

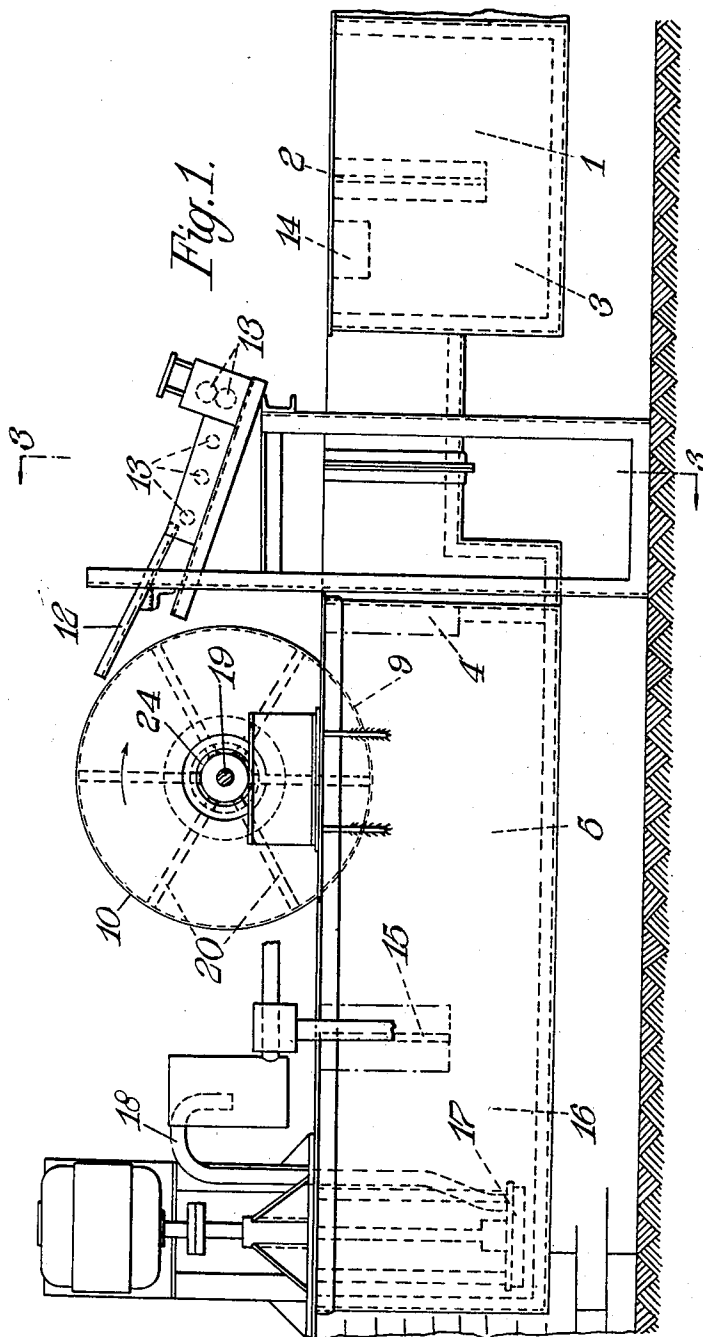
Feb. 14, 1956

G. K. WILLIAMS ET AL.
METHOD AND APPARATUS FOR SEPARATION OF
METALS, ALLOYS, OR COMPOUNDS FROM A
MOLTEN METALLIC SYSTEM

2,734,819

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3 Sheets-Sheet 1



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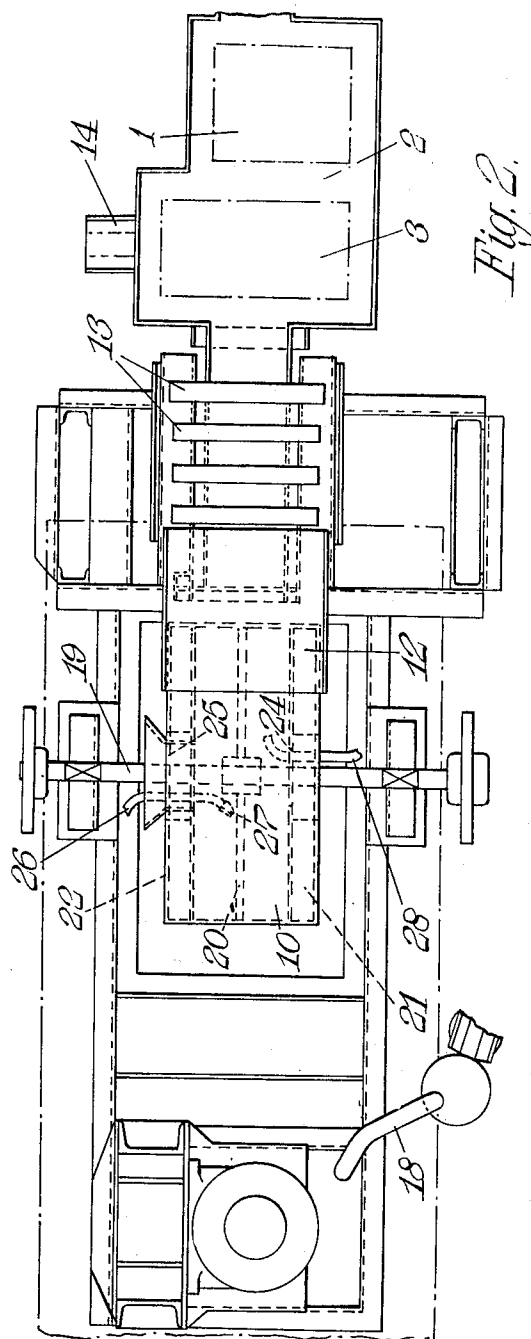


Fig. 2.

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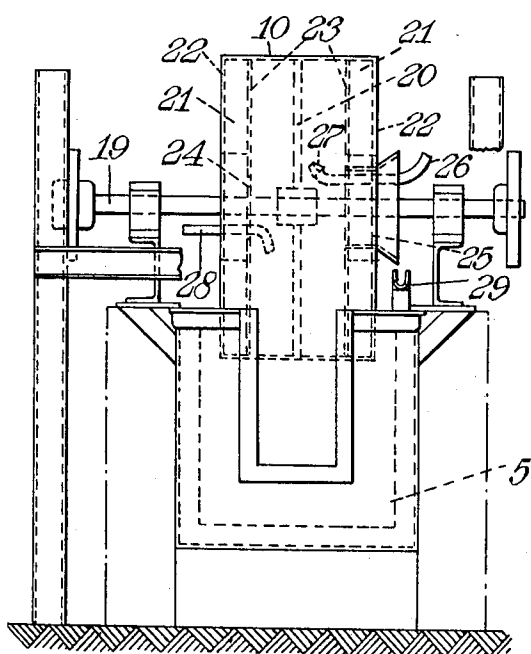


Fig. 3

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METHOD AND APPARATUS FOR SEPARATION OF METALS, ALLOYS, OR COMPOUNDS FROM A MOLTEN METALLIC SYSTEM

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Application May 5, 1951, Serial No. 224,698

Claims priority, application Great Britain May 8, 1950

22 Claims. (Cl. 75—63)

This invention relates to the separation of metals, alloys or compounds from a molten metallic system.

The invention consists in a method of separating metals, alloys or compounds from a molten metallic system comprising a solution of metals or of at least one metal and at least one compound in which this system is cooled by introducing a cooled moving surface into a bath of the molten metals whereby to cause separation of a metal, alloy or compound in solid form, while any metal, alloy or compound thus separating and adhering to the moving surface is continuously removed.

Such a method of separation may be used, for example, in the desilverisation of lead and in the removal of copper and copper sulphides from lead and also in the method of condensing zinc vapour by means of molten lead and subsequently recovering the zinc therefrom.

