

Further Biphenyl Lignans with a Tetrahydrofuran Moiety from *Gymnotheca chinensis*

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Four new biphenyl lignans, **1–4**, with a tetrahydrofuran moiety were isolated from the MeOH extract of the whole plant of *Gymnotheca chinensis*. Their chemical structures were established by means of spectroscopic analysis, including 1D and 2D NMR spectroscopy and comparison with previously reported data. Compounds **1–4** occur as mixtures of atropisomers as revealed by variable-temperature NMR experiments.

Introduction. – *Gymnotheca chinensis* DECNE, one of the endemic genera of seed plants in China, is a perennial herb of the family Saururaceae. The whole plants of *G. chinensis* have long been used as traditional herbal medicine to treat contusions and strains [1]. In our previous investigations on *G. chinensis*, 15 new lignans belonging to the dibenzocyclooctene, eupomatilone, and eupodienone series were isolated [2]. Eupomatilones are biphenyl lignans originally isolated from the Australian shrub *Eupomatia bennettii*. All members of this family contain a highly oxygenated biphenyl system with a γ -lactone ring or a tetrahydrofuran ring attached to one of the aryl rings, and some of the eupomatilone molecules occur as a mixture of fluctional atropisomers [3]. So far, this kind of degraded lignan was only isolated from *E. bennettii* and *G. chinensis*. As a consequence, these compounds have attracted interest of synthetic chemists [4–10]. Our recent phytochemical studies led to the isolation of four further new biphenyl lignans with a tetrahydrofuran moiety, named gymnothelignans P–S (**1–4**, resp.). The compounds occur as mixtures of atropisomers as revealed by variable-temperature NMR experiments.

Results and Discussion. – A MeOH extract of the whole plant of *G. chinensis* was suspended in H₂O, and successively extracted with petroleum ether and AcOEt. The petroleum ether and AcOEt extracts were combined and then further fractionated by various chromatographic techniques to yield four new biphenyl lignans **1–4** (Fig. 1).

Compound **1** was obtained as white powder, with the molecular formula, C₂₂H₂₆O₇, deduced from HR-ESI-MS (m/z 401.1571 ($[M-H]^-$), indicating ten degrees of unsaturation. In the NMR spectra (Tables 1 and 2), several signals were doubled, indicating the presence of two rotational stereoisomers in a ca. 1:1 ratio at room temperature. On raising the temperature of a (D₆)DMSO solution of **1**, the ¹H-NMR

