

Natural Product Research
Formerly Natural Product Letters

ISSN: 1478-6419 (Print) 1478-6427 (Online) Journal homepage: http://www.tandfonline.com/loi/gnpl20

Essential oil composition of Inula britannica L. from Bulgaria

Milka Todorova, Antoaneta Trendafilova, Viktoria Ivanova, Kalina Danova & Dimitar Dimitrov

To cite this article: Milka Todorova, Antoaneta Trendafilova, Viktoria Ivanova, Kalina Danova & Dimitar Dimitrov (2017): Essential oil composition of Inula britannica L. from Bulgaria, Natural Product Research, DOI: 10.1080/14786419.2017.1285295

To link to this article: http://dx.doi.org/10.1080/14786419.2017.1285295

View supplementary material

Published online: 13 Feb 2017.

Submit your article to this journal

View related articles

View Crossmark data

Full Terms & Conditions of access and use can be found at
http://www.tandfonline.com/action/journalInformation?journalCode=gnpl20

Download by: [Bulgarian Academy of Sciences] **Date:** 13 February 2017, At: 23:50

NATURAL PRODUCT RESEARCH, 2017
http://dx.doi.org/10.1080/14786419.2017.1285295

Taylor & Francis
Taylor & Francis Group

SHORT COMMUNICATION

Essential oil composition of *Inula britannica* L. from Bulgaria

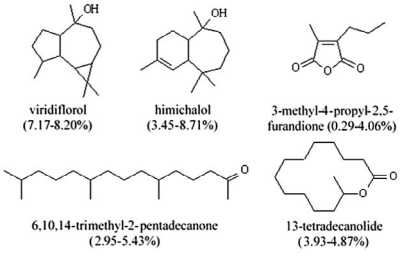
Milka Todorova[a], Antoaneta Trendafilova[a], Viktoria Ivanova[a], Kalina Danova[a] and Dimitar Dimitrov[b]

[a]Institute of Organic Chemistry with Centre of Phytochemistry, Bulgarian Academy of Sciences, Sofia, Bulgaria; [b]National Museum of Natural History, Bulgarian Academy of Sciences, Sofia, Bulgaria

ABSTRACT

The separately distilled flowers (**F**) and leaves' (**L**) essential oils of *Inula britannica* L. were investigated using capillary gas chromatography–flame ionization detector (GC–FID) and gas chromatography–mass spectrometry (GC–MS). A total of 83 constituents, representing 96.91% (**F**) and 96.73% (**L**) of the total oils, were registered. The oils were rich in terpenoids (57.85% and 77.28%), of which sesquiterpenoids dominated. The main constituents of the essential oils were viridiflorol (7.17%–8.20%) and himachalol (3.45%–8.71%) followed by 6,10,14-trimethyl-2-pentadecanone (5.43%–2.95%), 13-tetradecanolide (3.93%–4.87%) and 3-methyl-4-propyl-2,5-furandione (4.06%–0.29%).





ARTICLE HISTORY
Received 13 July 2016
Accepted 31 December 2016

KEYWORDS
Inula britannica L; Asteraceae; flower and leaf essential oil; GC; GC/MS

1. Introduction

Inula britannica L. is mainly distributed in Asia (China, Korea and Japan) and in most of the European countries. In Chinese folk medicine, it is used to treat bronchitis, digestive disorders and inflammation (Zhang et al. 2015). *I. britannica* had a wide variety of applications due to its anticancer, antioxidant, antiinflammatory, neuroprotective and hepatoprotective activities (Khan et al. 2010; Seca et al. 2014, 2015; Wang et al. 2014). Phytochemical study of this species is focused on sesquiterpene lactones, flavonoids and phenolic acids, to which are attributed its healing properties. Several reviews summarised the results on scientific investigations of bioactive secondary metabolites as well as biological activities of extracts, and individual

CONTACT Milka Todorova todorova@orgchm.bas.bg
Supplemental data for this article can be accessed at http://dx.doi.org/10.1080/14786419.2017.1285295.

