Notes

Revised Structures for Senegalensin and Euchrenone b_{10}

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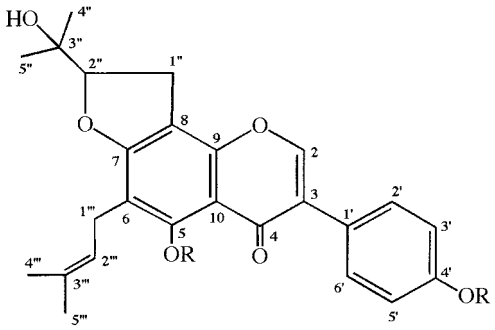
Two prenylated isoflavones (**1** and **2**) with a hydroxyisopropyldihydrofuran moiety have been isolated from the wood of *Erythrina suberosa* var. *glabrescence*. The structure of compound **1** was in agreement with that of the previously reported senegalensin, isolated from the stem bark of *Erythrina senegalensis*. The structure of senegalensin was revised from structure **2** to structure **1** by spectroscopic means. Compound **2**, the regioisomer of **1**, was confirmed as euchrenone b_{10} by comparison with the spectral data of the reported euchrenone b_{10}, isolated from the roots of *Euchresta horsfieldii*. The structure of **2** was established by 2D NMR spectroscopic analysis and by the X-ray crystallographic analysis of its *p*-bromobenzoyl derivative (**2b**).

Erythrina suberosa var. *glabrescence* has been used in Pakistan as an ornamental plant and a folk medicine, and the ethanol extract of the leaves has been reported to have antitumor activity.[1] In continuation of our study on the secondary metabolites of the genus *Erythrina*, we have reported the isolation of two isoflavones (eysubins A and B) from the wood of *E. suberosa* var. *glabrescence*.[2] Further investigation of this plant led to the isolation of two prenylated isoflavones (**1** and **2**) with a hydroxyisopropyldihydrofuran moiety, and we now describe revised structures of senegalensin (**1**) and its regioisomer, euchrenone b_{10} (**2**), which were established in the present study by spectroscopic methods, including 2D NMR experiments and X-ray crystallographic analysis of the *p*-bromobenzoyl derivative (**2b**).

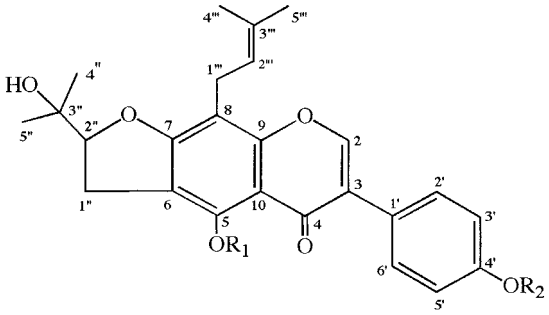
Silica gel chromatography of the dichloromethane extract of the wood of *E. suberosa* var. *glabrescence* gave the prenylated isoflavone **1** and the regioisomer **2**, together with three known isoflavones, alpinumisoflavone, wighteone, and laburnetin.[2]

Compound **1**, $C_{25}H_{26}O_6$, was obtained in a racemic form, and the IR spectrum showed the presence of conjugated carbonyl (1660 cm^{-1}) and hydroxyl (3600 cm^{-1}) groups. The UV spectrum and the characteristic singlet signal (δ 7.79),[3] ascribed to H-2 in the ^1H NMR spectrum (Table 1), provided evidence that compound **1** is an isoflavone derivative. The EIMS exhibited a molecular ion ($C_{25}H_{26}O_6$) at *m/z* 422 and characteristic fragments at *m/z* 363 $[M - 59]^+$ and *m/z* 59 of a hydroxyisopropyldihydrofuran moiety.[4] The ^1H NMR spectrum (in $CDCl_3$) revealed signals of a hydroxyl group at the C-5 position (δ 13.20), a prenyl group (δ 1.69, 1.79, 3.32, 3.37, and 5.29), and a set of *ortho*-coupled



