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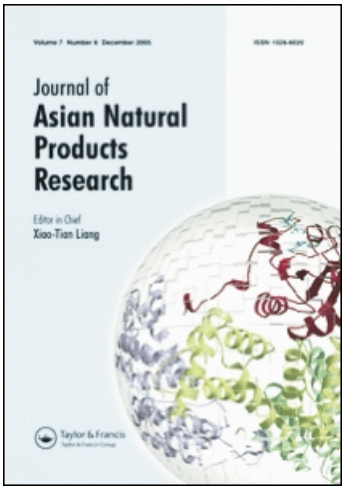

Journal of Asian Natural Products Research
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713454007

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To cite this Article Peng, Chong-Sheng , Wang, Feng-Peng and Jian, Xi-Xian(2000) 'New Norditerpenoid Alkaloids from *Aconitum Hemsleyanum* var. *Pengzhouense*', Journal of Asian Natural Products Research, 2: 4, 245 — 249
To link to this Article: DOI: 10.1080/10286020008041362
URL: http://dx.doi.org/10.1080/10286020008041362

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JANPR, Vol. 2, pp. 245–249
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NEW NORDITERPENOID ALKALOIDS FROM *ACONITUM HEMSLEYANUM* VAR. *PENGZHOUENSE*

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(Received 19 October 1999; Revised 12 November 1999; In final form 30 November 1999)

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Two new norditerpenoid alkaloids, 13-deoxyludaconitine (**1**) and 8-deacetylsungpaconitine (**3**), were isolated from the roots of *Aconitum hemsleyanum* Pritz var. *pengzhouense* and their structures were elucidated by spectral data.

Keywords: *Aconitum hemsleyanum* Pritz var. *pengzhouense*; *Ranunculaceae*; Norditerpenoid alkaloids; 13-Deoxyludaconitine; 8-Deacetylsungpaconitine

INTRODUCTION

The isolation and identification of the main norditerpenoid alkaloids from the roots of *Aconitum hemsleyanum* Pritz var. *pengzhouense* W.J. Zhang et G.H. Chen have been reported previously [1]. Continuation of our investigation on the plant led to the isolation of two new norditerpenoid alkaloids, 13-deoxyludaconitine (**1**) and 8-deacetylsungpaconitine (**3**). Here we report their isolation and structure elucidation.

RESULTS AND DISCUSSION

The two new bases, whose molecular formula were determined by EIMS and ^{13}C NMR, are norditerpenoid alkaloids according to characteristic signals in their NMR and MS spectra [2–4].

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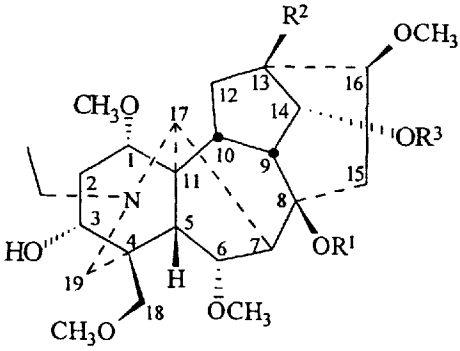
The ^{1}H NMR spectrum of 13-deoxyludaconitine (**1**), $C_{32}H_{45}NO_8$, exhibited the presence of an *N*-ethyl group (δ_H 1.16, 3H, *t*, $J = 6.6$ Hz; δ_C 48.9 *t* and 12.5 *q*), four methoxyl groups (δ_H 3.22, 3.30, 3.30 and 3.34, each 3H, *s*; δ_C 55.5 *q*, 56.0 *q*, 57.8 *q* and 59.1 *q*), and one benzoyl group (δ_H 7.39–8.05, 5H, *m*; δ_C 166.2 *s*, 130.9 *s*, 129.5 *d*, 128.4 *d* and 132.8 *d*). The presence of the 1H triplet ($J = 4.8$ Hz) signal at δ_H 5.15 attributable to H-14β [2,3] in the ^{1}H spectrum of **1** indicated that it had a benzoyl group at C-14. The NMR spectrum of **1** showed that it lacks a hydroxyl group when compared with ludaconitine (**2**) [1]. Comparison of the ^{13}C NMR data (Table I) of ring C between **1** and **2** indicated that the former had no hydroxyl group at the C-13 position. Therefore, the structure of compound **1** was determined as 13-deoxyludaconitine.



	R1	R2	R3
1	H	H	Bz
2	H	OH	Bz
3	H	H	Cn
4	Ac	H	Cn

Cn: -OC—C(H)=C(H)—C_6H_5

The NMR spectra of 8-deacetyl sungpaconitine (**3**), $C_{34}H_{47}NO_8$, gave the signals at δ_H 1.18 (3H, *t*, $J = 6.9$ Hz), δ_C 48.9 *t* and 12.9 *q*, for an *N*-ethyl group, δ_H 3.25, 3.27, 3.31 and 3.41 (each 3H, *s*), δ_C 55.6 *q*, 56.1 *q*, 57.8 *q* and 59.2 *q* for four methoxyl groups, δ_H 6.42, 7.33 (each 1H, *d*, $J = 16.0$ Hz), 7.32–7.54 (5H, *m*), δ_C 166.4 *s*, 117.7 *d*, 145.3 *d*, 130.4 *s*, 128.8 *d*, 128.2 *d* and 134.2 *d* for a cinnamoyl group. The 1H triplet ($J = 4.8$ Hz) at δ 4.99 in the ^{1}H NMR spectrum of **3** was assigned to H-14β [2,3], indicating that it has a cinnamoyl group at C-14. The presence of the 1-OCH3 group was deduced from an intense fragment ion peak at *m/z* 566 (M-31, 60) [4]. The ^{13}C NMR spectrum of **3** showed seven oxygenated signals at δ_C 82.6 *d*, 81.9 *d*, 81.7 *d*, 77.4 *t*, 76.6 *d*, 73.8 *s* and 71.4 *d* for C-1, C-16, C-6, C-18, C-14, C-8 and C-3, respectively, which were assigned by comparison with sungpaconitine (**4**) [5] (Table I). The NMR spectrum of **3** showed that it lacks an acetyl group at C-8 but has a tertiary hydroxyl group as compared with **4**, indicating that **3** is a 8-deacetyl derivative of sungpaconitine (**4**). Thus, the structure of compound **3** was determined as 8-deacetylsungpaconitine.

