Volman collector for gas chromatography

The problem of aerosol formation, or "fogging", is a normal complication of gas chromatographic purification of liquids. It is particularly troublesome with high molecular weight compounds, and results in low trapping efficiency. If traps are packed with defatted cotton, glass wool, glass beads, silica, or other materials, their efficiency for small samples can be increased, but it becomes necessary to rinse the packing with a suitable solvent to recover the sample. An additional disadvantage is that packed traps are prone to clog with large samples. An electrical precipitator has

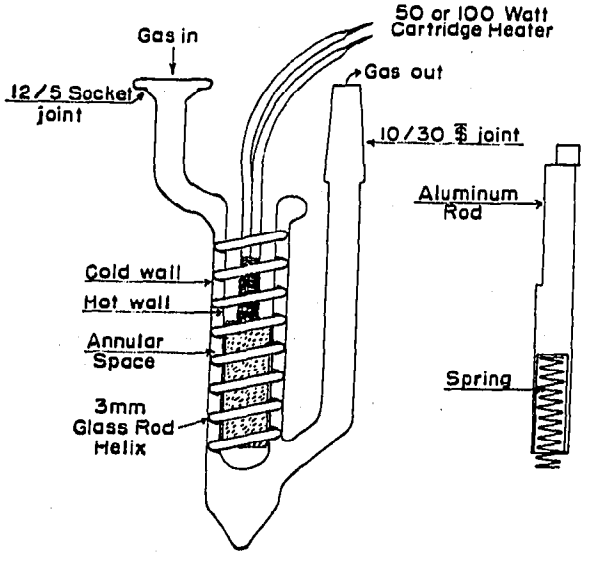

Fig. 1. Trap with cartridge or aluminum rod heaters.

been suggested[1,2] but the dangers of such a trap are obvious. The Volman trap, which can be made by any competent glass blower, is very effective in stopping the fogging phenomenon and, therefore, is practical for preparative work in gas chromatography. A large temperature gradient in the trap prevents fog formation[3].

Our variation of the Volman trap (Fig. 1) permits its use for collecting fractions from gas chromatographic runs. The gases emerging from the apparatus are forced in a spiral path down the annular space between the center well wall, heated by the cartridge, and the outer wall, cooled by ice, dry ice, or liquid nitrogen. The glass helix was found necessary in order to force the gas to take a longer path than without the helix; *i.e.*, some aerosol was observed in the effluent gas stream from the traps without the helix. If the cartridge heater is used, a thermocouple can be inserted with the heater in order to adjust the thermal gradient. Temperatures of 150–200° have been found to be sufficient for most cases.

For chromatographs that suspend traps from a rotating heated plate (*e.g.*, the Megachrom*), a spring-loaded aluminum rod held in contact with the plate can be used to conduct the heat to the center well (Fig. 1, inset). This eliminates the need for electrical wiring and permits assembly to rotate freely with the fraction cutter.

* Reference to a company or product name does not imply approval or recommendation of the product by the Department of Agriculture to the exclusion of others that may be suitable.

If the sample size is in the milligram region, then the material must be vacuum transferred. If the sample size is in the gram region, a Teflon tube can be inserted down the gas outlet tube of the collector, and the sample removed with a syringe or bulb. The collector can then be rinsed clean with appropriate solvents, dried, and thus be ready for another collection.

Trapping yield data have been obtained with ethyl caproate and limonene. In the 1-gram range, yields of 90–95 % were obtained with ice as the coolant. Yields slightly higher than 95 % were obtained with dry ice as the coolant.

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The isolation of DDT, parathion and lindane from biological fatty materials by liquid–liquid partition chromatography

The isolation of organic insecticides from biological fatty matter constitutes a fundamental step for their ultimate quantitative estimation. While studying the applicability of some of the methods described in the literature, the work of JONES AND RIDDICK[1] and that of BURCKFIELD AND STORRS[2] stimulated us to develop liquid–liquid partition chromatographic columns for the isolation of the above-mentioned insecticides.

JONES AND RIDDICK isolated several insecticides by partitioning them between acetonitrile and *n*-hexane. BURCKFIELD AND STORRS replaced acetonitrile by N,N-dimethyl-formamide. By using these solvent pairs in chromatographic columns we succeeded in recovering milligram quantities of DDT, parathion and lindane for more than 90 % out of their solutions in concentrated insect extractives or in peanut oil. One of the advantages of these columns is the possibility of using them more than once.

Application of this method to microgram quantities, often occurring in biological material, could not at the moment be checked owing to the lack of a detection method of adequate sensitivity.

Methods for isolating the insecticides

(1) *Isolation of DDT.* Celite 545 (JOHNS MANVILLE), from which the fines have been slibbed off, is washed with concentrated hydrochloric acid and then with distilled water and dried. Of this material 5 g are mixed thoroughly with 2 g of dimethyl-