Nortriterpenoids from *Chukrasia tabularis* var. *velutina*

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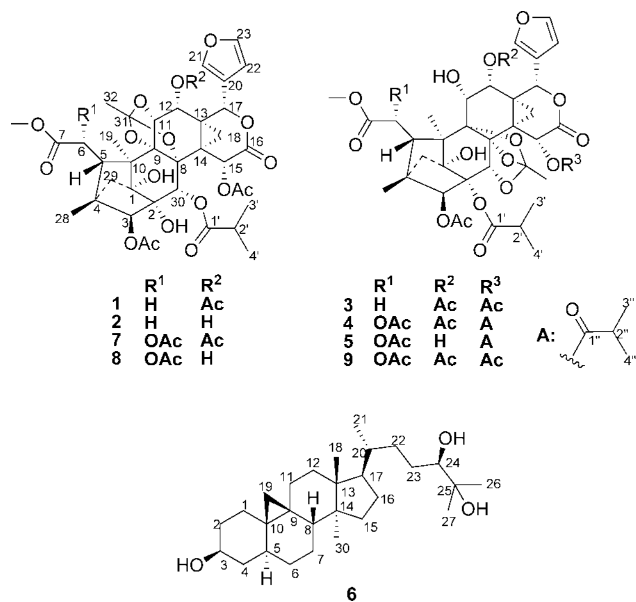
Received July 14, 2007

Five new limonoids, tabularisins E–I (**1–5**), and a new dinorcycloartane, (24*R*)-28,29-dinor-cycloartane-3β,24,25-triol (**6**), together with three known compounds, were isolated from the twigs and leaves of *Chukrasia tabularis* var. *velutina*. Their structures were elucidated primarily on the basis of spectroscopic methods.

Plants belonging to the Maliaceae family are rich sources of structurally diverse and biologically significant limonoids.[1] Previous chemical investigations on the genus *Chukrasia* led to the isolation of a number of phragmalin-type limonoids.[2] *C. tabularis* var. *velutina* (Wall.) King (Maliaceae), a timber tree that grows mainly in tropical areas of Asia such as India and southern China,[3] is known to contain β-sitosterol, daucosterol, and some aliphatic compounds.[4] In the current study, five new limonoids (**1–5**) and a new dinorcycloartane (**6**), as well as three known compounds, tabularisins A–C (**7–9**),[2d] were isolated from an ethanolic extract of the twigs and leaves of *C. tabularis* var. *velutina* collected in Xishuangbanna, Yunnan Province, China. We report herein the isolation and structural elucidation of these new compounds.



Results and Discussion

Tabularisin E (**1**), a white, amorphous powder, possessed a molecular formula of $C_{39}H_{46}O_{18}$ (calcd 802.2684) as determined by HREIMS ([M]$^+$, *m/z* 802.2700) and supported by NMR data (Tables 1 and 2). Strong IR absorptions showed the presence of OH (3435 cm^{-1}) and carbonyl groups (1730 and 1759 cm^{-1}). The ^{1}H (Table 1) and ^{13}C NMR (Table 2) data indicated the existence of one orthoacetate, one methoxyl, one isobutyryloxyl, three acetoxyl, two additional carbonyl groups that came from two esters, and one β-furyl ring. These functionalities accounted for 9 out of the 17 degrees of unsaturation, and the remaining 8 degrees of unsaturation were thus required for the presence of an octacyclic core in **1**. Two proton signals at δ 2.82 and 3.36 (s, each ca. 1H) showed no correlation with any carbon signals in the HSQC spectrum and were assigned to exchangeable OH protons. The aforementioned data and extensive analyses of its 1D NMR spectra indicated that compound **1** was a phragmalin-type limonoid.[2,5]

