Phenolic Glycosides from *Viscum angulatum*

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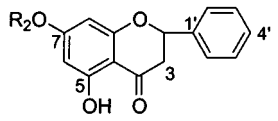
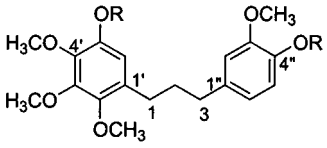
Three new glycosides, pinocembrin 7-*O*-apiosyl(1→5)apiosyl(1→2)-*β*-D-glucopyranoside (**1**), 2′,3′,4′,3″-tetramethoxy-1,3-diphenylpropane 5′,4″-di-*O*-*β*-D-glucopyranoside (**2**), and rhamnocitrin 3-*O*-apiosyl(1→5)-apiosyl(1→2)-[α-L-rhamnopyranosyl(1→6)]-*β*-D-glucopyranoside (**3**), were isolated from *Viscum angulatum* along with viscumneoside V, naringenin, and homoeriodictyol. Their structures were established by spectral and chemical methods.

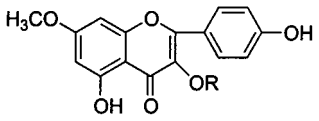
The mistletoes have been used to treat hypertension, atherosclerosis, rheumatism, neuralgia, etc., in Chinese medicine.[1] Flavonoids have been extensively isolated and characterized.[2−10] *Viscum angulatum* Heyne in DC. (Loranthaceae) is a perennial parasitic plant distributed at lower altitudes of the central and southern parts of Taiwan.[11] Its stems have been used for the treatment of arthritis and hypertension.[1] The only previous phytochemical studies afforded the isolation of long chain alkanes, long chain alcohols, long chain fatty acids, *β*-amyrin fatty acid esters, *β*-amyrin acetate, betulic acid, and oleanolic acid.[12] As a part of our interest in polyphenolic compounds, a continuing chemical investigation was conducted on the 70% acetone-soluble fraction. In this paper, we report the isolation and structural elucidation of three new phenolic glycosides (**1**−**3**).



1 R=apiosyl (1⟶5)apiosyl(1⟶2)–*β*-D-glucosyl
5 nonaacetate of **1**



2 R=*β*-D-glucosyl
4 R=H
6 octaacetate of **2**



3 R=apiosyl (1⟶5)apiosyl(1⟶2)-[*α*-
-L- rhamnosyl (1⟶6)]–*β*-D-glucosyl
7 decaacetate of **3**

Results and Discussion

An aqueous acetone extract from the whole plants of *Viscum angulatum* was partitioned between chloroform and water. After concentration, the water layer was subjected to column chromatography over Diaion HP-20, RP-18, and Sephadex LH-20 to yield glycosides **1**−**3** and the known flavanones viscumneoside V,[3] naringenin,[13] and homoeriodictyol.[11]

