

## Note

### Chromatographic analysis of major trace components in solvents with concentration prior to sample injection

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One of the commonest tasks in the chromatography of trace substances is the analysis of major trace components in solvents. The poor sensitivity because of the limited sample size and the masking of the trace zones by the zone of the main component (solvent) are unsolved problems<sup>1-4</sup>. In capillary chromatography, where the sample size is usually *ca.*  $10^{-3}$   $\mu\text{l}$ , these problems are serious<sup>2</sup>.

One of the ways of solving such problems is to carry out concentration of the trace component before injection into the chromatographic column. In this instance the first stage of the analysis is the concentration of the trace component on the cooled sorbent. A solid adsorbent and a solid support covered with liquid stationary phase can also be used. The second stage is desorption of the concentrated trace components by heating the sorbent. The concentration and desorption are carried out in a continuous flow of the carrier gas. Similar techniques have already been used in gas chromatography<sup>3,5,6</sup>.

In the method proposed here, the apparatus and techniques are simpler and more convenient than those described previously.

#### APPARATUS AND TECHNIQUE

The concentrator is situated in the thermostat between the injector and analytical column (Fig. 1). The device (Fig. 2) consists of a glass-walled, stainless-steel tube of length 100 mm and I.D. 1 mm Tenax GC (60-80 mesh) was used as the sorbent for the concentration step. The construction of the cooler is of the "tube in a tube"

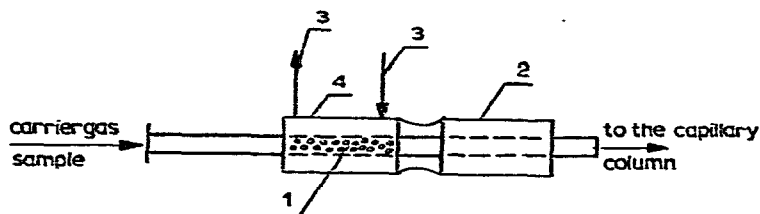


Fig. 1. Diagram of concentrator system. 1 = Concentrator filled with sorbent; 2 = heater; 3 = coolant; 4 = cooler.

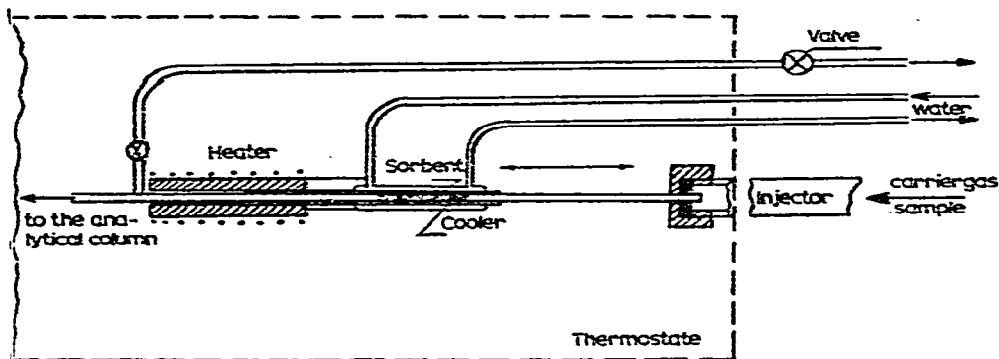


Fig. 2. Arrangement of the device in the thermostat.

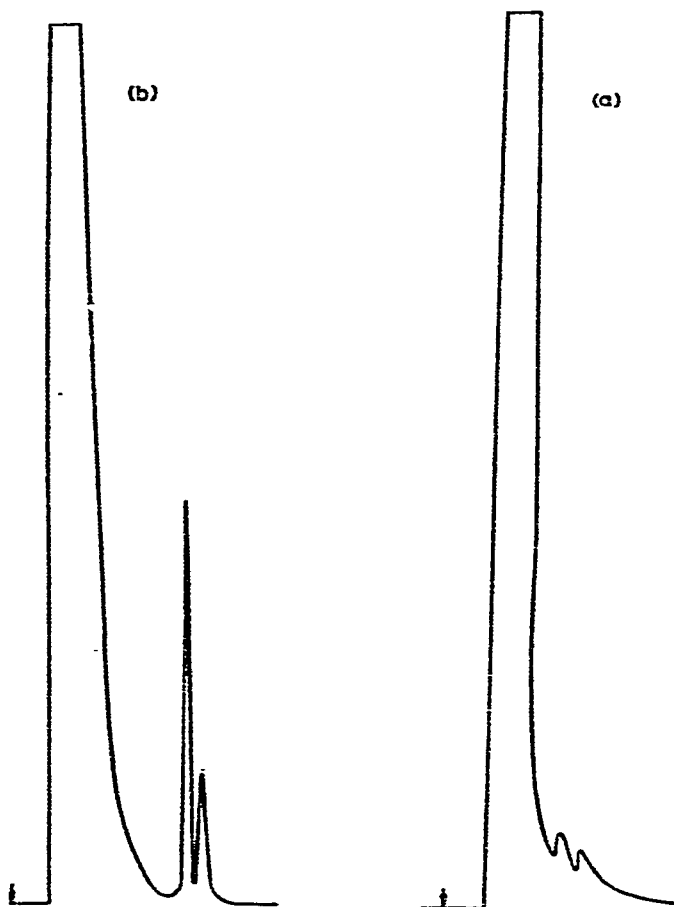


Fig. 3. Analysis of benzene and toluene at the  $10^{-5}\%$  level in *n*-pentane. (a) Direct sample injection; (b) with use of the proposed device. Peaks from left to right: *n*-pentane, benzene, toluene.

