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大理产升麻中一个新环阿尔廷三萜皂甙*

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摘要: 从升麻 (*Cimicifuga foetida* L.) 根茎中分离得到一个新的四环三萜皂甙和5个已知化合物, 它们分别是12β-乙酰基升麻醇-3-O-β-D-木糖甙 (**1**), 升麻醇 (**2**), 25-O-乙酰基升麻醇 (**3**), 升麻醇3-O-β-D-升麻醇木糖甙 (**4**), 阿科特素 (**5**), 小升麻甙B (**6**)。其化学结构经光谱解析和化学方法鉴定。

关键词: 升麻; 12β-乙酰基升麻醇-3-O-β-D-木糖甙; 环阿尔廷三萜皂甙
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A New Cycloartane Triterpenoid from the Rhizome of *Cimicifuga foetida* Collected in Dali

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Abstract: A new triterpene glycoside, 12β-acetoxycimigenol-3-O-β-D-xylopyranoside (**1**) together with five known compounds, cimigenol (**2**), 25-O-acetylcimigenol (**3**), cimigenol-3-O-β-D-xylpyranoside (**4**), actein (**5**), cimiaceroside B (**6**), were isolated from the rhizome extract of *Cimicifuga foetida* collected in Dali. All structures of the compounds were established by spectral interpretation and chemical methods.

Key words: *Cimicifuga foetida*; 12β-acetoxycimigenol-3-O-β-D-xylopyranoside; Cycloartane triterpene glycoside

Cimicifuga foetida L., a famous traditional Chinese medicine plant, is distributed in many Provinces of China, which has been employed as traditional medicines for the alleviation of fever, pain, inflammation, and antipyretic and analgesic remedy (Jiangsu New Medical College, 1977). Cycloartane triterpene glycosides are considered to be the main bioactive components and are used as mark compounds to standardize its extracts (Yu *et al*, 2000). Up to now, more than 100 triterpenes

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and triterpene glycosides have been reported from the genus *Cimicifuga*, and more than 20 triterpene glycosides have been isolated from *C. foetida* (Lin *et al*, 2002; Zhu *et al*, 2001). In the course of our screening of biologically active constituents, one new cycloartane triterpenoid, together with five known compounds, were isolated from *C. foetida* collected in Dali county. In the paper, we would like to present the isolation of the compound and its chemical structure identification.

