Four New Compounds from the Leaves of *Acer truncatum*

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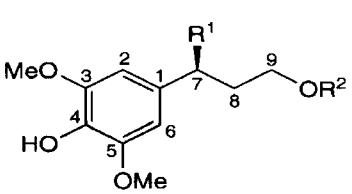
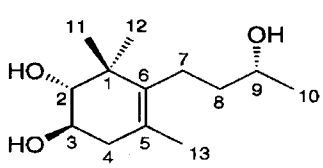
Four new compounds, 3-(4-hydroxy-3,5-dimethoxyphenyl)propyl formate (**1**), 2,6-dimethoxy-4-[(1*S*)-3-methoxypropyl]phenol (**2**), (1*R*,2*R*)-4-[(3*R*)-3-hydroxybutyl]-3,3,5-trimethylcyclohex-4-ene-1,2-diol (**3**), and (1*S*,3*R*,3a*R*,6*S*,7*S*,9a*R*)-decahydro-1-(hydroxymethyl)-1,7-dimethyl-3a,7-methano-3a*H*-cyclopentacyclooctene (**4**) were isolated from the leaves of *Acer truncatum*, together with twelve known compounds. Their structures were elucidated on the basis of extensive spectroscopic techniques. The absolute configuration of compound **3** was established by the modified *Mosher*'s method. All compounds were evaluated for antibacterial activities.

1. Introduction. – The plants of genus *Acer*, which belongs to the family of the Aceraceae and comprises over 200 species, are mainly distributed in the north temperate zone such as China and Japan. Particularly, there are over 150 species in China [1]. Chemical studies have revealed that the genus *Acer* mainly contained flavonoids, lignans, cyclic diarylheptanoids, and phenolic glycosides [2–6]. The roots of *Acer truncatum* have been used as folk medicine to treat lumbago, and its leaves have been used as sanitarian tea [7]. In our previous paper, we reported six flavonoid glycosides, which showed strong activity in thrombus [8]. We continued to investigate the chemical constituents of this plant and isolated four new compounds including two phenylpropanoids, one megastigmane, and one sesquiterpene. Here, we describe the isolation and structure elucidation of the four new compounds, **1**–**4**, besides twelve known compounds. At the same time, we studied the antibacterial activity against *Escherichia coli* (YMF3.0016), *Staphylococcus aureus* (YMF3.0017), *Micrococcus luteus* (YMF3.0018), and *Bacillus cereus* (YMF3.0019).



1 R^1 = H, R^2 = CHO
2 R^1 = MeO, R^2 = H



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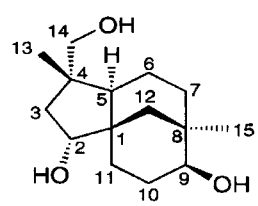

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2. Results and Discussion. – *Structure Elucidation*[1]). Compound **1**, a colorless gum, was determined to have the molecular formula $C_{12}H_{16}O_5$, which was based on HR-ESI-MS (*m/z* 263.0897 ($[M + Na]^+$; calc. 263.0895), corresponding to five degrees of unsaturation. The IR spectrum showed absorption bands at 3432 and 1115 cm^{-1} for a OH group, 1720 cm^{-1} for a COO group, 1613, 1519, and 1461 cm^{-1} for a benzene group. The ^{13}C-NMR (including DEPT) spectrum of **1** (*Table 1*) showed the signals of two MeO groups, three CH_2 (including one oxygenated CH_2), one CHO group, together with six aromatic C-atoms, indicating a 3-phenylpropanol skeleton. Comparison of the ^1H- and ^{13}C-NMR spectrum data of **1** with those of dihydrosyringenin (**10**) [9] indicated the same skeleton except the presence of a CHO group in **1**. In the HMBC experiment, the correlation of H (δ(H) 8.11 (*s*)) with C(9) (δ(C) 63.2 (*t*)) suggested that the ester of formic acid was located at C(9). Hence, on the basis of above evidence, the structure of **1** was established as 3-(4-hydroxy-3,5-dimethoxyphenyl)propyl formate (**1**).

