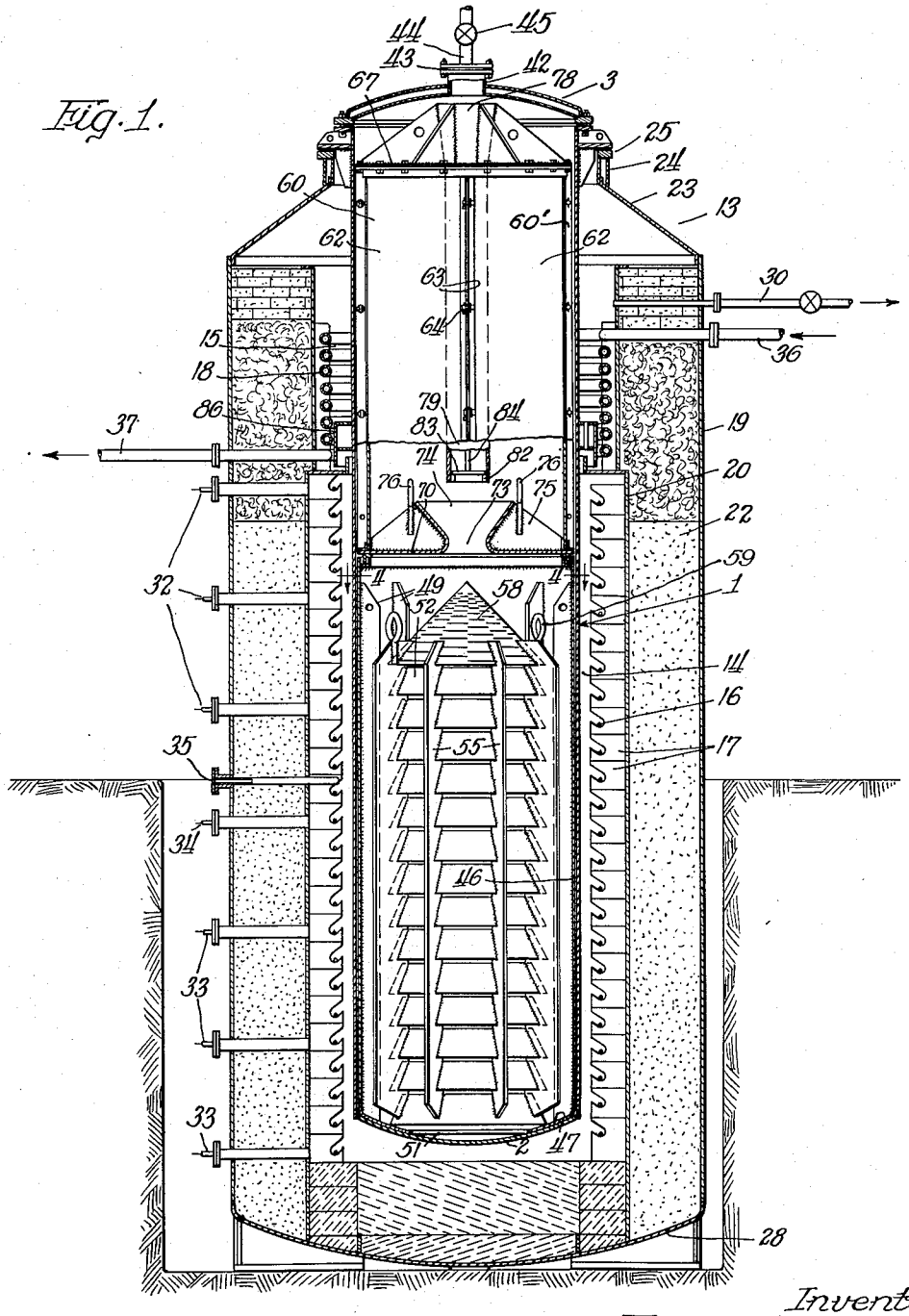
Dec. 18, 1945. F. J. HANSGIRG 2,391,156

APPARATUS FOR REFINING SUBLIMABLE MATERIAL

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Fig. 1.



