

# United States Patent

[11] 3,598,384

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[73] Assignee **S.A.E.S. Getters S.p.A.**  
Milan, Italy  
[32] Priority **Sept. 13, 1968**  
[33] **Italy**  
[31] **21,185A/68**

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Publication: The Review of Scientific Instruments; Vol. 35, No. 6; June, 1964 (Eichenbaum et al.)

Primary Examiner—Gerald A. Dost  
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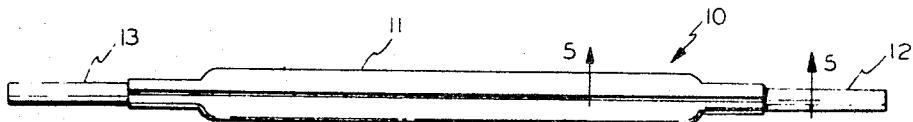
## [54] METAL VAPOR GENERATORS 10 Claims, 5 Drawing Figs.

[52] U.S. Cl. .... **266/39,**  
118/49, 219/275  
[51] Int. Cl. .... **C22b 61/00**  
[50] Field of Search. .... **266/1, 34**  
R, 34 V, 39; 118/48, 49, 49.1, 49.5; 219/271, 275;  
117/107, 107.2

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**ABSTRACT:** A particle free device for releasing a metal vapor comprising: a container having walls defining a chamber; an opening in at least one of said walls; a particulate, metal vapor releasing substance within the chamber; and a member within the chamber and adjacent to the opening whereby the opening is rendered permeable to metal vapor but impermeable to the particulate substance.



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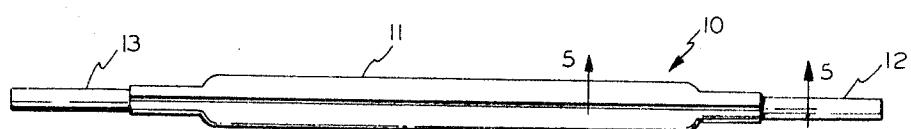


FIG. 1

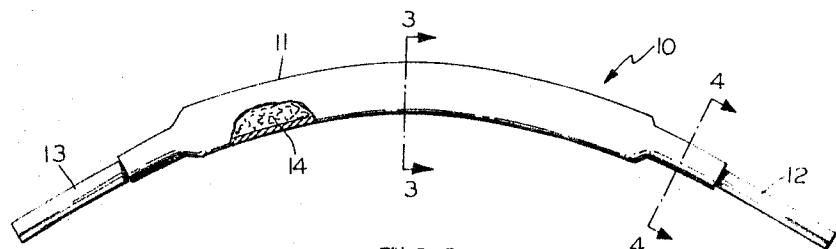


FIG. 2

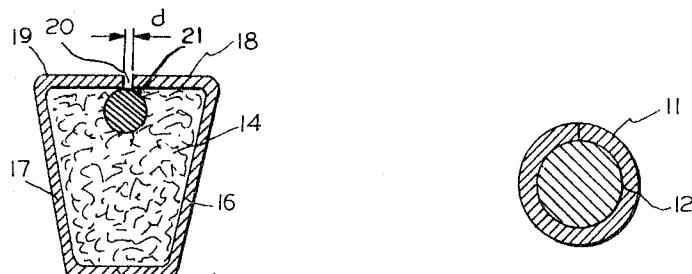


FIG. 3

FIG. 4

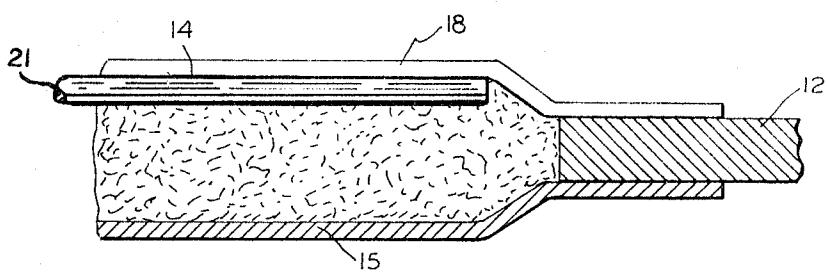


FIG. 5

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## METAL VAPOR GENERATORS

## DISCLOSURE

Devices which release metal vapor, such as those of mercury, cesium, potassium, or sodium, in vessels such as electron tubes, are well-known in the art as described, for example, in Italian U.S. Pat. No. 784,903, Italian compleutive application 21538A/67 of Nov. 12, 1967, and by Eichenbaum, et al., "Cesium Vapor Dispenser," The Review of Scientific Instruments, Vol. 35, No. 6, June 1964, pp. 691-693. Unfortunately, the above and other prior art generators suffer from a number of disadvantages. For example, they frequently release small particles before and/or after metal vapor release. These small particles are electrically conductive and can cause short circuits, damage photosensitive surfaces and cause other difficulties within the electron tubes. Another disadvantage is the undesirable absorption of water vapor and gasses from the atmosphere by that portion of the metal releasing substance within the device which comes in contact with atmospheric air. Another disadvantage is the nonuniform compression of the particulate metal vapor releasing substance within the device which generally occurs when the container of the generator is closed. The above and other disadvantages lead to poor reproducibility of results from one generator to another, causing numerable difficulties during production and use of the generators.

