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FRACTION COLLECTOR

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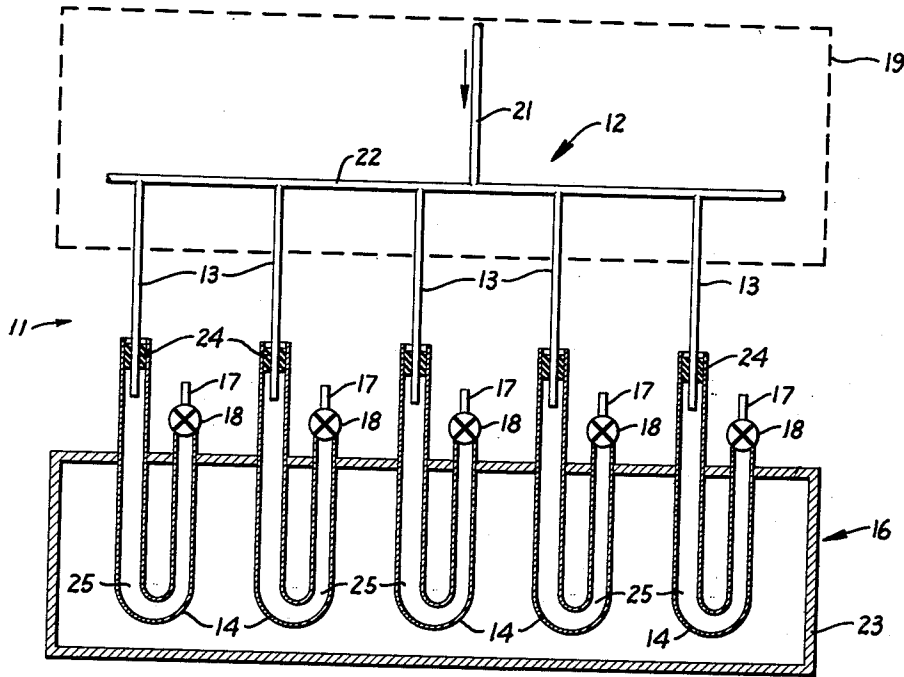


FIG. 1.

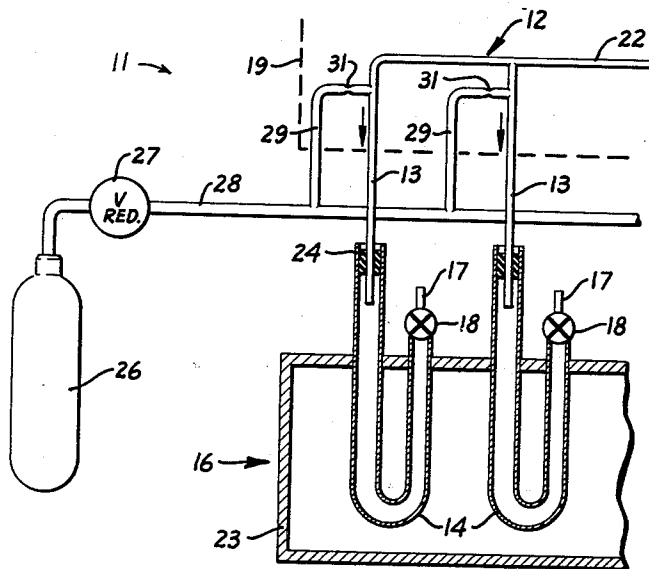


FIG. 2.

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FRACTION COLLECTOR

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4 Claims. (Cl. 165—71)

The present invention relates to improvements in a fraction collector and particularly to an automatic fraction collector suitable for collection of components from the effluent stream of a gas chromatograph.

In gas chromatography, one or more components are separated from a given material with the material in a vapor state, and the separated component or components are collected separately for further determination. In a typical operation, the separation is effected at elevated temperatures and the fractions are collected as liquids. In this way, certain selected components are collected in different containers.

In order to direct the fractions to be separated into separate containers, certain procedures have been developed which are capable of performing the desired operation, but which have presented certain problems that are overcome by the improved system herein described. For example, one method utilizes valves in a manifold to direct the fractions to the desired container. This procedure has the disadvantage of requiring the use of valves in a hot environment where the valve life is rather short. On the other hand, it has been considered desirable to use the valve in the heated gas line so that substantially no condensation of liquid occurs in the valve.

It has now been found that a suitable system may be used for collecting fractions that overcomes the above mentioned problem by a unique placement of the valves. This placement allows for automatic operation simply by automatic control of valves placed at the outlet of each container regulating the flow of fluid into each container according to a predetermined program.

