

CHROM. 37II

### Improved preparative collections from the Autoprep gas chromatograph

With the continual improvement of equipment, preparative-scale gas-liquid chromatography has become increasingly important for the isolation of many types of volatile compounds. However, the abrupt temperature change encountered at the column exit port frequently gives rise to stable aerosols which drastically reduce the efficiency of collection.

Application of the principle of the Cottrell precipitator for the collection of gas chromatograph eluates not otherwise trapped because of aerosol formation has been reported by several investigators<sup>1-3</sup>. The most recent paper<sup>3</sup> describes a modification primarily designed for the F & M model 770 preparative gas chromatograph.

We have devised a simple modification of the widely used Aerograph Model A-700 Autoprep (Varian Aerograph Inc., Walnut Creek, Calif.) (Fig. 1) to provide for the collection of aerosols by electrostatic precipitation. This instrument utilizes a

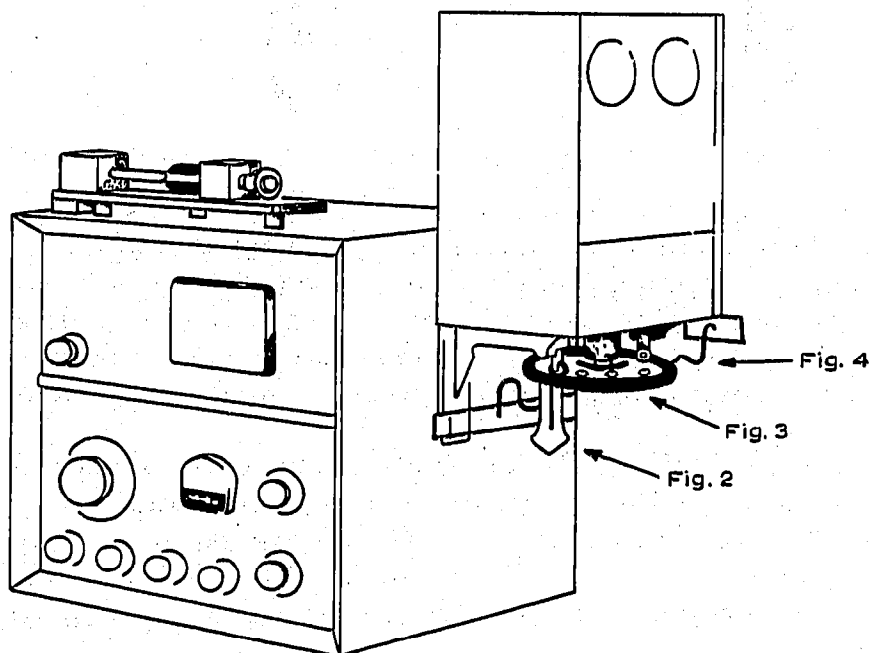


Fig. 1. Complete preparative chromatograph.

circular collector table to which collection bottles are attached. The collector table is rotated into place automatically upon activation by the recorder response so that the carrier gas and the vaporized sample are introduced serially into each collector bottle at the appropriate time.

Modification of the usual commercial collector bottle (Fig. 2a) allows the effluent from the chromatograph to pass through an electrostatic field which causes precipitation of the sample from the carrier gas. Cutting off the bent portion of the exit side (Fig. 2b) permits insertion of an inner electrode which is constructed of spring wire (A) embedded in a glass capillary (B) with epoxy resin and is held in the center