Table 1. *^1H-* (400 MHz) *and ^{13}C-NMR* (100 MHz) *Data of Compounds* **1** *and* **2**. In $CDCl_3$; δ in ppm, *J* in Hz.

	1		**2**	
	δ(C)	δ(H)	δ(C)	δ(H)
C(1)	132.0 (*s*)	–	132.7 (*s*)	–
H–C(2)	105.1 (*d*)	6.42 (*s*)	103.1 (*d*)	6.54 (*s*)
C(3)	147.1 (*s*)	–	147.2 (*s*)	–
C(4)	133.2 (*s*)	–	134.3 (*s*)	–
C(5)	147.1 (*s*)	–	147.2 (*s*)	–
H–C(6)	105.1 (*d*)	6.42 (*s*)	103.1 (*d*)	6.54 (*s*)
CH_2(7) or H–C(7)	32.2 (*t*)	2.65 (*t*, $J = 7.9$)	84.0 (*d*)	4.28 (*dd*, $J = 4.0, 9.0$)
CH_2(8)	29.7 (*t*)	1.99 (*td*, $J = 6.5, 7.9$)	40.6 (*t*)	1.82–1.86, 2.03–2.06 (2*m*)
CH_2(9)	63.2 (*t*)	4.21 (*t*, $J = 6.5$)	61.2 (*t*)	3.78 (*t*, $J = 6.0$)
MeO–C(3,5)	56.3 (*q*)	3.90 (*s*)	56.4 (*q*)	3.90 (*s*)
MeO–C(7)	–	–	56.5 (*q*)	3.23 (*s*)
H–C=O	161.0 (*s*)	8.11 (*s*)	–	–

Compound **2**, a colorless gum, had the molecular formula $C_{12}H_{18}O_5$, as derived from HR-ESI-MS (*m/z* 265.1057 ($[M + Na]^+$; calc. 265.1051). A comparison of the ^1H- and ^{13}C-NMR spectral data (*Table 1*) of **2** with those of **1** indicated that **2** had the same skeleton as **1**, except for the presence of a MeO group and the disappearance of the CHO group. The $^1H,^1H$–COSY correlations of H–C(8) (δ(H) 1.82–1.86, 2.03–2.06 (2*m*)) with H–C(7) (δ(H) 4.28 (*dd*, $J = 4.0, 9.0$)), and H–C(9) (δ(H) 3.78 (*t*, $J = 6.0$)) suggested the following fragment: CH(7)–CH_2(8)–CH_2(9)OH. In the HMBC experiment, the H-atom of the MeO group at δ(H) 3.32 (*s*) coupled with C(7) at δ(C) 84.0, suggesting that the MeO group (δ(C) 56.5) was located at C(7). The correlations between H–C(2)/H–C(6) (δ(H) 6.54 (*s*)) and C(1), C(3), C(4), C(5), C(7), two MeO (δ(H) 3.90 (*s*)) and C(3), C(5)) were correlated. So, the other two MeO groups were at C(3) and C(5).

[1]) For the discussion and in the *Tables*, an arbitrary C-atom numbering, shown in the Formulae **1**–**4**, has been used. For systematic names, *cf.* the *Exper. Part.*

The absolute configuration at C(7) was confirmed as (*S*) by comparing the optical rotations of **2** (−6.8) with (+)-(*R*)-4-(1-hydroxy-1,5-dimethyl-3-oxohex-4-enyl)benzaldehyde (+32.6) [10], and (−)-(10′*S*)-3-(10-hydroxyphenyldecyl)phenol (−8.3) [11]. Therefore, compound **2** was identified as 2,6-dimethoxy-4-[(1*S*)-3-methoxypropyl]phenol (**2**).

