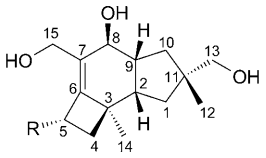
Novel Sesquiterpenes from the Mycelial Cultures of *Dichomitus squalens*

by **Hai-Hui Xie**, **Xin-Ya Xu**, **Yang Dan**, and **Xiao-Yi Wei***

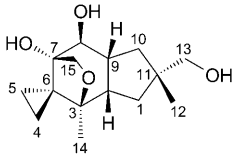
Key Laboratory of Plant Resources Conservation and Sustainable Utilization, South China Botanical Garden, Chinese Academy of Sciences, Guangzhou 510650, P. R. China
(phone: +86-20-37252538; fax: +86-20-37252537; e-mail: wxy@scbg.ac.cn)

Four novel sesquiterpenes, 5-hydroxydichomitol (**1**), dichomilludol (**2**), 3*β*,13-dihydroxyledol (**3**), and 2*β*,3*β*,13-trihydroxyledol (**4**), were isolated along with 2*β*,13-dihydroxyledol (**5**) from the AcOEt-soluble fraction of the mycelial solid cultures of *Dichomitus squalens*. Their structures were determined on the basis of spectroscopic analyses. The previously assigned structure of dichomitol was revised.

Introduction. – *Dichomitus squalens* is a commonly found white-rot Basidiomycetous fungus with the capability to degrade both natural and synthetic lignins and lignin derivatives [1][2]. In search for bioactive natural compounds from Basidiomycetes growing in South China, the EtOH extract of the mycelial cultures of this fungus was found to be nematicidal against a catastrophic pine wilt nematode (*Bursaphelenchus xylophilus*). Subsequent screening of the petroleum ether-, $CHCl_3$-, and AcOEt-soluble fractions from this extract showed that the $CHCl_3$-soluble fraction was more potent than the others. Our previous investigation on the $CHCl_3$-soluble fraction led to the isolation of three new sesquiterpenes, dichomitol, 2*β*,13-dihydroxyledol, and dichomitone, of which the second one was found to be nematicidal [3]. In continuation of our studies on this fungus, the AcOEt-soluble fraction was investigated, and four new sesquiterpenes, 5-hydroxydichomitol (**1**), dichomilludol (**2**), 3*β*,13-dihydroxyledol (**3**), and 2*β*,3*β*,13-trihydroxyledol (**4**), were isolated along with a previously isolated sesquiterpene, 2*β*,13-dihydroxyledol (**5**). Furthermore, the previously assigned structure of dichomitol was revised. Here, we describe the isolation and structure elucidation of these compounds and the structure revision of dichomitol.



1 R = OH
6 R = H



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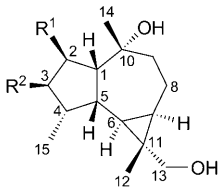

	R^1	R^2
3	H	OH
4	OH	OH
5	OH	H

Results and Discussion. – The AcOEt-soluble fraction, previously obtained from the EtOH extract of the mycelial solid cultures of *D. squalens* by liquid-liquid partition [3], was separated by silica-gel and *Develosil ODS* column chromatography and preparative HPLC to give compounds **1**–**5** (see *Exper. Part*).

