CHROM. 6222

A simple device for introducing liquids, solutions and thin-layer chromatography samples into a mass spectrometer via the direct inlet system

A low cost device which allows rapid introduction of liquids, solutions and thin-layer chromatography (TLC) samples into a mass spectrometer via the direct inlet system has been constructed. The TLC samples can be introduced into the mass spectrometer either in the absorbed state (on the chromatographic support) or after extraction with a small amount of solvent. The system described below has been constructed to fit an LKB-9000 mass spectrometer. However, it may be possible to adapt the device to other commercial mass spectrometers in common use.

The new sample device

The system is shown in Fig. 1 and consists of:

(a) screw-cap;
(b) sample container;
(c) insert (model dependent upon application):
  (1) glass capillary and two gold packings (used for liquids and solutions);
  (2) powder separator in three pieces (used for direct introduction of TLC scrapings);
(d) PVC ring on the DI probe;
(e) hexagonal tool for the screw cap.

The material used was stainless steel, but for substances that are unstable in contact with steel there should be no hindrance in using gold.

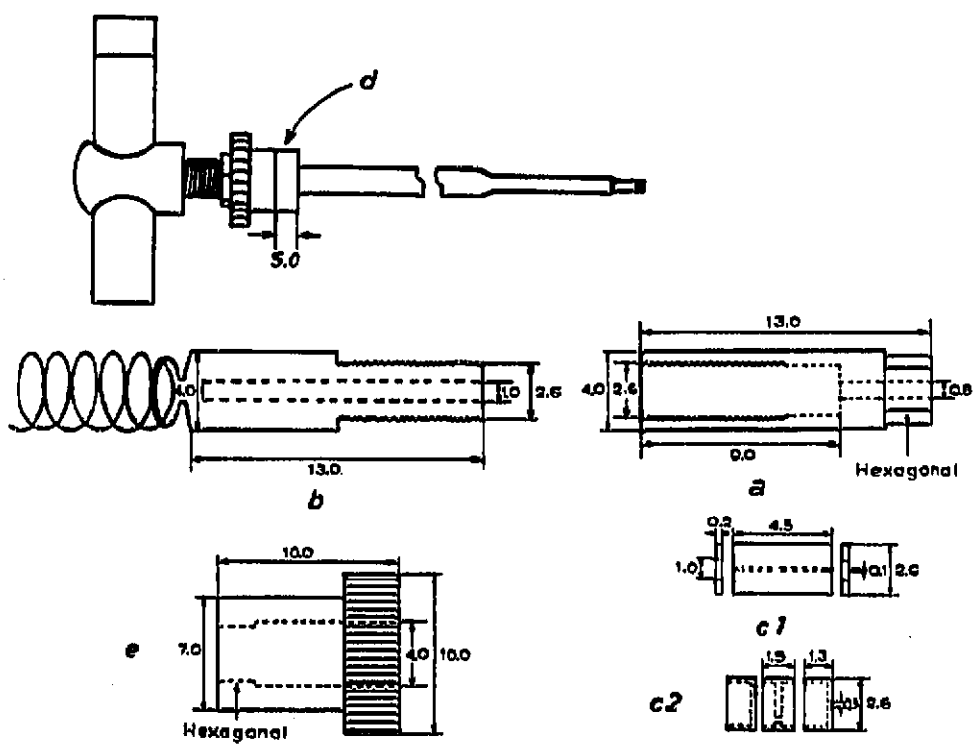

Fig. 1. The new sample device (all dimensions are in millimetres).

It should be noted that the outer dimensions are the same as those of the original mass spectrometer sample holder.

The capillary is cut into suitable lengths and the ends are planed.

The PVC ring (d) is applied to the DI probe to avoid the danger of discharge between the metal container and the ion source, otherwise serious damage to the mass spectrometer will occur. To allow for convenient handling of the equipment, the holder shown in Fig. 2 was used.

