

## Note

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### Use of Tenax-GC in single sample analysis of gas-liquid mixtures

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The analysis of a complex mixture of permanent gases and heavy organic compounds presents an interesting and difficult chromatographic problem, since no single column or phase can be expected to completely resolve a variety of components so different in molecular weights and boiling points. A number of investigators have approached this problem by utilizing a diverse number of chromatographic columns, each specifically tailored for a particular group of compounds<sup>1-6</sup>. Multiple-column methods either split the effluent into columns connected in parallel or the columns are connected in series and switching valves are used for routing the desired components into the appropriate column.

All of the previously reported methods work well for the particular application for which they were designed; they all however suffer from at least one of the following disadvantages: (1) inability to handle large amounts of water; (2) use of a molecular sieve column which accumulates non-eluted material and requires periodic regeneration or replacement; (3) need for polarity reversal of the detector.

Since its introduction into the market nearly a decade ago, Tenax-GC has proven invaluable in gas chromatographic analyses. Numerous papers have been published on its use in trace analysis, headspace analysis and as a column packing<sup>7-10</sup>. Due to its hydrophobic nature, Tenax-GC has the ability of adsorbing, reversibly, most organic compounds while not retaining water and the lighter permanent gases. It is this property of Tenax which was used in the method reported here.

This paper describes a method for the analysis of gas-liquid mixtures which makes use of a short precolumn of Tenax to adsorb quantitatively all the organic compounds while allowing permanent gases and water to be resolved using a combination of porous polymer columns. The Tenax is then thermally desorbed into a second chromatographic column for analysis of all other compounds. A system of valves allows the same injector and detector to be used for both parts of the analysis, thereby eliminating the need for the reversal of polarity.

## EXPERIMENTAL

### *Apparatus*

All work was carried out using an F & M Model 810 gas chromatograph equipped with a thermal conductivity detector. The instrument was modified as shown in Fig. 1 by addition of the switching valves V1 and V2 (Valco Instrument Co.,

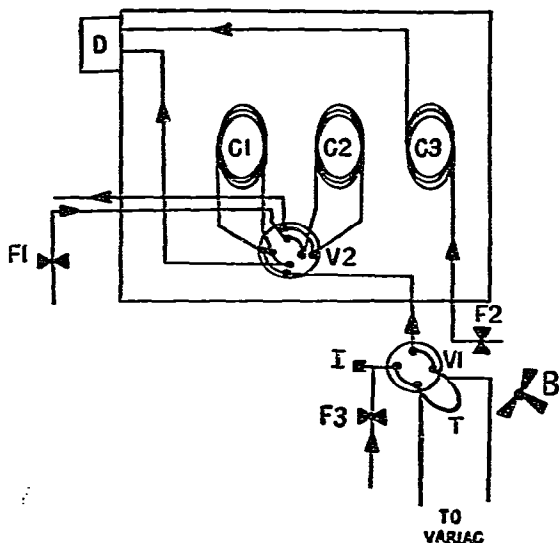


Fig. 1. Schematic layout of the modified apparatus. I = Injector; F1, F2 and F3 = flow regulators; V1 and V2 = switching valves; T = Tenax loop; D = T.C. detector; C1 and C2 = analytical columns; C3 = reference column packed with uncoated Chromosorb W; B = blower.

Houston, Texas, U.S.A.) and by making minor modifications to the pneumatic system. The injector, I, was fashioned from a short piece of metal tubing and a Swagelok "tee" fitted with a septum.

The adsorbent loop, T, contained *ca.* 0.3 g of Tenax-GC and was insulated with asbestos and wrapped with a heating tape connected to a variable transformer. This loop was maintained near room temperature by blowing air from a small cooling fan at all times, except during the desorption step. The temperature of the loop was monitored by means of an iron-Constantan thermocouple.

Helium was employed exclusively as the carrier gas and the flows through all columns were adjusted to 30 ml/min. The temperatures of the injector and detector were maintained at 250° and valve V1 was separately heated to 200°.

The column employed for the analysis of the permanent gases and water (C1) was stainless steel (6 ft.  $\times$  0.125 in. O.D.) packed with Chromosorb 101, connected in series with a stainless-steel column (3 ft.  $\times$  0.125 in. O.D.) packed with Porapak QS. The organic compounds were analyzed using a glass column (10 ft.  $\times$  2 mm I.D.) packed with 10% XE-60 deposited on Chromosorb W HP (C2). The gases were analyzed isothermally at 75° and then the temperature was programmed at 10°/min to 135° for the rest of the analysis.

### Procedure

The valve switching arrangement is shown in Fig. 2. The gas chromatographic analyses are carried out as follows. A sample of the appropriate volume is introduced through the septum in the injector, I, at which point it is volatilized and carried through the sample loop, T. Valve V1 is in position C and valve V2 is in position A.

