Registry No. L-**1**, 59865-23-5; D-**1**, 81177-28-8; **2**, 122213-51-8; **3**, 126459-24-3; **4**, 126459-25-4; **5**, 122213-53-0; **6**, 126459-26-5; **7**, 126459-27-6; **7** (R = $CH_2CH_2CH_3$), 126459-57-2; **8**, 126459-28-7; **9**, 126459-29-8; (4*S*)-**10**, 126459-30-1; (4*R*)-**10**, 126459-61-8; **11**, 126459-31-2; **12**, 126459-32-3; **13**, 126459-33-4; (*E*)-**14**, 126459-34-5; (*Z*)-**14**, 126459-58-3; (2′*R*)-**15**, 126459-35-6; (2′*S*)-**15**, 126459-59-4; **16**, 51703-97-0; (2′*R*)-**17**, 126459-36-7; (2′*S*)-**17**, 126459-60-7; (4*S*,1′*S*,2′*S*)-**19**, 126459-37-8; (4*S*,1′*S*,2′*R*)-**19**, 126576-03-2; (4*R*,1′*R*,2′*R*)-**19**, 126576-04-3; (4*R*,1′*R*,2′*S*)-**19**, 126576-05-4; **20**, 126459-38-9; **21**, 126459-39-0; **22**, 126459-40-3; **23**, 126459-41-4; **24**, 126459-42-5; **25**, 126459-43-6; **27**, 126459-44-7; **28**, 126459-45-8; **29**, 126459-46-9; **30**, 126459-47-0; **32**, 126501-62-0; **33**, 126459-48-1; (4*R*,5*S*,1′*S*)-**34**, 126459-49-2; (4*S*,5*R*,1′*R*)-**34**, 126459-62-9; **36**, 126459-50-5; (2*R*,3*S*,4*S*)-**37**, 126459-51-6; (2*S*,3*R*,4*R*)-**37**, 126576-06-5; L-(*R*)-**38**, 126459-52-7; D-(*R*)-**38**, 126637-91-0; (2*R*,3*S*,4*S*,6*Z*)-**39**, 126459-53-8; (2*S*,3*R*,4*R*,6*Z*)-**39**, 126576-07-6; **40**, 126459-54-9; L-**42**, 81135-31-1; L-(*R*)-**43**, 126459-55-0; BuLi, 109-72-8; EtLi, 811-49-4; EtMgBr, 925-90-6; $CH_3C{\equiv}CCH_2CH_2MgBr$, 126459-56-1; (*E*)-$CH_3CH{=}CHCH_2CH_2MgBr$, 37586-56-4; *s*-BuLi, 598-30-1; *s*-BuMgBr, 922-66-7; $BrCH_2CH_2C{\equiv}CCH_3$, 18719-27-2; $HOCH_2CH_2C{\equiv}CCH_3$, 10229-10-4; (*E*)-$HOCH_2CH_2CH{=}CHCH_3$, 764-37-4; (*E*)-$BrCH_2CH_2CH{=}CHCH_3$, 7515-62-0; Me_2CO, 67-64-1; (*R*)-(+)-PhCH(Me)NH_2, 3886-69-9; (±)-PhCH(Me)NH_2, 618-36-0; cyclosporin, 79217-60-0.

Tilifodiolide, Tetraline-Type Diterpenoid of Clerodanic Origin from *Salvia tiliaefolia*†

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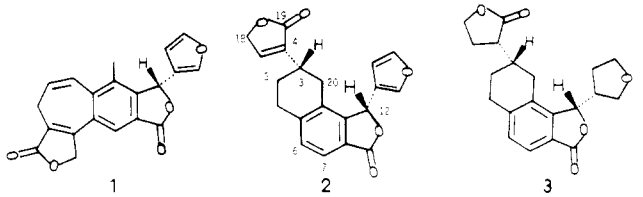
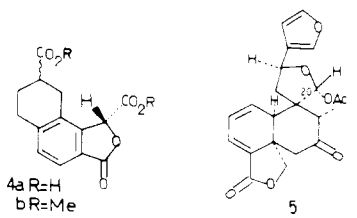
From the aerial parts of *Salvia tiliaefolia* Vahl, the known isosalvipuberulin (**1**) and two new clerodane diterpenoids, salvifolin and tilifodiolide, were isolated. The structure of salvifolin (**5**) was deduced from spectral data. The structure of tilifodiolide (**2**) was established by chemical and spectral means and X-ray diffraction analysis.

