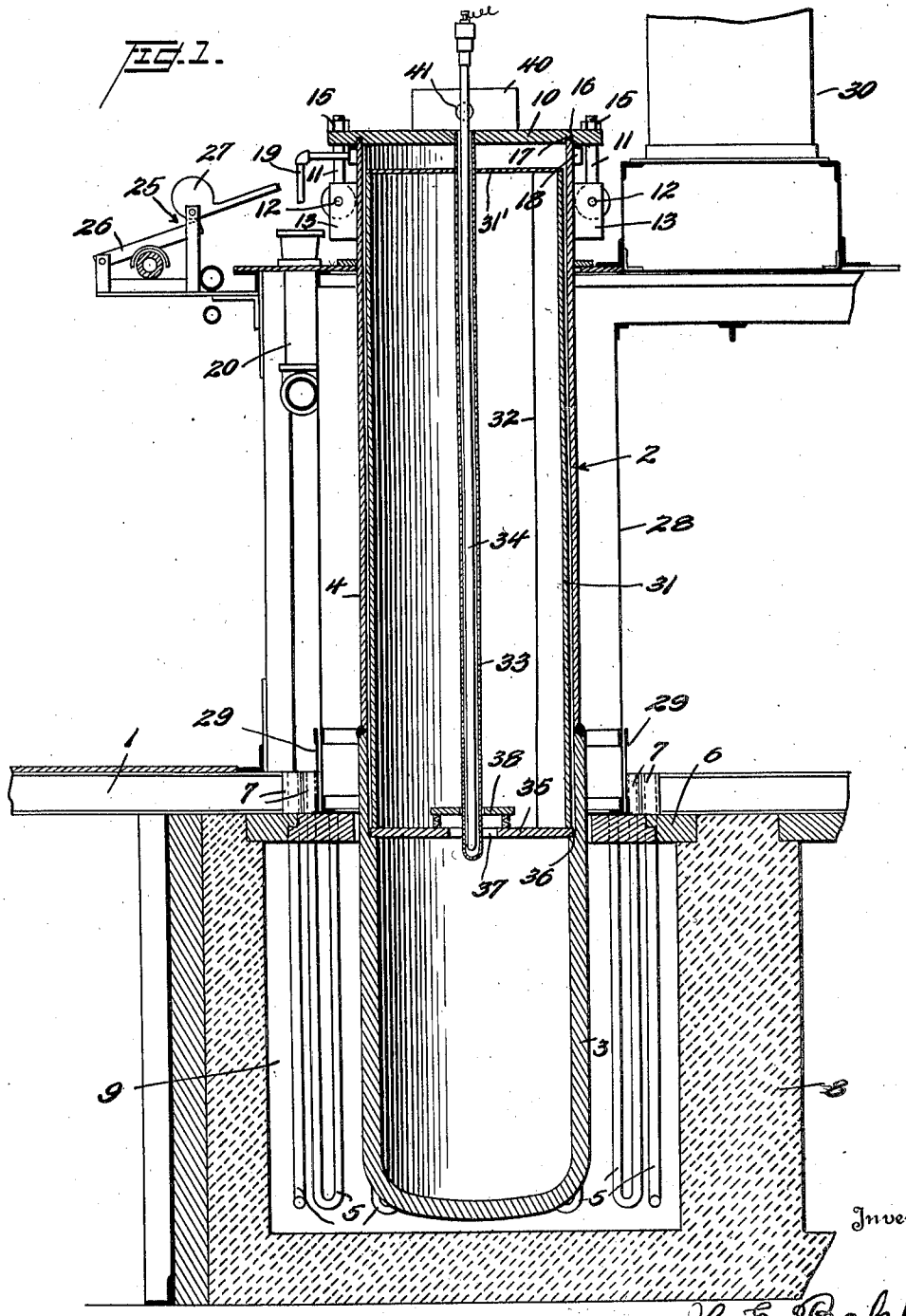
Aug. 3, 1926. 1,594,348

H. E. BAKKEN

SUBLIMATION APPARATUS

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FIG. 1.



