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Note

Direct desorption of traps for capillary column gas chromatography

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A simple and inexpensive attachment has been described by Peterson *et al.*[1] for the direct sampling of sorption traps used to collect volatile organic compounds. However, I found that their thermal focussing technique of lowering the column temperature below ambient, caused drastic loss of resolution with Carbowax 20M support-coated open tubular (SCOT) columns. This note describes modifications which enable the attachment to be used with any type of column and allow conventional syringe injection without detaching the column and removing the injector insert.

EXPERIMENTAL

Construction

The dimensions given in Fig. 1 are based on a Varian 2700 gas chromatograph fitted with 6.3-mm injectors. The equilibration tube was made from cold-drawn 6.3 mm O.D. stairless-steel tubing. The coupling (N) to the inlet of the injector and the O-ring seal assembly (K) were machined from 12.7-mm brass rod. Both fittings were silver soldered on to the stainless-steel tube. Alternatively the O-ring seal assembly may be made from a 1/8-in. Gould Eastman–Imperial compression fitting. (Needs to be partially drilled out with a 6.3-mm drill so that the O-ring is retained.) The seal (O) between the coupling N and the injection port was made from a low-bleed septum bored out with a cork borer of appropriate size.

The transfer line (I) consisting of 1.6 mm O.D. glass-lined stainless-steel tubing (Scientific Glass Engineering, Ringwood, Australia) was attached to the length of 6.3 mm O.D. stainless-steel tubing (G) by crimping the larger tubing and silver soldering. The PTFE seal (F) makes a tight fit with the 6.3 mm O.D. tubing (G) and the transfer line. The internal diameter of the seat (D) is such that the stainless-steel trap could slide into it without undue force. The transfer line (I) was terminated 3 mm from the seat (D). The syringe injection liner, when it is used, fits into this recess (E).

The traps (J) were made from 85 mm of cold-drawn 3.3 mm O.D. × 2.5 mm I.D. stainless-steel tubing (Fig. 1). One end was tapped so that the plunger (P) could be screwed into it. A carrier gas inlet hole (1.0 mm) was drilled at 10 mm from the threaded end. The other end was rounded over so that it could slide into the seat (D) without damaging it.

A 16-mm hole was drilled through the lid of the column oven. This hole was

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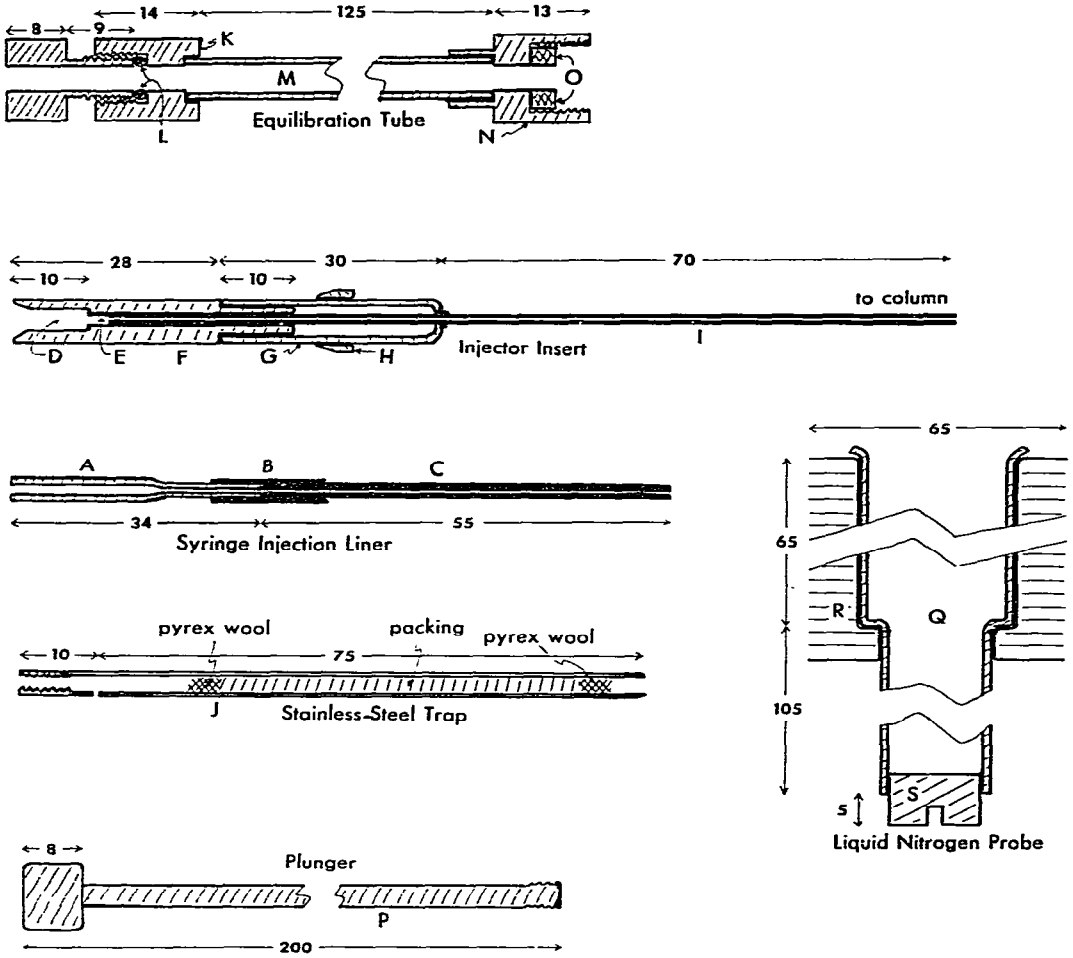

