Three New Compounds from *Kadsura longipedunculata*

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From the leaves and stems of *Kadsura longipedunculata* FINET et GAGNEP (Schisandraceae), a new triterpenoid, schisanlactone E (**1**), and two new lignans, 9-(*β*-D-glucopyranosyloxy)-3′-methoxy-3,4-(methylenedioxy)-7,9′-epoxylignan-4′-ol (**3**) and 3-methoxy-3′,4′-(methylenedioxy)-9,9′-epoxylignan-4,7′-diol (**4**), together with seven known compounds, were isolated. Their structures were elucidated by analysis of spectroscopic evidence including extensive 2D-NMR data.

Introduction. – The genus *Kadsura* (Schisandraceae) is closely related to *Schisandra*, and many of its species are used as a folk medicine in Taiwan, Japan, and mainland China, mostly as a substitute for *Schisandra* [1]. It has been reported that some species of this genus contain dibenzocyclooctadienlignans, lanostane, and cycloartane triterpenoids with pharmacological properties, including anti-HBeAg, antihepatotoxic, antitumor, anti-HIV, and anti-lipid-peroxidative activities [2–9]. *Kadsura longipedunculata* FINET et GAGNEP is a climbing plant widely distributed in the southern part of China. It has been used in folk medicine for the treatment of rheumatoid arthritis as well as gastric and duodenal ulcers [10][11]. Previous studies on the leaves and stems of *Kadsura longipedunculata* have resulted in the isolation of two series of novel triterpenoids with unique skeletons, kadlongilactones A and B and longipedlactones A–I [12][13]. Futher chemical analysis of this plant led to the isolation of three new compounds, schisanlactone E (**1**), 9-(*β*-D-glucopyranosyloxy)-3′-methoxy-3,4-(methylenedioxy)-7,9′-epoxylignan-4′-ol (**3**), and 3-methoxy-3′,4′-(methylenedioxy)-9,9′-epoxylignan-4,7′-diol (**4**), along with seven known compounds, *i.e.*, *β*-sitosterol [14], daucosterol [14], schizandronic acid [15], parkeol [16], mangiferolic acid [17], licarin A [18], and schizandriside [19] from the AcOEt extract of the Me_2CO extract. This paper describes the isolation and structural elucidation of these new compounds.

Results and Discussion. – Compound **1** was obtained as white powder. Its molecular formula $C_{30}H_{44}O_4$ was determined by high-resolution MS ($[M-H]^-$ at m/z 467.3156), in combination with ^{1}H- and ^{13}C-NMR data (*Table 1*), indicating 9 degrees of unsaturation. The IR spectrum showed the presence of an OH group (3435 cm^{-1}), a carbonyl group (1724 cm^{-1}), and an *α*,*β*-unsaturated *δ*-lactone group (1707 cm^{-1}).

