Rh-Catalyzed Enantioselective Diboration of Simple Alkenes: Reaction Development and Substrate Scope

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Supplementary Material

Table of contents

1) General remarks.

^{1}H NMR chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$: 7.24 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), coupling constants (Hz) and assignment. ^{13}C NMR chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal standard ($CDCl_3$: 77.0 ppm).

Liquid chromatography was performed using forced flow (flash chromatography) on silica gel (SiO_2, 32 to 63 μm). Thin layer chromatography (TLC) was performed on 0.25 mm silica gel plates.

Analytical gas-liquid chromatography (GLC) was performed on a Supelco β-dex 120 column with helium as the carrier gas. Analytical high performance liquid chromatography (HPLC) was performed using a Daicel Chiralcel OD-H column. Analytical supercritical fluid chromatography (SFC) was performed using a Daicel Chiralcel OD-H column.

All reactions were conducted in oven and flame dried glassware under an inert atmosphere of argon. Alkene starting materials were all commercially available unless otherwise described. All reagents were used as received.

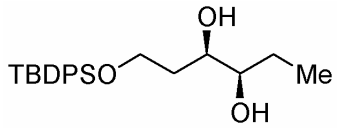
2) Experimental details.

Representative Procedure for Catalytic, Enantioselective Diboration Reaction.

An oven-dried 20 mL vial equipped with a stir-bar was charged with 6.1 mg (0.021 mmol) of (bicyclo[2.2.1]hepta-2,5-diene)-(2,4-pentanedionato)-rhodium (I) ((nbd)Rh(acac)), 9.2 mg (0.021 mmol) of (*S*)-Quinap, and 1.7 mL of THF under an inert atmosphere of argon in a dry-box. The resultant yellow solution was stirred for 5 minutes. After this time, 148 mg (0.62 mmol) of bis(catecholato)diboron was added to the solution under argon. The solution turned immediately from yellow to dark brownish-red. The solution was allowed to stir for 5 minutes. After this time, 84 mg (0.42 mmol) of trans-(7-methyl-octa-1,6-dienyl)-benzene was added to the solution under argon. The vial was sealed with a screw-cap and removed from the dry box, where the solution was allowed to stir for 14 hours at ambient temperature. After this time, the mixture was cooled to 0^{o}C and 1.25 mL of 3 M NaOH and then 0.800 mL of 30% H_2O_2 (dropwise with caution) were added under nitrogen. The solution was allowed to stir at ambient temperature for 6 hours. The solution was then quenched with 2 mL of saturated aqueous $Na_2S_2O_3$ and 10 mL of 1 M NaOH. The mixture was extracted with ethyl acetate (3 x 25 mL) and the combined organic layers were washed with brine (1 x 10 mL). The organic layers were then dried over anhydrous $MgSO_4$, filtered, and the solvent removed by rotary evaporation. The crude material was purified by silica gel chromatography (9 : 1 to 6 : 4 hexanes/ethyl acetate) to provide 63 mg (64%) of pure (1*R,* 2*R*)-7-methyl-1-phenyl-oct-6-ene-1,2-diol.

Compounds from table 4 (entries 1-3, 8-10), table 5 (entry 3) and table 6 (entry 4) were all previously reported[1]. Compounds from table 5 (entries 1-2, 4-7) were also previously reported[2].

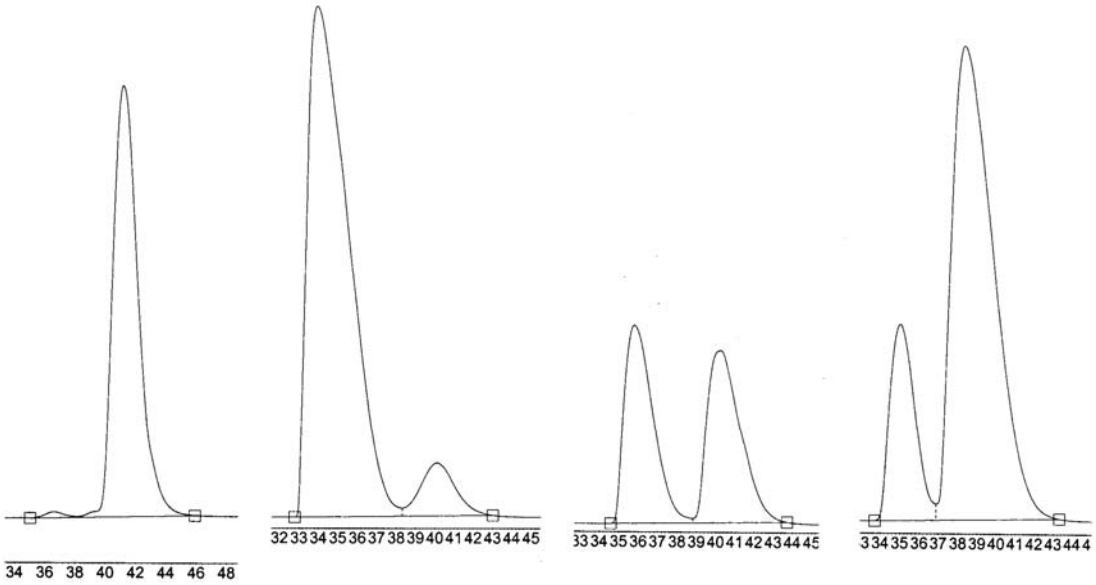
Table 4, entry 4



(3*R*, 4*R*)-1-(*tert*-butyldiphenylsilanyloxy)-hexane-3,4-diol[3]. IR (neat, ν cm^{-1}): 3481, 2964, 2933, 1640, 1472, 1428, 1391; ^{1}H NMR: δ 7.68-7.66 (4H, m), 7.45-7.37 (6H, m), 3.88 (2H, t, *J* = 5.1 Hz), 3.77-3.73 (1H, m), 3.41 (1H, d, *J* = 3.4 Hz), 3.39-3.33 (1H, m), 2.58 (1H, d, *J* = 5.4 Hz), 1.84-1.77 (1H, m), 1.71-1.64 (1H, m), 1.59-1.44 (2H, m), 1.05 (9H, s), 0.98 (3H, t, *J* = 7.4 Hz); ^{13}C NMR: δ 135.5, 132.8, 129.9, 127.8, 75.8, 73.5, 62.8, 35.1, 26.8, 26.3, 19.0, 10.1.

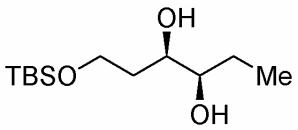
Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by osmium tetraoxide catalyzed dihydroxylation. Absolute stereochemistry established in comparison to authentic (3*S*, 4*S*) isomer prepared via a Sharpless asymmetric dihydroxylation (Becker, H.; King, S. B.; Taniguchi, M.; Vanhessche, K.; Sharpless, K. B. *J. Org. Chem.* **1995**, *60*, 3940).

SFC (OD-H, 150 psi, 40 ºC, flow = 2 mL/min, 1.5% MeOH) analysis of the product:



Diboration product	Authentic	Racemic	Racemic + Diboration product

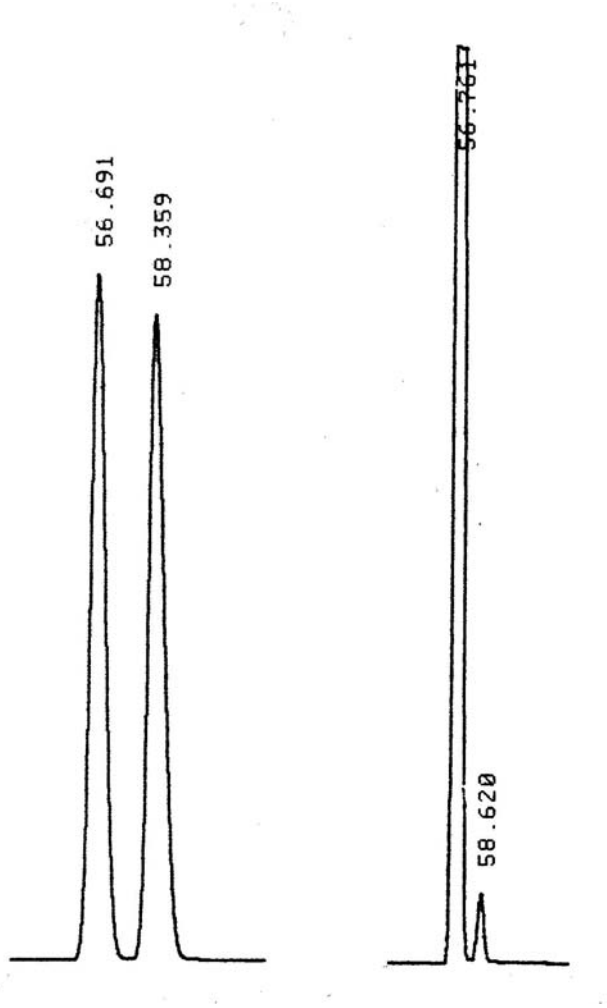
Table 4, entry 5



(3*R*, 4*R*)-1-(*tert*-butyldimethylsilanyloxy)-hexane-3,4-diol[4]. IR (neat, ν cm^{-1}): 3481, 2958, 2933, 1640, 1472, 1389; ^{1}H NMR: δ 3.90-3.80 (2H, m), 3.70-3.65 (1H, m), 3.50 (1H, d, *J* = 3.4 Hz), 3.36-3.31 (1H, m), 2.54 (1H, d, *J* = 5.5 Hz), 1.81-1.72 (1H, m), 1.69-1.62 (1H, m), 1.57-1.42 (2H, m), 0.96 (3H, t, *J* = 7.5 Hz), 0.87 (9H, s), 0.06 (6H, s); ^{13}C NMR: δ 75.7, 73.8, 62.0, 35.2, 26.3, 25.8, 18.1, 10.1, -5.6.

Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by osmium tetraoxide catalyzed dihydroxylation.

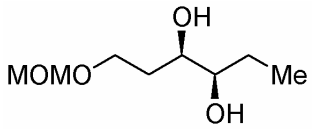
Chiral GLC (β-dex, Supelco, 100°C, 20 psi) analysis of the product:



Racemic

Diboration product

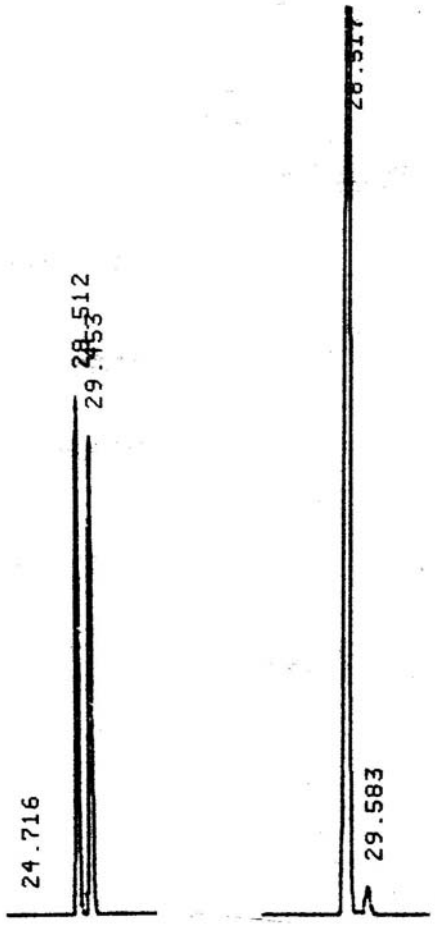
Table 4, entry 6



(3*R*, 4*R*)-1-methoxymethoxy-hexane-3,4-diol[5]. IR (neat, ν cm^{-1}): 3483, 3056, 2989, 2968, 2939, 1640, 1465, 1422; ^{1}H NMR: δ 4.62 (2H, s), 3.78-3.71 (2H, m), 3.67-3.63 (1H, m), 3.39-3.33 (1H, m), 3.36 (3H, s), 2.85 (1H, d, *J* = 4.0 Hz), 2.30 (1H, d, *J* = 5.3 Hz), 1.83-1.77 (2H, m), 1.60-1.42 (2H, m), 0.98 (3H, t, *J* = 7.5 Hz); ^{13}C NMR: δ 96.6, 75.8, 72.8, 65.8, 55.4, 33.2, 26.4, 10.1.

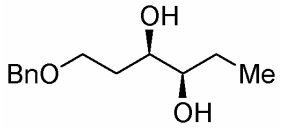
Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by osmium tetraoxide catalyzed dihydroxylation.

Chiral GLC (β-dex, Supelco, 100°C, 20 psi) analysis of the product:



Racemic Diboration product

Table 4, entry 7

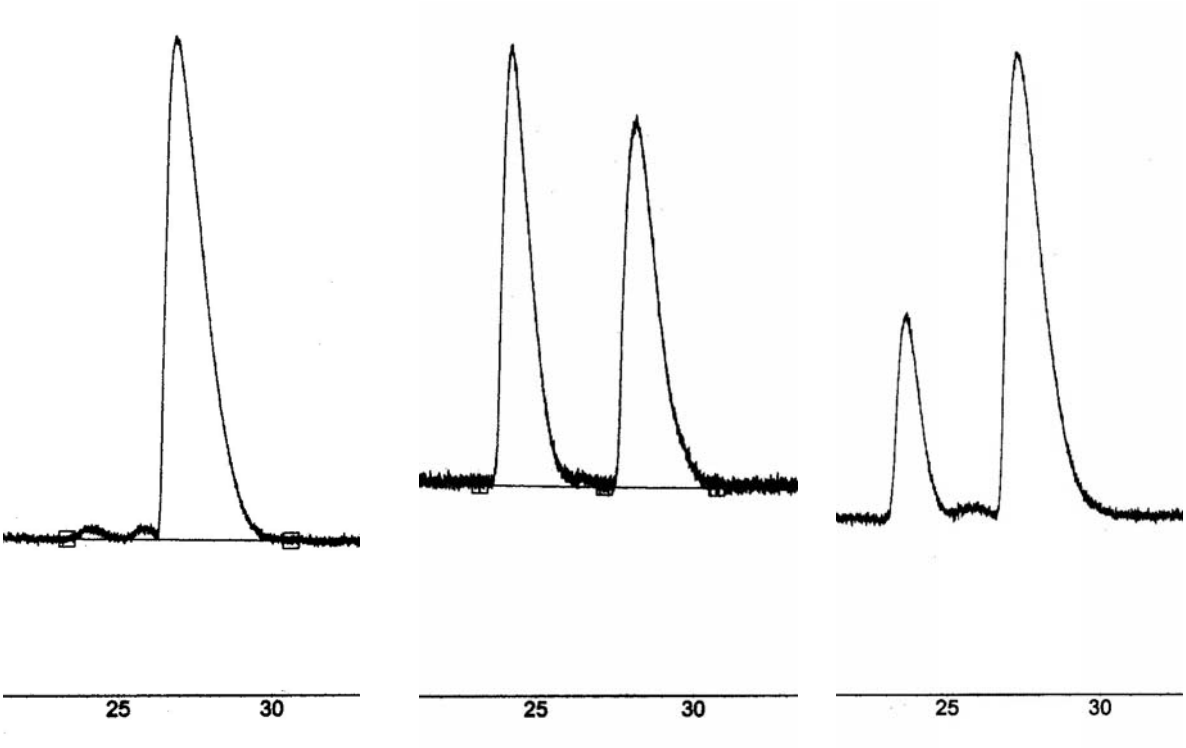


(3*R*, 4*R*)-1-benzyloxy-hexane-3,4-diol[6]. IR (neat, ν cm^{-1}): 3481, 3056, 2968, 2937, 2877, 1640, 1455, 1422, 1364; ^{1}H NMR: δ 7.36-7.28 (5H, m), 4.51 (2H, s), 3.74-3.65

(3H, m), 3.36-3.31 (1H, m), 3.09 (1H, d, J = 3.7 Hz), 2.41 (1H, d, J = 5.3 Hz), 1.91-1.82 (1H, m), 1.79-1.72 (1H, m), 1.60-1.41 (2H, m), 0.97 (3H, t, J = 7.5 Hz); ^{13}C NMR: δ 137.7, 128.5, 127.8, 127.7, 75.7, 73.4, 73.1, 68.5, 33.2, 26.3, 10.1.

Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by osmium tetraoxide catalyzed dihydroxylation.

