Research Note

A TRAP-SPLITTER UNIT FOR COUPLING PREPARATIVE GAS CHROMATOGRAPHIC COLUMNS TO ANALYTICAL CAPILLARY COLUMNS[1]

Gas chromatographic techniques, which involve preparative as well as analytical capillary columns, are essential in the separation and subsequent detection of trace components. Previous investigation conducted along this line[2] demonstrated that trace components can be separated with preparative columns without prior solvent pre-concentration, but the direct capillary analysis of the concentrated fractions cannot be obtained readily because commercially available traps and splitters are unsuitable for combining capillary with preparative columns. Coupling capillary to preparative columns presents complex problems because of uneven working temperatures, flow rates and pressures, and large differences in size of samples required for both types of columns. This note presents the development of an integrated trap-splitter unit which overcomes these difficulties.

The gas chromatographic and auxiliary equipment used for the construction and testing of the trap-splitter are described. The trap-splitter is shown in figure 1 and a schematic drawing of the system in figure 2.

In the trap-splitter the trap is made of U-bent 5 mm (i.d.) stainless steel 316 tubing. It allows collection of more than 10 μl. (0.01 ml) of concentrated sample without noticeable interruption of the carrier flow. Liquid N_2 or CO_2 is used in the cold bath, and the sample is evaporated from the trap with a modified flash heater. All tubing employed is stainless steel 316, 1.6 mm o.d., (0.25 mm i.d. for the splitter section, and 0.76 mm i.d. for the expander, inlet and outlet lines). The valves for the inlet, outlet and carrier lines are shut-off type with 3.2 mm orifice for 6.3 mm o.d. tubing, which can withstand temperatures higher than 140°C. A bellows type valve having the same inside dimensions but rated to 816°C, is used in the expander section. Connection between the valves and the capillary tubings is accomplished with 6 to 1.6 mm o.d. stainless steel reducing unions. A toggle-operated shut-off valve is used at the end of the splitter column. The heaters are cartridge type elements regulated with Hewlet Packard 1904A[3] temperature control accessory.

[1] Manuscript submitted to Editorial Board, August 11, 1975.

[2] Batiz, H., and Soltero, E., Preparative-capillary systems for trace analysis of rum, J. Agr. Univ. P.R. 60(4):559–84, 1976.

[3] Trade names are used solely for the purpose of providing specific information. Mention of trade names does not constitute a guarantee or warranty of the equipment by the Agricultural Experiment Station of the University of Puerto Rico or an endorsement over other equipment not mentioned.

