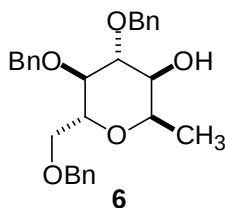


SUPPORTING INFORMATION FOR:
Aluminum and Boron Mediated C-Glycoside Synthesis From 1,2-
Anhydroglycosides

Jon D. Rainier* and Jason M. Cox

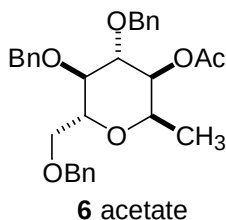
Department of Chemistry, The University of Arizona, Tucson, AZ 85721

Spectroscopic data for compounds **6-11** and **6-11 acetates**.

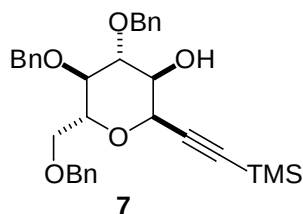


Alcohol **6**. Colorless oil; $[\alpha]_D^{23} = +29.64^\circ$ ($c = 2.35$, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 7.35-7.20 (m, 15H, PhH), 4.71 (d, $J = 11.4$ Hz, 1H, OCH_2Ph), 4.65 (d, $J = 11.2$ Hz, 1H, OCH_2Ph), 4.61 (d, $J = 11.2$ Hz, 1H, OCH_2Ph), 4.58 (d, $J = 12.4$ Hz, 1 H, OCH_2Ph), 4.57 (d, $J = 11.1$ Hz, 1 H, OCH_2Ph), 4.52 (d, $J = 11.8$ Hz, 1H, OCH_2Ph), 4.13-4.07 (m, 1H, OCH), 3.98 (ddd, $J = 9.9, 4.8, 4.8$ Hz, 1H, OCH), 3.80 (ddd, $J = 10.3, 5.7, 1.4$ Hz, 1H, OCH), 3.76-3.72 (m, 1H, OCH), 3.69 (dd, $J = 10.2, 4.6$ Hz, 1H, OCH), 3.61 (m, 2H, CH_2OBn), 2.77 (s, 1H, OH), 1.24 (dd, $J = 6.7, 1.5$ Hz, 3H, CH_3); ^{13}C NMR (125 MHz, CDCl_3) δ 138.2, 138.1, 137.6, 128.6, 128.5, 128.4, 127.9, 127.8, 127.7, 127.6, 78.5, 75.6, 73.7, 73.4, 73.2, 73.1, 70.5, 68.3, 68.2, 14.3; IR (neat) 3472, 1101 cm^{-1} ;

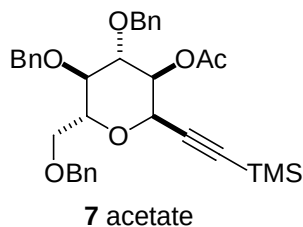
MS (FAB⁺) 449 (MH⁺), 447, 91 m/z; HRMS calcd for C₂₈H₃₃O₅ (MH⁺) 449.2328, found 449.2336.



6 Acetate: Colorless oil; $[\alpha]_D^{24} = +33.89^\circ$ ($c = 1.30$, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 7.34-7.26 (m, 13H, PhH), 7.15-7.13 (m, 2H, PhH), 5.01 (dd, $J = 9.0, 5.6$ Hz, 1H, CHOAc), 4.78 (d, $J = 11.5$ Hz, 1H, OCH₂Ph), 4.75 (d, $J = 10.9$ Hz, 1H, OCH₂Ph), 4.72 (d, $J = 11.7$ Hz, 1H, OCH₂Ph), 4.60 (d, $J = 12.2$ Hz, 1H, OCH₂Ph), 4.52 (d, $J = 12.2$ Hz, 1H, OCH₂Ph), 4.49 (d, $J = 11.1$ Hz, 1H, OCH₂Ph), 4.33 (ddd, $J = 12.6, 6.8, 6.8$ Hz, 1H, OCH), 3.82 (dd, $J = 8.6, 8.6$ Hz, 1H, OCH), 3.74 (m, 1H, OCH), 3.72-3.62 (m, 3H, OCH and CH₂OBn), 1.99 (s, 3H, OCOCH₃), 1.21 (d, $J = 6.9$ Hz, 3H, OCHCH₃); ¹³C NMR (125 MHz, CDCl₃) δ 170.0, 138.4, 138.1, 137.0, 128.4, 128.4, 128.0, 127.9, 127.8, 127.7, 127.6, 79.9, 77.8, 74.8, 73.5, 73.2, 71.8, 69.0, 68.5, 21.0, 12.9; IR (neat) 1750, 1384, 1098 cm⁻¹; MS (FAB⁺) 491 (MH⁺), 489, 91 m/z; HRMS calcd for C₃₀H₃₅O₆ (MH⁺) 491.2434, found 491.2454.

