New Prenylflavonoids from *Artocarpus communis*

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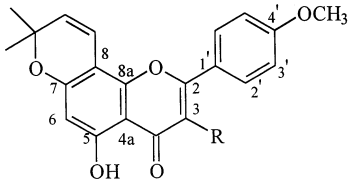
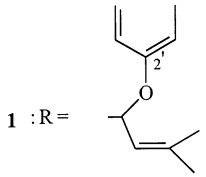
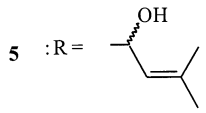
Five new prenylflavonoids, artocommunols CA (**1**), CB (**2**), CC (**3**), CD (**4**), and CE (**5**), were isolated from the cortex of the roots of *Artocarpus communis*, along with the known compound cyclomorusin. The structures of **1**−**5** were determined by spectral methods.

In previous papers,[1−18] we have reported the isolation and biological activities of phenolic compounds from *Artocarpus communis* Forst, *A. heterophyllus* Lamk, and *Artocarpus rigida* Blume (Moraceae). As part of a continued investigation on the constituents of *Artocarpus* species, five new prenylflavonoids, artocommunols CA (**1**), CB (**2**), CC (**3**), CD (**4**), and CE (**5**), were further isolated from *A. communis*, along with cyclomorusin.[1,19] In the present paper the isolation and structure elucidation of **1**−**5** are reported.

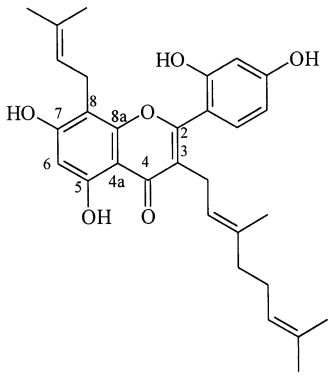




1 : R =



5 : R =



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The HREIMS of **1** revealed a $[M]^+$ peak at *m/z* 432.1580, which corresponded to a molecular formula of $C_{26}H_{24}O_6$. The IR spectrum of **1** showed hydroxyl and chelated carbonyl absorption bands at 3449 and 1654 cm^{-1}, respectively. The UV spectrum of **1** exhibited absorption maxima (220, 280, 360, and 380 nm) suggestive of a 5,7,2′,4′-tetraoxygenated flavone derivative.[1,19] The ^1H and ^{13}C NMR spectra (Table 1 and Experimental Section) were similar to those of cyclomorusin[1,19] except for an additional methoxyl proton signal at δ 3.84 (s) and a methoxyl carbon signal at δ 55.6 present in the ^1H and ^{13}C NMR spectra of **1**, respectively. Accordingly, artocommunol CA (**1**) was characterized as 4′-*O*-methylcyclomorusin (**1**) [5-hydroxy-4′-methoxy-7,8-(2,2-dimethyl-6*H*-pyrano)-9-(2-methylpropenyl)-9*H*-chromeno[4,3-*b*]chromen-4-one] (**1**). The ^1H and ^{13}C NMR data were assigned by comparing with those of related spectral data reported in the literature.[1,7,19]

