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GAS–LIQUID CHROMATOGRAPHIC ANALYSIS OF THE NEUTRAL FRACTION OBTAINED FROM ELDER

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SUMMARY

Gas–liquid chromatography has been used to investigate the neutral fraction obtained from the fruits of elder. The fraction contained vanillin and numerous unidentified compounds. Identities are suggested for some of these compounds.

INTRODUCTION

Elder (*Sambucus nigra*) is a medicinal plant. Fruits of the elder have laxative, diaphoretic, and slight analgesic activity and the flowers have uretic and vasotonic activity [1]. Previous phytochemical investigations have revealed the presence of saccharides [2], flavonoids [3], phenolic acids [4], terpenoids [5], anthocyanins [6], and cyanogenic glycosides [7]. The aliphatic acid composition of elder flowers has been thoroughly studied [8] and acids in the fruits of elder [9,10] have also been investigated. Amino acids have been reported to occur in the fruits [11]. Few amines have been found in elder [12], among these is choline [13].

In a previous paper we reported the gas–liquid chromatographic analysis of acid fractions of elder [14]. In this paper, we describe analogous analysis of the neutral fraction of elder.

EXPERIMENTAL

Analysis was performed on the fruits of elder. Samples were collected in villages close to Siedlce in October, cleaned, and dried at room temperature.

Isolation

The fruits (300 g) were ground and extracted twice with a mixture of acetone and water (1:1, 800 mL). The combined extracts were then filtered. The filtrate was concentrated under reduced pressure, the aqueous solution (900 mL) was made alkaline (pH 9.0) by use of NaOH solution (2 M) and extracted with ethyl acetate (3 × 200 mL). Organic extracts were dried with anhydrous sodium sulphate and evaporated. The residue (440 mg) was dissolved in a mixture of pyridine (1 mL) and acetic anhydride (1 mL) and next day a few drops of water, then dichloromethane (10 mL), and 10% hydrochloric acid were added. After shaking, the layers were separated and the organic layer was again washed with acid and excess acid was neutralised with $NaHCO_3$ solution. The organic extract was then concentrated and subjected to chromatography. Thin-layer chromatography was performed on silica gel 60 F_{254} plates with hexane–isopropanol, 9:1, as mobile phase. Numerous spots were obtained and were difficult to separate.

Gas–Liquid Chromatography

GLC was performed with an Shimadzu QP5050A instrument equipped with a mass-spectrometric detector and a 30 m × 0.25 mm i.d. × 0.25 μm SGE (Phenomenex) BPX 5 column. Helium was used as carrier gas at a flow rate of 0.7 mL min^{-1}.The injection volume was 1 μL (splitless) and the injector temperature was 180°C. The column was held at 80°C for 5 min after injection then programmed at 5° min^{-1} to 280°C which was held for 15 min. The detector was operated at 290°C. Compounds were identified on the basis of the NIST 107 library and their retention times (which were compared with those of standards).

RESULTS

Gas chromatographic analysis furnished sixteen peaks. Their retention times and important mass spectral data are given in Table I.

DISCUSSION

Compound 5, vanillin, was the only compound that could be identified unambiguously. The spectrum of compound 7 suggested it was 4-(2-propenyl)phenyl acetate and the spectrum of compound 13 was similar to that of the methyl ester of 9,10-dihydroxyoctadecanoic acid; standards of

Table I

Retention times and mass spectral data for compounds in the neutral fraction from Elder

Compound no.	Retention time (min)	Mass spectral peaks, *m*/*e*
1	8.37	131, 120, 105, 99, 84, 69
2	11.67	194, 152, 110
3	12.08	162, 149, 120, 107, 96, 76
4	12.19	182, 111, 96, 83, 69, 55
5	12.28	281, 194, 152
6	12.40	240, 207, 198, 181, 169, 141, 124, 99
7	12.82	240, 207, 198, 181, 169, 141, 124, 99
8	12.99	162, 120, 107, 91, 77
9	13.69	176, 134, 119, 107, 95, 77
10	14.17	234, 192, 150, 149, 133, 121, 107, 104
11	14.22	229, 207, 187, 178, 159, 149, 127, 98, 57
12	14.27	159, 145, 127, 116, 98, 84, 57
13	15.96	281, 227, 215, 185, 160, 155, 136, 117, 109, 98, 83
14	19.64	354, 341, 326, 311, 297, 284, 269, 255, 239, 159, 117
15	22.27	337, 307, 281, 262, 247, 221, 207, 193, 159, 135, 117
16	24.16	314, 272, 244, 229, 191, 173, 159, 147, 124

these compounds were not available, however. On the basis of the spectra obtained it seems probable that compounds 14 and 15 are derivatives (e.g. esters) of hexa- and octadecanoic acids. The conclusion can therefore be drawn that the fruits of elder contain some unknown compounds and are interesting material for further examination.

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