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云南铁杉中一个新的倍半木脂素*

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摘要：从云南铁杉（*Tsuga dumosa*）心材中分离得到 9 个化合物，采用波谱方法鉴定了它们的结构。其中化合物 1（3-(4-hydroxy-3-methoxy-benzyl)-5-[2-(4-hydroxy-3-methoxy-phenyl)-3-hydroxymethyl-7-methoxy-2，3-dihydro-benzofuran-5-yl]-4-hydroxymethyl-dihydro-furan-2-one）为一个新的倍半木脂素，命名为 dumosaol，2～9 为首次从该种植物中分离得到。

关键词：云南铁杉；倍半木脂素；Dumosaol
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A New Sesquilignan from *Tsuga dumosa*

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Abstract：A new sesquilignan，dumosaol **(1)** elucidated as 3-(4-hydroxy-3-methoxy-benzyl)-5-[2-(4-hydroxy-3-methoxy-phenyl)-3-hydroxymethyl-7-methoxy-2，3-dihydro-benzofuran-5-yl]-4-hydroxymethyl-dihydro-furan-2-one，was isolated from the methanol extract of the heartwoods of *Tsuga dumosa*，together with eight known compounds **(2－9)**. Their structures were elucidated on the basis of spectroscopic evidence. It is first time that compounds **2－9** were isolated from this plant.

Key words：*Tsuga dumosa*；Sesquilignan；Dumosaol

Tsuga dumosa is an economically important conifer indigenous to the Yunnan Province of China (Southwest College of Forestry and Yunnan Forestry Administration，1988). This plant has been extensively used for timbering and lumber products because of its resistance to decay. Sesquilignans and lignans from this genus have been reported，previously (Kawamura *et al*，1997). Further chemical investigation of the heartwoods of *T. dumosa* collected from the northern part of Yunnan province，China led to the isolation of a new sesquilignan dumosaol **(1)**，as well as eight known compounds (Fig. 1)，

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saussol (**2**) (Liu *et al*, 1989), 4, 4′-dihydroxy-3, 3′-dimethoxy-7-one-lignan-9, 9′-olid (**3**) (Nishibe *et al*, 1980), 4, 4′, 9, 7′-tetrahydroxy-3, 3′-dimethoxy-7, 9′-epoxylignan (**4**) (Huang *et al*, 1990), 4-hydroxy-cyclohexanecarboxylic acid (**5**) (Raston *et al*, 1994), 8-hydroxy-α-conidendric acid (**6**) (Kawamura *et al*, 1997), 8-hydroxy-α-conidendrine (**7**) (Kawamura *et al*, 1997), 4-(3-hydroxy-propenyl)-phenol (**8**) (Quideau *et al*, 1992) and 2′, 7-dihydroxy-4′-methoxyisoflavone (**9**) (Woodward *et al*, 1980). Their structures were determined by spectral methods.