Compound **1** was isolated as colorless oil. It had a molecular formula of $C_{15}H_{24}O_4$ as determined by its HR-ESI-MS and NMR data. In the ^{1}H- and ^{13}C-NMR spectra (*Table 1*), signals for 20 H-atoms and 15 C-atoms were observed. With the aid of ^{13}C,^{1}H-COSY spectrum, these signals were assigned to two tertiary Me groups (δ(H) 1.19 (*s*, Me(12)) and 1.48 (*s*, Me(14)), and δ(C) 23.3 (C(12)) and 22.1 (C(14))), five CH_2 groups (two O-bearing; δ(H) 3.74 (*s*, CH_2(13)), 4.92 and 4.86 (each *d*, $J = 13.0$, CH_2(15)), and δ(C) 71.4 (C(13)) and 59.5 (C(15))), four CH groups (two O-bearing; (δ(H) 5.37 (br. *d*, $J = 6.4$, H–C(5)) and 4.68 (*d*, $J = 8.8$, H–C(8)), and δ(C) 68.3 (C(5)) and 74.0 (C(8))), and four quaternary C-atoms including two olefinic C-atoms (δ(C) 146.9 (C(6)) and 137.2 (C(7))). Analysis of the ^{1}H,^{1}H-COSY spectrum showed connectivities of H–C(1) with H–C(2), H–C(2) with H–C(9), H–C(9) with both H–C(8) and CH_2(10), H–C(8) with H–C(5) *via* a C=C bond, and H–C(5) with CH_2(4). In the HMBC spectrum, long-range correlations were observed from both Me(12) and CH_2(13) to C(1) and C(10), from CH_2(13) to C(12) and C(11), and from Me(12) to CH_2(13), indicating that C(12) and C(13) were at C(11), which was further connected to both C(1) and C(10). Further HMBCs from Me(14) to C(2), C(3), C(4), and C(6), from CH_2(4) to C(2), C(3), and C(6), and from CH_2(15) to C(6), C(7), and C(8) were also observed, revealing the attachments of C(15) to C(7), C(14) to C(3), and of C(3) to C(2), C(4), and C(6). These findings indicated a planar structure of protoillud-6-ene-5,8,13,15-tetrol [4]. In the NOESY spectrum, mutual correlations H–C(2)/Me(12), H–C(9)/Me(12), H–C(9)/H_β–C(1), H–C(8)/Me(14), H–C(8)/H_α–C(10), and Me(14)/CH_2(13) were observed, while the correlation H–C(5)/Me(14) was not observed, indicating that H–C(2), H–C(5), H–C(9), HO–C(8), and Me(12) were at the same side and β-oriented, while Me(14), HO–C(5), and CH_2(13) were all in α-orientation. Thus, compound **1** was determined as protoillud-6-ene-5α,8β,13,15-tetrol.

A total synthesis of the previously reported dichomitol [3] had been accomplished by *Mehta* and *Pallavi* [5]. It became clear that the compound isolated in our studies was different from the authentic synthetic product. The actual structure of dichomitol had not been assigned until this new protoilludene **1** was isolated and characterized. Then, dichomitol was found to be closely related to this new compound by comparison of its ^{1}H- and ^{13}C-NMR spectra with those of **1**, except that in the 5-position there was a CH_2 group (δ(H) 2.74–2.81 and 2.61–2.66 (each 1 H, m), and δ(C) 25.1) instead of an O-bearing CH group (δ(H) 5.37 (br *d*, $J = 6.4$) and δ(C) 68.3) in **1**. This, in combination with its molecular formula, indicated a structure of protoillud-6-ene-8β,13,15-triol (**6**), a new protoilludene sesquiterpene. Re-examination of the ^{1}H,^{1}H-COSY, ^{13}C,^{1}H-COSY, HMBC, and NOESY spectra led to the re-assignment of the ^{1}H- and ^{13}C-NMR data as compiled in *Table 1* and supported the structure. Thus, the structure of dichomitol (**6**) was corrected to be as depicted. The error had resulted from misinterpretation of the HMBCs, *i.e.*, a ^4J correlation from CH_2(4) at δ(H) 1.81–1.89 to C(7) at δ(C) 145.8 had been misinterpreted as ^3J correlations, and a ^3J correlation from CH_2(15) to C(8) at δ(C) 74.3 had been erroneously explained by a

Table 1. *^{1}H- and ^{13}C-NMR Data* (at 400 and 100 MHz, resp.) *of* **1**, **2**, *and* **6**[a]). δ in ppm, J in Hz.

