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### **Combination of a gas chromatograph and a fraction collector using a small condenser**

It is often necessary, either for rechromatography or for further identification by such techniques as IR spectroscopy or nuclear magnetic resonance, to collect micro amounts of a resolved fraction of a complex mixture as they are eluted from the gas chromatograph. Conventional cold traps are inefficient for trapping high-boiling solutes which tend to form aerosols that are swept through the trap. A number of gas chromatographic traps of varying complexity, designed to prevent the formation of aerosols, have been described in the literature. Such devices may depend on the maintenance of a temperature differential between a heated inner wall and the cooled outer wall<sup>1</sup> or on the use of a cold trap and electrostatic precipitator<sup>2</sup>. A procedure for using argon<sup>3</sup> or carbon dioxide<sup>4</sup> as carrier gas and condensing a carrier gas along with the sample in a trap surrounded by liquid nitrogen has also been described.

The present paper describes a new collection method using simultaneous condensation of a sample and organic solvent vapor in a small Liebig-type condenser. This principle has also been applied to the combination of a gas chromatograph and IR spectrometer using a liquid flow cell<sup>5</sup>.

Many fraction collectors are commercially available and various designs have been published<sup>6,7</sup>. However, only some of them can be built conveniently for the collection of a relatively large number of gas chromatographic fractions. The present paper also describes the combination of a gas chromatograph and fraction collector which is commercially available.

#### *Experimental*

The flow diagram is shown in Fig. 1. The gas lines which connected 1, 2 and 3 in Fig. 1 were heated with tape heaters. The generator of the organic solvent vapor consists of a 500-ml flask and 20-cm resistant glass tube packed with sand or firebrick C-22. They are maintained at 2-7 degrees above the boiling temperature of the solvent<sup>5</sup>. In this experiment tetrachloroethylene was used as an organic solvent.

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The flow rate of the organic solvent vapor was indicated by the amount (mg/min) of tetrachloroethylene vapor condensed by a small condenser. Usually the flow rate of the vapor was set between 100–300 mg/min. It was constant if the temperature of the generator did not change.

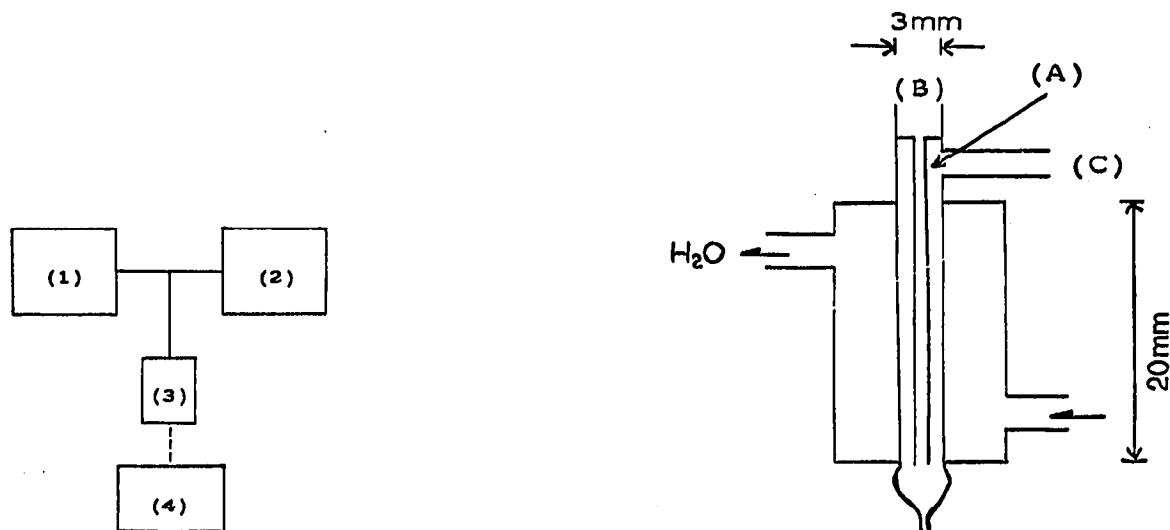


Fig. 1. Flow diagram. (1) Gas chromatograph; (2) generator of the vapor of organic solvent; (3) small condenser; (4) fraction collector.

