# RH-CATALYZED ENANTIOSELECTIVE HYDROGENATION OF VINYL BORONATES FOR THE CONSTRUCTION OF SECONDARY BORONIC ESTERS.

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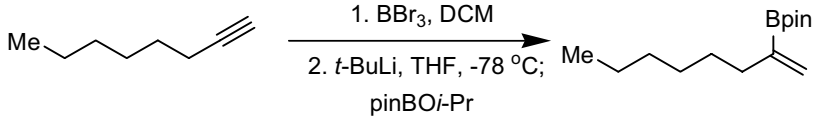
## Supporting Information

**General.** $^{1}$H NMR spectra were recorded on Bruker DRX 300 or 400 MHz spectrometers. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (CDCl3: 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 was recorded on a Bruker 400 MHz (100 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal standard ($CDCl_3$: 77.0 ppm). Mass spectrometry (m/z) was performed on either a Bruker BioTOF II or a Micromass Quattro II, operating in ESI or APCI mode, with only molecular ions reported. Infrared (IR) spectra were obtained on an ASI ReactIR 1000 or a Nicolet 560 Magna-IR, υmax in $cm^{-1}$. Bands are characterized as broad (br), strong (s), medium (m) and weak (w). Flash chromatography was performed on silica gel ($SiO_2$, 230 X 450 mesh) purchased from Sorbent Technologies, Inc. Thin layer chromatography (TLC) was performed on aluminium backed plates pre-coated with silica (0.2 mm, Merck DC-alufolien Kieselgel 60). Visualization was achieved using basic potassium permanganate solution, followed by heating. Analytical gas-liquid chromatography (GLC) was performed on a Hewlett-Packard 6890 Series chromatography equipped with a CTC Analysis Combi Pal autosampler by Leap Technologies (Carrboro, NC), a split mode capillary injection system, a flame ionization detector and a Supelco β-dex 120 column with helium as the carrier gas. All reactions were conducted in oven and flame dried glassware under an inert atmosphere of nitrogen unless otherwise stated. Walphos 1, (*R*)-1-[(*R*)-2-(2'-diphenylphosphinophenyl)ferrocenyl]ethyldi(bis-3,5-trifluoromethylphenyl)phosphine, was kindly donated by Sovias AG. Bis(norbornadiene) rhodium(I) tetrafluoroborate was purchased from Acros Organics. All other reagents were purchased from Aldrich Chemical Companies and used directly. Hydrogenations were performed in a stainless steel high-pressure vessel from Parr Instrument Company. Enantiomeric excesses of the boronate compounds were determined by chiral GC analysis after oxidation of the carbon boron bond followed by acetate derivatization. Absolute configuration of products determined by comparison of GC data of commercial non-racemic chiral alcohols.

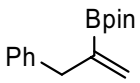
A note about NMR spectra: Due to the boron quadrupole, carbons directly attached to this element are often not detected in $^{13}$C spectra. See Wrackmeyer, B. *Prog. In NMR Spectroscopy,* **1979**, *12*, 227. In some cases, the $^{2}$J and $^{3}$J $^{11}$B/$^{1}$H and $^{10}$B/$^{1}$H coupling makes determination of some $^{1}$H/$^{1}$H coupling constants difficult.

**Representative Procedure for Preparation of Vinyl Boronic Acid Pinacol Esters: Synthesis of 2-(1-Hexyl-vinyl)-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane.**

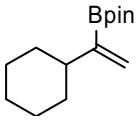



1-Octyne (1.3 ml, 9.1 mmol, 1 equiv) was added dropwise to a 1 M solution of boron tribromide (4.5 ml, 4.5 mmol, 0.5 equiv) at -78 °C. The resulting solution was allowed to warm to room temperature over 3 h. Glacial acetic acid (9 ml) was added to the mixture and stirred for 1 h. This mixture was quenched with water, extracted with DCM and dried over MgSO4. Filtration, followed by (careful) concentration in vacuo and passage through a short silica column (hexanes) provided the desired vinyl bromide sufficiently clean to continue on to the next step. The vinyl bromide was dissolved in THF (15 ml) and cooled to -78 °C. t-BuLi (1.5 M, 12 ml, 18 mmol, 2 equiv) was added dropwise, and the resulting mixture stirred for 0.5 - 1 h at -78 °C. 2-Isopropoxy-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane (2.8 ml, 14 mmol, 1.5 equiv) dissolved in THF (10 ml) was added dropwise via cannula and the mixture allowed to warm to room temperature over 3 h. 1 M HCl solution (20 ml) was added and the mixture was stirred for 0.5 h. Extraction with DCM, followed by drying

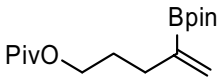
over MgSO4, filtration and concentration in vacuo provided the crude oil. Purification by flash chromatography (20:1 hexanes / EtOAc) furnished a colorless oil (1.8 g, 83%). IR (neat): 2975 (s), 2861 (s), 1721 (s), 1615 (s), 1142 (br) $cm^{-1}$. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 0.83 (3H, t, J = 6.4 Hz), 1.15-1.42 (20H, m), 2.11 (2H, t, J = 6.4 Hz), 5.57 (1H, br), 5.72 (1H, br). $^{13}C$ NMR (100.6 MHz, $CDCl_3$): δ 14.5, 23.0, 25.1, 29.4, 29.6, 32.2, 35.8, 83.7, 129.1.



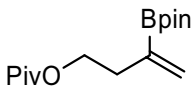

**2-(1-benzyl-vinyl)-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane.** IR (neat): 2981 (s), 2931 (s), 1947 (w), 1887 (w), 1713 (s), 1615 (s) $cm^{-1}$. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 1.21 (12H, s), 3.47 (2H, s), 5.51 (1H, br), 5.82 (1H, br), 7.13-7.25 (5H, m). $^{13}C$ NMR (100.6 MHz, $CDCl_3$): δ 24.8, 41.6, 83.7, 125.9, 128.3, 129.4, 130.1, 140.9.



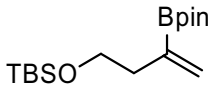

**2-(1-cyclohexyl-vinyl)-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane.** IR (neat): 2979 (s), 2925 (s), 2852 (s), 1611 (m), 1306 (s), 1144 (s) $cm^{-1}$. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 1.10-1.70 (22H, m), 2.00-2.16 (1H, m), 5.55 (1H, br), 5.70 (1H, br). $^{13}C$ NMR (100.6 MHz, $CDCl_3$): δ 14.1, 24.9, 26.9, 32.7, 43.0, 83.4, 126.1.



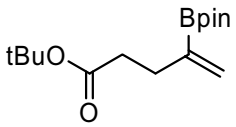

**2,2-dimethyl-propionic acid 4-(4,4,5,5-tetramethyl-[1,3,2]dioxaborolan-2-yl)-pent-4-enyl ester.** IR (neat): 3025 (s), 2900 (s), 1727 (s), 1484 (m) $cm^{-1}$. $^{1}H$ NMR (300 MHz, $CDCl_3$): δ 1.20 (9H, s), 1.26 (12H, s), 1.67-1.80 (2H, m), 2.22 (2H, t, J = 7.7 Hz), 4.04 (2H, t, J = 6.6 Hz), 5.62 (1H, br), 5.80 (1H, br). $^{13}C$ NMR (75.5 MHz, $CDCl_3$): δ 22.9, 25.0, 27.4, 28.4, 39.0, 64.1, 83.6, 130.1, 178.3.



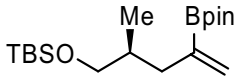

**2,2-dimethyl-propionic acid 3-(4,4,5,5-tetramethyl-[1,3,2]dioxaborolan-2-yl)-but-3-enyl ester.** IR (neat): 3025 (s), 2914 (s), 1756 (s), 1360 (s) cm-1. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 1.15 (9H, s), 1.24 (12H, s), 2.45 (2H, t, J = 6.8 Hz), 4.13 (2H, t, J = 7.0 Hz), 5.66 (1H, br), 5.84 (1H, br). $^{13}C$ NMR (100.6 MHz, $CDCl_3$): δ 25.2, 27.6, 35.0, 39.1, 63.8, 83.9, 132.3, 178.9.




**2-{1-[2-(tert-butyl-dimethyl-silanyloxy)-ethyl]-vinyl}-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane.** IR (neat): 2933 (s), 1723 (s), 1382 (s) $cm^{-1}$. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 0.01 (6H, s), 0.87 (9H, s), 1.26 (12H, s), 2.37 (2H, t, J = 7.2 Hz), 3.65 (2H, t, J = 7.2 Hz), 5.65 (1H, br), 5.82 (1H, br). $^{13}C$ NMR (100.5 MHz, $CDCl_3$): δ -5.2, 18.4, 24.7, 26.0, 39.1, 63.1, 83.3, 131.8.

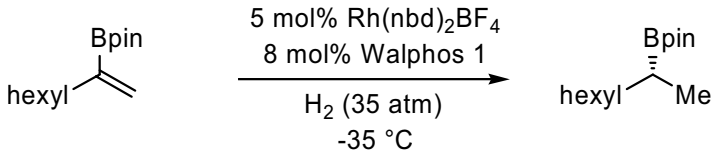



**2-{1-[2-(tert-butyl-dimethyl-silanyloxy)-ethyl]-vinyl}-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane.** IR (neat): 2979 (s), 2931 (m), 1733(s), 1370 (s), 1312 (s), 1142 (s) $cm^{-1}$. $^{1}H$ NMR (300 MHz, $CDCl_3$): δ 1.26 (12H, s), 1.44 (9H, s), 2.30-2.43 (2H, m), 5.61 (1H, br), 5.76 (1H, br). $^{13}C$ NMR (75.5 MHz, $CDCl_3$): δ 25.0, 28.4, 31.1, 35.3, 80.2, 83.6, 130.0, 173.0.




**t-Butyldimethyl(2-methyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-4-enyloxy)silane.** IR (neat): 2981 (s), 2929 (s), 2858 (s), 1615 (m), 1472 (s), 1372 (s), 1146 (s), 1092 (s) $cm^{-1}$. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 0.00 (6H, s), 0.81 (3H, d, J = 8.0 Hz), 0.87 (9H, s), 1.23 (12H, s), 1.70-1.90 (2H, m), 2.43-2.52 (1H, dd, J = 12, 8.0 Hz), 3.30 (1H, dd, J = 8.0, 8.0 Hz), 3.45 (1H, dd, J = 12, 8.0 Hz), 5.55 (1H, br), 5.77 (1H, br). $^{13}C$ NMR (100.6 MHz, $CDCl_3$): δ -5.3, 16.4, 18.1, 24.5, 25.8, 35.4, 39.0, 67.9, 83.1, 130.2.

