Notes

Acylated Flavone *C*-Glycosides from *Trollius ledebouri*

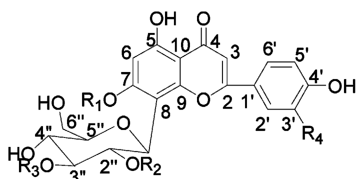
Jian-Hua Zou, JunShan Yang,* and Liang Zhou

Institute of Medicinal Plant Development, Chinese Academy of Medical Sciences and Peking Union Medical College, Beijing 100094, People's Republic of China

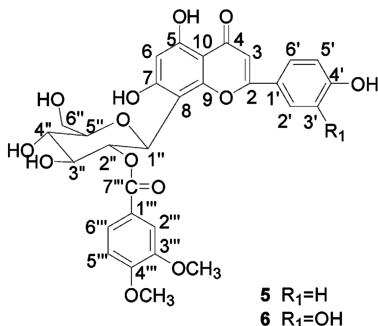
Received October 2, 2003

Six new acylated flavone *C*-glycosides, 2″-*O*-(2‴-methylbutyryl)isoswertisin (**1**), 3″-*O*-(2‴-methylbutyryl)-isoswertisin (**2**), 2″-*O*-(2‴-methylbutyryl)vitexin (**3**), 2″-*O*-(2‴-methylbutyryl)orientin (**4**), 2″-*O*-(3‴,4‴-dimethoxybenzoyl)vitexin (**5**), and 2″-*O*-(3‴,4‴-dimethoxybenzoyl)orientin (**6**), and the known flavone *C*-glycoside, isoswertisin, were isolated from the flowers of *Trollius ledebouri*. The structures of **1**−**6** were elucidated by spectroscopic methods.

The genus *Trollius*, of which 16 species grow in mainland China and 25 species are recorded worldwide, belongs to the family Ranunculaceae. *Trollius ledebouri* Reichb. is mainly distributed in the northern regions of China. Its flowers, commonly named "Jin Lian Hua", possess antimicrobial and antiviral actions and have been used for a long time as a well-known medicinal plant to treat colds, high fevers, chronic tonsillitis, and acute tympanitis.[1] Previous chemical investigations have shown the presence of an organic acid and flavone *C*-glycosides.[2] In the present study, six new acylated flavone *C*-glycosides (**1**−**6**) were isolated and identified from the ethanol extract of the flowers of *T. ledebouri*, together with the known flavone *C*-glycoside, isoswertisin,[3] that was obtained for the first time from this plant. This paper reports the isolation and structural elucidation of compounds **1**−**6**.



1 R_1=CH_3, R_2=Et(Me)CHCO, R_3=H, R_4=H
2 R_1=CH_3, R_2=H, R_3=Et(Me)CHCO, R_4=H
3 R_1=H, R_2=Et(Me)CHCO, R_3=H, R_4=H
4 R_1=H, R_2=Et(Me)CHCO, R_3=H, R_4=OH