1 R = H

1a R = Me



2 $R_1 = R_2 = H$

2a $R_1 = R_2 = Me$

2b $R_1 = H$, $R_2 = -C(=O)-C_6H_4-Br$

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Table 1. ^{1}H NMR Spectral Data of **1** and **2** and Reported Data

position	**1**[a]	**1**[b]	**2**[a]	**2**[b]	reported senegalensin[b,7]	reported euchrenone b_{10}[a,11]
2	7.79 s	8.16 s	7.90 s	8.25 s	8.14 s	7.89 s
2′	7.36 d (8.1)	7.45 d (8.8)	7.36 d (8.8)	7.47 d (8.8)	7.44 d (8.5)	7.39 d (9)
3′	6.86 d (8.1)	6.91 d (8.8)	6.84 d (8.8)	6.90 d (8.8)	6.90 d (8.5)	6.85 d (9)
5′	6.86 d (8.1)	6.91 d (8.8)	6.84 d (8.8)	6.90 d (8.8)	6.90 d (8.5)	6.85 d (9)
6′	7.36 d (8.1)	7.45 d (8.8)	7.36 d (8.8)	7.47 d (8.8)	7.44 d (8.5)	7.39 d (9)
1″	3.22 dd (15.4, 8.1)	3.29 dd (15.4, 9.2)	3.14 dd (15.8, 7.7)	3.16 dd (15.8, 9.5)	3.25 dd (9.5, 4.5)	3.12 dd (16, 8)
	3.26 dd (15.4, 9.5)	3.34 dd (15.4, 7.7)	3.23 dd (15.8, 9.5)	3.22 dd (15.8, 7.7)	3.30 dd (9.5, 6.5)	3.22 dd (16, 9)
2″	4.80 dd (9.5, 8.1)	4.87 dd (9.2, 7.7)	4.79 dd (9.5, 7.7)	4.84 dd (9.5, 7.7)	4.85 m	4.78 dd (9, 8)
4″	1.25 s	1.28 s	1.23 s	1.27 s	1.28 s	1.25 s
5″	1.37 s	1.28 s	1.36 s	1.28 s	1.28 s	1.34 s
1‴	3.32 dd (13.9, 6.6)	3.26−3.36 m	3.39 d (7.7)	3.36 dd (15.0, 7.7)	3.38 d (7.2)	3.37 brd (7)
	3.37 dd (13.9, 6.6)			3.43 dd (15.0, 7.7)		
2‴	5.29 t (6.6)	5.29 t (7.3)	5.22 t (7.7)	5.27 t (7.7)	5.28 t (7.2)	5.20 brt (7)
4‴	1.79 s	1.78 s	1.80 s	1.80 s[c]	1.77 s	1.79 brs
5‴	1.69 s	1.66 s	1.69 s	1.66 s[c]	1.65 s	1.68 brs
OH	1.58 brs	3.80 s	1.67 brs	3.76 brs	1.54 s	7.35 s
OH	5.20 brs	8.52 brs	5.65 brs	8.49 brs	8.60 s	
OH-5	13.20 s	13.49 s	12.97 s	13.16 s	13.50 s	12.94 s

[a] In $CDCl_3$. [b] In acetone-d_6. [c] Assignments may be interchanged.

Table 2. ^{13}C NMR Spectral Data of **1** and **2** and Reported Data of Senegalensin (in $CDCl_3$)

position	**1**	**2**	reported senegalensin[6]
2	151.9[a]	152.6	155.8
3	123.3	123.2	123.8
4	180.8	181.3	181.6
5	160.2[a]	155.0	165.7
6	107.6[a]	108.6	103.9
7	164.4[a]	164.0	160.7
8	102.6[a]	102.2	107.7
9	151.0	155.3	151.9
10	105.9	106.7	106.1
1′	123.4	123.0	123.4
2′	130.4	130.3	130.3
3′	115.5	115.6	115.5
4′	155.8[a]	156.0	160.1
5′	115.5	115.6	115.5
6′	130.4	130.3	130.3
1″	27.2	27.1	27.7
2″	91.0	91.3	91.0
3″	72.0	72.2	71.4
4″	24.1	23.9	24.0
5″	25.7[b]	25.6[b]	26.2
1‴	21.9	21.9	21.9
2‴	121.5	121.4	121.5
3‴	132.2	132.3	131.9
4‴	17.8	17.8	18.0
5‴	25.8[b]	25.7[b]	25.7

[a] Revised signals.[8] [b] Assignments in the same vertical column may be interchanged.

