

## SUPPLEMENTARY MATERIAL

### **Silver(I)-Catalyzed Addition of Zirconocenes to Glycal Epoxides. A New Synthesis of $\alpha$ -C-Glycosides.**

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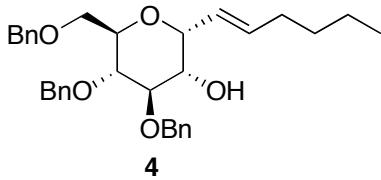
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Experimental procedures and spectral data for all new compounds, including copies of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra for **4**, **6**, **8**, **10**, **12**, **13**, **15**, and **16**.

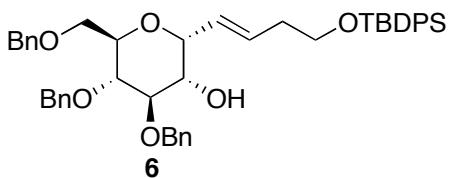
**General.** All reactions were performed under an N<sub>2</sub> atmosphere and all glassware was dried in an oven at 140 °C for 2 h prior to use. CH<sub>2</sub>Cl<sub>2</sub> was filtered using an alumina filtration system. Cp<sub>2</sub>ZrHCl and glycal epoxides **3**, **11** and **14** were prepared according to literature procedures.<sup>1,2</sup>

Reactions were monitored by TLC analysis (EM Science pre-coated silica gel 60 F<sub>254</sub> plates, 250 µm layer thickness) and visualization was accomplished with a 254 nm UV light and by staining with a PMA solution (5 g of phosphomolybdic acid in 100 mL of 95% EtOH), *p*-anisaldehyde solution (2.5 mL of *p*-anisaldehyde, 2 mL of AcOH, and 3.5 mL of conc. H<sub>2</sub>SO<sub>4</sub> in 100 mL of 95% EtOH), Vaughn's reagent (4.8 g of (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>•4 H<sub>2</sub>O and 0.2 g of Ce(SO<sub>4</sub>)<sub>2</sub> in 100 mL of a 3.5 N H<sub>2</sub>SO<sub>4</sub> solution) or a KMnO<sub>4</sub> solution (1.5 g of KMnO<sub>4</sub> and 1.5 g of K<sub>2</sub>CO<sub>3</sub> in 100 mL of a 0.1% NaOH solution). Flash chromatography on SiO<sub>2</sub> was used to purify the crude reaction mixtures. Infrared spectra were determined on a Nicolet Avatar 360 FT-IR spectrometer. <sup>1</sup>H spectra were obtained on a Bruker Avance instrument in CDCl<sub>3</sub> and <sup>13</sup>C NMR were obtained on a Bruker Avance 300 instrument in CDCl<sub>3</sub> unless otherwise noted. Chemical shifts were reported in parts per million with the residual solvent peak used as an internal standard. <sup>1</sup>H NMR spectra were run at 300, 500, or 600 MHz and are tabulated as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), number of protons, and coupling constant(s). <sup>13</sup>C NMR spectra were run at 76 MHz using the proton-decoupled pulse sequence with a d<sub>1</sub> of 3 seconds, and are tabulated by observed peak. Mass spectra were obtained on a Micromass Autospec double focusing instrument.

**Silver perchlorate on Celite®.** To a 100 mL round bottom flask charged with silver perchlorate (1.0 g, 4.8 mmol) in H<sub>2</sub>O (30 mL) was added Celite® (5.0 g). The resulting suspension was stirred for 30 min, filtered through a Buchner funnel, and dried in a vacuum oven (60 °C, 24 h) to yield 6.0 g of silver perchlorate on Celite® (20 wt %) as an off-white powder.

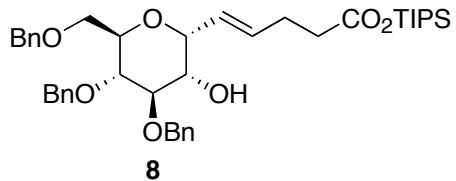


**(2*R*,3*S*,4*R*,5*R*,6*R*,*E*)-4,5-Bis(benzyloxy)-6-(benzyloxymethyl)-2-(hex-1-enyl)-tetrahydro-2*H*-pyran-3-ol (4).** **General Protocol A.** To a suspension of zirconocene hydrochloride (38 mg, 0.15 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1 mL) was added alkyne **1** (17  $\mu$ L, 0.15 mmol). The yellow reaction mixture was stirred at room temperature for 10 min, treated with AgClO<sub>4</sub> on Celite® (15.5 mg, 15.0  $\mu$ mol), stirred for 5 min, and treated dropwise with a solution of glycal epoxide **3** (44 mg, 0.10 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1 mL). The reaction mixture was stirred for 1 h, quenched with saturated aqueous NaHCO<sub>3</sub>, extracted with EtOAc, dried (MgSO<sub>4</sub>), and concentrated *in vacuo*. The residue was purified by chromatography on SiO<sub>2</sub> (3:7, EtOAc:hexanes) to yield 39 mg (76%) of **4** as a colorless oil: IR (neat) 3455, 3030, 2924, 2870, 1496, 1454, 1361, 1208, 1084, 1028, 734, 697 cm<sup>-1</sup>; <sup>1</sup>H NMR (600 MHz)  $\delta$  7.40-7.25 (m, 12 H), 7.25-7.15 (m, 3 H), 5.87 (dt, 3 H, *J* = 15.5, 6.7 Hz), 5.70 (dd, 1 H, *J* = 15.6, 6.7 Hz), 4.79 (d, 1 H, *J* = 11.5 Hz), 4.70 (t, 2 H, *J* = 10.9 Hz), 4.62 (d, 1 H, *J* = 12.1 Hz), 4.56 (d, 1 H, *J* = 11.1 Hz), 4.53 (d, 1 H, *J* = 12.1 Hz), 4.43 (t, 1 H, *J* = 5.5 Hz), 4.00-3.94 (m, 1 H), 3.78 (dd, 1 H, *J* = 10.5, 10.4 Hz), 3.77 (dd, 1 H, *J* = 9.0, 8.5 Hz), 3.70 (dd, 1 H, *J* = 10.5, 10.2 Hz), 3.70 (t, 1 H, *J* = 7.1 Hz), 3.66 (t, 1 H, *J* = 6.7 Hz), 2.50 (bs, 1 H), 2.10 (q, 2 H, *J* = 6.9 Hz), 1.42-1.37 (m, 2 H), 1.37-1.27 (m, 2 H), 0.90 (t, 3 H, *J* = 7.2 Hz); <sup>13</sup>C NMR (126 MHz)  $\delta$  138.3, 138.2, 137.8, 137.2, 128.6, 128.6, 128.5, 128.1, 128.0, 127.8, 124.0, 80.5, 76.6, 74.3, 74.1, 73.9, 73.5, 73.1, 71.0, 68.6, 32.5, 31.2, 22.4, 14.0; ESIMS *m/z* 539 ([M+Na]<sup>+</sup>, 100); HRMS (ESI) *m/z* calcd for C<sub>33</sub>H<sub>40</sub>O<sub>5</sub>Na (M+Na) 539.2773, found 539.2762.



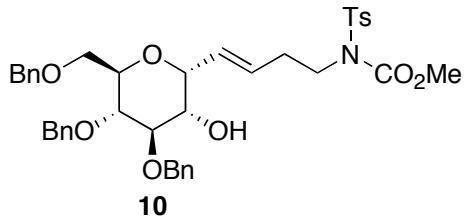
**(2*R*,3*S*,4*R*,5*R*,6*R*,*E*)-4,5-Bis(benzyloxy)-6-(benzyloxymethyl)-2-(4-(tert-butylidiphenylsilyloxy)but-1-enyl)-tetrahydro-2*H*-pyran-3-ol (6).** According to the General Protocol A, zirconocene hydrochloride (38 mg, 0.15 mmol), alkyne **5** (93 mg,

0.30 mmol), AgClO<sub>4</sub> on Celite® (15.5 mg, 15.0 µmol) and glycal epoxide **3** (44 mg, 0.10 mmol) afforded 55 mg (74%) of **6** as a colorless oil after purification on SiO<sub>2</sub> (1:9, EtOAc:hexanes): IR (neat) 3454, 3067, 3030, 2930, 2858, 1454, 1428, 1110, 736, 700 cm<sup>-1</sup>; <sup>1</sup>H NMR δ 7.67 (d, 4 H, *J* = 6.6 Hz), 7.44-7.35 (m, 6 H), 7.35-7.28 (m, 13 H), 7.20 (d, 2 H, *J* = 7.0 Hz), 5.87 (dt, 1 H, *J* = 15.6, 6.5 Hz), 5.76 (dd, 1 H, *J* = 15.7, 6.2 Hz), 4.77 (d, 1 H, *J* = 11.5 Hz), 4.71 (d, 1 H, *J* = 11.1 Hz), 4.67 (d, 1 H, *J* = 11.5 Hz), 4.60 (d, 1 H, *J* = 12.1 Hz), 4.55 (d, 1 H, *J* = 11.1 Hz), 4.51 (d, 1 H, *J* = 12.2 Hz), 4.43 (t, 1 H, *J* = 4.8 Hz), 3.98-3.93 (m, 1 H), 3.80-3.70 (m, 4 H), 3.70-3.64 (m, 3 H), 2.50 (d, 1 H, 6.4 Hz), 2.36 (q, 2 H, *J* = 6.5 Hz), 1.05 (s, 9 H); <sup>13</sup>C NMR δ 138.2, 138.0, 137.7, 135.5, 133.8, 133.0, 129.5, 128.5, 128.4, 128.3, 127.9, 127.8, 127.6, 127.6, 126.1, 80.4, 76.4, 74.7, 73.9, 73.7, 73.4, 73.0, 70.9, 68.4, 63.3, 36.1, 26.8, 19.2; ESIMS *m/z* 565 ([M+Na]<sup>+</sup>, 100); HRMS (ESI) *m/z* calcd for C<sub>47</sub>H<sub>54</sub>O<sub>6</sub>SiNa (M+Na) 765.3587, found 765.3550.