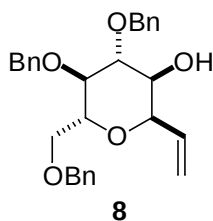


Alcohol **7**: Colorless oil; $[\alpha]_D^{24} = +93.66^\circ$ ($c = 1.20$, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 7.39-7.25 (m, 13H, *PhH*), 7.19-7.17 (m, 2H, *PhH*), 4.90 (d, $J = 11.3$ Hz, 1H, OCH_2Ph), 4.85 (d, $J = 11.3$ Hz, 1H, OCH_2Ph), 4.81 (d, $J = 10.5$ Hz, 1H, OCH_2Ph), 4.80 (d, $J = 4.2$ Hz, 1H, *CHCC*), 4.63 (d, $J = 12.1$ Hz, 1H, OCH_2Ph), 4.54 (d, $J = 12.6$ Hz, 1H, OCH_2Ph), 4.52 (d, $J = 12.4$ Hz, 1H, OCH_2Ph), 3.98 (ddd, $J = 9.6, 3.4, 1.8$ Hz, 1H, *OCH*), 3.78 (dd, $J = 10.9, 3.6$ Hz, 1H, *OCH*), 3.76-3.62 (m, 5H, *OCH* and CH_2OBn), 1.96 (s, 1H, *OH*), 0.19 (s, 9H, $\text{Si}(\text{CH}_3)_3$); ^{13}C NMR (125 MHz, CDCl_3) δ 138.5, 138.1, 137.9, 128.5, 128.5, 128.4, 128.1, 128.0, 128.0, 127.9, 127.7, 99.5, 95.9, 83.5, 77.6, 75.1, 73.9, 73.5, 71.2, 68.9, 68.5, -0.1; IR (neat) 3472, 2165, 1075 cm^{-1} ; MS (FAB^+) 531 (MH^+), 529, 91 m/z ; HRMS calcd for $\text{C}_{32}\text{H}_{39}\text{O}_5\text{Si}$ (MH^+) 531.2567, found 531.2562.



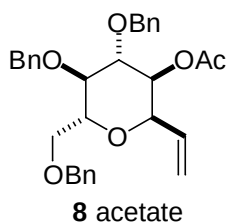
7 Acetate: Colorless oil; $[\alpha]_D^{24} = +86.48^\circ$ ($c = 0.85$, CHCl_3); ^1H NMR (500 MHz, C_6D_6) δ 7.34 (d, $J = 8.0$ Hz, 2H, *PhH*), 7.29-7.27 (m, 1H, *PhH*), 7.22-7.18 (m, 10H, *PhH*), 7.16-

