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Combination Gas Sampler and Fraction Collector for Gas Chromatography and Mass Spectrometer Application

MAURICE L. BAZINET AND JOHN T. WALSH

Pioneering Research Division, U. S. Army Quartermaster Research and Engineering Center, Natick, Massachusetts¹

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FREQUENTLY very small samples of gaseous mixtures require separation by gas chromatography into individual components or less complex groups prior to mass spectrometric analysis. Because of the minute sample size, the usual methods employing a syringe or gas pipette cannot be used to introduce the sample to the gas chromatography unit.

The apparatus to be described can function for sample introduction to either a gas chromatographic unit, or to a mass spectrometer, or for collection of samples from a gas chromatographic unit.

This device is similar to that described by Glew and Young,¹ but employs only a single stopcock so that all manipulations are rapid and convenient.

The unit (Fig. 1) consists of a four-way, twin-V bore, 2-mm stopcock,² approximately 8 in. of $\frac{1}{4}$ in. o.d. Pyrex glass tubing to form the enclosure of the trap, and an inlet and outlet arm with 12/30 male ground glass joints plus a removable glass blind fitted with a 12/30 female ground glass joint. If a trap of smaller volume is desired, the U-tube can be made of capillary tubing.

When the unit is used as a sampling device, a glass blind is placed on one side arm and the other side arm is attached to a vacuum system. After evacuation of the U-tube, with the stopcock in the vertical position (Fig. 1, B), the sample may be introduced from another source by free expansion, by cooling the sampling unit, or a combination of both. After sample introduction, the stopcock is turned to the horizontal position (Fig. 1, A) locking the sample in the U-tube for transfer to a gas chromatography unit or the mass spectrometer. The sample can be swept into a gas chromatographic unit with prior flushing of the air in the inlet and outlet section by connecting the unit in the carrier gas stream with Tygon tubing. A slight vertical deflection of approximately $\frac{1}{2}$ in. is observed when the sample is introduced, which serves to indicate the starting point of the scan. If necessary the sample may be volatilized by warming the lower section of the sampler before flushing into the chromatography unit.

To utilize the unit as a fraction collector, the chamber is first flushed with carrier gas while the chamber is immersed

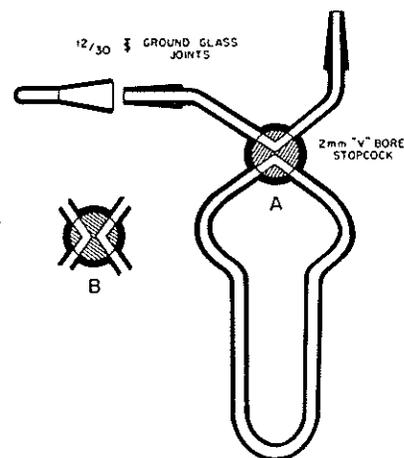


Fig. 1. U-type sampler-collector tube.

in the coolant selected for trapping of the components. Carrier gas then is locked in the chamber by turning the stopcock to the horizontal position. Carrier gas is allowed to bypass the chamber until a peak appears on the chromatographic record. The sample stream is diverted then into the chamber, and the component is condensed out. When the chamber is opened to allow the sample stream from the chromatographic column to pass through, only a small deflection, if any, is noticed on the chromatographic record. After the component has completely emerged from the column, the stream bypasses once again.

By having several collection units on hand during a gas chromatographic separation, a series of components emerging one after the other may be trapped by simply replacing collection units. A simple method is to provide a three-way stopcock at the exit end of the chromatographic column with two collection units attached. When a component has been collected in the first unit, the second may be opened to collect the next component and the first unit replaced with another collector. In this manner a collector is always available for an emerging component.

A similar type of sampler-collector unit may be constructed with a coiled chamber to provide a much larger surface area for condensing fractions.

¹ D. N. Glew, and D. M. Young, *Anal. Chem.* **30**, 1890 (1958).

² Made by Martin Glass Company, Evanston, Illinois.