# Lignans from *Phryma leptostachya* L.

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Three new lignans, haedoxan J (**1**), phrymarolin III (**2**), and phrymarolin IV (**3**), as well as eight known lignans, leptostachyol acetate, haedoxan A, 1-(4,6-dimethoxy-1,3-benzodioxol-5-yl)dihydro-4-(6-methoxy-1,3-benzodioxol-5-yl)-1*H*,3*H*-furo[3,4-*c*]furan-3a(4*H*)-yl acetate, 4-(4,6-dimethoxy-1,3-benzodioxol-5-yl)dihydro-1-(4-methoxy-1,3-benzodioxol-5-yl)-1*H*,3*H*-furo[3,4-*c*]furan-3a(4*H*)-yl acetate, 4-[(4,6-dimethoxy-1,3-benzodioxol-5-yl)oxy]dihydro-1-(6-methoxy-1,3-benzodioxol-5-yl)-1*H*,3*H*-furo[3,4-*c*]-furan-3a(4*H*)-yl acetate, leptostachyol acetate C, 4-(4,6-dimethoxy-1,3-benzodioxol-5-yl)dihydro-1-(6-methoxy-1,3-benzodioxol-5-yl)-1*H*,3*H*-furo[3,4-*c*]furan-3a(4*H*)-yl acetate, and phrymarin II, were isolated from the plant *Phryma leptostachya* L. The structures of the new compounds were elucidated by analyzing their spectroscopic data and comparing with data reported in the literature.

**Introduction.** – *Phryma leptostachya* L., the sole species of the family Phrymaceae, is widely distributed in the Himalayas, temperate Asia, and northern East America. It has long been used as an empirical treatment of human's scabies in the folk medicine of the Yi people in China [1]. In the Southwest district of China, this herb has been used to drive or kill mosquitos and flys [2], while in East Asia, the root of the plant has been traditionally used as a natural insecticide [3–6].

*P. leptostachya* has been shown to be rich of lignans, many of which have a unique oxygenated 3,7-dioxabicyclo[3.3.0]octane skeleton [7]. Several lignans isolated from this species are insecticidal, such as haedoxan A, phrymarolin I, and phrymarolin II, among which haedoxan A showed the highest bioactivity [7]. To search for the active constituents of the title plant, we investigated its chemical composition under the guidance of the flycide-activity assay. From the flycide part of the ethanol extract of the herb of *P. leptostachya*, three new lignans were isolated, along with eight known lignans. This article is about the isolation and structure elucidation of these three new lignans, compounds **1**–**3** (*Fig. 1*).

**Results and Discussion.** – The HR-ESI-MS of compound **1** showed a quasi-molecular-ion peak ($[M+Na]^+$) at $m/z$ 665.1841, suggesting that its molecular formula was $C_{32}H_{34}O_{14}$ which was supported by 32 C-atom signals in the $^{13}$C-NMR spectrum of **1**.

In the $^1$H-NMR spectrum, the signals at $\delta$(H) 6.99 (*d*, $J=1.8$ Hz, H–C(2″)), 6.85 (*dd*, $J=8.0$, 1.8 Hz, H–C(6″)), and 6.80 (*d*, $J=8.0$ Hz, H–C(5″)) manifested the presence of a 1,3,4-substituted benzene moiety; and the signals at $\delta$(H) 7.05 (*s*, H–C(2′)), 6.60 (*s*, H–C(5′)), and 6.56 (*s*, H–C(5)) suggested that there were two other

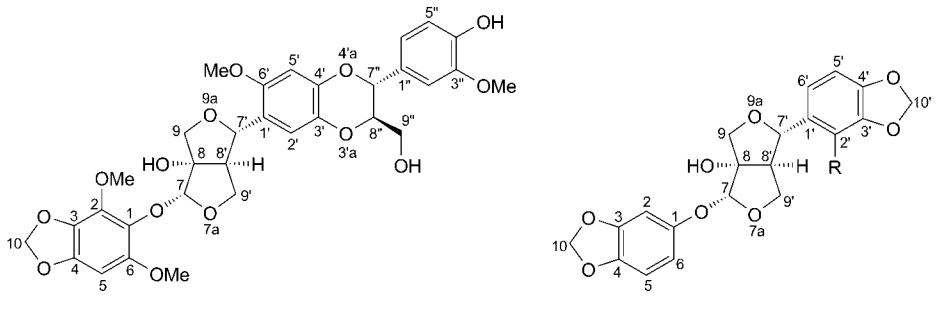


Fig. 1. *Compounds* **1**–**3**, *isolated from* Phryma leptostachya L.