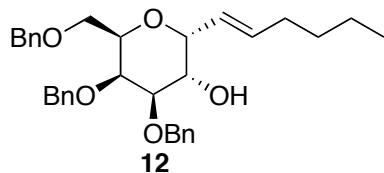


**(E)-Triisopropylsilyl 5-((2*R*,3*S*,4*R*,5*R*,6*R*)-4,5-bis(benzyloxy)-6-(benzyloxymethyl)-3-hydroxy-tetrahydro-2*H*-pyran-2-yl)pent-4-enoate (8).** According to the General Protocol A, zirconocene hydrochloride (38 mg, 0.15 mmol), alkyne **7** (76 mg, 0.30 mmol), AgClO<sub>4</sub> on Celite® (15.5 mg, 15.0 µmol) and glycal epoxide **3** (44 mg, 0.10 mmol) afforded 46 mg (67%) of **8** as a colorless oil after purification on SiO<sub>2</sub> (6:4, CH<sub>2</sub>Cl<sub>2</sub>:hexanes): IR (neat) 3479, 3030, 2944, 2867, 1716, 1454, 1365, 1073, 884, 735, 697 cm<sup>-1</sup>; <sup>1</sup>H NMR δ 7.38-7.28 (m, 13 H), 7.21 (d, 2 H, *J* = 6.1 Hz), 5.88 (dt, 1 H, *J* = 15.6, 6.0 Hz), 5.76 (dd, 1 H, *J* = 15.6, 6.1 Hz), 4.77 (d, 1 H, *J* = 11.5 Hz), 4.70 (t, 2 H, *J* = 10.9 Hz), 4.61 (d, 1 H, *J* = 12.1 Hz), 4.56 (d, 1 H, *J* = 11.2 Hz), 4.52 (d, 1 H, *J* = 12.2 Hz), 4.43 (t, 1 H, *J* = 4.5 Hz), 4.00-3.93 (m, 1 H), 3.80-3.73 (m, 2 H), 3.73-3.64 (m, 3 H), 2.62 (d, 1 H, *J* = 6.7 Hz), 2.51-2.45 (m, 2 H), 2.45-2.38 (m, 2 H), 1.34-1.25 (m, 3 H), 1.08 (d, 18 H, *J* = 7.4 Hz); <sup>13</sup>C NMR δ 173.0, 138.2, 138.0, 137.7, 134.3, 128.5, 128.4, 128.4, 127.9, 127.8, 127.7, 125.3, 80.1, 77.2, 76.2, 74.1, 73.6, 73.4, 73.2, 70.8, 68.4, 35.1,

28.2, 17.8, 11.9; ESIMS  $m/z$  711 ( $[M+Na]^+$ , 100), 515 (57); HRMS (ESI)  $m/z$  calcd for  $C_{41}H_{56}O_7SiNa$  ( $M+Na$ ) 711.3693, found 711.3687.

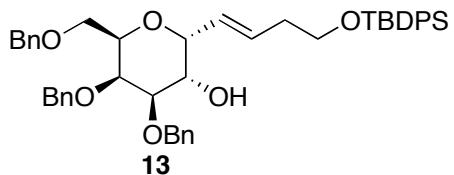


**Methyl (*E*)-4-((2*R*,3*S*,4*R*,5*R*,6*R*)-4,5-bis(benzyloxy)-6-(benzyloxymethyl)-3-hydroxytetrahydro-2*H*-pyran-2-yl)but-3-enyl(tosyl)carbamate (10).** According to the General Protocol A, zirconocene hydrochloride (38 mg, 0.15 mmol), alkyne **9** (84 mg, 0.30 mmol),  $\text{AgClO}_4$  on Celite® (15.5 mg, 15.0  $\mu\text{mol}$ ) and glycal epoxide **3** (44 mg, 0.10 mmol) afforded 50 mg (70%) of **10** as a colorless oil after purification on  $\text{SiO}_2$  (4:6, EtOAc:hexanes): IR (neat) 3521, 3030, 2921, 1736, 1453, 1359, 1168, 910, 734, 699  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR  $\delta$  7.84 (d, 2 H,  $J$  = 8.1 Hz), 7.39-7.23 (m, 15 H), 7.21 (d, 2 H,  $J$  = 6.0 Hz), 5.89-5.77 (m, 2 H), 4.79 (d, 1 H,  $J$  = 11.5 Hz), 4.72 (d, 1 H,  $J$  = 11.3 Hz), 4.62 (d, 1 H,  $J$  = 12.1 Hz), 4.54 (t, 2 H, 11.4 Hz), 4.45 (t, 1 H,  $J$  = 4.4 Hz), 3.97-3.88 (m, 3 H), 3.82-3.74 (m, 2 H), 3.73-3.64 (m, 6 H), 2.63 (d, 1 H,  $J$  = 6.7 Hz), 2.58-2.51 (m, 2 H), 2.42 (s, 3 H);  $^{13}\text{C}$  NMR  $\delta$  152.8, 144.5, 138.3, 138.2, 137.9, 136.8, 131.3, 129.3, 128.5, 128.4, 127.9, 127.8, 127.7, 127.6, 80.4, 74.1, 73.7, 73.4, 73.3, 71.0, 68.6, 53.7, 46.6, 33.4, 21.6; ESIMS  $m/z$  738 ( $[M+Na]^+$ , 100); HRMS (ESI)  $m/z$  calcd for  $C_{40}H_{45}O_9SNa$  ( $M+Na$ ) 738.2713, found 738.2692.

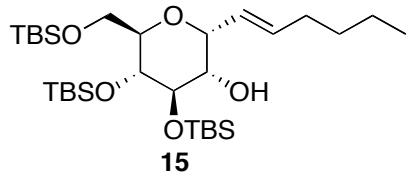


**(2*R*,3*S*,4*R*,5*S*,6*R*,*E*)-4,5-Bis(benzyloxy)-6-(benzyloxymethyl)-2-(hex-1-enyl)-tetrahydro-2*H*-pyran-3-ol (12).** According to the General Protocol A, zirconocene hydrochloride (38 mg, 0.15 mmol), alkyne **1** (17  $\mu\text{L}$ , 0.30 mmol),  $\text{AgClO}_4$  on Celite® (15.5 mg, 15.0  $\mu\text{mol}$ ) and glycal epoxide **11** (44 mg, 0.10 mmol) afforded 38 mg (73%) of **12** as a colorless oil after purification on  $\text{SiO}_2$  (1:9, EtOAc:hexanes): IR (neat) 3453,

3030, 2925, 2871, 1454, 1094, 735, 698 cm<sup>-1</sup>; <sup>1</sup>H NMR δ 7.33-7.23 (m, 15 H), 5.84 (dt, 1 H, *J* = 15.6, 6.7 Hz), 5.56 (dd, 1 H, *J* = 15.9, 5.5 Hz), 4.74 (d, 1 H, *J* = 11.6 Hz), 4.71 (d, 1 H, *J* = 11.6 Hz), 4.55 (d, 1 H, *J* = 11.7 Hz), 4.52 (d, 1 H, *J* = 5.7 Hz), 4.50 (d, 1 H, *J* = 6.1 Hz), 4.45 (d, 1 H, *J* = 11.9 Hz), 4.16-4.09 (m, 1 H), 4.04-3.97 (m, 2 H), 3.75-3.68 (m, 1 H), 3.65-3.57 (m, 2 H), 2.10-2.02 (m, 3 H), 1.38-1.22 (m, 4 H), 0.86 (t, 3 H, *J* = 7.3 Hz); <sup>13</sup>C NMR δ 138.5, 138.3, 138.1, 136.4, 128.6, 128.5, 128.4, 128.0, 130.0, 127.8, 123.1, 79.3, 73.8, 73.6, 73.5, 72.4, 68.8, 68.1, 32.5, 31.3, 22.4, 14.1; ESIMS *m/z* 539 ([M+Na]<sup>+</sup>, 100), 536 (20); HRMS (ESI) *m/z* calcd for C<sub>33</sub>H<sub>40</sub>O<sub>5</sub>Na (M+Na) 539.2773, found 539.2800.

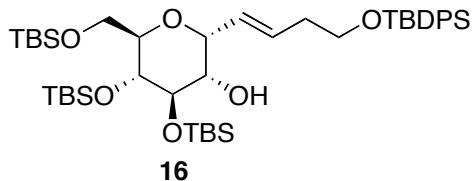


**(2*R*,3*S*,4*R*,5*S*,6*R*,*E*)-4,5-Bis(benzyloxy)-6-(benzyloxymethyl)-2-(4-(tert-butylidiphenylsilyloxy)but-1-enyl)-tetrahydro-2*H*-pyran-3-ol (13).** According to the General Protocol A, zirconocene hydrochloride (38 mg, 0.15 mmol), alkyne **5** (93 mg, 0.30 mmol), AgClO<sub>4</sub> on Celite® (15.5 mg, 15.0 µmol) and glycal epoxide **11** (44 mg, 0.10 mmol) afforded 53 mg (72%) of **13** as a colorless oil after purification on SiO<sub>2</sub> (hexanes to EtOAc:hexanes, 0.5:9.5): IR (neat) 3459, 3066, 3030, 2929, 2857, 1454, 1428, 1110, 736, 700 cm<sup>-1</sup>; <sup>1</sup>H NMR δ 7.69 (d, 4 H, *J* = 6.8 Hz), 7.45-7.35 (m, 6 H), 7.35-7.28 (m, 15 H), 5.89 (dt, 1 H, *J* = 15.6, 6.6 Hz), 5.68 (dd, 1 H, *J* = 15.7, 4.5 Hz), 4.78 (d, 1 H, *J* = 11.5 Hz), 4.72 (d, 1 H, *J* = 11.6 Hz), 4.62-4.55 (m, 2 H), 4.53 (d, 1 H, *J* = 6.7 Hz), 4.51 (d, 1 H, *J* = 6.3 Hz), 4.49-4.45 (m, 1 H), 4.19-4.13 (m, 1 H), 4.09-4.04 (m, 1 H), 4.04-4.01 (m, 1 H), 3.73 (t, 3 H, *J* = 6.4 Hz), 3.69-3.63 (m, 1 H), 3.61 (d, 1 H, *J* = 6.9 Hz), 2.37 (q, 2 H, *J* = 6.5 Hz), 2.11 (d, 1 H, *J* = 2.5 Hz), 1.06 (s, 9 H); <sup>13</sup>C NMR δ 138.4, 138.1, 138.0, 135.6, 133.8, 132.1, 129.6, 128.5, 128.3, 128.3, 128.1, 127.9, 127.8, 127.6, 127.6, 125.3, 79.2, 77.2, 73.7, 73.5, 73.3, 72.5, 72.2, 68.6, 67.8, 63.3, 36.1, 26.8, 19.2; ESIMS *m/z* 765 ([M+Na]<sup>+</sup>, 100), 391 (10); HRMS (ESI) *m/z* calcd for C<sub>47</sub>H<sub>54</sub>O<sub>6</sub>SiNa (M+Na) 765.3587, found 765.3582.



