New Sesquiterpenes from Edible Fungus *Clavicorona pyxidata*

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Three new sesquiterpenes, pyxidatol A–C (**1**–**3**, resp.), were obtained from the fermentation culture of *Clavaria pyxidata*, together with two known ones, tsugicoline E (**4**) and omphadiol (**5**). Their structures were elucidated through spectroscopic analyses, including 1D- and 2D-NMR experiments, HR-ESI-Q-TOF mass spectrometry, chemical correlation, and X-ray single-crystal diffraction.

Introduction. – Many sesquiterpenes have been isolated from the genus *Clavicorona*. These include clavicoronic acid [1], a novel inhibitor of reverse transcriptases from *Clavicorona pyxidata*, and four sesquiterpenes of protoilludane origin, divaricatines A and B, 7-epitsugicoline H and tsugicoline M, and the norsesquiterpene tsugicoline L and tsugicoline I from *Clavicorona divaricata* [2]. *Clavicorona pyxidata* is a wild mushroom which is widely used for curing gastric pain, dyspepsia, gout and heat-toxicity in traditional medicine in China. *Clavicorona pyxidata* YB2005 was isolated from the wild fruiting body in Jilin Province and identified by ITS methods. In exploring new bioactive metabolites from this medicinal mushroom, two new protoilludanes, named pyxidatol A (**1**), pyxidatol B (**2**), and one analogue of omphadiol, pyxidatol C (**3**), were isolated from this strain, together with two known ones, tsugicoline E (**4**) [3] and omphadiol (**5**) [4]. The structures of **4** and **5** were elucidated by their NMR data and chemical correlation and X-ray single crystal diffraction.

Results and Discussion. – *Clavicorona pyxidata* was cultured in *Petri* dishes with PDA medium and a total of 10 l for 25 d at 25°. The fermentation culture was extracted with AcOEt/MeOH/AcOH (80:15:5, *v*/*v*/*v*). The crude extract was partitioned between AcOEt and H_2O. The organic layer was dried over Na_2SO_4 (anh.) and concentrated *in vacuo* to afford 15.0 g extract (dark oil). The extract was purified by repeated column chromatography (*RP-18*, *Sephadex LH-20*, and SiO_2) to afford three new and two known sesquiterpenes.

Pyxidatol A (**1**) was obtained as a colorless oil. The molecular formula was determined as $C_{15}H_{26}O_6$ on the basis of HR-ESI-Q-TOF MS. The IR absorption at 3466 cm^{-1} indicated the presence of OH groups. The ^{13}C-NMR (DEPT) spectrum

(*Table 1*) of **1** revealed the presences of 15 signals: three Me, three CH_2 (one being oxygenated), five CH (three being oxygenated), and four quaternary C-atoms (two being oxygenated). Analysis of the ^{1}H-NMR spectrum (*Table 2*) indicated the presence of three Me signals at δ(H) 0.97 (*s*), 1.00 (*s*), and 1.07 (*s*). The connectivity of the H- and C-atoms was established by a HSQC spectrum. HMBC Data from Me(8) to C(4), C(6), C(7), and C(9), from H–C(6) to C(8), C(7), C(9), and C(5), and from H–C(5) to C(6), C(4), and C(2), and analysis of the ^{1}H,^{1}H-COSY data afforded fragment **1a** (*Fig. 1*). The ^{1}H,^{1}H-COSY cross-peaks between H–C(3) and H–C(13),

Table 1. *^{13}C-NMR Spectroscopic Data of* **1**, **2**, *and* **4**. Recorded at 150 MHz, δ in ppm, *J* in Hz. Arbitrary atom numbering.