benzene moieties in the molecule, a 1,2,4,5-substituted, and a 1,2,3,4,5-substituted one. The typical arene C-atom signals in the $^{13}$C-NMR spectrum of **1** confirmed the existence of three benzene rings in the molecule. In the $^{1}$H,$^{1}$H-COSY plot of **1**, the correlations of $\delta$(H) 2.44 (*m*, H–C(8′)) with $\delta$(H) 4.76 (*d*, $J$ = 5.8 Hz, H–C(7′)), 4.49 (*q*, $J$ = 7.9 Hz, $H_\alpha$–C(9′), and 3.82 (*dd*, $J$ = 9.0, 2.4 Hz, $H_\beta$–C(9′) implied the fragment CH(7′)–CH(8′)–$CH_2$(9′), which was located at C(1′) as inferred from the HMBC cross-peak $\delta$(H) 4.76 (H–C(7′))/$\delta$(C) 123.5 (C(1′)) (*Fig. 2*). Likewise, the existence of the fragment CH(7″)–CH(8″)–$CH_2$(9″) was suggested by the $^{1}$H,$^{1}$H-COSY cross-peaks between $\delta$(H) 4.01 (*m*, H–C(8″)) and $\delta$(H) 4.88 (*d*, $J$ = 7.8 Hz, H–C(7″)), 3.52 (br. *d*, $J$ = 12.0 Hz, $H_a$–C(9″)), and 3.32 (*dd*, $J$ = 12.0, 4.7 Hz, $H_b$–C(9″)), and the fragment was located at C(1″) as established by the HMBC cross-peak $\delta$(H) 4.88 (H–C(7″))/$\delta$(C) 128.1 (C(1″)) (*Fig. 2*). Another fragment, CH(7)–C(8)–$CH_2$(9), was revealed by the HMBC signals between $\delta$(C) 92.1 (C(8)) and $\delta$(H) 5.13 (*s*, H–C(7)), 4.12 (br. *d*, $J$ = 9.3 Hz, $H_\alpha$–C(9), and 3.51 (*m*, $H_\beta$–C(9), and was determined to be linked to C(1) *via* an O-atom as shown by the HMBC $\delta$(H) 5.13 (H–C(7))/$\delta$(C) 131.4 (C(1)) (*Fig. 2*). Therefore, the molecule of **1** contains three phenylpropyl moieties, hence it is a sesquilignan. The $^{1}$H-NMR spectrum of **1** resembled that of the known haedoxan A [6], suggesting the presence of a 6-aryl-2-(aryloxy)-3,7-dioxabicyclo[3.3.0]octane framework in the molecule. The four signals at $\delta$(H) 4.49 (*q*, $J$ = 7.9 Hz, $H_\alpha$–C(9′),

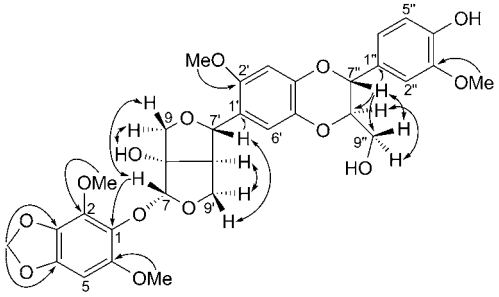

Fig. 2. *Key HMBC* (H → C) *and NOESY* (H ↔ H) *features of compound* **1**[1])

[1]) Trivial atom numbering; for systematic names, see *Exper. Part.*

3.82 (*dd*, $J = 9.0$, 2.4 Hz, $H_\beta$–C(9′) and 4.12 (br. *d*, $J = 9.3$ Hz, $H_\alpha$–C(9), and 3.51 (*m*, $H_\beta$–C(9) were similar to those of the corresponding H-atoms of haedoxan A, which implied that there are two furan rings in the molecule of **1**. The HMBC cross-peaks between $\delta$(H) 2.44 (*m*, H–C(8′)) and $\delta$(C) 92.1 (C(8)) (*Fig. 2*) suggested that the two phenylpropyl moieties were linked *via* C(8)–C(8′).

