Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Triphenylphosphine Promoted Regio and Stereoselective α-Halogenation of Ynamides

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SUPPORTING INFORMATION

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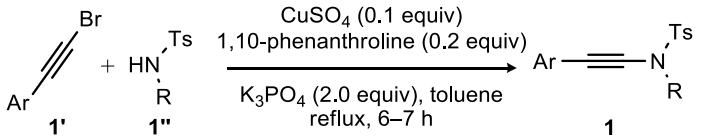
General Information: All the reactions were performed in an oven-dried Schlenk flask. Commercial grade solvents were distilled prior to use. Column chromatography was performed using 100−200 Mesh silica gel. Thin layer chromatography (TLC) was performed on silica gel GF254 plates. Visualization of spots on TLC plate was accomplished with UV light (254 nm) and staining over I_2 chamber.

Proton, carbon, and fluorine nuclear magnetic resonance spectra (^{1}H NMR, ^{13}C NMR, ^{19}F NMR, and ^{31}P NMR) were recorded based on the resonating frequencies as follows: (^{1}H NMR, 400 MHz; ^{13}C NMR, 101 MHz; ^{19}F NMR, 376 MHz) and (^{1}H NMR, 500 MHz; ^{13}C NMR, 126 MHz; ^{19}F NMR, 470 MHz; ^{31}P NMR, 202 MHz) having the solvent resonance as internal standard (^{1}H NMR, $CHCl_3$ at 7.26 ppm; ^{13}C NMR, $CDCl_3$ at 77.0 ppm) and the data for ^{1}H, ^{13}C NMR, ^{19}F, ^{31}P NMR were reported in terms of chemical shift (ppm). Few cases tetramethylsilane (TMS) at 0.00 ppm was used as reference standard. Data for ^{1}H NMR are reported as follows: chemical shift (ppm), multiplicity (s = singlet; bs = broad singlet; d = doublet; bd = broad doublet, t = triplet; bt = broad triplet; q = quartet; m =multiplet), coupling constants, *J*, in (Hz), and integration. IR spectra were reported in cm^{-1}. HRMS were obtained in ESI-TOF analyzer. LC−MS spectra were obtained (EI positive/nagative mode) with an ionization voltage of 70 eV; data are reported in the form of m/z (intensity relative to base peak 100). Elemental (C, H, N) analysis was carried out using an EA 1112 analyzer. Melting points were determined by electrothermal heating and are uncorrected.

Materials: Unless otherwise noted, all the reagents were obtained commercially and used without purification. Dichloromethane (CH_2Cl_2) and carbon tetrachloride (CCl_4) used from bottle grade. Iodoform (CHI_3), tetrabromomethane (CBr_4), carbon tetrachloride (CCl_4), and triphenylphosphine (Ph_3P) are purchased and used as received.

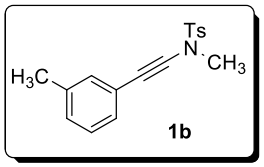
Experimental Section

Following the known synthetic procedure, **1a–r** are prepared. Analytical and spectral data of **1a**, **1d**, **1h**, **1i**, **1o**, **1p** and 1**r** are exactly matching with the reported values.[1,2]



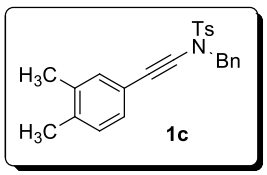
General Procedure for the Synthesis of 1 (GP 1):[1] To a mixture of **1″** (2.0 mmol), $CuSO_4 \cdot 5H_2O$ (0.2 mmol), 1,10-phenanthroline (0.4 mmol) and K_3PO_4 (4.0 mmol) in dry toluene (8.0 mL) was added 1-bromo-2-arylacetylene (**1′**). The reaction mixture was heated at 70 ºC under the nitrogen atmosphere. Progress of the reaction was monitored periodically by TLC. Upon completion, the reaction mixture was cooled to room temperature and diluted with dichloromethane (10 mL). The crude mixture was filtered through a small pad of Celite and concentrated under the reduced pressure. The crude residue was purified using column chromatography on silica gel to provide **1**.

N-Methyl-N-(*m*-tolylethynyl)-N-4-methylbenzenesulfonamide (1b):



Pale yellow solid (460 mg, 77% yield); mp 85–88 °C; R_f = 0.38 (9:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.21–7.14 (m, 3H), 7.12–7.06 (m, 1H), 3.14 (s, 3H), 2.45 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.7, 137.9, 133.2, 131.9, 129.8, 128.7, 128.4, 128.1, 127.8, 122.4, 83.6, 69.1, 39.3, 21.6, 21.2; IR (Neat)ν_{max} 2230, 1687, 1594, 1446, 1167, 1019, 865, 821, 783 cm^{-1}; MS (EI) *m/z* (%) 300 (M^+ + 1, 100), 207 (13); Anal. Calcd. for $C_{17}H_{17}NO_2S$: C, 68.20; H, 5.72; N, 4.68; S, 10.71 . Found: C, 68.35; H, 5.75; N, 4.61; S, 10.62.

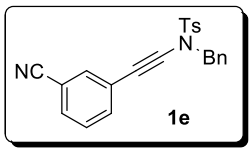
N-Benzyl-N-((3,4-dimethylphenyl)ethynyl)-4-methylbenzenesulfonamide (1c):



Colorless solid (710 mg, 91% yield); mp 105−107 °C; R_f = 0.4 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (bd, *J* = 7.6 Hz, 2H), 7.38–7.24 (m, 7H), 7.03 (s, 1H), 7.00 (s, 2H), 4.56 (s, 2H), 2.43 (s, 3H), 2.21 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.5, 136.6, 136.4, 134.7, 134.5, 132.4, 129.6, 129.4, 128.8, 128.76, 128.4, 128.2,

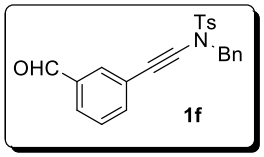
127.7, 119.9, 81.7, 71.3, 55.7, 21.6, 19.6, 19.5; IR (Neat)ν_{max} 2219, 1594, 1452, 1358, 953, 915, 756, 591, 531; cm^{-1}; MS (EI) *m/z* (%) 390 (M^+ + 1, 100); Anal. Calcd. for $C_{24}H_{23}NO_2S$: C, 78.81; H, 6.19; N, 3.59; O, 8.19; S, 8.21. Found: C, 74.15; H, 5.98; N, 3.65; S, 8.31.

N-Benzyl-N-((3-cyanophenyl)ethynyl)-4-methylbenzenesulfonamide (1e):



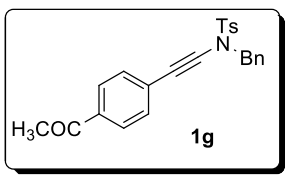
Colorless solid (672 mg, 87% yield); mp 147−148 °C; R_f = 0.41 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.0 Hz, 2H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.54–7.47 (m, 3H), 7.44–7.34 (m, 6H), 4.70 (s, 2H), 2.43 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 145.9, 134.8, 134.2, 133.7, 133.5, 131.8, 130.7, 129.4, 129.1, 128.9, 128.8, 128.0, 126.2, 117.9, 112.7, 89.3, 68.7, 55.8, 21; IR (Neat)ν_{max} 2235, 1693, 1490, 1364, 1304, 1084, 958, 810, 706 cm^{-1}; MS (EI) *m/z* (%) 441 (M^+ + 1, 100); Anal. Calcd. for $C_{23}H_{18}N_2O_2S$: C, 57.15; H, 3.88; N, 3.35; O, 7.25; S, 7.27. Found: C, 57.23; H, 3.82; N, 6.29; S, 7.32.

N-Benzyl-N-((3-formylphenyl)ethynyl)-4-methylbenzenesulfonamide (1f):



Pale yellow gummy liquid (638 mg, 82 % yield); R_f = 0.62 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.98 (s, 1H), 7.90 (d, *J* = 8.0 Hz, 2H), 7.86–7.80 (m, 1H), 7.73 (bs, 1H), 7.60–7.49 (m, 4H), 7.42–7.34 (m, 5H), 4.67 (s, 2H), 2.42 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 193.1, 145.7, 136.9, 136.4, 135.0, 134.3, 131.8, 130.7, 130.1, 129.3, 129.0, 128.8, 128.0, 123.5, 84.7, 70.4, 55.5, 21.6; IR (Neat)ν_{max} 2235, 1594, 1495, 1260, 1172, 1090, 936, 816, 723, 597 cm^{-1}; MS (EI) *m/z* (%) 389 (M^+ + 2, 100); Anal. Calcd. for $C_{23}H_{19}NO_3S$: C, 70.93; H, 4.92; N, 3.60; O, 12.32; S, 8.23. Found: C, 71.56; H, 4.61; N, 7.32; S, 8.38.

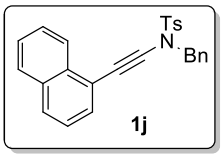
N-((4-Acetylphenyl)ethynyl)-N-benzyl-4-methylbenzenesulfonamide (1g):



Colorless solid (629 mg, 78% yield); mp 146−148 °C; R_f = 0.51 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.0 Hz, 4H), 7.52 (d, *J* = 12.8 Hz, 2H), 7.41–7.37 (m, 5H), 7.37–7.32 (m, 2H), 4.68 (s, 2H), 2.55 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 197.5, 145.8, 135.8, 135.0, 134.2, 130.7, 130.6, 129.3, 129.0, 128.9, 128.9, 128.0, 127.3, 86.8, 71.5, 55.5, 27.1, 21.6; IR (Neat)ν_{max} 1682, 1594, 1550, 1452, 1265, 1084, 761

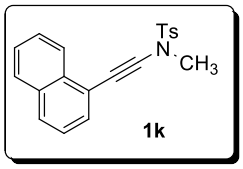
cm^{-1}; MS (EI) *m/z* (%) 389 (M^+ – 1, 100); Anal. Calcd. for $C_{24}H_{21}NO_3S$: C, 71.44; H, 5.25; N, 3.47; O, 11.90; S, 7.95. Found: C, 71.35; H, 5.28; N, 3.52; S, 7.89.

N-Benzyl-4-methyl-N-(naphthalen-1-ylethynyl)benzenesulfonamide (1j):



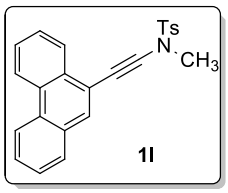
Colorless solid (667 mg, 81% yield); mp 112−114 °C; R_f = 0.32 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, *J* = 8.4 Hz, 2H), 7.83–7.69 (m, 3H), 7.47–7.42 (m, 2H), 7.41–7.36 (m, 3H), 7.36–7.28 (m, 6H), 4.67 (s, 2H), 2.42 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.7, 134.6, 134.4, 133.0, 132.9, 129.8, 129.2, 129.0, 128.6, 128.5, 128.4, 128.0, 127.9, 127.8, 126.5, 126.2, 126.1, 125.1, 120.5, 87.1, 69.9, 55.7, 21.6; IR (Neat)ν_{max} 2224, 1649, 1594, 1539, 1495, 1446, 1260, 931, 843, 591 cm^{-1}; MS (EI) *m/z* (%) 412 (M^+ + 1, 100); Anal. Calcd. for $C_{26}H_{21}NO_2S$: C, 75.88; H, 5.14; N, 3.40; O, 7.78; S, 7.79. Found: C, 75.73; H, 5.18; N, 3.46; S, 7.71.

N-Methyl-N-(naphthalen-1-ylethynyl)-N-4-Methylbenzenesulfonamide (1k):



Colorless solid (509 mg, 76% yield); mp 119−121 °C; R_f = 0.28 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26 (d, *J* = 8.4 Hz, 1H), 7.83 (d, *J* = 7.6 Hz, 2H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.57–7.48 (m, 2H), 7.42 (bt, *J* = 7.2 Hz, 1H), 7.30 (bt, *J* = 7.2 Hz, 1H), 7.19 (d, *J* = 7.6 Hz, 2H), 3.18 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.7, 132.8, 132.80, 132.78, 130.5, 129.6, 129.2, 128.0, 127.9, 127.7, 127.4, 127.2, 126.4, 126.1, 125.7, 124.9, 120.0, 88.5, 67.2, 39.1, 21.2; IR (Neat)ν_{max} 2230, 1594, 1550, 1364, 1260, 1167, 947, 800, 171 cm^{-1}; MS (EI) *m/z* (%) 336 (M^+ + 1, 100); Anal. Calcd. for $C_{20}H_{17}NO_2S$: C, 71.62; H, 5.11; N, 4.18; O, 9.54; S, 9.56. Found: C, 71.56; H, 5.18; N, 4.23; S, 9.49.

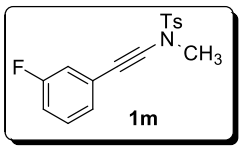
N-Methyl-N-(phenanthren-9-ylethynyl)-N-4-Methylbenzenesulfonamide (1l):



Pale yellow solid; (608 mg, 79% yield); mp 120−121 °C; R_f = 0.29 (9:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.68–8.59 (m, 2H), 8.35–8.30 (m, 1H), 7.93–7.85 (m, 3H), 7.82–7.52 (m, 1H), 7.71–7.53 (m, 4H), 7.34 (d, *J* = 8.0 Hz, 2H), 3.28 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.9, 133.3, 131.2, 131.1, 130.7, 130.0, 130.0, 129.9, 128.3, 127.8, 127.2, 127.17, 127.03, 126.9, 126.8, 122.7, 122.6, 119.1, 88.2, 67.7, 39.4, 21.6; IR (Neat)ν_{max} 2926, 1600, 1523, 1446, 1167, 1084, 931, 860, 745 cm^{-1}; MS (EI) *m/z* (%) 386 (M^+ +

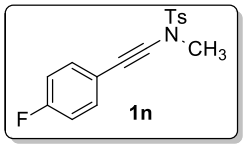
1, 100); Anal. Calcd. for $C_{24}H_{19}NO_2S$: C, 74.78; H, 4.97; N, 3.63; O, 8.30; S, 8.32 Found: C, 74.65; H, 4.91; N, 3.72; S, 8.38.

N-((3-Fluorophenyl)ethynyl)-N,4-dimethylbenzenesulfonamide (1m):



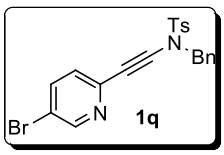
Pale yellow semi solid; (533 mg, 88% yield); R_f = 0.36 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, J = 8.4 Hz, 2H), 7.54 (d, J = 8.0 Hz, 2H), 7.45–7.38 (m, 1H), 7.24–7.18 (m, 3H), 3.15 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.3 (d, J = 245 Hz, 1C), 145.8, 132.7, 131.2 (d, J = 9.0 Hz, 1C), 130.7, 128.1, 127.62, 127.60, 124.4 (d, J = 10.0 Hz, 1C), 117.8 (d, , J = 23.0 Hz, 1C), 117.8 (d, J = 21 Hz, 1C), 58.7, 68.1, 21.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ −112.6; IR (Neat)ν_{max} 12241, 1698, 1490, 1347, 1161, 1024, 767, 673 cm^{-1}; MS (EI) *m/z* (%) 304 (M^+ + 1, 100); Anal. Calcd. for $C_{16}H_{14}FNO_2S$: C, 63.35; H, 4.65; F, 6.26; N, 4.62; O, 10.55; S, 10.57. Found: C, 63.41; H, 4.58; N, 4.56; S, 10.45.

N-((4-Fluorophenyl)ethynyl)-N,4-dimethylbenzenesulfonamide (1n):



Brownish gummy solid; (394 mg, 65% yield); R_f = 0.35 (9:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, J = 8.4 Hz, 2H), 7.54 (d, J = 8.0 Hz, 2H), 7.46–7.40 (m, 2H), 7.22 (t, J = 9.2 Hz, 2H), 3.13 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4 (d, J = 247 Hz, 1C), 145.7, 134.0, 133.9, 132.7, 130.7, 128.1, 118.7, 118.7, 116.4 (d, J = 22 Hz, 1C), 84.3, 67.9, 21.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ −111.2; IR (Neat)ν_{max} 2230, 1698, 1600, 1358, 1161, 1084, 959 cm^{-1}; MS (EI) *m/z* (%) 304 (M^+ + 1, 100); Anal. Calcd. for $C_{16}H_{14}FNO_2S$: C, 63.35; H, 4.65; F, 6.26; N, 4.62; O, 10.55; S, 10.57. Found: C, 63.26; H, 4.69; N, 4.58; S, 10.45.

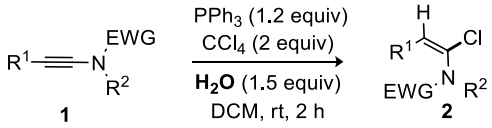
N-Benzyl-N-((5-bromopyridin-2-yl)ethynyl)-4-methylbenzenesulfonamide (1q):



Colorless solid; (573 mg, 65% yield); mp 126−127 °C; R_f = 0.2 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.54 (s, 1H), 7.78 (d, J = 8.0 Hz, 2H), 7.68 (d, J = 8.0 Hz, 1H), 7.39–7.25 (m, 7H), 7.08 (d, J = 8.4 Hz, 1H), 4.61 (s, 2H), 2.43 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.8, 144.9, 141.7, 138.6, 134.6, 134.1, 129.8, 128.7, 128.6, 128.4, 127.7, 127.3, 119.0, 84.4, 71.0, 55.6, 29.7, 21.6; IR (Neat)ν_{max} 2230, 1594, 1161, 1134, 1041, 827, 778, 657 cm^{-1}; MS (EI) *m/z* (%) 304 (M^+

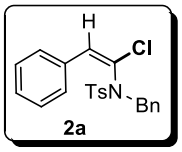
+ 1, 100); Anal. Calcd. for $C_{21}H_{17}BrN_2O_2S$: C, 71.48; H, 4.69; N, 7.25; O, 8.28; S, 8.30. Found: C, 70.85; H, 4.88; N, 3.56; S, 8.31.

General Procedure for the Chlorination of 1; Synthesis of 2 (GP 2):



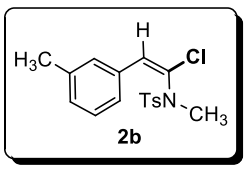
To a solution of corresponding ynamide **1** (0.5 mmol), PPh_3 (0.6 mmol), and CCl_4 (1.0 mmol) in CH_2Cl_2 (2.0 mL for 0.5 mmol) in a Schlenk tube was added H_2O (0.75 mmol). The resulting mixture was stirred at room temperature (25 °C). Progress of the reaction was monitored periodically by TLC. Upon completion, the reaction mixture was diluted with CH_2Cl_2 (10 mL). The crude mixture was filtered through a small pad of Celite and concentrated under the reduced pressure. The crude residue was purified through column chromatography on silica gel to provide **2**.

(*E*)-N-Benzyl-N-(1-chloro-2-phenylvinyl)-4-methylbenzenesulfonamide (2a):



Colorless solid; (*E*/*Z* = 99:1; 195 mg, 98% yield); mp = 168–169 ºC; R_f = 0.65 (4:1 hexane/EtOAc), [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, *J* = 8.4 Hz, 2H), 7.38–7.30 (m, 4H), 7.24–7.19 (m, 5H), 7.18–7.08 (m, 3H), 6.61 (s, 1H), 4.80 (bd, *J* = 9.6 Hz, 1H), 4.05 (bd, *J* = 10 Hz, 1H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.6, 135.0, 134.9, 133.4, 132.9, 129.8, 129.6, 128.9, 128.8, 128.5, 128.3, 128.2, 128.1, 127.5, 52.4, 21.7; IR (Neat)ν_{max} 1595, 1348, 1238, 1167, 1085, 937, 816, 740 cm^{-1}; HRMS (ESI) for $C_{22}H_{20}ClNNaO_2S$ $(M+Na)^+$: calcd 420.0801, found 420.0805.

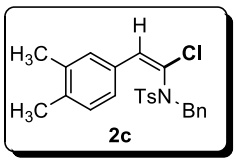
(*E*)-N-(1-Chloro-2-*m*-tolylvinyl)-N,4-dimethylbenzenesulfonamide (2b):



Colorless solid; (*E*/*Z* = 86:14; 164 mg, 97% yield); mp = 170–172 °C; R_f = 0.65 (4:1 hexane/EtOAc), [Silica, UV and I_2]. Major rotamer: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, *J* = 8.0 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.35–7.25 (m, 4H), 7.12 (d, *J* = 8.0 Hz, 1H), 6.62 (s, 1H), 3.02 (s, 3H), 2.43 (s, 3H), 2.34 (s, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.4, 138.1, 134.4, 133.0, 132.5, 129.63, 129.57, 129.5, 128.8, 128.6, 128.3, 125.7, 35.7, 21.6, 21.4 ppm; Minor rotamer (selected signals): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 (d, *J* = 8.0 Hz,), 6.9 (s), 3.07 (s), 2.44 (s), 2.36 (s) ppm; IR (Neat)ν_{max} 2356,

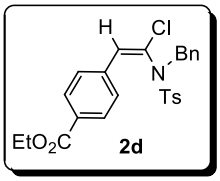
2338, 1631, 1356, 1163, 1087, 739 cm^{-1}; HRMS (ESI) for $C_{17}H_{22}ClN_2O_2S$ $(M+NH_4)^+$: calcd 353.1091, found 353.1091.

(*E*)-N-Benzyl-N-(1-chloro-2-(3,4-dimethylphenyl)vinyl)-4-methylbenzenesulfonamide (2c):



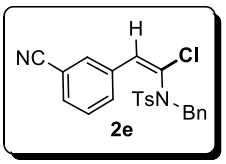
Colorless solid; (*E*/*Z* = 96:4; 183 mg, 86% yield); mp = 180–181 °C; R_f = 0.64 (4:1 hexane/EtOAc), [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.0 Hz, 2H), 7.36–7.23 (m, 4H), 7.17–7.08 (m, 4H), 7.01 (s, 1H), 6.94 (d, *J* = 7.6 Hz, 1H), 6.54 (s, 1H), 4.78 (bd, *J* = 12.4 Hz, 1H), 4.07 (bd, *J* = 12.8 Hz, 1H), 2.41 (s, 3H), 2.17 (s, 3H), 2.12 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.3, 137.2, 135.9, 135.1, 134.8, 133.6, 130.4, 129.7, 129.5, 129.3, 128.7, 128.1, 126.3, 126.2, 52.2, 21.5, 19.5; IR (Neat)ν_{max} 1596, 1453, 1359, 1161, 1087, 1020, 852, 731 cm^{-1}; HRMS (ESI) for $C_{24}H_{24}ClNO_2SNa$ $(M+Na)^+$: calcd 448.1114, found 448.1112.

(*E*)-Ethyl 4-(2-(N-benzyl-4-methylphenylsulfonamido)-2-chlorovinyl)benzoate (2d):



Colorless solid; (*E*/*Z* = 98:2; 107 mg, 73% yield); mp = 138–140 °C; R_f = 0.34 (4:1 hexane/EtOAc), [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89–7.82 (m, 4H), 7.35 (d, *J* = 8.4 Hz, 4H), 7.24–7.19 (m, 2H), 7.18–7.10 (m, 3H), 6.64 (s, 1H), 4.81 (bd, *J* = 12.8 Hz, 1H), 4.36 (q, *J* = 6.8 Hz, 2H), 4.03 (bd, *J* = 12.8 Hz, 1H), 2.46 (s, 3H), 1.39 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 166.2, 144.7, 137.2, 134.7, 134.0, 133.2, 130.0, 129.8, 129.7, 129.5, 129.2, 128.9, 128.5, 128.4, 128.3, 60.9, 52.4, 21.6, 14.3; IR (Neat) ν_{max} 1704, 1605, 1353, 1266, 1162, 773 cm^{-1}; HRMS (ESI) for $C_{25}H_{24}ClNNaO_4S$ $(M+Na)^+$: calcd 492.1012, found 492.1009.

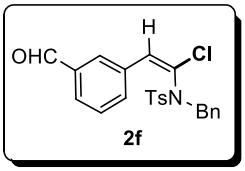
(*E*)-N-Benzyl-N-(1-chloro-2-(3-cyanophenyl)vinyl)-4-methylbenzenesulfonamide (2e):



Pale yellow solid; (*E*/*Z* = 100:0; 101 mg, 92% yield); mp = 168–170 °C; R_f = 0.44 (4:1 hexane/EtOAc), [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.44 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.30 (t, *J* = 7.6 Hz, 1H), 7.25–7.15 (m, 6H), 6.60 (s, 1H), 4.81 (bd, *J* = 12.4 Hz, 1H), 4.02 (bd, *J* = 12.8 Hz, 1H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 144.9, 134.6, 134.1, 133.1, 132.7, 132.6, 131.7, 131.5, 130.1, 129.8, 129.7, 128.9, 128.7, 128.6, 128.5, 118.3, 112.2, 52.2, 21.6; IR (Neat) ν_{max} 2219, 1595, 1359, 1233, 1162, 1014,

904, 789, 663 cm^{-1}; HRMS (ESI) for $C_{23}H_{19}ClN_2NaO_2S$ $(M+Na)^+$: calcd 445.0753, found 445.0757.

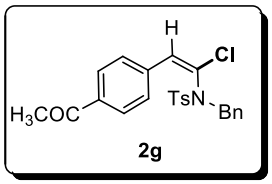
(*E*)-N-Benzyl-N-(1-chloro-2-(3-formylphenyl)vinyl)-4-methylbenzenesulfonamide (2f):



2f

Colorless solid; (*E*/*Z* = 100:0; 90 mg, 82% yield); mp = 185–186 °C; R_f = 0.37 (4:1 hexane/EtOAc), [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.84 (s, 1H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.71 (d, *J* = 7.6 Hz, 1H), 7.63 (bs, 1H), 7.60 (br d, *J* = 7.6 Hz, 1H), 7.38–7.32 (m, 3H), 7.25–7.21 (m, 2H), 7.17–7.09 (m, 3H), 6.68 (s, 1H), 4.82 (bd, *J* = 12.4 Hz, 1H), 4.04 (bd, *J* = 12.8 Hz, 1H), 2.46 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 192.0, 144.8, 136.1, 134.7, 134.3, 133.8, 133.6, 133.3, 130.7, 129.8, 129.7, 129.2, 128.8, 128.5, 128.3, 52.3, 21.6; IR (Neat) ν_{max} 2920, 1638, 1348, 1156, 1025, 904 cm^{-1}; HRMS (ESI) for $C_{23}H_{24}ClN_2O_3S$ $(M+NH_4)^+$: calcd 443.1196, found 443.1194.

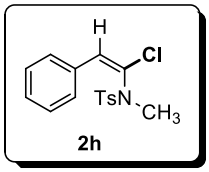
(*E*)-N-(2-(4-Acetylphenyl)-1-chlorovinyl)-N-benzyl-4-methylbenzenesulfonamide (2g):



2g

Colorless solid; (*E*/*Z* = 94:6; 80 mg, 73% yield); mp = 132–134 °C; R_f = 0.37 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.42–7.33 (m, 4H), 7.27–7.21 (m, 2H), 7.20–7.09 (m, 3H), 6.64 (s, 1H), 4.82 (bd, *J* = 12.8 Hz, 1H), 4.03 (bd, *J* = 12.8 Hz, 1H), 2.57 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 197.5, 144.8, 137.4, 136.5, 134.7, 133.9, 133.2, 129.8, 129.72, 129.67, 128.8, 128.7, 128.5, 128.3, 128.0, 52.4, 26.6, 21.6; IR (Neat) ν_{max} 1671, 1266, 1162, 1085, 707 cm^{-1}; HRMS (ESI) for $C_{24}H_{22}ClNNaO_3S$ $(M+Na)^+$: calcd 462.0907, found 462.0909.

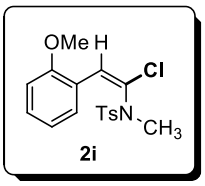
(*E*)-N-(1-Chloro-2-phenylvinyl)-N,4-dimethylbenzenesulfonamide (**2h**):



2h

Colorless solid; (*E*/*Z* = 100:0; 151 mg, 93% yield); mp = 201–202 °C; R_f = 0.55 (4:1 hexane/EtOAc), [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, *J* = 7.2 Hz, 2H), 7.63 (d, *J* = 7.6 Hz, 2H), 7.45–7.28 (m, 5H), 6.68 (s, 1H), 3.06 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.5, 134.2, 133.1, 132.4, 129.8, 129.5, 128.9, 128.82, 128.77, 128.69, 35.7, 21.7; IR (Neat) ν_{max} 1638, 1591, 1360, 1167, 1085, 960 cm^{-1}; HRMS (ESI) for $C_{16}H_{20}ClN_2O_2S$ $(M+NH_4)^+$: calcd 339.0934, found 339.0937.

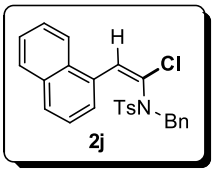
(*E*)-N-(1-Chloro-2-(2-methoxyphenyl)vinyl)-N,4-dimethylbenzenesulfonamide (2i):



2i

Colorless solid; (*E*/*Z* = 99:1; 118 mg, 67% yield); mp = 130–132 °C; R_f = 0.59 (4:1 hexane/EtOAc), [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.75 (d, *J* = 8.4 Hz, 2H), 7.32–7.24 (m, 3H), 7.04 (s, 1H), 6.96 (t, *J* = 7.6 Hz, 1H), 6.85 (d, *J* = 8.4 Hz, 1H), 3.82 (s, 3H), 3.00 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.8, 144.2, 134.3, 129.9, 129.5, 129.4, 128.7, 128.6, 126.5, 122.0, 120.7, 110.6, 55.5, 35.8, 21.6; IR (Neat) ν_{max} 1593, 1492, 1349, 1250, 1159, 962, 763, cm^{-1}; HRMS (ESI) for $C_{17}H_{22}ClN_2O_3S$ $(M+NH_4)^+$: calcd 369.1040, found 369.1040.

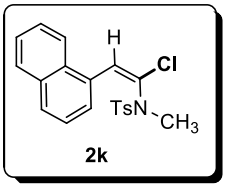
(*E*)-N-Benzyl-N-(1-chloro-2-(naphthalen-1-yl)vinyl)-4-methylbenzenesulfonamide (2j):



2j

Colorless solid; (*E*/*Z* = 100:0; 202 mg, 90% yield); mp = 193–195 °C; R_f = 0.57 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.76 (d, *J* = 6.5 Hz, 2H), 7.73 (dd, *J* = 8.0, 4.5 Hz, 2H), 7.46 (d, *J* = 7.0 Hz, 1H), 7.42–7.36 (m, 2H), 7.33 (t, *J* = 8.0 Hz, 1H), 7.31–7.27 (m, 1H), 7.25 (bs, 1H), 7.23 (bs, 2H), 6.92– 6.87 (m, 2H), 6.86–6.76 (m, 3H), 4.56 (bs, 1H), 4.05 (bs, 1H), 2.40 (s, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 144.4, 134.9, 133.1, 133.03, 132.97, 131.2, 129.8, 129.4, 129.3, 129.2, 128.8, 128.6, 128.1, 127.84, 127.81, 126.6, 125.9, 125.5, 125.2, 123.9, 52.3, 21.6; IR (Neat)ν_{max}1580, 1345, 1086, 919, 873, 798, 776, 704 cm^{-1}; HRMS (ESI) for $C_{26}H_{22}ClNNaO_2S$ $(M+Na)^+$: calcd 470.0957, found 470.0958.

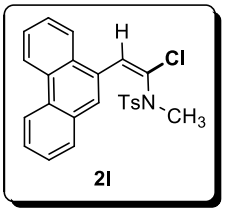
(*E*)-N-(1-Chloro-2-(naphthalen-1-yl)vinyl)-N,4-dimethylbenzenesulfonamide (2k):



2k

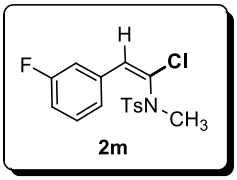
Colorless solid; (*E*/*Z* = 100:0; 175 mg, 94% yield); mp = 201–203 °C; R_f = 0.55 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.91–7.86 (m, 1H), 7.85–7.81 (m, 1H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.70 (d, *J* = 7.0 Hz, 1H), 7.53 (bd, *J* = 7.0 Hz, 2H), 7.50–7.46 (m, 2H), 7.44 (t, *J* = 7.8 Hz, 1H), 7.27 (s, 1H), 7.06 (d, *J* = 8.0 Hz, 2H), 2.99 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.0, 134.3, 133.4, 131.9, 131.2, 130.2, 130.1, 129.2, 128.8, 128.5, 128.3, 126.4, 126.3, 125.9, 125.4, 123.8, 36.2, 21.5; IR (Neat)ν_{max} 1644, 1594, 1085, 924, 891, 779, 705 cm^{-1}; HRMS (ESI) for $C_{20}H_{18}ClNO_2SNa$ $(M+Na)^+$: calcd 394.0644, found 394.0643.

(*E*)-N-(1-Chloro-2-(phenanthren-9-yl)vinyl)-N,4-dimethylbenzenesulfonamide (2l):



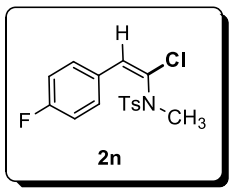
Yellow gummy liquid; (*E*/*Z* = 94:6; 177 mg, 84% yield); mp = 223–226 °C; R_f = 0.65 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.67 (d, *J* = 7.6 Hz, 1H), 8.63 (d, *J* = 8.4 Hz, 1H), 7.93 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.88–7.79 (m, 2H), 7.71–7.55 (m, 4H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.25 (bs, 1H), 6.84 (d, *J* = 8.0 Hz, 2H), 3.04 (s, 3H), 2.17 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.8, 134.7, 132.5, 131.2, 130.28, 130.26, 130.23, 130.17, 129.1, 129.0, 128.8, 128.0, 127.6, 127.1, 126.8, 126.74, 126.66, 124.7, 122.9, 122.5, 122.3, 36.4, 21.4; Minor rotamer (selected signals): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (s), 3.17 (s), 2.40 (s); ^{13}C NMR (101 MHz, $CDCl_3$) δ 129.7, 128.21, 31.5, 22.6; IR (Neat)ν_{max} 2360, 1637, 1353, 1164, 1087, 964 cm^{-1}; HRMS (ESI) for $C_{24}H_{24}ClN_2O_2S$ $(M+NH_4)^+$: calcd 439.1247, found 439.1247.

(*E*)-N-(1-Chloro-2-(3-fluorophenyl)vinyl)-N,4-dimethylbenzenesulfonamide (2m):



Yellow semi solid; (*E*/*Z* = 79:21; 160 mg, 94% yield); mp = 188–190 °C R_f = 0.52 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.78 (d, *J* = 8.0 Hz, 2H), 7.39–7.28 (m, 6H), 6.62 (s, 1H), 3.03 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 162.8 (d, *J* = 244.0 Hz, 1C), 144.6, 135.1, 134.1, 131.2, 130.1, 129.6, 129.3, 128.2, 124.6 (d, *J* = 3.0 Hz, 1C), 115.8 (d, *J* = 21.2 Hz, 1C), 115.3 (d, *J* = 23.2 Hz, 1C), 35.7, 21.6; ^{19}F NMR (376 MHz, $CDCl_3$) *δ* −111.5; Minor rotamer (selected signals): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.05–6.98 (m), 6.90 (s), 3.07 (s), 2.45 (s); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 162.5 (d, *J* = 247 Hz, 1C), 144.4, 135.1, 134.4, 131.1, 130.0, 129.7, 129.3, 128.2, 125.6 (d, *J* = 2.0 Hz, 1C), 115.9 (d, *J* = 21 Hz, 1C), 115.7 (d, *J* = 19 Hz, 1C), 35.9; IR (Neat) ν_{max} 1448, 1352, 1162, 994, 810, 784, 723, 706 cm^{-1}; HRMS (ESI) for $C_{16}H_{19}ClFN_2O_2S$ $(M+NH_4)^+$: calcd 357.0840, found 357.0841.

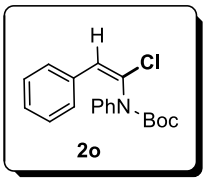
(*E*)-N-(1-Chloro-2-(4-fluorophenyl)vinyl)-N,4-dimethylbenzenesulfonamide (2n):



Pale yellow solid, (*E*/*Z* = 78:22; 155 mg, 91% yield); mp = 170–172 °C R_f = 0.53 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (500 MHz, $CDCl_3$): *δ* 7.71 (d, *J* = 8.5 Hz, 2H), 7.55–7.50 (m, 2H), 7.25 (t, *J* = 6.4 Hz, 2H), 7.19 (s, 1H), 7.0–6.95 (m, 2H), 6.54 (s, 1H), 2.95 (s, 2H), 2.37 (s, 3H); ^{13}C NMR (125.8 MHz, $CDCl_3$) *δ* 163.7 (d, *J* = 249.1 Hz, 1C), 144.6, 134.1, 131.2, 130.6

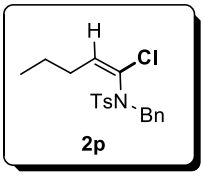
(d, J = 8.8 Hz, 1C), 129.6, 128.8, 128.3, 115.72 (d, J = 21.4 Hz, 1C), 35.7, 21.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ −112.5 (s); Minor rotamer (selected signals): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.68 (d), 6.54 (s), 2.38 (s), 3.0 (s), ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 131.3 (d, J = 7.5 Hz, 1C), 129.7, 129.4, 129.31, 129.28, 115.3 (d, J = 21.4 Hz, 1C); IR (Neat) ν_{max} 1502, 1349, 1081, 963, 818, 710 cm^{-1}; HRMS (ESI) for $C_{16}H_{19}ClFN_2O_2S$ $(M+NH_4)^+$: calcd 357.0840, found 357.0840.

(*E*)-*tert*-Butyl 1-chloro-2-phenylvinyl(phenyl)carbamate (2o):



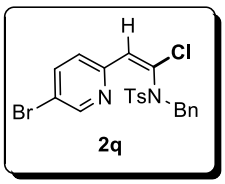
Colorless solid; (*E*/*Z* = 99:1; 213 mg, 97% yield); mp = 182–184 °C; R_f = 0.65 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43–7.37 (m, 4H), 7.36–7.30 (m, 4H), 7.30–7.27 (m, 1H), 7.25–7.17 (m, 1H), 6.68 (s, 1H), 1.34 (s, 9H), ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.8, 138.9, 133.8, 130.3, 128.90, 128.86, 128.7, 128.5, 127.6, 126.3, 124.4, 82.6, 27.9; IR (Neat)ν_{max} 3057, 2980, 2926, 1725, 1638, 1599, 1490, 1451, 1391, 1369, 1287, 1237, 1150, 1002, 892, 843 cm^{-1}; HRMS (ESI) for $C_{19}H_{20}ClNNaO_2$ $(M+Na)^+$: calcd 352.1080, found 352.1080.

(*E*)-N-Benzyl-N-(1-chloropent-1-enyl)-4-methylbenzenesulfonamide (2p):



Colorless solid; (*E*/*Z* = 99:1; 213 mg, 93% yield); mp = 168–170 °C; R_f = 0.35 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88–7.77 (m, 2H), 7.40–7.26 (m, 7H), 5.74–5.65 (m, 1H), 4.80 (d, J = 13.2 Hz, 1H), 3.92 (d, J = 13.2 Hz, 1H), 2.45 (s, 3H), 2.08–1.72 (m, 2H), 1.17–0.97 (m, 1H), 0.81–0.67 (m, 1H), 0.67–0.61 (m, 3H), ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.3, 137.8, 135.6, 134.5, 129.6, 128.41, 128.37, 128.2, 125.3, 51.4, 31.3, 21.7, 21.5, 13.5; IR (Neat)ν_{max} 1596, 1356, 1261, 1167, 1040, 904, 784, 752, cm^{-1}; HRMS (ESI) for $C_{19}H_{22}ClNNaO_2S$ $(M+Na)^+$: calcd 386.0957, found 386.0953.

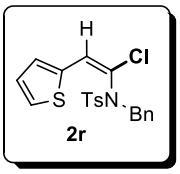
(*E*)-N-Benzyl-N-(2-(5-bromopyridin-2-yl)-1-chlorovinyl)-4-ethylbenzenesulfonamide (2q):



Brownish gummy liquid; (*E*/*Z* = 94:6; 213 mg, 77% yield); R_f = 0.51 (30% hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.45 (s, 1H), 7.86 (d, J = 8.0 Hz, 2H), 7.64–7.61 (m, 2H), 7.36 (d, J = 8.5 Hz, 2H), 7.28–7.23 (m, 2H), 7.22–7.13 (m, 3H), 6.76 (s, 1H), 4.85 (bs, 1H), 4.06 (bs, 1H), 2.47 (s, 3H), ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.5, 149.98, 144.9, 138.5,

134.7, 132.3, 131.5, 129.8, 129.7, 128.8, 128.6, 128.4, 124.3, 119.8, 52.6, 21.7; Minor rotamer (selected signals): 7.80 (d, J = 8.5 Hz), 7.65 (bd, J = 2.0 Hz), 7.60 (bd, J = 8.5 Hz), 6.72 (s), 2.46 (s); ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.6, 129.8, 128.8, 128.6, 128.1; IR (Neat)ν_{max} 1640, 1548, 1494, 1306, 1287, 1088, 1023, 879, 740 cm^{-1}; HRMS (ESI) for $C_{21}H_{18}BrClN_2NaO_2S$ $(M+Na)^+$: calcd 498.9859, found 498.9866.

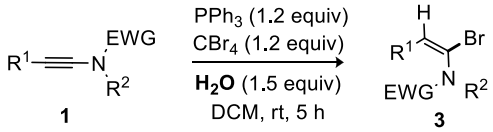
(*E*)-N-Benzyl-N-(1-chloro-2-(thiophen-2-yl)vinyl)-4-methylbenzenesulfonamide (2r):



2r

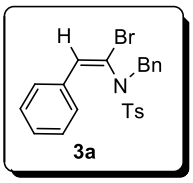
Brownish solid; (*E/Z* = 95:5; 174 mg, 86% yield); mp = 178–180 °C; R_f = 0.57 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, J = 8.0 Hz, 2H), 7.36 (d, J = 7.6 Hz, 4H), 7.25 (bd, J = 4.8 Hz, 1H), 7.16 (bd, J = 5.6 Hz, 3H), 6.99 (d, J = 3.2 Hz, 1H), 6.85 (t, J = 4 Hz, 1H), 6.77 (s, 1H), 4.89 (d, J = 12.8 Hz, 1H), 3.97 (d, J = 12.4 Hz, 1H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.6, 135.5, 134.6, 133.0, 130.2, 129.9, 129.6, 129.1, 128.7, 128.4, 128.0, 127.8, 125.9, 124.7, 52.4, 21.7; IR (Neat)ν_{max} 1594, 1086, 858, 808, 772, 715, 703 cm^{-1}; HRMS (ESI) for $C_{20}H_{19}ClNO_2S_2$ $(M+H)^+$: calcd 404.0546, found 404.0546.

General Procedure for the Bromination of 1; Synthesis of 3 (GP 3):



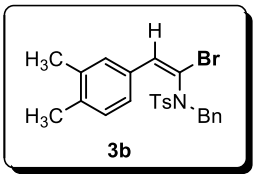
To a solution of corresponding ynamide **1** (0.5 mmol), PPh_3 (0.6 mmol), and CBr_4 (0.6 mmol) in CH_2Cl_2 (2.0 mL for 0.5 mmol) in a Schlenk tube was added H_2O (0.75 mmol). The resulting homogeneous mixture was stirred at room temperature (25 °C). Progress of the reaction was monitored periodically by TLC. The reaction mixture became heterogeneous after stirring for 5 h. Upon completion, the reaction mixture was diluted with CH_2Cl_2 (10 mL). The crude mixture was filtered through a small pad of Celite and concentrated under the reduced pressure. The crude residue was purified through column chromatography on silica gel to provide **3**.

(*E*)-N-Benzyl-N-(1-bromo-2-phenylvinyl)-4-methylbenzenesulfonamide (3a):



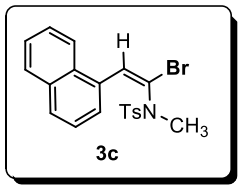
Colorless solid; (*E/Z*= 98:2; 213 mg, 96% yield); mp = 168–170 °C; R_f = 0.65 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Mixture of rotamers; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.0 Hz, 2H), 7.38 (bd, *J* = 8.0 Hz, 3H), 7.34 (bd, *J* = 6.4 Hz, 2H), 7.28 (bd, *J* = 5.2 Hz, 2H), 7.23 (bt, *J* = 7.6 Hz, 2H), 7.18–7.11 (m, 3H), 6.85 (s, 1H), 4.86 (d, *J* = 13.2 Hz, 1H), 3.97 (d, *J* = 12.8 Hz, 1H), 2.5 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.8, 139.8, 134.3, 133.6, 133.2, 129.9, 129.6, 129.2, 128.7, 128.6, 128.4, 128.2, 128.1, 119.7, 53.4, 21.7; IR (Neat)ν_{max} 1349, 1164, 1155, 1087, 1012, 866, 820, 747 cm^{-1}; HRMS (ESI) for $C_{22}H_{24}BrN_2O_2S$ $(M+NH_4)^+$: calcd 459.0742, found 459.0738.

(*E*)-N-Benzyl-N-(1-bromo-2-(3,4-dimethylphenyl)viny)-4-methylbenzenesulfonamide (3b):



Colorless solid; (*E/Z*= 88:12; 221 mg, 94% yield); mp = 205–208 °C; R_f = 0.65 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, *J* = 8.4 Hz, 2H), 7.38–7.27 (m, 4H), 7.21–7.11 (m, 3H), 7.07 (d, *J* = 7.6 Hz, 1H), 7.00 (s, 1H), 6.93 (d, *J* = 7.6 Hz, 1H), 6.76 (s, 1H), 4.82 (d, *J* = 13.2 Hz, 1H), 4.00 (d, *J* = 13.2 Hz, 1H), 2.43 (s, 3H), 2.19 (s, 3H), 2.11 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.6, 139.7, 137.5, 136.0, 134.7, 133.6, 131.3, 130.0, 129.8, 129.6, 129.4, 129.1, 128.32, 128.28, 126.3, 118.7, 53.3, 21.7, 19.7, 19.6; Minor rotamer (selected signals): 7.64 (d, *J* = 8.0 Hz), 7.24 (d, *J* = 9.6 Hz), 5.06 (s), 3.76 (s), 2.40 (s), 2.20 (s); ^{13}C NMR (101 MHz, $CDCl_3$) δ 129.7, 128.7, 128.5, 128.1, 127.7; IR (Neat) 1363, 1109, 1020, 906, 813, 767, 720 cm^{-1}; HRMS (ESI) for $C_{24}H_{24}BrNNaO_2S$ $(M+Na)^+$: calcd 492.0609, found 492.0599.

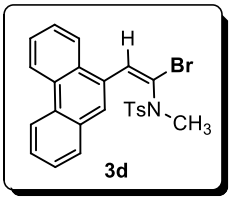
(*E*)-N-(1-Bromo-2-(naphthalen-1-yl)vinyl)-N,4-dimethylbenzenesulfonamide (3c):



Colorless solid; (*E/Z* = 96:4; 183 mg, 88% yield); mp = 168–171 °C; R_f = 0.5 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Mixture of rotamers: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83–7.69 (m, 4H), 7.60 (d, *J* = 7.2 Hz, 1H), 7.46–7.39 (m, 5H), 7.35 (bt, *J* = 7.6 Hz, 1H), 6.96 (d, *J* = 8.0 Hz, 2H), 2.87 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.1, 135.1, 133.8, 133.4, 131.02, 130.97, 129.2, 128.8, 128.5, 128.4, 126.3, 126.1, 125.9, 125.4, 123.8, 123.3, 37.0, 21.5; IR (Neat)ν_{max}

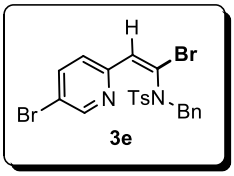
1593, 1400, 1132, 1021, 893, 927, 775, 704 cm^{-1}; HRMS (ESI) for $C_{20}H_{18}BrNO_2SNa$ $(M+Na)^+$: calcd 438.0139, found 438.0139.

(*E*)-N-(1-Bromo-2-(phenanthren-9-yl)vinyl)-N,4-dimethylbenzenesulfonamide (3d):



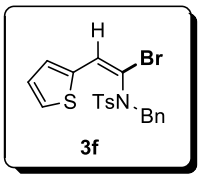
Yellow gummy liquid; (*E/Z* = 98:2; 203 mg, 87% yield); R_f = 0.53 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.67 (bd, *J* = 8.0 Hz, 1H), 8.62 (bd, *J* = 8.0 Hz, 1H), 7.92 (d, *J* = 7.6 Hz, 1H), 7.87–7.77 (m, 2H), 7.71–7.54 (m, 4H), 7.52–7.39 (m, 3H), 6.83 (d, *J* = 8.0 Hz, 2H), 3.00 (s, 3H), 2.16 (s, 3H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 143.9, 135.2, 134.4, 131.2, 130.3, 130.2, 129.9, 129.6, 129.1, 129.0, 128.1, 127.3, 127.1, 126.8, 126.73, 126.67, 124.7, 123.7, 122.9, 122.3, 37.2, 21.4; Minor rotamer (selected signals): 7.30 (d, *J* = 8.0 Hz), 3.13 (s), 2.40 (s); IR (Neat) ν_{max} 1451, 1163, 826, 768, 715, 704 cm^{-1}; HRMS (ESI) for $C_{24}H_{24}BrN_2O_2S(M+NH_4)^+$: calcd 483.0742, found 483.0745.

(*E*)-N-Benzyl-N-(1-bromo-2-(5-bromopyridin-2-yl)vinyl)-4-ethylbenzenesulfonamide (3e):



Pale yellow liquid; (*E/Z* = 99:1; 201 mg, 77% yield); R_f = 0.50 (2.3:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.42 (bd, *J* = 2.0 Hz, 1H), 7.87 (d, *J* = 8.4 Hz, 2H), 7.64–7.56 (m, 2H), 7.36 (d, *J* = 8.12 Hz, 2H), 7.33–7.28 (m, 2H), 7.23–7.14 (m, 3H), 7.0 (s, 1H), 4.88 (d, *J* = 13.2 Hz,1H), 3.97 (d, *J* = 13.2 Hz, 1H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.9, 150.0, 145.1, 139.3, 138.6, 134.2, 133.2, 129.83, 129.76, 129.1, 128.6, 128.4, 124.2, 123.7, 119.8, 53.6, 21.8; IR (Neat)ν_{max} 1349, 1166, 1091, 812, 738, 700, 658, 638 cm^{-1}; HRMS (ESI) for $C_{21}H_{19}Br_2N_2O_2S$ $(M+H)^+$: calcd 520.9534, found 520.9537.

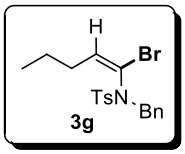
(*E*)-N-Benzyl-N-(1-bromo-2-(thiophen-2-yl)vinyl)-4-methylbenzenesulfonamide (3f)



Brown solid; (*E/Z* = 96:4; 213 mg, 83% yield,); mp = 156–158 °C; R_f = 0.57 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.0 Hz, 2H), 7.44–7.34 (m, 4H), 7.28–7.23 (m, 1H), 7.21–7.12 (m, 3H), 6.99 (bd, *J* = 3.6 Hz, 1H), 6.97 (s, 1H), 6.84 (t, *J* = 4.4 Hz, 1H), 4.92 (d, *J* = 12.8 Hz, 1H), 3.87 (d, *J* = 12.8 Hz, 1H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.8, 136.2, 134.1, 133.1, 132.9, 130.3, 130.1, 129.6, 129.4, 128.4, 128.0, 127.8, 125.9, 116.6, 53.4,

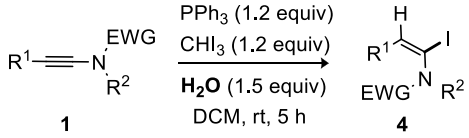
21.7; IR (Neat)ν_{max} 1455, 1362, 1166, 1021, 742, 703, 671, 609 cm^{-1}; HRMS (ESI) for $C_{20}H_{19}BrNO_2S_2$ $(M+H)^+$: calcd 448.0041, found 448.0040.

(*E*)-N-Benzyl-N-(1-bromopent-1-enyl)-4-methylbenzenesulfonamide (3g):



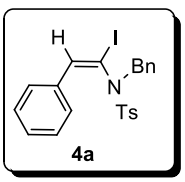
Colorless solid; (*E*/*Z* = 99:1; 201 mg, 85% yield); mp = 76–78 °C; R_f = 0.35 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.4 Hz, 2H), 7.42–7.25 (m, 7H), 5.91 (dd, *J* = 9.2, 6.0 Hz, 1H), 4.82 (d, *J* = 13.2 Hz, 1H), 3.84 (d, *J* = 13.6 Hz, 1H), 2.46 (s, 3H), 2.06–1.88 (m, 1H), 1.87–1.73 (m, 1H), 1.13–0.98 (m, 1H), 0.81–0.68 (m, 1H), 0.63 (br t, *J* = 7.2, Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.4, 142.8, 135.2, 134.4, 129.7, 129.6, 128.6, 128.4, 128.3, 116.5, 52.2, 32.5, 21.7, 21.3, 13.5; IR (Neat)ν_{max} 1596, 1494, 1356, 1209, 1167, 1027, 747, 699 cm^{-1}; HRMS (ESI) for $C_{19}H_{26}BrN_2O_2S$ $(M+NH_4)^+$: calcd 425.0898, found 425.0898.

General Procedure for the Iodination of 1; Synthesis of 4 (GP 4):



To a solution of corresponding ynamide **1** (0.5 mmol), PPh_3 (0.6 mmol), and CHI_3 (0.6 mmol) in CH_2Cl_2 (2.0 mL for 0.5 mmol) in a Schlenk tube was added H_2O (0.75 mmol). The resulting homogeneous mixture was stirred at room temperature (25 °C). Progress of the reaction was monitored periodically by TLC. The reaction mixture became heterogeneous after stirring for 5 h. Upon completion, the reaction mixture was diluted with CH_2Cl_2 (10 mL). The crude mixture was filtered through a small pad of Celite and concentrated under the reduced pressure. The crude residue was purified through column chromatography on silica gel to provide **4**.

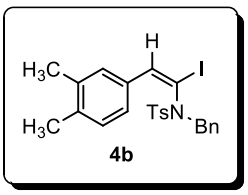
(*E*)-N-Benzyl-N-(1-Iodo-2-phenylvinyl)-4-methylbenzenesulfonamide (4a):



Reddish-orange solid; (*E*/*Z* = 99:1; 235 mg, 96% yield); mp = 212–114 °C; R_f = 0.65 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.29 (bd, *J* = 7.6 Hz, 4H), 7.24–7.09 (m, 7H), 4.86 (d, *J* = 13.2 Hz, 1H), 3.62 (d, *J* = 13.2 Hz, 1H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.3, 144.9, 134.9, 133.3, 132.98, 130.0, 129.63, 129.58,

128.8, 128.7, 128.4, 128.2, 128.0, 98.0, 55.2, 21.8; IR (Neat)ν_{max}1594, 1349, 1155, 1021, 910, 860, 761 cm^{-1}; HRMS (ESI) for$C_{22}H_{20}INNaO_2S$ $(M+Na)^+$: calcd 512.0157, found 512.0158.

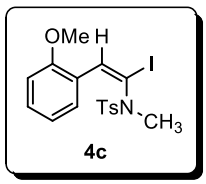
(*E*)-N-Benzyl-N-(1-iodo-2-(3,4-dimethylphenyl)vinyl)-4-methylbenzenesulfonamide (4b):



4b

Colorless solid; (*E*/*Z* = 91:9; 164 mg, 84% yield); mp = 186–189 °C; R_f = 0.64 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.4 Hz, 2H), 7.41–7.31 (m, 5H), 7.21–7.13 (m, 3H), 7.09–7.03 (m, 2H), 6.99 (s, 1H), 6.91 (d, *J* = 7.6 Hz, 1H), 4.85 (d, *J* = 13.2 Hz, 1H), 3.67 (d, *J* = 13.2 Hz, 1H), 2.45 (s, 3H), 2.16 (s, 3H), 2.11 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.2, 144.7, 137.6, 135.9, 133.8, 133.3, 132.6, 130.1, 129.9, 129.6, 129.5, 129.3, 128.33, 128.27, 126.4, 96.9, 55.1, 21.7, 19.7, 19.6; Minor rotamer (selected signals) ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 (s), 2.20 (d, *J* = 3.2 Hz); ^{13}C NMR (101 MHz, $CDCl_3$) δ 129.7, 129.4, 128.7, 128.5; IR (Neat) ν_{max} 1598, 1401, 1022, 812, 743, 715 cm^{-1}; HRMS (ESI) for $C_{24}H_{25}INO_2S$ $(M+H)^+$: calcd 518.0651, found 518.0649.

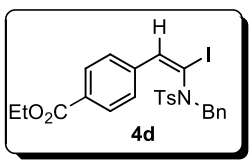
(*E*)-N-(1-Iodo-2-(2-methoxyphenyl)vinyl)-N,4-dimethylbenzenesulfonamide (4c):



4c

Colorless solid; (*E*/*Z* = 76:24; 133 mg, 60% yield); mp = 163–165 °C; R_f = 0.48 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.86 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.72 (d, *J* = 8.0 Hz, 2H), 7.51 (s, 1H), 7.29–7.23 (m, 3H), 6.93 (t, *J* = 5.2 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 3.80 (s, 3H), 2.80 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 156.5, 144.4, 139.7, 130.1, 129.5, 129.3, 129.2, 128.7, 123.9, 120.7, 110.6, 97.7, 55.5, 38.3, 21.6; Minor rotamer (selected signals): 7.85 (d, *J* = 1.6 Hz), 7.77 (d, *J* = 8.0 Hz), 7.62 (d, *J* = 8.0 Hz), 7.57 (d, *J* = 7.6 Hz), 7.38–7.32 (m), 7.30 (d, *J* = 1.6 Hz), 6.85 (d, *J* = 8.4 Hz), 3.83 (s), 2.89 (s), 2.45 (s), ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 157.1, 144.3, 133.9, 133.0, 130.4, 130.1, 128.7, 127.6, 127.6, 124.9, 121.0, 119.9, 110.5, 101.4, 38.8; IR (Neat) ν_{max} 1437, 1342, 1135, 1086, 892, 751, 705 cm^{-1}; HRMS (ESI) for $C_{17}H_{18}INNaO_3S$ $(M+Na)^+$: calcd 465.9950, found 465.9951.

(*E*)-Ethyl 4-(2-(N,4-dimethylphenylsulfonamido)-2-iodovinyl)benzoate (4d)

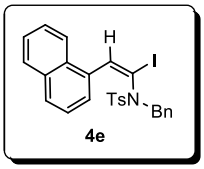


4d

Colorless solid (*E*/*Z* = 99:1; 133 mg, 94% yield); mp = 176–179 °C; R_f = 0.38 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz,

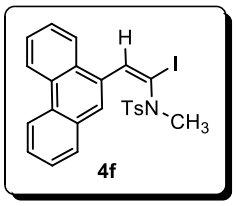
$CDCl_3$): δ 7.88–7.79 (m, 4H), 7.36 (d, J = 8.4 Hz, 2H), 7.33–7.28 (m, 4H), 7.21–7.13 (m, 4H), 4.88 (d, J = 13.2 Hz, 1H), 4.36 (q, J = 7.2 Hz, 2H), 3.62 (d, J = 13.2, 1H), 2.47 (s, 3H), 1.39 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.2, 147.2, 145.1, 139.0, 133.2, 132.8, 130.1, 129.98, 129.7, 129.5, 129.3, 128.6, 128.5, 128.4, 100.6, 61.0, 55.3, 21.8, 14.3; IR (Neat) ν_{max} 1496, 1456, 1309, 1125, 907, 750, 685 cm^{-1}; HRMS (ESI) for $C_{25}H_{24}INNaO_4S$ $(M+Na)^+$: calcd 584.0368, found 584.0374.

(*E*)-N-Benzyl-N-(1-iodo-2-(naphthalen-1-yl)vinyl)-4-methylbenzenesulfonamide (4e):



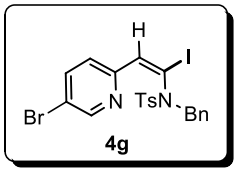
Pale yellow solid; (E/Z = 92:8; 229 mg, 89% yield); mp = 193–195 °C; R_f = 0.58 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Mixture of rotamers; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79–7.71 (m, 5H), 7.46–7.35 (m, 2H), 7.33–7.23 (m, 5H), 7.04–6.96 (m, 2H), 6.91–6.79 (m, 3H), 4.66 (d, J = 12.8 Hz, 1H), 3.65 (d, J = 13.6 Hz, 1H), 2.40 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.0, 144.6, 133.7, 132.9, 132.6, 132.0, 130.7, 129.6, 129.4, 129.4, 128.6, 128.0, 127.9, 126. 2, 125.9, 125.5, 125.2, 123.9, 99.1, 54.6, 21.7 IR (Neat)ν_{max} 1595, 1345, 1163, 1087, 920, 870, 812, 658 cm^{-1}; HRMS (ESI) for $C_{26}H_{22}INO_2SNa$ $(M+Na)^+$: calcd 562.0314, found 562.0305.

(E)-N-(1-Iodo-2-(phenanthren-9-yl)vinyl)-N,4-dimethylbenzenesulfonamide(4f):



Yellow semisolid; (E/Z = 84:16; 251 mg, 98% yield); R_f = 0.53 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.68 (dd, J = 8.4, 1.2 Hz, 1H), 8.63 (d, J = 8.4 Hz, 1H), 7.93–7.87 (m, 1H), 7.84–7.78 (m, 2H), 7.75 (bd, J = 0.8 Hz, 1H), 7.73–7.56 (m, 4H), 7.40 (d, J = 8.0 Hz, 2H), 6.82 (d, J = 8.0 Hz, 2H), 2.86 (s, 3H), 2.16 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.0, 134.0, 131.2, 131.0, 130.3, 130.2, 129.7, 129.65, 129.1, 129.0, 128.8, 128.3, 127.2, 127.17, 126.8, 126.7, 126.69, 124.7, 122.9, 122.4, 100.3, 38.8, 21.4; Minor rotamer (selected signals): 8.73–8.70 (m), 8.17–8.13 (m), 7.33 (d, J = 8.0 Hz), 3.0 (s), 2.42 (s); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.0, 140.0, 128.0, 127.1, 126.9, 125.5, 122.9, 122.6, 38.9, 21.51; IR (Neat) ν_{max} 1449, 1351, 1160, 941, 891, 745, 713 cm^{-1}; HRMS (ESI) for $C_{24}H_{24}IN_2O_2S$ $(M+NH_4)^+$: calcd 531.0603, found 531.0603.

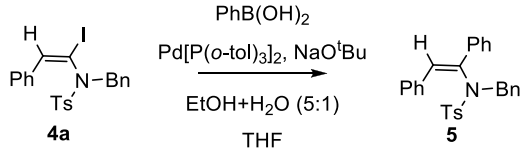
(*E*)-N-Benzyl-N-(1-iodo-2-(5-bromopyridin-2-yl)vinyl)-4-methylbenzenesulfonamide (4g):



4g

pale yellow liquid; (*E*/*Z* = 99:1; 211 mg, 78% yield); R_f = 0.5 (2.3:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (s, 1H), 7.86 (bd, *J* = 8.0 Hz, 2H), 7.74–7.60 (m, 2H), 7.36 (bd, *J* = 8.0 Hz, 2H), 7.26 (bd, *J* = 6.0 Hz, 2H), 7.24–7.13 (m, 3H), 6.76 (s, 1H), 4.86 (br s, 1H), 4.08 (br s, 1H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 150.5, 150.0, 144.9, 138.5, 134.7, 134.6, 133.3, 131.6, 129.7, 128.8, 128.5, 128.4, 124.3, 119.8, 52.6, 21.7; IR (Neat)ν_{max} 1456, 1093, 812, 741, 702, 661, 613 cm^{-1}; HRMS (ESI) for $C_{21}H_{19}BrIN_2O2S$ $(M+H)^+$: calcd 568.9395, found 568.9393.

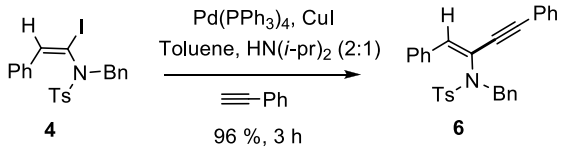
General Procedure for the Suzuki Reaction of 4a; Synthesis of (*Z*)-N-Benzyl-N-(1,2-diphenylvinyl)-4-methylbenzenesulfon amide 5 (GP 5):[3]



A mixture of α-iodoenamide **4a** (245 mg, 0.5 mmol), phenylboronic acid (74 mg, 0.6 mmol), $Pd[(P(o\text{-}Tol)_3]_2$ (15 mg, 10 mol %), and Na^{t}OBu (96 mg, 1.0 mmol) was dissolved in EtOH/H_2O (5:1; 3.0 mL. The resulting solution was stirred at room temperature for 3 h. Progress of the reaction was monitored by TLC. Upon completion, EtOH was removed under vacuum. The reaction mixture was diluted with EtOAc and washed with water and brine. The organic layer was dried over Na_2SO_4 and evaporated. The residue was purified by flash column chromatography to afford **5**. colorless gummy liquid; (*E/Z* = 81:19; 216 mg, 98% yield); R_f.= 0.65 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79 (d, *J* = 8.4 Hz, 2H), 7.35–7.28 (m, 3H), 7.28–7.24 (m, 3H), 7.20–7.15 (m, 3H), 7.14–7.08 (m, 2H), 7.08–7.03 (m, 2H), 6.85 (bd, *J* = 0.8 Hz, 1H), 6.84–6.79 (m, 3H), 6.56 (s, 1H), 4.51 (s, 2H), 2.46 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 143.6, 137.5, 137.3, 136.5, 136.3, 135.9, 135.6, 135.3, 130.6, 130.0, 129.7, 129.3, 129.0, 128.4, 128.3, 128.0, 127.9, 127.7, 52.2, 21.7; Minor rotamer (selected signals): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* = 8.4 Hz), 7.48 (d, *J* = 14.4 Hz), 7.24–7.20 (m), 4.64 (s), 2.41 (s); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 128.7, 128.6, 128.4,

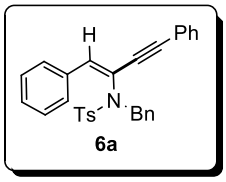
127.6, 127.2, 127.0, 126.9, 126.5, 115.3, 112.1, 49.5; IR (Neat)ν_{max} 1643, 1489, 1260, 1057, 942, 810 cm^{-1}; HRMS (ESI) for $C_{28}H_{26}NO_2S$ $(M+H)^+$: calcd 440.1684, found 440.1687.

