Sesquiterpenes from Cultures of the Basidiomycete *Conocybe siliginea*

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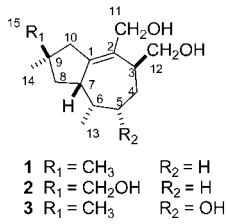
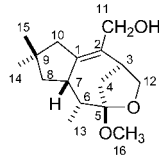
Six new tremulane sesquiterpenes, conocenol A–D (**2**–**5**), conocenolide A (**6**), and conocenolide B (**7**), have been isolated from cultures of the basidiomycete *Conocybe siliginea*. The structures of **2**–**7** were elucidated by the analysis of spectroscopic data, including heteronuclear multiple-bond correlation, heteronuclear single-quantum coherence, and ^{1}H,^{1}H correlation spectroscopy, and a comparison with known analogues.

The tremulanes constitute a class of sesquiterpenes that are characterized by an unusual carboskeletal array and were first isolated in 1993 from the aspen tree rotting fungus *Phellinus tremulae*.[1] Thus far, only 10 tremulane-type compounds have been reported. Over the last 10 years, our main research focus has been secondary metabolites from the untapped resources of higher fungi found in China.[2–5] The genus toward *Conocybe* belongs to the order Agaricales and family Bolbitiaceae and comprises more than 240 species widely distributed in the world. Previous investigations of basidiomycetes in the genus *Conocybe* have reported the isolation of hallucinogenic or toxic compounds, such as psilocybin,[6,7] psilocin,[7] and α-amanitin[8] that interfere with the normal action of brain serotonin in a similar way to lysergic acid diethylamide (LSD).[9–11] In this paper, we report the isolation and structure elucidation of six new tremulane sesquiterpenes (**2**–**7**) from the culture broth of *Conocybe siliginea*.

C. siliginea was collected in the Linglang county of the Yunnan Province, People's Republic of China, in July 2003. The fungus was grown in shake cultures (150 rpm) using PD medium. After the fungus was cultured for 25 days at 22 °C, the whole culture broth of *C. siliginea* (20 L) was filtered and the filtrate was extracted twice with EtOAc. The crude EtOAc extract (5.6 g) was subjected to column chromatography over silica gel with a $CHCl_3$/MeOH gradient to give eight fractions, which were further purified by silica gel and Sephadex LH-20 column chromatography to afford **2** (2.8 mg), **3** (3.7 mg), **4** (3.6 mg), **5** (4.7 mg), and a mixture of **6** and **7** (8.6 mg).



1 $R_1 = CH_3$ $R_2 = H$
2 $R_1 = CH_2OH$ $R_2 = H$
3 $R_1 = CH_3$ $R_2 = OH$



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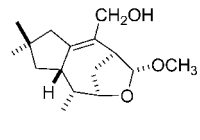

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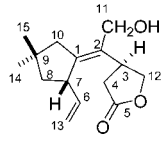

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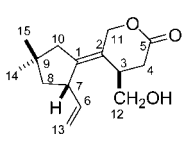

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Compound **2** was obtained as an oil and gave a quasi-molecular ion peak at *m/z* 277 $[M + Na]^+$ in its positive electrospray ionization mass spectrometry (ESIMS) spectrum and was assigned a molecular formula of $C_{15}H_{26}O_3$, as determined by HRESIMS (found $[M + Na]^+$ 277.1772, calcd for 277.1779) and nuclear magnetic resonance (NMR) data. The IR spectrum showed absorptions for a hydroxy group (3426 cm^{-1}) and a C=C double bond (1636 cm^{-1}). The ^{1}H NMR spectrum of **2** (Table 1) showed resonances for one secondary methyl [δ_H 0.90 (3H, d, $J = 7.2$ Hz)], one tertiary methyl [δ_H 1.07 (3H, s)], and three oxymethylene groups [δ_H 3.70 (2H, m), 3.22 (2H, s), and 4.09 (1H, d, $J = 11.2$ Hz), 3.94 (1H, d, $J = 11.2$ Hz)]. The ^{13}C and distortionless enhancement by polarization transfer (DEPT) NMR spectra of **2** (Table 3) revealed 15 carbon resonances, including two sp^2 quaternary carbons at δ_C 145.1 and 133.9, three oxymethylene carbons at δ_C 66.2, 61.7, and 68.7, as well as two methyl (δ_C 11.8 and 23.9), four methylene (δ_C 21.9, 33.0, 41.6, and 43.7), three methine carbons (δ_C 45.9, 33.2, and 47.4), and one quaternary carbon (δ_C 43.4). The above-mentioned data exhibited similarities with those of **1**,[1] which suggested that both compounds possessed the same tremulane skeleton. The key difference between the two compounds was that the methyl group (δ_C 26.9, C-15) in **1** was replaced by the hydroxymethylene moiety (δ_C68.7, C-15) in **2**. This assignment was in accordance with the observation of the downfield shifts of C-9 at δ_C 43.4 ($\Delta\delta$ +6.4 ppm) along with the upfield shift of C-8 at δ_C 41.6 ($\Delta\delta$ −3.9 ppm) and C-10 at δ_C 43.7 ($\Delta\delta$ −4.3 ppm) in **2** and confirmed by the heteronuclear multiple-bond correlations (HMBCs) of H-15 (δ_H 3.22, s) to C-8, C-9, and C-10. The relative configuration of **2** was determined by the ROESY correlations of H-5β (δ 1.96) with H-7 and H-12, 13-Me with H-5α (δ1.63) and H-8α (δ 1.34), and H-10α (δ 1.88) with H-8α and 14-Me, indicating that H-6 and H-7 were β-oriented, while H-3 and 14-Me were α-oriented. From the above evidence, the structure of conocenol A was established as **2**.