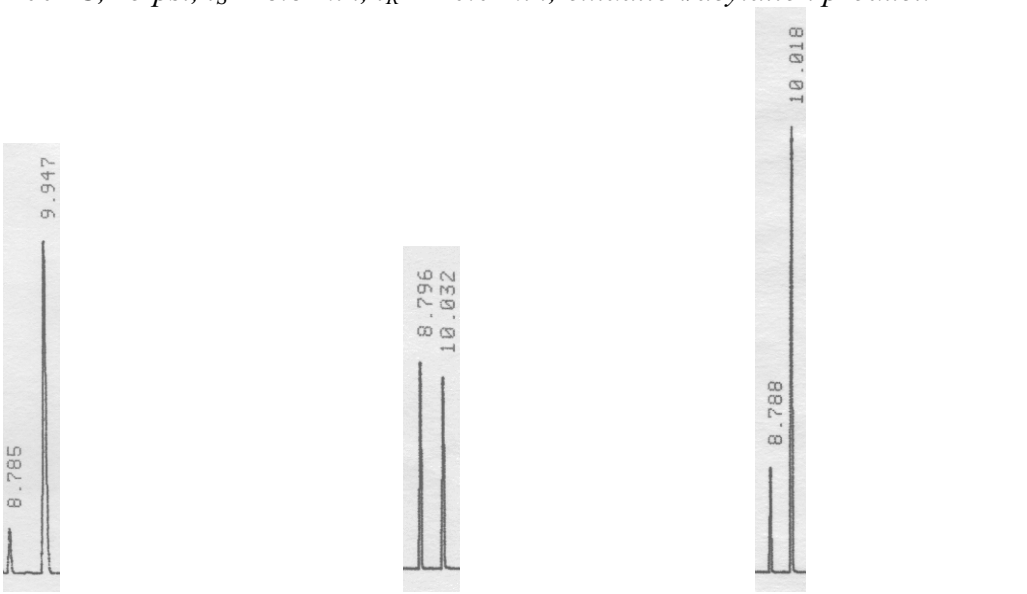
**Synthesis of 4,4,5,5-tetramethyl-2-(1-(R)-methyl-heptyl)-[1,3,2]dioxaborolane.[1]**




Reaction in 1,2-dichloroethane: A vial was charged with bis(norbornadiene)rhodium(I) tetrafluoroborate (3.9 mg, 0.010 mmol, 0.05 equiv), Walphos 1 (16 mg, 0.017 mmol, 0.08 equiv) and 1,2-dichloroethane (1 ml) in a dry-box and stirred at room temperature for 1 minute. 2-(1-Hexyl-vinyl)-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane (50 mg, 0.21 mmol, 1 equiv) was added to the catalyst solution and the vial was capped and removed from the dry-box. The cap was removed and the vial placed in a Parr Instruments pressure vessel. The vessel was partly submerged in a cryo-bath and cooled to approximately -35 °C, then purged with $H_2$. The vessel was charged with 30 bar $H_2$ and then depressurized. The vessel was re-charged with 30 bar $H_2$ and left stirring for 15 h. The pressure was released and the vessel removed from the cryo-bath. The vial was removed from the pressure vessel, and the volatiles were removed in vacuo. The resulting residue was purified by flash chromatography (20:1 hexanes/ethyl acetate) to furnish a colorless oil (50 mg, >95% yield). IR (neat): 2957 (m), 2927 (s), 2855 (m), 1464 (m), 1371 (s), 1313 (s), 1145 (s) $cm^{-1}$. $^1H$ NMR (300 MHz, $CDCl_3$): δ 0.83 (3H, t, J = 6.6 Hz), 0.94 (3H, br s), 1.1 -1.5 (27 H, m). $^{13}C$ NMR (75.5 MHz, $CDCl_3$): δ 14.0, 15.4, 22.6, 24.7 (2C), 28.9, 29.5, 31.8, 33.2, 82.7. GC of oxidation/acylation adduct: 85% ee; 100 °C, 25 psi, $t_S$=8.8 min, $t_R$=10.0 min.

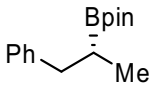
Reaction in toluene: A vial was charged with bis(norbornadiene)rhodium(I) tetrafluoroborate (3.9 mg, 0.010 mmol, 0.05 equiv), Walphos 1 (16 mg, 0.017 mmol, 0.08 equiv) and toluene (1 ml) in a dry-box and stirred at room temperature for 60 minutes. 2-(1-Hexyl-vinyl)-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane (50 mg, 0.21 mmol, 1 equiv) was added to the catalyst solution and the vial was capped and removed from the dry-box. The cap was removed and the vial placed in a Parr Instruments pressure vessel. The vessel was partly submerged in a cryo-bath and cooled to approx. -45 °C, then purged with $H_2$. The vessel was charged with 30 bar $H_2$ and then depressurized. The vessel was re-charged with 30 bar $H_2$ and left stirring for 40 h. The pressure was released and the vessel removed from the cryo-bath. The vial was taken from the pressure vessel, and the volatiles were removed in vacuo. The resulting residue was purified by flash chromatography (20:1 hexanes/ethyl acetate) to furnish a colorless oil (50 mg, >99% yield, 81% ee; GLC as above ).

*GC conditions: 100 °C, 25 psi, $t_S$ = 8.8 min, $t_R$ = 10.0 min; oxidation/acylation product.*



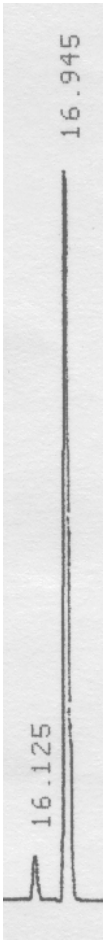

Product Racemate Product + Racemate

(1) Absolute configuration determined by GLC comparison to protected commercially available (*R*)-(-)-2-octanol.

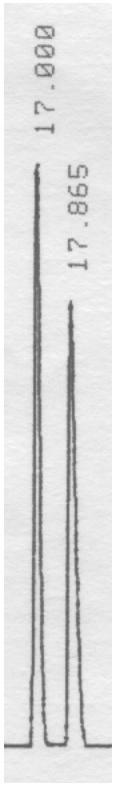



**4,4,5,5-tetramethyl-2-(1-(R)-methyl-2-phenyl-ethyl)-[1,3,2]dioxaborolane[2]:** IR (neat): 3062 (m), 3027 (m), 2979 (s), 2931 (s), 1457 (s), 1144 (s) $cm^{-1}$. $^{1}H$ NMR (300 MHz, $CDCl_3$): δ 0.94 (3H, d, J = 7.5 Hz), 1.17 (12H, s), 1.35 (1H, m), 2.54 (1H, dd, J = 13.8, 8.2 Hz), 2.78 (1H, dd, J = 13.5, 7.5 Hz), 7.16-7.28 (5H, m). $^{13}C$ NMR (75.5 MHz, $CDCl_3$): δ 15.2, 24.7 (2C), 39.0, 83.0, 125.5, 128.0, 128.0,142.3.

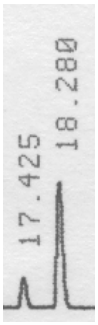
*GC conditions: 120 °C, 25 psi, $t_S$ = 17.0 min, $t_R$ = 17.9 min; oxidation/acylation product.*




Product

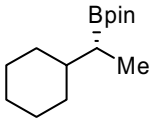



Racemate



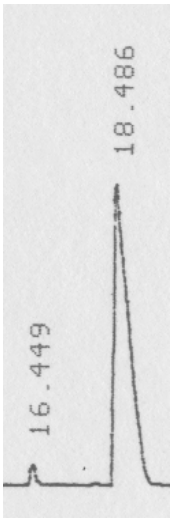

Product + Racemate

(2) Absolute configuration determined by GLC comparison to protected commercially available (*R*)-(-)-1-phenyl-2-propanol.

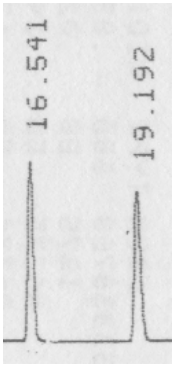



**2-(R)-(1-cyclohexyl-ethyl)-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane[3]:** IR (neat): 2979 (s), 2925 (s), 2852 (s), 1449 (s), 1380 (s), 1146 (s) $cm^{-1}$. $^{1}H$ NMR (300 MHz, $CDCl_3$): δ 0.9 - 1.40 (19H, s), 1.53-1.74 (6H, m), 1.95-2.05 (2H, m). $^{13}C$ NMR (100.5 MHz, $CDCl_3$): δ 13.5, 25.8, (2C), 27.7 , 32.8 (2C), 33.7 (2C), 41.5, 83.5. MS (A.P.C.I.+) m/z calc'd for $C_{14}H_{28}BO_2$: 239.2. Found: 239.1.

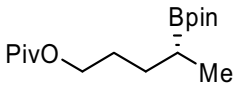
*GC conditions: 100 °C, 25 psi, $t_S$ = 16.5 min, $t_R$ = 19.2 min; oxidation/acylation product.*




Product

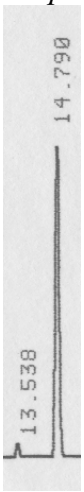



Racemate

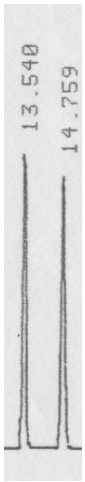



**2,2-dimethyl-propionic acid 4-(R)-(4,4,5,5-tetramethyl-[1,3,2]dioxaborolan-2-yl)-pentyl ester.** IR (neat): 2960 (s), 1723 (s), 1463 (s), 1382 (s) $cm^{-1}$. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 0.90-1.06 (4H, m), 1.15 (9H, s), 1.19 (12H, s), 1.23-1.37 (1H, m), 1.42-1.53 (1H, m), 1.54-1.65 (2H, m), 4.00 (2H, t, J = 6.6 Hz). $^{13}C$ NMR (100.5 MHz, $CDCl_3$): δ 15.4, 24.7, 27.2, 28.0, 29.4, 38.7, 64.6, 82.9, 178.8. MS (E.S.I.) m/z calc'd for $C_{16}H_{32}BO_4$: 299.2. Found: 299.2.

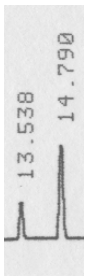
*GC conditions: 130 °C, 25 psi, $t_S$ = 13.5 min, $t_R$ = 14.8 min; oxidation/acylation product.*




Product

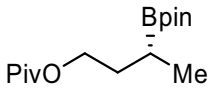



Racemate




Product + Racemate

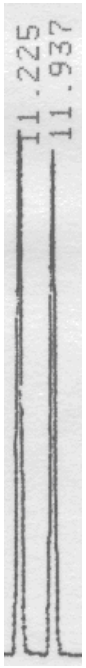
(3) Absolute configuration determined by comparison of derived Mosher's amide (vide infra) with authentic sample.




**2,2-dimethyl-propionic acid 3-(R)-(4,4,5,5-tetramethyl-[1,3,2]dioxaborolan-2-yl)-butyl ester[4]:** IR (neat): 2929 (m), 1723 (m), 1600 (s), 1470 (s) $cm^{-1}$. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 0.96 (3H, d, J = 7.6 Hz), 1.04-1.14 (1H, m), 1.16 (9H, s), 1.20 (12H, s), 1.51-1.63 (1H, m), 1.70-1.82 (1H, m), 3.98-4.10 (2H, m). $^{13}C$ NMR (100.5 MHz, $CDCl_3$): δ 15.0, 24.4, 24.5, 27.0, 31.4, 38.5, 63.7, 82.8, 178.4. MS (ESI) m/z calc'd for $C_{15}H_{30}BO_4$: 285.2. Found: 285.2

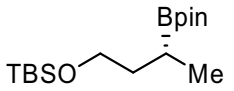
*GC conditions: 120 °C, 25 psi, $t_S$ = 11.2 min, $t_R$ = 11.9 min; oxidation/acylation product.*


Product



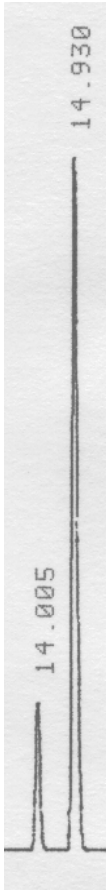

Racemate

(4) Absolute configuration determined by chiral GC analysis of protected commercially available (*R*)-(-)-1,3-butanediol.

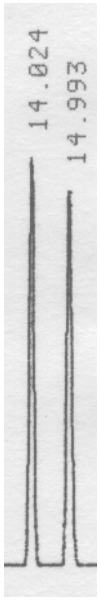



**(R)-tert-butyldimethyl(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butoxy)silane**[5]
IR (neat): 3072 (s), 2997 (s), 2866 (m), 2306 (s), 1434 (s), 1293 (s) $cm^{-1}$. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 0.02 (6H, s), 0.86 (9H, s), 0.95 (3H, d, J = 7.6 Hz), 1.04-1.13 (1H, m), 1.20 (12H, s), 1.42-1.52 (1H, m), 1.63-1.74 (1H, m), 3.53-3.65 (2H, m). $^{13}C$ NMR (100.5 MHz, $CDCl_3$): δ -4.8, 15.9, 18.8, 23.0, 25.1, 25.2, 26.4, 36.5, 63.0, 83.2. MS (ESI) m/z calc'd for $C_{16}H_{36}BO_3Si$: 315.2. Found: 315.2.

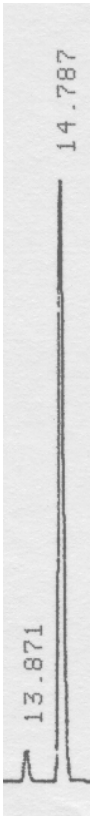
*GC conditions: 110 °C, 25 psi, $t_S$ = 14.0 min, $t_R$ = 15.0 min; oxidation/acylation product.*




Product+Racemate

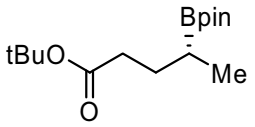



Racemate



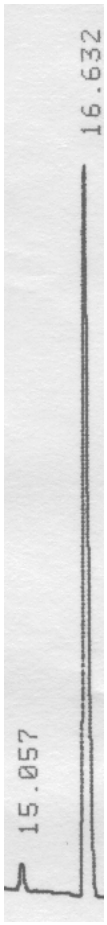

Product

(5) Absolute configuration determined by chiral GC analysis of protected commercially available (*R*)-(-)-1,3-butanediol.