The structure of **1** was further demonstrated by analysis of 2D NMR spectra, especially HMBC (Figure 1). Two proton resonances at δ 1.42 (brd, J = 7.0 Hz) and 2.64 (dd, J = 7.0 and 3.0 Hz) belonging to the methylene carbon C-18 (δ 18.6) showed correlations with C-13 (δ 31.0) and C-14 (δ 30.7), respectively, indicating that C-18 linked with both C-13 and C-14 to form a cyclopropyl ring similar to that of tabularisins A–C (**7–9**).[2d] The assignments of three acetoxyls to C-3 (δ 85.7), C-12 (δ 66.4), and C-15 (δ 69.7) were accomplished by the HMBC correlations from H-3 (δ 5.50, s), H-12 (δ 5.13, d, J = 3.1 Hz), and H-15 (δ 7.18, d, J = 3.0 Hz) to the corresponding carbonyls of three acetoxyl groups. An ester carbonyl carbon at δ 166.9 correlated with H-15 was assignable to C-16. The HMBC correlations from H-17 (δ 6.42, s) to C-21 (δ 142.0) and C-22 (δ 109.6) revealed that the β-furyl group was attached to C-17 (δ 71.4). Two proton signals of H_2-6 at δ 2.65 (brd, J = 16.5 Hz) and 2.43 (dd, J = 16.5, 12.2 Hz) showed HMBC correlations with C-4 (δ 44.8), C-5 (δ 38.0), C-7 (δ 173.7), and C-10 (δ 43.9), indicating attachment of the C-6–C-7-unit at C-5. The only methoxyl was linked to C-7 by the HMBC correlation between *Me*O and C-7. The attachment of an isobutyryloxyl at C-30 (δ 70.1) was revealed by the HMBC correlation from H-30 (δ 5.39, s) to its carbonyl carbon. Two proton resonances assignable to OH groups displayed HMBC correlations with C-1 (δ 82.8) and C-2 (δ 76.5), respectively, indicating OH groups at these positions. The remaining three oxygenated carbons were assigned to C-8 (δ 78.2), C-9 (δ 90.4), and C-11 (δ 74.8) by the mutual HMBC correlations of Me-19/C-9, H-30/C-8, H-30/C-9, H-11/C-8, H-11/C-9, H-11/C-12, and H-11/C-13, indicating the presence of an 8,9,11-orthoacetate[2d] though no direct HMBC correlations are available.

The relative configuration of **1** was fixed using a ROESY experiment (Figure 1). The ROESY cross-peaks of H-5/H-12, H-5/H-17, H-5/H-15, H-15/3-OAc, H-17/3-OAc, H-17/H-12, H-17/H-15, and H-15/H-30 indicated that H-5, 3-OAc, H-12, H-17, H-15, and H-30 are co-facial and randomly assigned as β-oriented. The ROESY correlation between H-18b (δ 1.42) and H-22 (δ 6.49, brd, J = 1.7 Hz) suggested that the cyclopropyl group and the β-furyl ring were α-oriented. ROESY correlations of Me-32/Me-3′, Me-32/H-18a (δ 2.64), Me-4′/1-OH, and Me-4′/2-OH revealed that the 8,9,11-orthoacetate, 30-isobutyryloxyl, 1-OH, and 2-OH were α-oriented. Furthermore, Me-19 and the CH_2-29 group were assigned to be α-oriented on the basis of ROESY correlations of H-11/Me-19, Me-19/H-29a, and H-3/H-29b. Thus, the structure of tabularisin E (**1**) was elucidated.

Tabularisin F (**2**) had the molecular formula $C_{37}H_{44}O_{17}$ as determined by HREIMS ([M]$^+$, *m/z* 760.2579). Direct comparison

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10.1021/np070345w CCC: $37.00 © 2007 American Chemical Society and American Society of Pharmacognosy
Published on Web 10/12/2007

Table 1. ^{1}H NMR Data of **1**–**5**[a]

