## A stream splitter and functional group tester for use with a flame ionization detector

A post-column stream-splitter is required when the effluent of a gas chromatograph equipped with a flame ionization detector (FID) is to be collected or psychometrically analyzed. Most FID chromatographs combust the entire column effluent in the detector.

This report describes an inexpensive splitter of unusual design for use with an F & M Model 1609* chromatograph. The device allows variable but controlled split ratios of maximum utility for fraction collection or effluent analysis and can be easily adapted to any FID.

*Experimental*

Fig. 1 is a diagram of the preliminary apparatus used to determine split ratios. Different lengths of 22-gauge Luer Lok needles attached to a B-D MS-10 stopcock (Becton Dickinson Company, Rutherford, New Jersey) are used to vary the amount of column effluent diverted to the detector. Short needles restrict the gas flow little and divert most of the column effluent from the FID inlet to the atmosphere. For determination of split ratios, only needle lengths were varied. All chromatographic conditions were held constant: column temperature (100°), sample size (0.5 $\mu$l *n*-undecane), carrier and hydrogen flow (both at 60 ml/min), and instrument electrometer setting (1000 × 2).

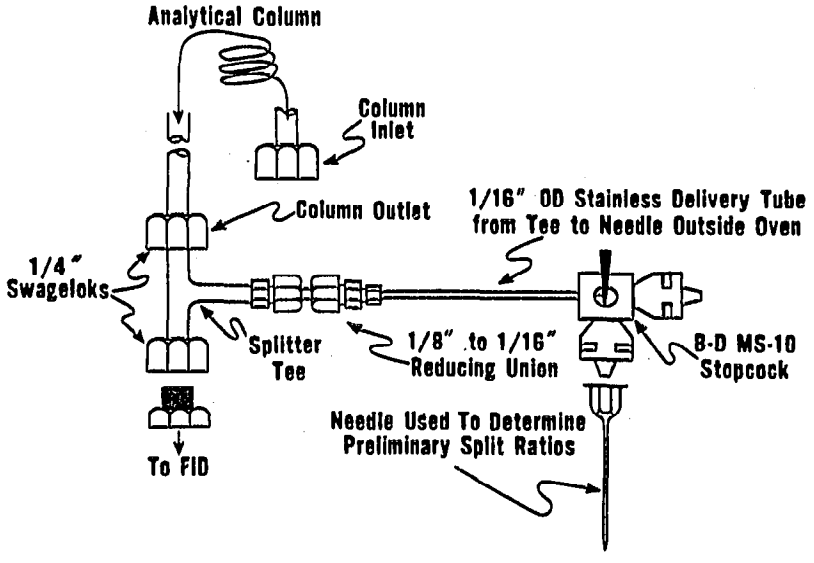


Fig. 1. Diagram of preliminary apparatus (located outside column oven) used to determine split ratios offered by needles of various lengths and gauges.

Split ratios were calculated as the ratio of peak height with the stopcock closed, to send all effluent to the FID, to peak height with the stopcock open, to allow part of the effluent to be exhausted through the needle to the atmosphere. Peak width is

* The mention of firm names or trade products does not constitute an endorsement by the U.S. Department of Agriculture over other firms or similar products not mentioned.

TABLE I

PEAK HEIGHT AND SPLIT RATIOS OFFERED BY 22-GAUGE NEEDLES OF VARIOUS LENGTHS (SPLITTER LOCATED OUTSIDE GAS CHROMATOGRAPH OVEN)

| | *Splitter closed** | *Needle length (in.)* | | |
|---|---|---|---|---|
| | | *3* | *2* | *1* |
| Peak height, cm (extrapolated where necessary) | 40.5 | 14.9 | 8.3 | 0.4 |
| Split ratio | | 3.0:1 | 5.4:1 | 112.5:1 |

* All effluent through flame ionization detector.

a function of elution time and is independent of post-column split ratio; therefore, peak height measurements were used to calculate the split. For example, a split ratio of 2:1 means that the peak recorded when the splitter was closed was twice as tall as when the splitter was open. One-half of the column effluent passed through the FID and was combusted while one-half was allowed to escape to the atmosphere. Table I shows the peak height ratio that each needle length provides.

