Chemical Constituents from *Alseodaphne andersonii*

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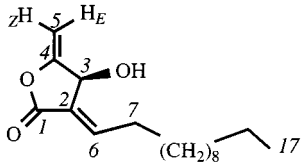
Five new compounds, including four C_{17} γ-lactones, dihydroisoobtusilactone (**1**), dihydroobtusilactone (**2**), 3-epilitsenolide D_2 (**3**), and 3-epilitsenolide D_1 (**4**), and one furanone alseodafuranone (**5**), were isolated from the root and stem of *Alseodaphne andersonii*. Their structures were elucidated mainly by spectral analysis (NMR and MS) and partially by chemical correlation.

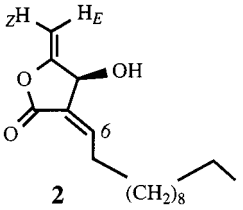
Cumulative studies have revealed that Lauraceous plants, particularly those of the *Litsea*, *Neolitsea*, *Phoebe*, *Cryptocarya*, and *Dehaasia* genera, contain various types of isoquinoline alkaloids.[1] Analysis of these alkaloidal compositions could serve as a basis for chemotaxonomy of these plants. Besides this, other components such as lactonic compounds[2] and flavonoid glycosides[3] were reported in certain Lauraceous species. To further clarify the application of chemotaxonomy in this family, a more thorough investigation on other genera is required, especially for those rarely studied ones, such as *Alseodaphne*. The following describes the outcome of our effort on the chemical study of *Alseodaphne andersonii* (King ex Hook. f.) Kosterm. (Lauraceae), a large tree indigenous to Yun-Nan Province, China.[4]

A preliminary study focusing on selective isolation of alkaloids by a general acid−base treatment of the ethanolic extract of the root and stem was performed, and it was found that this species contained very tiny amounts of alkaloids. Hence, a general liquid−liquid partitioning process was undertaken on the ethanolic extract, providing fractions soluble in hexane, $CHCl_3$, and MeOH−H_2O (1:1). Repeated chromatography on the hexane-soluble fraction yielded compound **1**. Repeated chromatography on the $CHCl_3$-soluble fraction yielded compounds **2**−**7**. Of these, compounds **2** and **6**, possessing very similar polarity but distinct skeletons, were separated via a Sephadex LH-20 column.

Compound **1**, a pale yellowish liquid, had molecular formula $C_{17}H_{28}O_3$, as deduced from HRFABMS. It contained an α,β'-conjugated γ-lactone moiety substituted with γ-exomethylene and β-hydroxy groups similar to isoobtusilactone[5] and isolancifolide,[2] as exemplified by the IR absorptions at 1769, 1672 cm^{-1}, UV absorption maximum at 222.5 nm, ^1H NMR signals at δ 7.04 (dt, J = 2.1, 7.8 Hz, H-6), 4.90 (dd, J = 2.7, 1.3 Hz, H_Z-5), 4.68 (dd, J = 2.7, 1.7 Hz, H_E-5), and 5.21 (m, H-3), and ^{13}C NMR signals at δ 166.89 (s, C-1), 127.29 (s, C-2), 150.15 (d, C-6), 157.67 (s, C-4), 91.29 (t, C-5), and 66.19 (d, C-3). However, it was found that **1** lacked a terminal vinyl group and its molecular formula had two more hydrogen atoms than that of isoobtusilactone. An NOED experiment enhanced H_E-5 (δ 4.68) (1.9%) and H-7 (δ 2.42) (3.3%) upon irradiation at H-3 signals, suggesting an *E*-configuration for $\Delta^{2(6)}$. These data and the optical property similar to isoobtusilactone, both levorotatory, indicated **1** to be 16,17-dihydroisoobtusilactone. An HMBC experiment was performed to assign most of its C-13 NMR signals (Table 2).



