

An Efficient Synthesis of Triazolo-carbohydrate Mimetics and Their Conformation Analysis

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1. Preparation of triazolo-mannose mimic **6b**

(2S,3S,4R)-2,3,4-Tri(benzyloxy)-1,1-dimethoxyhex-5-ene (2b). According to the synthetic procedure for **2a**, 1,1-dimethoxyhex-5-ene derivative **2b** was prepared in 100% yield (6.29 g, 13.6 mmol) by the reaction of (2S,3S,4R)-2,3,4-tribenzyloxyhex-5-enal **1b** (5.7 g, 13.6 mmol),¹ *p*TsOH·H₂O (30 mg) and HC(OMe)₃ (35 mL). Colorless oil. $[\alpha]_D^{25}$ (*c* 1.00, CHCl₃) -23.7; IR (neat) ν 3030, 2930, 1454, 1093, 735, 697 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.37 (3H, s), 3.40 (3H, s), 3.67-3.71 (1H, m), 3.72-3.75 (1H, m), 4.15 (1H, dd, *J* = 7.5, 5.6 Hz), 4.33 (1H, d, *J* = 11.8 Hz), 4.47 (1H, d, *J* = 11.5 Hz), 4.55 (1H, d, *J* = 4.7 Hz), 4.59 (1H, d, *J* = 11.8 Hz), 4.69 (1H, d, *J* = 11.5 Hz), 4.73 (1H, d, *J* = 11.5 Hz), 4.75 (1H, d, *J* = 11.5 Hz), 5.25 (1H, dd, *J* = 10.3, 1.5 Hz), 5.33 (1H, dd, *J* = 17.3, 1.5 Hz), 5.83 (1H, ddd, *J* = 17.3, 10.3, 7.5 Hz), 7.20-7.35 (15H, m); ¹³C NMR (100 MHz, CDCl₃) δ 55.2, 56.2, 70.6, 73.7, 74.7, 78.9, 81.0, 82.6, 105.4, 118.6, 127.4, 127.8, 127.8, 127.9, 128.1, 128.2, 128.2, 136.0, 138.6, 138.7, 138.9. MS (ESI-TOF) *m/z* 485 [M+Na]⁺. HRMS calcd for C₂₉H₃₄NaO₅ [M+Na]⁺, 485.2304; found, 485.2324. Anal. Calcd for C₂₉H₃₄O₅: C, 75.30; H, 7.41. Found: C, 75.37; H, 7.47.

(2S,3R,4S)-Tri(benzyloxy)-5,5-dimethoxypentanal (3b). According to the synthetic procedure for **3a**, 5,5-dimethoxypentanal derivative **3b** was prepared in 86% yield (4.39 g, 9.46 mmol) by the reaction of **2b** (5.1 g, 11 mmol), OsO₄ (0.11 M in H₂O, 4.5 mL, 0.5 mmol), NMO (3.8 g, 33 mmol) and NaIO₄ (5 g) in a mixture of acetone (200 mL), H₂O (50 mL) and *tert*-butyl alcohol (6.5 mL). Colorless oil. $[\alpha]_D^{25}$ (*c* 1.00, CHCl₃) -31.3. IR (neat) ν 3030, 2930, 1730, 1454, 1093, 735, 697 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 3.37 (3H, s), 3.44 (3H, s), 3.77-3.82 (1H, m), 4.03-4.05 (1H, m), 4.09-4.13 (1H, m), 4.53 (1H, d, *J* = 11.9 Hz), 4.55-4.63 (4H, m), 4.69 (1H, d, *J* = 11.8 Hz), 4.81 (1H, d, *J* = 11.5 Hz), 7.25-7.36 (15H, m), 9.69 (1H, brs); ¹³C NMR (100 MHz, CDCl₃) δ 55.7, 55.8, 73.1, 73.2, 74.3, 79.1, 79.5, 83.0, 105.0, 127.5, 127.8, 127.8, 127.9, 128.0, 128.1, 128.2, 128.3, 128.4, 137.3, 137.7, 138.2, 201.1; MS (ESI-TOF) *m/z* 487 [M+Na]⁺; HRMS calcd for C₂₈H₃₂NaO₆ [M+Na]⁺, 487.2097; found, 487.2082.

(2S,3S,4R)-2,3,4-Tri(benzyloxy)-1,1-dimethoxyhex-5-yne (4b). According to the synthetic procedure for **4a**, 1,1-dimethoxyhex-5-yne derivative **4b** was prepared in 52% yield (1.23 g, 2.72 mmol) by the reaction of **3b** (2.3 g, 13.5 mmol), Ohira-Bestmann reagent (2.88 g, 15 mmol) and K₂CO₃ (2.3 g, 13.5 mmol) in MeOH (400 mL). Colorless oil. $[\alpha]_D^{25}$ (*c* 1.00, CHCl₃) -50.3; IR (neat) ν 3030, 2931, 1454, 1070, 737, 697 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 2.55 (1H, d, *J* = 2.1 Hz), 3.39 (3H, s), 3.42 (3H, s), 3.87-3.89 (1H, m), 3.94 (1H, dd, *J* = 5.6, 4.9 Hz), 4.49-4.58 (3H, m), 4.63 (1H, d, *J* = 4.9 Hz), 4.76 (1H, d, *J* = 11.4 Hz), 4.79 (1H, d, *J* = 11.1 Hz), 4.87 (1H, d, *J* = 11.7 Hz), 4.98 (1H, d, *J* = 11.4 Hz), 7.25-7.42 (15H, m); ¹³C NMR (100 MHz, CDCl₃) δ 55.1, 56.1, 69.4, 70.9, 73.6, 74.8, 77.3, 78.7, 81.1, 81.6, 105.0, 127.4, 127.6, 127.9, 127.9, 128.1, 128.2, 128.3, 137.7, 138.4, 138.6; MS (ESI-TOF) *m/z* 483 [M+Na]⁺; HRMS calcd for C₂₉H₃₂NaO₅ [M+Na]⁺, 483.2147; found, 483.2135.

(4R,5S,6S,7R)-4,5,6-Tri(benzyloxy)-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (5b- α) and (4R,5S,6S,7S)-4,5,6-tri(benzyloxy)-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (5b- β).

According to the synthetic procedure for **5a**, these compounds were prepared in 91% yield (**5b- α** 88.0 mg, 0.187 mmol, 43% yield; **5b- β** 98.2 mg, 0.208 mmol, 48% yield) by the reaction of **4b** (200 mg, 0.43 mmol) and TMSN₃ (260 μ L, 2.2 mmol) in the presence of In(OTf)₃ (12.5 mg, 22 μ mol) in 1,2-dichloroethane (6.0 mL). **5b- α** as a less polar isomer. Colorless oil. $[\alpha]_D^{25}$ (*c* 1.05, CHCl₃) -22.3; IR (neat, ν) 3031, 2933, 1496, 1454, 1198, 1093, 740, 698 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 3.53 (3H, s), 4.22 (1H, dd, *J* = 3.4, 2.1 Hz), 4.26 (1H, dd, *J* = 7.7, 2.1 Hz), 4.64-4.75 (4H, m), 4.77 (1H, d, *J* = 11.5 Hz), 4.81 (1H, d, *J* = 11.5 Hz), 4.95 (1H, d, *J* = 7.7 Hz), 5.57 (1H, d, *J* = 3.4 Hz), 7.19-7.34 (15H, m), 7.53 (1H, s); ¹³C NMR (100 MHz, CDCl₃) δ 58.3, 71.4, 72.8, 73.2, 73.4, 76.0,

78.1, 86.4, 127.7, 127.8, 127.87, 127.94, 128.01, 128.03, 128.4, 128.47, 128.51, 132.1, 134.3, 137.2, 137.5, 137.7; MS (ESI-TOF) m/z 472 [M+H]⁺; HRMS calcd for C₂₈H₃₀N₃O₄ [M+Na]⁺, 472.2236; found, 472.2264. **5b-β** as a more polar isomer. Colorless oil. $[\alpha]_D^{25}$ (*c* 1.03, CHCl₃) -73.7; IR (neat) ν 3031, 2915, 1496, 1454, 1195, 1101, 740, 698 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.58 (3H, s), 4.23 (1H, dd, *J* = 3.4, 2.1 Hz), 4.28 (1H, dd, *J* = 7.5, 2.1 Hz), 4.64-4.75 (4H, m), 4.78 (1H, d, *J* = 12.0 Hz), 4.79 (1H, d, *J* = 12.0 Hz), 4.96 (1H, d, *J* = 7.5 Hz), 5.58 (1H, d, *J* = 3.4 Hz), 7.26-7.41 (15H, m), 7.57 (1H, s); ¹³C NMR (100 MHz, CDCl₃) δ 58.4, 71.5, 72.9, 73.3, 73.4, 76.1, 78.1, 86.5, 127.8, 127.8, 127.9, 128.0, 128.1, 128.5, 128.5, 128.6, 132.2, 134.3, 137.2, 137.6, 137.7; MS (ESI-TOF) m/z 472 [M+H]⁺; HRMS calcd for C₂₈H₃₀N₃O₄ [M+H]⁺, 472.2236; found, 472.2277.

(4R,5S,6S,7R)-4,5,6-Trihydroxy-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (6b-α). According to the synthetic procedure for **6a-α**, triol **6b-α** was obtained in 88% yield (76.1 mg, 0.379 mmol) by the debenzoylation reaction of **5b-α** (200 mg, 0.43 mmol) in the presence of Pd(OH)₂ on carbon (20 w/w%, 238 mg) in MeOH (5.0 mL) under H₂ atmosphere (1 atm). Colorless oil. $[\alpha]_D^{25}$ (*c* 1.02, MeOH) +28.9; IR (neat) ν 3335, 2928, 1457, 1273, 1126, 1071 cm⁻¹; ¹H NMR (400 MHz, D₂O) δ 3.64 (3H, s), 4.25 (1H, dd, *J* = 8.0, 2.2 Hz), 4.50 (1H, dd, *J* = 3.2, 2.2 Hz), 4.99 (1H, d, *J* = 8.0 Hz), 5.77 (1H, d, *J* = 3.2 Hz), 7.92 (1H, s); ¹³C NMR (100 MHz, CDCl₃) δ 60.1, 66.5, 72.5, 73.1, 90.6, 134.5, 138.7; MS (ESI-TOF) m/z 202 [M+H]⁺; HRMS calcd for C₂₈H₃₀N₃O₄ [M+H]⁺, 202.0828; found, 202.0818.

(4R,5S,6S,7S)-4,5,6-Trihydroxy-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (6b-β). According to the synthetic procedure for **6a-α**, triol **6b-β** was obtained in 79% yield (60.5 mg, 0.305 mmol) by the debenzoylation reaction of **5b-β** (180 mg, 0.39 mmol) in the presence of Pd(OH)₂ on carbon (20 w/w%, 218 mg) in MeOH (5.0 mL) under H₂ atmosphere (1 atm). Colorless oil. $[\alpha]_D^{25}$ (*c* 1.01, MeOH) -86.6; IR (neat) ν 3348, 2927, 1455, 1226, 1114, 1084 cm⁻¹; ¹H NMR (400 MHz, D₂O) δ 3.88 (3H, s), 4.04 (1H, dd, *J* = 7.4, 2.3 Hz), 4.59 (1H, dd, *J* = 3.3, 2.3 Hz), 5.05 (1H, d, *J* = 7.4 Hz), 5.80 (1H, d, *J* = 3.3 Hz), 7.89 (1H, s); ¹³C NMR (100 MHz, D₂O) δ 61.5, 66.1, 70.9, 74.2, 89.9, 133.9, 138.0; MS (ESI-TOF) m/z 202 [M+H]⁺; HRMS calcd for C₇H₁₂N₃O₄ [M+H]⁺, 202.0828; found, 202.0813.

2. Preparation of triazolo-galactose mimic 6c

(2R,3S,4S)-2,3,4-Tri(benzyloxy)-1,1-dimethoxyhex-5-ene (2c). According to the synthetic procedure for **2a**, 1,1-dimethoxyhex-5-ene derivative **2c** was prepared in 91% (1.26 g, 2.72 mmol) by the reaction of (2R,3S,4S)-2,3,4-tribenzyloxyhex-5-enal **1c** (1.3 g, 3.0 mmol),² *p*TsOH-H₂O (43 mg) and HC(OMe)₃ (7 mL). Colorless oil. $[\alpha]_D^{25}$ (*c* 1.00, CHCl₃) +18.1; IR (neat) ν 3030, 2928, 1454, 1094, 734, 697 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.27 (3H, s), 3.43 (3H, s), 3.73 (1H, dd, *J* = 7.7, 2.6 Hz), 3.80 (1H, dd, *J* = 7.1, 2.6 Hz), 4.09 (1H, t, *J* = 7.7 Hz), 4.12 (1H, d, *J* = 11.5 Hz), 4.47-4.56 (4H, m), 4.69 (1H, d, *J* = 11.3 Hz), 4.78 (1H, d, *J* = 11.5 Hz), 5.34 (1H, brd, *J* = 10.2 Hz), 5.41 (1H, brd, *J* = 17.5 Hz), 5.90 (1H, ddd, *J* = 17.5, 10.2, 7.7 Hz), 7.17-7.34 (15H, m); ¹³C NMR (100 MHz, CDCl₃) δ 53.9, 56.2, 69.9, 74.3, 74.4, 78.7, 79.8, 81.0, 105.6, 119.3, 127.3, 127.4, 127.5, 127.9, 128.0, 128.1, 128.2, 136.5, 138.5, 138.6, 138.9; MS (ESI-TOF) m/z 485 [M+Na]⁺; HRMS calcd for C₂₉H₃₄NaO₅ [M+Na]⁺, 485.2304; found, 485.2286.

(2R,3S,4R)-Tri(benzyloxy)-5,5-dimethoxypentanal (3c). According to the synthetic procedure for **3a**, 5,5-dimethoxypentanal derivative **3c** was prepared in 89% yield (1.03 g, 2.22 mmol) by the reaction of **2c** (1.2 g, 2.5 mmol), OsO₄ (0.11 M in H₂O, 1.2 mL,

0.13 mmol), NMO (0.5 g, 4.5 mmol) and NaIO₄ (3 g) in a mixture of acetone (40 mL), H₂O (10 mL) and *tert*-butyl alcohol (3 mL). Colorless oil. $[\alpha]_{\text{D}}^{25}$ (*c* 1.00, CHCl₃) +9.10; IR (neat) ν 3031, 2932, 1733, 1454, 1092, 737, 698 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 3.28 (3H, s), 3.42 (3H, s), 3.66 (1H, dd, *J* = 6.3, 3.3 Hz), 3.98-4.02 (2H, m), 4.27 (1H, d, *J* = 11.7 Hz), 4.45 (1H, d, *J* = 6.3 Hz), 4.51 (1H, d, *J* = 11.4 Hz), 4.55 (1H, d, *J* = 11.3 Hz), 4.57 (1H, d, *J* = 11.7 Hz), 4.63 (1H, d, *J* = 11.3 Hz), 4.77 (1H, d, *J* = 11.4 Hz), 7.19-7.30 (15H, m), 9.64 (1H, d, *J* = 1.4 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 54.7, 56.3, 72.3, 74.1, 74.3, 78.8, 79.6, 83.6, 105.5, 127.7, 127.7, 127.8, 128.2, 128.2, 128.3, 137.3, 137.8, 138.1, 201.2; MS (ESI-TOF) *m/z* 487 [M+Na]⁺; HRMS calcd for C₂₈H₃₂NaO₆ [M+Na]⁺, 487.2097; found: 487.2077.

(2*R*,3*S*,4*S*)-2,3,4-Tri(benzyloxy)-1,1-dimethoxyhex-5-yne (4c). According to the synthetic procedure for **4a**, 1,1-dimethoxyhex-5-yne derivative **4c** was prepared in 68% yield (0.69 g, 1.50 mmol) by the reaction of **3c** (1.0 g, 2.2 mmol), Ohira-Bestmann reagent (3.6 g, 19 mmol) and K₂CO₃ (3.0 g, 22 mmol) in MeOH (230 mL). Colorless oil. $[\alpha]_{\text{D}}^{25}$ (*c* 1.00, CHCl₃) +52.5; IR (neat) ν 3031, 2932, 1454, 1095, 736, 697 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 2.52 (1H, d, *J* = 2.0 Hz), 3.29 (3H, s), 3.45 (3H, s), 3.76 (1H, dd, *J* = 7.0, 2.5 Hz), 3.92 (1H, dd, *J* = 7.9, 2.5 Hz), 4.28 (1H, d, *J* = 11.4 Hz), 4.43 (1H, dd, *J* = 7.9, 2.0 Hz), 4.47 (1H, d, *J* = 11.5 Hz), 4.51 (1H, d, *J* = 7.0 Hz), 4.66 (1H, d, *J* = 11.2 Hz), 4.78 (1H, d, *J* = 11.5 Hz), 4.80 (1H, d, *J* = 11.4 Hz), 5.01 (1H, d, *J* = 11.2 Hz), 7.18-7.35 (13H, m), 7.40 (2H, d, *J* = 7.5 Hz). ¹³C NMR (100 MHz, CDCl₃) δ 53.9, 56.3, 68.6, 70.3, 74.4, 74.9, 75.2, 78.2, 80.3, 81.8, 105.3, 127.4, 127.5, 127.6, 127.9, 127.9, 128.1, 128.1, 128.3, 137.6, 138.5, 138.7; MS (ESI-TOF) *m/z* 483 [M+Na]⁺; HRMS calcd for C₂₉H₃₂NaO₅ [M+Na]⁺, 483.2147; found, 483.2122. Anal. Calcd for C₂₉H₃₂O₅: C, 75.63; H, 7.00. Found: C, 75.49; H, 7.00.

(4*S*,5*S*,6*R*,7*R*)-4,5,6-Tri(benzyloxy)-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (5c- α) and (4*S*,5*S*,6*R*,7*S*)-4,5,6-tri(benzyloxy)-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (5c- β).

According to the synthetic procedure for **5a**, these compounds were prepared in 90% yield (**5c- α** 30.2 mg, 0.064 mmol, 32% yield; **5c- β** 54.7 mg, 0.116 mmol, 58% yield) by the reaction of **4c** (92 mg, 0.20 mmol) and TMSN₃ (120 μ L, 1.0 mmol) in the presence of In(OTf)₃ (5.6 mg, 10 μ mol) in 1,2-dichloroethane (2.8 mL). **5c- α** as a less polar isomer. Colorless oil. $[\alpha]_{\text{D}}^{25}$ (*c* 1.01, CHCl₃) +35.4; IR (neat) ν 3031, 2931, 1454, 1205, 1092, 739, 698 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 3.65 (3H, s), 4.26 (1H, dd, *J* = 9.6, 3.8 Hz), 4.44 (1H, dd, *J* = 9.6, 3.6 Hz), 4.62 (1H, d, *J* = 12.2 Hz), 4.70 (1H, d, *J* = 12.2 Hz), 4.74 (1H, d, *J* = 11.8 Hz), 4.84 (1H, d, *J* = 3.8 Hz), 4.86 (1H, d, *J* = 12.0 Hz), 4.89 (1H, d, *J* = 11.8 Hz), 4.96 (1H, d, *J* = 12.0 Hz), 5.61 (1H, d, *J* = 3.6 Hz), 7.30-7.44 (15H, m), 7.56 (1H, s); ¹³C NMR (100 MHz, CDCl₃) δ 59.0, 66.7, 71.7, 73.7, 74.2, 74.7, 74.9, 86.4, 127.8, 127.8, 128.0, 128.1, 128.1, 128.4, 128.5, 128.5, 132.3, 132.5, 137.1, 137.7, 138.0; MS (ESI-TOF) *m/z* 472 [M+H]⁺; HRMS calcd for C₂₈H₃₀N₃O₄ [M+H]⁺, 472.2236; found, 472.2347. **5c- β** as a more polar isomer. White amorphous solid. Mp. 56.0-62.0 °C; $[\alpha]_{\text{D}}^{25}$ (*c* 1.00, CHCl₃) +51.7; IR (neat) ν 3030, 2929, 1454, 1191, 1118, 740, 698 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.69 (1H, dd, *J* = 9.2, 3.1 Hz), 3.79 (3H, s), 4.51 (1H, d, *J* = 12.5 Hz), 4.58 (1H, dd, *J* = 9.2, 4.6 Hz), 4.67 (1H, d, *J* = 12.0 Hz), 4.71 (1H, d, *J* = 12.5 Hz), 4.75 (1H, d, *J* = 3.1 Hz), 4.78 (1H, d, *J* = 3.1 Hz), 4.89 (1H, d, *J* = 11.3 Hz), 4.96 (1H, d, *J* = 11.3 Hz), 5.56 (1H, d, *J* = 4.6 Hz), 7.30-7.43 (15H, m), 7.57 (1H, s); ¹³C NMR (100.6 MHz, CDCl₃) δ 59.1, 65.1, 70.8, 72.6, 74.9, 77.9, 79.4, 90.9, 127.7, 127.8, 127.8, 127.9, 127.9, 128.0, 128.3, 128.4, 131.9, 131.9, 137.0, 137.5, 137.7; MS (ESI-TOF) *m/z* 472 [M+H]⁺; HRMS calcd for C₂₈H₃₀N₃O₄ [M+H]⁺, 472.2236; found, 472.2232; Anal. Calcd for C₂₈H₂₉N₃O₅: C, 71.32; H, 6.20; N, 8.91. Found: C, 71.01; H, 6.20; N, 8.99.

(4*S*,5*S*,6*R*,7*R*)-4,5,6-Trihydroxy-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (6c- α). According to the synthetic

procedure for **6a- α** , triol **6c- α** was obtained in 83% yield (34.9 mg, 0.174 mmol) by the debenzoylation reaction of **5c- α** (100 mg, 0.21 mmol) in the presence of Pd(OH)₂ on carbon (20 w/w%, 118 mg) in MeOH (3.0 mL) under H₂ atmosphere (1 atm). Colorless oil. $[\alpha]_D^{25}$ (*c* 1.00, CH₃OH) +91.2; IR (neat) ν 3358, 2937, 1448, 1191, 1110, 827 cm⁻¹; ¹H NMR (400 MHz, D₂O) δ 3.71 (3H, s), 4.33 (1H, dd, *J* = 9.6, 4.1 Hz), 4.44 (1H, dd, *J* = 9.6, 3.7 Hz), 5.24 (1H, d, *J* = 4.1 Hz), 5.92 (1H, d, *J* = 3.7 Hz), 7.93 (1H, s). ¹³C NMR (100 MHz, CDCl₃) δ 60.9, 62.9, 69.0, 69.4, 89.0, 135.1, 137.4; MS (ESI-TOF) *m/z* 202 [M+H]⁺; HRMS calcd for C₂₈H₃₀N₃O₄ [M+H]⁺, 202.0828; found, 202.0823.

(4S,5S,6R,7S)-4,5,6-Trihydroxy-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (6c- β). According to the synthetic procedure for **6a- α** , triol **6c- β** was obtained in 90% yield (37.9 mg, 0.189 mmol) by the debenzoylation reaction of **5c- β** (100 mg, 0.21 mmol) in the presence of Pd(OH)₂ on carbon (20 w/w%, 121 mg) in MeOH (3.0 mL) under H₂ atmosphere (1 atm). Colorless oil. $[\alpha]_D^{25}$ (*c* 1.00, CH₃OH) +11.2; IR (neat) ν 3358, 2927, 1456, 1254, 1115, 1056, 838 cm⁻¹; ¹H NMR (400 MHz, D₂O) δ 3.81 (3H, s), 4.08 (1H, dd, *J* = 8.7, 3.8 Hz), 4.42 (1H, dd, *J* = 8.7, 5.4 Hz), 5.18 (1H, d, *J* = 3.8 Hz), 5.61 (1H, d, *J* = 5.4 Hz), 7.90 (1H, s); ¹³C NMR (100 MHz, D₂O) δ 60.9, 63.1, 71.29, 71.33, 92.5, 134.5, 137.3; MS (ESI-TOF) *m/z* 202 [M+H]⁺; HRMS calcd for C₇H₁₂N₃O₄ [M+H]⁺, 202.0828; found, 202.0817.

3. Preparation of triazolo-gulose mimic **ent-5b**

tert-Butyl(dimethyl){[(2S,3S,4S)-2,3,4-tri(benzyloxy)hex-5-enyl]oxy}silane (7c). According to the synthetic procedure for **7a**, TBS ether **7c** was prepared in 96% yield (5.53 g, 10.4 mmol) by the reaction of (2S,3S,4S)-2,3,4-tri(benzyloxy)hex-5-en-1-ol (4.5 g, 11 mmol),³ which was prepared by NaBH₄ reduction of **1b**, imidazole (1.1 g, 16 mmol) and *tert*-butylchlorodimethylsilane (2.0 g, 13 mmol) in DMF (50 mL). Colorless oil. $[\alpha]_D^{25}$ (*c* 1.01, CHCl₃) +14.8; IR (neat) ν 3031, 2928, 2856, 1254, 1091, 837, 697 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ -0.05 (3H, s), -0.05 (3H, s), 0.83 (9H, s), 3.62 (2H, d, *J* = 6.0 Hz), 3.68 (1H, dd, *J* = 6.5, 3.6 Hz), 3.70-3.75 (1H, m), 4.04-4.09 (1H, m), 4.19 (1H, d, *J* = 11.7 Hz), 4.51-4.57 (3H, m), 4.59 (1H, d, *J* = 11.8 Hz), 4.67 (1H, d, *J* = 11.3 Hz), 5.32 (1H, brd, *J* = 17.5 Hz), 5.36 (1H, brd, *J* = 10.2 Hz), 5.92 (1H, ddd, *J* = 17.5, 10.2, 7.7 Hz), 7.19-7.30 (15H, m); ¹³C NMR (100 MHz, CDCl₃) δ -5.4, -5.4, 18.2, 25.9, 62.9, 70.1, 73.5, 74.5, 79.6, 80.1, 80.8, 119.2, 127.4, 127.4, 127.5, 127.6, 128.0, 128.1, 128.2, 128.3, 136.4, 138.6, 138.6, 139.0; MS (ESI-TOF) *m/z* 555 [M+Na]⁺; HRMS calcd for C₃₃H₄₄NaO₄Si [M+H]⁺, 555.2907; found, 555.2863. Anal. Calcd for C₃₃H₄₄O₄Si: C, 74.39; H, 8.32. Found: C, 73.99; H, 8.23.

(2R,3R,4S)-2,3,4-Tri(benzyloxy)-5-[(*tert*-butyl(dimethyl)silyl]oxy}pentanal (8c). According to the synthetic procedure for **8a**, pentanal derivative **8c** was prepared in 84% yield (4.22 g, 7.90 mmol) by the reaction of **7c** (5.0 g, 9.4 mmol), OsO₄ (0.11 M in H₂O, 9.1 mL, 1.0 mmol), NMO (3.0 g, 16.4 mmol) in a mixture of acetone (120 mL), H₂O (30 mL) and *tert*-butyl alcohol (9 mL) and following treatment of reaction mixture by NaIO₄ (5 g). Colorless oil. $[\alpha]_D^{25}$ (*c* 0.20, CHCl₃) +12.0; IR (neat) ν 3031, 2928, 2856, 1733, 1455, 1254, 1092, 837, 697 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 0.03 (3H, s), 0.04 (3H, s), 0.90 (9H, s), 3.70-3.75 (1H, m), 3.82-3.85 (2H, m), 4.01 (1H, m), 4.11 (1H, dd, *J* = 3.8, 1.4 Hz), 4.46 (1H, d, *J* = 11.8 Hz), 4.55 (1H, d, *J* = 11.8 Hz), 4.59-4.69 (4H, m), 7.26-7.38 (15H, m), 9.70 (1H, d, *J* = 1.4 Hz); ¹³C NMR (100 MHz, CDCl₃) δ -5.4, -5.4, 18.2, 25.9, 62.9, 72.7, 73.2, 73.7, 79.8, 80.0, 83.7, 127.7, 127.8, 127.8, 127.9, 128.0, 128.1, 128.3, 128.3, 128.4, 137.4, 137.8, 138.2, 201.6; MS (ESI-TOF) *m/z* 557 [M+Na]⁺; HRMS calcd for C₃₂H₄₂NaO₅Si [M+Na]⁺, 557.2699; found, 557.2689.

***tert*-Butyl(dimethyl){[(2*S*,3*R*,4*R*)-2,3,4-tri(benzyloxy)-5,5-dimethoxypentyl]oxy}silane.** This compound was obtained in 89% yield (3.87 g, 6.65 mmol) by the reaction of **8c** (4.0 g, 7.5 mmol) and HC(OMe)₃ (15 mL) in the presence of *p*TsOH·H₂O (50 mg). Colorless oil. $[\alpha]_{\text{D}}^{25}$ (*c* 1.03, CHCl₃) +11.7; IR (neat) ν 3030, 2928, 2856, 1455, 1254, 1073, 836, 697 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 0.04 (6H, s), 0.90 (9H, s), 3.41 (3H, s), 3.47 (3H, s), 3.75-3.83 (4H, m), 3.92-3.95 (1H, m), 4.59 (1H, d, *J* = 11.6 Hz), 4.61 (1H, d, *J* = 4.9 Hz), 4.61 (1H, d, *J* = 11.8 Hz), 4.67 (1H, d, *J* = 11.8 Hz), 4.70 (1H, d, *J* = 11.5 Hz), 4.73 (1H, d, *J* = 11.5 Hz), 4.84 (1H, d, *J* = 11.6 Hz), 7.26-7.36 (15H, m); ¹³C NMR (100 MHz, CDCl₃) δ -5.4, -5.4, 18.3, 25.9, 55.3, 56.0, 63.1, 73.1, 73.8, 74.2, 79.1, 79.5, 80.5, 105.3, 127.3, 127.3, 127.4, 127.6, 127.9, 127.9, 128.1, 128.2, 138.9, 139.1; MS (ESI-TOF) *m/z* 603 [M+Na]⁺; HRMS calcd for C₃₄H₄₈NaO₆Si [M+Na]⁺, 603.3118; found, 603.3127. Anal. Calcd for C₃₄H₄₈O₆Si: C, 70.31; H, 8.33. Found: C, 70.47; H, 8.29.

(2*S*,3*R*,4*R*)-2,3,4-Tri(benzyloxy)-5,5-dimethoxypentan-1-ol (9c). According to the synthetic procedure for **9a**, 5,5-dimethoxypentan-1-ol derivative **9c** was prepared in 88% yield (2.48 g, 5.31 mmol) by the reaction of *tert*-butyl(dimethyl){[(2*S*,3*R*,4*R*)-2,3,4-tri(benzyloxy)-5,5-dimethoxypentyl]oxy}silane (3.5 g, 6.0 mmol) and TBAF (1.0 M in THF, 12 mL, 12 mmol) in THF (5 mL). Colorless oil. $[\alpha]_{\text{D}}^{25}$ (*c* 0.21, CHCl₃) +16.7; IR (neat) ν 3459, 3030, 2832, 1454, 1207, 1072, 737, 698 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 2.45 (1H, brs), 3.33 (3H, s), 3.41 (3H, s), 3.67-3.74 (4H, m), 3.88 (1H, dd, *J* = 5.7, 3.3 Hz), 4.53-4.69 (6H, m), 4.76 (1H, d, *J* = 11.5 Hz), 7.21-7.32 (15H, m); ¹³C NMR (100 MHz, CDCl₃) δ 55.6, 55.8, 61.4, 72.6, 73.7, 74.2, 79.7, 79.9, 79.9, 105.1, 127.5, 127.5, 127.6, 127.8, 127.9, 127.9, 128.2, 128.3, 138.2, 138.4, 138.5; MS (ESI-TOF) *m/z* 489 [M+Na]⁺; HRMS calcd for C₂₈H₃₄NaO₆ [M+Na]⁺, 489.2253; found, 489.2255. Anal. Calcd for C₂₈H₃₄O₆: C, 72.08; H, 7.35. Found: C, 71.97; H, 7.37.

