carbonate in a substantially short time without the formation

of dry spots on the lead.

The surplus gas and vapor pass from the stack 2 through the pipe  $b^5$  into the stack 3, thence by the pipe  $b^6$  into the stack 4, thence by the pipe  $b^7$  into the stack 5, from which it passes through the pipe  $b^3$  into the chimney 105  $b^4$ . The chimney  $b^4$  is provided with a damper  $c^4$ , by which the draft of the chimney may be controlled, and the rate of circulation of the gas and vapor through the stacks may be regulated. To assist the natural draft of the 110 chimney, a gas or other burner  $c^5$  is located within the chimney.

The inlet-pipe  $b^2$  for the stack A (marked 2) is provided with a valve  $c^6$ , by which the inlet to the corroding-compartment of the stack 115 2 may be closed, if desired—as, for instance, at night-time, the lamp or burner  $b^{15}$  being

at such time extinguished.

During the day-time the lead fiber may be subjected to the corroding action of the hy- 120 drous carbonic-acid gas from the lamp or burner  $b^{15}$  and to the action of the carbonicacid gas and vapor arising from the compartment a' of the corroding-stack, as at that time the lamp or burner  $b^{15}$  may be watched 125 and controlled by a workman; but at night the lamp may be extinguished and the corroding action still maintained by the carbonicacid gas and vapor arising from the compartment a' of the stack.

Instead of the particular form of cooling apparatus, any other desired form of appara-

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tus may be employed—such, for instance, as shown in Fig. 3, wherein the gas is subjected to a shower of water from a rose or nozzle  $c^{10}$ on the end of a pipe  $c^{12}$ , connected to a suit-5 able source of supply.

By means of the natural draft of the chimney a circulation of gas and vapor may be maintained without the use of machinery,

such as a pump.

In practice the trays or supporting-frames covered with lead fiber are placed within the stacks and the doors securely closed. The lamp or burner  $b^{15}$  is started and the steam is admitted to the compartment a' to start the 15 fermentation or combustion of the tan-bark, cow-dung, or like material, and the corrosion is continued until all the metallic lead is converted into the carbonate, which takes from four to five days, at the end of which time 20 the trays holding the lead carbonate may be removed, and the carbonate, which is then of a creamy consistency, is dried and is ready to be used without subsequent treatment, such as grinding, as more commonly practiced.

We have herein shown a lamp by which the hydrous carbonic-acid gas is generated; but we do not desire to limit ourselves in this respect, as any desired form of burner may be

used.

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We claim—

1. The herein-described method of producing lead carbonate, which consists in confining finely-divided metallic lead in a closed chamber and subjecting the said finely-di-35 vided lead to the action of cooled hydrous carbonic-acid gas and an oxidizing agent, the said gas being generated by the combustion of refined petroleum or its described equivalent and cooled before entering the said cham-40 ber, substantially as described.

2. The herein-described method of producing lead carbonate, which consists in confining finely-divided metallic lead in a closed chamber and subjecting the said finely-di-45 vided lead to the corroding action of carbonicacid gas and acetic-acid vapor generated in the said chamber and to the corroding action of hydrous carbonic-acid gas and acetic acid vapor generated outside the chamber and 50 cooled before entering the said chamber, sub-

stantially as described.

3. The herein-described method of producing lead carbonate, which consists, first, in producing a purified hydrous carbonic-acid 55 gas, consisting of carbonic-acid gas and chemically-produced water obtained by the combustion of refined petroleum or its hereindescribed equivalent; second, cooling the hydrous carbonic-acid gas to substantially the 6c temperature herein specified, and, third, acting upon finely-divided metallic lead with the cooled hydrous carbonic acid in the presence

of an oxidizing agent, substantially as described.

4. The herein-described method of produc- 65 ing lead carbonate, which consists in confining finely-divided metallic lead in a closed chamber and subjecting the said finely-divided lead to the corroding action of carbonicacid gas and acetic-acid vapor generated in 70 the said chamber and to the corroding action of hydrous carbonic-acid gas generated outside the chamber and cooled before entering the said chamber, substantially as described.

5. In an apparatus for the production of 75 lead carbonate, the combination, with a chamber or stack provided with a perforated partition to form compartments, of a gas inlet and outlet for one of said compartments, a series of removable trays in the upper com- 80 partment to support the lead to be converted, a fermenting substance in the lower compartment, and means to heat the lower of said compartments, substantially as described.

6. In an apparatus for the production of 85 lead carbonate, the combination, with a stack or chamber to contain the lead, of an inletpipe for the said stack or chamber, a vessel to contain fluid, provided with a tube or pipe extended up through the vessel, of a burner 90 to co-operate with and heat said vessel, and a cooling apparatus located in the inlet-pipe,

substantially as described.

7. In an apparatus for the production of lead carbonate, the combination, with a stack 95 or chamber to contain the lead, of an inletpipe for the said stack or chamber, a vessel to contain fluid, provided with a tube or pipe extended up through the vessel, of a burner to co-operate with and heat said vessel, and ico a cooling apparatus located in the inlet-pipe, an outlet-pipe for said chamber or stack, and a chimney with which the outlet-pipe communicates, substantially as described.

8. In an apparatus for the production of 105 lead carbonate, the combination, with a stack or chamber to contain the lead, of an inletpipe for the said stack or chamber, a vessel to contain fluid, provided with a tube or pipe extended up through the vessel, of a burner 110 to co-operate with and heat said vessel and a cooling apparatus located in the inlet-pipe, an outlet-pipe for said chamber or stack, and a chimney with which the outlet-pipe communicates, and a burner located in said chim-115 ney, to operate substantially as described.

In testimony whereof we have signed our names to this specification in the presence of two subscribing witnesses.

> NORMAN K. MORRIS. JOHN W. BAILEY.

Witnesses:

C. T. WARD, R. T. ROYAL.