

Sept. 10, 1968

TAMOTSU NODA

3,400,514

CARRIER GAS SEPARATING DEVICE

Filed Sept. 20, 1966

FIG. 1

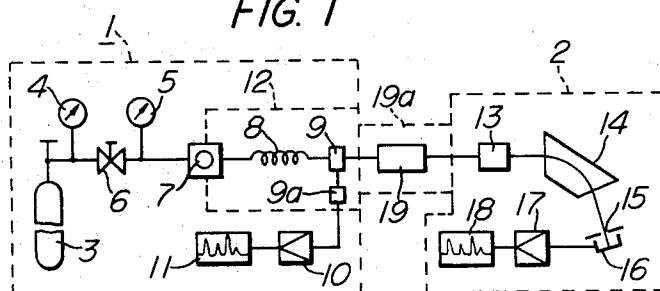


FIG. 2

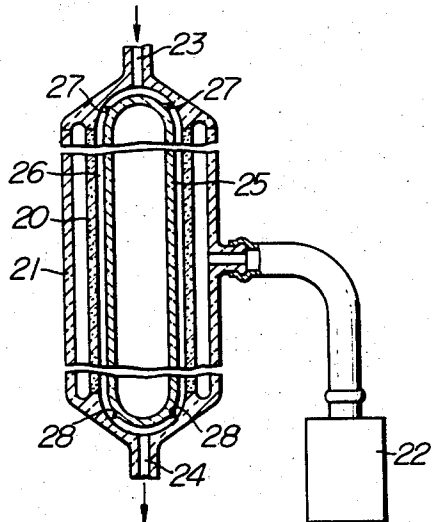


FIG. 4

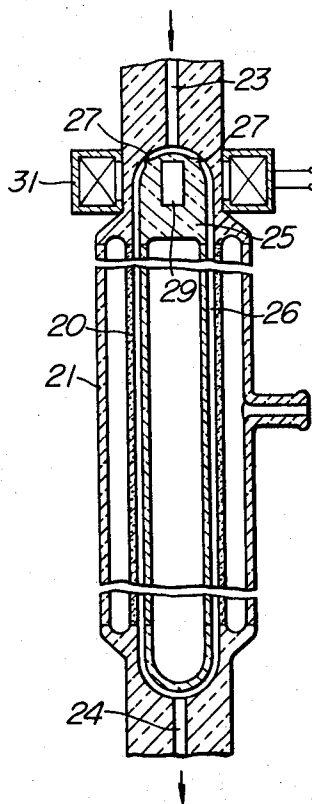
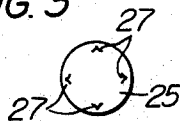


FIG. 3



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CARRIER GAS SEPARATING DEVICE

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Filed Sept. 20, 1966, Ser. No. 580,746

Claims priority, application Japan, Oct. 1, 1965,

40/79,571, 40/79,572

3 Claims. (Cl. 55-158)

ABSTRACT OF THE DISCLOSURE

A device for separating carrier gas from sample components for use in a system in which the sample components emerging from a gas chromatograph are subjected to mass spectrometric analysis by the use of a mass spectrometer, comprising a porous carrier gas separating tube, a vacuum jacket enclosing said tube, an inlet to said tube connected with a gas chromatograph and an outlet from said tube connected with a mass spectrometer, means for evacuating the interior of the vacuum jacket to remove a carrier gas, through the porous wall of the tube, which enters the tube through the inlet, together with sample components, from the gas chromatograph, and an air-tight inner space reduction member positioned within the tube, thus defining a uniform interstice between the member and the tube, which interstice provides for communication between the inlet and the outlet of the tube. According to an alternative embodiment of the present invention, the inner space reduction member is constructed so as to be movable axially within the porous tube to effect a blocking of communication between the inlet and outlet means of the tube. The movement of the inner space reduction member may be accomplished by electromagnetic means.

The present invention relates to a carrier gas separating device for use with a system in which sample components separated in and emerging from a gas chromatograph are led to a mass spectrometer and subjected to mass spectrometric analysis.

When performing mass analysis of sample components separated in and emerging from a gas chromatograph by a mass spectrometer directly coupled to the gas chromatograph, the presence of the carrier gas emerging from the gas chromatograph together with the separated sample components is quite unnecessary to the mass spectrometer and it is ideally desirable that the carrier gas be non-existent.

