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METHOD AND APPARATUS FOR STORING ALKALI METALS

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Fig. 1.

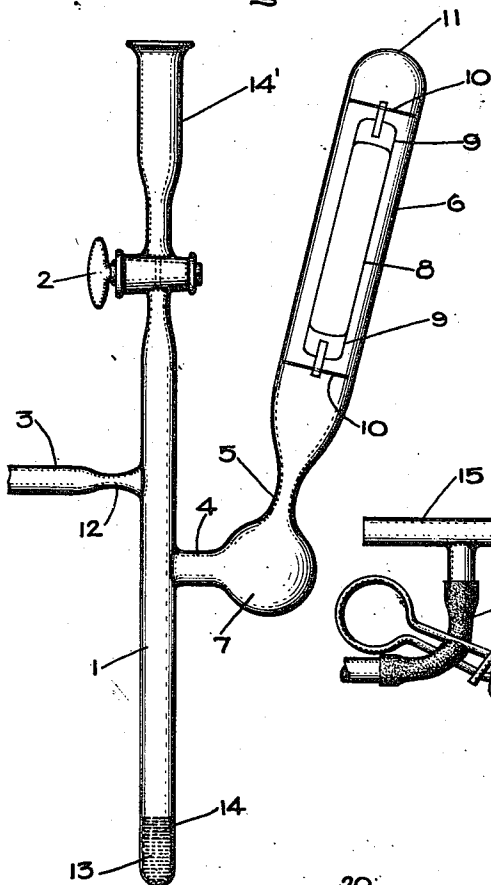


Fig. 2.

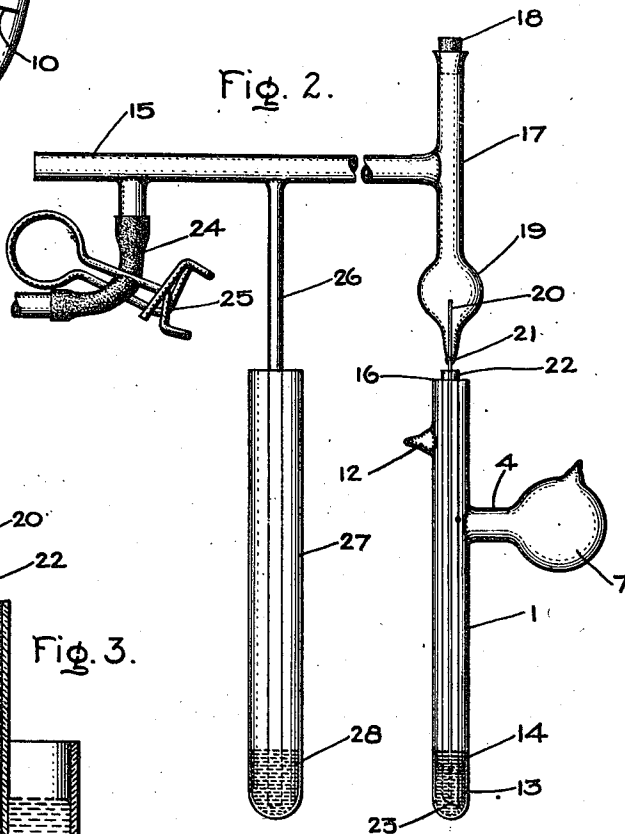
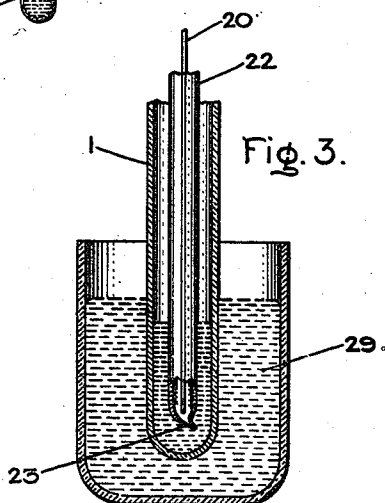


Fig. 3.