1



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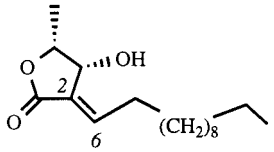

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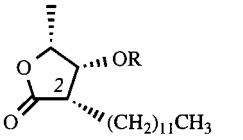
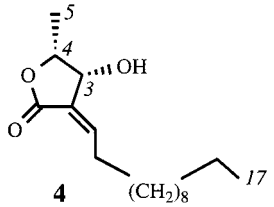


3a R= H
3b R= Ac



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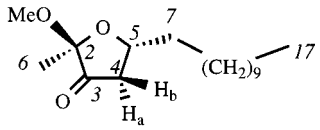

5

Compound **2**, a pale yellowish liquid, also had molecular formula $C_{17}H_{28}O_3$, as deduced from HRFABMS. Its spectral data (IR, UV, ^1H and ^{13}C NMR) were also very similar to those of **1**. The large difference between them appeared for H-6, δ 6.64 (dt, J = 2.0, 7.8 Hz) in **2** versus δ 7.04 in **1**, in their ^1H NMR spectra, suggesting a *Z*-configuration for $\Delta^{2(6)}$ in **2**. This proposal was confirmed by an NOED experiment which enhanced H_E-5 (δ 4.64) and H-6 (δ 6.64) upon irradiation at H-3 signals. Pooling these data and the optical property, $[\alpha]^{26}_D$ −44.7°, which established a 3*S*-configuration,[5,6] together established **2** to be 16,17-dihydroobtusilactone. The ^1H NMR and ^{13}C NMR data of **2**, except for some in the aliphatic side chain, were assigned by comparison with those of **1** (Tables 1 and 2).

Compound **3**, a colorless amorphous solid, mp 34−35 °C, had molecular formula $C_{17}H_{30}O_3$, as deduced from HRFABMS, two more hydrogen atoms than that in **1**. Its spectral data (IR, UV, ^1H and ^{13}C NMR) also indicated an α,β'-conjugated γ-lactone moiety having 3-hydroxy substitution, as exemplified by the IR absorption at 3487, 1732, 1678 cm^{-1}, UV absorption maximum at 215 nm, and ^1H NMR signals at δ 6.89 (dt, J = 1.4, 7.8 Hz, H-6), 4.77 (br d, J = 5.2 Hz, H-3). Its ^1H NMR spectrum lacked the signals for a γ-methylene group; instead an additional methyl doublet (δ 1.42) and a double quartet signal for a

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Table 1. ^{1}H NMR Data for Compounds **1**−**5** (δ/ppm, J in Hz) in $CDCl_3$ (400 MHz)

position	**1**	**2**	**3**	**4**	**5**[a]
3	5.21 m	5.08 dd (2.7, 3.4)	4.77 brd (5.2)	4.60 brd (5.4)	
4			4.50 dq (5.2, 6.6)	4.49 dq (5.4, 6.6)	2.63 dd (5.8, 18.4) (α) 2.05 dd (8.8, 18.4) (β)
5	4.68 dd (1.3, 2.7) (*E*) 4.90 dd (1.7, 2.7) (*Z*)	4.64 dd (1.7, 2.7) (*E*) 4.85 dd (1.7, 3.4) (*Z*)	1.42 d (6.6)	1.35 d (6.6)	4.25 ddt (5.8, 8.8, 6.2)
6	7.04 dt (2.1, 7.8)	6.66 dt (2.0, 7.8)	6.89 dt (1.4,7.8)	6.52 dt (1.2, 7.7)	1.34 s
7	2.42 m	2.73 m	2.36 dt (7.8, 7.4)	2.69 m	1.70 m
8	1.48 m	1.43 m	1.49 m	1.43 m	1.23 m
9−15	1.25m	1.23 m	1.23 m	1.22 m	1.23 m
16	1.25m	1.23 m	1.23 m	1.22 m	1.23 m
17	0.86 t (6.8)	0.86 t (6.8)	0.86 t (6.9)	0.84 t (6.9)	0.86 t (6.8)

[a] δ_{2-OMe} 3.24 (s).