In order to remove the carrier gas unnecessary to the mass spectrometer, an attempt has already been proposed in which a porous carrier gas separating tube enclosed by a vacuum jacket is connected between the gas chromatograph and the mass spectrometer to remove the unnecessary carrier gas emerging from the gas chromatograph into the vacuum jacket through the carrier gas separating tube (see, for example, Analytical Chemistry, vol. 37, No. 6, May 1964, pages 1135 to 1137).

However, the conventional carrier gas separating device is unable to remove the carrier gas satisfactorily owing to the small inner surface area of the carrier gas separating tube, i.e. contact surface to the carrier gas. If the inner surface of the carrier gas separating tube is made large, its inner space necessarily becomes larger, giving rise to the diffusion or remixing of the sample components separated in and emerging from the gas chromatograph in the carrier gas separating tube. Therefore, completely satisfactory results of analysis can not be expected.

When a gas chromatograph alone is operated, when the operations of a gas chromatograph and a mass spectroscopy are both suspended, or when a private sample introduction device for mass spectrometer is connected to

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the ion source of a mass spectrometer, it is necessary to keep carrier gas and effluents from the gas chromatograph from flowing into the mass spectrometer. To this end, it is necessary to provide a valve between the gas chromatograph and the mass spectrometer. However, if dead volume is formed around the valve by the provision of the valve, the diffusion or remixing of the sample components separated in and emerging from the gas chromatograph will take place in the dead volume. Therefore, the valve must be so constructed as not to provide the dead volume.

Therefore, an object of the present invention is to provide a carrier gas separating device in which the inner surface area of a carrier gas separating tube can be made sufficiently large without increasing the inner space thereof.

The carrier gas separating device according to the present invention comprises a carrier gas separating tube made of porous material, a vacuum jacket enclosing said tube, means for evacuating the interior of said jacket, an inner space reduction member inserted in said carrier gas separating tube in such a manner that interstice is formed between said member and said carrier gas separating tube, and an inlet and an outlet provided respectively at the opposite ends of said carrier gas separating tube and communicating with each other through said interstice.

According to the carrier gas separating device of the invention having such construction, the inner surface area of the carrier gas separating tube can be made large without increasing the inner space thereof. That is, putting the inner surface area of the carrier gas separating tube as A and the inner space thereof as V , the value of A/V can be made large by employing an inner space reduction member. In order to enhance the carrier gas separating effect while preventing the diffusion or mixing phenomenon of the separated sample components in the carrier gas separating tube, it is conceivable to reduce the inner radius of the carrier gas separating tube and to sufficiently increase the length thereof. However, such a tube has disadvantages in that the manufacture thereof is very difficult. Against this, according to the carrier gas separating device of the invention having the abovementioned construction, the carrier gas separating effect can be markedly enhanced while preventing the diffusion or mixing phenomenon of the separated sample components in the carrier gas separating tube.

Another object of the present invention is to provide a carrier gas separating device in which an inner space reduction member inserted in a carrier gas separating tube is also utilized as a valve.

The carrier gas separating apparatus according to the present invention comprises means for moving axially the inner space reduction member inserted in the carrier gas separating tube so as to interrupt the communication between the inlet and the outlet provided respectively at the opposite ends of the carrier gas separating tube.

According to the carrier gas separating device of the invention having the aforementioned construction, since said inner space reduction member having the function of increasing the value of A/V can also be utilized as a valve, a separate valve mechanism is unnecessary. Accordingly, in spite of the simple construction, the carrier gas and the sample components separated in and emerging from the gas chromatograph can be kept from flowing into the mass spectroscopy as occasion demands. Also, even if the inner space reduction member is utilized as a valve, as long as the inlet and the outlet of the carrier gas separating tube are communicating, no large dead volume is formed in the flow path in the carrier gas separating tube. Therefore, no diffusion or mixing phenomenon

non of the separated sample components takes place in the carrier gas separating tube.

Other objects and advantages of the invention will become more apparent from the following detailed description of the invention when read with reference to the accompanying drawings, in which:

FIG. 1 is a block diagram of an apparatus in which a gas chromatograph is directly coupled with a mass spectrometer;

FIG. 2 is a cross-sectional elevation view of an embodiment of the invention;

FIG. 3 is a plan view of the inner space reduction member shown in FIG. 2; and

FIG. 4 is a cross-sectional elevation view of another embodiment of the invention.