SFC (OD-H, 150 psi, 40 ºC, flow = 2 mL/min, 2.0% MeOH) analysis of the product:

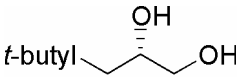


Diboration product

Racemic

Racemic + Diboration product

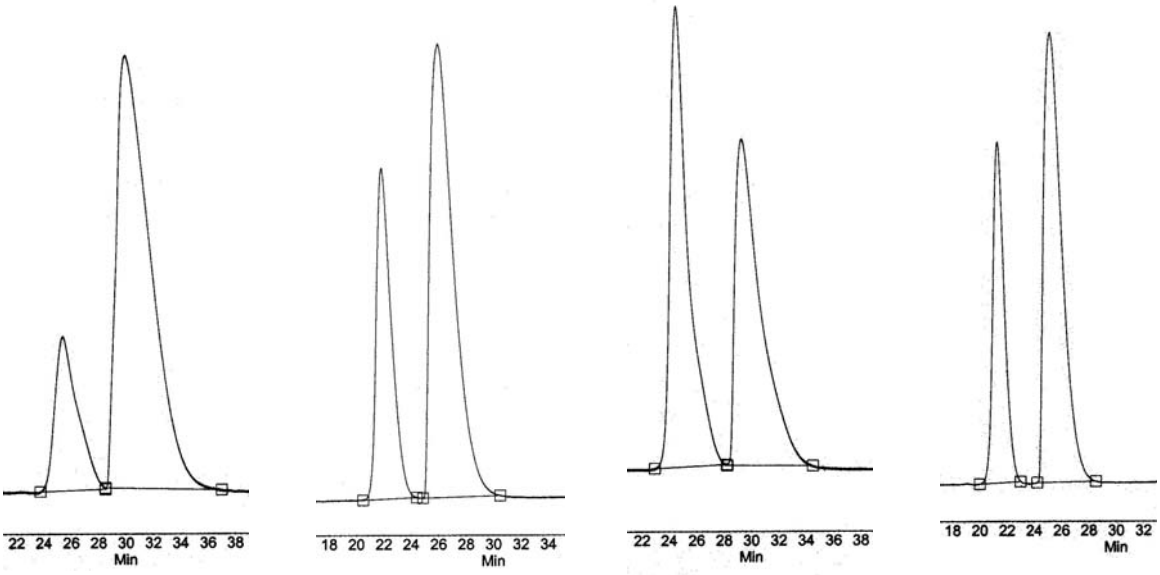
Table 5, entry 8



(2*S*)-4,4-dimethylpentane-1,2-diol. IR (neat, ν cm^{-1}): 3365, 2954, 2871, 1468, 1366, 1088, 1042; ^{1}H NMR: δ 3.79 (1H, m), 3.51 (1H, dd, *J* = 11.1, 3.2 Hz), 3.33 (1H, dd, *J* = 11.1, 8.4 Hz), 3.14 (2H, s), 1.31 (1H, dd, *J* = 14.6, 7.7 Hz), 1.20 (1H, dd, *J* = 14.5, 2.9 Hz), 0.93 (9H, s); ^{13}C NMR: δ 69.9, 68.0, 46.8, 30.0, 30.0; HRMS (CI) $(M+NH_4)^+$ calc'd for $C_7H_{20}NO_2$: 150.1489. Found: 150.1497.

Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by osmium tetraoxide catalyzed dihydroxylation. Absolute stereochemistry established in comparison to authentic (2*S*) isomer prepared via a Sharpless asymmetric dihydroxylation (Becker, H.; King, S. B.; Taniguchi, M.; Vanhessche, K.; Sharpless, K. B. *J. Org. Chem.* **1995**, *60*, 3940).

SFC (OD-H, 150 psi, 50 ºC, flow = 3 mL/min, 0% MeOH) analysis of the benzoate diester (BzCl / cat. DMAP, pyr) product:



Diboration product

Authentic

Racemic

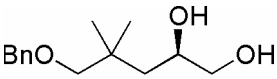
Racemic + Diboration product

Table 5, entry 9 (substrate)

1-((2,2-dimethylpent-4-enyloxy)methyl)benzene. Sodium hydride (60% in mineral oil) (67 mg, 1.68 mmol) was added to a round bottom flask and washed three times with pentane. THF (3.0 mL) was then added and the resulting suspension was cooled to 0°C. A solution of 2,2-dimethylpent-4-en-1-ol[7] (101 mg, 0.88 mmol) in THF (3.0 mL) was then added dropwise and the mixture was stirred 1h30 at room temperature. Benzyl bromide (0.16 mL, 1.33 mmol) and tetrabutylammonium iodide (33 mg, 8.8 μmol) were then added and mixture was stirred 16h at room temperature. A saturated NH_4Cl aqueous

solution (10 mL) was then added and mixture was extracted three times with Et_2O. The combined organic phases were dried with $MgSO_4$, filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography (100/0 to 95/5: hex/EtOAc) to yield the product (0.16 g, 88%) as a clear oil. IR (neat, ν cm^{-1}): 2958, 2860, 1102; ^{1}H NMR: δ 7.40-7.26 (5H, m), 5.87-5.76 (1H, m), 5.05-5.04 (1H, m), 5.04-5.00 (1H, m), 4.52 (2H, s), 3.15 (2H, s), 2.08 (2H, td, *J* = 7.5, 1.1 Hz), 0.93 (6H, s); ^{13}C NMR: δ 139.1, 135.4, 128.2, 127.3, 127.3, 116.9, 79.1, 73.2, 43.7, 34.9, 24.5.

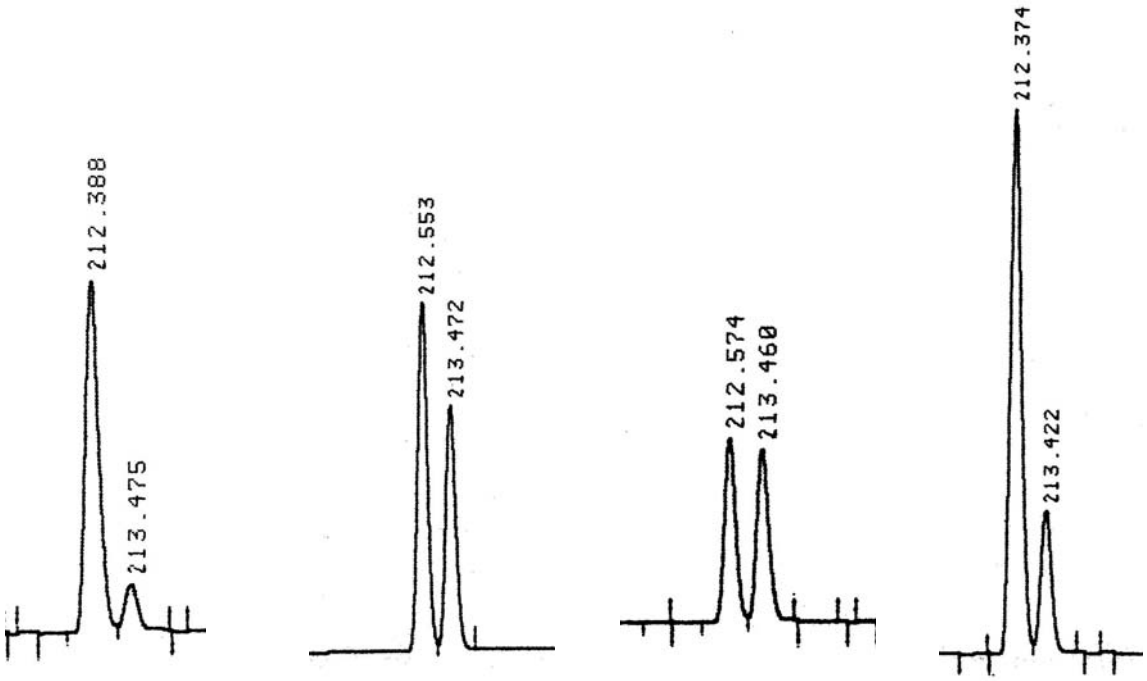
Table 5, entry 9



(2*R*)-5-(benzyloxy)-4,4-dimethylpentane-1,2-diol. IR (neat, ν cm^{-1}): 3394, 2954, 2929, 2871, 1096; ^{1}H NMR: δ 7.36-7.26 (5H, m), 4.52 (2H, d, *J* = 1.4 Hz), 3.82-3.76 (1H, m), 3.49 (1H, dd, *J* = 10.9, 3.5 Hz), 3.41-3.36 (3H, m), 3.27 (1H, d, *J* = 9.0 Hz), 3.23 (1H, dd, *J* = 9.0, 0.2 Hz), 1.47 (1H, dd, *J* = 14.8, 9.7 Hz), 1.32 (1H, dd, *J* = 14.7, 1.3 Hz), 0.99 (3H, s), 0.90 (3H, s); ^{13}C NMR: δ 137.8, 128.9, 128.3, 128.2, 80.0, 74.0, 68.8, 68.1, 45.2, 34.5, 28.4, 24.2; MS (ESI) $(M+Na)^+$ calc'd for $C_{14}H_{22}O_3Na$: 261.1. Found: 261.1.

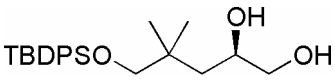
Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by osmium tetraoxide catalyzed dihydroxylation. Absolute stereochemistry established in comparison to authentic (2*R*) isomer prepared via a Sharpless asymmetric dihydroxylation (Becker, H.; King, S. B.; Taniguchi, M.; Vanhessche, K.; Sharpless, K. B. *J. Org. Chem.* **1995**, *60*, 3940).

Chiral GLC (β-dex, Supelco, 120°C 178 min then ↑1°C/min to 180°C, 20 psi) analysis of the acetonide (dimethoxypropane/cat. pTsOH) product:



Diboration product | Authentic | Racemic | Racemic + Diboration Product

Table 5, entry 10

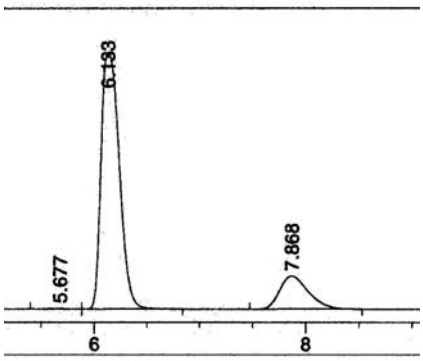


(2*R*)-5-(*tert*-butyldiphenylsilyloxy)-4,4-dimethylpentane-1,2-diol[8]. IR (neat, ν cm^{-1}): 3381, 2958, 2931, 2894, 2858, 1111, 1090; ^{1}H NMR: δ 7.67-7.64 (4H, m), 7.45-7.36 (6H, m), 3.89-3.84 (1H, m), 3.54 (1H, dd, *J* = 10.9, 3.5 Hz), 3.45-3.38 (3H, m), 3.15 (2H, br s), 1.51 (1H, dd, *J* = 14.7, 9.4 Hz), 1.35 (1H, dd, *J* = 14.7, 1.7 Hz), 1.08 (9H, s), 0.90 (3H,

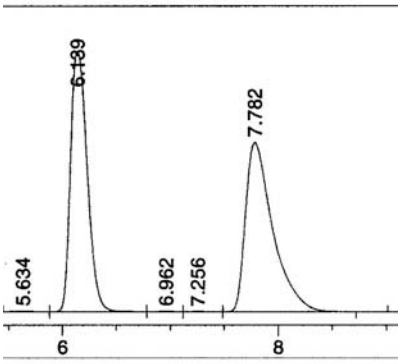
s), 0.80 (3H, s); ^{13}C NMR: δ 135.8, 135.7, 132.8, 132.7, 129.9, 129.8, 127.7, 127.7, 72.7, 68.6, 67.8, 44.0, 35.1, 26.9, 23.7, 19.3; MS (ESI) $(M+Na)^+$ calc'd for $C_{23}H_{34}O_3SiNa$: 409.2. Found: 409.2.

Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by osmium tetraoxide catalyzed dihydroxylation. Absolute stereochemistry established in comparison to authentic (2*R*) isomer prepared via a Sharpless asymmetric dihydroxylation (Becker, H.; King, S. B.; Taniguchi, M.; Vanhessche, K.; Sharpless, K. B. *J. Org. Chem.* **1995**, *60*, 3940).

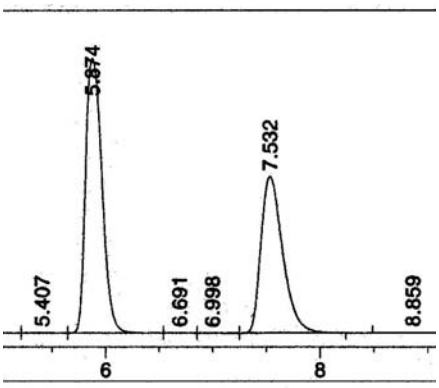
Chiral HPLC (Chiralcel OD-H, Daicel, 0.1% iPrOH in hexanes, 1.0 mL/min, wavelength: 220 nm) analysis of the acetonide (dimethoxypropane/cat. pTsOH) product:



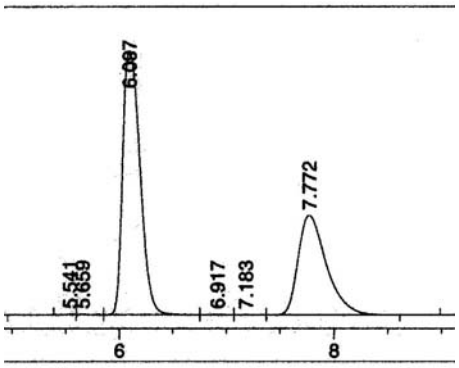
Diboration product



Racemic

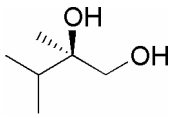


Authentic



Racemic + Diboration product

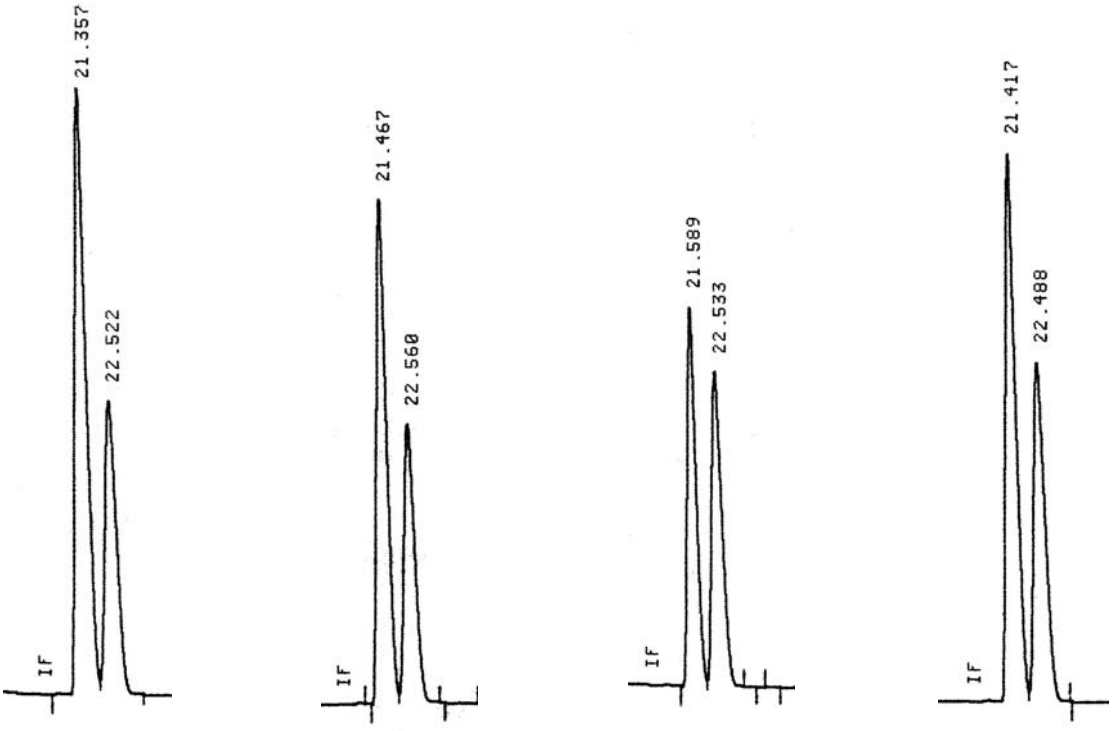
Table 6, entry 2



(2*R*)-2,3-dimethylbutane-1,2-diol. IR (neat, ν cm^{-1}): 3398, 2964, 2879; ^{1}H NMR: δ 3.43 (2H, dd, *J* = 48.7, 11.1 Hz), 3.10 (2H, br s), 1.77 (1H, sp, *J* = 6.9 Hz), 1.00 (3H, s), 0.90 (3H, d, *J* = 6.9 Hz), 0.82 (3H, d, *J* = 6.9 Hz); ^{13}C NMR: δ 75.3, 68.3, 34.1, 18.7, 17.6, 16.6; MS (ESI) $(M+Na)^{+}$ calc'd for $C_6H_{14}O_2Na$: 141.1. Found: 141.1.

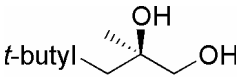
Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by osmium tetraoxide catalyzed dihydroxylation. Absolute stereochemistry established in comparison to authentic (2*R*) isomer prepared via a Sharpless asymmetric dihydroxylation (Becker, H.; King, S. B.; Taniguchi, M.; Vanhessche, K.; Sharpless, K. B. *J. Org. Chem.* **1995**, *60*, 3940).

