

The Biflavonoid Pattern of *Anacolia webbii**

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Biflavonoids

From *Anacolia webbii* the following five biflavonoids could be isolated: 2,3-dihydrophilonotisflavone, philonotisflavone, dicranolomin, 5',3'''-dihydroxyamentoflavone and 5',3'''-dihydroxyrobustafavone. The compounds were identified spectroscopically.

During a study of the biflavonoid patterns of the Bartramiaceae by two-dimensional TLC [1], we found that our standard chromatograms of *Anacolia webbii* are dominated by a spot with the chromatographic behaviour of 2,3-dihydrophilonotisflavone. Since this very large spot might well have obscured other compounds we decided to analyse this species on a preparative scale. By column chromatography on polyamide and Sephadex LH 20 we obtained from dried plant material ≈ 2400 ppm 2,3-dihydrophilonotisflavone (**1**), ≈ 200 ppm philonotisflavone (**2**), ≈ 230 ppm dicranolomin (**3**), ≈ 54 ppm 5',3'''-dihydroxyamentoflavone (**4**) and ≈ 13 ppm 5',3'''-dihydroxyrobustafavone (**5**). These compounds were identified by their spectroscopic data (^1H NMR, FAB-MS) [2–4] and by TLC with authentic material.

It is noteworthy that in this moss philonotisflavone is accompanied by a large amount of 2,3-dihydrophilonotisflavone, whereas 2,3-dihydrodicranolomin is absent, although dicranolomin itself occurs in the same concentration range as philonotisflavone. Bartramiaflavone and anhydrobartramiaflavone, which are characteristic of related *Bartramia* species [1, 5, 6] are absent from *Anacolia webbii*.

Experimental

Plant material

Gametophytic material of *Anacolia webbii* (Mont.) Schimp. was collected in Madeira, Pico de Arieiro, 14.10.1990, leg. et det. R. Mues. Voucher specimens are deposited in the Herbaria of R. Mues (Nr. 2573) and of Fachrichtung Botanik, Universität des Saarlandes ("SAAR", Nr. 3857).

Extraction and isolation

370 g air-dried plant material (freed from foreign matter) was preextracted five times with CHCl_3 2.5 l each. The flavonoids were extracted five times with $\text{EtOH}:\text{H}_2\text{O}$ (8:2) 2.5 l each and once with 2 l $\text{Me}_2\text{CO}:\text{H}_2\text{O}$ (8:2). To eliminate chlorophyll the combined flavonoid extracts were evaporated and the residue subjected to a four step Craig distribution between the upper and lower phases of $\text{DMF}:\text{H}_2\text{O}:\text{Et}_2\text{O}$ (4:1:8). The combined lower phases were reduced *in vacuo* to a thin syrup (about 100 ml). After addition of 60 ml dry polyamide-6 powder it was diluted with 1 l water. The resulting suspension was cautiously poured on top of a 3 l polyamide-6-column (wet packed). The column was eluted with 8 l H_2O ; 4 l each of $\text{Me}_2\text{CO}:\text{H}_2\text{O}$ (1:9; 2:8; 3:7); 10 l (4:6); 14 l (5:5); 7 l (6:4); 6 l each of (7:3; 8:2) and 8 l (9:1). The compounds were eluted as follows: **1**, **2**, **2+3**, **4**, **4+5**. Further separation and purification were achieved by CC on Sephadex LH20 with $\text{Me}_2\text{CO}:\text{H}_2\text{O}:\text{MeOH}$ (2:1:1).

Yields: 900 mg of **1**; 75 mg of **2**; 85 mg of **3**; 20 mg of **4**; 5 mg of **5**.

^1H NMR spectroscopy: Bruker AM 400, 400 MHz, $\text{DMSO}-d_6$, ambient temperature.

Mass spectra were recorded by FAB-MS (negative mode) on a Finnigan MAT 90 in a glycerine methanol-matrix with 4–8 keV xenon atoms.

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