Aug. 10, 1971 EIZO GOTO 3,598,518

METHOD OF PROVIDING A CONTAINER WITH AN OXYGEN-FREE GAS

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FIG. 1

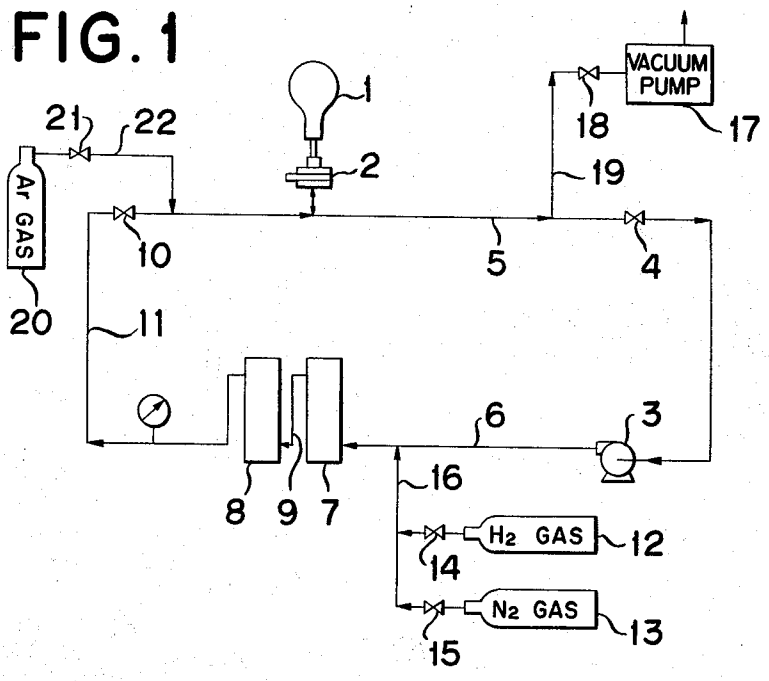


FIG. 3

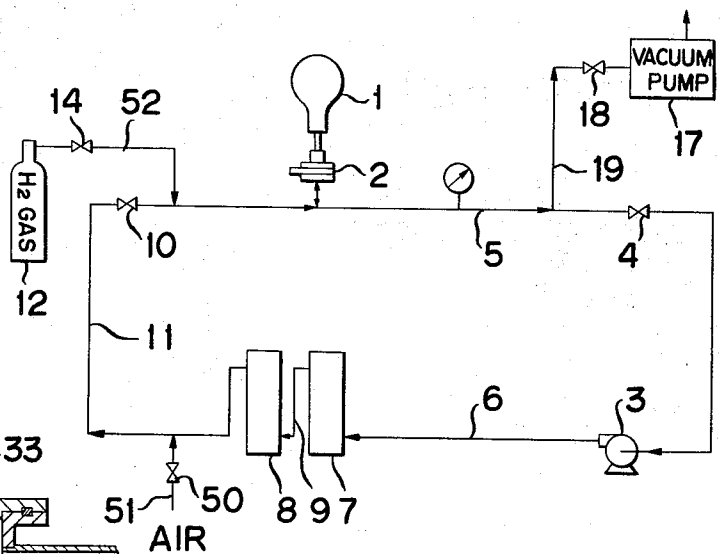
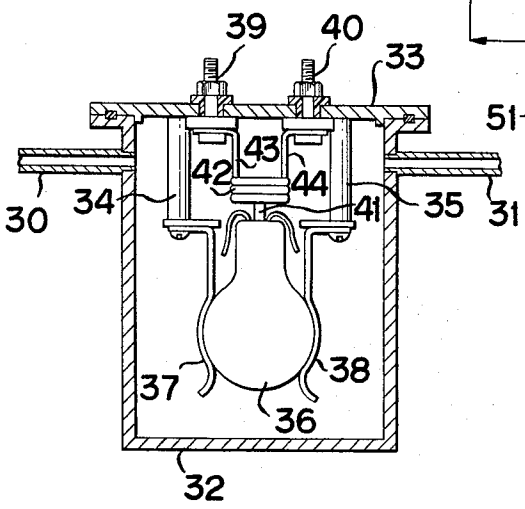


FIG. 2



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METHOD OF PROVIDING A CONTAINER WITH AN OXYGEN-FREE GAS

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ABSTRACT OF THE DISCLOSURE

Method of providing a container with an oxygen-free gas comprising purging the container with a hydrogen containing gas, withdrawing the gaseous mixture from the container, catalytically reacting the oxygen and hydrogen to form water, removing the water and recycling the gas to the container.