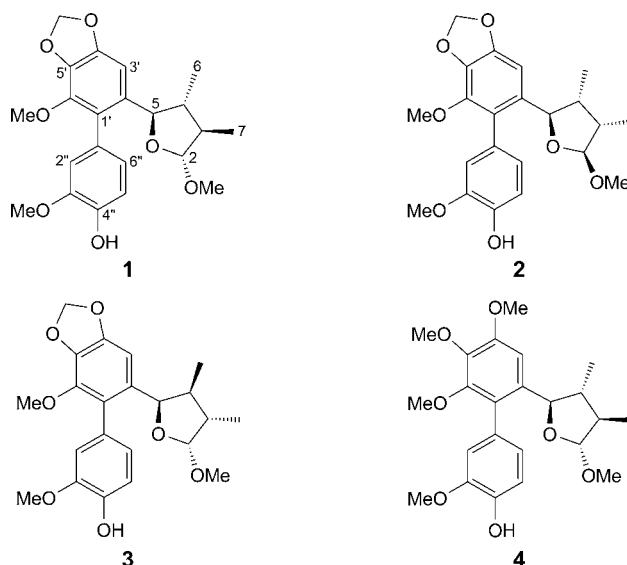


Fig. 1. Structures of compounds 1–4

signals broadened and coalesced, eventually yielding a single signal (Fig. 2), owing to the rapid interconversion of the two conformers at high temperature. The coalescence point for the two sets of signals was observed at *ca.* 100°. The presence of a OCH₂O group was evidenced by the HMBCs (Fig. 3) of OCH₂O to C(4') and C(5'). The correlations of MeO(6') to C(6'), and MeO(3'') to C(3'') positioned the two MeO groups to C(6') and C(3''), respectively. Similarly, a MeO group at C(2) was also assigned. HMBCs H–C(2)/C(5) indicated the connection of C(2) to C(5) *via* an ether bond. Detailed HMBC analysis suggested that the structure of **1** was very similar to that of gymnothelignan G [2], except for the absence of MeO at C(5''), which was revealed by the ¹H-NMR *AMX* spin system (δ (H) 6.57 (br. *d*, $J = 8.0$, H–C(6'')), 6.82 (*d*, $J = 8.0$, H–C(5'')), 6.70 (br. *s*, H–C(2''))). The orientations of the substituents of the tetrahydrofuran ring were deduced from NOESY correlations (Fig. 4). The cross-peaks H–C(2)/Me(7) and H–C(5)/Me(6) indicated that H–C(2) and Me(7) were cofacial, H–C(5) and Me(6) were on the opposite face. The vicinal coupling constants $J(2,3) = 4.9$ and $J(4,5) = 4.1$ were in close agreement with those observed in gymnothelignan G [2]. Gymnothelignan P (**1**) was thus identified as 5-(4''-hydroxy-6',3''-dimethoxy-4',5'-(methylenedioxy)-1,1'-biphenyl-2'-yl)-2*a*-methoxy-3 β ,4*a*-dimethyl-tetrahydrofuran¹⁾. The absolute configuration of **1** remains to be determined.

Compound **2** and **3** had the same molecular formula as those of **1** as deduced from HR-ESI-MS. The ¹H- and ¹³C-NMR, and HMBC spectra indicated that **2** and **3** were stereoisomers of **1**. In a NOESY experiment, the cross-peaks H–C(2)/Me(7), H–C(2)/H–C(5), and H–C(3)/H–C(4) were observed in compound **2**, and H–C(2)/H–C(3), H–C(2)/Me(6), H–C(3)/Me(6), and H–C(5)/H–C(4), Me(7)/H–C(4) were observed

¹⁾ For systematic names, see the *Exper. Part*.

Table 1. ¹H-NMR Data (400 MHz) of **1–4**. δ in ppm, *J* in Hz. Atom numbering as indicated in Fig. 1.

