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Peduncin, A New Indole Alkaloid from *Pueraria peduncualris*

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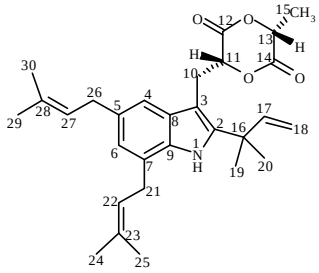
Abstract: A new indole alkaloid, peduncin was isolated from the roots of *Pueraria peduncularis*. Its structure was established as **1** by NMR spectroscopic and X-ray crystallographic analysis.

Key words: *Pueraria peduncularis*, Leguminosae, alkaloid, peduncin.

Pueraria peduncularis (Grah. ex Benth.) Benth. is a plant of Leguminosae which grows in the southwest of China. Different from other plants of *Pueraria*, it could not be used as a medicine due to its toxicity[1]. In order to use the *Pueraria* plants safer, we studied the constituents of *P. peduncularis*. Previous phytochemical studies of an *n*-BuOH extract afforded three triterpenoid saponins[2-3]. The continuing investigation on $CHCl_3$ extract led to the isolation of a new indole alkaloid. In this paper, we deal with the isolation and structural elucidation of this new compound.

Figure 1 The structure of peduncin.



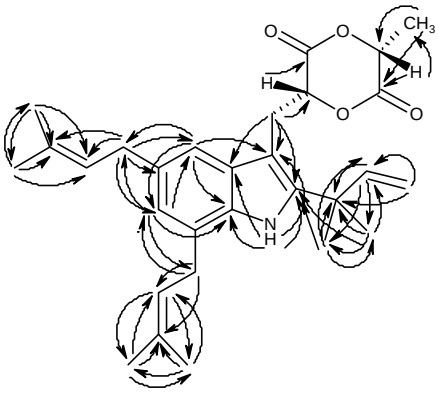
1

Dried roots of *P. peduncularis* were extracted with 95% EtOH three times under reflux. The extract was condensed and dissolved in water, then partitioned successively with $CHCl_3$ and *n*-BuOH. The $CHCl_3$ extract was subjected to a silica gel column chromatography to yield **1**.

Peduncin **1** was obtained as colorless platelets, mp >300°C. Its HR-FABMS *m/z* 463.3110 agreed with a molecular formula of $C_{29}H_{37}O_4N$ (calcd. for 463.3086). ^1H

NMR spectrum showed the presence of six quaternary methyl groups as singlets at *d* 1.51 (6H), 1.74 (6H), 1.81 (3H), 1.84 (3H) and a secondary methyl group at *d* 1.53 (*d*, 3H, *J*=6.9 Hz). Two isoprenyl groups, one 1,1-dimethylallyl group, one $CHCH_2$ fragment and one CH_3CH moiety could be elucidated by the combination of ^{1}H NMR, ^{1}H-^{1}H COSY, HMQC and HMBC spectra (**Table 1**). Besides these ^{1}H and ^{13}C NMR spectral signals, there were three proton signals at *d* 8.04 (*s*, 1H), 7.13 (*s*, 1H) and 6.80 (*s*, 1H) in ^{1}H NMR spectrum and two carbonyl carbons at *d* 168.5, 167.8, six quaternary carbons at *d* 141.4, 133.9, 132.3, 129.0, 123.4, 104.1, two methenyl carbons at *d* 122.9, 115.1 in ^{13}C NMR spectrum. It was very difficult to determine their linkages only by the HMBC spectrum, because there were too many quaternary carbons. In order to determine its structure, X-ray crystallography has been carried out.

Figure 2 Significant ^{13}C-^{1}H long-range correlations observed in the HMBC spectrum of **1**.