5 R_1=H
6 R_1=OH

Compound **1** was obtained as yellow powder and exhibited a positive magnesium hydrochloric acid test, indicating a flavonoid. The HRFABMS analysis established its molecular formula as $C_{27}H_{30}O_{11}$. It showed IR (3394, 1655 cm^{-1}) and UV absorptions (262, 336 nm) characteristic of a flavone.[4,5] The ^1H NMR spectrum of **1** (Table 1) indicated signals of two set of doublets at δ 8.09 (2H, d, J = 9 Hz) and 6.91 (2H, d, J = 9 Hz), due to the protons H-2′, 6′ and H-3′, 5′ of a 4′-hydroxyphenyl moiety, and two singlets at δ 6.84 and 6.48, due to the protons at C-3 and C-6 in rings C and A of a flavone, respectively.[3] The ^{13}C NMR spectrum (Table 1) revealed 27 carbon signals, which suggested the presence of a flavonoid moiety, a saccharide moiety, and an acyl group in **1**. The signal of C-6 at δ 94.4 exhibited the presence of a methoxyl group attached to the C-7 position. The six carbon signals of the sugar moiety were at δ 70.3, 71.3, 75.4, 70.2, 60.6, and 81.8 (C-5″), suggesting that **1** is a flavone *C*-glycoside. The sugar moiety was determined to be β-glucose from ^1H and ^{13}C NMR data. The site of the sugar linkage in **1** was also considered to be at the C-8 position of the aglycon moiety. The glycosidation position was unambiguously determined at the C-8 position by the appearance of cross-peaks of the glucosyl anomeric proton H-1″ (δ 4.88, d, J = 10 Hz) with the carbon signals at δ 103.1 (C-8), 162.5 (C-7), and 155.4 (C-9) in the HMBC spectrum. From these data, the sugar substituent at C-8 of the aglycon moiety gave a pattern of ^{13}C NMR signals similar to those in isoswertisin.[3] The signals of C-1″ (δ 70.3) and C-3″ (δ 75.4) of the sugar moiety showed upfield shifts of 2.9 and 3.1 ppm, compared with the corresponding data (δ 73.2, 78.5) of isoswertisin.[3] These data suggested that the acyl group was attached to the C-2″ hydroxyl of the sugar moiety. The ^1H NMR spectrum showed a doublet signal at δ 0.68 and a triplet signal at δ 0.58 ascribable to the presence of two methyl groups, together with the multiplets for the three protons (H-2‴ and H-3‴); the ^{13}C NMR spectrum exhibited the corresponding four carbon signals at δ 16.2 (C-5‴), 10.9 (C-4‴), 40.0 (C-2‴), and 25.5 (C-3‴), suggesting that **1** was acylated with a 2-methylbutyric acid. The latter observation was supported by the fragment ion peak at m/z 428 due to the loss of the 2-methylbutyric acid (102 mass units) from molecular ion (m/z 530) in the EIMS. Finally, the position of the acyl

* To whom correspondence should be addressed. Tel: +86-10-62899739. Fax: +86-10-62898425. E-mail: junshanyang@hotmail.com.

10.1021/np030443r CCC: $27.50 © 2004 American Chemical Society and American Society of Pharmacognosy
Published on Web 02/25/2004

Table 1. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) Spectral Data of **1**−**4** (in DMSO-d_6)

	1[a]		**2**[a]		**3**[a]		**4**[a]	
position	δ_H (J in Hz)	δ_C	δ_H (J in Hz)	δ_C	δ_H (J in Hz)	δ_C	δ_H (J in Hz)	δ_C
2		164.2 s		164.3 s		163.7 s		164.1 s
3	6.84 s	102.2 d	6.82 s	102.7 d	6.78 s	102.0 d	6.65 s	102.6 d
4		181.9 s		182.3 s		181.7 s		181.9 s
5		161.5 s		161.2 s		160.6 s		160.5 s
6	6.48 s	94.4 d	6.52 s	95.0 d	6.22 s	97.4 d	6.21 s	97.6 d
7		162.5 s		163.4 s		161.8 s		162.0 s
8		103.1 s		104.9 s		102.2 s		102.3 s
9		155.4 s		155.3 s		156.2 s		156.5 s
10		103.9 s		104.5 s		103.6 s		103.9 s
1′		121.2 s		121.6 s		121.3 s		122.0 s
2′	8.09 d (9)	128.9 d	8.06 d (9)	129.2 d	8.07 d (9)	128.7 d	7.52 d (2)	114.0 d
3′	6.91 d (9)	115.6 d	6.91 d (9)	115.8 d	6.91 d (9)	115.5 d		145.7 s
4′		161.0 s		161.1 s		160.2 s		149.5 s
5′	6.91 d (9)	115.6 d	6.91 d (9)	115.8 d	6.91 d (9)	115.5 d	6.88 d (8.5)	115.7 d
6′	8.09 d (9)	128.9 d	8.06 d (9)	129.2 d	8.07 d (9)	128.7 d	7.58 dd (8.5, 2)	119.5 d
1″	4.88 d (10)	70.3 d	4.80 d (10)	73.5 d	4.84 d (10)	70.6 d	4.83 d (10)	70.9 d
2″	5.35 t (10)	71.3 d	3.97 t (10)	68.5 d	5.33 t (10)	71.3 d	5.32 t (10)	71.6 d
3″	3.49 m	75.4 d	4.88 t (10)	78.9 d	3.47 m	75.2 d	3.48 m	75.7 d
4″	3.49 m	70.2 d	3.57 t (10)	68.2 d	3.46 m	70.2 d	3.44 m	70.7 d
5″	3.33 m	81.8 d	3.38 m	81.7 d	3.32 m	81.7 d	3.31 m	82.1 d
6″	3.58 dd (5.5, 12.5)	60.6 t	3.58 br d (10)	60.5 t	3.57 dd (6, 12)	60.6 t	3.59 dd (6, 12)	61.3 t
	3.78 m		3.73 br d (10)		3.79 m		3.80 m	
1‴		174.2 s		175.3 s		174.4 s		174.6 s
2‴	2.03 sxt (7)	40.0 d	2.29 m	40.4 d	2.04 sxt (7)	40.1 d	2.02 m	40.4 d
3‴	1.22 dqui (7, 14)	25.5 t	1.52 m	26.4 t	1.24 m	25.5 t	1.24 m	25.9 t
	1.13 dqui (7, 14)		1.33 m		1.14 m		1.14 m	
4‴	0.58 t (7)	10.9 q	0.78 t (7)	11.2 q	0.59 t (7)	10.9 q	0.59 t (7.5)	11.3 q
5‴	0.68 d (7)	16.2 q	1.03 d (7)	16.6 q	0.71 d (7)	16.2 q	0.71 d (7.5)	16.5 q
OCH_3-7	3.86 s	56.2 q	3.88 s	56.6 q				