It is therefore a primary object of this invention to provide an automatic fraction collector for use in a gas chromatograph to collect the fractions obtained therefrom in a plurality of containers by utilizing a novel valving arrangement capable of automatic control in which the valves are positioned in a favorable environment for long satisfactory service.

Another object of the invention is to provide an automatic fraction collector of the character described having means for providing a purging gas in the system to prevent any cross-contamination of the components collected in each vessel.

A further object of the invention is the provision of an automatic fraction collector of the character described that is simple in construction, capable of providing many years of satisfactory service, and which is not adversely affected by the temperatures encountered in the normal operation of the system.

Further objects and advantages of our invention will be apparent as the specification progresses, and the new and useful features of our fraction collector will be fully defined in the claims attached hereto.

The preferred forms of the invention are illustrated in the accompanying drawing, forming a part of this description, in which:

FIGURE 1 is a schematic representation of one form of fraction collector embodying the invention; and

FIGURE 2, a schematic fragmentary view illustrating a preferred form of the invention.

While we have shown only certain illustrative forms of our invention, it should be understood that various changes or modifications may be made within the scope of the

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claims attached hereto without departing from the spirit of the invention.

Referring to the drawing in greater detail, there is shown in FIGURE 1 an automatic fraction collector 11 comprising a heated manifold 12 having a plurality of outlet ducts 13, a plurality of containers 14 in communication with the outlet ducts, cooling means 16 for cooling said containers to provide condensation therein, outlets 17 on the containers 14, and valves 18 at said outlets for controlling the flow of material through the containers.

The manifold is kept in the heated area 19 which may be an oven or any other suitable temperature controlling means. In this way, the manifold may be maintained at a controlled temperature of say up to 400° C. or higher in order to maintain all the components in a gaseous state until they reach the container 14 or collection vessels. As best seen in FIGURE 1, a typical manifold consists of an effluent gas line 21 which carries material from the gas chromatograph in the vapor state, outlet ducts 13, and a header 22 which provides communication between the line 21 and the outlet ducts.

The outlet ducts then lead to the containers 14 which are maintained in a suitable condensation area such as refrigerator 23. It is important that the connection between the outlet ducts 13 and the containers 14 be made with a gas-tight seal for proper operation of the system. Thus the connections are made with connecting means 24 which may be a rubber stopper or any other device capable of providing the necessary seal.

Each container or collection vessel 14 is located in an area suitable for condensation of the component, and the temperature of the area is determined by the nature of the components to be collected. In other words, the condensation area may be refrigerated, ambient or even above ambient in some cases. As here shown, outlet ducts 13 lead through an ambient area to the containers 14 positioned in a cooled area 25 maintained in the refrigerator. With this arrangement, the fraction to be collected condenses and remains in a cooled area. The outlets of the container are provided at a position outside the refrigerator so that valves may be provided between the outlet point and the main body of the container without including the valves in the refrigerated area. In other words, the valves are provided in an ambient temperature zone.

The refrigerator 23 may be any unit capable of providing the desired cold temperature zone for substantially complete condensation of the components to be collected. Thus the refrigerator may simply be a chamber as shown suitable for holding ice, ice and brine mixture, or a Dry Ice and acetone mixture depending upon the temperature desired to be maintained in the cold zone. Alternatively, the temperature may be maintained at the desired low temperature by utilizing conventional refrigeration coils in the box.

The operation of the system is illustrated as follows: When all of the valves are closed, no gas flows in the effluent gas line 21 from the chromatograph and no components are collected. However, when one of the valves is opened to the air, gas is free to flow through effluent gas line 21, header 22 the outlet duct 13 in communication with the container having the open valve, through the container, and though the opened valve so that any carrier gas is free to pass through the outlet 17. No flow of the component takes place through the other containers, because the other valves are maintained in a closed position. After the fraction has been collected in one container, the next fraction may be collected in another container simply by closing the valve of the first container and opening the valve of the next container. In this way, all of the desired fractions are collected in the different containers.

Although five containers are shown in FIGURE 1, it

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should be understood that any number of containers may be provided according to the demands of the system. It is also seen that the system may be easily automated through the valves, because automatically controlled valves are well known. In other words, valves which are controlled electrically, pneumatically or hydraulically are available and could be used with electrical, pneumatic or hydraulic controls integrated into the system to depend upon the operation of the chromatograph itself.

