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Journal of Asian Natural Products Research

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713454007>

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To cite this Article Liu, Mei-Feng , Han, Yun , Xing, Dong-Ming , Shi, Yue , Xu, Li-Zhen , Du, Li-Jun and Ding, Yi(2004) 'A new stilbenoid from *Arundina graminifolia*', Journal of Asian Natural Products Research, 6: 3, 229 – 232

To link to this Article: DOI: 10.1080/10286020310001653219

URL: <http://dx.doi.org/10.1080/10286020310001653219>

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A NEW STILBENOID FROM *ARUNDINA GRAMNIFOLIA*

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(Received 28 July 2003; Revised 23 September 2003; In final form 30 September 2003)

A chemical investigation of the Orchidaceae *Arundina grammifolia* has led to the isolation of a novel stilbenoid, named arundinan (**1**). The structure of **1** has been elucidated as 2-(*p*-hydroxybenzyl)-3-hydroxy-5-methoxybibenzyl on the basis of physical and chemical evidence and spectral analysis.

Keywords: *Arundina grammifolia*; Orchidaceae; Stilbenoid; Arundinan

INTRODUCTION

Arundina grammifolia is a terrestrial plant belonging to the Orchidaceae. The genus is considered to possess activities of detoxification, antiarthritis and abirritation and is used as antidote and demulcent [1]. Stilbenoids are the major components in this plant [2,3], as well as triterpenes [4,5]. In a chemical investigation of medicinal plants, the ethanol extract of *Arundina grammifolia* was found to possess the function of regulating immunity. The ethanol extract was further fractionated into light petroleum, ethyl acetate, acetone and methanol parts. Further chemical investigation of the ethyl acetate part resulted in the isolation of a novel stilbenoid designated as arundinan (**1**). Its structure was established as 2-(*p*-hydroxybenzyl)-3-hydroxy-5-methoxybibenzyl from the following spectral and chemical evidence.

RESULTS AND DISCUSSION

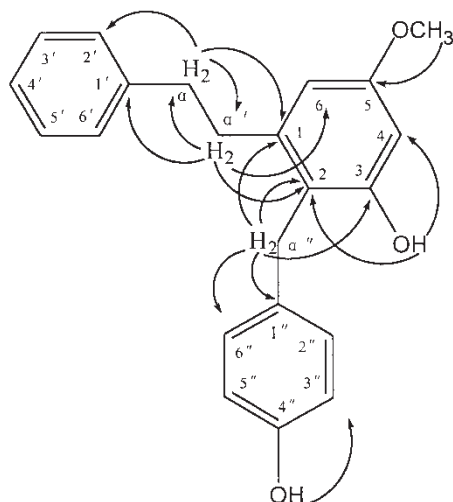
Arundinan (**1**) was obtained as a pink amorphous powder, mp. 152–154°C. Its UV spectrum exhibits absorptions typical of the bibenzyl chromophore at λ_{\max} 212, and 277 nm. Its IR spectrum showed hydroxyl (3322 cm⁻¹) and aromatic groups (1617, 1594, 1511 cm⁻¹). The phenolic nature of the compound was also indicated by its characteristic color reactions [FeCl₃: violet; phosphomolybdic acid: deep blue]. A molecular formula of C₂₂H₂₂O₃ was

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TABLE I ^1H and ^{13}C NMR data of **1** in CD_3COCD_3 (500 MHz for ^1H and 125 MHz for ^{13}C , δ in ppm, J in Hz)

No.	^1H	^{13}C	HMBC (^1H to ^{13}C)
1		143.5	
2		119.1	
3		157.0	
4	6.39(H, d, $J = 2.5$)	100.1	C-2, 3, 5, 6
5		159.6	
6	6.35(H, d, $J = 2.5$)	107.0	C-2, 4, 5, α'
1'		143.0	
2'(6')	7.14	129.2	C- α , 4', 6'(2')
3'(5')	7.23 (2H, t, $J = 7.5$)	129.0	C-1', 2'(6'), 5'(3')
4'	7.14	126.6	C-2'(6')
1''		133.5	
2'' (6'')	6.99 (2H, d, $J = 8.5$)	129.9	C-3''(5''), 4'', 6'' (2'')
3''(5'')	6.68 (2H, d, $J = 8.5$)	115.6	C-1'', 4'', 5'' (3'')
4''		156.1	
α	2.68 (2H, m)	38.1	C- α' , 1, 2'(6')
α'	2.79 (2H, m)	36.3	C-1', α , 2, 6
α''	3.93 (2H, s)	30.1	C-1, 2, 3, 1'', 2''(6'')
OCH_3	3.69 (3H, s)	55.2	C-5
3-OH	8.23 (s)		C-2, 4
4''-OH	7.99 (s)		C-3''(5'')

