Diastereoselective Silylene Transfer Reactions to Chiral Enantiopure Alkenes: Effects of Ligand Size and Substrate Bias

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Supporting Information

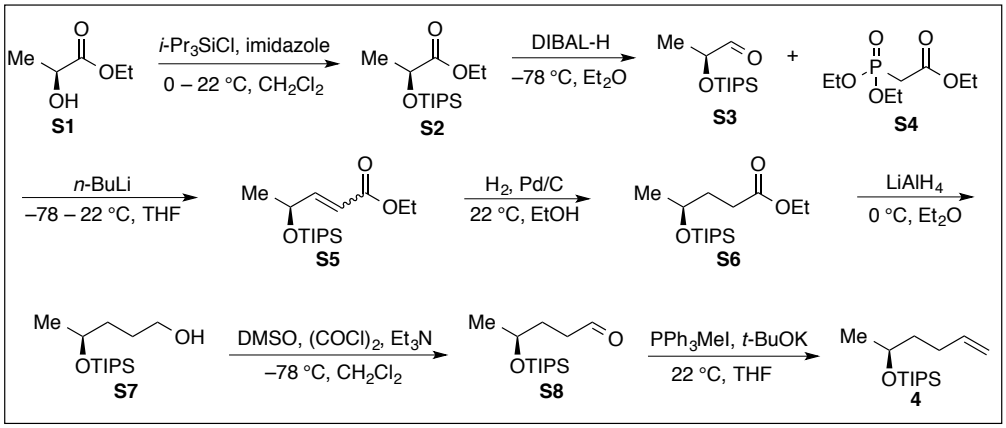
Contents

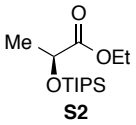
I. General Procedures.

^{1}H NMR, and ^{13}C NMR spectra were recorded at ambient temperature using Bruker AV–400 (400 and 100 MHz, respectively), AV–500 (500 and 125 MHz, respectively) or AVIII–600 (600 and 150 MHz, respectively) spectrometers, as indicated. ^{29}Si NMR spectra were recorded at ambient temperature using Bruker AVIII–400 (79 MHz) or AV–500 (99 MHz) spectrometers, as indicated. The data are reported as follows: chemical shift in ppm from internal tetramethylsilane or referenced to residual solvent (^{1}H NMR: C_6D_6 δ 7.16; $CDCl_3$ δ 7.26. ^{13}C NMR: C_6D_6 δ 128.1; $CDCl_3$ δ 77.2. ^{29}Si NMR: referenced to external tetramethylsilane C_6D_6 δ 0; $CDCl_3$ δ 0.) on the δ scale, multiplicity (appar = apparent, br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. Due to difficulties with purification for certain products, only distinctive peaks are listed in tabulated ^{1}H NMR and ^{13}C NMR spectroscopic data as indicated. ^{1}H NMR yields were determined relative to a known concentration of internal standard, mesitylene, using a single scan. Diastereomer ratios were determined using a combination of ^{1}H (single scan) and ^{13}C NMR spectroscopy.[1] Infrared (IR) spectra were obtained using a Thermo Nicolet *AVATAR* 360 FT-IR 5000 spectrometer using attenuated total reflectance (ATR). High-resolution mass spectra (HRMS) were acquired on an Agilent 6224 Accurate-Mass time-of-flight spectrometer and were obtained by peak matching. Microanalyses were performed by Atlantic Microlab Inc., Norcross, GA. Optical rotations were obtained using a digital polarimeter. Analytical thin layer chromatography was performed on Silicycle silica gel 60 Å F_{254} plates. Liquid chromatography was performed using forced flow (flash chromatography) of the indicated solvent system on Silicycle silica gel (SiO_2) 60 (230-400 mesh). Tetrahydrofuran, methylene chloride, 1,2-dichloroethane, chloroform, toluene, diethyl ether and triethylamine were dried by

filtration through alumina according to the method of Grubbs.[2] C_6D_6 and trifluorotoluene were dried over 3 Å molecular sieves for 48 h and degassed prior to use. Dimethyl sulfoxide was distilled over CaH_2. Ethanol was degassed prior to use. All reactions were run under an atmosphere of nitrogen in glassware that was flame-dried under a stream of nitrogen unless otherwise stated. Silacyclopropanes were stored and manipulated in a Vacuum Atmospheres nitrogen-atmosphere drybox. Cyclohexene silacyclopropane **2** was synthesized by known methods.[3] Methyltriphenylphosphonium iodide was prepared by known methods.[4]

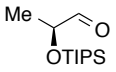
II. Preparation of Starting Materials





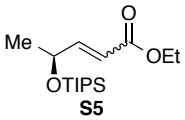
(*S*)-Ethyl 2-(Triisopropylsiloxy)propanoate S2. To a cooled (0 °C) solution of ethyl L-lactate **S1** (8.6 mL, 75 mmol) in CH_2Cl_2 (190 mL) were added triisopropylsilyl chloride (17.5 mL, 82.0 mmol) and imidazole (7.70 g, 113 mmol). The reaction mixture was allowed to warm to room temperature (22 °C). After 16 h, hexanes (50 mL) and H_2O (50 mL) were added, the layers were separated, and the aqueous layer was extracted with hexanes (3 x 20 mL). The combined organic layers were washed with H_2O (40 mL) and brine (60 mL), dried over Na_2SO_4, and concentrated *in vacuo* to provide protected lactate **S2** as a colorless oil and was used without further purification (20.4 g, 100%). The spectroscopic data are consistent with the data reported:[5] $[a]^{22}_D$ –16.99 (*c* 0.90, $CHCl_3$); ^1H

NMR (600 MHz, $CDCl_3$) δ 4.40 (q, *J* = 6.7, 1H), 4.22–4.14 (m, 2H), 1.42 (d, *J* = 6.7, 3H), 1.28 (t, *J* = 7.1, 3H), 1.07–1.05 (m, 21H); HRMS (TOF MS ES+) *m* / z calcd for $C_{14}H_{31}O_3Si$ $(M+H)^+$ 275.2037, found 275.2038.



S3

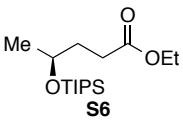
Aldehyde S3. To a cooled (–78 °C) solution of ester **S2** (2.1 g, 7.7 mmol) in Et_2O (75 mL) was added DIBAL-H (11 mL, 1.0 M in hexanes, 11 mmol) dropwise over 5 min. After 1 h, 2.0 M aqueous Rochelle Salt (30 mL) and H_2O (30 mL) were added. The aqueous layer was extracted with Et_2O (3 x 40 mL) and the combined organic layers were washed with brine (50 mL), dried over Mg_2SO_4 and concentrated *in vacuo* to provide aldehyde **S3** as a pale yellow oil that was used without further purification (1.7 g, 97%). The spectral data are consistent with the data reported:[6] $[a]^{20}_D$ –8.10 (*c* 1.10, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.66 (d, *J* = 1.7, 1H), 4.18 (qd, 6.8, 1.6, 1H), 1.31 (d, *J* = 6.8, 3H), 1.08–1.07 (m, 21H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 204.8, 74.0, 19.1, 18.1, 12.3; HRMS (TOF MS ES+) *m* / z calcd for $C_{12}H_{27}O_2Si$ $(M+H)^+$ 231.1775, found 231.1777.



S5

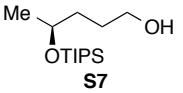
Enones S5. To a cooled (–78 °C) of triethyl phosphonoacetate **S4** (3.6 mL, 24 mmol) in THF (75 mL) was added *n*-BuLi (9.0 mL, 2.5 M in hexanes, 23 mmol) dropwise over 3 min. After 30 min, aldehyde **S3** (3.5 g, 15 mmol) was added as a solution in THF (30 mL), by cannula. The reaction mixture was warmed to room temperature (22 °C). After 16 h, the reaction mixture was concentrated *in vacuo* and CH_2Cl_2 (30 mL) was added to the residue. The organic layer was washed with 1 M HCl (10 mL), H_2O (15 mL), and brine (20 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash chromatography (3:97 EtOAc:hexanes) provided enones **S5** as a clear oil (2.7 g, 59%, 74:26 d.r.): ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.95 (dd, *J* = 15.5, 4.4, 1H), 6.26 (dd, *J* = 11.7, 7.6,

0.26H), 6.02 (dd, *J* = 15.5, 1.6, 1H), 5.64 (dd, *J* = 11.7, 1.4, 0.26H), 5.55–5.48 (m, 0.26H), 4.60–4.54 (m, 1H), 4.25–4.14 (m, 2.6H), 1.31–1.26 (m, 7.8H), 1.08–1.04 (m, 27H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 166.1, 155.4, 152.4, 119.1, 116.8, 68.0, 65.8, 60.5, 60.3, 24.1, 19.6, 18.23, 18.21, 18.14, 18.11, 18.07, 14.4, 12.5, 12.3; IR (ATR) 1720, 1090, 825 cm^{-1}; HRMS (TOF MS ES+) *m* / z calcd for $C_{16}H_{33}O_3Si$ $(M+H)^+$ 301.2193, found 301.2200. Anal. Calcd for $C_{16}H_{32}O_3Si$: C, 63.95; H, 10.73. Found: C, 64.03; H, 10.80.



S6

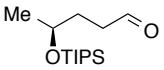
Ester S6. To a solution of enones **S5** (1.5 g, 5.0 mmol) in EtOAc (25 mL) was added Pd/C (0.53 g, 0.5 mmol Pd). The solution was sparged with N_2 and then stirred vigorously under an H_2 atmosphere (balloon). After 3 days, the reaction mixture was filtered through a short pad of Celite and concentrated *in vacuo*. Purification by flash chromatography (1:99 EtOAc:hexanes) provided ester **S6** as a colorless oil (1.26 g, 83%): $[a]^{17}_D$ +8.03 (*c* 2.00, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.12 (q, *J* = 7.1, 2H), 4.07–3.98 (m, 1H), 2.46–2.33 (m, 2H), 1.85–1.72 (m, 2H), 1.25 (t, *J* = 7.2, 3H), 1.17 (d, *J* = 6.1, 3H), 1.06 (s, 21H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 67.6, 60.6, 34.7, 30.1, 23.5, 18.33, 18.30, 14.4, 12.7; IR (ATR) 2943, 1464, 1091, 848 cm^{-1}; HRMS (TOF MS ES+) *m* / z calcd for $C_{16}H_{35}O_3Si$ $(M+H)^+$ 303.2350, found 303.2364. Anal. Calcd for $C_{16}H_{34}O_3Si$: C, 63.52; H, 11.33. Found: C, 63.79; H, 11.31.



S7

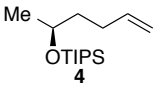
Alcohol S7. To a cooled (0 °C) solution of $LiAlH_4$ (0.31 g, 7.9 mmol) in Et_2O (24 mL) was added ester **S6** (1.2 g, 4.0 mmol) as a solution in Et_2O (40 mL) by cannula. After 1h, H_2O (20 mL) was added followed by 10% aqueous NaOH (100 mL) and H_2O (60 mL). The layers were separated and the aqueous layer was extracted with diethyl ether (5 x 15 mL). The combined organic layers were washed with brine (30 mL), dried over $MgSO_4$, and concentrated *in vacuo*. Purification by flash chromatography provided alcohol **S7**

as a colorless oil (0.95 g, 91%): $[a]^{20}_D$ +6.44 (*c* 1.10, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ 4.08–4.02 (m, 1H), 3.69–3.59 (m, 2H), 2.05 (br s, 1H), 1.74–1.55 (m, 4H), 1.20 (d, *J* = 6.2, 3H), 1.07 (s, 21H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 68.4, 63.4, 36.4, 28.3, 23.1, 18.32, 18.28, 12.6; IR (ATR) 3328, 2942, 1463 cm^{-1}; HRMS (TOF MS ES+) *m* / z calcd for $C_{14}H_{33}O_2Si$ $(M+H)^+$ 261.2244, found 261.2248. Anal. Calcd for $C_{14}H_{32}O_2Si$: C, 64.55; H, 12.38. Found: C, 64.48; H, 12.56.



S8

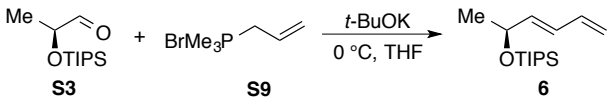
Aldehyde S8. To a cooled (–78 °C) solution of DMSO (0.11 mL, 1.5 mmol) in CH_2Cl_2 (10 mL) was added oxalyl chloride (0.10 mL, 1.2 mmol) dropwise over 2 min. After 30 min, alcohol **S7** (0.20 g, 0.76 mmol) was added as a solution in CH_2Cl_2 (1.5 mL) by cannula. After 30 min, Et_3N (0.55 mL, 4.0 mmol) was added. After 2 h, H_2O (5 mL) was added, the layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (3 x 5 mL). The combined organic layers were washed with H_2O (3 x 5 mL) and brine (10 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash chromatography (5:95 EtOAc:hexnes) provided aldehyde **S8** as a yellow oil (0.18 g, 90%). The aldehyde was used immediately in subsequent steps: $[a]^{18}_D$ +12.85 (*c* 1.25, $CHCl_3$); ^1H NMR (600 MHz, $CDCl_3$) δ 9.80 (t, *J* = 1.6, 1H), 4.09–4.04 (m, 1H), 2.59–2.49 (m, 2H), 1.88–1.82 (m, 1H), 1.79–1.73 (m, 1H), 1.18 (d, *J* = 6.1, 3H), 1.06 (s, 21H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 202.9, 67.5, 39.7, 31.8, 23.5, 18.33, 18.29, 12.7; HRMS (TOF MS ES+) *m* / z calcd for $C_{14}H_{30}NaO_2Si$ $(M+Na)^+$ 281.1907, found 281.1925.



4

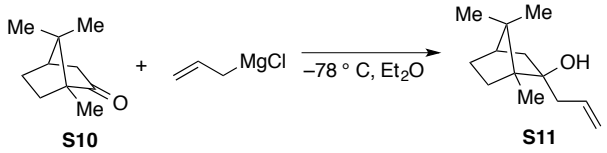
Alkene 4. To a solution methyltriphenylphosphonium iodide (0.55 g, 1.4 mmol) in THF (3.6 mL) was added *t*-BuOK (0.19 g, 1.7 mmol). After 2 h, aldehyde **S8** (0.28 g, 1.1 mmol) was added as a solution in THF (1.8 mL). After 20 min hexanes (10 mL)

and saturated aqueous NH_4Cl (10 mL) were added, the layers were separated, and the aqueous layer was extracted with hexanes (3 x 5 mL). The combined organic layers were washed with H_2O (10 mL) and brine (15 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash chromatography (hexanes) provided alkene **4** as a colorless oil (0.22 g, 79%): $[a]^{20}_D$ +5.75 (*c* 0.94, $CHCl_3$); ^{1}H NMR (600 MHz, $CDCl_3$) δ 5.86–5.79 (m, 1H), 5.01 (ddd, *J* = 17.1, 3.4, 1.6, 1H), 4.95–4.92 (m, 1H), 3.98–3.93 (m, 1H), 2.13–2.08 (m, 2H), 1.64–1.58 (m, 1H), 1.55–1.49 (m, 1H), 1.17 (d, *J* = 6.1, 3H), 1.06 (s, 21H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 139.2, 114.4, 68.2, 39.2, 29.8, 23.6, 18.4, 18.3, 12.7; IR (ATR) 2866, 1642 cm^{-1}; HRMS (TOF MS ES+) *m* / z calcd for $C_{15}H_{32}NaOSi$ $(M+Na)^+$ 279.2115, found 279.2109. Anal. Calcd for $C_{15}H_{32}OSi$: C, 70.24; H, 12.57. Found: C, 70.50; H, 12.73.



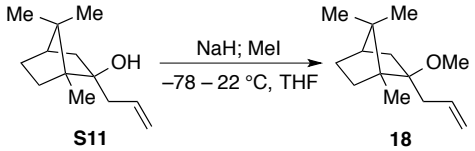
Diene 6. To a solution of allyltrimethylphosphonium bromide **S9** (0.087 g, 0.44 mmol) in THF (1 mL) was added *t*-BuOK (0.052 g, 0.46 mmol). After 30 min, aldehyde **S3** (0.106 g, 0.460 mmol) was added as a solution in THF (0.5 mL). After 16 h Et_2O (3 mL) and H_2O (5 mL) were added, the layers were separated, and the aqueous layer was extracted with Et_2O (5 x 3 mL). The combined organic layers were washed with brine (10 mL), dried over $MgSO_4$, and concentrated *in vacuo*. Purification by flash chromatography (hexanes) provided diene **6** as a colorless oil (0.041 g, 35%): $[a]^{19}_D$ +9.71 (*c* 0.58, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.32 (dt, *J* = 16.9, 10.3, 1H), 6.17 (dd, *J* = 15.2, 10.5, 1H), 5.73 (dd, *J* = 15.2, 5.9, 1H), 5.16 (d, *J* = 14.0, 1H), 5.04 (d, *J* = 10.1, 1H), 4.46–4.41 (m, 1H), 1.26 (d, *J* = 6.3, 3H), 1.06 (s, 21H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.3, 136.9, 128.9, 116.6, 69.0, 25.0, 18.2, 12.5; IR (ATR) 2943,

1463 cm^{-1}; HRMS (TOF MS ES+) *m* / z calcd for $C_{15}H_{31}OSi$ $(M+H)^+$ 255.2139, found 255.2141.[6]



S10 **S11**

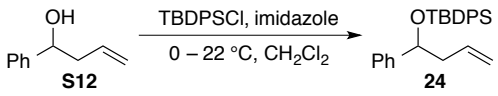
Alcohol S11. To a cooled (–78 °C) solution of (*R*)-camphor **S10** (3.1 g, 20 mmol) in Et_2O (100 mL) was added allylmagnesium chloride (25 mL, 1.0 M in Et_2O, 25 mmol) dropwise over 5 min. After 30 min, MeOH (5 mL) was added and the reaction mixture was warmed to 22 °C. H_2O (30 mL) was added, the layers were separated, and the aqueous layer was extracted with Et_2O (3 x 20 mL). The combined organic layers were washed with brine (25 mL), dried over $MgSO_4$, and concentrated *in vacuo*. Purification by flash chromatography (5:95 EtOAc:hexanes) provided alcohol **S11** as a yellow oil (3.8 g, 97%). The spectroscopic data are consistent with the data reported:[7] $[a]^{20}_D$ +8.02 (*c* 4.00, toluene); ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.98–5.90 (m, 1H), 5.18–5.14 (m, 2H), 2.34–2.25 (m, 2H), 1.97 (dt, *J* = 13.1, 3.7, 1H), 1.76–1.69 (m, 3H), 1.44–1.42 (m, 2H), 1.10 (s, 3H), 1.06–1.02 (m, 1H), 0.86 (s, 3H), 0.85 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 135.2, 119.1, 79.9, 52.4, 49.6, 46.2, 45.2, 44.7, 30.7, 27.2, 21.6, 21.1, 11.1.



S11 **18**

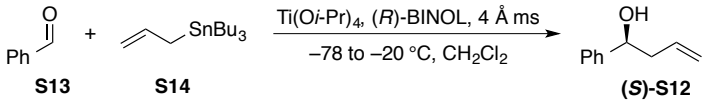
Homoallylic Ether 18. To a cooled (0 °C) solution of NaH (0.31 g, 60% in mineral oil, 7.9 mmol) in THF (10 mL) was added alcohol **S11** (0.75 g, 3.9 mmol) as a solution in THF (5 mL) by cannula. The reaction mixture was allowed to warm to room temperature (22 °C) over 3 h,

and iodomethane (0.60 mL, 9.7 mmol) was added. After 16 h, saturated aqueous $NaHCO_3$ (10 mL) and H_2O (10mL) were added and the aqueous layer was extracted with Et_2O (3 x 15 mL). The combined organic layers were washed with brine (20 mL), dried over $MgSO_4$, and concentrated *in vacuo*. Purification by flash chromatography (hexanes) provided allyl ether **18** as a colorless oil (0.70 g, 87%): $[a]^{20}_D$ +9.91 (*c* 0.40, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.94 (dddd, *J* = 17.2, 10.1, 8.9, 5.4, 1H), 5.08–5.00 (m, 2H), 3.13 (s, 3H), 2.71–2.66 (m, 1H), 2.20 (dt, *J* = 12.9, 3.7, 1H), 2.13 (dd, *J* = 15.7, 8.7, 1H), 1.76–1.68 (m, 1H), 1.66–1.65 (m, 1H), 1.57–1.52 (m, 1H), 1.38 (ddd, *J* = 13.3, 11.8, 5.6, 1H), 1.03 (ddd, *J* = 12.6, 9.2, 5.7, 1H), 0.96 (s, 3H), 0.88 (s, 3H and m, 1H), 0.82 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.4, 115.8, 85.6, 53.1, 50.3, 47.8, 45.1, 41.1, 40.0, 30.8, 27.4, 21.2, 21.1, 12.3; IR (ATR) 3074, 2963, 1639 cm^{-1}; Anal. Calcd for $C_{14}H_{24}O$: C, 80.71; H, 11.61. Found: C 80.95; H, 11.75.

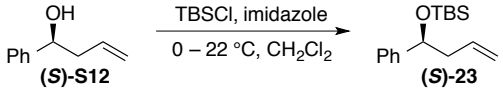


Silyl Ether 24. To a cooled (0 °C) solution of alcohol **S12** (0.51 g, 3.4 mmol) in CH_2Cl_2 (7 mL) were added imidazole (0.20 g, 4.1 mol) and *tert*-butyl(chloro)diphenylsilane (0.85 mL, 3.3 mmol). After 24 h, an additional portion of *tert*-butyl(chloro)diphenylsilane (0.5 mL, 1.9 mmol) was added. After 24 h, hexanes (15 mL) and H_2O (15 mL) were added, the layers were separated, and the aqueous layer was extracted with hexanes (3 x 10 mL). The combined organic layers were washed with H_2O (15 mL) and brine (20 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash chromatography (hexanes) provided silyl ether **24** as a colorless oil (0.467 g, 37%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71–7.69 (m, 2H), 7.46–7.31 (m, 6H), 7.26–7.18 (m, 7H), 5.58–5.48 (m, 1H), 4.87–4.78 (m, 2H), 4.72–4.69 (m, 1H), 2.48–2.35 (m, 2H), 1.04 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.2, 136.13, 136.10, 134.5, 134.4,

133.8, 129.8, 129.6, 128.0, 127.7, 127.5, 127.1, 126.5, 117.3, 75.7, 44.9, 27.2, 19.5; IR (ATR) 2931, 1454 cm^{-1}; HRMS (TOF MS ES+) *m* / z calcd for $C_{26}H_{30}NaOSi$ $(M+Na)^+$ 409.1958, found 409.1971. Anal. Calcd for $C_{26}H_{30}OSi$: C, 80.78; H, 7.82. Found: C, 80.74; H, 7.66.

