Phenylpropanoids from *Daphne feddei* and Their Inhibitory Activities against NO Production

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Chemical examination of the methanolic extract from the stem bark of *Daphne feddei* led to the isolation of five new phenylpropanoids, 4,4′-dihydroxy-3,3′-dimethoxy-9-butoxy-9,9′-epoxylignan (**1**), 4,4′-dihydroxy-3,3′-dimethoxy-9-ethoxy-9,9′-epoxylignan (**2**), daphneresinol (**3**), armaosigenin (**4**), and isocubebin (**5**), together with 33 known phenylpropanoids. All 38 compounds were isolated for the first time from *D. feddei*. All compounds were tested for inhibitory activity against LPS-induced NO production in RAW 264.7 macrophages. Compounds **2**, **8**, **9**, **12**, **13**, and **15** showed potent inhibitory activities against the production of NO with IC_{50} values of 0.091, 0.047, 0.005, 0.088, 0.004, and 0.074 μM/mL, respectively.

Daphne feddei levl. is a common evergreen shrub native to Yunnan, Sichuan, and Guizhou Provinces in China. Its stem bark is used for the treatment of injuries from falls and bruises in folk medicine.[1] In a previous chemical investigation of *D. feddei*, the occurrence of four diterpenes has been reported.[2] In the course of our study on chemical constituents of thymelaeaceous plants,[3−5] five new phenylpropanoids, 4,4′-dihydroxy-3,3′-dimethoxy-9-butoxy-9,9′-epoxylignan (**1**), 4,4′-dihydroxy-3,3′-dimethoxy-9-ethoxy-9,9′-epoxylignan (**2**), daphneresinol (**3**), armaosigenin (**4**), and isocubebin (**5**), together with 33 known phenylpropanoids, were isolated from the title plant. This paper deals with the structural elucidation of the five new compounds and inhibitory activities of all 38 compounds against LPS-induced NO production in macrophages.

Results and Discussion

The EtOAc-soluble fraction of the methanolic extract of the stem bark of *D. feddei* was subjected to column chromatography over silica gel, RP-18, and Sephadex LH-20 in various solvent systems to afford five new phenylpropanoids, 4,4′-dihydroxy-3,3′-dimethoxy-9-butoxy-9,9′-epoxylignan (**1**), 4,4′-dihydroxy-3,3′-dimethoxy-9-ethoxy-9,9′-epoxylignan (**2**), daphneresinol (**3**), armaosigenin (**4**), and isocubebin (**5**), together with 33 known phenylpropanoids. By comparing physical and spectroscopic data with reported data, the 33 known compounds were identified as (−)-pinoresinol (**6**),[6] (+)-medioresinol (**7**),[7] syringaresinol (**8**),[8] matairesinol (**9**),[7] arctigenin (**10**),[9] wikstromol (**11**),[10] (8*R*,8′*R*,9*R*)-4,4′,9-trihydroxy-3,3′-dimethoxy-9,9′-epoxylignan (**12**), (8*R*,8′*R*,9*S*)-4,4′,9-trihydroxy-3,3′-dimethoxy-9,9′-epoxylignan (**13**),[11] (+)-lariciresinol (**14**),[12] (+)-isolariciresinol (**15**),[13] secoisolariciresinol (**16**),[14] (+)-neoolivil (**17**),[15] eduesmine (**18**),[16] (−)-pinoresinol 4-*O*-β-D-glucopyranoside (**19**),[17] (−)-pinoresinol-4,4′-di-*O*-β-D-glucopyranoside (**20**),[18] eucommin A (**21**),[19] arctiin (**22**),[20] acanthoside B (**23**),[10] liriodendrin (**24**),[21] matairesinoside (**25**),[20] (−)-nortracheloside (**26**),[22] (+)-lariciresinol-4-*O*-β-D-glucopyranoside (**27**),[23] syringin (**28**),[8] sinapaldehyde (**29**),[24] coniferaldehyde (**30**),[25] methyl caffeate (**31**),[26] evofolin B (**32**),[27] guaicylglycerol (**33**),[28] isodaphneticin (**34**),[29] isodaphneticin 4″-*O*-α-D-glucopyranoside (**35**),[30] daphneticin (**36**),[31] demethoxydaphneticin (**37**),[29] and daphneticin 4″-*O*-β-D-glucopyranoside (**38**).[32] The structures of the new compounds were determined by spectroscopic methods.