Chiral GLC (β-dex, Supelco, 60°C, 20 psi) analysis of the acetonide (dimethoxypropane /cat. pTsOH) product:



Diboration product | Authentic | Racemic | Racemic + Diboration Product

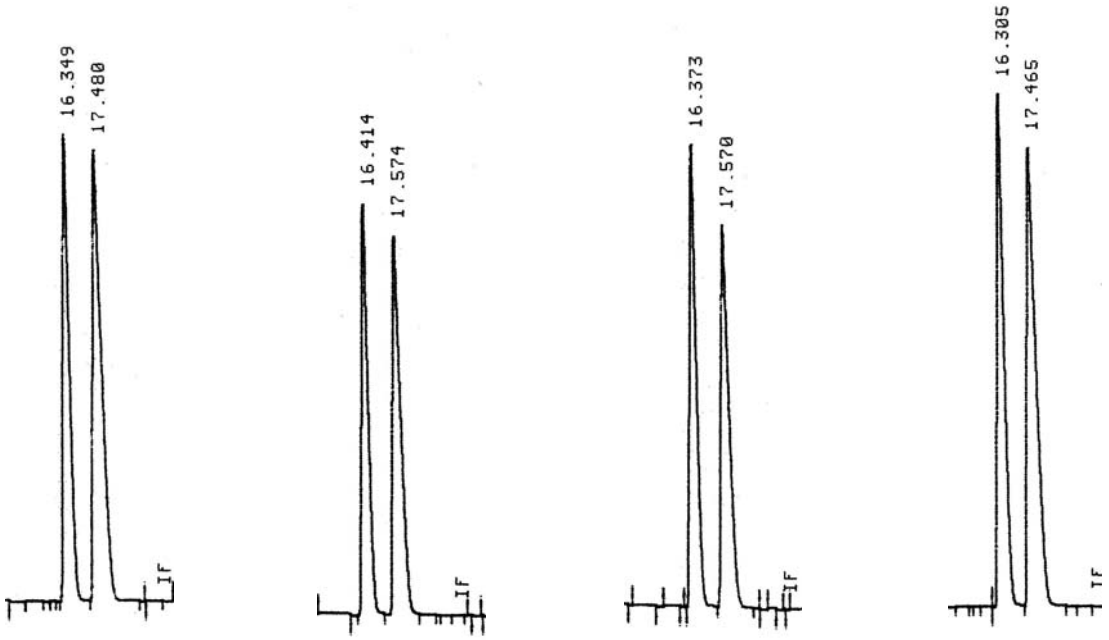
Table 6, entry 3



(2*R*)-2,4,4-trimethylpentane-1,2-diol. IR (neat, ν cm^{-1}): 3398, 2964, 2879; ^{1}H NMR: δ 3.36 (2H, dd, *J* = 41.0, 10.8 Hz), 2.58 (2H, br s), 1.44 (2H, dd, *J* = 48.2, 14.8 Hz), 1.24 (3H, s), 1.00 (9H, s); ^{13}C NMR: δ 74.2, 73.1, 50.7, 31.5, 31.1, 24.8; HRMS (ESI) $(M+Na)^{+}$ calc'd for $C_8H_{18}O_2Na$: 169.1199. Found: 169.1202.

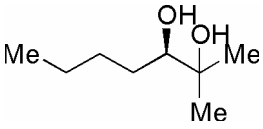
Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by osmium tetraoxide catalyzed dihydroxylation. Absolute stereochemistry established in comparison to authentic (2*R*) isomer prepared via a Sharpless asymmetric dihydroxylation (Becker, H.; King, S. B.; Taniguchi, M.; Vanhessche, K.; Sharpless, K. B. *J. Org. Chem.* **1995**, *60*, 3940).

Chiral GLC (β-dex, Supelco, 80°C, 20 psi) analysis of the acetonide (dimethoxypropane /cat. pTsOH) product:



Diboration product | Authentic | Racemic | Racemic + Diboration product

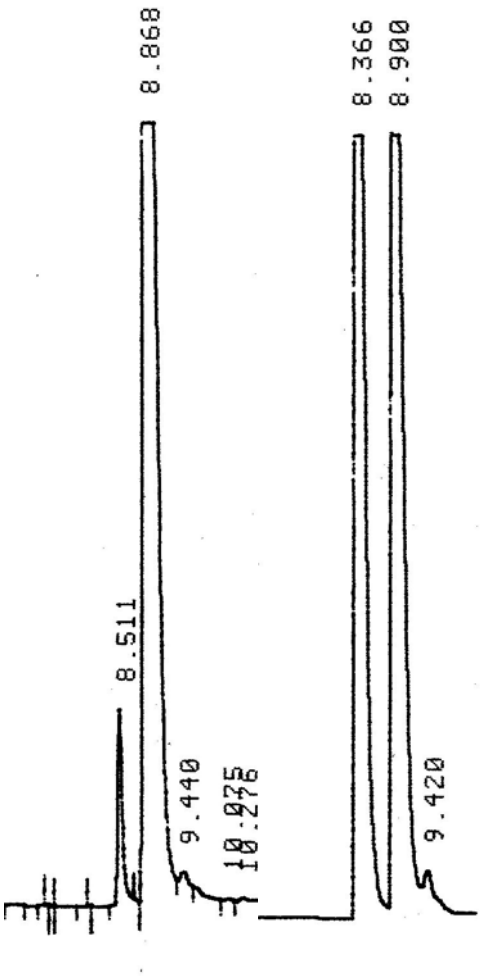
Table 6, entry 6



(3*R*)-2-methyl-heptane-2,3-diol. IR (neat, ν cm^{-1}): 3394, 2958, 2933, 2873, 2861, 1466, 1380, 1167, 1071; ^{1}H NMR: δ 3.35 (1H, dd, *J* = 10.1, 2.3 Hz), 1.66-1.22 (8H, m), 1.19 (3H, s), 1.14 (3H, s), 0.890 (3H, t, *J* = 7.3 Hz); ^{13}C NMR: δ 78.6, 73.1, 31.4, 28.9, 26.5, 23.1, 22.7, 14.0; MS (ESI) $(M+Na)^{+}$ calc'd for $C_8H_{18}O_2Na$: 169.1. Found: 169.1.

Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by osmium tetraoxide catalyzed dihydroxylation.

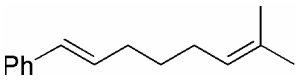
Chiral GLC (β-dex, Supelco, 130°C, 20 psi) analysis of the product:



Diboration product

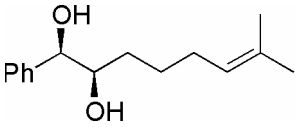
Racemic

Scheme 3 (substrate)



***trans*-(7-methyl-octa-1,6-dienyl)-benzene.** To a stirred suspension of isopropyl(triphenyl)phosphonium bromide (1.28 g, 3.33 mmol) in THF (6.0 mL) at 0°C was added a solution of (*E*)-6-phenyl-hex-5-enal[3] (0.29 g, 1.66 mmol) in THF (5.0 mL). The resulting mixture was stirred 0.5h at 0°C and 0.5h at room temperature. Water was then added and mixture was extracted three times with Et_2O. The combined organic phases were dried with $MgSO_4$, filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography (100/0 to 80/0: hex/EtOAc) to yield the product (0.17 g, 50%) as a clear oil. IR (neat, ν cm^{-1}): 2927, 2856, 1449; ^{1}H NMR: δ 7.36-7.17 (5H, m), 6.39 (1H, d, *J* = 15.8 Hz), 6.22 (1H, dt, *J* = 15.8, 6.9 Hz), 5.17-5.13 (1H, m), 2.22 (2H, q, *J* = 6.9 Hz), 2.04 (2H, q, *J* = 7.4 Hz), 1.71 (3H, s), 1.62 (3H, s), 1.52 (2H, qu, *J* = 7.3 Hz); ^{13}C NMR: δ 138.0, 131.6, 131.0, 129.9, 128.4, 126.7, 125.9, 124.4, 32.6, 29.5, 27.6, 25.7, 17.7.

Scheme 3 (product)

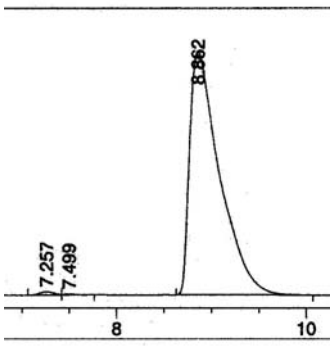


(1*R*, 2*R*)-7-methyl-1-phenyl-oct-6-ene-1,2-diol. IR (neat, ν cm^{-1}): 3375, 2927, 2860, 1455, 1081, 1054, 1025; ^{1}H NMR: δ 7.35-7.25 (5H, m), 5.03-4.98 (1H, m), 4.38 (1H, d, *J* = 6.8 Hz), 3.66-3.60 (1H, m), 2.78 (2H, br s), 1.92-1.85 (2H, m), 1.62 (3H, s), 1.52 (3H, s), 1.51-1.25 (4H, m); ^{13}C NMR: δ 141.3, 131.6, 128.4, 128.0, 126.8, 124.3, 77.8, 75.9,

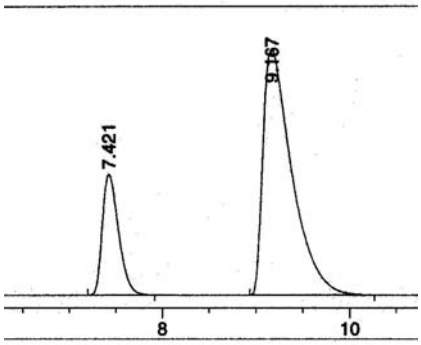
32.3, 27.8, 25.8, 25.6, 17.6; MS (ESI) $(M+Na)^+$ calc'd for $C_{15}H_{22}O_2Na$: 256.9. Found: 256.9.

Proof of Stereochemistry. Stereochemical ratios were determined in comparison to authentic racemic materials prepared by diboration with racemic QUINAP followed by oxidation.

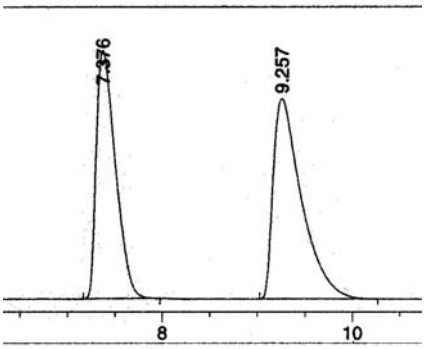
Chiral HPLC (Chiralcel OD-H, Daicel, 0.1% iPrOH in hexanes, 1.0 mL/min, wavelength: 220 nm) analysis of the acetonide (dimethoxypropane/cat. pTsOH) product:



Diboration product

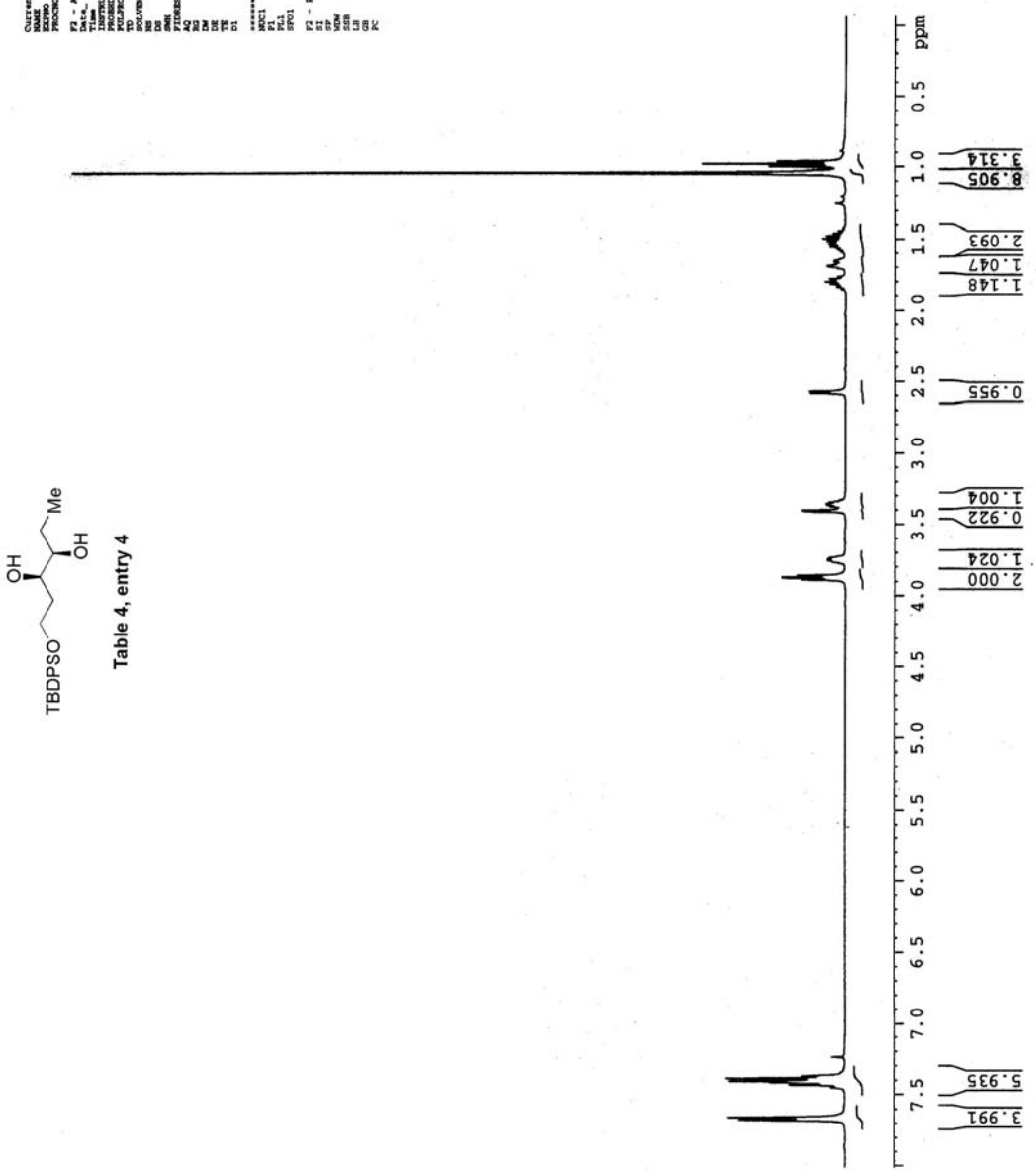


Racemic + Diboration product



Racemic

3) ^{1}H and ^{13}C NMR spectra



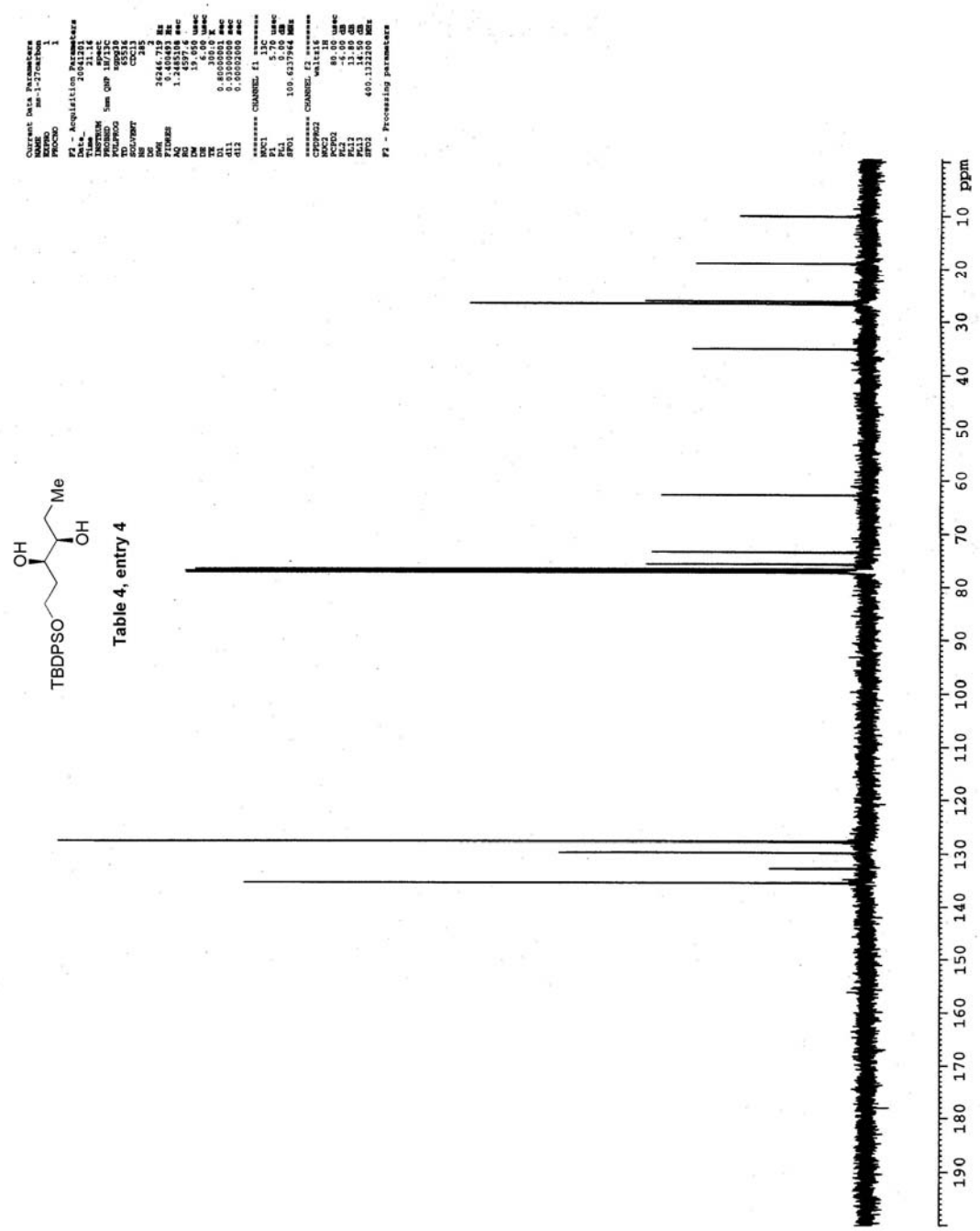


Table 4, entry 4
TBDPSO
OH
OH
Me
ppm

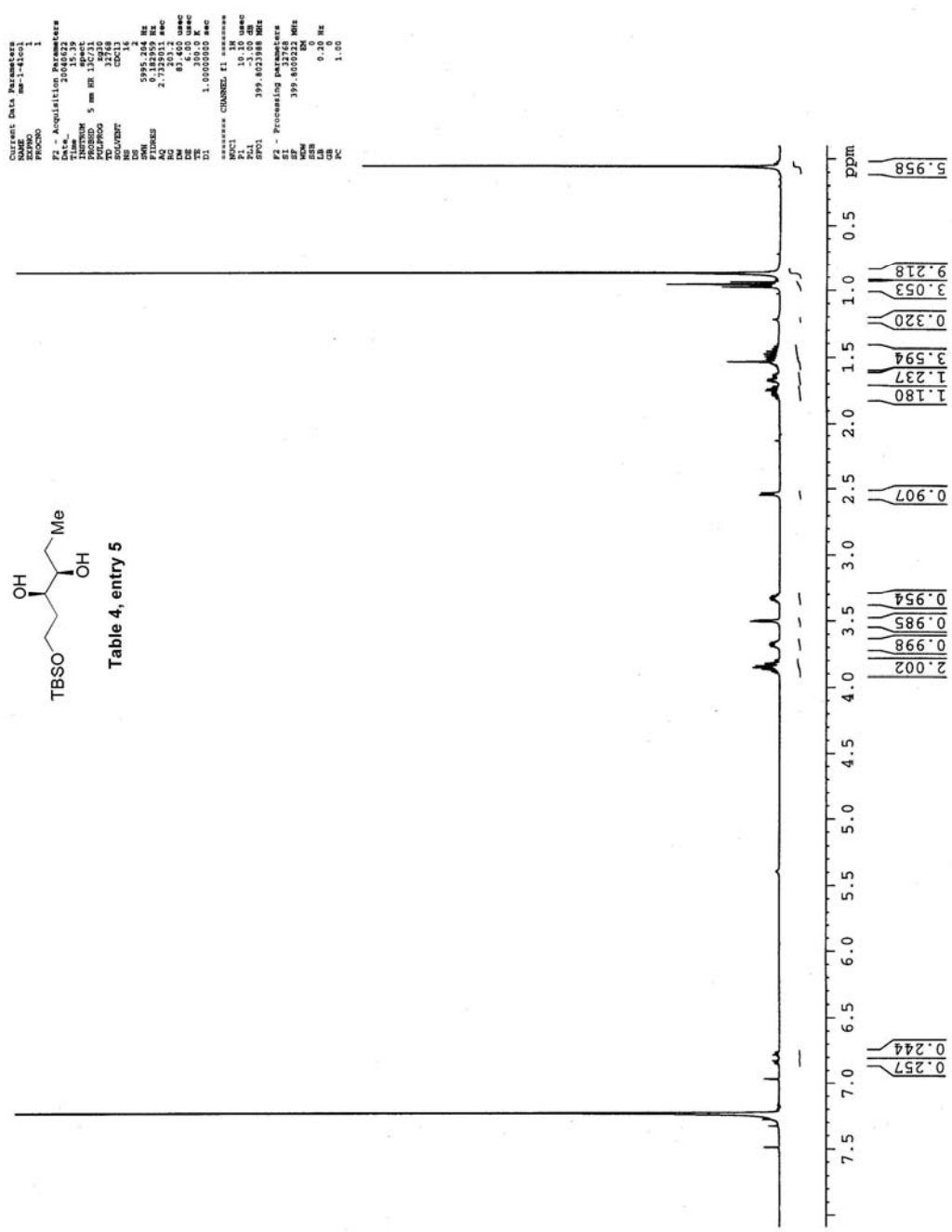
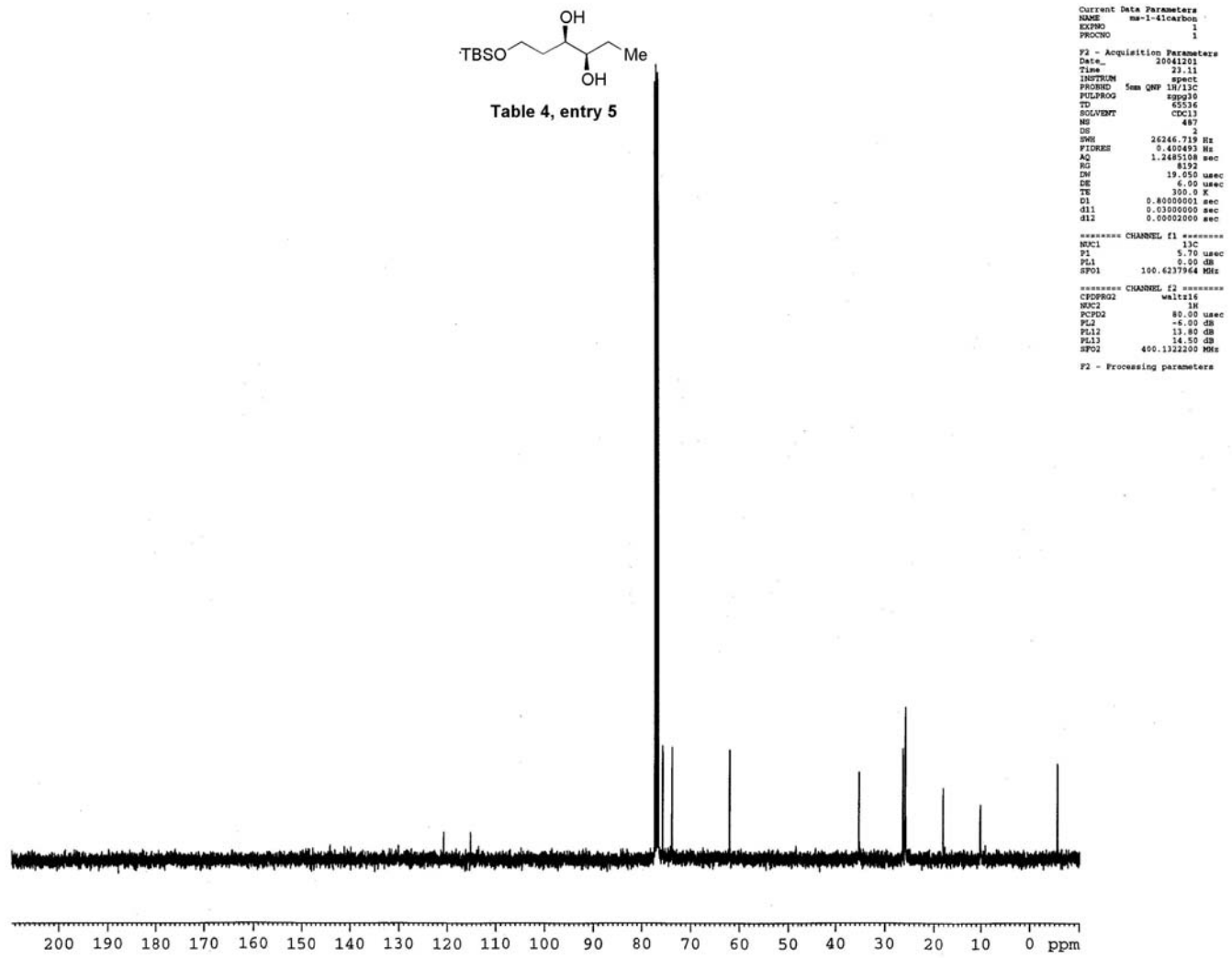
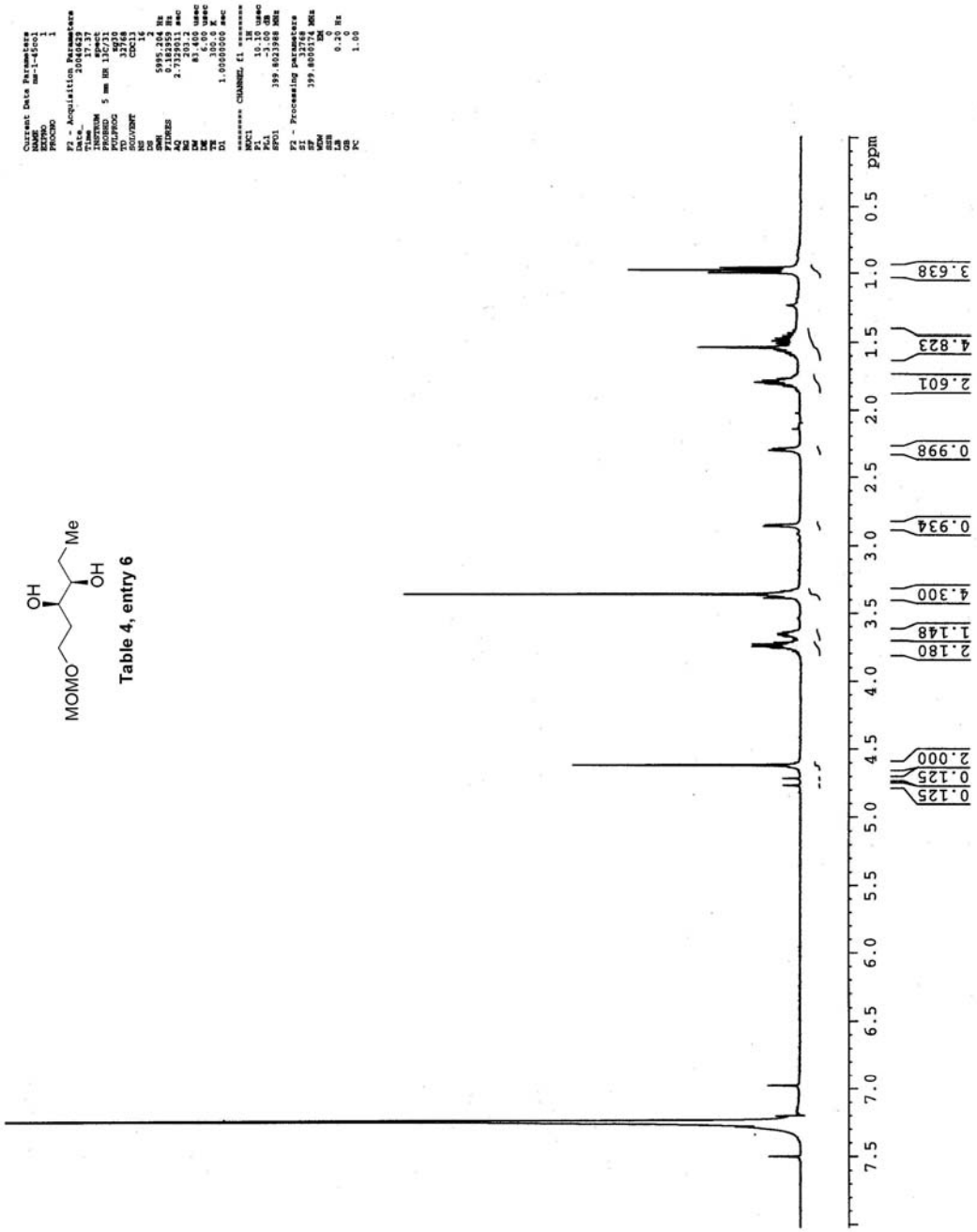
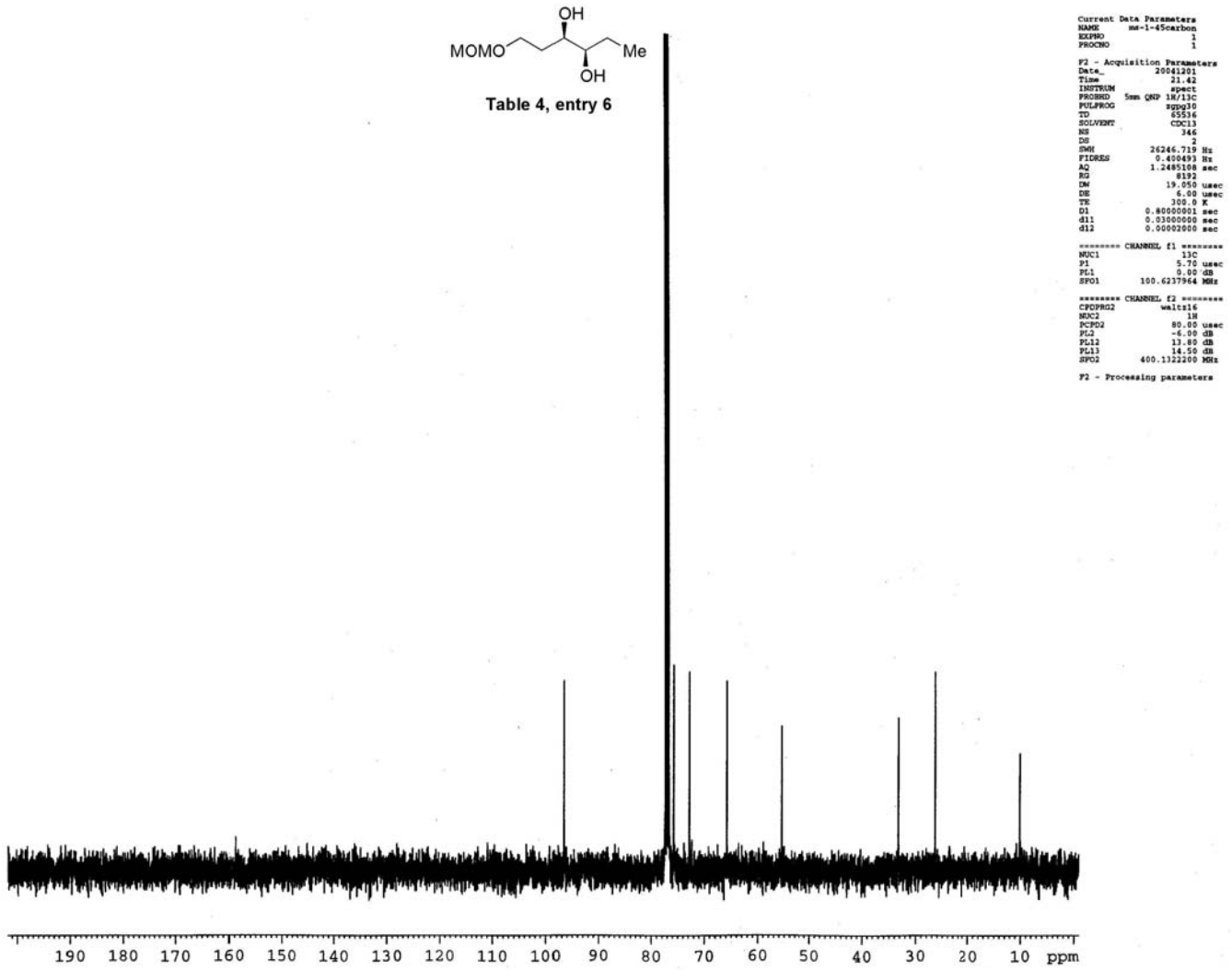


Table 4, entry 5
TBSO
OH
OH
Me
ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.257
0.244
2.002
0.998
0.985
0.954
0.907
1.180
1.237
3.594
0.320
3.053
9.218
5.958


OH
TBSO
Me
OH
Table 4, entry 5
Current Data Parameters
NAME ns-1-41carbon
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20941201
Time 23.11
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 487
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.70 usec
PL1 0.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.1322200 MHz
F2 - Processing parameters
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Current Data Parameters
NAME mm-1-45c01
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161117
Time 17.37
INSTRUM spect
PROBHD 5 mm BB 126/71
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.183000 Hz
AQ 2.7323011 sec
RG 203.2
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -3.00 dB
SFO1 399.8023988 MHz
F2 - Processing parameters
SI 32768
SF 399.8000174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
OH
Me
MOMO
Table 4, entry 6
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.125
0.125
2.000
2.180
1.148
4.300
0.934
0.998
2.601
4.823
3.638


