Five New Cadinane-Type Sesquiterpenes from the Heartwood of *Chamaecyparis obtusa* var. *formosana*

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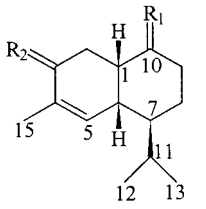
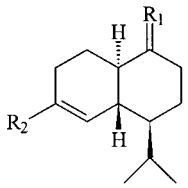
Muurola-4,10(14)-dien-3-one (**1**), 10-*O*-acetyl-15-oxo-α-cadinol (**2**), 15-hydroxy-α-cadinol (**3**), 4α-hydroxy-5*β*-ethoxy-*epi*-cubenol (**4**), and 4α-hydroxy-5*β*-acetoxy-*epi*-cubenol (**5**), five new bicyclic sesquiterpenes, have been isolated from the heartwood of *Chamaecyparis obtusa* var. *formosana* along with the known compounds *epi*-cubenol, T-cadinol, T-muurolol, *δ*-cadinol, and α-cadinol. The structures of the new constituents were elucidated through chemical and spectral studies.

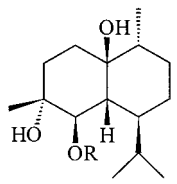
There are seven species of the genus *Chamaecyparis* (Cupressaceae), but only two endemic species, *C. formosensis* and *C. obtusa* var. *formosana*, are found in the central mountains of Taiwan. Both of these species are important building materials, and the latter species is believed to be more resistant to wood-decaying fungi. Although there are several earlier studies on the chemical constituents of *C. obtusa* var. *formosana*,[1−4] only essential oils and simple acidic components were isolated. We also recently reported the isolation and identification of the novel diterpene obtunone[5] and three new abietane diterpenes[6] from the heartwood of the plant. In this paper, we report five new bicyclic cadinane sesquiterpenes, muurola-4,10(14)-dien-3-one (**1**), 10-*O*-acetyl-15-oxo-α-cadinol (**2**), 15-hydroxy-α-cadinol (**3**), 4α-hydroxy-5*β*-ethoxy-*epi*-cubenol (**4**), and 4α-hydroxy-5*β*-acetoxy-*epi*-cubenol (**5**), together with five known sesquiterpenes, *epi*-cubenol (**6**),[7] T-cadinol (**7**),[8] T-muurolol (**8**),[8] *δ*-cadinol (**9**),[9] and α-cadinol (**10**).[10] Compound **3** was purified as its monoacetate **11**.



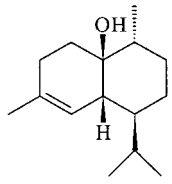


1 R_1=CH_2, R_2=O
8 R_1=β-OH, α-Me, R_2=H_2
9 R_1=β-Me, α-OH, R_2=H_2

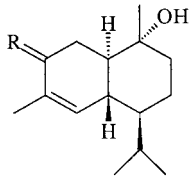
2 R_1=β-Me, α-OAc, R_2=CHO
3 R_1=β-Me, α-OH, R_2=CH_2OH
7 R_1=β-OH, α-Me, R_2=Me
10 R_1=β-Me, α-OH, R_2=Me
11 R_1=β-Me, α-OH, R_2=CH_2OAc
12 R_1=β-Me, α-OAc, R_2=Me
13 R_1=β-Me, α-OH, R_2=CHO