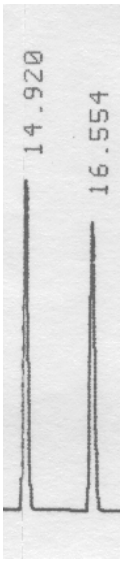



**(*R*)-tert-butyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate.** IR (neat): 2979 (s), 2933 (s), 2873 (s), 1735 (s), 1466 (m), 1364 (m), 1146 (m) $cm^{-1}$. $^{1}H$ NMR (300 MHz, $CDCl_3$): δ 0.94 (3H, s), 1.20 (12H, s), 1.40 (9H, s), 1.47-1.76 (3H, m), 2.20 (2H, t, J = 8.0 Hz). $^{13}C$ NMR (100 MHz, $CDCl_3$): δ 15.1, 24.5, 24.6, 27.9, 28.1, 34.7, 79.6, 82.7, 173.2. MS (ESI) m/z calc'd for $C_{15}H_{29}BO_4Na^+$: 307.2. Found: 307.4.

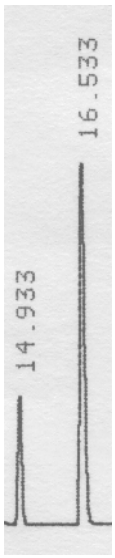
*GC conditions: 120 °C, 20 psi, $t_S$ = 14.9 min, $t_R$ = 16.6 min; oxidation/acylation product.*



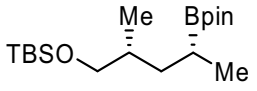

Product



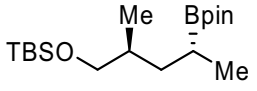

Racemate




Product + Racemate

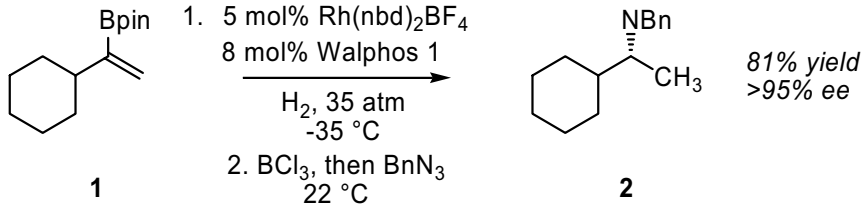



**t-butyldimethyl((2R,4R)-2-methyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentyloxy)silane[6]:** IR (neat): 2977 (s), 2929 (s), 2856 (s), 1472 (m), 1387 (s), 1315 (s), 1256 (s), 1146 (s), 1094 (s), 837 $cm^{-1}$. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 0.02 (6H, s), 0.82-1.15 (17H, m), 1.23 (12H, s), 1.45-1.67 (2H, m), 3.25 (1H, dd, J = 9.9, 7.2 Hz), 3.45 (1H, dd, J = 9.6, 5.1 Hz). $^{13}C$ NMR (100.5 MHz, $CDCl_3$): δ -5.5 (2C), 15.1, 16.4, 18.2, 24.5 (2C), 25.8, 34.2, 36.0, 68.5, 82.6. MS (ESI) m/z calc'd for $C_{18}H_{39}BO_3SiNa^+$: 365.3. Found: 365.4.



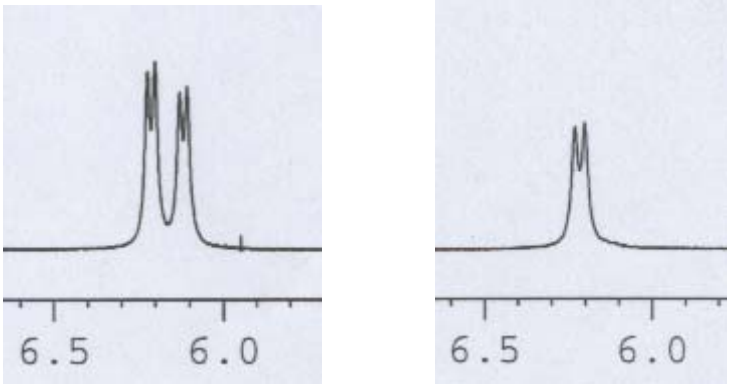

**t-butyldimethyl((2S,4R)-2-methyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentyloxy)silane[6]:** IR (neat): 2979 (s), 2931 (s), 2858 (s), 1459 (m), 1372 (s), 1318 (s), 1256 (s), 1146 (s), 1094 (s), 837 (s) $cm^{-1}$. $^{1}H$ NMR (300 MHz, $CDCl_3$): δ 0.02 (6H, s), 0.82-1.15 (17H, m), 1.23 (12H, s), 1.45-1.67 (2H, m), 3.25 (1H, dd, J = 9.9, 7.2 Hz), 3.45 (1H, dd, J = 9.6, 5.1 Hz). $^{13}C$ NMR (100.5 MHz, $CDCl_3$): δ -5.5 (2C), 16.0, 16.9, 18.2, 24.5 (2C), 25.8, 34.7, 36.8, 68.4, 82.6. MS (ESI) m/z calc'd for $C_{18}H_{39}BO_3SiNa^+$: 365.3. Found: 365.4..

(6) Relative stereochemistry verified by comparison of corresponding diol with literature $^{1}H$ NMR data: Chiarello, J.; Joullie, M. M. *Synth. Commun.* **1989**, *19*, 3379.



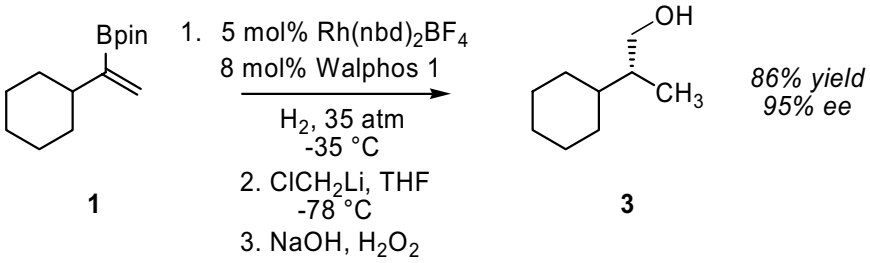

**Synthesis of (R)-N-benzyl-1-cyclohexylethanamine:** A vial was charged with bis(norbornadiene)rhodium(I) tetrafluoroborate (9.5 mg, 0.025 mmol, 0.05 equiv), Walphos 1 (38 mg, 0.041 mmol, 0.08 equiv) and 1,2-dichloroethane (2.4 ml) in a dry-box and stirred at room temperature for 1 minute. 2-(1-Cyclohexyl-vinyl)-4,4,5,5-tetramethyl-[1,3,2] dioxaborolane (120 mg, 0.51 mmol, 1 equiv) was added to the catalyst solution and the vial was capped and removed from the dry-box. The cap was removed and the vial placed in a Parr Instruments pressure vessel. The vessel was partly submerged in a cryo-bath and cooled to approx. -35 °C, then purged with $H_2$. The vessel was charged with 30 bar $H_2$ and then depressurized. The vessel was re-charged with 30 bar $H_2$ and left stirring for 15 h. The pressure was released and the vessel removed from the cryo-bath. The vial was taken from the pressure vessel, capped, and allowed to warm to ambient temperature. The vial was then taken back into the dry-box and boron trichloride (1M in $CH_2Cl_2$, 1.0 ml, 1.0 mmol, 2 equiv) was added. After 3 h, benzyl azide (0.16 ml, 1.3 mmol, 2.5 equiv) was added carefully (nitrogen evolution observed) and the mixture stirred for a further 4 h. The vial was removed from the dry-box and aqueous NaOH solution (2M, 1 ml) was added. The mixture was stirred for 0.5 h at ambient temperature, and then extracted with EtOAc. Evaporation of the volatiles in vacuo provided the crude residue, which was purified by flash chromatography (9:1 hexanes/EtOAc) to provide a colorless oil (91 mg, 82%). Spectral data are in agreement with that reported for this compound.[7] $^1$H NMR (400 MHz, CDCl3): δ 0.95 (3H, d, J = 5.3 Hz), 1.00-1.32 (7H, m), 1.50-1.72 (5H, m), 2.40-2.44 (1H, m), 3.62 (1H, d, J = 13.2 Hz), 3.76 (1H, d, J = 13.2 Hz), 7.18-7.27 (5H, m). $^{13}$C NMR (100.5 MHz, $CDCl_3$): δ 17.1, 27.0, 26.8, 26.9, 28.3, 30.1, 43.2, 51.8, 57.2, 127.0, 128.4, 128.6, 141.3.

*$^1$H NMR of debenzylated Mosher's amide; NH resonance:*

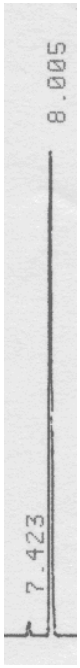



racemic reaction product

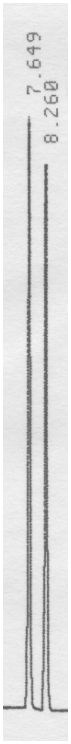
(7) Data consistent with literature: Majewski, M.; Gleave, D. M. *J. Org. Chem.* **1992**, *57*, 3599.




**Synthesis of (R)-2-cyclohexylpropan-1-ol:** A vial was charged with bis(norbornadiene)rhodium(I) tetrafluoroborate (9.5 mg, 0.025 mmol, 0.05 equiv), Walphos 1 (38 mg, 0.041 mmol, 0.08 equiv) and 1,2-dichloroethane (2.4 ml) in a dry-box and stirred at room temperature for 1 minute. 2-(1-Cyclohexyl-vinyl)-4,4,5,5-tetramethyl-[1,3,2] dioxaborolane (120 mg, 0.51 mmol, 1 equiv) was added to the catalyst solution and the vial was capped and removed from the dry-box. The cap was removed and the vial placed in a Parr Instruments pressure vessel. The vessel was partly submerged in a cryo-bath and cooled to approx. -35 °C, then purged with $H_2$. The vessel was charged with 30 bar $H_2$ and then depressurized. The vessel was re-charged with 30 bar $H_2$ and left stirring for 15 h. The pressure was released and the vessel removed from the cryo-bath. The vial was taken from the pressure vessel and the solvent removed in vacuo. THF (2 ml) was added followed by bromochloromethane (43 µl, 0.66 mmol, 1.3 equiv). The reaction mixture was then cooled to – 78 °C and n-BuLi (2.5 M in hexanes, 0.26 ml, 0.66 mmol, 1.3 equiv) added dropwise. The reaction was allowed to warm to ambient temperature overnight and then 30% aqueous hydrogen peroxide solution (0.5 ml) and aqueous NaOH solution (2M, 0.5 ml) were added. After a further 2.5 h, the reaction mixture was extracted with EtOAc and concentrated in vacuo. Purification by flash chromatography (9:1 hexanes/EtOAc) provided the product as a colorless oil (62 mg, 85 %). Spectral data are in agreement with that reported for this compound.[8] $^1$H NMR (300 MHz, $CDCl_3$): δ 0.88 (3H, d, J = 6.9 Hz), 0.90-1.40 (6H, m), 1.40-1.55 (1H, m), 1.60-1.80 (5H, m), 3.43 (1H, dd, J = 10.5, 6.9 Hz), 3.59 (1H, dd, J = 10.5, 5.7 Hz). $^{13}$C NMR (75 MHz, $CDCl_3$): δ 13.4, 26.8, 26.9, 27.0, 29.0, 31.2, 39.6, 41.2, 66.5.




Product

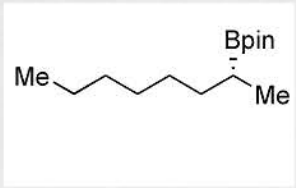



Racemate

(8) Data consistent with literature: Kondakov, D. Y.; Negishi, E.-I. *J. Am. Chem. Soc.* **1995**, *117*, 10771.