In connection with the last mentioned use we have described in our British Patent No. 572,961, (see U. S. Patent 2,464,262), a method in which a body of lead-zinc solution is cooled in its lower portion to a temperature below 418° (the solidifying point of the zinc or monotectic point) to precipitate the zinc therefrom while the upper portion is maintained above 418° C., whereby the precipitated zinc rises into the upper portion with the resultant remelting of the zinc and the formation of a supernatant layer of molten zinc.

We have further pointed out in our British application No. 17,837/49 (see co-pending U. S. application Serial No. 80,916, filed March 11, 1949, now U. S. Patent No. 2,671,725, issued March 9, 1954) that some of the precipitated zinc crystals tend to adhere to the sides of the vessel in which they are precipitated.

By cooling a lead-zinc solution down to 318° C. which is the eutectic temperature the zinc content of the lead can be reduced to 0.58%. The difficulty then arises of collecting the zinc as crystals and remelting it to cast it as ingots.

An object of the present invention is to provide an improved method of separating zinc from a lead-zinc solution which will permit of reduction of the zinc content to between about 0.58% and 1.7% and of continuous removal of the zinc separated from the solution.

Another object is to provide an improved method of reducing the zinc content of a lead-zinc solution having a zinc content of greater than 1.7% to between 0.58% and 1.7% by a continuous process supplying the separated zinc in a molten form.

Another object is to reduce the zinc content of the lead returned to the lead splash condenser such as that described in our British Patent No. 572,961 (see U. S. Patent 2,464,262) whereby to reduce the amount of oxidation of the zinc vapour being condensed, and hence of the consequent dross formation.

Further, according to the invention, a lead-zinc solution containing more than 0.58% zinc in solution and as to the bulk at a temperature not below the tempera-

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ture at which the zinc crystallises out is locally cooled by means of a moving surface in contact with the lead-zinc solution and which is at a temperature at which the zinc separates out in solid form and adheres to the moving surface, and the adhering strip of zinc is removed from the moving surface.

Conveniently the zinc strip may be melted by feeding it into a saturated solution of zinc in lead in a second vessel at a temperature above the melting point of zinc (418° C.). The zinc would then melt and float to the top of the lead, whence it could be removed.

Heat would, of course, have to be supplied to the melting vessel to furnish sensible heat and latent heat of fusion for the lead and zinc. If therefore the problem is to cool a solution of zinc in lead, which is at a temperature considerably in excess of 418° C., to a temperature below 418° C. and separate some of the dissolved zinc as liquid metal, the overall process involves removal of heat. The two-vessel process so far described involves a cooling stage and a heating stage. To avoid having to supply heat from an external source, we may use some of the sensible heat of the original hot lead to melt the solid mixture of zinc and lead produced from the cooler. This is effected by allowing the hot solution of zinc in lead to flow through the melting vessel on its way to the cooler. It may then not be necessary to apply any external heat to the melting pot or it may be necessary to apply some external heat but in less amount than would be necessary were the melting pot not in the incoming lead circuit.

Thus it will be seen how in a continuous process a lead-zinc solution may be first cooled to a temperature not lower than 418° C. in a vessel and may then be passed on to a second vessel in which it is locally cooled by means of a moving surface which is at a temperature at which zinc separates out in solid form and adheres to the moving surface, and the adhering strip of zinc may be removed from the moving surface and melted by being fed into the first vessel.

If the solution fed into the first vessel contains more than 1.7% zinc then, depending on the temperature to which the solution is cooled, molten zinc may separate out and rise to the surface.

With the process operated continuously, zinc as made would be withdrawn from the top of the melting vessel, while lead containing more than 1.7% of zinc in solution, would be withdrawn from the bottom of the melting vessel and returned to the cooling vessel.

The invention further consists of a method of desilverising lead in which molten lead containing zinc in solution is passed into the top of a full vessel and is drawn off from the bottom of the vessel, the temperature at the top of the vessel being such that the zinc-silver alloy separates out in molten form and the lead drawn off is fed to a second vessel in which it is cooled by means of a moving surface, the temperature at the bottom of the second vessel being the lead-zinc eutectic temperature (318° C.).