[a] Assignments based on HMQC and HMBC.

group was confirmed at C-2″ of the sugar moiety by the long-range correlation between the proton at δ 5.35 (H-2″) and the carbonyl signal at δ 174.2 (C-1‴) from the HMBC spectrum. On the basis of the above evidence, the structure of the acylated flavone *C*-glycoside **1** was established as 2″-*O*-(2‴-methylbutyryl)isoswertisin.

The molecular formula for **2** was determined as $C_{27}H_{30}O_{11}$ by HRFABMS, which was the same as **1**. Comparison of the ^{13}C NMR data (Table 1) of **2** with those of **1** indicated that the major difference was the position of the acyl group in the sugar moiety. The signals of C-2″ (δ 68.5) and C-4″ (δ 68.2) of the glucosyl residue were shifted upfield by 2.3 and 2.1 ppm, compared with the corresponding signals (δ 70.8, 70.3) of isoswertisin.[3] The HMBC spectrum showed a correlation between the H-3″ (δ 4.88, t, J = 10 Hz) and the C-1‴ (δ 175.3) signals. These data confirmed that the acyl group was attached to the C-3″ hydroxyl of the sugar moiety. The HMBC spectrum also provided the correlations between the proton H-1″ (δ 4.80) and the carbon signals at δ 104.9 (C-8), 163.4 (C-7), and 155.3 (C-9), which supported the attachment of the sugar moiety at C-8. The ^{1}H and ^{13}C NMR data (Table 1) were assigned completely from HMQC and HMBC spectra. Thus, the structure of **2** was established as 3″-*O*-(2‴-methylbutyryl)isoswertisin.

Compound **3** was assigned the molecular formula $C_{26}H_{28}O_{11}$ by HRFABMS, 14 mass units less than **1**. Comparison of the ^{13}C NMR data (Table 1) of **3** with those of vitexin[6] indicated that an acyl group was attached to the sugar moiety in **3**. The acyl group was identified as a methylbutyryl group from the ^{1}H and ^{13}C NMR spectra. The signals of C-1″ (δ 70.6) and C-3″ (δ 75.2) of the glucosyl residue were shifted upfield by 2.8 and 3.5 ppm, respectively, compared with the corresponding signals (δ 73.4, 78.7) of vitexin.[6] Moreover, the correlations between the proton at δ 5.33 (H-2″) and the carbonyl signal at δ 174.4 (C-1‴) confirmed that the acyl group was attached to the C-2″ hydroxyl of the glucosyl residue. Thus, the structure of **3** was established as 2″-*O*-(2‴-methylbutyryl)vitexin.

