

May 15, 1928.

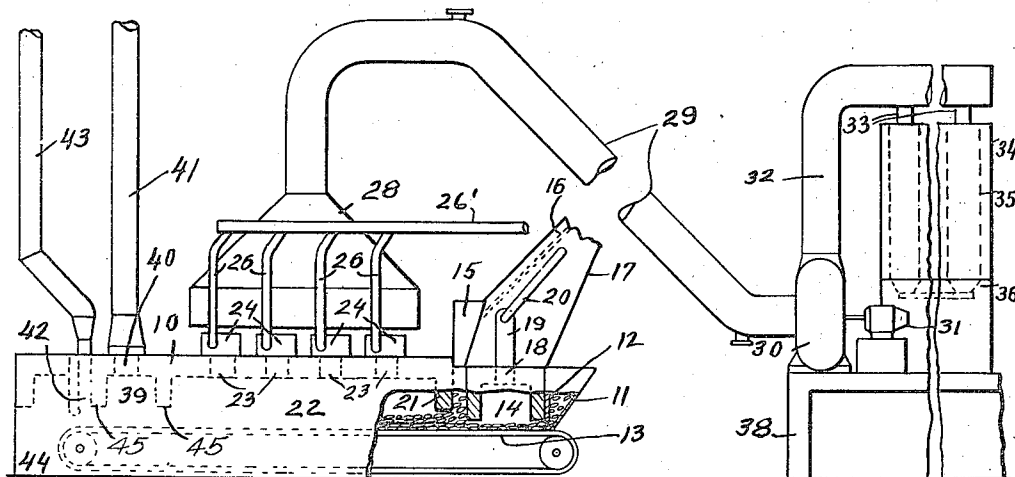
J. A. SINGMASTER ET AL

1,670,169

MANUFACTURE OF ZINC OXIDE

Filed March 27, 1926

Fig. 1.



Zinc oxide separated from
fume at temperature not less
than about 125°C

Fig. 2.

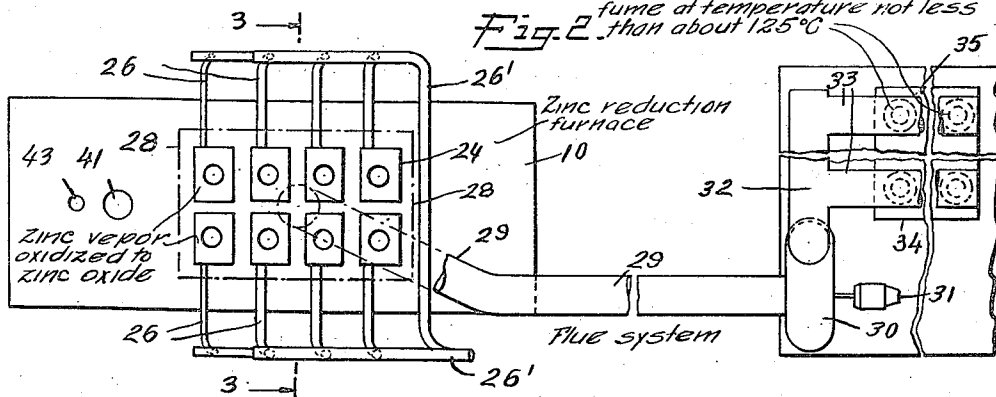
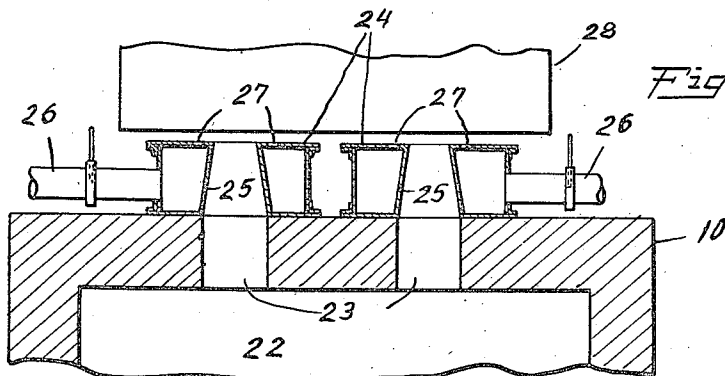


Fig. 3.