The invention further consists of apparatus for the separation of metals, alloys or compounds from a molten metal system comprising a vessel for receiving the molten metal system, a rotatable drum adapted to dip into the molten metal system in the vessel, cooling arrangements for the circumferential surface of the drum, and an arrangement for removing a sheet of metal from the circumferential surface of the drum.

The invention further consists of apparatus for the separation of metals, alloys or compounds from a molten metal system comprising a first vessel for receiving the molten metal system, a second vessel, a flow passage from the first vessel to the second vessel, a rotatably mounted drum for dipping into the metal system in the

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second vessel, a cooling arrangement for the circumferential surface of the drum, an arrangement for transferring a sheet of metal from the circumferential surface of the drum to the first vessel, and a flow passage from the second vessel.

An embodiment of an apparatus for carrying the invention into effect is shown in the accompanying drawings.

Figure 1 is a side elevation,

Figure 2 is a plan, and

Figure 3 is a section on the line 3—3 of Figure 1 with the pinch rollers omitted.

The lead comes in by a conduit to a vessel 1 and passes beneath an underflow weir 2 and enters vessel 3. Passing underneath another baffle 4 it enters vessel 5

in which dips the drum 10. The lower surface 9 of this drum cools the portion of the lead with which it comes into contact and thereby causes zinc to separate and adhere to the surface of the drum, entraining some lead with it and forming a sheet of metal on the surface of the drum which rotates in the direction indicated by the arrow. Just after the top of the drum the sheet of metal is deflected on to a plate 12 and then passes through pinch rollers 13.

It is then directed into the molten lead in vessel 3, the temperature of which, in spite of the cooling effect of the solid lead and zinc thus introduced, is still above 418°C ., the lead-zinc monotectic point. The zinc in excess above the amount soluble in lead at the temperature in this bath, separates out as a supernatant layer of liquid zinc, containing some lead in solution, and this is tapped off from the well 14.

The lead from vessel 5 now containing less zinc in solution, passes beneath baffle 15 into vessel 16. Thence it is sent by pump 17 into pipe 18 by which it is returned, say, to the lead-splash container from which it had originally come.

The mild steel drum 10 rotates on a horizontal shaft 19, to which it is fixed by spokes 20. The plane vertical faces of the drum are provided with heat-insulating lagging 21 (Fig. 3) enclosed between the two mild steel plates 22 and 23. On one vertical face of the drum is an orifice 24 and on the other side a larger orifice 25. Through orifice 25 is introduced a water pipe 26 from the spray-nozzle 27 of which water is directed into the drum. A second water pipe 28 directs water down to the lower part of the drum. The water which overflows from the lower edge of the orifice 25 falls into a gutter 29 by which it is conducted away.

In a specific example the lead entering the melting vessel 3 might contain 1.3% zinc, while the lead leaving the melting vessel would contain 1.8% zinc; entering the cooler 5 at 1.8% zinc it might leave containing 0.8% zinc. Under such conditions the weight of zinc removed in the cooler would be twice the net amount of zinc removed from the lead in flowing through the whole circuit.

To desilverize lead, zinc is dissolved in the lead at a temperature of, say, 600°C . and it is then cooled. The solution first separates into two layers, the upper one consisting chiefly of zinc and silver, the lower of lead saturated with zinc but containing very little silver. Above a certain temperature which, owing to the presence of the silver, may be a few degrees different from the 418°C . monotectic temperature when only zinc and lead are present, the upper layer is liquid; at lower temperatures the zinc-silver alloy separates as a solid crust.