Fig. 2. A small condenser. (A) Capillary tube; (B) inlet of gas mixture; (C) outlet.

The small condenser which has a small capillary gas inlet is shown in Fig. 2. The vapor of organic solvent was mixed with GC effluent, and the mixture was led into the condenser for the simultaneous condensation of the vapor and sample component. The gas mixture was passed through the capillary gas inlet and collided with the cold glass wall; almost all the condensable vapor was condensed around the outlet of the capillary. Then the condensed liquid was dropped from the condenser to a test tube in the fraction collector. The fraction collector was operated to renew a test tube every 15 sec.

The equipment used included a gas chromatograph with TCD made by Yanagimoto Seisakusho; a fraction collector, model No SF-200A, containing test tubes 13 cm deep with an I.D. of 1.5 cm was made by Toyo Kagaku Sangyo Ltd.; an IR-spectrometer, IR-E, was made by Japan Spectroscopic Co. Ltd.; and an NMR spectrometer, JNM-C-60, was made by Japan Electron Optics Laboratories.

### Results and discussion

Fig. 3 shows the chromatogram with thermo-conductance detector and trapping. The absorbance of the trapped solution of GC fractions was measured using an IR liquid cell. No obvious tailing was observed. This method was applicable to a sample with a boiling point up to 300° without obvious tailing.

*Recoveries of samples.* Recoveries of samples are shown in Table I. The recovery was estimated by the following procedure. For a 1-min elution of a sample from the separation column, the column temperature was adequately set, and the trapped GC fraction was rechromatographed after addition of an internal standard. When the

sample and vapor of organic solvent were condensed by the small condenser, the formation of aerosols was not observed. From Table I, the average recovery is above 80 %, and the higher the boiling point of the sample, the higher the recovery. The recovery of the sample was proportional to the logarithm of the amount of the flow rate of organic vapor<sup>5</sup>. Therefore, the higher average recovery may be obtained when the flow rate of organic vapor is more rapid.

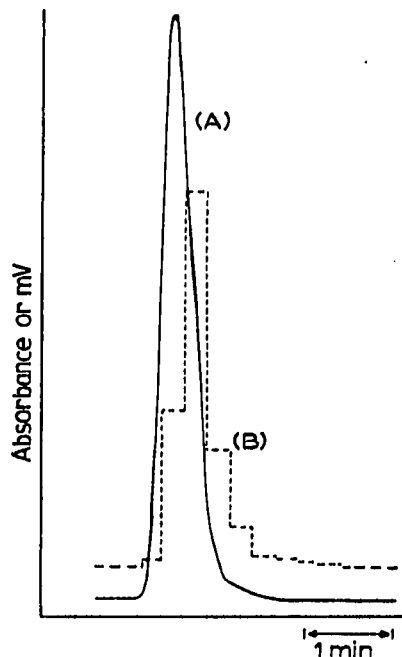


Fig. 3. Comparison of chromatograms with TCD (A) and trapping (B). Sample: *n*-butyl acetate, 5  $\mu$ l; flow rate of  $\text{Cl}_2\text{C}=\text{CCl}_2$  vapor: 290 mg/min; carrier gas:  $\text{H}_2$ , 14 ml/min; column: diethylene glycol sebacate polyester, 5 %; length, 145 cm; temperature, 100°.

TABLE I

RECOVERIES OF SAMPLES

Flow rate of  $\text{Cl}_2\text{C}=\text{CCl}_2$  vapor, approximately 180 mg/min; of carrier gas  $\text{H}_2$ , 20 ml/min.