INVENTORS

JAMES A. SINGMASTER, FRANK G. BREYER & EARL H. BUNCE

BY Bonnie, Davis, Marvin, & Edmonds
ATTORNEYS

UNITED STATES PATENT OFFICE.

JAMES A. SINGMASTER, OF BRONXVILLE, NEW YORK, AND FRANK G. BREYER AND
EARL H. BUNCE, OF PALMERTON, PENNSYLVANIA, ASSIGNORS TO THE NEW
JERSEY ZINC COMPANY, OF NEW YORK, N. Y., A CORPORATION OF NEW JERSEY.

MANUFACTURE OF ZINC OXIDE.

Application filed March 27, 1926. Serial No. 97,352.

This invention relates to the manufacture of zinc oxide and has for its object the provision of certain improvements in zinc oxide manufacture. More particularly, the invention aims to provide an improved process of making rubber reinforcing zinc oxide. Other objects of the invention will be brought out in the course of the following description.

United States Patent No. 1,522,097 (Breyer-Gaskill and Singmaster) dated January 6, 1925, describes an improved method of manufacturing zinc oxide in which a blast of relatively cool oxidizing gas is directed against an issuing stream of metallic zinc vapor. We have discovered that the physical properties of the zinc oxide product produced by the method of this patent depend to some extent upon the source of the issuing stream of zinc vapor, that is to say, the method used in producing the zinc vapor. When the zinc vapor is produced by the volatilization of high grade metallic zinc, the zinc oxide product resulting from the process of this patent is free, or substantially free, from impurities, such as chlorides, sulfates, etc. We have discovered, however, that when the zinc vapor is obtained from a charge of mixed zinciferous and carbonaceous materials, the zinc oxide product produced by the method of this patent almost invariably contains impurities which, although present in very minute quantities, exercise a pronounced deleterious effect on the zinc oxide when incorporated in rubber. As a result of our researches and investigations, we believe that minute amounts of sulfur-oxygen compounds, and the like, condense on the surface of the zinc oxide particles when the zinc vapor is obtained from a charge of mixed zinciferous and carbonaceous materials, and that these compounds are responsible, in large measure, for the aforementioned deleterious effects.

The present invention is based upon our discovery that the condensation of sulfur-oxygen compounds upon the surface of zinc oxide particles is very substantially inhibited when the separation of the zinc oxide particles from the gases in which these particles are suspended is carried out at temperatures in excess of about 125° C. Thus, we have

found that when the gases carrying the zinc oxide particles in suspension are passed through an appropriate zinc oxide collecting apparatus maintained at a temperature not less than 125° C., the sulfur-oxygen compounds (for the most part sulfur dioxide and sulfur trioxide) pass along with the exhaust gases and substantially little, if any, objectionable condensation of these compounds takes place on the surface of the zinc oxide particles. The present invention, in its broad aspect, thus contemplates the production of zinc oxide from a charge of mixed zinciferous and carbonaceous materials, and the separation of the zinc oxide particles from the gases in which these particles are suspended while maintained at a temperature not less than about 125° C.

As applied to the production of zinc oxide of optimum properties for rubber-reinforcing purposes, the present invention contemplates maintaining substantially complete non-oxidizing conditions above a charge of mixed zinciferous and carbonaceous materials supported on a perforated grate and through which a combustion supporting draft is passed. The resulting gaseous products containing metallic zinc vapor are conveyed to an oxidizing environment of such characteristics that the zinc oxide particles are substantially instantaneously cooled to a temperature not higher than about 700° C., and preferably lower. At the same time, in accordance with our present invention, this sudden chilling or cooling of the zinc oxide particles is so regulated and controlled that the gases in which the zinc oxide particles are suspended are not cooled below a temperature of 125° C. until after passage through the zinc oxide collecting apparatus. Thus, the gases and zinc oxide particles suspended therein are maintained at a temperature below 700° C., but not less than 125° C., and are conveyed to an appropriate zinc oxide collecting apparatus where the exhaust gases are removed from the zinc oxide product at a temperature not less than about 125° C.

