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Device for automatic preparative gas chromatographic collections on an equal time basis

In our work on the identification of volatile components of natural products, it was necessary to separate the complex material into a number of arbitrary crude fractions by preparative scale gas chromatography (GC).

We had available for this purpose the widely-used Aerograph Model A705 Autoprep gas chromatograph (Varian Aerograph Inc., Walnut Creek, Calif.). This is an automated preparative scale gas chromatograph which controls fraction collection by changing trap positions at a pre-set, but always identical, signal level of the recorder.

In complex natural products the relative concentrations of the components vary by a factor of a thousand or more and the recorder-actuated trap-changing switch is not applicable. One solution to this problem is to collect at a variably adjusted response level (*i.e.* use a peak height discriminator); however, this is not possible with the Autoprep. Another solution that has been proposed is to utilize a collection programmer[1] which incorporates a complex sensing and actuating mechanism to effect trap changing. Although such a system would achieve the required results, a simpler device was desired. Still another method for actuating the trap changing mechanism is to collect on an equal time basis, using a timer system. Although GC collection on a time basis has been suggested before[2,3] such a modification to a commercial instrument has not been previously described. Such a system is effective only if retention times are reasonably reproducible, and the Autoprep demonstrates acceptable reproducibility. The present communication describes a simple, inexpensive device to change trapping positions on an equal time basis.

The present modification, designed to allow collection on an equal time basis,

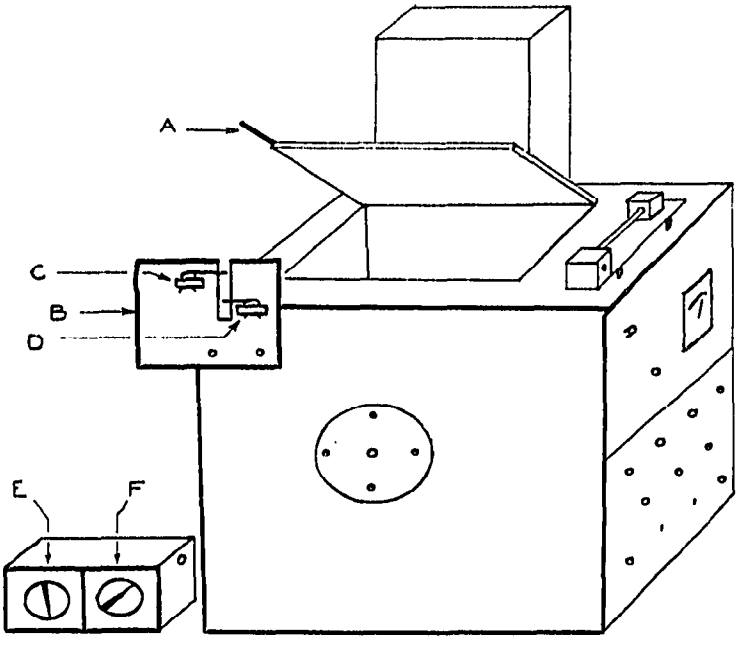

Fig. 1. Modifications to preparative chromatograph.

is shown in Fig. 1. The $\frac{1}{4}$ in. steel rod A is attached to the left side of the oven door and allowed to extend about $1\frac{1}{2}$ in. The 6 × 6 in. steel plate B which has a $\frac{1}{2} \times 3$ in. slot is attached to the rear left side of the instrument and adjusted so rod A will pass through the slot when the door closes. Micro-switch C is modified by cutting off the standard arm and replacing it with a piece of spring steel and is positioned as shown. This is wired in the normally open position in parallel with the Autoprep manual turntable advance pushbutton switch. It is positioned so that the contact is closed briefly by rod A as the oven door is closed. Micro-switch D is positioned such that the contacts are closed by rod A when the oven door is closed, and open when the oven door is open. Micro-switch D is wired such that when closed by rod A it actuates a Dual-Trol timer (Industrial Timer Corporation, Parsippany, N.J.). The Dual-Trol is a recycling timer consisting of two individual timing mechanisms sequentially pulsing a latch relay. When micro-switch D is closed, timer E, continuously variable between 0 and 5 min, will start its cycle. When the pointer reaches 0, the load contacts are closed and timer F, variable between 0 and 6 sec, is actuated. When timer F completes its cycle it restarts timer E and opens the load contact. This sequence continues until microswitch D is opened. The load contacts of timer F are connected to the leads of the recorder peak sensor switch which normally actuates the collector turntable. As long as the oven door is closed the collector turntable will rotate at equal time intervals, as determined by the time setting on timer E. In operation, timer E is set for the approximate collection interval (*i.e. ca.* 2 min); timer F is set for about 0.5 sec.

In the completely automated temperature programming mode, the following sequence of events occurs: as sample injection occurs and the heater is turned on, the oven door closes striking switch C and closing switch D. Switch C positions trap 1 in the collection position; switch D actuates timer E. At the end of the first collection cycle (*i.e.* 2 min) timer F is actuated momentarily and this actuates the turntable thus placing trap 2 in position. This sequence continues until the selected number of fractions has been collected. At this time the door opens, switch D opens, collection is terminated and the timers reset.

One of the advantages of this system is that up to sixteen fractions can be collected in a reproducible manner. In addition, by utilizing switch C the first trap can be positioned immediately after injection, thus allowing components with short retention times to be collected. The primary disadvantage of this system is that certain components will overlap two fractions if turntable rotation occurs while a peak is emerging.

Although this device was designed with the Aerograph Autoprep in mind, a similar modification should be possible for other automatic preparative GC units which collect at a constant signal response level.

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