(2*R*,3*R*,4*R*)-2,3,4-Tri(benzyloxy)-5,5-dimethoxypentanal (ent-3b). According to the synthetic procedure for **ent-3a**, 5,5-dimethoxypentanal derivative **ent-3b** was prepared in 88% yield (1.75 g, 3.78 mmol) by the reaction of **7c** (2.0 g, 4.3 mmol) and DMP (3.0 g, 7.2 mmol) in CH₂Cl₂ (15 mL). The physical data of **ent-3b** were coincident with those of **3b**, except for specific optical rotation. $[\alpha]_{\text{D}}^{25}$ (*c* 1.00, CHCl₃) +29.8.

(2*R*,3*R*,4*S*)-2,3,4-Tri(benzyloxy)-1,1-dimethoxyhex-5-yne (ent-4b). This compound was prepared from **ent-3b** under the same conditions in a case of **4b**. The physical data of **ent-3b** were coincident with those of **3b**, except for specific optical rotation. $[\alpha]_{\text{D}}^{25}$ (*c* 0.99, CHCl₃) +52.7.

(4*S*,5*R*,6*R*,7*S*)-4,5,6-Tri(benzyloxy)-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (ent-5b- α) and

(4*S*,5*R*,6*R*,7*R*)-4,5,6-tri(benzyloxy)-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (ent-5b- β).

According to the synthetic procedure for **5b**, these compounds were prepared in 86% yield (**ent-5b- α** 45.2 mg, 95 μ mol, 48% yield; **ent-5b- β** 36.0 mg, 76 μ mol, 38% yield) by the reaction of **ent-5b** (92.1 mg, 0.20 mmol) and TMSN₃ (120 μ L, 1.0 mmol) in the presence of In(OTf)₃ (5.6 mg, 10 μ mol) in 1,2-dichloroethane (3.0 mL). The physical data of **ent-5b- α** were coincident with those of **5b- α** , except for specific optical rotation. $[\alpha]_{\text{D}}^{25}$ (*c* 0.21, CHCl₃) +23.5. The physical data of **ent-5b- β** were also coincident with those of **5b- β** , except for specific optical rotation. $[\alpha]_{\text{D}}^{25}$ (*c* 1.00, CHCl₃) +74.5.

4. Preparation of triazolo-altrose mimic **ent-5c**

tert-Butyl(dimethyl){[(2R,3S,4R)-2,3,4-tri(benzyloxy)hex-5-enyl]oxy}silane (7b). According to the synthetic procedure for **7a**, TBS ether **7b** was prepared in 89% yield (3.98 g, 7.49 mmol) by the reaction of (2R,3S,4R)-2,3,4-tri(benzyloxy)hex-5-en-1-ol (3.5 g, 8.4 mmol),³ which was prepared by NaBH₄ reduction of **1b**, imidazole (743 mg, 11 mmol) and *tert*-butylchlorodimethylsilane (1.52 g, 10 mmol) in DMF (9.0 mL). Colorless oil. $[\alpha]_{\text{D}}^{25}$ (*c* 1.00, CHCl₃) -14.1; IR (neat) ν 2928, 2856, 1455, 1253, 1093, 836, 734, 697 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 0.05 (6H, s), 0.91 (9H, s), 3.67-3.72 (1H, m), 3.72-3.79 (1H, m), 3.83 (1H, dd, *J* = 10.9, 5.3 Hz), 3.96 (1H, dd, *J* = 10.9, 2.9 Hz), 4.13 (1H, dd, *J* = 7.7, 4.2 Hz), 4.34 (1H, d, *J* = 11.9 Hz), 4.40 (1H, d, *J* = 11.5 Hz), 4.63 (1H, d, *J* = 11.9 Hz), 4.65 (1H, d, *J* = 11.5 Hz), 4.70-4.73 (2H, m), 5.27 (1H, brd, *J* = 10.4 Hz), 5.33 (1H, brd, *J* = 17.4 Hz), 5.91 (1H, ddd, *J* = 17.4, 10.4, 7.7 Hz), 7.18-7.36 (15H, m); ¹³C NMR (100 MHz, CDCl₃) δ -5.4, 18.2, 25.9, 62.6, 70.5, 72.3, 74.7, 79.8, 80.5, 81.5, 118.2, 127.3, 127.3, 127.4, 127.6, 127.9, 128.0, 128.1, 128.1, 136.2, 138.5, 138.6, 138.7; MS (ESI-TOF) *m/z* 533 [M+H]⁺; HRMS calcd for C₃₃H₄₅O₄Si [M+H]⁺, 533.3087; found, 533.3120.

(2S,3R,4R)-2,3,4-Tri(benzyloxy)-5-{*tert*-butyl(dimethyl)silyl}oxy}pentanal (8b). According to the synthetic procedure for **8a**, pentanal derivative **8b** was prepared in 92% yield (3.49 g, 6.50 mmol) by the reaction of **7b** (3.78 g, 7.1 mmol), OsO₄ (0.11 M in H₂O, 6.4 mL, 0.7 mmol), NMO (1.6 g, 14 mmol) in a mixture of acetone (100 mL), H₂O (35 mL) and *tert*-butyl alcohol (3 mL) and following treatment of reaction mixture by NaIO₄ (15 g). Colorless oil. $[\alpha]_{\text{D}}^{25}$ (*c* 1.02, CHCl₃) -34.1; IR (neat) ν 3032, 2952, 2929, 1731, 1455, 1327, 1253, 1096, 1028, 736, 697 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 0.01 (3H, s), 0.02 (3H, s), 0.88 (9H, s), 3.66-3.74 (1H, m), 3.78 (1H, dd, *J* = 11.2, 4.2 Hz), 3.95 (1H, dd, *J* = 11.2, 2.9 Hz), 4.03-4.07 (2H, m), 4.32 (1H, d, *J* = 11.5 Hz), 4.47 (1H, d, *J* = 11.8 Hz), 4.52 (1H, d, *J* = 11.3 Hz), 4.57 (1H, d, *J* = 11.3 Hz), 4.62 (1H, d, *J* = 11.8 Hz), 4.64 (1H, d, *J* = 11.5 Hz), 7.18-7.30 (15H, m), 9.58 (1H, d, *J* = 1.4 Hz); ¹³C NMR (100 MHz, CDCl₃) δ -5.4, -5.4, 18.3, 25.9, 61.4, 72.0, 73.2, 74.1, 78.4, 78.9, 84.2, 127.5, 127.7, 127.8, 128.0, 128.1, 128.2, 128.3, 128.3, 128.4, 137.3, 137.8, 138.1, 202.2; MS (ESI-TOF) *m/z* 535 [M+H]⁺; HRMS calcd for C₃₂H₄₃O₅Si [M+H]⁺, 535.2880; found, 535.2889.

tert-Butyl(dimethyl){[(2R,3R,4S)-2,3,4-tri(benzyloxy)-5,5-dimethoxypentyl]oxy}silane. This compound was obtained in 100% yield (3.48 g, 6.00 mmol) by the reaction of **8b** (3.2 g, 6.0 mmol) and HC(OMe)₃ (15 mL) in the presence of *p*TsOH·H₂O (20 mg). Colorless oil. $[\alpha]_{\text{D}}^{25}$ (*c* 0.98, CHCl₃) -7.20; IR (neat) ν 3031, 2929, 2856, 1454, 1096, 1028, 836, 734, 697 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 0.02 (3H, s), 0.02 (3H, s), 0.89 (9H, s), 3.28 (3H, s), 3.43 (3H, s), 3.73 (1H, ddd, *J* = 7.2, 4.8, 2.3 Hz), 3.76-3.82 (2H, m), 3.88 (1H, dd, *J* = 7.2, 2.6 Hz), 4.01 (1H, dd, *J* = 11.2, 2.3 Hz), 4.32 (1H, d, *J* = 11.7 Hz), 4.50 (1H, d, *J* = 11.6 Hz), 4.53 (1H, d, *J* = 7.1 Hz), 4.63 (1H, d, *J* = 11.6 Hz), 4.70 (1H, d, *J* = 11.7 Hz), 4.72 (1H, d, *J* = 11.6 Hz), 4.81 (1H, d, *J* = 11.6 Hz), 7.18-7.35 (15H, m); ¹³C NMR (100 MHz, CDCl₃) δ -5.4, -5.3, 18.2, 25.9, 53.5, 55.9, 62.2, 71.8, 74.2, 74.3, 78.2, 78.8, 79.5, 105.4, 127.2, 127.4, 127.8, 127.9, 128.1, 128.2, 128.2, 138.9, 139.1; MS (ESI-TOF) *m/z* 603 [M+Na]⁺; HRMS calcd for C₃₄H₄₈NaO₆Si [M+Na]⁺, 603.3118; found, 603.3062.

(2R,3R,4S)-2,3,4-Tri(benzyloxy)-5,5-dimethoxypentan-1-ol (9b). According to the synthetic procedure for **9a**, 5,5-dimethoxypentan-1-ol derivative **9b** was prepared in 98% yield (2.74 g, 5.88 mmol) by the reaction of *tert*-butyl(dimethyl){[(2R,3R,4S)-2,3,4-tri(benzyloxy)-5,5-dimethoxypentyl]oxy}silane (3.4 g, 6.0 mmol) and TBAF (1.0 M in THF, 12 mL, 12 mmol) in THF (5 mL). Colorless oil. $[\alpha]_{\text{D}}^{25}$ (*c* 1.00, CHCl₃) -8.70; IR (neat) ν 3481, 3031, 2933, 1454, 1092, 1029, 735, 697 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.28 (3H, s), 3.43 (3H, s), 3.65-3.70 (2H, m), 3.74 (1H, brd, *J* = 12.0 Hz), 3.85 (1H, dd,

$J = 12.0, 3.6$ Hz), 3.90 (1H, dd, $J = 6.9, 2.9$ Hz), 4.29 (1H, d, $J = 11.6$ Hz), 4.48 (1H, d, $J = 12.0$ Hz), 4.50 (1H, d, $J = 6.7$ Hz), 4.51 (1H, d, $J = 12.0$ Hz), 4.63 (1H, d, $J = 11.5$ Hz), 4.72 (1H, d, $J = 11.5$ Hz), 4.81 (1H, d, $J = 11.6$ Hz), 7.19-7.34 (15H, m); ^{13}C NMR (100 MHz, CDCl_3) δ 54.1, 56.2, 60.2, 71.2, 74.1, 74.6, 78.1, 7.7, 78.9, 105.6, .127.5, 127.6, 127.6, 127.7, 128.0, 128.0, 128.2, 128.3, 128.4, 138.1, 138.4, 138.6; MS (ESI-TOF) m/z 489 $[\text{M}+\text{Na}]^+$; HRMS calcd for $\text{C}_{28}\text{H}_{34}\text{NaO}_6$ $[\text{M}+\text{Na}]^+$, 489.2253; found, 489.2232.

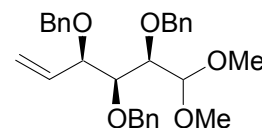
(2S,3R,4S)-2,3,4-Tri(benzyloxy)-5,5-dimethoxypentanal (ent-3c). According to the synthetic procedure for *ent-3a*, 5,5-dimethoxypentanal derivative *ent-3c* was prepared in 88% yield (2.20 g, 4.73 mmol) by the reaction of **9b** (2.5 g, 5.4 mmol) and DMP (3.3 g, 8.0 mmol) in CH_2Cl_2 (15 mL). The physical data of *ent-3c* were coincident with those of **3c**, except for specific optical rotation. $[\alpha]_{\text{D}}^{25}$ (c 1.00, CHCl_3) -8.20 .

(2S,3R,4R)-2,3,4-Tri(benzyloxy)-1,1-dimethoxyhex-5-yne (ent-4c). This compound was prepared from *ent-3c* under the same conditions in a case of **4c**. The physical data of *ent-4c* were coincident with those of **4c**, except for specific optical rotation. $[\alpha]_{\text{D}}^{25}$ (c 1.01, CHCl_3) -54.2 .

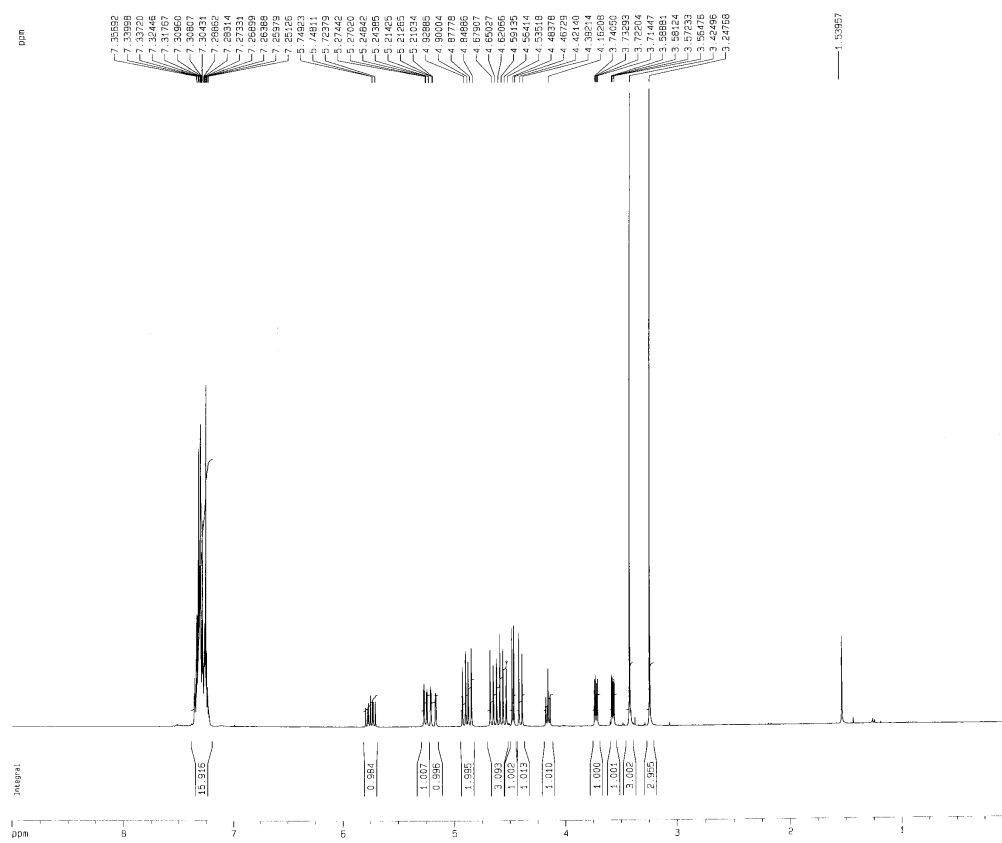
(4R,5R,6S,7S)-4,5,6-Tri(benzyloxy)-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (ent-5c- α) and (4R,5R,6S,7R)-4,5,6-tri(benzyloxy)-7-methoxy-4,5,6,7-tetrahydro[1,2,3]triazolo[1,5-*a*]pyridine (ent-5c- β).

According to the synthetic procedure for **5c**, these compounds were prepared in 89% yield (*ent-5c- α* 85.3 mg, 0.181 mmol, 28% yield; *ent-5c- β* 187.6 mg, 0.398 mmol, 61% yield) by the reaction of *ent-4c* (300 mg, 0.65 mmol) and TMSN_3 (0.40 mL, 3.3 mmol) in the presence of $\text{In}(\text{OTf})_3$ (18.2 mg, 32.5 μmol) in 1,2-dichloroethane (9.0 mL). The physical data of *ent-5c- α* were coincident with those of **5c- α** , excluding specific optical rotation. $[\alpha]_{\text{D}}^{25}$ (c 1.01, CHCl_3) -36.8 . The physical data of *ent-5c- β* were also coincident with those of **5c- β** , except for specific optical rotation. $[\alpha]_{\text{D}}^{25}$ (c 0.98, CHCl_3) -51.3 .

5. ^1H and ^{13}C NMR spectra of **2a**, **3a**, **4a**, **5a** and **6a**



2a



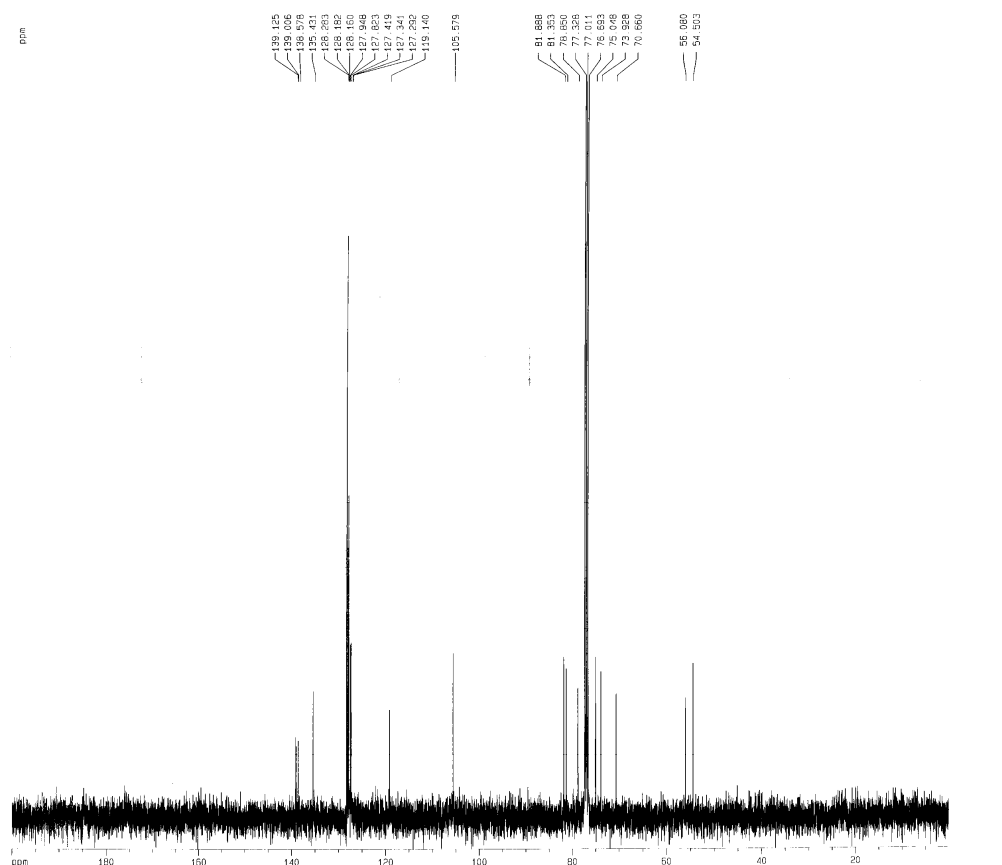
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EXPNO 5
PROCNO 1

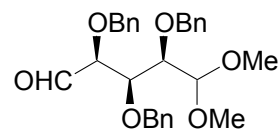
F2 - Acquisition Parameters
Date_ 20080228
Time 11.26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 26178.010 Hz
FIDRES 0.359445 Hz
AQ 1.2517875 sec
RG 11505.2
DM 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
SOL1 0.00000000 sec
SOL2 1.85999998 sec
MCREST 0.00000000 sec
MCWK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5966886 MHz

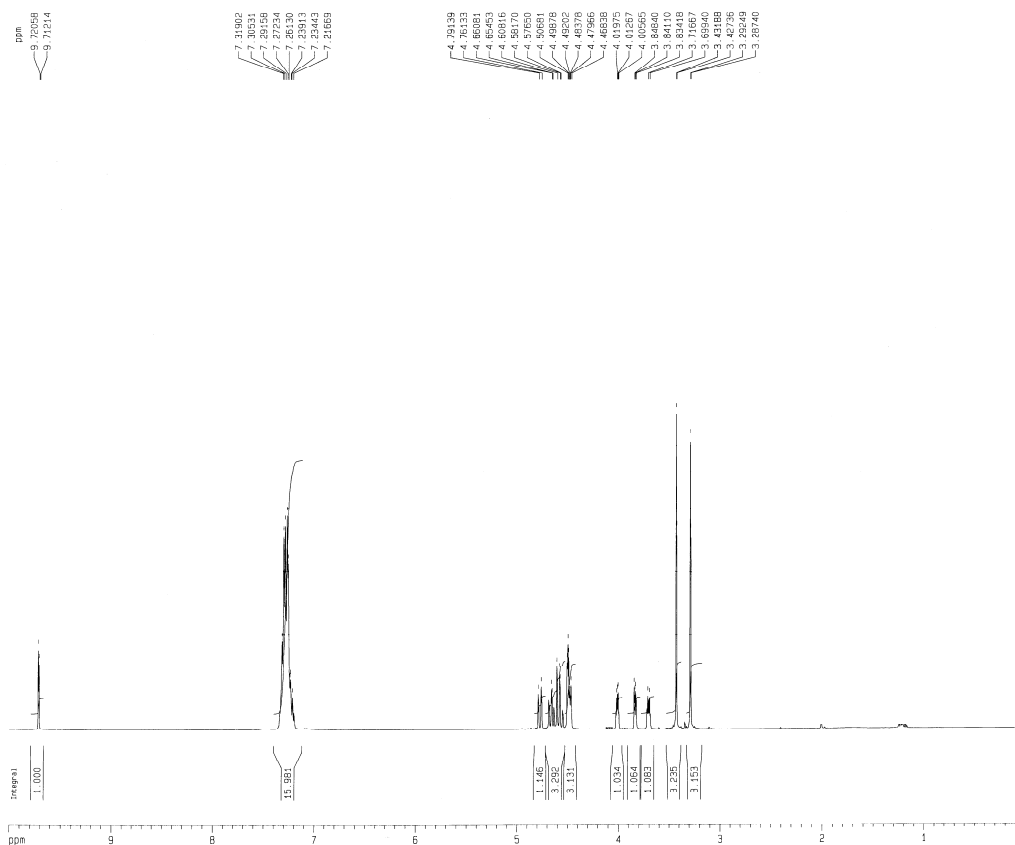
----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876230 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 19.00 cm
F1P 206.000 ppm
F2P 20117.52 Hz
F3P -0.000 ppm
F2 0.00 Hz
PPMCM 6.65657 ppm/cm
HZCM 670.58411 Hz/cm



3a



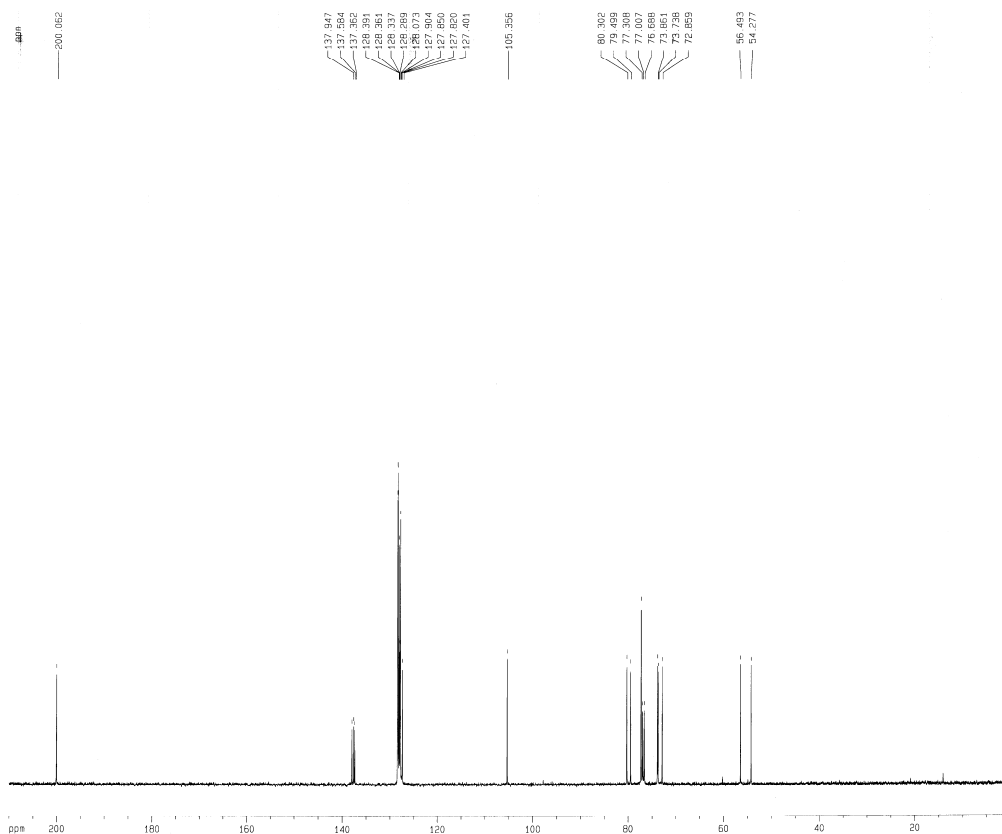
Current Data Parameters
NAME A3C2-5a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050728
Time 12.22
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PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT cdcl3
NS 32
DS 0
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 45.3
DM 73.600 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWVK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.40 usec
PL1 8.00 dB
SF01 400.1326354 MHz

F2 - Processing parameters
SI 16384
SF 400.1300319 MHz
WDW EM
SSB 0
IR 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
FPC 0.31250 ppm/cm
H2CM 125.04063 Hz/cm



Current Data Parameters
NAME A3C2-5a
EXPNO 2
PROCNO 1

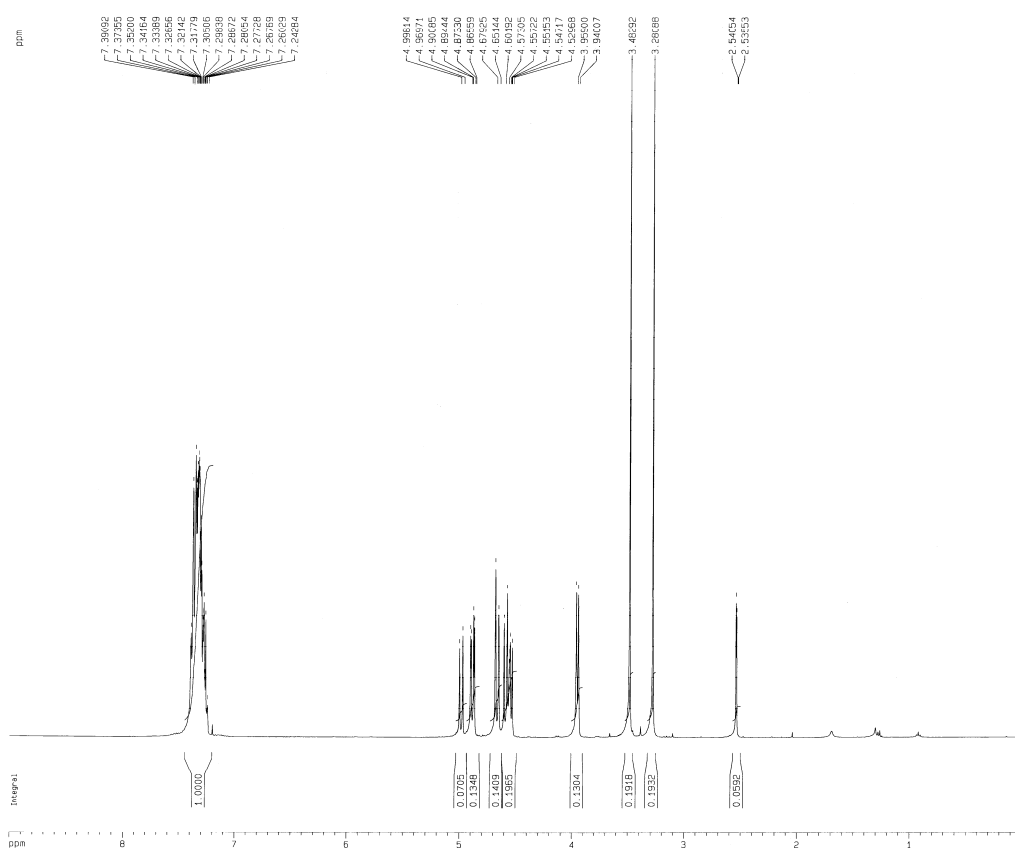
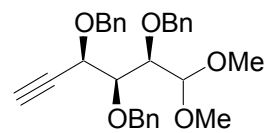
F2 - Acquisition Parameters
Date_ 20050728
Time 12.29
INSTRUM dp4400
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT cdcl3
NS 112
DS 2
SWH 26176.010 Hz
FIDRES 0.399444 Hz
AQ 1.2517875 sec
RG 16384
DM 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.88999999 sec
MCREST 0.00000000 sec
MCWVK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -6.00 dB
SF01 100.6238401 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL12 16.40 dB
PL13 16.40 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127853 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 10.00 cm
F1P 210.000 ppm
F1 21128.89 Hz
F2P 0.000 ppm
F2 0.00 Hz
FPC 6.56250 ppm/cm
H2CM 660.07140 Hz/cm



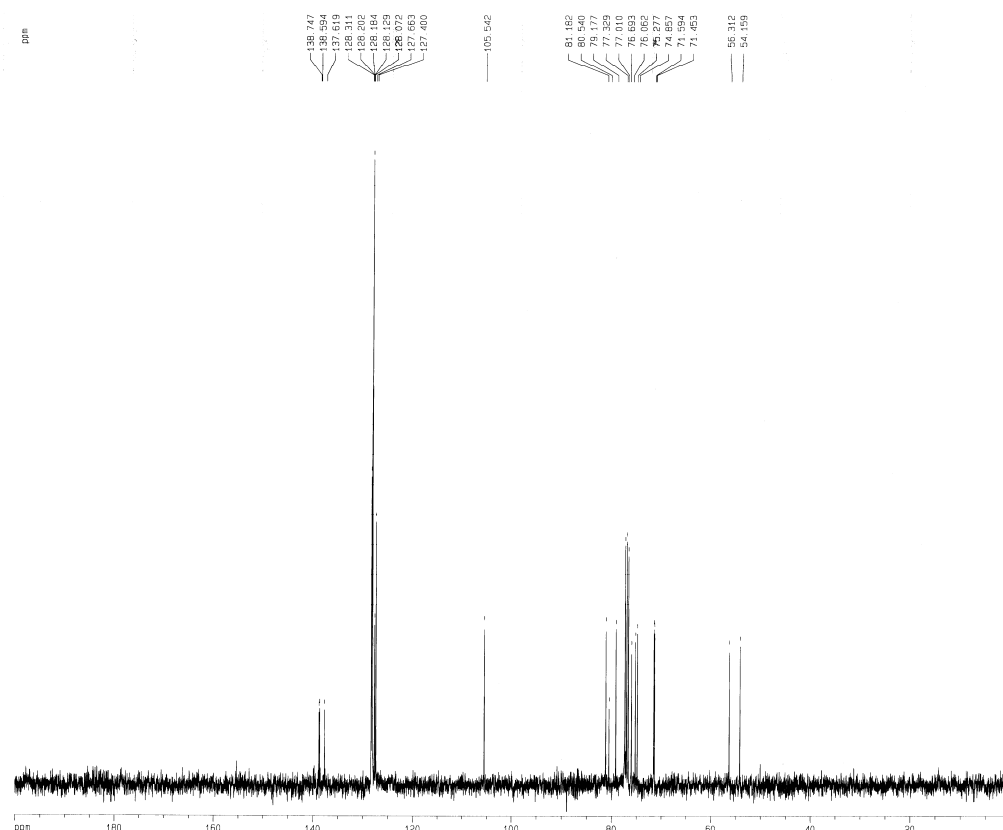
Current Data Parameters
NAME A3C2-7a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050730
Time 11.25
INSTRUM gpcx400
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 95.5
DM 73.500 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MREST 0.00000000 sec
MORCK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 7.40 usec
PL1 -6.00 dB
SFO1 400.1363954 MHz

F2 - Processing parameters
SI 32768
SF 400.1300332 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.53656 Hz/cm



