Jan. 2, 1934. B. N. McCRAVEN 1,941,569

MANUFACTURE OF ZINC OXIDE

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Fig. 1,

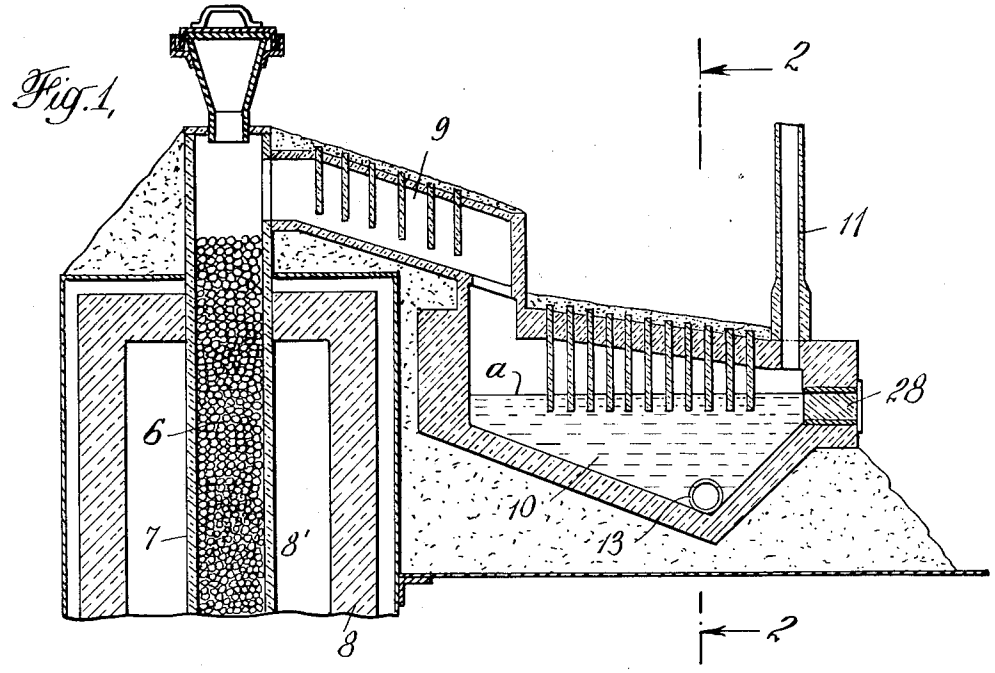
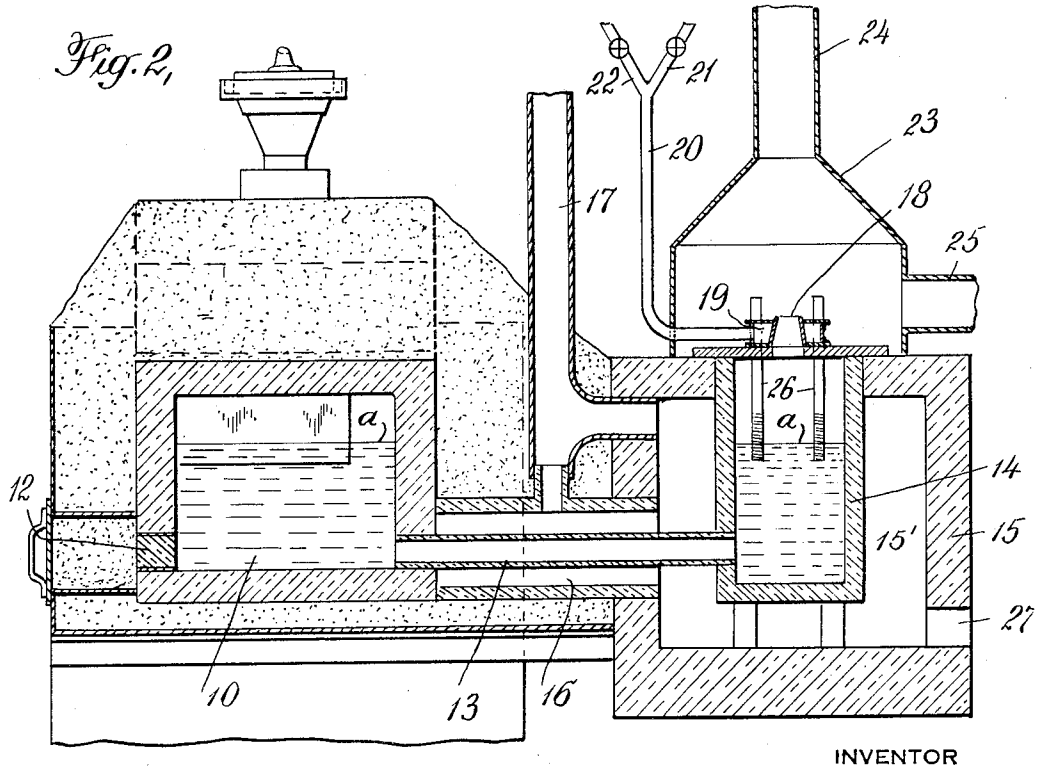