Compound **1** was isolated as a colorless amorphous solid. The UV characteristic absorption bands (284 and 326 sh nm) and the ABX system protons at *δ* 2.81 (dd, *J* = 17.0, 2.81 Hz), 3.14 (m), and 5.56 (dd, *J* = 12.0, 2.8 Hz) in the ^{1}H NMR spectrum showed **1** to be a flavanone[14] with the molecular formula $C_{31}H_{38}O_{17}$ as determined by ^{13}C and DEPT NMR and HRFABMS. The positive-ion FABMS of **1** showed a quasi-molecular ion $[M + Na]^+$ at *m*/*z* 705 and a $[M + H]^+$ at *m*/*z* 683, fragment peaks at *m*/*z* 551 $[M + H - 132]^+$ due to the cleavage of a pentose, at *m*/*z* 419 $[M + H - 132 - 132]^+$ due to the subsequent loss of another pentose, and the aglycone peak at *m*/*z* 257 $[M + H - 132 - 132 - 162]^+$ due to loss of the other hexosyl moiety. The ^{1}H NMR showed a monosubstituted phenyl group at *δ* 7.35−7.41 (3H, m) and 7.46 (2H, dd, *J* = 8.0, 2.0 Hz), two *meta*-coupled phenyl protons at *δ* 6.07 and 6.09 (1H each, d, *J* = 20 Hz), phenolic hydroxyl groups at *δ* 11.85 (D_2O exchangeable), and three anomeric protons at *δ* 4.67 (1H, br s), 5.02 (1H, d, *J* = 7.5 Hz), and 5.23 (1H, br s). The presence of a signal at *δ* 11.85 and a bathochromic shift of 24 nm of band II in the presence of $AlCl_3$/HCl indicated that C-5 was hydroxylated.[14] The 7-*O*-glycoside was determined by $^3J_{C-H}$ correlation between the anomeric proton at *δ* 5.02 (H-1″) and C-7 in the HMBC spectrum and NOESY correlations between H-1″ and H-6 and between H-1″ and H-8. In addition to FABMS fragmentations, the trisaccharide moiety of this molecule was evident from the signals of three anomeric protons [*δ* 4.67 (br s), 5.02 (d, *J* = 7.5 Hz), and 5.23 (br s)] in the ^{1}H NMR spectrum and their corresponding carbons at *δ* 109.3, 97.0, and 109.3 in the ^{13}C NMR spectrum, respectively. The chemical shifts of the anomeric carbons suggested one pyranoglycosyl and two furanoglycosyl groups. Comparison of the ^{1}H and ^{13}C NMR spectra of **1** with those of viscumneoside V showed they had the same glycosyl moiety.[3] Acidic hydrolysis of **1** yielded pinocembrin,[15] and glucose and apiose were detected from the filtrate by TLC after evaporation. The linkage of the sugar moiety was identified as apiosyl(1→5)-apiosyl (1→2)-*β*-D-glucopyranoside by 1D TOCSY, DEPT, and HMBC experiments. The HMBC revealed correlations between apiosyl H-1‴ and glucosyl C-2″ and between apiosyl H-1⁗ and apiosyl C-5‴. Thus, compound **1** was

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assigned as pinocembrin 7-*O*-[apiosyl(1→5)apiosyl(1→2)]-*β*-D-glucopyranoside. Acetylation of **1** afforded the corresponding nonaacetate (**5**). This result was also consistent with the proposed structure.

Compound **2** was obtained as a colorless amorphous solid. It had molecular formula $C_{31}H_{44}O_{16}$ from its HR-FABMS and ^{13}C NMR spectrum. The ^1H NMR spectrum revealed two *β*-configured anomeric protons at *δ* 4.82 (d, *J* = 7.0 Hz), four methoxyl groups at *δ* 3.70, 3.71, 3.72, and 3.74, three methylene protons at *δ* 1.69 (quin, *J* = 7.0 Hz), 2.42 (t, *J* = 7.0 Hz), and 2.51 (t, *J* = 7.0 Hz), an isolated phenyl proton at *δ* 6.63, and ABX system protons at *δ* 6.67 (dd, *J* = 8.0, 2.0 Hz), 6.79 (d, *J* = 2.0 Hz), and 6.96 (d, *J* = 8.0 Hz). The ^{13}C NMR spectrum showed 15 signals that were assigned to the aglycone moiety, 12 signals belonging to two glucoside moieties and four methoxyl groups from HMQC and HMBC. The aglycone moiety included three methylene carbons and two phenyl groups with six oxygenated carbons. The oxygenated carbons were assigned by $^3J_{C-H}$ correlations between methoxyl protons and the oxygenated carbon and between the anomeric proton and the oxygenated carbon in the HMBC spectrum and NOESY correlations between the methoxyl and H-2″, between one of the anomeric protons and H-5″, and between another anomeric proton and the isolated phenyl proton. Acetylation of **2** afforded octaacetate (**6**) (*δ* 2.04, 2.04, 2.05, 2.05, 2.06, 2.06, 2.08, and 2.10). Acid hydrolysis of **2** gave glucose and 5′,4″-dihydoxy-2′,3′,4′,3″-tetramethoxy-1,3-diphenylpropane (**4**). Compound **4** has not been reported previously. The structure of **4** was also determined by spectral methods. Accordingly, compound **2** was assigned as 2′,3′,4′,3″-tetramethoxy-1,3-diphenylpropane 5′,4″-di-*O*-*β*-D-glucopyranoside. The FABMS showed fragments at *m/z* 511 $[M + H - 162]^+$ and *m/z* 349 $[M + H - 162 - 162]^+$ also supporting the shown structure.