Position	**1**		**2**		**6**	
	δ(H)	δ(C)	δ(H)	δ(C)	δ(H)	δ(C)
H_α–C(1)	1.84 (*dd*, J = 12.4, 10.6)	36.3	2.09 (*dd*, J = 13.6, 12.0)	39.2	1.44 (*dd*, J = 12.0, 11.0)	36.0
H_β–C(1)	1.49 (*ddd*, J = 12.4, 8.4, 1.6)		1.32 (*dd*, J = 12.0, 6.4)		1.35 (*ddd*, J = 12.0, 8.0, 1.5)	
H–C(2)	2.48 (*ddd*, J = 11.8, 10.5, 8.4)	46.6	2.64 (*ddd*, J = 13.6, 8.9, 6.4)	54.5	2.40 (*td*, J = 11.0, 8.0)	45.5
C(3)		44.0		82.8		45.9
H_α–C(4)	2.14 (*dd*, J = 11.9, 2.1)	47.3	0.54 (*ddd*, J = 10.1, 4.2, 2.5)	0.1	1.81 – 1.89 (*m*)	36.1
H_β–C(4)	2.28 (*dd*, J = 11.9, 6.4)		1.03 – 1.08 (*m*)		1.81 – 1.89 (*m*)	
H_α–C(5)		68.3	0.71 (*ddd*, J = 9.4, 4.3, 3.4)	4.5	2.74 – 2.81 (*m*)	25.1
H_β–C(5)	5.37 (br. *d*, J = 6.4)		1.03 – 1.08 (*m*)		2.61 – 2.66 (*m*)	
C(6)		146.9		31.9		145.8
C(7)		137.2		78.3		129.1
H–C(8)	4.68 (*d*, J = 8.8)	74.0	4.18 (*d*, J = 8.9)	78.1	4.13 (br. *d*, J = 7.5)	74.3
H–C(9)	2.80 – 2.89 (*m*)	52.7	2.79 (*dq*, J = 8.9, 2.5)	45.1	2.29 – 2.36 (*m*)	50.5
H_α–C(10)	1.77 (*t*, J = 11.8)	42.6	2.39 (*dd*, J = 13.8, 2.5)	40.9	1.27 (*t*, J = 11.0)	40.8
H_β–C(11)	2.15 (*ddd*, J = 11.8, 6.5, 1.8)		1.93 (*dd*, J = 13.8, 8.9)		1.73 (br. *dd*, J = 11.0, 7.5)	
C(11)		46.1		45.5		45.1
Me(12)	1.19 (*s*)	23.3	1.16 (*s*)	26.5	0.99 (*s*)	22.7
CH_2(13)	3.74 (*s*)	71.4	3.69 (*s*)	71.7	3.48 (*d*, J = 11.0), 3.46 (*d*, J = 11.0)	72.1
Me(14)	1.48 (*s*)	22.1	1.11 (*s*)	20.7	1.07 (*s*)	20.3
CH_2(15)	4.92 (*d*, J = 13.0), 4.86 (*d*, J = 13.0)	59.5	3.94 (*dd*, J = 7.2, 1.4), 4.59 (br. *d*, J = 7.2)	70.5	4.22 (br. *d*, J = 12.5), 4.20 (br. *d*, J = 12.5)	59.0

[a]) Solvents: C_5D_5N for **1** and **2**, $CDCl_3$ for **6**.

^4J correlation. The author, *X. Wei*, apologizes for any inconveniences caused by this error.

Compound **2** was also isolated as colorless oil, and its molecular formula was deduced as $C_{15}H_{24}O_4$ from its HR-ESI-MS and NMR data. The ^{1}H- and ^{13}C-NMR spectra (*Table 1*), in combination with the ^{13}C,^{1}H-COSY experiment, indicated the presence of two tertiary Me groups (δ(H) 1.16 (*s*, Me(12)) and 1.11 (*s*, Me(14)), and δ(C) 26.5 (C(12)) and 20.7 (C(14))), six CH_2 groups, of which two displayed extremely upfield H-atom signals (δ(H) 0.54 (H_α–C(4)), 1.03 – 1.08 (H_β–C(4)), 0.71 (H_α–C(5)), and 1.03 – 1.08 (H_β–C(5)), and C-atom signals at δ(C) 0.1 (C(4)) and 4.5 (C(5)), and two were O-bearing at δ(H) 3.69 (CH_2(13)), 3.94 and 4.59 (CH_2(15), each 1 H), and δ(C) 71.7 (C(13)) and 70.5 (C(15))), three CH groups including an O-bearing CH (δ(H) 4.18 (H–C(8)) and δ(C) 78.1 (C(8))), and four quaternary C-atoms including two O-bearing ones ((δ(C) 82.8 (C(3)) and 78.3 (C(7))). Interpretation of the ^{1}H,^{1}H-COSY spectrum revealed partial structures as shown by bold lines in the *Figure*. Further connectivities were deduced from the HMBC spectrum (*Fig.*), in which correlations from both CH_2(13) and Me(12) to C(1), C(11), and C(10) revealed the connectivity of C(11) to both C(1) and C(10), with C(12) and C(13) attached to C(11). Further, correlations from both CH_2(4) and CH_2(5) to C(3), C(6), and C(7), from Me(14) to C(2), C(3), and C(6), and from CH_2(15) to C(6), C(7), and C(8) indicated a cyclopropane ring composed of C(4), C(5), and C(6), and a six-membered ring formed by C(2), C(3), C(6), C(7), C(8), and C(9), with C(14) attached to C(3), and C(15) attached to C(7) [6]. In addition, a correlation from CH_2(15) to C(3) was also observed, revealing the connectivity between C(15) and C(3) *via* an O-bridge. Thus, a 3,15-*O*-bridged illudane structure was assigned as shown. In the NOESY spectrum, correlations were observed between H–C(2) and Me(12), H–C(9) and Me(12), Me(12) and H_β–C(10), H–C(8) and H_α–C(10), CH_2(13) and H_α–C(1), CH_2(13) and H_α–C(10), and H–C(9) and H_b–C(15), indicating that H–C(2), H–C(9), Me(12), HO–C(8), and 3,15-*O*-bridge were at the same side and β-oriented, while H–C(8), CH_2(13), Me(14), and HO–C(7) were at the opposite side and in α-orientation. The presence of NOE correlations H_α–C(5)/H_α–C(1), H–C(8)/H_α–C(1), and H–C(8)/CH_2(13) indicated a chair ($^6C_9$) form of the six-membered ring. Therefore, the structure of **2** was determined as shown and trivially named dichomilludol.

