Electronic Supplementary Material (ESI) for Chemical Communications

Electric Supporting information for

Unexpected formation of stannolanes and trigonal bipyramidal tin complexes by radical cyclization reaction

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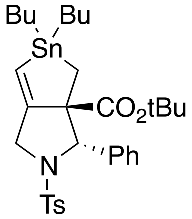
General

All ^{1}H and ^{13}C NMR spectra were recorded on JEOL JNM-ECA500 Delta2 (500 MHz for ^{1}H, 125 MHz for ^{13}C, and 186 MHz for ^{119}Sn) spectrometer. All the reactions in this paper were performed under nitrogen atmosphere unless otherwise mentioned. CH_2Cl_2 was dried over CaH_2, and distilled under nitrogen before use. Dry THF was purchased from Kanto Kagaku Co. Ltd. High resolution mass spectra (HRMS) were measured at Integrated Center for Sciences, Ehime University, Matsuyama, Japan.

Preparation of (*3S,3aS*)-methyl 5,5-dibutyl-3-(p-tolyl)-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2a): A mixture of (*S*)-methyl 2-((4-methyl-*N*-(prop-2-yn-1-yl)phenylsulfon-amido)(*p*-tolyl)methyl)acrylate (**1a**) (256.1 mg, 0.64 mmol), Bu_3SnH (0.21 mL, 0.77 mmol), and Et_3B in hexane solution (1.0 M, 0.77 mL) in toluene (64 mL) was placed in 100 mL two necked flask and purged by air at room temperature. After stirring for 1 h at room temperature, sat NH_4Claq (10 mL) was added. The organic phase was separated and aqueous phase was extracted with EtOAc (3 × 30 mL). The organic phases were combined, washed with brine (10 mL), and dried over Na_2SO_4. After filtration, the filtrate was concentrated. The residue was purified by flash chromatography (silica gel/hexane:EtOAc 50:1, 20:1, and 10:1) to give **2a** in 80% yield (320.7 mg, 80%). White solid, mp 68–69 ºC; $[\alpha]_D$ +17.1 (c 1.01, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 63.8 min ((*R*)-**2a**), t_R 79.5 min ((*S*)-**2a)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 80/20, 1.0mL/min] as >99%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.3 Hz, 2 H), 7.14 (d, *J* = 8.0 Hz, 2 H), 6.99 (d, *J* = 7.9 Hz, 2 H), 6.88 (d, *J* = 7.4 Hz, 2 H), 6.61 (s, 1 H, J^{119}Sn–^{1}H = 113.4 Hz), 5.38 (s, 1 H), 4.13 (dd, *J* = 13.3, 2.1 Hz, 1 H), 4.08 (dd, *J* = 13.3, 1.3 Hz, 1 H), 3.44 (s, 3 H), 2.36 (s, 3

H), 2.28 (s, 3 H), 1.49 – 1.02 (m, 12 H), 0.90 (d, *J* = 13.2 Hz, 1 H), 0.83 (t, *J* = 7.3 Hz, 3 H), 0.74 (t, *J* = 7.3 Hz, 3 H), 0.26 (d, *J* = 13.2 Hz, 1 H, J^{119}Sn–^{1}H = 54.3 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.2 (s), 157.8 (d, J^{13}C–^{119}Sn = 52.2 Hz), 142.7 (s), 137.2 (s), 136.8 (s), 136.2 (s), 129.2 (s), 128.9 (s), 127.9 (s), 127.5 (s), 69.0 (d, J^{13}C –^{119}Sn = 15.3 Hz), 68.5 (d, J^{13}C–^{119}Sn = 32.2 Hz), 52.7 (s), 50.6 (d, J^{13}C–^{119}Sn = 60.2 Hz), 29.0 (d, J^{13}C–^{119}Sn = 22.4 Hz), 28.8 (d, J^{13}C–^{119}Sn = 22.9 Hz), 27.1 (d, J^{13}C–^{119}Sn = 56.7 Hz), 27.0 (d, J^{13}C–^{119}Sn = 55.9 Hz), 21.5 (s), 21.2 (s), 13.73 (s), 13.73 (s), 13.67 (d, J^{13}C–^{117}Sn = 297.4 Hz, J^{13}C–^{119}Sn = 309.8 Hz), 13.0 (d, J^{13}C–^{117}Sn = 332.0 Hz, J^{13}C–^{119}Sn = 350.8 Hz), 12.4 (d, J^{13}C–^{117}Sn = 322.4Hz, J^{13}C–^{119}Sn = 337.2 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 141.6; IR (neat) 2920, 1726, 1614, 1344, 1159, 908 cm^{-1}; HRMS (FAB M+1) m/z 632.1856. Calcd for $C_{30}H_{42}NO_4SSn$ m/z 632.1856.

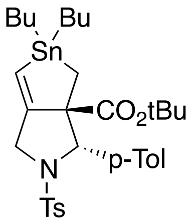
(3*S*,3a*S*)-*tert*-butyl 5,5-dibutyl-3-phenyl-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2b):



Colorless oil; $[\alpha]_D$ +11.3 (c 1.01, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 17.8 min ((*R*)-**2b**), t_R 34.7 min ((*S*)-**2b)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 88/12, 1.0mL/min] as >99%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.3 Hz, 2 H), 7.13 (d, *J* = 8.1 Hz, 2 H), 7.18 – 7.12 (m, 3 H), 6.90 (br, 2 H), 6.68 (s, 1 H, J^{119}Sn–^{1}H = 115.4 Hz), 5.42 (s, 1 H), 4.12 (dd, *J* = 12.9, 2.0 Hz, 1 H), 4.02 (d, *J* = 13.0 Hz, 1 H), 2.36 (s, 3 H), 1.36 (s, 9 H), 1.54 – 1.01 (m, 12 H), 0.92 (d, *J* = 13.3 Hz, 1 H), 0.85 (t, *J* = 7.3 Hz, 3 H), 0.74 (t, *J*

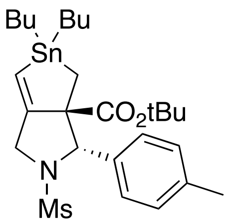
= 7.0 Hz, 3 H), 0.23 (d, *J* = 13.2 Hz, 1 H, J^{119}Sn–^{1}H = 53.4 Hz); ^{13}C NMR ($CDCl_3$, 126 MHz) δ 173.1, 158.2 (J^{119}Sn–^{13}C = 54.3 Hz), 142.6, 138.2, 136.0, 132.9, 128.9, 128.2, 127.9, 127.8, 126.9, 81.20, 69.47 (J^{119}Sn–^{13}C = 16.1 Hz), 68.4 (J^{119}Sn–^{13}C = 30.5 Hz), 50.4 (J^{119}Sn–^{13}C = 63.2 Hz), 28.7 (J^{119}Sn–^{13}C = 22.1 Hz), 28.4 (J^{119}Sn–^{13}C = 26.1 Hz), 27.5, 26.8 (J^{119}Sn–^{13}C = 56.4 Hz), 26.7 (J^{119}Sn–^{13}C = 58.2 Hz), 21.2 ,13.42 ,13.37, 12.8 (J^{117}Sn–^{13}C = 285.0 Hz, J^{119}Sn–^{13}C = 304.1 Hz), 12.6 (J^{117}Sn–^{13}C = 278.3 Hz, J^{119}Sn–^{13}C = 293.5 Hz), 12.0 (J^{117}Sn–^{13}C = 320.0 Hz, J^{119}Sn–^{13}C = 337.2 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 142.2; IR (neat) 2922, 1717, 1616, 1343, 1161, 908 cm^{-1}; HRMS (FAB^+ M+1) *m/z* 660.2185. calcd for $C_{32}H_{46}NO_4SSn$ 660.2170.

(3*S*,3a*S*)-*tert*-butyl 5,5-dibutyl-3-(*p*-tolyl)-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2c)



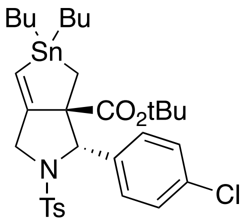
Pale yellow oil; $[\alpha]_D$ -1.1 (c 1.06, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 23.2 min ((*R*)-**2c**), t_R 47.7 min ((*S*)-**2c)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 88/12, 1.0mL/min] as 96%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.3 Hz, 2 H), 7.08 (d, *J* = 8.1 Hz, 2 H), 6.95 (d, *J* = 7.6 Hz, 2 H), 6.84 (br, 2 H), 6.56 (s, 1 H, J^{119}Sn–^{1}H = 115.4 Hz), 5.41 (s, 1 H), 4.14 (dd, *J* = 12.9, 2.1 Hz, 1 H), 4.01 (d, *J* = 12.9 Hz, 1 H), 2.34 (s, 3 H), 2.28 (s, 3 H), 1.37 (s, 9 H), 1.54 – 1.00 (m, 12 H), 0.93 (d, *J* = 11.9 Hz, 1 H), 0.85 (t, *J* = 7.3 Hz, 3 H), 0.75 (t, *J* = 7.0 Hz, 3 H), 0.27 (d, *J* = 13.2 Hz, 1 H, J^{119}Sn–^{1}H = 53.2 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.7, 158.3 (J^{119}Sn–^{13}C = 55.9 Hz), 142.4, 136.8, 136.4, 128.9, 128.9, 128.5, 127.8, 127.6, 127.1, 81.2, 69.4 (J^{119}Sn–^{13}C = 16.3 Hz), 68.2 (J^{119}Sn–^{13}C = 30.4 Hz), 50.4 (J^{119}Sn–^{13}C = 60.4 Hz), 28.7, 28.4 (J^{119}Sn–^{13}C = 22.7 Hz), 27.4, 26.8 (J^{119}Sn–^{13}C = 55.7 Hz), 26.7 (J^{119}Sn–^{13}C = 56.4 Hz), 21.1, 20.8, 13.4, 13.3, 12.7 (J^{117}Sn–^{13}C = 330.4 Hz, J^{119}Sn–^{13}C = 345.6 Hz), 12.5 (J^{117}Sn–^{13}C = 284.6 Hz, J^{119}Sn–^{13}C = 309.4 Hz), 11.9 (d, J^{117}Sn–^{13}C = 319.6 Hz, J^{119}Sn–^{13}C = 336.6 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 140.7; IR (neat) 2922, 1717, 1616, 1342, 1161, 909 cm^{-1}; HRMS (FAB^+ M+1) *m/z* 674.2338. calcd for $C_{33}H_{48}NO_4SSn$ 674.2326.

(3*S*,3a*S*)-tert-butyl 5,5-dibutyl-2-(methylsulfonyl)-3-(p-tolyl)-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2d)



Colorless oil; $[\alpha]_D$ +10.6 (c 1.10, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 22.1 min ((*R*)-**2d**), t_R 50.5 min ((*S*)-**2d)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 88/12, 1.0mL/min] as 96%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.07 (d, *J* = 8.0 Hz, 2 H), 6.99 (d, *J* = 7.7 Hz, 2 H), 6.64 (s, 1 H, J^{119}Sn–^{1}H = 115.6 Hz), 5.30 (s, 1 H), 4.20 (dd, *J* = 13.0, 1.8 Hz, 1 H), 4.11 (d, *J* = 13.1 Hz, 1 H), 2.62 (s, 3 H), 2.29 (s, 3 H), 1.65 – 1.05 (m, 12 H), 1.47 (s, 9 H), 0.97 (d, *J* = 13.2 Hz, 1 H), 0.86 (t, *J* = 7.3 Hz, 3 H), 0.78 (t, *J* = 7.0 Hz, 3 H), 0.28 (d, *J* = 13.2 Hz, 1 H, J^{119}Sn–^{1}H = 53.4 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.1, 158.3 (J^{119}Sn–^{13}C = 55.9 Hz), 137.5, 136.9, 129.1, 128.4 (J^{117}Sn–^{13}C = 335.2 Hz, J^{119}Sn–^{13}C = 350.6 Hz), 81.8 (J^{119}Sn–^{13}C = 39.5 Hz), 69.6 (J^{119}Sn–^{13}C = 14.9 Hz), 68.0 (J^{119}Sn–^{13}C = 30.7 Hz), 50.8 (J^{119}Sn–^{13}C = 61.3 Hz), 37.9, 29.1 (J^{119}Sn–^{13}C = 22.2 Hz), 28.8 (J ^{119}Sn–^{13}C = 22.6 Hz), 27.9, 27.1 (J ^{119}Sn–^{13}C = 56.9 Hz), 27.0 (J ^{119}Sn–^{13}C = 56.6 Hz), 21.2, 13.7, 13.6, 13.1 (J ^{117}Sn–^{13}C = 332.0 Hz, J ^{119}Sn–^{13}C = 347.2 Hz), 12.6 (J ^{117}Sn–^{13}C = 290.8 Hz, J ^{119}Sn–^{13}C = 306.5 Hz), 12.4 (J ^{117}Sn–^{13}C = 322.5 Hz, J ^{119}Sn–^{13}C = 339.0 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 144.8; IR (neat) 1714, 1337, 1155 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 598.2013 calcd for $C_{27}H_{44}NO_4SSn$ 598.2013.

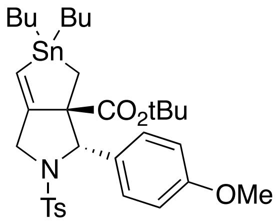
(3*S*,3a*S*)-*tert*-butyl 5,5-dibutyl-3-(4-chlorophenyl)-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2e)



Pale yellow oil; $[\alpha]_D$ -0.8 (c 1.05, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 13.3 min ((*R*)-**2e**), t_R 19.5 min ((*S*)-**2e)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 88/12, 1.0mL/min] as >99%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, J = 8.3 Hz, 2 H), 7.13 (d, J = 8.1 Hz, 4 H), 6.90 (br, 2 H), 6.58 (s, 1 H, J ^{119}Sn–^{1}H = 115.4 Hz), 5.42 (s, 1 H), 4.12 (dd, J = 12.9, 2.0 Hz, 1 H), 4.02 (d, J = 13.0 Hz, 1 H), 2.36 (s, 3 H), 1.36 (s, 9 H), 1.54 – 1.00 (m, 12 H), 0.90 (d, J = 13.2 Hz, 1 H), 0.85 (t, J = 7.3 Hz, 3 H), 0.77 (t, J = 7.0 Hz, 3 H), 0.19 (d, J = 14.2 Hz, 1 H, J ^{119}Sn–^{1}H = 53.3 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.2, 157.4 (J ^{119}Sn–^{13}C = 55.2 Hz), 142.6, 138.2, 136.0, 132.9, 129.1, 128.9, 128.2, 127.8, 126.9, 81.2, 69.1 (J ^{119}Sn–^{13}C = 17.0 Hz), 67.5 (J ^{119}Sn–^{13}C = 29.6 Hz), 50.1 (J ^{119}Sn–^{13}C = 60.2 Hz), 28.5 (J ^{119}Sn–^{13}C = 22.6 Hz), 28.3 (J ^{119}Sn–^{13}C = 23.1 Hz), 27.2, 26.6 (J ^{119}Sn–^{13}C = 57.7 Hz), 26.5 (J ^{119}Sn–^{13}C = 56.7 Hz), 21.0, 13.2, 13.1, 12.7 (J ^{117}Sn–^{13}C = 333.2 Hz, J ^{119}Sn–^{13}C = 348.6 Hz), 12.3 (J ^{117}Sn–^{13}C = 278.3 Hz, J ^{119}Sn–^{13}C = 280.8 Hz), 11.9

(J^{117}Sn–^{13}C = 316.4 Hz, J^{119}Sn–^{13}C = 328.5 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 140.1; IR (neat) 2922, 1717, 1616, 1343, 1161, 908 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 694.1771. calcd for $C_{32}H_{45}ClNO_4SSn$ 694.1780.

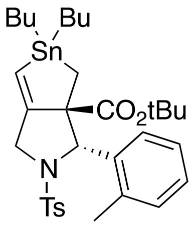
(3*S*,3a*S*)-*tert*-butyl 5,5-dibutyl-3-(4-methoxyphenyl)-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2f)



Colorless oil; $[\alpha]_D$ +1.4 (c 1.00, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 23.7 min ((*R*)-**2f**), t_R 46.6 min ((*S*)-**2f)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 85/15, 1.0mL/min] as 95%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.3 Hz, 2 H), 7.09 (d, *J* = 8.2 Hz, 2 H), 6.87 (br, 2 H), 6.68 (d, *J* = 8.3 Hz, 2 H), 6.56 (s, 1 H, J^{119}Sn–^{1}H = 115.4 Hz), 5.41 (s, 1 H), 4.15 (dd, *J* = 13.0, 2.1 Hz, 1 H), 3.99 (d, *J* = 13.8 Hz, 1 H), 2.33 (s, 3 H), 3.76 (s, 3 H), 1.38 (s, 9 H), 1.54 – 1.04 (m, 12 H), 0.91 (d, *J* = 13.3 Hz, 1 H), 0.85 (t, *J* = 7.3 Hz, 3 H), 0.76 (t, *J* = 7.1 Hz, 3 H), 0.28 (d, *J* = 12.7 Hz, 1 H, J^{119}Sn–^{1}H = 53.2 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.7, 158.3 (J^{119}Sn–^{13}C = 55.2 Hz), 158.9, 142.4, 136.4, 131.6, 129.0, 128.9, 127.7, 127.1, 113.2, 81.3, 69.5 (J^{119}Sn–^{13}C = 16.4 Hz), 68.0 (J^{119}Sn–^{13}C = 31.9 Hz), 55.0, 50.3 (J^{119}Sn–^{13}C = 61.4 Hz), 28.7 (J^{119}Sn–^{13}C = 21.8 Hz), 28.5 (J^{119}Sn–^{13}C = 22.8 Hz), 27.4, 26.8 (J^{119}Sn–^{13}C = 57.0 Hz), 26.7 (J^{119}Sn–^{13}C = 56.7 Hz), 21.2 ,13.4 ,13.3, 12.8 (J^{117}Sn–^{13}C = 330.2 Hz, J^{119}Sn–^{13}C = 346.0 Hz), 12.5 (J^{117}Sn–^{13}C = 261.2 Hz, J^{119}Sn–^{13}C = 296.2 Hz), 12.0 (J^{117}Sn–^{13}C = 319.2 Hz, J^{119}Sn–^{13}C = 336.8 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 140.0; IR (neat)

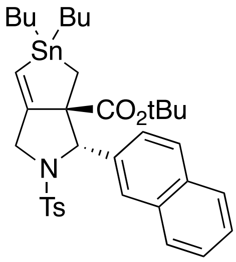
2930, 1719, 1612, 1343, 1247, 1161, 1098, 910 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 690.2261. calcd for $C_{33}H_{48}NO_5SSn$ 690.2275.

(3*S*,3a*S*)-*tert*-butyl 5,5-dibutyl-3-(*o*-tolyl)-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2g)



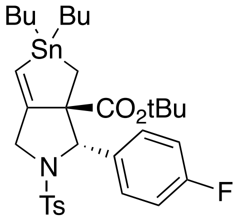
Colorless oil; $[\alpha]_D$ +16.9 (c 1.00, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 17.4 min ((*R*)-**2g**), t_R 19.1 min ((*S*)-**2g)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 88/12, 1.0mL/min] as 98%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.2 Hz, 2 H), 7.12 (d, *J* = 8.3 Hz, 2 H), 7.10 (d, *J* = 8.9 Hz, 1 H), 7.05 (td, *J* = 7.4, 1.1 Hz, 1 H, 6.87 (t, *J* = 7.3 Hz, 1 H), 6.60 (d, *J* = 7.9 Hz, 1 H), 6.58 (s, 1 H, J^{119}Sn–^{1}H = 115.4 Hz), 5.83 (s, 1 H), 4.17 (dd, *J* = 12.8, 2.2 Hz, 1 H), 4.06 (dd, *J* = 12.9, 0.9 Hz, 1 H), 2.45 (s, 3 H), 2.34 (s, 3 H), 1.37 (s, 9 H), 1.55 – 1.02 (m, 12 H), 0.95 (d, *J* = 13.2 Hz, 1 H), 0.84 (t, *J* = 7.3 Hz, 3 H), 0.73 (t, *J* = 7.0 Hz, 3 H), 0.18 (d, *J* = 13.2 Hz, 1 H, J^{119}Sn–^{1}H = 54.2 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.9, 158.0 (J^{119}Sn–^{13}C = 55.6 Hz), 142.6, 138.0, 136.3, 136.2, 129.7, 129.0, 128.1, 127.2, 127.0, 126.5, 125.8, 81.3, 69.4 (J^{119}Sn–^{13}C = 17.0 Hz), 64.1 (J^{119}Sn–^{13}C = 31.0 Hz), 50.6 (J^{119}Sn–^{13}C = 60.7 Hz), 28.8 (J^{119}Sn–^{13}C = 22.0 Hz), 28.3 (J^{119}Sn–^{13}C = 22.8 Hz), 27.5, 26.9 (J^{119}Sn–^{13}C = 55.3 Hz), 26.7 (J^{119}Sn–^{13}C = 55.2 Hz), 21.2, 19.7, 13.4, 13.3, 12.7 (J^{117}Sn–^{13}C = 329.4 Hz, J^{119}Sn–^{13}C = 345.0 Hz), 12.1 (J^{117}Sn–^{13}C = 266.8 Hz, J^{119}Sn–^{13}C = 281.8 Hz), 11.9 (J^{117}Sn–^{13}C = 324.0 Hz, J^{119}Sn–^{13}C = 340.0 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 139.3; IR (neat) 2924, 1717, 1616, 1341, 1159, 908 cm^{-1}; HRMS (FAB^{+} M+1) *m/z* 674.2320. calcd for $C_{33}H_{48}NO_4SSn$ 674.2326.

(3*S*,3a*S*)-*tert*-butyl 5,5-dibutyl-3-(naphthalen-2-yl)-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2h)



Pale yellow oil; $[\alpha]_D$ –21.9 (c 1.03, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 20.9 min ((*R*)-**2h**), t_R 30.3 min ((*S*)-**2h)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 88/12, 1.0mL/min] as 96%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.2 Hz, 2 H), 7.82 – 7.39 (m, 6 H), 7.06 – 6.93 (m, 1 H), 6.90 (d, *J* = 8.0 Hz, 2 H), 6.63 (s, 1 H, J^{119}Sn–^{1}H = 114.6 Hz), 5.61 (s, 1 H), 4.26 (dd, *J* = 13.0, 2.1 Hz, 1 H), 4.12 (d, *J* = 13.5 Hz, 1 H), 2.20 (s, 3 H), 1.42 (s, 9 H), 1.56 – 1.02 (m, 12 H), 0.96 (d, *J* = 13.3 Hz, 1 H), 0.83 (t, *J* = 7.3 Hz, 3 H), 0.64 – 0.50 (m, 3 H), 0.25 (d, *J* = 13.3 Hz, 1 H, J^{119}Sn–^{1}H = 53.2 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.8, 158.5 (J^{119}Sn–^{13}C = 54.6 Hz), 142.6, 136.9, 136.5, 133.1, 132.9, 129.5, 129.0, 128.2, 128.1, 128.0, 127.8, 127.5, 127.2, 126.0, 125.9, 81.8, 69.8 (J^{119}Sn–^{13}C = 16.0 Hz), 68.9 (J^{119}Sn–^{13}C = 30.7 Hz), 50.9 (J^{119}Sn–^{13}C = 61.7 Hz), 29.0 (J^{119}Sn–^{13}C = 22.1 Hz), 28.7 (J^{119}Sn–^{13}C = 22.6 Hz), 27.8, 27.1 (J^{119}Sn–^{13}C = 56.3 Hz), 26.9 (J^{119}Sn–^{13}C = 59.5 Hz), 21.4, 17.7, 13.7, 13.4, 13.1 (J^{117}Sn–^{13}C = 331.6 Hz, J^{119}Sn–^{13}C = 347.1 Hz), 12.2 (J^{117}Sn–^{13}C = 319.2 Hz, J^{119}Sn–^{13}C = 346.6 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 157.2; IR (neat) 2924, 1716, 1344, 1251, 1139, 665 cm^{-1}; HRMS (FAB^{+} M+2) *m/z* 711.2394. calcd for $C_{36}H_{49}NO_4SSn$ 711.2404.

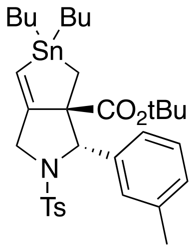
(3S,3aS)-tert-butyl 5,5-dibutyl-3-(4-fluorophenyl)-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2i)



Pale yellow oil; $[\alpha]_D$ +9.87 (c 1.02, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 13.7 min ((*R*)-**2i**), t_R 21.4 min ((*S*)-**2i)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 88/12, 1.0mL/min] as 92%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, J = 8.2 Hz, 2 H), 7.12 (d, J = 8.4 Hz, 2 H), 6.94 (s, 2 H), 6.85 (t, J = 8.4 Hz, 2H), 6.58 (s, 1 H, J^{119}Sn–^{1}H = 115.1 Hz), 5.45 (s, 1 H), 4.13 (dd, J = 12.9, 2.1 Hz, 1 H), 4.02 (d, J = 13.3 Hz, 1 H), 2.34 (s, 3 H), 1.56 – 1.03 (m, 12 H), 1.37 (s, 9 H), 0.91 (d, J = 13.1 Hz, 1 H), 0.85 (t, J = 7.3 Hz, 3 H), 0.76 (t, J = 7.0 Hz, 3 H), 0.20 (d, J = 13.3 Hz, 1 H, J^{119}Sn–^{1}H = 52.9 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.5, 163.1, 161.1, 157.8 (J^{119}Sn–^{13}C = 54.5 Hz), 142.7, 136.3, 135.6, 135.5, 129.1, 128.2, 127.2, 114.8 (J^{19}F–^{13}C = 21.4 Hz), 81.6, 69.5 (J^{119}Sn–^{13}C = 16.4 Hz), 67.7 (J^{119}Sn–^{13}C = 30.7 Hz), 50.4 (J^{119}Sn–^{13}C = 60.2 Hz), 28.9 (J^{119}Sn–^{13}C = 22.0 Hz), 28.6 (J^{119}Sn–^{13}C = 22.7 Hz), 27.6, 27.0 (J^{119}Sn–^{13}C = 51.6 Hz), 26.9 (d, J = 54.4 Hz), 21.3, 13.5, 13.4, 13.0 (J^{117}Sn–^{13}C = 330.0 Hz, J^{119}Sn–^{13}C = 346.0 Hz), 12.7 (J^{117}Sn–^{13}C = 270.2 Hz, J^{119}Sn–^{13}C = 292.9 Hz), 12.2 (J^{117}Sn–^{13}C = 322.4 Hz, J^{119}Sn–^{13}C = 338.0 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 140.6; IR (neat) 2924, 1716, 1508, 1344, 1161 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 678.2072. calcd for $C_{32}H_{45}FNO_4SSn$ 678.2075.

(3S,3aS)-tert-butyl 5,5-dibutyl-3-(m-tolyl)-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-car

boxylate (2j)

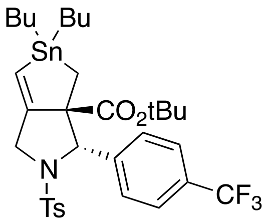


Pale yellow oil; $[\alpha]_D$ –1.84 (c 1.03, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 18.6 min ((*R*)-**2j**), t_R 33.9 min ((*S*)-**2j)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 88/12, 1.0mL/min] as 96%ee; ^{1}H NMR (500 MHz, $CDCl_3$) ™ 7.40 (d, J = 8.3 Hz, 2 H), 7.07 (d, J = 8.4 Hz, 2 H), 7.03 (d, J = 7.5 Hz, 1 H), 6.96 (d, J = 7.6 Hz, 1 H), 6.81 – 6.70 (m, 1 H), 6.67 – 6.58 (m, 1 H), 6.56 (s, 1 H, $^3J^{119}$Sn–^{1}H = 115.4 Hz), 5.41 (s, 1 H), 4.18 (dd, J = 12.9, 2.1 Hz, 1 H), 4.01 (dd, J = 12.9, 1.1 Hz, 1 H), 2.32 (s, 3 H), 2.17 (s, 3 H), 1.39 (s, 9 H), 1.54 – 1.04 (m, 12 H), 0.92 (d, J = 13.3 Hz, 1 H), 0.85 (t, J = 7.3 Hz, 3 H), 0.74 (t, J = 7.1 Hz, 3 H), 0.25 (d, J = 13.3 Hz, 1 H, J^{119}Sn–^{1}H = 53.2 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.7, 158.4 (J^{119}Sn–^{13}C = 55.5 Hz), 142.4, 139.14, 137.3, 136.5, 128.9, 128.1, 127.8, 127.7, 127.2, 81.4, 69.5 (J^{119}Sn–^{13}C = 16.3 Hz), 68.6 (J^{119}Sn–^{13}C = 31.4 Hz), 50.6 (J^{119}Sn–^{13}C = 61.7 Hz), 28.9 (J^{119}Sn–^{13}C = 22.1 Hz), 28.5 (J^{119}Sn–^{13}C = 22.7 Hz), 27.6, 27.0 (J^{119}Sn–^{13}C = 55.3 Hz), 26.8 (J^{119}Sn–^{13}C = 55.8 Hz), 21.3, 21.2, 13.5, 13.4, 12.9 (J^{117}Sn–^{13}C = 330.3 Hz, J^{119}Sn–^{13}C = 347.7 Hz), 12.7 (J^{117}Sn–^{13}C = 287.9 Hz, J^{119}Sn–^{13}C = 302.1 Hz), 12.1 (J^{117}Sn–^{13}C = 320.1 Hz, J^{119}Sn–^{13}C = 335.4 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 140.3; IR (neat) 2922, 1716, 1344, 1159 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 674.2314. calcd for $C_{33}H_{48}NO_4SSn$ 674.2326.

(3S,3aS)-tert-butyl

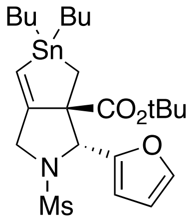
5,5-dibutyl-2-tosyl-3-(4-(trifluoromethyl)phenyl)-1,2,3,3a,4,5-hexahydrostannolo[3,

4-c]pyrrole-3a-carboxylate (2k)



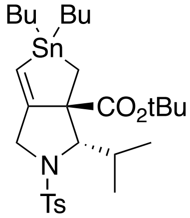
Pale yellow oil; $[\alpha]_D$ +2.24 (c 1.07, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 8.9 min ((*R*)-**2k**), t_R 10.3 min ((*S*)-**2k)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 88/12, 1.0mL/min] as 96%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, J = 8.3 Hz, 2 H), 7.40 (d, J = 7.9 Hz, 2 H), 7.09 (dd, J = 8.5, 0.5 Hz, 2 H), 7.11 – 7.03 (m, 2 H), 6.61 (s, 1 H, $^3J^{119}$Sn–^{1}H = 115.3 Hz), 5.49 (s, 1 H), 4.16 (dd, J = 13.0, 2.1 Hz, 1 H), 4.08 (dd, J = 12.9, 1.1 Hz, 1 H), 2.33 (s, 3 H), 1.37 (s, 9 H), 1.56 – 0.98 (m, 12 H), 0.92 (d, J = 13.3 Hz, 1 H), 0.85 (t, J = 7.3 Hz, 3 H), 0.72 (t, J = 7.1 Hz, 3 H), 0.12 (d, J = 13.3 Hz, 1 H, J^{119}Sn–^{1}H = 53.2 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.3, 157.5 (J^{119}Sn–^{13}C = 53.6 Hz), 143.8, 142.9, 136.2, 129.6 (q, J^{19}F–^{13}C = 32.3 Hz), 129.1, 128.7, 127.1, 124.9 (q, J^{19}F–^{13}C = 3.8 Hz), 81.8, 69.5 (J^{119}Sn–^{13}C = 16.7 Hz), 67.9 (J^{119}Sn–^{13}C = 29.9 Hz), 50.6 (J^{119}Sn–^{13}C = 54.5 Hz), 28.9 (J^{119}Sn–^{13}C = 22.2 Hz), 28.6 (J^{119}Sn–^{13}C = 23.0 Hz), 27.6, 27.0 (J^{119}Sn–^{13}C = 57.4 Hz), 26.7 (J^{119}Sn–^{13}C = 57.4 Hz), 21.3, 13.6, 13.3, 13.1 (J^{117}Sn–^{13}C = 301.6 Hz, J^{119}Sn–^{13}C = 336.5 Hz), 12.7 (J^{117}Sn–^{13}C = 259.0 Hz, J^{119}Sn–^{13}C = 278.4 Hz), 12.2 (J^{117}Sn–^{13}C = 334.1 Hz, J^{119}Sn–^{13}C = 349.5 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 141.1; IR (neat) 2926, 1716, 1508, 1344, 1161 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 728.2054. calcd for $C_{33}H_{45}F_3NO_4SSn$ 728.2043.

(3R,3aS)-tert-butyl 5,5-dibutyl-3-(furan-2-yl)-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2l)



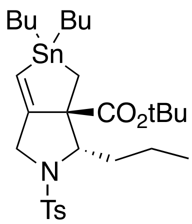
Pale yellow oil; $[\alpha]_D$ +16.9 (c 1.09, $CHCl_3$); the enantiomeric purity was determined by HPLC analysis, t_R 17.3 min ((*R*)-**2l**), t_R 23.4 min ((*S*)-**2l)** [CHIRALPAK IC (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 88/12, 1.0mL/min] as 87%ee; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, J = 8.3 Hz, 2 H), 7.10 (d, J = 8.5 Hz, 2 H), 7.02 (t, J = 1.0 Hz, 1 H), 6.49 (s, 1 H, $^3J^{119}$Sn–^{1}H = 116.0 Hz), 6.19 (d, J = 1.3 Hz, 2 H), 5.52 (s, 1 H), 4.18 (dd, J = 12.9, 2.1 Hz, 1 H), 3.86 (dd, J = 12.9, 1.3 Hz, 1 H), 2.34 (s, 3 H), 1.45 (s, 9 H), 1.57 – 1.09 (m, 12 H), 1.03 (d, J = 13.1 Hz, 1 H), 0.86 (t, J = 7.3 Hz, 3 H), 0.79 (t, J = 7.2 Hz, 3 H), 0.24 (d, J = 13.1 Hz, 1 H, J^{119}Sn–^{1}H = 52.8 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.1, 158.6 (J^{119}Sn–^{13}C = 52.2 Hz), 152.0, 142.4, 141.9, 136.2, 129.1, 127.0, 126.3, 109.9, 109.8, 81.8, 69.5 (J^{119}Sn–^{13}C = 17.8 Hz), 62.3 (J^{119}Sn–^{13}C = 34.1 Hz), 49.7 (J^{119}Sn–^{13}C = 62.8 Hz), 28.9 (J^{119}Sn–^{13}C = 19.9 Hz), 28.7 (J^{119}Sn–^{13}C = 22.5 Hz), 27.7, 27.0 (J^{119}Sn–^{13}C = 54.4 Hz), 26.9 (J^{119}Sn–^{13}C = 56.3 Hz), 21.4, 13.6 (J^{117}Sn–^{13}C = 289.1 Hz, J^{119}Sn–^{13}C = 309.9 Hz), 13.1 (J^{117}Sn–^{13}C = 333.6 Hz, J^{119}Sn–^{13}C = 349.2 Hz), 12.3 (J^{117}Sn–^{13}C = 321.5 Hz, J^{119}Sn–^{13}C = 334.8 Hz), 12.2; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 141.6; IR (neat) 2928, 1718, 1346, 1161 cm^{-1}; HRMS (FAB^+ M+1) *m/z* 650.1963. calcd for $C_{30}H_{44}NO_5SSn$ 650.1962.