aromatic protons (δ 6.86 and 7.36) as well as typical aliphatic protons of a dihydrofuran moiety (δ 1.25, 1.37, 3.22, 3.26, and 4.80). The placement of the dihydrofuran ring fused to the C-7 and C-8 positions was assigned on the basis of the HMBC spectrum, which showed correlations from the aliphatic protons at C-1″ (δ 3.22 and 3.26) to the sp^2 quaternary carbons at C-7 (δ 164.4) and C-8 (δ 102.6) (Table 2). The prenyl group was located at the C-6 position from the HMBC spectrum, indicating a correlation from the aliphatic proton at C-1‴ (δ 3.32) to an sp^2 quaternary carbon at C-6 (δ 107.6). The assignment of the prenyl group was further confirmed by the NOESY experiment of the dimethyl ether derivative **1a**, which showed NOE interactions between a methoxyl group at C-5 (δ 3.86) and the aliphatic protons at C-1‴ (δ 3.37 and 3.40) and between the methoxyl group and an aliphatic proton at C-2‴ (δ 5.21); therefore, compound **1** was determined to be 5,4′-dihydroxy-6-(3,3-dimethylallyl)-5-hydroxyisopropyldihydrofurano[2,3:7,8]isoflavone.[5] The ^{1}H NMR spectrum of **1** was in agreement with that of the reported senegalensin, isolated from the stem bark of the Cameroonian medicinal plant, *Erythrina senegalensis*.[6,7] The structure of the reported senegalensin was erroneously shown as **2** through interpretation of its 2D NMR spectroscopic data and its ^{13}C NMR spectrum. In the ^{13}C NMR spectrum of the reported senegalensin,[8] the signals of C-6 (δ 103.9) and C-8 (δ 107.7) were incorrectly assigned, and the reverse reassignment signals of C-6 (δ 107.6) and C-8 (δ 102.6) led senegalensin to structure **1**. Fukai and Nomura have reported that the chemical shift values of the OH-5 signal are useful for the characterization of 6- or 8-prenylated flavonoids having the same B and C rings;[9,10] the OH-5 signal does not shift with changes in concentration, while changes in chemical shifts dependent on the frequency of observational spectrometers are small. The OH-5 signal of 6-prenylated flavonoids appears more downfield than that of the 8-prenylated flavonoids. The OH-5 signal of the reported senegalensin (δ 13.50 in acetone-d_6) was consistent with that of structure **1** (δ 13.49 in acetone-d_6) rather than structure **2** (δ 13.16 in acetone-d_6). Thus, the downfield value of the OH-5 for senegalensin strongly suggested senegalensin to be the 6-prenylated flavonoid. The structure of senegalensin is, therefore, revised to **1**.

Compound **2**, a regioisomer of **1**, $C_{25}H_{26}O_6$, was also obtained in a racemic form. The IR spectrum was similar to that of **1**, and the EIMS also showed typical fragments (*m*/*z* 363 and 59) of a dihydrofuran moiety.[4] The ^{1}H NMR spectrum (in $CDCl_3$) displayed proton signals [a prenyl group (δ 1.69, 1.80, 3.39, and 5.22), *ortho*-coupled aromatic protons (δ 6.84 and 7.36), and a dihydrofuran moiety (δ 1.23, 1.36, 3.14, 3.23, and 4.79)] similar to those of **1** except for a more upfield signal (δ 12.97) of the hydroxyl group at the C-5 position. The prenyl group was located at the C-8 position by the HMBC experiment, which revealed correlations from the aliphatic protons at C-1‴ (δ 3.39) to the sp^2 quaternary carbons at C-7 (δ 164.0), C-8 (δ 102.2), and C-9 (δ 155.3). The placement of the dihydrofuran ring fused to the C-6 and C-7 positions was assigned on the basis of the HMBC spectrum, which showed correlations from the aliphatic protons at C-1″ (δ 3.14 and 3.23) to the sp^2 quaternary carbons at C-6 (δ 108.6) and C-7. Further, the