FIELD OF THE INVENTION

The present invention relates to a method of gas washing to remove an unnecessary or undesirable gaseous component from gases originally present in the envelope and an apparatus for carrying out said method.

BACKGROUND OF THE INVENTION

The "gas washing method" already established in this particular field of technology consists in replacing unnecessary or undesirable gases originally present in an envelope with desired gases before sealing the envelope. This method is useful in manufacturing electric bulbs or canned goods. After gas washing is completed, a filling gas is introduced into the envelope. The filling gas is generally nitrogen or argon for electric bulbs, and dried air or nitrogen for canned goods. The gas washing method includes a repetitive washing operation in which preliminarily air is removed from the envelope to a relatively low degree of vacuum, a purge gas such as argon or nitrogen is then introduced at a pressure of 200 to 300 mm. Hg for substantial removal of air and finally feeding the filling gas at a prescribed pressure. Since the gas which has been used in washing carries undesirable components therewith, it can not be reused, but generally wasted into the open air. Consequently, the aforementioned gas washing method has the drawback that large consumption of expensible purge gas naturally increases the cost of the washing operation.

SUMMARY OF THE INVENTION

The present invention provides an economical gas washing method comprising:

Forcedly introducing into an envelope with pressure a purge gas;

Drawing out the gas from said envelope;

Removing the gaseous impurity component from the gas; and

Recycling the gas refined in the foregoing step to the interior of said envelope.

According to the method of the present invention, a purge gas is forcedly introduced into an envelope and drawn therefrom along with part of the gas originally present therein, so that the interior of the envelope is substantially kept at an atmospheric pressure or higher throughout the washing operation. In this method the gas washing process is carried out at pressure at which the gas shows molecular flow, thereby eliminating a sucking step as has been required in the prior art gas washing method which has the fundamental drawback that the sucking efficiency is reduced after the envelope is evacuated to the pressure at which the gas in the envelope represents a viscous flow. Further, the purge gas soiled by use in purging is sufficiently purified for reuse by removing an undesirable gaseous component. Consequently the consumption of purge gas is considerably reduced with the resultant decrease in the cost of purging operation.

The present invention is also intended to provide an advantageous apparatus for carrying out the aforementioned process.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic flow diagram of apparatus for removing a gaseous impurity from a container according to the present invention adapted for purging air from the envelope of an incandescent lamp;

FIG. 2 shows is longitudinal section an envelope supporting means adapted in said apparatus; and

FIG. 3 is a schematic flow diagram of another embodiment of the apparatus according to the invention adapted for purging air from the envelope of an incandescent lamp.

DETAILED DESCRIPTION OF THE INVENTION

As described above, the method of the present invention allows hydrogen-containing purge gas to be forcedly introduced into a free oxygen-containing envelope with pressure to provide a pressurized gas mixture and drawn therefrom without sucking by vacuum pump. In the purging operation, the greater is the pressure with which the purge gas is introduced into an envelope, the more effectively will be removed the unnecessary gases originally contained therein. For instance, where a purge gas is first forcedly introduced with pressure continuously into an air-containing envelope at a pressure of one atmosphere until the interior pressure reaches 2 atmospheres, and thereafter the gas content of the envelope is released until said interior pressure falls to 1 atmosphere, then the amount of air remaining in the envelope will be reduced to what was initially present therein. And where the purge gas is continuously introduced with pressure into the air-containing envelope until the interior pressure rises to 4 atmospheres and thereafter is discharged along with part of the air, then the amount of remaining air will be reduced to one-fourth of its original amount. And this purging effect can be realized regardless of the concentration of unnecessary gas contained in the envelope and the inner volume thereof. Therefore the amounts of unnecessary gases can be reduced without any difficulties by repeated purging to the extent that the residual concentration of said unnecessary gases can be well permitted in the envelope. This purging effect can not be obtained with the prior art gas washing method in which an envelope is evacuated in advance, and then a purge gas is introduced into the envelope at a reduced pressure and drawn out. One of the reasons is that as is well known in the art, the evacuation of an envelope will be more obstructed with increasing degrees of vacuum and with larger capacities of the envelope. Furthermore, the present invention allows used purge gas to be purified for reuse.