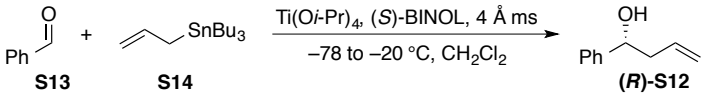


(*S*)-Alcohol S12. To a solution of (*R*)-BINOL (0.29 g, 1.0 mmol) in CH_2Cl_2 (20 mL) with powdered molecular sieves (4 Å, 4.3 g) was added Ti(O*i*-Pr)$_4$ (0.30 mL, 1.0 mmol) and the reaction mixture was heated to reflux. After 1 h, the reaction mixture was cooled to 22 °C and benzaldehyde **S13** (1.0 mL, 10 mmol) was added. After 15 min, the reaction mixture was cooled to –78 °C and allyltributyltin **S14** (4.0 mL, 13 mmol) was added. After 10 min, the reaction mixture was brought to –20 °C. After 3 d, the reaction mixture was warmed to 22 °C, diluted with CH_2Cl_2 (20 mL), and filtered. The organic layer was washed with saturated aqueous $NaHCO_3$ (20 mL) and brine (20 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash chromatography (10:90 EtOAc:hexanes) provided alcohol **(*S*)-S12** as a yellow oil (0.52 g, 35%). The spectroscopic data are consistent with the data reported:[8] $[a]^{20}_D$ +50.48 (*c* 0.53, $CHCl_3$); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38–7.34 (m, 4H), 7.29–7.26 (m, 1H), 5.85–5.78 (m, 1H), 5.19–5.14 (m, 2H), 4.75 (dd, *J* = 7.9, 5.0, 1H), 2.57–2.47 (m, 2H).

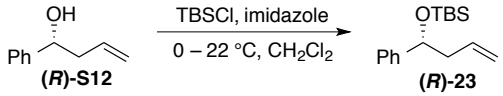


(*S*)-Homoallylic Ether 23. To a cooled (0 °C) solution of alcohol **(*S*)-S12** (0.50 g, 3.4 mmol) in CH_2Cl_2 (7 mL) were added imidazole (0.28 g, 4.1 mmol) and *tert*-butyl(chloro)dimethylsilane (0.51 g, 3.4 mmol) and the reaction mixture was allowed to warm

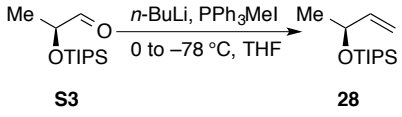
to room temperature (22 °C). After 16 h, hexanes (5 mL) and H_2O (5 mL) were added, the layers were separated, and the aqueous layer was extracted with hexanes (3 x 5 mL). The combined organic layers were washed with H_2O (5 mL) and brine (10 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash chromatography (hexanes) provided protected alcohol **(*S*)-23** as a colorless oil (0.50 g, 57%): $[a]^{18}_D$ +47.22 (*c* 1.00, $CHCl_3$); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32–7.29 (m, 4H), 7.24–7.21 (m, 1H), 5.81–5.74 (m, 1H), 5.03–4.99 (m, 2H), 4.69–4.67 (m, 1H), 2.48–2.36 (m, 2H), 0.88 (s, 9H), 0.02 (s, 3H), –0.13 (s, 3H).



(*R*)-Alcohol S12. To a solution of (*S*)-BINOL (0.29 g, 1.0 mmol) in CH_2Cl_2 (20 mL) with powdered molecular sieves (4 Å, 4.3 g) was added Ti(O*i*-Pr)$_4$ (0.30 mL, 1.0 mmol) and the reaction mixture was heated to reflux. After 1 h, the reaction mixture was cooled to 22 °C and benzaldehyde **S13** (1.0 mL, 10 mmol) was added. After 15 min, the reaction mixture was cooled to –78 °C and allyltributyltin **S14** (4.0 mL, 13 mmol) was added. After 10 min, the reaction mixture was brought to –20 °C. After 3 d, the reaction mixture was warmed to 22 °C, diluted with CH_2Cl_2 (20 mL), and filtered. The organic layer was washed with saturated aqueous $NaHCO_3$ (20 mL) and brine (20 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash chromatography (10:90 EtOAc:hexanes) provided alcohol **(*R*)-S12** as a yellow oil (0.68 g, 47%): $[a]^{20}_D$ –44.36 (*c* 1.55, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.34 (m, 4H), 7.30–7.27 (m, 1H), 5.87–5.77 (m, 1H), 5.20–5.13 (m, 2H), 4.77–4.72 (m, 1H), 2.58–2.46 (m, 2H).



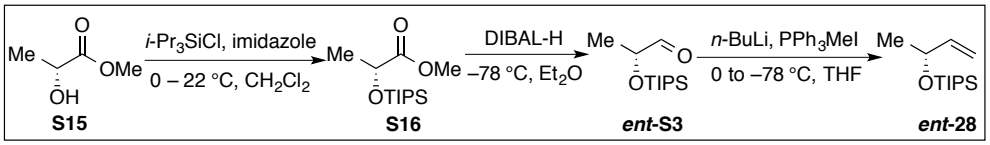
(*R*)-Homoallylic Ether 23 To a cooled (0 °C) solution of alcohol **(*R*)-S12** (0.68 g, 4.6 mmol) in CH_2Cl_2 (9 mL) were added imidazole (0.38 g, 5.5 mmol) and *tert*-butyl(chloro)dimethylsilane (0.56 g, 3.7 mmol). The reaction mixture was allowed to warm to room temperature (22 °C). After 16 h, hexanes (5 mL) and H_2O (5 mL) were added, the layers were separated, and the aqueous layer was extracted with hexanes (3 x 5 mL). The combined organic layers were washed with H_2O (5 mL) and brine (10 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash chromatography (hexanes) provided protected alcohol **(*R*)-23** as a colorless oil (0.72 g, 60%): $[a]^{18}_D$ –48.89 (*c* 1.02, $CHCl_3$); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30–7.29 (m, 4H), 7.23–7.21 (m, 1H), 5.81–5.74 (m, 1H), 5.02–4.99 (m, 2H), 4.67 (dd, *J* = 7.3, 5.2, 1H), 2.48–2.43 (m, 1H), 2.40–2.36 (m, 1H), 0.88 (s, 9H), 0.02 (s, 3H), –0.13 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 145.3, 135.5, 128.2, 127.1, 126.1, 117.0, 75.2, 45.7, 26.0, 18.4, –4.5, –4.7; IR (ATR) 1641, 1085, 832 cm^{-1}; HRMS (TOF MS ES+) *m* / z calcd for $C_{16}H_{27}OSi$ $(M+H)^+$ 263.1826, found 236.1814. Anal. Calcd for $C_{16}H_{26}OSi$: C, 73.22; H, 9.98. Found: C, 73.38; H, 9.97.

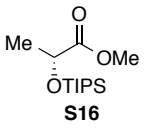


S3 **28**

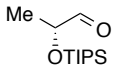
Alkene 28. To a cooled (–15 °C) solution of methyltriphentylphosphonium bromide (5.1 g, 14 mmol) in THF (150 mL) was added *n*-BuLi (6.0 mL, 2.5 M in hexanes, 15 mmol) dropwise over 5 min. After 15 min, the reaction mixture was cooled to –78 °C and aldehyde **S3** (1.7 g, 7.4 mmol) was added as a solution in THF (50 mL) by cannula. After 15 min, the reaction

mixture was brought to room temperature (22 °C). After 1 h, saturated aqueous NH_4Cl (75 mL) was added and the aqueous layer was extracted with Et_2O (3 x 45 mL). The combined organic layers were washed with brine (50 mL), dried over $MgSO_4$, and concentrated *in vacuo*. Purification by flash chromatography (3:97 EtOAc:hexanes) provided alkene **28** as a colorless oil (0.83 g, 48%): $[a]^{20}_D$ +14.12 (*c* 0.46, $CHCl_3$); ^{1}H NMR (600 MHz, $CDCl_3$) δ 5.87 (ddd, *J* = 17.0, 10.4, 5.5, 1H), 5.17 (dt, *J* = 17.2, 1.4, 1H), 4.98 (dt, *J* = 10.4, 1.3, 1H), 4.40–4.36 (m, 1H), 1.24 (d, *J* = 6.4, 3H), 1.07 (s, 10H), 1.06 (s, 11H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 143.5, 112.5, 69.8, 24.9, 18.3, 12.5; IR (ATR) 2943, 1464, 1091, 848 cm^{-1}; HRMS (TOF MS ES+) *m* / z calcd for $C_{13}H_{29}OSi$ $(M+H)^+$ 229.1988, found 229.1961. Anal. Calcd for $C_{13}H_{28}OSi$: C, 80.71; H, 11.61. Found: C, 80.95; H, 11.75.



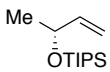


(*R*)-Methyl 2-(Triisopropylsiloxy)propanoate S16. To a cooled (0 °C) solution of methyl-D-lactate **S15** (0.50 mL, 5.2 mmol) in CH_2Cl_2 (13 mL) was added imidazole (0.54 g, 7.9 mmol). Triisopropylchlorosilane (1.3 mL, 6.1 mmol) was added dropwise over 5 min. After 16 h, hexanes (15 mL) was added, and the reaction mixture was washed with H_2O (3 x 15 mL) and brine (20 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Ester **S16** was obtained as a colorless oil without any further purification (1.2 g, 86%). The spectroscopic data are consistent with the data reported:[5a] ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.44 (q, *J* = 6.7, 1H), 3.72 (s, 3H), 1.43 (d, *J* = 6.7, 3H), 1.07–1.05 (m, 21H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.9, 68.7, 51.9, 22.0, 18.0, 12.3.



***ent*-S3**

Aldehyde *ent*-S3. To a cooled (–78 °C) solution of ester **S16** (1.2 g, 4.5 mmol) in Et_2O (45 mL) was added DIBAL-H (5.4 mL, 1.0 M in hexanes, 5.4 mmol) dropwise over 5 min. After 1 h, 2.0 M aqueous Rochelle Salt (10 mL) and H_2O (30 mL) were added and the aqueous layer was extracted with Et_2O (3 x 15 mL). The combined organic layers were washed with brine (20 mL), dried over $MgSO_4$, and concentrated *in vacuo*. Purification by flash chromatography (3:97 EtOAc:hexanes) provided aldehyde ***ent*-S3** as a colorless oil (0.55 g, 53%). The spectroscopic data are consistent with the data reported for the enantiomeric compound:[9] ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.66 (d, *J* = 1.6, 1H), 4.18 (qd, *J* = 6.8, 1.6, 1H), 1.31 (d, *J* = 6.8, 3H), 1.08–1.06 (m, 21H); HRMS (TOF MS ES+) *m* / z calcd for $C_{12}H_{27}O_2Si$ $(M+H)^+$ 231.1775, found 231.1781.



***ent*-28**

Alkene *ent*-28. To a solution of methyltriphenylphosphonium iodide (0.49 g, 1.3 mmol) in THF (3 mL) was added *t*-BuOK (0.16 g, 1.4 mmol). After 1 h, aldehyde ***ent*-S3** (0.29 g, 1.3 mmol) was added as a solution in THF (1.4 mL). After 10 min, hexanes (10 mL) and saturated aqueous NH_4Cl (10 mL) were added, the layers were separated, and the aqueous layer was extracted with hexanes (3 x 15 mL). The combined organic layers were washed with H_2O (10 mL) and brine (15 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash chromatography (hexanes) provided alkene ***ent*-28** as a colorless oil (0.212 g, 74%): $[a]^{20}_D$ –14.37 (*c* 0.53, $CHCl_3$); ^{1}H NMR (600 MHz, $CDCl_3$) 5.87 (ddd, *J* = 17.1, 10.4, 5.5, 1H), 5.17 (dt, *J* = 17.2, 1.5, 1H), 4.98 (dt, *J* = 10.4, 1.4, 1H), 4.40–4.36 (m, 1H), 1.24 (d, *J* = 6.3, 3H), 1.07 (s, 10H), 1.06 (s, 11H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.5, 112.5, 69.8, 24.9, 18.3, 12.5; HRMS (TOF MS ES+) *m* / z calcd for $C_{13}H_{29}OSi$ $(M+H)^+$ 229.1982, found 229.1972.

III. **Reaction Condition and Ligand Screening**

Figure S1. Complete List of Ligands Used

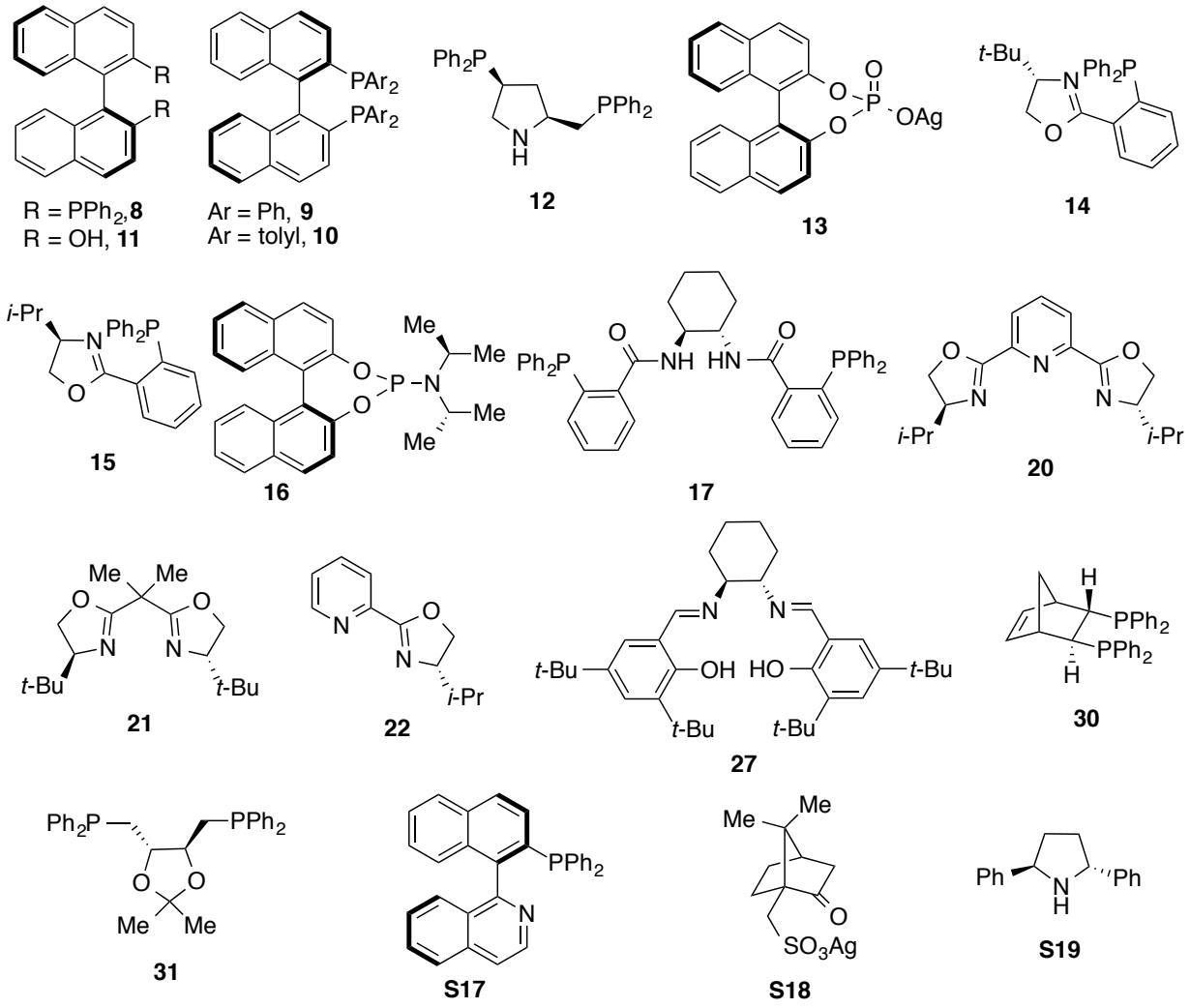
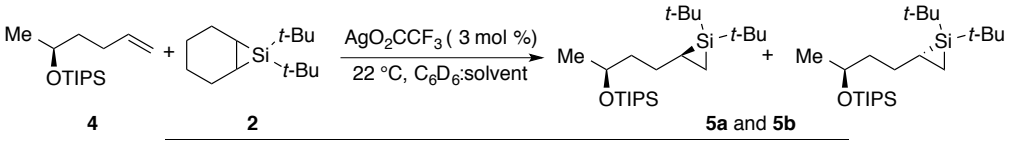


Table S1. Reaction Condition Screening for Alkene **4**

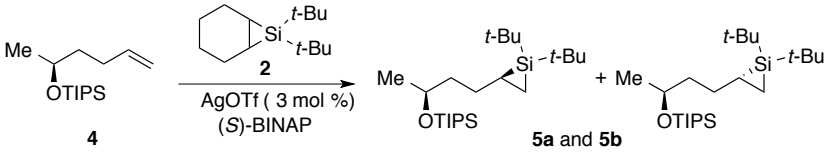


4 **2** **5a** and **5b**

Entry	Solvent	Conversion[a]	d.r.[b]
1	Trifluorotoluene	95	42:58
2	1,2-Dichloroethane	100	48:52
3	Chloroform	50	47:53
4	Tetrahydrofuran	0	-
5	Diethyl Ether	78	50:50
6	Toluene	78	47:53

[a]Determined by ^{1}H NMR spectroscopy.
[b]Determined by ^{1}H and ^{13}C NMR spectroscopy.

Table S2. Ligand Loading



4 **5a** and **5b**

Entry	Ag:BINAP	Complex[10]	Conversion (%)[a]	d.r.[b]
1	1:2	**A**	46	52:48
2	1:1	**A:B:C** (21:63:16)[c]	68	50:50
3	2:1	**C**	67	53:47

[a]Determined by ^{1}H NMR spectroscopy.
[b]Determined by ^{1}H and ^{13}C NMR spectroscopy.
[c]Ratio of complexes in solution.

Figure S2. BINAP Binding Modes in Solution

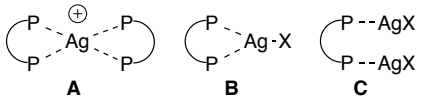
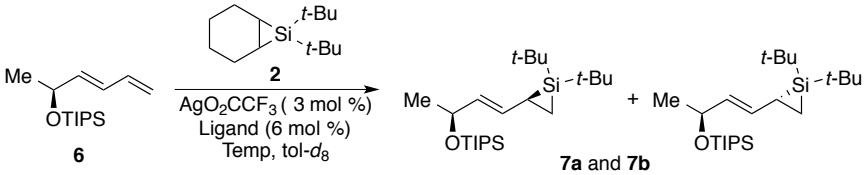


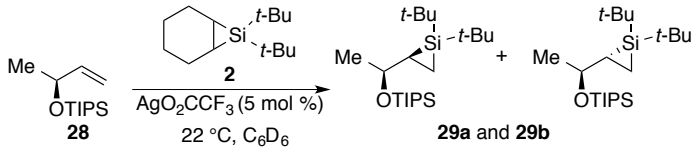
Table S3. Ligand and Temperature Screen for Alkene **6**



Entry	Ligand	Temperature (°C)	Time (h)	d.r.[a]
1	(*S*)-BINAP (**8**)	22	2	52:48
2	(*S*)-BINAP (**8**)	–20	7.5	54:46
3	(*S*)-BINAP (**8**)	–20	22	53:47
4	(*R*)-BINAP (**9**)	–20	22	51:49
5	(*R*)-BINAP (**9**)	–78	7.5	53:47

[a]Determined by ^{1}H NMR spectroscopy.

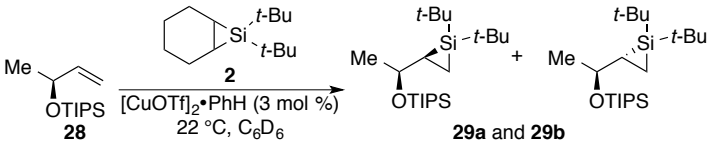
Table S4. Ligand Screening for Alkene **28** (Silver-Catalyzed)



Ligand (10 mol %)	Conversion (%)[a]	d.r.[a,b]
(*R*)-BINAP (**9**)	20	53:47
(*R*)-QUINAP (**S17**)[c]	67	55:45
(*S*,*S*)-NORPHOS (**30**)	0	-
(*S*,*S*)-DIOP (**31**)	0	-
(*R*,*R*)-pyrrolidine (**S19**)	61	54:46
(*S*,*S*)-salen (**27**)	79	54:46
(*S*,*S*)-*t*-Bu-BOX (**21**)	82	52:48

[a]Determined by ^{1}H NMR spectroscopy.
[b]Determined using ^{13}C NMR spectroscopy.
[c]Heated to 80 °C for 48 h.

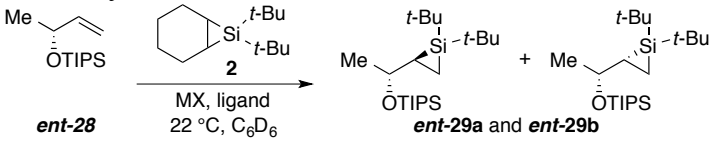
Table S5. Ligand Screening for Alkene **28** (Copper-Catalyzed)



Entry	Ligand (6 mol %)	Conversion (%)[a]	d.r.[a,b]
1	(*R*)-BINAP (**9**)	18	50:50
2	(*R*)-QUINAP (**S17**)	67	55:45
3	(*S*,*S*)-*i*-Pr-PyBOX (**20**)	0	-
4	(*S*,*S*)-*i*-Pr-PyOX **(22)**	84	60:40
5	(*S*,*S*)-*t*-Bu-BOX (**21**)	30	56:44
6	(*S*,*S*)-salen (**27**)	85	60:40
7	(*R*,*R*)-pyrrolidine (**S19**)	34	59:41

[a]Determined by ^{1}H NMR spectroscopy.
[b]Determined using ^{13}C NMR spectroscopy.

Table S6. Ligand and Catalyst Screen for ***ent*-28**



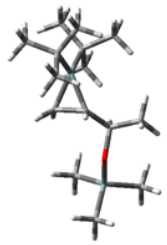
Entry	MX	Ligand	Yield (%)[a]	d.r.[a]
1	AgO_2CCF_3	None	77	54:46
2	AgO_2CCF_3	(*S*)-BINAP (**8)**	61	51:49
3	AgO_2CCF_3	(*R*)-QUINAP (**S17**)	53	53:47
4	$[Cu(OTf)_2]$•PhH	None	68	60:40
5	$[Cu(OTf)_2]$•PhH	(*S*,*S*)-*t*-Bu-BOX (**21**)	21	52:48
6	$[Cu(OTf)_2]$•PhH	(*R*)-QUINAP (**S17**)	4	50:50

[a]Determined by ^{1}H NMR spectroscopy.