Compound **3** was obtained as an oil and found to possess the same molecular formula as that of **2** based on the HRESIMS (found $[M + Na]^+$ 277.1786, calcd for 277.1779) and NMR data. A comparison of the ^{13}C NMR data of **3** with those of **1** suggested that they shared the same planar structure, except for the presence of a hydroxyl group at C-5 in **3** instead of C-15, causing the downfield chemical shift of C-4 at δ_C 30.2 ($\Delta\delta$ +7.7 ppm), C-5 at δ_C 72.9 ($\Delta\delta$ +40.3 ppm), and C-6 at δ_C 40.5 ($\Delta\delta$ +8.9 ppm) and the upfield chemical shift of C-13 at δ_C 6.1($\Delta\delta$ −5.5 ppm) in **3**. HMBCs of H-5 (3.98, m) with C-4 and 13-Me further supported the above assignment. On the basis of the rotating-frame Overhauser enhancement spectroscopy (ROESY) correlations, the relative configuration at C-3, C-6, and C-7 in **3** was the same as those of **1** and the α orientation of the 5-OH group in **3** was determined by the ROESY correlation from H-5 to H-7. Hence, the structure of conocenol B was deduced as **3**.

Compound **4** was assigned the molecular formula $C_{16}H_{26}O_3$ by positive HRESIMS (found $[M + Na]^+$ 289.1775, calcd for

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Table 1. ^{1}H (400 MHz) NMR Data for Compounds **2**–**5**[a]

number	**2**[b]	**3**[b]	**4**[c]	**5**[c]
3	2.56 (m)	2.64 (m)	3.00 (dd, 7.2, 7.2)	2.74 (d, 7.6)
4	1.75 (m)	1.95 (m)	2.04 (m)	2.05 (m)
		1.73 (ddd, 12.8, 12.8, 3.6)	1.74 (d, 12.4)	1.82 (d, 12.4)
5	1.96 (br t, 13.6)	3.98 (m)		4.60 (dd, 7.2, 7.2)
	1.63 (m)			
6	1.75 (m)	1.89 (m)	2.08 (m)	2.06 (m)
7	3.04 (br t, 9.4)	2.92 (br t, 9.4)	3.22 (m)	3.24 (m)
8	1.73 (m)	1.59 (ddd, 12.0, 12.0, 2.0)	1.47 (d, 10.2)	1.46 (dd, 12.0, 12.0)
	1.34 (dd, 12.8, 10.8)	1.51 (dd, 12.0, 12.0)		1.39 (br t, 12.0)
10	2.50 (dd, 16.0, 1.6)	2.31 (dd, 15.2, 2.0)	2.24 (d, 15.6)	2.25 (dd, 15.6, 2)
	1.88 (br d, 16.0)	1.99 (br d, 15.2)	1.97 (br d, 15.6)	1.95 (br d, 15.6)
11	4.09 (d, 11.2)	4.07 (d, 11.6)	4.15 (d, 11.6)	4.15 (d, 11.6)
	3.94 (d, 11.2)	3.96 (d, 11.6)	4.01 (d, 11.6)	4.03 (d, 11.6)
12	3.70 (m)	3.66 (m)	4.31 (dd, 10.0, 6.8)	5.01 (s)
			3.89 (d, 10.0)	
13	0.90 (d, 7.2)	0.82 (d, 6.8)	0.80 (d, 6.8)	0.74 (d, 7.0)
14	1.07 (s)	1.11 (s)	1.09 (s)	1.09 (s)
15	3.23 (d, 10.0)	0.91 (s)	0.90 (s)	0.93 (s)
	3.21 (d, 10.0)			
16			3.31 (s)	3.34 (s)