In the $^1$H-NMR spectrum of **1**, the signals at $\delta$(H) 3.89, 3.78, 3.71, and 3.71 (4*s*, each 3 H) and $\delta$(H) 5.93 (*s*, 2 H) implied the presence of four aromatic MeO groups and one methylenedioxy moiety. In the HMBC plot the correlation peaks $\delta$(H) 5.93 ($OCH_2O$)/$\delta$(C) 131.7 (C(3)) and 144.6 (C(4)) (*Fig. 2*) indicated that $OCH_2O$ was attached at C(3) and C(4), and $\delta$(H) 3.89/$\delta$(C) 147.4, $\delta$(H) 3.78/$\delta$(C) 138.6, $\delta$(H) 3.71/$\delta$(C) 148.0, and $\delta$(H) 3.71/$\delta$(C) 150.4 demonstrated that the MeO groups were located at C(2), C(6), C(6′), and C(3″) (*Fig. 2*). In the NOESY plot of **1**, the cross-peak $\delta$(H) 5.13 (H–C(7))/$\delta$(H) 3.51 $H_\beta$–C(9) indicated that H–C(7) was in $\beta$-configuration, while $\delta$(H) 5.31 (OH–C(8))/$\delta$(H) 4.12 ($H_\alpha$–C(9) established the $\alpha$-configuration of OH–C(8). Likewise, H–C(7′) was determined as $\beta$-oriented and H–C(8′) as $\alpha$-oriented by the NOEs $\delta$(H) 4.76 (H–C(7′))/$\delta$(H) 3.82 $H_\beta$–C(9′) and $\delta$(H) 2.44 (H–C(8′))/$\delta$(H) 4.49 $H_\alpha$–C(9′). The NOE H–C(8″)/$H_a$–C(9″) suggested that H–C(8″) was $\alpha$-configurated and the NOE of H–C(7″)/$H_b$–C(9″) implied that H–C(7″) was in $\beta$-configuration in the molecule (*Fig. 2*). The structure of **1** was thus established as shown in *Fig. 1* and it was named haedoxan J[1]).

Compound **2** gave a quasi-molecular ion at *m*/*z* 425.0843 ($[M + Na]^+$) in its HR-ESI-MS, and its molecular formula was inferred as $C_{20}H_{18}O_9$, which was supported by 20 C-atom signals in the $^{13}$C-NMR spectrum. The $^1$H- and $^{13}$C-NMR spectra of **2** (*Table*) showed the same pattern as those of phrymarolin II [5], except for the absence of the signals of an aromatic MeO and an AcO group and the presence of an aromatic OH and an aliphatic OH group, respectively, in **2**. This suggested that **2** contained two bezene moieties, a 1,3,4-substituted, and a 1,2,3,4-substituted one. In the HMBC plot of **2**, the cross-peaks $\delta$(H) 5.89 (*s*, $OCH_2O$)/$\delta$(C) 148.4 (C(3)) and 143.7 (C(4)) and $\delta$(H) 5.90 (*s*, $OCH_2O$)/$\delta$(C) 135.4 (C(3′)) and 149.4 (C(4′)) indicated that both of the benzene rings carried a methylenedioxy group, at C(3) and C(4), and at C(3′) and C(4′), respectively. The signal of C(7) ($\delta$(C) 102.6) located at lower field than that of C(7′) ($\delta$(C) 90.1), implied that C(7) was linked to C(1) *via* an O-atom, while C(7′) was attached directly to C(1′). The correlation pattern in the NOESY plot of **2** was also similar to that of phrymarolin II, indicating that the relative configurations of the stereogenic C-atoms C(7), C(8), C(7′), and C(8′) of **2** were just the same as those of phrymarolin II. Thus, the structure of **2** was established as shown in *Fig. 1*, and it was named phrymarolin III[1]).

Compound **3** gave a quasi-molecular-ion peak at *m*/*z* 439.2681 ($[M + Na]^+$) in its HR-ESI-MS, and its molecular formula was inferred as $C_{21}H_{20}O_9$ which was supported by the 21 C-atom signals in its $^{13}$C-NMR spectrum. The $^1$H- and $^{13}$C-NMR spectra of **3** (*Table*) had patterns analogous to those of **2**, except for the signals of an aromatic MeO group at $\delta$(H) 4.00 in the case of **3**. The linkage of C(7) to C(1) *via* an O-atom was deduced from the HMBC cross-peak of $\delta$(H) 5.30 (*s*, H–C(7))/$\delta$(C) 150.2 (C(1)). The HMBC $\delta$(H) 4.00 (*s*, MeO–(2′))/$\delta$(C) 125.3 (C(1′)), 139.4 (C(2′)), and 135.0 (C(3′)) indicated that the MeO was connected to C(2′). Just as compound **2**, the correlation pattern in the NOESY plot of **3** was identical to that of phrymarolin II, namely, the