Compound **1** was assigned the molecular formula $C_{24}H_{32}O_6$ by HRESIMS ($[M + Na]^+$ at *m*/*z* 439.2096). In the ^1H NMR spectrum, three aromatic protons in an ABX pattern (δ_H 6.66, d, J = 7.8 Hz, 6.53, dd, J = 1.8, 7.8 Hz, and 6.67, d, J = 1.8 Hz) and a singlet at δ_H 3.72 (3H) indicated the existence of a 4-hydroxy-3-methoxyphenyl ring. Three additional aromatic protons in an ABX pattern (δ_H 6.65, d, J = 7.8 Hz, 6.48, dd, J = 1.8, 7.8 Hz, and 6.61, d, J = 1.8 Hz) and a singlet at δ_H 3.71 (3H) indicated the existence of a second 4-hydroxy-3-methoxyphenyl ring. The interpretation of the ^1H and ^{13}C NMR data of **1**, together with the analysis of the $^1H-^1H$ COSY and HSQC spectra, allowed the assignment of a butoxy group due to the signals at δ_H 3.21 and 3.48 (each 1H, m, H-1″a and H-1″b), 1.39 (2H, m, H-2″a and H-2″b), 1.23 (2H, m, H-3″a and H-3″b), and 0.83 (3H, t, J = 7.2 Hz, H-4″). The NMR resonances were very similar to those of 4,4′,9-trihydroxy-3,3′-dimethoxy-9,9′-epoxylignan,[33] except for the signals due to a butoxy group. Furthermore, the proton resonances at δ_H 3.21 and 3.48 (each 1H, m, H-1′a and H-1′b) showed correlation with a carbon signal at δ_C 108.1 (C-9) in the HMBC spectrum, which indicated the structure of compound **1** to be 4,4′-dihydroxy-3,3′-dimethoxy-9-butoxy-9,9′-epoxylignan. The relative configuration of **1** was obtained through analysis of coupling constants and the NOESY spectrum. H-9, H-8, and H-8′ were determined to be α-, α-, and β-oriented, respectively, on the basis of the NOE correlation of H-9/H-8 and the small coupling constant (J = 1.8 Hz) between H-9 and H-8. Thus, compound **1** was deduced as 4,4′-dihydroxy-3,3′-dimethoxy-9-butoxy-9,9′-epoxylignan.

Compound **2** had a molecular formula of $C_{22}H_{28}O_6$, as deduced by HRESIMS ($[M + Na]^+$ at *m*/*z* 411.1786). The NMR data of **2** were very similar to those of compound **1**, except for signals due to an ethoxy group at δ_H 3.39 and 3.71 (each 1H, m, H-1′a and H-1′b) and 1.17 (3H, t, J = 6.6, H-2″) instead a butoxy group in **1**. The relative configuration of **2** was identical with **1** on the basis of the NOE correlation of H-9/H-8 and the small coupling constant (J = 1.2 Hz) between H-9 and H-8. Thus, compound **2** was determined as 4,4′-dihydroxy-3,3′-dimethoxy-9-ethoxy-9,9′-epoxylignan. However, EtOAc and *n*-butanol may be the sources of the *O*-butyl and *O*-ethyl units in compounds **1** and **2**, for they had been used in the process of extraction and isolation. This needs to be established.