(3*S*,3a*S*)-*tert*-butyl 5,5-dibutyl-3-isopropyl-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2m)



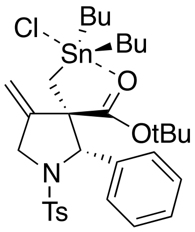
Colorless oil; $[\alpha]_D$ +9.9 (c 0.97, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.3 Hz, 2 H), 7.21 (d, *J* = 8.0 Hz, 2 H), 5.84 (s, 1 H), 4.40 (dd, *J* = 15.0, 1.9 Hz, 1 H), 4.08 (d, *J* = 15.2 Hz, 1 H), 3.34 (d, *J* = 9.1 Hz, 1 H), 2.38 (s, 3 H), 2.29 – 2.18 (m, 1H), 1.93 (d, *J* = 11.6 Hz, 1 H), 1.38 (s, 9 H), 1.66 – 1.09 (m, 12 H), 1.18 (d, *J* = 6.8 Hz, 3 H), 0.98 (d, *J* = 6.5 Hz, 3 H), 0.88 (t, *J* = 7.3 Hz, 3 H), 0.80 (t, *J* = 7.3 Hz, 3 H), 0.17 (d, *J* = 11.7 Hz, 1 H, J^{119}Sn–^{1}H = 54.7 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.3, 161.0 (J^{119}Sn–^{13}C = 39.6 Hz), 143.3, 135.5, 129.4, 128.5, 124.5 (J^{117}Sn–^{13}C = 310.6 Hz, J^{119}Sn–^{13}C = 324.6 Hz), 81.7, 79.3 (J^{119}Sn–^{13}C = 41.0 Hz), 68.8 (J^{119}Sn–^{13}C = 9.3 Hz), 55.0 (J^{119}Sn–^{13}C = 53.6 Hz), 31.3, 32.2, 29.1 (J^{119}Sn–^{13}C = 23.2 Hz), 28.9 (J^{119}Sn–^{13}C = 20.4 Hz), 27.9, 27.2 (J^{119}Sn–^{13}C = 51.0 Hz), 27.1 (d, J^{119}Sn–^{13}C = 58.9 Hz), 21.7, 20.4, 17.6 (J^{117}Sn–^{13}C = 321.6 Hz, J^{119}Sn–^{13}C = 336.2 Hz), 13.9 ,13.7, 12.8 (J^{117}Sn–^{13}C = 331.0 Hz, J^{119}Sn–^{13}C = 346.2 Hz), 12.4 (J^{117}Sn–^{13}C = 314.8 Hz, J^{119}Sn–^{13}C = 329.4 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 127.3; IR (neat) 2922, 1716, 1346, 1159 cm^{-1}; HRMS (FAB^+ M+2) *m/z* 627.2410. calcd for $C_{29}H_{49}NO_4SSn$ 627.2404.

(3S,3aS)-tert-butyl 5,5-dibutyl-3-propyl-2-tosyl-1,2,3,3a,4,5-hexahydrostannolo[3,4-c]pyrrole-3a-carboxylate (2n)



Colorless oil; isolated as an inseparable 1:1 mixture of the two diastereomer; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.3 Hz, 1 H), 7.67 (d, *J* = 8.3 Hz, 1 H), 7.25 (d, *J* = 8.8 Hz, 1 H), 7.23 (d, *J* = 8.8 Hz, 1 H), 6.40 (s, 0.5 H), 6.10 (s, 0.5 H), 4.48 (dd, *J* = 5.8, 4.5 Hz, 0.5 H), 4.40 (dd, *J* = 14.6, 2.1 Hz, 0.5 H), 4.07 (dd, *J* = 14.6, 1.0 Hz, 0.5 H), 3.89 (dd, *J* = 13.2, 2.2 Hz, 0.5 H), 3.77 (dd, *J* = 13.2, 1.2 Hz, 0.5 H), 3.33 (dd, *J* = 10.4, 4.5 Hz, 0.5 H), 2.38 (s, 1.5 H), 2.36 (s, 1.5 H), 2.26–2.17 (m, 2 H), 1.40 (s, 4.5 H), 1.25 (s, 4.5 H), 1.67 – 0.96 (m, 15 H), 0.93 – 0.78 (m, 9 H), 0.26 (d, *J* = 11.9 Hz, 0.5 H, J^{119}Sn–^{1}H = 54.0 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.0, 171.7, 160.0, 159.9, 143.2, 142.9, 136.9, 135.3, 129.5, 129.4, 127.7, 127.5, 125.4, 124.9, 81.7, 81.2, 72.2, 68.7, 68.6, 64.1, 54.3, 49.8, 35.8, 34.9, 29.2, 29.1, 29.0, 28.8, 27.93, 27.92, 27.63, 27.62, 27.2, 27.1, 27.0, 26.9, 21.6, 21.5, 20.7, 19.6, 18.8, 17.6, 14.5, 14.2, 13.8, 13.75, 13.73, 13.70, 13.1, 13.0, 12.6, 12.5, 12.3; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 137.0, 132.7; HRMS (FAB^+ M+2) *m/z* 627.2404. calcd for $C_{29}H_{49}NO_4SSn$ 627.2404

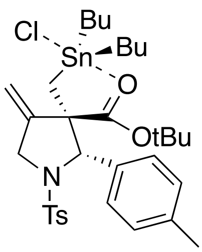
Preparарion of TBP tin complex (4b): To a solution of **2b** (120,0 mg, 0.18 mmol) in ether (20 mL) was added 12 M HCl aq (0.5 mL) and the resulting biphasic mixture was stirred at room temperature for 18 h. The organic phase was separated, washed with brine (10 ml x 2), and dried over Na_2SO_4. After filtration, the filtrate was concentrated in vacuo and the residue was purified through flash chromatography (hexane-EtOAc (20:1) to give **4b** in 100% yield (123.0 mg, 0.18 mmol).



White solid; mp 58 – 59 ºC; $[\alpha]_D$ –36.0 (c 1.05, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23 – 7.09 (m, 5 H), 6.99 (d, *J* = 8.4 Hz, 2 H), 6.89 (br, 2 H), 5.26 (s, 1 H), 5.11 (s, 2

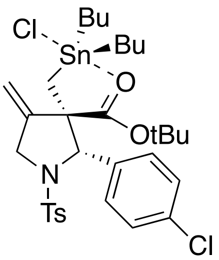
H), 4.55 (dt, J = 13.3, 2.0 Hz, 1 H), 4.07 (d, J = 13.3 Hz, 1 H), 2.30 (s, 3 H), 1.58 (s, 9 H), 1.44 – 1.14 (m, 12 H), 1.08 (d, J = 13.9 Hz, 1 H), 0.91 (t, J = 7.3 Hz, 3 H), 0.88 (t, J = 7.3 Hz, 3 H), 0.87 (d, J = 13.1 Hz, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.90 (J^{119}Sn–^{13}C = 14.9 Hz), 147.23 (J^{119}Sn–^{13}C = 28.5 Hz), 142.7, 137.1, 136.3, 129.0, 128.3, 128.2, 127.8, 126.7, 110.4, 86.7, 70.9 (J^{119}Sn–^{13}C = 38.1 Hz), 60.9 (J^{119}Sn–^{13}C = 19.9 Hz), 52.4, 27.9 (J^{119}Sn–^{13}C = 25.5 Hz), 27.9 (J^{119}Sn–13C = 25.5 Hz), 27.6, 26.5 (J^{117}Sn–^{13}C = 79.0 Hz, J^{119}Sn–^{13}C = 82.6 Hz), 26.4 (J^{117}Sn–^{13}C = 76.7 Hz, J^{119}Sn–^{13}C = 80.1 Hz), 21.3 (J^{117}Sn–^{13}C = 429.5 Hz, J^{119}Sn–^{13}C = 459.2 Hz), 21.5, 20.2 (J^{117}Sn–^{13}C = 439.3 Hz, J^{119}Sn–^{13}C = 459.9 Hz), 19.8 (J^{117}Sn–^{13}C = 435.8 Hz, J^{119}Sn–^{13}C = 455.9 Hz), 13.6, 13.5; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 24.8; IR (neat) 2922, 1659, 1343, 1161, 812 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 696.1931. calcd for $C_{32}H_{47}ClNO_4SSn$ 696.1936.

TBP complex (4c)



White solid; mp 92 – 93 ºC; $[\alpha]_D$ –36.0 (c 1.00, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.18 (d, J = 8.3 Hz, 2 H), 6.99 (d, J = 8.0 Hz, 2 H), 6.91 (br, 2 H), 6.75 (br, 2 H), 5.24 (s, 1 H), 5.10 (s, 1 H), 5.06 (s, 1 H), 4.53 (dt, J = 13.3, 2.2 Hz, 1 H), 4.06 (d, J = 13.4 Hz, 1 H), 2.32 (s, 3 H), 2.28 (s, 3 H), 1.56 (s, 9 H), 1.40 – 1.12 (m, 12 H), 1.08 (d, J = 13.9 Hz, 1 H, J^{119}Sn–^{1}H = 67.1 Hz), 0.90 (t, J = 7.2 Hz, 3 H), 0.90 (d, J = 14.3 Hz, 1 H, J^{119}Sn–^{1}H = 72.2 Hz), 0.88 (t, J = 7.3 Hz, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 179.1 (J^{119}Sn–^{13}C = 16.2 Hz), 147.4 (J^{119}Sn–^{13}C = 27.9 Hz), 142.7, 142.7, 138.0, 134.0, 129.0, 128.9, 127.9, 126.8, 110.30, 86.7, 70.9 (J^{119}Sn–^{13}C = 39.5 Hz), 61.0 (J^{119}Sn–^{13}C = 19.8 Hz), 52.5, 27.91 (J^{119}Sn–^{13}C = 26.8 Hz), 27.91 (J^{119}Sn–^{13}C = 26.8 Hz), 27.65, 26.64 (J^{119}Sn–^{13}C = 79.4 H), 26.61 (J^{119}Sn–^{13}C = 77.4 Hz), 21.5, 21.4 (J^{117}Sn–^{13}C = 430.2 Hz, J^{119}Sn–^{13}C = 448.0 Hz), 21.1, 20.3 (J^{117}Sn–^{13}C = 438.0 Hz, J^{119}Sn–^{13}C = 470.2 Hz), 19.8 (J^{117}Sn–^{13}C = 435.2 Hz, J^{119}Sn–^{13}C = 455.0 Hz), 13.7, 13.6; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 25.4; IR (neat) 2924, 1655, 1342, 1159, 907 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 710.2088. calcd for $C_{33}H_{49}ClNO_4SSn$ 710.2093.

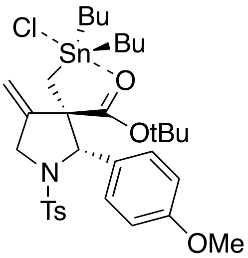
TBP complex (4e)



Pale yellow oil; $[\alpha]_D$ –50.6 (c 1.01, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 (d, J = 8.3 Hz, 1 H), 7.10 (br, 2 H), 7.04 (d, J = 8.1 Hz, 2 H), 6.82 (s, 2 H), 5.27 (s, 1 H), 5.10 (s, 1 H), 5.08 (s, 1 H), 4.54 (d, J = 13.4 Hz, 1 H), 4.09 (d, J = 13.4 Hz, 1 H), 2.34 (s, 3 H), 1.57 (s, 9 H), 1.47 – 1.13 (m, 12 H), 1.02 (d, J = 13.9 Hz, 1 H, J^{119}Sn–^{1}H = 73.5 Hz), 0.90 (t, J = 7.4 Hz, 3 H), 0.88 (t, J = 7.3 Hz, 3 H), 0.87 (d, J = 14.2 Hz, 1 H, J^{119}Sn–^{1}H = 72.7 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.5 (J^{119}Sn–^{13}C = 14.4 Hz), 146.8 (J^{119}Sn–^{13}C = 23.5 Hz), 143.1, 136.3, 135.7, 134.1, 129.1, 129.0, 128.5, 126.6, 110.8, 87.0, 70.1 (J^{119}Sn–^{13}C = 42.0 Hz), 60.8 (J^{119}Sn–^{13}C = 19.8 Hz), 52.4, 27.9 (J^{119}Sn–^{13}C = 24.1 Hz), 27.8 (J^{119}Sn–^{13}C = 25.6 Hz), 27.6, 26.56 (J^{117}Sn–^{13}C = 79.6 Hz, J^{119}Sn–^{13}C = 83.0 Hz), 26.53 (J^{117}Sn–^{13}C = 76.1 Hz, J^{119}Sn–^{13}C = 79.8 Hz), 21.6, 21.4 (J^{117}Sn–^{13}C = 442.5 Hz, J^{119}Sn–^{13}C = 468.9 Hz), 20.3 (J^{117}Sn–^{13}C = 440.6 Hz, J^{119}Sn–^{13}C = 460.8 Hz), 19.8 (J^{117}Sn–^{13}C = 446.4 Hz, J^{119}Sn–^{13}C = 466.1 Hz), 13.6, 13.5; ^{119}Sn NMR (186

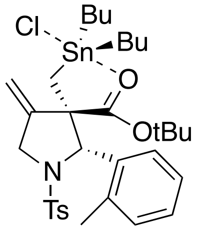
MHz, $CDCl_3$) δ 24.7; IR (neat) 2924, 1668, 1344, 1163, 912 cm^{-1}; HRMS (FAB^+ M+1) *m/z* 730.1546. calcd for $C_{32}H_{46}Cl_2NO_4SSn$ 730.1547.

TBP complex (4f)



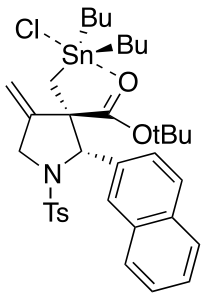
Colorless oil; $[\alpha]_D$ –50.1 (c 1.02, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.19 (d, *J* = 8.3 Hz, 2 H), 7.01 (d, *J* = 8.1 Hz, 2 H), 6.79 (br, 2 H), 6.66 (br, 2 H), 5.24 (s, 1 H), 5.11 (s, 1 H), 5.05 (s, 1 H), 4.53 (dt, *J* = 13.4, 2.2 Hz, 1 H)., 4.04 (d, *J* = 13.3 Hz, 1 H), 3.77 (s, 3 H), 2.32 (s, 3 H), 1.57 (s, 9 H), 1.41 – 1.16 (m, 12 H), 1.10 (d, *J* = 13.9 Hz, 1 H, J^{119}Sn–^{1}H = 67.9 Hz), 0.91 (t, *J* = 7.3 Hz, 3 H), 0.90 (d, *J* = 13.2 Hz, 1 H, J^{119}Sn–^{1}H = 66.5 Hz), 0.88 (t, *J* = 7.3 Hz, 3 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 179.1 (J^{119}Sn–^{13}C = 14.9 Hz), 159.5, 147.4 (J^{119}Sn–^{13}C = 29.6 Hz), 142.7, 136.5, 129.2, 129.0, 126.8, 113.8, 110.3, 86.7, 70.6 (J^{119}Sn–^{13}C = 39.1 Hz), 61.0 (J^{119}Sn–^{13}C = 19.4 Hz), 55.2, 52.3, 27.9 (J^{119}Sn–^{13}C = 27.3 Hz), 27.8 (J^{119}Sn–^{13}C = 26.6 Hz), 27.6, 26.6 (J^{117}Sn–^{13}C = 79.1 Hz, J^{119}Sn–^{13}C = 82.6 Hz), 26.5 (J^{117}Sn–^{13}C = 76.6 Hz, J^{119}Sn–^{13}C = 80.3 Hz), 21.4, 21.3 (J^{117}Sn–^{13}C = 426.8 Hz, J^{119}Sn–^{13}C = 456.1 Hz), 20.2 (J^{117}Sn–^{13}C = 439.5 Hz, J^{119}Sn–^{13}C = 460.1 Hz), 19.8 (J^{117}Sn–^{13}C = 435.4 Hz, J^{119}Sn–^{13}C = 455.4 Hz), 13.6. 13.5; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 25.1; IR (neat) 2926, 1657, 1342, 1250, 1161, 1098, 910 cm^{-1}; HRMS (FAB^+ M+1) *m/z* 726.2025. calcd for $C_{33}H_{49}ClNO_5SSn$ 726.2042.

TBP complex (4g)



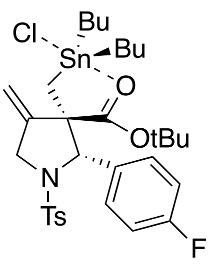
White solid; mp 84 – 84 ºC; $[\alpha]_D$ – 33.6 (c 1.05, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.16 – 7.09 (m, 3 H), 7.04 (td, *J* = 7.5, 1.2 Hz, 1 H)., 6.96 (d, *J* = 8.0 Hz, 2 H), 6.73 (t, *J* = 7.2 Hz, 1 H), 6.42 (d, *J* = 7.8 Hz, 1 H), 5.61 (s, 1 H), 5.35 (s, 1 H), 5.16 (s, 1 H), 4.56 (dt, *J* = 13.1, 2.3 Hz, 1 H), 4.05 (d, *J* = 13.0, 1 H), 2.38 (s, 3 H), 2.29 (s, 3 H), 1.61 (s, 9 H), 1.41 – 1.13 (m, 12 H), 0.97 (d, *J* = 14.1 Hz, 1 H, J^{119}Sn–^{1}H = 82.8 Hz), 0.90 (t, *J* = 7.3 Hz, 3 H), 0.89 (t, *J* = 7.3 Hz, 3 H), 0.68 (d, *J* = 14.2 Hz, 1 H, J^{119}Sn–^{1}H = 55.3 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 179.2 (J^{119}Sn–^{13}C = 17.6 Hz), 147.7 (J^{119}Sn–^{13}C = 5.3 Hz), 142.5, 137.2, 136.4, 135.3, 130.3, 128.9, 127.6, 127.2, 126.4, 126.2, 111.9, 87.1, 65.9 (J^{119}Sn–^{13}C = 62.4 Hz), 60.0 (J^{119}Sn–^{13}C = 19.7 Hz), 52.1, 27.9 (J^{119}Sn–^{13}C = 28.9 Hz), 27.9 (J^{119}Sn–^{13}C = 24.3 Hz), 27.7, 26.6 (J^{117}Sn–^{13}C = 75.6 Hz, J^{119}Sn–^{13}C = 79.5 Hz), 26.5 (J^{117}Sn–^{13}C = 77.0 Hz, J^{119}Sn–^{13}C = 81.0 Hz), 21.7 (J^{117}Sn–^{13}C = 403.5Hz, J^{119}Sn–^{13}C = 424.7 Hz), 21.3, 21.2 (J^{117}Sn–^{13}C = 441.9 Hz, J^{119}Sn–^{13}C = 462.5 Hz), 19.5, 19.2 (J^{117}Sn–^{13}C = 439.9 Hz, J^{119}Sn–^{13}C = 460.7 Hz), 13.6, 13.5; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 23.2; IR (neat) 2924, 1667, 1340, 1159, 910 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 710.2109. calcd for $C_{33}H_{49}ClNO_4SSn$ 710.2093.

TBP complex (4h)



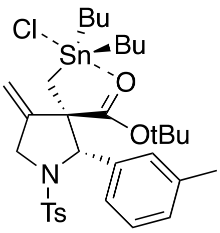
Colorless oil; $[\alpha]_D$ –61.5 (c 1.01, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 – 7.42 (m, 6 H), 7.10 (d, *J* = 6.8 Hz, 2 H), 6.95 – 6.80 (m, 1 H), 6.73 (d, *J* = 8.0 Hz, 2 H), 5.31 (s, 1 H), 5.26 (s, 1 H), 5.15 (s, 1 H), 4.61 (dd, *J* = 13.4, 1.6 Hz, 1 H), 4.18 (d, *J* = 13.5 Hz, 1 H), 2.13 (s, 3 H), 1.60 (s, 9 H), 1.60 – 1.48 (m, 4 H), 1.38 – 1.17 (m, 8 H), 1.07 (d, *J* = 14.0 Hz, 1 H), 0.97 (dd, *J* = 13.9, 1.4 Hz, 1 H), 0.87 (t, *J* = 6.5 Hz, 3 H), 0.85 (t, *J* = 7.3 Hz, 3 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 179.0 (J^{119}Sn–^{13}C = 14.2 Hz), 147.5 (J^{119}Sn–^{13}C = 27.5 Hz), 142.9, 136.5, 134.4, 133.1, 132.9, 130.1, 128.9, 128.4, 128.0, 127.6, 126.7, 126.5, 126.4, 125.2, 110.8, 87.0, 71.4 (J^{119}Sn–^{13}C = 42.5 Hz), 61.1 (J^{119}Sn–^{13}C = 15.2 Hz), 52.7, 28.1 (J^{119}Sn–^{13}C = 25.9 Hz), 28.0 (J^{119}Sn–^{13}C = 27.2Hz), 27.9, 26.8 (J^{117}Sn–^{13}C = 78.9 Hz, J^{119}Sn–^{13}C = 82.9 Hz), 26.7 (J^{117}Sn–^{13}C = 76.8 Hz, J^{119}Sn–^{13}C = 80.3 Hz), 21.8, 21.3 (J^{117}Sn–^{13}C = 409.6 Hz, J^{119}Sn–^{13}C = 422.4 Hz), 20.6 (J^{117}Sn–^{13}C = 439.8 Hz, J^{119}Sn–^{13}C = 459.2 Hz), 19.8 (J^{117}Sn–^{13}C = 435.2 Hz, J^{119}Sn–^{13}C = 455.6 Hz), 13.73, 13.73; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 25.67; IR (neat) 2956, 1660, 1342, 1161, 910 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 746.2074. calcd for $C_{36}H_{49}ClNO_4SSn$ 746.2093.

TBP complex (4i)



Colorless oil; $[\alpha]_D$ –29.0 (c 1.10, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.20 (d, J = 8.3 Hz, 2 H), 7.03 (d, J = 8.1 Hz, 2 H), 6.90 – 6.79 (m, 4 H), 5.27 (s, 1 H), 5.12 (s, 1 H), 5.10 (s, 1 H), 4.55 (dt, J = 13.4, 2.2 Hz, 1 H), 4.07 (d, J = 13.4 Hz, 1 H), 2.33 (s, 3 H), 1.57 (s, 9 H), 1.39 – 1.19 (m, 12 H), 1.06 (d, J = 13.9 Hz, 1 H, J^{119}Sn–^{1}H = 66.1 Hz), 0.91 (t, J = 7.3 Hz, 3 H), 0.88 (t, J = 7.3 Hz, 3 H), 0.84 (d, J = 13.9 Hz, 1 H, J^{119}Sn–^{1}H = 71.0 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.8 (J^{119}Sn–^{13}C = 14.1 Hz), 163.7, 161.6, 147.2 (J^{119}Sn–^{13}C = 27.8 Hz), 143.1, 136.6, 133.32, 133.30, 129.7, 129.2, 126.8, 115.4 (J^{19}F–^{13}C = 21.4 Hz), 110.8, 87.0, 70.2 (J^{119}Sn–^{13}C = 40.7 Hz), 61.0 (J^{119}Sn–^{13}C = 20.1 Hz), 52.5, 28.0 (J^{119}Sn–^{13}C = 26.3 Hz), 28.0 (J^{119}Sn–^{13}C = 26.4 Hz), 27.8, 26.7 (J^{117}Sn–^{13}C = 78.0 Hz, J^{119}Sn–^{13}C = 79.8 Hz), 26.6 (J^{117}Sn–^{13}C = 78.8 Hz, J^{119}Sn–^{13}C = 82.6 Hz), 21.8 (J^{117}Sn–^{13}C = 400.6 Hz, J^{119}Sn–^{13}C = 421.0 Hz), 21.4, 20.4 (J^{117}Sn–^{13}C = 439.8 Hz, J^{119}Sn–^{13}C = 460.6 Hz), 20.0 (d, J^{117}Sn–^{13}C = 436.0 Hz, J^{119}Sn–^{13}C = 455.4 Hz), 13.8, 13.6; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 24.5; IR (neat) 2924, 1658, 1508, 1157 cm^{-1}; HRMS (FAB^{+} M+1) *m/z* 714.1844. calcd for $C_{32}H_{46}ClFNO_4SSn$ 714.1842.

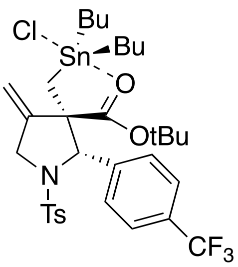
TBP complex (4j)



Pale yellow oil; $[\alpha]_D$ –23.8 (c 1.06, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 (d, J = 8.3 Hz, 2 H), 6.98 (d, J = 7.9 Hz, 4 H), 6.73 – 6.46 (m, 2 H), 5.25 (s, 1 H), 5.10 (s, 1 H), 5.06 (s, 1 H), 4.56 (dt, J = 13.4, 2.2 Hz, 1 H), 4.08 (d, *J* = 13.3 Hz, 1 H), 2.30 (s, 3 H), 2.14 (s, 3 H), 1.58 (s, 9 H), 1.44 – 1.16 (m, 12 H), 1.09 (d, *J* = 13.9 Hz, 1 H, J^{119}Sn–^{1}H = 67.7 Hz), 0.92 (t, *J* = 7.3 Hz, 3 H), 0.88 (t, *J* = 7.3 Hz, 3 H), 0.86 (d, *J* = 13.7 Hz, 1 H, J^{119}Sn–^{1}H = 70.0 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 179.0 (J^{119}Sn–^{13}C = 14.8 Hz), 147.3 (J^{119}Sn–^{13}C = 30.8 Hz), 142.6, 137.9, 136.8, 136.5, 128.9, 128.8, 128.2, 126.7, 110.3, 86.6, 71.0 (J^{119}Sn–^{13}C = 37.8 Hz), 60.9 (J^{119}Sn–^{13}C = 19.9 Hz), 52.5, 27.9 (J^{119}Sn–^{13}C = 27.1 Hz), 27.8 (J^{119}Sn–^{13}C = 26.0 Hz), 27.6, 26.5 (J^{117}Sn–^{13}C = 78.3 Hz, J^{119}Sn–^{13}C = 81.7 Hz), 26.5 (J^{117}Sn–^{13}C = 75.8 Hz, J^{119}Sn–^{1}H = 79.5 Hz), 21.5, 21.2

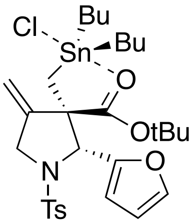
(J^{117}Sn–^{13}C = 409.3 Hz, J^{119}Sn–^{13}C = 426.1 Hz), 21.1, 20.2 (J^{117}Sn–^{13}C = 440.0 Hz, J^{119}Sn–^{13}C = 458.5 Hz), 19.9 (J^{117}Sn–^{13}C = 435.3.3 Hz, J^{119}Sn–^{1}H = 458.5 Hz), 13.6, 13.5; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 25.2; IR (neat) 2924, 1658, 1342, 1159 cm^{-1}; HRMS (FAB^+ M+1) *m/z* 710.2103. calcd for $C_{33}H_{49}ClNO_4SSn$ 710.2093.

TBP complex (4k)



Pale yellow oil; $[\alpha]_D$ –33.0 (c 1.00, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) ™ 7.36 (d, J = 6.4 Hz, 2 H), 7.18 (d, J = 8.2 Hz, 2 H), 7.06 – 6.94 (m, 2 H), 6.98 (d, J = 8.3 Hz, 2 H), 5.30 (s, 1 H), 5.15 (s, 1 H), 5.12 (s, 1 H), 4.58 (d, J = 13.5 Hz, 1 H), 4.15 (d, J = 13.5 Hz, 1 H), 2.29 (s, 3 H), 1.58 (s, 9 H), 1.64 – 1.19 (m, 12 H), 0.99 (d, J = 13.9 Hz, 1 H, J^{119}Sn–^{1}H = 65.6 Hz), 0.89 (t, J = 7.3 Hz, 3 H), 0.88 (t, J = 7.4 Hz, 3 H), 0.85 (d, J = 14.0 Hz, 1 H, J^{119}Sn–^{1}H = 72.6 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.2 (J^{119}Sn–^{13}C = 14.3 Hz), 146.6 (J^{119}Sn–^{13}C = 22.5 Hz), 143.2, 141.2, 136.2, 130.2 (q, J^{19}F–^{13}C = 32.6 Hz), 129.1, 126.5, 125.2 (q, J^{19}F–^{13}C = 3.7 Hz), 123.7 (q, J^{19}F–^{13}C = 272.3 Hz), 111.0, 87.1, 70.1 (J^{119}Sn–^{13}C = 43.5 Hz), 60.7 (J^{119}Sn–^{13}C = 19.6 Hz), 52.6, 27.8 (J^{119}Sn–^{13}C = 27.0 Hz), 27.6, 26.5 (J^{117}Sn–^{13}C = 73.4 Hz, J^{119}Sn–^{13}C = 83.2 Hz), 26.4 (J^{117}Sn–^{13}C = 73.6 Hz, J^{119}Sn–^{13}C = 86.7 Hz), 21.7 (J^{117}Sn–^{13}C = 398.4 Hz, J^{119}Sn–^{13}C = 416.9 Hz), 21.1, 20.4 (J^{117}Sn–^{13}C = 441.1 Hz, J^{119}Sn–^{13}C = 461.8 Hz), 19.7 (J^{117}Sn–^{13}C = 436.9 Hz, J^{119}Sn–^{13}C = 457.1 Hz), 13.5, 13.4; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 24.4; IR (neat) 2926, 1668, 1323, 1161 cm^{-1}; HRMS (FAB^+ M+1) *m/z* 764.1808. calcd for $C_{33}H_{46}ClF_3NO_4SSn$ 764.1810.

TBP complex (4l)

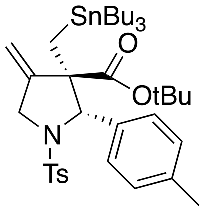


Pale yellow oil; $[\alpha]_D$ –34.9 (c 1.13, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 (d, J = 8.3 Hz, 2 H), 7.09 (d, J = 8.2 Hz, 2 H), 7.05 (d, J = 1.5 Hz, 1 H), 6.19 (dd, J = 3.2, 1.8 Hz, 1 H), 6.13 (d, J = 3.2 Hz, 1 H), 5.18 (s, 1 H), 5.13 (s, 2 H), 4.50 (d, J = 12.9 Hz, 1 H), 3.98 (d, J = 12.8 Hz, 1 H), 2.34 (s, 3 H), 1.73 – 1.58 (m, 4 H), 1.56 (s, 9 H), 1.49 – 1.25 (m, 8 H), 1.25 (d, J = 14.1 Hz, 1 H), 0.95 (t, J = 7.3 Hz, 3 H), 0.90 (t, J = 7.3 Hz, 3 H), 0.66 (d, J = 13.8 Hz, 1 H, J^{119}Sn–^{1}H = 64.3 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.8 (J^{119}Sn–^{13}C = 12.0 Hz), 149.7, 147.2 (J^{119}Sn–^{13}C = 44.9 Hz), 142.7, 136.2, 129.2, 126.6, 110.5, 109.9, 109.4, 86.5, 64.4 (J^{119}Sn–^{13}C = 28.0 Hz), 60.9 (J^{119}Sn–^{13}C = 20.3 Hz), 51.8, 27.9 (J^{119}Sn–^{13}C = 26.4 Hz), 27.8 (J^{119}Sn–^{13}C = 27.2 Hz), 27.5, 26.6 (J-^{117}Sn–^{13}C = 78.8 Hz, J^{119}Sn–^{13}C = 82.6 Hz), 26.5 (J^{117}Sn–^{13}C = 77.3 Hz, J^{119}Sn–^{13}C = 80.5 Hz), 21.3, 20.7 (J^{117}Sn–^{13}C = 402.0 Hz, J^{119}Sn–^{13}C = 422.4 Hz), 20.2 (J^{117}Sn–^{13}C = 432.4 Hz, J^{119}Sn–^{13}C = 452.4 Hz), 19.7 (J^{117}Sn–^{13}C = 436.8 Hz, J^{119}Sn–^{13}C = 457.2 Hz), 13.6, 13.5; ^{119}Sn NMR (186 MHz, $CDCl_3$) δ 28.2; IR (neat) 2942, 1661, 1344, 1161 cm^{-1}; HRMS (FAB$^+$ M+1) *m/z* 686.1730. calcd for $C_{30}H_{45}ClNO_5SSn$ 686.1729.

Radical cyclization of 1c under high Bu_3SnH concentration conditions (Scheme 2)

A mixture of **1c** (172.0 mg, 0.40 mmol) and AIBN (6.9 mg, 0.04 mmol) in Bu_3SnH (0.13 mL, 0.47 mmol) was heated at 110ºC for 1.5 h. The reselting reaction mixture was subjected to column chromatography (hexane then hexane-EtOAc 50:1 to 20:1) to give **2c** (111.7 mg, 0.17 mmol) and **3c** (125.1 mg, 0.17 mmol) in 42% and 44% yield, respectively.

(2*S*,3*S*)-*tert*-butyl 4-methylene-2-(p-tolyl)-1-tosyl-3-((tributylstannyl)methyl)pyrrolidine-3-carboxylate (3c)



Colorless oil; $[\alpha]_D$ –21.3 (c 1.04, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 (d, *J* = 8.3 Hz, 2 H), 6.95 (d, *J* = 8.1 Hz, 2 H), 6.90 (d, *J* = 7.8 Hz, 2 H), 6.82 (d, *J* = 7.2 Hz, 2 H), 5.17 (s, 2 H), 5.12 (s, 1 H), 4.40 (dt, *J* = 13.0, 2.2 Hz, 1 H), 3.96 (d, *J* = 13.0 Hz, 1 H), 2.29 (s, 3 H), 2.27 (s, 3 H), 1.47 (s, 9 H), 1.39 – 1.15 (m, 12 H), 1.00 (d, *J* = 12.8 Hz, 1 H), 0.82 (t, *J* = 7.1 Hz, 9 H), 0.76 – 0.59 (m, 6 H), 0.37 (d, *J* = 13.0 Hz, 1 H, J^{119}Sn–^{1}H = 44.9 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.2, 150.0 (J^{119}Sn–^{13}C = 40.3 Hz), 142.3, 137.5, 136.9 (J^{119}Sn–^{13}C = 47.4 Hz), 135.4, 128.9, 128.8, 127.0, 109.2, 70.2 (J^{119}Sn–^{13}C = 19.1 Hz), 61.4 (J^{119}Sn–^{13}C = 18.9 Hz), 82.1, 52.1, 29.1 (J^{119}Sn–^{13}C = 19.5 Hz), 27.8, 27.5 (J^{119}Sn–^{13}C = 60.0 Hz), 21.4, 21.1 ,13.8, 12.4 (J^{117}Sn–^{13}C = 271.2 Hz, J^{119}Sn–^{13}C = 283.9 Hz), 11.0 (J^{117}Sn–^{13}C = 315.2 Hz, J^{119}Sn–^{13}C = 329.9 Hz); ^{119}Sn NMR (186 MHz, $CDCl_3$) δ –23.1; IR (neat) 2988, 1716, 1342, 1161, 910 cm^{-1}; Anal. Calcd. for $C_{30}H_{41}NO_4SSn$: C, 57.16; H, 6.56; N, 2.22. Found: C, 57.20; H, 6.57; N, 2.20; HRMS (FAB$^+$ M+1) *m/z* 732.3100. calcd for $C_{37}H_{58}NO_4SSn$ 732.3109.

checkCIF/PLATON (full publication check)

Structure factors have been supplied for datablock(s) I

No syntax errors found. CIF dictionary

Please wait while processing .... Interpreting this report

Structure factor report

Datablock: I

Bond precision: C-C = 0.0061 A Wavelength=1.54187

Cell:	a=9.1613(5)	b=10.3390(6)	c=36.334(2)
	alpha=90	beta=90	gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	3441.5(3)	3441.5(4)
Space group	P 21 21 21	P 21 21 21
Hall group	P 2ac 2ab	P 2ac 2ab
Moiety formula	C33 H48 Cl N O4 S Sn	C33 H48 Cl N O4 S Sn
Sum formula	C33 H48 Cl N O4 S Sn	C33 H48 Cl N O4 S Sn
Mr	708.95	708.95
Dx, g cm-3	1.368	1.368
Z	4	4
Mu (mm-1)	7.462	7.471
F000	1472.0	1472.0
F000'	1478.31	
h,k,lmax	11,12,43	10,12,43
Nref	3587[6291]	6254
Tmin,Tmax	0.242,0.224	0.119,0.224

Tmin' 0.155

Correction method= MULTI-SCAN

Data completeness= 1.74/0.99 Theta(max)= 68.180

R(reflections)= 0.0382(5773) wR2(reflections)= 0.0792(6253)

S = 0.881 Npar= 371

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level C

RINTA01_ALERT_3_C The value of Rint is greater than 0.12

Rint given 0.123

PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual

Density 2.08

PLAT910_ALERT_3_C Missing # of FCF Reflections Below Th(Min) .....

