A micro-fraction collector for gas chromatography*,**

A number of fraction collectors are available commercially for the collection of gas chromatographic peak compounds. However, none of them is suitable for the collection of micro-amounts of fractions of a complicated gas chromatogram. The present non-automatic fraction collector is designed for samples such as flavor compounds isolated from foods. These compounds usually can only be obtained in micro-amounts and yield gas chromatograms of imperfect resolution with more than a dozen peaks.

Construction of the fraction collector

The main portion of the present fraction collector (Fig. 1) consists of one distributor (A) and three manifolds (B). They are constructed of borosilicate glass capillary tubings (6 mm O.D., 1 mm I.D.) and 1 mm bore three way stopcocks (Kontes Glass Company, Vineland, N.J.). The distributor directs the effluent gas from the chromatograph, according to the position of the stopcock (C), either to one of the three manifolds or to an exit port. The latter can be used to eliminate the effluent gas when no peak is indicated by the recorder. Each manifold consists of five stopcocks which can be arranged to direct the effluent gas to any one of the six collection ports. The three manifolds are connected to the distributor through S/J 12/1 joints. The 16 stopcocks are so arranged that the effluent gas can be directed to one collection port or to the exit port by turning only one stopcock.

The distributor and the three manifolds are each imbedded between two aluminum blocks. The upper block (D) is 1 in. in thickness and the lower one (E), 2 in. Channels, 0.1 in. larger in diameter than the capillary tubing, are engraved into each of the aluminum blocks. The clearance between the capillary tubing and the channel is to prevent breakage of the borosilicate glass tubing due to its difference in thermal expansion coefficient from that of aluminum. A well, $^5/_8$ in. diameter and $12^1/_2$ in. long, is drilled into the center of each lower aluminum block. An immersion heater (F) (Blue M Electric Co., No. TH 3012, Blue Island, Ill.) is fitted into each well. The distributor and the three manifolds are independently maintained at constant temperatures by connecting one variable transformer to each immersion heater. A thermocouple is inserted into each of the aluminum blocks at a position close to the capillary tubing. The thermocouples are connected to a pyrometer (G) through a five position rotary switch (H).

Connection of the fraction collector to the gas chromatograph

The distributor is extended to 4 in. from the sample exhaust port of the gas chromatograph through a piece of borosilicate glass capillary tubing with S/J 12/1 joints on both ends. This tubing is connected to the sample exhaust tube of the gas chromatograph through a piece of stainless steel tubing ($^1/_8$ in. O.D., $^1/_{25}$ in. I.D.). The latter is silver soldered on one end to a stainless steel S/J 12/1 joint which fits to the borosilicate capillary tubing. On the other end it has a stainless steel Swagelock fitting which connects to the sample exhaust tube of the gas chromatograph. In order to maintain these

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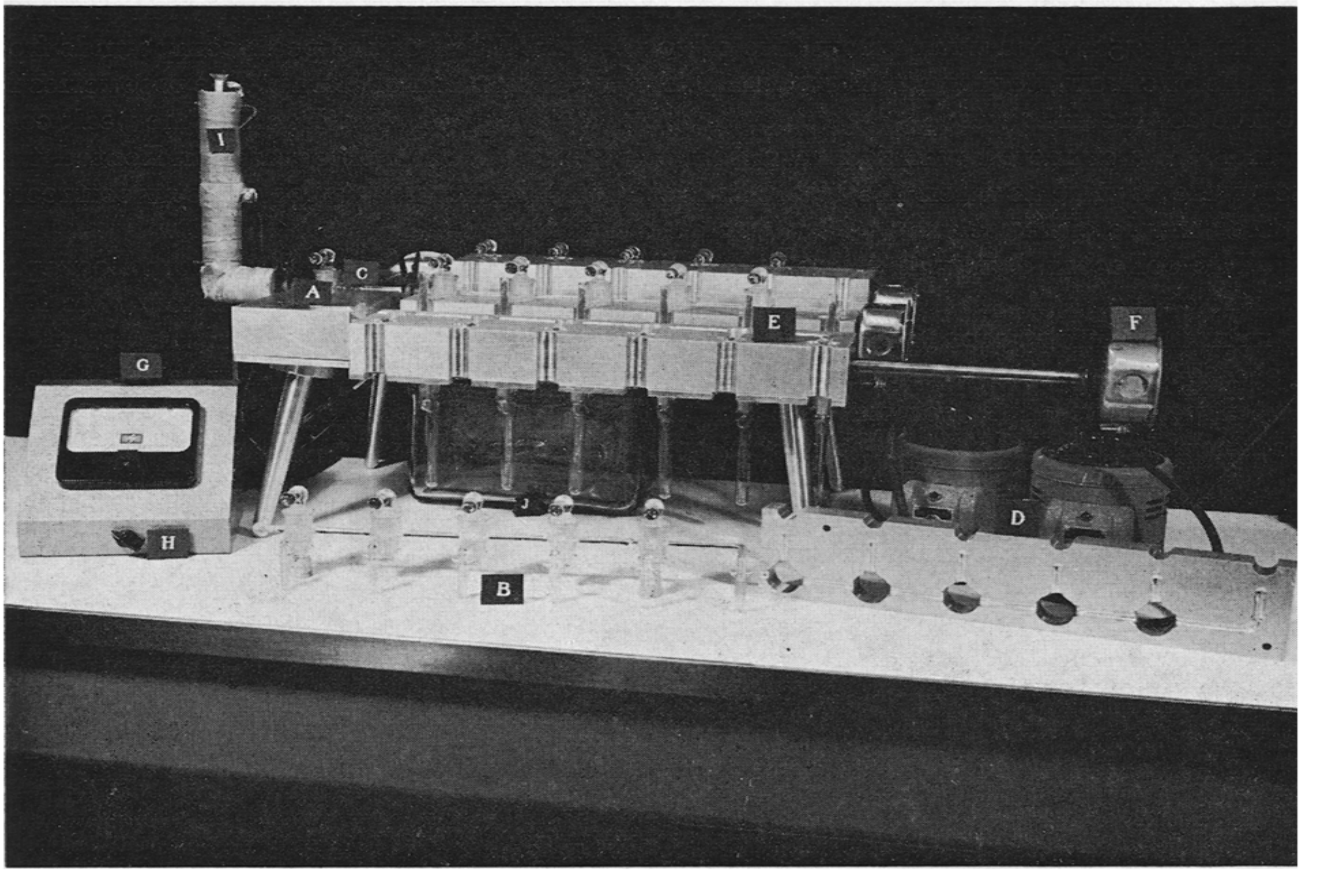