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Fig. 2.

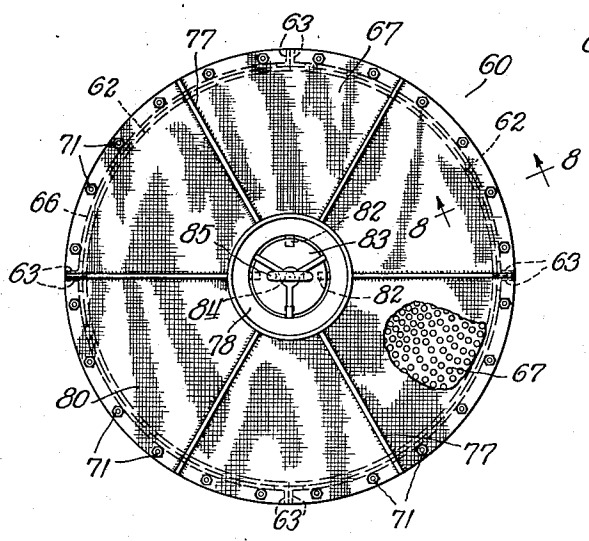


Fig. 8.

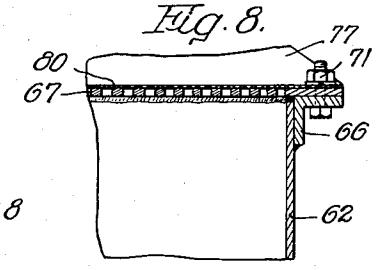


Fig. 9.

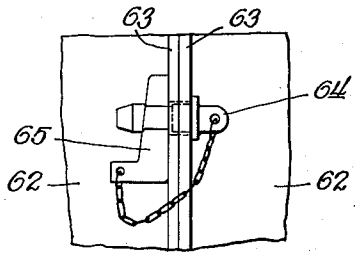


Fig. 10.

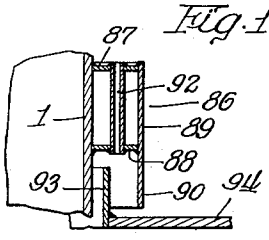


Fig. 3.

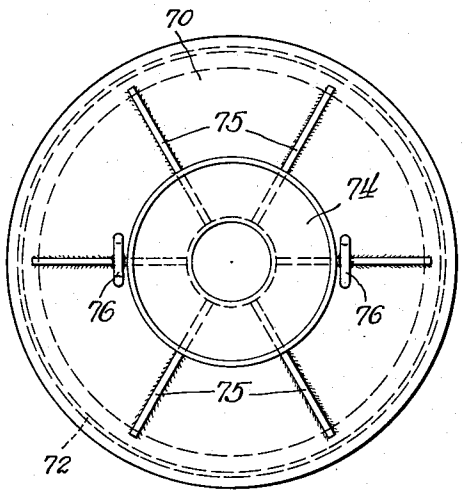
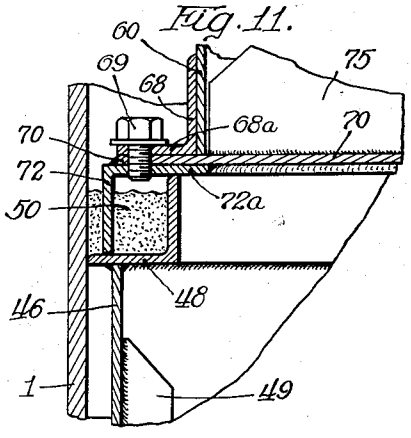


Fig. 11.



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APPARATUS FOR REFINING SUBLIMABLE MATERIAL

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Fig. 6.

