New Sesquiterpenoids from *Lycianthes marlipoensis*

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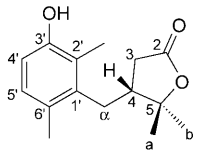
Two new sesquiterpenoids and one derivative, lycifuranone A (= (4*R*)-4,5-dihydro-4-(3-hydroxy-2,6-dimethylbenzyl)-5,5-dimethylfuran-2(3*H*)-one; **1**), lycifuranone B (= 4,5-dihydroxy-3-methyl-2-{[(3*R*)-tetrahydro-2,2-dimethyl-5-oxofuran-3-yl]methyl} benzaldehyde; **2**), and lycifuranone C (= (4*R*)-4-(3,4-dihydroxy-6-{(2*S*,4*R*,6*S*)-4-[2-(4-hydroxy-3-methoxyphenyl)ethyl]-6-pentyl[1,3]dioxan-2-yl}-2-methylbenzyl)-4,5-dihydro-5,5-dimethylfuran-2(3*H*)-one; **3**), respectively, have been isolated from the roots of *Lycianthes marlipoensis*, and their structures were established by spectroscopic methods.

Introduction. – The genus *Lycianthes* (Solanaceae) comprises *ca.* 180 species, nine species of which occur in South China. It is used as a treatment for swelling and rabies in some local areas because of its ability to eliminate toxins [1]. Isolation of steroid alkaloids and sapogenins from *Lycianthes biflora* has been reported [2][3]. Earlier, we also described research on the constituents of *L. biflora* [4]. However, there has been no report of any chemical investigation of *L. marlipoensis.* In this paper, we report the isolation and structural elucidation of two new sesquiterpenoids, **1** and **2**, and compound **3**, an acetal derivative of **2**, from *L. marlipoensis.* These compounds were named lycifuranone A, lycifuranone B, and lycifuranone C, respectively, and each of them contains a functionalized benzyl subunit, which occurs rarely in sesquiterpenoids.



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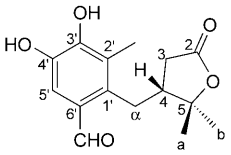

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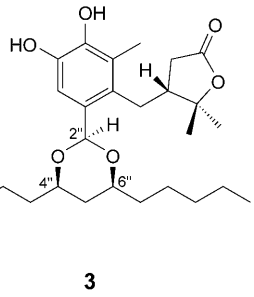

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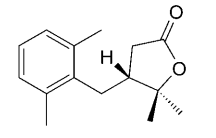

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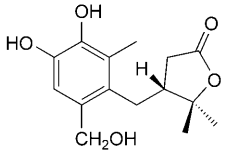

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[1]) Arbitrary numbering for compound **2**.

Results and Discussion. – Lycifuranone A (**1**) was obtained as a white amorphous powder. Its EI-HR-MS exhibited the molecular-ion peak M^+ at m/z 248.1415, corresponding to the molecular formula $C_{15}H_{20}O_3$. In the IR spectrum, absorption bands at 3517 (OH), 1591 (aromatic ring), and 1716 cm^{-1} (γ-lactone) were apparent. The ^{1}H-NMR (*Table 1*), ^{13}C-NMR (*Table 2*), and HSQC and HMBC (*Fig.*) data, and their comparison with those of the analogous solafuranone (= *(+)-(*R*)-4,5-dihydro-5,5-dimethyl-4-(2,6-dimethylbenzyl)furan-2(3*H*)-one*; **4**) isolated from *Solanum indicum* (Solanaceae) [5] led to the unambiguous assignment of all H- and C-atoms. Thus, the structure of **1** was concluded to be *4,5-dihydro-4-(3-hydroxy-2,6-dimethylbenzyl)-5,5-dimethylfuran-2(3*H*)-one*.

Table 1. *^{1}H-NMR Data of* **1** *and* **2**[1]). δ in ppm, J in Hz.