proton	**1** (mult., *J* in Hz)	**2** (mult., *J* in Hz)	**3** (mult., *J* in Hz)	**4** (mult., *J* in Hz)	**5** (mult., *J* in Hz)
3	5.50 s	5.50 s	5.30 s	5.28 s	5.27 s
5	2.58 brd (12.2)	2.50 brd (12.4)	2.44 brd (12.2)	2.78 s	2.66 s
6	a 2.65 brd (16.5)	a 2.48 brd (16.2)	a 2.87 brd (17.1)	6.16 s	5.36 s
	b 2.43 dd (16.5, 12.2)	b 2.42 dd (16, 2, 12.4)	b 2.32 dd (17.1, 12.2)		
11	4.17 d (3.1)	4.22 d (4.2)	4.22 d (3.9)	4.30 d (4.7)	4.18 d (5.0)
12	5.13 d (3.1)	3.86 brs	5.19 d (3.9)	5.35 d (4.7)	4.10 brs
15	7.18 d (3.0)	7.17 d (2.8)	7.03 d (2.6)	6.88 d (2.4)	6.84 d (2.4)
17	6.42 s	6.43 s	6.41 s	6.40 s	6.39 s
18a	2.64 dd (7.0, 3.0)	2.47 dd (6.8, 2.8)	2.94 dd (6.9, 2.6)	2.93 dd (6.8, 2.4)	2.59 dd (6.7, 2.4)
18b	1.42 brd (7.0)	1.36 brd (6.8)	1.49 brd (6.9)	1.47 brd (6.8)	1.36 brd (6.7)
19	1.30 s	1.32 s	1.30 s	1.29 s	1.29 s
21	7.46 brs	7.66 brs	7.54 dd (1.6, 0.7)	7.53 brs	7.69 dd (1.7, 0.8)
22	6.49 brd (1.7)	6.55 brd (1.2)	6.54 dd (1.7, 0.7)	6.53 brd (1.5)	6.53 brt (0.8)
23	7.38 brs	7.51 brt (1.5)	7.40 brt (1.6)	7.40 dd (1.5, 0.6)	7.46 brt (1.7)
28	0.82 s	0.80 s	0.75 s	0.92 s	0.90 s
29a	1.92 d (11.6)	1.92 d (10.0)	1.99 d (11.4)	2.26 d (11.3)	2.20 d (11.1)
29b	1.89 d (11.6)	1.89 d (10.0)	1.67 d (11.4)	1.69 d (11.3)	1.69 d (11.1)
30	5.39 s	5.39 s	5.05 s	4.90 s	4.89 s
32	1.66 s	1.67 s	1.73 s	1.71 s	1.71 s
2′	2.49–2.56 m	2.49–2.56 m	2.60–2.67 m	2.59–2.66 m	2.59–2.66 m
2″				2.68–2.75 m	2.66–2.73 m
3′	1.20 d (6.9)	1.20 d (7.5)	1.20 d (7.0)	1.18 d (7.0)	1.17 d (7.0)
3″				1.24 d (7.0)	1.23 d (7.0)
4′	1.18 d (6.9)	1.18 d (7.5)	1.18 d (7.0)	1.15 d (7.0)	1.15 d (7.0)
4″				1.19 d (7.0)	1.19 d (7.0)
1-OH	2.82 s	2.89 s	3.56 s	3.56 s	3.61 s
2-OH	3.36 s	3.37 s			
11-OH			2.11 s	2.11 s	2.43 brs
12-OH		not observed			2.43 brs
3-OAc	2.20 s	2.20 s	2.34 s	2.35 s	2.34 s
6-OAc				2.19 s	2.19 s
12-OAc	1.66 s		1.64 s	1.66 s	
15-OAc	2.33 s	2.33 s	2.20 s		
–OCH$_3$	3.75 s	3.76 s	3.75 s	3.78 s	3.79 s

[a] Data measured in CDCl$_3$ at 400 MHz; chemical shifts are expressed in ppm; the spin coupling (*J*) is given in parentheses (Hz).

of the ^{1}H, ^{13}C NMR (Tables 1 and 2) of **2** with those of **1** suggested that **2** differed from **1** only in the absence of one acetyl group, and this was supported by its molecular formula, which showed 42 mass unit less than that of **1**. In the ^{1}H NMR spectrum of **2**, H-12 (δ 3.86, brs) was upfield shifted ca. $\Delta\delta$ 1.27 as compared with that of **1**, indicating the presence of an OH at C-12 (δ 65.0) in **2** instead of the OAc as in **1**. As the result, C-11 (δ 76.4) and C-13 (δ 34.6) of **2** were downfield shifted ca. $\Delta\delta$ 1.6 and $\Delta\delta$ 3.6, respectively, as compared with those of **1** due to the different γ-gauche effects between the 12-OH of **2** and 12-AcO of **1**. The structure of **2** was further confirmed by a combined analysis of HSQC, HMBC, and ROESY spectra (for details see Supporting Information).