Position	1 ^{a)}	1 ^{b)}	1 ^{c)}	2 ^{b)}	3 ^{b)}	4 ^{a)}
2	4.97 (<i>d</i> , <i>J</i> = 4.9)	4.95 (<i>d</i> , <i>J</i> = 4.9)	4.96 (<i>d</i> , <i>J</i> = 4.9)	4.55 (s)	4.91 (<i>d</i> , <i>J</i> = 4.4)	5.01 (<i>d</i> , <i>J</i> = 4.4)
	4.98 (<i>d</i> , <i>J</i> = 4.9)	4.96 (<i>d</i> , <i>J</i> = 4.9)		4.55 (s)	4.91 (<i>d</i> , <i>J</i> = 4.4)	5.00 (<i>d</i> , <i>J</i> = 4.4)
3	2.30–2.41 (<i>m</i>)	2.25–2.36 (<i>m</i>)	2.29–2.38 (<i>m</i>)	2.28–2.38 (<i>m</i>)	1.83–1.89 (<i>m</i>)	2.31–2.43 (<i>m</i>)
	2.30–2.41 (<i>m</i>)	2.25–2.36 (<i>m</i>)		2.28–2.38 (<i>m</i>)	1.83–1.89 (<i>m</i>)	2.31–2.43 (<i>m</i>)
4	1.91–2.00 (<i>m</i>)	1.86–1.94 (<i>m</i>)	1.93–2.01 (<i>m</i>)	2.10–2.18 (<i>m</i>)	1.75–1.83 (<i>m</i>)	1.93–2.02 (<i>m</i>)
	1.91–2.00 (<i>m</i>)	1.86–1.94 (<i>m</i>)		2.10–2.18 (<i>m</i>)	1.75–1.83 (<i>m</i>)	1.93–2.02 (<i>m</i>)
5	4.58 (<i>d</i> , <i>J</i> = 4.1)	4.42 (<i>d</i> , <i>J</i> = 4.0)	4.47 (<i>d</i> , <i>J</i> = 4.6)	4.52 (<i>d</i> , <i>J</i> = 8.9)	4.99 (<i>d</i> , <i>J</i> = 8.5)	4.65 (<i>d</i> , <i>J</i> = 3.9)
	4.55 (<i>d</i> , <i>J</i> = 4.1)	4.42 (<i>d</i> , <i>J</i> = 4.0)		4.52 (<i>d</i> , <i>J</i> = 8.9)	5.04 (<i>d</i> , <i>J</i> = 8.5)	4.62 (<i>d</i> , <i>J</i> = 3.9)
6	0.61 (<i>d</i> , <i>J</i> = 7.2)	0.53 (<i>d</i> , <i>J</i> = 7.1)	0.60 (<i>d</i> , <i>J</i> = 7.1)	0.67 (<i>d</i> , <i>J</i> = 6.9)	0.66 (<i>d</i> , <i>J</i> = 7.5)	0.61 (<i>d</i> , <i>J</i> = 7.1)
	0.62 (<i>d</i> , <i>J</i> = 7.2)	0.53 (<i>d</i> , <i>J</i> = 7.1)		0.68 (<i>d</i> , <i>J</i> = 6.9)	0.68 (<i>d</i> , <i>J</i> = 7.5)	0.62 (<i>d</i> , <i>J</i> = 7.1)
7	0.85 (<i>d</i> , <i>J</i> = 7.2)	0.79 (<i>d</i> , <i>J</i> = 7.2)	0.82 (<i>d</i> , <i>J</i> = 7.2)	0.74 (<i>d</i> , <i>J</i> = 7.3)	0.92 (<i>d</i> , <i>J</i> = 6.6)	0.85 (<i>d</i> , <i>J</i> = 7.2)
	0.86 (<i>d</i> , <i>J</i> = 7.2)	0.79 (<i>d</i> , <i>J</i> = 7.2)		0.75 (<i>d</i> , <i>J</i> = 7.3)	0.92 (<i>d</i> , <i>J</i> = 6.6)	0.86 (<i>d</i> , <i>J</i> = 7.2)
3'	6.70 (s)	6.68 (s)	6.68 (s)	6.85 (s)	6.64 (s)	6.90 (s)
	6.70 (s)	6.68 (s)		6.85 (s)	6.64 (s)	6.90 (s)
2''	6.77 (<i>d</i> , <i>J</i> = 1.9)	6.66 (<i>d</i> , <i>J</i> = 1.8)	6.70 (br. s)	6.74 (br. s)	6.70 (br. s)	6.78 (<i>d</i> , <i>J</i> = 1.8)
	6.75 (<i>d</i> , <i>J</i> = 1.9)	6.69 (<i>d</i> , <i>J</i> = 1.8)		6.74 (br. s)	6.74 (br. s)	6.80 (<i>d</i> , <i>J</i> = 1.8)
5''	6.88 (<i>d</i> , <i>J</i> = 8.0)	6.80 (<i>d</i> , <i>J</i> = 8.0)	6.82 (<i>d</i> , <i>J</i> = 8.0)	6.84 (<i>d</i> , <i>J</i> = 8.1)	6.88 (<i>d</i> , <i>J</i> = 8.0)	6.88 (<i>d</i> , <i>J</i> = 8.0)
	6.88 (<i>d</i> , <i>J</i> = 8.0)	6.80 (<i>d</i> , <i>J</i> = 8.0)		6.85 (<i>d</i> , <i>J</i> = 8.1)	6.88 (<i>d</i> , <i>J</i> = 8.0)	6.88 (<i>d</i> , <i>J</i> = 8.0)
6''	6.61 (<i>ddd</i> , <i>J</i> = 8.0, 1.9)	6.51 (<i>ddd</i> , <i>J</i> = 8.0, 1.8)	6.57 (<i>d</i> , <i>J</i> = 8.0)	6.60 (<i>d</i> , <i>J</i> = 8.1)	6.59 (<i>d</i> , <i>J</i> = 8.0)	6.64 (<i>ddd</i> , <i>J</i> = 8.0, 1.8)
	6.66 (<i>ddd</i> , <i>J</i> = 8.0, 1.9)	6.56 (<i>ddd</i> , <i>J</i> = 8.0, 1.8)		6.62 (<i>d</i> , <i>J</i> = 8.1)	6.63 (<i>d</i> , <i>J</i> = 8.0)	6.69 (<i>ddd</i> , <i>J</i> = 8.0, 1.8)
2-MeO	3.25 (s)	3.18 (s)	3.22 (s)	3.38 (s)	3.21 (s)	3.25 (s)
	3.25 (s)	3.19 (s)		3.38 (s)	3.21 (s)	3.25 (s)
OCH ₂ O	6.00, 6.01 (2s)	6.00, 6.03 (2s)	5.99, 6.00 (2s)	6.02 (s)	6.01 (s)	
	6.00, 6.01 (2s)	6.00, 6.03 (2s)		6.02 (s)	6.01 (s)	
6'-MeO	3.73 (s)	3.68 (s)	3.69 (s)	3.72 (s)	3.74 (s)	3.56 (s)
	3.73 (s)	3.69 (s)		3.72 (s)	3.74 (s)	3.56 (s)
3''-MeO	3.83 (s)	3.71 (s)	3.77 (s)	3.82 (s)	3.83 (s)	3.84 (s)
	3.85 (s)	3.74 (s)		3.84 (s)	3.84 (s)	3.86 (s)
4'-MeO						3.87 (s)
5'-MeO						3.87 (s)
						3.81 (s)
						3.81 (s)
4''-OH	7.59 (s)	8.98 (s)	8.44 (br. s)	7.57 (br. s)	7.60 (br. s)	7.58 (br. s)
	7.59 (s)	8.98 (s)		7.57 (br. s)	7.60 (br. s)	7.59 (br. s)