General Procedure for the Sonagashira Reaction of 4; Synthesis of 6 (GP 6):[4]



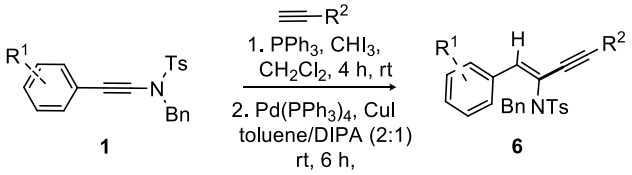
To a solution of α-iodoenamide **4** (245 mg, 0.5 mmol) in toluene/diisopropylamine (2:1; 3.0 mL) was added $Pd(PPh_3)_4$ (58 mg, 10 mol %). After 2 h, CuI (41 mg, 7 mol %) and phenylacetylene (66 µL, 0.6 mmol) were added to the reaction mixture and stirred for 6 h at room temperature. Upon completion, the crude reaction mixture was filtered through a small pad of Celite and concentrated under the reduced pressure. The crude residue was purified using column chromatography on silica gel to provide **6**.

Synthesis of (*Z*)-N-Benzyl-N-(1,2-diphenylvinyl)-4-methylbenzenesulfonamide (6a):



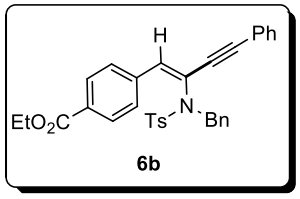
Brown gummy liquid; (*E*/*Z* = 67:33; 223 mg, 96% yield); R_f = 0.65 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer (*E*): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, *J* = 8.0 Hz, 2H), 7.77 (d, *J* = 6.8 Hz, 2H), 7.41–7.27 (m, 10H), 7.34–7.28 (m, 2H), 7.19–7.13 (m, 3H), 6.99 (s, 1H), 4.69 (s, 2H), 2.40 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.7, 141.5, 135.9, 134.1, 131.4, 130.2, 129.7, 129.6, 129.2, 129.0, 128.8, 128.5, 128.3, 128.2, 127.8, 122.1, 117.0, 96.7, 84.0, 52.01, 21.5; Minor rotamer (selected signals): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.0 Hz), 7.71 (bt, *J* = 1.6 Hz), 7.43 (d, *J* = 7.2 Hz), 7.34–7.28 (m), 7.13–7.08 (m), 6.84 (s,), 2.39 (s) ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.9, 140.9, 135.6, 135.0, 134.4, 133.9, 129.2, 129.1, 129.0, 128.6, 128.4, 128.3, 128.2, 128.1, 128.0, 122.2, 118.3, 91.3, 86.0, 53.4; IR (Neat)ν_{max} 2356, 1594, 1089, 925, 816, 755 cm^{-1}; HRMS (ESI) for $C_{30}H_{25}NO_2S$ $(M+Na)^+$: calcd 486.1504, found 486.1505.

General Procedure for the Sequential Iodination and Alkynylation of 1; Synthesis of 6 (GP 7):[5]



To a solution of corresponding ynamide **1** (0.5 mmol), PPh_3 (0.6 mmol, 1.2 equiv), and CHI_3 (0.6 mmol, 1.2 equiv) in CH_2Cl_2 (2.0 mL) in a Schlenk tube was added H_2O (0.75 mmol, 1.5 equiv). The homogeneous solution was stirred at 25 °C for 5 h. The reaction mixture turned to heterogeneous. Solvent CH_2Cl_2 was then completely evaporated. To this reaction mixture, $Pd(PPh_3)_4$ (10 mol %), toluene/diisopropylamine (2:1; 2.0 mL) was added. After 2 h, CuI (7 mol %) and alkyne (0.6 mmol) were added to the reaction mixture and stirred for 6 h at room temperature. Upon completion, the crude reaction mixture was filtered through a small pad of Celite and concentrated under the reduced pressure. The crude residue was purified using column chromatography on silica gel to provide **6**.

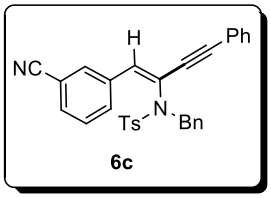
(*Z*)-Ethyl-4-(2-(N-benzyl-4-methylphenylsulfonamido)-4-phenylbut-1-en-3-ynyl)benzoate (6b)



Colorless gummy liquid; (*E*/*Z* = 63:37; 164 mg, 61% yield); R_f = 0.40 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer (*E*): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.81 (d, *J* = 8.0 Hz, 2H), 7.76 (d, *J* = 8.4 Hz, 2H), 7.32–7.27 (m, 8H), 7.17–7.08 (m, 4H), 6.96 (s, 1H), 4.64 (s, 2H), 4.43–4.32 (m, 2H), 2.35 (s, 3H), 1.44–1.33 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 166.1, 143.9, 140.0, 138.4, 135.7, 134.2, 131.5, 129.8, 129.7, 129.5, 129.5, 128.9, 128.7, 128.5, 128.3, 128.3, 128.1, 121.7, 119.2, 97.6, 83.6, 61.0, 52.2, 21.5, 14.3; Minor rotamer (selected signals): 7.92 (d, *J* = 8.8 Hz, 1.4 Hz), 7.85 (d, *J* = 8.0 Hz), (d, *J* = 8.4 Hz), 7.41–7.32 (m), 7.24–7.20 (m), 7.08–7.03 (m), 6.80 (s), 4.43–4.32 (m), 2.33 (s), 1.44–1.33 (m); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 166.3, 144.1, 139.6, 138.2, 125.5, 134.8, 131.5, 130.5, 130.3, 129.6, 129.3, 129.0, 128.9, 128.5, 128.2, 127.8, 121.9, 120.4, 92.4, 85.6, 61.1, 53.4, 14.4; IR (Neat)ν_{max} 2975,

1715, 1605, 1436, 1277, 1101, 1085 cm^{-1}; HRMS (ESI) for $C_{33}H_{29}NO_4S$ $(M+Na)^+$: calcd 558.1715, found 558.1716.

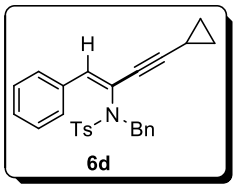
(*Z*)-N-Benzyl-N-(1-(3-cyanophenyl)-4-phenylbut-1-en-3-yn-2-yl)-4- methylbenzene sulfo namide (6c):



6c

Colorless gummy liquid; (*E*/*Z* = 53:47; 159 mg, 65% yield) was obtained in as; R_f = 0.65 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17 (s, 1H), 7.87 (t, *J* = 6.4 Hz, 2H), 7.81 (d, *J* = 8.4 Hz, 2H), 7.54 (bs, 1H), 7.39–7.23 (m, 9H), 7.22–7.17 (m, 1H), 7.13–7.04 (m, 2H), 6.74 (s, 1H), 4.63 (s, 2H), 2.34 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.2, 138.3, 135.3, 134.1, 133.6, 132.9, 131.9, 131.6, 131.5, 129.8, 129.7, 129.6, 129.0, 128.6, 128.4, 128.3, 128.1, 127.2, 120.9, 118.6, 112.2, 92.6, 85.0, 53.3, 21.5; Minor rotamer (selected signals): ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.76 (dd, *J* = 8.0, *J* = 1.6 Hz), 7.47 (d, *J* = 7.6 Hz), 7.42 (d, *J* = 8.0), 7.39–7.23 (m), 7.22–7.17 (m), 7.13–7.04 (m), 6.88 (s), 4.40 (bs), 4.12 (d, *J* = 6 Hz), 2.37 (s), ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.0, 138.3, 136.9, 136.3, 135.5, 135.1, 134.7, 131.8, 129.2, 129.0, 128.6, 127.9, 121.7, 121.3, 119.6, 112.6, 98.2, 83.1, 52.1, 47.3, IR (Neat)ν_{max} 2230, 2192, 1162, 1090, 1052, 1030, 663 cm^{-1}; HRMS (ESI) for $C_{31}H_{24}N_2NaO_2S$ $(M+Na)^+$: calcd 511.1456, found 511.1456.

(*Z*)-N-Benzyl-N-(4-cyclopropyl-1-phenylbut-1-en-3-yn-2-yl)-4-methylbenzene sulfonamide (6d)

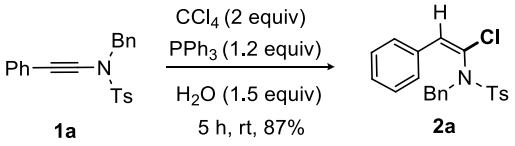


6d

Colorless gummy liquid; (*E/Z* = 86:14; 145 mg, 68% yield); R_f = 0.45 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.67–7.60 (m, 2H), 7.37–7.28 (m, 5H), 7.26–7.18 (m, 5H), 6.80 (s, 1H), 4.5 (s, 2H), 2.45 (s, 3H), 1.24–1.16 (m, 1H), 0.79–0.73 (m, 2H), 0.45–0.40 (m, 2H), ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 143.7, 140.0, 136.1, 136.0, 134.3, 129.3, 128.9, 128.83, 128.75, 128.6, 128.4, 128.1, 127.6, 117.3, 101.6, 70.4, 51.7, 21.6, 8.6, 0.13; Minor rotamer (selected signals): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, *J* = 8.4 Hz), 7.56–7.7.50 (m), 7.15–7.09 (m), 7.02–6.95 (m), 6.6 (s), 3.87 (s), 3.87 (s), 1.31–1.24 (m), 1.15–1.08 (m), 0.73–0.69 (m), 0.40–0.38 (m); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 144.9, 139.6, 136.6, 136.6, 134.6, 134.1, 129.8, 129.7, 129.6, 129.2, 128.5, 128.1, 127.98, 127.95, 127.75,

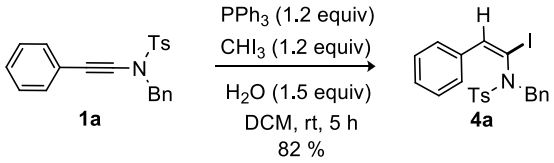
72.4, 53.0, 21.6, 8.5, -0.20; IR (Neat)ν_{max} 2197, 1704, 1452, 1085, 1030, 942 cm^{-1}; HRMS (ESI) for $C_{27}H_{25}NNaO_2S$ $(M+Na)^+$: calcd 450.1504, found 450.1506.

Gram Scale Synthesis of 2a:



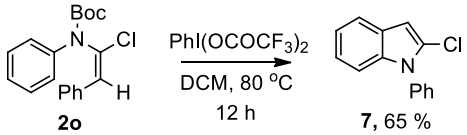
Following the general procedure (GP-2), a mixture of **1a** (1.0 g, 2.8 mmol), PPh_3 (871 mg, 3.3 mmol), and CCl_4 (0.5 mL, 5.5 mmol) in CH_2Cl_2 (6.0 mL) and H_2O (75 µL, 4 mmol) was taken in a Schlenk tube. The resulting mixture was stirred at room temperature (25 °C) for 3 h. The crude mixture was filtered through a small pad of Celite and concentrated under the reduced pressure. The crude residue was purified through column chromatography on silica gel to provide **2a** (959 mg) in 87 % yield.

Gram Scale Synthesis of 4a:



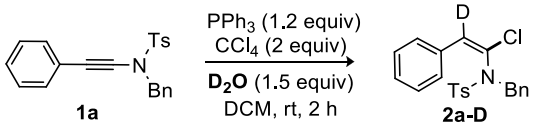
Following the general procedure (GP-4), a mixture of **1a** (1.0 g, 2.8 mmol), PPh_3 (871 mg, 3.3 mmol), and CHI_3 (1.3 g, 3.3 mmol) in CH_2Cl_2 (6.0 mL) and H_2O (75 µL, 4 mmol) was taken in a Schlenk tube. The resulting mixture was stirred at room temperature (25 °C) for 5 h. The crude mixture was filtered through a small pad of Celite and concentrated under the reduced pressure. The crude residue was purified through column chromatography on silica gel to provide **4a** (1.1 g) in 82 % yield.

Synthesis of 2-chloro-1-phenyl-1H-indole 7:[6]



A solution of enamide **2o** (165 mg, 0.5 mmol) and [bis(trifluoroacetoxy)iodo]benzene (259 mg, 0.6 mmol) in CH_2Cl_2 (2.0 mL) was heated in a Schlenk tube at 80 °C for 12 h. Upon completion, the crude mixture filtered through a small pad of Celite and concentrated under the reduced pressure. The crude residue was purified using column chromatography on silica gel to provide **7.** brown solid; (74 mg, 65% yield); mp = 210–212 °C; R_f = 0.43 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 7.6 Hz, 2H), 7.61–7.53 (m, 2H), 7.53–7.44 (m, 5H), 7.40 (t, J = 7.2 Hz, 1H), ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.6, 134.5, 132.0, 129.5, 129.2, 128.8, 128.7, 127.3, 126.3, 124.6, 120.0, 111.3; IR (Neat)ν_{max} 1644, 1594, 1356, 1155, 1085, 958, 924, 891, 844, 824, 799, 779, 730, 705 cm^{-1}; HRMS (ESI) for $C_{14}H_{11}ClN$ $(M+H)^+$: calcd 228.0580, found 228.0576.

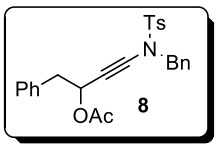
Chlorination of 1a in D_2O:



The ynamide **1a** (181 mg, 0.5 mmol), PPh_3 (158 mg, 0.6 mmol), and CCl_4 (97µL, 1 mmol) were dissolved with CH_2Cl_2 (2.0 mL) in a Schlenk tube followed by the addition of D_2O (14 µL, 0.8 mmol). The reaction mixture was stirred at room temperature (25 °C). After stirring for 2 h, the reaction mixture was diluted with CH_2Cl_2 (10 mL). The crude mixture was filtered through a small pad of Celite and concentrated under the reduced pressure. The crude residue was purified using column chromatography on silica gel to provide **2a-D** and **2a** (80:20; 192 mg) in 96 % yield as colorless solid. Analytical data is exactly matching with the values mentioned above for **2a**.

Ratio of **2a-D** and **2a** is determined based on the integration of the respective CH_3-proton of N-Ts of **2a-D** (δ = 2.46, s, 3H) and alkenyl proton of **2a** (δ = 6.60, s, 0.19H).

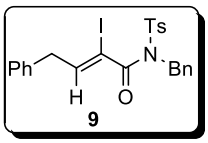
4-(N-Benzyl-4-methylphenylsulfonamido)-1-phenylbut-3-yn-2-yl acetate (8):[7]



Colorless thick liquid; (192 mg, 86% yield); R_f = 0.40 (4:1 hexane/EtOAc); [Silica, UV and I_2]; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, J = 6.4 Hz, 2H), 7.36–7.21 (m, 10H), 7.13 (dd, J = 5.6, 2.8 Hz, 2H), 5.60 (t, J = 5.2 Hz, 1H), 4.52 (d, J = 11.2 Hz, 1H), 4.39 (d, J = 11.2 Hz, 1H), 3.04–2.90 (m, 2H), 2.45 (s, 3H), 2.01 (s,

3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.7, 144.5, 135.9, 134.4, 129.6, 128.8, 128.7, 128.5, 128.3, 127.8, 127.2, 126.8, 80.1, 69.3, 65.1, 55.4, 40.9, 21.6, 20.9; IR (Neat)ν_{max} 2252, 1456, 1367, 1309, 1234, 1090 cm^{-1}; HRMS (ESI) for $C_{26}H_{25}NNaO_4S$ $(M+Na)^+$: calcd 470.1402, found 470.1402.

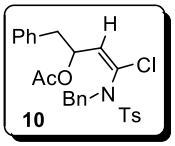
(*Z*)-N-Benzyl-2-iodo-4-phenyl-N-tosylbut-2-enamide (9):[7]



9

Brown gummy liquid (*E*/*Z* = 88:12; 114 mg, 96 % yield); $R_{f.}$ = 0.52 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (bd, *J* = 8.4 Hz, 2H), 7.40–7.25 (m, 8H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.11–7.04 (m, 2H), 6.51 (d, *J* = 14.8 Hz, 1H), 5.11 (s, 2H), 3.48 (br d, *J* = 6.0 Hz, 2H), 2.43 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.8, 149.1, 144.6, 137.3, 136.9, 136.8, 129.6, 128.9, 128.7, 128.6, 128.0, 127.72, 127.66, 126.7, 122.5, 49.3, 38.7, 21.6; Minor rotamer (selected signals): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.4–7.28 (m, 3H), 6.17 (d, *J* = 10.8 Hz), 4.91 (s), 2.47 (s); ^{13}C NMR (101 MHz, $CDCl_3$) δ 129.2, 128.5, 127.0, IR (Neat)ν_{max} 1677, 1353, 975, 915, 811 cm^{-1}; HRMS (ESI) for $C_{24}H_{23}INO_3S$ $(M+H)^+$: calcd 532.0443, found 532.0449.

(*E*)-4-(N-Benzyl-4-methylphenylsulfonamido)-4-chloro-1-phenylbut-3-en-2-yl acetate (10)



10

Colorless solid; (*E*/*Z* = 91:09; 103 mg, 95% yield); $R_{f.}$ = 0.45 (4:1 hexane/EtOAc); [Silica, UV and I_2]; Major rotamer: ^{1}H NMR (500 MHz, DMSO): δ 7.87 (d, *J* = 8.5 Hz, 2H), 7.45–7.15 (m, 12H), 6.84 (bs, 1H), 5.74 (bs, 1H), 4.85 (bs, 1H), 4.03 (bs, 1H), 3.26 (bs, 1H), 2.95 (bs, 1H), 2.50 (s, 3H), 1.76 (bs, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 144.7, 136.5, 134.8, 133.6, 133.2, 130.0, 129.8, 128.6, 128.2, 126.6, 122.1, 70.8, 52.0, 39.4, 21.7, 21.0; Minor rotamer (selected signals): ^{1}H NMR (500 MHz, DMSO): δ 7.52 (d, *J* = 8.0 Hz), 7.10–7.06 (m), 2.42 (s), 1.94 (br s); ^{13}C NMR (101 MHz, $CDCl_3$) δ 133.2, 127.7, 69.5, IR (Neat)ν_{max} 1732, 1496, 1458, 1359, 1090, 745 cm^{-1}; HRMS (ESI) for $C_{26}H_{26}ClNNaO_4S$ $(M+Na)^+$: calcd 506.1169, found 506.1169.

X-ray crystallography:

X-ray reflections for **2a** were collected on an Oxford Xcalibur Gemini Eos CCD diffractometer using Mo-Kα, radiation. Data reduction was performed using CrysAlisPro (version 1.171.33.55).[8] OLEX2-1.0 and SHELX-TL 97 programme were used to solve and refine the data.[9] All non-hydrogen atoms were refined anisotropically, and C−H hydrogens were fixed.

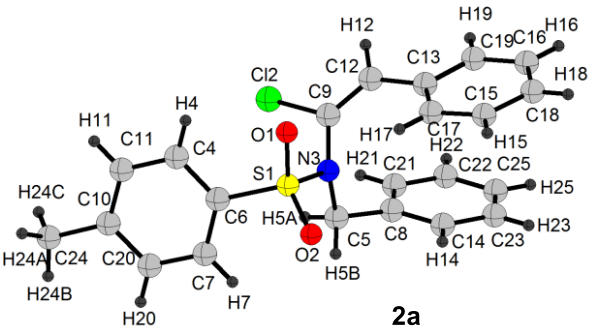


Figure 1. Molecular structures of compounds **2a;** thermal ellipsoids are set at 30% probability**.** Oxygen (red), nitrogen (blue), and sulphur (yellow).

Table 1. Crystallographic Data for Compound **2a**

Compound	**2a**
formula	$C_{22}H_{20}ClNO_2S$
Mw	397.90
crystal system	Monoclinic
space group	I a
T [K]	293 K
a [Å]	11.4861(4)
b [Å]	14.0594(4)
c [Å]	13.4860(4)
α [°]	90
β [°]	111.393(4)
γ [°]	90
V [Å^3]	2027.77(12)

Z	4
ρ_{calcd} [g cm^{-3}]	1.303
μ [mm^{-1}]	2.758
total reflns	3990
unique reflns	2273
observed	2237
$R_1[I>2\sigma(I)]$	0.0530
wR2 [all]	0.1455
GOF	1.075
Diffractometer	Xcalibur Gemini Eos CCD

References:

(1) (a) Y. Zhang, R. P. Hsung, M. R. Tracey, K. C. M. Kurtz, E. L. Vera, *Org. Lett*. 2004, **6**, 1151; (b) X. Zhang, Y. Zhang, J. Huang, R. P. Hsung, K. C. M. Kurtz, J. Oppenheimer, M. E. Petersen, I. K. Sagamanova, L. Shen, M. R. Tracey, *J. Org. Chem*. 2006, **71**, 4170.

(2) (**a**) A. H. Sato, K. Ohashi, T. Iwasawa, *Tetrahedron Lett*. 2013, **54**, 1309; (**b**) M. R. Tracey, Y. Zhang, M. O. Frederick, A. M. Jason, R. P. Hsung, *Org. Lett*. 2004, **6**, 2209; (**c**) W. D. Mackay, M. Fistikci, R. M. Carris, J. S. Johnson, *Org. Lett*. 2014, **16**, 1626; (**d**) T. Okitsu, K. Nakata, K. Nishigaki, N. Michioka, M. Karatani, A. Wada, *J. Org. Chem*. 2014, **79**, 5914; (**e**) H. Huang, G. He, G. Zhu, X. Zhu, S. Qiu, H. Zhu, *J. Org. Chem*. 2015, **80**, 3480; (**f**) S. J. Mansfield, C. D. Campbell, M. W. Jones, and E. A. Anderson, *Chem. Comm***.,** 2015, **51**, 3316.

(3) G.-P. Lu, K. R. Voigtritter, C. Cai, B. H. Lipshutz, *J. Org. Chem.*, 2012, **77**, 3700.

(4) K. Sonagashira, Y. Tohda, N. Hagihara, *Tetrahedron Lett.,* 1975, **16**, 4467.

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(6) Y. Du, R. Liu, G. Linn, K. Zhao, *Org. Lett*., 2006, **8**, 5919.

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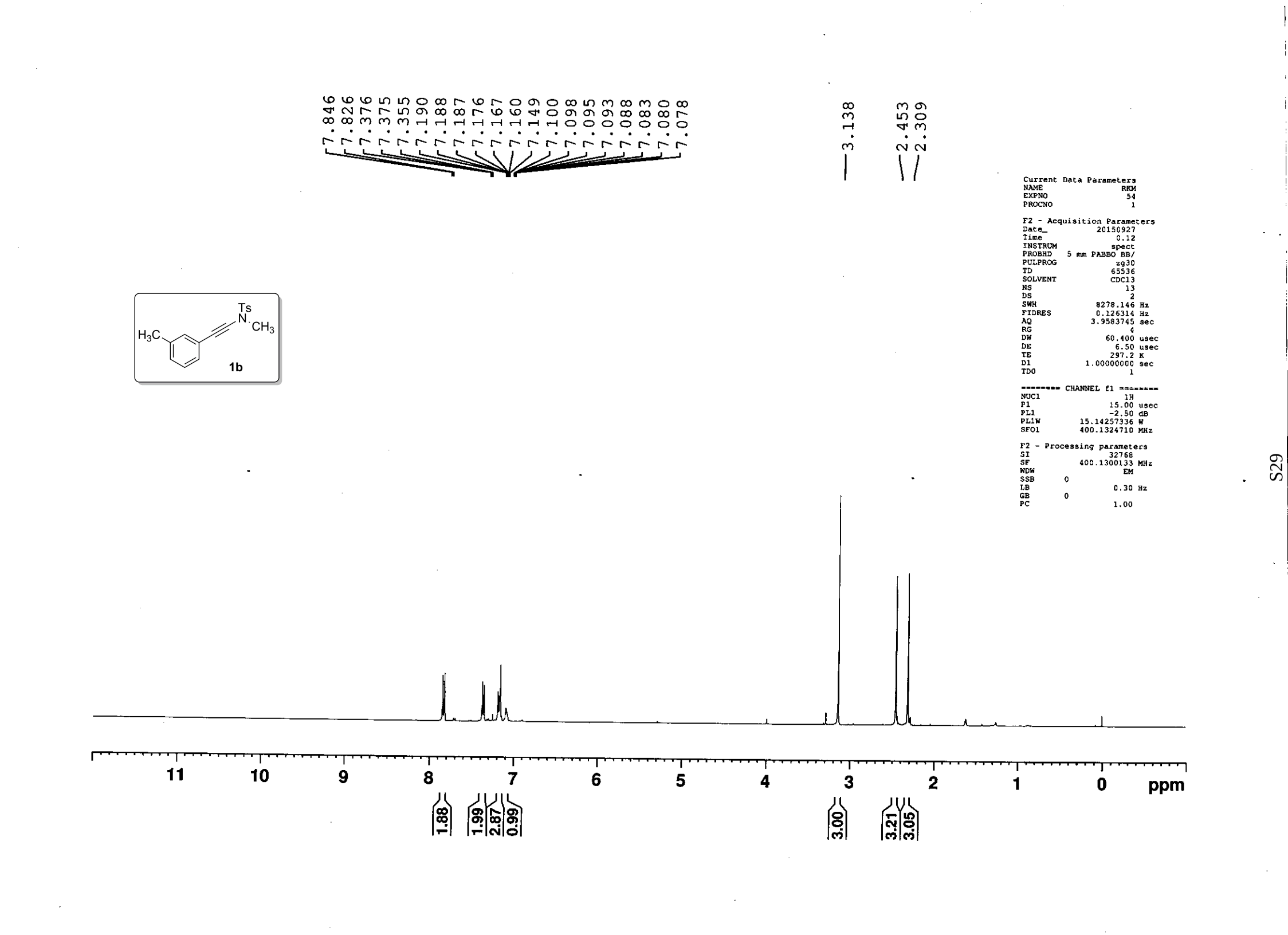
(9) (a) *SMART* (*version 5.625*), *SHELX-TL* (*version 6.12*), Bruker AXS Inc.Madison, WI, 2000; (b) G. M. Sheldrick, *SHELXS-97*, University of Gottingen, Germany, 1997**.**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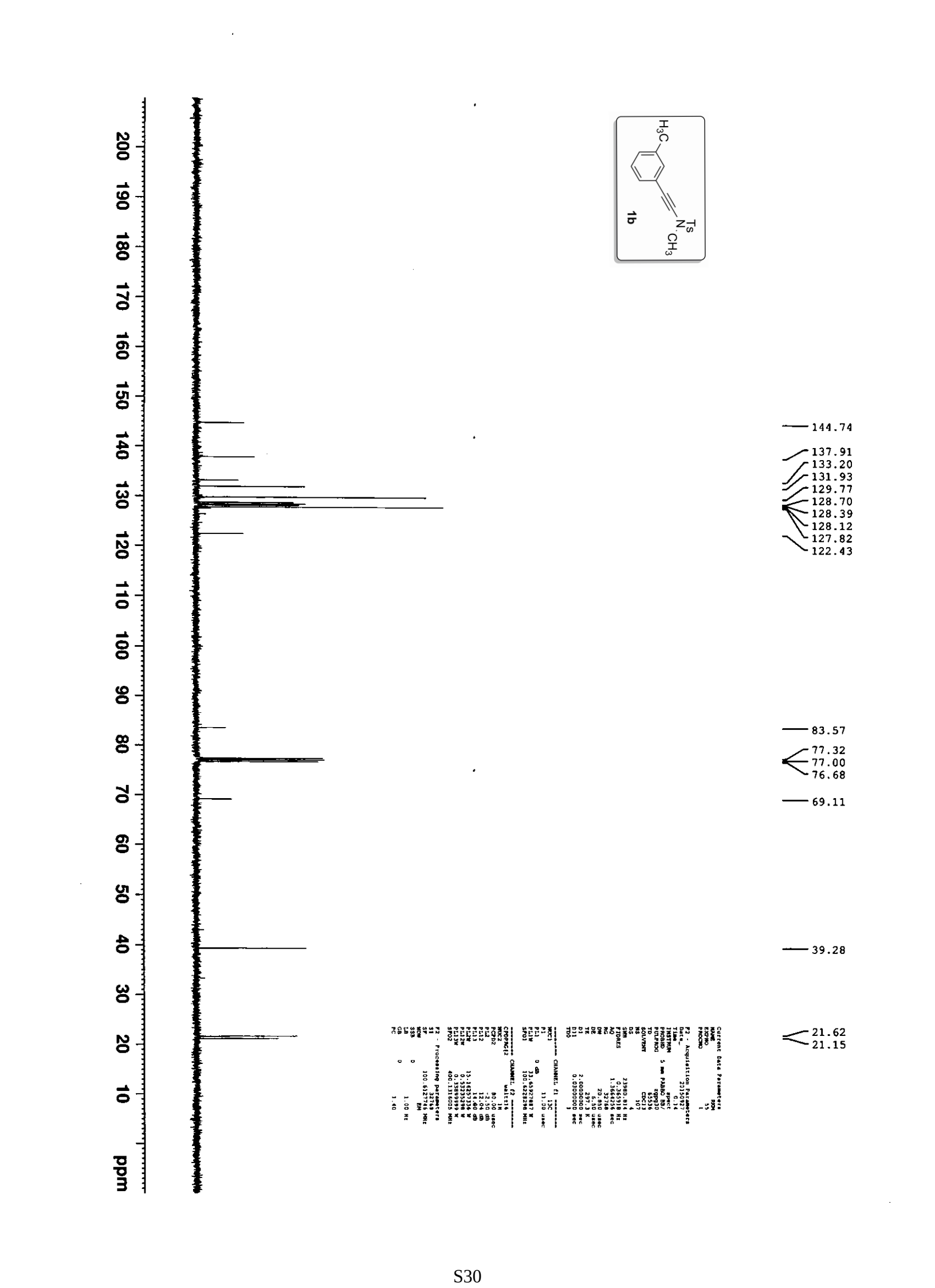
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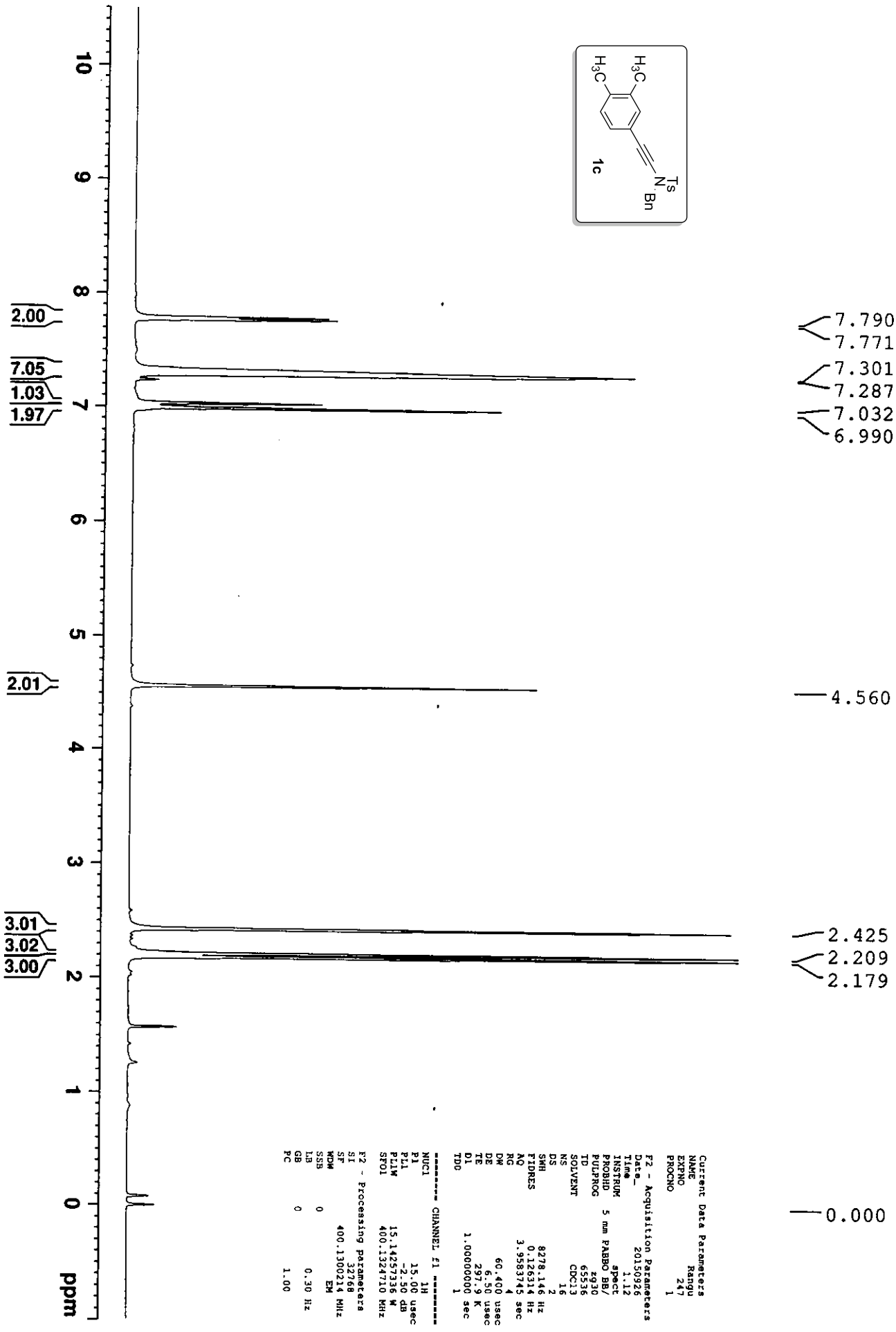
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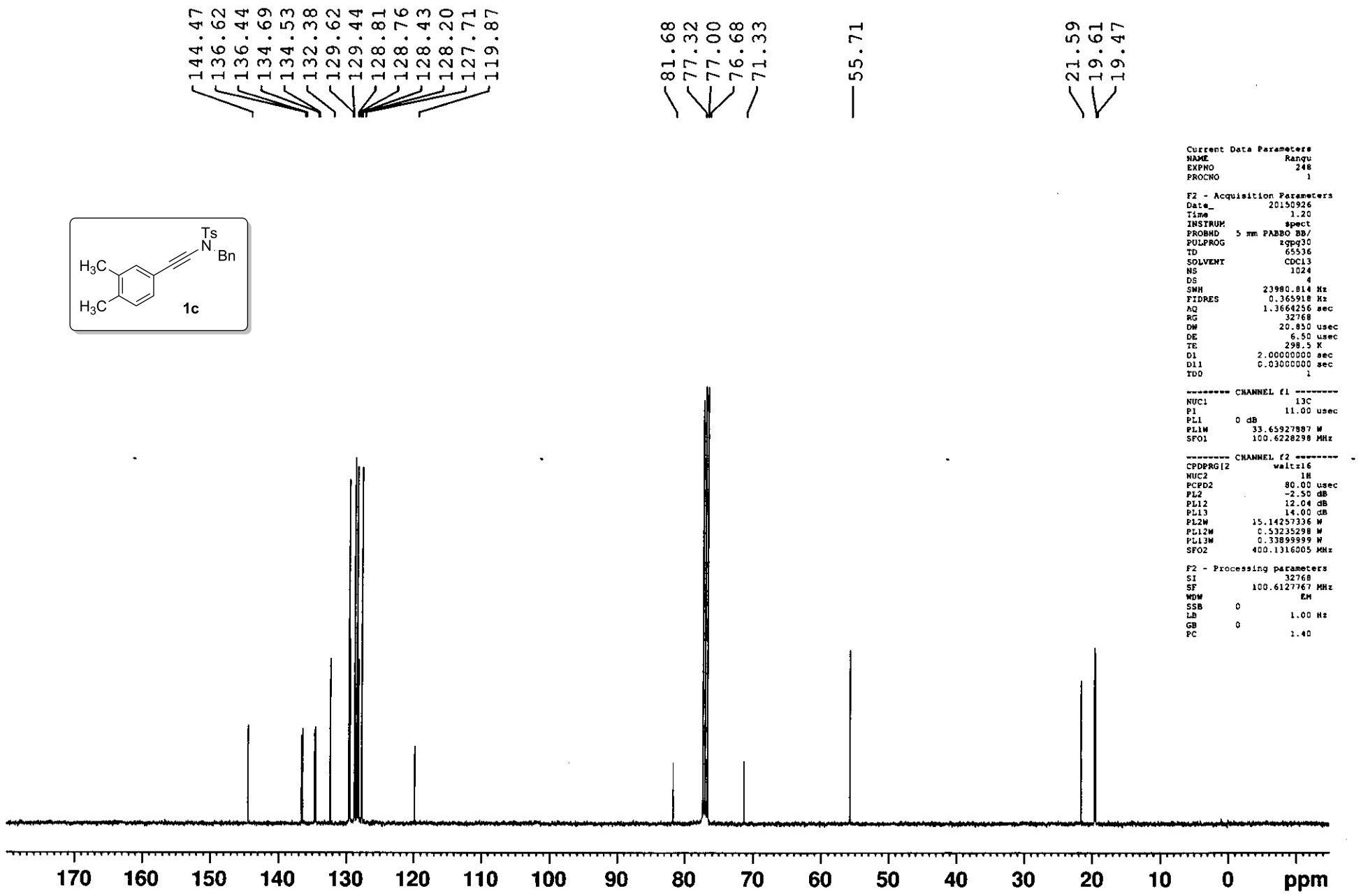
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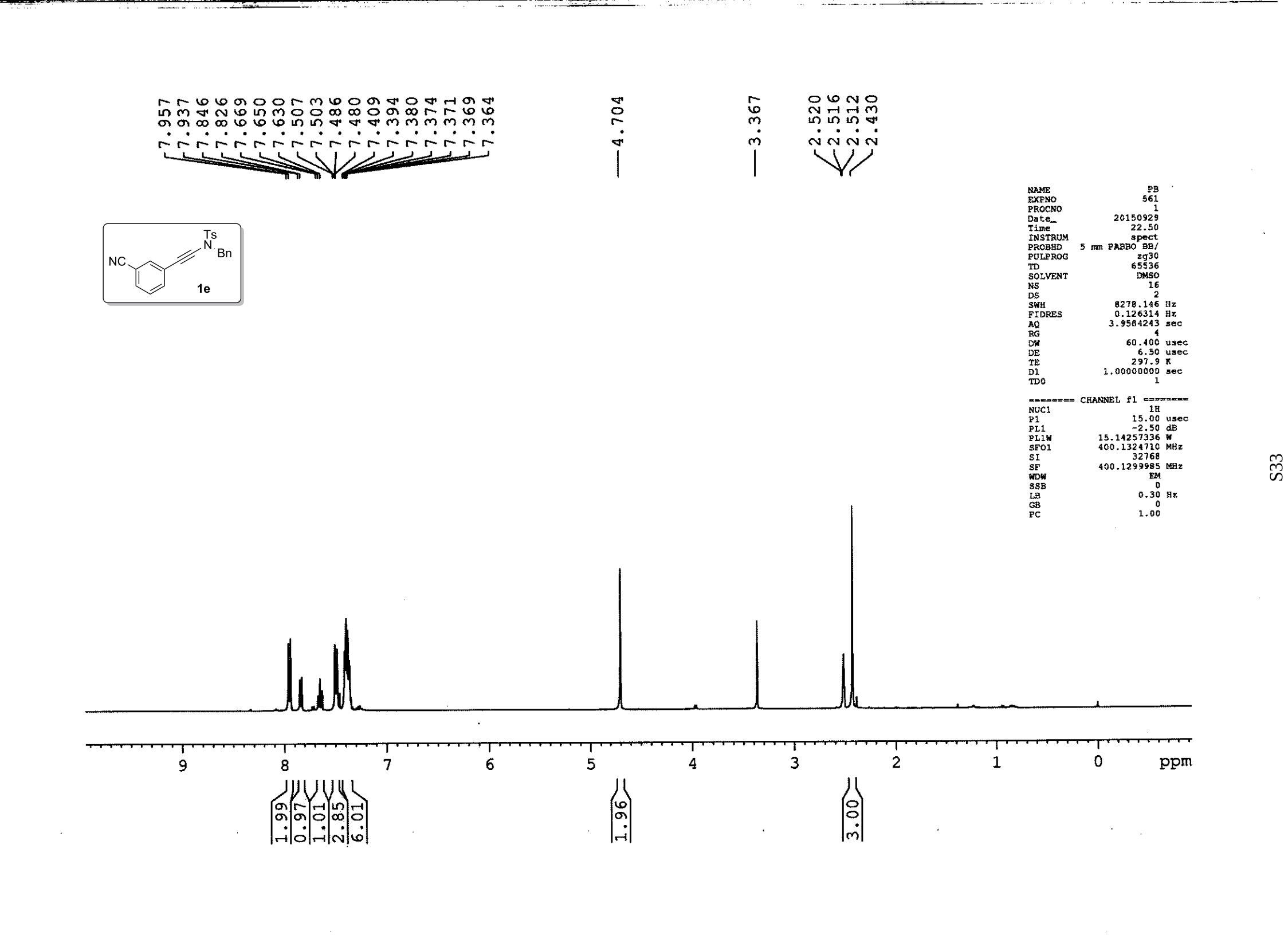
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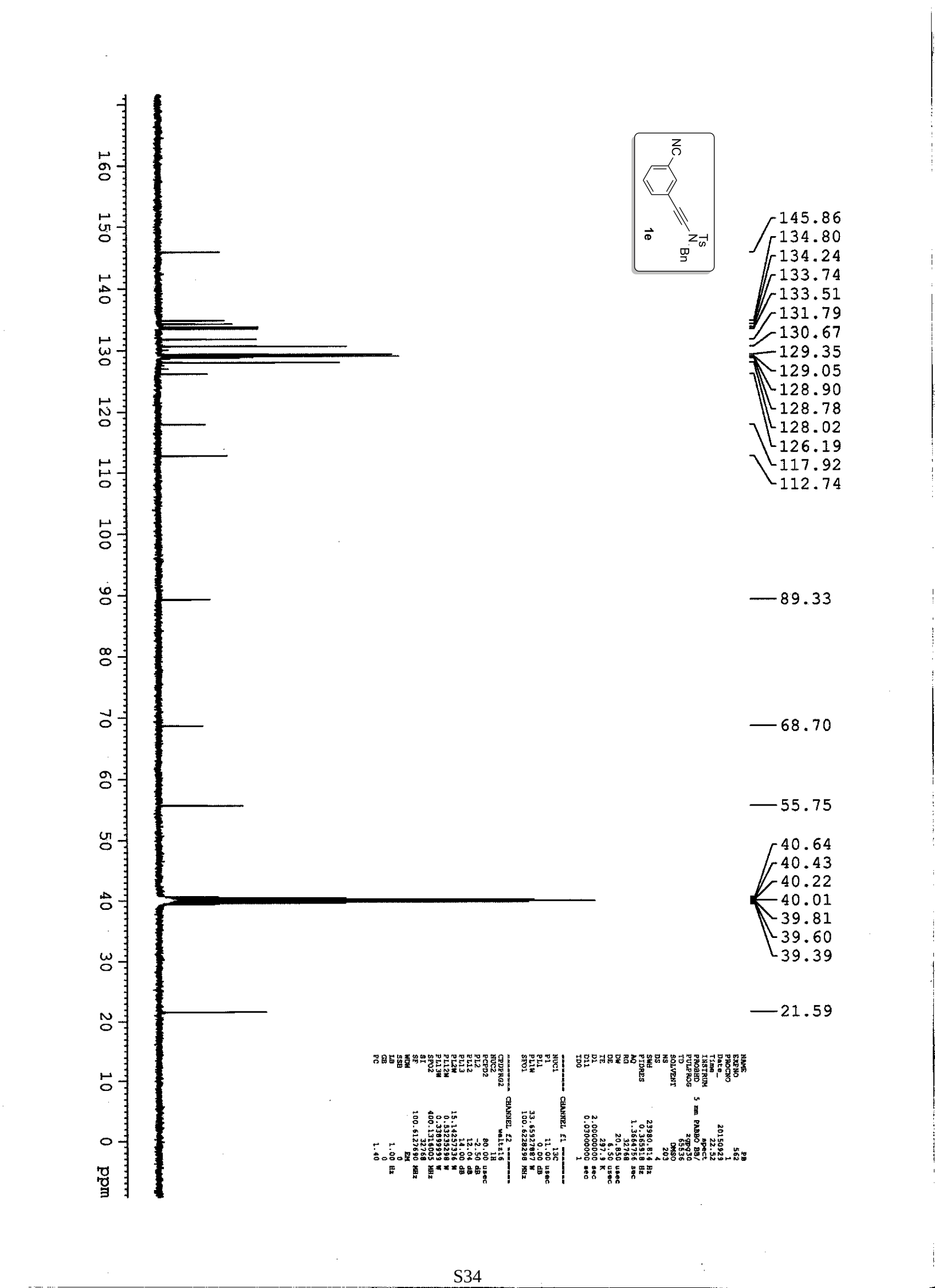
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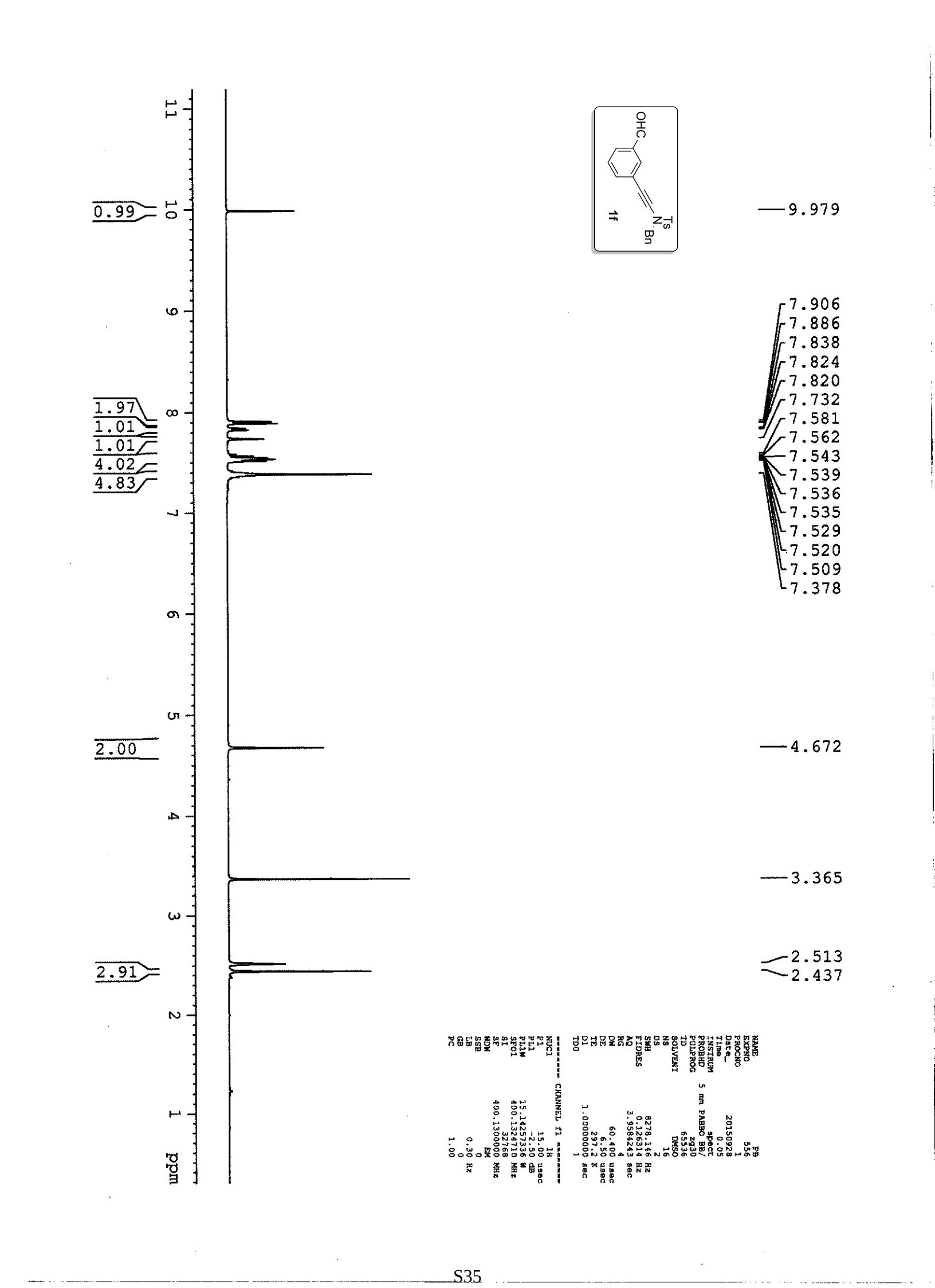
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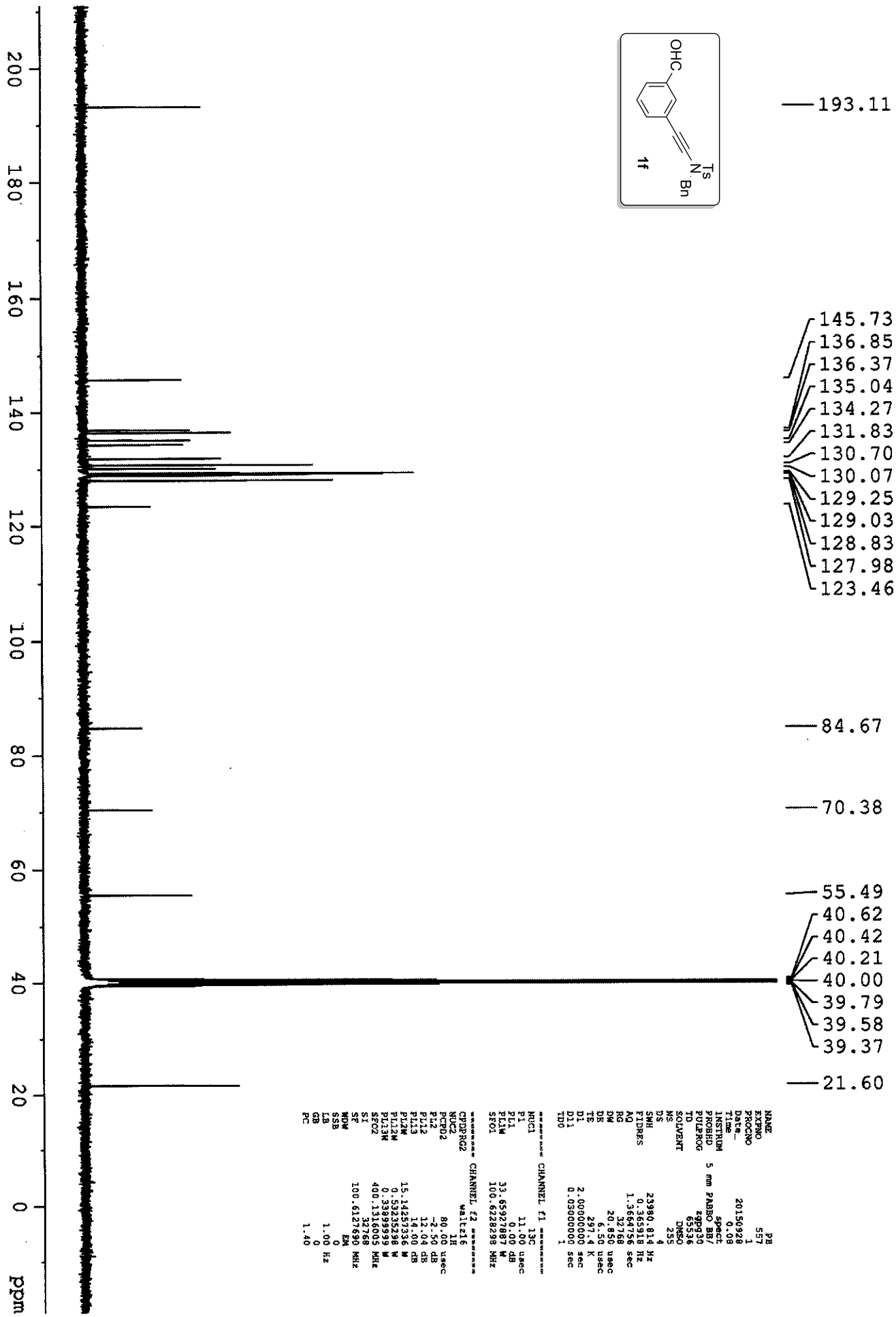
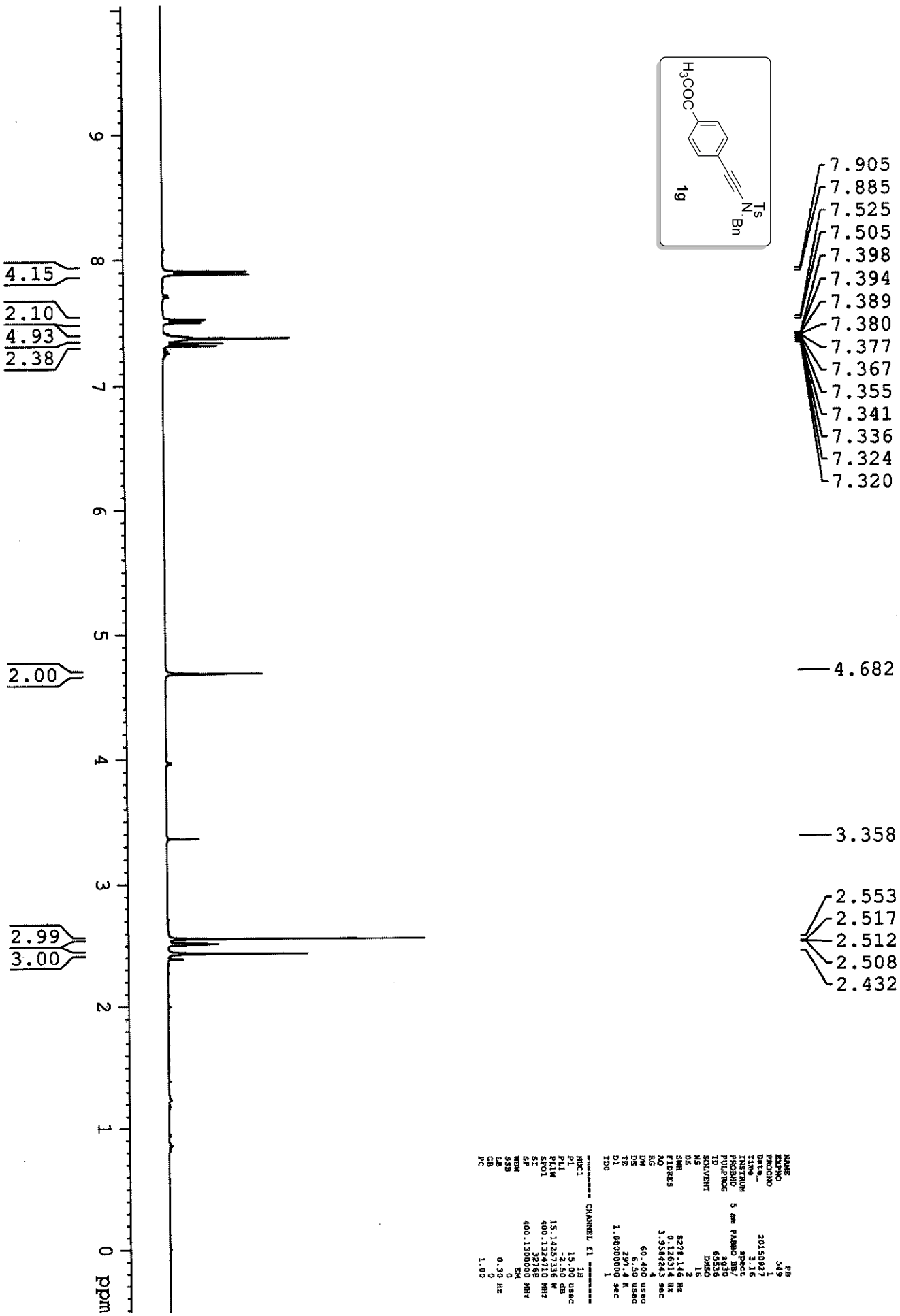
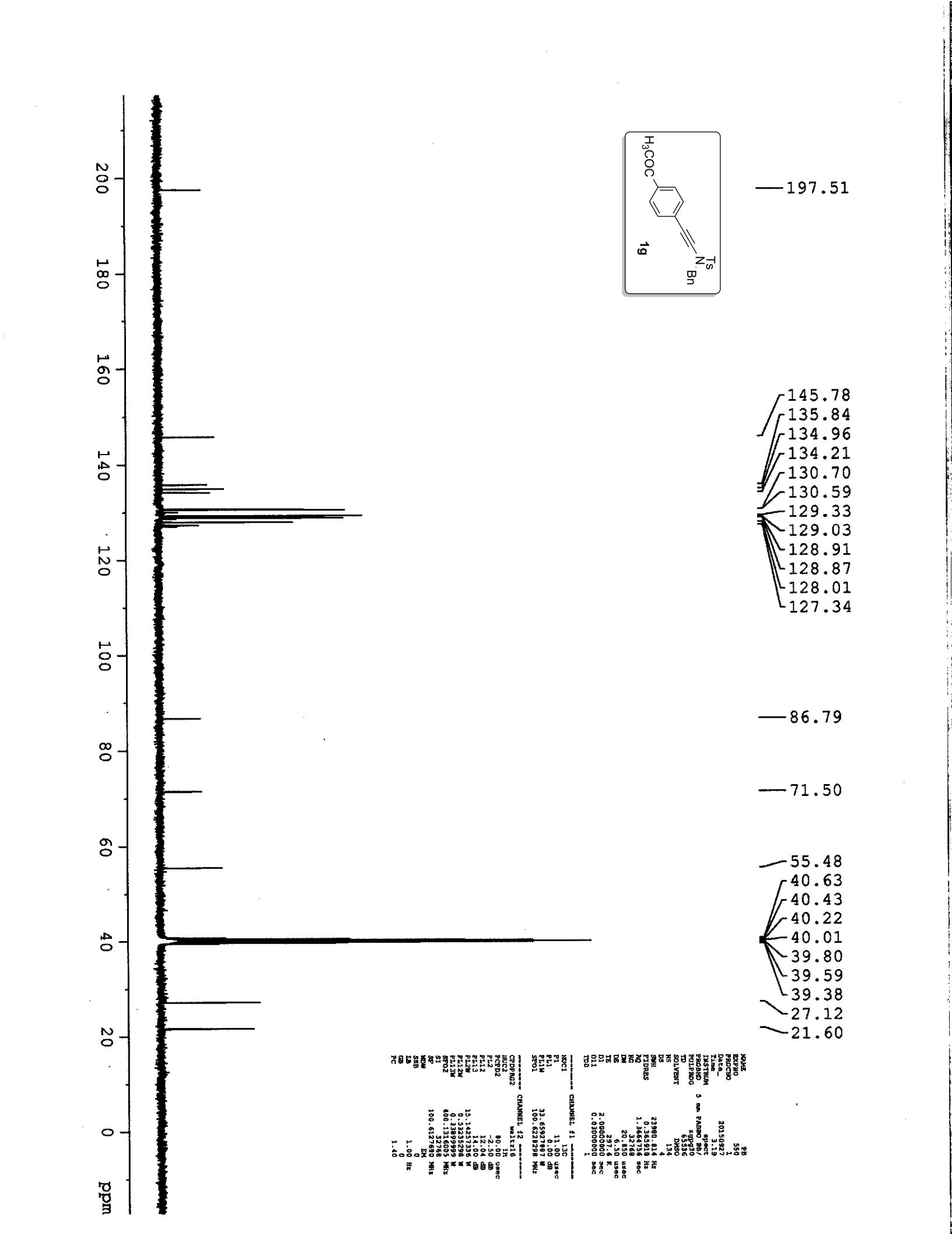
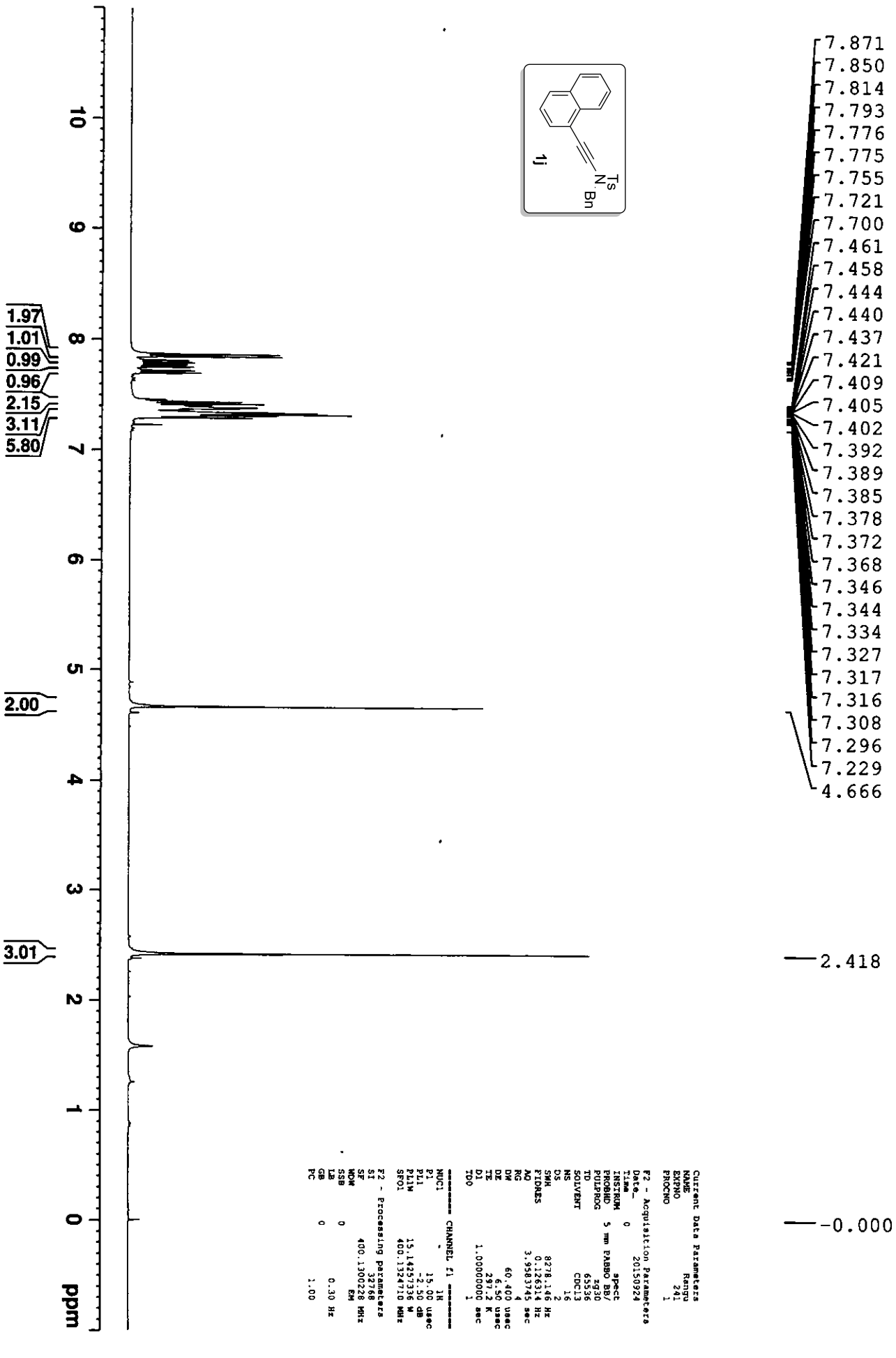
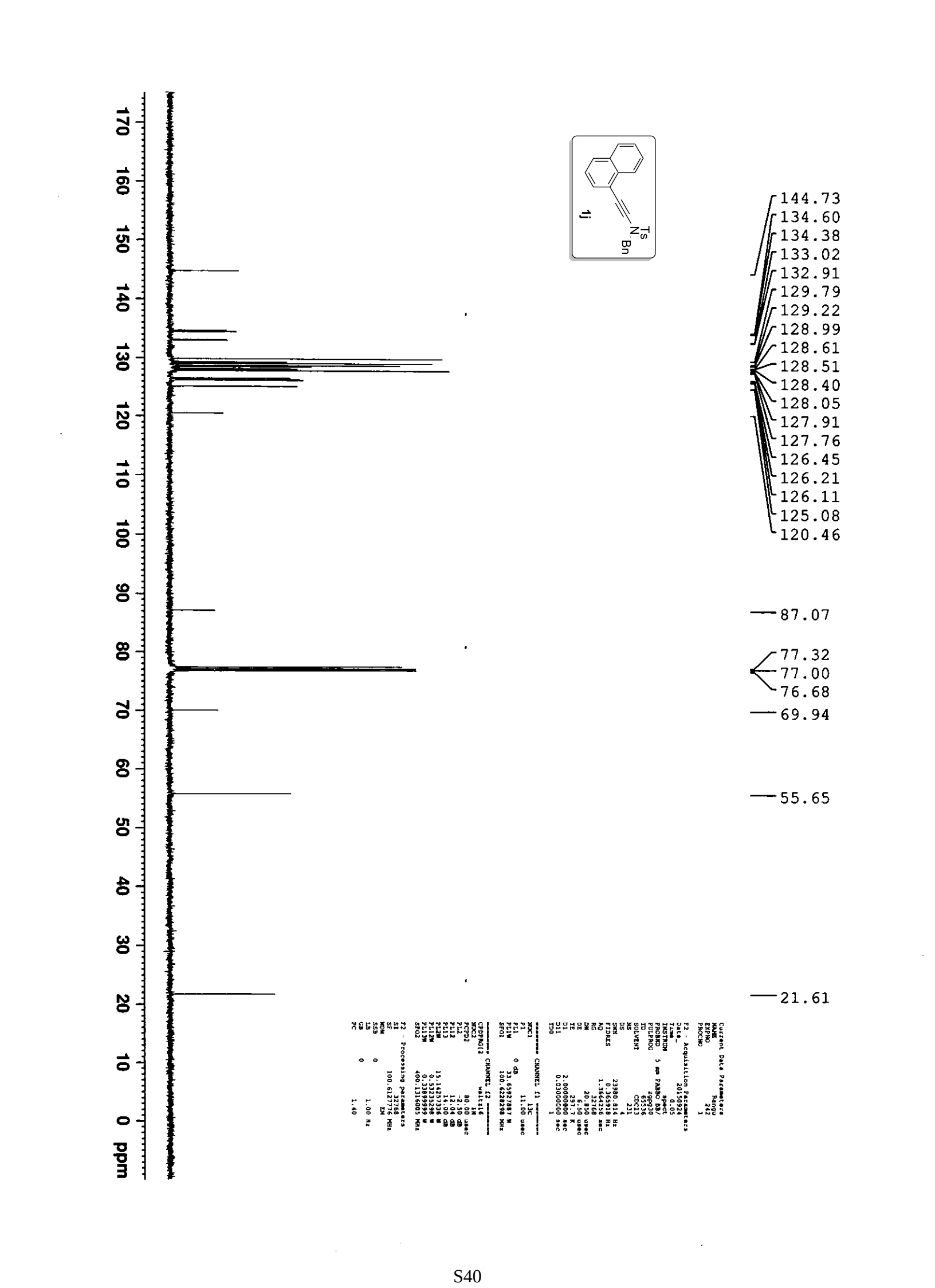
4.83
2.00
2.91
11
10
9
8
7
6
5
4
3
2
1
ppm
NAME PB
EXPNO 556
PROCNO 1
Date_ 20150928
Time 0.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