Compound **3** was assigned the molecular formula $C_{20}H_{26}O_7$ by HRESIMS ($[M]^+$ at *m*/*z* 378.1678). In the ^1H NMR spectrum, three aromatic protons in an ABX pattern (δ_H 6.70, d, J = 8.4 Hz, 6.81, dd, J = 1.8, 8.4 Hz, and 6.93, d, J = 1.8 Hz) and a singlet at δ_H 3.83 (3H) indicated the existence of a 4-hydroxy-3-methoxyphenyl ring. Three additional aromatic protons with an ABX pattern (δ_H 6.69, d, J = 8.4 Hz, 6.80, dd, J = 1.8, 8.4 Hz, and 6.93, d, J = 1.8

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Table 1. ^{13}C and ^{1}H NMR Spectroscopic Data of Compounds **1**−**3** (**1** in DMSO-d_6,**2** in CD_3Cl, **3** in CD_3OD)

	1		**2**		**3**	
position	δ_C	δ_H (mult. *J* Hz)	δ_C	δ_H (mult. *J* Hz)	δ_C	δ_H (mult. *J* Hz)
1	131.3		132.5		137.8	
2	112.9	6.67 (d, 1.8)	111.2	6.53 (d, 1.8)	113.3	6.93 (d, 1.8)
3	147.4		146.4		149.1	
4	144.7		143.9		145.9	
5	115.3	6.66 (d, 7.8)	114.1	6.80 (d, 7.8)	116.4	6.70 (d, 8.4)
6	120.9	6.53 (dd, 1.8, 7.8)	121.6	6.61 (dd, 1.8, 7.8)	121.9	6.81 (dd, 1.8, 8.4)
7	37.9	2.48 (m)	39.2	2.56 (m)	52.2	3.97 (d, 12.0)
8	52.2	2.01 (m)	52.2	2.15 (m)	45.1	2.62 (m)
9	108.1	4.73 (d, 1.8)	108.6	4.84 (d, 1.2)	44.0	1.95 (m)
10					63.8	3.70 (m)
11					59.9	3.62 (m)
						3.68 (m)
12					60.4	3.39 (m)
						3.54 (m)
1′	130.7		131.6		137.2	
2′	112.6	6.61 (d, 1.8)	111.0	6.43 (d, 1.8)	113.1	6.93 (d, 1.8)
3′	147.4		146.3		148.9	
4′	144.6		143.8		145.8	
5′	115.2	6.65 (d, 7.8)	114.0	6.77 (d, 7.8)	116.3	6.69 (d, 8.4)
6′	120.5	6.48 (dd, 1.8, 7.8)	121.2	6.55 (dd, 1.8, 7.8)	121.5	6.80 (dd, 1.8, 8.4)
7′	37.6	2.39 (m)	38.5	2.56 (m)		
		2.48 (m)		2.42 (dd, 15.6, 8.4)		
8′	45.7	2.07 (m)	45.7	2.14 (m)		
9′	74.2	3.48 (m)	72.1	4.00 (dd, 7.2, 8.4)		
		3.82 (t, 7.8)		3.67 (t, 7.8)		
1″	66.3	3.21 (m)	62.8	3.71 (m)		
		3.48 (m)		3.39 (m)		
2″	31.2	1.39 (m)	15.3	1.17 (t, 6.6)		
3″	18.8	1.23 (m)				
4″	13.6	0.83 (t, 7.2)				
3-OCH_3	55.5	3.72 (s)	55.7	3.81 (s)	56.6	3.83 (s)
3′-OCH_3	55.5	3.71 (s)	55.7	3.80 (s)	56.6	3.82 (s)