**(2*R*,3*S*,4*R*,5*R*,6*R*,*E*)-4,5-Bis(tert-butyldimethylsilyloxy)-6-((tert-butyldimethylsilyloxy)methyl)-2-(hex-1-enyl)-tetrahydro-2*H*-pyran-3-ol (15).**

According to the General Protocol A, zirconocene hydrochloride (38 mg, 0.15 mmol), alkyne **1** (17  $\mu$ L, 0.30 mmol), AgClO<sub>4</sub> on Celite® (15.5 mg, 15.0  $\mu$ mol) and glycal epoxide **14** (50 mg, 0.10 mmol) afforded 44 mg (74%) of **15** as a colorless oil after purification on SiO<sub>2</sub> (1:9, EtOAc:hexanes): IR (neat) 3532, 2930, 2858, 1472, 1254, 1093, 836, 778 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz)  $\delta$  5.81-5.66 (m, 2 H), 4.25-4.17 (m, 1 H), 4.10-3.93 (m, 2 H), 3.92-3.81 (m, 3 H), 3.82-3.78 (m, 1 H), 3.71 (d, 1 H, *J* = 11.5 Hz), 3.34-3.26 (m, 1 H), 2.15-2.00 (m, 2 H), 1.43-1.30 (m, 4 H), 0.96-0.84 (m, 30 H), 0.17-0.02 (m, 18 H); <sup>13</sup>C NMR  $\delta$  134.1, 127.9, 80.3, 72.3, 70.6, 69.8, 68.9, 61.3, 32.1, 31.2, 25.9, 25.8, 22.3, 17.9, 13.9, -4.9, -5.0, -5.1, -5.3; ESIMS *m/z* 611 ([M+Na]<sup>+</sup>, 100), 439 (20); HRMS (ESI) *m/z* calcd for C<sub>30</sub>H<sub>64</sub>O<sub>5</sub>Si<sub>3</sub>Na (M+Na) 611.3959, found 611.3936.

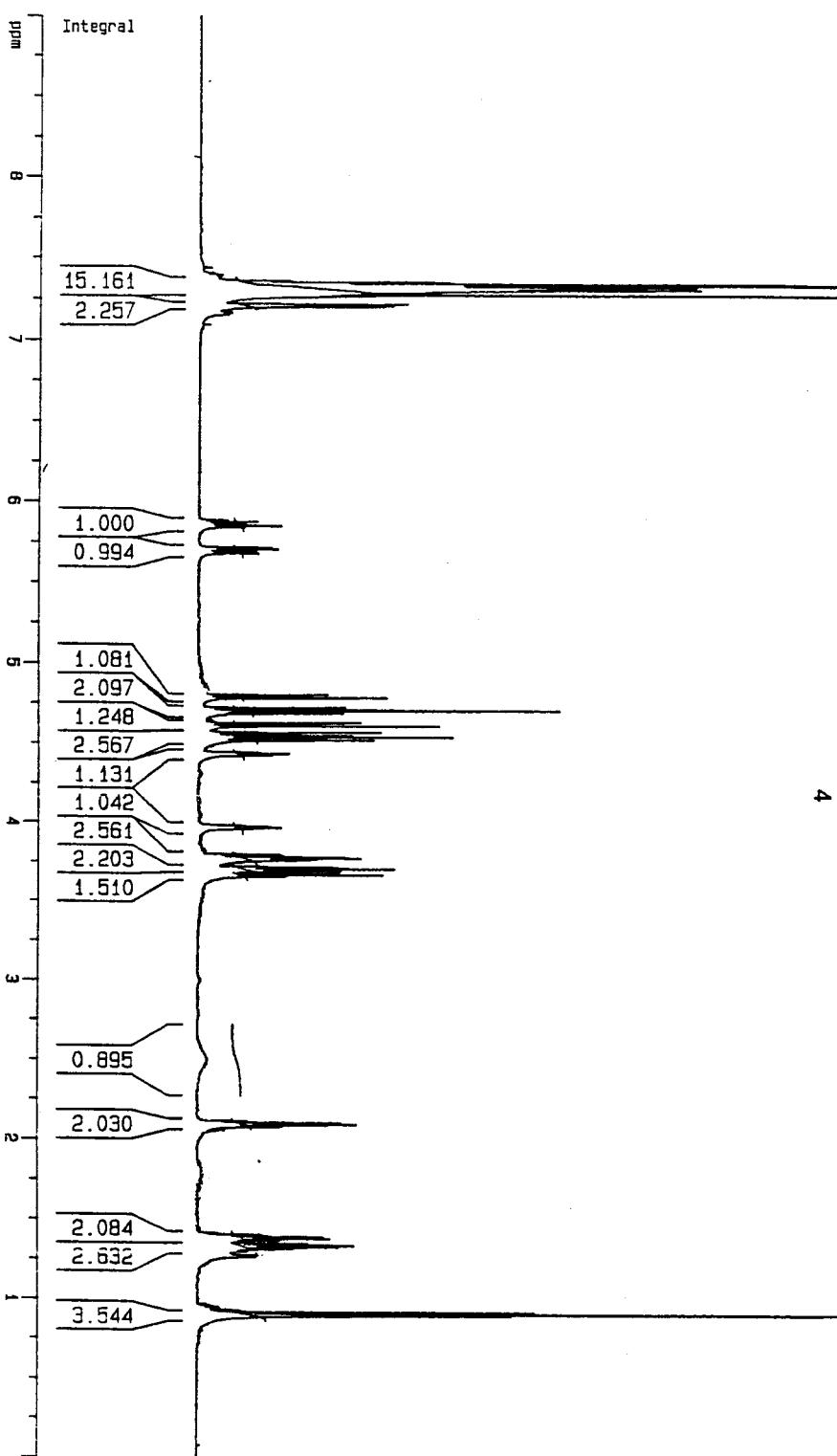
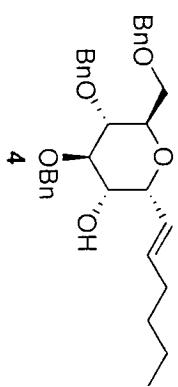
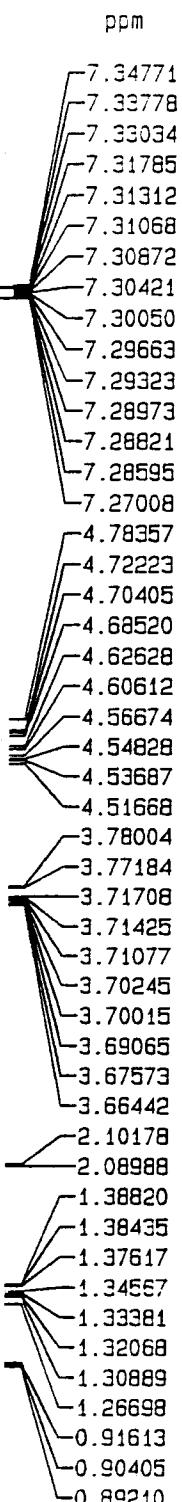


**(2*R*,3*S*,4*R*,5*R*,6*R*,*E*)-4,5-Bis(tert-butyldimethylsilyloxy)-6-((tert-butyldimethylsilyloxy)methyl)-2-(4-(tert-butyldiphenylsilyloxy)but-1-enyl)-tetrahydro-2*H*-pyran-3-ol (16).** According to the General Protocol A, zirconocene hydrochloride (38 mg, 0.15 mmol), alkyne **5** (93 mg, 0.30 mmol), AgClO<sub>4</sub> on Celite® (15.5 mg, 15.0  $\mu$ mol) and glycal epoxide **14** (50 mg, 0.10 mmol) afforded 58 mg (71%) of **16** as a colorless oil after purification on SiO<sub>2</sub> (hexanes to EtOAc:hexanes, 0.5:9.5): IR (neat) 3528, 2930, 2858, 1472, 1255, 1095, 836, 778, 702 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz)  $\delta$  7.70-7.64 (m, 4 H), 7.45-7.33 (m, 6 H), 5.84-5.70 (m, 2 H), 4.25-4.18 (m, 1 H), 3.91 (t, 1 H, *J* = 2.5 Hz), 3.89-3.79 (m, 3 H), 3.76-3.73 (m, 1 H), 3.71 (d, 2 H, *J* = 6.7 Hz), 3.35-3.27 (m, 1 H), 2.43-2.33 (m, 2 H), 1.04 (s, 9 H), 1.00-0.86 (m, 27 H), 0.13-0.05 (m, 18 H)

H);  $^{13}\text{C}$  NMR  $\delta$  135.7, 134.1, 130.5, 129.9, 129.6, 127.7, 80.3, 72.2, 70.4, 69.9, 68.8, 63.7, 61.3, 35.9, 31.7, 26.9, 26.0, 25.9, 22.8, 19.3, 18.3, 18.0, 14.2, -4.5, -4.9, -5.1, -5.2; ESIMS  $m/z$  765 ( $[\text{M}+\text{Na}]^+$ , 100), 391 (10); HRMS (ESI)  $m/z$  calcd for  $\text{C}_{47}\text{H}_{54}\text{O}_6\text{SiNa}$  ( $\text{M}+\text{Na}$ ) 765.3587, found 765.3582.

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- (1) Buchwald, S.L.; LaMaire, S.J.; Nielsen, R.B. *Org. Synth.* **1993**, *71*, 77.
  - (2) (a) Halcomb, R. L.; Danishefsky, S. J. *J. Am. Chem. Soc.* **1989**, *111*, 6661. (b) Adam, W.; Bialas, J.; Hadjiarapoglou, L. *Chem. Ber.* **1991**, *124*, 2377.