Table. *$^1$H-NMR* (500 MHz) *and $^{13}$C-NMR* (125 MHz) *Data of Compounds* **2** *and* **3**. Measured in $CDCl_3$; $\delta$ in ppm, $J$ in Hz.

| | **2** | | **3** | |
|---|---|---|---|---|
| | $\delta$(H) | $\delta$(C) | $\delta$(H) | $\delta$(C) |
| C(1) | | 151.2 | | 150.2 |
| H–C(2) | 6.59 (*d*, *J* = 2.5) | 120.4 | 6.66 (*d*, *J* = 2.5) | 118.1 |
| C(3) | | 148.4 | | 142.4 |
| C(4) | | 143.7 | | 147.2 |
| H–C(5) | 6.65 (*d*, *J* = 8.5) | 108.3 | 6.72 (*d*, *J* = 9.5) | 107.1 |
| H–C(6) | 6.48 (*dd*, *J* = 8.5, 2.5) | 110.1 | 6.56 (*dd*, *J* = 9.5, 2.0) | 108.9 |
| H–C(7) | 5.24 (*s*) | 102.6 | 5.30 (*s*) | 102.2 |
| C(8) | | 92.4 | | 91.2 |
| $H_\alpha$–C(9) | 4.30 (*d*, *J* = 10.0) | 77.6 | 4.33 (*d*, *J* = 9.5) | 76.8 |
| $H_\beta$–C(9) | 3.61 (*d*, *J* = 10.0) | | 4.05 (*dd*, *J* = 9.5, 2.5) | |
| $CH_2$(10) | 5.89 (*s*) | 101.6 | 5.92 (*s*) | 100.4 |
| C(1′) | | 120.0 | | 125.3 |
| C(2′) | | 139.6 | | 139.4 |
| C(3′) | | 135.4 | | 135.0 |
| C(4′) | | 149.4 | | 147.8 |
| H–C(5′) | 6.33 (*d*, *J* = 8.5) | 100.9 | 6.55 (*d*, *J* = 9.0) | 101.7 |
| H–C(6′) | 6.51 (*d*, *J* = 8.5) | 101.8 | 7.09 (*d*, *J* = 9.0) | 99.7 |
| H–C(7′) | 4.59 (*d*, *J* = 7.0) | 90.1 | 4.88 (*d*, *J* = 6.5) | 83.6 |
| H–C(8′) | 2.77 – 2.80 (*m*) | 57.9 | 2.58 – 2.61 (*m*) | 57.5 |
| $H_\alpha$–C(9′) | 4.25 (*dd*, *J* = 9.5, 7.0) | 69.7 | 4.35 (*dd*, *J* = 9.5, 6.5) | 69.7 |
| $H_\beta$–C(9′) | 3.79 (*dd*, *J* = 9.5, 2.5) | | 3.77 (*dd*, *J* = 9.5, 1.5) | |
| $CH_2$(10′) | 5.90 (*s*) | 100.8 | 5.94 (*s*) | 100.0 |
| 2′-MeO | | | 4.00 (*s*) | 58.5 |

relative configuration of **3** was the same as that of phrymarolin II. Thus, the structure of **3** was determined as shown in *Fig. 1*, and it was named phrymarolin IV[1]).

The eight known lignans were identified as leptostachyol acetate [6], haedoxan A [7], 1-(4,6-dimethoxy-1,3-benzodioxol-5-yl)dihydro-4-(6-methoxy-1,3-benzodioxol-5-yl)-1*H*,3*H*-furo[3,4-*c*]furan-3a(4*H*)-yl acetate [6], 4-(4,6-dimethoxy-1,3-benzodioxol-5-yl)dihydro-1-(4-methoxy-1,3-benzodioxol-5-yl)-1*H*,3*H*-furo[3,4-*c*]furan-3a(4*H*)-yl acetate [8], 4-[(4,6-dimethoxy-1,3-benzodioxol-5-yl)oxy]dihydro-1-(6-methoxy-1,3-benzodioxol-5-yl)-1*H*,3*H*-furo[3,4-*c*]furan-3a(4*H*)-yl acetate [8], leptostachyol acetate C [11], 4-(4,6-dimethoxy-1,3-benzodioxol-5-yl)dihydro-1-(6-methoxy-1,3-benzodioxol-5-yl)-1*H*,3*H*-furo[3,4-*c*]furan-3a(4*H*)-yl acetate [9], and phrymarin II [10] by comparing their m.p., UV, IR, $^1$H-, and $^{13}$C-NMR, and MS data with those reported.