WM-194B

No parameters

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76.639

33.217
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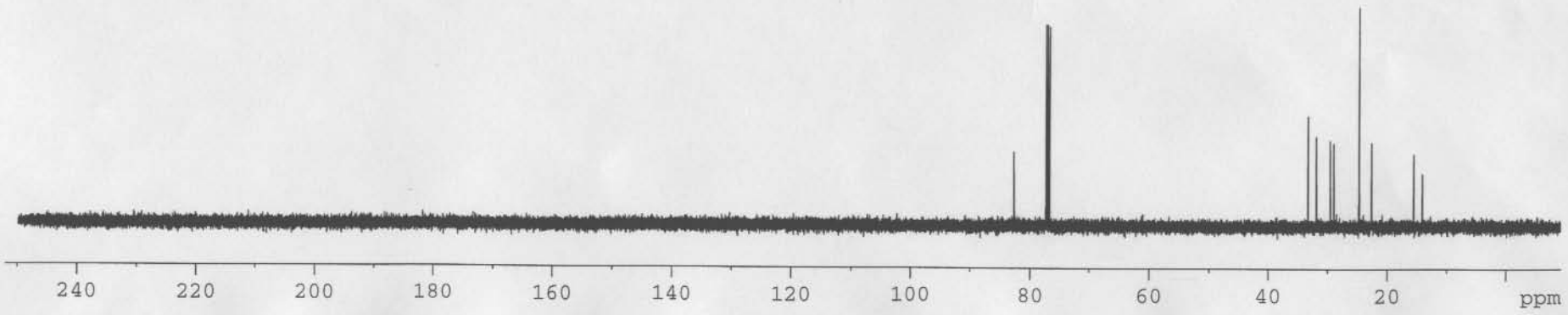
22.586
15.448
14.009



WM2-7B

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PROCNO                     1

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DS                        2
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DE                  168.57 usec
TE                   293.0 K
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P1                   11.50 usec
SFO1           300.1319000 MHz
NUCLEUS                1H

F2 - Processing parameters
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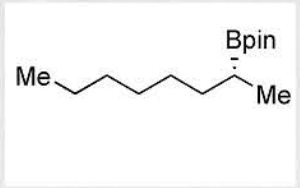
7.240

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1.369
1.243
1.238
1.210
1.171
0.999
0.982
0.936
0.859
0.846
0.824

8.5 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 ppm

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WM-242B.1

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PROCNO                 1

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DS                    2
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AQ              1.2517875 sec
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DW                19.100 usec
DE                32.36 usec
TE                300.0 K
D1              1.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   7.25 usec
PL1                  0.00 dB
SFO1         100.5418136 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
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
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76.716
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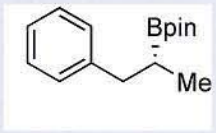
24.694
15.191

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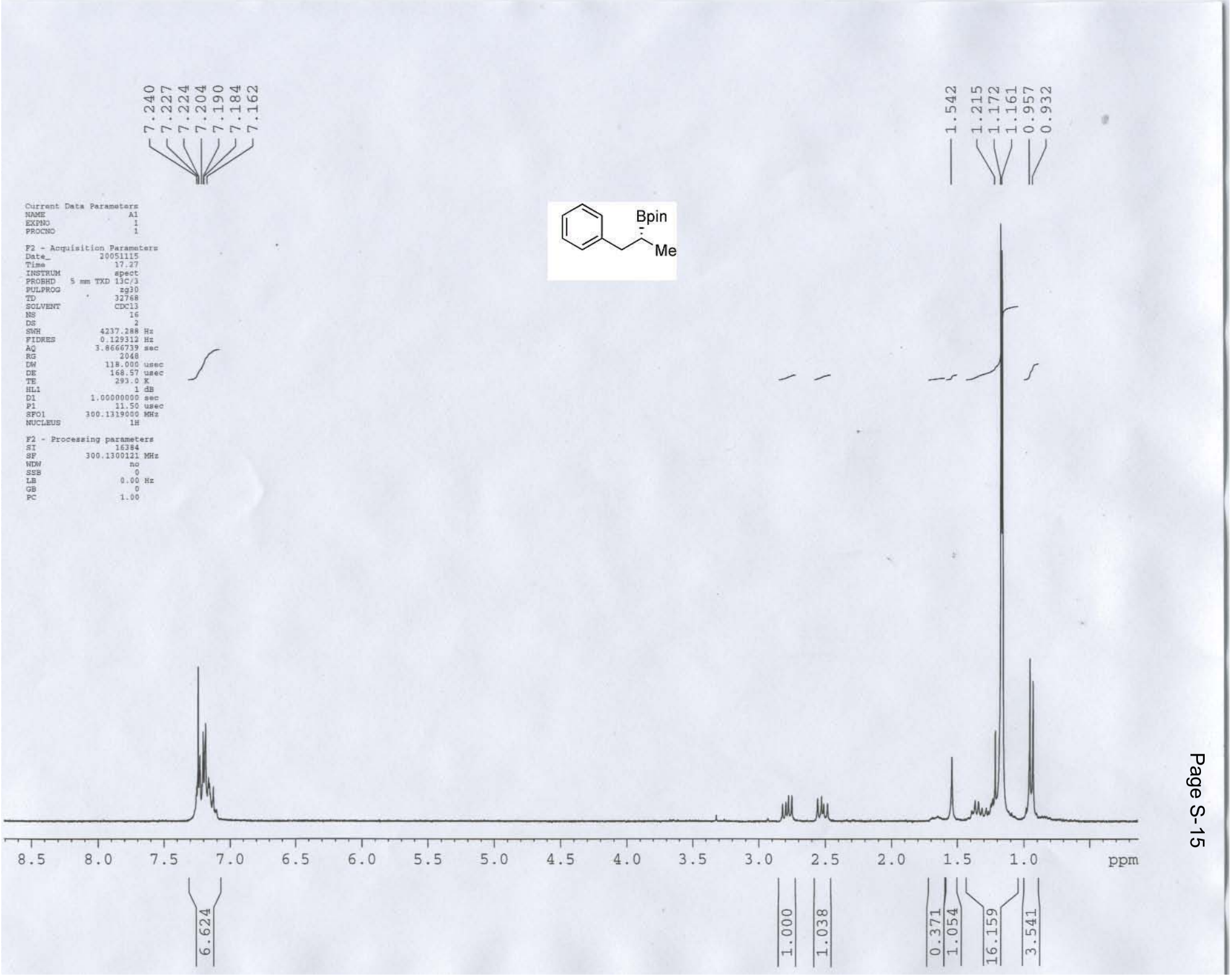
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7.204
7.190
7.184
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1.542
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1.172
1.161
0.957
0.932
Current Data Parameters
NAME A1
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PROCNO 1
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Date_ 20051115
Time 17.27
INSTRUM spect
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4237.288 Hz
FIDRES 0.129312 Hz
AQ 3.8666719 sec
RG 2048
DW 118.000 usec
DE 168.57 usec
TE 293.0 K
HL1 1 dB
D1 1.00000000 sec
P1 11.50 usec
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NUCLEUS 1H
F2 - Processing parameters
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SF 300.1300121 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Bpin
Me
8.5
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6.5
6.0
5.5
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1.038
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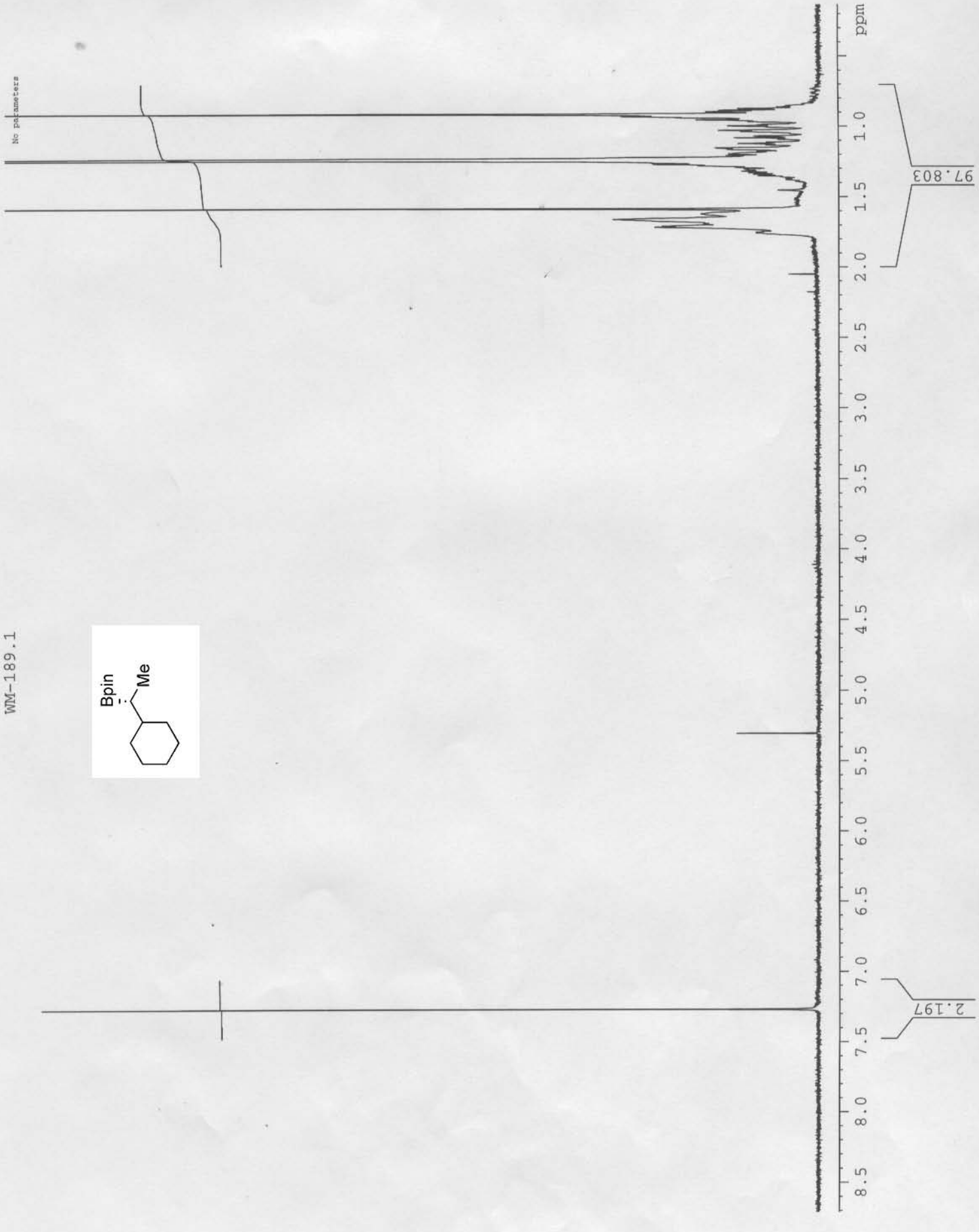
1.054
16.159
3.541


No parameters
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ppm
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4.5
4.0
3.5
3.0
2.5
2.0
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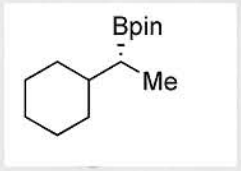
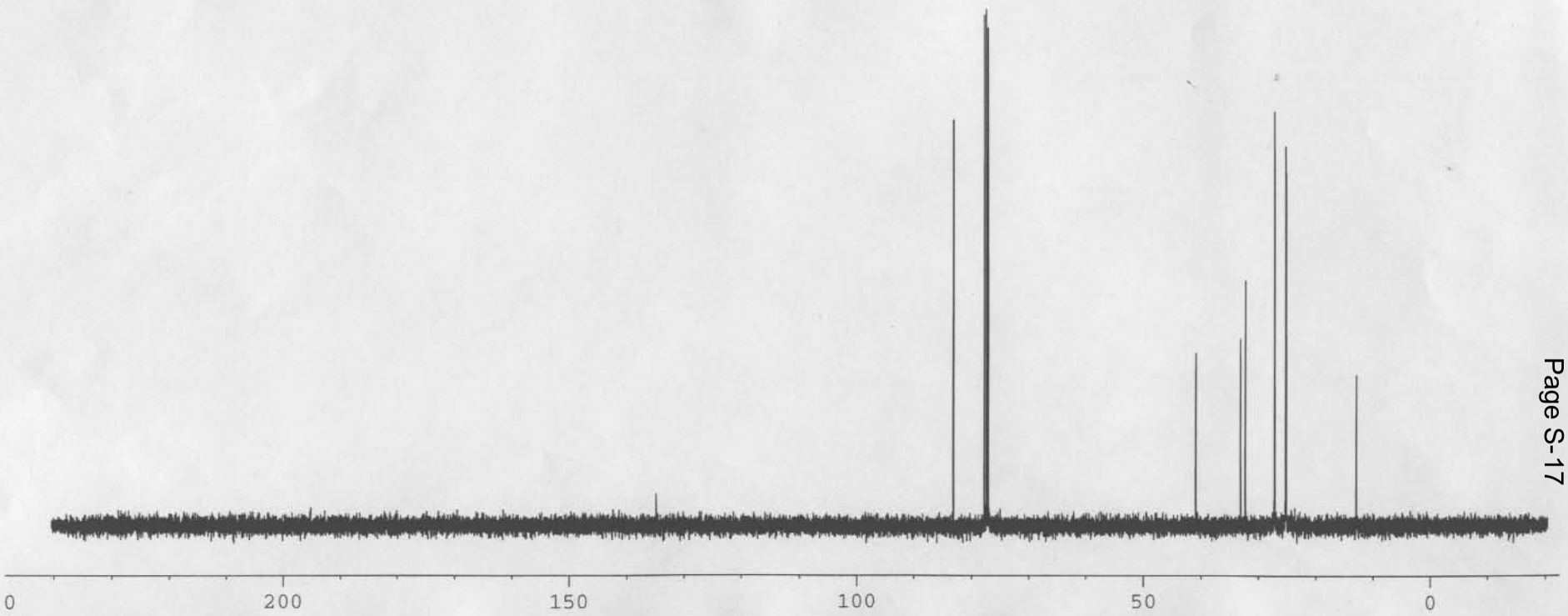
1.0

