

Article ID: 1005 - 0108(2009)02 - 0144 - 03

## Claviconol, a new metabolite, from the mycelia culture of *Claviconora pyxidata*

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**Abstract:** **Aim** To explore the new bioactive metabolites from the mycelia culture of the edible mushroom *Claviconora pyxidata*. **Methods** The constituents were isolated and purified by column chromatography, and their structures were identified by spectroscopic analyses, including 1D- and 2D-NMR data, and single-crystal X-ray diffraction. **Results** A new natural product, named claviconol (**1**) along with adenosine (**2**), were obtained. **Conclusion** Compound **1** is a new compound and stereochemistry of **2** was confirmed by a single-crystal X-ray diffraction.

**Key words:** *Claviconora pyxidata*; claviconol; adenosine; single-crystal X-ray diffraction

**CLC number:** Q 284 **Document code:** A

## 珊瑚菌菌体培养物的新天然产物

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**摘要:** 目的 对珊瑚菌 (*Claviconora pyxidata*) 菌体培养物的活性成分进行分离。方法 用多种色谱技术对化合物进行分离纯化, 并用光谱技术和单晶 X 射线衍射技术鉴定化合物的结构。结果 从中分离到 2 个化合物: claviconol (**1**)、腺苷 (**2**)。结论 化合物 **1** 为新化合物, 并通过单晶 X 射线衍射技术确定了 **2** 的立体构型。

**关键词:** 珊瑚菌; claviconol; 腺苷; 单晶 X 射线衍射

*Claviconora pyxidata* is a wild mushroom which was widely used for curing gastric pain, dyspepsia, gout and heat-toxicity in China. Some compounds have been isolated from *Claviconora pyxidata*, such as claviconic acid which was a novel inhibitor of reverse transcriptases<sup>[1]</sup>. *Claviconora pyxi-*

*data* YB2005 was isolated from the wild fruit body in Jilin Province and identified by ITS methods. In exploring the new bioactive metabolites from this medicinal mushroom, a new metabolite, claviconol (**1**) along with adenosine (**2**) were obtained. Their structures were elucidated by the analysis of NMR

**Received date:** 2008 - 08 - 28

**Foundation item:** This work was financially supported by the National Natural Science Foundation of China (30500632), the National Science Fund for Distinguished Young Scholars to Y. M. Shen (30325044)

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data ( $^1\text{H}$ ,  $^{13}\text{C}$ -NMR, DEPT, HSQC, HMBC,  $^1\text{H}$ - $^1\text{H}$  COSY and NOESY). The relative stereochemistry of

**2** was confirmed by a single-crystal X-ray diffraction

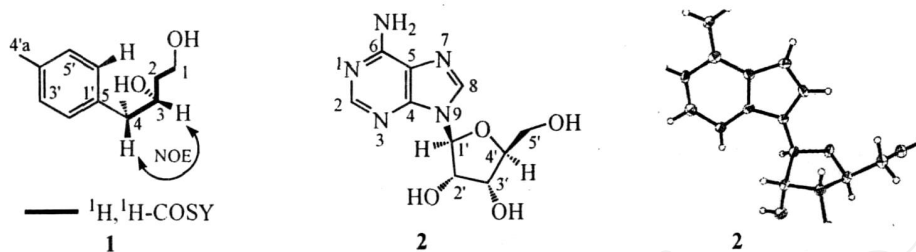


Figure 1 The structures of **1** and **2**

Clavicornol (**1**) was obtained as a colorless oil, [ $\alpha$ ] $_{\text{D}}^{20}$  + 4.56 (c 0.29,  $\text{CHCl}_3$ ). The molecular formula was determined as  $\text{C}_{12}\text{H}_{18}\text{O}_2$  according to the HR ESI-Q-TOF MS which showed the obvious quasi-molecular ion at  $m/z$  217.0933 [ $\text{M} + \text{Na}$ ] $^+$ . IR absorption implied the presence of hydroxyl ( $3353\text{ cm}^{-1}$ ), methyl ( $2924\text{ cm}^{-1}$ ) and aromatic ( $1593\text{ cm}^{-1}$ ) groups.  $^1\text{H}$ -NMR spectra of **1** (Table 1) showed the presence of four aromatic protons [at 7.09 (d, 2H,  $J = 7.8\text{ Hz}$ ); 7.17 (d, 2H,  $J = 7.8\text{ Hz}$ )] and indicated the 1, 4-bisubstitutions of aromatic moiety. The presence of aromatic moiety was further supported by the  $^{13}\text{C}$ -NMR resonances assigned to six aromatic carbons (Table 1). HMBC correlation from the methyl protons at 2.28 (H-4a) to C-3, C-4 and C-5 indicated the linkage of the methyl at C-4. The cross peaks between H-5 and H-4, H-4 and H-3, H-3 and H-2b, and H-1 and H-2a (2b) can be observed in the  $^1\text{H}$ - $^1\text{H}$ -COSY spectrum and allowed to establish the fragment of  $\text{CH}_3$  (5)-CH (4)-CH (3)- $\text{CH}_2$  (2)- $\text{CH}_2$  (1) (Figure 1). The key HMBC correlations from H-5 to C-4, C-3 and C-1, and H-4 to C-2, C-3, C-1 and C-2 (6), and H-1

to C-3 confirmed the connections of the fragment and allowed the linkage of the fragment with C-1. The hydroxyl groups were determined at C-1 and C-3 according to the downshift of the carbons at 61.0 and 74.9, respectively. The relative stereochemistry of **1** was determined by the NOE correlations between H-4 and H-3.

