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Three novel nortriterpenoids from *Notochaete hamosa* Benth. (Labiatae)

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Abstract—Three nortriterpenoids, notohamosin A (**1**), B (**2**) and C (**3**) with novel skeleton, and eight known compounds were isolated from the ethanol extract of the whole plants of *Notochaete hamosa* Benth. (Labiatae). On the basis of spectral evidence including 1D, 2D NMR, IR and MS data, their structures were elucidated. The relative configurations of compounds **1**, **2** and **3** were determined according to NOESY experiments.

1. Introduction

Notochaete hamosa Benth., one of the two plants of the genus *Notochaete* (Labiatae), is distributed in Southwest China, Nepal, India and Burma at an altitude of 1200–2500 m, and usually found in the edge of sub-tropical green-forest.[1,2] No chemical study on the plants of this genus was reported. This study led to the isolation of three new nortriterpenoids, notohamosin A (**1**), B (**2**) and C (**3**) possessing novel skeleton, and eight known compounds, β-sitosterol (**4**),[3] shanzhiside (**5**),[4] daucosterol (**6**),[3] 3,5-dimethoxy-4-hydroxybenzoic acid (**7**),[5] martynoside (**8**),[6] pinoresinol (**9**),[7] desrhamnosyl acteoside (**l0**),[8] and acteoside (**11**).[9] Their structures were elucidated by spectral evidence. The relative configurations of compounds **1**, **2** and **3** were determined by NOESY experiments.

2. Results and discussion

The ethanol extract of the whole plants of *N. hamosa* was divided into petroleum ether-, EtOAc- and *n*-BuOH-soluble fractions. Compounds **1**–**7** were isolated from the EtOAc-soluble extract, **8**–**11** from the *n*-BuOH-soluble fraction by column chromatography.

Notohamosin A (**1**) was obtained as a white powder. The color of **1** in acetic anhydride changed from red to dark-blue after being reacted with Liebermann–Burchard reagent. The molecular formula $C_{29}H_{46}O_5$ was calculated from the quasi-molecular ion peak at *m/z* 475.3395 $[M+H]^+$ in the HRAPCIMS (positive). Twenty-eight signals recognized in the ^{13}C NMR spectrum (DEPT, Table 1) represent 29 C-atoms in view of one overlapped signal which was confirmed by the HMQC cross signals at δ 1.50, 1.35 and 1.90, 1.58/32.7 (t). The ^1H NMR signals at δ 1.13, 0.84, 0.92 and 1.27 (each 3H, s) indicated four methyl groups at quaternary carbon atoms (H_3-25, H_3-26, H_3-27 and H_3-28) (Table 1). Three hydroxymethyl groups located on quaternary C-atoms were postulated from the HMQC cross signals at δ 4.63 and 4.43 (each 1H, d, *J*=10.8 Hz, H_2-23)/68.8 (t, C-23), 4.21 and 4.09 (each 1H, d, *J*=11.0 Hz, H_2-24)/63.5 (t, C-24), and 3.66 and 3.60 (each 1H, d, *J*=10.2 Hz, H_2-29)/70.8 (t, C-29). One Z-double bond (C-11 and C-12) was deduced from ^1H NMR signals at δ 5.73 and 6.00 (each 1H, d, *J*=10.0 Hz). One tri-substituted double bond (C-13 and C-18) was provided by the ^1H NMR signal at δ 5.52 (1H, s) and ^{13}C NMR signals (DEPT) at δ 136.1 (d) and 139.1 (s). The evidence mentioned above led to the conclusion that compound **1** is a nortriterpene.

Four moieties a, b, c and d were established from ^1H NMR and ^{13}C NMR spectral data, and HMQC and HMBC experiments (Table 1 and Fig. 1). The quaternary carbon atom resonated at δ 44.9 (s) could be assigned to C-17 according to the ^1H NMR signal at δ 5.52 (1H, s) for H-18. Thus, skeleton **1A** (Fig. 1) was determined.

