

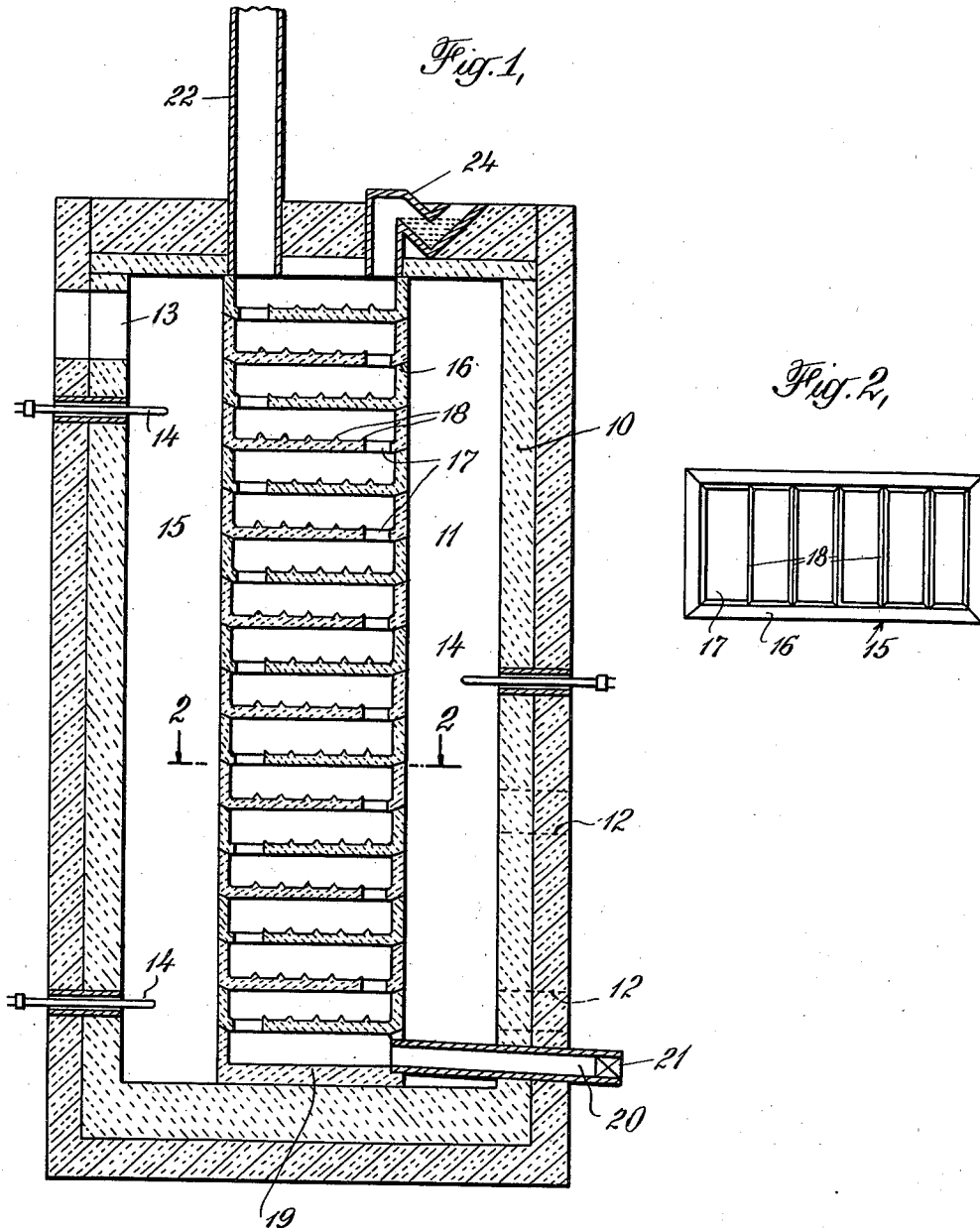
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1,994,356

