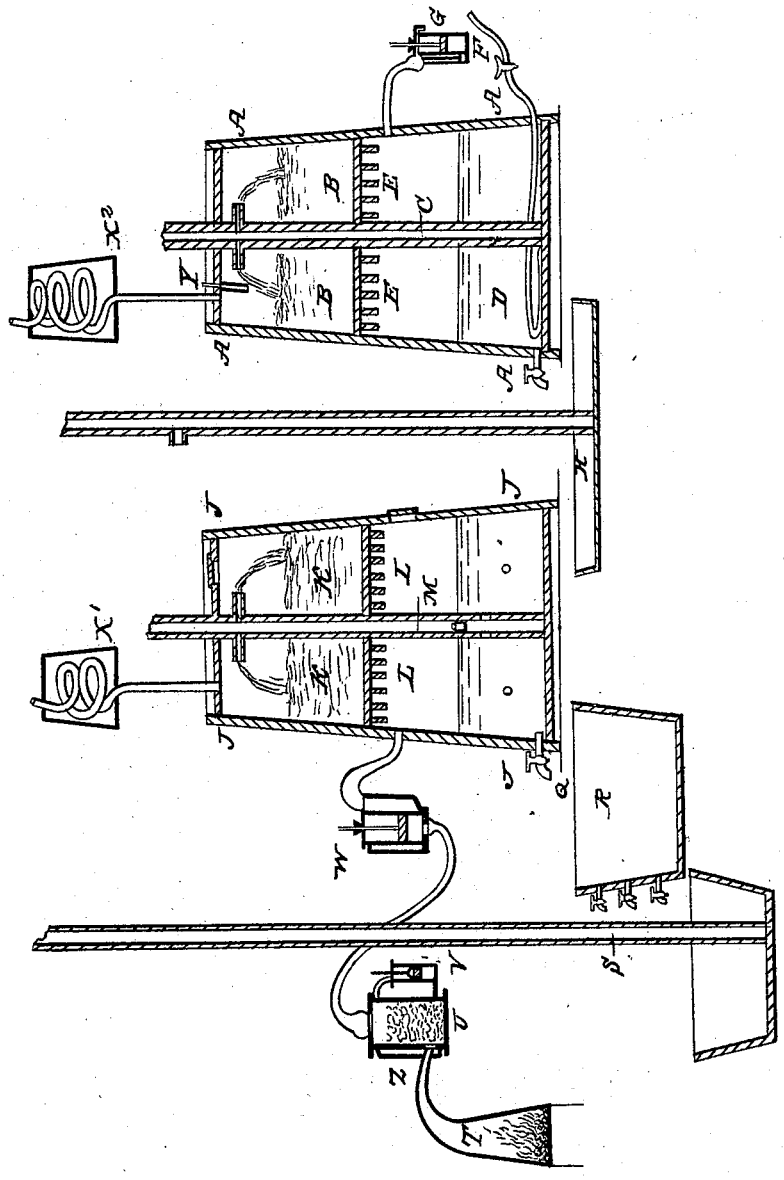
P. PHILLIPS.

Making White Lead.

No. 160. Patented April 17, 1837.



UNITED STATES PATENT OFFICE.

PEREGRINE PHILLIPS, OF CAMPBELL COUNTY, KENTUCKY.

PROCESS OF MANUFACTURING WHITE LEAD.

Specification of Letters Patent No. 160, dated April 17, 1837.

To all whom it may concern:

Be it known that I, PEREGRINE PHILLIPS, of the county of Campbell and State of Kentucky, have invented certain Improvements in the Manufacture of White Lead, and that the following is a full and exact description thereof.

I cause the lead which I wish to make into white lead to be melted and when sufficiently hot (which can be easily known by trying) to be poured in a small stream, or streams, into cold water, when the melted metal will immediately solidify in a great variety of angular and other forms, exposing a large proportion of surface. This process is commonly known by the term "feathering," and the metal in that state is called feathered metal.

