

A New Isoflavone from *Smilax glabra*

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Abstract: A new isoflavone, 7,6'-dihydroxy-3'-methoxyisoflavone, has been isolated from the roots of *Smilax glabra*. The structure was determined by 2D-NMR techniques.

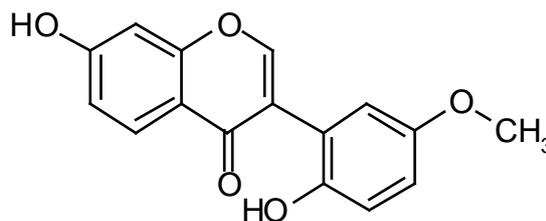
Keywords: *Smilax glabra*, isoflavone, 7,6'-dihydroxy-3'-methoxyisoflavone

Introduction

Smilax glabra Roxb. (Liliaceae), which was called "Tu Fu Ling", is widely distributed in east and southwest regions of China. In traditional Chinese medicine, the rhizome of *S. glabra* has been used clinically to prevent leptospirosis, and to treat syphilis, acute bacterial dysentery, acute and chronic nephritis, etc. [1].

Several chemical constituents have been isolated from this plant in our laboratory [2-5]. In the present work, silica gel chromatography of the EtOAc part of the EtOH extracts of the roots of *S. glabra* has led to the isolation of a new isoflavone. It was elucidated as 7,6'-dihydroxy-3'-methoxyisoflavone (**1**) based on MS and ¹H- and ¹³C-NMR spectroscopy, including ¹H-¹H COSY, COLOC and

NOEDS techniques. In this paper we report the structural elucidation of the new isoflavone **1**.



Formula 1

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Results and Discussions

Compound **1**, pale yellow needles (acetone), mp 250~252. Gave positive Mg-HCl (reddish) and FeCl₃ (greenish-brown) colour tests. In the ¹H-NMR spectrum, the singlet was observed at 7.7(s, 1H). In the UV spectrum, the maximum absorption was at 255 nm. Thus, **1** was deduced to be an isoflavone which was also confirmed by the ¹³C-NMR(DEPT) spectra (see Table 1).

The EI-MS of **1** exhibited [M]⁺ at m/z 284.0672 for C₁₆H₁₂O₅ (Calc. 284.0679) which is in accord with an isoflavone containing two hydroxyl groups and a methoxy group. In order to determine the substituted positions, 2D-NMR techniques were used. The COLOC spectrum showed a correlation between H-2 and C-9, H-8 and C-9, C-7. This correlation confirmed that the hydroxyl group in ring A was at the C-7 position.

Furthermore, the ion m/z 148 observed in the EIMS was derived from ring B and suggested that it carried one hydroxyl group and a methoxy group. In the ¹H-NMR, there are two groups of peaks of B ring, 6.5(1H, d, J=2Hz) and 6.3(2H, m). The position of methoxy group at

C-3' was deduced from the NOEDS of **1** that showed an enhancement of the peaks of methoxy and H-2 on irradiation of H-2'. In the ¹H-¹H-COSY, the correlations of H-2 and H-2', H-2 and MeO were shown. The position of the hydroxyl group at C-6' was confirmed by ¹H-¹H-COSY which displayed the correlation between methoxy and H-4'.

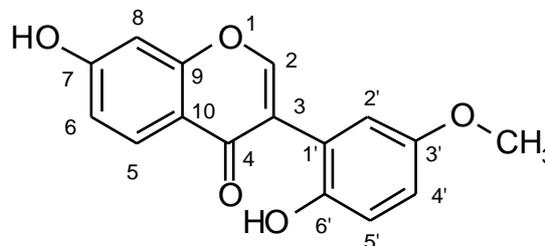


Figure 1. Atomic numbering of compound **1**.

The structure of **1** was fully in accordance with its NMR spectra including COLOC, DEPT and ¹H-¹H-COSY spectra. Thus, **1** was deduced to be 7,6'-dihydroxy, 3'-methoxy isoflavone.

Table 1. Correlated ¹³C- and ¹H-Data and COSY for compound **1**.

Atomic no.	C	CHn	H	Mult.	J(Hz)	COLOC (C to H)	¹ H- ¹ H COSY
2	153.4	CH	7.7	s		H-2	H-2', CH ₃
3	125.0	C				H-2,H-2'	
4	175.0	C				H-5, H-2,H-8	
5	127.6	CH	7.4	d	8.7	H-5	H-6
6	115.5	CH	6.3	m		H-8,OH-7	H-5, H-8
7	162.9	C				H-5, H-8	
8	102.4	CH	6.1	d	2.0	OH-7, H-8,H-6	H-5,H-6
9	157.7	C				H-5, H-2, H-8	
10	117.0	C				H-6, H-8	
1'	123.0	C				H-2',H-2,H-5'	
2'	120.1	CH	6.5	d	2.0	H-2',H-4'	H-2, H-4'
3'	147.8	C				CH ₃ , H-2',H-4'	
4'	116.8	CH	6.3	m		H-2',H-5'	CH ₃ ,H-2'
5'	112.2	CH	6.3	m		H-4'	H-2'
6'	146.3	C				H-2',H-5'	
OMe	55.9	CH3	3.8	s		CH ₃	H-2, H-4'

Experimental Section

Melting point was uncorrected. The IR spectrum was measured on a Nicolet IR-2000, ^1H NMR and ^{13}C NMR spectra were recorded on an ACF-300, DMSO- d_6 as solvent and TMS as int. standard. MS was recorded on a Finnigan FTMS-2000.

The dried roots of *Smilax glabra* were extracted with 95% EtOH under reflux. After evaporating the solvent, the crude extract was extracted with Et₂O and EtOAc, respectively to give 30g of EtOAc extract. Successive column chromatography using a CHCl₃-MeOH solvent system with increasing polarity, and repeated column chromatography of series (CHCl₃-MeOH, 20:1) afforded compound **1**.

Compound **1** is pale yellow crystal, mp 250-252°C. IR_{max} 3420, 1640 cm⁻¹. UV (MeOH) _{max} 227, 255, 290, 308 nm. EIMS m/z 284 (M⁺), 269, 253, 241, 213, 148, 137, 105. For ^1H NMR, ^{13}C NMR, ^1H - ^1H COSY, COLOC data, see Table 1.

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References and Notes

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Sample Availability: available from MDPI.