1

PLAT912_ALERT_4_C Missing # of FCF Reflections Above STh/L=

0.600 11

PLAT913_ALERT_3_C Missing # of Very Strong Reflections in FCF ....

1

Alert level G

REFLT03_ALERT_4_G Please check that the estimate of the number

of Friedel pairs is

correct. If it is not, please give the correct count in the

_publ_section_exptl_refinement section of the

submitted CIF.

From the CIF: _diffrn_reflns_theta_max 68.18

From the CIF: _reflns_number_total 6254

Count of symmetry unique reflns 3587

Completeness (_total/calc) 174.35%

TEST3: Check Friedels for noncentro structure

Estimate of Friedel pairs measured 2667

Fraction of Friedel pairs measured 0.744

Are heavy atom types Z>Si present yes

PLAT791_ALERT_4_G Note: The Model has Chirality at C3 (Verify) S

PLAT791_ALERT_4_G Note: The Model has Chirality at C4 (Verify) S

PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) still 80 Perc.

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain

0 **ALERT level B** = A potentially serious problem, consider carefully

5 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight

4 **ALERT level G** = General information/check it is not something unexpected

0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data

1 ALERT type 2 Indicator that the structure model may be wrong or deficient

4 ALERT type 3 Indicator that the structure quality may be low

4 ALERT type 4 Improvement, methodology, query or suggestion

0 ALERT type 5 Informative message, check

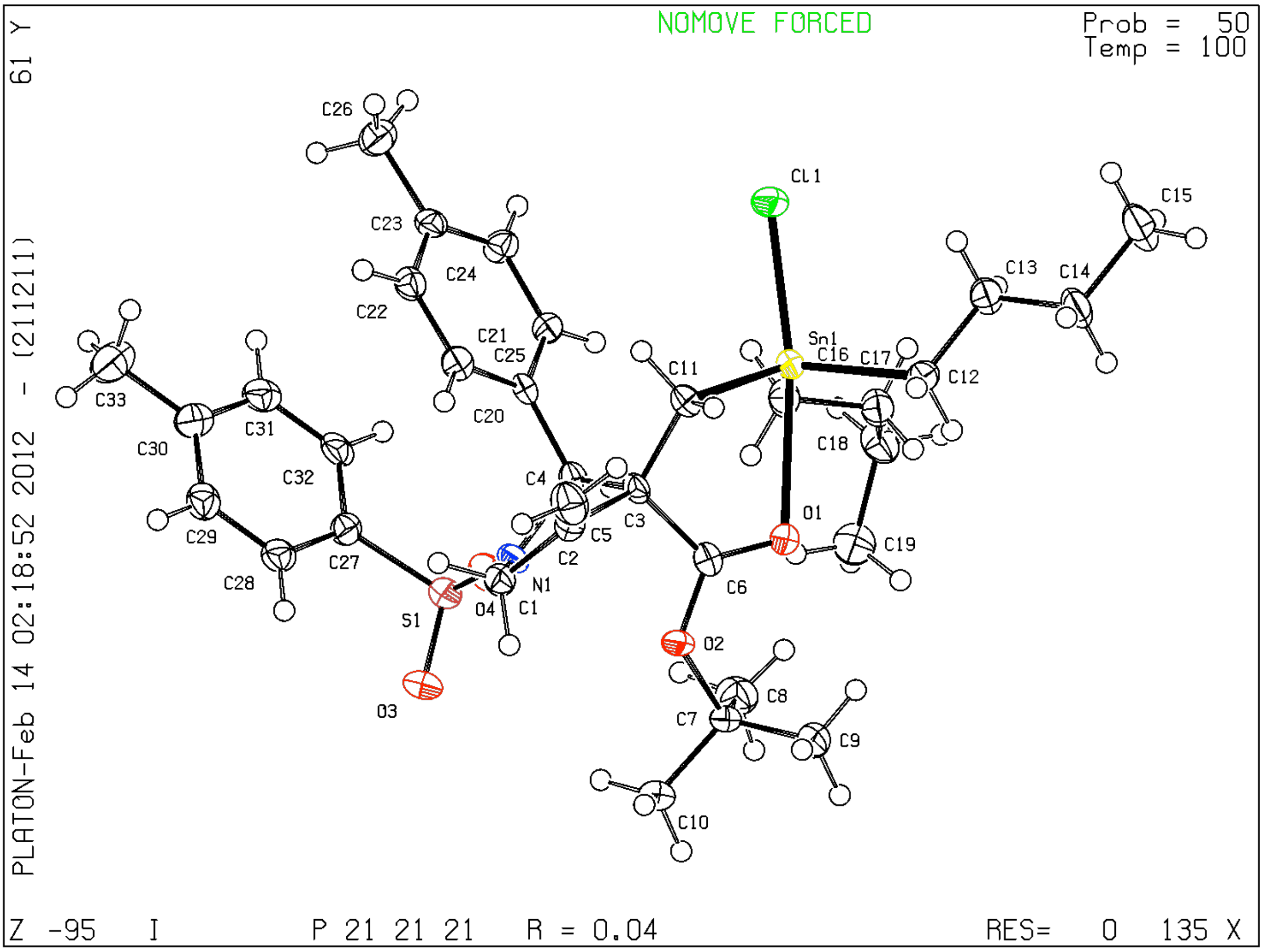
Publication of your CIF

You should attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the nature of your study may justify the reported deviations from journal submission requirements and the more serious of these should be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. *checkCIF* was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

If you wish to submit your CIF for publication in Acta Crystallographica Section C or E, you should upload your CIF viathe web. If your CIF is to form part of a submission to another IUCr journal, you will be asked, either during electronic submission or by the Co-editor handling your paper, to upload your CIF via our web site.

PLATON version of 21/12/2011; check.def file version of 16/12/2011

Datablock I - ellipsoid plot



checkCIF/PLATON (full publication check)

Structure factors have been supplied for datablock(s) I

No syntax errors found. CIF dictionary

Please wait while processing .... Interpreting this report

Structure factor report

Datablock: I

Bond precision: C-C = 0.0156 A Wavelength=0.71075

Cell:	a=8.5992(8)	b=9.5675(9)	c=19.3422(18)
	alpha=82.229(3)	beta=79.942(2)	gamma=89.865(3)

Temperature: 296 K

	Calculated	Reported
Volume	1552.1(3)	1552.1(3)
Space group	P 1	P 1
Hall group	P 1	P 1
Moiety formula	C30 H41 N O4 S Sn	C30 H41 N O4 S Sn
Sum formula	C30 H41 N O4 S Sn	C30 H41 N O4 S Sn
Mr	630.42	630.41
Dx, g cm-3	1.349	1.349
Z	2	2
Mu (mm-1)	0.923	0.923
F000	652.0	652.0
F000'	651.17	
h,k,lmax	11,12,25	11,12,25
Nref	7111[14222]	11472
Tmin,Tmax	0.766,0.912	0.661,0.912

Tmin' 0.751

Correction method= MULTI-SCAN

Data completeness= 1.61/0.81 Theta(max)= 27.470

R(reflections)= 0.0519(6095) wR2(reflections)= 0.1535(11468)

S = 1.092 Npar= 686

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level C

PLAT220_ALERT_2_C Large Non-Solvent C Ueq(max)/Ueq(min) ... 3.9 Ratio

PLAT222_ALERT_3_C Large Non-Solvent H Uiso(max)/Uiso(min) .. 5.0 Ratio

PLAT230_ALERT_2_C Hirshfeld Test Diff for C17 -- C22 .. 5.2 su

PLAT234_ALERT_4_C Large Hirshfeld Difference Sn1 -- C11 .. 0.16 Ang.

PLAT234_ALERT_4_C Large Hirshfeld Difference C18 -- C19 .. 0.16 Ang.

PLAT234_ALERT_4_C Large Hirshfeld Difference C20 -- C23 .. 0.17 Ang.

PLAT234_ALERT_4_C Large Hirshfeld Difference C26 -- C27 .. 0.16 Ang.

PLAT234_ALERT_4_C Large Hirshfeld Difference Sn2 -- C37 .. 0.16 Ang.

PLAT234_ALERT_4_C Large Hirshfeld Difference N2 -- C35 .. 0.16 Ang.

PLAT234_ALERT_4_C Large Hirshfeld Difference C38 -- C39 .. 0.24 Ang.

PLAT234_ALERT_4_C Large Hirshfeld Difference C51 -- C52 .. 0.18 Ang.

PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C15

PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C27

PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C39

PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C43

PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C45

PLAT342_ALERT_3_C Low Bond Precision on C-C Bonds .... 0.0156 Ang

PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C37 - C38 ... 1.43 Ang.

PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C39 - C40 ... 1.34 Ang.

PLAT411_ALERT_2_C Short Inter H...H Contact H37B .. H13D .. 2.07 Ang.

PLAT910_ALERT_3_C Missing # of FCF Reflections Below Th(Min) 10

PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.600 11

PLAT912_ALERT_4_C Missing # of FCF Reflections Above STh/L= 0.600 67

PLAT913_ALERT_3_C Missing # of Very Strong Reflections in FCF 1

PLAT915_ALERT_3_C Low Friedel Pair Coverage .... 62 Perc.

Alert level G

REFLT03_ALERT_4_G Please check that the estimate of the number of Friedel pairs is

correct. If it is not, please give the correct count in the

_publ_section_exptl_refinement section of the submitted CIF.

From the CIF: _diffrn_reflns_theta_max 27.47

From the CIF: _reflns_number_total 11472

Count of symmetry unique reflns 7111

Completeness (_total/calc) 161.33%

TEST3: Check Friedels for noncentro structure

Estimate of Friedel pairs measured 4361

Fraction of Friedel pairs measured 0.613

Are heavy atom types Z>Si present yes

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 7

PLAT003_ALERT_2_G Number of Uiso or Uij Restrained Atom Sites 11

PLAT301_ALERT_3_G Note: Main Residue Disorder .... 4 Perc.

PLAT791_ALERT_4_G Note: The Model has Chirality at C3 (Verify) S

PLAT791_ALERT_4_G Note: The Model has Chirality at C4 (Verify) S

PLAT791_ALERT_4_G Note: The Model has Chirality at C33 (Verify) S

PLAT791_ALERT_4_G Note: The Model has Chirality at C34 (Verify) S

PLAT860_ALERT_3_G Note: Number of Least-Squares Restraints 143

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14 ALERT type 4 Improvement, methodology, query or suggestion

0 ALERT type 5 Informative message, check

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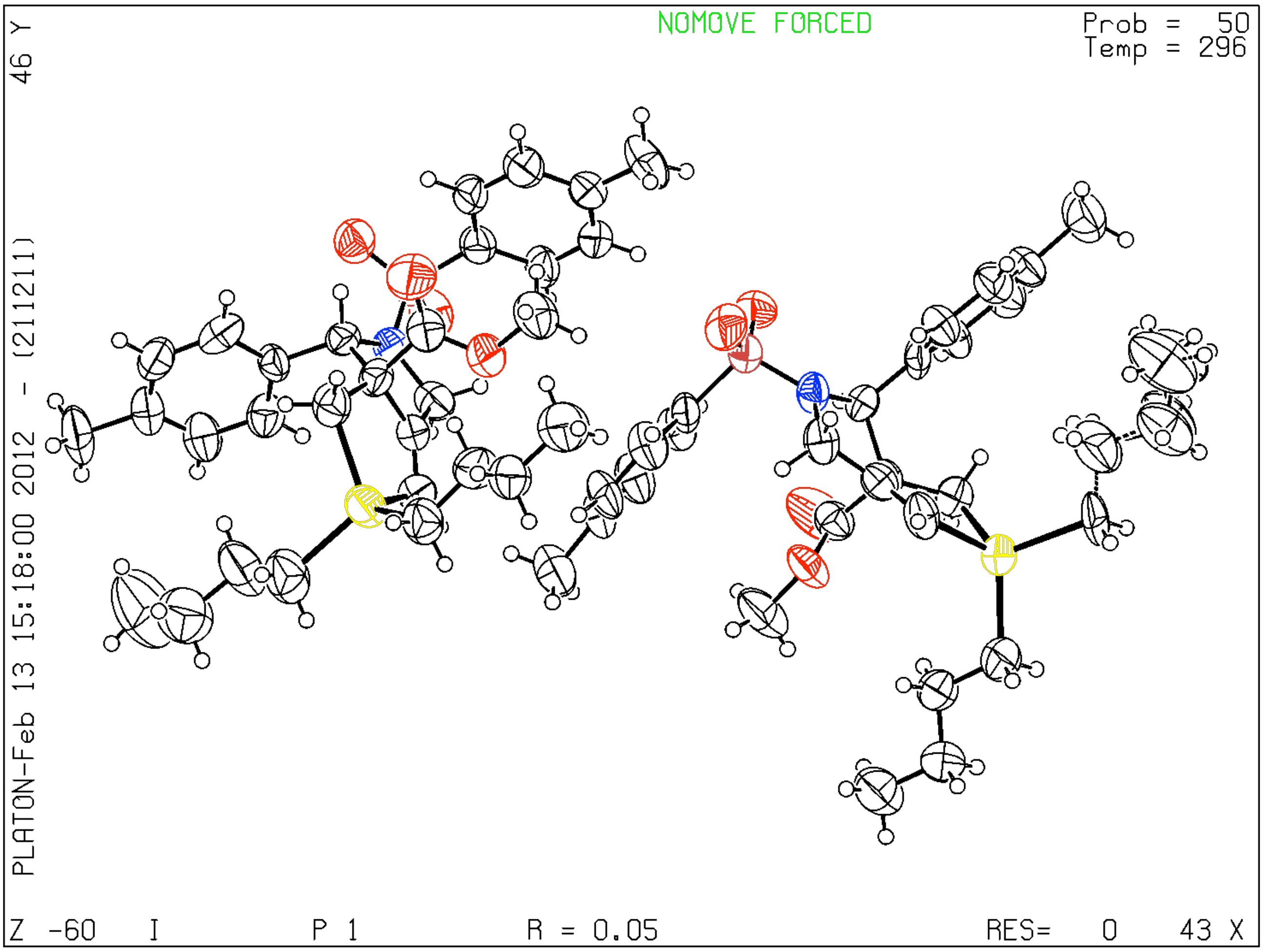
PLATON version of 21/12/2011; check.def file version of 16/12/2011

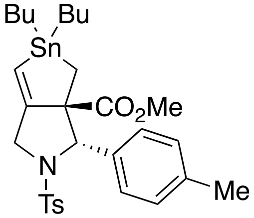
Datablock I - ellipsoid plot



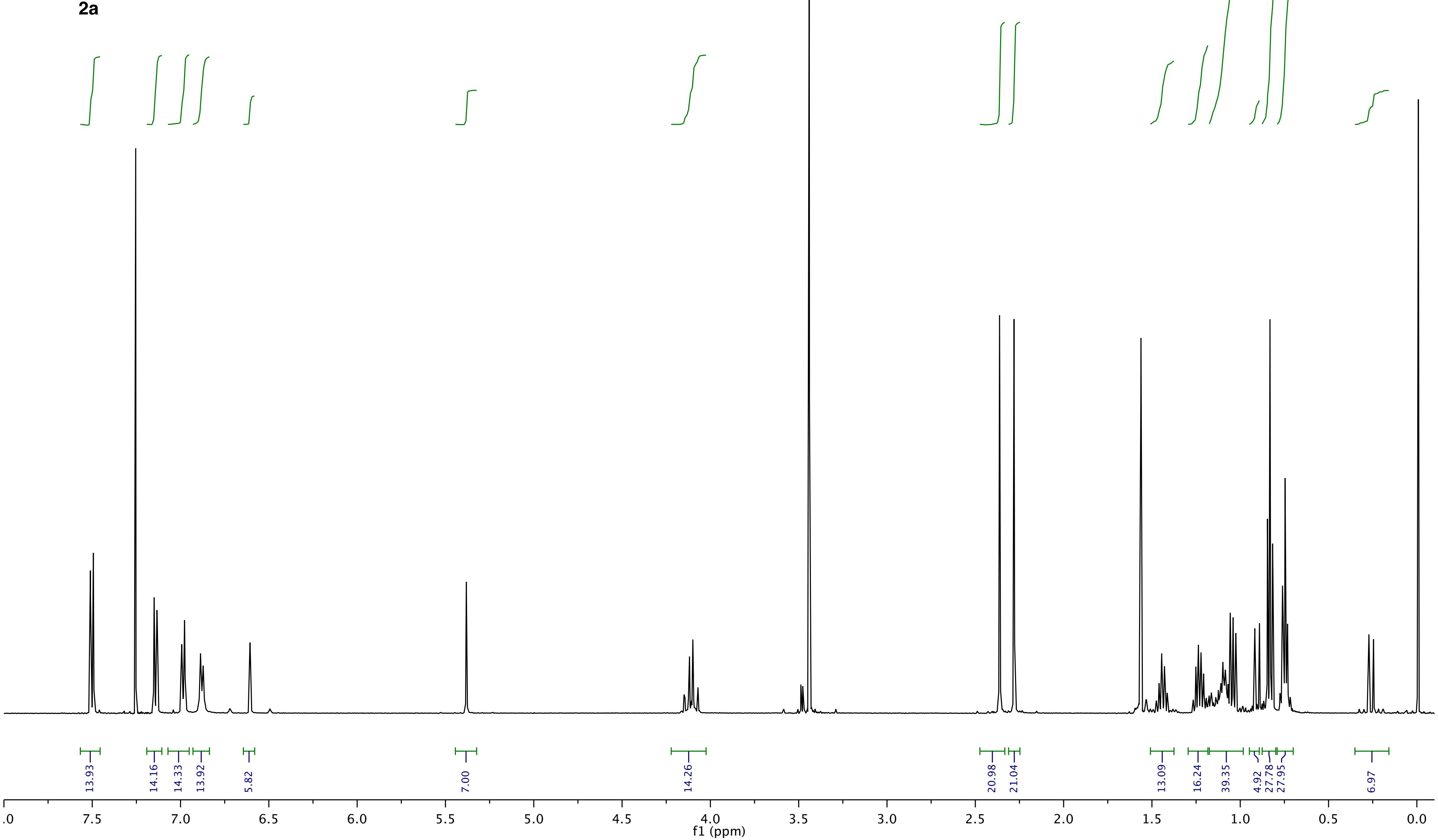
Download CIF editor (publCIF) from the IUCr

Download CIF editor (enCIFer) from the CCDC

Test a new CIF entry


Bu
Bu
Sn
CO2Me
N
Ts
Me

2a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13.09
16.24
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4.92
27.78
27.95
6.97
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7.5
7.0
6.5
6.0
5.5
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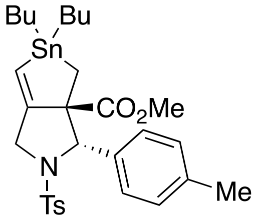
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CO2Me
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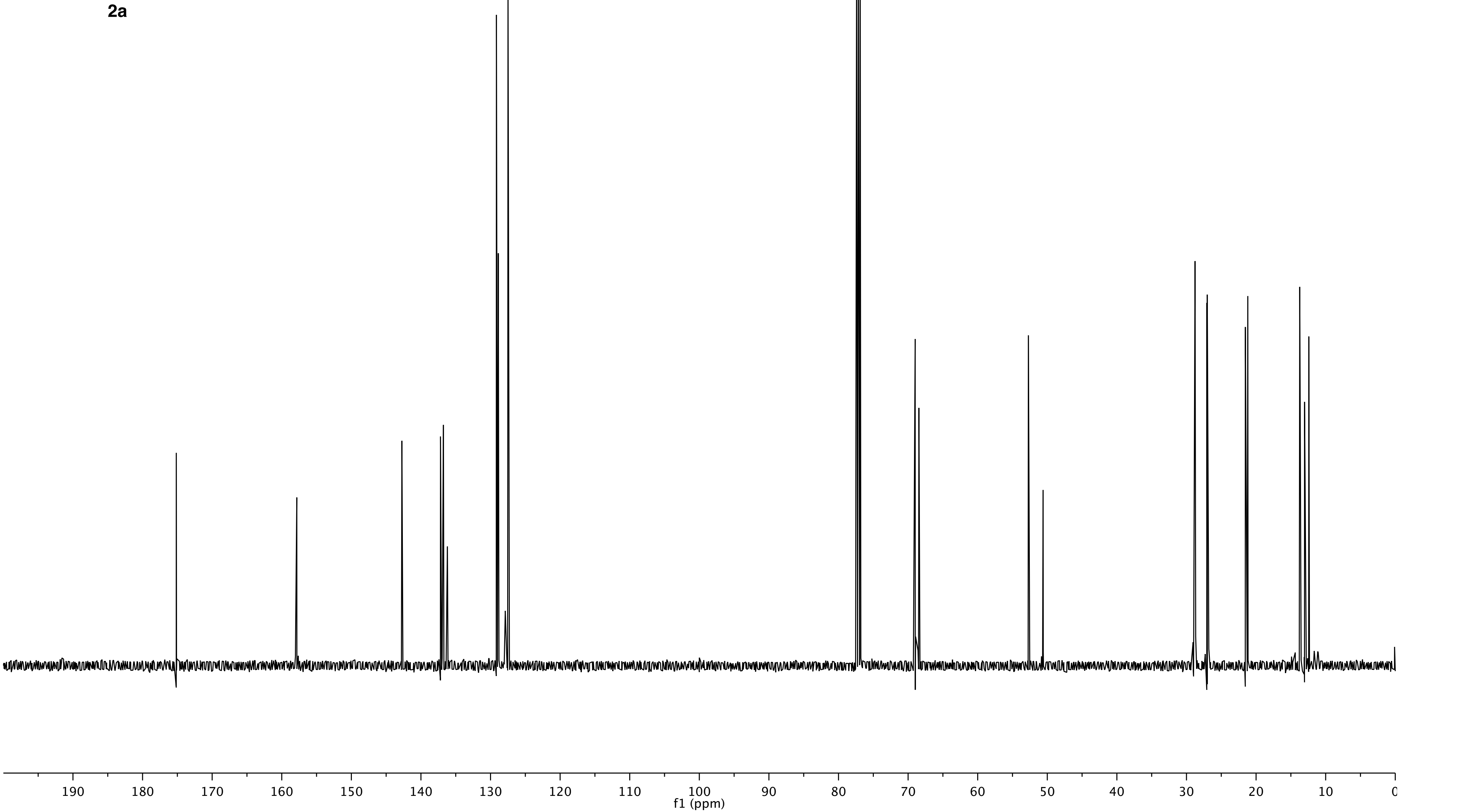
Ts
Me

2a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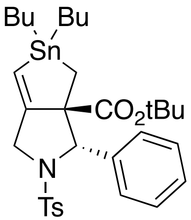
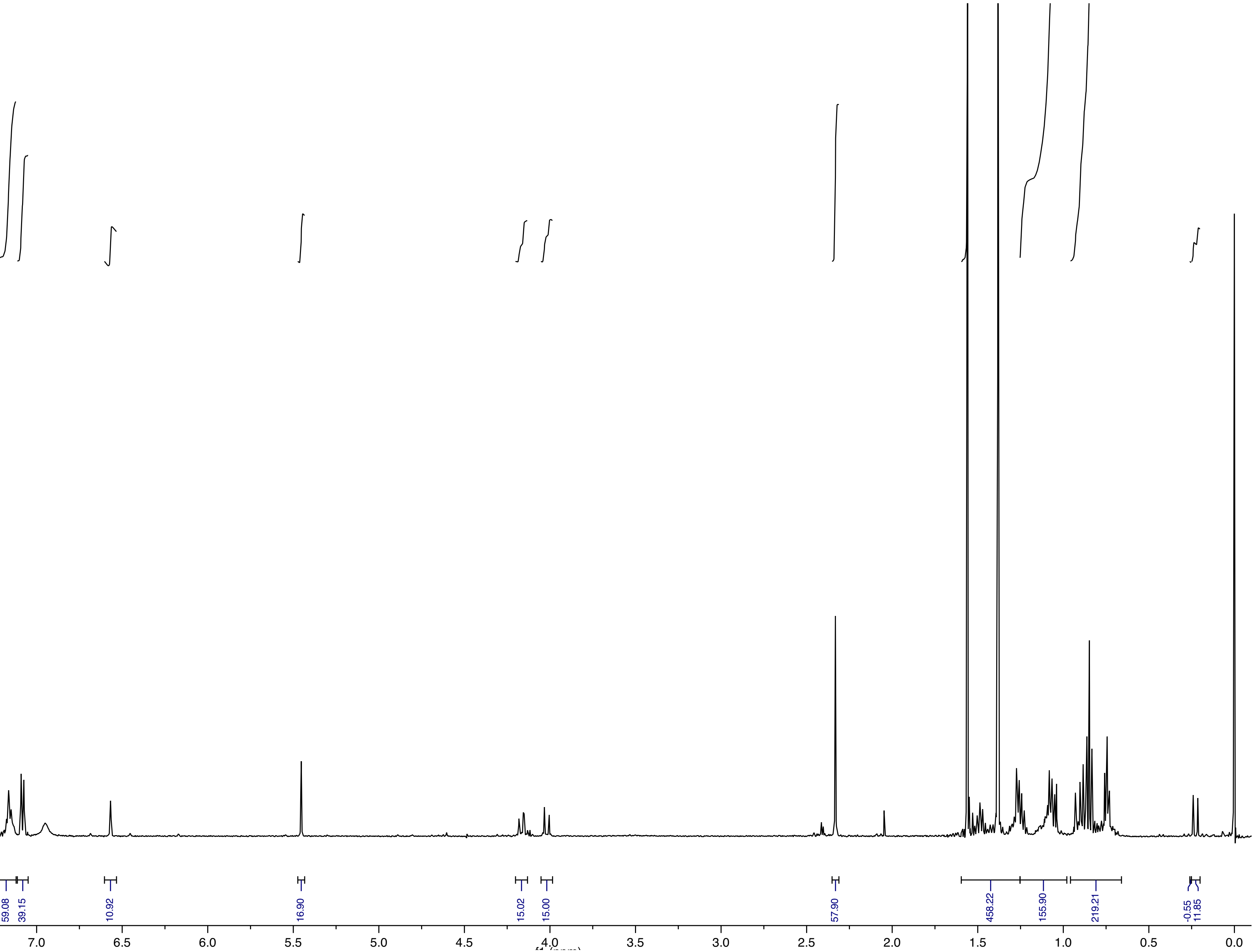

Bu Bu
Sn
CO2tBu
N
Ts

2b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10.92
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15.00
57.90
458.22
155.90
219.21
-0.55
11.85
8.0
7.5
7.0
6.5
6.0
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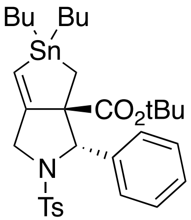
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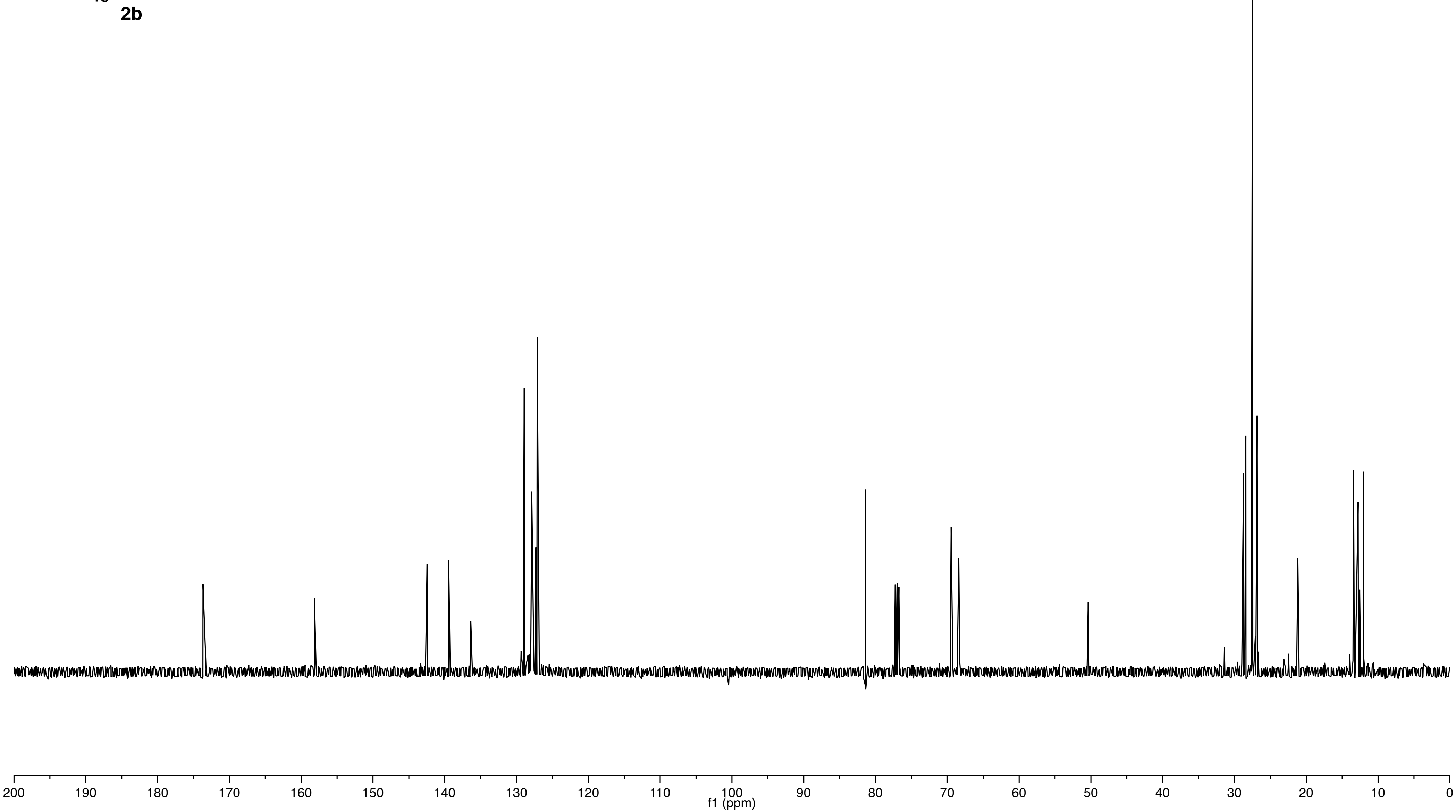
CO2tBu
N
Ts

2b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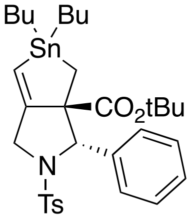
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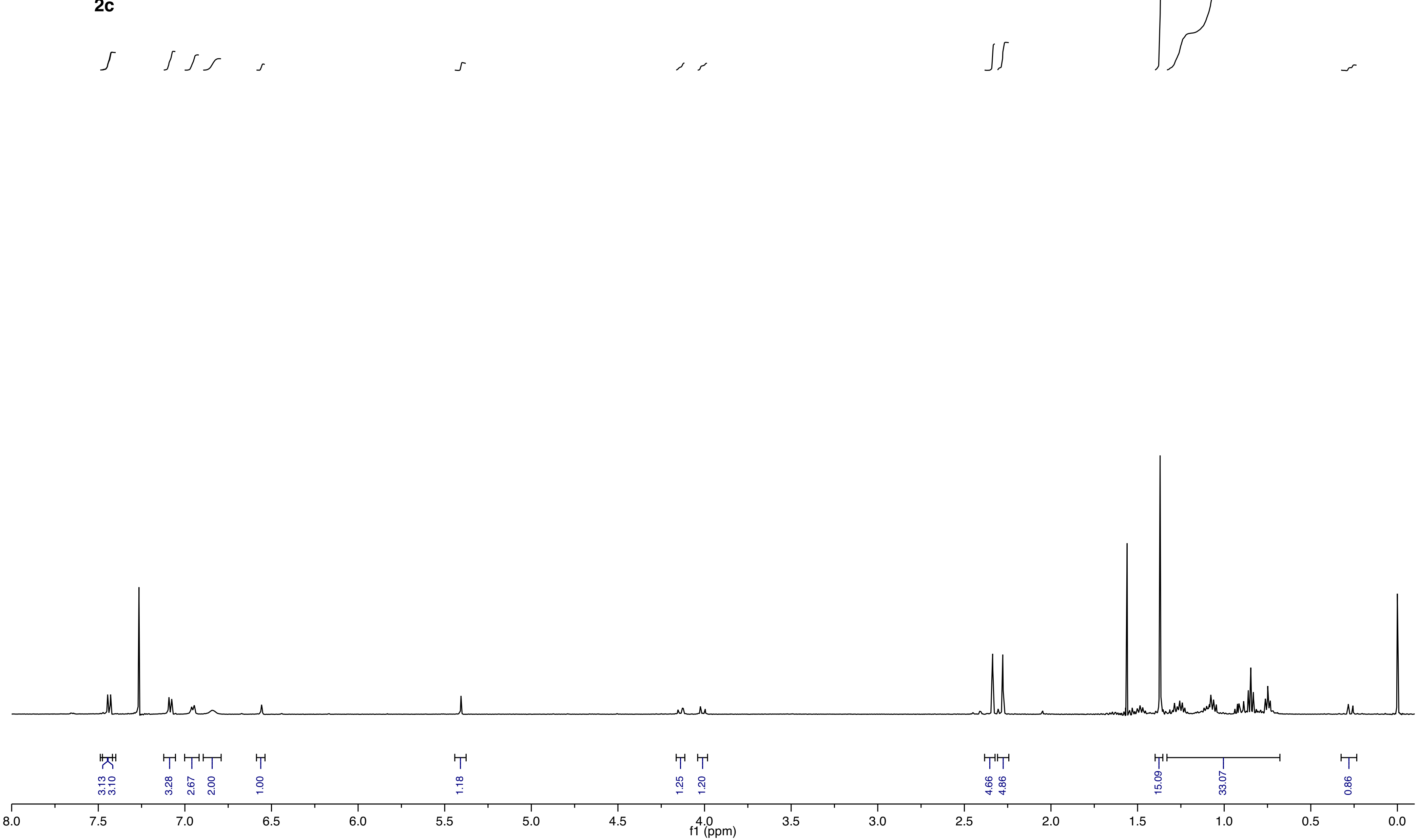
Bu
Sn
CO_2tBu
N
Ts

