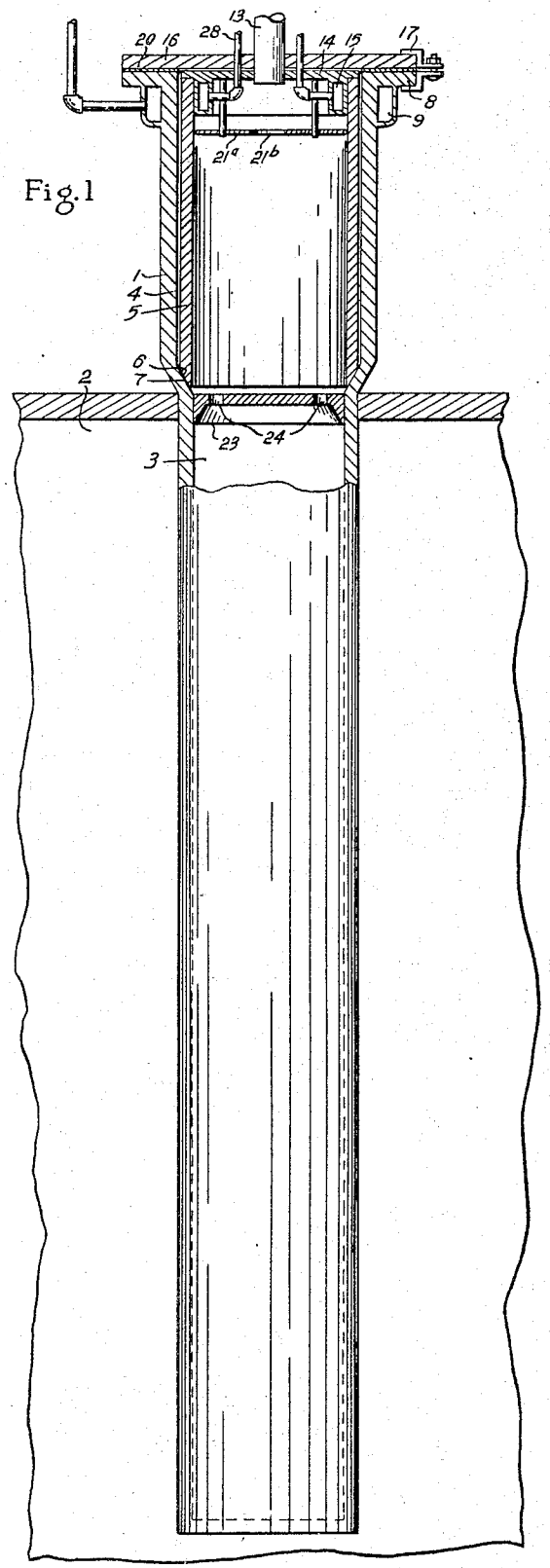
Sept. 21, 1943. L. M. PIDGEON 2,330,142

METHOD AND APPARATUS FOR RECOVERING VOLATILIZABLE METALS

Filed Nov. 3, 1941 2 Sheets-Sheet 1

Fig. 1



INVENTOR:
LLOYD M. PIDGEON

BY Alex. K. MacRae

ATTORNEY.

