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A new naphthaquinone derivative from *Chirita eburnea*

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A new compound, methyl 3-(4'-hydroxyphenethylamino)-1,4-dihydro-1,4-dioxonaphthalene-2-carboxylate (**1**) was isolated from *Chirita eburnea*. Its structure was elucidated on the basis of 1D NMR, 2D NMR and MS analysis.

Keywords: Methyl 3-(4'-hydroxyphenethylamino)-1,4-dihydro-1,4-dioxonaphthalene-2-carboxylate; Naphthaquinone; *Chirita eburnea*; Gesneriaceae

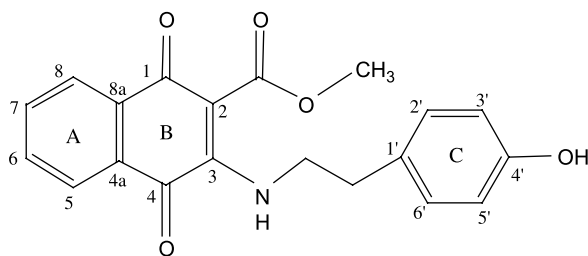
1. Introduction

Chirita eburnea Hance, belonging to the family Gesneriaceae, is distributed in Yunnan, Guangxi, Guangdong and Sichuan Provinces. It has been used as traditional Chinese medicine to treat cough with bleeding and other weak diseases [1]. Inoue *et al.* have extracted α,β -dunnione from *Streptocarpus dunnii* [2–4]. Many anthraquinones have also been isolated from other genera in Gesneriaceae. In this paper we report isolation of a new naphthaquinone derivative, named methyl 3-(4'-hydroxyphenethylamino)-1,4-dihydro-1,4-dioxonaphthalene-2-carboxylate; its structure was elucidated by means of 1D NMR, 2D NMR and HRESI-MS analysis.

2. Results and discussion

Compound **1** was deduced to have a molecular formula of $C_{20}H_{17}O_5N$ by HRESI-MS m/z 352.1175 ($[M + 1]^+$), in combination with 1H NMR, ^{13}C NMR and MS spectra. In its ^{13}C NMR spectrum, the signals of two conjugate ketone [δ_C 178.9 (s), 178.9 (s)] and eight phenyl carbons [δ_C 132.2 (s), 126.1 (d), 135.2 (d), 132.8 (d), 125.4 (d), 132.2 (s), 129.6 (s), 129.3 (s)] suggested a naphthaquinone skeleton [2]. In the 1H NMR and ^{13}C NMR spectra, four phenyl carbons [δ_C 126.1 (d), 135.2 (d), 132.8 (d), 125.4 (d)] and corresponding protons [δ_H 7.98 (d, 7.0 Hz) 7.75 (t, 7.0 Hz) 7.85 (t, 7.0 Hz) 7.93 (d, 7.0 Hz)] assumed the absence of

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Figure 1. Structure of **1**.

substituting groups on the benzene ring (A ring), which was also supported by its HMBC spectrum. Four downfield carbons were attributed to two phenyl signals [δ_C 132.2 (s), 132.2 (s)], and two substituted olefinic carbons [δ_C 129.6 (s), 129.3 (s)] by its HMBC data. A methylate ester was positioned at C-2 by the correlation between OCH₃ (δ_H 3.73) and —CO— (δ_C 167.6) in the HMBC spectrum. In the ¹H NMR and ¹³C NMR spectra, signals at [δ_C 128.1 (s), 155.9 (s), 129.3 (d), 115.2 (d)], and corresponding protons [δ_H 7.00 (2H, d, 8.0 Hz), 6.68 (2H, d, 8.0 Hz)] indicated a typical *p*-substituted benzene moiety. 4'-Hydroxylphenethylamino part was proposed based on the correlations between OH (δ_H 9.28) and C-3', 5' (δ_C 115.2) and C-4' (δ_C 155.9); δ_H 2.75 (t, 7, CH₂) and CH₂ (δ_C 33.6) and C-1' (δ_C 128.1). Therefore, this structure was deduced to be methyl 3-(4'-hydroxyphenethylamino)-1,4-dihydro-1,4-dioxonaphthalene-2-carboxylate (see figures 1 and 2).