Current Data Parameters
NAME A3C2-7a
EXPNO 2
PROCNO 1

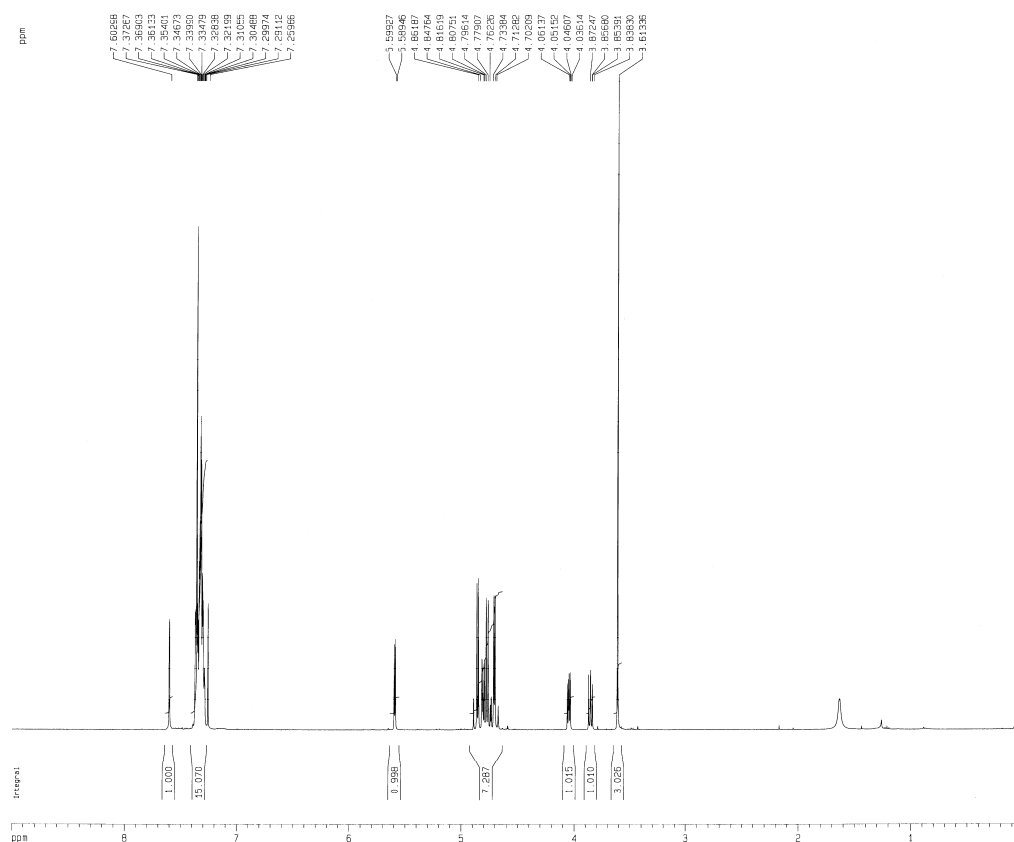
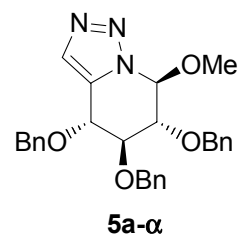
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Time 15.24
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PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 18
DS 2
SWH 26176.010 Hz
FIDRES 0.359445 Hz
AQ 1.2517875 sec
RG 2298 R
DM 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MREST 0.00000000 sec
MORCK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 8.00 usec
PL1 -6.00 dB
SFO1 100.6238401 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.40 dB
PL13 16.40 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127774 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 20.00 cm
F1P 200.900 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 626.62990 Hz/cm



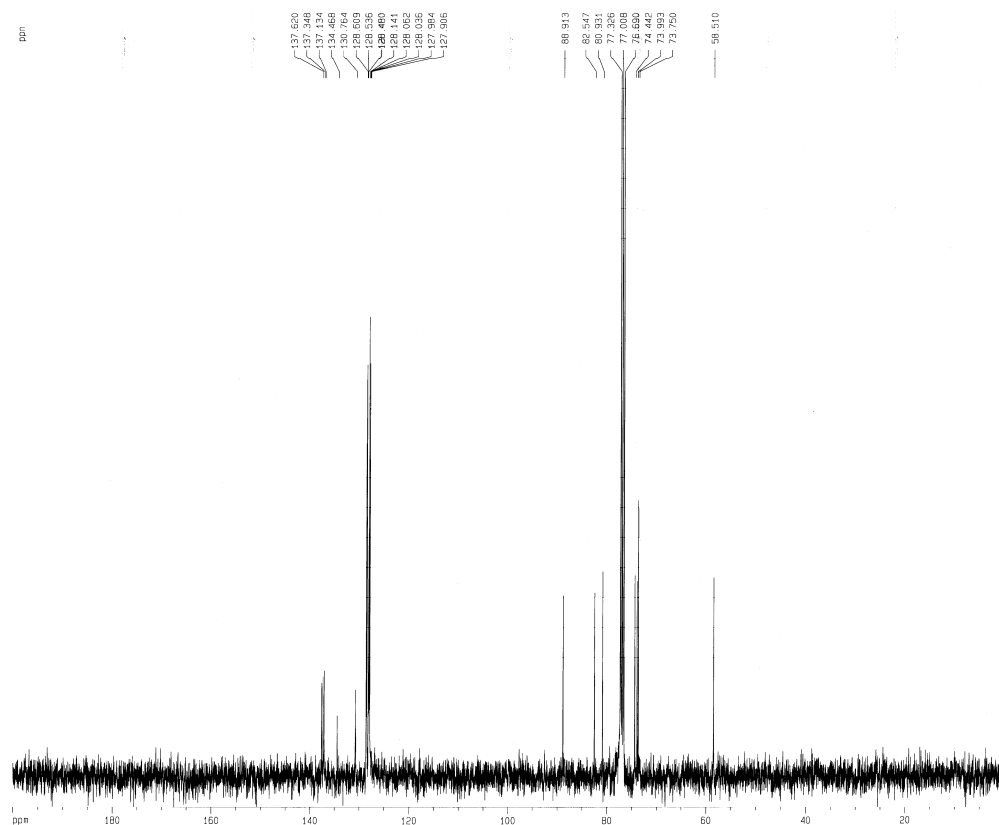
Current Data Parameters
NAME A3C2-8a-less
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050727
Time 20.36
INSTRUM gpc400
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6793.470 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 406.4
DM 73.600 usec
DE 9.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.40 usec
PL1 -6.00 dB
SFO1 400.1326324 MHz

F2 - Processing parameters
SI 62684
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPNMC 0.28125 ppm/cm
HZCM 112.53656 Hz/cm



Current Data Parameters
NAME A3C2-8a-less
EXPNO 2
PROCNO 1

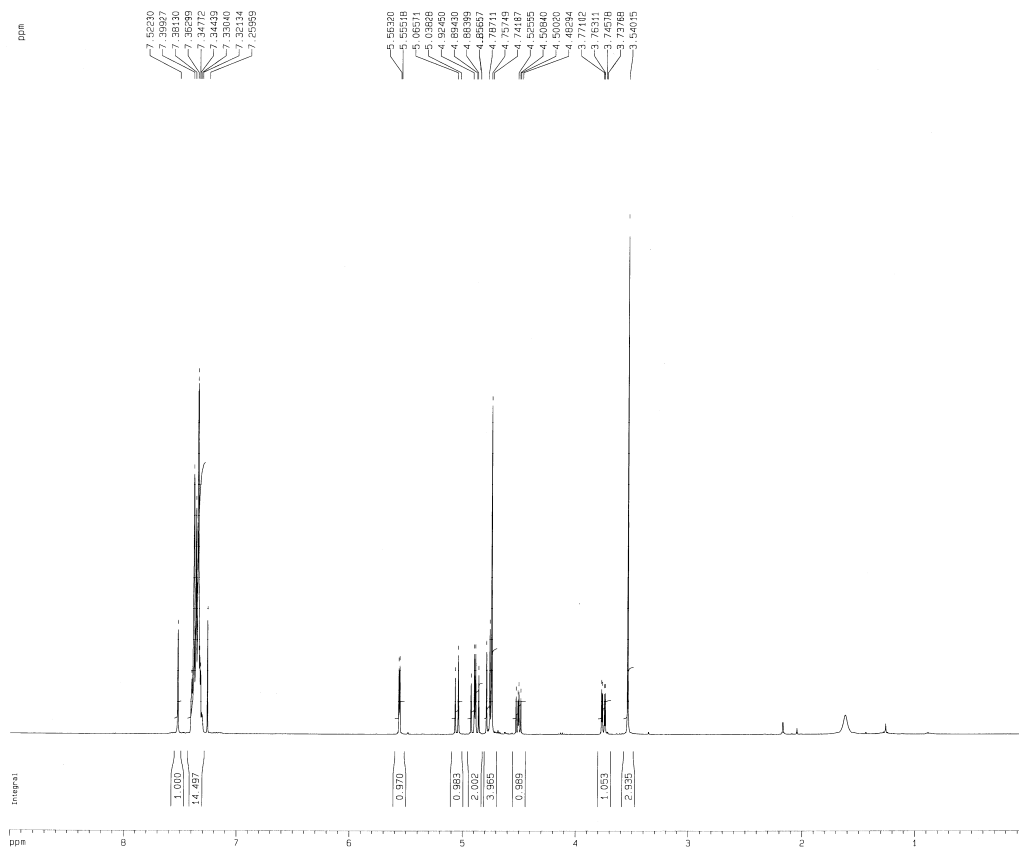
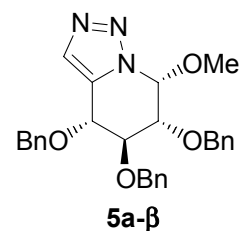
F2 - Acquisition Parameters
Date_ 20050727
Time 20.11
INSTRUM gpc400
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 173
DS 2
SWH 26178.010 Hz
FIDRES 0.399449 Hz
AQ 1.2517075 sec
RG 2048
DM 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -6.00 dB
SFO1 100.6238401 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL12 16.40 dB
PL13 16.40 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127710 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 45.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPNMC 6.25000 ppm/cm
HZCM 628.82383 Hz/cm



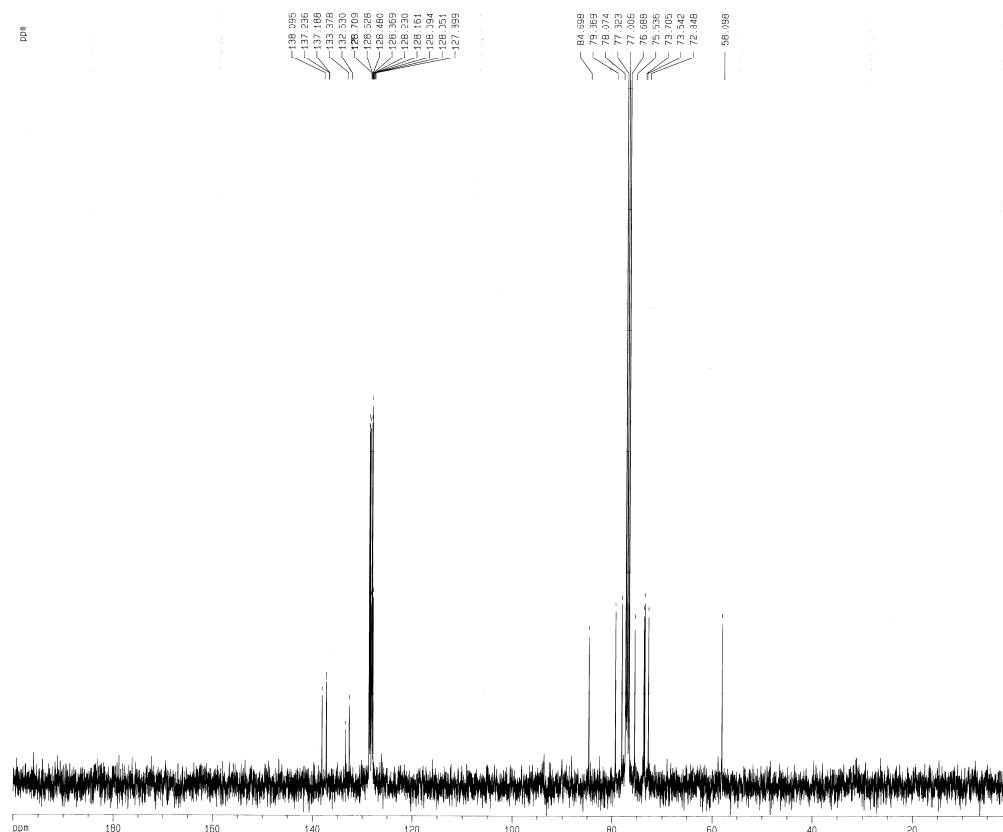
Current Data Parameters
 NAME A3C2-Ba-more
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050728
 Time 7.57
 INSTRUM dp400
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.4117749 sec
 RG 456.1
 DW 73.600 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCKRK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.40 usec
 PL1 -6.00 dB
 SF01 400.1326354 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300095 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 CY 45.00 cm
 F1P 9.000 ppm
 F1 3601.17 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.2815 ppm/cm
 HZCM 112.53656 Hz/cm



Current Data Parameters
 NAME A3C2-Ba-more
 EXPNO 2
 PROCNO 1

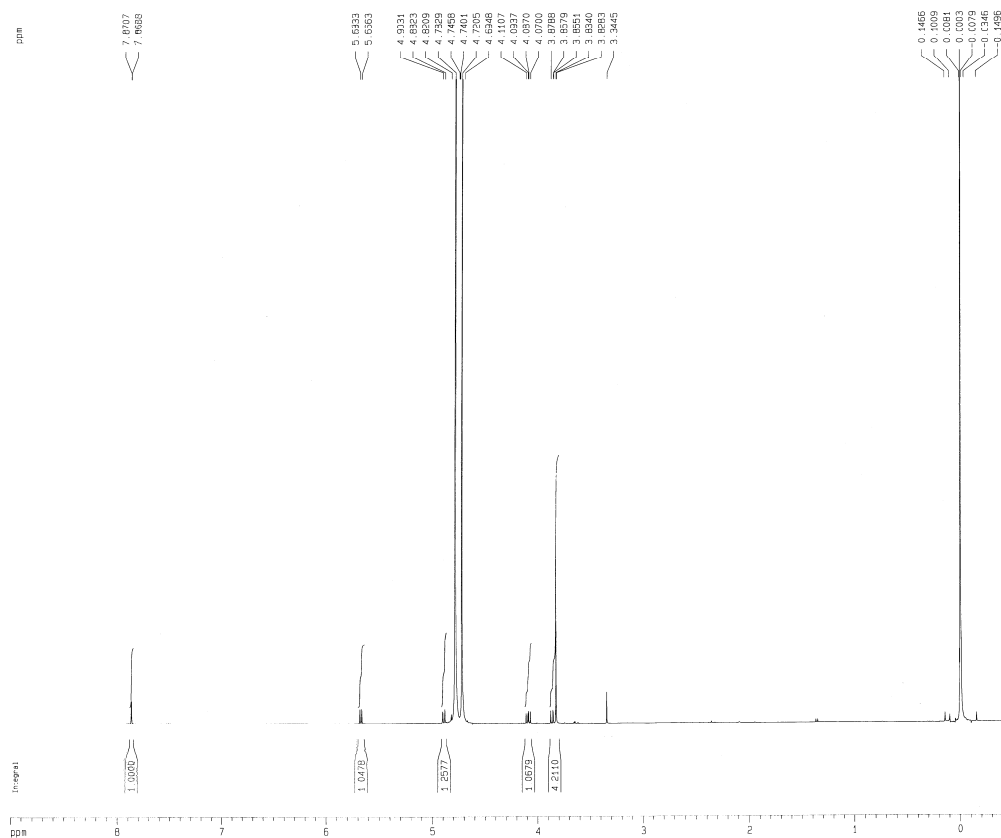
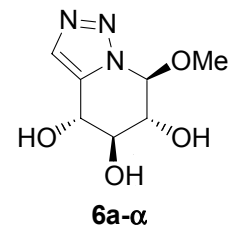
F2 - Acquisition Parameters
 Date_ 20050728
 Time 2.18
 INSTRUM dp400
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 173
 DS 2
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517875 sec
 RG 16304
 DW 19.100 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCKRK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 8.00 usec
 PL1 -6.00 dB
 SF01 100.6238401 MHz

***** CHANNEL f2 *****
 NUC2 1H
 P1 100.00 usec
 PL2 -6.00 dB
 PL12 16.40 dB
 PL13 18.40 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127710 MHz
 WDM EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 32.00 cm
 CY 45.00 cm
 F1P 200.000 ppm
 F1 20122.55 Hz
 F2P -0.000 ppm
 F2 -0.00 Hz
 PPMCM 6.25000 ppm/cm
 HZCM 628.82983 Hz/cm



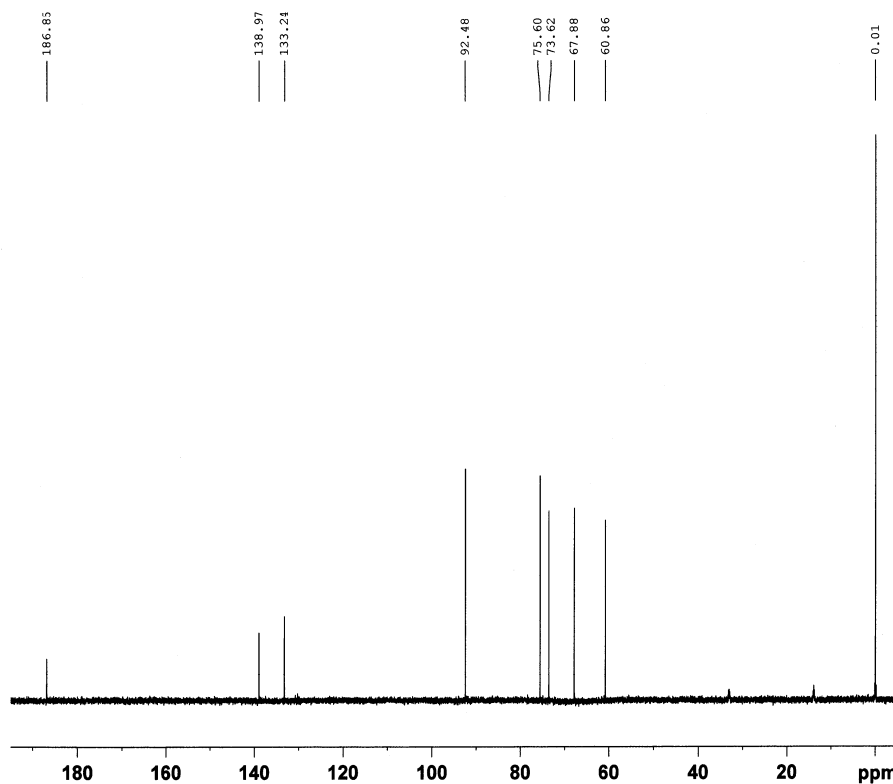
Current Data Parameters
 NAME g1c-dh-less
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071228
 Time 20.26
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 64
 DW 60.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.00000000 sec
 MCHREST 0.00000000 sec
 MCMRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -4.00 dB
 SFO1 400.0324703 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0299640 MHz
 MW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 CY 50.00 cm
 F1P 3.000 ppm
 F1 3600.27 Hz
 F2P -0.500 ppm
 F2 -200.01 Hz
 PRMCM 0.29588 ppm/cm
 HZCM 118.75880 Hz/cm