Sample	Amount of injection (mg)	Recovery (%)
Isoamyl alcohol	2.0	66
Cumene	1.5	77
Cyclohexanone	1.5	82
Methylbenzoate	2.2	90
Diisopropylbenzene	2.9	99

*Performance of the fraction collector connected to a gas chromatograph.* Since the concentration of the sample in the GC fraction is 0.5–10 % when it is in mg, its elution time is about 1 min and the flow rate of the vapor of organic solvent is 100–300 mg per min, its IR or NMR spectrum can be measured without concentrating the solution.

Gas chromatograph and fraction collector were operated on line in Fig. 4. Since 4 tubes of the fraction collector were used in 1 min and the flow rate of the vapor

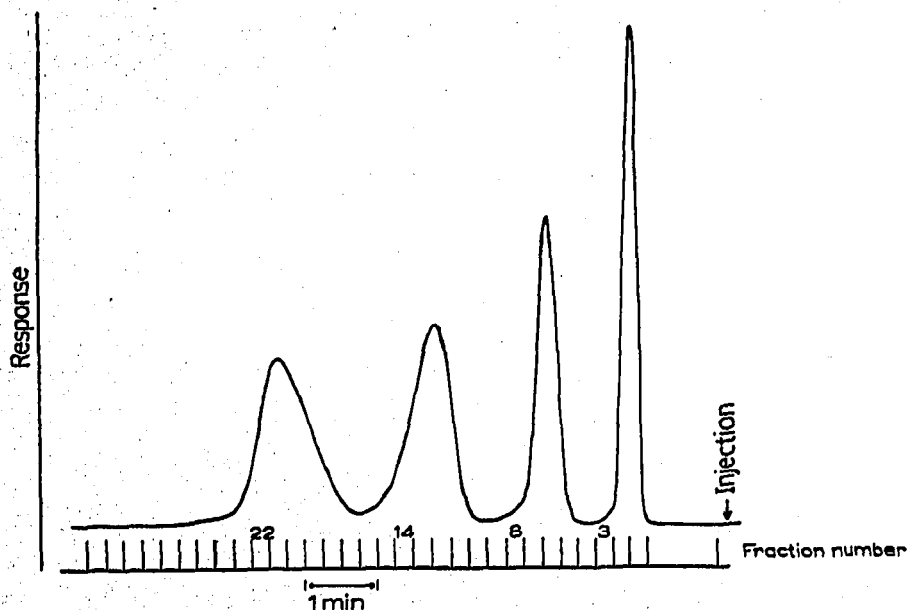


Fig. 4. Combination of gas chromatograph and fraction collector. Sample: a mixture of *n*-decane, diisopropylbenzene, aniline and dimethyl adipate, 10  $\mu$ l; flow rate of  $\text{Cl}_2\text{C}=\text{CCl}_2$ : 240 mg/min; carrier gas:  $\text{H}_2$ , 13 ml/min; cooling of condenser: water; recorder: two pens; one fraction time: 15 sec.

