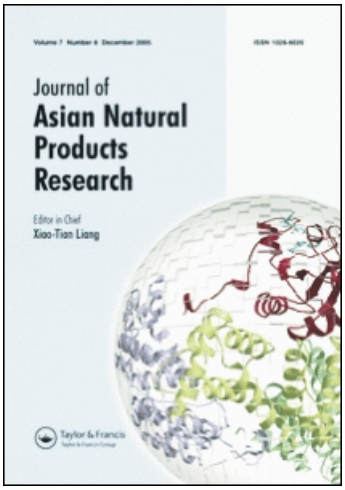

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Coumarins from the leaves of *Bambusa pervariabilis* McClure

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NOTE

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A new pyrone-coumarin, 7,8-dihydroxy-3-(3-hydroxy-4-oxo-4*H*-pyran-2-yl)-2*H*-chromen-2-one (**1**), along with two known coumarins, scopoletin (**2**) and scopolin (**3**), was isolated from the 95% EtOH extract of the leaves of *Bambusa pervariabilis* McClure. Their structures were determined on the basis of spectroscopic techniques and chemical methods.

Keywords: *Bambusa pervariabilis* McClure; coumarin; 7,8-dihydroxy-3-(3-hydroxy-4-oxo-4*H*-pyran-2-yl)-2*H*-chromen-2-one

1. Introduction

Bamboo comprises over 1300 species, and more than 500 species have been found in China. Chinese people realized the medical and health-care effects of bamboo leaf long ago, and used it or its extract as a traditional Chinese medicine and food additive. *Bambusa pervariabilis* McClure is one of the bamboo species found in China. Chinese people used it as a traditional medicine for the treatment of febrile disorder, exogenous diseases, cooling blood, and hemostasis [1]. Previous phytochemical research on bamboo leaves showed the presence of flavonoid, coumarin, and phenolic acids [2–4]. Coumarin, an important active component in plants, has various medical activities [5] such as anti-AIDS [6–9], anti-oxidant activities [10,11], and different types of cancer-preventive activity [12]. Extensive chromatography of the EtOH extract of the leaves of *B. pervariabilis* McClure had led to the isolation of a new pyrone-coumarin, and two known coumarins were reported from this species for the first time. This paper deals with the isolation and structural elucidation of the new pyrone-coumarin **1** (Figure 1).

2. Results and discussion

Compound **1** was obtained as a yellow amorphous powder, mp 278.6–279.8°C. The molecular formula, $C_{14}H_8O_7$, was deduced from the positive HR-ESI-MS at *m/z* 311.0158 $[M+Na]^+$. The ESI-MS (positive) showed ion peaks at *m/z* 289.3 $[M+H]^+$ and 311.3 $[M+Na]^+$. The IR spectrum displayed characteristic absorption bands for hydroxyl (3456 cm^{-1}), carbonyl (1744 and 1650 cm^{-1}) groups, and aromatic rings (1575 and 1541 cm^{-1}). The UV spectrum showed absorption maxima at 230, 277, and 311 nm, characteristic of the coumarin.

The ^1H NMR spectrum (Table 1) indicated the presence of five proton signals at δ_H 7.23 (s, 1H), 8.27 (d,

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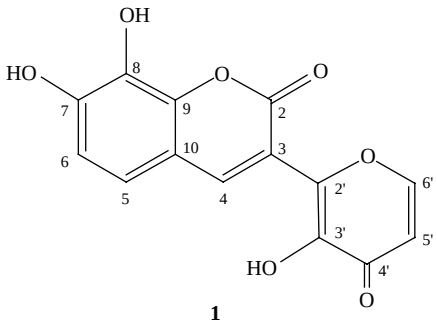

1

Figure 1. Structure of compound **1**.