The HREIMS of **2** gave a molecular ion peak at *m/z* 556.2860, indicating a molecular formula of $C_{35}H_{40}O_6$. The IR spectrum of **2** showed hydroxyl and chelated carbonyl absorption bands at 3373 and 1650 cm^{-1}, respectively. The UV spectrum of **2** exhibited absorption maxima similar to those of **1**. The ^1H NMR ($CDCl_3$) spectrum of **2** showed signals for a prenyl group at δ 1.57, 1.69 (each 3H, s), 3.32 (2H, d, J = 6.8 Hz), and 5.21 (1H, t, J = 6.8 Hz), a geranyl group at δ 1.25, 1.57, and 1.69 (each 3H, s), 1.69 and 1.75 (each 1H, m), 2.08 (2H, m), 3.14 (2H, d, J = 6.4 Hz), 5.06 (1H, t, J = 7.2 Hz), and 5.08 (1H, t, J = 6.4 Hz), a 2,2-dimethylpyran ring at δ 1.27 and 1.76 (each 3H, s), 5.44 (1H, d, J = 10 Hz), and 6.69 (1H, d, J = 10 Hz), three aromatic proton signals at δ 6.51 (1H, d, J = 8.4 Hz), 6.55 (1H, dd, J = 8.4, 2.0 Hz), and 7.16 (1H, d, J = 8.4 Hz), and three phenolic proton signals at δ 7.05 (1H, s), 7.26 (1H, s), and 13.14 (1H, s).[20] In addition, the UV spectrum of **2** showed no bathochromic shift upon addition of aluminum chloride and the presence of a bathochromic shift upon addition of sodium methoxide. On the basis of the above evidence, **2** could be suggested as being a 5,2′,4′-trihydroxy-3,6,7,8-tetrasubstituted flavone.[21] The HMBC correlations between the methylene proton signal at δ 3.32 and carbon signal at δ 158.2, the chelated phenolic proton signal at δ 13.14 and the carbon signal at δ 158.2, and the methylene proton signal at δ 3.14 and the carbon signal at δ 182.5, helped establish the structure of artocommunol CB (**2**) as 5,2′,4′-trihydroxy-3-geranyl-7,8-(2,2-dimethyl-6*H*-pyrano)-6-prenylflavone (**2**). The EIMS showed significant fragmentation peaks at *m/z* 541 $[M - 15]^+$, 513 $[M - a]^+$, and 473 $[M - b]^+$ (Figure 1), which further supported the structure of **2**. A combination of 2D NMR techniques, such as $^1H-^1H$ COSY, HMQC, HMBC, and NOESY experiments, also supported the characterization of **2** and enabled the assignment of the ^1H and ^{13}C NMR data for **2** (Table 1 and Experimental Section).

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Table 1. ^{13}C NMR Chemical Shifts of Compounds **1**−**5**[a]

carbon	**1**[b]	**2**[b]	**3**[c]	**4**[c]	**5**[c]
2	158.1	160.4	158.5	162.2	157.2
3	105.6	120.8	117.4	121.0	110.6
4	178.5	182.5	181.9	183.2	179.9
4a	101.3	104.4	104.3	105.1	106.7
5	151.1	158.2	162.5	160.7	160.6
6	100.2	112.3	99.7	98.6	101.1
7	161.8	157.5	160.5	161.7	160.6
8	108.5	100.4	101.5	106.6	103.0
8a	159.1	150.5	152.5	156.4	152.7
9	69.9	24.3	25.6	24.5	71.0
10	120.9	121.0	86.3	122.7	122.7
11	139.2	132.9	83.3	131.4	139.6
12	18.6	17.5	22.4	16.0	26.5
13	25.8	41.6	20.1	40.3	19.3
14	114.8	22.7	116.0	27.2	122.7
15	124.7	123.7	127.2	125.0	129.4
16	77.9	131.5	81.3	131.8	79.5
17	28.1	17.5	27.0	17.6	29.0
18	28.1	25.6	42.0	25.7	29.0
19		21.2	23.2	21.9	
20		122.0	124.7	123.0	
21		131.8	132.1	135.2	
22		25.7	17.6	16.0	
23		25.6	25.7	16.0	
24		115.6			
25		125.4			
26		80.4			
27		17.9			
28		26.9			
1′	109.7	112.1	114.7	113.0	109.0
2′	155.0	155.2	161.4	157.1	159.8
3′	102.2	103.6	108.5	103.7	105.5
4′	164.6	159.4	162.2	161.2	164.9
5′	109.1	108.2	112.2	107.8	111.6
6′	127.5	131.5	131.0	132.1	127.1
OMe	55.6				

[a] The number of protons directly attached to each carbon was verified by DEPT and HMQC experiments. [b] Measured in $CDCl_3$. [c] Measured in acetone-d_6.

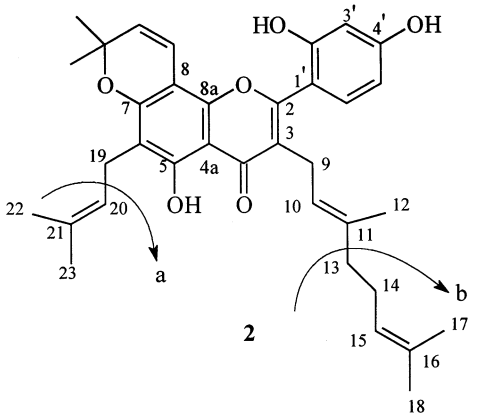

Figure 1. EIMS fragmentation patterns of **2**.

