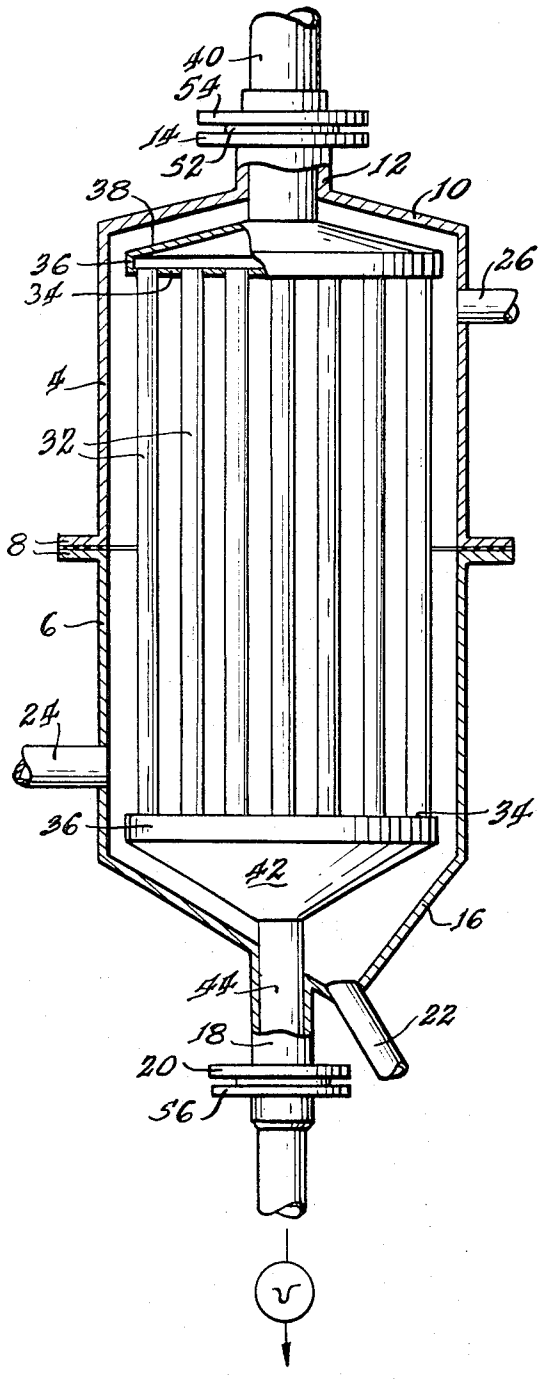
June 11, 1968 S. B. SKLADZIEN 3,387,969

OXYGEN SCAVENGING METHOD

Filed Aug. 2, 1965

Fig. 1



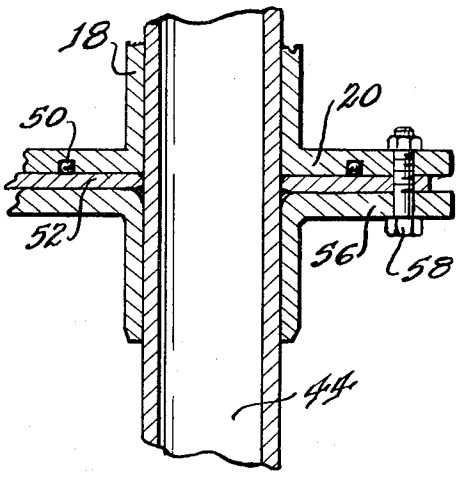


Fig. 2

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United States Patent Office 3,387,969 Patented June 11, 1968

3,387,969

OXYGEN SCAVENGING METHOD

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Filed Aug. 2, 1965, Ser. No. 476,767
3 Claims. (Cl. 75—66)

The invention relates to a method for removing oxide from molten alkali metal and maintaining the alkali metal in an oxide free condition.

In many alkali metal systems it is very important to keep oxide content as low as possible. The presence of oxide in the alkali metal substantially increases the corrosiveness of the alkali metal to many metals. Such metals are necessarily used for containers and other mechanical parts.

There are a number of presently available devices that can accomplish this purpose. All that is needed is a container filled with material that can scavenge oxide from the molten alkali metal. This scavenger can be in any bulky form offering a large amount of surface to the molten alkali metal.

However, these devices suffer from a number of severe shortcomings. It is extremely difficult to determine when the scavenger material is exhausted and severe damage may result before this becomes known. Further, these devices are normally fabricated into a closed system so that the system must be opened to replace a unit. With the known tendency of alkali metal to wet and adhere to metal surfaces, and its tendency to catch fire in air, such a change is a major undertaking.

It is an object of this invention to provide a method by which the oxygen scavenging material is continuously and automatically regenerated.

It is also an object of this invention to provide a deoxidizing device which will function uniformly during its entire life.

It is a further object of this invention to provide a device which eliminates the need of frequent, troublesome replacements.