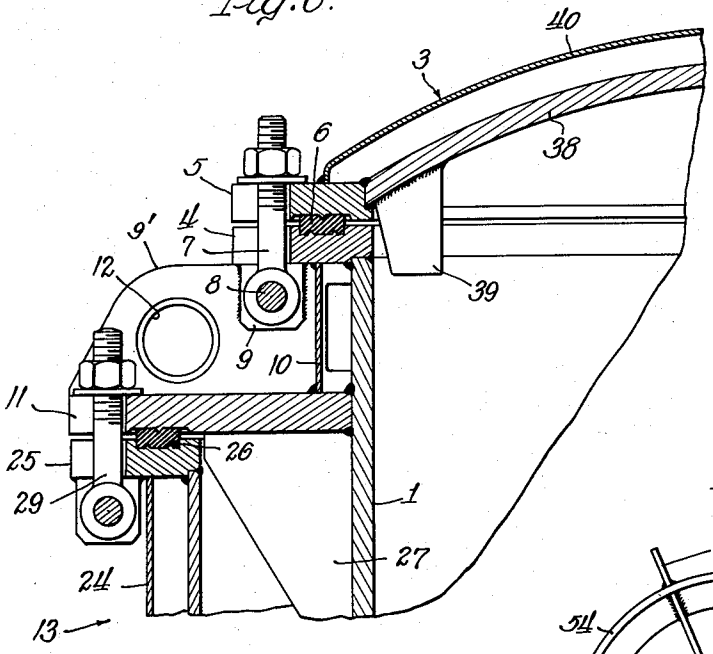


Fig. 5.

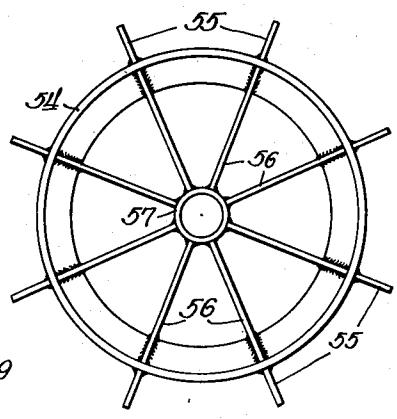


Fig. 4.

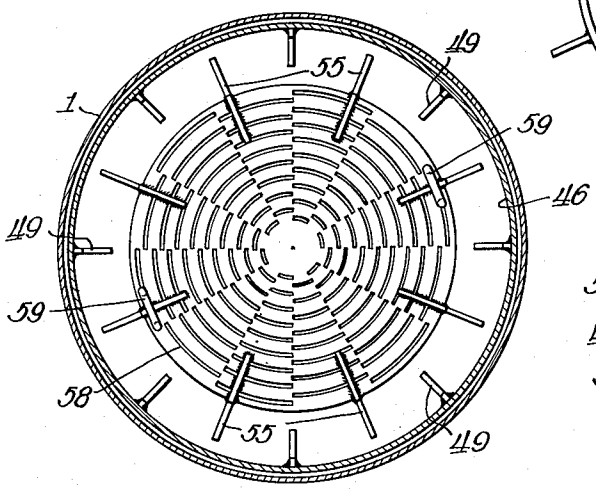
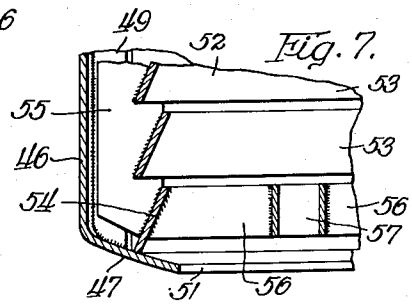


Fig. 7.