Fig. 2,



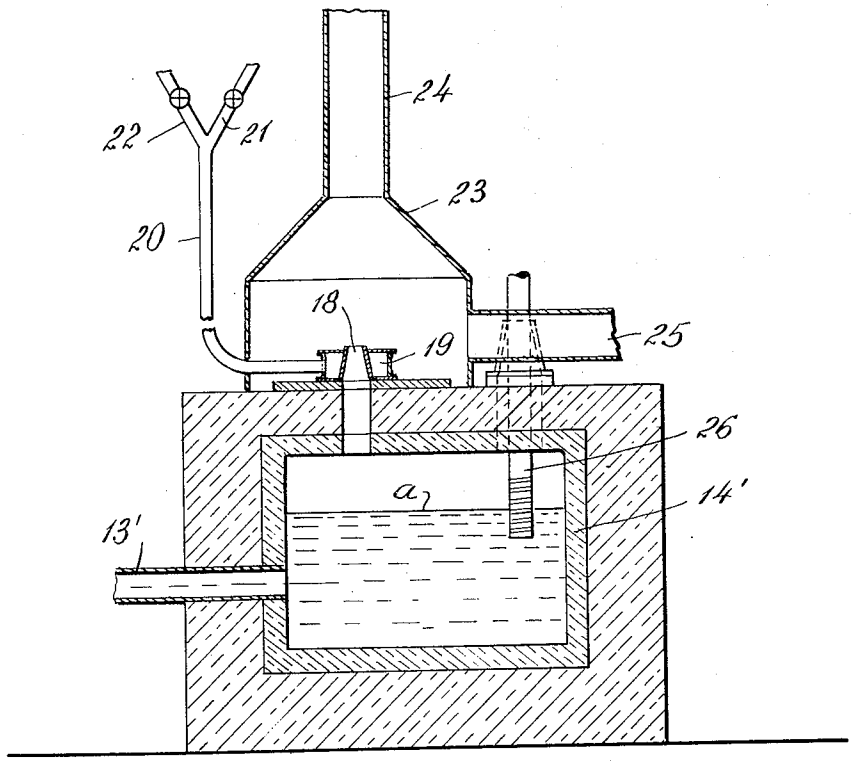
INVENTOR
Bonner N. McCraven
BY
Pennie, Davis, Marvin & Edmonds
ATTORNEYS

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Fig. 3,



INVENTOR
Bonner N. McCraven
BY
Pennie, Davis, Marvin & Edmonds
ATTORNEYS

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UNITED STATES PATENT OFFICE

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MANUFACTURE OF ZINC OXIDE

Bonner N. McCraven, Bloomfield, N. J., assignor to The New Jersey Zinc Company, New York, N. Y., a corporation of New Jersey

Application February 24, 1930. Serial No. 430,839

5 Claims. (Cl. 23—148)

This invention relates to the manufacture of zinc oxide, and has for its object the provision of an improved method of making zinc oxide.