OH
MOMO
Me
OH
Table 4, entry 6
Current Data Parameters
NAME mr-1-45carbon
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041201
Time 21.42
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 346
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.70 usec
PL1 0.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.1322200 MHz
F2 - Processing parameters
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

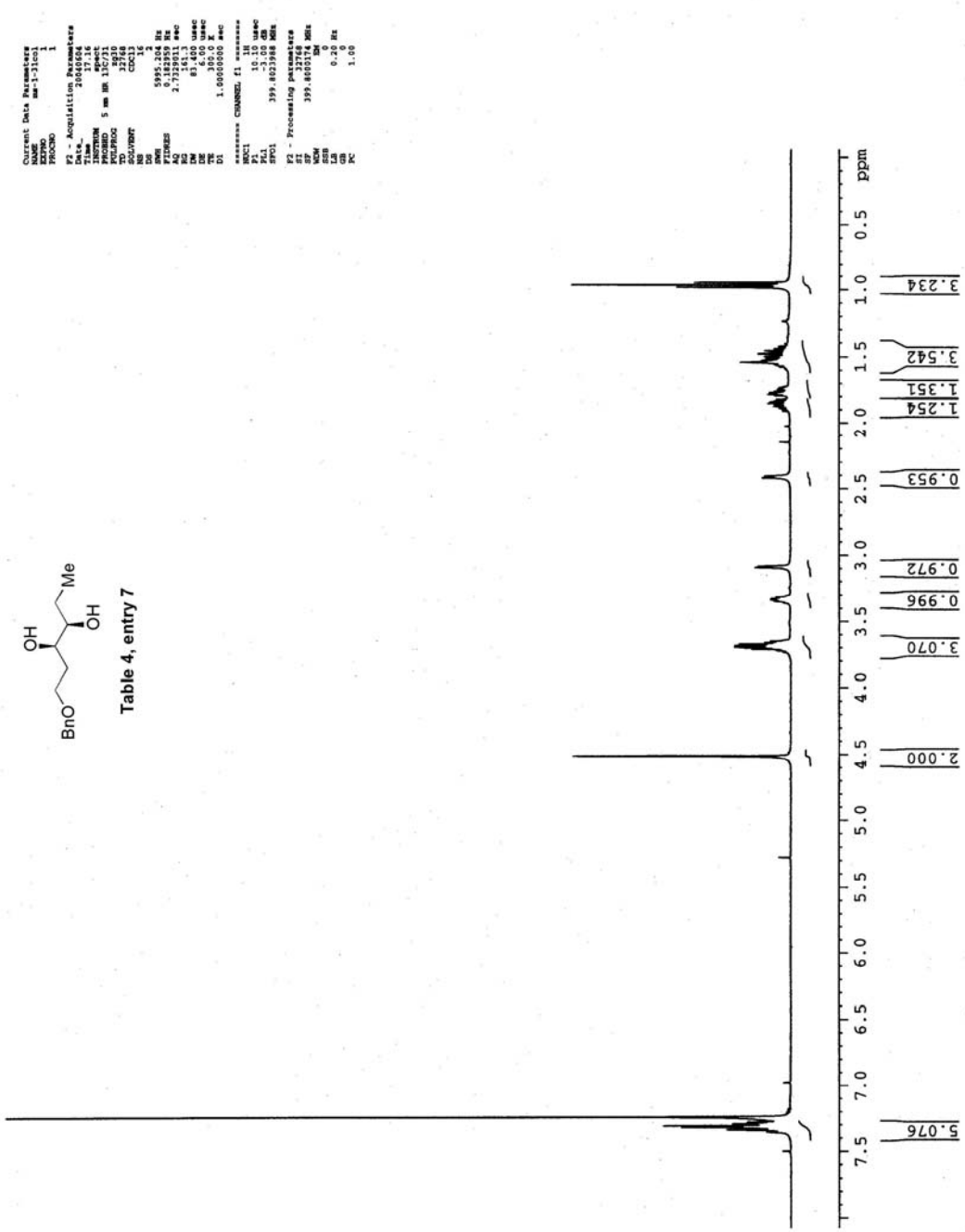
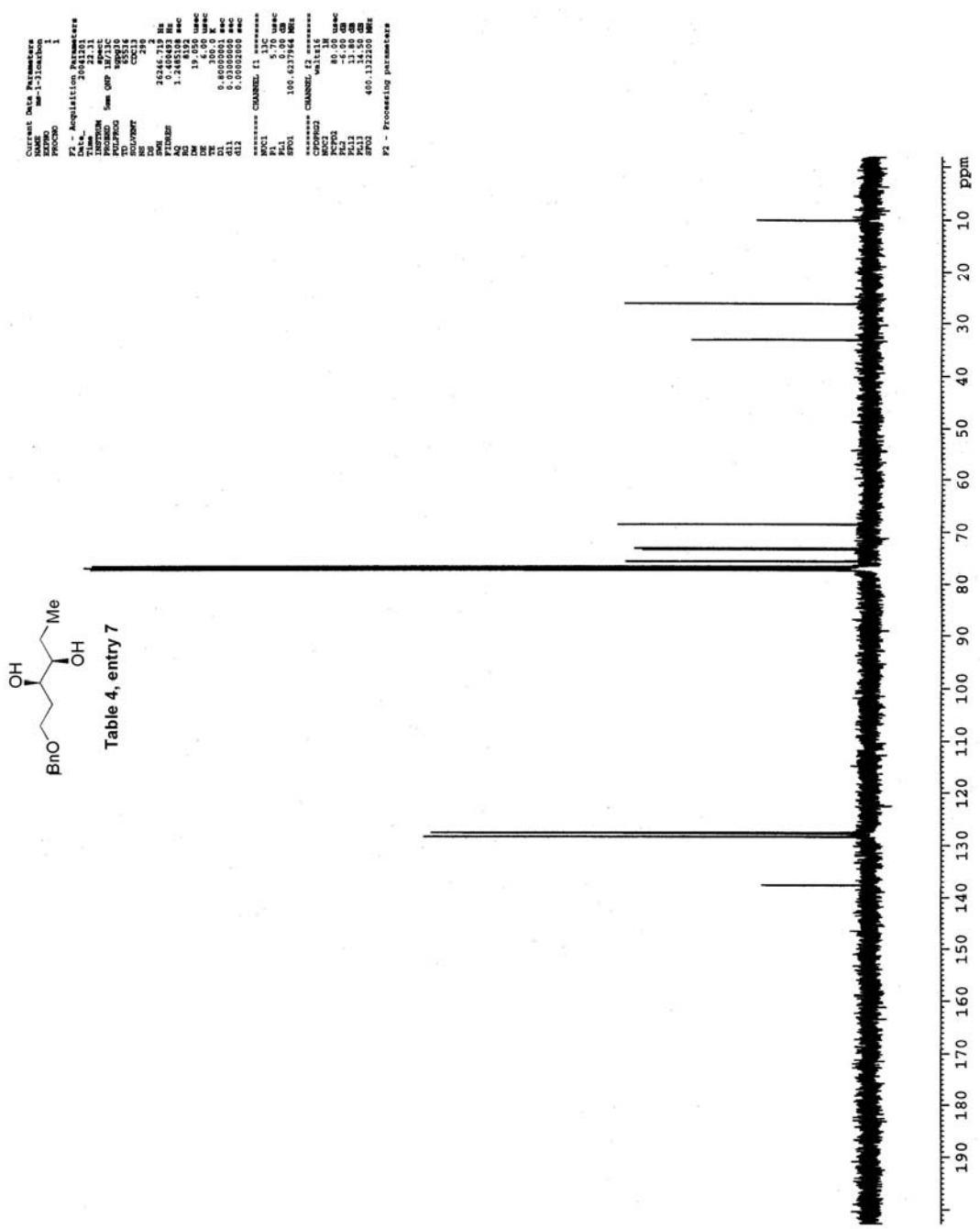


Table 4, entry 7
OH
Me
OH
BnO
5.076
2.000
3.070
0.996
0.972
0.953
1.254
1.351
3.542
3.234
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


Current Data Parameters
NAME mm-3-31-carbon
EXPNO 1
PROCNO 1
F1 - Acquisition Parameters
Data_ 20041201
TIME 22.31
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 26246.719 Hz
FIDRES 0.400543 Hz
AQ 1.2484352 sec
RG 8192
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
d11 0.03000001 sec
d1 1.00000001 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.70 usec
PL1 0.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 13.80 dB
PL13 14.00 dB
SFO2 400.1322300 MHz
F2 - Processing parameters
OH
Me
OH
BnO
Table 4, entry 7
ppm

Current Data Parameters
NAME ST-1-278
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041130
Time 15.43
INSTRUM spect
PROBHD 5 mm HR 13C/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 71.8
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SFO1 399.8023988 MHz
F2 - Processing parameters
SI 32768
SF 399.8000174 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
OH
OH
t-butyl
Table 5, entry 8
0.977
0.979
0.991
2.078
0.990
1.005
9.000
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

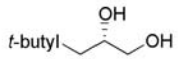
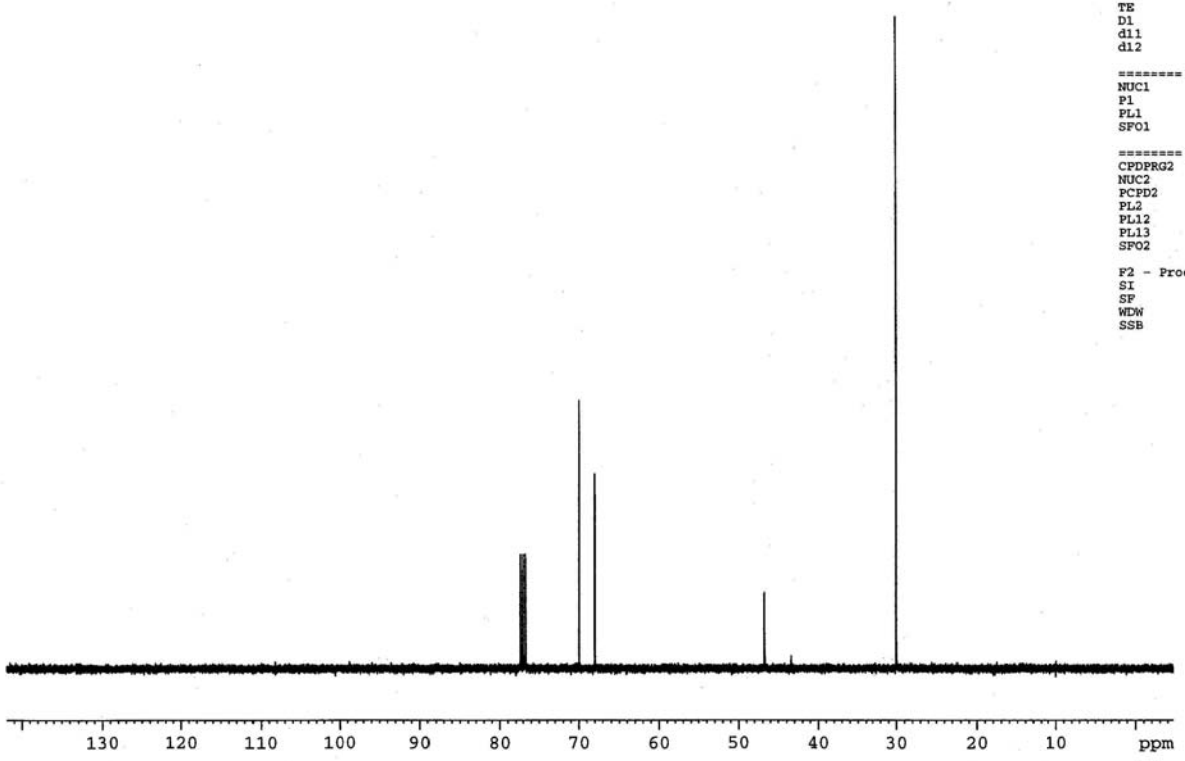


Table 5, entry 8

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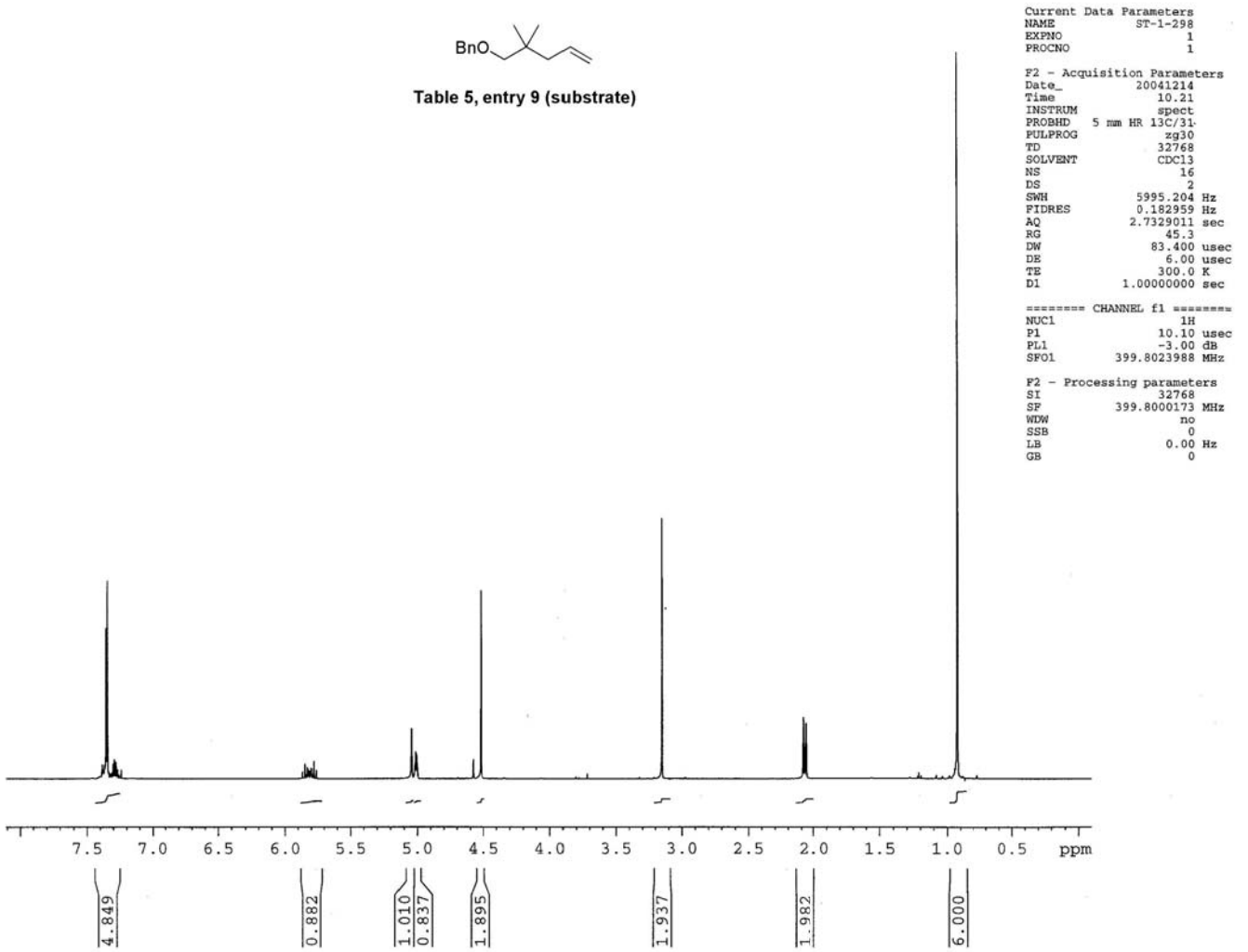
Current Data Parameters
NAME                  ST-1-278
EXPNO                        3
PROCNO                       1