This is accomplished by providing as the scavenger, a continuous thin zirconium-containing membrane, one surface of which is in contact with the alkali metal, the other with a molten calcium containing metal, which regenerates the membrane by virtue of the unique diffusion of oxide therethrough.

In the drawing:

FIGURE 1 is a vertical, partially sectional view of the apparatus; and

FIGURE 2 is an enlarged vertical sectional view of the bottom portion of FIGURE 1.

Referring to FIGURE 1, the containing vessel **2** consists of an upper shell **4** and a lower shell **6**, each shell being constructed of ¼" thick (AISI designation) 304 stainless steel. Shells **4** and **6** are cylinders 16" OD, 15" high. At their mating ends they are joined to flanges **8**, 19" OD. Upper shell **4** is completed with conical top **10**, rising 2" and joined to nipple **12**, 3" OD, 2" high terminating with 7½" diameter top flange **14**.

Lower shell **6** is closed by skewed conical bottom **16**, through which a 3" long, 2" OD nipple **18** is axially positioned. Nipple **18** is connected to flange **20**, which is 7½" OD. At the lowest point of conical bottom **16**, drain pipe **22**, a length of ¾" OD tubing extends downward. At some point on pipe **22**, a valve (not shown) permits the contents of containing vessel **2** to be drained, when desired. Inlet line **24** and outlet line **26** are attached to the lower shell **6** and upper shell **4** respectively, at opposite sides, 11" from the mating surfaces of flanges **8**.

37 tubes **32** are provided, each of which is a membrane

as mentioned above and is formed of zirconium or a zirconium alloy such as Zircaloy-II. Each tube is 12" long and 1/16" thick and has an outside diameter of 1". The tubes are parallel and have their axis on the corners of equilateral triangles 1" on a side, so that each tube, except the outermost ones, is surrounded by six tubes that have an axis spacing of 1" from one another and from said each tube. The tubes **37** as a group present the general outline of a regular hexagon. The tubes are inserted into tube sheets **34**, which take the form of discs of 3/16" thick, 14" diameter and are formed of zirconium or zirconium alloy. Closure is completed by inert arc welding. On each tube sheet **34**, circular bands **36** 1" wide, 3/16" thick extend outward. Upper manifold **38** is a 3/16" shallow conical vessel, to which is attached upper conduit **40**, which is 3/16" in thickness and 2½" in OD and is formed of zirconium or zirconium alloy. The upper end of conduit **40** leads to a molten calcium alloy storage vessel (not shown). Flow is controlled by a conventional valve (not shown). Lower manifold **42** is a conical vessel of zirconium or zirconium alloy, deeply sloped to avoid collection of sediment, terminating in outlet conduit **44** 3/16" thick, 1½" OD. This conduit **44** terminates in a valve, symbolically shown, which regulates the flow of molten metal to a receiving vessel (not shown).

After assembly, discs **52** of zirconium or zirconium alloy, ⅛" thick, 6½" OD, are positioned on upper conduit **40** and outlet conduit **44** and welded in place by inert arc welding. Flanges **14** and **20** are grooved to accommodate hollow zirconium O rings **50**. Against these O rings **50** are positioned discs **52** welded in position in situ, using inert arc welding. Closure is completed by positioning upper centering flange **54** and lower centering flange **56** against discs **52**. Bolts **58** complete the seal. Heating is accomplished by conventional means, preferably by inductive heat.

Obviously, other simple modifications could be used to carry out the teachings of the patent. All that is necessary is to provide approximately 20–30 square feet of thin walled zirconium or zirconium alloy such as Zircaloy-II, with contact on one surface with calcium metal and the alkali metal to be cleaned on the other surface.

Due to the ease of brittle compound formation between zirconium and iron alloys, such as stainless, all joints between these metals must be mechanical. Welding such members is not desirable.

Optimum operating temperature is 1200°–1300° F. This temperature is below the melting point of calcium, so that it is desirable to add sufficient magnesium to approach one of the eutectics, in the neighborhood of 21% magnesium by weight. A range of 15–30% magnesium should operate satisfactorily.

The present invention is employed to remove oxygen from sodium that is relatively low in oxygen contamination, for example, about 10 parts per million. This condition will have been reached by one or two treatments of the sodium in a cold trap that has wire mesh on which the oxygen is trapped out when the sodium temperatures drop below the saturation temperature, but not below about 225° F.

The need to employ the present invention to obtain sodium with less than 10 p.p.m. oxygen comes from the trend to higher temperatures and use of refractory metals for jackets in sodium reactor systems.

It is to be understood that the invention is not to be limited to the specific design shown or the details presented, but only by the scope of the appended claims.

The embodiment of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method of removing oxide from alkali metal comprising flowing said alkali metal past a membrane of

zirconium or zirconium alloy maintained at an elevated temperature, meanwhile flowing a calcium-containing alloy on the other side of said membrane.

2. The method of claim 1 where the temperature is maintained at 1200°–1300° F.

3. The method of claim 1 where the calcium containing alloy consists essentially of calcium and 15–30 weight percent of magnesium.

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