In the preferred practice of our present invention the charge of mixed zinciferous and carbonaceous materials is briquetted and supported upon a travelling or moving

perforated grate, through which a combustion supporting draft is maintained. This practice is more fully described in our United States Patent No. 1,322,143. The character and composition of the briquetted charge and the manipulation of the furnace are so controlled and regulated that a substantially complete non-oxidizing atmosphere is maintained above the charge. The resulting gaseous products contain metallic zinc vapor and are passed through appropriate gas outlets in the roof of the furnace and are conveyed into an oxidizing environment where such products are subjected to the action of a blast of relatively cool oxidizing gas whereby the flame temperature is lowered and the resulting particles of zinc oxide are substantially instantaneously cooled to a temperature not higher than about 700° C., and preferably not higher than about 500° C. The gases and zinc oxide particles suspended therein are then conveyed from this oxidizing environment, while maintained at a temperature not less than about 125° C., to the zinc oxide collecting apparatus, preferably asbestos fibre bags, also maintained at a temperature not less than about 125° C.

The novel features of the invention can be best explained by reference to the accompanying drawing in which:

Fig. 1 is a sectional side elevation of a traveling grate furnace and zinc oxide collecting apparatus;

Fig. 2 is a top plan view of the furnace and apparatus shown in Fig. 1; and

Fig. 3 is a detail section on the line 3—3 of Fig. 2.

The traveling grate furnace 10 is provided at one end thereof with a fuel-briquet feed hopper 11. An adjustable fuel-briquet leveler 12 depends from the furnace roof to within a predetermined height from the perforated traveling grate 13. The fuel-briquet ignition chamber 14 has a fuel-ore briquet feed hopper 15 immediately adjoining it. A grizzly chute 16 surrounded by a sheet steel casing 17 inclines downwardly into the fuel-ore briquet hopper.

The fuel-briquet ignition chamber 14 is provided with an outlet 18 through the furnace roof, in conjunction with which is attached an offtake pipe 19 connected with distributing pipes 20 leading under the grizzly chute 16. An adjustable fuel-ore briquet leveler 21, adjacent the fuel-ore briquet hopper 15, depends from the furnace roof to within a predetermined height from the perforated traveling grate 13. The main working chamber 22 extends along the greater part of the furnace length. It is provided with outlets 23 through the furnace roof. A gas compartment or wind-box 24, having a conical nozzle 25, rests axially over each outlet 23. These wind-

boxes are equipped with air supply pipes 26 as well as with circumferential slots 27 around the upper periphery of the nozzle 25. The pipes 26 are in turn connected with the main air blast supply pipes 28.

A hood 28 is appropriately placed over the wind-boxes 24. Suitable piping 29 connects the hood with the suction-blast fan 30 operatively connected with a motor 31. A feed pipe 32 extends from the fan to the distributing pipes 33. These pipes in turn lead into a zinc oxide collecting chamber or bag room 34, the walls and roof of which are insulated with asbestos board, or the like. Filter-bags 35, preferably of asbestos fibre, are hung within the chamber 34 and are connected at their upper ends to the distributing pipes 33, and at their lower ends to collecting hoppers 36. The chamber or bag room 34 is mounted on an appropriate foundation 38. Suitable outlets (not shown) are provided in the top of the bag room to permit the escape of exhaust gases from the filter-bags to the outside atmosphere.

Returning now to the furnace structure, a relatively short chamber 39 is provided, by depending bulkheads or partitions 45, beyond the main working chamber 22. The chamber 39 has an outlet 40 connected with a down-leg pipe 41 leading to a bag room or other suitable collecting apparatus (not shown).

A still shorter chamber 42 is provided near the discharge end of the traveling grate 13, and adjacent to the chamber 39. A stack 43 communicates with this chamber 42 and leads to the outside atmosphere.

