Dissectol A, A Novel Monoterpene Glycoside from *Incarvillea dissectifoliola*

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Abstract: Dissectol A, a novel monoterpene glycoside was isolated from the methanol part of the 95% EtOH extract of *Incarvillea dissecfoliola* and its structure was determined by 1D and 2D NMR data.

Keywords: Incarvillea dissectifoliola, dissectol A, monoterpene glycoside.

The genus Incarvillea (Bignoniaceae) is widely distributed in Yunnan Province and has various bioactivities. Incarvillea arguta is a Yi herbal medicine traditionally used to treat hepatitis, diarrhea and infectious diseases in Sichuan and Yunnan Provinces¹. I. sinensis is one of the traditional herbal medicines used to treat rheumatism and relieve pain². Literature reported that the crude extract of *I. arguta* display antibacterial activity against Staphylococcus aureus, Shigella sonnei, Pseudomonas aeruginosa and can inhibit influenza virus¹, and that argutone isolated form *I. arguta* had bacteriostatic and sedative activities³. Previous reports showed the isolation of monoterpene alkaloids including incarvillateine⁴, incarvilline⁵, incarvines A^6 , B, C^7 and incarvine D, methoxycarvillateine⁸, incarvillateines C and D⁹; N-oxides including incarvillateine N-oxide and incarvine A N-oxide⁸, and macrocyclic spermine alkaloids including incasines A, A', B, B' and C10 from I. sinensis, and 8-epideoxyloganic acid and alkaloids delayayines A, B and C from I. delayayi^{11, 12}. Moreover, some monoterpene alkaloids such as incarvillateine and delavayine A had antinociceptive activity 11, 13. Investigation on the phytochemistry of *I. dissectifoliola*¹⁴ was not reported before this work. Aerial parts of I. dissectifoliola collected in Yanyuan County, Sichuan Province were extracted with 95% EtoH. The alcoholic extract was separated into the petroleum ether, ethyl acetate and methanol parts. The methanol part was chromatographically isolated over silica gel and reversed-phase silica gel to afford a novel monoterpene glycoside, named dissectol A (1).

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Figure 1 Structural fragments of 1 and selected ROSEY (dashed \leftrightarrow) correlations.

The HRFABMS determined the molecular formula of **compound 1** to be $C_{16}H_{26}O_7$. The ^{13}C NMR and DEPT experiment (see **Table 1**) showed sixteen carbon signals for one methyl, seven methylene, five methane and three quaternary carbons including two functional groups, one ketone (δ 209.8) and a carbon-carbon double bond (δ 113.7 and 142.8). Fragments **1a**, **1b** and **1c** (**Figure 1**) were determined based on that the methyl protons at δ 0.88 had $^{1}H_{-}^{13}C$ long-range correlations (HMBC) with the carbons at δ 33.5, 36.5 and 67.6, and that in the $^{1}H_{-}^{1}H$ COSY, the proton at δ 1.34 showed correlations with the protons at δ 0.96 and 1.97, the proton at δ 3.90 showed correlations with the protons at δ 4.43 and 5.36, the proton at δ 3.84 showed correlations with the proton at δ 4.57. The proton at δ 3.90 (δ _C 78.7) showed $^{1}H_{-}^{13}C$ long-range correlations with carbons at δ 110.4 and 209.8, indicating that the carbon at δ 110.4 linked with the one at δ 78.7 *via* an oxygen, and the carbon at δ 209.8 linked with the one at δ 72.6, respectively, revealing the structure of the fragment **1d**. The fragment **1e** was constructed from the fragments **1b** and **1d** through the carbon at δ 87.4 connecting with the one at δ 209.8, and the

carbon at δ 51.6 connecting with the one at δ 87.4 because the proton at δ 5.36 showed $^{1}\text{H}^{-13}\text{C}$ long-range correlation with the carbon at δ 87.4, and the carbon at δ 87.4 showed $^{13}\text{C}^{-1}\text{H}$ long-range correlation with proton at δ 3.84. Moreover, the carbon at δ 87.4 showed $^{1}\text{H}^{-13}\text{C}$ correlations with the protons at δ 4.99 and 5.09, and carbon at δ 142.8 showed $^{1}\text{H}^{-13}\text{C}$ correlations with protons at δ 4.54 and 4.57, indicating that the carbon at δ 142.8 linked with carbon at δ 51.6 to form the fragment **1f**. The carbon at δ 142.8 showed $^{1}\text{H}^{-13}\text{C}$ correlations with the protons at δ 1.97 and 1.34, indicating the linkage of fragments **1c** and **1f** via the carbons at δ 142.8 and 37.3 to give the fragment **1g**. The carbon at δ 87.4 showed $^{1}\text{H}^{-13}\text{C}$ long-range correlations with the proton at δ 5.52 (s), and the carbon at δ 110.4d showed $^{1}\text{H}^{-13}\text{C}$ long-range correlations with the protons at δ 4.54 and 4.57, indicating that the carbon at δ 87.4 linked with the one at δ 110.4, and the carbon at δ 72.0 linked with the one at δ 110.4 through X, respectively. All oxygen substitutions were assigned based on ^{13}C NMR and HRFABMS data. The stereochemistry of C-2, C-4' and C-5' was determined to be shown in Figure 1 based on the correlations obtained from ROSEY experiment.