Compound **4** was assigned a molecular formula of $C_{26}H_{28}O_{12}$, as established from the molecular ion peak at *m*/*z* 532.1573 in the HREIMS, 16 mass units greater than those of **3**. Comparison of the ^{1}H NMR data (Table 1) of **4** with those of **3** indicated the presence of an ABX system in the ring B of **4**, instead of the AA′BB′ system in **3**, suggesting that the flavone moiety of **4** was luteolin.[7] The ^{13}C NMR spectrum (Table 1) of **4** showed 26 carbon signals, indicating that the structure was a luteolin derivative containing a sugar moiety and a methylbutyryl group. From the HMQC and HMBC spectra, the proton and carbon signals could be assigned completely. Thus, the structure of **4** was confirmed as 2″-*O*-(2‴-methylbutyryl)orientin.

Compound **5** gave a HRFABMS quasi-molecular ion at *m*/*z* 597.1611 $[M + H]^+$, corresponding to a molecular formula of $C_{30}H_{28}O_{13}$. Its UV (262 and 332 nm) and IR (3390 and 1658 cm^{-1}) spectral data were similar to those of **3**, which suggested that **5** possesses the same vitexin molecular skeleton as **3**. The ^{1}H NMR spectrum (Table 2) showed signals due to two aromatic methoxyl groups at δ 3.74 (3H, s) and 3.78 (3H, s) and an ABX system [δ 7.35 (1H, dd, J = 8.5, 2.0 Hz), 7.20 (1H, d, J = 2.0 Hz), 6.95 (1H, d, J = 8.5 Hz)] corresponding to a 1,3,4-trisubstituted aromatic ring. These data indicated the presence of a dimethoxybenzoyl group.[2] Comparison of the ^{13}C NMR data (Table 2) of **5** with vitexin[6] confirmed that there is a vitexin moiety in the structure of **5**, but the signals of C-1″ (δ 71.0) and C-3″ (δ 75.9) of **5** were shifted upfield by 2.4 and 2.8 ppm, respectively, compared with the corresponding signals (δ 73.4, 78.7) of vitexin.[6] These data showed that the acyl group was attached to the C-2″ hydroxyl of glucose. The correlation between the proton at δ 5.52 (H-2″) and the carbon at δ 164.7 (C-1‴) in the HMBC experiment confirmed that the dimethoxybenzoyl group was attached to the C-2″ position of the glucose moiety. The HMQC and HMBC spectra allowed for the complete assignments of the proton and carbon signals of **5**. Thus, the structure of **5** was established as 2″-*O*-(3‴,4‴-dimethoxybenzoyl)vitexin.

Table 2. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) Spectral Data of **5** and **6** (in DMSO-d_6)

	5[a]		**6**[a]	
positon	δ_H (J in Hz)	δ_C	δ_H (J in Hz)	δ_C
2		164.1 s		164.2 s
3	6.81 s	102.6 d	6.67 s	102.7 d
4		182.0 s		181.9 s
5		160.7 s		160.4 s
6	6.09 s	97.8 d	6.09 s	97.7 d
7		162.1 s		162.0 s
8		102.4 s		102.4 s
9		156.3 s		156.4 s
10		103.9 s		103.9 s
1′		121.6 s		122.1 s
2′	8.12 d (9)	129.1 d	7.55 d (2)	114.1 d
3′	6.93 d (9)	115.9 d		145.8 s
4′		161.3 s		149.6 s
5′	6.93 d (9)	115.9 d	6.91 d (8.5)	115.8 d
6′	8.12 d (9)	129.1 d	7.63 dd (8.5, 2)	119.6 d
1″	5.02 d (10)	71.0 d	5.01 d (10)	71.1 d
2″	5.52 t (10)	72.8 d	5.51 t (10)	72.7 d
3″	3.64 m	75.9 d	3.63 m	75.9 d
4″	3.56 m	70.6 d	3.52 m	70.7 d
5″	3.40 m	82.1 d	3.40 m	82.3 d
6″	3.82 m, 3.63 m	61.0 t	3.85 m, 3.65 m	61.3 t
1‴		122.0 s		122.1 s
2‴	7.20 d (2.0)	111.6 d	7.19 d (2)	111.6 d
3‴		148.2 s		148.2 s
4‴		152.7 s		152.7 s
5‴	6.95 d (8.5)	110.9 d	6.94 d (8.5)	110.9 d
6‴	7.35 dd (8.5, 2.0)	123.0 d	7.34 dd (8.5, 2)	123.1 d
7‴		164.7 s		164.7 s
OCH_3-3‴	3.74 s	55.5 q	3.73	55.5 q
OCH_3-4‴	3.78 s	55.7 q	3.78	55.7 q