The invention is particularly applicable to the production of zinc oxide from zinc vapor derived directly from a reducing or smelting operation. The zinc vapor produced by a reducing or smelting operation is diluted with carbon monoxide gas and contaminated with sulfur originating usually in the carbonaceous reducing agent (such as coal or coke). Zinc oxide resulting from the direct oxidation of such zinc vapor is accordingly contaminated with adsorbed sulfur compounds and resembles in quality the zinc oxide produced directly from ore by the so-called American process and is inferior in quality to French process zinc oxide (made from zinc metal) largely on account of its sulfur content. One of the principal aims of the present invention is to produce zinc oxide from a reducing or smelting operation without contamination with sulfur compounds. In accordance with the invention, the zinc vapor from the reducing or smelting operation is condensed in a condenser of appropriate capacity, whereby the carbon monoxide and sulfur gases are eliminated through the exhaust gas outlet of the condenser. The resulting molten zinc metal is permitted to flow from the condenser into a volatilization chamber maintained at a sufficiently high temperature to volatilize zinc and deliver the resulting zinc vapor to an appropriate oxidizing environment, and the resulting zinc oxide is collected in any appropriate manner.

The invention in its broad aspect is applicable to the production of zinc oxide from any source of zinc vapor. The zinc vapor is first condensed to molten zinc metal and the molten metal is delivered to a volatilization chamber. Preferably, the condensation of the zinc vapor takes place in a condenser of relatively large capacity having a bath of molten zinc in free communication with the bath of molten zinc in the volatilization chamber. Optimum results are attained when a substantially continuous supply of zinc vapor is delivered to the condenser, and a substantially continuous stream of zinc vapor issues from the volatilization chamber. It is my preferred practice to carry out the oxidation of the zinc vapor in an enclosed environment to which clean oxidizing gas is admitted in appropriate volume. Preferably oxidation of the zinc vapor is effected by a thermally regulated blast of oxidizing gas. These and other novel features of the invention will be best understood from the following description taken in conjunction with the accompanying drawings, in which

Fig. 1 is a sectional elevation of an apparatus adapted for the practice of the invention;

Fig. 2 is a sectional elevation of the same apparatus on the section line 2—2 of Fig. 1; and

Fig. 3 is a sectional elevation of a slightly modified volatilization chamber.

A vertical retort smelting operation is illustrated in the drawings as the source of the primary or initial zinc vapor. This smelting operation may advantageously be carried out as described in U. S. Patent 1,712,132 by progressively passing an agglomerated charge 6 of mixed zinciferous and carbonaceous materials through a vertical retort 7 within the heating chamber or laboratory 8' of an appropriate furnace structure 8. The upper end of the vertical retort 7 communicates with a down-draft condenser consisting of a baffled condensing chamber 9 and a communicating baffled condensing and collecting sump 10, generally of the type described in the copending patent application of Earl H. Bunce, Serial No. 400,064, filed October 16, 1929. The sump 10 has a chimney or stack 11 for the exhaust of gases, other than zinc vapor entering the condenser.

The bottom of the sump 10 is inclined lengthwise from each end and is provided on one side with a tap hole 12 at the apex of the inclined bottom surfaces. On the other side, at the apex of its inclined bottom surfaces, the sump is connected by a conduit 13 with a volatilization chamber or retort 14 appropriately mounted within a furnace structure 15. The conduit 13 is surrounded by a heating laboratory 16 in communication with the heating laboratory 15' surrounding the chamber 14, and the exhaust heating gases from the two laboratories are conveyed to a common stack or chimney 17.

The volatilization chamber 14 is closed at its top except for a vapor discharge nozzle 18. The nozzle 18 is surrounded by a wind box 19 adapted to direct an annular blast of oxidizing gas (such as air) against the stream of zinc vapor issuing through the nozzle, as more particularly described in U. S. Patent 1,522,097. Compressed air is supplied to the wind box by a pipe 20 communicating with a valved cold air supply pipe 21 and a valved hot air supply pipe 22. A hood 23 is mounted above the volatilization chamber and constitutes an enclosed oxidizing environment for the zinc vapor issuing through the nozzle 18. The hood is connected by an off-take pipe 24 to a bag-room or other zinc oxide collecting means (not shown).