The molecular formula of **3** was determined as $C_{30}H_{32}O_7$ by HREIMS (*m*/*z* 504. 2153 $[M]^+$). The IR absorptions of **3** implied the presence of OH (3365 cm^{-1}), conjugated CO (1650 cm^{-1}), and aromatic ring (1600 cm^{-1}) moieties. The UV spectrum of **3** exhibited absorption maxima similar to those of "compound A"[19] and artocarpol B.[16,19] The ^1H NMR data of **3** (Experimental Section) were very similar to those of artocarpol B, except for the proton signals of H-6, H-10, Me-12, Me-13, and H-14.[16] The ^{13}C NMR data of **3** (Table 1) were also very similar to those of artocarpol B, except for the carbon signals of C-5, C-6, C-8, C-8a, C-10, C-11, C-12, C-13, and C-2′.[16] The NOESY correlations of H_β-9/H-10 and H_α-9/H-13 suggested a β-configuration for H-10 with the bond between C-10 and C-11 located on the α-side

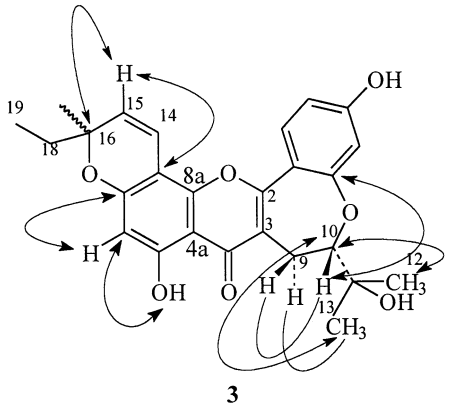

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Figure 2. Some key HMBC (↔) and NOESY (—) correlations of **3**.

of **3** (Figure 2), while the NOESY correlation of H_β-9/Me-13 and the coupling constants of H_2-9 and H-10 of artocarpol B suggested that the bonds between C-10 and C-11, and C-10 and O−C-2′, were located on the β- and α-sides of artocarpol B, respectively. In addition, the UV spectrum of **3** showed a bathochromic shift upon addition of aluminum chloride.[21] The OH-5 resonance showed a HMBC correlation with C-6 (Figure 1) and exhibited an optical rotation different from that of artocarpol B.[16] On the basis of the above evidence, artocommunol CC (**3**) was characterized as the C-10 stereoisomer of artocarpol B.[16] The stereochemistry at C-16 was not resolved in compound **3**.

The HREIMS of **4** gave a molecular ion peak at *m*/*z* 490.2362, which was consistent with the ^1H and ^{13}C NMR data. The IR absorption of **4** implied the presence of OH (3373 cm^{-1}), conjugated CO (1650 cm^{-1}), and aromatic ring (1617 cm^{-1}) moieties. The UV spectrum of **4** exhibited absorption maxima similar to that of **2**. The ^1H NMR (acetone-d_6) spectrum of **4** showed the signal of a prenyl group at δ 1.55 (6H, s), 3.35 (2H, d, J = 7.2 Hz), and 5.20 (1H, m), a geranyl group at δ 1.41, 1.50, and 1.57 (each 3H, s), 1.87 and 1.96 (each 2H, m), 3.11 (2H, d, J = 7.2 Hz), 5.00 (1H, m), and 5.10 (1H, m), a 2′,4′-dihydroxy-substituted B ring at δ 6.47 (1H, dd, J = 8.0, 2.0 Hz), 6.54 (1H, d, J = 2.0 Hz), and 7.17 (1H, d, J = 8.0 Hz), an aromatic singlet signal at δ 6.31, and a phenolic proton signal at δ 13.05 (1H, s). In addition, the UV spectrum of **4** showed bathochromic shifts upon addition of aluminum chloride, sodium acetate, and sodium methoxide. On the basis of the above evidence, **4** was suggested to be a 5,7,2′,4′-tetrahydroxy-3,8-disubstituted flavone.[21] The HMBC correlations between the methylene proton signals at δ 3.11 and 3.35 and carbon signal at δ 183.2 and 156.4, respectively, and chelated phenolic proton signal at δ 13.05 and carbon signal at δ 98.6, established the structure of artocommunol CD (**4**) as 5,7,2′,4′-tetrahydroxy-3-geranyl-8-prenylflavone (**4**). A combination of 2D NMR techniques also supported the characterization of **4** and enabled the assignments of the ^1H and ^{13}C NMR data for **4** (Table 1 and Experimental Section).