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UNITED STATES PATENT OFFICE.

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METHOD AND APPARATUS FOR STORING ALKALI METALS.

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The present invention relates to an apparatus and method for introducing an alkali or readily oxidizable metal into capillary tubing. While attempts have been made to fill capillary tubing with alkali metal, it has been heretofore impractical to provide a capillary tube filled with substantially pure alkali metal owing to the fact that the alkali metal usually becomes contaminated by contact with the metal of the capillary tube and by mixing with materials, such as oils, which are employed in the process of filling the tube. Since the alkali metal stored in the capillary tubing is employed in high vacuum electron discharge devices, it is not only desirable but essential that the alkali metal should be free from all impurities. It is one of the objects of the present invention to provide an improved method and apparatus for filling capillary tubing with substantially pure alkali or readily oxidizable metal.

The novel features which we believe to be characteristic of the present invention are set forth with particularity in the appended claims. The invention itself however will best be understood from reference to the following specification when considered in connection with the accompanying drawing, in which Fig. 1 is a view in elevation of the portion of the apparatus employed in carrying out the first steps in our improved method; Fig. 2 is a view in elevation of a portion of the apparatus employed in carrying out the remaining steps of the process; while Fig. 3 is a fragmentary view on an enlarged scale of a portion of the apparatus employed in the later steps of the process.

Referring to the drawing, I have indicated at 1 a glass tube provided with a suitable stopcock 2 and an extension 3 which is adapted to be connected to a vacuum pump or other suitable evacuating means. A second extension 4, provided with a contracted portion 5 connecting enlarged portions 6 and 7, is also mounted on the tube 1.

A mixture of cesium chloride and calcium is placed in a nickel cylinder 8. The ends of the cylinder are pressed together as indicated at 9 and provided with a plurality of thin spacing wires 10. The nickel cylinder is placed in the enlarged portion 6 of the extension 4, the end of which is sealed off as indicated at 11.

After the nickel cylinder has been enclosed in the chamber 6, the stopcock 2 is closed and

a vacuum produced by any suitable means attached to the connection 3. Any moisture in the apparatus is removed by heating the apparatus with a small flame. When a desired degree of vacuum has been obtained, cesium is distilled into the glass bulb 7 by applying a high frequency heating coil to the nickel cylinder 8. The glass tubes connecting bulb 7 and tube 1 and connecting the extension 3 with the tube 1 are so small that the distilled metal is prevented from going into the tube 1 and extension 3. The tube 6 and extension 3 are now sealed off at the points 5 and 12 respectively; the cesium in the bulb 7 is warmed until liquid, and the tube 1 tipped so that the cesium will run into the end of tube 1 as indicated at 13 in Fig. 1. Stopcock 2 is now opened, and a hydrocarbon oil 14, such as kerosene or molten paraffin, permitted to flow into tube 1 from a receiver 14' on the upper end of tube 1 until the oil reaches a depth of about 1 inch above the cesium, after which the stopcock and receiver are cut off as indicated at 16 in Fig. 2.

To introduce the cesium into a capillary tube, the apparatus indicated in Fig. 2 is employed. This apparatus comprises a horizontal glass tube 15, one end of which is adapted to be connected to an evacuating apparatus, while the other end is provided with an extension 17, the upper end of which is supplied with a stopper 18 and the lower end of which has a bulb portion or chamber 19 provided at its lower end with a small opening adapted to accommodate a capillary tube 20. Tube 20 extends about one-half inch into the bulb 19 and is sealed thereto by wax as indicated at 21. The portion of capillary tube 20 outside of chamber 19 is inserted into a small glass tube or casing 22, the lower end of which is closed and provided with a scratched or readily frangible portion 23 so that the end of the casing may be easily removed or broken away. Casing 22 and its enclosed capillary tube 20 is then inserted in tube 1 so that casing 22 extends well below the oil 14 which rests on the surface of the cesium 13. Pressure is then applied to the upper end of the casing 22 and the lower portion 23 broken off.