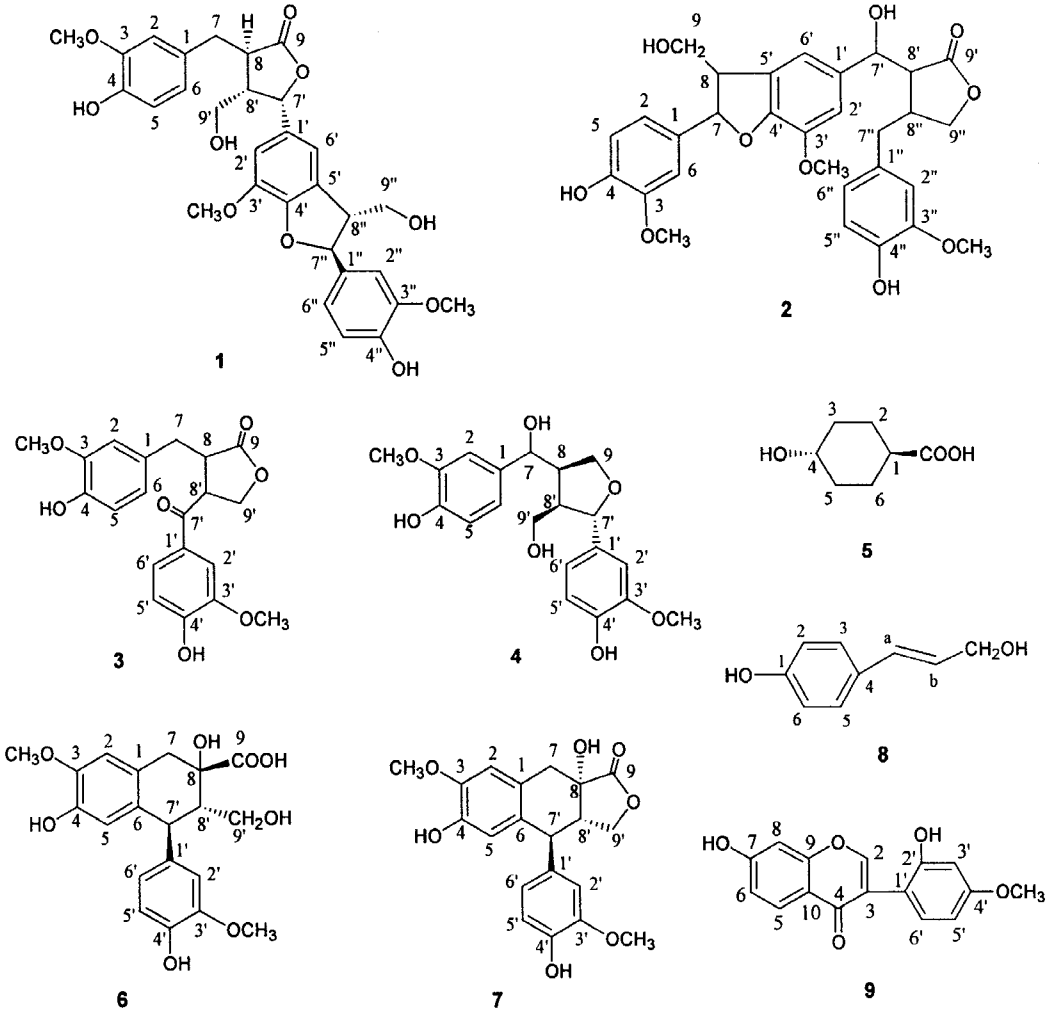

Fig. 1 Structures of compounds **1 – 9**

Compound **1** was obtained as amorphous powder, having the molecular formula of $C_{30}H_{32}O_{10}$ on the basis of EIMS (*m/z* 552, [M]$^+$) and HRESIMS ([M + Na] found: *m/z* 575.1897, calcd: 575.1893). The IR spectrum showed absorptions for hydroxyl groups (3442 cm^{-1}), carbonyl group (1749 cm^{-1}) and aromatic groups (1614, 1517 cm^{-1}). The ^{1}H NMR spectrum showed eight aromatic protons at δ_H 6.92 (1H, d, J = 1.9 Hz), 6.86 (1H, d, J = 1.8 Hz) and 6.81 – 6.73 ppm (6H, m)

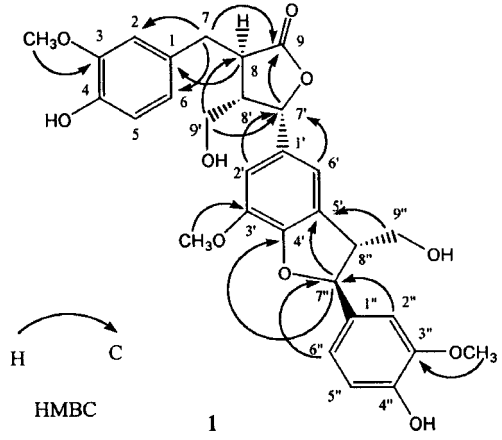

Fig. 2　The key HMBC correlations for compound **1**

and three aromatic methoxyl groups at δ_H 3.82, 3.82 and 3.78 ppm (3H each, s). The ^{13}C NMR and DEPT spectrum revealed the presence of 30 carbon atoms (Table 1), including one carbonyl (δ_C 180.6, C-9), three aromatic groups, three methoxys and eight other carbon atoms, four of them bearing oxygen atoms, which was analogous to those sesquilignans (Ichihara *et al*, 1976; 1977) except for the substitutions of C-7′ and C-8′. Chemical shift of C-7′ (δ_C 83.2) in **1** was consistent to compounds **4** (δ_C 85.8), magnone A (δ_C 83.76) (Jung *et al*, 1998) and fargesol (δ_C 84.6) (Huang *et al*, 1990), suggesting that the aromatic group link to C-7′. The HMBC spectrum showed the cross-peaks from H-2′ (δ_H 6.86) and H-6′ (δ_H 6.93) to C-7′ (δ_C 83.2), corresponding to the signal at δ_H 5.45 (1H, d, J = 2.7 Hz) (Fig. 2). The cross-peaks in HMBC spectrum from H-7 to C-2, C-6 and C-9, H-9′ to C-7′ and C-8, H-7′ to C-9, H-8 to C-1, H-7″ to C-4′ and C-5′, H-2″ and H-6″ to C-7″ and H-9″ to C-5′ were accordance with the assignment of **1**.