APPARATUS FOR VOLATILIZING ZINC

Original Filed May 12, 1933 2 Sheets-Sheet 1



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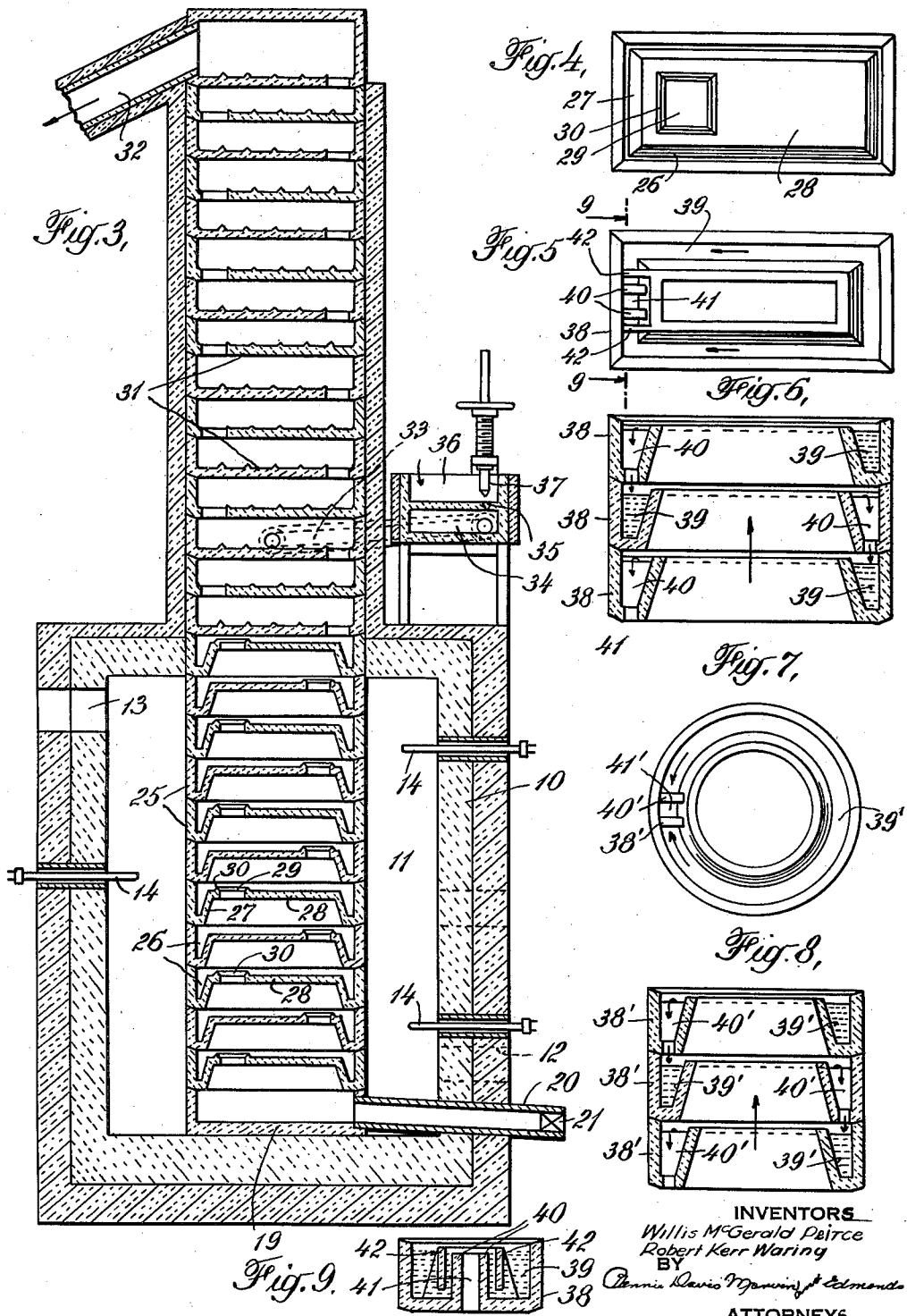
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APPARATUS FOR VOLATILIZING ZINC

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10 Claims. (Cl. 266—19)

This invention relates to the volatilizing of zinc metal and has for its object the provision of an apparatus for volatilizing zinc metal. The invention particularly aims to provide improvements in apparatus for use in volatilizing relatively large quantities of zinc metal in a substantially continuous operation. While the invention is applicable wherever zinc metal is to be volatilized, as for example in the manufacture of French process zinc oxide, it can be applied with particular advantage in redistilling impure zinc metal in the purification thereof by controlled distillation or by rectification treatment.

The present application is a division of our application Serial No. 670,648, filed May 12, 1933.