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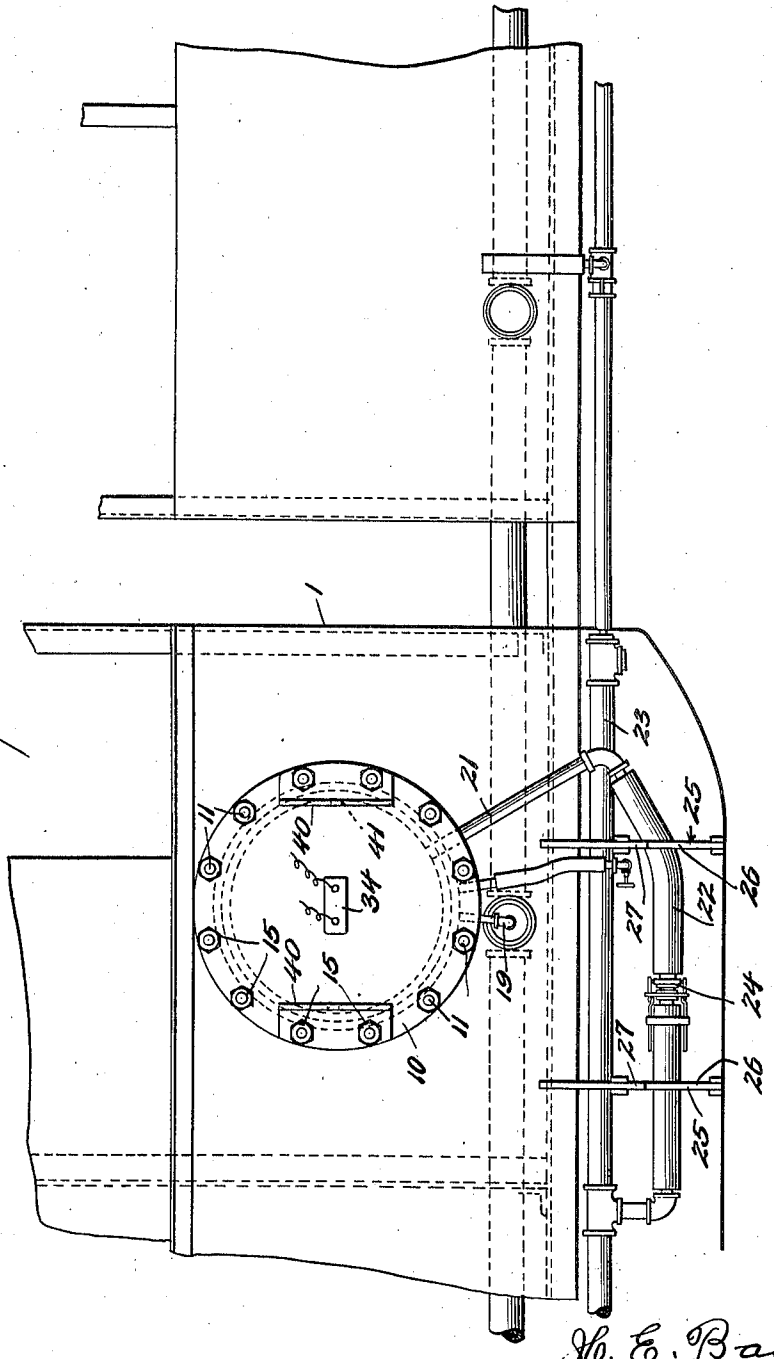
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SUBLIMATION APPARATUS

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Fig. 2.



