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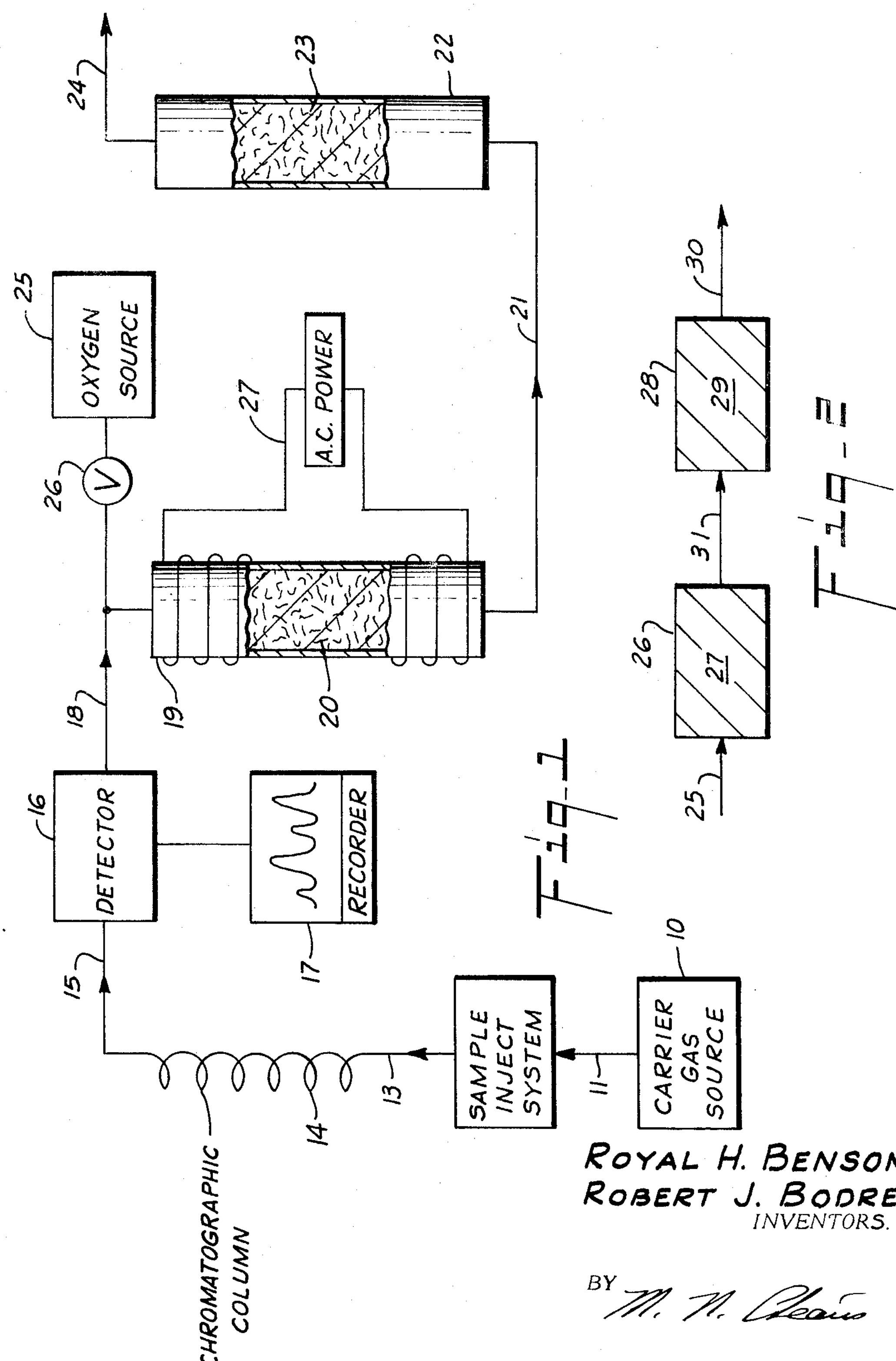

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SAFETY DEVICE FOR REMOVING TRITIUM GAS FROM A GAS STREAM

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ABSTRACT OF THE DISCLOSURE

Safety device for removing tritium gas from flowing fluid streams comprised of a means to convert the tritium in the stream to a labile-tritium-containing compound and a trapping means connected to the conversion means such that the fluid stream upon exiting from the conversion means flows through the trapping means, the trapping means comprising a means to prevent the escape of the labile-tritium-containing compound to atmosphere. In a specific embodiment, the invention is a safety device for use in combination with gas chromatographic detectors of the electron capture type which employ a tritiated material as a source of beta radiation wherein the converter means is connected to the outlet of the detector and the trapping means, which in the preferred embodiment takes the form of a tritium-exchange means, is connected to the outlet of the converter means such that the tritium in the labile-tritium-containing compound is exchanged with labile hydrogen in the tritium-exchange means.