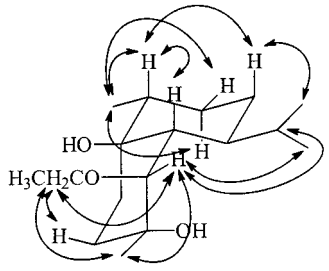
4 R=Et
5 R=Ac



6



14 R=O
15 R=α-OH, β-H



16 Key NOESY ⟷

Results and Discussion

HREIMS revealed compound **1** to be a sesquiterpene of formula $C_{15}H_{22}O$ [[M^+] = 218.1675]. The IR spectrum suggested the presence of an α,*β*-unsaturated carbonyl system with absorptions at ν_{max} 1672, 1663 cm^{-1} and an exocyclic double bond with absorption at 889 cm^{-1}. The ^{13}C NMR spectrum of **1** (Table 1) shows 15 carbon signals for a carbonyl, two double bonds, three methylenes, four methines, and three methyl groups. Five indices of hydrogen deficiency (IHD) were determined from the ^{13}C NMR spectrum and DEPT experiments. Four olefinic signals (δ_C 109.0, 134.9, 149.2, and 149.7) and the conjugated carbonyl at δ_C 199.5 implicated a bicyclic molecule. The one-proton doublet at δ 6.78 (J = 5.0 Hz, H-5) could be assigned to a vinylic hydrogen *β* to the carbonyl. A broad signal at δ 1.77 (3H) proved to be due to a methyl group on a double bond. The signal at δ_C 109.0 is typical of the terminal methylene carbon of an exocyclic double bond, while the singlet at δ 4.67 (2H) could be assigned to the terminal olefinic protons. The signals at δ 0.86 and 0.93 (each 3H, d, J = 6.9 Hz, H-12, -13) and 1.89 (1H, m, H-11; COSY correlation with δ 0.86 and 0.93), together with the MS spectral fragment at m/z at 175 [$M^+ - C_3H_7$]$^+$, established the presence of an isopropyl group. On the basis of the above evidence, **1** had the skeleton of the cadinane type of sesquiterpene.[11] The $^1H-^1H$ COSY spectrum of **1** showed connectivities for the H_2-protons [δ 2.25 (1H, dd, J = 16.9, 4.3 Hz) and 2.74

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Table 1. ^{1}H and ^{13}C NMR Spectral Data of **1**−**5** and **11** (400 and 100 MHz in $CDCl_3$)

	1	**2**	**3**	**11**	**4**		**5**	
no.	δ_C	δ_C	δ_C	δ_C	δ_H	δ_C	δ_H	δ_C
1	43.0	47.3	50.0	49.7		74.7		72.2
2	40.1	21.5	22.2	22.1	1.41m 1.64m[a]	32.1	1.51m 1.86m[b]	31.8
3	199.5	22.0	26.4	26.7	1.57m 1.72m	25.0	1.24m 1.85m	29.6
4	134.9	141.7	138.2	133.7		75.6		70.6
5	149.7	151.2	123.5	127.4	3.55br s	72.3	5.05br s	75.5
6	40.7	40.6	39.6	39.8	1.65[a]	42.5	1.83[b]	42.9
7	43.8	45.3	46.4	46.2	1.72m	37.4	1.46m	37.2
8	25.4	21.6	21.9	22.0	1.06m 1.67m[a]	23.7	1.05qd (12.8, 6.8) 1.65qd (12.8, 4.2)	23.7
9	30.5	36.2	42.0	42.1	1.32m 1.49m	30.4	1.42m 1.47m	29.8
10	149.2	84.2	72.3	72.3	1.32m	41.1	1.26m	41.5
11	27.5	26.2	25.9	26.0	2.05sep d (6.9, 3.0)	25.6	1.82m[b]	25.5
12	16.3	15.2	15.1	15.1	0.71d(6.9)	14.9	0.72d(6.9)	14.8
13	21.4	21.3	21.4	21.4	0.91d(6.9)	21.5	0.84d(6.9)	21.4
14	109.0	17.4	20.6	20.7	0.83d(6.3)	14.1	0.87d(6.7)	14.6
15	16.0	194.4	67.2	68.8	1.20s	21.7	1.16s	27.8
$OCOCH_3$		22.6		21.0			2.08s	21.1
$OCOCH_3$		170.5		171.0				169.2
OCH_2CH_3					1.11t(6.9)	16.2		
OCH_2CH_3					3.32dq (14.0, 6.9) 3.37dq (14.0, 6.9)	55.9		

[a,b] Overlapping each other.

