

Four New Nor-Diterpenoid Alkaloids from *Aconitum brachypodum*

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Four new C₁₉-nor-diterpenoid alkaloids, named brachyaconitines A–D (**1–4**), were isolated from the roots of *Aconitum brachypodum* DIELS. Their structures were elucidated as 3-*O*-acetyl-20-deethyl-20-formylaconitine (**1**), 3-*O*-acetyl-19,20-didehydro-20-deethylaconitine (**2**), 3-*O*-acetyl-8-de(acetyloxy)-7,8,17,20-tetrahydro-20-deethyl-7,17-secoaconitine (**3**), and 1-*O*-methylflavaconitine (**4**) by means of MS, IR, 1D- and 2D-NMR analyses. The structure of compound **1** was confirmed by an X-ray diffraction experiment.

Introduction. – *Aconitum brachypodum* DIELS., a commonly used folk-medicinal herb, is mainly distributed in Yunnan and Sichuan Provinces of China [1]. Its dried roots, ‘Xue-Shang-Yi-Zhi-Hao’ in the Chinese Pharmacopoeia [2], is widely used in traditional Chinese medicine for the treatment of rheumatism and pains [3]. As part of our ongoing phytochemical investigation on *A. brachypodum*, four new diterpenoid alkaloids, named brachyaconitines A–D (**1–4**; Fig. 1), were isolated from the

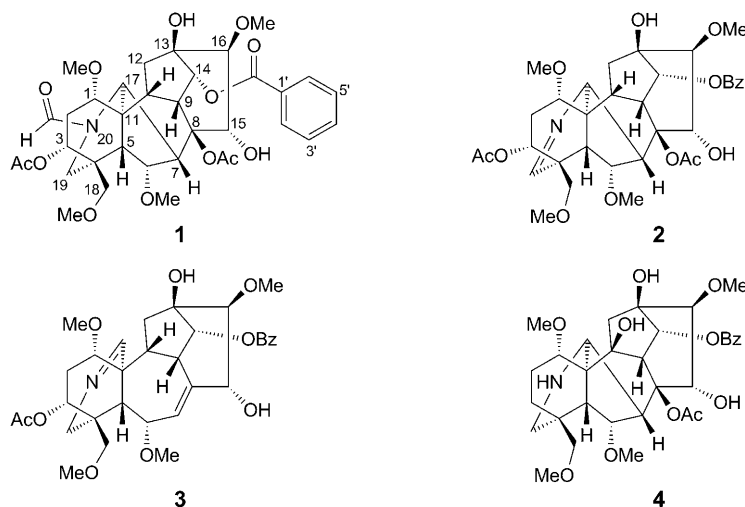


Fig. 1. Compounds **1–4**, isolated from *Aconitum brachypodum* DIELS.

95% EtOH extract of its roots, together with the five known compounds bullatine A [4], aconitine = (1 α ,3 α ,6 α ,14 α ,15 α ,16 β)-20-ethyl-1,6,16-trimethoxy-4-(methoxymethyl)-aconitane-3,8,13,14,15-pentol 8-acetate 14-benzoate [5], neoline [6], hypaconitine [7], and songorine [8]. All of the isolated compounds showed a positive reaction with *Dragendorff's* reagent. This article describes the isolation and structural elucidation of the four new compounds.

Results and Discussion. – Compound **1** was obtained as colorless prisms from pyridine. Its HR-ESI-MS exhibited a *quasi*-molecular-ion peak at m/z 710.2771 ($[M + Na]^+$), corresponding to the molecular formula C₃₅H₄₅NO₁₃ with 14 degrees of unsaturation. The IR spectrum showed the absorption bands for OH (3500 cm⁻¹), conjugated-ester C=O (1721 cm⁻¹), amide C=O (1664 cm⁻¹), and aromatic-ring functions (1602 and 1451 cm⁻¹). In the ¹H-NMR spectrum (*Table 1*), four MeO groups

Table 1. ¹H-NMR Data (CDCl₃) of Compounds **1–4**. δ in ppm, J in Hz.