[a] Assignments based on HMQC and HMBC.

The HRFABMS of **6** showed a quasi-molecular ion peak at *m/z* 613.1554 [M + H]$^+$, 16 mass units greater than those of **5**, indicating the molecular formula as $C_{30}H_{28}O_{14}$ and corresponding to 17 degrees of unsaturation. Comparison of the ^{1}H NMR data (Table 2) of **6** with those of **5** indicated a further ABX system in **6** compared with **5**. The additional ABX system [δ 7.63 (1H, dd, J = 8.5, 2 Hz), 7.55 (1H, d, J = 2 Hz), and 6.91 (1H, d, J = 8.5 Hz)] of **6** confirmed the presence of an *ortho*-dihydroxy group in ring B of the flavone aglycon.[7] The ^{13}C NMR, HMQC, and HMBC spectra confirmed that two hydroxy groups in **6** were attached to C-3′ and C-4′, respectively. On the basis of HMQC and HMBC spectra, the structure of **6** was established as 2″-*O*-(3‴,4‴-dimethoxybenzoyl)orientin.

The flavone *C*-glycosides **1**−**4** are esterified with 2-methylbutyric acid, which has not been reported previously in any flavonoid *C*-glycoside.[8,9] This is the first report of the occurrence in nature of two flavone glycosides, **5** and **6**, acylated with 3,4-dimethoxybenzoic acid (veratric acid).

Experimental Section

General Experimental Procedures. Melting points were determined on an X4 micro-melting point apparatus and are uncorrected. Optical rotations were measured on a Perkin-Elmer 341 digital polarimeter. UV spectra were measured with a Hitachi UV-2201 spectrophotometer and IR spectra with an Impact 400 FTIR spectrometer. NMR spectra were recorded in DMSO-d_6 with an INOVA 500 NMR spectrometer, using visual DMSO-d_6 resonances (^{1}H δ 2.49, ^{13}C δ 39.4) for internal reference. Mass spectra were recorded on an AutoSpec Ultima-TOF spectrometer. Silica gel 60H 400−500 mesh and silica gel 100−200 mesh (both from Qingdao Haiyang Chemical Co., Qingdao, People's Republic of China) were used for column chromatography. Sephadex LH-20 (25−100 μm, Sigma-Aldrich) was used for chromatography.

Plant Material. The flowers of *Trollius ledebouri* Reichb. were collected from Chengde, People's Republic of China, in August 2002, and identified by Prof. Wen-Yan Lian. A voucher specimen (HB-02-0128) is deposited at the Institute of Medicinal Plant Development, Chinese Academy of Medical Sciences and Peking Union Medical College, People's Republic of China.