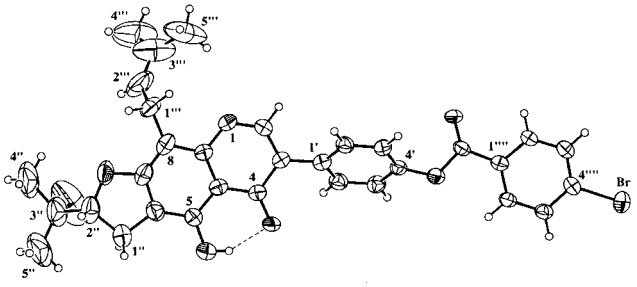
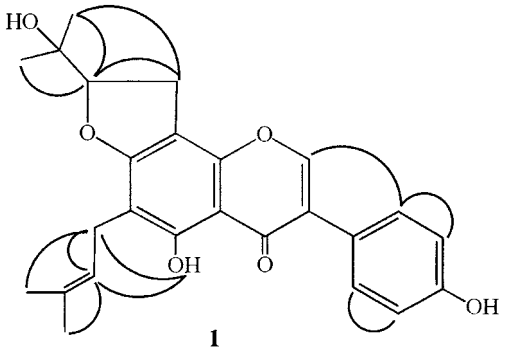
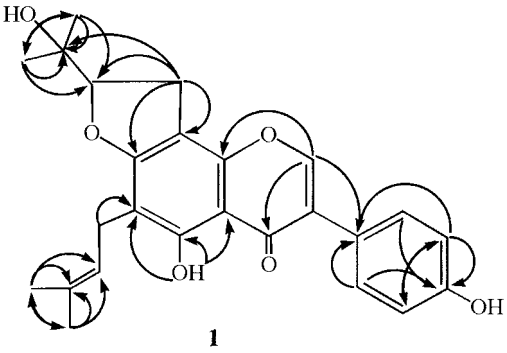


Figure 1. X-ray structure of **2b**.



1

Figure 2. NOESY correlations observed for compound **1**.



1

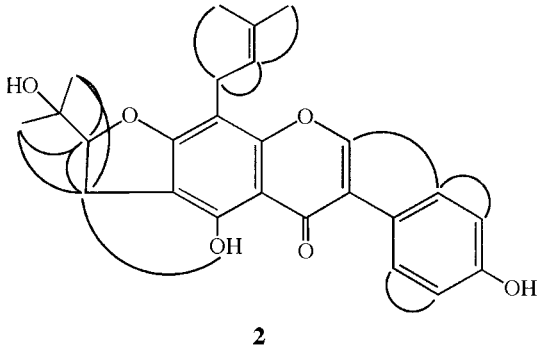
Figure 3. HMBC correlations observed for compound **1**.

placement of the dihydrofuran ring at the C-6, C-7 positions was confirmed by the NOESY experiment on its the dimethyl ether derivative **2a**, which showed NOE interactions between a methoxyl group at the C-5 position (δ 3.91) and aliphatic protons at C-1″ (δ 3.25 and 3.31). Compound **2** was assigned as 5,4′-dihydroxy-8-(3,3-dimethylallyl)-5-hydroxyisopropyldihydro[3,2:6,7]isoflavone.[5] This conclusion was confirmed by the X-ray crystallographic analysis of the *p*-bromobenzoyl derivative **2b** (see Experimental Section). The crystal structure of **2b** is illustrated in Figure 1. The ^{1}H NMR spectrum of **2** (OH-5, δ 12.97 in $CDCl_3$) was in agreement with the reported data of euchrenone b_{10} (OH-5, δ 12.94 in $CDCl_3$), previously described as structure **1**, which has been isolated from the roots of *Euchresta horsfieldii*.[11] Thus, the structure of euchrenone b_{10} is revised to structure **2**. The unambiguous assignments of the ^{1}H NMR and ^{13}C NMR signals of **1** and **2** were accomplished by analysis of their ^{1}H−^{1}H COSY, NOESY, HSQC, and HMBC spectra.

Experimental Section

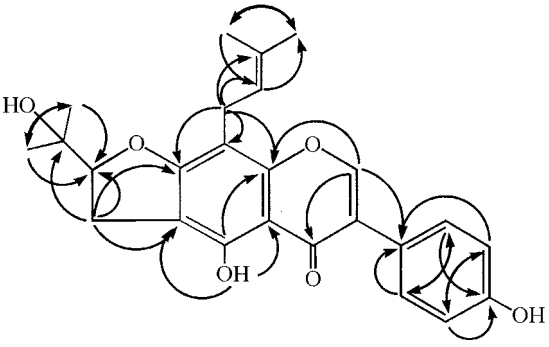
General Experimental Procedures. UV spectra were recorded on a Shimadzu UV-2100 spectrophotometer, and IR spectra were recorded on a JASCO IR-810 spectrophotometer. ^{1}H NMR spectra were obtained on a JEOL JNM-A 600 spectrometer, while ^{13}C NMR spectra were recorded at 150.8 MHz on the same instrument. Mass spectra were performed on a JEOL JMS-D 300 spectrometer. Column chromatography was performed using Si gel (230−400 mesh). TLC was carried out using Merck precoated Si gel 60 F_{254} plates. Spots were visualized using UV light and iodine vapor.