[a] Chemical shifts (δ) are in parts per million (ppm), and J is in hertz. [b] Measured in CD_3OD. [c] Measured in $CDCl_3$.

289.1779), corresponding to four degrees of unsaturation. This was corroborated by the ^{13}C and DEPT NMR spectra, which displayed 16 resonances for two quaternary carbons (δ_C 114.1 and 37.2), two disubstituted olefinic carbons (δ_C 143.1 and 136.2), three methine (δ_C 39.6, 39.8, and 41.5), five methylene (δ_C 32.9, 43.9, 47.6, 65.5, and 74.9), and four methyl (δ_C 10.9, 28.8, 26.7, and 49.0) carbons. These observations, in combination with the molecular formula, revealed that **4** possessed three rings and one OH group. Using 2D NMR spectra and a comparison of the ^{13}C NMR data with those of **1**, compound **4** was postulated as having a tremulane-type skeleton possessing structural fragment a, which is also present in compound **1**. The HMBC spectrum displayed the following correlations: H-3 with C-4, C-5, and C-12, H-4 with C-2, and H-12 with C-2, C-3, C-4, and C-5. The above observations, coupled with the proton spin system H-12/H-3/H-4, deduced from the ^{1}H–^{1}H correlation spectroscopy (COSY) spectrum, led to the determination of the structure of fragment b. The additional HMBCs of H-3 to C-1, C-2, and C-11 and 13-Me to C-5 established the direct linkage of C-5 with C-6 and C-2 with C-3, which permitted fragments a and b to be joined to get the planar structure of compound **4**. The *O*-methyl group was attached to C-5 in agreement with the cross-peak between 16-Me and C-5 in the HMBC spectrum. The relative configuration of the four stereogenic carbons, C-3, C-5, C-6, and C-7, was defined by ROESY, which showed correlations of H-3 and 16-OMe to H-4β (δ 2.04), 13-Me to H-4α (δ 1.74), and H-7 to H-6. This observation implied that H-3, H-6, H-7, and 5-*O*-methyl all possessed β orientations. On the basis of the above evidence, the structure of conocenol C was established as **4**.

Compound **5** was isolated as an oil. It possessed the molecular formula $C_{16}H_{26}O_3$, identical with **4**, as revealed by positive HRESIMS (*m*/*z* 289.1771 $[M + Na]^+$, calcd for 289.1779). With the assistance of 2D NMR spectra including ^{1}H–^{1}H COSY, heteronuclear single-quantum coherence (HSQC), and HMBC, compound **5** was shown to possess the same tricyclic carbon skeleton as that of compound **4**. Furthermore, a comparision the ^{13}C NMR data of **5** with those of **4** showed that they both were similar, except for the significant downfield shift at δ_C 109.9 because of C-12 ($\Delta\delta$ +35 ppm) and the upfield shift at δ_C 85.4 because of C-5 ($\Delta\delta$ −28.7 ppm) relative to those of **4**. Thus, **5** was presumed to be an isomer of **4**, differing in the position of the *O*-methyl group. The HMBC from Me-16 (δ_H 3.34, s) to C-12 exhibited that the *O*-methyl group was located at C-12 in **5**. On the basis of the ROESY correlations, the relative configuration at C-3, C-5, C-6, and C-7 in **5** was the same as those of **4** and the β orientation of H-12 was determined by the cross-peak between H-12 and H-7. From biogenetic considerations, compound **3** seemed to be the precursor of **5**, indicating the relative configuration at C-5 contrary to that deduced from ROESY correlations in **5**. The C-5 epimer of compound **3** had not been found in our current work. From the above evidence, the structure of conocenol D was determined as **5**.