Sept. 21, 1943. L. M. PIDGEON 2,330,142

METHOD AND APPARATUS FOR RECOVERING VOLATILIZABLE METALS

Filed Nov. 3, 1941 2 Sheets-Sheet 2

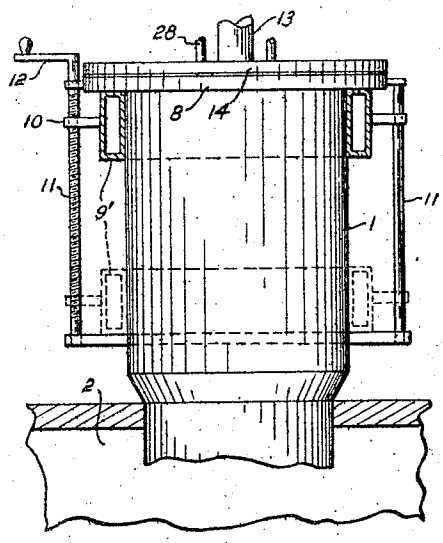


Fig. 2

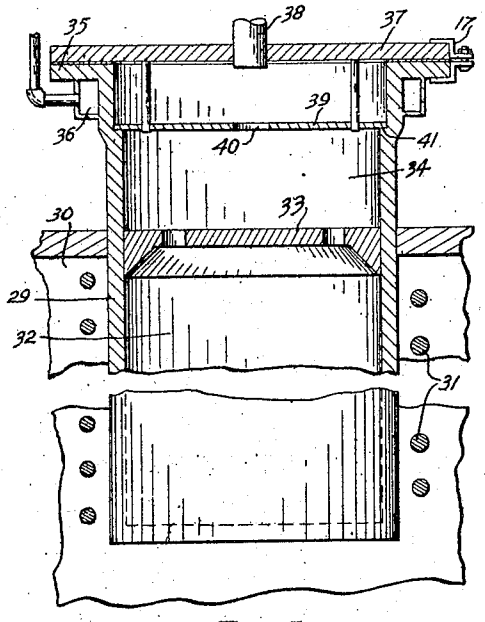


Fig. 5

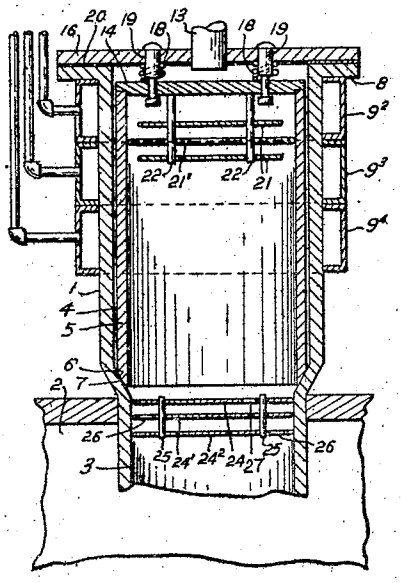


Fig. 3

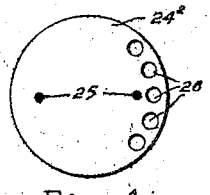


Fig. 4

INVENTOR:
LLOYD M. PIDGEON

BY Alex. E. MacLean

ATTORNEY.

Patented Sept. 21, 1943 2,330,142

UNITED STATES PATENT OFFICE

2,330,142

METHOD AND APPARATUS FOR RECOVERING VOLATILIZABLE METALS

Lloyd Montgomery Pidgeon, Rockcliffe, Ontario, Canada, assignor, by mesne assignments, to Dominion Magnesium Limited, Toronto, Ontario, Canada, a corporation of Canada

Application November 3, 1941, Serial No. 417,819
In Canada October 22, 1941

33 Claims. (Cl. 75—67)

This invention relates to a method and apparatus for the recovery of volatilizable metals and particularly to a retort and condenser for the thermal treatment of metal-containing materials and the recovery of volatilizable metals, such as magnesium and calcium, in substantially pure form.

The invention will be described with particular reference to the production of magnesium but it is to be understood that it may be utilized for the production of other volatilizable metals, such as cadmium and zinc, as well as in the removal of alkali metals and like impurities from calcium in the production of the pure metal.

In the production of magnesium by thermal methods difficulties are encountered in successfully condensing the magnesium vapour and many proposals have been made for the production and condensation of such vapours. For example, a frequent source of trouble is occasioned by the ignition of sodium liberated from the raw material in producing magnesium and calcium.