A spent residue or ash hopper 44 is provided at the extreme end of the furnace to catch exhausted charge clinkers remaining after the furnacing operation has been completed. It is so constructed that the ash may be removed from time to time as desired.

The operation of this apparatus in accordance with the process of our present invention is as follows: Carbonaceous fuel briquets, preferably coal, are charged onto the perforated traveling grate 13 by way of the fuel-briquet hopper 11 at one end of the furnace 10. The grate is made to travel (by machinery not shown, but more fully described in Patent No. 1,322,143, hereinbefore referred to) with its burden of fuel briquets toward the other end of the furnace as a continuous but regulated draft of combustion supporting gas (preferably air) is passed up through the perforated grate into the charge. These fuel briquets are thereupon spread over the grate to form a layer of predetermined depth by means of the adjustable levelers 12. They are quickly heated to a relatively high temperature and ignited as they progress through the ignition chamber 14.

In the meantime fuel-ore briquets are

passed down the grizzly chute 16 into the fuel-ore briquet hopper 15 which deposits the briquets upon the ignited layer of fuel briquets issuing from the ignition chamber

14. These fuel-ore briquets are fed from the briquetting press (not shown) directly to the furnace by way of the heating chamber 17 surrounding the grizzly chute 16. Regulated amounts of hot gaseous products of combustion are conducted from the ignition chamber 14 by way of the off-take pipe 19 and the distributing pipes 20 to a suitable outlet beneath the grizzly chute, where they escape and circulate in and among the descending fuel-ore briquets.

The fuel-ore briquets from the hopper 15 are spread to form a layer of predetermined thickness upon the ignited bed of fuel briquets by means of the adjustable leveler 21. Reduction of the compounds of zinc in the fuel-ore briquets to metallic zinc begins to take place almost immediately. The metallic zinc is likewise immediately volatilized to zinc vapors. The greater part of the reduction and volatilization of the zinc takes place as the traveling grate moves through the main working chamber 22 of the furnace. The draft of combustion supporting gas passed up through the perforated grate is so regulated that a substantially complete non-oxidizing atmosphere is at all times maintained above the charge.

As the combined charge of fuel and fuel-ore briquets passes through the main working chamber 22 of the furnace, the gaseous products of the operation, including the metallic zinc vapor, flow, to the outlets 23 and upwardly through the nozzles 25 of the wind boxes 24. A blast of relatively cool oxidizing gas is directed from the circumferential slots 27 against the stream of gaseous products issuing from the nozzles 25. This oxidizing gas, preferably air, is fed under pressure to the wind-boxes by means of pipes 26 and 26'.

The metallic zinc vapor issuing from the nozzles 25 immediately oxidizes or burns to zinc oxide, and as a consequence of the action of the blast of relatively cool oxidizing gas, the flame temperature is lowered and the resulting particles of zinc oxide are substantially instantaneously cooled to a temperature not more than 700° C., and preferably not higher than about 500° C. This lowering of the flame temperature and substantially instantaneous cooling prevents the initially very fine zinc oxide particles from fusing, subliming or growing into agglomerates or particles of larger size.

The temperature to which the resulting particles of zinc oxide should be substantially instantaneously cooled depends, to some extent at least, upon the concentration of the zinc vapor in the gaseous products issuing from the outlets 23 of the furnace.

Under the operating conditions hereinbefore specifically described this temperature may be as high as about 700° C., but preferably is lower than about 500° C.

The finely divided zinc oxide particles, suspended in the resulting gases of the oxidizing operation, are drawn into the hood 28 and piping system 29 by the suction fan 30 and conveyed to the bagroom 34. The bagroom is maintained at a temperature not below about 125° C. The zinc oxide particles are deposited in the bags 34, while the gases in which these particles were suspended filter through the bags and exhaust from the bag-room through appropriate outlets. The zinc oxide is collected in the hoppers 36, to facilitate which the bags 35 may be mechanically or otherwise shaken, and the product collecting in the hoppers is removed in any appropriate manner. If the zinc oxide product is held in the hoppers 36 for any length of time, it is desirable to maintain these hoppers and the oxide therein at a temperature not below about 125° C.

