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# CHEMICAL CONSTITUENTS OF *Inula montana* FLOWERS AND LEAVES

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2) 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.

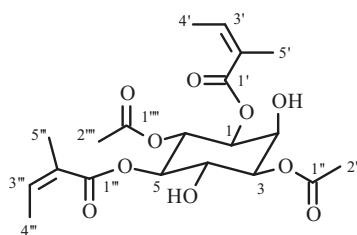
NOESY spectra defined the spatial orientation and enabled the myo-inositol structure to be characterized.

A CombiFlash Rf 200 flash chromatograph (Teledyne ISCO, USA) and PLC 2020 preparative HPLC (Gilson, USA) equipped with a Luna C18 column (150 × 21.2 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<sub>2</sub>Cl<sub>2</sub> to afford petroleum-ether extract (2.1 and 4.4 g) and CH<sub>2</sub>Cl<sub>2</sub> extract (1.0 and 0.7 g) of flowers and leaves, respectively.

Reversed-phase flash chromatography (C18, 100 g) of the CH<sub>2</sub>Cl<sub>2</sub> 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<sub>20</sub>H<sub>29</sub>O<sub>10</sub>, yellow oil, soluble in CH<sub>2</sub>Cl<sub>2</sub>, EtOH, MeOH. UV spectrum (MeOH, λ<sub>max</sub>, nm): 217. <sup>1</sup>H NMR spectrum (600 MHz, MeOD-d<sub>4</sub>, 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''). <sup>13</sup>C NMR spectrum (150 MHz, MeOD-d<sub>4</sub>, 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'), 171.7 (C-1''), 20.7 (C-2''), 168.2 (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<sub>2</sub>Cl<sub>2</sub> extract and was identified using NMR spectroscopy as loliolide, C<sub>11</sub>H<sub>16</sub>O<sub>3</sub>. UV spectrum: (MeOH, λ<sub>max</sub>, nm): 215 [3].

The CH<sub>2</sub>Cl<sub>2</sub> 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<sub>16</sub>H<sub>33</sub>NO<sub>3</sub>. 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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