Analysis of Industrial Essential Oils

The following is a summary of one of the papers presented at a Meeting of the South East Region and the Chromatography and Electrophoresis Group held on May 26th, 1977, at Chelsea College, London.

Some Practical Experiences of Reporting Integrators in Industrial Essential Oil Analysis

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A reporting integrator integrates a chromatographic signal in the conventional sense by giving peak-retention times and areas, etc., with the added feature of thermally plotting

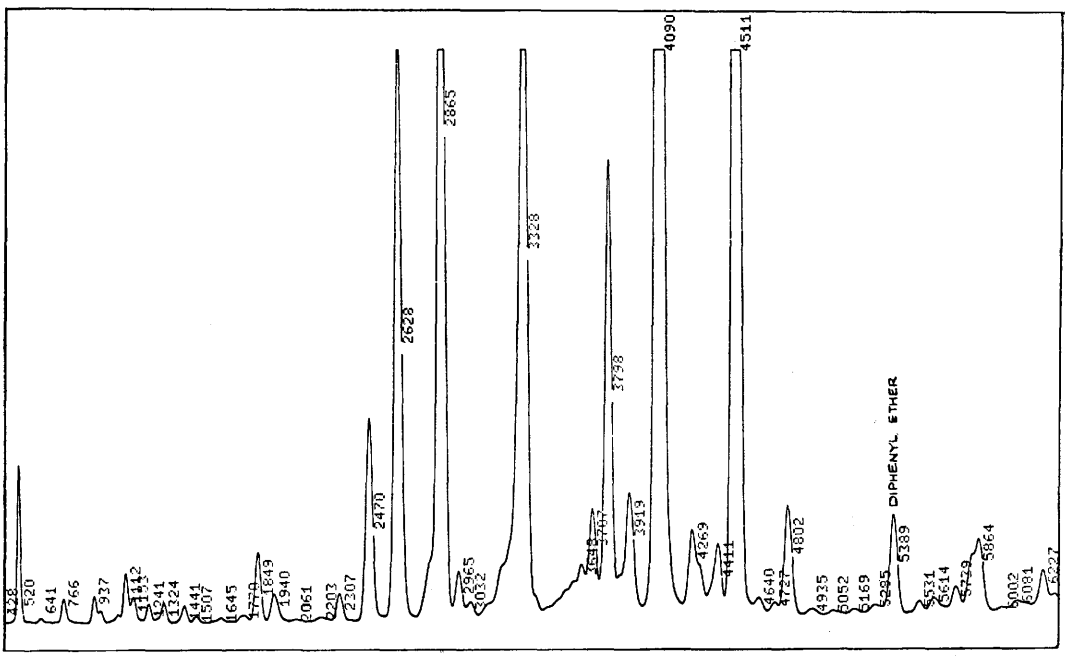

Fig. 1. Chromatogram of a commercial geranium bourbon oil sample. The numbers on the peaks are retention times in 1/100 min.