The separation of the zinc oxide particles from the gases in which these particles are suspended while maintained at a temperature not below about 125° C., effectively minimizes the condensation of sulfur-oxygen compounds on the surface of the zinc oxide particles. The condensation of such sulfur-oxygen compounds on the surface of the zinc oxide particles is not entirely inhibited by maintaining the temperature of the collecting system or bag-room at a minimum of 125° C. At this minimum temperature there is still some condensation of the sulfur-oxygen compounds, but the amount of such condensation in the time the zinc oxide is ordinarily held in the collecting system is not sufficient to deposit a harmful amount of the sulfur-oxygen compounds on the zinc oxide particles. When the zinc oxide is caught in settling chambers and held exposed to the sulfur-oxygen gases coming from the furnace for a longer time than in the ordinary bag-room practice, temperatures higher than 125° C. may be necessary to inhibit objectionable condensation of the sulfur-oxygen compounds on the particles of zinc oxide.

A lower grade of zinc oxide is produced in the traveling grate furnace as the fuel and fuel-ore briquets become worked off. Thus, when the charge has been carried by the traveling grate beyond the main working chamber 22, the carbonaceous content of the charge has become so far depleted that it is difficult to maintain a completely non-oxidizing atmosphere above the charge, and the chamber 39 is provided for the working off of the charge during this final stage of the operation. The gaseous products formed in the chamber 39 pass through the outlet 40 into the downleg 41 and thence to an ap-

propriate collecting apparatus where the zinc oxide is separated from the gases in which it is suspended.

The worked-off charge or residue is deposited from the discharge end of the traveling grate into the receiving hopper 44 and the traveling grate returns by way of its undercourse to the charging end of the furnace where fresh fuel briquets and fuel-ore briquets are being continuously deposited on the grate. The furnace is thus mechanically operated in a continuous manner.

While we have herein described the practice of our invention on a traveling grate furnace with a briquetted charge, it is to be understood that the invention may be carried out in various other types of furnaces, and with or without briquetting of the charge either in whole or in part.

We claim:

1. The improvement in the process of making zinc oxide where a charge of zinciferous and carbonaceous materials is supported on a perforated grate and a combustion supporting draft maintained there-through, which comprises maintaining substantially non-oxidizing conditions above the charge, conducting the resulting gaseous products containing metallic zinc vapor to an oxidizing environment where such products are subjected to the action of a blast of relatively cool oxidizing gas, and conveying the resulting gases carrying the zinc oxide particles in suspension while maintained at a temperature not less than about 125° C. to an appropriate zinc oxide collecting means where the exhaust gases are separated from the zinc oxide particles at a temperature not less than about 125° C.

2. The improvement in the process of making zinc oxide where a charge of zinciferous and carbonaceous materials is supported on a perforated grate and a combustion supporting draft maintained there-through, which comprises maintaining substantially non-oxidizing conditions above the charge and conveying the resulting gaseous products containing metallic zinc vapor to an oxidizing environment where such products are subjected to the action of a blast of relatively cool oxidizing gas and the resulting particles of zinc oxide are cooled substantially instantaneously to a temperature not higher than about 700° C., and separating the zinc oxide particles from the gases in which these particles are suspended while maintained at a temperature not less than about 125° C.

3. The improvement in the process of making zinc oxide where a charge of zinciferous and carbonaceous materials is heated to a temperature sufficiently high to reduce the compounds of zinc and to volatilize the metallic zinc, which comprises maintaining substantially non-oxidizing conditions above

the charge, conducting the resulting gaseous products containing metallic zinc vapor to an oxidizing environment, and conveying the resulting gases carrying the zinc oxide particles in suspension while maintained at a temperature not less than about 125° C., to an appropriate zinc oxide collecting means where the exhaust gases are separated from the zinc oxide particles at a temperature not less than about 125° C.

