Iron-Catalyzed Alkylations of Aryl Sulfamates and Carbamates

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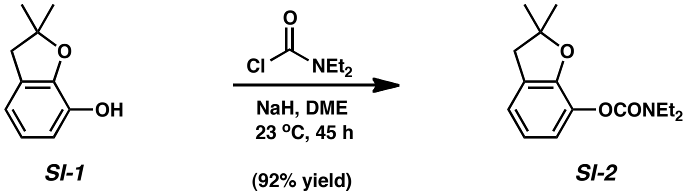
Supporting Information – Table of Contents

Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen using anhydrous solvents (either freshly distilled or passed through activated alumina columns). Unless otherwise stated, commercially obtained reagents were used as received. Commercial Grignard reagents were titrated,[1] and then used without further purification. Non-commercially available Grignard reagents were synthesized following protocols specified in Section C of the SI. Anhydrous $FeCl_2$ (98%) was obtained from Strem Chemicals and SIMes•HCl (**3**) was obtained from either Sigma Aldrich or Materia Inc. Reaction temperatures were controlled using an IKAmag temperature modulator, and unless stated otherwise, reactions were performed at room temperature (rt, approximately 23 °C). Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm) and visualized using a combination of UV, anisaldehyde, ceric ammonium molybdate, iodine, vanillin, and potassium permanganate staining. Silicycle Silia*Flash* P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^{1}H NMR spectra were recorded on Bruker spectrometers (at 300, 400, 500, and 600 MHz) and are reported relative to deuterated solvent signals. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. ^{13}C NMR spectra were recorded on Bruker Spectrometers (at 75, 125, and 150 MHz). Data for ^{13}C NMR spectra are reported in terms of chemical shift. IR spectra were recorded on a Perkin-Elmer 100 spectrometer and are reported in terms of frequency of absorption (cm^{-1}). Melting points are uncorrected and were obtained on a Laboratory Devices Mel-Temp II instrument. High resolution mass spectra were obtained from either the UC Los Angeles (ICT Premier) or UC Irvine Mass Spectrometry Facility. Inductively coupled plasma optical emission spectroscopy was obtained on a TJA Radial IRIS 1000 instrument and reported in parts per million (ppm).

Experimental Procedures.

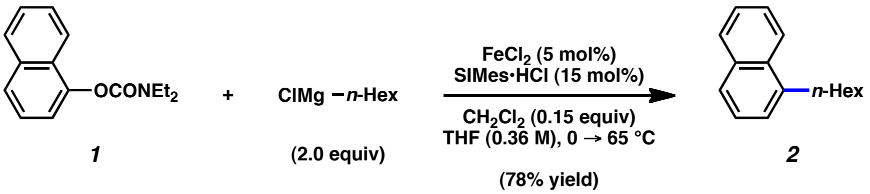
A. Synthesis of Aryl Carbamate and Sulfamate Substrates

Note: Supporting information for the synthesis of the aryl sulfamates and carbamate shown in Tables 1, 2, and 3 have previously been reported,[2] with the exception of the benzofuran carbamate **SI-2** shown below.



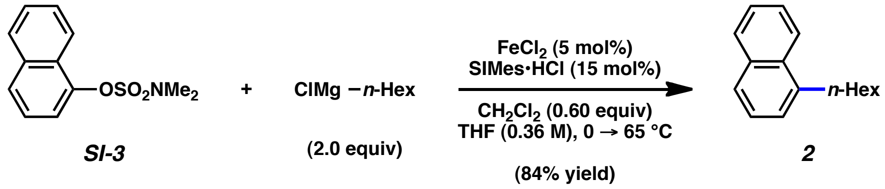
SI-2 (Table 2, entry 6b). A round bottom flask was charged with NaH (0.88 g, 21.92 mmol, 1.2 equiv, 60% dispersion in oil). A solution of dihydrobenzofuranol **SI-1** (3.0 mL, 18.27 mmol, 1 equiv) in DME (40 mL) was added dropwise via cannula to the NaH. A solution of *N,N*-diethylcarbamoyl chloride (2.2 mL, 17.36 mmol, 0.95 equiv) in DME (30 mL) was then added dropwise via cannula to the reaction vessel. The reaction was stirred at 23 °C for 45 h. After quenching with water (10 mL), the volatiles were removed under reduced pressure, and then Et_2O (30 mL) and H_2O (20 mL) were added. The layers were separated, and the organic layer was washed successively with 1 M KOH (20 mL) and H_2O (20 mL). The combined aqueous layers were extracted with Et_2O (3 x 20 mL). The combined organic layers were then washed with brine (20 mL), dried over $MgSO_4$, and evaporated to dryness. The crude residue was purified by flash chromatography (7:1 Hexanes:EtOAc) to yield carbamate **SI-2** as a clear oil (1.0 g, 92% yield). R_f 0.4 (100% Benzene); ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.95 (dd, J = 7.5, 1.5, 1H), 6.91 (app d, J = 8.0, 1H), 6.76 (app t, J = 7.5, 1H), 3.44–3.38 (m, 4H), 3.01 (s, 2H), 1.47 (s, 6H), 1.26–1.20 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.8, 150.6, 135.4, 129.3, 121.9, 121.8, 119.9, 87.8, 43.1, 42.3, 42.1, 28.2, 14.1, 13.4; IR (film): 2974, 2934, 1718, 1416, 1152, 1052 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{15}H_{21}NO_3Na$, 286.1419; found, 286.1420.

B. Cross-Couplings of Aryl Carbamates and Sulfamates

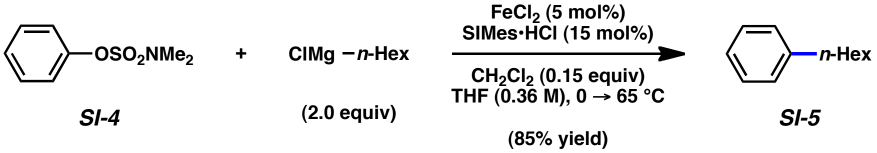


Representative Procedure (coupling of naphthylcarbamate 1, Table 1, entry 1b) is used as an example). 2. A flame-dried 4-mL reaction vial was charged with $FeCl_2$ (2.6 mg, 0.02 mmol, 5 mol%), SIMes•HCl (20.6 mg, 0.06 mmol, 15 mol%), the carbamate substrate **1** (97.3 mg, 0.40 mmol, 1 equiv) and a magnetic stir bar, all in a glove box. The vessel was removed from the glove box, and then placed under an atmosphere of N_2 on the bench. THF (0.62 mL) and CH_2Cl_2 (3.9 μL, 0.06 mmol, 0.15 equiv) were added and the resulting suspension was cooled to 0 °C. Next, a solution of *n*-hexylmagnesium chloride in THF (1.6 M in THF, 0.5 mL, 0.8 mmol, 2.0 equiv) was added slowly. The vial was sealed and heated to 65 °C for 3 h. After cooling the reaction vessel to 23 °C, the reaction was quenched with saturated aqueous NH_4Cl (2 mL). The layers were separated; the aqueous layer was extracted with EtOAc (2 x 3 mL). The combined organic layers were then washed with brine (3 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude residue was purified by flash chromatograpy (100% Pentanes) to yield 1-*n*-hexylnaphthylene **2** as a colorless oil (84.9 mg, 78% yield) and naphthalene (4.6 mg, 9% yield) as an inseparable mixture. R*f* 0.66 (100% Pentanes). Spectral data match those previously reported.[3]

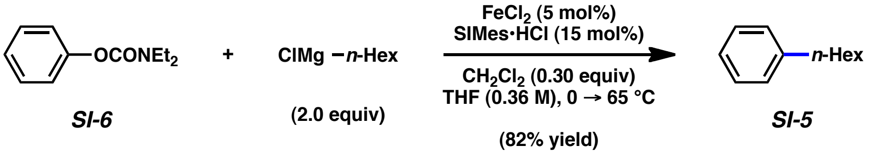
Any modifications of the conditions shown in this representative procedure are specified in the following schemes, which depict all of the results shown in Tables 1, 2, and 3. Note: Reactions can be set up outside of the glove box using a suspension of anhydrous $FeCl_2$ in THF and CH_2Cl_2 stored under inert atmosphere.



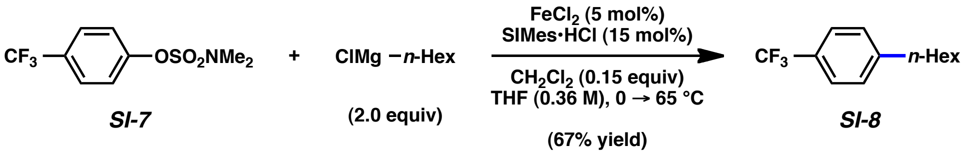
2 (Table 1, entry 1a). Purification by flash chromatography (100% Pentanes) afforded cross-coupled product **2** (84% yield) and naphthalene (16% yield) as an inseparable mixture. R*f* 0.61 (100% Pentanes). Spectral data match those previously reported.[3]



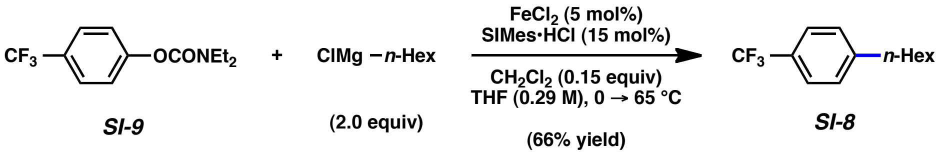
SI-5 (Table 1, entry 2a). Purification by flash chromatography (100% Hexanes) yielded cross-coupled product **SI-5** (85% yield) as a clear oil. R*f* 0.77 (100% Hexanes). Spectral data match those previously reported.[4]



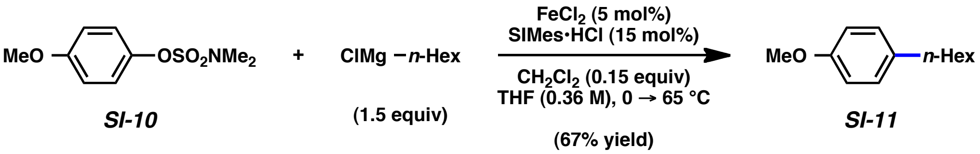
SI-5 (Table 1, entry 2b). Purification by flash chromatography (100% Hexanes) produced alkylated product **SI-5** (82% yield) as a clear oil. R*f* 0.77 (100% Hexanes). Spectral data match those previously reported.[4]



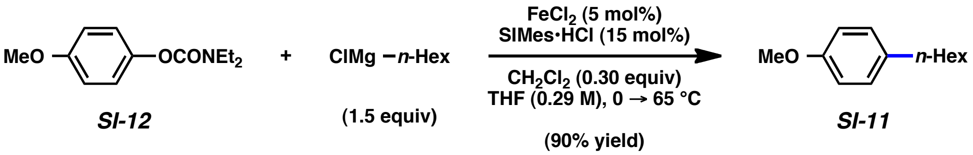
SI-8 (Table 1, entry 3a). Purification by flash chromatography (1:1 Hexanes:Benzene) afforded cross-coupled product **SI-8** (67% yield) as a clear oil. R*f* 0.88 (1:1 Pentanes:Benzene). Spectral data match those previously reported.[5]



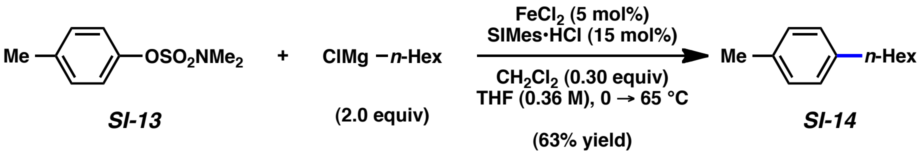
SI-8 (Table 1, entry 3b). Purification by flash chromatography (1:1 Hexanes:Benzene) supplied cross-coupled product **SI-8** (66% yield) as a clear oil. R*f* 0.88 (1:1 Pentanes:Benzene). Spectral data match those previously reported.[5]



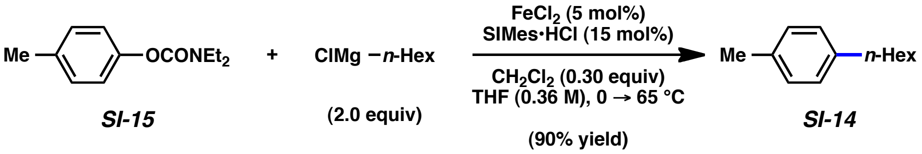
SI-11 (Table 1, entry 4a). Purification by flash chromatography (20:1 Pentanes:Benzene) yielded cross-coupled product **SI-11** (67% yield) as a clear oil. R*f* 0.32 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[6]



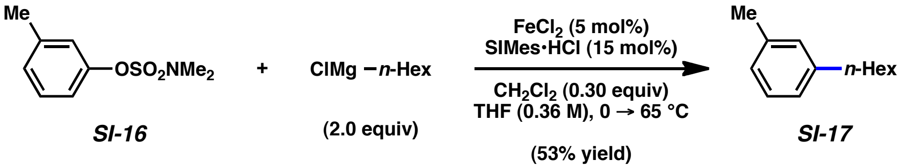
SI-11 (Table 1, entry 4b). Purification by flash chromatography (20:1 Pentanes:Benzene) produced alkylated product **SI-11** (90% yield) as a clear oil. R*f* 0.32 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[6]



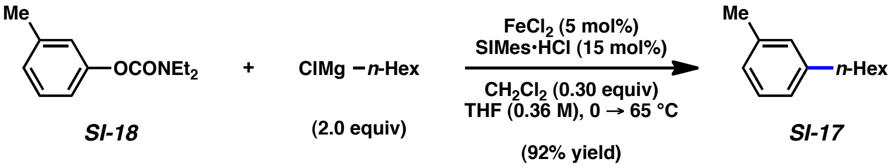
SI-14 (Table 1, entry 5a). Purification by flash chromatography (20:1 Pentanes:Benzene) afforded cross-coupled product **SI-14** (63% yield) as a clear oil. R*f* 0.84 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[6]



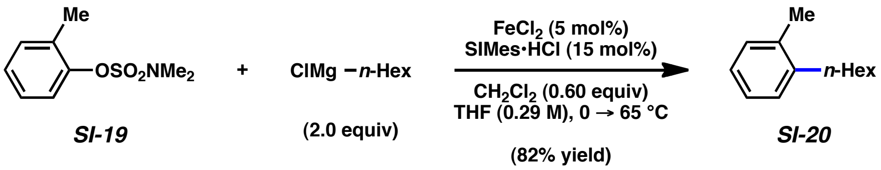
SI-14 (Table 1, entry 5b). Purification by flash chromatography (20:1 Pentanes:Benzene) supplied cross-coupled product **SI-14** (90% yield) as a clear oil. R*f* 0.84 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[6]



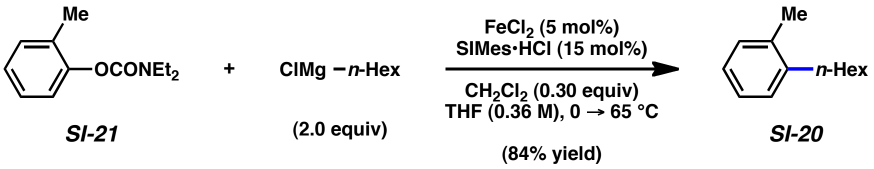
SI-17 (Table 1, entry 6a). Purification by flash chromatography (20:1 Pentanes:Benzene) yielded cross-coupled product **SI-17** (53% yield) as a clear oil. R*f* 0.84 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[7]



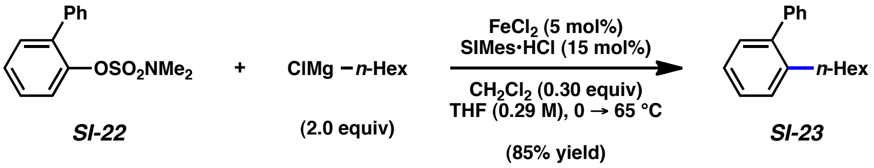
SI-17 (Table 1, entry 6b). Purification by flash chromatography (20:1 Pentanes:Benzene) produced alkylated product **SI-17** (92% yield) as a clear oil. R*f* 0.84 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[7]



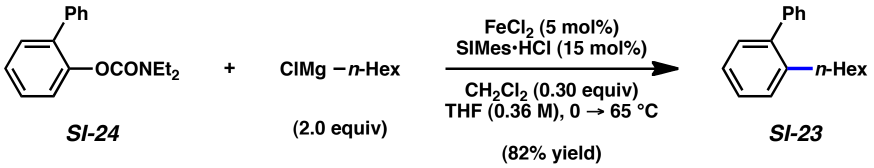
SI-20 (Table 2, entry 1a). Purification by flash chromatography (20:1 Pentanes:Benzene) afforded cross-coupled product **SI-20** (82% yield) as a clear oil. R_f 0.68 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[4]



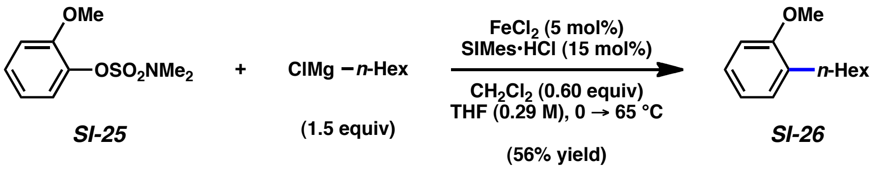
SI-20 (Table 2, entry 1b). Purification by flash chromatography (20:1 Pentanes:Benzene) supplied cross-coupled product **SI-20** (84% yield) as a clear oil. R_f 0.68 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[4]



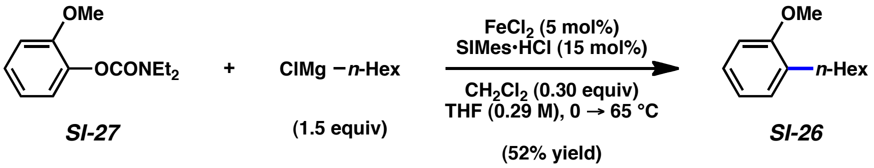
SI-23 (Table 2, entry 2a). Purification by flash chromatography (20:1 Pentanes:Benzene) yielded cross-coupled product **SI-23** (85% yield) as a clear oil. R_f 0.79 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[8]



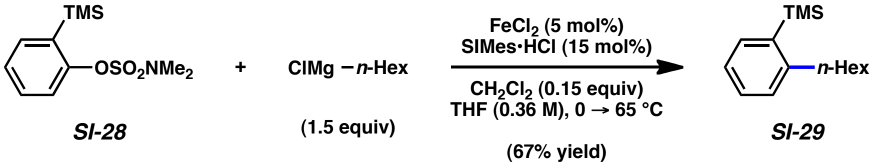
SI-23 (Table 2, entry 2b). Purification by flash chromatography (20:1 Pentanes:Benzene) produced product **SI-23** (82% yield) as a clear oil. R_f 0.79 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[8]



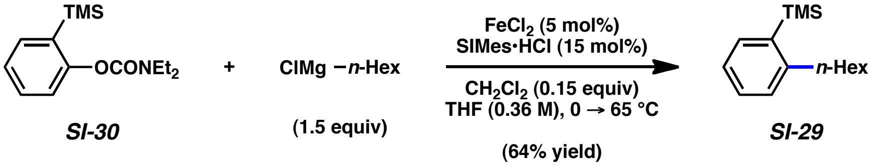
SI-26 (Table 2, entry 3a). Purification by flash chromatography (20:1 Pentanes:Benzene) afforded cross-coupled product **SI-26** (56% yield) as a clear oil. R$_f$ 0.42 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[9]



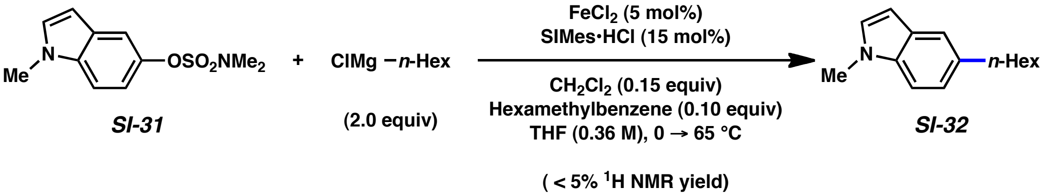
SI-26 (Table 2, entry 3b). Purification by flash chromatography (20:1 Pentanes:Benzene) supplied cross-coupled product **SI-26** (52% yield) as a clear oil. R$_f$ 0.42 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[9]



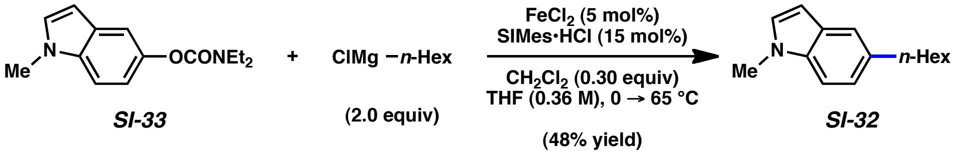
SI-29 (Table 2, entry 4a). Purification by flash chromatography (100% Pentanes) yielded cross-coupled product **SI-29** (67% yield) as a clear oil. R$_f$ 0.80 (100% Pentanes). ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.47 (dd, *J* = 7.4, 1.3, 1H), 7.31 (td, *J* = 7.5, 1.4, 1H), 7.21 (d, *J* = 7.6, 1H), 7.16 (td, *J* = 7.3, 1.2, 1H), 2.70 (t, *J* = 8.2, 2H); 1.65–1.57 (m, 2H), 1.45–1.38 (m, 2H), 1.34–1.31 (m, 4H), 0.90 (t, *J* = 6.9, 3H), 0.32 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 149.0, 138.0, 134.6, 129.3, 128.7, 125.1, 36.6, 32.8, 32.0, 29.8, 22.8, 14.2, 0.6; IR (film): 3054, 2955, 2928, 2858, 1249, 849, 836 cm^{-1}; HRMS-EI (*m/z*) [M]$^+$ calcd for $C_{15}H_{26}Si$, 234.1804; found 234.1792.



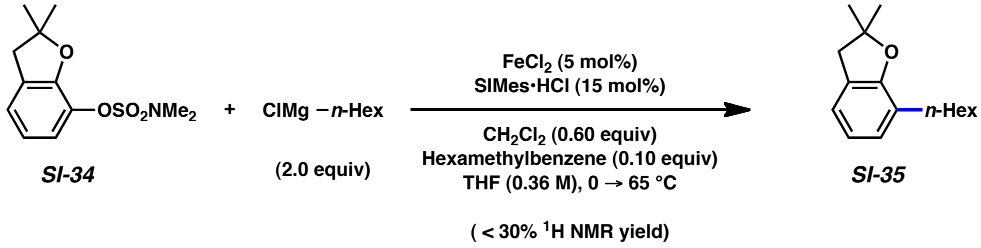
SI-29 (Table 2, entry 4b). Purification by flash chromatography (100% Pentanes) gernerated alkylated product **SI-29** (64% yield) as a clear oil. R$_f$ 0.80 (100% Pentanes). ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.47 (dd, *J* = 7.4, 1.3, 1H), 7.31 (td, *J* = 7.5, 1.4, 1H), 7.21 (d, *J* = 7.6, 1H), 7.16 (td, *J* = 7.3, 1.2, 1H), 2.70 (t, *J* = 8.2, 2H); 1.65–1.57 (m, 2H), 1.45–1.38 (m, 2H), 1.34–1.31 (m, 4H), 0.90 (t, *J* = 6.9, 3H), 0.32 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 149.0, 138.0, 134.6, 129.3, 128.7, 125.1, 36.6, 32.8, 32.0, 29.8, 22.8, 14.2, 0.6; IR (film): 3054, 2955, 2928, 2858, 1249, 849, 836 cm^{-1}; HRMS-EI (*m/z*) [M]$^+$ calcd for $C_{15}H_{26}Si$, 234.1804; found 234.1792.



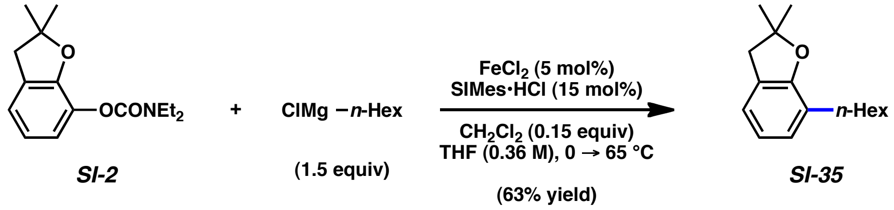
SI-32 (Table 2, entry 5a). The yield was determined by ^{1}H NMR analysis with hexamethylbenzene as internal standard.



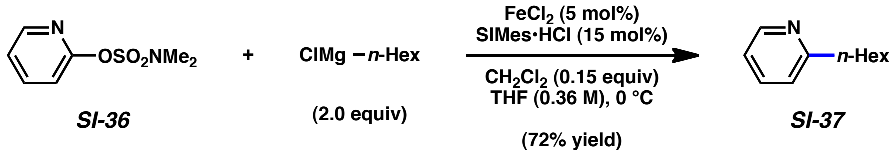
SI-32 (Table 2, entry 5b). Purification by flash chromatography (10:1 Hexanes:EtOAc) afforded cross-coupled product **SI-32** (48% yield) as a clear oil. R_f 0.59 (10:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.42 (d, J = 0.7, 1H), 7.24 (d, J = 8.4, 1H), 7.07 (dd, J = 8.4, 1.6, 1H), 7.01 (d, J = 3.1, 1H), 6.41 (dd, J = 3.1, 0.7, 1H), 3.77, (s, 3H), 2.70 (t, 7.6, 2H), 1.66 (quintet, J = 7.5, 2H), 1.40–1.27 (m, 6H), 0.89 (t, J = 6.8, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 135.4, 133.9, 128.9, 128.8, 122.7, 120.1, 109.0, 100.5, 36.2, 33.0, 32.5, 32.0, 29.2, 22.8, 14.3; IR (film): 2955, 2922, 2853, 1513, 1491, 1244 cm^{-1}; HRMS-ESI (m/z) $[M + H]^+$ calcd for $C_{15}H_{22}N$, 216.1752; found 216.1755.



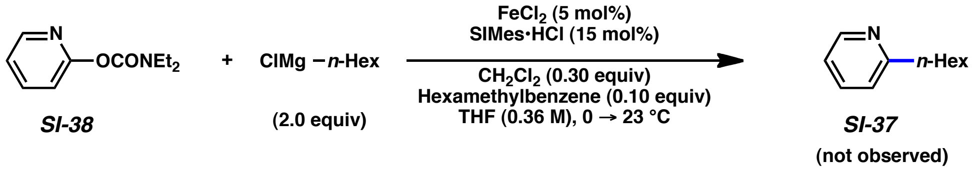
SI-35 (Table 2, entry 6a). The yield was determined by ^{1}H NMR analysis with hexamethylbenzene as internal standard.



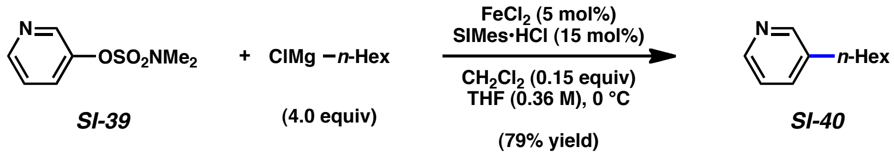
SI-35 (Table 2, entry 6b). Purification by flash chromatography (20:1 Pentanes:Benzene) afforded cross-coupled product **SI-35** (63% yield) as a clear oil. R_f 0.78 (100% Benzene). ^{1}H NMR (500 MHz, CDCl$_3$): δ 6.97 (dd, J = 7.2, 1.0, 1H), 6.93 (d, J = 7.5, 1H), 6.74 (t, J = 7.4, 1H), 2.99 (s, 2H), 2.54 (t, J = 7.6, 2H), 1.59 (quintet, J = 7.5, 2H), 1.46 (s, 6H), 1.35–1.26 (m, 6H), 0.91–0.86 (m, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 157.3, 128.2, 126.6, 124.8, 122.6, 119.8, 85.9, 43.3, 31.9, 29.9, 29.7, 29.2, 28.4, 22.8, 14.3; IR (film): 2955, 2927, 2856, 1597, 1454, 1368, 1294, 1263, 1137 cm^{-1}; HRMS-EI (*m/z*) $[M]^+$ calcd for $C_{16}H_{24}O$, 232.1827; found 232.1820.



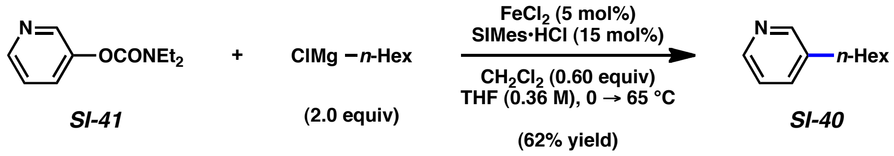
SI-36 (Table 2, entry 7a). Purification by flash chromatography (2:1 Hexanes:EtOAc) supplied cross-coupled product **SI-37** (72% yield) as a clear oil. R*f* 0.69 (2:1 Hexanes:EtOAc). Spectral data match those previously reported.[10]



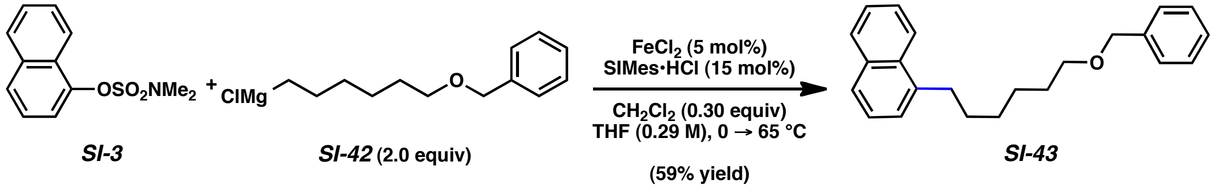
SI-37 (Table 2, entry 7b). **SI-37** was not observed, as determined by ^{1}H NMR analysis with hexamethylbenzene as internal standard.