JP-253-600, cdc13, 600MHz



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SF	600.8300276 MHz
MDW	E1
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

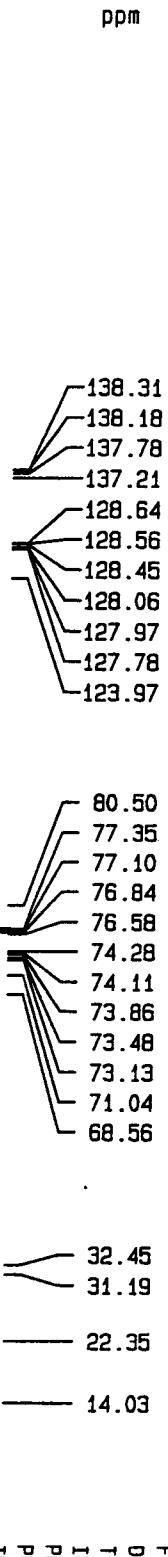
1D NMR plot parameters

CX	20.00 cm
F1P	9.000 ppm
F1	5407.47 Hz
F2P	0.000 ppm
F2	0.10 Hz
PPCM	0.45000 ppm/cm
PCM	270.37250 Hz/cm

10/28/04 jp1\_253 CDCl<sub>3</sub>

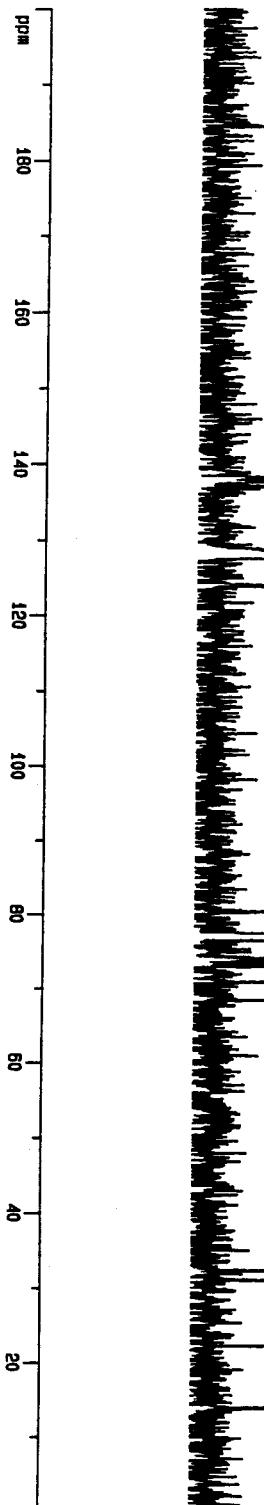
10

Current Data Parameters  
 NAME jp1\_253  
 EXPNO 2  
 PROCNO 4



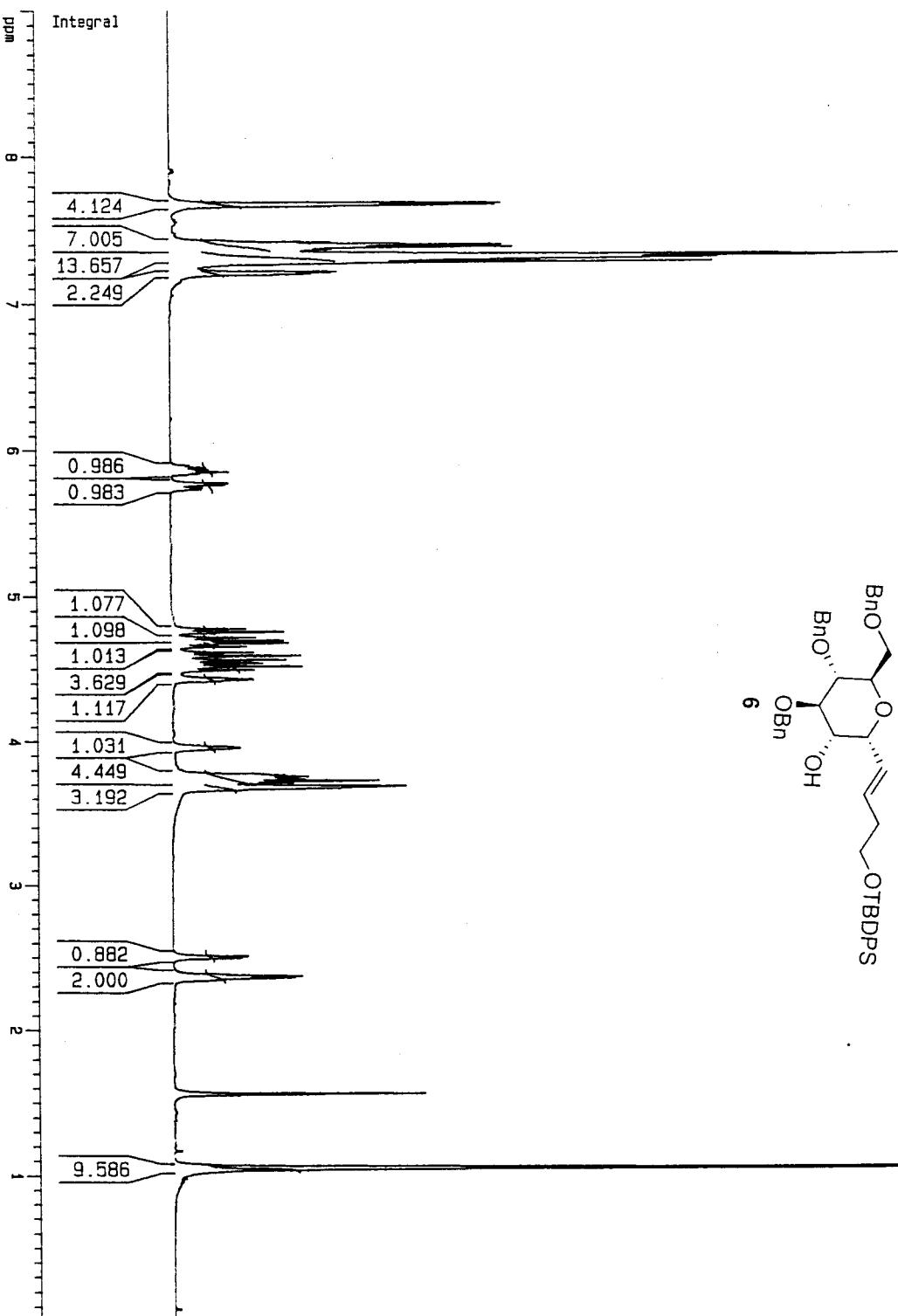
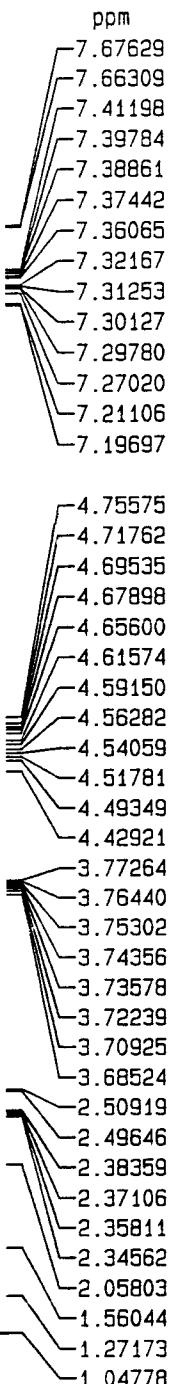
1D NMR plot parameters

CX	20.00	cm
FR	200.000	ppm
F1	25151.56	Hz
F2P	0.000	ppm
F2	0.00	Hz
PPCM	10.00000	ppm/cm <sup>-1</sup>
HZCM	1257.57788	Hz/cm <sup>-1</sup>



jp1\_271 1H NMR 500 MHz CDCl<sub>3</sub> 11-20-04

Current Data Parameters  
 NAME jp1\_271.p  
 EXPNO 1  
 PROCN 1

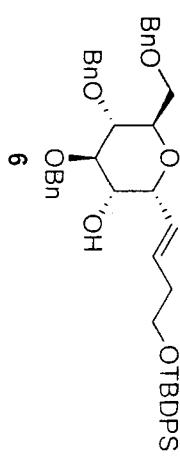
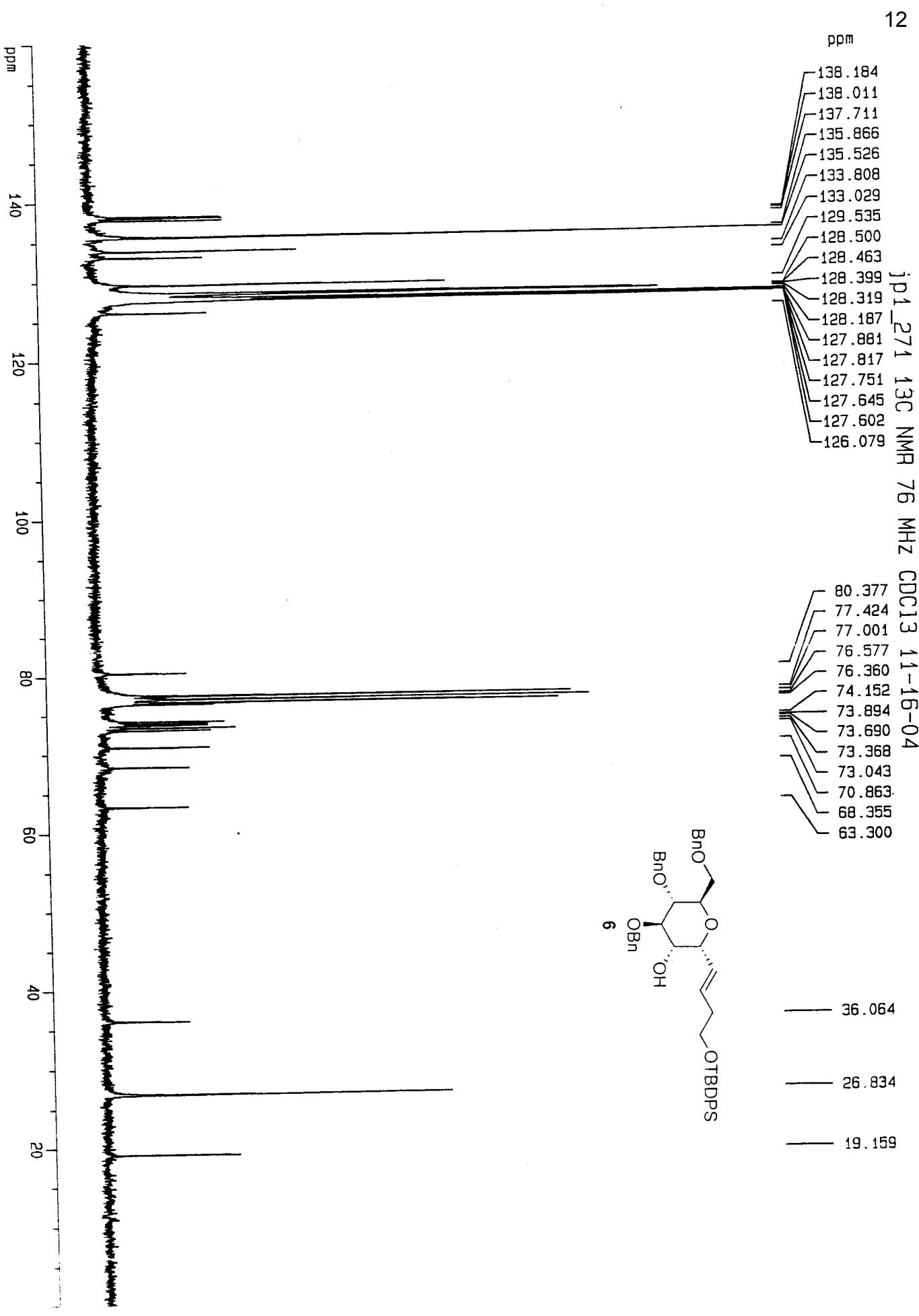