2c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6.0
5.5
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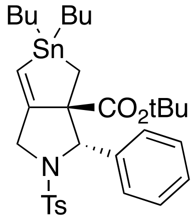
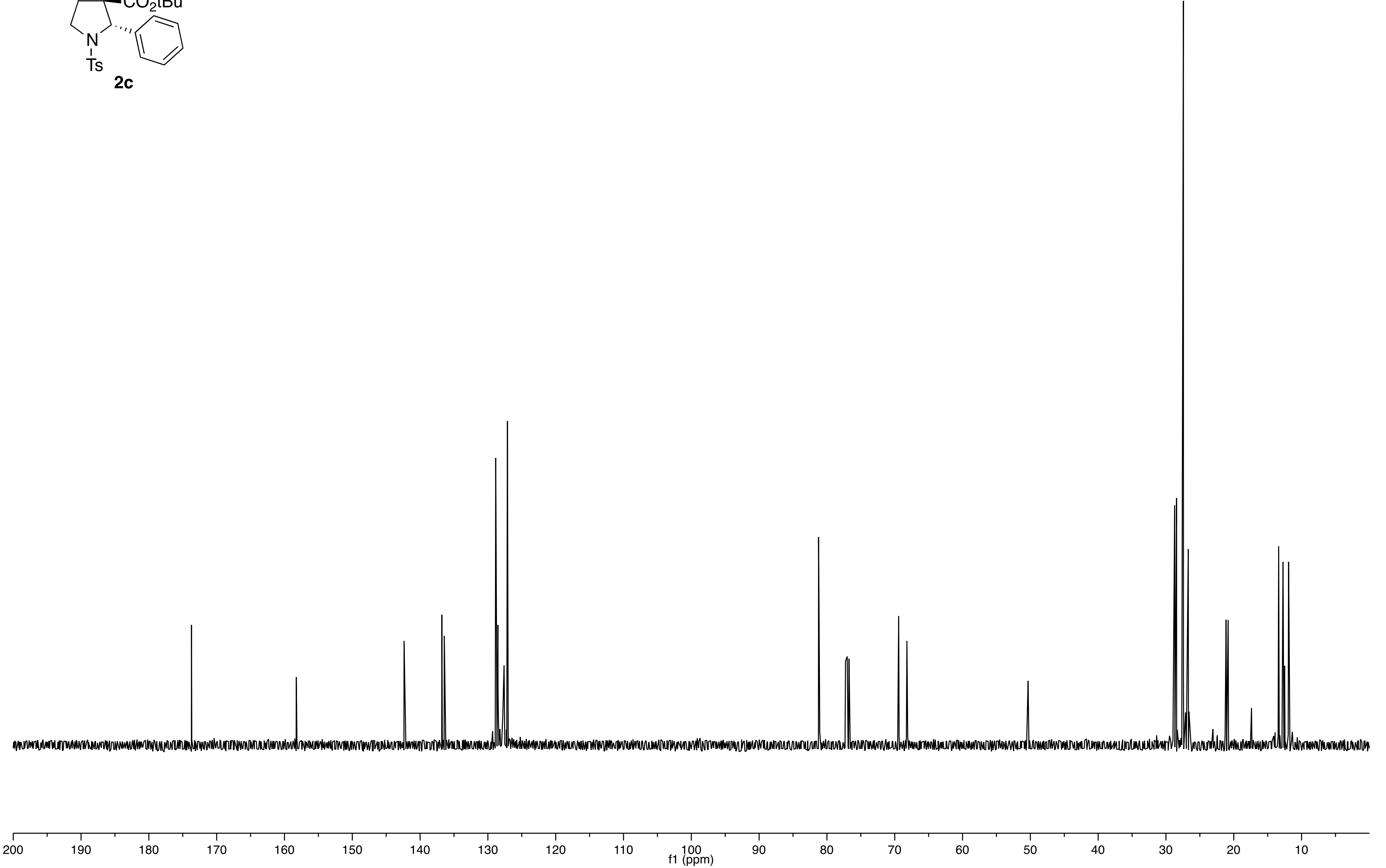

Bu Bu
Sn
CO2tBu
N
Ts

2c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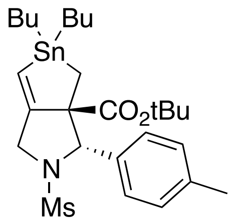
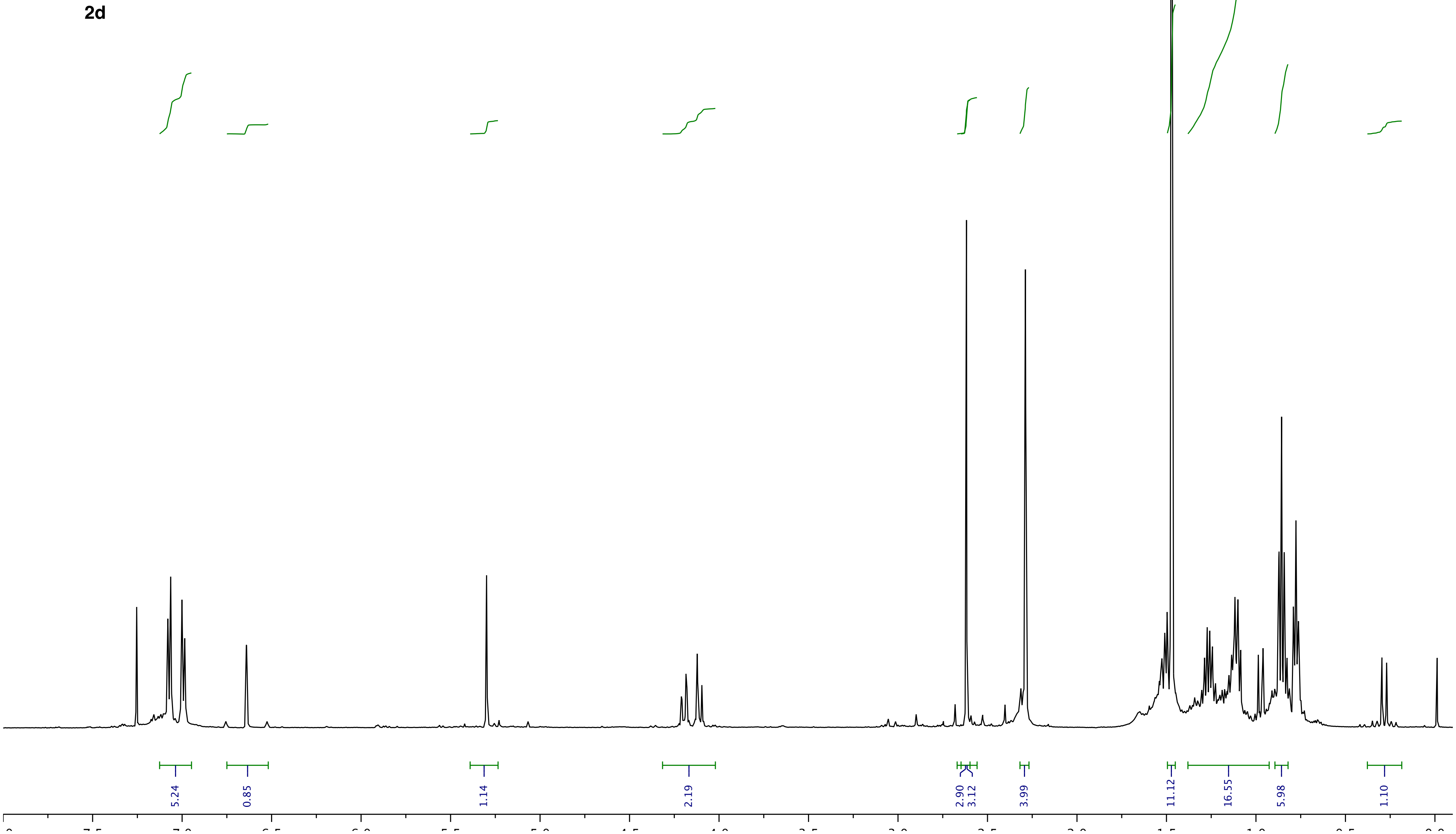

Bu
Bu
Sn
CO2tBu
N
Ms

2d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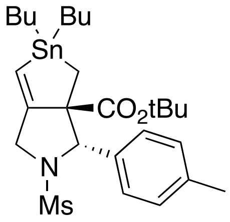
16.55
5.98
1.10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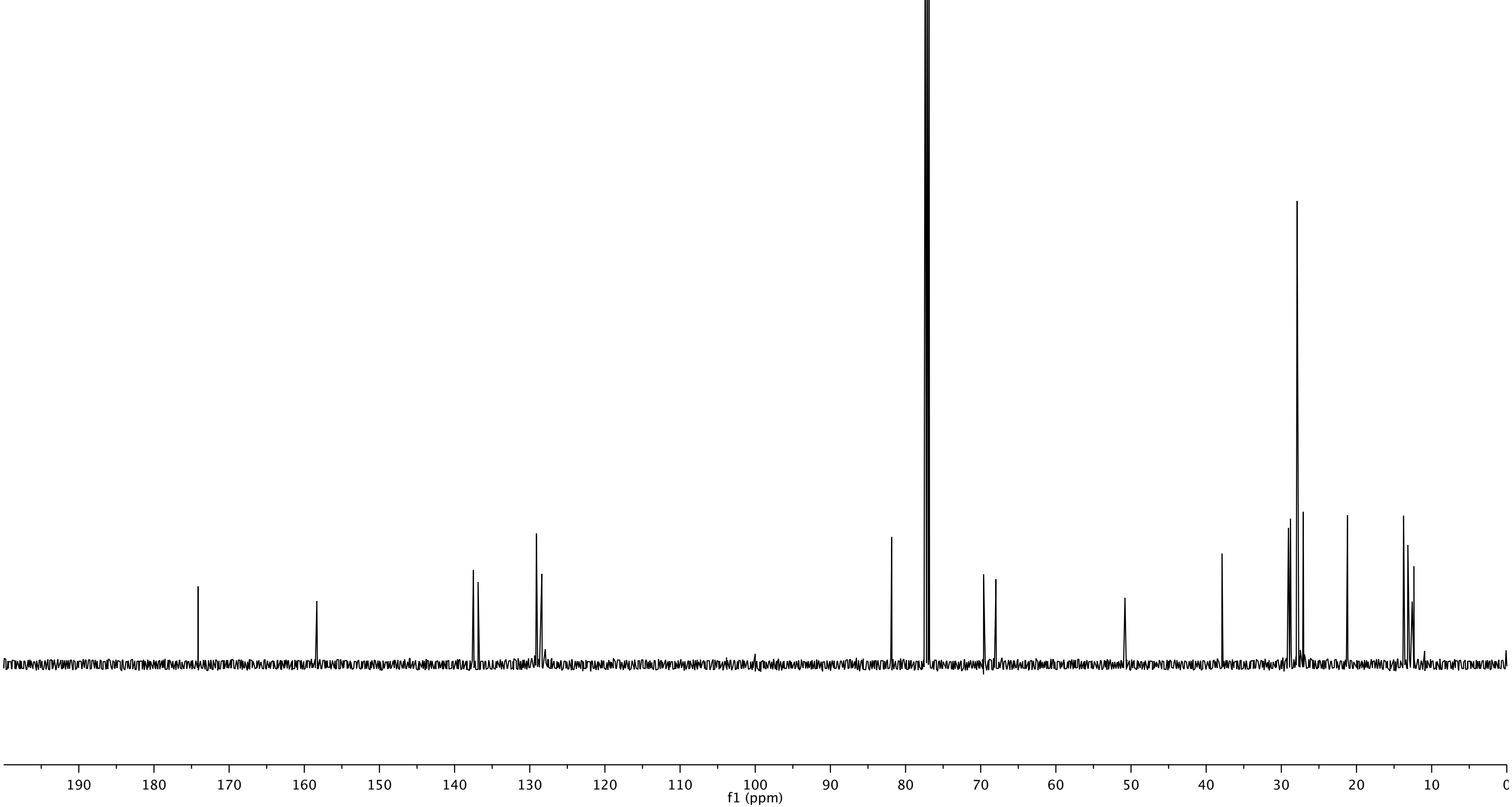
Bu
Sn
CO2tBu
N
Ms

2d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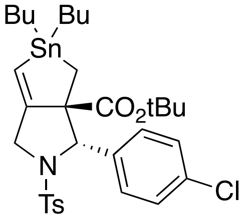
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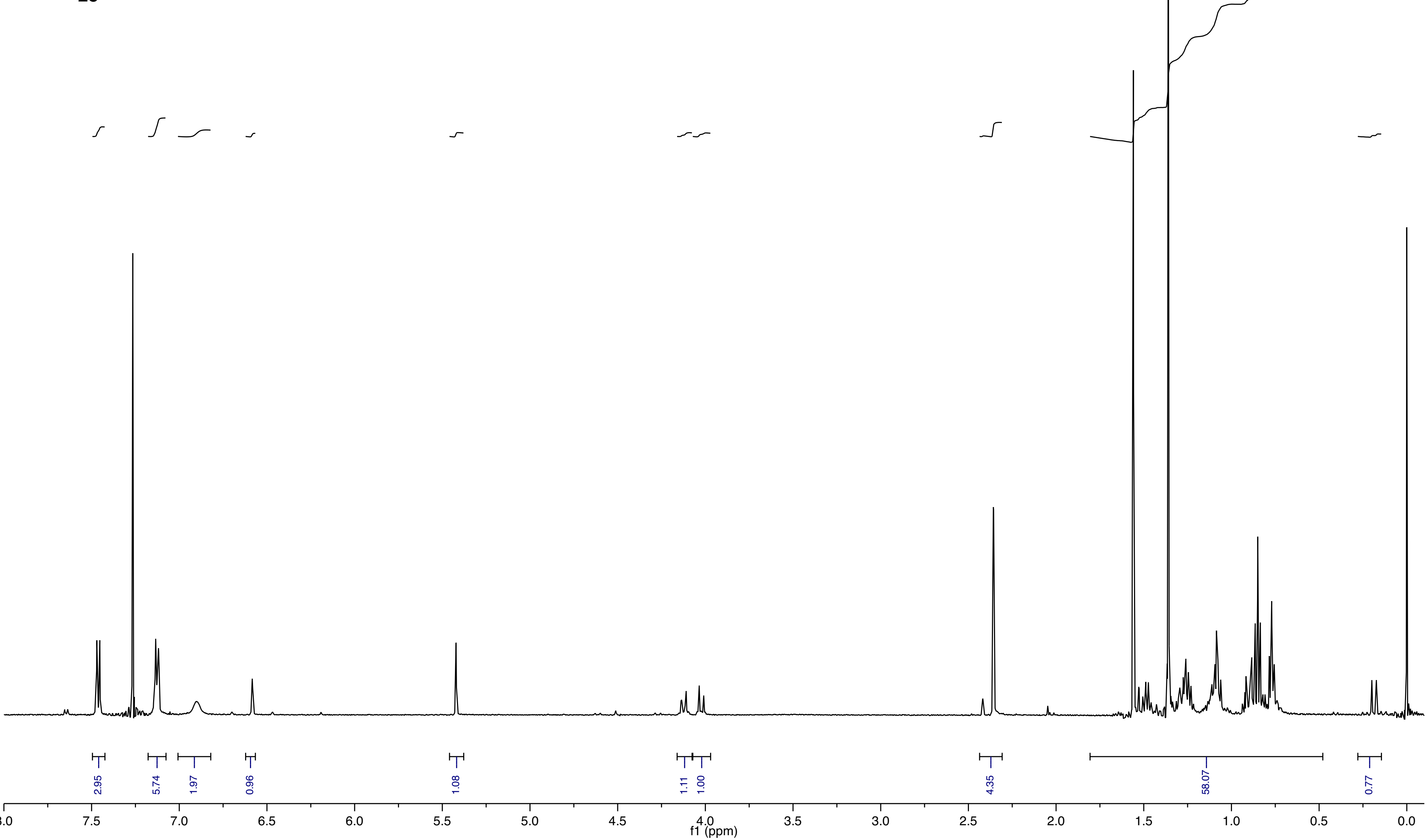
Sn
CO2tBu
N
Ts
Cl

2e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6.0
5.5
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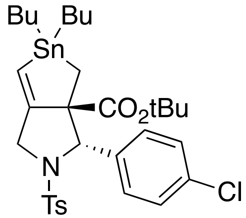
0.5
0.0
f1 (ppm)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Sn
CO_2tBu
N
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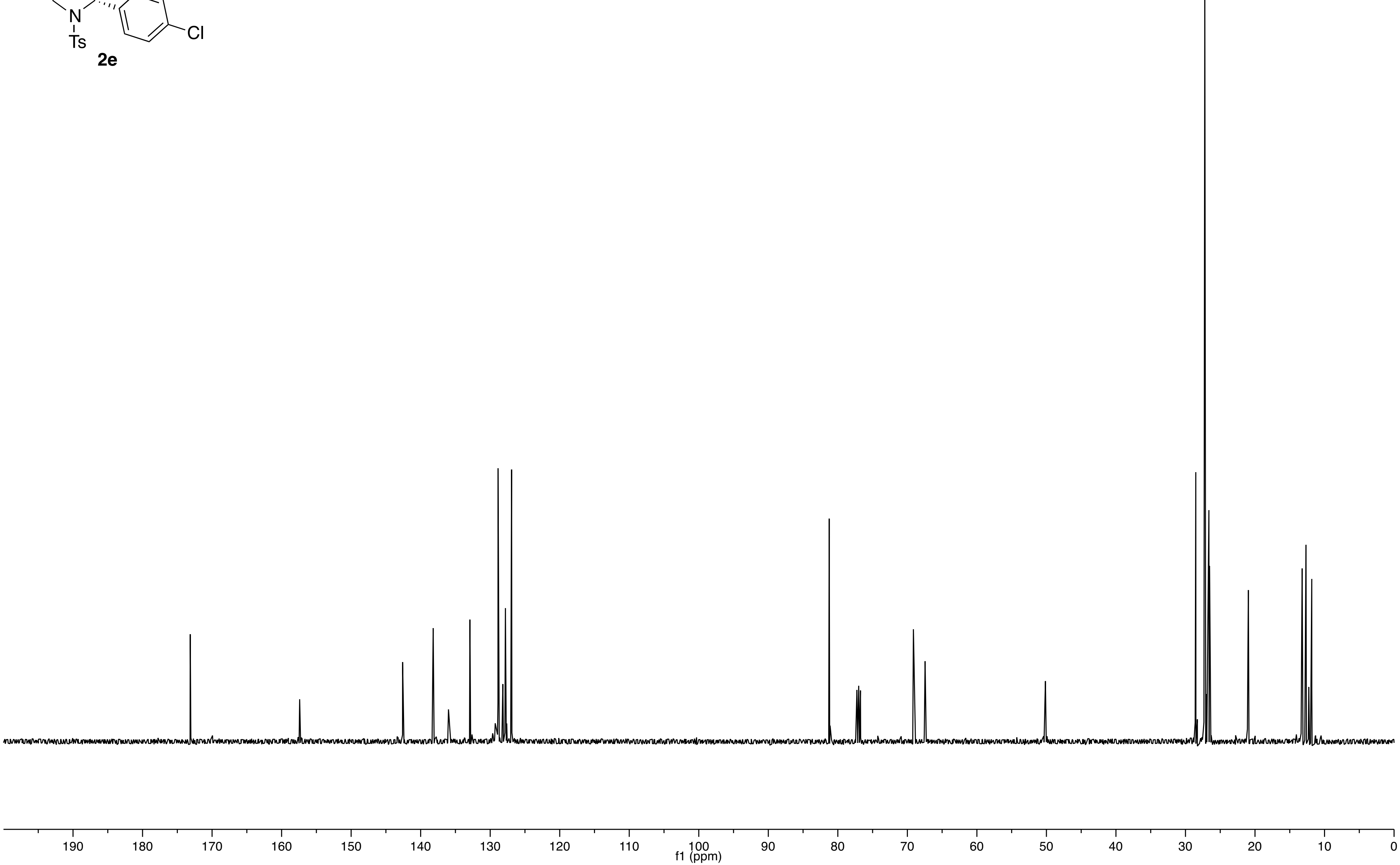
Cl

2e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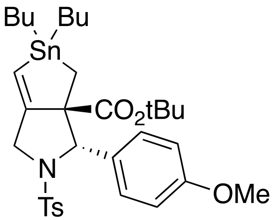
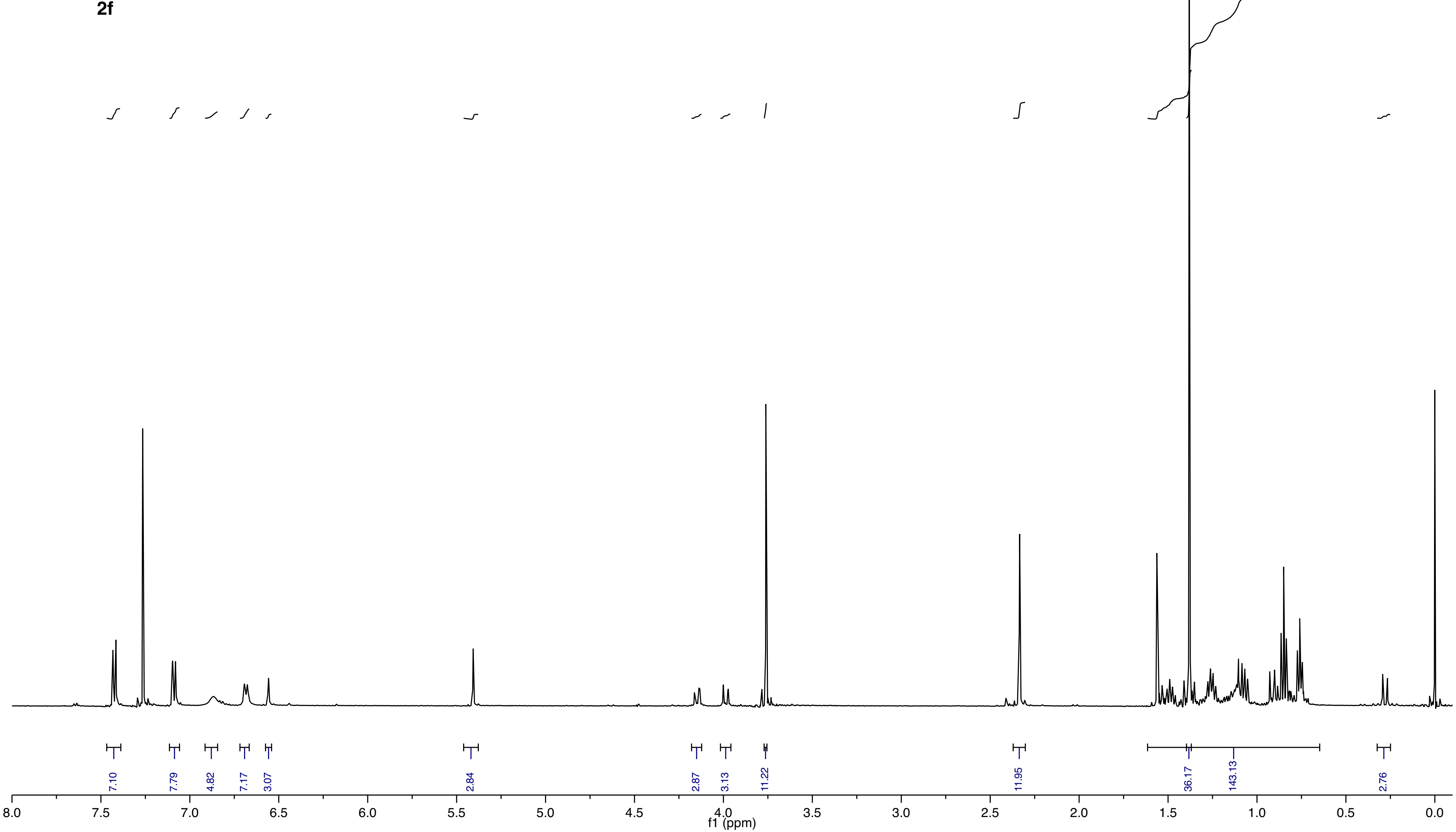
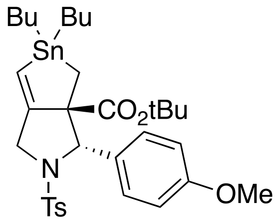
f1 (ppm)

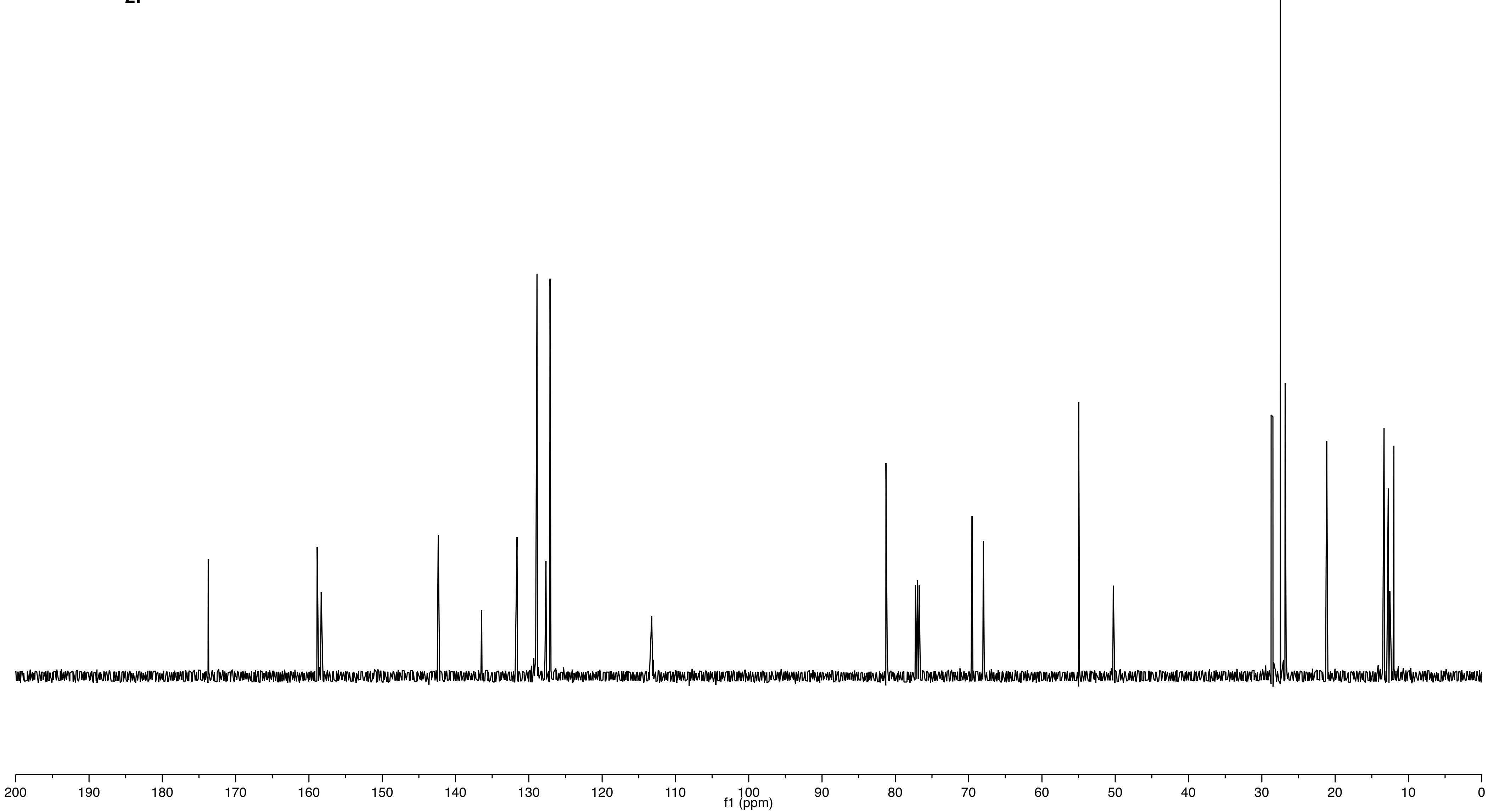

Bu Bu
Sn
CO2tBu
N
Ts
OMe

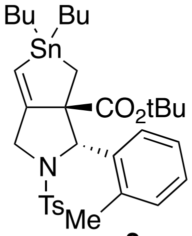
2f


7.10
7.79
4.82
7.17
3.07
2.84
2.87
3.13
11.22
11.95
36.17
143.13
2.76
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
0.0
f1 (ppm)

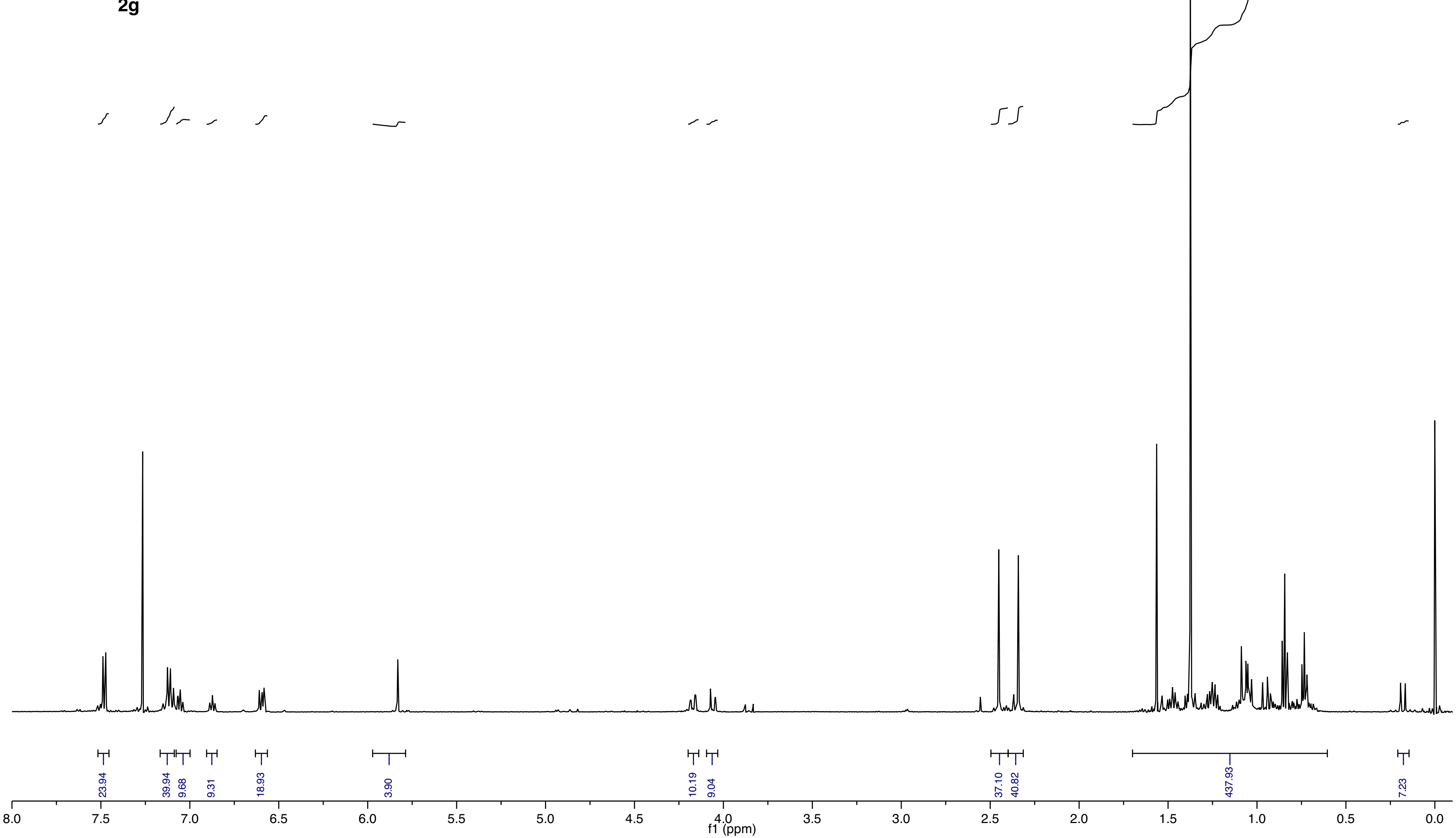

Bu
Bu
Sn
CO2tBu
N
Ts
OMe

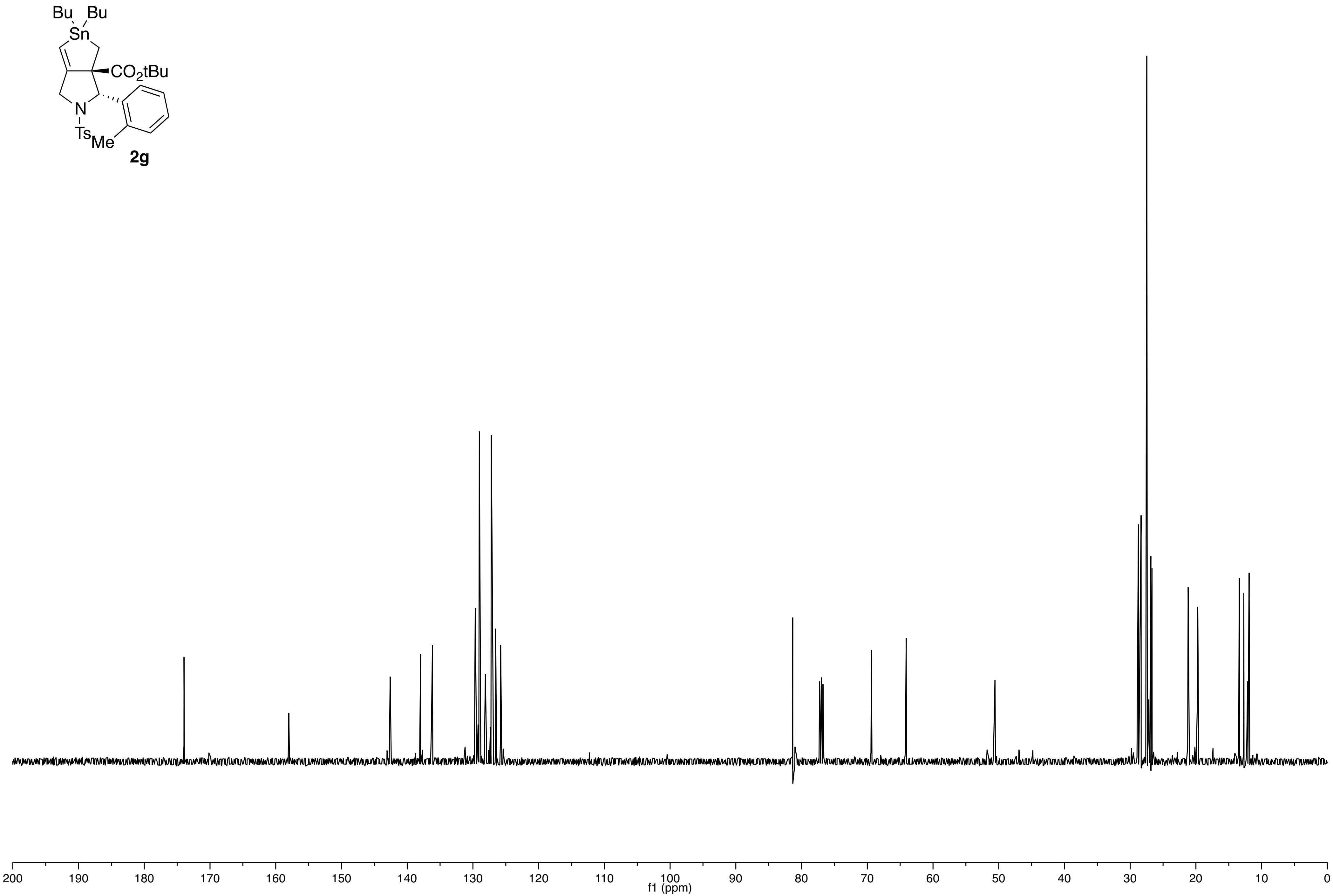
2f


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

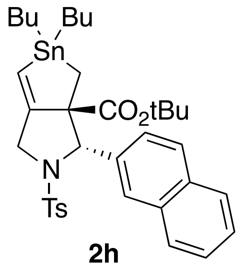


2g

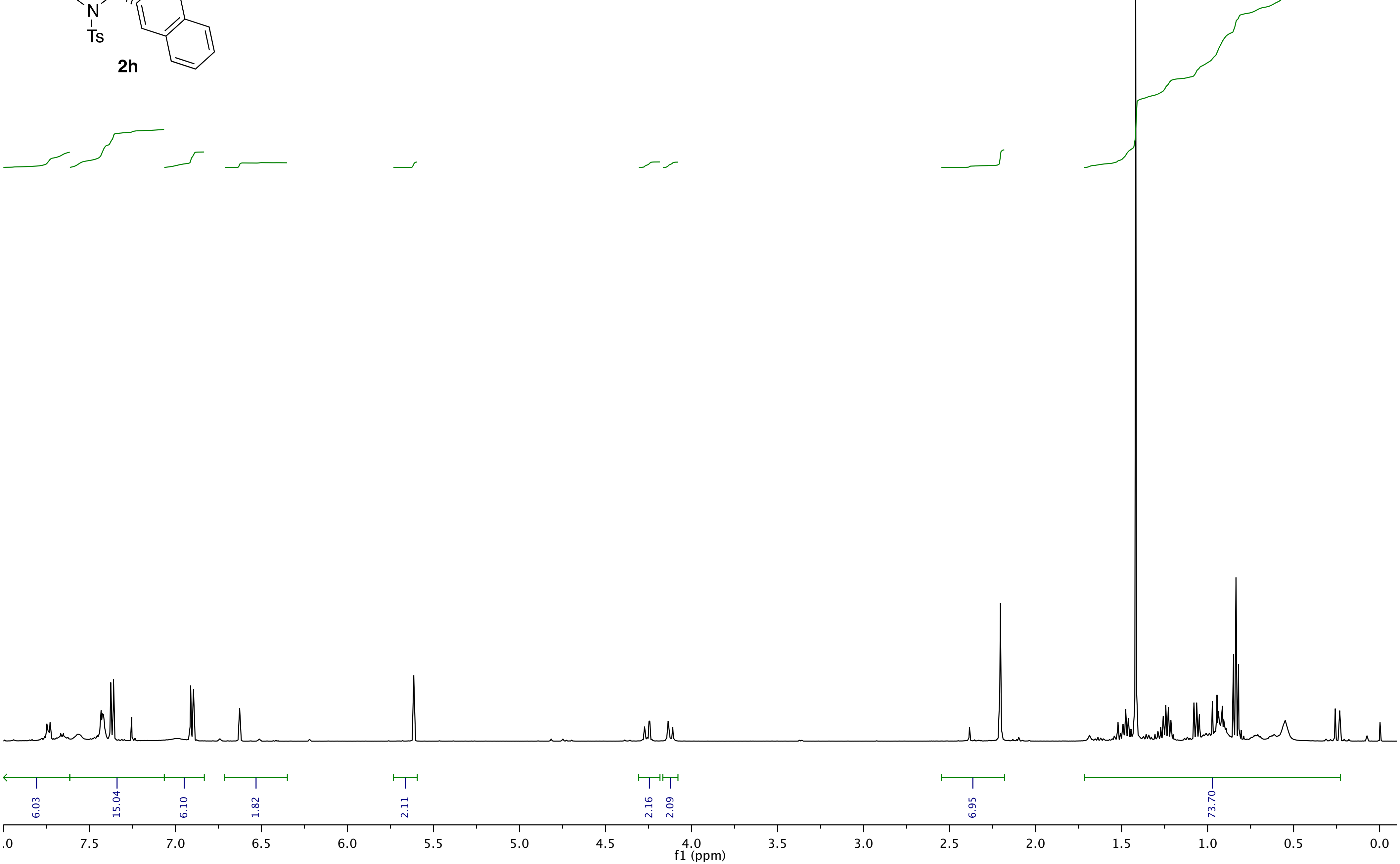

23.94
39.94
9.68
9.31
18.93
3.90
10.19
9.04
37.10
40.82
437.93
7.23
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