The *Salvia* spp. of Mexico and Central and South America have been classified in the Calosphace subgenus subdivided in 91 sections.[1] Systematic chemotaxonomic study of the Mexican *Salvia* species revealed an interesting relationship between the diterpenoid content of the species under study and the section to which it belongs.[2] There are however, some deviations from this generalization. Recently we described the structure elucidation of two new diterpenoids isolated from *Salvia puberula*,[3] which was classified in the Section Holwaya (Ramamoorthy)[4] closely related to Section Fulgentes.

In this paper we describe the diterpenoid constituents of *Salvia tiliaefolia* Vahl, a species classified[1] in Section Angulatae, Subsection Tiliaefoliae, which is not botanically related to Section Fulgentes. From the polar fraction of the acetone extract of *Salvia tiliaefolia*, we isolated the known isosalvipuberulin (**1**), previously obtained from



1 **2** **3**



4a R=H
b R=Me

5

Table I. ^1H NMR (400 MHz) and ^{13}C NMR (75 MHz) Data of Compound **2** ($CDCl_3$, TMS as Internal Standard)

C	^{13}C[a]	^1H δ[b]
1	29.2 t	3.01 ax, eq m
2	26.6 t	2.18 eq m; 1.76 ax, dddd (6, 9, 12, 13)
3	30.8 d	2.79 br t (12)
4	137.2 s	
5	143.8 d	6.95 dt (2, 1.5)
6	130.9 d	7.3 d (8)
7	123.0 d	7.7 d (8)
8	123.7 s	
9	130.5 s	
10	143.2 s	
11	147.2 s	
12	74.3 d	6.33 s
13	120.9 s	
14	142.1 d	7.52 t (1)
15	144.2 d	7.37 t (2)
16	108.7 d	6.08 dd (2, 1)
17	170.3 s	
18	173.3 s	
19	70.2 t	4.75 t (1.5)
20	30.4 t	2.90 eq, dd (4, 16); 2.21 ax, dd (1, 2, 16)

[a] SFORD multiplicities (at 20 MHz). [b] ax = pseudo-axial; eq = pseudo-equatorial, established by coupling patterns and NOE data.

Salvia puberula,[3] and a new diterpenoid named tilifodiolide, whose structure **2** was established by chemical and

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(3) The name puberulin has been previously used for a coumarin isolated from *Agathosma puberula* (*Phytochemistry* **1976**, *15*, 1080. Professor Rivett and Dr. S. A. Brown, personal communication); for this reason, we propose to name the diterpenoid described as "puberulin" (*J. Org. Chem.* **1988**, *53*, 3933) as *Salvipuberulin*.

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spectroscopic means and X-ray diffraction analysis. From a second population of *Salvia tiliaefolia*, salvifolin (**5**) was also isolated. Its structure was deduced from spectral data.

Results and Discussion

Tilifodiolide (**2**) had a molecular formula $C_{20}H_{16}O_5$ by high resolution mass spectrometry. Its IR spectrum showed the bands due to α,β-unsaturated γ-lactone functions (1770, 1650 cm^{-1}), a β-substituted furan (1500, 873 cm^{-1}), and an aromatic ring (1600 cm^{-1}).

The ^1H NMR spectrum of **2** (Table I) showed the characteristic signals due to a β-substituted furan ring at δ 6.08 (1 H, dd, J = 2, 1 Hz), 7.37 (1 H, t, J = 2 Hz), and 7.52 (1 H, t, J = 1 Hz). A singlet at δ 6.33 (1 H) was assigned to H-12. The presence of an abundant fragment at m/z 269 (100%, $M^+ - 68$) and at m/z 95 (10) in the mass spectrum is in agreement[5] with the existence of a lactone group and a furan ring bound to C-12. Two doublets at δ 7.3 (1 H, J = 8 Hz) and 7.7 (1 H, J = 8 Hz) were assigned to an aromatic AB system which suggested the presence of a tetrasubstituted aromatic ring in tilifodiolide (**2**). A triplet at δ 4.75 (2 H, t, J = 1.5 Hz) was attributed to C-19 methylene and was shown to be coupled to the vinylic H-5 observed at δ 6.95 (1 H, dt, J = 2, 1.5 Hz).

