No. 846,444. PATENTED MAR. 12, 1907.

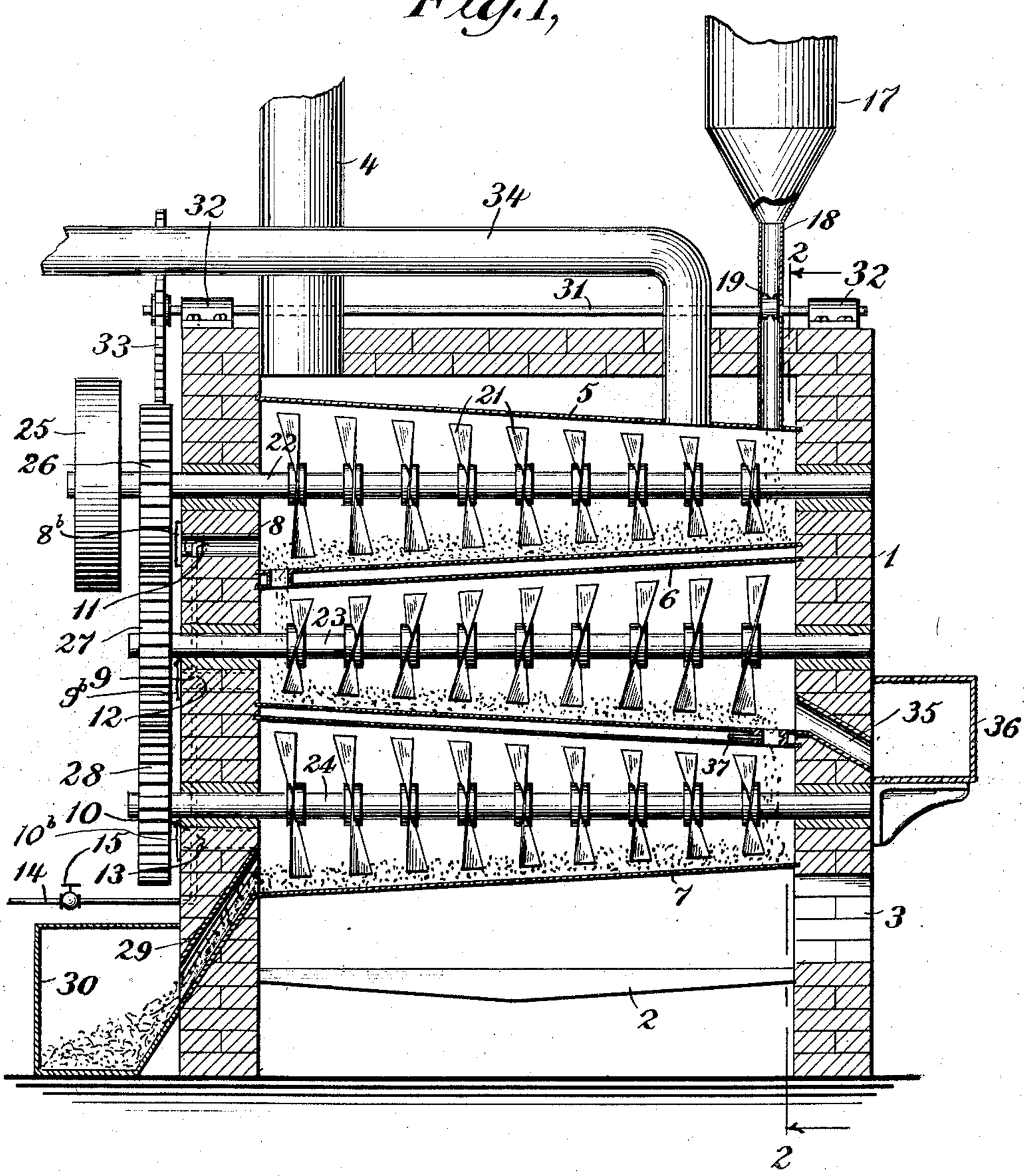
J. W. BAILEY.

PROCESS OF MAKING METALLIC OXIDS.

APPLICATION FILED MAR. 12, 1902.

2 SHEETS—SHEET 1.

Fig. 1,



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2 SHEETS—SHEET 2.

