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A Novel Metabolite of *Cinnamomum burmani*

Yang TL[1], Yeh HC[2], Li HT[3], Liu SL[4] and Chen CY[2]*

[1]*Department of Clinical Microbiology Laboratory, Yuan's General Hospital, Kaohsiung, Taiwan.*

[2]*School of Medical and Health Sciences, Fooyin University, 83102, Kaohsiung, Taiwan*

[3]*Department of Medical Laboratory Science and Biotechnology, Fooyin University, Kaohsiung 83102, Taiwan.*

[4]*Experimental Forest College of Bioresources and Agriculture, National Taiwan University, No.12, Sec. 1, Qianshan Rd., Zhushan Township, Nantou County, Taiwan (R.O.C.).*

***Correspondence:**
Chen CY, School of Medical and Health Sciences, Fooyin University, 83102, Kaohsiung, Taiwan.

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ABSTRACT

A new metabolite, burmanoside (1) was isolated from the plant flowers of Cinnamomum burmani (Nees & T. Nees) Blume (Lauraceae). The structure of 1 as a novel dioxane and tetrahydro-2H-pyran-3,4,5-triol skeleton. This structure was elucidated on the basis of physical and spectral analysis.

Keywords

Cinnamomum burmani, burmanoside, plant flowers, dioxane, tetrahydro-2*H*-pyran-3,4,5-triol.

Introduction

Cinnamomum burmani (Nees & T. Nees) Blume (*Lauraceae*) is a source of Indonesia cinnamon, and is widely used as a spice in Indonesia [1]. The chemical constituents of the roots of this plant have not yet been reported. Recently, we reported a new amide, a novel homosesquiterpenoid, along with five known amides from the stems of *C. burmani* [2,3]. Previously, we isolated 20 compounds, including one apocarotenoid, one triterpenoid, one coumarin, two steroids, and four benzenoids from the leaves of this plant [4,5]. In the course of screening for biologically and chemically novel agents from Formosan plants in the family Lauraceae [6-91], *C. burmani* was chosen for further phytochemical investigation. In this paper, we report the isolation and structural elucidation of this new metabolite (burmanoside (**1**)).

Compound **1**, obtained as colorless crystals, had the molecular formula $C_{14}H_{26}O_{11}$, as determined by HR-ESI-MS data (*m/z* 393.1377 ($[M+Na]^+$; calc. 393.1373)) in combination with its ^{1}H-NMR, ^{13}C-NMR and DEPT, requiring 2 degree of unsaturation. The IR spectrum show absorptions for hydroxyl group (3400 cm^{-1}). The ^{1}H- and ^{13}C-NMR spectra indicated ten OCH, two CH_2, and two Me groups. In the ^{1}H-NMR spectrum, there were typical signals for two OMe groups at δ_H 3.44 (3H, s, OMe-14), and 3.58 (3H, s, OMe-15), two hydroxymethyl moieties at δ_H 4.37 (2H, d, J = 10.0 Hz, H-6) & 4.38 (2H, d, J = 10.8 Hz, H-13), ten oxymethines signals overlapping between δ_H 4.01~5.16 corroborated by ^{13}C-NMR signals in Table 1 and were clarified and assigned based on COSY, NOESY (Figure 1), HSQC and HMBC (Table 1) spectra to confirm the existence of one dioxane and tetrahydro-2*H*-pyran-3,4,5-triol skeleton (Table 1). The HMBC correlations of H-4 (δ_H 4.39) to C-8 (δ_C 70.7), and H-8 (δ_H 4.52) to C-4 (δ_C 70.2) (Table 1) showed that the two moieties were bonded to C-4 and 8 by C-7, respectively. Two methoxyl groups δ_H 3.44 and 3.58 located at the C-1 and C-2 were determined by ^{13}C-NMR signals (δ 106.0 and 101.4) and the HMBC correlation of OMe-14/C-1 and OMe-15/C-2. The attachment of two OMe substituents at C-1 and C-2 were confirmed by the correlation between OMe-14/H-1, OMe-15/H-2 and OMe-14/OMe-15 in NOESY spectrum. The full assignment of **1** was further confirmed by COSY, HSQC, and HMBC spectra.