Compound **3** was isolated as a yellowish amorphous solid. The IR spectrum showed hydroxyl, conjugated carbonyl, and aromatic absorption bands. On the basis of the bathochromic shift with diagnostic reagents in the UV spectrum and ^1H and ^{13}C NMR spectral data, compound **3** was a flavonol with 3-*O*-glycosidation.[14,16] The HRFABMS gave molecular formula $C_{38}H_{49}O_{23}$. Positive ion FABMS of **3** indicated a quasi-molecular ion at *m/z* 895 $[M + Na]^+$ and *m/z* 873 $[M + H]^+$ and fragments at *m/z* 763 $[M + Na - 132]^+$ and *m/z* 301 $[M + H - 132 - 132 - 146 - 162]^+$. Acidic hydrolysis of **3** gave rhamnocitrin,[17] and apiose, glucose, and rhamnose were detected. The ^1H NMR spectrum showed four anomeric protons at *δ* 4.25 (br s), 4.90 (br s), 5.30 (br s), and 5.34 (d, *J* = 7.0 Hz) and their corresponding carbons at *δ* 101.2, 109.3, 110.6, and 99.4 in the ^{13}C NMR spectrum, respectively. Comparison of the NMR spectra data with compound **1** revealed that the signals of the sugar moiety had one more rhamnosyl unit and the C-6 carbon signal of glucosyl downfield shifted. Acetylation of **3** afforded decaacetate (**7**). Analysis of 1D TOCSY, DEPT, and HMQC spectra of **7** revealed no downfield shift of H-6 of the glucosyl unit upon acetylation. Therefore, compound **3** was established as rhamnocitrin 3-*O*-apiosyl(1→5)apiosyl(1→2)-[α-L-rhamnopyranosyl(1→6)]-*β*-D-glucopyranoside (**3**).

Experimental Section

General Experimental Procedures. Melting points were determined on a Yanagimoto micromelting point apparatus and are uncorrected. IR spectra were recorded on a Nicolet Avatar 320 FT-IR spectrophotometer. Optical rotations were recorded on a JASCO DIP-370 polarimeter. UV spectra were measured on a Hitachi U-3200 spectrophotometer. NMR were run on a Varian Unity INOVA-500 spectrometer. Mass spectra (EIMS, FABMS, and HRFABMS) were taken on JEOL JMS-HX110 and JEOL SX-102A mass spectrometers, respectively. Column chromatography was performed on Diaion HP-20 (Mitsubishi) and RP-18 and Sephadex LH-20 (Pharmacia). Silica gel 60F254 (Merck) was used for TLC with the lower phase of $CHCl_3$−MeOH−H_2O (65:35:10) as developing solvent.

Plant Material. Whole plants of *V. angulatum* (parasitic on *Diospyros kaki*) were collected from Cha-Yi County, Taiwan, in May 1998. The plant was identified by Mr. Nien-Yung Chiu, Technician of the Institute of Chinese Pharmaceutical Science, China Medical College, Taichung, Taiwan, and comparison with the voucher specimens already deposited at the Herbarium of the Department of Botany, National Taiwan University, Taipei, Taiwan (no. TAI 041085, collected in 1975).