The object of the present invention is to provide an improved and conveniently operated method and apparatus for reducing magnesia containing material or for treating magnesium to form magnesium vapour and for condensing the vapour to form the metal in macro crystalline form. Other more specific objects are: to provide against ignition of the metal during discharge of the condenser, to avoid formation of pyrophoric powder, to provide that the retort may remain at substantially operating temperature to avoid deterioration due to repeated changes in temperature when charging and discharging the retort and discharging the condenser, to provide effective temperature control within the condenser, to provide for the fractional separation and ready removal of metals more volatile than magnesium, such as sodium and the like, from the magnesium, and to provide a retort having a plurality of inter-communicating condensing zones in which metals of different vapour pressures may be separately condensed. Other objects will appear from the detailed description of the apparatus now to be given with reference to the accompanying drawings in which

Figure 1 is a longitudinal sectional view of one form of the apparatus,

Figure 2 is an elevation of a modified form,

Figure 3 is a sectional view of a further modified form,

Figure 4 is a plan view of one of the discs of the radiation shield of Figure 3, and

Figure 5 is a sectional view of an alternative form.

Referring to Figures 1, 2 and 3 of the drawings 1 is a metal retort adapted to be located in a furnace 2 in which it is externally heated in any desired way. The retort includes the reduction zone 3 and the condenser zone 4. Within the latter is located a removable condenser 5 the inner end of which is tapered, as at 6, to seat snugly against the complementary bevel 7 in the wall of the retort. The internal diameter of the condenser should be at least as great as, and preferably somewhat greater than, that of the reduction zone of the retort. The retort and condenser constitute a unit, any desired number of which may be used in one furnace. The external diameter of the condenser is sufficiently less than the internal diameter of the retort in the condensing zone that due clearance is provided for easy removal of the condenser. The condenser seating arrangement prevents escape of vapours between the condenser and retort wall.

The condenser end of the retort is provided with a flange 8 and cooling means 9. As shown in Figure 1, the cooling means consists of an annular chamber through which cooling water may circulate in contact with the wall of the retort and the flange. As shown in Figure 2, the cooling means consists of a chamber 9′ which is movable along the wall of the condenser end of the retort by any suitable means. As illustrated, the chamber is supported on a frame 10 and a threaded shaft 11 which may be rotated by the crank 12 is provided for moving the chamber. As illustrated in Figure 3, the cooling means may consist of a plurality of chambers 9^2, 9^3, 9^4 located successively along the wall of the condenser portion of the retort and each having independent means for circulating cooling liquid therethrough.

The unit is provided with a removable closure device which includes a conduit 13 for producing a vacuum within the retort, a pressure plate 14 adapted to rest upon the end of the condenser and press the condenser snugly against the tapered seat 7, and a closure plate 16 adapted to rest on the retort flange 8 and to be secured by any suitable clamps 17. The plates 14 and 16 are arranged to provide in the closure device assembly a resiliency which insures the exertion of a positive pressure on the condenser by the plate 14 and on the flange 8. As shown in Fig-

ure 1, the plates 14 and 16 are secured to the pipe 13 and, as the plate 16 is clamped to the retort, the pipe 13 exerts a positive pressure on the plate 14 resting on the condenser. Alternatively, as shown in Figure 3, coil springs 18 may be arranged between the plates by means of bolts or the like 19. A gasket 20 is preferably inserted between the flange 8 and the plate 16. As shown in Figure 1, an internal cooler 15 with the pipes 28 for circulating cooling fluid is provided. The cooler snugly fits within the discharge end of the condenser and is removable with the closure device.

Carried by the closure device is auxiliary means for condensing vapours of sodium and like metals of relatively high vapour pressure and for removing such metals independently of the magnesium. Preferably this consists of a series of spaced metal plates 21 and 21' carried by the plate 14 by means of the supporting members 22, as shown in Figure 3. The plates 21 are of lesser diameter than the retort while the intermediate plate or plates 21' are cut away at the centre to provide a circuitous path of flow of the sodium vapours about the plates on which the vapours are condensed. The innermost of the plates is substantially independent of the cooler and in operation does not accumulate condensate. As shown in Figure 1, this auxiliary fractionating means is provided by the plate 21a which divides the condenser into two chambers which are in communication through the opening 21b. The plate is carried by the closure device through the supporting members 22.