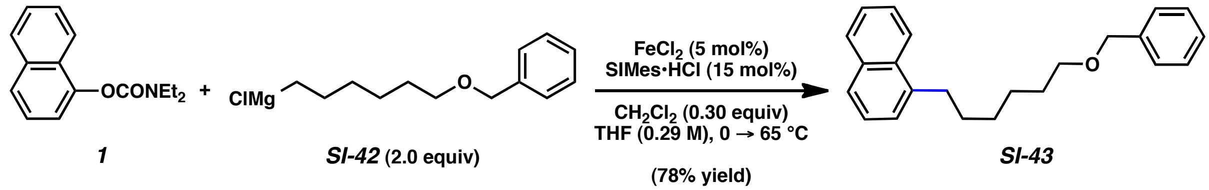
SI-40 (Table 2, entry 8a). Purification by flash chromatography (4:1 Hexanes:EtOAc) afforded cross-coupled product **SI-40** (79% yield) as a clear oil. R*f* 0.45 (2:1 Hexanes:EtOAc). Spectral data match those previously reported.[11]



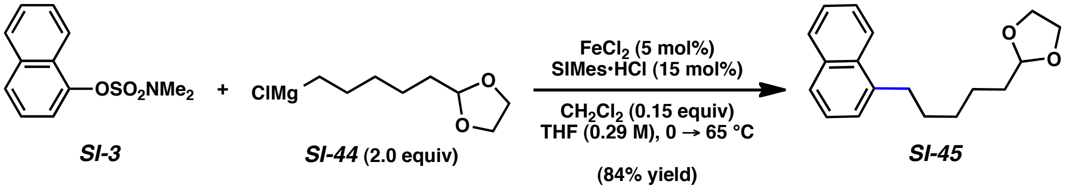
SI-40 (Table 2, entry 8b). Purification by flash chromatography (4:1 Hexanes:EtOAc) supplied cross-coupled product **SI-40** (62% yield) as a clear oil. R*f* 0.45 (2:1 Hexanes:EtOAc). Spectral data match those previously reported.[11]



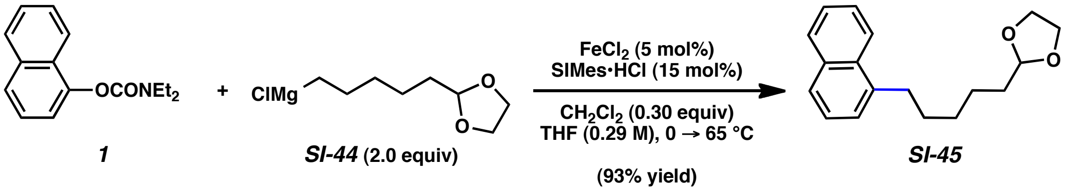
SI-43 (Table 3, entry 1a). Purification by flash chromatography (50:1:1 Hexanes:Benzene:EtOAc) yielded cross-coupled product **SI-43** (59% yield) as a yellow oil. R$_f$ 0.05 (50:1:1 Hexanes:Benzene:EtOAc). ^{1}H NMR (600 MHz, CDCl$_3$): δ 8.05 (d, *J* = 8.2, 1H), 7.86 (d, *J* = 7.6, 1H), 7.71 (d, *J* = 8.2, 1H), 7.25–7.46 (m, 2H), 7.40 (dd, *J* = 8.0, 7.2, 1H), 7.37–7.31 (m, 5H), 7.31–7.27 (m, 1H), 4.51 (s, 2H), 3.48 (t, *J* = 6.7, 2H), 3.08 (t, *J* = 7.9, 2H), 1.78 (quintet, *J* = 7.4, 2H), 1.65 (quintet, *J* = 6.7, 2H), 1.50–1.44 (m, 4H); ^{13}C NMR (150 MHz, CDCl$_3$): δ 139.0, 138.8, 134.0, 132.0, 128.8, 128.5, 127.7, 127.6, 126.5, 125.9, 125.7, 125.6, 125.5, 124.0, 73.0, 70.5, 33.2, 30.9, 29.9, 29.7, 26.2; IR (film): 3032, 2931, 2855, 1596, 1509, 1495, 1453, 1362, 1215, 1097, 1028 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{23}H_{26}OH$, 320.2096; found 320.2119.



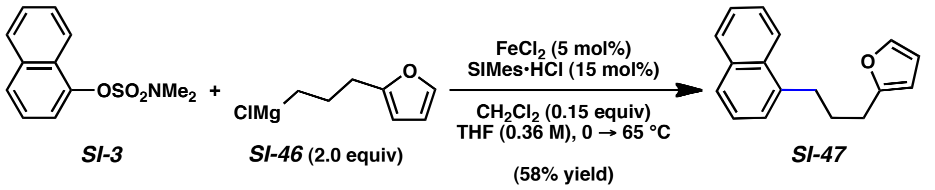
SI-43 (Table 3, entry 1b). Purification by flash chromatography (50:1:1 Hexanes:Benzene:EtOAc) generated alkylated product **SI-43** (78% yield) as a yellow oil. R$_f$ 0.05 (50:1:1 Hexanes:Benzene:EtOAc). See spectral data above.



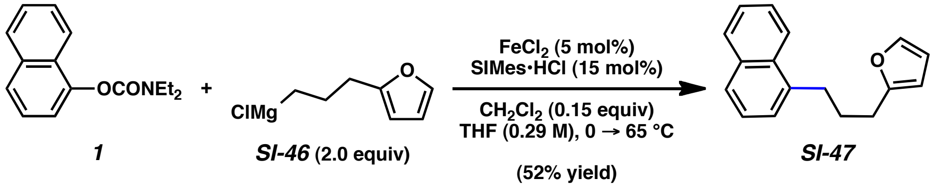
SI-45 (Table 3, entry 2a). Purification by flash chromatography (9:1 Hexanes:EtOAc) afforded cross-coupled product **SI-45** (84% yield) as a colorless oil. R$_f$ 0.27 (9:1 Hexanes:EtOAc). ^{1}H NMR (600 MHz, CDCl$_3$): δ 8.04 (d, *J* = 8.4, 1H), 7.86 (d, *J* = 7.9, 1H), 7.71 (d, *J* = 8.1, 1H), 7.52–7.46 (m, 2H), 7.39 (dd, *J* = 8.1, 7.1, 1H), 7.32 (d, *J* = 6.9, 1H), 4.86 (t, *J* = 4.9, 1H), 4.00–4.94 (m, 2H), 3.88–3.83 (m, 2H), 3.08 (t, *J* = 7.8, 2H), 1.81–1.76 (m, 2H), 1.71–1.67 (m, 2H), 1.55–1.47 (m, 4H); ^{13}C NMR (150 MHz, CDCl$_3$): δ 139.0, 134.1, 132.1, 128.9, 126.6, 126.0, 125.8, 125.6, 125.5, 124.0, 104.8, 65.0, 34.0, 33.1, 30.9, 29.8, 24.1; IR (film): 2934, 2859, 1395, 1129, 1032 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{18}H_{22}O_2H$, 271.1698; found 271.1695.



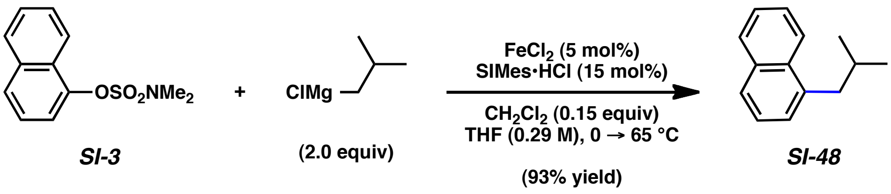
SI-45 (Table 3, entry 2b). Purification by flash chromatography (9:1 Hexanes:EtOAc) supplied cross-coupled product **SI-45** (93% yield) as a yellow oil. R$_f$ 0.27 (9:1 Hexanes:EtOAc). See spectral data above.



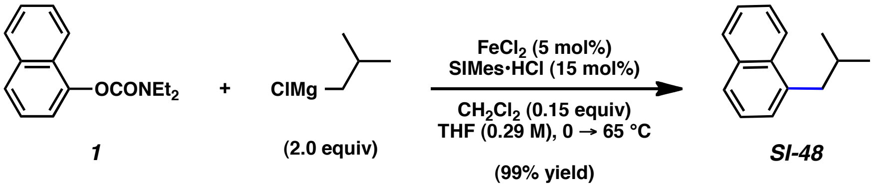
SI-47 (Table 3, entry 3a). Purification by flash chromatography (50:1:1 Hexanes:Benzene:EtOAc) afforded cross-coupled product **SI-47** (58% yield) as a yellow oil. R$_f$ 0.27 (50:1:1 Hexanes:Benzene:EtOAc). ^{1}H NMR (600 MHz, CDCl$_3$): δ 8.02 (d, *J* = 8.3, 1H), 7.88 (d, *J* = 8.2, 1H), 7.74 (d, *J* = 8.1, 1H), 7.54–7.48 (m, 2H), 7.42 (dd, *J* = 8.1, 7.2, 1H), 7.37–7.34 (m, 2H), 6.32 (dd, *J* = 3.0, 1.9, 1H), 6.06 (d, *J* = 2.6, 1H), 3.15 (t, *J* = 7.4, 2H), 2.78 (t, *J* = 7.4, 2H), 2.14 (quintet, *J* = 7.6, 2H); ^{13}C NMR (150 MHz, CDCl$_3$): δ 156.1, 141.0, 138.2, 134.1, 132.0, 128.9, 126.8, 126.2, 125.9, 125.7, 125.6, 123.9, 110.3, 105.2, 32.5, 29.1, 28.0; IR (film): 3046, 2929, 2867, 1737, 1596, 1507, 1239, 1147, 1007 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{17}H_{16}OH$, 237.1279; found 237.1278.



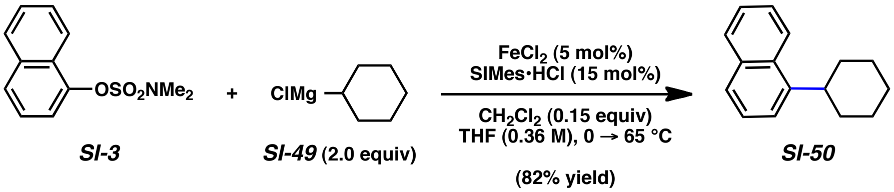
SI-47 (Table 3, entry 3b). Purification by flash chromatography (50:1:1 Hexanes:Benzene:EtOAc) supplied cross-coupled product **SI-47** (52% yield) as a yellow oil. R$_f$ 0.27 (50:1:1 Hexanes:Benzene:EtOAc). See spectral data above.



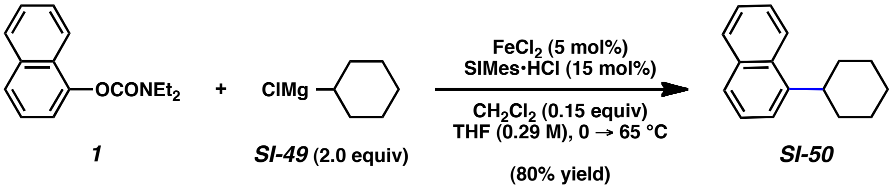
SI-48 (Table 3, entry 4a). Purification by flash chromatography (20:1 Pentanes:Benzene) yielded cross-coupled product **SI-48** (93% yield) as a clear oil. R$_f$ 0.73 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[12]



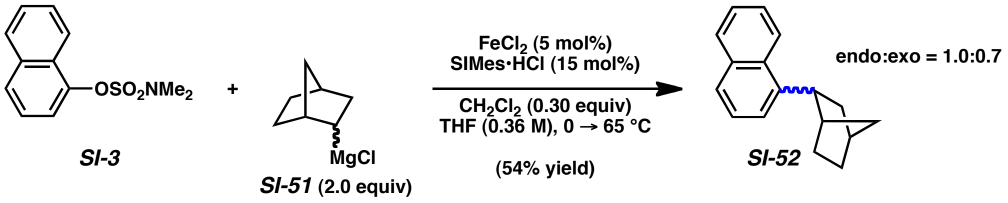
SI-48 (Table 3, entry 4b). Purification by flash chromatography (20:1 Pentanes:Benzene) generated alkylated product **SI-48** (99% yield) as a clear oil. R_f 0.73 (20:1 Pentanes:Benzene). Spectral data match those previously reported.[12]



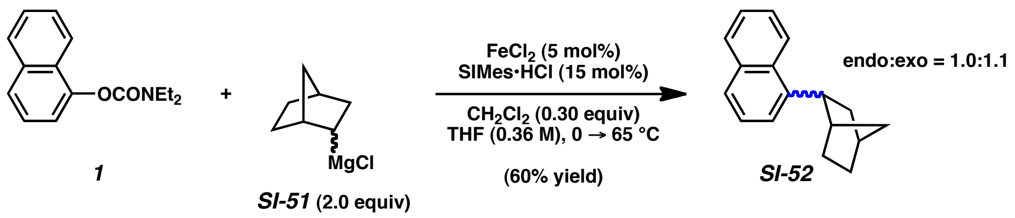
SI-50 (Table 3, entry 5a). Purification by flash chromatography (100% Pentanes) afforded cross-coupled product **SI-50** (82% yield) as a clear oil. R_f 0.56 (100% Pentanes). Spectral data match those previously reported.[13]



SI-50 (Table 3, entry 5b). Purification by flash chromatography (100% Pentanes) supplied cross-coupled product **SI-50** (80% yield) as a clear oil. R_f 0.56 (100% Pentanes). Spectral data match those previously reported.[13]

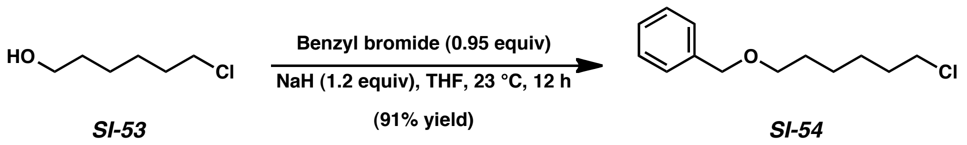


SI-52 (Table 3, entry 6a). Purification by flash chromatography (39:1 Pentanes:Benzene) afforded cross-coupled products **SI-52** (54% yield as a 1.0:0.7 mixture of endo:exo isomers) as a clear oil. R_f 0.47 (39:1 Pentanes:Benzene). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.19 (d, J = 8.6, 1H), 7.88–7.85 (m, 1.5H), 7.73–7.72 (m, 1H), 7.67(d, J = 7.5, 0.6H), 7.54–7.40 (m, 6.3H), 4.06–4.03 (m, 1H), 3.41 (dd, J = 8.6, 5.6, 0.5H), 2.61–2.59 (m, 1.5H), 2.44–2.41 (m, 1.5H), 2.07–1.99 (m, 1.6H), 1.82–1.52 (m, 7.4H), 1.49–1.39 (m, 1.7H), 1.31 (d, J = 9.5, 1.6H), 1.27–1.16 (m, 1.9H); ^{13}C NMR (125 MHz, $CDCl_3$): (33 of 34 C) δ 143.2, 138.8, 134.2, 134.1, 133.1, 132.1, 128.9, 128.9, 126.6, 126.2, 125.7, 125.5, 125.4, 125.4, 125.2, 124.4, 124.1, 123.4, 121.8, 43.4, 42.4, 42.3, 41.7, 41.4, 39.7, 37.9, 37.2, 36.7, 34.1, 30.6, 29.9, 29.5, 23.6; IR (film): 3047, 2947, 2868, 1597, 1509, 1396 cm^{-1}; HRMS-EI (*m/z*) $[M]^+$ calcd for $C_{17}H_{18}$, 222.1409; found 222.1411.

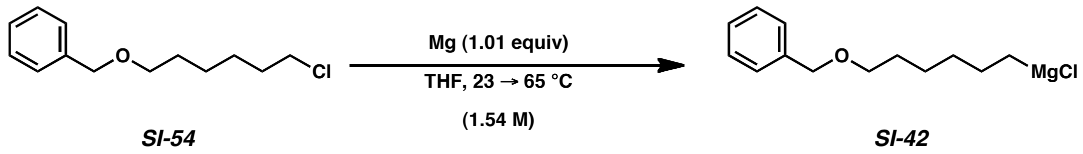


SI-52 (Table 3, entry 6b). Purification by flash chromatography (39:1 Pentanes:Benzene) supplied cross-coupled products **SI-52** (60% yield, as a 1.0:1.1 mixture of endo:exo isomers) as a clear oil. R$_f$ 0.47 (39:1 Pentanes:Benzene). See above for spectral data.

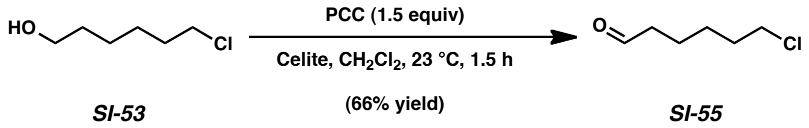
C. Synthesis of Grignard Reagents



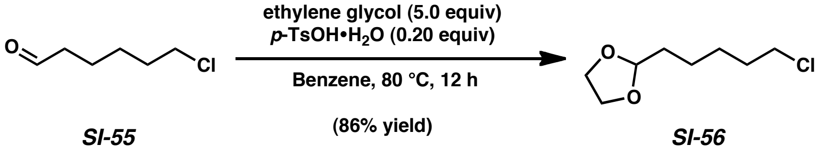
SI-54. A flame-dried round bottom flask was charged with NaH (1.43 g, 35.8 mmol, 1.2 equiv, 60% dispersion in mineral oil). The flask was cooled to 0 ºC, and then a solution of 6-chloro-1-hexanol (**SI-53**) (4.0 mL, 29.9 mmol, 1 equiv) in THF (72.4 mL) was added dropwise via cannula. After 30 minutes, benzyl bromide (4.85 g, 3.4 mL, 28.4 mmol, 0.95 equiv) was added dropwise to the reaction vessel. The reaction was allowed to warm to 23 °C and stirred for 12 h. The reaction was quenched with a solution of 1 M NaOH (10 mL), and then EtOAc (150 mL) and 1 M aqueous solution of NaOH (50 mL) were added. The layers were separated, and the aqueous layer was extracted with EtOAc (2 x 150 mL). The organic layer was washed successively with H_2O (40 mL) and brine (40 mL). The combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The crude residue was purified by flash chromatography (4:1 Hexanes:EtOAc) to yield chloro-benzyl ether **SI-54** as a yellow oil (5.85 g, 91% yield). R$_f$ 0.36 (4:1 Hexanes:EtOAc). Spectral data match those previously reported.[14]



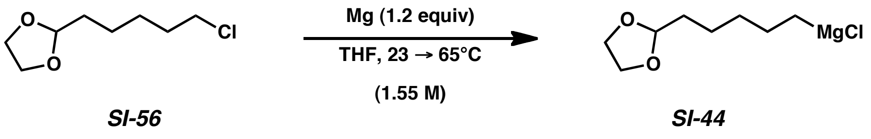
SI-42. A flame-dried round bottom flask was charged with magnesium powder (541.4 mg, 22.27 mmol, 1.01 equiv). The flask was fitted with a water-cooled condenser, and then flame-dried under vacuum to activate the magnesium powder. Dry THF (6.35 mL) was added and the suspension was heated to reflux for 1 h. To the refluxing suspension was added chloro-benzyl ether (**SI-54**) (5.0 g, 22.05 mmol, 1.0 equiv) dropwise over 30 minutes. Upon completion, dry THF (1.0 mL) was added, and the reaction was held at reflux for 1 h. The reaction yielded alkyl benzyl magnesium chloride **SI-42**; the solution was found to be 1.54 M in THF.[1]



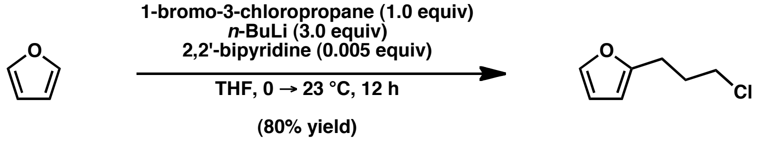
SI-55. A round bottom flask was charged with pyridinium chlorochromate (PCC) (14.47 g, 67.14 mmol, 1.5 equiv) and Celite (14.47 g). The material was suspended in CH_2Cl_2 (120 mL) and then 6-chloro-1-hexanol (**SI-53**) (6.0 mL, 44.76 mmol, 1.0 equiv) was added dropwise over several minutes. The mixture was allowed to stir for 1.5 h at 23 ºC. The reaction mixture was then filtered through a pad of basic alumina and Celite. The pad was washed with Et_2O (360 mL) and the volatiles were removed under reduced pressure. The crude residue was purified by flash chromatography (6:1 Pentanes:Et_2O) to yield 6-chloro-1-hexanal **SI-55** as a colorless oil (3.98 g, 66% yield). R_f 0.31 (6:1 Pentanes:Et_2O). Spectral data match those previously reported.[15]



SI-56. To a solution of 6-chloro-1-hexanal (**SI-55**) (1.33g, 9.90 mmol, 1.0 equiv) and ethylene glycol (2.76 mL, 49.5 mmol, 5.0 equiv) in benzene (113 mL) was added *p*-toluenesulfonic acid monohydrate (*p*–TsOH•H_2O) (376.6 mg, 1.98 mmol, 0.20 equiv). The resulting solution was heated to reflux. After 12 h, the reaction mixture was cooled to 23 ºC, the volatiles were removed under reduced pressure, and the reaction mixture was diluted with Et_2O (50 mL), and then poured into water (25 mL). The layers were separated and the organic layer was washed sequentially with a solution of saturated aqueous $NaHCO_3$ (25 mL) and brine (25 mL). The solution was dried over $MgSO_4$ and evaporated to dryness. The crude residue was purified by flash chromatography (20:1 Hexanes:EtOAc) to yield 1-chlorohexanal ethylene acetal **SI-56** as a pale yellow oil (1.51 g, 86% yield). R_f 0.28 (20:1 Hexanes:EtOAc), bp = 115 ºC at 2.5 mmHg. Spectral data match those previously reported.[16]

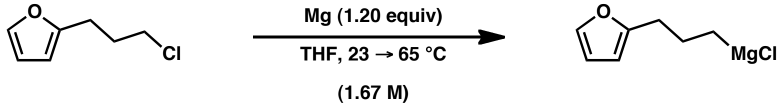


SI-44. A dry round bottom flask was charged with magnesium powder (635.2 mg, 26.13 mmol, 1.2 equiv). The flask was fitted with a water-cooled condenser and flame-dried under vacuum to activate the magnesium powder. To further activate the magnesium, I_2 (single crystal) was added and the flask was heated under N_2. Dry THF (6.0 mL) was added and the suspension heated to reflux for 1 h. To the heated suspension were added 1,2-dibromoethane (0.1 mL) and 1-chlorohexanal ethylene acetal (**SI-56**) (3.89 g, 21.77 mmol, 1.0 equiv) dropwise. Next, dry THF (1.0 mL) was added, and the reaction was held at reflux for 1 h to afford the Grignard reagent **SI-44**; the solution was found to be 1.55 M in THF.[1]



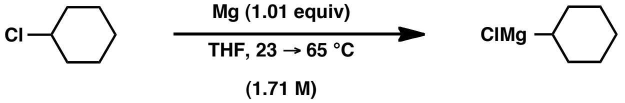
SI-57 ***SI-58***

SI-58. A dry 2 L three-neck round bottom flask was fitted with two dry 250 mL pressure equalizing addition funnels. The flask was charged with 2,2'-bipyridine (270 mg, 1.74 mmol, 0.005 equiv) and dry THF (540 mL), and the solution was cooled to 0 ºC. Furan (**SI-57**) (37.3 mL, 387.14 mmol, 3.7 equiv) was added in one portion via addition funnel, and the resulting solution was held at 0 ºC for 30 minutes. A solution of *n*-BuLi (2.07 M in hexanes, 149 mL, 307.8 mmol, 3.0 equiv) was added to the addition funnel via cannula, and then added dropwise to the reaction flask. The mixture was allowed to stir at 0 ºC in an ice bath for 2 h before 1-bromo-3-chloropropane (10.26 mL, 102.6 mmol, 1.0 equiv) was added dropwise. The reaction remained in the bath and was allowed to warm slowly 0 → 23 ºC over 12 h. The reaction was quenched with a saturated, aqueous solution of NH_4Cl (270 mL), and then hexanes (270 mL) were added. The layers were separated, and the aqueous layer was extracted with hexanes (350 mL x 3). The organic layer was washed with brine (275 mL x 2). The combined organic layers were dried over $MgSO_4$, and concentrated under reduced pressure. The crude residue was purified by fractional vacuum distillation, 77–80 ºC at 2 mmHg yielding 2-(3-chloropropyl)furan **SI-58** as a yellow oil (10.6 g, 80% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.31 (dd, *J* = 1.7, 0.6, 1H), 6.29 (dd, *J* = 3.0, 1.9, 1H), 6.04 (dd, *J* = 3.1, 0.7, 1H), 3.56 (t, *J* = 6.5, 2H), 2.81 (t, *J* = 7.2, 2H), 2.11 (quintet, *J* = 6.9, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 154.5, 141.4, 110.3, 105.8, 44.3, 31.0, 25.3; IR (film): 2959, 1597, 1507, 1443, 1294, 1148, 1007 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for C_7H_9ClONa, 167.0153; found 167.0156.



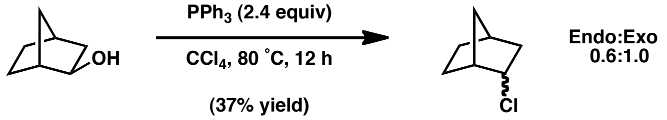
SI-58 ***SI-46***

SI-46. A dry round bottom flask was charged with magnesium powder (1.01g, 41.50 mmol, 1.2 equiv). The flask was fitted with a water-cooled condenser and flame-dried under vacuum to activate the magnesium powder. Dry THF (10.5 mL) was added and the resulting suspension was heated to reflux for 1 h. To the refluxing suspension was added 2-(3-chloropropyl)furan (**SI-58**) (5.0 g, 34.58 mmol, 1.0 equiv) dropwise over 45 minutes. Next dry THF (1.0 mL) was added, and the reaction was held at reflux for 1 h. The reaction yielded (3-(furan-2-yl)propyl)magnesium chloride **SI-46**; the solution was found to be 1.67 M in THF.[1]



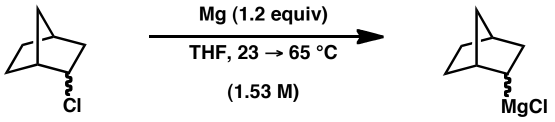
SI-59 ***SI-49***

SI-49. A dry round bottom flask was charged with magnesium powder (1.03 g, 42.58 mmol, 1.01 equiv). The flask was fitted with a water-cooled condenser and flame-dried under vacuum to activate the magnesium powder. Dry THF (13.1 mL) was added and the suspension was heated to reflux for 1 h. To the heated suspension was added chlorocyclohexane (**SI-59**) (5.0 g, 42.16 mmol, 1.0 equiv) dropwise over 20 minutes. Next, dry THF (1.0 mL) was added, and the reaction was held at reflux for 1 h. The reaction yielded cyclohexylmagnesium chloride **SI-49**; the solution was found to be 1.71 M in THF.[17,1]



SI-60 ***SI-61***

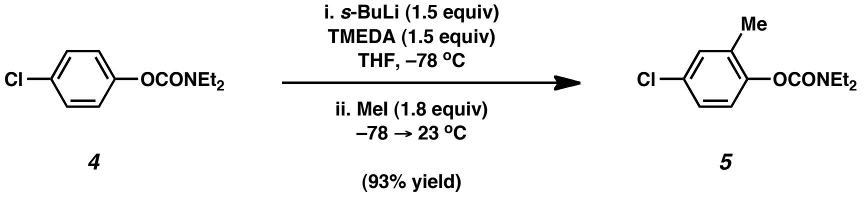
SI-61. In a flame-dried round bottom flask, *exo*-norborneol (**SI-60**) (5.0 g, 44.57 mmol, 1 equiv) and triphenylphosphine (28.1 g, 106.97 mmol, 2.4 equiv) were dissolved in dry CCl_4 (127.3 mL). The reaction was allowed to stir for 12 h at 80 ºC, and was then cooled to 23 °C. The reaction was diluted with pentanes (100 mL), filtered through a pad of silica with additional pentanes (400 mL), and then concentrated under reduced pressure. The crude residue was purified by fractional distillation at 25 mbar, 50 ºC to yield 2-chloronorborane **SI-61** as a white solid (2.2 g, 37% yield). ^{1}H NMR (600 MHz, $CDCl_3$): δ 4.26–4.22 (m, 0.6H), 3.89 (dt, *J* = 7.3, 2.0, 1H), 2.42–4.21 (m, 1.6H), 2.34–2.31 (m, 1H), 2.23 (t, *J* = 4.3, 0.6H), 2.20–2.15 (m, 0.6H), 2.04–2.00 (m, 0.6H), 1.94 (dd, *J* = 7.4, 2.3, 0.6H) 1.91, (dd, *J* = 7.4, 2.2, 0.6H), 1.83–1.76 (m, 2H), 1.65–1.56 (m, 1.6H), 1.49–1.37 (m, 2.9H), 1.35–1.31 (m, 0.7H), 1.26–1.22 (m, 2H), 1.19 (d, *J* = 1.3, 1H), 1.16–1.12 (m, 1H), 1.10–1.06 (m, 1H), 1.00 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 62.7, 46.2, 43.7, 36.7, 35.2, 28.3, 26.9. Spectral data match those previously reported.[18]



SI-61 ***SI-51***

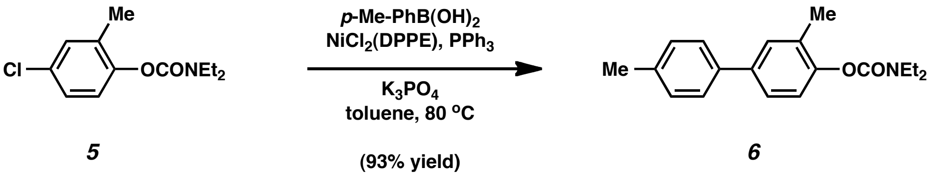
SI-51. A dry round bottom flask was charged with magnesium powder (1.02 g, 41.80 mmol, 1.2 equiv). The flask was fitted with a water-cooled condenser and flame-dried under vacuum to activate the magnesium powder. To further activate the magnesium, I_2 (single crystal) was added and the flask gently heated with a heat gun under N_2. Dry THF (6.1 mL) was added, the suspension was heated to reflux for 1 h. To the refluxing suspension were added 1,2-dibromoethane (0.5 mL) and 2-chloronorborane (**SI-61**) (4.55 g, 34.83 mmol, 1.0 equiv) dropwise. Next, dry THF (1.0 mL) was added, and the reaction was held at reflux for 1 h. The reaction yielded 2-norborylmagnesium chloride **SI-51**; the solution was found to be 1.53 M in THF.[19,1]

D. Synthetic Application (Figure 3)

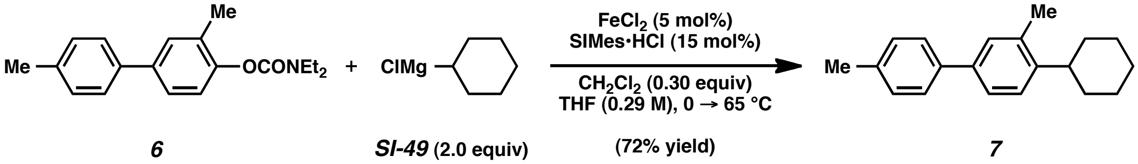


4 ***5***

5. To a solution of known carbamate **4**[20] (0.569 g, 2.50 mmol, 1.0 equiv) and TMEDA (0.56 mL, 3.75 mmol, 1.5 equiv) in THF (27.8 mL) at –78 °C was added *s*-BuLi (3.5 mL, 1.07 mmol, 1.5 equiv) over 15 min. After stirring for 60 min, MeI (0.28 mL, 4.50 mmol, 1.8 equiv) was added over 5 min. The reaction was allowed to stir at –78 °C for 1 h, then warmed to 23 °C over 1 h, and then quenched with saturated aqueous NH_4Cl (20 mL). The layers were separated, and the aqueous layer was extracted with Et_2O (3 x 30 mL). The combined organic layers were washed with brine (30 mL), dried over $MgSO_4$ and concentrated under reduced pressure, yielding methylated product **5** (0.5606 g, 93% yield) as a light yellow oil. The crude residue was used without further purification. R_f 0.48 (3:1 Hexanes:Et_2O). Spectral data match those previously reported.[21]



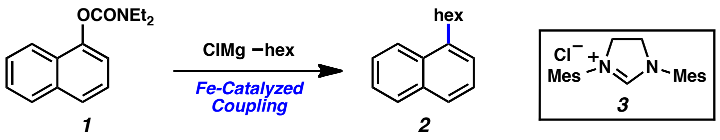
6. A dry 20 mL vial was charged with K_3PO_4 (1.15 g, 5.4 mmol, 4.5 equiv), Ni(DPPE)Cl_2 (31.7 mg, 0.06 mmol, 5 mol%), triphenylphosphine (31.5 mg, 0.12 mmol, 10 mol%), *p*-Me-PhB$(OH)_2$ (489.5 mg, 3.6 mmol, 3.0 equiv), and aryl chloride **5** (290.1 mg, 1.2 mmol, 1.0 equiv). The headspace was evacuated and backfilled with N_2 (3 times). Toluene (6.0 mL) was added, and the resulting mixture was heated to 80 ºC for 14 h. The reaction was then cooled to 23 °C and purified by flash chromatography (3:1 Hexanes:Et_2O) to produce carbamate **6** (331.5 mg, 93% yield) as a colorless solid. R_f 0.67 (3:1 Hexanes:Et_2O). mp: 53.0–56.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45 (d, *J* = 8.1, 2H), 7.41 (d, *J* = 1.8, 1H), 7.38 (dd, *J* = 8.3, 2.1, 1H), 7.23 (d, *J* = 7.8 2H), 7.12 (d, *J* = 8.2 1H), 3.49 (q, *J* = 7.0, 2H), 3.42 (q, *J* = 6.9, 2H), 2.39 (s, 3H), 2.28 (s, 3H), 1.31–1.26 (m, 3H), 1.26–1.21 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.1, 149.5, 138.5, 138.1, 136.9, 130.7, 129.7, 129.5, 127.1, 125.5, 122.6, 42.4, 42.0, 21.2, 16.6, 14.4, 13.6; IR (film): 2977, 2932, 1712, 1420, 1273, 1273, 1210, 1210, 1158 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{19}H_{23}NO_2H$, 298.1807; found 298.1806.