IV. **Details of Computation**

Major isomer, 29b and 29b' (anti). A conformational search (Monte Carlo) for the tri(isopropyl)silyl ether **29b** using molecular mechanics (MMFF force field) as implemented in Spartan16[11] produced >300 conformers, mostly with similar energies (note that the enantiomeric structure was calculated, which would not affect the results). Minimizing just the lowest energy conformer with DFT (B3LYP/6-31G*) was prohibitively slow, so it was not practical to calculate several hundred conformations at the higher level. The trialkylsilyl ether moiety was changed to a trimethylsilyl group, considering that these two different types of silyloxy groups should be conformationally similar.[12] With the trimethylsilyl ether (compound **29b'**), 20 low-energy structures were identified (MMFF). Those structures were optimized using the PM3 model in Spartan16. The geometries were then optimized with Gaussian09[13] using density functional theory (DT/6-31G*) to optimize geometry (keywords: opt b3lyp/6-31g(d) geom=connectivity).

Of the 20 structures, the lowest two were nearly identical in structure and energy; the structure of the lowest energy structure (the lower of these two) is listed below (E = –1246.34122902 au). Frequency calculations were performed on the lowest energy conformer (keywords: freq b3lyp/6-31g(d) geom=connectivity), which confirmed that it had no imaginary frequencies. Three more structures were found only 0.2 kcal/mol higher, and another three were 1.6 kcal/mol higher in energy. The only difference among these structures were differences in rotation of the trimethylsilyl group.



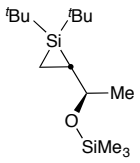


Table S7. Cartesian coordinates for Major isomer, **29b'**.

Atom	X (Å)	Y (Å)	Z (Å)
Si	-1.669300	-0.100800	-0.215300
C	-0.239400	-0.640100	-1.292900
C	0.044300	-0.745000	0.236000
C	1.250300	-0.038500	0.826700
O	2.430700	-0.764700	0.439200
Si	3.831100	-0.143800	-0.230400
C	-3.110300	-1.377700	-0.007400
C	-4.026100	-1.018000	1.182800
C	-2.519800	-2.781800	0.245700
C	-3.955000	-1.440600	-1.301500
C	-2.103100	1.785600	-0.121100
C	-2.324800	2.222500	1.344800
C	-3.382800	2.088500	-0.933100
C	-0.957200	2.629500	-0.719000
C	4.656500	1.090800	0.943000
C	3.483200	0.720200	-1.877100
C	4.943900	-1.637200	-0.498800
H	0.335000	0.132200	-1.806300
H	-0.241600	-1.573800	-1.855300
H	0.028700	-1.780600	0.583100
H	-3.469300	-0.964600	2.126200
H	-4.538800	-0.060000	1.040900
H	-4.805000	-1.785000	1.304600
H	-1.969000	-2.833400	1.191100
H	-1.843700	-3.098400	-0.556900
H	-3.333200	-3.519900	0.296600
H	-3.345700	-1.725100	-2.167000
H	-4.750700	-2.192700	-1.194800
H	-4.439700	-0.486500	-1.533700
H	-1.424100	2.078100	1.951700
H	-3.141700	1.672700	1.824500
H	-2.582000	3.291300	1.385100
H	-3.593500	3.167500	-0.905500

H	-3.278500	1.801400	-1.986100
H	-4.263800	1.577900	-0.530200
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H	-0.767400	2.379400	-1.768600
H	-1.222200	3.695800	-0.674400
H	4.918800	0.617500	1.896600
H	4.003600	1.944000	1.165000
H	5.579500	1.492600	0.505900
H	2.813400	1.581100	-1.761000
H	3.019600	0.033500	-2.594800
H	4.414000	1.092300	-2.324100
H	5.145300	-2.152900	0.447200
H	5.907700	-1.344700	-0.933200
H	4.475300	-2.359300	-1.177100
C	1.204300	0.021600	2.354500
H	2.104000	0.504200	2.750600
H	1.154400	-0.992700	2.766100
H	0.326900	0.578500	2.700900
H	1.307800	0.986000	0.430100

Minor isomer, 29a' (syn). A conformational search of the trimethylsilyl ether was performed as discussed above, which produced 15 low-energy structures. These structures were optimized using the PM3 model in Spartan16. This minimization produced eight unique structures, which were then optimized using Gaussian09 using density functional theory (DFT/6-31G*) as discussed above. Of these structures, the lowest three were essentially of the same energy and structure (<0.0003 kcal/mol). The lowest energy of these conformers is shown below (E = –1246.34012163 au). Frequency calculations, as discussed above, confirmed that it had no imaginary frequencies. Three other structures were only 0.05 kcal/mol higher in energy; they exhibited a ∠Si–C–C–O dihedral angle of –38°. By comparison, that angle was 78° in the lowest energy conformers. The remaining two structures were considerably higher energy (>3.5 kcal/mol).

The assignment of this conformer results from the energy differences between the two isomeric forms of **29a'** compared to **29b'**. All of the conformers of **29a'** are >0.7 kcal/mol higher than for the lowest energy conformers of **29b'**. Although this number is not large, it does suggest which isomer is thermodynamically preferred. The energy difference is likely to be larger in the triisopropyl silyl ether series. In isomer **29a'**, the groups on the two silicon atoms occupy similar regions of space, which is not the case for **29b'**, so making the groups larger on the silyl ether should increase that interaction and destabilize the minor isomer.

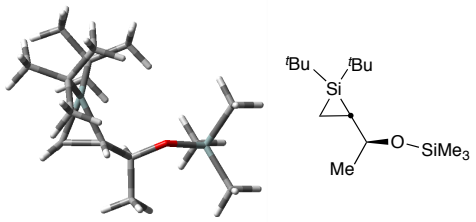

Table S8. Cartesian coordinates for Minor isomer, **29a'**.

Atom	X (Å)	Y (Å)	Z (Å)
Si	-1.69260	-0.14800	-0.35860
C	-1.24050	-1.14140	-1.88450
C	-0.20850	-1.24170	-0.71830
C	1.23940	-0.86130	-0.98490
O	1.86500	-0.54600	0.26430
Si	3.37310	0.12470	0.54780
C	1.98020	-1.99560	-1.70490
C	-3.03610	-0.93210	0.79180
C	-3.00820	-0.30460	2.20300
C	-2.77150	-2.44660	0.93450
C	-4.44170	-0.74640	0.17520
C	-1.54620	1.78050	-0.43060
C	-0.75310	2.28610	0.79650
C	-2.93810	2.45120	-0.44550
C	-0.80520	2.21270	-1.71380
C	3.23210	0.96180	2.22940
C	3.80420	1.39250	-0.78910
C	4.71930	-1.20000	0.63270
H	-0.88570	-0.59440	-2.75970
H	-1.75520	-2.05880	-2.17080
H	-0.25530	-2.20350	-0.20270

H	1.26830	0.02750	-1.63210
H	3.00160	-1.70590	-1.97770
H	1.45050	-2.26990	-2.62400
H	2.03440	-2.87950	-1.05920
H	-2.03130	-0.42870	2.68540
H	-3.24210	0.76570	2.18970
H	-3.75700	-0.79050	2.84570
H	-1.81670	-2.65230	1.43020
H	-2.76890	-2.96060	-0.03390
H	-3.56510	-2.90360	1.54320
H	-4.51170	-1.21180	-0.81480
H	-5.20010	-1.21830	0.81750
H	-4.71660	0.30790	0.06860
H	0.24000	1.82770	0.84740
H	-1.26780	2.06650	1.73880
H	-0.62470	3.37760	0.74150
H	-2.82240	3.54280	-0.51560
H	-3.54090	2.13050	-1.30360
H	-3.51190	2.24750	0.46490
H	-1.32490	1.88010	-2.61910
H	-0.74210	3.31000	-1.75100
H	0.21970	1.82960	-1.75270
H	2.91480	0.24550	2.99610
H	2.49610	1.77380	2.21090
H	4.19280	1.38610	2.54660
H	3.90220	0.93550	-1.78120
H	4.75960	1.88130	-0.56060
H	3.04130	2.17730	-0.85760
H	5.68100	-0.75800	0.92370
H	4.86750	-1.71120	-0.32510
H	4.46700	-1.96240	1.37940

V. **References and Notes**

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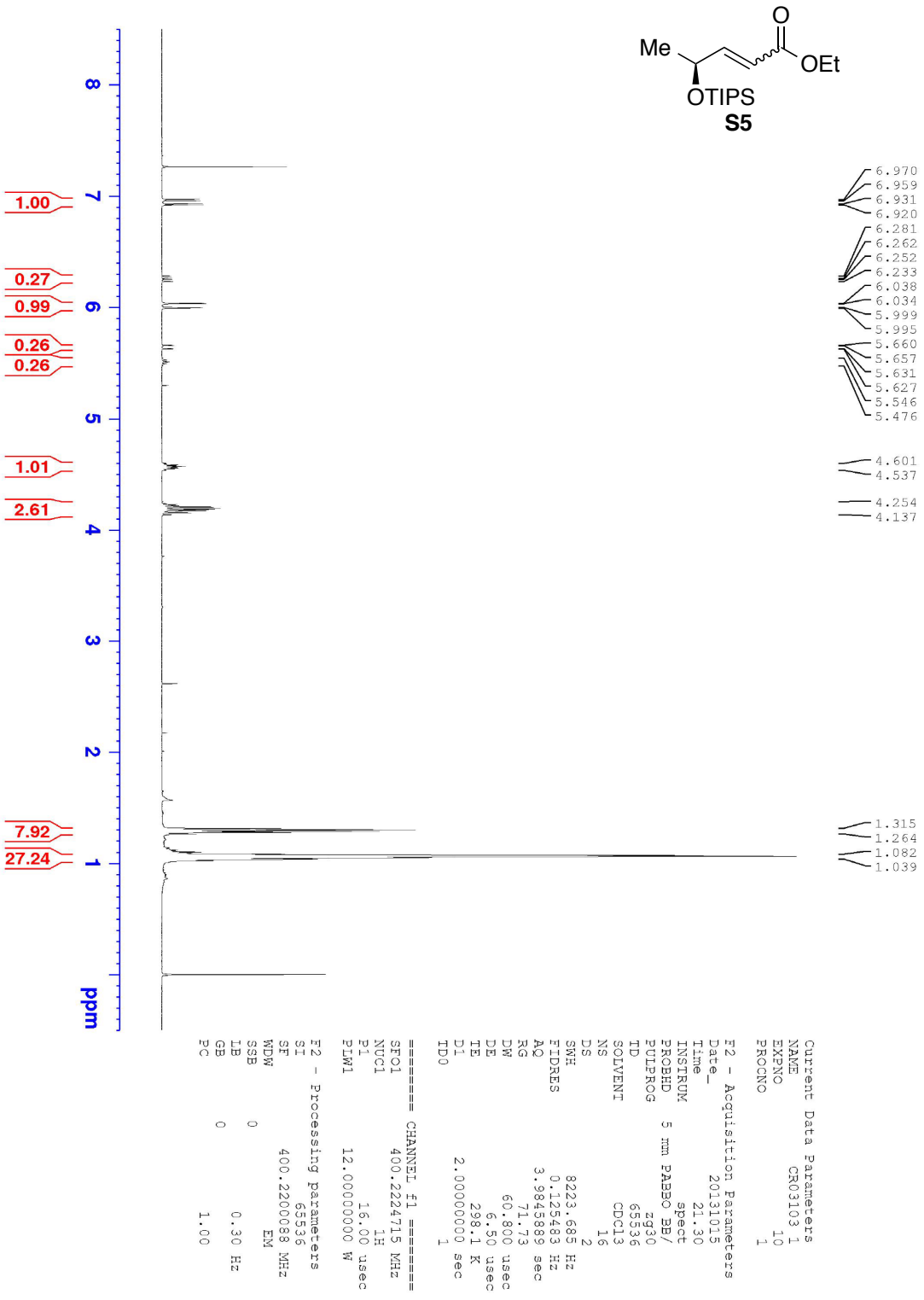
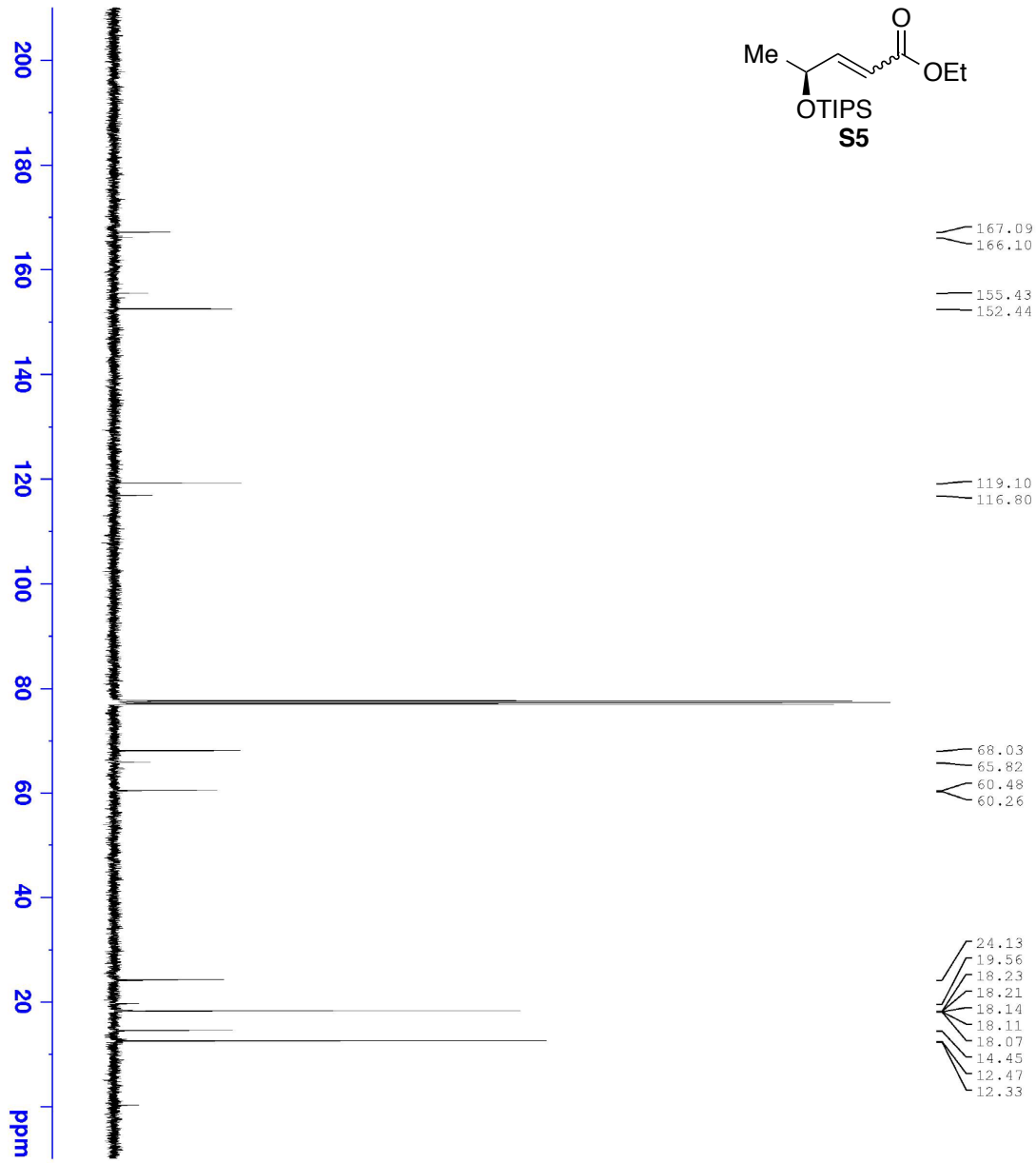
VI. **Selected Spectra**