We have found that the rate at which zinc can be vaporized in a refractory container depends on the percentage of the area of the heated wall which has liquid zinc in contact with its inner surface. This contact maintains the inside wall temperature at approximately the boiling point of zinc and thereby increases the temperature gradient between the heating chamber temperature and the inside wall temperature. The increased temperature gradient increases the rate of heat transfer from the heating medium to the zinc to be vaporized.

In accordance with the present invention, the liquid zinc is dispersed in a plurality of baths so that a high percentage of the inner wall of the refractory container in which the zinc is volatilized is in contact with liquid zinc, and so that no large individual area of inner wall surface is free from contact with liquid zinc. Thus, in one of its practical aspects, the invention is characterized by heating a series of vertically spaced pools or baths of molten zinc metal to a sufficiently high temperature to volatilize the metal, and maintaining these pools of molten metal by charging molten metal into the top of the series and permitting overflow of molten metal from pool to pool. The vertically spaced pools of molten metal may be conveniently maintained in an upright refractory chamber internally packed to permit counter-current flow of descending molten metal and ascending vapor. The chamber may advantageously be packed with superposed and vertically spaced transverse pots or trays having appropriate openings permitting the passage of descending molten metal and ascending vapor from tray to tray.

The improved apparatus of the present invention permits of the advantageous use of relatively large zinc volatilizing retorts or zinc boilers compared with the usual practice of the prior art.

Zinc metal has heretofore been commonly volatilized in horizontal or slightly inclined cylindrical retorts, generally resembling the retorts of the Belgian spelter furnace, or in somewhat larger bottle-shaped retorts. In a small retort, such as the retorts or bottles commonly used in the prior art, the percentage of wall surface in contact with liquid zinc is limited by the necessity of maintaining a vapor space at the top of the retort. If the size of the retort is increased in order to provide a unit of large capacity, several difficulties are encountered:

1. Retorts or bottles of the form used in the prior art will, if made of large size and filled to the customary degree, contain deep baths of liquid zinc. The hydrostatic head of such deep baths of the heavy liquid zinc rapidly becomes too great for a refractory container safely to sustain.

2. A monolithic container becomes structurally impossible; and if a retort similar in form to those of the prior art is built up of separate bricks, the joints between the bricks will be, according to experience, unable to withstand the pressure of liquid zinc without leaking.

3. Carrying out vaporization in a deep bath of a heavy liquid, such as zinc, results in increased boiling point at the bottom and also results in violent ebullition, which is objectionable because it contaminates the vapor with a spray of impure metal.

The apparatus of the present invention obviates these difficulties and thus permits of the volatilization of zinc in units of large capacity and high output, as follows:

1. The distribution of the liquid (molten) zinc in a plurality of baths divides the hydrostatic head of the molten zinc so that it is not sufficiently great at any point to endanger the refractory container.

2. The apparatus of the present invention may be built up of individual units in such a way that no joint is exposed to the pressure of liquid zinc, thus obviating leakage of liquid zinc through the joints.

3. The distribution of the liquid zinc in a plurality of baths renders each bath relatively shallow, and thus makes negligible the increase in the boiling point of zinc at the bottom of the bath brought about by the hydrostatic head of the liquid zinc. The relative shallowness of the individual baths diminishes the violence of ebullition, and furthermore, the degree of ebullition present here is not disadvantageous in the apparatus of the present invention, in view of the fact that the molten metal flowing down from bath to bath

in counter-current with ascending zinc vapor rinses out of the zinc vapor the spray of impure molten metal with which ebullition contaminates the zinc vapor.

5 To attain liquid metal contact with the maximum percentage of heated wall area without unduly increasing the weight of liquid zinc to be supported by each unit of the retort or boiler, we may employ a unit in which a trough along each wall holds liquid zinc in contact with a large proportion of the total wall area. In such a vaporizing process as is contemplated by this invention, the impurities with higher boiling points and higher densities than zinc, such as lead and iron, will tend to concentrate and remain in the individual troughs. We have discovered two means of limiting this concentration by continuously removing these impurities from the troughs.

1. By providing a sufficient violence of ebullition, these impurities are kept in suspension and carried from one trough to the next and finally out of the last trough, together with a limited quantity of zinc.