$J = 5.4$ Hz, 1H), 7.02 (d, $J = 8.4$ Hz, 1H), 6.62 (d, $J = 5.4$ Hz, 1H), 6.70 (d, $J = 8.4$ Hz, 1H), and three hydroxyl groups at δ 10.30 (br s, 1H), 9.68 (br s, 1H), and 12.80 (br s, 1H). The ^{13}C NMR spectrum (Table 1) showed 14 carbon signals, including two carbonyl signals at δ_C 173.3 and 162.3, and six aromatic carbons at δ_C 122.7, 117.7, 147.4, 144.8, 146.4, and 116.3. In the ^{1}H NMR spectrum, two proton signals at δ 6.62 (d, $J = 5.4$ Hz, 1H) and 8.27 (d, $J = 5.4$ Hz, 1H) indicated the existence of a *cis*-configured double bond, and the ^{13}C NMR spectrum showed one carbonyl signal at δ 173.3 and four carbon signals at δ 156.8, 155.8 144.5, and 116.3. All these data were similar to the corresponding data of maltol [13,14]. In the HMBC spectrum, the correlations of H-6′ at δ 8.27 (d, $J = 5.4$ Hz, 1H) with C-4′ at δ 173.3 and H-5′ at δ 6.62 (d, $J = 5.4$ Hz, 1H) with C-3′ at δ 144.5 suggested that a hydroxypyrone moiety existed in **1**. Additionally, a proton signal at δ 7.23 (s, 1H) and two anomeric proton signals at δ 7.02 (d, $J = 8.4$ Hz, 1H) and 6.70 (d, $J = 8.4$ Hz, 1H) in the ^{1}H NMR spectrum and nine carbon signals at δ 162.3, 123.9, 124.7, 122.7, 117.7, 147.4, 144.8, 146.4, and 116.3 in the ^{13}C NMR spectrum probably suggested the presence of a coumarin skeleton. The ^{1}H and ^{13}C NMR spectroscopic data of **1** were similar to the corresponding data of scopoletin [15,16], except for additional hydroxypyrone and hydroxyl groups. This was confirmed by further analysis of its HMBC and ^{1}H–^{1}H COSY spectra. The correlation of H-5 at δ 7.02 (d, $J = 8.4$ Hz, 1H) with H-6 at δ 6.70 (d, $J = 8.4$ Hz, 1H) was shown in the ^{1}H–^{1}H COSY spectrum (Table 1), while the HMBC spectrum showed the correlations between H-4 at δ 7.23 (s, 1H) and C-5 at δ 122.7, C-9 at δ 146.4, C-2 at δ

Table 1. ^{1}H and ^{13}C NMR spectral data for compound **1** in DMSO-d_6[a].

No.	δ_H	δ_{OH}	δ_C	DEPT	^{1}H–^{1}H COSY
2			162.3	C	
3			123.9	C	
4	7.23 (s)		124.7	CH	
5	6.70 (d, $J = 8.4$, 1H)		122.7	CH	H-6
6	7.02 (d, $J = 8.4$, 1H)		117.7	CH	H-5
7			147.4	C	
7-OH		10.30 (br s, 1H)			
8			144.8	C	
8-OH		9.68 (br s, 1H)		C	
9			146.4	C	
10			116.3	C	
2′			155.8	C	
3′			144.5	C	
3′-OH		12.80 (br s, 1H)			
4′			173.3	C	
5′	6.62 (d, $J = 5.4$, 1H)		116.3	CH	H-6′
6′	8.27 (d, $J = 5.4$, 1H)		156.8	CH	H-5′

Note: [a] The ^{1}H and ^{13}C NMR spectral data were measured at 600 MHz, and the J values are given in Hz.

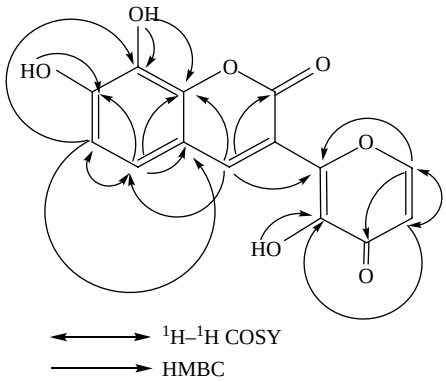

Figure 2. Significant HMBC and ^{1}H–^{1}H COSY correlations of compound **1**.