An improved form of the invention is shown in FIGURE 2, which is constructed in similar fashion to FIGURE 1 but which also includes means for supplying an inert gas purge to each of the outlet ducts 13 at a position between the manifold 12 and the containers 14. Preferably the inert gas is admitted in the heated zone as shown although this is not essential.

A typical inert gas system includes a gas supply source 26, a control means 27 for controlling the pressure in the inert gas purge line 28 which supplies inert gas to the ducts 13 through feeder lines 29. As best seen in FIGURE 2, a gas supply source 26 may be a cylinder of inert gas under pressure, and the control means 27 is arranged to produce a pressure line 28 slightly higher than that in any of the feeder lines 13. With this arrangement, the purge gas extends to pass through each line 13 in reverse direction except through the line 13 which is connected to a container with open valve 18. The reverse flow of purge gas through all closed lines 13 prevents cross-contamination of vapors.

In order to insure a positive pressure in line 28 and lines 29, a constricted area 31 is provided in each line 29 in the preferred embodiment as shown in the drawing. In this way, any likelihood of cross-contamination of the samples is positively avoided.

In operation, the inert gas purge is applied to the system and each valve is opened individually to provide an inert gas purge through the system so as to give the system an inert atmosphere at the beginning. The gas chromatograph is then connected to effluent line 21 and the valves 18 are operated individually as mentioned above. When none of the valves are opened, none of the containers collect any gas, but as each valve is opened one at a time the fraction to be called flows through appropriate vessels and the inert gas purge prevents cross-contamination. The inert gas so utilized passes out through the outlet 17 associated with the open valve 18. The embodiment shown in FIGURE 2 is thus also capable of simple automation because the entire controls of the operation of the system is through the valves 18 which may be controlled automatically as indicated above.

From the foregoing description, it is seen that we have provided an automatic fraction collector for collecting fractions of gas from gas chromatograph at elevated temperatures in plurality of containers maintained at low temperatures using a comparatively simple system completely controlled by the valves which are maintained in an ambient temperature zone. In this way, the valves are protected from the adverse effect of high or low temperatures and are easily suited for automation.

We claim:

1. An automatic fraction collector for collecting fractions of gas components from the effluent stream of a gas chromatograph delivered at elevated temperature in a plurality of containers at low temperatures, comprising

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a heated manifold having an inlet duct for receipt of the effluent stream of a gas chromatograph and a plurality of outlet ducts, a plurality of containers in communication with said outlet ducts, means for supplying an inert purge gas to the outlet ducts in the heated manifold, means for cooling said containers below the temperature of said manifold to provide condensation of gas components therein, outlets on said containers, and valves on said outlets for controlling the flow of the effluent stream and purge gas through said containers.

2. An automatic fraction collector for collecting fractions of gas components from the effluent stream of a gas chromatograph delivered at elevated temperature in a plurality of containers at low temperatures, comprising a heated manifold having an inlet duct for receipt of the effluent stream of a gas chromatograph and a plurality of outlet ducts, means for supplying an inert purge gas to the outlet ducts in the heated manifold, means for providing a condensation temperature region, a plurality of containers in said temperature region with each container being connected to one of said outlet ducts, said outlet ducts being connected to the containers in a fluid-tight relationship so that all the fluid passing through each duct must pass through the container attached thereto, outlets on said containers, and valves on said outlets for controlling the flow of the effluent stream and purge gas through said containers.

3. An automatic fraction collector for collecting fractions of gas components from the effluent stream of a gas chromatograph delivered at elevated temperature in a plurality of containers at low temperatures, comprising a heated manifold having an inlet duct for receipt of the effluent stream of a gas chromatograph and a plurality of outlet ducts, means for supplying an inert purge gas to the outlet ducts in the heated manifold, cooling means for providing a cold temperature region substantially less than ambient, and a plurality of containers in said cold temperature region with each container being connected to one of said outlet ducts, said outlet ducts being connected to the containers in a fluid-tight relationship so that all the fluid passing through each duct must pass through the container attached thereto, each of said containers having an outlet extending beyond the cold temperature region into a substantially ambient temperature region with each outlet containing a valve in the ambient temperature region for controlling the flow of the effluent stream and purge gas through the container.

4. The fraction collector defined in claim 3, in which the means for supplying an inert purge gas includes a gas supply line, means for providing purge gas in the supply line at a controlled pressure, and a branch line connecting each of the outlet ducts in the heated manifold to the gas supply line, each of said branch lines having a constricted area near the connection to the outlet duct.

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