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Note

A simple semi-micro gas-liquid chromatography sample trap for aerosol-forming substances

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The trapping of material from a gas-liquid chromatographic (GLC) column is frequently complicated by the formation of aerosols (especially for compounds with boiling points above 175° at 760 mm). Several methods have been used in order to overcome this problem. A thermal gradient, either longitudinal^{1,2} or radial³, is useful in breaking down these aerosols. For instance, a high-efficiency trap suitable for large-scale work (although of fairly complicated construction) that uses a radial thermal gradient has been described by Teranishi *et al.*³. High-tension electrostatic traps (Cottrell-type precipitators) have been used² but the inconveniences (electric shock, ignition) of such devices are obvious.

This paper describes a simple trap system for the 1–100 mg range using combined longitudinal and radial thermal gradients. It is made up of a simple glass collector tube, an electrically heated glass insert, a copper sleeve that penetrates the wall of the GLC oven and a simple splitter at the column outlet. Substances from the chromatographic column are collected by pushing the collector tube (with heater insert fitted) through the copper tube against the rubber septum (Fig. 1, component 9) on the splitter. The copper tube is mounted on a copper plate inside the oven and provides a longitudinal temperature gradient; the heater insert provides a radial gradient.

The trap system was tested using a number of compounds that fumed heavily by injection of accurately measured amounts of pure material and re-collection of the material in weighed traps. The recovery of material varied markedly with the heater insert temperature (Fig. 2). When the insert was not heated, heavy white fumes appeared, but at the maximum recovery almost no fumes could be seen. No condensation could be detected on the insert when it was heated.

The collector tubes were usually air-cooled. When a small water cooling jacket was fitted to the collector tube (rubber stoppers), there was a slight improvement in yield (see curves 5 and 6 in Fig. 2).

The material in the tubes could be distilled out or sealed off in the narrow part of the tube after collection by centrifugation. Alternatively, the material could be washed out, for instance directly into an NMR tube. Samples of about 30 mg can be readily collected from two or three injections of moderately pure material.

A slightly modified Varian 600 chromatograph was used. A 13-mm hole was

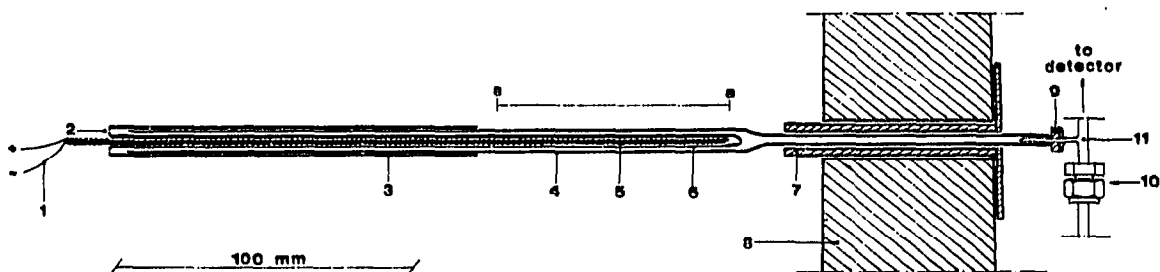


Fig. 1. GLC trap with heated insert. 1, Kanthal wire, 0.2 mm, total resistance 50Ω ; 2, pressure release vents; 3, guide tube to center the heater insert; 4, collector tube; 5, glass capillary with heater winding; 6, glass envelope of heater insert; 7, copper tube; 8, gas chromatograph oven wall; 9, rubber septum; 10, Swagelok connection; 11, splitter.

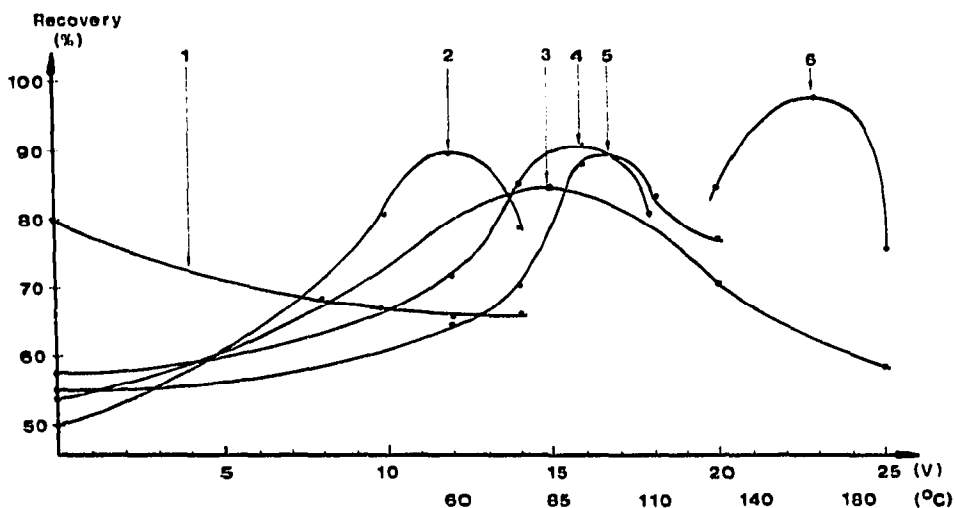


Fig. 2. Recovery versus heater insert voltage (or approximate temperature). Column, 2.5 m \times 4 mm I.D.; 20% DC-550 on Chromosorb A, 45-60 mesh; oven temperature, 220°. 1, *n*-Nonane (b.p. 151°); 2, *n*-dodecane (b.p. 216°); 3, quinoline (b.p. 237°); 4, *n*-tetradecane (b.p. 254°); 5, *n*-hexadecane (b.p. 287°); 6, *n*-hexadecane collected using water cooling on the section a-a in Fig. 1.

drilled in the oven wall for mounting the copper tube and a simple splitter (*ca.* 100:1) was fitted between the flame detector and the end of the column.

The trapping system described here has given ideal performance in routine collection of small samples for NMR and other forms of spectroscopy.

ACKNOWLEDGEMENT

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