(1H, dd, J = 16.9, 12.8 Hz)] with the bridgehead proton H-1 (δ 2.92, dt, J = 12.8, 4.3 Hz), which in turn was connected to the other bridgehead proton H-6 (δ 2.34, m). H-6 was coupled to the methine proton at δ 1.59 (m, H-7), and the coupling chain continued from H-7 to H-8 (δ 1.68−1.73, m) and then to H-9 (δ 2.15−2.23, m). H-11 (δ 1.89, m) showed a COSY correlation with H-7, H-12, and H-13 and also a NOESY correlation with H-5. This evidence confirmed the location of the isopropyl group at C-7. The relative stereochemistry of **1** was deduced from analyses of coupling constants and the NOESY correlation between H-1 and H-6. In the ^{1}H NMR spectrum of **1**, H-1 appears as a doublet of triplets, with the larger doublet coupling (J = 12.8 Hz) to H_{ax}-2 (δ 2.74) and smaller triplet couplings (J = 4.3 Hz) to H_{eq}-2 (δ 2.25) and H-6 (δ 2.34). This evidence established compound **1** as a *cis*-fused ring system. Additionally, H-5 appears as a doublet coupled with H-6 (J = 5.0 Hz), providing further evidence for the *cis*-fusion: in *trans*-fused isomers, such as T-cadinol (**7**), α-cadinol (**10**), and 4-cadinene-9α-ol-3-one,[12] the olefinic proton appears as a broad singlet, while in *cis*-fused muurolene derivatives[8,13] the olefinic proton resonates as a doublet. Thus, based on the above evidence, the structure of **1** was established as muurola-4,10(14)-dien-3-one.

Compound **2** was isolated as an oil and showed a pseudomolecular ion at m/z 218 [M^+ − AcOH], analyzing for $C_{17}H_{26}O_3$. The IR spectrum of **2** showed bonds attributable to an acetoxyl group (1734, 1245, and 1160 cm^{-1}), geminal dimethyl group (1380 and 1369 cm^{-1}), and a conjugated aldehyde (2723, 1690, and 1642 cm^{-1}). The latter group was further confirmed from the UV absorption at λ_{max} 232 nm. The ^{1}H NMR spectrum shows signals for an isopropyl group at δ 0.82, 0.96 (each 3H, d, J = 6.9 Hz), and 2.20 (1H, m, H-11), a three-proton singlet at δ 1.41 for a methyl attached to a quaternary carbon bearing an acetoxyl group (δ 1.97, s), a conjugated aldehyde signal at δ 9.43 (s), and a singlet at δ 6.84 for the olefinic proton β to the carbonyl group (H-5). The COSY spectrum of **2** displayed the connectivity of H-5 to H-6 (δ 2.07, m), which was also coupled to H-1 (δ 1.72, m) and H-7 (δ 1.23, m). The location of the isopropyl at C-7 was confirmed by the NOESY correlations observed between H-5 and the two methyls of the isopropyl group. The protons at δ 2.05 (m) and 2.44 (m) were assigned to the allylic H-3 protons, which were correlated in the COSY spectrum with H-2 [δ 1.64 and 2.06 (each 1H, m)]. H-1 showed a NOESY correlation to H-2α, but no correlation to H-6. The signal for the olefinic proton (H-5) appeared as a broad singlet and showed a NOESY correlation to the methyls of the isopropyl group, thus indicating a *trans*-fused ring and an equatorial isopropyl.[8,13,14] The ^{1}H and ^{13}C NMR data of **2** are very similar to those of α-cadinyl acetate (**12**),[15] except for differences attributable to the aldehyde group in **2**. On the basis of the above evidence, as well as HMBC and NOESY techniques, compound **2** was determined to be 10-*O*-acetyl-15-oxo-α-cadinol. Saponification of **2** yielded alcohol **13** (see below), which is an oxidative product from α-cadinol (**10**). The chemical correlation further confirmed the structure of **2**.