4. The improvement in the process of making zinc oxide where a charge of zinciferous and carbonaceous materials is heated to a temperature sufficiently high to reduce the compounds of zinc and to volatilize the metallic zinc, which comprises maintaining substantially non-oxidizing conditions above the charge, conducting the resulting gaseous products containing metallic zinc vapor to an oxidizing environment, cooling the resulting particles of zinc oxide to a temperature not higher than about 700° C., and separating the zinc oxide particles from the gases in which these particles are suspended while maintained at a temperature not less than about 125° C.

5. The improvement in the process of making zinc oxide where a charge of zinciferous and carbonaceous materials is heated to a temperature sufficiently high to reduce the compounds of zinc and to volatilize the metallic zinc, which comprises maintaining substantially non-oxidizing conditions above the charge, conducting the resulting gaseous products containing metallic zinc vapor to an oxidizing environment where such products are subjected to the action of a blast of relatively cool oxidizing gas, and conveying the resulting gases carrying the zinc oxide particles in suspension while maintained at a temperature not less than about 125° C. to an appropriate zinc oxide collecting means where the exhaust gases are removed from the zinc oxide particles at a temperature not less than about 125° C.

6. The improvement in the process of making zinc oxide where a charge of zinciferous and carbonaceous materials is heated to a temperature sufficiently high to reduce the compounds of zinc and to volatilize the metallic zinc, which comprises maintaining substantially non-oxidizing conditions above the charge, conducting the resulting gaseous products containing metallic zinc vapor to an oxidizing environment where such products are subjected to the action of a blast of relatively cool oxidizing gas and the resulting particles of zinc oxide are cooled substantially instantaneously to a temperature not higher than about 700° C., and separating the zinc oxide particles from the gases in which these particles are suspended while maintained at a temperature not less than about 125° C.

7. The improvement in the process of

making zinc oxide from an appropriately heated charge of zinciferous and carbonaceous materials, which comprises conveying the gases in which the particles of zinc oxide are suspended while maintained at a temperature not less than about 125° C. to an appropriate zinc oxide collecting means where the exhaust gases are removed from the zinc oxide particles at a temperature not less than about 125° C.

8. The improvement in the process of making zinc oxide from an appropriately heated charge of zinciferous and carbonaceous materials, which comprises conducting the formation of the zinc oxide particles in an oxidizing environment where the particles of zinc oxide are cooled to a temperature not higher than about 700° C., substantially instantaneously after their formation, and separating the zinc oxide particles from the gases in which these particles are suspended while maintained at a temperature not less than about 125° C.

9. The improvement in the process of making zinc oxide where a charge of zinciferous and carbonaceous materials is heated to a temperature sufficiently high to reduce the compounds of zinc and to volatilize the metallic zinc, which comprises conducting the resulting gaseous products containing metallic zinc vapor to an oxidizing environment and there subjecting such products to a blast of relatively cool oxidizing gas whereby the flame temperature is lowered and the resulting particles of zinc oxide are cooled substantially instantaneously to a temperature not higher than about 700° C., and conveying the resulting gases carrying the zinc oxide particles in suspension while maintained at a temperature not less than about 125° C. to an appropriate zinc oxide collecting means and there separating the exhaust gases from the zinc oxide particles at a temperature not less than about 125° C.

10. The improvement in the process of making zinc oxide where a charge of mixed zinciferous and carbonaceous materials is supported on a perforated grate and a combustion supporting draft maintained therethrough, which comprises maintaining substantially non-oxidizing conditions above the charge, conducting the resulting gaseous products containing metallic zinc vapor to an oxidizing environment and there subjecting such products to a blast of relatively cool oxidizing gas whereby the flame temperature is lowered and the resulting particles of zinc oxide are cooled substantially instantaneously to a temperature not higher than about 700° C., and conveying the resulting gases carrying the zinc oxide particles in suspension while maintained at a temperature not less than about 125° C. to an appropriate zinc oxide collecting means and there separating the exhaust gases from

the zinc oxide particles at a temperature not less than about 125° C.