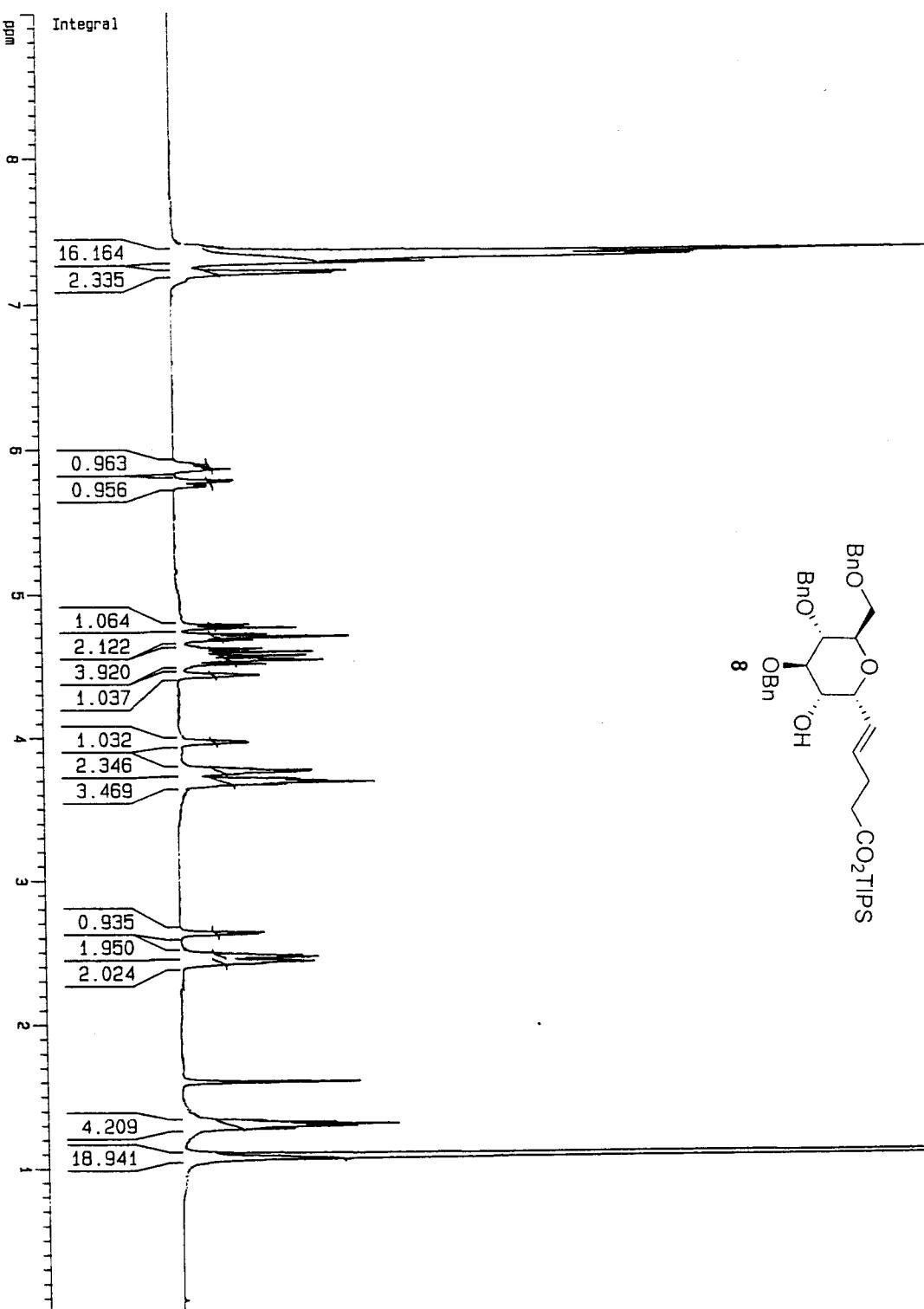
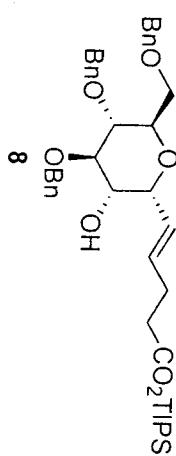
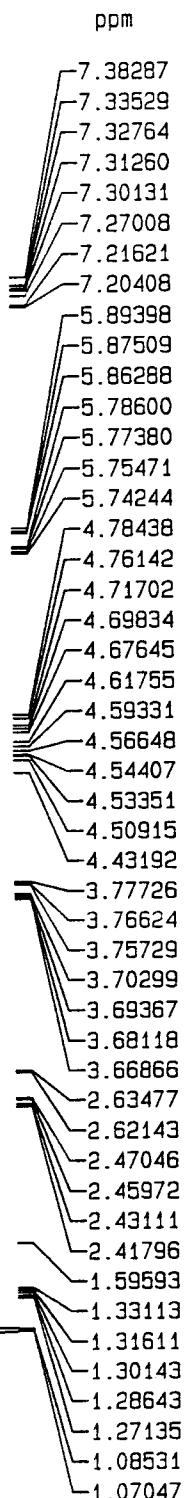
F2 - Acquisition Parameters  
 Date 5/00/00  
 Time 23:02  
 INSTRUM spect  
 PROBHD 5 mm TXI 13C  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl<sub>3</sub>  
 NS 16  
 DS 0  
 SWH 7507.507 Hz  
 FIDRES 0.229111 Hz  
 AQ 2.182398 sec  
 RG 128  
 DW 66.600 usec  
 DE 6.00 usec  
 TE 290.0 K  
 D1 6.0000000 sec  
 P1 10.50 usec  
 DE 6.00 usec  
 SF 01 500.1330008 MHz  
 NUC1 <sup>1</sup>H  
 PL1 0.00 dB

F2 - Processing parameters

ST 32768  
 SF 500.1300235 MHz  
 WDW EM  
 SSB 0  
 LB 0.40 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P g.000 ppm  
 F1 450.17 Hz  
 F2P 0.000 ppm  
 F2 0.00 Hz  
 PPMCM 0.45000 ppm/cm  
 HZCM 225.05952 Hz/cm



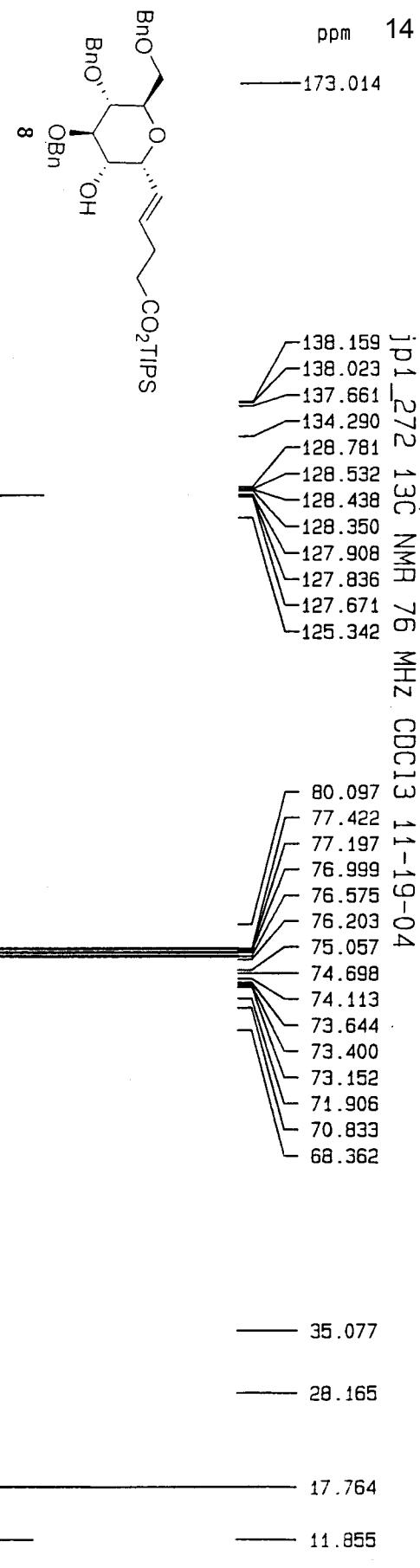
jp1\_272 1H NMR 500 MHz CDCl<sub>3</sub> 11-19-04