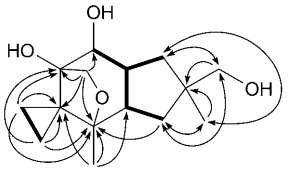

Figure. *^{1}H,^{1}H-COSY* (bold lines) *and key HMBC* (arrows) *correlations of* **2**

Compound **3**, obtained as colorless oil, had a molecular formula of $C_{15}H_{26}O_3$ as deduced from its HR-ESI-MS and NMR data. The ^{1}H- and ^{13}C-NMR (*Table 2*), and DEPT spectra revealed the presence of two tertiary Me groups, a secondary Me group, four CH_2 groups, six CH groups, and two quaternary C-atoms, of which a CH_2 group, a CH group, and a quaternary C-atom were O-bearing. Combined analysis of the ^{1}H,^{1}H-

COSY, ^{13}C,^{1}H-COSY, and HMBC spectra led to full assignment of the ^{1}H- and ^{13}C-NMR data as compiled in *Table 2*, indicating a structure closely similar to 2β,13-dihydroxyledol (**5**) [3], a known aromadendrane sesquiterpene occurring in this fungus and re-isolated in the present study, except that HO at C(2) in **5** migrated to C(3) in **3**. The β-orientation of HO–C(3) was deduced from the NOE correlations of H–C(3) with both H–C(6) and Me(15) observed in the NOESY spectrum. Thus, compound **3** was determined as 3β,13-dihydroxyledol.

Table 2. *^{1}H- and ^{13}C-NMR Data* (400 and 100 MHz, resp.; C_5D_5N) *of* **3** and **4**. δ in ppm, J in Hz.

	3		**4**	
	δ(H)	δ(C)	δ(H)	δ(C)
H–C(1)	2.72 (*ddd*, *J* = 13.4, 7.2, 4.4)	56.1	2.53 (*dd*, *J* = 9.2, 4.8)	63.2
H_α–C(2)	2.25–2.34 (*m*)	37.9	4.49 (*dd*, *J* = 9.2, 8.0)	72.0
H_β–C(2)	2.00–2.05 (*m*)			
H–C(3)	4.35 (br. *t*, *J* = 7.2)	77.7	4.17 (*t*, *J* = 8.0)	76.5
H–C(4)	2.25–2.34 (*m*)	50.0	2.19–2.28 (*m*)	46.0
H–C(5)	2.45 (*dt*, *J* = 9.6, 4.4)	40.4	2.39 (*dt*, *J* = 10.0, 4.8)	35.9
H–C(6)	0.53 (*t*, *J* = 9.6)	20.8	0.51 (*t*, *J* = 10.0)	21.9
H–C(7)	1.09 (*ddd*, *J* = 11.4, 9.6, 6.1)	26.2	1.02–1.09 (*m*)	25.7
H_α–C(8)	1.73–1.79 (*m*)	19.1	1.73–1.80 (*m*)	19.2
H_β–C(8)	2.00–2.05 (*m*)		2.00–2.05 (*m*)	
H_α–C(9)	1.80–1.83 (*m*)	38.3	2.00–2.05 (*m*)	40.1
H_β–C(9)	1.80–1.83 (*m*)		1.95–2.03 (*m*)	
C(10)		72.9		73.3
C(11)		26.2		26.7
Me(12)	1.36 (*s*)	12.5	1.34 (*s*)	12.2
CH_2(13)	3.55 (*d*, *J* = 10.4), 3.85 (*d*, *J* = 10.4)	72.0	3.52 (*d*, *J* = 10.8), 3.81 (*d*, *J* = 10.8)	71.9
Me(14)	1.36 (*s*)	32.7	1.84 (*s*)	33.0
Me(15)	1.33 (*d*, *J* = 6.8)	14.8	1.32 (*d*, *J* = 7.2)	14.9

