Chemical Constituents of *Inula montana* Flowers and Leaves

E. Garayev, G. Herbette, Fathi Mabrouki, P. Chiffolleau, David Roux,
Evelyne Ollivier, Riad Elias, B. Baghdikian

► To cite this version:

E. Garayev, G. Herbette, Fathi Mabrouki, P. Chiffolleau, David Roux, et al.. Chemical Constituents of *Inula montana* Flowers and Leaves. *Chemistry of Natural Compounds*, 2018, 54 (4), pp.755-756. 10.1007/s10600-018-2463-3 . hal-01865221

HAL Id: hal-01865221

<https://hal.science/hal-01865221>

Submitted on 28 Mar 2019

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

CHEMICAL CONSTITUENTS OF *Inula montana* FLOWERS AND LEAVES

E. Garayev,^{1*} G. Herbette,² F. Mabrouki,¹ P. Chiffolleau,³ D. Roux,⁴ E. Ollivier,¹ R. Elias,¹ and B. Baghdikian¹

1) Aix Marseille Univ, Avignon Universite, CNRS, IRD, IMBE, Laboratoire de Pharmacognosie, Marseille, France, e-mail: elnurgar@gmail.com;

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 CH₂Cl₂ to afford petroleum-ether extract (2.1 and 4.4 g) and CH₂Cl₂ extract (1.0 and 0.7 g) of flowers and leaves, respectively.

Reversed-phase flash chromatography (C18, 100 g) of the CH₂Cl₂ 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, C₂₀H₂₉O₁₀, yellow oil, soluble in CH₂Cl₂, 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'), 171.7 (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 CH₂Cl₂ extract and was identified using NMR spectroscopy as loliolide, C₁₁H₁₆O₃. UV spectrum: (MeOH, λ_{max}, nm): 215 [3].

The CH₂Cl₂ 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, C₁₆H₃₃NO₃. 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.

REFERENCES

1. M. Amir, *Les Cueillettes de Confiance*, Forcalquier, 1998, 247 pp.
2. E. Garayev, G. Herbette, C. Di Giorgio, P. Chiffolleau, D. Roux, H. Sallanon, E. Ollivier, and B. Baghdikian, *Fitoterapia*, **120**, 79 (2017).
3. G. Erosa-Rejon, L. M. Pena-Rodriguez, and O. Sterner, *J. Mex. Chem. Soc.*, **53** (2), 44 (2009).
4. M. Fernandez-Perez and C. Otero, *Enzyme Microb. Technol.*, **33** (5), 650 (2003).