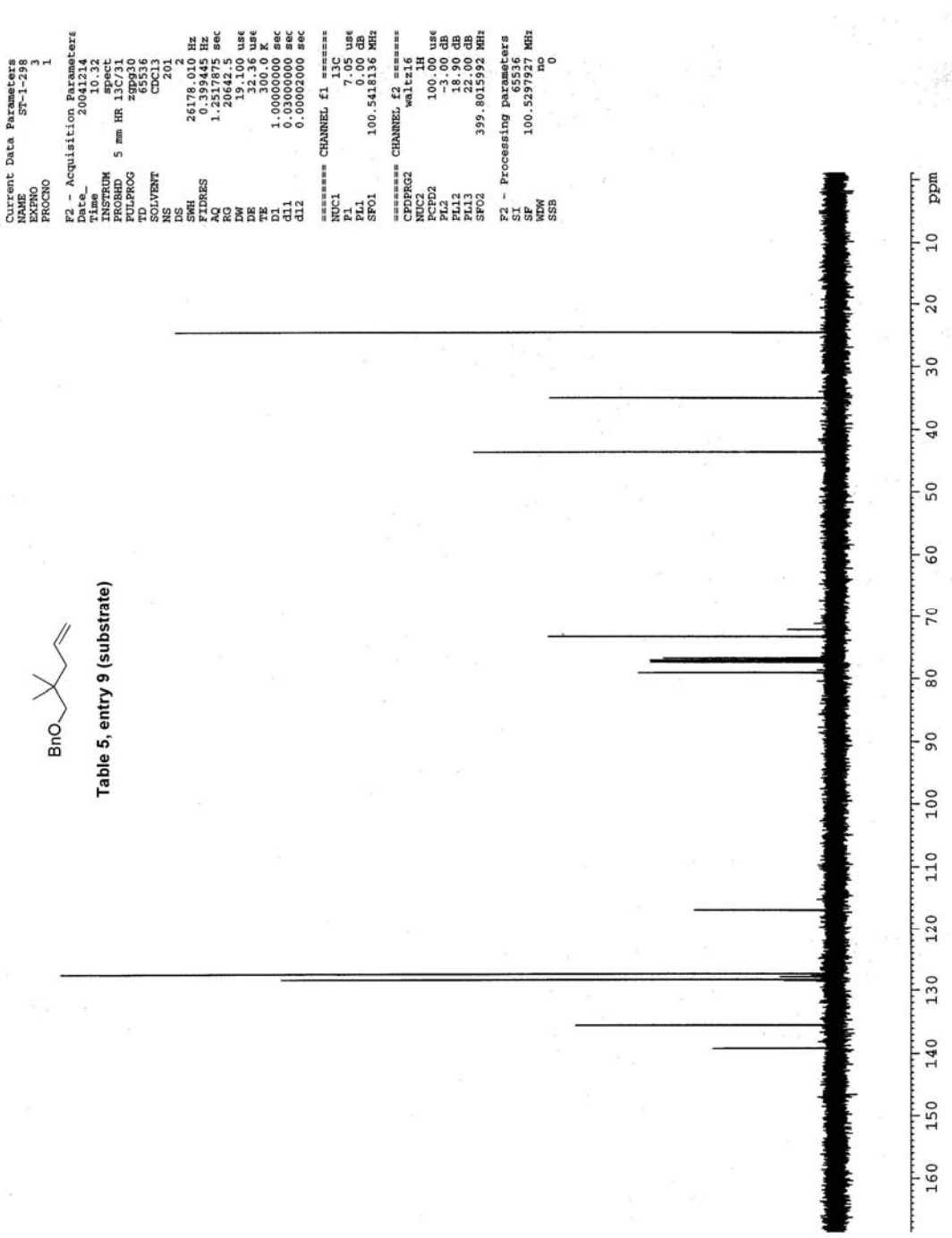
F2 - Acquisition Parameters
Date_              20041130
Time                   15.54
INSTRUM                spect
PROBHD   5 mm HR 13C/31
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                        357
DS                          2
SWH                26178.010 Hz
FIDRES             0.399445 Hz
AQ                 1.2517875 sec
RG                   14596.5
DW                    19.100 use
DE                    32.36 use
TE                    300.0 K
D1                 1.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

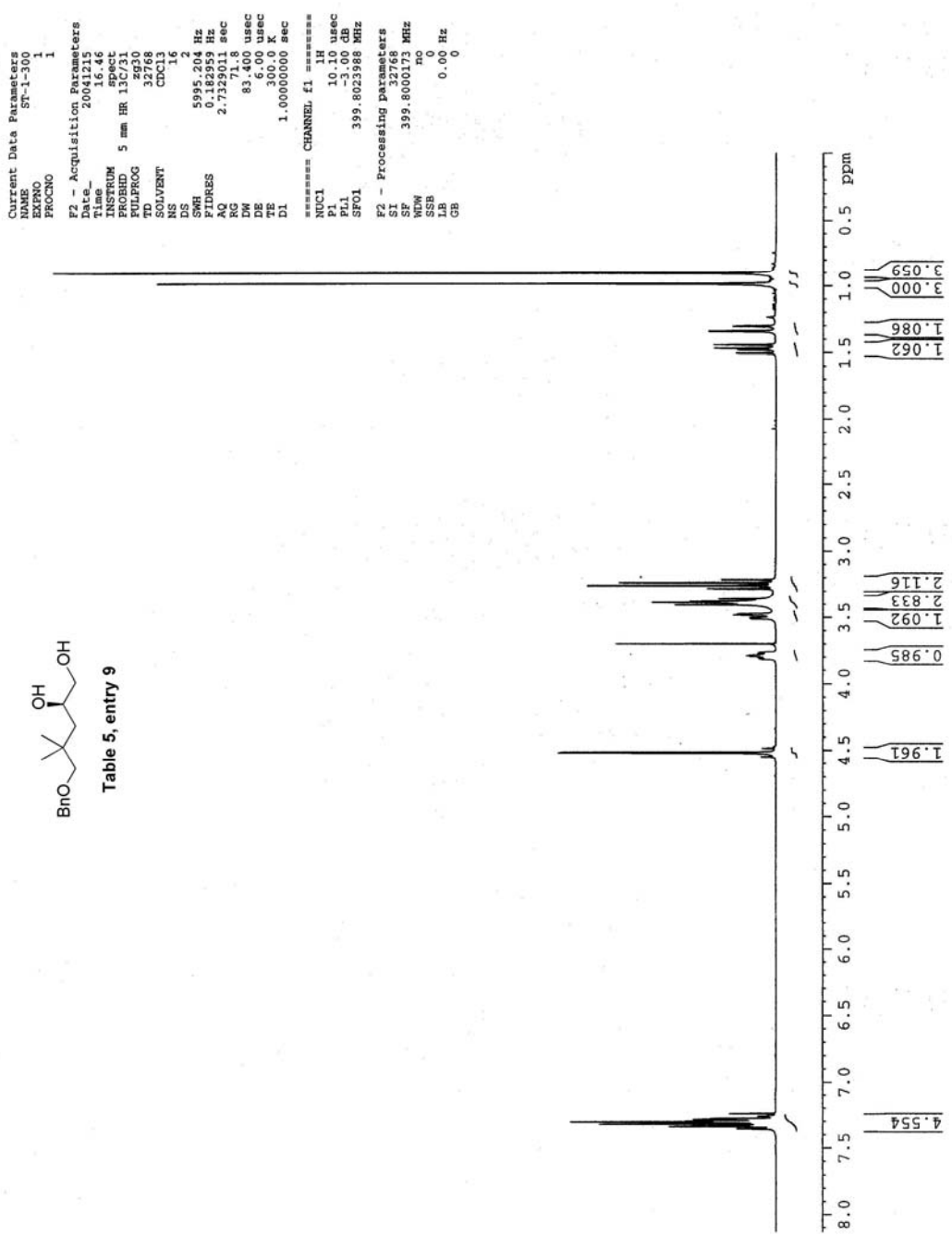
======== CHANNEL f1 =======
NUC1                     13C
P1                      7.05 use
PL1                     0.00 dB
SFO1             100.5418136 MHz

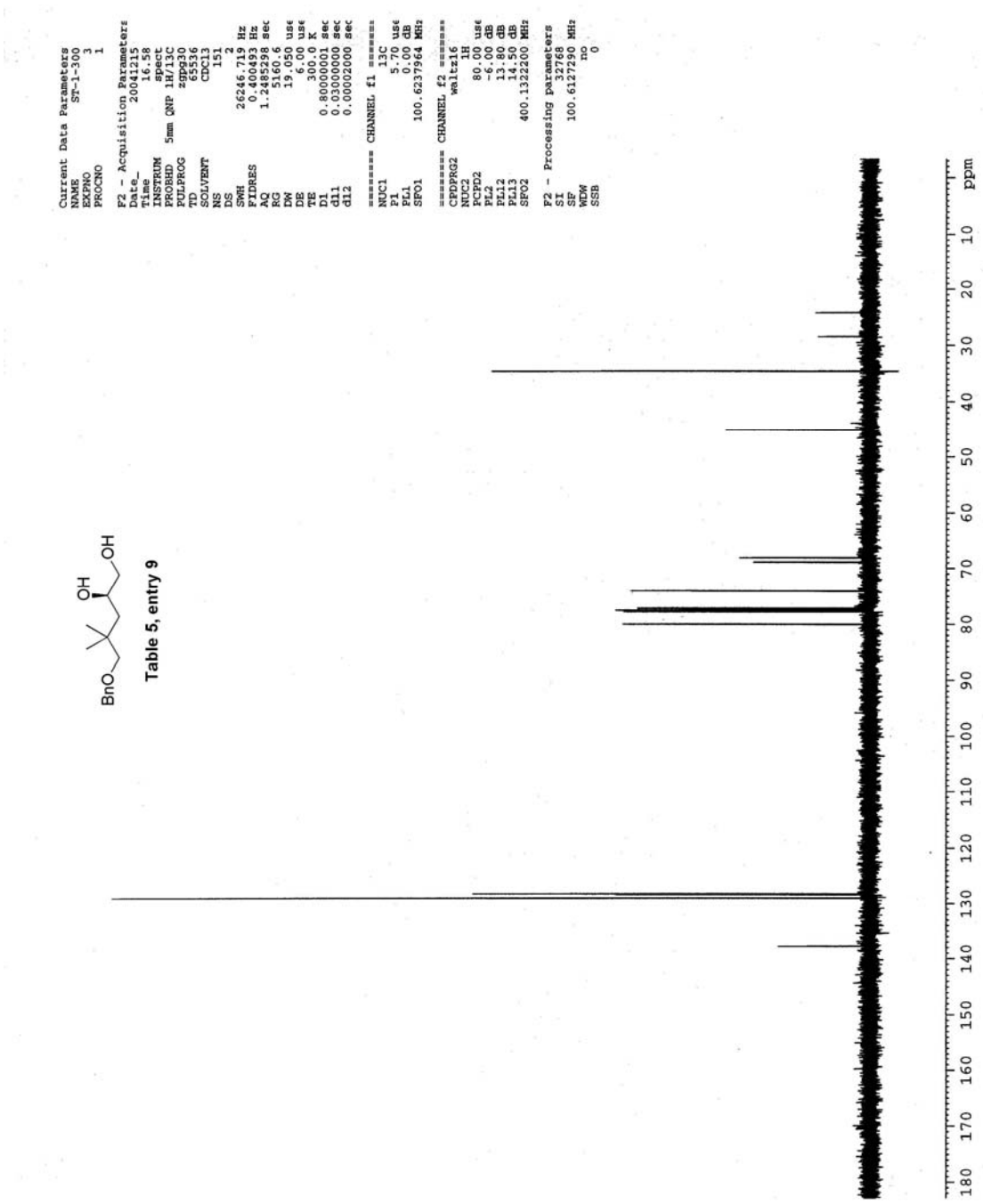
======== CHANNEL f2 =======
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 100.00 use
PL2                    -3.00 dB
PL12                   18.90 dB
PL13                   22.00 dB
SFO2             399.8015992 MHz

