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Non-alkaloid constituents of *Vinca major*

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[ABSTRACT] The present study was designed to investigate the non-alkaloid compounds from the leaves and stems of *Vinca major* cultivated in Yunnan Province, China. The compounds were isolated using chromatographic techniques. The structures were elucidated by 1D- and 2D-NMR spectroscopic methods in combination with UV, IR, and MS analyses. The 1, 1-diphenyl-2-picrylhydrazyl (DPPH)-scavenging activity of Compounds **1**–**7** were evaluated. One new iridoid glycoside (compound **1**), together with 11 known compounds, were isolated from *Vinca major*. Compounds **1**, **5**, and **6** showed moderate DPPH-scavenging activity, with IC_{50} values being 70.6, 32.8, and 62.2 μmol·L^{-1}, respectively. In conclusion, compound **1** is a newly identified iridoid glycoside with moderate antioxidant activity.

[KEY WORDS] *Vinca major*; Iridoid glycoside; Vinmaside A; Antioxidant

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Introduction

The genus *Vinca*, belonging to the Apocynaceae family, is mainly native to Europe, northwest Africa, and southwest Asia, which has attracted considerable attention as a source of indole alkaloids with diverse structures and biological properties [1-6]. *Vinca major* has been cultivated widely in China as a flowering evergreen ornamental plant. Previous phytochemical investigations on the leaves and stems of *V. major* are focused on the alkaloid fraction and have led to the discovery of a series of indole alkaloids [7-11]. However, the non-alkaloid constitutes of this plant have seldomly been reported. As a continued systematic research on the chemical constitutes, our investigation on the non-alkaloid part of *V. major* resulted in the isolation a new iridoid glycoside, together with 11 known compounds (**2**–**12**). The structure of compound **1** was elucidated by means of spectroscopic methods and the known compounds were identified by comparison of their spectroscopic data with those reported in the literature. In addition, the antioxidant activities of compounds **1**–**7** were determined using the 1, 1-diphenyl-2-picryldyhydrazyl (DPPH) radical-scavenging assay were determined.

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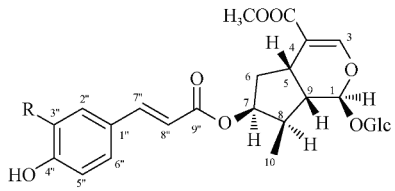
Results and Discussion

The molecular formula of compound **1** was assigned as $C_{27}H_{34}O_{13}$ on the basis of the negative quasi-molecular ion peak at *m/z* 565 $[M - H]^-$ and positive-ion HR-EI-MS at *m/z* 566.1993 $[M]^+$, in combination with its ^{13}C NMR spectrum. Its UV spectrum exhibited maximum absorptions at 204 and 290 nm. The ^1H NMR spectrum displayed the signals for an aromatic ABX spin system at δ_H 7.08 (1H, d, J = 8.2 Hz), 6.81 (1H, dd, J = 8.2, 1.9 Hz), and 7.21 (1H, d, J = 1.9 Hz), and two *trans*-substituted double bonds proton signals at δ_H 7.61 (1H, d, J = 15.9 Hz) and 6.39 (1H, d, J = 15.9 Hz), indicating the existence of a feruloyl moiety in combination with the methoxy group signal at δ_H 3.89 (3H, s) [12] (Table 1). Additionally, a methyl doublet signal at δ_H 1.12 (3H, d, J = 6.8 Hz, CH_3-10), a methoxy group signal at δ_H 3.69 (3H, s, $COOCH_3$), a trisubstituted olefinic proton signal at δ_H 7.44 (1H, s), an oxymethine doublet signal at δ_H 5.30 (1H, d, J = 5.0 Hz), and an anomeric proton signal at δ_H 4.66 (1H, d, J = 7.9 Hz)

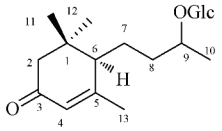
were also observed, which were characteristic signals for an iridoid glycoside. Besides feruloyl and glucoside groups, compound **1** possessed 12 carbon signals in the ^{13}C NMR spectrum, ascribable to one sp^2 methine (δ_C 152.6), five sp^3 methines (δ_C 97.6, 78.4, 47.0, 41.0, and 32.7), one sp^3 methylene (δ_C 40.4), one methoxyl group (δ_C 51.7), one methyl group (δ_C 13.7), and two sp^2 quaternary carbons (δ_C 169.3 and 113.1). All the above spectroscopic data were in a good agreement with 7-*O*-(*p*-coumaryl)-loganin [13], suggesting that compound **1** was an iridoid glycoside bearing a feruloyl moiety and a loganin moiety. In the HMBC spectrum, the methine proton signal at δ_H 5.27 (m, H-7) showed correlation with feruloyl carbonyl signal at δ_C 168.8 (C-9"), which revealed that the feruloyl moiety was located at C-7 (Fig. 2).

Table 1 ^1H and ^{13}C NMR spectral data of compound 1 in methnol-d_4 at 400 MHz

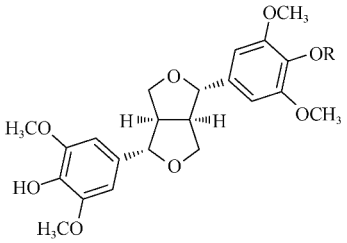
entry	δ_H	δ_C	entry	δ_H	δ_C
1	5.30, d (5.0)	97.6, d	4'	3.28, m	71.5, d
3	7.44, s	152.6, d	5'	3.32, m	78.4, d
4		113.1, s	6'a	3.91, d (11.7)	62.7, t
5	3.15, m	32.7, d	6'b	3.67, m	
6a	2.33, m	40.4, t	1"		127.6, s
6b	1.78, m		2"	7.21, d (1.9)	111.6, d
7	5.27, m	78.4, d	3"		149.3, d
8	2.18, m	41.0, d	4"		150.6, s
9	2.11, m	47.0, d	5"	7.08, d (8.2)	116.4, d
10	1.12, d (6.8)	13.7, q	6"	6.81, dd (8.2, 1.9)	124.1, d
11		169.3, s	7"	7.61, d (15.9)	115.6, d
$COOCH_3$	3.69, s	51.7, q	8"	6.39, d (15.9)	146.8, d
1'	4.66, d (7.9)	100.1, d	9"		168.8, s
2'	3.21, d (8.0)	74.7, d	OCH_3	3.89, s	56.4, q
3'	3.38, m	77.9, d			



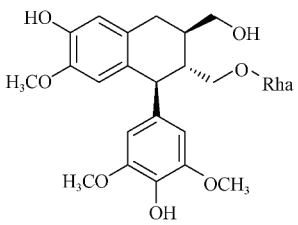
1 R=OCH_3
2 R=H



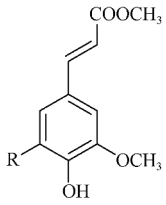
3 $\Delta^{7,8}$
4



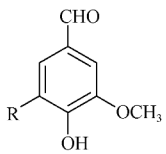
5 R=Glc
6 R=H



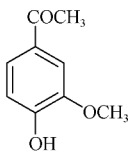
7



8 R = H
9 R = OCH_3



10 R = H
11 R = OCH_3



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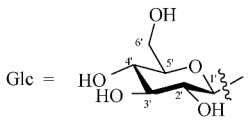
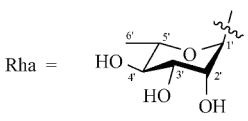


Glc =



Rha =

Fig. 1 Structures of compounds 1–12