

(No Model.)

R. W. E. MACIVOR.

WHITE LEAD.

No. 413,528.

Patented Oct. 22, 1889.

Fig. 1.

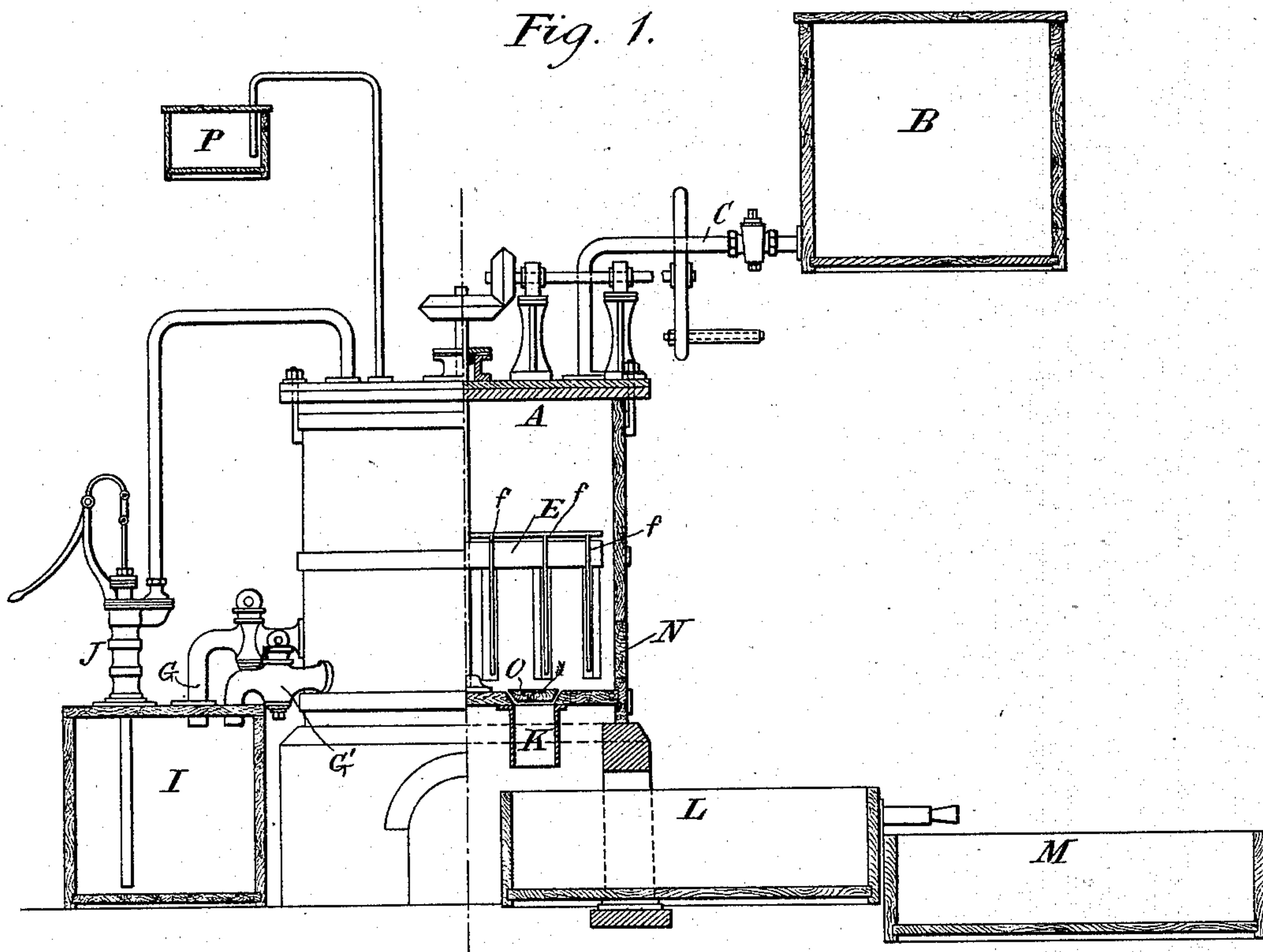
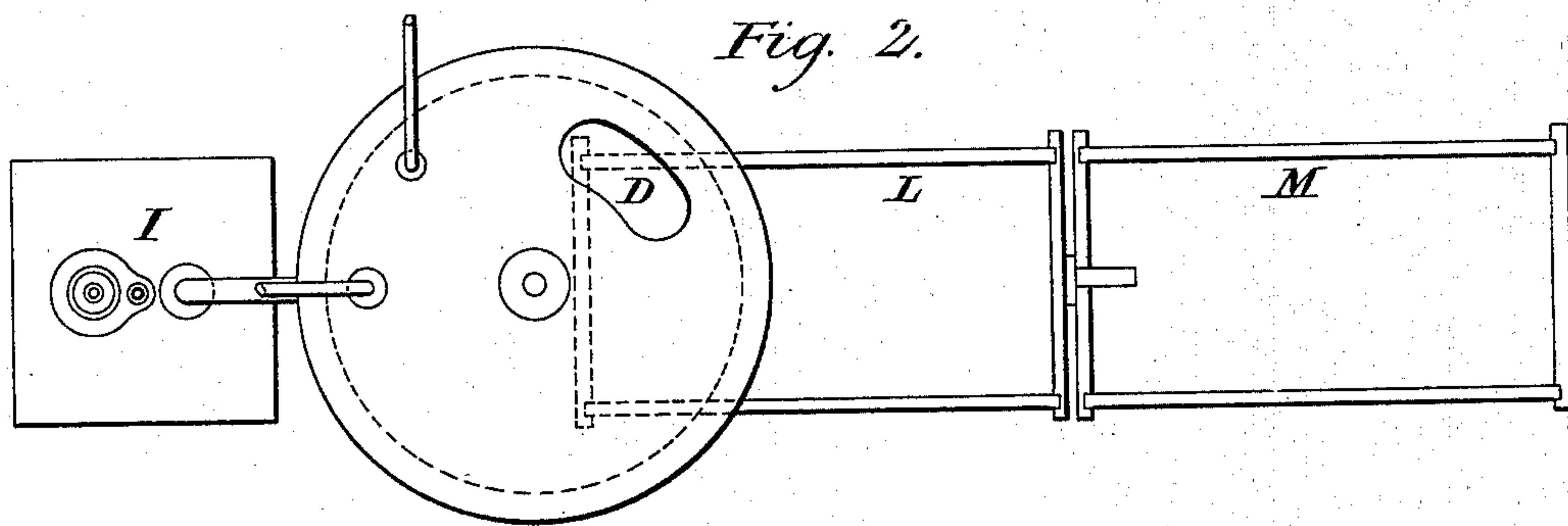