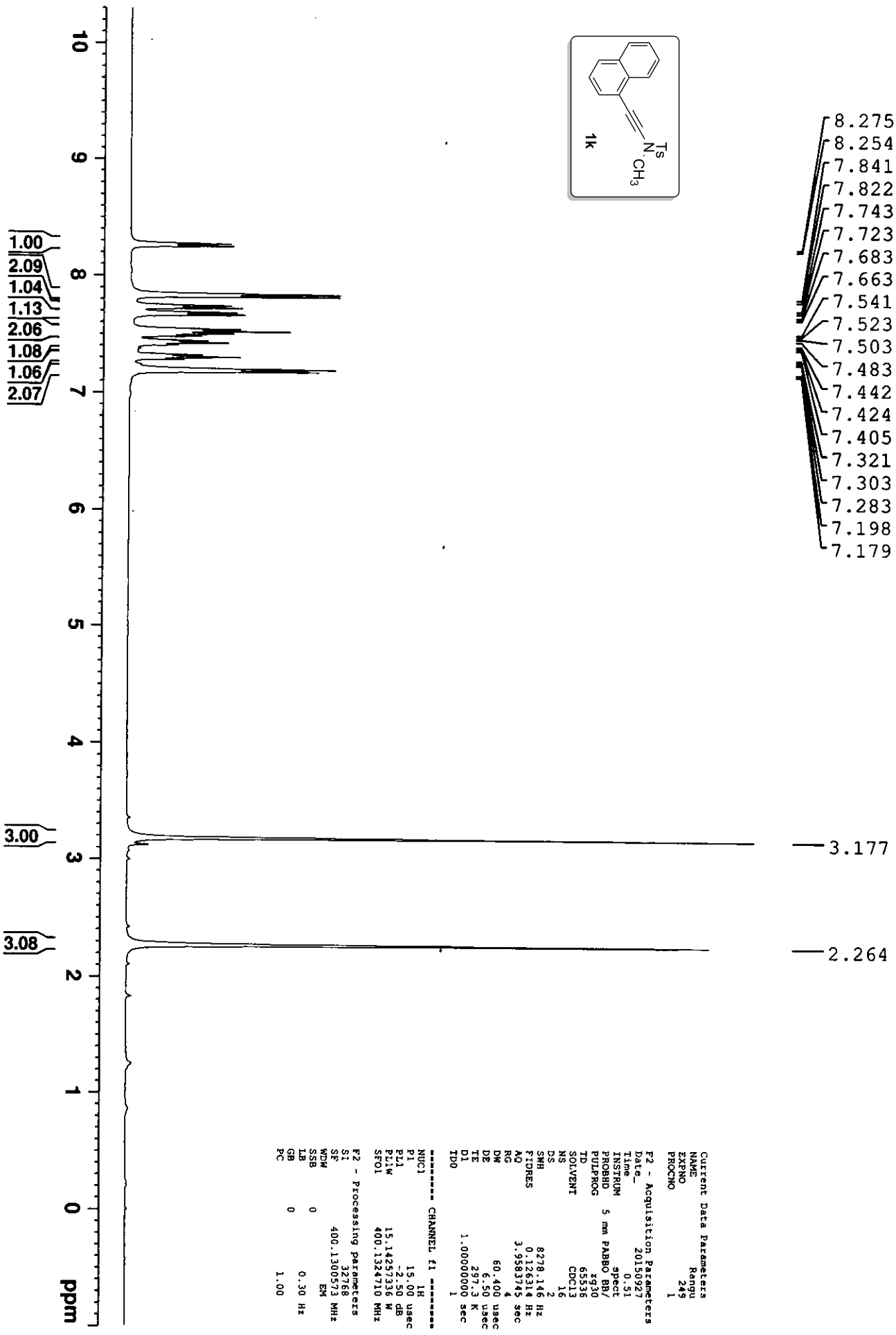

OHC
Ts
N
Bn
1f
193.11
145.73
136.85
136.37
135.04
134.27
131.83
130.70
130.07
129.25
129.03
128.83
127.98
123.46
84.67
70.38
55.49
40.62
40.42
40.21
40.00
39.79
39.58
39.37
21.60
200
180
160
140
120
100
80
60
40
20
0
ppm

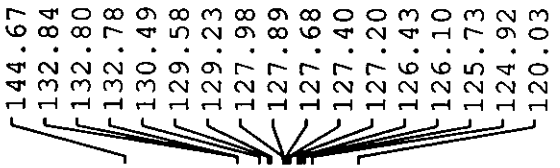

H3COC
Ts
N
Bn
1g
7.905
7.885
7.525
7.505
7.398
7.394
7.389
7.380
7.377
7.367
7.355
7.341
7.336
7.324
7.320
4.682
3.358
2.553
2.517
2.512
2.508
2.432
4.15
2.10
4.93
2.38
2.00
2.99
3.00
ppm

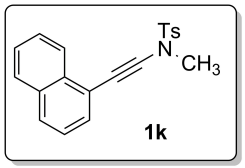

H3COC
Ts
N
Bn
1g
197.51
145.78
135.84
134.96
134.21
130.70
130.59
129.33
129.03
128.91
128.87
128.01
127.34
86.79
71.50
55.48
40.63
40.43
40.22
40.01
39.80
39.59
39.38
27.12
21.60
200
180
160
140
120
100
80
60
40
20
0
ppm


1j
Ts
N
Bn
7.871
7.850
7.814
7.793
7.776
7.775
7.755
7.721
7.700
7.461
7.458
7.444
7.440
7.437
7.421
7.409
7.405
7.402
7.392
7.389
7.385
7.378
7.372
7.368
7.346
7.344
7.334
7.327
7.317
7.316
7.308
7.296
7.229
4.666
2.418
-0.000
1.97
1.01
0.99
0.96
2.15
3.11
5.80
2.00
3.01
ppm
Current Data Parameters
NAME Rangu
EXPNO 241
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150924
Time 0
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300228 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

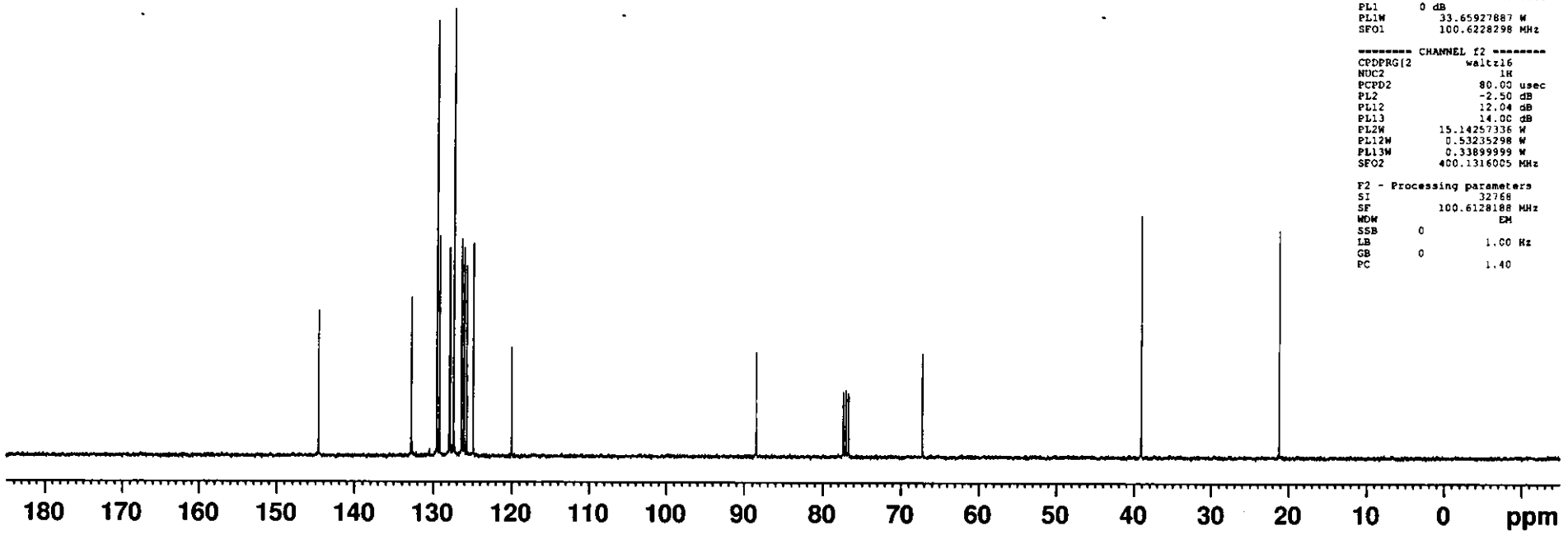

1j
Ts
N
Bn
144.73
134.60
134.38
133.02
132.91
129.79
129.22
128.99
128.61
128.51
128.40
128.05
127.91
127.76
126.45
126.21
126.11
125.08
120.46
87.07
77.32
77.00
76.68
69.94
55.65
21.61
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

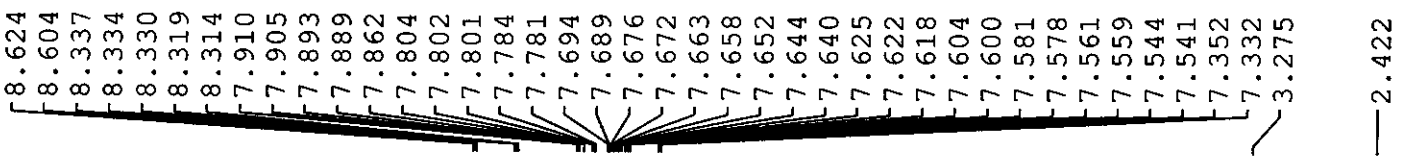

1k
Ts
N
CH3
8.275
8.254
7.841
7.822
7.743
7.723
7.683
7.663
7.541
7.523
7.503
7.483
7.442
7.424
7.405
7.321
7.303
7.283
7.198
7.179
3.177
2.264
1.00
2.09
1.04
1.13
2.06
1.08
1.06
2.07
3.00
3.08
10
9
8
7
6
5
4
3
2
1
0
ppm
Current Data Parameters
NAME Rangu
EXPNO 249
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150927
Time 0.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300573 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

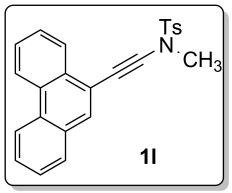





```
Current Data Parameters
NAME                  Rangu
EXPNO                   250
PROCNO                    1

F2 - Acquisition Parameters
Date_              20150927
Time                   0.55
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      53
DS                       4
SWH              23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664256 sec
RG                   32768
DW                  20.850 usec
DE                   6.50 usec
TE                  297.6 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   11.00 usec
PL1        0 dB
PL1W        33.65927887 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.50 dB
PL12                 12.04 dB
PL13                 14.00 dB
PL2W        15.14257336 W
PL12W        0.53235298 W
PL13W        0.33899999 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF           100.6128188 MHz
WDW                      EM
SSB        0
LB                    1.00 Hz
GB         0
PC                    1.40
```





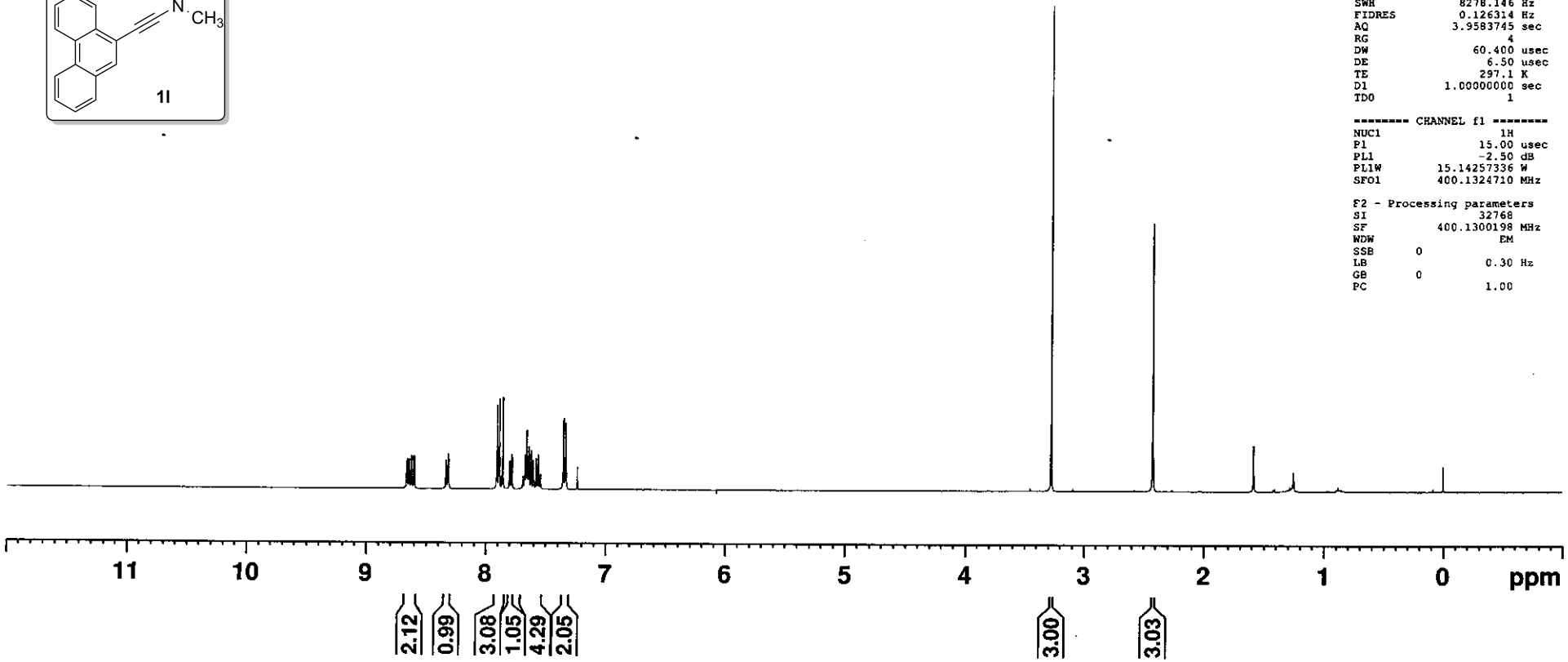


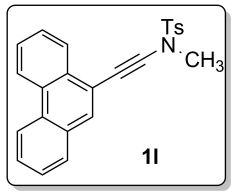
Current Data Parameters
NAME RKM
EXPNO 49
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150926
Time 23.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1

--------- CHANNEL f1 ---------
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300198 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



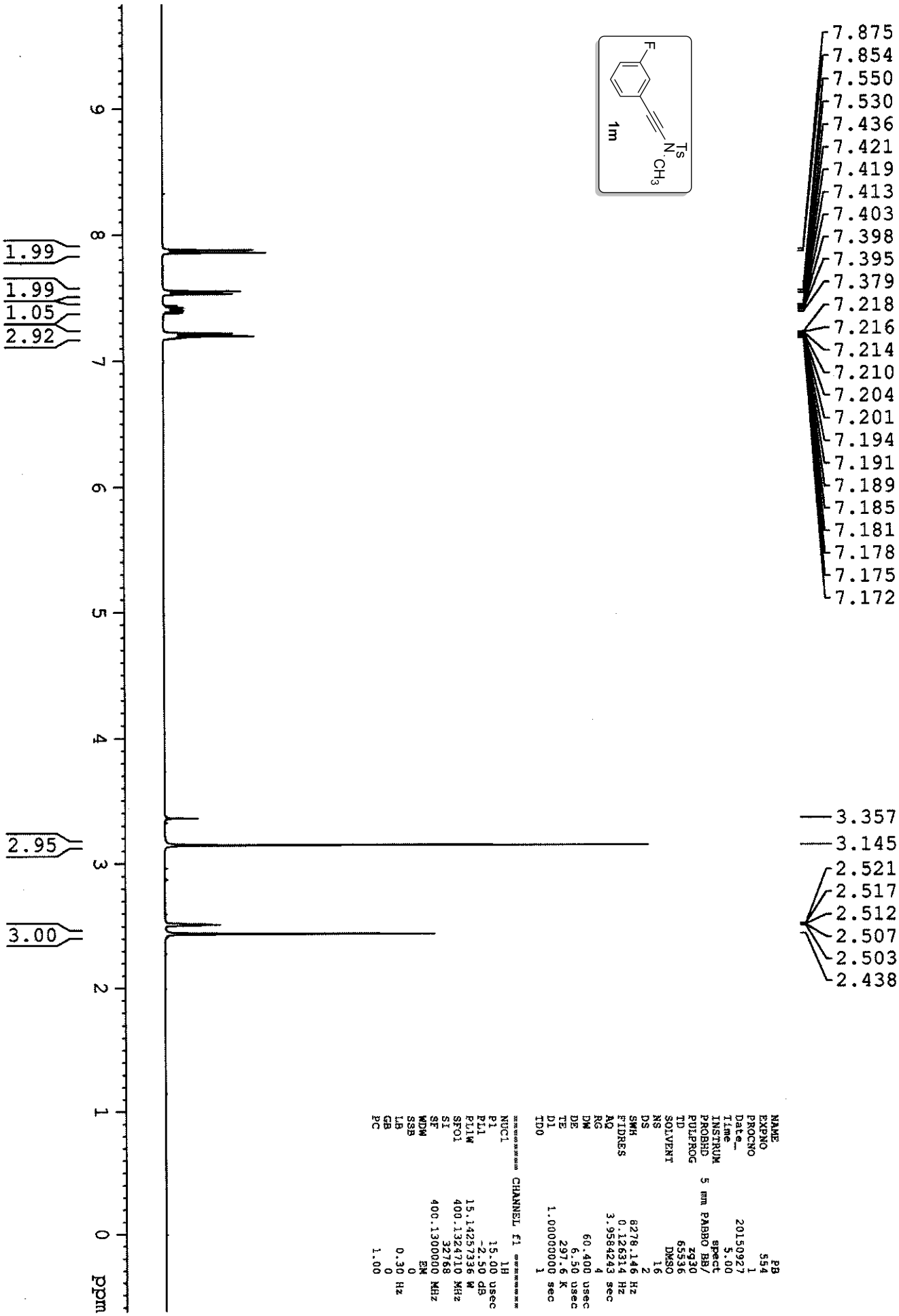


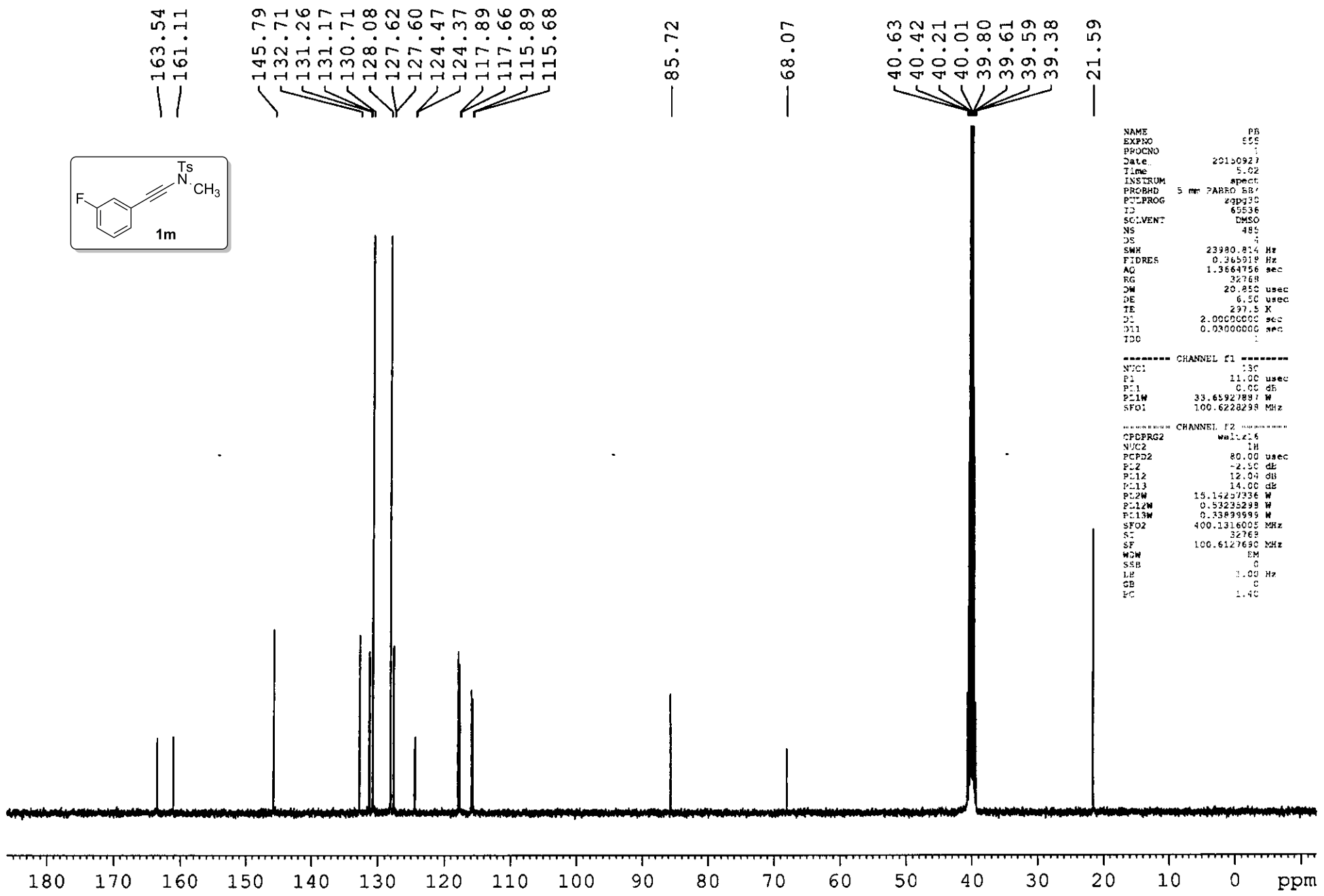
1l

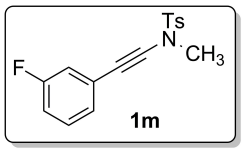
144.89
133.29
131.20
131.09
130.71
130.02
129.94
129.89
128.26
127.84
127.17
127.03
126.99
126.90
126.84
122.69
122.55
119.11
88.17
77.32
77.00
76.69
67.73
39.37
21.62

```
Current Data Parameters
NAME                  ROH
EXPNO                  50
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150926
Time                23.44
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    104
DS                      4
SWH           23980.814 Hz
FIDRES            0.365918 Hz
AQ            1.3664256 sec
RG                  32768
DW                 20.850 usec
DE                  6.50 usec
TE                297.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.00 usec
PL1        0 dB
PL1W       33.65927887 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 2.50 dB
PL12               12.04 dB
PL13               14.00 dB
PL2W       15.14257336 W
PL12W       0.53235298 W
PL13W       0.33899999 W
SFO2       400.7316005 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127762 MHz
WDW                    EM
SSB        0
LB                  1.00 Hz
GB         0
PC                  1.40
```

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


1m
7.875
7.854
7.550
7.530
7.436
7.421
7.419
7.413
7.403
7.398
7.395
7.379
7.218
7.216
7.214
7.210
7.204
7.201
7.194
7.191
7.189
7.185
7.181
7.178
7.175
7.172
3.357
3.145
2.521
2.517
2.512
2.507
2.503
2.438
1.99
1.99
1.05
2.92
2.95
3.00
ppm
NAME PB
EXPNO 554
PROCNO 1
Date_ 20150927
Time_ 5.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


163.54
161.11
145.79
132.71
131.26
131.17
130.71
128.08
127.62
127.60
124.47
124.37
117.89
117.66
115.89
115.68
85.72
68.07
40.63
40.42
40.21
40.01
39.80
39.61
39.59
39.38
21.59
Ts
N
CH3
F
1m
NAME PB
EXPNO 555
PROCNO 1
Date_ 20150927
Time 5.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 485
DS 4
SWH 23980.814 Hz
FIDRES 0.365919 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
PL1W 33.65927887 W
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.50 dB
PL12 12.04 dB
PL13 14.00 dB
PL2W 15.14257336 W
PL12W 0.53236298 W
PL13W 0.33899999 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



-112.62

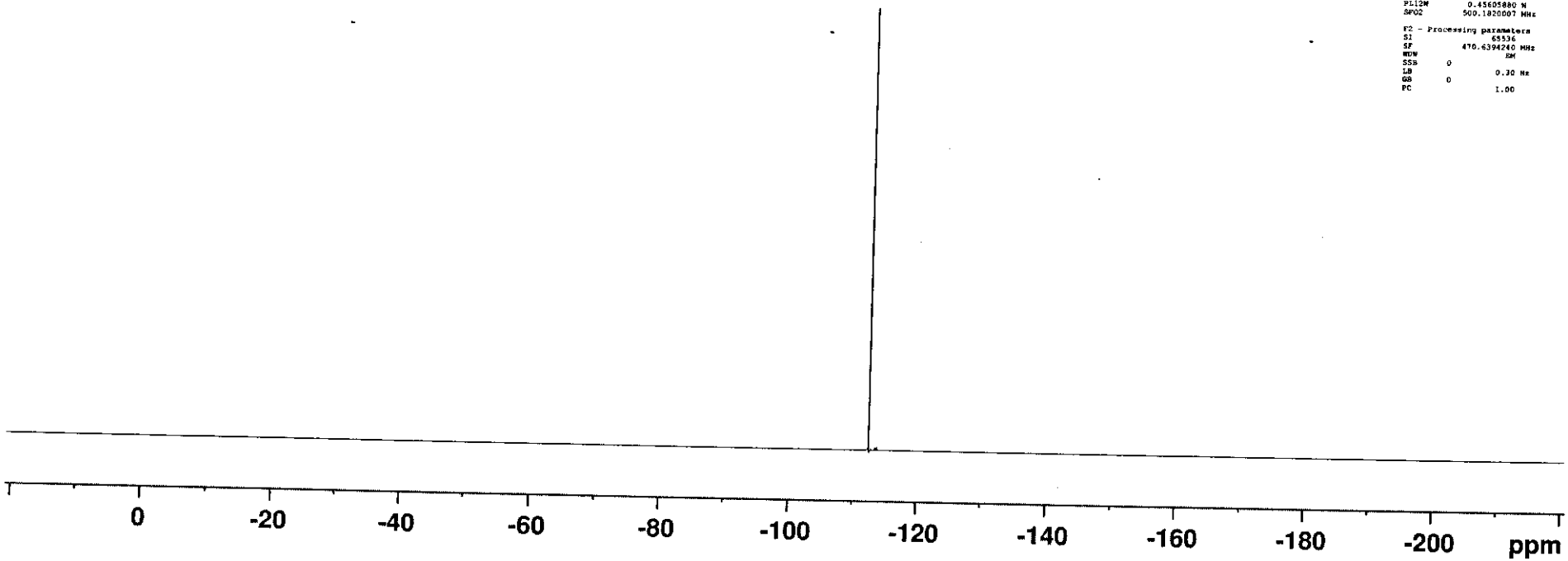
Current Data Parameters
NAME XF
EXPNO 67
PROCNO 1

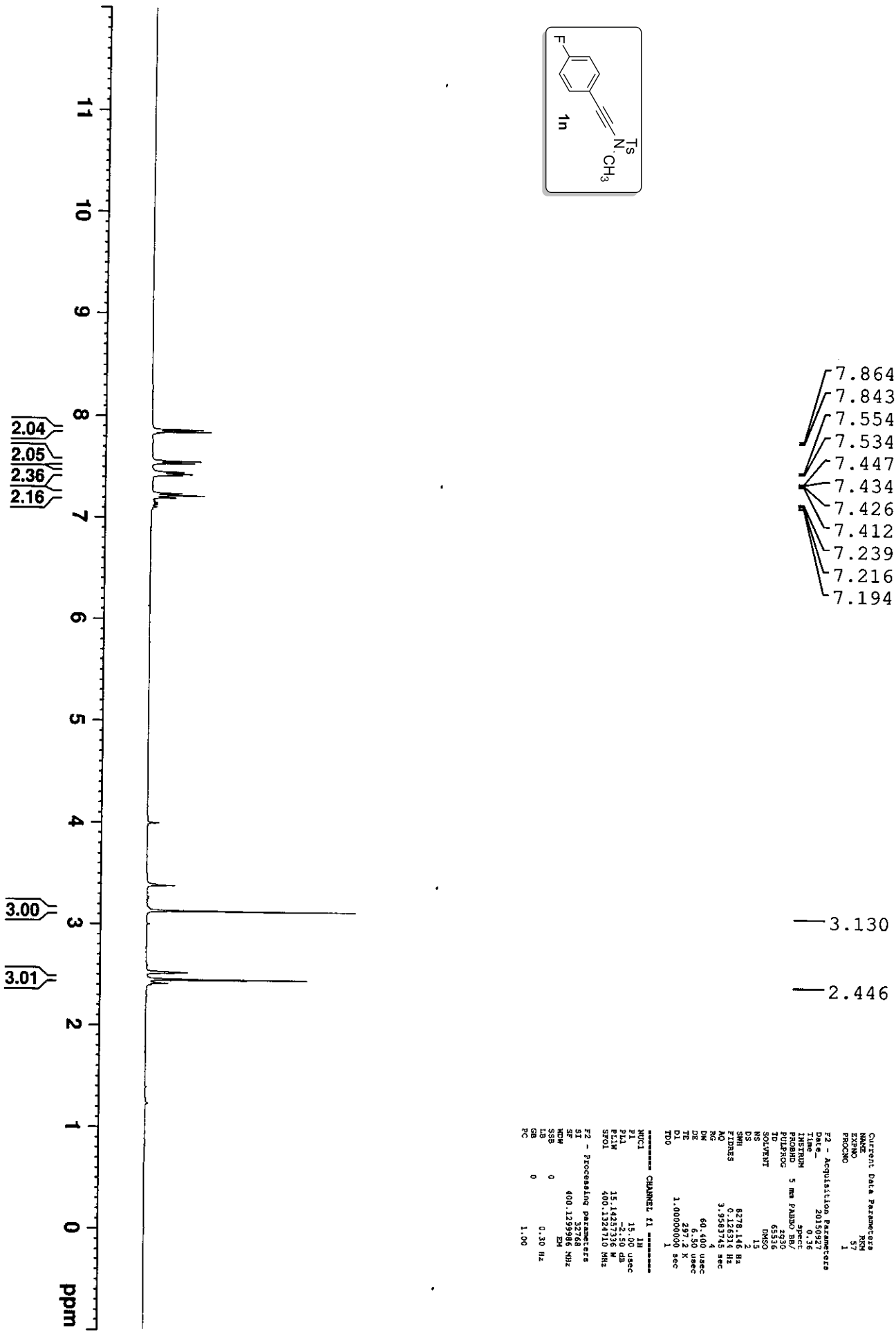
F2 - Acquisition Parameters
Date_ 20150929
Time 2.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgfhigqn
TD 131072
SOLVENT DMSO
NS 16
DS 4
SWH 113636.367 Hz
FIDRES 0.866977 Hz
AQ 0.5767168 sec
RG 2050
DW 4.400 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 19F
P1 14.50 usec
PL1 -0.50 dB
PL1W 21.58881378 W
SFO1 470.5923603 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.50 dB
PL2W 12.85348415 W
PL12W 0.45605880 W
SFO2 500.1820007 MHz

F2 - Processing parameters
SI 65536
SF 470.6394240 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

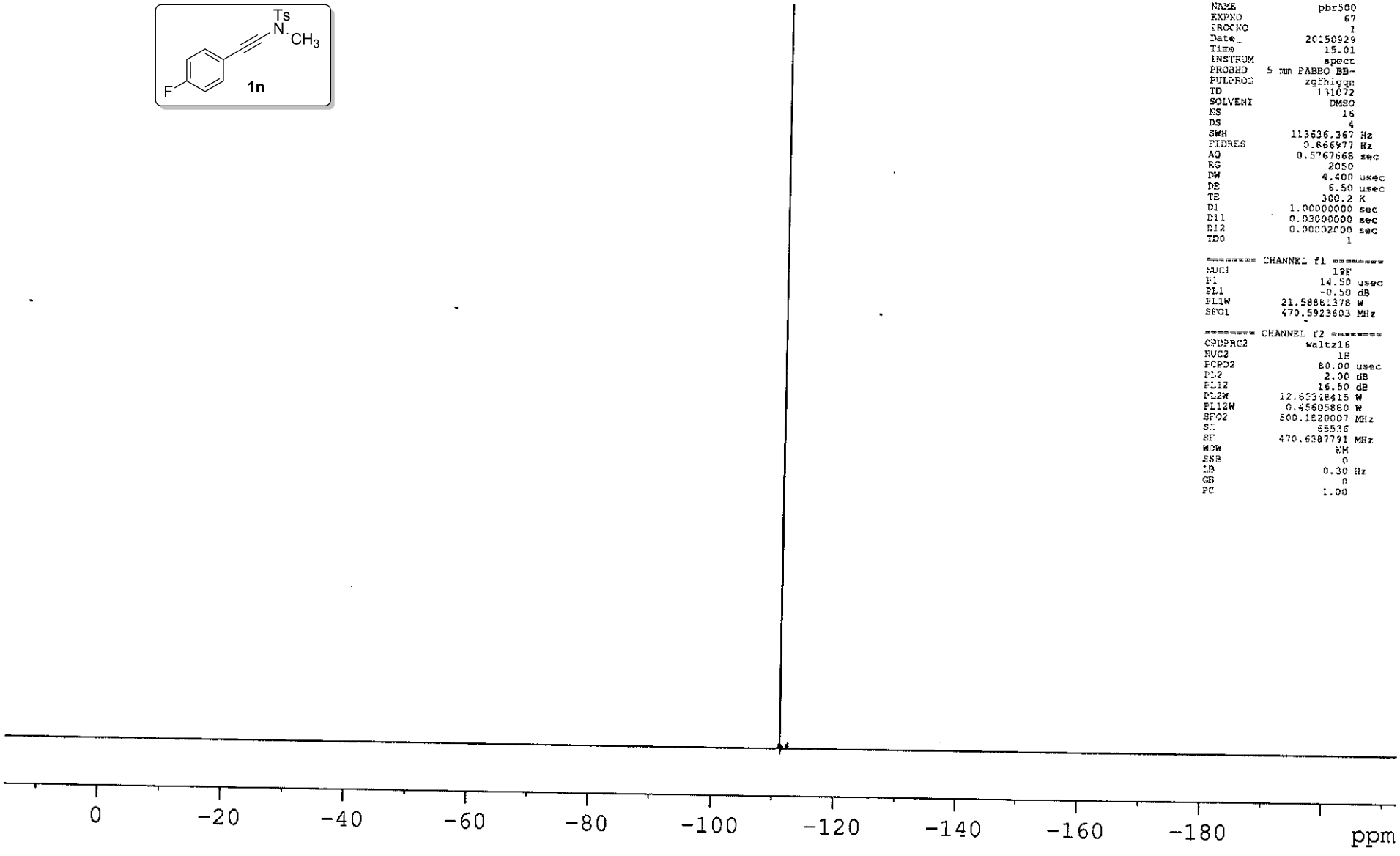


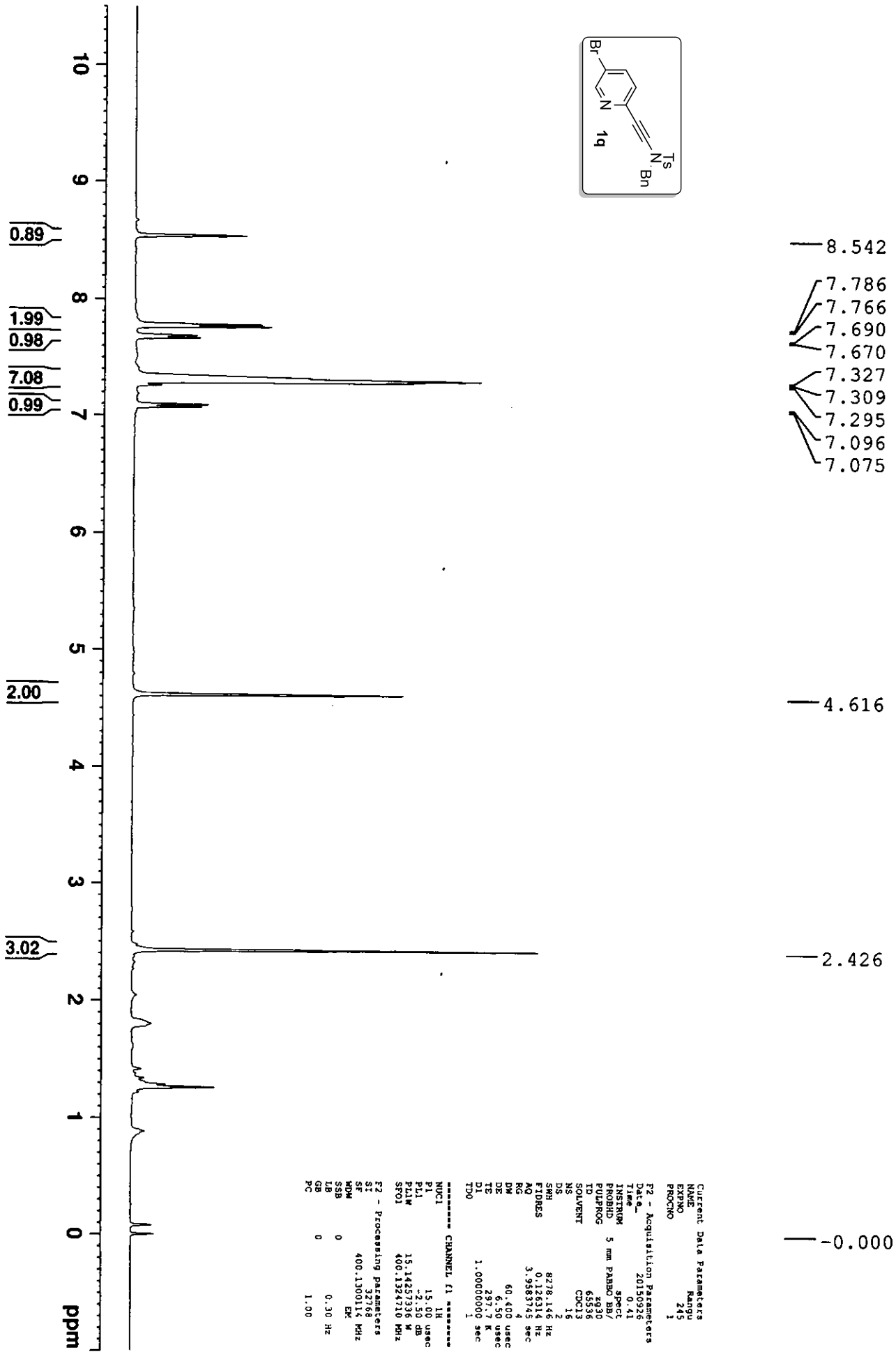

F
Ts
N
CH3
1n
7.864
7.843
7.554
7.534
7.447
7.434
7.426
7.412
7.239
7.216
7.194
3.130
2.446
2.04
2.05
2.36
2.16
3.00
3.01
ppm
Current Data Parameters
NAME RKM
EXPNO 57
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150927
Time 0.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126214 Hz
AQ 3.9583745 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1299986 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

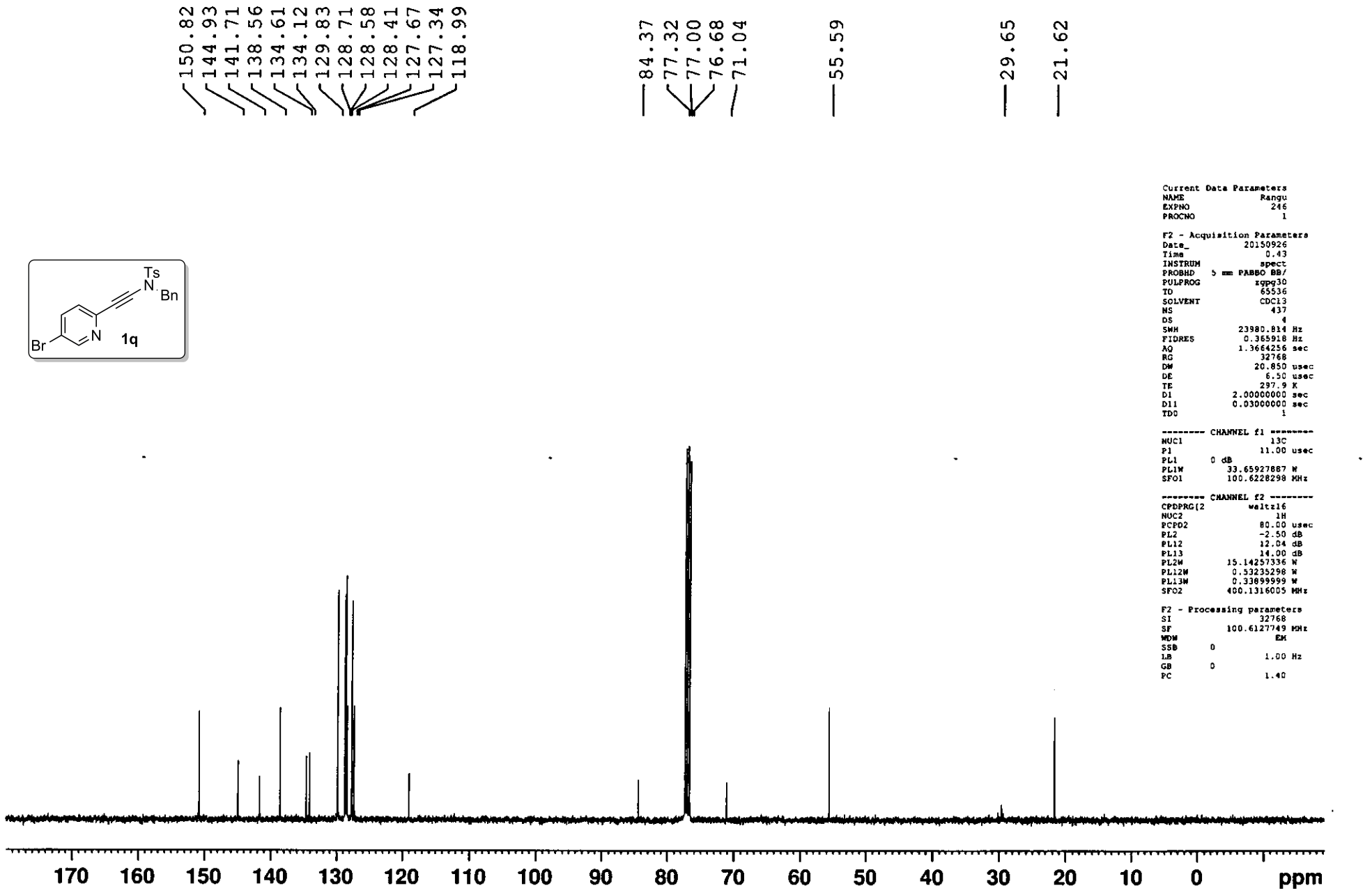
F
Ts
N
CH3
1n
163.44
160.99
145.70
133.99
133.90
132.73
130.67
128.05
118.72
118.69
116.48
116.26
84.32
67.85
40.63
40.42
40.21
40.00
39.79
39.67
39.58
39.38
21.59
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

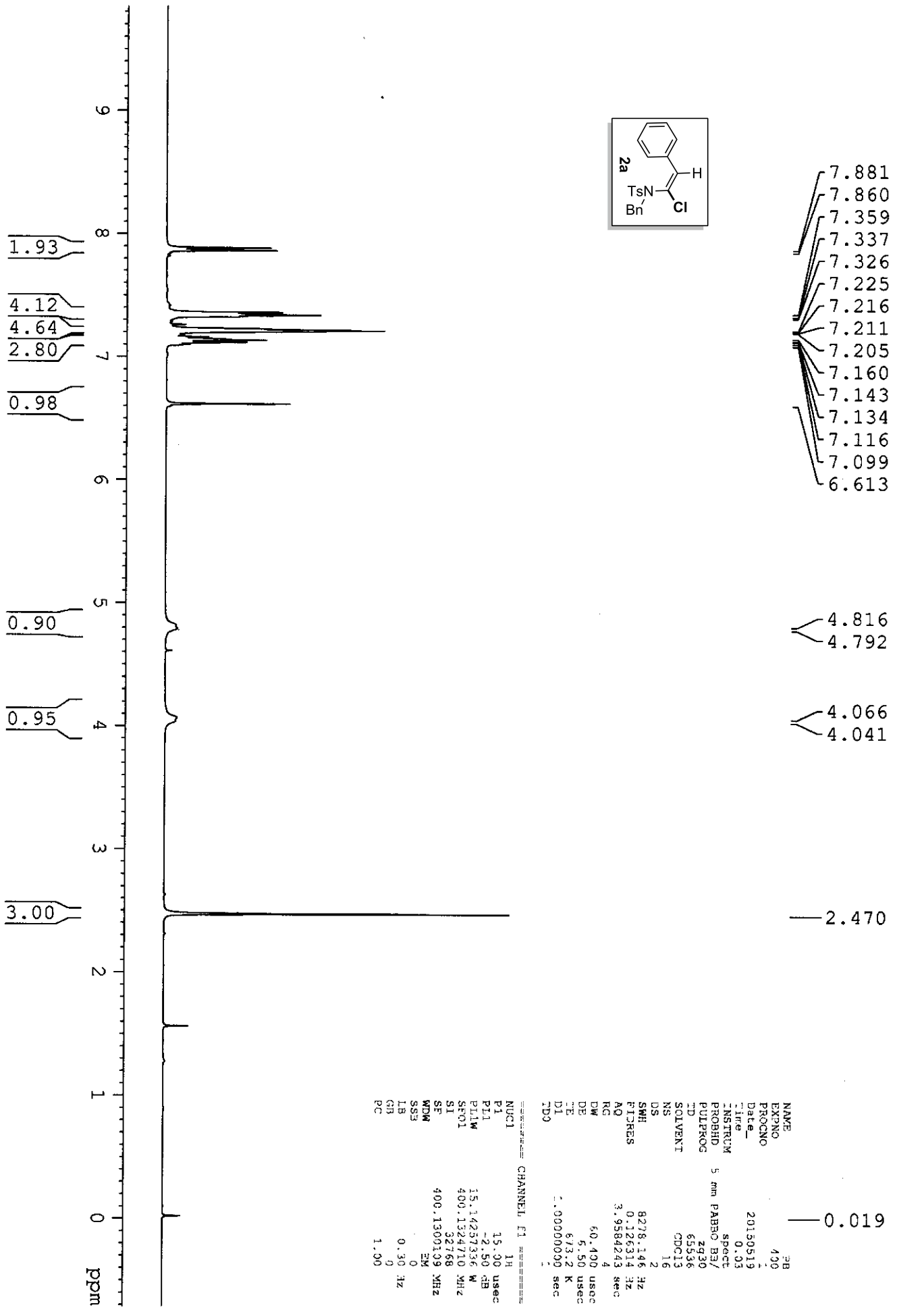
-111.25

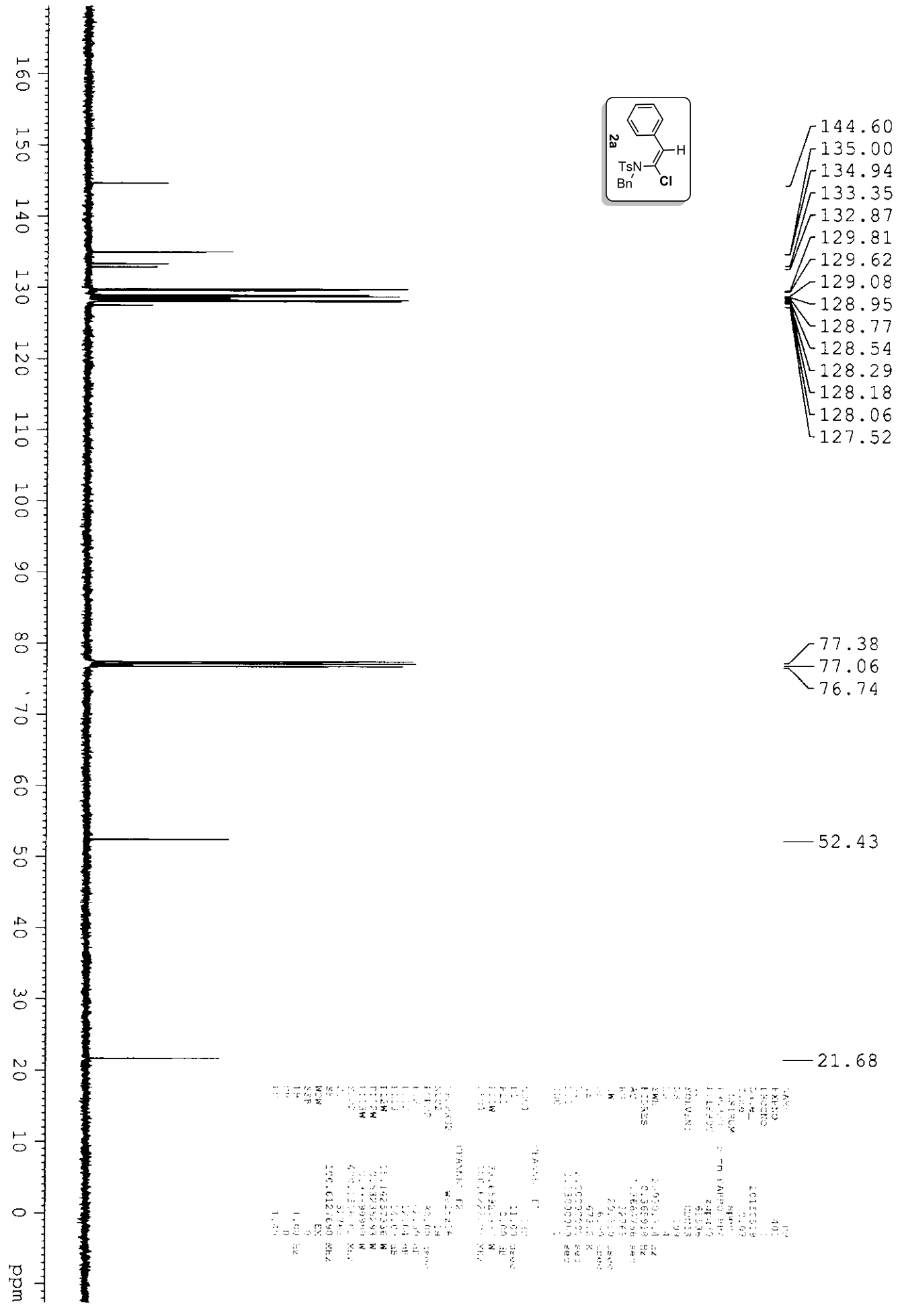
NAME	pbr500
EXPNO	67
PROCNO	1
Date_	20150929
Time	15.01
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgfhigqn
TD	131072
SOLVENT	DMSO
NS	16
DS	4
SWH	113636.367 Hz
FIDRES	0.866977 Hz
AQ	0.5767668 sec
RG	2050
DW	4.400 usec
DE	6.50 usec
TE	300.2 K
D1	1.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	19F
P1	14.50 usec
PL1	-0.50 dB
PL1W	21.58861378 W
SFO1	470.5923603 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.50 dB
PL2W	12.85348415 W
PL12W	0.45605880 W
SFO2	500.1820007 MHz
SI	65536
SF	470.6387791 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

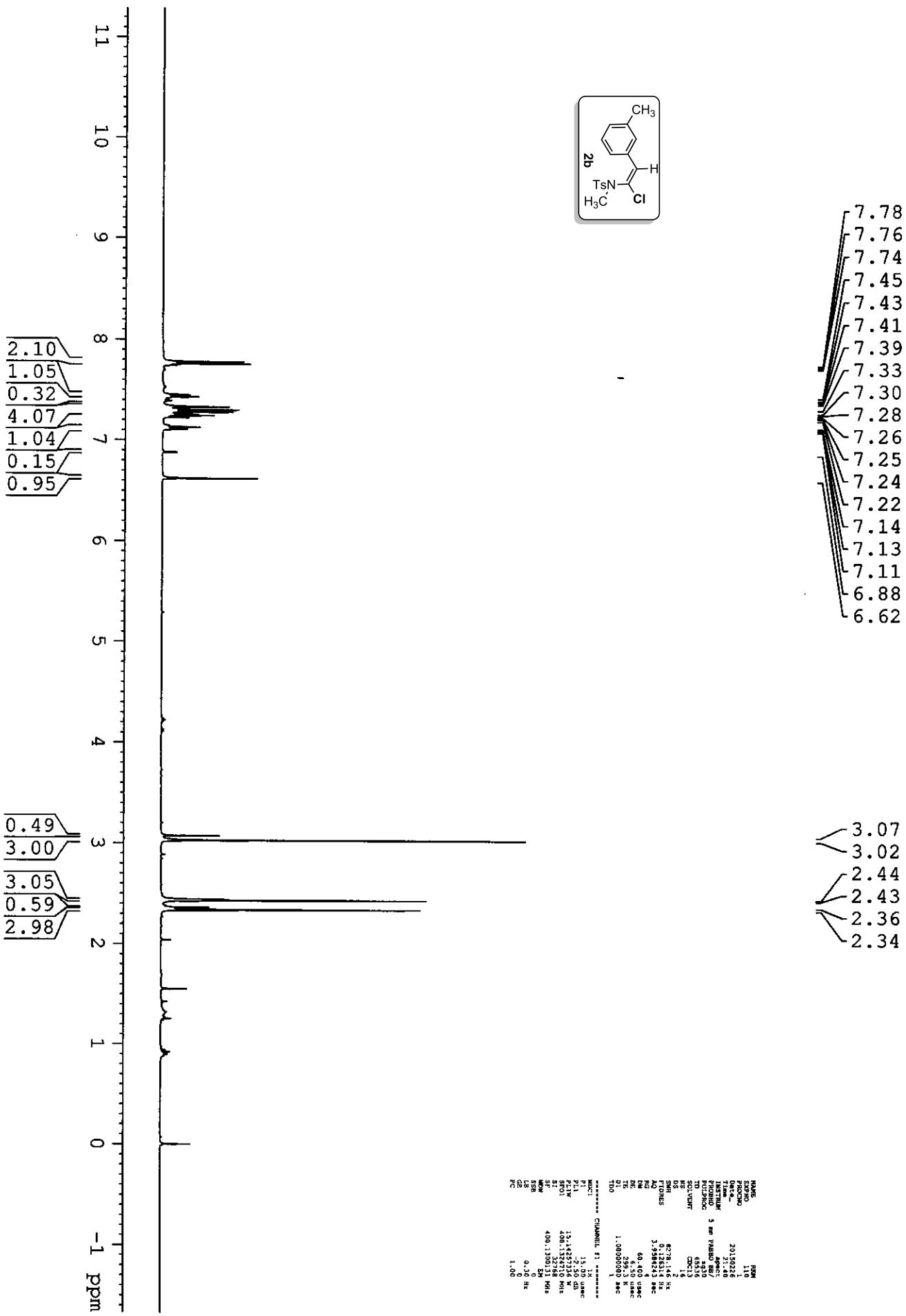


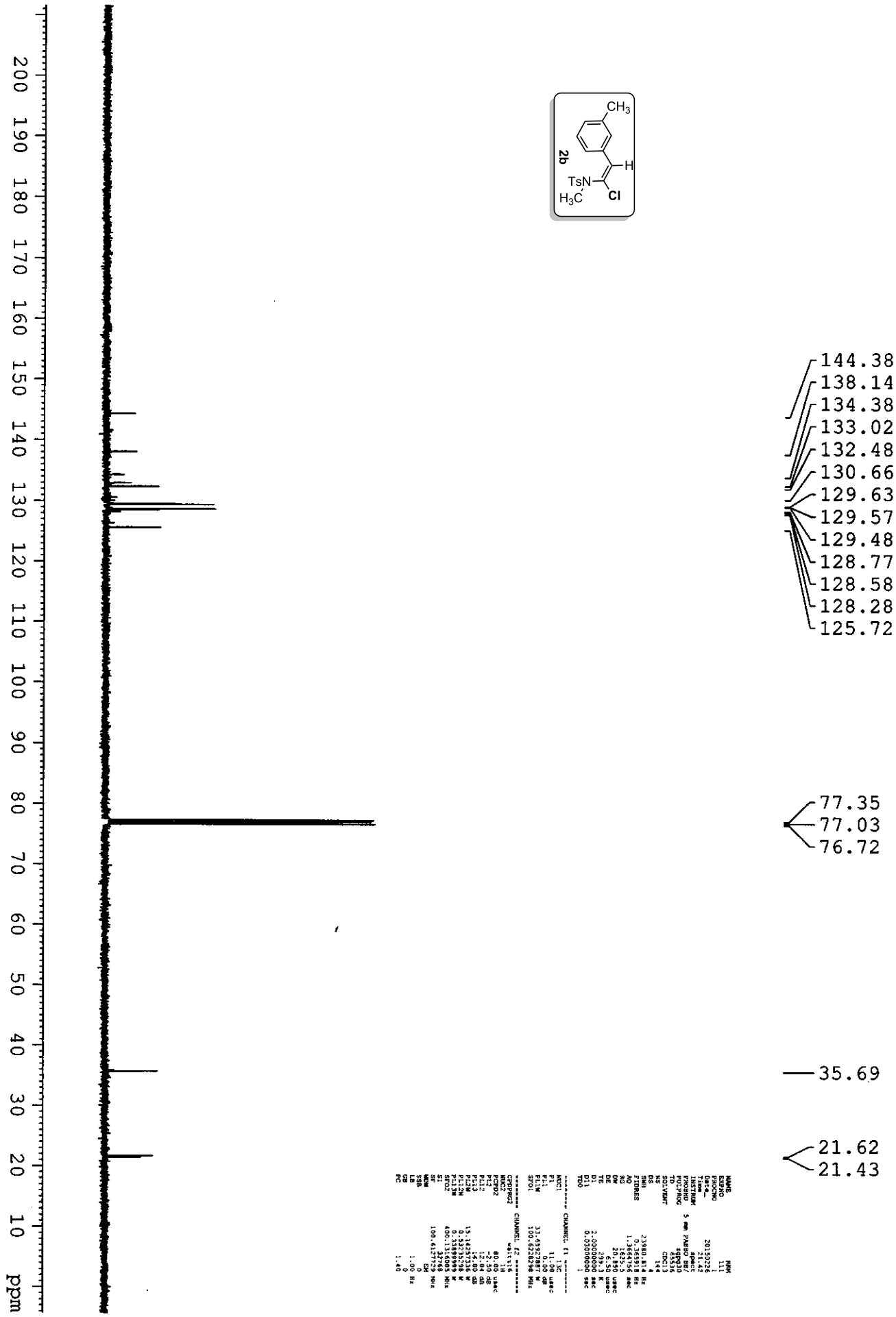

Br
N
1q
Ts
N
Bn
8.542
7.786
7.766
7.690
7.670
7.327
7.309
7.295
7.096
7.075
4.616
2.426
-0.000
0.89
1.99
0.98
7.08
0.99
2.00
3.02
ppm
Current Data Parameters
NAME Rangu
EXPNO 245
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150926
Time 0.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PLIW 15.14257336 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

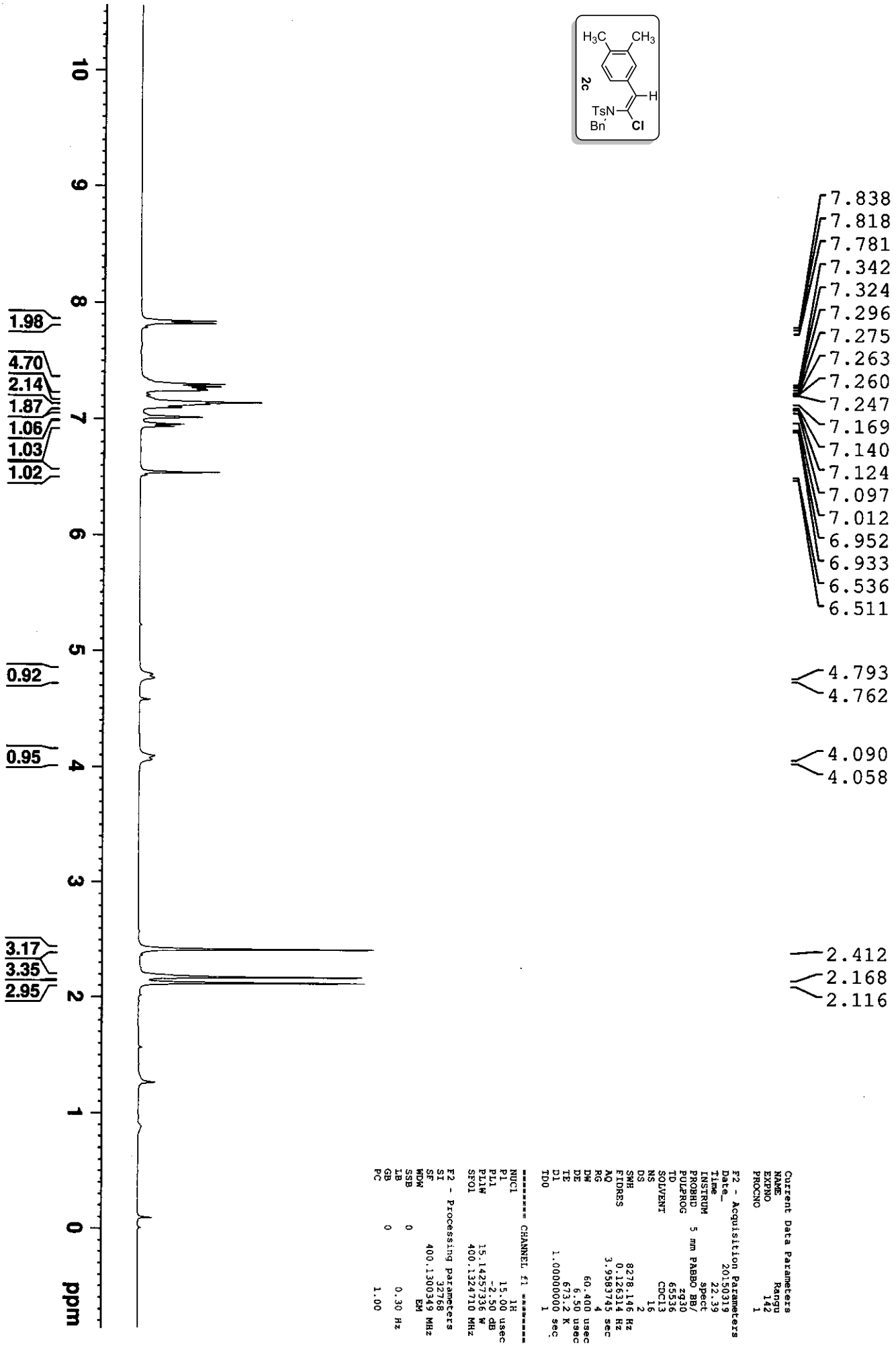

150.82
144.93
141.71
138.56
134.61
134.12
129.83
128.71
128.58
128.41
127.67
127.34
118.99
84.37
77.32
77.00
76.68
71.04
55.59
29.65
21.62
Ts
N
Bn
Br
N
1q
Current Data Parameters
NAME Rangu
EXPNO 246
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150926
Time 0.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 437
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 11.00 usec
PL1 0 dB
PL1W 33.65927887 W
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.50 dB
PL12 12.04 dB
PL13 14.00 dB
PL2W 15.14257336 W
PL12W 0.53235298 W
PL13W 0.33899999 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127749 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