Extraction and Isolation. Dried flowers (15 kg) were extracted with 95% ethanol two times. The concentrated extract was suspended in water and extracted with petroleum ether, ethyl acetate, and *n*-butanol. The ethyl acetate-soluble fraction (150 g) was chromatographed over a silica gel (100−200 mesh) column, eluting with $CHCl_3$−MeOH mixtures to afford 15 fractions. Fraction 5 (7 g) was chromatographed on a silica gel 60H (400−500 mesh) column eluting with $CHCl_3$−MeOH (19:1) to afford **1** (2.1 g), and on Sephadex LH-20, eluting with MeOH, to afford **2** (27.7 mg). Fractions 8 (8 g) and 9 (8 g) were further purified on a silica gel 60H (400−500 mesh) column eluting with $CHCl_3$−MeOH (19:1) and on Sephadex LH-20 eluting with MeOH to afford **5** (110 mg) and **3** (50 mg), respectively. A mixture (11 g) of fractions 11 and 12 was chromatographed by MPLC over silica gel 60H (400−500 mesh), and elution with $CHCl_3$−MeOH−H_2O (90:10:0.5) gave 10 fractions. Fraction 4 was purified by Sephadex LH-20 with MeOH to give **4** (145 mg). Fractions 7−9 were purified by Sephadex LH-20 (MeOH) to afford **6** (50 mg) and isoswertisin (59 mg).

2″-*O*-(2‴-Methylbutyryl)isoswertisin (1): yellow powder; mp 155−157 °C; $[\alpha]^{20}_D$ −21.2° (*c* 0.066, CH_3OH); UV (CH_3OH) λ_{max} (log ϵ) 262 (4.15), 316 (4.19), 336 (4.16) nm; IR (KBr) ν_{max} 3394, 2968, 2877, 1728, 1655, 1604, 1496, 1444, 1365, 1277, 1244, 1178, 1120, 1080, 1020, 891, 837 cm^{-1}; ^{1}H and ^{13}C NMR data (Table 1); EIMS *m/z* 530 [M]$^+$ (9), 428 (4), 410 (4), 380 (3), 326 (34), 313 (25), 297 (79), 284 (18), 267 (11), 255 (5), 195 (11), 179 (22), 149 (9), 121 (14), 105 (13), 85 (26), 74 (33), 57 (100); HRFABMS *m/z* 531.1867 [M + H]$^+$ (calcd for $C_{27}H_{31}O_{11}$, 531.1866).

3″-*O*-(2‴-Methylbutyryl)isoswertisin (2): yellow powder; mp 231−233 °C; $[\alpha]^{20}_D$ −41.1° (*c* 0.036, CH_3OH); UV (CH_3OH) λ_{max} (log ϵ) 264 (4.22), 312 (4.21), 332 (4.22) nm; IR (KBr) ν_{max} 3377, 2970, 2877, 1722, 1655, 1604, 1512, 1444, 1336, 1244, 1180, 1115, 1020, 903, 837 cm^{-1}; ^{1}H and ^{13}C NMR data (Table 1); FABMS *m/z* 531 [M + H]$^+$; HRFABMS *m/z* 531.1883 [M + H]$^+$ (calcd for $C_{27}H_{31}O_{11}$, 531.1866).

2″-*O*-(2‴-Methylbutyryl)vitexin (3): yellow powder; mp 179−181 °C; $[\alpha]^{20}_D$ −22.0° (*c* 0.064, CH_3OH); UV (CH_3OH) λ_{max} (log ϵ) 264 (4.16), 314 (4.19), 334 (4.16) nm; IR (KBr) ν_{max} 3367, 2970, 2877, 1716, 1657, 1610, 1574, 1508, 1361, 1242, 1178, 1082, 1001, 891, 839 cm^{-1}; ^{1}H and ^{13}C NMR data (Table 1); EIMS *m/z* 498 [M − H_2O]$^+$ (7), 396 (5), 378 (21), 360 (27), 283 (79), 270 (49), 254 (11), 242 (16), 165 (27), 121 (28), 87 (27), 74 (100), 57 (88); HRFABMS *m/z* 539.1550 [M + Na]$^+$ (calcd for $C_{26}H_{28}O_{11}Na$, 539.1529).

