Jan. 10, 1967 M. VAN SWAAY 3,297,062

FRACTION COLLECTOR FOR OPERATION AT REDUCED PRESSURE

Filed June 25, 1964 2 Sheets-Sheet 1

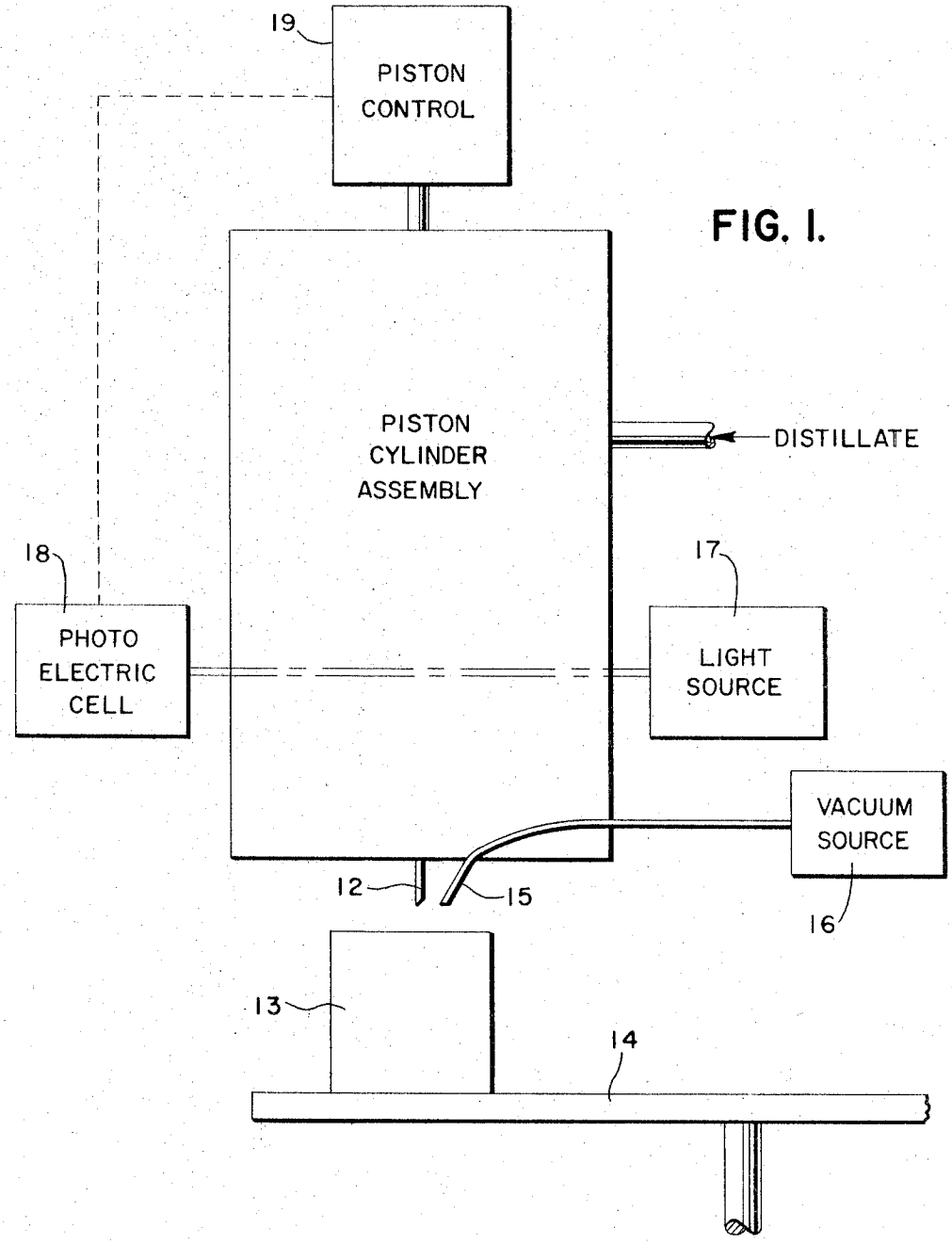


FIG. I.