Fig. 2,

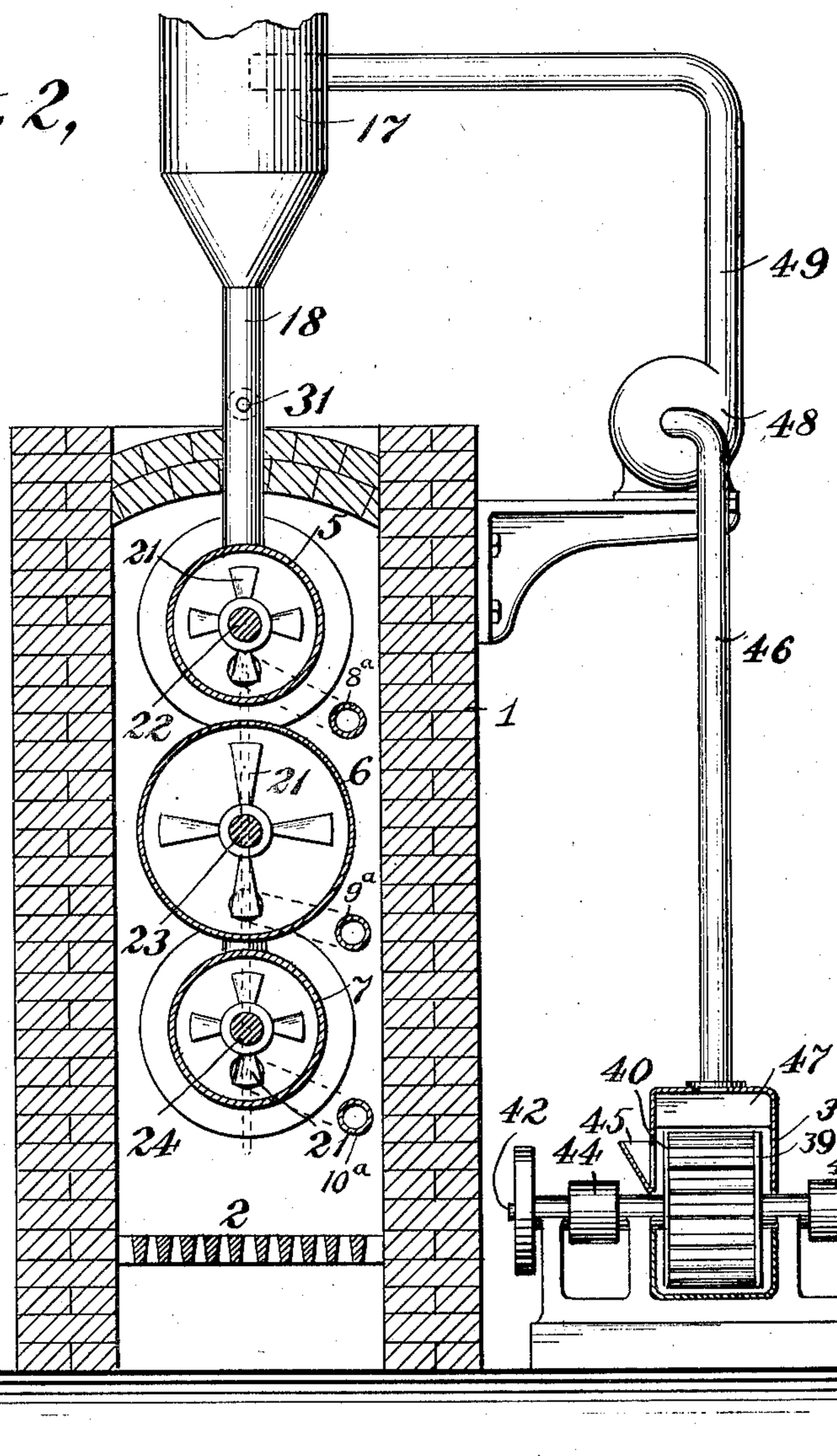
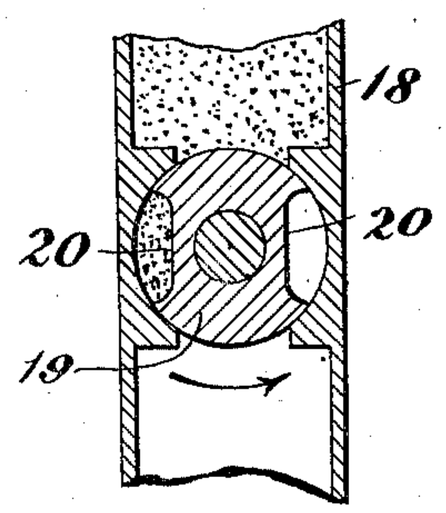


Fig. 3,



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PROCESS OF MAKING METALLIC OXIDS.