2″-*O*-(2‴-Methylbutyryl)orientin (4): yellow powder; mp 185−187 °C; $[\alpha]^{20}_D$ −16.8° (*c* 0.054, CH_3OH); UV (CH_3OH) λ_{max} (log ϵ) 250 (4.21), 262 (4.21), 340 (4.25) nm; IR (KBr) ν_{max} 3381, 2968, 1722, 1655, 1610, 1512, 1361, 1261, 1188, 1080, 987, 928, 845 cm^{-1}; ^{1}H and ^{13}C NMR data (Table 1); EIMS *m/z* 532 [M]$^+$ (18), 514 (8), 430 (8), 412 (27), 394 (50), 376 (13), 352 (25), 328 (33), 299 (100), 286 (23), 165 (25); HREIMS *m/z* 532.1573 [M]$^+$ (calcd for $C_{26}H_{28}O_{12}$, 532.1581), 514.1421 [M − H_2O]$^+$ (calcd for $C_{26}H_{26}O_{11}$, 514.1475).

2″-*O*-(3‴,4‴-Dimethoxybenzoyl)vitexin (5): pale yellow powder; mp 277−279 °C; $[\alpha]^{20}_D$ −182.7° (*c* 0.064, CH_3OH); UV (CH_3OH) λ_{max} (log ϵ) 262 (4.47), 292 (4.32), 314 (4.30), 332 (4.33) nm; IR (KBr) ν_{max} 3390, 2943, 1712, 1658, 1604, 1577, 1512, 1439, 1354, 1273, 1176, 1134, 1086, 1018, 939, 841, 760 cm^{-1}; ^{1}H and ^{13}C NMR data (Table 2); FABMS *m/z* 597 [M + H]$^+$; HRFABMS *m/z* 597.1611 [M + H]$^+$ (calcd for $C_{30}H_{29}O_{13}$, 597.1608).

2″-*O*-(3‴,4‴-Dimethoxybenzoyl)orientin (6): yellow powder; mp 202−204 °C; $[\alpha]^{20}_D$ −72.2° (*c* 0.054, CH_3OH); UV (CH_3OH) λ_{max} (log ϵ) 256 (4.39), 288 (4.15), 340 (4.24) nm; IR (KBr) ν_{max} 3383, 1705, 1655, 1604, 1514, 1442, 1358, 1271, 1221, 1176, 1117, 1022, 843, 758 cm^{-1}; ^{1}H and ^{13}C NMR data (Table

2); FABMS m/z 613 [M + H]$^+$; HRFABMS m/z 613.1554 [M + H]$^+$ (calcd for $C_{30}H_{29}O_{14}$, 613.1557).

Acknowledgment. The authors thank Prof. W.-Y. He (Institute of Materia Medica, Chinese Academy of Medical Sciences and Peking Union Medical College) for the NMR data.

References and Notes

(1) *Florae Reipublicae Popularis Sinicae*, Tomus 27; Science Press: Beijing, 1979; p 85.
(2) Huang, W. Z.; Wang, L.; Duan, J. A. *Chin. Trad. Herbal Drugs* **2000**, *31*, 731−732.
(3) Della Greca, M.; Ferrara, M.; Fiorentino, A.; Monaco, P.; Previtera L. *Phytochemistry* **1998**, *49*, 1299−1304.
(4) Xie, J. X.; Chang, J. B.; Wang, X. M. *Infrared Spectra in Organic Chemistry and Medicinal Chemistry*; Science Press: Beijing, 2001; pp 404−405.
(5) Ke, Y. K.; Dong, H. R. *Handbook of Analytical Chemistry*; Fascicule 3, Spectrographic Analysis, 2nd ed.; Chemical Industry Press: Beijing, 1998; p 655.
(6) Zhang, P. C.; Xu, S. X. *J. Asian Nat. Prod. Res.* **2003**, *5*, 131−136.
(7) Kato, T.; Morita, Y. *Chem. Pharm. Bull.* **1990**, *38*, 2277−2280.
(8) Chari, V. M.; Jordan, M.; Wagner, H.; Thies, P. W. *Phytochemistry* **1977**, *16*, 1110−1112.
(9) Merfort, I.; Wendisch, D. *Planta Med.* **1992**, *58*, 355−357.

NP030443R