The assignments were further verified by significant NOE correlations (Figure 1) and further supported the relative configuration of each substituents on the **1**. The ^{1}H- and ^{13}C-NMR (Table 1), COSY,

NOESY (Figure 1), HSQC and HMBC (Table 1) experiments confirmed the structure as ((8*S**,9*R**,10*S**,11*R**,12*S**)-8-((*S**)-hydroxy((1*S**,2*R**,4*S**,5*R**)-4- (hydroxymethyl)-1,2-dimethoxy-3,6-dioxan-5-yl)methyl)-12-(hydroxymethyl)tetrahydro-2*H*-pyran-9,10,11-triol and designated as burmanoside (**1**).

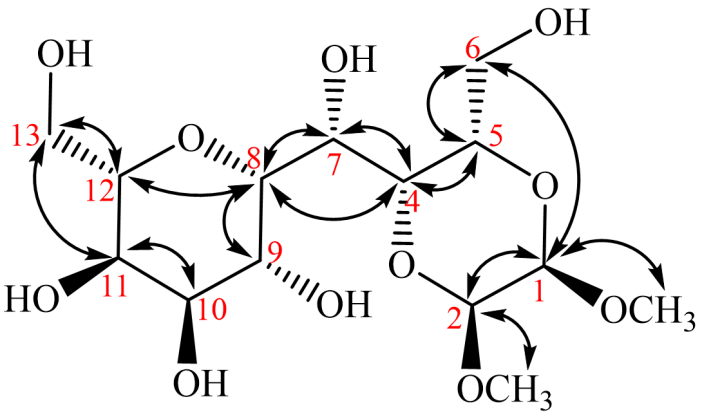

Figure 1: Structure of compound **1** & major NOESY (H↔H) correlations of **1**.

Table 1: ^{13}C (Py-d_5) and ^{1}H HMR (Py-d_5) data of **1**.

No	δ_C	δ_H	*mult., J (Hz)*	*HMBC ($^1H \rightarrow {}^{13}C$)*
1	106.0	5.16	d, 4.0	C-2, C-14
2	101.4	4.63	d, 4.0	C-1, C-15
4	70.2	4.39	t, 6.8	C-5, C-6, C-7, C-8
5	72.2	4.26	td, 10.0, 6.8	C-4, C-6, C-7
6	62.4	4.37	d, 10.0	C-4, C-5
7	70.0	4.47	d, 6.8	C-4, C-5, C-8, C-9
8	70.7	4.52	d, 6.8	C-4, C-7, C-9, C-10
9	71.4	4.48	d, 6.8	C-7, C-8, C-10, C-11
10	72.3	4.41	d, 6.8	C-8, C-9, C-11, C-12
11	76.6	4.01	t-like, 6.8	C-9, C-10, C-12, C-13
12	75.0	4.22	td, 10.8, 6.8	C-10, C-11, C-13
13	62.1	4.38	d, 10.8	C-11, C-12
14	55.0	3.44	s	C-1
15	56.5	3.58	s	C-2

Experimental

General

IR spectra were measured on a Hitachi 260-30 spectrophotometer. ^{1}H NMR (400 MHz, Py-d_5) and 2D spectra were obtained on Varian-Mercury-400 spectrometers. Low-resolution ESI-MS spectra were obtained on an API 3000 (Applied Biosystems) and high-resolution ESI-MS spectra on a Bruker Daltonics APEX II 30e spectrometer. Silica gel 60 (Merck, 70~230 mesh, 230~400 mesh) was used for column chromatography. Precoated Silica gel plates (Merck, Kieselgel 60 F-254), 0.20 mm and 0.50 mm, were used for analytical TLC and preparative TLC, respectively, visualized with 50% H_2SO_4.