7. Compound **7** (119.0 mg, 0.4 mmol) was prepared using the representative procedure described on page S2. Purification by flash chromatography (100% Pentanes) afforded cross-coupled product **7** (76.5 mg, 72% yield) as a colorless oil. R_f 0.42 (100% Pentanes). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.49 (dt, *J* = 8.1, 1.6, 2H), 7.39 (dd, *J* = 8.0, 1.9, 1H), 7.37 (d, *J* = 1.7, 1H), 7.28 (d, *J* = 8.0, 1H), 7.23 (d, *J* = 7.9, 2H), 2.78–2.71 (m, 1H), 2.40 (s, 3H), 2.39 (s, 3H), 1.93–1.76 (m, 5H), 1.48–1.42 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.9, 138.5, 138.4, 136.7, 135.6, 129.5, 128.9, 127.0, 126.0, 124.8, 40.1, 33.9, 27.3, 26.5, 21.2, 19.7; IR (film): 3020, 2922, 2850, 1495, 1448, 809 cm^{-1}. HRMS-ESI (*m/z*) $[M + NH_4]^+$ calcd for $C_{20}H_{24}NH_4$, 282.2222; found 282.2220.

E. Key Optimization Studies

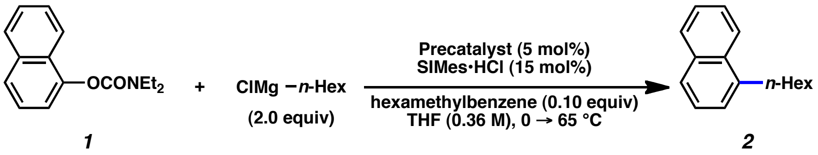
Table SI-1. Selected results from optimization studies



entry	iron salt	solvent	ligands/additives	temp (time)	yield[a]	conditions
1	$Fe(acac)_3$ (10 mol%)	THF	TMEDA (2 equiv)	–20 °C (1.5 h)	<5%	Skrydstrup's conditions
2	$Fe(acac)_3$ (10 mol%)	THF	NMP (5.8 equiv)	23 °C (1.5 h)	58%	Fürstner's conditions
3	$FeCl_2$ (0.5 mol%)	THF	*3* (4 mol%)	25 °C (2 h)	29%	Shi's conditions
4	$FeCl_2$ (5 mol%)	THF	*3* (15 mol%)	0 → 65 °C (3 h)	50–85%	Shi's conditions, with heat
5	$FeCl_2$ (5 mol%)	THF	*3* (15 mol%) CH_2Cl_2 (15 mol%)	0 → 65 °C (10 min)	93%	Optimal conditions

[a] The yield was determined by ^{1}H NMR analysis with hexamethylbenzene as internal standard.

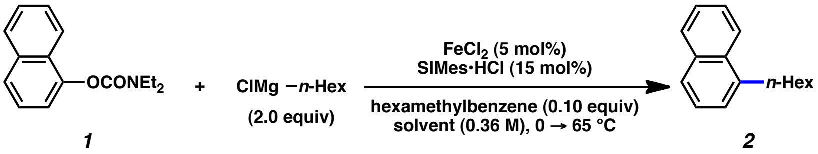
Table SI-2. Iron Precatalysts



entry	Precatalyst	yield (mol %)[a]
1	$Fe(acac)_3$	*84*
2	$FeCl_3$	*75*
3	$FeCl_2$	*86*

[a] The yield was determined by ^{1}H NMR analysis with hexamethylbenzene as internal standard.

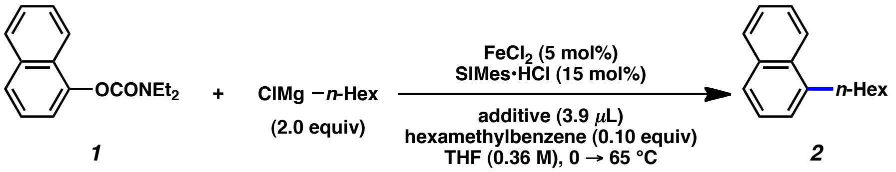
Table SI-3. Influence of Solvents



entry	solvent	yield (mol %)[a]
1[b]	Et_2O	*85*
2	dimethoxyethane	*84*
3	dioxane	*71*
4	toluene	*31*
5[b]	CH_2Cl_2	*0*

[a] The yield was determined by ^{1}H NMR analysis with hexamethylbenzene as internal standard. [b] Reaction heated to 35 ºC.

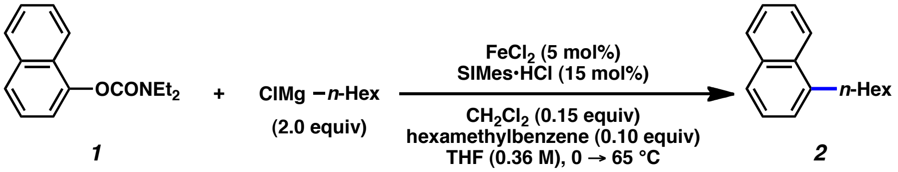
Table SI-4. Influence of chlorinated additives



entry	additive	yield (mol %)[a]
1	No additive	*50–85*
2	CH_2Cl_2	*95*
3	CH_2Br_2	*85*
4	$CHCl_3$	*82*
5	1,2-dichloroethane	*82*
6[b]	1,1-dichloroethane	*90*
7	CCl_4	*94*

[a] The yield was determined by ^{1}H NMR analysis with hexamethylbenzene as internal standard. [b] 1,1-dichloroethane (4.9 μL, 0.15 equiv).

Table SI-5. Influence of Solvent Choice with CH_2Cl_2 additive present



entry	solvent	yield (mol %)[a]
1	Et_2O	*86*
2	dimethoxyethane	*77*
3	dioxane	*71*
4	toluene	*62*

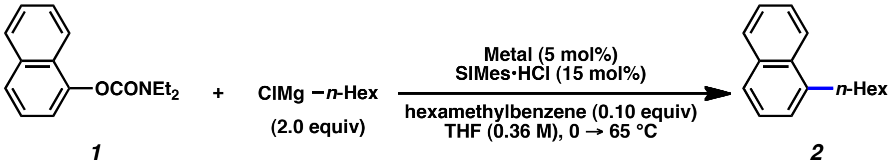
[a] The yield was determined by ^{1}H NMR analysis with hexamethylbenzene as internal standard.

F. Trace Metal Impurities[a]

entry	Vender (Fe %)	Cu (ppm)	Ni (ppm)	Pd (ppm)	Combined% (w/w)
1 $FeCl_2$ (Lot # A4048080)	Strem (98%)	111	131	298	0.05
2 $FeCl_2$ (Lot # A5268039)	Strem (98%)	324	82	56	0.05
3 $FeCl_2$	Aldrich (99.998%)	44	10	18	0.01
4 THF		0	0	0	0
5 Dichloromethane		2	0	1	0

[a] All measurements were taken using ICP-OES.

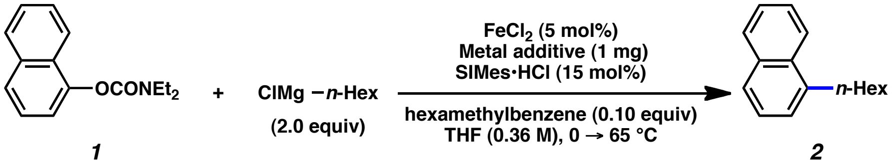
Table SI-6. Metal Catalyst.



entry	Metal	yield (mol %)[a]
1	$FeCl_2$	95
2	$NiCl_2$	4
3	$Ni(acac)_2$	3
4	$Ni(cod)_2$	3
5	$PdCl_2$	3
6	$Pd_2(dba)_3$	6
7	CuCl	0
8	$CuCl_2$	10
9	$NiCl_2$ (2.5%) + $PdCl_2$ (2.5%)	3
10	$Ni(cod)_2$ (2.5%) + $Pd_2(dba)_3$ (2.5%)	11

[a] The yield was determined by ^{1}H NMR analysis with hexamethylbenzene as internal standard.

Table SI-7. Iron with Various Metal Additives.

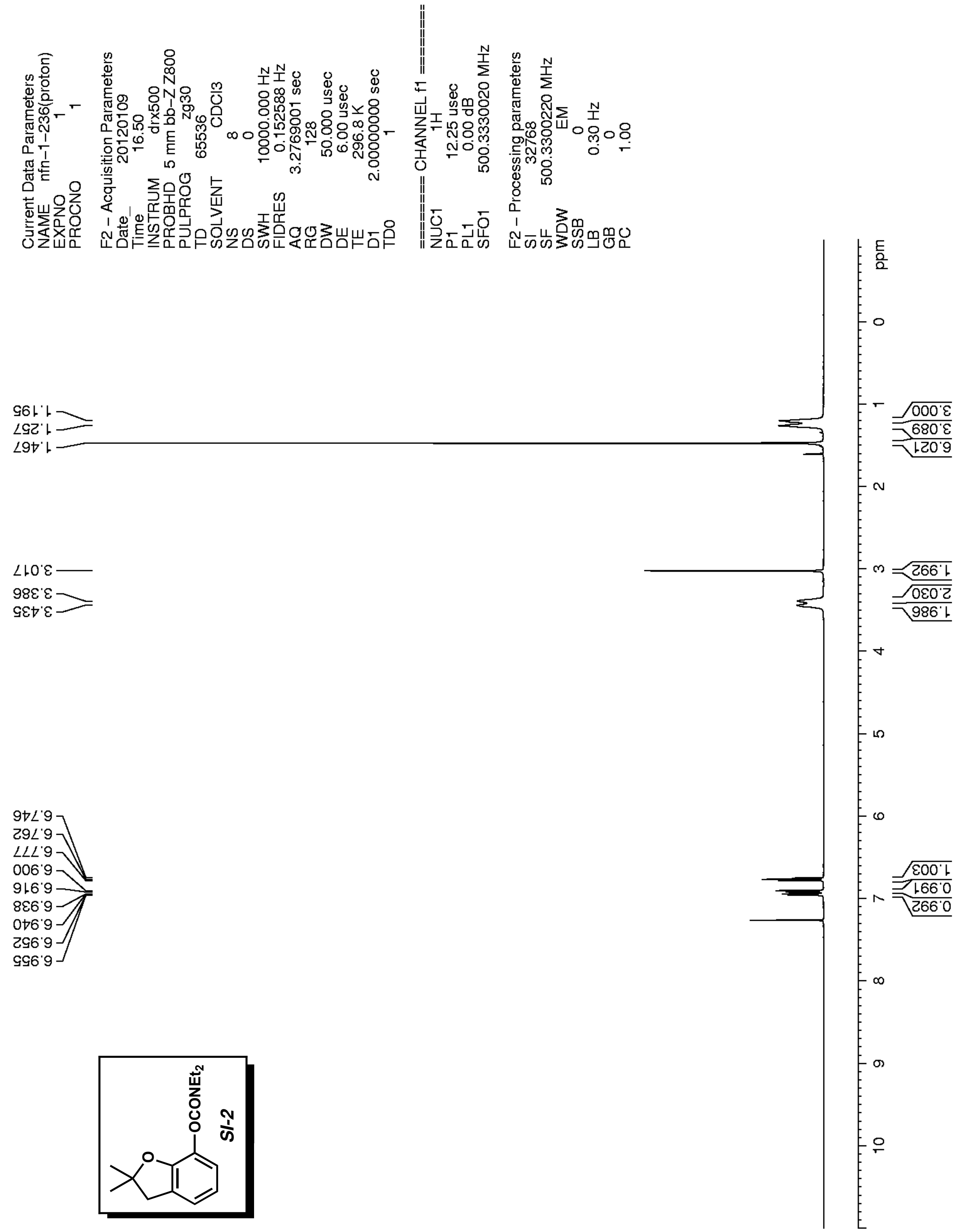


entry	Metal additive	yield (mol %)[a]
1	None	95
2	$NiCl_2$	81
3	$Ni(acac)_2$	78
4	$Ni(cod)_2$	75
5	$PdCl_2$	85
6	$Pd_2(dba)_3$	56
7	CuCl	87
8	$CuCl_2$	92

[a] The yield was determined by ^{1}H NMR analysis with hexamethylbenzene as internal standard.

^{1}H and ^{13}C

NMR Spectra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PROCNO 1
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
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SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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1.257
1.467
3.017
3.386
3.435
6.746
6.762
6.777
6.900
6.916
6.938
6.940
6.952
6.955
3.000
3.089
6.021
1.992
2.030
1.986
1.003
0.991
0.992
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0
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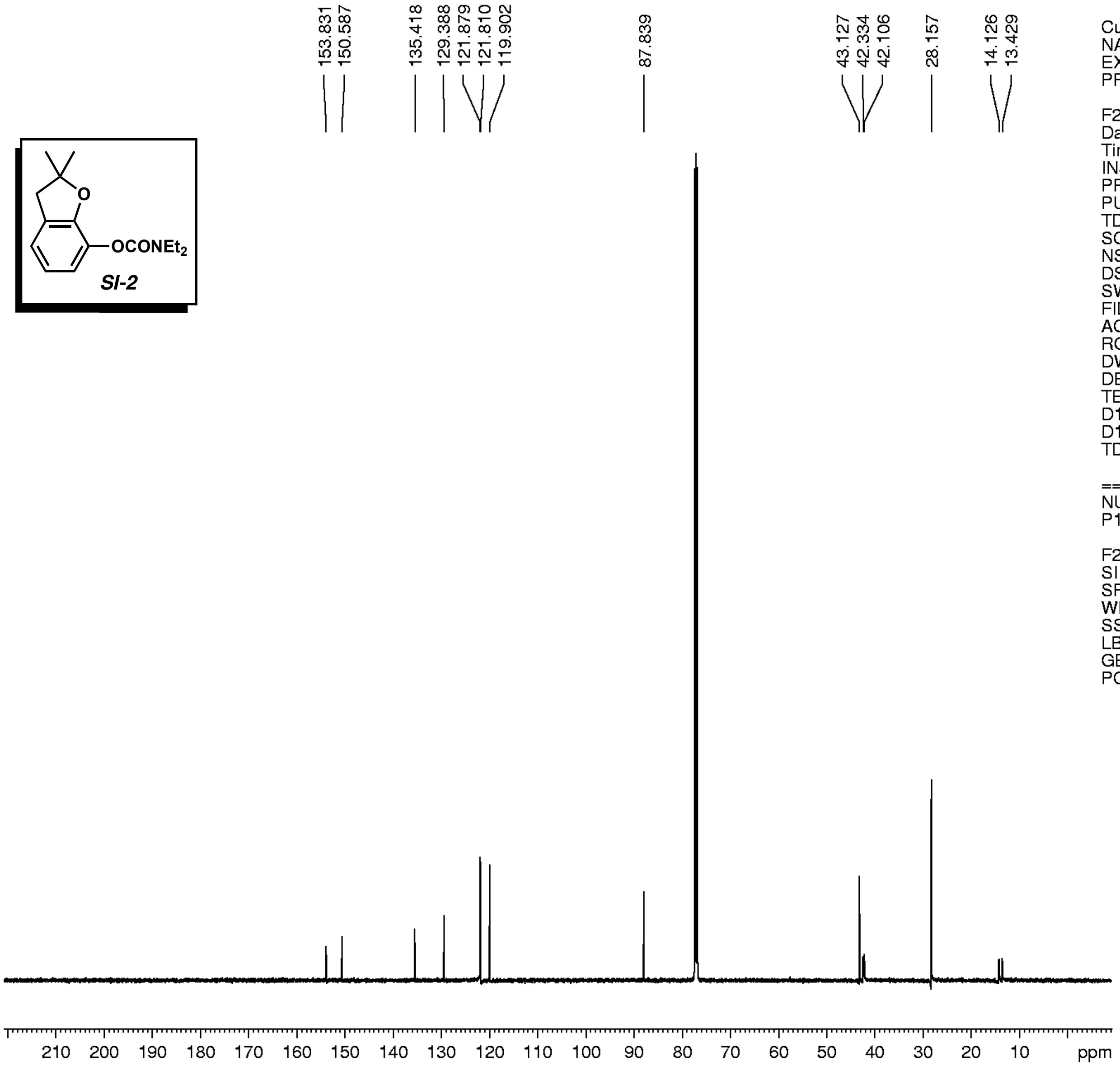
2
3
4
5
6
7
8
9
10
OCONEt2
SI-2

SI-2



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EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20111212
Time            14.14
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PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD              65536
SOLVENT         CDCl3
NS                 32
DS                  2
SWH         31250.000 Hz
FIDRES       0.476837 Hz
AQ          1.0486259 sec
RG                 11
DW             16.000 usec
DE              18.00 usec
TE              296.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1              13C
P1               9.63 usec

F2 - Processing parameters
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SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
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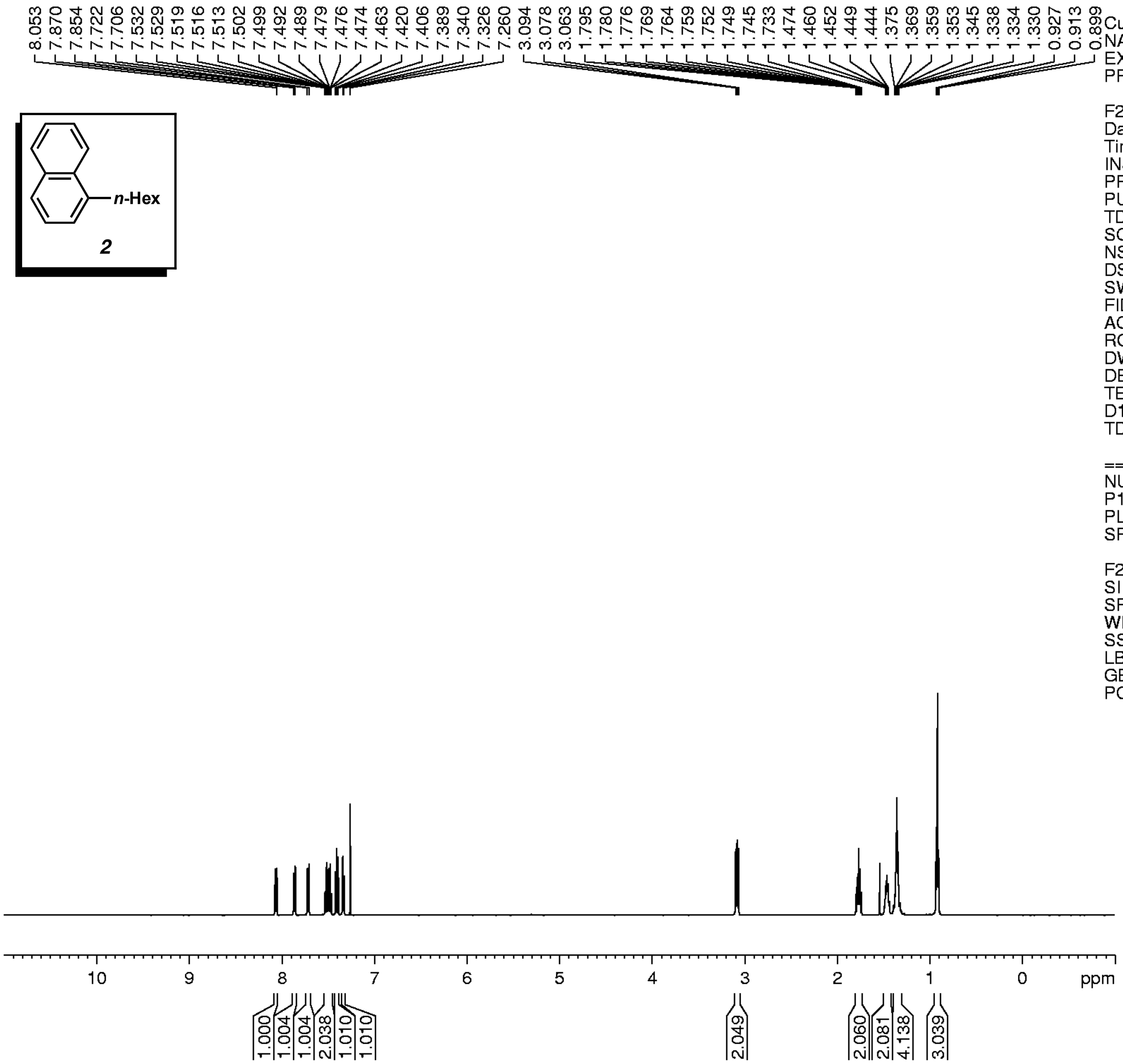

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EXPNO 1
PROCNO 1

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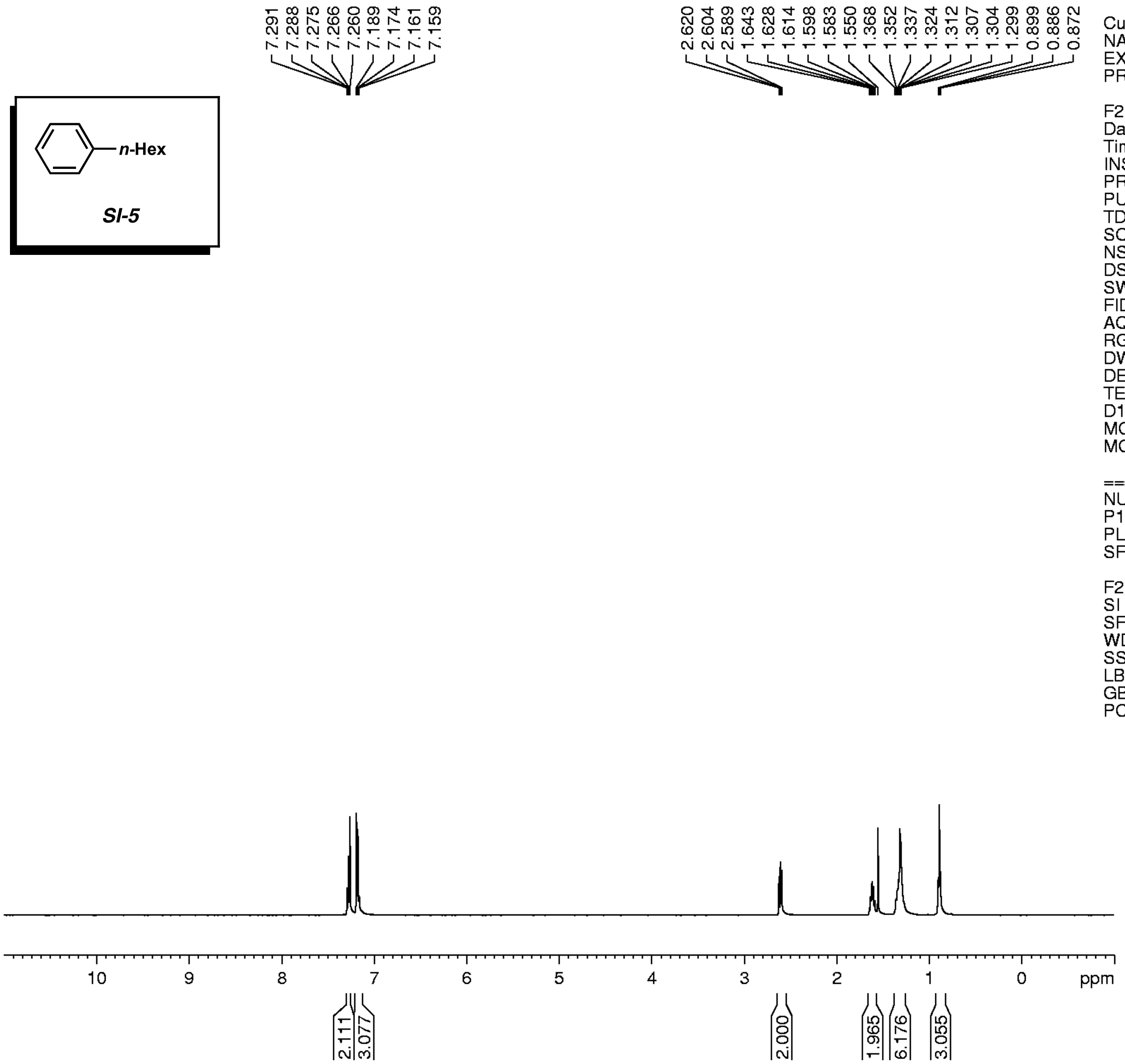
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 114
DW 50.000 usec
DE 6.00 usec
TE 297.4 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300219 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


SI-5

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PROCNO             1

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PULPROG          zg30
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SOLVENT         CDCl3
NS                 16
DS                  0
SWH       10000.000 Hz
FIDRES     0.152588 Hz
AQ        3.2769001 sec
RG                181
DW           50.000 usec
DE             6.00 usec
TE            296.2 K
D1       2.00000000 sec
MCREST   0.00000000 sec
MCWRK    0.01500000 sec

======== CHANNEL f1 ========
NUC1               1H
P1            12.25 usec
PL1            0.00 dB
SFO1    500.3330020 MHz

F2 – Processing parameters
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WDW                EM
SSB                 0
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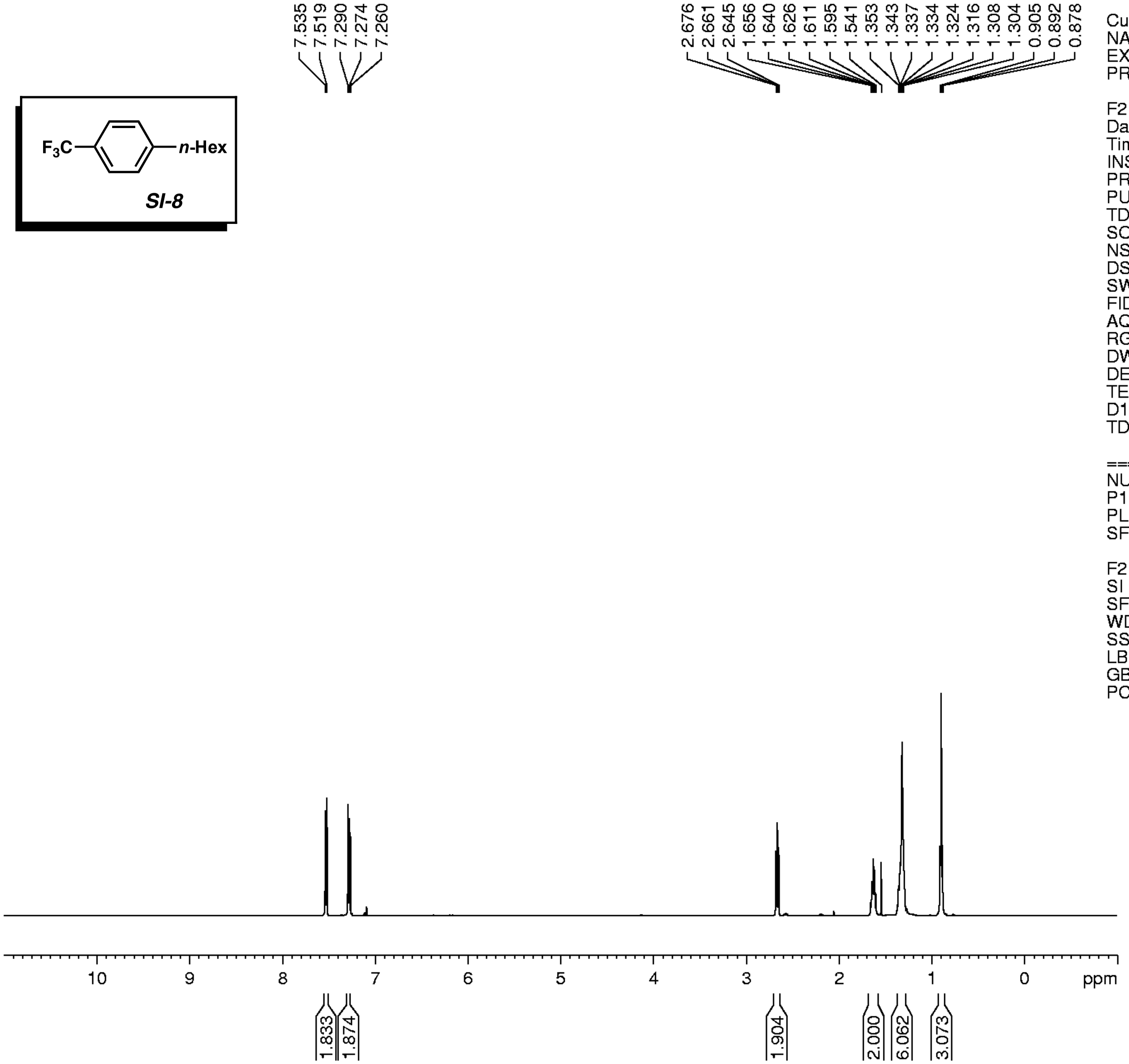
SI-8