7.12 (m, 2H, PhH), 5.34 (d, $J = 5.9$ Hz, 1H, OCH), 5.25 (dd, $J = 9.8, 5.9$ Hz, 1H, CHOAc), 4.89 (d, $J = 11.1$ Hz, 1H, OCH₂Ph), 4.87 (d, $J = 11.4$ Hz, 1H, OCH₂Ph), 4.84 (d, $J = 11.4$ Hz, 1H, OCH₂Ph), 4.68 (d, $J = 11.2$ Hz, 1H, OCH₂Ph), 4.51 (d, $J = 12.2$ Hz, 1H OCH₂Ph), 4.42 (d, $J = 12.1$ Hz, 1H, OCH₂Ph), 4.40 (ddd, $J = 9.8, 3.8, 1.7$ Hz, 1H, OCH), 4.31 (dd, $J = 9.4, 9.4$ Hz, 1H, CH₂OBn), 3.90 (dd, $J = 9.4, 9.2$ Hz, 1H, CH₂OBn), 3.84 (dd, $J = 11.1, 3.9$ Hz, 1H, OCH), 3.73 (dd, $J = 11.1, 1.8$ Hz, 1H, OCH), 1.68 (s, 3H, OCOCH₃), 0.19 (s, 9H, Si(CH₃)₃); ¹³C NMR (125 MHz, CDCl₃) δ 170.1, 138.5, 138.0, 137.9, 128.4, 128.4, 128.2, 127.9, 127.9, 127.8, 127.7, 99.2, 95.1, 80.9, 77.6, 75.3, 73.7, 73.5, 72.2, 68.4, 65.9, 20.9, -0.1; IR (neat) 2191, 1758, 1083 cm⁻¹; MS (FAB⁺) 573 (MH⁺), 573, 91 m/z; HRMS calcd for C₃₄H₄₁O₆Si (MH⁺) 573.2672, found 573.2694.



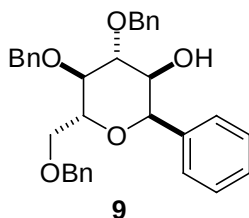
Alcohol **8**: Colorless oil; $[\alpha]_D^{24} = +73.11^\circ$ ($c = 0.80$, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 7.34-7.25 (m, 13H, PhH), 7.20-7.19 (m, 2H, PhH), 6.03 (ddd, $J = 17.4, 10.8, 5.5$ Hz, 1H, CHCH₂), 5.39 (dd, $J = 17.5, 1.5$ Hz, 1H, CHCH₂), 5.35 (dd, $J = 10.8, 1.4$ Hz, 1H, CHCH₂), 4.76 (d, $J = 11.6$ Hz, 1H, OCH₂Ph), 4.68 (d, $J = 11.2$ Hz, 1H, OCH₂Ph), 4.64 (d, $J = 11.6$ Hz, 1H, OCH₂Ph), 4.59 (d, $J = 12.1$ Hz, 1H, OCH₂Ph), 4.54 (d, $J = 11.2$ Hz, 1H, OCH₂Ph), 4.51 (d, $J = 12.1$ Hz, 1H, OCH₂Ph), 4.46 (m, 1H, OCH),

4.01 (ddd, $J = 9.9, 5.0, 5.0$ Hz, 1H, OCH), 3.76 (dd, $J = 10.4, 5.0$ Hz, 2 H, OCH or CH_2OBn), 3.69 (dd, $J = 10.4, 4.1$ Hz, 1H, OCH or CH_2OBn), 3.66 (m, 2 H, OCH or CH_2OBn), 2.65 (d, $J = 5.1$ Hz, 1H, OH); ^{13}C NMR (125 MHz, CDCl_3) δ 138.1, 138.1, 137.6, 133.0, 128.6, 128.5, 128.4, 127.9, 127.8, 127.7, 118.9, 79.9, 76.3, 74.1, 73.7, 73.6, 73.4, 73.3, 70.6, 68.3; IR (neat) 3446, 1109 cm^{-1} ; MS (FAB $^+$) 461 (MH^+), 91 m/z ; HRMS calcd for $\text{C}_{29}\text{H}_{33}\text{O}_5$ (MH^+) 461.2328, found 461.2334.

