

## Notes

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### A new technique for solid injection in gas-liquid chromatography

#### Analysis of barbiturates in urine

In connection with our GLC investigations of lower terpenes present in micro-quantities of plant material we have for some years used a solid sample injector as shown in Fig. 1. Small amounts of plant material can be directly introduced into the flash heater, where the volatile compounds immediately evaporate in the inert carrier gas and are carried onto the gas-liquid chromatographic (GLC) column<sup>1</sup>.

Lately we have used this injector for GLC of barbiturates in extracts of urine: The stainless-steel holder of the injector is flamed to remove traces of organic substances. A small piece of filter paper is placed in the holder and a suitable amount of a purified ethyl ether extract of acidified urine, containing the barbiturates, is applied to the filter paper. The solvent is evaporated and the application of the ether extract is repeated. The injector is mounted on the gas chromatograph.

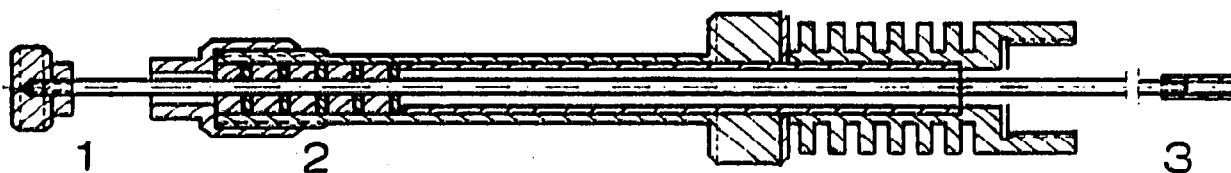


Fig. 1. Solid sample injector. 1 = Stainless-steel rod going through gas-tight Teflon O-rings (2); 3 = holder for material to be analysed.

The sample is brought into the flash heater by means of the stainless-steel rod and withdrawn after 20 sec. The volatile substances evaporate immediately whereas the non-volatile compounds remain on the filter paper. No decomposition of the barbiturates could be detected. For other substances a gilded holder may be necessary instead of stainless steel in order to prevent decomposition.

The advantages of the technique described are: (1) quite dilute solutions can be concentrated on the filter paper, the evaporation surface of the filter paper being great; (2) no interference of solvent peaks on the chromatogram; (3) non-volatile compounds in the urine extract will not contaminate the column.

Fig. 2 shows a typical chromatogram of a purified ethyl ether extract of acidified (HCl) urine containing barbital, pentobarbital, hexobarbital and phenobarbital. GLC conditions: glass column, 2 m  $\times$  2 mm I.D., Chromosorb 60-80 mesh, a.w.,

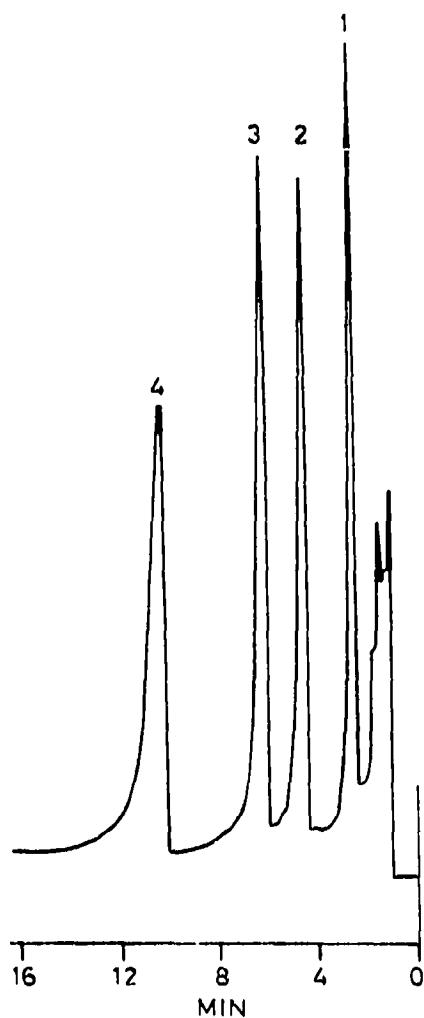


Fig. 2. Chromatogram of barbiturates obtained by solid sample injection of a purified ethyl ether extract of acidified urine. 1 = Barbital; 2 = pentobarbital; 3 = hexobarbital; 4 = phenobarbital.

Apiezon L 1%; column temperature 195°; flash heater temperature 270°; carrier gas, nitrogen; Becker-Delft Model 409 gas chromatograph.

*Department of Pharmacognosy,  
University of Leiden,  
Leiden (The Netherlands)*

K. E. RASMUSSEN  
S. RASMUSSEN  
A. BAERHEIM SVENDSEN

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