2. By so designing the troughs and overflows that the flow of liquid metal from one unit to the next carries with it such impurities, which because of their greater density tend to settle out in the bottom of the trough.

In practicing the present invention as a part of a purification system to remove high boiling impurities, it is known that these impurities, for example lead, are carried into the vapor stream by mechanical entrainment of the mist due to boiling. The amounts so carried increase the impurity content of the vapor to an amount greater than would otherwise be present in the vapor by direct vaporization. By suitable arrangement of the retort units, to baffle and scrub the vapor, we may accomplish the removal of a substantial proportion of such entrained mist. By keeping the inner walls of the retort covered with liquid metal, we may also minimize the superheating of the vapor and consequent vaporization of such mist. If such vaporization does occur to some extent, thereby increasing the content of impurity vapor in the vapor stream, a means of reabsorbing this excess impurity vapor by contact with the liquid metal is provided.

We may also decrease the amount of entrained mist by so adjusting the proportions of the trough as to avoid violent ebullition. This may, if desirable, be carried to a point where the unit becomes a shallow tray, though a loss in vaporizing capacity results.

The invention may be embodied in various forms and types of apparatus, and in the accompanying drawings we have illustrated certain apparatus with which we have secured excellent results in the practice of the invention. In the drawings,

Fig. 1 is a sectional elevation of an apparatus for volatilizing zinc metal in accordance with the principles of the invention,

Fig. 2 is a plan of one of the transverse trays of the apparatus of Fig. 1,

Fig. 3 is a sectional elevation of an apparatus with a modified form of transverse tray in conjunction with a lead-eliminating rectifying column,

Fig. 4 is a plan of the transverse tray of the retort or volatilizing chamber of the apparatus of Fig. 3,

Figs. 5, 6, 7 and 8 illustrate still further modified forms of transverse trays adapted as inter-

nal packing for the volatilizing chamber or retort of the invention, and

Fig. 9 is a vertical section on the line 9-9 of Fig. 5.

Referring to Fig. 1 of the drawings, there is shown an upright furnace structure 10 having a heating chamber or laboratory 11 provided with burner openings 12 and an outlet 13 for the exhausted heating gases. Pyrometers 14 extend through the walls of the furnace structure into the heating chamber 11 for indicating and recording the temperature of the heating gases therein. It will, of course, be understood that any other appropriate means of heating may be used in lieu of the burners and hot gases of combustion indicated in the apparatus of Fig. 1.

A vertical volatilizing chamber or retort is built within the heating chamber 11. This retort is built up of superposed rectangular trays or pots 15 of silicon carbide or equivalent refractory material of good heat conductivity. The top and bottom edges of the trays are bevelled as at 16 so that they may be stacked one above the other to form the vertical retort. The joints between the trays are made zinc-vapor-tight by an appropriate cement, such as a cement of silicon carbide with a small amount of clay. The bottom of each tray has a transverse opening 17 approximate one end thereof. The upper surface of the bottom of each tray is provided with transverse ribs 18 parallel to the opening 17; the ribs being of progressively increasing height from the end nearest the opening to the end furthest from the opening. The trays 15 are stacked in staggered relationship with respect to the openings 17 so that the bottoms thereof form a column of superposed and vertically spaced baffles through which metallic vapor ascends and molten metal descends in tortuous or zig-zag paths. The ribs 18 serve to retain shallow pools of molten metal on the bottoms of the trays. The cascade effect of molten metal pouring over the ribs and falling from tray to tray facilitates intimate contact between ascending vapor and descending molten metal.

The bottom of the retort is formed by a tray 19 having a lateral opening communicating with a discharge pipe 20, the orifice or tap hole of which is closed by a plug 21. An outlet 22 is mounted on the top of the retort for withdrawing the zinc vapor evolved therein. The outlet 22 is operatively connected with any appropriate apparatus or device (not shown) for utilizing or treating the zinc vapor evolved from the retort. At its top, the retort is provided with a charging device 24 for the introduction of molten zinc metal under conditions preventing the escape therethrough of any vapor evolved in the retort.

