

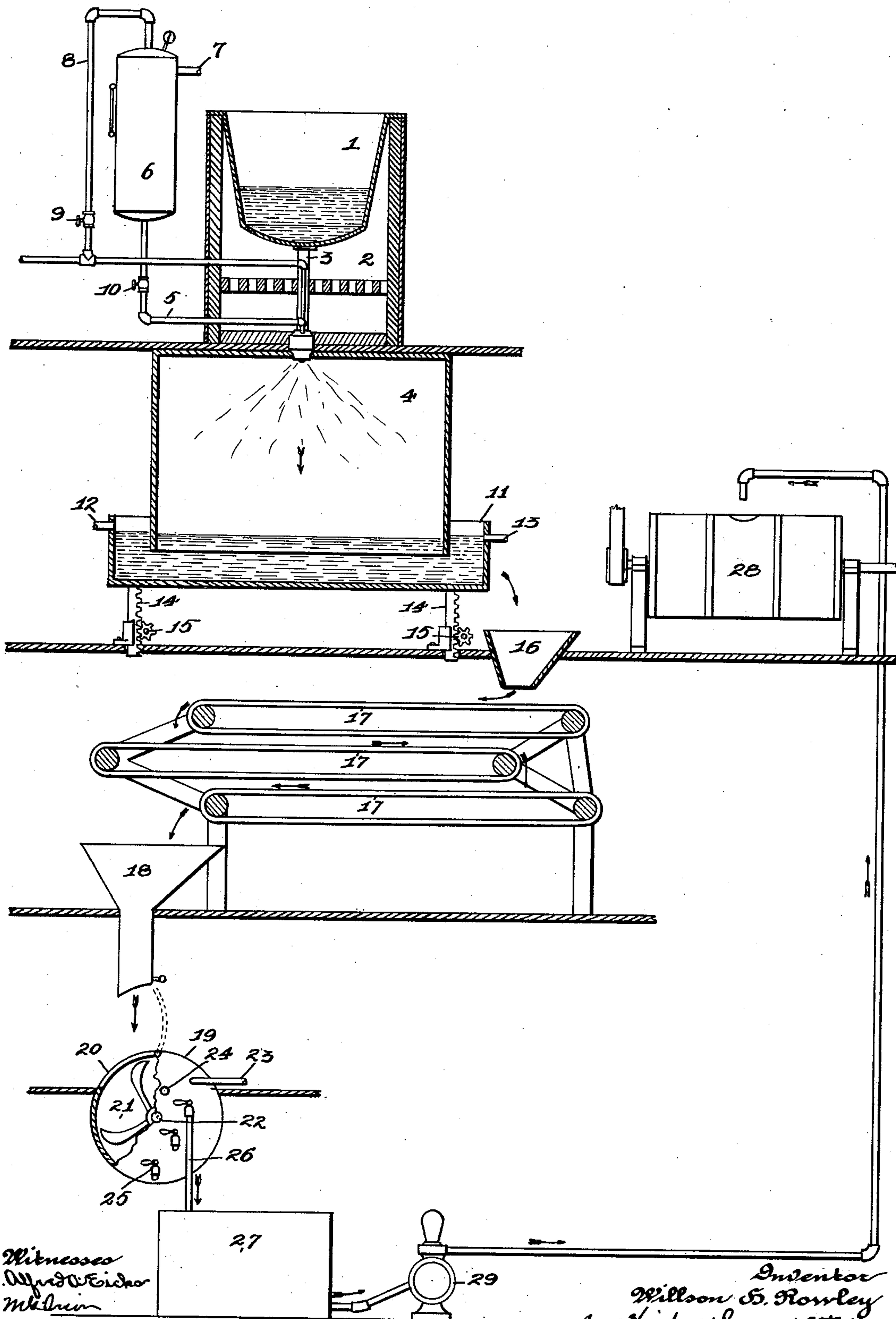
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Patented Nov. 11, 1902.

W. H. ROWLEY.
PROCESS OF MAKING WHITE LEAD.

(Application filed July 21, 1902.)