Table 1　^{13}C NMR data for compounds **1** and **4** in CD_3OD (100 MHz)

1						**4**			
No.	C	No.	C	No.	C	No.	C	No.	C
1	133.6	1′	132.1	1″	134.6	1	140.0	1′	136.2
2	114.4	2′	110.0	2″	110.6	2	115.9	2′	111.1
3	149.3[a]	3′	145.3	3″	149.1[a]	3	149.1	3′	149.1
4	147.7[b]	4′	148.1	4″	147.5[b]	4	147.3[e]	4′	147.4[e]
5	116.3[c]	5′	130.3	5″	116.1[c]	5	115. 9	5′	111.3
6	118.0	6′	118.8	6″	119.8	6	120.7[f]	6′	120.8[f]
7	31.8	7′	83.2	7″	89.1	7	75.6	7′	85.8
8	42.9	8′	48.4	8″	55.2	8	52.9	8′	56.0
9	180.6	9′	60.9	9″	64.8	9	71.2	9′	63.3
3-OCH_3	56.8[d]	3′-OCH_3	56.5[d]	3″-OCH_3	56.4[d]	3-OCH_3	56.4	3′-OCH_3	56.4

[a,b,c,d,e,f] Assignments may be interchangeable.

The assignment of **1** was further confirmed by HMQC and ^{1}H-^{1}H COSY spectrum exhibiting important correlations between H-7 / H-8, H-8 / H-8′, H-7′ / H-8′, H-9′ / H-8′, H-7″ / H-8″, H-7″ / H-8″ and H-9″ / H-8″. The chemical shift of H-7′ at δ 5.45 ppm suggested a *cis*-orientation of this configuration at the C-7′ / C-8′ bond (Huang *et al*, 1990). A *trans*-orientation at C-7″ / C-8″ bond can be proposed based on the chemical shift of H-7″ at δ 5.48 ppm (Ichihara *et al*, 1979). The NOESY experiment revealed clear correlations between H-8 / H-9′a, b, suggesting a *trans*-orientation at C-8′ / C-8 bond. The NOE correlations between H-7″ / H-9″a, b and H-7′ / H-8′ further confirmed the relative configuration of C-7″ / C-8″ bond and C-7′ / C-8′ bond. Thus, the relative configuration of **1** can be assigned from the above evidence. Based on above spectral analysis, **1** was

elucidated to be 3 -(4-hydroxy-3-methoxy-benzyl)-5-[2-(4 -hydroxy-3-methoxy-phenyl)-3-hydroxymethyl-7-methoxy-2, 3-dihydro-benzofuran-5-yl]-4-hydroxymethyl-dihydro-furan-2-one, which was a new sesquilignan named dumosaol.

Experimental section

General Experimental Procedures Melting point was measured on a XRC-1 micro-melting point apparatus and was uncorrected. Optical rotation was determined on a JASCO-20 polarimeter. IR spectra were obtained on KBr pellets using a Bio-Rad FTS-135 spectrometer. UV spectra were recorded on a UV 210A spectrometer. MS spectra were carried out on a VG Auto Spec-3000 spectrometer. The 1D and 2D NMR spectra were obtained on BRUKER AV-400 and DRX-500 spectrometers. TLC was carried out on silica gel G precoated plates. Separation and purification were performed by column chromatography on silica gel (200 – 300 mesh).

Plant Material The heartwoods of *Tsuga dumosa* were collected in the Dayao of Yunnan province, in July 2002 and identified by Prof. Jun Zhou of Kunming Institute of Botany, Chinese Academy of Sciences. A voucher specimen was deposited in the State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences.

