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Three new sesquiterpene alkaloids from the root of *Tripterygium wilfordii*

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Abstract

Three new sesquiterpene alkaloids, 1-desacetylwilforgine **(1)**, 1-desacetylwilforine **(2)**, and 9′-hydroxy-2-nicotinoylwilforine **(3)** were isolated from the roots of *Tripterygium wilfordii* Hook f., along with six known alkaloids. Their structures were established on the basis of spectral analysis.

Keywords: *Tripterygium wilfordii*; Celastraceae; Sesquiterpene alkaloid

Tripterygium wilfordii Hook f. has been used as traditional Chinese medicine to treat cancer, rheumatoid arthritis, autoimmune diseases, skin disorders, and in male-fertility control for many years [1]. In the previous studies, we isolated a new triterpenoid 3,4,6-trihydroxy-2-oxo-1(10), 3,5,7-tetraen-23, 24-nor-D: A-friedooleana-29-oic acid from the root of *T. wilfordii* [2]. In continuing studies on the chemical components of this species, we have isolated three new sesquiterpene alkaloids 1-desacetylwilforgine (**1**), 1-desacetylwilforine (**2**), 9′-hydroxy-2-nicotinoylwilforine (**3**), together with six known alkaloids: wilforgine (**4**) [1], wilfordine (**5**) [3], wilfortrine (**6**) [4], wilforine (**7**) [5], wilforidine (**8**) [6], wifornine (**9**) [7] from the root of *T. wilfordii*.

The roots of *T. wilfordii* were collected in Taining Prefecture, Fujian Province (2007) and identified by Jin Ming, the associate chief pharmacist of Fujian Institute for Drug Control, Fuzhou, China.

The air-dried root barks (5.3 kg) of *T. wilfordii* were extracted with 75% EtOH (3 × 20 L) for 10, 8 and 8 h, respectively. The EtOH extracts were evaporated to dryness under reduced pressure, and the residues were suspended in H_2O. The suspensions were extracted with petroleum ether, ethyl acetate, and n-butanol in turn. Then the ethyl acetate part was evaporated to give 174 g of a residue, which was subjected to CC (silica gel 200–300 mesh, 1.2 kg). The column was eluted with solvents of increasing polarity petroleum ether/ethyl acetate to give 10 frs (fr: 1–10). Fr 5 was further purified by HPLC (CH_3CN:H_2O 55:45) to give **1** (11.6 mg), **2** (5.5 mg), **3** (3.5 mg).

Compound **1** was obtained as white needle crystals. Its HREI-MS showed at *m/z* 816.2710 $[M+H]^+$ (calcd. for $C_{39}H_{45}NO_{18}$ 816.2714). The ^1H NMR spectrum of **1** (Table 1) showed two tetrarnary and one tertiary methyl groups

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Table 1
^{1}H and ^{13}C NMR data of **1**–**3** in $CDCl_3$ (400 MHz for ^{1}H, 100 MHz for ^{13}C, δ in ppm, J in Hz).