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EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
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Time 20.41
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PROBHD 5 mm bb–Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 101.6
DW 50.000 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
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F2 – Processing parameters
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SF 500.3300219 MHz
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SSB 0
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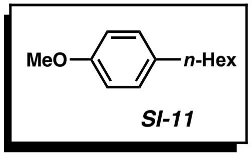
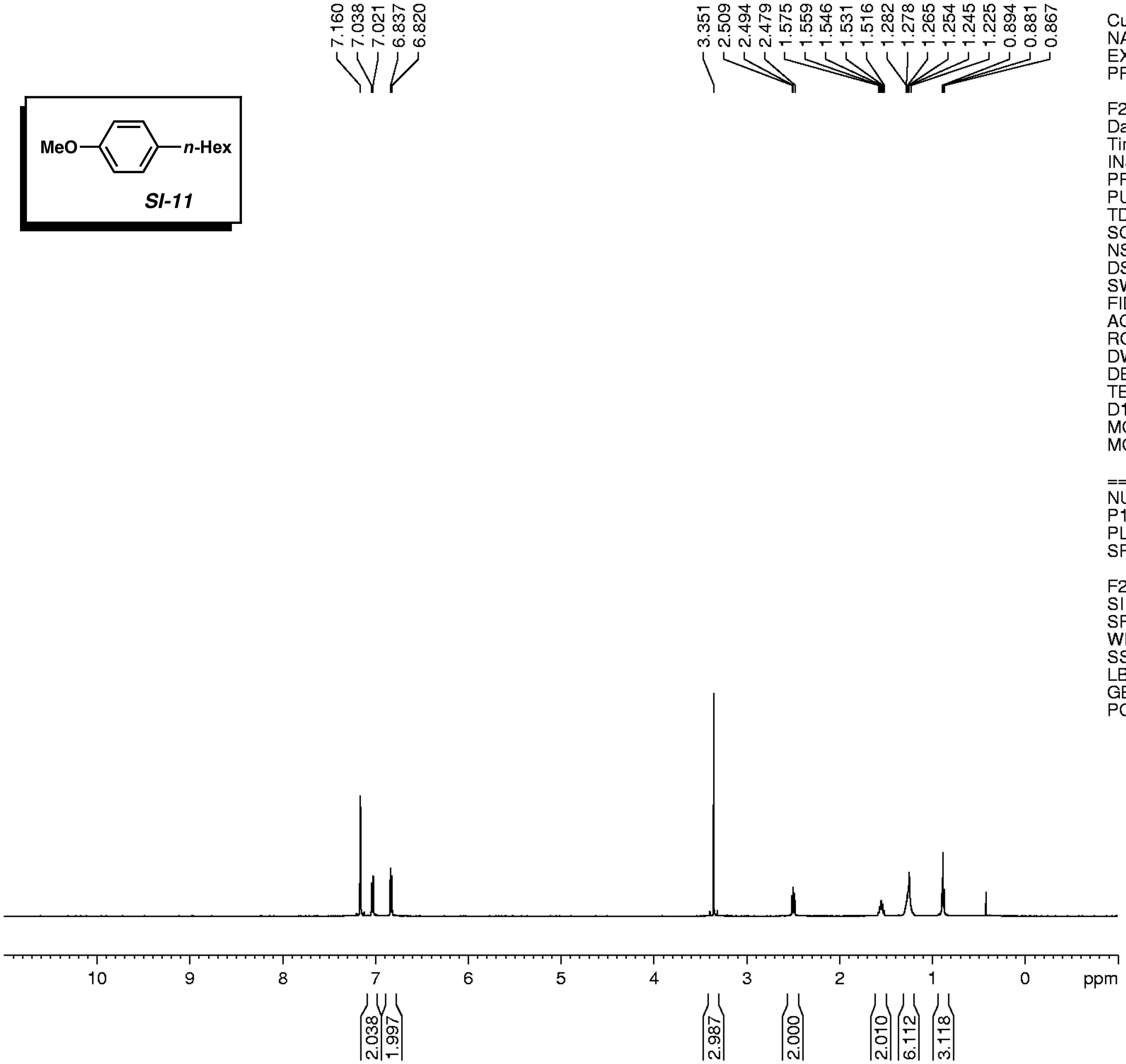
GB 0
PC 1.00



SI-11



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EXPNO              1
PROCNO              1

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PULPROG            zg30
TD              65536
SOLVENT            C6D6
NS                  16
DS                   0
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FIDRES       0.152588 Hz
AQ          3.2769001 sec
RG                 128
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DE               6.00 usec
TE              295.0 K
D1         2.00000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1              12.25 usec
PL1              0.00 dB
SFO1       500.3330020 MHz

F2 – Processing parameters
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WDW                 EM
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LB                0.30 Hz
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PC                1.00
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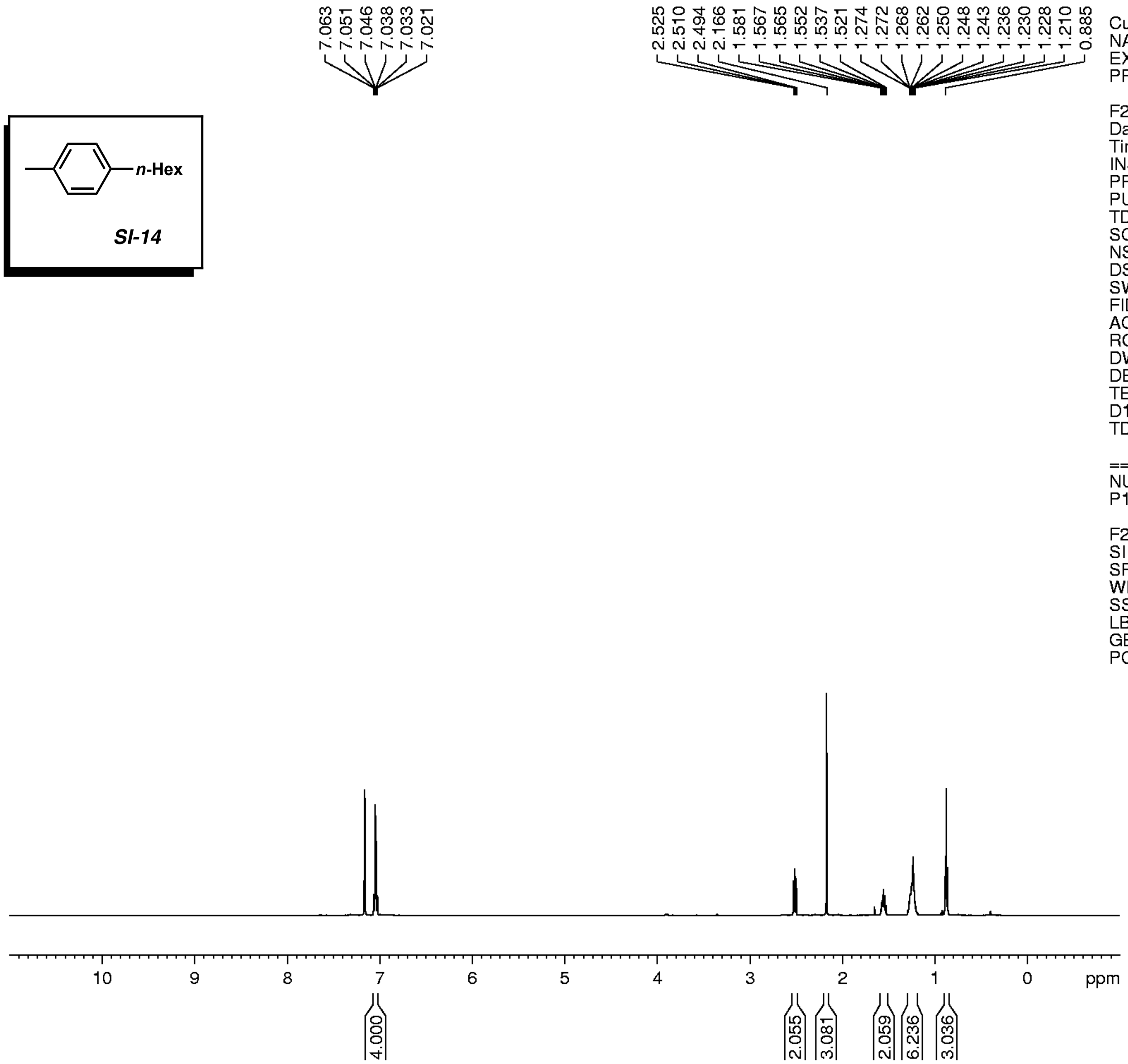

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EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
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Time            16.42
INSTRUM         av500
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PULPROG          zg30
TD              65536
SOLVENT          C6D6
NS                  3
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ        3.2768500 sec
RG                 11
DW           50.000 usec
DE            10.00 usec
TE              296.0 K
D1       2.00000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1               1H
P1            10.00 usec

F2 - Processing parameters
SI              65536
SF       500.1299974 MHz
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LB            0.30 Hz
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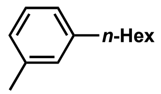
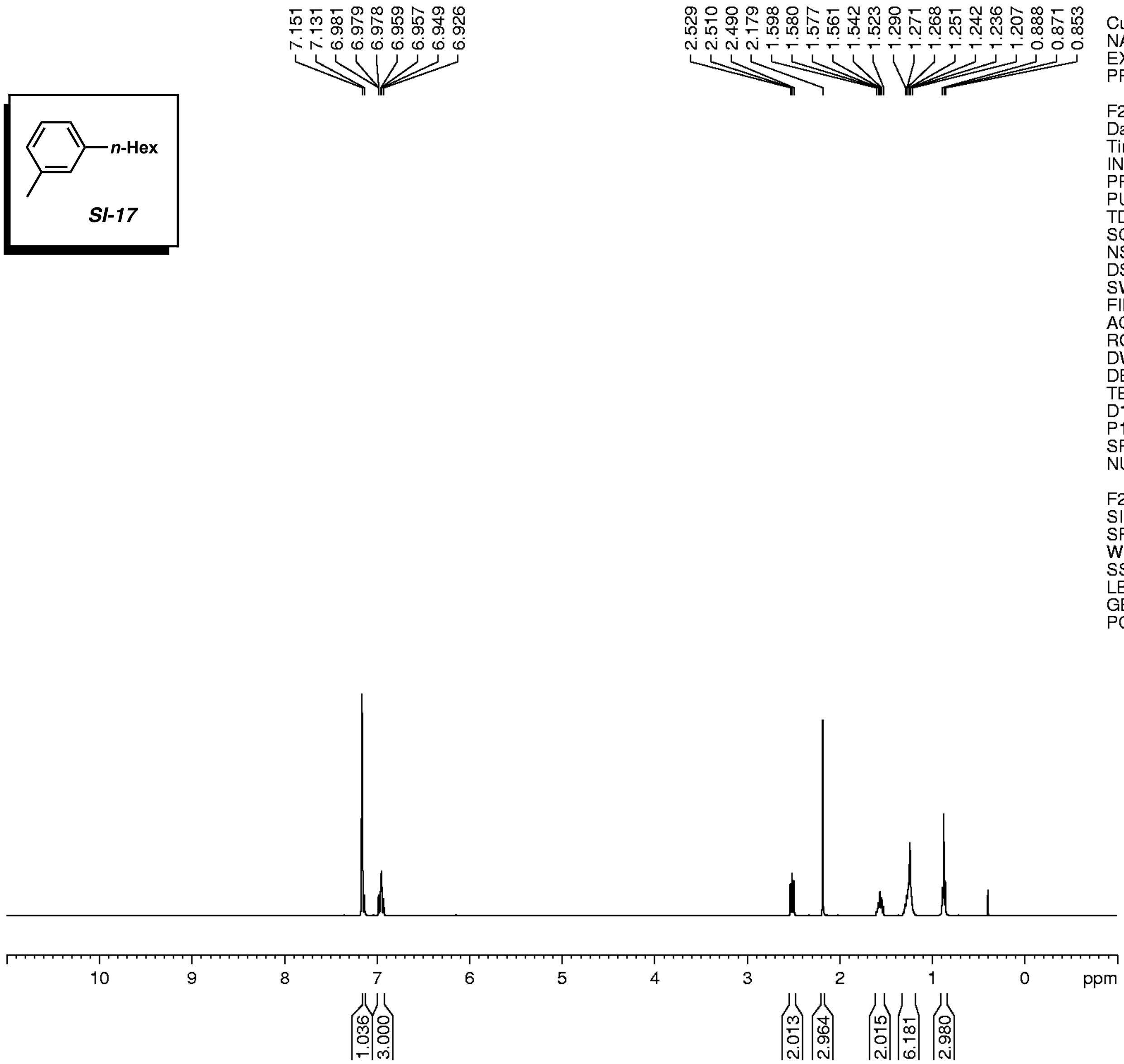


SI-17



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PROCNO 1

F2 – Acquisition Parameters
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Time 18.20
INSTRUM arx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 0
SWH 8064.516 Hz
FIDRES 0.123055 Hz
AQ 4.0632820 sec
RG 1430
DW 62.000 usec
DE 88.57 usec
TE 300.0 K
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P1 7.50 usec
SFO1 400.1324008 MHz
NUCLEUS 1H

F2 – Processing parameters
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SF 400.1300445 MHz
WDW EM
SSB 0
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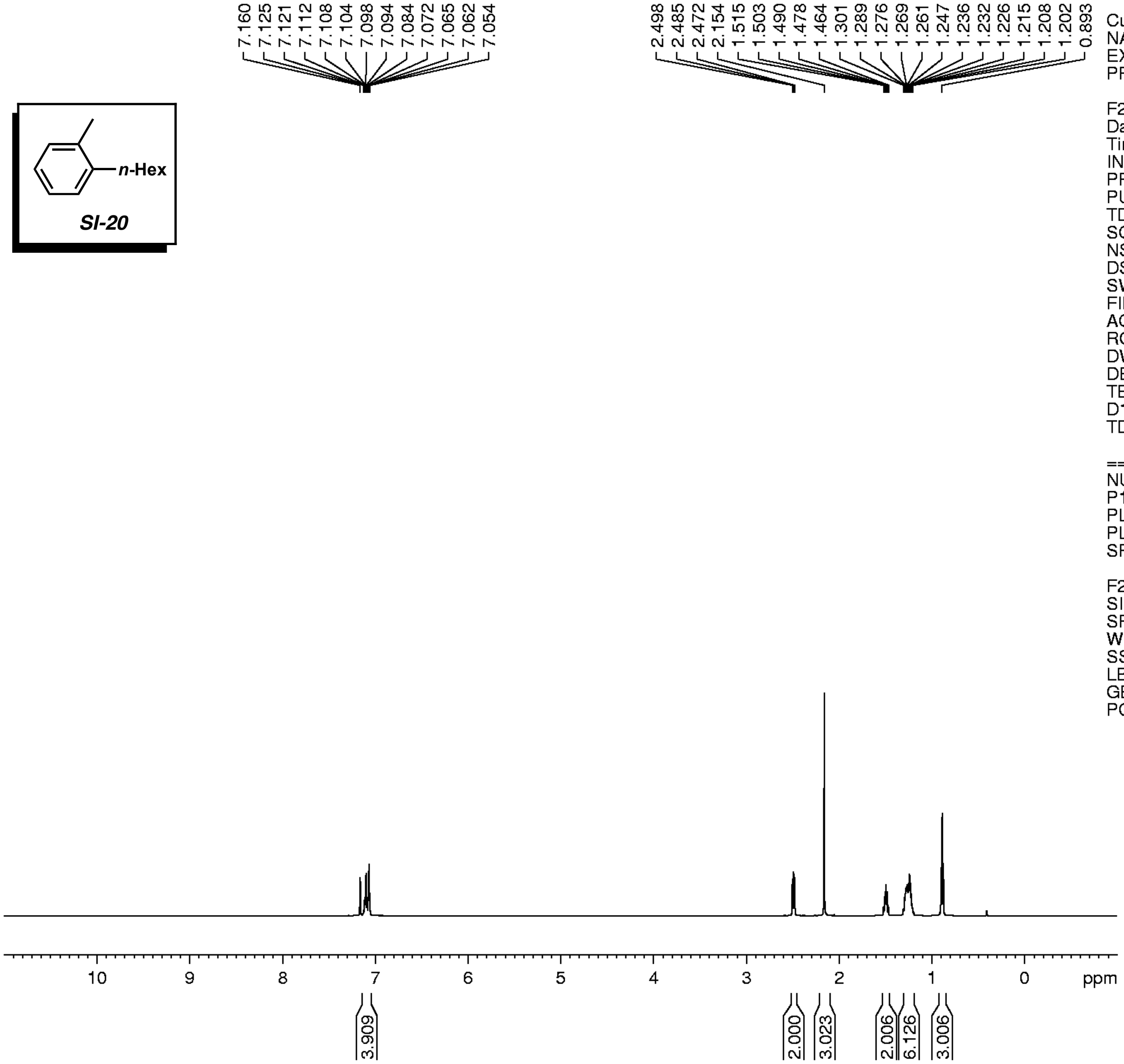
GB 0
PC 1.00

SI-20



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EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
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Time              9.17
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PROBHD   5 mm BB5
PULPROG           zg30
TD               65536
SOLVENT           C6D6
NS                  16
DS                   0
SWH          12376.237 Hz
FIDRES        0.188846 Hz
AQ           2.6477449 sec
RG                  32
DW              40.400 usec
DE                6.50 usec
TE               294.3 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               14.00 usec
PL1              -1.00 dB
PL1W       31.62277603 W
SFO1     600.1336008 MHz

F2 - Processing parameters
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WDW                 EM
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LB                0.30 Hz
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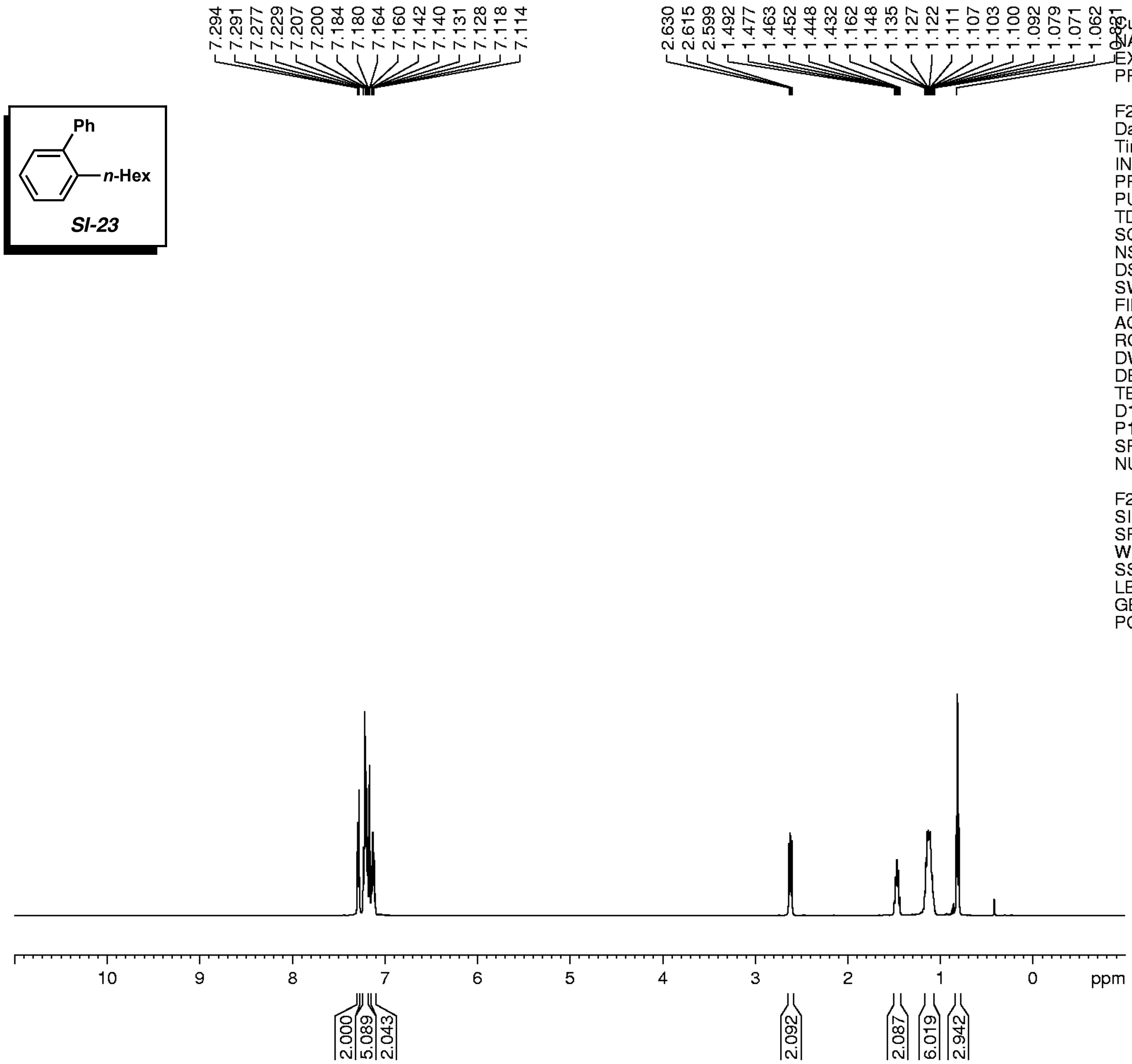

SI-23

Current Data Parameters	
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PROCNO	1
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PROBHD	5 mm broadban
PULPROG	zg30
TD	65536
SOLVENT	C6D6
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
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DW	50.000 usec
DE	71.43 usec
TE	300.0 K
D1	2.00000000 sec
P1	12.20 usec
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NUCLEUS	1H
F2 – Processing parameters	
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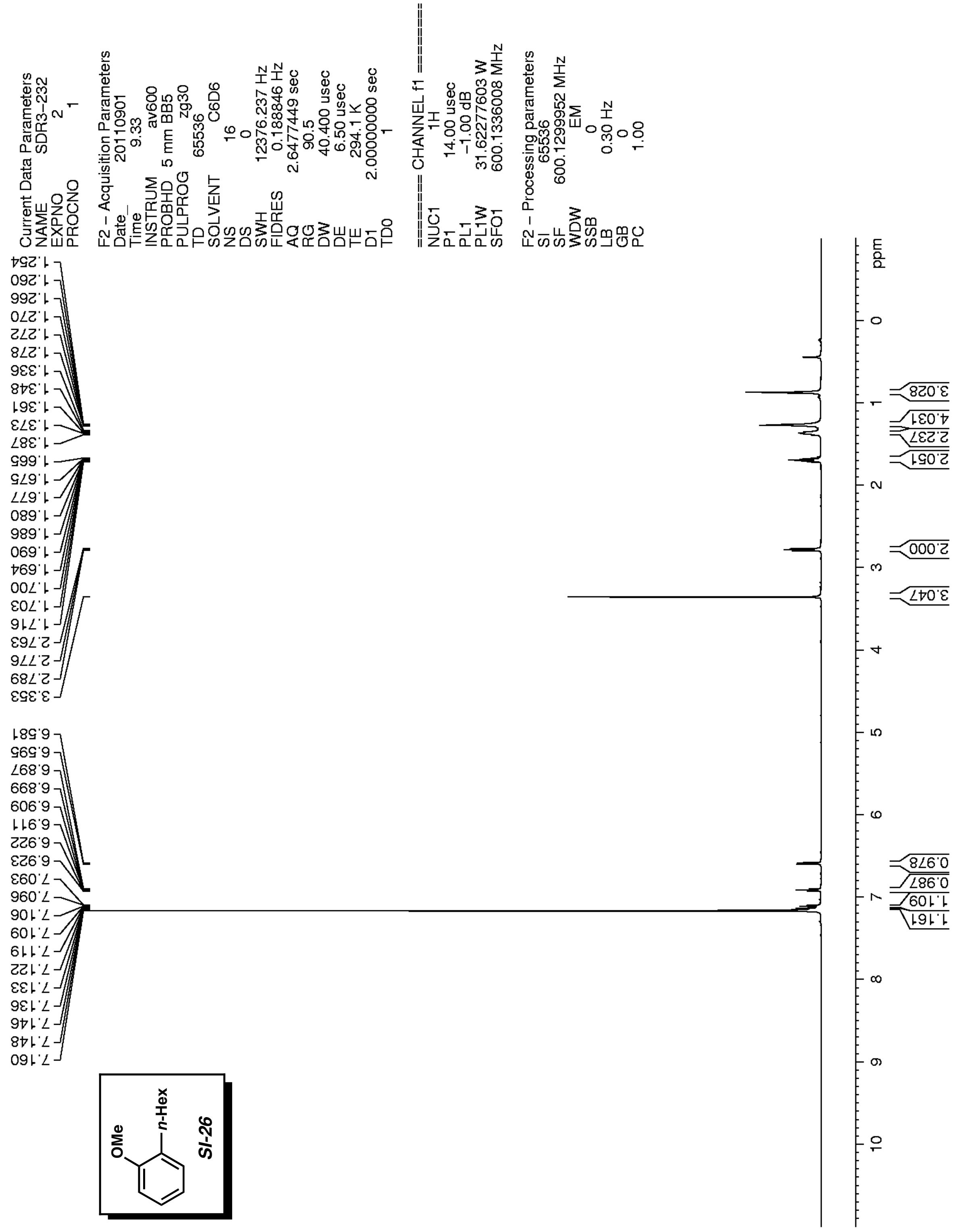
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



Current Data Parameters
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EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
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Time_ 9.33
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PROBHD 5 mm BB5
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6477449 sec
RG 90.5
DW 40.400 usec
DE 6.50 usec
TE 294.1 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 31.62277603 W
SFO1 600.1336008 MHz

F2 – Processing parameters
SI 65536
SF 600.1299952 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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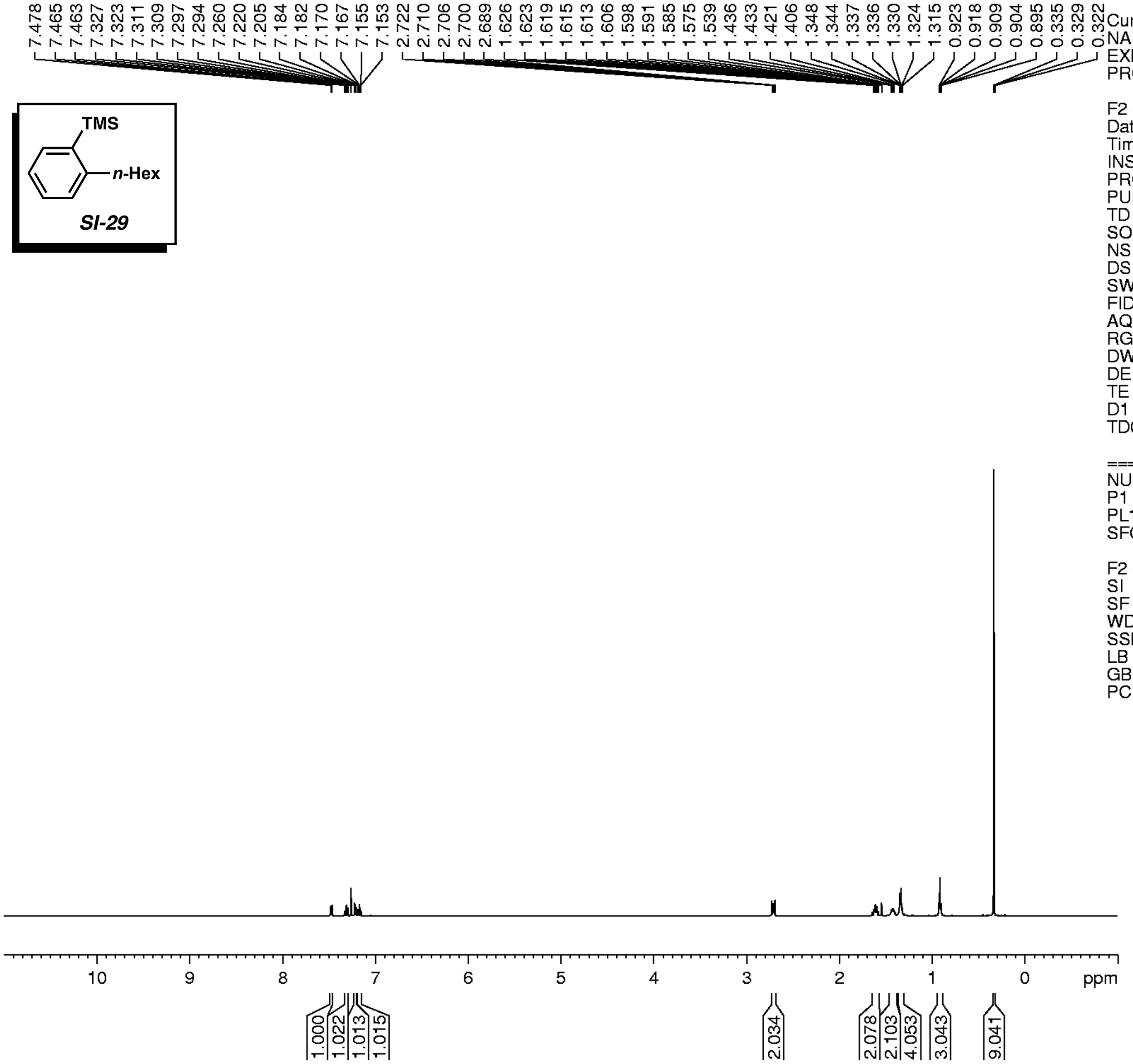
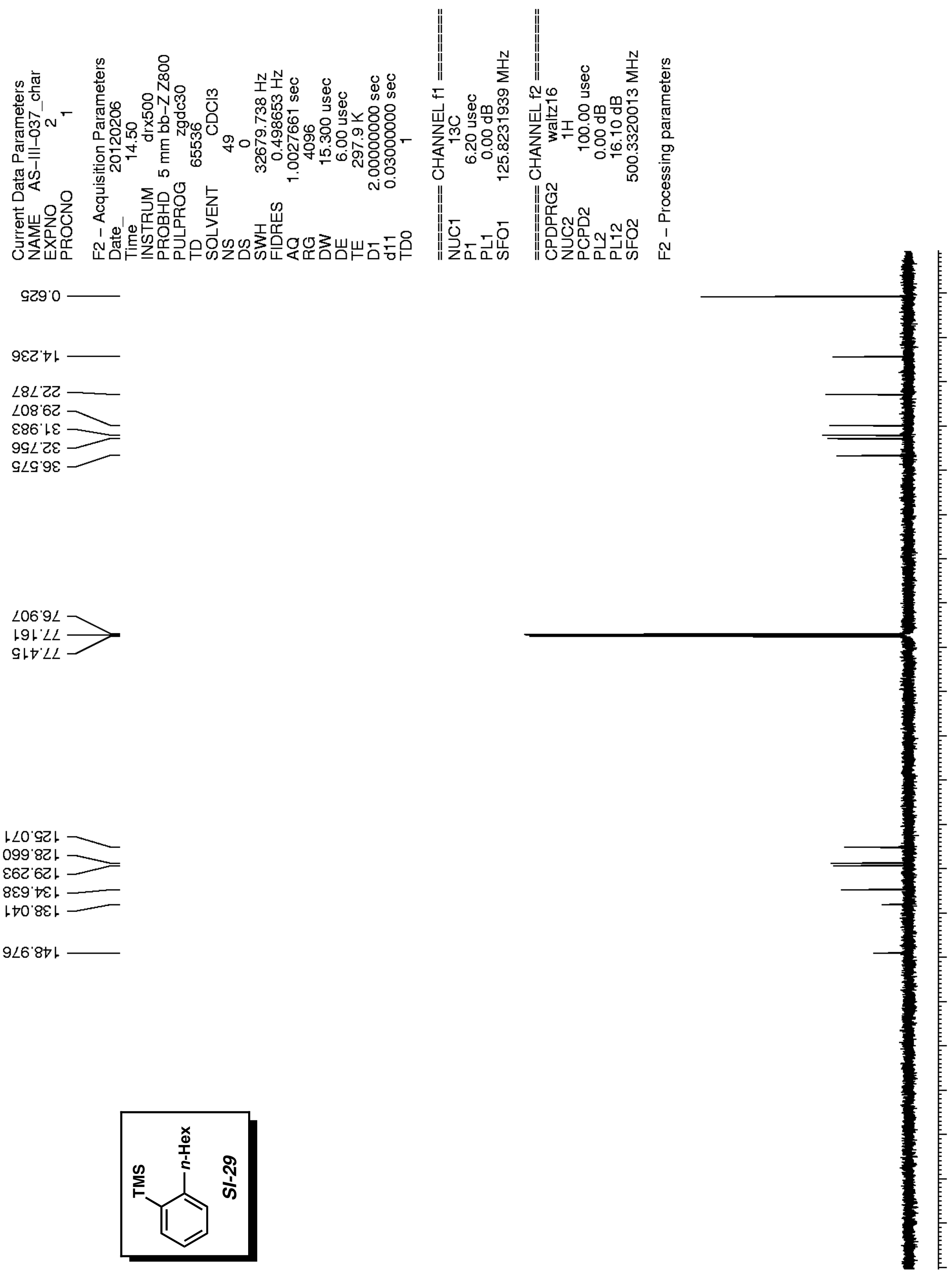


