UNITED STATES PATENT OFFICE.

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MANUFACTURE OF WHITE LEAD.

SPECIFICATION forming part of Letters Patent No. 414,935, dated November 12, 1889.

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To all whom it may concern:

Be it known that I, TURNER D. BOTTOME, a citizen of the United States, residing at Hoosick, in the county of Rensselaer and State of New York, have invented certain new and useful Improvements in the Manufacture of White Lead; 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 object of the present invention is the commercial manufacture, by the agency of an electric current, of white lead in the form of the hydrated carbonate, which can thus be effected quickly and uniformly, instead of the tedious oxidation process hereinbefore generally used.

The invention comprises the preparation of the electrolyte, the electrodes, and the various steps of the prccess, substantially as hereinafter fully described and claimed.

The invention consists in electrically dissolving a lead electrode in an electrolyte containing nascent or free carbon dioxide, whereby the lead compound formed by electrolytic action is precipitated to form hydrated carbonate of lead or pure white lead, which is then removed, washed, and dried.

One method of carrying my invention into practical effect is as follows: The electrolytic solution can be prepared by dissolving one-half pound each of sodium nitrate and ammonium nitrate in one gallon of water, and then saturating the solution thus formed with carbon dioxide, which can be done in various ways. Sodium carbonate and ammonium carbonate may be used in the place of the nitrates; but in that case nitric acid must be added until the bath is about neutral, which results in the larger portion of the carbon dioxide being driven off during effervescence. The electrolytic solution is then placed in a suitable vessel or tank and electrodes of metallic lead are immersed in the same. The electrodes are then connected to a source of electricity, and for practical work I prefer a current density of about fifteen ampères per square foot of anode surface. Upon the passage of such a current between the electrodes through the bath the white lead begins to fall very rapidly. As the carbon dioxide is taken up from the bath to form the hydrated carbonate of lead, it is of course necessary to have the bath replenished with additional carbon dioxide as the process continues. This may be done in several ways. Porous pots filled with sodium or ammonium carbonate may be placed in the bath, and will there slowly dissolve out into the bath for the purpose of supplying the latter with the required carbon dioxide; but this method involves the ultimate oversaturation of the bath with sodium hydrate and free ammonia, which are of no use when once formed, and only tend to interfere with the most advantageous working of my process. I prefer to supply the carbon dioxide in some other way, and obviously the best method will be one which will supply it unaccompanied by any other material. A convenient way of doing this is by burning limestone, washing the gas produced by the disassociation of the constituents of the limestone, and supplying such gas directly to the bath by any suitable means. In this case no detrimental effect takes place in the electrolytic solution, and the latter remains in a normal condition while the precipitation of the white lead is obviously going on under the electrolytic action of the current.

Where the electrolytic solution is made up of sodium and ammonium nitrates in water and the solution is saturated with carbon dioxide in its free state, the reactions taking place upon the passage of the electric current may be described as follows: The bath is decomposed, yielding at the anode nitrogen pentoxide, ozone, and oxygen, and at the cathode sodium hydrate, ammonia, and hydrogen. The lead is attacked by the powerfully-oxidizing nitrogen pentoxide (N_2O_5) and ozone; but nitrogen pentoxide in the presence of water is decomposed and forms nitric acid, (HNO_3.) During the double decomposition which takes place nitric acid (HNO_3) and plumbic acid or hydroxide of lead, $PbO(OH)_2$, are formed. The nitric acid combines with the free ammonia and sodium hydrate to again form sodium and ammonium nitrate, while the plumbic acid is precipitated by the free carbon dioxide present and forms

finally the hydrated carbonate of lead, $2PbO\,CO_2+Pb(OH)_2$. Thus it is seen that although the solution is decomposed by electrolysis it is regenerated by the chemical reactions taking place, and the only loss sustained is carbon dioxide and water, which can be readily supplied. The white lead is from time to time removed from the tank, washed, and dried, and on mixing with a suitable vehicle into a paint it is found to have much greater covering properties than ordinary commercial white lead formed by dissolving lead in acetic acid in the presence of carbonic acid, since the latter is slightly crystalline and less opaque than the hydrated carbonate produced by the action of carbonic acid on plumbic acid.

I have given what I consider the most practical and economical solution; but I wish to state that I do not confine the scope of my invention thereto, as various solutions may be employed without departing from my invention. For instance, I may use the nitrate or carbonate of any of the alkaline metals or earths; but I give preference to the combination hereinbefore named. Likewise the particular means adapted for the introduction of carbon dioxide into the electrolytic bath may be varied from those given—such, for instance, as the action of diluted sulphuric acid on sodium carbonate or calcium carbonate, and in either of these cases the residue becomes a marketable product. The strength of the bath may be weak or strong without materially affecting the results; but I prefer the proportions given with regard to quantities, as also the amount of current per square foot of anode surface, and I also prefer that the bath should be kept at about 60° Fahrenheit.

I claim as my invention—

1. The process of manufacturing white lead, consisting in electrolytically dissolving a lead anode in an alkaline aqueous solution while supplying the latter with carbon dioxide.

2. The process of manufacturing white lead by electrolytically dissolving an electrode of metallic lead in an alkaline solution containing free carbon dioxide.

3. The process of manufacturing white lead by electrolytically forming an oxygen compound of lead from a lead electrode in an alkaline solution containing free carbon dioxide, which then unites with the oxygen compound of lead to form the hydrated carbonate of lead.

4. The process of manufacturing white lead, consisting in electrically converting lead into hydrated lead carbonate by subjecting an electrolyte of an alkaline nitrate to electrolysis with leaden anodes while the bath is kept saturated with free carbon dioxide, substantially as herein described.

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

TURNER D. BOTTOME.

Witnesses:
D. P. GRIFFITH,
G. E. BABCOCK.