No. 846,444. Specification of Letters Patent. Patented March 12, 1907.

Application filed March 12, 1902. Serial No. 97,848.

To all whom it may concern:

Be it known that I, JOHN W. BAILEY, a citizen of the United States, and a resident of Jersey City, in the county of Hudson and State of New Jersey, have invented a certain new and useful Improved Process of Making Metallic Oxids, of which the following is a specification.

My invention relates to a process of making metallic oxids, and more especially to a process of making lead oxids for use as pigments.

The process commonly employed heretofore for making the oxids of lead consists in exposing a mass of the molten metal in a reverberatory furnace to the products of combustion of the fuel mixed with more or less air, the metal being continuously stirred to expose fresh surfaces to the action of the oxidizing agents. Lead has also been oxidized by placing the metal in a finely-divided state in a revolving cylinder through which the products of combustion of an adjoining furnace are conducted. In the case of zinc the common process has been to volatilize the metal either from a molten mass of it or from the decomposition of its ores, the metallic vapor being burned to produce the oxid. Each of these processes is, owing, among other things, to the form in which the metal is treated, slow and tedious and requires a large expenditure of fuel. The product, moreover, is more or less impure, being contaminated with earthy and other matters from the fuel, and it is recovered in a form which requires washing, grinding, and other treatment before it is fit for use.

My invention has for an object to provide a process of making metallic oxids which is simple and easily carried into effect and every step of which, especially the oxidation of the metal, is rapidly performed and is under perfect control, also one which may be carried out continuously and with a minimum expenditure of labor, also one which is economical in the use of fuel and in which the oxidation of the metal is rapidly effected, also to provide a process the product of which is preferably pure and is produced in a form suitable for use without further treatment. These and other objects of the invention will more fully appear from the following description.

My invention is based on the discovery that metallic lead when divided in the usual manner, as by blowing a jet of steam or compressed air through a stream of the molten metal, can be further reduced to a very fine dust or impalpable powder by means of a suitable pulverizing or attrition mill and when thus finely divided can be quickly and economically reduced to the state of an oxid by being exposed to the action of a current of hot air or of hot air and moisture. When the metallic powder is thus treated in a closed muffle that is suitably heated without subjecting the metal to the action of the products of combustion, a purer product is produced which is of a superior quality and is in a suitable condition for use without further treatment—such as grinding, washing, &c.—and this product is produced in much less time than is required to carry out the processes in common use.

In order that my invention may be fully understood, reference is made to the accompanying drawings, in which is illustrated one form of apparatus by which my invention may be carried into effect, it being understood that my process is not restricted to the use of the apparatus which I am about to describe or to any particular apparatus or means of performing the various steps of the process.

Of the drawings, Figure 1 is a vertical longitudinal central sectional view of a suitable form of apparatus devised by me for carrying my process into effect, said apparatus forming the subject-matter of a companion application, Serial No. 97,849, filed March 12, 1902. Fig. 2 is a vertical transverse section of the apparatus, taken on the line 2 2 of Fig. 1; and Fig. 3 is a sectional view illustrating a detail.

Like reference-numerals refer to like parts wherever they occur throughout the drawings.

This apparatus consists, essentially, in the combination, with a closed muffle, of means for heating the same, means for maintaining a current of air in the muffle, means for introducing finely-comminuted metal into one end of the muffle, means for advancing the metal therethrough, and a discharge-passage for the oxidized or partly-oxidized metal communicating with the other end of the muffle. In accordance with the preferred form of the apparatus there is also provided means for supplying moisture to the interior

of the muffle and for agitating the metal as it is advanced therethrough. In the best embodiment of the apparatus a plurality of muffles are provided, the same being arranged to communicate with each other in series. In order that this series of muffles may be suitably heated and maintained at progressively-increasing temperatures, they are arranged one above another in a suitable furnace, the means for introducing the metal being connected to the upper muffle of the series and the discharge-passage for the oxid being connected to the lower muffle of the series. The means for supplying the moisture consists of a series of steam-jets, one for each muffle, arranged in the air-supply passage so as to act at the same time to induce a current of air through each muffle and to suitably heat the air as it is introduced.

