Journal of Chromatography, 94 (1974) 276–278

CHROM. 7413

Note

A device for low-pressure gas injection in gas chromatography

H. NIENHUIS and M. SEVENS

Philips Gloeilampenfabrieken, Light Division, Eindhoven (The Netherlands)

(First received December 17th, 1973; revised manuscript received February 12th, 1974)

Many types of injection valve for introducing a gas sample into a gas chromatograph have been described[1–6], but it has always been a problem to analyze a gaseous sample at a very low pressure. The sudden reduction in the pressure in the inlet system after introducing a sampling loop, filled at a low pressure, results in an output signal that is difficult to interpret, and there is always a high, long tailed peak due to the decrease in pressure. Therefore, a system has been devised in which it is possible to eliminate the pressure difference between the sampling loop and the inlet side of the column.

EXPERIMENTAL AND RESULTS

The technique of Cundall *et al.*[2] was used in a special valve (Fig. 1) made of stainless steel. It consists of a barrel in which a key moves laterally, with three fixed positions. In the key are seven grooves with O-rings. The clearance between the barrel and key is very small (*ca.* 100 μm). Inside the key is an extra valve, which enables this device to be used for introducing gas samples from high-pressure cylinders. When analyzing gas samples from small glass ampoules, the ampoules are placed in a sample holder, in which it is possible to crack the glass by screwing a hardened steel point into the wall of the ampoule after evacuating the sample holder.

This valve is used with a double-column gas chromatograph (Perkin-Elmer 900). An important advantage of this valve is its vacuum tightness. Because of the low vacuum tightness of the original valve of the Perkin-Elmer gas chromatograph, it was not possible to operate the system under vaccum.

In Fig. 1, the valve is shown in its three positions.

In position 1, the carrier gas flows directly to the column (1–4). The sampling loop and the sample holder are connected to the vacuum line.

In position 2, the carrier gas flows through the same route (1–4). The ampoule can now be opened and the sampling loop is filled by expansion with the gas to be analyzed (3–5).

In position 3, the pressure in the sampling loop is equalized with the pressure at the inlet side of the column (1–5). The gas from the sampling loop is then injected into the column (1–5–4).

If the volume V is sufficiently large with respect to the volume of the sampling loop, then there will be a smaller decrease in the pressure at the inlet side of the column,

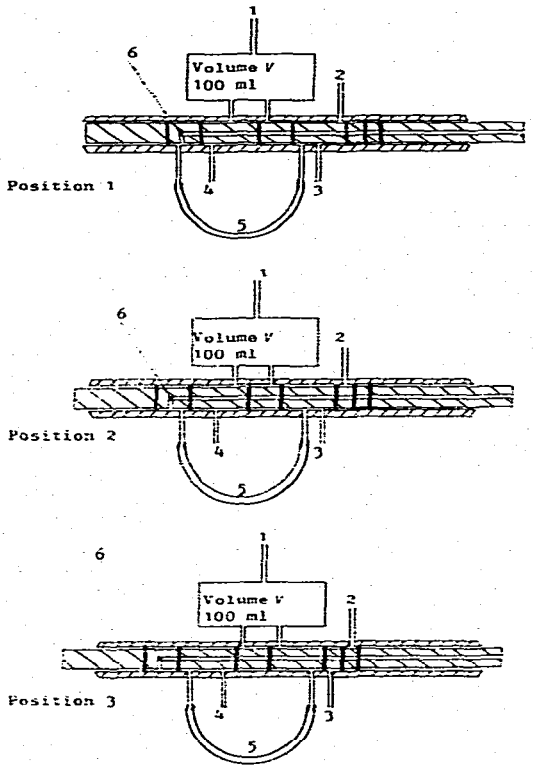

Fig. 1. The three positions of the injection valve. 1 = carrier gas; 2 = vacuum system; 3 = sample holder; 4 = column; 5 = sampling loop; 6 = extra valve.

which will result in a decreased long tailed peak, the occurrence of which has already been mentioned. In this case, a volume of 100 ml is used and the maximum volume of the sampling loop is 5 ml.

This valve is mounted in one of the column lines of the gas chromatograph. Each of the column lines has a flow regulator. Because of the presence of the volume V in one line, the flow regulation is not equal for both lines, especially when a temperature programme with a variable column temperature is used. Therefore, another identical volume was inserted in the other line so as to ensure that there was no shift of the baseline, even when the temperature of the column varied.

With this system, it is possible to measure gas pressures down to $2 \cdot 10^{-4}$ torr in a sampling loop of 5 ml. The carrier gas is helium at a flow-rate of 30 ml/min, and a hot-wire detector is used.

It is also possible to analyze gases from high-pressure cylinders. The cylinder is then connected in the place of the sample holder. The valve inside the key is open. With the key in position 1 and a closed vacuum line, the gas is led through the sampling loop and by switching the key to position 3 via position 2, the gas from the sampling loop is injected into the column.

REFERENCES

1 D. N. Glew and D. M. Young, *Anal. Chem.*, 30 (1958) 1890.
2 G. L. Pratt and J. H. Purnell, *Anal. Chem.*, 32 (1960) 1213.
3 W. H. Wahl and G. G. Meisels, *Rev. Sci. Instrum.*, 36 (1965) 1508.
4 R. B. Cundall, K. Hay and P. W. Lemeunier, *J. Sci. Instrum.*, 43 (1966) 652.
5 D. J. Malcolm-Lawes and D. S. Urch, *J. Chromatogr.*, 44 (1969) 609.
6 J. C. Cavagnol and W. R. Betker, in L. S. Ettre and A. Zlatkis (Editors), *The Practice of Gas Chromatography*, Interscience, New York, 1967, p. 107.