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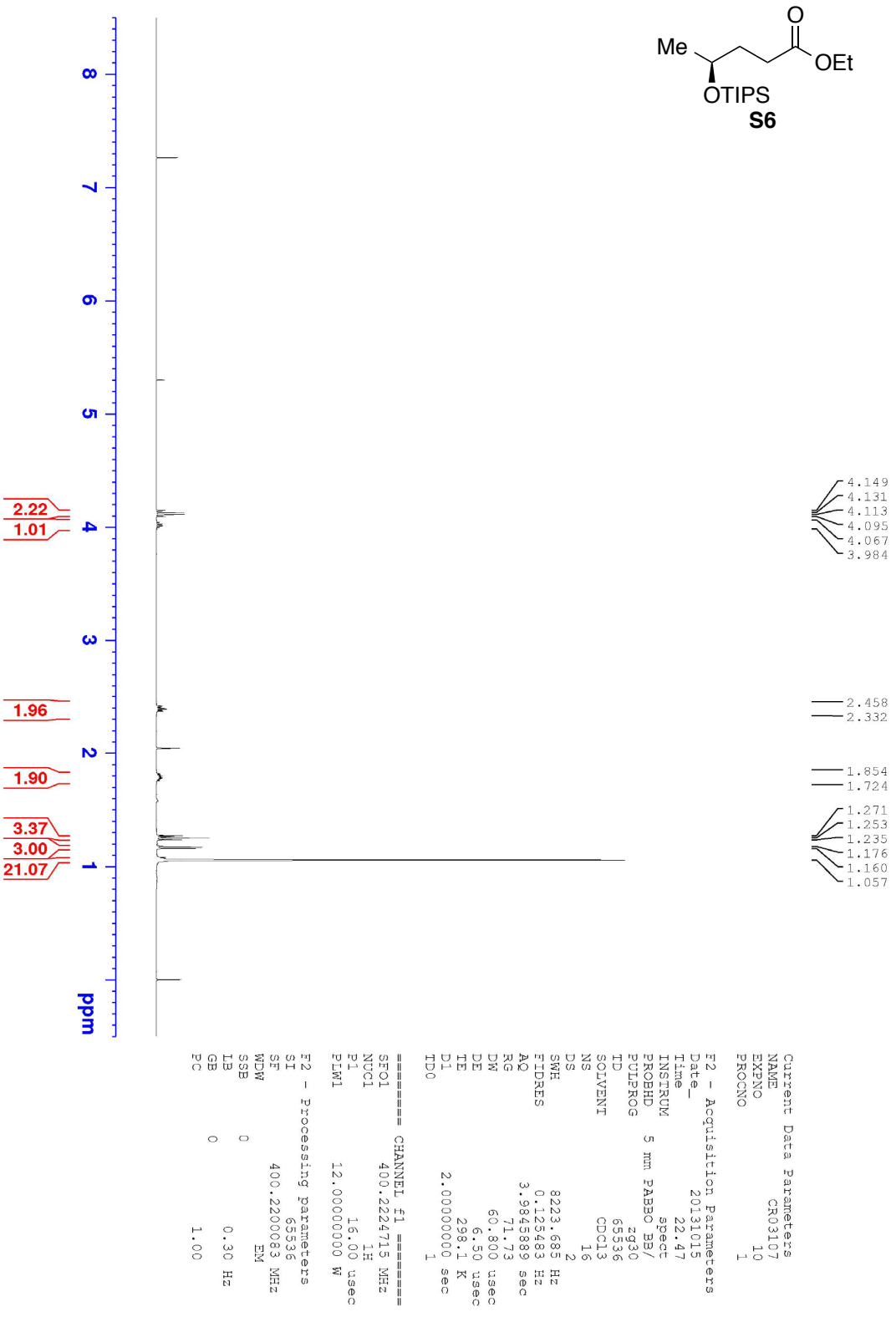
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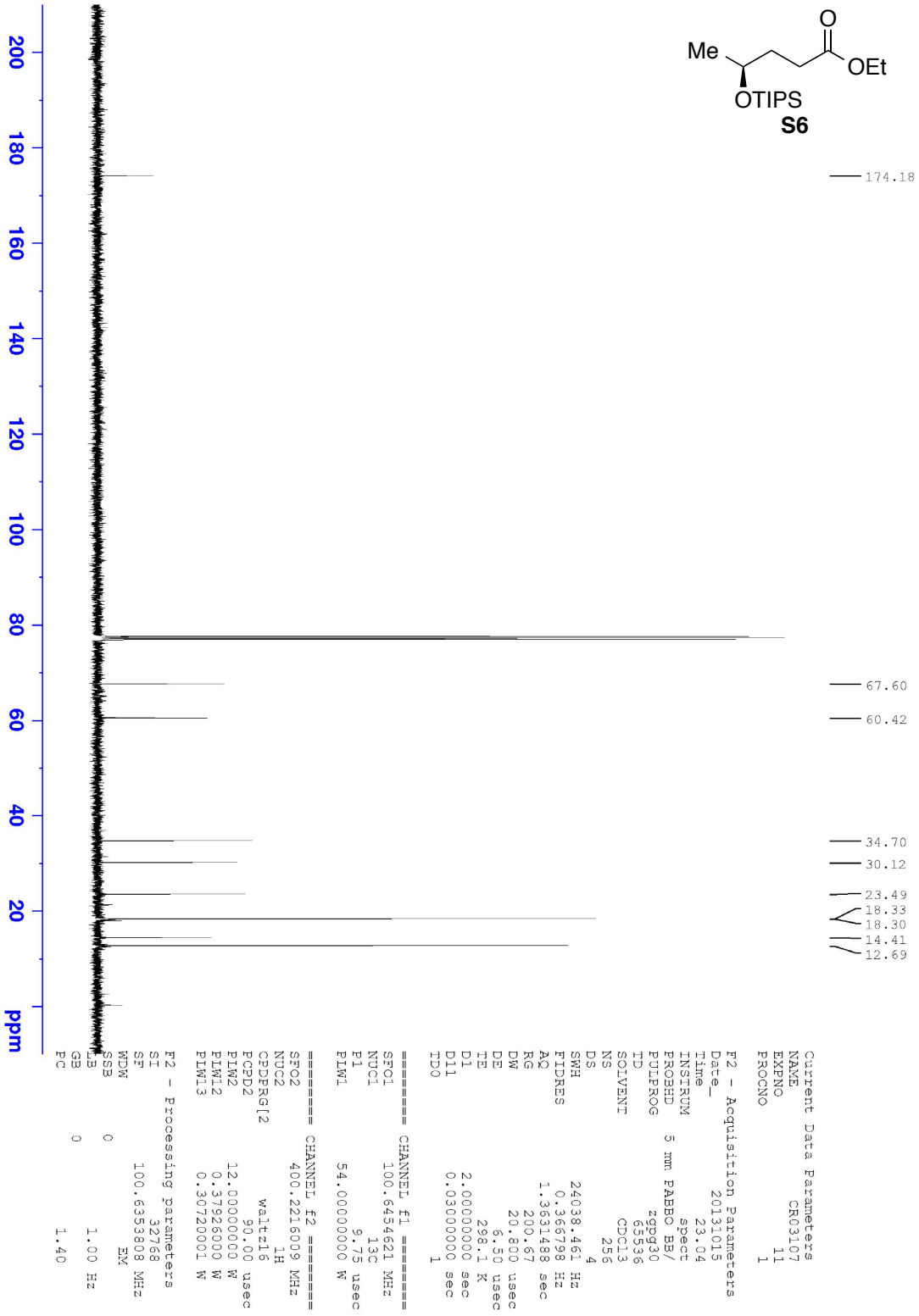
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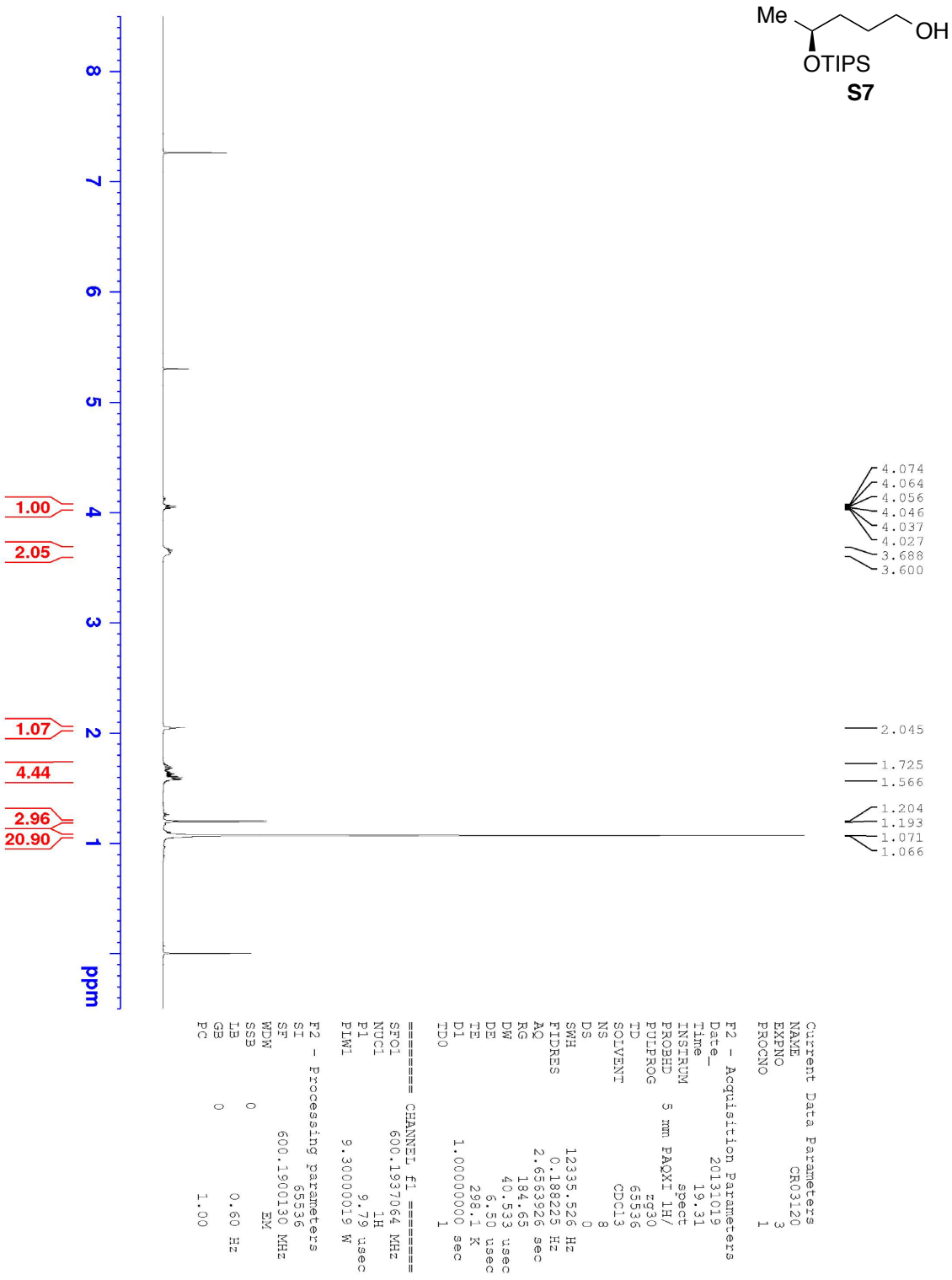
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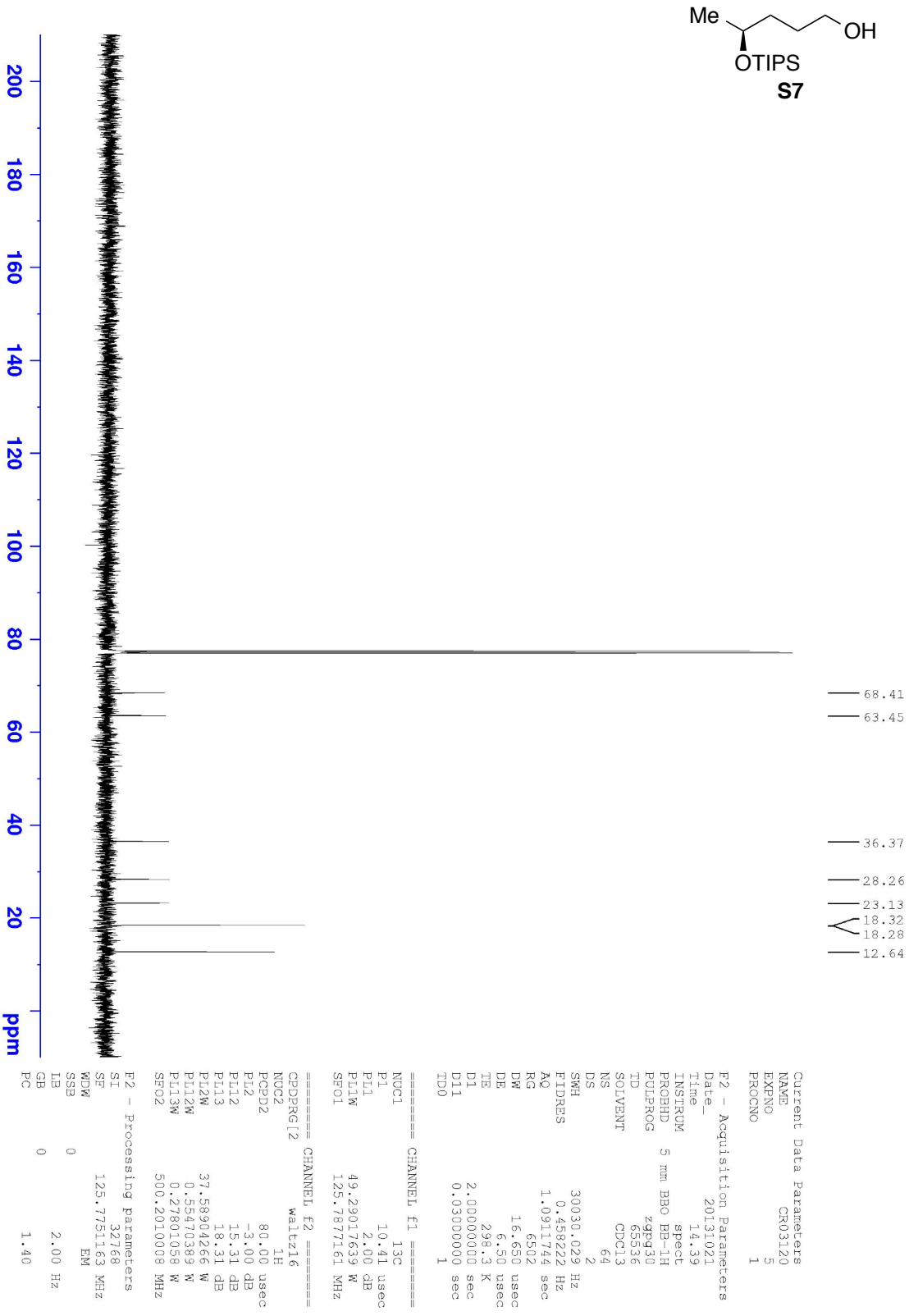
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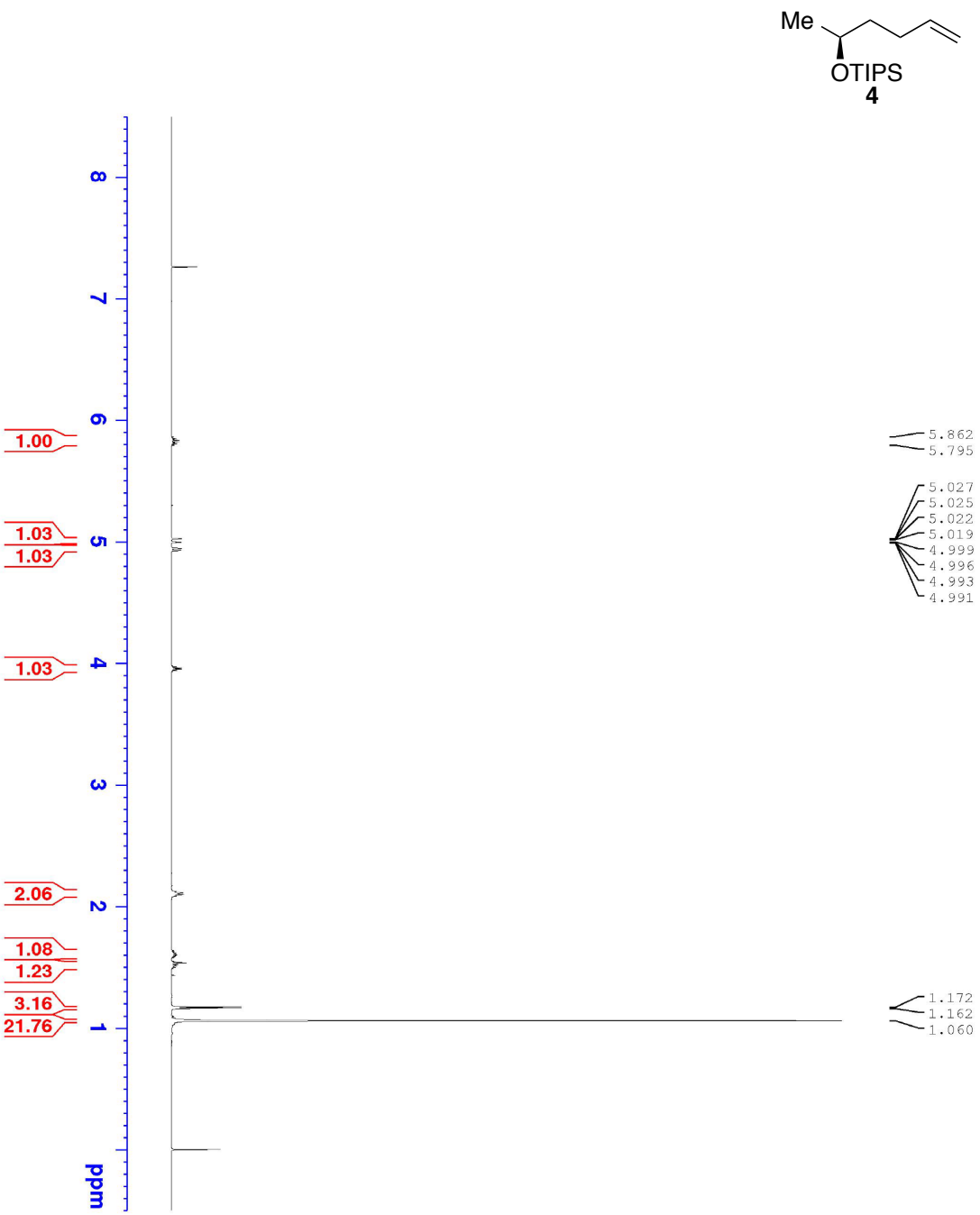
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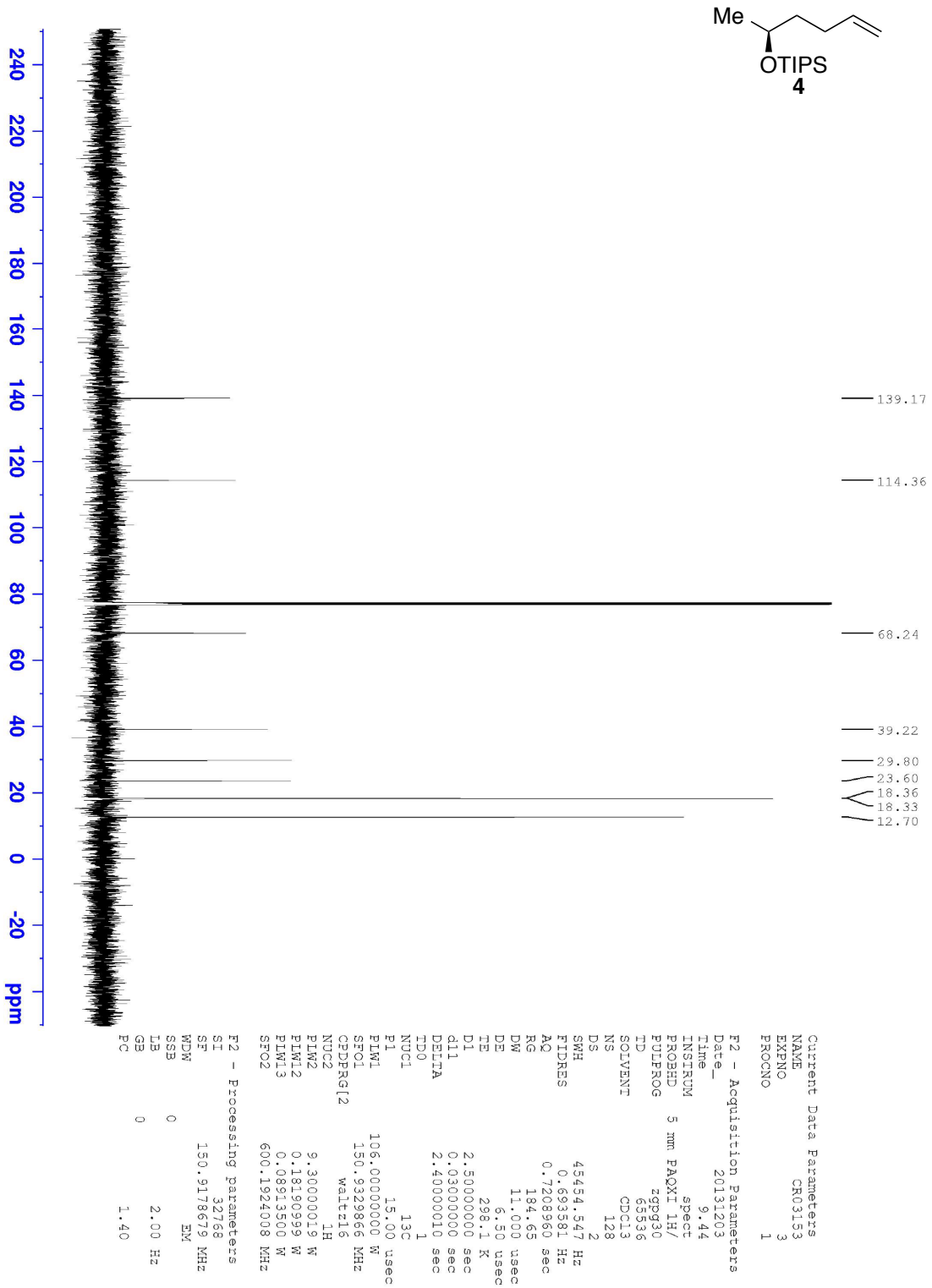
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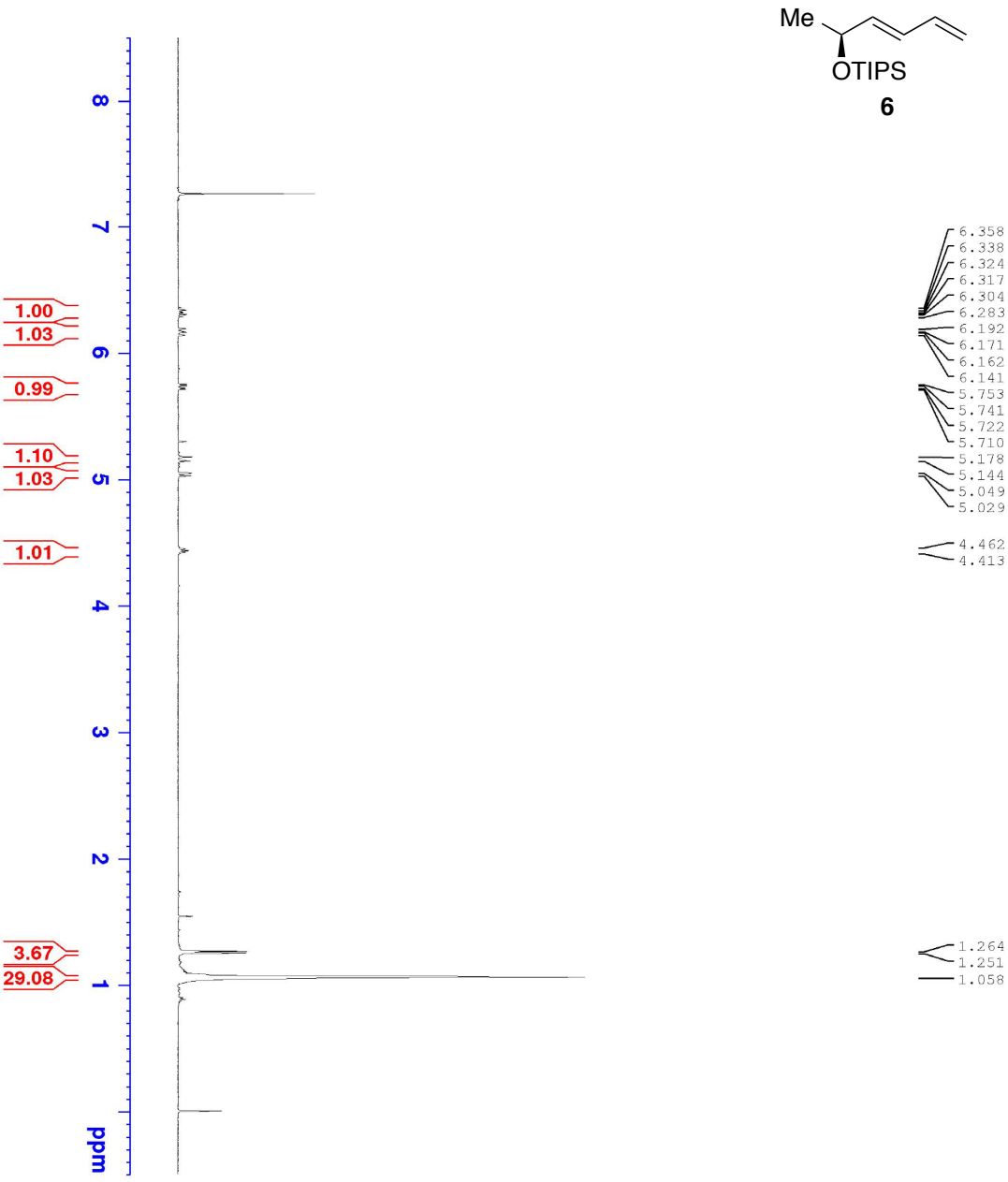
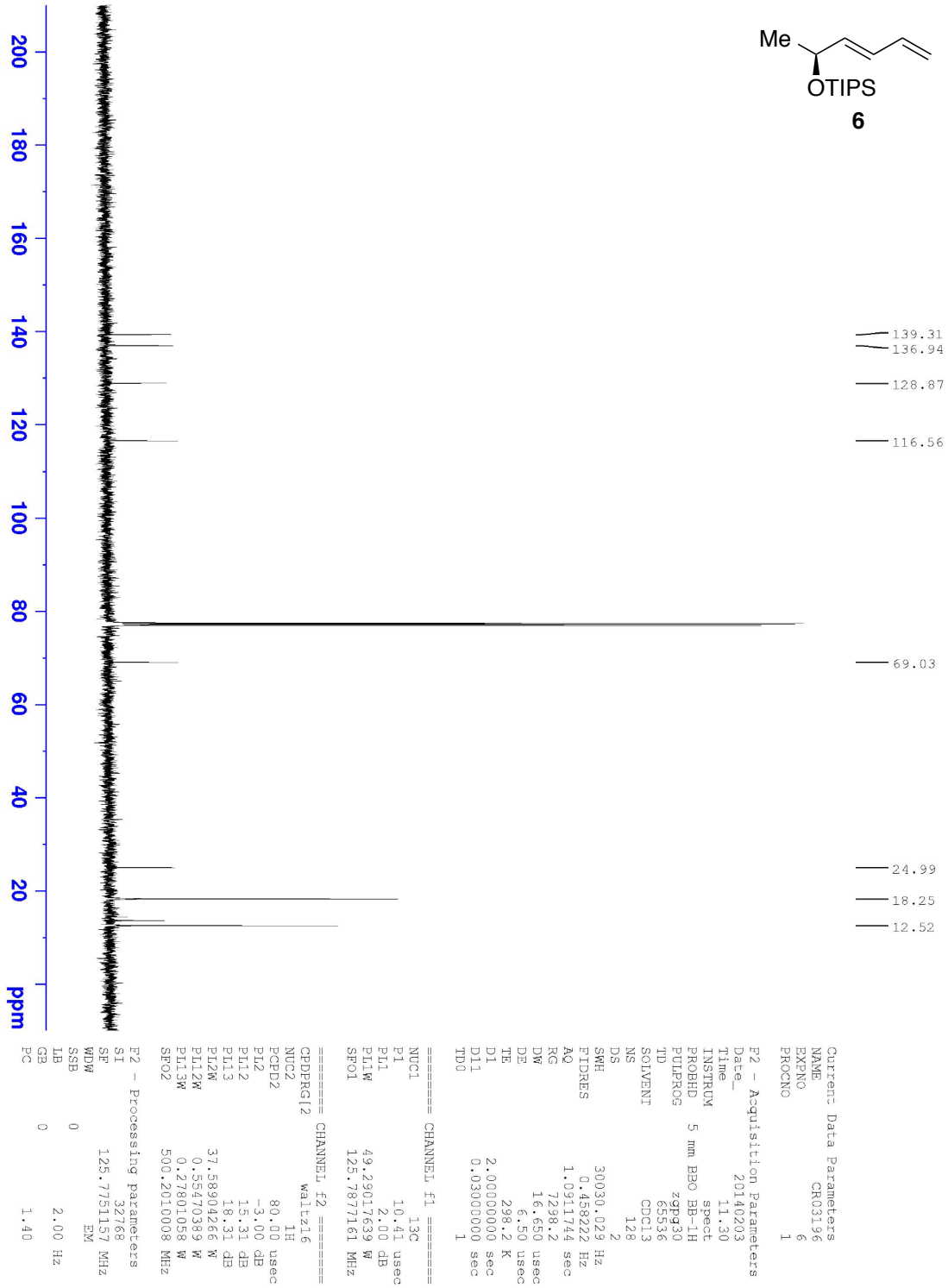
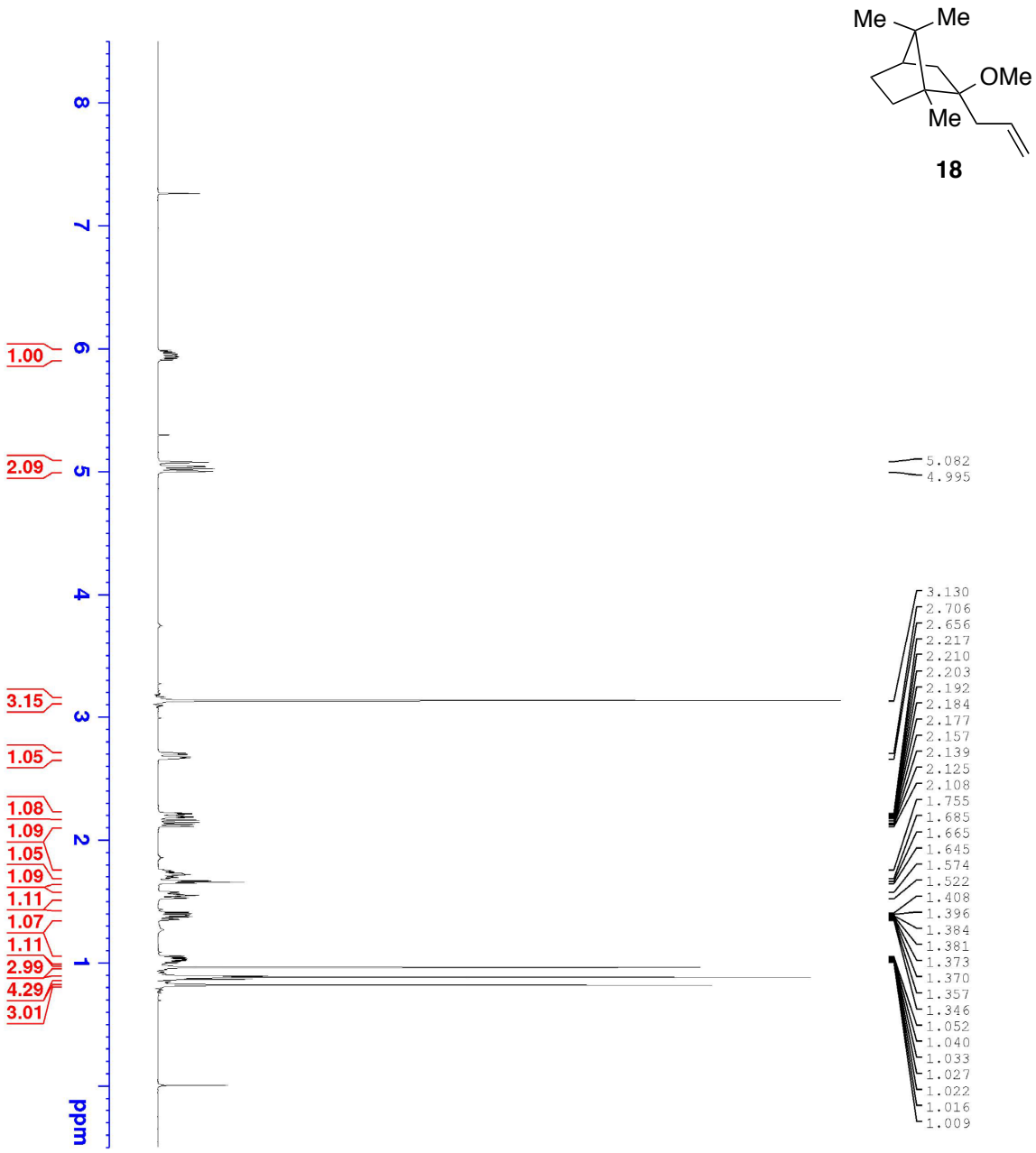
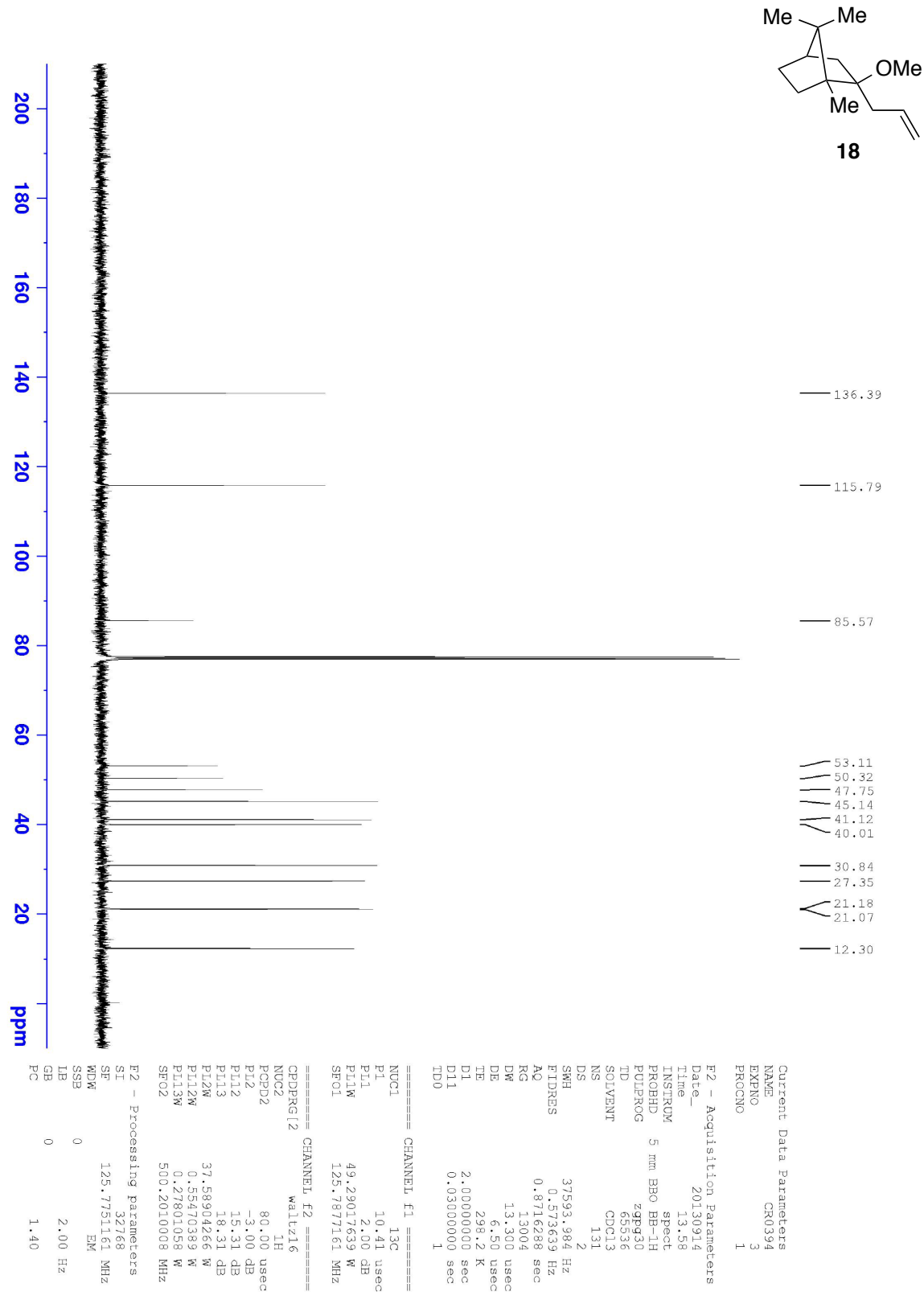
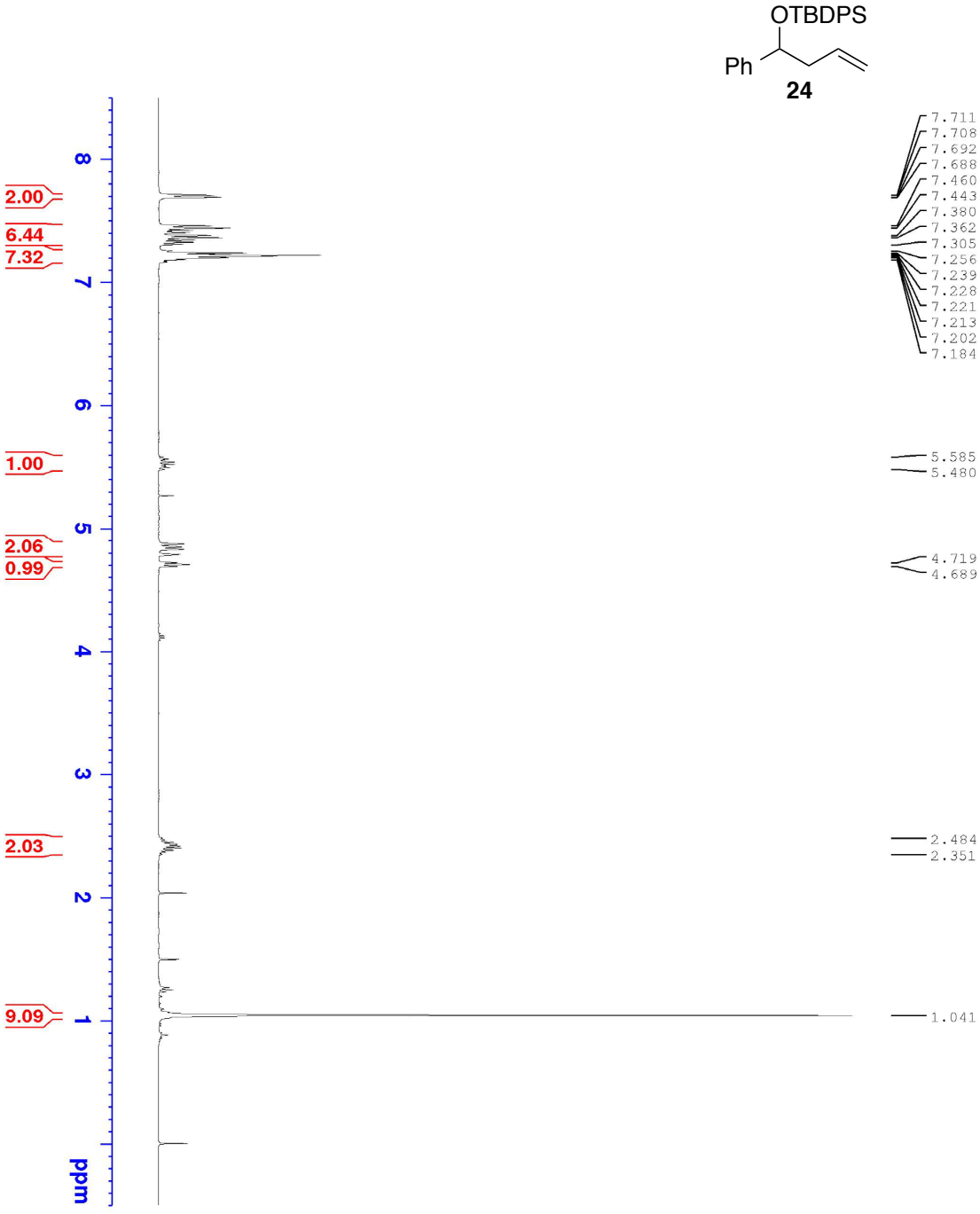
PLW2 9.30000019 W
PLW12 0.18190999 W
PLW13 0.08913500 W
SFO2 600.1924008 MHz
F2 - Processing parameters
SI 32768
SF 150.9178679 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