Extraction and Isolation. The air-dried *V. angulatum* (5 kg) was extracted with 70% acetone (each 20 L for 7 days, × 3) at room temperature. The extracts were combined and evaporated under reduced pressure to give an aqueous suspension. The aqueous suspension was partitioned with $CHCl_3$ to give $CHCl_3$ and H_2O layers. The water layer was concentrated to about 1.1 L and subjected to column chromatography over Diaion HP-20 (7.5 × 38 cm) using H_2O−MeOH gradient elution to give nine fractions. The fractions of 90% MeOH (77 g) and MeOH (11 g) eluates were rich in flavonoids. The MeOH eluate was separated by a Sephadex LH-20 column (5.0 × 45 cm, eluted with 50% MeOH−80% MeOH gradient). The 80% MeOH eluate (250 mg) was chromatographed on a Si gel column (3.0 × 30 cm, $CHCl_3$−15% EtOAc gradient) to give homoeriodictyol[11] (37 mg) and naringenin[13] (45 mg). The 90% MeOH eluate was further purified by a Sephadex LH-20 column (8 × 50 cm, eluted with H_2O, 10% and 20% MeOH, sequentially) to give the 10−20% MeOH eluted fractions rich in flavonoid glycosides (15 g), which were combined and subjected to Diaion HP-20 column chromatography (7.5 × 58 cm, 30−90% MeOH gradient). The 50−80% MeOH eluate (3.2 g) was further purified by Sephadex LH-20 (40−60% MeOH) and Cosmosiol 75 C_{18} OPN columns (4.0 × 42 cm, 30−50% MeOH) to give **1** (767 mg), **2** (43 mg), and **3** (43 mg) and viscumneoside V3 (248 mg).

Compound 1: colorless amorphous solid; $[\alpha]_D$ −119° (*c* 0.3, MeOH); UV (MeOH) λ_{max} (log ϵ) 284 (4.54), 326sh (3.78); (+$AlCl_3$) λ_{max} (log ϵ) 308 (4.51), 381 (3.70); (+$AlCl_3$ + HCl) λ_{max} (log ϵ) 307 (4.50), 382 (3.71); IR (KBr) ν_{max} 3409 (OH), 3012, 1636 (C=O), 1561, 1519 (aromatic), 1177, 1084, 1022, 820 cm^{-1}; ^1H NMR (DMSO-d_6, 500 MHz) *δ* 2.81 (1H, dd, *J* = 17.0, 2.81 Hz, H*cis*-3), 3.14 (1H, m, H*trans*-3), 3.23−3.37 (4H, m, H-2″, H-4″, H-5‴, H-5⁗), 3.37−3.52 (6H, m, H-2″, H-3″, H-4″, H-6″, H-5‴), 3.62−3.81 (3H, m, H-5″, H-4‴, H-4⁗), 4.67 (1H, br s, H-1⁗), 5.02 (1H, d, *J* = 7.5 Hz, H-1″), 5.23 (1H, br s, H-1‴), 5.56 (1H, dd, *J* = 12.0, 2.8 Hz, H-2), 6.07 (1H, d, *J* = 2.0 Hz, H-6), 6.09 (1H, d, *J* = 2.0 Hz, H-8), 7.35−7.41 (3H, m, H-3′, -4′, -5′), 7.46 (2H, dd, *J* = 8.0, 2.0 Hz, H-2′, -6′), 11.85 (1H, br s, O*H*); ^{13}C NMR (DMSO-d_6, 125 MHz) *δ* 40.1 (t, C-3), 61.0 (t, glc C-6″), 63.8 (t, api C-5⁗), 70.1 (d, glc C-4″), 70.4 (t, api C-5‴), 73.9 (t, api C-4⁗), 74.3 (t, api C-4‴), 76.3 (d, api C-2‴), 76.8 (d, api C-2⁗), 77.1 (d, glc C-3″), 77.2 (d, glc C-2″), 77.3 (d, glc C-5″), 79.3 (s, api C-3‴), 79.3 (s, api C-3⁗), 96.0 (d, C-8), 97.0 (d, glc C-1″), 98.3 (d, C-6), 104.0 (s, C-10), 109.3 (d, api C-1⁗), 109.3 (s, api C-1‴), 127.3 (d, C-2′, −6′), 129.4 (d, C-3′, 5′), 129.5 (d, C-4′), 128.9 (s, C-1′), 163.2 (s, C-9), 163.3 (s, C-5), 165.5 (s, C-7), 197.3 (s, C-4); HMBC correlations H″-1/C-7; H-1‴/C-2″, -2‴, -3‴, -4‴; H-1⁗/C-2⁗, -3⁗, -4⁗, -5⁗; H-6/C-5, -7, -8, -10; H-8/C-6, -7, -9, -10; H-3/C-1′, -2, -4, -10; NOESY correlations H-1″/H-6, H-1″/H-8; FABMS *m/z* 705 $[M + Na]^+$ (12), 683 $[M + H]^+$ (5), 551 $[M + H - 132]^+$(5), 419 $[M + H - 132 - 132]^+$ (8), 257 $[[M + H - 132 - 132 - 162]^+$ (100); HRFABMS *m/z* 683.2224 $[M + H]^+$ (calcd for $C_{31}H_{39}O_{17}$ 683.2187).

