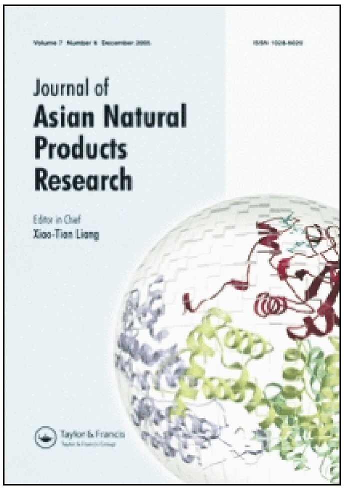
This article was downloaded by:
On: *22 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Journal of Asian Natural Products Research
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713454007

Two new compounds from *Ixeris sonchifolia*

Nan Zhang[a]; A-Li Lv[a]; Zhe Zheng[a]; Yi-Mei Zeng[a]; Ying-Na Li[a]; Yue-Hu Pei[a]
[a] School of Traditional Chinese Materia Medica, Shenyang Pharmaceutical University, Shenyang, China

To cite this Article Zhang, Nan , Lv, A-Li , Zheng, Zhe , Zeng, Yi-Mei , Li, Ying-Na and Pei, Yue-Hu(2008) 'Two new compounds from *Ixeris sonchifolia*', Journal of Asian Natural Products Research, 10: 3, 211 — 213
To link to this Article: DOI: 10.1080/10286020701395362
URL: http://dx.doi.org/10.1080/10286020701395362

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

Journal of Asian Natural Products Research
Vol. 10, No. 3, March 2008, 211–213

Downloaded At: 19:01 22 January 2011

Two new compounds from *Ixeris sonchifolia*

Nan Zhang, A-Li Lv, Zhe Zheng, Yi-Mei Zeng, Ying-Na Li and Yue-Hu Pei*

School of Traditional Chinese Materia Medica, Shenyang Pharmaceutical University, Shenyang 110016, China

(*Received 2 November 2006; final version received 29 March 2007*)

Two new compounds, named as sonchifolactone E (**1**) and sonchifolinin B (**2**), have been isolated from the whole plant of *Ixeris sonchifolia*, along with one known compound, sonchifolatone A (**3**). Their structures and stereochemistry were determined by spectroscopic methods.

Keywords: *Ixeris sonchifolia*; Compositae; sonchifolatone E; sonchifolinin B

1. Introduction

Ixeris sonchifolia (Bge.) Hance (Compositae) is abundantly distributed throughout northeastern China and commonly found in dry places. It has been used as a folk medicine in China for invigorating circulation of blood, normalising menstruation and eliminating blood stasis.[1] Studies on other species of this genus revealed the presence of sesquiterpene lactones[2] which showed wide biological activities, such as cytotoxicity,[3] anti-repellant [4] and anti-feedant to some insects.[5] Further investigation on this paper has led to the isolation of two new compounds, sonchifolactone E (**1**) and sonchifolinin B (**2**, Figure 1), and one known compound, sonchifolactone A (**3**). We report here the isolation and structural elucidation of these compounds.