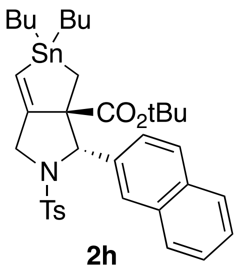


Bu
Bu
Sn
CO2tBu
N
Ts
Me
2g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

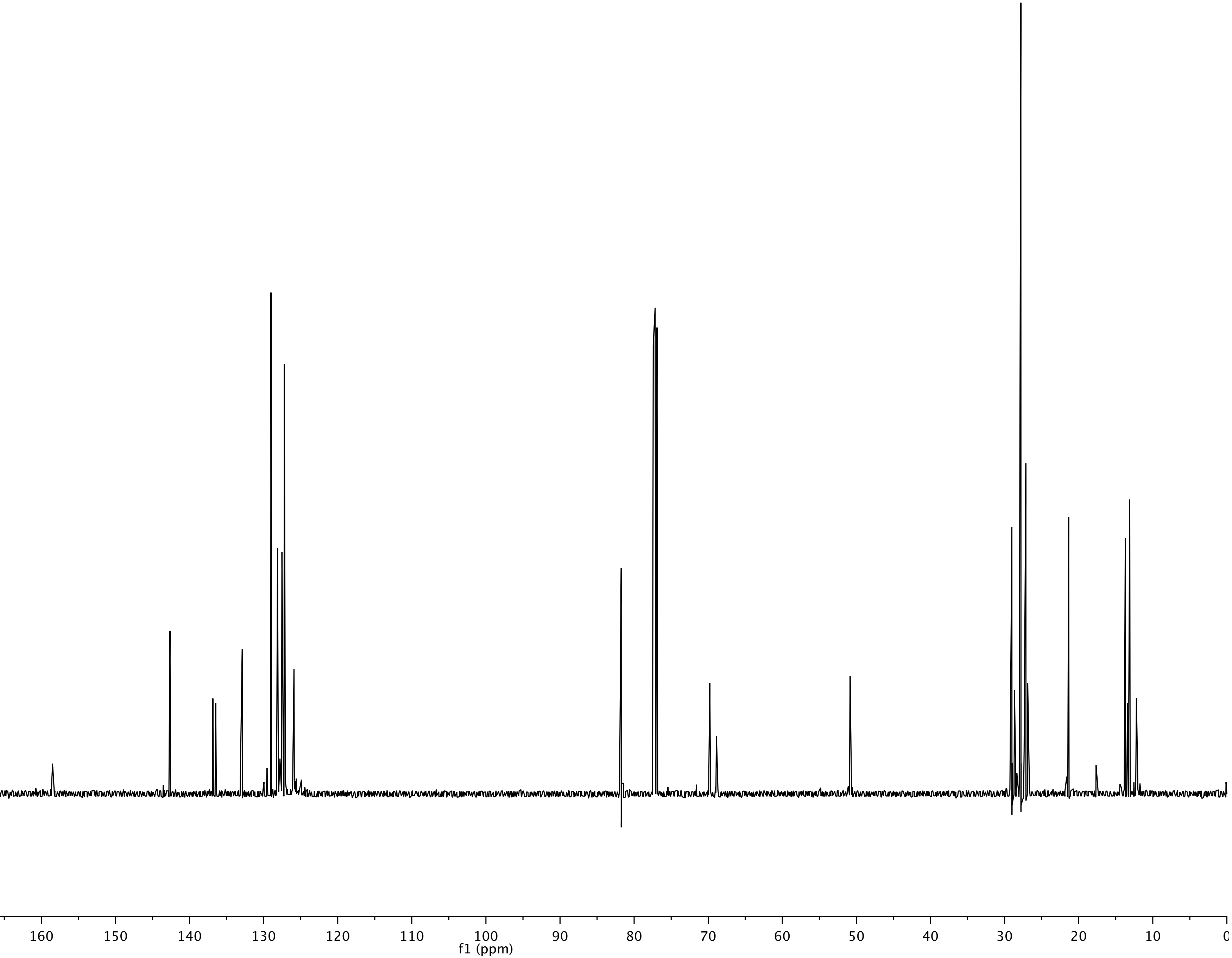

Bu
Bu
Sn
CO2tBu
N
Ts

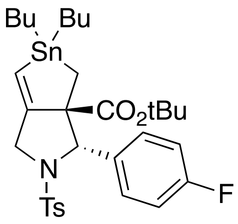
2h


6.03
15.04
6.10
1.82
2.11
2.16
2.09
6.95
73.70
.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
0.0
f1 (ppm)

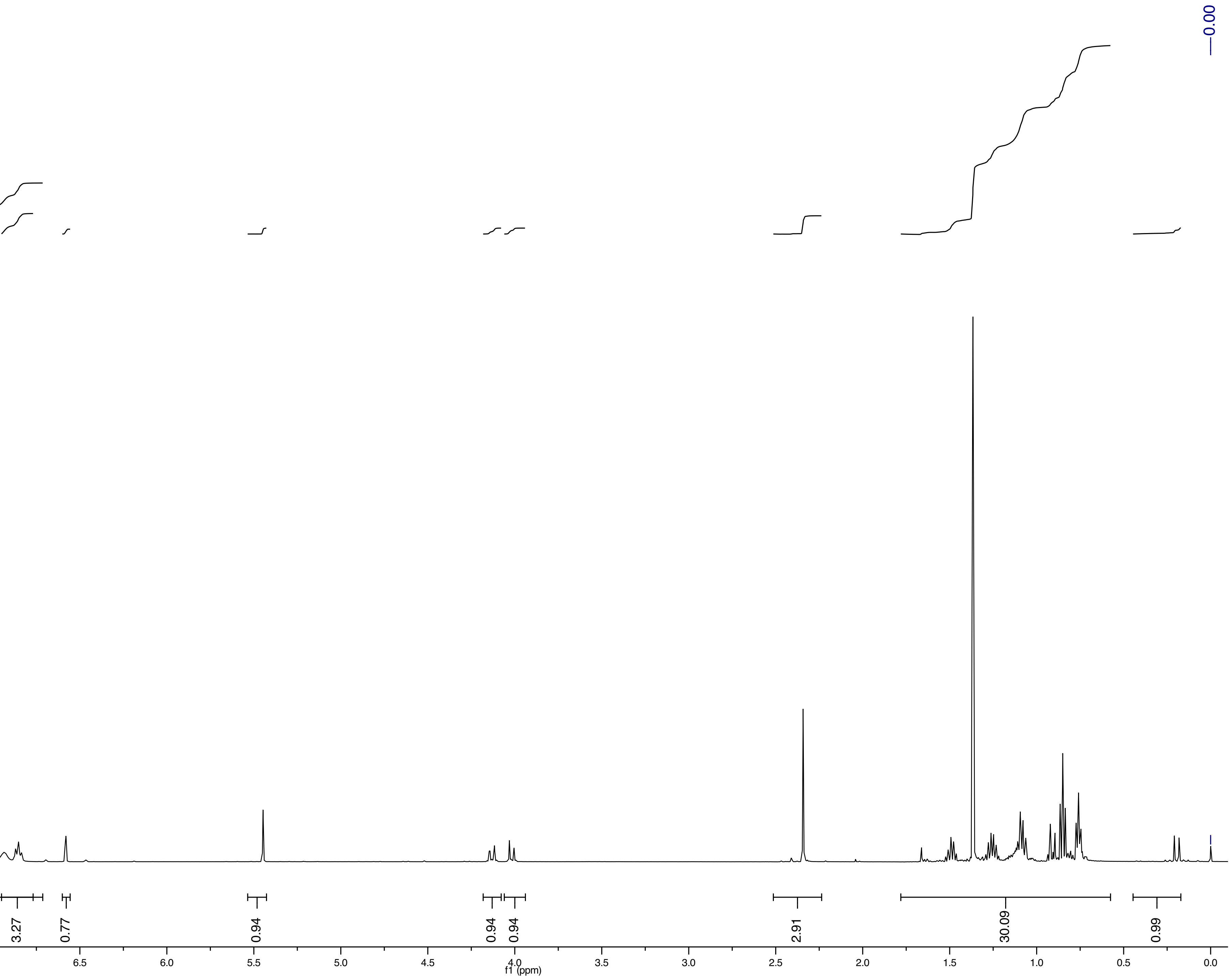

Bu
Bu
Sn
CO2tBu
N
Ts

2h

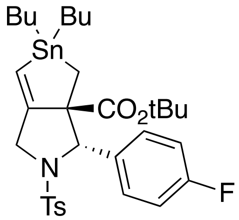

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



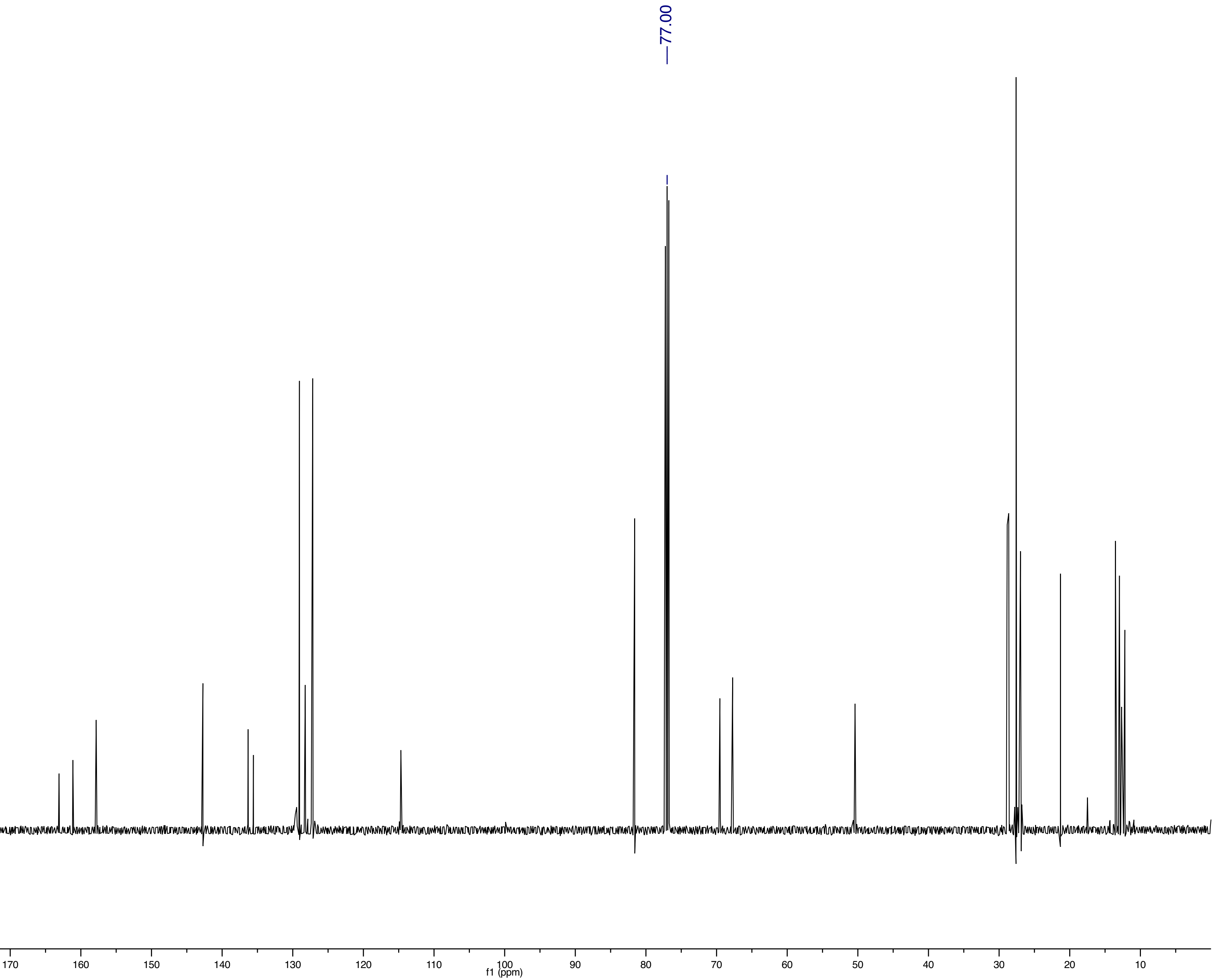
2i

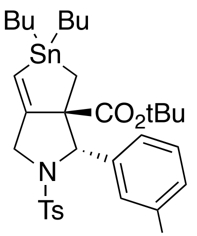


0.00
8.14
3.27
0.77
0.94
0.94
0.94
2.91
30.09
0.99
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

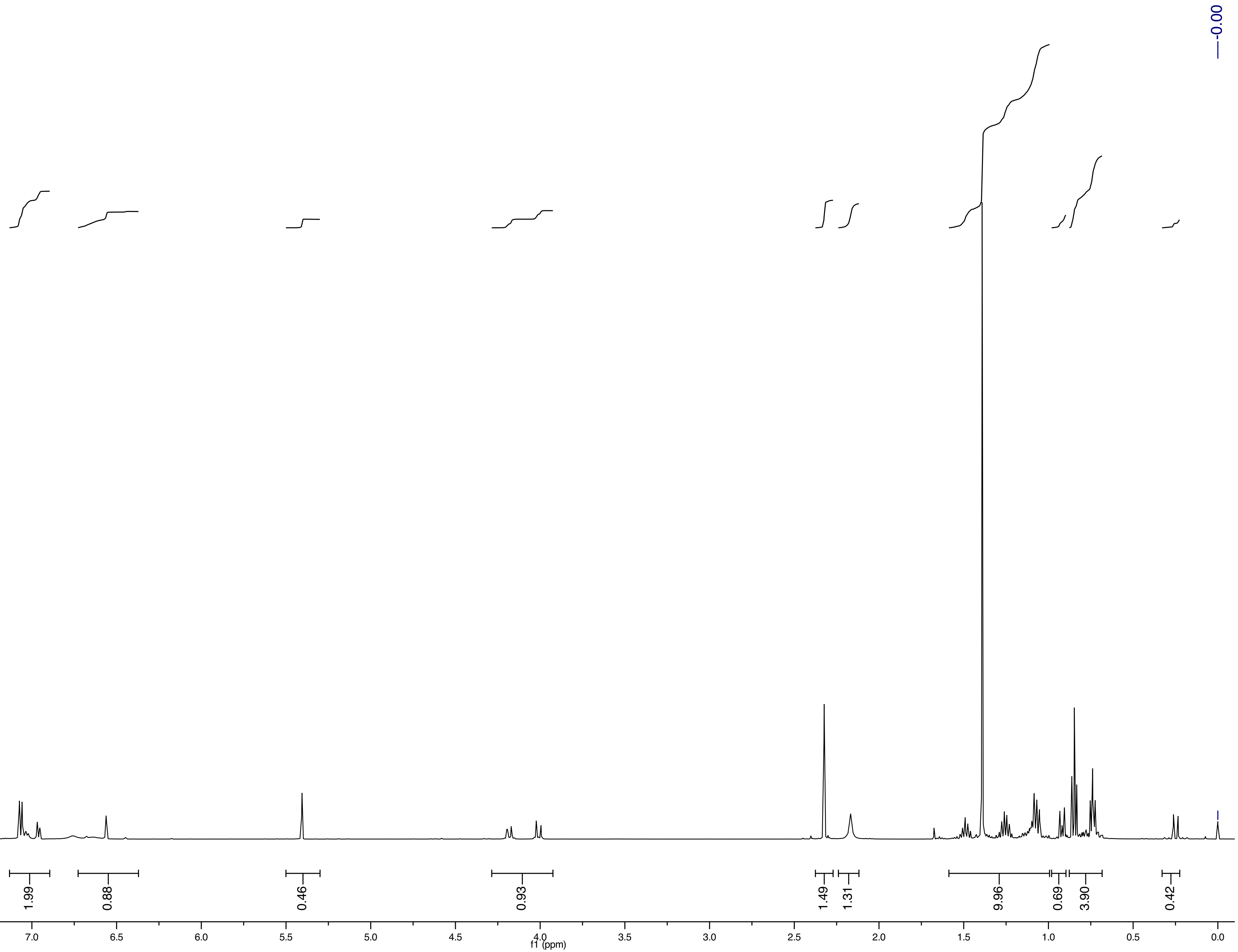

Bu
Bu
Sn
CO2tBu
N
Ts
F

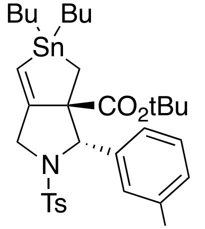
2i


77.00
200
190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10

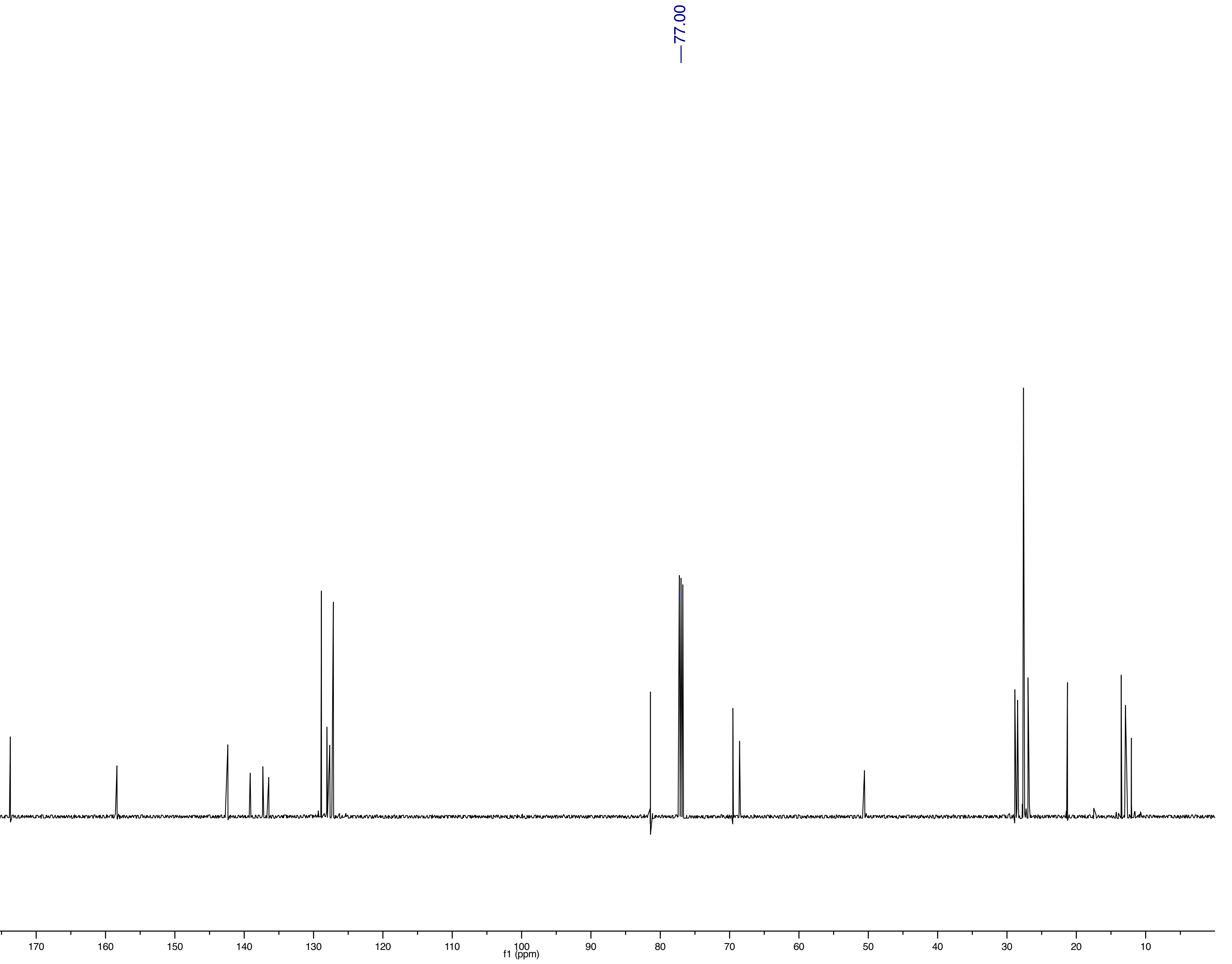

Bu
Bu
Sn
CO2tBu
N
Ts
Me

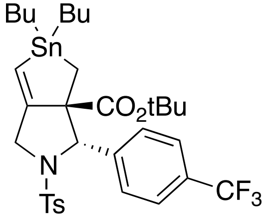
2j


-0.00
1.00
1.99
0.88
0.46
0.93
1.49
1.31
9.96
0.69
3.90
0.42
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
0.0
f1 (ppm)

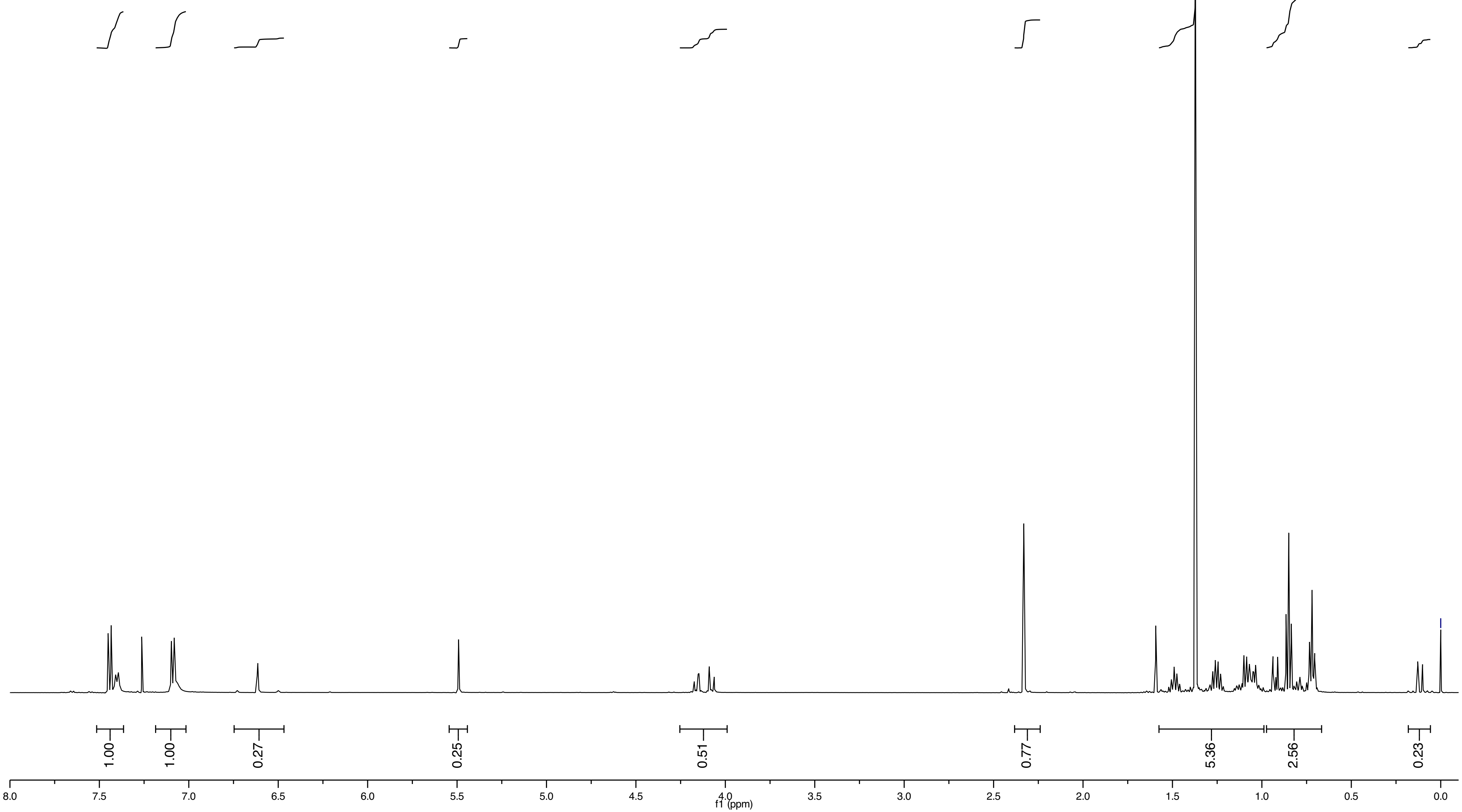

Bu
Bu
Sn
CO2tBu
N
Ts
Me

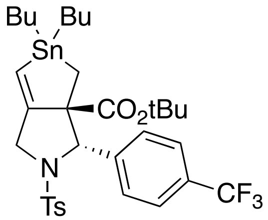
2j


77.00
200
190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10

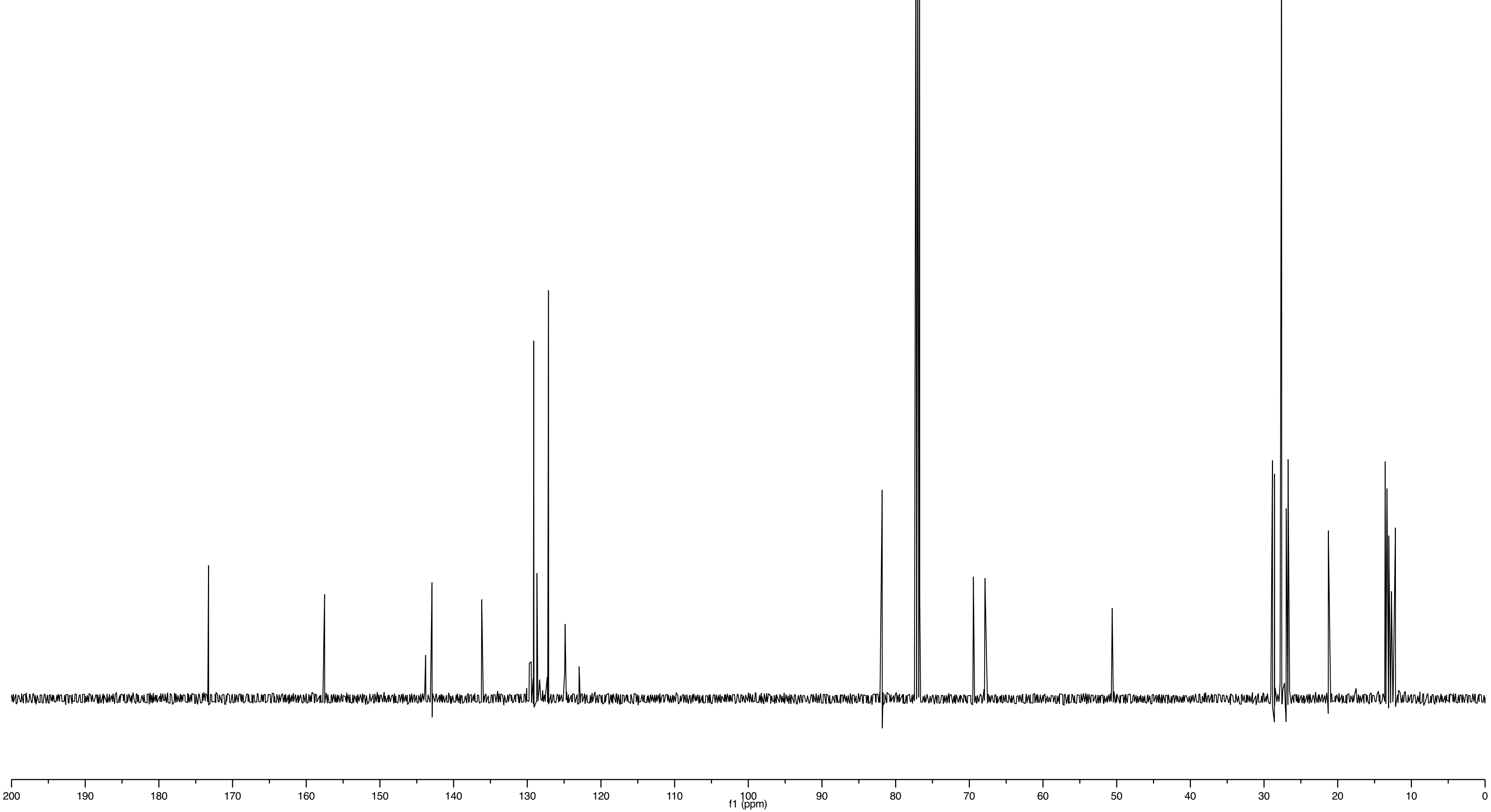

Bu
Bu
Sn
CO_2tBu
N
Ts
CF_3

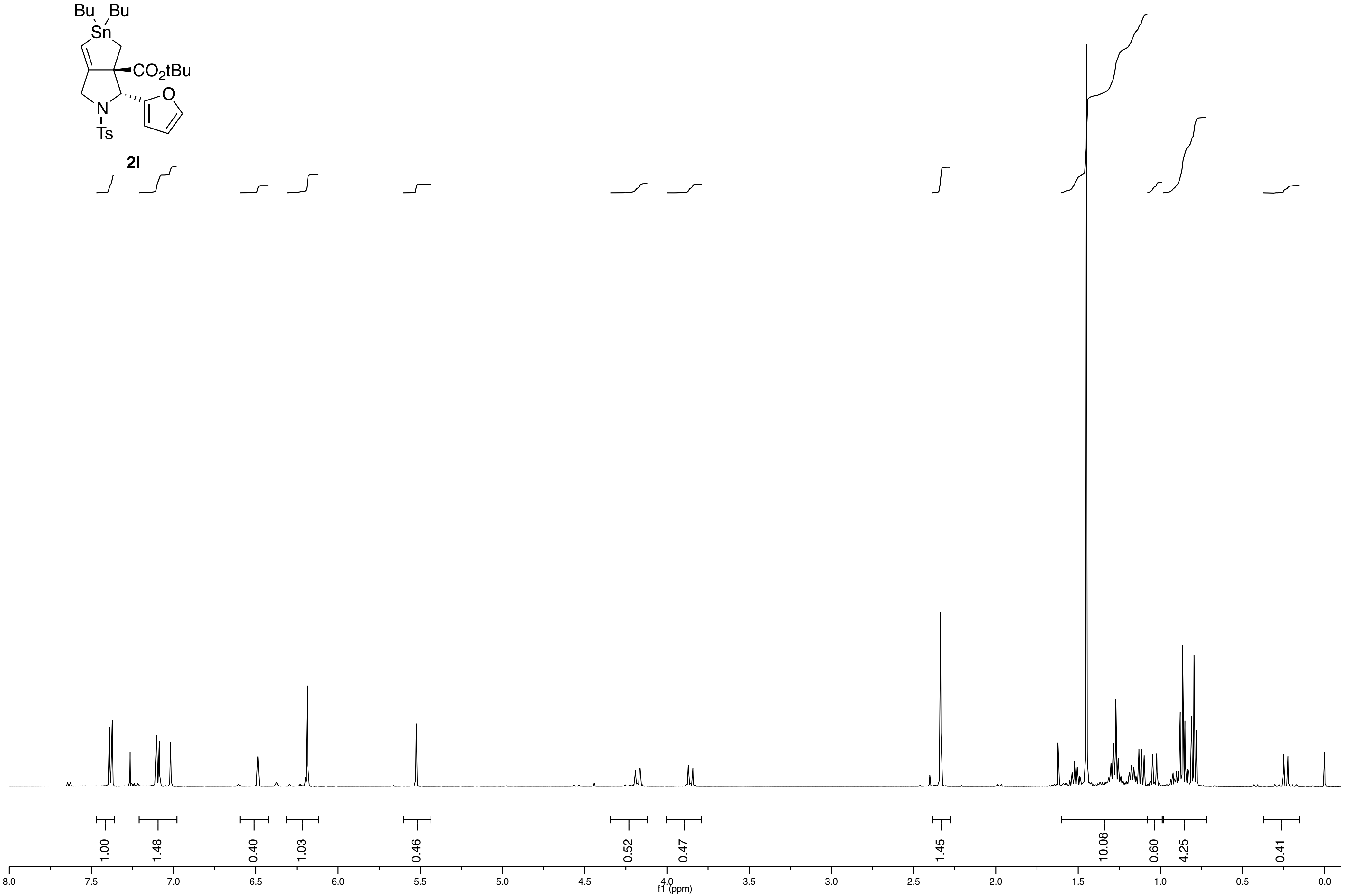
2k


0.00
1.00
1.00
0.27
0.25
0.51
0.77
5.36
2.56
0.23
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
0.0
f1 (ppm)