Another two hydroxyl groups, concluded from molecular formula, could be located at C-2 and C-3 according to the HMQC correlation δ 4.50 (1H, brd, *J*=11.2 Hz, H-2)/66.1

Keywords: *Notochaete hamosa* Benth; nortriterpeonids; notohamosin A; notohamosin B; notohamosin C.
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Table 1. NMR data of compound **1** in pyridine-d_5

	δ_H[a,b]	δ_C[b,c]	HMBC (selected)	NOESY (selected)
CH_2(1)	2.27 (1H, dd, 11.2, 3.6), 1.92 (1H, m)	42.7 (t)		
H–C(2)	4.50 (1H, brd, 11.2)	66.1 (d)		H_2-24, H_3-25
H–C(3)	4.91 (1H, s)	73.7 (d)	C-1, C-2, C-5	
C(4)		47.5 (s)		
H–C(5)	2.23 (1H, brd, 12.4)	44.4 (d)	C-4, C-6, C-24, C-25	H-9, H_2-23
CH_2(6)	1.01 (m), 0.82 (m)	18.9 (t)		
CH_2(7)	1.50 (m), 1.35 (m)	32.7 (t)		
C(8)		41.0 (s)		
H–C(9)	2.37 (1H, s)	54.7 (d)	C-10, C-25, C-11, C-26	H-5, H_3-27
C(10)		38.0 (s)		
H–C(11)	5.73 (1H, d, 10.0)	125.9 (d)	C-8, C-10, C-13	H-12
H–C(12)	6.00 (1H, d, 10.0)	130.7 (d)	C-9, C-14, C-18	H-11, H-18
C(13)		139.1 (s)		
C(14)		40.4 (s)		
CH_2(15)	1.66 (m), 1.03 (m)	26.3 (t)		
CH_2(16)	1.90 (m), 1.58 (m)	32.7 (t)		
C(17)		44.9 (s)		
H–C(18)	5.52 (1H, s)	136.1 (d)	C-12, C-16, C-19, C-22	H-12, Ha-19
CH_2(19)	(a) 1.97 (1H, d, 13.2) (b) 1.37 (1H, d, 13.2)	52.0 (t)		H-18, H_2-29
C(20)		44.6 (s)		
CH_2(21)	1.97 (m), 1.50 (m)	35.6 (t)		
CH_2(22)	1.68 (m), 1.60 (m)	39.6 (t)		H_3-27
CH_2(23)	4.63 (1H, d, 10.8) 4.43 (1H, d, 10.8)	68.8 (t)	C-3, C-4, C-5, C-24	H-5
CH_2(24)	4.21 (1H, d, 11.0) 4.09 (1H, d, 11.0)	63.5 (t)	C-3, C-4, C-23	H-2, H_3-25
Me(25)	1.13 (3H, s)	19.2 (q)	C-1, C-10, C-9, C-5	H-2, H_2-24, H_3-26
Me(26)	0.84 (3H, s)	16.7 (q)	C-9, C-8, C-14, C-7	H_3-25
Me(27)	0.92 (3H, s)	20.0 (q)	C-13, C-14, C-15	H-9, H_2-22
Me(28)	1.27 (3H, s)	26.9 (q)	C-19, C-20, C-21, C-29	
CH_2(29)	3.66 (1H, d, 10.2) 3.60 (1H, d, 10.2)	70.8 (t)	C-19, C-21, C-28, C-20	Ha-19

[a] *J* in Hz.
[b] Signals were assigned by HMQC, HMBC and COSY experiments, δ in ppm.
[c] Multiplicity were determined by DEPT.