WM-189



WM2-41A

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PROCNO                1

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TD                65536
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DS                    2
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FIDRES            0.399445 Hz
AQ            1.2517875 sec
RG                32768
DW               19.100 usec
DE               32.36 usec
TE               300.0 K
D1            1.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

======== CHANNEL f1 ========
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P1                 7.25 usec
PL1                0.00 dB
SFO1         100.5418136 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              90.00 usec
PL2                -3.00 dB
PL12               18.90 dB
PL13               22.00 dB
SFO2         399.8015992 MHz

F2 - Processing parameters
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WDW                   no
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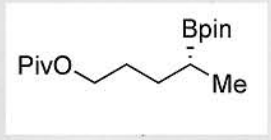
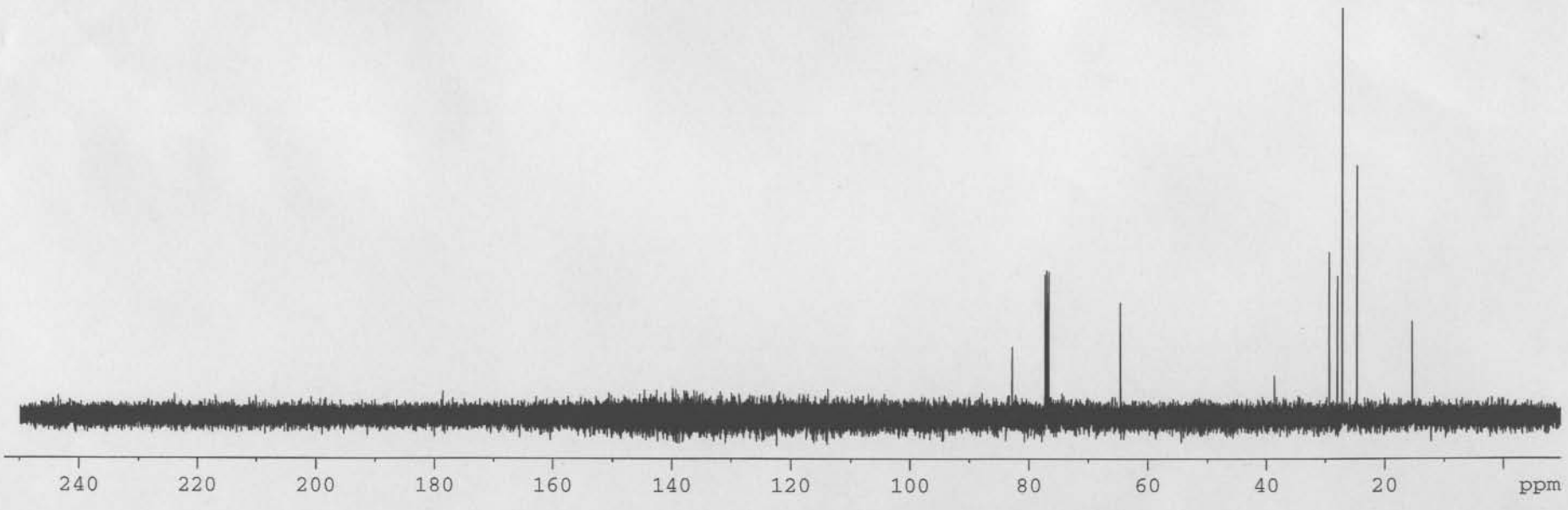
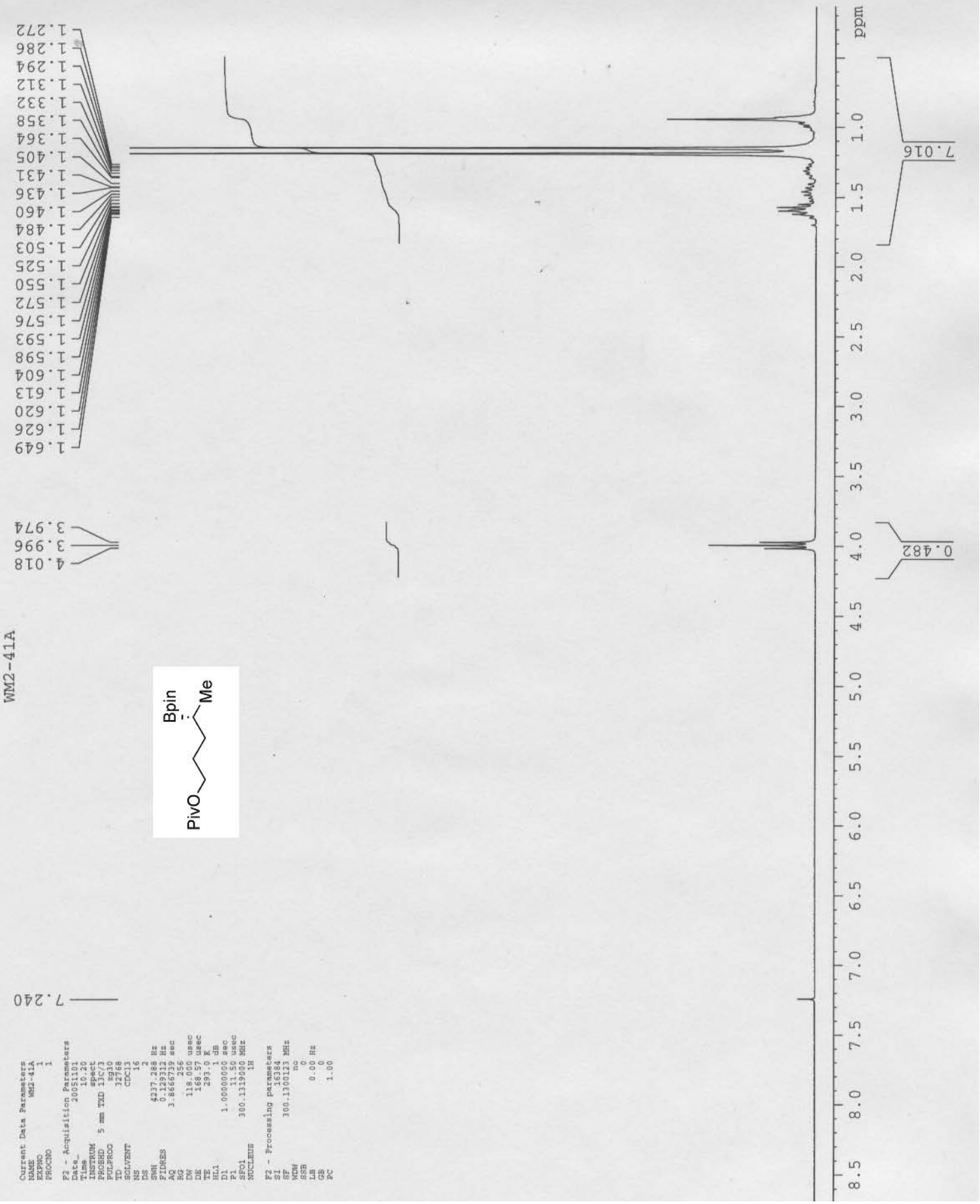



82.867
77.311
76.995
76.678
64.624
38.688
29.379
27.960
27.182
24.729
24.676
15.417