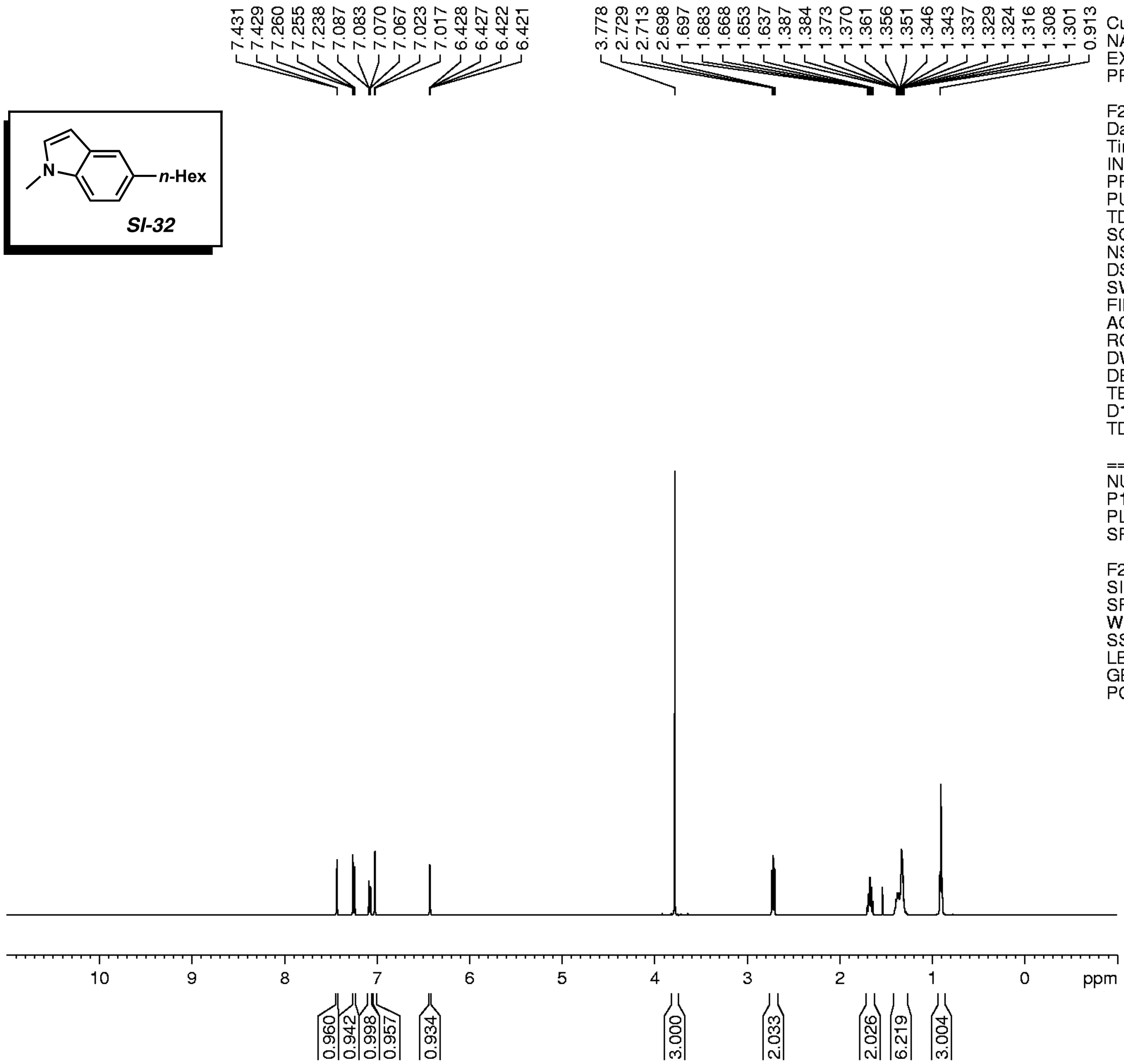
Current Data Parameters
NAME AS–III–037_char
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20120206
Time 14.41
INSTRUM drx500
PROBHD 5 mm bb–Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 80.6
DW 50.000 usec
DE 6.00 usec
TE 297.4 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300219 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


Current Data Parameters
NAME AS–III–037_char
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20120206
Time 14.50
INSTRUM drx500
PROBHD 5 mm bb–Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 49
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
0.625
14.236
22.787
29.807
31.983
32.756
36.575
76.907
77.161
77.415
125.071
128.660
129.293
134.638
138.041
148.976
TMS
n-Hex
SI-29
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210

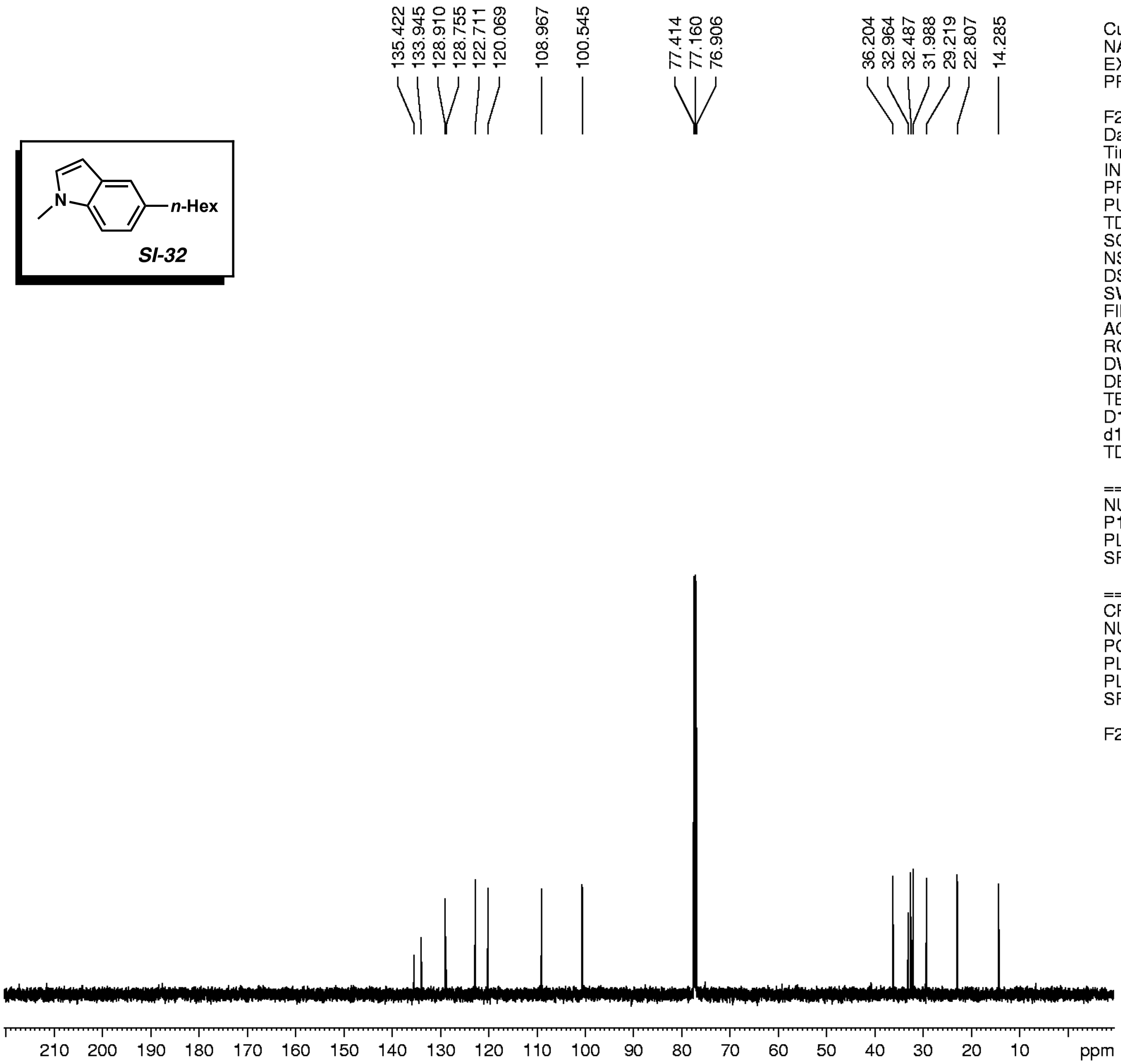


```
Current Data Parameters
NAME     AS-III-038_char
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20120206
Time            21.23
INSTRUM        drx500
PROBHD   5 mm bb-Z Z800
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                  8
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ        3.2769001 sec
RG                128
DW           50.000 usec
DE             6.00 usec
TE             297.8 K
D1       2.00000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1               1H
P1             12.25 usec
PL1             0.00 dB
SFO1     500.3330020 MHz

F2 - Processing parameters
SI              32768
SF        500.3300219 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```



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```
Current Data Parameters
NAME     AS-III-038_char
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_         20120206
Time             21.29
INSTRUM         drx500
PROBHD   5 mm bb-Z Z800
PULPROG        zgdc30
TD              65536
SOLVENT          CDCl3
NS                  56
DS                   0
SWH         32679.738 Hz
FIDRES       0.498653 Hz
AQ         1.0027661 sec
RG               13004
DW            15.300 usec
DE              6.00 usec
TE             298.2 K
D1        2.00000000 sec
d11       0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1               6.20 usec
PL1              0.00 dB
SFO1      125.8231939 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2          100.00 usec
PL2            120.00 dB
PL12            16.10 dB
SFO2      500.3320013 MHz

F2 - Processing parameters
```

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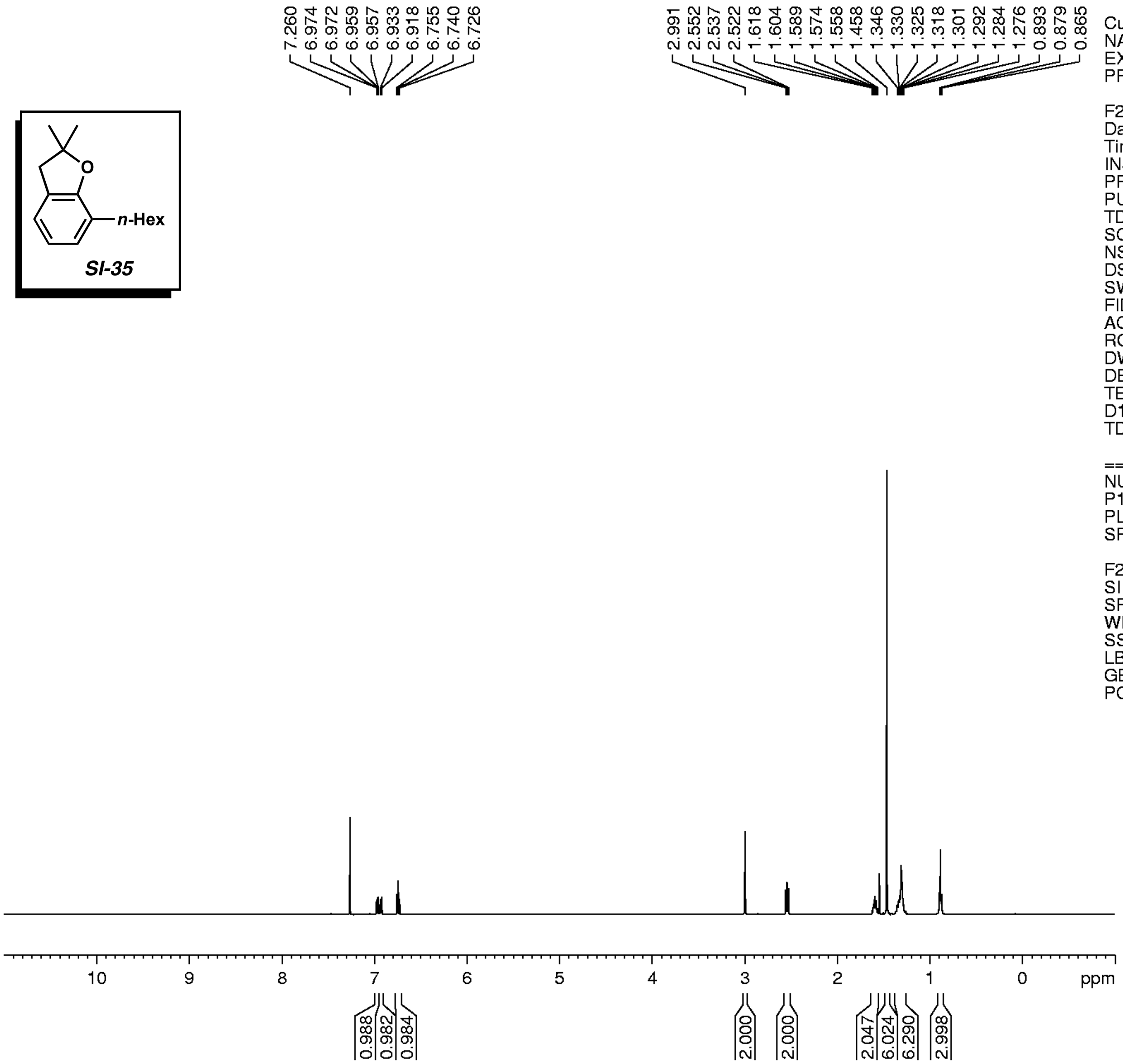
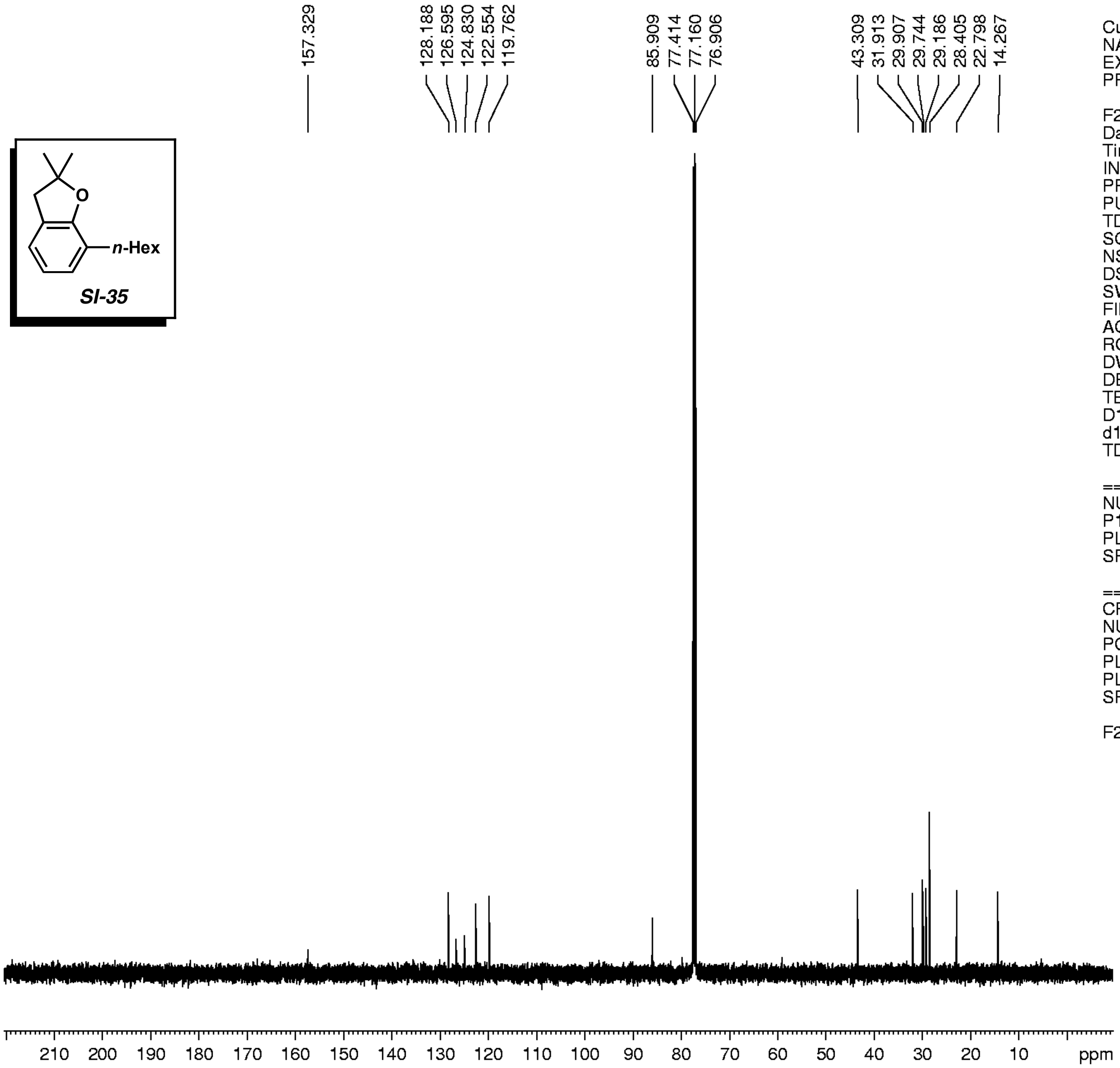


```
Current Data Parameters
NAME     AS-III-053_char
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_       20120206
Time           20.58
INSTRUM       drx500
PROBHD   5 mm bb-Z Z800
PULPROG         zg30
TD             65536
SOLVENT        CDCl3
NS                16
DS                 0
SWH       10000.000 Hz
FIDRES     0.152588 Hz
AQ        3.2769001 sec
RG             203.2
DW            50.000 usec
DE              6.00 usec
TE             297.6 K
D1        2.00000000 sec
TD0                1

======== CHANNEL f1 ========
NUC1              1H
P1             12.25 usec
PL1             0.00 dB
SFO1     500.3330020 MHz

F2 - Processing parameters
SI             32768
SF       500.3300219 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              1.00
```



SI-35

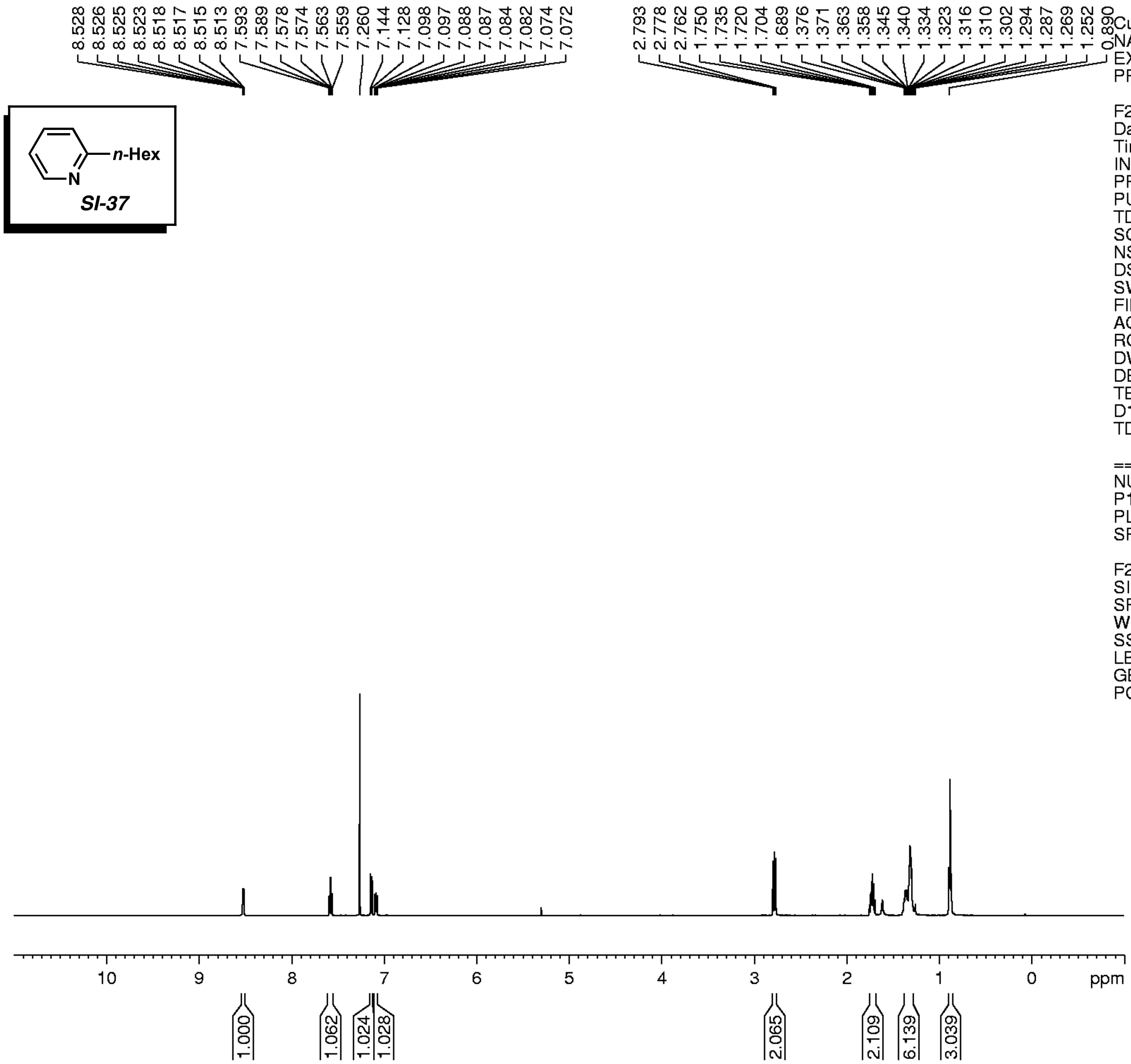
```
Current Data Parameters
NAME     AS-III-053_char
EXPNO              4
PROCNO             1

F2 - Acquisition Parameters
Date_        20120206
Time            21.19
INSTRUM        drx500
PROBHD   5 mm bb-Z Z800
PULPROG        zgdc30
TD             65536
SOLVENT         CDCl3
NS               149
DS                 0
SWH        32679.738 Hz
FIDRES      0.498653 Hz
AQ         1.0027661 sec
RG             13004
DW            15.300 usec
DE              6.00 usec
TE             298.2 K
D1        2.00000000 sec
d11       0.03000000 sec
TD0                1

======== CHANNEL f1 ========
NUC1             13C
P1              6.20 usec
PL1             0.00 dB
SFO1     125.8231939 MHz

======== CHANNEL f2 ========
CPDPRG2       waltz16
NUC2              1H
PCPD2         100.00 usec
PL2             0.00 dB
PL12           16.10 dB
SFO2     500.3320013 MHz

F2 - Processing parameters
```

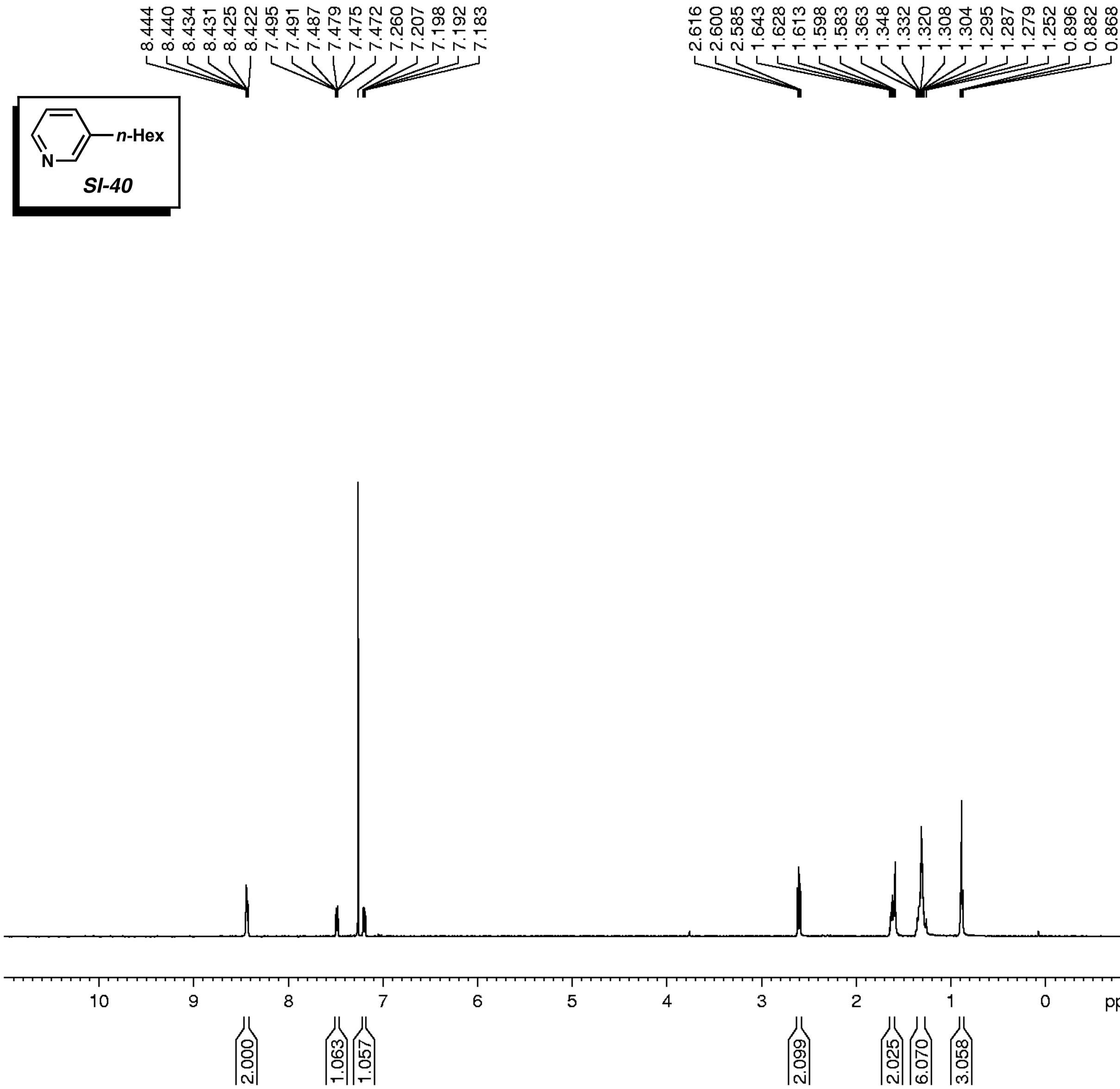


```
Current Data Parameters
NAME     AS-III-051_char
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_         20120206
Time             14.26
INSTRUM         drx500
PROBHD   5 mm bb-Z Z800
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   9
DS                   0
SWH         10000.000 Hz
FIDRES       0.152588 Hz
AQ          3.2769001 sec
RG               203.2
DW              50.000 usec
DE                6.00 usec
TE               297.4 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               12.25 usec
PL1               0.00 dB
SFO1       500.3330020 MHz

F2 - Processing parameters
SI               32768
SF         500.3300219 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

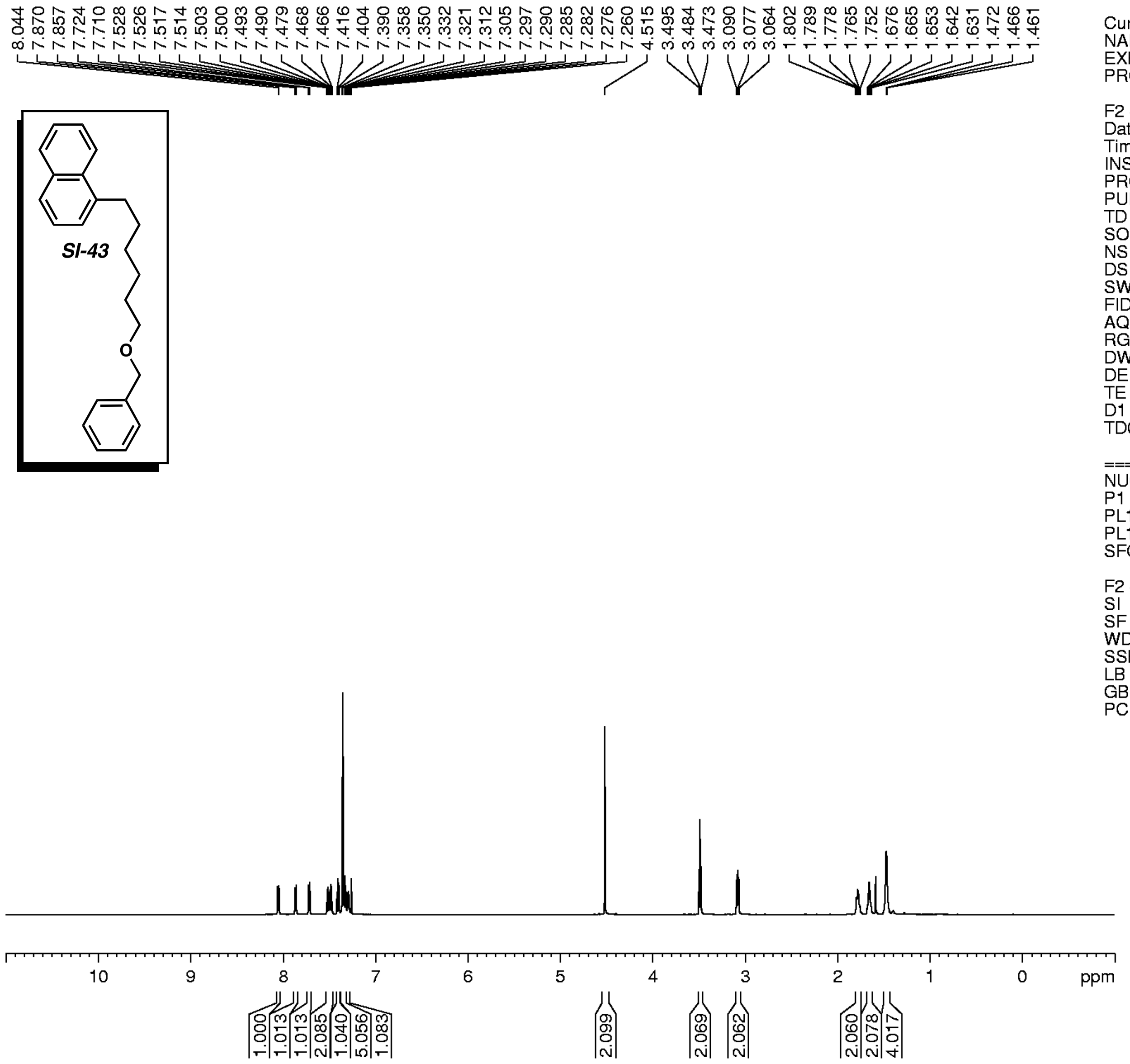