The authors are grateful to *Tao Gu* for gathering plants and for assistance with the extraction of the material.

## Experiment Part

*General.* All reagents were of anal. grade. Column chromatography (CC): silica gel ($SiO_2$, 200 – 300 and 300 – 400 mesh; *Qingdao Marine Chemical Factory*, Qingdao, China), and *Lichroprep RP$_{18}$* gel (40 – 60 μm, *Merck*, Darmstadt, Germany). Anal. and prep. TLC: silica gel plates ($SiO_2$, *GF$_{254}$*; *Yantai Institute*

*of Chemical Technology*, Yantai, China). UV Spectra: *Shimadzu-UV-260* spectrophotometer, in anh. MeOH; $\lambda_{max}$ (log $\varepsilon$) in nm. IR Spectra: *Avatar-360-ESP* spectrophotometer (*Thermo Nicolet*); KBr tablets; $\tilde{\nu}$ in $cm^{-1}$. $^1H$- and $^{13}C$-NMR Spectra: *Bruker-DRX-500* spectrometer; in $CDCl_3$; $\delta$ in ppm rel. to $Me_4Si$ as internal standard, *J* in Hz. HR-ESI-MS: *Bruker-APEX-7.0-Tesla* FT-MS apparatus (*Bruker*, Germany); in *m*/*z*.

*Plant Material.* The whole plants of *P. leptostachya* were collected in May 2009 in Hanyuan, Sichuan Province, China, and identified by Dr. *Hong-Ping Deng*. A voucher specimen (PL20090523) has been deposited with the Herbarium of Materia Medica, College of Pharmaceutical Sciences, Southwest University, China.

*Extraction and Isolation.* The dried and powdered material (9 kg) was extracted with 95% EtOH by percolation, and the percolate was concentrated to give 1.1 kg of extract. The latter was then suspended in $H_2O$ (2500 ml) and partitioned in turn by extraction with petroleum ether ($3 \times 2000$ ml), AcOEt ($3 \times 2000$ ml), and BuOH ($3 \times 2000$ ml). The AcOEt-soluble part (200 g) was subjected to CC ($SiO_2$ (200–300 mesh, 2.8 kg, $12 \times 100$ cm), petroleum ether/AcOEt $100:0 \rightarrow 0:100$): *Fractions 1–11. Fr. 8* (68.5 g) was subjected to repeated CC ($SiO_2$, petroleum ether/acetone 9:1) to give three subfraction which were recrystallized to afford **1** (60 mg), leptostachyol acetate (32 mg), and haedoxan A (70 mg). *Fr. 7* was subjected to CC ($SiO_2$, petroleum ether/AcOEt 8:2) followed by prep. TLC (petroleum ether/acetone 4:1) and semi-prep. HPLC (MeOH/$H_2O$ 4:1): **2** (130 mg), 8-(acetyloxy)-6,2′,6′-trimethoxy-3,4,4′,5′-dimethylenedioxyphenyl-7,7′-dioxabicyclo[3.3.0]octane (25 mg), 8-(acetyloxy)-2,2′,6′-trimethoxy-3,4,4′,5′-dimethylenedioxyphenyl-7,7′-dioxabicyclo[3.3.0]octane (8 mg), and 8-(acetyloxy)-2,6-dimethoxyphrymarolin II (13 mg). The petroleum ether-soluble part (200 g) was subjected to CC ($SiO_2$, petroleum ether/AcOEt $100:0 \rightarrow 0:100$): *Frs. I–VIII. Fr. VII* was subjected to repeated CC ($SiO_2$, petroleum ether/acetone 95:5) then to prep. TLC (petroleum ether/acetone 9:1): **3** (12 mg), leptostachyol acetate C (8 mg), 8-(acetyloxy)-2,6,2′-trimethoxy-3,4,4′,5′-dimethylenedioxyphenyl-7,7′-dioxabicyclo[3.3.0]octane (27 mg), and 8-(acetyloxy)-2,2′-dimethoxy-4,5,4′,5′-dimethylenedioxyphenyl-7,7′-dioxabicyclo[3.3.0]octane (33 mg).