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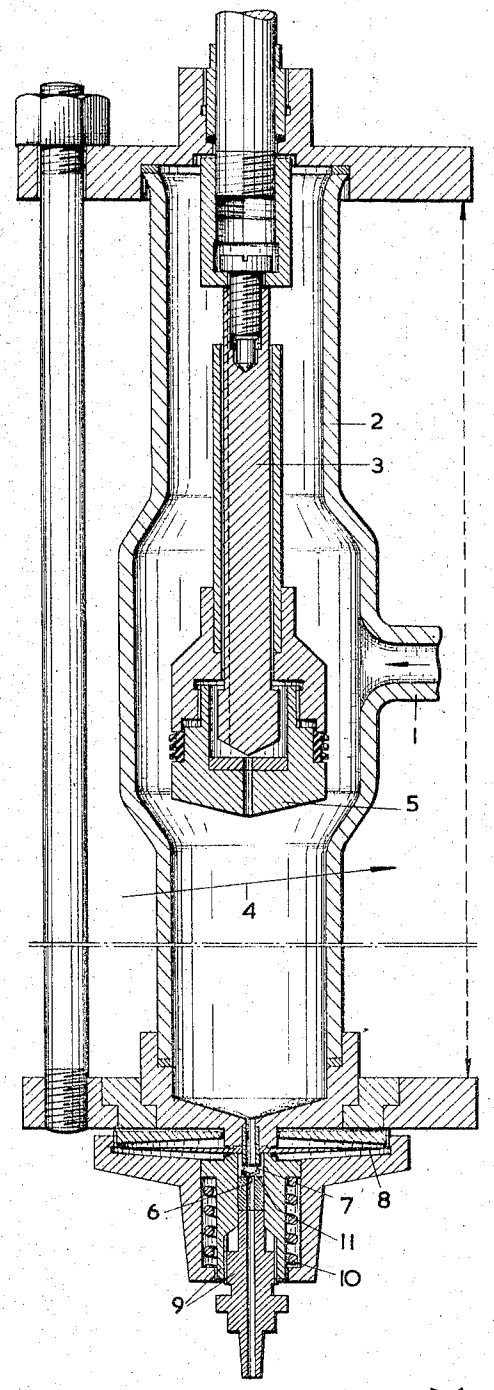


FIG. 2

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FRACTION COLLECTOR FOR OPERATION AT REDUCED PRESSURE

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Claims priority, application Netherlands, July 8, 1963, 295,047
6 Claims. (Cl. 141—130)

The invention relates to an apparatus for continuously drawing off given quantities of liquid samples which are liberated at reduced pressure, in particular for analytical distillation, the collecting vessels for the samples being mounted on a revolving table and being successively moved underneath an apparatus communicating with the liquid column from which the samples have to be taken.

Analytical distillation is frequently performed as a charge process, in which suitable quantities of fractions are collected and are subsequently analyzed by different methods, such as gas chromatography and spectrometry. The composition of the distillate can be determined very rapidly by these methods, while the availability of this information has made possible a very effective control of the distillation process. When the distillation is carried out at atmospheric pressure, the quantity of the fraction can be determined from the volume or the weight of a siphon or by counting drops. Transfer of the sample to the collecting vessel is achieved by means of gravity, while the collecting vessels may or may not be closed. They can be so closed, for instance, by dropping a ball into the neck of the vessel.

The construction of a fraction collector of this type, designed for operation at reduced pressure, involves mechanical difficulties because all the parts, including the frequently voluminous apparatus in which the collectors are accommodated, must be mounted in a vacuum-tight enclosure. Moreover the removal of individual fractions is very cumbersome, so that the distillation process must be adjusted while the composition of the distillate is unknown.

In view of the above, fractions are usually collected by hand during distillations at reduced pressure.

It is the object of the invention to furnish an apparatus for drawing off samples at reduced pressure. To achieve this object, the apparatus according to the invention comprises a piston adapted to move up and down in a cylinder, which piston is moved down at a predetermined level of the sample collected in the cylinder, while a valve which is adapted to move along the piston and which in the uppermost position of the piston is located in a wider part of the cylinder is forced into the lower-narrower-part of the cylinder, in which the valve fits closely.

It is a further object of the invention to furnish an apparatus capable of operating completely automatically. To this end the cylinder, which is provided on its underside with a shut-off valve and a needle which has to bring about the communication with the collector located underneath the apparatus, can be moved up and down, all this in such a way that upon continued downward movement of the piston the increased pressure of the sample in the lower part of the cylinder will shut the piston valve further as well as move down the cylinder with the needle, while at the same time opening the shut-off valve.

In this way it is ensured that during the operation of the column at reduced pressure the individual fractions are accessible at atmospheric pressure, while it is possible to collect them under protective gas and in sealed collectors under a vacuum with only small modifications of the equipment.