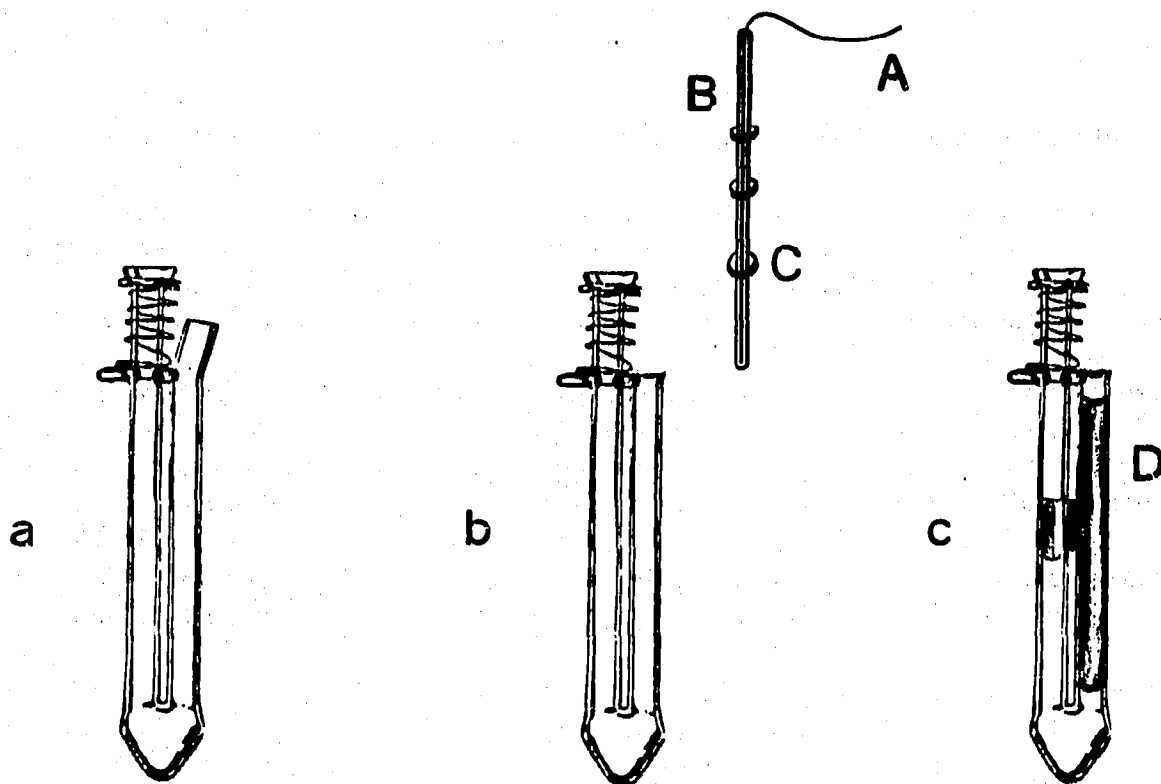


Fig. 2. Collector bottle detail (see text).

of the exit tube by Teflon spacers (C) cut from sheet with a cork-borer. This design permits withdrawal of the electrode to facilitate removal of the collected sample.

The outer electrode is formed by wrapping aluminum foil tape ("Permacel") (D) around the exit side of the collector bottle (Fig. 2c) and can be readily removed after the collection has been completed. Wide variation in the dimensions of collector bottles is possible so long as the effluent is passed through an electrostatic field of sufficient intensity.

In order to provide high voltage safely to the inner electrode A, the standard collector table E was modified as shown in Fig. 3. An upper conducting metal ring, F, was cemented with epoxy resin to a larger insulator ring G constructed of a 1/8 in. (3 mm) thickness of hardboard. A similar pair of rings was cemented to the lower side of the collector table, and the F rings were connected electrically by a Teflon-insulated piece of 12 gauge copper wire, H.

A sketch of the assembled collector (Fig. 4) shows the relationship of the modified units to each other. The upper end of each electrode wire and a spring wire brush, I, were arranged to maintain continuous contact with opposite F rings. To complete the high-voltage circuit, a short length of wire, J, was attached to the strip of insulation material K bolted to the collector mounting bracket. The wire was shaped to make contact with the metal foil on the collector bottle as it moved to the collection position. The metal chromatograph cabinet was grounded.