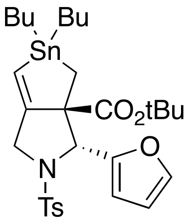

Bu
Bu
Sn
CO2tBu
N
Ts
CF3

2k

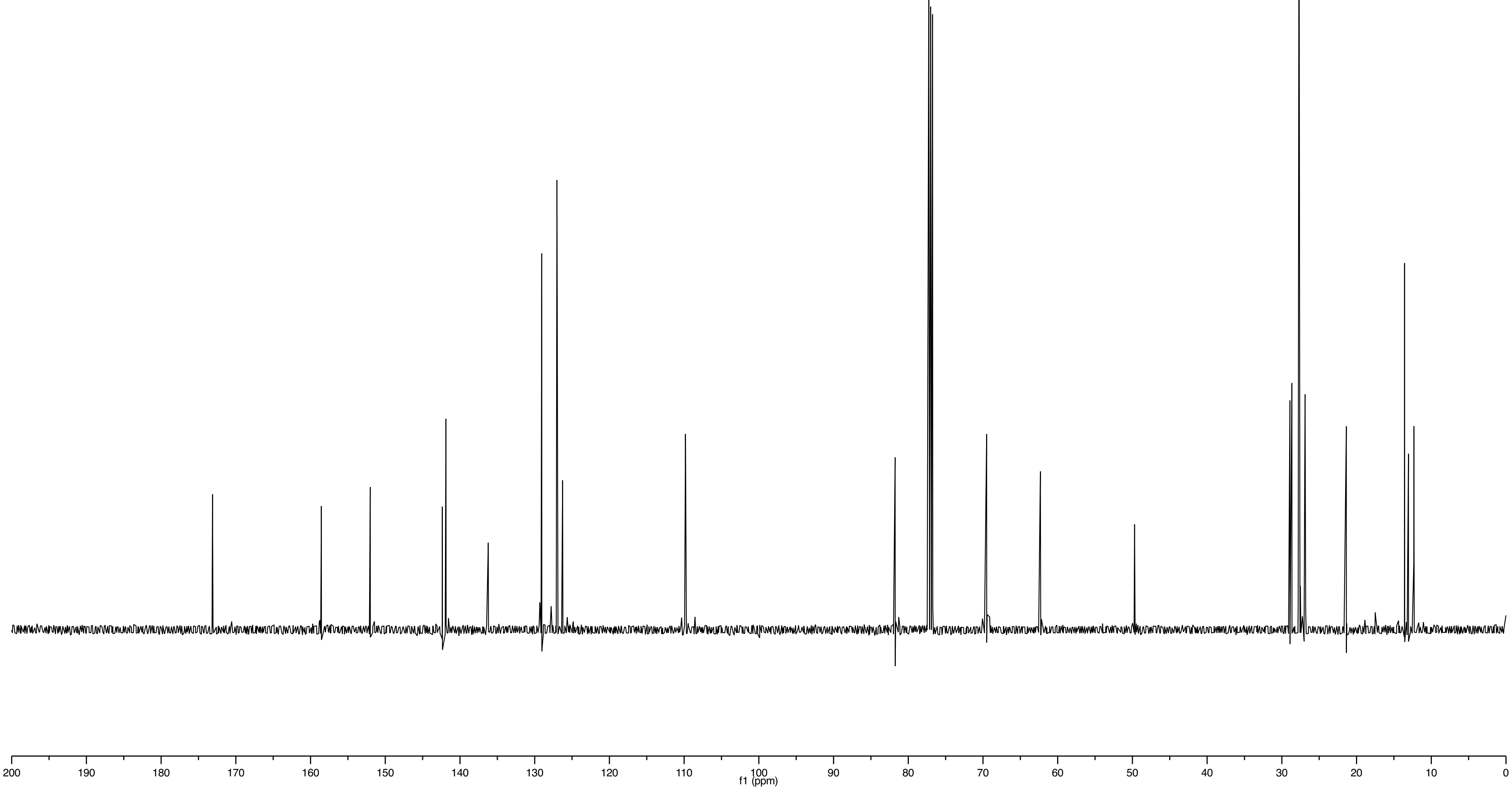

77.00
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

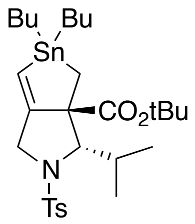


Bu Bu
Sn
CO2tBu
O
N
Ts
2l
1.00
1.48
0.40
1.03
0.46
0.52
0.47
1.45
10.08
0.60
4.25
0.41
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

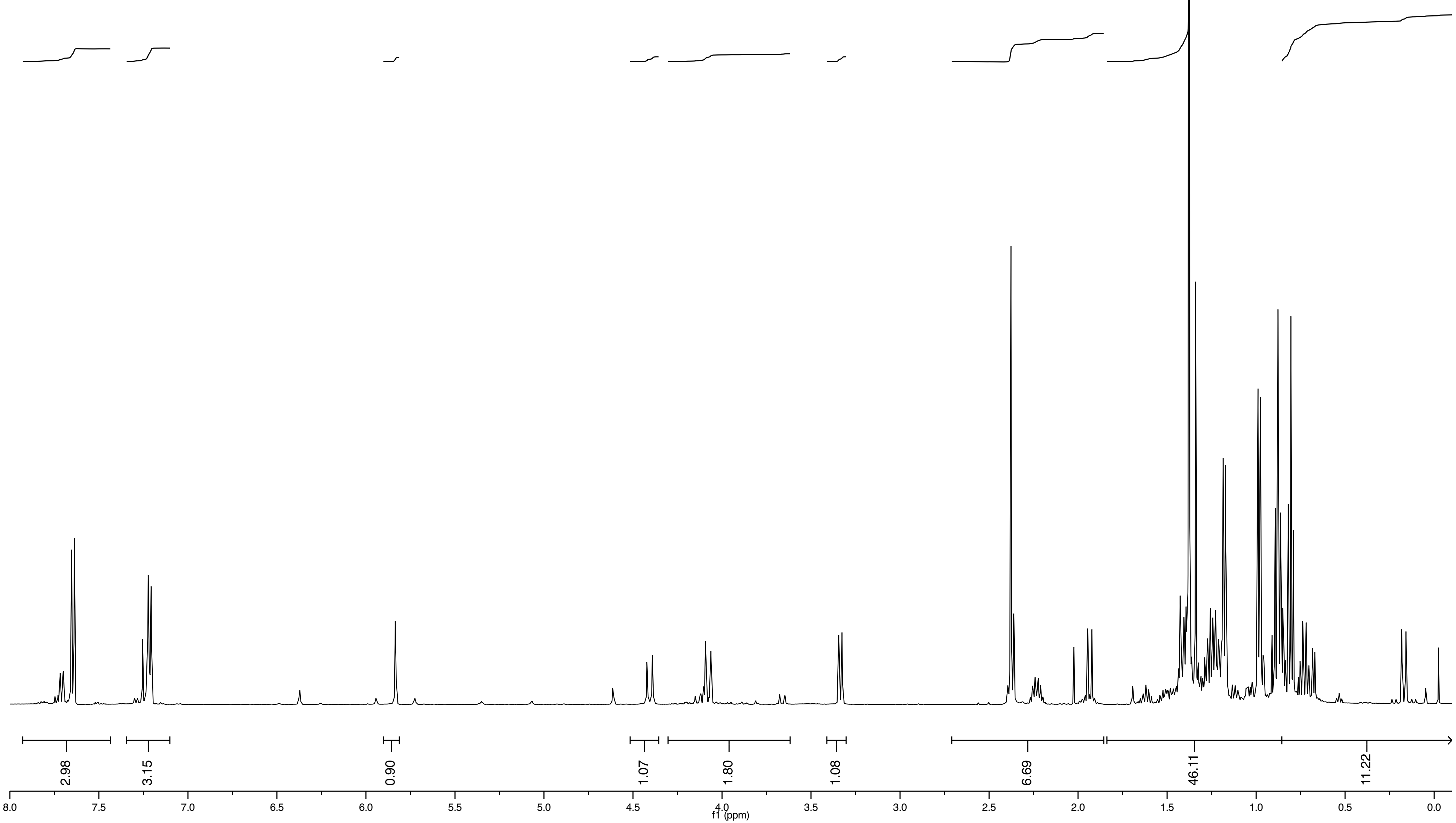

Bu Bu
Sn
CO2tBu
O
N
Ts

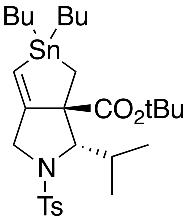
2l


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

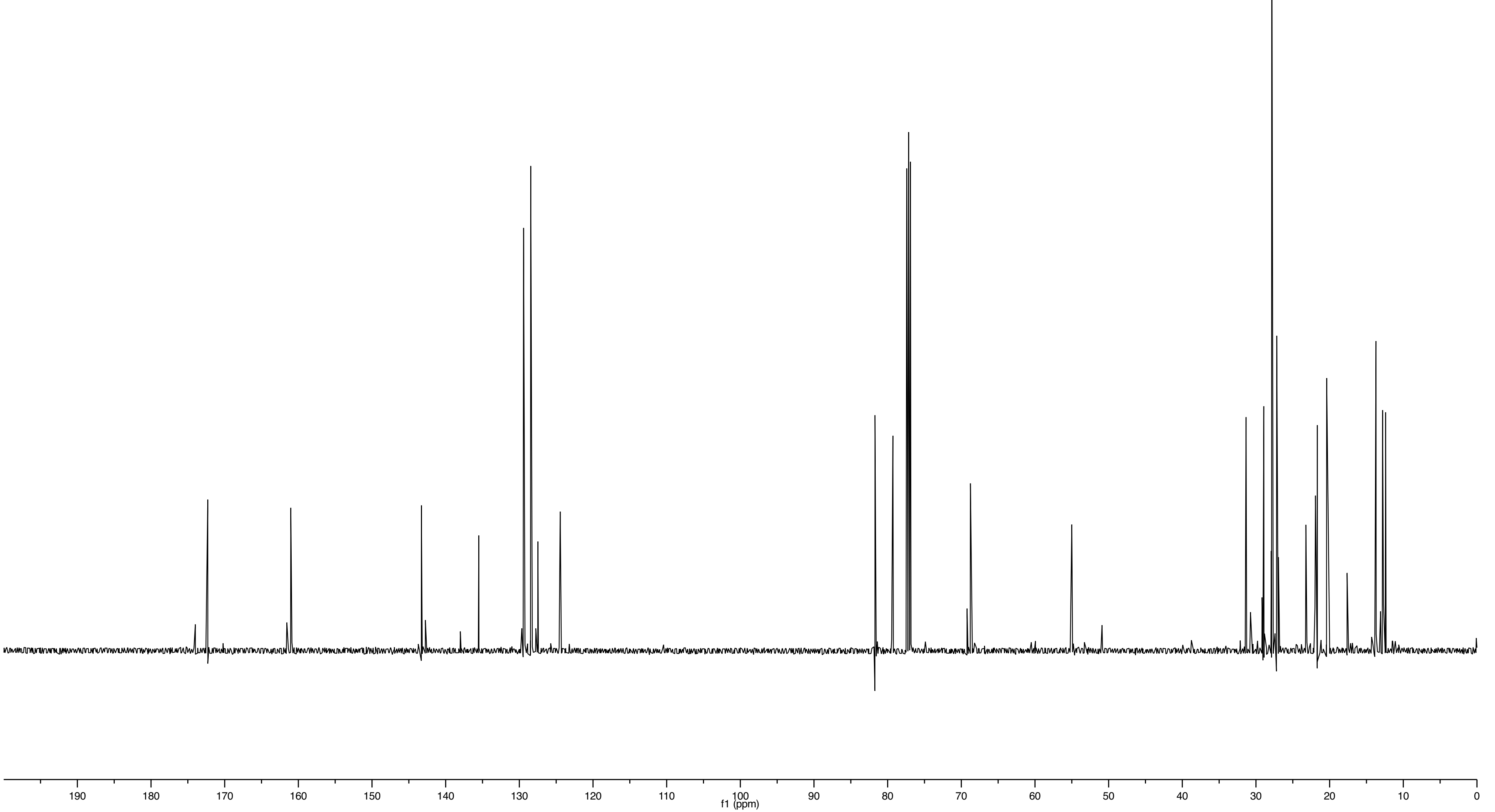


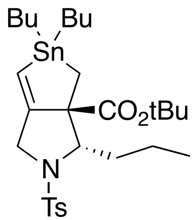
2m


2.98
3.15
0.90
1.07
1.80
1.08
6.69
46.11
11.22
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
0.0
f1 (ppm)

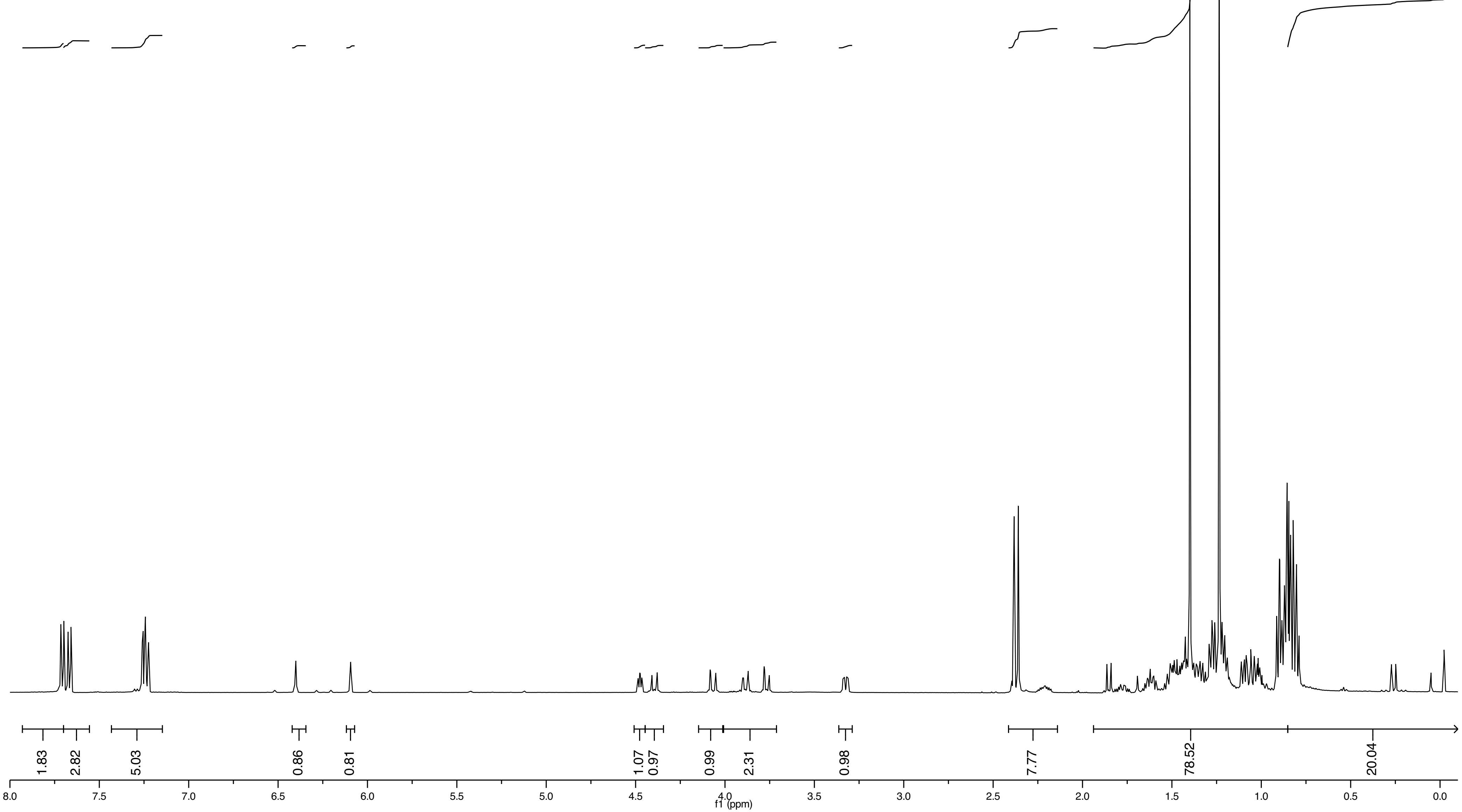

Bu
Bu
Sn
CO2tBu
N
Ts

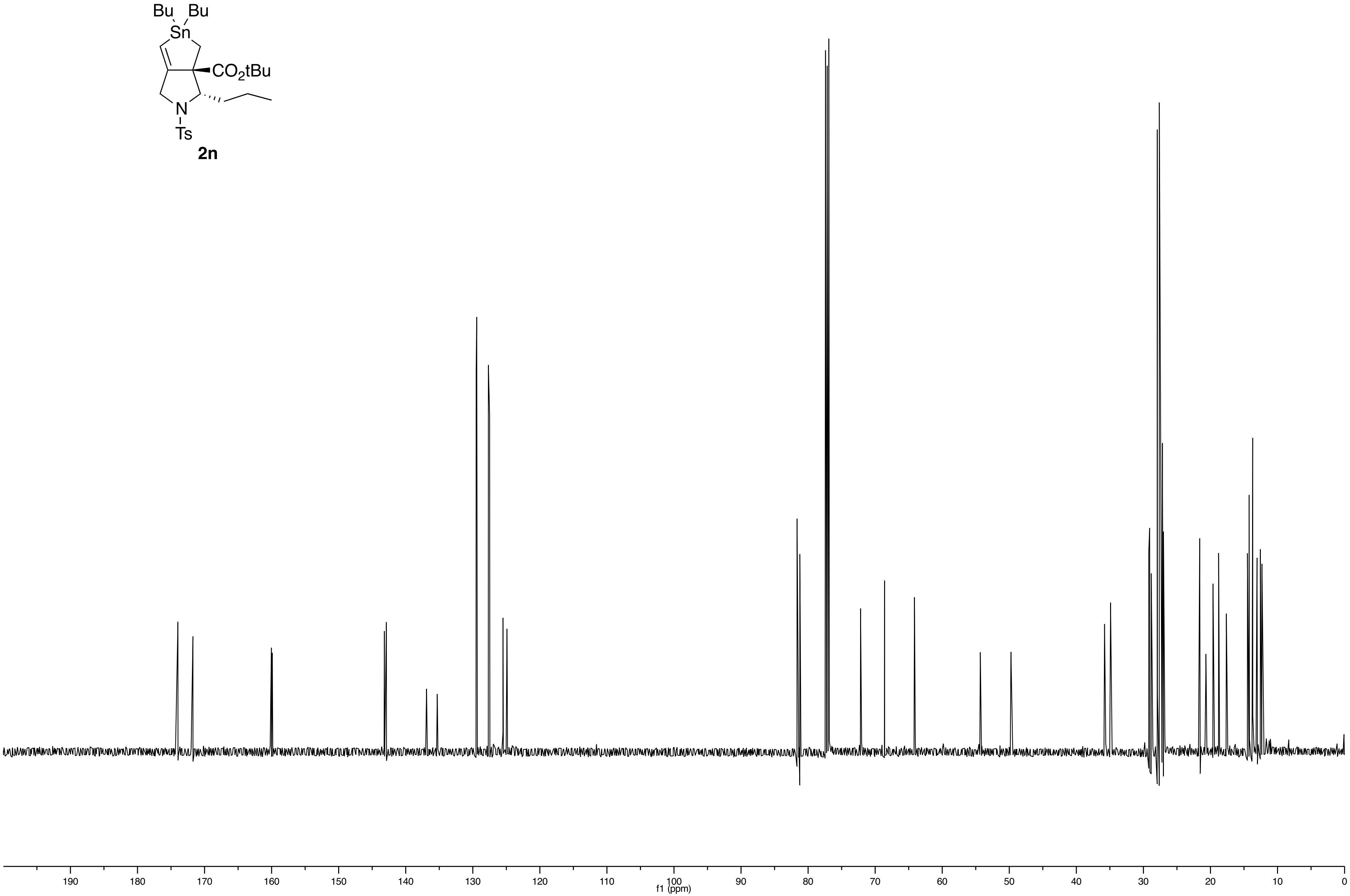
2m

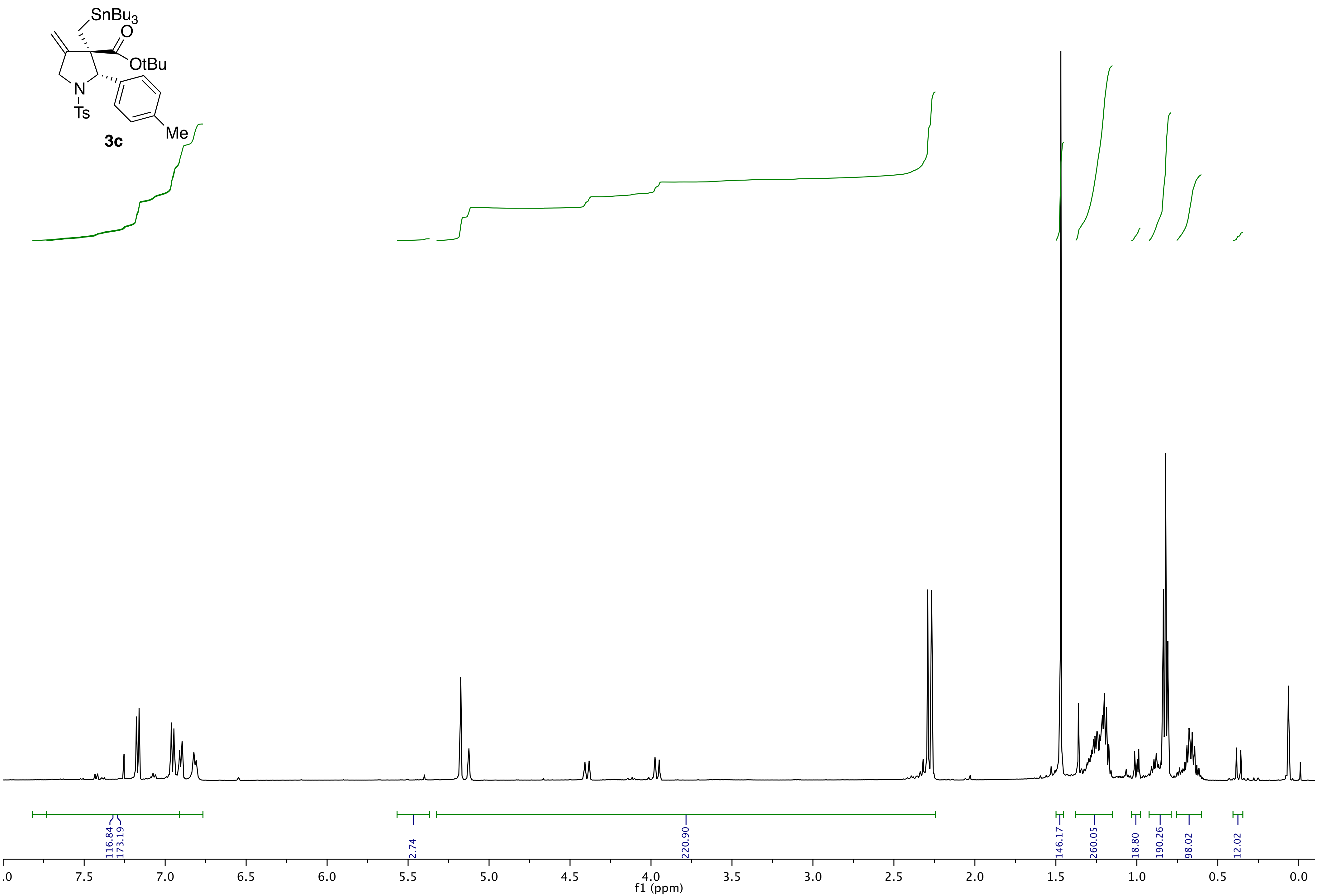

190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10
0

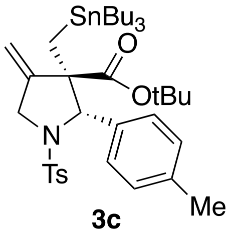

Bu
Bu
Sn
CO2tBu
N
Ts

2n

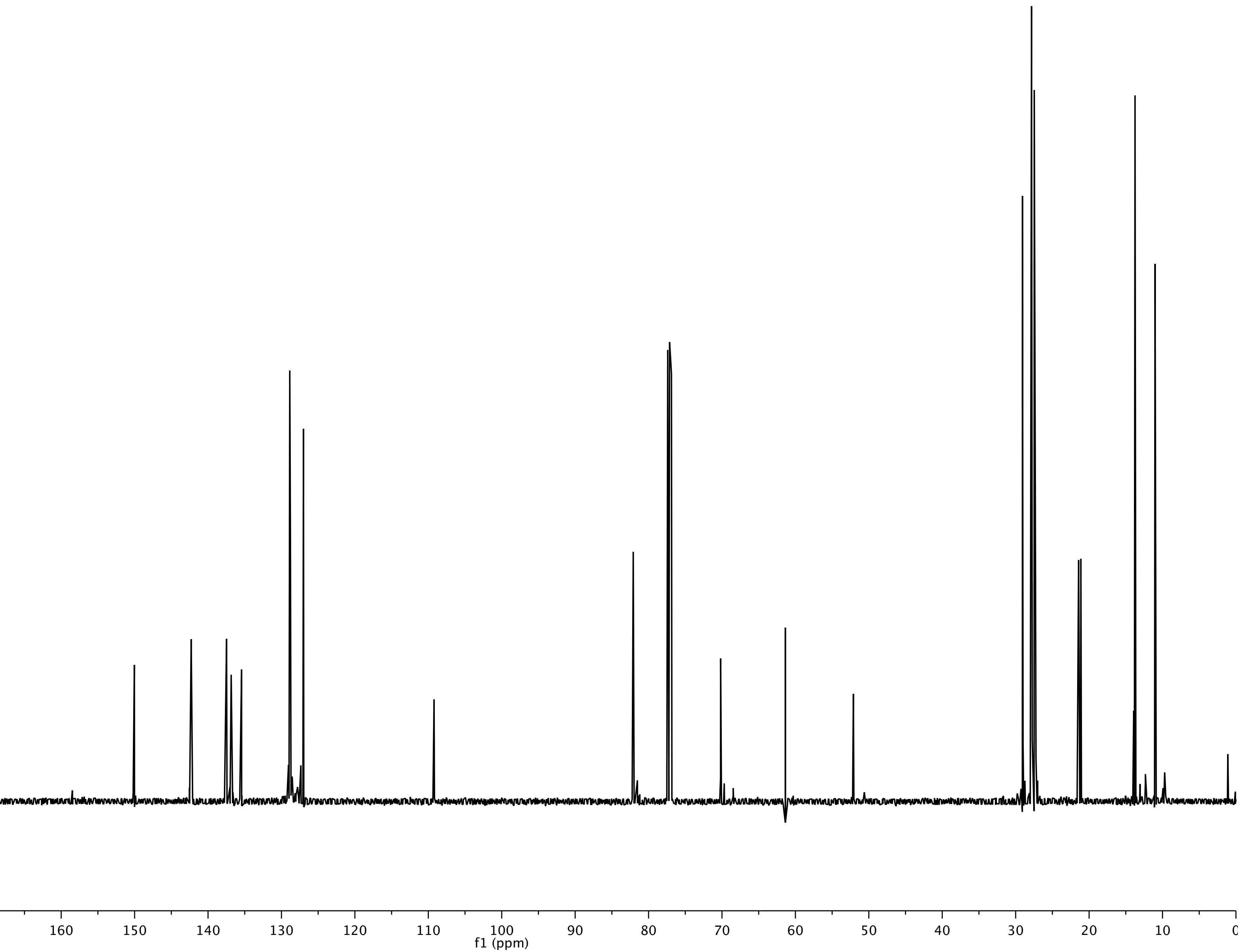

1.83
2.82
5.03
0.86
0.81
1.07
0.97
0.99
2.31
0.98
7.77
78.52
20.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

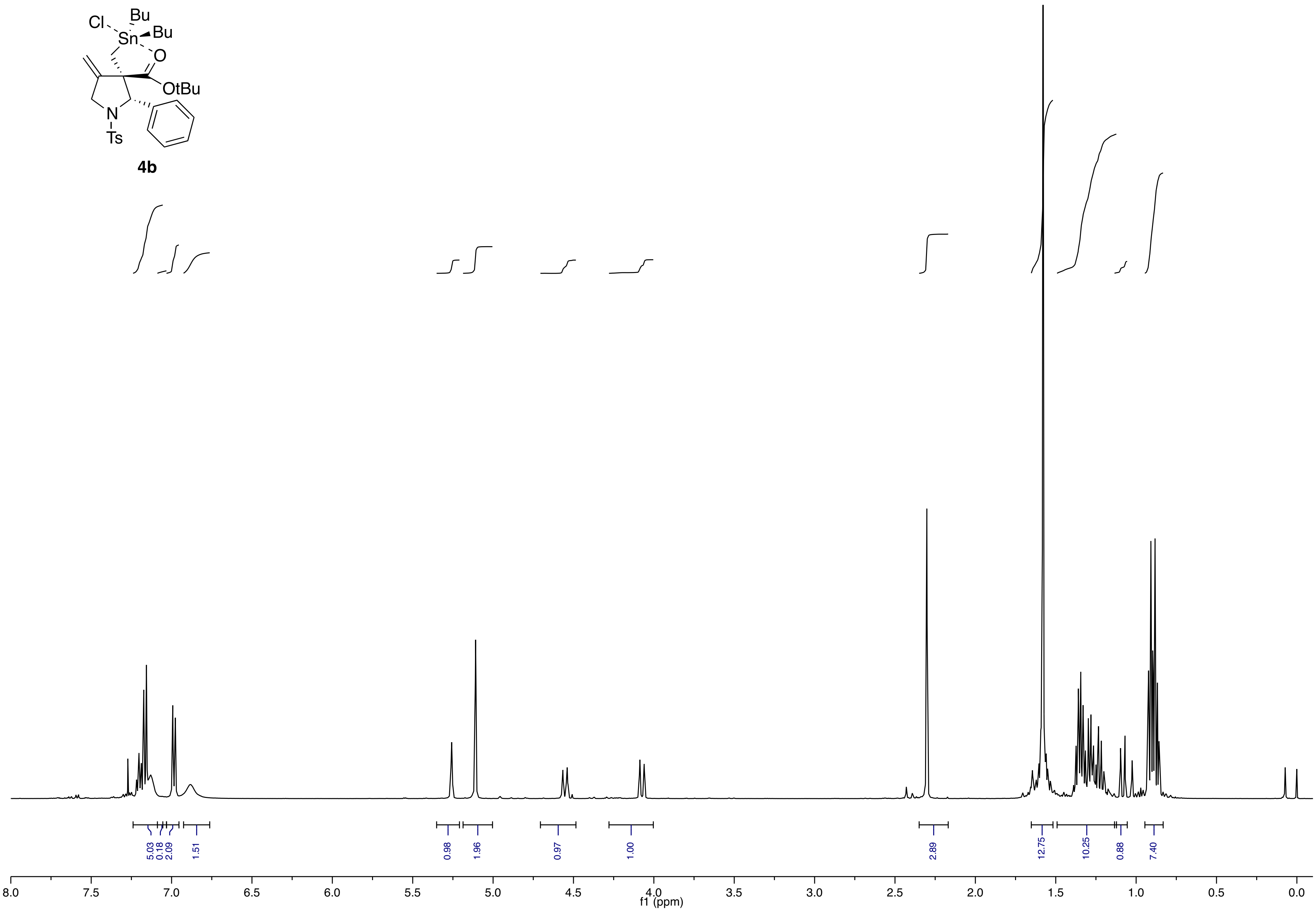

Bu
Bu
Sn
CO2tBu
N
Ts
2n
190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10
0

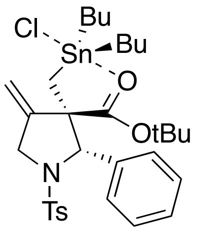

SnBu3
O
OtBu
N
Ts
Me
3c
116.84
173.19
2.74
220.90
146.17
260.05
18.80
190.26
98.02
12.02
.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


SnBu3
O
OtBu
N
Ts
Me

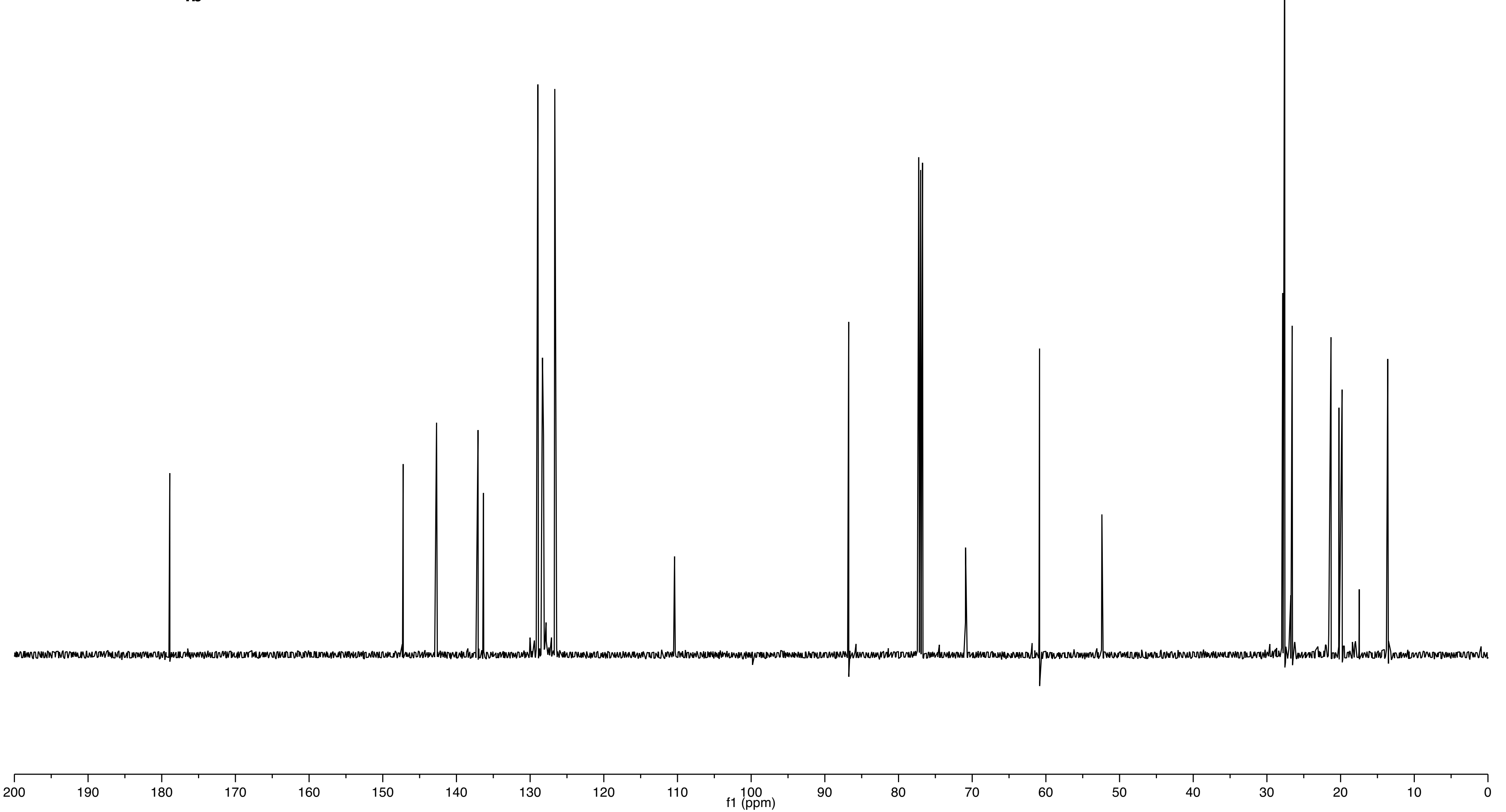
3c

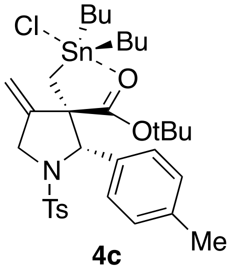

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Cl
Bu
Sn
Bu
O
OtBu
N
Ts
4b
5.03
0.18
2.09
1.51
0.98
1.96
0.97
1.00
2.89
12.75
10.25
0.88
7.40
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
0.0
f1 (ppm)