Fig. 2.



Witnesses.

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UNITED STATES PATENT OFFICE.

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WHITE LEAD.

SPECIFICATION forming part of Letters Patent No. 413,528, dated October 22, 1889.

Application filed August 14, 1889. Serial No. 320,685. (No specimens.) Patented in England July 18, 1888, No. 10,426.

To all whom it may concern:

Be it known that I, RALPH WALDO EMERSON MAC IVOR, F. I. C., F. C. G., M. Phy. Society, London, F. R. G. S., &c., analytical and consulting chemist, a subject of the Queen of Great Britain, residing at St. George's Club, Hanover Square, London, in the county of Middlesex, England, have invented a certain new and useful Improvement in the Production of White Lead or "Carbonate of Lead," (for which I have received Letters Patent in Great Britain, No. 10,426, dated July 18, 1888,) of which the following is a specification.

This invention relates to the production of white lead or basic carbonate of lead by the treatment of monoxide of lead—that is to say, litharge or massicot with a solution of acetate of ammonia, whereby the monoxide of lead is transformed into hydrate of lead, which is subsequently converted into basic carbonate of lead by the injection of carbonic acid. The hydrate of lead readily unites with the carbonic acid to yield the final product—namely, basic carbonate of lead.

In order that my said invention may be fully understood and readily carried into effect, I will proceed to describe the process more in detail by the aid of the drawings hereunto annexed.

Figure 1 is a vertical section of the apparatus which I employ, and Fig. 2 is a plan of the same.

A is a vat, which may be made of wood or other material capable of resisting the chemicals employed. It may conveniently be six feet in diameter and five feet deep. It is provided with a closely-fitting cover.

B is a cistern situated at a higher level and intended to contain a solution of acetate of ammonia.

C is a pipe by which the solution can be drawn down from the cistern into the vat A. A cock is provided upon this pipe, as the drawings indicate. There is a man-hole D in the cover of the vat, and the vat contains an agitator E with a vertical shaft, which can be turned by gearing, as shown; or the agitator may be driven by any suitable motor. The shaft of the agitator is hollow, and pipes *ff*, which stand immediately behind the stir-

ring-tines, are connected with the hollow shaft to deliver the carbonic-acid gas into the vat; or this may be effected by means of concentric rings laid at the bottom of the vat and pierced with small holes. The pipes *ff* are open at their lower ends.

G is a cock by which the liquor can be drawn off from the vat into the receiver I. A pump J is provided upon the cover of the receiver, by which the liquor may be returned into the vat A.