2. Results and discussion

Compound **1** with $[\alpha]_D^{25}+81$ (*c* 0.005, MeOH) and mp 164–166°C, was obtained as yellow needles. The molecular formula, $C_{15}H_{18}O_5$, was determined by HRFAB-MS, which showed the $[M+H]^+$ ion peak at *m*/*z* 279.1150. IR spectrum showed the presence of hydroxyl (3450 cm^{-1}) and a γ-lactone carbonyl (1770, 1670 cm^{-1}) groups. The ^1H NMR spectrum (Table 1) displayed three methyl groups, of which two were assigned to 14,15-vinyl methyls [δ 2.37 (3H, s, H-14), δ 1.99 (3H, s, H-15)], and one to the α-methyl in a γ-lactone ring [δ 1.26 (3H, s, H-13)]. The signal at δ 3.72 (1H, t, $J=10.0$ Hz) was assigned to H-6, which coupled with both H-5 [δ 3.36 (1H, d, $J=10.0$ Hz)] and H-7 [δ 2.07 (1H, dd, $J=10.0$, 2.0 Hz)] and thus established the trans-diaxial relationship of these three protons. Since the naturally occurring guaianolides have an α-oriented H-7,[6] this meant that the orientations of H-5 and H-6 were α and β, respectively. In addition, the ^1H NMR spectrum established the presence of two hydroxyl signals at δ 9.21 (1H, brs, 3-OH) and 5.81 (1H, brs, 11-OH). Fifteen carbon signals were observed in the ^{13}C NMR spectrum (Table 1), the two most downfield signals were assigned to an α,β-unsaturated ketone carbonyl (δ 189.0) and a γ-lactone carbonyl (δ 176.8). The other signals were those for four tertiary olefinic carbons, two methylenes, one tertiary carbon, three methines, and three methyls. In the HMBC spectrum of compound **1**, the H-5 at δ 3.36 showed long-range correlations with C-1 (δ 128.8), C-3 (δ 152.7), C-4 (δ 135.8), and C-7 (δ 56.8); H-6 (δ 3.72) with C-1 (δ 128.8) and C-8 (δ 21.2), respectively. Furthermore, the HMBC spectrum exhibited correlations from the methyl protons 13-Me (δ 1.26) to C-12 (δ 176.8), C-7 (δ 56.8), and C-11 (δ 72.6); 14-Me (δ 2.37) to C-1 (δ 128.8), C-10 (δ 153.3), and C-9 (δ 36.3); 15-Me (δ 1.99) to C-3 (δ 152.7) and C-4 (δ 135.8), respectively. Therefore, all of the signals were assigned according to HMQC and HMBC spectra. Additionally, the relative configuration of 11-OH was determined as β orientation from NOESY correlations of 6β-H with 8β-H; 13-Me with 8α-H, respectively (Figure 1). Thus, compound **1** was shown to be 3,11β-dihydroxyl-1(10),3-guaiadiene-12,6-olide-2-one, named sonchifolactone E.

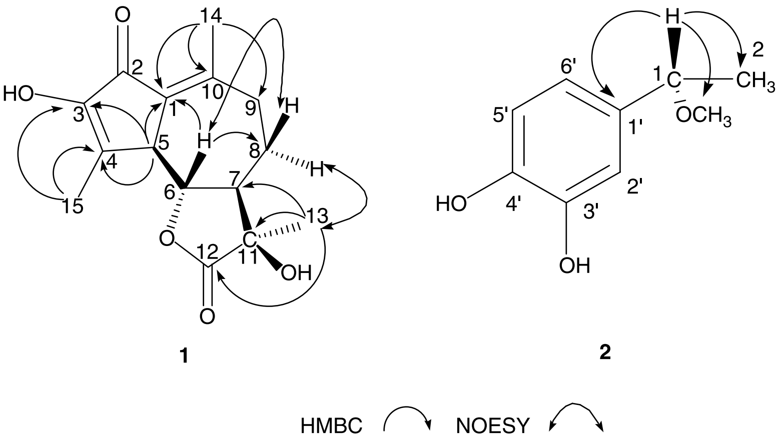
Compound **2**, yellow solid, $[\alpha]_D^{25}+11$ (*c* 0.004, MeOH), mp 135–138° ×, was assigned a molecular formula of $C_9H_{12}O_3$, on the basis of HRFAB-MS *m*/*z* 169.0779 $[M+H]^+$. UV spectrum showed absorption maxima at 230 and 280 nm. The ^1H NMR spectrum (Table 2) displayed an ABX system at δ 6.68 (1H, d, $J=8.1$ Hz, H-5′), 6.52 (1H, dd, $J=1.8$, 8.1 Hz, H-6′), and 6.67 (1H, d, $J=1.8$ Hz, H-2′), which suggested the presence of a 1,3,4-trisubstituted benzene moiety. In addition, the ^1H NMR spectrum showed a methyl proton at δ 1.25 (3H, d, $J=6.4$ Hz, H-2) which was coupled