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TABLE I ^{13}C NMR data of compounds **1**, **2** [1], **3** and **4** [5]

Carbon	**1**	**2**	**3**	**4**
1	82.4	83.0	82.6	83.8
2	33.0	33.4	32.4	33.5
3	71.2	71.8	71.4	71.8
4	43.1	43.2	43.2	43.2
5	45.8	47.6	46.3	48.6
6	81.7	82.4	81.7	82.4
7	53.4	53.3	53.2	47.7
8	73.9	73.8	73.8	85.9
9	45.8	47.6	53.2	48.8
10	37.1	41.9	37.0	38.1
11	50.4	50.2	50.4	50.6
12	28.4	35.8	28.7	28.6
13	44.5	75.9	44.6	44.9
14	76.5	80.1	76.6	76.6
15	41.4	42.1	41.5	39.4
16	81.7	82.4	81.9	82.8
17	62.2	61.9	61.9	61.2
18	77.1	77.3	77.4	77.3
19	48.6	47.5	48.4	47.0
N–CH_2	48.9	48.9	48.9	48.8
CH_3	12.5	13.4	12.9	13.3
1α-OCH_3	56.0	56.3	55.6	55.6
6α-OCH_3	57.8	58.2	57.8	58.0
16β-OCH_3	55.5	57.5	56.1	56.6
18-OCH_3	59.1	59.5	59.2	59.2
O=C	...	...	...	169.7
CH_3	...	...	...	22.4
O=C	166.2	166.7	...	...
1′	130.9	129.7	...	...
2′	129.5	129.5	...	...
3′	128.4	128.5	...	...
4′	132.8	133.1	...	...
O=C	...	...	166.4	166.0
CH	...	...	117.7	118.5
HC	...	...	145.3	145.1
1′	...	...	130.4	130.4
2′	...	...	128.8	129.0
3′	...	...	128.2	128.1
4′	...	...	134.2	134.4

EXPERIMENTAL SECTION

General Experimental Procedures

Optical rotations were measured on a Perkin Elmer 241 spectrophotometer. EIMS data were recorded with a Finnigan M-80A GC/MS spectrometer. ^1H- and ^{13}C NMR spectra were measured in $CDCl_3$, with TMS as internal

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standard, on a Bruker AC-E 200 spectrometer. Other procedures for extraction and isolation, see Ref. [1].

Plant Material

The plants *Aconitum hemsleyanum* Pritz var. *pengzhouense* W.J. Zhang et G.H. Chen were collected in September 1993 in Peng county of Sichuan province, China, and authenticated by Professor W.T. Wang, Institute of Botany, Chinese Academy of Sciences, where a voucher specimen has been deposited.

Extraction and Isolation

The total alkaloids (186 g) obtained by the use of an ion exchange resin from 30 kg of the roots of *Aconitum hemsleyanum* var. *pengzhouense* [1] were divided into five parts, A (pH 2: 32 g), B (pH 5: 6.5 g), C (pH 7: 8 g), D (pH 9: 35 g) and E (pH 11: 3.5 g) by pH gradient separation.

Column chromatography of part A using $CHCl_3$–MeOH (98 : 2 → 90 : 10) led to fractions A (290 mg), B (4.13 g), C (835 mg), D (792 mg) and E (260 mg, 13-deoxyludaconitine). Column chromatography of fraction D eluting with $CHCl_3$–MeOH (95 : 5) gave 8-deacetyl sungpaconitine (72 mg). Separation and identification (TLC, m.p., MS, ^{1}H- and ^{13}C NMR) of the known alkaloids, see Ref. [1].

13-Deoxyludaconitine (1)

This was obtained as a homogenous amorphous substance, 260 mg, $[\alpha]_D^{17}$ +22.2 (c 0.5, $CHCl_3$). ^{1}H NMR (200 MHz): δ 1.16 (3H, *t*, $J = 6.6$ Hz, NCH_2CH_3), 3.22, 3.30, 3.30, 3.34 (each 3H, s, 4× OCH_3), 5.15 (1H, *t*, $J = 4.8$ Hz, H-14β), 7.39–8.05 (5H, *m*, H–Ar); ^{13}C NMR data, see Table I; EIMS *m/z* (%): 571 (M^+, 5), 540 (M-31, 13), 105 (100).

8-Deacetylsungpaconitine (3)

This was obtained as a homogenous amorphous substance, 72 mg, $[\alpha]_D^{17}$ +38.0 (c 0.45, $CHCl_3$); ^{1}H NMR (200 MHz): δ 1.18 (3H, *t*, $J = 7.1$ Hz, NCH_2CH_3), 3.25, 3.27, 3.31, 3.41 (each 3H, *s*, 4× OCH_3), 4.99 (1H, *t*, $J = 4.8$ Hz, H-14β), 6.42, 7.33 (each ^{1}H, *d*, $J = 16.0$ Hz, –CH=CH–), 7.32–7.54 (5H, *m*, H–Ar); ^{13}C NMR data: see Table I; EIMS *m/z* (%): 597 (M^+, 5), 566 (M-31, 60), 131 (68), 103 (29), 58 (100).

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