The molecular formula of **5** was determined to be $C_{25}H_{24}O_7$ by HREIMS (*m*/*z* 418.1425 $[M - 18]^+$). The IR absorption of **5** implied the presence of OH (3394 cm^{-1}), conjugated CO (1657 cm^{-1}), and aromatic ring (1617 cm^{-1}) moieties. The UV spectrum of **5** exhibited absorption maxima similar to that of **1**. The ^1H NMR (acetone-d_6) spectrum of **5** showed a 2,2-dimethylpyran ring at δ 1.47 (6H, s), 5.77 (1H, d, J = 10 Hz), and 6.91 (1H, d, J = 10 Hz), two tertiary methyl signals at δ 1.68 (3H, s) and 1.94 (3H, s), a methine proton signal at δ 6.19 (1H, d, J = 9.6 Hz), a 2′,4′-dihydroxy-substituted B ring at δ 6.43 (1H, d,

J = 2.4 Hz), 6.63 (1H, dd, J = 8.8, 2.4 Hz), and 7.79 (1H, d, J = 8.8 Hz), a singlet aromatic signal at δ 6.15, and a phenolic proton signal at δ 12.92 (1H, s). In addition, the UV spectrum of **5** showed bathochromic shifts upon addition of aluminum chloride and sodium methoxide. On the basis of the above evidence, **5** was suggested to be a 5,2′,4′-trihydroxy-3,7,8-trisubstituted flavone.[21] The HMBC correlations of Me-12 and Me-13/C-11, H-10/C-12, and H-9/C-2 and C-3, the ^{1}H−^{1}H COSY correlation between H-9/H-10, the NOESY correlation between Me-13/H-9, and the HMQC correlation between H-9/C-9 established the connectivity between C-3 and C-9. In addition, the HMBC correlations of H-6/C-5, H-14/C-7 and C-8, and H-15/C-8 and C-16 established the proposed structure for artocommunol CE (**5**) as 5,2′,4′-trihydroxy-7,8-(2,2-dimethyl-6*H*-pyrano)-3-(9-hydroxy)prenylflavone (**5**). Further experiments are required to elucidate the absolute configuration of **5**.

Experimental Section

General Experimental Procedures. Melting points were recorded on a Yanaco micro-melting point apparatus and reported uncorrected. Optical rotations were obtained on a JASCO model DIP-370 digital polarimeter. UV spectra were obtained on a JASCO model 7800 UV−vis spectrophotometer. IR spectra were recorded on a Perkin-Elmer system 2000 FT-IR spectrophotometer. ^{1}H (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a Varian Unity-400 spectrometer, and MS were obtained on a JMS-HX 100 mass spectrometer.

Plant Material. The roots of *Artocarpus communis* (13.5 kg) were collected at Kaohsiung Hsien, Taiwan, during November 2001, and a voucher specimen (2001-3) has been deposited in the Department of Medicinal Chemistry, School of Pharmacy, Kaohsiung Medical University.

Extraction and Isolation. The cortex of the roots (0.52 kg) of *A. communis* was chipped and extracted with $CHCl_3$ at room temperature. The $CHCl_3$ extract of the cortex was chromatographed over a Si gel column, and elution with *n*-hexane−EtOAc (5:1) yielded cyclomorusin (2.7 g), while elution with *n*-hexane−EtOAc (6:1) yielded **1** (8.9 mg) and with CH_2Cl_2−EtOAc (15:1) yielded **2** (11.6 mg). Elution with $CHCl_3$−EtOAc (4:1) yielded **3** (350 mg), while elution with *n*-hexane−EtOAc (5:1) yielded **4** (21.6 mg) and with *n*-hexane−EtOAc (7:3) yielded **5** (17.3 mg). Cyclomorusin was identified by spectroscopic methods and comparison with the spectral data obtained from an authentic sample.[1,19]

