BUTTON & DYER.

Making White Lead.

No. 1,115. Patented Apr. 10, 1839.

Fig. 2.

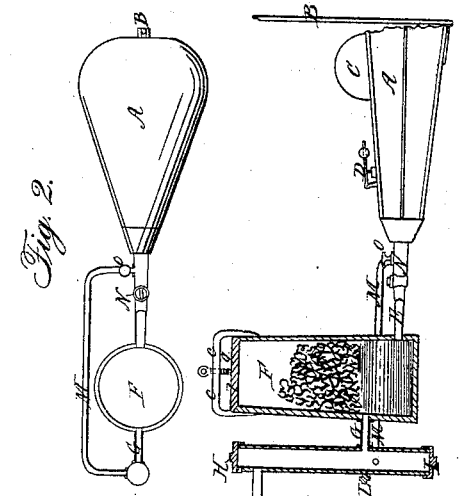


Fig. 1.

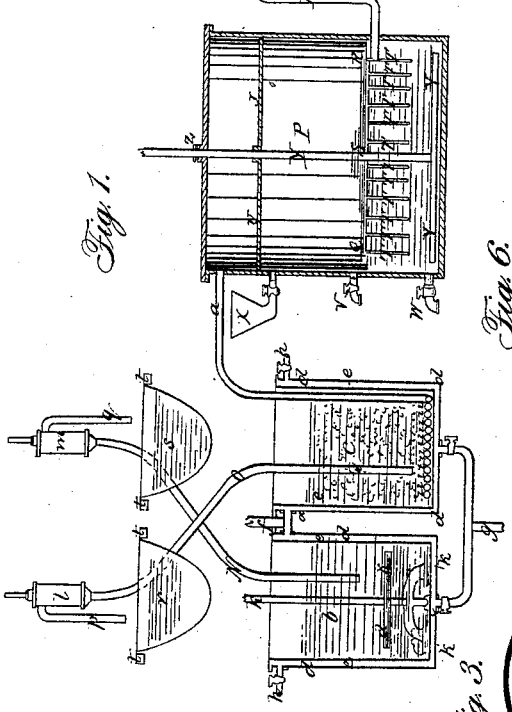


Fig. 5.

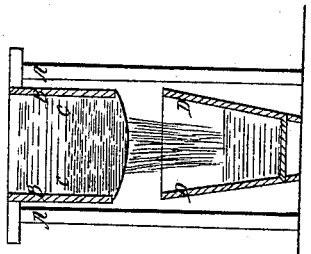


Fig. 7.

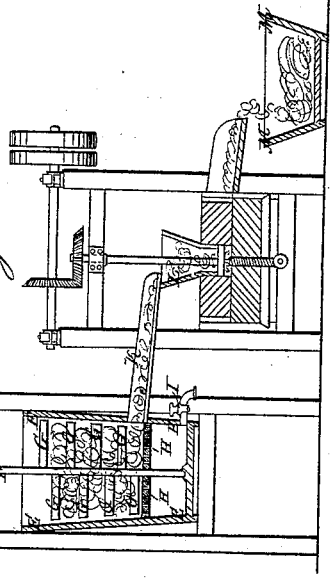


Fig. 6.

Fig. 3.

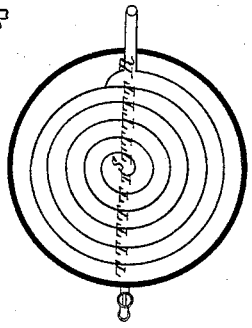
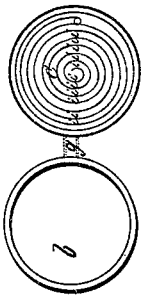


Fig. 4.



Inventor:

Chas. Button

Witnesses: Geo. Dyer

UNITED STATES PATENT OFFICE.

CHARLES BUTTON AND HARRISON GREY DYAR, OF LONDON, ENGLAND.

PROCESS OF MANUFACTURING CARBONATE OF LEAD, CALLED WHITE LEAD.

Specification of Letters Patent No. 1,115, dated April 10, 1839.