Accordingly, it is an object of the present invention to provide novel metal vapor generators which are substantially free of one or more of the disadvantages of the prior art. Another object is to provide metal vapor generators which are substantially free of loose particles before and/or after release of the metal vapor. A further object is to provide metal vapor generators having a reduced tendency to absorb water vapor and gases from the air. Another object is to provide metal vapor generators with a uniformly compressed particulate metal vapor releasing substance adjacent to the opening of the generator. Additional objects and advantages will be apparent to those skilled in the art by reference to the following detailed description and drawings wherein:

FIG. 1 is a plan view of a generator of the present invention;  
FIG. 2 is an elevation view of the generator of FIG. 1;

FIG. 3 is an enlarged sectional view taken along line 3-3 of FIG. 2;

FIG. 4 is an enlarged sectional view taken along line 4-4 of FIG. 2; and

FIG. 5 is an enlarged sectional view taken along line 5-5 of FIG. 1.

According to the present invention, there is provided a particle free device for releasing a metal vapor. This device comprises a container having continuous walls defining a chamber. The extremities of the walls are juxtaposed parallel to one another to form an opening. A particulate metal vapor releasing substance is within the chamber. Also within the chamber and adjacent to the opening is a member placed such that the opening is rendered permeable to metal vapor but impermeable to the particulate substance.

The container can have any convenient shape, but in one preferred embodiment of the present invention is made from a generally rectangular sheet of material, bent around its longer axis such that the longitudinal extremities of the sheet material are juxtaposed parallel to one another, and define an opening through which the metal vapor is released. In another preferred embodiment, the container is made of a material of high electrical resistance permitting activation of the metal vapor releasing substance by imposing an electrical potential across the ends of the container to heat the container.

The particulate metal vapor releasing substance within the chamber can be virtually any suitable material but is generally a mixture of a compound of the desired metal and a reducing agent for the compound. Reducible metallic compounds include, among others, cesium dichromate, or more preferably

cesium chromate, potassium chromate, and sodium chromate. Mercuric oxide can also be employed if mercury is the desired metal. Any suitable reducible agent such as those described in Eichenbaum or the Italian patent supra can be employed, examples of which include, among others, silicon and aluminum. When the desired metal is barium, any of the well known barium aluminum alloys can be employed. The metal vapor releasing substance is particulate in order to provide a large surface area for vaporization of the metal and/or chemical reaction. 10 However if the particles are too small they tend to escape from the device. Generally those which pass through a U.S. standard screen of 170 mesh/inch and are retained on a screen of 45 mesh/inch, i.e. those exhibiting a particle size of about 10 to 90 $\mu$ , are suitable. 15 The member within the chamber is located adjacent to the opening whereby the opening is rendered permeable to metal vapor, but impermeable to the particulate substance. According to the broadest aspects of the invention, the member can have any suitable geometric shape but is preferably elongated 20 having its axis parallel to the extremities of the walls or sheet. The cross section can be any regular or irregular polygon and, thus, can be triangular, square, or hexagonal but is preferably round since such a cross section gives the best seal against loose particles. In the most preferred embodiment of the present invention, the member within the chamber is a wire having a diameter greater than the distance between the extremities of the walls.

Referring now to the drawings and in particular FIGS. 1 and 2 thereof, there is shown a device 10 for generating metal vapors. The device 10 comprises a container or tube 11 having terminals 12 and 13 in each end thereof. The terminals 12 and 13 seal the ends of the tube 11 and together with the walls thereof, define a chamber containing the metal vapor releasing substance 14 which preferably fills the chamber. As shown in FIG. 3, the tube 11 is formed from a continuous sheet of material such that the tube 11 has a lower wall 15 connected to sidewalls 16 and 17 which in turn are connected to top walls 18 and 19. The edges of the sheet which is comprised of top walls 18 and 19 meet in juxtaposed relationship defining an opening 20 of width  $d$  which is generally 100 to 300 microns. Within the chamber containing the substance 14 is a member 21 which is preferably a wire of circular cross section. The wire 21 is held in place by the substance 14. As shown in FIG. 45, the diameter of the wire 21 is greater than the dimension  $d$ . As shown in FIG. 5, the axis of the wire 21 is parallel to the longitudinal edge of the top wall 18 and, therefore, also the top wall 19. By this arrangement, metal vapors produced by the substance 14 can pass by the wire 21 and out the opening 20, whereas loose particles in the substance 14 are kept within the chamber. Another advantage of this arrangement is the decreased tendency for the substance 14 to absorb water vapor and other gases from the air since the chamber is almost effectively sealed by the wire 21 minimizing entry of air into the chamber.