	1 ^{a)}	2 ^{a)}	3 ^{a)}	4 ^{b)}
CH(1)	3.14–3.18 ^{c)}	3.20–3.25 ^{c)}	3.22–3.27 ^{c)}	4.01 ($d, J=6.2$)
CH ₂ (2)	1.37 ($dd, J=12.7, 10.4$), 2.47–2.52 (m)	1.73–1.79 (m), 2.00–2.06 (m)	1.14 ($dd, J=12.1, 12.1$), 2.29–2.32 (m)	1.36–1.43 ^{c)} , 1.91–1.94 (m)
CH(3) or CH ₂ (3)	4.41 ($dd, J=13.0, 5.4$)	5.12 ($d, J=6.3$)	4.92 ($dd, J=15.3, 8.7$)	1.36–1.43 ^{c)} , 1.77–1.80 (m)
CH(5)	2.51 ($d, J=6.8$)	2.31 ($d, J=6.9$)	2.21–2.28 ^{c)}	2.55 ($d, J=6.5$)
CH(6)	4.16 ($d, J=7.0$)	3.99 ($d, J=7.1$)	4.52 ($dd, J=10.6, 7.7$)	4.52 ($d, J=5.2$)
CH(7)	2.68 ($br. s$)	2.90 ($br. s$)	5.66 ($d, J=5.6$)	2.80 ($br. s$)
CH(9)	2.85 ($dd, J=6.7, 5.8$)	2.72 ($t, J=4.8$)	2.31–2.35 ^{c)}	2.75 ($d, J=5.0$)
CH(10)	2.17 ($dd, J=6.9, 5.9$)	2.15–2.19 ^{c)}	2.45–2.52 ^{c)}	–
CH ₂ (12)	2.05–2.31 (m), 2.94 ($dd, J=11.6, 5.2$)	2.15–2.19 (m), 2.35–2.39 (m)	2.45–2.52 ^{c)}	2.17 ($br. s$), 2.51 ($br. s$)
CH(14)	4.87 ($d, J=5.0$)	4.89 ($t, J=4.7$)	5.10 ($t, J=4.1$)	5.39 ($d, J=5.1$)
CH(15)	4.47 ($d, J=5.1$)	4.48 ($d, J=4.3$)	4.86 ($d, J=4.3$)	3.70 ($br. s$)
CH(16)	3.32 ($d, J=5.1$)	3.40–3.46 ^{c)}	3.27–3.33 ^{c)}	3.37 ($d, J=5.2$)
CH(17)	4.03 ($br. s$)	4.17 ($br. s$)	7.85 ($br. s$)	2.88 ($br. s$)
CH ₂ (18)	3.08 ($d, J=8.9$), 3.94 ($d, J=8.9$)	3.40–3.46 ^{c)} , 4.05 ($d, J=8.5$)	2.98 ($d, J=8.8$), 4.02 ($d, J=8.8$)	3.01 ($d, J=8.3$), 3.58 ($d, J=8.4$)
CH ₂ (19) or CH(19)	2.94 ($dd, J=13.9, 5.4$), 4.02 ($d, J=13.2$)	7.37 ($br. s$)	2.31–2.35 ^{c)} , 2.77–2.83 ^{c)}	2.16–2.20 ^{c)} , 3.22–3.27 ^{c)}
N–CHO	8.10 ($br. s$)	–	–	–
MeO–C(1)	3.14 (s)	3.06 (s)	3.21 (s)	3.14 (s)
MeO–C(6)	3.19 (s)	3.18 (s)	3.22 (s)	3.26 (s)
MeO–C(16)	3.20 (s)	3.25 (s)	3.24 (s)	3.29 (s)
MeO–C(18)	3.75 (s)	3.75 (s)	3.76 (s)	3.76 (s)
AcO–C(3)	2.03 (s)	2.06 (s)	2.07 (s)	–
AcO–C(8)	1.32 (s)	1.33 (s)	–	1.39 (s)
CH(2',6')	8.01 ($d, J=7.6$)	8.01 ($d, J=7.6$)	8.05 ($d, J=7.2$)	8.01 ($d, J=7.2$)
CH(3',5')	7.45 ($dd, J=7.6$)	7.44 ($dd, J=7.5$)	7.45 ($dd, J=7.2$)	7.45 ($dd, J=7.3$)
CH(4')	7.57 ($t, J=7.6$)	7.57 ($t, J=7.5$)	7.57 ($t, J=7.3$)	7.57 ($t, J=7.3$)

^{a)} 500 MHz. ^{b)} 400 MHz. ^{c)} Overlapped.