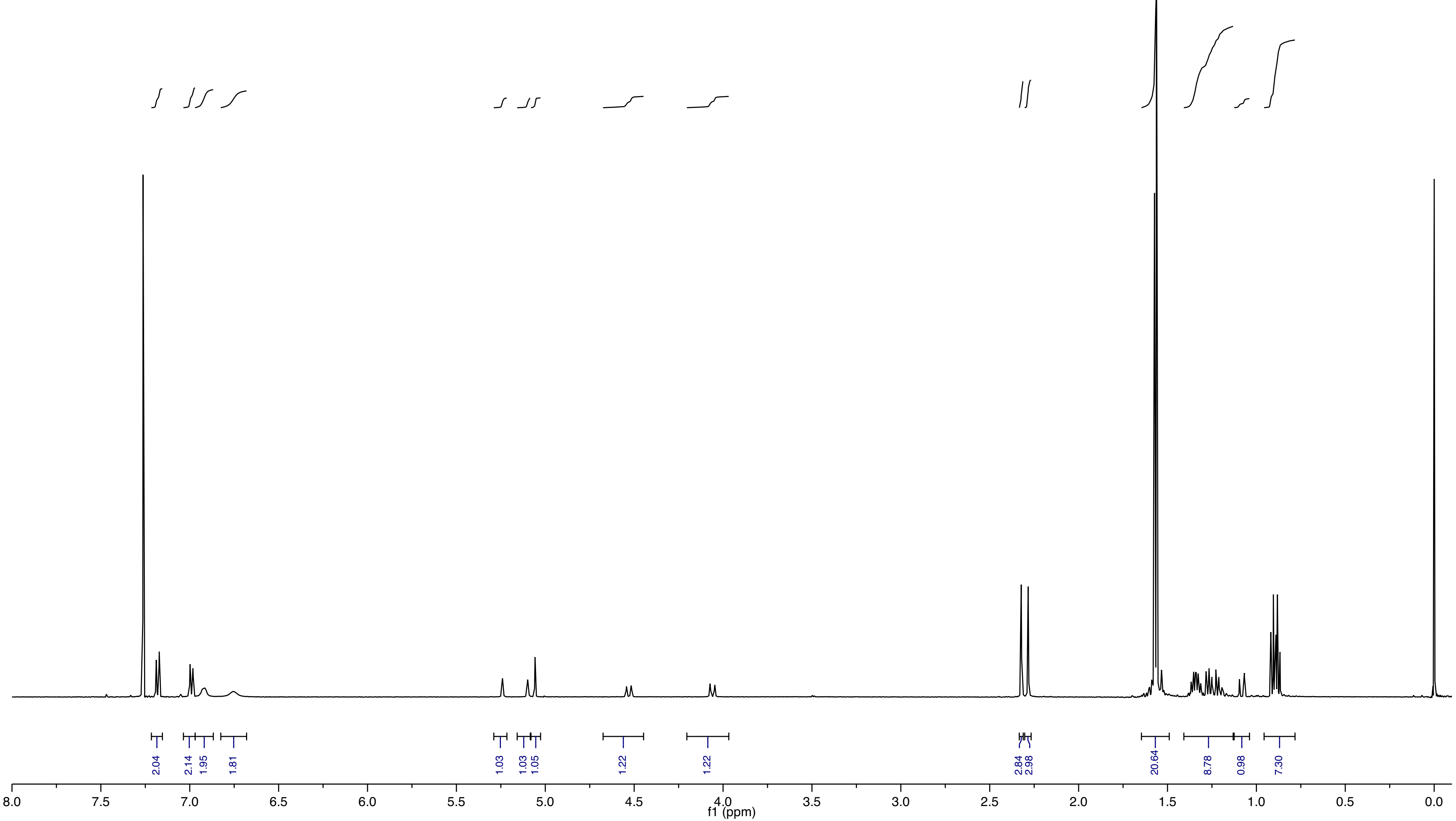

Cl
Bu
Sn
Bu
O
OtBu
N
Ts

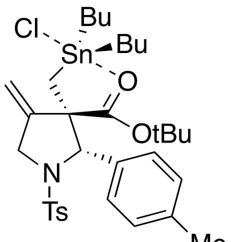
4b


200
190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10
0

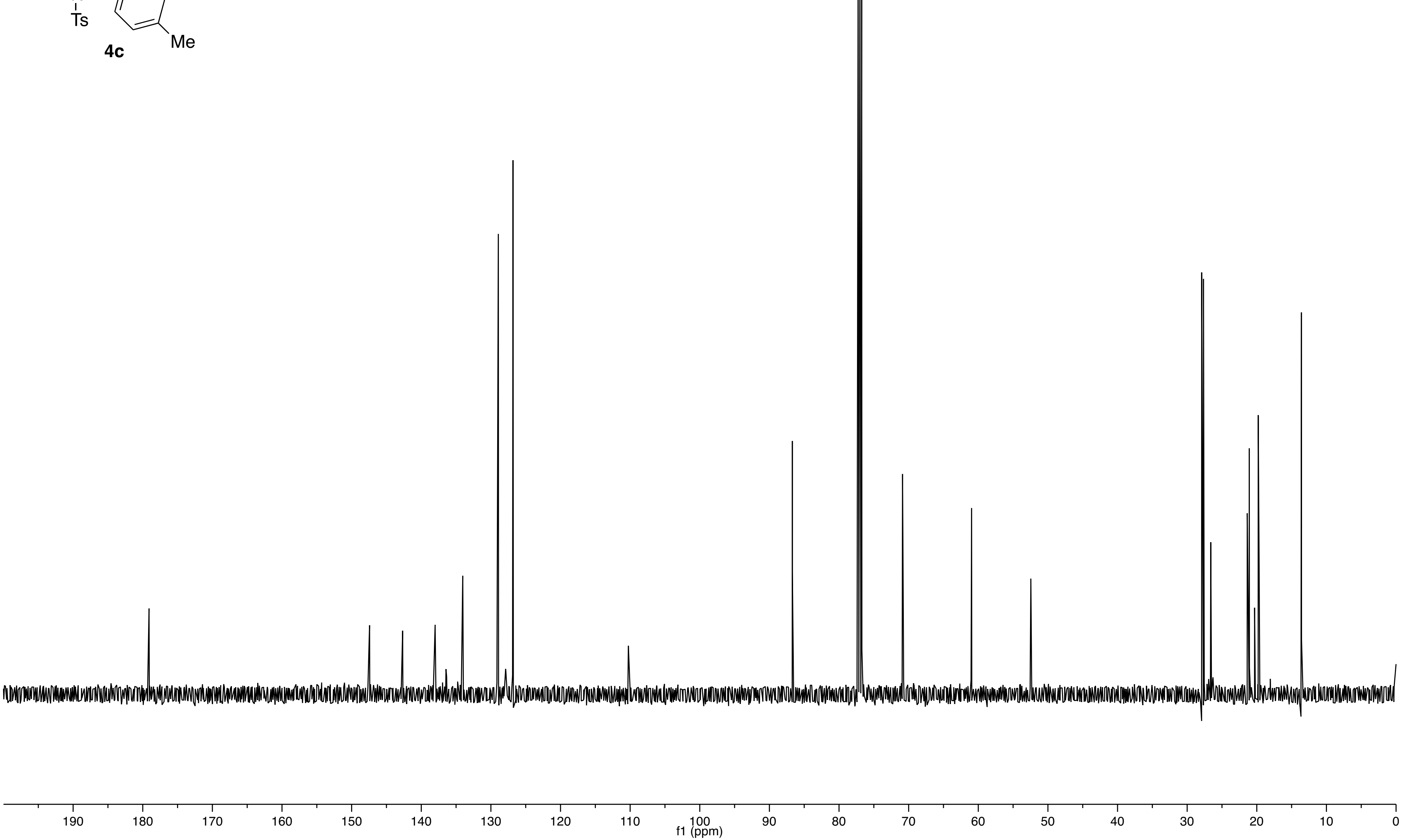


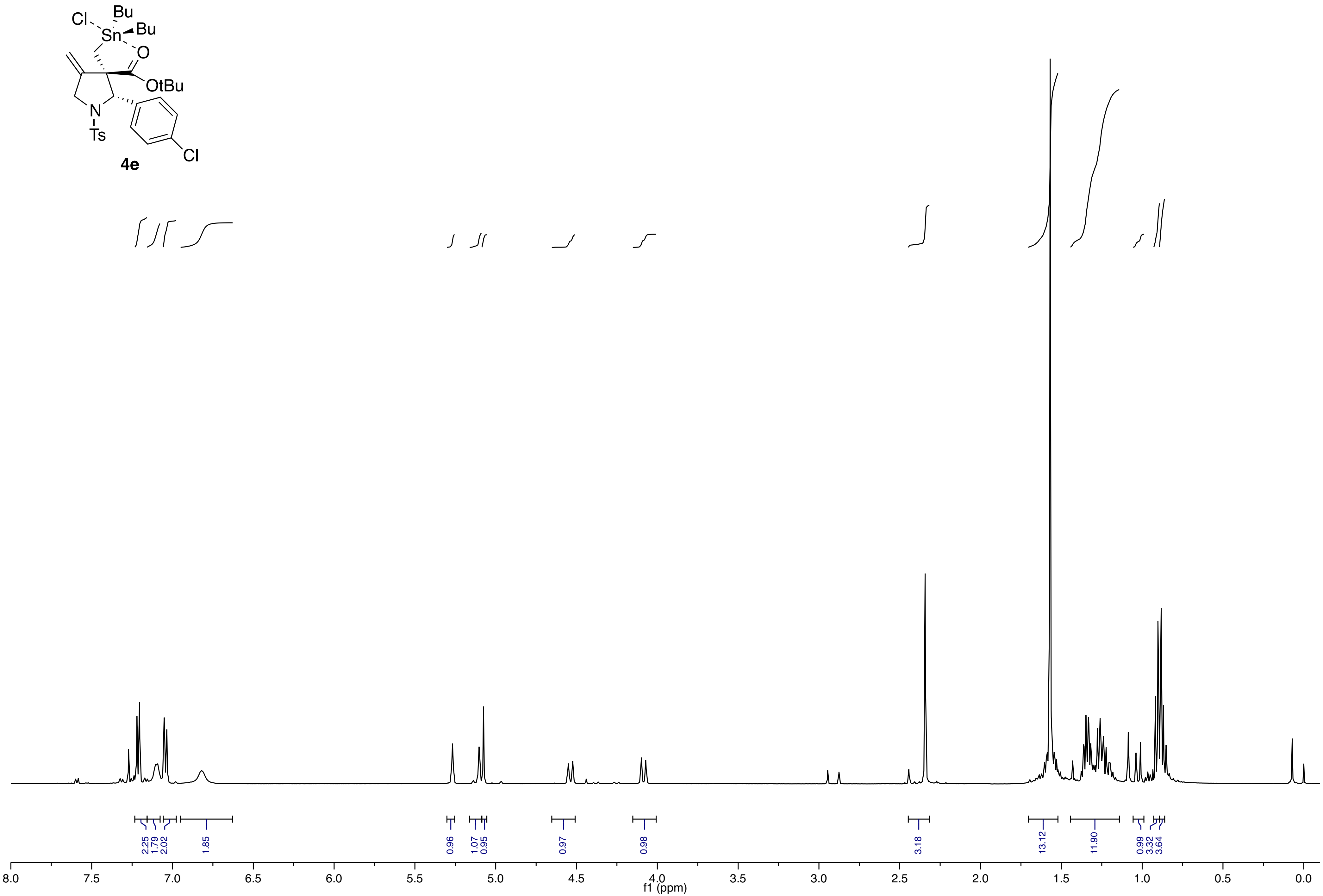
4c

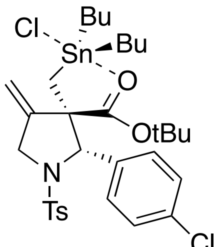

2.04
2.14
1.95
1.81
1.03
1.03
1.05
1.22
1.22
2.84
2.98
20.64
8.78
0.98
7.30
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Cl
Bu
Bu
Sn
O
OtBu
N
Ts
Me

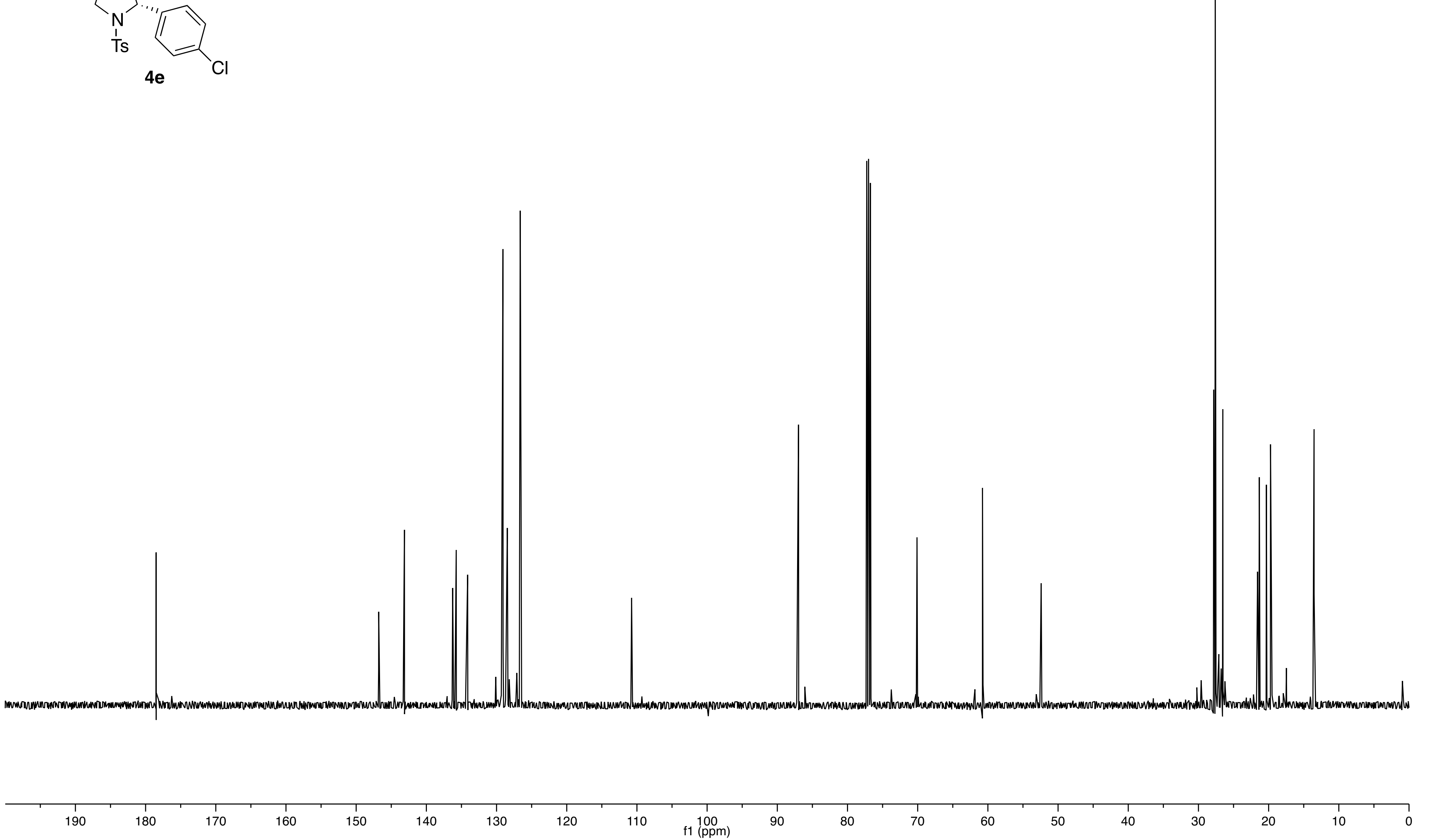
4c

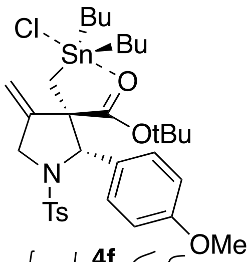

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Cl
Bu
Sn
Bu
O
OtBu
N
Ts
Cl
4e
2.25
1.79
2.02
1.85
0.96
1.07
0.95
0.97
0.98
3.18
13.12
11.90
0.99
3.32
3.64
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
0.0
f1 (ppm)

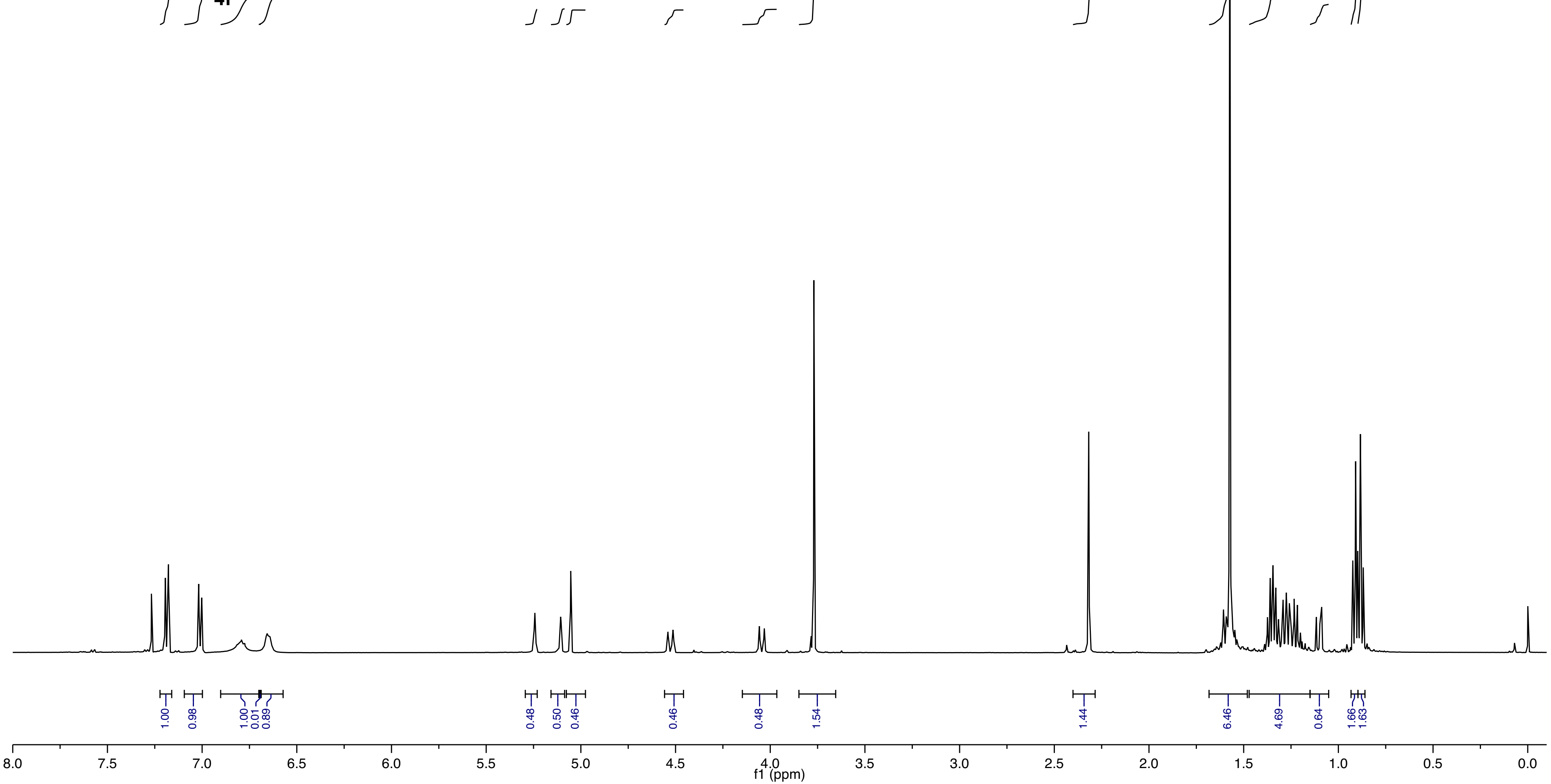

Cl
Bu
Sn
Bu
O
OtBu
N
Ts
Cl

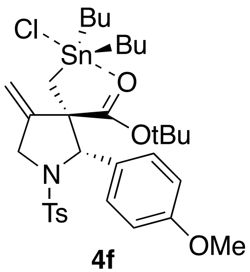
4e


190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

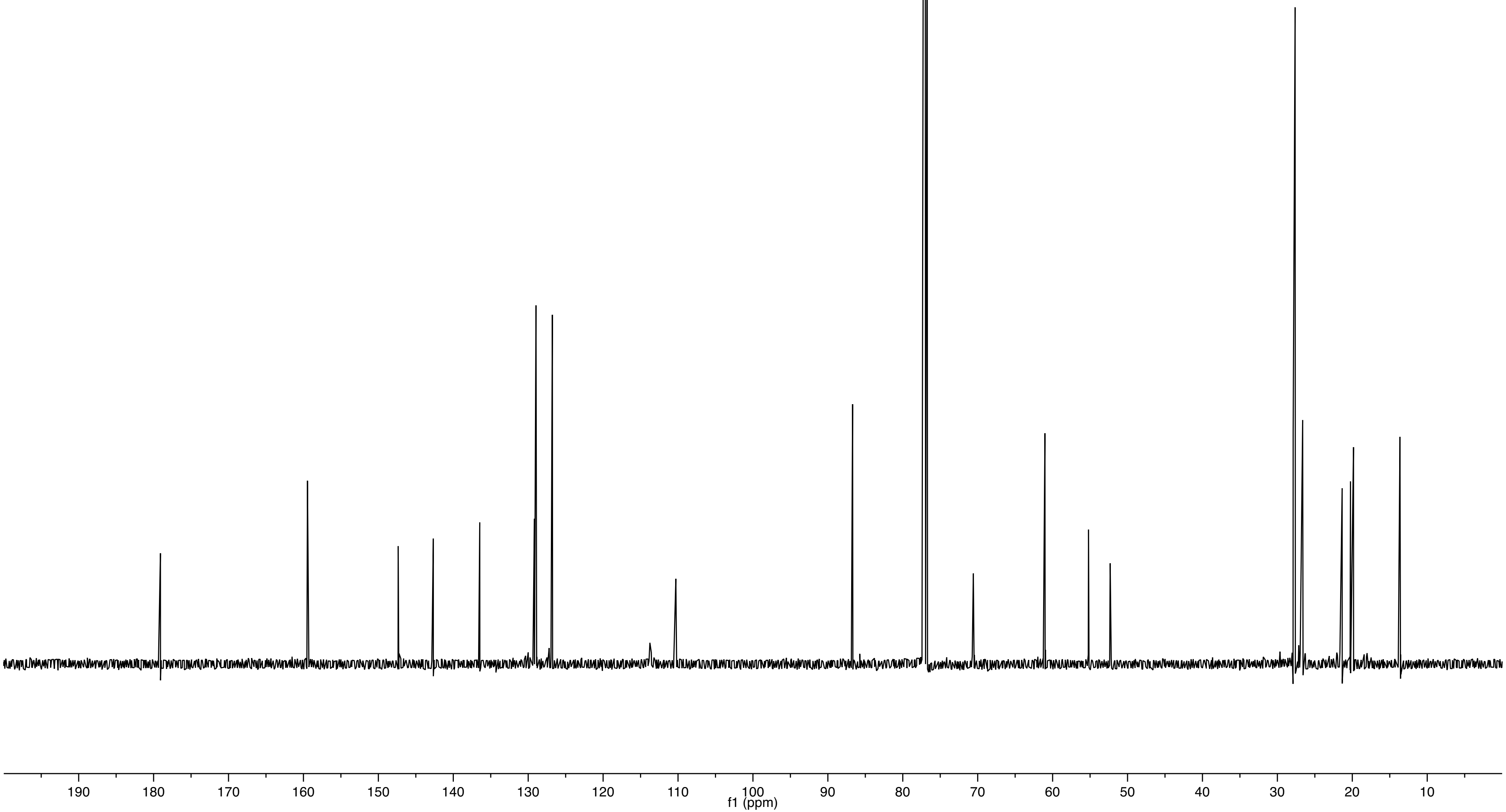


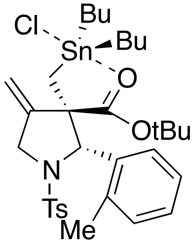
4f


1.00
0.98
1.00
0.01
0.89
0.48
0.50
0.46
0.46
0.48
1.54
1.44
6.46
4.69
0.64
1.66
1.63
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
0.0
f1 (ppm)

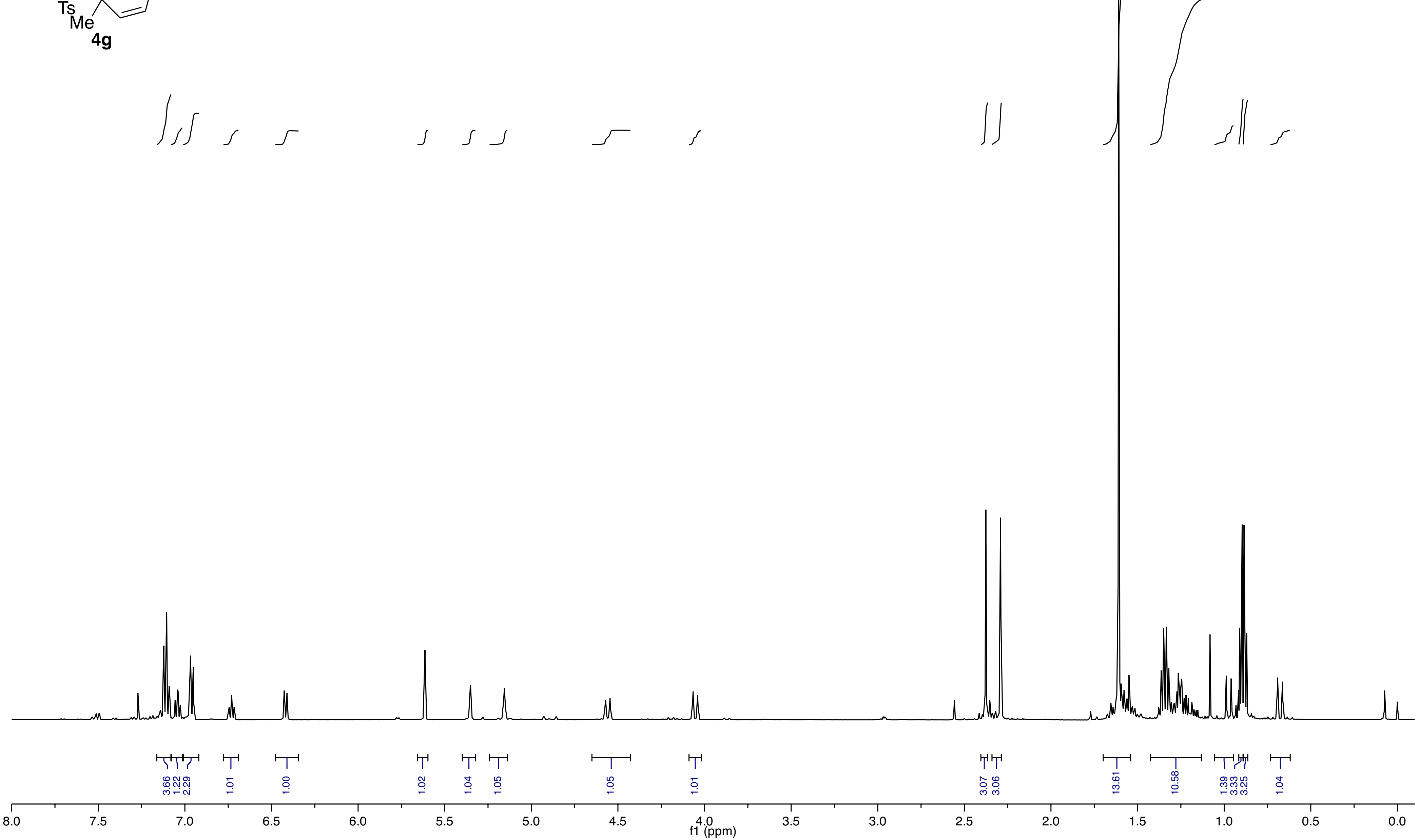

Cl
Bu
Sn
Bu
O
OtBu
N
Ts
OMe

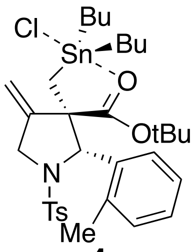
4f


190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10

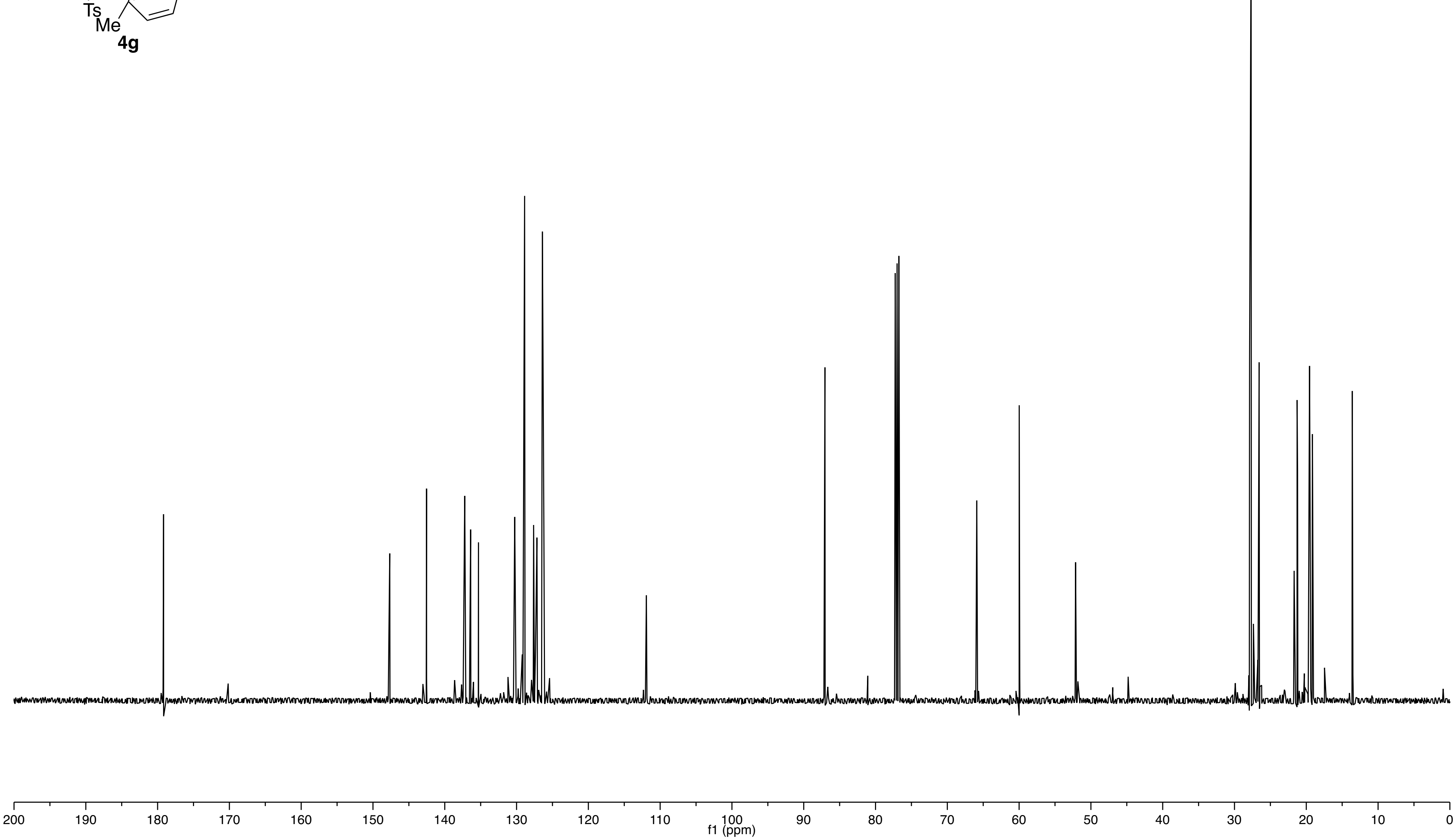

Cl
Bu
Sn
Bu
O
OtBu
N
Ts
Me

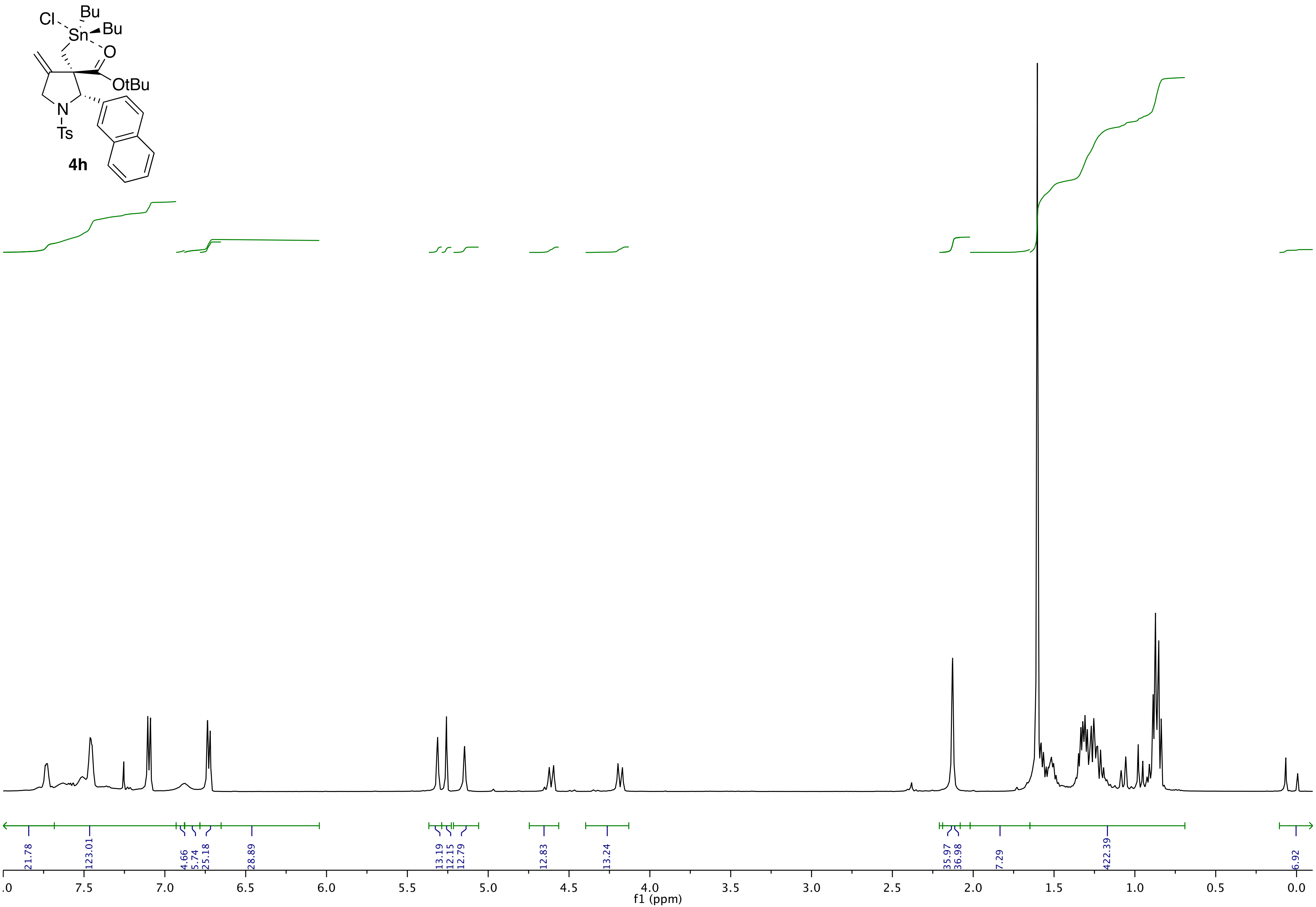
4g

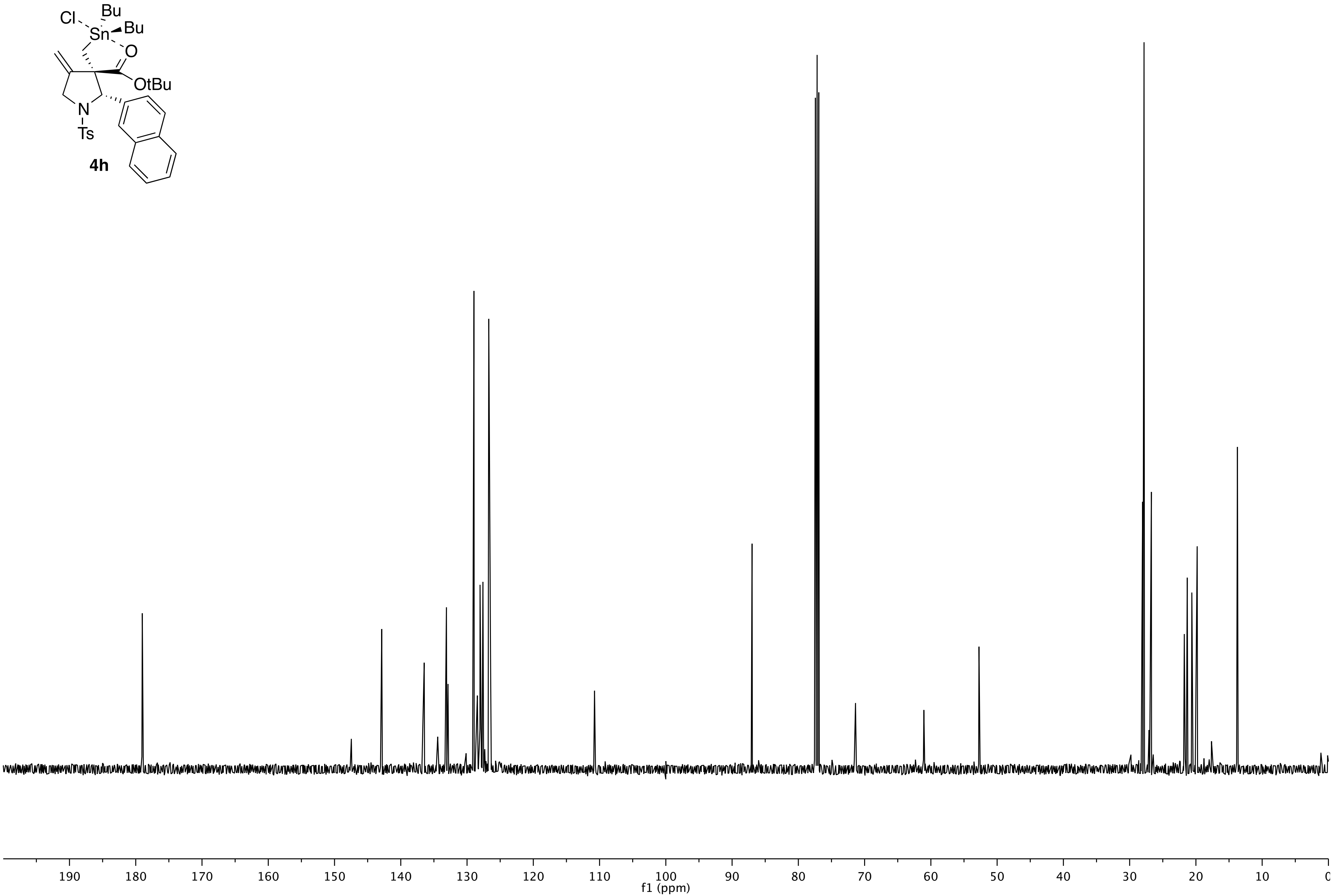

3.66
1.22
2.29
1.01
1.00
1.02
1.04
1.05
1.05
1.01
3.07
3.06
13.61
10.58
1.39
3.33
3.25
1.04
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