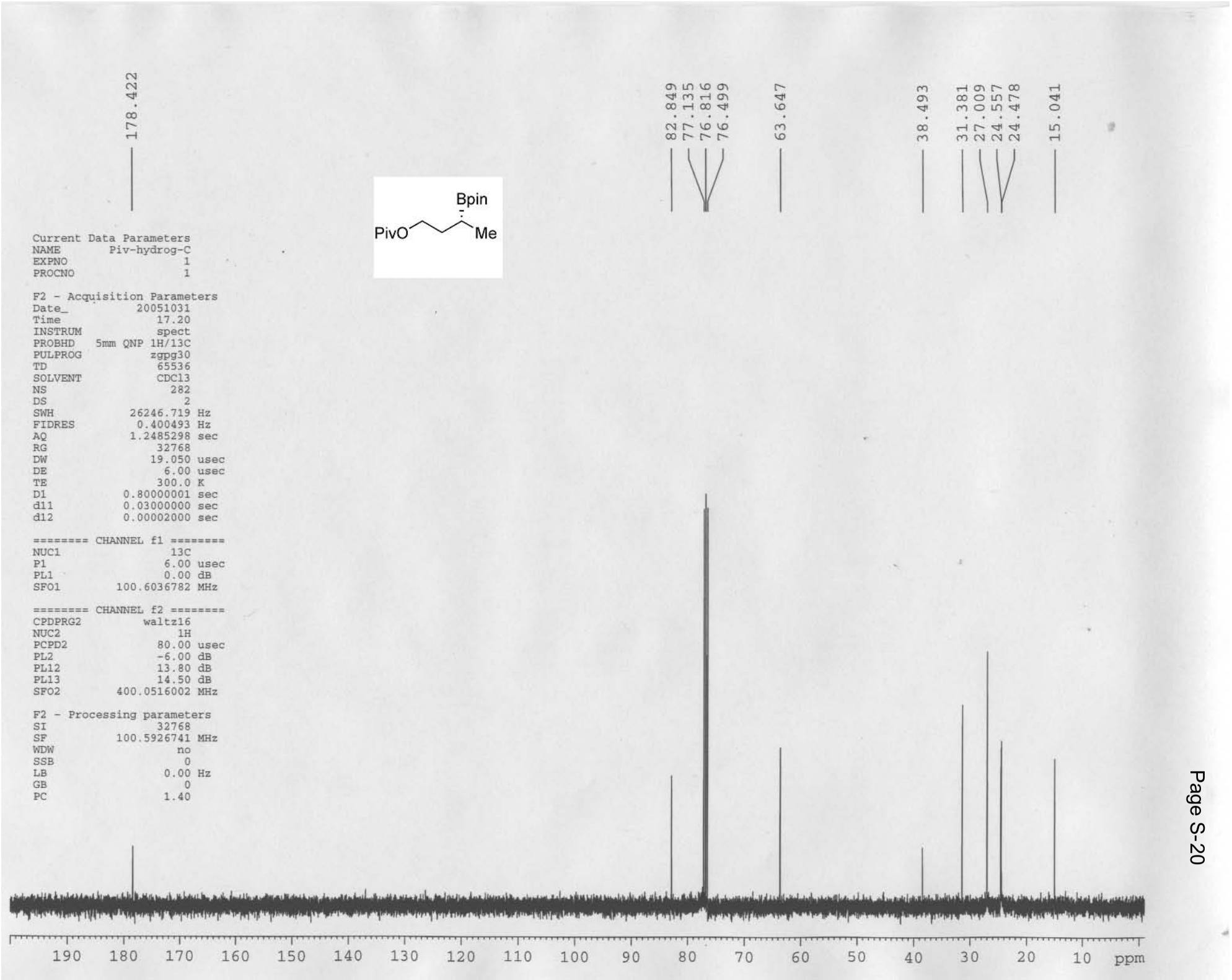




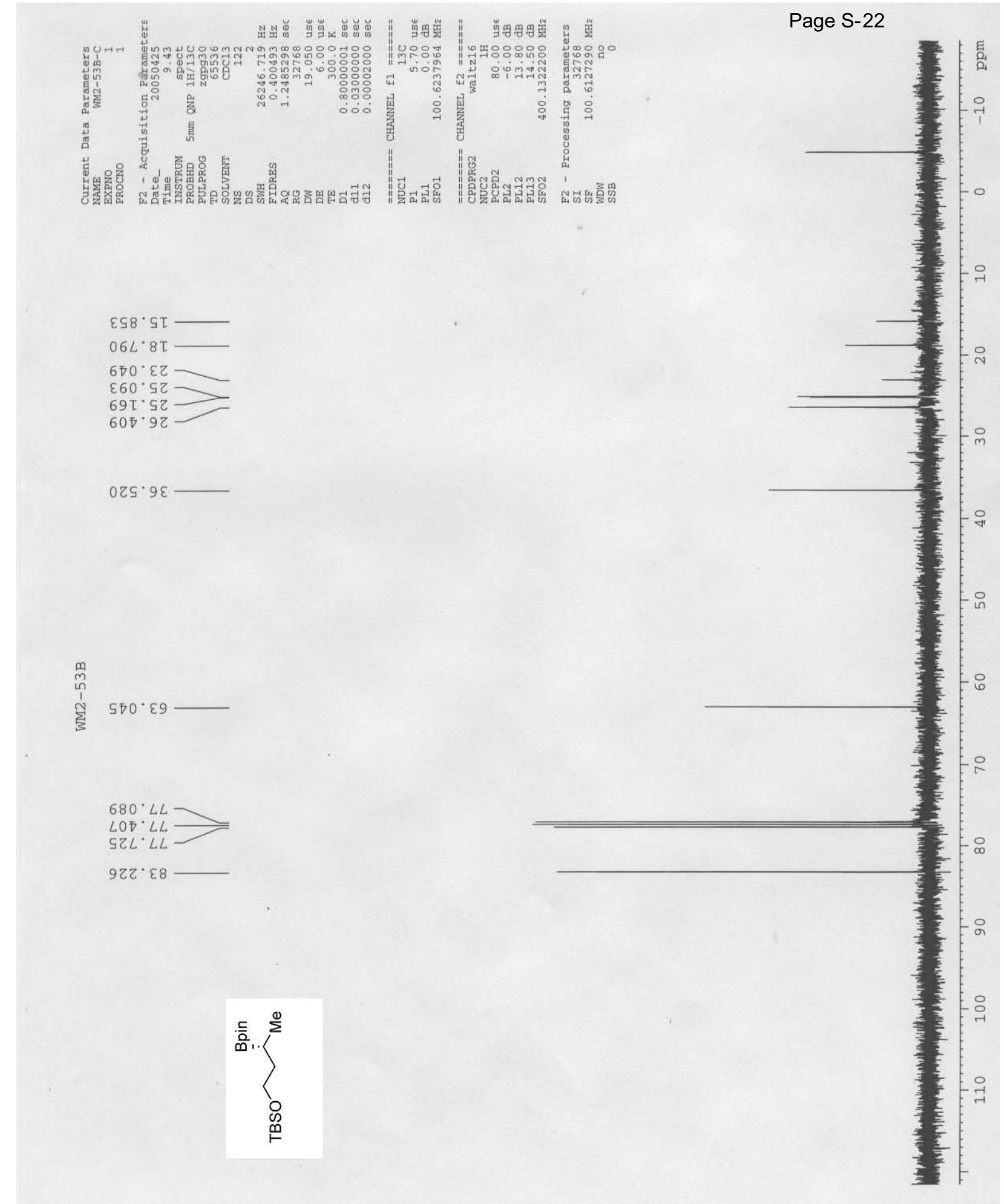

Current Data Parameters
NAME WM2-41A
EXPNO 1
PROCNO 1

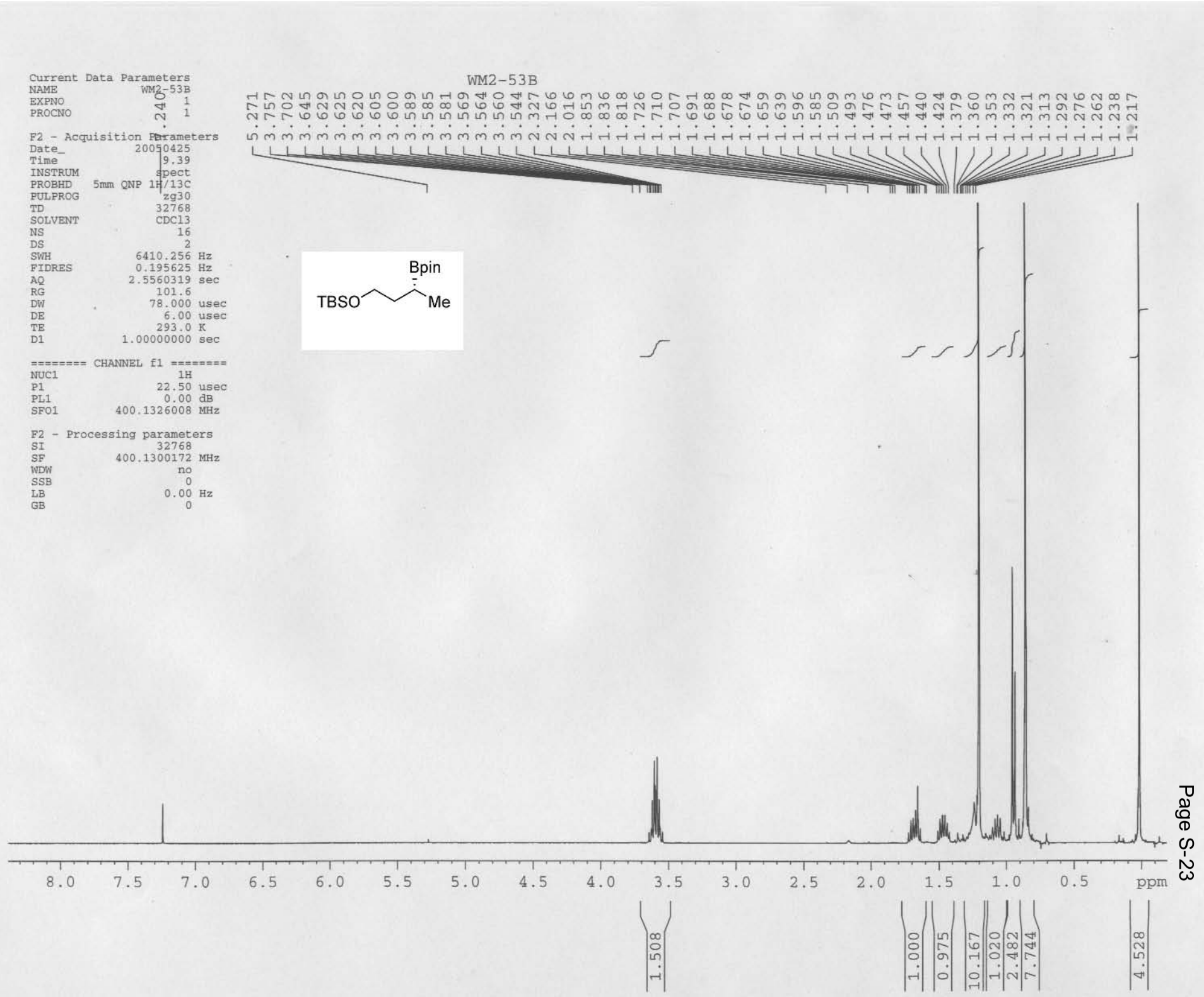
F2 - Acquisition Parameters
Date_ 20051101
Time 10.20
INSTRUM spect
PROBHD 5 mm TXD 13C/3
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4237.288 Hz
FIDRES 0.129312 Hz
AQ 3.8666739 sec
RG 256
DW 118.000 usec
DE 168.57 usec
TE 293.0 K
HL1 1 dB
D1 1.00000000 sec
P1 11.50 usec
SFO1 300.1319000 MHz
NUCLEUS 1H

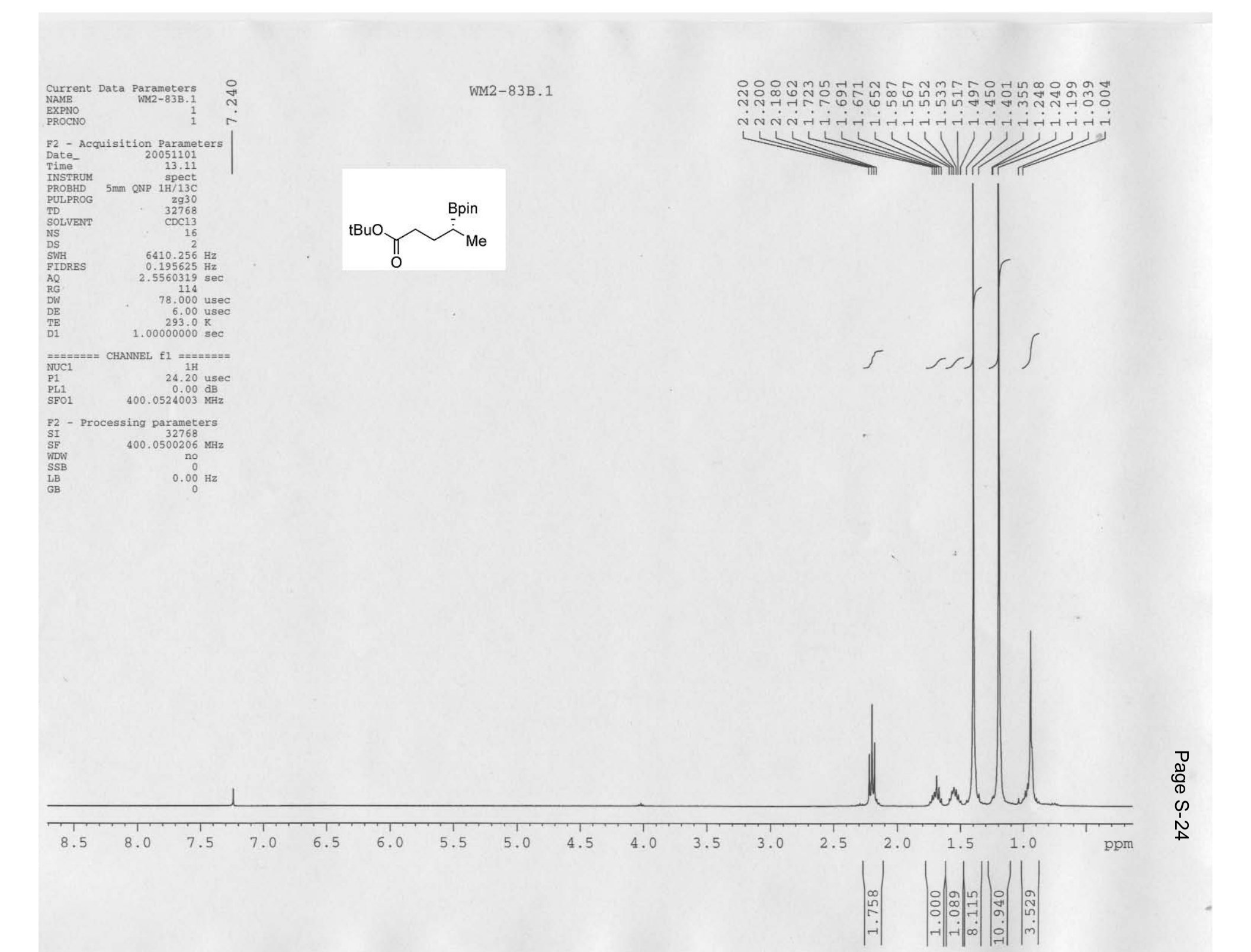
F2 - Processing parameters
SI 16384
SF 300.1300123 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00


178.422
82.849
77.135
76.816
76.499
63.647
38.493
31.381
27.009
24.557
24.478
15.041
Bpin
PivO
Me
Current Data Parameters
NAME Piv-hydrog-C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051031
Time 17.20
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 282
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 32768
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 6.00 usec
PL1 0.00 dB
SFO1 100.6036782 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.0516002 MHz
F2 - Processing parameters
SI 32768
SF 100.5926741 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