	1		**2**
	$CDCl_3$, 400 MHz	$(CD_3)_2CO$, 500 MHz	CD_3OD, 400 MHz
CH_2(3)	2.34–2.36 (*m*), 2.42[a])	2.25[b]), 2.55–2.59 (*m*)	2.16 (*dd*, *J* = 16.5, 7.2) 2.57 (*dd*, *J* = 16.5, 11.4)
H–C(4)	2.44[a])	2.50–2.54 (*m*)	2.46 (*ddd*, *J* = 11.4, 7.2, 3.7)
$CH_2(\alpha)$	2.72–2.75 (*m*)	2.77 (*dd*, *J* = 13.7, 11.0), 2.84 (*dd*, *J* = 13.7, 3.1)	3.10 (br. *d*, *J* = 13), 3.38 (br. *d*, *J* = 13)
Me_a–C(5)	1.43 (*s*)	1.45 (*s*)	1.44 (*s*)
Me_b–C(5)	1.56 (*s*)	1.56 (*s*)	1.55 (*s*)
H–C(4′)	6.59 (*d*, *J* = 8.1)	6.67 (*d*, *J* = 8.1)	
H–C(5′)	6.87 (*d*, *J* = 8.1)	6.82 (*d*, *J* = 8.1)	7.14 (*s*)
Me–C(2′)	2.20 (*s*)	2.20 (*s*)	2.23 (*s*)
Me–C(6′) or H–C(7′)	2.24 (*s*)	2.25 (*s*)[b])	9.88 (*s*)

[a]), [b]) Overlapped.

Table 2. *^{13}C-NMR Data of* **1**–**5**[1]). δ in ppm.

	1 $CDCl_3$, 100 MHz	**2** CD_3OD, 100 MHz	**3**[a]) $CDCl_3$, 100 MHz	**4**[b]) $(CD_3)_2CO$, 125 MHz	**5** CD_3OD, 100 MHz
C(2)	176.0	178.9	176.0	175.2	179.1
C(3)	34.3	35.4	34.0	34.8	35.6
C(4)	45.9	48.6	46.2	46.4	48.3
C(5)	86.9	89.2	86.6	86.7	89.3
C(α)	28.7	27.5	27.0	29.1	28.8
C_a	21.7	22.4	21.9	21.7	22.3
C_b	27.0	27.9	27.2	27.1	27.8
C(1′)	136.8	135.6	128.3	137.2	131.7
C(2′)	122.4	128.8	122.8	137.0	129.4
C(3′)	152.5	152.0	143.6	129.3	145.0
C(4′)	113.1	144.8	141.0	127.0	144.5
C(5′)	128.6	120.0	112.2	129.3	115.6
C(6′)	127.7	126.4	127.9	137.0	125.2
C–C(2′)	12.2	12.5	12.3	20.4	13.3
C–C(6′) or C(2″)	19.9	194.2	99.7	20.4	64.2

[a]) For other ^{13}C-NMR data, see *Exper. Part.* [b]) Reported data from [5].

The ^{1}H-NMR spectrum revealed the presence of four Me groups, including two aromatic Me groups (δ 1.43 (*s*), 1.56 (*s*), 2.20 (*s*), 2.24 ppm (*s*)), and a tetrasubstituted Ph group (δ 6.59 (*d*, J = 8.1 Hz), 6.87 ppm (*d*, J = 8.1 Hz)), the presence of which was supported by the MS fragment at *m*/*z* 135 (base peak), which derived from the dimethyl-hydroxybenzyl moiety. The ^{13}C-NMR DEPT spectra showed one aromatic ring, one lactone group, one oxygenated quaternary C-atom (δ 86.9 ppm), one CH, two CH_2, and four Me C-atoms. The connectivity of the ^{1}H- and ^{13}C-NMR signals was determined by a HSQC spectrum, and the gross structure of **1** was established by HMBC analysis (*Fig.*), in which the correlations of Me_a (δ 1.43 ppm), Me_b (δ 1.56 ppm) to C(5) (δ 86.9 ppm) indicated that C(5) of the γ-lactone was substituted by two Me groups; the correlations of $CH_2(\alpha)$ (δ 2.74 ppm) with C(4) (δ 45.9 ppm) and C(1′) (δ 136.8 ppm) and correlations of Me_a (δ 1.43 ppm), Me_b (δ 1.56 ppm) with C(4) (δ 45.9 ppm) suggested that a benzyl group was located at C(4). The positions of the two Me and the OH groups at the aromatic ring were assigned from the HMBC correlations of *Me*–C(6′), *Me*–C(2′) to C(1′) and Me–C(2′) to C(3′).

The absolute configuration of **1** was tentatively assigned as (4*R*), based on a comparison of the optical rotation ($[\alpha]_D^{20} = +24.3$) with the one of compound **4** ($[\alpha]_D^{20} = +14.0$) [5].