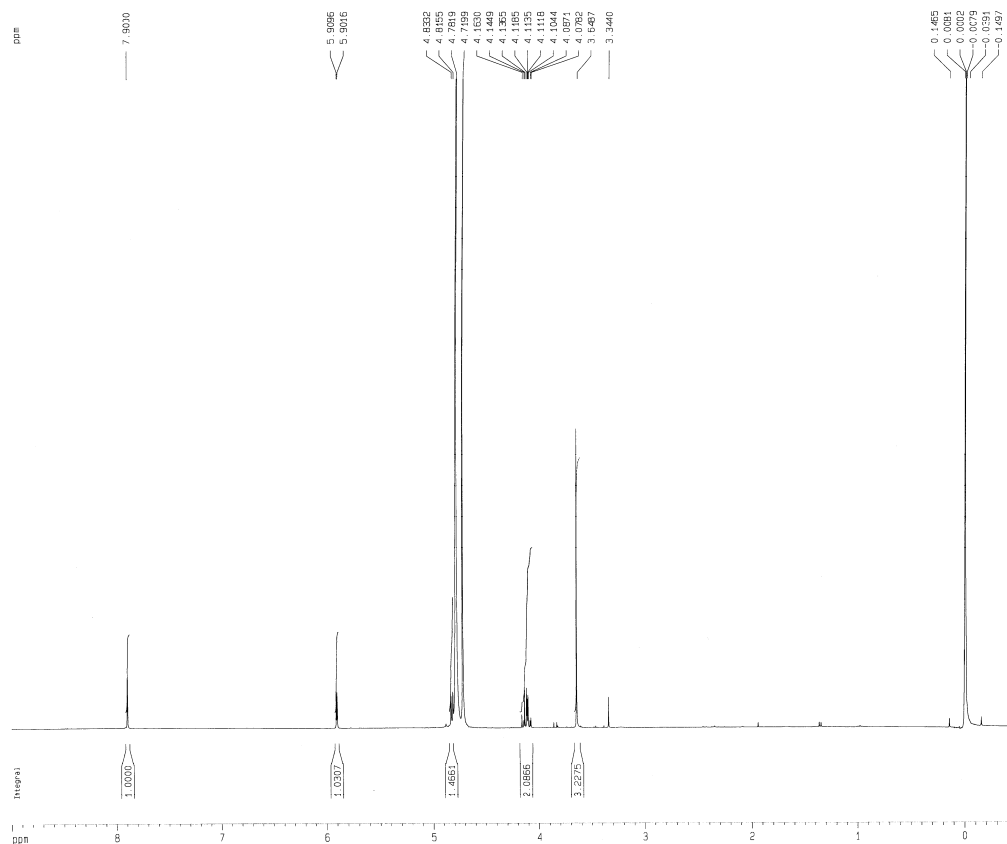
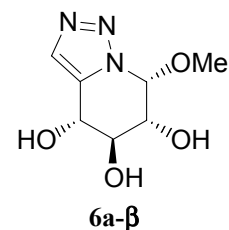
Current Data Parameters
 NAME g1cchl
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071225
 Time 10.13
 INSTRUM AV600
 PROBHD 5 mm CPTCI 1H-
 PULPROG zgpg30
 TD 65402
 SOLVENT D2O
 NS 256
 DS 16
 SWH 35971.223 Hz
 FIDRES 0.5550002 Hz
 AQ 0.9091517 sec
 RG 9200
 DW 13.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 15.40 usec
 PL1 -6.00 dB
 SFO1 150.9194083 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -5.40 dB
 PL12 12.00 dB
 PL13 12.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 32768
 SF 150.9025026 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



```

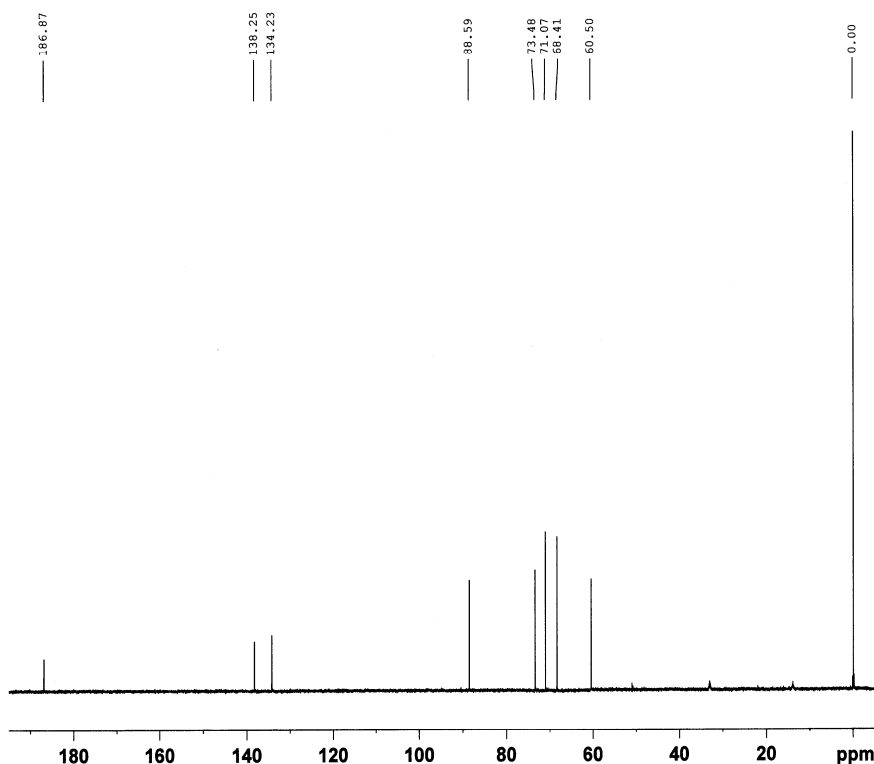
Current Data Parameters
NAME      Bic-DH-more
EXPNO    4
PROCNO   1

F2 - Acquisition Parameters
Date_    20071228
Time     21.27
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       64
DS       2
SWH      8278.146 Hz
FIDRES   0.126314 Hz
AQ       3.9584243 sec
RG       71.0
DM       60.400 usec
DE       6.00 usec
TE       300.2 K
D1       1.00000000 sec
MCREST   0.00000000 sec
MCWRR    0.01500000 sec

===== CHANNEL f1 =====
NUC1     1H
P1       11.50 usec
PL1      -4.00 dB
SFO1     400.0324703 MHz

F2 - Processing parameters
SI       32768
SF       400.0299645 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00

1D NMR plot parameters
CX       32.00 cm
CY       50.00 cm
F1P      9.000 pph
F1       360.20 Hz
F2P      -0.500 ppm
F2       -200.01 Hz
PPMCM    0.29666 ppm/cm
HZCM     118.75890 Hz/cm
    
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```

Current Data Parameters
NAME      glicohm
EXPNO    4
PROCNO   1

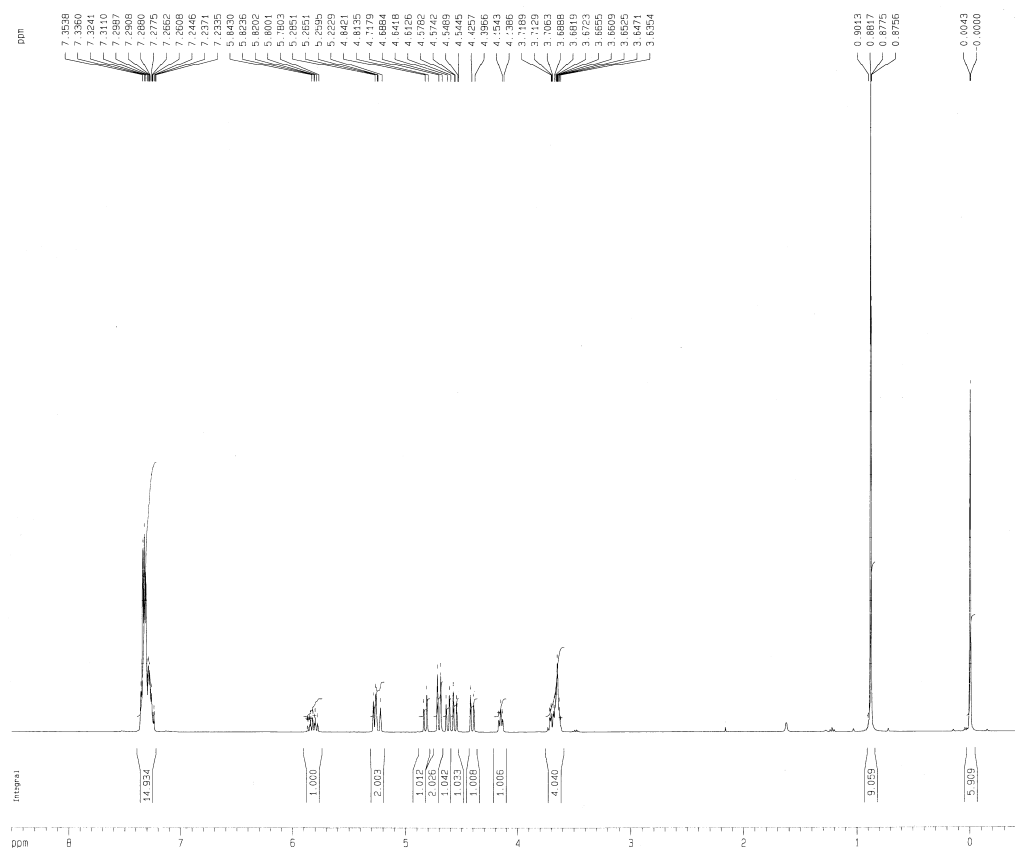
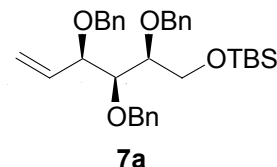
F2 - Acquisition Parameters
Date_    20071225
Time     9.29
INSTRUM  AV600
PROBHD   5 mm CPTCI 1H-
PULPROG  zgpg30
TD       65402
SOLVENT  D2O
NS       288
DS       16
SWH      35971.223 Hz
FIDRES   0.550002 Hz
AQ       0.9091517 sec
RG       9200
DM       13.900 usec
DE       6.00 usec
TE       300.0 K
D1       2.00000000 sec
d11      0.03000000 sec
DELTA    1.899999998 sec
TD0      1

===== CHANNEL f1 =====
NUC1     13C
P1       15.40 usec
PL1      -6.00 dB
SFO1     150.9194083 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      -5.40 dB
PL12     12.00 dB
PL13     12.00 dB
SFO2     600.1324005 MHz

F2 - Processing parameters
SI       32768
SF       150.9025026 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
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6. ¹H and ¹³C NMR spectra of **7a**, **8a** and **9a**



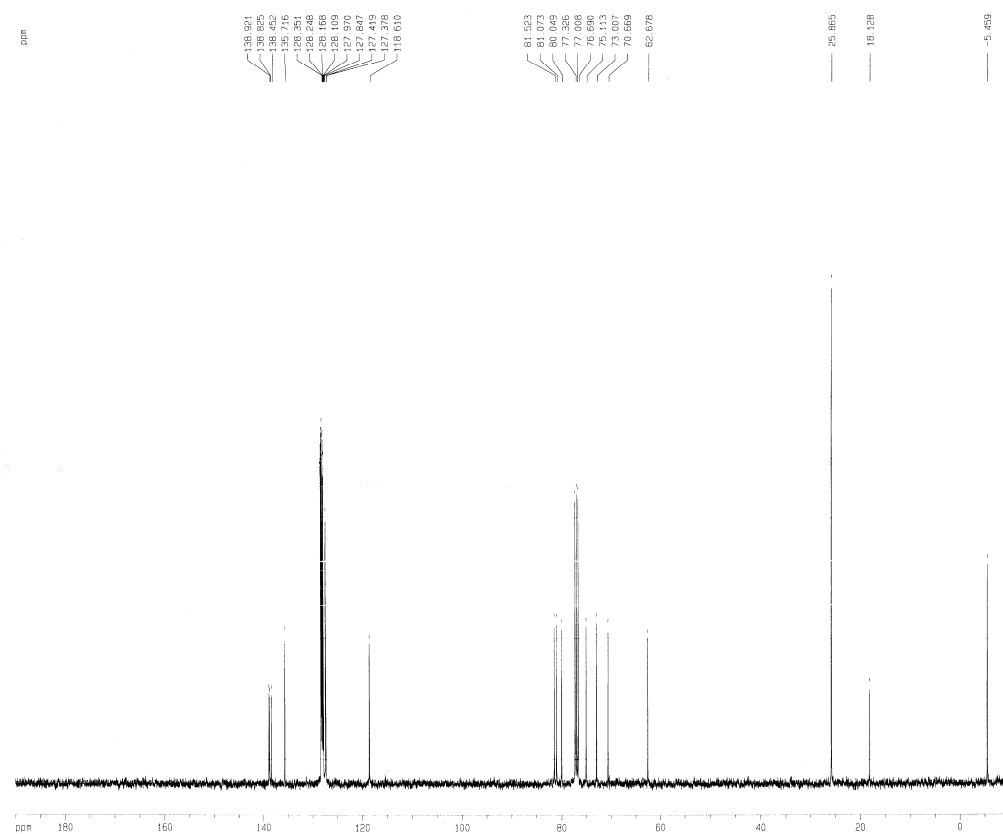
Current Data Parameters
NAME 43C2-10a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070713
Time 20.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 655
DM 73.600 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.40 usec
PL1 -6.00 dB
SFO1 400.1326354 MHz

F2 - Processing parameters
SI 16384
SF 400.1300150 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.53656 Hz/cm



Current Data Parameters
NAME 43C2-10a
EXPNO 2
PROCNO 1

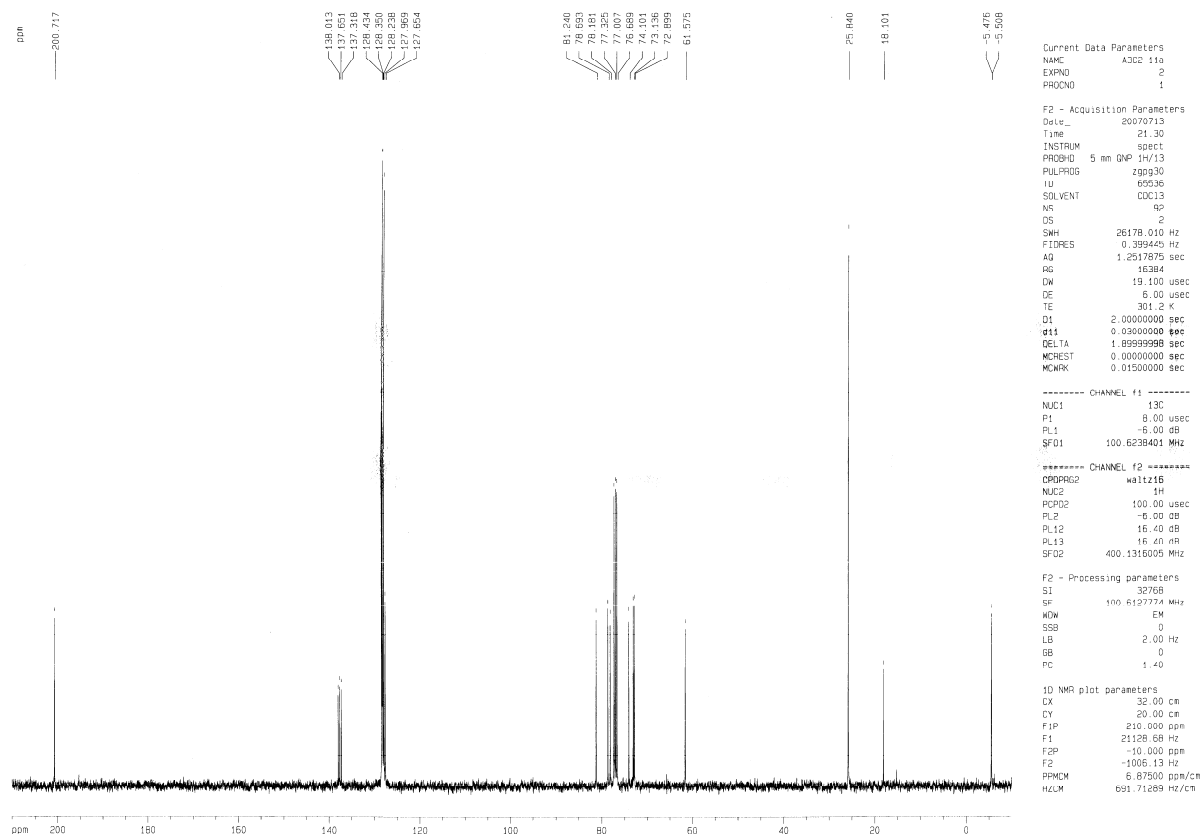
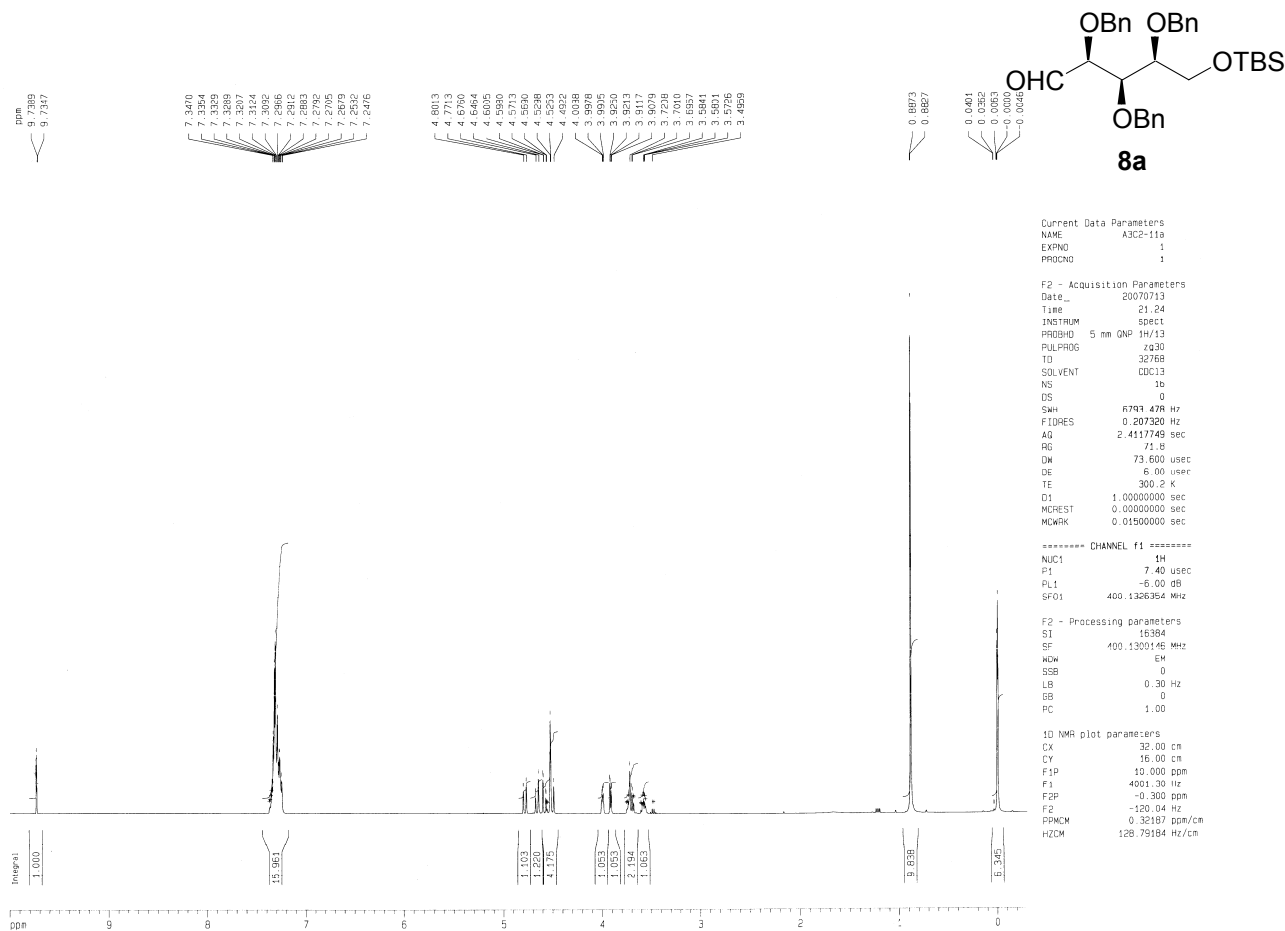
F2 - Acquisition Parameters
Date_ 20070713
Time 21.02
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 104
DS 2
SWH 26178.010 Hz
FIDRES 0.293445 Hz
AQ 1.2517675 sec
RG 16384
DM 19.100 usec
DE 6.00 usec
TE 301.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d11A 1.00000000 sec
MCREST 0.00000000 sec
MCWK 0.01500000 sec

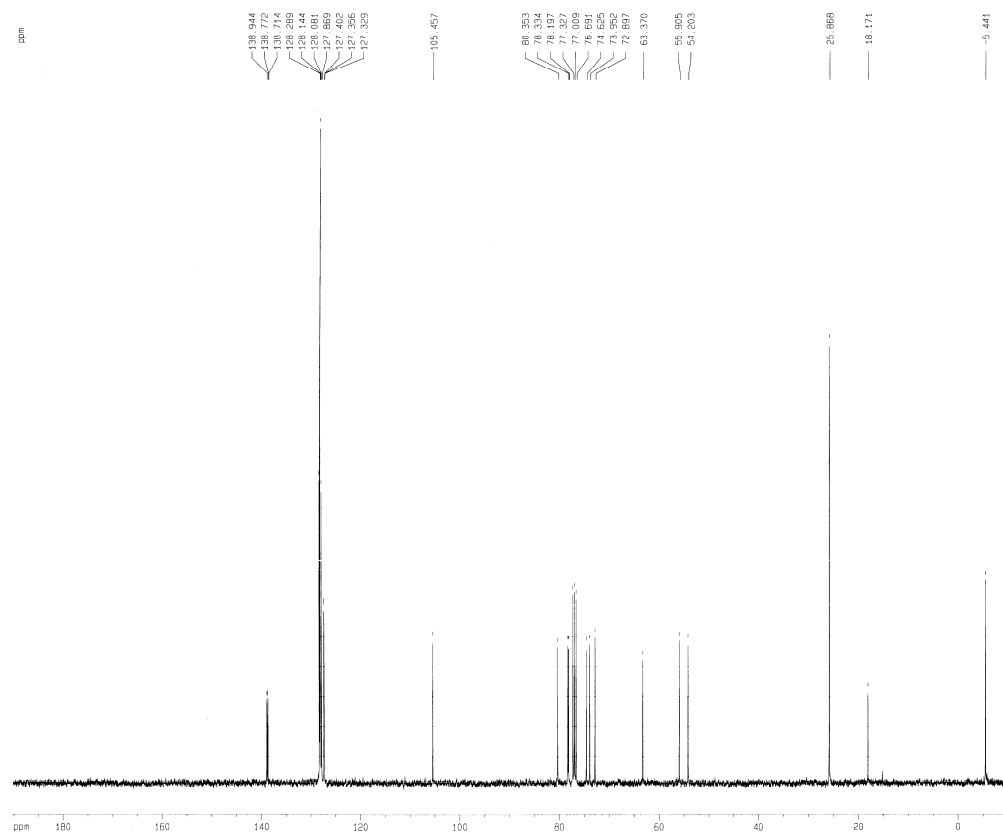
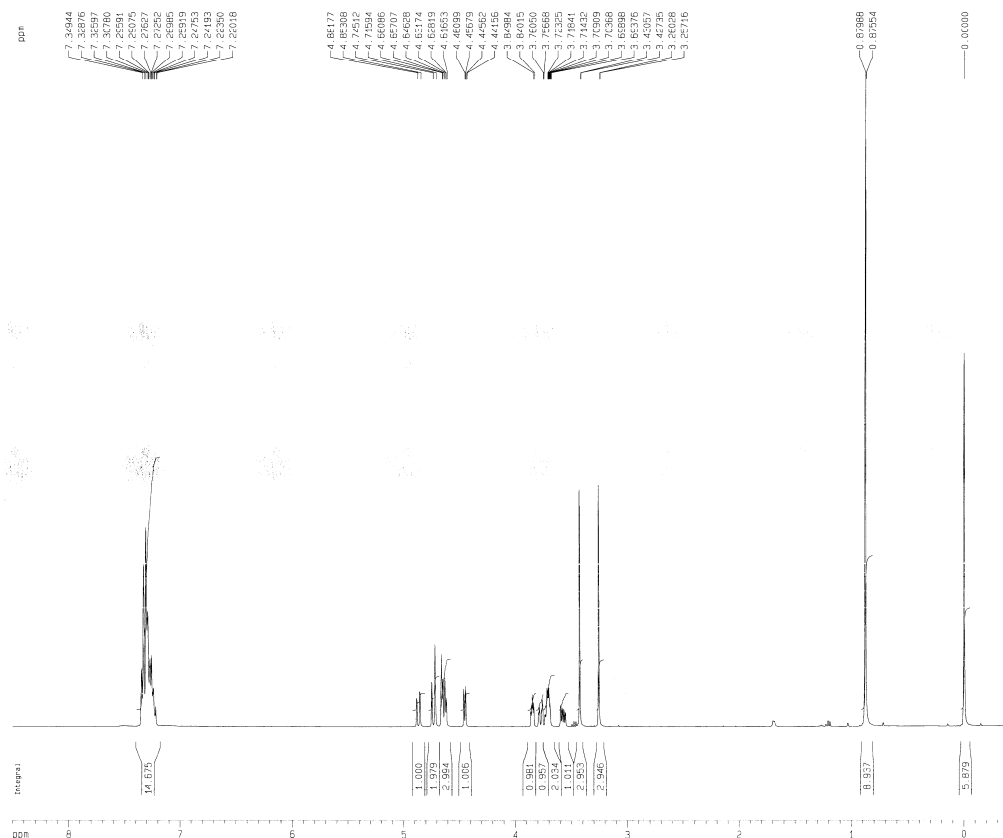
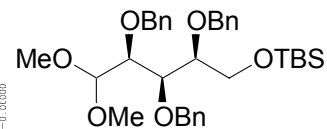
===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -6.00 dB
SFO1 100.6284601 MHz

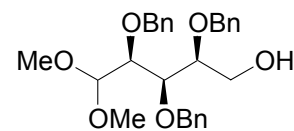
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2P2 100.00 usec
PL2 -6.00 dB
PL12 16.40 dB
PL13 16.40 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127774 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

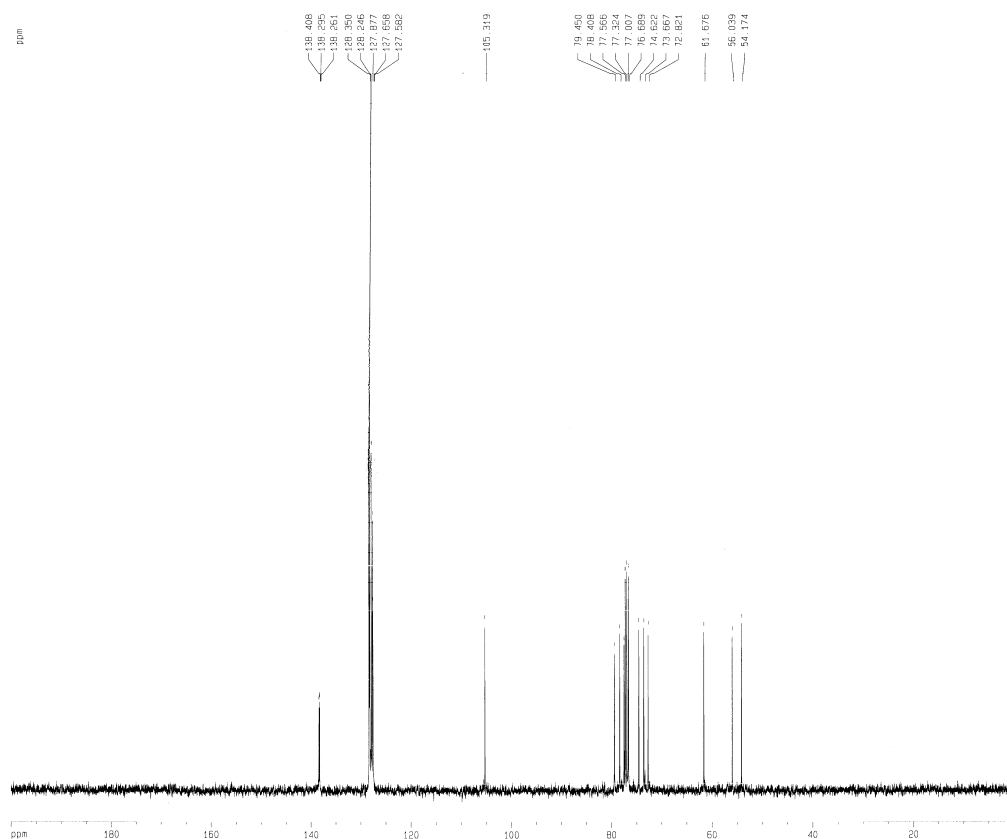
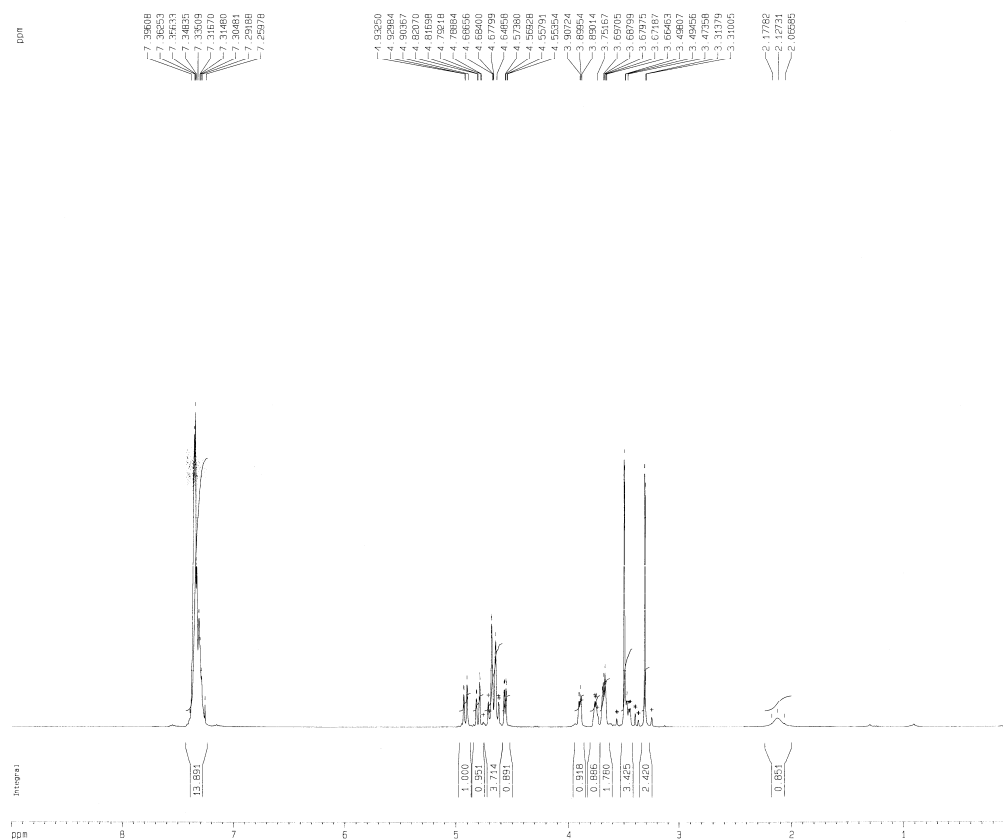
1D NMR plot parameters
CX 32.00 cm
CY 16.00 cm
F1P 190.000 ppm
F1 19116.43 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.82990 Hz/cm



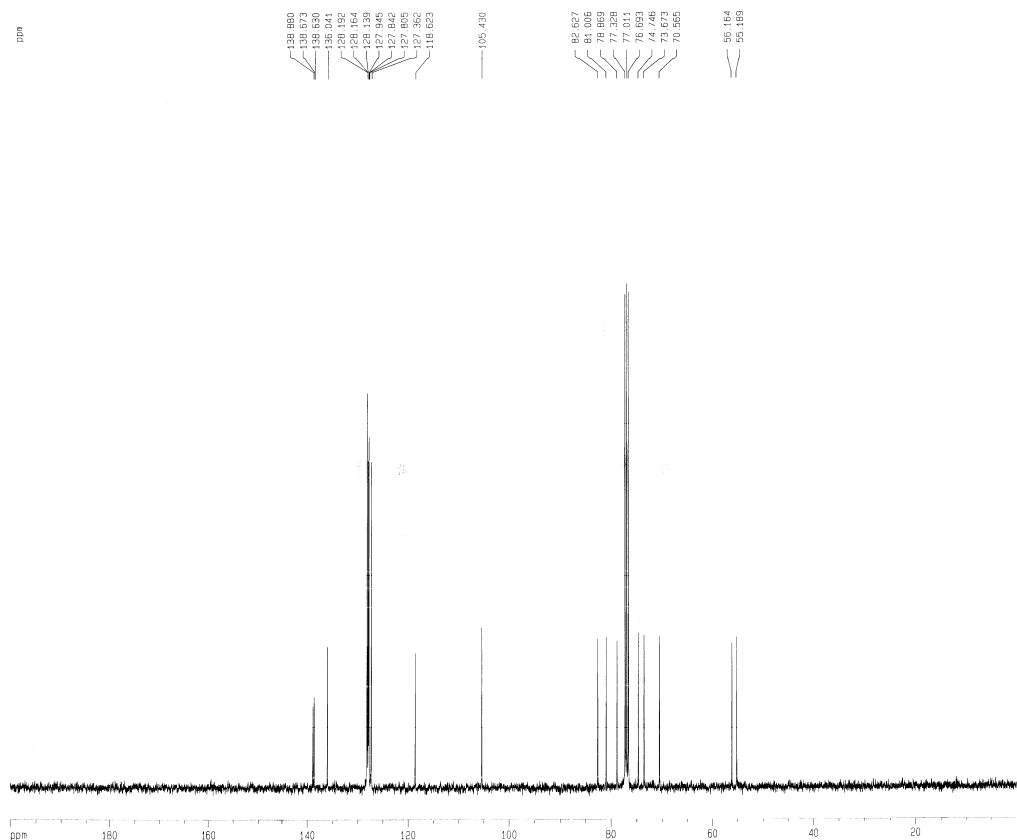
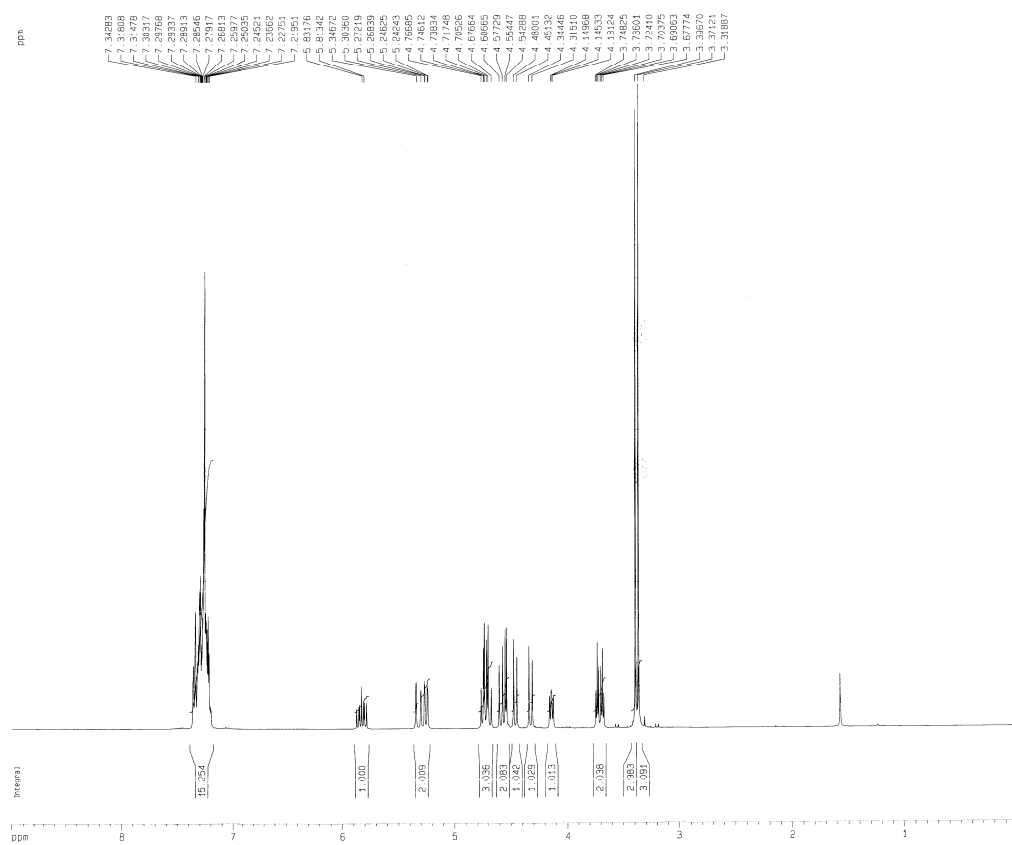
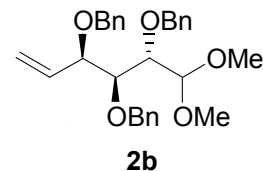


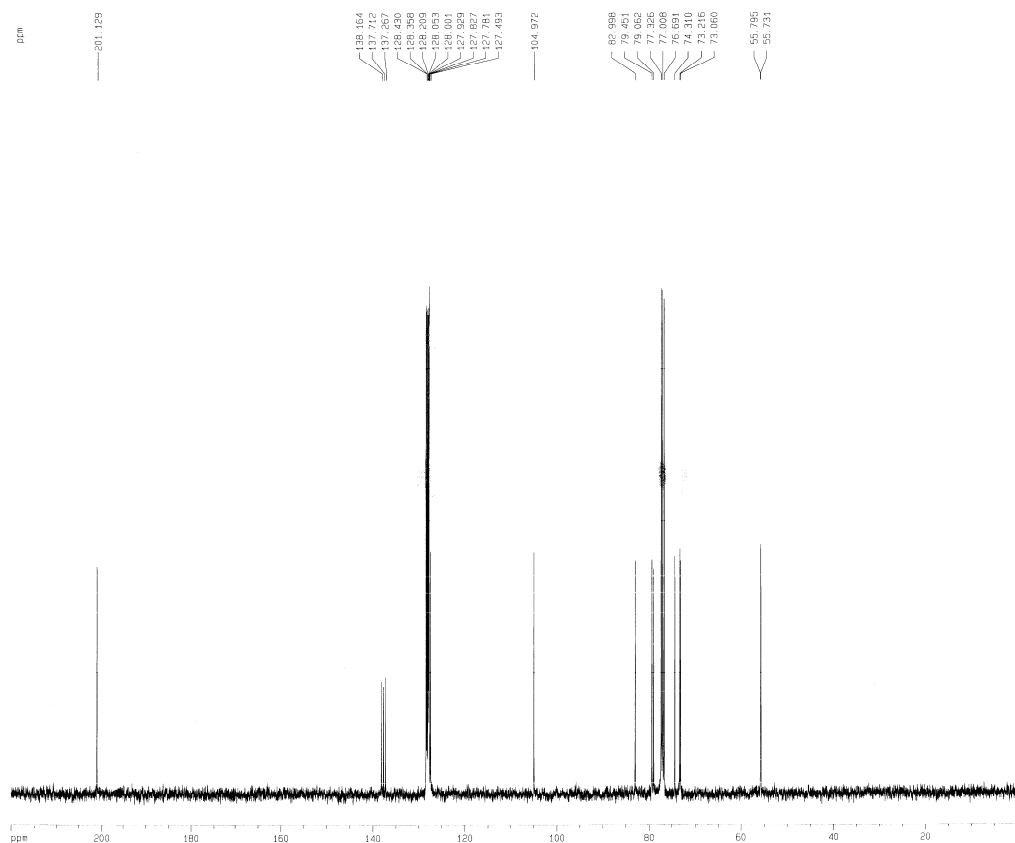
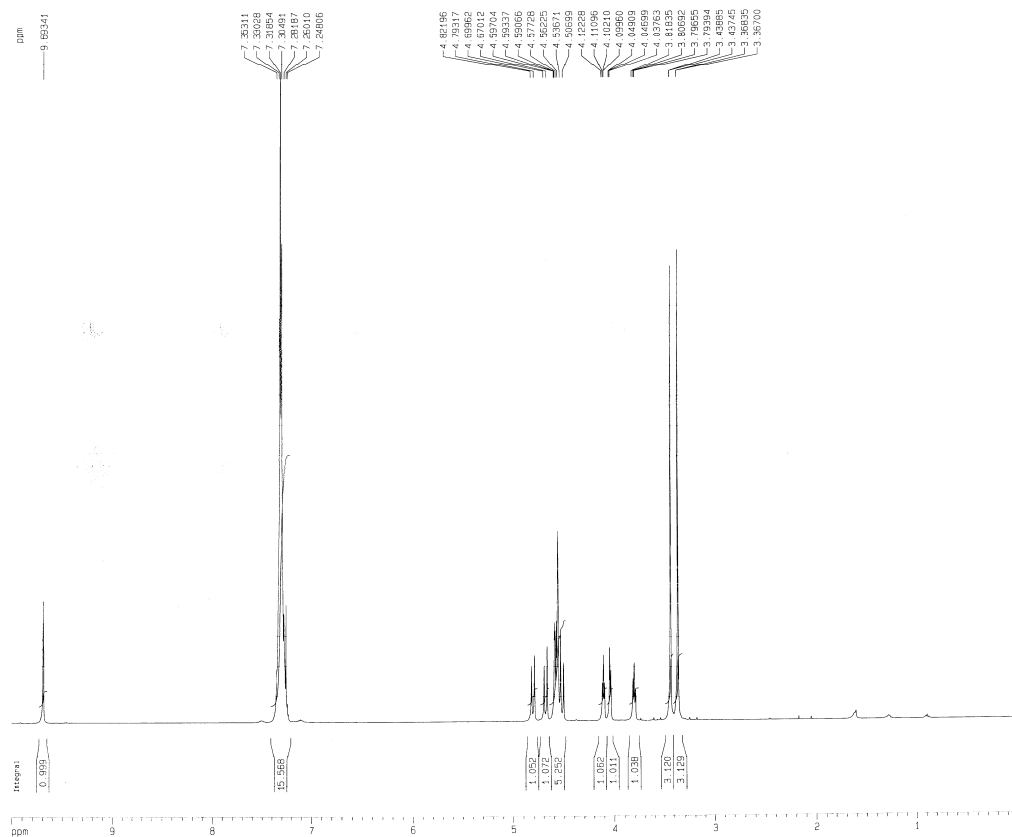
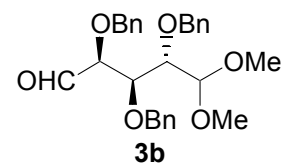


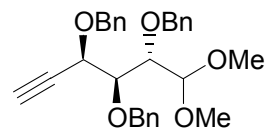
9a



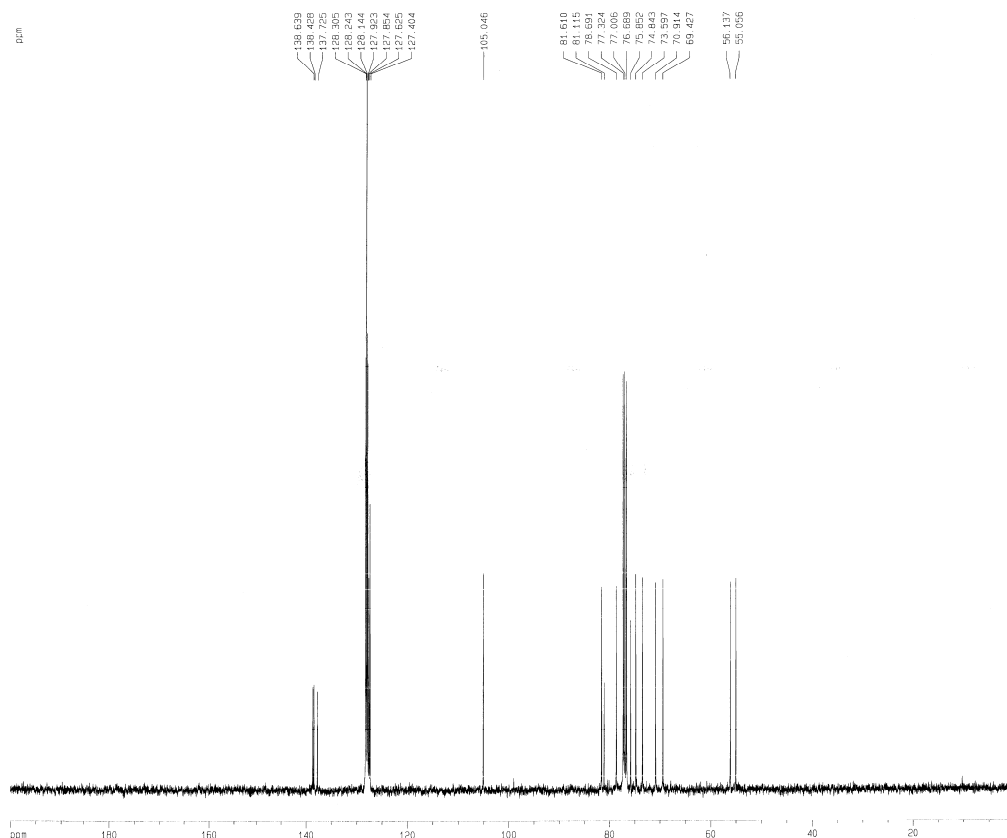
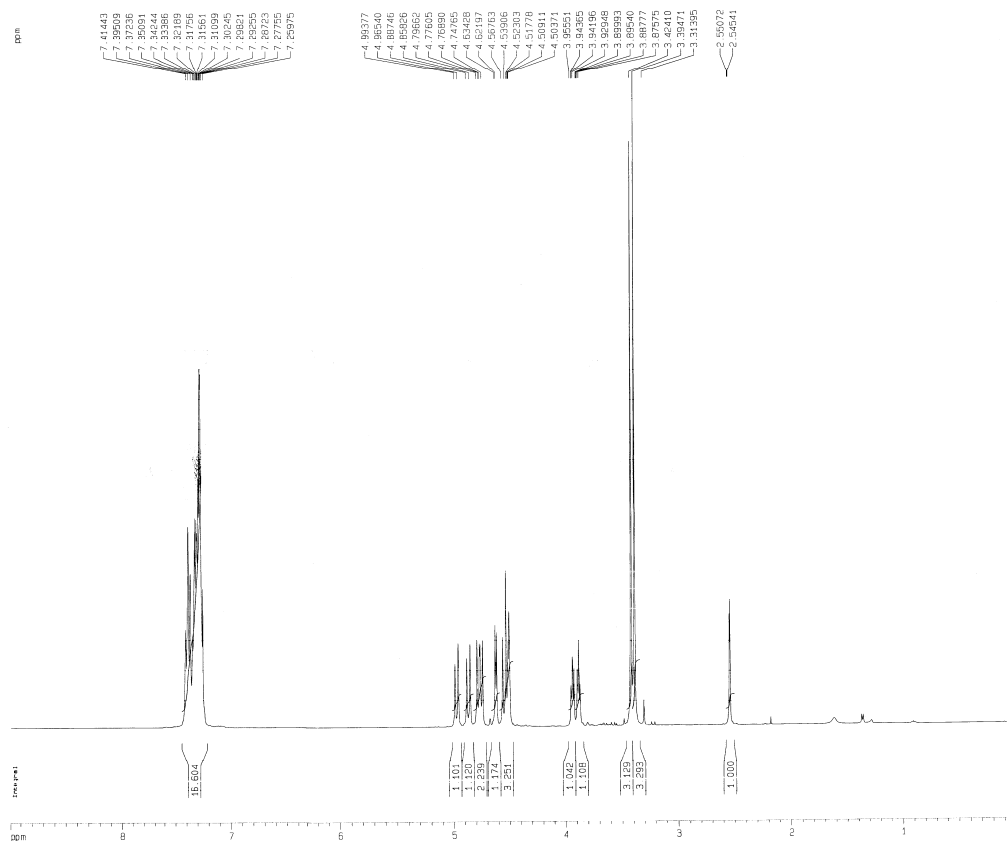
7. ¹H and ¹³C NMR spectra of **2b**, **3b**, **4b**, **5b** and **6b**

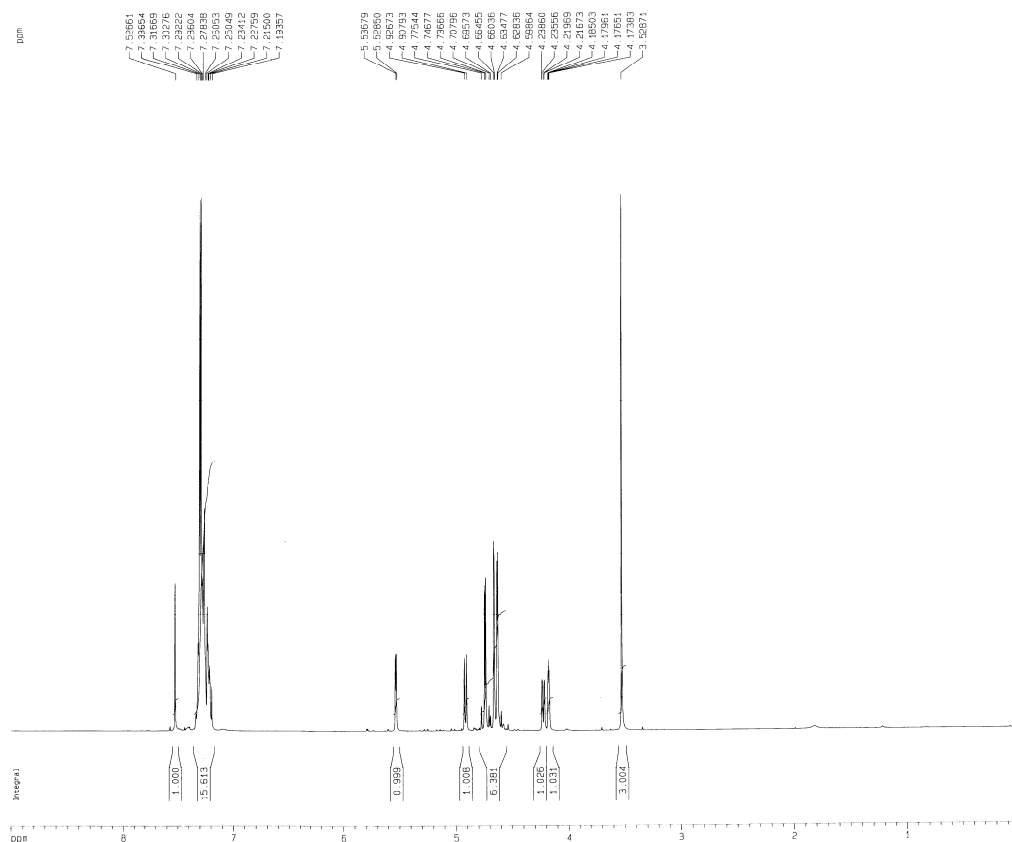
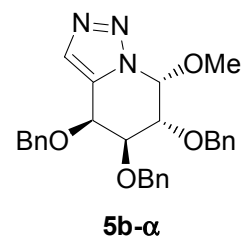






4b





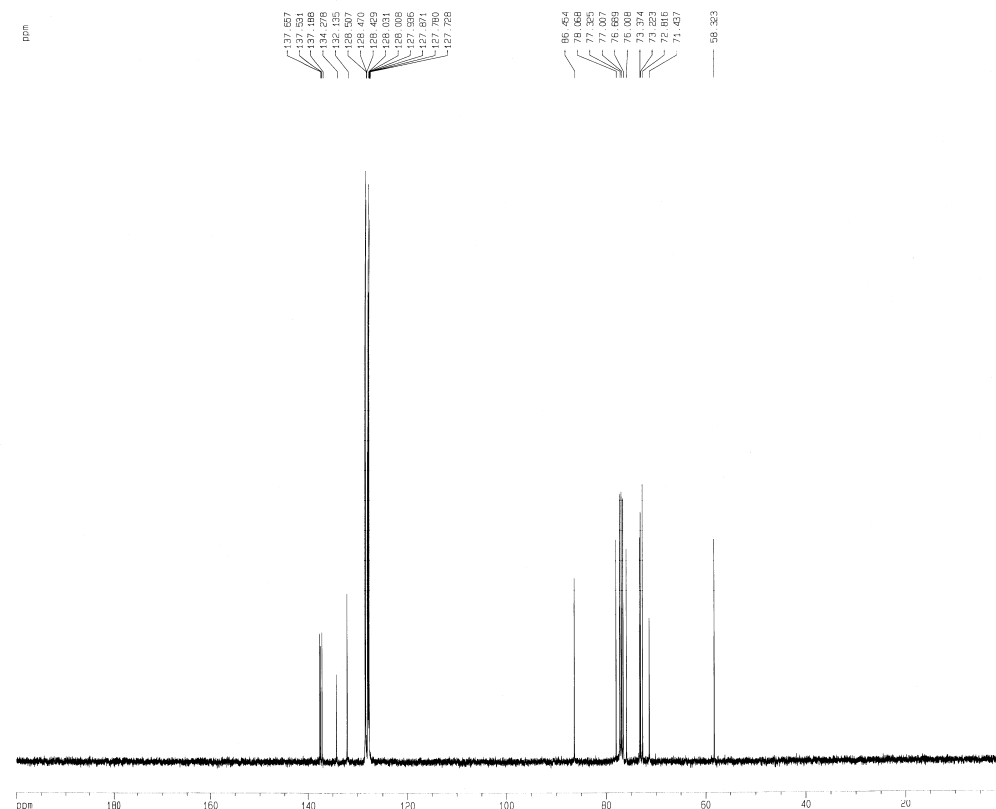
Current Data Parameters
NAME M9-less
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071006
Time 15.52
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 40.3
DM 60.400 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300330 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 17.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PRCM 0.29125 ppm/cm