```
Current Data Parameters
NAME     AS-III-052_char
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_       20120206
Time           14.33
INSTRUM       drx500
PROBHD   5 mm bb-Z Z800
PULPROG         zg30
TD             65536
SOLVENT        CDCl3
NS                 8
DS                 0
SWH       10000.000 Hz
FIDRES     0.152588 Hz
AQ        3.2769001 sec
RG               256
DW          50.000 usec
DE            6.00 usec
TE             297.4 K
D1       2.00000000 sec
TD0                1

======== CHANNEL f1 ========
NUC1              1H
P1           12.25 usec
PL1            0.00 dB
SFO1    500.3330020 MHz

F2 - Processing parameters
SI             32768
SF      500.3300222 MHz
WDW               EM
SSB                0
LB           0.30 Hz
GB                 0
PC              1.00
```



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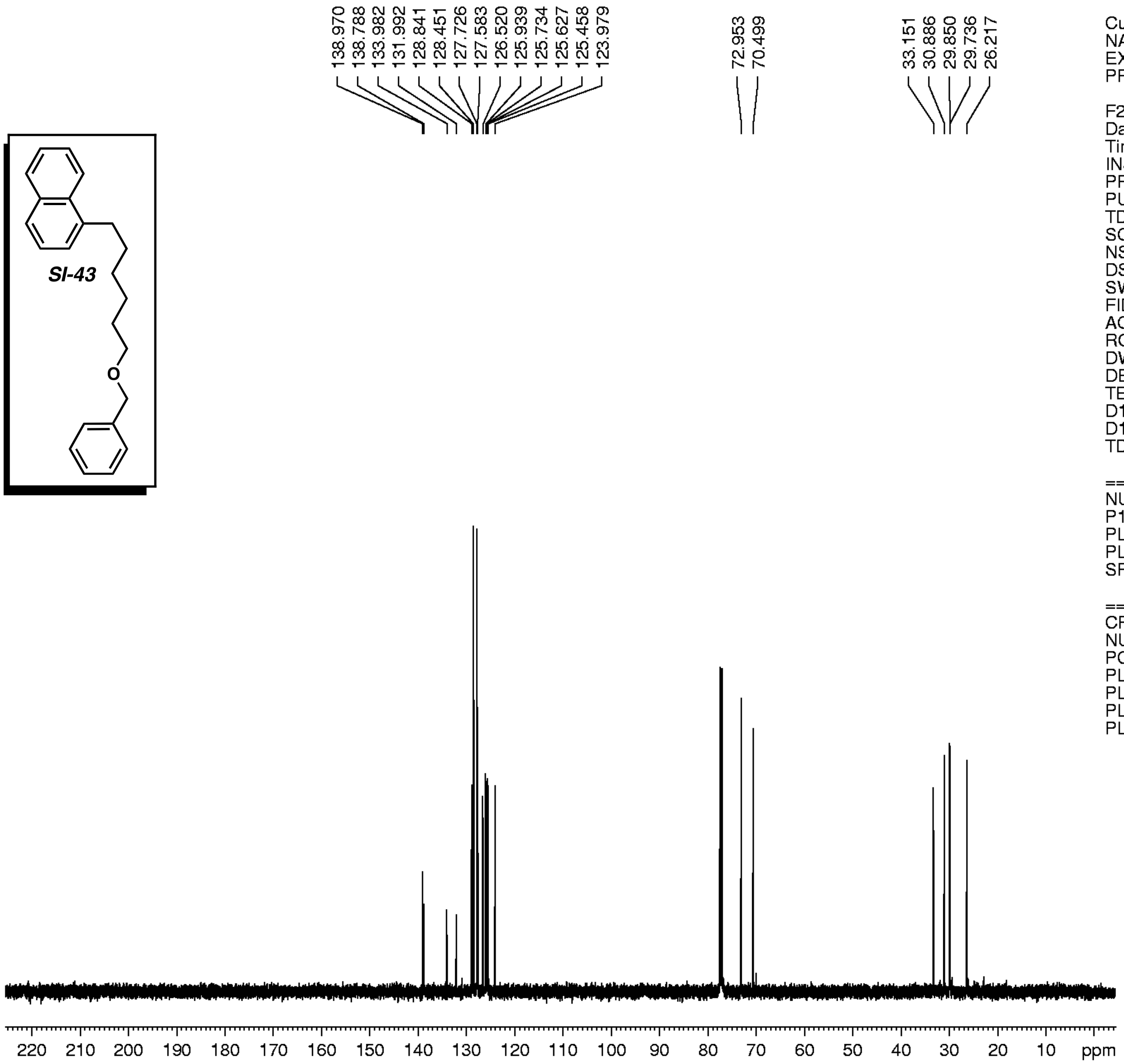
```
Current Data Parameters
NAME         SDR4-061
EXPNO              11
PROCNO              1

F2 - Acquisition Parameters
Date_        20111026
Time            20.17
INSTRUM         av600
PROBHD   5 mm BB5
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 16
DS                  0
SWH        12376.237 Hz
FIDRES      0.188846 Hz
AQ         2.6477449 sec
RG                114
DW             40.400 usec
DE               6.50 usec
TE              294.0 K
D1         2.00000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1               1H
P1              14.00 usec
PL1             -1.00 dB
PL1W      31.62277603 W
SFO1     600.1336008 MHz

F2 - Processing parameters
SI              65536
SF       600.1300286 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```



Current Data Parameters

NAME	SDR4–061
EXPNO	3
PROCNO	1

F2 – Acquisition Parameters

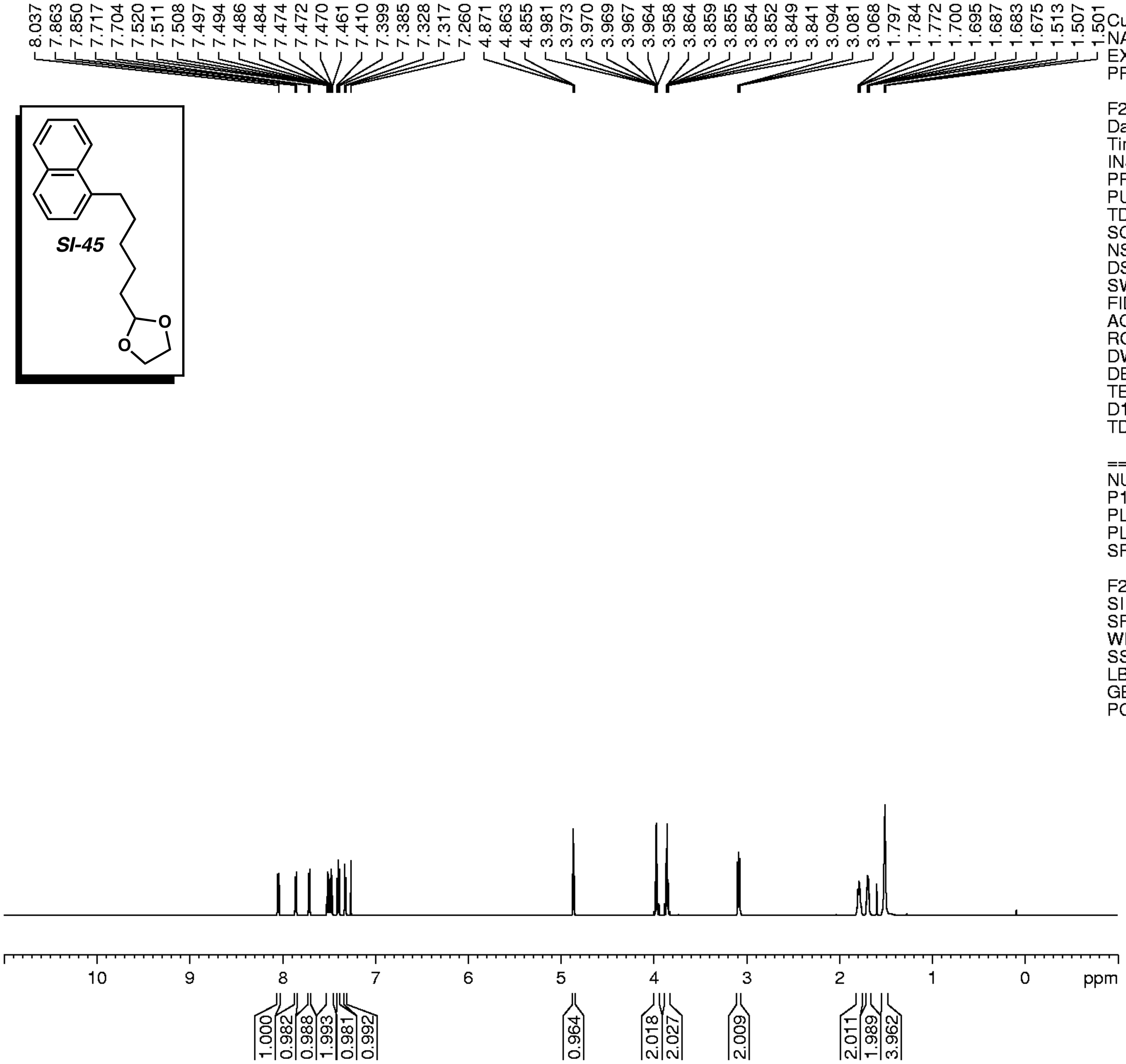
Date_	20111024
Time	20.17
INSTRUM	av600
PROBHD	5 mm BB5
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl3
NS	51
DS	0
SWH	37593.984 Hz
FIDRES	0.573639 Hz
AQ	0.8716921 sec
RG	18390.4
DW	13.300 usec
DE	6.50 usec
TE	294.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

NUC1	13C
P1	9.75 usec
PL1	0.00 dB
PL1W	75.35659027 W
SFO1	150.9209173 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	–1.00 dB
PL12	14.14 dB
PL2W	31.62277603 W
PL12W	0.96827775 W

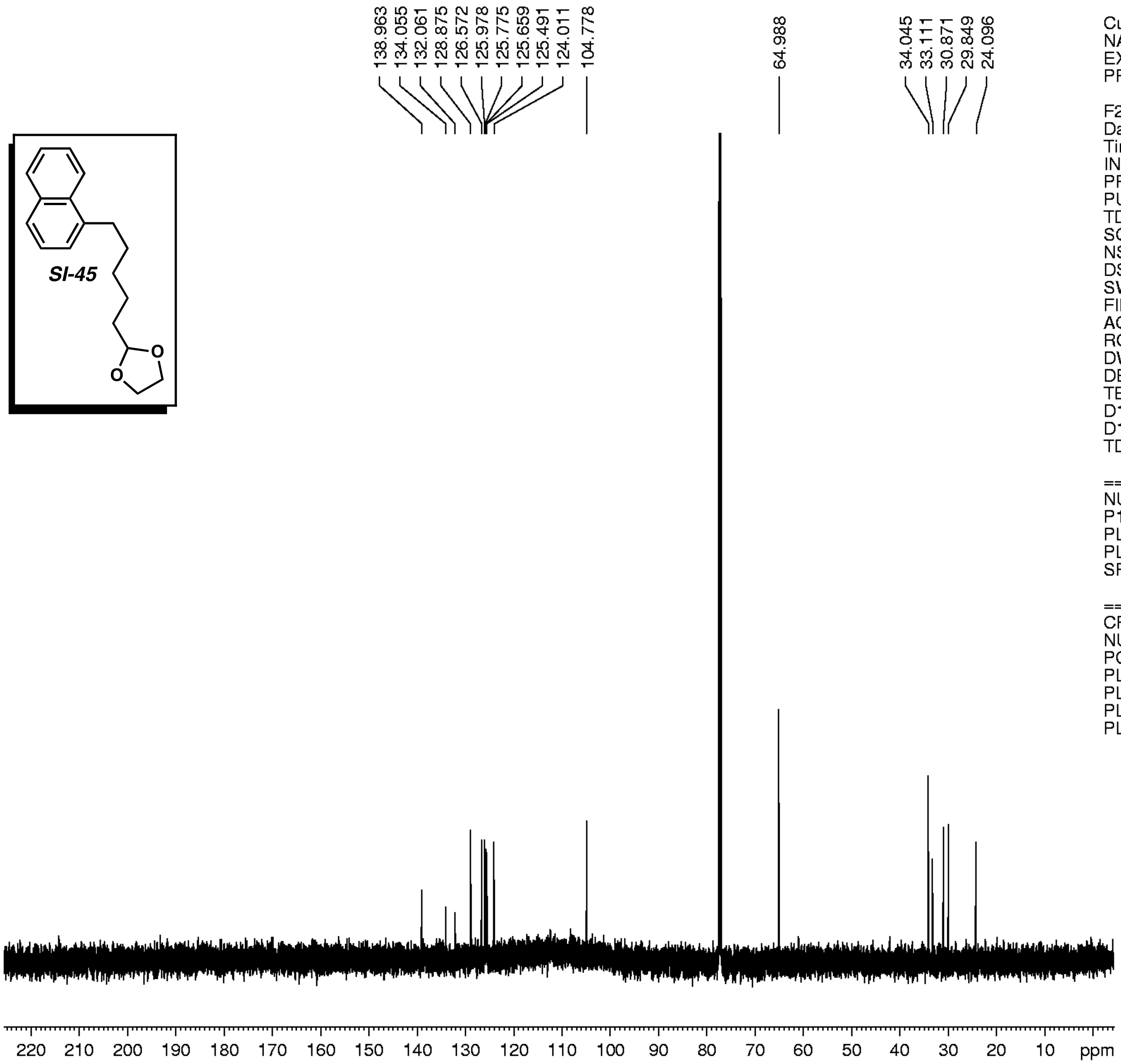


```
Current Data Parameters
NAME          SDR4-134
EXPNO               12
PROCNO               1

F2 - Acquisition Parameters
Date_         20111216
Time              9.19
INSTRUM          av600
PROBHD   5 mm TBI5
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   0
SWH         12376.237 Hz
FIDRES       0.188846 Hz
AQ          2.6477449 sec
RG                  64
DW              40.400 usec
DE               6.50 usec
TE               294.8 K
D1         2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                9.10 usec
PL1              -2.00 dB
PL1W       39.81071854 W
SFO1      600.1336008 MHz

F2 - Processing parameters
SI               65536
SF        600.1300293 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

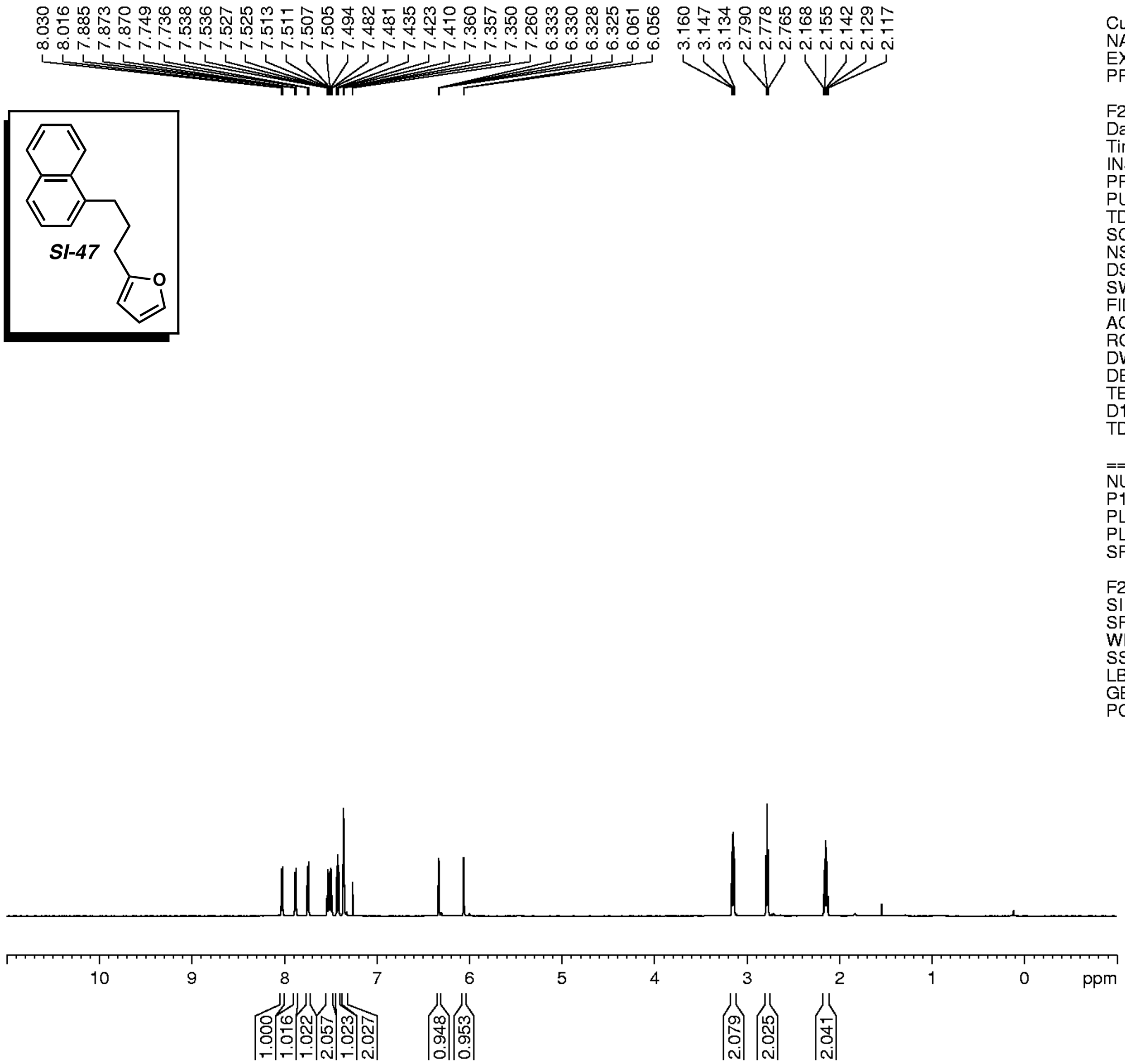


```
Current Data Parameters
NAME          SDR4-134
EXPNO              112
PROCNO               1

F2 - Acquisition Parameters
Date_         20111216
Time              9.27
INSTRUM          av600
PROBHD   5 mm TBI5
PULPROG        zgdc30
TD              65536
SOLVENT          CDCl3
NS                 212
DS                   0
SWH         37593.984 Hz
FIDRES       0.573639 Hz
AQ          0.8716921 sec
RG             18390.4
DW             13.300 usec
DE               6.50 usec
TE              294.9 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.75 usec
PL1               0.00 dB
PL1W       75.35659027 W
SFO1      150.9209173 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             14.14 dB
PL2W       31.62277603 W
PL12W       0.96827775 W
```



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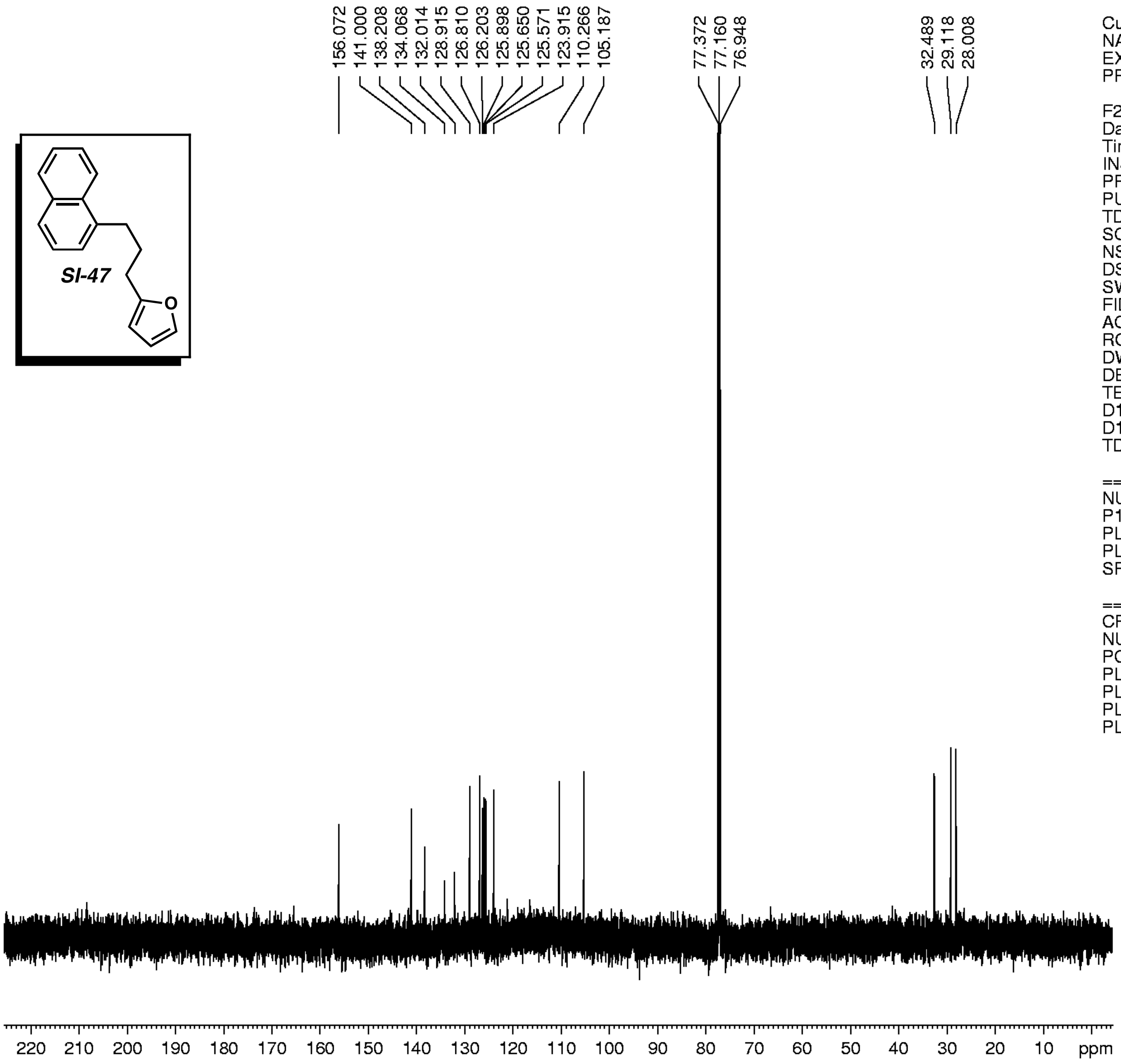
```
Current Data Parameters
NAME          SDR4-106
EXPNO               11
PROCNO               1

F2 - Acquisition Parameters
Date_          20111201
Time              11.00
INSTRUM           av600
PROBHD   5 mm TBI5
PULPROG            zg30
TD               65536
SOLVENT           CDCl3
NS                  16
DS                   0
SWH          12376.237 Hz
FIDRES        0.188846 Hz
AQ           2.6477449 sec
RG                  57
DW              40.400 usec
DE                6.50 usec
TE               294.1 K
D1         2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                9.10 usec
PL1              -2.00 dB
PL1W       39.81071854 W
SFO1      600.1336008 MHz

F2 - Processing parameters
SI               65536
SF        600.1300292 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

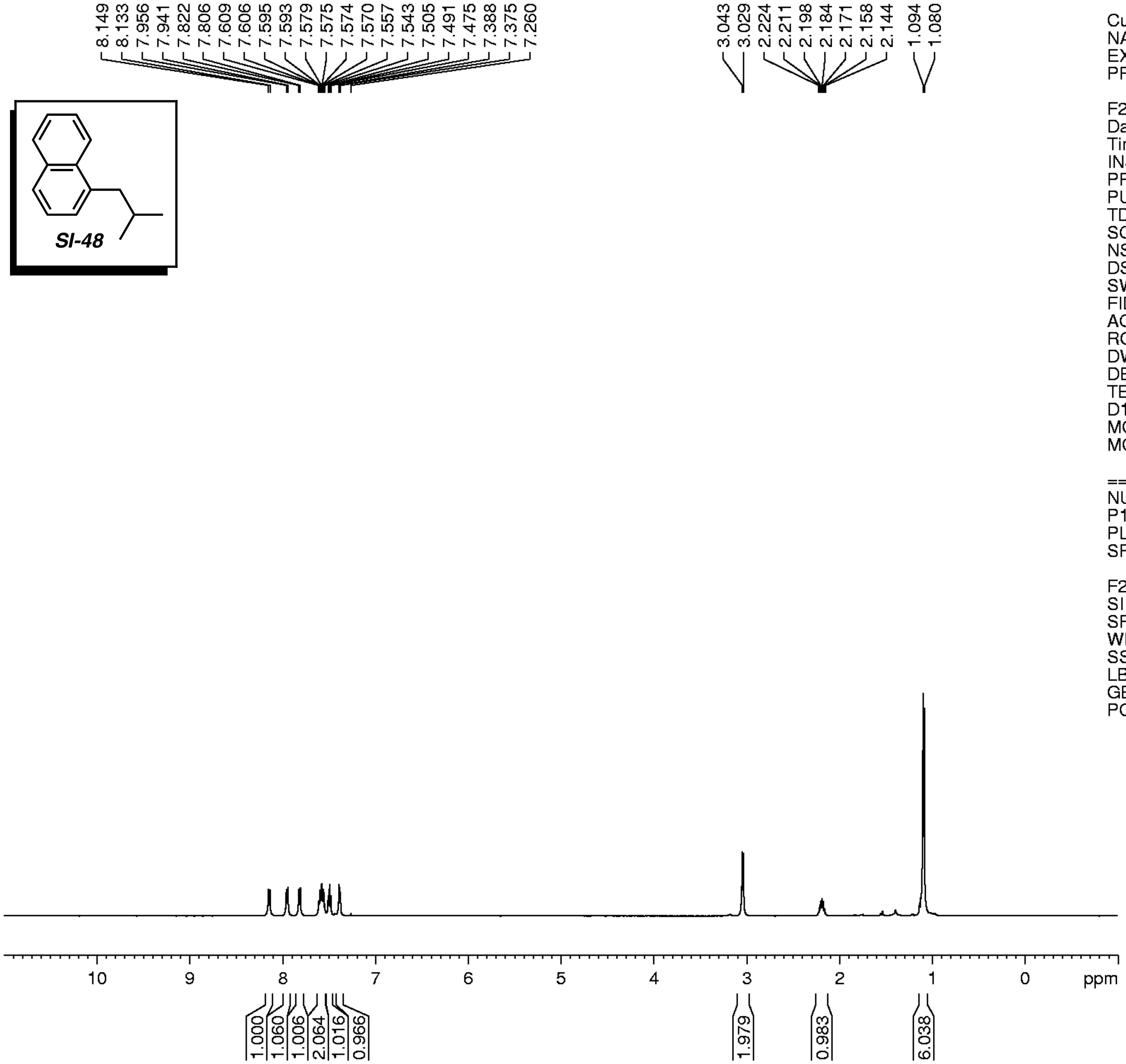


```
Current Data Parameters
NAME          SDR4-106
EXPNO              111
PROCNO               1

F2 - Acquisition Parameters
Date_         20111201
Time             11.11
INSTRUM          av600
PROBHD   5 mm TBI5
PULPROG         zgdc30
TD              65536
SOLVENT          CDCl3
NS                 100
DS                   0
SWH          37593.984 Hz
FIDRES        0.573639 Hz
AQ           0.8716921 sec
RG             20642.5
DW              13.300 usec
DE                6.50 usec
TE               294.3 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.75 usec
PL1               0.00 dB
PL1W       75.35659027 W
SFO1       150.9209173 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             14.14 dB
PL2W       31.62277603 W
PL12W       0.96827775 W
```