(d, C-2) and 4.91 (1H, s, H-3)/73.7 (d, C-3) and HMBC correlations between H-3 and C-1, C-2 and C-5. The relative configurations of C-2, C-3, C-5, C-8, C-9, C-10, C-14, C-17 and C-20 were determined by NOESY experiments (Fig. 1 and Table 1). The 2-OH group should be α-orientated considering the NOESY correlations between H-2 and H_3-25, and between H-2 and H_2-24. The negligible coupling constant between H-2 and H-3 revealed 3α-OH. The NOESY correlations between H-5 and H-9, between H-5 and H_2-23, and between H-9 and H_3-27 indicated α-oriented H-5, H-9 and H_3-27. The relative configuration of C-17 was determined upon the NOESY cross signals between H_3-27 and H_2-22. The NOESY correlation between δ_H 5.52 (H-18) and δ_H 1.97 suggested that the proton resonated at δ_H 1.97 should be α-oriented (H-19a). The key NOESY correlation between H-19a and H_2-29 suggested 20α-CH_2OH group (C-29). Thus, the structure of notohamosin A (**1**) was established as that illustrated in Figure 1.

The visualization of notohamosin **B** (**2**) with Liebermann–Buchard reagent was the same as that of **1**. The quasi-molecular ion peak at *m/z* 459.3454 in the HRAPCIMS (positive) of **2** provided the molecular formula $C_{29}H_{46}O_4$. Twenty-nine signals observed in the ^{13}C NMR spectrum, five methyl groups and two hydroxymethyl groups at quaternary C-atoms recognized from the ^1H NMR spectrum suggested that **2** is also a nortriterpene (Fig. 1 and Table 2). Comparing the NMR data and MS data of **2** with those of **1**, it is obvious that **2** bears one more methyl group and one less hydroxymethyl group than **1**. C-24 of **2** was not substituted by a hydroxyl group, which was confirmed by HMQC correlation at δ 0.90 (3H, s, H_3-24)/17.1 (q, C-24), HMBC cross signals between H_3-24 and C-4, C-3 and C-23 (Table 2), as well as NOESY correlations between H_3-24, H-2 and H_3-25 (Fig. 1 and Table 2). **2A** (Fig. 1) was postulated on the basis of HMQC and HMBC experiments. The relative configurations of C-2, C-3, C-4, C-5, C-8, C-9, C-10, C-14, C-17 and C-20 were determined to be the same as those in compound **1** based on the NOESY experiments (Fig. 1 and Table 2). Thus, the structure of **2** (notohamosin **B**) could be determined as that depicted in Figure 1.

Notohamosin C (**3**) was isolated as a white powder. In the ^1H NMR spectrum four methyl groups at quaternary C-atom (H_3-25, H_3-26, H_3-27 and H_3-28) resonated at δ 1.16, 1.00, 1.02 and 1.04 (each 3H, s) (Table 3). The ^1H NMR signals at δ 4.59 and 4.40 (each d, J=10.5 Hz), 4.22 and 4.07 (each d, J=11.0 Hz), as well as ^{13}C NMR signals at δ 69.2 (t) and 64.2 (t) (Table 3), could be assigned to two hydroxymethyl groups at quaternary C-atom. One methoxy group was deduced from HMQC signal at δ 3.36 (3H, s)/54.7 (q). One tri-substituted double bond (C-12 and C-13) was suggested by the ^1H NMR signal at δ 6.07 (IH, t, J=2.0 Hz) and ^{13}C