HZCM 112.50845 Hz/cm



Current Data Parameters
NAME M9-less
EXPNO 2
PROCNO 1

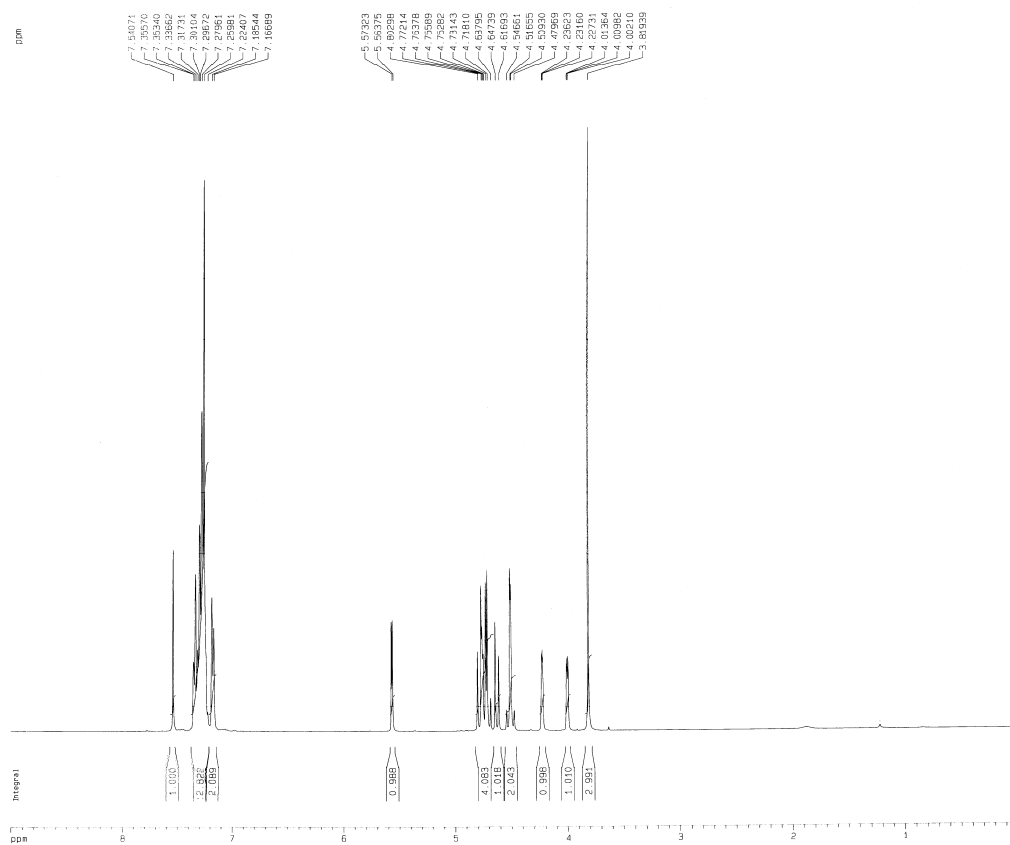
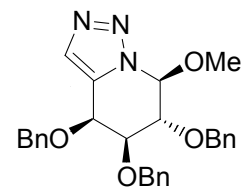
F2 - Acquisition Parameters
Date_ 20071006
Time 16.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 8192
DM 15.100 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89299998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

===== CHANNEL f2 =====
CHPROG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.6267034 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PRCM 6.25000 ppm/cm
HZCM 628.67273 Hz/cm



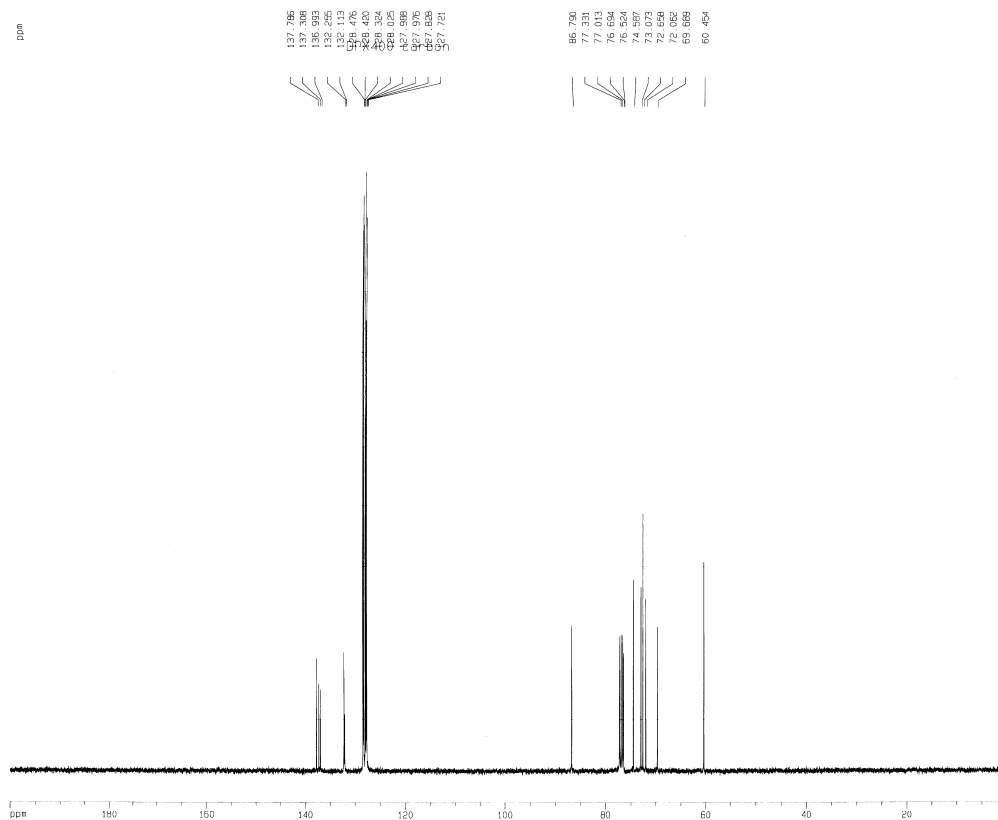
Current Data Parameters
 NAME M9-more
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071006
 Time 14.53
 INSTRUM spect
 PROUID 5 mm UNP 1H/13
 PULPROG zg30
 ID 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.3584243 sec
 RG 40.3
 DM 60.400 usec
 DE 6.00 usec
 TE 298.2 K
 O1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -4.00 dB
 SFO1 400.0324703 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0300345 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 CY 15.00 cm
 FIP 9.000 ppm
 F1 3600.27 Hz
 F2P 0.000 ppm
 F2 -0.00 Hz
 PRMCM 0.28125 ppm/cm
 HZCM 112.50845 Hz/cm



Current Data Parameters
 NAME M9-more
 EXPNO 2
 PROCNO 1

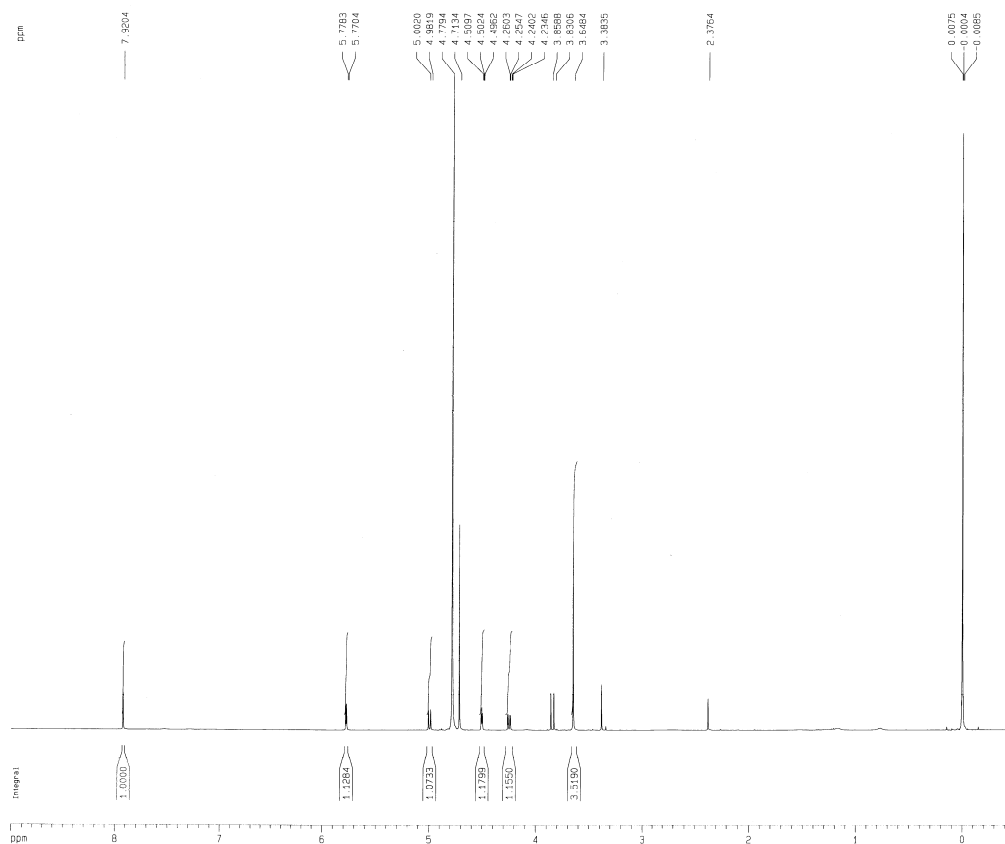
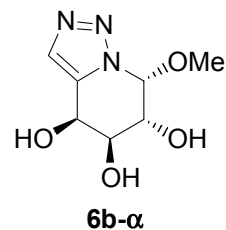
F2 - Acquisition Parameters
 Date_ 20071006
 Time 15.02
 INSTRUM spect
 PROUID 5 mm UNP 1H/13
 PULPROG zgpg30
 ID 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517875 sec
 RG 8192
 DM 14.100 usec
 DE 6.00 usec
 TE 298.2 K
 O1 2.00000000 sec
 O11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -3.50 dB
 SFO1 100.5906006 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 100.00 usec
 PL2 -4.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
 SFO2 400.0316001 MHz

F2 - Processing parameters
 SI 32768
 SF 100.5876362 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 32.00 cm
 CY 15.00 cm
 FIP 200.000 ppm
 F1 20117.53 Hz
 F2P -0.000 ppm
 F2 -0.00 Hz
 PRMCM 6.25000 ppm/cm
 HZCM 620.67273 Hz/cm



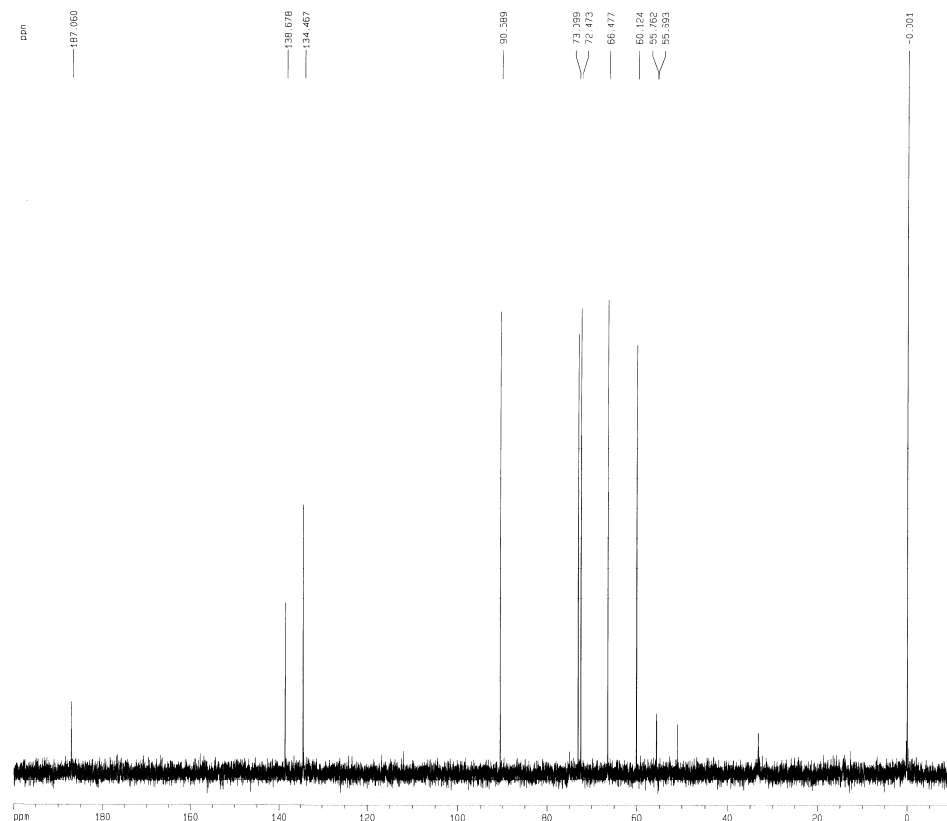
Current Data Parameters
NAME Man-OH-less
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071109
Time 20.12
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.148 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DM 60.400 usec
DE 5.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0299650 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 9.000 ppm
F1 3500.27 Hz
F2P -0.500 ppm
F2 -200.01 Hz
PMCM 0.29688 ppm/cm
HZCM 119.75890 Hz/cm



Current Data Parameters
NAME Man-OH-less
EXPNO 2
PROCNO 1

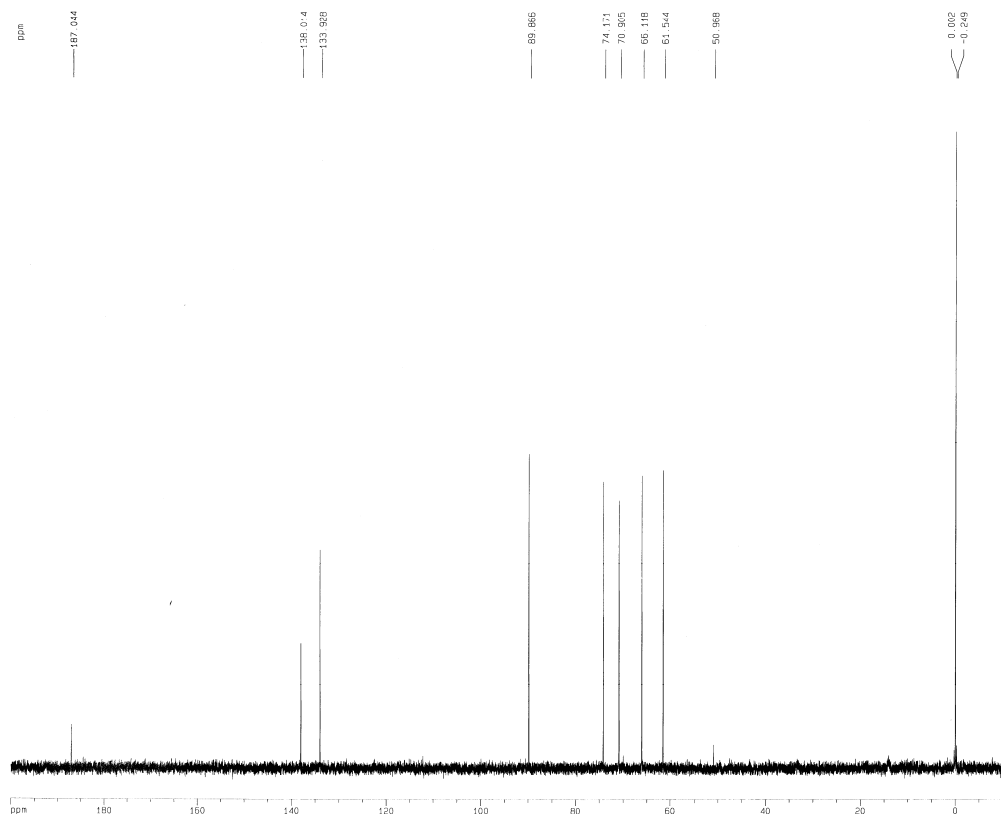
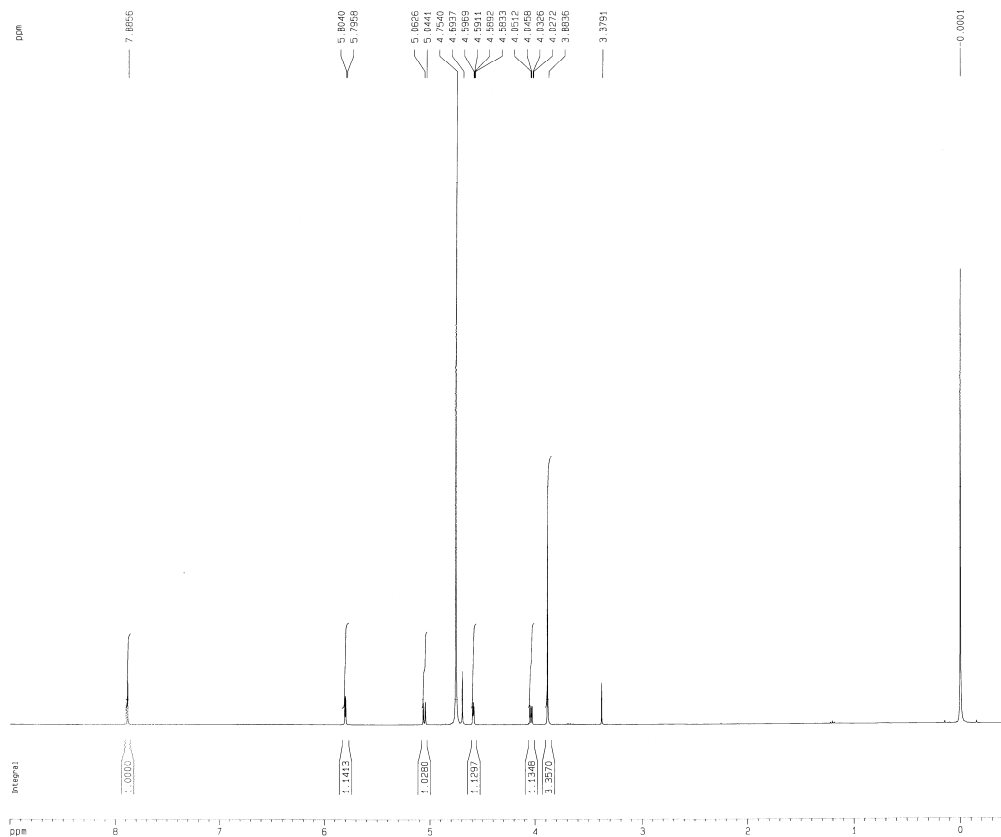
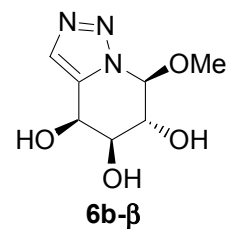
F2 - Acquisition Parameters
Date_ 20071109
Time 20.29
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 26178.010 Hz
FIDRES 0.389445 Hz
AQ 1.2317075 sec
RG 4096
DM 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999996 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.6281000 MHz

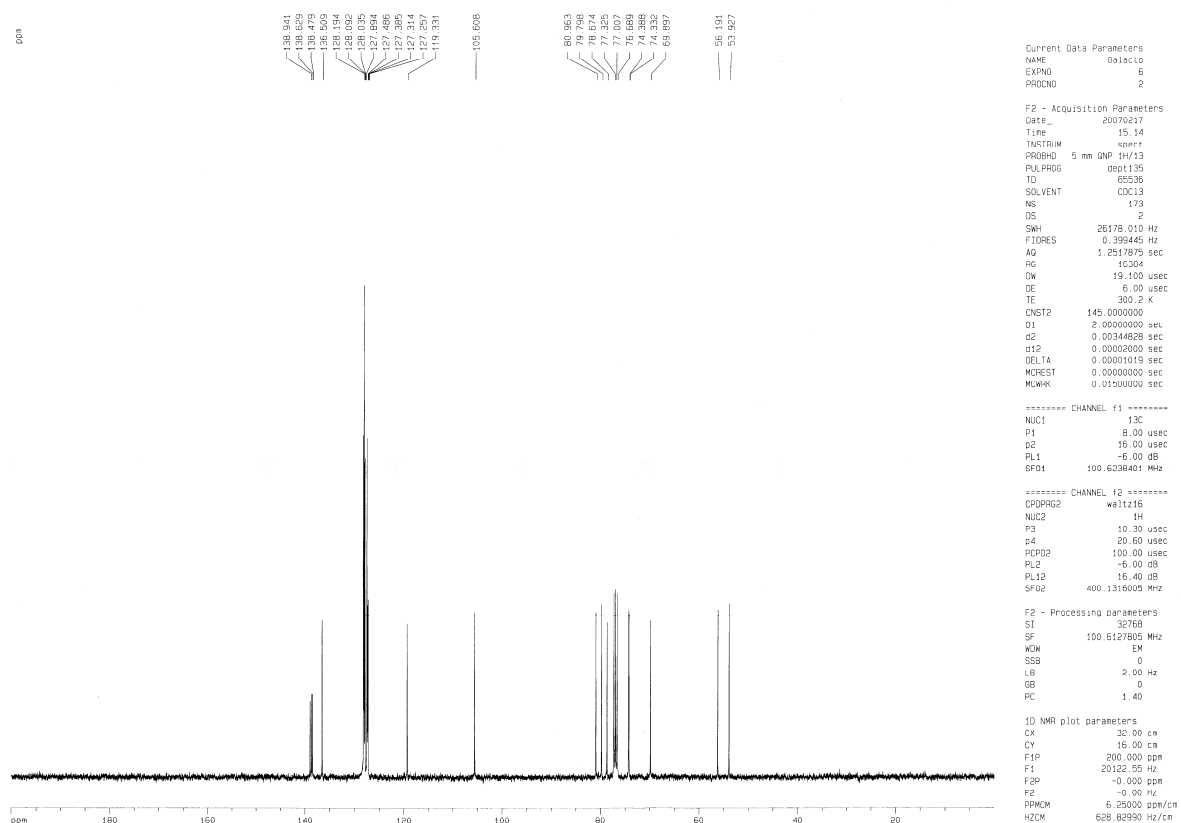
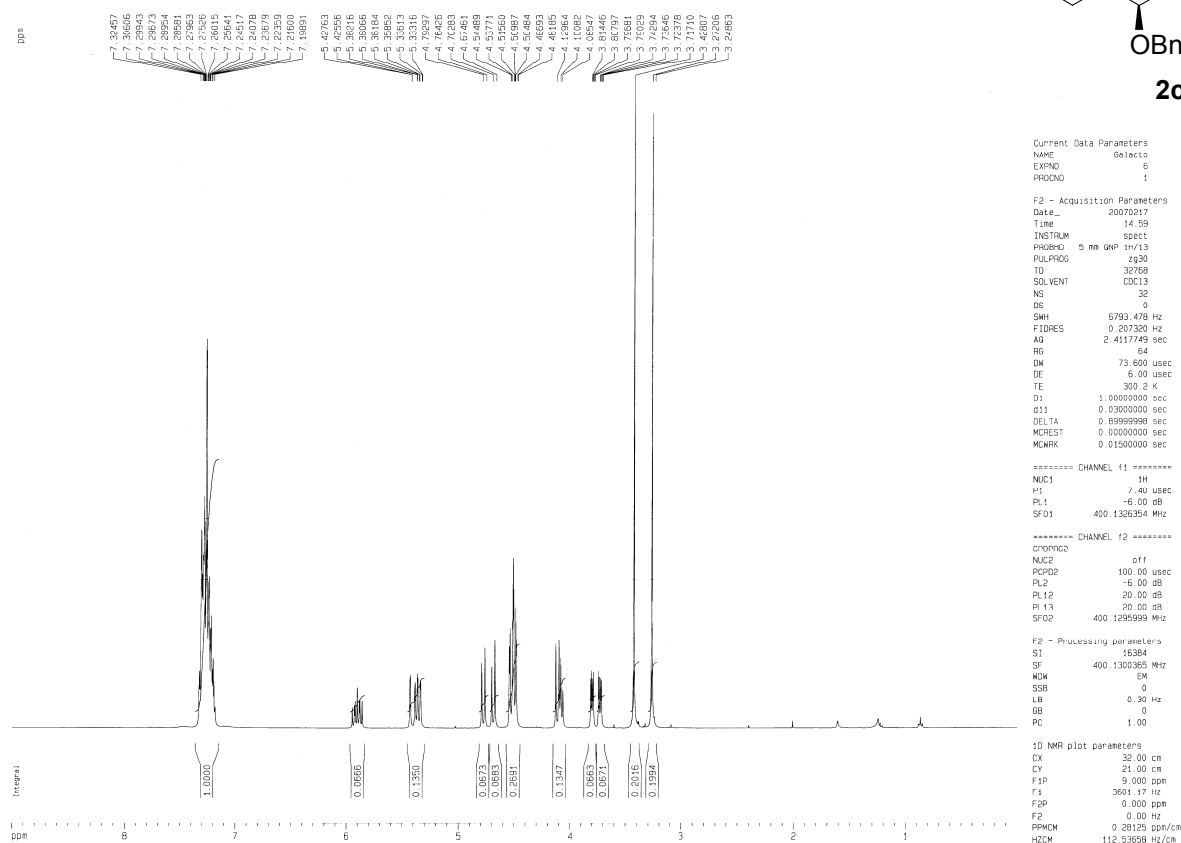
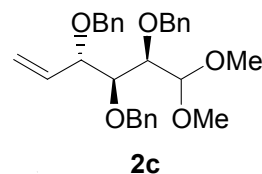
----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

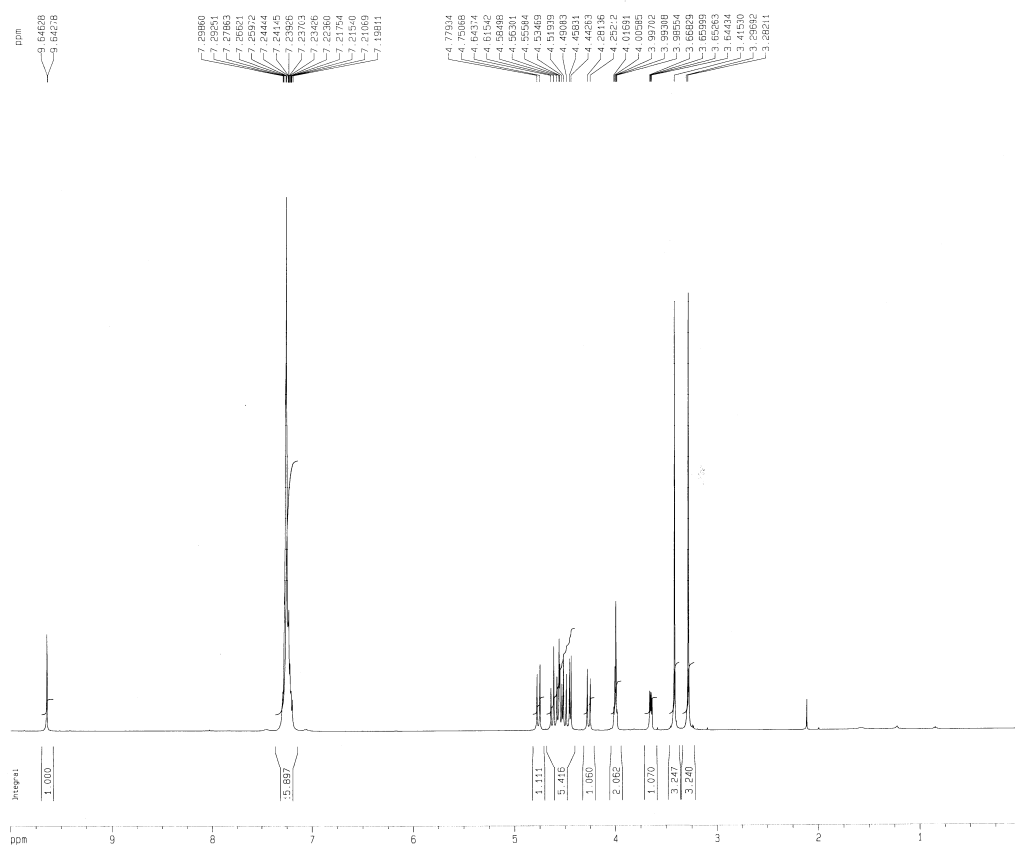
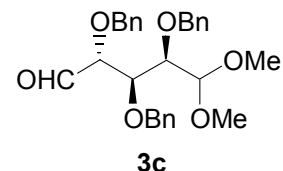
F2 - Processing parameters
SI 32768
SF 100.6274163 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 15.00 cm
F1P 200.000 ppm
F1 201.45 Hz
F2P -10.000 ppm
F2 -1005.87 Hz
PMCM 7.00000 ppm/cm
HZCM 704.11185 Hz/cm



8. ¹H and ¹³C NMR spectra of 2c, 3c, 4c, 5c and 6c





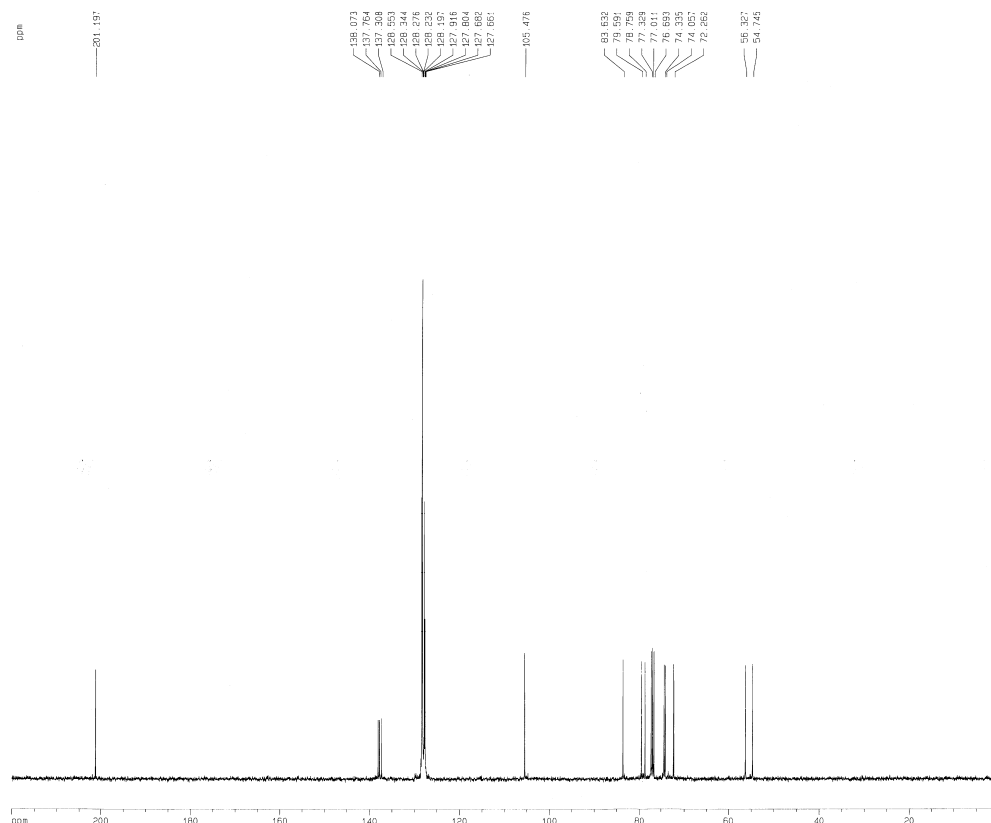
Current Data Parameters
NAME Galacto
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070302
Time 16.22
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 64
DK 73.600 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.40 usec
PL1 -6.00 dB
SFO1 400.1326354 MHz

F2 - Processing parameters
SI 16384
SF 400.1300336 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 17.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHMC 0.31250 ppm/cm
HZCM 125.04063 Hz/cm



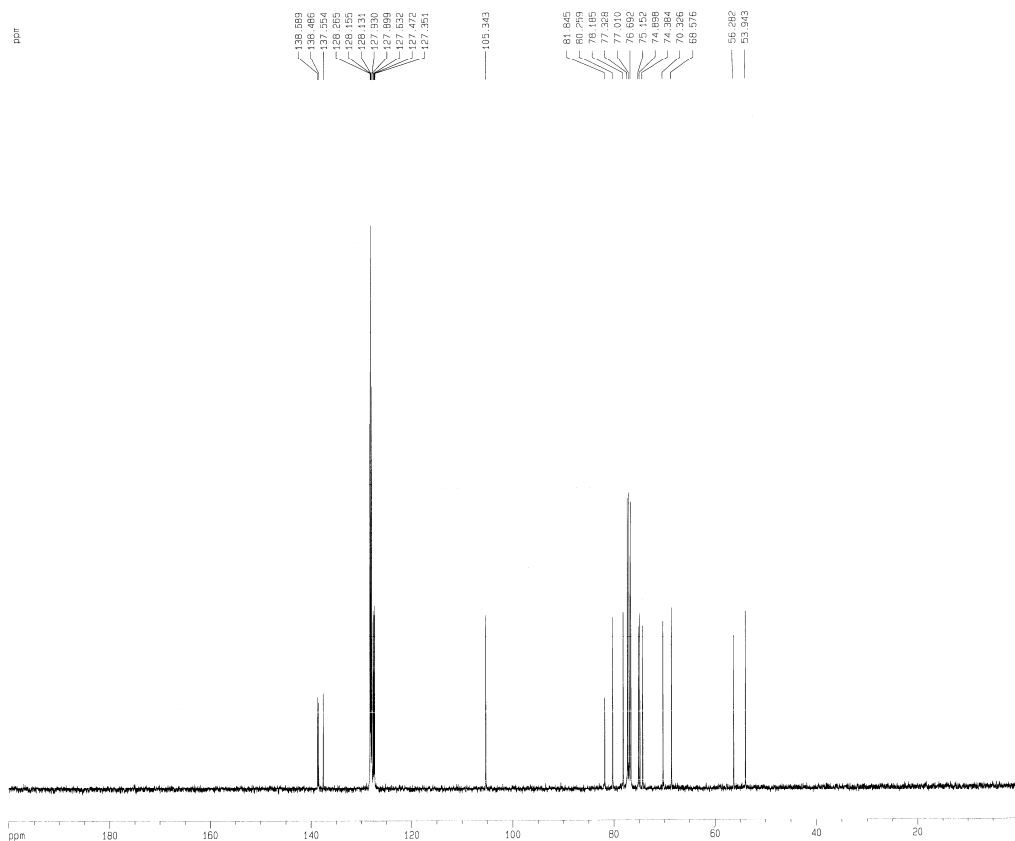
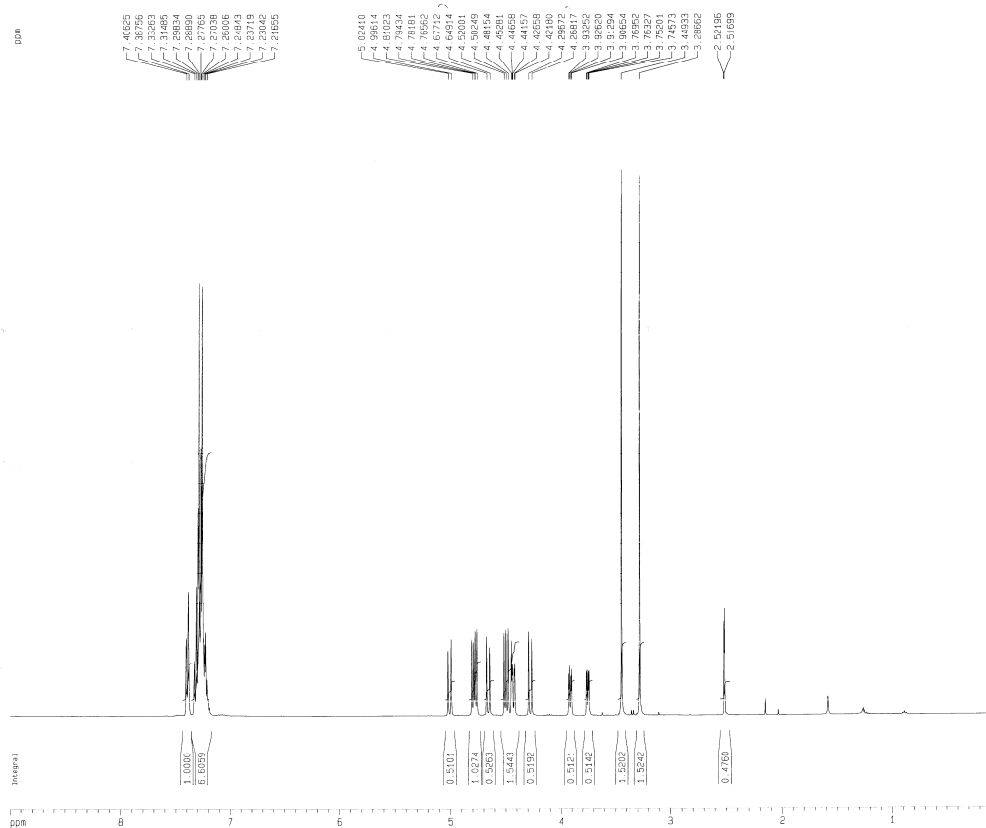
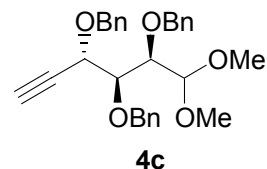
Current Data Parameters
NAME Galacto
EXPNO 7
PROCNO 2

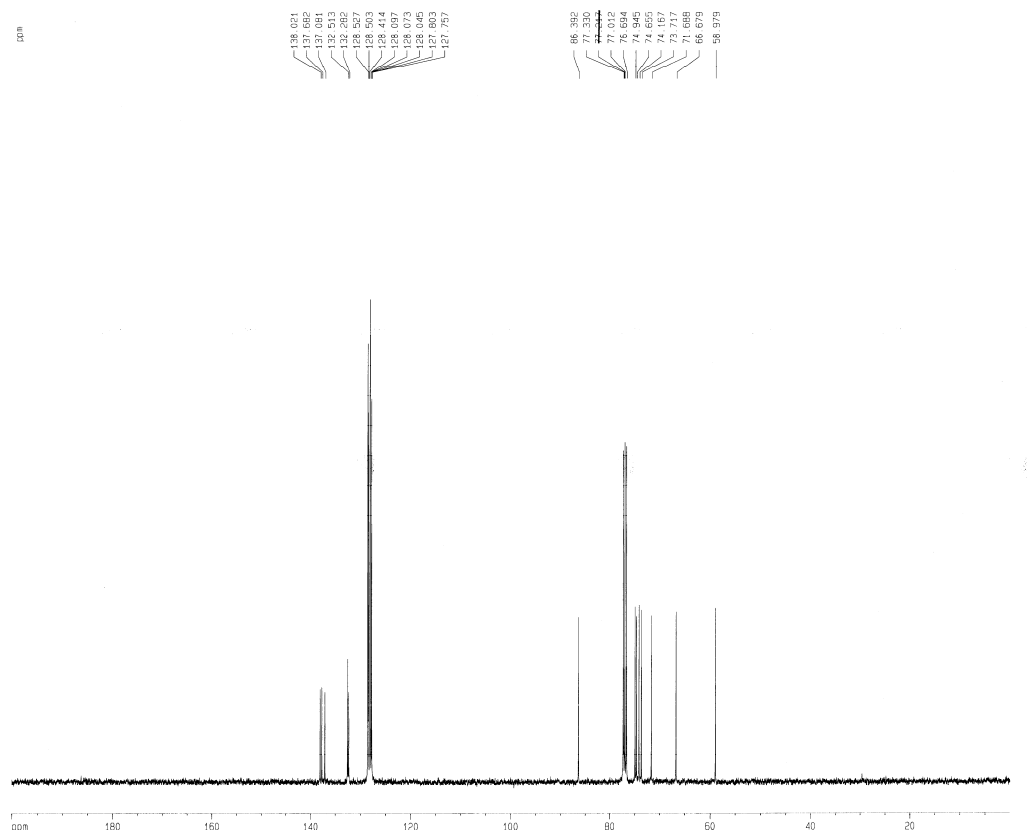
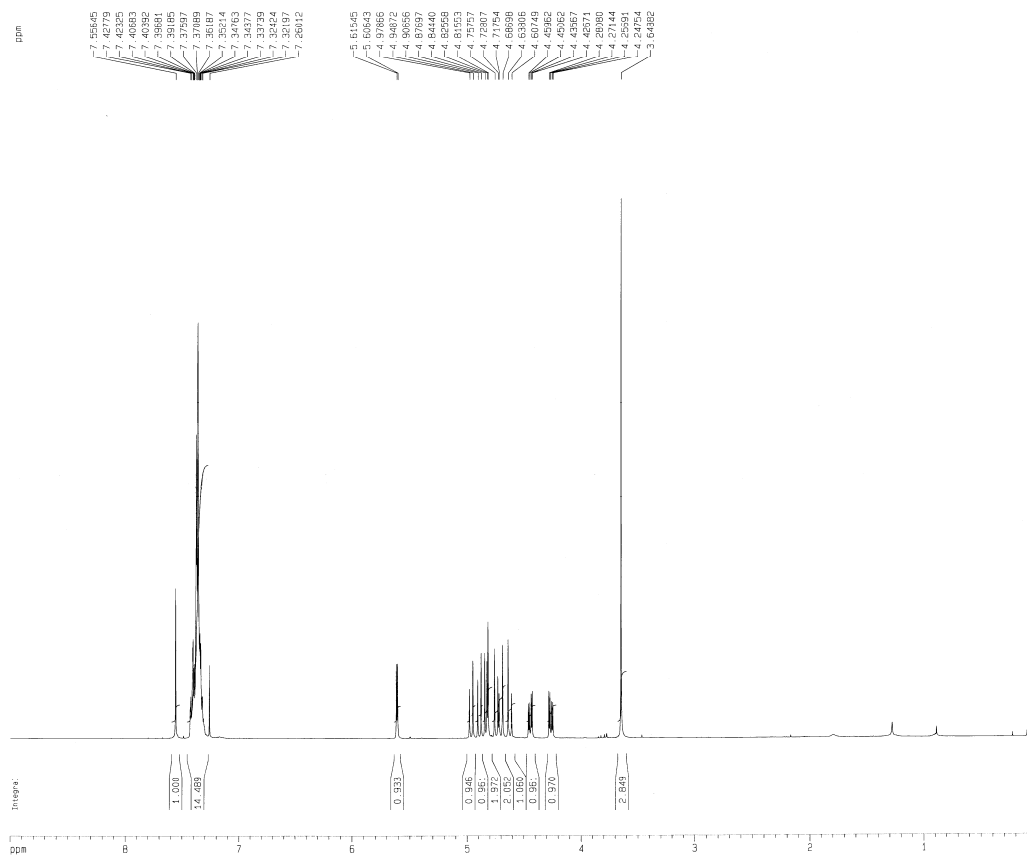
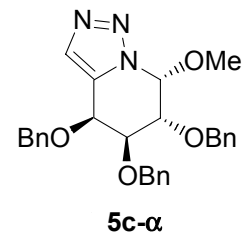
F2 - Acquisition Parameters
Date_ 20070221
Time 17.00
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 67
DK 73.600 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

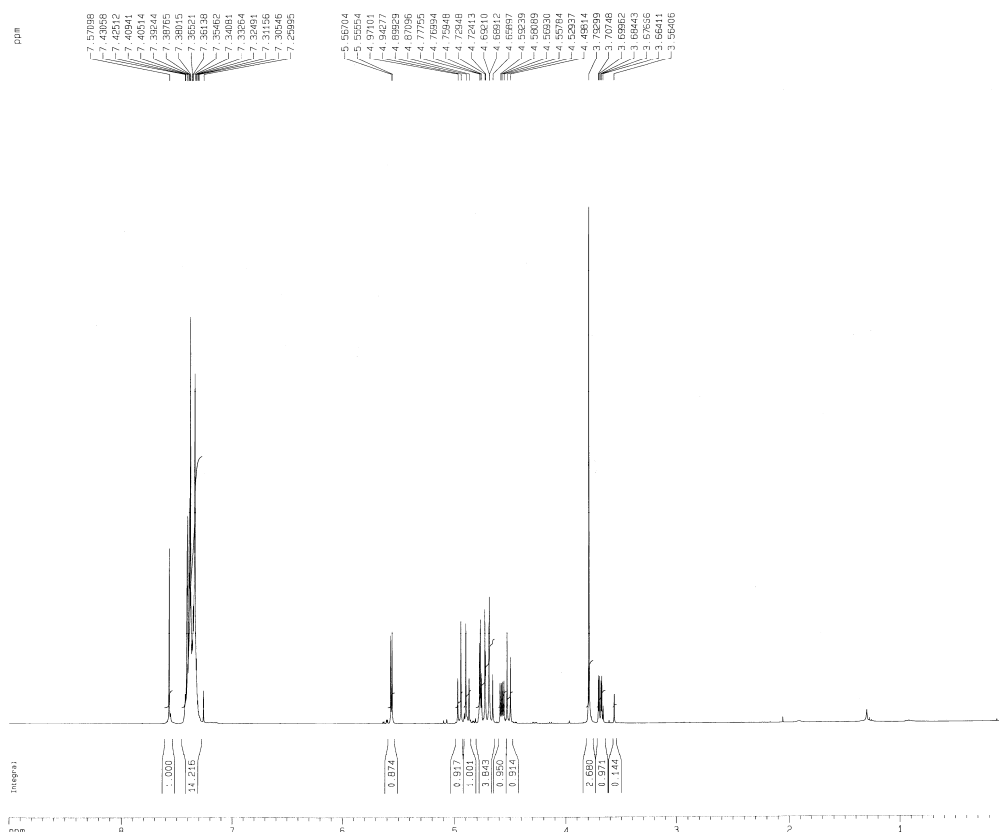
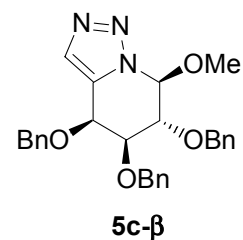
===== CHANNEL f1 =====
NUC1 1H
P1 7.40 usec
PL1 -6.00 dB
SFO1 400.1326354 MHz

F2 - Processing parameters
SI 100.6127813 MHz
SF 100.6127813 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 16.00 cm
F1P 220.000 ppm
F1 22134.61 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHMC 6.87500 ppm/cm
HZCM 691.71289 Hz/cm







