May 6, 1958 D. HARVEY 2,833,151

DEVICE FOR DELIVERING MEASURED QUANTITIES OF GASES OR VAPOURS

Filed May 11, 1955

FIG. 1

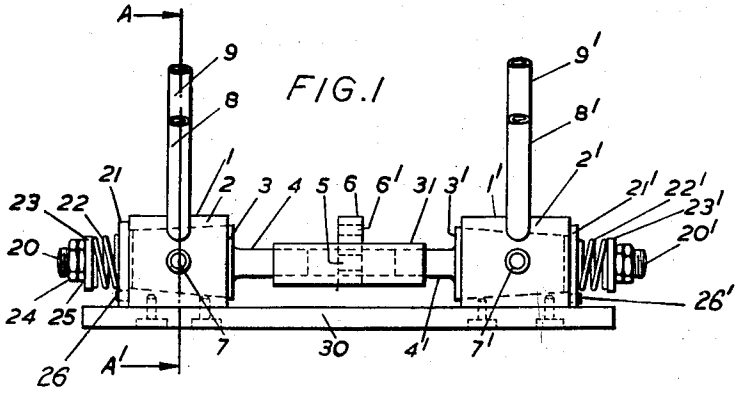


FIG. 2.

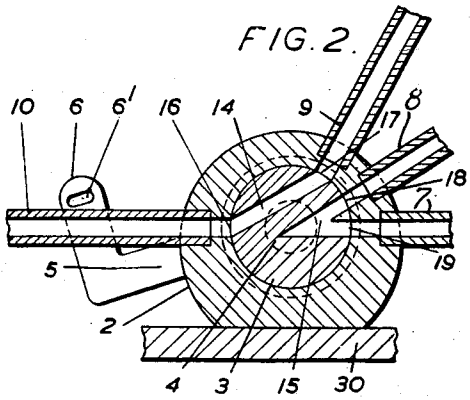


FIG. 6.

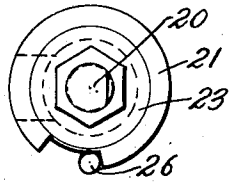


FIG. 3

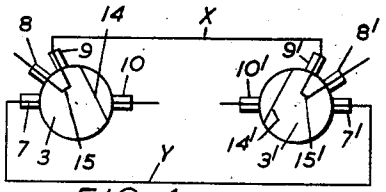


FIG. 4.

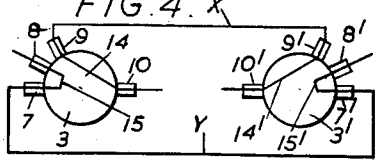
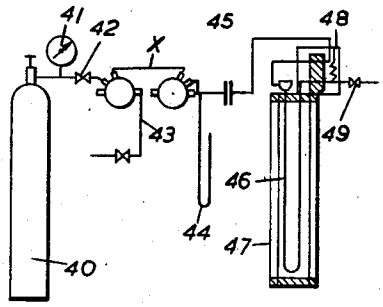


FIG. 5.



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DEVICE FOR DELIVERING MEASURED QUANTITIES OF GASES OR VAPOURS

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6 Claims. (Cl. 73—422)

This invention relates to methods of providing or supplying accurate volumes, especially small volumes, of gases for various uses.

It is commonly required to be able to provide accurately measured quantities of gases, for example in gas analysis. More recently the same problem has arisen in vapour phase chromatography. Previously it has been customary to employ glass measuring vessels or pipettes with associated glass stop cocks for the purpose, but these suffer from the disadvantages of fragility, that the fabrication of multi-way cocks is time consuming and difficult, and hence the number of cocks required for a given assembly is increased.