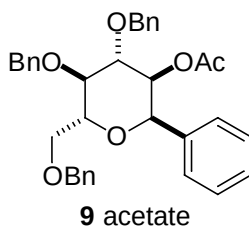


8 Acetate: Colorless oil; $[\alpha]_{\text{D}}^{24} = +87.85^\circ$ ($c = .30$, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 7.31-7.25 (m, 13H, PhH), 7.13-7.11 (m, 2H, PhH), 5.95 (ddd, $J = 17.1, 10.9, 5.9$ Hz, 1H, CHCH_2), 5.40 (d, $J = 17.6$ Hz, 1H, CHCH_2), 5.37 (d, $J = 11.2$ Hz, 1H, CHCH_2), 5.05 (dd, $J = 9.4, 6.0$ Hz, 1H, CHOAc), 4.77 (d, $J = 11.9$ Hz, 1H, OCH_2Ph), 4.75 (d, $J = 11.7$ Hz, 1H, OCH_2Ph), 4.72 (d, $J = 11.5$ Hz, 1H, OCH_2Ph), 4.66 (dd, $J = 5.9, 5.9$ Hz, 1H, OCH), 4.61 (d, $J = 12.2$ Hz, 1H, OCH_2Ph), 4.50 (d, $J = 11.8$ Hz, 1H, OCH_2Ph), 4.48 (d, $J = 10.5$ Hz, 1H, OCH_2Ph), 3.80 (m, 1 H, OCH or CH_2OBn), 3.77 (dd, $J = 9.0, 9.0$ Hz, 1 H, OCH or CH_2OBn), 3.69 (partially obscured dd, $J = 10.7, 4.8$ Hz, 1 H, OCH or CH_2OBn), 3.66 (m, 2H, OCH or CH_2OBn), 1.95 (s, 3H, OCOCH_3); ^{13}C NMR (125 MHz, CDCl_3) δ 169.9, 138.4, 138.0, 138.0, 131.3, 128.4, 128.4, 128.0, 127.9, 127.8, 127.7, 127.6, 120.5, 80.6, 78.0, 75.0, 74.9, 73.5, 73.2, 73.0, 72.5, 68.9, 20.9; IR (neat)

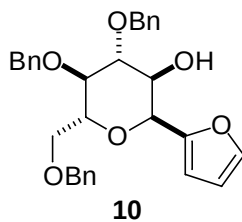
1750, 1109 cm^{-1} ; MS (FAB⁺) 503 (MH⁺), 501, 91 m/z; HRMS calcd for C₃₁H₃₅O₆ (MH⁺) 503.2434, found 503.2413.



Alcohol **9**: Colorless oil; $[\alpha]_{\text{D}}^{24} = -7.49^\circ$ ($c = 1.20$, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 7.43 (d, $J = 7.6$ Hz, 2H, PhH), 7.36-7.23 (m, 18H, PhH), 4.95 (bs, 1H, OCH), 4.62 (d, $J = 11.7$ Hz, 1 H, OCH_2Ph), 4.61 (s, 2H, OCH_2Ph), 4.58 (d, $J = 11.9$ Hz, 1 H, OCH_2Ph), 4.55 (d, $J = 12.2$ Hz, 1H, OCH_2Ph), 4.51 (d, $J = 12.2$ Hz, 1H, OCH_2Ph), 4.34 (m, 1H, OCH), 3.95 (dd $J = 3.4, 3.4$ Hz, 1H, OCH or CH_2OBn), 3.89 (dd $J = 10.0, 6.5$ Hz, 1H, OCH or CH_2OBn), 3.86-3.84 (m, 1H, OCH), 3.81 (dd $J = 10.0, 6.4$ Hz, 1H, OCH or CH_2OBn), 3.73-3.72 (m, 1H, OCH or CH_2OBn), 3.28 (d, $J = 10.0$ Hz, 1H, OH); ^{13}C NMR (125 MHz, CDCl_3) δ 139.0, 138.2, 137.8, 137.3, 128.6, 128.6, 128.4, 128.1, 128.0, 128.0, 127.7, 127.3, 126.8, 75.9, 74.8, 73.4, 73.2, 72.7, 72.2, 71.3, 70.3, 67.7; IR (neat) 3532, 1083 cm^{-1} ; MS (FAB⁺) 511 (MH⁺), 509, 91 m/z; HRMS calcd for C₃₃H₃₅O₇ (MH⁺) 511.2484, found 511.2495.