If it is desired to eliminate water in the envelope as in the case of canned goods, water is removed from the remaining gas by passing the gas through a dehumidifying agent. Where the impurity is oxygen, the purification of purge gas can be carried out by adding to the purge gas 2 mols or more of hydrogen per mol of the oxygen carried by the purge gas and catalytically reacting the hydrogen and oxygen to form water and removing the water from the purge gas. Generally, the gaseous components which are most desired to exclude from the interior of almost all envelopes are oxygen and water vapor. Either or both of hydrogen and nitrogen or other inert gases such as argon will not cause any practical harm even if they are

filled in an envelope. Therefore the gas which has been substantially deprived of oxygen and water is well adapted for purging the interior of an envelope.

The catalyst for reaction of oxygen and hydrogen may consist of any of those generally used in such reaction, for example, platinum, palladium, etc. The water formed by the catalytic reaction is removed from the gas by an ordinary dehumidifying means. The most common type of dehumidifier is a tower packed with a dehumidifying agent such as silica gel or phosphorus pentoxide.

There will now be described the present invention by reference to the accompanying drawings.

FIG. 1 is a schematic flow diagram of an apparatus for removing a gaseous impurity from a container of the present invention suitably arranged to fill argon into the bulb or envelope of an incandescent lamp. A supporting means **2** hold the glass bulb **1** of an incandescent lamp and provided with a gas passage communicating with the interior of the envelope **1** communicates with a circulating system which comprises a blower or compressor **3** for compressing the circulating gas, and a pair of valves **4** and **5**. The outlet of the compressor **3** opens to an inlet to a reaction tower **7** through a line **6**. The outlet of the reaction tower **7** is connected through a line **9** to an inlet to the following dehumidifying tower **8**. The outlet of the dehumidifying tower **8** is connetced to the supporting means **2** through a line **11** having a valve **10**. To the line **6** are connected a hydrogen gas container **12** containing hydrogen gas and a nitrogen gas container **13** containing nitrogen gas via valves **14** ad **15** respectively through a line **16**. To the line **5** between the supporting means **2** and the valve **4** is connected a vacuum pump **17** for finally sucking a purge gas out of the envelope through a line **19** having a valve **18**. To the line **11** between the supporting means **2** and the valve **10** is connected an argon gas container **20** for feeding argon gas to the interior of the envelope **1** finally evacuated through a line **22** having a valve **21**.

Into the interior of the envelope **1** of an incandescent lamp fitted to the supporting means **2**, in which an inert gas is to be filled, is introduced forcedly with pressure a purge gas consisting of a mixture of nitrogen and hydrogen gases from the nitrogen gas container **13** and hydrogen gas container **12** through the lines **16** and **11**, respectively. At this time the valve **21** for supplying argon gas, the valve **18** on the line **19** connected to the vacuum pump **17** and the valve **4** on the line **5** are all closed. When the interior pressure of the envelope **1** reaches a prescribed level, for example, 4 atmospheres due to the pressurized introduction of purge gas, the valves **14** and **15** are closed to stop the feeding of the purge gas. The envelope **1** is preferably heated so as to accelerate the release of gases, particularly water contained in the wall of the envelope. Next, the purge gas feed valve **10** is closed and the valve **4** on the discharge side is opened and the compressor is also set in operation. Thus the pressurized gas mixture in the envelope **1** is conducted through the line **5** into the compressor **3** and then through the packed tower **7** via the line **6**. While passing through the packed tower **7**, the purge gas containing oxygen and hydrogen is brought into contact with the catalyst filled therein, and the oxygen and hydrogen are reacted to form water. During transit through the dehumidifying tower **8**, the gas carrying said water is deprived of it. Since the gas is substantially free form oxygen and water, it can be reused in the subsequent purging cycle. The purging operation is repeated until the oxygen and water content of the envelope **1** falls to below the allowable limit. The second and following purging cycles do not always require the make up supply of nitrogen. Upon completion of purging, the valves **10** and **4** for supplying purge gas are closed, and after opening the valve **18**, the vacuum pump **17** is operated to draw purge gas out of the envelope **1**. After the purge gas has been completely discharged therefrom, the valve **21** is opened and argon gas is filled through the lines **22** and **10** into the envelope **1** at a prescribed pressure. Thereafter the exhaust tube of the envelope **1** is hermeticallly sealed by an ordinary process.