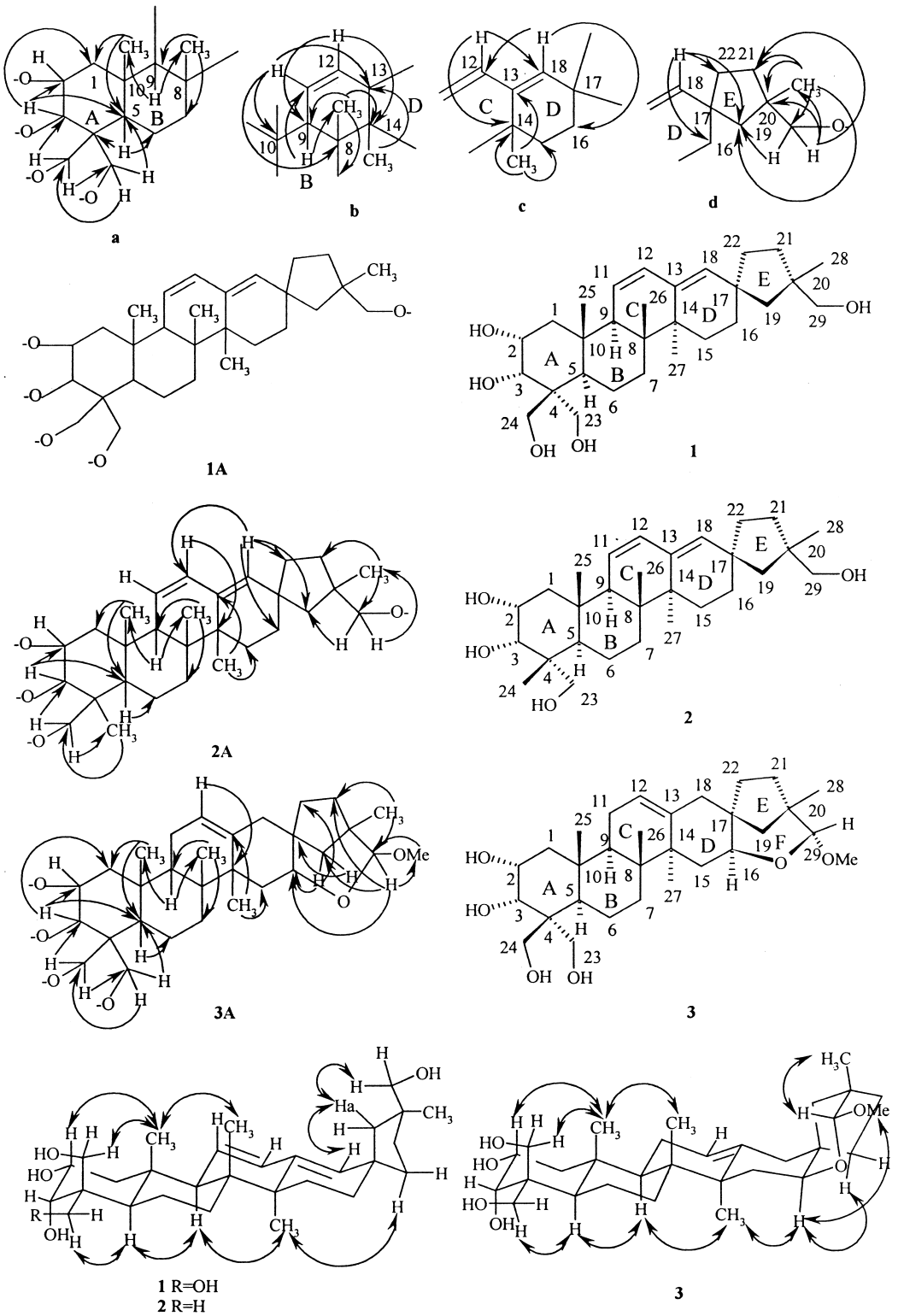

Figure 1. Structures of **1**, **2** and **3**, and major HMBC (→) and key NOESY correlation (↔).

NMR signals (DEPT) at δ 119.2 (d) and 138.5 (s). The quasi-molecular ion peak $[M+H]^+$ at *m/z* 505.3515 in HRAPCIMS gave the molecular formula $C_{30}H_{48}O_6$. From ^{1}H NMR, ^{13}C NMR, HMBC and HMQC (Table 3), structure **3A** (Fig. 1) was concluded.