A vacuum device is attached to the end of tube 15, and as a result cesium rises in the capillary tube 20. Owing to the manner in which the capillary tube 20 is inserted in the

cæsium, the cæsium is entirely free from oil or other impurities. To regulate the degree of vacuum in the system, a flexible connector 24 provided with a pinchcock 25 is connected to tube 15 and to the atmosphere.

The capillary tube 20 may be made of glass or aluminum. If a metal capillary is employed, it is advantageous to employ a gauge in order to determine the height of the cæsium in the capillary tube. This may be accomplished by providing a glass capillary tube 26 connected to tube 15 and extending into a receptacle 27 containing carbon tetrachloride 28 which has about the same density as cæsium. The tube 26 has an inner diameter substantially equal to the inner diameter of the capillary tube 20. During the filling of the capillary tube 20, tube 1 is immersed in an oil bath 29, see Fig. 3, consisting of kerosene or paraffin, which is kept heated in any suitable manner and to a temperature sufficiently high to maintain the alkali metal 13 in a fluid condition.

When the metal capillary tube is completely filled, the upper end portion is caught with pliers at a point below 19 and closed. The capillary is then cut away from bulb 19 at a point slightly above the closed point of the tube. The opposite end of the tube is closed by pressure on the pliers, and the ends sealed with sealing wax. The tube may then be stored for future use.

If desired, the capillary tube may be cut into convenient lengths, depending upon the amount of cæsium to be used. The ends of the cut portions should be closed and made air-tight. This may be accomplished by spot welding or other suitable means. If glass capillaries are used, they may be divided into convenient lengths by the application of heat, the ends of the small portions being fused air-tight. Although aluminum is entirely satisfactory for the capillary tubing, any metal which will not react with molten cæsium and which can be made into capillary tubes may be employed.

If the cæsium or other alkali metal is to be employed in connection with vacuum tubes, it is of course essential, if a metal capillary is employed, that the capillary be melted by high frequency current and at a temperature sufficiently low to avoid injury to any part of the vacuum tube. The oil employed to cover the cæsium 2 in the tube 1 should be a hydrocarbon oil or organic liquid containing no hydroxyl group or groups which would react with cæsium. Kerosene or molten paraffin as well as an oil known to the trade as Nujol may be employed for this purpose.

What we claim as new and desire to secure by Letters Patent of the United States is:

1. In combination, a liquid metal covered with a layer of material which will not readily react with the metal, a casing having a closed and readily frangible lower end portion extending through said material and into the metal, a capillary tube within the casing, and means for filling the capillary tube with said metal.

2. In combination, a liquid metal covered with a layer of oil, a casing having a closed and readily frangible lower end portion extending into the metal, a capillary tube within the casing, means for filling the tube with said metal, and means for indicating the height of the metal in the capillary tubing.

3. In combination, a liquid metal covered with a layer of oil, a casing having a closed and readily frangible lower end portion extending into the metal, a capillary tube within the casing, means for filling the capillary tube with said metal, and means for regulating the height of the metal in the capillary tube.

4. The method of storing readily oxidizable material in capillary tubing which comprises placing the metal under oil, placing a capillary tube in a casing closed at its lower end, inserting the casing and tube through the oil and into the metal, removing the lower end portion of the casing, and filling the tube.

5. The method of storing a readily oxidizable metal in capillary tubing which comprises placing the metal in a container and under a material which will not readily react with the metal, inserting a casing having a closed readily frangible lower end portion through the material and into the metal, inserting a capillary tube through the casing, breaking the lower end portion of the casing, and applying suction to the upper end of the capillary tubing.

6. The method of filling a capillary tube with readily oxidizable metal which comprises covering the metal with a material which will not react with the metal, liquefying the metal, inserting a casing having a readily frangible lower end portion through the material and into the metal, inserting a capillary tube into said casing, breaking the end portion of the casing, and drawing the metal into the capillary tube.

In witness whereof, we have hereunto set our hands this 26th day of August, 1927.

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