To all whom it may concern:

Be it known that we, CHARLES BUTTON, of Holborn Bars, chemist, a subject of the Queen of Great Britain, and HARRISON GREY DYAR, of Mortimer street, Cavendish square, gentleman, a citizen of the United States of America, both in the county of Middlesex, in the Kingdom of England, have invented or discovered certain new and useful Improvements in the Manufacture of White Lead; and we, the said CHARLES BUTTON and HARRISON GREY DYAR, do hereby declare the nature of our said invention to consist, first, in purifying the gases or vapors arising from stone or mineral coal when used in the process of manufacturing carbonate of lead by a mode of combustion induced by a peculiar application of atmospheric air to the combustion and also by a process of washing in a mixture of carbonate of soda whiting and white lead or other materials of like chemical properties as hereinafter explained. Secondly, in the use of basic nitrates of lead for producing carbonate of lead. Thirdly, in causing white lead to be made from litharge massicot or protoxid of lead by boiling together or otherwise mixing nitric acid or nitrate of lead upon litharge massicot or the protoxid of lead and exposing the mixture or solution while hot to be acted upon by carbonic acid gas; and, fourthly, in a mode of further purifying and condensing the said carbonate of lead when so produced; and we, the said CHARLES BUTTON and HARRISON GREY DYAR, do hereby describe the manner in which our said invention is to be performed by the following statement thereof, reference being had to the drawings annexed and to the figures and letters marked thereon—that is to say, first as to the machinery or apparatus used for our improvements in the said manufacture.

Figure 1 is a vertical section of our apparatus so far as it is necessary in producing white lead from litharge massicot or protoxid of lead.

A is a pair of forge bellows; B, the rod by which the bellows are kept in action. C is a weight or load on the bellows necessary to overcome the friction and heads of water or solutions against which they have to contend. D is a safety valve on the bellows. E is the nozzle pipe of the bellows going into the furnace F, which is a cast iron vessel. Red *t*, is a cast iron cap or cover which can be taken off at pleasure; red *c* is a bridle of wrought iron through which the screw red *g* works so as to fasten the cover red *t*, which we make air tight with a little fire clay. G is the exit pipe of the flame from the furnace. H, I, is an iron chamber, which may be designated as the flame chamber. K is the exit pipe from the flame chamber. L is a plug (which may be unscrewed in case the pipe G be obstructed) by which the passage can be freed from dust. At H and I are two caps which may be unscrewed so as to remove any accumulation of dust which might collect in the flame chamber. M is a pipe leading from the nozzle of the bellows into the flame chamber which pipe will convey atmospheric air from the bellows into the flame chamber without going through the furnace F, the arrangements of this pipe in its relation to the furnace and flame chamber may be more fully understood by reference to Fig. 2 which is an horizontal section of the furance flame chamber, pipe M and the nozzle of the bellows. Similar letters being used to denote similar parts in each; but to revert to Fig. 1, at N and O are cocks the one in the nose of the bellows, the other in the pipe leading from the bellows to the flame chamber and the area of the opening in the latter cock should be one fifth of that in the former. P, is a large closed cylindrical iron vessel which may be demoninated the washing vessel into which the burnt gases or products from the furnace pass. Q, R is a plate of iron. T, T, T, show the sections of a spiral coil of flat sheet iron open at bottom and attached vertically to the plate Q, R, in such a manner that the air may enter the outermost part of the coil at R and circulate around the coil until it arrives at the center S at which place there is an opening through the plate Q R by which the vapors and gases may pass into the upper part of the vessel P.

Fig. 3, is an horizontal section of the coil T, T, T, showing that the burnt air may enter at R and circulate around the coil and pass out at S. Similar letters denoting similar parts in each. U, U, is a diaphragm of fine wire gauze drawn across the washing vessel P, and supported by perforated copper plates. V and W are cocks. X, is a funnel with cock attached. Y, Y, Y, is a shaft and

arms of a stirrer working through a stuffing box at Z. The burnt air after passing first through the coil T, T, T, then through the wire gauze U U makes its exit through the pipe *a*. *b* and *c* are two cylindrical copper vessels. *d*, *d*, *d*, *d* are copper jackets or casings inclosing the copper vessels *b* and *c*. *e e e e* are steam tight vacant spaces between the said jackets and the said copper vessel. *f* is a steam pipe opening into these vacant spaces. *g*, is an exit pipe for condensed water. *h*, *h* are air cocks. The copper vessel *c* may be designated as the white lead vessel, the copper vessel *b* may be designated the litharge vessel. *i*, *i*, *i*, *i*, is a copper pipe coiled around the bottom of the vessel *c* shown in plan at Fig. 4 the one end being closed and the other connected with the pipe *a*. The coil *i*, *i*, *i*, *i*, is pierced with a great number of small holes so that the vapors and gases passing down the pipe *a* to the coil *i*, *i*, *i*, *i*, will pass out of that coil in a great number of little streams or bubbles into the open vessel *c*. *k*, *k*, *k*, *k* is the shaft and arms of a stirrer or agitator working in the litharge vessel *b*. *l*, *m* are two copper pumps drawing out the vessel *c* and *b* by means of the copper suction pipes *n* and *o* and discharging through the pipes *p* and *q*, into the filtering bags *r* and *s*. The filtering bags *r* and *s* are linen cloth stretched on square wooden frames *t*, *t*, *t*, *t*.