The ^{13}C NMR signal at δ 74.6 (d) could be assigned to oxygenated C-16 in view of the HMQC correlation at δ 4.24 (1H, brs, H-16)/74.9 (d, C-16) and HMBC cross signal at δ 1.06 (1H, d, J=11.0 Hz, Hb-19)/74.9 (d, C-16). The HMQC signal at δ 4.33 (1H, s, H-29)/106.8 (d, C-29) suggested that C-29 should be an acetal carbon atom. From the HMBC correlation between H-29 and C-16 and the molecular formula of compound **3**, it could be

Table 2. NMR data of compound **2** in pyridine-d_5

	δ_H[a,b]	δ_C[b,c]	HMBC (selected)	NOESY (selected)
CH_2(1)	2.22 (1H, dd, 12.0, 4.0) 1.86 (1H, t, 12.0)	42.3 (t)		
H–C(2)	4.34 (1H, brd, 12.0)	66.0 (d)		H_2-24, H_3-25
H–C(3)	4.17 (1H, s)	78.8 (d)	C-1, C-2, C-5	
C(4)		41.8 (s)		
H–C(5)	2.21 (1H, brd, 11.6)	43.3 (d)	C-4, C-6, C-24, C-25	H-9, H_2-23
CH_2(6)	1.02 (m), 0.82 (m)	18.2 (t)		
CH_2(7)	1.48 (m), 1.34 (m)	32.1 (t)		
C(8)		41.0 (s)		
H–C(9)	2.33 (1H, s)	54.5 (d)	C-10, C-25, C-11, C-26	H-5, H_3-27
C(10)		38.0 (s)		
H–C(11)	5.71 (1H, d, 10.0)	130.7 (d)	C-8, C-10, C-13	H-12
H–C(12)	6.00 (1H, d, 10.0)	125.9 (d)	C-9, C-14, C-18	H-11, H-18
C(13)		139.0 (s)		
C(14)		40.4 (s)		
CH_2(15)	1.66 (m), 1.03 (m)	26.3 (t)		
CH_2(16)	1.73 (m), 1.65 (m)	32.7 (t)		
C(17)		44.9 (s)		
H–C(18)	5.53 (1H, s)	136.1 (d)	C-12, C-16, C-19, C-22	H-12, Ha-19
CH_2(19)	(a) 1.93 (1H, d, 13.2) (b) 1.35 (1H, d, 13.2)	52.0 (t)		H-18, H_2-29
C(20)		44.6 (s)		
CH_2(21)	2.00 (m), 1.55 (m)	35.6 (t)		
CH_2(22)	1.68 (m), 1.60 (m)	39.6 (t)		H_3-27
CH_2(23)	3.91 (1H, d, 10.8) 3.77 (1H, d, 10.8)	71.1 (t)	C-3, C-4, C-5, C-24	H-5
Me(24)	0.90 (3H, s)	17.1 (q)	C-3, C-4, C-5, C-23	H-2, H_3-25
Me(25)	1.13 (3H, s)	19.4 (q)	C-1, C-10, C-9, C-5	H-2, H_2-24, H_3-26
Me(26)	0.85 (3H, s)	16.8 (q)	C-7, C-8, C-9, C-14	H_3-25
Me(27)	0.93 (3H, s)	20.0 (q)	C-13, C-14, C-15	H-9, H_2-22
Me(28)	1.27 (3H, s)	26.8 (q)	C-19, C-20, C-21, C-29	
CH_2(29)	3.66 (1H, d, 10.0) 3.60 (1H, d, 10.0)	70.6 (t)	C-19, C-20, C-21, C-28	Ha-19

[a] *J* in Hz.
[b] Signals were assigned by HMQC, HMBC and COSY experiments, δ in ppm.
[c] Multiplicity were determined by DEPT.

concluded that C-16 and C-29 should be connected via an ether bond. The assignment of ring F was then achieved according to the key HMBC correlations derived from H-29 with C-16, C-19 and 29-O*C*H_3, from H_3-28 with C-19, C-20, C-21 and C-29, and from H-19 with C-16, C-17, C-20, C-21 and C-29.

