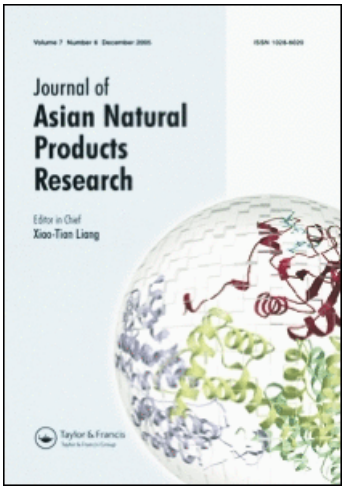

Journal of Asian Natural Products Research
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713454007

A new alkaloid from the stem of *Sparganium stoloniferum* Buch.-Ham

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Online publication date: 21 April 2010

To cite this Article Li, Shu-Xian , Wang, Feng , Deng, Xiao-Hui and Liang, Sheng-Wang(2010) 'A new alkaloid from the stem of *Sparganium stoloniferum* Buch.-Ham', Journal of Asian Natural Products Research, 12: 4, 331 — 333
To link to this Article: DOI: 10.1080/10286021003730330
URL: http://dx.doi.org/10.1080/10286021003730330

Journal of Asian Natural Products Research
Vol. 12, No. 4, April 2010, 331–333

NOTE

A new alkaloid from the stem of *Sparganium stoloniferum* Buch.-Ham

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(*Received 24 November 2009; final version received 24 February 2010*)

A new alkaloid (**1**), together with five known compounds (**2**–**6**), has been isolated from the stem of *Sparganium stoloniferum* Buch.-Ham. The structure of the new compound was elucidated as 3-isobutyl-tetrahydro-imidazo[1,2-*a*]pyridine-2,5-dione on the basis of physical and chemical evidence and spectral analysis. Compound **6** was obtained for the first time from the *Sparganium* genus.

Keywords: *Sparganium stoloniferum* Buch.-Ham; alkaloid; 3-isobutyl-tetrahydro-imidazo[1,2-*a*]pyridine-2,5-dione

1. Introduction

Sparganium stoloniferum Buch.-Ham is a traditional Chinese medicine used for the treatment of uterine myoma, hyperplasia of mammary glands, ovarian cysts, infertility, and dysmenorrhea. Volatile oil, flavonoids, saponins, and phenylpropanoids are the major chemical components in this plant [1]. As part of our systematic studies on the chemical constituents of commonly used traditional Chinese medicines, we carried out the chemical study of *S. stoloniferum* Buch.-Ham. A new alkaloid, 3-isobutyl-tetrahydro-imidazo[1,2-*a*]pyridine-2,5-dione (**1**), was isolated along with five known compounds: β-sitosterol (**2**), daucosterol (**3**), hydroxy cinnamic acid (**4**) [2], 3,4-dihydroxybenzoic acid (**5**) [3], and cyclo-(Phe-tyr) (**6**) [4,5]. Compound **6** was obtained for the first time from the *Sparganium* genus. The known compounds were identified by comparing their spectral data with reported data or with those of the authentic sample.

2. Results and discussion

Compound **1** was obtained as white needles. ESI-MS (negative mode) showed a pseudomolecular ion peak at *m/z* 209.1 $[M-H]^-$ and its molecular formula was determined as $C_{11}H_{18}O_2N_2$ on the basis of its HR-EI-MS spectrum (*m/z* 210.1363 $[M]^+$). Seventeen proton signals were observed in the ^1H NMR spectrum and 11 carbon signals in the ^{13}C NMR spectrum (Table 1), which were sorted by the DEPT experiment as two methyls, four methylenes, three methines, and two quaternary carbons, of which two amide groups resonating at δ 172.6 and 168.9 were suggested based on the chemical shifts. The existence of one reactive hydrogen linked to the N atom of one amide was revealed by the evidence that 18 hydrogens in the compound were suggested by the molecular weight but 17 were observed in the ^1H NMR spectrum. The ^1H and ^{13}C NMR spectral data were assigned by the ^1H–^1H COSY, HSQC, and

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ISSN 1028-6020 print/ISSN 1477-2213 online

DOI: 10.1080/10286021003730330
http://www.informaworld.com

Table 1. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectral data for compound **1** in CD_3OD.

No.	δ_C	δ_H	HMBC
2	168.9		
3	54.6	4.12 (1H, m)	1′, 2′, 2
5	172.6		
6	46.4	3.50 (2H, m)	7, 8
7	23.7	2.01 (1H, m)	8
		1.91 (1H, m)	8
8	29.1	2.30 (1H, m)	6
		2.02 (1H, m)	6
9	60.3	4.25 (1H, td, $J = 8.0$, 1.7 Hz)	3, 5, 8
1′	39.4	1.90 (1H, m)	2′, 4′, 2, 3
		1.51 (1H, m)	4′
2′	25.8	1.89 (1H, m)	
3′	23.3	0.96 (3H, d, $J = 6.4$ Hz)	1′, 2′
4′	22.2	0.95 (3H, d, $J = 6.3$ Hz)	

HMBC spectra (Table 1). Two methyl groups linked to the same carbon atom were indicated in the ^{1}H NMR spectrum (Table 1) at δ 0.96 (3H, d, $J = 6.4$ Hz) and 0.95 (3H, d, $J = 6.3$ Hz).