Plant Material

The flowers of *C. burmani* (Nees & T. Nees) Blume were collected from Nantou County, Taiwan, in June 2020. Plant material was identified by Dr. Su-Ling Liu (Experimental Forest College of Bioresources and Agriculture, National Taiwan University). A voucher specimen was deposited at the Department of Medical Technology, School of Medical and Health Sciences, Fooyin University, Kaohsiung, Taiwan.

Extraction and Isolation

The plant flowers (304 g) of *C. burmani* were airdried and extracted repeatedly with MeOH (2 L × 5) at room temperature. The combined MeOH extracts (22.4 g) were then evaporated and further separated into 6 fractions by column chromatography on silica gel (4.5 kg, 70-230 mesh) with gradients of *n*-hexane/CH_2Cl_2/acetone/ MeOH. Part of fraction 1 (3.5 g) was subjected to silica gel chromatography by eluting with *n*-hexane–acetone (70:1) enriched with acetone to furnish three further fractions (1-1–1-3). Fraction 1-1 (2.8 g) was further purified on a silica gel column using *n*-hexane–acetone mixtures to obtain 5-hydroxy-4,7-dimethoxyflavonoid (13 mg). Part of fraction 1-3 (2.3 g) was subjected to silica gel chromatography by eluting with *n*-hexane–acetone (50:1) enriched gradually with acetone to furnish five fractions (1-3-1–1-3-5). Fraction 1-3-2 (0.9 g) was further purified on a silica gel column using *n*-hexane–acetone mixtures to yield luteolin-7,3,4-trimethyl ether (4 mg) and 5,3-dihydroxy-7,4-dimethoxy- flavone (2 mg). Part of fraction 2 (2.1 g) was subjected to silica gel chromatography by eluting with *n*-hexane–acetone (40:1) enriched with acetone to furnish two further fractions (2-1–2-2). Fraction 2-1 (3.4 g) was further purified on a silica gel column using *n*-hexane–acetone mixtures to obtain persicogenin (11 mg) and genkwanin (14 mg). Part of fraction 3 (2.8 g) was subjected to silica gel chromatography by eluting with CH_2Cl_2–MeOH (100:1) enriched with MeOH to furnish five fractions (3-1–3-5). Fraction 3-3 (1.4 g) was further purified on a silica gel column using CH_2Cl_2–MeOH mixtures to obtain apigenin (7 mg) and luteolin (11 mg). Fraction 5 (3.8 g) eluted with *n*-hexane–EtOAc (1:1) was repeatedly subjected to silica gel CC and gave kaempferol (12 mg) and quercetin (16 mg). Part of fraction 6 (7.6 g) was subjected to silica gel chromatography by eluting with EtOAc-MeOH (20:1), enriched with MeOH to furnish five further fractions (6-1–6-5). Fraction 6-1 (3.2 g) was further purified on a silica gel column using *n*-hexane/acetone mixtures to obtain kaempferol 3-*O*-L-rhamnopyranoside (12 mg) and quercetin 3-*O*-L-rhamnopyranoside (9 mg). Fraction 6-3 (1.6 g) was further purified on a silica gel column using *n*-hexane/acetone mixtures to obtain burmanoside (**1**) (13 mg).

Burmanoside (1)

Colorless crystals. mp 194-196 °C. $[\alpha]^{25}_{D}$ + 12.5° (c 0.55, MeOH). IR (KBr, ν_{max}, cm^{-1}) ν_{max}: 3400 (OH) cm^{-1}. HR-EI-MS-: *m/z* $[M+Na]^+$ calcd for $C_{14}H_{26}O_{11}Na$: 393.1373; found: 393.1377. ^{1}H and ^{13}C NMR (400 MHz, Py-d_5, δ, ppm, J/Hz): see Table 1.

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