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Note

A simple on-column injector for capillary gas chromatography

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Capillary columns are widely used in gas chromatography (GC) because of their high resolving capacity. Split, splitless and on-column techniques are used to introduce the sample.

A conventional vaporizing GC injector can easily be converted into a split/splitless injector. However, it cannot be used for on-column injection, which is regarded as the best injection technique for quantitation of sample components with a wide boiling point range[1]. In addition thermolabile compounds are less susceptible to degradation at the low injection temperatures normally encountered with on-column injection[2].

More- and less sophisticated on-column injectors have been reported[2–6] and some are commercially available. Although the principles of on-column injection are relatively simple, the design of the injectors is often complicated and they are expensive. This has led us to design an easily built, low-cost on-column injector, based on the principle of maintaining constant pressure at the column head during injection[3].

Construction and operation

The injector (Fig. 1) consists of a glass inlet tube, a modified reducing union for gas tubings, a "buffer tank" for the carrier gas and a directing sleeve to lead the injection needle into the column.

At one end of the glass inlet there is a constriction to centre the injection needle (0.17 mm fused silica) before entering the column. The other end is sealed with a glass stopper which is connected to the injector via a PTFE tube held firmly to the glass walls by constrictions[7]. Two holes in the walls of the glass tube allow the carrier gas to enter the capillary column head. Two constrictions in the central part of the tube prevent pressure drop during injection.

An 1/8–1/16 in. union (Scientific Glass Engineering, U.K.) was modified in the following way. A 2-mm side hole was drilled in the body of the union. The 1/8-in. hole was enlarged to 4 mm and deep enough to pass the 2-mm side hole, the outer part of which was expanded to receive an 1/8-in. metal tube for the carrier gas.

The carrier gas is regulated by a constant-pressure regulator and passes via a buffer tank (30 cm × 1/4 in. metal tube) before being introduced into the injector. It is important that the carrier gas is not restricted by any narrow parts between the pressure regulator and the injector.

The directing sleeve is made from a piece of 1/16 in. stainless-steel capillary

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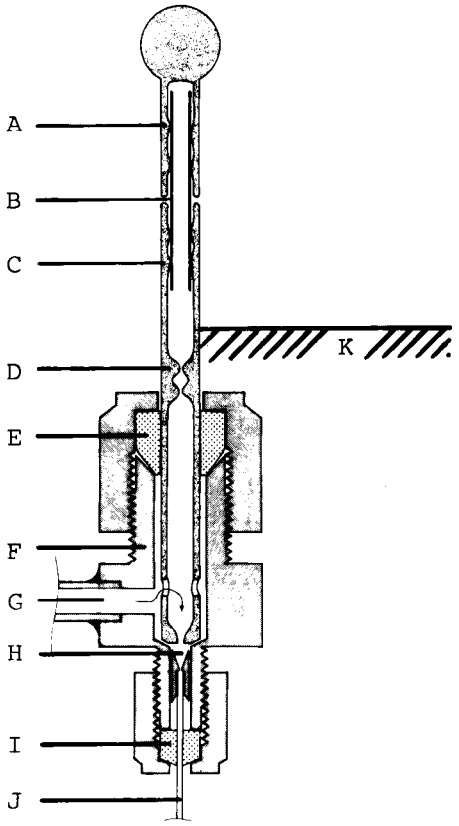

Fig. 1. On-column injector for capillary columns. A = Glass stopper; B = PTFE tube; C = 3-mm glass inlet tube with two holes (I.D. = 1 mm) in the wall; D = constrictions (I.D. = 0.20 mm); E = 1/8-in. Vespel ferrule; F = 1/8–1/16 in. union; G = carrier gas inlet; H = directing sleeve; I = graphite ferrule; J = capillary column; K = oven wall.

tube. The lower part is enlarged to receive the capillary column. The upper part is recessed on a lathe with a tapered scalpel. The sleeve is mounted on the capillary column and the assembly is centered with the help of a clean metal thread. The directing sleeve should not make contact with the inlet glass tube.

The injector and gas buffer tank are kept inside the column oven. Only the injector head protrudes from the column oven. All metal parts that are heated consist of stainless steel and are silver brazed.

After removing the glass stopper, the injection needle is inserted into the double constrictions in the glass tube and led via the bottom constriction into the final directing sleeve and further into the column.

Performance

The injector is based on the principle of maintaining constant pressure at the column head during injection. The double constrictions in the glass inlet tube and the buffer tank prevent rapid pressure changes at the column head when removing

the glass stopper. When connecting a pressure gauge instead of the column, the pressure drop was found to be 4.2% of the initial value (8 p.s.i.) as the glass stopper was removed. The sample loss due to back-flow through the injector will therefore be minimal. This means that the dead volume of the injector is not of importance.

