NASA TECHNICAL MEMORANDUM NASA TM-77505

HELIUM IONIZATION DETECTION APPARATUS

Shimazu Seisakujo Co., Ltd.

Translation of Japanese Kokai Patent Publication No. 58-172544, published October 11, 1983; Application No. 57-55563, filed April 2, 1982; inventor: Ryukou Nagai; assignee: Shimazu Seisakujo Co., Ltd.

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16. Abstract

In a gas chromatograph apparatus comprising a gas supply (He carrier gas), a sample injection apparatus, a chromatograph column, a He ion detector, and connecting tubes, a foreign gas (other than He) injection apparatus is installed between the sample injection apparatus and the detector. Mixing of the sample gas and foreign gas takes place readily, the sample gas is always maintained at a stable concentration range, and accurate measurements are possible, especially at low sample gas concentrations.

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HELIUM IONIZATION DETECTION APPARATUS

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Specifications

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1. Title of Invention: Helium Ionization Detection Apparatus

2. Scope of Patent Request

A helium ionization detection apparatus is composed of the successive connection of a gas supply, a sample injection apparatus, a column and a detector with connecting tubes. This setup is completed by the installation of a foreign gas injection apparatus either between the sample injection apparatus and the detector, or in the detector.

3. Detailed Explanation of Invention

The invention concerns a helium ionization detection apparatus which uses a helium ion detector in a gas chromatographic method. Specifically, the helium ionization detection apparatus is composed of the successive connection of a gas supply, a sample injection apparatus, a column and a detector with connecting tubes. This setup is completed by the installation of a foreign gas injection apparatus either between the sample injection apparatus and the detector, or in the detector.

The helium ionization apparatus generates helium atoms by the energy of electronic discharge and radioactive rays, which is used to ionize all other atoms or molecules. It is also used as the detector for the chromatograph. However, hydrogen, oxygen and nitrogen record a negative peak if we use helium gas with high purity. In order to avoid this situation, there is a technique where foreign gas such as hydrogen is added to the helium carrier gas in advance. In the past, as Fig. 1 shows, a foreign gas was added in the connecting tubes by the foreign gas injection apparatus (c) which is placed between

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*Numbers in the margin indicate pagination in the foreign text.

the gas supply (a) and the sample injection apparatus (c). Figure 2 shows that the sample injection apparatus (b) injects the sample by switching the connecting tubes (d) with a cock (e), while Fig. 2A shows the condition when samples are injected into a measuring device (f) and Fig. 2B shows the condition when samples are injected into the connecting tubes (d) from a measuring device (f). If the samples are injected under this condition, they proceed to the detector through the column without mixing with the foreign gas that is added from the foreign gas injection apparatus (c) in the connecting tubes. A negative peak will also be recorded. There is a strong tendency that is true especially in the case when the sample gas has a lower concentration rate. Thus, it is necessary that the portion of the sample gas be within the stable concentration range in order to obtain a normal positive peak on the detector. In the past, the position of the foreign gas injection apparatus was located in front of the sample injection apparatus. Therefore, we did not obtain a satisfactory condition for detection. In addition, in the example above the cock is cited as a sample injection apparatus; however, the same problem will occur when we conduct sample injection with the use of a gas-tight cylinder.

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Reflecting on the above problem, we find that this invention enables us to conduct an accurate detection regularly with the use of a lower sample gas concentration by facilitating the mixture of the sample gas and the foreign gas.

A detailed explanation of the invention follows, with the experiment described in figures; however, the invention is not limited to the experiment described below.

Figure 3 shows the whole composition of the invention. The gas supply (2), the sample injection apparatus (3), the column (4) and the detector (5) are series-connected by the connecting tubes. The measuring device (6) that is installed