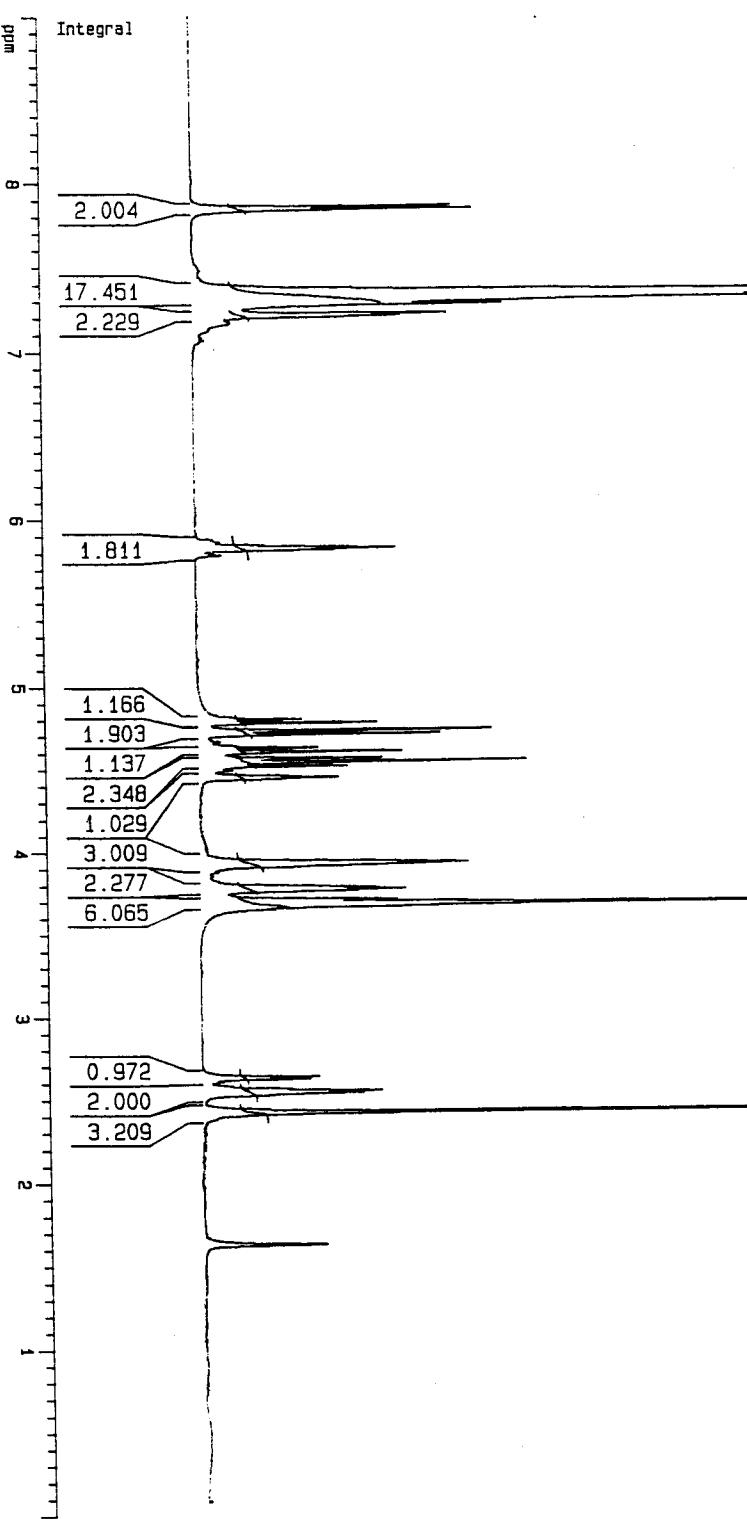
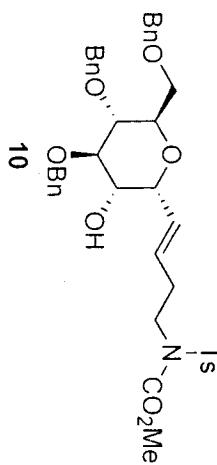
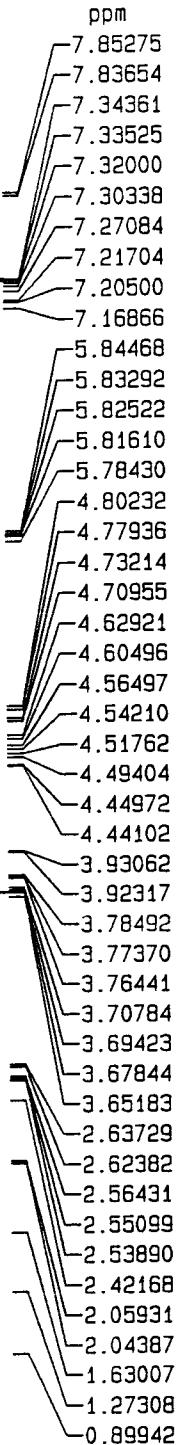
## F2 - Processing parameters

SI	32768
SF	500.1300230 MHz
MDW	E1
SSB	0
LB	0.40 Hz
GB	0
PC	1.00

1D NMR plot parameters	20.00 cm
CX	9.003 ppm
F1P	4501.17 Hz
F1	0.009 ppm
F2P	0.09 Hz
F2	0
PPMCM	0.45000 ppm
HZCM	225.05852 Hz/cm

Current Data Parameters	jp1_272.p
NAME	jp1_272.p
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date	500000
Time	22.36
INSTRUM	spect
PROBHD	5 mm TXI 13r
PULPROG	zg
TD	32768
SOLVENT	CDCl <sub>3</sub>
NS	16
DS	0
SWH	7507.507 Hz
FLDRES	0.229111 Hz
AQ	2.1823988 sec
RG	45.3
DW	66.600 usec
DE	6.00 usec
TE	290.0 K
D1	6.0000000 sec
P1	10.60 usec
DE	6.00 usec
SFO1	500.1330008 MHz
NUC1	<sup>1</sup> H
PL1	0.00 dB

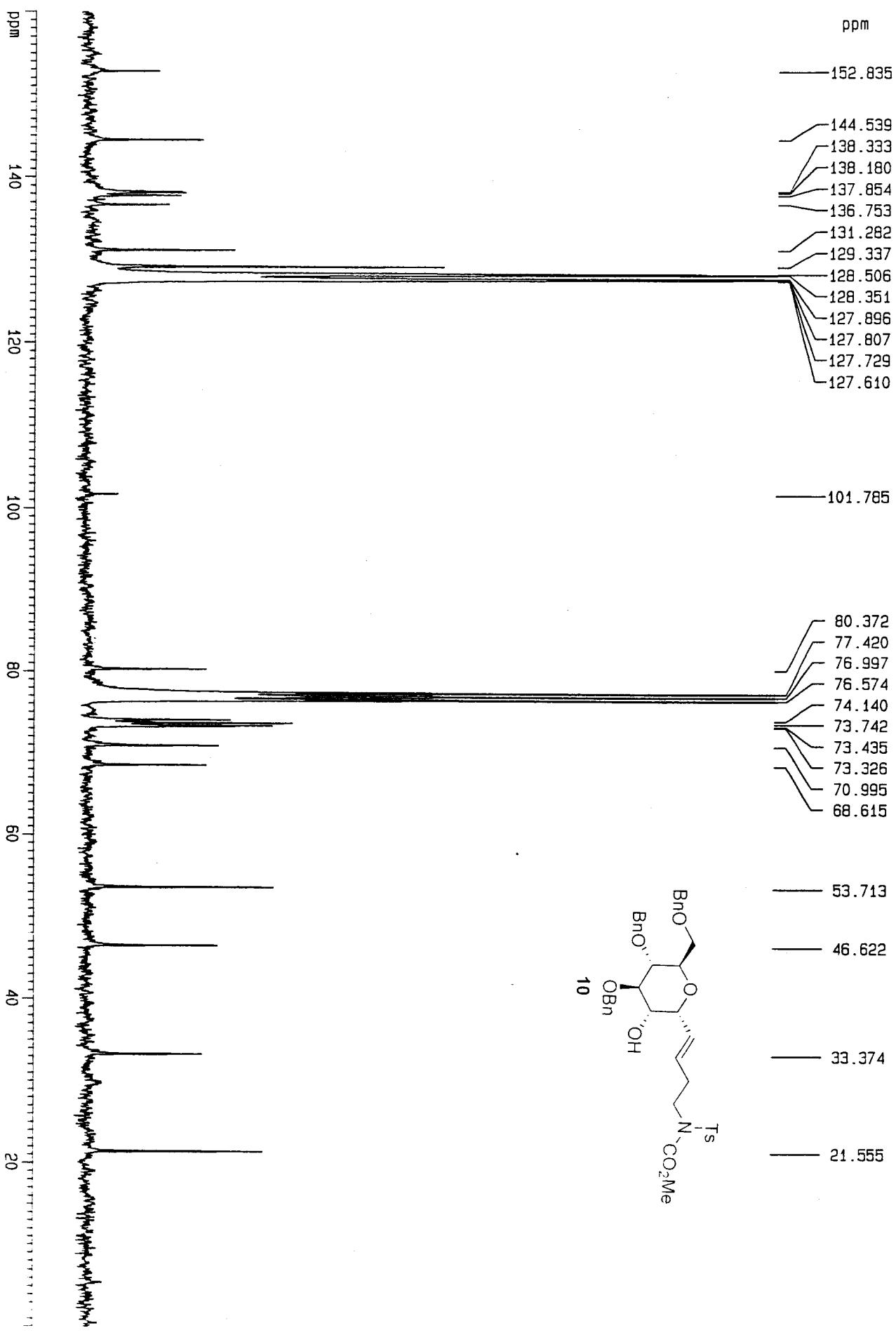


jp1\_270 1H NMR 500 MHz CDCl<sub>3</sub> 11-19-04

F2 - Processing parameters	
SI	32768
SF	500.1300233 MHz
WDW	E1
SSB	0
LB	0.40 Hz
GB	0
PC	1.00

1D NMR plot parameters	
CX	20.00 cm
F1P	9.000 ppm
F1	4501.17 Hz
F2P	0.009 ppm
F2	0.09 Hz
PPCM	0.45001 ppm/cm
HZCM	225.059E? Hz/cm