One method of desilverizing lead is that described in British Patent No. 267,104. There is used a tall kettle, through which the lead, containing zinc in solution, continuously flows in at the top and out at the bottom. In practice, the lead offtake, starting at the bottom, is taken up through the liquid and out at the top, not by a spout leaving the side of the vessel at the bottom, as shown in the drawing of that specification; this difference, how-

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ever, is merely a detail of design. The top of the kettle is at a temperature such that the zinc-silver alloy which separates is molten; the bottom of the kettle is at the zinc-lead eutectic temperature, about 318°C . As the lead flows down the kettle, it slowly cools; the solubility of zinc-silver alloy in lead decreases as the temperature falls. In the lower part of the kettle the zinc-silver alloy which is coming out of solution is solid, but as it rises through the hot solution higher up it melts, so that all the zinc-silver alloy which separates finally accumulates as a liquid layer on the lead at the top of the kettle. The lead which leaves the bottom of the kettle retains in solution only a very small amount of silver which is still soluble at the eutectic temperature in lead which is saturated with respect to zinc.

In this desilverisation process the lead at the top of the kettle must be at a sufficiently high temperature for the silver-zinc alloy which separates to be liquid, and at the bottom it must be at a low temperature, near the eutectic point, in order that the lead run off should contain the smallest possible quantity of silver. A tall kettle has to be used to permit the necessary cooling of the flowing lead to take place. Particularly in the lower temperature range, a close control of temperature is necessary to prevent the lead round the bottom of the off-take pipe from freezing, and a small temperature gradient is therefore necessary when the lower temperature limit is being approached. Rapid cooling would be permissible, however, in the upper temperature range.

In an alternative method of procedure according to the present invention the lead would first be passed into a pot containing lead at the bottom with a layer of molten zinc-silver alloy floating thereon. The temperature of this first pot would be about 600°C . The lead put into this pot would pass through the upper layer of zinc and would flow away saturated or nearly saturated with zinc. It would then pass to a second pot similar to that illustrated in British Patent No. 267,104, but of only about half the height. Dipping into the surface of the lead in this second pot would be a drum cooler. This would reduce the temperature of the lead, which enters at say 550°C . considerably, to a temperature which need not be specified but might be in the region of 450°C . There would be a tendency for a molten zinc-alloy to separate at this temperature but this, together with any adherent lead, would be frozen by contact with the cooled drum. The sheet of metal on the drum would be transferred back to the first pot.

In the second pot the lead, already cooled in bulk to, say, 450°C . would be cooled further as it descended, the cooling from the sides of this pot being so controlled that the temperature at the bottom is at the lead-zinc eutectic temperature of 318°C . During this cooling, more zinc-silver alloy would separate from the descending lead and float up to the surface, to be collected by the drum cooler.

Various modifications will be possible within the scope of the invention.

We claim:

1. In the method of separating a component of the group consisting of metals or alloys of metals from a liquid molten metallic body thereof, the improvement which comprises maintaining a body of molten metallic liquid like that to be treated in a cooling zone, said body being separated generally into two superposed layers, the upper layer being relatively shallow and consisting chiefly of the component to be recovered and the lower layer being relatively deep and consisting chiefly of the remainder of the molten metallic liquid, passing the body of molten metallic liquid to be treated into the body of molten metallic liquid in the cooling zone, cooling the molten metallic body to a temperature not lower than that at which the component separates out in solid form, passing the body of molten metallic liquid thus partially cooled into a second cooling zone, moving a cooling

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surface through the molten metallic body in the second cooling zone at a temperature at which the desired component separates out and adheres thereto in solid form, withdrawing from the second cooling zone liquid from the molten metallic body thus denuded of said component, removing said component from the moving surface and passing it into the liquid molten metallic body in the first cooling zone for the enrichment of the molten metallic body therein to the point where said component separates out and rises to the top of said body to form a supernatant overlying liquid layer of said component and an underlying liquid layer of the remainder of the molten metallic body still containing some of said component, continuing to pass liquid from the underlying layer into the second cooling zone for treatment with said moving cooling surface, and removing molten component from the supernatant liquid layer in the first cooling zone.