The high voltage required was supplied by a transformer with a 120 VAC primary and a 6000 VAC secondary (such as a commercial neon-light transformer). Voltage to the electrodes was varied by adjustment of the primary voltage with an autotransformer (Variac). To determine the voltage required for precipitation, a

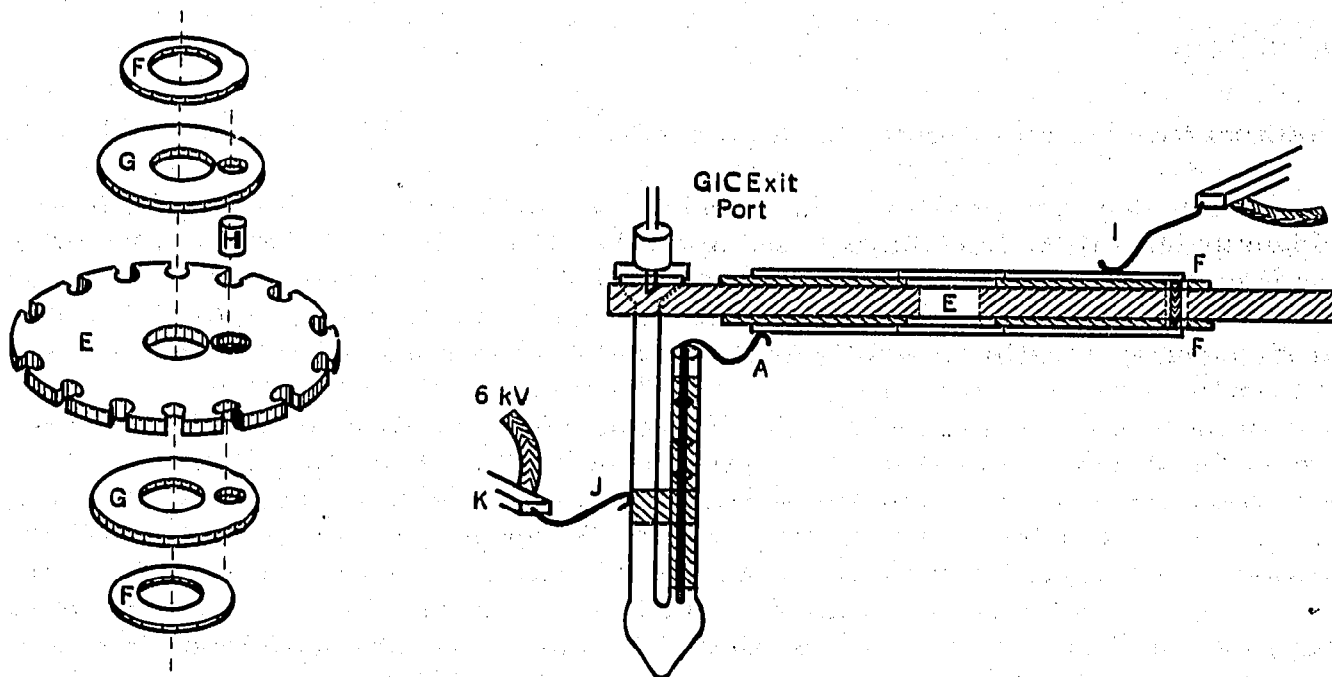


Fig. 3. Collector table detail (see text).

Fig. 4. Bottle in place for collection.

sample was injected with power at zero; when the desired fraction passed through the collector bottle, the voltage was increased until emergence of the aerosol from the bottle no longer was observed. A primary voltage of 50–60 VAC was required for the modified collector bottle described above.

This electrostatic collector has been used successfully with samples in which each fraction represented approximately 5 mg of collected material per injection on a 1.6 cm  $\times$  150 cm column; collection efficiency was essentially quantitative. Collected fractions were rechromatographed and checked by infrared spectroscopy for signs of degradation due to the electrostatic field; no evidence of degradation was ever observed.

A Plexiglass shield with an interlocking safety switch can be readily incorporated and provides complete safety for the operator.

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Financial support, in part, by the Chevron Chemical Company and the U.S. Public Health Service (Grant ES-00054) is gratefully acknowledged.

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