Tabularisin G (**3**) showed the same molecular formula ($C_{39}H_{46}O_{18}$) as **1**. IR absorptions exhibited the presence of OH (3435 cm^{-1}) and carbonyl groups (1736 and 1768 cm^{-1}). The ^{1}H (Table 1) and ^{13}C NMR data of **3** (Table 2) implied that they had the same limonoid core as compound **1**, and the differences were in the positions of the OH, ester, and orthoacetate groups. Acetoxyl groups were assignable to C-3 (δ 85.4), C-12 (δ 68.3), and C-15 (δ 70.6) on the basis of their corresponding HMBC correlations (Figure 2). The HMBC correlations of 1-OH/C-1 and 11-OH/C-11 allowed the assignments of hydroxyls to C-1 (δ 84.6) and C-11 (δ 67.4), respectively. One methyl (4′-Me) of the isobutyryl showed ROESY correlations with H-29b (δ 1.67, d, $J = 11.4$ Hz) and H-3 (δ 5.30, s), favoring linkage of an isobutyryloxyl to C-2 (δ 83.1). Thus, the orthoacetate was attached to C-8 (δ 87.3), C-9 (δ 84.6), and C-30 (δ 76.8). The relative configuration of **3** was established by the ROESY spectrum (Figure 2). The ROSEY correlations of H-5/H-12, H-5/H-15, H-5/H-17, H-12/H-17, and H-15/H-17 indicated that they are co-facial and arbitrarily assigned as β-oriented. ROSEY correlations of H-15/H-30, 3-OAc/H-15, and 3-OAc/H-17 revealed that 3-OAc and H-30 also were β-oriented. ROESY correlations of H-18b/H-22, H-18a/11-OH, H-11/Me-19, H-29a/Me-19, and H-29b/H-3 indicated that the furyl ring, cyclopropyl group, Me-19, and CH$_2$-29 were α-oriented, and H-11 was β-oriented. The ROESY correlations of Me-32/1-OH, H-3/Me-4′, H-29b/Me-4′, and Me-19/1-OH revealed that the 8,9,30-orthoacetate, 2-isobutyryloxyl, and 1-OH were α-oriented.

Tabularisin H (**4**) was determined to have the molecular formula $C_{43}H_{52}O_{20}$ (HREIMS). The ^{1}H (Table 1) and ^{13}C NMR (Table 2) data of **4** showed high similarity to those of tabularisin C (**9**);[2d] the only structural difference was that one acetyl of **9** was replaced by an isobutyryl in **4**. The isobutyryloxy group was then placed at C-15 (δ 69.9) by the HMBC correlation between its carbonyl and H-15 at δ 6.88 (d, $J = 2.4$ Hz). The complete structural assignment of **4** was further confirmed by 2D NMR spectra (Supporting Information).

Tabularisin I (**5**), a white, amorphous powder, possessed a molecular formula of $C_{41}H_{50}O_{19}$ as determined by HREIMS. Analysis of the ^{1}H and ^{13}C NMR data of **5** showed that it was likely a deacetyl derivative of **4**, and this was supported by 42 mass units less in the molecular formula of **5** than that of **4**. On further comparison with compound **4**, H-12 of **5** at δ 4.10 (1H, brs) was upfield shifted ca. $\Delta\delta$ 1.25, indicating that **5** was the 12-deacetyl derivative of **4**. The structure of **5** was finally confirmed by 2D NMR experiments, including HSQC, HMBC, and ROESY spectra (Supporting Information).

(24*R*)-28,29-Dinor-cycloartane-3β,24,25-triol (**6**), a white, amorphous powder, was determined to have a molecular formula of $C_{28}H_{48}O_3$ (calcd 432.3603) by HREIMS; [M]$^+$, *m*/*z* 432.3597. The strong IR absorption at 3423 cm^{-1} revealed the presences of OH groups. The ^{1}H NMR data (Table 3) exhibited proton resonances at δ 0.88 (3H, s), 0.89 (3H, brd, $J = 3.4$ Hz), 0.96 (3H, s), 1.16 (3H, s), and 1.21 (3H, s) due to five methyl groups, an AB system centered at δ 0.07 (1H, d, $J = 3.7$ Hz) and 0.42 (1H, d, $J = 3.7$ Hz) due to the methylene of a cyclopropyl group, and two proton signals at δ 3.28