If it be preferred the lead may be cast in sheets, and then rolled up, or it may be shotted of various sizes, but I prefer feathering, as exposing more surface with the least trouble. This feathered lead I cause to be placed in a large cask, vat, or other vessel (which to distinguish it I shall call the oxidizing vat) previously fitted with a false bottom, perforated with small holes or any other contrivance that will allow liquid to pass down and yet retain the lead. This false bottom should be elevated in the vessel nearly or quite half way up it and firmly and securely supported there. There is also to be fixed in the said vessel a pump (by preference of wood with a bell metal chamber in it) this pump to be so fixed as to be capable of pumping from the bottom of the said vessel to near the top. If it be preferred, this pump may be situated outside the vat and draw from near the bottom of the vat by means of a pipe reaching from the vessel to the pump and conveying the liquor from the pump by means of a pipe or chute to the top of the vessel; or the pump may be fixed in a tub or well into which the liquor may be let run from the vat by means of a cock or tap, but I prefer having the pump fixed in the vat and as near the center as convenient. There must also be placed in the said vat or vessel a pipe of pure tin, pewter, or lead, (but by preference of pure tin or of pewter) communicating on the outside at one end with a steam boiler. This pipe after passing once or more around the said vat or vessel should pass out something below the level of its entrance for the purpose of allowing the condensed steam to run out. There must also communicate with the said vessel an air force pump or bellows of any construction suitable for forcing atmospheric air, which air should pass into the said vessel, close under the level of the aforesaid false bottom, with the intention of its passing up the interstices of the lead, before mentioned; or if it be preferred, the air may be drawn through the interstices of the lead by leaving a hole or holes above the level of the liquid and under or near the level of the false bottom and applying the pump or bellows to exhaust the air from the top of the lead; taking care to keep the head of the vat quite tight; or the current of air may be formed by taking a pipe from the top of the lead, (aforesaid) to a pipe or chimney, where the air may be rarified by heat or the air may be drawn or forced downward, passing it in above the lead and out below it, but in general I should prefer the action of an air force pump, forcing the air in below the lead and out above it as the most certain and convenient in practice.

The feathered lead, having been placed in the vat, or vessel, and around the pump, any quantity of distilled vinegar, or dilute acetic acid, about the usual strength of distilled vinegar, (that is containing about 2 or 3 per cent. of dry acetic acid) may be also placed in the said vessel, provided it does not rise so high as to interfere with the passage of the air through the interstices of the lead. Then the pump in the center (being perforated all around with holes, which may be fitted with spouts close under the top of the vessel), may be worked by steam, water, or any other power that may be most convenient, with the intention of keeping the feathered lead continually moistened by a current of dilute acetic acid or vinegar passing down, which said vinegar or acetic acid, should be heated when necessary by means of the steam pipe, to about the temperature of 85 to 90 degrees by Fahrenheit's thermometer, at the same time the air pump, or bellows, should be kept continually at work, throwing in atmospheric air below the lead; and which is obliged to pass up the interstices of the lead to which it rapidly communicates oxygen, through the medium of the acetic acid; thus converting the external surface of the lead into oxide of lead, and which oxide of lead is as rapidly dissolved by the acetic acid, and thus converted into acetate of lead; so that

in a short time the liquor which was put in the vat, as dilute acetic acid, becomes converted into a dilute solution of acetate of lead. When the acid becomes fully saturated, it should be drawn off into another vessel to cool, and a fresh supply of dilute acetic acid or distilled vinegar immediately put in to continue the like process, until all the lead is dissolved, or so much diminished, as to require a fresh supply. The time when the acid is saturated with lead will vary according to the relative proportions of acid and lead, but it may be known by trying its specific gravity from time to time by any of the well known methods of taking specific gravity, that may be preferred, the specific gravity of the liquor will gradually increase, until the point of saturation is attained, when it no longer increases, it may be considered as attained.

When the liquor before mentioned has cooled down to the temperature of the atmosphere, or to 60 degrees Fahrenheit (if the atmosphere should be below that) it must be pumped into another vat or vessel, which I shall call the carbonating vat, fitted up somewhat similar to the before mentioned oxidizing vat, omitting the pipe for the steam and substituting for the feathered lead, a quantity of brush wood or siliceous pebbles, or any other substance not acted upon by the acetic acid, and which will at the same time expose a large moistened surface, when the pump situated in the said vat is worked so as to throw the liquor upon the aforesaid brush wood or other substance. This pump also, if it be preferred, may be placed outside the vat, and may be worked in different ways, as I described with reference to the first pump. Below the false bottom which supports the aforesaid brush wood, or other substance, and above the surface of the liquor in the vat, there must be pumped or forced in a quantity of carbonic acid gas, or carbonic acid gas and other air, prepared in a way to be hereafter mentioned, this carbonic acid gas passing up through the interstices of the brush wood or other substance, comes extensively in contact with the solution of lead passing down, decomposes it, forms carbonate of lead commonly called white lead, which is gradually precipitated, in the form of an impalpable white powder; during the process of this operation, the specific gravity of the liquor will gradually decrease, the rapidity of this decrease will be an indication of the rapidity of the formation of white lead. When it is judged from this, that little, or no more, white lead will be precipitated, the whole of the liquor should be let out of the vat, into another vessel, for the final precipitation of the white lead.