Compound **3** was purified as its monoacetate **11**, obtained by treating **3** with acetic anhydride in pyridine. The molecular formula of **11** was assigned as $C_{17}H_{28}O_3$ based on HREIMS. The IR spectrum of **11** shows absorption bands for a hydroxyl (ν_{max} 3424 cm^{-1}), a trisubstituted double bond (ν_{max} 3041, 1670, 825 cm^{-1}), and an acetoxyl group (ν_{max} 1743 and 1245 cm^{-1}). The ^{1}H NMR spectrum of **11** shows signals for an isopropyl group at δ 0.75, 0.90 (each 3H, d, J = 6.9 Hz) and 2.09 (1H, m, COSY correlation to two doublet methyl groups), a three-proton singlet (δ 1.09) for a methyl group attached to a quaternary carbon bearing a hydroxyl group, an oxymethylene group (δ 4.44, 2H, s) linked between acetoxyl and olefinic groups, an acetoxyl group (δ 2.05, s), and a trisubstituted olefinic proton (δ 5.81, br s). By using COSY, HMQC, and HMBC

spectral methods, all protons, carbons, and carbon−proton connectivities were confirmed. The data were consistent with a 15-acetoxycadinol general structure. The *trans*-fused **11** was revealed from the presence of a broad singlet for the olefinic proton, H-5, and the C-7 equatorial isopropyl was confirmed by observing a NOE correlation between H-5 and an isopropyl methyl group. The strong NOE correlation between H_3-14 and H-6 indicated an axial orientation (β) of the methyl at C-10. By comparison of ^{13}C NMR data of **11** with that of α-cadinol (Table 1), the structure of **11** was determined as 15-acetoxy-α-cadinol. Saponification of compound **11** yielded the purified natural product **3** (see Experimental Section for physical data), which was also chemically correlated to α-cadinol (**10**) and **13**. Oxidation of α-cadinol (**10**) with SeO_2 under reflux in ethanol solution gave four products, **13**, **14**,[17] **15**,[15] and **3**.

Compound **4** was isolated as an oil and shown by HREIMS to have molecular formula $C_{17}H_{32}O_3$. Only sp^3 carbon signals, including three oxygenated carbon signals, were observed in the ^{13}C NMR spectrum of **4**. The hydroxyl group in **4** was evident from its IR spectrum (ν_{max} 3418 cm^{-1}). An ethoxyl group attached to a chiral carbon was revealed from ethoxyl signals at δ 3.32, 3.37 (each 1H, dq, J = 14.0, 6.9 Hz), and 1.11 (3H, t, J = 6.9 Hz). A broad signal at δ 3.55 (1H, H-5, resonate at δ_C 72.3), which showed HMBC correlation with δ_C 55.9 ($-OCH_2CH_3$) and NOESY correlations with signals at δ 3.32 and 3.37 ($-OCH_2CH_3$), was assigned as geminal to an ethoxy group. A doublet methyl signal (δ 0.83, d, J = 6.3 Hz), a singlet methyl signal (δ 1.20) on a carbon bearing a hydroxyl, and an isopropyl group [δ 0.71, 0.91 (each 3H, d, J = 6.9 Hz), 2.05 (1H, sep. d, J = 6.9, 3.0 Hz)] were also observed. Analysis of the ^1H and ^{13}C NMR (Table 1) of **4** clearly showed it to be structurally similar to *epi*-cubenol (**6**), the obvious differences being the absence of any double bonds and the presence of an additional hydroxyl and one ethoxyl group in **4**. The placement of a hydroxyl and the ethoxyl group at C-4 and C-5, respectively, was based on the following data. The signal at δ 3.55 was assigned as C-5$_\alpha$ (equatorial) due to having NOESY (see structure **16**) correlation with H_3-12 and H-11 of the isopropyl group. By using HMQC and HMBC techniques, the ^1H and ^{13}C signals were assigned. H-10 (δ 1.32, m) exhibited NOESY correlations with H-6 (δ 1.65, overlapped with other signals), H_β-8 (δ 1.06, m), and H_3-14, thus confirming the equatorial orientation of the C-10 methyl group. The absence of a NOE between H-10 and H_{ax}-2 (δ 1.41) suggested a *cis*-fused decaline structure, as in **16**. The presence of NOE cross-peaks between H-15 and H-4, and H-15 and $-OCH_2CH_3$, clearly established the β-equatorial orientation of the C-4 methyl. The ethoxy group on C-5 was assigned the β-axial orientation, since $-OCH_2CH_3$ showed NOE correlation with the H_β-3 (δ 1.57) axial proton. Thus, compound **4** was identified as 4α-hydroxy-5β-ethoxy-*epi*-cubenol.