Plant Material. The dried wood of *E. suberosa* var. *glabrescence* was collected in Karachi, Pakistan, in July 1997. A voucher specimen (# 970701) was deposited in the Department



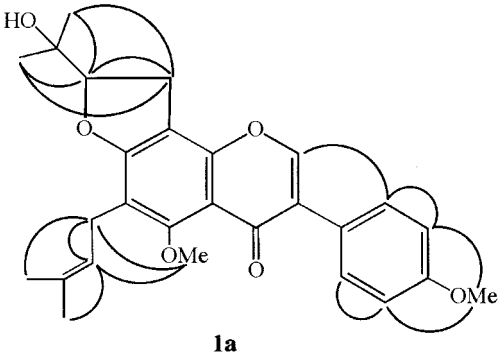
2

Figure 4. NOESY correlations observed for compound **2**.



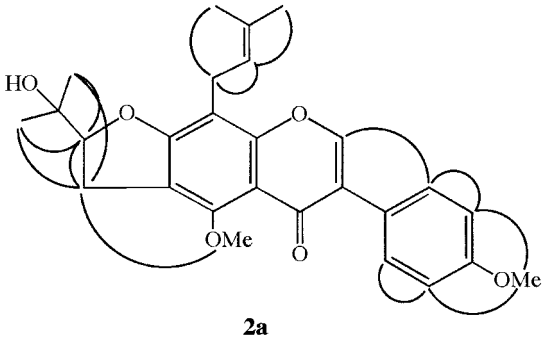
2

Figure 5. HMBC correlations observed for compound **2**.



1a

Figure 6. NOESY correlations observed for compound **1a**.



2a

Figure 7. NOESY correlations observed for compound **2a**.

of Natural Product Chemistry in the Faculty of Pharmacy, Meijo University.

Extraction and Isolation. The wood (2.55 kg) was extracted with acetone (36 L) and evaporated to give a dark green residue (133.7 g). The residue was divided into *n*-hexane-, CH_2Cl_2-, and EtOAc-soluble fractions. The CH_2Cl_2-soluble fraction (50 g) was chromatographed on Si gel and eluted with $CHCl_3$ (fractions A1−10), $CHCl_3$−acetone (10:1) (fractions A11−32), $CHCl_3$−acetone (1:1) (fractions A33−69), and $CHCl_3$−MeOH (10:1) (fractions A70−81) (each fraction; 200 mL, column A). Fraction A45 (2.5 g) was separated by Si gel column chromatography [benzene−EtOAc (10:1) (fractions B1−25), benzene−EtOAc (3:1) (fractions B26−41), and benzene−EtOAc (1:1) (fractions B42−60) (each fraction; 20 mL, column B)] to afford alpinumisoflavone[2] (73 mg; 0.0029% yield) (fractions B10−15) and wighteone[2] (124 mg; 0.0049% yield) (fractions B35−42) and fractions B43−60 (674 mg). The latter fractions were chromatographed on Si gel [benzene−EtOAc (10:1) (fractions $B_1$1−30) and benzene−EtOAc (5:1) (fractions $B_1$31−60) (each fraction; 15 mL, column B_1). Fractions $B_1$30−33 (32 mg) were purified by Si gel column chromatography [*n*-hexane−acetone (2:1)] to yield laburnetin[2] (3.2 mg; 0.00013% yield), and fractions $B_1$34−51 were separated by Si gel column chromatography [$CHCl_3$−acetone (20:1) (fractions $B_2$1−33) (each fraction; 5 mL, column B_2)] to furnish **1** (11.4 mg; 0.00045% yield) (fractions $B_2$15−20) and **2** (140.9 mg; 0.0055% yield) (fractions $B_2$25−30).

Compound 1 (senegalensin): colorless oil; UV (MeOH) λ_{max} (log ϵ) 202 (4.48), 216 (sh, 4.38), 271 (4.48) nm; IR ($CHCl_3$) ν_{max} 3600, 1660, 1630, 1610 cm^{-1}; ^1H NMR, see Table 1; ^{13}C NMR, see Table 2; EIMS *m*/*z* 422 ($[M]^+$, 91), 407 (21), 379 (72), 367 (100), 363 (6), 349 (14), 335 (14), 321 (11), 307 (39), 295 (41), 59 (9); HREIMS *m*/*z* 422.1722 (calcd for $C_{25}H_{26}O_6$, 422.1728); R_f 0.54 [$CHCl_3$−acetone (10:1.5)].