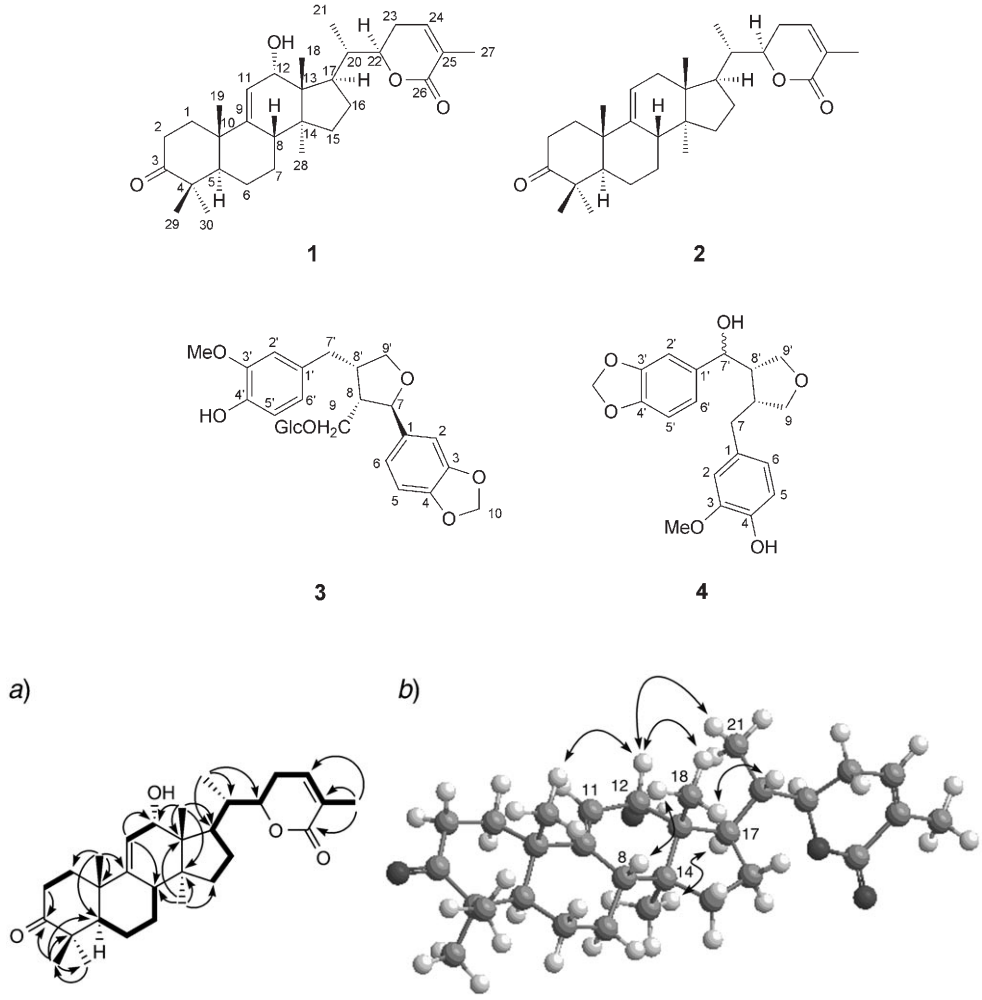

Fig. 1. a) *$^1H,^1H$-COSY* (▬) *and Key HMBC* (H → C) *correlations of* **1**. b) *Key ROESY* (↔) *correlations of* **1**.

Detailed analysis of the HSQC, HMBC, and ROESY data (*Fig. 1*) established the structure of **1** as (12α)-12-hydroxyschisanlactone D[1]), **1** was named schisanlactone E.

Analysis of the 1D-NMR data and HSQC spectra revealed that **1** contained two carbonyl C-atoms (including an α,β-unsaturated lactone), six quaternary C-atoms (including two olefinic C-atoms), and eight CH (including two olefinic and two oxygenated ones), seven CH_2, and seven Me groups. Apart from two C=C bonds and two C=O groups, the remaining elements of the unsaturation in **1** were assumed to be due to the presence of five rings. Comparison of the ^{1}H- and ^{13}C-NMR data with those of the known schisanlactone D (**2**) [20] showed the presence of the same pentacyclic triterpenoid skeleton,

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

Table 1. *^{1}H- and ^{13}C-NMR Data* (400 MHz, (D_5)pyridine) *of* **1** *and* **2**. δ in ppm, J in Hz.