The remaining assigments were established by a careful analysis of the ^1H and ^{13}C NMR data. The methine proton H-3 was observed as a broad triplet at δ 2.79 (1 H, J = 12 Hz). COSY spectra showed a long-range coupling of this proton with H-5 and H-19. Two double doublets at δ 2.90 (J = 4 and 16 Hz) and 2.21 (J = 12 and 16 Hz) were assigned to the ψ equatorial and axial protons of C-20. A complex signal at δ 1.76 (dddd, J = 6, 9, 12, and 13 Hz) could be attributed to H-2ψ axial. A COSY experiment established a long-range coupling between H-12 and H-6, H-7, CH_2-1, CH_2-20, besides the expected interaction with the furan protons.

The ^{13}C NMR spectrum (Table I), APT,[16] and SFORD experiments revealed the presence of one methine and three methylene sp^3 carbon atoms. A triplet at δ 70.2 ppm was assigned to C-19 and a doublet at 74.3 to C-12. Two resonances at 170 ppm and 173 ppm were assigned to the lactone carbonyl groups. The assignments of the protonated ^{13}C resonances were obtained from a HETCOR[18] spectrum and selective INEPT[19] experiments led to the assignment of the quaternary sp^2 carbon resonances (Table I), which were confirmed by a ^{13}C–^{13}C correlation experiment. The furan carbon assignments are in agreement with the resonances observed in related structures.[3]

The selective INEPT experiments established the following long-range ^{13}C–^1H correlation (two or three bonds): H-2 (ax) to C-1, 3, 4, 10, and 20; H-19 to C-4, 5, and 18; H-12 to C-13, 14, 8, 9, 11, and 17; H-5 to C-4, 19, 18, and 3; H-6 to C-10, 1, 8, and 9; H-7 to C-17, 11, and 10.

The relative stereochemistry, assigned to the chiral centers C-3 (*S*) and C-12 (*R*) represented in **2**, was established taking into consideration the coupling constants of the protons attached to these carbons, and the NOESY[21] experiments in which a larger NOE effect is observed between H-12 and H-20 eq than between H-12 and H-20 ax. A NOE effect is also observed between H-5 and H-3, H-2 eq, H-2 ax, H-20 ax, and H-20 eq, being larger with H-20 ax than with H-20 eq.

The structure **2** proposed for tilifodiolide is quite unusual; consequently an X-ray diffraction analysis was carried out. Tilifodiolide crystallizes with two independent molecules per asymmetric unit (molecules A and B). Both molecules are closely similar in their bond lengths and angles, except for the bond distances C(1B)–C(10B), C(1A)–C(2A), C(3B)–C(20B), and C(6B)–C(7B), which are shorter than their counterparts by more than 3σ. Other bond-length differences involving the atoms of furan rings can be explained in terms of some thermal disorder.

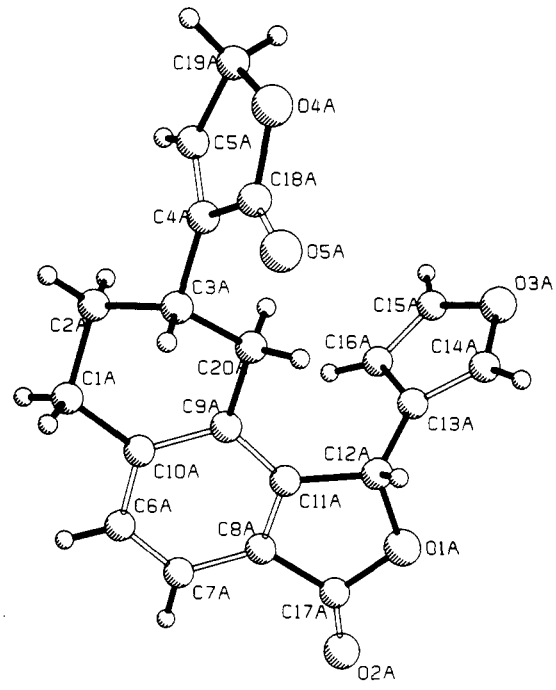

Figure 1. View of tilifodiolide molecule A showing atom numbering.