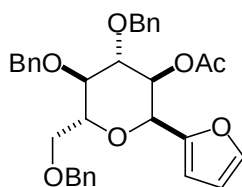


9 Acetate: Colorless oil; $[\alpha]_D^{29} = +54.04^\circ$ ($c = 1.15$, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 7.52 (d, $J = 7.6$ Hz, 2H, PhH), 7.41-7.28 (m, 16H, PhH), 7.22-7.21 (m, 2H PhH), 5.34 (dd, $J = 6.4, 4.2$ Hz, 1H, CHOAc), 5.29 (d, $J = 4.1$ Hz, 1H, OCH), 4.85 (d, $J = 11.7$ Hz, 1H, OCH₂Ph), 4.80 (d, $J = 11.7$ Hz, 1H, OCH₂Ph), 4.74 (d, $J = 11.1$ Hz, 1H, OCH₂Ph), 4.66 (d, $J = 12.1$ Hz, 1H, OCH₂Ph), 4.56 (d, $J = 11.1$ Hz, 1H, OCH₂Ph), 4.55 (d, $J = 12.1$ Hz, 1H, OCH₂Ph), 4.09 (m, 1H, OCH), 3.92-3.73 (m, 4H, OCH or CH₂OBn), 1.93 (s, 3H, OCOCH₃); ^{13}C NMR (125 MHz, CDCl_3) δ 170.0, 138.2, 138.1, 138.0, 137.5, 128.5, 128.4, 128.3, 128.2, 127.9, 127.9, 127.8, 127.7, 127.7, 127.6, 127.4, 79.0, 76.6, 73.8, 73.6, 73.5, 73.4, 72.9, 72.4, 68.9, 20.9; IR (neat) 1752, 1065 cm^{-1} ; MS (FAB⁺) 553 (MH⁺), 551, 91 m/z ; HRMS calcd for $\text{C}_{35}\text{H}_{37}\text{O}_6$ (MH⁺) 553.2590, found 553.2597.



Alcohol **10**: Colorless oil: $[\alpha]_D^{23} = +63.57^\circ$ ($c = 1.70$, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 7.40 (d, $J = 1.0$ Hz, 1H, OCHCHCHC) 7.36-7.24 (m, 13H, PhH), 7.20-7.18 (m, 2H, PhH), 6.46 (d, $J = 3.2$ Hz, 1H, OCHCHCHC), 6.35 (dd, $J = 3.2, 1.8$ Hz, 1H, OCHCHCHC), 5.09 (d, $J = 5.0$ Hz, 1H, OCH), 4.86 (d, $J = 11.5$ Hz, 1H, OCH₂Ph), 4.77 (d, $J = 11.4$ Hz, 1H, OCH₂Ph), 4.75 (d, $J = 10.9$ Hz, 1H, OCH₂Ph), 4.61 (d, $J = 12.1$ Hz, 1H, OCH₂Ph), 4.57 (d, $J = 10.9$ Hz, 1H, OCH₂Ph), 4.50 (d, $J = 12.1$ Hz, 1H, OCH₂Ph), 4.05 (dd, $J = 15.3, 8.1$ Hz, 1H, OCH), 4.04-4.01 (m, 1H, OCH), 3.84 (ddd, $J = 7.6, 3.8,$

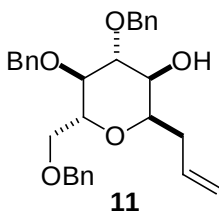
3.8 Hz, 1H, *OCH*), 3.78 (dd, $J = 10.6, 4.2$ Hz, 1H, *OCH* or CH_2OBn), 3.74 (dd $J = 7.5, 7.5$ Hz, 1H, *OCH* or CH_2OBn), 3.67 (dd, $J = 10.6, 3.2$ Hz, 1H, *OCH* or CH_2OBn), 2.51 (d, $J = 4.6$ Hz, 1H, *OH*); ^{13}C NMR (125 MHz, CDCl_3) δ 151.2, 142.6, 138.3, 138.0, 137.8, 128.6, 128.5, 128.4, 128.0, 127.9, 127.9, 127.7, 11.0, 110.3, 81.1, 77.3, 74.6, 74.2, 74.0, 73.5, 70.7, 70.2, 68.4; IR (neat) 3480, 1092 cm^{-1} ; MS (FAB^+) 501 (MH^+), 499, 91 m/z ; HRMS calcd for $\text{C}_{31}\text{H}_{33}\text{O}_6$ (MH^+) 501.2277, found 501.2271.