Lycifuranone B (**2**) was obtained as colorless needles from AcOEt and gave the molecular-ion peak M^+ at *m*/*z* 278.1147 in the EI-HR-MS, which is consistent with the molecular formula $C_{15}H_{18}O_5$. The IR spectrum showed the absorption bands of OH groups (3388 cm^{-1}), an aromatic ring (1578 cm^{-1}), a γ-lactone (1735 cm^{-1}), and an aldehyde (1676 cm^{-1}). Comparison of the ^{1}H-NMR (*Table 1*) and ^{13}C-NMR (*Table 2*) spectroscopic data of **1** and **2** revealed that compound **2** is structurally closely related to **1**. Compound **2** was assigned the structure of *4,5-dihydroxy-3-methyl-2-{[(3*R*)-tetrahydro-2,2-dimethyl-5-oxofuran-3-yl]methyl}benzaldehyde* on the basis of the following evidences.

In the ^{1}H-NMR spectrum[1]), the signals at δ 1.44 (*s*), 1.55 (*s*), and 2.23 ppm (*s*) showed the presence of three Me groups, the last one being an aromatic substituent. A set of signals at δ 3.10 (br. *d*, H_a–C(α)) and 3.38 ppm (br. *d*, H_b–C(α)) was attributed to the geminal H-atoms of the benzylic CH_2 group. A CH H-atom at δ 2.46 (*ddd*, J = 11.4, 7.2, 3.7 Hz, H–C(4)) was coupled with two sets of *dd*s at δ 2.16 (*dd*, J = 16.5, 7.2, H_a–C(3)) and 2.57 ppm (*dd*, J = 16.5, 11.4, H_b–C(3)). It was supported by an important MS fragment at *m*/*z* 165, implying the loss of a 5,5-dimethyl-γ-lactone ring ($[M - C_6H_9O_2]^+$). Signals due to an aromatic H-atom (δ 7.14 ppm (*s*)) and a CHO group (δ 9.88 ppm) were also observed in the ^{1}H-NMR spectrum. These data suggested the presence of a pentasubstituted benzene ring endowed with two phenolic OH groups, a Me group, a CHO group, and a CH_2 unit containing the γ-lactone moiety. All H-atoms were assigned to the corresponding C-atoms by a HSQC experiment. In addition, the positions of two OH groups and a CHO group at the aromatic ring were confirmed by NOESY correlations of H–C(5′) with a CHO group at C(6′) and a OH group at C(4′) (*Fig.*). These conclusions were also supported by the observed HMBC correlations.

Although compound **2** was optically active with specific rotation $[\alpha]_D^{20} = -20$, it can be transformed with $NaBH_4$ into **5**, which has a specific rotation $[\alpha]_D^{20} = +20$. Therefore, the configuration at C(4)[1]) of **2** was assigned tentatively to be the same as that of compound **4**. Its reversed specific rotation was thought to be caused by the presence of the aldehyde.

Lycifuranone C (**3**), a pale yellow powder, has a molecular formula of $C_{32}H_{44}O_8$ (HR-EI-MS: *m*/*z* 556.3027 (M^+)). The IR data suggested the presence of OH groups (3423 cm^{-1}), an aromatic ring (1516 cm^{-1}), and a γ-lactone (1743 cm^{-1}). The following analysis of the NMR data of **3** (*Tables 2* and *3*) established the structure of *(4*R*)-4-(3,4-dihydroxy-6-{(2*S*,4*R*,6*S*)-4-[2-(4-hydroxy-3-methoxyphenyl)ethyl]-6-pentyl[1,3]dioxan-2-yl}-2-methylbenzyl)-4,5-dihydro-5,5-dimethylfuran-2(3*H*)-one* for this metabolite.

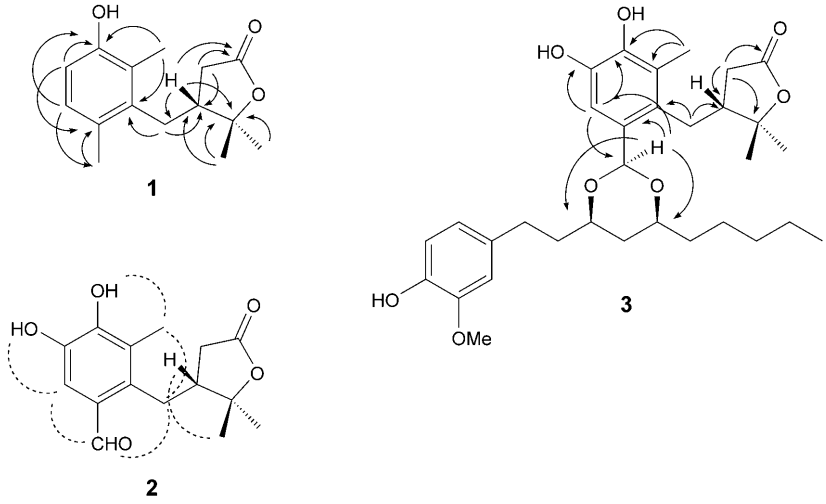

Figure. *Selected HMBC correlations of* **1** *and* **3** (in $CDCl_3$) *and key NOESY correlations of* **2** ((D_6)DMSO)