In operating the apparatus of Fig. 1 in accordance with the present invention, molten zinc metal is introduced into the top of the vertical retort (15) through the charging device 24. As molten metal is added to the charging device, either continuously or at frequent intervals, the tray or charging well thereof overflows into the top transverse tray 15 of the vertical retort or boiler. Molten metal flows downwardly through the openings 17 from tray to tray, and pools of molten metal are retained on the bottoms of the trays by the ribs 18. The vertical retort is heated to a sufficiently high temperature by the hot gases surrounding it in the heating chamber 11 to actively boil the shallow pools of molten metal held on the trays. The heated walls of the retort are wetted with a plurality of baths of

molten zinc metal over a relatively large proportion of the wall area, and there are a plurality of baths of molten metal of relatively large surface area exposed to radiant heat, and in consequence of these two factors (particularly the first) very efficient volatilization of the molten metal takes place. Molten zinc metal is charged into the top of the retort in sufficient amount to maintain pools of molten metal on all of the trays of the retort, and this condition is established and maintained when a small amount of molten metal continuously overflows into the bottom tray 19 of the retort. In general, the rate of feed of molten metal to the retort approximates its boiling capacity. Where this rate of feed of molten metal is exceeded, the excess molten metal withdrawn from the bottom of the retort (if not too high in impurities) may be recharged into the top.

In Fig. 3 of the drawings the vertical retort or boiler is mounted within a heating chamber as in the apparatus of Fig. 1, but the retort is built up of trays or pots 25 having a V-shaped peripheral trough or groove 26 for holding molten metal. This groove is formed between the outer wall of the pot and an inner inclined wall 27 of the bottom 28 of the pot. It will be understood that all these parts of the tray or pot constitute an integrally molded article. The bottom 28 has an opening 29 approximate one end thereof, and the upper edge of the opening has a rib 30 for retaining a shallow layer of molten metal on the top surface of the bottom member 28.

A lead-eliminating reflux or rectifying column is mounted above the top of the vertical retort or boiler formed by the trays (25) to receive vapor therefrom. The lead-eliminating column comprises a vertical series of spaced transverse trays or baffles 31 generally resembling in configuration, structure and mounting the trays 15 shown in Figs. 1 and 2 of the drawings. An outlet 32 at the top of the column formed by the trays 31 convey zinc vapor (purified with respect to lead) to a condenser or to other appropriate apparatus for the recovery, utilization or subsequent treatment of the vapor.

Molten zinc metal to be volatilized in the retort formed by the trays (25) is introduced into one of the lower trays of the rectifying column through a pipe 33 communicating with a charging well 34. A continuous stream of molten zinc metal flows into the charging well 34 through an opening 35 in the bottom of a superposed charging well 36. The amount of molten metal flowing through the opening 35 is regulated by adjustment of the position of a cooperating valve stem 37. An appropriate level of molten metal is continuously maintained in the upper charging well 36.

Molten metal descends from tray to tray of the retort through the openings 29. Any impurities that may tend to collect in the troughs 26 are kept in suspension by violent ebullition prevailing therein and are in consequence swept out of the troughs and carried down through the retort by the descending stream of molten zinc. The impurities present less volatile than zinc, along with a limited quantity of zinc, escape from the retort through the discharge tube 20 and the tap-hole thereof.

The rectifying column formed by the trays (31) is thoroughly insulated throughout its length, except at the extreme top thereof. This uninsulated top of the rectifying column serves as a dephlegmator or condenser to supply molten

metal for refluxing back through the column. The retort or boiler formed by the trays (25) operates continuously to volatilize large quantities of impure zinc metal, and the resulting vapor is subjected to a rectification treatment in the rectifying column for removing lead therefrom as described in the copending application of Messrs. Holstein and Ginder, Serial No. 540,566, filed May 28, 1931. If desired the lead-free zinc vapor flowing from the top of the rectifying column may be conducted to a cadmium-eliminating rectifying column for removing cadmium therefrom as described in the copending application of Messrs. Ginder, Peirce and Waring, Serial No. 620,634, filed July 2, 1932.

In Figs. 5, 6 and 9, there is shown a still further modified form of tray or pot for the vertical retort or boiler of the invention. The tray 38 is rectangular in section and has an inner peripheral trough or groove 39 for holding molten metal to be volatilized. At one end of the tray the groove 39 has spaced curtains 42 and spaced dams 40; the molten metal flows under the curtains 42 and over the dams 40, and escapes from the tray through an opening 41 in the bottom of the groove and between the dams 40. In flowing under the curtains 42, the molten metal sweeps out of the grooves any impurities denser than molten zinc (such as lead or iron) that may tend to collect therein. With this form of tray the volatilized zinc metal passes freely up through the open center of the retort while the descending molten metal flows through the openings 41 from tray to tray and in zig-zag fashion through the grooves 39 thereof.