Hz) and a singlet at δ_H 3.82 (3H) indicated the existence of a second 4-hydroxy-3-methoxyphenyl ring. The $^{1}H-^{1}H$ COSY correlations of H-8 (δ_H 2.62) with H-7 (δ_H 3.97), H-9 (δ_H 1.95), and H-12 (δ_H 3.39 and 3.54) and of H-9 (δ_H 1.95) with H-8 (δ_H 2.62), H-10 (δ_H 3.70), and H-11 (δ_H 3.62 and 3.68) allowed the assignment of a 2,3-dihydroxymethylbutanol fragment. The proton at δ_H 3.97 (H-7) showed long-range correlations with carbon resonances at δ_C 113.3 (C-2), 121.9 (C-6), 113.1 (C-2′), and 121.5 (C-6′), suggesting that the six-carbon unit was attached to two 3,4-dimethoxyphenyl groups at C-7. The relative configuration of **3** was obtained through analysis of coupling constants and the NOESY spectrum. H-7, H-8, and H-9 were determined to be β-, α-, and α-oriented, respectively, on the basis of the NOE correlation H-9/H-8 and the coupling constant ($J = 12.0$ Hz) between H-7 and H-8. Compound **3**, named daphneresinol, is the first example of a 2-benzhydryl-3-hydroxymethylbutane-1,4-diol skeleton isolated from a natural source.

Compound **4** had a molecular formula of $C_{21}H_{24}O_8$ by HRESIMS ($[M + Na]^+$ at *m/z* 427.1368). The ^{1}H and ^{13}C NMR (DEPT) spectra showed signals assignable to three *O*-methyl groups [δ_H 3.86 (6H, s) and 3.81 (3H, s); δ_C 56.9 and 56.4], an aldehyde group (δ_H 9.62, d, $J = 7.8$ Hz and δ_C 196.0), and a *trans* double bond (δ_H 7.58, d, $J = 15.6$ Hz and 6.74, dd, $J = 7.8$, 15.6 Hz; δ_C 155.2 and 129.1). In the ^{1}H NMR spectrum, three aromatic protons in an ABX pattern (δ_H 6.72, d, $J = 7.8$ Hz, 6.78, dd, $J = 1.8$, 7.8 Hz, and 6.97, d, $J = 1.8$ Hz) indicated the existence of a 1,3,4-trisubstituted aromatic ring. Two additional aromatic protons [δ_H 6.99 (2H, br s)] indicated the existence of a 1,3,4,5-tetrasubstituted aromatic ring. The NMR data of **4** were very similar to those of known armaoside,[34] except for the absence of a D-glucose group. The NOE correlation of H-7/H-8, together with a small coupling constant ($J = 5.4$ Hz) between H-7 and H-8,[35] allowed the assignment of the relative configuration of **4**. Consequently, compound **4** was named armaosigenin.

Compound **5** had the molecular formula $C_{20}H_{20}O_6$ by HRESIMS ($[M]^+$ at *m/z* 356.1256). In the ^{1}H NMR spectrum, three aromatic protons with an ABX pattern (δ_H 6.73, d, $J = 7.8$ Hz, 6.64, dd, $J = 1.2$, 7.8 Hz, and 6.68, d, $J = 1.2$ Hz) indicated the existence of a 1,3,4-trisubstituted aromatic ring. Three additional aromatic protons with an ABX pattern (δ_H 6.76, m, 6.76, m, and 6.84, d, $J = 1.2$) were attributed to a second 1,3,4-trisubstituted aromatic ring. The ^{1}H and ^{13}C NMR (DEPT) spectra showed signals assignable to two $-OCH_2O-$ groups [δ_H 5.93 (4H, d, $J = 6.6$ Hz); δ_C 100.9 and 100.8]. The NMR data of **5** were very similar to those of cubebin,[36] except for the signals due to a $-CH-$ group (δ_H 4.80, d, $J = 6.6$ Hz and δ_C 82.8) instead of the signals due to a $-CH-$ group (δ_H 6.40, d, $J = 6.6$ Hz and δ_C 107.3). The relative configurations of H-7, H-8, and H-8′ were determined to be β-, α-, and β-oriented, respectively, on the basis of the NOE correlation H-7/H-8′ and the coupling constant ($J = 6.6$ Hz) between H-7 and H-8. Thus, compound **5** was deduced and named isocubebin. Although (±)-isocubebin was obtained in 1992 by chemical synthesis,[37] isocubebin is obtained from a natural source for the first time.