According to the present invention there is provided an apparatus adapted for measuring and delivering at controlled time intervals an accurate volume of gas and suitable for mechanical operation which comprises two valves each having a fixed metal body formed with an interior frusto-conical hole, preferably tapering outwardly, and situated in the said hole a co-operating closely fitting rotatable metal valve plug; a metal spindle rigidly connecting said valve plugs provided with means adapted for rotating the spindle and valve plugs; four metal tubes radiating from the body of each valve and in free connection with the frusto-conical hole therein; the first tube of the first valve being in connection with the supply of gas being measured, the second forming one limb of a pipette, the third being in connection with a supply of inert gas under pressure, the fourth being in connection with the fourth tube of the second valve; the first tube of the second valve being open to atmosphere, the second forming the other limb of the pipette, the third being in connection with the apparatus to which the gas is being supplied, the fourth being in connection with the fourth tube of the first valve; and situated in each of said valve plugs a first transverse duct and a second separate V-shaped duct or roughly sector shaped recess, each having ports in the surface of the valve plug: the area of the ports and the circumferential distance between them being such that in a first configuration the first duct is in communication between the first and second tubes when the second duct or recess is in communication between the third and fourth tubes; and that in a second configuration the second duct is in communication between the second and third tubes while the first duct is isolated; and there being stop means provided for register of the ducts and tubes.

Preferably the axes of the frusto-conical holes are substantially horizontal and preferably the body of each valve is substantially cylindrical. The apparatus is of great value when used as the gas measuring element of a vapour phase chromatographic apparatus.

Preferably the valve plugs each have at their small ends a short projection on which is located a compression spring provided with washers and adjusting screws. This spring urges the said projection away from the valve body and thus presses the valve plug into the valve body and gives a tight joint. When the spindle comprises two

halves connected by a collar, an arrangement which facilitates dismantling for cleaning, this tightness may be achieved alternatively although not so conveniently by locating a compression spring within the collar which urges each half of the spindle outwardly.

In order to provide good register between the ducts and the tubes in the two configurations, stops are provided. A preferred arrangement comprises a metal disc rigidly connected to the outer end face of each valve plug and having a peripheral slot which co-operates with a peg situated on the outer end face of the corresponding valve body.

Preferably the means adapted for rotating the spindle and valve plugs comprises a lever arm. This lever arm may be connected by a suitable link to a source of power such as a diaphragm valve operated by compressed air, e. g. at 20 lb./sq. in. gauge, and rocked at specified time intervals between the aforesaid configurations. The time intervals can be dictated by a process controller. Register between ducts and tubes may then be achieved by providing the lever arm with limiting stops.

The circumferential width of the ducts at the plug body interface of a valve and their angular distribution are selected relatively to the circumferential length of the said interface so that substantially no leakage occurs anywhere along the interface. We have found for this reason that the said angular distribution should with medium sized plugs preferably be such that: the angle between the axes of the second and third tubes, which is the same as that between the axes of the third and fourth tubes is not less than 30°; and that the angle between the first and second tubes is not less than 30°; and that between the first and fourth tubes not less than 60°. When the angles are less than those specified we have found there is increasing danger of leakage and consequent inaccuracy and/or contamination of the sample. Preferably the said angle between the second and third and between the third and fourth tubes should not be greater than 65° in order to keep down the distance travelled by the lever or other rocking means. With tubes of smaller diameter it is advantageous to reduce the circumferential width of the ducts and/or increase the angle between the axes of adjacent tubes. It is preferred to use tubes and ducts of circular cross-section because they are easily fabricated, but if desired other sections, e. g. elliptical may be used, which afford some benefit with plugs of narrow diameter.

Among the advantages of this apparatus are:

(1) By reason of its robustness it can be rocked. This permits mechanical driving and therefore renders the device automatic. Glass by reason of its fragility would not afford this.

(2) Automatic operation makes possible operation at such high frequency that for all practical purposes continuity is substantially achieved. Thus it has been possible with some gaseous mixtures to obtain a complete chromatogram every three minutes.

(3) These advantages are obtained without any sacrifice of accuracy. If anything the accuracy is greater than that obtained with glass.

(4) The apparatus is substantially gas tight.

(5) It has good durability and long life.