Position	**1**	**2**	**4**
1	61.1 (*t*)	64.8 (*t*)	71.6 (*t*)
2	68.1 (*s*)	137.8 (*s*)	79.8 (*s*)
3	73.8 (*d*)	126.5 (*d*)	73.2 (*d*)
4	70.2 (*s*)	45.8 (*d*)	54.9 (*d*)
5	76.3 (*d*)	75.2 (*d*)	106.4 (*s*)
6	78.0 (*d*)	70.1 (*d*)	72.8 (*d*)
7	40.1 (*s*)	44.9 (*s*)	34.9 (*s*)
8	14.1 (*q*)	20.7 (*q*)	16.8 (*q*)
9	47.6 (*d*)	43.5 (*d*)	45.3 (*d*)
10	43.3 (*t*)	37.7 (*t*)	41.8 (*t*)
11	37.6 (*s*)	43.2 (*s*)	34.3 (*s*)
12	43.2 (*t*)	43.9 (*t*)	41.9 (*t*)
13	45.8 (*d*)	39.0 (*d*)	42.0 (*d*)
14	32.4 (*q*)	72.0 (*t*)	30.5 (*q*)
15	31.8 (*q*)	27.5 (*q*)	30.2 (*q*)

Table 2. *^{1}H-NMR Spectroscopic Data of* **1**, **2**, *and* **4**. Recorded at 600 MHz in CD_3OD (**1**, **4**) and in $(CD_3)_2CO$ (**2**); δ in ppm, *J* in Hz. Arbitrary atom numbering.

Position	**1**	**2**	**4**
1a	4.13 (*d*, *J* = 12.6)	3.97 (*s*, 2 H)	4.02 (*d*, *J* = 10.8)
1b	3.86 (*d*, *J* = 12.6)		3.91 (*d*, *J* = 10.8)
3	3.27 (*d*, *J* = 12.0)	5.45 (*s*)	3.61 (*d*, *J* = 11.4)
4		2.03 (*dd*, *J* = 13.1, 6.7)	1.93 (*s*, 3 H)
5	4.34 (*d*, *J* = 6.2)	3.87 (overlap)	
6	3.62 (*d*, *J* = 6.2)	3.86 (overlap)	4.14 (*s*)
8	0.97 (*s*, 3 H)	1.17 (*s*, 3 H)	1.06 (*s*, 3 H)
9	2.35–2.40 (*m*)	2.06 (overlap)	2.19–2.24 (*m*)
10α	1.38 (*dd*, *J* = 12.4, 6.6)	1.60 (*dd*, *J* = 12.7, 7.3)	1.51 (*dd*, *J* = 6.5, 12.4)
10β	1.30 (overlap)	0.94 (*t*, *J* = 12.7)	1.40 (*t*, *J* = 12.4)
12α	1.55 (*dd*, *J* = 14.0, 6.9)	1.94 (*dd*, *J* = 13.1, 8.1)	1.66 (*dd*, *J* = 6.8, 13.8)
12β	1.78 (*dd*, *J* = 14.0, 1.9)	1.34 (*d*, *J* = 13.1)	1.91 (*d*, *J* = 13.8)
13	2.04–2.09 (*m*)	2.60 (br. *s*)	2.15 (*ddd*, *J* = 6.4, 6.8, 11.2)
14	1.07 (*s*, 3 H)	0.82 (*s*, 3 H)	1.17 (*s*, 3 H)
15a	1.00 (*s*, 3 H)	3.25 (*dd*, *J* = 10.2, 3.4)	1.06 (*s*, 3 H)
15b		3.21 (*dd*, *J* = 10.2, 3.4)	