Table 2. ^{13}C NMR Data (δ/ppm)[a] for **1**−**5** (100 MHz) and HMBC Data for **1** and **3** in $CDCl_3$ (400 MHz)

	1		**2**	**3**		**4**	**5**[c]
position	δ_C (mult)[b]	HMBC corr C#	δ_C (mult)	δ_C (mult)	HMBC corr C#	δ_C (mult)	δ_C (mult)
1	166.9 s		165.2 s	170.2 s		168.9 s	
2	127.3 s		126.8 s	130.5 s		129.2 s	100.5 s
3	66.2 d	1, 2	68.8 d	67.7 d	1, 2, 4, 6	71.2 d	209.0 s
4	157.7 s		157.5 s	79.0 d	3, 5	78.0 d	40.1 t
5	91.3 t	3, 4	90.3 t	13.9 q	3, 4	14.1 q	73.5 d
6	150.2 d	1, 3, 7	151.3 d	147.7 d	1, 2, 3, 7	149.6 d	14.1 q
7	∼29.5 t	2, 6, 8, 9	∼29.5 t	∼29.5 t	2, 6, 8, 9	∼29.5 t	35.3 t
8	28.3 t	6	28.3 t	28.4 t	7, 9, 10	27.8 t	∼29.6 t
15	31.8 t		31.9 t	31.9 t		31.9 t	31.9 t
16	22.6 t	15, 17	22.6 t	22.6 t	15, 17	22.6 t	22.7 t
17	14.0 t	15, 16	14.0 t	14.1 t	15, 16	14.1 t	16.4 q

[a] The chemical shifts for C-9 through C-14 are between δ 29.7 and 28.7. [b] Multiplicities were obtained from DEPT experiments. [c] In **5**, δ_{2-OMe} 48.8 (q).

carbinoyl proton (δ 4.50) were present. These data suggested **3** to be a 4,5-dihydro analogue of **1**, possibly the C-3 isomer of litsenolide D_2.[7] An NOED experiment which enhanced H-4 (7.3%), H-7 (δ 2.36) (5.3%), and H-8 (δ 1.49) (1.5%) upon irradiation at H-3 suggested a cis relationship of 3-OH and 4-Me and an *E*-conformation for $\Delta^{2(6)}$ for lack of significant NOE between H-3 and H-6.

To establish the absolute configuration at C-3 and C-4, **3** was catalytically hydrogenated, followed by *O*-acetylation and elimination of the acetoxy group to yield 2-dodecyl-4α-methyl-2-butenolide, $[\alpha]^{26}_D$ −37.3° (*c* 1.0, dioxane) (lit. −29.8°),[8] having spectral data identical to those reported.[8] Since the C-4 stereochemistry of this product had been determined to be the *R*-configuration by further chemical degradation to the known methyl lactate,[8] **3** has a 3*R*,4*R*-configuration, different from 3*S*, 4*R* in litsenolide D_2. Therefore, compound **3** is a new compound and is named 3-epilitsenolide D_2.

Compound **4** had molecular formula $C_{17}H_{30}O_3$, as deduced from HRFABMS, the same as **3**. Its spectral data (IR, UV, ^{1}H and ^{13}C NMR) were also very similar to those of **3**. The large difference between them was the H-6 signal, δ 6.52 (dt, J = 1.2, 7.7 Hz) in **4** versus δ 6.89 in **3**, in their ^{1}H NMR spectra, suggesting a *Z* conformation for $\Delta^{2(6)}$ in **4**. The close CD curves, both having a large negative Cotton effect at 226 (**3**) and 222 nm (**4**), suggested the same 3*R*,4*R*-stereochemistry. Hence, **4** is a C-3 isomer of the known litsenolide D_1[7] and is a new compound named 3-epilitsenolide D_1.