All 38 isolates were tested for inhibitory activities against LPS-induced NO production in RAW 264.7 macrophages. Compounds **2**, **8**, **9**, **12**, **13**, and **15** showed inhibitory activities against the production of NO with IC_{50} values of 0.091, 0.047, 0.005, 0.088, 0.004, and 0.074 μM/mL, respectively (Table 3).

Nitric oxide (NO) plays an important role in the inflammatory process;[38] therefore, inhibitors of NO release may be considered as a therapeutic agent in inflammatory diseases.[39] Although a number of natural products have been reported to inhibit NO release,[40−42] only a limited number of phenylpropanoids, e.g., pinoresinol, were studied.[43,44] Our investigation showed that compounds **9** and **13** strongly inhibited nitric oxide release and may represent potential nitric oxide synthase inhibitors. Our research also implied that phenylpropanoids may be responsible for the traditional usages of *D. feddei* to treat injuries from falls and bruises.

Table 2. ^{13}C and ^{1}H NMR Spectroscopic Data of Compounds **4** and **5** (in CD_3OD)

	4		**5**	
position	δ_C	δ_H (mult. *J* Hz)	δ_C	δ_H (mult. *J* Hz)
1	133.9		137.0	
2	111.6	6.97 (d, 1.8)	106.2	6.84 (d, 1.2)
3	148.7		147.8	
4	147.0	6.72 (d, 7.8)	146.8	
5	115.7	6.78 (dd, 1.8, 7.8)	108.2	6.76 (m)
6	120.9		119.0	6.76 (m)
7	74.3	4.89 (d, 5.4)	82.8	4.80 (d, 6.6)
8	87.6	4.39 (m)	52.6	2.35 (m)
9	61.9	3.63 (dd, 2.4, 12)	60.8	3.75 (dd, 6.6, 10.8)
		3.90 (dd, 1.8, 12)		3.88 (dd, 5.4, 10.8)
10			100.9	5.93 (d, 6.6)
1′	131.4		134.1	
2′	107.4	6.99 (s)	108.9	6.68 (d, 1.2)
3′	154.9		147.7	
4′	140.0		145.9	
5′	154.9		108.0	6.73 (d, 7.8)
6′	107.4	6.99 (s)	121.4	6.64 (dd, 1.2, 7.8)
7′	155.2	7.58 (d, 15.6)	33.2	2.53 (dd, 10.2, 13.8)
				2.87 (dd, 5.4, 13.8)
8′	129.1	6.74 (dd, 7.8, 15.6)	42.3	2.70 (m)
9′	196.0	9.62 (d, 7.8)	72.8	3.72 (dd, 6.6, 8.4)
				3.74 (dd, 6.6, 8.4)
10′			100.8	5.93 (d, 6.6)
OCH_3	56.4	3.81 (s)		
OCH_3	56.9	3.86 (s)		
OCH_3	56.9	3.86 (s)		

Experimental Section

General Experimental Procedures. NMR spectra were recorded on a Bruker Avance 600 or Avance 400 NMR spectrometer with TMS as interal standard. ESIMS were measured on an Agilent LC/MSD Trap XCT mass spectrometer, whereas HRESIMS were measured using a Q-TOF micro mass spectrometer (Waters, USA). Optical rotations were acquired with a Perkin-Elmer 341 polarimeter, whereas IR spectra were recorded on a Bruker Vector 22 spectrometer with KBr pellets. Materials for CC were silica gel (100−200 mesh; Huiyou Silical Gel Development Co. Ltd. Yantai, China), silica gel H (10−40 μm; Yantai), Sephadex LH-20 (40−70 μm; Amersham Pharmacia Biotech AB, Uppsala, Sweden), and YMC-GEL ODS-A (50 μm; YMC, Milford, MA). Preparative TLC (0.4−0.5 mm) was conducted with glass precoated silica gel GF_{254} (Yantai).

