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RESEARCH NOTE

Micro infra-red spectrometry of gas chromatographic fractions

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Abstract—A simple apparatus is described by means of which gas chromatographic fractions are vacuum deposited on KBr powder. Good infra-red spectra were obtained for sample sizes down to 10–1 μg.

Introduction

The techniques for infra-red analysis of gas chromatographic fractions usually involve condensation, transfer and finally presentation of the component to the sample beam of the spectrometer using allied micro-sampling and beam condensing systems. Gas chromatographic fractions can best be collected in glass or teflon tubing [1] or in a tube containing gas liquid chromatography packing [2]. Transferring of the fraction to the small amount of KBr must be done with a minimum of manipulative losses [3].

The combination of the collecting method [2] and the modified subliming method of Maehly [4] give a high recovery and high quality infra-red spectrum. Moreover the introduction of impurities on the KBr pellet from column bleeding can be minimized by this method.

Method

The apparatus consists (Fig. 1) of three parts: the main glass body 1·3 cm i.d., the glass tube with the collected fraction (1–15 g) and an Aluminium cooling bar. To the side arm in the main

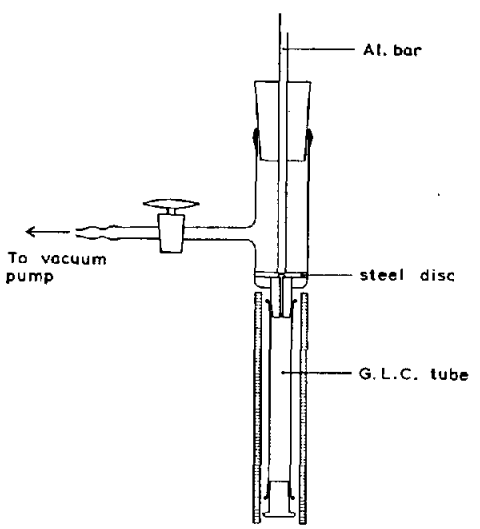

Fig. 1. Apparatus for vacuum deposition of gas liquid chromatography fractions on KBr powder.

[1] S. C. Brooks and V. C. Godefroi, *Anal. Biochem.* **7**, 135 (1964).
[2] M. Cartwright and A. Heywood, *Analyst* **91**, 337 (1966).
[3] J. H. van der Maas, Ph.D. Thesis, University of Utrecht (1965).
[4] A. C. Meahly, *Analyst* **87**, 116 (1962).

body is connected to the vacuum line. The steel disc with an orifice of 0·5 mm or 1·5 mm fits on the top of a glass capillary 1·0 or 0·3 mm i.d. is centrated on the orifice of the steel disc. The glass capillary is fitted with a cone in which the collecting tube is placed. Unpressed KBr is cooled by an Aluminium bar immersed in liquid nitrogen. The collecting tube is inserted in a small copper tube, which can electrically be heated to 250°C. After cooling and evacuation (an oil pump giving 0·1 mm of mercury) of the apparatus, the deposition of the compound is usually finished in 6–8 min. The pellet is prepared in the usual way pressing or applying vacuum to the microdisc.

Results

The technique was tested on compounds with boiling points above 150°C. Among these were various steroids, vanilline and benzophenone. The infra-red spectra were recorded with a

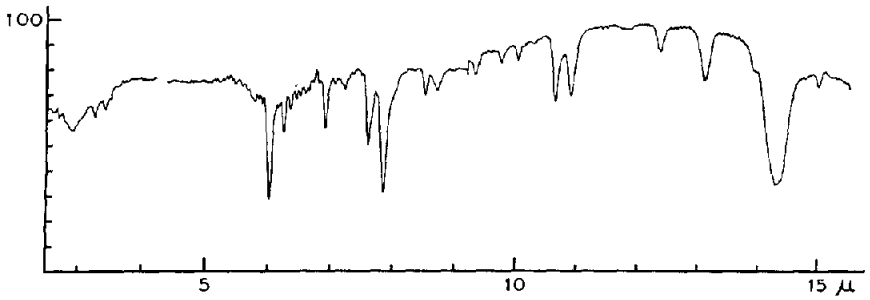

Fig. 2. Infrared spectrum of 1 microgram of benzophenone recovered from the gas liquid chromatography collecting tube.

Perkin–Elmer model 21 Infrared Spectrophotometer. To improve energy transmission through the micropellets a Perkin–Elmer mirror beam condensing unit in the sample beam was used. The resulting spectrum of 1 μg benzophenone is shown in Fig. 2.

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