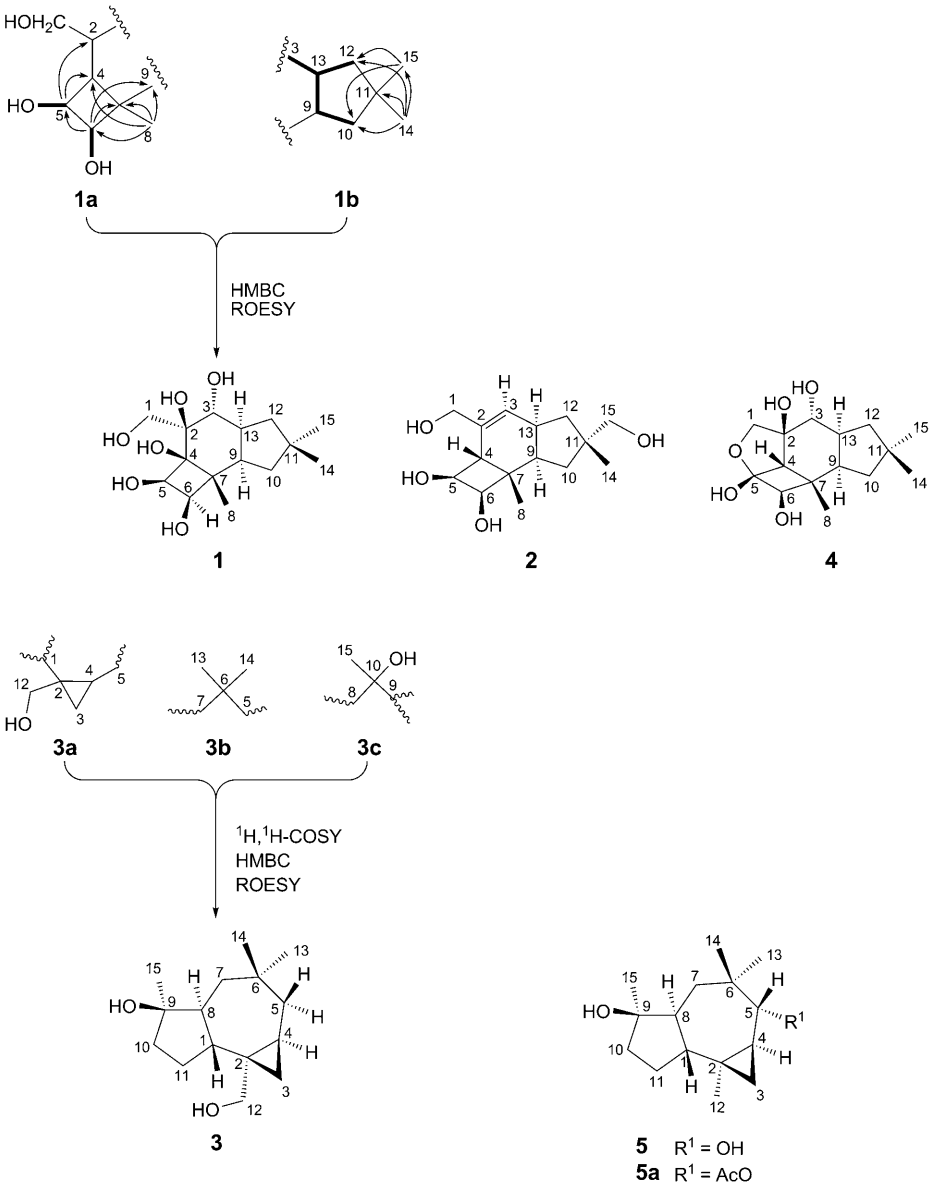

Fig. 1. *Structure fragments of* **1** *and* **3**, *and selected* ^{1}*H,*^{1}*H-COSY and HMBC data*

H–C(12) and H–C(13), H–C(9) and H–C(13), and H–C(9) and H–C(10), and HMBC data from Me(14) to C(10), C(11), C(12), and C(15) and from Me(15) to C(10), C(11), C(12), and C(14) led to establish a *gem*-dimethyl-substituted cyclopentane ring **1b** (*Fig. 1*). The HMBC experiments also showed long-range correlations between H–C(3) and C(2) and C(4) in fragment **1a**, and C(12) and C(13) in fragment

1b, which determined the connection of fragments **1b** and **1a** and gave the full structure. Therefore, the formula of **1** could be established.

