Notes

CHROM. 5477

An efficient gas chromatographic effluent trap for collecting air- and moisture-sensitive compounds

The gas chromatographic purification of air- and moisture-sensitive compounds requires the use of an efficient sample introduction and collection system. Two techniques are commonly used to introduce samples of this type into chromatography units. One technique (for liquids), employs a syringe and a special transfer tube such as the one described by GOKHALE *et al.*[1]. The other technique (for gaseous samples or liquids that can be easily vaporized) employs a gas-sampling valve (or other similar assembly), one part of which is connected to a vacuum line. Various devices are available for collecting effluents from chromatographs, although in handling air- or moisture-sensitive compounds particular care must be taken to minimize the exposure of the purified samples to the atmosphere. In this report we describe an easily constructed trap that can be conveniently used to collect such compounds on a preparative scale in the complete absence of air or moisture.

A general drawing of the trap is shown in Fig. 1. It consists of a standard U-shaped trap attached as shown to a 2 mm four-way high-vacuum capillary stopcock*. The spring-held plug of the stopcock has one straight and one oblique bore. The trap contains a loose plug of glass-wool to aid in trapping the sample. The glass tubing above the oblique bore leads to a 13 mm OD tube that is fitted with a 10 mm *medium* porosity fritted disk. A small layer of mercury (height about 2 mm) is placed on the top of the frit. The glass tubing from the straight bore of the stopcock is attached to an 18/7 glass ball-joint. This ground-glass connection permits the trap to be attached to a glass manifold-type tube leading from the exit port of the chromatograph**. The manifold presently has four 18/7 glass socket joints attached via 3-way vacuum stopcocks. The 3-way vacuum stopcocks are arranged to permit one to divert the effluent to a particular trap or to bypass one trap and divert the flow to another trap. The arrangement permits the collection of pure compounds in each trap from more than one sample injection.

In using the trap, one adds mercury to cover the frit and first evacuates the trap on a vacuum line with the stopcock in position B***. The stopcock is then turned 90° to a position that is intermediate between the A and B positions and it is placed on the exit manifold of the chromatograph. The carrier gas (helium) is allowed to

* Stopcocks are commercially available from Eck and Krebs Scientific Laboratory Glass Apparatus, Inc., 27-09 40th Ave., Long Island City, N.Y. 11101 (Cat. No. 5105).

** The tube that contains the glass frit may have to be bent towards the back of the trap (still being positioned vertically though) if it interferes with the manifold assembly.

*** We recommend cautiously testing all newly constructed traps of this type to be sure that mercury does not pass through the frit on evacuation. Occasionally some medium frits were found to be unsatisfactory.

pass through the trap with the stopcock in position B. The trap is then surrounded by a Dewar flask containing liquid nitrogen and the assembly is ready to be used for collecting a sample. Other traps are added to the manifold in the same way when more than one sample is to be collected. The layer of mercury on the frit allows the carrier gas to pass through the traps, but prevents oxygen or water vapor from back-condensing with the sample. We have found that most materials are almost quantitatively stopped in the complete absence of any air or moisture when these traps are used with liquid nitrogen as coolant to trap the condensable vapors.

At the conclusion of the chromatographic separation, the stopcock is moved to a position that is intermediate between positions A and B and the trap assembly, still cooled with liquid nitrogen, is returned to the vacuum line where trapped carrier gas is pumped away with the stopcock in position A. Any air which may enter the small volume between the frit and the stopcock while the trap is being moved (owing to the mercury movement) does not enter the U-trap and can be neglected. When the carrier gas is removed, the sample can be quantitatively condensed to another part of the vacuum line for identification or further work.

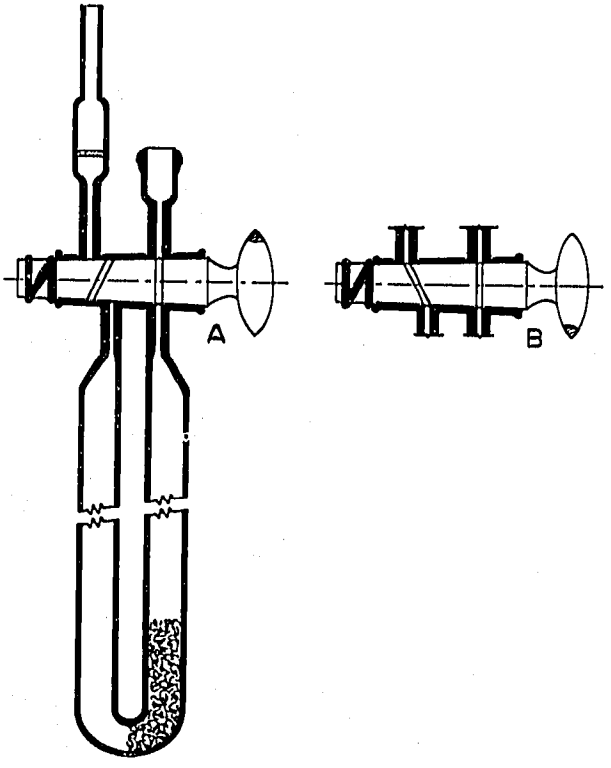

Fig. 1. Trap for the collection of air- and moisture-sensitive compounds.

We have successfully used this type of trap in the collection of various moisture and/or air sensitive compounds such as the parent silanes* and germanes, their halogen derivatives, PF_5, and numerous other compounds which we have reported elsewhere in the literature[2-6**].

* Silane and the parent silanes react very explosively with oxygen and extreme caution is recommended when handling these compounds with the apparatus described.

** A Varian-Aerograph A90-P3 gas chromatograph equipped with a gas sampling valve is currently in use with the collection system described above. Illustrations of columns, flow rates, etc. have been given in previous publications [2-6].

We recommend taking special care to dry the carrier gas before attempting to purify compounds such as PF_5 that are particularly moisture-sensitive. We often have used PF_5 to test whether or not our gas chromatographic set-up is completely moisture-free. Any trace amount of moisture present will convert PF_5 to POF_3 which can easily be detected by its characteristic IR spectrum[7]. Passing the helium carrier gas through a standard drying column and a coiled tube in liquid nitrogen is generally sufficient to remove all traces of moisture.

Department of Chemistry, Carnegie-Mellon University, Pittsburgh, Pa. 15213 (U.S.A.) CHARLES H. VAN DYKE

1 S. D. GOKHALE, J. E. DRAKE AND W. L. JOLLY, *J. Inorg. Nucl. Chem.*, 27 (1965) 1911.
2 S. CRADOCK, G. A. GIBBON AND C. H. VAN DYKE, *Inorg. Chem.*, 6 (1967) 1751.
3 J. T. WANG AND C. H. VAN DYKE, *Inorg. Chem.*, 6 (1967) 1741; 7 (1968) 1319; *Chem. Commun.*, (1967) 612.
4 G. A. GIBBON, J. T. WANG, AND C. H. VAN DYKE, *Inorg. Chem.*, 6 (1967) 1989.
5 M. A. FINCH, L. H. MARCUS, C. SMIRNOFF AND C. H. VAN DYKE, *Syn. Inorg. Metalorg. Chem.*, 1 (1971) 103.
6 G. A. GIBBON, E. W. KIFER AND C. H. VAN DYKE, *Inorg. Nucl. Chem. Lett.*, 6 (1970) 617.
7 H. S. GUTOWSKY AND A. D. LIEHR, *J. Chem. Phys.*, 20 (1952) 1652.

Received May 25th, 1971