(No Model.)



UNITED STATES PATENT OFFICE.

WILLSON H. ROWLEY, OF ST. LOUIS, MISSOURI.

PROCESS OF MAKING WHITE LEAD.

SPECIFICATION forming part of Letters Patent No. 713,155, dated November 11, 1902.

Application filed July 21, 1902. Serial No. 116,375. (No specimens.)

To all whom it may concern:

Be it known that I, WILLSON H. ROWLEY, of the city of St. Louis, State of Missouri, have invented certain new and useful Improvements in Processes of Making White Lead, of which the following is a full, clear, and exact description.

My invention relates to a process of making white lead.

10 The object of my invention is to simplify and reduce the cost of the production of white lead and improve the quality of same.

One form of apparatus for carrying out my improved process is shown in the accompanying drawing, in which the figure is a sectional elevation with parts broken away.

15 Pure metallic lead is first melted in a kettle 1, heated by a furnace 2, and the lead is to be kept at the lowest temperature sufficient to retain it in a liquid state and yet permit it to flow readily, and it is then run through a vertical pipe 3 (attached at its upper end to the bottom of the said kettle) downwardly and discharged into the upper end of a suitable air-tight chamber 4, where the lead is atomized (according to a well-known principle) into a lead-powder by coming into immediate contact with moistened steam, which is discharged into said chamber under high pressure. The steam is moistened by the addition of water supplied at the lower end of the pipe 3 by means of a suitable water-pipe 5. Said water-pipe 5 is connected to the lower end of a water-tank 6, and water is supplied to said tank by means of the supply-pipe 7. Steam-pressure is admitted on top of the water in said tank by means of a steam-pipe 8, controlled by the valve 9. Steam to the atomizer is controlled by the valve 10.

40 By means of the atomizer above described the lead is atomized to a powdered state and carried along by the pressure of the steam and discharged forcibly into a basin of cool water 11 at the lower end of the said air-tight chamber 4 and where the lead is immediately covered by the water contained in said basin. The chamber 4 is preferably in the form of an inverted air-tight drum, with its lower end closed by the water contained within said basin 11 in a manner similar to that in which the lower end of an ordinary gas-receiver is closed. The basin 11 is continuously sup-

plied with running water by means of suitable pipes 12 and 13. The water in the basin is thus kept comparatively cool by circulation. The steam under high pressure passes downward in the manner before stated and striking the cool water in the basin 11 is partially condensed and continuously fills the room with moisture, and the lead passing from the atomizer and coming in contact with the moistened steam where the same is discharged from the atomizer passes at high speed through the chamber 4, filled with moisture and devoid of air and is not oxidized to any appreciable extent, it being speedily precipitated into the basin of water. The result will be powdered lead almost absolutely free from any high oxid and containing but slight oxidation, if any. Thus the pig-lead is at practically one operation reduced to a perfectly divided state, being metallic lead powder in water from which can be readily built up by degrees a uniform protoxid of lead without getting any high oxids. After the metallic lead powder in the basin 11 has cooled the water is drained off or otherwise removed and the basin containing it is lowered by means of the racks 14 and pinions 15 or other suitable devices until the said basin 11 is in a plane below that of the lower end of the chamber 4, and then the mass of lead powder contained in said basin is permitted to remain in said basin until it has partially combined with water and oxygen from the remaining water with which it forms a plastic mass. The combination of water and oxygen with the lead will be quite noticeable from the fact that the lead in this massed condition will create considerable heat. When this lead mass has become nearly dry, it is stirred and dried by suitable means in free air, being preferably thrown into a hopper 16 and thence discharged onto a series of slowly-moving drying-aprons 17, and during all this time it is readily changed to lower oxids of lead and forms a dry metallic lead powder with sub-oxid and protoxid of lead containing no high oxids, and then the lead powder is discharged from the apron 17 to another hopper 18 into a drum 19 by way of its door 20, said drum containing a set of plows or agitating blades 21, mounted upon a shaft 22, extending through said drum. The lead is then sprin-