Compound 2: colorless amorphous solid; UV (MeOH) λ_{max} (log ϵ) 228 sh (3.74), 279 (4.33); IR (KBr) ν_{max} 3369 (OH), 3020, 1588, 1513 (aromatic), 1158, 1100, 1073, 1050, 785 cm^{-1}; ^1H NMR (DMSO-d_6, 500 MHz) *δ* 1.69 (2H, quin, *J* = 7.0 Hz, H-2), 2.42 (2H, t, *J* = 7.0 Hz, H-3), 2.51 (2H, t, *J* = 7.0 Hz, H-1),

3.17−3.45 (12H, m, glucosyl H-2, H-3, H-4, H-5, and H-6), 3.70, 3.71, 3.72, and 3.94 (3H each, s, OCH_3), 4.82 (2H, d, J = 7.0 Hz, anomeric H), 6.63 (1H, s, H-6′), 6.67 (1H, dd, J = 8.0, 2.0 Hz, H-6″), 6.79 (1H, d, J = 2.0 Hz, H-2″), 6.96 (1H, d, J = 8.0 Hz, H-5″); ^{13}C NMR (DMSO-d_6, 125 MHz) δ 23.0 (t, C-3), 31.4 (t, C-2), 35.0 (t, C-1), 55.9 (q, O*C*H$_3$), 56.0 (q, O*C*H$_3$), 60.9 (q, O*C*H$_3$), 61.0 (t, GLC C-6‴), 61.0 (t, glc C-6⁗), 61.1 (q, O*C*H$_3$), 69.9 (d, glc C-4‴), 70.2 (d, glc C-4⁗), 73.4 (d, glc C-2‴), 73.4 (d, glc C-2⁗), 76.7 (d, glc C-3‴), 76.9 (d, glc C-3⁗), 77.0 (d, glc C-5‴), 77.3 (d, glc C-5⁗), 96.7 (d, C-6′), 100.7 (d, C-1‴), 101.3 (d, C-1⁗), 113.1 (d, C-2″), 115.9 (d, C-5″), 117.2 (s, C-1′), 120.4 (d, C-6″), 120.4 (s, C-1″), 136.4 (s, C-4′), 144.7 (s, C-4″), 149.0 (s, C-3″), 149.4 (s, C-5′), 151.8 (s, C-3′), 153.3 (s, C-2′); HMBC correlations OCH_3 (δ 3.72)/C-3′; OCH_3 (δ 3.70)/C-2′; OCH_3 (δ 3.71)/C-3′; OCH_3 (δ 3.72)/C-4′; OCH_3 (δ 3.94)/C-3‴; H‴-1/C-5′; H-1⁗/C-4″; H-6′/C-1, -2′, -4′, -5′; H-2″/C-1″, -3, -3″, -4″, -6″; H-5″/C-1″, -3″, -4″, -6″; FABMS m/z 695 [M + Na]$^+$ (100), 673 [M + H]$^+$ (5), 511 [M + H − 162]$^+$ (5), 349 [[M + H − 162 − 162]$^+$ (50); HRFABMS m/z 695.2620 [M + Na]$^+$ (calcd for $C_{31}H_{44}O_{16}Na$ 695.2629).

