Notes

A trap for the collection of a series of fractions in the preparative scale gas chromatography of high molecular weight compounds*

We have been experimenting at this laboratory with the method of preparative scale gas–liquid chromatography for the separation and isolation of pure samples of methyl esters of the polyenoic acids of marine oils. Runs have been carried out on large polyester columns with injected samples of up to 3 g of mixed esters at temperatures around 200° and with nitrogen carrier gas flow rates of 3 l/min. It was found in our earliest experiments that under these conditions ester was eluted from the column as an aerosol which defied quantitative condensation in liquid air traps. A study of the literature indicated that the problem of aerosol formation in the use of this technique was well known and its cause recognised[1]. A number of devices for overcoming it have been reported, the best known of these probably being the VOLMAN collector[2], in which the formation of a persistent aerosol is prevented by passing the effluent through a large temperature gradient. A variation on the VOLMAN trap has also been reported[3]. Other methods based on electrostatic precipitation[4] and re-vaporizing the aerosol in a single hot zone followed by secondary condensation in a falling temperature gradient[5] have also been published. A method recently described is based on passage of the column effluent through a number of consecutive hot and cold zones, the trap being constructed by simple modification of a reflux coil condenser[6].

Not one of these devices entirely fulfilled our immediate requirements, which included simplicity of construction, ready availability of parts and, probably most important, the suitability for collection of a series of fractions at timed intervals with rapid removal of the fraction from the trap, leaving it uncontaminated at the end of each period. This last requirement was necessary as our laboratory-made preparative chromatograph was not fitted with a detector. Fractions were collected as stated above and each was then examined for its contents on an analytical gas chromatograph.

In attempts to trap the effluent from the chromatograph quantitatively, a series of Drechsel bottles fitted with sintered glass distributors and containing acetone were connected in series with the outlet. Acetone was used as the solvent since it dissolved both the esters and the small amount of polyester "bleed" from the column. Separation of the fatty acid ester from the polyester can be effected by dissolution in petroleum ether, in which the polyester is insoluble. A series of three such bottles appeared to be an effective scrubber. It was decided to construct a vessel which would have the effective scrubbing properties of the three Drechsel bottles and which could be easily emptied without removal from the outlet of the chromatograph.

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Construction of the trap

This is best described by reference to Fig. 1.

The trap consists of a central tube (E) fitted with three sealed-in sintered glass discs (F) of porosity No. 1 and with two side limbs (D) and (G). A water condenser (A) is fitted at the B14 joint at the top of E. The limb D has a B14 socket which carries a stoppered dropping funnel (B) and also a short side arm which connects the trap through the ball and socket union (C) to the heated outlet of the chromatograph. A short extension of the outlet internal to the ball and socket was found to reduce losses and serves to prevent intercontamination of fractions due to condensation at this point. The outlet (H) on the limb (G) is closed by a sealed tube carrying a B10 socket which fits over the outlet and is held secure by a spring attached to lugs on both parts.

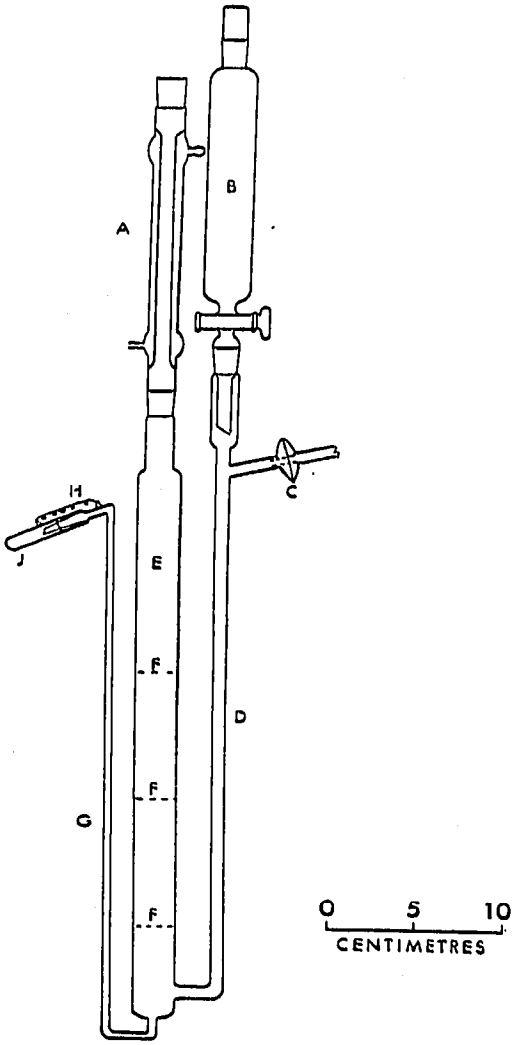

Fig. 1. Diagram of G.L.C. collection trap.

Operation of the trap

With the sealing tube (J) in place, the dropping funnel (B) is filled with acetone. The tap is opened and by applying pressure by means of a rubber blow-ball fitted with a B14 cone to the top of the funnel, solvent is forced into the main chamber.

This is continued until there is a layer of solvent about one inch deep above the upper sintered disc and rather deeper layers on the other two. The tap is closed and the funnel stoppered. The trap is now ready for scrubbing the effluent gas. In order to cleanse any condensate from the limb (D), a slow solvent drip from the funnel may be used or alternatively a quick wash from the tap-funnel may be given before changing fractions. To change fractions the procedure is as follows: The tap-funnel (B) is closed and lifted from its socket. This allows the sealing tube (J) to be removed without discharging the trap. On replacing the tap-funnel the solution in that part of the trap under the lower disc is discharged through tube H into the collecting vessel by the gas flow from the chromatograph. The solvent above the discs is then forced into the lower part of the trap and expelled through tube H by gentle pumping with a rubber blow-ball at the top of the condenser. If it is so desired, a little wash solvent can be added at the top of the condenser and then pumped through. The seal (J) is then replaced and the trap re-filled with solvent as before. The procedure, which in description is rather complex, is in practice very simple and a change of fraction can be performed in less than one minute. Consideration has been given to the possibility of sophistication of the trap, for example by the incorporation of various taps to make discharge automatic but it was felt that this would probably be obtained only at the expense of increased fragility, at risk of contamination, and with little or no gain in efficacy.

On test runs with samples of fatty acid methyl esters, recoveries have been better than 95 %. The trap is easily constructed by a glass-blower, all parts being readily available. (The main chamber of our original trap was made by joining three sintered glass Gooch crucibles together.) It appears to have certain advantages over the traps hitherto described in that it is suited to the collection of a large series of fractions and requires no electrical connections, sources of heat or refrigeration.

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