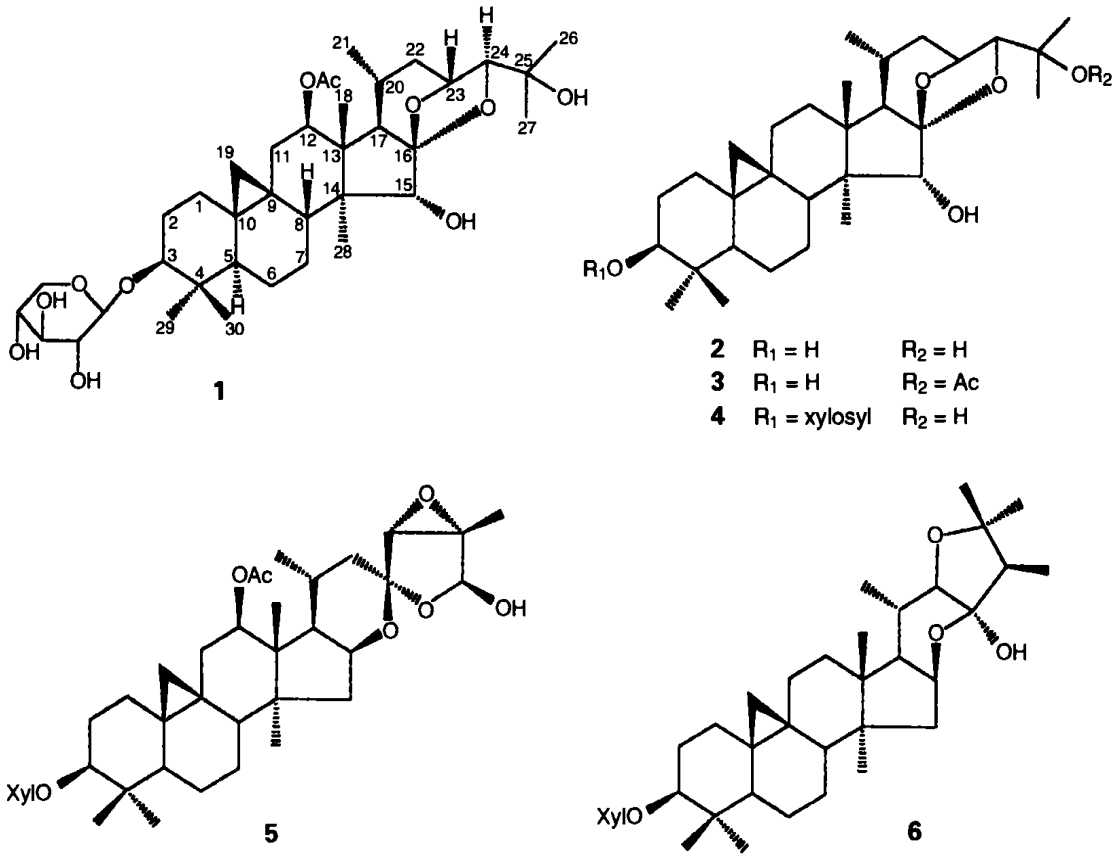

Fig. 1 Chemical structures of compounds **1 – 6**

Result and Discussion

Compound **1** was obtained as a white powder, with a molecular formula of $C_{37}H_{57}O_{11}$ indicated by the quasi-molecular ion peak at *m/z* 677.4057 $[M-1]^-$ in its HRFABMS (calcd. for $C_{37}H_{57}O_{11}$ *m/z* 677.4087), which was consistent with its NMR data. Its IR spectrum showed absorption at 3441 cm^{-1} for hydroxyl and 1732 cm^{-1} for carbonyl groups. The ^1H NMR spectrum showed two characteristic cyclopropane methylene signals at δ 0.28 and 0.57 (each 1H, d, $J = 3.5$ Hz) in the upfield region; six methyl singlet peaks due to tert-methyl groups at δ 0.99, 1.20, 1.28, 1.31, 1.46 and 1.48 (each 3H, s), an obvious methyl signal for an acetyl group at δ 2.11 (3H, s), one methyl doublet ascribable to a secondary methyl group at δ 0.85 (3H, d, $J = 6.9$ Hz), a typical doublet attributable to an anomeric proton of a sugar at δ 4.83 (1H, d, $J = 7.5$ Hz). 37 carbons signals were observed in the ^{13}C NMR and DEPT spectra (Table 1), which was indicative of a triterpenoid, including a five-carbons at δc 107.6 (d, C-1′), 75.6 (d, C-2′), 78.7 (d, C-3′), 71.5 (d, C-4′),