```

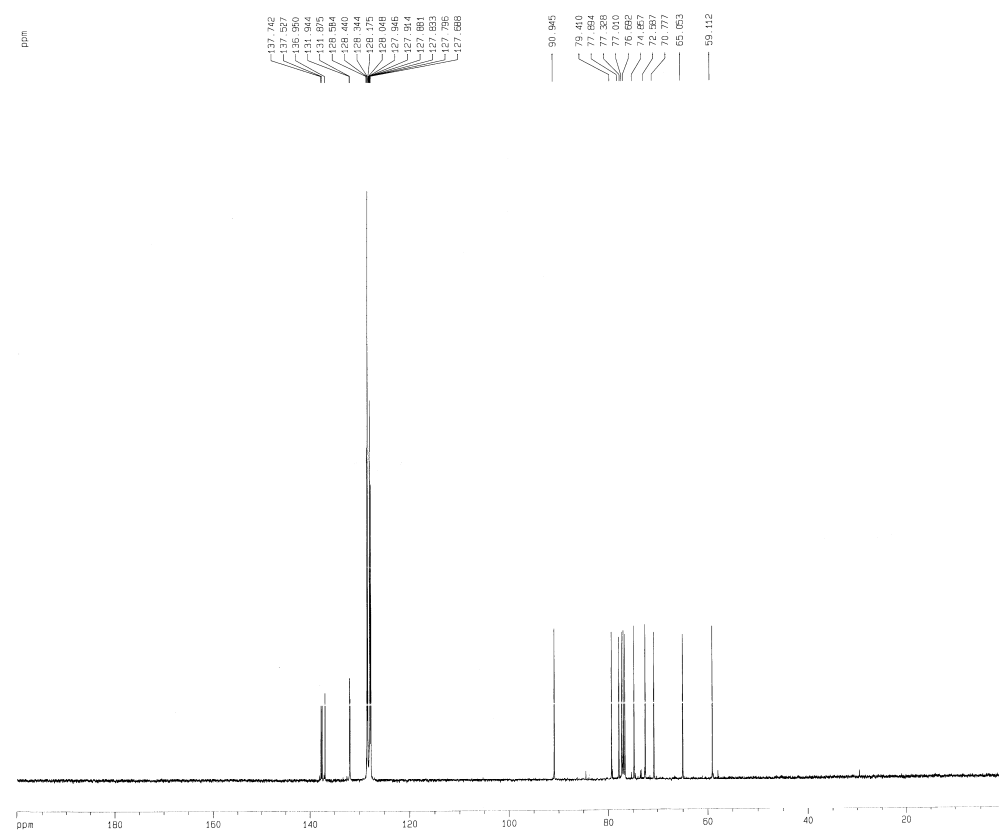
Current Data Parameters
NAME          Ga195
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_        20070324
Time         13.35
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zg30
TD           32768
SOLVENT      CDCl3
NS           16
DS           0
SWH          6793.476 Hz
FIDRES      0.207320 Hz
AQ          2.4117249 sec
RG          40.3
DW          73.600 usec
DE          6.00 usec
TE          300.2 K
D1          1.0000000 sec
MCREST      0.0000000 sec
MCWK       0.0150000 sec

===== CHANNEL f1 =====
NUC1         1H
P1           7.40 usec
PL1         -6.00 dB
SFO1        400.1326394 MHz

F2 - Processing parameters
SI           65384
SF          400.1300991 MHz
WDW          EM
SSB          0
LB          0.30 Hz
GB          0
PC          1.00

1D NMR plot parameters
CX          32.00 cm
CY          17.00 cm
F1P        9.000 ppm
F1         3601.17 Hz
F2P        0.000 ppm
F2         0.00 Hz
PPMCM      0.28125 ppm/cm
HZCM       112.53656 Hz/cm
    
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```

Current Data Parameters
NAME          Ga195
EXPNO        2
PROCNO       1

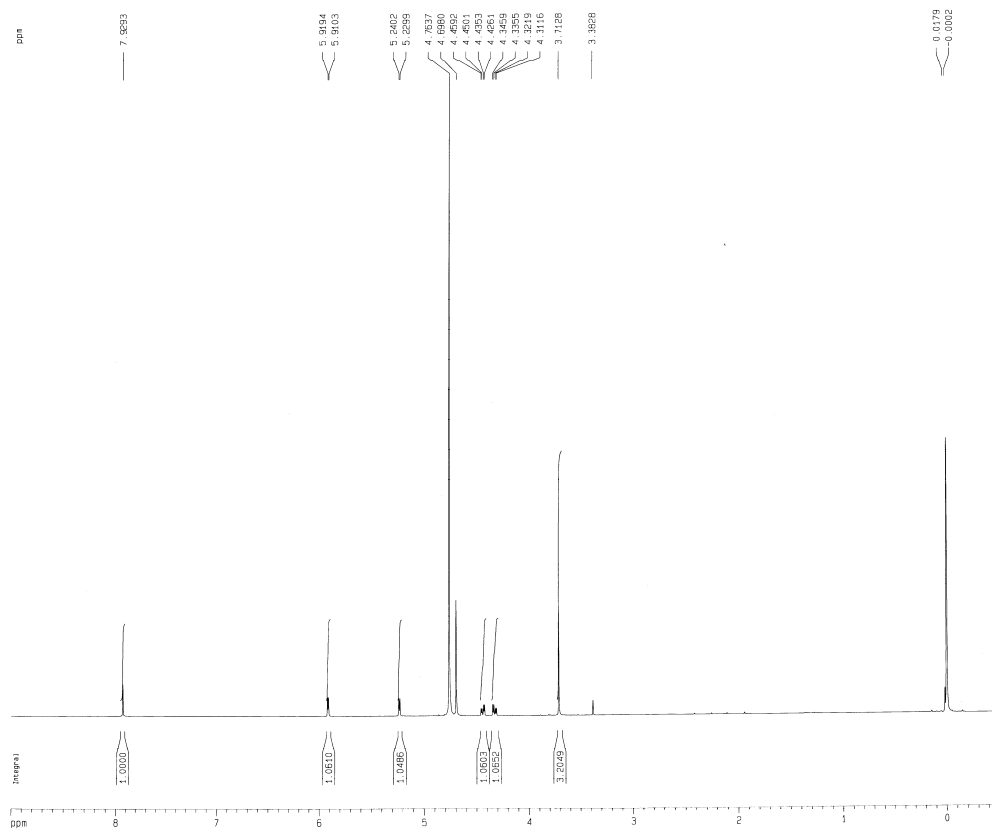
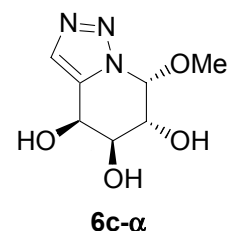
F2 - Acquisition Parameters
Date_        20070324
Time         23.43
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           256
DS           2
SWH          26178.010 Hz
FIDRES      0.398445 Hz
AQ          1.2517875 sec
RG          4997.6
DW          19.100 usec
DE          6.00 usec
TE          300.2 K
D1          2.0000000 sec
d11         0.0300000 sec
DELTA       1.8995999 sec
MCREST      0.0000000 sec
MCWK       0.0150000 sec

===== CHANNEL f1 =====
NUC1         13C
P1           8.00 usec
PL1         -6.00 dB
SFO1        100.6238401 MHz

===== CHANNEL f2 =====
CPDPRG2     waltz16
NUC2         1H
PCPD2       100.00 usec
PL2         -6.00 dB
PL12        16.40 dB
PL13        16.40 dB
SFO2        400.1316076 MHz

F2 - Processing parameters
SI           32768
SF          100.6127861 MHz
WDW          EM
SSB          0
LB          2.00 Hz
GB          0
PC          1.40

1D NMR plot parameters
CX          32.00 cm
CY          19.00 cm
F1P        200.000 ppm
F1         20122.56 Hz
F2P        -0.000 ppm
F2         -0.00 Hz
PPMCM      5.25000 ppm/cm
HZCM       628.82990 Hz/cm
    