Compound **5** had molecular formula $C_{17}H_{32}O_3$, as deduced from HREIMS. Its IR spectrum revealed a strong absorption at 1774 cm^{-1}, suggesting a five-membered carbonyl function, confirmed by a carbon signal at δ 208.99 (s, C-3). The ^{1}H NMR spectrum displayed a MeO (δ 3.26, 2-OMe), one methyl singlet (δ 1.34, 2-Me), and two geminal protons at δ 2.63 (dd, J = 18.4, 5.8 Hz) and 2.05 (dd, J = 18.4 and 8.8 Hz), both of which coupled to a carbinoyl proton at δ 4.25 (ddt, J = 8.8, 5.8, 6.2 Hz, H-5), evidenced by a COSY-45 spectrum. This COSY spectrum also revealed the coupling of the latter proton (δ 4.25) to a methylene group at δ_H 1.70 (2H, m, H-6), which was a part of a long aliphatic chain with the terminal methyl group at δ 0.86 (t, J = 6.8 Hz). Its ^{13}C NMR data showed a ketal carbon at δ 100.53 (s, C-2) and a carbinoyl carbon at δ 73.53 (d, C-5). These data taken together would build up a 2-methoxy-2-methyl-5-alkyl-3(2*H*)-dihydrofuranone structure for **5**. The aliphatic side chain would contain 11 carbons, $-(CH_2)_{10}CH_3$, as deduced by the subtraction of this furan moiety from the formula. This proposed skeleton was confirmed by analysis of an HMBC spectrum, which revealed the coupling of both methyl (^2J) and methoxy (^3J) singlets to the ketal carbon and the methyl singlet (^3J) and γ-methylene protons' (^2J) signals to the β-carbonyl signal. The relative stereochemistry at C-2 and C-5 was determined by two NOED experiments. That irradiation at the frequency of H-5 did not enhance the intensity of 2-Me and 2-OMe and irradiation at the methyl singlet only enhanced the 2-OMe signal (2.6%) would suggest the trans relationship between the 5-alkyl group and 2-OMe. Hence the structure of compound **5** was elucidated as (2,5-*E*)-2-methoxy-2-methyl-5-undecyl-3(*H*)-dihyrofuranone. The absolute stereochemistry in **5**, however, remains to be clarified. To our knowledge, **5** represents the first occurrence of such a compound, and it was named alseodafuranone.

Two additional compounds had molecular formula $C_{10}H_{12}O_4$, as deduced from EIMS and NMR spectral data. They contained a phenolic group H-bonded to an ester carbonyl group, as exemplified by IR absorption at 3405 and 1629 cm^{-1}, a bathochromic shift in the UV spectrum under alkaline conditions, and a D_2O exchangeable proton signal at δ 12.01 (s, 2-OH). These were identified as methyl β-orcinolcarboxylate[9] and ethyl orsellinate,[9,10] respectively, by comparison of their spectral data (^{1}H NMR, UV, MS) to those reported. These two structures were further con-

firmed by NOED experiments. The complete ^{13}C NMR assignment of β-orcinolcarboxylate was made by analysis of an HMBC spectrum as listed in the Experimental Section. The complete ^{13}C NMR assignment of orsellinate listed in the Experimental Section was made by comparing to that of β-orcinolcarboxylate and correlation with that calculated from the substitution effect on the benzene ring.

This study resulted in the isolation and characterization of four new lactonic compounds (**1**−**4**) and one new furanone (**5**). Among these, similar lactonic type compounds had been isolated frequently from the *Clinostemon*,[5] *Lindera*,[6,11] and *Litsea*[7] genera. The above findings and the low alkaloid content in *Alseodaphne andersonii* could serve to differentiate the plants of this genus from those of the genera mentioned above.