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UNITED STATES PATENT OFFICE

2,391,156

APPARATUS FOR REFINING SUBLIMABLE MATERIAL

Fritz J. Hansgirg, Los Altos, Calif., assignor, by mesne assignments, to The Anglo California National Bank of San Francisco, San Francisco, Calif., a national banking association, as trustee

Application February 4, 1942, Serial No. 429,484

4 Claims. (Cl. 266—19)

My invention relates to an apparatus for refining vaporizable, especially sublimable material such as metals, and has in the first instance in view a device which is adapted for refining, by sublimation, crude, alloyed or impure magnesium, particularly magnesium dust as is obtained as an intermediate product in the course of the carbothermic reduction of magnesium oxide-containing material according to my patents Nos. 1,884,993 and 1,943,601. This dust includes not only impurities coming from the ore started with, but also considerable amounts of magnesium oxide and carbon.

In my copending applications S. N. 380,589 filed February 26, S. N. 387,643, filed April 9, and S. N. 411,784, filed September 22, all in 1941, of which this application is a continuation in part, I have disclosed a process and an apparatus especially suited for freeing this dusty intermediate product from said impurities by a sublimation operation capable of being commercially carried out so as to produce magnesium of outstanding purity at a reasonable cost. The herein disclosed apparatus is an improvement on that described and claimed in my aforesaid prior applications, and while embodying the same general principles of operation and construction contains in addition thereto features which permit closer control of the process. A further object of my present invention is, to produce higher durability of the apparatus by enabling the exhausted material under treatment to be more easily discharged.

Other objects and advantages will be apparent from the following specification taken in connection with the accompanying drawings.

The essential elements of the apparatus subject of my present invention include a retort member adapted to be heated for evolving vapor from the material charged therein, a condenser member adapted to be kept cool for condensing the vapor evolved, a liner removably positioned in the condenser member spaced from the walls thereof, and means adapted to establish proper communication between the interior of the charge and the condenser. According to a preferred mode of execution of my invention this means is centrally situated within the charge so as to reduce, by displacement, the radial extent thereof. Moreover, this means are preferably constructed such as to permit controlling the speed of the vapor rising in the condenser.

The removable liner provided in the condenser member to cause the sublimed magnesium to condense thereon and thus to facilitate the removal of the sublimate, is known to the art by itself. It is, however, an important feature of my invention that this liner be positioned so as to provide an annular interspace between the outer face of the liner and the inner face of the condenser wall coacting with said outer face of the liner through a seal for preventing the vapor from rising in the interspace. This arrangement aims at avoiding sudden cooling down of the hot metal vapor and results in obtaining the sublimate in a coarse or even massive crystalline condition which is a considerable advantage, since the melting loss is markedly decreased thereby.

There may moreover be a charge container with a discharge opening in its bottom placed removably in the retort member in which case the means adapted to establish proper communication between the interior of the charge and the condenser, is constructed such that it operates at the same time as a removable locking mechanism for the discharge opening of the charge container.

An apertured partition may be placed between the retort and condenser member.

As disclosed in my prior applications, I prefer to superimpose the condenser member upon a substantially vertical retort member. According to the preferred mode of execution, the apparatus consists of a cylinder-shaped retort having a protracted upper part adapted to serve as a condenser.

Now in order to acquaint those skilled in the art with the manner of constructing and operating an embodiment of my invention, I shall describe, in conjunction with the accompanying drawings forming a part of this specification, a specific embodiment of the invention and the mode of use of the same.

Referring to the drawings:

Figure 1 is a vertical longitudinal section through the apparatus embodying my invention;

Figure 2 is a top plan view of a liner disposed in the upper part of the apparatus;

Figure 3 is a top plan view of a partition positioned between the upper retort and condenser member;

Figure 4 is a section taken on the line 4—4 of Figure 1;

Figure 5 is a bottom plan view of a vapor flow regulator situated in the lower part of the apparatus;

Figure 6 is a fragmentary section taken on an enlarged scale, showing details of the removable cover of the apparatus and of parts associated therewith;

Figure 7 is a fragmentary sectional view on an

enlarged scale, illustrating details of the vapor flow regulator;

Figure 8 is a fragmentary section taken on the line 8—8 of Figure 2;

Figure 9 is a view of details in connection with Figure 2;

Figure 10 is a fragmentary section showing a restriction provided between the upper and the lower part of the mounting; and

Figure 11 is a fragmentary section showing a modified form of construction in connection with the arrangement of the partition.