In both A and B molecules, the α,β unsaturated lactone, the hydrobenzofuranone, and the furan rings are planar within experimental error. The six-membered ring composed of C(1), C(2), C(3), C(20), C(9), and C(10) exhibit half-chair conformation, and the substituents at C(3) (lactone) and C(12) (furan) hold a syn relationship.

The major differences between molecules A and B arise from the orientations of the lactone and furan moieties with respect to the hydrobenzofuranone moiety. While in both molecules the furan rings are almost perpendicular, with angles between planes of 81.2° for the A molecule (92.0° for the B molecule), the inclination of the lactone changes drastically from 111.2° for molecule A to 26.2° on molecule B. This dramatic change in orientation probably results from the head-to-tail packing between molecules A and B in order to minimize repulsions.

Exhaustive catalytic hydrogenation of tilifodiolide (**2**) gave the hexahydro derivative **3** whose ^1H NMR spectrum showed the aromatic protons 6 and 7 as an AB system at δ 7.25 (1 H, d, J = 8 Hz) and 7.62 (1 H, d, J = 8 Hz). No other vinylic or aromatic protons were observed.

Ozonolysis of **2** followed by oxidative treatment of the ozonide (see Experimental Section) gave the diacid **4a** which was converted to the dimethyl ester **4b** with ethereal diazomethane. NMR analysis (see Experimental Section) indicated that **4b** was an equimolar mixture of C-3 epimers.

Attempts to aromatize the A ring of tilifodiolide (**2**) were unsuccessful.

The presence of isosalvipuberulin A and tilifodiolide (**2**) in *Salvia tiliaefoliae* suggest that they may be derived from a common biogenetic precursor such as salvigenolide A. Thus, a functionalization from salvigenolide A at C-20 could give **2** as outlined in Scheme I.

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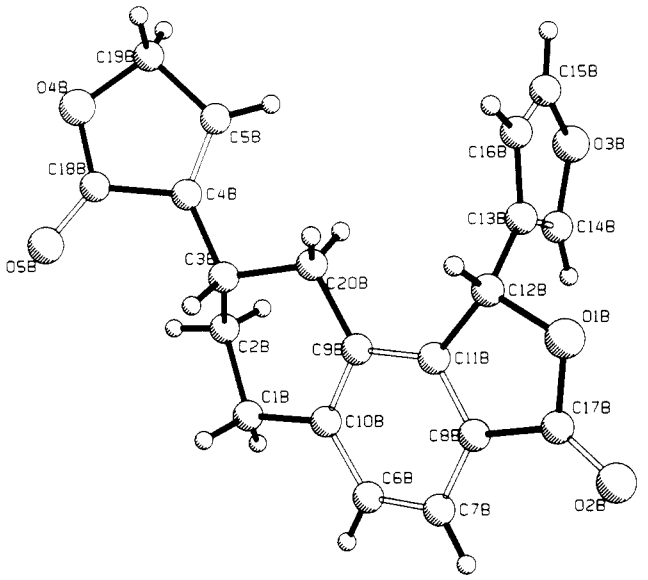
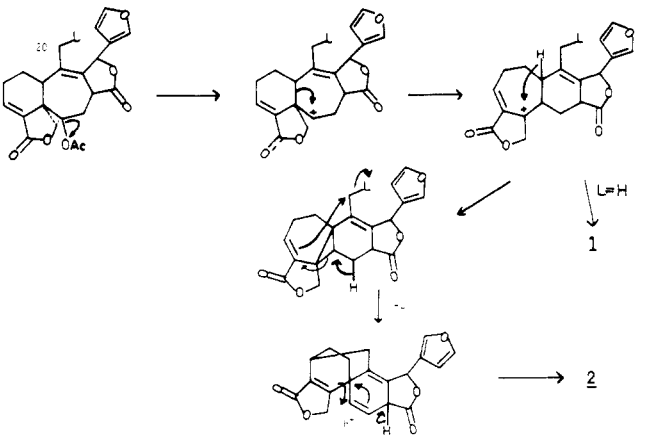


Figure 2. View of tilifodiolide (molecule B).

Scheme I



Tilifodiolide (**2**) constitutes, to our knowledge, the first natural product with a substituted tetralin skeleton, which could be biogenetically derived from a clerodane precursor.