kled with water supplied by a suitable water-pipe 23 until it reaches the form of a paste, and then the plows revolving in said drum carry the lead mass around with a rotary movement and constantly circulate it and present fresh surfaces of the air, which is circulated in the said drum, the air having free access through the door 20, and this door should of course be open during the operation just described. Such operation allows the lead gradually to take on oxygen, and the lead will heat up readily under this treatment to a temperature of about 130° Fahrenheit, which is very favorable for proper oxidation, as the lead constantly agitated by the plows and presented to the oxygen of the air within the drum has its pellicles of oxid removed by attrition, and the general mass is thereby worked up to a uniform protoxid of lead. This drum 19 is also provided with an air-supply pipe 24, through which air may be forced, in which case of course the door 20 should be closed. After the mass within the drum has taken on a general condition of protoxid the door 20 is closed and air is forced into the drum by way of the pipe 24 to finish the oxidation of the mass. The dry lead powder when placed in the said drum 19 has a greenish color, caused by the combination with the lead of water and oxygen under the previously-described treatment and acts as a means to readily assist further oxidation of a uniform condition until the mass assumes a general yellow color, which is indicative of protoxid of lead. By reason of the agitation produced by the plows 21 the lead contained in said drum under the said comparatively high temperature will often stiffen up and should then be sprinkled down with water to keep it in the form of a paste. After reaching this stage of protoxid of lead water is run into the drum while the plows are still agitating the mass, and the pellicles of oxid are thereby more thoroughly separated from those larger particles of lead that are not yet thoroughly oxidized, although they appear in the form of minute particles. It will be found that only the surface of the larger particles has been oxidized, and it is through this rotary motion in water after the oxidation that the protoxid of lead is removed from the metallic lead by the agitation of the plows. This protoxid of lead is washed out of the drum, being discharged by way of the series of cocks 25 and pipe or hose 26 into the tank 27. This protoxid of lead is then discharged into a common rotary cylinder 28 by means of a pump 29, where it is subjected in the usual manner to a stream of carbonic-acid gas to form white lead. As is well known, the uniform protoxid of lead will freely take up carbonic-acid gas and form a basic carbonate of lead.

In determining, as near as possible, the reaction and chemical formula of the mass during manufacture I will say when the lead has been atomized into the basin of water the ag-

gregated mass has a dull dark bluish color, in which can be noticed metallic lead and likewise a black color. No greenish, pink, or brown colors can be found under careful examination. In accounting for this it is clear that the dark bluish color is due to metallic lead and its particles with a pellicle (black) suboxid of lead, (Pb_2O), which is similar to metallic lead ordinarily exposed after being cut with a knife. The absence of the other colors named above should indicate the lead mass void of higher oxids. This lead mass, previously cooled and drained of its water, evolves considerable heat in this wet massed condition and soon loses the water remaining, during which time the bluish tinge is lost to a blacker color, which fades to a dull greenish color when it becomes dry. Since evaporation of water is cooling, this natural heat may represent a chemical change by the lead mass absorbing the water brought about through the condition of the many surfaces for attack and their close contact to one another with only sufficient water to hold the mass in a heavy paste, which if allowed to stand without agitation would become a solid cemented mass. The blacker color indicates the formation of suboxid of lead, (Pb_2O), which is rapidly converted into the protoxid of lead (PbO) when in contact with heat and moisture. Thus the greenish color may be accounted for as a pellicle of the protoxid of lead (PbO) on the particles of metallic lead, which is shown by working part of this mass with water in a mortar, when the greenish color changes to a yellow substance—protoxid of lead—which is rubbed off and floats with the water, leaving the metallic lead. The lead mass agitated in a paste form immediately loses its greenish appearance to the black color before named and gradually changes color to first a dark olive green, which assumes lighter greens until a yellow color becomes general, during which time it takes on high temperature. These conditions may be accounted for by the pellicles of protoxid of lead (PbO) on the particles of lead being rubbed off by attrition and lost sight of through the preponderance of the metallic lead and suboxid of lead which in contact with the oxygen of the air and its chemical heat readily changes the suboxid as it is formed to the protoxid of lead, and thus changes the color of the mass.