Table 2. ^{1}H (500 MHz) and ^{13}C (125 MHz) NMR Data for Compounds **6** and **7** ($CDCl_3$)[a]

	6		**7**	
number	δ_H	δ_C	δ_H	δ_C
1		148.3 s		143.2 s
2		129.0 s		123.5 s
3	3.81 (m)	37.1 d	3.38 (m)	35.4 d
4	2.57 (m)	32.8 t	2.76 (dd, 15.5, 4.5)	32.1 t
			2.57 (overlapped)	
5		177.6 s		172.5 s
6	5.69 (m)	142.0 d	5.63 (m)	139.9 d
7	3.37 (m)	46.1 d	3.44 (m)	46.1 d
8	1.80 (m)	48.1 t	1.80 (m)	48.4 t
	1.37 (m)		1.37 (m)	
9		37.8 s		37.8 s
10	2.30 (dd, 15.5, 2)	46.0 t	2.14 (d, 13.6)	46.3 t
	2.19 (br d, 15.5)		2.10 (d, 13.6)	
11	4.26 (overlapped)	60.3 t	4.81 (d, 14.0)	69.3 t
	4.16 (d, 12.0)		4.70 (d, 14.0)	
12	4.36 (dd, 9.0, 9.0)	71.2 t	3.68 (dd, 10.0, 4.5)	63.3 t
	4.27 (overlapped)		3.55 (dd, 10.0, 8.5)	
13	4.99 (overlapped)	113.5 t	5.05 (overlapped)	114.5 t
14	1.10 (s)	28.6 q	1.10 (s)	28.5 q
15	0.90 (s)	27.2 q	0.90 (s)	27.3 q

[a] Chemical shifts (δ) are in ppm, and J is in hertz.

Compounds **6** and **7** were obtained as an inseparable mixture in the ratio of approximately 2:1 deduced from the intensity of ^{1}H NMR resonances. The ^{1}H and ^{13}C NMR spectra of these two compounds showed two sets of resonances, respectively, and could be assigned clearly to each compound because their unequal concentrations resulted in an obvious difference of resonance intensities in the ^{1}H NMR spectra. Both compounds had the same molecular formula of $C_{15}H_{22}O_3$ by positive HRESIMS (*m*/*z* 273.1456 $[M + Na]^+$, calcd for 273.1466) and NMR data, which suggested a sesquiterpene skeleton with five degrees of unsaturation. The ^{13}C NMR and DEPT NMR spectra of the mixture contained 15 carbon resonances for each compound that were attributable in each case to two methyl, six methylene (two oxygenated and one olefinic), three methine (one olefinic), and four quaternary carbons (two olefinic and one carbonyl). Consideration of the above data led to the conclusion that **6** and **7** possessed two rings and one OH group, respectively.

Table 3. ^{13}C (100 MHz) NMR Spectroscopic Data for Compounds **1**–**5**[a]

number	**1**[b]	**2**[c]	**3**[c]	**4**[b]	**5**[b]
1	145.6 s	145.1 s	144.9 s	143.1 s	144.1 s
2	132.3 s	133.9 s	133.1 s	136.2 s	133.1 s
3	45.4 s	45.9 d	43.4 d	39.6 d	47.3 d
4	22.5 t	21.9 t	30.2 t	32.9 t	29.7 t
5	32.6 t	33.0 t	72.9 d	114.1 s	85.4 d
6	31.6 d	33.2 d	40.5 d	39.8 d	35.7 d
7	46.0 d	47.4 d	43.9 d	41.5 d	41.0 d
8	45.5 t	41.6 t	46.4 t	43.9 t	43.6 t
9	37.0 s	43.4 s	38.3 s	37.2 s	36.8 s
10	48.0 t	43.7 t	48.4 t	47.6 t	47.6 t
11	65.6 t	66.2 t	65.9 t	65.5 t	65.6 t
12	63.2 t	61.7 t	62.4 t	74.9 t	109.9 d
13	11.6 q	11.8 q	6.1 q	10.9 q	12.0 q
14	28.5 q	23.9 q	28.8 q	28.8 q	28.9 q
15	26.9 q	68.7 t	27.2 q	26.7 q	26.7 q
16				49.0 q	54.8 q

[a] Chemical shifts (δ) are in ppm, and J is in hertz. [b] Measured in $CDCl_3$. [c] Measured in CD_3OD.

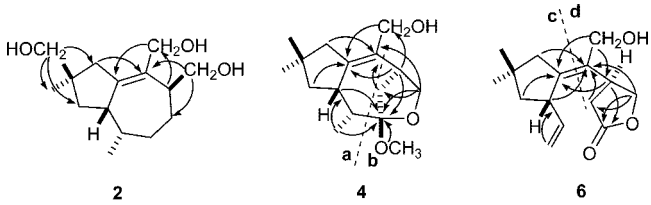

Figure 1. Key HMBCs of compounds **2**, **4**, and **6**.