Table 1	The ¹ H, ¹³ C NMR	data, ¹ H- ¹³ C long-ran	1 Ige and 1 H- 1 H correlations 1
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position	¹³ C	$^{1}\mathrm{H}^{b}$	HMBC	¹ H- ¹ H COSY
1	72.0	4.54 (dd, Hβ)	C-2, C-3, C-1', C-2'	H-2
		4.57 (dd, Hα)	C-2, C-3, C-1'	H-2
2	51.6	3.84 (t, 9.5)	C-1, C-3, C-3a, C-4, C-2', C-3'	Ha-1, Hb-1
3	142.8	/	/	/
3a	113.7	4.99 (s, Ha)	C-2, C-3, C-4, C-2'	Hb-3a
		5.09 (s, Hb)	C-2, C-3, C-4, C-2'	Hb-3a
4	37.3	1.97 (m, 2H)	C-2, C-3, C-3a, C-5, C-6	H-5
5	25.6	1.34 (m, 2H)	C-3, C-4, C-6	H-4, H-6
6	33.5	0.96 (m, Ha)	C-5, C-8	H-5
		1.36 (m, Hb)	C-3, C-5, C-7a, C-8	Ha-6, H-8
7	36.5	1.65 (m)	C-5, C-6	Ha-6, Ha-7a
7a	67.6	3.54 (dd, 5.7, 10.4, Ha)	C-6, C-8	H-7
		3.62 (dd, 5.7, 10.4, Hb)	C-6, C-8	H-7
8	17.4	0.88 (d, 7.4, 3H)	C-6, C-7, C-7a	H-7
1'	110.4	5.52 (s)	C-1, C-2, C-2'	/
2'	87.4	/	/	/
3'	209.8	/	/	/
4'	72.6	5.36 (d, 10.0)	C-2c, C-2', C-3', C-5', C-6'	H-5'
5'	78.7	3.90 (br d, 10.0)	C-1', C-3'	H-4', H-6'
6'	62.1	4.43 (m, 2H)	C-4', C-5'	H-5'

^a ¹H, ¹³C-NMR and HMBC, ¹H-¹H COSY and ROESY spectra were obtained at 500 MHz, 125 MHz and 500MHz, and recorded in C₅D₅N at room temperature, respectively.

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^b Coupling constants are presented in Hz. Unless otherwise indicated, all proton signals integrate to ¹H.

^c Signals of ¹H-¹³C long-range correlations are weak.

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References

- 1. G. D. Li, Zhong Cao Yao, 1986, 17 (6), 272.
- X. Wang, J. R. Cui, Z. P. Xiao, Y. H. Zhang, CH. L. Li, Y. Y. Zhao, SH. Q. Cai, Journal of Beijing Medical University, 1998, 30 (2), 145.
- 3. M. K. Yang, Y. SH. Tang, L. M. Cai etc., Acta Pharmaceutica Sinica, 1987, 22 (9), 711.
- 4. Y. M. Chi, W. M. Yan , J. SH. Li, Phytochemistry, 1990, 29 (7), 23768.
- Y. M. Chi, W. M. Yan, D. CH. Chen, H. Noguchi, Y. Iitaka etc., *Phytochemistry*, 1992, 31 (8), 2930
- 6. Y. M. Chi, F. Hashimoto, W. M. Yan, T. Nohara, *Phytochemistry*, **1995**, 40 (1), 353.
- 7. Y. M. Chi, F. Hashimoto, W. M. Yan, T. Nohara, Phytochemistry, 1995, 39 (6), 1485.
- 8. Y. M. Chi, F. Hashimoto, W. M. Yan, T. Nohara, *Phytochemistry*, **1997**, 46 (4), 763.
- 9. M. Nakamura, Y. M. Chi, J. Kinjo, W. M. Yan, T. Nohara, Phytochemistry, 1999, 51, 595.
- 10. Y. M. Chi, F. Hashimoto, W. M. Yan, T. Nohara, Tetrahedron Letters, 1997, 38 (15), 2713.
- 11. M. Nakamura, K. Kido, J. Kinjo, T. Nohara, Phytochemistry, 2000, 53, 2536.
- 12. M. Nakamura, K. Kido, J. Kinjo, T. Nohara, Chem. Pharm. Bull., 2000, 48 (11), 1826.
- 13. M. Nakamura, Y. M. Chi, W. M. Yan, A. Yonezawa, Y. Nakasugi, T. Yoshizawa, F. Hashimoto, J. Kinjo, T. Nohara, SH. Sakurada, *Planta Med*, **2001**, *67*, 114.
- 14. Q. S. Zhao, Acta Phytotaxonomica Sinica, 1988, 26 (1), 78.

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