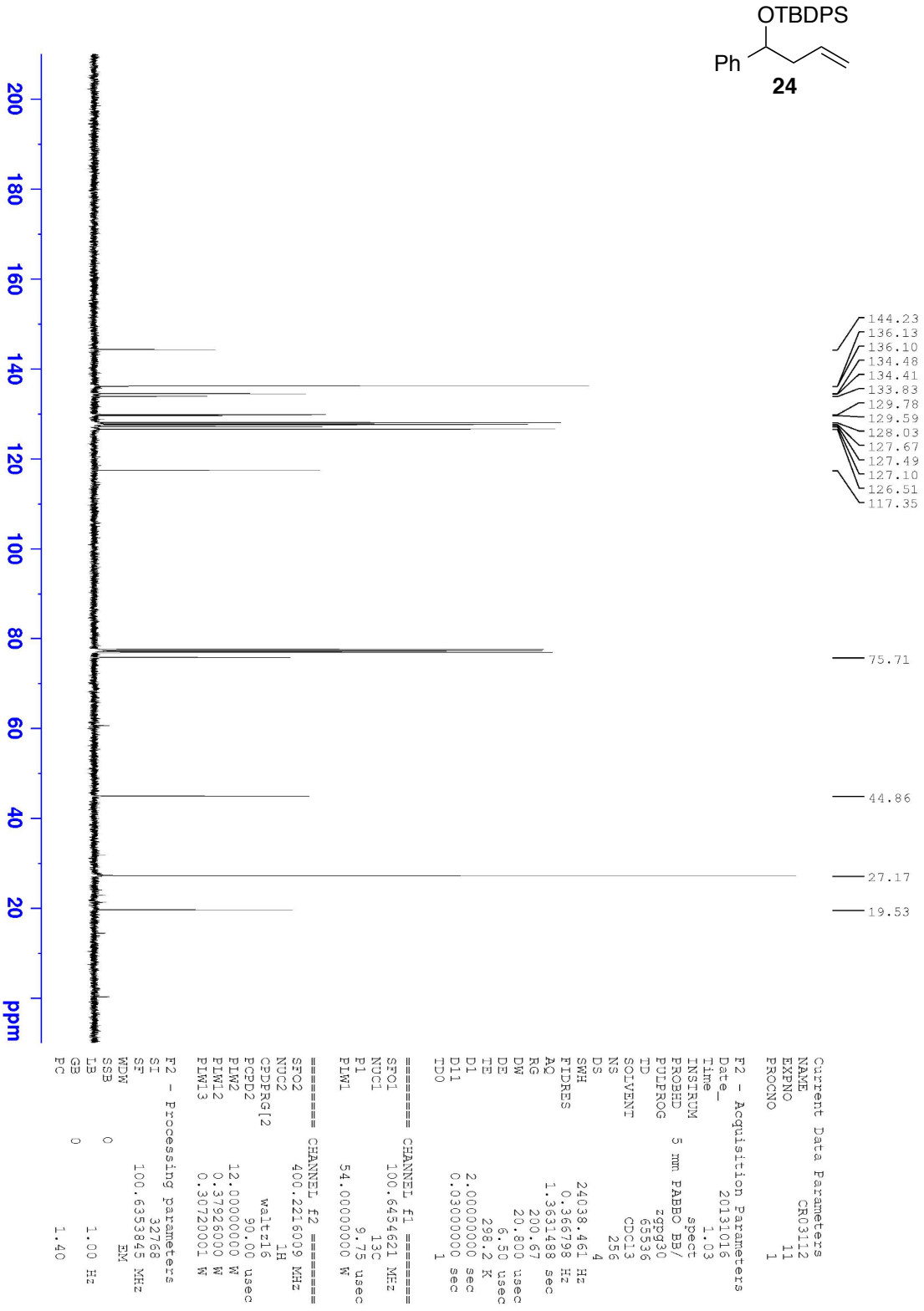

Me
OTIPS
6
6.358
6.338
6.324
6.317
6.304
6.283
6.192
6.171
6.162
6.141
5.753
5.741
5.722
5.710
5.178
5.144
5.049
5.029
4.462
4.413
1.264
1.251
1.058
1.00
1.03
0.99
1.10
1.03
1.01
3.67
29.08
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME CR03196
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140203
Time 11.23
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 128
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.72 usec
PL1 -3.00 dB
PL1W 37.58904266 W
SFO1 500.2020889 MHz
F2 - Processing parameters
SI 32768
SF 500.1990147 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

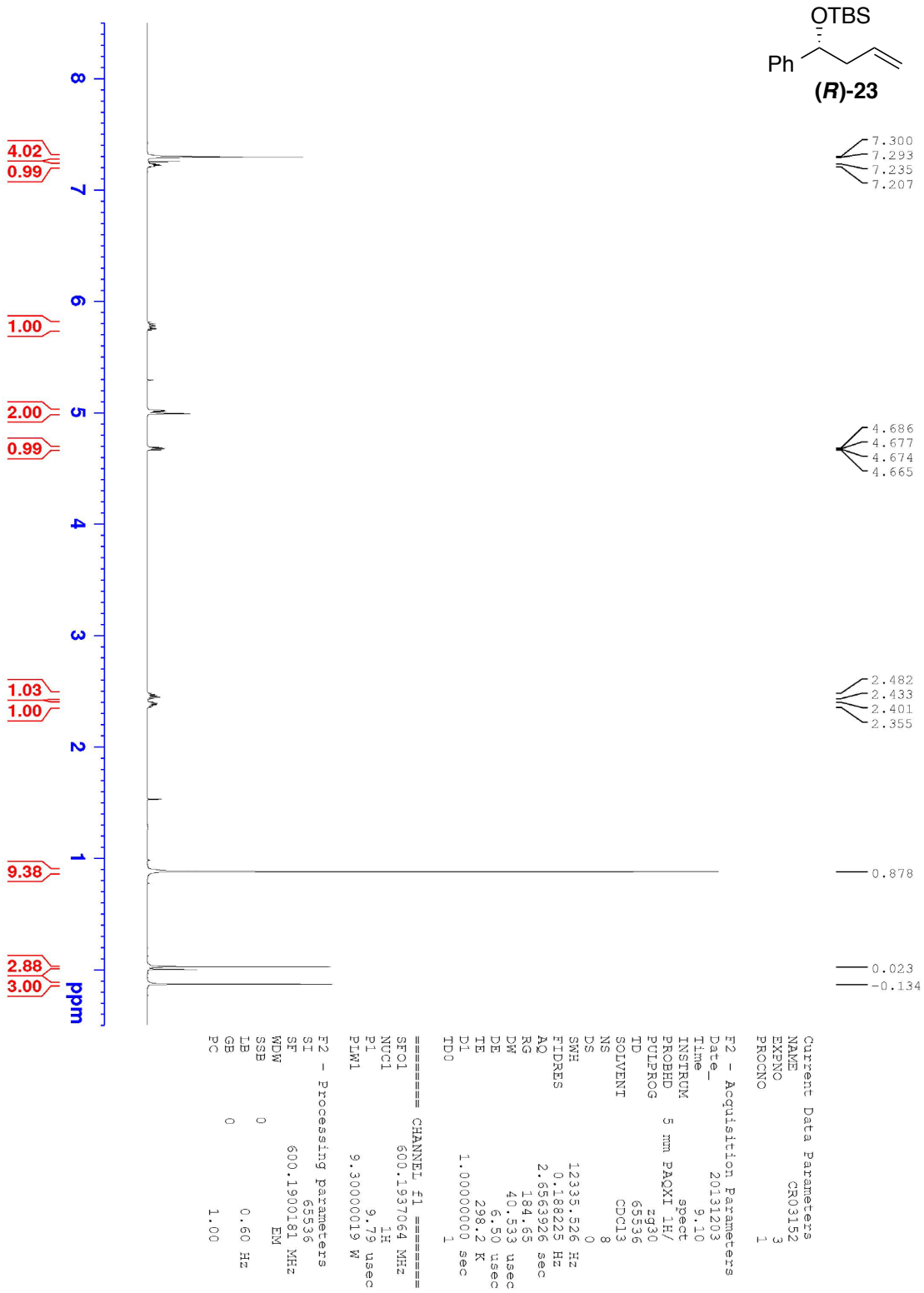

Me
OTIPS
6
139.31
136.94
128.87
116.56
69.03
24.99
18.25
12.52
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME CR03196
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140203
Time 11.30
INSTRUM spect
PROBHD 5 mm PBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 7298.2
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.41 usec
PL1 2.00 dB
PL1W 49.29017639 W
SFO1 125.7877161 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.31 dB
PL13 18.31 dB
PL2W 37.58904266 W
PL12W 0.55470389 W
PL13W 0.27801058 W
SFO2 500.2010008 MHz
F2 - Processing parameters
SI 32768
SF 125.7751157 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

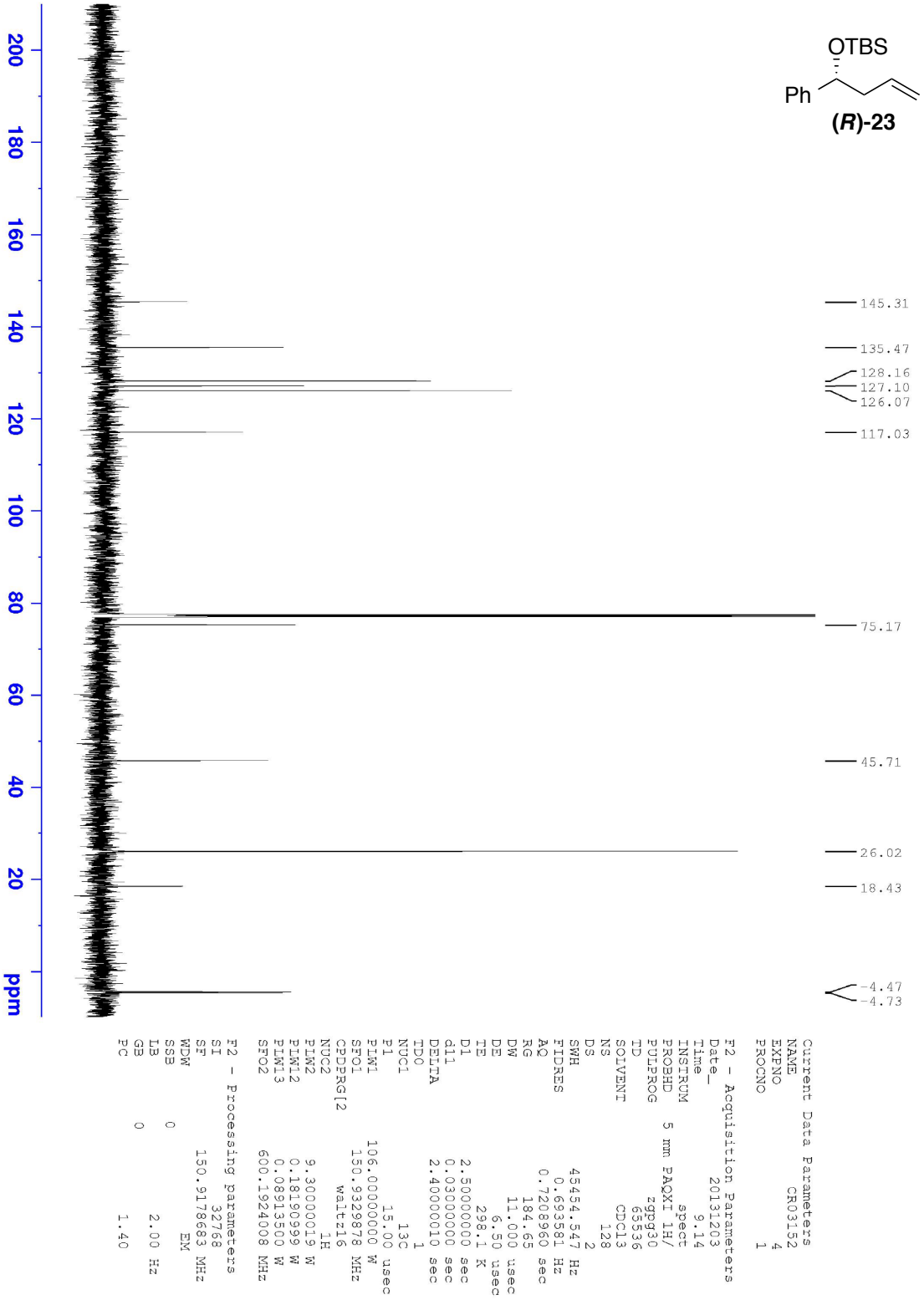

Me
Me
OMe
Me
18
1.00
2.09
3.15
1.05
1.08
1.09
1.05
1.09
1.11
1.07
1.11
2.99
4.29
3.01
ppm
5.082
4.995
3.130
2.706
2.656
2.217
2.210
2.203
2.192
2.184
2.177
2.157
2.139
2.125
2.108
1.755
1.685
1.665
1.645
1.574
1.522
1.408
1.396
1.384
1.381
1.373
1.370
1.357
1.346
1.052
1.040
1.033
1.027
1.022
1.016
1.009
Current Data Parameters
NAME CR0394
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130914
Time 13.51
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 9.72 usec
PL1 -3.00 dB
PL1W 37.58904266 W
SFO1 500.2020889 MHz
F2 - Processing parameters
SI 32768
SF 500.1990147 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