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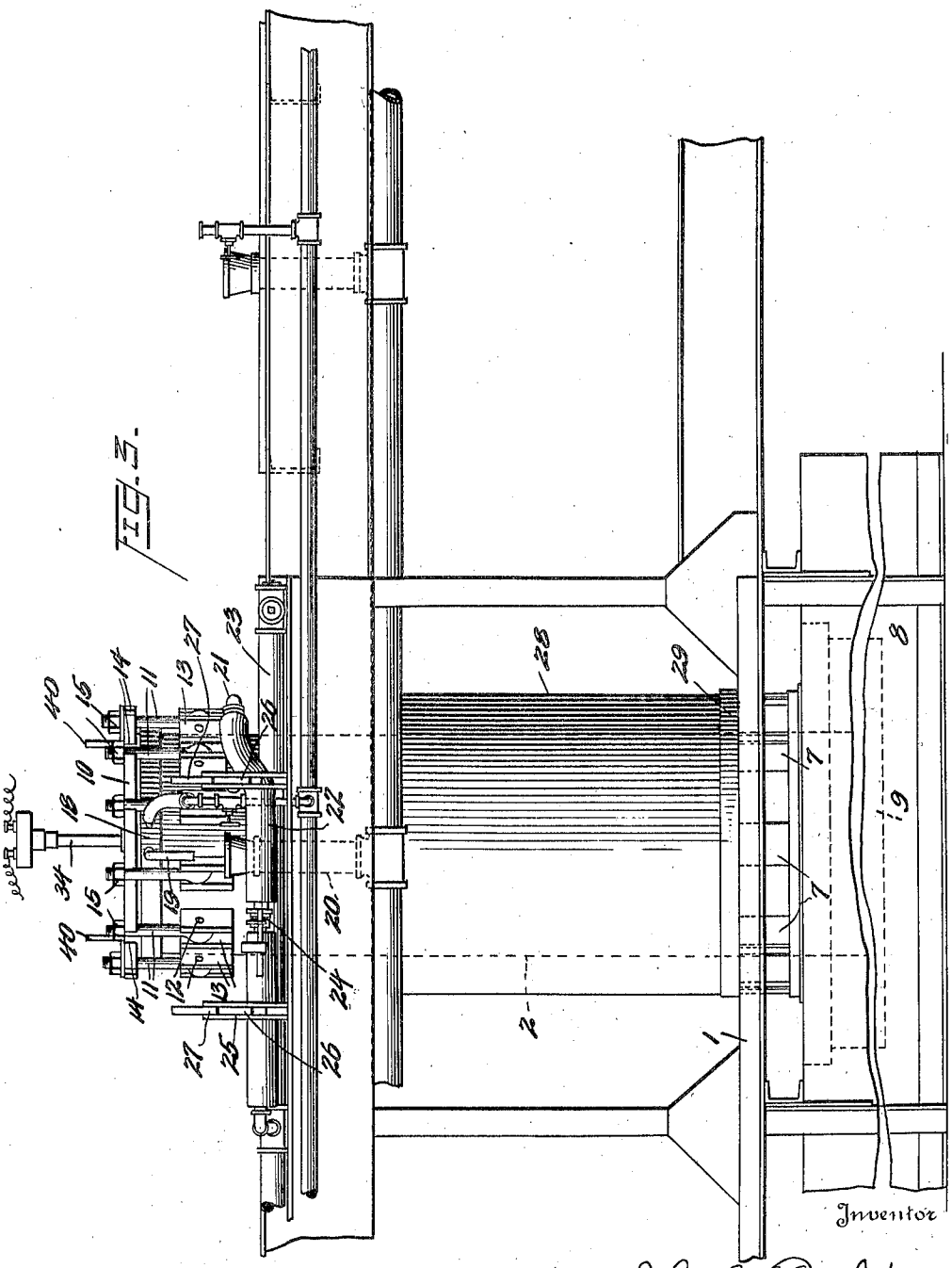
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Aug. 3, 1926. 1,594,348

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SUBLIMATION APPARATUS

Filed Feb. 10, 1925 3 Sheets-Sheet 3



Patented Aug. 3, 1926. 1,594,348

UNITED STATES PATENT OFFICE.

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SUBLIMATION APPARATUS.

Application filed February 10, 1925. Serial No. 8,159.

My invention relates to an apparatus for subliming metals and has particularly in view a device which is adapted for refining crude, alloyed or impure magnesium.

In my co-pending application, S. N. 598,-292, filed November 1, 1922, of which this application is a continuation in part, I have disclosed a process and apparatus for producing sublimed magnesium in a crystallized form and substantially free from non-metallic inclusions and metallic impurities.