	1		**2**
	δ(H)	δ(C)	δ(C)
CH_2(1)	1.68–1.71 (*m*, H_α), 2.00–2.04 (*m*, H_β)	36.8 (*t*)	36.8 (*t*)
CH_2(2)	2.34–2.40 (*m*, H_α), 2.68–2.75 (*m*, H_β)	34.9 (*t*)	34.9 (*t*)
C(3)		215.5 (*s*)	216.7 (*s*)
C(4)		47.7 (*s*)	46.7 (*s*)
H–C(5)	1.39–1.43 (overlapped)	53.4 (*d*)	53.6 (*d*)
CH_2(6)	1.53–1.56 (*m*)	22.6 (*t*)	22.6 (*t*)
CH_2(7)	1.36–1.39 (overlapped, H_α), 1.63–1.67 (*m*, H_β)	28.6 (*t*)	27.8 (*t*)
H–C(8)	2.22 (*dd*, J = 4.9, 12.7)	42.2 (*d*)	42.0 (*d*)
C(9)		148.6 (*s*)	147.4 (*s*)
C(10)		39.3 (*s*)	39.2 (*s*)
H–C(11)	5.64 (*d*, J = 4.4)	120.4 (*d*)	116.1 (*d*)
H–C(12) or CH_2(12)	4.07 (*s*)	73.0 (*d*)	37.2 (*t*)
C(13)		48.7 (*s*)	44.9 (*s*)
C(14)		46.0 (*s*)	47.7 (*s*)
CH_2(15)	1.37–1.41 (overlapped, H_α), 1.46–1.49 (*m*, H_β)	35.5 (*t*)	34.0 (*t*)
CH_2(16)	1.78–1.85 (*m*, H_α), 1.34–1.38 (overlapped, H_β)	26.7 (*t*)	26.9 (*t*)
H–C(17)	2.61 (*dd*, J = 9.3, 20.6)	40.2 (*d*)	47.0 (*d*)
Me(18)	0.70 (*s*)	15.0 (*q*)	14.3 (*q*)
Me(19)	1.16 (*s*)	22.1 (*q*)	22.0 (*q*)
H–C(20)	2.04–2.08 (overlapped)	39.9 (*d*)	39.2 (*d*)
Me(21)	1.32 (*d*, J = 6.4)	12.3 (*q*)	13.3 (*q*)
H–C(22)	4.55 (*dd*, J = 3.4, 6.9)	80.9 (*d*)	80.6 (*d*)
CH_2(23)	2.08–2.12 (overlapped, H_α), 2.28–2.31 (*m*, H_β)	23.9 (*t*)	23.6 (*t*)
H–C(24)	6.48 (*d*, J = 5.9)	140.3 (*d*)	139.3 (*d*)
C(25)		128.0 (*s*)	128.5 (*s*)
C(26)		166.4 (*s*)	166.5 (*s*)
Me(27)	1.93 (*s*)	17.2 (*q*)	16.9 (*q*)
Me(28)	1.19 (*s*)	20.7 (*q*)	18.6 (*q*)
Me(29)	1.14 (*s*)	26.0 (*q*)	25.7 (*q*)
Me(30)	1.04 (*s*)	22.1 (*q*)	21.8 (*q*)
OH–C(12)	5.74 (br. *s*)		

except for the presence of an oxygenated CH group (δ(C) 73.0) in **1** and the absence of a CH_2 group assigned to C(12) (δ(C) 37.2) of **2**, indicating that CH_2(12) of **2** was replaced by an oxygenated CH(12) in **1**. This assignment was in accord with the observation of significant downfield shifts of the C(11), C(12), and C(13) signals from δ(C) 116.1, 37.2, and 44.9 in **2** to δ(C) 120.4, 73.0, and 48.7 in **1**, respectively. This was further confirmed by the HMBC correlations from H–C(11) (δ(H) 5.64) and Me(18) (δ(H) 0.70) to C(12), and by the ^{1}H,^{1}H-COSY correlation H–C(11)/H–C(12) (*Fig. 1,a*). The α-configuration of OH–C(12) was deduced from the ROESY correlations (H–C(11)/H–C(12), H–C(12)/Me_β(18), and H_β–C(8)/Me_β(18)) (*Fig. 1,b*), which was also supported by the upfield shift of C(17) ($\Delta\delta$(C) = –6.8) caused by the *syn*-γ effect between OH–C(12) and H_α–C(17) [21].