Experimental Section

General Experimental Procedures. A JASCO DIP-181 digital polarimeter; a Perkin-Elmer 1760-X infrared FT spectrometer (KBr); a Hitachi 2000 UV (MeOH); a JASCO J-710 spectropolarimeter (MeOH); a JEOL JMX-HX110 mass spectrometer; and a Bruker AMX-400 NMR spectrometer in $CDCl_3$ (δ_H 7.24, δ_C 77.0) were used using Bruker's standard pulse programs: in the HMQC and HMBC experiments, $\Delta = 1$ s and $J = 140$, 8 Hz, respectively, and the correlation maps consisted of 512 × 1K data points per spectrum, each composed of 32−64 transients.

Plant Material. The plant material, collected in July 1994 in Xishuangbanna, Yunnan Province, Mainland China, was authenticated by Dr. Su-Lin Young and colleagues in the Institute of Medicinal Plant Development, Chinese Academy of Medical Sciences, Beijing, China. A voucher specimen has been deposited in that Institute.

Extraction and Isolation. The dry powder of the stem (14.2 kg) was percolated with 95% EtOH (100 L × 3) to give the EtOH extract (1.05 kg) upon concentration under reduced pressure. A portion of the extract (500.0 g) was triturated with 80% $MeOH_{aq}$ (0.5 L × 3). The soluble fraction obtained by centrifugation was then partitioned against hexane (1 L × 3) to give the hexane-soluble fraction (28.2 g). The MeOH layer was then adjusted to 50% $MeOH_{aq}$ by adding H_2O, and the resultant suspension was partitioned with $CHCl_3$ (1 L × 3) to give a precipitate (35.5 g) and two fractions soluble in $CHCl_3$ (8.4 g) and 50% MeOH (34.1 g) upon evaporation.

The hexane-soluble fraction (20.2 g) was chromatographed on a silica gel (550 g, finer than 230 mesh) column, eluted with EtOAc (0−20%) in hexane to give three fractions. Fractions 1 and 3 were found to be lipids and fatty acids. Fraction 2 (1.06 g) obtained from 10% EtOAc elution was rechromatographed on a silica gel (40 g, 230−400 mesh) column, eluted with MeCN (1−3%) in hexane to give **1** (131 mg). The $CHCl_3$-soluble fraction (7.3 g) was chromatographed on a silica gel (290 g, 230−400 mesh) column, eluted with MeOH (0−30%) in $CHCl_3$, to give eight fractions. Fractions 1 and 3 were found to be lipids and fatty acids. Fraction 2 (326 mg) obtained from $CHCl_3$ elution was rechromatographed on a Sephadex LH-20 column [200 mL, $CHCl_3$−MeOH (7: 3)] to give compounds **2** (255 mg) and β-orcinolcarboxylate (3.2 mg). Fraction 4 (194 mg), obtained from 0.5% MeOH elution, was rechromatographed on a silica gel (10 g, 230−400 mesh) column eluted with EtOAc (0−8%) in hexane to give compound **4** (91 mg). Fraction 6 (137 mg) was rechromatographed on a silica gel (10 g, 230−400 mesh) column eluted with EtOAc (0−10%) in hexane to give compounds **5** (5.3 mg), orsellinate (6.2 mg), and **3** (79.6 mg).

16,17-Dihydroisoobtusilactone (1): colorless liquid; R_f 0.20 [^{i}PrOH−hexane (1:19)]; $[\alpha]^{26}_D$ −36.8° (*c* 0.57, $CHCl_3$); IR ν_{max} 3435 (br, OH), 2925, 2854, 1768 (s, γ-lactone), 1671, 1466, 1272, 1023 cm^{-1}; UV λ_{max} (MeCN, log ϵ) 222.5 (4.12) nm; CD (3.57×10^{-5} M, MeCN) ($\Delta\epsilon$) 270 (−1.49), 226 (−5.46); ^1H and ^{13}C NMR data, see Tables 1 and 2; FABMS (pos.) *m*/*z* [M + H]$^+$ 281 (44), [M + H − H_2O]$^+$ 263 (24), 55 (74), 43 (100); HRFABMS (pos.) *m*/*z* [M + H]$^+$ 281.2127 (calcd for $C_{17}H_{29}O_3$ 281.2117).