Compound **4**, also obtained as colorless oil, had a molecular formula of $C_{15}H_{26}O_4$, containing one O-atom more than compound **3**. The ^{1}H- and ^{13}C-NMR spectra (*Table 2*) were similar to those of **3** except that the signals for one of the CH_2 groups were absent. Instead, the H- and C-atom resonances for an additional O-bearing CH group at δ(H) 4.49 (H–C(2)) and δ(C) 72.0 (C(2)) were observed. Analysis of the ^{1}H,^{1}H-COSY, ^{13}C,^{1}H-COSY, and HMBC spectra revealed a planar structure of aromadendrane-2,3,10,13-tetrol. The presence of NOE correlations H–C(1)/H–C(4), H–C(1)/Me(14), H–C(2)/H–C(6), H–C(3)/H–C(6), H–C(3)/Me(15), H–C(5)/Me(12), and H–C(6)/CH_2(13) in the NOESY spectrum, as well as the coupling constants, J(1,5) = 4.8, J(1,2) = 9.2, J(2,3) = 8.0, and J(5,6) = 10.0 Hz in the ^{1}H-NMR spectrum (*Table 2*) indicated that **4** had the same relative configuration as that of ledol [7], and both HO–C(2) and HO–C(3) were β-oriented [8]. Consequently, compound **4** was established as 2β,3β,13-trihydroxyledol.

Compounds **1**–**4** were evaluated for the cytotoxicity and nematicidal activity according to the previously described methods [3][9]. However, they were all found to be relatively non-cytotoxic ($IC_{50} > 35$ μM) against the test cancer cell lines, including human breast cancer (MCF-7 and MDA-MB-231/ATCC), leukemia (K-562), and non-

small lung cancer (NCI-H460) cells, and inactive against the test nematode *Bursaphelenchus xylophilus* ($LC_{50} > 1000$ μg/ml).

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Experimental Part

General. Column chromatography (CC) and TLC: silica gel *60* (SiO_2; 100–200 mesh; *Qingdao Haiyang Chemical Co., Ltd.*, Qingdao, P. R. China), *Develosil ODS* (*S-75* μm; *Nomura Chemical Co., Ltd.*, Seto, Japan), silica gel $HSGF_{254}$ plates (*Yantai Jiangyou Silica Gel Development Co., Ltd.*, Yantai, P. R. China), and *RP-18* F_{254S} plates (*Merck Japan Ltd.*, Tokyo, Japan). Prep. HPLC: *Shimadzu LC-6A* pump and *Shimadzu RID-10A* refractive index detector; *YMC-Pack ODS-A* column (*S-5* μm, 12 nm; 250 mm × 20 mm i.d.). Optical rotations: *Perkin-Elmer 341* polarimeter. 1D- and 2D-NMR spectra: *Bruker DRX-400* instrument; the residual peaks of C_5D_5N at δ(H) 8.71 and δ(C) 149.9 ppm as internal references. ESI-MS: *MDS SCIEX API 2000* LC/MS/MS apparatus. HR-ESI-MS: *API QSTAR* TOF IIIQ mass spectrometer.

Producing Fungus and Fermentation. Fruiting bodies of *D. squalens* were collected in Dinghu Mountain, Guangdong, P. R. China, and authenticated by Prof. *T. Li*, Guangdong Institute of Microbiology, Guangdong, P. R. China. Mycelia were isolated from tissue plugs of a young fruiting body. The mycelia grown on MEA (membrane electrode assembly) plates were used to inoculate ten 500-ml *Erlenmeyer* flasks containing 100 ml of YMG medium (glucose 0.4%, malt extract 1.0%, yeast extract 0.4%, and pH 5.5). The flasks were incubated on a rotary shaker for 5 d at 28° with shaking at 120 rpm. The cultures were transferred into ten 5,000 ml flasks containing 1,000 ml of YMG medium and 500 g of wheat grains, and the cultivation was carried out in the stationary phase at 28° for 13 d [3].