In Figs. 7 and 8 there is shown a type of tray resembling that shown in Figs. 5, 6 and 9 except that the tray is circular instead of rectangular and is not provided with curtains. Thus, the circular tray 38' has an inner circular peripheral groove 39' with spaced dams 40' and opening 41' in the bottom of the groove and between the dams.

In the actual operation of a five foot high retort built up of ten trays of the type shown in Figs. 3 and 4, 12" by 24" external dimensions and a wall thickness of 1 1/4", we have volatilized zinc metal at the rate of about 20,000 pounds per day of 24 hours. Such a retort has an outside heated surface of 30 square feet, so that the boiling rate per square foot of outside heated surface is about 666 pounds per day. This boiling rate per square foot of outside heated surface is from 3 to 6 times as great as in retorts of the type heretofore commonly used in volatilizing zinc metal.

The invention is of advantage wherever large quantities of zinc metal are to be volatilized, and is also adapted for the production of zinc vapor low in lead by controlled redistillation of zinc metal with relatively high lead content. For this application of the invention the internal packing of the retort or boiler should give intimate contact between the ascending vapor and descending molten metal, such as provided in the apparatus of Figs. 1 to 4 of the drawings so as to remove superheat and metallic mist from the zinc vapors. The retort is charged with impure zinc metal containing lead and/or other contaminants with boiling points exceeding that of zinc at a rate somewhat in excess of the boiling capacity of the retort. The retort may be so operated that the zinc vapor evolved from the retort contains an amount of lead that approaches that corresponding to the partial pressure of lead

in the alloy charged into the retort at the boiling point of the alloy. The relative purity of the zinc vapor is due to the following factors:

1. The scrubbing action of the shower of molten zinc metal descending through the ascending vapor tends to remove superheat and thus a portion of the lead vapor evaporated by this superheat is condensed or redissolved, and at the same time the scrubbing action removes metallic mist from the ascending vapor.

2. Superheat is substantially decreased not only by the scrubbing action, but also in consequence of the large bath surface exposed to heat radiation from the walls of the retort and by the great extent to which the walls of the retort are covered with or scrubbed by molten metal.

Furthermore, the metallic vapor leaving the retort or boiler at the top has on account of its intimate contact with the descending molten metal ample opportunity to approach closely to a state of equilibrium with the descending molten metal. In view of this circumstance the vapor leaving the retort may have a lead content approaching closely to the lead content of vapor in equilibrium with the molten metal charged into the retort. The purest zinc vapor evolved in the retort is the vapor that is evolved by the molten lead-zinc alloy just entering the retort. The vapor evolved from a boiling zinc-lead alloy contains a lower percentage of lead and a greater percentage of zinc than the molten zinc-lead alloy. In consequence, the molten zinc-lead alloy becomes gradually enriched in lead as it descends through the retort. Moreover, the vapor evolved from the molten metal also becomes richer in lead as the lead content of the molten metal is enriched during its descent through the retort. The metallic vapor evolved at the bottom of the retort is thus richer in lead than that evolved at the top. However, the excess lead content in the vapor evolved from the molten metal near the bottom of the retort tends to be removed from this vapor by the scrubbing action of the descending molten metal so that the lead content of the vapor as it leaves the retort is nearly in equilibrium with the entering molten metal.

When operated in the foregoing manner, the vertical retort of the invention is capable of producing large amounts of relatively pure zinc vapor from impure zinc by distillation without refluxing or rectification, scrubbing by the descending molten metal charged into the retort being relied upon to secure relatively pure zinc vapor. In many cases this zinc vapor will be sufficiently pure for direct utilization thereof and may be conveyed to any appliance for utilizing it, such as a canister for producing zinc dust, a combustion chamber for the production of zinc oxide, etc. Where a further removal of lead from the vapor evolved from the retort is necessary, the apparatus of Fig. 3 of the drawings with its superposed lead rectifying column is employed.

