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CHEMICAL CONSTITUENTS OF Inula montana FLOWERS AND LEAVES

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Aix Marseille Univ, Spectropole, FR1739, Service 511, Campus Saint-Jerome, 13397 Marseille, France; 3) Luberon Natural Regional Park, 60, Place Jean Jaures, 84400 Apt, France; 4) Avignon University, Qualisud, UMR 95, F-84000, Avignon, France. Translated from *Khimiya Prirodnykh Soedinenii*, No. 4, July–August, 2018, pp. 639–640. Original article submitted January 30, 2018.

Inula montana L. from the flora of France was studied in continuation of a search for new potential sources of biologically active compounds.

I. montana (Asteraceae, Compositae) is widely used in folk medicine of Provence to treat hematomas as a replacement for *Arnica montana* [1].

Herein, the new myo-inositol derivative myo-inositol-1,5-diangelate-3,6-diacetate is characterized. Previously, four new myo-inositol derivatives were isolated from this species [2].

PMR and COSY spectra showed resonances of neighboring H-1–H-2–H-3–H-4–H-5–H-6, suggesting an inositol-type ring, two acetates, and two angelates.

The HMBC spectrum exhibited correlations between H-1–C-1' and H-5–C-1''' for the angelates; between H-3–C-1'' and H-6–C-1'''' for acetates; and between methyl H atoms and acetate C atoms H-2''–C-1'' and H-2'''–C-1'''. All these data were consistent with 1- and 5-angelates and 3- and 6-acetates on the inositol ring.

NOESY spectra defined the spatial orientation and enabled the myo-inositol structure to be characterized.

A CombiFlash Rf 200 flash chromatograph (Teledyne ISCO, USA) and PLC 2020 preparative HPLC (Gilson, USA) equipped with a Luna C18 column ($150 \times 21.2 \text{ mm}$, 3 µm) was used for chromatographic separations. NMR spectra were recorded on a Bruker AM-600 spectrometer; UV spectra, on a Shimadzu UV-1650pc spectrophotometer; high-resolution mass spectra, on a SYNAPT G2 High Definition Mass Spectrometer (Waters, USA).

Raw material for the study was collected in June 2015 in the vicinity of Merindol in Luberon Natural Regional Park, France.

Air-dried and ground flowers (0.2 kg) and leaves (0.2 kg) of *I. montana* were extracted (3×) with EtOH (95%) by percolation in a 1:3 ratio. The extracts were combined and evaporated *in vacuo* to an aqueous residue that was worked up sequentially with petroleum ether and CH_2Cl_2 to afford petroleum-ether extract (2.1 and 4.4 g) and CH_2Cl_2 extract (1.0 and 0.7 g) of flowers and leaves, respectively.

Reversed-phase flash chromatography (C18, 100 g) of the CH_2Cl_2 extract of leaves (0.6 g) isolated 1 (0.3 mg) that was identified based on NMR spectroscopic data as the new compound myo-inositol-1,5-diangelate-3,6-diacetate.



Compound 1, $C_{20}H_{29}O_{10}$, yellow oil, soluble in CH_2Cl_2 , EtOH, MeOH. UV spectrum (MeOH, λ_{max} , nm): 217. ¹H NMR spectrum (600 MHz, MeOD-d₄, δ , ppm, J/Hz): 6.16 (1H, qq, J = 7.3, 1.6, H-3'''), 6.13 (1H, qq, J = 7.3, 1.6, H-3'), 5.62 (1H, br.t, J = 10.2, H-6), 5.19 (1H, br.t, J = 9.8, H-5), 5.05 (1H, dd, J = 10.5, 2.6, H-1), 4.85 (1H, dd, J = 10.2, 2.6, H-3), 4.31 (1H, t, J = 2.6, H-2), 4.12 (1H, br.t, J = 9.9, H-4), 2.12 (2H, s, H-2''), 1.95 (6H, dq, J = 7.3, 1.6, H-4', 4'''), 1.91 (3H, s, H-2''''), 1.87 (3H, pentet, J = 1.6, H-5'), 1.84 (3H, pentet, J = 1.6, H-5'''). ¹³C NMR spectrum (150 MHz, MeOD-d₄, δ , ppm): 72.4 (C-1), 68.2 (C-2), 74.5 (C-3), 69.6 (C-4), 74.0 (C-5), 71.4 (C-6), 167.9 (C-1'), 128.5 (C-2'), 140.2 (C-3''), 15.7 (C-4'''), 20.4 (C-5'''), 171.2 (C-1''''), 20.4 (C-2'''').

Compound **2** (6.2 mg) was isolated from the same CH_2Cl_2 extract and was identified using NMR spectroscopy as loliolide, $C_{11}H_{16}O_3$. UV spectrum: (MeOH, λ_{max} , nm): 215 [3].

The CH_2Cl_2 extract of flowers (0.9 g) was purified by reversed-phase flash chromatography (C18, 100 g) and then preparative HPLC to isolate **3** (11.6 mg) that was identified as *N*,*N*-di(2-hydroxyethyl)dodecanamide, $C_{16}H_{33}NO_3$. The structure was elucidated using NMR spectroscopy. The length of the lipid chain was determined using high-resolution mass spectroscopy. The exact monoisotopic mass is 287.2460 Da [4].

All three compounds were identified in I. montana for the first time.

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