Secondly, as to the mode of working this apparatus we remove the cap or cover from the furnace F and we then throw a little lighted coal into the furnace at the same time setting the bellows A into action, the cock N being open and O being closed we then fill up the furnace with the best stone coal after the fire is well lighted we put on the cap red *t*, and screw it down air tight, we then turn on the cock O, we allow the coal in the lower part of the furnace at and below the pipe G to become completely ignited before the gas is applied to use after which the volatile matter liberated from the coal which lies in the furnace above G will be very much decomposed in passing through the ignited coal lying about the pipe G before it escapes into the flame chamber H, I. When it escapes into the flame chamber it is at a very high degree of temperature and is then insected and mixed with a fresh portion of atmospheric air passing from the bellows through the pipe M into the flame chamber which fresh supply of atmospheric air is so proportioned in quantity to that which passes through the furnace as to insure the conversion of any sulfureted hydrogen into sulfurous acid and vapor of water and the carbonic acid into carbonic acid and any compounds of carbon and hydrogen into carbonic acid and water at the same time carefully preventing such an excess of atmospheric air from passing through the pipe M as to endanger the sinking of the temperature of the gases in the flame chamber below that temperature at which sulfureted hydrogen or other volatile matter will combine with the oxygen gas in the atmospheric air. Into the washing vessel P, we put a mixture of ten pounds each of carbonate of soda, whiting, and carbonate of lead or white lead and through the funnel X we pour in water till it rises to the gage cock V a little above the plate Q, R. We fill the vessels *b* and *c* nearly full of distilled water, into the vessel *b*, we place about fifty pounds of litharge, to this we add twenty pounds of commercial nitrate of lead or such quantity that the nitrate of lead is in weight about one thirtieth the weight of water in the vessel. We then turn the steam into the steam jackets through the pipe *f* which leads from an ordinary steam boiler making the liquid in the vessels *b* and *c* to boil. We then set the stirrer *k*, *k*, *k*, in motion by a strap from a drum on a shaft and passing on a pulley attached to the shaft of the stirrer at *k* but not shown in the drawing. The piston rods of the pumps *l* and *m* are severally attached to two cranks upon a shaft which makes thirteen revolutions per minute so that the pump *m* will draw the liquid from the vessel *b* and discharge it into the filter *s* whence passing through that filter it descends into the open vessel *c* while the pump *l* draws the liquid and some white lead from the vessel *c* and discharges it into the filter *r* when the liquor passes through that filter and falls into the open vessel *b*. In this condition of things the air which is taken into the bellows passes through the flame chamber also through the washing vessel and through the pipe *a* into the coil *i*, *i*, *i*, whence it escapes through the small holes made in that coil and will be found to rise up through the liquid in the vessel *c* in a great number of bubbles. And the apparatus being now in the process of making white lead that substance will be found to be pumped up out of the vessel *c* with the liquor and thrown into the filter *r* where the white lead will be arrested while the liquor filters through into the vessels *b* at the same time a small quantity of litharge and impurities will be found to be collected in the filter *s*. The filter *r* which contains the white lead is removed or exchanged for another as often as it becomes filled with white lead and the filter *s* is also exchanged when the pores of the filter become filled with litharge and impurities.

The furnace F will require to be refilled about three times in twenty-four hours when in constant action—the agitator in the washing vessel is kept slowly in motion by a pulley on the shaft Y not shown in the drawing. From time to time it is necessary to add fresh litharge into the vessel *b* taking care not to allow the vessel *b* either to get empty

of litharge or so much to accumulate as to overload the agitator *k*, *k*, *k*,—occasionally fresh water must be poured into the funnel X whence passing through the cock it enters the washing vessel P so that the liquor in this vessel shall never be found much below the gage cock V. In the process of working after a long time the material placed in this vessel P with the accumulations of solid matter require to be drawn out through the cock W and fresh materials and water put in as at first.