The relative configuration of **1** was determined by analysis of the ROESY spectrum. The presence of ROESY correlations between H–C(9) and H–C(13), H–C(9) and H–C(6), H–C(9) and Me(15), H–C(5) and H–C(6), and H–C(5) and Me(15) indicated that H–C(9), H–C(6), H–C(13), H–C(5), and Me(15) are in α-orientation, while H–C(3) and Me(14) are in β-orientation (*Fig. 1*). All NMR assignments are consistent with literature values for the structurally similar, known compound tsugicoline E [3], and its relative configuration was confirmed by X-ray single-crystal diffraction of **4**. The compound **4** could be derived from **1** *via* regioselective oxidation at C(5) of **1**. Thus, from the above data, the structure of **1** was established.

Pyxidatol B (**2**) was isolated as a colorless oil. The molecular formula was determined as $C_{15}H_{24}O_4$ according to the HR-ESI-Q-TOF MS and NMR data. The IR absorptions at 3281, 2922, and 1652 cm^{-1} indicated the presence of OH and Me groups, and of a C=C bond. The NMR data of **2** were similar to those of **1**, except that the Me group at C(15) in **1** was replaced by a $HOCH_2$ group in **2**, and C(4) was a CH group, and the C=C bond was formed between C(2) and C(3) in **2**. The configuration of **2** was determined based on its ROESY correlations, as in the case of compound **1**.

Pyxidatol C (**3**) was obtained as a colorless oil. The molecular formula was determined as $C_{15}H_{26}O_2$ according to the HR-ESI-Q-TOF MS and NMR data. The IR spectrum (film) exhibits absorption bands for OH (3344 cm^{-1}) and Me groups (2926 cm^{-1}). The ^{13}C-NMR (DEPT) spectrum (*Table 3*) of **3** revealed the presence of 15 signals: three Me, six CH_2 (one being oxygenated), three CH, and three quaternary C-atoms (one being oxygenated). The ^{1}H-NMR and HSQC spectrum (*Table 3*) of **3** showed signals for the presence of two OH groups at δ(H) 2.80 (*s*) and δ(H) 3.42 (*t*, J = 5.0).

HMBC data from H–C(12) to C(1), C(2), C(3), and C(4) and from H–C(3) to C(1), C(2), C(4), C(5), and C(12), and the upfield chemical shifts of H_α–C(3) (δ(H) 0.70 (*dd*, J = 8.5, 3.8)), H_β–C(3) (δ(H) 0.14 (*t*, J = 3.8)) and H–C(4) (δ(H) 0.78 (*m*)) allowed the formulation of fragment **3a** (*Fig. 1*). HMBC Correlations from Me(13) to C(5), C(6), C(7), and C(14) and from Me(14) to C(5), C(6), C(7), and C(13) established the *gem*-dimethyl-substituted fragment **3b** (*Fig. 1*). The obvious HMBC data from Me(15) to C(8), C(9), and C(10), and from the H-atom (δ(H) 2.80 (*s*)) of the OH group to C(8), C(9), C(10), and C(15) led to fragment **3c** (*Fig. 1*). The ^{1}H,^{1}H-COSY cross-peaks between H–C(4) and H–C(5), CH_2(7) and H–C(8), and, H–C(1) and H–C(8) allowed to join the above fragments to form the constitutional formula of **3**, which is supported by the additional HMBC data from H–C(1) to C(3), C(2), and C(8), from CH_2(7) to C(1), C(5), C(6), C(13), and C(14), from CH_2(11) to C(1) and C(2), and from CH_2(10) to C(11) and C(8).

The comparison of ^{1}H- and ^{13}C-NMR data (*Table 3*) of **3** and omphadiol (**5**) [4] revealed that the two compounds have a similar basic structure. However, the Me(12) group and HO–CH(5) in **5** was replaced by a $HOCH_2$(12) substituent and CH_2(5) in **3**. The configuration of omphadiol (**5**) was determined by the crystal data of the 3,5-dinitrobenzoate derivative of omphadiol [4]. We also obtained crystals of the acetylated product of **5** (**5a**) (*Fig. 2*). The relative configuration of **3** was determined

Table 3. *^{1}H-and ^{13}C-NMR Data for* **3**, **5**, *and* **5a**. Recorded in $(CD_3)_2CO$ for **3** and **5**, $CDCl_3$ for **5a**.