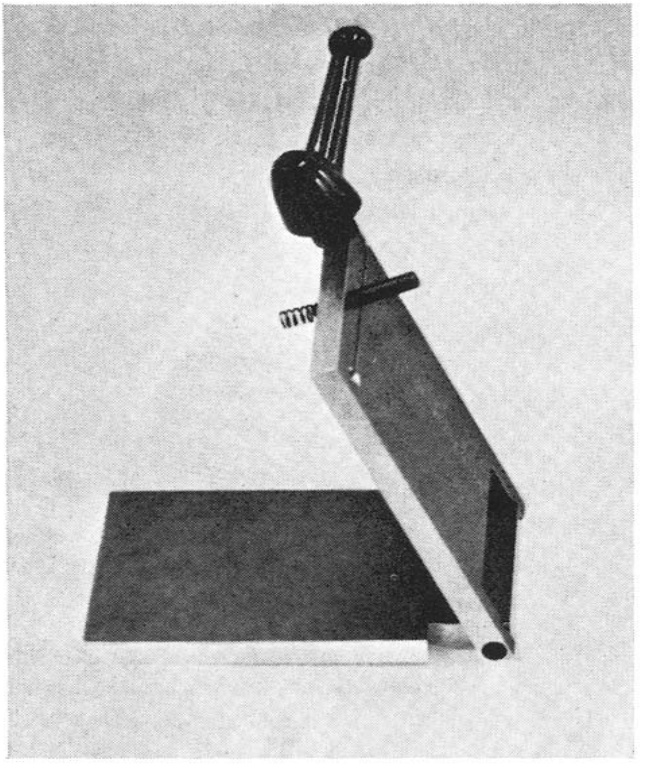

Fig. 2. The holder.

Elution of TLC spots

(1) Insert a cotton-wool plug in an ordinary melting-point capillary. The plug is tamped by compressing it from both sides with steel rods. The tube and the plug are then washed by drawing freshly distilled ether through the tube with a vacuum line, and then dried by drawing air through it.

(2) One end of the capillary is drawn out to a fine tip in a small flame.

(3) The TLC spot is loaded into the open end of the tube, and the powder is packed against the cotton-wool plug.

(4) The powder is then eluted with 2×10 μl of a suitable solvent, which is applied with a 10 μl injection syringe.

(5) When the elution is completed, the solvent is forced out of the capillary into the container under slight pressure, *e.g.*, with a 1-ml injection syringe connected with the capillary by a small piece of rubber tubing.

(6) The screw-cap is fitted without previous evaporation of the solvent and the container is introduced into the mass spectrometer via the direct inlet.

(7) After the solvent has evaporated, mass spectra are obtained in the usual manner.

Steps (1)–(3) of this procedure are a modification of CLARKE's method[1].

Introduction of liquids and solutions

The liquid (solution) is introduced into the container with an injection syringe of a suitable size, then step (6) above is followed.

Introduction of TLC powder

The layer is scraped off the plate and loaded into the container. This can be carried out via a melting-point capillary that is slightly pointed, sufficiently to serve as a funnel. Then the powder separator is applied and the container introduced into the mass spectrometer.

Peak matching

The system allows peak matching in two different ways:

(1) mixing the substance with a suitable standard in the container;

(2) peak matching of unknown substances introduced via the gas chromatograph, while the standard is introduced with the container.

Adjustment of the mass marker and resolution testing

As the container allows continuous introduction of a standard, *e.g.*, perfluorokerosene, at a constant rate over a long period, it is excellent for adjusting the mass marker and testing the resolution.

Experimental

An LKB-9000 mass spectrometer was used, operated with the ion source at 270° and 70 eV. The glass capillary was kindly provided by Göteborgs Termometerfabrik.

Results

Liquids. Liquids of b.p. 60° or higher were analyzed. Sample sizes between 0.05 and 0.5 μl were adequate. The larger volume is required for low-boiling substances. Even lower boiling substances could probably be analyzed by using a thinner capillary.

Solutions. Solutions of testosterone and pentachloronitrobenzene have been analyzed with good results. A 1-μl volume of a solution containing 1 μg per microlitre of solvent (ethanol) was sufficient to give a very good spectrum.

TLC spots. A 5-μg amount of pentachloronitrobenzene was applied on to a TLC plate (0.25 mm Silica Gel GF_{254}) and eluted 10 cm on the plate with ethanol. The spot was then scraped off and analyzed without previous extraction of the substance.

TLC spots extracted. If the TLC layer with the spot was scraped off and extracted as outlined in steps (1)–(7) above, 1 μg of substance was sufficient to obtain a good spectrum.

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Department of Organic Chemistry,
University of Umeå,
S-901 87 Umeå (Sweden)

CARL-AXEL NILSSON
ÅKE NORSTRÖM
KURT ANDERSSON

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