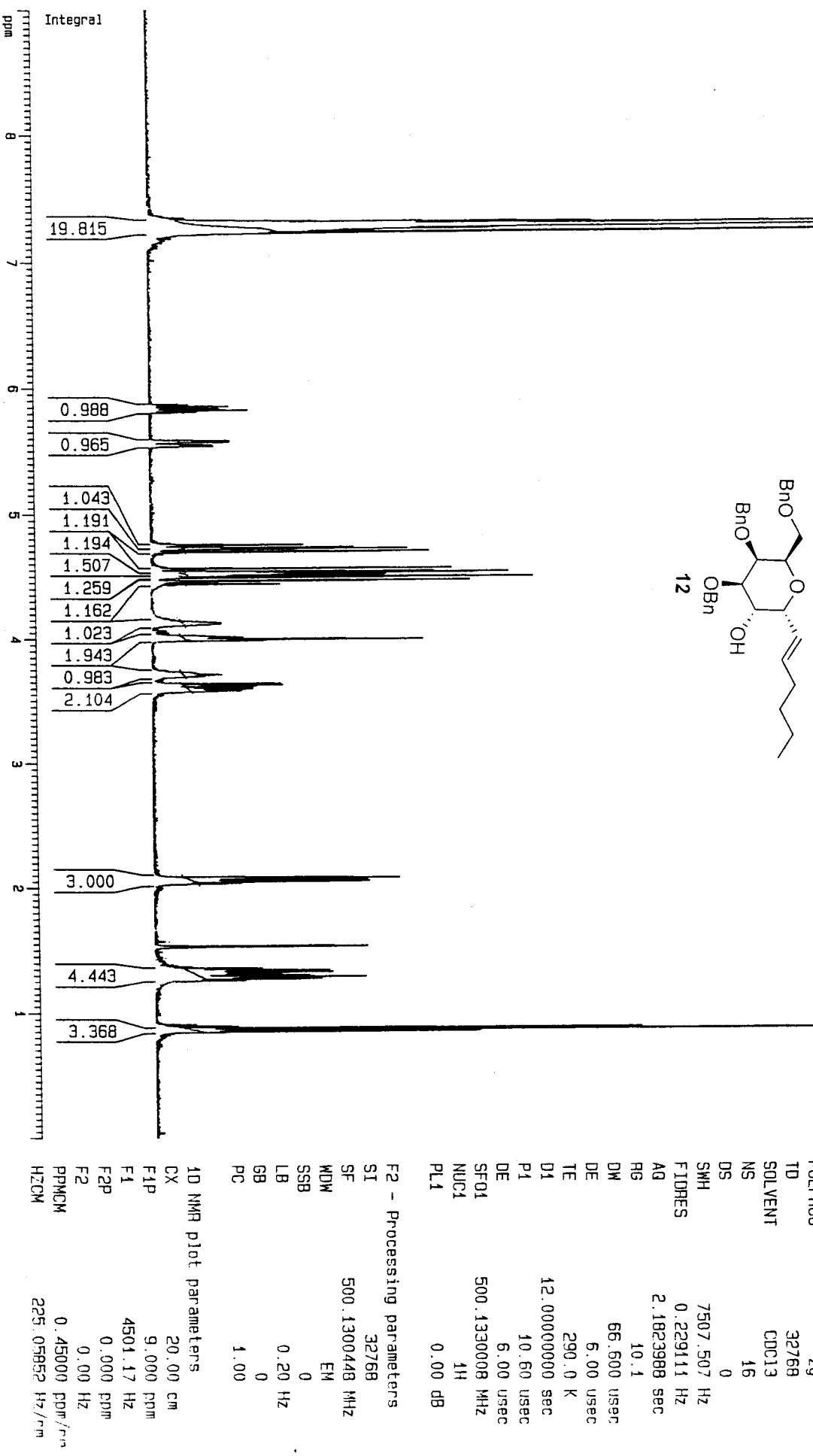
Current Data Parameters	
NAME	jp1_270_P
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
DATE	50000
TIME	22.19
INSTRUM	spect
PROBHD	5 mm T1X13C
PULPROG	zg
TD	32768
SOLVENT	CDCl <sub>3</sub>
NS	16
DS	0
SWH	7507.507 Hz
ETR	0.229111 Hz
AQ	2.1823988 sec
RG	40.3
DW	66.600 usec
DE	6.00 usec
TE	290.0 K
D1	6.0000000 sec
P1	10.60 usec
DE	6.00 usec
SF01	500.1330008 MHz
NUC1	<sup>1</sup> H
PL1	0.00 dB



jp1\_280 1H NMR 500 MHz CDCl<sub>3</sub> 11-19-04

NAME	ppm
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
TD	500000
Time	15.00
INSTRUM	spec
PROBHD	5 mm TXY 13C
PULPROG	zg
TD	32768
SOLVENT	CDCl <sub>3</sub>
NS	16
DS	0
SWH	7507.507 Hz
FIRES	0.22911 Hz
AQ	2.182988 sec
RG	10.1
DW	66.600 usec
DE	6.00 usec
TE	290.0 K
DA	12.000000 sec
P1	10.60 usec
DE	6.00 usec
NUC1	500.133008 MHz
PL1	1H
PL1	0.00 dB

Current Data Parameters
NAME jp280
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 32768
SF 500.1300448 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

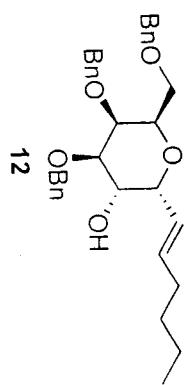
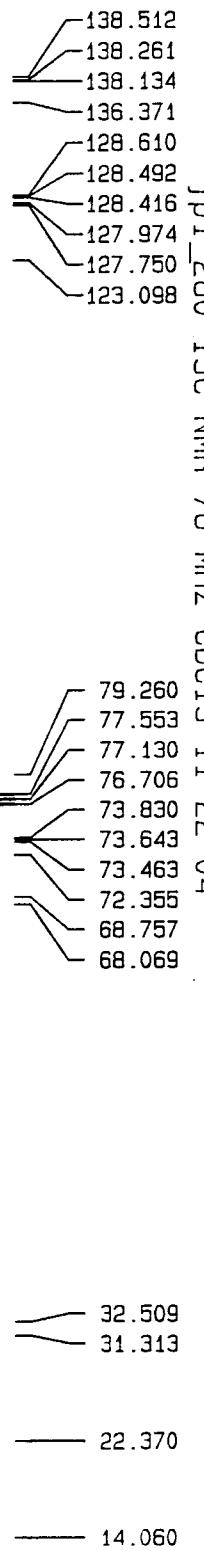