C-2 and C-3 were found to be substituted by hydroxyl groups by comparing its ^{13}C NMR data with those of **1**. The relative configurations of C-2, C-3, C-4, C-5, C-8, C-9, C-10 and C-14 were determined to be the same as those in compound **1** based on the NOESY experiments (Fig. 1 and Table 3). H-16 and 29-OCH_3 should be α-oriented in view of NOESY correlations between H-16 and H_3-27, and between H-16 and 29-OMe (Fig. 1 and Table 3). The relative configuration of C-17 was assigned by NOESY correlation between H-16 and Ha-22. The relative configuration of C-29 could be determined by taking the rigidities of rings D, E and F into account, which was confirmed by the key NOESY correlation between H-29 and H_3-28. Therefore, the structure of compound **3** was determined as shown in Figure 1.

The known compounds, β-sitosterol (**4**),[3] shanzhiside (**5**),[4] daucosterol (**6**),[3] 3,5-dimethoxy-4-hydroxybenzoic acid (**7**),[5] martynoside (**8**),[6] pinoresinol (**9**),[7] desrhamnosyl acteoside (**10**),[8] and acteoside (**11**)[9] were identified by co-TLC with authentic samples and by comparison of their spectral data with those reported.

3. Experimental

3.1. General

Melting points were determined on an XRC-1 micro-melting point apparatus and are uncorrected. Optical rotations were measured on a Perkin–Elmer 241 automatic polarimeter. IR spectra were recorded on a Nicolet Protege 460 spectrometer using KBr disc and ν_{max} are given in cm^{-1}. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were recorded at room temperature with a Brucker Avance 500 spectrometer. The chemical shifts (δ) are given in ppm and the coupling constants (*J*) are given in Hz. HRAPCIMS were obtained on a BRUCKER FTMS APEX3 mass spectrometer. FABMS were obtained on a VG AutoSpec-3000 (glycerol as matrix) mass spectrometer. ESIMS were carried out on a Finnigan LCQ^{DECA} mass spectrometer. EIMS were obtained on a VG 7070E (70 eV) mass spectrometer. Column Chromatography (CC) was performed on self-packed open column with 200–300 mesh of silica gel purchasing from Qingdao Ocean Chemical Engineering Company (QOCEC). Thin Layer Chromatography (TLC) analyses were made on plates precoated with

Table 3. NMR data of compound **3** in pyridine-d_5

	δ_H[a,b]	δ_C[b,c]	HMBC (selected)	NOESY (selected)
CH_2(1)	2.06 (1H, dd, 11.0, 2.0) 1.86 (1H, m)	43.4 (t)		
H–C(2)	4.44 (1H, dt, 11.0, 2.0)	66.4 (d)		H_2-24, H_3-25
H–C(3)	4.85 (1H, d, 2.0)	73.8 (d)	C-1, C-5, C-4, C-2, C-24	
C(4)		47.6 (s)		
H–C(5)	2.15 (1H, brd, 11.5)	44.9 (d)	C-23, C-24, C-25, C-4, C-10, C-7, C-6	H-9, H_2-23
CH_2(6)	1.87 (m), 1.65 (m)	19.0 (t)		
CH_2(7)	1.65 (m), 1.42 (m)	34.5 (t)		
C(8)		39.8 (s)		
H–C(9)	1.83 (1H, s)	48.0 (d)	C-10, C-25, C-26	H-5, H_3-27
C(10)		38.3 (s)		
CH_2(11)	2.04 (2H, m)	23.4 (t)		
H–C(12)	6.07 (1H, t, 2.0)	119.2 (d)		
C(13)		138.5 (s)		
C(14)		44.2 (s)		
CH_2(15)	1.56 (m), 0.98 (m)	27.6 (t)		
H–C(16)	4.24 (1H, brs)	74.9 (d)		H_3-27, Ha-22, H–OMe
C(17)		46.4 (s)		
CH_2(18)	1.82 (m), 1.01 (m)	28.8 (t)		
CH_2(19)	(a) 1.94 (1H, d, 11.0) (b) 1.06 (1H, d, 11.0)	44.9 (t)	C-16, C-17, C-18, C-20, C-21, C-22, C-28, C-29	H-16
C(20)		43.7 (s)		
CH_2(21)	(a) 1.72 (1H, dd, 12.5, 4.0) (b) 1.30 (1H, dd, 12.5, 4.0)	36.0 (t)		
CH_2(22)	(a) 1.63 (1H, d, 13.0) (b) 1.20 (1H, d, 13.0)	31.2 (t)		H-16
CH_2(23)	4.59 (1H, d, 10.5) 4.40 (1H, d, 10.5)	69.2 (t)	C-3, C-4, C-5, C-24	H-5
CH_2(24)	4.22 (1H, d, 11.0) 4.07 (1H, d, 11.0)	64.2 (t)	C-3, C-4, C-5, C-23	H-2, H_3-25
Me(25)	1.16 (3H, s)	17.4 (q)	C-1, C-10, C-9, C-5	H-2, H_2-24, H_3-26
Me(26)	1.00 (3H, s)	17.7 (q)	C-9, C-8, C-14, C-7	H_3-25
Me(27)	1.02 (3H, s)	22.9 (q)	C-13, C-14, C-15	H-9, H-16
Me(28)	1.04 (3H, s)	21.8 (q)	C-19, C-20, C-21, C-29	H-29
H–C(29)	4.33 (1H, s)	106.8 (d)	C-16, C-19, C–OMe	H_3-28
–OMe	3.36 (3H, s)	54.7 (q)	C-29	H-16