BACKGROUND OF THE INVENTION

The present invention relates to a device for preventing radiation hazards caused by the accidental release of tritium to the atmosphere. More particularly, the present invention relates to a safety device for use with analytical instruments employing tritiated materials in the detector system.

In certain types of instrumentation employed in chemical analysis, radioactive materials are employed. For example, in the field of gas chromatography which is a widely employed analytical technique, certain detector systems contain a tritiated material as a radiation source. Two such detector systems are the electron capture detector and the argon ionization detector both of which are described in detail, as to construction and operation, in an article entitled "Ionization Methods for the Analysis of Gases and Vapors" by J. E. Lovelock which appeared in Anal. Chem., 33, 161 (1961). In particular the electron capture detector has been found to be particularly useful in the chromatographic analysis of halogenated materials, sulfur containing materials and other such materials containing atoms which will interact with or "capture" certain forms of radiation from a suitable source. Electron capture detectors and the like utilize very pure metallic foils which have been heated in a gaseous tritium atmosphere at very high temperatures for several hours or longer. Heating of the metallic foil at elevated temperatures in the tritium atmosphere causes some of the tritium to form metallic tritides. It is also believed that the metallic foil

contains tritium in other forms both physically and chemically bonded to the metal. Whatever the form of the tritium in the tritiated metallic foil, it is used as a source of beta radiation in electron capture detectors and the like. The most commonly employed tritiated metallic foil is tritiated titanium foil. In use, detectors employing the titanium tritide source are operated at high temperatures, generally in the range of from 100 to 200° C. Systems employing such detectors have safety switches built into the system which are designed to prevent overheating thereof. This is necessitated by the fact that titanium tritide has a decomposition temperature of around 225° C., and should this temperature be exceeded, dangerous levels of tritium could be released in the effluent from the chromatograph, which in most cases is vented to atmosphere. However, if the safety switches fail and the temperature of the detectors is allowed to exceed 225° C., there is no provision for preventing the tritium from escaping to atmosphere via the effluent from the chromatographic system.

Over and above the potential radiation hazard posed by the use of the detector systems described above, there are numerous other occasions when tritium gas may be accidentally released to atmosphere and create a serious health hazard. The use of tritium as a radioactive labeling material is widespread in industrial and educational laboratories. Such extensive use of tritium carries with it the probability that significant quantities may be accidentally released into a laboratory creating a health menace to the personnel therein. While the use of a venthood will serve to remove the released tritium from a room, laboratory, etc., it allows the tritium to go unchecked into the atmosphere which is quite undersirable from a health standpoint.

SUMMARY OF THE INVENTION

It is therefore an object of the present invention to provide a safety device to prevent radiation hazards caused by accidental release of tritium to atmosphere.

Another object of the present invention is to provide an improved analysis system particularly for use in gas chromatography.

Still another object of the present invention is to provide a safety device for use with analytical instruments employing certain radiation sources.

Another object of the present invention is to provide a safety device for use with electron capture detectors and the like which employ a metallic tritide as a radiation source.

These and other objects of the present invention will become apparent from the description given herein, the drawings and the appended claims.

In its broadest aspect, the invention contemplates the removal of tritium gas from a flowing fluid stream using a conversion means through which the fluid stream is passed, said conversion means being a means to convert tritium gas to a labile-tritium-containing compound and a trapping means connected to the conversion means such that the fluid stream from the conversion means flows through the trapping means, the trapping means comprising a means to prevent the escape of the labile-tritium-containing compound to atmosphere. Generally, the trapping means takes the form of a tritium-exchange means whereby the labile tritium in the labile-tritium-containing compound produced in the conversion means exchanges for labile hydrogen in the tritium-exchange means.