A common method of producing the device 10 is to compress the particulate substance 14 and form the walls 15, 16, 17, 18, and 19 into the general form of FIG. 3 but with the walls 16 and 17 angled outward at a greater angle than shown, thus making  $d$  much larger. The walls 16 and 17 are then pressed towards one another to reduce  $d$  to the desired value. However, in the absence of the wire 21, the compressed material 14 adjacent to the opening 20 fractures and produces loose particles.

In a nonlimiting example of operation, the device 10 is placed in a vessel such as an image intensifier tube in which it is desired to introduce cesium vapor. The terminals 12 and 13 are connected to a source of potential not shown, and the tube evacuated by any known means. Current is then caused to flow through the nichrome tube 11 by impressing a potential across the terminals 12 and 13 until the tube 11 is heated to about 1,000° K, whereupon the substance 14, which is a mixture of one part by weight of  $Cs_2CrO_4$  and two parts by weight of Si, releases cesium vapor. No loose particles are observed in the vessel either before or after cesium vapor release.

Although the invention has been described in considerable detail with reference to certain preferred embodiments thereof, it will be understood that variations and modifications can be effected within the spirit and scope of the invention as described above and as defined in the appended claims.

I claim:

1. A particle free device for releasing a metal vapor, said device comprising:
  - A. a container having continuous walls defining a chamber;
  - B. the extremities of the walls juxtaposed parallel to one another defining an opening;
  - C. a particulate metal vapor releasing substance within the chamber; and
  - D. a member within the chamber and adjacent to the opening whereby the opening is rendered permeable to metal vapor but impermeable to the particulate substance wherein the chamber is completely filled with the metal vapor releasing substance which holds the member in place.
2. The device of claim 1 wherein the container is a material of high electrical resistance.
3. The device of claim 1 wherein the metal vapor is that of an alkali metal.
4. The device of claim 1 wherein the metal vapor is that of mercury.
5. The device of claim 1 wherein the metal vapor is that of barium.
6. The device of claim 1 wherein the metal vapor releasing substance is a mixture of an alkali metal chromate and a reducing agent for the chromate.
7. The device of claim 1 wherein the member is a wire having a diameter greater than the transverse dimension of the opening.
8. A particle free device for releasing a metal vapor, said device comprising:
  - A. a container having continuous walls defining a chamber;

- B. the extremities of the walls juxtaposed parallel to one another defining an opening;
- C. a particulate metal vapor releasing substance within the chamber; and
- D. a wire within the chamber adjacent to the extremities of the walls, the wire having a diameter greater than the distance between the extremities of the walls wherein the chamber is completely filled with the metal vapor releasing substance which holds the wire in place.
9. A particle free device for releasing an alkali metal vapor, said device comprising:
  - A. a container having continuous walls of a rectangular sheet material of high electrical resistance defining a chamber;
  - B. the longitudinal extremities of the sheet material juxtaposed parallel to one another defining an opening;
  - C. a metal vapor releasing substance in the chamber, said substance comprising an alkali metal chromate and a reducing agent for the chromate; and
  - D. a wire within the chamber adjacent to the longitudinal extremities of the sheet material, the axis of the wire being parallel to the longitudinal extremities of the sheet, the diameter of the wire being greater than the distance between the extremities of the sheet wherein the chamber is completely filled with the metal vapor releasing substance which holds the wire in place.
10. A particle free device for releasing a metal vapor, said device comprising; a container having walls defining a chamber; an opening in at least one of said walls; a particulate, metal vapor releasing substance within the chamber; and a member within the chamber and adjacent to the opening whereby the opening is rendered permeable to metal vapor but impermeable to the particulate substance wherein the chamber is completely filled with the metal vapor releasing substance which holds the member in place.

UNITED STATES PATENT OFFICE  
CERTIFICATE OF CORRECTION

Patent No. 3,598,384 Dated Aug. 10, 1971

Inventor(s) Mario Zucchinelli

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Col. 1, line 10, delete "U.S."

Col. 1, line 20 delete "absorption", insert --sorption--

Col. 1, line 36 delete "absorb", insert --sorb--

Col. 1, line 45 delete "enlarge", insert --enlarged--

Col. 2, line 13 delete "45", insert --450--

Col. 2, line 21 delete "of", insert --or--

Col. 2, line 52 delete "absorb" insert --sorb--

Signed and sealed this 22nd day of February 1972.

(SEAL)

Attest:

EDWARD M. FLETCHER, JR.  
Attesting Officer

ROBERT GOTTSCHALK  
Commissioner of Patents