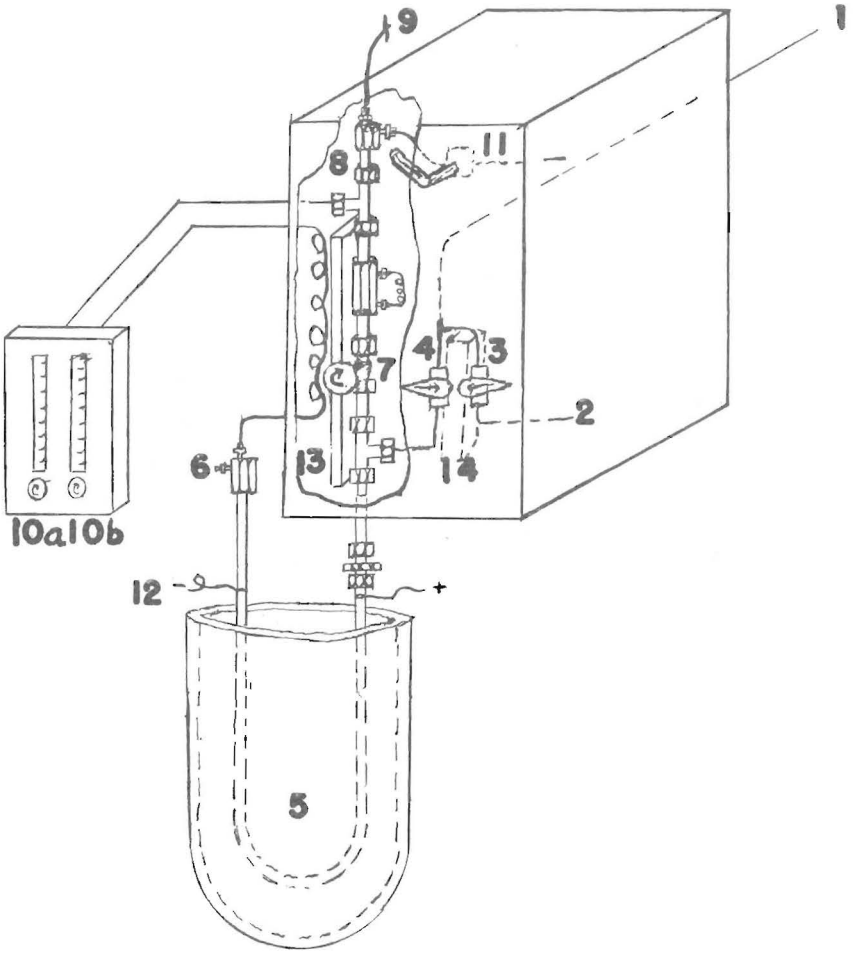

FIG. 1.—Trap-splitter apparatus. 1, sample inlet; 2, sample outlet; 3, and 4, shut-off valves; 5, U-bent trap in liquid N_2 bath; 6, exit; 7, expander with bellows valve; 8, main capillary carrier entrance; 9, capillary column entrance; 10, flowmeter; 11, splitter toggle valve; 12, flash heater; 13, and 14, cartridge heaters.

The sample inlet tubing is heated with a 12 V direct current power supply. Two Hewlett Packard gas chromatographs were used for the tests. The preparative apparatus was a model 5754B provided with both dual flame ionization detector (FDI) and thermal conductivity detector (TCD), a two-pen two-channel recorder, and a heated backflush valve. The analytical chromatograph was a model 5750B with dual FID and a two-pen two-channel recorder. The preparative column was a 3 m × 12.7 mm o.d. stainless steel 316 tube filled with 10% Carbowax 20M on acid

washed 60-80 mesh, Chromosorb W. The capillary column was a small bore capillary Golay column, stainless steel 316, 91 m × 0.25 mm i.d. coated with polyethylene glycol (Ucon) 75H 90,000.

For operating the trap-splitter system (fig. 2), an accurate volume of sample is introduced in the preparative column by syringe injection. The carrier gas and/or sample emerging from the preparative chromatogram goes to the trap-splitter (fig. 1) through inlet 1 and is vented outside when valve 3 is opened and valve 4 is closed. After fractionation, desirable portions of the sample are collected in the trap, previously

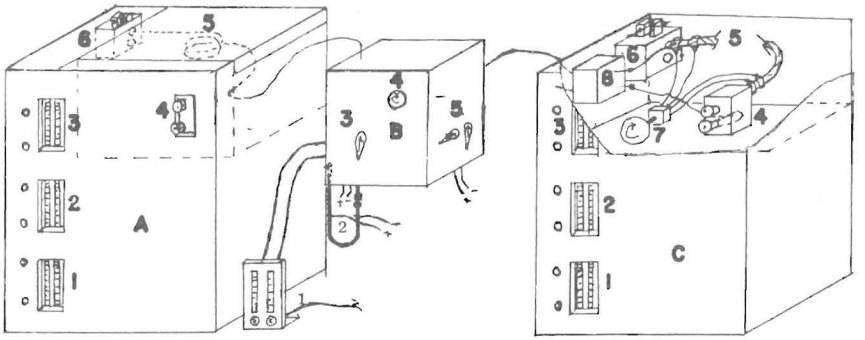

FIG. 2. – Gas chromatographic system for coupling capillary to preparative columns, A and C oven modules 5750B and 57543 gas chromatographs; 1, carrier flowmeter; 2, air flowmeter; 3, hydrogen flowmeter; 4, injection port; 5, column; 6, FID; 7, backflush valve; and 8, TCD. B. Trap-splitter. 1, carrier flowmeter; 2, trap; 3, expander valve; 4, splitter valve; and 5, inlet and outlet valves.

TABLE 1. – *Chromatographic condition for capillary column 91 m × 0.25 mm i.d., Ucon 75 H 90,000 and trap-splitter*

Carrier	He
Make up gas	He at 25 ml/min at 0.42 kg/cm^2
Carrier flow	1 ml/min at 1.68 kg/cm^2
Splitter ratio	1:63
Oven temperature, programmed	10° C/min at ethyl alcohol
Oven temperature, initial	40° C
Oven temperature, final	125° C
FID temperature	200° C
Injection temperature	200° C
Trap-splitter line temperature	
Trap entrance	200° C
Trap outlet	200° C
Inside splitter	150° C
Splitter outlet	130° C
Temperature of trap	200° C for 3 min
Column material	Stainless steel 316

placed in liquid N_2, by simultaneously closing valve 3 and opening valve 4. During this operation trap outlet 6 is kept open and valve 7 closed, while carrier gas is allowed at exit 6, and through main capillary entrance 8 to the capillary column 9 with needle valve of the flowmeter 10 open all the way and valve 11 closed. Components separated with a preparative column are trapped individually or as a single fraction. In order to stop the collection procedure, valve 4 and exit 6 are closed