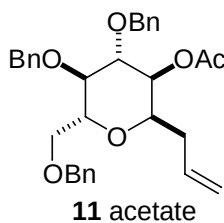
10 acetate

10 Acetate: $[\alpha]_D^{29} = +83.20^\circ$ ($c = 0.50$, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 7.41-7.40 (m, 1H, *OCHCHCHC*) 7.34-7.23 (m, 13H, *PhH*), 7.15-7.13 (m, 2H, *PhH*), 6.36 (d, $J = 3.2$ Hz, 1H, *OCHCHCHC*), 6.32 (dd, $J = 2.4, 2.0$ Hz, 1H, *OCHCHCHC*), 5.30 (d, $J = 6.6$ Hz, 1H, *OCHfuryl*), 5.17 (dd, $J = 9.8, 6.6$ Hz, 1H, *CHOAc*), 4.85 (d, $J = 11.4$ Hz, 1H, OCH_2Ph), 4.80 (d, $J = 10.6$ Hz, 1H, OCH_2Ph), 4.79 (d, $J = 11.4$ Hz, 1H, OCH_2Ph), 4.60 (d, $J = 12.1$ Hz, 1H, OCH_2Ph), 4.54 (d, $J = 10.7$ Hz, 1H, OCH_2Ph), 4.48 (d, $J = 12.1$ Hz, 1H, OCH_2Ph), 4.29 (dd, $J = 9.3, 9.3$ Hz, 1H, *OCH* or CH_2OBn), 3.79 (dd, $J = 9.1, 9.1$ Hz, 1H, *OCH* or CH_2OBn), 3.77-3.67 (m, 2H, *OCH* or CH_2OBn), 3.61 (d, $J = 9.6$ Hz, 1H, *OCH*), 1.84 (s, 3H, OCOCH_3); ^{13}C NMR (125 MHz, CDCl_3) δ 170.0, 150.4, 143.0, 138.5, 138.0, 138.0, 128.4, 128.3, 128.0, 127.9, 127.8, 127.7, 127.6, 111.6, 110.0, 80.9, 78.0, 75.2, 75.1, 73.6, 73.5, 72.8, 68.8, 68.6, 20.8; IR (neat) 1744, 1106 cm^{-1} ;

MS (FAB⁺) 543 (MH⁺), 541, 91 m/z; HRMS calcd for C₃₃H₃₅O₇ (MH⁺) 543.2383, found 543.2377.