Now referring to FIG. 1, a description of an apparatus constructed by directly coupling a gas chromatograph with a mass spectrometer will first be given. In FIG. 1, a gas chromatograph 1 is constituted by a carrier gas bottle 3, pressure gauges 4 and 5, a needle valve 6, a sample inlet 7, a column 8, a flow splitter 9, a detector 9a, a recorder 11 connected through the intermediary of an amplifier 10 to the detector 9a, and a temperature controlled oven for keeping the column 8 and the detector 9a at a constant temperature. A mass spectrometer 2 comprises an ion source 13, magnetic poles 14, a slit 15, an ion collector 16, and a recorder 18 connected through an amplifier 17 to the ion collector 16. Between the gas chromatograph 1 and the mass spectrometer 2 is connected a carrier gas separating device 19 enclosed by an oven 19a.

Now, while letting the carrier gas contained in the gas bottle 3 flow into the column 8 and watching the pressure gauges 4 and 5, the needle valve 6 is adjusted to set the carrier gas to a predetermined pressure. Then, if a predetermined quantity of sample is injected into the sample inlet 7, the sample is introduced into the column 8 by the carrier gas. Since the column 8 is filled with an adsorbent, the sample introduced into the column 8 is separated in accordance with the differences between the affinities of the components of the sample and the adsorbent. The separated components are detected by the detector 9a. The detector 9a then generates electric signals corresponding to the amount of sample components, which is fed, after being amplified by the amplifier 10, to the recorder 11. Thus, a peak corresponding to the sample component appears on the recorder. When the mass analysis of the sample components separated in and emerging from the gas chromatograph is carried out by coupling the gas chromatograph directly to the mass spectrometer, the detector in the gas chromatograph is not necessarily required. In such case, the detector 9a, the amplifier 10 and the recorder 11 can be excluded or their operations can be suspended.

On the other hand, the sample components separated by the gas chromatograph 1 fed into the mass spectrometer 2 on the carrier gas, but the carrier gas itself is utterly unnecessary to the mass spectrometer. Hence, the carrier gas is separated for the most part by the carrier gas separating device 19 connected between the gas chromatograph 1 and the mass spectrometer 2. Since the mass spectrometer 2 has been evacuated to a high vacuum by pumping systems (not shown), the sample components including a small amount of the carrier gas are introduced into the mass spectrometer 2 due to the vacuum suction. Then, the sample components are ionized in the ion source 13. The ions generated in the ion source 13 are directed to the magnetic poles 14, and are dispersed and focussed depending on the masses of the ions, when passing through the magnetic field developed by the said magnetic poles. Consequently, if the magnetic field is scanned by scanning system (not shown), the ions dispersed depending on their masses pass through the slit 15 and are sequentially collected on the ion collector 16. The collected ions are converted at the ion collector 16 into an

electric signal which is, after being amplified by the amplifier 17, supplied to the recorder 18. Thus mass spectrum is displayed on the recorder 18.

The carrier gas separating device according to the present invention is to be used as the carrier gas separating device 19 in FIG. 1.

FIGS. 2 and 3 show an example of the carrier gas separating device according to the invention. A carrier gas separating tube 20 is made of fritted glass having fine pores of the order of, for example, a few microns. The carrier gas separating tube 20 is enclosed by a vacuum jacket 21, the interior of which is evacuated by means of a pumping system 22. At the upper and lower ends of the carrier gas separating tube 20 are respectively provided an inlet port 23 and an outlet 24. In the interior of the carrier gas separating tube 20 is mounted an inner space reduction member 25 so that an interstice 26 may be left therebetween. The upper and lower end portions of the inner space reduction member 25 are of spherical form and provided respectively with a plurality of protuberances 27 and 28 which support the inner space reduction member 25 within the carrier gas separating tube 20 so as to maintain the communication between the inlet 23 and the outlet 24. The inlet 23 of the carrier gas separating tube 20 is to be connected to the outlet of a gas chromatograph and the outlet 24 of the tube 20 is to be coupled with the inlet of a mass spectrometer.