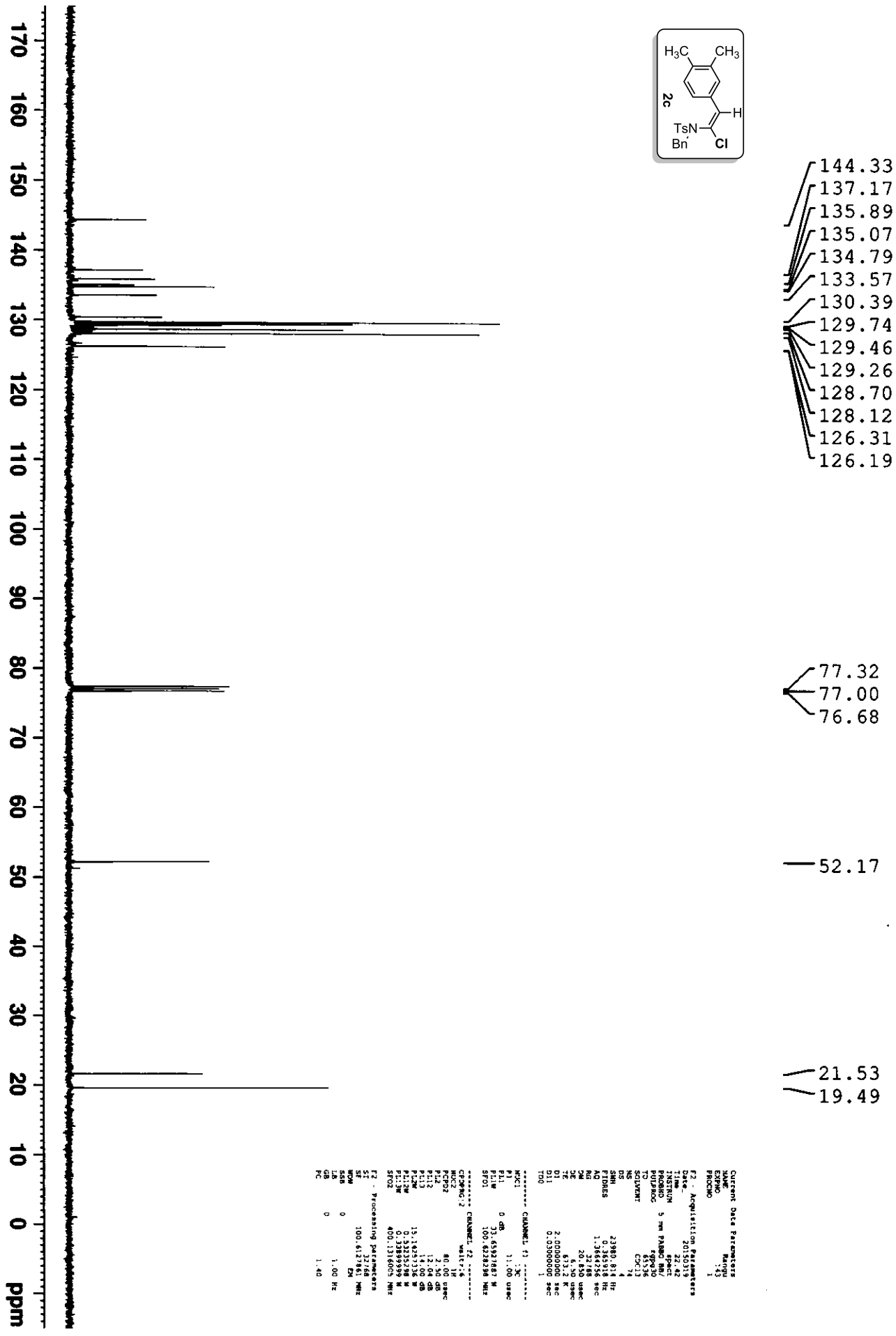

2a
TsN
Bn
H
Cl
7.881
7.860
7.359
7.337
7.326
7.225
7.216
7.211
7.205
7.160
7.143
7.134
7.116
7.099
6.613
4.816
4.792
4.066
4.041
2.470
0.019
1.93
4.12
4.64
2.80
0.98
0.90
0.95
3.00
ppm

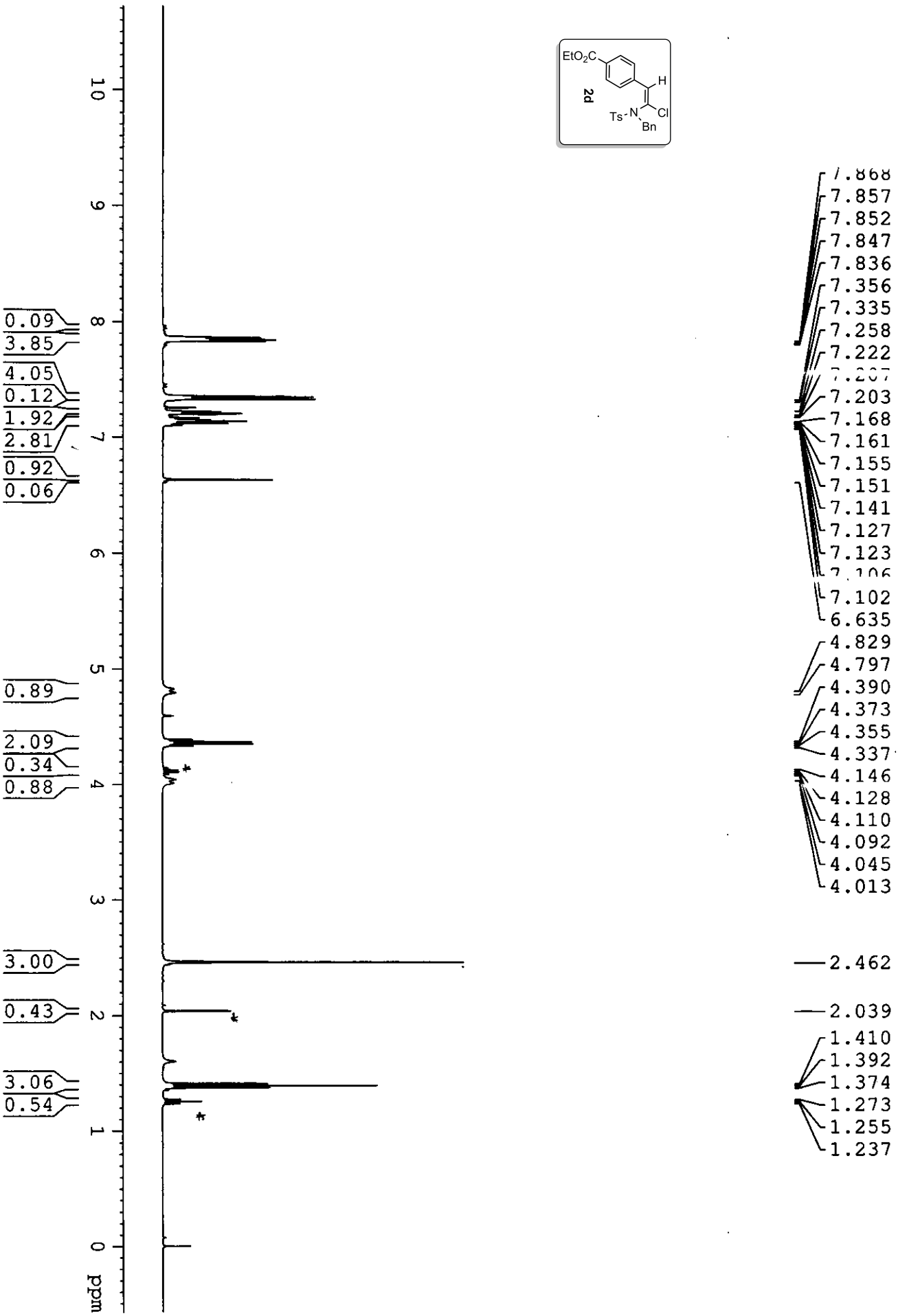

2a
TsN
Bn
H
Cl
144.60
135.00
134.94
133.35
132.87
129.81
129.62
129.08
128.95
128.77
128.54
128.29
128.18
128.06
127.52
77.38
77.06
76.74
52.43
21.68
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

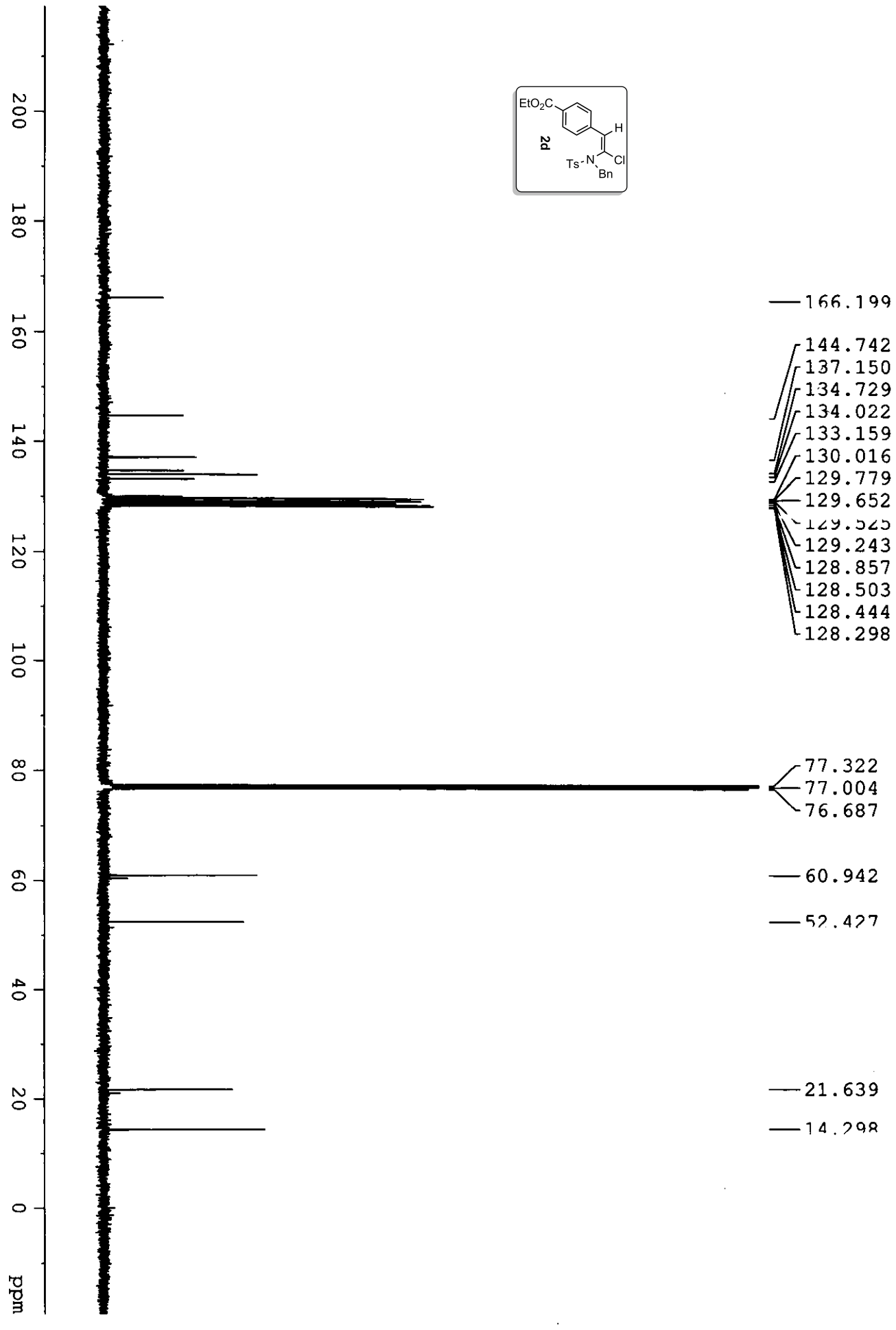

2b
CH3
H
TsN
H3C
Cl
7.78
7.76
7.74
7.45
7.43
7.41
7.39
7.33
7.30
7.28
7.26
7.25
7.24
7.22
7.14
7.13
7.11
6.88
6.62
3.07
3.02
2.44
2.43
2.36
2.34
2.10
1.05
0.32
4.07
1.04
0.15
0.95
0.49
3.00
3.05
0.59
2.98
11
10
9
8
7
6
5
4
3
2
1
0
-1 ppm

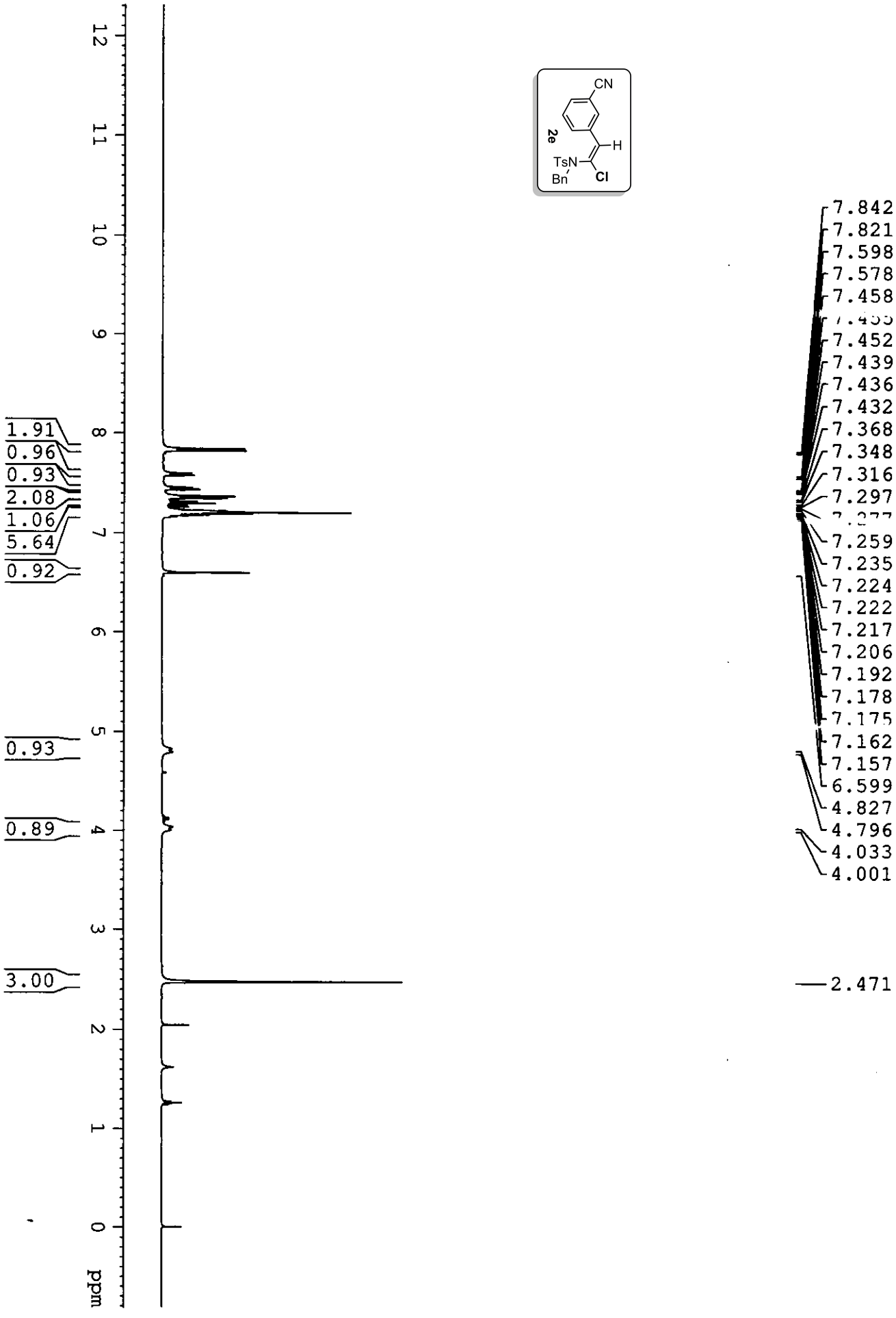

CH3
H
TsN
H3C
Cl
2b
144.38
138.14
134.38
133.02
132.48
130.66
129.63
129.57
129.48
128.77
128.58
128.28
125.72
77.35
77.03
76.72
35.69
21.62
21.43
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

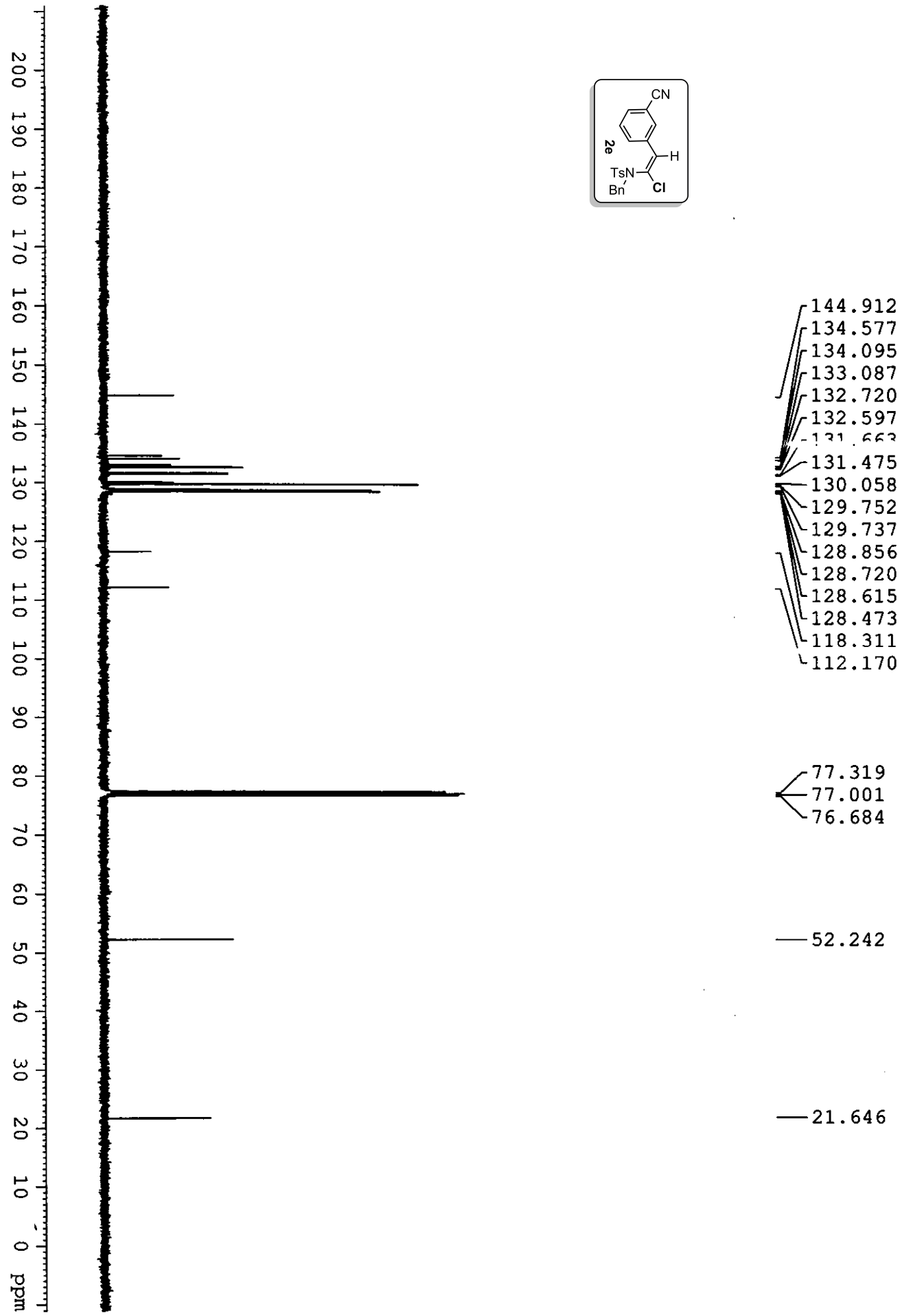

2c
7.838
7.818
7.781
7.342
7.324
7.296
7.275
7.263
7.260
7.247
7.169
7.140
7.124
7.097
7.012
6.952
6.933
6.536
6.511
4.793
4.762
4.090
4.058
2.412
2.168
2.116
1.98
4.70
2.14
1.87
1.06
1.03
1.02
0.92
0.95
3.17
3.35
2.95
ppm

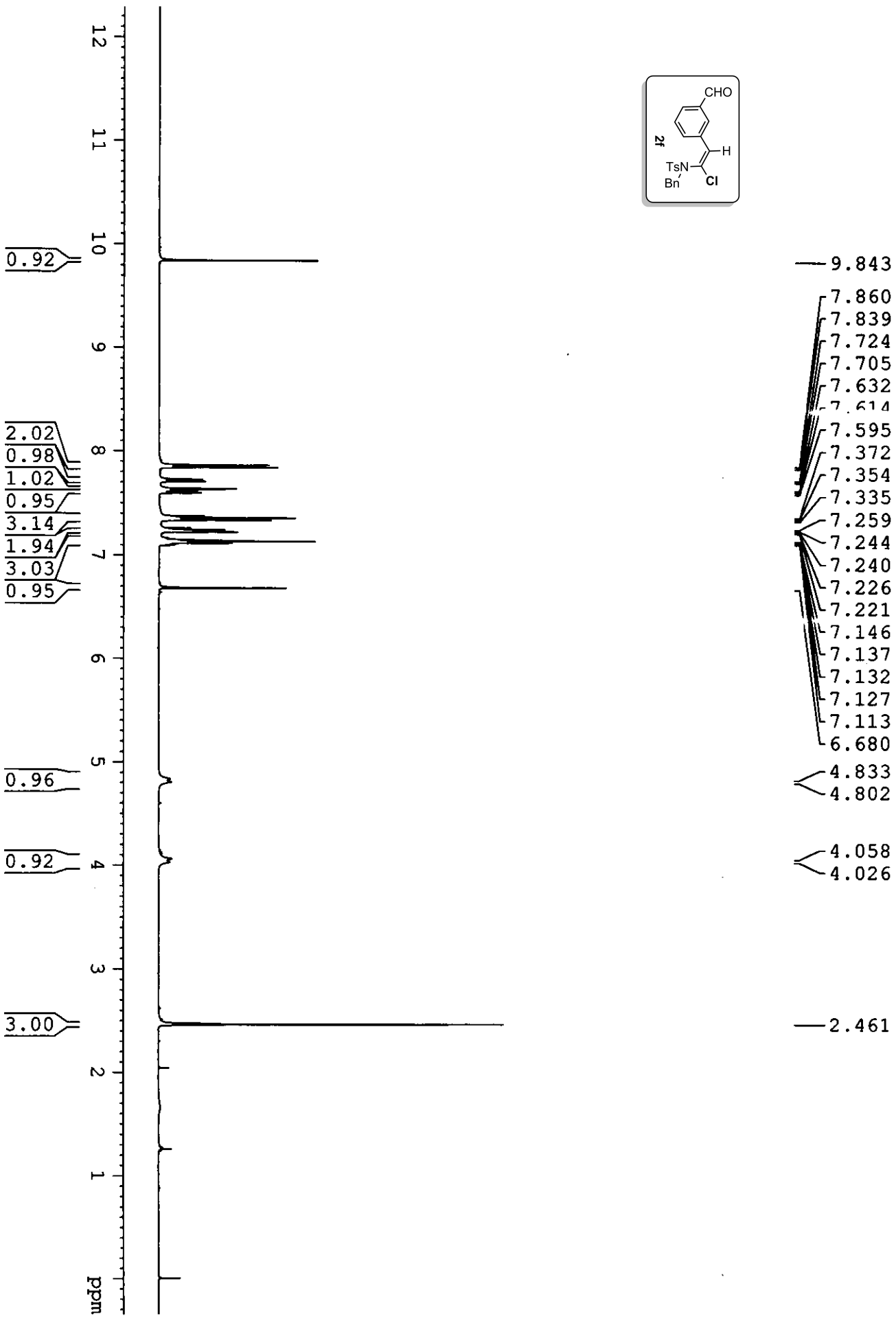

144.33
137.17
135.89
135.07
134.79
133.57
130.39
129.74
129.46
129.26
128.70
128.12
126.31
126.19
77.32
77.00
76.68
52.17
21.53
19.49
2c
H3C
CH3
H
TsN
Bn
Cl
ppm

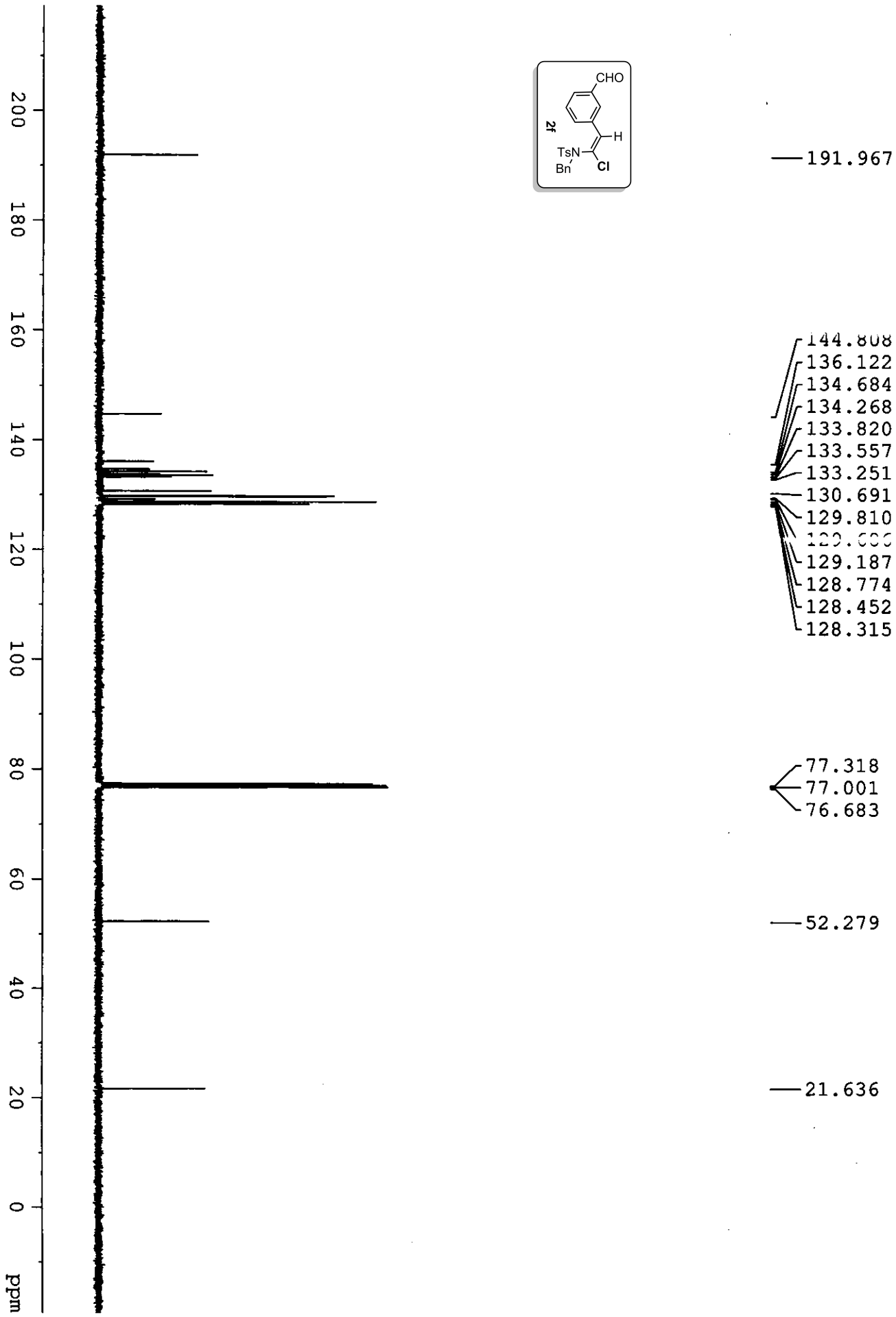

EtO2C
H
Cl
Ts-N
Bn
2d
7.868
7.857
7.852
7.847
7.836
7.356
7.335
7.258
7.222
7.203
7.168
7.161
7.155
7.151
7.141
7.127
7.123
7.102
6.635
4.829
4.797
4.390
4.373
4.355
4.337
4.146
4.128
4.110
4.092
4.045
4.013
2.462
2.039
1.410
1.392
1.374
1.273
1.255
1.237
0.09
3.85
4.05
0.12
1.92
2.81
0.92
0.06
0.89
2.09
0.34
0.88
3.00
0.43
3.06
0.54
ppm

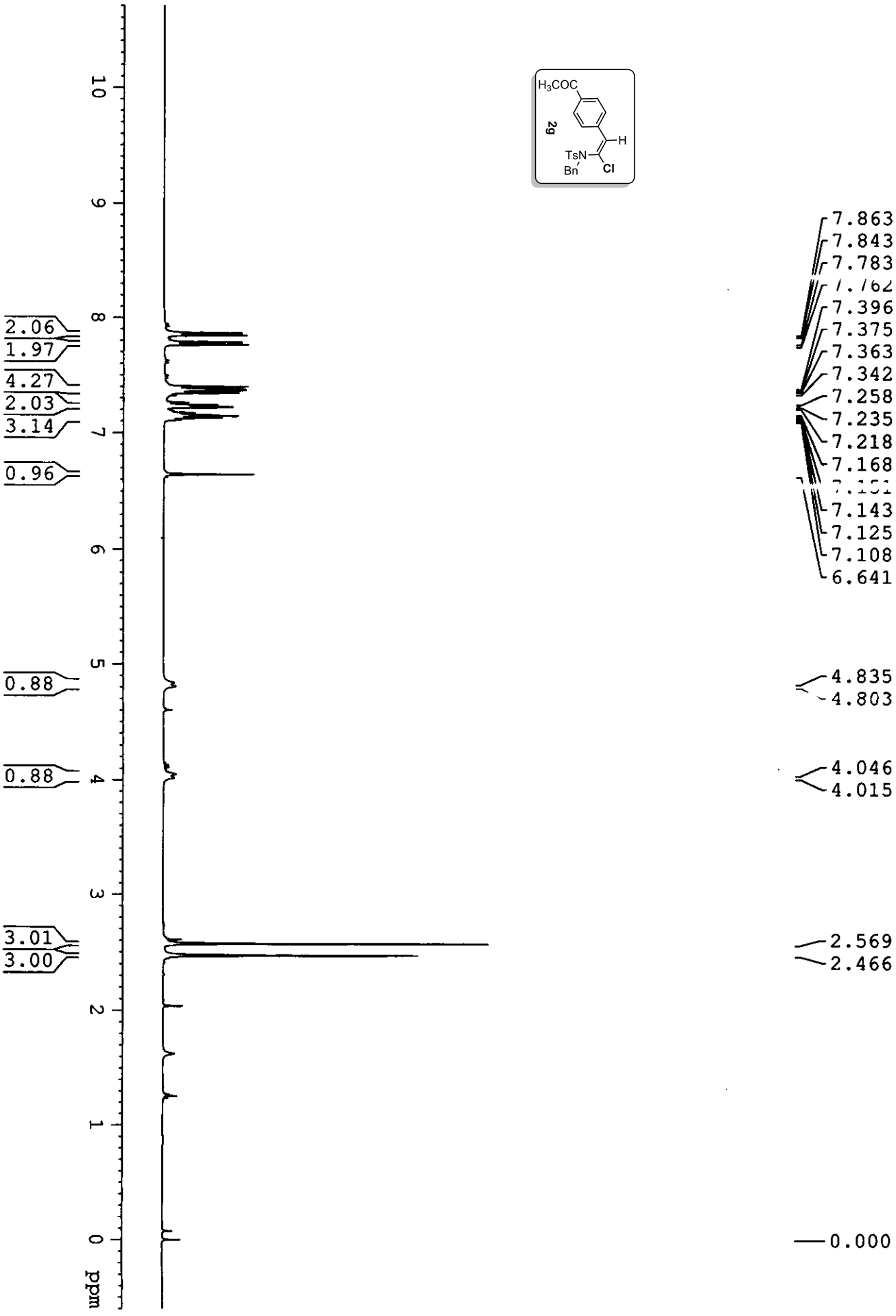

EtO2C
H
2d
Ts-N
Cl
Bn
166.199
144.742
137.150
134.729
134.022
133.159
130.016
129.779
129.652
129.525
129.243
128.857
128.503
128.444
128.298
77.322
77.004
76.687
60.942
52.427
21.639
14.298
200
180
160
140
120
100
80
60
40
20
0
ppm

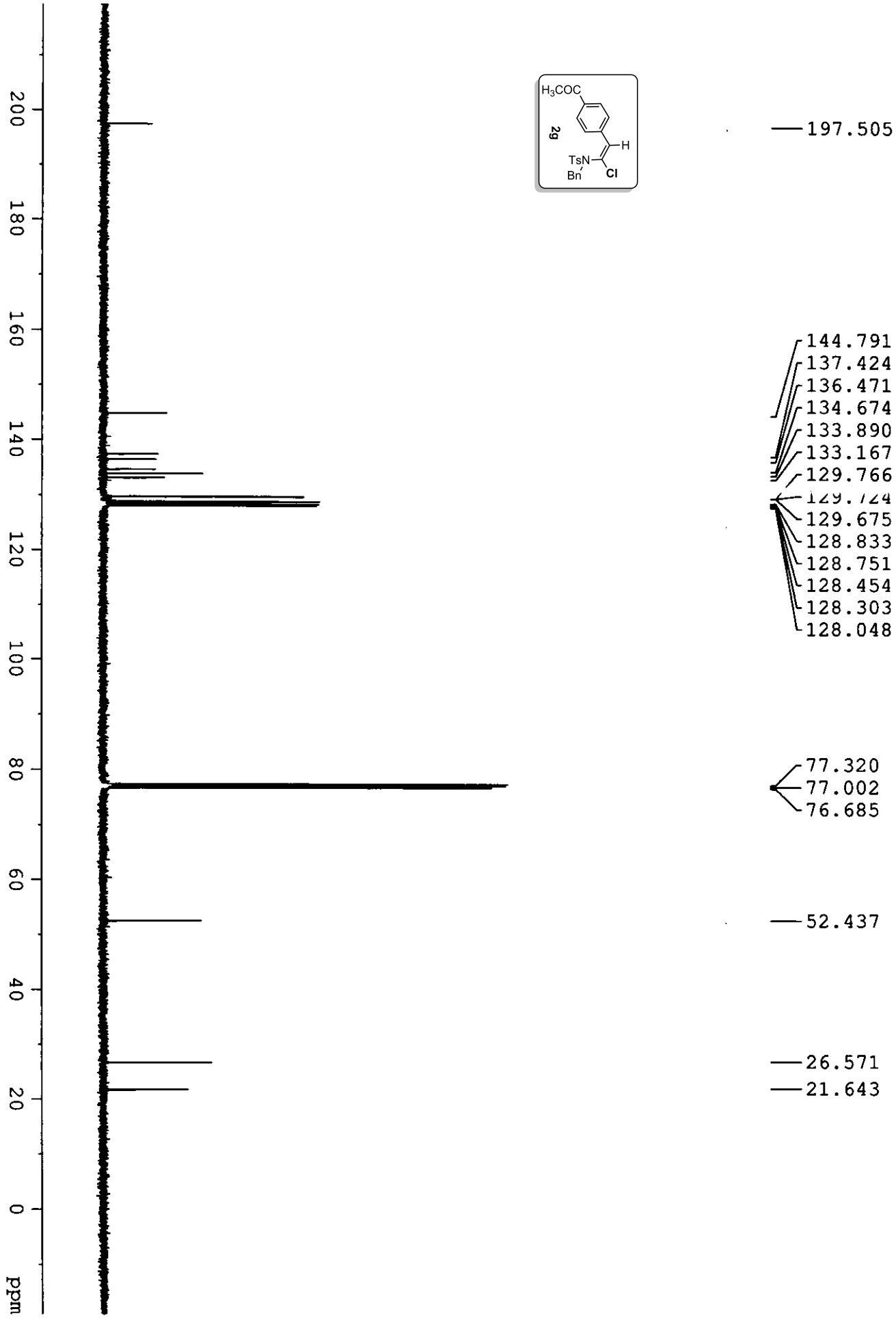

CN
2e
H
TsN
Bn
Cl
7.842
7.821
7.598
7.578
7.458
7.452
7.439
7.436
7.432
7.368
7.348
7.316
7.297
7.259
7.235
7.224
7.222
7.217
7.206
7.192
7.178
7.175
7.162
7.157
6.599
4.827
4.796
4.033
4.001
2.471
1.91
0.96
0.93
2.08
1.06
5.64
0.92
0.93
0.89
3.00
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm

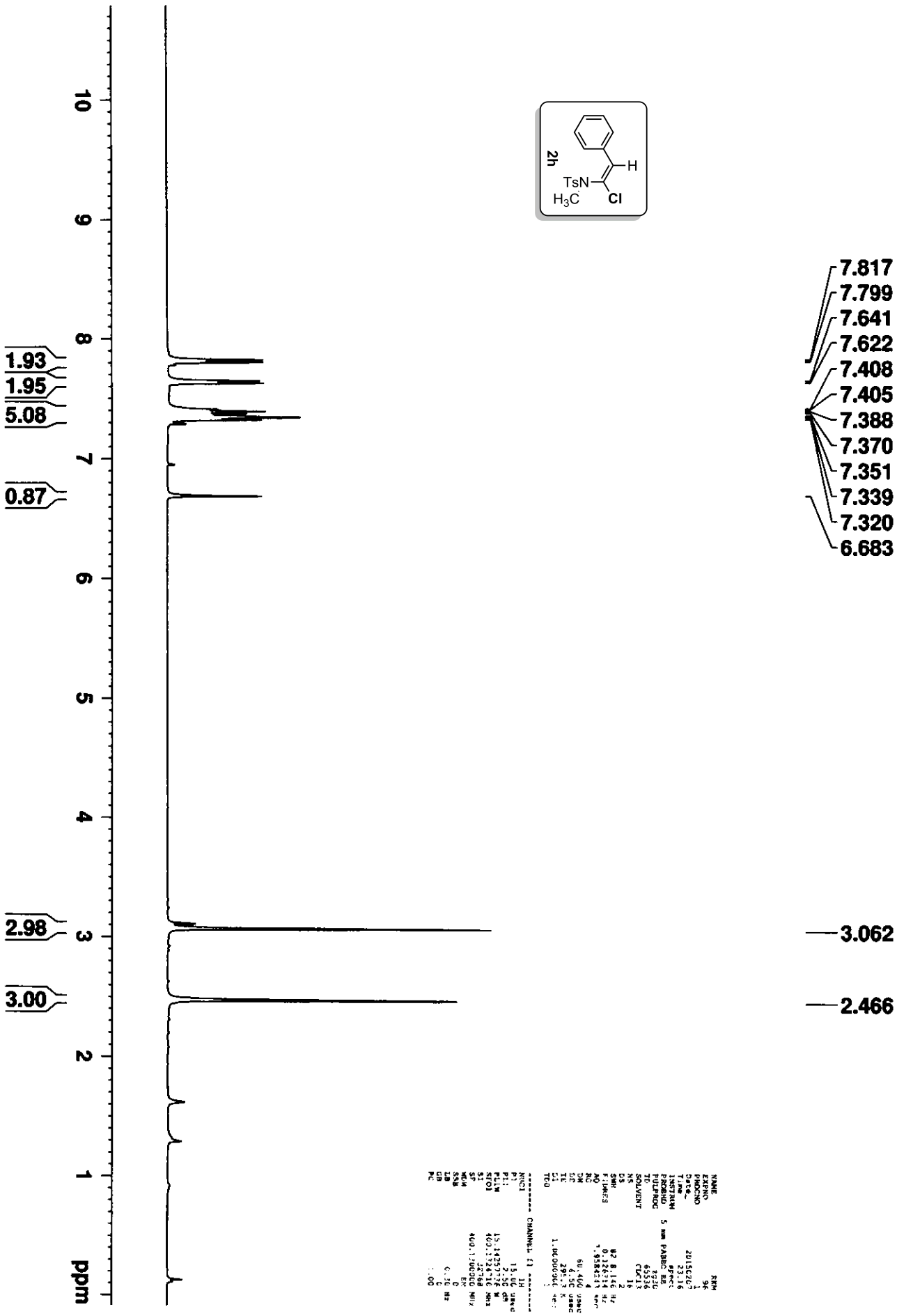

CN
2e
H
TsN
Bn
Cl
144.912
134.577
134.095
133.087
132.720
132.597
131.475
130.058
129.752
129.737
128.856
128.720
128.615
128.473
118.311
112.170
77.319
77.001
76.684
52.242
21.646
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

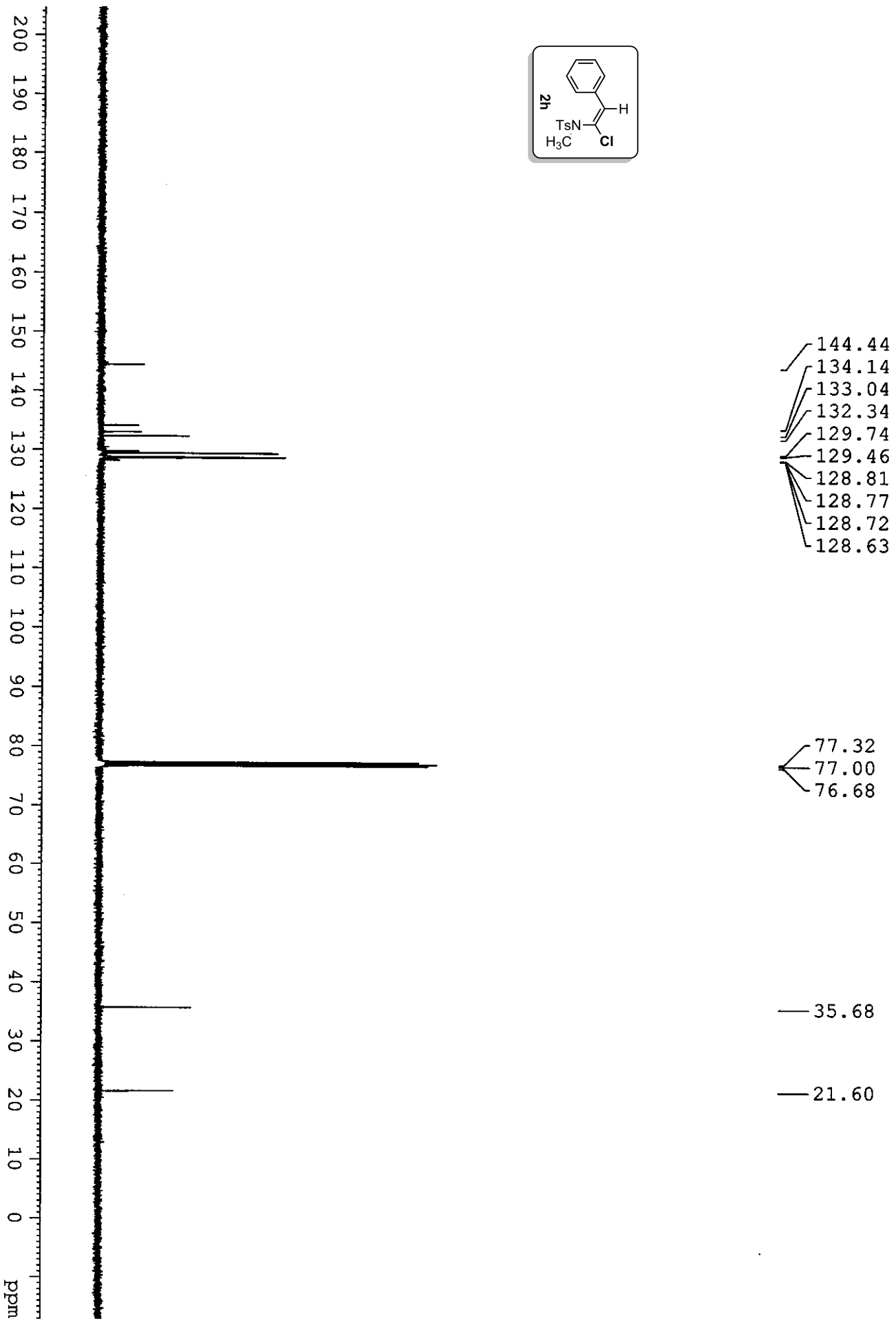

CHO
2f
H
TsN
Bn
Cl
0.92
2.02
0.98
1.02
0.95
3.14
1.94
3.03
0.95
0.96
0.92
3.00
ppm
9.843
7.860
7.839
7.724
7.705
7.632
7.595
7.372
7.354
7.335
7.259
7.244
7.240
7.226
7.221
7.146
7.137
7.132
7.127
7.113
6.680
4.833
4.802
4.058
4.026
2.461

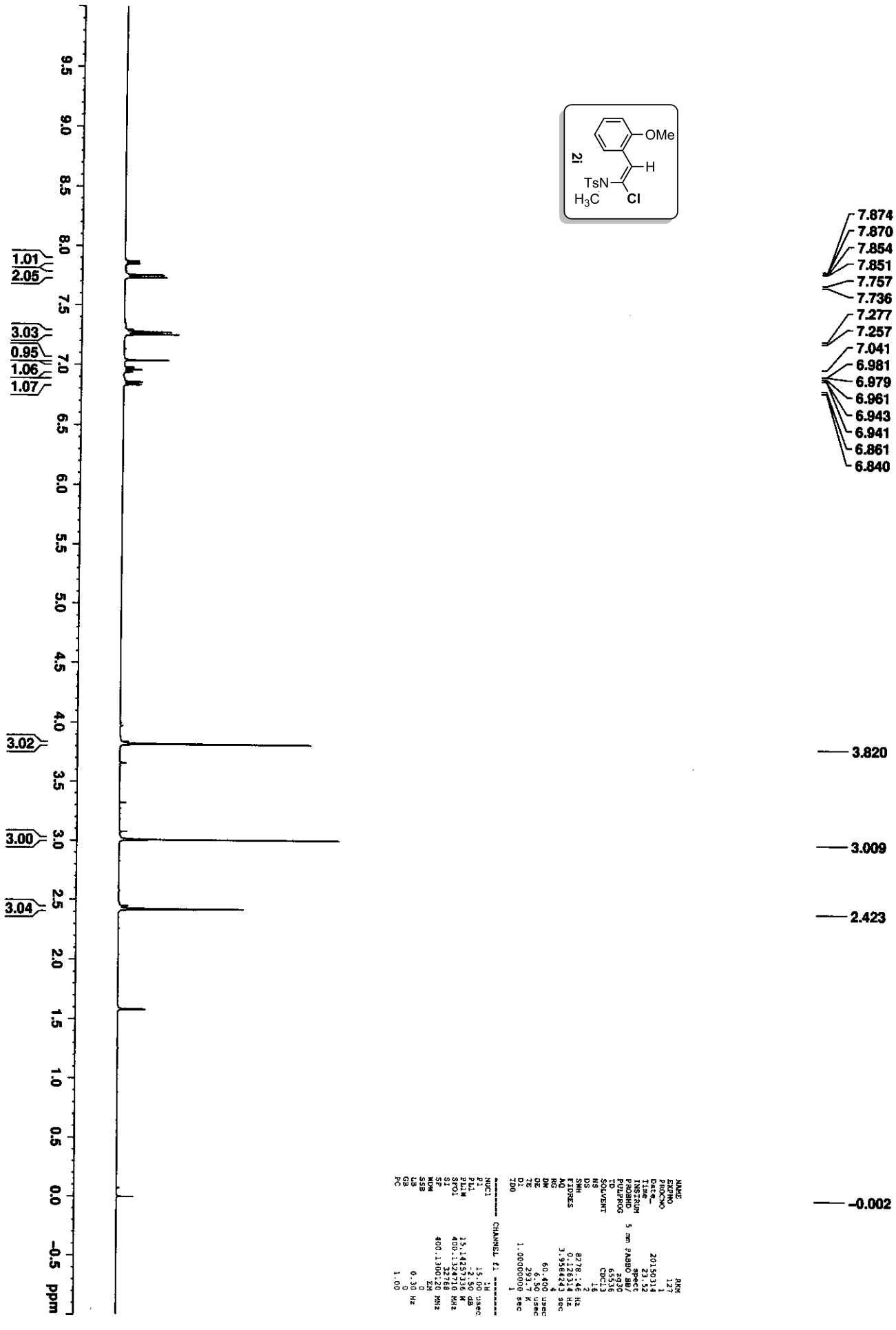

CHO
3f
H
TsN
Bn
Cl
191.967
144.808
136.122
134.684
134.268
133.820
133.557
133.251
130.691
129.810
129.187
128.774
128.452
128.315
77.318
77.001
76.683
52.279
21.636
200
180
160
140
120
100
80
60
40
20
0
ppm

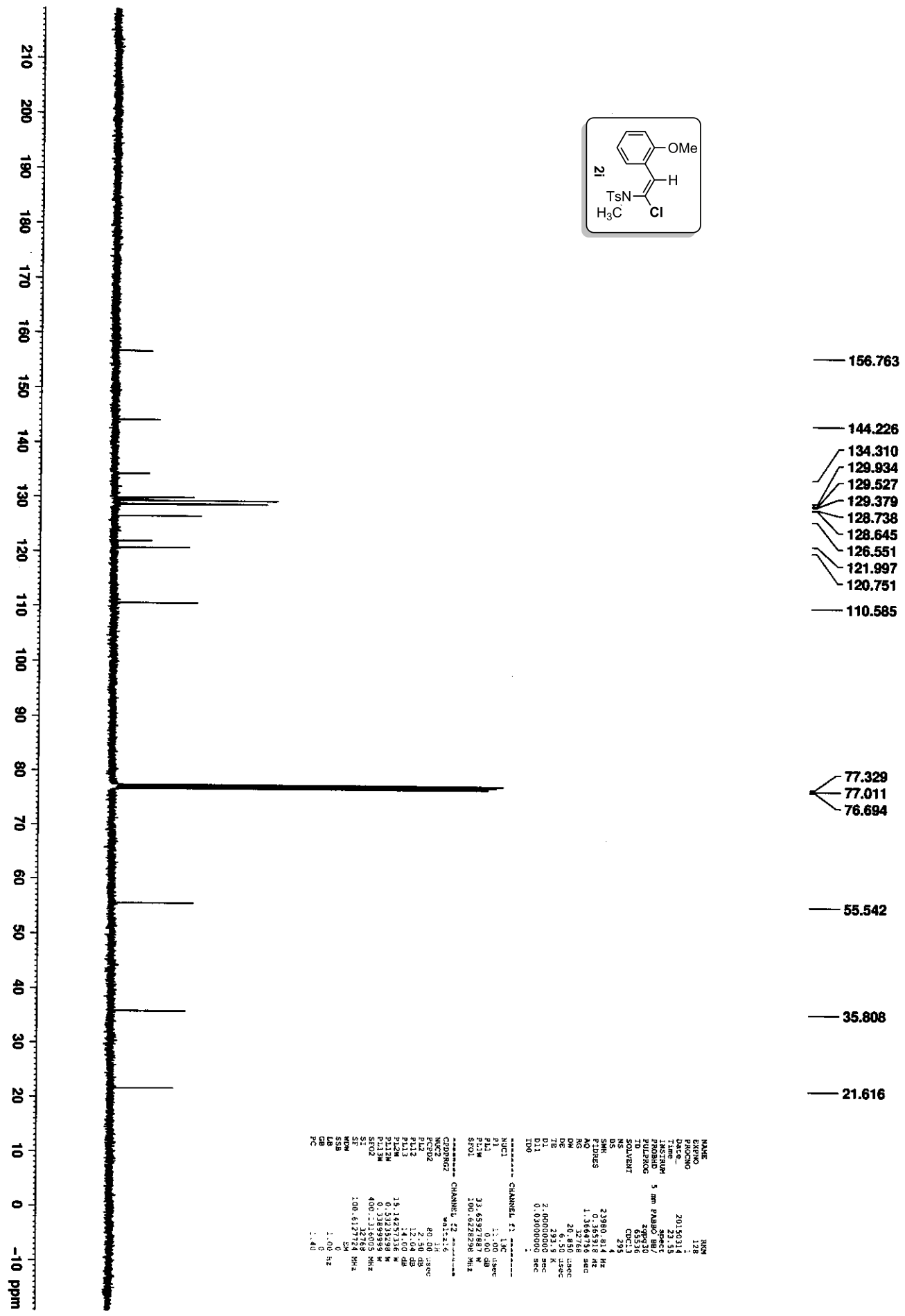

H3COC
2g
TsN
Bn
Cl
H
7.863
7.843
7.783
7.762
7.396
7.375
7.363
7.342
7.258
7.235
7.218
7.168
7.143
7.125
7.108
6.641
4.835
4.803
4.046
4.015
2.569
2.466
0.000
2.06
1.97
4.27
2.03
3.14
0.96
0.88
0.88
3.01
3.00
10
9
8
7
6
5
4
3
2
1
0
ppm

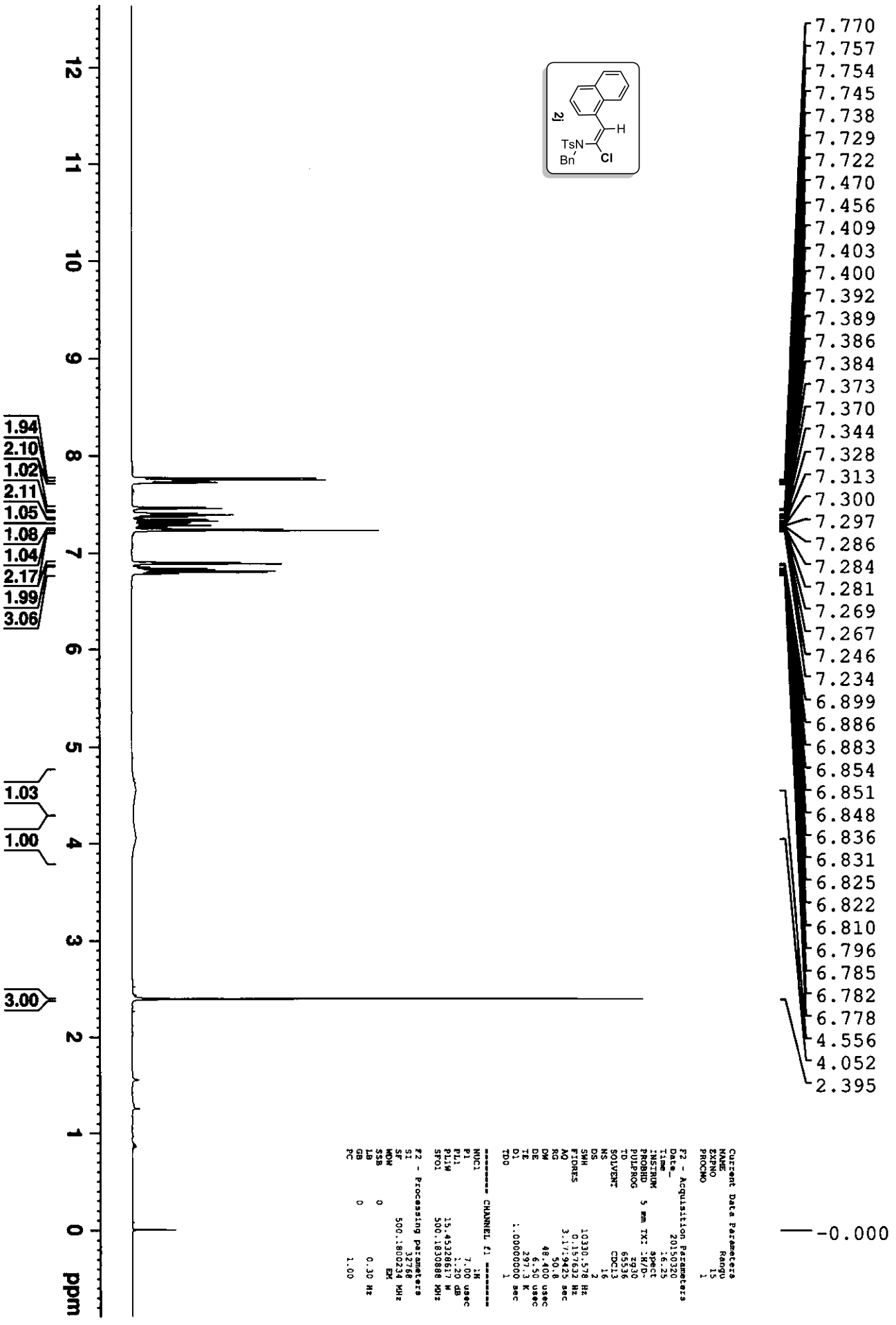

H3COC
2g
TsN
Bn
Cl
H
197.505
144.791
137.424
136.471
134.674
133.890
133.167
129.766
129.724
129.675
128.833
128.751
128.454
128.303
128.048
77.320
77.002
76.685
52.437
26.571
21.643
200
180
160
140
120
100
80
60
40
20
0
ppm

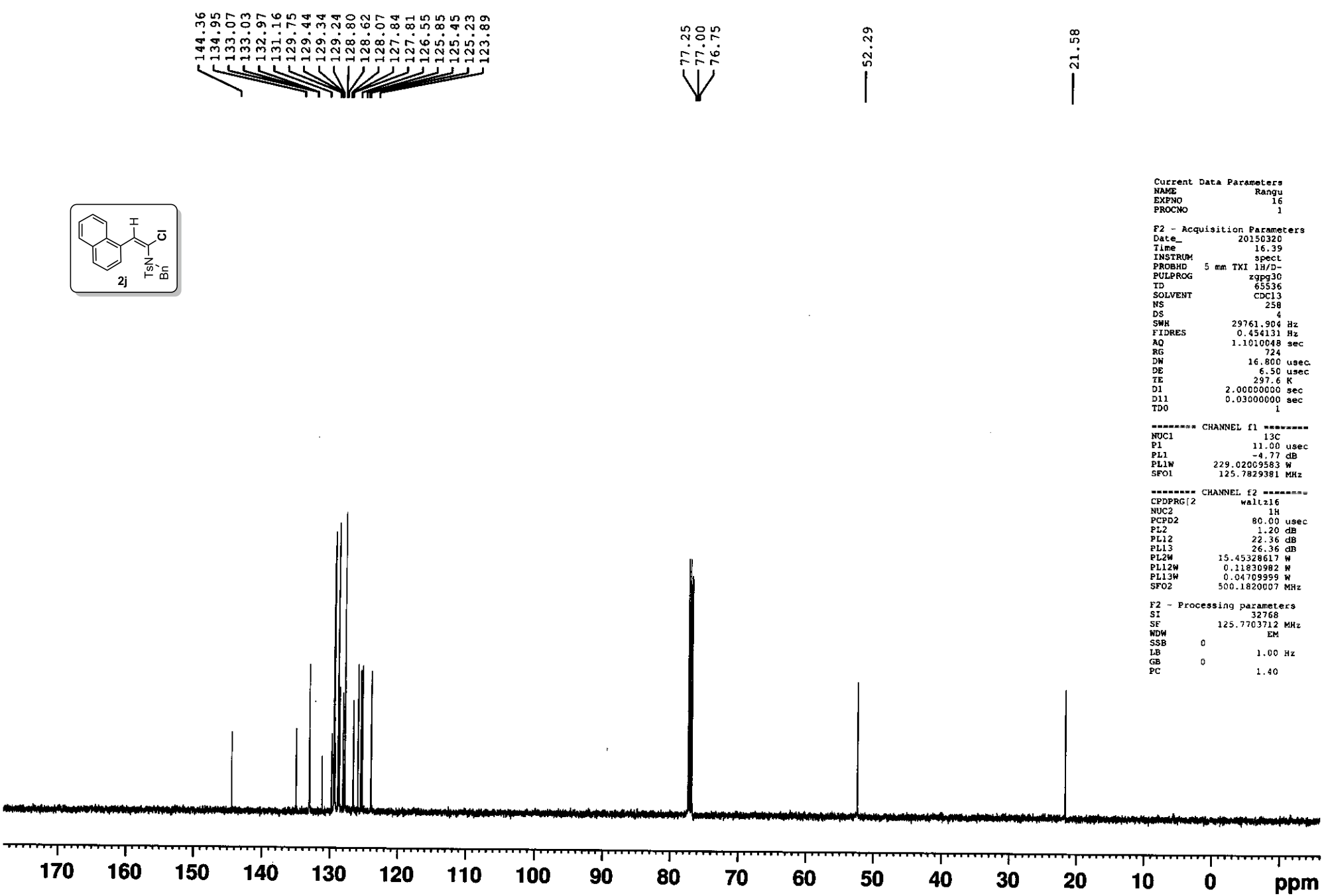

2h
TsN
H
H3C
Cl
7.817
7.799
7.641
7.622
7.408
7.405
7.388
7.370
7.351
7.339
7.320
6.683
3.062
2.466
1.93
1.95
5.08
0.87
2.98
3.00
ppm

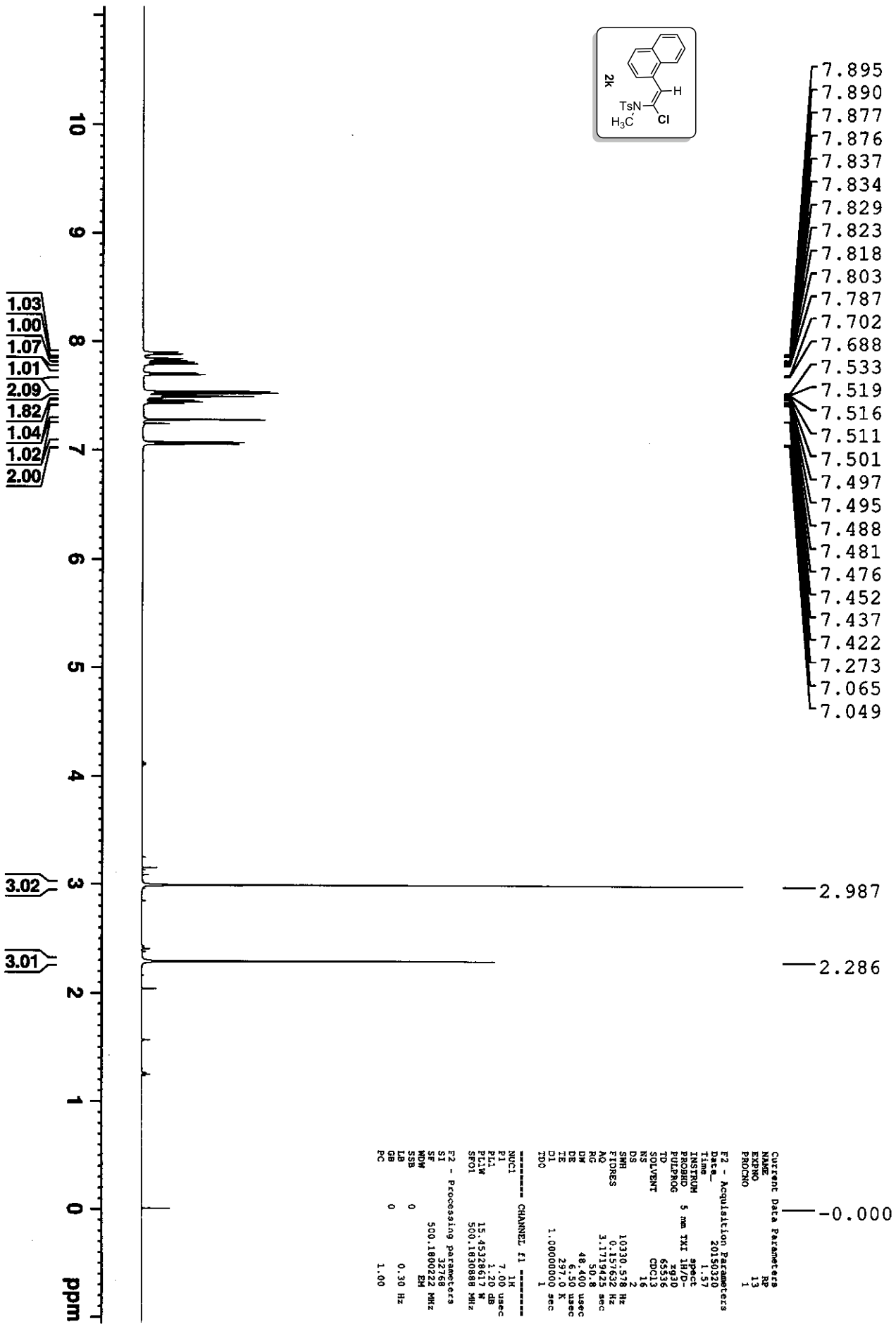

2h
TsN
H3C
Cl
H
144.44
134.14
133.04
132.34
129.74
129.46
128.81
128.77
128.72
128.63
77.32
77.00
76.68
35.68
21.60
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

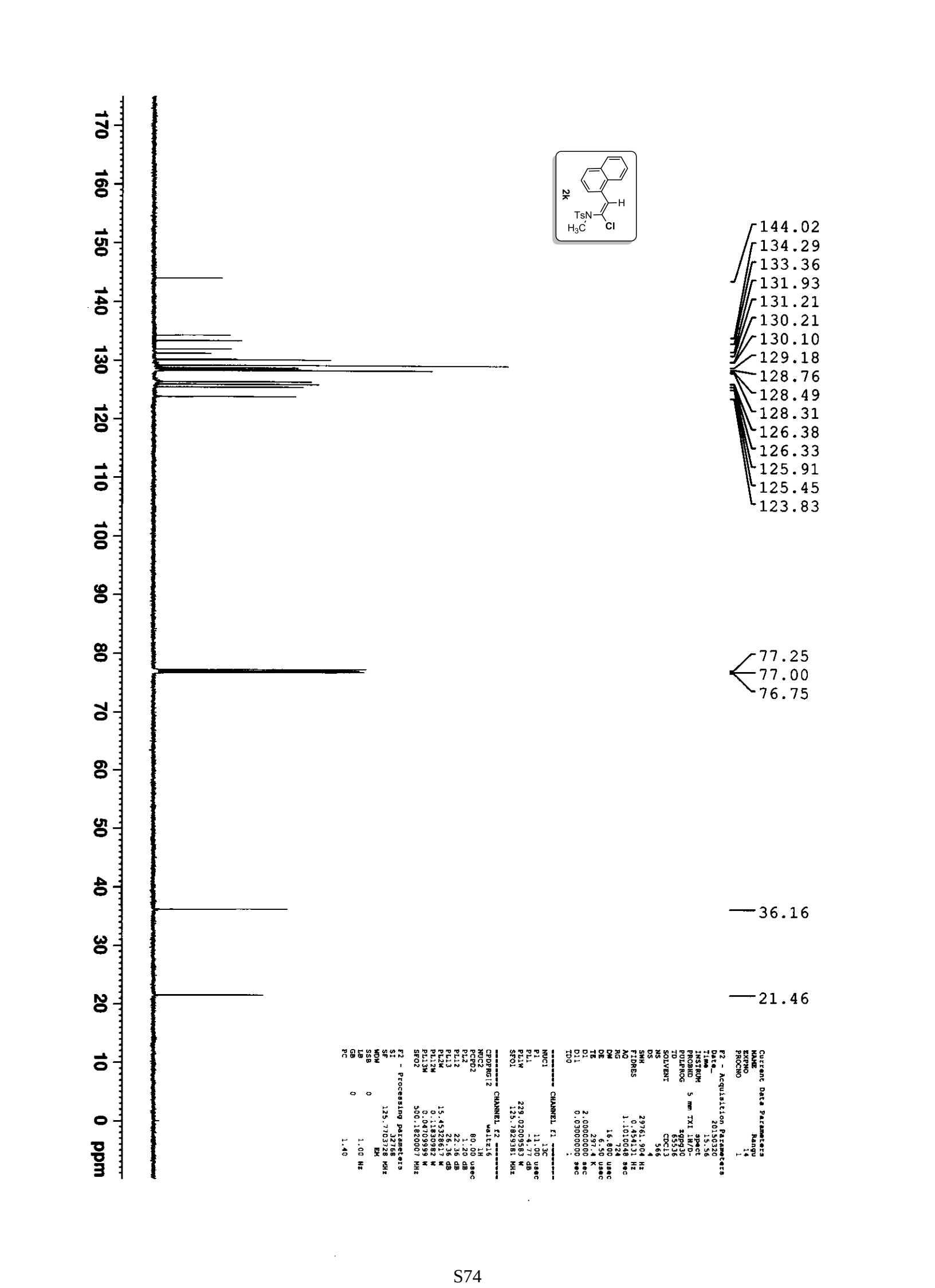

2l
OMe
H
TsN
H3C
Cl
7.874
7.870
7.854
7.851
7.757
7.736
7.277
7.257
7.041
6.981
6.979
6.961
6.943
6.941
6.861
6.840
3.820
3.009
2.423
-0.002
1.01
2.05
3.03
0.95
1.06
1.07
3.02
3.00
3.04
ppm

