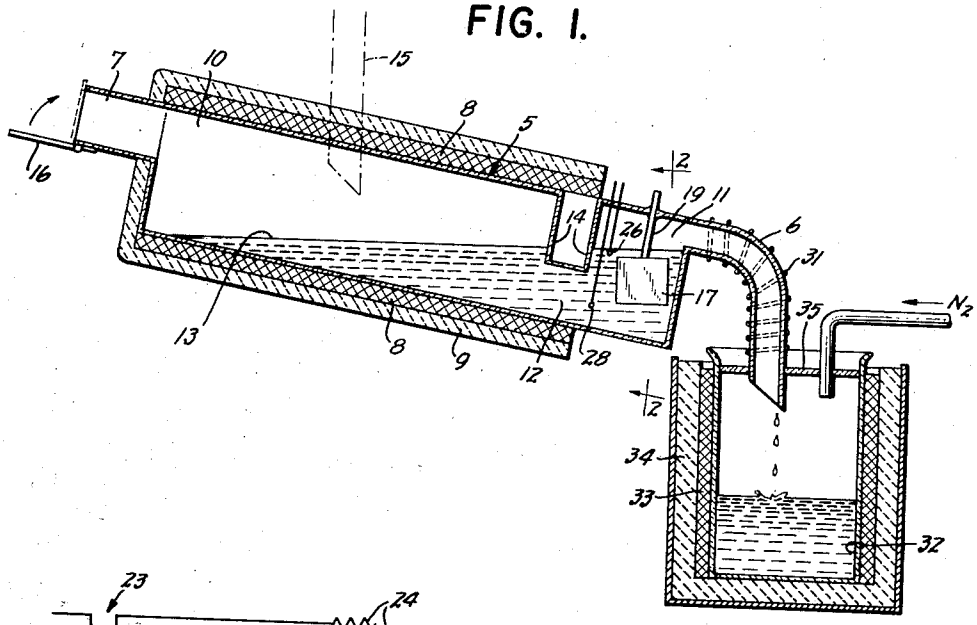
May 12, 1959 J. JERGER, JR., ET AL 2,886,491

METHOD OF GLASS MANUFACTURE

Filed Jan. 12, 1953

FIG. 1.



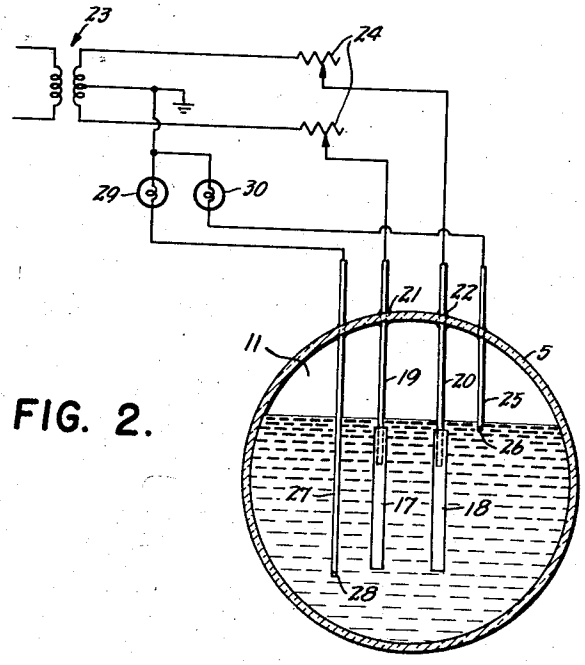


FIG. 2.

INVENTORS
JOSEPH JERGER JR.
WALTER A. FRASER
BY
Mitchell & Beckert
ATTORNEYS

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METHOD OF GLASS MANUFACTURE

Joseph Jerger, Jr., New Hyde Park, and Walter A. Fraser, Bayville, N.Y.

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6 Claims. (Cl. 202—39)

Our invention relates to a method and means for refining materials, such as certain glasses, having substantially a single boiling point or a relatively narrow boiling range.

It is an object of the invention to provide an improved method and means of the character indicated.

It is another object to provide an improved method and means for refining materials which become electrically conductive only when liquified or when heated to temperatures near the boiling points for such materials.

It is a further object to provide improved glass-refining methods and apparatus.

It is a specific object to meet the above objects with equipment lending itself to relatively continuous operation.

Another specific object is to provide improved liquid-level indicating means for refining equipment of the character indicated.

Other objects and various further features of novelty and invention will be pointed out or will occur to those skilled in the art from a reading of the following specification in conjunction with the accompanying drawings. In said drawings, which show, for illustrative purposes only, a preferred form of the invention:

Fig. 1 is a simplified sectional view illustrating a preferred refining apparatus according to the invention; and

Fig. 2 is a sectional view taken in the plane 2—2 of Fig. 1 and showing electrical connections to certain of the parts of Fig. 1.