^{a)} Recorded in (D₆)acetone, at 25°. ^{b)} Recorded in (D₆)DMSO, at 25°. ^{c)} Recorded in (D₆)DMSO, at 100°.

Table 2. $^{13}\text{C-NMR}$ Data (100 MHz, in (D_6) acetone, at 25°) of **1–4**. δ in ppm. Atom numbering as indicated in Fig. 1.

Position	1	2	3	4
2	108.3	111.5	106.2	108.3
	108.2	111.5	106.2	108.3
3	40.3	44.0	42.6	40.2
	40.3	44.0	42.6	40.2
4	44.3	44.4	46.5	44.2
	44.4	44.5	46.8	44.3
5	85.0	84.0	79.5	85.1
	85.0	84.0	79.5	85.1
6	15.1	11.6	16.3	15.2
	15.2	11.6	16.5	15.3
7	9.1	11.2	12.6	9.1
	9.1	11.2	12.6	9.1
1'	128.5	128.4	128.6	128.7
	128.6	128.5	128.7	128.8
2'	137.9	137.1	136.9	138.9
	137.9	137.1	136.9	138.9
3'	101.3	102.7	102.9	105.9
	101.3	102.7	102.9	105.9
4'	149.3	149.5	150.0	153.6
	149.3	149.5	150.0	153.6
5'	136.8	136.3	134.4	142.1
	136.8	136.3	134.4	142.1
6'	141.9	141.4	141.9	152.2
	141.9	141.4	141.9	152.2
1''	128.2	129.8	128.4	128.6
	128.2	129.8	128.4	128.7
2''	115.3	115.1	115.3	115.3
	115.5	115.3	115.3	115.4
3''	148.0	147.7	147.8	147.9
	148.0	147.8	147.9	148.0
4''	146.5	146.5	146.4	146.4
	146.5	146.5	146.5	146.5
5''	115.4	114.4	114.3	114.2
	115.9	115.9	115.6	115.8
6''	123.3	123.5	123.5	123.3
	124.9	125.0	124.2	124.9
2-MeO	54.8	54.9	54.5	54.8
	54.9	55.0	54.6	54.8
OCH ₂ O	102.0	102.0	102.0	
	102.0	102.0	102.0	
6'-MeO	60.0	60.0	60.0	61.1
	60.0	60.0	60.0	61.1
3''-MeO	56.4	56.3	56.3	56.4
	56.4	56.3	56.3	56.4
4'-MeO				56.3
				56.3
5'-MeO				60.7
				60.7