Compound 3: yellowish amorphous solid; UV (MeOH) λ_{max} (log ϵ) 266 (4.36), 346 (4.22); (+AlCl$_3$) λ_{max} (log ϵ) 275 (4.35), 303 (4.12), 355 (4.23), 406 (4.23); (+AlCl$_3$ + HCl) λ_{max} (log ϵ) 276 (4.35), 304 (4.11), 354 (4.22), 405 (4.22); IR (KBr) ν_{max} 3415 (OH), 1655 (C═O), 1599, 1530 (aromatic), 1167, 1073, 1028, 880 cm^{-1}; ^{1}H NMR (DMSO-d_6, 500 MHz) δ 0.89 (3H, d, J = 6.0 Hz, rha H-6), 3.14−3.93 (m, glycosyl H), 3.82 (3H, s, OCH_3), 4.25 (1H, br s, rha H-1), 4.90 (1H, br s, api H-1⁗), 5.30 (1H, br s, api H-1‴), 5.34 (1H, d, J = 7.0 Hz, glc H-1″), 6.29 (1H, d, J = 2.0 Hz, H-6), 6.59 (1H, d, J = 2.0 Hz, H-8), 6.86 (2H, d, J = 8.0 Hz, H-3′, -5′), 7.99 (2H, d, J = 8.0 Hz, H-2′, -6′), 10.13 and 13.54 (1H each, br s, O*H*); ^{13}C NMR (DMSO-d_6, 125 MHz) δ 18.0 (q, rha C-6), 56.6 (q, O*C*H$_3$), 63.8 (t, api C-5⁗), 67.6 (t, glc C-6″), 68.8 (d, rha C-5), 70.4 (d, glc C-4″), 70.4 (d, rha C-2), 70.9 (d, rha C-3), 70.9 (t, api C-5‴), 71.1 (d, rha C-4), 74.4 (t, api C-4‴), 74.4 (t, api C-4⁗), 76.6 (d, api C-2⁗), 76.8 (d, glc C-3″), 77.1 (d, api C-2‴), 77.1 (d, api C-2″), 77.8 (d, glc C-5″), 79.3 (s, api C-3⁗), 79.8 (s, api C-3‴), 92.9 (d, C-8), 98.5 (d, C-6), 99.4 (d, glc C-1″), 101.2 (d, rha H-1), 105.5 (s, C-10), 109.3 (s, api C-1⁗), 110.6 (d, api C-1‴), 115.7 (d, C-3′, -5′), 121.6 (s, C-1′), 131.6 (d, C-2′, -6′), 133.7 (s, C-3), 157.1 (s, C-9), 157.7 (s, C-2), 160.3 (s, C-4′), 161.1 (s, C-5), 165.7 (s, C-7), 178.0 (s, C-4); HMBC correlations OCH_3/C-7; H″-1/C-3; H-1‴/C-2″, -2‴, -3‴, -4‴; H-1⁗/C-2⁗, -3⁗, -4⁗, -5⁗; rhamnosyl H-1/C-6″; H-6/C-5, -7, -8, -10; H-8/C-6, -7, -9, -10; FABMS m/z 895 [M + Na]$^+$ (10), 873 [M + H]$^+$ (10), 763 [M + Na − 132]$^+$ (5), 301 [M + H − 132 − 132 − 146 − 162]$^+$ (55); HRFABMS m/z 873.2650 [M + H]$^+$ (calcd for $C_{38}H_{49}O_{23}$ 873.2664).