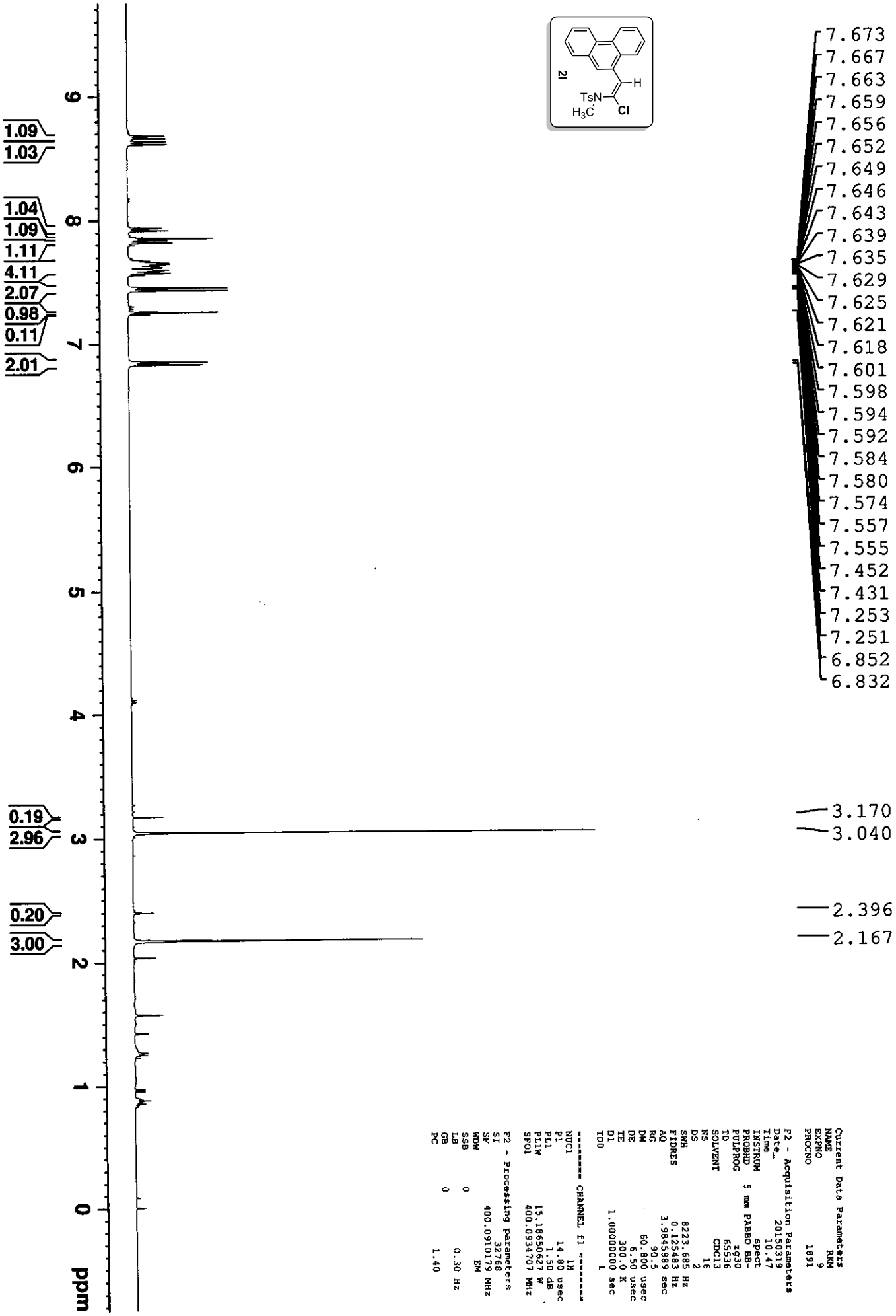

OMe
H
TsN
H3C
Cl
2i
156.763
144.226
134.310
129.934
129.527
129.379
128.738
128.645
126.551
121.997
120.751
110.585
77.329
77.011
76.694
55.542
35.808
21.616
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10 ppm

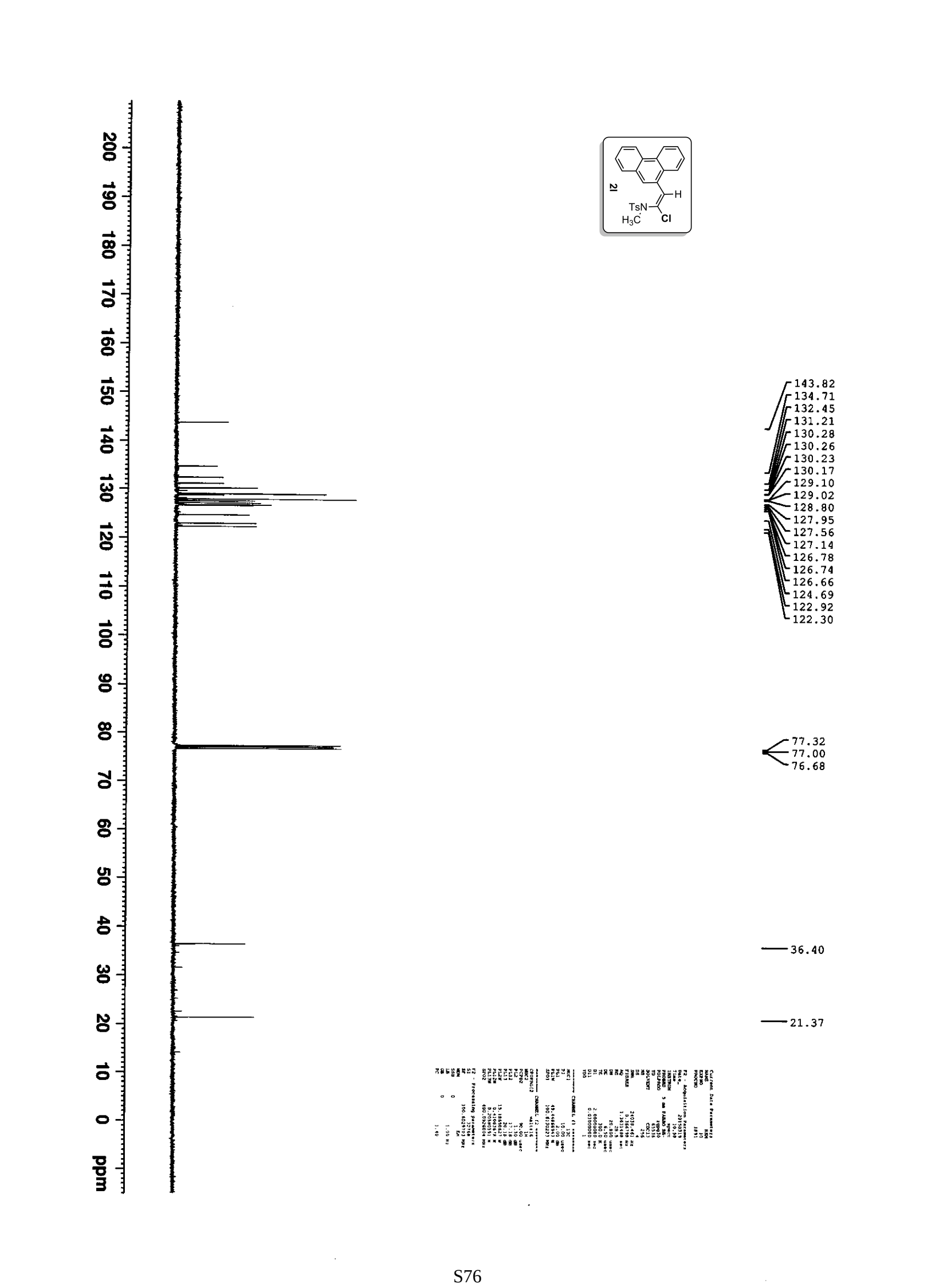

2j
TsN
Bn
H
Cl
1.94
2.10
1.02
2.11
1.05
1.08
1.04
2.17
1.99
3.06
1.03
1.00
3.00
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
7.770
7.757
7.754
7.745
7.738
7.729
7.722
7.470
7.456
7.409
7.403
7.400
7.392
7.389
7.386
7.384
7.373
7.370
7.344
7.328
7.313
7.300
7.297
7.286
7.284
7.281
7.269
7.267
7.246
7.234
6.899
6.886
6.883
6.854
6.851
6.848
6.836
6.831
6.825
6.822
6.810
6.796
6.785
6.782
6.778
4.556
4.052
2.395
-0.000
Current Data Parameters
NAME Rangu
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150320
Time 16.25
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 50.8
DW 48.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 1.20 dB
PL1W 15.45328617 W
SFO1 500.1830888 MHz
F2 - Processing parameters
SI 32768
SF 500.1800234 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

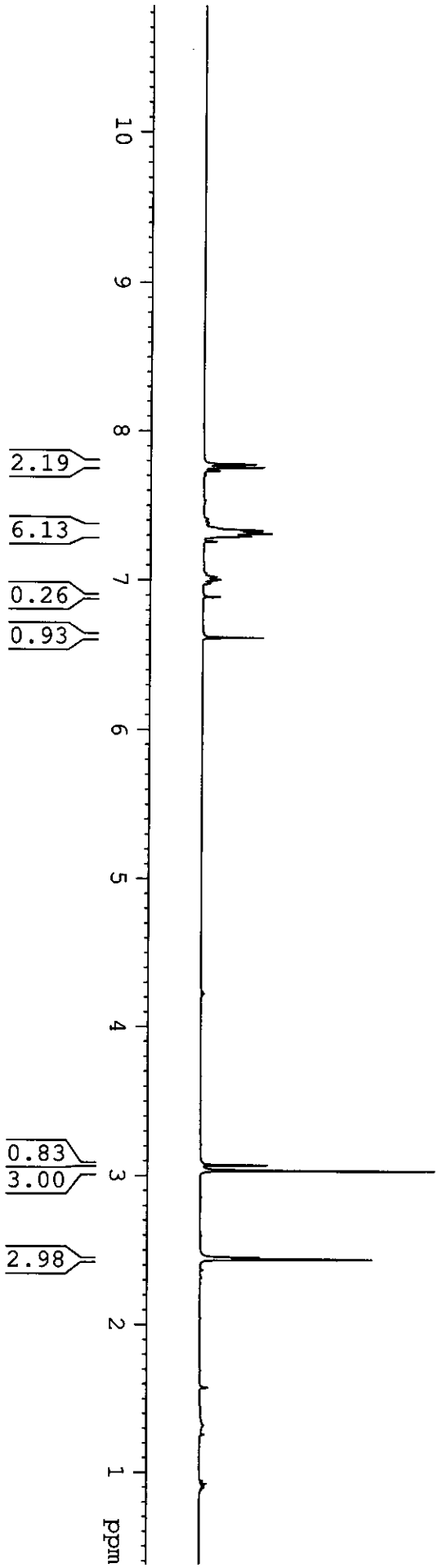

144.36
134.95
133.07
133.03
132.97
131.16
129.75
129.44
129.34
129.24
128.80
128.62
128.07
127.84
127.81
126.55
125.85
125.45
125.23
123.89
77.25
77.00
76.75
52.29
21.58
2j
Current Data Parameters
NAME Rangu
EXPNO 16
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150320
Time 16.39
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 258
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 724
DW 16.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 -4.77 dB
PL1W 229.02009583 W
SFO1 125.7829381 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.20 dB
PL12 22.36 dB
PL13 26.36 dB
PL2W 15.45328617 W
PL12W 0.11830982 W
PL13W 0.04709999 W
SFO2 500.1820007 MHz
F2 - Processing parameters
SI 32768
SF 125.7703712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

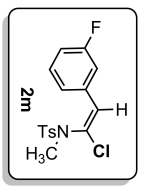

2k
TsN
H3C
Cl
H
7.895
7.890
7.877
7.876
7.837
7.834
7.829
7.823
7.818
7.803
7.787
7.702
7.688
7.533
7.519
7.516
7.511
7.501
7.497
7.495
7.488
7.481
7.476
7.452
7.437
7.422
7.273
7.065
7.049
2.987
2.286
-0.000
1.03
1.00
1.07
1.01
2.09
1.82
1.04
1.02
2.00
3.02
3.01
ppm
Current Data Parameters
NAME RP
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150320
Time 1.57
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 50.8
DW 48.400 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 1.20 dB
PL1W 15.45328617 W
SFO1 500.1830888 MHz
F2 - Processing parameters
SI 32768
SF 500.1800222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


2k
TsN
H3C
Cl
H
144.02
134.29
133.36
131.93
131.21
130.21
130.10
129.18
128.76
128.49
128.31
126.38
126.33
125.91
125.45
123.83
77.25
77.00
76.75
36.16
21.46
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


2l
TsN
H3C
Cl
H
7.673
7.667
7.663
7.659
7.656
7.652
7.649
7.646
7.643
7.639
7.635
7.629
7.625
7.621
7.618
7.601
7.598
7.594
7.592
7.584
7.580
7.574
7.557
7.555
7.452
7.431
7.253
7.251
6.852
6.832
3.170
3.040
2.396
2.167
1.09
1.03
1.04
1.09
1.11
4.11
2.07
0.98
0.11
2.01
0.19
2.96
0.20
3.00
ppm
Current Data Parameters
NAME RKM
EXPNO 9
PROCNO 1891
F2 - Acquisition Parameters
Date_ 20150319
Time_ 10.47
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 90.5
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.80 usec
PL1 1.50 dB
PL1W 15.18650627 W
SFO1 400.0934707 MHz
F2 - Processing parameters
SI 32768
SF 400.0910179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40


2l
TsN
H3C
Cl
H
143.82
134.71
132.45
131.21
130.28
130.26
130.23
130.17
129.10
129.02
128.80
127.95
127.56
127.14
126.78
126.74
126.66
124.69
122.92
122.30
77.32
77.00
76.68
36.40
21.37
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm





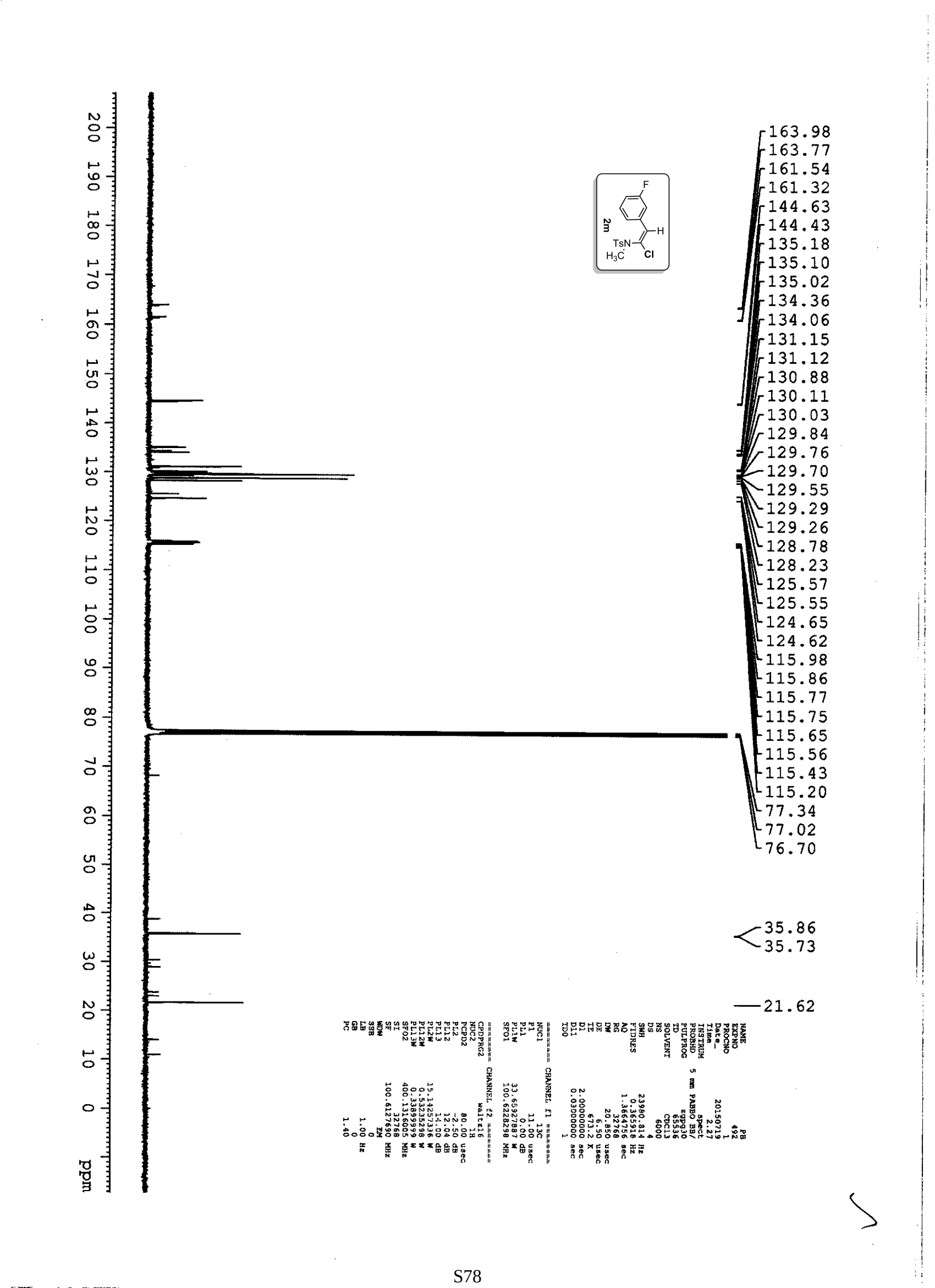
7.777
7.756
7.732
7.333
7.321
7.313
7.301
7.293
7.257
7.025
7.021
7.014
7.002
6.997
6.984
6.981
6.888
6.617

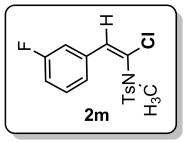
3.069
3.032

2.448
2.436

NAME RRM
EXPNO 112
PROCNO 1
Date_ 20150226
Time 22.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

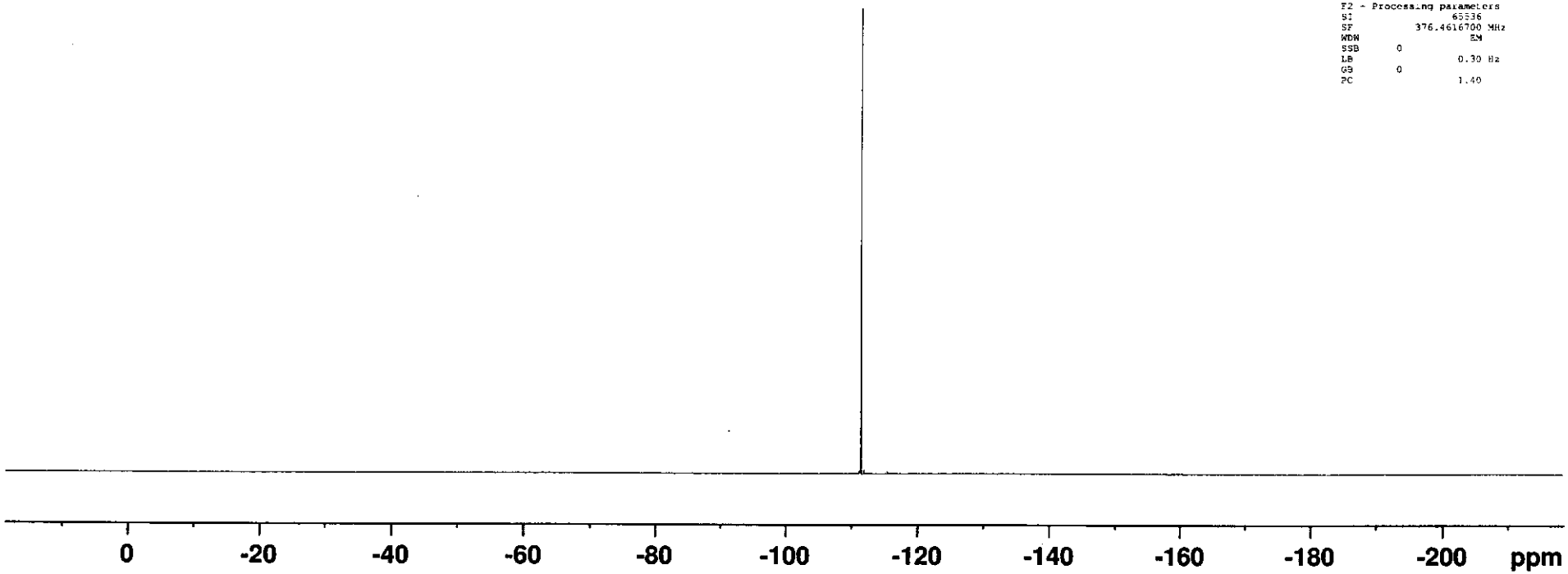
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300217 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

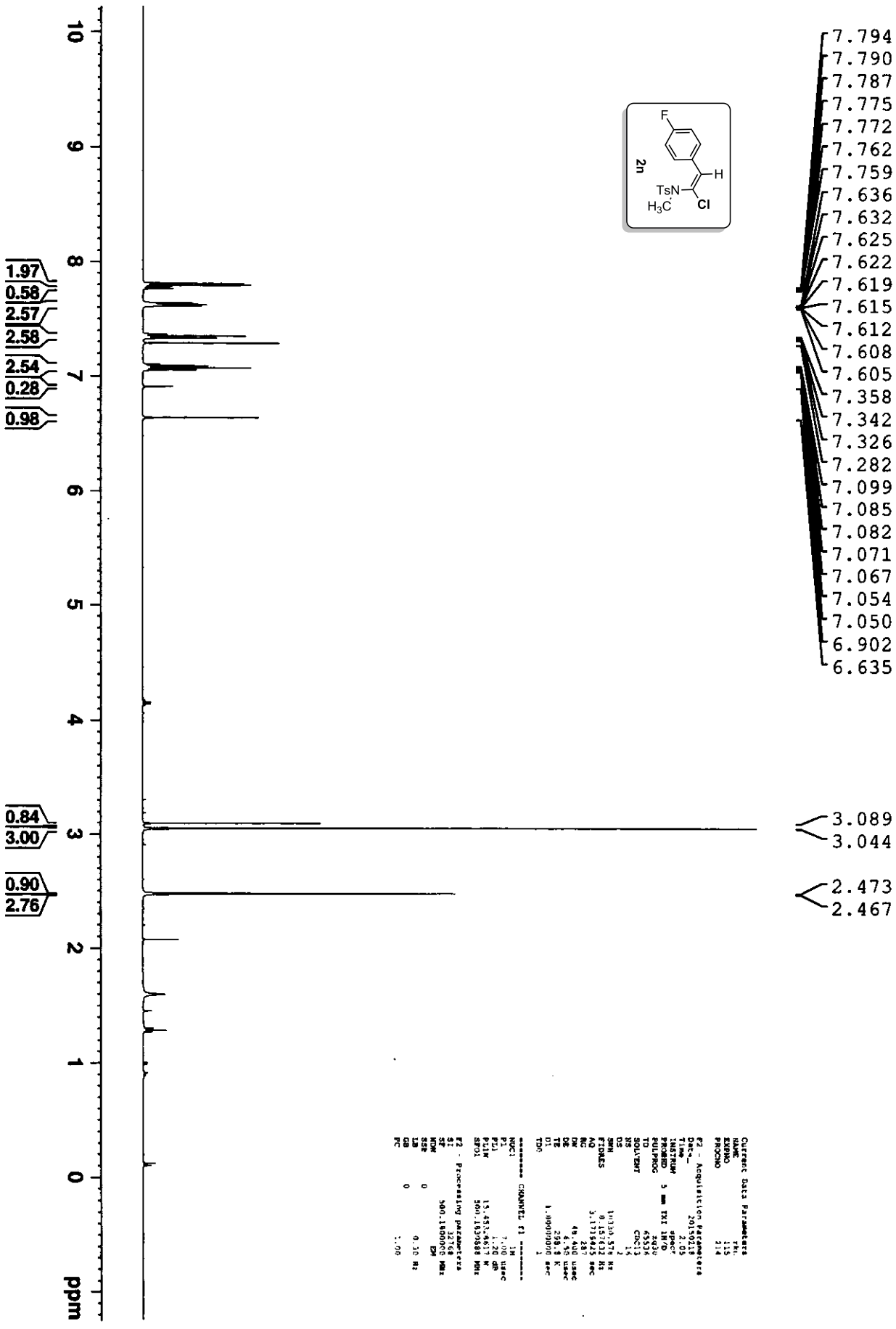

F
2m
H
TsN
H3C
Cl
163.98
163.77
161.54
161.32
144.63
144.43
135.18
135.10
135.02
134.36
134.06
131.15
131.12
130.88
130.11
130.03
129.84
129.76
129.70
129.55
129.29
129.26
128.78
128.23
125.57
125.55
124.65
124.62
115.98
115.86
115.77
115.75
115.65
115.56
115.43
115.20
77.34
77.02
76.70
35.86
35.73
21.62
NAME PB
EXPNO 492
PROCNO 1
Date_ 20150719
Time 2.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
PL1W 33.65927887 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.50 dB
PL12 12.04 dB
PL13 14.00 dB
PL2W 15.14257336 W
PL12W 0.53235298 W
PL13W 0.33899999 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

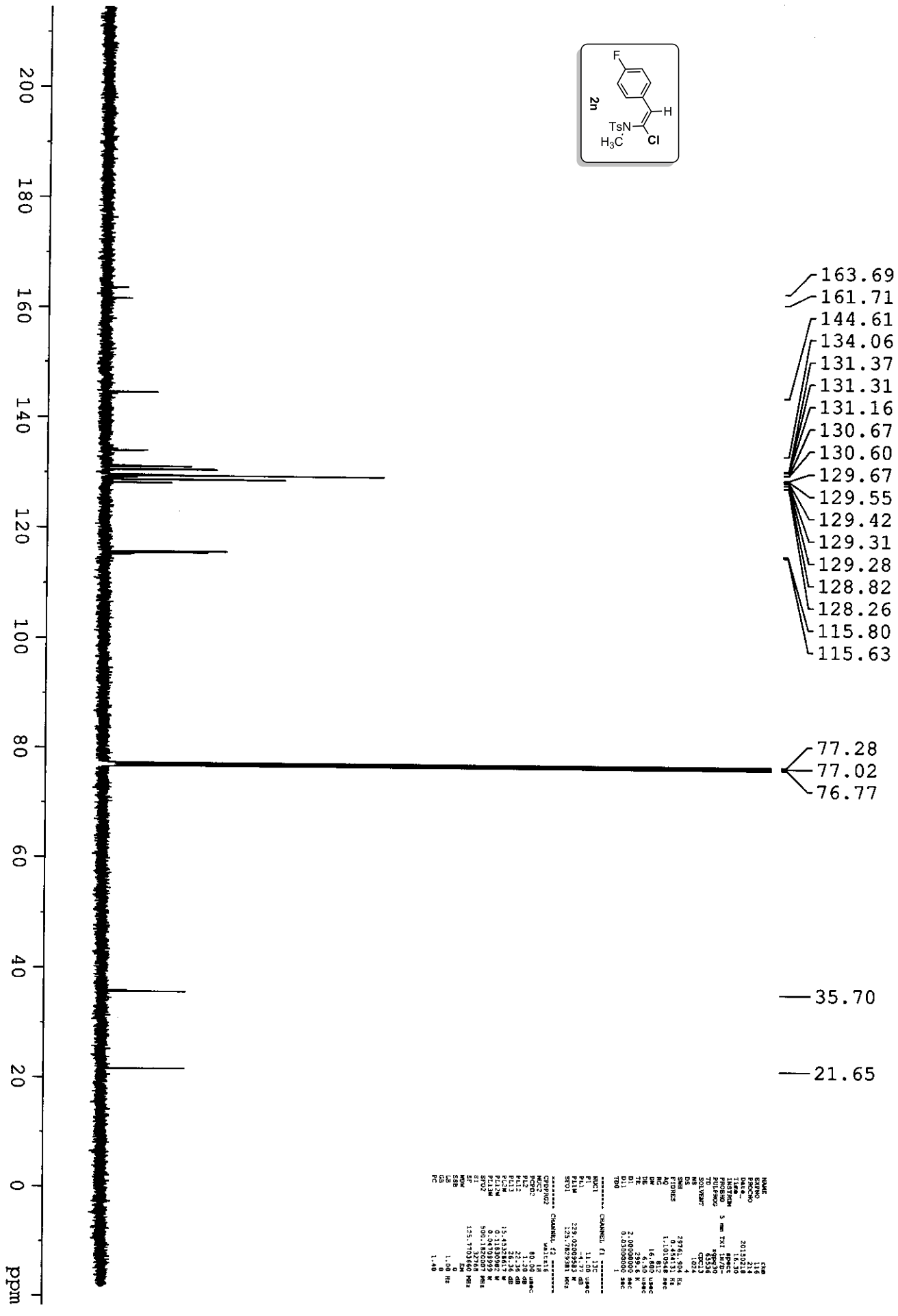


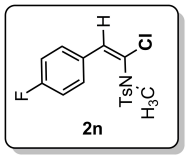
-111.48

```
Current Data Parameters
NAME                  RKM
EXPNO                  26
PROCNO               1891

F2 - Acquisition Parameters
Date_           20150515
Time                15.45
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgfhigqn
TD                131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH             89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340032 sec
RG                   2050
DW                5.600 usec
DE                6.50 usec
TE                298.9 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  19F
P1                 14.80 usec
PL1                -1.20 dB
PL1W         18.11287308 W
SFO1         376.4240234 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                 1.50 dB
PL12               17.18 dB
PL2W         15.18650627 W
PL12W         0.41063678 W
SFO2         400.0926004 MHz

F2 - Processing parameters
SI                  65536
SF           376.4616700 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.40
```




7.794
7.790
7.787
7.775
7.772
7.762
7.759
7.636
7.632
7.625
7.622
7.619
7.615
7.612
7.608
7.605
7.358
7.342
7.326
7.282
7.099
7.085
7.082
7.071
7.067
7.054
7.050
6.902
6.635
3.089
3.044
2.473
2.467
1.97
0.58
2.57
2.58
2.54
0.28
0.98
0.84
3.00
0.90
2.76
ppm
2n
F
H
TsN
H3C
Cl

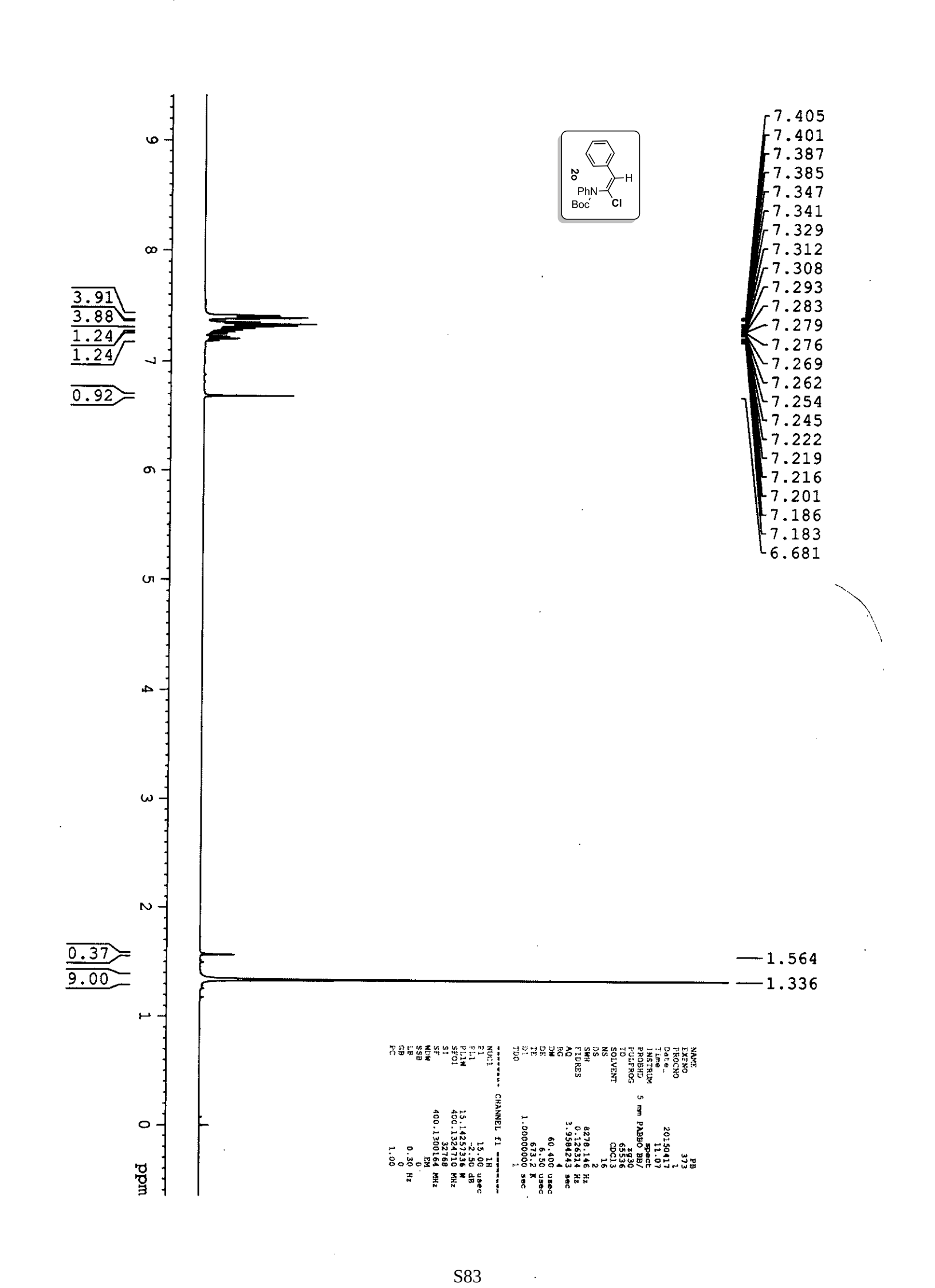

2n
F
H
TsN
H3C
Cl
163.69
161.71
144.61
134.06
131.37
131.31
131.16
130.67
130.60
129.67
129.55
129.42
129.31
129.28
128.82
128.26
115.80
115.63
77.28
77.02
76.77
35.70
21.65
ppm
200
180
160
140
120
100
80
60
40
20
0

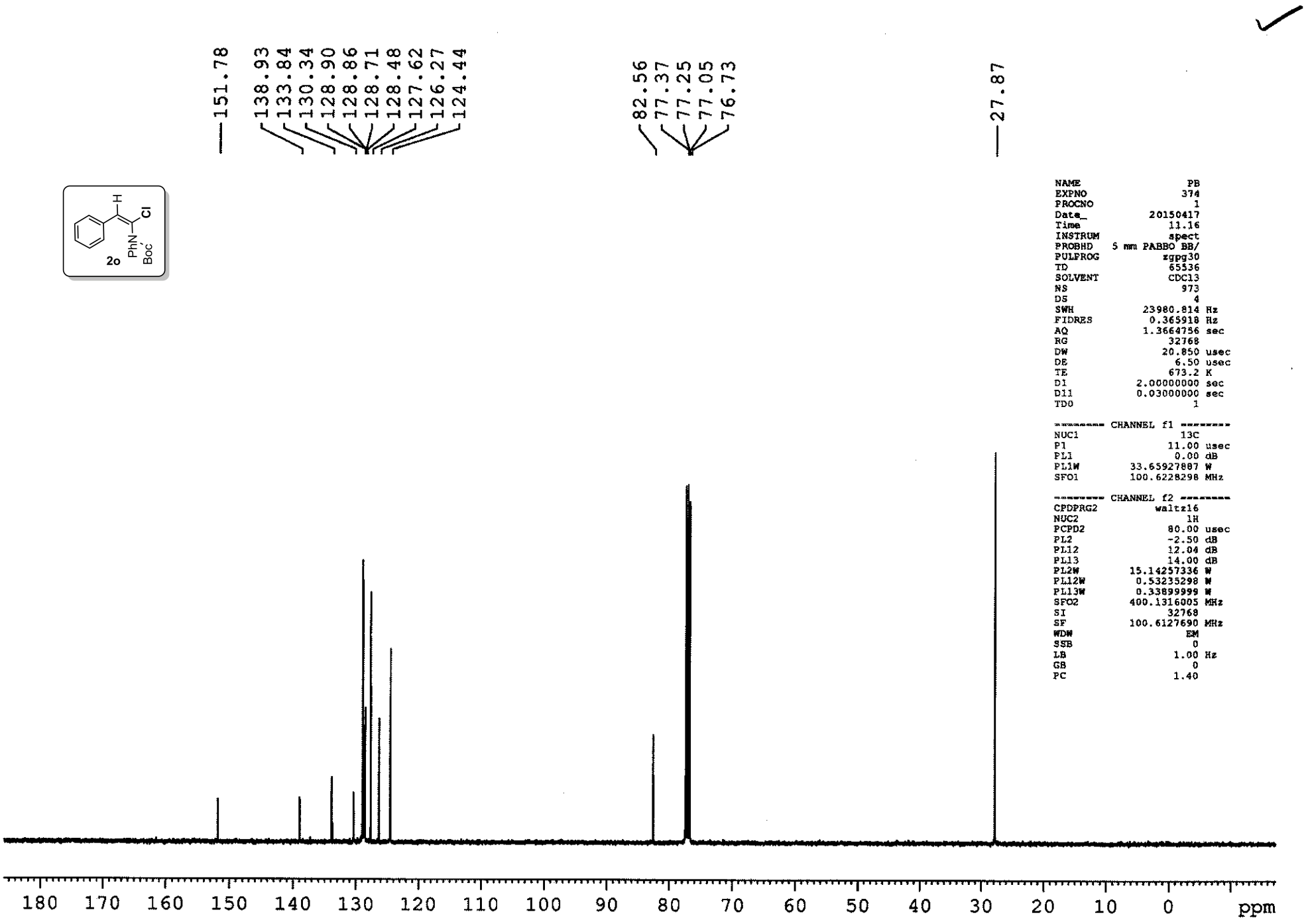


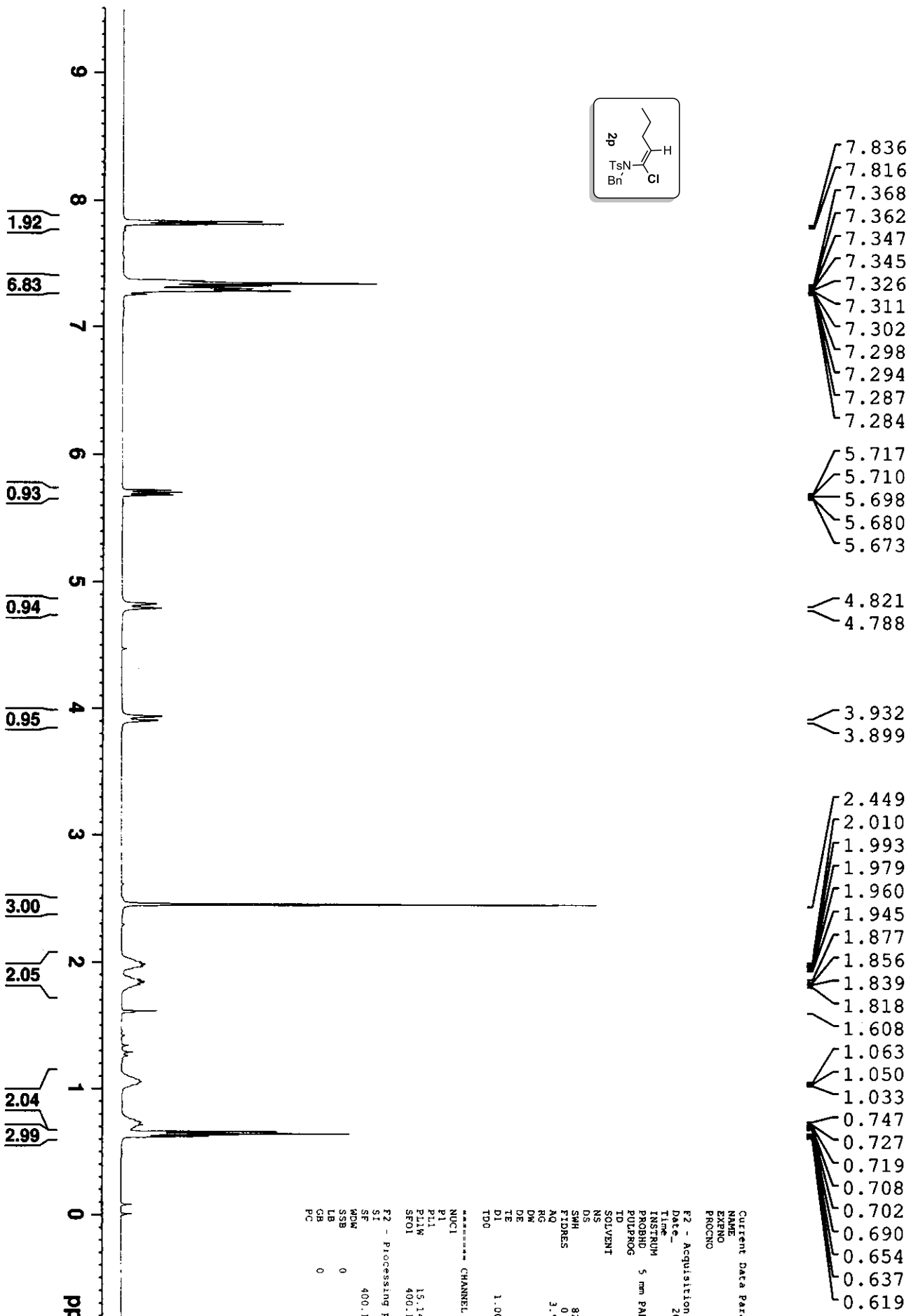
2n

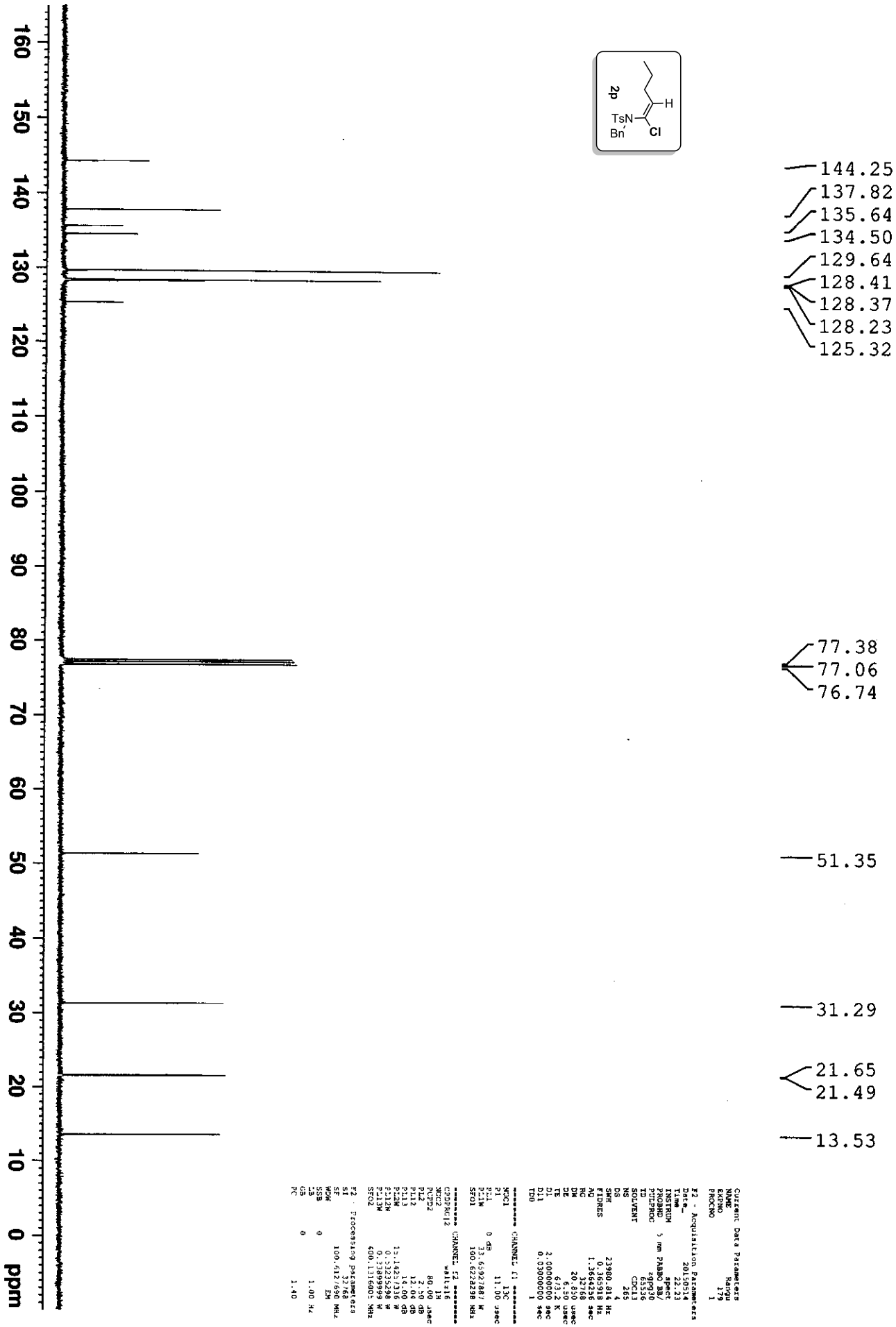
-112.53

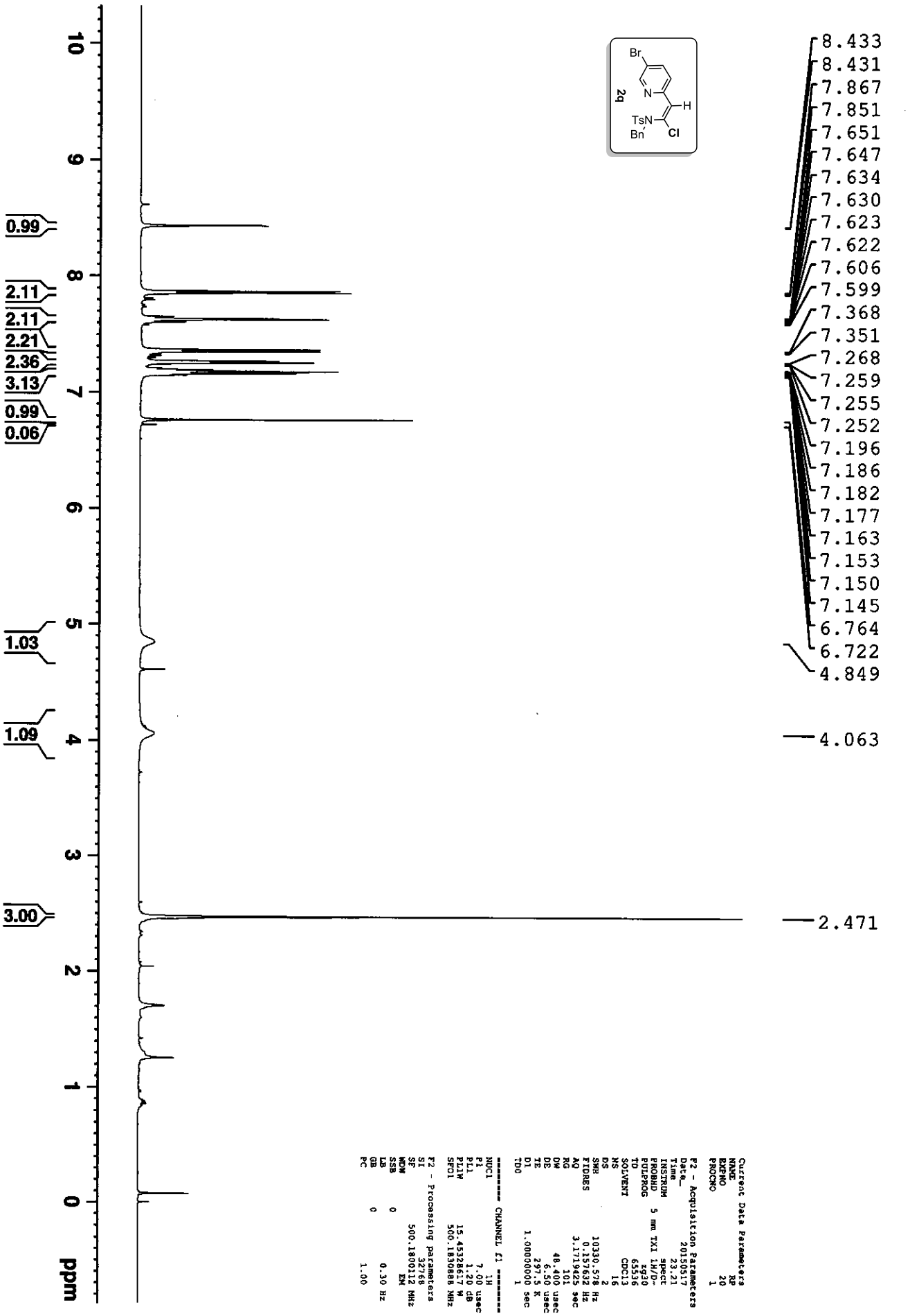
```
Current Data Parameters
NAME                  RKM
EXPNO                  25
PROCNO               1891

F2 - Acquisition Parameters
Date_              20150515
Time                 15.41
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG          zgfhigqn
TD                 131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH                89285.711 Hz
FIDRES             0.681196 Hz
AQ                 0.7340032 sec
RG                      2050
DW                     5.600 usec
DE                      6.50 usec
TE                     299.0 K
D1                1.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     19F
P1                     14.80 usec
PL1                    -1.20 dB
PL1W            18.11287308 W
SFO1            376.4240234 MHz

======== CHANNEL f2 ========
CPDPRG12              waltz16
NUC2                     1H
PCPD2                  90.00 usec
PL2                     1.50 dB
PL12                   17.18 dB
PL2W            15.18650627 W
PL12W            0.41063678 W
SFO2            400.0926004 MHz

F2 - Processing parameters
SI                      65536
SF              376.4616700 MHz
WDW                        EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.40
```

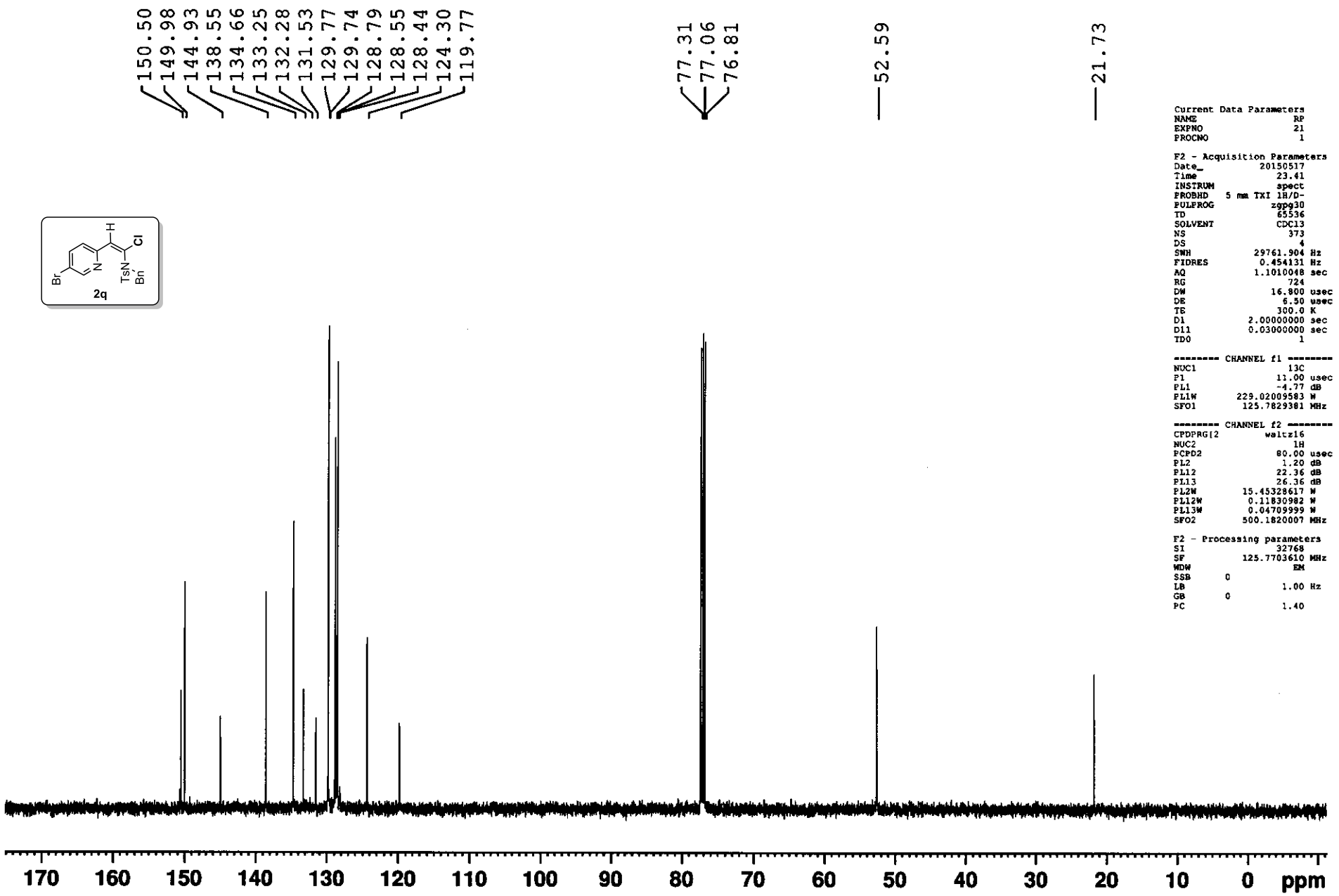

7.405
7.401
7.387
7.385
7.347
7.341
7.329
7.312
7.308
7.293
7.283
7.279
7.276
7.269
7.262
7.254
7.245
7.222
7.219
7.216
7.201
7.186
7.183
6.681
3.91
3.88
1.24
1.24
0.92
0.37
9.00
1.564
1.336
2o
PhN
Boc
H
Cl
ppm

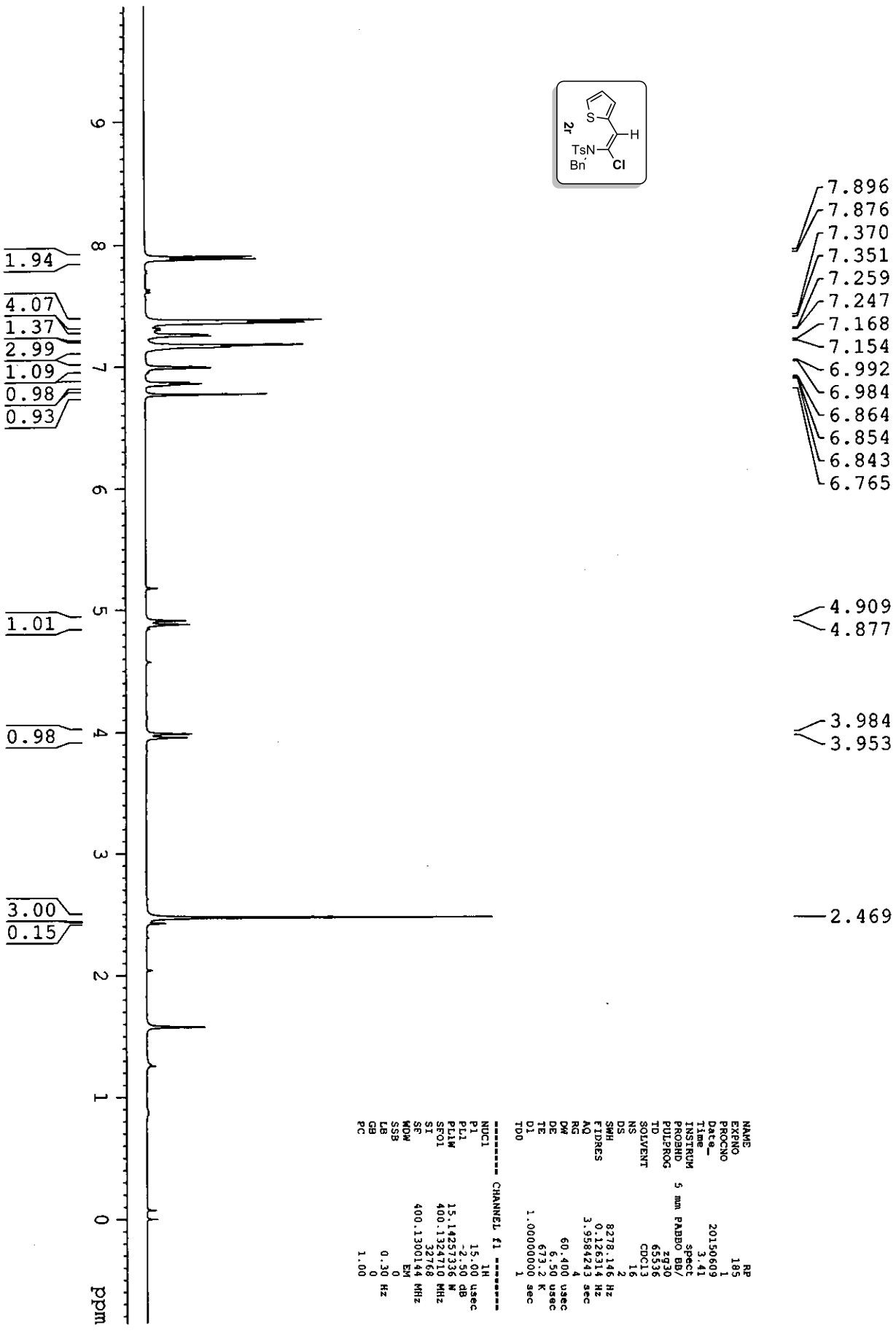

151.78
138.93
133.84
130.34
128.90
128.86
128.71
128.48
127.62
126.27
124.44
82.56
77.37
77.25
77.05
76.73
27.87
2o
NAME PB
EXPNO 374
PROCNO 1
Date_ 20150417
Time 11.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 973
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
PL1W 33.65927887 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.50 dB
PL12 12.04 dB
PL13 14.00 dB
PL2W 15.14257336 W
PL12W 0.53235298 W
PL13W 0.33899999 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

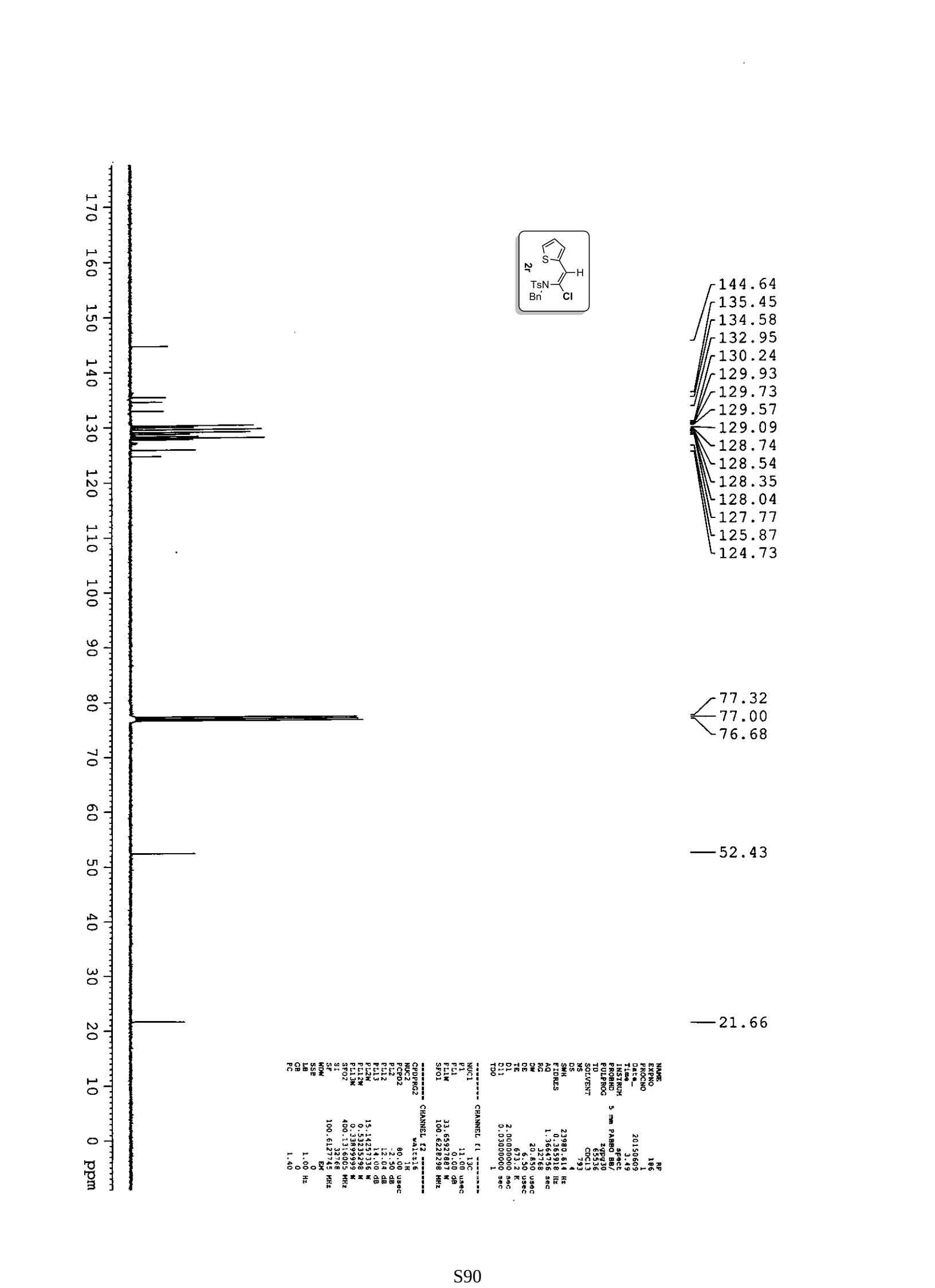

2p
TsN
Bn
H
Cl
7.836
7.816
7.368
7.362
7.347
7.345
7.326
7.311
7.302
7.298
7.294
7.287
7.284
5.717
5.710
5.698
5.680
5.673
4.821
4.788
3.932
3.899
2.449
2.010
1.993
1.979
1.960
1.945
1.877
1.856
1.839
1.818
1.608
1.063
1.050
1.033
0.747
0.727
0.719
0.708
0.702
0.690
0.654
0.637
0.619
1.92
6.83
0.93
0.94
0.95
3.00
2.05
2.04
2.99
ppm
Current Data Parameters
NAME Rangu
EXPNO 178
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150514
Time 22.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PLW 15.14257336 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300155 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

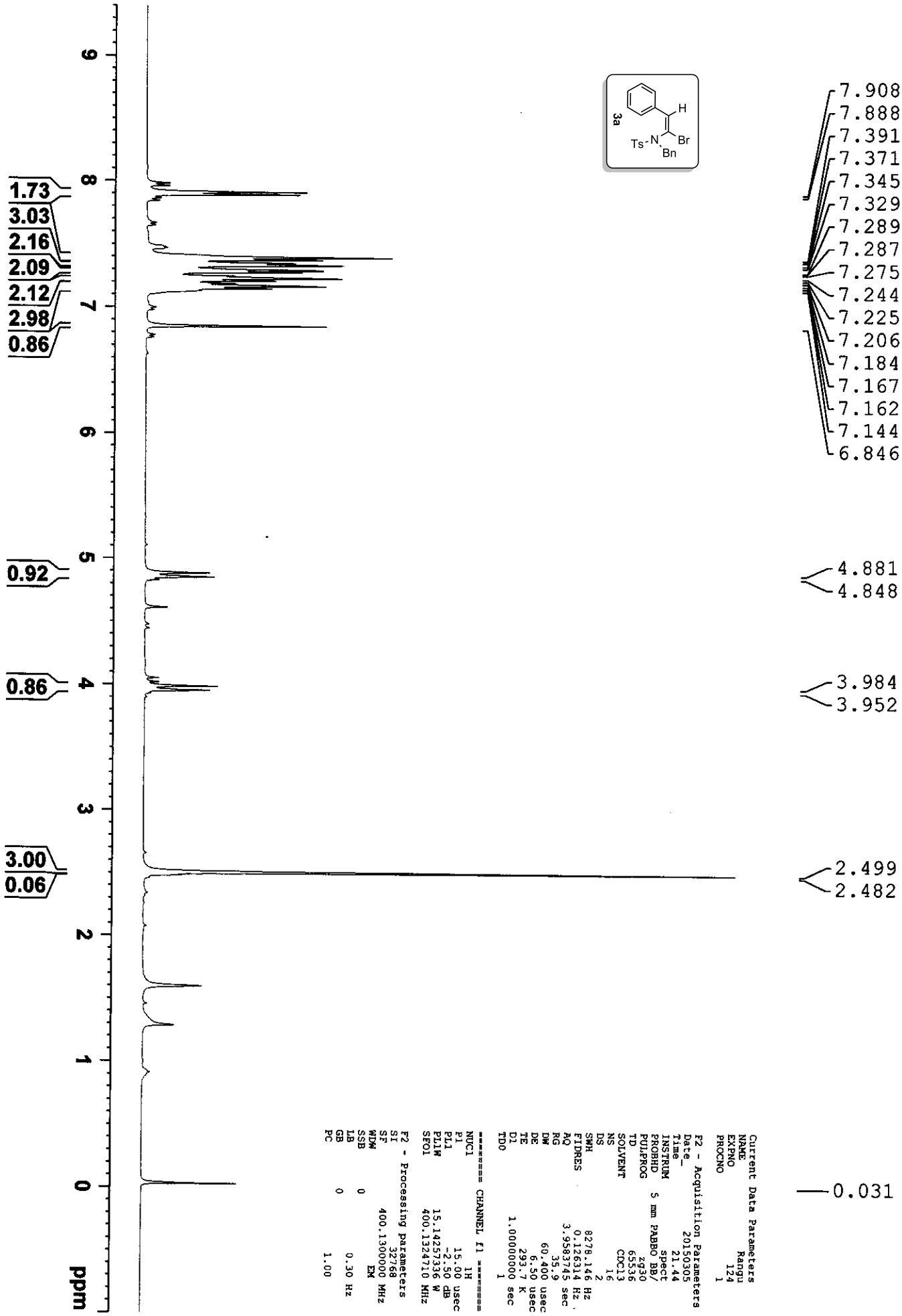

2p
TsN
Bn
H
Cl
144.25
137.82
135.64
134.50
129.64
128.41
128.37
128.23
125.32
77.38
77.06
76.74
51.35
31.29
21.65
21.49
13.53
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