The invention therein disclosed comprises the purification of magnesium by converting the solid metal directly into the state of vapor and then condensing it directly to the solid state without intermediate liquefaction, and in particular it consists in the discovery of suitable means and conditions whereby such purification can be made practically operable and capable of being commercially carried out so as to produce pure magnesium at a reasonable cost.

It is known that magnesium boils at atmospheric pressure at about 1120 degrees C. Purification by distillation at this temperature, however, is in practice very difficult because of the fact that the necessary apparatus deteriorates rapidly at such a high temperature. By reducing the pressure in the apparatus, i. e. by carrying on the distillation under diminished pressure, the boiling point of the metal can be reduced, until it coincides with the melting point at a pressure of approximately 2 millimeters of mercury.

I have discovered, however, that if the pressure inside the apparatus is still further reduced, say to less than about 1 mm. rapid sublimation of the magnesium may be accomplished at temperatures which are within the range where ordinary iron or steel apparatus will function satisfactorily. At these low temperatures the difficulty of obtaining apparatus which will withstand continued external pressure is also greatly reduced over that incurred when the operation is carried out under reduced pressure at a somewhat higher temperature. In order to obtain rapid sublimation, it is necessary to so adjust or regulate the operating pressure that the vapor pressure of the magnesium at the temperature of the solid metal being sublimed is slightly greater than the total absolute pressure within the condenser. Under these conditions, the evolution of magnesium vapor will be rapid and if proper condensation facilities are provided the rate of sublimation is only limited by the rate at which heat can be supplied to the metal being sublimed.

The invention disclosed in my said co-pending application consists in placing in one end of a closed retort, crude metallic magnesium or magnesium scrap, such as castings, alloys, etc., and then heating the said metal at a temperature of about 300 to 651° C. for a suitable period, say, 5 to 6 hours, while maintaining an absolute pressure less than the vapor pressure of magnesium at the temperature employed. The pressure may vary from approximately .001 mm. at 300° C. to approximately 2 mm. at 651° C. Under such conditions, the magnesium will rapidly sublime and condense in the cooler end of the retort in the form of a loosely cohering mass of aggregated crystals. When the sublimation is completed and the apparatus cooled somewhat, the crystallized masses can be removed in a suitable manner.

The herein disclosed apparatus is an improvement on that disclosed in my aforesaid parent application and while embodying the same general principles of operation and construction contains in addition thereto features which permit closer control of the process and produces as well a more durable apparatus.

One form of apparatus embodying my invention is shown in the accompanying drawings, in which:—

Figure 1 is a vertical section of the furnace;

Fig. 2 is a plan view; and

Fig. 3 is a fragmentary front elevation.

Supported vertically in a suitable frame work 1 is the metal retort 2 comprising a cast steel boot 3 welded to a thinner wrought iron condenser 4. Surrounding the boot 3 are electrical resistance heating elements 5 made of nichrome rods, supported by fire brick slabs 6 and connected to electrical

terminals 7 from whence the necessary current is received. The heating elements 5 are surrounded by fire brick walls 8 suitably spaced therefrom and providing thereby a heating chamber 9 for the boot 3.

The open end of the retort is closed by a cover 10 which is clamped down tight by means of eyebolts 11 mounted to swing on pivots 12 carried by lugs 13 which are welded to the upper end of the retort. The upper ends of the eye bolts swing into notches 14 in the edge of the cover 10 and the cover is then set down tight by screwing up the nuts 15 engaging the threaded ends of the said bolts. For placing and removing the cover 10 in position, it is provided with hoisting angles 40, secured thereto and having apertures 41 for engaging a hoisting means.