Table 2. ^{13}C NMR Data of **1–5**[a]

carbon	1	2	3	4	5
1	82.8	82.9	84.6	84.4	84.2
2	76.5	76.4	83.1	83.0	82.7
3	85.7	85.7	85.4	85.3	85.0
4	44.8	44.8	44.6	44.3	44.1
5	38.0	37.7	38.6	43.6	42.8
6	32.9	33.6	33.4	71.0	71.0
7	173.7	173.5	174.0	171.7	170.9
8	78.2	77.9	87.3	87.2	86.6
9	90.4	90.6	84.6	84.9	84.9
10	43.9	44.0	48.4	49.2	49.0
11	74.8	76.4	67.4	67.2	69.0
12	66.4	65.0	68.3	68.2	65.7
13	31.0	34.6	30.0	29.8	33.3
14	30.7	31.7	24.9	25.0	25.8
15	69.7	69.8	70.6	69.9	69.8
16	166.9	167.0	165.7	165.6	165.6
17	71.4	71.3	71.9	71.9	72.4
18	18.6	18.2	17.0	17.0	16.1
19	14.5	14.6	16.5	17.4	17.1
20	122.0	122.0	122.5	122.4	122.4
21	142.0	142.6	141.9	141.8	141.9
22	109.6	108.7	109.9	109.8	109.3
23	143.3	144.8	143.2	143.2	143.5
28	14.1	14.2	14.3	15.3	15.2
29	38.8	38.7	39.7	40.7	40.5
30	70.1	70.0	76.8	76.4	76.1
31	119.7	119.5	116.4	116.2	115.9
32	16.2	16.2	15.7	15.7	15.5
1′	173.4	173.5	175.6	175.4	175.1
1″				174.8	174.6
2′	33.9	33.9	34.5	34.2	34.1
2″				33.7	33.6
3′	19.4	19.4	18.7	18.8	18.6
3″				18.6	18.4
4′	18.7	18.7	18.7	18.7	18.5
4″				18.4	18.2
3-OAc	169.1	169.1	168.6	168.4	168.3
	20.9	21.0	21.0	20.9	20.7
6-OAc				169.1	169.1
				20.9	20.8
12-OAc	170.6		170.9	170.9	
	19.7		19.7	19.5	
15-OAc	172.1	172.2	169.0		
	21.4	21.4	20.5		
–OCH$_3$	52.4	52.4	52.3	53.5	53.4

[a] Data were measured in CDCl$_3$ at 100 MHz; chemical shifts (δ) are expressed in ppm.

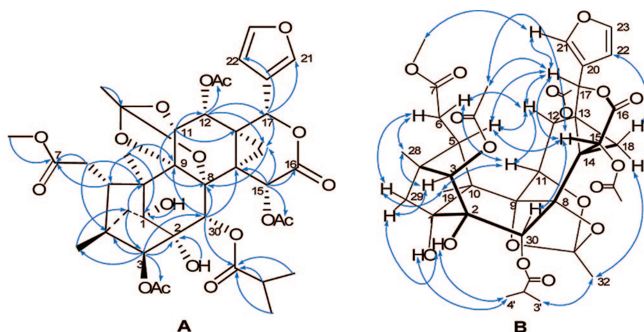

Figure 1. Key HMBC (A: H→C) and ROESY (B: ↔) correlations of **1**.

(1H, m) and 3.68 (1H, brd, J = 9.6 Hz) due to two oxygenated methines. Comparison of the ^{1}H and ^{13}C NMR (Table 3) of **6** with those of 29-nor-cycloartane-3β,24ξ,25-triol,[6] which was formerly classified as the steroid 4α,14-dimethyl-9,19-cyclocholestane-3β,24ξ,25-triol,[7] indicated that **6** was likely to be 28,29-dinor-cycloartane-3β,24,25-triol. The NMR data of the two compounds showed high similarity except for the absence of the C-28 methyl in **6**. In its ^{13}C NMR spectrum, C-3 (δ 71.2) and C-5 (δ 37.1) were upfield shifted

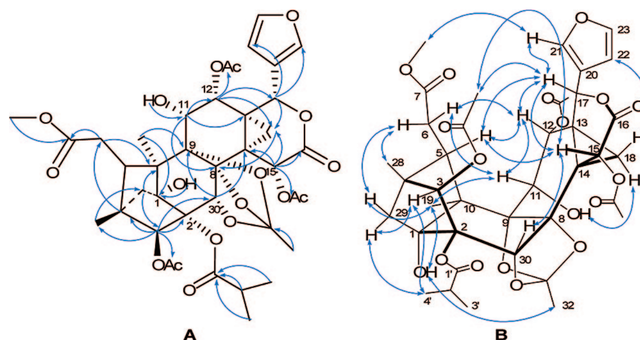

Figure 2. Key HMBC (A: H→C) and ROESY (B: ↔) correlations of **3**.