Fig. 1. Fraction collector for gas chromatographic peak compounds.

connecting tubings at a required constant temperature, the capillary glass tubing is imbedded in the center of an aluminum rod (1 in. diameter), which is split into two halves with channels engraved in each of them (I). The aluminum rod is wrapped with a Briskeat heating tape ($^1/_2$ in. wide, 4 ft. long). A thermal couple is inserted into the rod at a position close to the borosilicate glass capillary tubing. This thermocouple is connected to the pyrometer through one position of the rotary switch (H). The stainless steel tubing is inserted into an aluminum tubing ($^1/_4$ in. O.D., $^1/_8$ in. I.D.) which is wrapped with a 10 in. piece of Briskeat heating tape.

Construction of the cold traps

Each of the peak compounds is collected in a cold trap connected to one of the exit ports of the manifolds through a S/T 10/30 borosilicate glass joint. The traps used are the same as that described by CHANG *et al.*[1] except that borosilicate glass capillary tubing (3 mm O.D.), bent into U shape, and restrictions drawn out at every 2 cm, is used to replace the polyethylene capillary tubing.

Performance of the fraction collector

The present fraction collector can be used to trap 18 peak compounds eluted from a gas chromatograph without loss of resolution. A mixture of acetone and isoamyl alcohol, was chromatographed with a Carbowax 1000 column at 130°, and a mixture

of caproic, caprylic and capric methyl esters was chromatographed with a DEGS column at 175°, both with a flow rate of 70 ml/min. Each of the peak compounds from six runs of chromatograph was accumulatively collected in one trap. The recoveries for the five compounds were 95, 100, 94, 90 and 100 % respectively. Each of the peak compounds thus collected was rechromatographed. Their chromatograms indicated that they were as pure as when they were collected with individual traps directly connected to the sample exhaust tube of the gas chromatograph.

The collector does not create measurable back pressure to the chromatographic column even when the flow rate is as high as 150 ml/min. When the flow rate is 60 ml/min, the time lag for the carrier gas to reach the trap farthest from the detector is $^1/_3$ sec. For compounds of a higher boiling point which have a tendency to form "fog", the temperature of the coolant in bath (J) for the cold traps should not be lower than necessary for condensing the compound.

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[1] S. S. CHANG, K. M. BROBST, C. E. IRELAND AND H. TAI, *Appl. Spectry.*, 16 (1962) 106.

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The effect of ultraviolet irradiation on chlorpromazine*

I. Aerobic condition

Introduction

The picture of the metabolic end-products of chlorpromazine in psychotic patients has become clearer in recent years following a number of reports on the isolation and identification of the metabolites. Glucuronidation was found to be the major mechanism of the metabolism of this drug. A number of studies[1–4] concerning the excretion of non-polar metabolites, namely, unchanged chlorpromazine (CP) and its sulfoxides (CPO), have been reported. Chlorpromazine sulfoxide was found to be 1–18 % and unchanged drug was reported to be less than 1 % of the administered dose[2]. The ratio of the unchanged drug to its sulfoxide was approximately 1:16. The CPO was identified to be a mixture of chlorpromazine sulfoxide and two demethylated products, namely Nor_1-CPO and Nor_2-CPO in which Nor_2-CPO predominates. The same findings have been reported by FISHMAN AND GOLDENBERG[5]. In the report[6] on a quantitative analysis of four groups of urinary metabolites in psychotic patients, chlorpromazine glucuronides (CPGL) were found to be the major metabolites. An average of 44.6 % of CPGL was found in urine followed by CPO (7.7 %), $CPOH_1$**

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** $CPOH_1$ = Hydroxyl chlorpromazine (urinary metabolite, R_F 0.57, reacted blue with 50 % H_2SO_4, purple with 5 % $FeCl_3$, I.R.: 2.70–2.85 μ)[6].