Referring to the drawings in detail, 1 represents the inclosing walls of a suitable furnace, and 2 represents the grate-bars thereof, to which the fuel is supplied through a suitable opening 3.

4 is the uptake or escape-pipe for the products of combustion.

In the apparatus shown three muffles 5, 6, and 7 are provided, each consisting of a horizontally-arranged longitudinally-tapering shell which is fixed at its opposite ends in the walls of the furnace, the various muffles of the series being, as regards their taper, alternately arranged in reverse positions. For the purpose of supplying air to the muffles air-passages 8, 9, and 10 are provided in one of the walls of the furnace, one passage for each of the muffles. In each of these air-passages is provided an injector-nozzle, said nozzles being numbered, respectively, 11, 12, and 13. In order that the currents of air induced by these injector-nozzles may be suitably heated and at the same time supplied with a suitable amount of moisture, the injector-nozzles are preferably connected to a steam-supply pipe 14, which is provided with a suitable regulating-valve 15.

17 represents a supply reservoir or hopper for the finely-divided metal, said hopper communicating at its lower end with the supply-pipe 18, which passes down through the top of the furnace and enters the upper muffle 5, near the small end thereof, as shown. In the supply-pipe 18 is mounted a suitable feeding device, the same consisting in the construction shown of a wheel or cylinder 19, which, as shown in Fig. 3, is fitted to a suitable chamber in the supply-pipe and is provided with one or more circumferential recesses 20, adapted upon the revolution of the wheel to supply the finely-comminuted metal to the muffle in regulated quantities.

For the purpose of agitating the metal in the muffles, so as to thoroughly and uniformly expose all the parts thereof to the air in the muffles, each of them is provided with a suitable agitating device. The agitating device, as shown, consists of a series of radial blades 21, arranged at intervals along the shafts 22, 23, and 24, centrally arranged in the muffles 5, 6, and 7, respectively. The blades 21 are made successively longer from one end of each of the shafts to the other, so that their ends will all come in close proximity to the interior walls of the muffles. In order that the blades may act to advance the metal through the muffles, as well as to agitate the same, the blades are preferably flattened at their ends and arranged at such an angle to the axis of the shafts as to advance the metal with which they come in contact to a greater or less amount, dependent upon the speed of rotation of the shaft. This advancing movement of the metal is also facilitated somewhat by the inclination of the bottoms of the muffles, due to their tapering form. The shafts 22, 23, and 24 are driven by any suitable means, that shown consisting of a pulley 25 on the upper shaft and the gears 26, 27, and 28, which transmit the motion of the upper shaft 22 to the lower shafts 23 and 24.

29 indicates a suitable outlet-passage for the oxidized metal, the same communicating at one end with the larger end of the lower muffle 7 and at the other end with a suitable receptacle 30 for the finished product.

In order that the metal may be supplied to the muffles in automatically-regulated quantities, the feeding-cylinder 19 is mounted on a shaft 31, which is journaled in suitable bearings 32 and is provided at one end with a gear 33, which meshes with the gear 26 on the shaft 22.

34 indicates an escape-pipe for the air supplied to the muffles by the air-passages 8, 9, and 10, provision being thus made to maintain a continuous current through each of the muffles. The pipe 34 preferably communicates near the small end of the upper muffle 5, and in order that any particles of the metallic dust or of the oxid which is carried out by the current of air may be recovered the air-pipe 34 should terminate in a dust-settling chamber which, as it forms no part of my present invention, and as it may be of any of the usual constructions, is not shown or described herein.

In Fig. 2 is indicated a form of pulverizing-mill adapted to reduce comminuted metal to an impalpable powder. As shown, this mill consists of a suitable casing 38, in which are mounted a pair of rotary heads 39 and 40, the same being mounted on oppositely-arranged alined shafts 41 and 42, which are driven in opposite directions by pulleys 43 and 44.

45 is a feed-hopper through which the metal in a fine sand-like form is introduced into the mill.

A discharge-pipe 46 communicates with an enlarged chamber 47, formed at the top of

the casing 38. The discharge-pipe 46 communicates with the suction-port of a suitable blower 48, the delivery-port of which is connected by a suitable pipe 49 to the chamber 17.