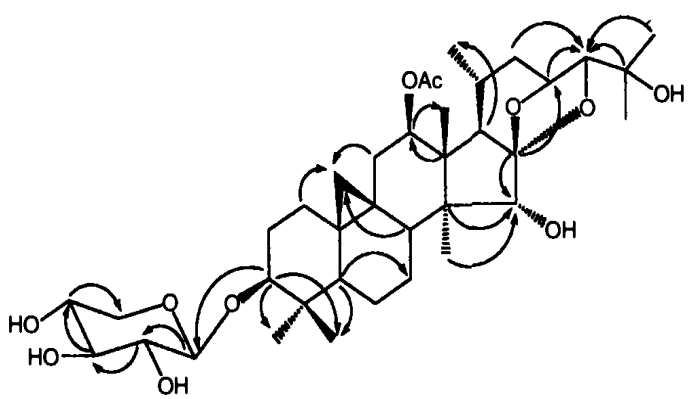

Fig. 2 Selected HMBC correlations of **1**

67.1 (t, C-5′), an acetyl group δc 170.6 (s, $\underline{C}OCH_3$) and δc 21.7 (q, $CO\underline{C}H_3$). A characteristic ketalic quaternary carbon signal at δ 112.0 (s, C-16), together with two oxygen-bearing methine signals at δc 88.3 (d, C-15) and 90.0 (d, C-24) and the cyclopropane methylene at δ_c 30.9 (t, C-19), suggested that **1** was a highly oxygenated 9, 19-cycloartane cimigenol-type monoglycoside with an acetoxy substituent. Through the combination of ^{1}H-^{1}H COSY, HMQC, and HMBC experiments, the ^{1}H and ^{13}C signals for compound **1** were fully assigned (Table 1). Comparing the ^{13}C NMR data of **1** with those of a known compound, 12β-acetoxycimigenol-3-O-β-L-arabinopyranoside (Yu *et al*, 2000), we disclosed that both compounds have the same aglycone. The configurations of C-23 and C-24 were also assigned as R and S form, respectively, by comparing the coupling constants of the C-23 and C-24 proton signals of **1** with those of known 9, 19-cycloartane triterpene glycosides (Kusano *et al*, 1994; Ye *et al*, 1999). And the sugar moiety was identified as β-D-xylose by acid hydrolysis followed by TLC analysis with an authentic sample and by the HMBC spectrum. In the HMBC spectrum, correlation observed between the δ_H 4.83 (d, 1H, J = 7.5 Hz) and δc 88.3 (d, C-3), suggested that the sugar moiety was located at the C-3 position. Based on all analyses above data, chemical structure of compound **1** was elucidated as 12β-acetoxycimigenol-3-O-β-D-xylopyranoside.

Experimental

General Experimental Procedures Melting points were determined on XRC-1 apparatus and uncorrected. Optical rotations were obtained on JASCO-20C digital polarimeter. Infrared spectra were recorded on a Bio-Rad FTS-135 spectrometer. FAB-MS were determined on a VG Auto Spec-3000 mass spectrometer. 1D and 2D NMR spectra were measured on Bruker AV-400 MHz or DRX-500 MHz instruments, and chemical shifts (δ) were reported with the solvent (Pyridine-d_5) as the reference. Silica gel (200 – 300 mesh and 10 – 40 μm) was used for column chromatography. Thin-layer chromatography was performed on precoated TLC plates (GF_{254}. Qingdao, People's Republic of China). The spots were visualized by spraying with 20% H_2SO_4 and followed by heating. Reversed-phase column chromatography was carried out on Merck lobar lichroprep RP-18 columns (EM Science, Germany).

Plant Material The roots/rhizomes of *Cimicifuga foetida* were collected in Dali county of Yunnan province, China, in September 2000, and identified by Prof. Wang Z Y. A voucher specimen (KUN 0273201) was deposited in the Herbarium of Kunming Institute of Botany, the Chinese Academy of Sciences.

Extraction and Isolation The dried, milled roots/rhizomes (2.8 kg) of *Cimicifuga foetida* were exhaustively extracted with 90% MeOH (3 × 10 L) under reflux. The MeOH extract was evaporated to yield a black syrup-like residue (459 g) under reduced pressure. A sample (439 g) of the residue was suspended in water-MeOH (9:1, 1300 mL) and fractionated by successive partitions with EtOAc (3 × 1800 mL) and n-BuOH (3 × 1800 mL) to give EtOAc-soluble (214

g) and n-BuOH-soluble fractions (82 g). A portion (200 g) of the EtOAc-soluble fraction was subjected to silica gel column chromatography (7.5 × 120 cm, 200 – 300 mesh, 1200 g), eluting with $CHCl_3$-MeOH (10:0, 9:1, 8:2, 7:3, 6:4, 0:10; each 10 L) gradient system to yield six fractions: A (oil), B (14 g), C (9 g), D (88 g), E (43 g), F (12 g).