Methylation of Compound 1. A mixture of **1** (4.3 mg) and trimethylsilyldiazomethane (2.0 M solution in *n*-hexane) (0.5 mL) in MeOH (0.5 mL) was stirred at room temperature overnight. After the excess of trimethylsilyldiazomethane was decomposed with AcOH, the reaction mixture was evaporated to dryness. The resulting residue was purified by column chromatography on Si gel eluting with $CHCl_3$−acetone (10:1) to give **1a** (2.3 mg, 50%) as a colorless oil: UV (MeOH) λ_{max} (log ϵ) 202 (4.79), 217 (sh, 4.64), 259 (4.70), 313 (sh, 4.10) nm; IR (KBr) ν_{max} 3440, 1640, 1610 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.25, 1.37 (each 3H, each s, Me-4″ and -5″), 1.68, 1.79 (each 3H, each s, Me-4‴ and -5‴), 3.28 (1H, dd, J = 15.4, 8.8 Hz, H-1″), 3.33 (1H, dd, J = 15.4, 9.5 Hz, H-1″), 3.37 (1H, dd, J = 13.9, 7.3 Hz, H-1‴), 3.40 (1H, dd, J = 13.9, 7.3 Hz, H-1‴), 3.75 (1H, brs, OH), 3.83 (3H, s, OMe-4′), 3.86 (3H, s, OMe-5), 4.82 (1H, dd, J = 9.5, 8.8 Hz, H-2″), 5.21 (1H, t, J = 7.3 Hz, H-2‴), 6.96 (2H, d, J = 8.8 Hz, H-3′ and −5′), 7.45 (2H, d, J = 8.8 Hz, H-2′ and -6′), 7.77 (1H, s, H-2); EIMS *m*/*z* 450 ($[M]^+$, 67), 435 (75), 419 (18), 407 (25), 393 (19), 391 (11), 381 (100), 363 (27), 345 (14), 335 (18), 323 (13), 321 (13), 309 (58), 59 (12); HREIMS *m*/*z* 450.2047 (calcd for $C_{27}H_{30}O_6$, 450.2041).

Compound 2 (euchrenone b_{10}): colorless oil; UV (MeOH) λ_{max} (log ϵ) 202 (4.67), 214 (4.64), 269 (4.72) nm; IR ($CHCl_3$) ν_{max} 3600, 1670, 1630, 1620 cm^{-1}; ^1H NMR, see Table 1; ^{13}C NMR, see Table 2; EIMS *m*/*z* 422 ($[M]^+$, 100), 407 (19), 389 (18), 363 (25), 349 (29), 335 (18), 321 (17), 307 (13), 295 (25), 59 (12); HREIMS *m*/*z* 422.1728 (calcd for $C_{25}H_{26}O_6$, 422.1728); R_f 0.43 [$CHCl_3$−acetone (10:1.5)].

Methylation of Compound 2. A solution of **2** (12 mg) in MeOH (1.5 mL) was treated with trimethylsilyldiazomethane (2.0 M solution in *n*-hexane) (1.5 mL) and worked up in the same way as described for **1a** to give **2a** (8.2 mg, 64%) as a colorless oil: UV (MeOH) λ_{max} (log ϵ) 203 (4.69), 219 (sh, 4.53), 259 (4.69), 310 (sh, 4.17) nm; IR (KBr) ν_{max} 3430, 1640, 1610 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.24, 1.36 (each 3H, each s, Me-4″ and -5″), 1.60 (1H, brs, OH), 1.71, 1.81 (each 3H, each s, Me-4‴ and -5‴), 3.25 (1H, dd, J = 16.1, 8.1 Hz, H-1″), 3.31 (1H, dd, J = 16.1, 9.5 Hz, H-1″), 3.44 (2H, d, J = 7.3 Hz, H-1‴), 3.83 (3H, s, OMe-4′), 3.91 (3H, s, OMe-5), 4.75 (1H, dd, J = 9.5, 8.1 Hz, H-2″), 5.25 (1H, t, J = 7.3 Hz, H-2‴), 6.96 (2H, d, J = 8.8 Hz, H-3′ and −5′), 7.48 (2H, d, J = 8.8 Hz, H-2′ and -6′), 7.85 (1H, s, H-2); EIMS *m*/*z* 450 ($[M]^+$, 100), 421 (8), 391 (13), 373 (10), 361 (8), 331 (11), 323 (7), 178 (43), 59 (7); HREIMS *m*/*z* 450.2047 (calcd for $C_{27}H_{30}O_6$, 450.2041).