A molecular formula of $C_{26}H_{32}O_{11}$ was established for compound **3** from its HR-ESI-MS (*m*/*z* 543.1843 for $[M + Na]^+$). The ^{1}H-NMR (*Table 2*) disclosed a substituted-diarene epoxylignan skeleton [22]. The structure and relative configuration of **3** were established from its ^{1}H- and ^{13}C-NMR (*Table 2*), ^{1}H,^{1}H-COSY, HSQC, HMBC, and

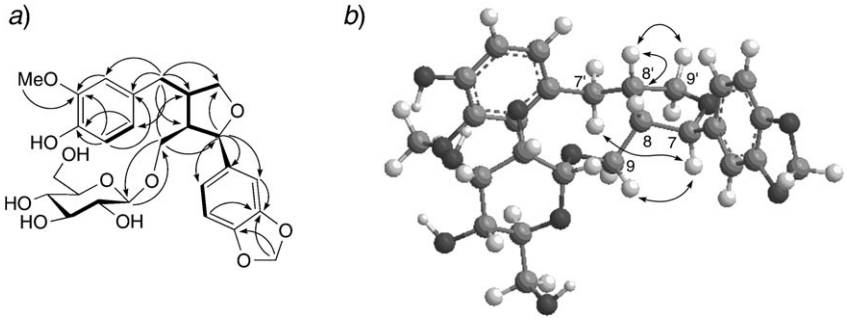

Fig. 2. a) *$^1H,^1H$-COSY* (▬) *and Key HMBC* (H → C) *correlations of* **3**. b) *Key ROESY* (↔) *correlations of* **3**.

ROESY data (*Fig. 2*) as 9-(*β*-D-glucopyranosyloxy)-3′-methoxy-3,4-(methylenedioxy)-7,9′-epoxylignan-4′-ol[1]).

The ^{1}H-NMR of **3** showed the presence of only one downfield-shifted benzylic CH group (*δ*(H) 4.82–4.85, H–C(7[1])), one benzylic CH_2 group (*δ*(H) 2.47–2.51 and 2.92, CH_2(7′)), a primary OCH_2 group (*δ*(H) 3.72–3.77 and 4.04, CH_2(9)), and two aliphatic CH groups (*δ*(H) 2.43–2.47, H–C(8); *δ*(H) 2.70–2.79, H–C(8′)). Both arene moieties showed a 1,3,4-trisubstitution pattern (see *Table 2*). The ^{13}C-NMR data (*Table 2*) and DEPT experiments clearly indicated the basic C_{18}-lignan skeleton of the 7,9′-monoepoxy type [23] and a sugar moiety (*δ*(C) 62.8, 71.7, 75.2, 78.0, 78.1, and 104.7). In the HMBC experiment (*Fig. 2,a*), correlations from H–C(6′) to C(4′), from H–C(5′) to C(4′) and C(3′), from H–C(2′) to C(3′), from MeO to C(3′), and from CH_2(10) to C(3) and C(4), determined that the OH, MeO, and OCH_2O groups were located at C(4′), C(3′), and C(3),C(4), respectively. ^{1}H-NMR Coupling constants and ^{13}C-NMR chemical-shift data indicated that the monosaccharide unit was a glucose, corresponding with the fragment ion at *m/z* 359 $[M - \text{Glc} + \text{H}]^+$ in the FAB-MS experiment, and the *β*-configuration was determined by the *J*(H,H) of the anomeric proton H–C(1″) (*δ* 4.29 (*d*, *J* = 7.7)). The HMBC correlation of H–C(1″) with C(9) required that the glucose be attached to C(9). The ROESY data (*Fig. 2,b*) established the *cis*-configuration of H–C(8) and H–C(8′) and the *trans*-orientation of H–C(7) and H–C(8), which was in accordance with precedented cases [22][23].