Fraction A did not show any significant spots, and was not further isolated. Fration B (14 g) was dissolved in acetone and then filtered, and its filter residue was washed with acetone to give compound **3** (56 mg), the remained part of this fraction was not isolated further because no major or interesting components were detected by TLC. Fraction C (9 g) was rechromatographied on silica gel (4 × 70 cm, 200 – 300 mesh, 100 g) eluting with solvent mixtures of $CHCl_3$-MeOH (100:1, 80:1, 65:1, 50:1, each 2 L) to give four subfractions (C_1-C_4). Compound **2** (45 mg) was crystallized from subfraction C_1 (0.4 g). Fraction D (88 g) was subjection to silica gel CC (7.5 × 120 cm, 200 – 300 mesh, 800 g), developing with $CHCl_3$-MeOH (40:1, 25:1, 10:1, each 5 L) to give three sub-fractions (D_1-D_3). Subfraction D_1 (2.9 g) was rechromatographied over a silica gel column (4 × 125 cm, 200 – 300 mesh, 80 g), developing with $CHCl_3$-MeOH (40:1, 2.5 L) to afford compound **5** (427 mg). The remaining (0.3 g) of fraction D_1 was chromatographied over RP_{18} silica gel (3 × 40 cm, 65 mg) eluting with MeOH-H_2O (7:3, 2 L) to afford compound **1** (35 mg). Subfraction D_2 (27 g) was rechromatographied on silica gel (3 × 40 cm), eluting with $CHCl_3$-MeOH (25:1, 1.5 L) to provide compound **4** (534 mg) and **6** (73 mg).

Table 1 ^1H NMR and ^{13}C NMR data of compound **1** in C_5D_5N (δ in ppm)

Position	^1H	^{13}C	Position	^1H	^{13}C
1	1.08 (m), 1.58 (m)	32.4t	20	1.68 (m)	24.1d
2	1.86 (m), 2.28 (m)	30.1t	21	0.85 (d, J=6.9 Hz)	
3	3.46 (dd, J=4.1, 11.6 Hz)	88.3d	22	1.06 (m), 2.30 (m)	38.6t
4		41.3s	23	4.20 (dd, J=4.9, 9.1 Hz)	71.3d
5	1.32 (m)	47.2d	24	3.76 (s)	90.0d
6	0.70 (m), 1.54 (m)	20.8t	25		71.0s
7	1.11 (m), 2.02 (m)	26.0t	26	1.46 (s, 3H)	25.5q
8	1.79 (m)	47.3d	27	1.48 (s, 3H)	25.7q
9		20.0s	28	1.20 (s, 3H)	11.9q
10		26.9s	29	1.28 (s, 3H)	27.1q
11	1.09 (m), 2.92 (dd, J=9.4, 16.0 Hz)	37.5t	30	0.99 (s, 3H)	15.4q
12	5.25 (dd, J=7.6, 16.0 Hz)	77.3d	12-$\underline{C}OCH_3$		170.6s
13		46.3s	12-CO$\underline{C}H_3$	2.11 (s, 3H)	21.7q
14		48.5s	Xyl-1′	4.83 (d, J=7.5 Hz)	107.6d
15	4.37 (s, 1H)	79.2d	2′	4.01 (t, J=8.0 Hz)	75.6d
16		112.0s	3′	4.14 (t, J=8.5 Hz), 4.01 (t, J=8.0 Hz)	78.7d
17	1.73 (dd, J=4.3, 16.6 Hz)	59.3d	4′	4.73 (d, J=8.9 Hz)	71.5d
18	1.31 (s, 3H)	12.7q	5′	3.70 (t, J=10.0 Hz),	67.1t
19	0.28 (d, J=3.5 Hz), 0.57 (d, J=3.5 Hz)	30.9t		4.33 (dd, J=4.9, 11.2 Hz)	

[a] ^1H and ^{13}C-NMR spectra were obtained at 500 Hz and 400 Hz, and recorded in C_5D_5N at room temperature, respectively.

[b] Coupling constants are presented in Hz. Unless otherwise indicated, all proton signals integrated to ^1H.

Compound **1**, white powder; mp 185 – 187℃, $[\alpha]_D^{24}$ – 41° [*c* 0.55 MeOH:$CHCl_3$ (1:1)], negative FAB-MS *m/z* 677.4057 [M-1]$^-$ (calcd. for $C_{37}H_{57}O_{11}$ *m/z* 677.4087); ^1H and ^{13}C NMR data, Table 1.