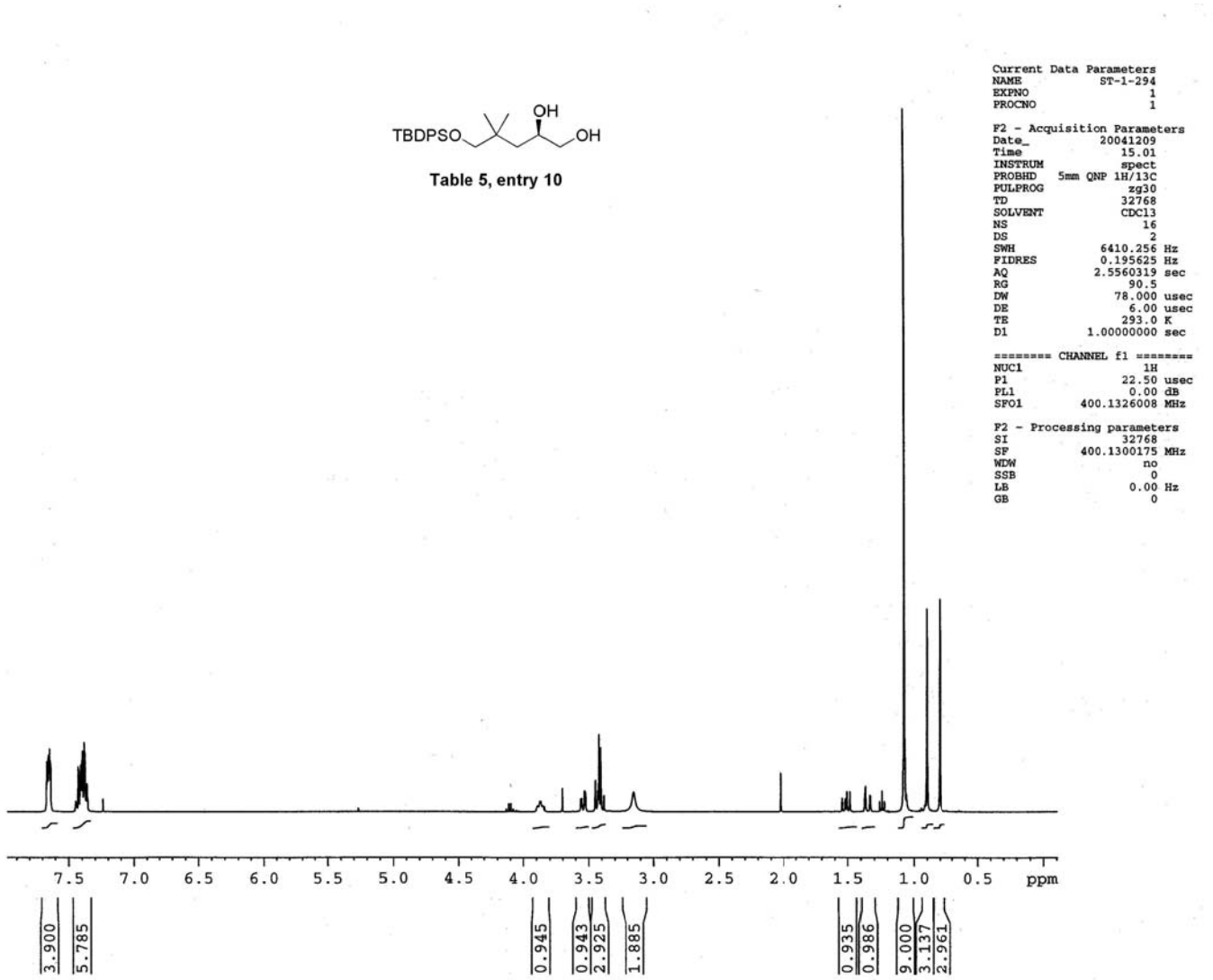
F2 - Processing parameters
SI                       65536
SF               100.5297899 MHz
WDW                         no
SSB                          0
```

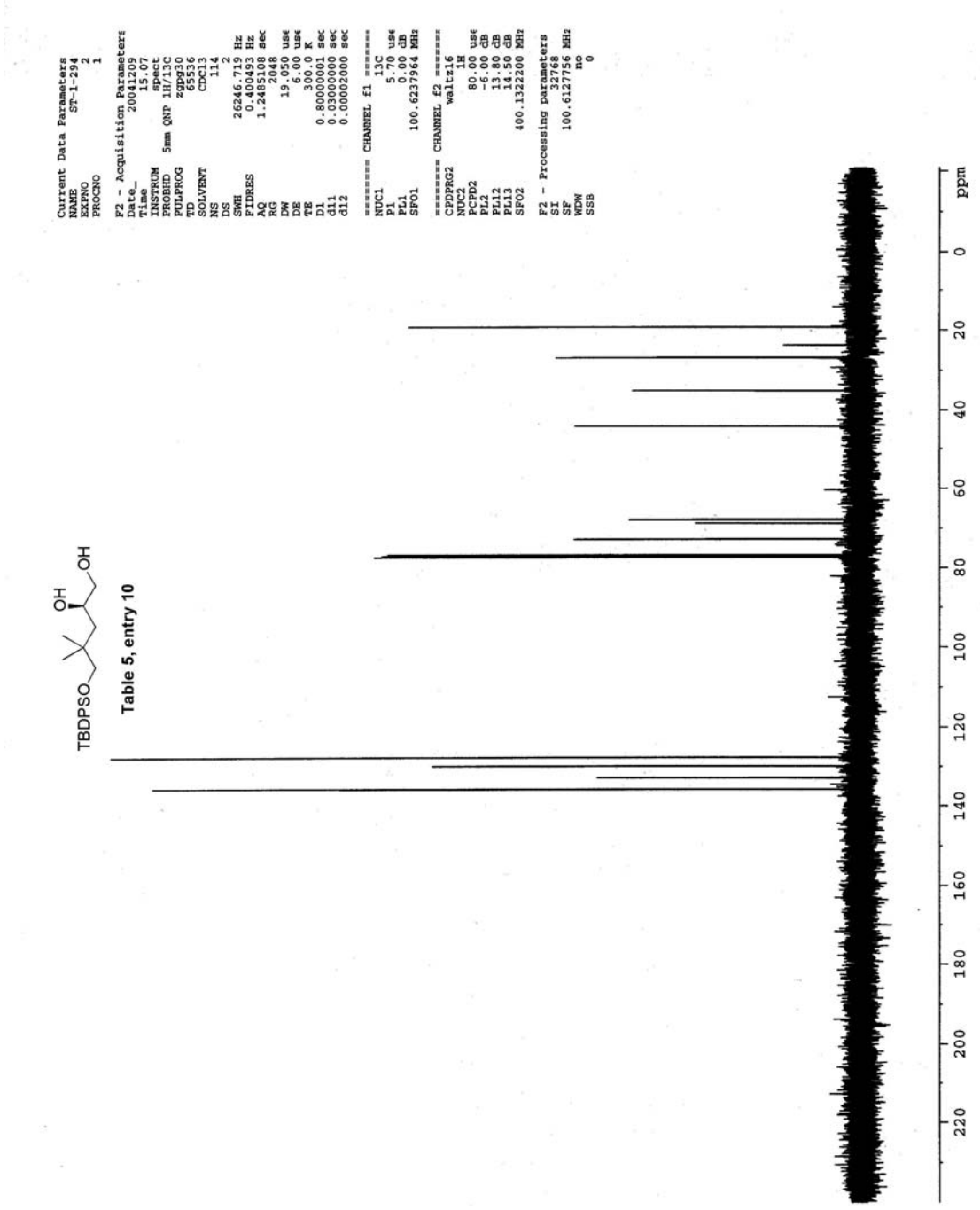

BnO
Table 5, entry 9 (substrate)
Current Data Parameters
NAME ST-1-298
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041214
Time 10.21
INSTRUM spect
PROBHD 5 mm HR 13C/31-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 45.3
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -3.00 dB
SFO1 399.8023988 MHz
F2 - Processing parameters
SI 32768
SF 399.8000173 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.849
0.882
1.010
0.837
1.895
1.937
1.982
6.000


Current Data Parameters
NAME ST-1-298
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041214
Time 10.32
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 201
DS 2
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 20642.5
DW 19.100 use
DE 32.36 use
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.05 use
PL1 0.00 dB
SFO1 100.5418136 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 18.90 dB
PL13 22.00 dB
SFO2 399.8015992 MHz
F2 - Processing parameters
SI 65536
SF 100.5297927 MHz
WDW no
SSB 0
BnO
Table 5, entry 9 (substrate)
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

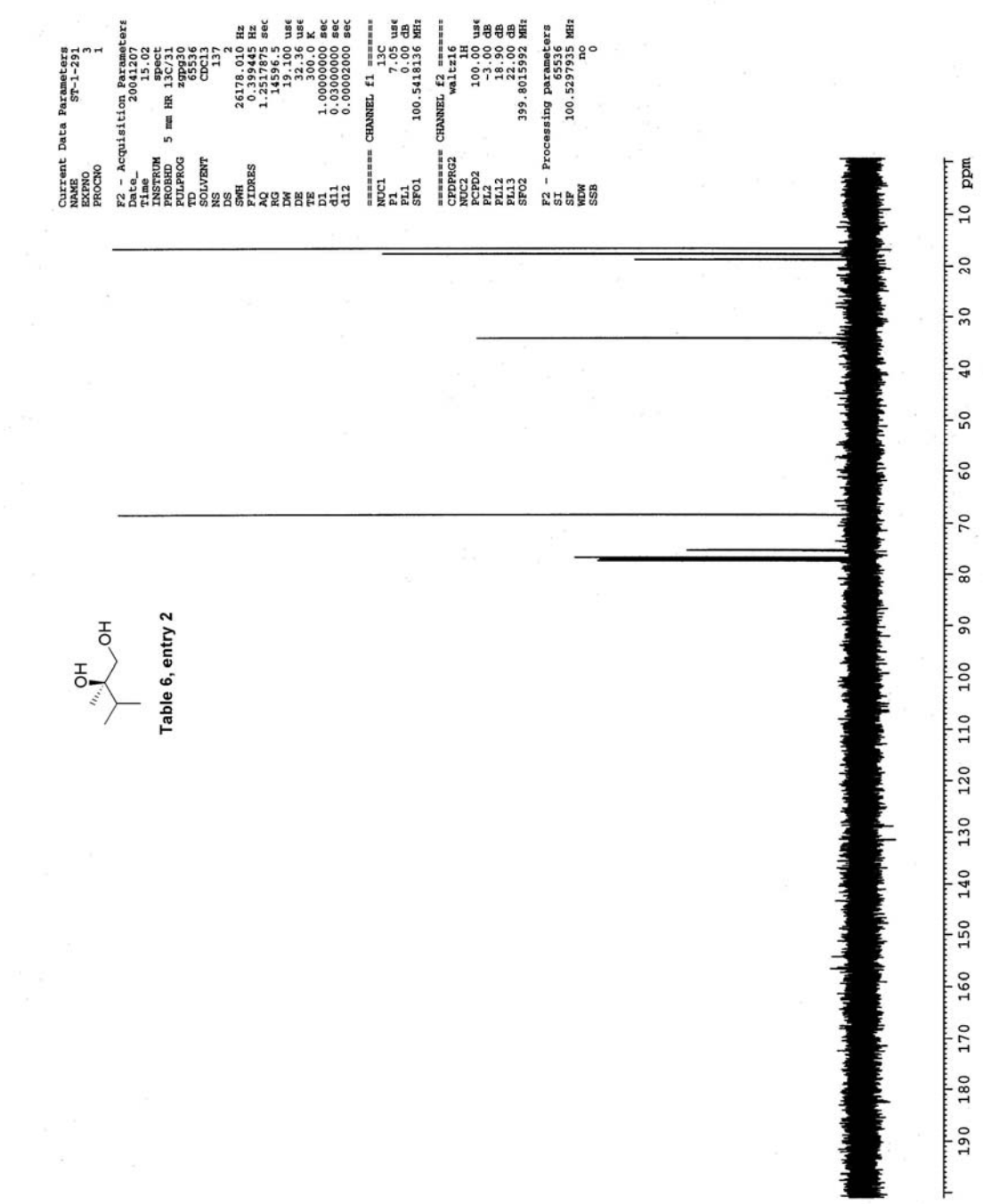

Current Data Parameters
NAME ST-1-300
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041215
Time 16.46
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 71.8
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -3.00 dB
SFO1 399.8023988 MHz
F2 - Processing parameters
SI 32768
SF 399.8000173 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
OH
OH
BnO
Table 5, entry 9
4.554
1.961
0.985
1.092
2.833
2.116
1.062
1.086
3.000
3.059
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


Current Data Parameters
NAME ST-1-300
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041215
Time 16.58
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 151
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 5160.6
DW 19.050 use
DE 6.00 use
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.70 use
PL1 0.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.1322200 MHz
F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW no
SSB 0
OH
OH
BnO
Table 5, entry 9
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

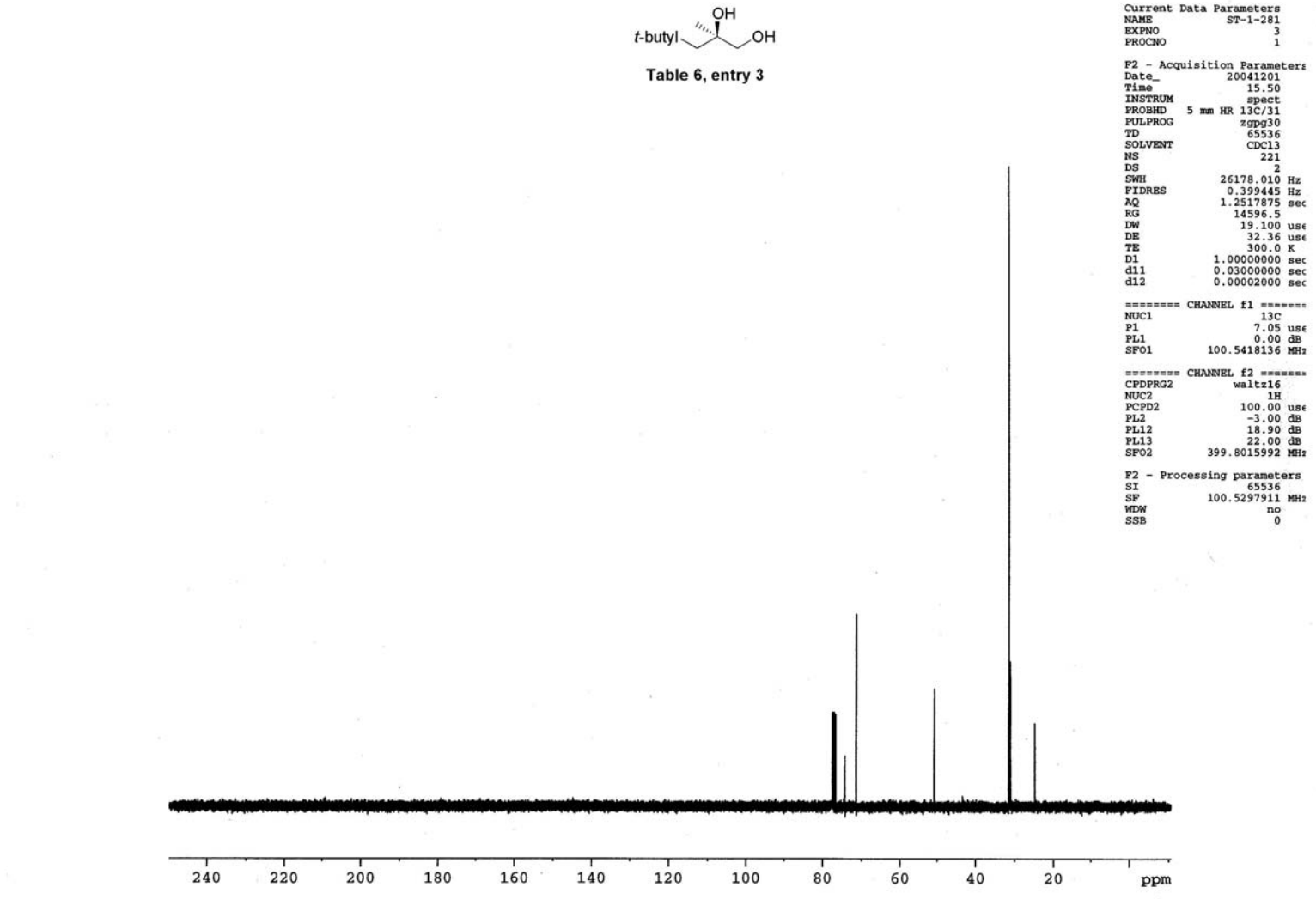

TBDPSO
OH
OH
Table 5, entry 10
Current Data Parameters
NAME ST-1-294
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041209
Time 15.01
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5560319 sec
RG 90.5
DW 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 22.50 usec
PL1 0.00 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 32768
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
3.900
5.785
0.945
0.943
2.925
1.885
0.935
0.986
9.000
3.137
2.961


TBDPSO
OH
OH
Table 5, entry 10
Current Data Parameters
NAME SF-1-294
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041209
Time 15.07
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 114
DS 2
SWH 26246.719 Hz
FIDRES 0.400443 Hz
AQ 1.248510 sec
RG 2048
DW 19.050 use
DE 6.00 use
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.70 use
PL1 0.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.1322200 MHz
F2 - Processing parameters
SI 32768
SF 100.6127756 MHz
WDW no
SSB 0
220
200
180
160
140
120
100
80
60
40
20
0
ppm

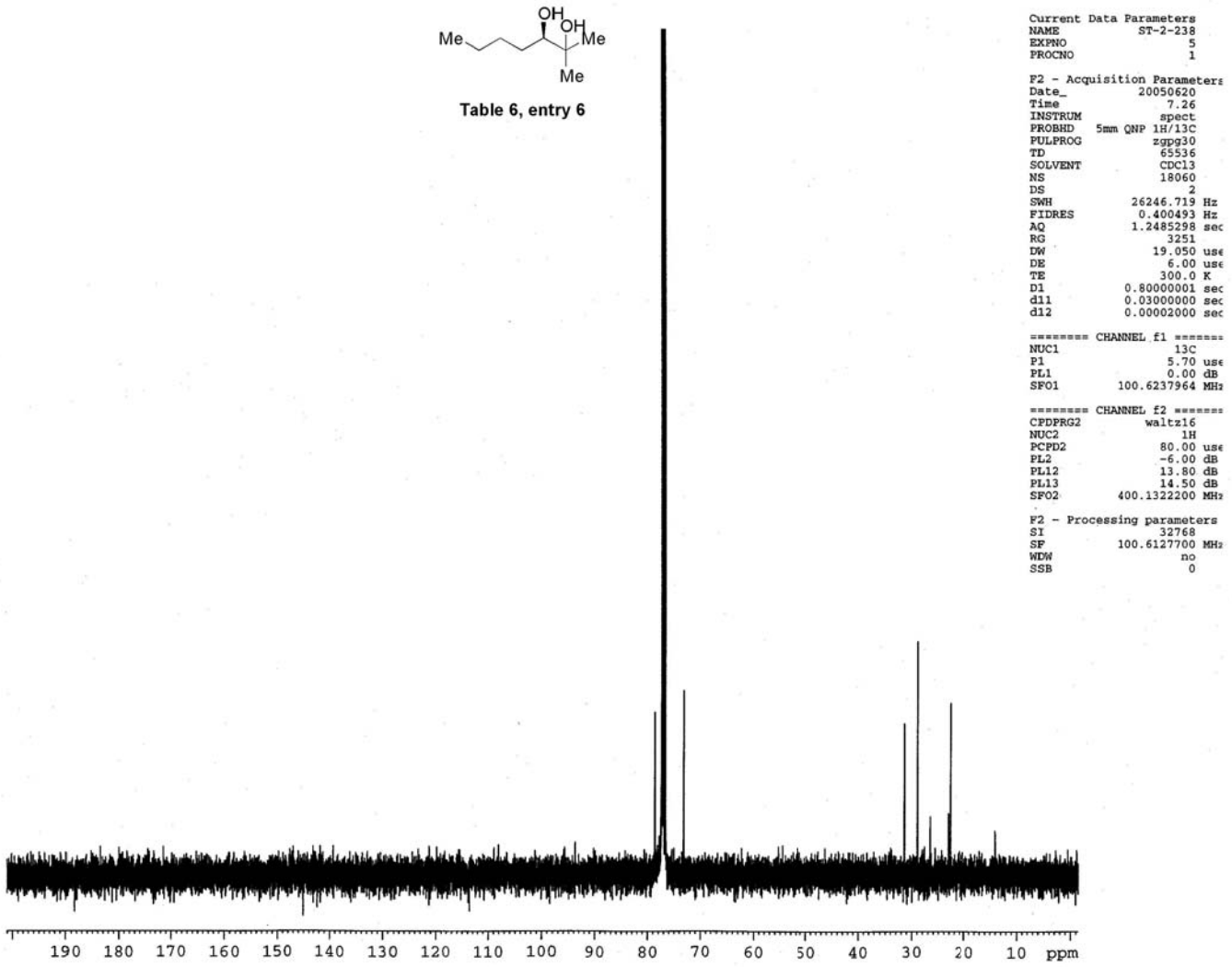
Current Data Parameters
NAME ST-1-291
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041207
Time 14.52
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 40.3
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -1.00 dB
SFO1 399.8023988 MHz
F2 - Processing parameters
SI 32768
SF 399.8000173 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
OH
OH
Table 6, entry 2
2.931
3.002
2.960
1.000
2.312
0.951
0.932
ppm

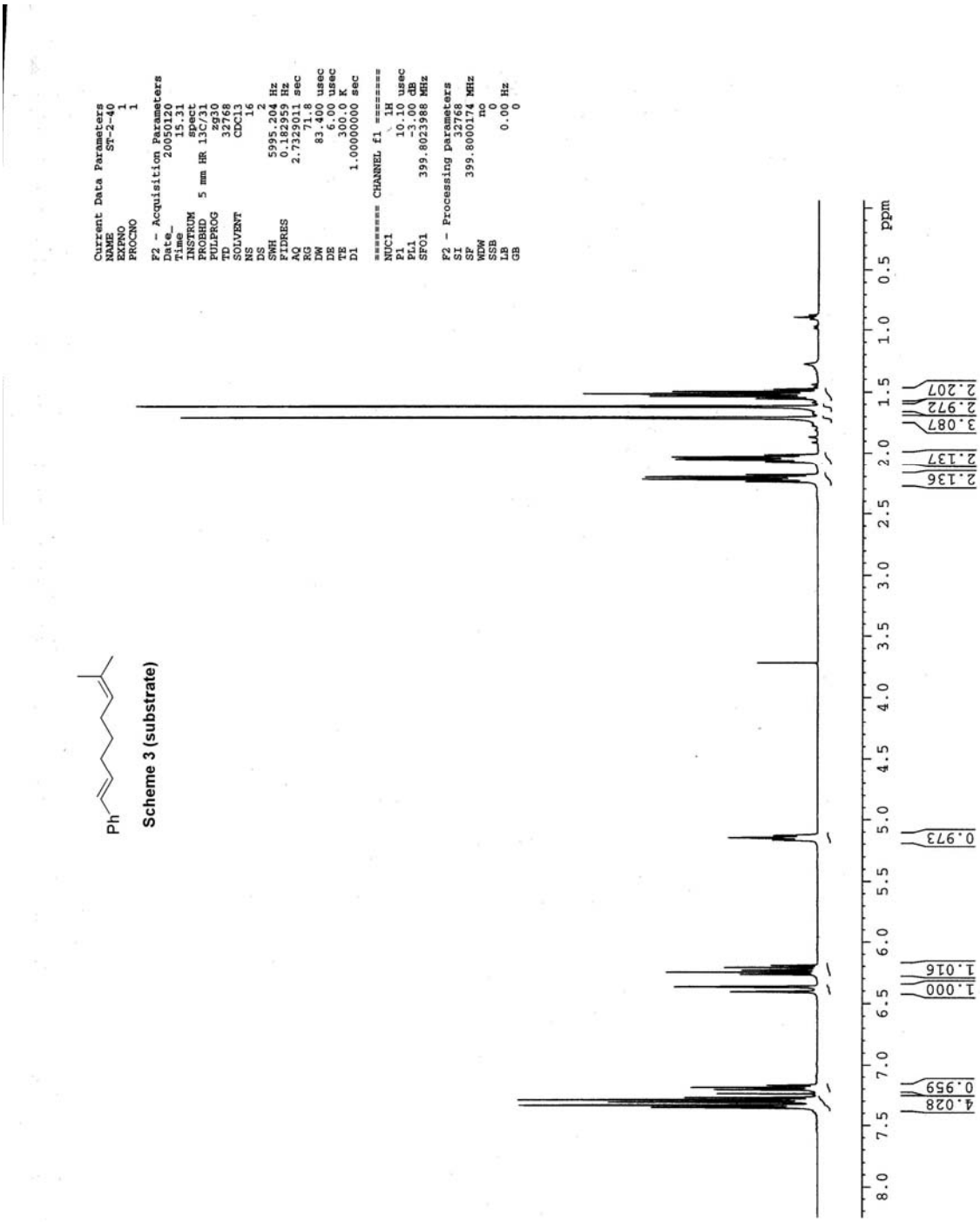

Current Data Parameters
NAME ST-1-291
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041207
Time 15.02
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 137
DS 2
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2479875 sec
RG 14596.5
DW 19.100 use
DE 32.36 use
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.05 use
PL1 0.00 dB
SFO1 100.5418136 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 3.00 dB
PL12 18.90 dB
PL13 22.00 dB
SFO2 399.8015992 MHz
F2 - Processing parameters
SI 65536
SF 100.5297935 MHz
WDW no
SSB 0
OH
OH
Table 6, entry 2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