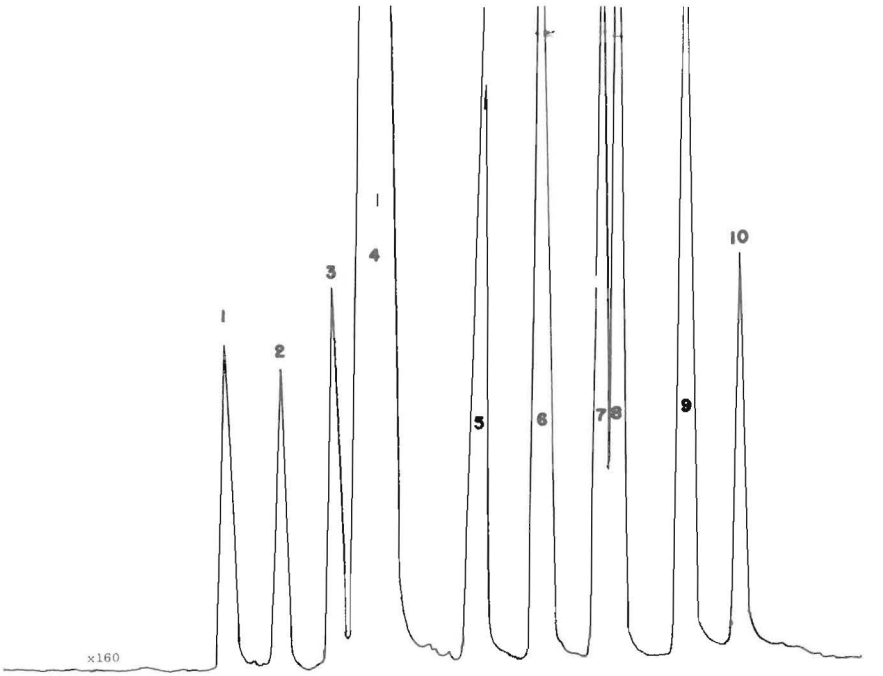

FIG. 3. – Capillary chromatogram of 1 μl reference solution (PC-2). 1, acetaldehyde; 2, methyl acetate; 3, ethyl acetate; 4, ethyl alcohol; 5, propyl alcohol; 6, isoamyl acetate; 7, isobutyl alcohol; 8, butyl alcohol; 9, isoamyl alcohol, 10, amyl alcohol. Recorder speed, 6.3 mm/min.

simultaneously while valve 3 is opened. The carrier gas from the preparative column is vented outside again through outlet 2. The first fraction collected in the trap is analyzed at the capillary column, while the preparative separation continues uninterrupted by opening the splitter valve 11 for 2 or 3 min previous to analysis, then rotameter valve 10a is closed, expander valve 7 is opened, the liquid N_2 bath is removed, and flash heater 12 is heated to about 200°C for several minutes. Finally, the flow through 10a is restored and valves 7 and 11 are closed again. Valve 10b, which supplies carrier gas to the trap is kept open all the time, and

carrier flow through the capillary and preparative column is uninterrupted. Other fractions of the sample are analyzed similarly after undesirable components have been expelled from the preparative column when the trap-splitter is restored to collect position; but any other material trapped at this point of the separation is to be analyzed at the capillary column only after analysis of the preceding fraction is completed.

Reference samples of different volumes (1 μl to 10 μl) were introduced directly into the trap through the preparative column and then analyzed with the capillary column to test the trap-splitter and for subsequent identification of trace components in the capillary chromatograms. The reference sample (PC-2) was made about 4% each of acetaldehyde, methyl acetate, ethyl acetate, acetal, isoamyl acetate, and propyl, butyl, isobutyl, isoamyl and amyl alcohols. The capillary chromatograms obtained by direct injection of 1 μl of a reference sample PC-2 is shown in figure 3 and conditions are given in table 1. All the signals are well resolved even for a 10 μl sample with practically no tailing.

Heriberto Batiz
Eduardo Rosado
Rum Pilot Plant