Extraction and Isolation The air-dried heartwoods of *T. dumosa* (5.0 kg) were extracted with 15 L EtOH (90% × 3) at 60℃ for 4 h each time. The EtOH extraction was evaporated in vacuum. The residue (365 g) was subjected to column chromatography on silica gel (200 – 300 mesh), eluted with gradient chloroform/methanol/water (12∶1∶0.1) to yield 5 fractions. Fraction 2 was further separated over Si-gel column developed with chloroform/methanol (10∶1) and petroleum ether/chloroform/methanol (15∶12∶1) to afford compound **3** (80 mg) and **6** (90 mg). Fraction 3 was purified by repeated silica gel column chromatography eluted with petroleum ether/EtOAc (1∶1) and petroleum ether/acetone/EtOAc (3∶2∶1) to yield **4** (950 mg), **9** (10 mg), **7** (34 mg) and **8** (25 mg), respectively. Fraction 4 was further separated over Sephadex LH-20 and Si-gel column (petroleum ether/chloroform/acetone 1∶1∶1) to afford two sesquilignans **1** (19 mg) and **2** (110 mg). Fraction 5 chromatographed with petroleum ether/acetone/$CHCl_3$ (1∶2∶1) to give **5** (40 mg).

Dumosaol (1) $C_{30}H_{32}O_{10}$, amorphous powder; mp∶70 – 72℃; $[\alpha]_D^{27}$ + 30.44° (*c* 0.77, MeOH); UV max (MeOH)∶204.8, 281.6, 382.0 nm; IR bands (KBr)∶3442, 2926, 2854, 1749, 1614, 1517, 1456, 1274, 1210, 1144 and 1031 cm^{-1}; ^1H NMR (400 MHz, CD_3OD) δ 6.92 (1H, d, J = 1.9 Hz, H – 2″), 6.86 (1H, d, J = 1.8 Hz, H – 2′), 6.81 – 6.73 (6H, m, H – 2, 5, 6, 6′, 6″, 5″), 5.48 (1H, d, J = 6.3 Hz, H – 7″), 5.45 (1H, d, J = 2.7 Hz, H – 7′), 3.89 (1H, dd, J = 11.0, 4.5 Hz, H – 9′a), 3.80 (1H, m, H – 9″a), 3.82, 3.82, 3.78 (3H each, s, $3OCH_3$), 3.75 (1H, m, H – 9″b), 3.70 (1H, dd, J = 11.1, 6.4 Hz, H – 9′b), 3.47 (1H, m, H – 8″), 3.17 (1H, m, H – 8), 3.11 (1H, dd, J = 14.7, 5.8 Hz, H – 7a), 2.86 (1H, dd, J = 14.0, 9.1 Hz, H – 7b), 2.58 (1H, m, H – 8′); ^{13}C-NMR spectral data see Table 1; HR-ESIMS *m/z*∶575.1897 [M ($C_{30}H_{32}O_{10}$) + Na], calcd. 575.1893; EI-MS *m/z*∶552 $[M]^+$ (1), 534 (1), 481 (6), 466 (4), 441 (5), 298 (10), 279 (12), 256 (20), 238 (22), 192 (7), 178 (14), 152 (100), 123 (23), 109 (27), 81 (40).

Saussol (2) $C_{30}H_{32}O_{10}$, amorphous powder; mp∶141 – 143℃; EI-MS *m/z*∶552 $[M]^+$; ^1H NMR (400 MHz, CD_3OD) δ 6.96 – 6.42 (8H, m, H – 2, 2′, 2″, 5, 5″, 6, 6′, 6″), 5.50 (1H, d, J = 6.6 Hz, H – 7), 4.63 (1H, d, J = 6.6 Hz, H – 7′), 4.11 – 3.73 (4H, m, H – 9a, 9b, 9″a, 9″b), 3.80, 3.79, 3.78 (9H, s, $3ArOCH_3$), 3.34 (1H, m, H – 8), 2.94 – 2.61 (4H, m, H – 8′, 8″, 7″a, 7″b); ^{13}C NMR data were consistent with those in the literature (Liu *et al*, 1989).

