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B R I E F C O M M U N I C A T I O N S

CONSTITUENTS OF *Maydis stigma* CHLOROFORM EXTRACT

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We have investigated the chloroform extract of a commercially available herbal drug *Maydis stigma* (dried cut stigmata of maize, *Zea mays* L. ssp. *mays*, *Poaceae*), purchased from the herbal apothecary of the Institute for Medicinal Plant Research "Dr. Josif Pancic", Belgrade, Serbia and Montenegro. Powdered plant material (1.5 kg) was extracted with $CHCl_3$ in a Soxhlet apparatus until exhausted. Dry $CHCl_3$ extract (15 g) was fractionated by silicagel flash CC, using an *n*-hexane to *n*-hexane-acetone (50:50, v/v) gradient with 1% rate, followed by an *n*-hexane–acetone (50:50, v/v) to MeOH gradient with 5% increase. Final elution: MeOH. Fractions with similar composition (monitored by TLC) were pooled together. Compound **1** (200 mg) was isolated from fraction 12 by recrystallization from petroleum ether at +4°C. Compound **2** (1600 mg) was isolated from pooled fractions 24–27 by recrystallization from acetone at +4°C. Compound **3** (50 mg) was isolated from pooled fractions 28–56 by LPLC on Lobar Fertigsäule Grosse B (310–25) LiChroprep Si 60 (40–63 mm) für die Flussigkeits-Chromatographie (Merck, Darmstadt, Germany) and ChemCo Low-Prep Pump, Model 81-M-2 (ChemCo, Japan), using toluene–ethyl acetate gradient, at a flow rate of 1 ml/min. Compound **4** (500 mg) was isolated from pooled fractions 170–194 by recrystallization from acetone at +4°C.

Compound 1 (white, amorphous flakes). Mass spectrum (EI, 70 eV), *m/z* (I_{rel}, %): 396 $[M]^+$ (6), 382 $[M]^+$ - 14 (8), 368 (28), 354 (9), 340 (11), 326 (6), 312 (4), 298 (3), 60 (45, basal ion), 45 (100). **1** was identified as hexacosanoic acid. This compound was isolated from *M. stigma* for the first time.

Compound 2 (colorless prismatic crystals). Mass spectrum (EI, 70 eV), *m/z* (I_{rel}, %): 414 $[M]^+$ (67), 399 $[M]^+$-Me (27), 396 $[M]^+$-H_2O (34), 381 $[M]^+$–Me–H_2O (24), 329 (27), 303 (35), 273 (27), 255 (46), 231 (24), 213 (40), 145 (47), and *m/z* (I_{rel}, %): 412 $[M]^+$ (39), 300 (21), 271 (27), 253 (8), 229 (18), 211 (5). PMR (250 MHz, $CDCl_3$, δ, ppm, J, Hz): 0.6–1.0 (s, CH_3-18, 19, 21, 26, 27 and 29, J = 6–7), 3.53 (m, H-3a), 5.33 (d, H-6), 5.12 (dd, H-22), 5.00 (dd, H-23). According to previously published spectral data [1–3], 2 was identified as a mixture of sitosterol and stigmasterol (4:1 ratio).

Compound 3 (colorless semisolid). PMR (250 MHz, $CDCl_3$, δ, ppm): 3.48 (1H, s, OH), 2.44 (2H, s, aliphatic CH_2), 1.99 (3H, s, CH_3), 1.05 (6H, s, $2CH_3$). ^{13}C NMR (90 MHz, $CDCl_3$, d): 210.5 (C=O), 69.2 (*tert*-C–OH), 53.7 (aliphatic CH_2), 31.4 (CH_3 at C=O), 28.9 ($2CH_3$). The presented spectral data are in good agreement with published data on 4-hydroxy-4-methyl-2-pentanone [4]. To the best of our knowledge, **3** was isolated from *M. stigma* for the first time.

Compound 4 (white, amorphous solid). The PMR spectra (250 MHz, $CDCl_3$, δ, ppm) are similar to **2**, except: 4.91 (1H, d, anomeric H-1′ of glucose), 4.20 (1H, d, H-3′), 4.43 (1 H, t, H-4′), 4.86–4.87 (1H, dd, H-6′). The presented data are consistent with previously published data on daucosterol (β-sitosteryl-3-O-β-D-glucoside, sitosteroline) [3].

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