No.	**1**			**2**		**3**	
	δ_C	δ_H	HMBC (position)	δ_C	δ_H	δ_C	δ_H
1	74.1	5.41 d (3.2)	9	74.2	5.32 d (3.2)	73.1	5.75 d (3.6)
2	69.1	5.52 dd (3.2, 3.2)	2-OFur	69.3	5.50 d (3.2)	68.9	5.54 d (3.6)
3	75.0	5.08 d (2.8)	10, 11′	75.1	5.14 d (2.8)	77.7	5.09 d (3.2)
4	71.7			71.2		69.8	
5	73.4	5.71 d (3.6)	5-OAc	73.6	5.76 d (3.2)	73.6	6.93 s
6	52.5	2.45 d (4.0)	5, 7	52.4	2.46 d (4.0)	51.1	2.40 d (4.0)
7	69.3	5.38 d (3.6)	5, 7-OAc	69.8	5.49 d (3.6)	69.8	5.47 dd (3.2, 3.2)
8	71.0	5.40 d (6.0)	9, 8-OAc	71.2	5.37 d (5.6)	70.7	5.45 d (6.0)
9	50.9			50.7		52.0	
10	92.7			92.7		94.0	
11a	60.9	5.42 d (13.2)	8, 9, 10, 11-OAc	61.1	5.40 d (13.2)	60.3	5.53 d (13.6)
11b		4.36 d (13.2)			4.46 d (13.2)		4.38 d (13.6)
12	23.4	1.89 s	3, 4, 10	23.9	1.99 s	23.0	1.69 s
13	85.0			85.1		84.8	
14	18.2	1.67 s	6, 13, 15	18.2	1.67 s	17.9	1.6 s
15a	71.1	5.86 d (12.0)	13, 12′	71.2	5.38 d (12.0)	69.8	5.78 d (12.0)
15b		3.73 d (12.0)	12′		3.73 d (12.0)		3.74 d (12.0)
2′	153.6	8.77 dd (4.8, 1.6)	3′,4′	153.6	8.76 dd (4.8, 1.6)	152.3	8.71 dd (4.8, 1.6)
3′	121.2	7.29 dd (4.8, 8.0)	5′	121.2	7.29 dd (4.8, 8.0)	120.6	7.22 dd (4.8, 8.0)
4′	138.7	8.37 dd (1.6, 8.0)	6′	138.7	8.36 dd (1.6, 8.0)	137.8	8.37 dd (1.6, 8.0)
5′	123.9			123.9		125.5	
6′	165.1			164.9		165.2	
7a′, 7b′	33.0	4.10 m, 2.89 m		33.0	4.06 m, 2.86 m	31.4	3.97 m, 2.96 m
8a′, 8b′	33.5	2.38 m, 1.84 m		33.4	2.38 m, 1.98 m	38.5	2.48 m, 2.19 m
9′	38.1	2.30 m		38.2	2.20 m	77.7	
10′	19.0	1.18 d (6.4)	8′, 9′, 11′	18.9	1.20 d (6.4)	28.0	1.27 s
11′	175.3			175.3		172.5	
12′	167.1			167.1		168.0	
1″				129.0			
2″	148.5	8.20 s	3″, 4″, 5″	129.8	8.02 t (1.2, 7.6)	151.2	9.31 d (1.6)
3″	118.4			128.8	7.50 t (7.6, 7.6)	124.8	
4″	109.6	6.80 d (1.2)	3″, 5″	133.8	7.63 t (1.2, 7.6)	137.3	8.15 dd (1.6, 3.2)
5″	144.3	7.49 s	3″	128.8	7.50 t (7.6, 7.6)	123.7	7.50 dd (1.6, 3.2)
6″				129.8	8.02 t (1.2, 7.6)	154.3	8.88 dd (1.6, 3.2)
2-C=O	161.0			165.3		163.7	
1-Ac						20.5	2.19 s
						169.7	
5-Ac	21.0	2.16 s	5-OAc	20.9	2.18 s	21.0	2.22 s
	170.0			169.8		169.8	
7-Ac	20.5	1.85 s	7-OAc	20.6	1.88 s	21.0	2.19 s
	169.5			169.7		170.2	
8-Ac	20.4	1.93 s	8-OAc	20.4	1.90 s	20.5	1.97 s
	169.0			169.0		169.0	
11-Ac	21.1	2.07 s	11-OAc	21.0	1.85 s	21.5	1.86 s
	170.1			170.0		170.0	

[δ 1.67(H-14), and 1.89(H-12), 1.18(H-10′)], four acetyl methyl groups [δ 1.85(OAc-7), 1.93(OAc-8), 2.07(OAc-11), 2.16(OAc-5)], two methylene groups connected to oxygen atoms [δ 5.86 and 3.73(d, each 1H, J = 12.0 Hz, H-15), δ 5.42 and 4.36(d, each 1H, J = 13.2 Hz, H-11)], six methine groups connected to oxygen atoms [δ 5.41(H-1), 5.52(H-2), 5.08(H-3), 5.71(H-5), 5.38(H-7), 5.40(H-8)]. Also evidence in the ^{1}H NMR spectrum were, a 5′, 6′-substituted pyridine ring [δ 8.77(dd, J = 1.6, 4.8 Hz), 8.37(dd, J = 1.6, 8.0 Hz), 7.29(dd, J = 8.0, 4.8 Hz)] and a 3-furanoyl group [δ 8.20(s, 1H), 7.49(s, 1H), 6.80(d, 1H, J = 1.2 Hz)]. The ^{13}C and DEPT NMR spectra showed the presence of four acetyls, ten tetrarnary carbons, fourteen methine carbons, four methylene and three methyl. The ^{1}H and ^{13}C NMR spectroscopic data of **1** were similar to those of **4** [1] indicating that these compounds have the same basic skeleton.