4，4′-Dihydroxy-3，3′-dimethoxy-7-one-lignan-9，9′-olid (3) $C_{20}H_{20}O_7$，white amorphous powder；mp：70 – 72℃；EI-MS *m/z*：372 $[M]^+$；^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (1H, d, J = 2.0 Hz, H – 2), 7.18 (1H, dd, J = 8.3, 2.0 Hz, H – 6), 6.86 (1H, d, J = 8.3 Hz, H – 5), 6.71 (1H, d, J = 8.1 Hz, H – 5′), 6.58 (1H, d, J = 1.8 Hz, H – 2′), 6.53 (1H, dd, J = 8.1, 1.8 Hz, H – 6′), 4.35, 4.09 (2H, m, H – 9′), 4.08 (1H, m, H – 8), 3.89, 3.71 (3H each, s, $2ArOCH_3$), 3.49 (1H, m, H – 8′), 2.98 (2H, m, H – 7)；^{13}C NMR (100 MHz, $CDCl_3$) δ 195.0 (s, C – 7′), 177.4 (s, C – 9), 151.3 (s, C – 3′), 146.9 (s, C – 3), 146.5 (s, C – 4′), 144.5 (s, C – 4), 128.8 (s, C – 1′), 128.5 (s, C – 1), 123.5 (d, C – 6), 122.2 (d, C – 6′), 114.3 (d, C – 5), 113.8 (d, C – 5′), 111.6 (d, C – 2), 109.9 (d, C – 2′), 68.3 (t, C – 9′), 56.0, 56.7 (q, $2ArOCH_3$), 46.4, 44.9 (d, C – 8, 8′), 34.3 (t, C – 7).

4，4′，9，7′-Tetrahydroxy-3，3′-dimethoxy-7，9′-epoxylignan (4) $C_{20}H_{24}O_7$，amorphous powder；mp：146 – 148℃；EI-MS *m/z*：376 $[M]^+$；^{1}H NMR (400 MHz, CD_3OD) δ 6.99 (1H, d, J = 1.7 Hz, H – 2′), 6.92 (1H, d, J = 1.5 Hz, H – 2), 6.81 – 6.75 (4H, m, H – 5, 5′, 6, 6′), 4.51 (1H, d, J = 8.6 Hz, H – 7), 4.48 (1H, d, J = 8.7 Hz, H – 7′), 3.86, 3.84 (6H, s, $2ArOCH_3$), 3.74 – 3.57 (4H, m, H – 9, 9′), 2.62, 2.26 (1H each, m, H – 8, 8′)；^{13}C NMR spectral data see Table 1.

4-Hydroxy-cyclohexanecarboxylic acid (5) $C_{20}H_{24}O_7$，colorless needles (MeOH)；mp：153 – 155℃；FAB$^+$ MS *m/z*：145 $[M+H]^+$；^{1}H NMR (500 MHz, CD_3OD) δ 3.49 (1H, m, H – 4), 2.20 (1H, m, H – 1), 1.99, 1.44 (4H, m, H – 3, 5), 1.96, 1.27 (4H, m, H – 2, 6)；^{13}C NMR (125 MHz, CD_3OD) δ 179.5 (s, COOH), 70.5 (d, C – 4), 43.5 (d, C – 1), 35.3 (t, C – 3, 5), 28.5 (t, C – 2, 6).

8-Hydroxy-α-conidendric acid (6) $C_{20}H_{22}O_8$，amorphous powder；mp：132 – 134℃；EI-MS *m/z*：390 $[M]^+$；^{1}H NMR (400 MHz, CD_3OD) δ 6.68 (1H, d, J = 8.0 Hz, H – 5′), 6.84 (1H, d, J = 1.3 Hz, H – 2′), 6.77 (1H, s, H – 2), 6.75 (1H, dd, J = 8.0, 1.3 Hz, H – 6′), 6.57 (1H, s, H – 5), 3.85 (1H, d, J = 7.4 Hz, H – 7′), 3.87, 3.85 (3H each, s, $2ArOCH_3$), 3.78 (2H, d, J = 8.9 Hz, H – 9′), 3.33 (1H, d, J = 13.5 Hz, H – 7a), 3.02 (1H, d, J = 13.5 Hz, H – 7b), 2.61 (1H, m, H – 8′)；^{13}C NMR (100 MHz, CD_3OD) δ 179.0 (s, C – 9), 149.4 (s, C – 3′), 147.9 (s, C – 3), 146.6 (s, C – 4′), 145.7 (s, C – 4), 135.2 (s, C – 1′), 132.9 (d, C – 6), 125.8 (s, C – 1), 122.6 (d, C – 6′), 116.7 (d, C – 5), 116.4 (d, C – 5′), 114.1 (d, C – 2), 113.1 (d, C – 2′), 72.2 (s, C – 8), 72.0 (t, C – 9′), 56.4 (q, $2ArOCH_3$), 50.9 (d, C – 8′), 44.4 (d, C – 7′), 37.2 (t, C – 7).