11. The improvement in the process of making zinc oxide where a charge in whole or in part briquetted of mixed zinciferous and carbonaceous materials is supported on a perforated grate and a combustion supporting draft maintained therethrough, which comprises maintaining substantially non-oxidizing conditions above the charge, conducting the resulting gaseous products containing metallic zinc vapor to an oxidizing environment and there subjecting such products to a blast of relatively cool oxidizing gas whereby the flame temperature is lowered and the resulting particles of zinc oxide are cooled substantially instantaneously to a temperature not higher than about 700° C., and conveying the resulting gases carrying the zinc oxide particles in suspension while maintained at a temperature not less than about 125° C. to an appropriate zinc oxide collecting means and there separating the exhaust gases from the zinc oxide particles at a temperature not less than about 125° C.

12. The improvement in the process of making zinc oxide where a charge of mixed zinciferous and carbonaceous materials is advanced through a combustion zone and a combustion supporting gas is simultaneously passed into the charge, which comprises maintaining substantially non-oxidizing conditions above the charge, conducting the resulting gaseous products containing metallic zinc vapor to an oxidizing environment where such products are subjected to the action of a blast of relatively cool oxidizing gas, and conveying the resulting gases carrying the zinc oxide particles in suspension while maintained at a temperature not less than about 125° C. to an appropriate zinc oxide collecting means and there separating the exhaust gases from the zinc oxide particles at a temperature not less than about 125° C.

13. The improvement in the process of making zinc oxide where a charge of mixed zinciferous and carbonaceous materials is advanced through a combustion zone and a combustion supporting gas is simultaneously passed into the charge, which comprises maintaining substantially non-oxidizing conditions above the charge, conveying the resulting gaseous products containing metallic zinc vapor to an oxidizing environment where such products are subjected to the action of a blast of relatively cool oxidizing gas and the resulting particles of zinc oxide are cooled substantially instantaneously to a temperature not higher than about 700° C., and separating the zinc oxide particles from the gases in which these particles are suspended while maintained at a temperature not less than about 125° C.

14. The improvement in the process of making zinc oxide where a charge in whole or in part briquetted of zinciferous and carbonaceous materials is advanced through a combustion zone and a combustion supporting gas is simultaneously passed into the charge, which comprises maintaining substantially non-oxidizing conditions above the charge, conducting the resulting gaseous products containing metallic zinc vapor to an oxidizing environment where such products are subjected to the action of a blast of relatively cool oxidizing gas, and conveying the resulting gases carrying the zinc oxide particles in suspension while maintained at a temperature not less than about 125° C. to an appropriate zinc oxide collecting means where the exhaust gases are separated from the zinc oxide particles at a temperature not less than about 125° C.

15. The improvement in the process of making zinc oxide where a charge in whole or in part briquetted of zinciferous and carbonaceous materials is advanced through a combustion zone and a combustion supporting gas is simultaneously passed into the charge, which comprises maintaining substantially non-oxidizing conditions above the charge, conveying the resulting gaseous products containing metallic zinc vapor to an oxidizing environment and there subjecting such products to the action of a blast of relatively cool oxidizing gas whereby the resulting particles of zinc oxide are cooled substantially instantaneously to a tempera-

ture not higher than about 700° C., and separating the zinc oxide particles from the gases in which these particles are suspended while maintained at a temperature not less than about 125° C.

16. The improvement in the method of making zinc oxide by the American process whereby the deposition of sulfur-oxygen compounds on the zinc oxide particles is substantially inhibited, which comprises conveying the zinc oxide laden fume while maintained at a temperature not less than about 125° C. to an appropriate zinc oxide collecting means and there separating the exhaust gases containing the sulfur-oxygen compounds from the zinc oxide particles at a temperature not less than about 125° C.

17. The improvement in the method of making zinc oxide by the American process whereby condensation of acid gases on the particles of zinc oxide is substantially inhibited, which comprises conveying the zinc oxide laden fume while maintained at a temperature not less than about 125° C. to an appropriate zinc oxide collecting means, and separating the zinc oxide particles from the gases in which these particles are suspended while maintained at a temperature not less than about 125° C.

In testimony whereof we affix our signatures.

JAMES A. SINGMASTER.
FRANK G. BREYER.
EARL H. BUNCE.