A study of the spectroscopic data (^1H–^1H COSY, HSQC, and HMBC) prompted us to consider that **6** was a 5,6-*seco*-tremulane sesquiterpene. The ^1H–^1H COSY and HSQC spectrum revealed the presence of the partial structures CH (3)–CH_2 (4), CH (7)–CH_2(8), and CH (7)–CH (6) = CH_2 (13). Analysis of the HMBC spectrum of **6** showed correlations from both 14-Me and 15-Me to C-8, C-9, and C-10 and from both H-7 and H-10 to C-1 and C-2, which indicated that C-8 and C-10 were connected via C-9 and that C-7 and C-10 were attached to the sp^2 quaternary carbon C-1. Furthermore, the HMBCs of H-3, H-4, and H-12 with C-5, H-4 and H-12 with C-2, and H-3 with C-1 and C-2 revealed the connectivity of C-2 to C-3 and C-5 to C-12 via an oxygen atom to form a five-membered lactone ring. In addition, the hydroxymethylene group (δ_C 60.3) assigned as C-11 was indicated by the HMBCs from H-11 to C-1, C-2, and C-3. Thus, the planar structure of **6** was unambiguously established as shown in Figure 1. The geometry of the double bond at C-1 and C-2 was determined to be *E* based on the ROESY correlations of H-10 to H-11. The relative configuration of C-3 and C-7 could not be determined by the NMR data alone, because the bonds between C-2 and C-3 and C-6 and C-7 show free rotation.

By a comparison of the ^{13}C NMR data with those of **6**, compound **7** was suggested to possess the partial structure c of compound **6**, which was also confirmed by analysis of 2D NMR data. The HMBCs of H-3, H-4, and H-11 to C-5, H-4 to C-2, and H-11 to C-1 and C-2 allowed us to determine the presence of a six-membered lactone ring in **7**. Furthermore, the HMBC cross-peaks of H-12 with C-2, C-3, and C-4 indicated that the hydroxymethylene group (δ_C 63.3) was located at C-3. The six-membered lactone ring and fragment c were connected via the double bond, attributable to C-1 and C-2, to give the planar structure of **7**. The geometry of the double bond at C-1 and C-2 was confirmed to be *E* based on the ROESY correlation of H-11 to H-10. ROESY correlations were inconclusive in establishing the relative configuration of **7** as a result of overlapping resonances in the mixture.

Biogenetically, we could assume that compounds **6** and **7** are biosynthesised via steps involving the equivalents of Baeyer–Villiger oxidation and elimination of compound **3** followed by lactonization, which led us to propose that compounds **6** and **7** should share the same configuration at C-3 and C-7 as that already established for compound **3**.

Experimental Section

General Experimental Procedures. Optical rotations were measured on a Horiba SEPA-300 polarimeter. IR spectra were recorded on a Bruker Tensor 27 spectrometer with KBr pellets. Both 1D and 2D NMR experiments were performed on a Bruker AM-400 or DRX-500 spectrometer with tetramethylsilane (TMS) as the internal standard. Mass spectra were recorded on a VG Auto Spec-3000 or an API QSTAR Pulsar 1 spectrometer. Column chromatography was performed on silica gel (200–300 mesh; Qingdao Marine Chemical Ltd., Qingdao, People's Republic of China) and Sephadex LH-20 (Amersham Biosciences, Sweden). TLC analysis was carried out on silica gel GF_{254} precoated plates (0.20–0.25 mm; Qingdao) with detection by heating silica gel plates sprayed with 10% H_2SO_4 in ethanol.

Fungal Material. *C. siliginea* was isolated from the tissue culture of its fruiting bodies collected from a moist woodland (dominated by pines) of the Linglang county in the Yunnan Province, People's Republic of China, in July 2003, and authenticated by Prof. Mu Zang, Kunming Institute of Botany, Chinese Academy of Sciences (CAS). A voucher specimen (KIB03071801) was deposited in the Herbarium of Kunming Institute of Botany, CAS.

Fermentation and Isolation. The culture medium consisted of potato (peel off) (200 g), glucose (20 g), KH_2PO_4 (3 g), $MgSO_4$ (1.5 g), citric acid (0.1 g), and thiamin hydrochloride (10 mg) in 1 L of deionized H_2O. The fungus was grown in reagent bottles (500 mL; media of 300 mL). The pH was adjusted to 6.5 before autoclaving. Fermentation was carried out on a shaker at 22 °C and 150 rpm for 25 days.