© 2017 Informa UK Limited, trading as Taylor & Francis Group

compounds isolated from different *Inula* species (Khan et al. 2010; Seca et al. 2014, 2015; Wang et al. 2014). Extracts and fractions from certain *Inula* species like *I. crithmoides* L. and *I. helenium* L. exhibited different biological activities (Bucchini et al. 2015; Lee & Kang 2016). The promising results could be used for the further bioguided extensive phytochemical research. The literature survey revealed that the information about essential oil composition of *I. britannica* is scarce. To the best of our knowledge, only two articles on volatile components are published (Zha et al. 2005; Erenko et al. 2012). One more article dealt with SF-CO_2 extraction of essential oil without data on its chemical composition (Zhao et al. 2013). Therefore, in continuation of our research on secondary metabolites in *I. britannica*, the essential oil composition of the taxon of Bulgarian origin is presented in this paper.

2. Results and discussion

The obtained essential oils from flowers (**F**) and leaves (**L**) separately were subjected to GC analysis. Eighty-three components in concentration above 0.15% representing 96.91% and 96.73% of the total oils in **F** and **L**, respectively, are listed in Table S1. Nine of them were not identified, but their type was determined on the basis of their mass-spectral fragmentation, while one (MW 188) was unknown. The other substances were identified on the basis of mass spectra and RI. Various structural types of components were detected – terpene and aliphatic hydrocarbons, alcohols, ketones, lactones, aromatic and furan derivatives. As can be seen, the two oils **F** and **L** differed from each other qualitatively in the presence/absence of 15 components. Most of them were in concentrations less than 1%, but one unidentified sesquiterpene alcohol reached 3.00% in **F**. It should be noted that no one component in the studied oils exceeded 10%. The main constituents in both oils were viridiflorol (8.20% in **L**) and himachalol (8.71% in **L**) followed by 6,10,14-trimethyl-2-pentadecanone (5.43% in **F**), 13-tetradecanolide (4.87% in **L**) and 3-Me-4-propyl-2,5-furandione (4.06% in **F**). The principal group representing 60.64% (**F**) and 71.82% (**L**) consisted of oxygen containing compounds, regardless their chemical class, while hydrocarbons were 34.92% and 25.39% in flower and leaf oils, respectively. Saturated aliphatic hydrocarbons C_{23}–C_{29} were almost six times higher in flowers. Among them $C_{25}H_{52}$ dominated similarly to the earlier reported data for *I. britannica* (Erenko et al. 2012). Normal and ramified alkanes have been detected in *I. viscosa* (Haoui et al. 2011; Madani et al. 2014; Mahmoudi et al. 2016) as well as in *I. verbascifolia* (Fontana et al. 2014). Further, the total amount of terpenoids [57.85% (**F**) and 77.28% (**L**)] prevailed over the other types of components [39.06% (**F**) and 19.45% (**L**)]. Similar to *I. verbascifolia* (Fontana et al. 2014), the amount of sesquiterpenoids 45.02% (**F**) and 64.83% (**L**) dominated over monoterpenods, which were 10.47% and 10.33%, respectively. On the other hand, sesquiterpenoids in **L** were with 20% more in comparison with **F**, while the concentration of monoterpenoids was the same.

Further, different types of lactones reaching levels 5.69% (**F**) and 6.86% (**L**) were identified. Thus, the macrolide 13-tetradecanolide was registered in significant amount 3.93% (**F**) and 4.87% (**L**), while 16-hexadecanolide was found in the leaves only. Any macrolide has not been described before for *Inula* essential oils. 13-Tetradecanolide is identified in some *Achillea* species (Hayran & Demirci 2015). Macrocyclic lactones (C_{14}–C_{17}) are found in *Angelica archangelica* reaching maximum 25% in one of the studied samples (Nivinskiene et al. 2003). γ-Palmito lactone, dihydroactinidiolide, ivalin and 3-Me-4-propyl-2,5-furandione were compounds containing furanone functional group.

It is worth to note that the previously reported main components thymol, carvacrol, borneol and cis-chrysantenol in Ukrainian *I. britannica* (Erenko et al. 2012) were not detected now. Surprisingly, in the same oil, alantolactone and isoalantolactone, characteristic for *I. helenium,* have been identified, but no one of them was found neither in the oil nor in the extract (unpublished data) obtained from the Bulgarian *I. britannica*. 2,3,4,5-Tetrahydro-1-benzoxepin-3-ol, which was a major substance in the oil from this species of Chinese origin (Zha et al. 2005), also lacks in the studied now sample. Thus, the above discussed deviations in essential oil composition are an indication for intraspecific variability of *I. britannica*. This statement is in accordance with differences in the lactone profiles published up to now for *I. britannica* of different origins.

Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This work was supported by the National Science Fund, Ministry of Education and Science, Bulgaria.

References

Bucchini A, Ricci D, Messina F, Marcotullio MC, Curini M, Giamperi L. 2015. Antioxidant and antifungal activity of different extracts obtained from aerial parts of Inula crithmoides L. Nat Prod Res. 29:1173–1176.

Erenko EK, Mazulin OV, Smoylovska GP, Grechana OV, Mazulin GV. 2012. Study of component composition of essential oils *Inula helenium* L., *Inula britannica* L. of Ukraine flora. Pharm. J. (Фармац. Журн). 4:85–89 (in Ukrainian).

Fontana G, Bruno M, Senatore V, Formisano C. 2014. Volatile constituents of aerial parts of two Mediterranean species of Inula: Inula crithmoides L. and I. verbascifolia (Willd.) Hausskn. (Asteraceae). Nat Prod Res. 28:984–993.

Haoui IE, Derriche R, Madani L, Oukali Z. 2011. Analysis of the chemical composition of essential oil from Algerian *Inula viscosa* (L.) Aiton. Arabian Journal of Chemistry. 8:587–580.

Hayran M, Demirci B. 2015. Characterization of volatile compounds of eleven *Achillea* species from Turkey and biological activities of essential oil and methanol extract of *A. hamzaoglui Arabacı & Budak*. Molecules. 20:11432–11458.

Khan AL, Hussain J, Hamayun M, Gilani SA, Ahmad S, Rehman G, Kim YH, Kang SM, Lee IJ. 2010. Secondary metabolites from Inula britannica L. and their biological activities. Molecules. 15:1562–1577.

Lee SG, Kang H. 2016. Anti-neuroinflammatory effects of ethanol extract of Inula helenium L (Compositae). Trop J Pharm Res. 15:521–526.

Madani L, Derriche R, Haoui IE. 2014. Essential oil of Algerian *Inula viscosa* leaves. JEOBP. 17:164–168.

Mahmoudi H, Hosni K, Zaouali W, Amri I, Zargouni H, Hamida N, Kaddour R, Hamrouni L, Nasri MB, Ouerghi Z.. 2016. Comprehensive phytochemical analysis, Antioxidant and antifungal activities of *Inula viscosa* Aiton leaves. J Food Saf. 36:77–88.

Nivinskiene O, Butkiene R, Moskute D. 2003. Changes in the chemical composition of essential oil of *Angelica archangelica* L. root during storage. Chemija (Vilnius). 14:52–56.

Seca AML, Grigore A, Pinto DCGA, Silva AMS. 2014. The genus *Inula* and their metabolites: from ethnopharmacological to medicinal uses. J Ethnopharmacol. 154:286–310.

Seca AML, Pinto DCGA, Silva AMS. 2015. Metabolomic profile of the genus Inula. Chem Biodivers. 12:859–906.

Wang GW, Qin JJ, Cheng XR, Shen YH, Shan L, Jin HZ, Zhang WD. 2014. *Inula* sesquiterpenoids: structural diversity, cytotoxicity and anti-tumor activity. Expert Opin Investig Drugs. 23:318–345.

Zha J, Fu Y, Wu Y, Guo C, Zhang D, Wang Y. 2005. Study of chemical constituents of the essential oil from *Inula britannica* L. by GC-MS. J Chin Med Mater. 28:466–468.
Zhang XF, Du JL, Ren J, Ye FM, Xie YG, Cheng XR, Yan SK, Jin HZ. 2015. Three new sesquiterpene lactones from *Inula britannica*. Arch Pharm Res. 38:666–672.
Zhao T, Gao F, Zhou L, Song T-Y. 2013. Essential oil from *Inula britannica* extraction with SF-CO2 and its antifungal activity. J Integr Agric. 12:1791–1798.

