

a temp. of 20–21°. The filaments were harvested by filtration and washed twice with H₂O. The resulting material (wet wt. 8.74 g) was then extracted three times with CHCl₃-MeOH (2:1, v/v) after first disrupting the cells by cycles of freezing and thawing followed by grinding with acid-washed silver sand. The chlorosulpholipids were then obtained from this extract as described previously for *O. danica*⁵ and converted to their diols by solvolysis.⁶ The diol mixture was then silylated and subjected to GC-MS using a Pye 104 gas chromatograph linked via a single-stage silicone rubber membrane separator⁷ to an AE1 MS-30 mass spectrometer. The GLC separation was performed on a 5 ft × 4 mm i.d. glass column packed with 1% SE-30 on 80–100 mesh Gas Chrom Q programmed to 5 min isothermal at 210° followed by a linear increase of 5°/min up to 265° and then held. The He gas flow through the column was 50 ml/min. Low resolution MS were obtained with an electron energy of 24 eV, an emission current of 300 µA and a source temp. of 250°.

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CRYSTALLINE CHEMICAL COMPONENTS OF *CHEILANTHES LONGISSIMA*

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Key Word Index—*Cheilanthes longissima*; Polypodiaceae; fern; cheilarinosin; 7-*O*-methylapigenin; 3,7-di-*O*-methylkaempferol; 3,7,4'-tri-*O*-methylkaempferol; sitosterol; sitosterol D-glucoside.

Plant. *Cheilanthes longissima*. *Geographical source.* Darjeeling (Himalayas). *Previous work:* on this species nil; on other *Cheilanthes* species, *C. farinosa*,^{1–4} *C. tenuifolia*⁵ and *C. mysurensis*.⁶

Present work. Dried and coarsely powdered fern (aerial parts) was successively extracted with boiling petrol and C₆H₆. The extracts, being identical (TLC), were mixed. On concentration a solid was deposited. The filtered solid (1) and the evaporated mother liquor (2) were separately chromatographed on silica gel. From chromatography of (1): CHCl₃-MeOH (49:1) eluted cheilarinosin; CHCl₃-MeOH (97:3) eluted 3,7-di-*O*-methylkaempferol and then a flavonoid mixture; CHCl₃-MeOH (19:1) eluted sitosterol D-glucoside. The flavonoid mixture was separated by preparative TLC using C₆H₆:dioxan:AcOH (90:25:4) into 3,7-di-*O*-methylkaempferol and 7-*O*-methylapigenin. From chromatography of (2): Petrol-C₆H₆ (9:1) eluted a triterpene compound in very low yields; C₆H₆ eluted sitosterol; C₆H₆-EtOAc (49:1) eluted 3,7,4'-tri-*O*-methylkaempferol. Appropriate

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derivatives were prepared in each case. The properties of the parent substances and the derivatives which led to their identification are described below: (a) *Cheilarinosin*, m.p. 175°, $[\alpha]_D + 13.7^\circ$ (MeOH) (l^3 m.p. 177°, $[\alpha]_D + 11.7^\circ$). (b) *7-O-methylapigenin*, m.p. 295–296° (l^7 m.p. 285°), $C_{16}H_{12}O_5$, identification aided by reduction tests, ferric colour and by UV-visible spectrum in methanol before and after addition of shift reagents for flavonoids; diacetate m.p. 195–198° (l^7 m.p. (197–198°; dimethyl ether (trimethyl ether of apigenin) m.p. 157° (l^7 m.p. 156°). (c) *3,7-Di-O-methylkaempferol*, m.p. 261–263° (l^2 m.p. 252°), $C_{17}H_{14}O_6$, identification aided by colour reactions as above and UV-visible spectrum; dimethyl ether (tetra-*O*-methylkaempferol) m.p. 157° (l^2 m.p. 159°); diacetate m.p. 161–163°, because of disparity in m.p. from l^2 value (148°) its NMR spectrum was taken to confirm the presence of the functional groups and other protons: δ 2.23 (s, 3H, –Ac), 2.31 (s, 3H, –Ac), 3.78 (s, 6H, 2 × OMe), 6.64 (d, 2H, *J*, 3Hz, 6–H and 8–H), 6.99 (1H) and 7.15 (1H) (3'–H and 5'–H), 7.83 (1H) and 7.98 (1H) (2'–H and 6'–H). (d) *3,7,4'-Tri-O-methylkaempferol*, m.p. 142–144° (l^2 m.p. 143–145°), $C_{18}H_{16}O_6$, identification aided by colour reactions and UV-visible spectrum; monoacetate m.p. 156–158° (l^8 m.p. 158–160°). (e) *Sitosterol*, m.p. 142–144°, $[\alpha]_D - 30.5^\circ$ (CHCl₃), $C_{29}H_{50}O$, positive Liebermann–Burchard test; acetate m.p. 135–136°, $[\alpha]_D - 38.5^\circ$ (CHCl₃). (f) *Sitosterol D-glucoside*, m.p. 296° (decomp.), $[\alpha]_D - 35.0^\circ$ (pyridine), $C_{35}H_{60}O_6$, positive Liebermann–Burchard and Molisch tests; hydrolysis yielded sitosterol and D-glucose; hydrolysis after initial permethylation yielded sitosterol and 2,3,4,6-tetra-*O*-methylglucose identified by paper chromatography.

The identity of all compounds (except b) and their crystalline derivatives was further confirmed by direct comparison with authentic samples available in our collection, employing m.m.p., TLC and IR spectra.

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⁸ IYER, R. T. (1971) Ph.D. thesis, Delhi University, p. 60.