---

*Corresponding author. Email: peiyueh@vip.163.com

ISSN 1028-6020 print/ISSN 1477-2213 online
© 2008 Taylor & Francis
DOI: 10.1080/10286020701395362
http://www.informaworld.com

Downloaded At: 19:01 22 January 2011



1

2

Figure 1. Structures of compounds **1** and **2**.

with a benzylic proton at δ 4.11 (1H, q, $J = 6.4$ Hz, H-1). A methoxyl proton at δ 3.04 (3H, s, OCH_3) and two hydroxyl proton at δ 8.85 (2H, brs, 3′,4′-OH) were also observed. The ^{13}C NMR spectrum (Table 2) indicated that there were a methyl at δ 23.8 (C-2), a methine which assigned to an oxygen-bearing carbon at δ 78.4 (C-1), a methoxyl at δ 55.5 (OCH_3), in addition to six aromatic carbons in the molecule. Furthermore, the HMBC spectrum suggested correlations of the benzylic proton H-1 (δ 4.11) to C-2 (δ 23.8), C-1′ (δ 134.3), and 1-OCH_3 (δ 55.5). All of the signals were assigned according to HMQC and HMBC spectra. The absolute configuration at stereocentre C-1 was determined by CD spectrum. In the CD spectrum, compound **2** gave a positive Cotton effect at 280 nm which indicated that **2** had the *R* configuration at C-1.[7] Thus, compound **2** was revealed as (1*R*)-3′,4′-dihydroxyphenylmethoxylethane, named sonchifolinin B.

Table 1. ^{13}C NMR (150 MHz) and ^{1}H NMR (600 MHz) spectral data of compound **1** (DMSO-d_6).

Position	δ_H	δ_C
1		153.3
2		189.0
3	9.21 (brs, —OH)	152.7
4		135.8
5	3.36 (d, $J = 10.0$ Hz)	46.5
6	3.72 (t, $J = 10.0$ Hz)	84.9
7	2.07 (dd, $J = 10.0, 2.0$ Hz)	56.8
8	1.35 (m)	
1.74 (m)	21.2	
9	2.22 (m)	
2.42 (m)	36.3	
10		128.8
11	5.81 (brs, —OH)	72.6
12		176.8
13	1.26 (s)	20.6
14	2.37 (s)	20.9
15	1.99 (s)	14.2

3. Experimental

3.1 General experimental procedures

Optical rotation was obtained in CH_3OH at 20°C, using a P-E 241 MC; Melting points were determined with a Yanaco micro-melting point apparatus and are uncorrected. CD was taken on a JASCO polarimeter; IR spectra were recorded on a NEXUS-470 spectrophotometer; NMR spectra were recorded on a Bruker-ARX-300 and 600 spectrometer, using DMSO-d_6 as solvent and TMS as internal standard; HRESI-MS was carried on Q-trap LC-MS-MS; Column chromatography was carried out on silica gel (200–300 mesh) and Sephadex LH-20; HPLC was performed with a Daojin LC-10AT*vp* and LC-8A. Fractions were monitored by TLC and spots were visualised by silica gel GF254 plates sprayed with 10% H_2SO_4.

3.2 Plant material

The whole plant of *Ixeris sonchifolia* was collected in August 2005 at Shenyang city, Liaoning province, China, and identified by Professor Qi-Shi Sun, Department of Pharmacognosy, Shenyang Pharmaceutical University. The voucher specimen has been deposited in the Department of Phytochemistry, Shenyang Pharmaceutical University (No.6032).

3.3 Extraction and isolation

Dried whole plant (9 kg) was extracted three times with H_2O at boiling temperature. The combined extracts (1.5 kg) were successively partitioned with petroleum ether, $CHCl_3$, EtOAc, and *n*-BuOH. The *n*-BuOH fraction (100 g) was subjected to column chromatography on silica gel and eluted with $CHCl_3$/MeOH (100:0 to 1:1) to yield 12 fractions. Fraction 3 was purified by Sephadex LH-20 and eluted with $CHCl_3$/MeOH (1:1) to

Table 2. ^{13}C (150 MHz) and ^{1}H NMR (600 MHz) spectral data of compound **2** (DMSO-d_6).