Fig. 1. Direct desorption attachment. All dimensions are in mm. A = 3 mm O.D. Pyrex tubing drawn out as shown; B = heat shrink PTFE tubing; C = 1.6 mm O.D. glass-lined stainless-steel tubing (GLT); D = trap seat (makes sliding seal with trap); E = syringe injection liner seat 3 mm long (makes sliding seal with C); F = PTFE adapter, machined from 6.3 mm rod —makes tight fit with G and I; G = 6.3 mm O.D. stainless-steel tubing; H = Vespel ferrule (Swagelok nut is not shown); I = 1.6 mm O.D. GLT transfer line silver soldered to G (note that it extends up to the seat E); J = trap made from 3.2 mm O.D. cold-drawn stainless-steel tubing threaded (3 mm × 0.5) and drilled (1 mm) through side as shown; K = O-ring seal assembly machined from 12.7-mm brass rod, the I.D. is 3.5 mm; L = 6.3 mm O.D. × 1.6 mm section silicone rubber O-ring; M = 6.3 mm O.D. stainless-steel tubing; N = coupling machined from 12.7-mm brass rod and threaded to match that of the injection port; O = bored-out low-bleed silicone rubber septum; P = plunger made from 3.1-mm stainless-steel rod threaded to take J; Q = liquid nitrogen reservoir made from lengths of 15 mm O.D. and 21 mm O.D. copper tubing silver soldered together; R = expanded polystyrene insulation; S = 12.7 mm copper rod silver soldered to Q. The slot, 3.5 mm deep × 1.8 mm wide, fits over the transfer line I.

positioned so that the liquid nitrogen-cooled probe (Fig. 1) or the heated probe could be quickly applied near the mid-point of the transfer line (I) extending beyond the injector. The heated probe (not shown) consists of 90 mm of 15.9-mm copper rod

terminated at one end with a slot as in S. At the other end 100 mm of 6.3 mm O.D. stainless-steel tubing was silver soldered on for a handle.

Assembly and operation

A short length of 6 mm O.D. × 4 mm I.D. Pyrex tubing was placed inside the injector between the injection port and the injector insert to guide the trap into the PTFE seat (D) during trap insertion. The injector insert was introduced into the injector from the outlet end and secured with a 1/4-in. Swagelok nut. The position of the ferrule (H) was adjusted so that the end of A was approximately 2 mm from the septum when the syringe injection liner is properly seated in E. The liner could be easily removed by withdrawing it through the injection port with the aid of a short length of stiff wire slightly bent at one end, so that it lightly gripped the inside wall of A. I used the plunger from a 10-μl syringe. The column was attached to the end of the transfer line (I) using a zero dead volume union. Next the equilibration tube assembly was attached in place of the septum and septum nut via the coupling N.

The procedure for using the attachment is now described. The test sample was a mixture containing 1 μl of each of ethyl acetate, ethyl butanoate, pentan-3-ol, and pent-1-en-3-ol, in 50 ml of water.

The stainless-steel trap was packed with 100 mg of Chromosorb 105[2] and was conditioned initially at 190°C with nitrogen at 20 ml/min for 48 h. Immediately before use the trap was conditioned for a further 1 h. The non-threaded end of the trap was attached, using a Swagelok reducing union, to 120 mm of 6 mm O.D. Pyrex tubing. A low wattage thermostated tube heater was placed around the Pyrex tubing. A 50-μl aliquot of the aqueous test mixture was deposited into the Pyrex tubing, and flushing into the trap with nitrogen (15 ml/min) over a 20-min period. During this period the temperature of the Pyrex tube was slowly increased to 100°C. The trap containing the sample was flushed with more nitrogen (20 ml/min) at 40°C for 30 min to remove adsorbed water. The plunger was screwed into the trap and the assembly partially inserted through K into the equilibration tube so that the 1 mm hole in the trap was still visible. After 5 min (to allow air to be flushed out of the trap) the assembly was pushed in until the trap was inside the equilibration tube. It was held in this position until the column flow (head pressure) had re-established. The cold probe was lowered through the oven lid so that the transfer line was in the slot of the probe tip (S). Liquid nitrogen was poured into the probe reservoir. The trap was then pushed in until it was seated in the PTFE seat (D). Liquid nitrogen was maintained in the reservoir during the 7-min desorption period. The trap was then withdrawn from the seat but not out of the injector. The cold probe was then quickly exchanged for the heated probe (200°) in order to "inject" the sample.

