

## Slingerland

[15]     **3,656,277**

[45] **Apr. 18, 1972**

**[54] METHOD AND DEVICE FOR GAS CHROMATOGRAPHY**

|           |         |                        |        |
|-----------|---------|------------------------|--------|
| 3,063,286 | 11/1962 | Nerheim.....           | 55/197 |
| 3,150,516 | 9/1964  | Linnenborn et al. .... | 55/197 |

[72] Inventor: **Peter Slingerland, Pijnacker, Netherlands**

[73] Assignee: **Nederlandse Organisatie Voor Toegepast-Natuurwetenschappelijk Onderzoek Ten Behoeve Van Nijverheid, Handel en Verkeer, The Hague, Netherlands**

[22] Filed: Mar. 24, 1970

[21] Appl. No.: 22,184

[30] Foreign Application Priority Data

Mar. 28, 1969 Netherlands.....6904800

[52] U.S. Cl.....55/67, 55/197, 55/386

[51] Int. Cl. .... B01d 15/08

[58] **Field of Search** ..... 55/67, 197, 386

[56] **References Cited**

## UNITED STATES PATENTS

3,513,636 5/1970 Halasz et al.....55/197

## OTHER PUBLICATIONS

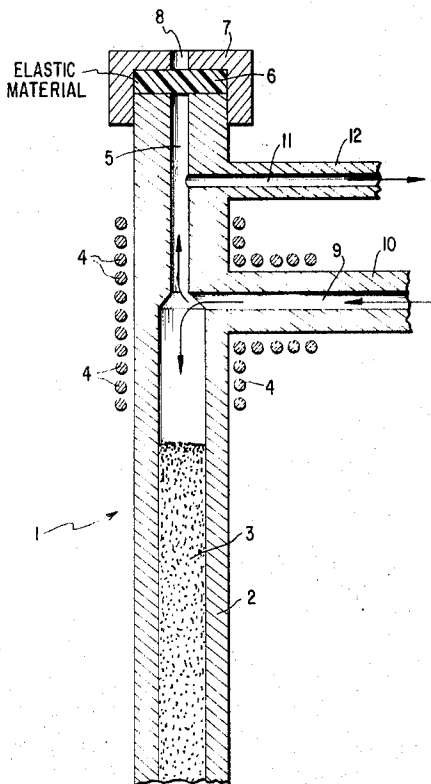
Brudeweck, et al., *Journal of Gas Chromatography*, May 1967, pp. 217-225.

**Primary Examiner—Charles N. Hart**  
**Attorney—Spencer & Kaye**

[57] **ABSTRACT**

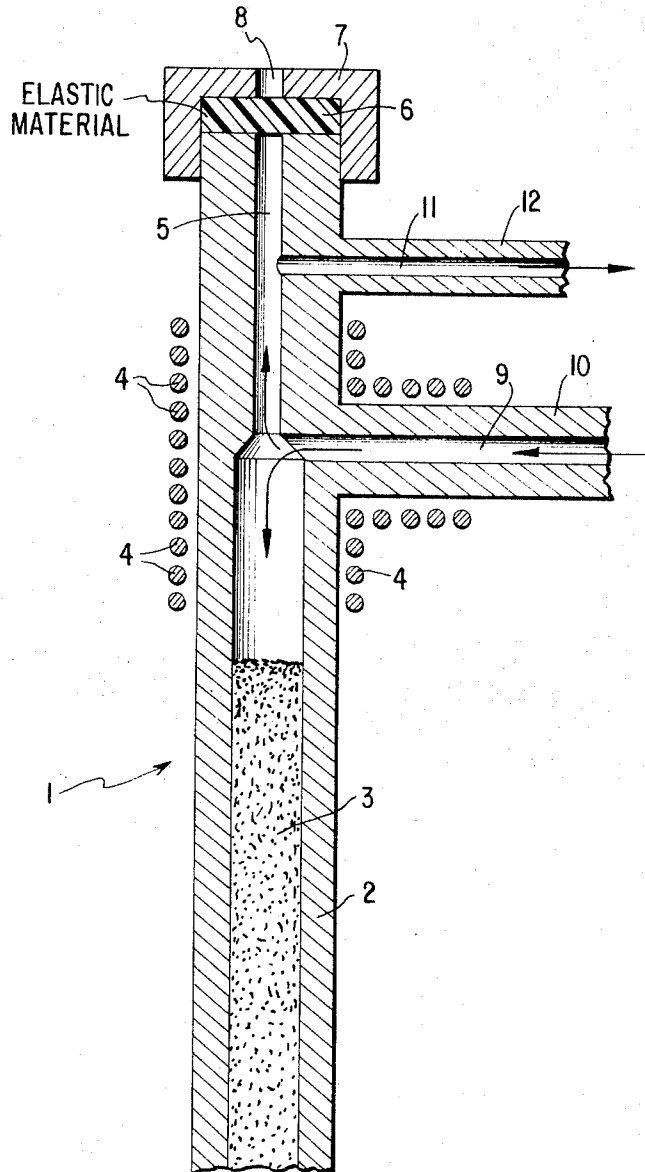
An improved method and device for gas chromatography in which the ill effect of septum-bleeding is eliminated by allowing a small stream of carrier gas to leak out of the top section of the column into the atmosphere at a location between the septum and the main stream of carrier gas.

## 2 Claims, 1 Drawing Figure



PATENTED APR 18 1972

3,656,277



INVENTOR

Peter Slingerland

BY *Spencer & Kaye*

**ATTORNEYS.**

**METHOD AND DEVICE FOR GAS CHROMATOGRAPHY**

The invention relates to a method for the separation of the substances present in a mixture, in which method a small quantity of the mixture is introduced into the separating column of a gas chromatograph by means of a needle syringe, whose needle is pierced through the septum which consists of an elastic material and which seals the top section of the column, and in which method the mixture injected is carried through the separating column with the aid of a carrier gas stream introduced at the top section of the column.