Hydrolysis of 1, 2, and 3. Compound **1** (10 mg) was dissolved in 5% HCl/H_2O (2 mL) and refluxed for 1 h, then filtered. The residue was recrystallized from EtOH to yield pale yellow crystals (3 mg) identified as pinocembrin[15] by its ^{1}H NMR spectrum. The filtrate was concentrated under reduced pressure. The sugar components were identified by TLC (on Si gel, developed with *n*-butyl acetate/2-butanone/acetic acid/H_2O = 6.0:2.5:1.2:0.3) as apiose and glucose in comparison with authentic samples. Compounds **2** and **3** were hydrolyzed by the same method. Acidic hydrolysis of **2** gave the colorless amorphous aglycone (2 mg), 2′,3′,4′,3″-tetramethoxy-1,3-diphenylpropane (**4**): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.78 (2H, m, H-2), 2.52−2.58 (4H, m, H-1, H-3), 3.79, 3.81, 3.82, and 3.85 (3H each, s, OCH_3), 6.28 (1H, s, H-6′), 6.91 (1H, d, J = 8.0 Hz, H-5″), 6.92 (1H, d, J = 2.0 Hz, H-2″), 6.79 (1H, dd, J = 8.0, 2.0 Hz, H-6″); EIMS m/z 348 (M$^+$); HREIMS m/z 349.1665 [M + H]$^+$ (calcd for $C_{19}H_{24}O_6$ 349.1658). Hydrolysis of compound **3** yielded rhamnocitrin,[17] and apiose, rhamnose, and glucose were detected in the concentrated filtrate by TLC as above.

Acetylation of 1, 2, and 3. Compounds **1**, **2**, and **3** (10 mg) were acetylated in pyridine (0.5 mL) and Ac_2O (0.5 mL) to yield the corresponding colorless amorphous solids **5**, **6**, and **7**, respectively. ^{1}H NMR of **5** (nonaacetate) ($CDCl_3$) δ 1.96, 2.01, 2.03, 2.05, 2.06, 2.08, 2.10, 2.12, and 2.39, 4.84 (1H, br s), 5.08 (1H, d, J = 7.0 Hz), 5.15 (1H, br s), 6.37 and 6.59 (1H each, d, J = 2.0 Hz), 7.39−7.46 (5H, m); ^{1}H NMR of **6** (octaacetate) ($CDCl_3$) δ 2.04, 2.05 and 2.06 (6H each, s), 2.08 and 2.10 (3H each, s), 3.75, 3.77, 3.82, and 3.83 (3H each, s), 5.27 (1H, d, J = 7.0 Hz), 5.28 (1H, d, J = 6.5 Hz), 6.47 (1H, s), 6.71 (1H, dd, J = 8.0, 2.0 Hz), 6.75 (1H, d, J = 2.0 Hz), 7.02 (1H, d, J = 8.0 Hz); ^{1}H NMR of **7** (decaacetate) ($CDCl_3$) δ 0.95 (3H, d, J = 6.0 Hz), 1.97, 1.99, 2.01, 2.03, 2.05, 2.06, 2.08, 2.10, 2.33, and 2.40 (3H each, s), 3.91 (3H, s), 4.46 (1H, br s), 4.98 (1H, br s), 5.17 (1H, br s), 5.63 (1H, d, J = 7.2 Hz), 6.61 and 6.82 (1H each, d, J = 2.0 Hz), 7.23 and 8.06 (2H each, d, J = 8.5 Hz).

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