AN IMPROVED TECHNIQUE FOR BACK-FLUSHING GAS CHROMATOGRAPHIC COLUMNS

D. R. DEANS
Imperial Chemical Industries Limited, Heavy Organic Chemicals Division, Billingham, Durham (Great Britain)
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INTRODUCTION

Back-flushing of gas chromatographic columns is a procedure that is widely practised when the sample to be analysed contains components that are eluted after the compounds of interest. Its virtue is that it can cut down analysis time and it may be used to protect the detector or a second column from contamination with undesirable compounds.

Back-flushing may be used to obtain a separation of compounds where a straight through system fails. Two columns with different stationary phases are used in series, the first column separating the compounds into two groups, A and B. The group with the longest retention time, B, is back-flushed off the first column. The separation of group A is completed on the second column. If back-flushing is not used, one or more of the compounds in group B may interfere with the final separation of group A on the second column.

THE TRADITIONAL METHOD OF BACK-FLUSHING

Fig. 1 shows a schematic flow diagram of a typical back-flushing system. With the taps in the position shown, the column operates normally. When both taps are turned through 90° the first column is back-flushed and the second column continues on forward flow. Both four-way taps have to be at the column temperature, the inlet tap, in order to avoid contamination of the inlet carrier gas with purge material. The needle valve is adjusted to maintain the same flow of carrier gas through the second column, when the first column is being back-flushed.

A number of variations of this back-flushing system are used but they are all likely to suffer from reduced separation efficiency or peak distortion compared with a straight through system. The source of the trouble is the lack of suitable multiway taps for use in the sample path. The taps available usually have a comparatively large dead volume, which if they are used with packed columns tends to broaden the peaks, and which if they are used with capillary columns destroys the separation efficiency. A technique of adding carrier gas to the flow through a four-way tap has been used to reduce the effect of the dead volume[1]. The technique, while useful, is limited in its applicability and it dilutes the sample to the detector.

The difficulties associated with multiway taps are not confined to the problem of dead volume; the tap has to withstand the column conditions of temperature and pressure without leaking, yet the use of grease is undesirable as absorption of components can occur. A constructional drawback is that the barrels of the taps have to be situated in the oven while they must be capable of being operated from outside the oven.

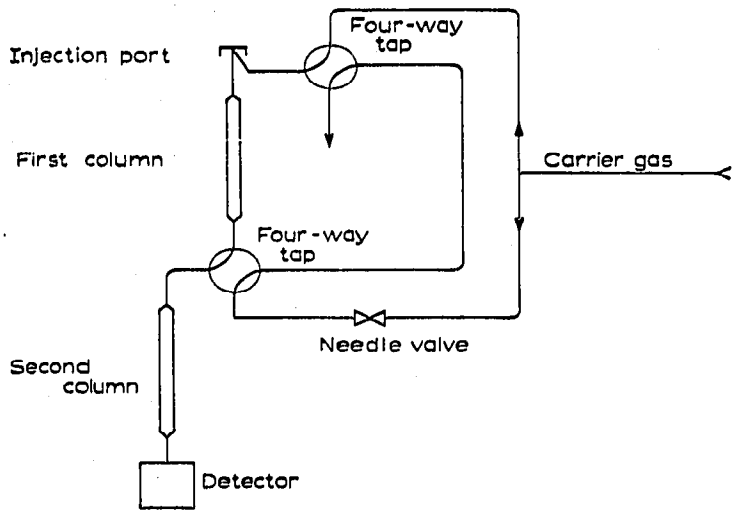

Fig. 1. Schematic flow diagram of a typical back-flushing system.

DESIGN AND CONSTRUCTION OF THE IMPROVED METHOD

A system of back-flushing has been developed which avoids the use of taps in the sample path or in the oven. Fig. 2 shows the scheme applied to two packed or two capillary columns and Fig. 3 that applied to one packed and one capillary column.

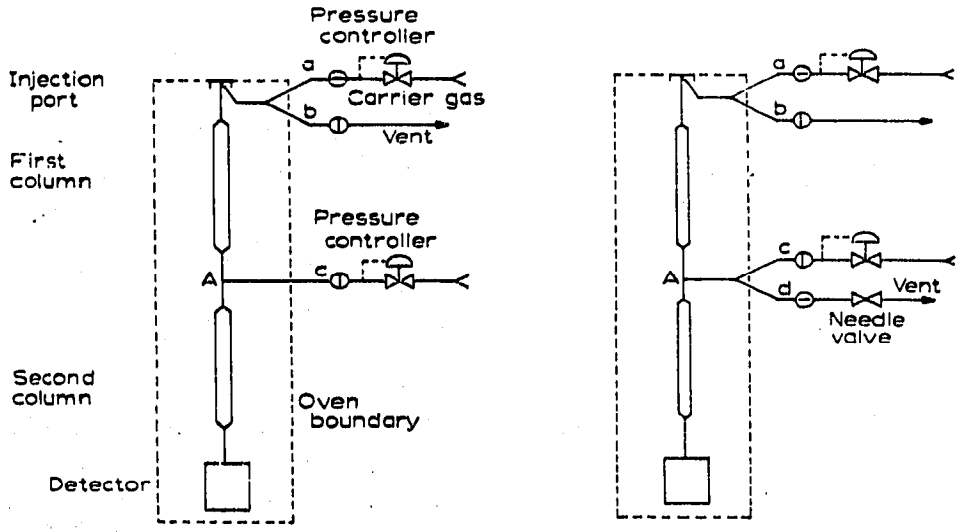

Fig. 2. Back-flushing system applied to two packed or two capillary columns.

