UNITED STATES PATENT OFFICE.

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PROCESS OF MAKING LEAD CARBONATE.

SPECIFICATION forming part of Letters Patent No. 649,507, dated May 15, 1900.

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To all whom it may concern:

Be it known that I, GEORGE D. COLEMAN, a citizen of the United States, residing at Bridgewater, in the county of Plymouth and State of Massachusetts, have invented certain new and useful Improvements in Processes of Making Lead Carbonate; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

The present invention relates to an improvement in the art of making lead carbonate, and more particularly to the art of making lead carbonate from metallic lead.

All processes of making carbonate of lead known to me are more or less objectionable because of the fact that they are either tedious, expensive, or result in an impure product.

The objects of my invention are to improve the quality of the product and to produce a very pure carbonate of lead, to decrease its cost of manufacture, and to reduce to a minimum the time required in carrying out the process, as well as to secure better hygienic conditions in the works.

To these ends, therefore, my invention consists in the art of making carbonate of lead hereinafter described, and more particularly set forth in the claims.

In general my process consists in exposing metallic lead to the action of a suitable oxidizing reagent, such as air, to corrode or oxidize it, then in exposing the product to the action of a suitable carbonating reagent, such as carbonic acid, whereby the product of the oxidation is largely transformed into lead carbonate, and then exposing the product of the carbonating step to the action of a suitable oxidizing reagent, such as air, to transform the small quantities of metallic lead still found in the mass to the suboxids of lead, and at the same time to transform the small quantities of suboxid of lead thus formed and present in the mass at the beginning of the step to nascent lead protoxid. Then the product of the last step is subjected to the action of a suitable carbonating reagent, such as carbonic acid, thereby reducing the protoxid of lead to the carbonate, so that the product of this step is pure carbonate of lead.

While my process of making carbonate of lead is thus seen to consist in the alternate exposure of the material, which at the beginning was metallic lead, to the action of oxidizing and carbonating reagents alternately until all of the lead originally subjected to the action of the reagents employed is entirely transformed into carbonate of lead, the manner in which I carry out this process may vary in many respects without departing from the spirit of my invention. Still I prefer to carry out this process in the following manner:

First. Metallic lead, preferably comminuted by grinding or cutting into very small pieces, by which the action of the reagents thereupon, owing to the greater surface exposed, is materially facilitated, is subjected to the action of an oxidizing reagent, preferably pure air, containing no carbonic acid, and in order to remove protoxid of lead, which forms very rapidly upon the surface of the metallic lead when so exposed, the whole mass is agitated by stirring or tumbling, preferably in the presence of water, so that the lead shall be exposed to the oxidizing influence of the oxidizing reagent and then subjected to the washing action of the water, whereby the newly-formed oxid will be washed off to present a clean surface to the renewed action of the said reagent. Any suitable means may be employed for this purpose—as, for example, the apparatus for corroding lead illustrated and described in Letters Patent granted me August 16, 1892, No. 481,004. This corroding or oxidizing step may be carried on at the ordinary temperature; but by preference the mass will be maintained at a temperature of 127° Fahrenheit, as I have found that the corroding or oxidizing action is more rapid at or near this temperature. Of course I do not confine myself to this precise temperature, as good results can be obtained by using lower or higher temperatures. The product of this corroding or oxidizing operation will be about eighty-five per cent. lead protoxid, eight per cent. lead hydrate, five per cent. lead suboxid, and two per cent. of unaffected finely-divided metallic lead, although under varying conditions it may depart considerably from the proportions above given.

Second. The product of the first corroding

or oxidizing step is now subjected to the action of a carbonating reagent, preferably carbonic acid. By preference the product of the corroding or oxidizing operation before being exposed to the carbonic acid will be cooled as much as is conveniently possible in order that the carbonating process may be more rapidly carried on, as I have found that carbonating progresses less rapidly at higher than at lower temperatures. In order to accelerate the rate of carbonating the mass, I subject it to agitation by stirring or tumbling, as in oxidizing. This carbonating operation is continued until the product of the corroding or oxidizing step has been almost entirely transformed into a lead carbonate, the resulting mass being approximately ninety-six per cent. lead carbonate, two per cent. suboxid of lead, and two per cent. unaffected metallic lead, as before, although under varying conditions it may depart considerably from these proportions. It will be observed that by this operation the protoxid of lead and the hydrate of lead have been transformed into lead carbonate and that some of the suboxid of lead has taken up oxygen mixed with the carbonic acid or water, becoming protoxid of lead, so that when this operation is completed there still remains present about two-fifths the quantity of suboxid of lead present at the beginning of the operation. Whatever metallic lead was present is of course chemically unaffected by this step, although the attrition incident to the step will result in its being more finely divided than before.

