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Note

High-performance liquid chromatographic separation of shallot volatile oil

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The use of high-performance liquid chromatography (HPLC) for the separation of essential oils has already been tried on several occasions and has been discussed by Ross[1]. However, the column resolving power is often a limiting factor.

In the work reported here, a volatile oil from shallots was separated by HPLC and more than 90 peaks were detected by a UV detector. The resolution offered by HPLC is better than that offered by capillary column gas–liquid chromatography, which gives less than 90 peaks[2]. Six fractions obtained by HPLC separation can be identified directly by gas chromatography–mass spectrometry (GC–MS) without concentration; however, most other fractions have not yet been analysed.

EXPERIMENTAL

Shallots were purchased from the local market, chopped and steam-distilled by the AOAC method[3] to give volatile shallot oil.

HPLC was carried out with a Hewlett-Packard 1084B system with a 25 cm × 6.2 mm I.D. DuPont pre-packed Zorbax ODS (6 μm particles). Reagent-grade methanol was purchased from E. Merck (Darmstadt, G.F.R.), distilled using a 30-plate Kontes Oldershaw column and filtered through a 0.5-μm fluorocarbon filter (Millipore, Bedford, MA, U.S.A.). The methanol and Milli-Q deionized water (Millipore) were degassed for 30 min by stirring under vacuum before being used as eluents.

The shallot oil was diluted 50-fold with distilled methanol and filtered through a 0.2-μm fluorocarbon filter (Millipore), and 30 μl of the diluted shallot oil were applied to the HPLC system. Detection was effected with a Hewlett-Packard HP 79870A UV detector (254 nm). The solvent system was 62.5% B (methanol) in A (water) for the first 5 min, then a linear gradient programmed from 62.5% B to 90% B in the test 65 min, at a constant flow-rate of 2 ml/min.

Fractions were collected from each HPLC run and injected directly into a Hewlett-Packard 5985B GC–MS system equipped with a 74 cm × 2 mm I.D. glass column packed with 2% OV-101 + 0.2% Carbowax 20M on 100–120-mesh Chromosorb W HP. The operating conditions of mass spectrometer were ionization voltage 70 eV, source temperature 200°C and accelerating voltage 1800 V.

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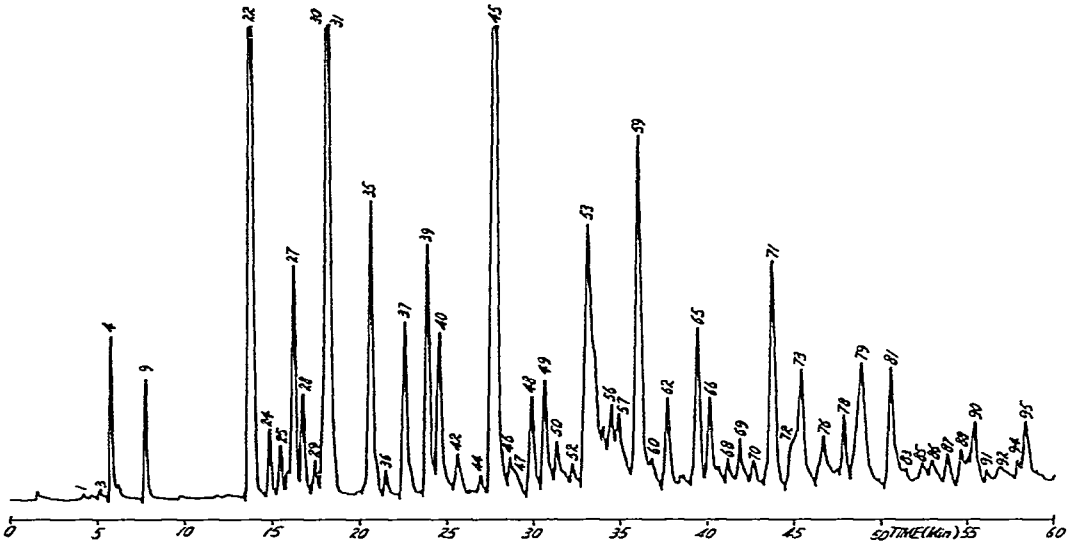

Fig. 1. High-performance liquid chromatogram of shallot oil.

RESULTS AND DISCUSSION

Fig. 1 shows the separation of shallot oil by HPLC. The integration of the peaks in Fig. 1 is shown in Table I. These data are valuable if HPLC is used for

TABLE I

PERCENTAGE OF EACH PEAK RELATIVE TO THE TOTAL PEAK AREA IN THE HIGH-PERFORMANCE LIQUID CHROMATOGRAM OF SHALLOT OIL

Percentages were calculated according to the absorbance of each peak at 254 nm. Peak Nos. ref to Fig. 1.

Peak No.	*Percentage*	*Peak No.*	*Percentage*
4	1.057	56	1.270
9	0.996	59, 60	6.684
22	9.895	62	1.304
24	0.754	65	2.381
25	0.622	66	1.372
27	2.828	68	0.254
28	1.277	69	0.665
29	0.488	70	0.331
30, 31	10.623	71	3.690
35	4.110	73, 74	3.116
36	0.294	78	0.750
37	2.531	79	3.221
39	3.667	81	2.424
40	2.586	85	0.122
42	0.785	86	0.198
44	0.113	87	0.407
45	12.480	88	0.299
48	1.475	90	0.552
49	1.680	95	0.906
53, 54	6.612		

TABLE II

IDENTIFICATION OF COMPOUNDS IN SHALLOT OIL

Peak Nos. refer to Fig. 1.

Peak No.	*Compound*
22	Dimethyl trisulphide
37	1-Methylthiopropyl ethyl disulphide
45	Methyl propyl trisulphide
53	Dipropyl trisulphide
59	Propyl propenyl trisulphide
71	Dipropyl trisulphide

quality evaluation. Liquid chromatography is the method of choice in the evaluation of essential oils, as they may be sensitive to temperature.

Table II shows the fractions from HPLC identified directly by GC–MS analysis. The other minor fractions cannot be examined owing to the low solute concentration. During the past year considerable progress has been made with combined liquid chromatography–mass spectrometry[4,5]. However, the ability to combine reversed-phase liquid chromatography with on-line mass spectrometry, without seriously compromising either technique, is still a difficult problem. In this paper, a reversed-phase LC–GC–MS technique is proposed.

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