Position	**3**		**5**		**5a**	
	δ(H) (multicity, J in Hz)	δ(C)	δ(H) (multicity, J in Hz)	δ(C)	δ(H) (multicity, J in Hz)	δ(C)
1	1.70 (overlap)	48.2 (*d*)	1.45 (overlap)	49.2 (*d*)	1.56 (overlap)	49.1 (*d*)
2		26.8 (*s*)		18.8 (*s*)		19.3 (*s*)
3α	0.70 (*dd*, J = 8.5, 3.8)	19.8 (*t*)	0.62 (*dd*, J = 8.2, 4.0)	22.5 (*t*)	0.67 – 0.68 (*m*)	23.0 (*t*)
3β	0.14 (*t*, J = 3.8)		0.34 (*t*, J = 4.4)		0.61 (overlap)	
4	0.73 – 0.78 (*m*)	18.8 (*d*)	0.51 – 0.55 (*m*)	30.3 (*d*)	0.61 (overlap)	27.0 (*d*)
5α	1.85 (overlap)	43.2 (*t*)	3.05 (*dd*, J = 9.0, 4.2)	79.8 (*d*)	4.55 (*d*, J = 8.6)	81.9 (*d*)
5β	1.11 – 1.14 (*m*)					
6		32.8 (*s*)		37.8 (*s*)		37.5 (*s*)
7α	1.42 – 1.45 (*m*)	43.7 (*t*)	1.42 (overlap)	42.4 (*t*)	1.48 (*d*, J = 13.3)	41.9 (*t*)
7β	1.17 – 1.24 (*m*)		1.25 – 1.29 (*m*)			
8	1.98 (overlap)	49.1 (*d*)	1.59 (overlap)	48.3 (*d*)	1.60 (overlap)	48.0 (*d*)
9		79.3 (*s*)		79.4 (*s*)		80.9 (*s*)
10α	1.72 (overlap)	41.6 (*t*)	1.65 (overlap)	41.4 (*t*)	1.68 (overlap)	41.4 (*t*)
10β	1.61 – 1.64 (*m*)		1.58 (overlap)		1.31 (overlap)	
11α	1.96 (overlap)	23.0 (*t*)	1.77 – 1.80 (*m*)	23.0 (*t*)	1.82 – 1.88 (*m*)	23.2 (*t*)
11β	1.82 (overlap)		1.64 (overlap)		1.68 (overlap)	
12a	3.61 (*dd*, J = 11.2, 5.0)	66.0 (*t*)	0.98 (*s*, 3 H)	19.0 (*q*)	1.00 (*s*, 3 H)	19.4 (*q*)
12b	3.49 (*dd*, J = 11.2, 5.0)					
13	1.07 (*s*, 3 H)	23.7 (*q*)	0.97 (*s*, 3 H)	30.0 (*q*)	0.91 (*s*, 3 H)	28.4 (*q*)
14	0.88 (*s*, 3 H)	33.8 (*q*)	0.94 (*s*, 3 H)	18.9 (*q*)	1.04 (*s*, 3 H)	20.2 (*q*)
15	1.19 (*s*, 3 H)	25.4 (*q*)	1.21 (*s*, 3 H)	25.0 (*q*)	1.27 (*s*, 3 H)	25.6 (*q*)
16						170.4 (*s*)
17					2.07 (*s*, 3 H)	21.1 (*q*)
5-OH			3.40 (*d*, J = 4.2)			
9-OH	2.80 (*s*)		2.90 (*s*)			
12-OH	3.42 (*t*, J = 5.0)					

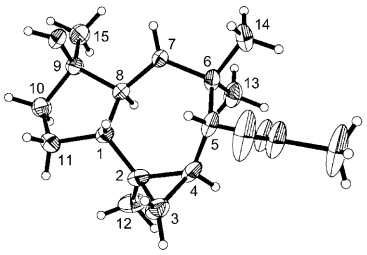

Fig. 2. *Crystallographic structure of* **5a**

by comparison with **5a** and was further supported by the observation of a ROESY correlation between H–C(1) and H–C(14), which showed that they are oriented on the same side of the molecule, and between H–C(8) and H–C(4), and H–C(12) and H–C(15), which are oriented on the other side.

