Aufarbeitung. 12 g Moos werden mit Äther extrahiert, der Extrakt mit 10-proz. methanolischer KOH verseift und der Neutralanteil über 10 g Al_2O_3 (Aktivität II, neutral) chromatographiert.

Ergebnis. 500 ml Benzol eluieren 150 mg (1%) wachsartiges Material vom Schmp. 76–78°, im Schmelz- und Mischschmelzpunkt sowie IR-Spektrum identisch mit *Cerylalkohol.*

Moos. Tortella inclinata (Hedwig fil.) Limpr.

Herkunft. Aufgelassene Muschelkalkgrube bei Köllme, westlich von Halle, im September 1970 gesammelt.

Aufarbeitung. 584 g Moos werden mit Äther extrahiert, der Extrakt eingedampft und der Rückstand mit methanolischer KOH verseift.

Ergebnis. Chromatographie des Neutralanteils an Al_2O_3 (Aktivität II, neutral) gibt 180 mg (0,03%) *Cerylalkohol* vom Schmp. 75–76° und 100 mg (0,016%) *β-Sitosterin* vom Schmp. 137–139°.

Anerkennungen—Fräulein M. Nörr, Halle, danke ich für die Bestimmung der Moosproben und Herrn Dr. G. Köllner, Ruhla, für die Aufsammlung von *Plagiothecium undulatum.*

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VOLATILE COMPOUNDS FROM *FONTINALIS ANTIPYRETICA**

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Abstract—At least 27 steam volatile compounds have been isolated from *Fontinalis antipyretica*. Ten of these have been identified, the main compound being hexanal. Tetracosanoic acid has also been isolated from this moss.

IT IS well known that liverworts generally contain essential oils which give them their characteristic odour.[1–3] Several of these oils have recently been found to contain sesquiterpenes.[4–7] Since some mosses also exhibit a rather characteristic odour, we have investigated one of these, *Fontinalis antipyretica*, which is a big moss growing in water. It develops a rather strong odour during drying at room temp.

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GLC studies of the ethereal extract obtained from the moss as well as of the colourless oil obtained after steam distillation of the evaporated ethereal extract revealed the presence of a complex mixture. From the steam-distilled fraction only the main component could be isolated in an amount sufficient for spectral investigation. It was identified as hexanal and found to be mainly responsible for the characteristic odour of the moss. For further investigation the steam-distilled fraction was subjected to vacuum distillation and a fraction boiling at 48–51°/12–13 mm was collected. No alteration of the volatile components in this fraction could be detected, the peaks being the same as those traced in the chromatogram of the ethereal moss extract. A combined gas chromatograph-mass spectrometer equipped with a 15-m capillary column was used to resolve the low boiling fraction. No less than about 40 peaks were obtained, and 27 mass spectra were taken (Table 1). Besides hexanal, nine components were tentatively identified by their mass spectra and their identities confirmed by GLC co-chromatography with authentic specimens on two other columns. From the mass spectral data of the other components (Table 1) it is evident that nearly all are aliphatic compounds with rather low molecular weights. It cannot be excluded that some of the more volatile compounds originate from the ether used for the extraction procedures, although it was redistilled and no trace of these compounds could be found in the gas chromatogram of the evaporated residue.

During the isolation, a crystalline product was obtained and identified as tetracosanoic acid by IR, MS and mixed m.p.