2. In the method of recovering zinc dissolved in a body of molten lead used cyclically in a zinc vapor condensing zone as a condensing medium for zinc vapor obtained in a zinc smelting operation, the resulting molten zinc-rich lead being treated in a zinc separating zone to recover zinc, the improvement which comprises maintaining a body of molten lead-zinc in a cooling zone, said body being separated generally into two superposed layers, the upper layer being relatively shallow and consisting chiefly of zinc with a little lead and the lower layer being relatively deep and consisting chiefly of lead and some zinc, passing the body of molten lead-zinc to be treated from the condensing zone into the body of molten lead-zinc in the cooling zone, cooling the body of molten lead-zinc to a temperature not lower than that at which the zinc separates out in solid form, passing the body of molten lead-zinc thus partially cooled into a second cooling zone, moving a cooling surface through the body of molten lead-zinc in the second cooling zone at a temperature at which zinc and some lead separate out and adhere thereto in solid form, withdrawing from the second cooling zone resulting molten lead lean in zinc for re-use in the condensing zone, removing the zinc-lead in solid form from the moving surface and passing it into the body of molten lead-zinc in the first cooling zone for melting and the enrichment of the molten lead-zinc therein to the point where zinc separates out and rises to the top of said body to form a supernatant overlying liquid layer of zinc and an underlying liquid layer of the remainder of the zinc-containing lead, continuing to pass molten lead-zinc from the underlying layer into the second cooling zone for treatment with said moving cooling surface, and removing molten zinc from the supernatant liquid layer in the first cooling zone.

3. Method according to claim 2, in which the cooling surface is moved through the top portion of the body of molten lead-zinc in the second cooling zone.

4. Method according to claim 2, in which the resulting molten lead lean in zinc is removed from the bottom portion of the second cooling zone.

5. Method according to claim 2, in which the cooling surface is moved through the top portion of the body of molten lead-zinc in the second cooling zone, and the resulting molten lead lean in zinc is removed from the bottom portion of the second cooling zone.

6. In the method of recovering zinc dissolved in a body of molten lead used cyclically in a zinc vapor condensing zone as a condensing medium for zinc vapor obtained in a zinc smelting operation, the resulting molten zinc-rich lead being treated in a zinc separating zone to recover zinc, the improvement which comprises maintaining a body of molten lead-zinc in a cooling zone, said body being separated generally into two superposed layers, the upper layer being relatively shallow and consisting chiefly of zinc with a little lead and the lower layer being relatively deep and consisting chiefly of lead and some zinc, passing the body of molten lead-zinc to be

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treated from the condensing zone into the body of molten lead-zinc in the cooling zone, cooling the body of molten lead-zinc to a temperature not lower than that at which the zinc separates out in solid form, passing the body of molten lead-zinc thus partially cooled into a second cooling zone, moving a cooling surface through the body of molten lead-zinc in the second cooling zone at a temperature at which zinc and a little lead separate out and adhere thereto in solid form, withdrawing from the second cooling zone resulting molten lead lean in zinc for re-use in the condensing zone, removing the zinc-lead in solid form from the moving surface and passing it into a body of molten lead saturated with zinc at a temperature maintained above the melting point of the zinc, melting the solid zinc therein and thereby enriching the molten lead-zinc to the point where zinc separates out and rises to the top of said body to form a supernatant overlying liquid layer of zinc and an underlying liquid layer of lead saturated with zinc, and removing molten zinc from the supernatant liquid layer.

7. In the method of desilverizing a body of molten lead containing zinc in solution, the improvement which comprises maintaining an upright elongated body of molten lead-zinc-silver in a cooling zone, said body of molten metals being separated generally into two superposed layers, the upper layer being relatively shallow and consisting chiefly of zinc and silver with a little lead and the lower layer being relatively deep and consisting chiefly of lead with some zinc and a little silver, passing the body of molten lead-zinc to be desilverized into the body of molten metals in the cooling zone, passing molten lead containing some zinc and a little silver from the lower layer into a second cooling zone, permitting molten zinc-silver with a little lead to separate out as a top layer and the bulk of the molten lead to form as a bottom layer in the second cooling zone, moving a cooling surface through the body of molten metals in the second cooling zone at a temperature at which zinc-silver and a little lead separate out and adhere thereto in solid form, withdrawing from the bottom layer in the second cooling zone resulting molten lead lean in zinc and silver for re-use in recovering more silver, removing the zinc-silver in solid form from the moving surface and passing it into the body of molten lead-zinc-silver in the first cooling zone for melting and the enrichment of the body of molten lead-zinc-silver therein to the point where zinc-silver separates out, rises and merges with said upper layer of zinc-silver in that cooling zone, and removing molten zinc-silver from the upper layer in the first cooling zone.