```



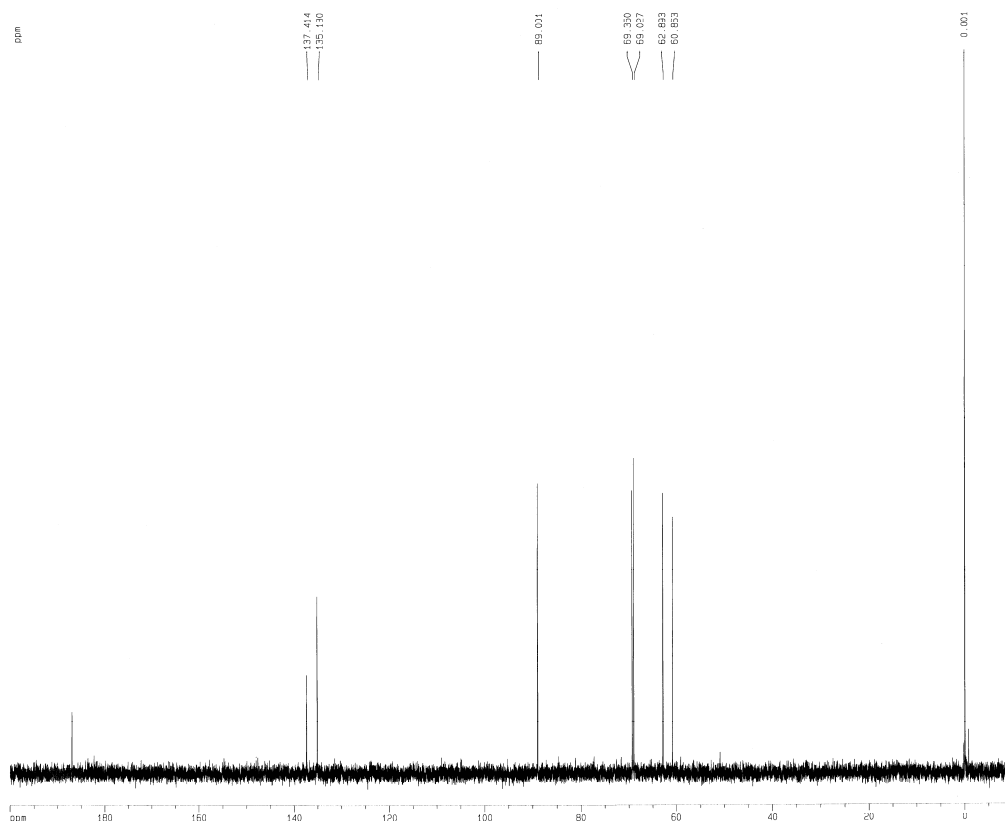
Current Data Parameters
 NAME Gal-OH-less
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071102
 Time 14.51
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8078.145 Hz
 FIDRES 0.186314 Hz
 AQ 3.9584243 sec
 RG 90.5
 DW 60.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCMK 0.01500000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 11.50 usec
 PL1 -4.00 dB
 SFO1 400.0324703 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0299726 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 CY 21.00 cm
 F1P 9.000 ppm
 F1 3600.27 Hz
 F2P -0.500 ppm
 F2 -200.01 Hz
 PPMCM 0.2968 ppm/cm
 HZCM 116.75890 Hz/cm



Current Data Parameters
 NAME Gal-OH-less
 EXPNO 1
 PROCNO 1

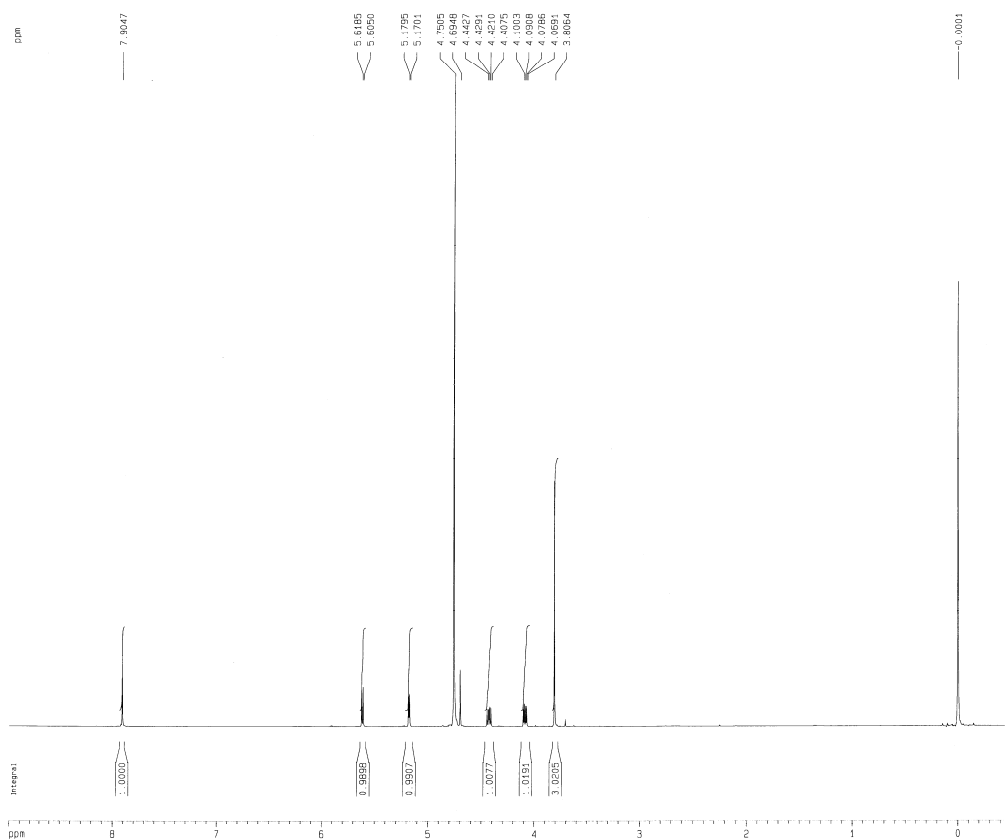
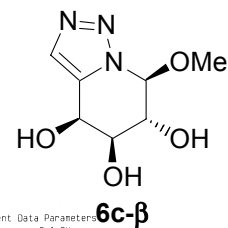
F2 - Acquisition Parameters
 Date_ 20071102
 Time 15.01
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 330
 DS 4
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517075 sec
 RG 3643.1
 DM 19.100 usec
 DE 6.00 usec
 TE 300.2 K
 D1 0.00000000 sec
 D11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCMK 0.01500000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 10.00 usec
 PL1 -3.50 dB
 SFO1 100.6261800 MHz

----- CHANNEL f2 -----
 NUC2 1H
 P1P1P 100.00 usec
 PL2 -4.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
 SFO2 400.0316001 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6274293 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 32.00 cm
 CY 10.00 cm
 F1P 200.000 ppm
 F1 20117.48 Hz
 F2P -10.000 ppm
 F2 -1005.87 Hz
 PPMCM 6.56250 ppm/cm
 HZCM 660.10492 Hz/cm



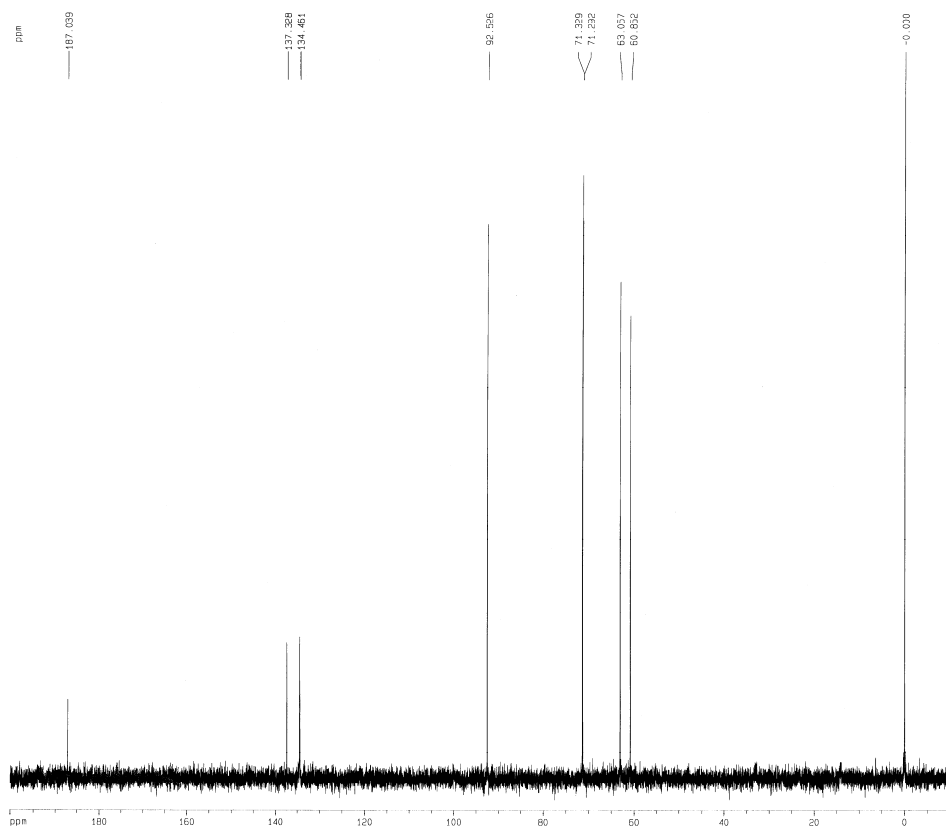
Current Data Parameters
 NAME Gal-DH-more
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071031
 Time 16.57
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 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TO 65336
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 65
 DW 60.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 11.50 usec
 PL1 -4.00 dB
 SFO1 400.0324703 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0299769 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 CY 21.00 cm
 F1P 9.000 ppm
 F1 3600.27 Hz
 F2P -0.500 ppm
 F2 -200.01 Hz
 FWHM 0.29688 ppm/cm
 HZCM 118.75889 Hz/cm



Current Data Parameters
 NAME Gal-DH-more
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071031
 Time 17.07
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TO 65336
 SOLVENT CDCl3
 NS 279
 DS 4
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517875 sec
 RG 8192
 DW 19.100 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

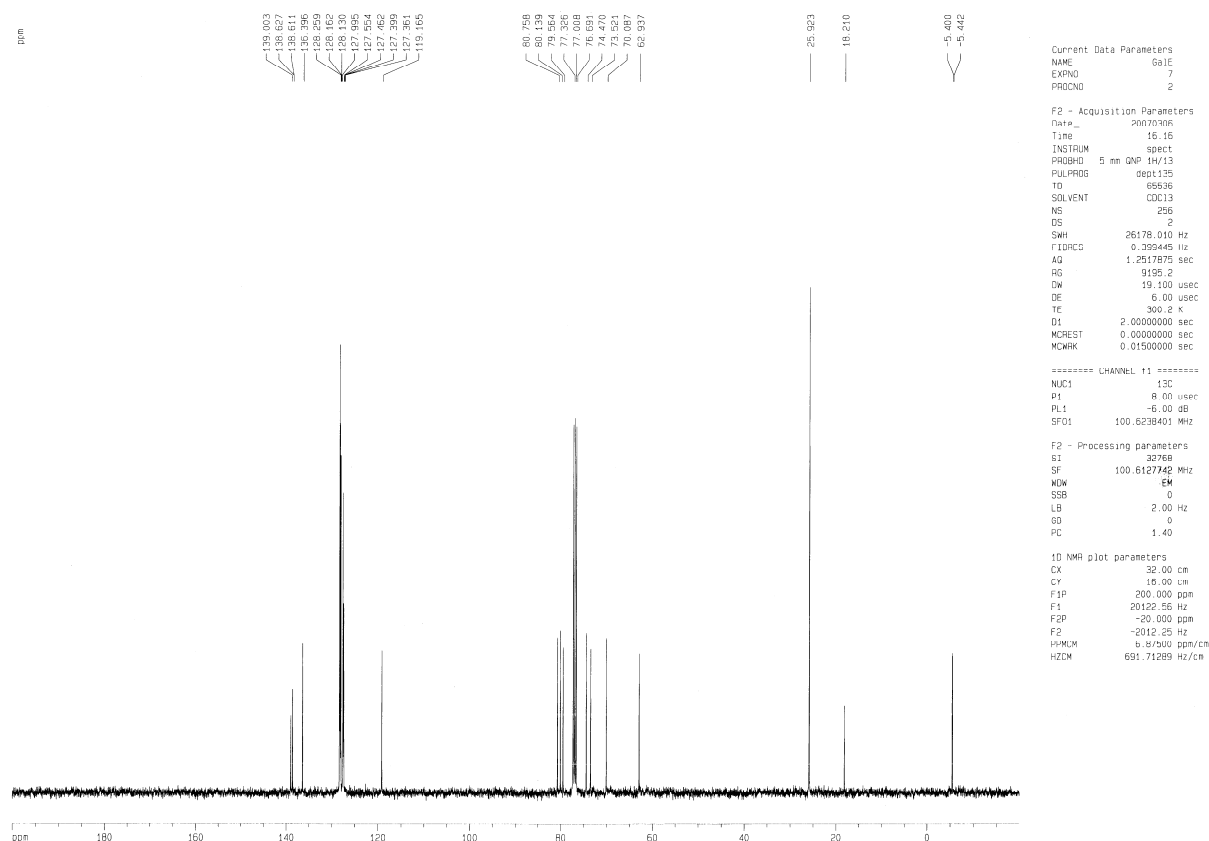
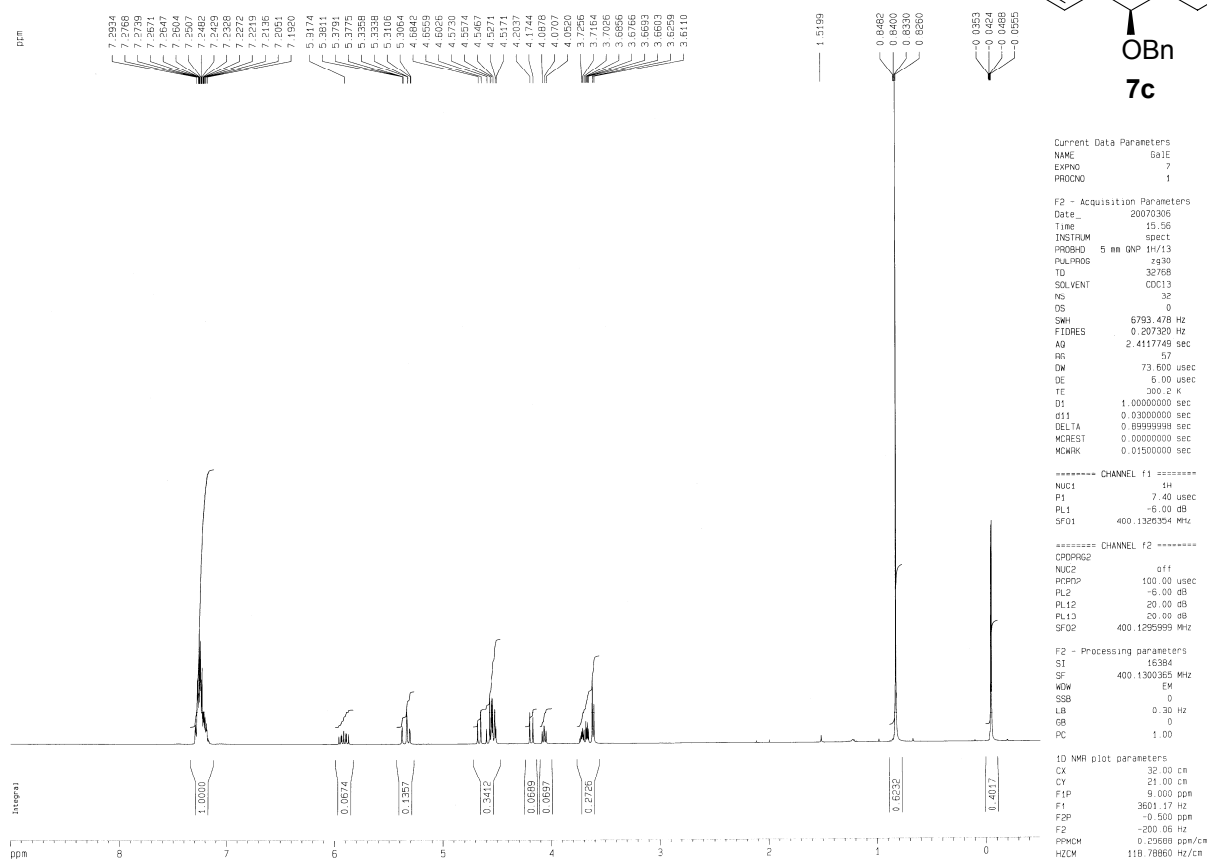
----- CHANNEL f1 -----
 NUC1 13C
 P1 10.00 usec
 PL1 -3.50 dB
 SFO1 100.5986886 MHz

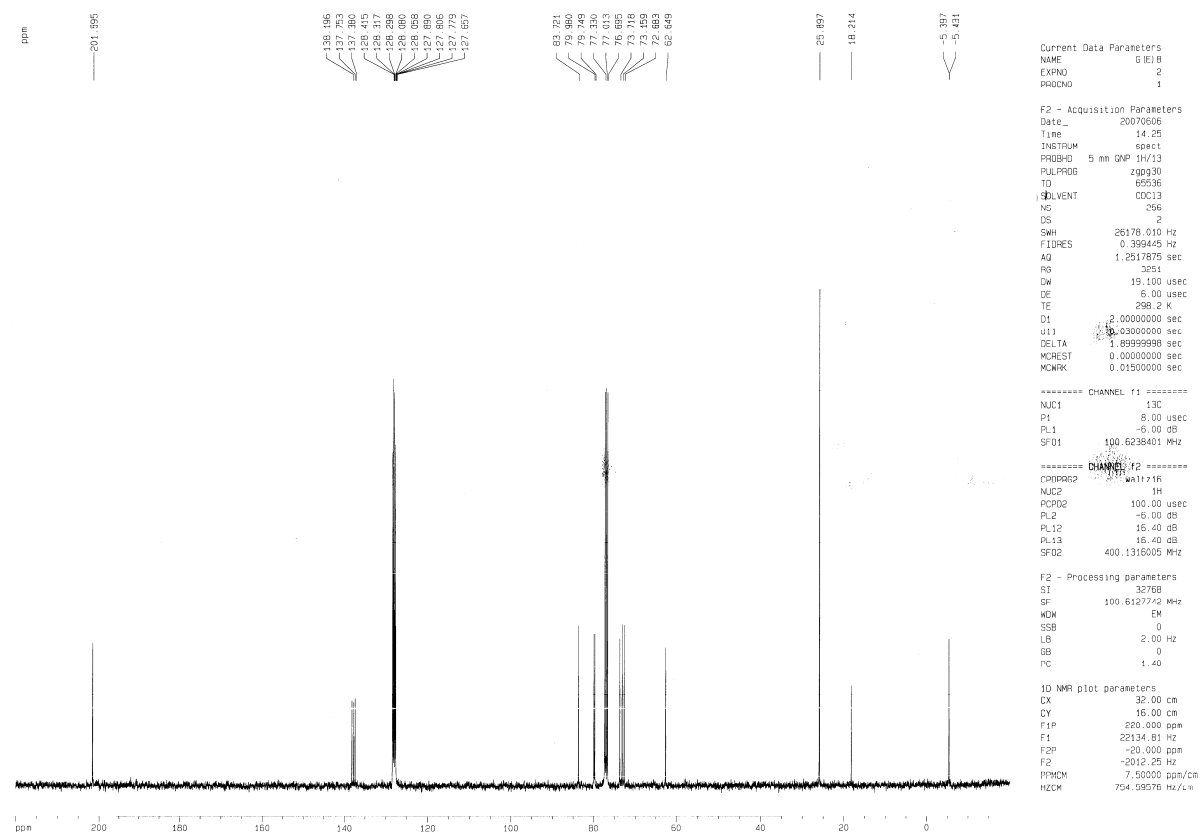
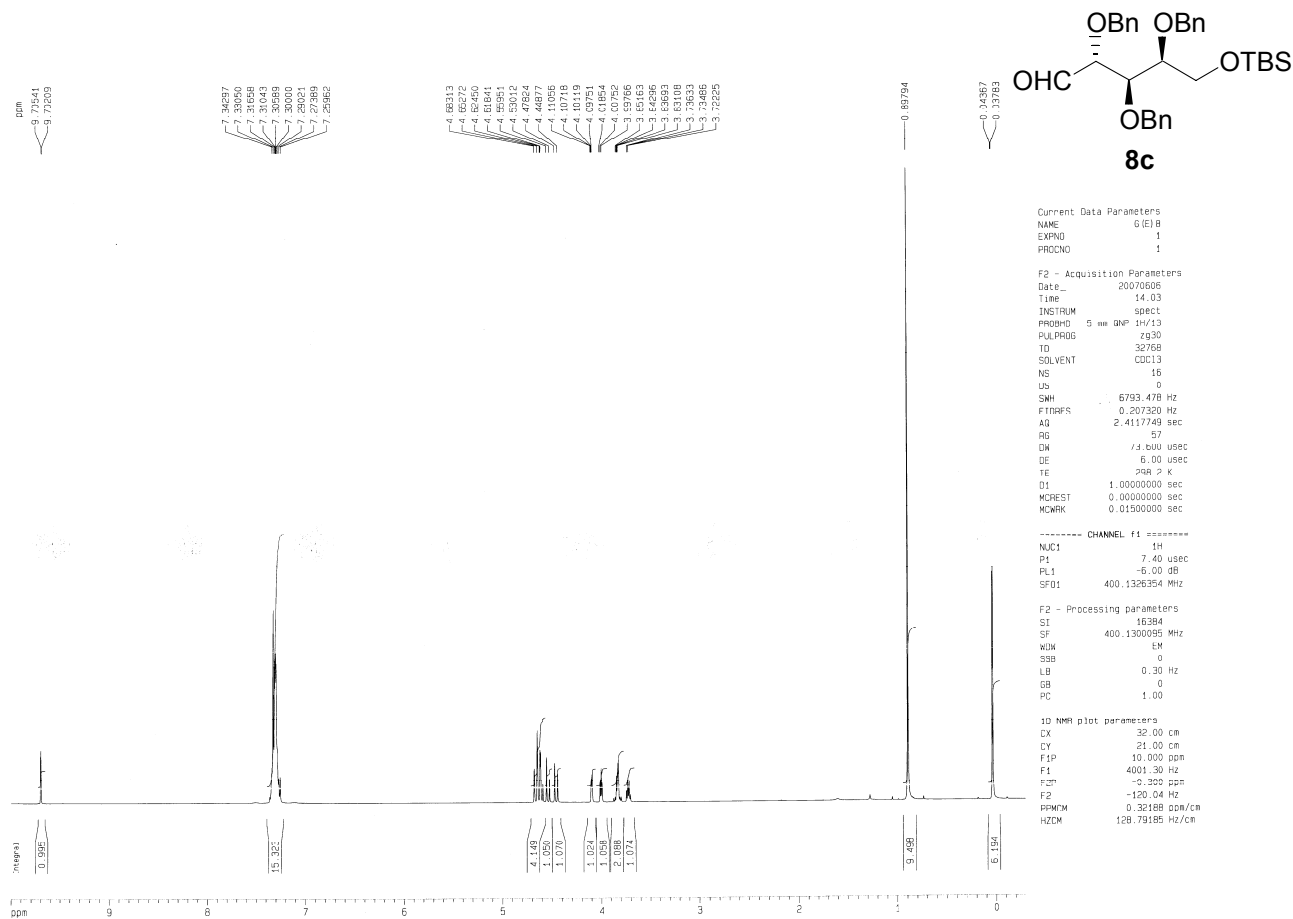
----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -4.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
 SFO2 400.0316001 MHz

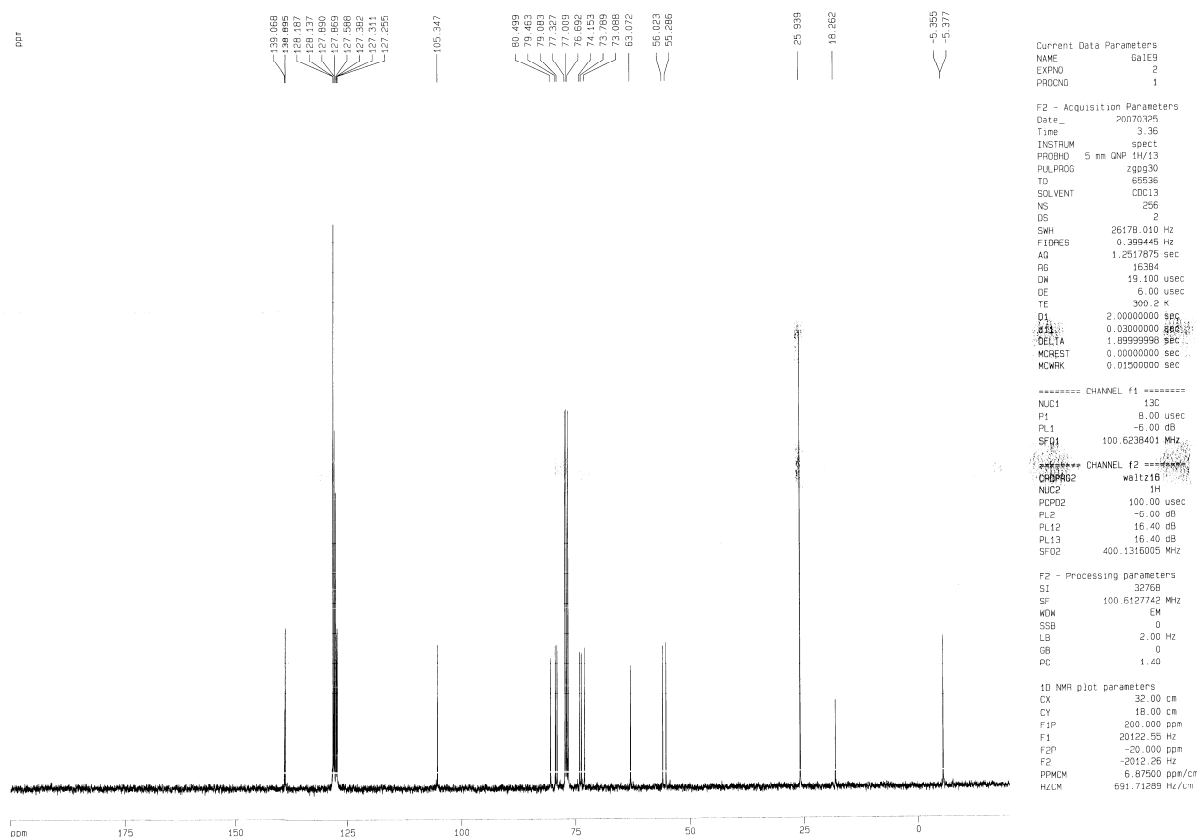
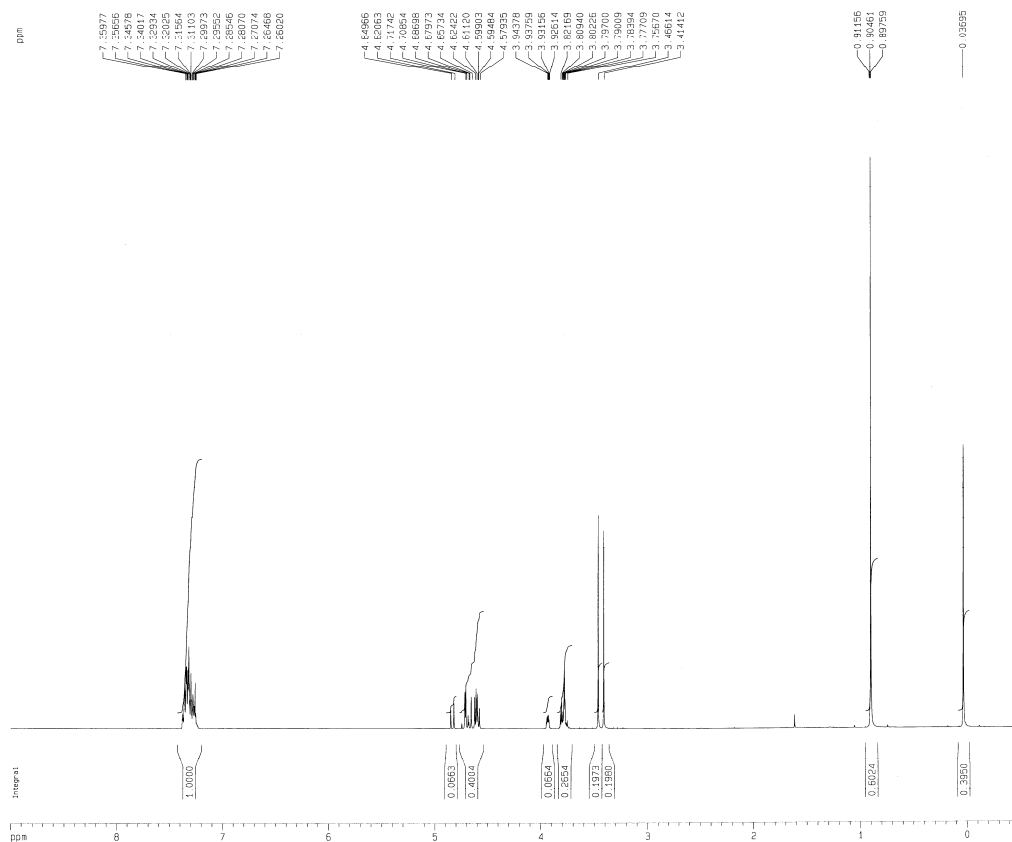
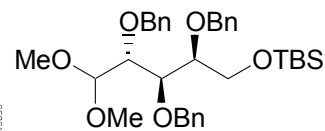
F2 - Processing parameters
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 SF 100.5874203 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

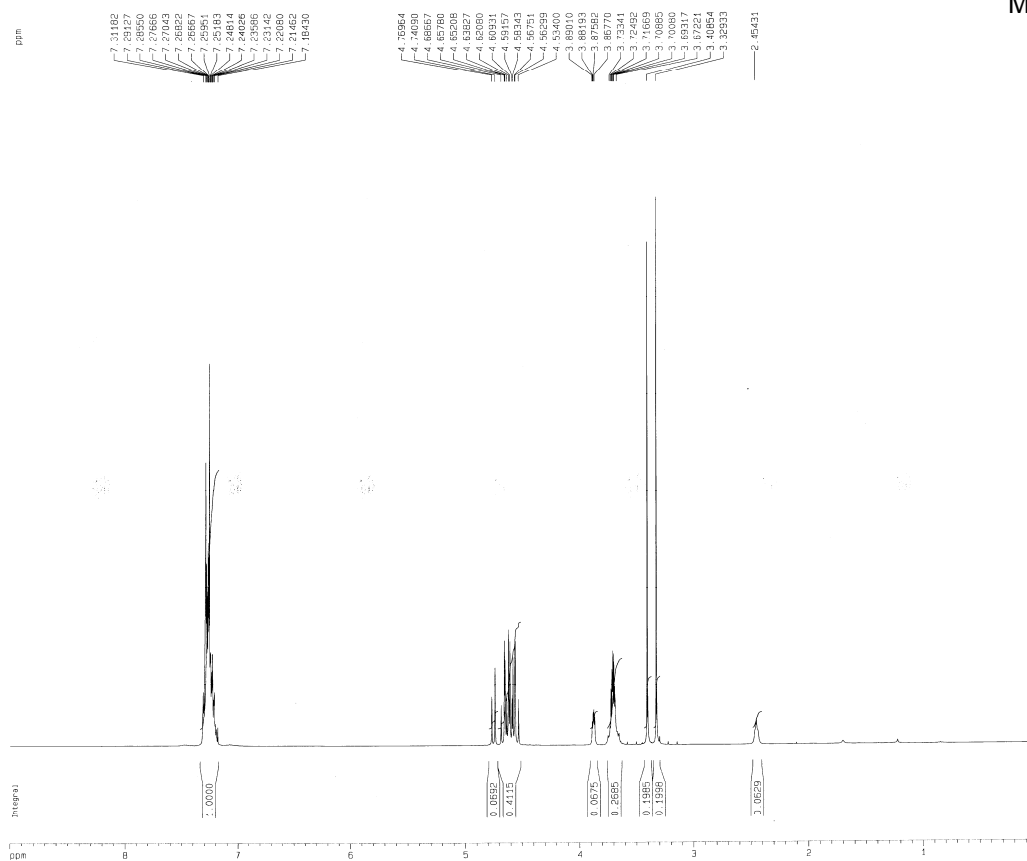
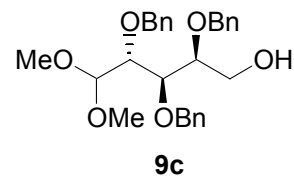
1D NMR plot parameters
 CX 30.00 cm
 CY 19.00 cm
 F1P 200.000 ppm
 F1 20117.48 Hz
 F2P -10.000 ppm
 F2 -1005.87 Hz
 FWHM 7.50000 ppm/cm
 HZCM 704.11188 Hz/cm

9. ¹H and ¹³C NMR spectra of **7c**, **8c** and **9c**









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Current Data Parameters
NAME          GaIE
EXPNO        10
PROCNO       1

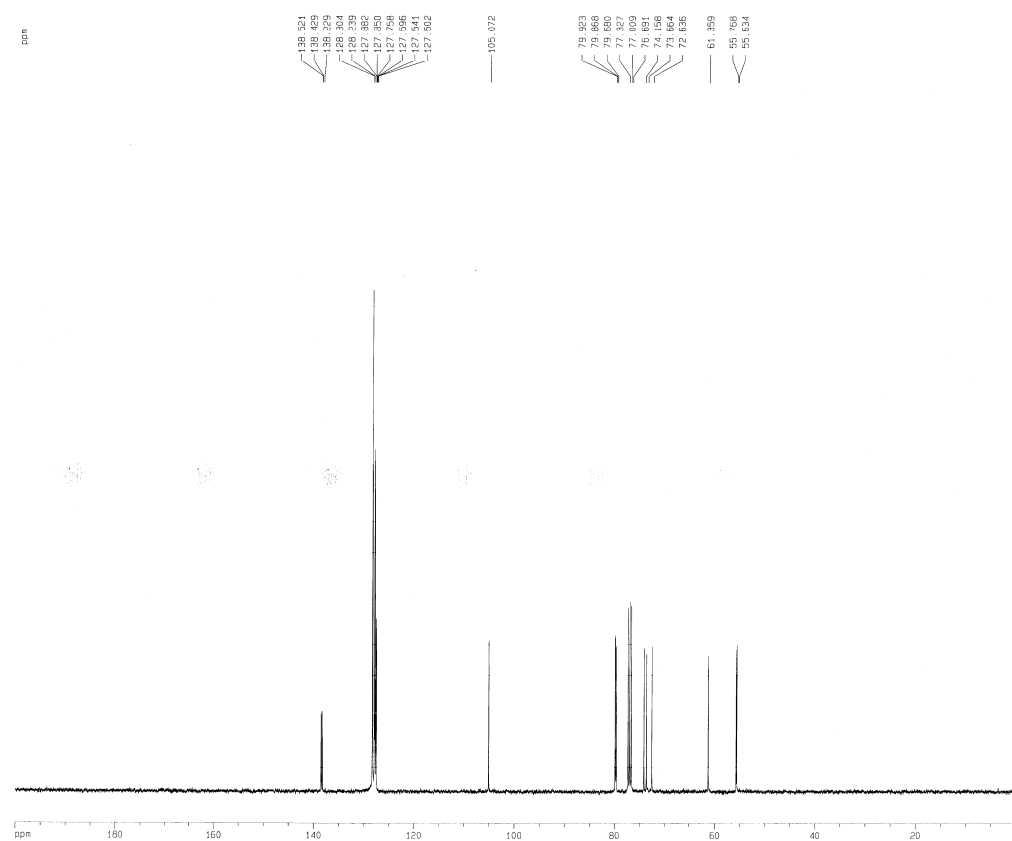
F2 - Acquisition Parameters
Date_        20070323
Time         1.39
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zg30
TD           32768
SOLVENT      CDCl3
NS           16
DS           0
SMH          6793.478 Hz
FIDRES       0.237300 Hz
AQ           2.417749 sec
RG           64
DW           73.600 usec
DE           6.00 usec
TE           300.2 K
D1           1.00000000 sec
d11          0.03000000 sec
DELTA        0.09899999 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

===== CHANNEL f1 =====
NUC1         1H
P1           7.40 usec
PL1         -6.00 dB
SFO1        400.1363654 MHz

===== CHANNEL f2 =====
UNPRG2
NUC2         off
PCPD2       100.00 usec
PL2         -6.00 dB
PL12        20.30 dB
PL13        20.00 dB
SFO2        400.1259999 MHz

F2 - Processing parameters
SI           16384
SF           400.1300394 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00

1D NMR plot parameters
CX           32.00 cm
CY           17.00 cm
F1P          6.000 ppm
F1           3601.17 Hz
F2P          0.000 ppm
F2           0.00 Hz
RPMCM        0.2912 ppm/cm
HZCM         112.53658 Hz/cm
    
```



```

Current Data Parameters
NAME          GaIE
EXPNO        10
PROCNO       2

F2 - Acquisition Parameters
Date_        20070323
Time         2.13
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      dept135
TD           65536
SOLVENT      CDCl3
NS           256
DS           2
SMH          26178.010 Hz
FIDRES       0.399445 Hz
AQ           1.2517875 sec
RG           16384
DW           19.100 usec
DE           6.00 usec
TE           300.2 K
CNST2       145.0000000 sec
d2           2.00000000 sec
d21          0.00344828 sec
d12          0.00002000 sec
DELTA        0.0001919 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

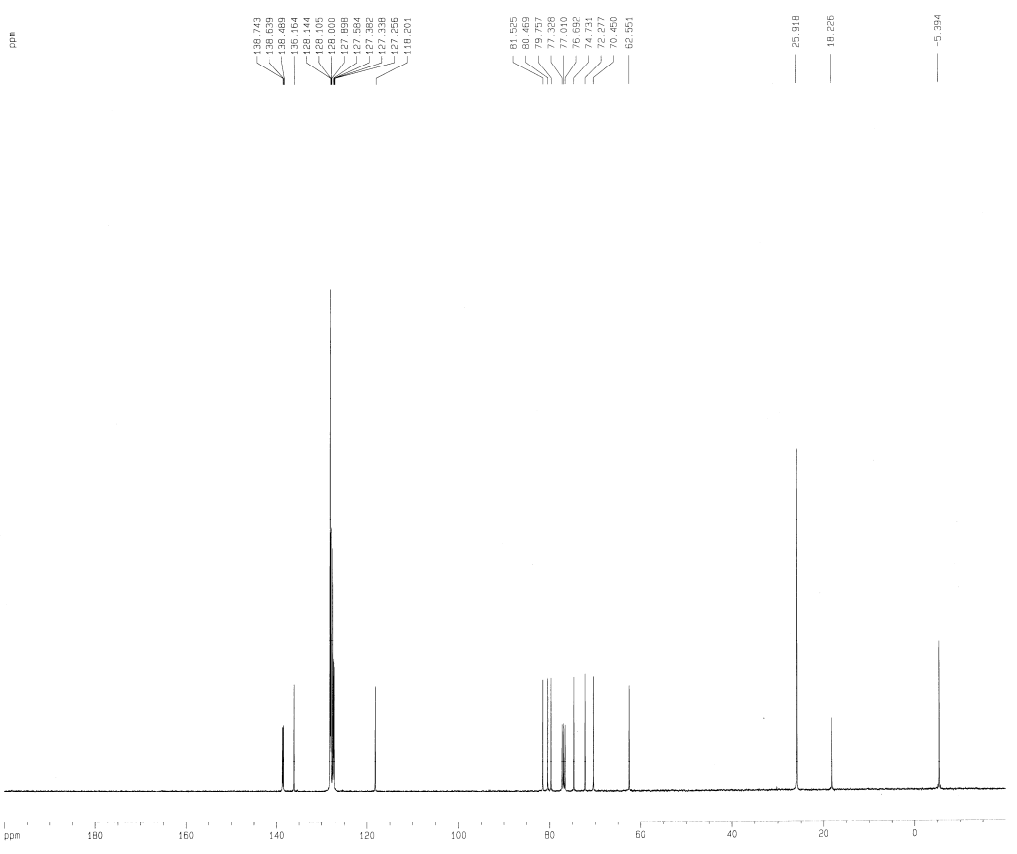
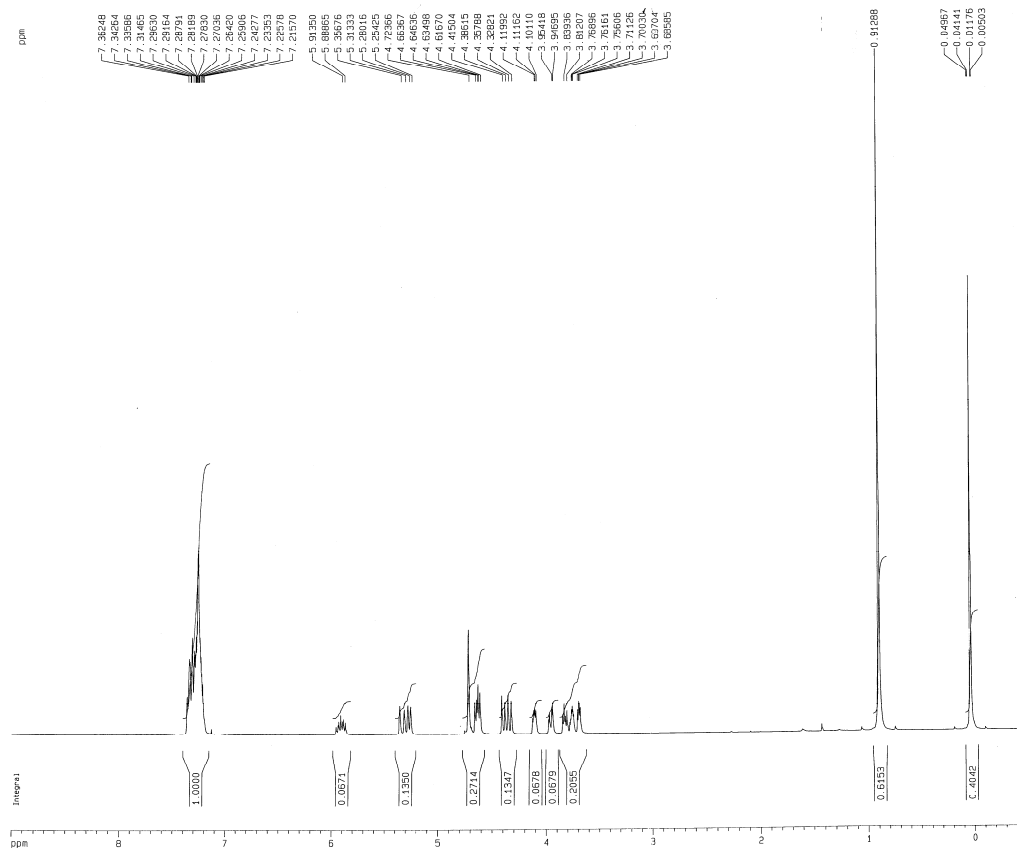
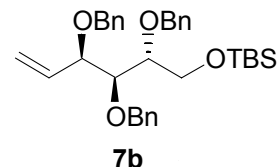
===== CHANNEL f1 =====
NUC1         13C
P1           8.00 usec
P2          16.00 usec
PL1         -6.00 dB
SFO1        100.6238401 MHz

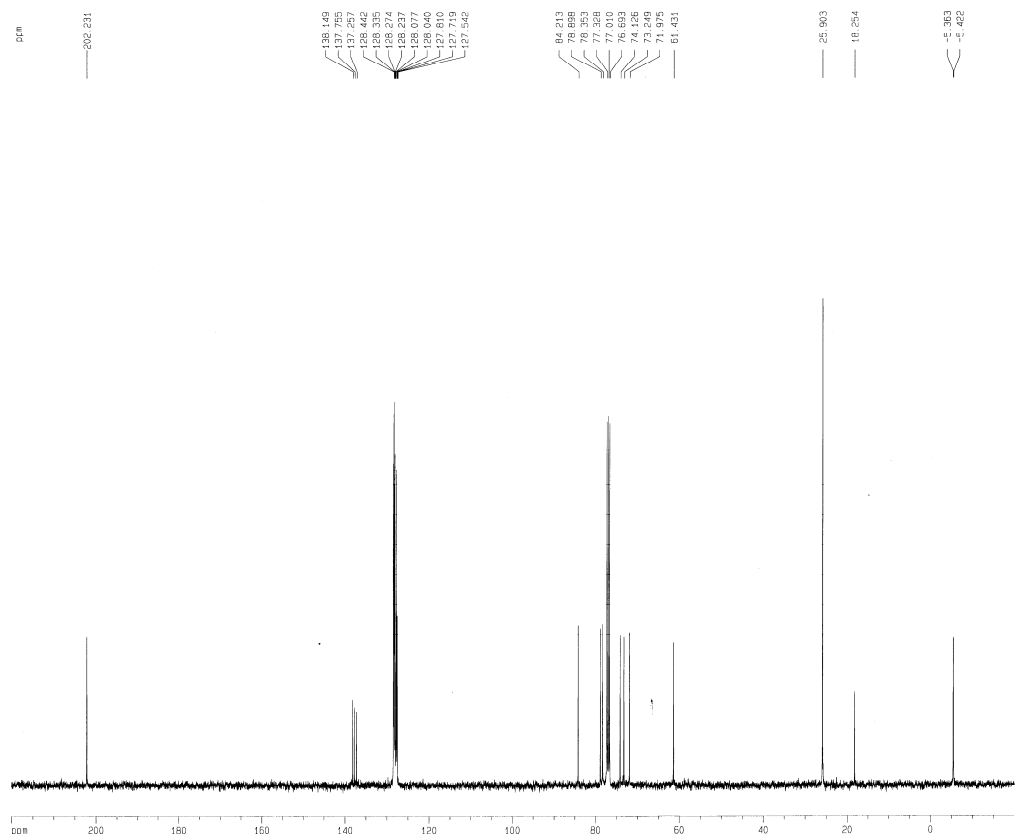
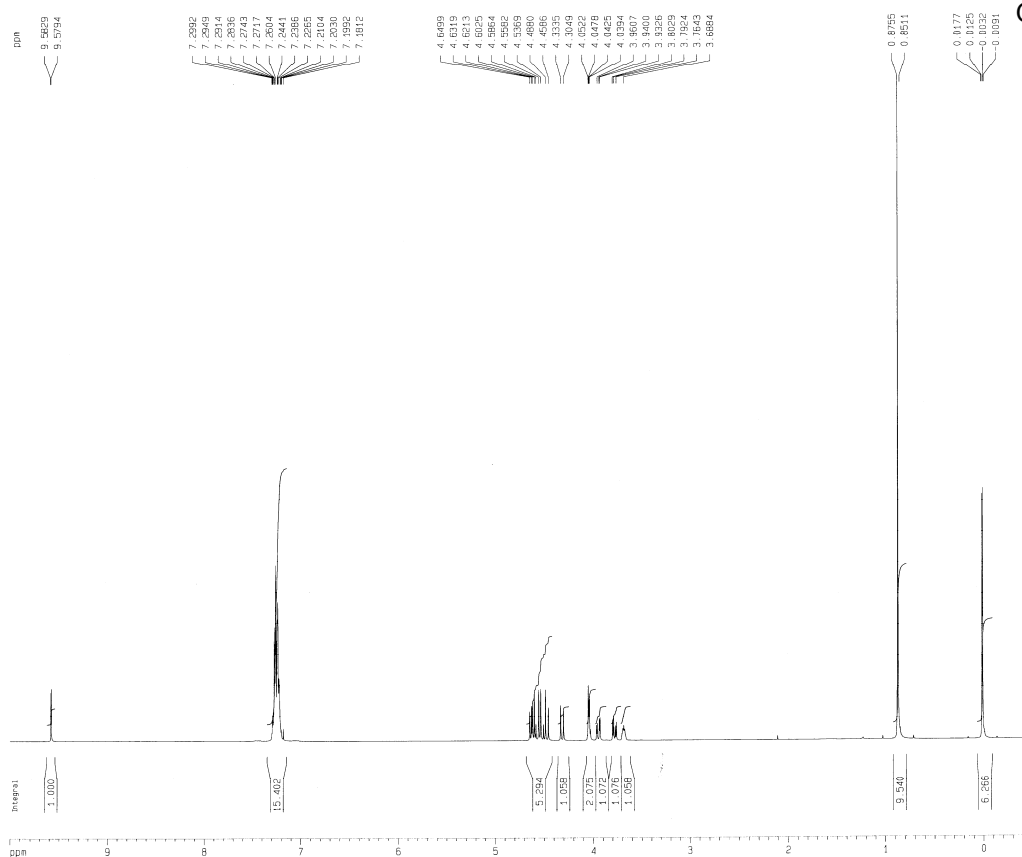
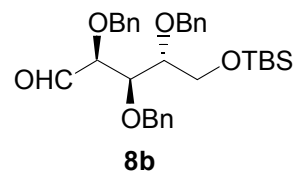
===== CHANNEL f2 =====
UNPRG2
wait2       1H
NUC2         1H
P3           10.30 usec
P4           20.60 usec
PCPD2       100.00 usec
PL2         -6.00 dB
PL12        16.40 dB
SFO2        400.1316005 MHz

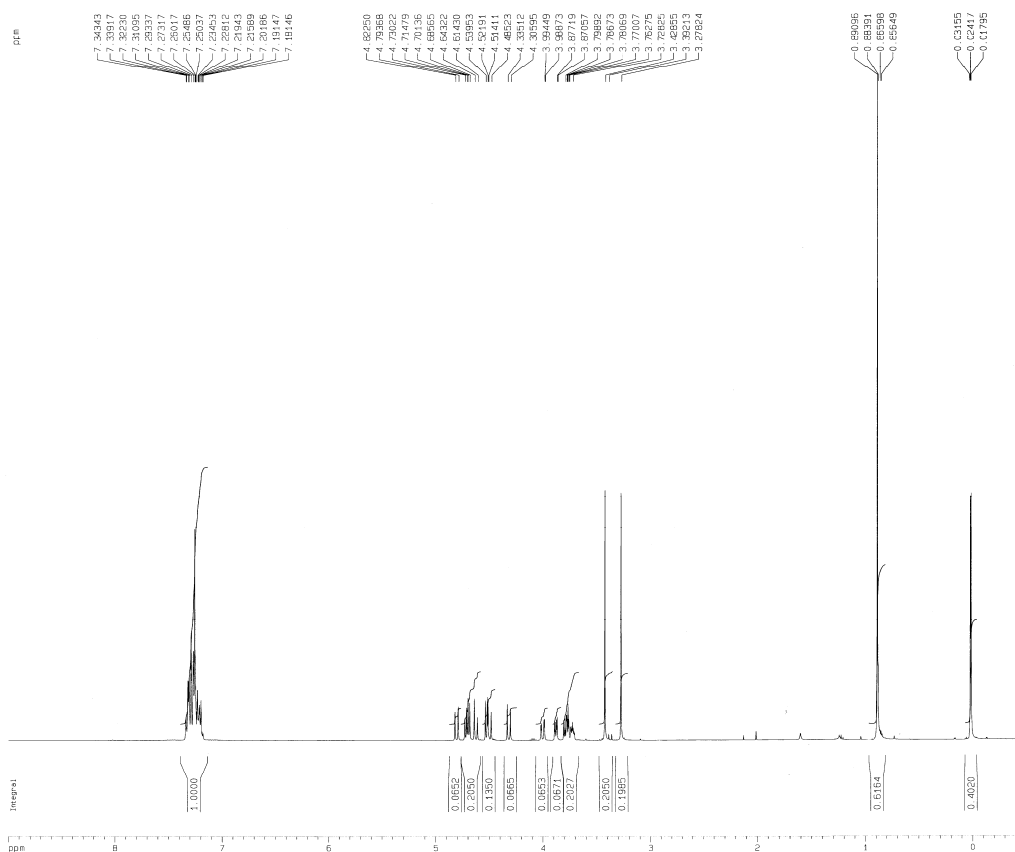
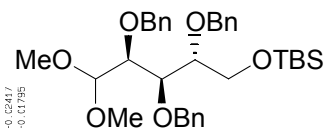
F2 - Processing parameters
SI           32768
SF           100.6127805 MHz
WDW          EM
SSB          0
LB           2.00 Hz
GB           0
PC           1.40

1D NMR plot parameters
CX           32.00 cm
CY           16.00 cm
F1P          200.600 ppm
F1           20122.55 Hz
F2P          -0.000 ppm
F2           -0.00 Hz
RPMCM        6.25000 ppm/cm
HZCM         628.82950 Hz/cm
    
```