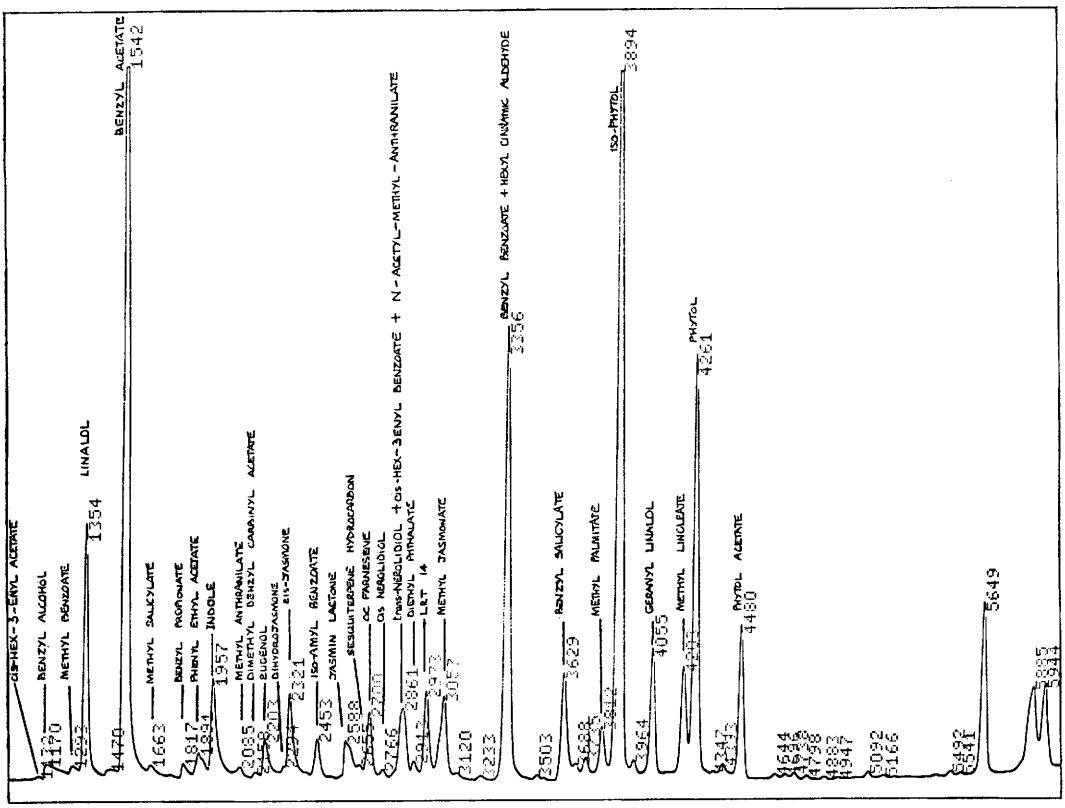

Fig. 2. Chromatogram of an Italian jasmin absolute oil sample obtained on column 1. The numbers on the peaks are retention times in 1/100 min.

the chromatogram. The integrals and chromatograms are tabulated as a combined report on heat-sensitive paper. The features of reporting integrators compared to a conventional integrator plus recorder are as follows.

(*i*) Convenience: all the data and the chromatogram are on one piece of chart, making storage easier, particularly on microfilm.

(*ii*) Ease of calculation: the chromatogram has retention times printed next to the peaks, which is easier for reference and retention index calculations, especially with complex chromatograms.

(*iii*) Reliability: after 20 000 reports, the only servicing done by the author has been to clean air filters.

(*iv*) Chart cost: the cost per foot is similar to that for the accurately gridded recorder chart.

Some reporting integrators can be used directly for calculating the Kováts index and the temperature-programmed retention index, including tabulating with the report. Examples were shown.

A report of a commercial geranium bourbon oil sample was shown, in which an unusual peak was found at the 1% level. This was shown to be diphenyl ether by a retention index of **1 374** on stationary phase SE-30 and of **2 013** on Carbowax 20M, both columns being standardised.[1] The diphenyl ether does not occur naturally, but has a strong geranium-type odour and was probably added in order to enhance an inferior oil. Fig. 1 shows a chromatogram obtained by using a column of 15% Carbowax 20M on Chromosorb W AW DMCS support, 2.10 m long, 4 mm in diameter, of 4 000 theoretical plates, polarity 1.277, and temperature programmed from 75 to 225 °C at 2 °C min^{-1}.

Italian jasmin absolute oil, selected by odour as being the best of 30 jasmine samples in

1976, was critically analysed. Two SE-30 columns were used: 1, of 3 000 theoretical plates, 1.50 m by 4 mm, polarity 1.004, programmed from 60 to 300 °C at 4 °C min^{-1}; and 2, of 200 000 theoretical plates, 50-m wall-coated open tubular capillary, programmed from 80 to 310 °C at 5 °C min^{-1} after "splitless injection." The chromatograms obtained by use of columns 1 and 2 are shown in Figs. 2 and 3, respectively.