3. Experimental

3.1 General experimental procedures

NMR spectra were run on a Bruker DRX-500 (500 MHz for ¹H NMR and 2D NMR, 125 MHz for ¹³C NMR) instrument with TMS as internal standard; IR spectra were measured on a Bio-Rad FTS-135 spectrometer with KBr pellets. ESI-MS spectra and EI-MS spectra were recorded on a VG Auto Spec-3000 spectrometer. UV spectra were obtained on a Shimadzu double-beam 210A spectrophotometer. EI-MS: 70 eV; Silica gel (200–300 mesh).

3.2 Plant material

The whole plant of *Chirita eburnea* was collected and identified by Dr De-Shan Deng in August 2002 in Guangxi Province, China. A voucher specimen is deposited in herbarium of the Department of Taxonomy, Kunming Institute of Botany, Academia Sinica, Kunming, China.

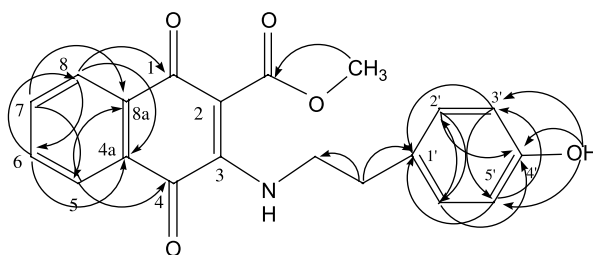
Figure 2. Selected HMBC correlations of **1**.

Table 1. NMR spectral data of compound **1** (500 MHz for ^1H NMR and 125 MHz for ^{13}C NMR, in DMSO, J in Hz).

No.	δ_{H} (Hz)	δ_{C}	HMBC
1		178.9 s	
2		129.6 s	
3		129.3 s	
4		180.9 s	
4a		132.2 s	
5	7.98 (d, 7.0)	126.1 d	C-4, C-7, C-8a
6	7.75 (t, 7.0)	135.2 d	C-8, C-4a
7	7.85 (t, 7.0)	132.8 d	C-5, C-8a
8	7.93 (d, 7.0)	125.4 d	C-1, C-6, C-4a
8a		132.2 s	
COOCH ₃		167.6 s	
OCH ₃	3.73 (s)	51.8 q	—CO
—NH—CH ₂	3.25 (Br. s)	44.6 t	
—CH ₂ —	2.75 (t, 7)	33.6 t	—NH—CH ₂ , C-1'
1'		128.1 s	
2',6'	7.00 (d, 8.0)	129.3 d	C-4', 2', 6'
3',5'	6.68 (d, 8.0)	115.2 d	C-1', 3', 5'
4'		155.9 s	
OH	9.35 (s)		C-3', 4', 5'

3.3 Extraction and isolation

Dried whole plant (4.0 kg) was crushed and extracted with MeOH at room temperature. After removal of the MeOH under reduced pressure, the viscous concentration was partitioned between H₂O and EtOAc. Column chromatography of the EtOAc layer on silica gel, eluting with a gradient mixture of CHCl₃/CH₃COCH₃, gave 10 fractions (I–X). Fraction VI was chromatographed over silica gel, eluting with CHCl₃/CH₃COCH₃ 4:1 to give compound **1** (100 mg).

Compound **1**: C₂₀H₁₇O₅N, yellow crystals, mp 200–202°C, UV (MeOH) λ_{max} (log ϵ): 226 (3.83), 271 (3.43), 442 (2.32) nm; IR (KBr) ν_{max} (cm⁻¹): 3409 (—OH), 3298 (—NH), 1710 and 1689 (—C=O), 1602, 1565, 1515, 1459, 721. ESI-MS m/z 374 [M + Na]⁺ (100); EI-MS (70 eV) m/z 351 [M]⁺ (20), 317 (15), 245 (50), 212 (100), 107 (50). HRESI-MS m/z 352.1175 [M + 1]⁺ (calcd for C₂₀H₁₈O₅N: 352.1184). ^1H NMR, ^{13}C NMR and HMBC spectral data: see table 1.

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