Table 3. *^{1}H-NMR Data of* **3**–**5**. δ in ppm, J in Hz.

	3[a]) $CDCl_3$, 400 MHz	**4**[b]) $(CD_3)_2CO$, 500 MHz	**5** CD_3OD, 400 MHz
CH_2(3)	2.28–2.35 (*m*), 2.52[c])	2.21 (J = 22, 13), 2.53 (*m*)	2.22[d]), 2.57[e])
H–C(4)	2.49[c])	2.53 (*m*)	2.58[e])
$CH_2(\alpha)$	2.81 (br. *d*, J = 6.4)	2.78 (J = 14, 11), 2.86 (J = 14, 3)	2.74 (*dd*, J = 14.0, 11.0) 2.85 (*dd*, J = 14.0, 3.0)
Me_a–C(5)	1.41 (*s*)	1.43 (*s*)	1.44 (*s*)
Me_b–C(5)	1.52 (*s*)	1.55 (*s*)	1.55 (*s*)
H–C(3′)		7.00 (*s*)	
H–C(4′)		7.00 (*s*)	
H–C(5′)	7.02 (*s*)	7.00 (*s*)	6.71 (*s*)
Me–C(2′)	2.18 (*s*)	2.34 (*s*)	2.20 (*s*)[d])
C(2″) or Me–C(6′) or CH_2–C(6′)	5.49 (*s*)	2.34 (*s*)	4.50 (*AB*, *q*, J = 12)

[a]) For other ^{1}H-NMR data, see *Exper. Part.* [b]) Reported data from [5]. [c])–[e]) Overlapped.

The ^{13}C-NMR spectrum revealed 32 peaks, of which a subset of 15 signals were very similar to those of compound **2** except that the CHO group was replaced by a CH group adjacent to two O-atoms. Signals for the remaining 17 C-atoms and signals in the ^{1}H-NMR spectrum revealed the presence of a unit derived from *1-(4-hydroxy-3-methoxyphenyl)decane-3,5-diol* [6], which was also isolated from this plant and identified by us. Thus, compound **3** was elucidated as the aldehyde acetal of compound **2** with 1-(4-hydroxy-3-methoxyphenyl)decane-3,5-diol. This fact was supported by the HMBC spectrum (*Fig.*). Furthermore, compound **3** hydrolyzed in 1N HCl within 5 min to compound **2** and (3*R*,5*S*)-1-(4-hydroxy-3-methoxyphenyl)decane-3,5-diol. The relative configuration of compound **3** was confirmed by NOE spectroscopy. When the signals at δ 3.76 ppm (*m*, H–C(4″) and H–C(6″)) were irradiated, the signal at δ 5.49 ppm (*s*, H–C(2″)) showed an obvious NOE enhancement.

Experimental Part

General. Column chromatography (CC): Silica gel (200–300 mesh; *Qingdao Marine Chemical*, China), *Sephadex LH-20* (*Pharmacia Fine Chemical Co. Ltd*). TLC: pre-coated silica-gel *GF254* plates (*Yantai Marine Chemical*, China); detection at 254 nm, and by spraying with 10% H_2SO_4 soln. followed by heating. ORD: *Perkin-Elmer 341* polarimeter. UV/VIS: *Varian CARY 300 Bio* UV/VIS spectrometer; λ_{max} in nm. IR: *Perkin-Elmer 577* IR spectrometer; ν in cm^{-1}. NMR: *Brucker AM-400*, Me_4Si as internal standard; δ in ppm, J in Hz. EI-MS and HR-EI-MS: *Finnigan MAT-95*; in m/z (rel. %).

Plant Material. The roots of *Lycianthes marlipoensis* were collected in June 2003 in Wenshan, Yunnan province, China and identified by Prof. *Shengli Pan*, Department of Pharmacognosy, Fudan University. A voucher specimen (LA 030918) was deposited in our laboratory.