K is an outlet by which the white lead is discharged from the vat into the washing-cisterns L and M.

N is a man-hole, which may be opened to facilitate the emptying of the vat.

O is a plug, which is removed to let the white lead run out of the vat.

P is an ammonia catch box. It is charged with acetic acid. All the metal work of the apparatus with which the acetate solution comes into contact should be thoroughly tinned or made of acid-proof metal.

The operation is by preference conducted in the following manner, but the details admit of variation: The charge of monoxide of lead for an apparatus of the dimensions indicated may weigh about eleven hundred and twenty pounds. The monoxide should be in fine powder, and may be either moist or dry. Having received this charge, which is introduced by the upper man-hole, the vat is closed and solution of acetate of ammonia is let down upon the charge from the cistern B or pumped out of the receiver I. The vat A should be charged with the solution of acetate of ammonia in the proportion of three parts of said solution to one part of lead monoxide, by weight. It is convenient to employ a solution containing five per cent. of acetate of ammonia, and the quantities above stated are suited to a solution of this strength; but the strength of the acetate solution may be varied within wide limits, as hereinafter explained. The charge of monoxide of lead and acetate of ammonia in the vat should be kept constantly stirred by the agitator, when it will be found that the monoxide of lead has become converted into hydrate. The workman will know that this change is

complete when the reddish appearance of the monoxide of lead disappears and the mass in the vat becomes whitish in color. The hollow axis of the agitator is connected with a pipe, by which carbonic-acid gas is supplied to it under pressure sufficient to cause the gas to pass through the contents of the vat. This gas escapes at the lower ends of the pipes *f* and ascends through the liquid in which the hydrate of lead is held suspended. A free flow of this gas should be maintained and some excess allowed to pass away by a pipe on the cover into the ammonia catch box P. The pipe dips down into the acetic acid which this box contains, and any ammonia passing off with the carbonic-acid gas is caught by the acid and forms acetate of ammonia. When the hydrate of lead is completely converted into basic carbonate, and this the workman will know by its changed appearance, the motion of the agitator is caused to cease and the white lead allowed to settle. There is a gage-glass on the side of the vat A, and in this glass the changes of appearance can be recognized by which the workman regulates the process. When the white lead is deposited, the liquor is drawn off into the receiver I by opening the cock G. The plug O is then displaced and the white lead allowed to descend into the washing-cistern L. Below the cock G there is another cock G', in order that any further quantity of liquor may be drawn off into the cistern I if required. The taper-plug O is then raised by being pushed up from underneath the vat A through pipe K. The white lead requires to be well washed with clean cold water. When a fresh charge of monoxide of lead has been placed in the vat A, the liquor is pumped up onto it from the receiver I, and then a further quantity of solution is drawn from the cistern B and also from the ammonia catch box until the proper quantity has been supplied, which is determined by the gage. The rapidity with which the conversion of monoxide of lead into hydrate can be effected depends upon the strength of the acetate-of-ammonia solution employed—that is to say, the weaker the solution the slower will be the conversion.

I have found that for commercial purposes the solution of acetate of ammonia may be used with advantage of any strength between five and twenty-five per cent. A strength of twenty-five per cent. operates in a comparatively short space of time, but a strength as low as half a per cent. will effect the hydration if there be a sufficient quantity of the weak solution, the lead oxide being in a fine state of division and time of no object. This half per cent. strength, however, or any strength below five per cent., is not recommended for commercial purposes, having regard to the time required for completing the operation. The conversion of the oxide of lead into the hydrate of lead is effected in the cold. Heat may be used, but for commercial operation it is not recommended. The solution of acetate of ammonia may be repeatedly used for the conversion of further quantities of oxide of lead into hydrate of lead, which hydrate is converted into basic carbonate of lead by the injection of carbonic acid. Theoretically, a given weight of acetate of ammonia in solution should be capable of converting an unlimited quantity of oxide of lead into hydrate; but during the manufacture of white lead by this process it may be reckoned that there will necessarily be some loss of acetate of ammonia.

What I claim is—

1. The process for the manufacture of white lead, consisting in the treatment of monoxide of lead with acetate of ammonia, whereby hydrate of lead is formed and the conversion of this into basic carbonate of lead by the subsequent injection of carbonic acid, substantially as herein described.

2. The process for the manufacture of white lead, consisting in submitting monoxide of lead to agitation with a solution of acetate of ammonia in a close vessel, and afterward, when the monoxide has been converted into hydrate, passing streams of carbonic-acid gas through the contents of the vessel.

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Witnesses:

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