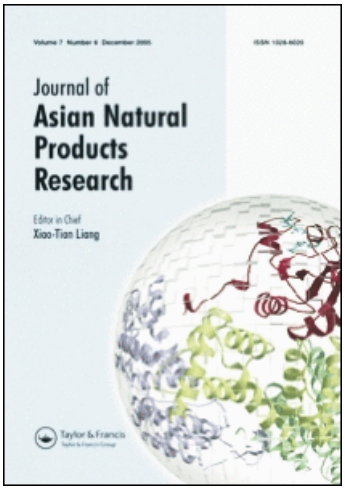
This article was downloaded by:
On: *22 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



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

A new compound from *Gentianopsis paludosa*

H.-D. Wang[a]; C.-Y. Tan[a]; X.-F. Bai[a]; Y.-G. Du[a]; B.-C. Lin[b]
[a] Graduate School of the Chinese Academy of Sciences, Dalian, China [b] Laboratory of Microfluidics for Systems Biology, Dalian Institute of Chemical Physics, Chinese Academy of Sciences, Dalian, China

To cite this Article Wang, H.-D. , Tan, C.-Y. , Bai, X.-F. , Du, Y.-G. and Lin, B.-C.(2007) 'A new compound from *Gentianopsis paludosa*', Journal of Asian Natural Products Research, 9: 3, 307 — 309
To link to this Article: DOI: 10.1080/10286020600727459
URL: http://dx.doi.org/10.1080/10286020600727459

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. 9, No. 3, April–May 2007, 307–309

Taylor & Francis
Taylor & Francis Group

A new compound from *Gentianopsis paludosa*

H.-D. WANG†‡§, C.-Y. TAN†‡, X.-F. BAI†‡, Y.-G. DU†‡* and B.-C. LIN‡¶

†The Natural Products and Glycoconjugate Research Group 1805, Dalian Institute of Chemical Physics, Chinese Academy of Sciences, Dalian 116023, China
‡Graduate School of the Chinese Academy of Sciences, Dalian, China
¶Laboratory of Microfluidics for Systems Biology, Dalian Institute of Chemical Physics, Chinese Academy of Sciences, Dalian 116023, China

Downloaded At: 19:10 22 January 2011

(Received 16 February 2006; revised 18 February 2006; in final form 18 February 2006)

A new compound, named gentianopfluorenone (**1**), along with three known compounds,1-*O*-β-D-glucopyranosyl-5-hydroxy-3-methoxyxanthone (**2**), 1-*O*-[β-D-xylopyranosyl-(1 → 6)-β-D-glucopyranosyl]-7,8-dihydroxy-3–methoxyxanthone (**3**), and apigenin (**4**), were isolated from the whole herb of *Gentianopsis paludosa*. On the basis of spectral and chemical evidence, the structure of **1** was elucidated as 4,4a,6-trihydroxy-5-methoxy-fluoren-2,9-dione. Compounds **2**–**4** were isolated from the plant for the first time.

Keywords: *Gentianaceae*; *Gentianopsis paludosa*; Gentianopfluorenone; Xanthone

1. Introduction

Gentianopsis paludosa (Hook.f.) Ma (Gentianaceae) is an annually growing herb in bosk, meadow and damp sidehills at an altitude of 2500–4500 m in China, India, Nepal, Bhutan and Sikkim [1]. In China, it grows in the northwest region and is called 'ji he di' by local people. The whole herb is used for the treatment of inflammatory and gastrointestinal problems, such as gastroenteritis and diarrhoea [2,3]. In the previous studies, we have reported the isolation of fourteen compounds from the whole herb of *Gentianopsis paludosa* [4,5]. Here, we report the isolation and structural elucidation of a new compound, gentianopfluorenone, and three known compounds, 1-*O*-β-D-glucopyranosyl-5-hydroxy-3-methoxyxanthone (**2**) [6], 1-*O*-[β-D-xylopyranosyl-(1 → 6)-β-D-glucopyranosyl]-7,8-dihydroxy-3-methoxyxanthone (**3**) [7], and apigenin (**4**) [8] from further investigation of this plant.

2. Results and discussion

Compound **1** was obtained as yellow powder and showed a positive reaction with $FeCl_3$. Its molecular formula $C_{14}H_{10}O_6$ was determined by HREI-MS, which gave a molecular ion peak at *m*/*z* 274.0481 $[M]^+$. The IR spectrum suggested the presence of hydroxyl group

*Corresponding author. Email: duyg@dicp.ac.cn
§Email: whd77@163.com

Journal of Asian Natural Products Research
ISSN 1028-6020 print/ISSN 1477-2213 online © 2007 Taylor & Francis
http://www.tandf.co.uk/journals
DOI: 10.1080/10286020600727459

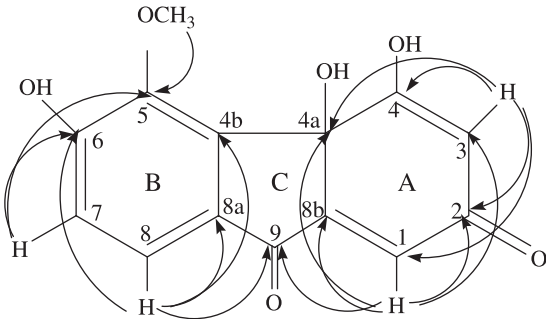


Figure 1. Main HMBC correlations of compound **1**.