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Experimental Part

General. Column chromatography (CC): silica gel (SiO_2; 200–300 and 80–100 mesh; *Qingdao Marine Chemical Factory*, Qingdao, China), SiO_2 *GF*$_{254}$ (*Merck*), *RP-18* gel (*Merck*), or *Sephadex LH-20* gel (*Amersham Biosciences*). TLC: precoated silica gel *GF*$_{254}$ plates (0.20–0.25 mm, *Qingdao*). Optical rotations: *AUTOPOL®IV* automatic polarimeter. UV Spectra: *Genesys*™ *2 Thermospectronic*, λ_{max} (ε); in nm. IR Spectra: *Thermo Nicolet 380* FT-IR spectrophotometer, with KBr cells; in cm^{-1}. NMR Spectra: *Bruker ARX 600* spectrometer operating, at 600/150 MHz, δ in ppm rel. to Me_4Si; *J* in Hz. HR-Q-TOF-MS: *BioTOF*™-*Q* mass spectrometer (*Bruker*); in *m*/*z*. X-Ray single diffraction: *Oxford Gemini S Ultra* single crystal diffraction, graphite monochromator.

Fermentation and Isolation. Clavaria pyxidata was cultured in dishes laid with *ca.* 20 ml PDA medium with the total volume of 10 l for 25 d at 28°. The mycelium together with culture medium was firstly extracted with 80% AcOEt, 15% MeOH, and 5% AcOH. The crude extract was partitioned again with AcOEt and doubly-distilled H_2O. The AcOEt-soluble fraction was dried over Na_2SO_4 (anh.) and the solvent evaporated under reduced pressure to afford 15.0 g of a crude org. extract (dark oil).

Fractionation and Isolation. The crude extract was subjected to MPLC over *RP-18* SiO_2 (170 g) using a stepwise gradient of 10, 30, 50, 70, and 100% (*v*/*v*) MeOH in H_2O and to afford *Fr. 1* (1.1 g) obtained from 20% MeOH and *Fr. 2* (1.2 g) obtained from 50% MeOH. *Fr. 1* was further subjected to *Sephadex LH-20* eluted with MeOH to afford *Fr. 1.1* (80 mg) and *Fr. 1.2* (360 mg). *Fr. 1.1* and *Fr. 1.2* were subjected to SiO_2 chromatography using a $CHCl_3$/MeOH solvent gradient to yield **1** (17 mg), **2** (3.5 mg), and **4** (27 mg), resp. *Fr. 2* was chromatographed on *Sephadex LH-20* and eluted with MeOH, MPLC eluted with MeOH and H_2O and SiO_2 column using PE/acetone to yield **3** (1.8 mg) and **5** (2 mg).

Pyxidatol A (= rel-*(1*R,*2*S,*2a*S,*3*S,*4*R,*4a*S,*7a*R,*7b*R*)-Decahydro-3-(hydroxymethyl)-6,6,7b-trimethyl-2a*H*-cyclobut[*e*]indene-1,2,2a,3,4-pentol*; **1**). Colorless oil. $[\alpha]_D^{20} = -22.3$ ($c = 0.15$, MeOH). IR (film): 3466, 1636. ^{1}H- and ^{13}C-NMR: *Tables 2* and *1*. HR-ESI-Q-TOF MS: 303.2035 ($[M+H]^+$, $C_{15}H_{27}O_6^+$; calc. 303.1808).