If it be preferred the carbonic acid gas may be forced in near the top of the vat and allowed to pass out below, or, it may be drawn through the said vessel, by the action of an air pump exhausting either from above, or below, the false bottom, taking care to let the carbonic acid gas enter at as distant a point as possible, so that it may not be drawn out, by the action of the pump, before it has come extensively in contact with the solution of lead. But I prefer forcing the gas up through the brush wood or other substance, by an air force pump as the most certain and convenient in practice.

When the white lead is precipitated, and the liquor has become clear, the latter should be drawn off, and pumped as soon as convenient again into the first mentioned vat containing the feathered lead, to act again upon the lead, and so continue, again, and again, the same round of operation without end. The precipitated white lead should now be washed with a small quantity of water to free it from any acid, or acetate of lead which water when drained off, should be mixed with a small quantity of strong acetic acid, to make up for the unavoidable loss by evaporation, or other causes, and then passed with the other liquor to the feathered lead. After this the before mentioned white lead, may be more fully washed, then precipitated and dried for sale.

If it should be preferred, the brush wood, siliceous pebbles, or other substances may be altogether omitted from the carbonating vat, and in lieu thereof, a false top, placed a little way under the real top, which false top should be well perforated with small holes, and upon this top, the liquor from the pump, should first strike, and passing down the small holes, fall in numerous streams or drops, down through the atmosphere of carbonic acid and other gases contained in the vat, in a manner somewhat similar to the common, and well known, shower baths. Or a force pump, or pumps, may be used to force the liquor through the atmosphere in the vat, in numerous jets or streams, but I prefer the method first described as the most convenient and economical in practice.

The carbonic acid gas, before mentioned, may be prepared by any of the well known methods, of decomposing any of the alkaline, or earthy, carbonates; or by the fermentation of any saccharin liquor, but I recommend as the most convenient, and economical, that it be prepared by the combustion of well burned charcoal in a close vessel. The heat of the charcoal fire, may be used for the drying of the white lead, and the vapor may be purified from any dust or carbonaceous matter, by causing it to pass through a light vessel (by preference of iron) well filled with rounded pebbles, or brush wood, (by preference pebbles) and containing a small quantity of water, which

water may be made by means of a small force pump, to be continually running over the said pebbles, through which the vapor of the charcoal will be drawn, by the action of the air pump which forces it into the vat.

With respect to the proportions to be observed in the several parts, it must be remembered, that every 104 pounds of lead, will require 8 pounds of oxygen, to supply which will require about 533 cubic feet of atmospheric air, the same quantity of lead in the carbonating vat will require 22 pounds carbonic acid to supply which will require about 975 cubic feet of air, but as we cannot in practice attain this theoretical perfection it will be necessary to have the oxidizing air pump or bellows, capable of throwing in at least from 700 to 800 cubic feet of air for every 100 pounds of lead, that is to be oxidized in a given time, while the carbonating air pump should be capable of throwing from 1400 to 1600 cubic feet air in the same time. The size of the liquor pumps must depend in some measure on the shape of the vats; they should be sufficiently large to throw liquor enough to keep the materials below them, continually in a moistened state; a pump whose box is 5 inches in diameter and which works 14 inches at a stroke and 40 strokes per minute, will with a judicious distribution of the liquor be sufficient for a well proportioned vat of 4,000 gallons. It must also be understood that the oxidizing and carbonating vats should be made as perfectly tight as good materials, and workmanship, can make them; assisted on the outside by a good coat of paint or varnish to prevent the leakage of air. If brush wood should be used for exposing a surface in the carbonating vat, it should be sufficiently scalded previously, to extract its coloring matter.

When the vat containing the lead to be oxidated is large, it will conduce to the better distribution of the liquor over the lead, for there to be placed, a false top, under the real top and over the lead, which false top should be well perforated with small holes, and upon which the liquor from the pump should first strike. It should be also understood that when the metallic lead is known, or suspected, to be impure, the impurities may be separated by filtering the solution of lead, as it comes from the oxidizing vat, or before it goes into the carbonating vat, provided the impurities are not soluble in acetic acid, so that a pure white lead may be made from an impure metallic lead, attended however with a loss in weight in proportion to the impurity.

In drying the white lead I should also recommend that while in a moist state, it be preserved as much as possible from the light to prevent any slight discoloration by the action of the sun's rays upon any acetate of lead, otherwise sugar of lead, which may exist in it in a minute portion and which will not be affected by the light when once dry.

For the better illustration of the nature of my said improvements to persons who may not be conversant with the subject, I annex a sectional drawing of the manner in which the apparatus and utensils may be fixed up, but I wish it to be distinctly understood, that I do not confine myself to that particular arrangement of the utensils nor to any particular shape or size of them, which may be varied to suit the shape or form of the building, and the circumstances and situation of the operator.