8. Method according to claim 7, in which the body of molten lead-zinc to be desilvered is passed downwardly through said upper layer of molten zinc-silver in the first cooling zone to facilitate the separation and formation of more zinc-silver in that layer.

9. Method according to claim 7, in which the cooling surface is moved through the top layer of zinc-silver in the second cooling zone.

10. Method according to claim 7, in which the resulting molten lead lean in zinc and silver for re-use in recovering more silver is removed from the bottom of the bottom layer in the second cooling zone.

11. Method according to claim 7, in which the cooling surface is moved through the top layer of zinc-silver in the second cooling zone, and the resulting molten lead lean in zinc and silver for re-use in recovering more silver is removed from the bottom of the bottom layer in the second cooling zone.

12. In the method of recovering zinc dissolved in a body of molten lead used cyclically in a zinc vapor condensing zone as a condensing medium for zinc vapor obtained in a zinc smelting operation, the resulting molten zinc-rich lead being treated in a zinc separating zone to recover zinc, the improvement which comprises maintaining a body of molten lead-zinc in a cooling zone, said

body being separated generally into two superposed layers, the upper layer being relatively shallow and consisting chiefly of zinc with a little lead and the lower layer being relatively deep and consisting chiefly of lead and some zinc, passing the body of molten lead-zinc to be treated from the condensing zone into the body of molten lead-zinc in the cooling zone, cooling the body of molten lead-zinc to a temperature not lower than that at which the zinc separates out in solid form, passing the body of molten lead-zinc thus partially cooled into a second cooling zone, moving a cooling surface through the body of molten lead-zinc in the second cooling zone at a temperature at which zinc and some lead separate out and adhere thereto in solid form, withdrawing from the second cooling zone resulting molten lead lean in zinc for re-use in the condensing zone, removing the zinc-lead from the moving surface and passing it into the body of molten lead-zinc in the first cooling zone for the enrichment of the molten lead-zinc therein to the point where zinc separates out and rises to the top of said body to form a supernatant overlying liquid layer of zinc and an underlying liquid layer of the remainder of the zinc-containing lead, continuing to pass molten lead-zinc from the underlying layer into the second cooling zone for treatment with said moving cooling surface, and removing molten zinc from the supernatant liquid layer in the first cooling zone.

13. In the method of desilverizing a body of molten lead containing zinc in solution, the improvement which comprises maintaining an upright elongated body of molten lead-zinc-silver in a cooling zone, said body of molten metals being separated generally into two superposed layers, the upper layer being relatively shallow and consisting chiefly of zinc and silver with a little lead and the lower layer being relatively deep and consisting chiefly of lead with some zinc and a little silver, passing the body of molten lead-zinc to be desilverized into the body of molten metals in the cooling zone, passing molten lead containing some zinc and a little silver from the lower layer into a second cooling zone, permitting molten zinc-silver with a little lead to separate out as a top layer and the bulk of the molten lead to form as a bottom layer in the second cooling zone, moving a cooling surface through the body of molten metals in the second cooling zone at a temperature at which zinc-silver and a little lead separate out and adhere thereto in solid form, withdrawing from the bottom layer in the second cooling zone resulting molten lead lean in zinc and silver for re-use in recovering more silver, removing the zinc-silver from the moving surface and passing it into the body of molten lead-zinc-silver in the first cooling zone for the enrichment of the body of molten lead-zinc-silver therein to the point where zinc-silver separates out, rises and merges with said upper layer of zinc-silver in that cooling zone, and removing molten zinc-silver from the upper layer in the first cooling zone.

