Jan. 2, 1968 N. L. GREGORY 3,361,907

APPARATUS FOR GAS ANALYSIS HAVING VOLTAGE MEANS TO ACCELERATE ELECTRONS FROM AN IONIZATION CHAMBER TO A DETECTION CHAMBER

Filed Dec. 2, 1963

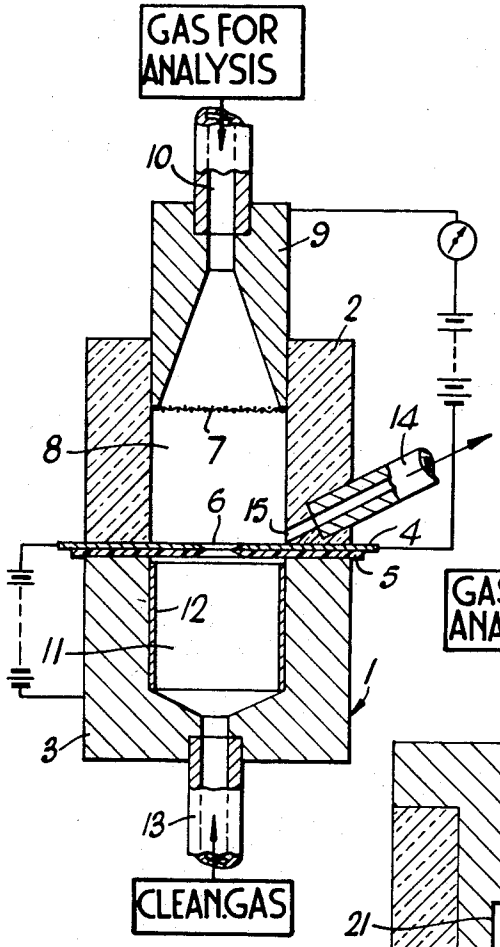


FIG. 1.

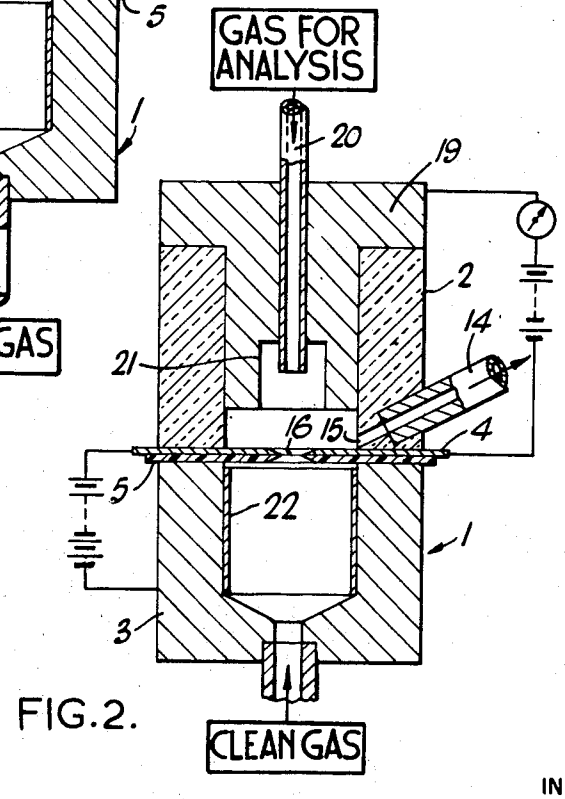


FIG. 2.

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United States Patent Office 3,361,907
Patented Jan. 2, 1968

3,361,907

APPARATUS FOR GAS ANALYSIS HAVING VOLTAGE MEANS TO ACCELERATE ELECTRONS FROM AN IONIZATION CHAMBER TO A DETECTION CHAMBER

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Filed Dec. 2, 1963, Ser. No. 327,211
Claims priority, application Great Britain, Dec. 6, 1962, 46,167/62
5 Claims. (Cl. 250—43.5)

This invention relates to gas or vapour detectors comprising ionising means, and more particularly, though not solely, to such detectors for use in chromatographic and like processes.

In accordance with the invention, the body of the detector is provided with a chamber or the like for the radio-active ionising means, which is separated from a detector chamber or the like in such a way as, in operation, to enable ionising means within the first said chamber to be maintained substantially isolated from gas or vapour components entering the detecting chamber. Thus the body of the detector may comprise two chambers separated by one or more orifices through which "scavenging" gas or vapours can be directed substantially to prevent ingress of these components to the ionising means, while permitting entry of ionisation inducing means into the detecting chamber.