Based on these preliminary split-ratio determinations, the apparatus diagram-

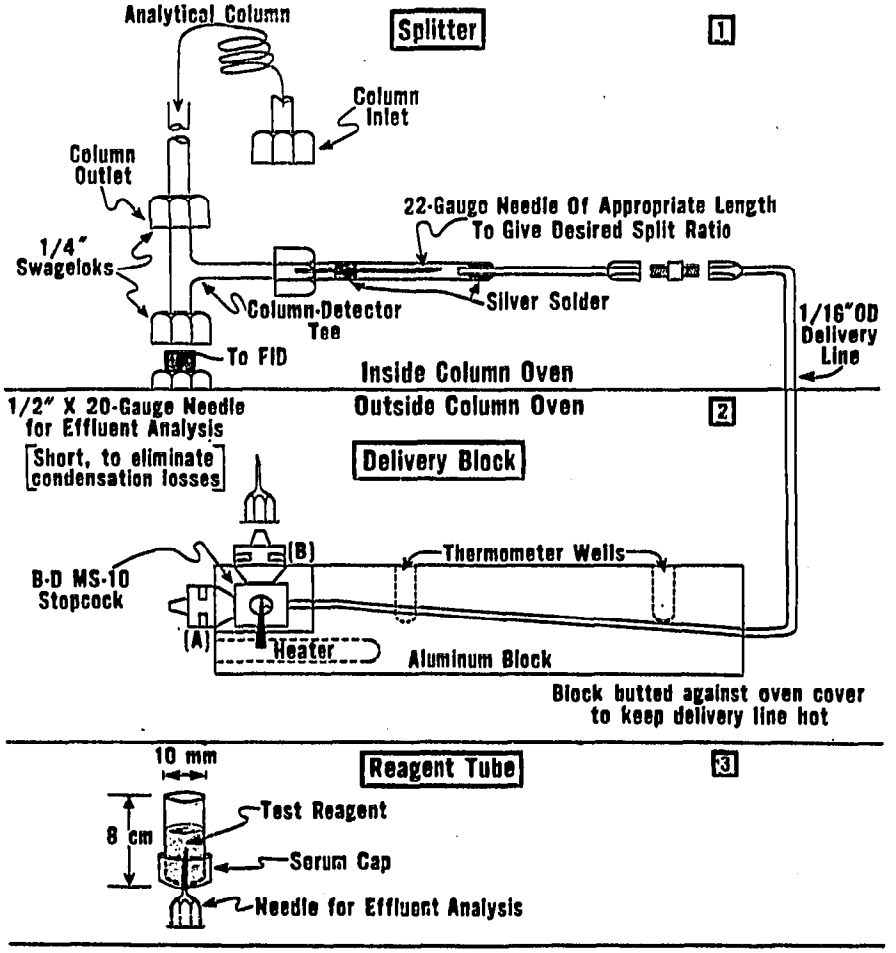


Fig. 2. (1) Inside-oven splitter details. (2) Diagram of delivery block. (3) Test reagent tube.

med in Fig. 2 was built. Its heated block prevents effluent condensation and provides a convenient support for the stopcock and delivery line. Fig. 3 is a photograph of the apparatus with the delivery block, stopcock, and splitter connected to the instrument. The splitter needle is located inside the oven and is directly attached to the column-