in the detector (5) and the foreign gas injection apparatus (7) are set between the sample injection apparatus (3) and the column (4). The gas supply is filled with helium gas which is used as the carrier gas, and it proceeds to the detector (5) when the gas supply (2) opens. Accompanying the release of the gas supply (2), the foreign gas injection apparatus consecutively injects the foreign gas such as hydrogen. As the foreign gas injection apparatus we used a gas cylinder, a permeation tube, etc. In addition, the foreign gas means other than helium gas. Under the condition when the carrier gas and the foreign gas pass inside the connecting tube (1), 1 to 5 ml of the sample gas such as an atmospheric gas are injected by the sample injection apparatus which is the same shape as the one in Fig. 2. Since the foreign gas is consecutively injected, the sample gas and foreign gas mix easily when the sample gas passes through this section of the foreign gas injection apparatus. In the case when a 1 ppm concentration of sample gas is used, a 10 ppm concentration of foreign gas will be used. Since the sample gas is a small quantity, the concentration of the mixed gas will be nearly the same as the concentration of the foreign gas and this concentration rate enables us to obtain normal detection by the detector. We used as a fixed substance on the inside of the column a general hygroscopic chemical such as silica gel or a molecular sieve. Figure 4 shows the experimental example where the foreign gas injection apparatus (7) is set between column (4) and detector (5). Figures 3 and 4 show the generation of the helium atoms in the detector (5) which is done by the use of radioactive energy (7) of a radioactive element such as tritium. Figure 5 shows the foreign gas injection apparatus (7) installed in the detector. In this case, the mixed gas, the mixture of the foreign gas and helium gas, is injected by the foreign gas injection apparatus (7) which is installed inside the detector (5). The helium atom of the mixed gas is generated when we provide it with the energy from the

electronic discharge from the electrode. Under this condition we mix the mixed gas and the sample gas from column (4) in order to prevent impurities from lowering the function, which happens when the electrode directly contacts the sample gas.

Further, the analysis of the detection of the sample is based on the chromatographic method which specifies the quantity of the gas from the time at which the sample is injected to the time of the peak occurrence on the chromatogram. Moreover, the quantity of gas is based on the general chromatographic method which calculates the area at the peak. In the past, when a negative peak was obtained, the problem occurred because accurate calculation was not available.

In the experimental example above, I refer to the use of atmospheric gas as the sample gas. The gases with which the helium ionization detection apparatus is apt to record a negative peak are hydrogen, oxygen and nitrogen. From this point of view, it is clear that the invention is more effective when it is used to detect the gases listed above.

As stated above, the invention is comprised in such a way that the sample gas and the foreign gas mix easily, maintaining /247
the portion of the sample gas within the stable concentration range. Thus the detector is able to perform accurate detection and when the sample gas has a lower concentration rate, the invention is more effective.

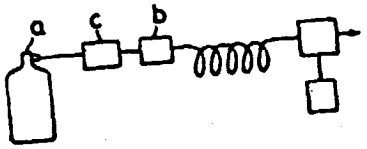
4. Brief Explanation of Figures

Figure 1 is an illustration of the whole composition of the example above. Figures 2A and 2B are illustrations of the above example of the enlarged composition of the sample injection apparatus. Figure 3 is an illustration of the whole composition of the experimental sample of the invention.

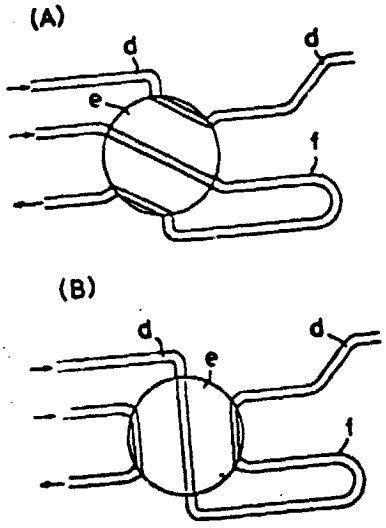
Figures 4 and 5 also illustrate the whole composition of the experimental example of the invention.

(1) Connecting tube
(2) Gas supply
(3) Sample injection apparatus
(4) Column
(5) Detector
(7) Foreign gas injection apparatus

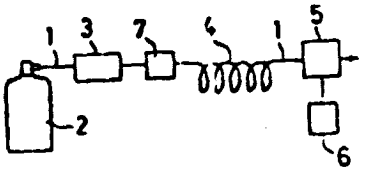
第1図



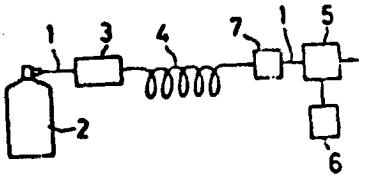
第2図



第3図



第4図



第5図