From a second population of *Salvia tiliaefolia* collected in Cuernavaca (Mexico), isosalvipuberulin (**1**) and tilifodiolide (**2**) were isolated together with a new neoclerodane diterpenoid, salvifolin, whose structure **5** was deduced on spectroscopic evidence. Salvifolin (**5**), $C_{22}H_{22}O_7$, had IR frequencies attributed to a furan ring (1504 and 875 cm^{-1}) and an unsaturated γ-lactone function (1755, 1668, 1596 cm^{-1}). A band at 1718 cm^{-1} could be ascribed to a cyclohexanone absorption. The ^1H NMR spectrum of **5** (Table II) revealed the presence of an $\alpha,\beta,\gamma,\delta$-diunsaturated γ-lactone function in ring A, similar to the functionality found in linearolactone[6] and related neoclerodane diterpenes.[7] The ^{13}C NMR spectrum (Table II) confirmed this assignment. A doublet observed at δ 1.16 (3 H, J = 7 Hz) was ascribed to the C-17 secondary methyl group. A singlet (3 H) at δ 1.96 was due to an acetyl function. The ^1H NMR spectrum of **5** showed a singlet at δ 5.69 which was assigned to a ketalic proton; a doublet at δ 97.9 in the ^{13}C NMR spectrum of **5** (Table II) confirmed this assignment. This resonance was assigned to C-20 by comparison

Table II. ^1H NMR (80 MHz) and ^{13}C NMR (20 MHz) Data of 5 ($CDCl_3$, TMS as Internal Standard)

C	^{13}C δ^a	^1H δ^b
1	126.2 d	6.19 br dd (9, 3)
2	128.6 d	6.50 ddd (9, 5, 3)
3	134.9 d	7.03 d (5)
4	130.6 s	
5	59.1 s	
6	43.6 t	
7	206.7 s	
8	49.1 d	2.95 q (7)
9	59.1 s	
10	45.3 d	3.15 br s
11	43.6 t	(a) 2.71 dd (13, 8); (b) 2.27 dd (13, 8)
12	74.6 d	5.22 t (8)
13	128.6 s	
14	108.6 d	6.32 dd (2, 1)
15	143.8 d	7.36 t (2)
16	139.6 d	7.31 br s
17	9.4 q	1.16 d (7)
18	167.8 s	
19	74.8 t	pro*R* 4.12 d (9), pro*S* 3.81 dd (9, 1.5)
20	97.9 d	5.69 s
CH_3CO	21.4 q	1.96 s
CH_3CO	169.1 s	

[a] SFORD multiplicities (20 MHz). [b] The assignments were confirmed by double resonance experiments. Chemical shifts in δ (J values in parentheses in Hz).

with the data described for similar functionality found in certain neoclerodane diterpenes isolated from *Teucrium* species.[8] The ^{13}C NMR spectrum of **5** also showed a singlet at δ 206.7 ascribed to the C-7 ketonic group.

The relative stereochemistry shown in **5** was deduced from the following evidence. Comparison of the ^{13}C NMR spectrum of **5** (Table II) with that of linearolactone, whose structure was established by X-ray diffraction analysis,[9] suggested an A/B cis ring fusion in **5**. The chemical shift and multiplicity found for H-12 (see Table II) is comparable with the data described for similar structures;[8] therefore, the configuration at this chiral center must be *S*. The ketalic H-20 observed at δ 5.69 in **5** is shifted upfield with respect to the chemical shift observed for this proton in teuvicentin[8] (δ 6.83, 20*S*); therefore, the configuration at C-20 in **5** must be *R*.

Experimental Section

The plant material of the first population was collected in Texcoco (Mexico), and a voucher specimen was deposited at the Herbarium of the Universidad de Chapingo. The second population was collected in Cuernavaca (Morelos, Mexico), and a voucher specimen was deposited at the Herbarium of the Instituto de Biologia (Universidad de Mexico).

The APT,[16] HETCOR,[18] and selective INEPT[19] spectra were obtained on a Varian XL-300 spectrometer (300 MHz for ^1H).

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The COSY[17] spectra were obtained on a Varian XL-400 spectrometer (400 MHz); the long-range coupling between protons were emphasized using additional delays of 100 ms before and after the second 90° pulse. The NOESY[21] and the $^{13}C-^{13}C$[20] correlation spectra were obtained on a General Electric GN-500 spectrometer (500 MHz). The mixing time used for the NOESY spectrum is 0.6 s.