SUPPLEMENTARY MATERIAL

Essential oil composition of *Inula britannica* L. from Bulgaria

Milka Todorova[a*], Antoaneta Trendafilova[a], Viktoria Ivanova[a], Kalina Danova[a], Dimitar Dimitrov[b]

[a] *Institute of Organic Chemistry with Centre of Phytochemistry, Bulgarian Academy of Sciences, Acad. G. Bonchev str., bl.9, 1113 Sofia, Bulgaria*

[b] *National Museum of Natural History, Bulgarian Academy of Sciences, 1, Tsar Osvoboditel Blvd., 1000 Sofia, Bulgaria*

Abstract: The separately distilled flowers (**F**) and leaves (**L**) essential oils of *Inula britannica* L. were investigated using capillary gas chromatography–flame ionization detector (GC–FID) and gas chromatography–mass spectrometry (GC–MS). A total of eighty-three constituents, representing 96.91% (**F**) and 96.73% (**L**) of the total oils were registered. The oils were rich in terpenoids (57.85% and 77.28%), of which sesquiterpenoids dominated. The main constituents of the essential oils were viridiflorol (7.17% - 8.20%) and himachalol (3.45% - 8.71%) followed by 6,10,14-trimethyl-2-pentadecanone (5.43% - 2.95%), 13-tetradecanolide (3.93%- 4.87%) and 3-methyl-4-propyl-2,5-furandione (4.06% - 0.29%).

Key words: *Inula britannica* L.; Asteraceae; flower and leaf essential oil; GC; GC/MS.

Experimental

Plant material and isolation of essential oil

Aerial parts of *I. britannica* were collected in July, 2015 in full flowering stage from Northern Balkan Region in Bulgaria. Species was identified by Prof. D. Dimitrov and a voucher specimen (SOM 172474) was deposited in the Herbarium of the Institute of Biodiversity and Ecosystem Research, Bulgarian Academy of Sciences. Air-dried flowers and leaves separately were subjected to a micro distillation-extraction in a Likens-Nickerson apparatus for 3 hours using diethyl ether as a solvent. The essential oil dissolved in diethyl ether was dried over anhydrous Na_2SO_4. The solvent was removed

under N_2 flow, and the sample was stored at 4ºC before analysis. The oil yield was 0.6 mg/g DW from flowers and 0.4 mg/g DW from leaves.

***Gas chromatography (GC) and gas chromatography mass spectrometry (GC/MS)*:**

GC analysis was carried out on a HP 5890 Gas Chromatograph fitted with an HP 5 MS capillary column (30 m x 0.25 mm, 0.25 μm film thickness), carrier gas nitrogen, linear velocity 25 cm/s, split ratio 1:100. The injector and detector temperature was 260ºC; column temperature was programmed from 60ºC (5 min isotherm) then to 230ºC at a rate of 4 ºC/min and then held for 10 min at 230ºC. Quantitative data were obtained from the electronic integration of the FID peak areas.

GC/MS analysis was performed on a HP 6890 Plus instrument with MS detector 5973 of the same company which was operated in EI mode (70 eV). All GC conditions and the capillary column used were as described above, but the carrier gas was helium. Retention indices (RI) of the oil components were calculated by using retention times of C_8-C_{30} n-alkanes under the same chromatographic conditions according to Van den Dool's method (Van den Dool and Kratz 1963). The individual components were identified by their RI, referring to known compounds from the literature (Adams 2009, Tkachev 2008) and also by comparison of their MS with those of NIST 98, WILEY and home-made MS databases.

Table S1. Essential oil composition (%) in different plant parts of *I. britannica* L.

RI	Component	Flowers	Leaves
848	*trans*-2-Hexenal	0.75	0.60
865	1-Hexenol	0.71	-
975	1-Octen-3-ol	0.61	0.28
976	β-Pinene	1.71	1.21
985	Hexenoic acid (3E)	1.11	0.59
996	2*E*,4*Z*-Heptadienal	-	0.38
1005	2*E*,4*E*-Heptadienal	0.47	0.49
1026	Limonene	0.25	-
1028	1,8-Cineole	2.00	1.06
1031	Benzyl alcohol	0.21	0.25
1040	Phenyl acetaldehyde	0.28	0.64
1059	γ-Terpinene	0.23	0.11
1064	*trans*-Sabinene hydrate	0.27	0.30
1096	*cis*-Sabinene hydrate	0.40	0.51
1101	Nonyl aldehyde	0.56	0.22
1114	Phenyl ethyl alcohol	0.34	0.24
1118	3-Methyl-4-propyl-2,5-furandione	4.06	0.29
1137	*trans*-Pinocarveol	0.82	1.38
1147	Z-Nonenal	0.37	0.32
1164	Pinocarvone	0.59	0.91
1175	Terpinen-4-ol	0.71	0.51
1187	n-Decanal	0.40	0.69
1191	α-Terpineol	0.57	-
1195	Myrtenol	0.86	0.9
1197	Myrtenal	0.68	1.1
1212	Verbenone	-	0.66
1269	Decanol	0.22	0.60
1312	2*E*,4*E*-Decadienal	1.11	0.42