The HR-ESI-MS of compound **4** gave a quasi-molecular ion peak at *m/z* 381.1311 ($[M + \text{Na}]^+$), corresponding to the molecular formula $C_{20}H_{22}O_6$, requiring 10 degrees of unsaturation. The ^{1}H- and ^{13}C-NMR spectra (*Table 2*) suggested that **4** might be a 9,9′-epoxydibenzylbutane lignan [24] with an MeO, an OCH_2O, and two OH groups. Its functional groups were also deduced from IR spectral bands at 3426 (OH), 1609 and 1515 (aromatic) cm^{-1}. The relative configuration of **4** was shown to be as depicted in *Fig. 3,b*, by the correlations observed in a ROESY experiment. Finally, the structure of **4** was established as 3-methoxy-3′,4′-(methylenedioxy)-9,9′-epoxylignane-4,7-diol[1]).

In the ^{1}H-NMR spectrum of **4**, two *ABC* systems at *δ*(H) 6.63 (*dd*, *J* = 1.5, 8.1, H–C(6)), 6.70 (*d*, *J* = 8.1, H–C(5)), and 6.77–6.82 (overlapped, H–C(2)), and at *δ*(H) 6.75 (*dd*, *J* = 1.5, 7.7, H–C(5′)), 6.75–6.80 (overlapped, H–C(6′)), and 6.83 (*d*, *J* = 1.5, H–C(2′)), respectively, were typical of the substituted arene moieties. The functional groups were assigned on the basis of HMBC and ^{1}H,^{1}H-COSY studies (*Fig. 3,a*). Thus, the correlations of an OCH_2O proton to C(3′) and C(4′), of the aromatic proton H–C(6) to C(4), and of the OCH proton H–C(7′) to C(1′), C(2′), C(6′), C(8′), C(9′), and C(8), as well as the correlation of MeO to C(3), confirmed the positions of the substituents without doubt. ROESY

Table 2. *^{1}H- and ^{13}C-NMR Data* (400 MHz, CD_3OD) *of* **3** and **4**[1]). δ in ppm, J in Hz.