The detailed structural analysis was obtained from the HMBC spectrum. In the HMBC spectrum, the long-range correlations between H-3′ at δ 0.96 and C-4′ at δ 22.2, C-2′ at δ 25.8, C-1′ at δ 39.4 suggested the existence of the isobutyl group. Moreover, it was observed in the HMBC spectrum that the cross-peaks between H-9 at δ 4.25 and C-8 at δ 29.1, C-3 at δ 54.6, C-5 at δ 172.6, between H-3 at δ 4.12 and C-2 at δ 168.9 showed the presence of the tetrahydro-imidazo[1,2-*a*]pyridine-2,5-dione moiety. Isobutyl linked at C-3 was supported by the correlations observed in the HMBC spectrum of H-1′ (δ 1.51)/C-3 (δ 54.6), C-2 (δ 168.9) (see Figure 1). Because the nitrogen was linked to the carbonyl, the bond between the carbon and nitrogen tended to be double bonds and all atoms linked to the amide group were approximately in the same plane. So, only the stereochemistry of C-3 and C-9 would be taken into consideration. The cross-peak between H-3 and H-9 (see Figure 2) in the ROESY experiment implied that they were in the same side. Thus, the structure of **1** was elucidated as *cis*-3-isobutyl-tetrahydro-imidazo[1,2-*a*]pyridine-2,5-dione.

3. Experimental

3.1 General experimental procedures

Melting points were determined on a Yanaco MP-S_3 micro-melting point apparatus and are uncorrected. ESI-MS were obtained on an LCQ^{TM} DECA mass spectrometer.

NMR spectra were determined on a Bruker AVANCE AV 400 and Varian UNITY INOVA 500 spectrometer using TMS as the internal standard. Column chromatography was performed using silica gel (Qingdao Hayang Chemical

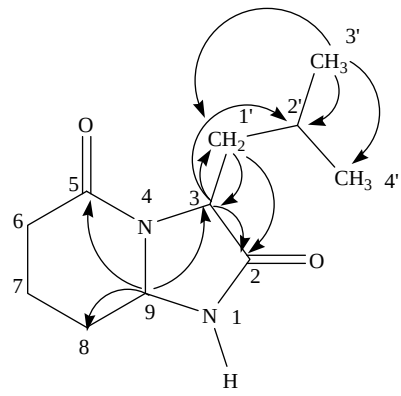

Figure 1. Important HMBC correlations of **1**.

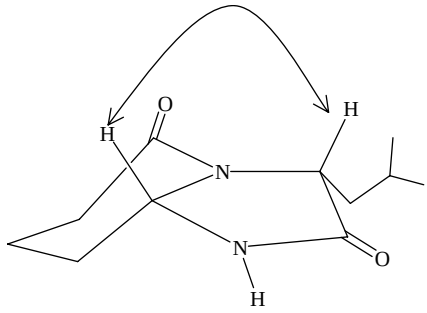

Figure 2. Key NOE correlations of **1**.

Group Co., Qingdao, China). TLC was conducted on silica gel GF254 (Qingdao Haiyang Chemical Group Co.) and monitored at 254 nm.

3.2 Plant material

The stems of *S. stoloniferum* Buch.-Ham were collected from Nanyang, Henna, China in March 2008 and identified by Prof. Shu-Yuan Li of the School of Traditional Chinese Medicine, Guangdong Pharmaceutical University. A voucher specimen (080410) of the plant has been deposited in our school.

3.3 Extraction and isolation

The air-dried and powdered stem barks of *S. stoloniferum* Buch.-Ham (14 kg) were extracted with 70% ethanol (85 liters × 3) under reflux. The ethanol was evaporated under reduced pressure to give a residue (356 g), which was suspended in water (0.5 liters), defatted with petroleum ether (1.0 liters × 5), and extracted with EtOAc (1.5 liters × 4) to give a residue (8.5 g), which was divided into five subfractions A–E by silica gel (500 g, 80 × 310 mm) column chromatography, eluted with $CHCl_3$–MeOH (50:1, 30:1, 10:1, 5:1, each 500 ml). Subfraction B (1.5 g) was separated by column chromatography on silica gel (40 g, 40 × 100 mm), eluted with $CHCl_3$–MeOH (60:1, 30:1, each 500 ml) to afford **2** (19 mg) and **3** (33 mg). Compounds **4** (7.6 mg) and **5** (9.3 mg) were obtained from subfraction C (2.8 g) by silica gel (280 g, 50 × 230 mm) column chromatography with $CHCl_3$–MeOH (15:1, 2.0 liters) as solvents. Subfraction D (1.1 g) was separated by silica gel (35 g, 38 × 90 mm) column chromatography and eluted with $CHCl_3$–MeOH (10:1, 1.5 liters) to give **1** (4.2 mg). Compound **6** (10 mg) was isolated from subfraction E (1.4 g) by silica gel (35 g, 38 × 90 mm) column chromatography using petroleum ether–acetone (8:1, 1.0 liters) as solvents.

*3.3.1 Cis-3-isobutyl-tetrahydro-imidazo[1,2-a]pyridine-2,5-dione (**1**)*

White needle crystal ($CHCl_3$–MeOH); mp 301–303°C; IR (KBr) ν_{max} (cm^{-1}): 3260 (NH), 1667 (C═O), 1635 (C═O). ^1H and ^{13}C spectral data: see Table 1. ESI-MS (negative mode) *m/z*: 209.1 $[M-H]^-$. HR-EI-MS *m/z*: 210.1363 $[M]^+$ (calcd for $C_{11}H_{18}O_2N_2$, 210.1368).

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