Clean oxidizing gas is supplied to the interior of the hood 23 through a pipe 25, and a fan or other appropriate suction means is associated with the off-take pipe 24 to induce the desired suction or draft within the oxidizing environment provided by the hood 23.

A pair of hollow graphite resistors 26 extend through the top of the chamber 14 into the bath of molten metal in the chamber. The resistors are provided (for an appropriate length) with spiral convolutions, as more particularly described in U. S. Patent 1,674,947. The resistors are connected to an appropriate source of electric energy.

In Fig. 3 of the drawings, the volatilization chamber 14' and conduit 13' are surrounded with a relatively thick layer of heat insulating material, in order to dispense with the heating laboratories illustrated in Fig. 2. The resistors 26 are positioned outside the hood 23 and near one end of the volatilizing chamber.

In practicing the invention in the apparatus illustrated in the drawings, the zinc vapor from the vertical smelting retort 7 is condensed to molten zinc metal in the condensing chamber 9 and condensing sump 10. The carbon monoxide and sulfur gases entering the condenser with the zinc vapor escape through the exhaust stack 11. The condensed molten zinc collecting in the sump 10 flows through the conduit 13 into the volatilizing chamber 14 so that the level of molten metal (a) is the same in both the sump 10 and chamber 14. Hot gases at a temperature of from 900–1000° C. (preferably the exhaust heating gases from the reducing furnace 8) enter the heating laboratory 15' through an inlet port 27 and pass around the chamber 14 and conduit 13 and assist in heating the molten zinc in the chamber. The further heat required for volatilizing zinc at adequate velocity is supplied by the electric resistors 26 which dip into the bath of molten zinc in the volatilization chamber.

The arrangement of the electric resistors 26 makes it possible to determine at any given time the exact level (a) of molten zinc in the condensing sump 10 and to regulate this level with precision. Since the sump 10 communicates freely with the chamber 14 through the conduit 13 the level of molten zinc in both the sump and chamber is always the same. As the depth of the bath of molten zinc in the chamber 14 increases, the resistors 26 become more deeply immersed in the molten metal and the effective length of the resistors is shortened so that an increased electric current flows through the resistors and more heat is evolved, and the quantity of zinc volatilized is accordingly increased. As the level of molten metal in the chamber 14 falls, the effective length of the resistors is increased, thus increasing the electric resistance and decreasing the current flow, so that less heat is supplied to the bath of molten metal, and the quantity of zinc volatilized accordingly decreases. In consequence, the velocity with which zinc is volatilized varies directly with the depth of the bath of molten zinc in the chamber 14, increasing as the depth increases and decreasing as the depth diminishes. This in itself tends to create an automatic regulation of the level of molten zinc in both the sump and the volatilization chamber.

If too much molten zinc builds up in the sump 10, so that the automatically increased supply of heat from the resistors 26 cannot compensate for the increase by volatilizing more zinc from the chamber 14, molten zinc may be tapped from the sump through the tapping hole 12 or a tapping hole 28 at one end of the sump approximate the normal level of molten metal therein. In case it is desired to produce metallic zinc and zinc oxide simultaneously, the amount of heat supplied to the volatilization chamber by the resistors is deliberately kept below the amount required to volatilize all the zinc collecting in the sump, and the excess molten zinc is tapped off periodically as it accumulates in the sump.

The molten zinc metal in the volatilization chamber 14 is maintained at a sufficiently high temperature to volatilize zinc and deliver the resulting zinc vapor through the nozzle 18 in a substantially continuous stream. As the stream of zinc vapor issues from the nozzle it encounters the annular blast of oxidizing gas from the wind box 19, and the zinc is immediately burnt or oxidized to zinc oxide. The suspended particles of zinc oxide are drawn through the off-take pipe 24 into the oxide-collecting system.