Briefly stated, our invention contemplates improved methods and means for the refinement of a material having essentially a single boiling point or boiling range and thus capable of refinement by distillation. The method and apparatus lend themselves particularly to the refinement of materials which are conductive, at least in their melted state, as for example, certain glasses and metals. If the material is conductive essentially only when melted, then the method of the invention may be said to comprise, first, the melting of the material, and then the passage of an electric current through a volume of the melted material, the current being of a magnitude to induce boiling, so that the refined product may be available through distillation. A heated receiver may hold the distillation products treated in the liquid state, until ready for annealing or other processing into the solid state.

Referring to the drawings, our invention is shown in application to chamber means 5 having a distillation exhaust outlet 6 and a loading inlet 7. The chamber means 5 may contain the material to be refined, and if such material is not conductive in the solid state, then the chamber means 5 may include melting or first heating means 8, such as a jacket of electrical heating coils, encompassing the substantial outer confines of the chamber means 5. The heating means 8 may itself be jacketed by insulation means 9, which may be a belt of refractory material, as suggested by the shading of the drawing.

The chamber means 5 may comprise two vessels 10—11 joined to each other by fluid-communicating means 12 located beneath the intended fluid level 13 so as to define baffle or trap means 14 between both chambers 10—11. However, in the form shown, we form the

chamber means 5 as a single structure which, for the refinement of certain low-melting point glasses, metallic selenium, metallic tellurium, and the like, may be of Pyrex glass, fused silica, or ceramic-coated stainless steel. This single structure is therefore formed with baffle 14 as a reentrant wall or barrier dividing the chamber means 5 into the two parts 10—11 already identified.

In certain cases, it may be desired to feed the loading or melting chamber 10 with liquid material, as from a synthesizing unit (not shown); in that event, a vertically extending tube, suggested by the phantom outline 15, may project into the chamber 10 from above. However, for ordinary batch preparations, it will be found convenient to load solid material into the chamber 10 through a side-loading entrance 7 located above the liquid level 13. In order to prevent the exhaust of vaporous products from chamber 10, we prefer that the loading inlet 7 shall include means 16 for closing off the inlet execept when loaded. The means 16 may be a simple door or flap, spring-urged to closing position, as will be understood. The bottom of the chambers 10—11 are preferably continuous with each other and are downwardly inclined from the horizontal in the direction of the chamber 11, as shown, so that melted batch materials may more readily gravitate to the chamber 11.

In accordance with the invention, we refine the melted material by electric distillation in chamber means 5, achieved upon passing a sufficiently strong electric current through a localized volume of the melted material. For this purpose, we show two spaced electrodes 17—18 having horizontally opposed substantial areas and contained within the volume beneath the fluid level 13 at the distillation end 11 of the chamber means 5. The electrodes 17—18 may be carbon-graphite plates and include means such as carbon electrode rods 19—20 for vertically suspending the electrode plates 17—18 in the fluid, and for energizing these plates. The rods 19—20 may be sealed, as by fusing at 21—22, to the chamber means 5 and brought externally for connection to a suitable electrical source; of course, if chamber means 5 is of fused quartz, carbon rods 19—20 cannot be fused thereto, but by using a refractory clay or cement at 21—22, an effectively air-tight seal may be produced upon drying. We have suggested that a conventional A.-C. source may be sufficient, and show a transformer 23 having a secondary connected across both plates 17—18 and with means 24 to regulate the applied voltage.

In accordance with another feature of the invention, we provide liquid-level indicating means within the boiling chamber 11. The indicating means may comprise a first electrode 25 contained within the chamber means 5 and effective at an upper level 26 corresponding substantially with the upper limit of the electrode plates 17—18. The electrode 25 may comprise a carbon rod, sealed to the chamber 11 in the same manner as described at 21—22. Similarly, a second indicating electrode 27 may have an effective level 28 substantially at the lower limit of the electrode plates 17—18 and may be sealed to the chamber 11. Single indicating means may be characterized by separate responses to circuit continuity through electrode 25 and to circuit continuity through electrode 27, but in the form shown we illustrate separate indicators. One of these indicators may suitably comprise a first lamp 29 electrically in series with a circuit to ground (midpoint of the transformer secondary) and through one (17) of the boiling electrodes; similarly, a second indicating lamp 30 may be connected electrically in series with another circuit to ground and through the other boiling electrode (18) and the other indicating electrode 27.

In operation, the material to be refined may be introduced continuously or periodically through one of the means 7—15 and maintained in melted condition substan-

tially at the level 13 by means of the heating element 8. In the distillation chamber 11, the heating occasioned by passage of electric current between electrodes 17—18 may boil the glass or other material, and baffle 14 will prevent the discharge of boiling vapors at any point except through the distillation exhaust or condensing tube 6. Ordinarily, chamber means 5 should be loaded as near to the level 13 as possible, as indicated when both indicating lamps 29—30 are lighted; if the liquid drops below the level 13, then the upper lamp 30 will be extinguished and will thus display the need for a further supply. Finally, as when exhausting the chamber means 5, extinction of the lower lamp 29 will indicate that the electrodes 17—18 will no longer function to boil the melted glass.