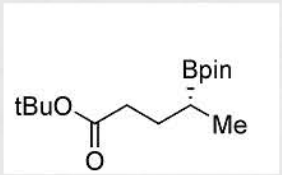

7.240
4.070
4.064
4.052
4.047
4.035
4.029
1.792
1.775
1.758
1.741
1.586
1.568
1.551
1.534
1.206
1.157
1.122
1.103
1.084
1.066
0.995
0.982
0.964
Bpin
PivO
Me
Current Data Parameters
NAME Piv-hydrog
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051031
Time 17.14
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5560319 sec
RG 143.7
DW 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 24.20 usec
PL1 0.00 dB
SFO1 400.0524003 MHz
F2 - Processing parameters
SI 32768
SF 400.0500204 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.5
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
ppm
1.833
1.000
1.204
20.411
2.695


WM2-53B
Current Data Parameters
NAME WM2-53B-C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050425
Time 9.43
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 122
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 32768
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
15.853
18.790
23.049
25.093
25.169
26.409
36.520
63.045
77.089
77.407
77.725
83.226
Bpin
Me
TBSO
ppm


Current Data Parameters
NAME WM2-53B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050425
Time 9.39
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 101.6
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
WM2-53B
5.271
3.757
3.702
3.645
3.629
3.625
3.620
3.605
3.600
3.589
3.585
3.581
3.569
3.564
3.560
3.544
2.327
2.166
2.016
1.853
1.836
1.818
1.726
1.710
1.707
1.691
1.688
1.678
1.674
1.659
1.639
1.596
1.585
1.509
1.493
1.476
1.473
1.457
1.440
1.424
1.379
1.360
1.353
1.332
1.321
1.313
1.292
1.276
1.262
1.238
1.217
Bpin
TBSO
Me
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
1.508
1.000
0.975
10.167
1.020
2.482
7.744
4.528


Current Data Parameters
NAME WM2-83B.1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051101
Time 13.11
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 114
DW 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 24.20 usec
PL1 0.00 dB
SFO1 400.0524003 MHz
F2 - Processing parameters
SI 32768
SF 400.0500206 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
7.240
WM2-83B.1
Bpin
tBuO
Me
O
2.220
2.200
2.180
2.162
1.723
1.705
1.691
1.671
1.652
1.587
1.567
1.552
1.533
1.517
1.497
1.450
1.401
1.355
1.248
1.240
1.199
1.039
1.004
8.5
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
ppm
1.758
1.000
1.089
8.115
10.940
3.529

WM2-83B.1



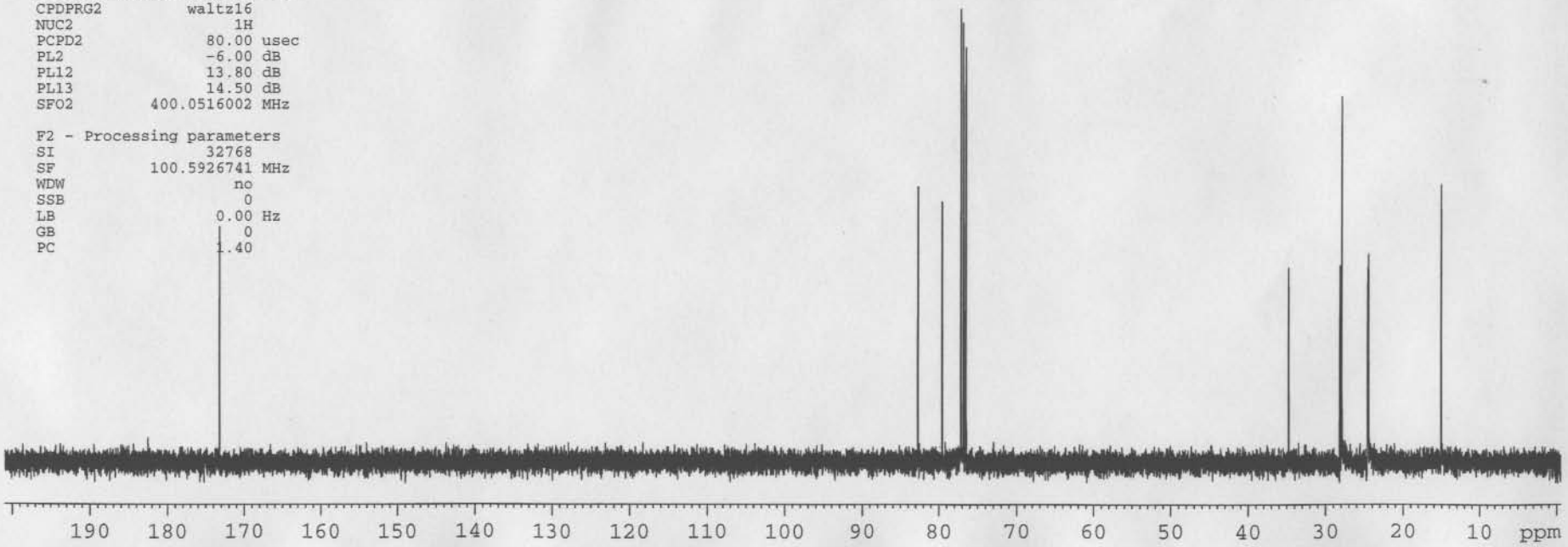

173.206

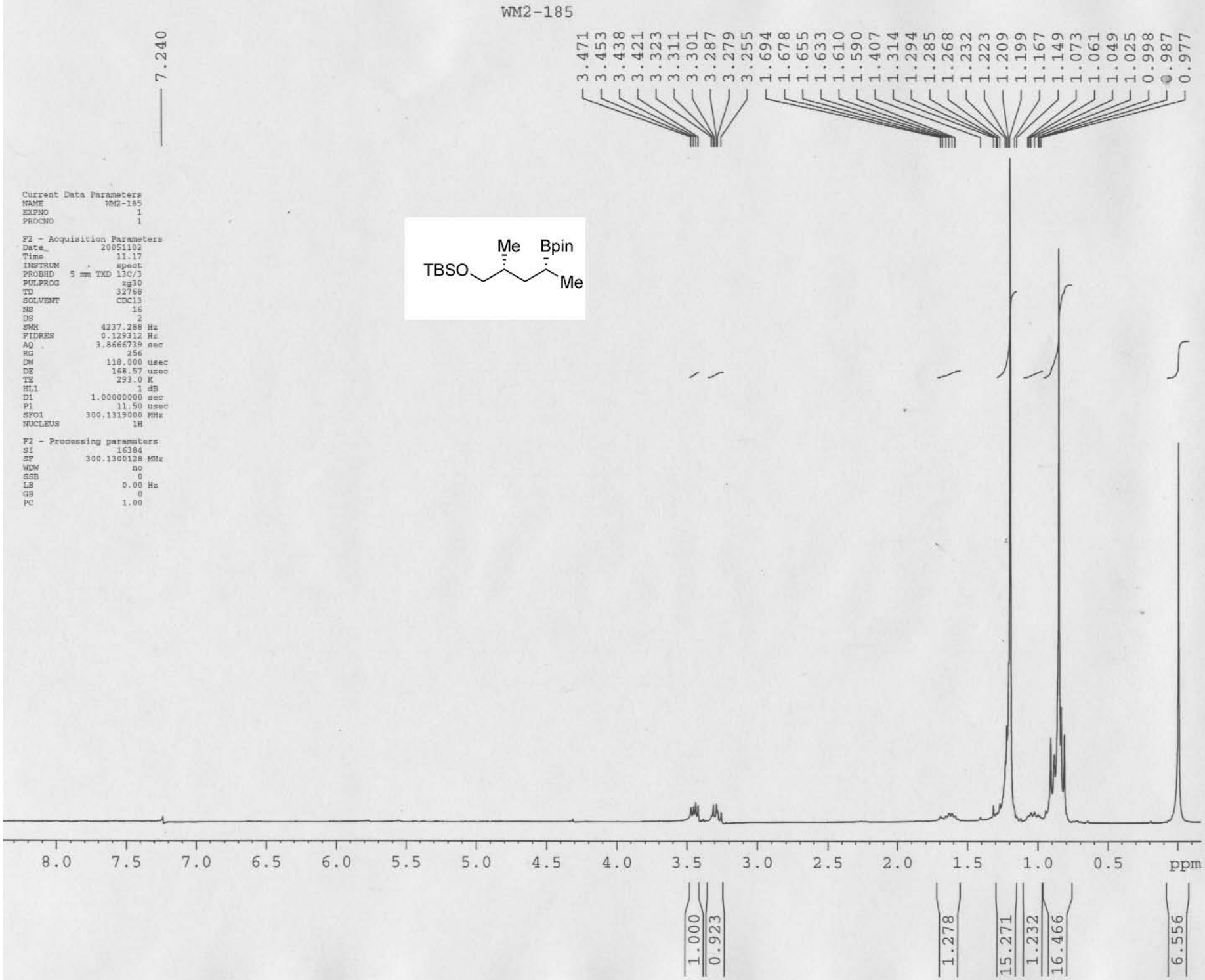
82.725
79.572
77.136
76.819
76.501

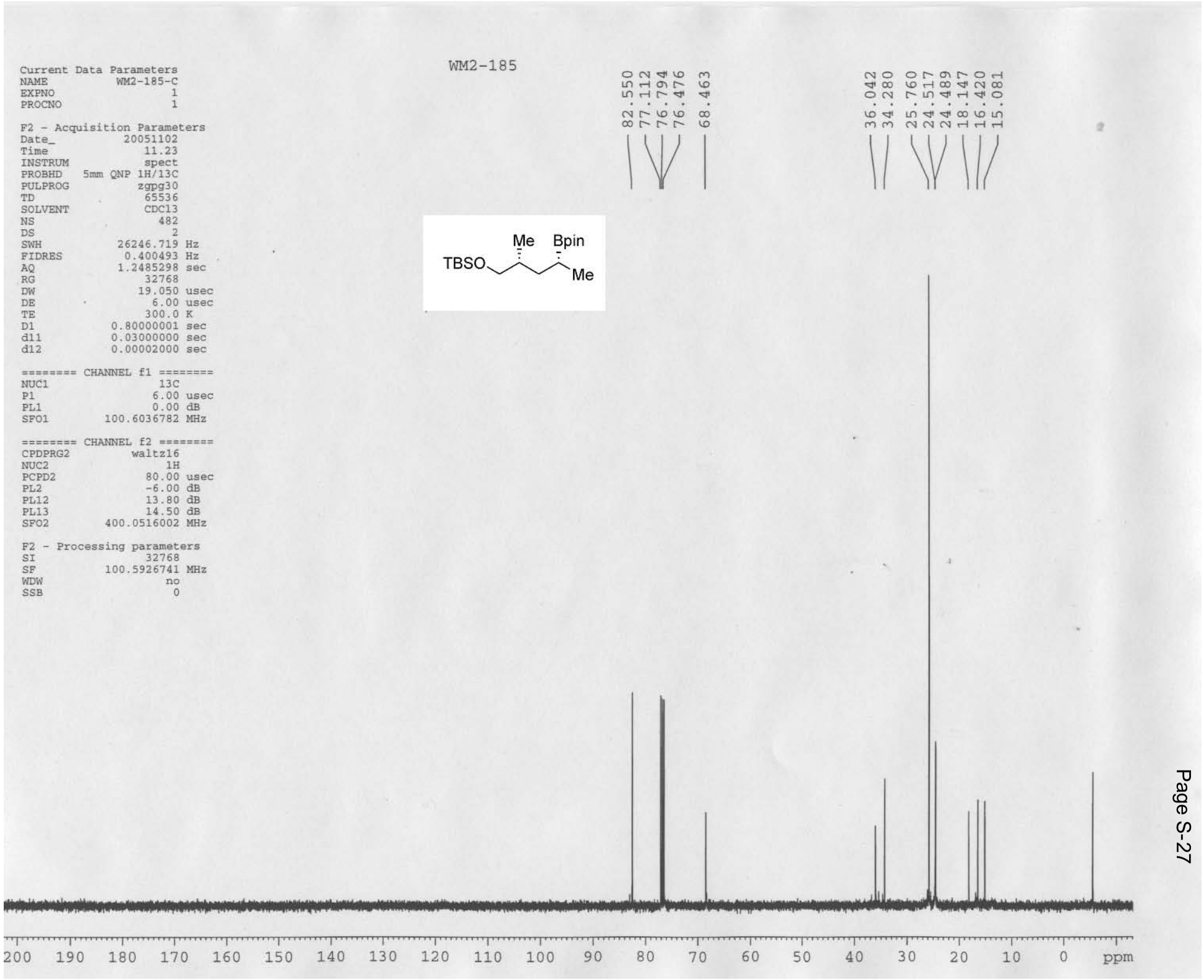
34.749
28.149
27.909
24.557
24.474

15.050

```
Current Data Parameters
NAME         WM2-83B.1-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20051101
Time               17.21
INSTRUM             spect
PROBHD   5mm QNP 1H/13C
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   256
DS                     2
SWH            26246.719 Hz
FIDRES          0.400493 Hz
AQ             1.2485298 sec
RG                 32768
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                 6.00 usec
PL1                0.00 dB
SFO1         100.6036782 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -6.00 dB
PL12              13.80 dB
PL13              14.50 dB
SFO2         400.0516002 MHz

F2 - Processing parameters
SI                 32768
SF           100.5926741 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
```