Extraction and Isolation. The roots (20 kg) were extracted with hot 95% aq. EtOH. The extract was evaporated to yield a syrup, which was partitioned successively with petroleum ether/H_2O, AcOEt/H_2O, and BuOH/H_2O to afford an AcOEt fraction (70 g). This fraction was subjected to CC (silica gel, petroleum ether/AcOEt 6:1, 4:1, 3:1, 2:1, 1:1) to give nine fractions: *Fr. 1–9*. *Fr. 2* was repeatedly chromatographed (silica gel, cyclohexane/acetone 5:1, 3:1, 2:1, 1.5:1) and then further purified (*Sephadex LH-20*, $CHCl_3$/MeOH 1:1) to yield **1** (11 mg). *Fr. 5* was repeatedly chromatographed (silica gel, cyclohexane/acetone 5:1, 3:1, 2:1, 1.5:1) and then further purified (*Sephadex LH-20*, $CHCl_3$/MeOH 1:1) to yield **2** (18 mg) and **3** (8 mg).

Lycifuranone A (= *(4*R*)-4,5-Dihydro-4-(3-hydroxy-2,6-dimethylbenzyl)- 5,5-dimethylfuran-2(3*H*)-one*; **1**). White powder. $[\alpha]_D^{20} = +24.3$ ($c = 1.03$, $CHCl_3$), +19.4 ($c = 0.72$, MeOH). UV ($CHCl_3$): 283. IR (KBr): 3517, 3257, 2981, 1716, 1591, 1377, 1273, 1122, 1055, 951, 816, 650. ^{1}H-NMR: see *Table 1*. ^{13}C-NMR: see *Table 2*. EI-MS: 248 (35, M^+), 233 (3), 215 (5), 188 (25), 173 (14), 147 (13), 135 (100). HR-EI-MS: 248.1415 (M^+, $C_{15}H_{20}O_3$; calc. 248.1413).

Lycifuranone B (= *4,5-Dihydroxy-3-methyl-2-{[(3*R*)-tetrahydro-2,2-dimethyl-5-oxofuran-3-yl]methyl}-benzaldehyde*; **2**). Colorless needles. M.p. 207–208° (AcOEt). $[\alpha]_D^{20} = -20$ ($c = 0.215$, MeOH). UV (MeOH): 214.0, 237.5, 291.5. IR (KBr): 3388, 3261, 2972, 1735, 1676, 1578, 1415, 1379, 1307, 1176, 1117, 1028, 955, 642. ^{1}H-NMR: see *Table 1*. ^{13}C-NMR: see *Table 2*. EI-MS: 278 (13, M^+), 263 (7), 260 (17), 235 (28), 220 (100), 175 (56), 165 (62), 137 (40). HR-EI-MS: 278.1147 (M^+, $C_{15}H_{18}O_5$; calc. 278.1155).

Lycifuranone C (= *(4*R*)-4-(3,4-Dihydroxy-6-{(2*S*,4*R*,6*S*)-4-[2-(4-hydroxy-3-methoxyphenyl)ethyl]-6-pentyl[1,3]dioxan-2-yl}-2-methylbenzyl)-4,5-dihydro-5,5-dimethylfuran-2(3*H*)-one*; **3**). Pale yellow powder. $[\alpha]_D^{20} = +15.0$ ($c = 0.26$, $CHCl_3$). UV (MeOH): 282.0. IR (KBr): 3423, 2931, 2858, 1743, 1604, 1516, 1464, 1302, 1273, 1234, 1122, 1034, 1001, 956, 876. ^{1}H-NMR: 0.87 (*t*, $J = 6.9$, *Me*$(CH_2)_4$–C(6″)); 1.2–1.4 (*m*, $(CH_2)_4$–C(6″)); 1.64 (*m*, CH_2(5″)); 1.73, 1.80 (2*m*, C(4″)–*C*H_2–CH_aH_b); 2.60 (*ddd*, $J = 13.9$, 8.9, 6.4, C(4″)–CH_2–CH_aH_b); 2.71 (*ddd*, $J = 13.9$, 9.9, 6.0, C(4″)–CH_2–CH_aH_b); 3.76 (*m*, H–C(4″), H–C(6″)); 3.82 (*s*, MeO–C(3‴)); 6.64 (*dd*, $J = 8.0$, 1.9, H–C(6‴)); 6.67 (*d*, $J = 1.9$, H–C(2‴)); 6.80 (*d*, $J = 8.0$, H–C(5‴)); for the assignment of other H-atoms, see *Table 3*. ^{13}C-NMR: 14.0 (*Me*$(CH_2)_4$–C(6″)); 22.5 (Me*C*$H_2(CH_2)_3$–C(6″)); 24.6 (Me$(CH_2)_2$*C*H_2CH_2–C(6″)); 31.0 (C(4″)–CH_2–*C*H_2); 31.8 (MeCH_2*C*$H_2(CH_2)_2$–C(6″)); 35.9 (Me$(CH_2)_3$*C*H_2–C(6″)); 36.7 (C(5″)); 37.7 (C(4″)–*C*H_2–CH_2); 55.9 (*Me*O–C(3‴)); 77.0 (C(4″)); 77.0 (C(6″)); 111.0 (C(2‴)); 114.2 (C(5‴)); 120.8 (C(6‴)); 133.4 (C(1‴)); 143.7 (C(4‴)); 146.4 (C(3‴)); for the assignment of other C-atoms, see *Table 2*. EI-MS: 556 (11, M^+), 278 (85), 261 (11), 220 (17), 165 (18), 137 (100). HR-EI-MS: 556.3027 (M^+, $C_{32}H_{44}O_8$; calc. 556.3037).