($\delta(\text{H})$ 3.14, 3.19, 3.20, and 3.75 (4s)) were observed together with two AcO groups ($\delta(\text{H})$ 1.32 and 2.03 (2s)), and a Bz unit ($\delta(\text{H})$ 7.45 (*dd*, $J=7.6$, 2 H), 7.57 (*t*, $J=7.6$, 1 H), and 8.01 (*d*, $J=7.6$, 2 H)). Its ^{13}C -NMR (DEPT) spectrum (Table 2) displayed 35 C-atom signals including 6 Me, 4 CH_2 , and 17 CH groups, and 8 quaternary C-atoms, suggesting that compound **1** might be an aconitine-type C_{19} -nor-diterpenoid alkaloid, bearing the following groups: one $\text{C}_{19}\text{H}_{19}$, two OH, four MeO, two AcO, one BzO, and one NCHO. Careful analyses of the ^1H - and ^{13}C -NMR data suggested that the structure of compound **1** was similar to that of 3-*O*-acetylaconitine [9]. The main difference between the two compounds is that compound **1** contains an *N*-formyl unit instead of a *N*-ethyl group in 3-*O*-acetylaconitine. The long-range HMBCs (Fig. 2) between the formyl H-atom ($\delta(\text{H})$ 8.10 (br. s)) and C(17) and C(19) confirmed the location of the additional formyl group. Compound **1** was presumed to possess a similar relative

Table 2. ^{13}C -NMR Data (CDCl_3) of Compounds **1**–**4**. δ in ppm.

