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FLAVONOID AGLYCONES AND STEROL FROM *Chrysanthemum fontanesii*

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Chrysanthemum species have been reported to exhibit antibacterial and antiviral activities [1–4] and are known to be a rich source of secondary metabolites with a variety of biological activities [5–9].

The genus *Chrysanthemum* (Compositae) is represented by about 20 species in Algeria [10]. As part of our ongoing program of research on plants of this genus [11], we report our results on *C. fontanesii* B. et R., an endemic species in the Maghreb [10], which has not been previously investigated.

Aerial parts of *Chrysanthemum fontanesii* were collected during flowering near Bejaia, North East Algeria (May 2003) and authenticated by Prof. M. Kaabeche (Biology Department, University of Setif, Algeria). A voucher specimen (CCF05/04/03) has been deposited in the Herbarium of Nature and Life Sciences Department, Mentouri University of Constantine.

Air-dried flowers of *C. fontanesii* (1516 g) were extracted at room temperature with MeOH–H_2O (80:20 v/v) for 24 h three times. After filtration, the filtrates were combined, concentrated, and successively extracted with $CHCl_3$, EtOAc, and *n*-BuOH. The organic layers were dried with Na_2SO_4 to give, after removal of solvents under reduced pressure, $CHCl_3$ (7.20 g), EtOAc (30.50 g), and *n*-BuOH (59.10 g) extracts.

The chloroform extract (7.20 g) was submitted to silica gel (230–400 mesh) column chromatography using *n*-hexane–EtOAc with increasing polarity as eluent to yield 25 fractions (F_1–F_{25}) obtained by combining the eluates on the basis of TLC analysis. Fraction F_{10} (90:10) was rechromatographed on a silica gel column using *n*-hexane–EtOAc (90:10) to give **1** (9 mg). Fractions F_{15}, F_{16} (60:40) and fraction F_{21} (50:50) were submitted to preparative TLC on silica gel GF_{254} eluted with $CHCl_3$–CH_3COCH_3 (12:1), $CHCl_3$–MeOH (16:1), and $CHCl_3$–MeOH (14:1), respectively, to give **2** (90 mg), **3** (100 mg), and **4** (42 mg), respectively.

The structures of the isolated compounds were elucidated by UV, ^{1}H NMR, ^{13}C NMR, and MS analysis. All these results were in good agreement with the literature data [12].

Compound 1. White crystals, mp 136–138°C. The Liebermann–Burchard test indicated its steroidal nature. The EI-MS spectrum presented a molecular ion at *m/z* 414 according to the molecular formula $C_{29}H_{50}O$. The ^{13}C and DEPT spectra recorded in $CDCl_3$ showed 29 signals from which those relative to a quaternary carbon atom at δ 140.7 and two CH groups at δ 121.7 and 71.8 were characteristic of C-5, C-6, and C-3, respectively, of the *β*-sitosterol [13]. The structure was also confirmed by results of co-chromatography with an authentic sample on silica gel plates eluted with petroleum ether–EtOAc (8:2), (R_f 0.58).

Compound 2. $C_{16}H_{12}O_6$, yellow needles, mp 299–302°C. The UV spectrum, recorded in MeOH, showed characteristic bands of a flavonoid. The value of λ_{max} at 352 nm of the band I and the deep purple fluorescence under UV radiation at 365 nm were indicative of 3-OR flavonol. This compound was characterized as 4′,5,7-trihydroxy-3-methoxyflavone (isokaempferide) [14].

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Compound 3. $C_{17}H_{14}O_7$, yellow needles, mp 257–260°C. The spectral data led to the structure of 4′,5,7-trihydroxy-3,3′-dimethoxyflavone (3-*O*-methylisorhamnetin) [15].

Compound 4. $C_{16}H_{12}O_7$, yellow crystals, mp 273–275°C. This compound was characterized as 3′,4′,5, 7-tetrahydroxy-3-methoxyflavone (3-*O*-methylquercetin) [16].

All these compounds are isolated from *C. fontanesii* B. et R. for the first time.

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