The IR spectrum of **5** suggested the presence of hydroxyl (ν_{max} 3430 cm^{-1}) and acetoxyl groups (ν_{max} 1735 and 1224 cm^{-1}), which was further supported by ^1H and ^{13}C NMR signals at δ 2.08 (s), δ_C 21.1, and 169.2. The molecular formula of $C_{17}H_{30}O_4$ was established by HREIMS. Besides the acetyl group, signals for 15 carbons were evident in the ^{13}C NMR spectrum. Thus, **5** was proposed to be a sesquiterpene. The signals at δ 0.87 (3H, d, J = 6.7 Hz, H-14), 1.16 (3H, s, H-15), 5.05 (1H, br s, H-5), 1.82 (1H, m, H-11), 0.72 and 0.84 (each 3H, d, J = 6.9 Hz, H-12, −13), suggested **5** is an *epi*-cubenol derivative. The signal for H-5 (δ 5.05) showed an NOE correlation with the signals at δ 1.82, 0.72, and 0.84 (isopropyl group), indicating an α-equatorial orientation of H-5. Therefore, hydroxyl and acetoxyl groups were located at C-4 and C-5, respectively. Similarity of the ^1H and ^{13}C NMR data of **5** and **4** and the absence of a NOE correlation of H-10 to H_{ax}-2 (δ 1.51) provided evidence that **5** is a *cis*-fused decalin. The NOE observed between H_3-15 and both H-5 and H_2-3 (δ 1.24 and 1.85) suggested the β-equatorial orientation of H_3-15. Thus, compound **5** was identified as 4α-hydroxy-5β-acetoxy-*epi*-cubenol.

Experimental Section

General Experimental Procedures. Melting points were determined with a Yanagimoto micromelting point apparatus and are uncorrected. IR spectra were recorded on a Perkin-Elmer 983G spectrophotometer. ^1H and ^{13}C spectra were run on a Bruker DMX-400 spectrometer. EIMS and specific rotations were taken on a JEOL JMS-HX 300 mass spectrometer and a JASCO DIP-1000 digital polarimeter, respectively. Extracts were chromatographed on silica gel (Merk 70-230 mesh, 230-400 mesh, ASTM) and purified with a semipreparative normal-phase HPLC column (250 × 10 mm, 7 μm, LiChrosorb Si 60) taken on LDC Analytical-III.

Plant Material. The heartwood of *C. obtusa* var. *formosana* was collected from Taichung, Taiwan, in 1996. The plant was identified by Mr. Muh-Tsuen Gun, formerly of the Department of Botany, National Taiwan University. A voucher specimen has been deposited at the Herbarium of the Department of Botany, National Taiwan University, Taipei, Taiwan.

Extraction and Isolation. The dried heartwood of *C. obtusa* var. *formosana* (11 kg) was extracted with Me_2CO (120 L) at room temperature (3 days × 2). To the evaporated Me_2CO extract was added H_2O to bring the total volume to 1 L, and this phase was then partitioned with ethyl acetate (1 L × 3). The combined ethyl acetate layer afforded a black syrup (680 g) that was chromatographed on Si gel and by HPLC (normal phase on Lichrosorb Si 60), repeatedly using a hexane−EtOAc gradient solvent system. Muurola-4,10(14)-diene-3-one (**1**) (5.5 mg), *epi*-cubenol (**6**) (11.8 mg), T-cadinol (**7**) (11.2 mg), T-muurolol (**8**) (9.3 mg), δ-cadinol (**9**) (5.8 mg), α-cadinol (**10**) (16.7 mg), 10-*O*-acetyl-15-oxo-α-cadinol (**2**) (5.1 mg), 15-hydroxy-α-cadinol (**3**) (crude weight 15 mg), 4α-hydroxy-5β-ethoxy-*epi*-cubenol (**4**) (6.5 mg), and 4α-hydroxy-5β-acetoxy-*epi*-cubenol (**5**) (5.6 mg) were eluted with 5%, 5%, 10%, 10%, 10%, 10%, 20%, 30%, 50%, and 50% EtOAc in hexane solvent systems, respectively. Acetylation of crude **3** (15 mg) with Ac_2O and pyridine yielded **11** (14 mg).