8-Hydroxy-α-conidendrine (7) $C_{20}H_{20}O_7$，amorphous powder；mp：148 – 150℃；EI-MS *m/z*：372 $[M]^+$；^{1}H NMR (400 MHz, CD_3OD) δ 6.80 (1H, d, J = 8.0 Hz, H – 5′), 6.75 (1H, d, J = 1.5 Hz, H – 2′), 6.75 (1H, s, H – 2), 6.69 (1H, dd, J = 8.0, 1.5 Hz, H – 6′), 6.31 (1H, s, H – 5), 4.33 (1H, dd, J = 10.7, 8.3 Hz, H – 9′a), 4.12 (1H, d, J = 12.0 Hz, H – 7′), 4.04 (1H, dd, J = 8.0, 7.5 Hz, H – 9′b), 3.83, 3.79 (3H each, s, $2ArOCH_3$), 3.37 (1H, d, J = 16.7 Hz, H – 7a), 3.11 (1H, d, J = 16.7 Hz, H – 7b), 2.65 (1H, m, H – 8′)；^{13}C NMR (100 MHz, CD_3OD) δ 179.0 (s, C – 9), 149.4 (s, C – 3′), 147.9 (s, C – 3), 146.6 (s, C – 4′), 145.7 (s, C – 4), 135.2 (s, C – 1′), 132.9 (d, C – 6), 125.8 (s, C – 1), 122.6 (d, C – 6′), 116.7 (d, C – 5), 116.4 (d, C – 5′), 114.1 (d, C – 2), 113.1 (d, C – 2′), 72.2 (s, C – 8), 72.0 (t, C – 9′), 56.4 (q, $2ArOCH_3$), 50.9 (d, C – 8′), 44.4 (d, C – 7′), 37.2 (t, C – 7).

4-(3-Hydroxy-propenyl)-phenol (8) $C_9H_{10}O_2$，white powder；mp：122 – 124℃；FAB-MS *m/z*：149 $[M-H]^-$；^{1}H NMR (400 MHz, CD_3OD) δ 7.22 (2H, d, J = 8.6 Hz, H – 3, 5), 6.71 (2H, d, J = 8.6 Hz, H – 2, 6), 6.46 (1H, d, J = 15.9 Hz, H – a), 6.16 (1H, dt, J = 6.0, 15.8 Hz, H – b), 4.16 (2H, d, J = 5.8 Hz, CH_2OH)；^{13}C NMR (100 MHz, CD_3OD) δ 158.2 (s, C – 1), 131.9 (d, C – a), 130.0 (s, C – 4), 128.7

(d, C-3, 5), 126.7 (d, C-b), 116.3 (d, C-2, 6), 64. .0 (t, CH_2OH).

2′, 7-Dihydroxy-4′-methoxyisoflavone (9) $C_{16}H_{12}O_5$, colorless needles (MeOH); mp: 215-217℃; FAB-MS *m/z*: 283 $[M-H]^-$; ^1H NMR (400 MHz, C_5D_5N) δ 8.43 (1H, d, J = 8.6 Hz, H-5), 8.18 (1H, s, H-2), 7.81 (1H, d, J = 1.9 Hz, H-8), 7.32 (1H, dd, J = 1.9, 8.3 Hz, H-6′), 7.21 (1H, dd, J = 2.0, 7.9 Hz, H-6), 7.10 (1H, d, J = 1.9 Hz H-3′), 7.03 (1H, d, J = 8.3 Hz, H-5′), 3.77 (3H, s, OCH_3); ^{13}C NMR (100 MHz, C_5D_5N) δ 175.7 (s, C-4), 164.1, 148.8, 148.1 (s, C-7, 2′, 4′), 158.6 (s, C-9), 152.8 (d, C-2), 128.3 (d, C-5), 126.4 (s, C-3), 124.9 (s, C-1′), 120.5 (d, C-6′), 118.1 (s, C-10), 117.9 (d, C-6), 115.9 (d, C-5′), 112.5 (d, C-8), 103.2 (d, C-3′), 56.0 (q, OCH_3).

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