	3		**4**	
	δ(H)	δ(C)	δ(H)	δ(C)
C(1)		138.4 (*s*)		133.5 (*s*)
H–C(2)	6.85 (*d*, J = 1.7)	107.4 (*d*)	6.77–6.82 (overlapped)	113.5 (*d*)
C(3)		149.2 (*s*)		149.0 (*s*)
C(4)		148.4 (*s*)		145.9 (*s*)
H–C(5)	6.74 (*d*, J = 8.2)	108.8 (*d*)	6.70 (*d*, J = 8.1)	116.2 (*d*)
H–C(6)	6.81 (*dd*, J = 1.7, 8.2)	120.4 (*d*)	6.63 (*dd*, J = 1.5, 8.1)	122.2 (*d*)
H–C(7) or CH_2(7)	4.82–4.85 (overlapped)	84.1 (*d*)	2.48 (*dd*, J = 11.4, 13.6, H_α), 2.90 (*dd*, J = 4.8, 13.6, H_β)	33.6 (*t*)
H–C(8)	2.43–2.47 (overlapped)	51.8 (*d*)	2.71 (*ddd*, J = 6.6, 12.1 18.0)	43.9 (*d*)
CH_2(9)	3.72–3.77 (overlapped, H_α), 4.04 (*dd*, J = 6.6, 9.9, H_β)	68.4 (*t*)	3.71 (*dd*, J = 5.9, 8.3, H_α), 3.97 (*dd*, J = 6.6, 8.3, H_β)	73.6 (*t*)
C(1′)		133.6 (*s*)		138.5 (*s*)
H–C(2′)	6.85 (*d*, J = 1.7)	113.4 (*d*)	6.83 (*d*, J = 1.5)	107.3 (*d*)
C(3′)		149.0 (*s*)		148.4 (*s*)
C(4′)		145.8 (*s*)		149.3 (*s*)
H–C(5′)	6.70 (*d*, J = 8.2)	116.2 (*d*)	6.75 (*dd*, J = 1.5, 7.7)	108.8 (*d*)
H–C(6′)	6.64 (*dd*, J = 1.7, 8.2)	122.2 (*d*)	6.75–6.80 (overlapped)	120.4 (*d*)
CH_2(7′) or H–C(7′)	2.47–2.51 (overlapped, H_α), 2.92 (*dd*, J = 4.9, 13.7, H_β)	33.8 (*t*)	4.75 (*d*, J = 6.6)	84.0 (*d*)
H–C(8′)	2.70–2.79 (*m*)	43.8 (*d*)	2.32 (*dd*, J = 7.0, 14.0)	54.2 (*d*)
CH_2(9′)	3.71–2.76 (overlapped, H_α), 3.97 (*dd*, J = 6.6, 8.2, H_β)	73.7 (*t*)	3.79–3.84 (overlapped, H_α), 3.62 (*dd*, J = 6.6, 11.0, H_β)	60.5 (*t*)
H–C(1″)	4.29 (*d*, J = 7.7)	104.7 (*d*)		
H–C2(″)	3.21 (*d*, J = 7.7)	75.2 (*d*)		
H–C(3″)	3.36 (*d*, J = 8.2)	78.1 (*d*)		
H–C(4″)	3.25–2.29 (overlapped)	71.7 (*d*)		
H–C(5″)	3.27–3.32 (overlapped)	78.0 (*d*)		
CH_2(6″)	3.86 (*dd*, J = 2.2, 12.1, H_α), 3.67 (*dd*, 5.5, J = 12.1, H_β)	62.8 (*t*)		
MeO–C(3′)	3.82 (*s*)	56.5 (*s*)	3.82 (*s*)	56.4 (*s*)
OCH_2O	5.90 (*s*)	102.3 (*t*)	5.91 (*s*)	102.3 (*t*)

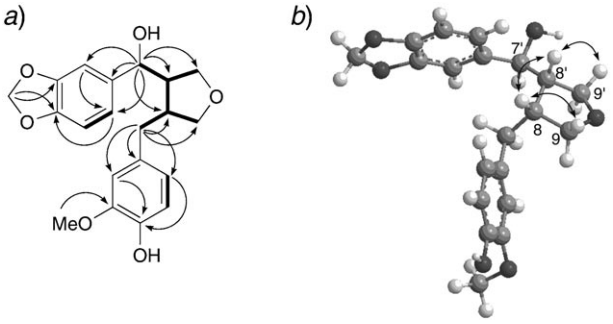

Fig. 3. a) *^{1}H,^{1}H-COSY* (▬) *and Key HMBC* (H → C) *correlations of* **4**. b) *Key ROESY* (↔) *correlations of* **4**.

correlations were observed between H–C(8′) and H–C(8) as well as H_β–C(9′), and H–C(8) gave a ROESY correlation to H_β–C(9), which confirmed that H–C(8) and H–C(8′) were in β-orientation. Because of free rotation around the connecting bond between C(7′) and C(8′), we could not determine the relative configuration at C(7′).

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Experimental Part

General. Column chromatography (CC) and TLC: silica gel (200–300 mesh) from *Qingdao Marine Chemical Factory*, Quingdao, People's Republic of China. Melting points: *XRC-1* micro melting point apparatus; uncorrected. Optical rotations: *Jasco DIP-370* digital polarimeter. IR Spectra: *Bio-Rad FtS-135* spectrophotometer; KBr pellets; in cm^{-1}. UV Spectra: *UV-210A* spectrometer; λ_{max}(log ε) in nm. 1D- and 2D-NMR Spectra: *Bruker DRX-500* instruments; $SiMe_4$ as an internal standard. MS: *VG Auto-Spec-3000* spectrometer; in *m/z* (rel. %).