Muurola-4,10(14)-dien-3-one (1): colorless oil; $[\alpha]^{25}_D$ −12.8° (*c* 0.31, $CHCl_3$); IR (KBr) ν_{max} 1672, 1663, 1655, 889, 830 cm^{-1}; UV (MeOH) λ_{max} (log ϵ) 238 (3.91) nm; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.86, 0.93 (each 3H, d, J = 6.9 Hz, H-12, -13), 1.59 (1H, m, H-7), 1.67−1.73 (2H, m, H-8), 1.77 (3H, br s, H-15), 1.89 (1H, m, H-11), 2.15−2.23 (2H, m, H-9), 2.25 (1H, dd, J = 16.9, 4.2 Hz, H_a-2), 2.34 (1H, m, H-6), 2.74 (1H, dd, J = 16.9, 12.8 Hz, H_b-2), 2.92 (1H, dt, J = 12.8, 4.3 Hz, H-1), 4.67 (2H, br s, H-14), 6.78 (1H, d, J = 5.0 Hz, H-5); ^{13}C NMR data, see Table 1; EIMS *m/z* 218 $[M]^+$ (6), 203 (3), 190 (70), 175 (45), 147 (100), 133 (40), 119 (41), 105 (68), 91 (80), 69 (48); HREIMS *m/z* 218.1675 (calcd for $C_{15}H_{22}O$, 218.1671).

10-*O*-Acetyl-15-oxo-α-cadinol (2): colorless oil; $[\alpha]^{25}_D$ −18.3° (*c* 0.75, $CHCl_3$); IR (KBr) ν_{max} 2723, 1734, 1690, 1642, 1380, 1369, 1245, 1114, 1025 cm^{-1}; UV (MeOH) λ_{max} (log ϵ) 232 (4.04) nm; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.82, 0.96 (each 3H,d J = 6.9 Hz, H-12, -13), 1.14−1.18 (2H, m, H-8), 1.23 (1H, m, H-7), 1.41(3H, s, H-14), 1.64 (2H, m, H_a-2, H_a-9), 1.72 (1H, m, H-1), 1.97 (3H, s, OC=OCH_3), 2.05 (1H, m, H_a-3), 2.06 (1H, m, H_b-2), 2.07 (1H, m, H-6), 2.20 (1H, m, H-1), 2.44 (1H, m, H_b-3), 2.64 (1H, d, J = 12.4 Hz, H_b-9), 6.84 (1H, br s, H-5), and 9.43 (1H, s, H-15); ^{13}C NMR data, see Table 1; EIMS *m/z* 218 $[M^+ - HOAc]$ (82), 189 (74), 175 (100), 157 (23), 148 (31), 105 (34); HREIMS *m/z* 218.1682 (calcd for $C_{15}H_{22}O$, 218.1671).

15-Hydroxy-α-cadinol (3): amorphous solid; $[\alpha]^{25}_D$ −30.6° (*c* 0.85, $CHCl_3$); IR (KBr) ν_{max} 3363, 3020, 1687, 1214, 1066,

1027, 757 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.73, 0.88 (each 3H, d, J = 6.9 Hz, H-12, -13), 1.03 (2H, m, H-7), 1.06 (3H, s, H-14), 1.21 (1H, m, H-1), 1.17, 1.58 (each 1H, m, H-8), 1.39 (1H, td, J = 12.5, 3.7 Hz, H-9), 1.72 (1H, m, H-6), 1.76 (1H, dt, J = 12.5, 2.9 Hz, H-9), 2.08 (2H, m, H-2), 2.12 (1H, m, H-11), 2.18 (2H, m, H-3), 3.94, 3.97 (each 1H, d, J = 13.0 Hz, H-15); ^{13}C NMR data, see Table 1; EIMS m/z 238 [M^+] (2), 220 ($M^+ - H_2O$)(26), 193 (54), 190 (93), 159 (100), 147 (78), 119 (68), 91 (100); HREIMS m/z 238.1935 (calcd for $C_{15}H_{26}O_2$, 238.1926).