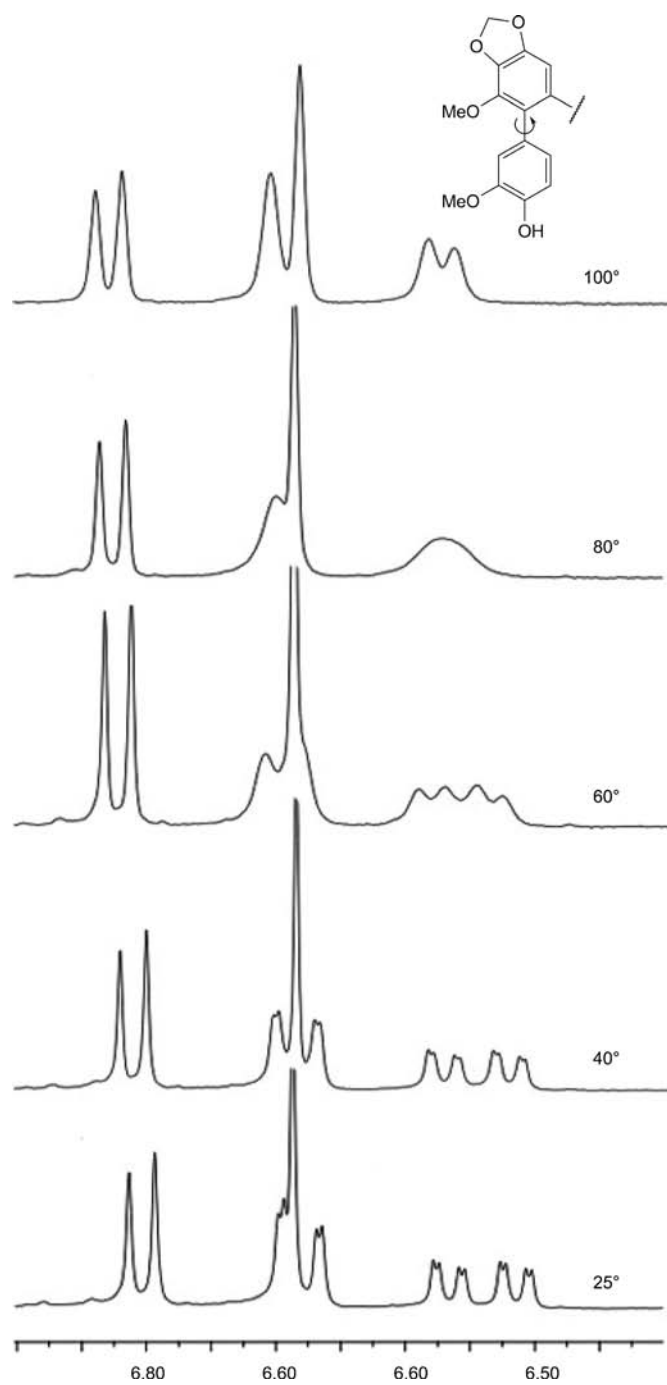
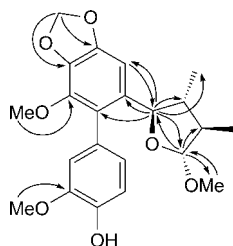
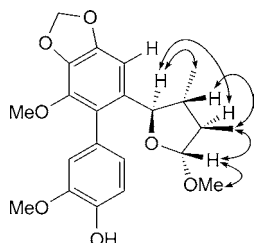


Fig. 2. Temperature dependence of the $^1\text{H-NMR}$ signals (400 MHz, in $(\text{D}_6)\text{DMSO}$) of the aryl H-atoms of **1**

Fig. 3. Key HMB (H → C) correlations of compound **1**Fig. 4. Key NOESY (H ↔ H) correlations of compound **1**

in compound **3**. The vicinal coupling constants $J(2,3) \approx 0$ in **2** and $J(2,3) = 4.4$ in **3**, and $J(4,5) = 8.9$ in **2** and $J(4,5) = 8.5$ in **3** were in close agreement with those observed in gymnothelignans F and H [2]. Thus, gymnothelignan Q (**2**) and gymnothelignan R (**3**) were identified as 2,3-diepi-**1** and 3,4-diepi-**1**, respectively.