[a] J in Hz.
[b] Signals were assigned by HMQC, HMBC and COSY experiments, δ in ppm.
[c] Multiplicity were determined by DEPT.

10–40 μm of silica gel G purchasing from QOCEC. Visualization on TLC was carried out by spraying 8% phosphomolybdic acid–ethanol solution (w/v) followed by heating. Fractions from all columns were generally collected by hand and auto-collect apparatus according to TLC analyses.

3.2. Plant material

The whole plants of *N. hamosa* Benth. were collected from Wuliangshan, Jingdong County, Yunnan Province, China, in September 1999, and identified by Professor X. W. Li (Kunming Institute of Botany, the Chinese Academy of Sciences). A voucher specimen (GF-05) is deposited at the Herbarium of Chengdu Institute of Biology, the Chinese Academy of Sciences.

3.3. Extraction and isolation

A sample of cut and dried whole plants (7.2 kg) was soaked with 92% ethanol (50 L×3, seven days each time) at room temperature. After being concentrated in vacuum, ca. 990 g residue was obtained. The syrup was dissolved in warm water 2.0 L (about 50°C), cooled to room temperature and successively extracted with petroleum ether (60–90°C) (1.0 L×10), ethyl acetate (1.0 L×10) and *n*-BuOH (0.5 L×12).

The EtOAc-partitionated extract (50 g) was divided into five fractions FrB1-5 by CC gradient eluted with $CHCl_3$/MeOH (from 20:1, 10:1 to 5:1, elute was combined after TLC analyses). Compounds **1** (25 mg) and **2** (22 mg) were obtained from FrB2 by CC repeatedly eluted with petroleum ether (60–90°C):acetone (4:1). **3** (18 mg) was isolated from FrB3 by CC eluted with $CHCl_3$:MeOH (15:1). FrB1 was subjected to CC repeatedly eluted with petroleum ether (60–90°C):acetone (4:1) to give compounds **4** (108 mg) and **5** (12 mg). Compound **6** (1.08 g) was precipitated from FrB5. Compound **7** (36 mg) was isolated from FrB4 by CC eluted with $CHCl_3$:MeOH (15:1). The *n*-BuOH extract (161 g) was dissolved in 1.2 L of warm water (about 50°C) and absorbed on CC packed with macroporus resin (D_{101}). The CC was eluted by water until no sugar in the elute was detected, then eluted by MeOH to yield 43 g residue, which was divided into four fractions FrC1-4 by CC eluted with $CHCl_3$:MeOH:acetone (4:1:1). Compound **8** (23 mg) was obtained from FrC1 by CC eluted with $CHCl_3$:MeOH (5:1). Compound **9** (18 mg) were isolated from FrC2 by CC eluted with $CHCl_3$:MeOH:H_2O (4:1:0.1). FrC3 was first separated by CC with $CHCl_3$:MeOH:H_2O (4:1:0.1), then separated by

CC packed with C-18 bonded silica gel with the elution of acetonitrile and water (1:3) to yield compounds **10** (67 mg) and **11** (44 mg).