Alcohol **11**: White solid; mp 74-75 °C; $[\alpha]_D^{29} = +26.60^\circ$ (c = 0.55, CHCl₃); ¹H NMR (500 Mhz, CDCl₃) δ 7.37-7.26 (m, 15H, PhH), 5.86 (m, 1H, CH₂CHCH₂), 5.17 (dd, *J* = 17.1, 1.0 Hz, 1H, CH₂CHCH₂), 5.09 (dd, *J* = 10.2, 0.9 Hz, 1H, CH₂CHCH₂), 4.68 (d, *J* = 11.7 Hz, 1H, OCH₂Ph), 4.64 (d, *J* = 11.5 Hz, 1H, OCH₂Ph), 4.60 (d, *J* = 11.6 Hz, 1H, OCH₂Ph), 4.60 (d, *J* = 11.9 Hz, 2 H, OCH₂Ph), 4.53 (d, *J* = 12.1 Hz, 1H, OCH₂Ph), 4.09 (dd, *J* = 10.5, 5.2 Hz, 1H, OCH), 3.96 (ddd, *J* = 8.6, 5.5, 3.0 Hz, 1H, OCH), 3.85 (dd *J* = 10.2, 5.8 Hz, 1H, OCH or CH₂OBn), 3.79 (dd *J* = 5.3, 5.3 Hz, 1H, OCH or CH₂OBn), 3.73 (dd *J* = 10.2, 5.2 Hz, 1H, OCH or CH₂OBn), 3.70-3.68 (m, 1H, OCH or CH₂OBn), 3.67 (dd, *J* = 4.6, 4.6 Hz, 1H, OCH or CH₂OBn), 2.94 (d, *J* = 8.3 Hz, 1H, OH), 2.43 (m, 2H, OCHCH₂CHCH₂); ¹³C NMR (125 MHz, CDCl₃) δ 138.2, 137.9, 137.4, 134.7, 128.5, 128.5, 128.3, 127.9, 127.7, 127.6, 127.6, 117.0, 77.4, 74.7, 73.5, 73.2, 72.8, 71.0, 69.0, 68.0, 33.2; IR (neat) 3493, 1082 cm⁻¹; MS (FAB⁺) 475 (MH⁺), 473, 91 m/z; HRMS calcd for C₃₀H₃₅O₅ (MH⁺) 475.2484, found 475.2494.



11 Acetate: White solid; mp 72-73 °C; $[\alpha]_D^{29} = +56.66^\circ$ ($c = 1.00$, CHCl_3); ^1H NMR (500 MHz, C_6D_6) δ 7.41 (d, $J = 7.2$ Hz, 3H, PhH), 7.36-7.34 (m, 2H, PhH), 7.29-7.26 (m, 7H, PhH), 7.22-7.18 (m, 3H, PhH), 5.88 (m, 1H, CH_2CHCH_2), 5.42 (dd, $J = 8.1, 5.1$ Hz, 1H, CHOAc), 5.17-5.12 (m, 2H, CH_2CHCH_2), 4.84 (d, $J = 11.8$ Hz, 1H, OCH_2Ph), 4.83 (d, $J = 11.5$ Hz, 1H, OCH_2Ph), 4.80 (d, $J = 11.9$ Hz, 1H, OCH_2Ph), 4.64 (d, $J = 11.4$ Hz, 1H, OCH_2Ph), 4.58 (d, $J = 12.2$ Hz, 1H, OCH_2Ph), 4.51 (d, $J = 12.2$ Hz, 1H, OCH_2Ph), 4.42 (ddd, $J = 10.0, 5.0, 5.0$ Hz, 1H, OCHCH_2CH), 3.97 (dd, $J = 8.0, 8.0$ Hz, 1H, OCH or CH_2OBn), 3.95-3.92 (m, 1H, OCH or CH_2OBn), 3.86 (dd, $J = 8.7, 7.8$ Hz, 1H, OCH or CH_2OBn), 3.82 (dd, $J = 10.7, 4.3$ Hz, 1H, OCH or CH_2OBn), 3.78 (dd, $J = 10.7, 2.8$ Hz, 1H, OCH or CH_2OBn), 2.58-2.51 (m, 1H, $\text{OCHCH}_2\text{CHCH}_2$), 2.32-2.27 (m, 1H, $\text{OCHCH}_2\text{CHCH}_2$), 1.69 (s, 3H, OCOCH_3); ^{13}C NMR (125 MHz, C_6D_6) δ 169.1, 138.9, 134.4, 128.3, 128.3, 128.2, 128.0, 127.7, 127.5, 127.4, 116.8, 80.1, 77.7, 74.2, 74.1, 73.3, 72.7, 72.6, 71.6, 69.4, 31.7, 20.2; IR (neat) 1752, 1106 cm^{-1} ; MS (FAB^+) 517 (MH^+), 515, 91 m/z ; HRMS calcd for $\text{C}_{32}\text{H}_{37}\text{O}_6$ (MH^+) 517.2590, found 517.2587.