Short Communication

A new method for programmed temperature gas chromatography at high sensitivities*

The dual-column technique used in temperature-programmed gas chromatography[1] appears to be applicable only at low or moderate sensitivities. At high sensitivities the two columns and all the components of the "dual" system must be perfectly matched. Such matched columns were unobtainable in our laboratory. Temperature programming at high sensitivities is particularly difficult when liquid phases of higher volatility must be used. Unfortunately, many of the highly selective liquid phases are also excessively volatile. For the solution of this problem we made use of the following observation: the rate of migration of the vapors of a volatile liquid phase in another liquid phase (of lower volatility) was found to be extremely slow at the maximum operable temperature of the more volatile liquid phase. This fact was used to prevent the column bleed from entering the high sensitivity detector by attaching a short "bleed-absorbing column" to the partitioning column. The bleed-absorbing column consisted of an ordinary short column containing a low volatility liquid phase which was attached to the outlet of the analytical column. For instance, an 8-in. column, 1/8-in. O.D., packed with 15% w/w of Carbowax 20M-TPA on Anakrom ABS 70—80 mesh which had been preconditioned at 245°, was found to be suitable as bleed-absorbing column when attached to a 37 ft., 1/8-in. O.D., column packed with 1% w/w of Tergitol NP-35 on 60–80 mesh Chromosorb G (DMCS treated). Temperature-programmed runs of the Tergitol NP-35 column with and without the bleed-absorbing column, using an Aerograph Model 660 flame ionization gas chromatograph at its highest sensitivity, showed the successful application of the bleed absorption principle in single-column temperature programming. Without the bleed-absorbing column the Tergitol column was inoperable above 125°, whereas with a freshly preconditioned Carbowax 20M-TPA bleed-absorbing column the Tergitol column could be programmed up to 190° without any drift of the baseline. Bleed-absorbing columns must be frequently "reactivated", *i.e.*, preconditioned in order to elute from them the volatile liquid phase absorbed during the temperature programmed runs. The liquid phase for the bleed-absorbing column must also be carefully selected to match the properties of the liquid phase used in the partitioning column as far as the order of elution is concerned.

The details of this work and the application of the technique for the reduction of

* This work was supported by the National Research Council of Canada and Distillers Corporation Limited.

the background mass spectra in temperature programmed gas chromatography—mass spectrometry will be published shortly.

Department of Plant Science and Department of Chemistry, University of Manitoba, Winnipeg (Canada)

R. L. LEVY*
H. D. GESSER
F. W. HOUGEN

1 W. E. HARRIS AND H. W. HABGOOD, *Programmed Temperature Gas Chromatography*, Wiley, New York, 1966, p. 228.

Received June 24th, 1967

* Present address: Midwest Research Institute, Kansas City, Mo. 64110, U.S.A.

Notes

Observations on the gas chromatographic behaviour of some amines used in anorectic formulations

Medicinal amines have been analyzed by gas–liquid chromatography in this laboratory without evidence of decomposition, *e.g.* amphetamine[1], the ephedrines[2], lignocaine[3], and 34 others[4]. In contrast, VAN ZWOL[5] recently reported that the gas chromatographic separation and identification of ten sympathomimetic amines, using large amounts of stationary phase on non-KOH coated supports, was difficult owing to amine decomposition. Chromatographic conditions similar to those used by VAN ZWOL were therefore investigated to clarify the discrepancy and the reported decomposition.

Experimental

Perkin-Elmer F 11 gas chromatographs, with hydrogen-flame ionization detectors, were used together with Leeds and Northrup Type G (0–5 mV) and Hitachi 159 (0–2.5 mV) recorders.

Column packing materials and operating conditions are listed in Table I.

Solutions of the ten amines* studied by VAN ZWOL were obtained by extraction of alkaline aqueous solutions of their corresponding salts with freshly distilled AnalaR diethyl ether. Each solution contained approximately 4 μg base per μl (slightly less for phenylpropanolamine because of water favourable partition characteristics). A solution of all ten amines was prepared by mixing 5 ml of each concentrated ethereal solution.

Results and discussion

Chromatography of 1 μl of the mixture of the ten amines on the 22.5 % Carbo-

* Amphetamine, methylamphetamine, isopropylhexedrine, phenylpropanolamine, diethylpropion, phenmetrazine, phendimetrazine, chlorphentermine, methylphenidate and phentermine.