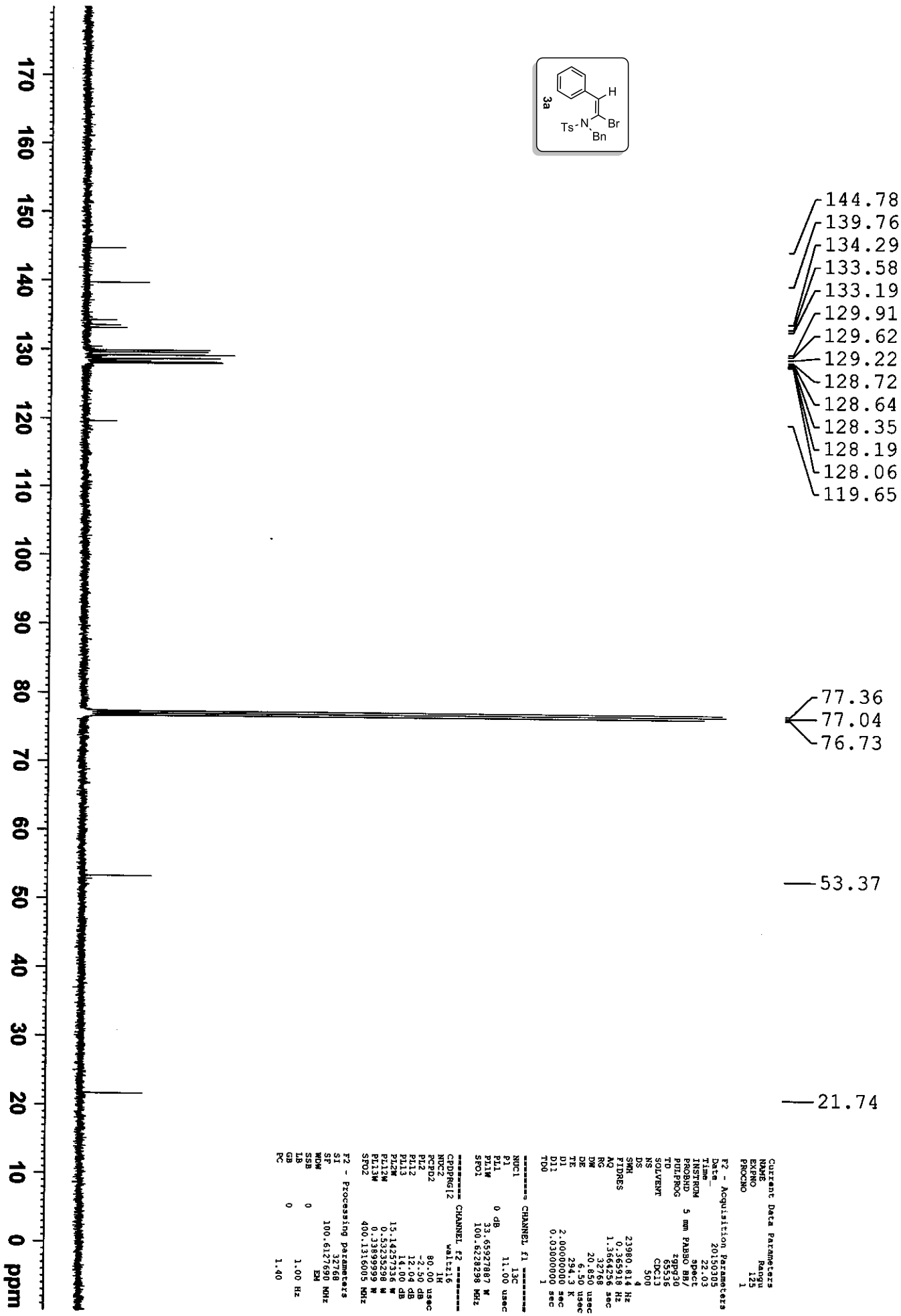

Br
N
H
TsN
Bn
Cl
2q
8.433
8.431
7.867
7.851
7.651
7.647
7.634
7.630
7.623
7.622
7.606
7.599
7.368
7.351
7.268
7.259
7.255
7.252
7.196
7.186
7.182
7.177
7.163
7.153
7.150
7.145
6.764
6.722
4.849
4.063
2.471
0.99
2.11
2.11
2.21
2.36
3.13
0.99
0.06
1.03
1.09
3.00
10
9
8
7
6
5
4
3
2
1
0
ppm
Current Data Parameters
NAME RP
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150517
Time 23.21
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 101
DW 48.400 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 1.20 dB
PL1W 15.45328617 W
SFO1 500.1830888 MHz
F2 - Processing parameters
SI 32768
SF 500.1800112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


150.50
149.98
144.93
138.55
134.66
133.25
132.28
131.53
129.77
129.74
128.79
128.55
128.44
124.30
119.77
77.31
77.06
76.81
52.59
21.73
Br
N
H
Cl
TsN
Bn
2q
Current Data Parameters
NAME RP
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150517
Time 23.41
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 373
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 724
DW 16.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 -4.77 dB
PL1W 229.02009583 W
SFO1 125.7829381 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.20 dB
PL12 22.36 dB
PL13 26.36 dB
PL2W 15.45328617 W
PL12W 0.11830982 W
PL13W 0.04709999 W
SFO2 500.1820007 MHz
F2 - Processing parameters
SI 32768
SF 125.7703610 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


2r
TsN
Bn
S
H
Cl
7.896
7.876
7.370
7.351
7.259
7.247
7.168
7.154
6.992
6.984
6.864
6.854
6.843
6.765
4.909
4.877
3.984
3.953
2.469
1.94
4.07
1.37
2.99
1.09
0.98
0.93
1.01
0.98
3.00
0.15
ppm


144.64
135.45
134.58
132.95
130.24
129.93
129.73
129.57
129.09
128.74
128.54
128.35
128.04
127.77
125.87
124.73
77.32
77.00
76.68
52.43
21.66
2f
TsN
Bn
Cl
H
S
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


3a
H
Br
Ts-N
Bn
7.908
7.888
7.391
7.371
7.345
7.329
7.289
7.287
7.275
7.244
7.225
7.206
7.184
7.167
7.162
7.144
6.846
4.881
4.848
3.984
3.952
2.499
2.482
0.031
1.73
3.03
2.16
2.09
2.12
2.98
0.86
0.92
0.86
3.00
0.06
ppm
Current Data Parameters
NAME Rangu
EXPNO 124
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150305
Time 21.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 35.9
DW 60.400 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PLIW 15.14257336 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



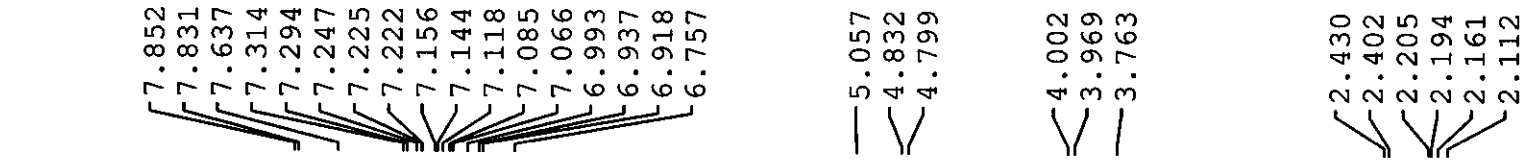
Current Data Parameters
NAME Rangu
EXPNO 125
PROCNO 1

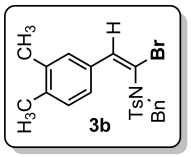
F2 - Acquisition Parameters
Date_ 20150305
Time_ 22.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 655336
SOLVENT CDCl3
NS 500
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 294.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0 dB
PL1W 33.65927887 W
SFO1 100.6228298 MHz

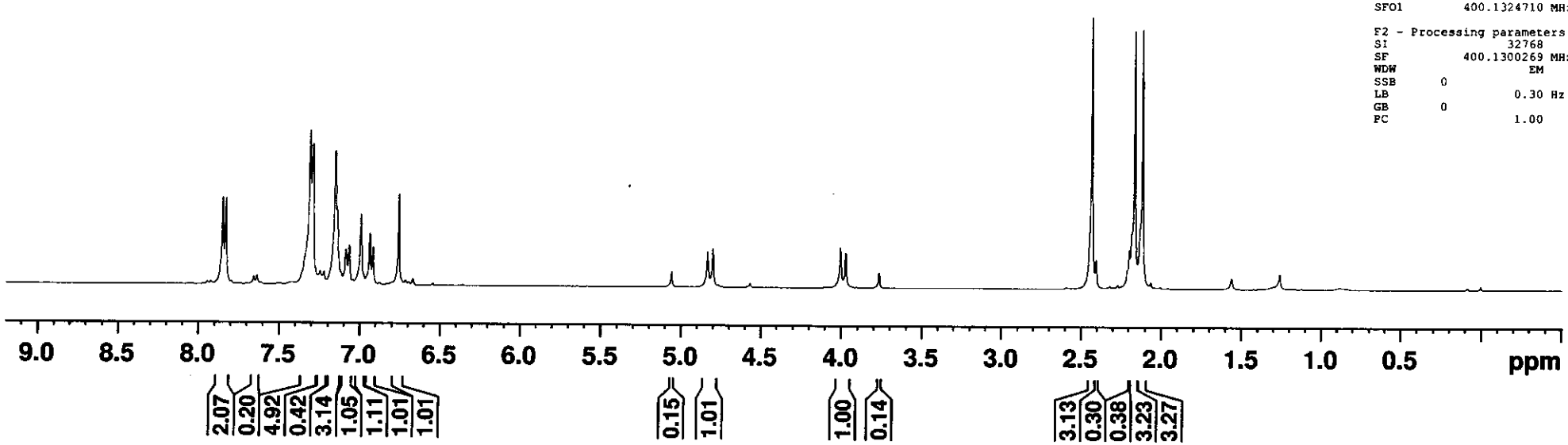
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.04 dB
PL13 14.04 dB
PL2W 15.14257336 W
PL12W 0.53233398 W
PL13W 0.33699999 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