rel-*(1*R*,3a*R*,4*S*,6a*S*)-1-[(2*S*,3*S*)-2,3-Dihydro-2-(4-hydroxy-3-methoxyphenyl)-3-(hydroxymethyl)-7-methoxy-1,4-benzodioxin-6-yl]-4-[(4,6-dimethoxy-1,3-benzodioxol-5-yl)oxy]dihydro-1*H,*3*H*-furo[3,4-c]furan-3a(4*H*)-ol* (**1**). Colorless prism crystals (MeOH). M.p. 158–159°. $[\alpha]_D^{25} = +125$ ($c = 0.32$, EtOH/$CH_2Cl_2$). UV (MeOH): 292 (4.06), 234 (4.07). IR (KBr): 3480, 3000, 2940, 2890, 1600, 1500, 1445, 1434, 1330, 980, 930. $^1H$-NMR (600 MHz, $(D_6)$DMSO)[1]): 7.05 (*s*, H–C(5′)); 6.99 (*d*, *J* = 1.8, H–C(2″)); 6.85 (*dd*, *J* = 8.0, 1.8, H–C(6″)); 6.80 (*d*, *J* = 8.0, H–C(5″)); 6.60 (*s*, H–C(2′)); 6.56 (*s*, H–C(5)); 5.93 (*s*, $CH_2$ (10)); 5.31 (OH); 5.13 (*s*, H–C(7)); 4.88 (*d*, *J* = 7.8, H–C(7″)); 4.76 (*d*, *J* = 5.8, H–C(7′)); 4.49 (*q*, *J* = 7.9, $H_\alpha$–C(9′)); 4.12 (br. *d*, *J* = 9.3, $H_\alpha$–C(9)); 3.99–4.01 (*m*, H–C(8″)); 3.89 (*s*, MeO–C(3″)); 3.82 (*dd*, *J* = 9.0, 2.4, $H_\beta$–C(9′)); 3.78 (*s*, MeO–C(2)); 3.71 (*s*, MeO–C(6)); 3.71 (*s*, MeO–(6′)), 3.52 (br. *d*, *J* = 12.0, $H_a$–C(9″)); 3.50–3.52 (*m*, $H_b$–C(9)); 3.32 (*dd*, *J* = 12.0, 4.7, $H_\beta$–C(9″)); 2.43–2.45 (*m*, H–C(8′)). $^{13}C$-NMR (150 MHz, $(D_6)$DMSO)[1]): 150.4 (C(6′)); 148.0 (C(6)); 147.5 (C(4″)); 147.4 (C(3″)); 144.6 (C(4)); 143.2 (C(4′)); 138.6 (C(2)); 137.1 (C(3′)); 131.7 (C(3)); 131.4 (C(1)); 128.1 (C(1″)); 123.5 (C(1′)); 120.9 (C(6″)); 115.8 (C(5″)); 114.5 (C(5′)); 112.1 (C(2″)); 104.3 (C(7)); 101.5 (C(1)); 100.8 (C(2′)); 92.1 (C(8)); 91.2 (C(5)); 83.9 (C(7′)); 78.3 (C(8″)); 78.2 (C(9)); 76.5 (C(7″)); 70.9 (C(9′)); 60.8 (C(9″)); 60.3 (*Me*O–(3″)); 57.4 (*Me*O–(6)); 57.4 (C(8′)); 57.4 (*Me*O–(6′)); 56.4 (MeO–(2)).

rel-*(1*R*,3a*R*,4*S*,6a*S*)-4-(1,3-Benzodioxol-5-yloxy)dihydro-1-(4-hydroxy-1,3-benzodioxol-5-yl)-1*H*,3*H*-furo[3,4-c]furan-3a(4*H*)-ol* (**2**): Colorless acicular crystals ($CHCl_3$/MeOH). M.p. 160–161°. $[\alpha]_D^{25} = +155$ ($c = 3.6$, dioxane). UV (MeOH): 297 (4.06), 236 (3.97). IR (KBr): 3000, 2760, 1743, 930, 1630–1650. $^1H$- and $^{13}C$-NMR: *Table.*

rel-*(1*R*,3a*R*,4*S*,6a*S*)-4-(1,3-Benzodioxol-5-yloxy)dihydro-1-(4-methoxy-1,3-benzodioxol-5-yl)-1*H*,3*H*-furo[3,4-c]furan-3a(4*H*)-ol* (**3**): Colorless acicular crystals (AcOEt). M.p. 160–161°. $[\alpha]_D^{25} = +154°$ ($c = 2.5$, dioxane). UV (MeOH): 297 (4.07), 234 (3.96). IR (KBr): 3000, 2762, 1745, 930, 1630–1650. $^1H$- and $^{13}C$-NMR: *Table.*

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