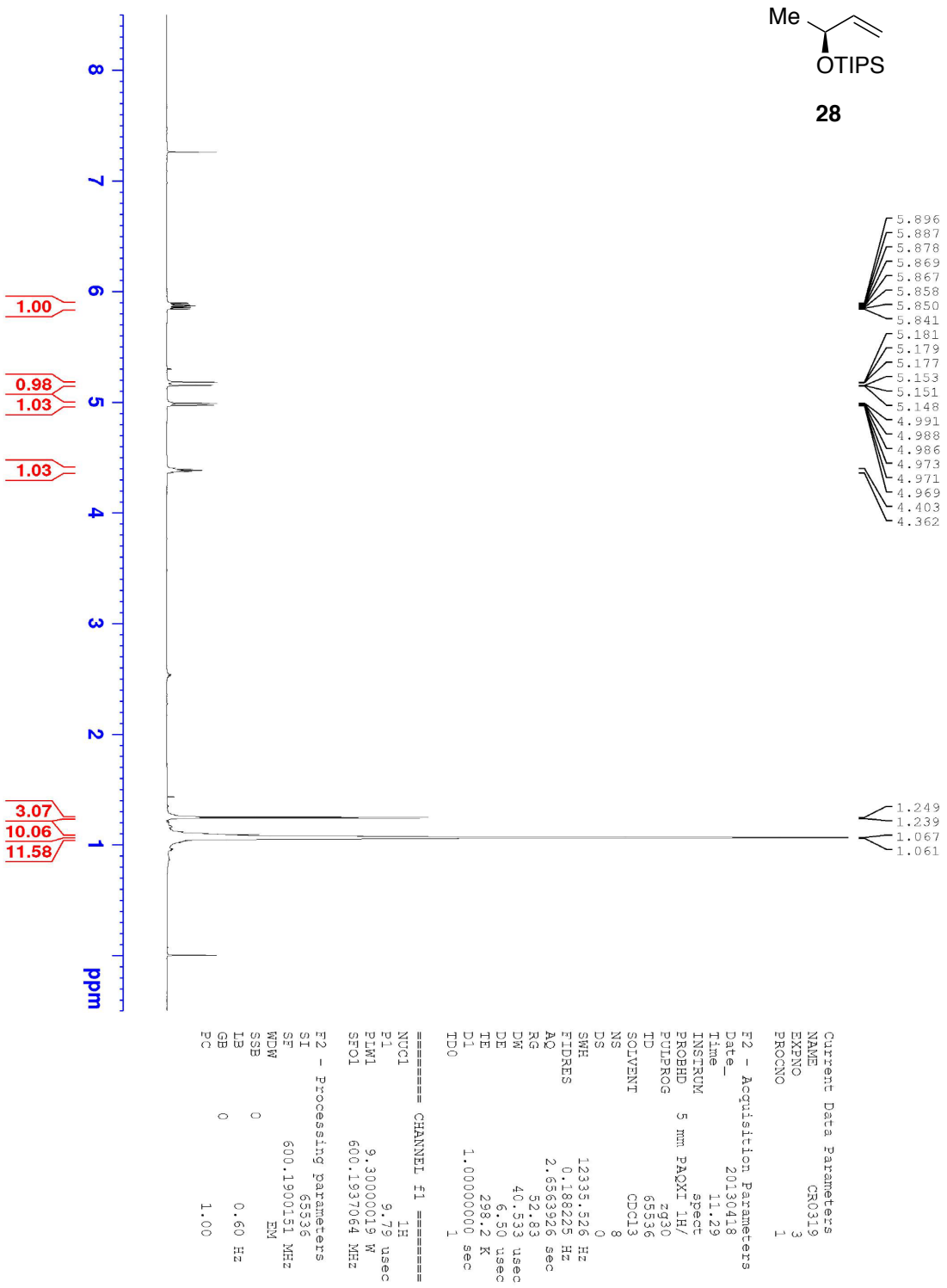

Me
Me
OMe
Me
18
136.39
115.79
85.57
53.11
50.32
47.75
45.14
41.12
40.01
30.84
27.35
21.18
21.07
12.30
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME CR0394
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130914
Time_ 13.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 131
DS 2
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716288 sec
RG 13004
DW 13.300 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 13C
P1 10.41 usec
PL1 2.00 dB
PL1W 49.29017639 W
SFO1 125.7877161 MHz
========== CHANNEL f2 ==========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.31 dB
PL13 18.31 dB
PL2W 37.58904266 W
PL12W 0.55470389 W
PL13W 0.27801058 W
SFO2 500.2010008 MHz
F2 - Processing parameters
SI 32768
SF 125.7751161 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

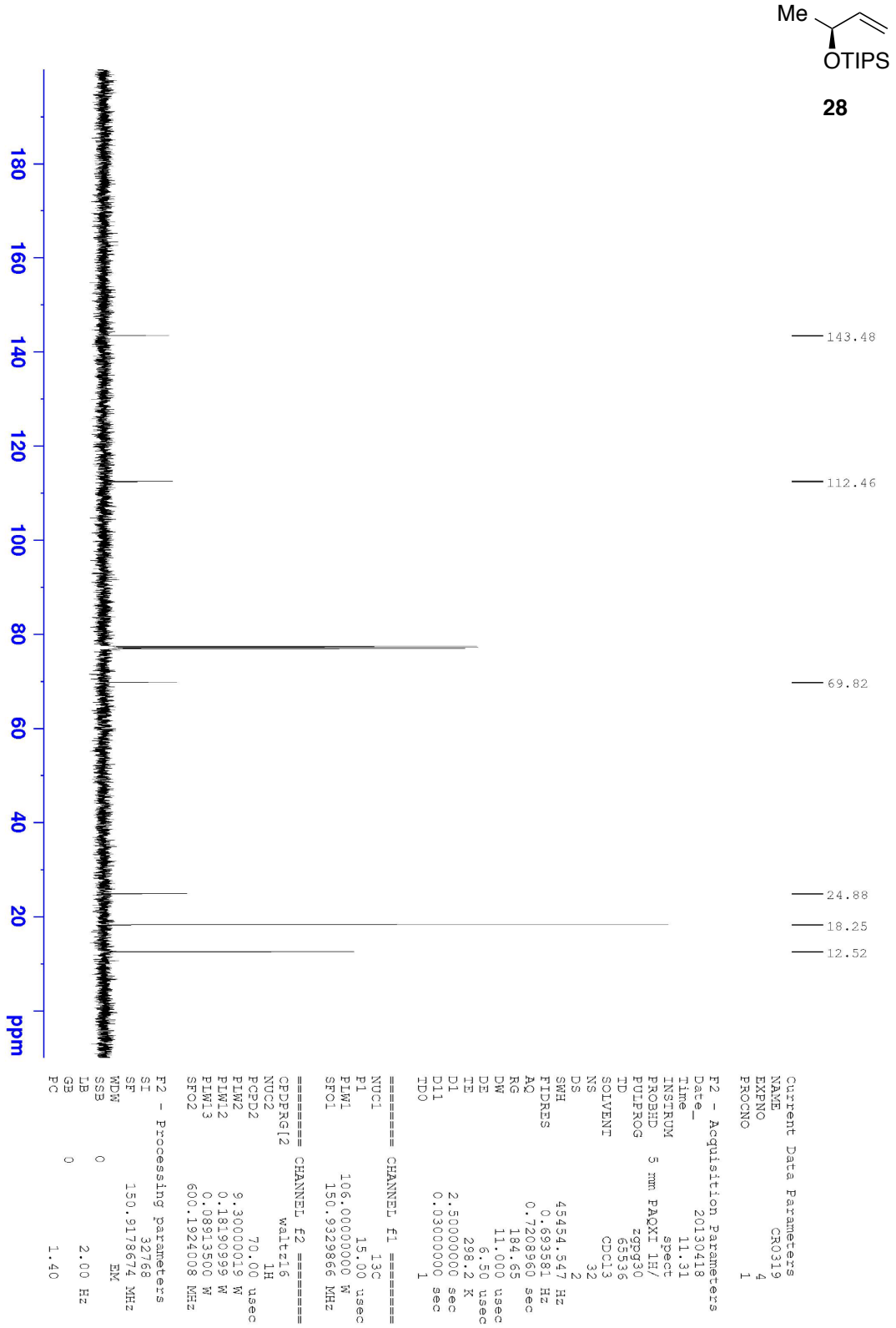

OTBDPS
Ph
24
7.711
7.708
7.692
7.688
7.460
7.443
7.380
7.362
7.305
7.256
7.239
7.228
7.221
7.213
7.202
7.184
5.585
5.480
4.719
4.689
2.484
2.351
1.041
2.00
6.44
7.32
1.00
2.06
0.99
2.03
9.09
ppm
Current Data Parameters
NAME CR03112
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131016
Time 0.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 71.73
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
========== CHANNEL f1 ==========
SFO1 400.2224715 MHz
NUC1 1H
P1 16.00 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 65536
SF 400.2200223 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

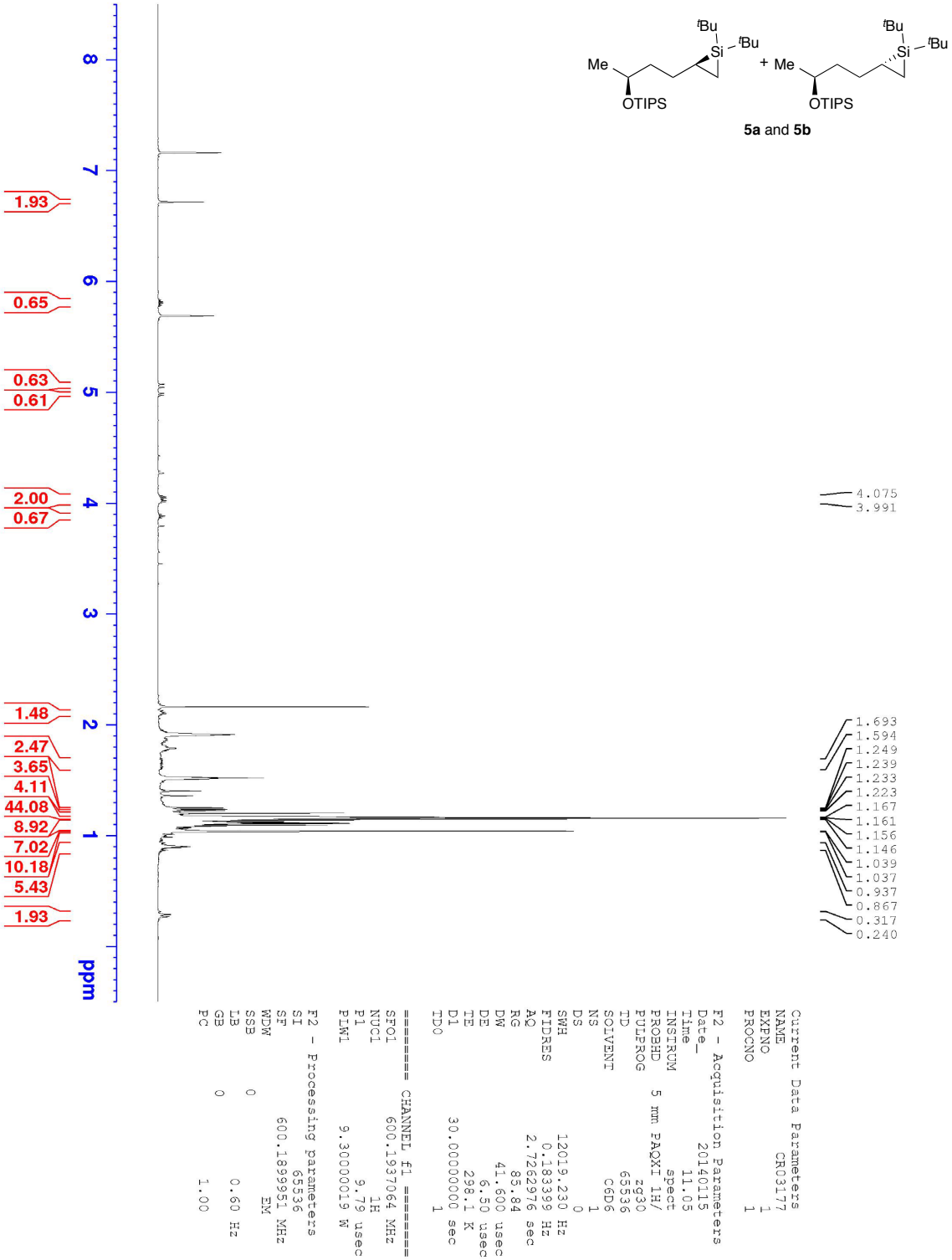

OTBDPS
Ph
24
144.23
136.13
136.10
134.48
134.41
133.83
129.78
129.59
128.03
127.67
127.49
127.10
126.51
117.35
75.71
44.86
27.17
19.53
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME CR03112
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131016
Time 1.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 200.67
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6454621 MHz
NUC1 13C
P1 9.75 usec
PLW1 54.00000000 W
======== CHANNEL f2 ========
SFO2 400.2216009 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.37926000 W
PLW13 0.30720001 W
F2 - Processing parameters
SI 32768
SF 100.6353845 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

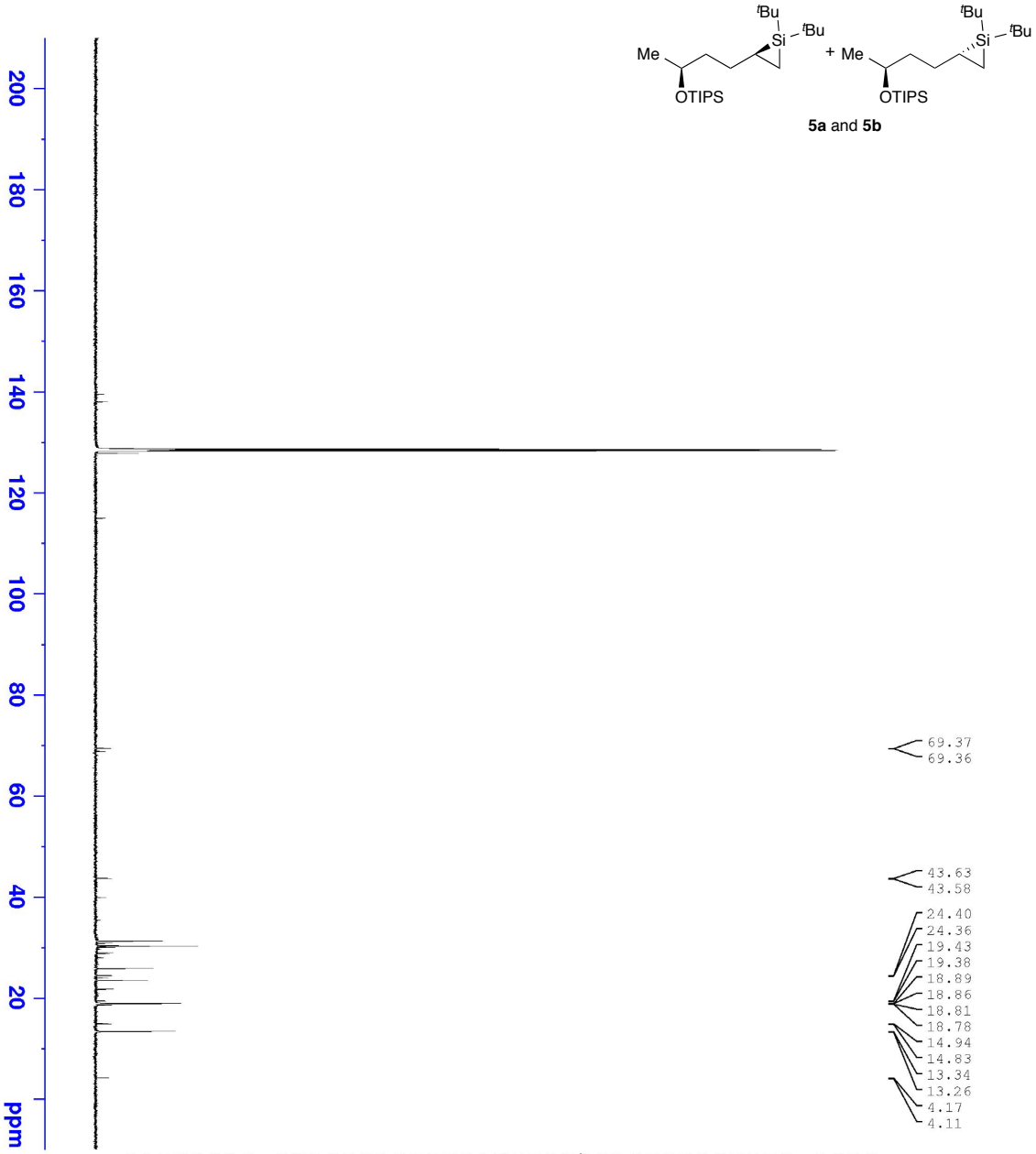

OTBS
Ph
(R)-23
7.300
7.293
7.235
7.207
4.686
4.677
4.674
4.665
2.482
2.433
2.401
2.355
0.878
0.023
-0.134
4.02
0.99
1.00
2.00
0.99
1.03
1.00
9.38
2.88
3.00
ppm
Current Data Parameters
NAME CR03152
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131203
Time 9.10
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6563926 sec
RG 184.65
DW 40.533 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1937064 MHz
NUC1 1H
P1 9.79 usec
PLW1 9.30000019 W
F2 - Processing parameters
SI 65536
SF 600.1900181 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

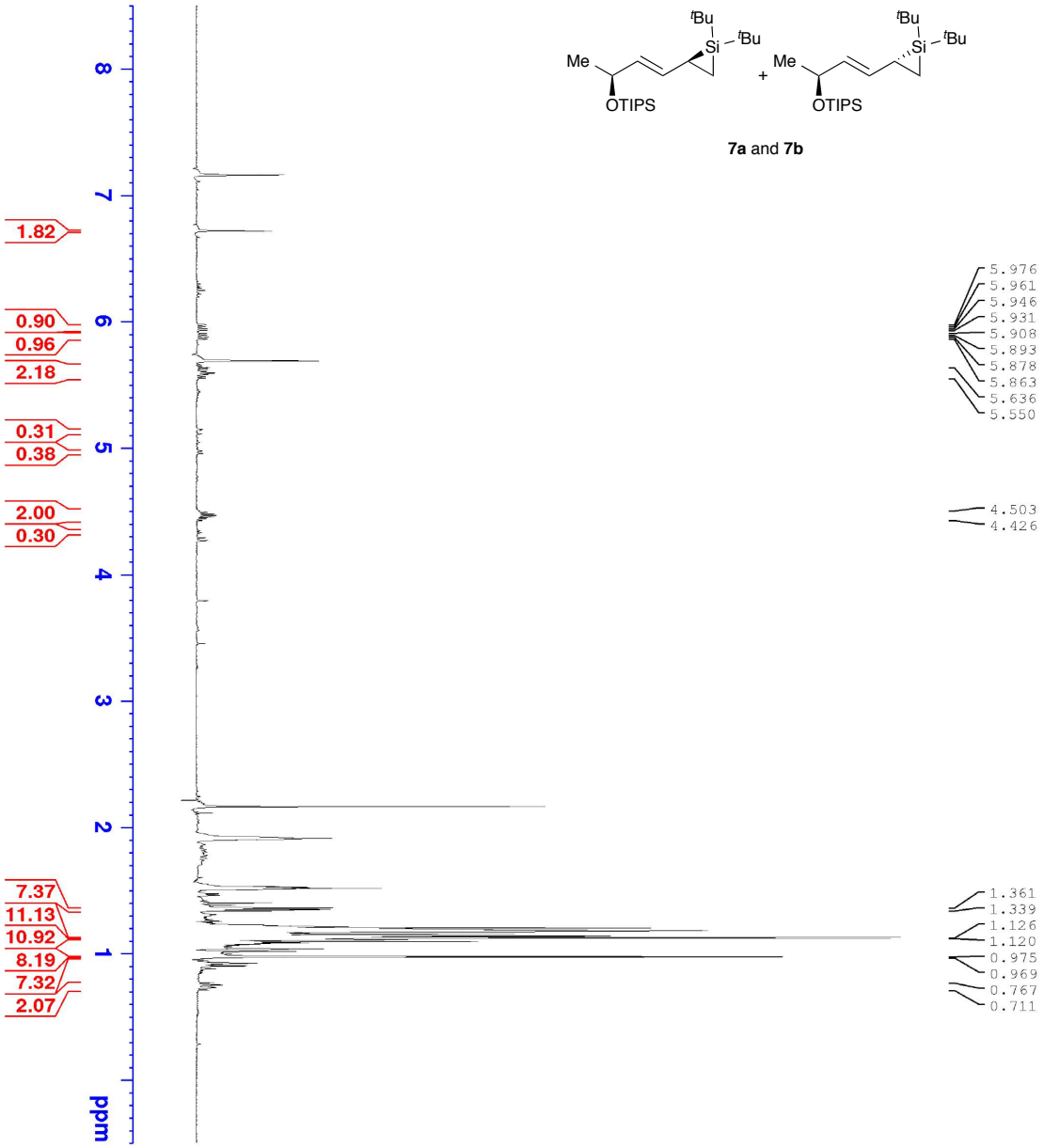

OTBS
Ph
(R)-23
145.31
135.47
128.16
127.10
126.07
117.03
75.17
45.71
26.02
18.43
-4.47
-4.73
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME CR03152
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131203
Time 9.14
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 45454.547 Hz
FIDRES 0.693581 Hz
AQ 0.7208960 sec
RG 184.65
DW 11.000 usec
DE 6.50 usec
TE 298.1 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
NUC1 13C
P1 15.00 usec
PL1 106.00000000 W
SFO1 150.9329878 MHz
CPDPRG[2 waltz16
NUC2 1H
PL2 9.30000019 W
PL12 0.18190999 W
PL13 0.08913500 W
SFO2 600.1924008 MHz
F2 - Processing parameters
SI 32768
SF 150.9179683 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