The whole culture broth of *C. siliginea* (20 L) was filtered, and the filtrate was extracted twice with EtOAc. The organic layer was concentrated under reduced pressure to give an oily residue (5.6 g) that was subjected to column chromatography over silica gel (200–300 mesh) eluting with $CHCl_3$/MeOH (from 1:0 to 0:1) to afford fractions A–H. Fraction B was subjected to further column chromatography over silica gel, eluting with a gradient of EtOAc/petroleum ether (from 15:1 to 1:1), to give fractions B_1–B_4. Subfraction B_3 was repeatedly chromatographed on a silica gel column eluted with $CHCl_3$/MeOH (125:1) to afford a mixture of **6** and **7** (8.6 mg). Subfraction B_4 was purified by a Sephadex LH-20 column eluting with MeOH and repeated chromatography over silica gel using $CHCl_3$/MeOH (100:1) as the eluent to yield compounds **4** (3.6 mg) and **5** (4.7 mg). Fraction C, obtained from $CHCl_3$/MeOH (97:3), was passed over a Sephadex LH-20 column with MeOH as the eluent and then repeatedly applied to a silica gel column eluted with $CHCl_3$/MeOH (55:1) to yield compounds **2** (2.8 mg) and **3** (3.7 mg).

Conocenol A (2): colorless oil. $[\alpha]_D^{14.5}$ +41.1 (*c* 0.15, MeOH). IR (KBr) ν_{max}: 3426, 2926, 2868, 1636, 1463, 1027 cm^{-1}. ^1H and ^{13}C NMR: see Tables 1 and 3, respectively. ESIMS *m/z*: 277 $[M + Na]^+$. HRESIMS *m/z*: 277.1772 $[M + Na]^+$ (calcd for $C_{15}H_{26}O_3Na$, 277.1779).

Conocenol B (3): colorless oil. $[\alpha]_D^{14.7}$ +84.4 (*c* 0.15, MeOH). IR (KBr) ν_{max}: 3426, 2951, 2931, 2880, 1630, 1461, 1012 cm^{-1}. ^1H and ^{13}C NMR: see Tables 1 and 3, respectively. ESIMS *m/z*: 277 $[M + Na]^+$. HRESIMS *m/z*: 277.1786 $[M + Na]^+$ (calcd for $C_{15}H_{26}O_3Na$, 277.1779).

Conocenol C (4): colorless oil. $[\alpha]_D^{16.1}$ +12.3 (*c* 0.19, $CHCl_3$). IR (KBr) ν_{max}: 3425, 2952, 2929, 2866, 1729, 1464, 1132, 1054 cm^{-1}. ^1H and ^{13}C NMR: see Tables 1 and 3, respectively. ESIMS *m/z*: 289 $[M + Na]^+$, 555 $[2M + Na]^+$. HRESIMS *m/z*: 289.1775 $[M + Na]^+$ (calcd for $C_{16}H_{26}O_3Na$, 289.1779).

Conocenol D (5): colorless oil. $[\alpha]_D^{16.5}$ +69.3 (*c* 0.22, $CHCl_3$). IR (KBr) ν_{max}: 3427, 2952, 2932, 2868, 1637, 1463, 1377, 1099, 1059 cm^{-1}. ^1H and ^{13}C NMR: see Tables 1 and 3, respectively. ESIMS *m/z*: 289 $[M + Na]^+$, 555 $[2M + Na]^+$. HRESIMS *m/z*: 289.1771 $[M + Na]^+$ (calcd for $C_{16}H_{26}O_3Na$, 289.1779).

Conocenolide A (6) and Conocenolide B (7) (Mixture, 2:1): colorless oil. $[\alpha]_D^{16.6}$ −28.3 (*c* 1.06, $CHCl_3$). IR (KBr) ν_{max}: 3432, 2955, 2927, 2869, 1772, 1664, 1637, 1465, 1417, 1385, 1367, 1183, 1023, 916, 852, 678 cm^{-1}. ^1H and ^{13}C NMR: see Table 2. FABMS *m/z*: 249 $[M - H]^-$. HRESIMS *m/z*: 273.1456 $[M + Na]^+$ (calcd for $C_{15}H_{22}O_3Na$, 273.1466).

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References and Notes

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