Journal of Chromatography, 172 (1979) 366–370

CHROM. 11,697

Note

Simple trapping system for collecting fractions from a gas chromatograph

JOHN F. MARCH*

Food Research Institute, Agricultural Research Council, Colney Lane, Norwich NR4 7UA (Great Britain)

(Received December 8th, 1978)

It was required to trap fractions of derivatized amino acids and associated components from a Pye Unicam 104 gas chromatograph, but the standard procedure was found to be inadequate.

The normal splitter used in the Pye Unicam instrument utilized a stainless-steel sinter to restrict the column effluent in one arm of the splitter. This type of splitter tended to cause degradation of the samples being studied and was replaced with a simplified, but much more effective device. Based on experience with the total trapping technique described by Swoboda[1], and the heated line technique described by Wooley[2], a modified procedure was developed. The splitter consisted essentially of a T-junction immediately following the column exit, and the split in effluent stream was controlled by a capillary tube inserted up into the jet of the flame-ionization detector (FID). By varying either the length or the internal diameter of the capillary tube, or both, a range of flow-rates could be produced. To minimize sample breakdown, I was anxious to use glass surfaces, and for all except the T-joint, the FID insert and the jet itself, this was achieved. The other surfaces were stainless steel. The delivery line to the trap was heated throughout its length to prevent condensation prematurely in the line. The hot emergent gas stream was directed on to the cold bottom inside surface of the collection tube. A high differential temperature was maintained between the bottom of the tube and the upper part. This ensured condensation of the sample well, within the tube.

EXPERIMENTAL

The splitter set up for the gas chromatographic (GC) column effluent is shown in Fig. 1. The capillary tubing used was of approximately 0.2 mm I.D., and a length of 130 mm gave a splitting ratio of 11.1 in favour of the trap at the working temperature (280°). This ratio was directly dependent on the operating temperatures and varied greatly if the outlet line temperature fell relative to the detector temperature. For example, when the outlet line heaters were off, the splitting ratio changed to about 40:1 in favour of the trap.

The collection system is shown in Fig. 2. The glass-lined metal tubing (GLT

* Present address: John Innes Institute, Colney Lane, Norwich, NR4 7UH, Great Britain.

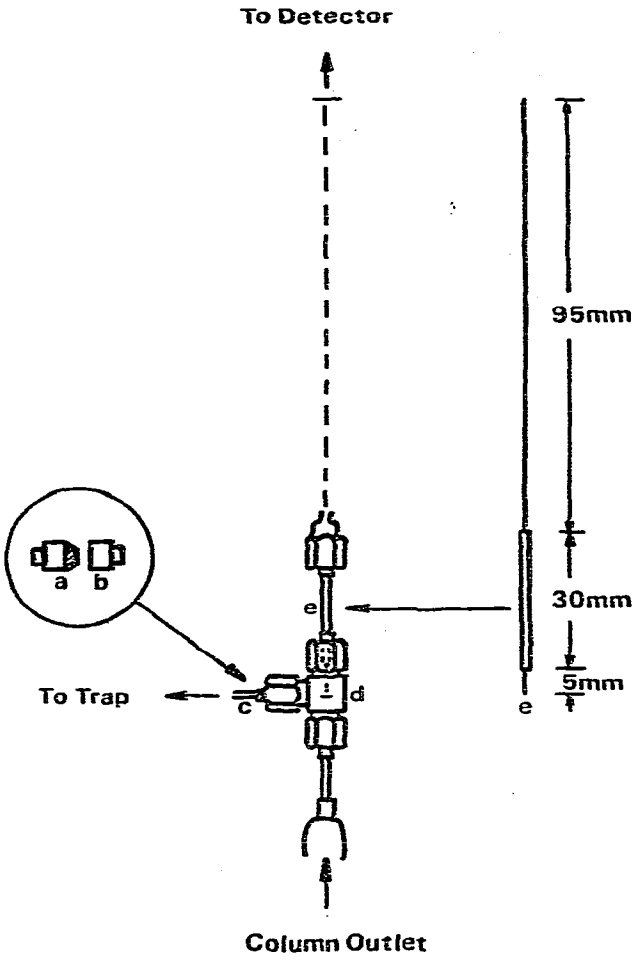

Fig. 1. Splitter assembly using a standard stainless-steel $^1/_{16}$-in. T-joint and a stainless-steel capillary jet insert. a = Modified 1/16-in. stainless-steel ferrule (the hatched part was filed off); b = standard 1/16-in. Graphlok ferrule, which fitted in place of the section filed off from a (this allowed a secure connection between the glass-lined tubing and the T-joint); c = glass-lined tubing leading to the trap; d = standard 1/16-in. stainless-steel T-joint placed between the column outlet and the detector; e = jet insert shown separately, and in position connected to the T-joint (the "insert" consisted of a 130-mm continuous length of capillary tubing with a 30-mm sleeve of standard 1/16-in. stainless-steel tubing silver-soldered 5 mm from one end; this arrangement allowed the "insert" to be connected between the detector and the T-joint with standard fittings). The column outlet was modified so that the terminal point was 35 mm lower than the standard analytical column used in the Pye Unicam 104 chromatograph. This allowed room for the T-joint to be fitted.