Fig. 3. Back-flushing system applied to one packed and one capillary column.

Metal columns have been used in all cases but there is no obvious reason why glass should not be used. To keep the volume of the T-piece at A small, T-pieces made of brass or stainless steel $^3/_4$ in. long were used with a 42 thou hole to take a 20 thou I.D. capillary column, or $^1/_8$ in. I.D. holes to take $^1/_8$ in. O.D. connections to packed columns. Spring loaded glass taps have been used successfully up to an inlet pressure of 30 p.s.i.g. Edwards pressure controllers type V.P.C.1 have been found satisfactory for regulating the carrier gas inlet pressures.

METHOD OF OPERATION

When a sample is injected, tap a (Fig. 2) is open and taps b and c are shut. Carrier gas flowing through tap a carries the sample through the first column. When the components of interest have passed on to the second column taps b and c are opened and tap a is shut. Carrier gas now enters through tap c and divides into two streams at point A. One stream back-flushes the first column carrying unwanted components to atmosphere through tap b. The other stream flows through the second column, where separation of the components already on this column is completed.

Base line disturbance on the recorder trace on turning the taps is avoided by adjusting the two pressure controllers so that the pressure at point A is the same irrespective of the direction of flow in the first column. This is achieved by setting the pressure controller before tap a to give the optimum flow conditions for the particular separation being studied and then adjusting the pressure controller before tap c until the base line kick on changing from forward flow to back-flush is eliminated. To avoid pressure build up behind closed taps the pressure controllers used should incorporate a bleed system, enabling the pressure to be controlled when there is no flow along the line.

The method of operation and setting up is slightly different when the scheme is applied to a packed first column and a capillary second column.

On forward flow the taps are set as shown in Fig. 3. Carrier gas entering through tap a is split at the end of the first column, the major part of it going to atmosphere through tap d and the needle valve. This makes it possible for the optimum flow rates to be maintained in both the packed and capillary columns; in other words, the packed column and vent are acting as the conventional sample splitter. Back-flushing is achieved by closing taps a and d and opening taps b and c.

In setting up this system a pressure gauge connected by a capillary T-piece to point A is useful. The pressure at point A to give the optimum flow rate through the capillary column is first determined. The inlet pressure and the needle valve setting are then adjusted on forward flow to maintain the pressure at point A and to give the optimum flow rate through the packed column.

THE EFFECT OF COLUMNS WITH UNEQUAL PNEUMATIC RESISTANCE

Where both the first and second columns have the same pneumatic resistance, the pressure difference from the inlet to point A will be the same as the pressure difference from point A to atmosphere at the detector. When the first column is back-flushed the pressure at point A remains unchanged but the pressure at the injection point drops to atmospheric. The back-flush gas flow rate will be the same

as the forward flow rate because the pressure difference across the first column will be the same but in the reverse direction.

As a consequence the time required to back-flush off any unwanted compounds will be the same as the time elapsed from injection to starting to back-flush.

If the pneumatic resistance of the first column is less than that of the second column the rate of reverse flow will be greater than the rate of forward flow as the pressure difference from point A to atmosphere will be greater than the pressure difference from the inlet to point A. This will not affect the operation of the system in any way except that unwanted compounds will be back-flushed off the first column more quickly. A limitation may be reached, when the pneumatic resistance of the first column is much less than that of the second, where the back-flush flow becomes so large that the pressure controller before tap c is unable to maintain the required pressure. In this case the back-flush flow rate can easily be reduced by introducing a needle valve or other restriction into the purge line before tap b.

Where the pneumatic resistance of the first column is greater than that of the second the operation will not be affected in any way except that the time required to back-flush off unwanted components may become appreciable. In order to increase the back-flush flow rate a choice of methods is available; probably the easiest is to introduce a restriction between the second column exit and the detector; a short length of capillary restricted by flattening has been found satisfactory. Another method which gives excellent results is to connect the purge line to a controlled vacuum system; a vacuum pump and Edwards V.P.C.1 controller have been used. A third method is to operate the system with the detector above atmospheric pressure; an Edwards V.P.C.1 controller, on the exit of a katharometer or a flame detector, has been used. Any of these methods may be used where a second column is not required.

ADVANTAGES OF THE IMPROVED TECHNIQUE

The advantage of this method of back-flushing over the conventional use of multiway taps are:

1. The dead volume associated with the use of taps in the sample path is eliminated. Consequently capillary columns may be back-flushed without loss of efficiency.

2. The absence of taps in the sample path eliminates the possibility of absorption of sample components on tap lubricants or low friction plastics.

3. The taps used are outside the oven, at ambient temperature, consequently no special design of tap or oven is required and the system is easy to add to an existing chromatograph.

This method has been used successfully in many applications including the analysis of trace impurities in phenols and esters and for removal of heavy materials from reactor products during analysis.

SUMMARY

A technique of back-flushing gas chromatographic columns is described which avoids the use of taps in the sample path. The dead volume and the danger of ab-

sorption of compounds in the tap lubricant normally associated with back-flushing systems is eliminated. The taps used can be located outside the oven at ambient temperature, consequently the apparatus is easy to construct and existing chromatographs can be readily modified. Capillary and other high efficiency columns have been successfully back-flushed using this technique.

REFERENCE

1 E. R. FITT, *Anal. Chem.*, 35, No. 3 (1963) 419.