Plant Material. The leaves and stems of *K. longipedunculata* were collected in the Erlang mountain region of Sichuan Province, China, in August 2004, and identified by Prof. *Xi-Wen Li*, Kunming Institute of Botany. A voucher specimen has been deposited in the Herbarium of the Kunming Institute of Botany, Chinese Academy of Sciences.

Extraction and Isolation. The air-dried and powdered stems and leaves (11 kg) of *K. longipedunculata* were extracted with 70% aq. Me_2CO (4 × 30 l) at r.t. to yield an extract, which was successively extracted with petroleum ether and AcOEt. The AcOEt extract (300 g) was subjected to CC (silica gel (1.5 kg; 200–300 mesh), $CHCl_3$/Me_2CO gradient 9:1, 8:2, 7:3, 6:4, 5:5): *Fractions 1–5*. Compound **3** (13 mg) was obtained from *Fr. 1*. Compounds **1** (8 mg) and **4** (3 mg) were obtained from *Fr. 3* after repeated CC (silica gel ($CHCl_3$/^{i}PrOH), followed by semiprep. HPLC (*Agilent-1100* HPLC system, *Zorbax SB-C-18* (*Agilent*), 9.4 mm × 25 cm, MeOH/H_2O).

*(12α,22*R*)-12,22-Dihydroxy-3-oxolanosta-9(11),24-dien-26-oic Acid δ-Lactone* (**1**): White powder. $[\alpha]_D^{25.8} = +175.0$ ($c = 0.28$, pyridine). UV (MeOH): 370 (2.17), 205 (4.52), 191 (4.08). IR (KBr): 3435, 2972, 2949, 2933, 2866, 1724, 1707, 1651, 1632, 1122, 1028. NMR: see *Table 1*. FAB-MS (neg.): 559 (8, $[M + Gly - H]^-$), 467 (100, $[M - H]^-$). HR-ESI-MS (pos.): 467.3156 ($C_{30}H_{43}O_4^-$, $[M - H]^-$; calc. 467.3161).

*{(2*R**,3*S**)-2-(1,3-Benzodioxol-5-yl)tetrahydro-4-[(4-hydroxy-3-methoxyphenyl)methyl]furan-3-yl}-methyl β-*D*-Glucopyranoside* (**3**): Pale yellow powder. $[\alpha]_D^{15.0} = -22.4$ ($c = 0.98$, pyridine). UV (MeOH): 350 (3.24), 283 (4.15), 203 (5.15). IR (KBr): 3417*s* (br.), 2927, 2886, 1713, 1607, 1516, 1489, 1445, 1376, 1271, 1248, 1157, 1124, 1098, 1076, 1036. NMR: see *Table 2*. FAB-MS (pos.): 613 (5, $[M + Gly + H]^+$), 521 (4, $[M + H]^+$), 274 (100). HR-ESI-MS (pos.): 543.1843 ($C_{26}H_{32}O_{11}Na^+$, $[M + Na]^+$; calc. 543.1842).

*α-{(3*S**,4*R**)-Tetrahydro-4-[(4-hydroxy-3-methoxyphenyl)methyl]furan-3-yl}-1,3-benzodioxole-5-methanol* (**4**): Pale yellow powder. $[\alpha]_D^{15.3} = +1.6$ ($c = 0.32$, MeOH). UV (MeOH): 283 (4.00), 203 (4.93). IR (KBr): 3426, 2932, 1709, 1609, 1515, 1489, 1445, 1369, 1272, 1247, 1208, 1124, 1098, 1038. NMR: *Table 2*. FAB-MS (pos.): 543 (2, $[M + 2\,Gly + H]^+$), 451 (7, $[M + Gly + H]^+$), 358 (44, M^+), 331 (41), 237 (40), 219 (72), 137 (100). HR-ESI-MS (pos.): 381.1311 ($C_{20}H_{22}O_6Na^+$, $[M + Na]^+$; calc. 381.1314).

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