Preferably, hydrogen is added to the purge gas to be initially introduced into the envelope **1** in reaction equivalents to the oxygen originally contained therein. The volumes of gases present in the envelope **1** gradually decrease with the progress of purging at the approximately fixed rate, so that there will always be present in the envelope the reaction equivalents of hydrogen to the oxygen. A concrete example of a supporting means for the envelope is presented in FIG. **2**. The supporting means includes a vessel body **32** provided with a pair of pipes **30** and **31** for the introduction and release of purge gas respectively and a cap member **33** airtightly engageable with the body **32**. To the cap member **33** are fitted a pair of rod members **34** and **35** extending into the body **32**. To the ends of both rods are fitted elastic members **37** and **38** for supporting the envelope **36** of an electric lamp. On the cap member **33** are provided a pair of terminals **39** and **40**. Below the terminals there is suspended by lead wires **43** and **44** connected to the terminals a coiled heater for thermally sealing the exhaust tube **41** of the envelope. Where required, there may be provided a plurality of elastic members **37** and **38** and a heater **42** respectively. In such case it is possible to treat a plurality of envelopes at once. The aforementioned supporting means may be disposed between lines **10** and **5** in place of the supporting means **2** shown in FIG. 1 by connecting the pipe **30** to the line **10** and the pipe **31** to the line **5**.

FIG. 3 is a schematic flow diagram of a modification of a gas washing apparatus according to the present invention adapted for the filling of nitrogen gas in the bulb or envelope of an incandescent lamp. The same parts of FIG. 3 as those of FIG. 1 are denoted by the same reference numerals and description thereof is omitted. The apparatus of FIG. 3 is different from that of FIG. 1 in the following points:

(i) An argon gas holder **20** is omitted;

(ii) A line **16** and a means for feeding hydrogen and nitrogen gases connected thereto are omitted;

(iii) Between the dehumidifying tower **8** on the line **11** and the valve **10** is provided an air-introducing line **51** with a valve **50** whose outer end opens to the air; and

(iv) Between the valve **10** on the line **11** and the supporting means **2** is connected a hydrogen gas container **12** through a line **52** having a valve **14**.

The reaction tower utilized in this modification may be packed with platinum-palladium catalyst or copper catalyst.

In operating the apparatus of FIG. 3, the valves **4** and **10** are first closed, and the valve **14** is opened to introduce hydrogen with pressure through the line **52** into the envelope at the rate of at least 2 mols per mol of air present in the line defined by the valves **4** and **10**. Then the valve **4** is opened and the compressor **3** is operated to conduct a mixture of air and hydrogen to a tower **7** packed with a catalyst through the line **5**, compressor **3** and the line **6**. During transit through the packed tower **7** and dehumidifying tower **8**, the gaseous mixture is dehydrated, thus making the purge gas dry and oxygen-free as described above. This operation is repeated until the content of gaseous impurities, such as oxygen, hydrogen and water in the envelope **1** falls to below the allowable limit. Upon completion of purging, filling gas mainly consisting of nitrogen gas is filled into the envelope at a prescribed pressure and the exhaust tube of the envelope **1** is hermetically sealed by a known process capable of filling the gas which is free from oxygen hydrogen and water, without the necessity of providing a purified gas.

The foregoing embodiments relate to the filling of inert gas in the envelope of an incandenscent lamp. However, it will be apparent that the present invention is not limited

to such use, but more broadly applicable in replacing the gases of an envelope used for canning and other purposes. It will be understood that changes or modifications clearly known to those skilled in the art may be practised, insofar as the scope and spirit of the present invention is not departed.

What is claimed is:

1. A method of providing a container with an oxygen-free gas, comprising the sequential steps of:

(a) forcedly introducing under pressure a hydrogen-containing purge gas into a free oxygen-containing container to form a pressurized gas mixture;

(b) withdrawing the pressurized gas mixture from said container;

(c) reacting hydrogen and oxygen contained in the withdrawn gas mixture to form a reaction mixture containing water;

(d) removing water from said reaction mixture and recovering a water-free purge gas;

(e) recycling said water-free purge gas obtained in step (d) to step (a).

2. The method according to claim **1**, wherein the oxygen-free gas in step (a) contains at least 2 molar proportions of hydrogen per molar proportion of oxygen originally present in said container.

3. The method according to claim **1**, wherein the container is heated following step (a) and prior to step (b) to accelerate the release of gases therefrom.

4. The method according to claim **1**, wherein the container is exacuated by repeating steps (a) to (e) until oxygen and water contents in the container fall to below an allowable limit, and a prescribed volume of the resulting oxygen-free gas is charged to the container.

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EARL C. THOMAS, Primary Examiner

U.S. Cl. X.R.

23—209, 281; 99—189; 141—66; 316—21