1314	Theaspirane B	0.23	-
1352	α-Longipinene	0.66	-
1357	Eugenol	0.58	0.34
1376	α-Copaene	0.3	0.28
1388	β-Bourbonene	0.86	0.95
1391	β-Elemene	0.97	2.22
1415	α-Cedrene	0.25	0.74
1419	MW 188	0.18	0.60
1424	β-Ylangene	0.16	-
1433	β-Copaene	0.24	0.20
1436	Neryl acetone	0.80	1.34
1451	α-Himachalene	0.51	0.48
1461	*allo*-Aromadendrene	0.49	0.51
1477	γ-Muurolene	1.42	2.23
1481	β-Ionone-epoxide	-	0.35
1485	α-Amorphene	1.81	2.05
1490	β-Selinene	3.42	3.60
1499	α-Selinene	-	0.45
1524	δ-Cadinene	1.54	2.23
1529	*cis*-Calamenene	0.47	0.67
1534	Dihydroactinidiolide	-	0.85
1559	$C_{15}H_{24}O$ (MW 220)	1.21	2.50
1579	Spathulenol	1.12	1.63
1584	Caryophyllene oxide	1.53	5.40
1595	Viridiflorol	7.17	8.20
1604	Ledol	-	0.78
1620	$C_{15}H_{24}O$ (MW 220)	-	1.03
1645	T-Cadinol	1.78	2.06
1647	T-Muurolol	1.94	3.22
1650	$C_{15}H_{26}O$ (MW 222)	1.58	2.66

1653	13-Tetradecanolide	3.93	4.87
1658	Himachalol	3.45	8.71
1667	Intermedeol	2.97	2.44
1687	Eudesma-4(15),7-diene-1β-ol	1.51	1.10
1706	$C_{15}H_{24}O$ (MW 220)	-	0.96
1733	$C_{15}H_{24}O$ (MW 220)	1.13	2.37
1735	$C_{15}H_{24}O$ (MW 220)	0.87	1.10
1743	$C_{15}H_{22}O$ (MW 218)	-	0.82
1782	$C_{15}H_{26}O_2$ (MW 238)	0.65	0.77
1795	E-Isovalencenol	0.82	1.01
1809	$C_{15}H_{24}O$ (MW 220)	3.00	-
1844	6,10,14-Trimethyl-2-pentadecanone	5.43	2.95
1851	16-Hexadecanolide	-	0.57
1862	Farnesyl acetone	0.49	0.50
1869	Ivalin	0.52	0.36
2068	Dihydrophytol	1.03	0.25
2115	Phytol	1.33	1.87
2121	γ-Palmitolactone	1.24	0.21
2300	Tricosane	0.37	0.22
2400	Tetracosane	0.53	0.19
2500	Pentacosane	7.44	1.46
2600	Hexacosane	0.53	0.19
2700	Heptacosane	4.77	1.04
2800	Octacosane	0.47	-
2900	Nonacosane	2.92	0.54
	Hydrocarbons	32.5	22.17
	Oxygenated hydrocarbons	64.41	74.56
	Terpenoids	57.85	77.28
	Monoterpenoids	10.47	10.33

Sesquierpenoids	45.02	64.83
Others	39.06	19.45
Total	96.91	96.73
Oil yield (% w/w)	0.6 mg/g	0.4 mg/g

References:

Adams RP. 2009. Identification of Essential Oil Components by Gas Chromatography/Mass Spectrometry. 4th ed., Allured Publ. Corp., Carol Stream, IL.

Tkachev AV. 2008. Analysis of plant volatile compounds. Novosibirsk, Ofset Publ. Company.

Van den Dool H and Kratz PD. 1963. A generalization of the retention index system including linear temperature programmed gas-liquid partition chromatography. Journal of Chromatography. 11:463-471