

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 Novel Phenolic Triterpenes from *Tripterygium Wilfordii*

Guang-Zhong Yang^a; Ming-Lei Xi^a; Yuan-Chao Li

^a Shanghai Institute of Materia Medica, Chinese Academy of Sciences, Shanghai

To cite this Article Yang, Guang-Zhong , Xi, Ming-Lei and Li, Yuan-Chao(2011) 'Two Novel Phenolic Triterpenes from *Tripterygium Wilfordii*', Journal of Asian Natural Products Research, 3: 2, 83 – 88

To link to this Article: DOI: 10.1080/10286020108041374

URL: <http://dx.doi.org/10.1080/10286020108041374>

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.

TWO NOVEL PHENOLIC TRITERPENES FROM *TRIPTERYGIUM WILFORDII*

GUANG-ZHONG YANG, MING-LEI XI and YUAN-CHAO LI*

*Shanghai Institute of Materia Medica, Chinese Academy of Sciences,
294 Tai-yuan Road, Shanghai, 200031*

(Received 10 April 2000; In final form 18 May 2000)

Two novel phenolic triterpenes were isolated from *Tripterygium wilfordii* Hook. f., their structures were identified to be 2,3-dihydroxy-1,3,5(10),8-tetra-ene-6 α -(2'-hydroxyethyl)-24-nor-D:A-friedooleanane-29-oic acid **1**, named triptotin **F**, and 2,3-dihydroxy-1,3,5(10),8-tetra-ene-6 β -(2'-hydroxyethyl)-24-nor-D:A-friedooleanane-29-oic acid **2**, named triptotin **G** on the basis of spectroscopic studies.

Keywords: *Tripterygium wilfordii* Hook. f.; *Celastraceae*; Triterpenes; Friedooleanane

INTRODUCTION

Tripterygium wilfordii Hook. f., commonly called “Lei Gong Teng” in China, is a perennial twining vine belonging to the family *Celastraceae*. *Tripterygium wilfordii* Hook. f. is known to contain a number of constituents, some of which appear to be toxic. Because of its toxicity, “Lei Gong Teng” was only used as pesticide in previous times. Recently, “Lei Gong Teng” and its preparation “Lei Gong Teng Duo Dai” have been used for the treatment of various diseases, including dermatitis, rheumatoid arthritis, systemic acne rosacea, and nephritis [1]. In the course of our continuing search for novel active components from this plant, two novel phenolic triterpenes were isolated and their structures were identified to be 2,3-dihydroxy-1,3,5(10),8-tetra-ene-6 α -(2'-hydroxyethyl)-24-nor-D:

*Corresponding author. Tel.: +86-21-64311833-422, Fax: +86-21-64370269, e-mail: ycli@mail.shenc.ac.cn

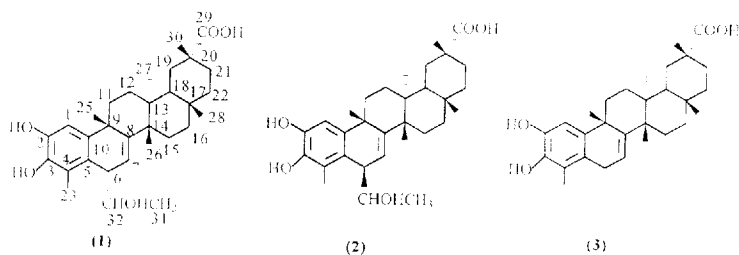


FIGURE 1 Structures of compounds (1)–(3).

A-friedooleanane-29-oic acid **1**, named triptotin **F**, and 2,3-dihydroxy-1, 3, 5(10), 8-tetra-cene-6 β -(2'-hydroxyethyl)-24-nor-D:A-friedooleanane-29-oic acid **2**, named triptotin **G** (Fig. 1).