16,17-Dihydroobtusilactone (2): colorless liquid; R_f 0.37 [^{i}PrOH−hexane (1:9)]; $[\alpha]^{26}_D$ −44.7° (*c* 0.76, $CHCl_3$); IR ν_{max} 3435 (br, OH), 2923, 2853, 1766 (s, γ-lactone), 1678, 1463, 1367, 1090, 965, 855, 813, 723 cm^{-1}; UV λ_{max} (MeCN, log ϵ) 225 (4.07) nm; CD (3.57×10^{-5} M, MeCN) ($\Delta\epsilon$) 273 (−1.88), 227 (−3.54); ^1H and ^{13}C NMR data, see Tables 1 and 2; FABMS (pos.) *m*/*z* [M + H]$^+$ 281 (17), [M + H − H_2O]$^+$ 263 (7), 139 (15), 69 (38), 55 (75), 43 (100); HRFABMS (pos.) *m*/*z* [M + H]$^+$ 281.2122 (calcd for $C_{17}H_{29}O_3$ 281.2117).

3-Epilitsenolide D$_2$ (3): amorphous solid, mp 34 °C; R_f 0.17 [EtOAc−hexane (1:4)]; $[\alpha]^{26}_D$ −69.8° (*c* 1.29, dioxane); IR ν_{max} 3387 (br, OH), 2924, 2848, 1729 (s, γ-lactone), 1693, 1672, 1460, 1219, 1038, 993, 919, 734 cm^{-1}; UV λ_{max} (MeCN, log ϵ) 215 (4.27) nm; CD (3.75×10^{-5} M, MeCN) ($\Delta\epsilon$) 266 (+0.61), 226 (−9.74); ^1H and ^{13}C NMR data, see Tables 1 and 2; FABMS (pos.) *m*/*z* [M + H]$^+$ 283 (100), 265 (19, [M + H − H_2O]$^+$), 214 (15), 57 (50); HRFABMS (pos.) *m*/*z* [M + H]$^+$ 283.2267 (calcd for $C_{17}H_{31}O_3$ 283.2273).

3-Epilitsenolide D$_1$ (4): amorphous solid, mp 50 °C; R_f 0.42 [MeOH−$CHCl_3$ (3:97)]; $[\alpha]^{26}_D$ −34.7° (*c* 1.04, dioxane); IR ν_{max} 3486 (br, OH), 2919, 2849, 1732 (s, γ-lactone), 1677, 1470, 1377, 1214, 1038, 997, 929, 674 cm^{-1}; UV λ_{max} (MeCN, log ϵ) 217.5 (4.02) nm; CD (3.57×10^{-5} M, MeCN) ($\Delta\epsilon$) 222 (−3.61); ^1H and ^{13}C NMR data, see Tables 1 and 2; FABMS (pos.) *m*/*z* [M + H]$^+$ 283 (100), [M + H − H_2O]$^+$ 265 (15); HRFABMS (pos.) *m*/*z* [M + H]$^+$ 283.2275 (calcd for $C_{17}H_{31}O_3$ 283.2273).

Alseodafuranone (5): R_f 0.42 [EtOAc−hexane (1:4)]; $[\alpha]^{26}_D$ −21.4° (*c* 0.14, $CHCl_3$); IR ν_{max} 2925, 2854, 1774 (s, C=O), 1463, 1101 cm^{-1}; UV λ_{max} (MeCN, log ϵ) 240 (3.02) nm; ^1H and ^{13}C NMR data, see Tables 1 and 2; HMBC ($CDCl_3$) data: 1-OMe to C-1, H-3 to C-2, C-4, and C-6, H-5 to C-1 and C-2, H-15 to C-16, H-16 to C-14 and C-15; EIMS *m*/*z* [M]$^+$ 284 (5), 253 (20, [M − MeO]$^+$), 209 (18, M − $C_3H_7O_2$]$^+$), 182 (28, [M − $C_4H_6O_3$]$^+$), 97 (34), 75 (100), 43 (72); HREIMS *m*/*z* [M]$^+$ 284.2345 (calcd for $C_{17}H_{32}O_3$ 284.2351).