14. In apparatus for separating a component of the group consisting of metals, alloys or compounds of metals from a molten metallic liquid body thereof, the improvement comprising a cooling chamber for receiving the molten body of metallic liquid to be treated and for separating the same generally into two superposed layers, the upper layer being relatively shallow and consisting chiefly of the component to be recovered and the lower layer being relatively deep and consisting chiefly of the remainder of the molten metallic liquid containing some of the component, a second cooling chamber for receiving molten metallic liquid from the first cooling chamber and for separating the same generally into two superposed layers, the upper layer being relatively shallow and consisting chiefly of the component to be recovered and the lower layer being relatively deep and consisting chiefly of the remainder of the molten metallic liquid containing the unseparated portion of the component, a conduit con-

necting the lower portion of the first cooling chamber with the lower portion of the second cooling chamber for the passage therethrough of molten metallic liquid from the lower layer in the first cooling chamber to the lower layer in the second cooling chamber, a passageway in the lower portion of the second cooling chamber for the withdrawal of molten metallic liquid lean in said component, movable cooling means associated operatively with the second cooling chamber for movement into and out of the body of molten metallic liquid confined therein to cool the same and to effect the deposition in solid form of a substantial amount of the component thereon, transfer means associated with the movable cooling means and the first cooling chamber for moving the solid component so deposited into the first cooling chamber for melting in the body of molten metallic liquid confined therein and enriching the same with said component, and an outlet at the top portion of the first cooling chamber for the withdrawal of molten component from its upper layer of molten metallic liquid.

15. Apparatus according to claim 14, in which a passageway at the lower portion of the first cooling chamber communicates with the lower portion of a well for receiving initially the molten body of metallic liquid to be treated.

16. Apparatus according to claim 14, in which the passageway in the lower portion of the second cooling chamber communicates with the lower portion of a receiving chamber for the cooled molten metallic liquid lean in said component, and an outlet is provided in the lower portion of the latter chamber for the withdrawal of such cooled liquid.

17. Apparatus according to claim 14, in which the movable cooling means includes a rotatable drum extending into the second cooling chamber sufficiently to dip into the body of molten metallic liquid confined therein, and the transfer means includes a deflector exteriorly of the second cooling chamber for separating a sheet of such deposited component from the drum and diverting it toward and into the first cooling chamber.

18. Apparatus according to claim 14, in which the movable cooling means includes a horizontally disposed drum extending at least in part across the second cooling chamber and dipping into the body of molten metallic liquid confined therein, and the transfer means includes a deflector exteriorly of the cooling chambers for separating a sheet of such deposited component from the drum and diverting it toward and into the first cooling chamber.

19. Apparatus according to claim 14, in which the movable cooling means includes a horizontally disposed drum extending at least in part across the second cooling chamber and dipping only into the upper layer of the body of molten metallic liquid confined therein, and the transfer means includes a deflector exteriorly of the cooling chambers for separating a sheet of such deposited component from the drum and diverting it toward and into the first cooling chamber.

20. Apparatus according to claim 14, in which the movable cooling means includes a horizontally disposed drum extending at least in part across the second cooling chamber and dipping into the body of molten metallic liquid confined therein, the drum being provided with special cooling means for lowering its temperature, and the transfer means includes a deflector exteriorly of the cooling chambers for separating a sheet of such deposited component from the drum and diverting it toward and into the first cooling chamber.

21. Apparatus according to claim 14, in which the movable cooling means includes a horizontally disposed drum extending at least in part across the second cooling chamber and dipping only into the upper layer of the body of molten metallic liquid confined therein, the drum being provided with special cooling means for lowering its temperature, and the transfer means includes a deflector exteriorly of the second cooling chamber for separating a

sheet of such deposited component from the drum and diverting it toward and into the first cooling chamber.

22. Apparatus according to claim 14, in which the movable cooling means includes a horizontally disposed drum extending at least in part across the second cooling chamber and dipping into the body of molten metallic liquid confined therein, the drum being provided with a pipe for spraying cooling water therein, and means for collecting water overflowing from an orifice in the drum.

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