Plant Material. The plant material was collected in July 2006 in Kunming City, Yunnan Province, China, and identified as *Daphne feddei* levl. by Prof. Li-Shan Xie of Kunming Institute of Botany. A voucher specimen has been deposited in the Herbarium of the School of Pharmacy, Second Military Medical University, Shanghai (No. 200607-12).

Assay for Inhibition Ability against LPS-Induced NO Production. RAW 264.7 macrophages were seeded in 24-well plates (10^5 cells/well). The cells were co-incubated with drugs and LPS (1 μg/mL) for 24 h. The amount of NO was assessed by determining the nitrite concentration in the cultured RAW 264.7 macrophage supernatants with Griess reagent. Aliquots of supernatants (100 μL) were incubated, in sequence, with 50 μL of 1% sulfanilamide and 50 μL of 0.1% naphthylethylenediamine in 2.5% phosphoric acid solution. The absorbances at 570 nm were read using a microtiter plate reader.[41]

Extraction and Isolation. The air-dried and powdered stem bark of *D. feddei* (6.5 kg) was extracted with MeOH for 3 × 50 L × 2 h. The solvent was evaporated under vacuum. Then the extract was suspended in H_2O and partitioned with petroleum ether, EtOAc, and *n*-butanol successively. The EtOAc extract (400 g) was subjected to CC on silica gel (200−300 mesh, 1000 g) and eluted successively with gradient $CHCl_3$−MeOH mixtures of increasing polarity. The 2% MeOH eluates were rechromatographed on silica gel with $CHCl_3$−MeOH to give **1** (25 mg), **2** (30 mg), **5** (200 mg), **6** (2 g), **7** (700 mg), **9** (2 g), **10** (20 mg), **11** (200 mg), **29** (380 mg), **30** (30 mg), and **31** (18 mg). The 4% MeOH eluates were rechromatographed on silica gel with $CHCl_3$−MeOH followed by Sephadex LH-20 with MeOH to give **3** (80 mg), **4** (4 mg), **8** (250 mg), **12** (140 mg), **13** (110 mg), **14** (80 mg), **15** (70 mg), **16** (100 mg), **17** (300 mg), **18** (110 mg), **22** (10 mg), **32** (20 mg), **34** (100 mg), **36** (120 mg), and **37** (140 mg). The 10% MeOH eluates were rechromatographed on ODS (CH_3OH−H_2O, 10:100−100:0) followed by Sephadex LH-20 with MeOH to give **19** (2 g), **20** (15 mg), **21** (150 mg), **23** (8 mg), **24** (120 mg), **25** (50 mg), **26** (17 mg), **27** (15 mg), **28** (30 mg), **33** (18 mg), **35** (80 mg), and **38** (130 mg).

Table 3. Effect of Phenylpropanoids on LPS-Induced NO Production in RAW264.7 Macrophages (n = 4, means ± SD)[a]

compound	IC_{50} (μM/mL)	compound	IC_{50} (μM/mL)
AG	0.021	**1**	0.146
2	0.091	**6**	0.266
7	0.245	**8**	0.047
9	0.005	**10**	0.260
12	0.088	**13**	0.004
14	0.252	**15**	0.074
16	0.172	**24**	0.112
26	0.127	**32**	0.144
38	0.163	OCs	>0.300

[a] LPS: negative control; AG: aminoguanidine, positive control; OCs: other compounds, including compounds **3**, **4**, **5**, **11**, **17**, **18**, **19**, **20**, **21**, **22**, **23**, **25**, **27**, **28**, **29**, **30**, **31**, **33**, **34**, **35**, **36**, **37**.