Extraction and Isolation. The AcOEt-soluble fraction (15.65 g), previously obtained from the EtOH extract of the mycelial cultures of *D. squalens* [3], was subjected to CC (SiO_2 (313 g); $CHCl_3$/MeOH 9:1 → 6:4): *Frs. 1–9*. *Fr. 4* (0.37 g) was further subjected to CC (*Develosil ODS* (11 g); MeOH/H_2O 10:90 → 60:40): *Frs. 4-1–4-6*. *Fr. 4-3* (17.9 mg) was purified by HPLC (45% MeOH/H_2O; 5 ml/min): **3** (t_R 22.1 min; 3.7 mg) and **5** (t_R 25.0 min; 11.0 mg). *Fr. 5* (1.29 g) was further subjected to CC (*Develosil ODS* (39 g), MeOH/H_2O 10:90 → 60:40): *Frs. 5-1–5-9*. *Fr. 5-5* (94 mg) was purified by HPLC (20% MeOH/H_2O; 5 ml/min): **4** (t_R 21.1 min; 14.9 mg). *Fr. 5-6* (87 mg) was purified by HPLC (25% MeOH/H_2O; 5 ml/min): **2** (t_R 47.4 min; 5.8 mg). *Fr. 7* (0.97 g) was further subjected to CC (*Develosil ODS* (29 g); MeOH/H_2O 10:90 → 55:45): *Frs. 7-1–7-4*. *Fr. 7-3* (125 mg) was purified by HPLC (40% MeOH/H_2O; 5 ml/min): **1** (t_R 29.0 min; 10.2 mg).

5-Hydroxydichomitol (= *(2*S*,4*S*,4a*R*,6*R*,7a*S*,7b*R*)-2,4,4a,5,6,7,7a,7b-Octahydro-3,6-bis(hydroxymethyl)-6,7b-dimethyl-1*H*-cyclobuta[*e*]indene-2,4-diol*; **1**). Colorless oil. $[\alpha]_D^{20} = -42.5$ ($c = 1.00$, MeOH). ^{1}H- (400 MHz) and ^{13}C-NMR (100 MHz): see *Table 1*. ESI-MS: 291 ($[M+Na]^+$), 303 ($[M+Cl]^-$). HR-ESI-MS (pos.): 291.1558 ($C_{15}H_{24}NaO_4^+$; calc. 291.1572).

Dichomilludol (= *(5′*S*,5a′*R*,7′*S*,8a′*S*)-Hexahydro-7′-(hydroxymethyl)-1′,7′-dimethyl-1′*H*-spiro[cyclopropane-1,9′-[2]oxa[1,4]methanocyclopenta[*c*]oxepine]-4′,5′(3′*H*)-diol*; **2**). Colorless oil. $[\alpha]_D^{20} = +10.2$ ($c = 0.58$, MeOH). ^{1}H- (400 MHz) and ^{13}C-NMR (100 MHz): see *Table 1*. ESI-MS: 291 ($[M+Na]^+$), 267 ($[M-H]^-$), 303 ($[M+Cl]^-$). HR-ESI-MS (pos.): 291.1559 ($C_{15}H_{24}NaO_4^+$; calc. 291.1572).

3β,13-Dihydroxyledol (= *(1*S*,1a*R*,4*R*,4a*S*,6*S*,7*S*,7a*R*,7b*S*)-Decahydro-1-(hydroxymethyl)-1,4,7-trimethyl-1*H*-cyclopropa[*e*]azulene-4,6-diol*; **3**). Colorless oil. $[\alpha]_D^{20} = -44.3$ ($c = 0.37$, MeOH). ^{1}H- (400 MHz) and ^{13}C-NMR (100 MHz): see *Table 2*. ESI-MS: 277 ($[M+Na]^+$), 253 ($[M-H]^-$). HR-ESI-MS (neg.): 253.1812 ($C_{15}H_{25}O_3^-$; calc. 253.1804).

2β,3β,13-Trihydroxyledol (= *(1*S*,1a*R*,4*R*,4a*R*,5*S*,6*R*,7*S*,7a*R*,7b*S*)-Decahydro-1-(hydroxymethyl)-1,4,7-trimethyl-1*H*-cyclopropa[*e*]azulene-4,5,6-triol*; **4**). Colorless oil. $[\alpha]_D^{20} = -16.4$ ($c = 1.00$, MeOH).

^{1}H- (400 MHz) and ^{13}C-NMR (100 MHz): see *Table 2*. ESI-MS: 293 ($[M+Na]^+$), 305 ($[M+Cl]^-$). HR-ESI-MS (pos.): 293.1725 ($C_{15}H_{26}NaO_4^+$; calc. 293.1729).

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