Compound **2**, $C_{30}H_{48}O_5$, white powder, mp 197 – 198℃, positive FAB-MS *m/z*: 489 [M + 1]$^+$, ^1H NMR (400 MHz, C_5D_5N) δ: 3.53 (1H, dd, *J* = 4.5, 11.2 Hz, H – 3), 1.31 (1H, dd, *J* = 3.4, 12.9 Hz, H – 5), 0.70 (1H, dd, *J* = 9.6, 10.2 Hz, H – 6α), 1.66 (1H, m, H – 6β), 4.40 (1H, s, H – 15), 1.19 (3H, s, Me – 18), 0.34 (1H, d, *J* = 4.1 Hz, H – 19), 0.60 (1H, d, *J* = 4.0 Hz, H – 19), 0.86 (3H, d, *J* = 6.5 Hz, Me – 21), 4.74 (1H, d, *J* = 9.0 Hz, H – 23), 3.76 (1H, s, H – 24), 1.46 (3H, s, Me – 26),

1.48 (3H, s, Me-27), 1.21 (3H, s, Me-28), 1.19 (3H, s, Me-29), 1.09 (3H, s, Me-30). ^{13}C NMR (100 MHz, C_5D_5N) δ: 32.7 (t, C-1), 31.4 (t, C-2), 78.0 (d, C-3), 41.1 (s, C-4), 47.5 (d, C-5), 21.4 (t, C-6), 26.6 (t, C-7), 48.7 (d, C-8), 20.0 (s, C-9), 27.0 (s, C-10), 26.5 (t, C-11), 34.1 (t, C-12), 41.9 (s, C-13), 47.4 (s, C-14), 80.0 (d, C-15), 112.0 (s, C-16), 59.6 (d, C-17), 19.6 (q, C-18), 31.1 (t, C-19), 24.1 (d, C-20), 19.6 (q, C-21), 38.2 (t, C-22), 71.8 (d, C-23), 90.2 (d, C-24), 71.0 (s, C-25), 27.2 (q, C-26), 26.2 (q, C-27), 11.9 (q, C-28), 25.4 (q, C-29), 14.9 (q, C-30).

Compound **3**, $C_{32}H_{50}O_6$, white powder, mp 213-214℃, positive FAB-MS *m/z*: 531 $[M+1]^+$, ^{1}H NMR (400 Hz, C_5D_5N) δ: 3.53 (1H, dd, *J*=4.5, 11.4 Hz, H-3), 1.94 (1H, s, H-7), 4.27 (1H, s, H-15), 1.46 (1H, d, *J*=11.1 Hz, H-17), 1.20 (3H, s, Me-18), 0.32 (1H, d, *J*=3.8 Hz, H-19), 0.59 (1H, d, *J*=3.6 Hz, H-19), 0.85 (3H, d, *J*=6.5 Hz, Me-21), 4.58 (1H, d, *J*=9.0 Hz, H-23), 4.11 (1H, s, H-24), 1.67 (3H, s, Me-26), 1.65 (3H, s, Me-27), 1.19 (3H, s, Me-28), 1.08 (3H, s, Me-29), 1.15 (3H, s, Me-30). ^{13}C NMR (100 MHz, C_5D_5N) δ: 32.7 (t, C-1), 31.1 (t, C-2), 78.0 (d, C-3), 41.1 (s, C-4), 47.3 (d, C-5), 21.4 (t, C-6), 26.5 (t, C-7), 48.8 (d, C-8), 19.7 (s, C-9), 26.8 (s, C-10), 26.6 (t, C-11), 34.1 (t, C-12), 41.7 (s, C-13), 47.5 (s, C-14), 80.2 (d, C-15), 112.5 (s, C-16), 59.4 (d, C-17), 19.5 (q, C-18), 31.3 (t, C-19), 24.0 (d, C-20), 19.5 (q, C-21), 38.0 (t, C-22), 71.7 (d, C-23), 86.8 (d, C-24), 83.2 (s, C-25), 24.1 (q, C-26), 23.4 (q, C-27), 11.9 (q, C-28), 26.2 (q, C-29), 14.9 (q, C-30), 25-$\underline{C}OCH_3$ (s, 170.2), 25-CO$\underline{C}H_3$ (q, 21.5).