Compound **4** was obtained as white powder. The molecular formula was determined as $C_{23}H_{30}O_7$ by analysis of the HR-ESI-MS (m/z 441.1891 ($[M + Na]^+$), indicating nine degrees of unsaturation. The 1H - and ^{13}C -NMR spectra (Tables 1 and 2) were very similar to those of **1** except for the replacement of the OCH_2O moiety by two MeO groups at C(4') and C(5'). Gymnothelignan S (**4**) was then determined as 5-(4'-hydroxy-4',5',6',3'-tetramethoxy-1,1'-biphenyl-2'-yl)-2 α -methoxy-3 β ,4 α -dimethyl tetrahydrofuran¹).

In conclusion, we identified four new biphenyl lignans with a tetrahydrofuran moiety, gymnothelignans P–S (**1–4**, resp.), from *G. chinensis*. These compounds were shown to have the same C-skeleton as eupomatilonones, and may be biogenetically derived from eupodienones [2][11]. Besides the eupomatilonones, no structurally related compounds were found in plants.

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Experimental Part

General. Column chromatography (CC): silica gel (SiO_2 , 300–400 mesh; *Qingdao Marine Chemical Co.*), MCI CHP-20 gel (75–150 μm ; *Mitsubishi*), and ODS (40–63 μm ; *LiChroprep*). TLC: Precoated plates GF 254 (*Qingdao Marine Chemical Co.*). Semi-prep. HPLC: LC3000 System (*Beijing*

ChuangXingTongHeng Science and Technology Co., Ltd.); column, *C18* (5 μm , i.d. 10 \times 250 mm; *Kromasil*). Optical rotations: *PerkinElmer-241* polarimeter. UV Spectra: *PerkinElmer Lambda 35*. IR Spectra: *Nicolet-MX-I* spectrometer; $\bar{\nu}$ in cm^{-1} . NMR Spectra: *Bruker Ascend 400* instrument; δ in ppm rel. to Me₄Si as internal standard, *J* in Hz. HR-ESI-MS: *MicrOTOF-Q II* mass spectrometer (*Bruker*, Germany); in *m/z*. ESI-MS: *Micromass Xevo triple-quadrupole* mass spectrometer (*Waters Corp.*, USA); in *m/z*.

Plant Material. The plant was collected from Jinshan in Chongqing City, P. R. China, in October 2012, and identified as *G. chinensis* by Prof. S. R. Yi, Chongqing Institute of Pharmaceutical Plant. A voucher specimen (20130125) was deposited with the herbarium of the Chengdu Institute of Biology, Chinese Academy of Sciences.

Extraction and Isolation. Dried and powdered whole plants of *G. chinensis* (1.45 kg) were extracted with MeOH at r.t. to give an extract (152 g), which was suspended in H₂O (1 l) and extracted with petroleum ether (PE) and AcOEt (3 \times 1 l, 3 h each) successively. The PE and AcOEt extracts (8 and 19 g, resp.) were combined and then subjected to CC (*MCI* gel (85 \times 100 mm); MeOH/H₂O 90 : 10). In total, two fractions, *A* and *B*, were obtained based on TLC analysis. *Fr. A* (15 g) was further separated by medium-pressure CC (SiO₂ (49 \times 460 mm); PE/acetone 100 : 1 \rightarrow 1 : 1) to give twelve fractions. *Fr. 7* (800 mg) was purified by CC (*ODS* (40–63 μm , 36 \times 310 mm); MeOH/H₂O 40 : 60 \rightarrow 100 : 0) to afford three main fractions, *Frs. 7A–7C*. Compound **4** (*t_R* 24.2 min; 4 mg) was obtained from *Fr. 7A* by semi-prep. HPLC (MeOH/H₂O 66 : 34; flow rate, 3 ml/min). Compounds **2** (*t_R* 23.1 min; 8 mg), **3** (*t_R* 35.4 min; 4 mg), and **1** (*t_R* 38.3 min, 10 mg) were obtained from *Fr. 7B* by semi-prep. HPLC (MeOH/H₂O 70 : 30; flow rate, 3 ml/min).