Isolation of the Constituents of *Salvia tiliaefolia*, Vahl (Texcoco Population). Dried and powdered aerial parts of *Salvia tiliaefolia* (5 kg) were extracted with Me_2CO for 1 week at room temperature. The solvent was removed in vacuo to yield 100 g of a gummy residue which was subjected to partition between MeOH–H_2O (4:1) (A) and benzene–petroleum ether (1:1) (B). Chromatography of the nonpolar fraction (77 g) on silica gel[10] using benzene–petroleum ether gave sitosterol, with ethyl acetate/petroleum ether a mixture of oleanolic and ursolic acids identified by comparison of their methyl esters with authentic samples, and with ethyl acetate the β-glucoside of sitosterol.

The aqueous methanolic fraction (A) was concentrated in vacuo, water was added, and the mixture was extracted with CH_2Cl_2. The extract was dried and the solvent removed to yield 28 g of a gum. Treatment with acetone yielded 5 g of an amorphous solid which after purification was identified as the mixture of oleanolic and ursolic acids.

The acetone-soluble fraction was submitted to column chromatography on silica gel using CH_2Cl_2 and CH_2Cl_2–MeOH of increasing polarity as eluents. Elution with CH_2Cl_2–MeOH (99:1) gave a yellow crystalline product (125 mg), mp 223–225 °C, identified as the flavone xanthomycrol by comparison of its spectral data with those reported.[12]

Repeated chromatography of the fractions eluted with CH_2Cl_2–MeOH (49:1) yielded two products. The least polar compound (165 mg, mp 200–203 °C) was identified as isosalvipuberulin (1), by comparison with an authentic sample. The most polar compound was a solid, mp 164–165 °C, tilifodiolide (2), (300 mg, $[\alpha]_D$ −137° (*c* 0.21, $CHCl_3$); IR ($CHCl_3$) ν_{max} (cm^{-1}) 1770, 1650, 1600, 1500, 1480, 1450, 1075, 1020, 990, 960, 910, 873, 830; λ_{max} [nm (ε)] 205 (48000), 240 (12700), 275 (1500), 283 (1300); ^1H NMR, see Table I; MS *m/z* (rel intensity) 336 (M^+ 8.5) 268 (100), 222 (35), 165 (23.4), 152 (23); HR MS 337.1092 (calc for $C_{20}H_{17}O_5$: 337.107 598 9).

Catalytic Hydrogenation of 2. Compound **2** (100 mg) in EtOAc (5 mL) was hydrogenated with Pd/C (100 mg, 10%) as catalyst at room temperature and pressure for 36 h. After the usual workup, the product was purified by column chromatography to give the hexahydro derivative **3** (75 mg) as a noncrystalline substance: IR ($CHCl_3$) ν_{max} (cm^{-1}) 1759, 1600, 1479, 1161, 1077, 1025, 842, 757; ^1H NMR (80 MHz, $CDCl_3$) δ 7.25 (H-6, d, *J* = 8 Hz), 7.62 (H-7, d, *J* = 8 Hz).

Ozonolysis of Tilifodiolide. **2** (100 mg) in EtOAc (5 mL) was treated with ozone at −70 °C until saturation. The excess ozone was removed and the solvent distilled under vacuum at low temperature. The residue, in MeOH (5 mL), was treated with H_2O_2 (30%, 1 mL) and K_2CO_3 (0.2 g) in H_2O (0.5 mL) and boiled under reflux for 2 h. The solvent was removed at reduced pressure, water was added, and the reaction mixture was acidified and extracted with EtOAc. The extract was washed with brine and dried over Na_2SO_4; the solvent was evaporated. The product **4a** was esterified with ethereal diazomethane solution and the ester was purified by flash chromatography.[13] Elution with petroleum ether–EtOAc (7:3) gave **4b** as a crystalline mixture of epimers at C-3: mp 98–100 °C (CH_2Cl_2–petroleum ether) (40 mg); IR ($CHCl_3$) ν_{max} (cm^{-1}) 1775, 1745, 1600; ^1H NMR ($CDCl_3$, 80 MHz) δ 7.25 (H-6, d, *J* = 8 Hz), 7.65 (H-7, d, *J* = 8 Hz), 5.77 (H-12, s), 5.80 (H-12, s), 3.7, 3.75, 3.78, 3.80 (4s, 4 OCH_3); (benzene-d_6, 80 MHz) δ 6.6 (H-6, d, *J* 8 Hz), 7.45 (H-7, d, *J* = 8 Hz), 5.25 (H-12, s), 5.30 (H-12, s), 3.15, 3.18, 3.35, 3.38 (4 OCH_3); MS *m/z* (rel intensity) 304 (M^+ 15), 272 (5), 245 (41), 185 (100), 128 (37), 77 (15) ($C_{16}H_{16}O_6$ requires M^+ 304).