Artocommunol CA (1): yellowish needles ($CHCl_3$); mp 190−192 °C; $[\alpha]_D^{25}$ 61.8° (*c* 0.1, $CHCl_3$); UV (MeOH) λ_{max} (log ϵ) 220 (4.36), 280 (4.23), 360 (3.91), 380 (3.92) nm, (MeOH−$AlCl_3$) 210, 260 (sh), 285, 385, 425 nm, (MeOH−NaOAc) unchanged, (MeOH−NaOAc−H_3BO_3) unchanged, (MeOH−NaOMe) unchanged; IR (KBr) ν_{max} 3449, 1654, 1570 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.47 (6H, s, Me-17 and Me-18), 1.70 (3H, s, Me-12), 1.98 (3H, s, Me-13), 3.84 (3H, s, OMe-4′), 5.44 (1H, d, J = 9.6 Hz, H-10), 5.61 (1H, d, J = 10.0 Hz, H-15), 6.25 (1H, s, H-6), 6.26 (1H, d, J = 9.6 Hz, H-9), 6.48 (1H, d, J = 2.8 Hz, H-3′), 6.61 (1H, dd, J = 8.4, 2.8 Hz, H-5′), 6.67 (1H, d, J = 10.0 Hz, H-14), 7.66 (1H, d, J = 8.4 Hz, H-6′), 12.83 (1H, s, OH-5); ^{13}C NMR ($CDCl_3$), see Table 1; EIMS (70 eV) *m*/*z* 432 $[M]^+$ (37), 417 (100), 377 (40), 361 (29), 203 (40); HREIMS *m*/*z* $[M]^+$ 432.1580, calcd for $C_{26}H_{24}O_6$, 432.1573.

Artocommunol CB (2): yellow needles ($CHCl_3$); mp 217−219 °C; UV (MeOH) λ_{max} (log ϵ) 240 (4.61), 280 (4.59), 325 (4.24) nm, (MeOH−$AlCl_3$) unchanged, (MeOH−NaOAc) unchanged, (MeOH−NaOAc−H_3BO_3) unchanged, (MeOH−NaOMe) 210, 265 (sh), 390 nm; IR (KBr) ν_{max} 3373, 1650, 1550 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.25 (3H, s, Me-12), 1.27 (3H, s, Me-27), 1.57 (6H, s, Me-17 and Me-22), 1.69, 1.75 (each 1H, m, H-13), 1.69 (6H, s, Me-18 and Me-23), 1.76 (3H, s, Me-28), 2.08 (2H, m, H-14), 3.14 (2H, d, J = 6.4 Hz, H-9), 3.32 (2H, d, J = 6.8 Hz, H-19), 5.06 (1H, t, J = 7.2 Hz, H-15), 5.08 (1H, d, J = 6.4 Hz, H-10), 5.21 (2H, t, J = 6.8 Hz, H-20), 5.44 (1H, d, J = 10 Hz, H-25), 6.51 (1H, dd, J = 8.4 Hz, H-5′), 6.55 (1H, d, J = 2.0, H-3′), 6.69 (1H, d, J = 10 Hz, H-24), 7.16 (1H, d, J = 8.4 Hz, H-6′); ^{13}C NMR ($CDCl_3$), see Table 1; EIMS (70 eV) *m*/*z* 556 $[M]^+$ (21), 541 $[M - 15]^+$ (6), 513 $[M - a]^+$ ($M - C_3H_7$, 6), 501 (10), 473 $[M - b]^+$ ($M - C_6H_{11}$,100), 215 (45); HREIMS, *m*/*z* $[M]^+$ 556.2860, calcd for $C_{35}H_{40}O_6$, 556.2825.

Artocommunol CC (3): yellow amorphous powder; $[\alpha]_D^{25}$ 43.1° (*c* 0.1, MeOH); UV (MeOH) λ_{max} (log ϵ) 240 (4.58), 275 (4.61), 340 (4.34) nm, (MeOH−$AlCl_3$) 270, 290, 365 nm, (MeOH−NaOMe) 280, 385 nm, (MeOH−NaOAc) unchanged, (MeOH−NaOAc−H_3BO_3) unchanged; IR (KBr) ν_{max} 3365, 1650, 1600, 1560 cm^{-1}; ^{1}H NMR (acetone-d_6, 400 MHz) δ 1.30 (3H, s, Me-13), 1.44 (6H, s, Me-12 and Me-17), 1.55 (3H, s, Me-22), 1.63 (3H, s, Me-23), 1.76 (2H, m, H-18), 2.10 (2H, m, H-19), 2.59 (1H, dd, J = 16.8, 9.6 Hz, H_α-9), 3.54 (1H, dd, J = 16.8, 2.0 Hz, H_β-9), 4.35 (1H, dd, J = 9.6, 2.0 Hz, H-10), 5.10 (1H, t, J = 7.2 Hz, H-20), 5.70 (1H, d, J = 10.4 Hz, H-15), 6.14 (1H, s, H-6), 6.61 (1H, d, J = 2.0 Hz, H-3′), 6.78 (1H, dd, J = 8.0, 2.0 Hz, H-5′), 6.88 (1H, d, J = 10.4 Hz, H-14), 8.01 (1H, d, J = 8.0 Hz, H-6′); ^{13}C NMR (acetone-d_6), see Table 1; EIMS (70 eV) *m*/*z* 504 $[M]^+$ (3.9), 421 (100), 403 (10), 363 (19), 345 (14), 333 (8), 203 (24); HREIMS *m*/*z* $[M]^+$ 504.2153, calcd for $C_{30}H_{32}O_7$, 504.2148.