In accordance with my process the metallic lead is reduced in the mill described to such a fine state that it will be floated in the air and carried thereby to a suitable settling-chamber, which may be the supply-chamber 17 of the oxidizing apparatus. The heat of the furnace is so regulated that the upper retort 5 is maintained at a temperature somewhat below that at which the metal fuses. The lower muffles 6 and 7 are preferably maintained at a somewhat higher temperature than that of the upper muffle 5, and the lowest muffle, in which the lead has all been changed to the form of an oxid, may be maintained at a temperature considerably higher than that at which the metal fuses; but it should not be heated to such a temperature as to fuse the oxid therein. The supply of steam to the injector-nozzles is so adjusted as to supply such a quantity of air and moisture to the muffles as will cause the metallic particles to become gradually oxidized, and the speed of rotation of the agitating devices is so regulated as to advance the metal through the muffles at such a rate of speed that when the metal reaches the outlet-passage it will be oxidized to the desired extent. If, for instance, it is desired to produce litharge, (PbO,) the agitating devices will be run at such a speed that the metal will have been oxidized to that extent only when it is discharged. If, however, it is desired to produce red lead, (Pb_3O_4,) the agitating devices will be operated at a slower speed, so that the lead will be further oxidized to the desired extent. The degree of oxidation and the rapidity thereof may of course be also regulated more or less by varying the temperature of the furnace and by varying the temperature and the amount of the steam and air supplied to the chambers, it being within the judgment and skill of the operator to determine which of these elements of regulation can best be made use of to produce the desired result. If desired, moreover, additional heating devices may be provided for increasing the temperature of the air supplied to the muffles. Such heating devices are shown in Fig. 2, wherein 8^a, 9^a, and 10^a indicate air-supply pipes which pass through the furnace and communicate with the air-supply passages 8, 9, and 10, respectively. Cut-offs 8^b, 9^b, and 10^b are provided to close the outer ends of the passages 8, 9, and 10 when it is desired to use the supplemental heaters. In fact, all of the heat provided to expedite the oxidation of the metal may be supplied to the air, if desired. If desired, moreover, additional outlet-passages for the product may be provided, the same being arranged to communicate with various parts of the muffles in order to discharge the metal therefrom when it has reached the desired degree of oxidation. Such a passage is indicated at 35 in Fig. 1, the same communicating at one end with the larger end of the intermediate muffle 6 and at the other end with a suitable receptacle 36. The passage between muffles 6 and 7 is closed by a cut-off 37 when the oxid is to be delivered from muffle 6.

It is obvious that a greater or less number of the muffles may be used, if desired.

It will be observed that in accordance with my invention the process of oxidation of the metal is a gradual one and under perfect control from start to finish. Owing principally to the fine condition of the metal, however, it is oxidized more rapidly and more uniformly than by the processes heretofore in common use. The product, moreover is discharged from the apparatus in a very pure and finely-divided or amorphous condition and is ready for use without further treatment.

What I claim as new, and desire to secure by Letters Patent, is—

1. The process of making an oxid of a metal, which process consists in reducing the metal to an impalpable metallic powder and then slowly oxidizing the powder by heating it in the presence of an oxidizing agent.

2. The process of making an oxid of a metal, which process consists in reducing the metal to an impalpable metallic powder and then oxidizing the powder by subjecting it to the action of heated air.

3. The process of making an oxid of a metal, which process consists in reducing the metal to an impalpable metallic powder and then subjecting the powder to the action of an oxidizing agent in such manner as to oxidize the metal without vaporization of the oxid.

4. The process of making an oxid of a metal, which process consists in reducing the metal to an impalpable metallic powder and then subjecting the powder to the action of heated air in such manner as to oxidize the metal without vaporization of the oxid.

5. The process of making an oxid of a metal, which process consists in reducing the metal to an impalpable metallic powder and then heating the powder in a closed chamber to a temperature below the fusing-point of the metal and at the same time subjecting the metal to the action of an oxidizing agent.

6. The process of making an oxid of metallic lead suitable for pigment, said process consisting in reducing the lead to an impalpable metallic powder and then slowly oxidizing the powder.

7. The process of making an oxid of metallic lead suitable for pigment, said process consisting in reducing the lead to an impalpable metallic powder and then slowly oxi-

dizing the powder by subjecting it to the action of a heated oxidizing agent.

8. The process of making an oxid of metallic lead suitable for pigment, said process consisting in reducing the lead to an impalpable metallic powder and then subjecting the powder to the action of an oxidizing agent in such manner as to oxidize the metal without vaporization of the oxid.