10. ¹H and ¹³C NMR spectra of **7b**, **8b** and **9b**







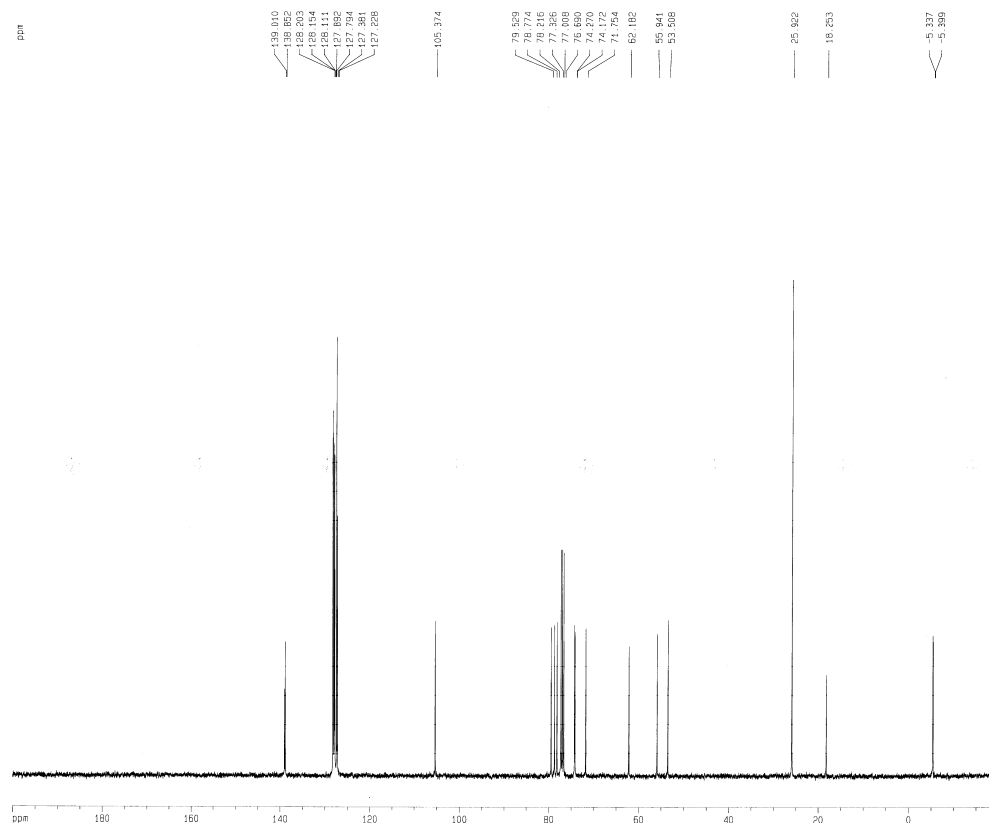
Current Data Parameters
NAME Manno
EXPNO 9
PROCNO 2

F2 - Acquisition Parameters
Date_ 00070203
Time 16.20
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TO 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 57
DW 73.600 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.40 usec
PL1 -6.00 dB
SFO1 400.1326354 MHz

F2 - Processing parameters
SI 16384
SF 400.1300323 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
FIP 9.000 ppm
F1 3601.7 Hz
F2 -200.06 Hz
F3 -200.06 Hz
PRMCM 0.29668 ppm/cm
HZCM 118.78860 Hz/cm



Current Data Parameters
NAME Manno
EXPNO 9
PROCNO 1

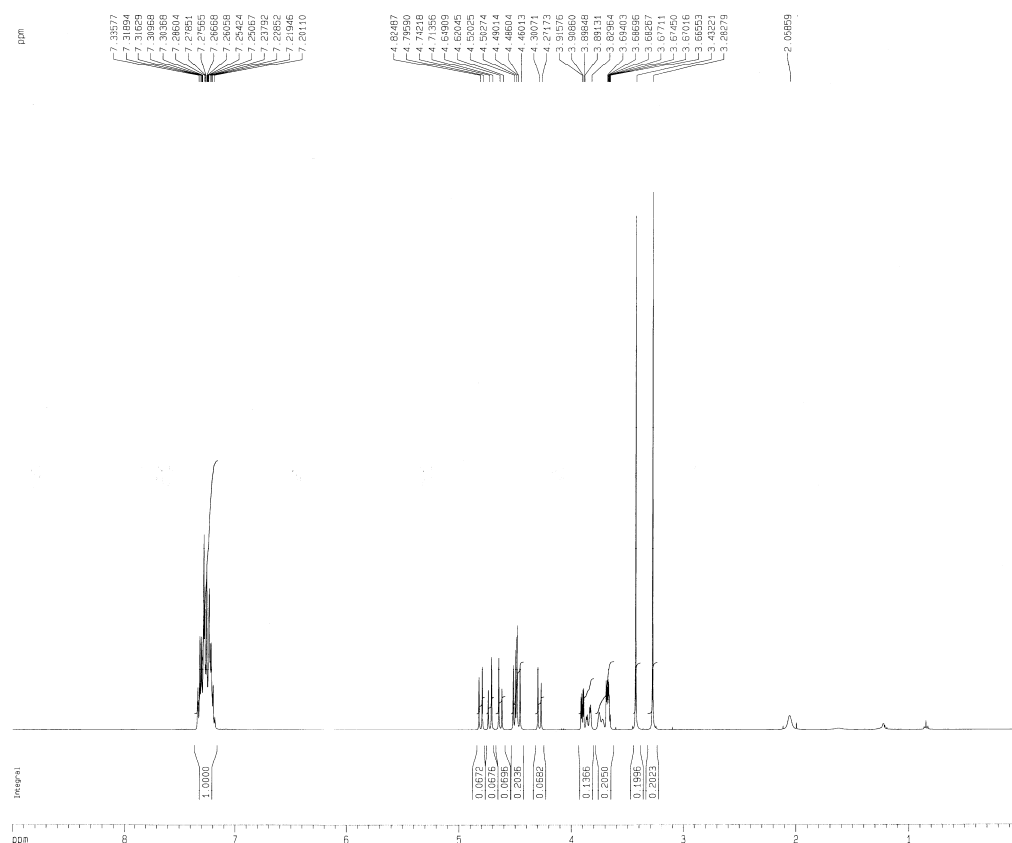
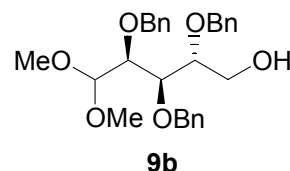
F2 - Acquisition Parameters
Date_ 20070221
Time 17.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TO 85536
SOLVENT CDCl3
NS 256
DS 2
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517075 sec
RG 5160.6
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.09999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -6.00 dB
SFO1 100.6238401 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL3 16.40 dB
PL13 16.40 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127781 MHz
WDW DM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 16.00 cm
FIP 200.000 ppm
F1 20122.56 Hz
F2 -20.000 ppm
F3 -2012.25 Hz
PRMCM 6.87500 ppm/cm
HZCM 691.71289 Hz/cm



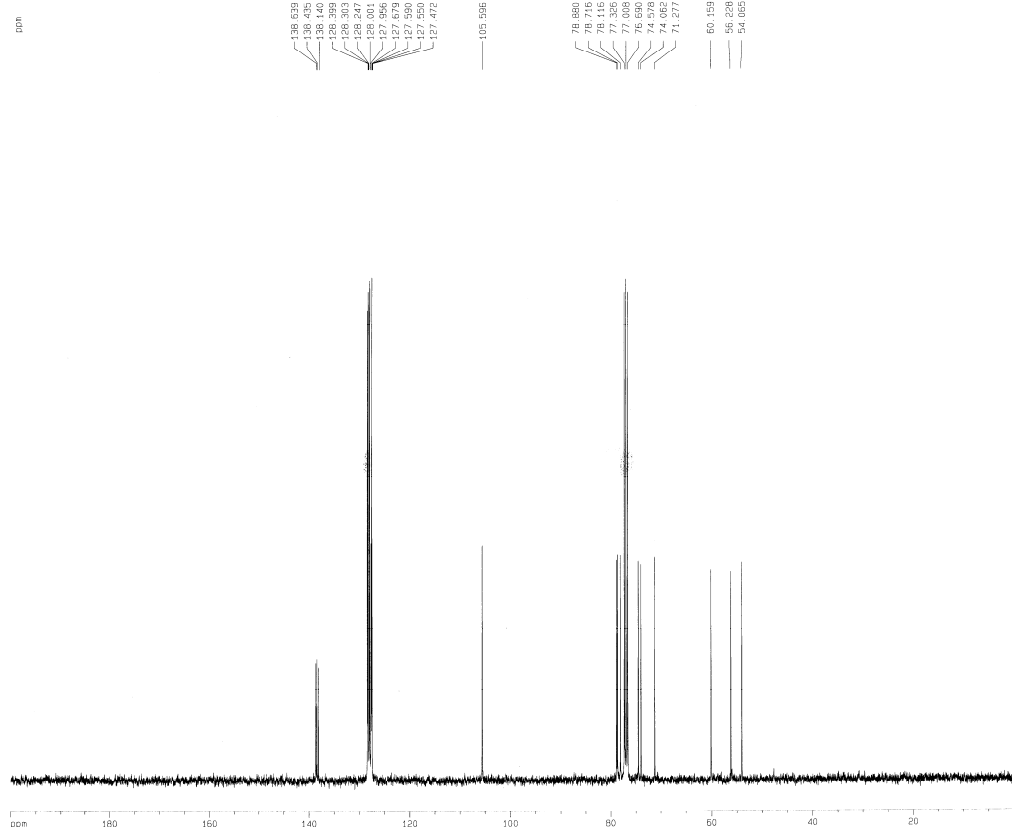
Current Data Parameters
NAME Manno
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070303
Time 15:26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 57
DW 73.600 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWK 0.01500000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 7.40 usec
PL1 -6.00 dB
SFO1 400.1362614 MHz

F2 - Processing parameters
SI 16384
SF 400.1300332 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 17.00 cm
FID 9.300 ppm
F1 3601.17 Hz
F2 0.000 ppm
F2 0.00 Hz
PRMCM 0.28125 ppm/cm
HZCM 112.53658 Hz/cm



Current Data Parameters
NAME Manno
EXPNO 10
PROCNO 2

F2 - Acquisition Parameters
Date_ 20070302
Time 17:19
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 57
DW 73.600 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWK 0.01500000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 7.40 usec
PL1 -6.00 dB
SFO1 400.1326354 MHz

F2 - Processing parameters
SI 32768
SF 100.6127758 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 16.00 cm
FID 200.000 ppm
F1 20122.55 Hz
F2 -0.000 ppm
F2 -0.00 Hz
PRMCM 6.29500 ppm/cm
HZCM 528.82390 Hz/cm

References

1. M. Kleban, U. Kautz, J. Greul, P. Hilgers, R. Kugler, H.-Q. Dong, V. Jaeger, *Synthesis* 2000, 1027-1033.
2. P. R. Skaanderup, L. Hyldtoft, R. Madsen, *Monatsh. Chem.* 2002, **133**, 467-472.
3. J. Désiré, J. Prandi, *Eur. J. Org. Chem.* 2000, 3075-3084.