Table 3. ^{1}H and ^{13}C NMR Data of **6**[a]

position	δ_H (mult., J in Hz)	δ_C	position	δ_H (mult., J in Hz)	δ_C
1α	1.51 m	30.5	14		49.0
1β	1.28 m		15	1.29 m, 2H	35.0
2α	1.97 m	35.2	16α	1.88 m	28.0
2β	1.30 m		16β	1.35 m	
3	3.68 m	71.2	17	1.59 m	52.2
4α	1.81 m	42.4	18	0.96 s, 3H	17.4
4β	1.11 m		19a	0.42 d (3.7)	25.8
5	1.53 m	37.1	19b	0.07 d (3.7)	
6α	1.36 m	24.6	20	1.41 m	36.4
6β	0.77 m		21	0.89 br d, 3H (3.4)	18.5
7α	1.11 m	27.7	22a	1.76 m	33.5
7β	1.27 m		22b	1.01 m	
8	1.71 m	46.1	23a	1.58 m	28.7
9		23.2	23b	1.13 m	
10		29.8	24	3.28 br d (9.6)	79.6
11α	1.88 m	27.0	25		73.2
11β	1.28 m		26	1.21 s, 3H	23.1
12	1.60 m, 2H	32.8	27	1.16 s, 3H	26.5
13		45.3	30	0.88 s, 3H	18.9

[a] Data were measured in CDCl$_3$ at 400 MHz (^{1}H) and 100 MHz (^{13}C); chemical shifts (δ) are expressed in ppm; the spin coupling (J) is given in parentheses (Hz).

ca. $\Delta\delta$ 5.3 and 6.3 from C-3 (δ 76.5) and C-5 (δ 43.4) in 29-nor-cycloartane-3β,24ξ,25-triol, respectively, supporting this deduction. Further, the chemical shifts of C-17 (δ 52.2) and the carbons of the side chain in **6** were essentially the same as those (C-17, ca. δ 52.3; C-20, ca. δ 36.4; C-21, ca. δ 18.4; C-22, ca. δ 33.5; C-23, ca. δ 28.7; C-24, ca. δ 79.6; C-25, ca. δ 73.2; C-26, ca. δ 23.2; C-27, ca. δ 26.6) of (24*R*)-cycloartane-3β,24,25-triol and slightly different from those (C-17, ca. δ 52.4; C-20, ca. δ 35.9; C-21, ca. δ 18.2; C-22, ca. δ 33.1; C-23, ca. δ 28.4; C-24, ca. δ 78.8; C-25, ca. δ 73.7; C-26, ca. δ 23.3; C-27, ca. δ 26.6) of (24*S*)-cycloartane-3β,24,25-triol,[8] indicating that compound **6** was (24*R*)-28,29-dinor-cycloartane-3β,24,25-triol. The structure of **6** was finally confirmed by analyses of 2D NMR, including HSQC, HMBC, and ROESY spectra (Figure 3 and Supporting Information).

Three known compounds were identified to be tabularisins A–C (**7–9**)[2d] on the basis of their ^{1}H and ^{13}C NMR and EIMS data and confirmed by co-TLC with authentic samples.

Experimental Section

General Experimental Procedures. IR spectra were recorded on a Perkin-Elmer 577 spectrometer with a KBr disc. UV spectra were measured on a Shimadzu UV-2550 UV–visible spectrophotometer. Optical rotations were made on a Perkin-Elmer 341 polarimeter at room temperature. NMR spectra were measured on a Bruker AM-400 spectrometer with TMS as internal standard. EIMS (70 eV) and ESIMS were carried out on a Finnigan MAT 95 mass spectrometer and an Esquire 3000plus LC-MS instrument, respectively. All solvents used were of analytical grade (Shanghai Chemical Plant, Shanghai, People's Republic of China). Silica gel (300–400 mesh), C18 reversed-phase silica gel (150–200 mesh, Merck), and MCI gel

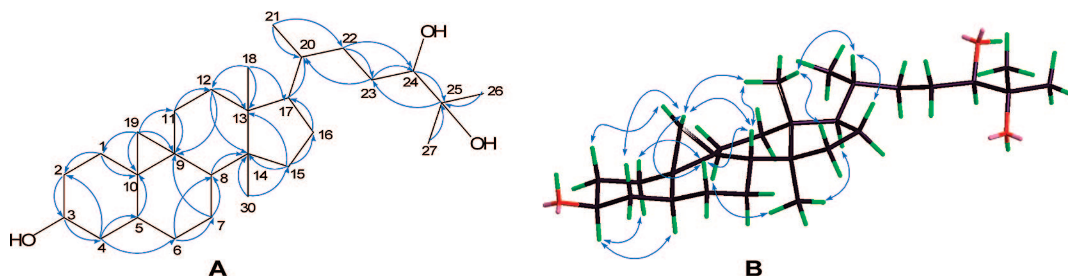

Figure 3. Key HMBC (A: H→C) and ROESY (B: ↔) correlations of **6**.