The invention as applied to certain analytical set-ups is best exemplified by a gas chromatographic system. In such a system, a sample mixture containing generally several components is injected into a separatory column whose function it is to resolve the individual components of the sample mixture. Each component then elutes from the separatory column individually and is detected by some suitable means. A carrier gas is employed which acts as the driving force to carry the sample mixture and the components through the separatory column and the detector. Upon exiting from the detector, the flow which is comprised of carrier gas and the separated component is generally allowed to vent to atmosphere. As explained above, if the detector happens to be an electron capture detector or such employing tritium tritide or some other metallic tritide, it is conceivable, due to failure of the safety devices provided with the instruments, that the effluent from the detector would contain significant amounts of tritium which would be vented to atmosphere which in most cases would be in a laboratory. The improvement proposed herein which would render the above-described system free from such a radiation hazard comprises the above-described conversion means connected to the outlet of said detector which serves the purpose of converting the tritium gas to a labile-tritium-containing material and a tritium trapping means connected to the outlet of the conversion means in which the tritiated material produced in the conversion means is prevented from escaping to atmosphere.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a flow diagram of a typical gas chromatographic system showing a specific embodiment of the safety device of the present invention.

FIG. 2 is a block diagram of the safety device of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Reference is now made to FIG. 2 for a general description of the safety device of the present invention. A flowing fluid stream in line 25 enters housing 26 containing means 27 which converts any tritium gas present in the fluid stream entering from line 25, to a labile-tritium-containing compound. The fluid stream, now containing any labile-tritium-containing compound, leaves housing 26 via line 31 and enters housing 28 containing trapping means 29 which traps the labile-tritium-containing compound so as to prevent its release to atmosphere via line 30.

The flowing fluid stream in line 25 can come from enumerable sources. For example, line 25 could be the outlet of a venthood used to pull vapors from a laboratory. Also, the flowing fluid stream in line 25 could be the outlet of an air purification system wherein stale air is brought in, passed through charcoal beds and the like to remove odors and then vented back into the room. In general, the safety device of the present invention can be utilized in any situation where there exists a flowing fluid stream which can be diverted through the conversion means and the trapping means constituting the present invention. So long as the flowing fluid stream containing the tritium gas passes through the conversion means and the trapping means, the ultimate release of tritium to the atmosphere will be prevented.

Reference is now made to FIG. 1 for a description of the use of the safety device of the present invention in an analysis system. While the safety device of the present invention is shown as being used with a gas chromatographic system, it is to be understood that it can be used equally well with other instruments which employ tritiated materials as part of the detector or other systems and wherein there exists the possibility that tritium gas may escape to atmosphere. Referring then to FIG. 2, a carrier gas source is connected via line 11 to sample inject system 12. Sample inject system 12 generally comprises a heated block or other such heating source wherein when a liquid sample is injected, it is instantaneously vaporized. Sample inject system 12 is connected via line 13 to chromatographic column 14 which may be any well known means commonly employed in gas chromatography for separating components of a sample. Column 14 is connected via line 15 to detector 16 which in this case, is an electron capture detector employing a titanium tritide foil as part of the detector system. A recorder 17 or other such readout device is responsively connected to detector 16. In most typical chromatographic systems the carrier gas flow upon exiting from detector 16 is allowed to escape to atmosphere. In the system shown in FIG. 1, however, detector 16 is shown connected via line 18 to column 19, which as shown, is a tubular housing packed with a substance 20 which is capable of catalyzing the reaction of tritium to labile-tritium-containing compound. Column 19 in turn is connected via line 21 to column 22 also a tubular housing but packed with a substance 23 which can trap or hold the labile-tritium-containing compound either chemically or physically or contains exchangeable or labile-hydrogen atoms. Column 22 is vented to atmosphere via line 24. There is also provided in the system a source of oxygen or air 25 which depending on whether or not valve 26 is open or closed can be fed into line 18 and thus flow through column 19. Column 19 is also provided with heating source 27 to heat substance 20 if desired.

In operation, a sample is injected into the system through sample inject system 12 and is swept through separating column 14 by means of a carrier gas coming from source 10. The components of the sample upon being separated in column 14 pass to detector 16 to be individually detected. The separated components of the sample mixture plus the carrier gas then flow through columns 19 and 22 ultimately venting to atmosphere through line 24. So long as detector 16 operates properly and does not overheat, the safety device of the present invention comprised of columns 19 and 22 has no effect on the system. However, should for some reason there be a failure of the safety switches which serve to limit the upper temperature of the titanium tritide foil used in detector 16 such that the temperature reaches about 225° C., or higher, the safety device prevents the release of tritium from the system. As can be seen, any tritium gas released from detector 16 will be swept by the carrier gas out of detector 16 and into line 18 and then into the safety device consisting of columns 19 and 22. For example, substance 20 in column 19 could be a metallic oxide such as copper oxide and substance 23 in column 22 could be a labile-hydrogen-containing material such as hydrated calcium sulfate. In this case, the tritium upon entering column 19 would be converted to water by the reaction of the tritium with the copper oxide. The tritiated water thus produced would leave column 19, flow through line 21 and enter column 22. Upon entering column 22, the tritium in the tritiated water would exchange with the labile-hydrogen atoms present in the hydrated calcium sulfate such that when the sample exited column 22 through line 24, the level of tritium in the water would be at a harmless level insofar as posing a radiation hazard is concerned.