	1 ^{a)}	2 ^{a)}	3 ^{a)}	4 ^{b)}
C(1)	78.9 (<i>d</i>)	80.3 (<i>d</i>)	79.7 (<i>d</i>)	82.9 (<i>d</i>)
C(2)	30.8 (<i>t</i>)	30.0 (<i>t</i>)	29.9 (<i>t</i>)	23.9 (<i>t</i>)
C(3)	70.5 (<i>d</i>)	72.9 (<i>d</i>)	71.7 (<i>d</i>)	28.9 (<i>t</i>)
C(4)	41.1 (<i>s</i>)	49.7 (<i>s</i>)	47.3 (<i>s</i>)	38.7 (<i>s</i>)
C(5)	46.5 (<i>d</i>)	44.3 (<i>d</i>)	42.6 (<i>d</i>)	39.6 (<i>d</i>)
C(6)	82.9 (<i>d</i>)	83.8 (<i>d</i>)	86.4 (<i>d</i>)	79.8 (<i>d</i>)
C(7)	51.0 (<i>d</i>)	50.2 (<i>d</i>)	131.2 (<i>d</i>)	48.5 (<i>d</i>)
C(8)	90.1 (<i>s</i>)	90.1 (<i>s</i>)	137.1 (<i>s</i>)	89.5 (<i>s</i>)
C(9)	43.0 (<i>d</i>)	42.4 (<i>d</i>)	41.3 (<i>d</i>)	52.7 (<i>d</i>)
C(10)	40.3 (<i>d</i>)	40.4 (<i>d</i>)	41.5 (<i>d</i>)	78.9 (<i>s</i>)
C(11)	48.5 (<i>s</i>)	49.5 (<i>s</i>)	42.2 (<i>s</i>)	55.0 (<i>s</i>)
C(12)	34.0 (<i>t</i>)	35.5 (<i>t</i>)	38.5 (<i>t</i>)	46.1 (<i>t</i>)
C(13)	74.1 (<i>s</i>)	74.0 (<i>s</i>)	75.3 (<i>s</i>)	74.7 (<i>s</i>)
C(14)	78.3 (<i>d</i>)	78.8 (<i>d</i>)	79.2 (<i>d</i>)	78.3 (<i>d</i>)
C(15)	78.7 (<i>d</i>)	78.5 (<i>d</i>)	73.8 (<i>d</i>)	78.8 (<i>d</i>)
C(16)	89.9 (<i>d</i>)	89.6 (<i>d</i>)	92.2 (<i>d</i>)	88.9 (<i>d</i>)
C(17)	57.7 (<i>d</i>)	60.7 (<i>d</i>)	165.0 (<i>d</i>)	57.3 (<i>d</i>)
C(18)	71.2 (<i>t</i>)	72.4 (<i>t</i>)	71.9 (<i>t</i>)	80.0 (<i>t</i>)
C(19)	39.2 (<i>t</i>)	163.0 (<i>d</i>)	52.1 (<i>t</i>)	49.7 (<i>t</i>)
N–CHO	163.1 (<i>d</i>)	–	–	–
MeO–C(1)	55.7 (<i>q</i>)	55.8 (<i>q</i>)	57.0 (<i>q</i>)	55.4 (<i>q</i>)
MeO–C(6)	57.8 (<i>q</i>)	57.4 (<i>q</i>)	58.2 (<i>q</i>)	58.0 (<i>q</i>)
MeO–C(16)	61.0 (<i>q</i>)	61.0 (<i>q</i>)	61.6 (<i>q</i>)	61.2 (<i>q</i>)
MeO–C(18)	58.9 (<i>q</i>)	58.9 (<i>q</i>)	58.7 (<i>q</i>)	59.1 (<i>q</i>)
AcO–C(3)	170.1 (<i>s</i>), 21.0 (<i>q</i>)	170.4 (<i>s</i>), 21.0 (<i>q</i>)	170.1 (<i>s</i>), 21.2 (<i>q</i>)	–
AcO–C(8)	172.2 (<i>s</i>), 21.2 (<i>q</i>)	172.0 (<i>s</i>), 21.2 (<i>q</i>)	–	172.0 (<i>s</i>), 21.2 (<i>q</i>)
COO–C(14)	165.9 (<i>s</i>)	165.9 (<i>s</i>)	166.1 (<i>s</i>)	166.1 (<i>s</i>)
C(1')	129.6 (<i>s</i>)	129.5 (<i>s</i>)	129.7 (<i>s</i>)	129.5 (<i>s</i>)
C(2',6')	129.6 (<i>d</i>)	129.5 (<i>d</i>)	129.9 (<i>d</i>)	129.6 (<i>d</i>)
C(3',5')	128.7 (<i>d</i>)	128.6 (<i>d</i>)	128.5 (<i>d</i>)	128.7 (<i>d</i>)
C(4')	133.4 (<i>d</i>)	133.3 (<i>d</i>)	133.3 (<i>d</i>)	133.4 (<i>d</i>)

^{a)} 125 MHz. ^{b)} 100 MHz.