Now, if the interior of a vacuum jacket 21 is evacuated by a pumping system 22, the carrier gas is almost entirely removed, through the fine pores of the carrier gas separating tube 20, while the mixture of the carrier gas and the sample component flow into the interstice 26 through the inlet 23. Since the mass spectrometer is evacuated in high degree of vacuum, the sample components passing through the interspace 26 are led to the mass spectrometer through the outlet 24.

Since the inner space reduction member 25 is mounted within the carrier gas separating device 20 the inner surface area of the carrier gas separating tube 20 has been made sufficiently large without increasing the inner space thereof. Consequently, the diffusion or mixing of the separated sample components in the carrier gas separating tube 20 can reasonably be prevented as well as the carrier gas removing effect can be strikingly promoted. Moreover, it is unnecessary to elongate the tube 20 in order to enlarge its inner surface area, resulting in no fabrication difficulty.

FIG. 4 illustrates another carrier gas separating device according to the invention. Similar parts of the embodiment of FIG. 4 to that of FIGS. 2 and 3 are designated by the same reference numerals as used in FIGS. 2 and 3. The carrier gas removing apparatus of FIG. 4 is different from that shown in FIGS. 2 and 3 in that a piece of magnet 29 is embedded in the upper end portion of the inner space reduction member 25, in that no protuberances are provided at the lower end of the inner space reduction member 25, and in that an electro-magnetic coil 31 for electro-magnetically attracting the magnetic piece 29 is provided surrounding the upper end portion of the carrier gas separating tube 20.

Now, when the electro-magnetic coil 31 is energized, the magnetic piece 29, and accordingly the inner space reduction member 25 is displaced upwardly due to the electro-magnetic attractive force between the coil 31 and the magnetic piece 29. FIG. 4 shows the apparatus in this state. When the inner space reduction member 25 is displaced upwardly as illustrated, the inlet 23 and the outlet 24 communicate with each other through the interstice 26. Therefore, it will readily be understood that the carrier gas removing effect is strikingly promoted and, at the same time, the diffusion or mixing of the separated sample components in the interior of the carrier gas separating tube is prevented in quite the same manner as in the carrier gas separating device of FIG. 2.

When the electro-magnetic coil 31 is deenergized, the

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inner space reduction member 25 drops downwardly due to the force of gravity to stop the outlet 24 with the lower end thereof, resulting in the interruption of the communication between the inlet 23 and the outlet 24.

Although, some preferred embodiments of the invention has been described, it will readily be understood that other changes and modifications can be made easily.

What I claim is:

1. A carrier gas separating device for use in a system in which sample components separated in and emerging from a gas chromatograph are subjected to mass spectrometric analysis by the use of a mass spectrometer, comprising:

a carrier gas separating tube made of a porous material,

inlet means and outlet means provided respectively at the opposite ends of said tube, said inlet means being connected with a gas chromatograph and said outlet means being connected with a mass spectrometer,

a vacuum jacket enclosing said tube, means for evacuating the interior of said jacket so as to effect the removal, through the porous wall of said tube, of a carrier gas flowing into said tube, through said inlet means, from a gas chromatograph, together with sample components, and,

an air-tight inner space reduction member positioned within said tube so as to define a uniform interstice between said member and said tube, said interstice providing for communication between said inlet means and said outlet means.

2. A carrier gas separating device comprising a carrier gas separating tube made of porous material, a vacuum jacket enclosing said tube, means for evacuating the in-

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terior of said jacket, and an inlet and an outlet provided respectively at the opposite ends of said carrier gas separating tube, characterized in that an inner space reduction member is arranged within said carrier gas separating tube so that an interstice is formed between said inner space reduction member and said carrier gas separating tube, and that said inlet and said outlet communicate with each other through said interstice, and further comprising means for moving axially said inner space reduction member arranged in said carrier gas separating tube so as to interrupt the communication between said inlet and said outlet provided respectively at the opposite ends of said carrier gas separating tube.

3. A carrier gas separating device according to claim 2 characterized in that said means for moving axially said inner space reduction member comprises a piece of magnet embedded in the upper end portion of said inner space reduction member, and an electro-magnetic coil for axially moving said inner space reduction member due to the electromagnetic attractive force between said coil and said piece of magnet.

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