Gymnothelignan P (=2-Methoxy-4-[4-methoxy-6-[(2*R**,3*R**,4*R**,5*R**)-2,3,4,5-tetrahydro-5-methoxy-3,4-dimethylfuran-2-yl]-1,3-benzodioxol-5-yl]phenol; **1**). White powder. $[\alpha]_{\text{D}}^{20} = -90$ ($c = 0.04$, MeOH). UV (MeOH): 267 (3.69). IR (KBr): 3421, 2935, 1596, 1518, 1487, 1463, 1402, 1253, 1195, 1137, 1094, 1010. ¹H- and ¹³C-NMR: *Tables 1* and 2, resp. ESI-MS: 401 ($[M - H]^-$), 425 ($[M + Na]^+$). HR-ESI-MS: 401.1571 ($[M - H]^-$, C₂₂H₂₅O₇⁻; calc. 401.1600).

Gymnothelignan Q (=2-Methoxy-4-[4-methoxy-6-[(2*R**,3*R**,4*S**,5*S**)-2,3,4,5-tetrahydro-5-methoxy-3,4-dimethylfuran-2-yl]-1,3-benzodioxol-5-yl]phenol; **2**). White powder. $[\alpha]_{\text{D}}^{20} = -64$ ($c = 0.048$, MeOH). UV (MeOH): 267 (3.49). IR (KBr): 3276, 2970, 1598, 1518, 1477, 1390, 1233, 1212, 1082, 1034, 916. ¹H- and ¹³C-NMR: *Tables 1* and 2, resp. ESI-MS: 401 ($[M - H]^-$), 425 ($[M + Na]^+$). HR-ESI-MS: 401.1568 ($[M - H]^-$, C₂₂H₂₅O₇⁻; calc. 401.1600).

Gymnothelignan R (=2-Methoxy-4-[4-methoxy-6-[(2*R**,3*S**,4*S**,5*R**)-2,3,4,5-tetrahydro-5-methoxy-3,4-dimethyltetrahydrofuran-2-yl]-1,3-benzodioxol-5-yl]phenol; **3**). White powder. $[\alpha]_{\text{D}}^{20} = -97$ ($c = 0.041$, MeOH). UV (MeOH): 267 (3.60). IR (KBr): 2961, 1616, 1518, 1476, 1374, 1269, 1081, 936. ¹H- and ¹³C-NMR: *Tables 1* and 2, resp. ESI-MS: 401 ($[M - H]^-$), 425 ($[M + Na]^+$). HR-ESI-MS: 401.1604 ($[M - H]^-$, C₂₂H₂₅O₇⁻; calc. 401.1600).

Gymnothelignan S (=2',3,3',4'-Tetramethoxy-6-[(2*R**,3*R**,4*R**,5*R**)-2,3,4,5-tetrahydro-5-methoxy-3,4-dimethylfuran-2-yl]-1,1'-biphenyl-4-ol; **4**). White powder. $[\alpha]_{\text{D}}^{20} = -17$ ($c = 0.036$, MeOH). UV (MeOH): 266 (3.43). IR (KBr): 3421, 2935, 1597, 1519, 1487, 1463, 1253, 1138, 1094, 1010. ¹H- and ¹³C-NMR: *Tables 1* and 2, resp. ESI-MS: 417 ($[M - H]^-$), 441 ($[M + Na]^+$). HR-ESI-MS: 441.1884 ($[M + Na]^+$, C₂₃H₃₀NaO₇⁺; calc. 441.1889).

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