RESULTS AND DISCUSSION

Compound **1**, amorphous powder, high-resolution mass spectrum gave a molecular formula of $C_{31}H_{44}O_5$. 1H NMR spectrum revealed the presence of seven methyls [δ 1.21, 1.25, 1.44, 1.47, 1.97, 2.50 (each 3H, s), δ 1.61 (3H, d, $J=6.3$ Hz)], one methine [δ 4.46 (1H, m)] attached to one oxygen function, one olefinic proton [δ 6.20 (1H, d, $J=6$ Hz)], and one aromatic proton [δ 7.13 (1H, s)]. ^{13}C NMR spectrum of **1** showed one carbonyl carbon signal (δ 181.3), six aromatic carbon signals [δ 110.7 (d), δ 143.4 (s), δ 145.2 (s), δ 121.4 (s), δ 126.7 (s), δ 142.6 (s)], two olefinic carbon signals [δ 119.6 (d), δ 151.1 (s)], seven methyl carbon signals, seven methylene carbon signals, one oxygenated methine carbon signal [δ 70.8], two methine carbon signals, and five quaternary carbon signals. The formula required ten unsaturated equivalents. Except for one carbonyl group, one benzene ring, and one double bond, the four remaining degrees of unsaturation were ascribed to four carbocyclic systems. Thus triptotin **F** should be a pentacyclic triterpenoid. We found that the ^{13}C NMR spectrum data of **1** were very similar to those of triptohypol **C** **3** which was isolated from *Tripterygium hypoglaucum* [2]. Analyses of these signals by HMQC and HMBC experiments proved that the structures of A, C, D, E ring in compound **1** were similar to those of triptohypol **C** and the double bond was located at C-7 and C-8. From 1H - 1H COSY and HMQC spectrum two partial structures (see Fig. 2) were obtained in relation to ring B.

So, the carbon signal at δ 45.4 could be assigned to C-6. In HMBC spectrum (see Fig. 3), the proton signal at δ 1.61 (3H, d, $J=6.3$ Hz) was

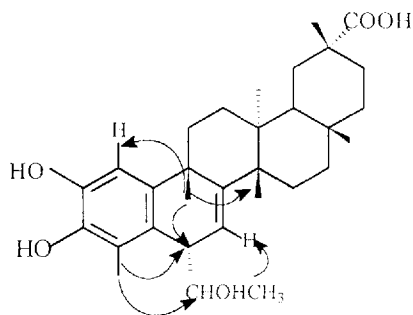


FIGURE 4 NOESY for 1.

EXPERIMENTAL SECTION

General Experimental Procedures

IR spectra were recorded on a Perkin-Elmer 599B IR spectrometer. Optical rotations were measured on a JASCO DIP-181 polarimeter, using a 10-cm microcell. MS spectra were measured on a MAT-711 spectrometer. $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectra were recorded on Bruker AM-400 instrument with TMS as internal standard. Silica gel chromatography was performed using silica gel 60H at Qingdao Haiyang Chemical Group Co.

Plant Material

The roots of *Tripterygium wilfordii* were collected in Fujian Province, China. The plant material was identified by Prof. Guan-Yuan Gu, Scientific and Technical Archives of Shanghai Medical University, Shanghai, China.

Extraction and Isolation

The air-dried roots (200 kg) of *Tripterygium wilfordii* were powdered and extracted with 95% EtOH. The EtOH extract was extracted with CHCl_3 . The CHCl_3 -soluble fraction (500 g) was chromatographed on silica gel with CHCl_3 -MeOH (95:5, 9:1, 8:2, MeOH) to give 5 fractions (A-E). Fraction B (95 g) was chromatographed over a silica gel column with CHCl_3 -MeOH (98:2, 95:5, 9:1, MeOH) to give 8 fractions. Fraction 4 (15 g) was chromatographed over a silica gel column eluting with CH_2Cl_2 -MeOH (98:2) to give 3 fractions (fractions 4.1-4.3), and frac-

tion **4.1** (7.5 g) was then subjected to column chromatography on silica gel with cyclohexane–acetone (5:1, 3:1, 1:1) to give **4** fractions (fractions **4.1.1–4.1.4**). Fraction **4.1.3** (2.5 g) was chromatographed over a silica gel column eluting with CH₂Cl₂–MeOH (98:2) to afford **3** fractions (fractions **4.1.3.1–4.1.3.3**). Fraction **4.1.3.3** (1.1 g) was subjected to RP-18 column chromatography with MeOH–H₂O (3:2) and column chromatography on silica gel with CH₂Cl₂–MeOH (98:2) to give compound **1** (37.6 mg). Fraction **6** (25 g) was chromatographed over a silica gel column eluting with petroleum ether–ethyl acetate (5:1, 5:2, 3:1, 1:1) to give **9** fractions (fractions **6.1–6.9**). Fraction **6.5** (3.7 g) was chromatographed over a silica gel column eluting with CH₂Cl₂–MeOH (98:2) to give compound **2** (250 mg).