Interposed between the upper end of the retort and the said cover is a compressible gasket 16 fitting in a groove 17 in the said cover. In order to prevent deterioration of the gasket from the heat and thus avoid leakage, there is provided adjacent thereto, a water cooled ring 18 welded to the upper end of the retort. The circulating water for the ring is fed through a supply pipe 19 and led off by a drain pipe 20. For producing and maintaining the necessary vacuum in the retort, the upper end is connected to a vacuum pipe 21, which in turn is connected to a rubber hose, 22, the latter being connected to the vacuum line 23. For the purpose of controlling the vacuum during operation of the process, there is interposed in the rubber hose line a quick acting coupling 24 of a conventional type together with hose clamps 25 for shutting off the vacuum from the retort. The hose clamps 25 consist of a pivoted lever 26, which pinches the rubber hose, when the said lever is forced downwards by the cam 27.

Surrounding and spaced from the upper end of the retort is a housing 28 for controlling the temperature of the condenser, having dampers 29 at its lower end and connected at its upper end to a stack 30. Within the condensing end of the retort, I provide a closely fitting removable liner 31 on which the crystals of magnesium condense and adhere. This liner may be split longitudinally as at 32 to facilitate removal of the deposited crystals. The liner may be provided with a removable cover 31'.

Depending from and secured to the cover 10 is a pyrometer well 33 adapted to receive a pyrometer 34, which indicates the vaporizing temperature in the retort.

The removable liner rests at its lower end on a platform 35 removably supported on a shoulder 36 of the boot 3. To prevent the carrying over of solid particles from the volatilizing to the condensing end of the retort, the platform 35 is provided with an aperture 37, through which the gases pass and overlying the aperture is a baffle 38 spaced from the platform. Between the baffle and the platform a screen 39 of suitable mesh may be interposed.

Operation.

The impure magnesium which is to be refined is placed in the lower end of the retort, the cover 10 is placed in position, sealed and vacuum applied. Water is caused to circulate through the cooling ring 18. When the manometer shows that the desired degree of vacuum has been obtained, heat is applied, care being taken to maintain the absolute pressure in the condenser below the vapor pressure of magnesium at its melting point. The temperature of sublimation and conversely of solidification may vary from approximately 300 degrees C. at 0.001 mm. pressure to 651 degrees C. at approximately 2 mm. pressure. I have found that successful operating conditions will involve heating a charge of the metal at a temperature of about 600 degrees C. for a period of 5 to 6 hours and under an absolute pressure of approximately 0.5 to 0.2 mm. of mercury.

It is, of course, necessary that the temperature in the condensing area should be below that of the metal being sublimed, since the difference in vapor pressure due to this difference in temperature is the driving force causing the rapid transfer of the magnesium vapor from the subliming to the condensing end of the system.

For closely controlling the temperature of the air cooled condensing end of the retort, the amount of cooling air passing around the upper end of retort can be regulated by adjusting the size of the openings in the damper. The condensing end of the retort may also be cooled by the use of air under pressure, cooling water or other suitable means.

Under the conditions outlined, the magnesium does not melt but passes directly from the solid into the vapor state. The vapor passes into the cooler portion of the retort where it condenses on the liner.

The connection with the vacuum line having been shut off by pinching the rubber hose through the medium of the hose clamps, the retort cover is removed. The inner surface of the liner will be found to be covered with crystallized magnesium which can be removed in a suitable manner.

My method of causing the sublimed magnesium to condense on a removable surface is an important feature of my invention. In the example given I have shown a cylindrical liner closely fitting the condenser section of the retort and arranged so as to be readily removable. It is found that if this

arrangement or an equivalent one is not used, it will be very difficult to remove the crystallized magnesium, since it may form a compact adherent layer, an inch or two in thickness, on the walls of the retort, from which it would be quite difficult to separate and remove it. The removal of the crystals from the liner is also greatly facilitated by having it made in two sections, rendering the metallic deposits more accessible for removal. Other forms of a removable condensing surface could be employed, such as a suitably cooled cone suspended from the cover, and all such equivalent condensing surfaces are included within the scope of my invention.

I claim:—

1. An apparatus for refining impure magnesium by sublimation comprising in combination a retort, means for heating the impure metal in the retort to volatilize the magnesium, means for reducing and maintaining the absolute pressure in the retort below the vapor pressure of magnesium at its melting point, a portion of the retort being kept cool to cause solidification of the magnesium from the vapor.