determined on the basis of the molecular ion of HR-MS at m/z 335.1632 ($[\text{M} + \text{H}]^+$) with significant fragment ion peaks at m/z 241, 229, 137, 107, 93 and 91. The ^1H NMR spectrum (Table I) of **1** showed signals for two phenolic hydroxyl protons at δ 7.99 and 8.23 and two doublets at δ 6.99 (2H, d, $J = 8.5$ Hz) and 6.69 (2H, d, $J = 8.5$ Hz) due to a pair of protons having an A_2B_2 system, which is characteristic of a *p*-substituted aromatic ring. In addition, the ^1H NMR spectrum exhibits a signal at δ 3.93 (2H, s), which is attributed to benzylic methylene. Furthermore, signals are present for the seven aromatic protons of bibenzyl, five of them at δ 7.23 (2H, t, $J = 7.5$ Hz) and 7.14 (3H, m), assignable to H-3', H-5', H-2', H-4' and H-6' of one aromatic ring from their chemical shifts and splitting patterns; the remaining two appear as a pair of doublets at δ 6.39 (H, d, $J = 2.5$ Hz) and 6.36 (H, d, $J = 2.5$ Hz), corresponding to two *meta*-coupled protons of the other aromatic ring. In addition, the ^1H NMR spectrum showed that **1** consists a methoxyl group at δ 3.69 as one singlet, and two methylenes of the bibenzyl appear at δ 2.77–2.86 (2H) and 2.65–2.69 (2H) as two multiplets. Its ^{13}C NMR spectrum (Table I) combined with DEPT spectra showed signals for three ethylenes, one methoxyl and 18 aromatic carbons (δ 100.0–160.0) of which 11 aromatic carbons are protonated; the other seven are quaternary (δ 159.6, 157.0, 156.1, 143.5, 143.0, 133.5, 119.1) and three are oxygen-bearing carbons at δ 159.6, 157.0, 156.1. All of the signals were assigned with the assistance of the HMQC spectrum. The structure of **1** was further confirmed by its HMBC spectrum. Cross peaks are observed for δ 6.39 (H-4)/119.1 (C-2), 157.0 (C-3), 159.6 (C-5), and 106.9 (C-6); δ 6.36 (H-6)/119.1 (C-2), 100.1 (C-4), δ 36.3 (C- α') and 159.6 (C-5); δ 7.14 (H-2' or H-6')/126.6 (C-4'), 38.1 (C- α), 129.2 (C-6' or C-2'); δ 7.23 (H-3' or 5')/129.2 (C-2' or 6'), 143.0 (C-1'), 129.0 (C-5' or 3'); δ 2.68 (H- α)/143.5 (C-1), 129.2 (C-2', 6') and 36.3 (C- α'); δ 2.79 (H- α')/143.0 (C-1'), 106.9 (C-6), 119.1 (C-2) and 38.1 (C- α); δ 3.93 (H- α'')/143.5 (C-1), 119.1 (C-2), 157.0 (C-3), 133.5 (C-1'') and 129.9 (C-2'' and C-6''); δ 8.23 (3-OH)/119.1 (C-2) and 100.1 (C-4); δ 7.99 (4''-OH)/115.6 (C-3'' and C-5''); and δ 3.69 (OCH_3)/159.6 (C-5) (Fig. 1). On basis of the above spectral evidence, the structure of the new compound (**1**) was established as 2-(*p*-hydroxybenzyl)-3-hydroxy-5-methoxybibenzyl.

FIGURE 1 Important HMBC correlations for **1**.

EXPERIMENTAL

General Experimental Procedures

Melting points were determined on an X-4 micro-melting point apparatus and are uncorrected. UV spectra were measured on a TU-1800PC spectrophotometer. IR spectra were recorded in KBr pellets on a Perkin-Elmer GX-FTIR spectrophotometer. HR-FABMS were obtained on an APEXII mass spectrometer. NMR spectra were determined on a Bruker AM500 instrument, using TMS as internal standard. Column chromatography was performed on silica gel (Qingdao Haiyang Chemical Group Co., Qingdao, China); TLC was conducted on Silica gel 60 F₂₅₄ (Merck Co., Germany) and monitored at 254 nm. ODS SP-120-ODS-BP and Sephadex LH-20 were purchased from Beijing Jinouya Technology and Development Co.

Plant Material

Rhizomes of *Arundina graminifolia* were collected from Yunnan Province in April 2002 and were identified by Professor Yang Cong-ren of the Kunming Institute of Botany, Chinese Academy of Sciences. A voucher specimen (02041) of the plant has been deposited in Laboratory of Pharmacy and Pharmacology, Department of Biological Science and Biotechnology, Tsinghua University.

Extraction and Isolation

The air-dried powder of rhizomes of *Arundina graminifolia* (4 kg) was refluxed with 95% EtOH (3 ×). The EtOH extract was concentrated under reduced pressure to 200 mL and mixed with silica gel (1 kg), then eluted with light petroleum, ethyl acetate, acetone and methanol sequentially. The ethyl acetate extract (70 g) was fractionated on a silica-gel column eluted with gradients of ethyl acetate in light petroleum to give 340 fractions. The combined Fr 112–125 (5 g) was then subjected to Sephadex LH-20 column chromatography and eluted with acetone to give fractions A and B; fraction A (1 g) was then subjected to ODS column chromatography, eluting with MeOH–H₂O (1:1), to give arundinan (**1**).

Characterization of **1**

2-(*p*-Hydroxybenzyl)-3-hydroxy-5-methoxybibenzyl (**1**) is a pink amorphous powder (MeOH). UV λ_{\max} (MeOH) (nm): 212 and 277. IR ν_{\max} (KBr) (cm^{-1}): 3322, 1617, 1594, 1511. ^1H and ^{13}C NMR data see Table I. Positive-HR-MS m/z 335.1632 (calcd for $\text{C}_{22}\text{H}_{22}\text{O}_3$, 335.1642) $[\text{M} + \text{H}]^+$, negative-FAB-MS m/z (rel. int): 333, $[\text{M} - \text{H}]^-$.

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