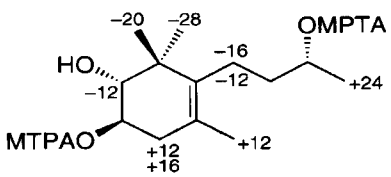
Compound **3** was obtained as a colorless gum. Its molecular formula was deduced as $C_{13}H_{24}O_3$ by HR-ESI-MS (*m/z* 251.1626 ($[M + Na]^+$; calc. 251.1623) and ^{13}C-NMR. The ^{1}H- and ^{13}C-NMR spectra of **3** (*Table 2*) indicated 13 C-atoms with 21 directly attached H-atoms, which consisted of four Me, three CH_2, three CH groups with O-functions, one quaternary C-atom (δ(C) 41.7 (C(1))), and a tetrasubstituted C=C bond. These implied a megastigmane skeleton for **3**. A tetrasubstituted C=C bond in a megastigmane skeleton was only possible between the C(5) and C(6). In the ^{1}H,^{1}H-COSY spectrum, the cross-peaks between H–C(3) and H–C(2), and H_a–CH(4) and H_b–CH(4) confirmed the presence of CH(2)–CH(3)–CH_2(4) groups. The CH_2(7)–CH_2(8)–CH(9)–Me(10) moiety was derived from the following ^{1}H,^{1}H-COSY correlations: H–C(7)/H–C(8)/H–C(9)/Me(10). Furthermore, three oxygenated CH groups were corresponding to three OH groups in compound **3**. In the HMBC experiment (*Table 2*), the correlations of H–C(2) with C(1), C(3), C(4), C(11), C(12), H–C(3) with C(1), C(2), C(4), and H–C(9) with C(7), C(8) suggested that one OH group was at C(9), and the other two OH groups were at C(2) and C(3).

Table 2. *^{1}H-* (400 MHz) *and ^{13}C-NMR* (100 MHz) *Data, Including COSY and HMBC* (500 MHz) *Data of Compound* **3**. In $CDCl_3$; δ in ppm, *J* in Hz.

	δ(C)	δ(H)	COSY (^{1}H,^{1}H)	HMBC (^{1}H,^{13}C)
C(1)	41.7 (*s*)	–	–	–
H–C(2)	80.1 (*d*)	3.23 (br. *d*, *J* = 10.2)	H–C(3)	C(1), C(3), C(4), C(11), C(12)
H–C(3)	67.8 (*d*)	3.70–3.75 (*m*)	H–C(2), H_a–C(4), H_b–C(4)	C(1), C(2), C(4)
H_a–C(4)	39.5 (*t*)	2.00–2.07 (*m*)	H–C(3), H_b–C(4)	C(3), C(5), C(6)
H_b–C(4)		2.28 (*dd*, *J* = 6.4, 16.7)	H–C(3), H_a–C(4)	C(2), C(3), C(5), C(6), C(13)
C(5)	123.4 (*s*)	–	–	–
C(6)	136.5 (*s*)	–	–	–
H_a–C(7)	24.7 (*t*)	1.83–1.91 (*m*)	H_b–C(7), H–C(8)	C(1), C(5), C(6)
H_b–C(7)		2.13–2.19 (*m*)	H_a–C(7), H–C(8), H–C(9)	C(5), C(6), C(8)
CH_2(8)	39.7 (*t*)	1.43–1.55 (*m*)	H_a–C(7), H_b–C(7), H–C(9)	C(7), C(9)
H–C(9)	68.6 (*d*)	3.77–3.82 (*m*)	H–C(8), Me(10)	C(7), C(8)
Me(10)	23.2 (*q*)	1.21 (*d*, *J* = 6.2)	H–C(9)	C(8), C(9)
Me(11)	25.4 (*q*)	1.09 (*s*)	–	C(1), C(2), C(6), C(12)
Me(12)	21.4 (*q*)	0.92 (*s*)	–	C(1), C(2), C(6), C(11)
Me(13)	19.4 (*q*)	1.59 (*s*)	–	C(4), C(5), C(6)