Referring first to Figure 1, a relatively long and thin steel cylinder 1, which has a closed bottom 2 and releasable cover 3, forms the main wall of the apparatus. This cylinder 1 (see Figure 6) has a clamping flange 4 welded or otherwise secured to its open upper end, and the cover 3 has a mating flange 5 cooperating with the flange 4 and with a yielding seal ring 6. Hinged bolts 7 mounted on hinges 8 in ears 9 swing up through slots formed in the flanges 4 and 5 for releasably clamping the cover 3 to the cylinder 1 which cover is supported in suspension on a flange 11 and provided with reinforcing brackets 9' and a bracing ring 10 to render the flange 11 rigid. The brackets 9' are perforated as indicated at 12 to form ears by which the cylinder 1 can be removed from its mounting 13. This mounting comprises the under zone 14 heated by the electric resistance conductors 16 which are mounted on suitable insulating refractories 17, and an upper zone 15 containing a cooling coil 18.

The mounting 13 includes an outer shell 19 and an inner shell 20, both of metal with insulation indicated at 22 between them. The outer shell 19 is closed at the bottom 28 and has an inwardly tapered or conical wall 23 at its upper end which terminates in a double walled neck portion 24 surmounted by the flange 25. This flange is slotted to cooperate with the slotted flange 11, hinged bolts 29, and a sealing ring 26 to produce an air-tight joint. A vacuum pump connection is indicated at 30 communicating with the enclosed space surrounding cylinder 1. Triangular brackets or buttresses 27 are welded to the flange 11 and to the side walls of cylinder 11 (Figure 6).

The electric heater may preferably be formed in two separate sections, the upper section having the three phase terminals 32 and the lower winding having the three phase terminals 33 leading to the outside with a ground connection such as 34, common to the two of them. One or more pyrometers as indicated at 35 may be provided at suitable points in the mounting. The two sets of heater conductors are independently controllable for applying and distributing the desired amount of heat. The cooling coil 15 is preferably made of steel tubing having an inlet connection at 36 and an outlet connection at 37. All of these connections are made airtight with the outer shell.

The cover 3 of cylinder 1 has a main convex wall 38 arched upwardly and provided with guiding fins 39 around its periphery. An outer thin wall of sheet metal 40 is welded to the flange 5 and spaced from the main wall 38, thereby providing a suitable air space for the thermal insulation of the cover member 38 (Figure 6). At its center the cover member 3 is provided with a short neck 42 surmounted by a clamping flange 43. A vacuum connection 44, controlled by a valve 45, is releasably connected to the tubular connection 42.

The removable liner 60 positioned in the condenser part of cylinder 1 comprises a series of releasable flanged sections, for instance four in number. These flanged sections 62, 62 (Figures 2 and 9) have longitudinally extending mating flanges 63, 63 which are provided with registering holes, with wedge bolts 64 projected through said holes, and with wedges 65 projected through slots in the shanks of the bolts 64. The section 62 are provided at their upper ends with circular flange sections 66 which may be joined to a common cover member 67 (Figure 2). The flanges 66 are provided with bolt holes whereby the margin of the cover member 67 may be bolted to the flanged sections 66 by the usual bolt and nut attachment, as indicated at 71. The removable liner 60 is placed in the condenser part in such position as to provide an annular interspace 60' between the outer face of the liner and the inner face of the condenser wall, sealing means for preventing the rising of vapor from the retort part of the cylinder 1 into said annular interspace being provided.