4α-Hydroxy-5*β*-ethoxy-*epi*-cubenol (4): amorphous solid; $[\alpha]^{25}_D$ −9.9° (*c* 0.12, $CHCl_3$); IR (KBr) ν_{max} 3418, 1113, 1069, 1013, 875, 775 cm^{-1}; ^1H and ^{13}C NMR data, see Table 1; EIMS m/z 284 [M^+] (3), 185 (65), 149 (13), 100 (100), 86 (14); HREIMS m/z 284.2354 (calcd for $C_{17}H_{32}O_3$, 284.2351).

4α-Hydroxy-5*β*-acetoxy-*epi*-cubenol (5): amorphous solid; $[\alpha]^{25}_D$ +10.2° (*c* 0.12, $CHCl_3$); IR (KBr) ν_{max} 3430, 1735, 1224, 1016, 996 cm^{-1}; ^1H and ^{13}C NMR data, see Table 1; EIMS m/z 298 [M^+] (1), 280 (28), 227 (36), 220 (25), 195 (30), 177 (60), 167 (100), 159 (22); HREIMS m/z 298.2148 (calcd for $C_{17}H_{30}O_4$, 298.2144).

15-Acetoxy-α-cadinol (11): amorphous solid; $[\alpha]^{25}_D$ −34.7° (*c* 0.36, $CHCl_3$); IR (KBr) ν_{max} 3424, 3041, 1743, 1670, 1245, 1123, 1026 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.10 (1H, m, H-7), 1.12, 1.62 (each 1H, m, H-8), 1.24 (1H, m, H-1), 1.24, 2.07 (each 1H, m, H-2), 1.41, 1.79 (each 1H, m, H-9), 1.76 (1H, m, H-6), 2.00−2.10 (2H, m, H-3); ^{13}C NMR data, see Table 1; EIMS m/z 262 [$M^+ - H_2O$] (4), 187 (12), 177 (19), 159 (100), 147 (18), 119 (26), 105 (23); HREIMS m/z 262.1941 (calcd for $C_{17}H_{26}O_2$, 262.1933).

Saponification of 2 and 11 in Methanolic NaOH. Compound **2** (5 mg) or **11** (5 mg) was dissolved in 1 N NaOH methanolic solution (1 mL) for 5 h under stirring, and the solution was quenched with 15 mL of H_2O. After removal of MeOH by evaporating in vacuo, the product was extracted with CH_2Cl_2 and dried ($MgSO_4$) to afford **13** (3 mg) or **3** (3 mg).

Oxidation of α-Cadinol (10) with Selenium Dioxide. α-Cadinol (**10**) (from *Taiwania cryptomerioides* with same specific rotation from *C. obtusa* var. *formosana*) (1.543 g) and SeO_2 (1.06 g) in 30 mL of 95% ethanol were refluxed 6 h. The reaction mixture was filtrated through Celite, and the filtrate was purified by SiO_2 chromatography and then HPLC to yield four products: **13** (100 mg), **14** (264 mg),[15] **3** (81 mg), and **15** (123 mg).[15] The physical data of **13** are as follows: amorphous solid; $[\alpha]^{30}_D$ −6.3° (*c* 3.52, $CHCl_3$); IR (KBr) ν_{max} 3432, 3031, 2735, 1683, 1639, 1129, 1070 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz) δ 0.81, 0.95 (each 3H, d, J = 7.0 Hz), 1.10 (3H, s), 6.82 (1H, br s), and 9.40 (1H, s); EIMS m/z 236 [M^+] (6), 218 (42), 193 (100), 178 (44), 175 (75), 135 (44), 91 (80).

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