(CHP20P, 75–150 μM, Mitsubishi Chemical Industries Ltd.) were used for column chromatography, and precoated silica gel GF254 plates (Qingdao Marine Chemical Plant, Qingdao, People's Republic of China) were used for TLC.

Plant Material. The twigs and leaves of *C. tabularis* var. *velutina* were collected from Xishuangbanna, Yunnan Province, China, and were authenticated by Professor You-Kai Xu of Xishuangbanna Tropical Botanical Garden, Chinese Academy of Sciences. A voucher specimen (CTvv-2005-1Y) has been deposited in the Shanghai Institute of Materia Medica.

Extraction and Isolation. The air-dried powder of twigs and leaves (5 kg) of *C. tabularis* var. *velutina* was extracted with 95% ethanol extensively at room temperature to give a dark green residue (238 g), which was then partitioned between EtOAc and water to give the EtOAc-soluble fraction E (110 g). The E fraction was first subjected to a column of MCI gel eluted with MeOH–H_2O (3:7 to 9:1) to give four fractions (F1–F4). F2 (13.5 g) was chromatographed on a silica gel column eluted with petroleum ether–EtOAc (gradient from 4:1 to 1:2) to afford five subfractions (F3a–F3e). F3d was subjected to a column of reversed-phase C_{18} silica gel eluted with MeOH–H_2O (from 6:4 to 8:2) to give three major fractions, F3d1–F3d3. F3d1 was then purified by silica gel CC eluted with $CHCl_3$–MeOH (200:1) to yield compound **6** (30 mg). F3d2 was also purified by silica gel CC eluted with $CHCl_3$–MeOH (300:1) to yield **7** (58 mg). F3d3 was extensively purified by silica gel CC eluted with $CHCl_3$–MeOH (400:1) and then a column of reversed-phase C_{18} silica gel eluted with MeOH–H_2O (7:3) to give **8** (13 mg). F3e was subjected to a column of reversed-phase C_{18} silica gel eluted with MeOH–H_2O (from 5:5 to 7:3) to give five major fractions, F3e1–F3e5. F3e2 was separated on a silica gel column eluted with $CHCl_3$–MeOH (200:1) to yield compounds **2** (8 mg) and **5** (14 mg). F3e4 was separated into three major subfractions by silica gel CC eluted with $CHCl_3$–MeOH (150:1), and each of them was then purified on a column of reversed-phase C_{18} silica gel eluted with MeOH–H_2O (6:4) to yield compounds **3** (12 mg), **4** (5 mg), and **9** (11 mg), respectively. F3e5 was purified by silica gel CC eluted with $CHCl_3$–MeOH (200:1) to afford compound **1** (20 mg).

Tabularisin E (1): white, amorphous powder; $[\alpha]^{20}_D$ +23 (*c* 0.124, $CHCl_3$); UV (MeOH) λ_{max} (log ε) 206 (3.88) nm; IR (KBr) λ_{max} 3547, 3435, 2972, 1759, 1730, 1421, 1369, 1211, 1022, 889 cm^{-1}; for ^{1}H NMR data, see Table 1; for ^{13}C NMR data, see Table 2; ESIMS *m/z* 825 [M + Na]$^+$; EIMS *m/z* 802 [M]$^+$ (6.5), 771 (7), 742 (100), 682 (22), 612 (90), 552 (63), 492 (78), 464 (60), 182 (58), 95 (69), 71 (97); HREIMS *m/z* 802.2700 (calcd for $C_{39}H_{46}O_{18}$ 802.2684).