was obtained from Scientific Glass Engineering (London, Great Britain) and was the standard 1/16-in. O.D. and 0.7 mm I.D. The collection tubes used in most instances were Sovirel SVL 15 × 100 mm tubes, but longer tubes (up to 150 mm) have been used satisfactorilty. These tubes have a screw-cap with a polytetrafluoroethylene PTFE)-lined seal.

The GLT was formed to the desired shape after heating to medium red-heat according to the manufacturer's instructions. A continuous length was used from the

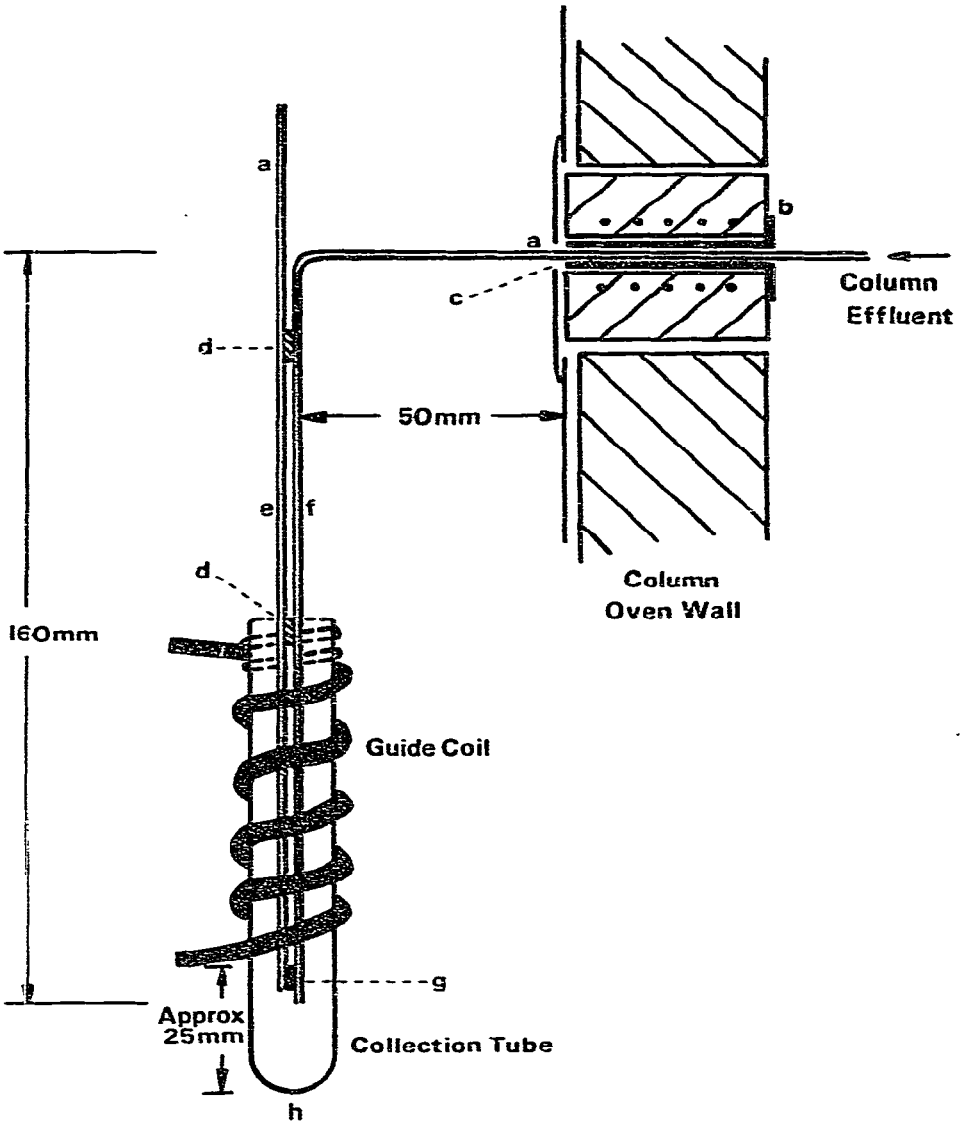

Fig. 2. Collection system indicating the procedure used to heat the delivery line. a = Connection points for low-voltage heating of the exposed delivery line; b = standard Pye Unicam outlet line heater; c = a thermocouple was inserted into the delivery line port at this point; d = soda-glass joints; e = glass-lined tubing which acted only to provide electrical conductivity to the tip of the delivery line; f = delivery line consisting of a single length of glass-lined tubing from the splitter: g = silver-soldered joint; h = cooling was applied only at the tip of trapping tube with a liquid nitrogen bath.