The sample passes through the Tenax, which retains all the organic compounds while allowing the water and the permanent gases to pass into column, C1, where

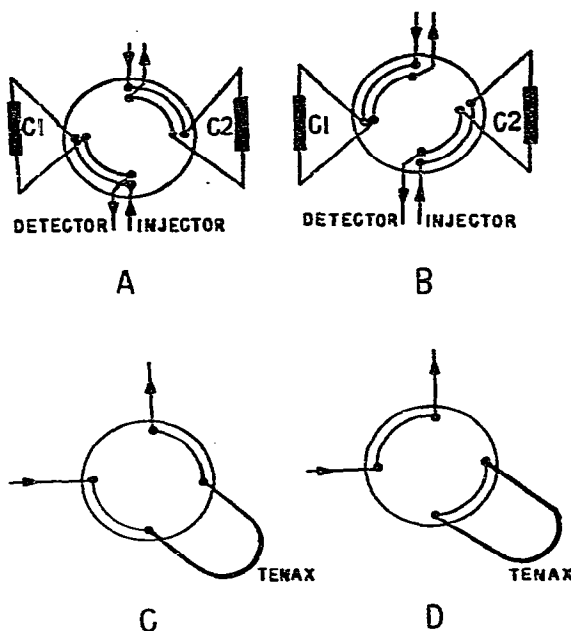


Fig. 2. Switching valves arrangements. Positions A and B refer to valve V2. Positions C and D refer to valve V1.

they are resolved and analyzed. After that portion of the analysis is completed, valve V2 is switched to position B and the Tenax loop is isolated by moving valve V1 to position D.

Desorption of the Tenax is accomplished by heating the loop to 230°. The valve (V1) is then switched to its former position and the desorbed gases are purged through valve V2 and into column C2 for analysis.

## RESULTS AND DISCUSSION

The primary purpose of our investigation was to develop an analytical method for the analysis of mixtures of terpene aldehydes and alcohols containing large amounts of water (>95%) and dissolved gases. Fig. 3 shows a chromatogram of such a synthetic mixture. The separation of all the desired compounds was achieved in as short a time as possible (under 25 min) and the system was able to handle an excessive amount of water with little "tailing" showing in the chromatogram.

A major concern during this work was whether the Tenax was able to adsorb all the organic material in a quantitative manner. Novotny *et al.*<sup>9</sup> have shown that Tenax has a saturation point past which normally adsorbed compounds will not be retained but we expected quantitative adsorption because of the relatively large amount of Tenax contained in the loop.

The ability of the polymer to completely adsorb all of the organic material was demonstrated by injecting equal volumes of the standard mixture diluted to various concentrations with acetone, and observing the relative signal of several choice component peaks which were desorbed from the Tenax. Fig. 4 shows the straight

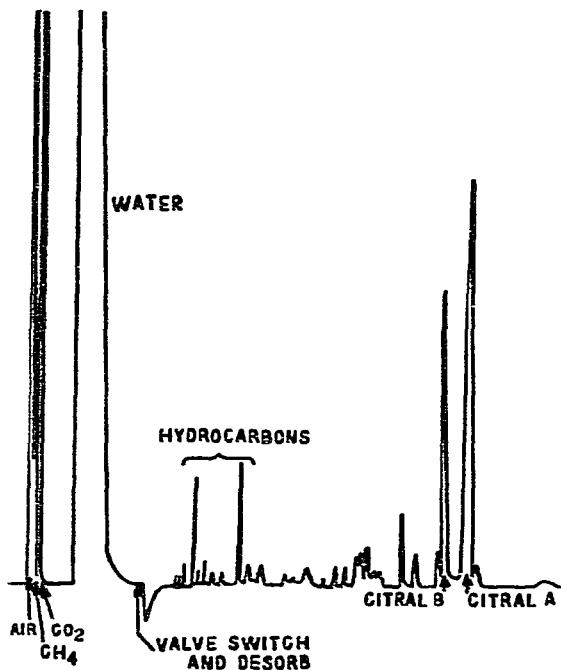


Fig. 3. Typical chromatogram of gas-liquid mixture containing a large amount of water. Chromatographic conditions are given in the Experimental. Peak identification as labeled.

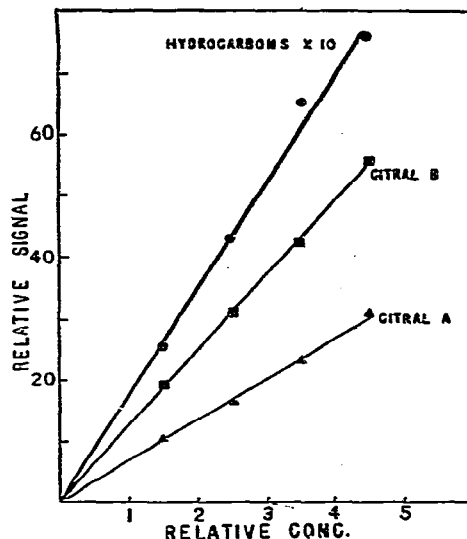


Fig. 4. Analytical curves obtained by analyses of various concentrations of the synthetic test mixture.

line relationship that exists between the concentration of the standard and signal. This indicates that the Tenax is, in fact, trapping all or nearly all of the organic material and desorbing it quantitatively. Further, the straightness of the analytical curve makes the utilization of external standards for quantization very desirable.

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