162.3, C-2′ at δ 155.8, H-6 at δ 7.02 (d, $J = 8.4$ Hz, 1H) and C-8 at δ 144.8, C-10 at δ 116.3, and H-5 at δ 6.70 (d, $J = 8.4$ Hz, 1H) and C-7 at δ 147.4, C-9 at δ 146.4. In addition, the key correlations between hydroxyl 8-OH at δ 9.68 (br s, 1H) and C-8 at δ 144.8, 7-OH at δ 10.30 (br s, 1H) and C-7 at δ 147.4 were observed in the HMBC spectrum (Figure 2). Furthermore, the correlation of H-4 at δ 7.23 (s) with C-2′ at δ 155.8 in the HMBC spectrum identified that C-3 of the coumarin section was connected with C-2′ of the hydroxypyrone moiety by the C—C bond. Finally, the structure of **1** was elucidated as 7,8-dihydroxy-3-(3-hydroxy-4-oxo-4*H*-pyran-2-yl)-2*H*-chromen-2-one.

Additionally, the two known compounds were identified by spectroscopic methods as scopoletin (**2**) [15,16] and scopolin (**3**) [17].

3. Experimental

3.1 General experimental procedures

Melting point was determined with Shenguang WRX-1S thermal values analyzer with microscope and is uncorrected. UV spectra were obtained on Waters 2695 HPLC with a photodiode array detector. IR spectra were taken on a Thermo Nicolet FT-IR NEXUS 670 spectrophotometer with KBr pellets. NMR spectra were recorded on Varian System-600 and Bruker System-300. HR-ESI-MS were performed on an AutoSpec Ultima-TOF mass spectrometer and ESI-MS data were obtained with an Agilent 1100 Series mass spectrometer.

3.2 Plant material

The leaves of *B. pervariabilis* McClure were collected from Nanning City, Guangxi Province, China in September 2008, and identified by Prof. Dayong Huang, Bamboo Research Institute, Nanning Academy of Forestry, Nanning, China. A voucher specimen (No. 200810-01) is deposited at the International Centre for Bamboo and Rattan (ICBR), Beijing, China.

3.3 Extraction and isolation

The shade-dried leaves of *B. pervariabilis* McClure (8.24 kg) were extracted with 95% EtOH by cold percolation for three times. A residue of 765.3 g was obtained after the removal of the solvent by evaporation. The residue was suspended in H_2O and extracted with petroleum ether. The fraction after being extracted with petroleum ether was subjected to macroporous absorption resin (AB-8) and eluted with H_2O, 20% EtOH, 40% EtOH, 60% EtOH, 80% EtOH, and acetone. The 60% EtOH fraction (42.9 g) was then chromatographed over Sephadex LH-20 and eluted with MeOH repeatedly, to yield compounds **1** (11.3 mg), **2** (18.4 mg), and **3** (13.2 mg).

3.3.1 7,8-Dihydroxy-3-(3-hydroxy-4-oxo-4H-pyran-2-yl)-2H-chromen-2-one (1)

Yellow amorphous powder (MeOH), mp 278.6–279.8°C; UV λ_{max} (nm): 230, 277, 311; FT-IR (KBr) γ_{max} (cm^{-1}): 3456, 1744, 1650, 1575, 1541, 1319, 1281, 843, 825; ^{1}H and ^{13}C NMR spectral data (see Table 1); HR-ESI-MS: *m/z* 311.0158 $[M+Na]^+$ (calcd for $C_{14}H_8NaO_7^+$

311.0168); positive ESI-MS: *m/z* 289.3 $[M+H]^+$, 311.3 $[M+Na]^+$.

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