The retort part of the cylinder 1 which is within the heating zone may be provided with a thin walled steel cylinder 46 slidable in the retort cylinder 1, which bucket serves for receiving the charge. The lower end of this bucket 46 has an inclined flange 47 (shown in detail in Figure 7) which rests upon the bottom wall 2 of cylinder 1 (Figure 1). At its upper end, the bucket 46 coacts with the wall of cylinder 1 through a sand seal. Ribs or flanges 49, which project radially inwardly (Figures 1 and 4) are welded to the side wall of the bucket to perform the dual function of reinforcing and serving as heat conductors to the charge which is disposed in the bucket in contact with them. Convenient means for lifting the bucket out of the retort are provided. The lower end of the bucket 46 has an opening 51 (Figures 1 and 7) for discharging the spent charge. This opening is normally closed by the vapor flow controlling means 52 which is formed of a series of annular louver members 53 (shown in section in Figure 7) having the shape of hollow truncated cones with the parts of smaller diameter being disposed under the parts of larger diameter of the next upper ring. These rings may be rigidly connected with longitudinally running fins or strips 55, which are preferably disposed, as appears from Figure 4, alternating with the ribs 49. A pair of rings or eyes 59 are provided for lifting and lowering the vapor flow controlling means as unitary structure. As appears from Figure 7, bottom ring 54 rests against the flange 47 and thereby closes off the opening 51. This lowermost ring constituting the valve ring, is braced internally and reinforced by radially extending strips or braces 56 which are welded together to a junction ring 57 at the center. At its upper end the vapor flow controlling means 52 is provided with a conical cap or cover 58 which is perforated to form a screen.

Between the retort member and the condenser member there is provided a partition 70 with a central opening 73 through which the vapor released from the charge passes into the interior of the liner 60 (Figures 1 and 3). This opening is surrounded by a funnel-shaped wall 74 for guiding the charge into the charge container. The funnel 74 is reinforced by gussets or bracket-like webs 75 welded in place. Ears or rings 76 provide convenient means for lifting the partition 70 out of the way when it is desired to withdraw the charge container 46.

In the modified form of construction corre-

sponding to Figure 11, in which there is shown a retort 1, the apertured partition 70 is releasably connected to the lower end of the liner 60, so that the partition can be drawn out of the retort in conjunction with the liner. To this end, the liner sections are provided with angle iron reinforcements 68, the horizontally extending flanges 68a of which rest upon the margins of the top surface of plate 70. The said plate has, moreover, a depending annular flange 72 formed by an angle iron ring connected by horizontally extending flanges 72a to the margins of the bottom surface of plate 70. Flanges 68a and 72a are provided with registering holes through which are projected cap screws 69. The depending annular flange 72 forms a seal between plate 70 and the retort wall 1 through the medium of the sand luting 50.

As shown in Figures 1 and 2, the cover of the removable liner 60 comprises a circular disc 67 with a central aperture and having bolt holes around its outer margin for clamping it to the flanges 66 at the upper ends of the liner sections 62. This disc is reinforced by a series of radially extending gussets or braces 77 which join the disc to a central funnel-shaped member 78, the lower end of which is continued as a cylindrical tube 79. Disc 67 is perforated as illustrated in Figures 2 and 8, and the perforations are covered with a screen 80. The tubular duct forming the extension of the funnel 78 terminates above the funnel 74 in the partition 70. It is provided with a valve consisting of a disc 83 resting upon lugs 82 welded to the inside of the lower end of the tube 79. A valve stem 84 extends up to a point within the funnel 78 where a convenient handle 85 (Figure 2) permits the withdrawal of the valve and stem from the charging tube 79. The valve stem is held in central position by a spider near its upper end.

A restriction 86 is disposed between the upper and lower section of mounting 13, to prevent radiation of heat from the hot zone to the cold zone without cutting off the communication with the vacuum conduit. This is shown in detail in Figure 10. A pair of rings 87 and 88 are welded to the side walls of the cylinder 1 and an annular cylinder member 89 providing a depending flange 90 is welded to the outer margins of the annular rings 87 and 88 to provide a dead air chamber which may be pierced by tubing stays 92. The depending annular flange 90 telescopes loosely with a similar upwardly projecting annular flange 93 supported on the shoulders of the inner metal lining 20 of the retort mounting.