The conversion means chosen for use in the safety device of the present invention can be any means which is capable of converting tritium gas to a labile-tritium-containing compound. For example, the conversion means can be a combustion chamber wherein the tritium is burned with oxygen to produce water. More conveniently, however, the conversion means will be a housing having an inlet and an outlet and containing a reactive or catalytic material capable of converting tritium to a labile-tritium-containing compound. The catalytic material can be a substance capable of converting the tritium to tritiated ammonia or some other nitrogen-containing compound in which the tritium thereof is of the labile form. Preferably, however, the conversion means will comprise a housing containing a catalyst capable of converting the tritium to an oxygenated material and especially to tritiated water. Suitable catalytic materials for converting tritium to tritiated water include the metal oxides, particularly those of Groups VIII and I–B of the Periodic Table. Also suitable as catalytic materials for converting tritium to tritiated water include metal catalysts such as the precious metals of Groups VI, VIII and I–B of the Periodic Table. When a precious metal catalyst is employed, it is necessary to provide the conversion means with a source of oxygen which can either be air or pure oxygen. It is also preferable, when a catalyst is employed to convert the tritium into a labile-tritium-containing compound, that the conversion means be equipped with a heating means to facilitate the reaction of the tritium and the catalytic or reactive material. The heating means can be any known method to heat a housing containing a catalytic material. More generally, the heating means will take the form of a hearing tape or Nichrome resistance wire wound around the housing and connected to a suitable source of electrical power.

The amount of the catalytic or reactive material employed in the conversion means will of course depend on the amount of tritium present in the tritiated material used as the radiation source or the amount of tritium being handled in the laboratory, hood, etc. Once the amount of tritium is known, the conversion means need only have enough catalytic material to convert all of the tritium passing therethrough to a labile-tritium-containing compound. Since the conversion means will be one to quantitatively convert the tritium to a labile-tritium-containing compound, the amount of catalytic material in the conversion means can be quite accurately ascertained.

When the conversion means comprises a housing containing a catalytic substance, the particular shape of the housing is not critical. However, preferably, because of ease of assembly and availability, a tubular member containing the catalytic material is generally chosen particularly when the safety device is used with analytical instruments such as gas chromatographs. A tubular member offers the additional advantage that it can be uniformly and easily heated by simply winding the heating tape or Nichrome resistant wire around it externally. The size of the tubular member will, of course, be dictated by the amount of the catalytic substance chosen which as explained above is determined by the amount of tritium in the tritiated-radiation source or which is being handled.

The trapping means employed in the safety device of the present invention can be any means by which a labile-tritium-containing compound is prevented from escaping therefrom. For example, the trapping means can comprise a housing having an inlet and an outlet and containing a substance which either absorbs or adsorbs the labile-tritium-containing compound thereby preventing its passage completely therethrough. Such a "trapping substance" can be any one of the numerous minerals, chemical compounds, synthetic materials, etc., which have the capability of either physically or chemically retaining labile-tritium-containing compounds. Trapping substances include such materials as activated aluminum, activated silica gel, certain types of molecular sieves, anhydrous calcium chloride, anhydrous calcium sulfate, and numerous other substances which exhibit the capability of picking up and retaining labile-tritium-containing compounds. In general, any material having so-called active sites on the surface which permit chemical or physical bonding of labile tritium thereto can be used. Alternately, the trapping substance can be a liquid which will absorb the labile-tritium-containing compound or perhaps react chemically therewith. Liquids have certain disadvantages in handling and present certain other problems, discussed more fully below, particularly when employed with analytical devices such as gas chromatographs.

Preferably, the trapping means will comprise a tritium-exchange means which is capable of converting the labile-tritium-containing compound produced in the conversion means to a labile-hydrogen-containing compound, i.e., one which is capable of exchanging hydrogen for the tritium. More generally, the tritium exchange means will take the form of a suitable housing with an outlet and inlet and containing an "exchange substance," which contains exchangeable or labile-hydrogen atoms. Virtually any material containing any labile-hydrogen atoms can be employed as the exchanging substance in the tritium-exchange means. It is preferred, however, that the substance chosen for use in the tritium-exchange means containing greater than 0.1% by weight of labile-hydrogen atoms. Obviously, the greater the concentration of labile-hydrogen atoms in the exchanging substance, the less of same which must be used in the tritium-exchange means, and the more efficient is the exchange process.