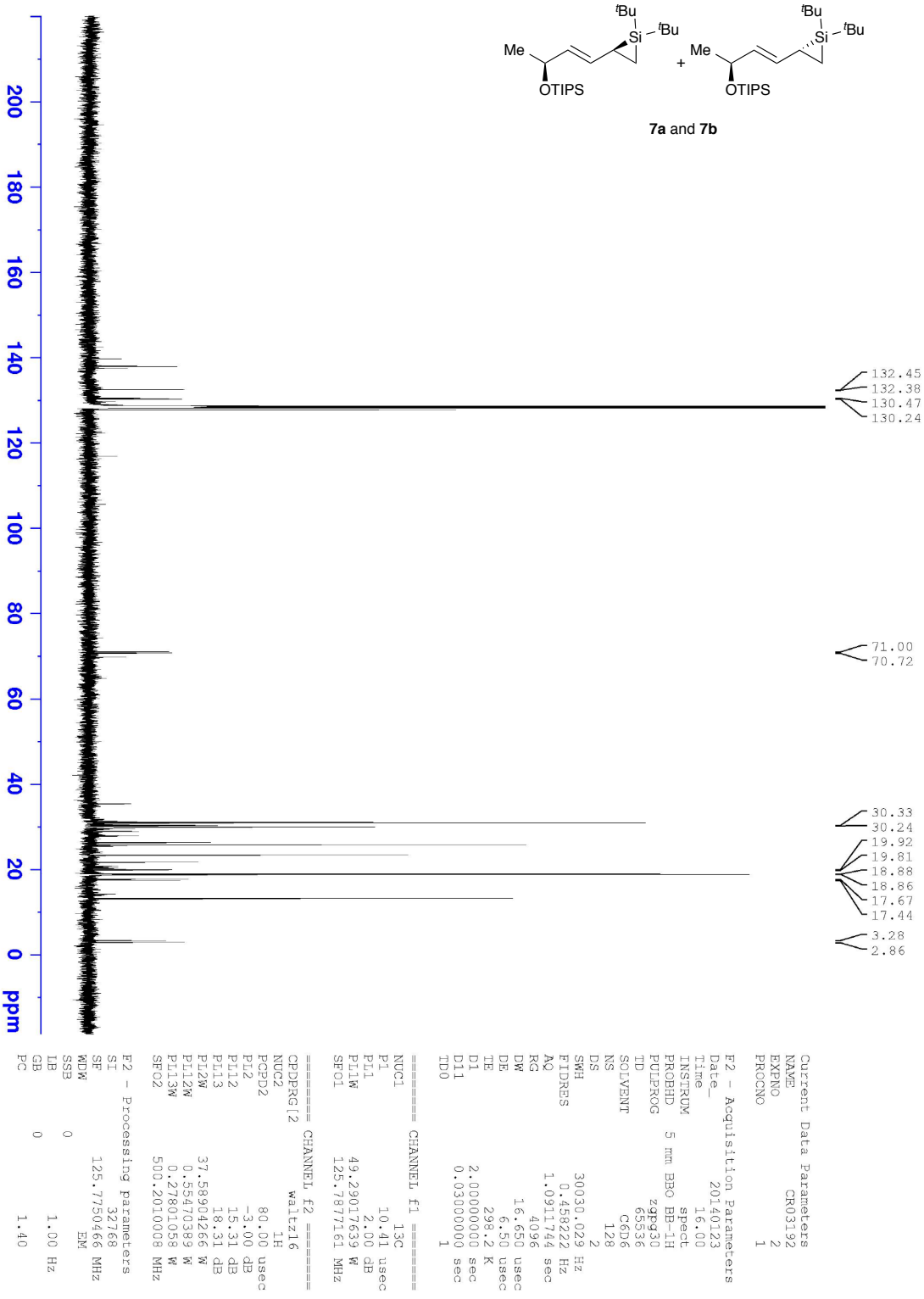

Me
OTIPS
28
5.896
5.887
5.878
5.869
5.867
5.858
5.850
5.841
5.181
5.179
5.177
5.153
5.151
5.148
4.991
4.988
4.986
4.973
4.971
4.969
4.403
4.362
1.249
1.239
1.067
1.061
1.00
0.98
1.03
1.03
3.07
10.06
11.58
ppm
Current Data Parameters
NAME CR0319
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130418
Time 11.29
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6563926 sec
RG 52.83
DW 40.533 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 9.79 usec
PLW1 9.30000019 W
SFO1 600.1937064 MHz
F2 - Processing parameters
SI 65536
SF 600.1900151 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

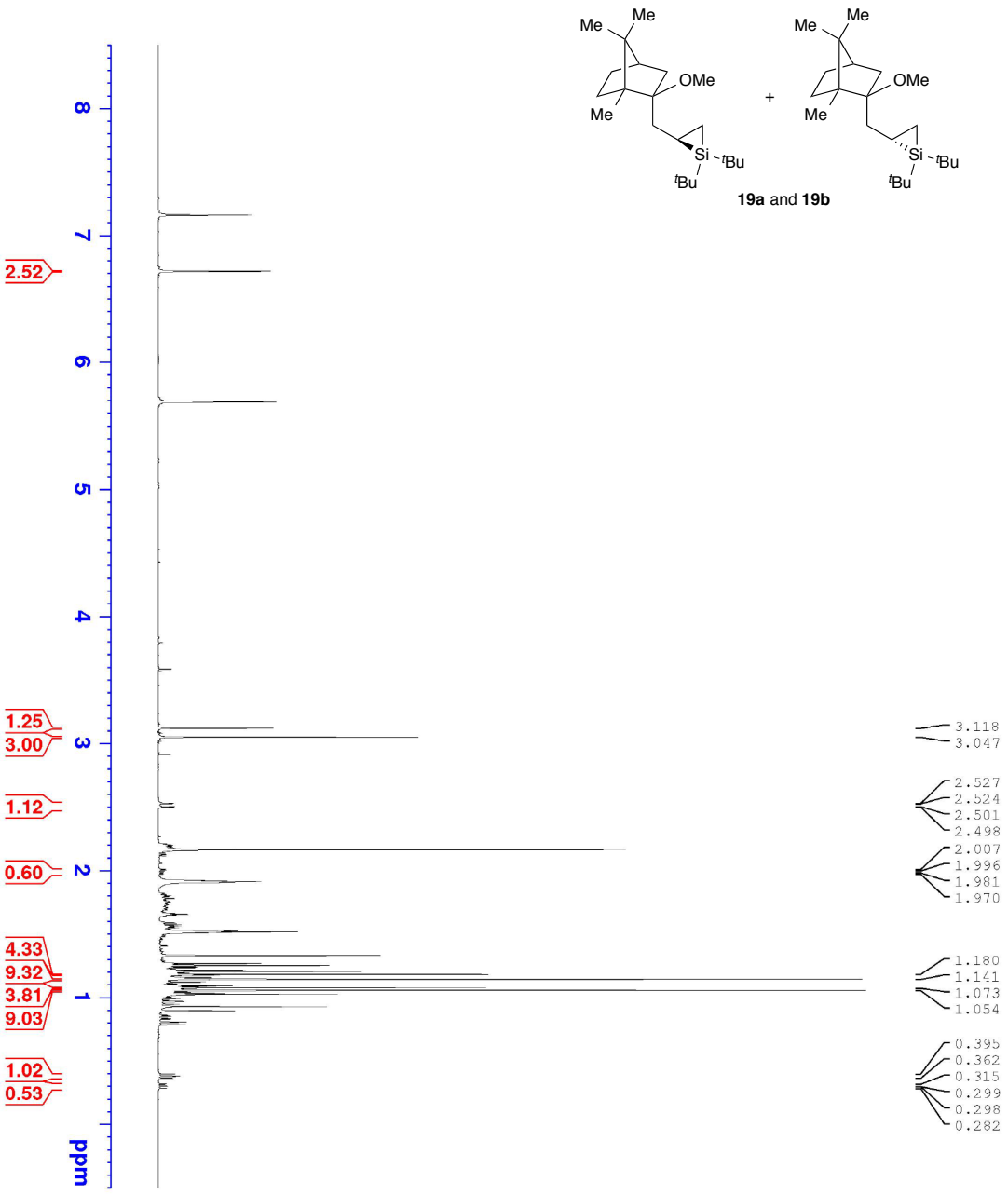

Me
OTIPS
28
143.48
112.46
69.82
24.88
18.25
12.52
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME CR0319
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130418
Time 11.31
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 45454.547 Hz
FIDRES 0.693581 Hz
AQ 0.7208960 sec
RG 184.65
DW 11.000 usec
DE 6.50 usec
TE 298.2 K
D1 2.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PLW1 106.00000000 W
SFO1 150.9329866 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 9.30000019 W
PLW12 0.18190999 W
PLW13 0.08913500 W
SFO2 600.1924008 MHz
F2 - Processing parameters
SI 32768
SF 150.9178674 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

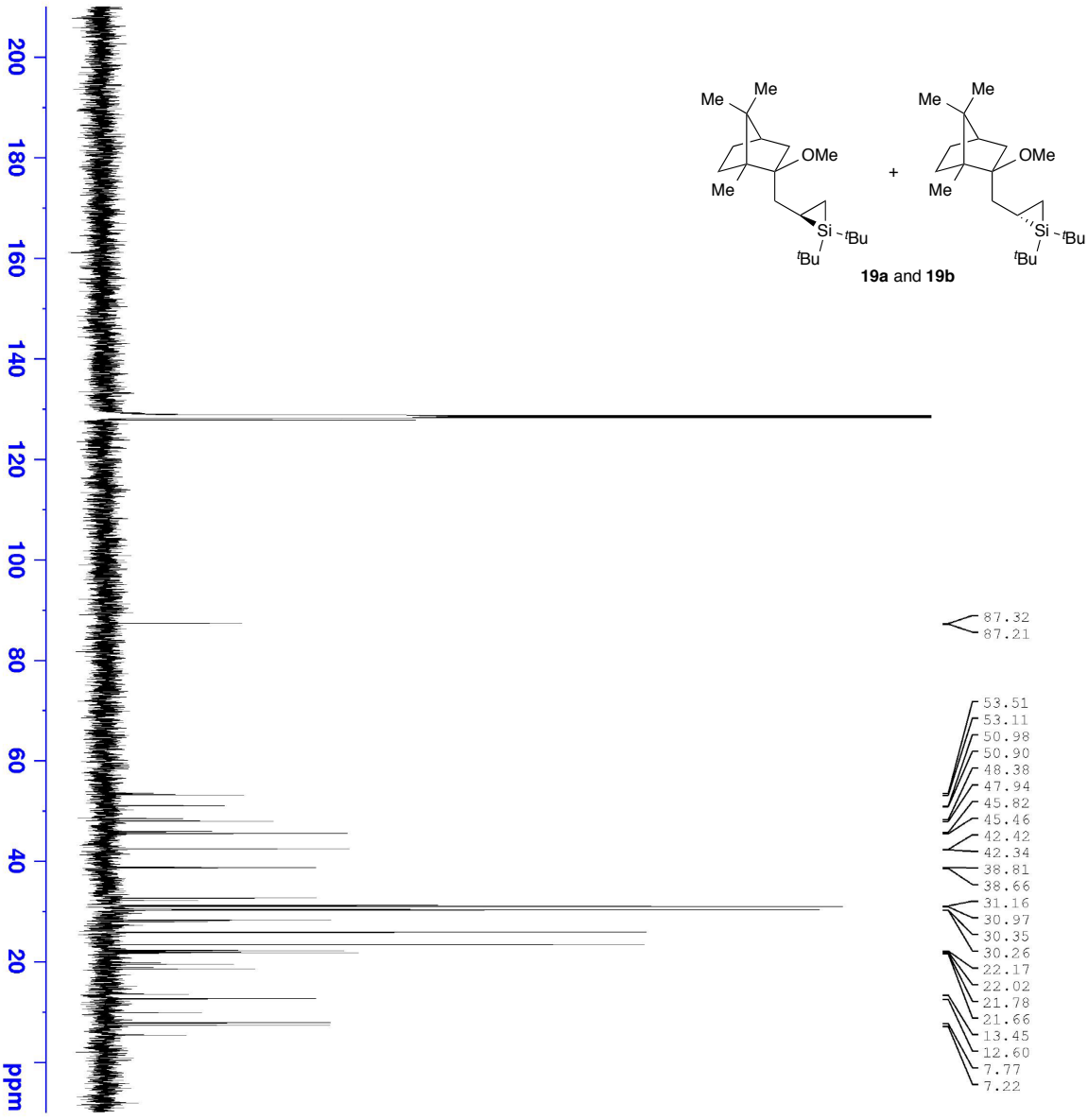

tBu
tBu
Si
Me
OTIPS
+ Me
5a and 5b
1.93
0.65
0.63
0.61
2.00
0.67
1.48
2.47
3.65
4.11
44.08
8.92
7.02
10.18
5.43
1.93
ppm
4.075
3.991
1.693
1.594
1.249
1.239
1.233
1.223
1.167
1.161
1.156
1.146
1.039
1.037
0.937
0.867
0.317
0.240
Current Data Parameters
NAME CR03177
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140115
Time 11.05
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 85.84
DW 41.600 usec
DE 6.50 usec
TE 298.1 K
D1 30.0000000 sec
TD0 1
========== CHANNEL f1 ==========
SFO1 600.1937064 MHz
NUC1 1H
P1 9.79 usec
PL1 9.30000019 W
F2 - Processing parameters
SI 65536
SF 600.1899951 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00


tBu
tBu
Si
Me
OTIPS
+ Me
5a and 5b
69.37
69.36
43.63
43.58
24.40
24.36
19.43
19.38
18.89
18.86
18.81
18.78
14.94
14.83
13.34
13.26
4.17
4.11
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME CR03185
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140120
Time_ 16.12
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 128
DS 2
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 184.65
DW 13.867 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
NUC1 13C
P1 15.00 usec
PLW1 106.00000000 W
SFO1 150.9329866 MHz
CPDPRG[2 waltz16
NUC2 1H
PLW2 9.30000019 W
PLW12 0.18190999 W
PLW13 0.08913500 W
SFO2 600.1924008 MHz
F2 - Processing parameters
SI 32768
SF 150.9177839 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40

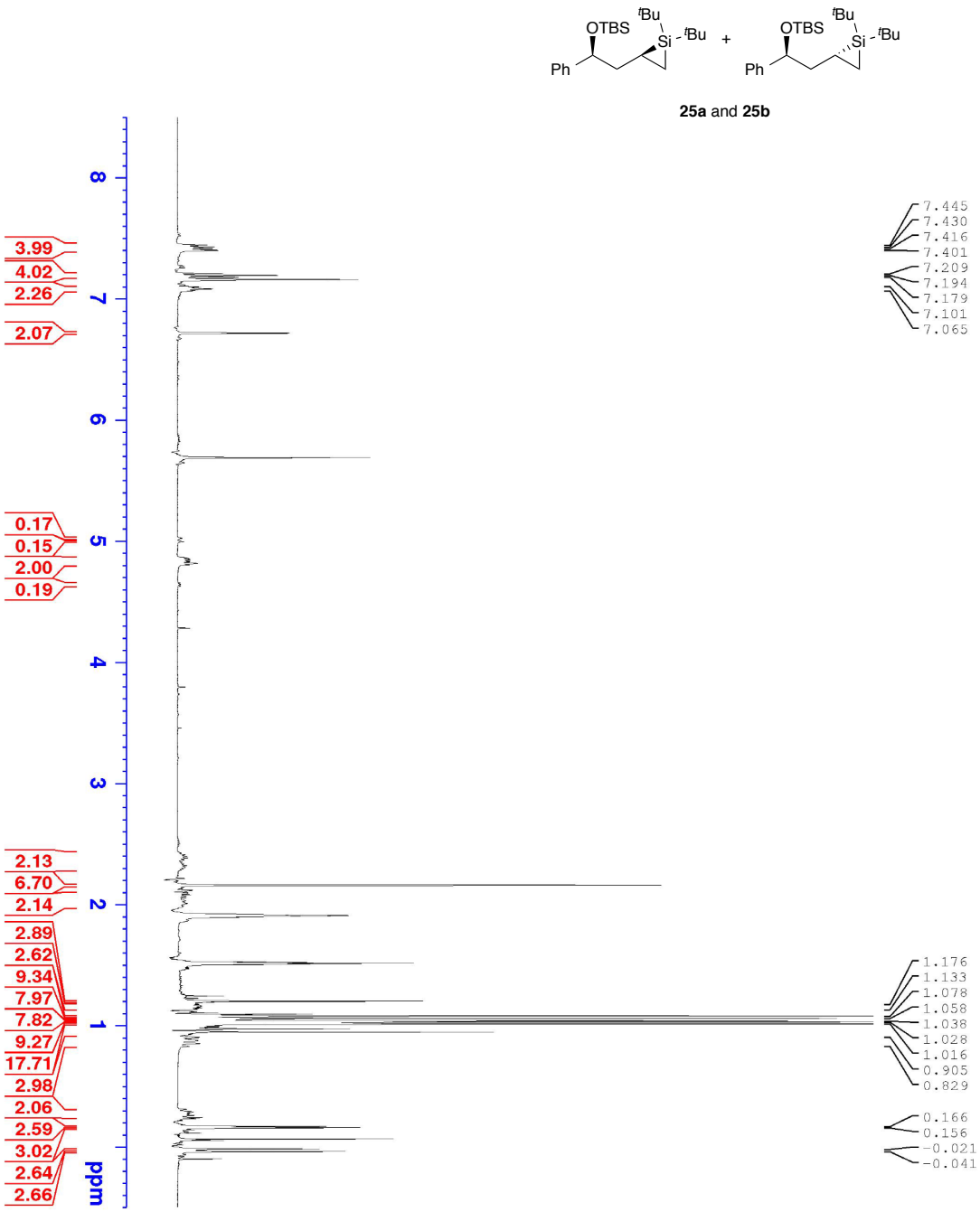

tBu
Si
tBu
Me
OTIPS
+
tBu
Si
tBu
Me
OTIPS
7a and 7b
5.976
5.961
5.946
5.931
5.908
5.893
5.878
5.863
5.636
5.550
4.503
4.426
1.361
1.339
1.126
1.120
0.975
0.969
0.767
0.711
1.82
0.90
0.96
2.18
0.31
0.38
2.00
0.30
7.37
11.13
10.92
8.19
7.32
2.07
ppm
Current Data Parameters
NAME CR03192
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140123
Time_ 15.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 30.0000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 7.55 usec
PL1 0 dB
PL1W 18.83914757 W
SFO1 500.2020889 MHz
F2 - Processing parameters
SI 32768
SF 500.1989983 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

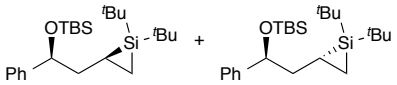

tBu
Si
tBu
Me
OTIPS
+
Me
OTIPS
7a and 7b
132.45
132.38
130.47
130.24
71.00
70.72
30.33
30.24
19.92
19.81
18.88
18.86
17.67
17.44
3.28
2.86
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME CR03192
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140123
Time 16.00
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 128
DS 2
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 4096
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.41 usec
PL1 2.00 dB
PL1W 49.29017639 W
SFO1 125.7877161 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.31 dB
PL13 18.31 dB
PL2W 37.58904266 W
PL12W 0.55470389 W
PL13W 0.27801058 W
SFO2 500.2010008 MHz
F2 - Processing parameters
SI 32768
SF 125.7750466 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


Me
Me
OMe
Me
Si
tBu
tBu
+
19a and 19b
2.52
1.25
3.00
1.12
0.60
4.33
9.32
3.81
9.03
1.02
0.53
ppm
3.118
3.047
2.527
2.524
2.501
2.498
2.007
1.996
1.981
1.970
1.180
1.141
1.073
1.054
0.395
0.362
0.315
0.299
0.298
0.282
Current Data Parameters
NAME CR0389
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130905
Time 16.46
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 85.84
DW 41.600 usec
DE 6.50 usec
TE 298.1 K
D1 30.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1937064 MHz
NUC1 1H
P1 9.79 usec
PLW1 9.30000019 W
F2 - Processing parameters
SI 65536
SF 600.1899948 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