Compound **4**, $C_{35}H_{56}O_9$, colorless needles (MeOH), mp 267-268℃, negative FAB-MS *m/z*: 621 $[M-1]^-$, ^{1}H NMR (400 Hz, C_5D_5N) δ: 3.53 (1H, dd, *J*=4.5, 11.2 Hz, H-3), 0.76 (1H, dd, *J*=9.6, 10.2 Hz, H-6), 4.47 (1H, d, *J*=11.1 Hz, H-15), 1.31 (3H, s, Me-18), 0.27 (1H, d, *J*=2.7 Hz, H-19), 0.52 (1H, d, *J*=2.1 Hz, H-19), 2.0 (1H, s, H-20), 0.85 (3H, d, *J*=5.1 Hz, Me-21), 2.40 (1H, dd, *J*=4.5, 13.2 Hz, H-22), 4.74 (1H, d, *J*=7.3 Hz, H-23), 3.76 (1H, s, H-24), 1.45 (3H, s, Me-26), 1.48 (3H, s, Me-27), 1.30 (3H, s, Me-28), 1.40 (3H, s, Me-29), 1.04 (3H, s, Me-30), 4.84 (1H, d, *J*=6.0 Hz, H-1′), 4.02 (1H, t, *J*=7.7 Hz, H-2′), 4.15 (1H, t, *J*=8.2 Hz, H-3′), 4.34 (1H, dd, *J*=4. 9, 12.6 Hz, H-4′), 4.74 (1H, m, H-5′), 4.24 (1H, m, H-5′). ^{13}C NMR (100 MHz, C_5D_5N) δ: 32.5 (t, C-1), 30.2 (t, C-2), 88.6 (d, C-3), 41.9 (s, C-4), 47.7 (d, C-5), 21.1 (t, C-6), 26.5 (t, C-7), 48.7 (d, C-8), 20.1 (s, C-9), 26.7 (s, C-10), 26.4 (t, C-11), 34.1 (t, C-12), 41.9 (s, C-13), 47.3 (s, C-14), 80.3 (d, C-15), 112.0 (s, C-16), 59.6 (d, C-17), 19.6 (q, C-18), 30.9 (t, C-19), 24.1 (d, C-20), 19.6 (q, C-21), 38.2 (t, C-22), 71.3 (d, C-23), 90.2 (d, C-24), 71.0 (s, C-25), 25.5 (q, C-26), 25.8 (q, C-27), 11.9 (q, C-28), 26.4 (q, C-29), 15.5 (q, C-30), 107.6 (d, C-1′), 75.6 (d, C-2′), 78.6 (d, C-3′), 71.9 (d, C-4′), 67.1 (t, C-5′).

Compound **5**, $C_{37}H_{56}O_{11}$, white powder, mp 232-233℃, negative FAB-MS *m/z*: 675 $[M-1]^-$, ^{1}H NMR (400 Hz, C_5D_5N) δ: 3.42 (1H, dd, *J*=11.0, 4.0 Hz, H-3), 1.22 (1H, s, H-5), 2.69 (1H, dd, *J*=16.0, 9.0 Hz, H-11), 5.05 (1H, dd, *J*=9.0, 4.0 Hz, H-12), 4.57 (1H, dd, *J*=14.0, 7.1 Hz, H-16), 1.34 (3H, s, Me-18), 0.59 (1H, d, *J*=4.0 Hz, H-19), 0.25 (1H, d, *J*=4.0 Hz, H-19), 0.97 (3H, d, *J*=6 Hz, Me-21), 3.86 (1H, s, H-24), 1.75 (1H, s, Me-26), 1.66 (1H, s, Me-27), 0.79 (3H, s, Me-28), 1.27 (3H, s, Me-29), 0.96 (3H, s, Me-30), 2.11 (3H, s, 12′-$COCH_3$), 4.78 (1H, d, *J*=7.0 Hz, H-1′), 3.94 (1H, t, *J*=6.7 Hz, H-2′), 4.10 (1H, dd, *J*=2.7,