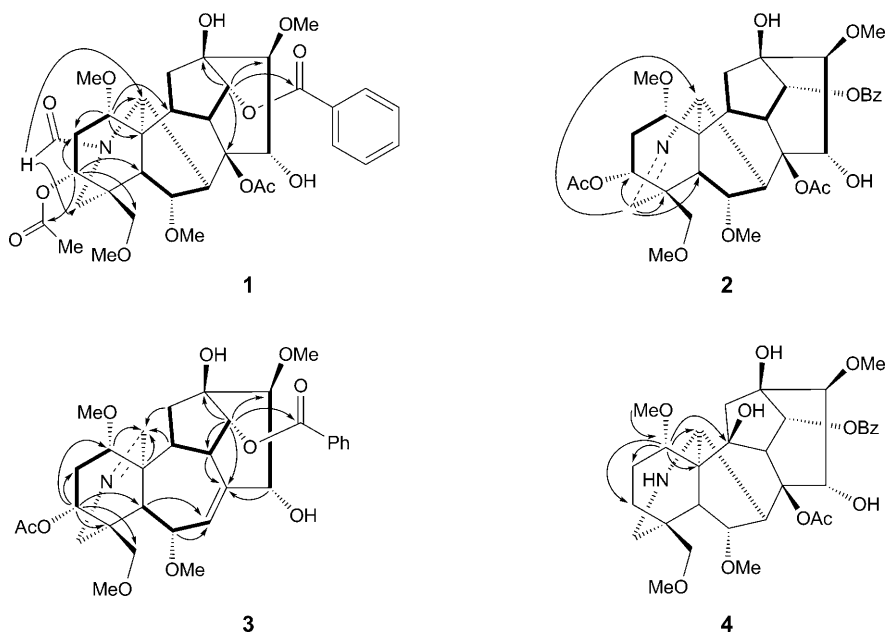


Fig. 2. Selected $^1\text{H},^1\text{H}$ -COSYs (\rightarrow) and HMBCs ($\text{H} \rightarrow \text{C}$) of compounds **1–4**

configuration as 3-*O*-acetylaconitine, based on their almost identical ^1H - and ^{13}C -NMR data (Tables 1 and 2) and the ROESY correlations (Fig. 3). The structure and relative configuration of **1** was confirmed by an X-ray crystallographic analysis (Fig. 4), and thus compound **1** was characterized as 3-*O*-acetyl-20-deethyl-20-formylaconitine (**1**).

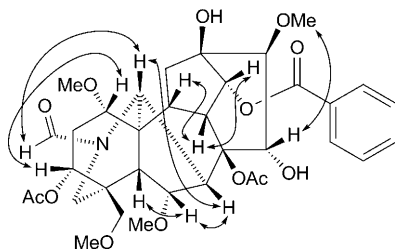


Fig. 3. Selected ROESY correlations of compound **1**

Compound **2** was isolated as colorless prisms and assigned the molecular formula $\text{C}_{34}\text{H}_{43}\text{NO}_{12}$ by analyses of the ESI-MS (m/z 658 ($[M + \text{H}]^+$)) and HR-ESI-MS (m/z 658.2846 ($[M + \text{H}]^+$)). The NMR data of **2** (Tables 1 and 2) were essentially identical with those of compound **1**, suggesting that **2** was also an aconitine-type C_{19} -nor-diterpenoid alkaloid. Compound **2** differed from **1** mainly at C(19) where a $\text{N}=\text{CH}(19)$ moiety was deduced by comparing the NMR data with those of 20-demethyl-19,20-didehydrodelphinine [10]. The presence of a double bond between the N-atom and

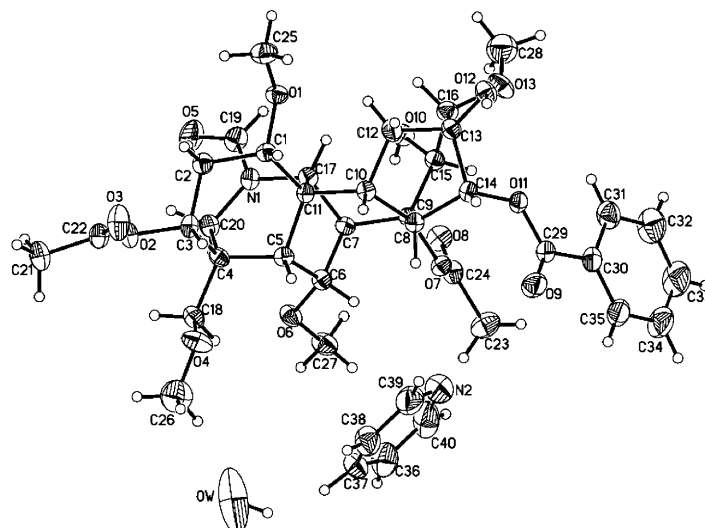


Fig. 4. X-Ray crystal structure of compound **1**. Arbitrary atom numbering.