Pyxidatol B (= rel-*(1*R,*2*S,*2a*R,*4a*R,*6*S,*7a*R,*7b*S*)-2,2a,4a,5,6,7,7a,7b-Octahydro-3,6-bis(hydroxymethyl)-6,7b-dimethyl-1*H*-cyclobut[*e*]indene-1,2-diol*; **2**). Colorless oil. $[\alpha]_D^{20} = +0.95$ ($c = 0.18$, MeOH). IR (film): 3281, 2922, 1652. ^{1}H- and ^{13}C-NMR: *Tables 2* and *1*. HR-ESI-Q-TOF MS: 291.2231 ($[M+Na]^+$, $C_{15}H_{24}NaO_4^+$; calc. 291.1572).

Pyxidatol C (= rel-*(1a*S,*4a*S,*5*R,*7a*R,*7b*S*)-Decahydro-7b-(hydroxymethyl)-3,3,5-trimethyl-1*H*-cycloprop[*e*]azulen-5-ol*; **3**). Colorless oil. $[\alpha]_D^{20} = +10.0$ ($c = 0.13$, MeOH). IR (film): 3344, 2926, 1593, 1035. ^{1}H- and ^{13}C-NMR: *Table 3*. HR-ESI-Q-TOF MS: 261.2376 ($[M + Na]^+$, $C_{15}H_{26}NaO_2^+$; calc. 261.1830).

Tsugicoline E (= rel-*(1*R,*1a*R,*3a*S,*4*R,*4a*S,*7a*R,*7b*S,*7c*S*)-Octahydro–6,6,7b-trimethyl-2-oxacyclobut[cd]-s-indacene-1,1a,3a,4(1*H*,3*H*)-tetrol*; **4**). White powder. $[\alpha]_D^{20} = -13.3$ ($c = 0.15$, MeOH). IR (KBr): 3354, 2950, 2928, 1744, 1031. ^{1}H- and ^{13}C-NMR: *Tables 2* and *1*. HR-ESI-Q-TOF MS: 307.2049 ($[M + Na]^+$, $C_{15}H_{24}NaO_5^+$; calc. 307.1521).

Crystal Data of **4**. $C_{15}H_{24}O_5$; 284.34; orthorombic, $a = 6.0148(8)$, $b = 9.7935(14)$, $c = 24.286(3)$ Å, 1430.6(3) Å^3, space group $P2_12_12_1$, $Z = 4$, $D_\chi = 1.320$ Mg m^{-3}, $\mu = 0.098$ mm^{-1}, $F(000) = 616$; colorless prismatic crystals, dimension $0.28 \times 0.30 \times 1.0$ mm^3. CCDC-707995 contains the supplementary crystallographic data for this work. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html (or from the *Cambridge Crysatallographic Data Center*, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

Acetylation of Omphadiol (**5**). Omphadiol (**5**) was dissolved in dry pyridine (0.5 ml) and treated with Ac_2O (1 ml) overnight at r.t. The mixture was evaporated and subjected to *Sephadex LH-20* eluted with acetone to afford **5a** (*rel-*(1a*S*,2*R*,4a*S*,5*R*,7a*R*,7b*S*)-decahydro-5-hydroxy-3,3,5,7b-tetramethyl-1*H*-cycloprop[*e*]azulen-2-yl acetate; 1.5 mg). White powder. $[\alpha]_D^{20} = +22.2$ ($c = 0.08$, $CHCl_3$). IR (KBr): 3434, 2957, 2926, 1736, 1244. ^{1}H- and ^{13}C-NMR: *Table 3*. ESI-MS: 303.2 ($[M + Na]^+$).

Crystal Data of **5a**. $C_{17}H_{28}O_3$; 280.39; orthorombic, $a = 7.6506(3)$, $b = 14.2512(6)$, $c = 15.1720(8)$ Å, 1654.21(13) Å^3, space group $P2_12_12_1$, $Z = 4$, $D_\chi = 1.126$ Mg m^{-3}, $\mu = 0.075$ mm^{-1}, $F(000) = 616$; colorless prismatic crystals, dimension $0.19 \times 0.21 \times 1.0$ mm^3. CCDC-685508 contains the supplementary crystallographic data for this work. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/

retrieving.html (or from the *Cambridge Crysatallographic Data Center*, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

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