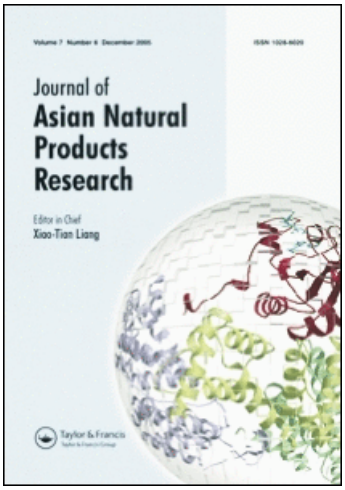

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A CINNAMIDE DERIVATIVE FROM *SOLANUM VERBASCIFOLIUM* L.

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A new cinnamide derivative together with *N* (*p*-hydroxyphenylethyl) *p*-coumaramide and vanillic acid has been isolated from the stems of *Solanum verbascifolium* L., the structure of the new compound was elucidated as *N*-2-hydroxy-2 (*p*-hydroxyphenylethyl) *p*-coumaramide (**1**) on the basis of physical and chemical evidence and spectral analysis.

Keywords: *Solanum verbascifolium*; Solanaceae; *p*-hydroxyphenylethyl; Cinnamide

INTRODUCTION

Solanum verbascifolium is a traditional folk medicine used for the treatment of metrorrhagia, edema, gout, carbuncles, eczema, toothache and dermatitis [1]. Solasonine is the major component in this plant [2]. As part of our systematic studies on the chemical constituents of commonly used traditional Chinese medicines, we carried out the chemical study on *S. verbascifolium*. A new cinnamide derivative, *N*-2-hydroxy-2 (*p*-hydroxyphenylethyl) *p*-coumaramide (**1**), along with *N* (*p*-hydroxylphenethyl) *p*-coumaramide [3] and vanillic acid [4] has been isolated from the title plant.

RESULTS AND DISCUSSION

Compound **1** was obtained as a colorless amorphous powder ($CHCl_3$–MeOH), mp. 236–238°C. $[\alpha]_D$: −29.0 (EtOH; $c = 0.031$). The UV spectrum of **1** exhibited typical absorption of a *trans*-cinnamide chromophore at λ_{max} 205, 225, 290 and 320 nm. Its IR spectrum showed the presence of hydroxyl (3236 cm^{-1}), carbonyl (1657 cm^{-1}), double bond and aromatic groups (1600,1587,1550,1512 cm^{-1}). A molecular formula of $C_{17}H_{17}O_4N$ was determined on the basis of quasi-molecular ion of HR-MS at *m/z* 322.1090 ($[M + Na]^+$).

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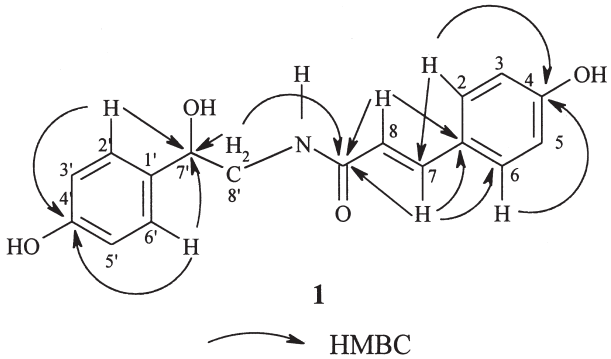

FIGURE 1 Important HMBC correlations of **1**.

And FAB-MS (positive) gave a molecular ion peak at *m/z* 300 (100%, $[M + H]^+$) and other fragments at *m/z* 283 (25%, $[M + H - OH]^+$), 282 (90%, $[M + H - H_2O]^+$), 207 (32%), 177 (22%), 164 (20%), 147 ($C_9H_7O_2$, 95%), 136 (20%), 120 (C_8H_8O, 17%) 115 (43%) and 107 (C_7H_7O, 20%). The ^{1}HNMR spectrum of **1** (Table I) showed signals of aromatic protons at δ 6.37–6.71 and a pair of AB doublet ($J = 16$ Hz) at δ 7.31 and 6.50, which are attributed to *trans*-olefinic protons of H-7 and H-8. Its ^{13}CNMR spectrum (Table I) also suggested the presence of *trans*-cinnamide and a substituted phenyl ethyl moiety. The structure of **1** was further confirmed by HMBC spectrum. Cross peaks were observed as follows: H-8′ (δ 3.36)/C-7′ (δ 71.2) and C = O (δ 165.6), H-8 (δ 6.50)/ C-1 (δ 125.9) and C = O (δ 165.6), H-7 (δ 7.31)/C-2, 6 (δ 129.2), C-1 (δ 125.9) and C = O (δ 165.6), H-2′, 6′ (δ 7.14)/C-4′ (δ 156.4) and C-7′ (δ 71.2), H-2, 6 (δ 7.37)/C-4 (δ 158.8) and C-7 (δ 138.6), respectively (Fig. 1). On the basis of the above spectral characteristics, the structure of the new compound (**1**) was established as *N*-2-hydroxy-2- (*p*-hydroxyphenylethyl) *p*-coumaramide.