To determine the absolute configuration at the stereogenic center in compound **3**, the modified *Mosher*'s method was applied [12]. The (*S*)-MTPA (MTPA = α-methoxy-α-(trifluoromethyl)phenylacetic acid) esters **3a** and (*R*)-MTPA esters **3b** of **3** were prepared by the procedure described in the *Exper. Part.* In the ^{1}H-NMR spectra of **3a** and **3b**, two MeO signals and ten signals for aromatic H-atoms were observed. Comparing the ^{1}H-NMR-spectral data with those of **3**, H–C(3), H–C(9) were shifted

downfield ($\Delta\delta \approx 1.4$ ppm), but H–C(2) was slightly changed, suggesting that the two *O*-MTPA moieties were at H–C(3) and H–C(9), respectively. Presumably, there is a higher steric hindrance of 2-OH, so the 2-*O*-MTPA ester of **3** was not formed under the esterification conditions. In accordance with $\Delta\delta = \delta(S) - \delta(R)$ (at 400 MHz, in Hz; *Fig. 1*), both C(3) and C(9) were (*R*)-configured. The absolute configuration at C(2) was confirmed as (*R*) by the ROESY spectrum (*Fig. 2*), displaying the correlations between H–C(3) and Me(12) (δ(C) 21.4 (*q*)), H–C(2), and Me(11) (δ(C) 25.4 (*q*)). In conclusion, the structure of **3** was elucidated as (1*R*,2*R*)-4-[(3*R*)-3-hydroxybutyl]-3,3,5-trimethylcyclohex-4-ene-1,2-diol.



3,9-di-*O*-(*S*)-MTPA ester **3a**
3,9-di-*O*-(*R*)-MTPA ester **3b**

Fig. 1. *Results with the modified* Mosher's *method for* **3**. $\Delta\delta$ ($=\delta(S) - \delta(R)$) in Hz (400 MHz).

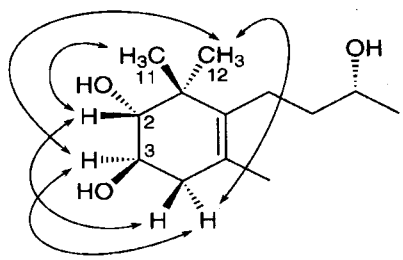


Fig. 2. *Key ROESY correlations observed in* **3**

Compound **4**, colorless needles, has a molecular formula of $C_{15}H_{26}O_3$ determined by HR-ESI-MS (*m/z* 277.1783 $[M + Na]^+$; calc. 277.1779). The IR spectroscopy showed a strong band at 3331 cm^{-1}, indicating OH groups. The signals of C-atoms found in ^{13}C-NMR and DEPT spectra of **4** included three oxygenated C-atoms (δ(C) 80.4 (*d*), 74.5 (*d*), 71.9 (*t*)), along with two Me, six CH_2, one CH groups, and three quaternary C-atoms (*Table 3*). The ^{1}H- and ^{13}C-NMR data indicated that **4** was very similar to (–)-clovane-2,9-diol [13], except for the appearance of one oxygenated CH_2 C-atom (δ(C) 71.9 (*t*)) and disappearance of one Me group. The HMBC correlations (*Table 3*) were observed between H–C(2) and C(1), C(3), C(4), C(5), C(11), C(12), H–C(9) and C(8), C(10), C(11), C(12), C(15), H–C(14) and C(3), C(4), C(5), C(13), in conjunction with the the ^{1}H,^{1}H–COSY correlations: H–C(2)/H–C(3), H–C(5)/H–C(6)/H–C(7), H–C(10)/H–C(11), which established the structure of compound **4** as clovane-2,9,14-triol. The relative configuration of **4** was assumed to be the same as (–)-clovane-2,9-diol on the basis of the following key ROESY correlations (*Fig. 3*): H–C(2)/Me(13), H–C(5)/H–C(14), H–C(5)/H–C(9), H–C(5)/H–C(15), H–C(9)/H–C(15). A comparison of optical rotation of **4** (– 3.9) and (–)-clovane-2,9-diol ((1*R*,2*R*,5*R*,8*S*,9*S*); (– 3.5) [13]) strongly suggested that the absolute configuration of **4** was the same as (–)-clovane-2,9-diol (*i.e.*, (1*R*,2*R*,5*R*,8*S*,9*S*)). Therefore, compound **4** was elucidated as (–)-(1*R*,2*R*,4*S*,5*R*,8*S*,9*S*)-clovane-2,9,14-triol (=