Zinc oxide produced in accordance with the invention is of high quality comparable with high grade French process oxide, and is devoid of the contaminating sulfur compounds associated with zinc oxide made in accordance with the American process. The method of the invention makes it possible to control the particle size of the zinc oxide produced to meet the demands of both a paint and rubber pigment. This regulation of particle size can be secured by regulating the intensity and/or the temperature of the air blast supplied by the wind box 19. The cold air supplied by the pipe 21 and the hot air supplied by the pipe 22 can be mixed in any desired proportions to feed air of a predetermined temperature into the wind box 19. The wind box is preferably provided with a thermometer or other temperature indicating means for continuously indicating, and recording if desired, the temperature of the air flowing through the wind box. The air in the hot air pipe 22 may be heated by carrying the pipe through the heating laboratory 15' of the furnace structure 15, or a compressed air reservoir may be thermally associated with the heating laboratory or the exhaust gases therefrom.

The air supplied by the wind box is not adequate to transport the zinc oxide formed through the off-take pipe 24 to the oxide-collecting means, and it is therefore necessary to supply other clean air to the oxidizing environment. In the apparatus illustrated in the drawings, this is effected by the air inlet pipe 25 which conducts clean air, taken from a point at a distance from any source of soot or dust, and preferably cleaned by air filters, into the oxidizing environment within the hood 23.

The invention, practiced in the apparatus illustrated in the drawings, is adapted for the production of zinc metal and zinc oxide of any desired particle size without any substantial readjustment in passing from one operation to the other. All or any part of the zinc vapor derived from the smelting furnace can be recovered as either zinc oxide or zinc metal. The volatilization chamber cooperates advantageously in maintaining proper operation conditions of the condenser of the smelting operation. The presence of a controlled amount of molten zinc in the condenser is necessary for optimum condensing conditions. The volatilization chamber acts both as a means of automatically regulating the depth of molten metal in the condenser, and as an indicator of the amount of molten metal actually present in the condensing sump, thus

rendering it more easy to operate the condenser efficiently.

I claim:—

1. A method of manufacturing zinc oxide which comprises subjecting a charge of zinciferous material to a reduction operation that results in the liberation of zinc vapor and the evolution of gases, condensing the zinc vapor to a bath of molten zinc while permitting the gases to escape from the system, continuously flowing molten zinc from said bath of molten zinc by gravity while substantially conserving its heat and unaccompanied by gases and not in contact with air into a second bath of molten zinc excluded from contact with said gases, volatilizing the said second bath of molten zinc excluded from contact with gases to zinc vapor, and burning the resulting zinc vapor to zinc oxide.

2. A method of manufacturing zinc oxide according to the preceding claim, which comprises utilizing the exhaust heat of the reduction operation to supply heat to the molten zinc volatilization operation.

3. A method of manufacturing zinc oxide according to claim 1, which comprises maintaining the second bath of molten zinc substantially constant in volume by volatilizing zinc therefrom at substantially the same rate as zinc vapor is condensed in the first bath of molten zinc.

4. A method of manufacturing zinc oxide according to claim 1, which comprises effecting the volatilization of the second bath of molten zinc by heat supplied by resistance electrodes dipping into the bath of molten zinc so that a circuit is completed through the bath of molten zinc, whereby an increase in the depth of the bath of molten zinc shortens the length of resistor to be traversed by electric current, then increasing the flow of current and the supply of heat so as to increase the amount of zinc volatilized, and whereby a decrease in the depth of the bath of molten zinc extends the length of resistor to be traversed by the electric current, thus increasing the flow of current and the supply of heat so as to decrease the amount of zinc volatilized, whereby the depth of the first and second baths of molten zinc is automatically regulated with resulting increase in efficiency of the condensation operation.

5. A method of manufacturing zinc oxide, which comprises reducing zinciferous material to produce zinc vapor, maintaining by the condensation of said zinc vapor a molten bath of zinc metal in full communication by gravity with a second molten bath of zinc metal from which reduction gases are excluded, volatilizing the zinc in said second bath, and burning the resulting zinc vapor to zinc oxide.

BONNER N. McCRAVEN.