Position	δ_H	δ_C
1	4.11 (q, $J = 6.4$ Hz)	78.4
2	1.25 (d, $J = 6.4$ Hz)	23.8
1′		134.3
2′	6.67 (d, $J = 1.8$ Hz)	113.4
3′	8.85 (brs, —OH)	144.6
4′	8.85 (brs, —OH)	145.3
5′	6.68 (d, $J = 8.1$ Hz)	115.4
6′	6.52 (dd, $J = 1.8$, 8.1 Hz)	117.3
1″	3.04 (s)	55.5

give compound **3** (0.1 g). Then fraction 4 was subjected to Sephadex LH-20 by eluting with $CHCl_3$/MeOH (1:1) to afford fraction C_{1-4}. Fraction C_2 was further separated by reversed-phase preparative HPLC using MeOH/H_2O (50:50) as mobile phase to yield compounds **1** (9 mg) and **2** (7 mg).

*3.3.1 Compound **1***

Yellow needles (MeOH); $[\alpha]_D^{25} + 81$ (*c* 0.005, MeOH); mp 164–166°C; UV (MeOH) λ_{max}: 234, 215 nm; IR (KBr) cm^{-1}: 3450, 1770, 1670, 1620, 1400, 1210; ^{1}H NMR and ^{13}C NMR spectral data are given in Table 1; HRFAB-MS: *m/z* 279.1150 $[M + H]^+$ (calcd for $C_{15}H_{18}O_5$, 279.1232).

*3.3.2 Compound **2***

Yellow solid (MeOH); $[\alpha]_D^{25} + 11$ (*c* 0.004, MeOH); mp 135–138°C; UV (MeOH) λ_{max}: 230, 280 nm; IR (KBr) cm^{-1}: 3250, 1620, 1570, 1500, 1450, 1240, 1150; ^{1}H NMR and ^{13}C NMR spectral data are given in Table 2; HRFAB-MS: *m/z* 169.0779 $[M + H]^+$ (calcd for $C_9H_{12}O_3$, 169.0865).

*3.3.3 Compound **3***

By comparison of NMR and IR spectral data with those in the literature,[8] **3** was identified as sonchifolactone A.

Downloaded At: 19:01 22 January 2011

Acknowledgements

The authors are very grateful to Professor Qishi Sun of Shenyang Pharmaceutical University for identifying the plant materials and Mr. Yi Sha, Analytical Centre of Shenyang Pharmaceutical University for some NMR measurements.

References

[1] Jiangsu Medical College. *Encyclopedia of Chinese Materia Medica* (Shanghai People's Publisher, Shanghai, 1977), p. 1300.
[2] H.S. Chung and S. Jalim. *Phytochemistry* **35**, 1583 (1994).
[3] A.L. Okunade and D.F. Wiemer. *Phytochemistry* **24**, 1199 (1985).
[4] M. Seto, T. Miyase, K. Umehara, A. Ueno, Y. Hirano, and N. Otani. *Chem. Pharm. Bull.* **36**, 2423 (1988).
[5] R. Srivastava, P. Proksch, and V. Wray. *Phytochemistry* **29**, 3445 (1990).
[6] K. Nishimura, T. Miyase, A. Uneo, T. Noro, M. Kuroyanagi, and S. Fukushima. *Phytochemistry* **25**, 2375 (1986).
[7] T. Ishida, V.J. Bounds, J. Caldwell, A. Drake, and M. Takeshita. *Tetrahedron* **7**, 3113 (1996).
[8] J.Y. Ma, Z.T. Wang, L.S. Xu, G.J. Xu, S. Kadota, and T. Namba. *Phytochemistry* **48**, 201 (1998).