A detector in accordance with the invention may be of the electron-capture type, the electron mobility type, or of that type known as the argon detector type in which case the ionisation inducing means may be free electrons: additionally or alternatively in the argon detector type the ionisation inducing means may be long-lived, excited-state molecules.

Moreover the detector body may be adapted to receive alternative parts to render it suitable for use either as an electron-capture detector or as an argon detector at will, thus providing a multi-purpose device. In order that the invention may be more clearly understood, reference will now be made by way of example, to the accompanying drawing which illustrates in the two somewhat diagrammatic figures, one embodiment of the invention in a detector which may be adapted for use as such a multi-purpose device.

As shown in FIGURE 1 of the drawing, an electron-capture detector comprises a cylindrical body 1 formed of a part 3 of electrically conductive material, such as brass, and a part 2 of insulating material, such as polytetrafluoroethylene, the two parts being separated by a cathode 4, the latter being insulated from the part 3 by a sheet 5 of insulating material, such as polytetrafluoroethylene. The parts will be secured together by suitably insulated through-bolts (not shown). The cathode 4 is formed with a central orifice at 6, the insulator 5 being formed with a matching orifice. A gauze anode 7 for the detecting chamber 8 in part 2 of the body, is carried on an insert 9 which may be of insulating material, or of brass, secured to the body 1 by suitable means, electrical connection to the anode 7 being provided in either case. A potential difference of up to, say 100 volts, is applied between the anode 7 and cathode 4. The insert is formed with a passage, a continuation of the tube 10, for leading the mixture containing the component(s) for detection into the detector.

The part 3 of the detector body is formed with a chamber 11 for the ionising means, which is a radio-active body. It may for example contain the isotope Radium 226 in the usual form of a sandwich between gold or silver foils; it is shown here in the form of a cylinder 12.

By means of a tube 13, a clean inert gas for example

nitrogen, can be introduced into the chamber 11 in order to provide a flow of gas through the orifice 6, as a result of which only an insignificant amount, if any, of the contents of the detecting chamber can pass into the chamber 11. This gas will be necessarily clean in that it should be free from entrainments which may be likely to provide adulterating ionisation in the detecting chamber. A further pipe 14 in gas-tight connection in the body of the device, leads to a hole 15 located towards the inner end of the detecting chamber 8, and this provides an outlet passage for the contents of the detecting chamber, which will include the clean gas flowing through the orifice 6.

Under the influence of an electric field arising from the application of a suitable potential difference between the cathode 4 and the part 3 of the body (comparable, for example, to that between cathode 4 and anode 7), free electrons generated by the ionising action of the isotopic source 12 will be directed towards the cathode 4 and, consequently, there will be a passage of electrons through the hole 6 into the detecting chamber 8. Depending upon the potential difference applied between the electrodes in the detecting chamber, molecules of an appropriate gas or vapour component, when introduced into the detecting chamber, will capture some of these electrons and ionisation will occur; thus a change of electron current through the detecting chamber upon admission of the gas or vapour component, in the manner normal with an electron-capture detector device, will indicate the presence of the component. By suitably following changes in electron current with different applied potentials, or by correlation of the reduction of current with that resulting from the presence of a known concentration of the component, in certain cases, the gas or vapour may be identified in ways known to those skilled in the art.

It will be noted that the cathode 4 and the insulator 5 are bevelled at the orifices. This is to minimise the effect of collision between the free electrons produced in the source chamber and the walls of the orifices, but the orifices would probably not be bevelled, in any case, if very thin cathode and insulator materials could be used. The size of the orifice will be determined by such considerations as electric field penetration to ensure that transfer of electrons is efficient, while still minimising entry of component gases or vapours from the detecting chamber into the other chamber, as referred to above. The size of orifice may also depend upon the type of ionising means used.

The detector is particularly useful for the detection of different components of a sample after passing through a separating column of either a packed form or the capillary type, and in that case the gas supplied from the tube 13 to maintain the ionisation chamber free from the contents of the detecting chamber will preferably be the same as the carrier gas used to carry the sample through the separating column.

Making use of the main parts of the detector but removing the insert 9, it is possible, by providing another insert 19 as shown in FIGURE 2 of the drawing, to adapt the detector for use as an argon detector type of device. The insert includes a tube 20, for the introduction of components for detection into the detecting chamber. This inlet tube, which is electrically conducting and forms the anode in the detecting chamber, terminates within a pocket 21 formed in the insert 19 as is usual with this type of detector.