EXPERIMENTAL SECTION

General Experimental Procedures

Melting points were determined on an XT4-100X micro melting point apparatus and are uncorrected. UV spectra were obtained on a Shimadzu UV-240 spectrophotometer. IR spectra were obtained in KBr on a Perkin–Elmer 683 infrared spectrophotometer. NMR spectra were determined on a Bruker AM 500 and Mercury 300 spectrometer using TMS as internal standard. FABMS were obtained on an Autospec-UltimaETOF mass spectrometer. Column chromatography was performed using silica gel (Qing Dao Hai Yang Chemical

TABLE I ^{1}H and ^{13}C-NMR data for **1** in DMSO (300 MHz for ^{1}H and 75 MHz for ^{13}C, δ in ppm, *J* in Hz)

No.	^{1}H	^{13}C	No.	^{1}H	^{13}C
1′		134.1	1		125.9
2′ (6′)	7.14 (2H, dd, 8.5)	127.2	2 (6)	7.37 (2H, dd, 8.5)	129.2
3′ (5′)	6.71 (2H, dd, 8.5)	114.7	3 (5)	6.78 (2H, dd, 8.5)	115.7
4′		156.4	4		158.4
7′	4.55 (1H, m)	71.2	7	7.31 (1H, d, 16)	138.6
8′	3.36 (2H, m)	47.1	8	6.50 (1H, d, 16)	118.8
C = O		165.6			

All assignments were confirmed by ^{1}H-^{13}CNMR and HMBC spectra (500 MHz for ^{1}H and 75 MHz for ^{13}C).

Group Co., Qing Dao, China), TLC was conducted on Si gel GF_{254} (Qing Dao Hai Yang Chemical Group Co.) and monitored at 254 nm. The polystyrene resin (RA) and Sephadex LH-20 were purchased from Beijing Chemical Factory and Shanghai Chemical Factory, respectively.

Plant Material

The stems of *Solanum verbascifolium* were collected from Yunnan province of the People's Republic of China in September 1998 and identified by Professor Wang Hong of Kunming Institute of Botany, Chinese Academy of Sciences. A voucher specimen (98041) of the plant has been deposited in our institute.

Extraction and Isolation

The stems of *Solanum verbascifolium* (4 kg) were extracted three times with EtOH under reflux. The combined EtOH extract was concentrated to give a residue (49 g), which was separated by chromatography on polystyrene resin RA with gradient elution of H_2O and EtOH. The fractions of H_2O, 70% EtOH and 90% EtOH were collected separately. The 70% EtOH fraction (37.3 g) was subject to Sephadex LH-20 and eluted with 80% EtOH to give A and B fractions. Fr. B (6.0 g) was purified by silica gel chromatography eluted with CH_2Cl_2–MeOH–H_2O (8:1.5:1 lower phase) to obtain compound 1, *N* (*p*-hydroxyphenylethyl) *p*-coumaramide and vanillic acid.

Characterization of New Compound

N-2-hydroxy-2(*p*-hydroxyphenylethyl) *p*-coumaramide (1) a colorless amorphous powder, mp 236-238°C ($CHCl_3$–MeOH). $[\alpha]_D$: −29.0 (EtOH; $c = 0.031$). UVλ_{max}^{MeOH} nm: 205, 225, 290, 320. IRυ_{max}^{KBr} cm^{-1}: 3236, 1657, 1600, 1587, 1550, 1512. ^1H and ^{13}C spectrum see Table I. HR-FABMS *m/z* 322.1090 (calcd for $C_{17}H_{17}O_4NNa$, 322.1055), FABMS *m/z*(rel. int.): 300 (100%, $[M + H]^+$), 283 (25%, $[M + H - OH]^+$), 282 (90%, $[M + H - H_2O]^+$), 207 (32%), 177 (22%), 164 (20%), 147 (95%), 136 (20%), 120 (17%), 115 (43%) and 107 (20%).

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