The means for accomplishing the foregoing objects

and other advantages, which will be apparent to those skilled in the art, are set forth in the following specification and claims, and are illustrated in the accompanying drawings dealing with a basic embodiment of the present invention. Reference is made now to the drawings in which:

FIG. 1 illustrates the invention in a schematic representation; and

FIG. 2 is a vertical section through the piston-cylinder assembly.

Through a conduit 1 distillate is fed into a glass cylinder 2. A piston 3 is present in cylinder 2 and in its position of rest hangs freely in a widened part of the cylinder 2. The distillate then collects in the lower part of the cylinder 2. When the distillate in the cylinder 2 reaches a given level, corresponding, for instance, to a volume of 100 ml., the liquid surface interrupts a beam of light 4, which is directed from a light source 17 towards a photoelectric cell 18, owing to the total reflection of said surface. The photoelectric cell serves to actuate a piston control 19 whereby the downward stroke of the piston 3 is initiated. As soon as the piston enters the lower part of the cylinder 2, a closing force is exerted by friction between the valve 5 and cylinder 2, which is adapted to move along the piston 3. The sample collected is now separated from the distillation column.

When the piston 3 moves further down, an additional closing force is exerted on the piston valve 5 by the increasing pressure in the enclosed volume. On the other hand the enclosed volume exerts a downwardly directed force on the cylinder 2, which is adapted to move vertically (by means not shown) so as to make possible a vertical movement of 2 cm., for instance. Owing to this movement a needle 12 on the underside of the cylinder 2 is inserted through a partition into a collector 13, which is mounted on a revolving table 14. Upon a further increase of the pressure underneath the piston 3, a valve 6 on the underside of the cylinder opens and the sample is transferred to the collector underneath. The opening of the valve 6 takes place because via openings 7 in the valve 6 the increasing pressure is propagated between the diaphragm 8, and the structure 9 is moved down, against the force of the spring 10, as the pressure reaches a given value, in consequence of which the seat 11 of the valve 6 moves away from said valve 6.

The air or the inert gas in the collector can escape via a second needle 15, provided adjacent to the sample needle. The air can also be removed from the vessel beforehand by means such as needle 15 and a vacuum source 16. This can be achieved by means of a needle fitted above the next collector on a bracket which is connected with the cylinder flange, so that this needle is inserted and withdrawn simultaneously with the sample needle. At the end of the transfer of the sample to the collector the gas present above the sample blows all the remaining liquid from the cylinder valve 6.

This valve 6 is self-closing, while the reduced pressure in the column exerts an additional closing force.

When the piston 3 is now moved up, the piston valve 5 opens, so that no pressure disturbances can occur when the sealing with the cylinder wall near the wider part of the cylinder 2 is broken. The cylinder 2 is returned to its uppermost position either by friction of the piston 3 or by the piston unit reaching the flange at the top of the cylinder 2. The next collector can then be placed underneath the needle by rotation of the revolving table, and the cycle starts again.

What I claim is:

1. An apparatus for continuously drawing off given quantities of liquid samples which are liberated at reduced pressure, in particular, for analytical distillation

comprising a cylindrical trap chamber, a lower portion of which is of a reduced diameter for receiving and trapping a liquid sample therein, means to feed said liquid to said chamber, piston means movably mounted within said chamber, means responsive to the level of the liquid within said chamber to initiate a downward movement of said piston means, valve means mounted at the lower end of said reduced portion and responsive to an increase in pressure caused by said piston on said liquid, collector means positioned below said valve, and means for directing the liquid from said valve into said collector means.

2. An apparatus according to claim 1 wherein said piston is a lost motion piston and said cylindrical trap chamber is mounted to move vertically with an initial movement of said piston, said chamber movement bringing said directing means into communication with said collector means.

3. An apparatus according to claim 1 wherein said valve means is a pop valve comprising a tubular portion having therein an opening, a valve, a spring-biased valve seat movably mounted on a diaphragm, an increase in pressure acting on said diaphragm against said spring biasing means to cause said valve to open.

4. An apparatus according to claim 1 in which said liquid-level sensing device comprises a light source and a photoelectric cell disposed on opposite sides of said chamber, said photoelectric cell being connected to energize means for initiating movement of said piston when the presence of a liquid is detected.

5. An apparatus according to claim 1 wherein said collector means comprises a plurality of receiving units mounted on a revolving table, said table selectively positioning said units under said liquid directing means.

6. An apparatus according to claim 1 wherein needle means connected to a vacuum source are provided, said needle means penetrating said collecting means with the downward movement of said chamber, said collecting means being evacuated prior to delivery of said liquid sample.

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