This method is the normal use of conventional devices for gas chromatography and is generally known in the art. It is also known in the art that in operating with a programmed proceeding of the column temperature and sensitive detectors a part of the measuring result may be composed of signals not originating from the substances in the sample injected. Such signals may very much interfere with the interpretation of the chromatogram.

The signals not related to the sample are often effected by substances emanating from the septum. Those cumulate at the top section of the column when the latter is rapidly being cooled at the end of a temperature programme and at a following temperature programme they behave like a sample injected.

Attempts have been made already to solve this troublesome problem of septum bleeding by composing the septum of three layers whose intermediate layer consists of a very flexible material and yields a good sealing, and whose two other layers consist of a less flexible material that emits fewer volatile substances. Another attempt to come to a solution of the problem is heating the septum beforehand in vacuo or in an inert stream of gas so as to remove the volatile substances. Experience has taught, however, that neither of these methods is satisfactory.

It is the object of the invention to provide a method by means of which the problem of septum bleeding can be solved completely in a simple and inexpensive way.

For this purpose the invention is characterized in that the gas stream is carried in and through the column in such a way that a section of the column comprising the septum is situated outside the gas stream and in that via this section of the column a small part of the gas stream is discharged from the column.

By this implementation of the method it is achieved that volatile substances released from the septum, are taken along and conveyed outside the column by the part of the gas stream that is immediately discharged from the column, before they could have come into contact with the gas stream. Consequently no particular materials, compositions or pretreatments need to be applied for the septum, but an inexpensive quality will suffice; the volatile substances being released do not reach the detector and are not signaled by the latter.

The amount of the part of the gas stream introduced that is immediately discharged at the top section of the column together with substances, if any, emanating from the septum, is not essential. On discharging quantities of 1 to 10 ml/min, fully satisfactory results were obtained. Besides, the discharge of gas has no effect whatsoever on the operation of the gas chromatograph.

The invention also relates to a gas chromatograph for the implementation of the method described, whose injector, which forms the top section of the separating column of the gas chromatograph, is provided in the usual way with a feed-through channel for gas and an opening sealed by the septum consisting of an elastic material. The gas chromatograph according to the invention is characterized in that the said opening is situated in a section of the injector that branches off from the feed-through gas channel and in that this part of the injector is connected with the environment by a small gas discharge opening.

In order to enable adjustment of the gas discharge to a suitable quantity, the discharge opening is preferably provided with a fine adjustment screw or with a capillary whose length is chosen in accordance with the discharge velocity desired.

The invention will be further elucidated below with reference to the drawing, which shows an embodiment of the injector section of a gas chromatograph.

Injector 1 forms the top section of column 2 of the gas chromatograph. Column 2, which consists of a glass or metal tube, is filled up to injector 1 with the fine-grained adsorbent 3. Column 2 is situated in a thermostat (not shown in the drawing), whose temperature can be adjusted to a constant value, or be programmed. Injector 1 is provided with heater 4 so as to be heated up to about 25° to 50° C above the column temperature.

Injector 1 comprises several channels. Of these, channel 5 is sealed by septum 6, consisting of a disc of an elastic material, which is pressed down by nut 7. The latter is provided with opening 8, which is situated in line with channel 5.

Channel 9 is situated in cross piece 10, to which a hose or tube for the supply of the carrier gas can be connected.

Channel 11 in cross piece 12 communicates with channel 5 between channel 9 and septum 6. Via an adjusting device, if desired, it forms an open communication of channel 5 with the environment.

When a gas chromatogram has to be made of a mixture of substances, a sample of this mixture is brought onto column filling 3, with the aid of a needle syringe, whose needle is pierced through septum 6 via opening 8. Upon subsequent withdrawal of the needle syringe, septum 6 is sealed by the elasticity of the material. In the meantime a gas stream is fed through column 2 via channel 9, and the column thermostat and injector heater 4 have been adjusted to the temperature or temperature programme desired.

Because of the high temperature of injector 1, volatile substances are emanating from the material of septum 6. Without any further measures these substances would mix with the gas stream that streams from channel 9 into column 2, and together with components of the mixture injected, be fed through column 2, the result being that the gas chromatogram would also contain the signals of these volatile substances. However, through the presence of channel 11, a small part of the gas stream that is fed through channel 9, is released in a reverse flow into the atmosphere via channel 5 and channel 11. This gas stream carries the substances emanating from septum 6 into the atmosphere. The flow, directed towards septum 6, in the part of channel 5 that lies between channels 9 and 11, guarantees that nothing of these substances can reach column 2.

I claim:

1. A method of operating a gas chromatographic column having a channel extending between a septum of elastic material and a means for separating substances present in a mixture, comprising maintaining in the portion of the channel adjacent the means for separating a flow of carrier gas into the means for separating while maintaining in a section of said channel between said portion and said septum a reverse flow of carrier gas toward the septum and through a channel outlet.

2. A gas chromatographic column comprising separating means for separating substances present in a mixture, a septum of elastic material, channel means sealed to said septum and disposed for forming a channel extending between said separating means and said septum, means arranged relative to said channel means for introducing a carrier gas into said channel means, whereby carrier gas may flow through said separating means, and conduit means arranged relative to said channel means for creating a reverse flow of a part of said carrier gas away from said separating means, toward said septum, in said channel means.

\* \* \* \* \*