Tabularisin F (2): white, amorphous powder; $[\alpha]^{20}_D$ +12 (*c* 0.75, $CHCl_3$); UV (MeOH) λ_{max} (log ε) 206 (3.97) nm; IR (KBr) λ_{max} 3538, 3472, 2974, 1755, 1728, 1369, 1213, 1134, 1022, 874 cm^{-1}; for ^{1}H NMR data, see Table 1; for ^{13}C NMR data, see Table 2; ESIMS *m/z* 783 [M + Na]$^+$; EIMS *m/z* 760 [M]$^+$ (26), 729 (8), 700 (45), 605 (26), 570 (21), 510 (27), 464 (25), 182 (57), 81 (31), 71 (100); HREIMS *m/z* 760.2579 (calcd for $C_{37}H_{44}O_{17}$ 760.2578).

Tabularisin G (3): white, amorphous powder; $[\alpha]^{20}_D$ +17 (*c* 0.700, $CHCl_3$); UV (MeOH) λ_{max} (log ε) 206 (3.74) nm; IR (KBr) λ_{max} 3435, 2974, 1768, 1736, 1375, 1207, 1034, 892 cm^{-1}; for ^{1}H NMR data, see Table 1; for ^{13}C NMR data, see Table 2; ESIMS *m/z* 825 [M + Na]$^+$; EIMS *m/z* 802 [M]$^+$ (1), 742 (1), 714 (7), 627 (46), 566 (33), 534 (36), 506 (41), 464 (41), 182 (80), 95 (83), 71 (100); HREIMS *m/z* 802.2685 (calcd for $C_{39}H_{46}O_{18}$ 802.2684).

Tabularisin H (4): white, amorphous powder; $[\alpha]^{20}_D$ +25 (*c* 0.910, $CHCl_3$); UV (MeOH) λ_{max} (log ε) 206 (3.82) nm; IR (KBr) ν_{max} 3440, 2976, 1767, 1736, 1374, 1205, 1038, 875 cm^{-1}; for ^{1}H NMR data, see Table 1; for ^{13}C NMR data, see Table 2; ESIMS *m/z* 911 [M + Na]$^+$; EIMS *m/z* 888 [M]$^+$ (2), 828 (1), 801 (6), 713 (40), 610 (16), 592 (25), 550 (22), 180 (16), 149 (13), 95 (35), 71 (100); HREIMS *m/z* 888.3047 (calcd for $C_{43}H_{52}O_{20}$ 888.3052).

Tabularisin I (5): white, amorphous powder; $[\alpha]^{20}_D \approx 0$ (*c* 0.730, $CHCl_3$); UV (MeOH) λ_{max} (log ε) 206 (3.99) nm; IR (KBr) ν_{max} 3496, 2976, 1763, 1736, 1375, 1207, 1038, 876 cm^{-1}; for ^{1}H NMR data, see Table 1; for ^{13}C NMR data, see Table 2; EIMS *m/z* 846 [M]$^+$ (6), 800 (2), 671 (9), 592 (11), 564 (12), 550 (20), 180 (12), 149 (27), 95 (55), 71 (100); HREIMS *m/z* 846.2941 (calcd for $C_{41}H_{50}O_{19}$ 846.2946).

(24*R*)-28,29-Dinor-cycloartane-3β,24,25-triol (6): white, amorphous powder; $[\alpha]^{20}_D$ +31 (*c* 0.165, $CHCl_3$); IR (KBr) ν_{max} 3423, 2927, 2868, 1635, 1736, 1466, 1377, 1161, 1105, 1074, 1030 cm^{-1}; for ^{1}H and ^{13}C NMR data, see Table 3; ESIMS *m/z* 455 [M + Na]$^+$; EIMS *m/z* 432 [M]$^+$ (22), 414 (100), 399 (74), 381 (66), 320 (49), 287 (86), 269 (73), 175 (82), 161 (57), 147 (68), 145 (45), 135 (62), 127 (42), 121 (80), 107 (75), 95 (95), 81 (62), 59 (81); HREIMS *m/z* 432.3597 (calcd for $C_{28}H_{48}O_3$ 432.3603).

Acknowledgment. Financial support from the Key Project of National Natural Science Foundation (Grant No. 30630072) and from the Shanghai Municipal Scientific Foundation (Grant No. 06DZ22028) of the People's Republic of China is gratefully acknowledged. We thank Prof. Y.-K. Xu of Xishuangbanna Tropical Botanical Garden, Chinese Academy of Sciences, for the collection and identification of the plant material.

Supporting Information Available: Key HMBC and ROESY correlations of **2**, **4**, and **5** (figures); IR, MS, 1D and 2D NMR spectra of **1**–**6**. This material is available free of charge via the Internet at http://pubs.acs.org.

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NP070345W