Journal of Chromatography, 120 (1976) 197–199

CHROM. 8874

Note

Injection system for small volatile samples in gas chromatography

ROLF JÖNSSON and JAN ÅKE JÖNSSON
Department of Analytical Chemistry, Chemical Centre, University of Lund, S-220 07 Lund 7 (Sweden)
(Received October 17th, 1975)

Two precision gas chromatographs for studies of column phenomena were built in this department[1–3], both of which utilize a system for the injection of small amounts of simple samples in a volatilized form, designed to give an injection time with high precision and fair reproducibility. The system is easily automated.

In Fig. 1, the present version of the system is shown. The sample, which must be in liquid form, is housed in a thermostatically controlled vessel (most commonly a test-tube with a ground-glass joint). The vapour pressure over the liquid is adjusted by dilution with a non-volatile solvent or heating.

A stream of gas (preferably of the same type as the carrier gas) is fed through the sample vessel, after which it contains sample in a volatilized form. This gas stream, hereafter called the sample gas, is led to a system of two three-way solenoid valves of

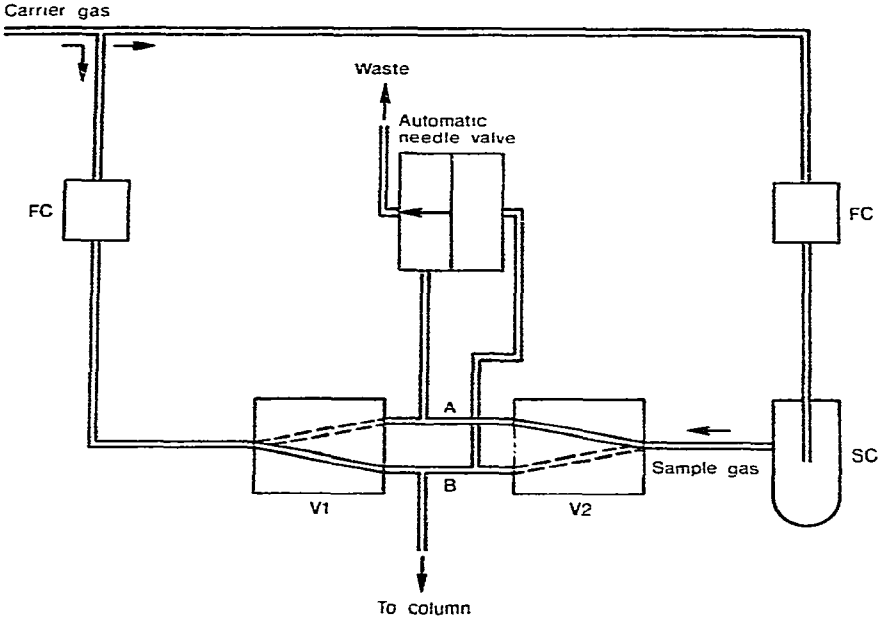

Fig. 1. Injection system. FC = flow controllers; SC = sample chamber; V1, V2 = solenoid valves.

the type 95HV from Hoke-Tomco (Cresskill, N.J., U.S.A.). The function of these valves is to switch either the sample gas stream or the carrier gas stream to the column. In the normal position, the carrier gas is led to the column and the sample gas to waste via an automatic needle valve (see below). In the other position, the sample gas is led to the column and the carrier gas to waste.

A pressure difference between points A and B in Fig. 1 will influence the injection. To check this effect, the automatic needle valve in Fig. 1 is replaced with an ordinary needle valve, and it is then possible to adjust the pressure at the sample side (point A) of the valves. In Fig. 2, the amount of sample injected (measured as the peak height) is plotted against the opening time of the injection valves for three cases: (1) when the pressures at A and B are equal; (2) when A has higher pressure than B (6 mmHg); and (3) when B has higher pressure than A (5 mmHg). It can be seen that the amount injected is dependent on the pressure difference and is proportional to the opening time of the valves when the pressures are equal. Therefore, it is important that the pressure of the sample side (A) is regulated to the same value as the column head pressure (B). In the first equipment[1], this was effected manually with the needle valve at the waste outlet. However, when the column head pressure is changed, for example as a result of a change in flow or temperature, the sample stream pressure must also be changed. In the later, more automated equipment[3], this regulation of pressure was effected automatically by means of a special valve. This valve is part of a flow controller taken from a PE 800 gas chromatograph. It consists of two chambers separated by a membrane, to which the regulating needle is attached. When this valve is connected as shown in Fig. 1, the pressure at the left-hand side of the membrane is kept very near the pressure at the right-hand side, thus making the pressures at the

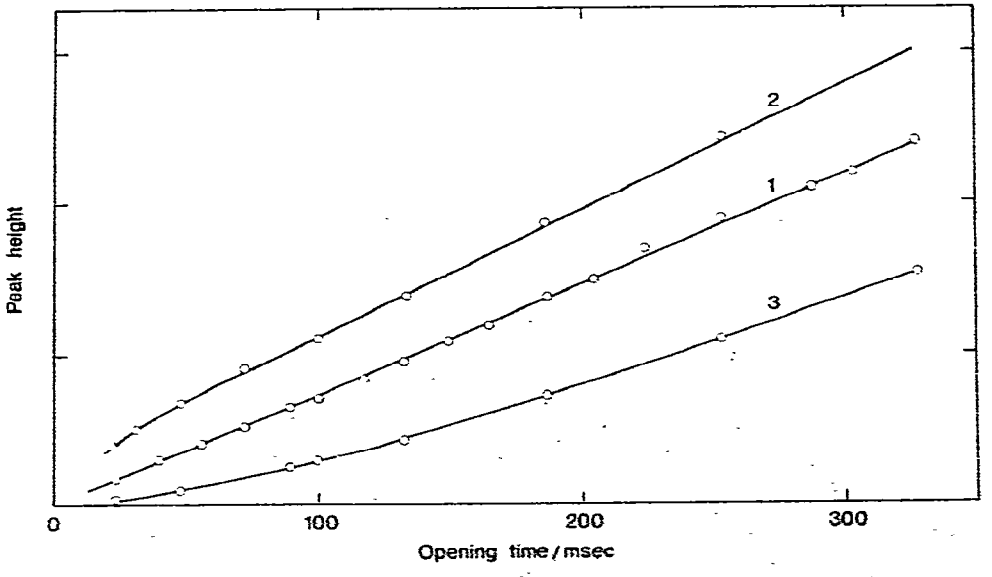

Fig. 2. Influence of pressure differences on the injection. For 1, 2 and 3, see text.

sample gas side and the column side almost equal. This simple arrangement worked satisfactory.

This type of injection system has been used in several investigations[1,4–6]. Its main advantages are that it is easy to change the sample, to adjust the amount injected (both by changing the opening time of the valves and by adjusting the dilution of the sample) and to operate by means of automatic control.

An extension of the present system is planned in which the simple test-tube is replaced with a system of 10 test-tubes connected via solenoid valves to provide for automatic change of sample type.

ACKNOWLEDGEMENT

We express our gratitude to Dr. Lars Haraldson for valuable discussions.

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