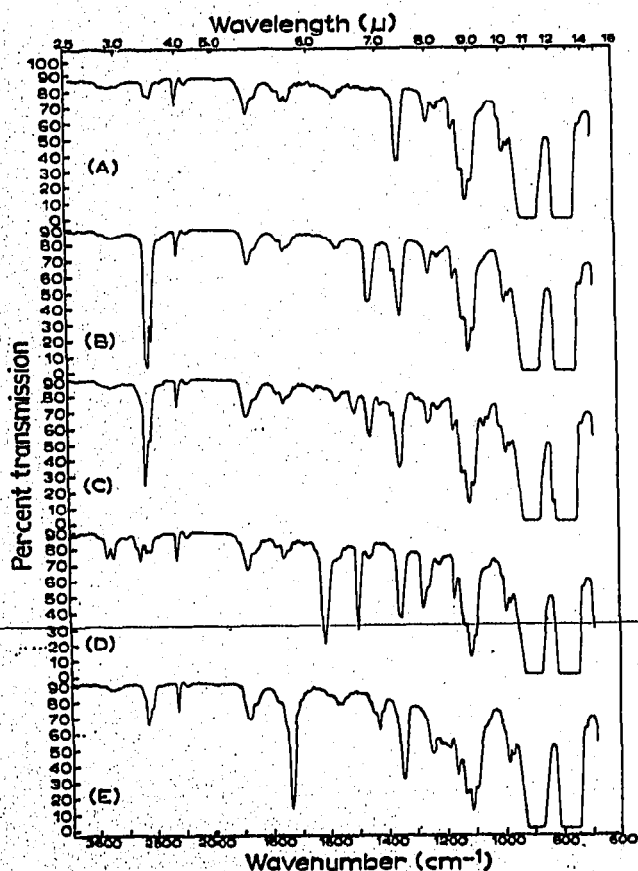


Fig. 5. IR spectrum of each solution of GC fraction. (A) solvent, the condensed liquid of  $\text{Cl}_2\text{C}=\text{CCl}_2$  vapor; (B) fraction number 3, identified as *n*-decane; (C) fraction number 8, identified as diisopropylbenzene; (D) fraction number 14, identified as aniline; (E) fraction number 22, identified as dimethyl adipate.

was 240 mg/min, about 60 mg or 37  $\mu$ l was in one test tube. The amount of one sample component was 2  $\mu$ l. Therefore, the concentration of it in the GC fraction was 1-4%. Thus its IR spectra, shown in Fig. 5, could be measured using the liquid cell in which the cell spacer and volume were 0.4 mm and 30  $\mu$ l, respectively.

NMR spectrum was also measured using a micro tube, made by Kusano Kagakukiki Seisakusho. An equal volume mixture of diisopropylbenzene and ethanol, 6  $\mu$ l, was injected and separated by the gas chromatograph. After trapping, the NMR spectrum of the former was measured (Fig. 6).

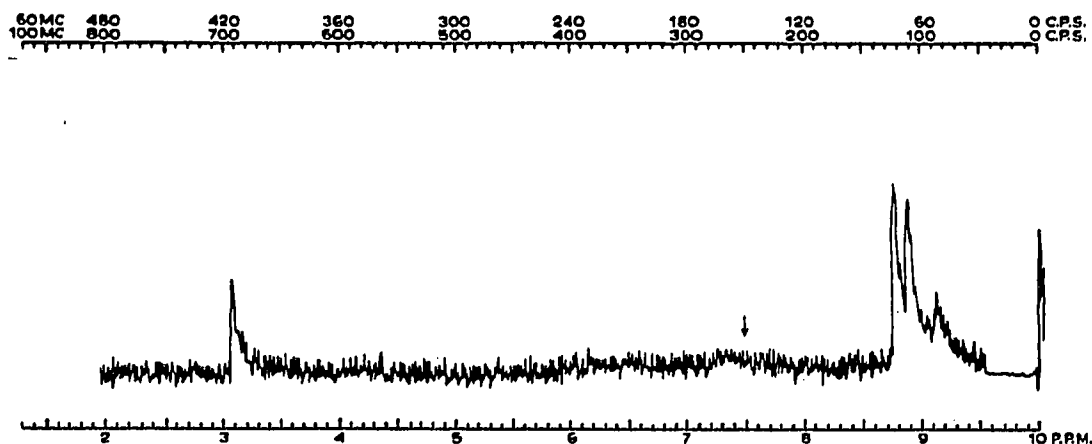


Fig. 6. NMR spectrum of diisopropylbenzene which was trapped by a small condenser. About 130  $\mu$ l of diisopropylbenzene-tetrachloroethylene solution was measured. The signal of 9.13 p.p.m. might come from impurity.

This new method of collecting GC fractions has two merits. First, the recovery of the sample component is comparatively high and the concentration of the sample in the GC fraction is enough to measure its IR or NMR spectrum without a concentration process. Second, the combination of GC and a fraction collector, which is commercially available, can be done easily using the small condenser.

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