At the outlet end of the reducing zone of the retort is located a baffle or thermal radiation shield, which as represented in Figure 1 is a plate 23 having perforations 24 therein for the passage of metallic vapours formed in the reducing zone. A modified form of baffled thermal radiation shield is illustrated in Figures 3 and 4. A series of spaced plates or discs 24, 24', 24^2 are mounted on frame members 25. All but the outer disc of the series may be perforated, as at 26, around a section of their periphery as shown to provide a sinuous path of travel for the vapours which on reaching the outer disc pass through the perforations 27 to the condenser. These perforations are preferably arranged more centrally of the disc than those of the other discs. This baffle or shield conserves heat by restricting radiation from the heating zone to the condenser zone.

Referring to Figure 5 of the drawings, 29 is the retort located in a furnace 30 which, as illustrated, may be heated by any desired electrical means represented by the elements 31. The heating or reducing zone 32 of the retort is separated by the perforated thermal radiation shield 33, from the condenser zone 34. The condenser end of the retort has a flange 35 and an annular chamber 36 through which cooling water may circulate. The retort is closed by a plate 37 through which passes a conduit 38 for producing a vacuum within the retort. A fractionating plate 39 having a perforation 40 divides the condenser portion of the retort into two communicating zones which during operation are at different temperatures for the fractionation of vapours of metals of different vapour pressures. This plate is shown as held in place within the retort against the shoulder 41 by the rods 42. Preferably it is carried by the closure 37 so that it is removable therewith.

In operation, with the closure device, condenser and baffle plate removed, a charge of magnesia containing material and reducing agent, or other desired charge, is fed into the reducing or heating zone of the retort, the retort and condenser are closed as indicated and the retort heated in the furnace to volatilize the metal of the charge. For the reduction of magnesia containing material the temperature in the retort is about 1100–1400° C. depending on the nature of the charge. The vapourized metal or metals pass through the radiation shield to the condenser, the temperature of which is controlled.

In the production of magnesium it is desirable to cause the metal vapour to condense in the form of macro crystals. To avoid the formation of pyrophoric powder the vapour should be condensed at a temperature of 200–400° C. under reduced pressure conditions commercially available. When the evolution of vapours from the charge is completed and the vapours condensed, the closure device is removed and the condenser is withdrawn for the removal of the metal. The residue of the charge is removed from the retort and the operating cycle repeated.

It will be observed that as metal vapours are evolved from the charge in the retort they pass through the baffle 24 into the condenser. Vapours of metal of low vapour pressure condense in the initial zone, while those of metal of higher vapour pressure pass through the opening in the fractionating plate assembly to the secondary condensing zone, where they condense and collect independently. Thus, metals, such as sodium, are separated from the magnesium or calcium, constituting the main product and are removed from the condenser independently of the main condensate. This avoids ignition of the main condensate and the sodium may be burned off or otherwise removed from the fractionating condenser plate assembly.

Best results are obtained by maintaining a vacuum in the neighbourhood of 0.1 mm. or less of mercury in the retort. As the metal vapours pass through the baffle plate they condense in the form of crystals on the wall of the condenser. Under conditions in which ignition of the metal, in the hotter end of the condenser, upon opening of the condenser becomes hazardous the variable cooling arrangement may be brought into operation to reduce the temperature below the danger zone after sublimation is complete and before the condenser is opened to the atmosphere.

While a retort has been specifically described, it will be understood that the invention as defined in the appended claims may be adapted to any apparatus or device for producing volatilizable metals.

I claim:

1. A method of producing volatilizable metals from alkali and alkaline earth containing materials which comprises heating the materials in a non-oxidizing atmosphere in a retort to volatilize and sublime the metals in said materials, condensing metal of lower vapour pressure in a condenser within the retort, condensing metal of higher vapour pressure and higher flamability in an auxiliary space within said condenser, opening the retort and removing the condensed metal of higher vapor pressure while leaving the condensed metal of lower vapor pressure in the retort, and then removing the condensed metal of lower vapor pressure from the retort.