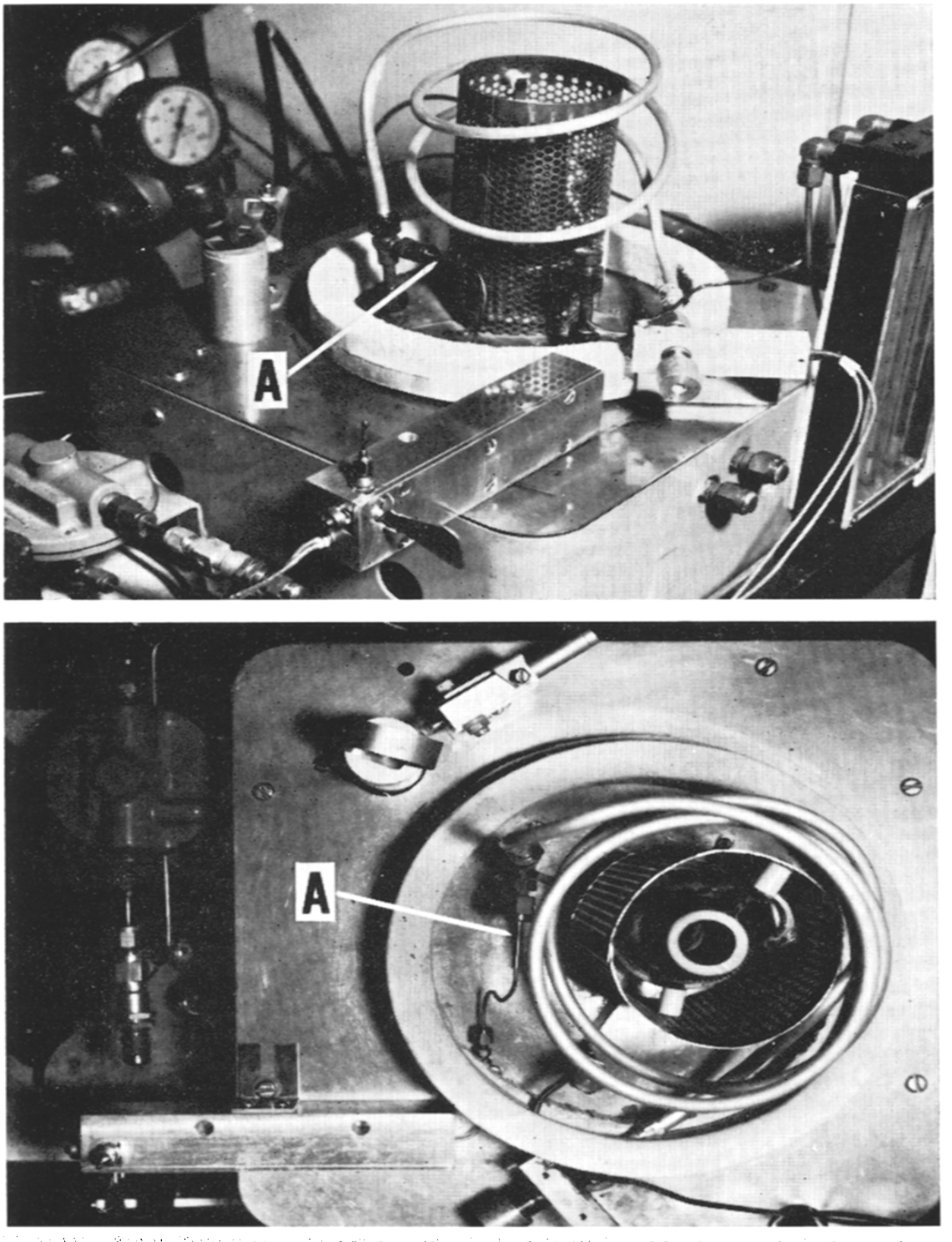


Fig. 3. Photograph of assembled splitter and delivery block connected to chromatograph. Split needle at arrow (A).

detector tee. Table II summarizes split ratios obtained from 22-gauge needles of various lengths. Apparent discrepancies in split ratios between 3-in. needles, listed in Tables I and II, occur because differences in effluent viscosity result when the temperature is changed. When located outside the oven, the split needle allows division of a cooled low-viscosity column effluent. When located inside the oven, the split is

TABLE II

SPLIT RATIOS OFFERED BY 22-GAUGE NEEDLES OF VARIOUS LENGTHS (SPLITTER INSIDE COLUMN OVEN)

| | *Needle length (in.)* | | |
|---|---|---|---|
| | *3* | *1–1/4* | *5/8* |
| Split ratio | 1.7:1 | 3.5:1 | 45:1 |

accomplished on a hot, more viscous, effluent stream. Since the inside splitter divides a higher viscosity effluent that reduces the amount allowed to escape, more effluent is delivered to the FID. Splitters located inside the oven are preferred because condensation plugs those outside.

A 45:1 split from a 5/8-in. needle proved to be the most useful. For this split ratio, the more than adequate sensitivity of an FID allows collection or analysis of 98 % of the column effluent. During temperature programming the amount of effluent diverted to the FID increases. This ratio is low enough to compensate for effluent viscosity changes caused by temperature programming without initially starving the flame of sample. Different needle lengths and diameters make a variety of split ratios available.

The stopcock port (A) on the delivery block, Fig. 2, is a convenient juncture for a multiple-port fraction collector[1]. Port (B) is a convenient position for attachment of a short 20-gauge needle through which the column effluent may be delivered for functional group analysis. The techniques of WALSH AND MERRITT[2] are particularly adaptable to functional group analysis when used with the reagent holder diagrammed in Fig. 2, part 3. When a component emerges from the instrument, the test reagent held in the tube is exposed to the effluent stream by piercing the serum cap with the verticle needle.

This splitter-collection-test unit can be built inexpensively to meet a variety of analytical situations and can be adapted to any gas chromatograph.

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1 R. L. HOFFMANN AND A. SILVEIRA, Jr., *Anal. Chem.*, 36 (1964) 447.
2 J. T. WALSH AND C. MERRITT, Jr., *Anal. Chem.*, 32 (1960) 1378.

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