TABLE 1. VOLATILE COMPOUNDS FROM *Fontinalis antipyretica*

Peak No.	Identity or mass spectral data
1	Ethanal
2	Ethyl formate
3	Ethyl acetate
4	Ethanol
5	Unknown: parent peak *m/e* 88
6	Unknown: parent peak *m/e* 88
7	Hexanal
8	Unknown: major peaks at *m/e* 43, 29, 44, 41, 42
9	Unknown: 43, 29, 27, 88, 57
10	Unknown: parent peak *m/e* 144
11	2-Heptanone
12	Unknown: 56, 43, 55, 41, 42
13	Ethyl hexanoate
14	1-Hexyl acetate
15	2-Octanone
16	Unknown: 56, 43, 55, 41, 42
17	Ethyl heptanoate
18	Unknown: 41, 43, 57, 28, 54
19	Unknown: 43, 28, 41, 55, 54
20	Unknown: 57, 43, 41, 29, 56
21	Unknown: 96, 95, 39, 28, 29
22	Unknown: 43, 55, 70, 41, 99
23	Unknown: 43, 117, 56, 99, 84
24	Unknown: 105, 28, 77, 43, 122
25	Unknown: 99, 28, 43, 41, 55
26	Unknown: 43, 28, 41, 55, 56
27	Unknown: 60, 73, 41, 43, 27

EXPERIMENTAL

Plant material. Fontinalis antipyretica was collected in the parish of Sälen in northwestern Dalecarlia where it grew in a mountain stream unpolluted by human habitation or industrial refuse. It was stored at − 20° until used. Reference compounds for GLC and mass spectrometry were obtained from commercial sources.

Isolation of volatile compounds. The moss was washed with distilled water, air dried, picked and ground. The powder (400 g) was extracted with redistilled ether (3 × 24 hr). After drying with $MgSO_4$ the ethereal solution was evaporated. The residue (25 g) was subjected to steam distillation using an all-glass system. The distillate (20 l.) was extracted with ether which after drying was evaporated yielding a colourless oil (1 g). Since by preparative GLC only the main compound could be separated, the oil was further purified by distillation. The fraction (0·3–0·4 g) boiling at 48–51°/12–13 mm was collected and used for the GLC-analyses.

Trials were also made to purify the volatile components by chromatography on alumina (activity II, neutral). A hexane extract obtained directly from the moss gave after elution with hexane some crystalline material. After recrystallization from pentane it melted at 78·5–79·5°. The m.p. was not depressed on admixture with an authentic specimen of tetracosanoic acid. Also their IR and MS spectra were identical.

Gas chromatography. The ethereal extract and the steam distillate were investigated in a Perkin–Elmer model F 21 preparative gas chromatograph (flame ionization detector) fitted with a 2·7 m × 0·95 cm stainless steel column packed with 20% Carbowax 20 M on Chromosorb A (45–60 mesh). Column temp.: Programmed 60–200° at 2°/min. Injection temp. 180–200°; carrier gas nitrogen 200 ml/min at 60°. Besides hexanal, identified by IR and co-chromatography on two other columns with an authentic specimen, only mixtures were obtained.

The fraction boiling at 48–51°/12–13 mm was investigated in a Perkin–Elmer model 270 combined gas chromatograph-mass spectrometer (flame ionization detector) fitted with a 15 m × 0·05 cm stainless steel open tubular column ('SCOT-column') coated with Carbowax 20 M. Column temp.: Programmed 50–150° at 10°/min. after 3·7 min and isothermal at 150°; injection temp. 180°; carrier gas helium 2–4 ml/min; sample size 0·5 μl. The mass spectra were recorded at 70 eV and compared with reference spectra and spectra given in the literature (Table 1).

The low boiling fraction was further analysed in a Pye model 64 gas chromatograph equipped with a flame ionization detector. Two different stainless steel 'SCOT-columns' 15 m × 0·05 cm were used. They were coated with:

(1) Carbowax 20M-TPA (Carbowax modified with terephthalic acid). Column temp.: 50° (3 min), programmed 50–180° at 10°/min, and isothermal at 180°.

(2) OV 1 (methylsilicone-gum). Column temp. 50° (3 min), programmed 50–180° at 5°/min, and isothermal at 180°. On-column injection, detector temp. 210°, carrier gas nitrogen 1·9 ml/min.

The identities of the different components were determined by co-chromatography in turn of the low boiling fraction with authentic specimens.

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