9. The process of making an oxid of metallic lead suitable for pigment, said process consisting in reducing the lead to an impalpable metallic powder and then heating the powder in a closed chamber to a temperature below the fusing-point of the metal and at the same time subjecting the metal to the action of an oxidizing agent.

10. The process of making an oxid of a metal, which process consists in reducing the metal to an impalpable metallic powder, heating the powder in the presence of an oxidizing agent, and agitating the powder to uniformly expose all parts thereof to the oxidizing agent.

11. The process of making an oxid of a metal, which consists in reducing the metal to an impalpable metallic powder and then heating the powder in the presence of air and moisture.

12. The process of making an oxid of a metal, which consists in reducing the metal to an impalpable metallic powder and then heating the powder in the presence of a current of air and steam.

13. The process of making an oxid of a metal which consists in reducing the metal to an impalpable metallic powder and then heating the powder in a muffle and at the same time subjecting it to the action of a current of heated air and steam.

14. The process of making an oxid of a metal, which consists in heating the metal in the form of an impalpable powder to a temperature below the fusing-point in a closed muffle and at the same time subjecting the metal to the action of a current of heated air.

15. The process of making an oxid of a metal, which consists in heating the metal in the form of an impalpable powder to a temperature below the fusing-point in a closed muffle and at the same time subjecting the metal to the action of a current of heated air and steam.

16. The process of making an oxid of a metal, which consists in heating the metal in the form of an impalpable powder to a temperature below the fusing-point in a closed muffle and at the same time agitating the metal and subjecting the metal to the action of a current of heated air.

17. The process of making an oxid of a metal, which consists in heating the metal in the form of an impalpable power to a temperature below the fusing-point in a closed muffle and at the same time agitating the metal and subjecting the metal to the action of a current of heated air and steam.

18. The process of making an oxid of a metal, which process consists in heating the metal in the form of an impalpable powder in such manner as to gradually transform the metal into the character of oxid desired.

19. The process of making an oxid of a metal, which process consists in gradually oxidizing the metal in the form of an impalpable powder by subjecting the powder to the action of an oxidizing agent and moisture.

20. The process of making an oxid of a metal, which consists in subjecting the metal in the form of an impalpable powder to the action of a current of air and moisture heated to a temperature lower than that at which the metal fuses.

21. The process of making lead oxid suitable for pigment, said process consisting in reducing the lead to an impalpable metallic powder and then heating the powder in the presence of air and moisture.

22. The process of making lead oxid suitable for pigment, said process consisting in reducing the lead to an impalpable metallic powder and then heating the powder to a temperature below the fusing-point of the metal in the presence of air and moisture.

23. The process of making an oxid of a metal, which consists in reducing the metal to an impalpable metallic powder, feeding the powder and steam into a heated muffle in regulated quantities and subjecting the powder to the action of a current of heated air.

24. The process of making an oxid of a metal, which consists in reducing the metal to an impalpable metallic powder, feeding the powder and steam in regulated quantities into a closed muffle heated to a temperature below the fusing-point of the metal, agitating the powder in the muffle and at the same time subjecting the powder to the action of a current of heated air.

25. The process of making an oxid of a metal whch consists in reducing the metal to an impalpable metallic powder and then subjecting the powder to a gradually-increasing temperature in the presence of air and moisture.

26. The process of making an oxid of a metal which consists in reducing the metal to an impalpable metallic powder and then subjecting the powder to a gradually-increasing temperature in the presence of a current of heated air and steam.

27. The process of making an oxid of a metal which consists in reducing the metal to an impalpable metallic powder, then heating the powder to a temperature below the fusing-point of the metal in the presence of air, and then as the metal becomes oxidized increasing the temperature thereof.

28. The process of making an oxid of a metal which consists in reducing the metal to an impalpable metallic powder, then heating the powder to a temperature below the fusing-point of the metal in the presence of air and moisture, and then as the metal becomes oxidized increasing the temperature thereof.

29. The process of making lead oxid, which process consists in heating the lead in the form of an impalpable powder in such manner as to gradually transform the lead into the character of oxid desired.

30. The process of making lead oxid, which process consists in gradually oxidizing the lead in the form of an impalpable powder by subjecting the powder to the action of an oxidizing agent and moisture.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

JOHN W. BAILEY.

Witnesses:

SIDNEY MANN,

T. E. RAFTERY.