2. Apparatus for recovering volatilizable metals from materials containing them, comprising

a retort having therein a heating zone and a condensing zone, means dividing said condensing zone into at least two communicating chambers, one of said chambers being adapted to condense vapour of metal of relatively low vapour pressure and at least one of the other chambers being adapted to condense vapour of metal of higher vapour pressure.

3. Apparatus as defined in claim 2, having a perforated plate dividing the condensing zone of the retort into two chambers, a removable closure for the retort and means connecting said plate to the closure for removal therewith.

4. Apparatus of the character described comprising a retort having a volatilizing zone and a condensing zone therein, a removable condenser in the latter zone having an internal diameter at least as great as that of the volatilizing zone of the retort, a removable closure device for the condenser and retort and cooling means carried by said closure device and projecting within the condenser.

5. Apparatus as defined in claim 4 wherein the inner wall of the retort is outwardly bevelled adjacent the condenser zone and the inner end of the condenser has a complementary bevel to provide a seat for the condenser.

6. Apparatus for volatilizing metals and subliming the same comprising a retort having a volatilizing zone and a condensing zone therein, a removable condenser in the latter zone, removable means for closing the retort and condenser and auxiliary means for fractionally condensing metal vapours within the condenser, said auxiliary means being removable from the condenser independently of metal collected on said removable condenser.

7. Apparatus as defined in claim 6 wherein said auxiliary condensing means comprises a series of spaced plates suspended from the closure device within the condenser and arranged transversely of the condenser to provide a sinuous path of travel for metal vapours.

8. Apparatus as defined in claim 4 having a perforated baffle plate within the retort between the volatilizing and condensing zones thereof.

9. Apparatus for volatilizing metals and subliming the same comprising a retort, a condenser in one end of the retort, a removable closure for the condenser and retort, and cooling means and a fractionating condenser carried by said closure and projecting within the condenser.

10. Apparatus for volatilizing metals and subliming the same comprising a retort having an outwardly inclined shoulder in its inner wall, a condenser within the retort having its end bevelled to rest against said shoulder and a resilient removable closure device clamped to said retort and pressing the condenser against said shoulder.

11. Apparatus for the thermal production of volatilizable metals including a retort, a removable condenser within one end of the retort and a removable closure therefor comprising a pressure plate to close the condenser, and an auxiliary condenser carried by said plate and extending within the condenser and a plate to close the retort and force said pressure plate against the end of the condenser.

12. Apparatus for the thermal production of magnesium comprising a furnace, a retort having a heating and volatilizing zone within the furnace and a condensing zone without the furnace, a main condenser removably arranged within the condensing zone, a removable closure head for the retort and condenser internal cooling means carried by the head, and external cooling means adapted to cool successively various portions of the condenser.

13. Apparatus as defined in claim 12 wherein an auxiliary fractionating condenser is carried by said head.

14. Apparatus as defined in claim 12 wherein a perforated baffle plate is located in the retort between the volatilizing and condensing zones.

15. Apparatus as defined in claim 12 wherein said external cooling means is movable longitudinally of the retort.

16. Apparatus for recovering volatile metals from materials containing them comprising a retort having therein a heating zone and a condensing zone, a removable condenser in the latter zone, said condenser having therein a plurality of communicating chambers of different temperatures for fractionating metal vapours.

17. Apparatus for recovering volatile metals from materials containing them comprising a retort having therein a heating zone and a condensing zone, a removable condenser in the latter zone, said condenser having a plurality of communicating chambers of different temperatures and having at its outer end a closure device having means attached thereto for forming one wall of at least two of said chambers.

18. Apparatus for volatilizing metals and subliming the same comprising a retort, a condenser in one end of the retort, a removable closure for the condenser and retort, and means within the condenser for collecting metals of relatively high vapour pressure and removing them independently of condensed metal of lower vapour pressure.

19. In apparatus of the character described a condenser for metallic vapours, and within the condenser means for collecting solid metal of relatively high vapour pressure and removing them independently of condensed metal of lower vapour pressure.