C(19) was verified by the HMBs (Fig. 2) between the olefinic H–C(19) ($\delta(\text{H})$ 7.37 (br. s)) and C(3), C(4), C(5), C(17), and C(18). Consequently, brachyaconitine B (**2**) was defined as 3-*O*-acetyl-19,29-didehydro-20-deethylaconitine (**2**).

Compound **3** was obtained as a white powder. The molecular formula $\text{C}_{32}\text{H}_{41}\text{NO}_{10}$ was deduced by ESI-MS (m/z 600 ($[M + \text{H}]^+$)) and HR-ESI-MS (m/z 600.2786 ($[M + \text{H}]^+$)). Comparison of its 1D-NMR spectra (Tables 1 and 2) with those of secokaraconitine [11] showed high similarity (karaconitine = (1 α ,2 α ,6 α ,14 α ,15 α ,16 β)-20-ethyl-1,6,16-trimethoxy-4-(methoxymethyl)aconitane-2,8,13,14,15-pentol 8-acetate 14-benzoate), except that there was an additional AcO group in compound **3** ($\delta(\text{H})$ 2.07 (s, 3 H); $\delta(\text{C})$ 170.1 (s) and 21.2 (q)). The AcO group was determined to be linked at C(3) by the HMBs between $\delta(\text{H})$ 4.92 (dd, $J = 15.3, 8.7$, H–C(3)) and $\delta(\text{C})$ 170.1 (AcO, C=O), C(2), C(4), C(5), and C(19). The full NMR data assignments of compound **3** were performed with the aid of the ^1H , ^1H -COSY, HSQC, and HMBC data. Consequently, the structure of compound **3**, named 3-*O*-acetyl-8-de(acetyloxy)-7,8,17,20-tetradecydro-20-deethyl-7,17-secoaconitine (**3**), was elucidated as shown in Fig. 1.

Compound **4** had the molecular formula $\text{C}_{32}\text{H}_{43}\text{NO}_{11}$ as derived from ESI-MS (m/z 618 ($[M + \text{H}]^+$)), HR-ESI-MS (m/z 618.2912 ($[M + \text{H}]^+$)), and the ^{13}C -NMR data (Table 2). Compound **4** had a MeO rather than a OH group at C(1) as deduced from the comparison of its 1D-NMR (Tables 1 and 2) and MS data with those of flavaconitine (= (1 α ,6 α ,14 α ,15 α ,16 β)-6,16-dimethoxy-4-(methoxymethyl)aconitane-1,8,10,13,14,15-hexol 8-acetate 14-benzoate) [6]. The position of this MeO group was established by the correlation of MeO–C(1) to C(1) in the HMBC spectrum (Fig. 2). Hence, compound **4** was defined as 1-*O*-methylflavaconitine (**4**).

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Experimental Part

General. Column chromatography (CC): silica gel (SiO₂; 200–300 mesh, Qingdao Meigao Chemical Ltd., Qingdao, P. R. China); Al₂O₃ (Shanghai Wusi Chemical Reagents Company, Ltd.); Sephadex LH-20 (Pharmacia Fine Chemical Co., Ltd., Germany). M.p.: XRC-1 micro melting-point apparatus; uncorrected. Optical rotations: Horiba-SEPA-300 polarimeter. UV Spectra: Shimadzu-UV-2401A spectrophotometer; λ_{max} (log ε) in nm. IR Spectra: Bio-Rad-FTS-135 spectrometer; ν̄ in cm⁻¹. 1D- and 2D-NMR Spectra: Bruker-AM-400 and -DRX-500 spectrometers; chemical shifts δ in ppm with reference to the solvent signals, J in Hz. EI- and ESI-MS: VG-Autospec-3000 spectrometer at 70 eV; in m/z (rel. %). HR-ESI-MS: API-Qstar-Pulsar-1 spectrometer; in m/z (rel. %).