The exchanging substance used in the tritium-exchange means can be either a liquid or a solid. The use of a liquid with analytical systems such as gas chromatographs suffers from several disadvantages one of which is that the effluent from the conversion means must usually be scrubbed through the liquid to effect good exchange. This can result in excessive back pressure to the carrier gas flow and cause tailing or other undesirable effects on the chromatographic technique being employed. Obviously, when the safety device is used with a vent hood, air purifier, or the like, this back pressure effect is of no consequence. However, unless the liquid has an extremely low vapor pressure, it will vent tritium-containing material into the atmosphere. Nonetheless, if desired, a liquid may be employed in the tritium-exchange means. Obviously, any number of liquids qualify for use in the tritium-exchange means, including such compounds as alcohols, acids, amines, water and any other of the well-known labile-hydrogen-containing liquids.

For all around ease of operation and convenience, it is preferable that the material used as the exchanging substance in the tritium-exchange means and which contains the labile-hydrogen atoms be a solid in either particulated or fibrous form. The use of particulated or fibrous solids makes it relatively easy to prepare a packed column out of a tubular member which offers minimum resistance to carrier gas flow, thus minimizing any possible back pressure effect and which offers maximum contact between the tritium-containing compound produced in the conversion means and the labile-hydrogen-containing substance, i.e., the exchanging substance. The term particulated as used herein, refers to solid pieces having an average size ranging from 325 mesh up to chunks having an average diameter of ½ inch. The shape of the solid particles is not critical. When a solid is employed as the substance in the tritium-exchange means, it can be any one of numerous solid substances containing hydroxyl groups, amine groups, amide groups or other such labile-hydrogen-containing groups. Examples include alumina, silica, silica gel, clays such as kaolin, diatomaceous earth, molecular sieves, hydrated salts such as hydrated calcium sulfate, hydrated calcium chloride, polymeric aromatic amines, polymeric aromatic amides, and numerous other solid materials having like characteristics. In general, any solid material containing labile-hydrogen atoms is a suitable substance for use in the tritium-exchange means of the present invention.

Preferably, the amount of the substance in the tritium exchange means should be ten times or greater the amount by weight of tritium (1) contained in the tritiated radiation source, or (2) being handled and potentially releasable to be picked up by the safety device herein.

While smaller amounts can be employed if desired, the use of a ten-fold excess results in maximum safety.

Whereas the temperature of the conversion means is generally raised to facilitate the conversion of the tritium to a labile-tritium-containing compound, it is preferred that the temperature of the tritium-exchange means be maintained at or near ambient to minimize the possibility of "stripping off" the labile-hydrogen atoms present in the substance as water or the like. However, higher temperatures can be employed in the tritium-exchange means, if desired, the kinetics of the exchange obviously being favored by higher temperatures.

Pressures and flow rates in both the conversion means and the trapping means, when the safety device is employed with a gas chromatographic system, will be dependent upon the carrier gas flow rate and the particular system to which the safety device is attached. Flow rates in typical gas chromatographic systems generally range from 1 ml. per minute up to 200 mls. per minute.

The use of the safety device of the present invention will prevent the immediate escape of harmful quantities of tritium gas to the atmosphere. For example, in the case of the device with a gas chromatographic system, once it has been determined that the safety switches protecting the detector employing the tritiated-radiation source have failed, and that the temperature of the detector has been raised to a level sufficient to release tritium gas, the operator of the system will be apprised of the fact that the safety device now contains, in the tritium-exchange portion, a potentially hazardous amount of tritium. At this point, the tritium-exchange means is removed and the tritium contained therein disposed of in a suitable manner. This procedure is necessitated by the fact that, although the tritium is initially exchanged and held in the tritium-exchange means, if allowed to remain, it will generally re-exchange with any labile-hydrogen compounds which may be passing through the system and would eventually be eluted off. Thus, the use of the safety device of the present invention with an analysis system such as a gas chromatographic system provides a method of preventing immediate and uncontrolled release of excessive amounts of tritium to the atmosphere.

As previously indicated, the safety device of the present invention is not limited to usage with instruments or analysis systems containing detectors which employ tritiated materials. A suitable safety device incorporating the conversion means and trapping means can be used on the vent of laboratory hoods, exhausts of room air purifiers, or any other system where there exists a flowing fluid stream which can be diverted through the conversion means and then the trapping means.