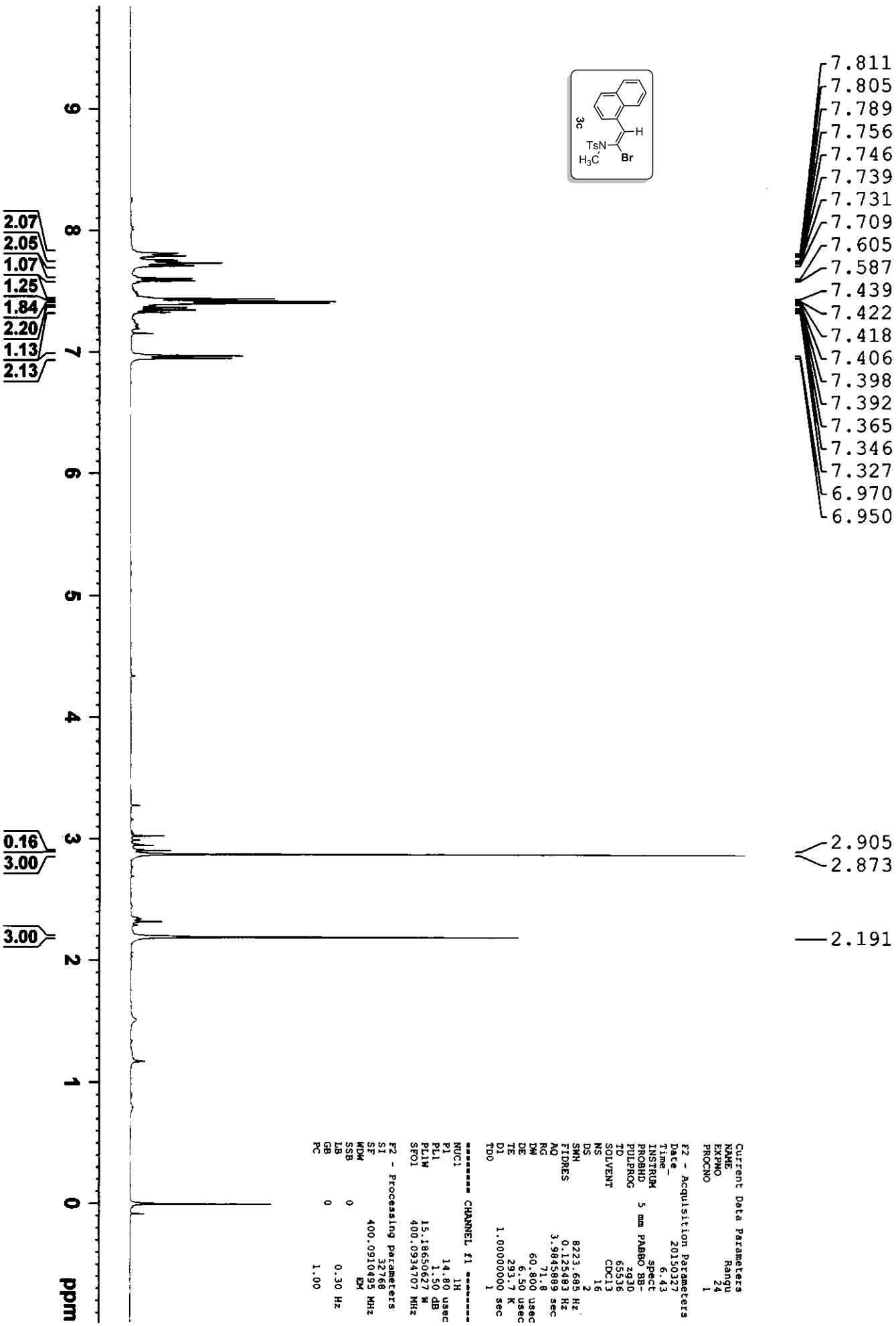


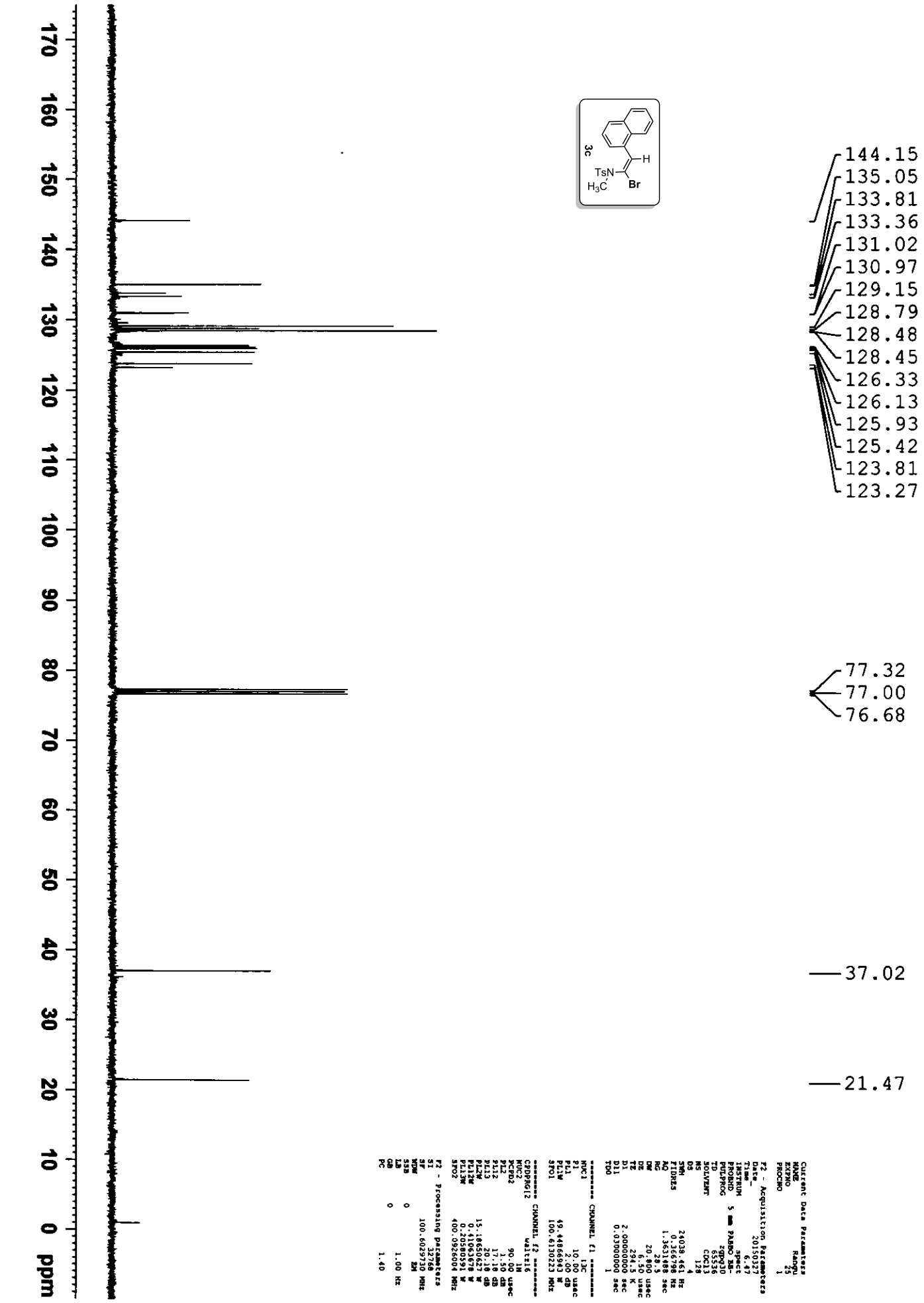


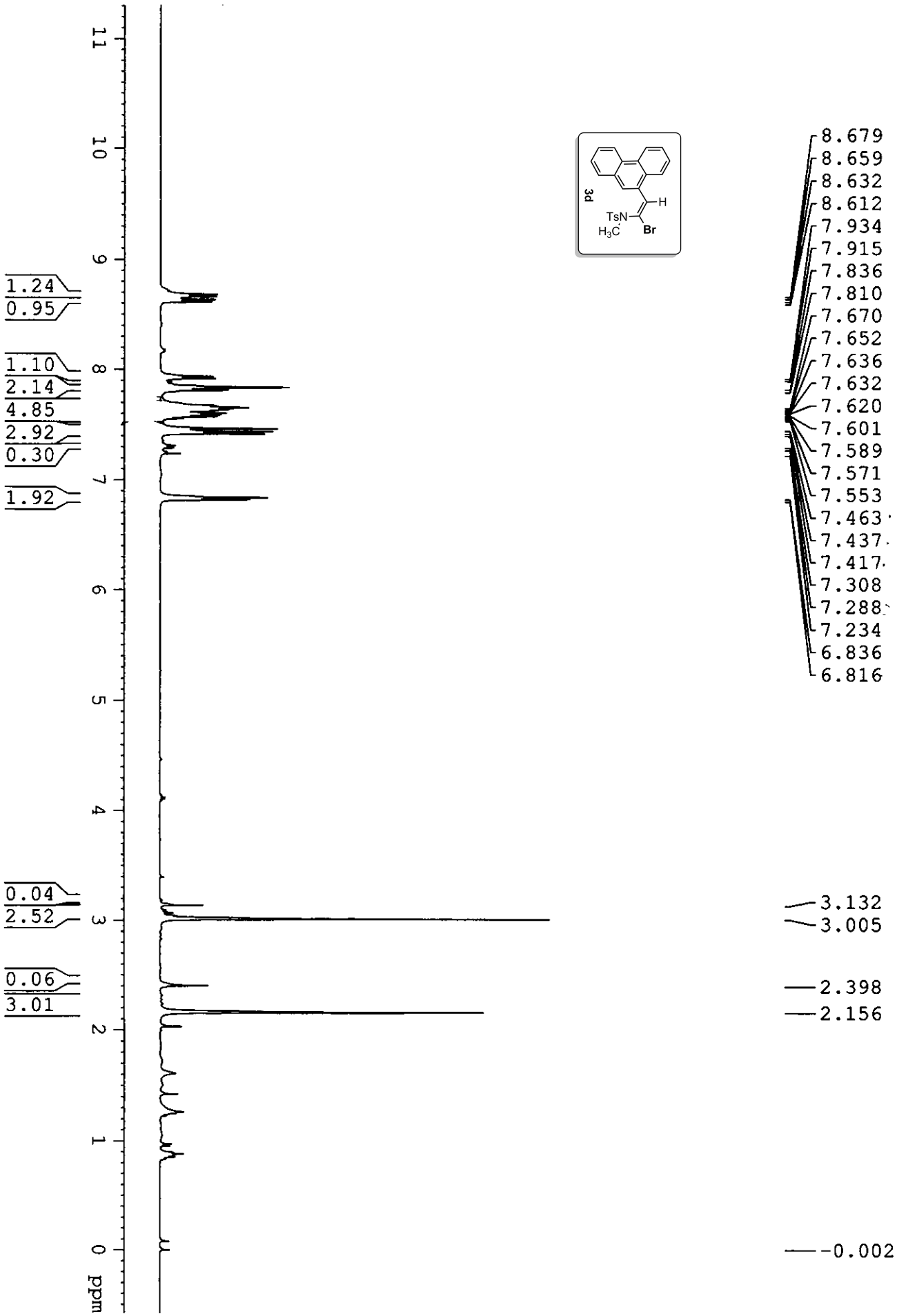

```
Current Data Parameters
NAME                  Rangu
EXPNO                   144
PROCNO                    1

F2 - Acquisition Parameters
Date_              20150326
Time                  21.22
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9583745 sec
RG                        4
DW                   60.400 usec
DE                    6.50 usec
TE                   673.2 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    15.00 usec
PL1                   -2.50 dB
PL1W           15.14257336 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                    32768
SF             400.1300269 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```

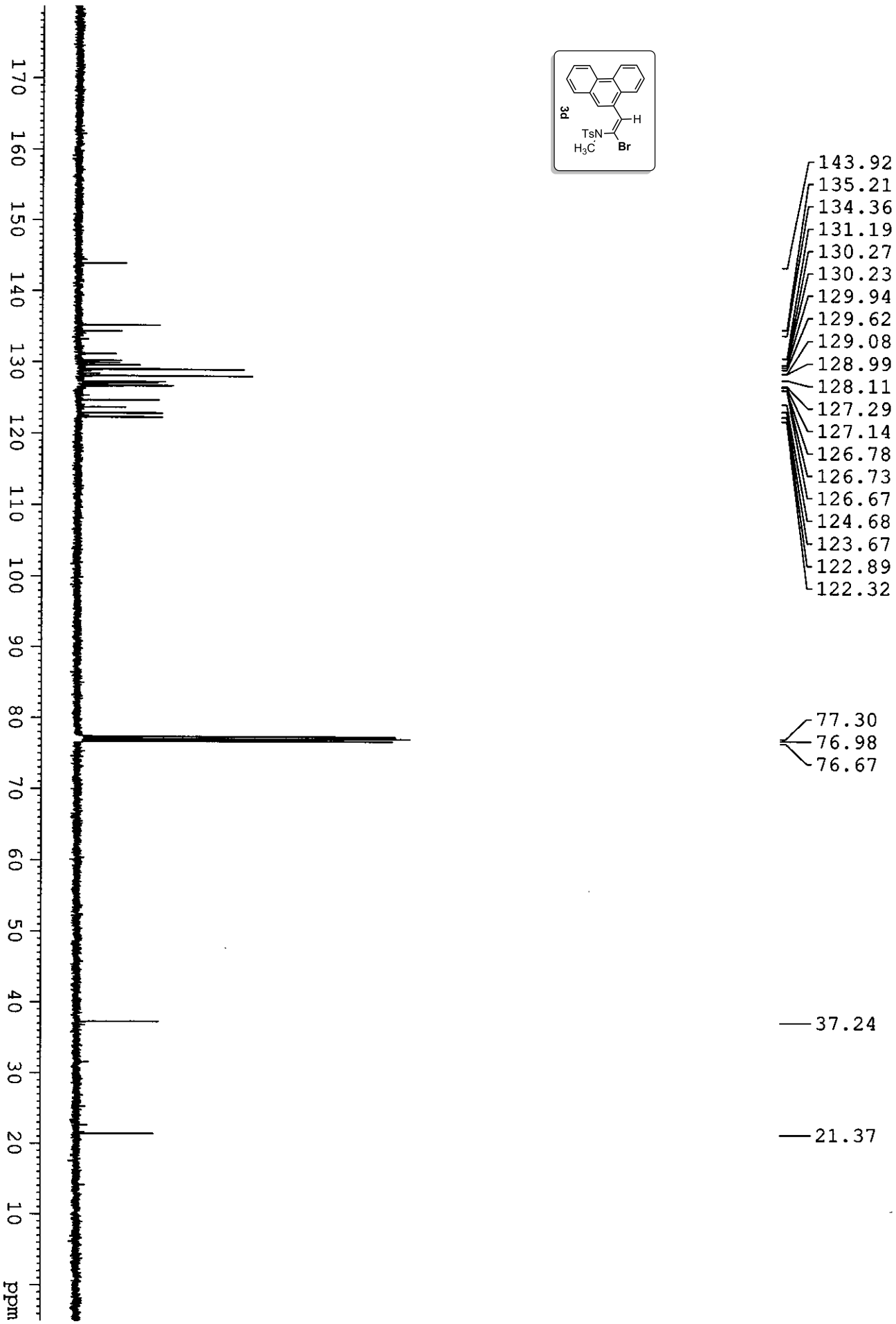


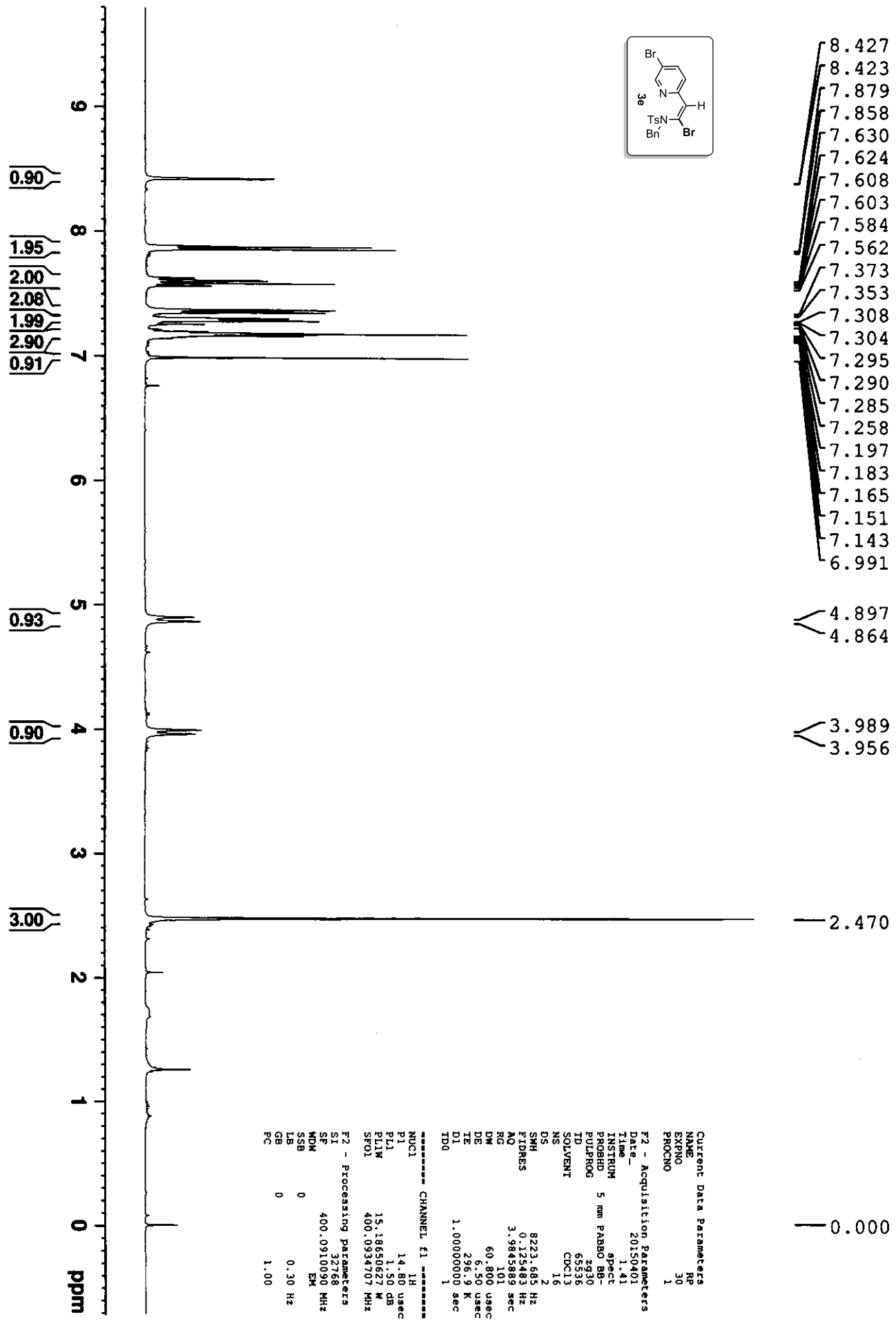
3b
144.63
139.70
137.45
136.04
134.69
133.59
131.28
130.00
129.83
129.59
129.38
129.12
128.32
128.28
126.33
118.66
77.43
77.12
76.80
53.32
21.71
19.67
19.61
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

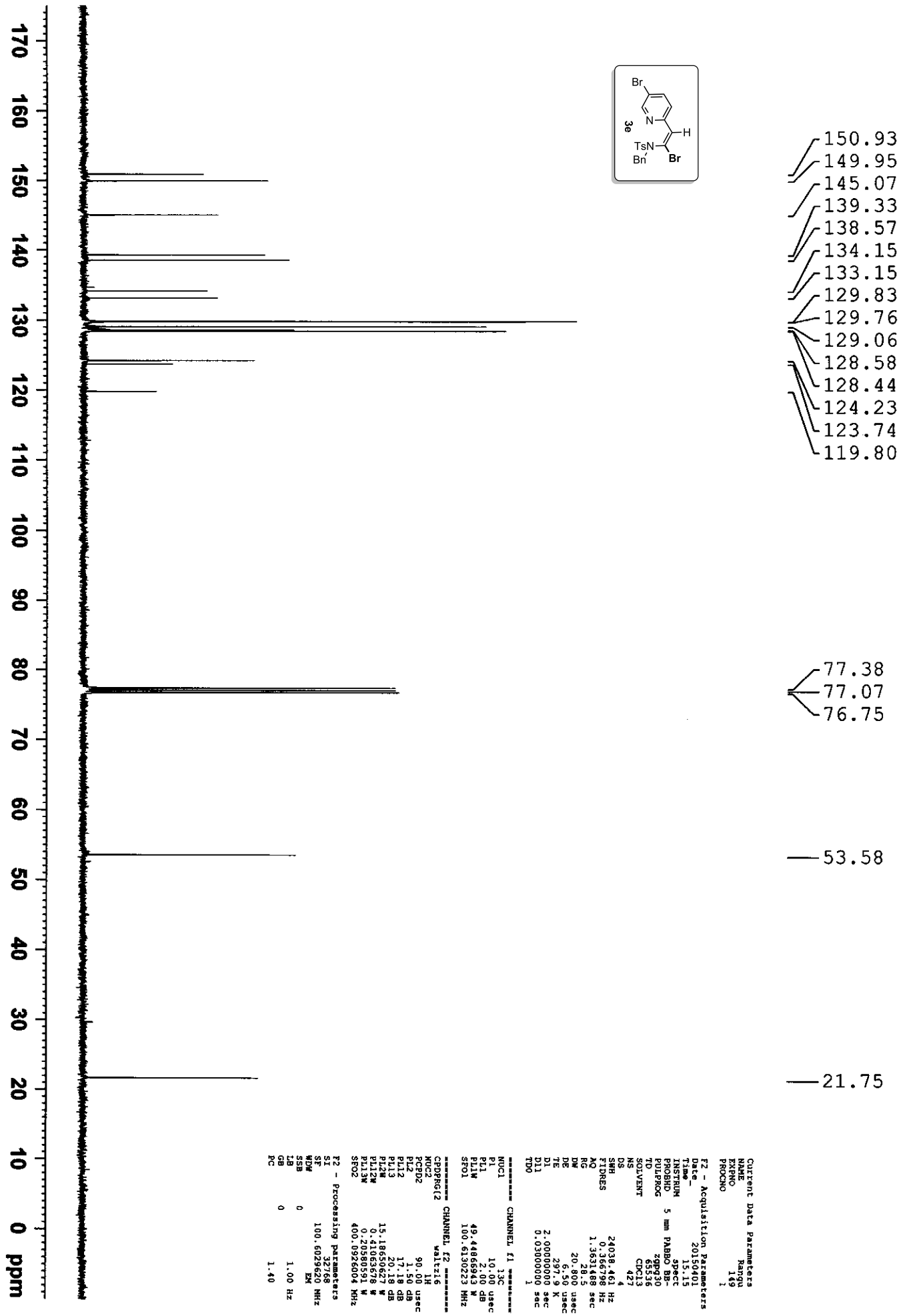

7.811
7.805
7.789
7.756
7.746
7.739
7.731
7.709
7.605
7.587
7.439
7.422
7.418
7.406
7.398
7.392
7.365
7.346
7.327
6.970
6.950
2.905
2.873
2.191
2.07
2.05
1.07
1.25
1.84
2.20
1.13
2.13
0.16
3.00
3.00
3c
TsN
H3C
H
Br
ppm
Current Data Parameters
NAME Rangu
EXPNO 24
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150327
Time 6.43
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 71.8
DW 60.800 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.80 usec
PL1 1.50 dB
PL1W 15.18650627 W
SFO1 400.0934707 MHz
F2 - Processing parameters
SI 32768
SF 400.0910495 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


3c
TsN
H3C
Br
H
144.15
135.05
133.81
133.36
131.02
130.97
129.15
128.79
128.48
128.45
126.33
126.13
125.93
125.42
123.81
123.27
77.32
77.00
76.68
37.02
21.47
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


3d
TsN
H3C
Br
H
8.679
8.659
8.632
8.612
7.934
7.915
7.836
7.810
7.670
7.652
7.636
7.632
7.620
7.601
7.589
7.571
7.553
7.463
7.437
7.417
7.308
7.288
7.234
6.836
6.816
3.132
3.005
2.398
2.156
-0.002
1.24
0.95
1.10
2.14
4.85
2.92
0.30
1.92
0.04
2.52
0.06
3.01
ppm


3d
TsN
H3C
Br
H
143.92
135.21
134.36
131.19
130.27
130.23
129.94
129.62
129.08
128.99
128.11
127.29
127.14
126.78
126.73
126.67
124.68
123.67
122.89
122.32
77.30
76.98
76.67
37.24
21.37
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.427
8.423
7.879
7.858
7.630
7.624
7.608
7.603
7.584
7.562
7.373
7.353
7.308
7.304
7.295
7.290
7.285
7.258
7.197
7.183
7.165
7.151
7.143
6.991
4.897
4.864
3.989
3.956
2.470
0.000
0.90
1.95
2.00
2.08
1.99
2.90
0.91
0.93
0.90
3.00
ppm
Br
N
H
TsN
Bn
Br
3e
Current Data Parameters
NAME RP
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150401
Time 1.41
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 101
DW 60.800 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.80 usec
PL1 1.50 dB
PL1W 15.18650627 W
SFO1 400.0934707 MHz
F2 - Processing parameters
SI 32768
SF 400.0910090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



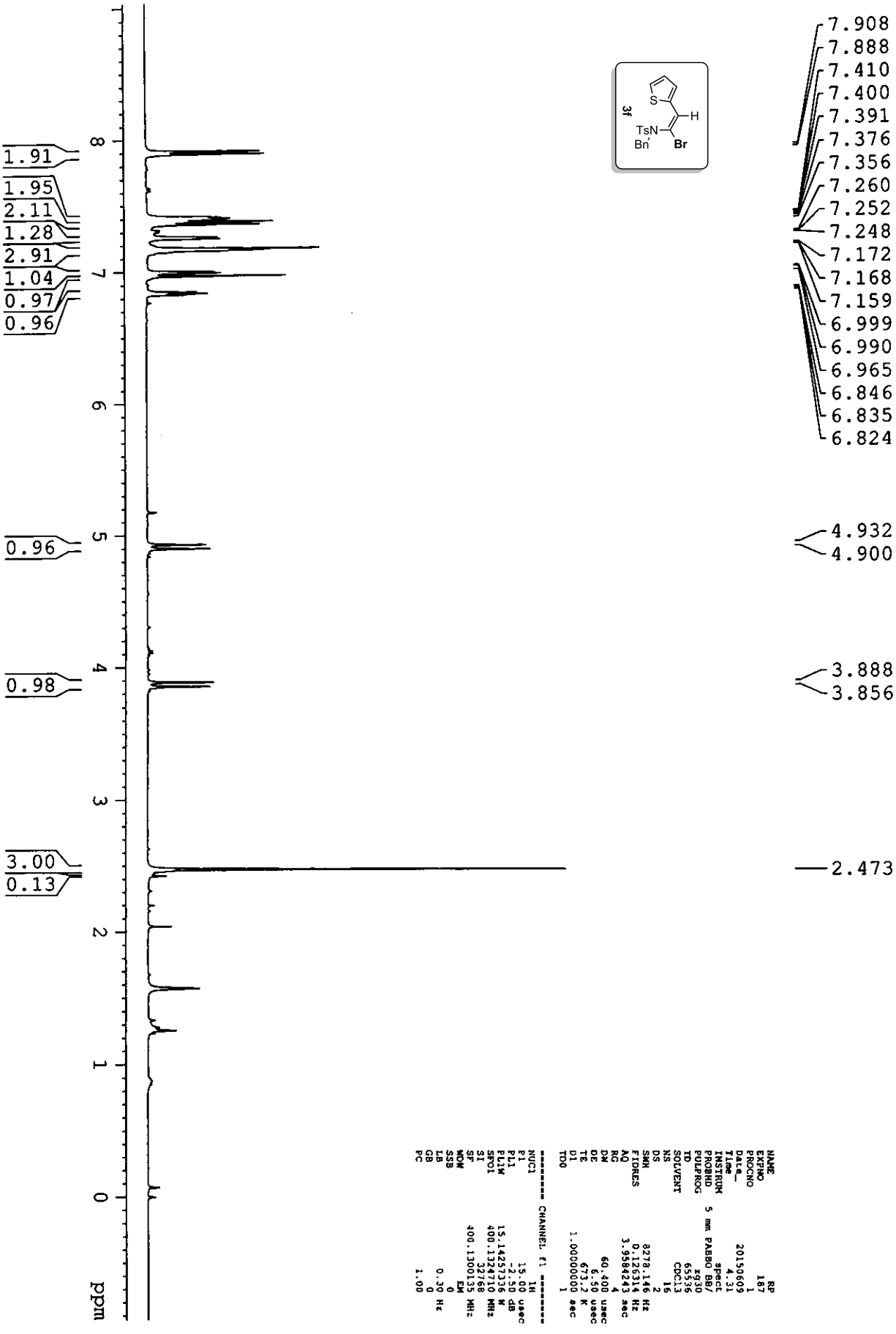
Current Data Parameters
NAME
EXPNO 149
PROCNO 1

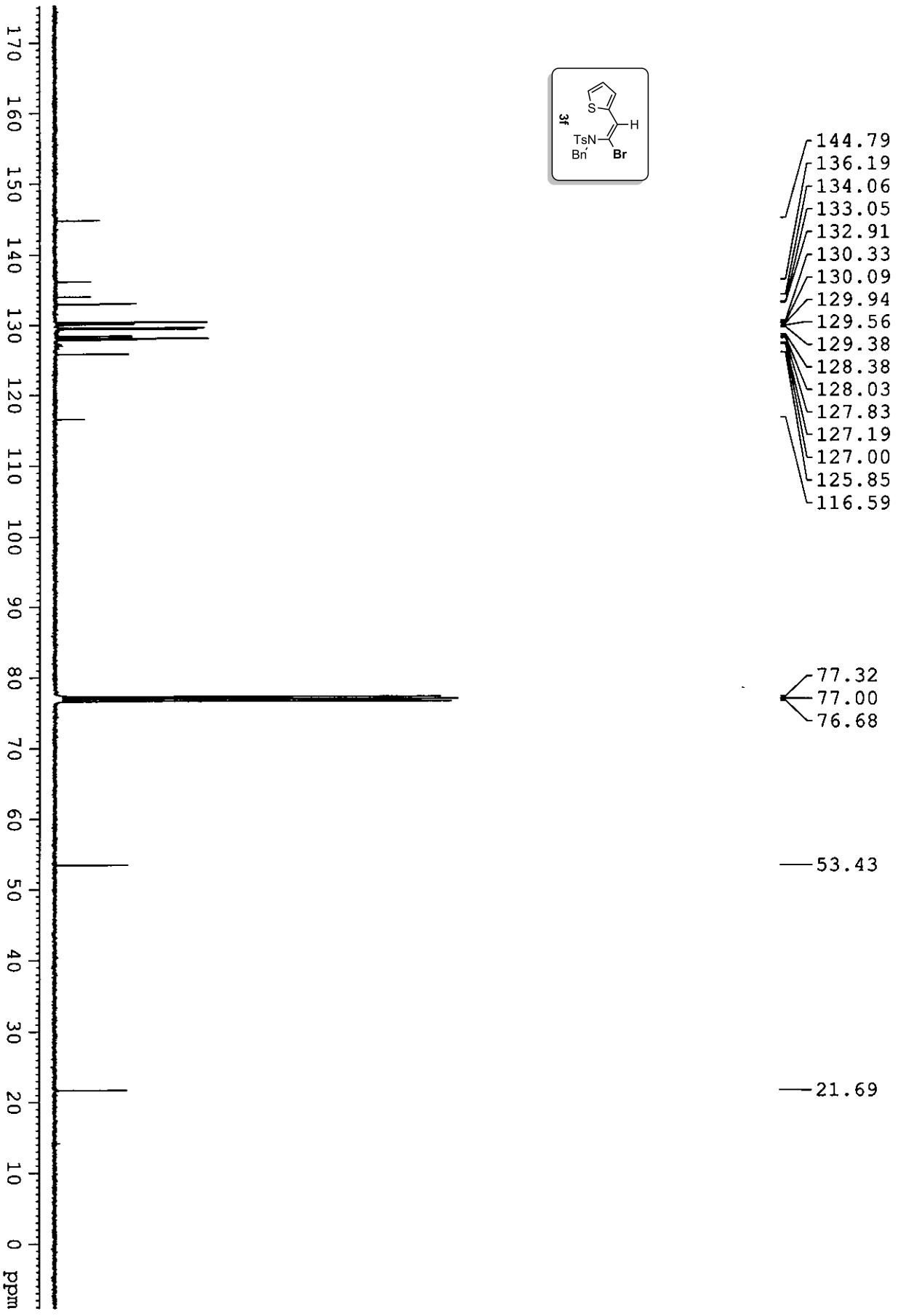
F2 - Acquisition Parameters
Date_ 20150401
Time 15.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 427
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 28.5
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

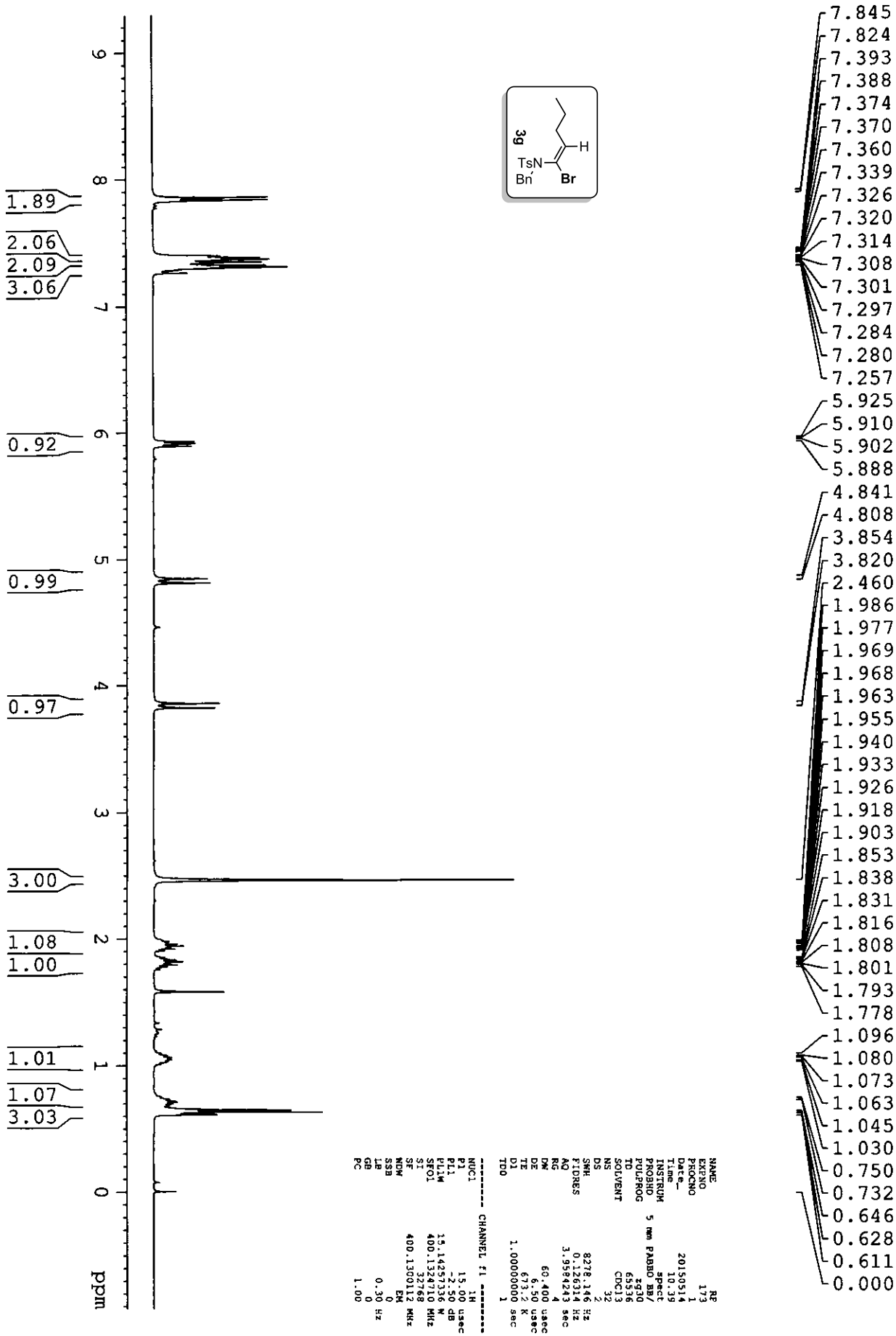
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.00 dB
PL1W 49.44866943 W
SFO1 100.6130223 MHz

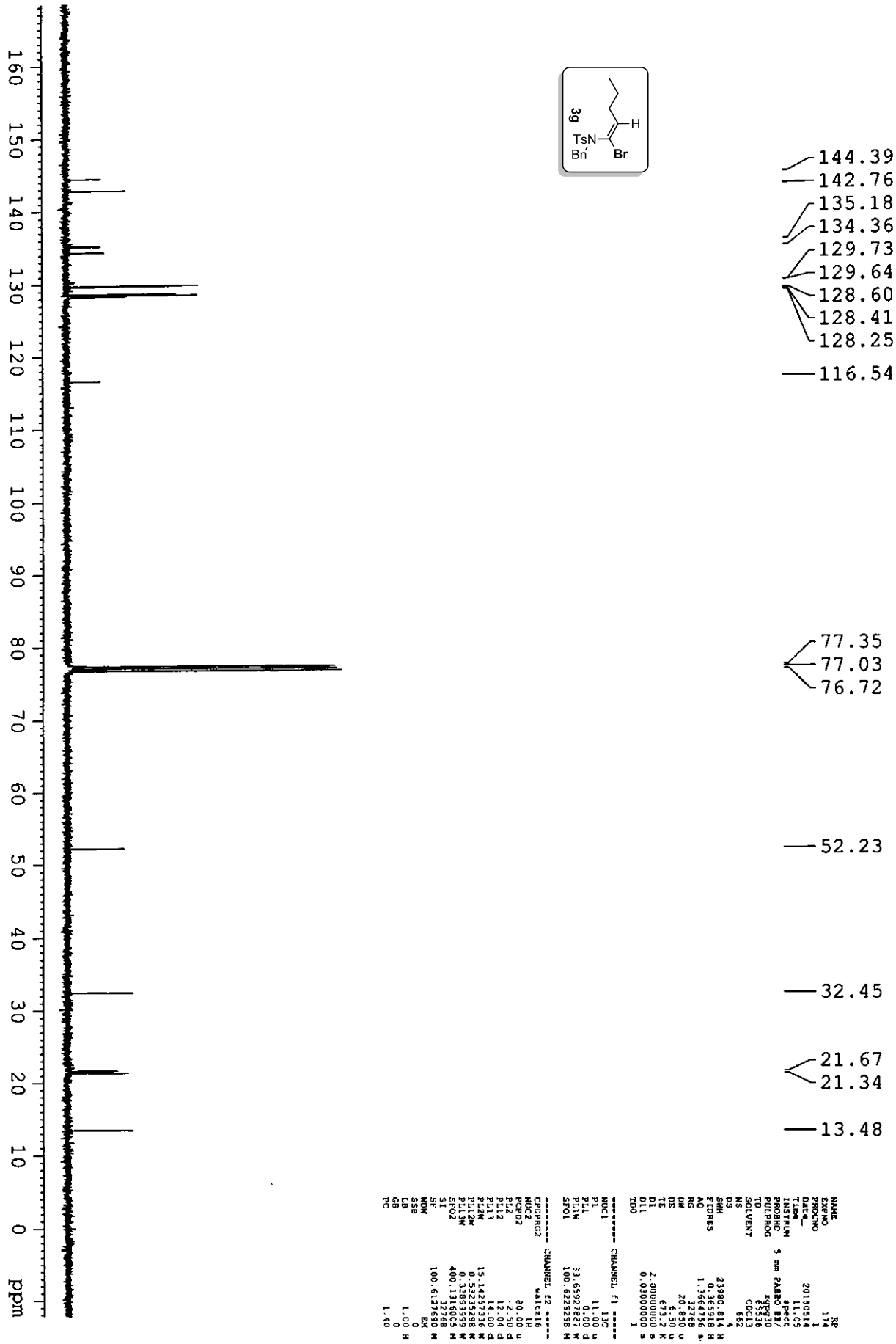
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.50 dB
PL12 17.18 dB
PL13 20.18 dB
PL2W 15.18650627 W
PL12W 0.41063678 W
PL13W 0.20580591 W
SFO2 400.0926004 MHz

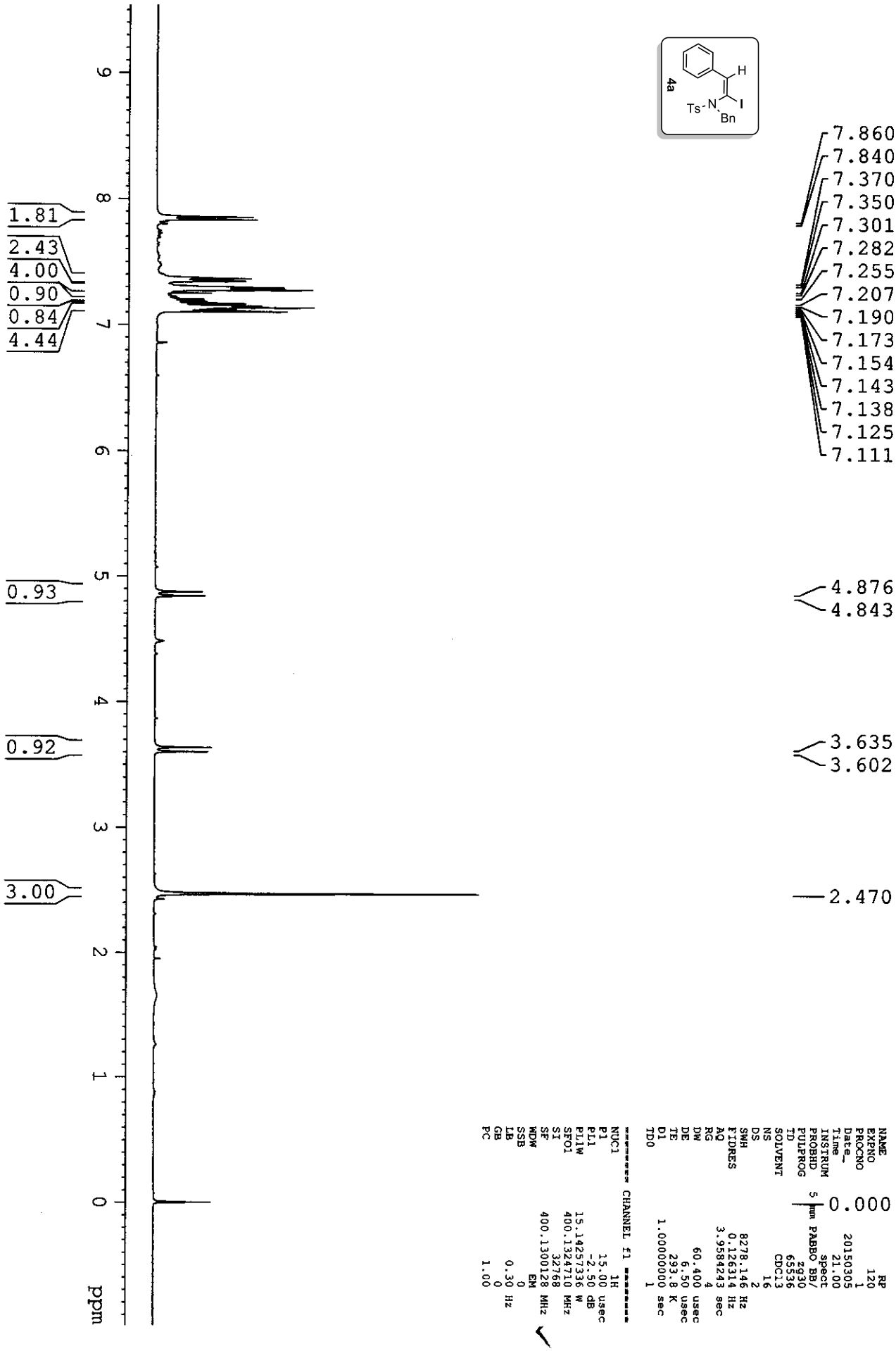
F2 - Processing parameters
SI 32768
SF 100.6029620 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

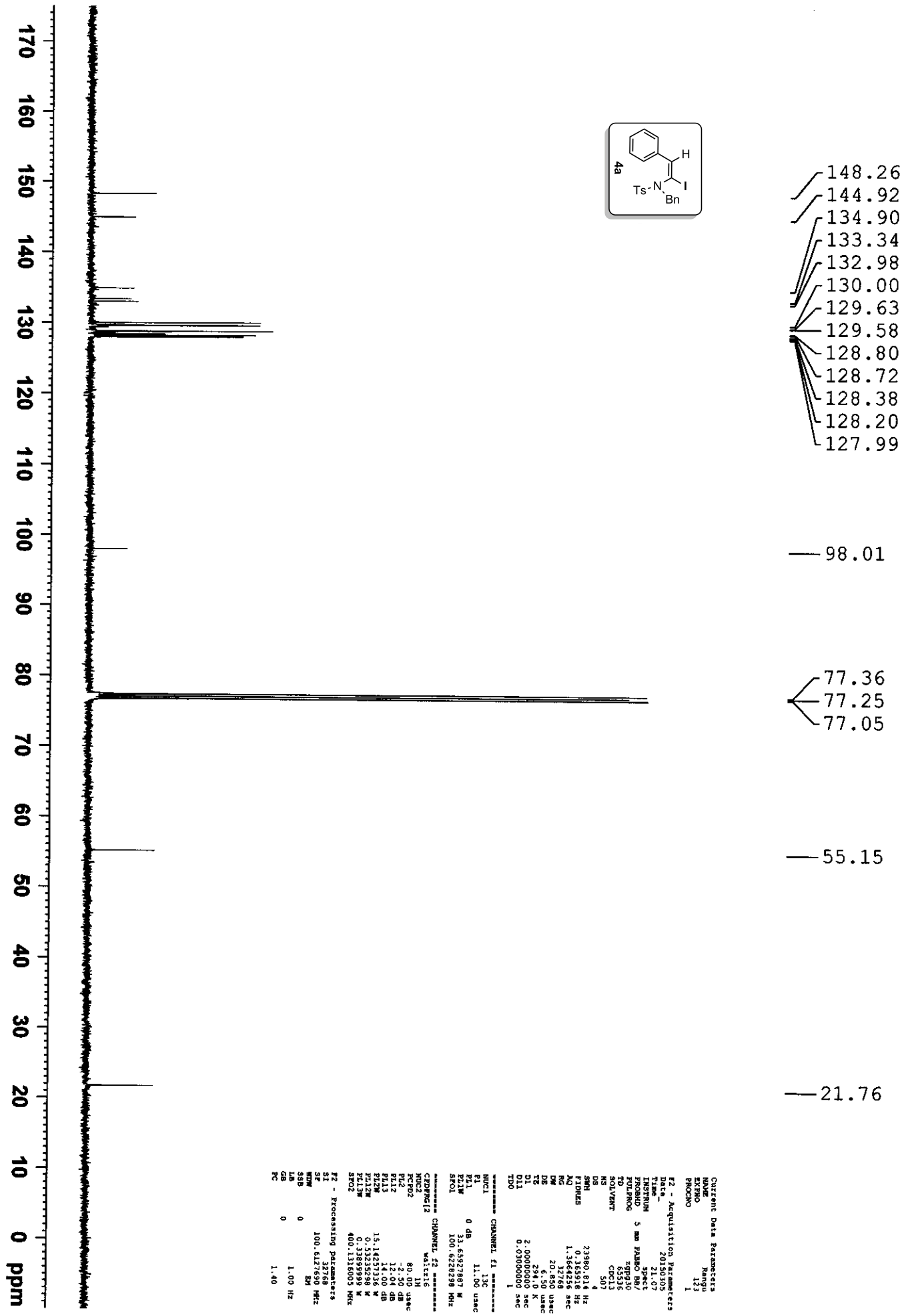

7.908
7.888
7.410
7.400
7.391
7.376
7.356
7.260
7.252
7.248
7.172
7.168
7.159
6.999
6.990
6.965
6.846
6.835
6.824
4.932
4.900
3.888
3.856
2.473
1.91
1.95
2.11
1.28
2.91
1.04
0.97
0.96
0.96
0.98
3.00
0.13
3f
S
H
TsN
Bn
Br
ppm

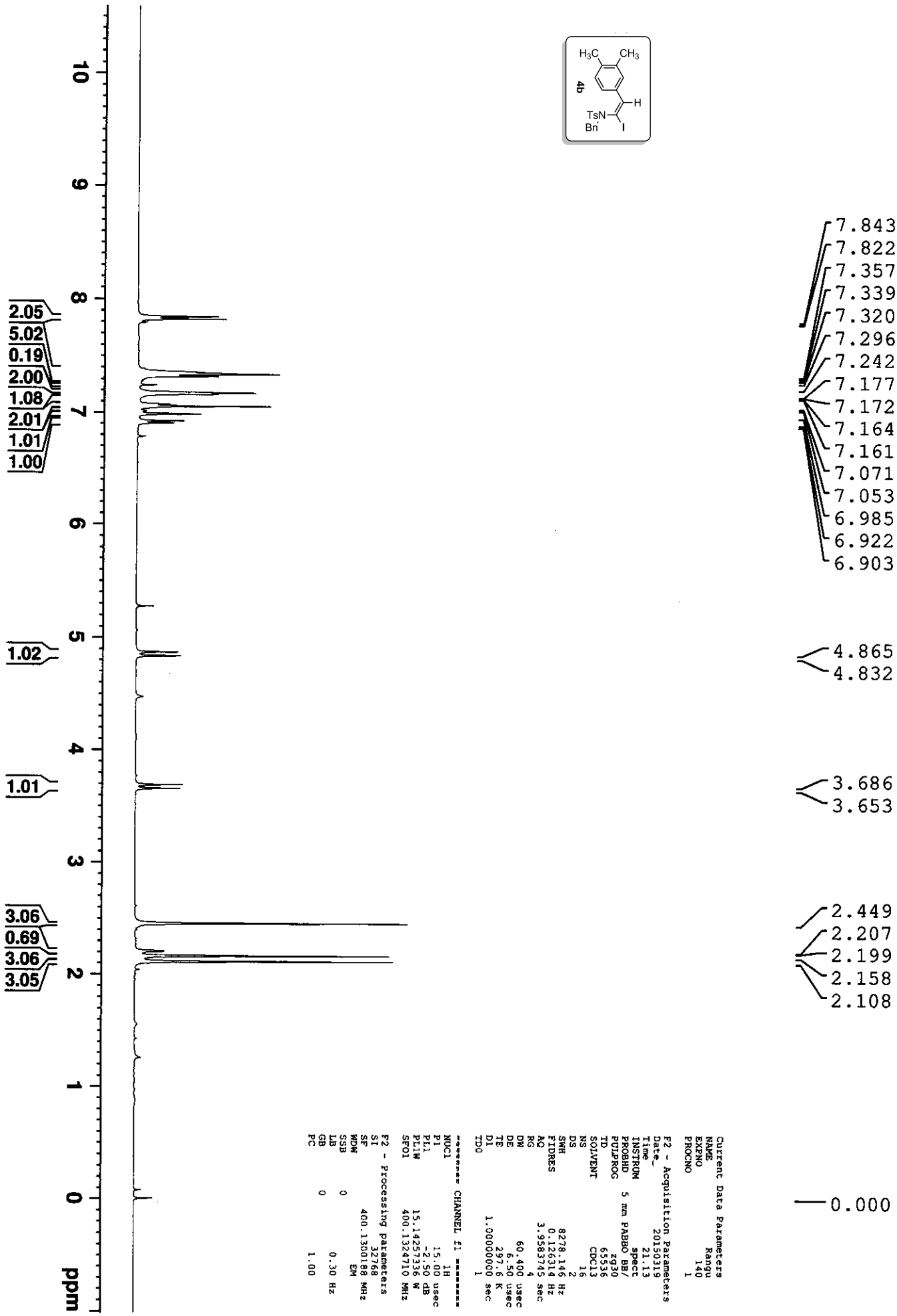

3f
TsN
Bn
Br
144.79
136.19
134.06
133.05
132.91
130.33
130.09
129.94
129.56
129.38
128.38
128.03
127.83
127.19
127.00
125.85
116.59
77.32
77.00
76.68
53.43
21.69
ppm

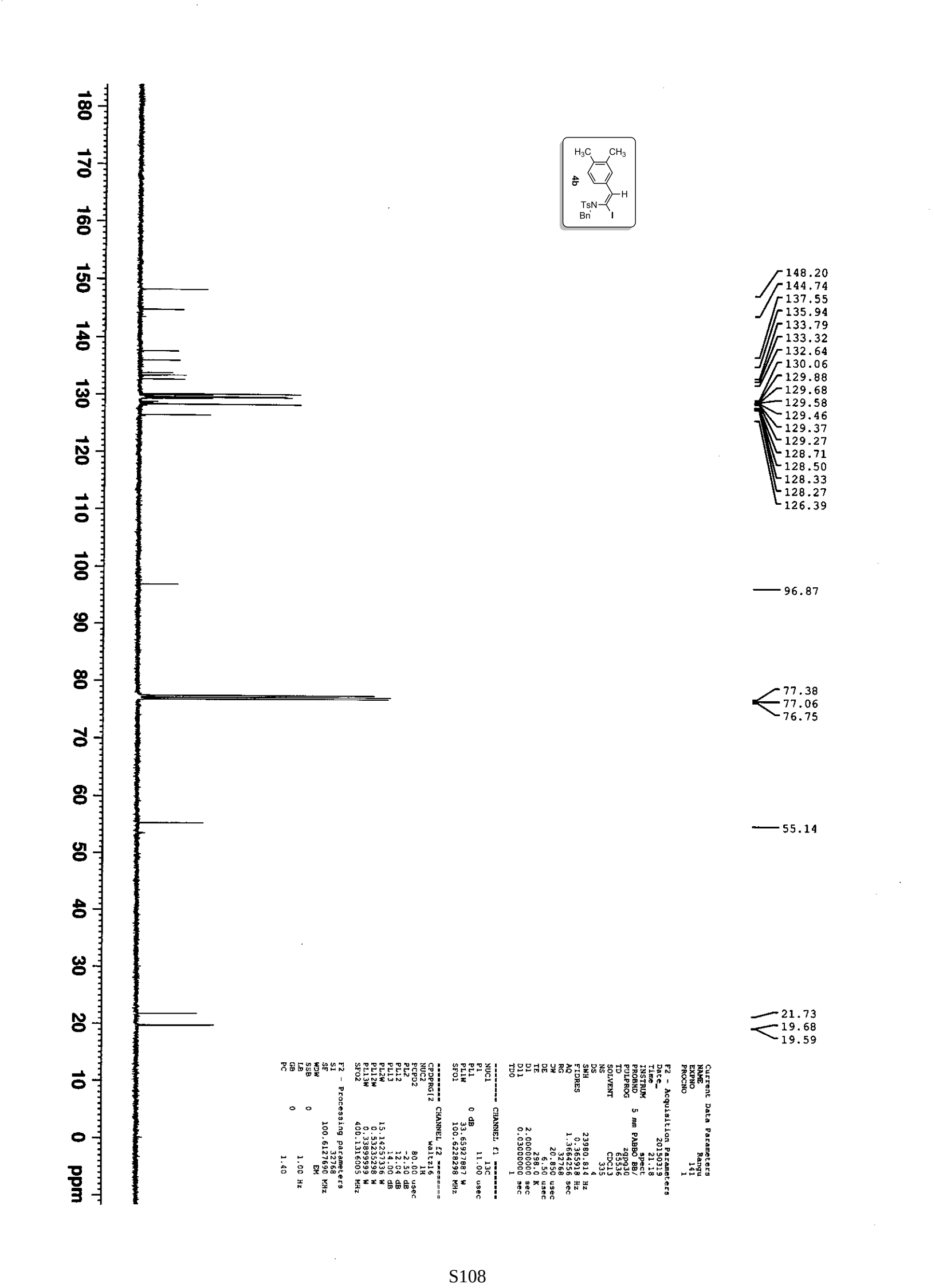

3g
TsN
Bn
H
Br
7.845
7.824
7.393
7.388
7.374
7.370
7.360
7.339
7.326
7.320
7.314
7.308
7.301
7.297
7.284
7.280
7.257
5.925
5.910
5.902
5.888
4.841
4.808
3.854
3.820
2.460
1.986
1.977
1.969
1.968
1.963
1.955
1.940
1.933
1.926
1.918
1.903
1.853
1.838
1.831
1.816
1.808
1.801
1.793
1.778
1.096
1.080
1.073
1.063
1.045
1.030
0.750
0.732
0.646
0.628
0.611
0.000
1.89
2.06
2.09
3.06
0.92
0.99
0.97
3.00
1.08
1.00
1.01
1.07
3.03
ppm
NAME RE
EXPNO 173
PROCNO 1
Date_ 20150514
Time 10.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
---------- CHANNEL f1 ----------
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PLW1 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

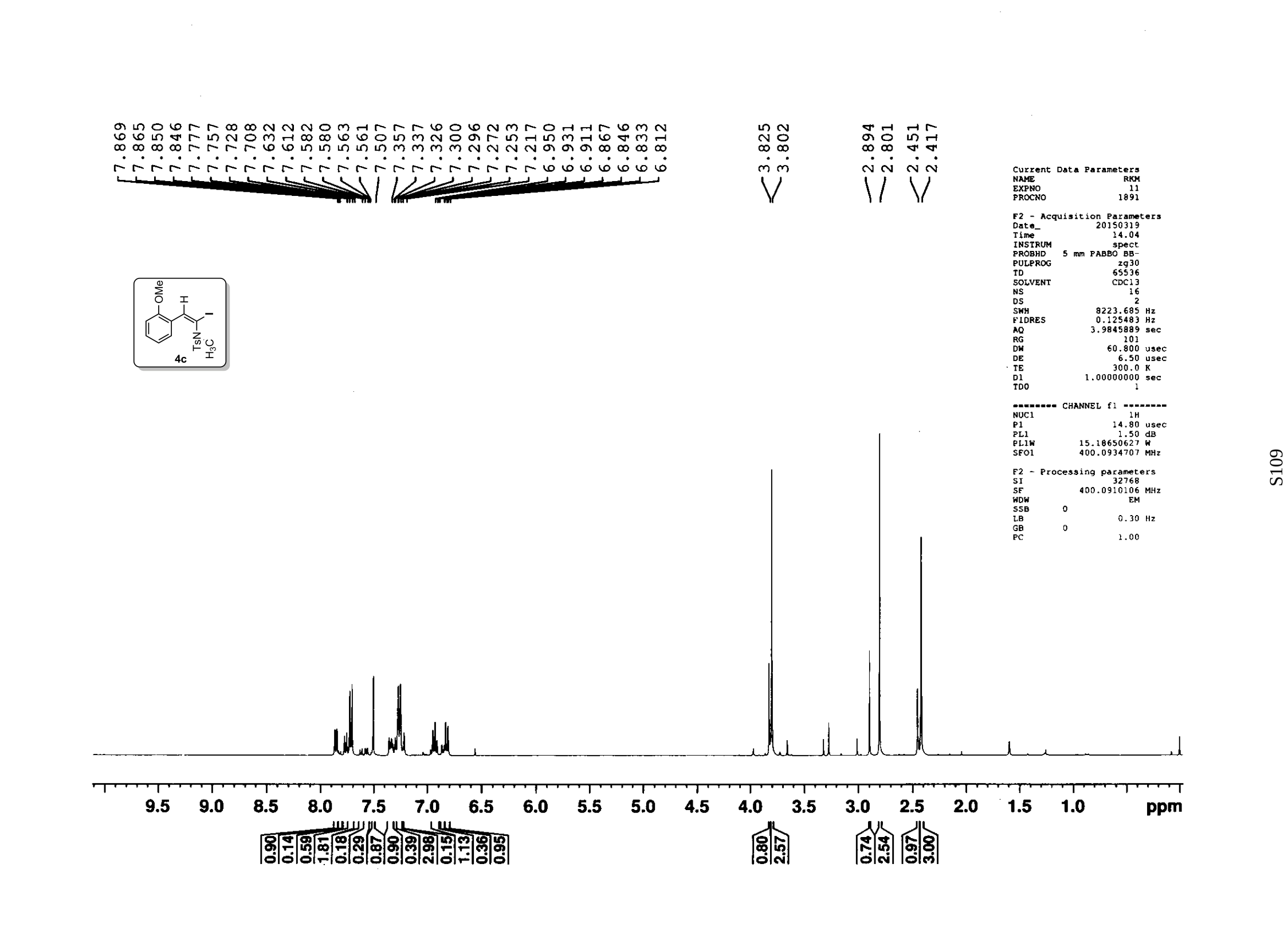

3g
TsN
Bn
H
Br
144.39
142.76
135.18
134.36
129.73
129.64
128.60
128.41
128.25
116.54
77.35
77.03
76.72
52.23
32.45
21.67
21.34
13.48
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

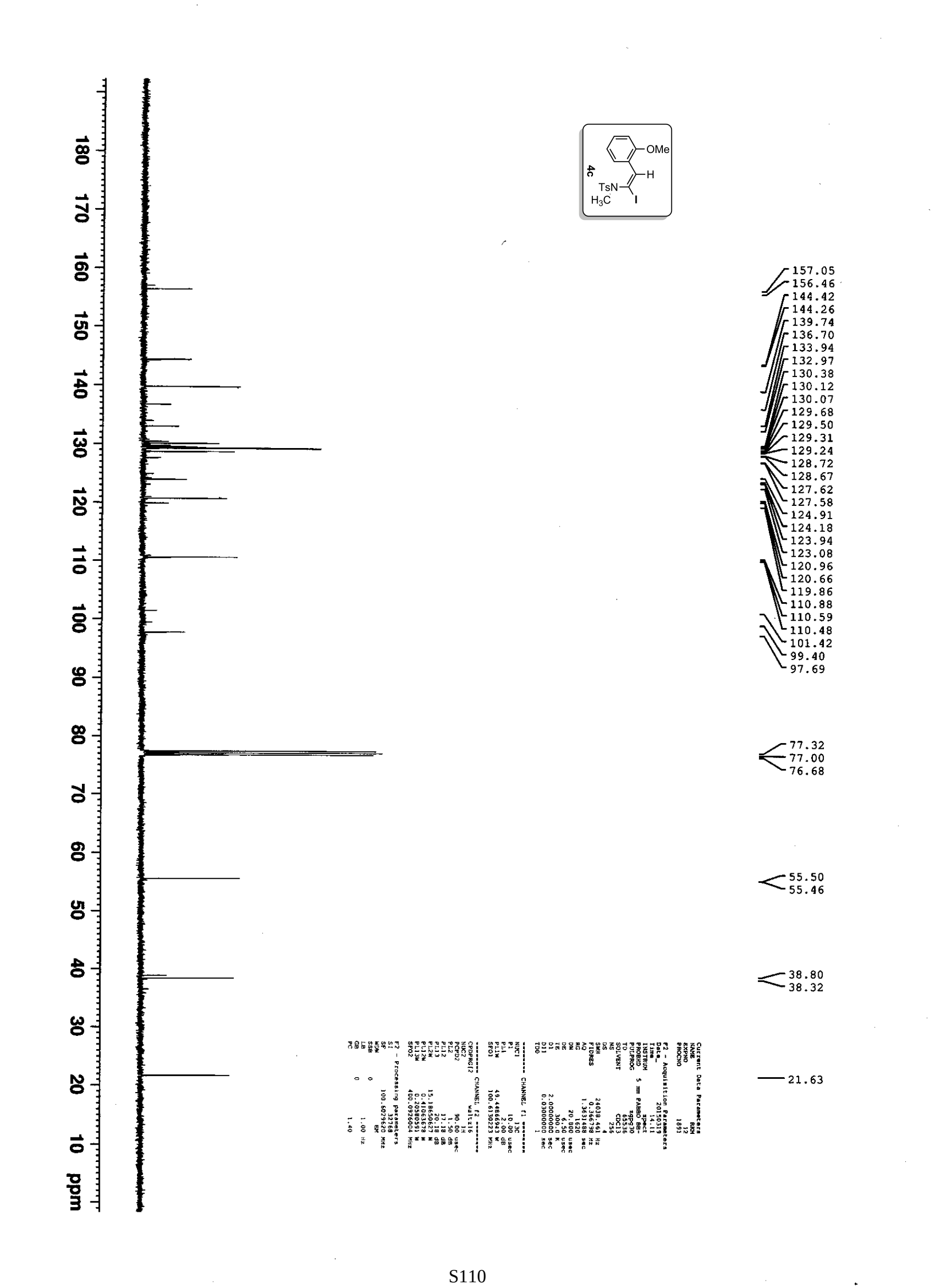

4a
Ts-N
Bn
H
I
7.860
7.840
7.370
7.350
7.301
7.282
7.255
7.207
7.190
7.173
7.154
7.143
7.138
7.125
7.111
4.876
4.843
3.635
3.602
2.470
0.000
1.81
2.43
4.00
0.90
0.84
4.44
0.93
0.92
3.00
ppm
NAME RP
EXPNO 120
PROCNO 1
Date_ 20150305
Time 21.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

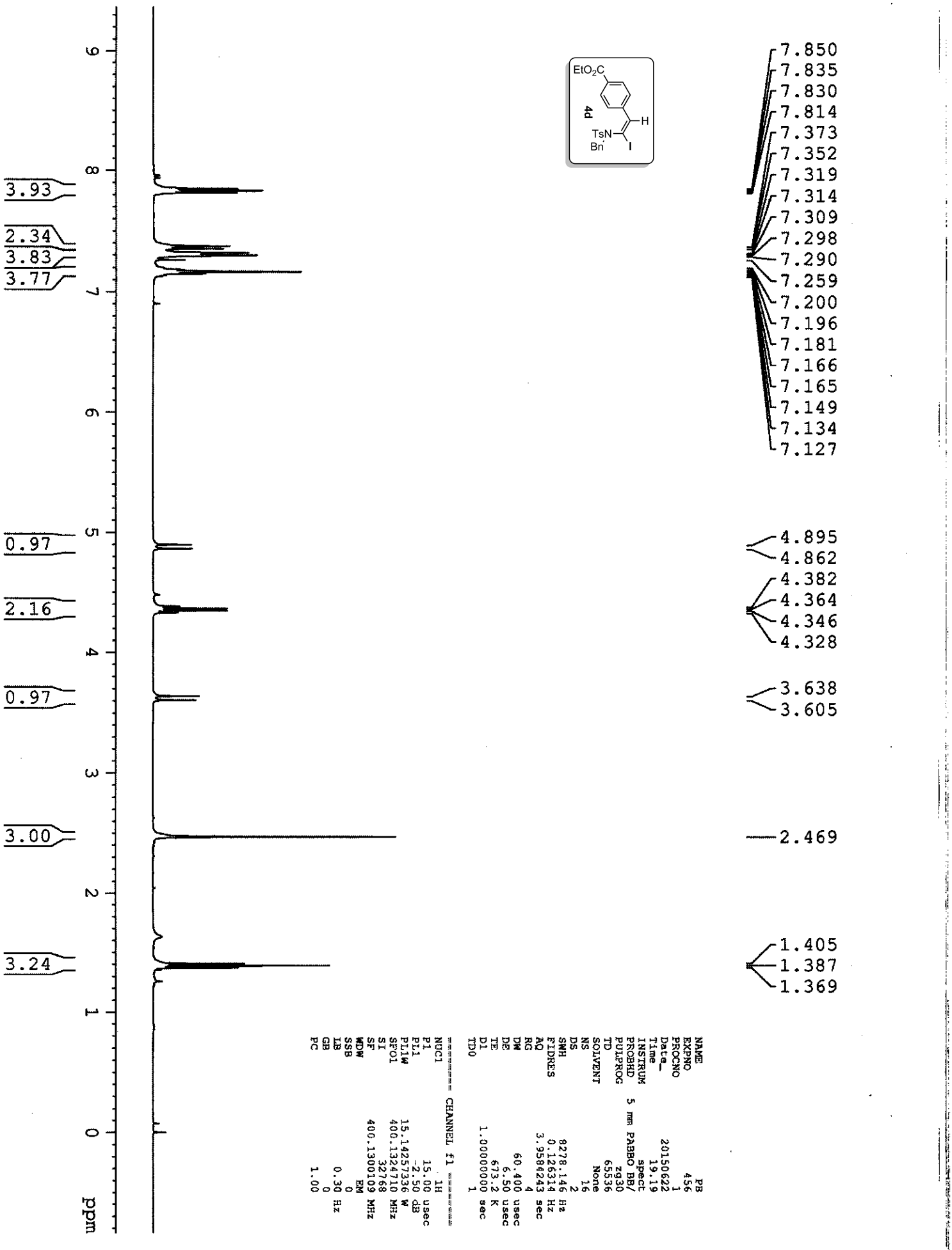

4a
Ts-N
Bn
H
I
148.26
144.92
134.90
133.34
132.98
130.00
129.63
129.58
128.80
128.72
128.38
128.20
127.99
98.01
77.36
77.25
77.05
55.15
21.76
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

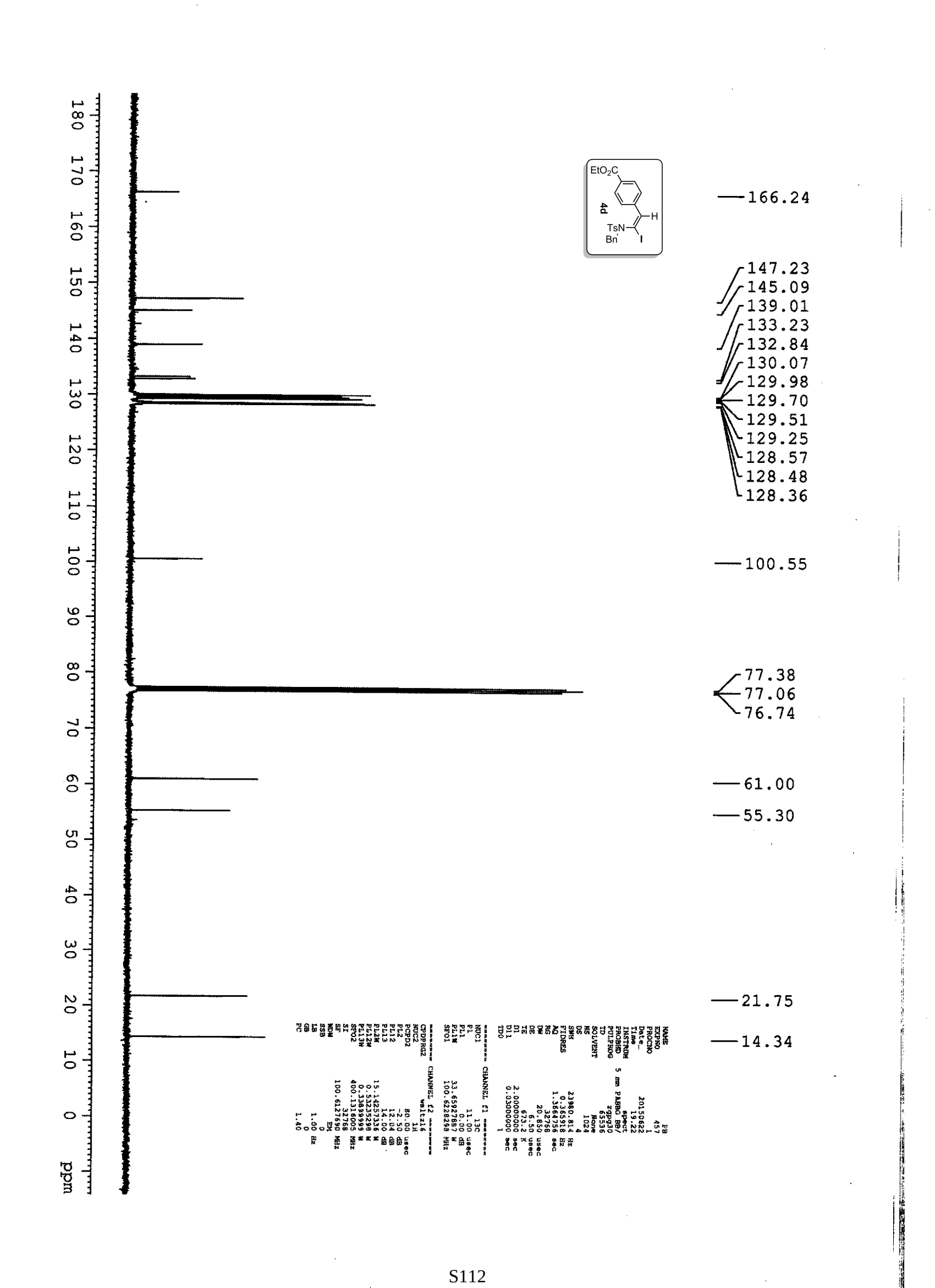

4b
7.843
7.822
7.357
7.339
7.320
7.296
7.242
7.177
7.172
7.164
7.161
7.071
7.053
6.985
6.922
6.903
4.865
4.832
3.686
3.653
2.449
2.207
2.199
2.158
2.108
0.000
2.05
5.02
0.19
2.00
1.08
2.01
1.01
1.00
1.02
1.01
3.06
0.69
3.06
3.05
ppm

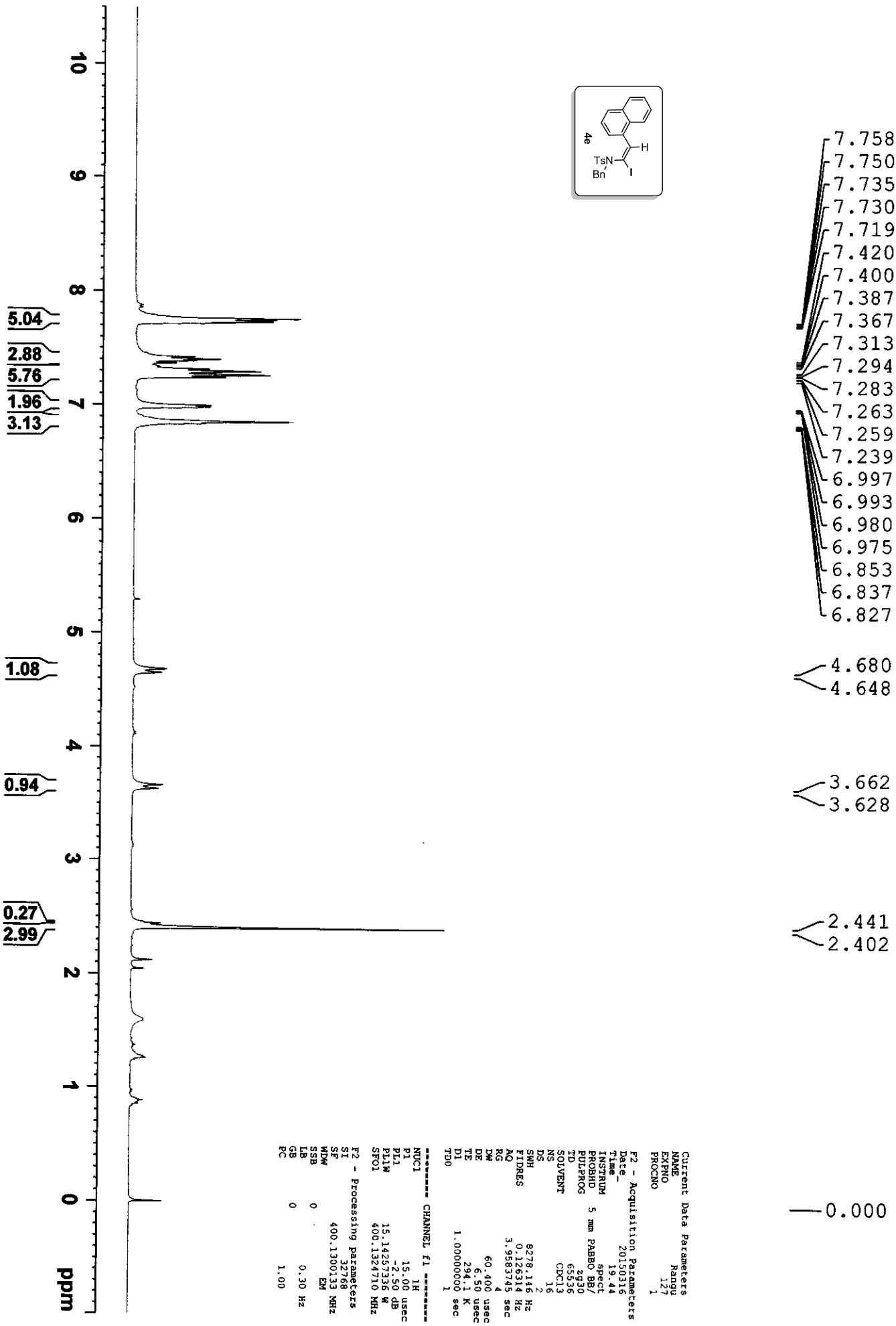

4b
148.20
144.74
137.55
135.94
133.79
133.32
132.64
130.06
129.88
129.68
129.58
129.46
129.37
129.27
128.71
128.50
128.33
128.27
126.39
96.87
77.38
77.06
76.75
55.14
21.73
19.68
19.59
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

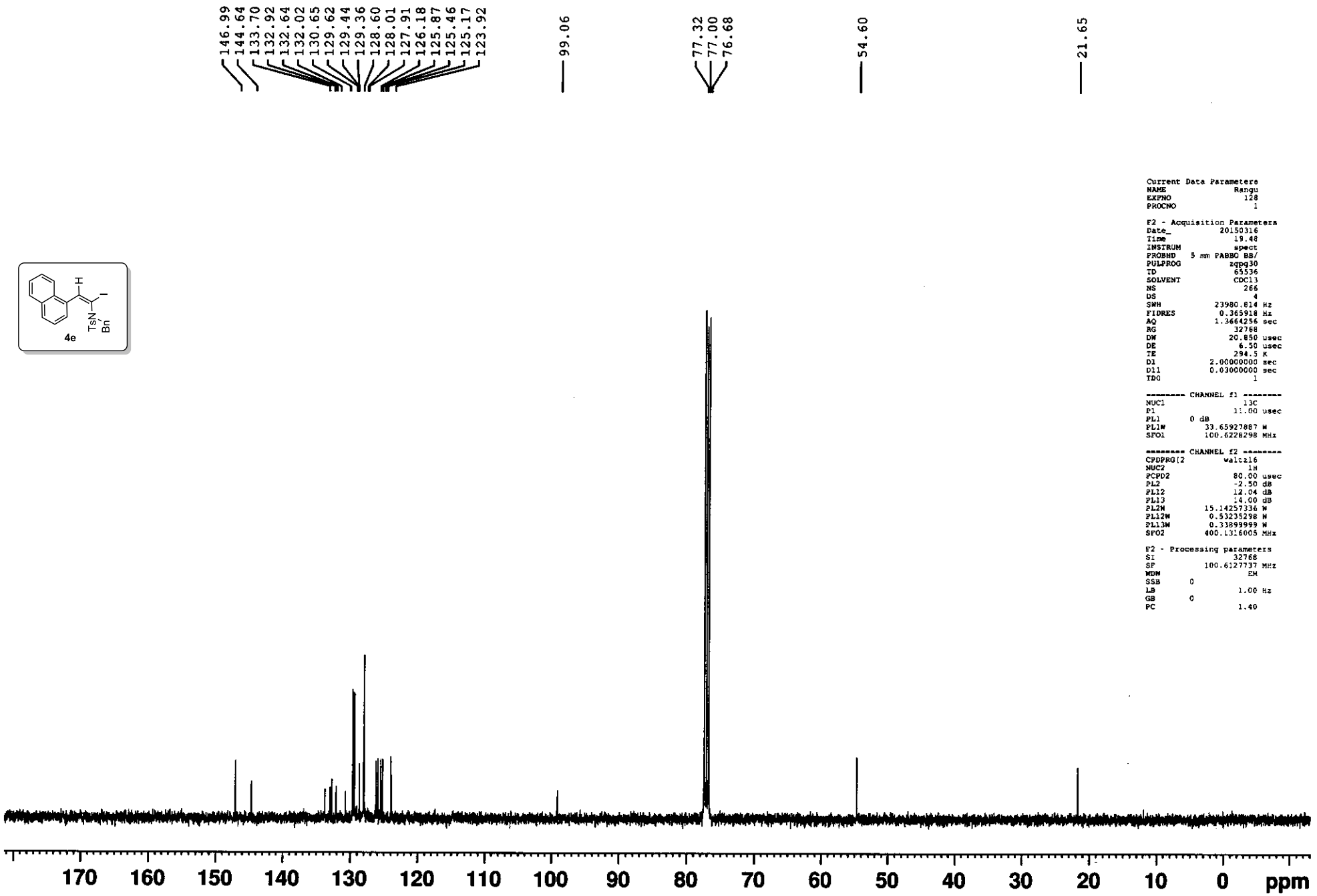

7.869
7.865
7.850
7.846
7.777
7.757
7.728
7.708
7.632
7.612
7.582
7.580
7.563
7.561
7.507
7.357
7.337
7.326
7.300
7.296
7.272
7.253
7.217
6.950
6.931
6.911
6.867
6.846
6.833
6.812
3.825
3.802
2.894
2.801
2.451
2.417
OMe
H
I
TsN
H3C
4c
Current Data Parameters
NAME RKM
EXPNO 11
PROCNO 1891
F2 - Acquisition Parameters
Date_ 20150319
Time 14.04
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 101
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.80 usec
PL1 1.50 dB
PL1W 15.18650627 W
SFO1 400.0934707 MHz
F2 - Processing parameters
SI 32768
SF 400.0910106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
0.90
0.14
0.59
1.81
0.18
0.29
0.87
0.90
0.39
2.98
0.15
1.13
0.36
0.95
0.80
2.57
0.74
2.54
0.97
3.00

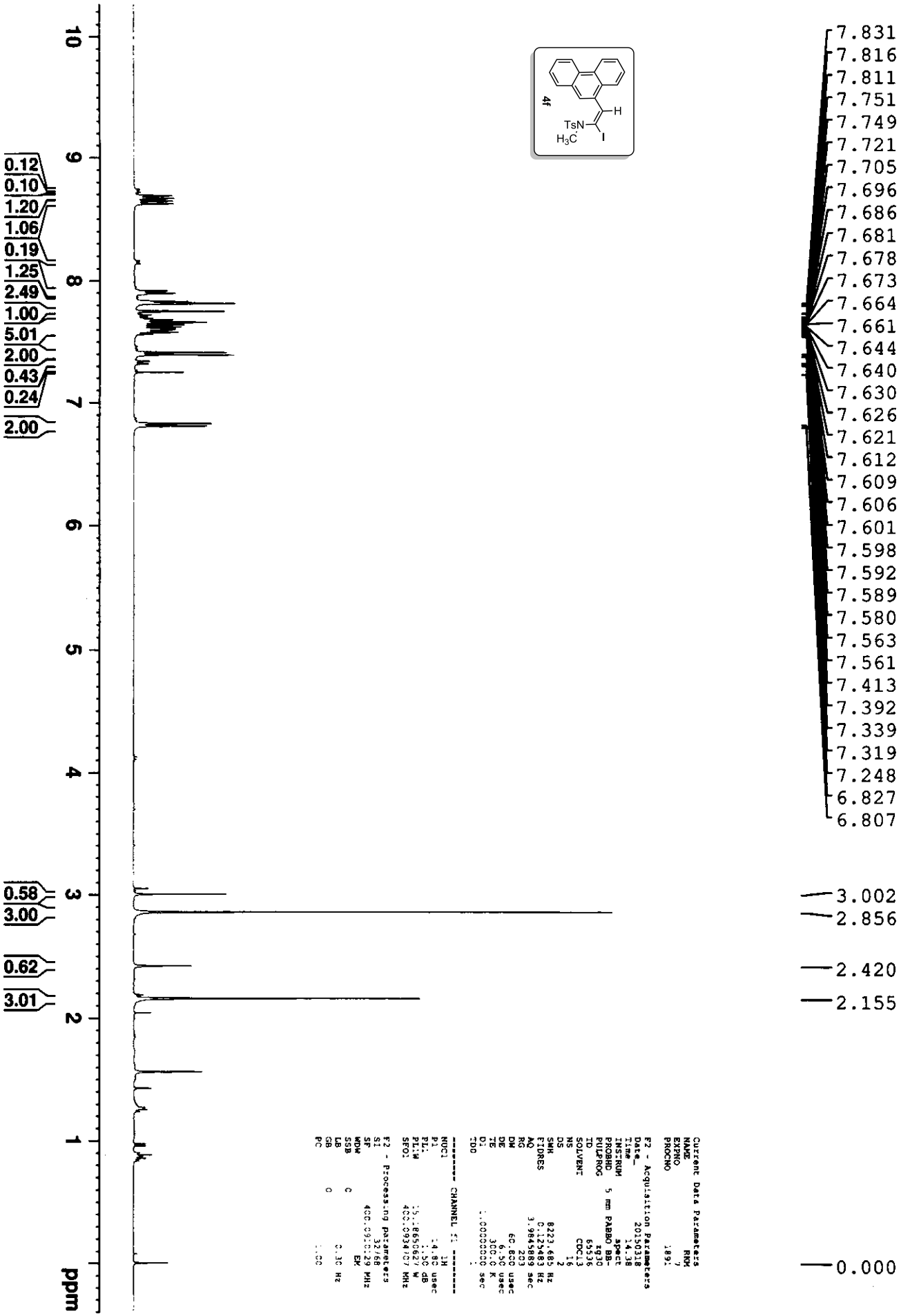

4c
OMe
H
TsN
H3C
I
157.05
156.46
144.42
144.26
139.74
136.70
133.94
132.97
130.38
130.12
130.07
129.68
129.50
129.31
129.24
128.72
128.67
127.62
127.58
124.91
124.18
123.94
123.08
120.96
120.66
119.86
110.88
110.59
110.48
101.42
99.40
97.69
77.32
77.00
76.68
55.50
55.46
38.80
38.32
21.63
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

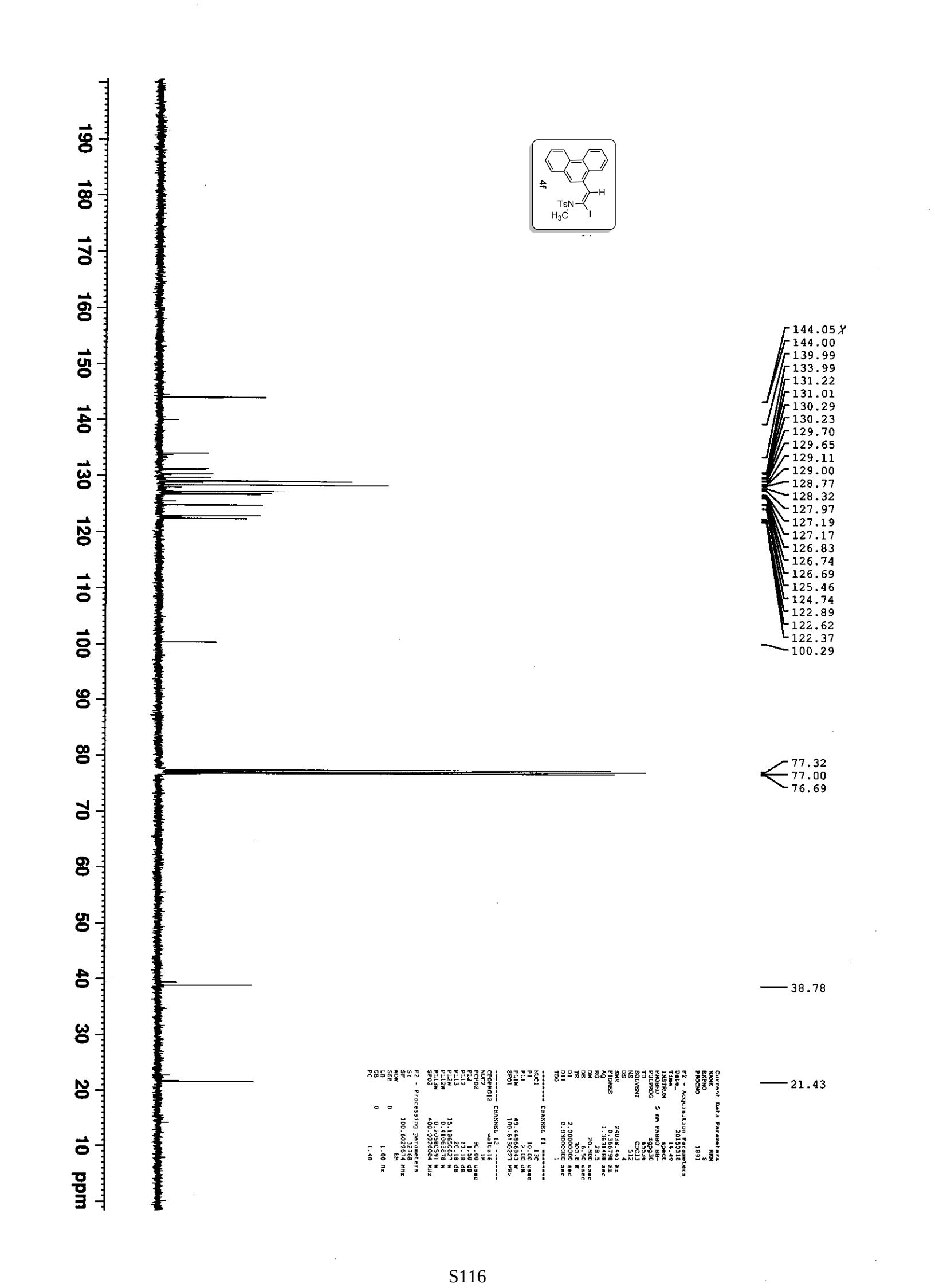

7.850
7.835
7.830
7.814
7.373
7.352
7.319
7.314
7.309
7.298
7.290
7.259
7.200
7.196
7.181
7.166
7.165
7.149
7.134
7.127
4.895
4.862
4.382
4.364
4.346
4.328
3.638
3.605
2.469
1.405
1.387
1.369
3.93
2.34
3.83
3.77
0.97
2.16
0.97
3.00
3.24
ppm
EtO2C
4d
TsN
Bn
H
I
NAME PB
EXPNO 456
PROCNO 1
Date_ 20150622
Time 19.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT None
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300109 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

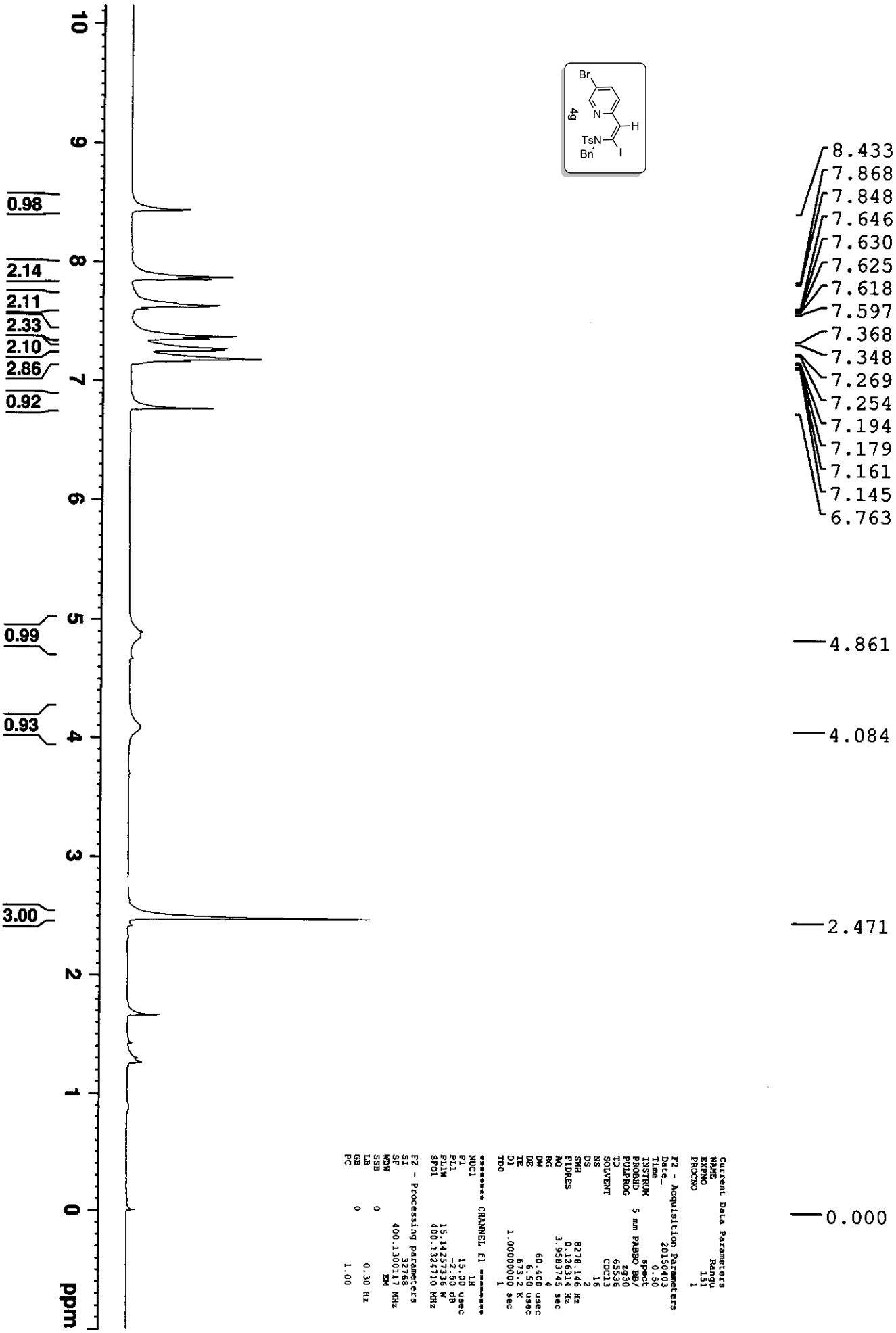

EtO2C
4d
TsN
Bn
H
I
166.24
147.23
145.09
139.01
133.23
132.84
130.07
129.98
129.70
129.51
129.25
128.57
128.48
128.36
100.55
77.38
77.06
76.74
61.00
55.30
21.75
14.34
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


4e
TsN
Bn
H
I
7.758
7.750
7.735
7.730
7.719
7.420
7.400
7.387
7.367
7.313
7.294
7.283
7.263
7.259
7.239
6.997
6.993
6.980
6.975
6.853
6.837
6.827
4.680
4.648
3.662
3.628
2.441
2.402
0.000
5.04
2.88
5.76
1.96
3.13
1.08
0.94
0.27
2.99
10
9
8
7
6
5
4
3
2
1
0
ppm
Current Data Parameters
NAME Rangu
EXPNO 127
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150316
Time 19.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PLW1 15.14257336 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300133 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


146.99
144.64
133.70
132.92
132.64
132.02
130.65
129.62
129.44
129.36
128.60
128.01
127.91
126.18
125.87
125.46
125.17
123.92
99.06
77.32
77.00
76.68
54.60
21.65
4e
Current Data Parameters
NAME Rangu
EXPNO 128
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150316
Time 19.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zqpg30
TD 65536
SOLVENT CDCl3
NS 266
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 294.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 11.00 usec
PL1 0 dB
PL1W 33.65927887 W
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.50 dB
PL12 12.04 dB
PL13 14.00 dB
PL2W 15.14257336 W
PL12W 0.53235298 W
PL13W 0.33899999 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127737 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


4f
TsN
H3C
H
I
0.12
0.10
1.20
1.06
0.19
1.25
2.49
1.00
5.01
2.00
0.43
0.24
2.00
0.58
3.00
0.62
3.01
ppm
7.831
7.816
7.811
7.751
7.749
7.721
7.705
7.696
7.686
7.681
7.678
7.673
7.664
7.661
7.644
7.640
7.630
7.626
7.621
7.612
7.609
7.606
7.601
7.598
7.592
7.589
7.580
7.563
7.561
7.413
7.392
7.339
7.319
7.248
6.827
6.807
3.002
2.856
2.420
2.155
0.000


4f
TsN
H3C
H
I
144.05
144.00
139.99
133.99
131.22
131.01
130.29
130.23
129.70
129.65
129.11
129.00
128.77
128.32
127.97
127.19
127.17
126.83
126.74
126.69
125.46
124.74
122.89
122.62
122.37
100.29
77.32
77.00
76.69
38.78
21.43
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

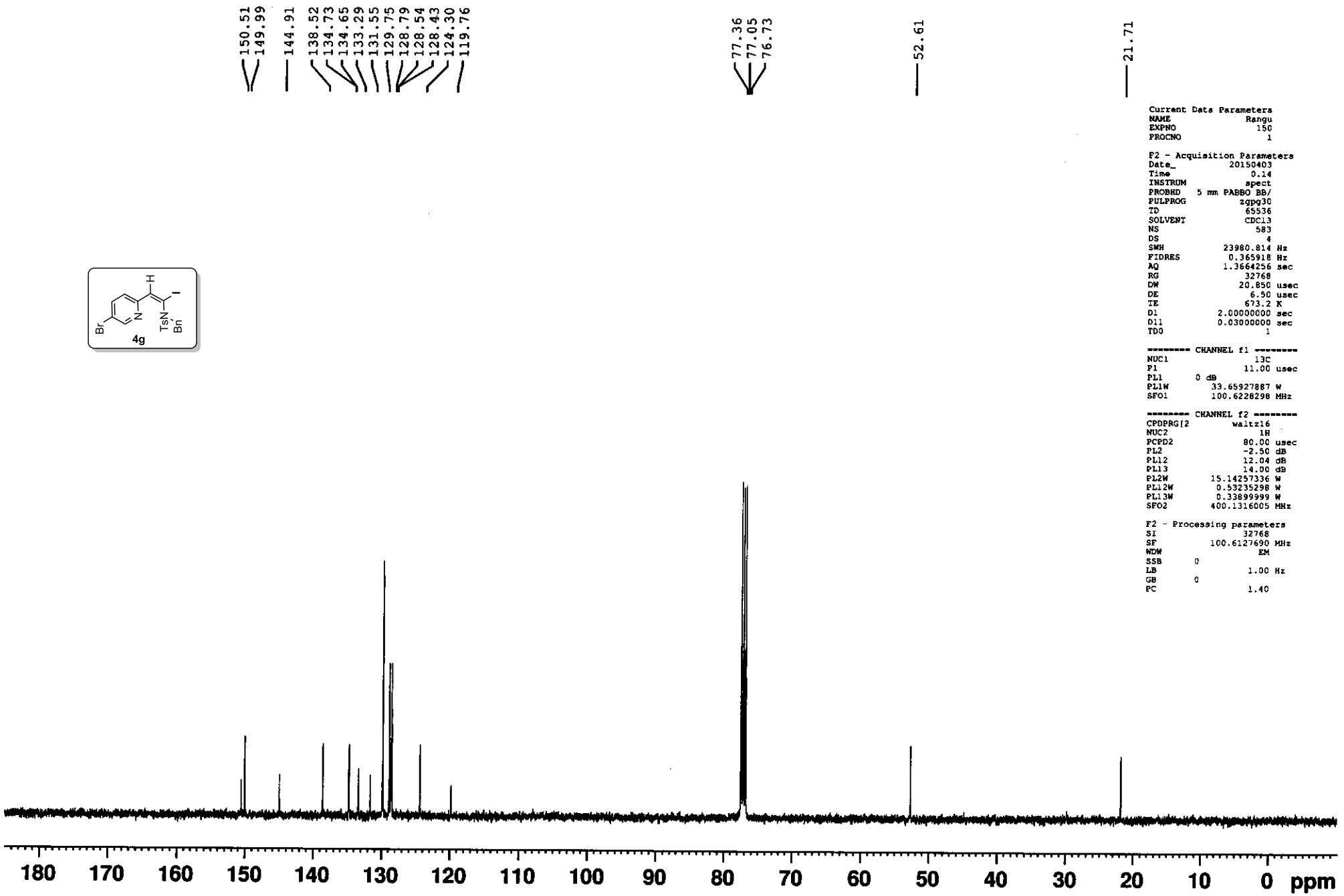


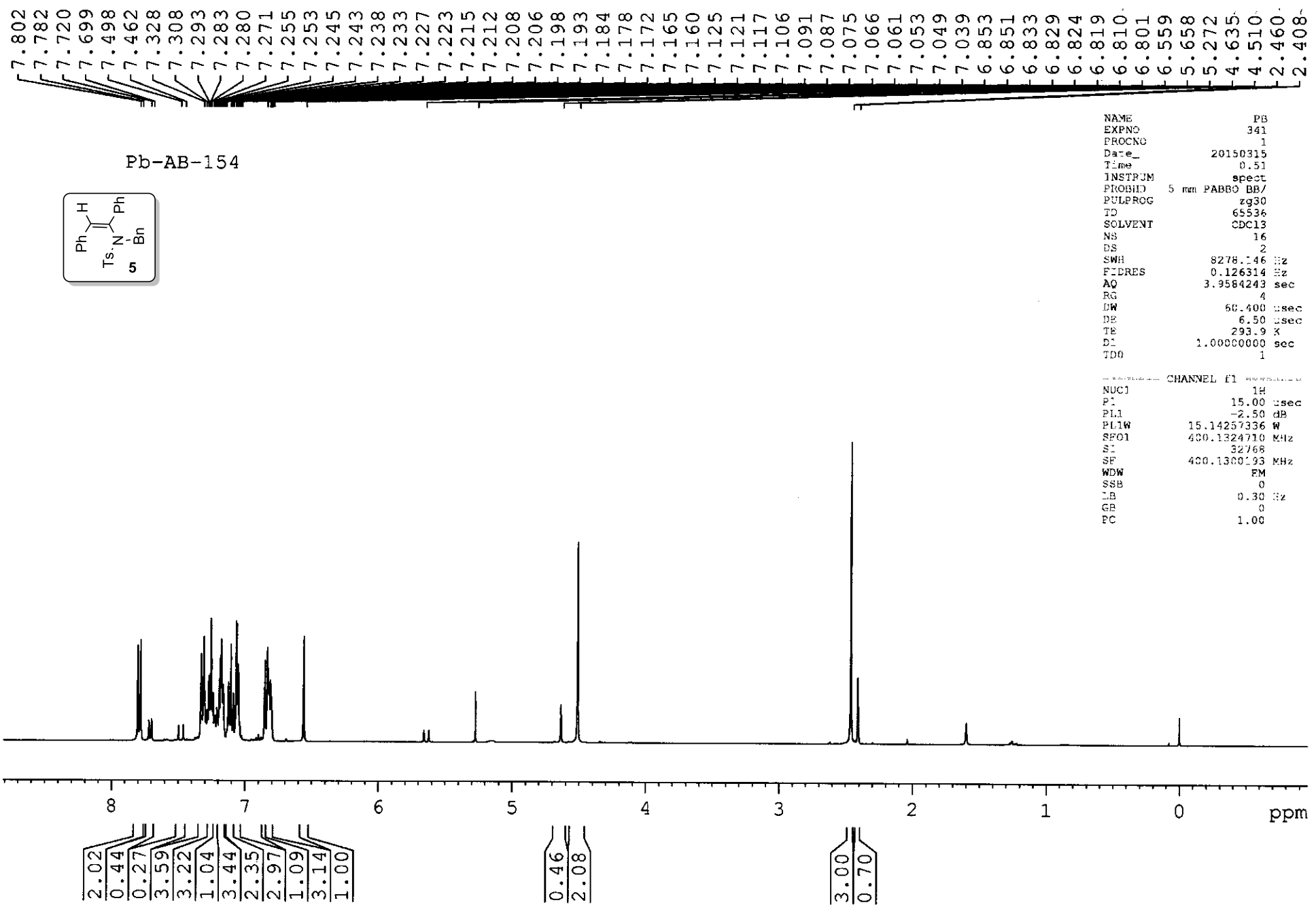
Current Data Parameters
NAME
EXPNO 151
PROCNO 1

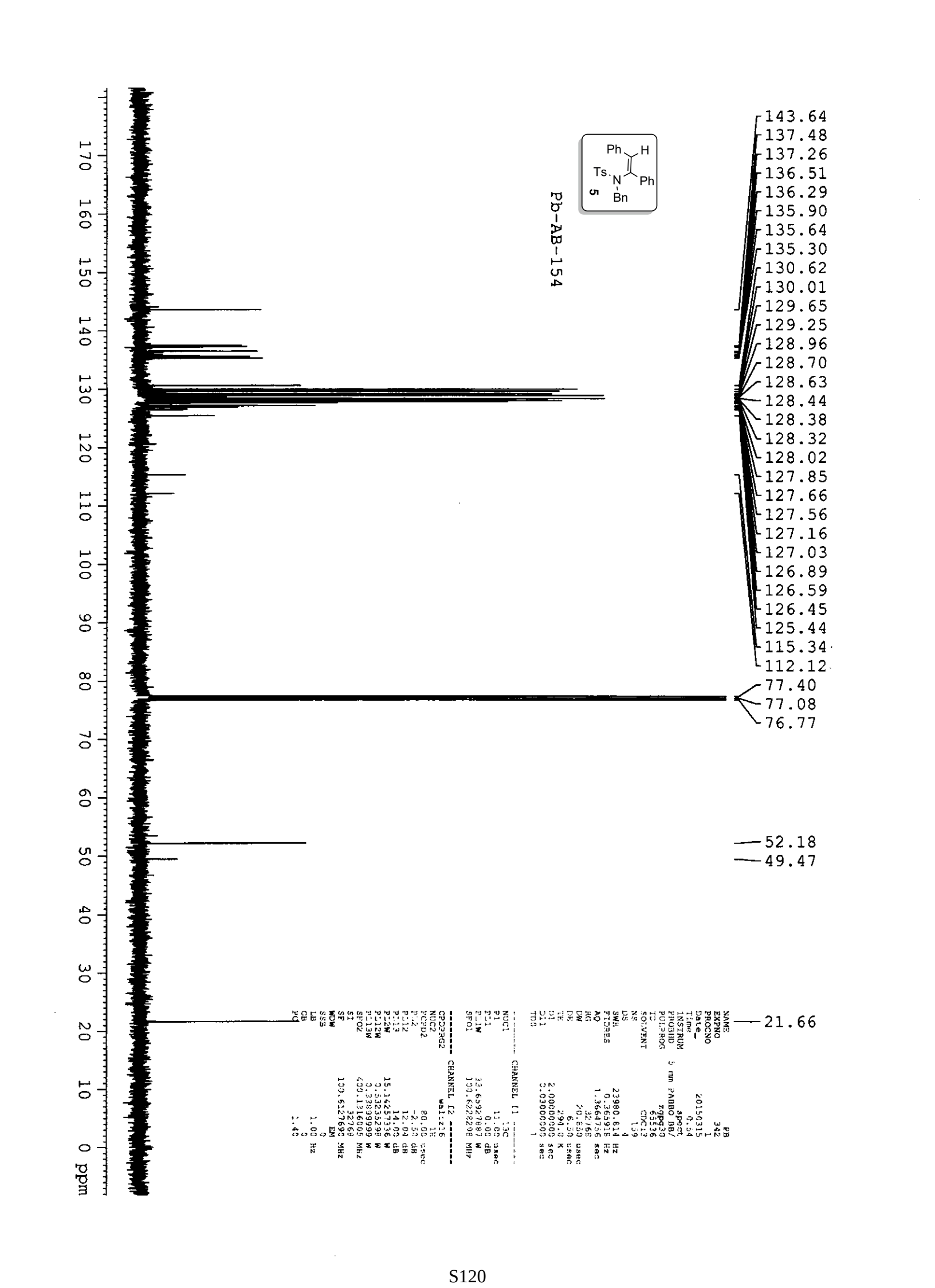
F2 - Acquisition Parameters