Reduction of Compound **2**. To a soln. of **2** (5.0 mg) in 5 ml MeOH, $NaBH_4$ was added, and the mixture was stirred at 0° for 30 min. The soln. was poured into 5 ml H_2O and neutralized with 1% AcOH. After removing the MeOH *in vacuo*, the residue was extracted with AcOEt (3 × 5 ml). The combined org. layers were evaporated to dryness and subjected to CC (*Sephadex LH-20*, CH_3Cl/MeOH 1:1) to give **5** (5.1 mg).

*(4*R*)-4,5-Dihydro-4-[3,4-dihydroxy-6-(hydroxymethyl)-2-methylbenzyl]-5,5-dimethylfuran-2(3*H*)-one* (**5**). White powder. $C_{15}H_{20}O_5$. $[\alpha]_D^{20} = +20.0$ ($c = 0.21$, MeOH). IR (KBr): 3423, 2976, 2927, 1741, 1618, 1458, 1377, 1304, 1122, 1033, 955, 864, 652. ^{1}H-NMR: see *Table 3*. ^{13}C-NMR: see *Table 2*. EI-MS: 280 (4, M^+), 262 (20), 232 (21), 220 (22), 202 (20), 166 (36), 150 (100), 137 (40), 121 (59).

Acid Hydrolysis of Compound **3**. A soln. of **3** (4 mg) in 0.5 ml MeOH and 0.5 ml 1N HCl was allowed to stand at r.t. for 5 min. The soln. was poured into 5 ml sat. aq. $NaHCO_3$ and subsequently extracted with AcOEt (3 × 5 ml). The org. layer was evaporated to a small volume and separated by prep. TLC (MeOH/$CHCl_3$ 3:97) to give **2** (1.8 mg) and *1-(4-hydroxy-3-methoxyphenyl)decane-3,5-diol* (1.8 mg). Furthermore, the configuration of the latter was assigned as (3*R*,5*S*) based on a comparison of the optical rotation with the one of an authentic sample.

*(3*R,5S*)-1-(4-Hydroxy-3-methoxyphenyl)decane-3,5-diol*. Oil. $C_{17}H_{28}O_4$. $[\alpha]_D^{20} = +11.0$ ($c = 0.18$, $CHCl_3$). ^{1}H-NMR: 0.88 (*t*, *J* = 6.9, Me(10)); 1.2–1.5 (*m*, CH_2(6, 7, 8, 9)); 1.49 (*dt*, *J* = 10.1, 14.4, H_a–C(4)); 1.61(*dt*, *J* = 2.4, 14.4, H_b–C(4)); 1.74 (*m*, CH_2(2)); 2.60 (*ddd*, *J* = 13.9, 9.2, 6.9, H_a–C(1)); 2.69 (*ddd*, *J* = 13.9, 9.6, 6.0, H_b–C(1)); 3.85 (*m*, H–C(3), H–C(5)); 6.67 (*dd*, *J* = 8.0, 1.9, H–C(6′)); 6.70 (*d*, *J* = 1.9, H–C(2′)); 6.82 (*d*, *J* = 8.0, H–C(5′)). EI-MS: 296 (30, M^+), 278 (23), 150 (15), 137 (100).

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Received March 16, 2005