3.3.1. Notohamosin A (1). White powder; mp 298–300°C ($CHCl_3$:CH_3OH (10:1)); $[\alpha]_D^{20}=-54$ (*c* 0.001, MeOH: pyridine (10:1)); IR ν_{max} (KBr): 3385, 2940, 2857, 1638, 1448, 1378 and 1033 cm^{-1}; HRAPCIMS (positive) *m/z*: 475.3395 ($[M+H]^+$, $C_{29}H_{47}O_5$, calcd: 475.3418); FABMS (negative) *m/z* (rel. int): 565 (46, $[M+glycerol-H]^-$) and 473 (100, $[M-H]^-$); ESIMS (positive) *m/z* (rel. int): 971 (76, $[2M+Na]^+$) and 497 (100, $[M+Na]^+$); ESIMS (negative) *m/z* (rel. int): 983 (30, $[2M+Cl]^-$), 509.6 (22, $[M+Cl]^-$) and 473 (100, $[M-H]^-$); EIMS *m/z* (rel int): 474 (16, $[M]^+$), 456 (21, $[M-H_2O]^+$), 438 (14, $[M-2\times H_2O]^+$), 325 (100); NMR data, see Table 1.

3.3.2. Notohamosin B (2). White powder; mp 284–286°C ($CHCl_3$:CH_3OH (10:1)); $[\alpha]_D^{20}=-25$ (*c* 0.001, MeOH: pyridine (10:1)); IR ν_{max} (KBr): 3424, 2927, 2857, 1638, 1459 and 1041 cm^{-1}; HRAPCIMS (positive) *m/z*: 459.3454 ($[M+H]^+$, $C_{29}H_{47}O_4$, calcd: 459.3469); FABMS (negative) *m/z* (rel. int): 549 (46, $[M+glycerol-H]^-$) and 457 (100, $[M-H]^-$); ESIMS (positive) *m/z* (rel. int): 939 (100, $[2M+Na]^+$) and 481 (70, $[M+Na]^+$); ESI-MS (negative) *m/z* (rel. int): 951 (10, $[2M+Cl]^-$), 915 (20, $[2M-H]^-$) and 458 (100, $[M]^-$); EIMS *m/z* (rel. int): 458 (27, $[M]^+$), 440 (19, $[M-H_2O]^+$), 325 (100); NMR data, see Table 2.

3.3.3. Notohamosin C (3). White powder; mp 272–274°C ($CHCl_3$:CH_3OH (10:1)); $[\alpha]_D^{20}=+44.4$ (*c* 0.064, MeOH: pyridine (10:1)); IR ν_{max} (KBr): 3424, 2923, 2855, 1638, 1457, 1377 and 1042 cm^{-1}; HRAPCIMS (positive) *m/z*: 505.3515 ($[M+H]^+$, $C_{30}H_{49}O_6$, calcd: 505.3524); FABMS (negative) *m/z* (rel. int): 595 (30, $[M+glycerol-H]^-$) and 503 (100, $[M-H]^-$); ESIMS (positive) *m/z* (rel. int): 1031 (60, $[2M+Na]^+$) and 527 (100, $[M+Na]^+$); ESIMS (negative) *m/z* (rel. int): 1043 (10, $[2M+Cl]^-$),1007 (16, $[2M-H]^-$), 539.9 (10, $[M+Cl]^-$) and 503 (100, $[M-H]^-$); EIMS *m/z* (rel.int): 235 (100); NMR data, see Table 3.

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