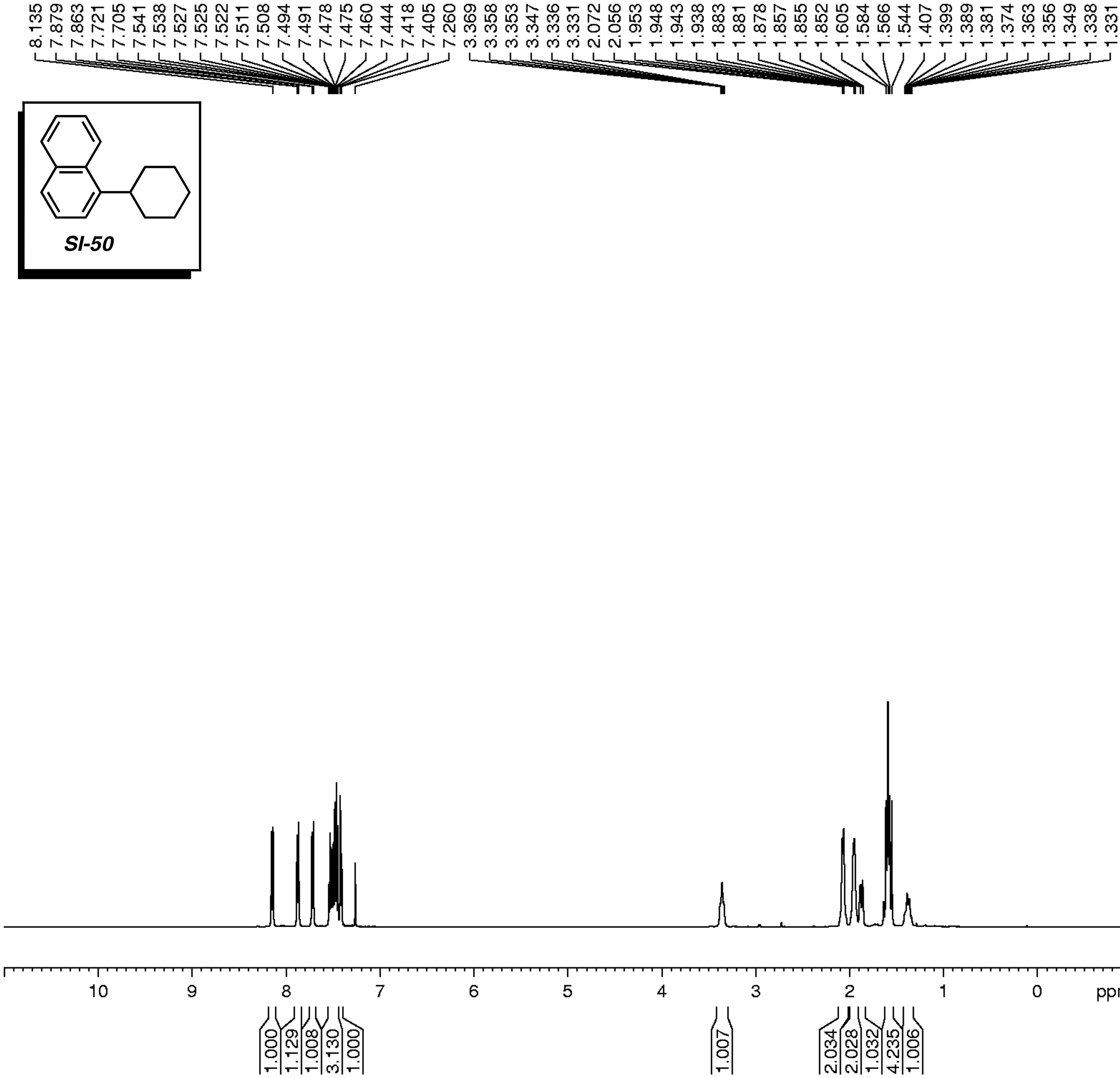


```
Current Data Parameters
NAME          SDR4-026
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20110929
Time              7.46
INSTRUM      avance500
PROBHD   5 mm bb-Z Z800
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   0
SWH         10000.000 Hz
FIDRES       0.152588 Hz
AQ          3.2769001 sec
RG                50.8
DW              50.000 usec
DE                6.00 usec
TE               296.5 K
D1          2.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1               12.25 usec
PL1               0.00 dB
SFO1       500.3330020 MHz

F2 - Processing parameters
SI               32768
SF         500.3300213 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

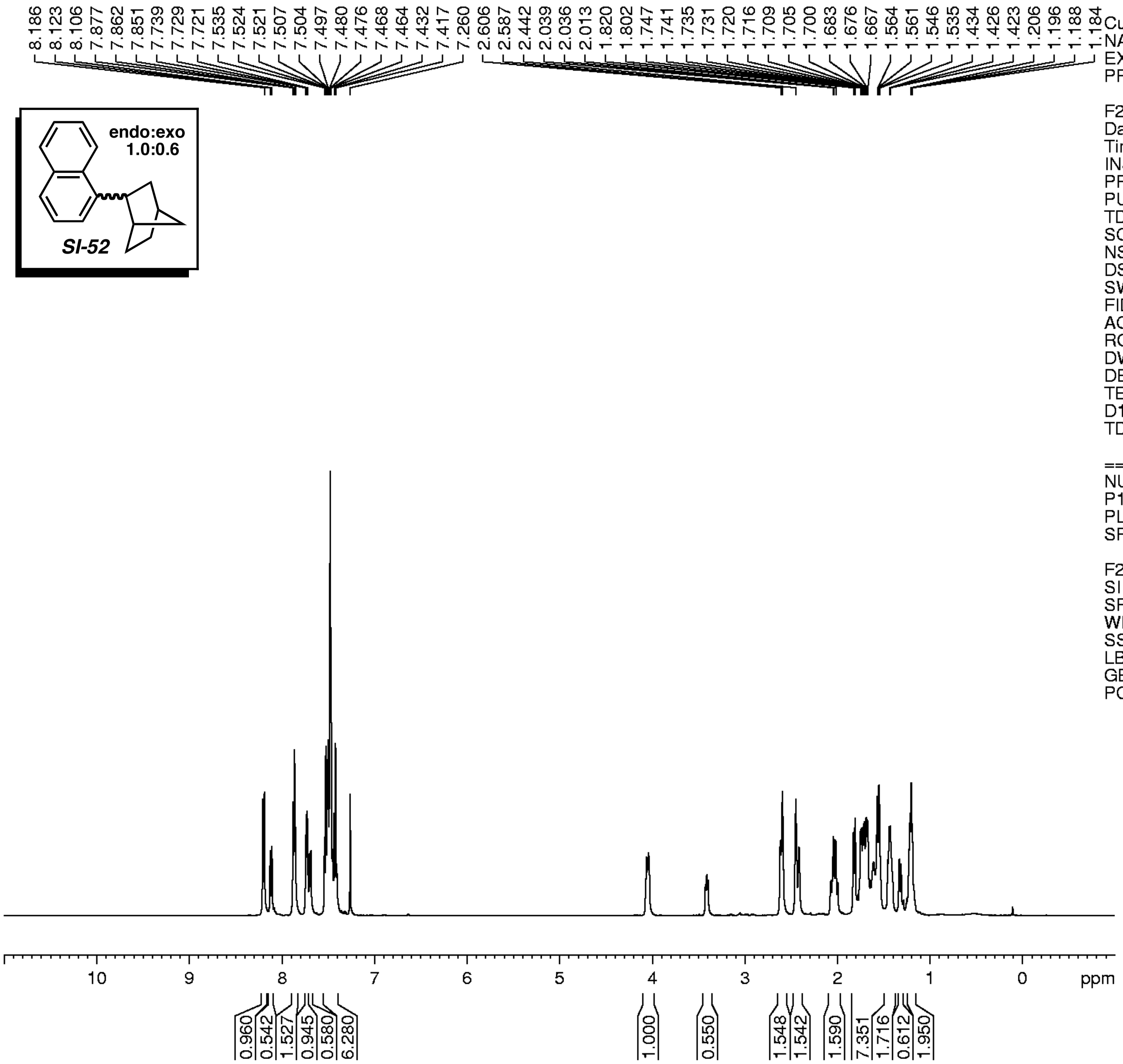


SI-50

Current Data Parameters	
NAME	SDR3–279
EXPNO	11
PROCNO	1

F2 – Acquisition Parameters	
Date_	20110920
Time	8.28
INSTRUM	arx500
PROBHD	5 mm broadban
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	715
DW	50.000 usec
DE	71.43 usec
TE	300.0 K
D1	2.00000000 sec
P1	12.20 usec
SFO1	500.1330008 MHz
NUCLEUS	1H

F2 – Processing parameters	
SI	32768
SF	500.1300234 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

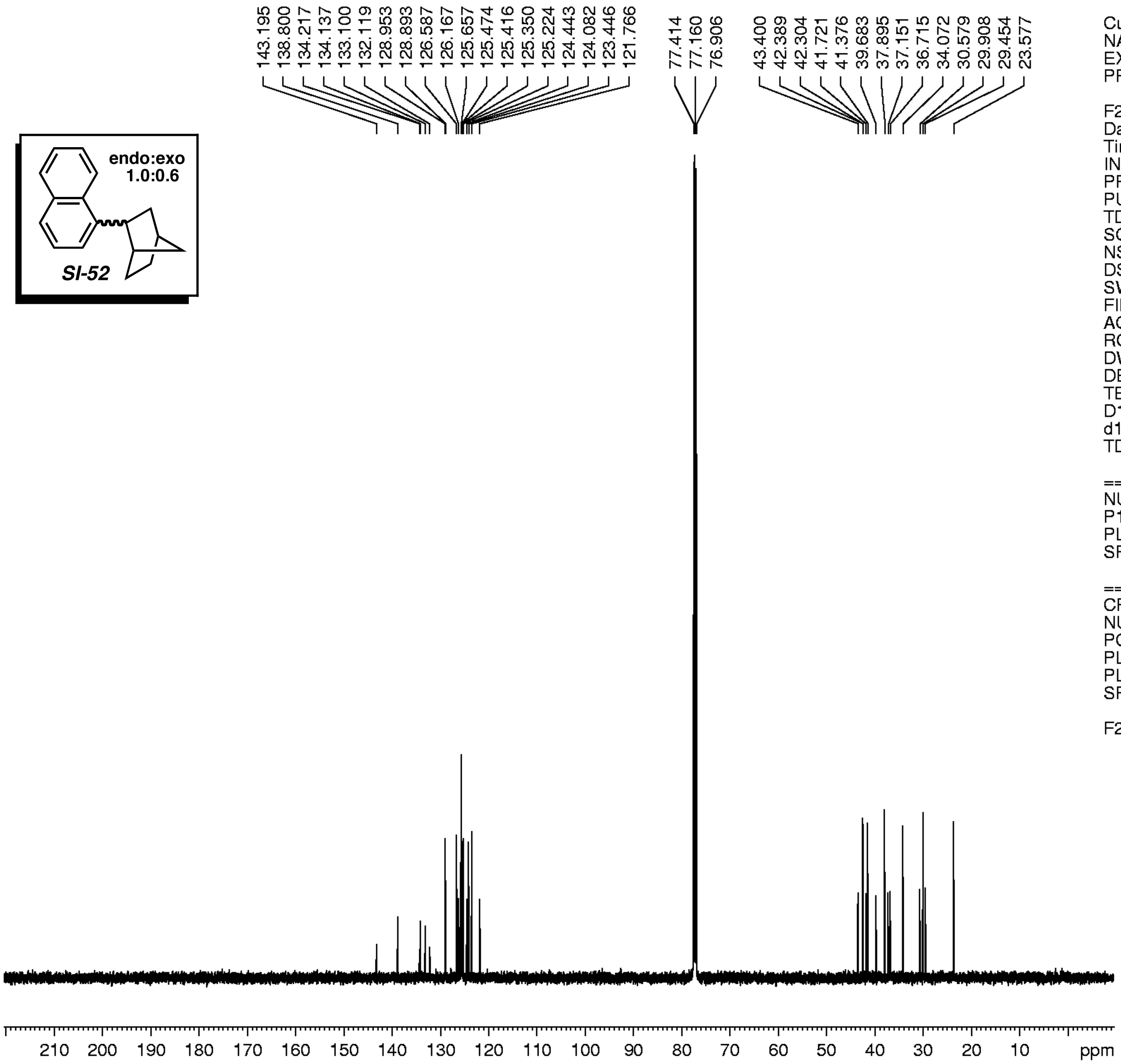


```
Current Data Parameters
NAME            SDR4-080
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111113
Time               10.02
INSTRUM           drx500
PROBHD   5 mm bb-Z Z800
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH         10000.000 Hz
FIDRES       0.152588 Hz
AQ         3.2769001 sec
RG                  80.6
DW            50.000 usec
DE             6.00 usec
TE               296.8 K
D1        2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1            12.25 usec
PL1              0.00 dB
SFO1      500.3330020 MHz

F2 - Processing parameters
SI                 32768
SF        500.3300220 MHz
WDW                   EM
SSB                    0
LB               0.30 Hz
GB                     0
PC                  1.00
```



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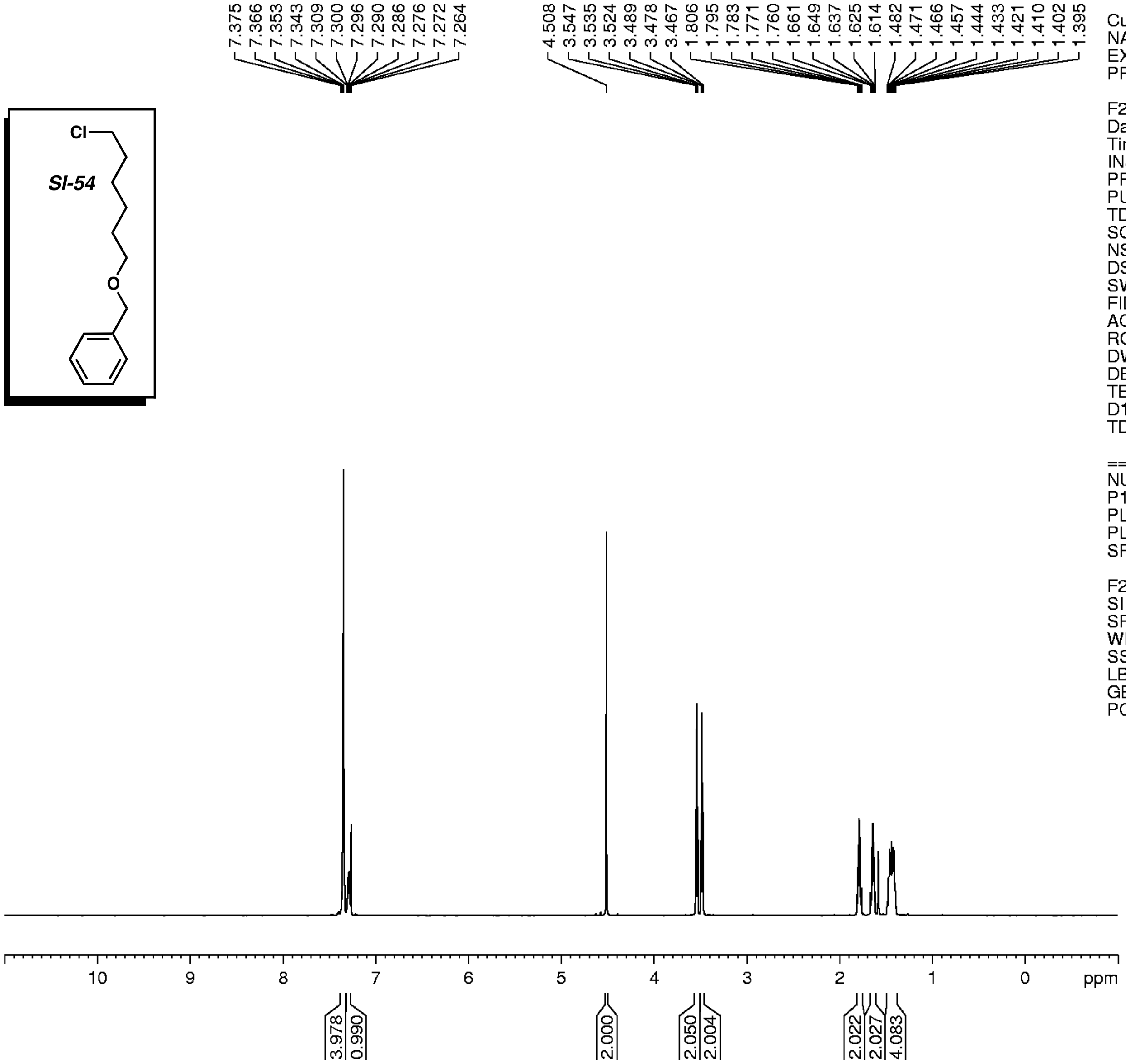
```
Current Data Parameters
NAME          SDR4-080
EXPNO              112
PROCNO               1

F2 - Acquisition Parameters
Date_         20111113
Time             10.06
INSTRUM         drx500
PROBHD   5 mm bb-Z Z800
PULPROG        zgdc30
TD               65536
SOLVENT          CDCl3
NS                 256
DS                   0
SWH         32679.738 Hz
FIDRES       0.498653 Hz
AQ          1.0027661 sec
RG                4096
DW              15.300 usec
DE                6.00 usec
TE               296.9 K
D1          2.00000000 sec
d11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                6.20 usec
PL1               0.00 dB
SFO1       125.8231939 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2             120.00 dB
PL12             16.10 dB
SFO2       500.3320013 MHz

F2 - Processing parameters
```



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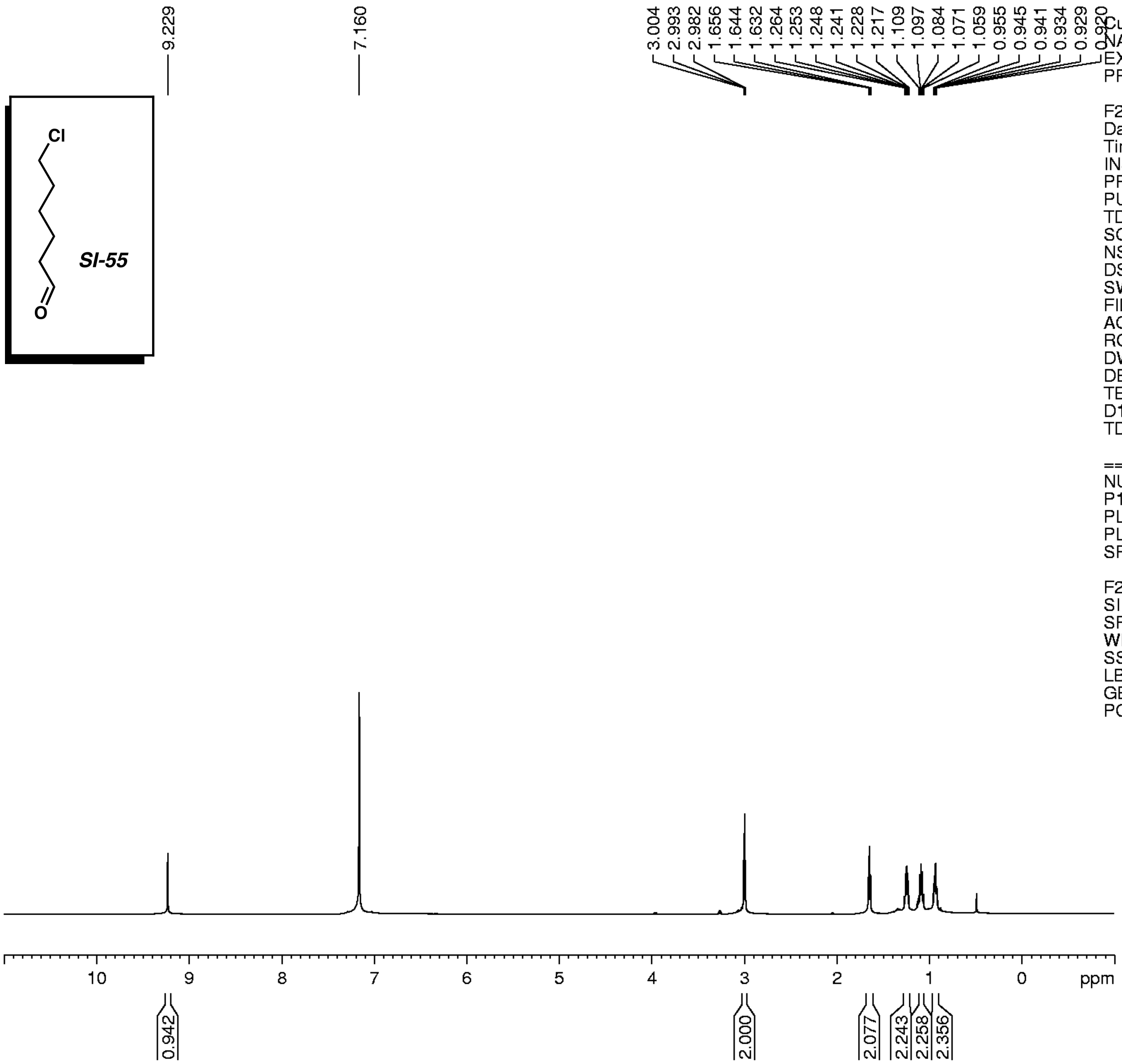
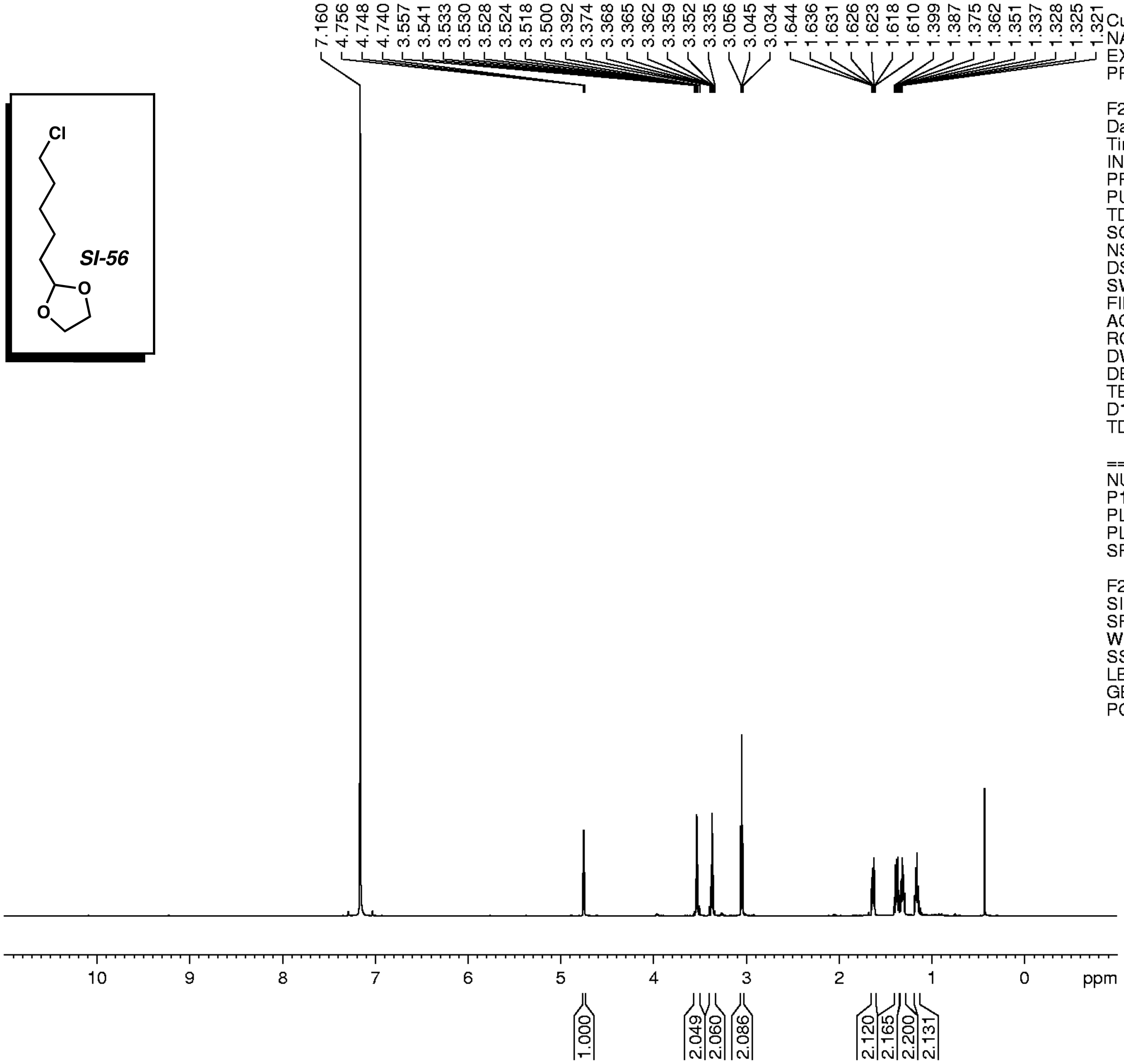
```
Current Data Parameters
NAME          SDR4-042
EXPNO                4
PROCNO               1

F2 - Acquisition Parameters
Date_          20111017
Time              18.34
INSTRUM           av600
PROBHD   5 mm TBI5
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH          12376.237 Hz
FIDRES        0.188846 Hz
AQ          2.6477449 sec
RG                  114
DW               40.400 usec
DE                 6.50 usec
TE                294.0 K
D1         2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.10 usec
PL1               -2.00 dB
PL1W        39.81071854 W
SFO1      600.1336008 MHz

F2 - Processing parameters
SI                65536
SF        600.1300273 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

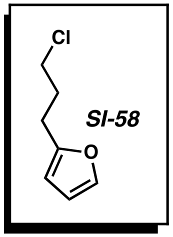


SI-55

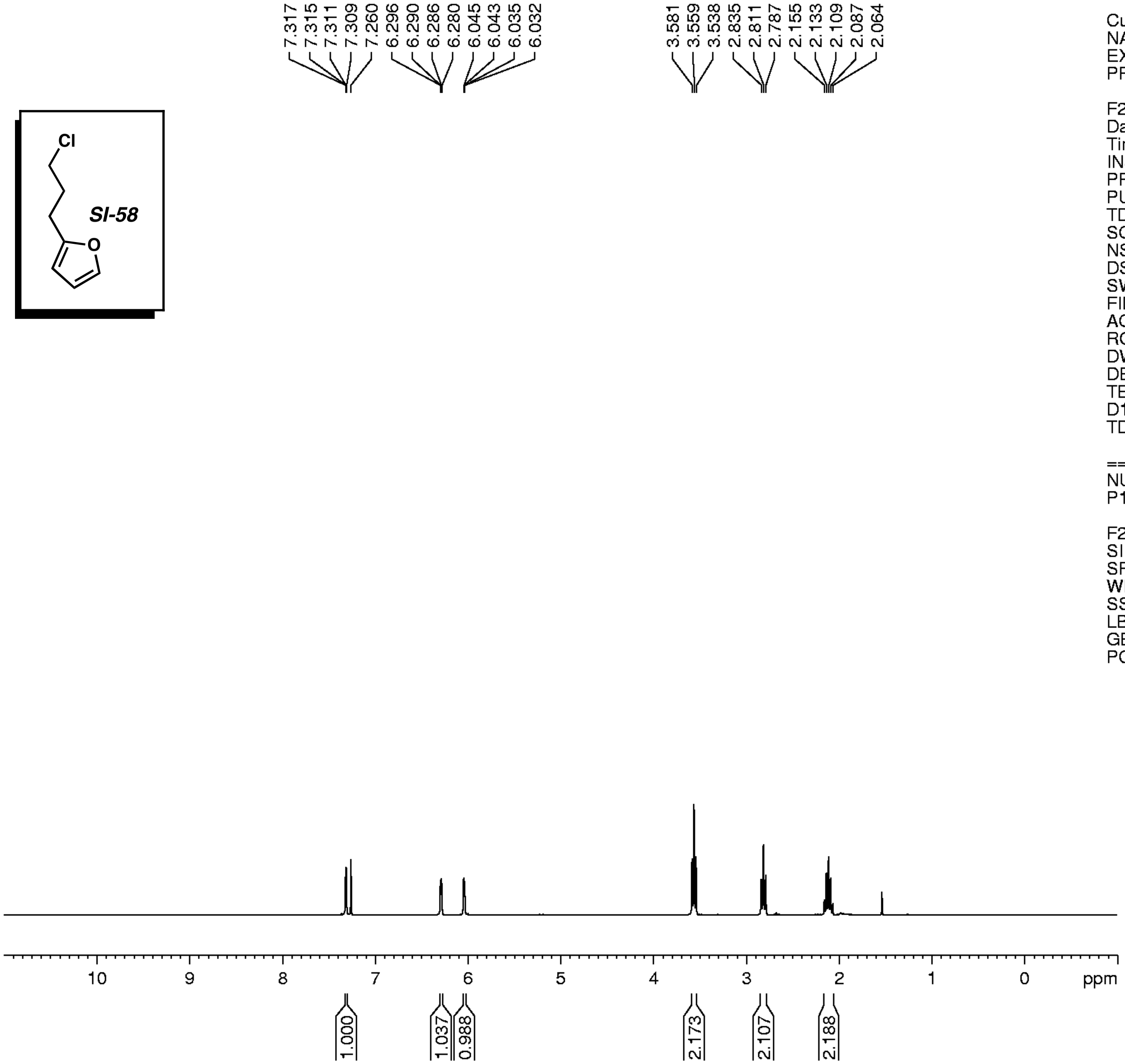
```
Current Data Parameters
NAME          SDR3-159
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_         20110628
Time             18.33
INSTRUM          av600
PROBHD   5 mm TBI5
PULPROG           zg30
TD               65536
SOLVENT           C6D6
NS                  16
DS                   0
SWH         12376.237 Hz
FIDRES       0.188846 Hz
AQ          2.6477449 sec
RG                90.5
DW              40.400 usec
DE                6.50 usec
TE               293.7 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                8.60 usec
PL1              -2.00 dB
PL1W       39.81071854 W
SFO1      600.1336008 MHz

F2 - Processing parameters
SI               65536
SF        600.1299974 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



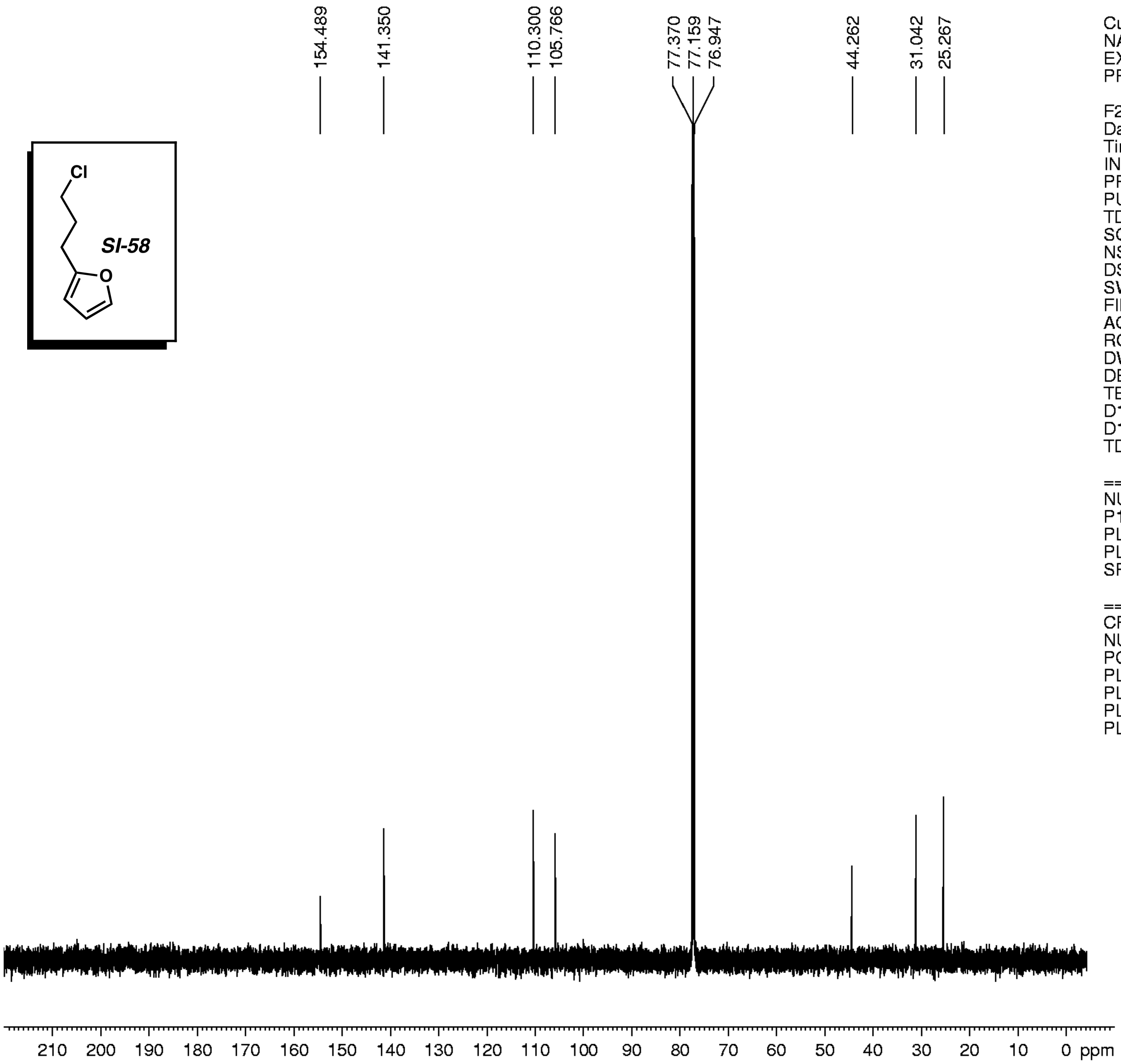
```
Current Data Parameters
NAME          SDR3-160
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_         20110701
Time             16.35
INSTRUM          av600
PROBHD   5 mm TBI5
PULPROG           zg30
TD               65536
SOLVENT           C6D6
NS                  16
DS                   0
SWH          12376.237 Hz
FIDRES        0.188846 Hz
AQ           2.6477449 sec
RG                 181
DW              40.400 usec
DE                6.50 usec
TE               293.6 K
D1         2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                8.60 usec
PL1              -2.00 dB
PL1W       39.81071854 W
SFO1     600.1336008 MHz

F2 - Processing parameters
SI               65536
SF       600.1299979 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