Preparation of 2-Dodecyl-4α-methyl-2-butenolide. Compound **3** (30.7 mg) dissolved in EtOH (3.5 mL) was hydrogenated (H_2, 1atm) over 10% Pd−C (20 mg) at room temperature overnight. After general workup, the reaction mixture was purified on a Si gel column eluted with $CHCl_3$ to give the 2,6-dihydro product **3a** (16.2 mg): ^1H NMR ($CDCl_3$) δ 4.45 (1H, dq, $J = 3.0$, 6.4 Hz, H-4), 4.29 (1H, br s), 2.55 (1H, m, H-2), 1.41 (3H, d, $J = 6.4$ Hz), 0.85 (3H, t, $J = 7.2$ Hz, H-17). **3a** (16 mg) was *O*-acetylated (Ac_2O−py, each 1 mL) overnight, and the essentially pure acetylated product **3b** (15 mg) was obtained via evaporation of the reaction mixture under vacuum. **3b**: ^1H NMR ($CDCl_3$) δ 5.57 (1H, dd, $J = 3.4$, 5.1 Hz), 4.54 (1H, dq, $J = 3.4$, 6.5 Hz, H-4), 2.68 (1H, m, H-2), 2.11 (3H, s, OAc), 1.29 (3H, d, $J = 6.5$ Hz), 0.85 (3H, t, $J = 6.7$ Hz, H-17). **3b** was then passed through an aluminum oxide column (basic, 7.66 g) eluted with $CHCl_3$ to give 2-dodecyl-4α-methyl-2-butenolide (6.7 mg), a colorless viscous liquid: $[\alpha]^{26}_D$ −37.3° (*c* 1.0, dioxane) [lit. −29.8° (*c* 0.541)];[8] ^1H NMR ($CDCl_3$) δ 6.96 (1H, br s, H-3), 4.97 (1H, br q, $J = 6.8$ Hz, H-4), 2.24 (2H, t, $J = 7.0$ Hz, H-6), 1.38 (3H, d, $J = 6.8$ Hz, H-5), 0.85 (3H, t, $J = 6.4$ Hz, H-17); FABMS (pos.) *m*/*z* [M + H]$^+$ 267 (100).

Additional data for methyl α-orcinolcarboxylate: R_f 0.36 [^{i}PrOH−hexane (1:9)]; ^{13}C NMR ($CDCl_3$) δ 172.47 (s, C-7), 162.92 (s, C-2), 158.01 (s, C-4), 140.12 (s, C-6), 110.44 (d, C-5), 108.41 (s, C-3), 105.18 (s, C-1), 51.75 (q, 7-OMe), 24.06 (q, 6-Me), 7.61 (q, 3-Me); HMBC ($CDCl_3$ + D_2O) H-5 to C-1, C-3, and 6-Me, 7-OCH_3 to C-7, 3-CH_3 (δ 2.08) to C-2, C-3, and C-4, 6-CH_3 to C-1, C-5, and C-6.

Additional data for ethyl orsellinate: R_f 0.26 [EtOAc−hexane (1:4)]; ^{13}C NMR ($CDCl_3$) δ 171.67 (s, C-7), 165.36 (s, C-4), 160.16 (s, C-2), 144.02 (s, C-6), 111.27 (s, C-1), 108.80 (d, C-5), 101.28 (d, C-3), 61.28 (t, 7-OCH_2CH$_3$), 24.32 (q, 6-Me), 14.22 (q, 7-OCH$_2$CH_3).

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References and Notes

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