Triptotin **F** (**1**), amorphous powder; $[\alpha]_D^{20} - 67.4$ (*c* 0.38, CHCl₃); IR $\nu_{\max}^{\text{KBr}} \text{cm}^{-1}$: 3400, 1697, 1606, 1461, 1209, 1202, 871; ¹HNMR (C₅D₅N, 400 MHz): δ 1.21 (3H, s, H₃-28), 1.25 (3H, s, H₃-27), 1.44 (3H, s, H₃-26), 1.47 (3H, s, H₃-30), 1.97 (3H, s, H₃-25), 2.50 (3H, s, H₃-23), 1.61 (3H, d, *J* = 6.3 Hz, H₃-31), 3.85 (1H, dd, *J* = 6, 3.5 Hz, H-6), 4.46 (1H, m, H-32), 6.20 (1H, d, *J* = 6 Hz, H-7), 7.13 (1H, s, H-1), ¹³CNMR (see Tab. I); EIMS *m/z* (rel.int.): 496[M]⁺ (3), 451 (19), 215 (21), 201 (100), 95 (17), 69 (10), HRMS *m/z* 496.3155 [M]⁺ (C₃₁H₄₄O₅, required 496.3189).

Triptotin **G** (**2**), amorphous powder; $[\alpha]_D^{20} - 56.0$ (*c* 0.28, MeOH); IR $\nu_{\max}^{\text{KBr}} \text{cm}^{-1}$: 3412, 1700, 1462, 1377, 1288, 1207, 1018, 871; ¹HNMR (C₅D₅N, 400 MHz): δ 1.20 (3H, s, H₃-28), 1.23 (3H, s, H₃-27), 1.37 (3H, s, H₃-26), 1.46 (3H, s, H₃-30), 1.73 (3H, s, H₃-25), 2.75 (3H, s, H₃-23), 1.45 (3H,

TABLE I ¹³CNMR data of compound **1–3***

C	1	2	3	C	1	2	3
1	110.7	110.6	109.8	17	31.1	30.9	30.9
2	143.4	141.3	140.7	18	45.1	44.9	44.8
3	145.2	143.1	142.7	19	31.3	31.1	31.2
4	121.4	122.5	121.4	20	40.8	40.6	40.6
5	126.7	126.0	124.2	21	31.4	31.2	30.5
6	45.4	46.1	28.3	22	35.9	35.7	35.5
7	119.6	120.9	118.4	23	13.4	13.8	12.2
8	151.1	+	149.9	24			
9	38.3	38.1	37.1	25	36.5	35.7	34.8
10	142.6	145.1	145.1	26	22.7	22.4	23.1
11	37.6	37.7	35.1	27	18.9	18.8	18.8
12	31.4	30.6	30.7	28	32.0	31.9	31.8
13	38.8	38.7	38.2	29	181.3	181.0	181.1
14	44.7	44.5	44.0	30	33.5	33.3	33.3
15	29.9	29.7	29.4	31	22.7	21.4	
16	37.6	37.4	37.3	32	70.8	72.1	

* Compound **1–3** in C₅D₅N.

+ Signal is hidden by a solvent peak.

d, $J=6$ Hz, H₃-31), 4.15 (1H, t, $J=6$ Hz, H-6), 4.41 (1H, m, H-32), 6.27 (1H, d, $J=6$ Hz, H-7), 7.24 (1H, s, H-1); ¹³CNMR (see Tab. 1); EIMS m/z (rel.int.): 496[M]⁺ (3), 451 (16), 215 (20), 201 (100); HRMS m/z 496.3193 [M]⁺ (C₃₁H₄₄O₅, required 496.3189).

References

- [1] Chen, K., Shi, Q., Fujioka, T., Zhang, D. C., Hu, C. Q., Jin, J. Q., Kilkuskie, R. and Lee, K. H. (1992), *J. Nat. Prod.*, **55**, 88–92.
- [2] Duan, H. Q., Kawazoe, K., Bando, M., Kido, M. and Takaishi, Y. (1997). *Phytochemistry*, **46**, 535–543.