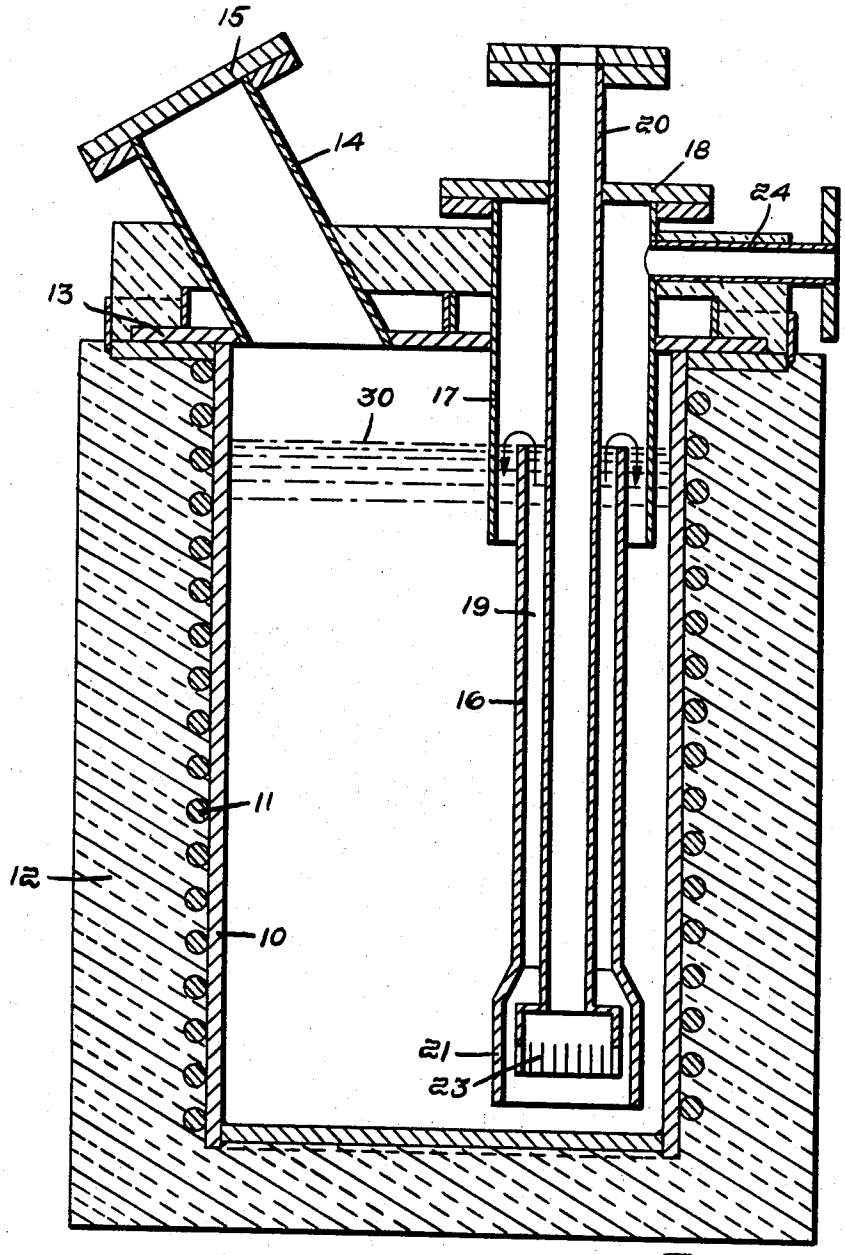
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METHOD FOR PREPARING DICYCLOPENTADIENYL MAGNESIUM

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METHOD FOR PREPARING DICYCLOPENTADIENYL MAGNESIUM

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ABSTRACT OF THE DISCLOSURE

The invention is directed to an improvement in a method for preparing magnesium dicyclopentadienyl wherein cyclopentadiene is reacted at an elevated temperature with magnesium to form magnesium dicyclopentadienyl, the improvement comprising bringing cyclopentadiene in contact with a molten alloy containing about 35 weight percent of magnesium and about 65 weight percent of lead at said elevated temperature at a selected point within a confined reaction zone while causing the molten alloy to move along an endless path passing through said point, a portion of said path extending upwardly from said point.

This invention contemplates a novel method for the preparation of a metaldicyclopentadienyl, such as dicyclopentadienylmagnesium, and an apparatus for use in the preparation of such compounds.

The United States patent to William A. Barber, No. 2,788,377, describes a method for preparing dicyclopentadienyl by reacting cyclopentadiene with solid magnesium, such as magnesium turnings, at a temperature between about 450° C. and about 600° C. or higher. We found that in the practice of this method carbon or magnesium carbide or magnesium acetylide collects on the metal surface, and eventually reaction ceases.