Third. The product is now transformed to another vessel and subjected to the action of an oxidizing reagent, preferably air, the whole mass being properly heated to a temperature of 125° Fahrenheit, although it may vary within considerable limits without departing from the spirit of my invention and agitated by tumbling or stirring, as before, so as to bring the oxidizing reagent into intimate contact with the metallic lead and the suboxid of lead, thereby transforming the metallic lead into the suboxid and at the same time converting the suboxid of lead present at the beginning of this operation and that formed from the metallic lead by oxidation into lead protoxid. The completion of the step may be determined by chemical analysis or by the change of color of the mass from white with a tinge of blue, due to the presence of finely-divided metallic lead, to white with a tinge of yellow, due to the presence of lead protoxid. The composition of the mass at the end of this operation will be ninety-six per cent. lead carbonate and four per cent. lead protoxid, although under varying conditions it may depart considerably from these proportions, thus leaving the carbonate unaffected by this operation, but entirely removing all traces of metallic lead and suboxid of lead from the mass.

I have found that the "light tinge of blue" which is referred to in the specification of the patent to Gardner of August 28, 1840, No. 1,744, is due to the presence of metallic lead or suboxid of lead, or both, and while that specification states that this "light tinge of blue" disappears in the process of drying I have found that it only disappears from the surface of the mass by reason of the oxidation of the surface metallic lead by the air and the subsequent transformation of the suboxid of lead thus produced or originally present in the mass into carbonate by taking up carbonic acid from the air, and thus preventing the recognition of the presence of metallic lead or suboxid of lead by the eye. Still it does not remove it from the body of the mass, and it is for the very purpose of removing this very finely-divided lead or suboxid of lead, or both, indicated by this "light tinge of blue," that at this stage in my process of making lead carbonate I subject it to this further oxidizing process in order to completely remove metallic lead or suboxid of lead, or both, from the mass. In this important respect my invention is very sharply distinguished from the patent to Gardner above referred to, which results, for the reasons stated, in a product containing metallic lead or suboxid of lead, or both, and while this is not the only important distinction between my process and that of Gardner it is one that it is pertinent here to advert to, reserving for a more appropriate place another distinction hereinafter to be pointed out.

Fourth. The product is now subjected to the action of a carbonating reagent, preferably carbonic acid. While this may be done by simply cutting off the supply of air and introducing the carbonating reagent into the vessel containing the mass, I prefer first to cool the entire mass by pumping it out of the vessel and passing it through suitable cooling pipes or vessels to reduce the temperature to the normal temperature of the air or still further, if convenient. It will be understood that the operation of carbonating, as before stated, is more rapidly carried out when the liquid is cool than when warm. I also subject the mass to agitation by stirring or tumbling and by passing the carbonating reagent into the bottom of the vessel, so as to percolate up through the mass to thereby bring the reagent into intimate contact with all portions of the same. This operation is continued until all of the lead protoxid is transformed into lead carbonate, thus producing a mass which consists entirely of lead carbonate and water.

It is to be observed that in the specification of the patent above referred to it is proposed to make white lead by introducing "carbonic acid in conjunction with a portion of atmospheric air" into a vessel containing granulated metallic lead, while by my process the oxidizing reagent and the carbonating reagent are introduced alternately into the vessel containing the lead to be treated. I have found that the operation is very much more satis-

factorily and economically carried out if these operations are made successive and not coincident besides which, by heating the mass during the oxidizing operations and by cooling it during the carbonating operations, I am able to secure better working conditions than if the two operations were carried on at the same time. So, while I may or may not use heat during the oxidizing operations, my process is clearly distinguished from the said Gardner patent by reason of introducing the reagents separately into the vessel containing the material to be operated upon. This is the second important difference between my process and the process described in the above-named patent.

Having thus described my invention, I claim as new and desire to secure by Letters Patent of the United States—

1. The art of making pure lead carbonate which consists in subjecting metallic lead and the successive products therefrom to the separate, successive and repeated actions of an oxidizing reagent and a carbonating reagent, such actions being repeated until all the metallic lead has been converted into lead carbonate, substantially as described.

2. The art of making pure lead carbonate which consists in subjecting metallic lead and the successive products therefrom to the separate, successive and repeated actions of an oxidizing reagent and a carbonating reagent and heating the mass during the oxidizing operations and cooling it during the carbonating operations, such actions being repeated until all the metallic lead has been converted into lead carbonate, substantially as described.

3. The art of making pure lead carbonate which consists in subjecting metallic lead in the presence of water to the oxidizing action of atmospheric air at a temperature of approximately 125° Fahrenheit, in subjecting the product of the last-named step to the action of carbonic acid at a lower temperature, in subjecting the product of the last-named step to the oxidizing action of atmospheric air at a temperature of approximately 125° Fahrenheit, and in subjecting the product of the last-named step to the action of carbonic acid at a lower temperature, whereby all the metallic lead is converted into lead carbonate, substantially as described.

In testimony whereof I affix my signature in presence of two witnesses.

GEORGE D. COLEMAN.

Witnesses:
T. HART ANDERSON,
JAMES V. ROE.