ON SOME LIPIDIC COMPONENTS FROM THE ROOT OF *Petasites hybridus* (L.) G. M. SCH.*

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In the chloroform extract from the root of *Petasites hybridus* (L.) G. M. SCH. there were detected by means of chromatographic and spectrophotometric methods in addition to compounds of the terpenoid nature the n-alkanes, esters of sterols (mainly of β-sitosterol) with higher aliphatic acids (mainly the $C_{16:0}$, $C_{18:2}$, and $C_{18:3}$ acids), and triglycerides containing the same acids.

In connection with investigations on the constituents[1-3] of *Petasites hybridus* (L.) G. M. SCH. (Syn.: *P. officinalis* MOENCH.) we have been interested in some so far poorly examined lipidic components. In the present paper, we wish to report on the analysis of the chloroform extract of roots.

EXPERIMENTAL

Material. Slices of the root were dried at room temperature, powdered (2·50 kg), the powder placed into a column, and washed at room temperature with chloroform (12 l). Evaporation of the chloroform extract yielded 62 g of the residue. A portion (20 g) was dissolved in chloroform (100 ml) and adsorbed to 50 g of deactivated silica gel. The solvent was removed and the residue chromatographed on a column (5·6 × 68 cm) of the Pitra[4] silica gel (particle size, 0·10—0·25 mm; 800 g) previously deactivated by the addition of water (12·5%). Light petroleum (b.p. 45—65°C) containing increasing amounts of ether (up to 20%) was used as eluant. The fractions were monitored by thin-layer chromatography on silica gel as well as by gas chromatography.

Methods. Gas chromatography was performed on a Perkin-Elmer F 11 apparatus with flame ionisation detectors and a dual system of glass columns (0·4 × 150 cm) packed with 3% SE-30 G.C. Grade on Gas-Chrom Q (80—100 mesh) or 20% diethylene glycol succinate on HMDS-Chromosorb W (80—100 mesh). Gas chromatography-mass spectrometry was performed on a combination of PYE Series 104 Chromatograph Model 64 and A.E.I. MS 902 apparatus with the use of the Watson–Biemann separator. Ionic source temperature, 150—200°C (according to the substance type); electron energy, 70 eV. Infrared spectroscopy was carried out in a UR-20 apparatus in 0·01 cm cells. Concentration of samples, 6% in tetrachloromethane.

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RESULTS

n-*Alkanes* (0·11% of the extract residue) form a homologous series (C_{15}—C_{37}) with dominating odd members. The C_{23} (7·9%), C_{25} (15·2%), C_{27} (30·9%), C_{29} (23·5%), and C_{31} (7·8%) homologues were the most abundant. It may be seen from the chromatographic record that the n-alkanes are as usually accompanied by a small amount of monomethylalkanes[5].

Terpenoids (4·0% of the extract residue). A systematic identification of sesquiterpenoid hydrocarbons has been performed earlier[6,7]. Nevertheless, some additional components of column chromatography fractions were completely or partly identified by means of gas chromatography–mass spectrometry. Thus, the presence of M = = 152 (base peak M − (26 + 15)) and M = 162 (main fragments 95, M − 15, M − 29) substances was recorded. There was also present a sesquiterpenic alcohol (M = 220; main fragments 41, 91, 81) absorbing in the IR region at 3620, 3440 and 1040 cm^{-1}. Moreover, the sesquiterpenic hydrocarbon β-santalene[8] (M = 204; main fragments 94, 41, 122) was newly identified. The subsequent fraction obtained from the chromatographic column (0·63% of the extract residue) mainly contains sesquiterpenic lactones[2,3,6] with IR absorption at 1770 and 1193 cm^{-1} (lactone group), at 3620 and 3440 cm^{-1} (—OH), at 1040 cm^{-1} (—O—), and at 1594 cm^{-1} (>C=C<). The most abundant components showed the following values: M = 218 (main fragments 103 ≫ 123, 218; 23·6%), M = 234 (main fragments M − 2, 123, 41, 109, 110; 49·3%), M = 234 (main fragments M − 2, 123, 109, 41, 110; 9·9%), and M = 232 (main fragments 109, 41, 91, 123, 232, 161; 9·0%).

Esters. The subsequent fraction (6·9% of the extract residue) exhibited IR absorption bands of the ester grouping (1732, 1249 and 1178 cm^{-1}) and of the double

TABLE I
Fatty Acids Obtained from Esters (A) and Glycerides (B)

Acid	A (%)	B (%)	Acid	A (%)	B (%)
12 : 0	—	[a]	17 : 1	0·3	0·2
14 : 0	0·5	0·6	18 : 0	1·0	1·3
15 : 0	0·4	1·8	18 : 1	2·0	3·7
?	0·8	—	18 : 2	64·3	54·8
16 : 0	12·9	16·1	18 : 3	17·2	18·5
16 : 1	0·6	3·0	20 : 0	[a]	[a]
17 : 0	—	[a]	22 : 0	[a]	—

[a] Trace amount.

bond (1642 cm^{-1}). After the transesterification[9], there was isolated a mixture of methyl esters of higher fatty acids (Table I) and a mixture of sterols. By means of mass spectrometry of trimethylsilylated hydroxy derivatives[10-12], the $C_{16:1}$ acid was identified as 9-hexadecenoic acid, the $C_{18:1}$ acid as 9-octadecenoic acid (oleic acid), and the $C_{18:2}$ acid as 9,12-octadecadienoic acid (linoleic acid). This method failed in determination of double bond positions in the $C_{18:3}$; the retention times in gas chromatography corresponded to those of authentic linolenic acid. The sterols were identified by gas chromatography and mass spectrometry[13] as campesterol (9·3%), stigmasterol (7·9%), and β-sitosterol[6] (72·8%); identification of the remaining three sterols (6·1, 2·9, and 1·0%) failed by this method.

Triglycerides. The subsequent chromatographic fraction (5·9% of the extract residue) exhibited the IR absorption at 1745 and 1162 cm^{-1} (ester group) and at 3015 cm^{-1} (>C=C<). On thin-layer chromatography (silica gel), the R_F value was identical with that of the synthetic triglyceride trimyristin. After the transesterification[9], there was isolated and identified by gas chromatography a mixture of higher fatty acids in the form of methyl esters (Table I); glycerol was identified in the form of the trimethylsilyl derivative[14]. As it may be inferred from Table I, composition of fatty acids in glycerides is very similar to that of acids bound to sterols.

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