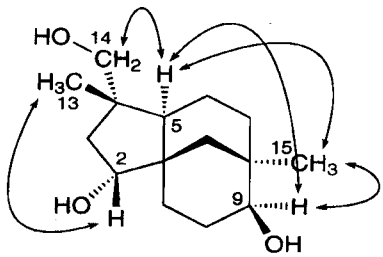


Fig. 3. *Key ROESY correlations observed in* **4**

Table 3. *^1H-* (400 MHz) *and ^{13}C-NMR* (100 MHz) *Data, Including COSY and HMBC* (500 MHz) *Data of Compound* **4**. In (D_5)pyridine; δ in ppm, J in Hz.

	δ(C)	δ(H)	COSY (^{1}H,^{1}H)	HMBC (^{1}H,^{13}C)
C(1)	46.3 (*s*)	–	–	–
H–C(2)	80.4 (*d*)	4.13 (*t*, *J*=6.4)	H_a–C(3), H_b–C(3)	C(1), C(3), C(4), C(5), C(11), C(12)
H_a–C(3)	44.0 (*t*)	2.00 (*d*, *J*=5.5)	H–C(2), H_b–C(3)	C(1), C(2), C(4), C(13), C(14)
H_b–C(3)		2.21 (*d*, *J*=4.8)	H–C(2), H_a–C(3)	C(1), C(2), C(4), C(5), C(13), C(14)
C(4)	44.3 (*s*)	–	–	–
H–C(5)	44.9 (*d*)	2.14–2.15 (*m*)	–	C(1), C(4), C(6), C(11), C(13), C(14)
H_a–C(6)	22.3 (*t*)	1.51–1.56 (*m*)	H_b–C(6), H_a–C(7), H_b–C(7)	C(4), C(5), C(7)
H_b–C(6)		1.58–1.68 (*m*)	H_a–C(6), H_a–C(7), H_b–C(7)	C(1), C(4), C(7), C(8)
H_a–C(7)	34.5 (*t*)	1.23–1.29 (*m*)	Ha–C(6), H_b–C(6), H_b–C(7)	C(5), C(8), C(9), C(15)
H_b–C(7)		1.39–1.44 (*m*)	H_a–C(6), H_b–C(6), H_a–C(7)	C(5), C(6), C(8), C(9), C(15)
C(8)	35.6 (*s*)	–	–	–
H–C(9)	74.5 (*d*)	3.58 (br. *s*)		C(8), C(10), C(11), C(12), C(15)
H_a–C(10)	27.6 (*t*)	2.37–2.43 (*m*)	H_b–C(10), H_b–C(11)	C(1), C(9), C(12)
H_b–C(10)		1.17 (br. *d*, *J* = 5.4)	H_a–C(10), H_a–C(11), H_b–C(11)	C(1), C(9), C(11), C(12)
H_a–C(11)	28.4 (*t*)	1.98 (*d*, *J* = 5.4)	H_b–C(10), H_b–C(11)	C(2), C(12)
H_b–C(11)		2.17–2.19 (*m*)	H_a–C(10),H_b–C(10), H_a–C(11), H_b–C(11)	C(1), C(2), C(5), C(10), C(12)
H_a–C(12)	36.7 (*t*)	1.09 (br. *s*)	H_b–C(11), H_b–C(12)	C(1), C(2), C(8), C(9), C(11), C(15)
H_b–C(12)		2.11 (*d*, *J* = 7.1)	H_a–C(11), H_b–C(11), H_a–C(12)	C(1), C(5), C(7), C(8), C(9), C(11), C(15)
Me(13)	21.9 (*q*)	1.12 (*s*)	–	C(3), C(4), C(5), C(14)
CH_2(14)	71.9 (*t*)	3.67 (*dd*, *J* = 3.7, 8.2)	–	C(3), C(4), C(5), C(13)
Me(15)	29.6 (*q*)	1.12 (*s*)	–	C(7), C(8), C(9), C(12)

(1*S*,3*R*,3a*R*,6*S*,7*S*,9a*R*)-decahydro-1-(hydroxymethyl)-1,7-dimethyl-3a,7-methano-3a*H*-cyclopentacyclooctene.