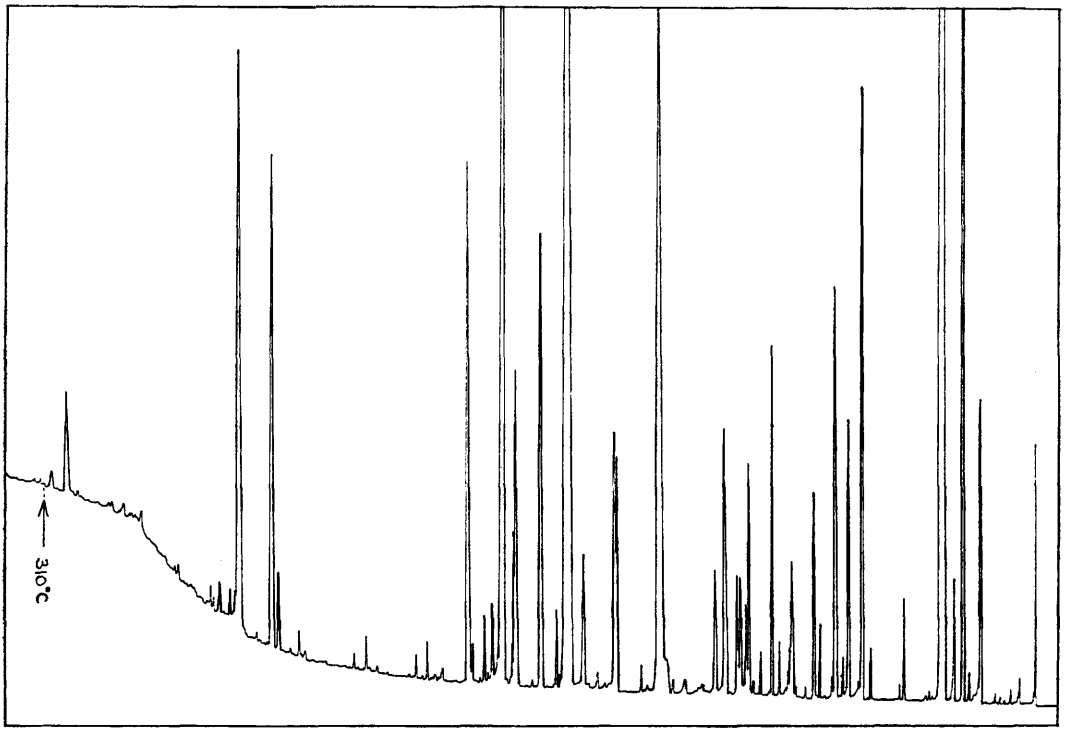

Fig. 3. Chromatogram of an Italian jasmin absolute oil sample obtained on column 2.

Column 1 gave 57 peaks, 34 of which were identified; most of the others were components specific to jasmin or components too small to identify with certainty (<0.1%). Gas chromatography - mass spectrometry was used for the identification and gas chromatography - infrared spectrometry and retention indices were used for confirmation. Column 2 revealed 180 peaks. This sample was of interest because four of the major components occurring naturally are also available as high-purity synthetics at low cost, *viz.*, linalol, benzyl acetate, benzyl benzoate and isophytol. Some of the more odoriferous, less abundant components were also present in higher amounts than in other samples, *viz.*, indole, the jasmones and anthranilates. This sample also contains many components thought not to occur naturally in jasmin, *e.g.*, diethyl phthalate and LRT 14 (2-pentyl-2-acetonylcyclopentanone).

I express my appreciation for the technical help given on the jasmin sample by R. J. H. Duprey and Barrie Frost.

Reference

1. van den Dool, H., "Standardisation of Gas Chromatographic Analysis of Essential Oils," Rijksuniversiteit te Groningen—Proefschrift, Rotterdam, 1974.