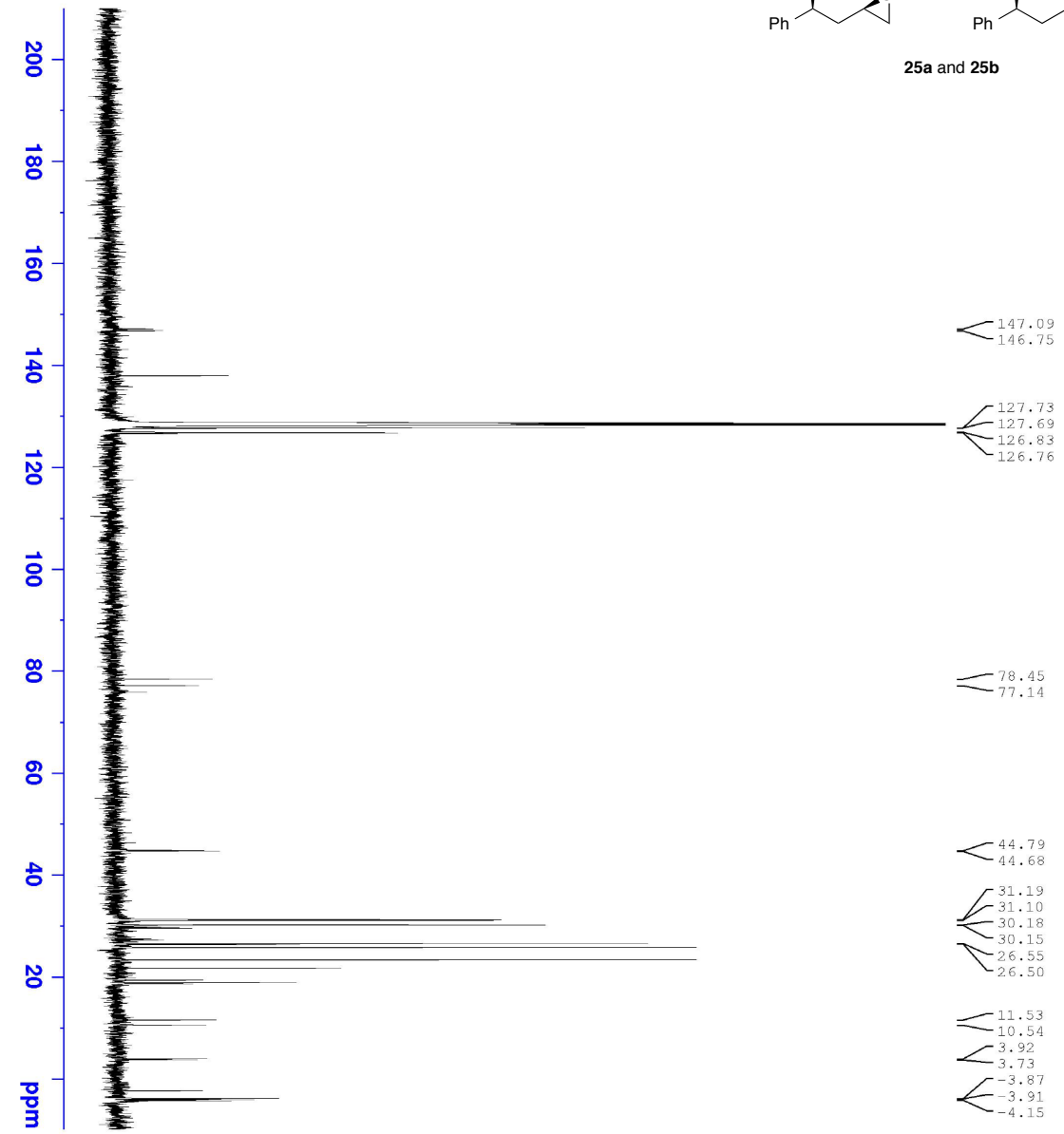



```
Current Data Parameters
NAME                  CR0376
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20130822
Time                   15.08
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG             zgpg30
TD                   65536
SOLVENT              C6D6
NS                     66
DS                      2
SWH              37593.984 Hz
FIDRES               0.573639 Hz
AQ                0.8716288 sec
RG                   9195.2
DW                  13.300 usec
DE                   6.50 usec
TE                  298.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.41 usec
PL1                    2.00 dB
PL1W            49.29017639 W
SFO1           125.78771611 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   -3.00 dB
PL12                  15.31 dB
PL13                  18.31 dB
PL2W            37.58904266 W
PL12W            0.55470389 W
PL13W            0.27801058 W
SFO2           500.20100008 MHz

F2 - Processing parameters
SI                     32768
SF             125.77750479 MHz
WDW                       EM
SSB                        0
LB                      2.00 Hz
GB                         0
PC                      1.40
```

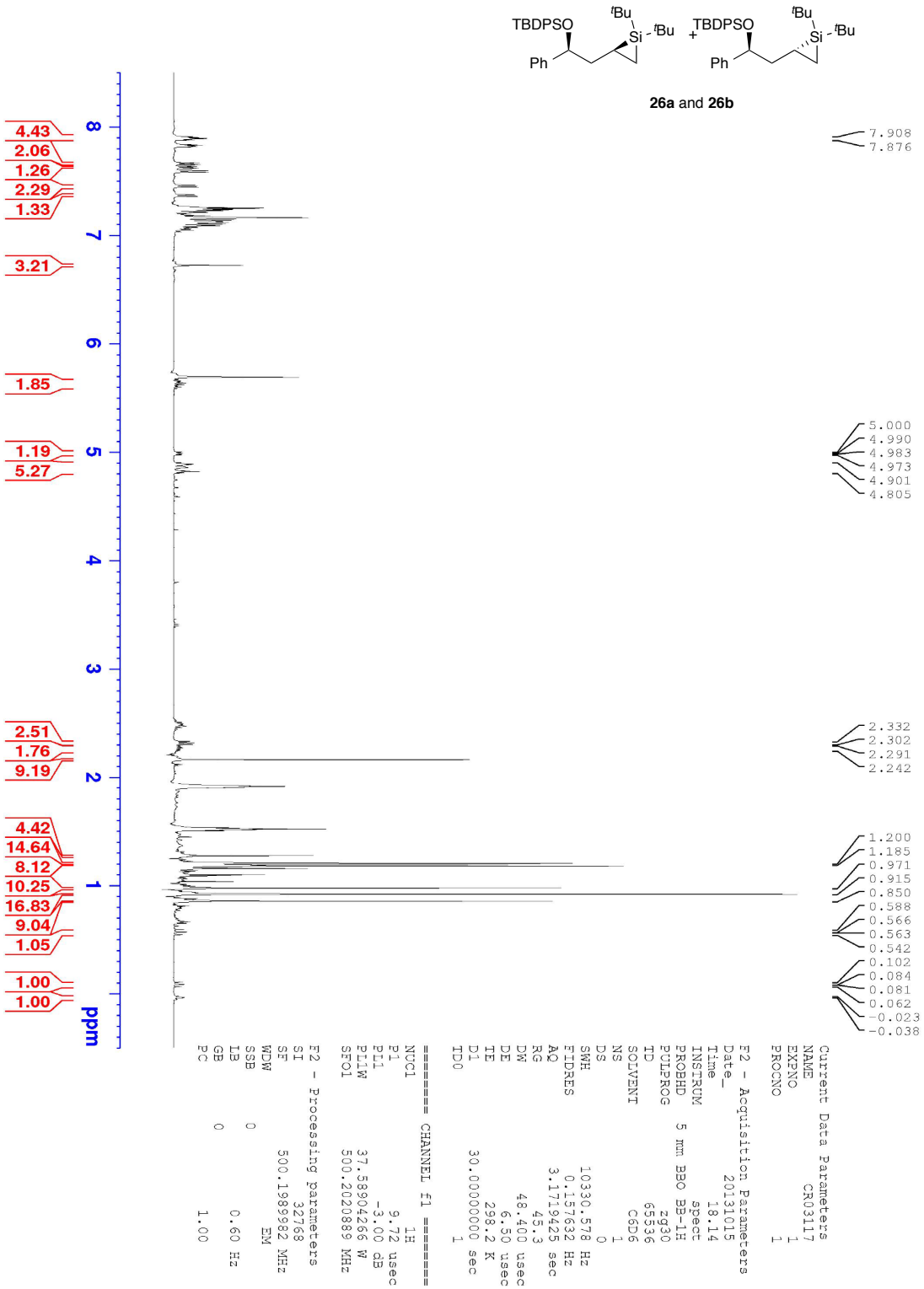

OTBS
tBu
Si
tBu
Ph
+
OTBS
tBu
Si
tBu
Ph
25a and 25b
7.445
7.430
7.416
7.401
7.209
7.194
7.179
7.101
7.065
1.176
1.133
1.078
1.058
1.038
1.028
1.016
0.905
0.829
0.166
0.156
-0.021
-0.041
3.99
4.02
2.26
2.07
0.17
0.15
2.00
0.19
2.13
6.70
2.14
2.89
2.62
9.34
7.97
7.82
9.27
17.71
2.98
2.06
2.59
3.02
2.64
2.66
ppm
Current Data Parameters
NAME CR03126
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131025
Time 11.56
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 45.3
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 30.0000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 9.72 usec
PL1 -3.00 dB
PL1W 37.58904266 W
SFO1 500.2020889 MHz
F2 - Processing parameters
SI 32768
SF 500.1989984 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

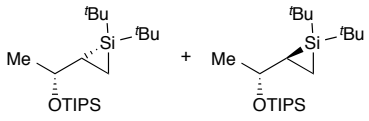


25a and **25b**

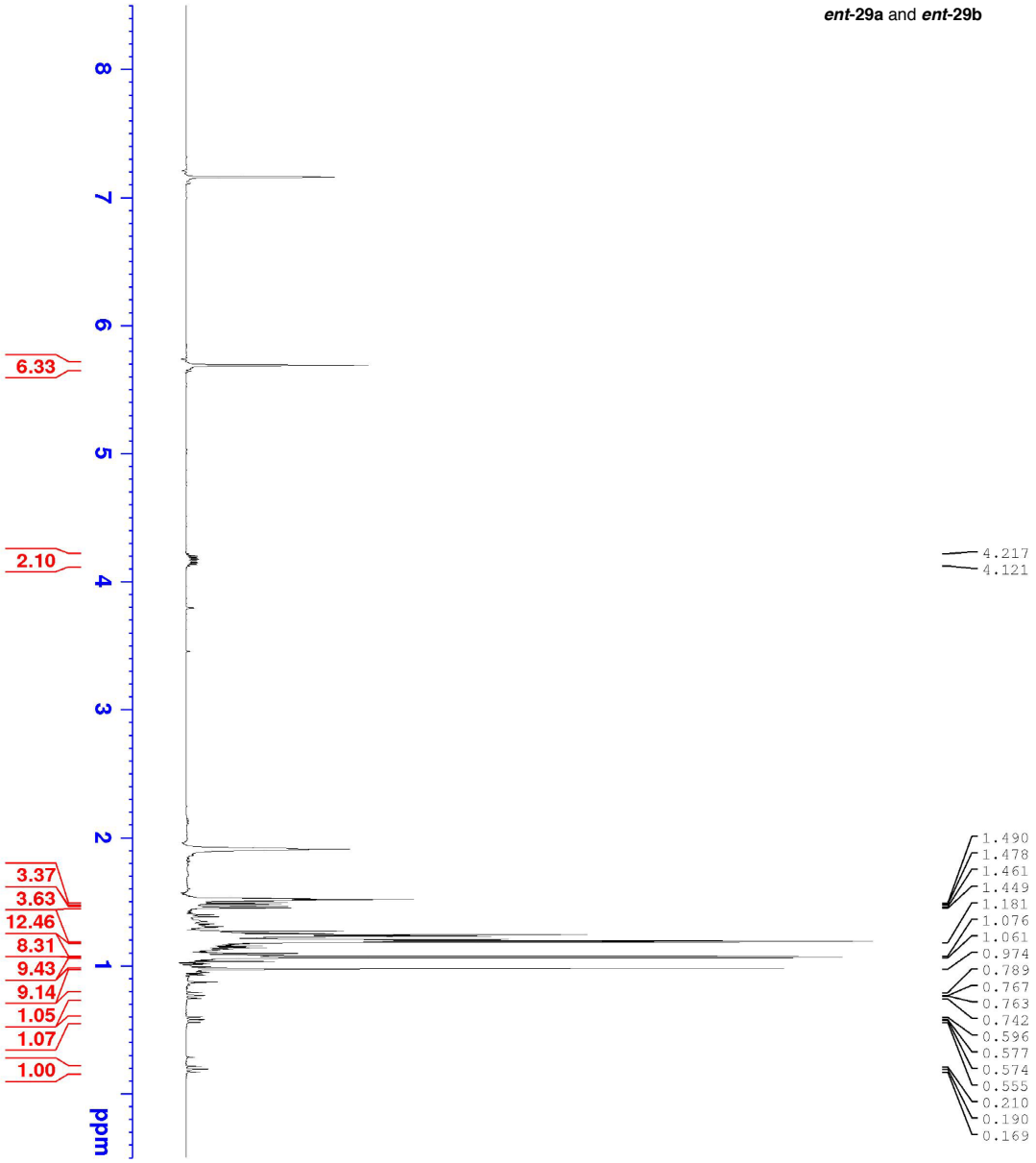



```
Current Data Parameters
NAME                  CR03126
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20131025
Time                   12.00
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG             zgpg30
TD                   65536
SOLVENT                CDCl3
NS                       256
DS                         2
SWH                30030.029 Hz
FIDRES                0.458222 Hz
AQ                    1.0911744 sec
RG                         8192
DW                       16.650 usec
DE                        6.50 usec
TE                      298.3 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                       13C
P1                       10.41 usec
PL1                       2.00 dB
PL1W                49.29017639 W
SFO1               125.7877161 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                      -3.00 dB
PL12                     15.31 dB
PL13                     18.31 dB
PL2W                37.58904266 W
PL12W                0.55470389 W
PL13W                0.27801058 W
SFO2               500.2010008 MHz

F2 - Processing parameters
SI                         32768
SF                 125.7750480 MHz
WDW                        EM
SSB                         0
LB                       2.00 Hz
GB                          0
PC                       1.40
```

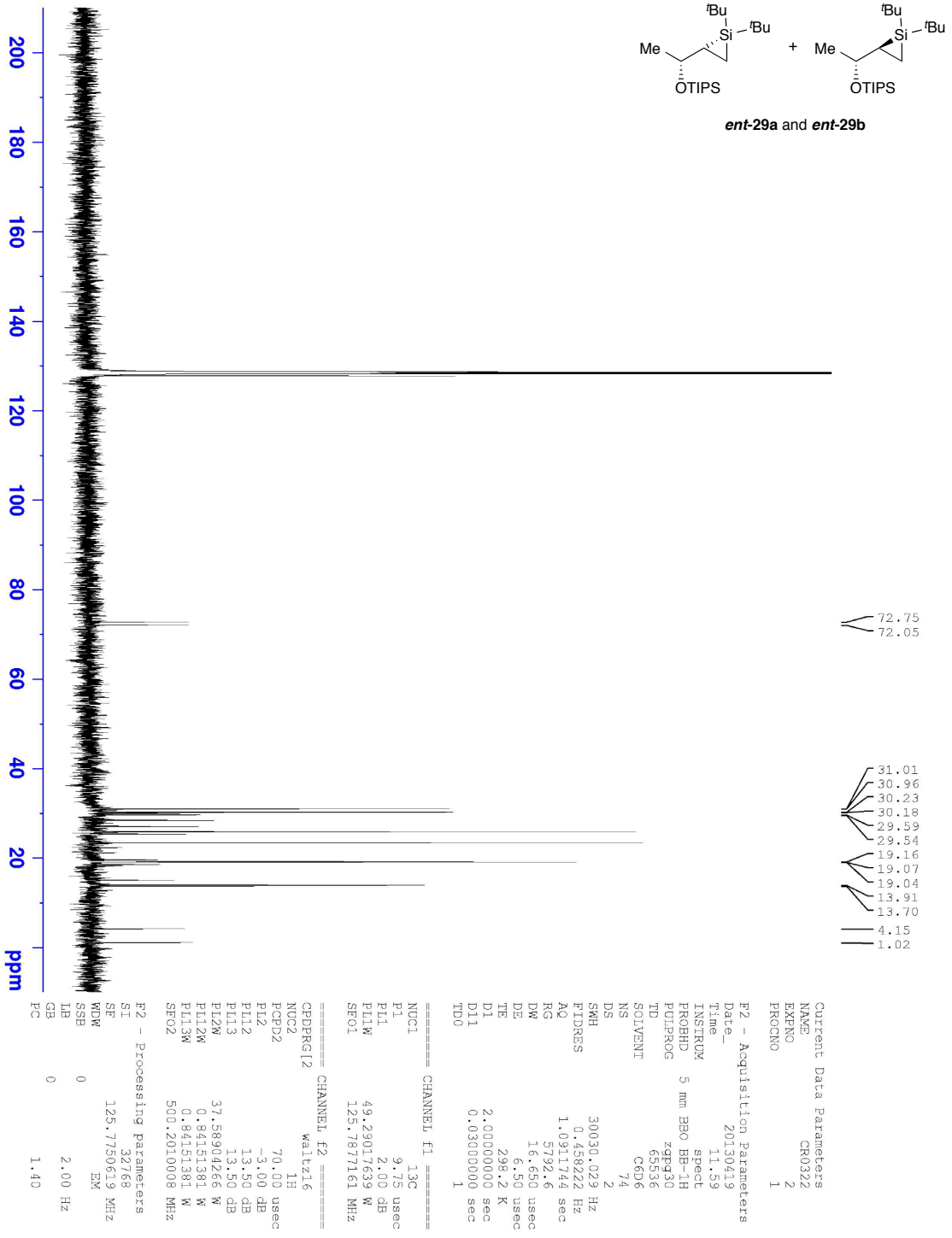

TBDPSO
tBu
Si
tBu
Ph
+
26a and 26b
4.43
2.06
1.26
2.29
1.33
3.21
1.85
1.19
5.27
2.51
1.76
9.19
4.42
14.64
8.12
10.25
16.83
9.04
1.05
1.00
1.00
ppm
7.908
7.876
5.000
4.990
4.983
4.973
4.901
4.805
2.332
2.302
2.291
2.242
1.200
1.185
0.971
0.915
0.850
0.588
0.566
0.563
0.542
0.102
0.084
0.081
0.062
-0.023
-0.038
Current Data Parameters
NAME CR03117
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131015
Time 18.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 45.3
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 30.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.72 usec
PL1 -3.00 dB
PL1W 37.58904266 W
SFO1 500.2020889 MHz
F2 - Processing parameters
SI 32768
SF 500.1989982 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

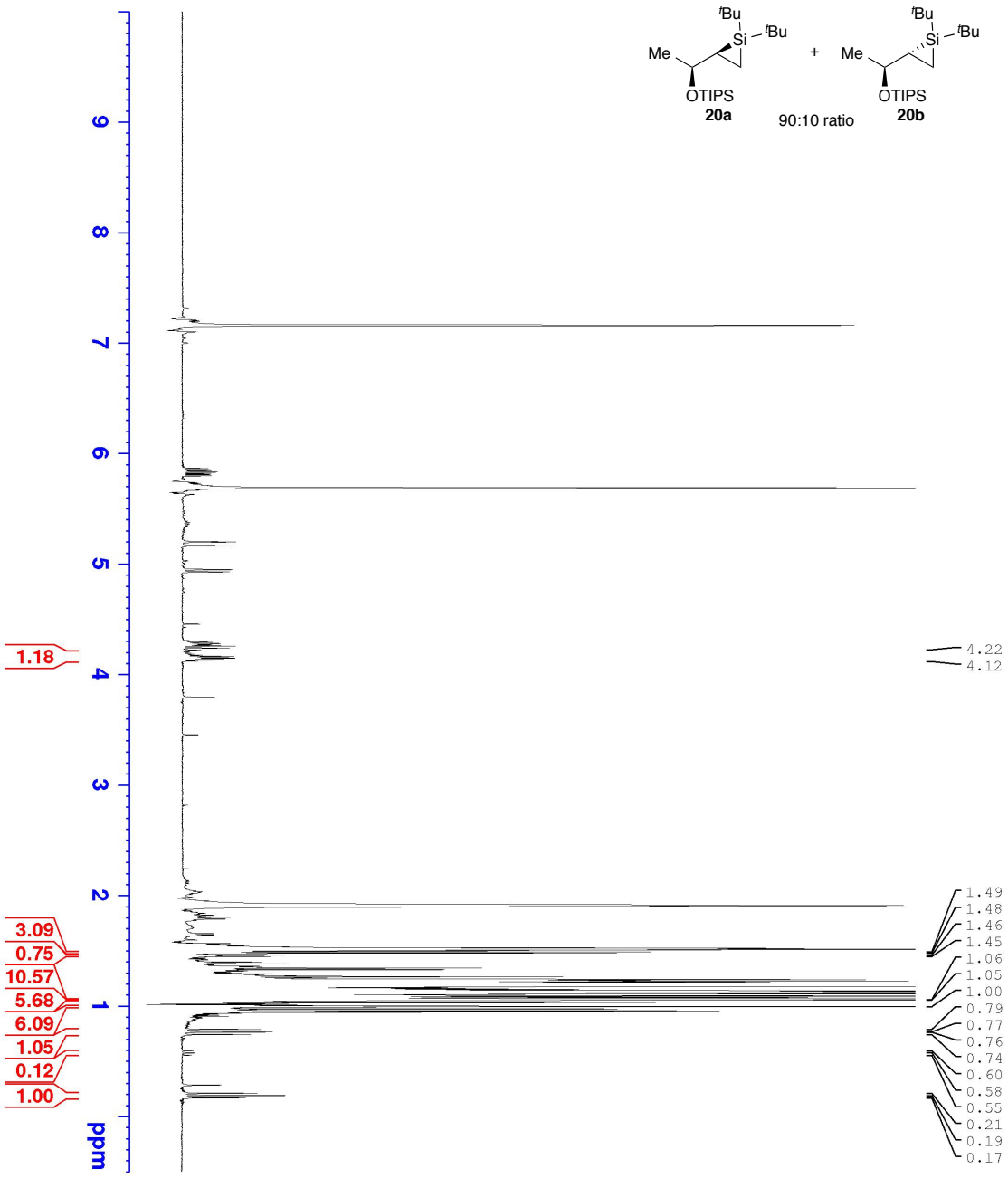


***ent*-29a** and ***ent*-29b**




```
Current Data Parameters
NAME                  CRO322
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20130419
Time               11.54
INSTRUM            spect
PROBHD     5 mm BBO BB-1H
PULPROG            zg30
TD                 65536
SOLVENT            C6D6
NS                 1
DS                 0
SWH          10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719425 sec
RG                 40.3
DW                 48.400 usec
DE                 6.50 usec
TE                 298.2 K
D1            2.00000000 sec
TD0                1

======== CHANNEL f1 ========
NUC1               1H
P1                 9.20 usec
PL1               -3.00 dB
PL1W      37.58904266 W
SFO1      500.2020889 MHz

F2 - Processing parameters
SI                 32768
SF            500.1990581 MHz
WDW                EM
SSB                0
LB                 0.60 Hz
GB                 0
PC                 1.00
```


tBu
Si
tBu
Me
OTIPS
+
Me
OTIPS
ent-29a and ent-29b
72.75
72.05
31.01
30.96
30.23
30.18
29.59
29.54
19.16
19.07
19.04
13.91
13.70
4.15
1.02
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME CR0322
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130419
Time_ 11.59
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 74
DS 2
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 5792.6
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.75 usec
PL1 2.00 dB
PL1W 49.29017639 W
SFO1 125.7877161 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.50 dB
PL13 13.50 dB
PL2W 37.58904266 W
PL12W 0.84151381 W
PL13W 0.84151381 W
SFO2 500.2010008 MHz
F2 - Processing parameters
SI 32768
SF 125.7750619 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40


tBu
Si
tBu
Me
OTIPS
20a
+
90:10 ratio
20b
1.18
3.09
0.75
10.57
5.68
6.09
1.05
0.12
1.00
ppm
4.22
4.12
1.49
1.48
1.46
1.45
1.06
1.05
1.00
0.79
0.77
0.76
0.74
0.60
0.58
0.55
0.21
0.19
0.17
Current Data Parameters
NAME CR0323
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130420
Time 14.49
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.17194425 sec
RG 45.3
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.20 usec
PL1 -3.00 dB
PL1W 37.58904266 W
SFO1 500.2020889 MHz
F2 - Processing parameters
SI 32768
SF 500.1990583 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00