CHROM. 6116

A micromanipulator for compounds eluted from a gas chromatograph

We have reported in an accompanying paper[1] on the analysis by thin-layer chromatography (TLC) and, especially, by mass spectrometry (MS) of fractions eluted from a gas–liquid chromatograph where the two instruments were physically separate. The handling of such samples has been greatly facilitated by the micromanipulator described in this paper. While our own work has been concerned with carbohydrates the technique is suitable for many compounds of low volatility which are amenable to gas–liquid chromatography (GLC).

The technique involves manipulation of the sample in a glass collecting tube in the way in which it was collected, *i.e.* with heat and a flowing gas. If the sample is reasonably stable it may be manipulated using only heat since, as the sample is heated, its viscosity is reduced and the sample will flow as the tube is passed through the oven. Gravity or a gentle flow of dry nitrogen may be used to increase the rate of movement of the sample for less stable compounds, minimizing any degradation. In order to handle 25-μg samples it is necessary to reduce the bore of the melting point capillary to the size of a vacuum leak capillary. In addition the capillary action helps draw the compound to the tip of the melting point tube. Positioning in the fine capillary facilitates removal of the sample to the tip of the mass spectrometer probe, or for TLC or infrared analysis. Since only a microgram sample is required for MS, ample material for examination by other techniques is still available.

A description of the micromanipulator is given in Fig. 1.

The melting tube (A) should be fire polished at both ends for clean insertion through the holed septum in the exit port of the gas chromatograph. By inserting the tube in this manner all the carrier gas and compound will pass through the tube and maximum collection will be achieved. The capillary (B) must be drawn after the collection because the extremely fine bore desired on the end of the tube restricts the gas flow. The minimum sample that can be handled this way was weighed at 25 μg and represented a peak approximately 1 in. high on an F & M 720 gas chromatograph at maximum sensitivity. The upper limit of size for the collection of a component, even when the capacity of the tube is enlarged with a small bulb, would appear to be about 7–10 mg. A sample larger than this is simply blown through the tube.

If gas is required to help move the sample, a lecture bottle (C) of pure nitrogen may be mounted on the board with the small oven (D). Extreme care must be exercised when using gas as it is very easy to blow the sample out of the microcapillary tip. A large opening (E) in the gas line, such that when open there is no gas flow into the capillary, is an excellent safety valve and avoids loss of sample. This opening may conveniently be closed by placing *on* it an oversized rubber stopper (F) which is easily removed when the sample nears the tip of the tube.

The oven (D) was machined from a block of lava (American Lava Company, grade A) which, after firing at over 1000°, is non-conducting and very hard. A hole (*ca.* 0.8 cm diameter) was drilled in the centre of the block and a second smaller lava cylinder to fit this hole was machine-threaded on the lathe for winding on the

resistance wire. The smaller cylinder was in turn drilled through the centre to permit insertion of the capillary tube. The resistance wire used was nichrome (wire gauge 26) and the oven temperature was controlled by the use of a variable transformer (Fisher powerstat 9-521). The oven is normally operated at 240° and corresponds to a setting of 8–10 on the transformer (full scale = 140).

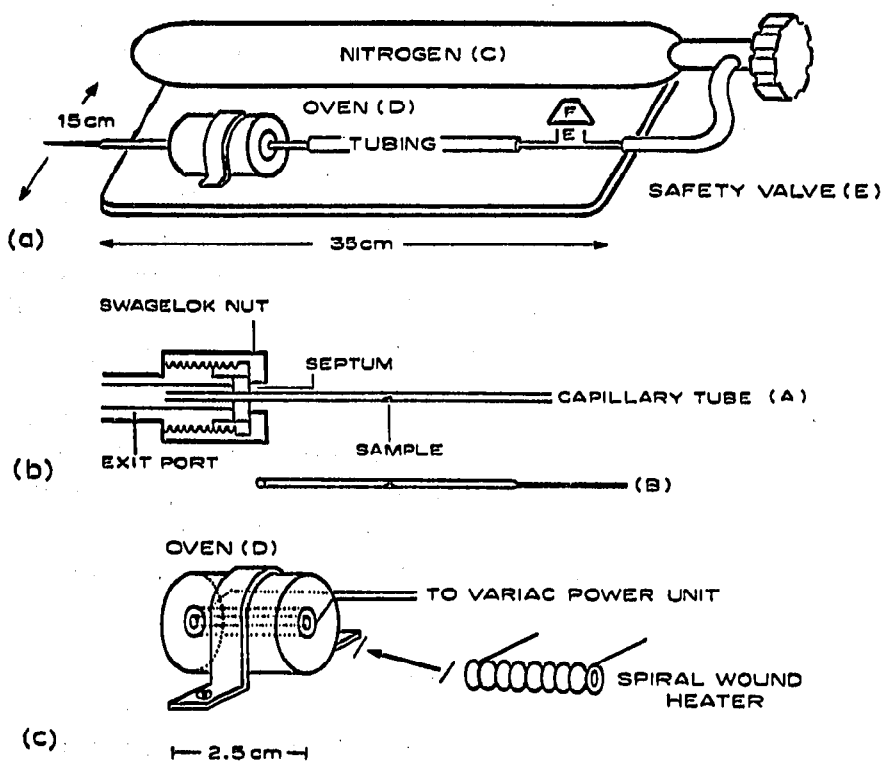

Fig. 1. (a) Micromanipulator assembled on wooden board. The safety valve (E) is closed by placing a rubber stopper (F) *on* the opening. (b) Collection of samples from the exit port of a GC column into a capillary tube (A). Tube (A) drawn to a capillary (B) after collection of sample. (c) Detail of the oven (D), see text for explanation.

The capillary is connected to the gas flow by a piece of small bore rubber tubing which is sufficiently flexible to permit the capillary to be moved slowly through the oven (D). The contents of the capillary are then concentrated in the tip. If the capillary and rubber tubing are disconnected at the safety valve (E) subsequent squeezing of the tubing will eject material from the tip of the capillary on to a TLC plate or the probe of a mass spectrometer. Alternatively, both ends of the capillary may be sealed, without drawing the fine tip, for storage of the sample.

This technique has been invaluable in analyzing, by MS and by TLC, mixtures of partially methylated alditol acetates where individual components differed in amount by as much as 100:1. Use of this micromanipulator is an excellent expedient when a combined GC–MS instrument is not available. The method should be applicable to a wide variety of other classes of compounds especially those of low volatility which do not pose any problem in the initial collection. The system of transfer described obviates the necessity for using organic solvents with the attendant possibility of contamination.

We thank the National Research Council of Canada and the University of British Columbia for financial support and BEATRIX KRIZSAN for the drawing.

Department of Chemistry,
The University of British Columbia,
Vancouver, B. C. (Canada)

G. G. S. DUTTON
K. B. GIBNEY

1 Y.-M. CHOY, G. G. S. DUTTON, K. B. GIBNEY, S. KABIR AND J. N. C. WHYTE, *J. Chromatogr.*, 72 (1972) 13.

First received December 22nd, 1971; revised manuscript received April 24th, 1972