I claim—

1. In the manufacture of white lead, atomizing the lead in an inverted air-tight chamber from which the air is excluded, substantially as herein specified.

2. In the manufacture of white lead, atomizing the lead in the presence of moistened steam in an inverted air-tight chamber from which the air is excluded, substantially as herein specified.

3. In the manufacture of white lead, atomizing the lead by means of moistened steam, whereby the same will be brought into im-

mediate contact with moisture as it is atomized, substantially as herein specified.

4. In the manufacture of white lead, atomizing the lead by causing the same to come into intimate contact with moisture as it is atomizing, and thereby covering each particle of lead with a coating of moisture and preventing it from being converted into the higher oxids while in a highly-heated condition, substantially as herein specified.

5. In the manufacture of white lead, atomizing the lead in an inverted air-tight chamber from which the air is excluded and which is filled with moistened steam, and immediately discharging the particles into a cooling-bath of water without allowing the same to come in contact with air, substantially as specified.

6. In the manufacture of white lead, atomizing the lead in an inverted air-tight chamber from which the air is excluded and which is filled with moistened steam, and immediately discharging the particles into a cooling-bath of water without allowing the same to come in contact with air until a mass of metallic lead powder and suboxid of lead is produced in said bath, then draining the water from said mass and permitting the same to absorb oxygen and hydrogen from the associated water, substantially as herein specified.

7. In the manufacture of white lead, atomizing the lead in an inverted air-tight chamber from which the air is excluded and which is filled with moistened steam, and immediately discharging the particles into a cooling-bath of water without allowing the same to come in contact with air until a mass of metallic lead powder and suboxid of lead is produced in said bath, then draining the water from said mass and permitting the same to absorb hydrogen and oxygen from the associated water, and further mildly oxidizing the lead mass by drying the same in free air, substantially as herein specified.

8. In the manufacture of white lead, atomizing the lead in an inverted air-tight chamber from which the air is excluded and which is filled with moistened steam, and immediately discharging the particles into a cooling-bath of water without allowing the same to come in contact with air until a mass of metallic lead powder and suboxid of lead is produced in said bath, then draining the water

from said mass and permitting the same to absorb oxygen and hydrogen from the associated water, and further mildly oxidizing the lead mass by drying the same in free air, and circulating the lead mass until it is dried to a protoxid of lead combined with metallic lead powder, substantially as herein specified.

9. In the manufacture of white lead from a mass of wet mildly-oxidized lead particles, starting an active oxidation therein by agitating the mass in free air until it is substantially dry, thereby oxidizing some of the lead to suboxid of lead and protoxid of lead, then further agitating the mass with water and injecting air into the same to assist the oxidation uniformly throughout said mass, substantially as herein specified.

10. In the manufacture of white lead, the conversion of a general mass of metallic lead and suboxid of lead to a uniform protoxid of lead, by constant agitation while in a paste condition and in contact with free air; building up gradually the general mass to the protoxid of lead, and as the protoxid is formed (as a pellicle on the particles of lead) removing it by the attrition caused through agitation, and thus continually presenting new surfaces for oxidation; working the lead mass in a heavy paste form bringing the particles of lead in close contact with one another during its agitation, to keep the mass in a general condition of metallic lead and protoxid of lead void of higher oxids, as the suboxid of lead formed is rapidly converted into protoxid of lead, allowing none of the particles of lead in the mass an opportunity to form high oxids; working the lead mass by agitation in this condition until gradually a fixed protoxid of lead is formed; then subjecting the mass to a more severe treatment of oxidation by forcing air into the mass in agitation in a drum supplied with a door at its top, said door being open under the milder treatment of free air but closed when the air is forced into the same; and finally washing out and carbonating the protoxid of lead, substantially as herein specified.

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

WILLSON H. ROWLEY.

Witnesses:

ALFRED A. EICKS,
JOHN C. HIGDON.