Downloaded At: 19:10 22 January 2011

(3444 cm^{-1}), methyl (2929, 2854 cm^{-1}), conjugated carbonyl (1654 cm^{-1}) and aromatic ring (1600, 1580 cm^{-1}). In the ^1H NMR spectrum, two aromatic proton signals at δ 7.26 (1H, d, $J = 9.0$ Hz, H-7) and 7.08 (1H, d, $J = 9.0$ Hz, H-8), two olefinic proton signals at δ 5.89 (1H, s, H-1) and 5.79 (1H, s, H-3), and the methoxyl proton signal at δ 3.78 (3H, s, 5-OCH_3) were observed. The ^{13}C NMR spectrum suggested the presence of two carbonyls (δ 178.0, 173.5). In the COSY spectrum, the H-7 was correlated to H-8. In the HSQC spectrum, correlated peaks were observed between H-7 and C-7 (δ 122.7), H-8 and C-8 (δ 112.6), H-1 and C-1 (δ 94.6), H-3 and C-3 (δ 99.9), the methoxyl proton and the methoxyl carbon (δ 60.9). In the HMBC spectrum, the H-8 and H-1 all showed correlations with C-9 (δ 178.0), and H-1 and H-3 showed correlations with C-2 (δ 173.5) and C-4a (δ 99.6). Furthermore, the HMBC spectrum also showed that the proton of —OCH_3 correlated with C-5 (δ 145.2), indicating that the methoxyl group was located at C-5. Therefore, compound **1** was established as 4,4a,6-trihydroxy-5-methoxy-fluoren-2,9-dione, and was named gentianop-fluorenone. The main HMBC correlations are shown in figure 1.

3. Experimental

3.1 General experimental procedures

Melting points were determined on an X-4 micro-melting point apparatus and are uncorrected. NMR spectra were recorded on a Bruker DRX-400 spectrometer (TMS as internal standard). IR and UV spectra were recorded on Bruker IS-55 and Jenway 6505 UV/Vis spectrometers, respectively. APIES-MS and HREI-MS were obtained on HP 100 MSD and GCT-ES mass spectrometers, respectively. TLC employed precoated silica gel GF_{254} plates.

3.2 Plant material

The whole herb of *Gentianopsis paludosa* was collected in August 2004 from Qinghai province, China. The plant was identified by Professor Bao-Chen Zhang, Northwest Institute of Plateau Biology, Chinese Academy of Sciences. A voucher specimen (No. NPGR-04-08) of the plant is deposited in our laboratory.

3.3 Extraction and isolation

The air-dried plant material (1.0 kg) was crushed and extracted three times with refluxing 75% EtOH. The extract was filtered and concentrated under vacuum to dryness. The residue (237 g) was suspended in H_2O and partitioned with petroleum ether, CH_3Cl_3, and n-BuOH successively. The n-BuOH extract (60 g) was separated on a polyamide column (80–100 mesh) and eluted with H_2O, 30%, 50%, 70%, 95% EtOH successively to give fractions A–E. Fraction B was chromatographed on a silica gel column (200–300 mesh) eluted with EtOAc/MeOH (10:0 to 5:5) to give compounds **2** (4 mg) and **3** (32 mg). Fraction C was also chromatographed on a silica gel column (200–300 mesh) eluted with petroleum ether-EtOAc (10:1 to 1:1) to give subfractions a–d. Subfraction a was separated by preparative TLC using petroleum ether/EtOAc (2.5:1) to yield compound **1** (7 mg). Subfraction c was purified by recrystallisation from MeOH to yield compound **4** (5 mg).

3.3.1 Compound 1. Yellow powder (MeOH); mp 229–231°C; IR (KBr) ν_{max} (cm^{-1}): 3444, 2925, 2854, 1654, 1600, 1580, 1428, 1380, 1315, 1282, 1174, 1058, 819; UV λ_{max} (MeOH) (nm): 245, 255, 310, 355, 380; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.26 (1H, d, $J = 9.0$ Hz, H-7), 7.08 (1H, d, $J = 9.0$ Hz, H-8), 5.89 (1H, s, H-1), 5.79 (1H, s, H-3), 3.78 (3H, s, 5-OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 94.6 (C-1), 173.5 (2-C═O), 99.9 (C-3), 162.9 (C-4), 99.6 (C-4a), 146.7 (C-4b), 145.2 (C-5), 148.9 (C-6), 122.7 (C-7), 112.6 (C-8), 115.0 (C-8a), 157.2 (C-8b), 178.0 (9-C═O), 60.9 (5-OCH_3); APIES-MS (*m*/*z*): 273 $[M-1]^{-1}$; HREI-MS (*m*/*z*): 274.0481 $[M]^+$ (calcd for $C_{14}H_{10}O_6$, 274.0477).

Downloaded At: 19:10 22 January 2011

References

[1] S.W. Liu. *Flora Qinghaiica*, p. 75, Qinghai People's Publishing House, Xining (1996).
[2] B.Z. Guo. *Medicinal Plants of Qinghai Province*, p. 458, Qinghai People's Publishing House, Xining (1987).
[3] L.Y. Tu, C.N. Cao, Q.Y. Zhao, J. Qinghai. *Norm. Uni. (Nat. Sci.)*, **1**, 69 (2005).
[4] H.D. Wang, C.Y. Tan, X.F. Bai, Y.G. Du. *Chin. J. Chin. Mater. Med.*, **11**, 1055 (2004).
[5] H.D. Wang, C.Y. Tan, X.F. Bai, Y.G. Du, B.C. Lin. *Chin. Pharm. J.*, **14**, 1059 (2005).
[6] J.L. Wolfender, M. Hamburger, J.D. Msonthi, K. Hostettmann. *Phytochemistry*, **30**, 3625 (1991).
[7] L.J. Ji, J.Y. Ding, S.F. Fan, H.F. Sun. *Acta Bota. Sin.*, **34**, 203 (1992).
[8] W. Li, C. Chan, H. Yeung. *Pharm. Pharmacol. Commun.*, **4**, 415 (1998).