(6) It permits the use of higher pressures, which facilitate the use of considerably longer columns without unduly increasing the time required for the analysis of a sample. This results in more complete separation between the components of the mixture, which in turn affords improved differentiation between the portions of the chromatograph corresponding to the various components of the mixture.

The invention is illustrated in a preferred form in the accompanying drawings, of which

Figure 1 is a vertical elevation of the valve unit, Figure 2 is a vertical cross section taken on the line AA' in Figure 1, Figures 3 and 4 are diagrammatic sketches illustrating the operation of the valve, and Figure 5 is a general view showing the valve and pipette as an element in a vapour phase chromatographic apparatus. Fig. 6 is an end elevational view of part of the structure of Fig. 1. Like parts of the twin valves are indicated by the same numerals and are distinguished between themselves by the addition of a prime thus '.

In the drawings numeral **1** represents the valves each comprising a substantially cylindrical body **2** fixed to the heavy metal base **30**; **3** the co-operating valve plug, which preferably is tapped into the valve body to afford tight sealing; **4** the spindle, conveniently formed of two like halves **4** and **4'** coupled by the collar **31**; **5** the lever arm provided with an elbow **6** which may be slotted at **6'** for connection to suitable driving means; **14** the first duct provided with ports **16** and **17**, and **15** the second (V-shaped) duct provided with ports **18** and **19**; **7**, **8**, **9** and **10** the tubes. **22** is the compression spring situated between the disc **21** and the washer **23** and adjusted by screws **24** and **25**, which urges the plug **3** into the body **2**. The disc **21** is rigidly connected to the valve plug **3** and is provided with a peripheral slot co-operating with a stop **26** on the outer end face of the valve body **2**, the slot being of such length that the "stop positions" correspond to the two configurations of the valve.

When used in conjunction with a vapour phase chromatographic apparatus operation is as follows, referring to Figures 3 and 4.

Figure 4 shows the configuration after the sample has been transferred to the sorption column and the latter is working on it. Carrier nitrogen under a pressure of say 20 lb. per sq. in. gauge enters by the tube **8**, passes through the V-shaped duct **15** to the limb Y, and thence through tube **7'**, V-shaped duct **15'**, and tube **8'** to the column. Meanwhile tube **10** is in communication with the gas being treated and gas is flowing thence through duct **14**, tube **9**—pipette X—tube **9'**, duct **14'** and tube **10'** to atmosphere.

After a set period, which experience has shown to be adequate for analysis, a process controller initiates an electric circuit, which in turn opens a compressed air or inert gas valve. The compressed air is applied to a spring loaded diaphragm attached to a rod which rocks the lever arm **6**, and the system is then moved into the configuration in Figure 3, register being effected by the slotted discs **21** and stops **26**.

Nitrogen entering by tube **8** and V-duct **15** now sweeps the volume of pure gas which is contained in tube **9**—pipette X—tube **9'** through V-duct **15'** and tube **8'** into the sorption column. Meanwhile the system **7Y7'** is isolated, as can be seen from Figure 3. After running for a specified time interval in this way the process controller completes the circuit and the system is switched to the configuration shown in Figure 4.

As already indicated the valve pipette has its greatest value in relation to vapour phase chromatography. As a further feature the invention therefore includes apparatus for vapour phase chromatography characterised by the use in combination of the pipette: an actuating member such as a lever; a spring loaded diaphragm; an electric circuit, which may include a solenoid; a motor and a process controller of commercial type for dictating the time intervals. The process controller and motor may be replaced by a synchronous clock together with a cam, or by a clock driven by compressed air together with a cam. The cam may be slotted at spaced intervals to operate a switch which controls an electrical circuit including a solenoid adapted to open or close a valve situated in a compressed air or inert gas line.

