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FOUR FLAVONOIDS FROM THE AERIAL PART OF *Ononis angustissima* SPECIES

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Ononis angustissima Lam. is an endemic plant of the north of Algerian Sahara (Guardaia, Bechar, Biskra, Boussaada and Beni Abbas). This plant was collected in the region of Bechar on April 1998 and was authenticated by Pr. A. Kaabeche (Biology department, University of Setif, Algeria). It has a yellow flower and belongs to the Leguminoseae family [1], which is very rich in flavonoids [2].

The Mediterranean species of this genus have been studied, in which more than 20 aglycon flavonoids were identified; as natural compounds, some of them are rare [3]. The aerial parts of *Ononis angustissima* Lam. (500 g) were air dried and extracted with MeOH–H_2O (70:30, v/v) three times during 24 hours. The solution was concentrated and extracted with solvents, starting with $CHCl_3$, AcOEt and *n*-BuOH.

A 7 g portion of chloroform extract was chromatographed on a silica gel (230–400 mesh) column using hexane–AcOEt as eluant, yielding 17 fractions. The isolated compounds were purified on silica gel TLC plates using as eluants diethyl ether–toluene–$CHCl_3$ (1:3:3, v/v/v), toluene–AcOEt–formic acid (95:5:1), $CHCl_3$–AcOEt (1:1, v/v), toluene–AcOEt–formic acid (95:2.5:0.5, v/v/v), hexane–AcOEt, toluene–$CHCl_3$, toluene–AcOEt, and hexane–acetone.

Compound 1: $C_{18}H_{16}O_6$, mp 100–102°C, UV spectrum (MeOH, λ_{max}, nm): 281, 339; +NaOH: 281, 342; +$AlCl_3$: 287, 343; +$AlCl_3$/HCl: 299, 357; +NaOAc: 283, 344; + NaOAc/H_3BO_3: 283, 344. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.92 (3H, s, OCH_3-6), 3.99 (3H, s, OCH_3-7), 4.09 (3H, s, OCH_3-8), 6.65 (1H, s, H-3), 7.48 (3H, m, H-3′, H-4′, H-5′), 7.88 (2H, m, H-2′, H-6′), 12.56 (1H, s, OH-5). ^{13}C NMR (100 MHz, $CDCl_3$): 61.0 (OCH_3-6), 61.6 (OCH_3-8), 62.1 (OCH_3-7), 105.0 (C-3), 106.9 (C-10), 126.0 (C-2′, C-6′), 128.9 (C-3′, C-5′), 131.0 (C-4′), 131.2 (C-7), 131.8 (C-1′), 136.4 (C-6), 145.8 (C-9), 149.4 (C-5), 153.1(C-8), 163.8 (C-2), 182.8 (C-4). Mass spectrum (EI, 70 eV), *m/z* (I_{rel}, %): 328.11 $[M]^+$ (69.52), 313.10 $[M–15]^+$ (100), 298.08 $[M–OCH_3]^+$ (9.27), 270.06 $[M–59]^+$ (5.20), 105.02 (3.89), 103.04 103.04 (2.33). Identified as 5-hydroxy-6,7,8-trimethoxyflavone (alnetin) [4].

Compound 2: $C_{17}H_{14}O_5$, mp 156–158°C, UV spectrum (MeOH, λ_{max}, nm): 275, 346; +NaOH: 275, 351; +$AlCl_3$: 293, 328; +$AlCl_3$/HCl: 289, 328; +NaOAc: 275, 346; + NaOAc/H_3BO_3: 275, 346. ^{1}H NMR ($CDCl_3$, δ, ppm): 3.89 (3H, s, OCH_3-7), 3.90 (3H, s, OCH_3-6), 6.39 (1H, s, H-3), 6.63 (1H, s, H-8), 7.50 (3H, m, H-3′, H-4′, H-5′), 7.89 (2H, m, H-2′, H-6′), 12.40 (1H, s, OH-5). ^{13}C NMR ($CDCl_3$): 56.1 (OCH_3-7), 61.5 (OCH_3-6), 95.8 (C-8), 105.3 (C-3), 126.3 (C-2′, C-6′), 128.9 (C-3′, C-5′), 129.1 (C-10), 131.3 (C-4′), 131.9 (C-1′), 147.5 (C-9), 149.4 (C-7), 157.5 (C-5), 158.7 (C-6), 163.9 (C-2), 182.7 (C-4). Mass spectrum (EI, 70 eV), *m/z* (I_{rel}, %): 298.07 $[M]^+$ (45.38), 283.05 $[M–15]^+$ (100), 255.06 $[M–15–CO]^+$ (2), 181.00 $[M–15–C_8H_6]^+$ (9.88), 153.01 (16.32), 149.06 (5.66), 105.03 (1.85), 102.04 (4.43). Identified as 5-hydroxy-6,7-dimethoxyflavone (6,7-di-*O*-methylbaicalein) [5–8].

Compound 3: $C_{16}H_{12}O_5$, mp 235–239°C, UV spectrum (MeOH, λ_{max}, nm): 275, 335; +NaOH: 286, 359; +$AlCl_3$: 286, 359; +$AlCl_3$/HCl: 291, 331; +NaOAc: 282, 345; + NaOAc/H_3BO_3: 281, 345. ^{1}H NMR ($CDCl_3$, δ, ppm): 4.10 (3H, s, OCH_3-7), 6.48 (1H, s, H-8), 6.70 (1H, s, H-3), 7.70 (3H, m, H-3′, H-4′, H-5′), 8.10 (2H, m, H-2′, H-6′), 12.50 (1H, s, OH-5). Identified as 5,6-dihydroxy-7-methoxyflavone (negletein) [7–10].

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Compound 4: $C_{15}H_{10}O_5$, mp 284–286°C, ^{1}H NMR ($CDCl_3$, δ, ppm, J/Hz): 6.21 (1H, d, J = 2.0, H-6), 6.58 (1H, d, J = 2.0, H-8), 6.78 (1H, s, H-3), 7.80 (3H, m, H-3′, H-4′, H-5′), 8.04 (2H, m, H-2′, H-6′), 12.50 (1H, s, OH-5). Identified as 5,7-dihydroxyflavone (chrysin) [11].

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