In use, the radio-active source 22 can be identical with source 12, but the difference betwen the detector in this form and that of the form in FIGURE 1, is that the gas used will be argon, helium, or other gas from which may be derived long-lived, excited state, molecules, and the ionisation inducing means passing through the orifice 16, into the detecting chamber, may include metastable

molecules, as well as electrons, the metastables effecting ionisation by collision with the molecules of the component(s) for detection. Alternatively, the electric field in the source chamber may be arranged to be such that metastables are produced in quantity, in which case the metastables may predominate over, or take the place of, electrons as the ionisation inducing means passing into the detecting chamber. The argon detector form of this embodiment of the invention may be used in the same way as known argon detectors, with the advantage, which also applies to the form of the detector, as shown in FIGURE 1, that it may be used at elevated temperatures without risk of radio-active leakage, since, as distinct from usage of the known detectors, only clean gas comes into contact with the radio-active source and the likelihood of radio-active particles being liberated from the source is remote; moreover the source itself is protected from deterioration from oxidation, corrosion or hydrogen exchange reactions which may result from the action of components entering the detecting chamber in the known types of detector. When the detector is used in conjunction with a chromatographic column, it sometimes happens that certain stationary phases "bleed" from the column and the present proposal prevents the deposition of films of such phases on the ionising means. One yet further advantage is that the primary generation of the ionisation inducing means takes place outside the detecting chamber and therefore out of the presence of components being detected. Additionally, in the electron-capture type of detector, secondary effects, such as peak reversal increases of current, are minimised by the use of the invention.

An additional advantage accruing from use of the invention, particularly in the electron-capture detector, is that the ionising means may be a radio-active material of any kind capable of producing the required number of free electrons or metastable molecules, because the source can be sufficiently well screened from the detecting chamber to make the ionisation current resulting from direct ionisation in the detecting chamber by primary particles from the source very much less than the current due to flow of electrons through the orifice separating the two chambers. This latter consideration will be yet another criterion in the determination of the size of orifice between the chambers.

It is not intended that the invention should be limited in scope to the particular design of detector described; it is possible that the inlet tubes for component gases and "scavenging" need not be in line, nor need the radio-active source be of the cylindrical form illustrated. It may be found, however, to be necessary to provide some form of filtration in the outlet tube **14** so that, even if radio-active products of decomposition are present, they are not entrained into the atmosphere, but this will be a matter of normal precautionary measures being taken.

I claim:

1. Apparatus for detecting a gaseous component in a mixture with at least one other gaseous component comprising a detector enclosure and an auxiliary enclosure which communicates with the detector enclosure, inlet and outlet means for the detector enclosure whereby a gaseous stream may be passed through the detector enclosure without passing through the auxiliary enclosure, further inlet means for the auxiliary enclosure whereby a further gaseous stream may be passed into the auxiliary enclosure and thence into the detector enclosure, detector means for responding to the presence of said gaseous component in the detector enclosure, and a radio-active source disposed within the auxiliary enclosure, and including a means for establishing an electric potential between said detector enclosure and said auxiliary enclosure for accelerating electrons from the auxiliary enclosure to the detector enclosure.

2. Apparatus according to claim **1** wherein said detecting means includes a first voltage means for inducing a voltage drop across the detector enclosure.

3. Apparatus for detecting a gaseous component in a mixture with at least one other gaseous component, comprising in combination a first enclosure for ionization and detection of said gaseous component therein, inlet and outlet means to the first enclosure for passage of the gaseous mixture therethrough, a first electrode in the first enclosure, a second enclosure housing a radio-active source and having a wall of electrically conductive material, inlet means to the second enclosure for introduction of scavenging gas thereto, and a second electrode separating the two enclosures and electrically insulated from said wall and from the first electrode, the second electrode being apertured for passage into the first enclosure of both the scavenging gas and ionization inducing particles produced by the action of said source and means for maintaining the second electrode at a positive potential with respect to the said wall for accelerating electrons from the second enclosure to the first enclosure.

4. Apparatus according to claim **3**, in which the first electrode is porous and is disposed adjacent the inlet means for the detector enclosure for passage therethrough of the gaseous mixture.

5. Apparatus according to claim **3**, in combination with means for connection between the first and second electrodes for indicating changes of ionization current produced in the first enclosure.

References Cited

UNITED STATES PATENTS

3,009,098	11/1961	Simons	250—83.6
3,087,113	4/1963	Foster	250— 83.6 X
3,134,898	5/1964	Burnell et al.	250—43.5
3,247,375	4/1966	Lovelock	250—83.6 X

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