type, and it moves along the outside surface of the column-concentrator and covers the zone of the sorbent during the concentration. The length of the cooler is 42 mm, whereas the length of the concentration zone is 38 mm, with a diameter of 1 mm. The cooler is made of brass, and the cooling agent is water at 18°. The heater can apply the temperature of the thermostat, but in our case the heating was independent. The length of the heater was chosen as 42 mm. In the first stage the cooler is situated on the sorbent zone so that the temperature in this zone is decreased to 20°. In our work water was used as the coolant, but other media can be used. The sample is injected and passes into the cooled zone, where concentration of the trace components takes place, and the excess of the solvent passes through the concentrator and enters the chromatographic column. When the beginning of the first peak (in this instance due to the excess of solvent) is recorded, the heater is moved over the cooled concentration zone. Desorption then takes place and the desorbed trace components enter the column. The heater is made of heat-conducting metal, *e.g.*, brass, and can be provided with heating independent of the thermostat. In our work the temperature in the desorption zone was 240°.

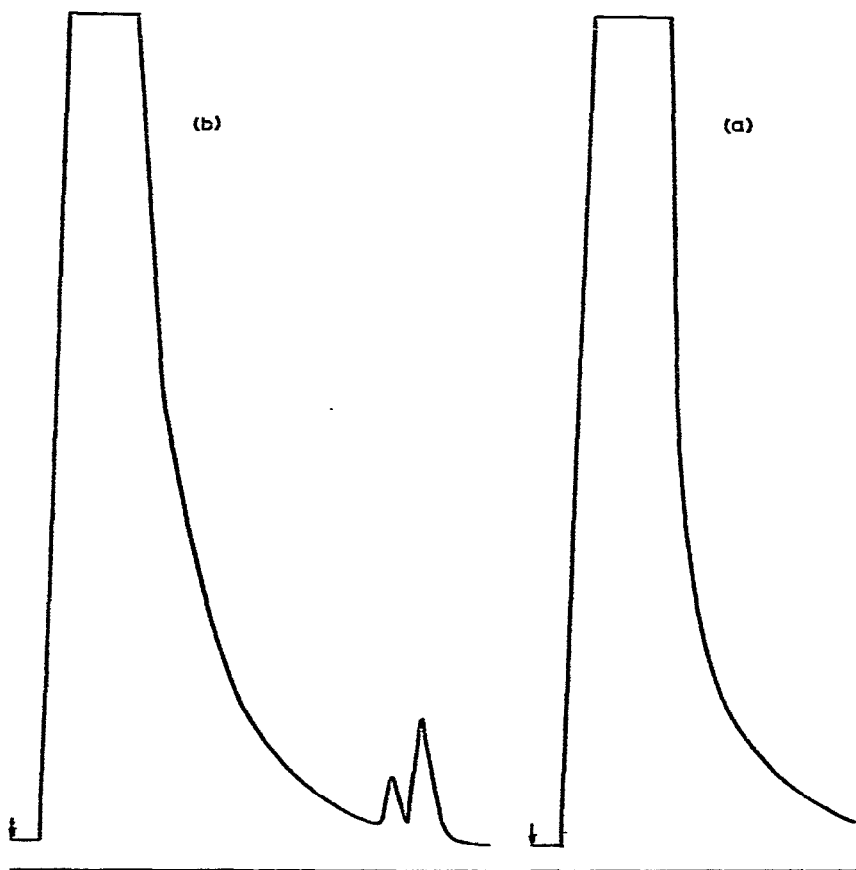


Fig. 4. Analysis of benzene and toluene at the  $10^{-7}\%$  level in *n*-pentane. (a) Direct sample injection; (b) with use of the proposed device. Peaks as in Fig. 3.

As an example of possible applications, Fig. 3 shows the analysis of a mixture of benzene and toluene at concentrations of  $10^{-5}\%$  (v/v) in *n*-pentane.

Fig. 3a is a chromatogram resulting from direct introduction of the sample into the chromatographic column. The sample volume was  $1\ \mu\text{l}$  and the splitting ratio was 1:100 or 1:50. The glass capillary column ( $50\ \text{m} \times 1\ \text{mm}$  I.D.) was of the WCOT type with polyethylene glycol 20 as the stationary phase. It can be seen that the peaks of benzene and toluene are almost completely masked by the peak of the solvent.

Fig. 3b shows the result of the separation of the same mixture with the use of the suggested device. Tenax GC (60–80 mesh) was used as the sorbent.

Under the same conditions, a mixture of benzene and toluene at concentrations of  $10^{-7}\%$  (v/v) in *n*-pentane was analysed (Fig. 4). A comparison of the results with direct sample injection (Fig. 4a) and with the use of the proposed device (Fig. 4b) indicate the possibilities of the device.

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