A Comparison of Carrier Gases Upon Chromatograms When Using a Flame Ionization Detector with Sub-Ambient Temperature Programming*

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In gas liquid chromatography the choice of a carrier gas has not been considered as critical as in gas solid chromatography wherein a polar gas may compete with the solute for adsorption sites. The effect of carrier gas on the sensitivity of response of a gas density balance has been reported recently (1). Similarly it has been observed that partition coefficients are dependent on column pressures of the carrier gas and that the absolute retention of a solute is affected by the carrier gas above ambient temperature (2, 3).

Merritt and Walsh (4) reported that the sensitivity of response of an argon ionization detector was enhanced 15-20 fold when sub-ambient temperature programming was used in the separation and detection of volatiles from such food products as coffee aroma and irradiated beef. These observations have been confirmed in this laboratory using a Sr^{90} Argon ionization detector; however, in addition, it was observed that the different gases had an unexplainable and a very noticeable effect on the response to these components when sub-ambient temperature programming was used in conjunction with a flame ionization detector. This paper describes some of the results of this study.

A Barber-Colman, Model 10****, gas chromatograph unit was used for the study. It was equipped with a flame ionization detector and a sub-ambient temperature apparatus as described by Merritt and Walsh (4). A 25 ft. coil of 0.02 inch i.d. stainless steel tubing was placed in the cell bath (150°C), between the column and detector to prevent flame quenching. The columns were of Pyrex glass (5 mm by 6 ft.) packed with 10% B B′-iminodipropionitrile on 60-80 mesh Firebrick (gas-chrom R, Applied Science Laboratories, Inc., State College, Pa.). Partition of the volatile components was accomplished at a constant pressure of 18 psig (5). Flow rate was measured for each carrier gas at the split exit of the column and found to be comparable (32 ml/min) for the carrier gases used over the programmed temperature range. Argon, nitrogen, helium and carbon dioxide were selected as carrier gases. Dry ice-ethanol was used as the coolant liquid, and the temperature was programmed from sub-ambient (−48°C) to room temperature (4). Each run was carried out over a period of 240 minutes. The column was then equilibrated for 1 hr. at room temperature with a different carrier gas, and then the column cooled to sub-ambient temperature from 10-15 min. before injection of the sample. In no case were peaks observed during the equilibration period.

Imported garlic oil (supplied by Fritzsche Brothers, Inc., New York, New York) was used as the source of volatile components. A five ml sample of the garlic oil vapor was analyzed by gas chromatography. It was observed that response to components in the mixture was related to the carrier gas (Fig. 1). The response to components decreased in the order of nitrogen, argon, carbon dioxide and helium. The use of nitrogen and argon at sub-ambient temperatures resulted in comparable responses by the detector. However, compared to the other gases a very striking difference was observed when helium was used as indicated by the reduction in number of peaks. Possibly these observations may be due to differences in the solubility of the carrier gases in the stationary phase used and to carrier gas-solute interactions over this temperature range. Consequently for analysis of garlic oil vapor at programmed sub-ambient temperatures, either nitrogen or argon could be recommended as providing the best results whereas helium and carbon dioxide are unsatisfactory as carrier gases.

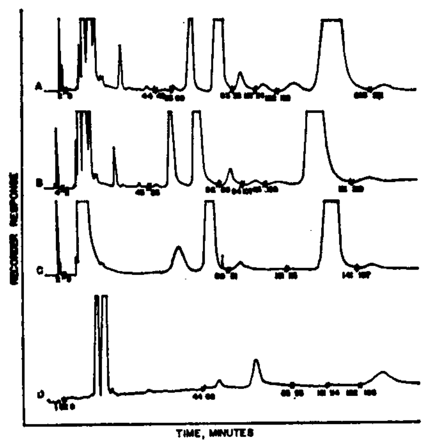

Figure 1. Chromatograph of imported garlic oil programmed from —48° to 26°C using different carrier gases. A= Nitrogen, B = Argon, C = Carbon dioxide, D = Helium.

In addition to garlic oil vapor, cigarette smoke was subjected to analysis. Similar results were observed even though cigarette smoke is different in its chemical composition.

One should not assume that helium or any one of the carrier gases should not be used in sub-ambient temperature programming in GLC when using the flame ionization detector. The selection of a carrier gas for sub-ambient temperature programming should be considered an important factor as illustrated in this paper. ■

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Literature Cited

1. Guillemin, C. L., and Amicourt, Melle F., J. Gas Chromatog. **2,** (5), 156 (1964).
2. Locke, D. C., and Brandt, W. W., 4th Intern. Gas Chromatog. Symp., Michigan State Univ., East Lansing, Mich. (June 17-21, 1963).
3. Desty, D. H., Goldup, A., Luckhurst, G. R., and Swanton, W. T., Gas Chromatography (1962) M. Van Sway ed., pp. 67-81, Butterworths, London, 1962.
4. Merritt, C., Jr., and Walsh, J. T., Anal. Chem. **35,** (1), 110, (1963).
5. Rowan, Robert, Jr., Anal. Chem. **34,** (9), 1042, (1962).

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