20. Apparatus as defined in claim 19 wherein said collecting means comprises a plurality of spaced plates arranged transversely of the condenser, the innermost of said plates being out of direct thermal contact with the condenser wall.

21. Apparatus for the production of magnesium by direct thermal reduction under reduced pressure of magnesium containing material comprising an externally heated retort, a removable condenser in one end of the retort for collecting magnesium, and means within the condenser for collecting metal of higher vapour pressure and removing the same in air independently of and without ignition of the magnesium within the condenser.

22. Apparatus for the production of magnesium by direct thermal reduction under reduced pressure of magnesium containing material comprising a furnace, a retort having a reducing and volatilizing zone within the furnace and a condensing zone without the furnace, a magnesium condenser removably arranged within the condensing zone, and an auxiliary condenser within the main condenser for collecting sodium and removing the same in air independently of and without ignition of magnesium in the main condenser.

23. Apparatus as defined in claim 22 having between said reducing zone and the condensing zone a series of spaced plates providing a sinuous path of travel for vapours passing from the reducing to the condensing zone of the retort.

24. In the production of magnesium by direct thermal reduction under reduced pressure of magnesium containing material, the method which comprises heating the material in a retort to form metal vapour, condensing and collecting metal of high vapour pressure and removing the same from the retort in air independently of the magnesium.

25. In the production of magnesium by direct thermal reduction under reduced pressure in a retort, the method which comprises separately condensing magnesium and sodium vapours and withdrawing sodium and such pyrophoric material from the retort before withdrawing magnesium.

26. A method as defined in claim 25 wherein said pyrophoric material is withdrawn in air.

27. In the production of metallic magnesium by direct thermal reduction under reduced pressure of magnesia containing material, the method which comprises heating the material to form metal vapours in a metal retort disposed within a furnace under normal pressure and with the inside of the retort under subatmospheric pressure with an end portion without the furnace to receive and condense said vapours, cooling said end portion, retarding the flow of said vapours at a point within the condenser adjacent to but removed from the outer end of the condenser portion of the retort to condense therein magnesium vapour to solid form in one zone, further cooling the remaining vapours adjacent the outer end of the retort, discharging the retort while hot in air and recharging the hot retort.

28. Apparatus for the production of magnesium by thermal reduction under reduced pressure comprising a furnace, a metallic retort having a reducing and volatilizing portion fixed within said furnace under normal pressure and at least one condensing portion without the furnace, a removable cover to close the condensing portion to the atmosphere, a magnesium condenser removably arranged within the condensing portion, a partition spaced from said cover dividing the condenser portion into a plurality of zones, a vapour passage through said partition and means for providing reduced pressure in the retort.

29. Apparatus as defined in claim 28 wherein said partition comprises a plurality of spaced plates arranged to provide a sinuous vapour passage therethrough.

30. Apparatus as defined in claim 28 having a heat retarding shield within the retort between the reducing and condensing portions thereof.

31. Apparatus as defined in claim 28 having a heat retarding shield comprising a series of spaced plates providing a sinuous path of travel for vapours passing from said reducing portion to the condensing portion of the retort.

32. Apparatus for the production of magnesium by thermal reduction under reduced pressure, comprising a furnace, a metal retort having a reducing and volatilizing portion fixed within said furnace under normal pressure and at least one condensing portion without the furnace, a removable cover to close said condensing portion, means for providing reduced pressure in the closed retort, a removable condenser within the condensing portion to receive magnesium vapour condensed in solid form, and a partition having an opening therein spaced from said cover to divide the condensing portion into at least two zones and to provide a relatively cool zone adjacent said cover.

33. In a furnace for producing metallic magnesium by thermal reduction under reduced pressure, a metallic retort which in operation comprises a hot reducing and volatilizing zone and a cooler condensing portion, said condensing portion consisting of a relatively warm zone and a cooler zone, the two zones of the condenser being separated by a partition having a vapour passage therethrough.

LLOYD MONTGOMERY PIDGEON.