Plant Material. The roots of *Aconitum brachypodum* DIELS. were collected in Dongchuan of Yunnan Province, P. R. China, in November 2006, and authenticated by Prof. Dr. Li-Gong Lei from Kunming Institute of Botany. A voucher specimen (No. KIB 2006-11-03) had been deposited with the Group of Anti-virus and Natural Medicinal Chemistry, Kunming Institute of Botany, Chinese Academy of Sciences.

Extraction and Isolation. The roots of *A. brachypodum* (50 kg) were powdered and extracted three times with 90% EtOH under reflux for 2 h. After evaporation of the solvent, the crude extract was dissolved in 2% aq. HCl soln. (20 l) and then filtrated. The acidic soln. was basified to pH 9.0 with 25% NH₃ soln. and extracted with CHCl₃ and the org. phase concentrated to furnish a crude alkaloid extract (520 g). The extract was purified by CC (SiO₂ (5.2 kg; 200–300 mesh), petroleum ether/acetone/Et₂NH 15:1:1 → 3:1:1): Fractions A–E. Fr. B (45.1 g) was subjected to CC (SiO₂, petroleum ether/acetone/Et₂NH 15:1:1), followed by CC (Al₂O₃, petroleum ether/acetone 7:1) and finally CC (Sephadex LH-20, CHCl₃/MeOH 1:1): bullatine A (15.3 g), neoline (1.2 g), and songorine (18.5 g). Fr. C (50.9 g) was purified by CC (SiO₂, petroleum ether/acetone/Et₂NH 15:3:1) and further by CC (Al₂O₃, petroleum ether/acetone 5:1): **1** (80 mg), **2** (23 mg), **3** (33 mg), **4** (15 mg), aconitine (0.5 g), and hypaconitine (0.6 g).

Brachyaconitine A (=rel-(1α,3α,6α,14α,15α,16β)-3,8-Bis(acetyloxy)-14-(benzoyloxy)-13,15-dihydroxy-1,6,16-trimethoxy-4-(methoxymethyl)aconitane-2D-carboxaldehyde; **1**): Colorless prisms (pyridine). M.p. 232–233°. [α]_D^{23.0} = –44.35 (c = 3.74, MeOH). UV (MeOH): 230 (4.17). IR (KBr): 3500, 2937, 2825, 1721, 1664, 1602, 1451, 1440, 1279, 713. NMR: Tables 1 and 2. ESI-MS (pos.): 710 ([M + Na]⁺). HR-ESI-MS (pos.): 710.2771 ([M + Na]⁺, C₃₅H₄₅NNaO₁₃; calc. 710.2789).

Brachyaconitine B (=rel-(1α,3α,6α,14α,15α,16β)-19,20-Didehydro-1,6,16-trimethoxy-4-(methoxymethyl)aconitane-3,8,13,14,15-pentol 3,8-Diacetate 14-Benzoate; **2**): Colorless prisms (pyridine). M.p. 150–151°. [α]_D^{23.7} = +69.50 (c = 0.94, MeOH). UV (MeOH): 230 (4.15). IR (KBr): 3500, 2935, 1723, 1640, 1603, 1563, 1452, 1371, 1279, 1106, 712. NMR: Tables 1 and 2. ESI-MS (pos.): 658 ([M + H]⁺). HR-ESI-MS (pos.): 658.2846 ([M + H]⁺, C₃₄H₄₄NO₁₂; calc. 658.2864).

Brachyaconitine C (=rel-(1α,3α,6α,14α,15α,16β)-7,8,17,20-Tetrahydro-1,6,16-trimethoxy-4-(methoxymethyl)-7,17-secoaconitane-3,13,14,15-tetrol 3-Acetate 14-Benzoate; **3**): White powder. M.p. 216–217°. [α]_D^{23.7} = +11.49 (c = 0.24, MeOH). UV (MeOH): 230 (4.19). IR (KBr): 3422, 2933, 1730, 1639, 1602, 1450, 1234, 1101, 719. NMR: Tables 1 and 2. EI-MS: 599 (2, M⁺), 568 (21, [M – MeO]⁺), 540 (37), 105 (100). HR-ESI-MS (pos.): 600.2786 ([M + H]⁺, C₃₂H₄₂NO₁₀; calc. 600.2809).