Together with compounds **1**–**4**, the following known constituents were isolated from the leaves of *A. truncatum:* myrsinionoside E (**5**) [14], 1β-D-glucopyranosyloxy-6α-hydroxyeudesman-4(15)-ene (**6**) [15], gallic acid (**7**) [16], ethyl 3,4,5-trihydroxybenzoate (**8**) [17], ω-hydroxypropioguaiacone (**9**) [18], dihydrosyringenin (**10**) [9], 2-[4-(3-hydroxypropyl)-2-methoxyphenoxy]propane-1,3-diol (**11**) [19], 4-(3-hydroxypropyl)-2-methoxyphenol (**12**) [20], (3,4,5-trimethoxyphenyl)methanol (**13**) [21], 2-(3-methoxy-4-hydroxyphenyl)propane-1,3-diol (**14**) [22], icariside B_6 (**15**) [23], and linarionoside A (**16**) [24].

Antibacterial Assays. Escherichia coli (YMF 3.0016), *Staphylococcus aureus* (YMF 3.0017), *Micrococcus luteus* (YMF 3.0018), and *Bacillus cereus* (YMF 3.0019) were used. All stock cultures were grown on tryptic soy agar plates. The test strain was transferred to fresh tryptic soy broth before use, and a disk containing only DMSO was used as negative control.

All of the compounds, **1**–**16**, were tested for their antibacterial activities against *E. coli*, *S. aureus*, *M. luteus*, and *B. cereus* using the paper-disk method; except gallic acid (**7**) and icariside B_6 (**15**), all compounds were found to be inactive at concentrations up to 50 μg/disk. Gallic acid (**7**) at 30 μg/disk afforded inhibitory zone sizes of 10 mm against *Saccharomyces cerevisiae.* Icariside B_6 (**15**) showed moderate antibacterial activity at 24 μg/disk in standard *Petri*-plate assays, affording inhibitory zone sizes of 14 and 17 mm against *Bacillus cereus* and *Micrococcus luteus*, respectively.

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Experimental Part

General. (+)-(*R*) and (−)-(*S*)-α-methoxy-α-(trifluoromethyl)phenylacetic acids (MTPA) were from *Aldrich.* Column chromatography (CC): silica gel (200–300 mesh; *Qingdao Marine Chemicals Co. Ltd.*), *Sephadex LH-20* gel (*Amershan Biosciences*, Sweden). MPLC: *Büchi Pump Module C-605*, *Büchi Pump Manager C-615*, *Büchi Fraction Collector C-660.* M.p. *Yuhua 104* melting-point apparatus (uncorrected). Optical rotations: *JASCO DIP-370* digital polarimeter. UV: *UV-210A* spectrophotometer; λ_{max} in nm (log ε). IR: *Bio-Rad FTS-135*; in cm^{-1}. NMR: 1D-NMR: *Bruker AM-400*; 2D-NMR: *Bruker DRX-500*; δ in ppm, *J* in Hz. MS: *VG Autospec-3000* (70 eV for EI); in *m/z* (rel. int.). HR-ESI-MS: *API Qstar pulsa.*

Plant Material. The leaves of *A. truncatum* were collected in Kunming, Yunnan province, P. R. China, in August 2004. The plants were identified by Prof. *T.-Z. Xu*, Kunming Institute of Botany, Chinese Academy of Science.