RESULTS AND DISCUSSION

Fig. 2 shows a chromatogram of the test mixture introduced with the direct desorption attachment in conjunction with a trap packed with Chromosorb 105. The peaks are sharp and symmetrical.

When direct sampling of sorption traps is used with capillary columns some method of sample concentration is essential during the desorption period. I have found the thermal focussing technique in which the column temperature is lowered to ambient or below difficult to control with the Varian 2700 gas chromatograph. It also

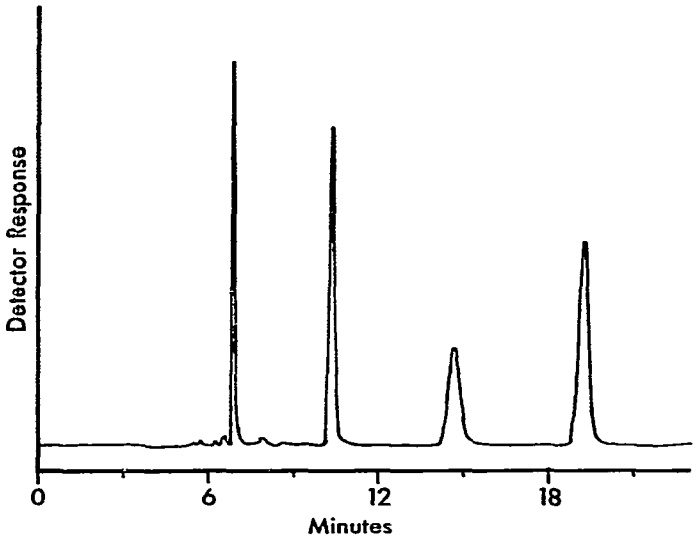

Fig. 2. Typical gas chromatogram obtained with direct desorption attachment. Trap packing: Chromosorb 105. Column: 60 m × 0.5 mm I.D. SCOT coated with Carbowax 20M. Carrier gas: nitrogen at 18 cm/sec. Temperature program: isothermal for 15 min at 50°C then 1°C/min. Temperatures: injector 175°C, detector 150°C. Sample: ethyl acetate, ethyl butanoate, pentan-3-ol, pent-1-en-3-ol (in increasing retention time).

resulted in poor resolution when a Carbowax 20M SCOT column was installed. This is probably due to solidification of the stationary phase. Our cold probe method which is similar to that described by Murray *et al.*[2] overcame these problems.

In over 12 months of intensive use of the direct desorption attachment I have not encountered any problems with accelerated degradation of column performance attributable to the entry of air or water on to the column. However, care has always been taken to lower the column temperature before venting the injector to the atmosphere. Excessive sample or water loading of the trap could cause a blockage of the transfer line during the desorption process but with sample sizes compatible with capillary columns this should not occur. The system has been used at desorption temperatures as high as 230°C without problems. Higher desorption temperatures were avoided by a suitable choice of packing for the trap (*e.g.*, we have used 10% OV-101 on silanised Pyrex wool for farnesol).

This direct sampling accessory makes it very easy to check for homogenity of gas chromatography (GC) peaks by trapping and analysing on liquid phases of different polarity. Samples eluting from the gas chromatograph could be collected directly into a suitably packed trap with little or no cooling in essentially quantitative yields. In cases where a stationary phase unsuitable for GC–mass spectrometry (MS) is necessary to obtain adequate separation, this trapping and re-chromatographing procedure makes it possible for the actual GC–MS analysis to be carried out on a column more compatible with GC–MS with little increase in the amount of sample required.

Syringe injection of samples could be readily accomplished without disconnecting the column to replace the injector insert. Instead the equilibration tube assembly

is removed, the syringe injection liner (Fig. 1) inserted into the seat E, and the injection port sealed with the septum and septum holder in the conventional manner. The ease of changing between direct desorption of traps and syringe injections has proved to be very convenient for checking column performance and for introducing standards, especially for GC–MS analyses.

In the design of the simple and inexpensive desorption device described above, I have given high priority to the ease of using it on an unmodified injector and to the use of dimensions that are compatible with flow-rates associated with capillary columns. I have found the accessory to be invaluable in the GC and GC–MS analyses of a wide range of compounds present at low levels in biological systems.

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