2. An apparatus for refining impure magnesium by sublimation comprising in combination a retort, means for heating the impure metal in the retort to volatilize the magnesium, means for reducing and maintaining the absolute pressure in the retort below the vapor pressure of magnesium at its melting point, a portion of the retort being kept cool to cause solidification of the magnesium from the vapor, and a removable liner in the cool portion of the retort on which the solidified magnesium deposits and adheres.

3. An apparatus for refining impure magnesium by sublimation comprising in combination a retort, means for heating the impure metal in the retort to volatilize the magnesium, means for reducing and maintaining the absolute pressure in the retort below the vapor pressure of magnesium at its melting point, a portion of the retort being kept cool to cause solidification of the magnesium from the vapor, and a removable split liner in the cool portion of the retort on which the solidified magnesium deposits and adheres.

4. An apparatus for refining impure magnesium by sublimation comprising in combination a retort, means for heating the impure metal in the retort to volatilize the magnesium, means for reducing and maintaining the absolute pressure in the retort below the vapor pressure of magnesium at its melting point, a portion of the retort being kept cool to cause solidification of the magnesium from the vapor on the walls of the retort, and means for removing the solidified magnesium therefrom.

5. An apparatus for refining impure magnesium by sublimation comprising in combination an elongated retort of substantially uniform cross-section, means for heating the impure metal in one end of the retort to volatilize the magnesium, means for controllably cooling the other end to cause solidification of the magnesium on the walls thereof, and means for reducing and maintaining the absolute pressure in the retort below the vapor pressure of magnesium at its melting point.

6. An apparatus for refining impure magnesium by sublimation comprising in combination an elongated retort of substantially uniform cross-section, means for heating the impure metal in one end of the retort to volatilize the magnesium, means for controllably cooling the other end, a removable liner closely fitting the cool end and on which the solidified magnesium deposits and adheres, and means for reducing and maintaining the absolute pressure in the retort below the vapor pressure of magnesium at its melting point.

7. An apparatus for refining impure magnesium by sublimation comprising in combination an elongated retort of substantially uniform cross-section, having a heating end and a cooling end, the heating end being permanently closed and the cooling end being sealed by a removable pressure tight cover, means for heating the impure metal in the closed end to vaporize the magnesium and means for controllably cooling the sealed end to cause solidification of the vaporized magnesium and means connected to the cool end for reducing and maintaining the absolute pressure in the retort below the vapor pressure of magnesium at its melting point.

8. An apparatus for refining impure magnesium by sublimation comprising in combination an elongated retort of substantially uniform cross-section, having a heating end and a cooling end, the heating end being permanently closed and the cooling end being sealed by a removable pressure tight cover, a compressible gasket between the cover and the retort, water cooling means adjacent the gasket, means for heating the closed end to vaporize the magnesium, the cold end causing solidification of the magnesium from the vapor, and means for reducing and maintaining the absolute pressure in the retort below the vapor pressure of magnesium at its melting point.

9. An apparatus for refining impure magnesium by sublimation comprising in combination a retort, means for heating the impure magnesium in the retort to volatilize the magnesium, means for reducing and maintaining the absolute pressure in the retort below the vapor pressure of magnesium at its melting point, a portion of the retort being kept cool to cause solidification of the

magnesium from the vapor, and a baffle interposed between the volatilizing and condensing ends of the retort.

10. An apparatus for the production of sublimed magnesium comprising in combination a retort, means for heating one end of the retort to volatilize the magnesium, means for cooling the other end to cause solidification of the magnesium on the walls thereof and comprising a housing spaced from the walls of the retort, and damper-controlled openings in the housing to permit the passage of cold air upwards around the retort, and means for reducing and maintaining the absolute pressure in the retort below the vapor pressure of magnesium at its melting point.

11. An apparatus for the production of sublimed magnesium comprising in combination a heavy cast steel container for the material to be heated welded to a thinner wrought iron condenser.

In testimony whereof I affix my signature.

HERMAN E. BAKKEN.