column exit T-junction to the exit at the trap. Electrical conduction in the delivery line was achieved with a second piece of GLT, which was silver-soldered near the exit point as used by Woolley[2]. The tubes were kept apart for the remainder of their lengths by two beads of glass fused at red-heat. Soda-glass was found to be mos suitable (from a Pasteur pipette); Pyrex glass was unsuitable, it tended to shatter

The line was heated by two methods. The portion of line which passed through the wall of the Pye Unicam 104 GC oven was heated by the normal "outlet" heater, and the emergent line was heated electrically by a low voltage. The applied voltage was variable and depended upon the exact set-up. The electrical contacts were made with crocodile clips. The line temperatures were set so they were just above the maximal column temperature used. These temperatures were measured by two methods. The temperature in the oven wall was measured by a thermocouple placed near the exit of the delivery line, and the exposed line temperature was measured with thermocrayons. The GLT was connected to the standard Pye Unicam stainless-steel 1/16-in. T-coupling with a modified Graphlok ferrule as shown in Fig. 1.

A metal coil made from 1/8-in. diameter welding rod was used as a guide to ensure centralization of the delivery line within the collection tube, and as a means of heating the upper part of the collection tube while the tip of the tube was cooled in liquid nitrogen. The metal coil itself was heated by radiation from the hot delivery line when the collection tube was not in place in the coil. During collection the tube was simply hand-held in position with the aid of a pair of metal tongs, so that the delivery line was approximately 10 mm from the bottom of the tube. No seal was used between the delivery line assembly and the collect on tube. Peaks were collected over periods of 1–2 min. Immediately after collection of the fractions, the tubes were placed in a desiccator over sodium hydroxide pellets and phosphorus pentoxide and allowed to reach room temperature before being sealed.

RESULTS AND DISCUSSION

The trapping system described has given good results and has enabled heptafluorobutyryl *n*-propylamino acids[3] to be trapped from a GC column with high yields. To prove that the trapped fractions were not degraded the amino acid derivatives were re-chromatographed. In some instances the derivatives were hydrolysed back to the original amino acids, re-derivatized, and verified by mass spectrometry.

The trapping efficiency was not measured accurately, but approximate values calculated from samples re-chromatographed indicated recoveries to be well above 80%.

Woolley[2] used a plastic seal between the delivery line and the trap. Past experience in these laboratories has made me very wary of all plastics when using mass spectrometry for identification. With the exception of the PTFE seals in the screw-caps of the Sovirel tubes, this technique avoided the use of plastics.

The collection tube guide coil proved very effective. Sufficient heat was obtained by radiation from the hot delivery line to give a useful temperature gradient within the tube when the tube tip was just immersed in liquid nitrogen. Condensation of the sample occurred in the bottom of the tube, and co-condensation of moisture or other atmospheric gases was not a problem. If with different operating conditions the radiant heat proved to be insufficient to heat the collection tube, it would be easy to increase the heating effect. For example, a coil with a greater number of turns could be used so that more heat would be absorbed when the tube was not in position, or it could be replaced with one made of copper which would absorb more heat. The application of a low voltage to the coil could also be used to increase the heat output. any one of these procedures or a combination of them would probably achieve the

desired level of heating. The ratio of the internal diameter of the delivery line to that of the collection tube was large (*ca.* 200). This favoured high trapping efficiencies because expansion would have been considerable before cooling occurred. The distance of the delivery line outlet from the base of the tube was found to be optimal at about 10 mm under the conditions used. Some fog formation was observed, but this was only in the lower part of the tube and it quickly dispersed maintaining condensation at the tip.

The Sovirel tubes used have many useful features. It should be noted that they were used for preparation of the original samples for GC[3] as well as the traps described here. These tubes avoided wasteful transfers which would be inevitable with larger vessels. They could also be coupled to a rotary evaporator[3].

All of the experiments were carried out with an 18 ft. × 4 mm I.D. packed column with effluent flow-rates of about 60 ml/min and column temperatures between 100 and 260°.

The splitter described is not novel. It has been used by a number of workers in these laboratories but with normal stainless-steel lines. However, I can find no recorded mention of its use. Although the system described was used with a Pye Unicam 104 chromatograph there is no reason why it should not be adapted to any similar commercial instrument.

REFERENCES

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