7.6 Hz, H-3′), 4.18 (1H, m, H-4′), 3.67 (1H, m, H-5′), 4.30 (1H, dd, J=5.0, 10.5 Hz, H-5′). ^{13}C NMR (100 Hz, C_5D_5N) δ: 31.9 (t, C-1), 29.9 (t, C-2), 88.1 (d, C-3), 41.2 (s, C-4), 47.0 (d, C-5), 20.1 (t, C-6), 25.7 (t, C-7), 45.7 (d, C-8), 20.1 (s, C-9), 26.7 (s, C-10), 36.7 (t, C-11), 77.1 (t, C-12), 48.7 (s, C-13), 47.8 (s, C-14), 43.6 (t, C-15), 73.1 (d, C-16), 59.4 (d, C-17), 13.5 (q, C-18), 29.5 (t, C-19), 26.0 (d, C-20), 21.0 (q, C-21), 37.6 (t, C-22), 105.8 (s, C-23), 63.5 (s, C-24), 65.6 (s, C-25), 98.4 (s, C-26), 13.1 (q, C-27), 19.5 (q, C-28), 25.7 (q, C-29), 15.3 (q, C-30), 171.61 (s, 12-$\underline{C}OCH_3$), 21.67 (q, 12-$CO\underline{C}H_3$), 107.6 (d, C-1′), 75.6 (d, C-2′), 78.7 (d, C-3′), 71.3 (d, C-4′), 67.2 (t, C-5′).

Compound **6**, $C_{35}H_{56}O_9$, white powder, mp 278-280℃, negative FAB-MS m/z: 619 [M-1]$^-$, ^{1}H NMR (400 Hz, C_5D_5N) δ: 3.49 (1H, dd, J=11.8, 4.8 Hz, H-3), 1.22 (1H, s, H-5), 4.93 (1H, dd, J=15.6, 7.8 Hz, H-16), 1.19 (3H, s, Me-18), 0.47 (1H, d, J=4.1 Hz, H-19), 0.17 (1H, d, J=4.1 Hz, H-19), 3.86 (1H, d, J=7.5 Hz, H-22), 4.19 (1H, s, H-24), 1.76 (1H, s, Me-26), 1.67 (1H, s, Me-27), 0.83 (3H, s, Me-28), 1.32 (3H, s, Me-29), 1.03 (3H, s, Me-30), 2.11 (3H, s, 12′-$COCH_3$), 4.85 (1H, d, J=6.9 Hz, H-1′), 4.03 (1H, t, J=8.0 Hz, H-2′), 4.13 (1H, t, J=8.3 Hz, H-3′), 4.22 (1H, m, H-4′), 3.72 (1H, t, J=11.0 Hz, H-5′), 4.32 (1H, dd, J=5.0, 11.1 Hz, H-5′). ^{13}C NMR (100 Hz, C_5D_5N) δ: 32.2 (t, C-1), 30.1 (t, C-2), 88.4 (d, C-3), 41.4 (s, C-4), 47.5 (d, C-5), 21.0 (t, C-6), 26.3 (t, C-7), 47.6 (d, C-8), 19.8 (s, C-9), 26.4 (s, C-10), 26.6 (t, C-11), 34.8 (t, C-12), 46.9 (s, C-13), 45.3 (s, C-14), 43.3 (t, C-15), 72.4 (d, C-16), 52.4 (d, C-17), 20.7 (q, C-18), 30.1 (t, C-19), 34.8 (d, C-20), 17.5 (q, C-21), 86.9 (d, C-22), 106.0 (s, C-23), 83.3 (d, C-24), 83.6 (s, C-25), 27.8 (q, C-26), 24.9 (q, C-27), 19.7 (q, C-28), 25.8 (q, C-29), 15.5 (q, C-30), 107.6 (d, C-1′), 75.6 (d, C-2′), 78.7 (d, C-3′), 71.3 (d, C-4′), 67.2 (t, C-5′).

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