The entire assembly is mounted vertically upon a suitable foundation.

The operation of the device described is as follows:

The cover 3 is removed and liner 60, partition 70, bucket 46, and vapor flow regulator 52 are brought in place. Then the valve 83 is withdrawn from the charging tube 79 and the charge, which may consist of pellets or tablets of compacted magnesium powder, is fed in through the funnel 78 and the charging tube 79. This material drops down through the opening 73 and is spread by the cone 58 in the open annular space between the fins 49 on the inside of the bucket 46 and the fins or strips 55 on the outside of the vapor flow regulator. Thereupon the valve 83 is replaced, the cover member 3 securely fastened in position, and vacuum applied, through connections 44 and 30, to produce a residual pressure below 2 mm. as well in the interior of the cylinder 1, as in the interspace surrounding it. Heat is now applied to maintain the charge at a temperature below the melting point of magnesium. The vapor disengaged passes through the lateral openings of member 52 into the central channel formed by the interior of the superimposed rings thereof, escapes through the perforations in the cone 58, and rises then through the aperture 73 of partition 70 in the condenser 62, where it condenses as a thick annular deposit of massive crystalline character adhering to the inner face of the liner 60.

There follow the steps of completely cooling down, opening cylinder 1, and lifting out the liner 60 for recovering the sublimate. If not connected to the liner 60, the partition 70 is then removed separately. The charge container 46 with the vapor flow regulator 52 and residue contained therein, can now be drawn out of the retort and passed to a suitable point, where, by raising the flow regulator with its valve member 54, the spent charge is discharged by gravity through opening 51. Finally the vapor flow regulator 52 is dropped down into position closing the opening 51, and the bucket 46, partition 70 and liner 60 are rearranged in their proper places as shown in the assembly of Figure 1 for repetition of the cycle.

In case no bucket 46 be used the removable liner is supported, for example, by angles fastened to the container wall to hold the liner in proper position within the condenser. In this case the spent charge is discharged by turning over the container to tilt out the residue from the retort.

I claim:

1. An apparatus for extracting metal in the vapor state from material bearing such metal, comprising a longitudinally extended retort member closed at the bottom and adapted to be heated for evolving vapor from material charged therein, and superimposed thereupon an elongated condenser member provided with a removable cover and adapted to be kept cool for causing the vapor evolved to condense thereon as a solid deposit, in combination with a removable liner positioned in the condenser member so as to provide an annular interspace between the outer face of the liner and the inner face of the condenser member wall, and a charge container with a discharge opening in the bottom, liftably placed in the retort member; means for establishing proper communication between the interior of the charge and the condenser member, adapted to control the speed of the vapor rising in the condenser member, and constituting a liftable blocking mechanism for the discharge opening of the charge container and being substantially centrally positioned in the retort member, so as to reduce by displacement the radial extent of the charge under treatment.

2. In a retort, a substantially vertical retort cylinder having a closed bottom, a removable charge container disposed in the lower end of the cylinder, said charge container comprising a bucket having an opening in the lower end thereof, a central tubular member having lateral openings, the lower end of said tubular member constituting a removable closure for said opening.

3. In a retort, a substantially vertical retort cylinder having a closed bottom, a removable charge container disposed in the lower end of the cylinder, said charge container comprising a bucket having an opening in the lower end thereof, and a valve member for closing said

opening, said valve member having an operating member disposed at the upper end of said charge container, whereby the charge container may be lifted out of the cylinder and the valve member raised to dump the bucket.

4. In a retort, a substantially vertical retort cylinder having a closed bottom, a removable charge container disposed in the lower end of the cylinder, said charge container comprising a bucket having an opening in the lower end thereof, and a lift valve member closing the said opening in the bucket.

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