Artocommunol CD (4): pale yellow needles (acetone); mp 183−185 °C; UV (MeOH) λ_{max} (log ϵ) 210 (4.62), 265 (4.39), 325 (3.93) nm, (MeOH−$AlCl_3$) 220, 275, 340 nm; (MeOH−NaOAc) 215, 270, 330 nm; (MeOH−NaOMe−H_3BO_3) unchanged; (MeOH−NaOMe) 220, 280, 370 nm; IR (KBr) ν_{max} 3373, 1650, 1617, 1558 cm^{-1}; ^{1}H NMR (acetone-d_6, 400 MHz) δ 1.41 (3H, s, Me-18), 1.50 (3H, s, Me-17), 1.55 (6H, s, Me-22 and Me-23), 1.57 (3H, s, Me-12), 1.87 (2H, m, H-13), 1.96 (2H, m, H-14), 3.11 (2H, d, J = 7.2 Hz, H-9), 3.35 (2H, d, J = 7.2 Hz, H-19), 5.00 (1H, m, H-15), 5.10 (1H, m, H-10), 5.20 (1H, m, H-20), 6.31 (1H, s, H-6), 6.47 (1H, dd, J = 8.0, 2.0 Hz, H-5′), 6.54 (1H, d, J = 2.0 Hz), 7.17 (1H, d, J = 8.0 Hz), 13.05 (1H, s, OH-5); ^{13}C NMR (acetone-d_6), see Table 1; EIMS (70 eV) *m*/*z* 490 $[M]^+$ (6), 421 (7), 367 (17), 311 (13), 219 (43), 165 (100); HREIMS *m*/*z* $[M]^+$ 490.2362, calcd for $C_{30}H_{34}O_6$, 490.2355.

Artocommunol CE (5): yellow amorphous powder; $[\alpha]_D^{25}$ 45.5° (*c* 0.1, MeOH); UV (MeOH) λ_{max} (log ϵ) 225 (4.08), 275 (4.19), 375 (2.60) nm, (MeOH−$AlCl_3$) 265, 285, 415 nm, (MeOH−NaOMe) 240, 285, 410 nm, (MeOH−NaOAc) unchanged, (MeOH−NaOAc−H_3BO_3) unchanged; IR (KBr) ν_{max} 3394, 1657, 1617, 1563 cm^{-1}; ^{1}H NMR (acetone-d_6, 400 MHz) δ 1.47 (6H, s, Me-17 and Me-18), 1.68 (3H, d, J = 1.6 Hz, Me-12), 1.94 (3H, d, J = 1.6 Hz, H-13), 5.48 (1H, d, J = 9.6 Hz, H-10), 5.77 (1H, d, J = 10 Hz, H-15), 6.15 (1H, s, H-6), 6.19 (1H, d, J = 9.6 Hz, H-9), 6.43 (1H, d, J = 2.4 Hz, H-3′), 6.63 (1H, dd, J = 8.8, 2.4 Hz, H-5′), 6.91 (1H, dd, J = 10.0 Hz, H-14), 7.79 (1H, d, J = 8.8 Hz, H-6′); ^{13}C NMR (acetone-d_6), see Table 1; EIMS (70 eV) *m*/*z* 418 $[M - H_2O]^+$ (42), 403 (100), 363 (23), 203 (17), 194 (11); HREIMS *m*/*z* $[M - 18]^+$ 418.1425, calcd for $C_{25}H_{22}O_6$, 418.1416.

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