Adenosine (**2**) was elucidated by analysis of NMR data ( $^1\text{H}$ ,  $^{13}\text{C}$  NMR, HSQC, HMBC) (Table 2). Compound **2** was an enantiomer of  $\beta$ -adenosine whose NMR data ( $^1\text{H}$ ,  $^{13}\text{C}$ ) was reported by Ciuffreda<sup>[2]</sup> and coworkers. The stereochemistry of **2** was established by single-crystal X-ray diffraction (CCDC number: 685692) (Figure 1). It was reported that some edible fungi contained the high level of nucleosides and nucleobases<sup>[3]</sup>. Adenosine, one of nucleoside, possessed many kinds of bioactivities and have been used in clinic.

Compounds **1** - **2** showed no antibacterial (Escherichia coli, Bacillus Subtilis and Staphylococcus aureus) and anti-yeast (Candida albicans) activities at the concentration  $100\text{ }\mu\text{g}\cdot\text{mL}^{-1}$  using a similar MIC method and exhibited low cytotoxicities against HeLa cells by MTT assay.

Table 1  $^1\text{H}$ -NMR and  $^{13}\text{C}$ -NMR data for clavicornol (**1**) in  $(\text{CD}_3)_2\text{CO}$

Position	$^1\text{H}$ (600 MHz)	$^{13}\text{C}$ (150 MHz)	HMBC (600 MHz)
1	3.70 (d, 1H, $J = 5.4\text{ Hz}$ )	61.0 (t)	C-3
2a	1.64 (m, 1H)	37.3 (t)	C-1, C-3
2b	1.48 (m, 1H)		
3	3.91 (m, 1H)	74.9 (d)	
4	2.78 (m, 1H)	46.4 (d)	C-2, C-3, C-5, C-1, C-2 (6)
5	1.27 (d, 3H, $J = 7.2\text{ Hz}$ )	17.7 (q)	C-3, C-4, C-1
1		142.1 (s)	
2	7.09 (d, 1H, $J = 7.8\text{ Hz}$ )	129.4 (d)	C-4, C-4, C-3
3	7.17 (d, 1H, $J = 7.8\text{ Hz}$ )	129.2 (d)	C-4, C-4 a, C-2

(to be continued)

Continued Table 1

Position	$^1\text{H}$ (600 MHz)	$^{13}\text{C}$ (150 MHz)	HMBC (600 MHz)
4		135.9 (s)	
4 a	2.28 (s, 3H)	21.0 (q)	C-4, C-3 (5)
5	7.17 (d, 1H, $J=7.8$ Hz)	129.2 (d)	C-4, C-4 a, C-6
6	7.09 (d, 1H, $J=7.8$ Hz)	129.4 (d)	C-4, C-4, C-5
OH	3.64 (overlap, 1H)		
OH	3.64 (overlap, 1H)		

Table 2  $^1\text{H}$ -NMR and  $^{13}\text{C}$ -NMR data for adenosine(2) in  $\text{DMSO}-d_6$ 

Position	$^1\text{H}$ (600 MHz)	$^{13}\text{C}$ (150 MHz)	HMBC (600 MHz)
2	8.13 (s, 1H)	152.4 (d)	C-4, C-6
4		149.0 (s)	
5		119.3 (s)	
6		156.1 (s)	
8	8.34 (s, 1H)	139.9 (d)	C-4, C-5, C-1
1	5.88 (d, 1H, $J=6.1$ Hz)	87.9 (d)	C-4, C-8, C-2
2	4.61 (d, 1H, $J=6.1$ Hz)	73.4 (d)	C-1, C-4
3	4.14 (d, 1H, $J=1.9$ Hz)	70.6 (d)	C-1, C-5
4	3.96 (d, 1H, $J=1.9$ Hz)	85.9 (d)	C-3
5 a	3.68 (m, 1H)	61.6 (t)	C-3, C-4
5 b	3.56 (m, 1H)		
$\text{NH}_2$	7.36 (s, 2H)		C-6
2'-OH	5.47 (overlap, 1H)		C-1, C-2, C-3
3'-OH	5.21 (d, 1H, $J=3.9$ Hz)		C-2, C-3, C-4
5'-OH	5.46 (overlap, 1H)		C-4, C-5

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