WM2-185
7.240
3.471
3.453
3.438
3.421
3.323
3.311
3.301
3.287
3.279
3.255
1.694
1.678
1.655
1.633
1.610
1.590
1.407
1.314
1.294
1.285
1.268
1.232
1.223
1.209
1.199
1.167
1.149
1.073
1.061
1.049
1.025
0.998
0.987
0.977
Current Data Parameters
NAME WM2-185
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051102
Time 11.17
INSTRUM spect
PROBHD 5 mm TXD 13C/3
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4237.288 Hz
FIDRES 0.129312 Hz
AQ 3.8666739 sec
RG 256
DW 118.000 usec
DE 168.57 usec
TE 293.0 K
HL1 1 dB
D1 1.00000000 sec
P1 11.50 usec
SFO1 300.1319000 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 16384
SF 300.1300128 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
TBSO
Me
Bpin
Me
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
1.000
0.923
1.278
15.271
1.232
16.466
6.556



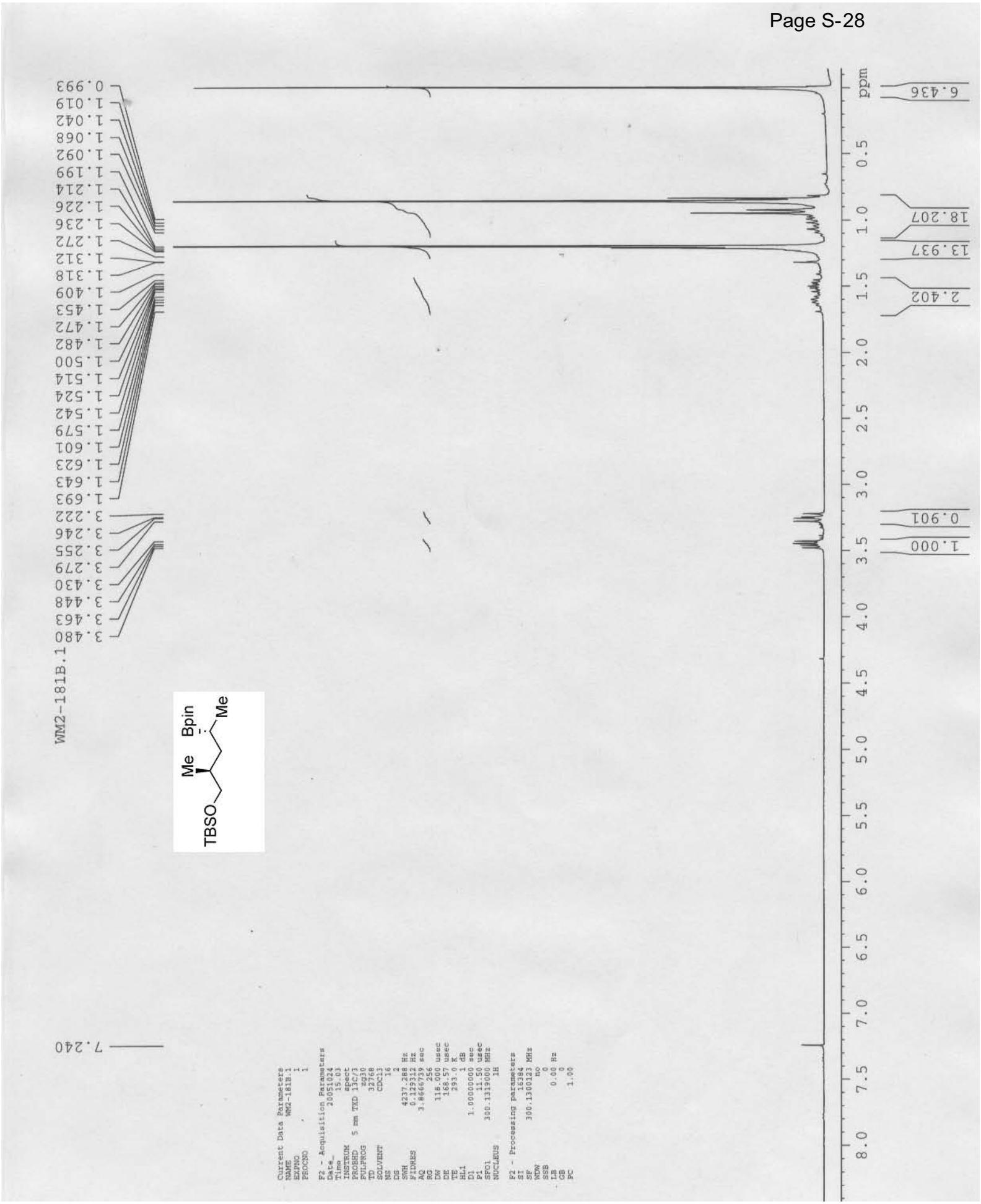

Current Data Parameters
NAME WM2-185-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051102
Time 11.23
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 482
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 32768
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 6.00 usec
PL1 0.00 dB
SFO1 100.6036782 MHz

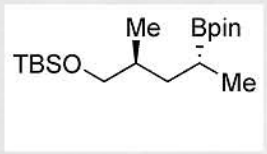
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.0516002 MHz

F2 - Processing parameters
SI 32768
SF 100.5926741 MHz
WDW no
SSB 0


WM2-181B.1
TBSO
Me
Bpin
Me
7.240
3.480
3.463
3.448
3.430
3.279
3.255
3.246
3.222
1.693
1.643
1.623
1.601
1.579
1.542
1.524
1.514
1.500
1.482
1.472
1.453
1.409
1.318
1.312
1.272
1.236
1.226
1.214
1.199
1.092
1.068
1.042
1.019
0.993
6.436
18.207
13.937
2.402
0.901
1.000
ppm
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
Current Data Parameters
NAME WM2-181B.1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051024
Time 15.03
INSTRUM spect
PROBHD 5 mm TXD 13C/3
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4237.288 Hz
FIDRES 0.129312 Hz
AQ 3.8666739 sec
RG 256
DW 118.000 usec
DE 168.57 usec
TE 293.0 K
D1 1 dB
D1 1.00000000 sec
P1 11.00000 usec
SFO1 300.1319000 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 16384
SF 300.1300123 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

WM2-181B

```
Current Data Parameters
NAME          WM2-181B-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20051102
Time               12.24
INSTRUM            spect
PROBHD   5mm QNP 1H/13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    2
SWH           26246.719 Hz
FIDRES         0.400493 Hz
AQ             1.2485298 sec
RG                32768
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                 6.00 usec
PL1                0.00 dB
SFO1        100.6036782 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2              80.00 usec
PL2                -6.00 dB
PL12               13.80 dB
PL13               14.50 dB
SFO2        400.0516002 MHz

F2 - Processing parameters
SI                 32768
SF          100.5926741 MHz
WDW                   no
SSB                    0
```

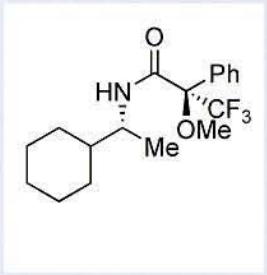


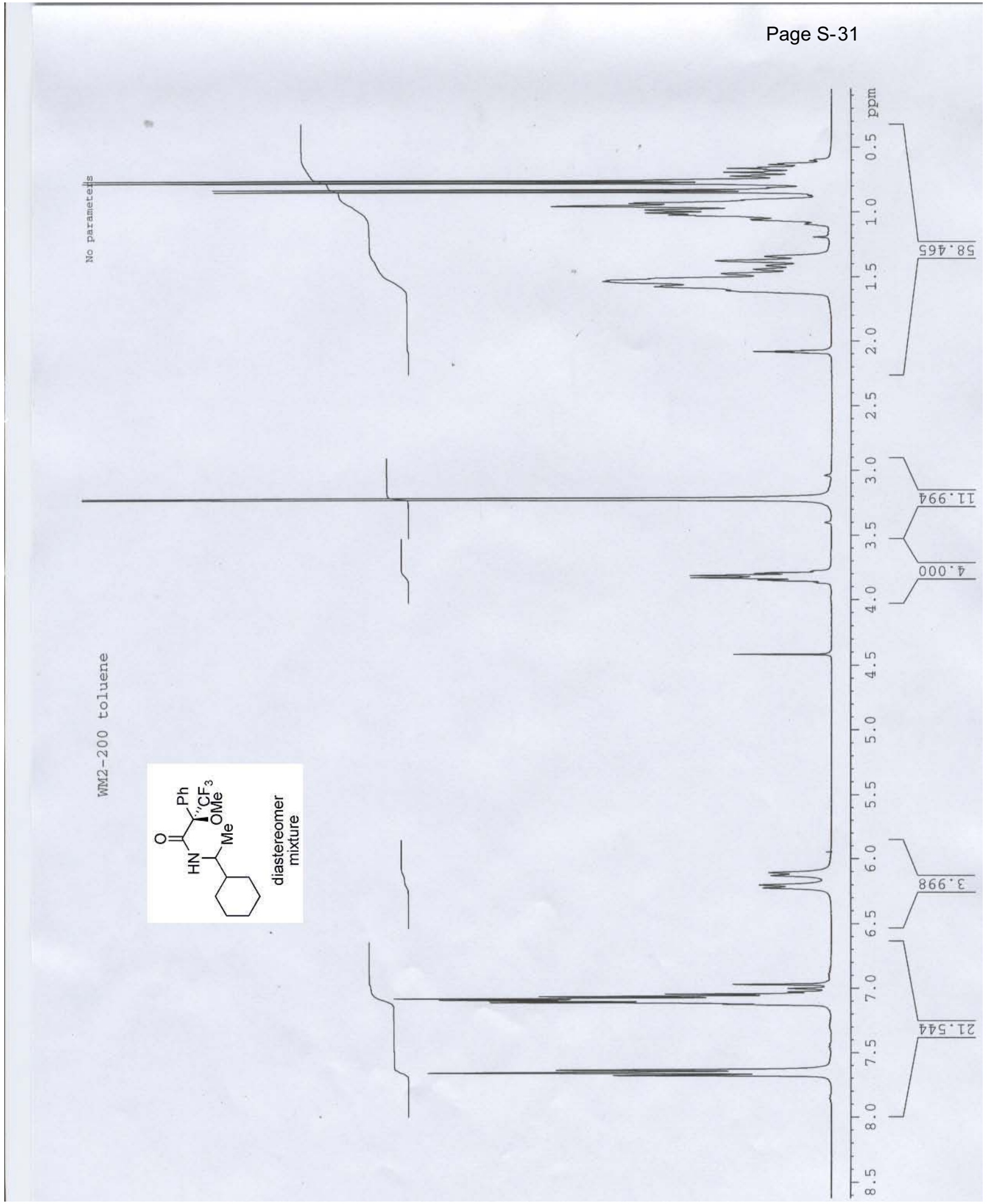

82.549
77.121
76.803
76.487
68.368
36.765
34.703
25.775
24.504
18.148
16.874
15.988

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

WM2-201 in toluene

```
Current Data Parameters
NAME                  WM2-201T
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20051220
Time                 16.46
INSTRUM              spect
PROBHD   5 mm TXD 13C/3
PULPROG              zg30
TD                  32768
SOLVENT              Tol
NS                      16
DS                       2
SWH               4237.288 Hz
FIDRES            0.129312 Hz
AQ                3.8666739 sec
RG                     256
DW                 118.000 usec
DE                   6.50 usec
TE                   293.0 K
HL1                      1 dB
D1              1.00000000 sec
P1                   11.50 usec
SFO1          300.1319000 MHz
NUCLEUS                  1H

F2 - Processing parameters
SI                   16384
SF            300.1300041 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```




WM2-200 toluene
No parameters
diastereomer
mixture
Ph
CF3
OMe
Me
HN
O
ppm
8.5
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
21.544
3.998
4.000
11.994
58.465