A vapour phase chromatographic apparatus including the novel valve pipette as a feature is shown in Figure 5 and comprises a source of inert gas under pressure, e. g. a nitrogen cylinder **40**, fitted with a pressure gauge **41**; a reducing valve **42**; the valve pipette with pipette limb X, a manometer **44**; a flowmeter **45**; a chromatographic column **46** packed with a suitable sorption agent, for example silica gel, contained in a jacketed tube of wide diameter; **48** a hot wire Katharometer, with which is used a high speed recording potentiometer e. g. of the Brown Electronic type; and a valve **49**.

This operates as follows. Nitrogen is released from the cylinder **40** and controlled at a suitable pressure e. g. from about 2 to about 20 p. s. i. gauge by means of the valve **42**. The gas or vapour to be analysed enters the pipette X by line **43**. The gas sample is measured and delivered to the chromatographic column using configurations of the valve pipette shown in Figures 3 and 4. Nitrogen streams through the column continuously. The composition of the gas issuing from the column is continuously determined by measuring its thermal conductivity relative to that of the carrier gas and recording this on the Brown Electronic recording potentiometer. The amplitude of the peaks corresponding to the desired compound is measured and the concentration of the compound can be immediately obtained from calibration charts. The valve **49** is used to adjust the pressure difference so that the rate of sorption is that which gives good differentiation in the sorbabilities of the components of the gas mixture.

If desired, suction may be applied to the exit end of the column, while the inlet end is either above atmospheric, or at atmospheric, or at a sub-atmospheric pressure greater than the pressure at the exit end.

Other suitable sorption agents are, for example activated carbon or charcoal in particulate form, or a liquid of low volatility, such as medicinal paraffin, supported on an inert support in particulate form, such as diatomaceous earth.

Instead of a Katharometer, similar or other appropriate analyzing devices may be used.

I claim:

1. Apparatus adapted for measuring and delivering at controlled time intervals an accurate volume of gas and suitable for mechanical operation which comprises two valves each having a fixed metal body formed with an interior frusto-conical hole tapering outwardly, and situated in the said hole a co-operating closely fitting rotatable metal valve plug; a metal spindle rigidly connecting said valve plugs provided with means adapted for rotating the spindle and valve plugs; four metal tubes radiating from the body of each valve and in free connection with the frusto-conical hole therein; the first tube of the first valve being in connection with the supply of gas being measured, the second forming one limb of a pipette, the third being in connection with a supply of inert gas under pressure, the fourth being in connection with the fourth tube of the second valve; the first tube of the second valve being open to atmosphere, the second forming the other limb of the pipette, the third being in connection with the apparatus to which the gas is being supplied, the fourth being in connection with the fourth tube of the first valve; and situated in each of said valve plugs a first transverse duct and a second separate V-shaped duct, each having ports in the surface of the valve plug; the area of the ports and the circumferential distance between them being such that in a first configuration the first duct is in communication between the first and second tubes when the second duct is in communication between the third and fourth tubes; and that in a second configuration the second duct is in communication between the second and third tubes while the first duct is isolated and the first and fourth tubes are blocked; and there being stop means provided for register of the ducts and tubes.

2. Apparatus as claimed in claim 1 in which the second V-shaped duct is a roughly sector-shaped recess.

3. Apparatus as claimed in claim 1 in which the axes of the frusto-conical holes are substantially horizontal.

4. Apparatus as claimed in claim 1 in which each of the valve plugs is provided with an element for pressing it into the hole with which it co-operates, which element comprises a compression spring located on a short projecting portion of the plug.

5. Apparatus as claimed in claim 1 in which the stop means comprises a metal disc rigidly connected to the outer end face of each valve plug and provided with a peripheral slot co-operating with a peg on the outer face of the valve body, the slot being of such length that the

"stop positions" correspond to the two configurations of the valve.

6. Apparatus as claimed in claim 1 in which the angle between any two of the tubes is at least 30°, and that between the first and fourth tubes is at least 60°.

References Cited in the file of this patent

UNITED STATES PATENTS

2,398,818	Turner	Apr. 23, 1946
2,434,723	Shook	Jan. 20, 1948
2,591,762	Zaikowski	Apr. 8, 1952
2,604,248	Gorham	July 22, 1952