Explanation of the drawing.—A A A A, the oxidizing vat containing (B B) the feathered lead; C, the liquor pump which is continually pumping from (D) the liquor that is weak acetic acid, or solution of lead in that acid; E, sections of joists that support the perforated false bottom, upon which rests the feathered lead; F, steam pipe for heating the liquor; G, air pump for throwing in atmospheric air, to oxidize the lead; H, cooler to receive the liquor when sufficiently charged with lead; I, pump to convey the liquor from cooler H to (J J J J) carbonating vat, containing (K K) brush wood or siliceous pebbles resting upon cross lathing, or else a perforated false bottom which is again supported by (L L) the joists, which may rest upon strong trestles; M, the liquor pump which draws from (N) the sucking holes, or entrance for the liquor into the pump, which should be but a short way under the surface of the liquor, to prevent as much as possible the entrance of the precipitated white lead, into the pump. This pump should discharge itself by several holes or spouts as close under the top head of the vat as convenient, and should be worked sufficiently rapid to distribute liquor over nearly all the surface of the brush wood or pebbles; O O, the body of liquor which supplies the pump M and which is continually replenished by the liquor, which having passed up the pump falls down through the brush wood, or pebbles, bringing with it more or less white lead, in mechanical solution; P, man hole in the vat for the purpose of allowing a man to go in, or reach in, when the precipitated white lead cannot all be drained out, with the liquor. This manhole must at all other times be kept air tight by means of a close fitting cover and gasket, or any other contrivance that may be preferred; Q, the cock or tap for running off the liquor and the white lead into (R) one of the tubs in which the white lead is to be finally precipitated: and which should be furnished with several taps, for the purpose of running off the clear liquor, (with-

out disturbing the white lead) into another vessel, in which is placed (S) a pump for conveying the liquor (from which the white lead has been precipitated) back again to vat A A A A A; T, furnace for the production of carbonic acid gas, by the combustion of charcoal, which gas is to be drawn through; U, the gas refiner, a tight vessel by preference or iron nearly filled with pebbles, and containing in the bottom a small quantity of water, which water is thrown by (V) a small force pump, continually over the before mentioned pebbles, for the purpose of keeping them moist, so that they may retain any dust, or smoky particles, from passing through; it will also conduce to the equal distribution of the water, if upon the pebbles, an iron plate be laid, perforated with small holes, upon which plate, the water from the force pump, should first strike; W, the air pump which draws the atmospheric air through the furnace T where by the combination of the charcoal, the oxygen of the air is converted into carbonic acid gas. These gases, that is, the carbonic acid gas, and about four times its bulk of nitrogen gas (derived from the atmosphere) are also drawn, by the action of this same pump, through the gas refiner U, and forced into the vat J J J J; where meeting with the solution of lead in acetic acid, it decomposes it, the carbonic acid gas unites with the oxid of lead, and forms carbonate of lead, otherwise white lead, and the nitrogen passes out through the top where is situated; X^1, a pewter, or a pure tin pipe, or worm, immersed in cold water, for the purpose of condensing any acetic acid, or vapor, that may be carried off by the superfluous gas; X^2, a similar pipe or worm for condensing any acid, or vapor, that may be carried from the vat A A A A A; Y, a long thermometer, 3 or 4 feet long well secured from injury, by being inclosed on all sides by wood, except a small part near the top, for the purpose of ascertaining the temperature of the liquor in the vat A; Z, a small glass tube, communicating both at top, and bottom, with the gas refiner U, for the purpose of showing at a glance of the eye, whether there be any water in it, and how much.

The improvements which I claim as my own invention, and not previously known in this described process of manufacturing white lead, are—

1. The oxidation of the lead, and dissolving the said oxid by means of pumping or throwing diluted acetic acid, or vinegar over lead, when in a state, exposing a large proportion of surface, at the same time forcing or drawing a current of atmospheric air through the interstices of the said lead. And I claim this only for the purpose of preparing a liquor to be used in the manufacture of white lead.

2. The pumping or throwing the aforesaid solution of lead over any substance or materials, placed in an atmosphere composed either wholly or in part of carbonic acid gas, or for pumping or throwing the aforesaid solution into the atmosphere as before said, so that it shall fall down through the said atmosphere, for the purpose of manufacturing white lead.

3. The purifying of the vapor of burning charcoal, by causing the same to pass through a stratum of pebbles, or other substances, which substance, shall be kept moist by the constant, or occasional, injection of water over them. And I claim this only for the purpose of assisting in the manufacture of white lead.

Witness my hand this 14 day of December 1836.

PEREGRINE PHILLIPS.

Witnesses:

MARSTON ALLEN,

W. H. HARRISON.