X-ray analysis of peduncin: crystal data: $C_{29}H_{37}O_4N$, MW=463.62. Orthorhombic in the space group $P2_1$, with Z=2, a=12.422 (1), b=6.235 (1), c=18.207 (1) Å, β=102.385 (3) and V=1377.3 (3) Å^3. It calculated density Dc=1.118 g/cm^3. Independent friedel (2448) were measured on MAC DIP-2030K image plate diffractometer with graphite-monchromated MoKα, radiation in *w*-scan mode. Friedal pairs (2416) were observed according to the criterion $|F|^2 \geq 8\sigma |F|^2$. The structure was resolved by direct method (SHELXS-86). The E-map gave the positions of 18 atoms. By using least-squares and Fourier difference methods, the positons of non-hydrogen atoms were established. The positions of all hydrogen atoms were determined by the geometric and Fourier difference methods, with R_f =0.083, R_w =0.073 ($w=1/\sigma^2|F|$), S =3.387, $(\Delta/\sigma)_{max}$ =0.106, $(\Delta\rho)_{min}$ = –0.250e/Å^3, $(\Delta\rho)_{max}$ =0.330e/Å^3.

From the X-ray crystallographic study (**Figure 3**), the structure of **1** was confirmed as shown in **Figure 1**. The ^{1}H NMR and ^{13}C NMR spectral data were assignable by a combination of 1D and 2D NMR techniques.

Figure 3 Stereoview of peduncin from X-ray diffraction analysis.

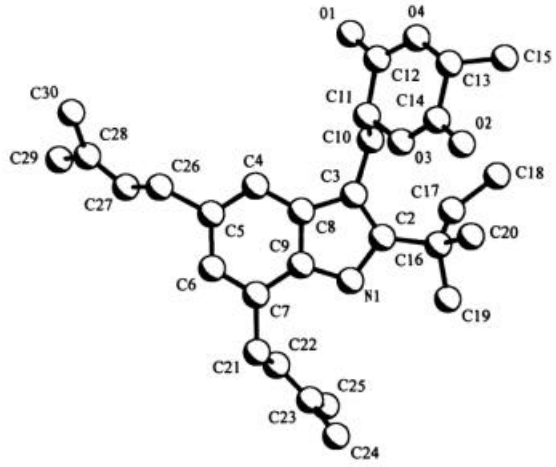


Table 1 ^{1}H NMR and ^{13}C NMR spectral data for **1***

	d_C	d_H (J Hz)	^{1}H-^{1}H	HMBC
1	N	8.04. s		C-2. C-3. C-8. C-9
2	141.4			
3	104.1			
4	115.1	7.13, s		C-3, C-6, C-9, C-26
5	133.9			
6	122.9	6.80, s		C-4, C-9, C-21, C-26
7	123.4			
8	129.0			
9	132.3			
10	29.5	3.66, dd (3.6, 14.7)	H-11	C-2, C-3, C-8, C-11
11	54.6	4.39, dd (11.5, 3.6)	H-10	C-12
12	168.5			
13	50.8	4.09, q (6.9)	H-15	C-14, C-15
14	167.8			
15	19.9	1.53, d (6.9)	H-13	C-13, C-14
16	39.0			
17	145.8	6.10, dd (17.2, 11.0)	H-18	C-16, C-19, C-20
18	112.3	5.16, d (17.2)	H-17	C-16, C-17
19	28.0	1.51, s		C-2, C-16, C-17, C-20
20	27.9	1.51, s		C-2, C-16, C-17, C-19
21	31.4	3.53, d (7.3)	H-22	C-6, C-22, C-23
22	122.9	5.42, t (7.3)	H-21	C-24, C-25
23	132.9			
24	17.9	1.84, s		C-22, C-23, C-25
25	25.8	1.81, s		C-22, C-23, C-24
26	34.6	3.39, d (7.2)	H-27	C-4, C-5, C-6, C-27, C-28
27	124.5	5.35, t (7.2)	H-26	
28	131.6			
29	25.7	1.74, s		C-27, C-28, C-30
30	17.9	1.74, s		C-27, C-28, C-29

* Measured in $CDCl_3$ at 400 MHz for ^{1}H and 100 MHz for ^{13}C NMR

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References

1. Z. P. Gu, B. Z. Chen, R. Z. Feng, *et al.*, *Acta Pharm. Sin.*, **1996**, *31*, 387.
2. N. Li, Y. L. Yang, Z. D. Min, H. M. Wu, *J. China Pharm. Univ.*, **1999**, *30* (3), 166.
3. N. Li, Z. D. Min, H. M. Wu, *Chin. Chem. Lett.* **2000**, *11*, 343.

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