Extraction and Islotion. Dried leaves of *A. truncatum* were extracted with H_2O. The extract was evaporated *in vacuo* to give a black-brown gum, which was subjected to *ADS-7* porous resin divided into four fractions: H_2O fraction, 30% EtOH fraction, 70% EtOH fraction, and 90% EtOH fraction. Evaporated 30% EtOH fraction (256 g) was subjected to CC (SiO_2; $CHCl_3$/MeOH 9:1 → 7:3) to afford ten fractions (*Fr. 1–10*) as judged by TLC. *Fr. 2* (20 g) was further purified by CC (1. SiO_2; petroleum ether/AcOEt; 2. *RP-18* gel) to afford **1** (10 mg), **2** (10 mg), **5** (43 mg), **9** (16 mg), **10** (210 mg), **12** (156 mg), and **13** (98 mg). *Fr. 4* (18 g) was subjected to CC (SiO_2; petroleum ether/AcOEt) to yield **3** (78 mg) and **8** (100 mg). *Fr. 5* (19 g) was subjected to CC (SiO_2; $CHCl_3$/AcOEt) to yield **11** (13 mg) and **14** (10 mg), *Fr. 7* (23 g) was subjected to CC (SiO_2; petroleum ether/AcOEt) to afford **6** (62 mg), **15** (14 mg), and **16** (27 mg). *Fr. 8* (20 g) was subjected to CC (1. SiO_2; $CHCl_3$/MeOH 2. *RP-18* gel) to afford **4** (171 mg). *Fr. 10* (300 mg) was subjected to CC (SiO_2; $CHCl_3$/MeOH) to afford **7** (70 mg).

Synthesis of (S)- and (R)-MTPA Esters of **3**. Dicyclohexylcarbodiimide (DCC; 30 mg), 4-(dimethylamino)pyridine (DMAP; 15 mg), and (*S*)- or (*R*)-MTPA (42 mg) were added to a soln. of **3** (3 mg) in THF (1 ml). The mixture was stirred at 25° for 24 h and then washed successively, with 0.5M HCl, sat. $NaHCO_3$ soln., and H_2O, and dried (Na_2SO_4). After removal of the solvent by evaporation, the residue was purified by CC (silica gel; petroleum ether/AcOEt 10:1) to afford the (*S*)-MTPA ester **3a** (2.3 mg) and (*R*)-MTPA ester **3b** (3 mg).

3-(4-Hydroxy-3,5-dimethoxyphenyl)propyl Formate (**1**). Colorless gum. UV ($CHCl_3$): 240 (4.1), 272 (3.5), 355 (2.59). IR: 3432, 2927, 2853, 1720, 1613, 1519, 1461, 1428, 1115. ^{1}H- and ^{13}C-NMR: see *Table 1*. EI-MS: 240 (64, M^+), 212 (6), 194(16), 182 (3), 168 (52), 167 (100), 123 (16). HR-ESI-MS: 263.0897 ($[M + Na]^+$, $C_{12}H_{16}NaO_5^+$; calc. 263.0895).

4-[(1S)-3-Hydroxy-1-methoxypropyl]-2,6-dimethoxyphenol (**2**). Colorless gum. $[\alpha]_D^{25} = -6.8$ ($c = 0.050$, $CHCl_3$). UV ($CHCl_3$): 220 (3.5), 241 (4.0), 271 (3.5). IR: 3440, 2924, 2854, 1617, 1519, 1462, 1428, 1114. ^{1}H- and ^{13}C-NMR: see *Table 1*. EI-MS: 242 (17, M^+), 198 (14), 197 (100), 182 (24), 151 (9), 97 (17), 71 (18). HR-ESI-MS: 265.1057 ($[M + Na]^+$, $C_{12}H_{18}NaO_5^+$; calc. 265.1051).

(1R,2R)-4-[(3R)-3-Hydroxybutyl]-3,3,5-trimethylcyclohex-4-ene-1,2-diol (**3**). Colorless gum. $[\alpha]_D^{25} = -66.3$ ($c = 0.056$, $CHCl_3$). IR: 3425, 2969, 2932, 2877, 1712, 1639, 1048. ^{1}H-and ^{13}C-NMR: see *Table 2*. EI-MS: 228 (6, M^+), 210 (5), 192 (34), 153 (39), 135 (100), 121 (73), 109 (98), 107 (86). HR-ESI-MS: 251.1626 ($[M + Na]^+$; $C_{13}H_{24}NaO_3^+$; calc. 251.1623).