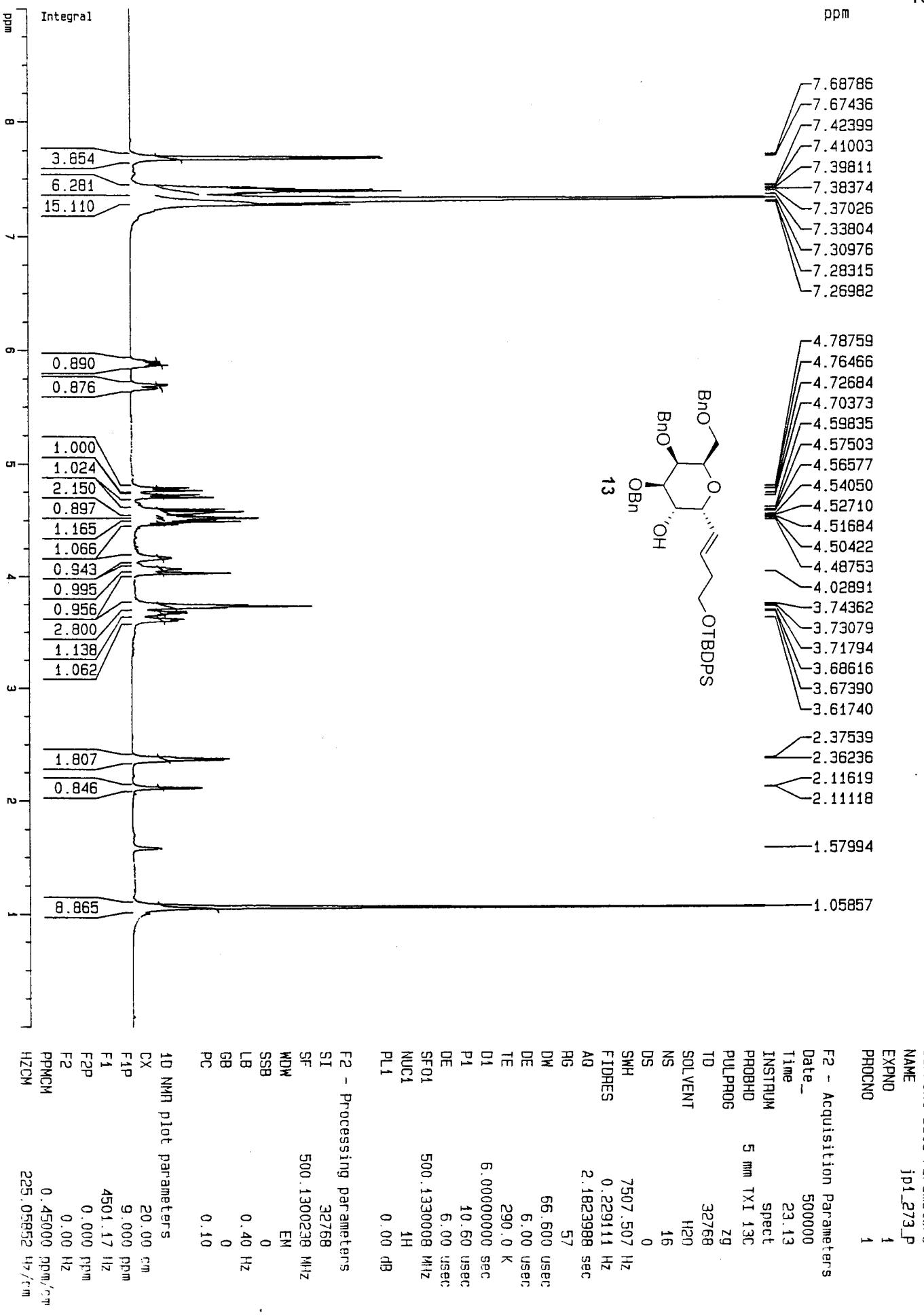
18

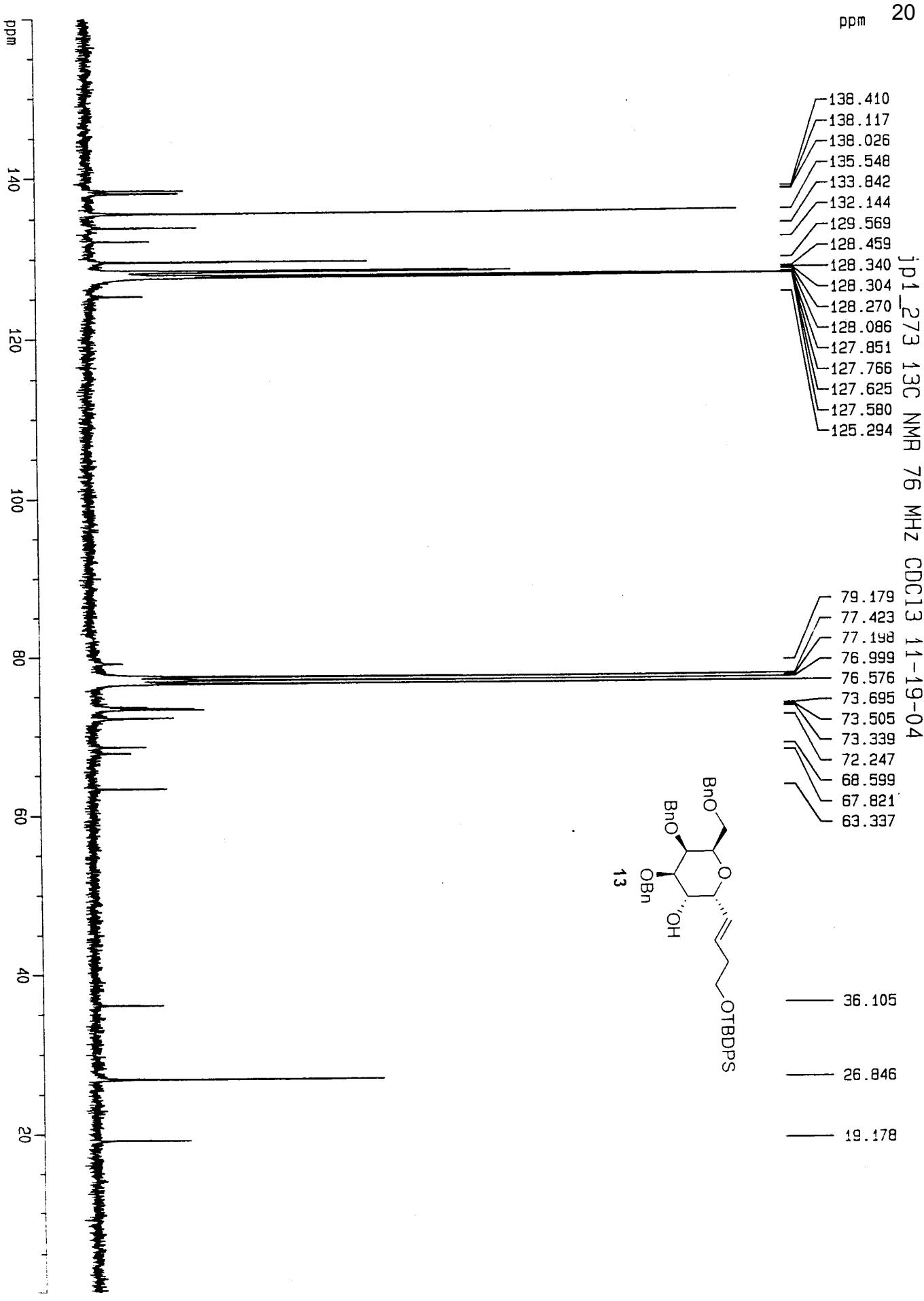
ppm

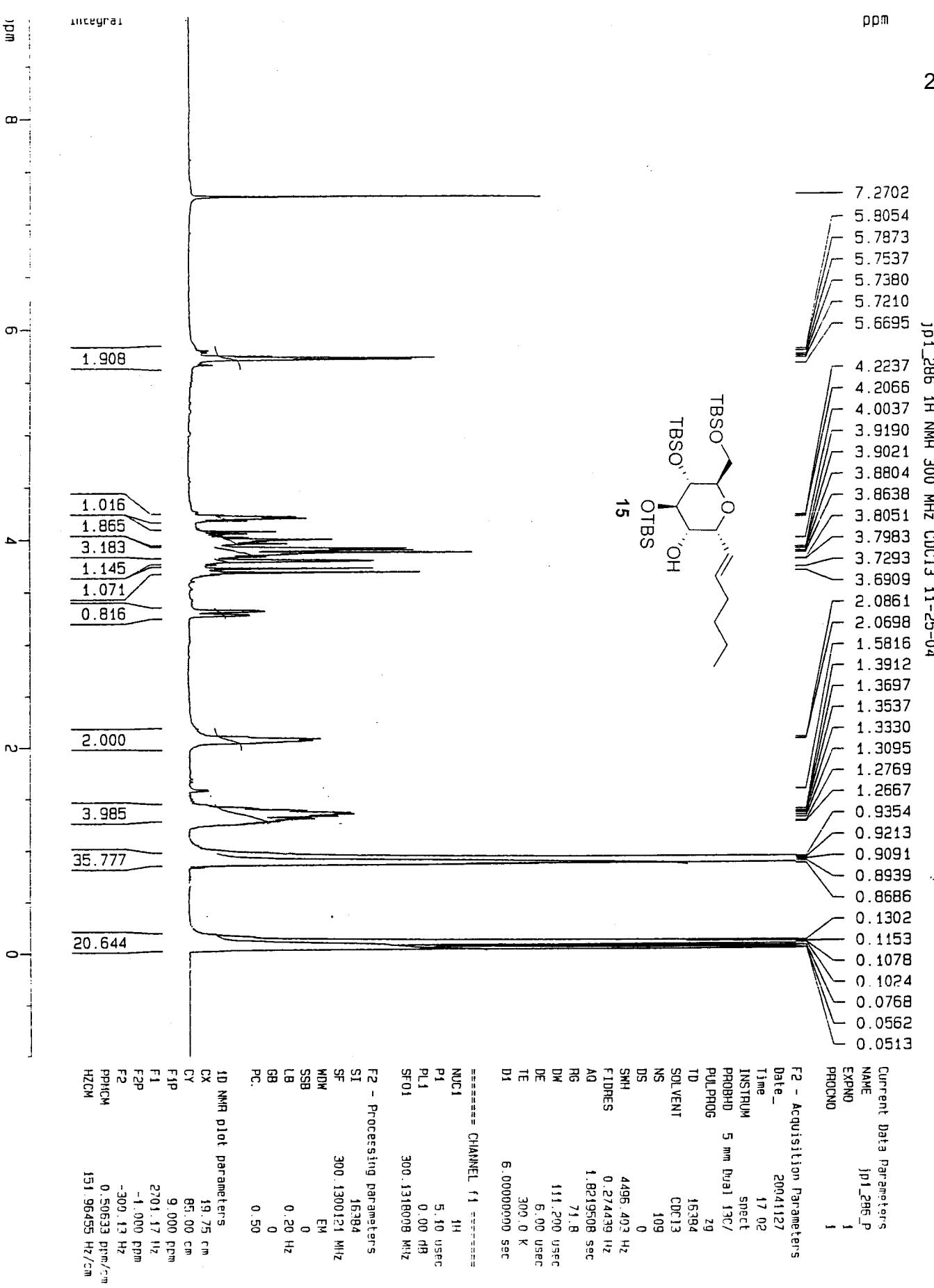
<sup>13</sup>C NMR 76 MHz CDCl<sub>3</sub>

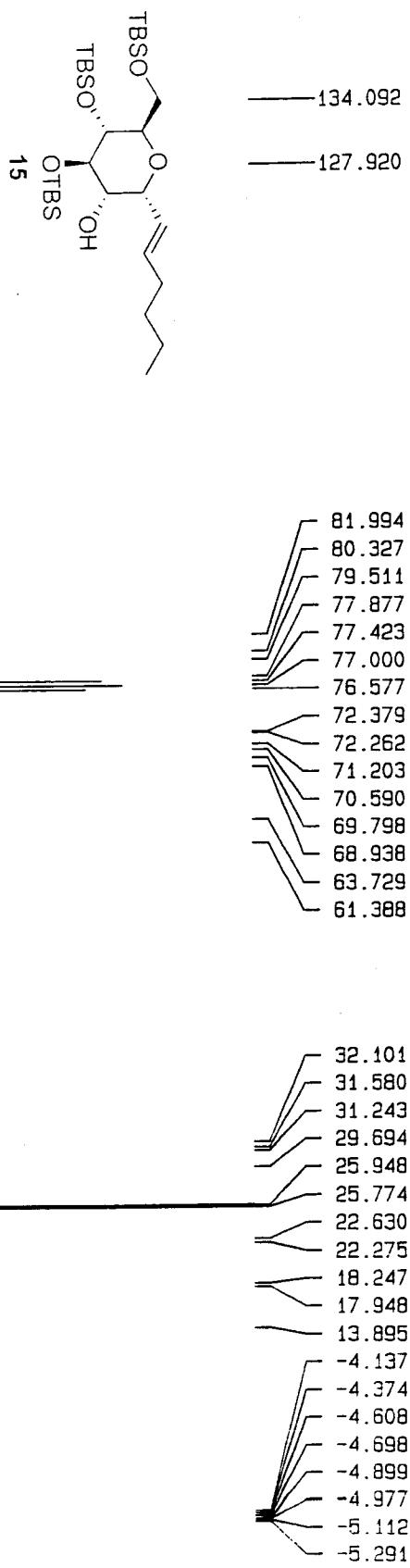
11-22-04



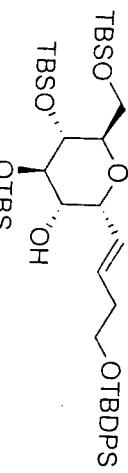
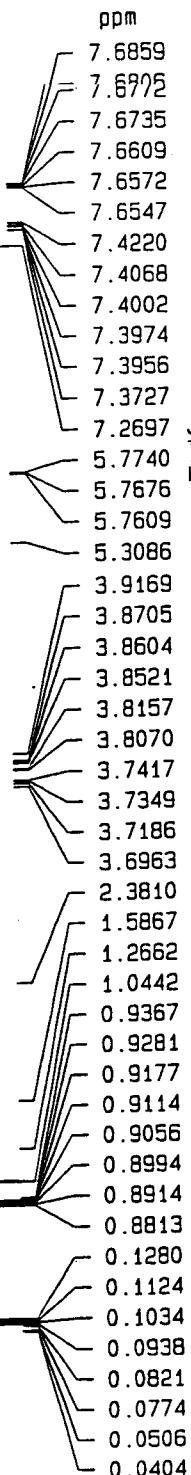
jp1\_273 1H NMR 500 MHz CDCl<sub>3</sub> 11-20-04







jp1\_285 1H NMR 300 MHz CDCl<sub>3</sub> 11-26-04



F2 - Acquisition Parameters  
 Date 20041129  
 Time 9.51  
 INSTRUM spect  
 PROBHD 5 mm Dual 13C/  
 PULPROG zg  
 TD 16384  
 SOLVENT CDCl<sub>3</sub>  
 NS 44  
 DS 0  
 SWH 4495.403 Hz  
 FIDRES 0.274459 Hz  
 AQ 1.8219508 sec  
 RG 71.8  
 DW 111.200 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 6.0000000 sec

===== CHANNEL f1 =====

NUC1 <sup>1</sup>H  
 P1 5.10 usec  
 PL1 0.00 dB  
 SF01 300.1318008 MHz

F2 - Processing parameters

SI 16384  
 SF 300.1300121 MHz  
 HDM EM  
 SSB 0  
 LB 0.20 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 19.75 cm  
 CY 50.00 cm  
 F1P 9.000 ppm  
 F1 2701.17 Hz  
 F2P -1.000 ppm  
 F2 -300.13 Hz  
 FPPCM 0.50633 ppm/cm  
 HZCM 151.96455 Hz/cm

ppm 24

jp1\_285 13C NMR 76 MHz CDCl<sub>3</sub> 11-22-04