It will be proper from time to time to examine the liquor in the vessels *b* and *c* to ascertain if by any waste, leakages, or otherwise this liquor has become too weak or contains too little of the nitrate of lead. The test for keeping this liquor at the right strength will be found by taking out a little liquid from the vessel *b* while it is boiling hot and setting it aside and allowing it to cool when a crystallization or precipitation ought to be produced of basic nitrate of lead. If it should be found that there is no appearance of such crystals this will indicate that by some means the liquor has become too weak and that some fresh nitrate of lead must be added. By testing after this manner the hot liquor taking out and allowed to cool soon after the vessels *b* and *c* were first charged the operator will be enabled to ascertain by comparison when the working liquor is at the proper strength.

We have thus described our apparatus so far as necessary for the producing white carbonate of lead. We now proceed to describe the apparatus and means used for giving this white lead a body best adapted for commercial purposes and at the same time preventing any sensible loss or waste of the solution of nitrate of lead which otherwise might be carried off with the white lead taken from the filter *r*.

By referring to Fig. 5 we have a stand AAAA into which we place a square box BBBB open at the top across the bottom of which box there is drawn a linen cloth as a filter. The contents of the before mentioned filter *r* is removed and put into the box BBBB which we may suppose to fill the box up to C on this white lead we pour pure water to wash out the solution of nitrate of lead remaining mixed with the white lead this process of pouring on water we repeat until the wash water which fllters through the white lead and drops from the filtering cloth is so free from the solution of nitrate of lead that a portion caught in a glass and poured into a solution of carbonate of soda will not render that solution opaque or milky. The accumulations of this wash water fall into the tub DDDD where it is collected and from time to time returned to the litharge vessel *b* (Fig. 1) so that not any of the wash water containing the solution of nitrate of lead shall be lost which likewise may be so arranged that the vessels *b* and *c* shall be thus supplied to compensate the loss of water by evaporation. The washed white lead from the vessel BBBB is now removed to the tub EEEE Fig. 6 which we call the mashing tub in which there is an upright shaft made of gun metal F, F with arms GGG, these arms being slightly inclined so as to cut through the white lead and which thus have a tendency to press it toward the bottom as the arms revolve, thus continually mashing the white lead which brings the particles nearer together, allowing the water held by capillary attraction to separate from the white lead through the bottom HH, which is full of holes over which a filtering cloth is drawn thus allowing the water which is liberated from the white lead by the process of mashing to fall into the vacant space H, E, where it accumulates and may be drawn off by the cock I when required. The process of mashing is continued until water ceases to drip from the white lead after which this washed white lead is allowed to pass through the channel K into the hopper L of the horizontal grinding mill Fig. 7 and thence (being ground) it passes into the vessel M, M. This lead if required for immediate use may be compressed by any convenient mode by which a portion of the remaining water may be pressed out and it may then be removed and dried by any of the usual and well known modes practised by white lead manufacturers.

Now whereas we do not claim any particular mode of generating carbonic acid gas nor of introducing the same into our hot solution, although we have shown the best which we are at present acquainted with for the purpose of obtaining those two objects nor do we claim any particular apparatus as necessary to be made in order to obtain a more perfect combustion and purification of the vapors and gases arising from the fuel in the furnace, but

We claim as our invention—

1. The acting upon the vapors or gases arising from the furnace used in the process of making white lead in such manner (by the introduction of atmospheric air and by washing hereinbefore described) as enables us to use that cheap kind of coal called stone or mineral coal in the said furnace and to arrest the smoke and other impurities injurious to the color or quality of white lead so that we are enabled to use the carbonic acid gas obtained therefrom in the process of manufacturing white lead.

2. In the manufacture of white lead from such compounds of nitric acid with oxid of lead in which the quantity of oxid bears a greater ratio to the nitric acid combined therewith than the oxid of lead bears to the

nitric acid in the ordinary nitrate of lead of commerce and which nitrates we call basic nitrates of lead.

3. Making white lead by boiling as hereinbefore described or otherwise acting upon the litharge massicot or protoxid of lead with either nitric acid or the common nitrate of lead of commerce and while in a boiling state or otherwise submitted to the action of or brought minutely into contact with carbonic acid in order to produce pure carbonate of lead or as it is usually called white lead and in such manner that the same nitric acid or nitrate of lead may be used over and over again many times with fresh portions of litharge as hereinbefore described.

4. The process of further purifying and condensing the white lead obtained as aforesaid by what we call mashing it as hereinbefore described.

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HARRISON GREY DYAR.

Witnesses:
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E. S. CURLEY.