The system was tested for back-flow in the following ways. (1) The injector was kept open during a full chromatographic analysis. The retention times of the sample components were on average 4.4% longer due to the reduced pressure. With the precision given in Table I, no loss of sample was noticed. (2) The syringe body and the glass inlet tube of the injector were connected to a piece of silicon tubing for 20 sec after injection. Equal amounts of the sample components were detected with and without this seal.

The reproducibility was tested by injecting 2-μl portions of C_{10}–C_{36} alkanes in *n*-octane (Table I). The retention times varied by less than 1% except for the first alkane. The fact that, the detected amounts varied more than has been shown by other authors[3] was probably due to variances in the injected sample volumes.

Two injectors are routinely used with 0.2 and 0.3 mm I.D. fused-silica columns at our laboratory, where biological samples are analysed for chlorinated organic residues (Fig. 2). They are accurate and fulfil our demands on the injection system. A similar injector is also routinely operated with a 1.6 mm O.D. SCOT glass column.

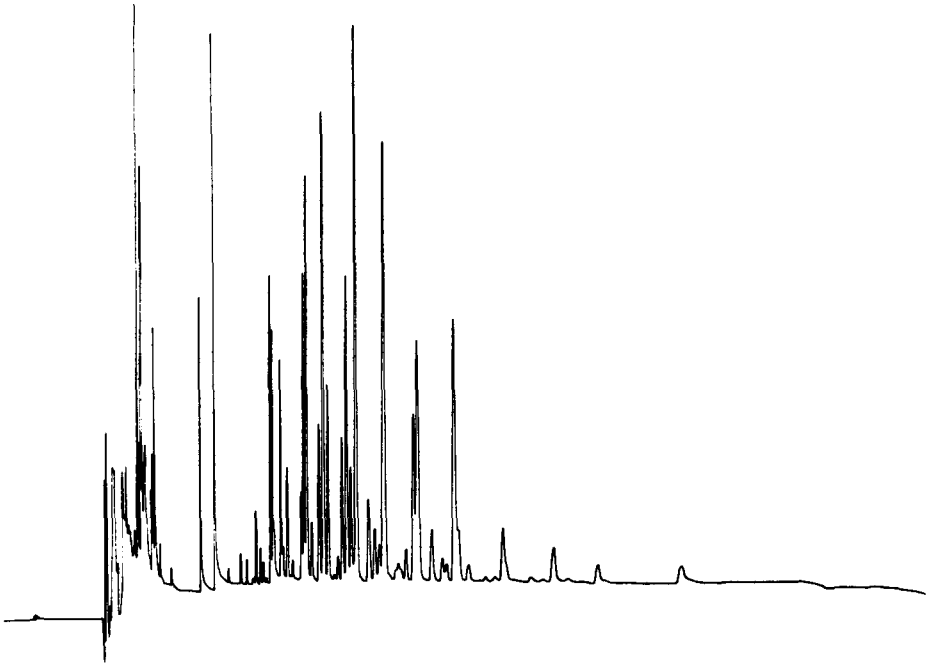

Fig. 2. On-column injection of 1-μl Clophen A 50 (1 ng of polychlorinated biphenyl mixture). Chromatographic conditions: Varian 3700 GC-ECD; column, SE 54, 15 m × 0.3 mm I.D. fused silica; carrier gas, hydrogen, 1.8 ml/min (p = 5 p.s.i.); make-up gas, nitrogen, 30 ml/min; column oven temperature, 80°C (1 min), then at 15°C/min to 180°C.

TABLE I

PRECISION OF THE CHROMATOGRAPHIC SYSTEM

Number of determinations, $n = 12$. Chromatographic conditions: Varian 3700 GC-FID; column, S.G.E. 0.25-μm QC2/BP5, 23 m × 0.2 mm I.D. fused silica. carrier gas, hydrogen, 1.0 ml/min ($P = 8$ p.s.i.); make-up gas, nitrogen, 30 ml/min; fuel gases, hydrogen, 30 ml/min, air, 300 ml/min; column oven temperature, 50°C (5 min), then at 10°C/min to 300°C; detector temperature, 320°C.

Alkane	*Average retention time (min)*	*Rel. S.D. (%)*	*Average amount (area units)*	*Rel. S.D. (%)*
C_{10}	5.33	2.4	2350	8.3
C_{12}	9.71	0.8	2525	7.3
C_{14}	12.78	0.7	2763	7.2
C_{16}	15.31	0.5	3143	8.0
C_{24}	23.08	0.2	2039	7.3
C_{28}	26.10	0.1	1702	8.0
C_{32}	28.79	0.1	1964	7.7
C_{36}	31.38	0.1	1693	8.6

We have not found it necessary to install a special cooling system for the injector; due to its low mass, and hence low thermal inertia, it is ready for injection when the temperature of the column oven has returned to the start temperature after a chromatographic analysis using temperature programming. Consequently, this on-column injector should be particularly valuable as a low-cost alternative in upgrading GC instruments not originally constructed for on-column capillary GC.

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