SI-58

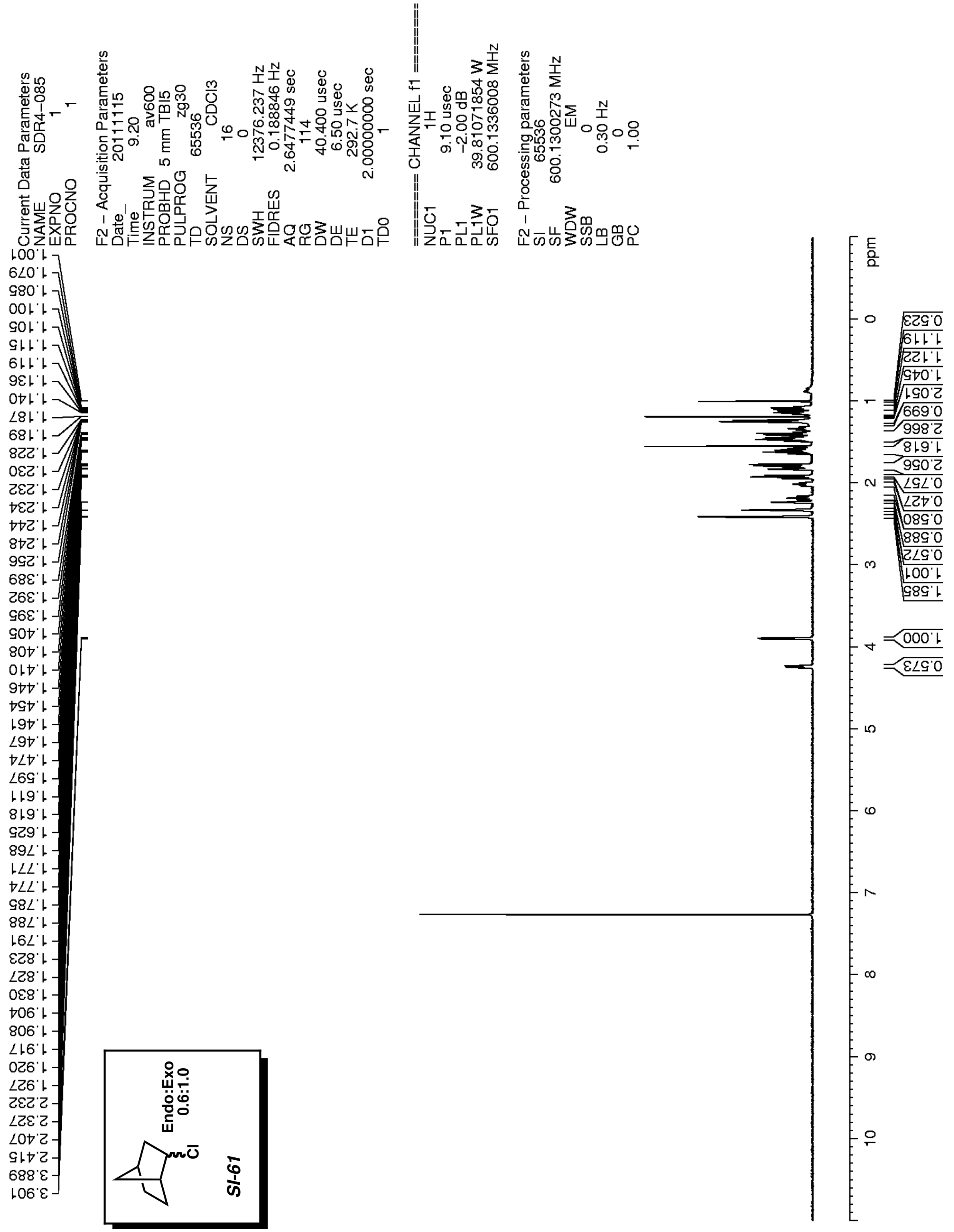


```
Current Data Parameters
NAME          SDR4-076
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_         20111114
Time              9.04
INSTRUM          av300
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   0
SWH        5995.204 Hz
FIDRES     0.091480 Hz
AQ        5.4657526 sec
RG               574.7
DW          83.400 usec
DE            6.50 usec
TE             297.8 K
D1      2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1          11.75 usec

F2 - Processing parameters
SI               65536
SF      300.1300119 MHz
WDW                 EM
SSB                  0
LB             0.30 Hz
GB                   0
PC                1.00
```



SI-58

```
Current Data Parameters
NAME          SDR4–072
EXPNO                3
PROCNO               1

F2 – Acquisition Parameters
Date_          20111107
Time              14.57
INSTRUM           av600
PROBHD   5 mm BB5
PULPROG         zgdc30
TD               65536
SOLVENT          CDCl3
NS                  30
DS                   0
SWH         37593.984 Hz
FIDRES       0.573639 Hz
AQ          0.8716921 sec
RG             20642.5
DW             13.300 usec
DE               6.50 usec
TE              294.2 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.75 usec
PL1               0.00 dB
PL1W       75.35659027 W
SFO1      150.9209173 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             14.14 dB
PL2W       31.62277603 W
PL12W       0.96827775 W
```

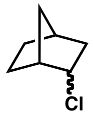


Current Data Parameters
NAME SDR4–085
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20111115
Time 9.20
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6477449 sec
RG 114
DW 40.400 usec
DE 6.50 usec
TE 292.7 K
D1 2.00000000 sec
TD0 1

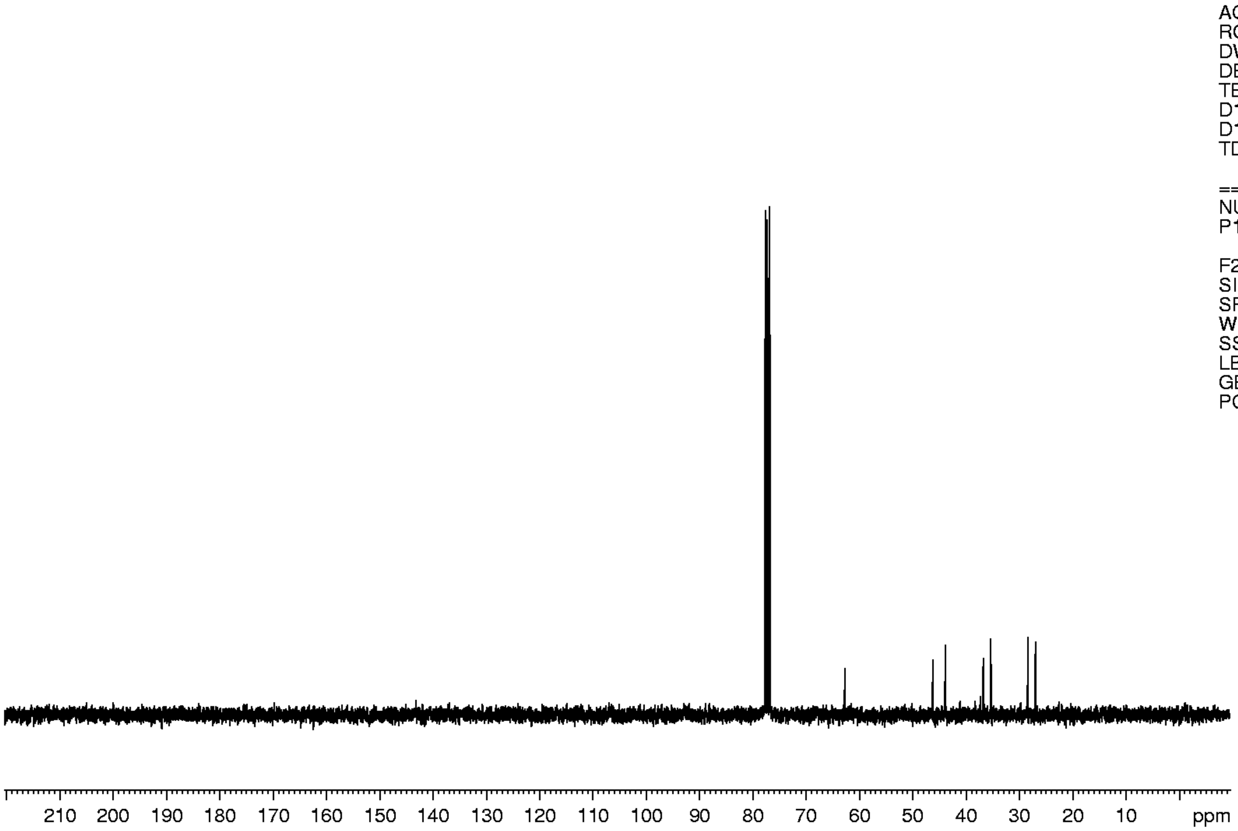
======== CHANNEL f1 ========
NUC1 1H
P1 9.10 usec
PL1 –2.00 dB
PL1W 39.81071854 W
SFO1 600.1336008 MHz

F2 – Processing parameters
SI 65536
SF 600.1300273 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

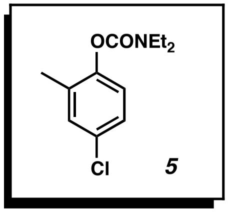


Endo:Exo
0.6:1.0

SI-61



```
Current Data Parameters
NAME          SDR4-032
EXPNO               12
PROCNO               1

F2 - Acquisition Parameters
Date_         20111005
Time              8.39
INSTRUM          av300
PROBHD   5 mm PABBO BB/
PULPROG         zgdc30
TD               65536
SOLVENT          CDCl3
NS                 126
DS                   0
SWH       18832.393 Hz
FIDRES     0.287360 Hz
AQ        1.7400308 sec
RG               26008
DW          26.550 usec
DE           6.50 usec
TE             297.1 K
D1      2.00000000 sec
D11     0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1            7.00 usec

F2 - Processing parameters
SI               32768
SF       75.4677391 MHz
WDW                 EM
SSB                  0
LB             1.00 Hz
GB                   0
PC                1.40
```



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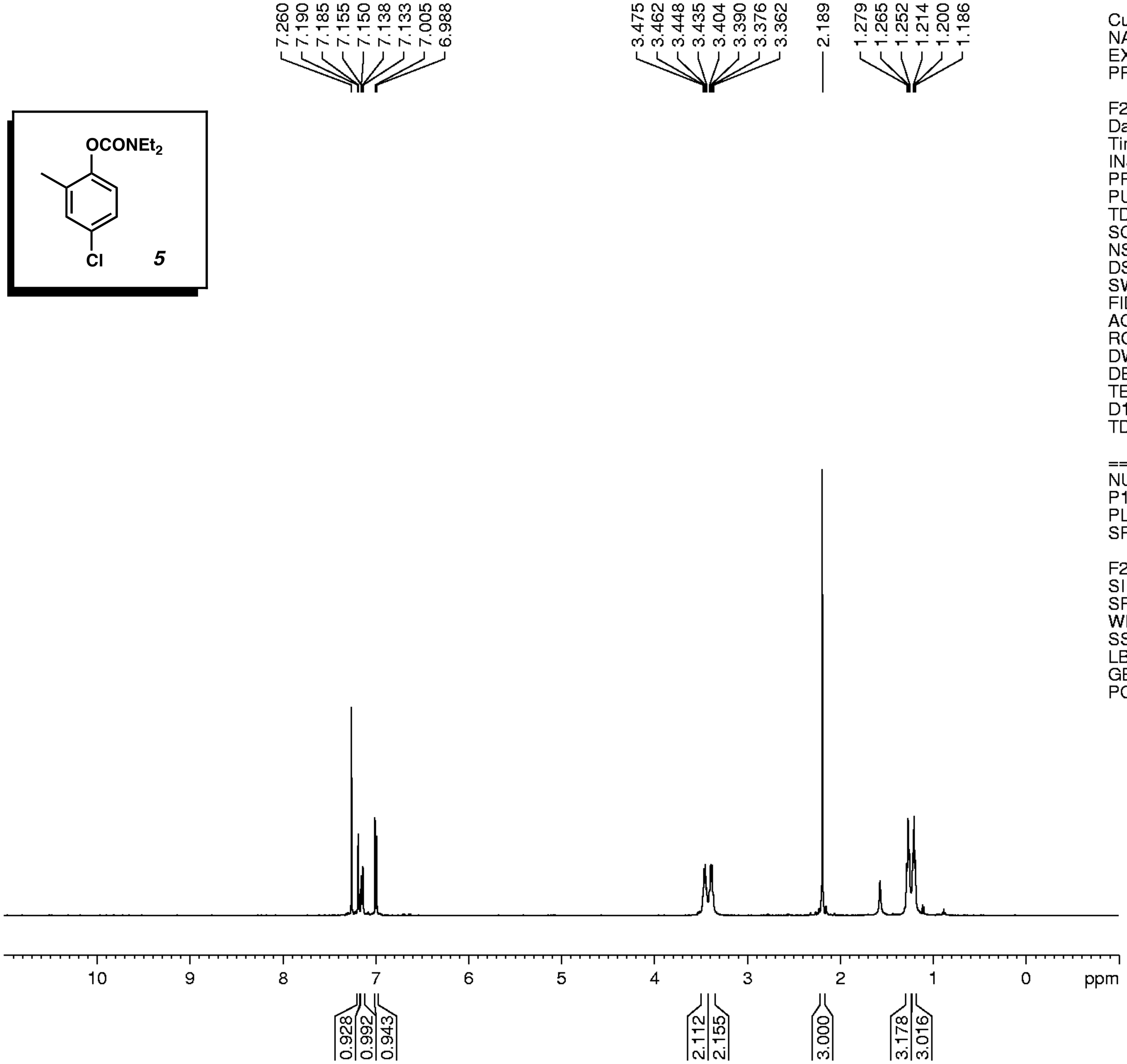


Current Data Parameters

NAME	AS–III–166
EXPNO	1
PROCNO	1

F2 – Acquisition Parameters

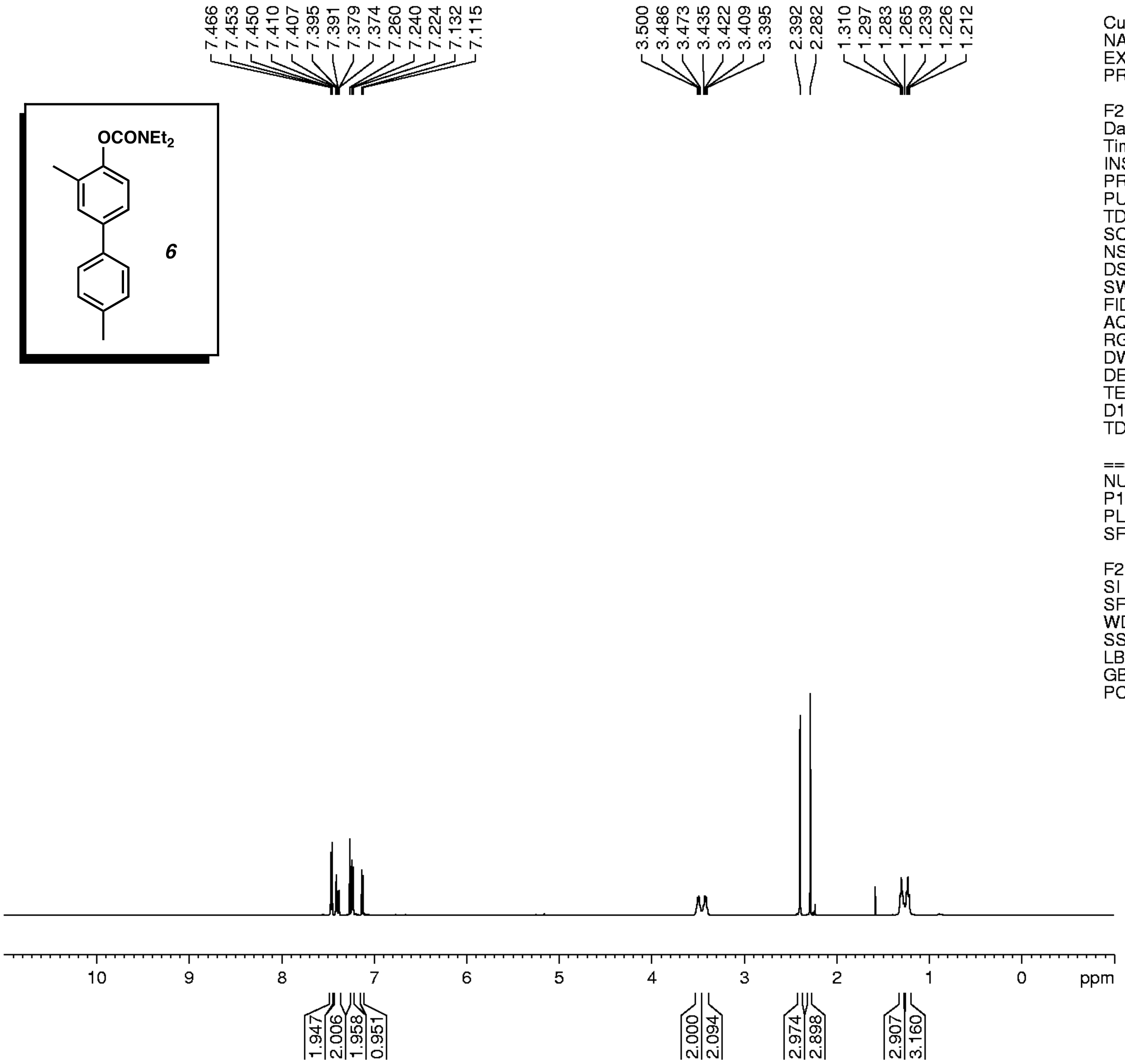
Date_	20111213
Time	21.23
INSTRUM	drx500
PROBHD	5 mm bb–Z Z800
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	10
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2769001 sec
RG	181
DW	50.000 usec
DE	6.00 usec
TE	297.2 K
D1	2.00000000 sec
TD0	1

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

NUC1	1H
P1	12.25 usec
PL1	0.00 dB
SFO1	500.3330020 MHz

F2 – Processing parameters

SI	32768
SF	500.3300222 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



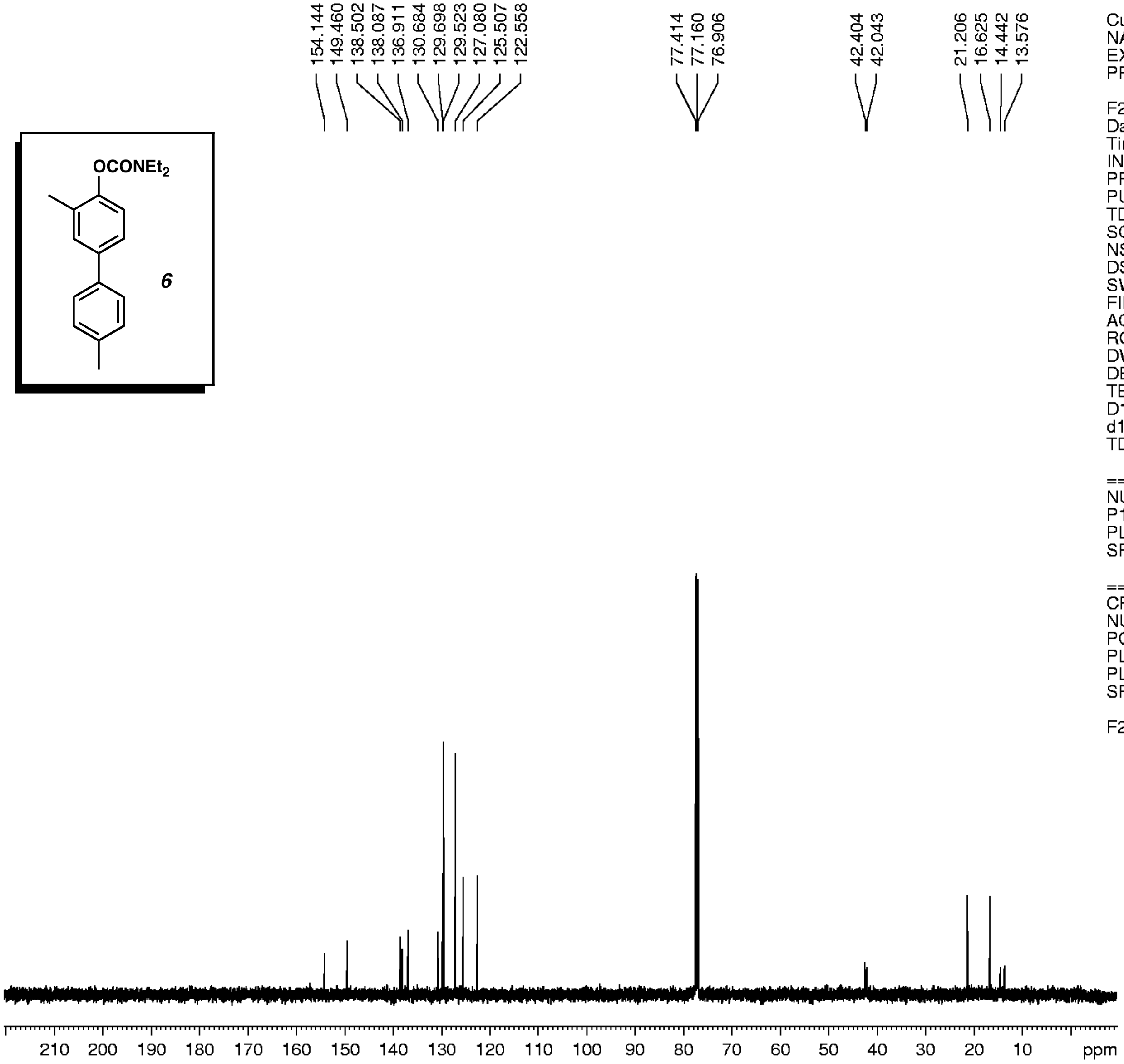
6

```
Current Data Parameters
NAME        AS-III-189
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20120112
Time            10.01
INSTRUM        drx500
PROBHD   5 mm bb-Z Z800
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                  8
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ        3.2769001 sec
RG                128
DW             50.000 usec
DE              6.00 usec
TE             296.1 K
D1       2.00000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1               1H
P1             12.25 usec
PL1             0.00 dB
SFO1      500.3330020 MHz

F2 - Processing parameters
SI              32768
SF        500.3300219 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```



6

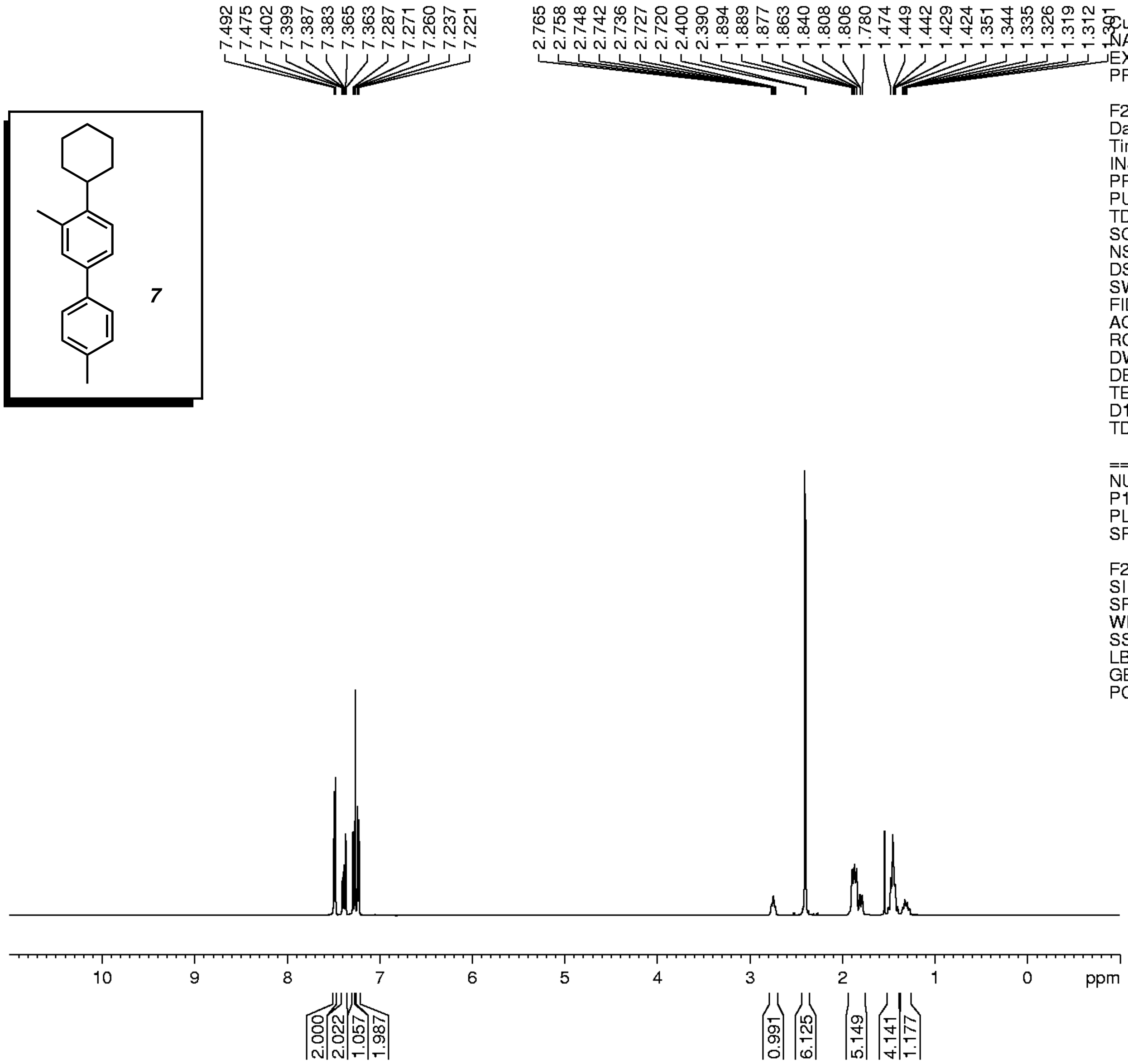
```
Current Data Parameters
NAME     AS-III-192_char
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_          20120207
Time              12.18
INSTRUM          drx500
PROBHD   5 mm bb-Z Z800
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                   51
DS                    0
SWH          32679.738 Hz
FIDRES        0.498653 Hz
AQ           1.0027661 sec
RG                13004
DW               15.300 usec
DE                 6.00 usec
TE                298.2 K
D1          2.00000000 sec
d11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 6.20 usec
PL1                0.00 dB
SFO1        125.8231939 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2              120.00 dB
PL12              16.10 dB
SFO2        500.3320013 MHz

F2 - Processing parameters
```



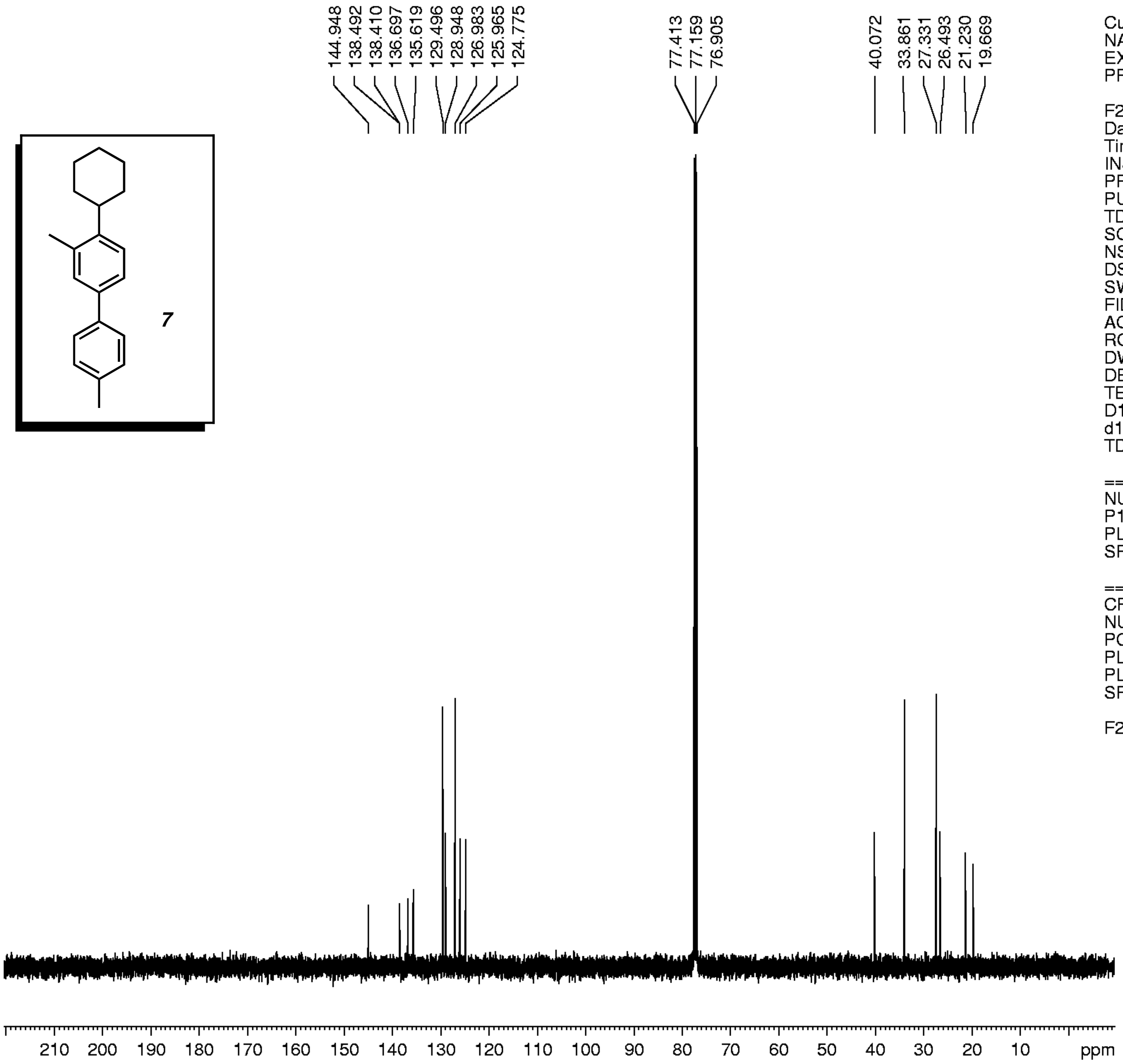
```
Current Data Parameters
NAME     AS-III-196.2_char
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20120206
Time            23.25
INSTRUM        drx500
PROBHD   5 mm bb-Z Z800
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                 16
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ         3.2769001 sec
RG              143.7
DW             50.000 usec
DE               6.00 usec
TE              297.6 K
D1         2.00000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1               1H
P1              12.25 usec
PL1              0.00 dB
SFO1      500.3330020 MHz

F2 - Processing parameters
SI              32768
SF        500.3300219 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```

7



```
Current Data Parameters
NAME     AS–III–196.1_char
EXPNO               2
PROCNO              1

F2 – Acquisition Parameters
Date_          20120206
Time             23.18
INSTRUM         drx500
PROBHD   5 mm bb–Z Z800
PULPROG         zgdc30
TD              65536
SOLVENT          CDCl3
NS                 65
DS                  0
SWH         32679.738 Hz
FIDRES       0.498653 Hz
AQ          1.0027661 sec
RG               4096
DW             15.300 usec
DE              6.00 usec
TE              298.1 K
D1         2.00000000 sec
d11        0.03000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1              13C
P1              6.20 usec
PL1              0.00 dB
SFO1       125.8231939 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2              1H
PCPD2          100.00 usec
PL2            120.00 dB
PL12            16.10 dB
SFO2       500.3320013 MHz

F2 – Processing parameters
```

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