While as indicated above, the safety device of the present invention is useful with any system wherein tritium is being handled and where the tritium is picked up in a flowing fluid stream or a tritiated-radiation source is being contacted by a carrier gas, and where consequently there exists the possibility that harmful amounts of tritium could escape to the atmosphere, the safety device finds particular use with gas chromatographic systems employing electron capture detectors, argon ionization detectors and the like. In such detectors, as pointed out, a metallic tritide is employed as the radiation source. The metallic tritide is generally used in the form of a metallic foil. Most such detectors employ, as the radiation source, titanium tritide, or at any rate, titanium foil which has been treated with tritium.

What is claimed is:

1. A safety device for the removal of tritium gas from a flowing fluid stream comprising:

a conversion means for flowing said fluid stream through, said conversion means comprising a means to convert tritium gas to a labile-tritium-containing compound; and

a trapping means connected to said conversion means whereby the fluid stream from said conversion means flows through said trapping means, said trapping means comprising means to prevent the escape of said labile-tritium-containing compound to atmosphere.

2. The safety device of claim **1** wherein said trapping means comprises a tritium-exchange means, said tritium-exchange means comprising means to exchange the labile-tritium in said labile-tritium-containing compound for labile-hydrogen.

3. The safety device of claim **1** wherein said conversion means comprises a housing having an inlet and an outlet, said housing containing a catalyst for converting tritium to tritiated water and there is provided means to heat said housing.

4. The safety device of claim **3** wherein said catalyst comprises a metal oxide.

5. The safety device of claim **3** wherein said catalyst comprises a precious metal and there are means provided to introduce a source of oxygen to said conversion means.

6. The safety device of claim **2** wherein said tritium exchange means comprises a housing having an inlet and an outlet, said housing containing a substance having labile-hydrogen atoms, said substance being present in an amount to effect substantially complete exchange of the labile-tritium in said tritium-containing compound with the labile-hydrogen in the labile-hydrogen-containing substance.

7. The safety device of claim **6** wherein said labile-hydrogen-containing substance has greater than 0.1% by weight of labile-hydrogen atoms.

8. The safety device of claim **7** wherein said substance is a particulated solid.

9. The safety device of claim **7** wherein said substance is a fibrous solid.

10. In an apparatus having a detector system employing a tritiated material as a source of radiation, and means to flow a carrier fluid through said detector system whereby said tritiated material is exposed to said carrier fluid, a safety device comprising:

a conversion means connected downflow from said detector system whereby the effluent from said detector system flows through said conversion means, said conversion means comprising means to convert tritium gas to a labile-tritium-containing compound; and

a trapping means connected downflow from said conversion means whereby the effluent from said conversion means flows through said trapping means, said trapping means comprising means to prevent the escape of said labile-tritium-containing compound to atmosphere.

11. The safety device of claim **10** wherein said trapping means comprises a tritium-exchange means, said tritium-exchange means comprising means to exchange the labile-tritium in said labile-tritium-containing compound for labile-hydrogen.

12. The safety device of claim **10** wherein said conversion means comprises a housing having an inlet and an outlet, said housing containing a catalyst for converting tritium to tritiated water.

13. The safety device of claim **12** wherein said housing comprises a tubular member and there are means provided to heat said tubular member.

14. The safety device of claim **13** wherein said catalyst comprises a metal oxide.

15. The safety device of claim **13** wherein said catalyst comprises a precious metal and there are means provided to introduce a source of oxygen to said conversion means.

16. The safety device of claim **11** wherein said tritium-exchange means comprises a housing having an inlet and an outlet, said housing containing a substance having labile-hydrogen atoms, said substance being present in an amount to effect substantially complete exchange of the labile-tritium in said tritium-containing compound with the labile-hydrogen in the labile-hydrogen-containing substance.

17. The safety device of claim **16** wherein said labile-hydrogen-containing substance has greater than 0.1% by weight of labile-hydrogen atoms.

18. The safety device of claim **17** wherein said substance is a particulated solid.

19. The safety device of claim **17** wherein said substance is a fibrous solid.

20. The safety device of claim **16** wherein said housing comprises a tubular member.

21. The safety device of claim **10** wherein said apparatus comprises a gas chromatograph.

22. The safety device of claim **10** wherein said tritiated material comprises a metallic tritide.

23. The safety device of claim **22** wherein said metallic tritide is titanium tritide.

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