Brachyaconitine D (=rel-(1α,6α,14α,15α,16β)-1,6,16-Trimethoxy-4-(methoxymethyl)aconitane-8,10,13,14,15-pentol 8-Acetate 14-Benzoate; **4**): Colorless prisms (MeOH). M.p. 165–166°. [α]_D^{22.1} = +23.81 (c = 0.80, MeOH). UV (MeOH): 230 (4.15). IR (KBr): 3497, 2936, 1723, 1603, 1453, 1280, 1098, 716. NMR: Tables 1 and 2. ESI-MS (pos.): 618 ([M + H]⁺). HR-ESI-MS (pos.): 618.2912 ([M + H]⁺, C₃₂H₄₄NO₁₁; calc. 618.2914).

X-Ray Crystal Structure Data of Compound 1. A colorless prismatic crystal was obtained from pyridine. Crystal data: C₃₅H₄₅NO₁₃, M, 687.84; triclinic, space group P1; crystal dimensions: 0.20 × 0.20 ×

0.30 mm; unit-cell dimensions: $a = 8.854(18) \text{ \AA}$, $b = 11.159(2) \text{ \AA}$, $c = 11.544(2) \text{ \AA}$, $\alpha = 106.89(3)^\circ$, $\beta = 105.27(3)^\circ$, $\gamma = 103.63(3)^\circ$, $V = 990.8(3) \text{ \AA}^3$; $Z = 1$; $D_x = 1.315 \text{ g/cm}^3$. Data were collected with a *MAC-DIP-2030K* diffractometer, a graphite monochromator (ω scan, $2\theta_{\text{max}} = 24.0^\circ$), and MoK_α radiation. The total number of independent reflections was 3847, of which 3456 were observed $|F|^2 \geq 2\sigma|F|^2$. The structure was solved by a direct method, with SHELXS-97, expanded with difference Fourier techniques, and refined with NOMCSDP and full-matrix least-squares calculations. Final indices: $R_1 = 0.056$, $wR_2 = 0.145$, $S = 1.327$. CCDC-741409 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/data_request/cif.

REFERENCES

- [1] ‘Zhongyao Da Cidian (The Dictionary of Chinese Crude Drugs)’, Ed. Jiangsu New Medical College, Shanghai Science and Technology Press, Shanghai, China, 1977, pp. 2089–2090.
- [2] China Pharmacopoeia Committee, ‘Pharmacopoeia of China’, People’s Medical Publishing House, Beijing, China, 1977, p. 580.
- [3] G. N. Li, ‘Yunnan Zhiwu Zhi (Chinese Medicine Record of Yunnan)’, Yunnan Science and Technology Press, Yunnan, China, 1990, pp. 472–473.
- [4] L. S. Ding, E. F. Wu, Y. Z. Chen, *Nat. Prod. Res. Dev.* **1994**, *6*, 50.
- [5] S. W. Pelletier, Z. Djarmati, *J. Am. Chem. Soc.* **1976**, *98*, 2626.
- [6] S. Y. Chen, S. H. Li, X. J. Hao, *Acta Bot. Sin.* **1986**, *28*, 86.
- [7] Y. G. Wang, Y. L. Zhu, R. H. Zhu, *Acta Pharm. Sin.* **1980**, *15*, 526.
- [8] H. Hikino, Y. Kuroiwa, C. Konno, *J. Nat. Prod.* **1983**, *46*, 178.
- [9] L. M. Liu, H. C. Wang, Y. L. Zhu, *Acta Pharm. Sin.* **1983**, *18*, 39.
- [10] Y. Bai, H. K. Desai, S. W. Pelletier, *J. Nat. Prod.* **1994**, *57*, 963.
- [11] M. N. Sultankhodzhaev, Atia-tul-Wahab, M. I. Choudhary, Atta-ur-Rahman, *Chem. Nat. Compd.* **2003**, *39*, 512.

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