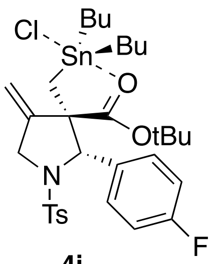

Cl
Bu
Sn
Bu
O
OtBu
N
Ts
Me

4g

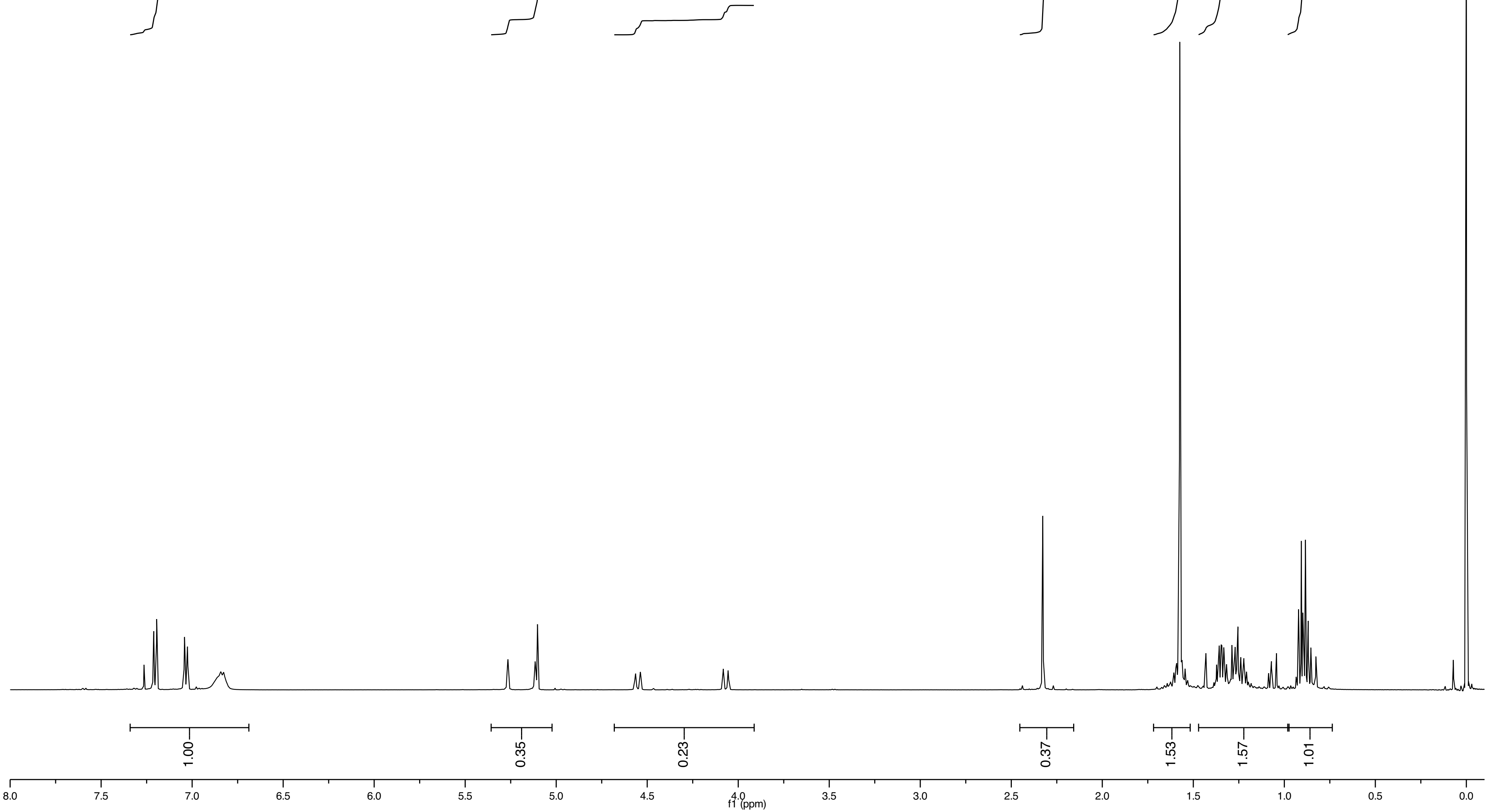

200
190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10
0


Cl
Bu
Sn
Bu
O
OtBu
N
Ts
4h
21.78
123.01
4.66
5.74
25.18
28.89
13.19
12.15
12.79
12.83
13.24
35.97
36.98
7.29
422.39
6.92
.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
0.0
f1 (ppm)


Cl
Bu
Sn
Bu
O
OtBu
N
Ts
4h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Cl
Bu
Sn
Bu
O
OtBu
N
Ts
F

4i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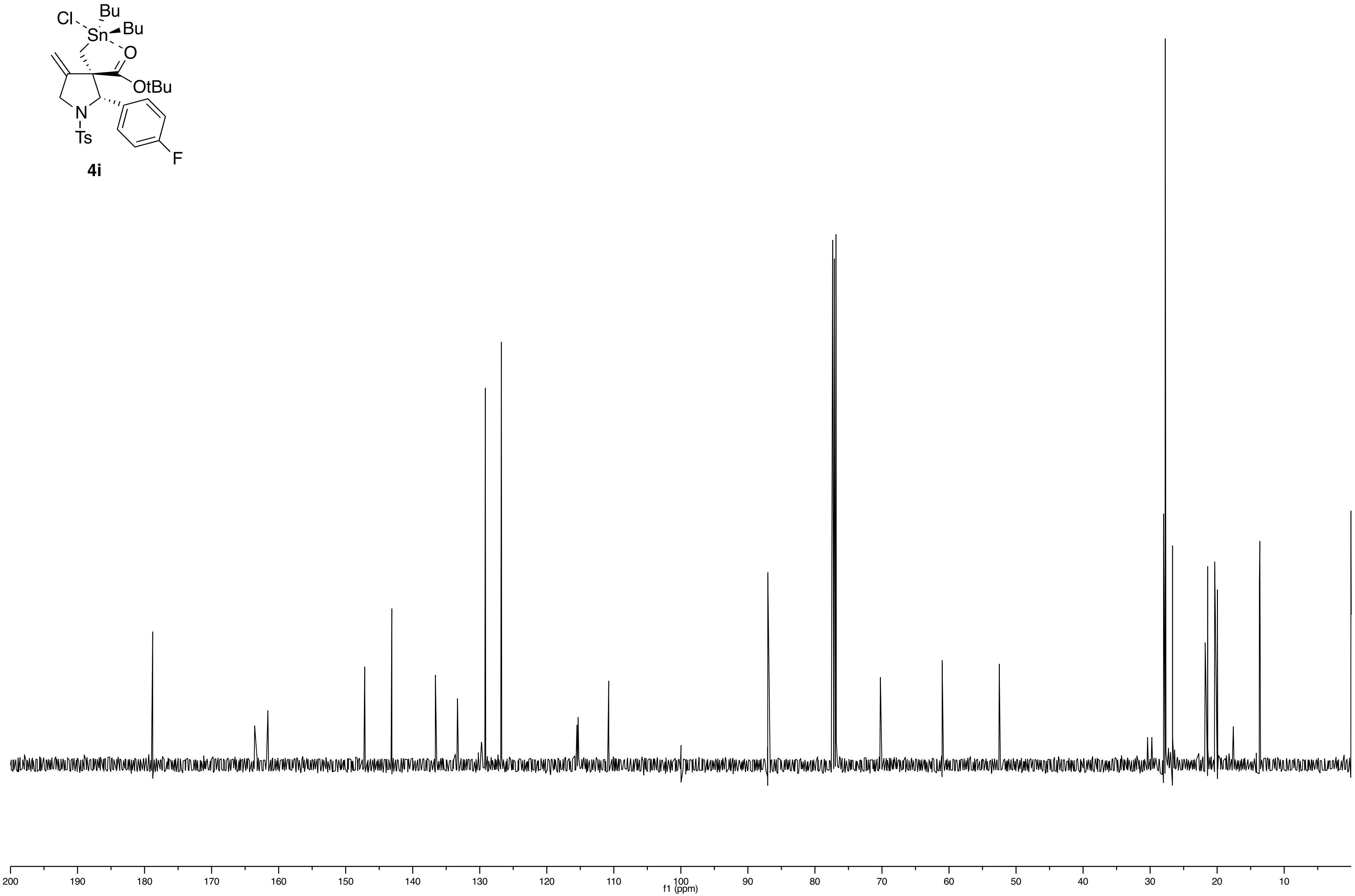
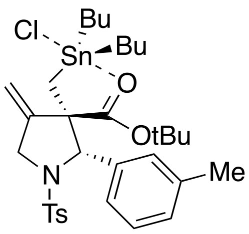
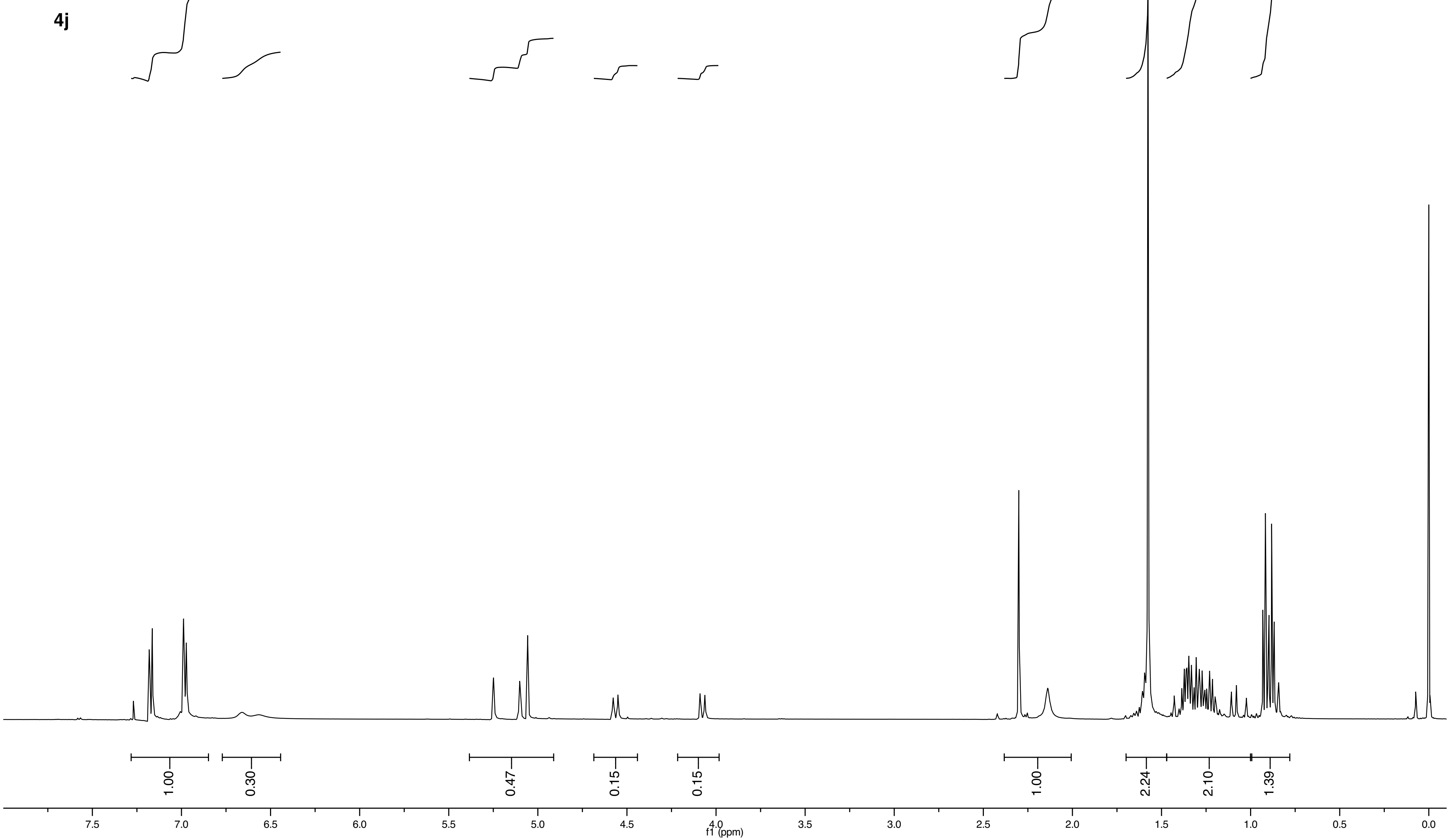
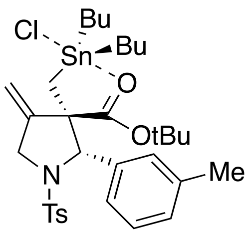
0.35
0.23
0.37
1.53
1.57
1.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
f1 (ppm)
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


Cl
Bu
Sn
Bu
O
OtBu
N
Ts
F
4i
200
190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10

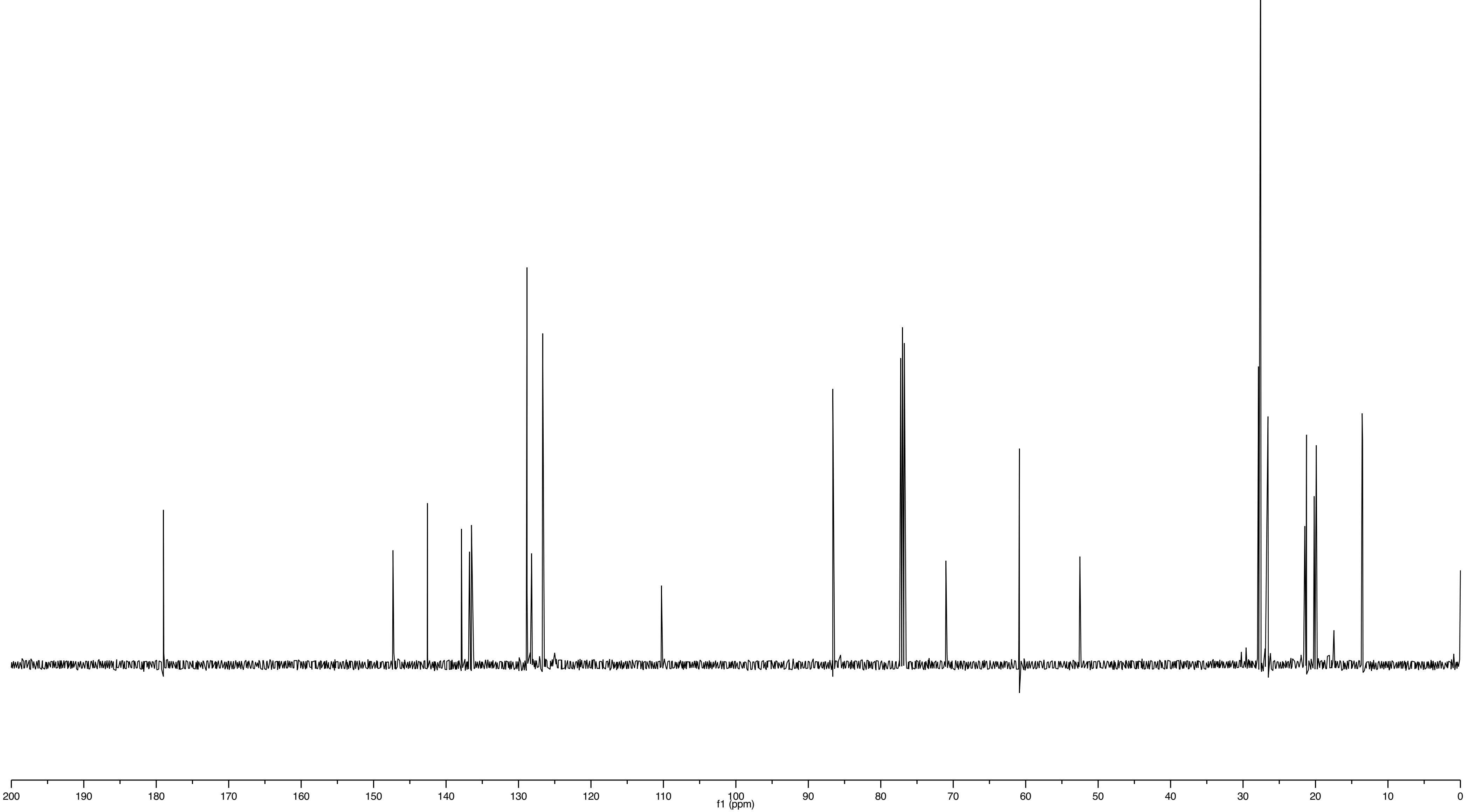

Cl
Bu
Sn
Bu
O
OtBu
N
Ts
Me

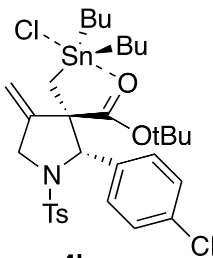
4j


1.00
0.30
0.47
0.15
0.15
1.00
2.24
2.10
1.39
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
f1 (ppm)
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

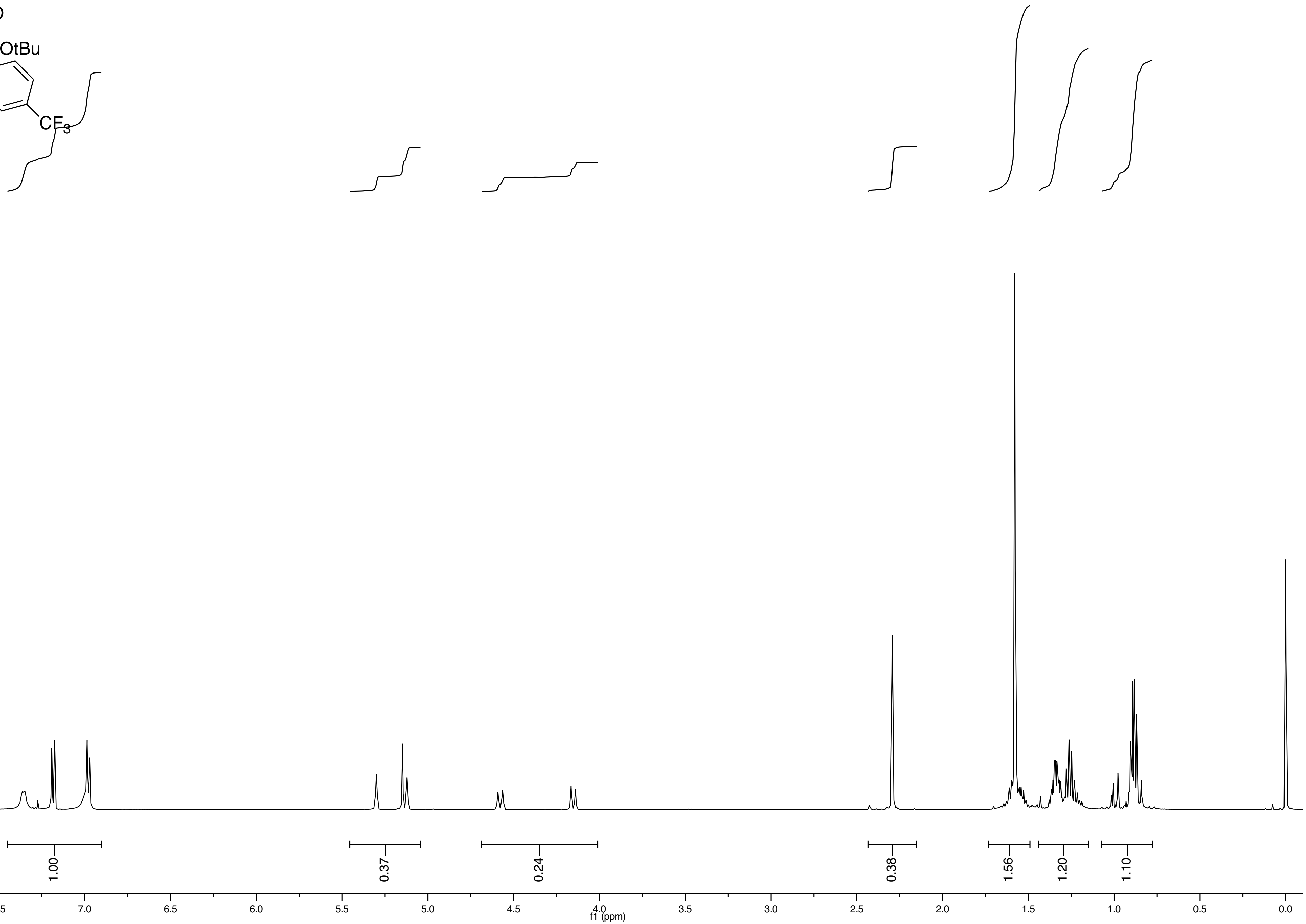

Cl
Bu
Sn
Bu
O
OtBu
N
Ts
Me

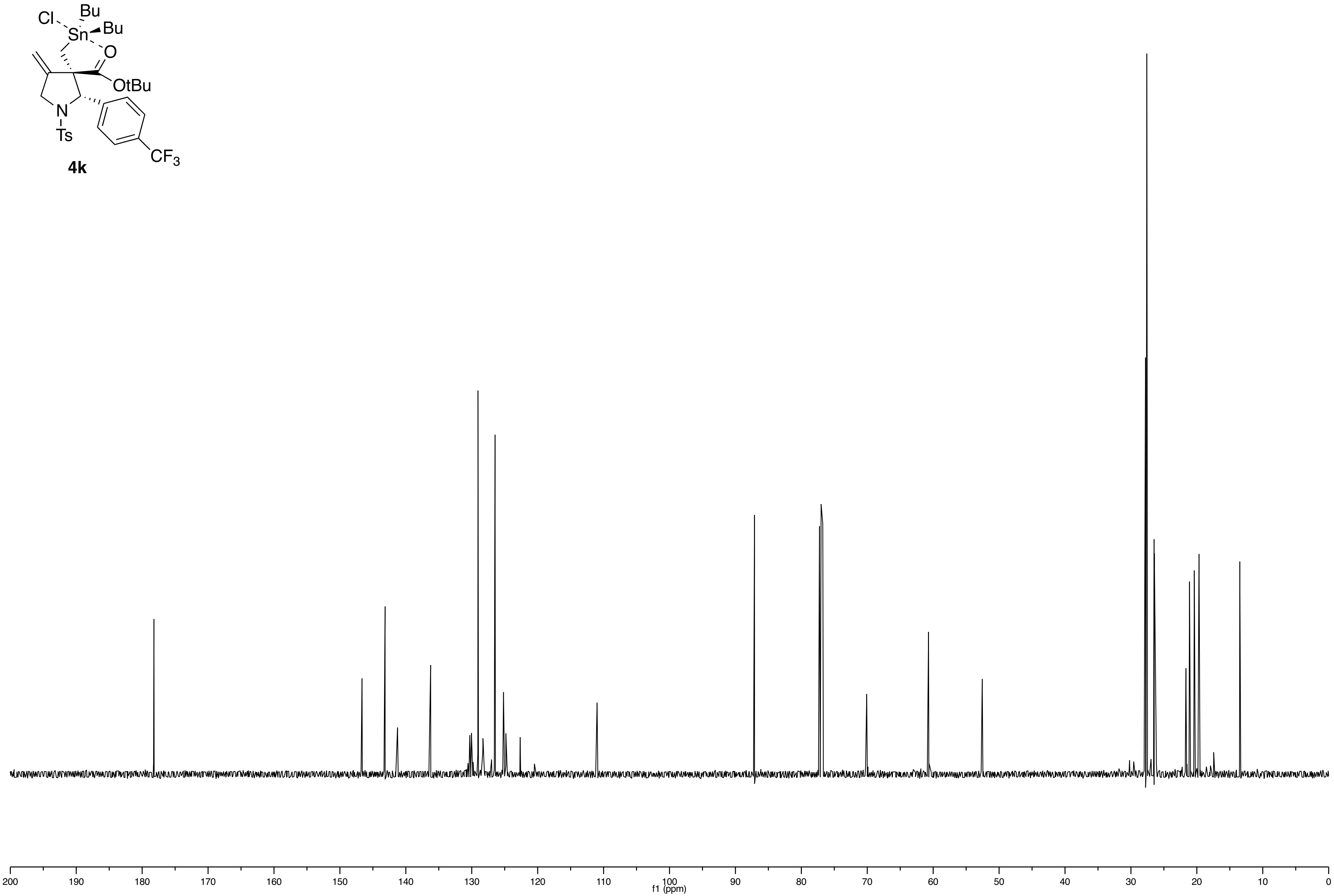
4j

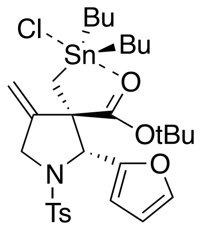

200
190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10
0


Cl
Bu
Sn
Bu
O
OtBu
N
Ts
CF3

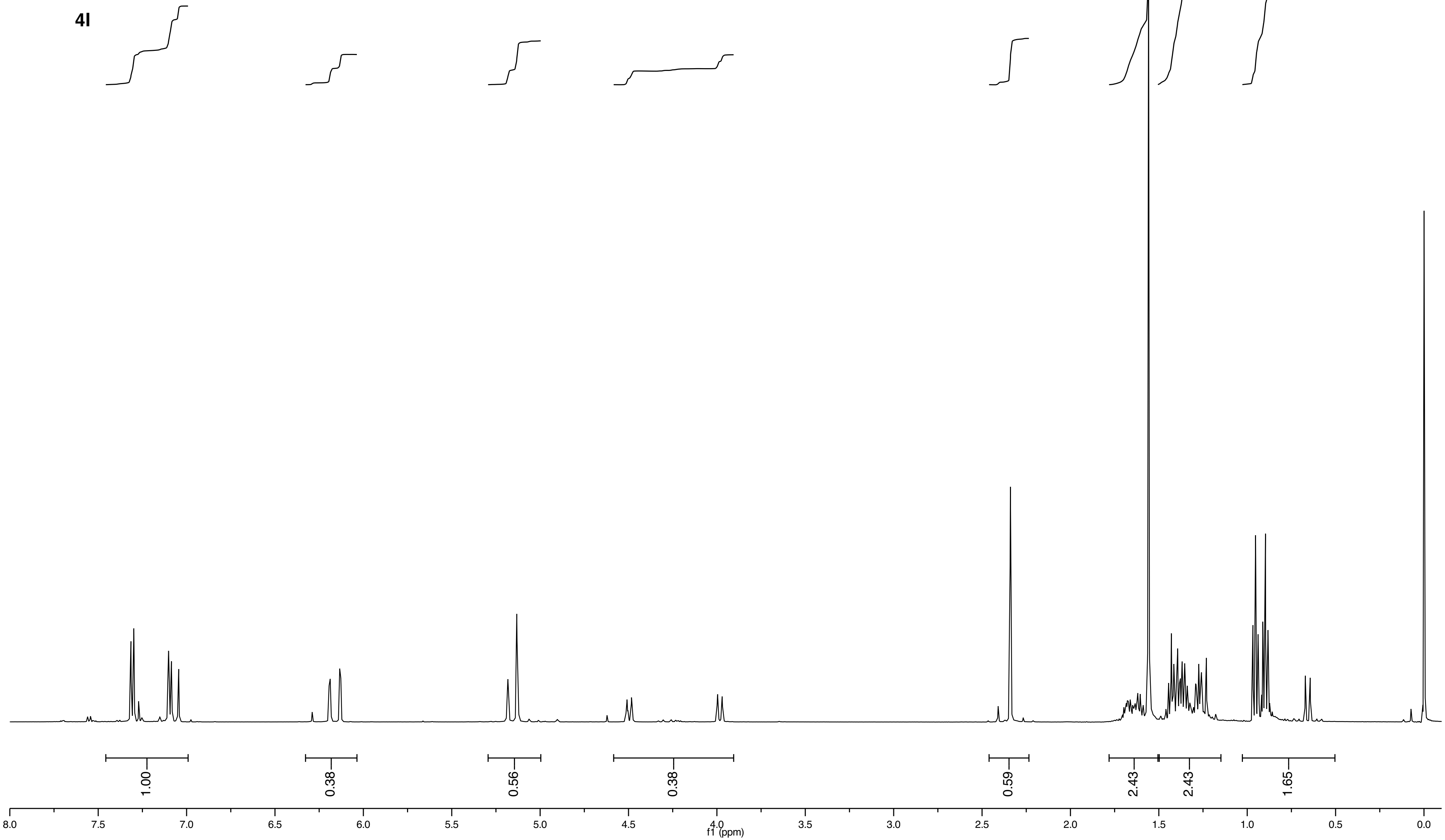
4k

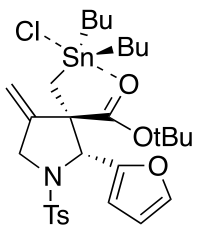

1.00
0.37
0.24
0.38
1.56
1.20
1.10
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Cl
Bu
Sn
Bu
O
OtBu
N
Ts
CF3
4k
200
190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10
0

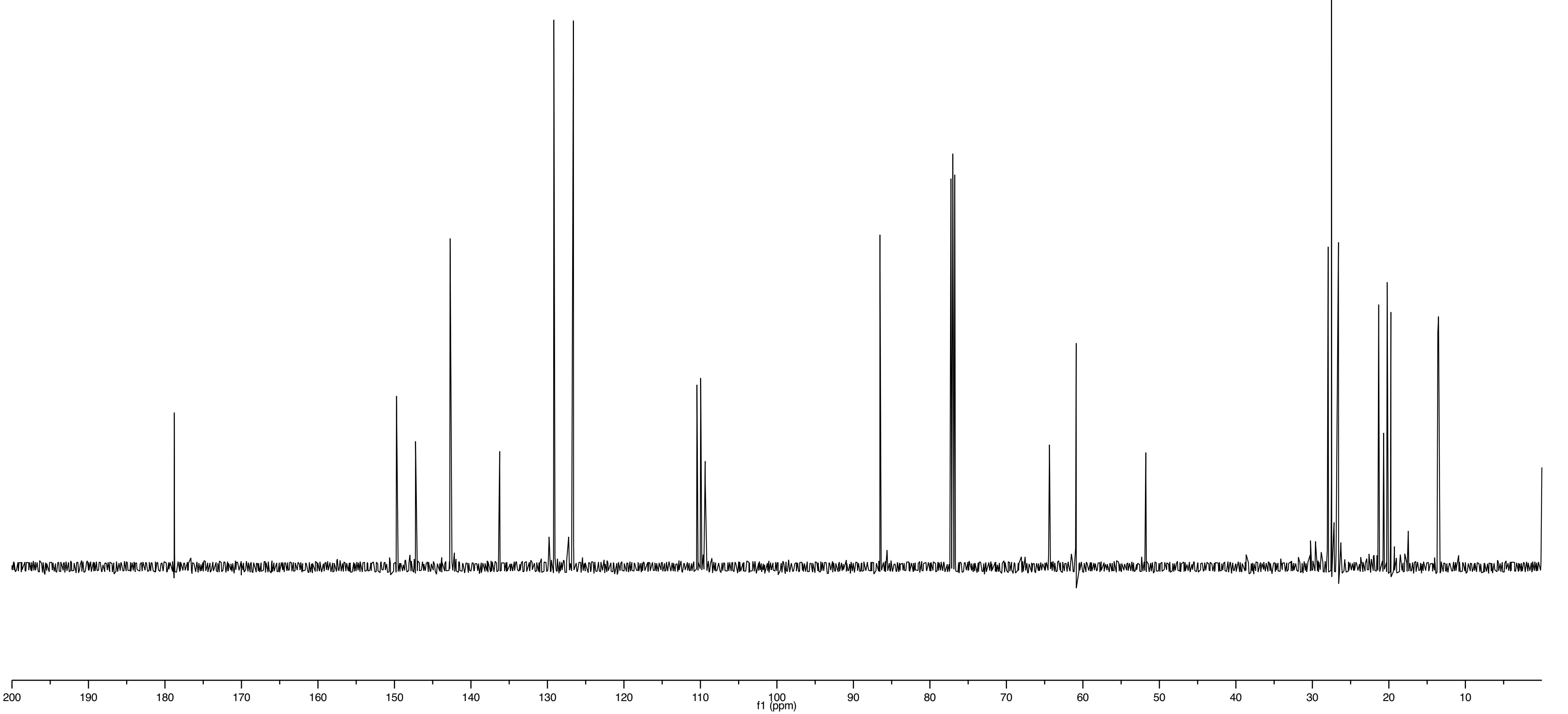

Cl
Bu
Sn
Bu
O
OtBu
N
O
Ts

4l


1.00
0.38
0.56
0.38
0.59
2.43
2.43
1.65
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
0.0
f1 (ppm)


Cl
Bu
Sn
Bu
O
OtBu
O
N
Ts

4l


200
190
180
170
160
150
140
130
120
110
100
f1 (ppm)
90
80
70
60
50
40
30
20
10