Date_ 20150403
Time 0.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1

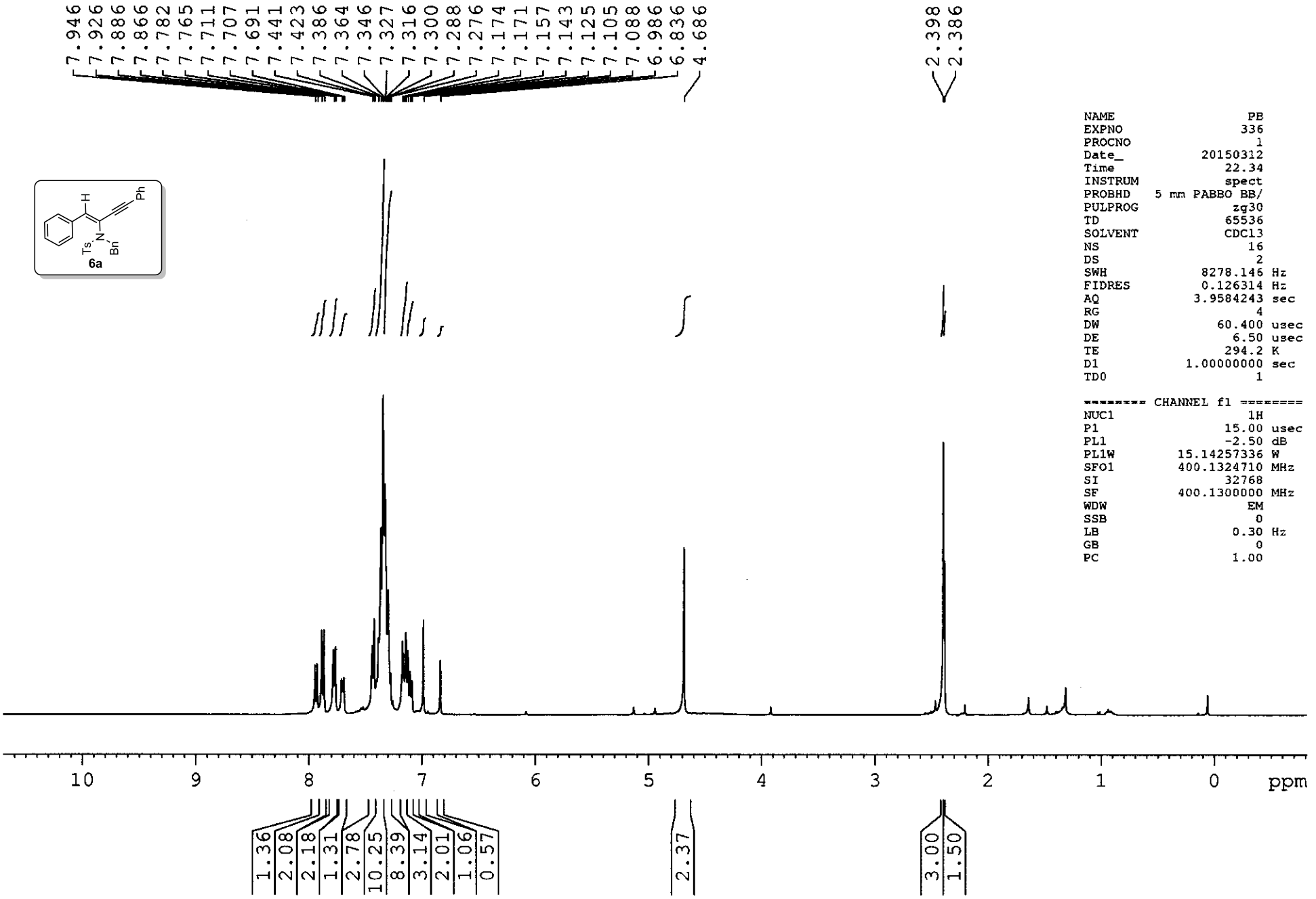
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz

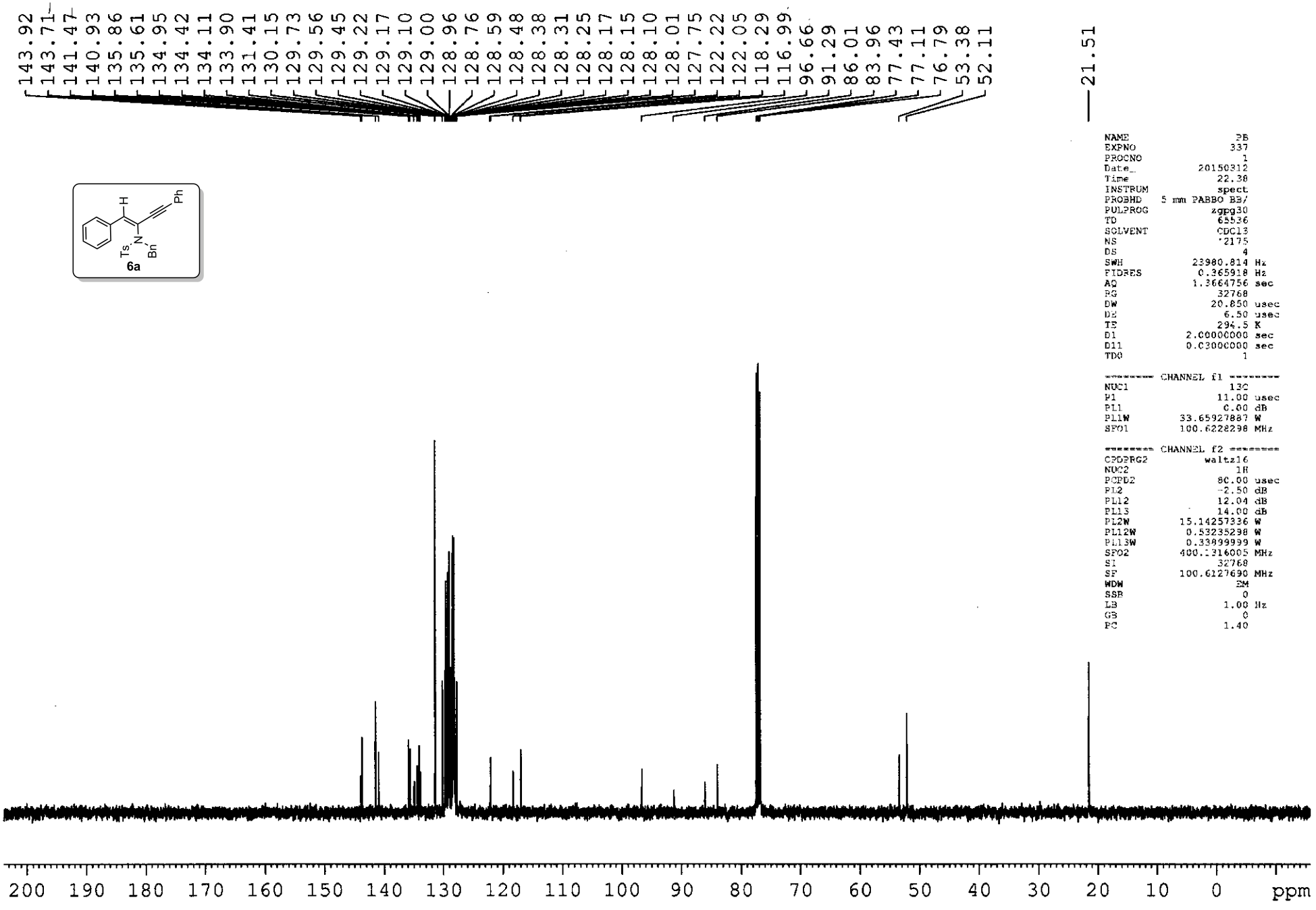
F2 - Processing parameters
SI 32768
SF 400.1300117 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

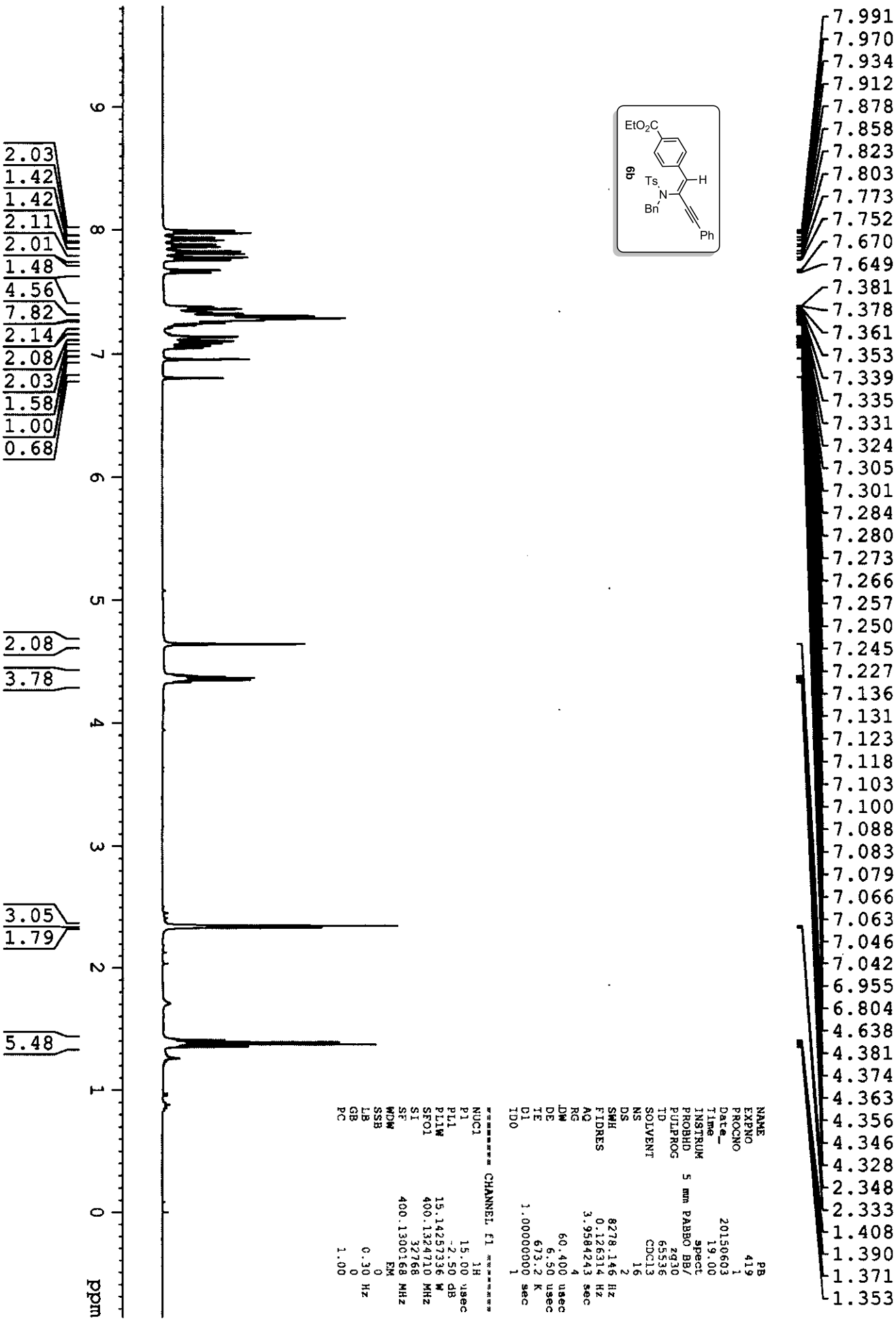

150.51
149.99
144.91
138.52
134.73
134.65
133.29
131.55
129.75
128.79
128.54
128.43
124.30
119.76
77.36
77.05
76.73
52.61
21.71
4g
Current Data Parameters
NAME Rangu
EXPNO 150
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150403
Time 0.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 583
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0 dB
PL1W 33.65927887 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.50 dB
PL12 12.04 dB
PL13 14.00 dB
PL2W 15.14257336 W
PL12W 0.53235298 W
PL13W 0.33899999 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

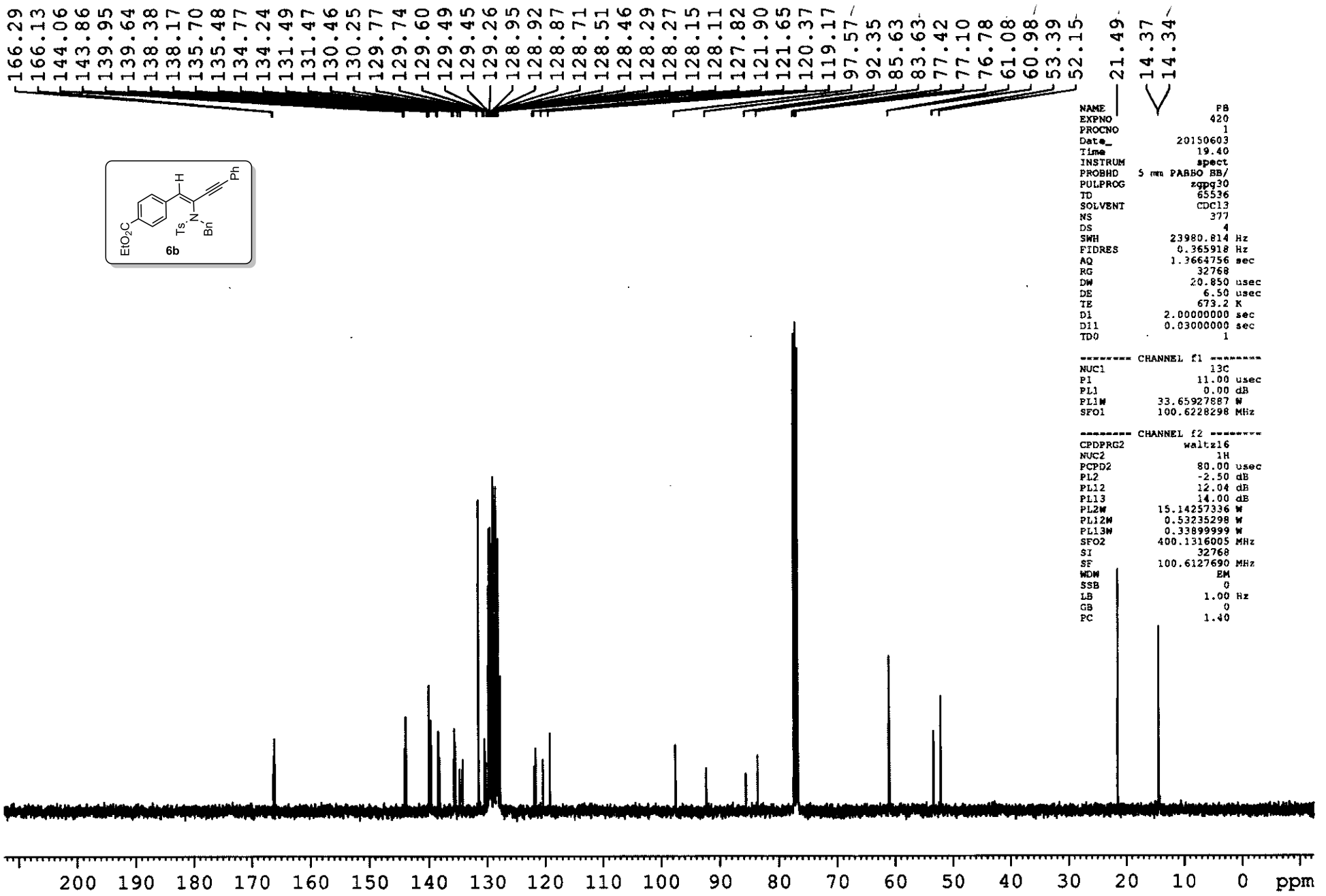

7.802
7.782
7.720
7.699
7.498
7.462
7.328
7.308
7.293
7.283
7.280
7.271
7.255
7.253
7.245
7.243
7.238
7.233
7.227
7.223
7.215
7.212
7.208
7.206
7.198
7.193
7.184
7.178
7.172
7.165
7.160
7.125
7.121
7.117
7.106
7.091
7.087
7.075
7.066
7.061
7.053
7.049
7.039
6.853
6.851
6.833
6.829
6.824
6.819
6.810
6.801
6.559
5.658
5.272
4.635
4.510
2.460
2.408
Pb-AB-154
Ph
H
Ph
Ts
N
Bn
5
NAME Pb
EXPNO 341
PROCNO 1
Date_ 20150315
Time 0.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0
ppm
2.02
0.44
0.27
3.59
3.22
1.04
3.44
2.35
2.97
1.09
3.14
1.00
0.46
2.08
3.00
0.70

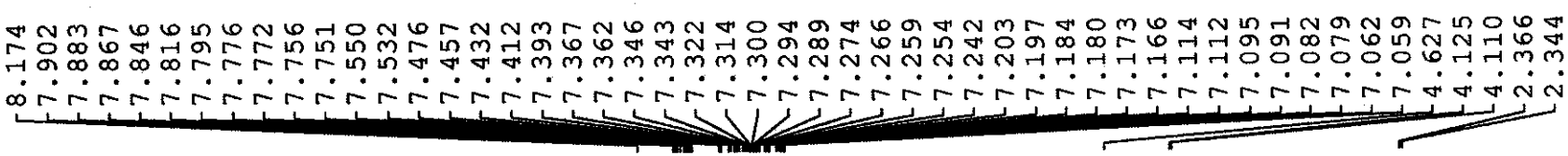

Pb-AB-154
Ph
H
Ts
N
Ph
Bn
5
143.64
137.48
137.26
136.51
136.29
135.90
135.64
135.30
130.62
130.01
129.65
129.25
128.96
128.70
128.63
128.44
128.38
128.32
128.02
127.85
127.66
127.56
127.16
127.03
126.89
126.59
126.45
125.44
115.34
112.12
77.40
77.08
76.77
52.18
49.47
21.66
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

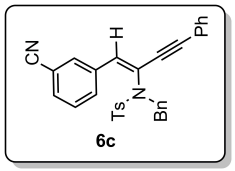

7.946
7.926
7.886
7.866
7.782
7.765
7.711
7.707
7.691
7.441
7.423
7.386
7.364
7.346
7.327
7.316
7.300
7.288
7.276
7.174
7.171
7.157
7.143
7.125
7.105
7.088
6.986
6.836
4.686
2.398
2.386
6a
NAME PB
EXPNO 336
PROCNO 1
Date_ 20150312
Time 22.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.36
2.08
2.18
1.31
2.78
10.25
8.39
3.14
2.01
1.06
0.57
2.37
3.00
1.50


143.92
143.71
141.47
140.93
135.86
135.61
134.95
134.42
134.11
133.90
131.41
130.15
129.73
129.56
129.45
129.22
129.17
129.10
129.00
128.96
128.76
128.59
128.48
128.38
128.31
128.25
128.17
128.15
128.10
128.01
127.75
122.22
122.05
118.29
116.99
96.66
91.29
86.01
83.96
77.43
77.11
76.79
53.38
52.11
21.51
Ph
H
Ts
N
Bn
6a
NAME PB
EXPNO 337
PROCNO 1
Date_ 20150312
Time 22.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS *2175
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 294.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
PL1W 33.65927887 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.50 dB
PL12 12.04 dB
PL13 14.00 dB
PL2W 15.14257336 W
PL12W 0.53235298 W
PL13W 0.33999999 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.991
7.970
7.934
7.912
7.878
7.858
7.823
7.803
7.773
7.752
7.670
7.649
7.381
7.378
7.361
7.353
7.339
7.335
7.331
7.324
7.305
7.301
7.284
7.280
7.273
7.266
7.257
7.250
7.245
7.227
7.136
7.131
7.123
7.118
7.103
7.100
7.088
7.083
7.079
7.066
7.063
7.046
7.042
6.955
6.804
4.638
4.381
4.374
4.363
4.356
4.346
4.328
2.348
2.333
1.408
1.390
1.371
1.353
EtO2C
6b
Ts
N
Bn
H
Ph
2.03
1.42
1.42
2.11
2.01
1.48
4.56
7.82
2.14
2.08
2.03
1.58
1.00
0.68
2.08
3.78
3.05
1.79
5.48
ppm
NAME PB
EXPNO 419
PROCNO 1
Date_ 20150603
Time 19.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300168 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


166.29
166.13
144.06
143.86
139.95
139.64
138.38
138.17
135.70
135.48
134.77
134.24
131.49
131.47
130.46
130.25
129.77
129.74
129.60
129.49
129.45
129.26
128.95
128.92
128.87
128.71
128.51
128.46
128.29
128.27
128.15
128.11
127.82
121.90
121.65
120.37
119.17
97.57
92.35
85.63
83.63
77.42
77.10
76.78
61.08
60.98
53.39
52.15
21.49
14.37
14.34
Ph
H
N
Ts
Bn
EtO2C
6b
NAME PB
EXPNO 420
PROCNO 1
Date_ 20150603
Time 19.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 377
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
PL1W 33.65927887 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.50 dB
PL12 12.04 dB
PL13 14.00 dB
PL2W 15.14257336 W
PL12W 0.53235298 W
PL13W 0.33899999 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

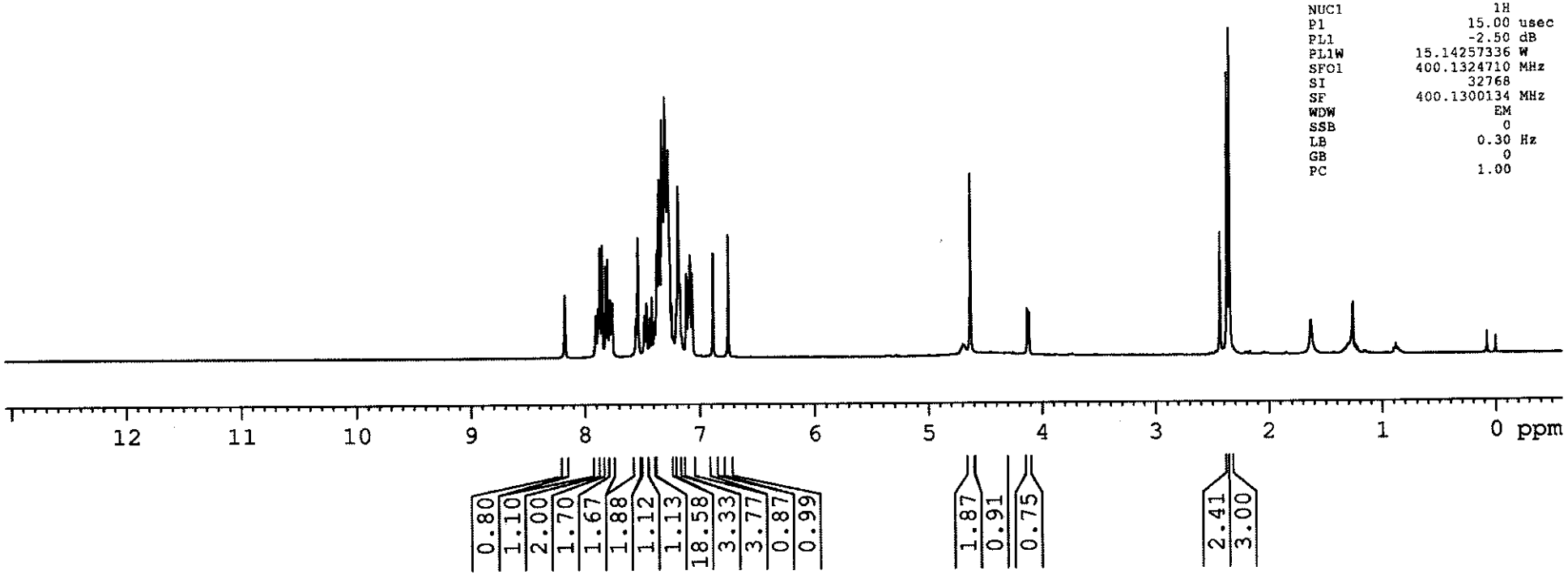


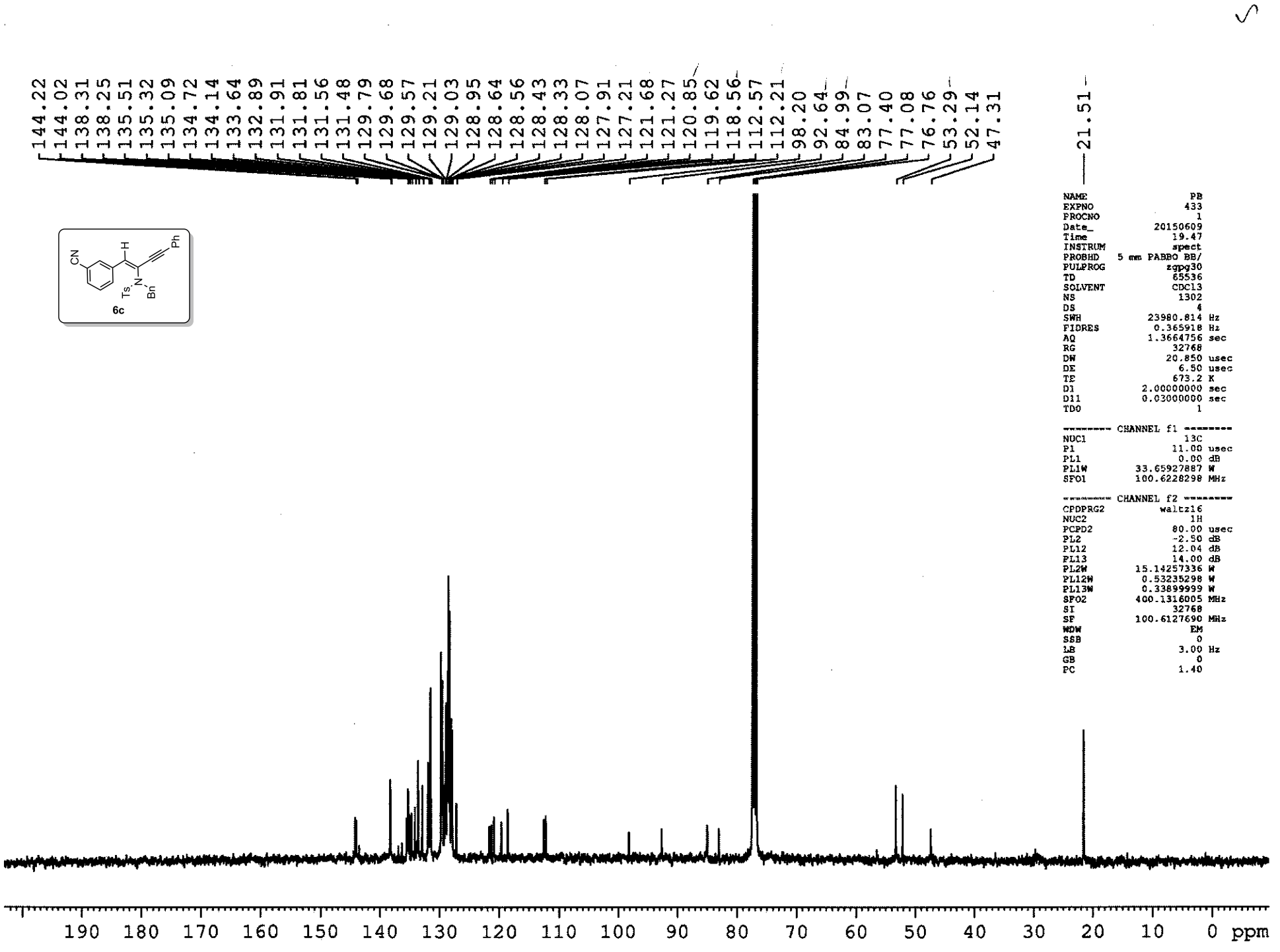


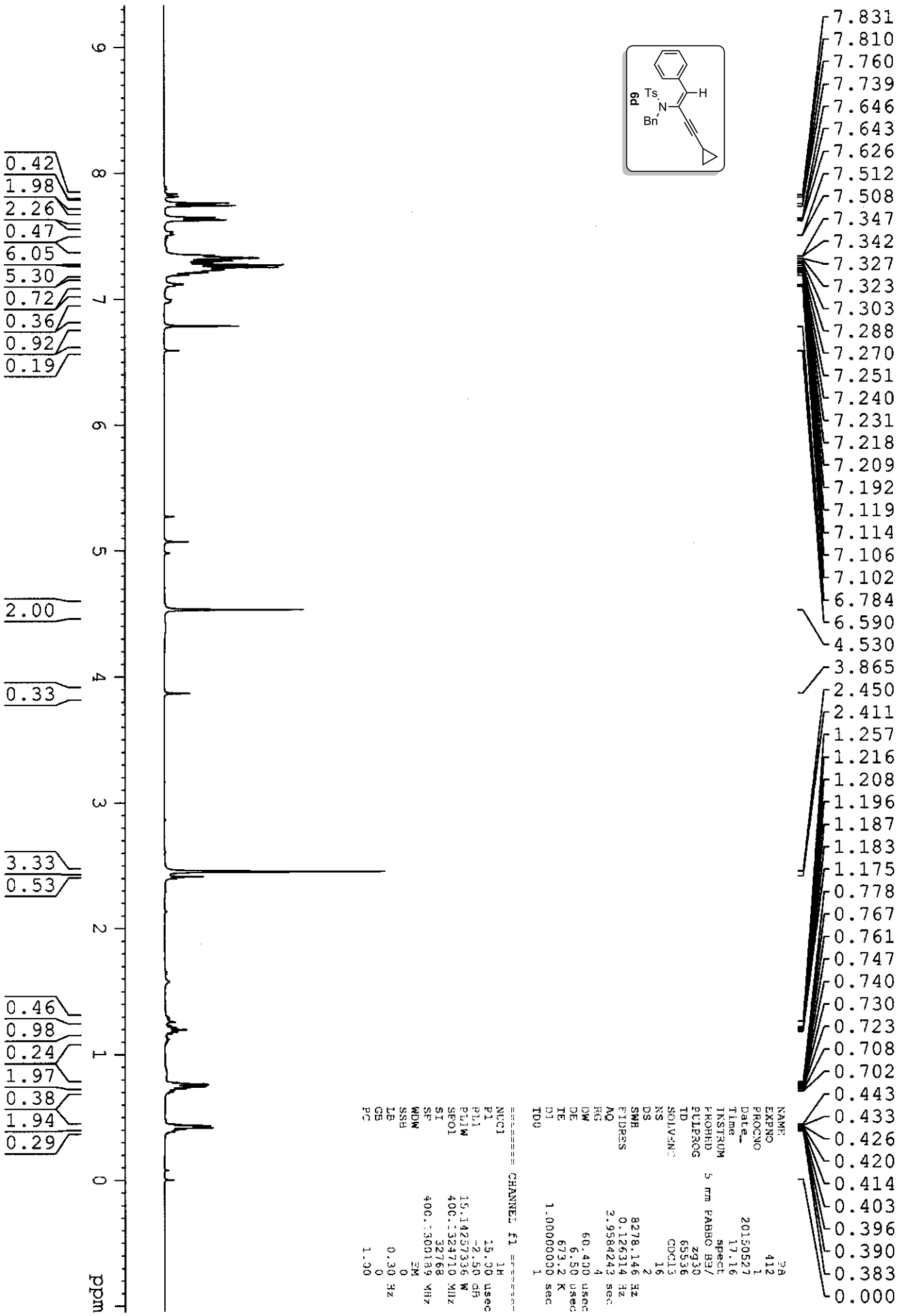
NAME	PB
EXPNO	432
PROCNO	1
Date_	20150609
Time	19.45
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	4
DW	60.400 usec
DE	6.50 usec
TE	673.2 K
D1	1.00000000 sec
TD0	1

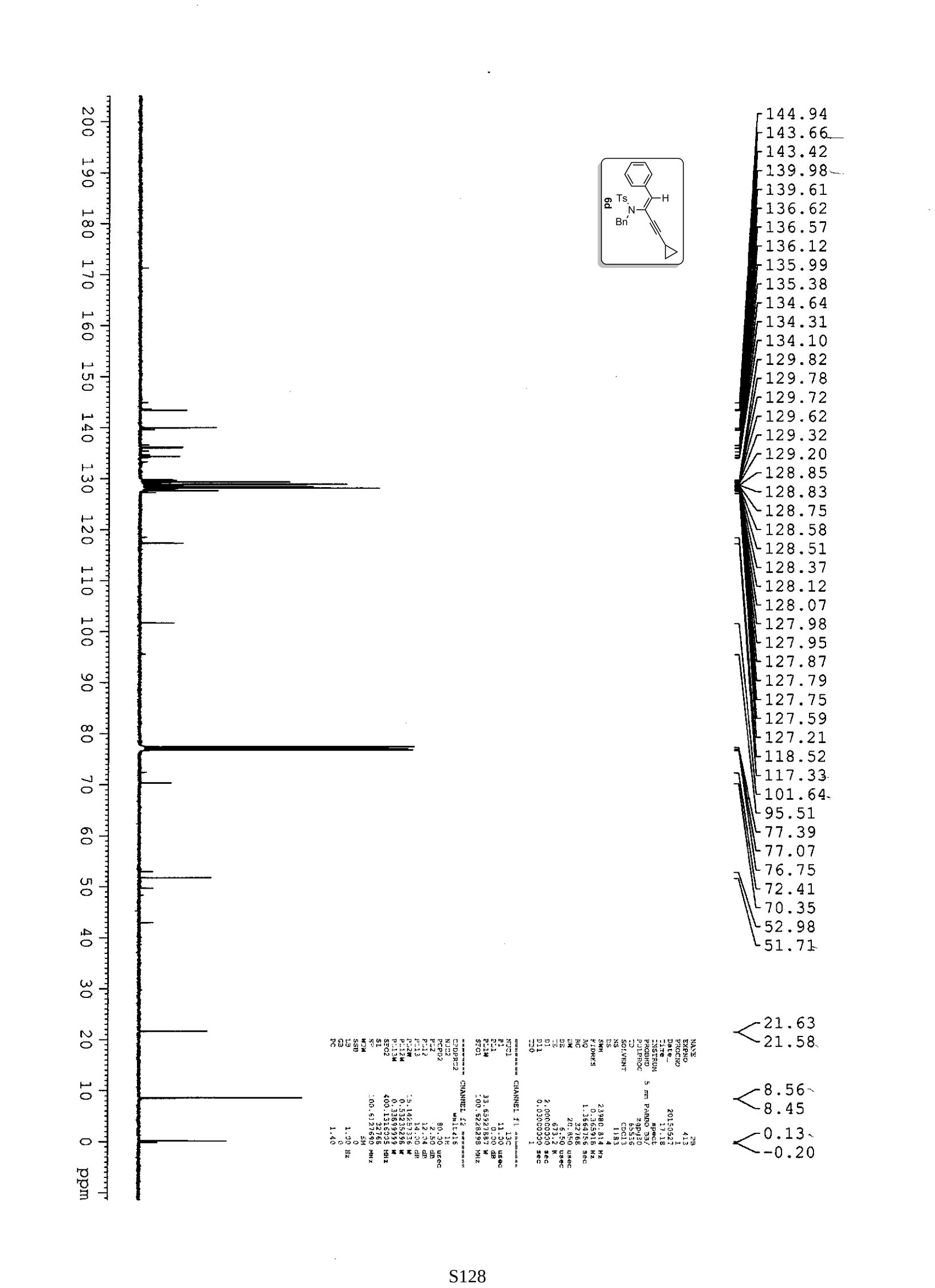
======== CHANNEL f1 ========

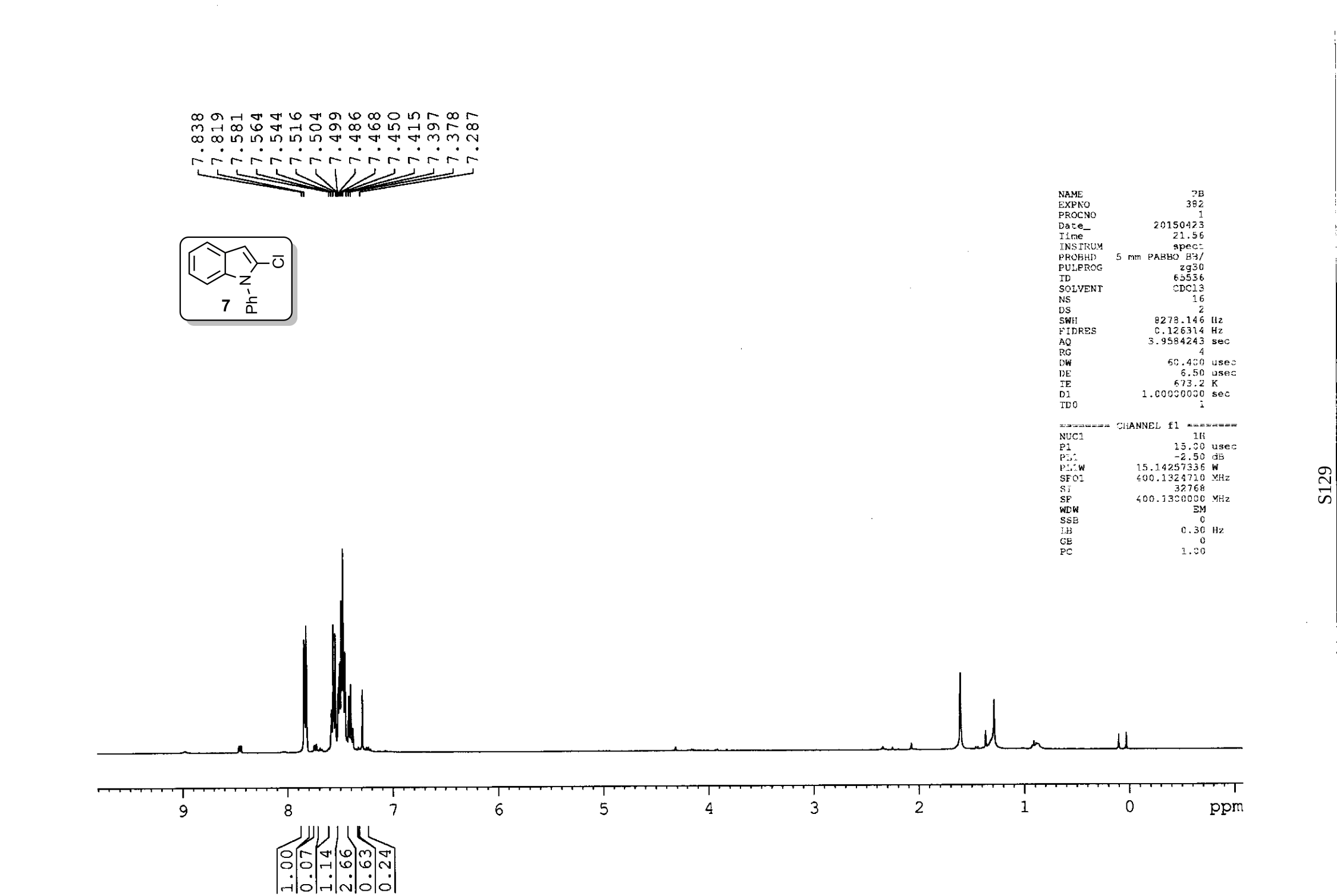
NUC1	1H
P1	15.00 usec
PL1	-2.50 dB
PL1W	15.14257336 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300134 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

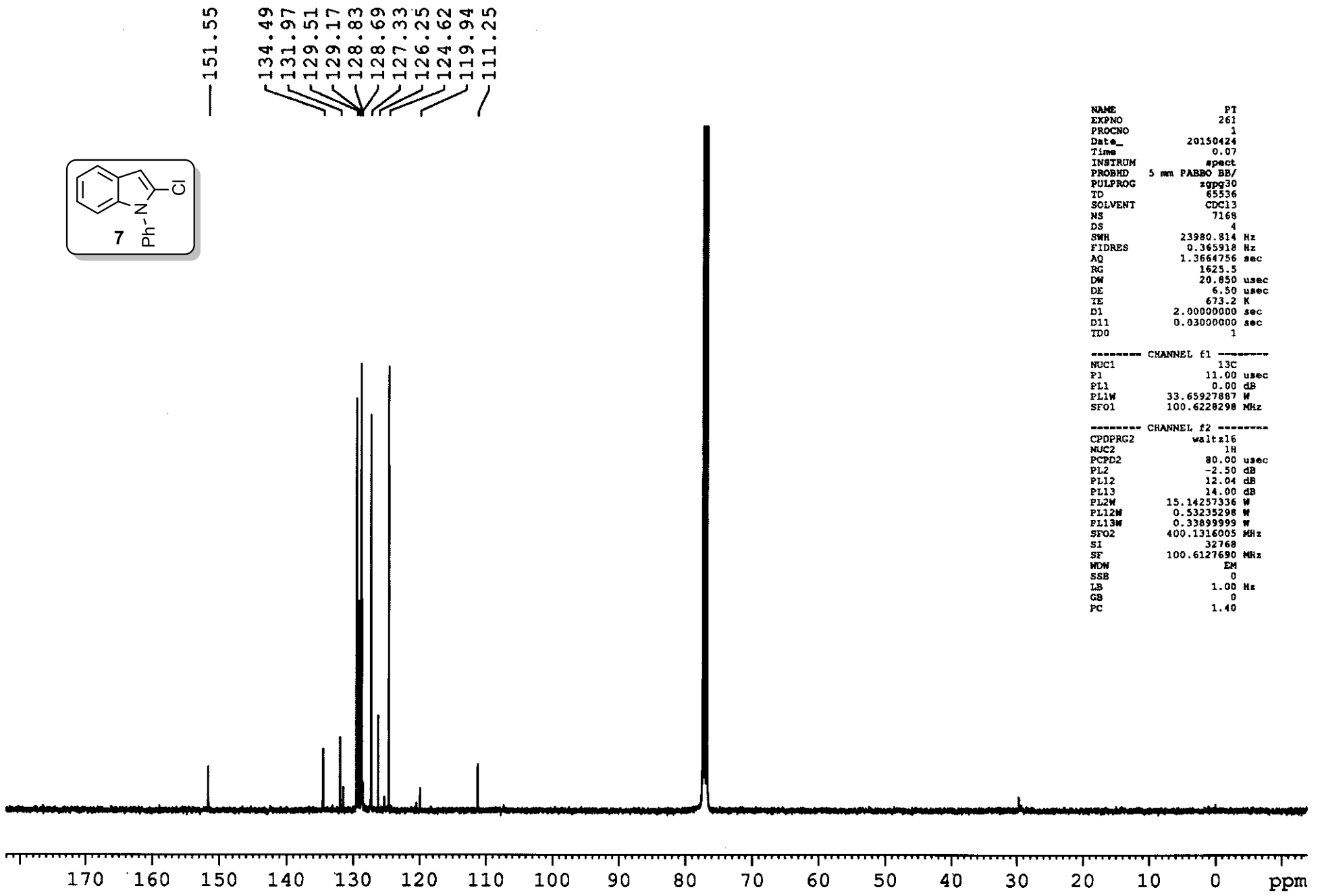



144.22
144.02
138.31
138.25
135.51
135.32
135.09
134.72
134.14
133.64
132.89
131.91
131.81
131.56
131.48
129.79
129.68
129.57
129.21
129.03
128.95
128.64
128.56
128.43
128.33
128.07
127.91
127.21
121.68
121.27
120.85
119.62
118.56
112.57
112.21
98.20
92.64
84.99
83.07
77.40
77.08
76.76
53.29
52.14
47.31
21.51
CN
H
Ph
Ts
N
Bn
6c
NAME PB
EXPNO 433
PROCNO 1
Date_ 20150609
Time 19.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1302
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
PL1W 33.65927887 W
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.50 dB
PL12 12.04 dB
PL13 14.00 dB
PL2W 15.14257336 W
PL12W 0.53235298 W
PL13W 0.33899999 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

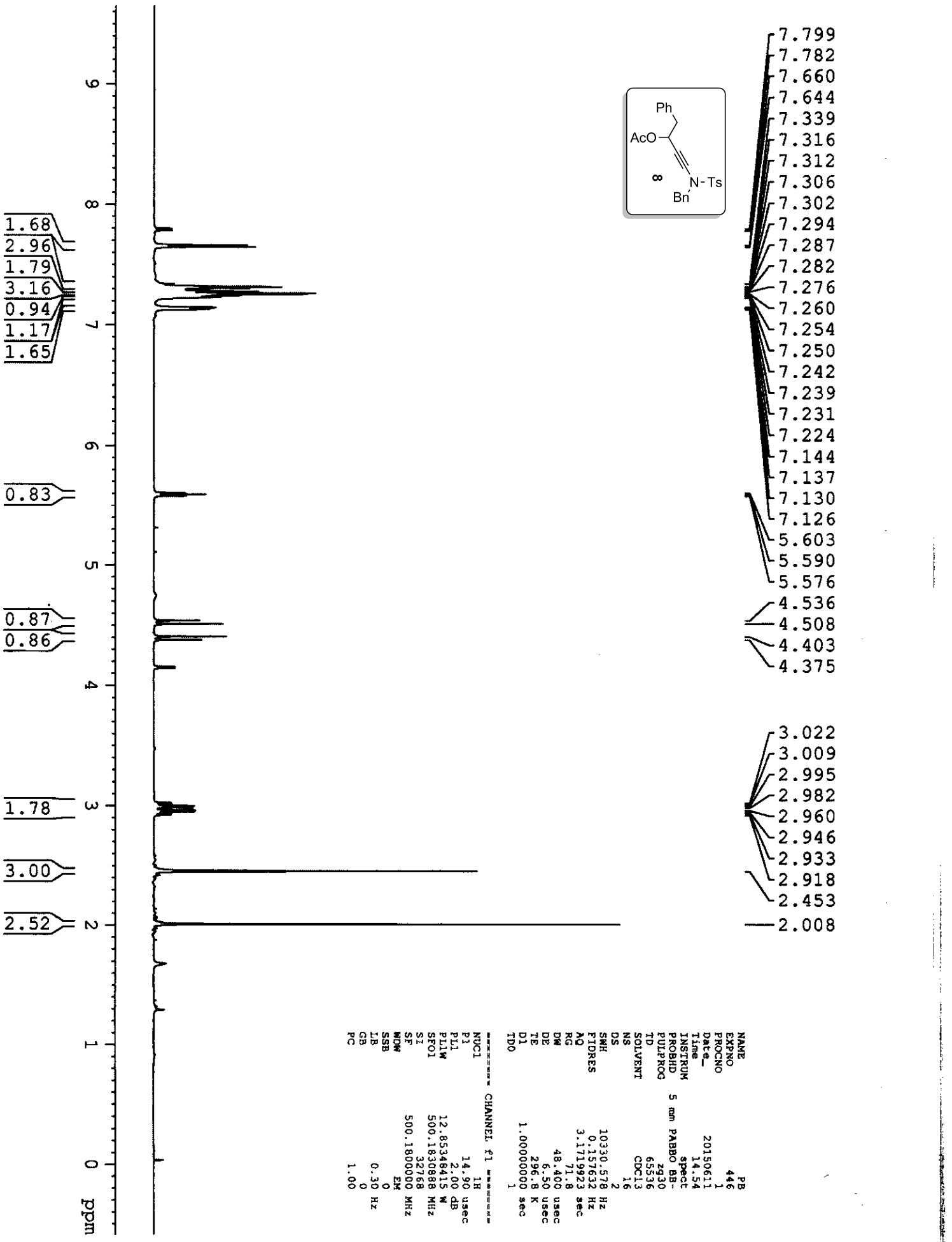

6d
Ts
N
Bn
H
7.831
7.810
7.760
7.739
7.646
7.643
7.626
7.512
7.508
7.347
7.342
7.327
7.323
7.303
7.288
7.270
7.251
7.240
7.231
7.218
7.209
7.192
7.119
7.114
7.106
7.102
6.784
6.590
4.530
3.865
2.450
2.411
1.257
1.216
1.208
1.196
1.187
1.183
1.175
0.778
0.767
0.761
0.747
0.740
0.730
0.723
0.708
0.702
0.443
0.433
0.426
0.420
0.414
0.403
0.396
0.390
0.383
0.000
0.42
1.98
2.26
0.47
6.05
5.30
0.72
0.36
0.92
0.19
2.00
0.33
3.33
0.53
0.46
0.98
0.24
1.97
0.38
1.94
0.29
ppm

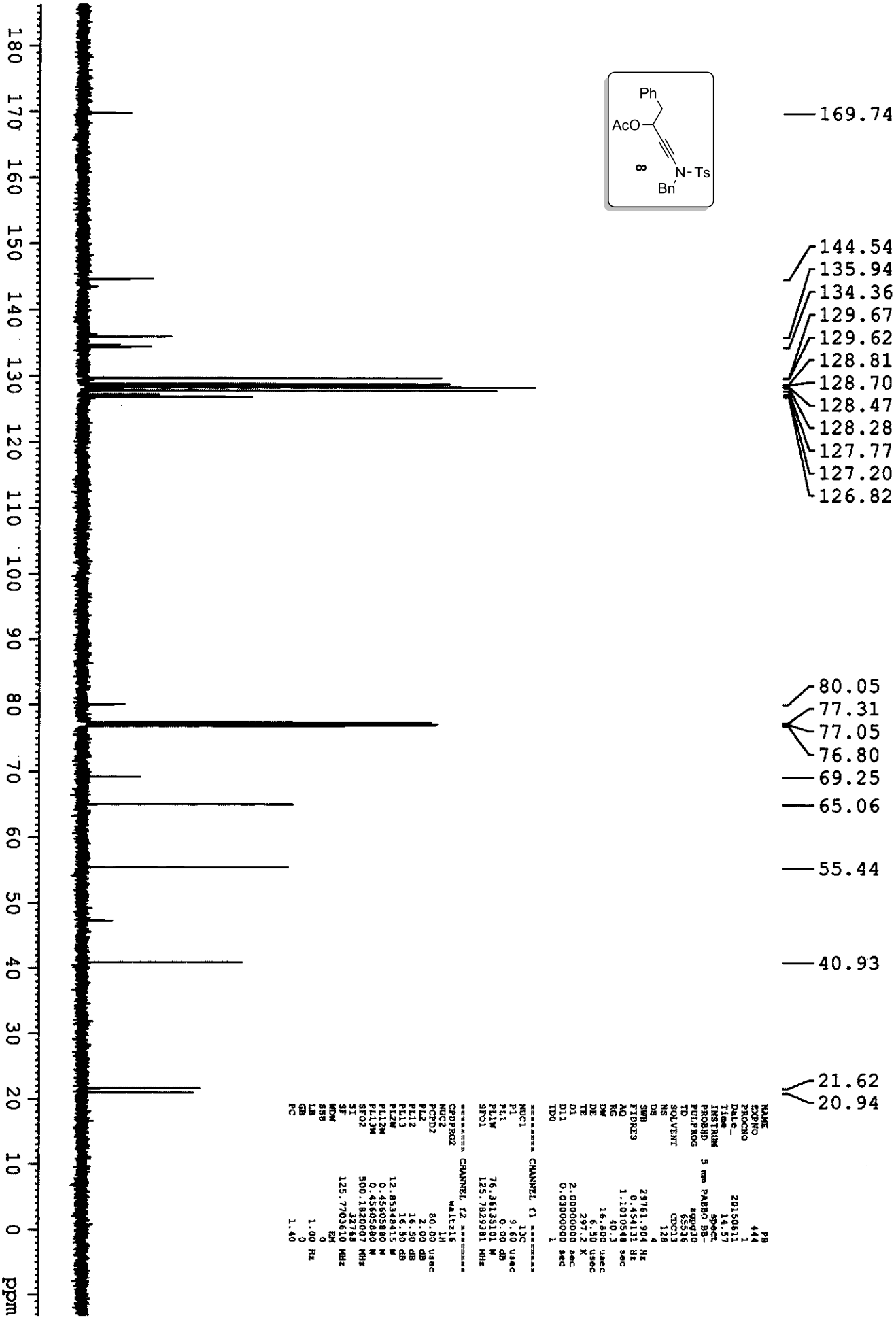

6d
Ts
N
Bn
H
144.94
143.66
143.42
139.98
139.61
136.62
136.57
136.12
135.99
135.38
134.64
134.31
134.10
129.82
129.78
129.72
129.62
129.32
129.20
128.85
128.83
128.75
128.58
128.51
128.37
128.12
128.07
127.98
127.95
127.87
127.79
127.75
127.59
127.21
118.52
117.33
101.64
95.51
77.39
77.07
76.75
72.41
70.35
52.98
51.71
21.63
21.58
8.56
8.45
0.13
-0.20
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

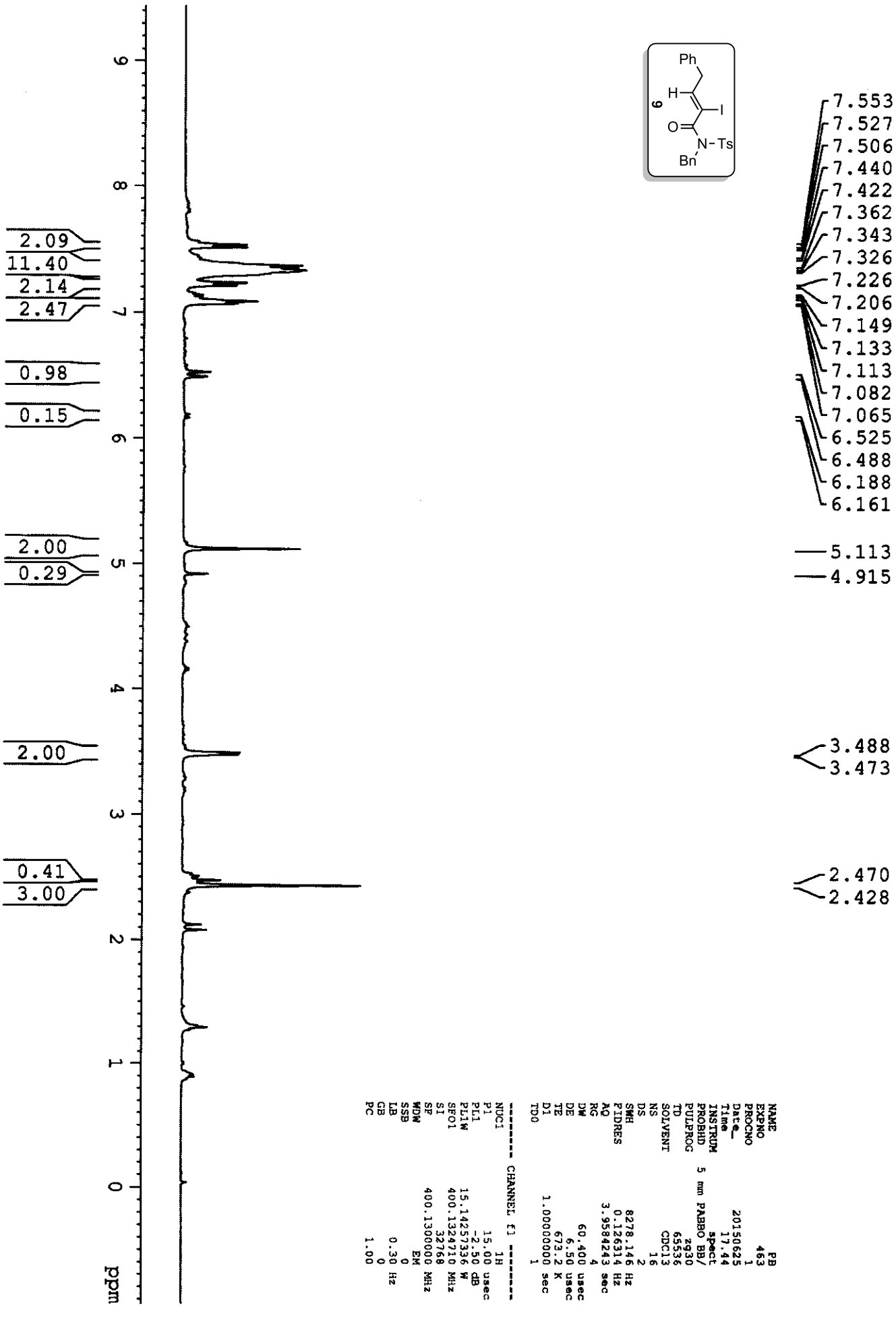

7.838
7.819
7.581
7.564
7.544
7.516
7.504
7.499
7.486
7.468
7.450
7.415
7.397
7.378
7.287
Cl
N
Ph
7
NAME 2B
EXPNO 382
PROCNO 1
Date_ 20150423
Time 21.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.07
1.14
2.66
0.63
0.24

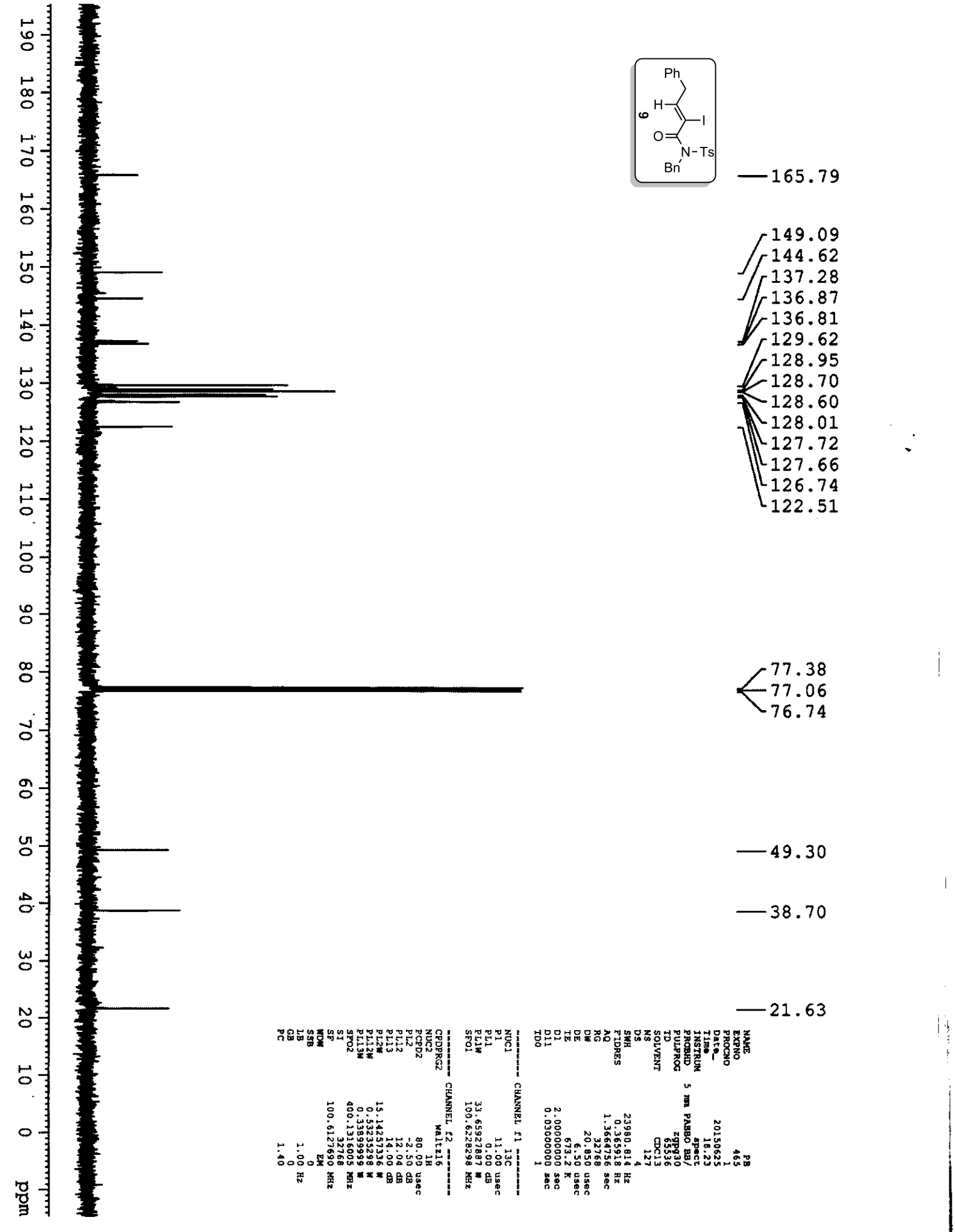


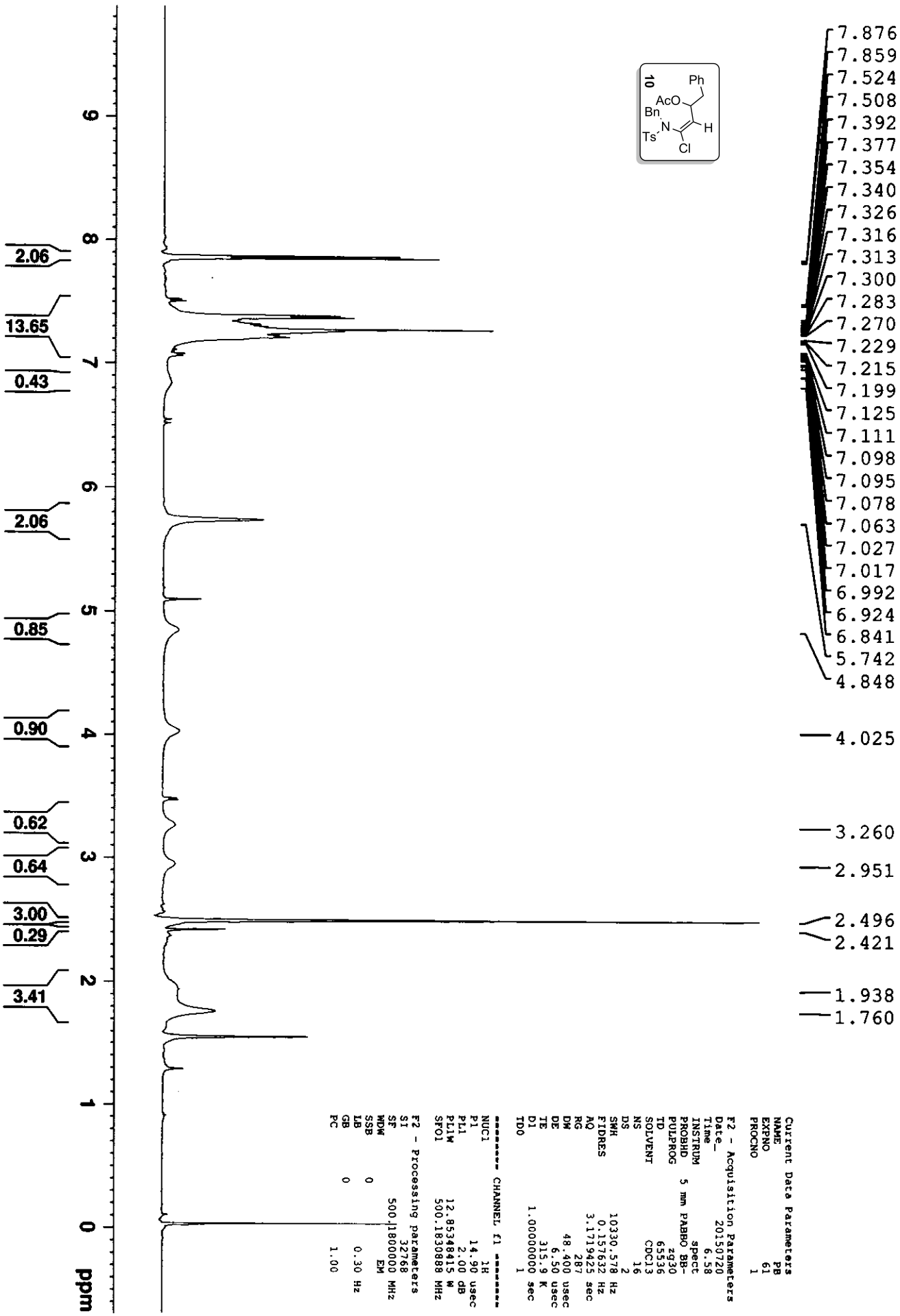
NAME	PT
EXPNO	261
PROCNO	1
Date_	20150424
Time	0.07
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	7168
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	1625.5
DW	20.850 usec
DE	6.50 usec
TE	673.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
NUC1	13C
P1	11.00 usec
PL1	0.00 dB
PL1W	33.65927887 W
SFO1	100.6228298 MHz
-------- CHANNEL f2 --------	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.50 dB
PL12	12.04 dB
PL13	14.00 dB
PL2W	15.14257336 W
PL12W	0.53235298 W
PL13W	0.33899999 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

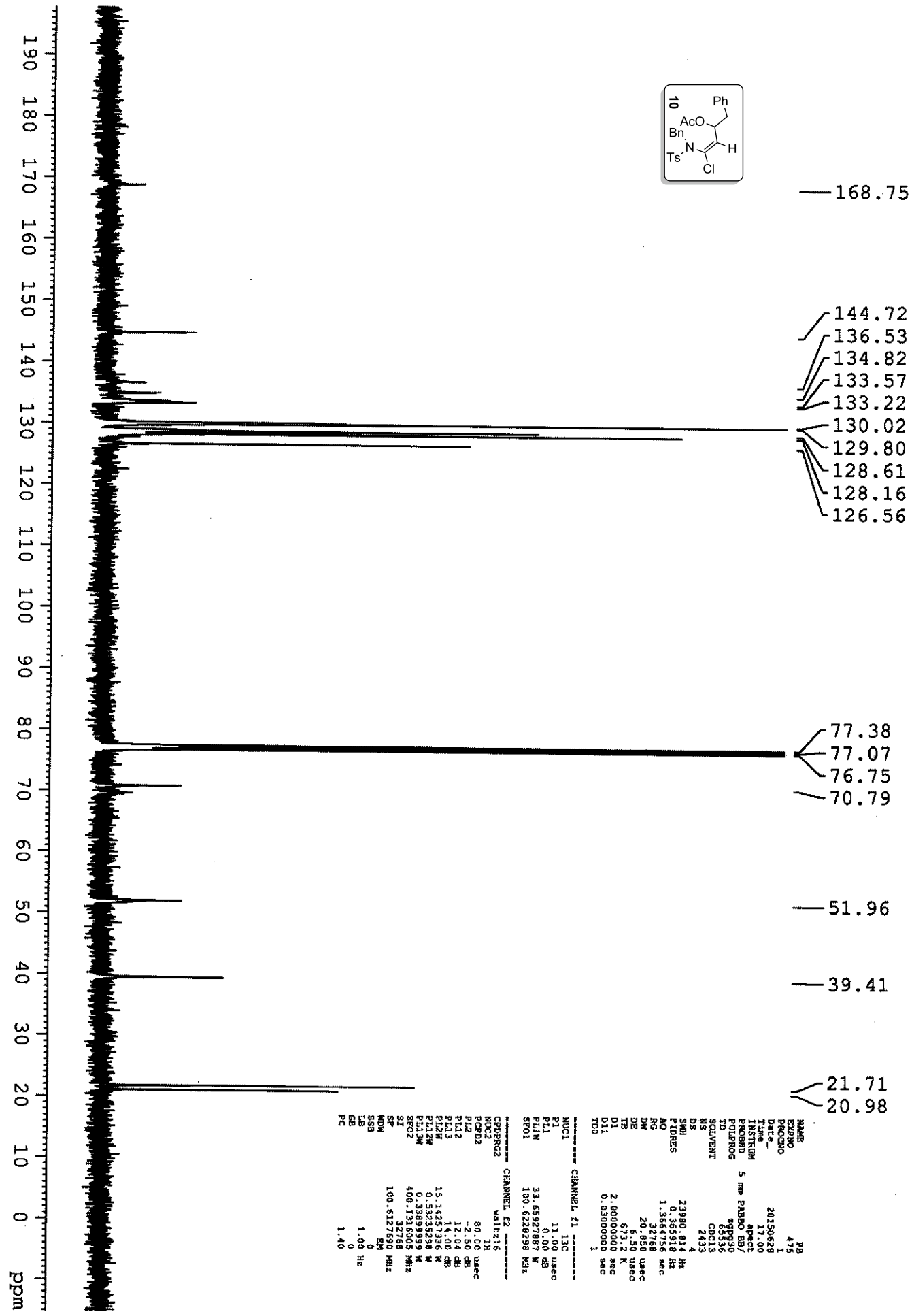

Ph
AcO
8
N-Ts
Bn
7.799
7.782
7.660
7.644
7.339
7.316
7.312
7.306
7.302
7.294
7.287
7.282
7.276
7.260
7.254
7.250
7.242
7.239
7.231
7.224
7.144
7.137
7.130
7.126
5.603
5.590
5.576
4.536
4.508
4.403
4.375
3.022
3.009
2.995
2.982
2.960
2.946
2.933
2.918
2.453
2.008
1.68
2.96
1.79
3.16
0.94
1.17
1.65
0.83
0.87
0.86
1.78
3.00
2.52
ppm
NAME PB
EXPNO 446
PROCNO 1
Date_ 20150611
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 2.00 dB
PL1W 12.85348415 W
SFO1 500.1830888 MHz
SI 32768
SF 500.1800000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

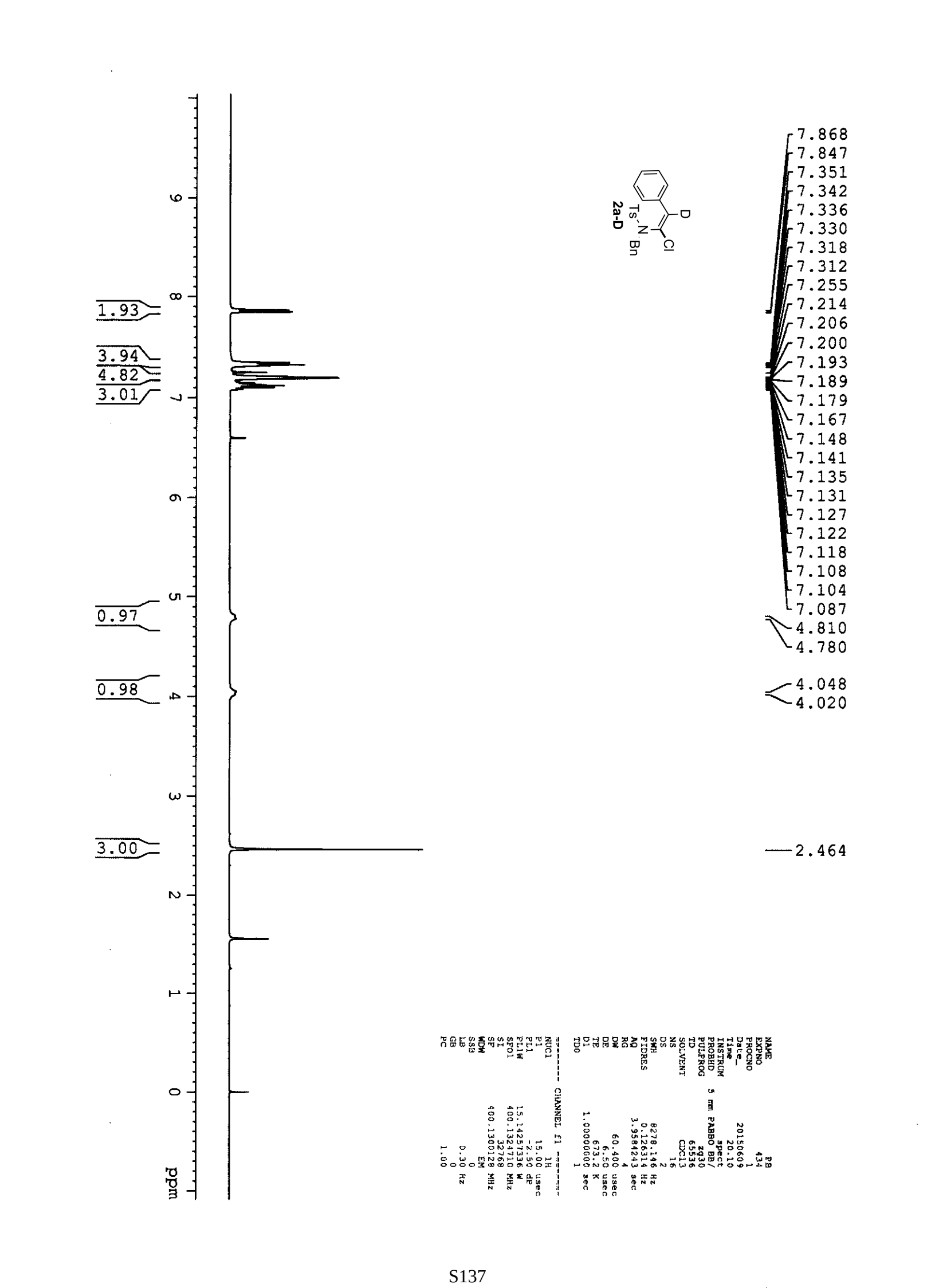

Ph
AcO
8
N-Ts
Bn
169.74
144.54
135.94
134.36
129.67
129.62
128.81
128.70
128.47
128.28
127.77
127.20
126.82
80.05
77.31
77.05
76.80
69.25
65.06
55.44
40.93
21.62
20.94
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

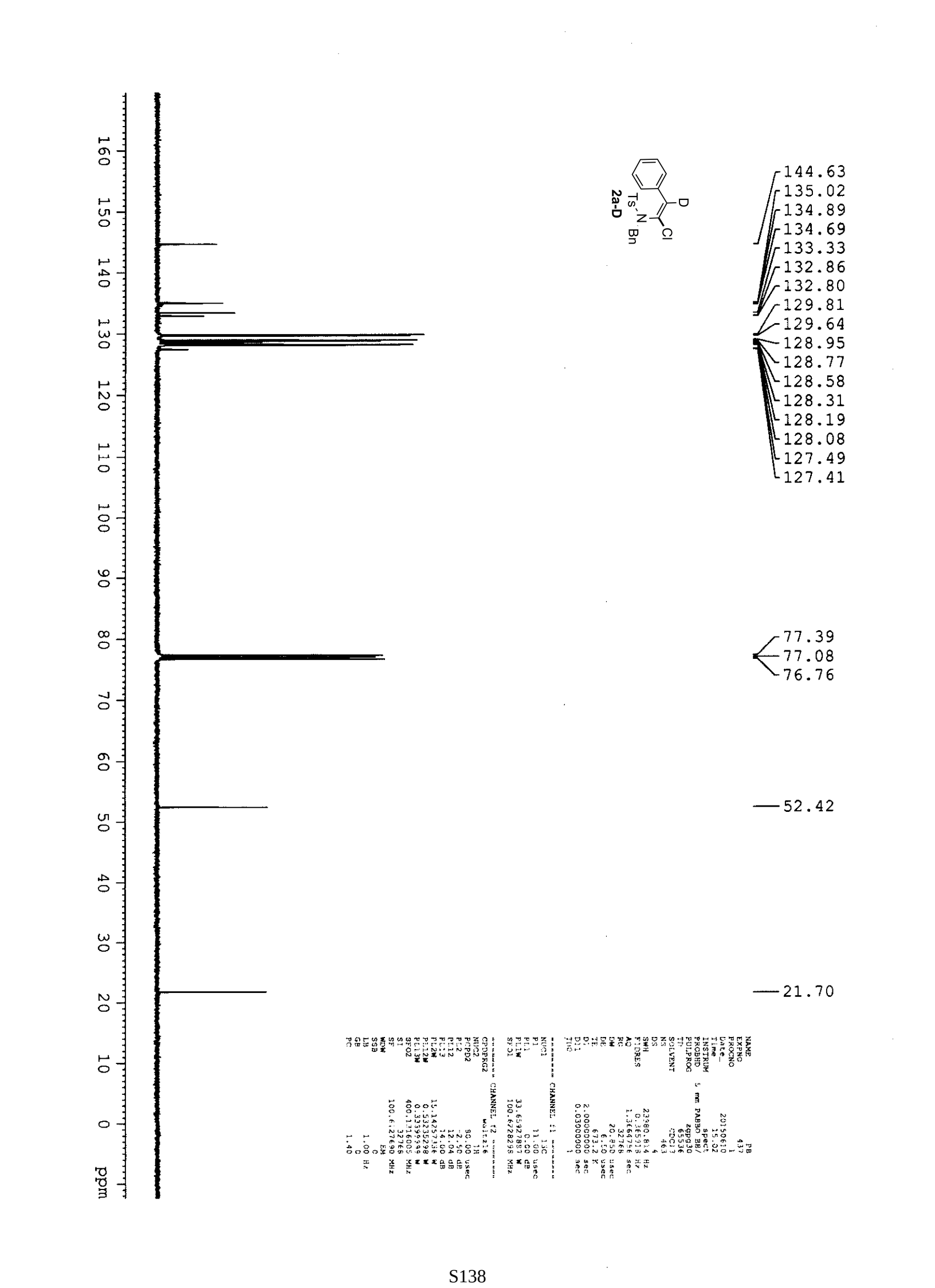

Ph
H
I
O
N-Ts
Bn
9
7.553
7.527
7.506
7.440
7.422
7.362
7.343
7.326
7.226
7.206
7.149
7.133
7.113
7.082
7.065
6.525
6.488
6.188
6.161
5.113
4.915
3.488
3.473
2.470
2.428
2.09
11.40
2.14
2.47
0.98
0.15
2.00
0.29
2.00
0.41
3.00
ppm
NAME PB
EXPNO 463
PROCNO 1
Date_ 20150625
Time 17.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


Ph
H
I
O
N-Ts
Bn
9
165.79
149.09
144.62
137.28
136.87
136.81
129.62
128.95
128.70
128.60
128.01
127.72
127.66
126.74
122.51
77.38
77.06
76.74
49.30
38.70
21.63
ppm


10
Ph
AcO
Bn
N
Ts
H
Cl
7.876
7.859
7.524
7.508
7.392
7.377
7.354
7.340
7.326
7.316
7.313
7.300
7.283
7.270
7.229
7.215
7.199
7.125
7.111
7.098
7.095
7.078
7.063
7.027
7.017
6.992
6.924
6.841
5.742
4.848
4.025
3.260
2.951
2.496
2.421
1.938
1.760
2.06
13.65
0.43
2.06
0.85
0.90
0.62
0.64
3.00
0.29
3.41
ppm
Current Data Parameters
NAME PB
EXPNO 61
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150720
Time_ 6.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 287
DW 48.400 usec
DE 6.50 usec
TE 315.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 2.00 dB
PL1W 12.85348815 W
SFO1 500.1830888 MHz
F2 - Processing parameters
SI 32768
SF 500.1800000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


10
Ph
AcO
Bn
N
Ts
H
Cl
168.75
144.72
136.53
134.82
133.57
133.22
130.02
129.80
128.61
128.16
126.56
77.38
77.07
76.75
70.79
51.96
39.41
21.71
20.98
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.868
7.847
7.351
7.342
7.336
7.330
7.318
7.312
7.255
7.214
7.206
7.200
7.193
7.189
7.179
7.167
7.148
7.141
7.135
7.131
7.127
7.122
7.118
7.108
7.104
7.087
4.810
4.780
4.048
4.020
2.464
1.93
3.94
4.82
3.01
0.97
0.98
3.00
ppm
2a-D
D
Cl
Ts
N
Bn
NAME pB
EXPNO 434
PROCNO 1
Date_ 20150609
Time 20.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 4
DW 60.400 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.50 dB
PL1W 15.14257336 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


144.63
135.02
134.89
134.69
133.33
132.86
132.80
129.81
129.64
128.95
128.77
128.58
128.31
128.19
128.08
127.49
127.41
77.39
77.08
76.76
52.42
21.70
D
Cl
Ts
N
Bn
2a-D
ppm