

## FLAVONOID GLYCOSIDES FROM *Ammoides pusilla*

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The present study is a part of our search concerning flavonoids in *Apiaceae* and their relationship to therapeutic activity.

The genus *Ammoides* (*Apiaceae*) includes two species in Algeria. one of them is endemic: *Ammoides atlantica* (Coss. et Dur.) Wolf; the other one *Ammoides pusilla* (Brot.) Breistr. is widespread in the Mediterranean region [1].

*Ammoides pusilla* is used in folk medicine as an infusion to treat headache, fever, flu, and diarrhea [2–4].

Aerial parts of *Ammoides pusilla* were collected during the flowering phase in eastern Algeria (May 2002) and authenticated by Dr. Hocine Laouer from the Department of Biology (University of Setif, Algeria) on the basis of Quezel and Santa [1].

Dried powder (435 g) was extracted with 70% MeOH. The MeOH extract was concentrated to dryness, the residue was dissolved in boiling water, and the concentrate was taken up with AcOEt and *n*-BuOH after filtration. The concentrate was evaporated and the residue was dissolved in small volumes of MeOH.

Two-dimensional paper chromatography using 15% AcOH and BAW(*n*-BuOH–AcOH–H<sub>2</sub>O, 4:1:5 upper phase) as solvents shows that the AcOEt and *n*-BuOH extract contains almost the same compounds representing flavonoids and phenolic acids.

The *n*-BuOH extract was applied to a column of polyamide MN SC6 and eluted with a gradient of toluene – MeOH with increasing polarity. Three flavonoid glycosides (**1–3**) contained in several fractions were isolated by Whatman 3MM paper using 15% AcOH as solvent and by TLC chromatography on polyamide DC6 with 13:3:3:1 (water–MeOH–methylketone–acetylacetone). Purification of each compound for spectral analysis was carried out using MeOH over sephadex LH-20.

The structures of these compounds were elucidated by UV and <sup>1</sup>H NMR, and all these data were in good agreement with the respective literature data [5–6].

**Quercetin 3-O-β-glucoside (1)**, mp 218–212°C, UV ( $\lambda_{\max}$ , MeOH, nm): 257, 353; + NaOH: 272, 331, 404; + AlCl<sub>3</sub>: 273, 300 sh, 434; AlCl<sub>3</sub>/HCl: 269, 359 sh; 396.

<sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>,  $\delta$ , ppm, J/Hz): 7.60 (1H, d, J = 2.3, H-2'); 7.60 (1H, m, H-6'); 6.85 (1H, d, J = 8.9, H-5'); 6.40 (1H, d, J = 1.9, H-8); 6.20 (1H, d, J = 1.9, H-6); 5.40 (1H, d, J = 6.9, H-1''glu); 3.2–4.9 (sugar protons).

Acid hydrolysis of **1** produced quercetin and D-glucose.

**Luteolin 7-O-α-L-rhamnosyl-(1-6)-β-D-glucopyranoside (2)**, mp 250°C (decomp), UV ( $\lambda_{\max}$ , MeOH, nm): 265, 342; + NaOH: 269, 394; + AlCl<sub>3</sub>: 272, 346 sh, 403; + AlCl<sub>3</sub>/HCl: 274, 346 sh, 381. <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>,  $\delta$ , ppm, J/Hz): 7.79 (1H, d, J = 2.1, H-2'); 4.79 (1H, m, H-6'); 6.95 (1H, d, J = 8.3, H-5'); 6.72 (1H, s, H-3); 6.72 (1H, d, J = 2.0, H-8); 6.47 (1H, d, J = 2.0, H-6); 5.10 (1H, d, J = 7.4, H-1''glu); 4.60 (1H, d, H-1'''rha); 1.05 (3H, d, CH<sub>3</sub> rha); 3.2–4.0 (sugar protons).

**Apigenin 7-O-β-glucoside (3)**, mp 220–222°C, UV ( $\lambda_{\max}$ , MeOH, nm): 269, 334; + NaOH: 267, 381; + AlCl<sub>3</sub>: 277, 349 sh, 383; + AlCl<sub>3</sub>/HCl: 279, 346 sh, 382.

<sup>1</sup>H NMR (250 MHz, CD<sub>3</sub>OD,  $\delta$ , ppm, J/Hz): 7.91 (2H, d, J = 8.8, H-2', H-6'); 6.95 (2H, d, J = 8.8, H-3', H-5'); 6.83 (1H, d, J = 2.5, H-8); 6.51 (1H, s, H-3), 6.54 (1H, d, J = 2.55, H-6); 5.20 (1H, d, H-1''glu); 3.2–4.7 (sugar protons).

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Acid hydrolysis of **2** produced apigenin and D-glucose.  
Compounds **1–3** have been isolated from genus *Ammoides* for the first time.

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