(1R,2R)-2-Hydroxy-4-[(3R)-3-[(S)-α-methoxy-α-(trifluoromethyl)phenylacetoxy]butyl]-3,3,5-trimethylcyclohex-4-enyl (S)-α-Methoxy-α-(trifluoromethyl)phenylacetate (**3a**). Amorphous power. ^{1}H-NMR($CDCl_3$)[2]): 0.94 (*s*, Me(12)); 1.02 (*s*, Me(11)); 1.36 (*d*, J=6.2, Me(10)); 1.57 (*s*, Me(13)); 1.79–1.87 (*m*, H_a–C(7)); 1.99–2.08 (*m*, H_a–C(4)); 2.13–2.19 (*m*, H_b–C(7); 2.51 (*dd*, $J = 6.4$, 16.7, H_b–C(4)); 3.46 (*d*, $J = 10.2$, H–C(2)); 3.58 (*s*, 2 MeO); 5.10–5.13 (*m*, H–C(3)); 5.14–5.15 (*m*, H–C(9)); 7.38–7.42 (*m*, 6 arom. H); 7.54–7.55 (*m*, 4 arom. H). FAB-MS (pos.): 661 ($[M+1]^+$).

(1R,2R)-2-Hydroxy-4-[(3R)-3-[(R)-α-methoxy-α-(trifluoromethyl)phenylacetoxy]butyl]-3,3,5-trimethylcyclohex-4-enyl (R)-α-Methoxy-α-(trifluoromethyl)phenylacetate (**3b**). Amorphous power. ^{1}H-NMR ($CDCl_3$)[2]): 0.99 (*s*, Me(12)); 1.09 (*s*, Me(12)); 1.30 (*d*, J=6.2, Me(10)); 1.54 (*s*, Me(13)); 1.84–1.91 (*m*, H_a–C(7)); 1.97–2.05 (*m*, H_a–C(4)); 2.16–2.22 (*m*, H_b–C(7); 2.47 (*dd*, $J = 6.4$, 16.7, H_b–C(4)); 3.49 (*d*, $J = 10.2$, H–C(2)); 3.53 (*s*, MeO), 3.58 (*s*, MeO); 5.11–5.13 (*m*, H–C(3)); 5.14–5.15 (*m*, H–C(9)); 7.41–7.43 (*m*, 6 arom. H); 7.52–7.56 (*m*, 4 arom. H). FAB-MS (pos.): 661 ($[M+1]^+$).

(–)-(1S,3R,3aR,6S,7S,9aR)-Decahydro-1-(hydroxymethyl)-1,7-dimethyl-3a,7-dimethano-3aH-cyclopentacyclooctene (**4**). Colorless needles (pyridine). M.p. 209–210°. $[\alpha]_D^{25} = -3.9$ ($c = 0.052$, MeOH). IR: 3331, 2986, 2945, 2863, 1038. ^{1}H- and ^{13}C-NMR: see *Table 3*. EI-MS: 254 (2, M^+), 236 (4), 218 (6), 205 (31), 187 (36), 147 (100), 95 (29), 93 (36). FAB-MS: 255 ($[M + Na]^+$). HR-ESI-MS: 277.1783 ($[M + Na]^+$, $C_{15}H_{26}NaO_3^+$; calc. 277.1779).

Antibacterial Activity. Antibacterial activity was detected by the disk-diffusion method with minor modifications [25]. *E. coli*, *S. aureus*, and *M. luteus*, *B. cereus* were subcultured in tryptic soy broth (TSB) and incubated for 18 h at 37°, and then the bacterial cells were suspended, according to the *McFarland* protocol, in saline soln. to produce a suspension of *ca.* 10^{-5} CFU ml^{-1}. This suspension (15 µl) was mixed with 15 ml of sterile tryptic soy agar (TSA) at 40° and poured onto an agar plate in a laminar flow cabinet. Each test compound was dissolved in DMSO and added to a paper disk (6-mm diameter) that was dried and placed on the agar plate containing the bacterial cells (5 samples/disk plus control). A disk containing only DMSO was used as negative control. The susceptibility of the bacteria to the test compounds was determined by the formation of an inhibitory zone after 18 h of incubation at 37°. Experiments were run in triplicate, and the results are presented as mean values of the three measurements.

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[2]) C-Atom numbering as shown in formula **3**.

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