Relatively greater ebullition takes place in the troughs 26 of the pots of Fig. 3 than does on the more shallow pools held on the trays 15 of Fig. 1. The trough type of pot 25 has a greater boiling capacity than does the tray 15. The open-center pots of Figs. 5 to 8 of the drawing may often be used with advantage where it is unnecessary or undesirable to operate the retort or boiler with particular attention to evolving vapor of a lead content in equilibrium with the molten metal charged into the retort.

We claim:

1. An apparatus for volatilizing zinc metal comprising an upright chamber having a plurality of integrally formed transverse superposed trays provided with ribs on their upper surfaces and adapted to hold pools of molten metal, each of said trays being provided with an opening to permit the downward flow of molten metal from tray to tray and the upward flow of vapor, and means for heating said chamber to a sufficiently high temperature to volatilize molten metal in said pools.

2. An apparatus for volatilizing zinc metal comprising a built-up retort having a multiplicity of vertically spaced integrally formed transverse trays each of which is provided with one or more ribs on its upper surface and adapted to hold a pool of molten metal, an opening being provided in each tray whereby molten metal flows downwardly through said retort from tray to tray and vapor flows upwardly through the retort, and means for heating the retort to a sufficiently high temperature to volatilize substantial quantities of molten metal in said pools.

3. An apparatus for volatilizing zinc metal comprising an upright chamber having a plurality of vertically spaced integrally formed transverse trays, the side walls of the trays forming the side walls of the chamber, each of said trays having a peripheral trough formed integrally therewith and adapted to hold a pool of molten metal and means permitting the overflow of molten metal in said trough to the next lower tray in the chamber, the said overflow means being located at a level below the top of the side walls of the tray, the arrangement of said trays being such as to permit an upward flow of vapor through the chamber, means near the top of the chamber for introducing molten metal, and means for withdrawing vapor evolved in the chamber.

4. An apparatus for volatilizing zinc metal comprising a retort built up of superposed integrally formed units each of which is adapted to hold a bath of molten zinc metal, said units being so constructed and assembled that joints between any two units are located above the bath of molten zinc metal in the lower of the two units and no joint in the built-up structure is exposed to the hydrostatic head of any bath of molten zinc metal therein.

5. An apparatus for volatilizing zinc metal comprising a retort built up of superposed integrally formed units each of which has a peripheral trough adapted to hold a pool of molten metal in contact with the heated wall of the retort, said units being so constructed and assembled that joints between any two units are located above the pool of molten metal in the lower of the two units and no joint in the built-up structure is exposed to the hydrostatic head of any pool of molten metal therein, means permitting the downward flow of molten metal from trough to trough and the upward flow of vapor, means near the top of the retort for introducing molten metal, means for withdrawing vapor evolved in the retort, and means for heating the retort to a sufficiently high temperature to volatilize molten metal in said pools.

6. An apparatus for volatilizing zinc metal which comprises a retort built up of a plurality of superposed integrally formed trays the sides of which form the wall of the retort, damming means on the upper surfaces of the trays adapted to hold pools of molten zinc, and means whereby

heat is applied to the wall of the retort to volatilize zinc therein.

7. An apparatus for volatilizing zinc metal which comprises a retort built up of a plurality of superposed integrally formed trays the sides of which form the wall of the retort and the bottoms of which form a series of baffles, whereby ascending zinc vapor and descending molten zinc are forced to pursue a tortuous path, and damming means on the upper surfaces of the trays adapted to hold pools of molten zinc.

8. An apparatus for volatilizing zinc metal which comprises a retort built up of a series of superposed integrally formed units the sides of which form walls of the retort, said units having troughs adjacent the sides and formed integrally therewith adapted to retain pools of molten zinc.

9. An apparatus for volatilizing zinc metal comprising a retort built up of superposed inte-

grally formed units each of which provides a container formed integrally with its respective unit and so constructed and arranged as to retain a bath of molten zinc metal contacting with a large proportion of the inner surface of the heated wall of the retort.

10. An apparatus for volatilizing zinc metal which comprises a retort built up of a plurality of superposed integrally formed units, the sides of which form walls of the retort, said units having troughs adjacent their sides adapted to contain molten zinc, spaced curtains located within said troughs under which surplus molten zinc must flow in order to leave the troughs, and overflow means disposed between said curtains whereby the troughs are kept substantially full of molten zinc.

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