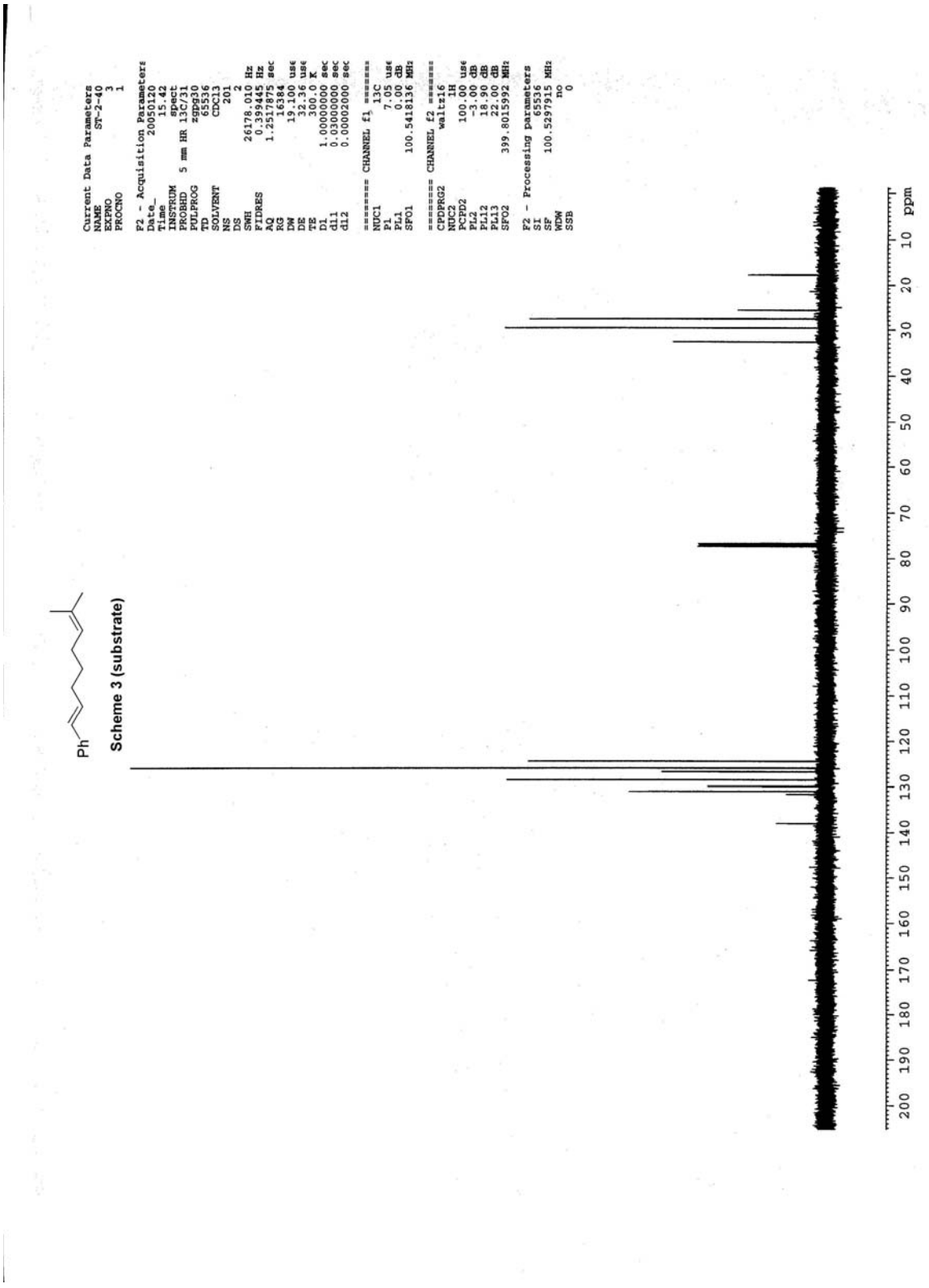
Current Data Parameters
NAME ST-1-281
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041201
Time 13.40
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 45.3
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -1.00 dB
SFO1 399.8023988 MHz
F2 - Processing parameters
SI 32768
SF 399.8000174 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
OH
OH
t-butyl
Table 6, entry 3
1.017
0.985
2.058
1.076
1.081
3.053
9.000
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

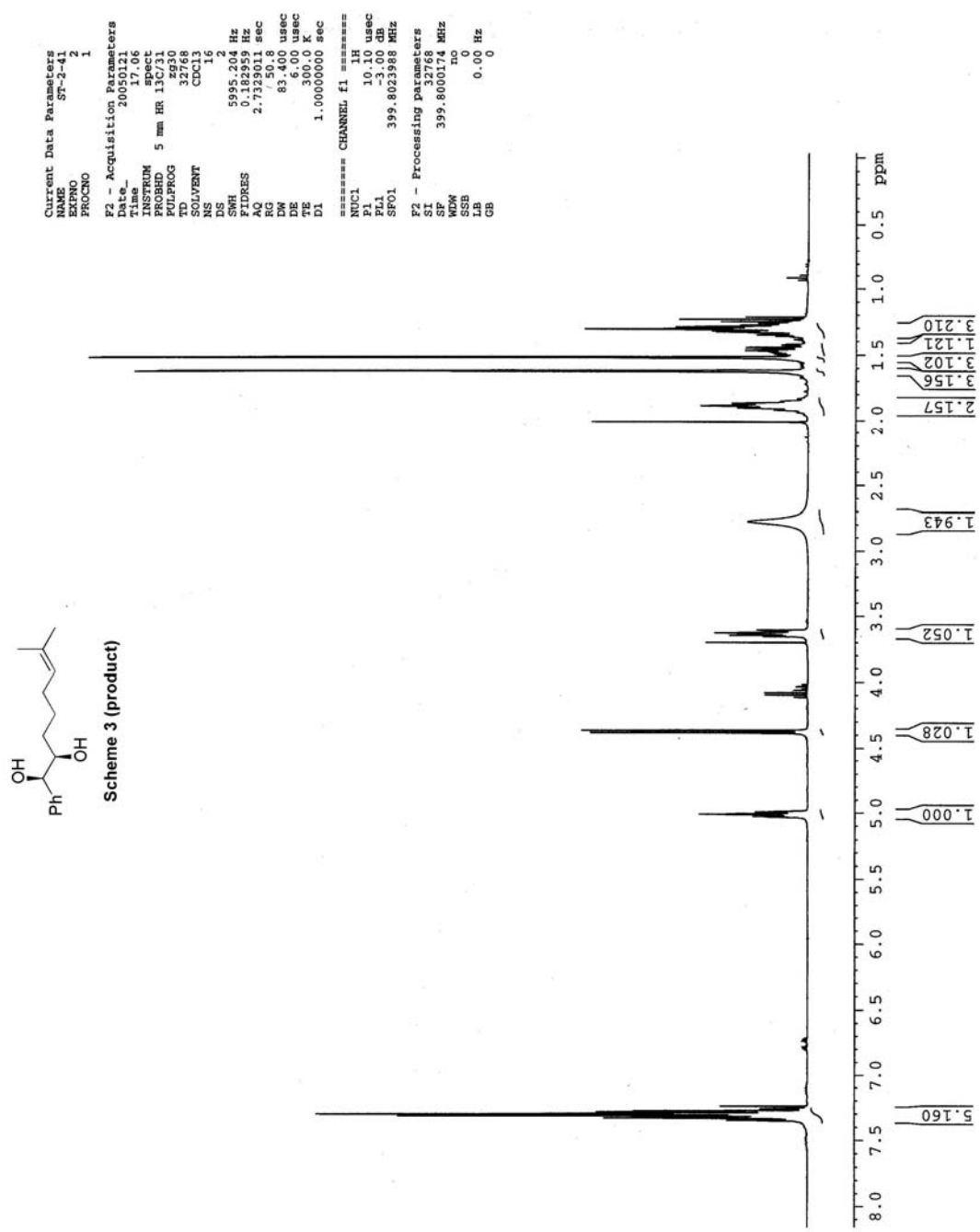

OH
t-butyl
OH
Table 6, entry 3
Current Data Parameters
NAME ST-1-281
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041201
Time 15.50
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 221
DS 2
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 14596.5
DW 19.100 use
DE 32.36 use
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 =======
NUC1 13C
P1 7.05 use
PL1 0.00 dB
SFO1 100.5418136 MHz
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 18.90 dB
PL13 22.00 dB
SFO2 399.8015992 MHz
F2 - Processing parameters
SI 65536
SF 100.5297911 MHz
WDW no
SSB 0
240
220
200
180
160
140
120
100
80
60
40
20
ppm

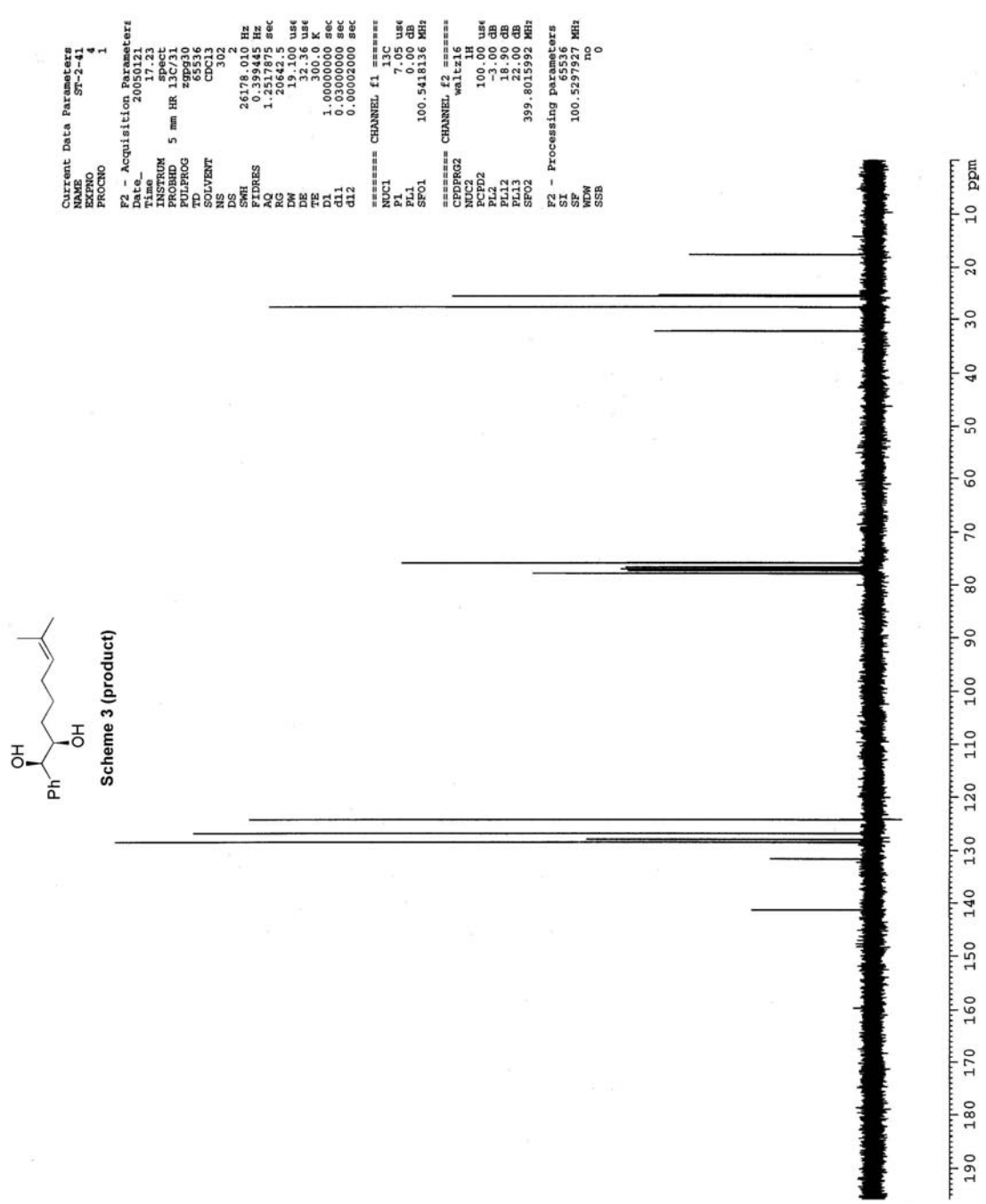
Current Data Parameters
NAME ST-2-238
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050617
Time 13.28
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.55603197 sec
RG 1625.5
DW 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 22.50 usec
PL1 0.00 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 32768
SF 400.1300172 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
OH
OH
Me
Me
Me
Table 6, entry 6
1.000
6.007
8.681
3.313
3.262
3.766
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


Me
OH
OH
Me
Me
Table 6, entry 6
Current Data Parameters
NAME ST-2-238
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050620
Time 7.26
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 18060
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 3251
DW 19.050 use
DE 6.00 use
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 =======
NUC1 13C
P1 5.70 use
PL1 0.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.1322200 MHz
F2 - Processing parameters
SI 32768
SF 100.6127700 MHz
WDW no
SSB 0
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Current Data Parameters
NAME ST-2-40
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050120
Time 15.31
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 71.8
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -3.00 dB
SFO1 399.8023988 MHz
F2 - Processing parameters
SI 32768
SF 399.8000174 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
Ph
Scheme 3 (substrate)
4.028
0.959
1.000
1.016
0.973
2.136
2.137
3.087
2.972
2.207
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


Ph
Scheme 3 (substrate)
Current Data Parameters
NAME SM-2-40
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050120
Time 15.42
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 201
DS 2
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 16384
DW 19.100 use
DE 32.36 use
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.05 use
PL1 0.00 dB
SFO1 100.5418136 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 18.90 dB
PL13 22.00 dB
SFO2 399.8015992 MHz
F2 - Processing parameters
SI 65536
SF 100.5297915 MHz
WDW no
SSB 0
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Current Data Parameters
NAME ST-2-41
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050121
Time 17.06
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 50.8
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -3.00 dB
SFO1 399.8023988 MHz
F2 - Processing parameters
SI 32768
SF 399.8000174 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
OH
OH
Ph
Scheme 3 (product)
5.160
1.000
1.028
1.052
1.943
2.157
3.156
3.102
1.121
3.210
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


Current Data Parameters
NAME ST-2-41
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050121
Time 17.23
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 302
DS 2
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 20642.5
DW 19.100 use
DE 32.36 use
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.05 use
PL1 0.00 dB
SFO1 100.5418136 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 18.90 dB
PL13 22.00 dB
SFO2 399.8015992 MHz
F2 - Processing parameters
SI 65536
SF 100.5297927 MHz
WDW no
SSB 0
OH
OH
Ph
Scheme 3 (product)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

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