In accordance with the present invention cyclopentadiene is introduced into a body of a molten magnesium alloy at a point a substantial distance below the surface of the molten alloy whereby the cyclopentadiene reacts with the magnesium in the molten alloy to form hydrogen and gaseous dicyclopentadienyl, which separate from the molten alloy at its surface and are caused to pass into a condenser, where the dicyclopentadienyl is condensed to a liquid or solid. Any solid by-products of the reaction are carried out of the zone of reaction to the surface of the molten alloy by upwardly flowing gas stream. The temperature of the molten alloy is maintained between about 450° C. and about 600° C. but a temperature as high as 1000° C. may be used. We have found a temperature between about 540° C. and about 555° C. particularly suitable. Solid magnesium metal may be added from time to time to replace the magnesium consumed by the reaction.

In the practice of the invention we prefer to employ an alloy of magnesium and lead. However, the lead may be replaced by other metals which form sufficiently low-melting binary alloys with magnesium, such as aluminum, silver, bismuth, calcium, cadmium, copper, gallium, mercury, nickel, tin, thallium or zinc. Ternary alloys containing these and other metals also may be used. The preferred alloy contains about 35 weight percent of magnesium and about 65 weight percent of lead.

A particularly suitable apparatus for use in the practice of the invention is illustrated in the single figure of the accompanying drawing. This apparatus comprises a

cylindrical vessel **10**, preferably of mild steel, wound with an electrical heating coil **11**, and provided with suitable insulation **12**. The vessel **10** is provided with a hermetically sealed top or cover **13**, pierced by a conduit **14**, through which the molten alloy, or replacement magnesium metal, may be introduced into the vessel **10**. The outer end of the conduit **14** is normally closed by a detachable cover **15**.

A mild steel tube **16** is suitably suspended within the vessel **10** by means (not shown) with its open bottom end spaced from the bottom of the vessel and its open top end spaced from the top of the vessel, the lower end, portion **21**, of the tube **16** being slightly enlarged. A mild steel tube **17**, supported by the cover **13** by means (not shown), extends through the cover **13** into the vessel **10** and surrounds the tube **16** with its lower open end below the upper end of the tube **16** to provide an annular passage therebetween. The upper end of the tube **17** is closed by a cover **18** secured thereto by means (not shown). A conduit **20** for introducing cyclopentadiene into the vessel **10** extends through the cover **18** of the tube **17** and into the tube **16** with its lower end slightly above the lower end of the tube **16**, the conduit **20** being suitably supported by means (not shown) to provide an annular passage **19** between its outer surface and the inner surface of the tube **16**. Preferably, the conduit **20** is provided with an enlarged slotted lower end, portion **23**. The upper end of the conduit **20** communicates with a source (not shown) of cyclopentadiene under pressure. A conduit **24** provides communication between the interior of the tube **17** and a suitable condenser (not shown) for condensing gaseous dicyclopentadienylmagnesium to liquid or solid.

In operation of the apparatus, molten magnesium alloy is introduced into the vessel **10** to a level indicated at **30**, namely, about 18 inches above the lower end of the tube **16**, and the apparatus is purged with an inert gas, such as argon. The molten alloy is maintained at a desired temperature by the heating coils **11**. Cyclopentadiene is introduced into the conduit **20** under a pressure sufficient to overcome the hydraulic head of the molten alloy and passes into the passage **19** in contact with the molten alloy. As the cyclopentadiene reacts with the molten magnesium alloy in the passage **19** the gaseous dicyclopentadienylmagnesium and hydrogen formed flow upwardly in the passage **19** and separate from the molten alloy at the surface of the latter and then pass through the conduit **24** to the condenser. The upward flow of the gaseous products causes molten metal to flow in an endless path upwardly in the passage **19**, downwardly between the tubes **16** and **17**, and thence upwardly in the passage **19**. If desired, flow of the molten metal may be augmented by a pump withdrawing molten metal from within the vessel **10** and discharging into the bottom open end of the tube **16**. In a given run of 40 hours, 50 liters of liquid cyclopentadiene (run at a flow rate corresponding to 1 to 2 liters liquid cyclopentadiene per hour) were converted in 32 percent yield to the desired dicyclopentadienylmagnesium.

We claim:

1. In a method for preparing magnesium dicyclopentadienyl wherein cyclopentadiene is reacted at an elevated temperature with magnesium to form magnesium dicyclopentadienyl, the improvement wherein the cyclopentadiene is contacted with a molten alloy of magnesium and lead at said elevated temperature at a selected point within a confined reaction zone while causing molten alloy to move along an endless path passing through said point, a portion of said path extending upwardly from said point, said alloy containing about 35

weight percent of magnesium and about 65 weight percent of lead.

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