Benzoylation of Compound 2. A solution of *p*-bromobenzoyl chloride (154 mg) in CH_2Cl_2 (3 mL) was added dropwise to a stirred mixture of **2** (99 mg) and *N*,*N*-diisopropylethylamine (219 mg) in CH_2Cl_2 (20 mL) at 0 °C under a nitrogen atmosphere. The reaction mixture was stirred at room temperature overnight and then evaporated to dryness. The resulting residue was purified by column chromatography on Si gel eluting with $CHCl_3$−acetone (10:1.5) to give a *p*-bromobenzoate (90 mg) as a crude solid. The solid was recrystallized from EtOAc to afford **2b** (49 mg, 35%) as colorless needles: mp 176−178°; UV ($CHCl_3$) λ_{max} (log ϵ) 257 (sh, 4.58), 272 (4.61) nm; IR ($CHCl_3$) ν_{max} 3580, 1740, 1670, 1630 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.23, 1.35 (each 3H, each s, Me-4″ and -5″), 1.55 (1H, brs, OH-3″), 1.70, 1.80 (each 3H, each s, Me-4‴ and -5‴), 3.15 (1H, dd, J = 15.8, 7.7 Hz, H-1″), 3.23 (1H, dd, J = 15.8, 9.5 Hz, H-1″), 3.41 (2H, d, J = 7.3 Hz, H-1‴), 4.79 (1H, dd, J = 9.5, 7.7 Hz, H-2″), 5.23 (1H, t, J = 7.3 Hz, H-2‴), 7.29 (2H, d, J = 8.8 Hz, H-2′ and -6′), 7.62 (2H, d, J = 8.8 Hz, H-3′ and -5′), 7.67 (2H, d, J = 8.4 Hz, H-3⁗ and -5⁗), 7.98 (1H, s, H-2), 8.08 (2H, d, J = 8.4 Hz, H-2⁗ and -6⁗), 12.91 (1H, s, OH-5); EIMS *m*/*z* 606 ($[M + 2]^+$, 63), 604 ($[M]^+$, 63), 591 (7), 589 (7), 573 (6), 571 (6), 547 (9), 545 (9), 533 (6), 531 (6), 421 (15), 294 (5), 185 (100), 183 (100), 157 (19), 155 (19), 104 (6), 59 (12); HREIMS *m*/*z* 604.1084 (calcd for $C_{32}H_{29}O_7Br$, 604.1095).

X-ray Diffraction Study of 2b. Cell dimensions and diffraction data were measured on Rigaku AFC5R instrument, and the structure was solved by the direct method using SHELXS-97. Crystal data of **2b**: formula = $C_{32}H_{29}BrO_7$, M_r = 605.46, crystal size = 0.6 × 0.4 × 0.06 mm^3, color = yellow, monoclinic, *C*2/*c*, a = 39.908(7) Å, b = 11.446(2) Å, c = 13.789(2) Å, β = 92.11(1)°, V = 6294.7(19) $Å^3$, Z = 8, F(000) = 2496, μ(Cu Kα) = 2.138 mm^{-1}, D_x = 1.278 g cm^{-3}, absorption correction type = sphere (SPHERABS), T_{min}/T_{max} = 0.728/0.739, number of reflections = 4573, θ_{max} = 67.5°, number of reflections with over $2\sigma(I)$ = 3817, number of parameters = 363, R_1 = 0.1071, R_w = 0.2676, goodness of fit = 1.064, $(\Delta/\sigma)_{max}$ = 0.025, $\Delta\rho_{max}$ = 1.676 e $Å^{-3}$, $\Delta\rho_{min}$ = −0.606 e $Å^{-3}$. The space group of *C*2/*c* indicated that **2b** was a racemic mixture. The structure of **2b** was refined by SHELXL-97. Non-hydrogen atoms were refined with anisotropic temperature factors, and hydrogen atoms calculated on the ideal positions were refined with isotropic temperature factors and included in the calculations of the structure factors.

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References and Notes

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