X-ray Analysis of Tilifodiolide. Suitable colorless crystals of tilifodiolide were prepared by slow evaporation of a methanol solution. A crystal with overall dimensions ca. 0.2 × 0.28 × 0.42 mm was used for data collection on a Nicolet P3/F diffractometer with Ni-filtered Cu Kα radiation (λ = 1.541 78 Å). Unit cell parameters determined from 25 reflections were *a* = 12.310 (2), *b* = 10.514 (2), *c* = 12.990 (3) Å; β = 97.34 (2); space group $P2_1$ (from systematic absences) with two formula units $C_{20}H_{16}O_5$ per asymmetric unit. Of 2239 independent reflections collected and corrected by Lp effect, 2113 showed intensities $I > 3\sigma(I)$ and were used for structure solution and refinement. No absorption correction was made, μ(Cu Kα) = 7.52 cm^{-1}.

The structure was solved by direct methods but not routinely. After several unsuccessful attempts at automatic solution, the "best one" among several starting sets was selected by hand and tested by its converge map for the first 50 strong reflections; it was used to start the MITHRIL[14] multisolution approach. The *E*-map for the first ranked solution based on CFOM yielded 40 of 50 expected atoms; the rest of atoms were found on a difference Fourier map.

The structure was refined by full-matrix least-squares with anisotropic temperature factors for the nonhydrogen atoms. The H atoms of tertiary CH and secondary CH_2 groups were assigned coordinates based on the expected geometry with an isotropic temperature factor 1.2 times that of the bearing atom. A weighting scheme based on $\sigma^2(I)$ was applied, and the usual discrepancy factors on the last cycle of refinement were $R = 0.037$, $R_w = 0.055$. All computations were performed on a Vax-Station II with TEXSAN[15] system of programs.

Isolation of Constituents from *Salvia tiliaefolia* (Cuernavaca Population). Ground and dried aerial parts of a second population of *Salvia tiliaefolia* (830 g) were treated as above. Chromatography over silica gel of the benzene–petroleum ether soluble fraction (27 g) yielded sitosterol (1 g) and the mixture of oleanolic and ursolic acids (2.44 g).

The CH_2Cl_2-soluble portion of the methanolic fraction was chromatographed over silica gel using mixtures of petroleum–EtOAc of increasing polarity. From the fractions eluted with petroleum ether–EtOAc (4:1), isosalvipuberulin (**1**) (6.4 mg, 7 × 10^{-4}% yield) and xanthomycrol (46 mg) were isolated.

Elution with petroleum ether–EtOAc (7:3) yielded, after purification, tilifodiolide (**2**) (3.6 g, 0.42% yield) and salvifolin (**5**) (129 mg, 15 × 10^{-3}% yield), $[\alpha]_D$ +27.1° (*c* 0.229, $CHCl_3$), mp 220–223 °C (Me_2CO–petroleum ether); IR ($CHCl_3$) ν_{max} (cm^{-1}) 1755, 1716, 1668, 1596, 1504, 875; UV λ_{max} [nm (ε)] 215 (11 000), 293 (5000); ^1H NMR, see Table II; ^{13}C NMR, see Table II; MS *m/z* (rel intensity) 356 (M^+ − 42,19), 338 (12), 267 (15), 185 (20), 108 (59), 94 (86), 81 (53), 43 (100); CI MS 399 (M^+ + 1) ($C_{22}H_{22}O_7$ requires M^+ at *m/z* 398).

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Supplementary Material Available: Tables of atomic coordinates and thermal parameters and bond distances and angles for molecules A and B of tilifodiolide (12 pages); table of observed and calculated structure factors for tilifodiolide (15 pages). Ordering information is given on any current masthead page.

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