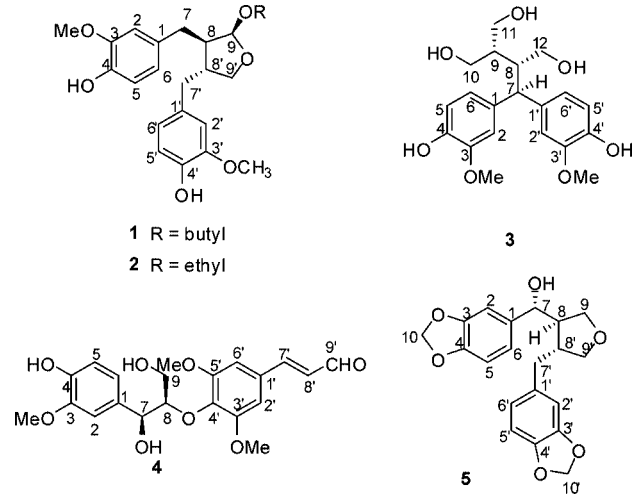

Figure 1. Structrues of compounds **1**−**5**.

Compound 1: pale yellow, viscous oil; $[\alpha]^{20}_D$ +50 (*c* 0.20, $CHCl_3$); IR ν^{KBr}_{max} (cm^{-1}) 3422, 2933, 1739, 1607, 1515, 1465, 1429, 1271, 1236, 1035, 852, 624 cm^{-1}; ^1H and ^{13}C NMR spectroscopic data, see Table 1; positive HRESIMS found 439.2096, calcd 439.2097 for $C_{24}H_{32}O_6Na$ $[M + Na]^+$.

Compound 2: pale yellow, viscous oil; $[\alpha]^{20}_D$ +32 (*c* 0.15, $CHCl_3$); IR ν^{KBr}_{max} (cm^{-1}) 3419, 2937, 1769, 1598, 1514, 1466, 1393, 1271, 1236, 1034, 1042, 854, 562 cm^{-1}; ^1H and ^{13}C NMR spectroscopic data, see Table 1; positive HRESIMS found 411.1786, calcd 411.1784 for $C_{22}H_{28}O_6Na$ $[M + Na]^+$.

Compound 3: pale yellow, viscous oil; $[\alpha]^{20}_D$ −26 (*c* 0.11, CH_3OH); IR ν^{KBr}_{max} (cm^{-1}) 3380, 2936, 2884, 2838, 1601, 1516, 1466, 1277, 1128, 1033, 1003, 824, 656 cm^{-1}; ^1H and ^{13}C NMR spectroscopic data, see Table 1; positive HRESIMS found 378.1678, calcd. 378.1679 for $C_{20}H_{26}O_7$ $[M]^+$.

Compound 4: pale yellow, viscous oil; $[\alpha]^{20}_D$ 0 (*c* 0.09, CH_3OH); IR ν^{KBr}_{max} (cm^{-1}) 3420, 2959, 2918, 2850, 1672, 1577, 1422, 1335, 1160, 1125, 1012, 823, 650 cm^{-1}; ^1H and ^{13}C NMR spectroscopic data, see Table 2; positive HRESIMS found 427.1368, calcd 427.1369 for $C_{20}H_{26}O_7$ $[M + Na]^+$.

Compound 5: pale yellow, viscous oil; $[\alpha]^{20}_D$ −9 (*c* 0.20, $CHCl_3$); IR ν^{KBr}_{max} (cm^{-1}) 3419, 2920, 2882, 1723, 1608, 1505, 1487, 1442, 1395, 1247, 1189, 1125, 1037, 928, 721 cm^{-1}; ^1H and ^{13}C NMR spectroscopic data, see Table 2; positive HRESIMS found 356.1256, calcd 356.1260 for $C_{20}H_{26}O_7$ $[M]^+$.

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References and Notes

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