In order to prevent freezing in the condensing tube 6, we show electric heating means 31 jacketing the tube and maintaining a melting temperature on the inner wall thereof. Condensed droplets may be collected in a receiver 32, which preferably includes heating means 33, for maintaining melting temperatures as long as a batch is being collected. Insulating means 34 may conserve the heating energy. In order to keep the room air out and the heat in the receiver 32, a removable closure 35 may be employed. If desired, a steady stream of an inert gas such as nitrogen may be fed into the receiver as long as the refined product is being collected, thus preventing oxidation of the product.

It will be seen that we have described relatively simple methods and means for refining a material having essentially a single boiling point or boiling range, and not necessarily conductive in the solid state. We have found the equipment to be specifically useful in the refinement of glasses having lower melting points than silica glasses and, in particular, we have successfully refined large batches of arsenic trisulphide and selenide and telluride glasses. As indicated generally above, the method and apparatus lend themselves also to the refinement of metals as, for example, metallic selenium and tellurium.

While we have described the invention in detail for the preferred method and form shown, it will be understood that modifications may be made within the scope of the invention as defined in the claims which follow.

We claim:

1. A method for refining a glass-forming material of the type selected from the group consisting of arsenic trisulfide, selenide and telluride glasses chracterized essentially by a single boiling range which comprises, confining and melting said glass-forming material in an enclosed melting zone maintained at a temperature below the boiling point of said material, passing said melted material to a confined boiling zone via means adapted to prevent the backward flow of vapors, heating said material in said boiling zone by means of electrical conduction to above the boiling temperature via immersed electrodes to form a vapor product, and removing said vapor product and condensing the same to form a liquid condensate at a temperature between the melting and boiling points of said condensate.

2. The method of claim 1 wherein the said liquid condensate is stirred and homogenized while maintained at a temperature between said boiling and melting points.

3. Apparatus for the refining of glass-forming materials of the type selected from the group consisting of arsenic trisulfide, selenide and telluride glasses characterized essentially by a single boiling range and being electrically conductive in the molten condition which comprises, a unitary structure having an enclosed melting chamber with a first heating means to melt the contents thereof to a glass-melting temperature, a distillation chamber connected to receive the melted contents of said melting chamber by fluid connecting means below the fluid level of said distillation chamber, said fluid connecting means adapted to prevent the back distillation of vapors into the melting chamber from said distillation chamber, a second heating means for said distillation chamber comprising spaced

electrodes adapted to extend below the fluid level in said distillation chamber, current supply means connected to said electrodes for boiling said glass-forming material by the passage of sufficient current therethrough, a distillation exhaust outlet connected to said distillation chamber above the fluid level thereof, third heating means associated with said exhaust outlet at a temperature intermediate the melting and boiling point of said glass, and a condensate receiver for the product discharged from said exhaust outlet.

4. Apparatus for the refining of glass-forming materials of the type selected from the group consisting of arsenic trisulfide, selenide and telluride glasses characterized essentially by a single boiling range and being electrically conductive in the molten condition, which comprises a unitary structure having an enclosed melting chamber with a first heating means to melt the contents thereof to a glass-melting temperature, said melting chamber having a loading opening above the fluid level and a vapor-locking device associated therewith to prevent the escape of noxious vapors therethrough, a distillation chamber connected to receive the melted contents of said melting chamber by fluid connecting means below the fluid level of said distillation chamber, said fluid connecting means adapted to prevent the back distillation of vapors into the melting chamber from said distillation chamber, a second heating means for said distillation chamber comprising spaced electrodes adapted to extend below the fluid level in said distillation chamber, current-supply means connected to said electrodes for boiling said glass-forming material by the passage of sufficient current therethrough, a heated distillation exhaust outlet connected to said distillation chamber above the fluid level thereof, and a heated condensate receiver for the product discharged from said exhaust outlet, said exhaust outlet and said receiver being maintained at a temperature intermediate the melting and the boiling point of said glass.

5. The apparatus as defined in claim 4 wherein said melting chamber is characterized by means for introducing bath material therein and wherein said receiver includes means for closing the same except for reception of the products of said exhaust outlet, whereby warm condensate in the receiver will not be exposed to ambient air.

6. Apparatus for the refining of glass-forming materials of the type selected from the group consisting of arsenic trisulfide, selenide and telluride glasses characterized essentially by a single boiling range and being electrically conductive in the molten condition, which comprises a unitary structure having an enclosed melting chamber with a first heating means to melt the contents thereof to a glass-melting temperature, a distillation chamber connected to receive the melted contents of said melting chamber by fluid connecting means below the fluid level of said distillation chamber, said fluid connecting means adapted to prevent the back distillation of vapors into the melting chamber from said distillation chamber, the bottom of one chamber being coextensive and merging with the bottom of the other, said apparatus bottom being downwardly inclined below the horizontal in the direction towards said distillation chamber, a second heating means for said distillation chamber comprising spaced electrodes adapted to extend below the fluid level in said distillation chamber, current-supply means connected to said electrodes for boiling said glass-forming material by the passage of sufficient current therethrough, a distillation exhaust outlet connected to said distillation chamber above the fluid level thereof, third heating means associated with said exhaust outlet at a temperature intermediate the melting and the boiling point of said glass, and a condensate receiver for the product discharged from said exhaust outlet.

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