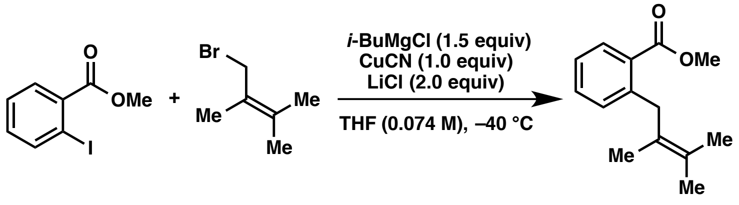
Supporting Information – Table of Contents

Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen and commercially obtained reagents were used as received. Non-commercially available substrates were synthesized following protocols specified in Section A in the Experimental Procedures. Prior to use, toluene was purified by distillation and taken through three freeze-pump-thaw cycles. 2-Halobenzoic acids derivatives **SI-6, SI-8, SI-10** were obtained from Combi-Blocks; **SI-4** and **SI-1** were obtained from Oakwood; **SI-12** was obtained from AstaTech; and **SI-16** was obtained from Ark Pharm. Ni(cod)$_2$ and Benz-ICy•HCl (**9**) were obtained from Strem Chemicals. Reductive coupling ligands **SI-35**[1] and **SI-41**[2] were prepared from known literature procedures. Reaction temperatures were controlled using an IKAmag temperature modulator, and unless stated otherwise, reactions were performed at room temperature (approximately 23 °C). Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm for analytical chromatography and 0.50 mm for preparative chromatography) and visualized using a combination of UV, anisaldehyde, iodine, and potassium permanganate staining techniques. Silicycle Siliaflash P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^{1}H NMR spectra were recorded on Bruker spectrometers (at 400 and 500 MHz) and are referenced to the residual solvent peak 7.26 ppm for CDCl$_3$. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz), integration.^{13}C NMR spectra were recorded on Bruker spectrometers (at 125 MHz) and are referenced to the residual solvent peak 77.16 ppm for CDCl$_3$. Data for ^{13}C NMR are reported as follows: chemical shift (δ ppm), multiplicity, and coupling constant (Hz). ^{19}F NMR spectra were recorded on Bruker spectrometers (at 376 MHz) and are reported in terms of chemical shift in CDCl$_3$. IR spectra were recorded on a Perkin-Elmer 100 spectrometer and are reported in terms of frequency absorption (cm^{-1}). High-resolution mass spectra were obtained from the UC Irvine and UCLA Mass Spectrometry Facilities using TOF and Orbitrap mass analyzers, respectively.

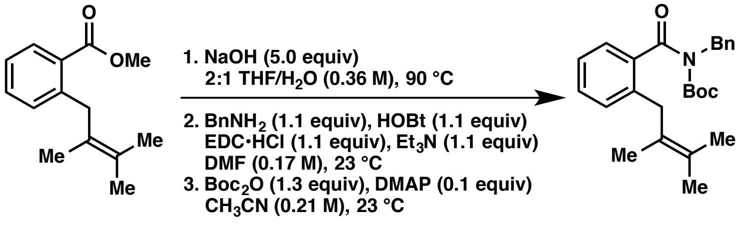
A. Syntheses of Heck Cyclization Substrates

A.1. Scalable Synthesis of Imide 5 for Reaction Discovery.



SI-1 ***SI-2*** **(31% yield)** ***SI-3***

Ester SI-3. Following a modification of the general procedure reported by Querolle and co-workers,[3] a flask containing a stir bar was charged with CuCN (1.03 g, 11.5 mmol, 1.0 equiv) and LiCl (971 mg, 22.9 mmol, 2.0 equiv) in the glovebox. The flask was removed from the glovebox, and the solids were suspended in THF (39 mL). The resulting mixture was stirred vigorously until a completely dissolved solution of CuCN•2LiCl was formed. In a separate flask containing a solution of methyl-2-iodobenzoate (**SI-1**) (3.02 g, 11.45 mmol, 1.0 equiv) in THF (115 mL) at –40 °C was added *i*-BuMgCl (8.6 mL of a 2.0 M solution in THF, 17.2 mmol, 1.5 equiv) dropwise over 1 min. After this mixture was stirred at –40 °C for 1 h, the solution of CuCN•2LiCl was added via cannula. The combined mixture stirred at –40 °C for an additional 15 min, at which point bromide **SI-2** (4.65 g, 28.6 mmol, 2.5 equiv) was added dropwise over 1 min. After stirring at –40 °C for an additional hour, the reaction was poured into 9:1 sat. aq. NH_4Cl:NH_4OH (150 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 100 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated under reduced pressure. The crude mixture was purified via flash chromatography (4:1 Benzene:Hexanes) to afford ester **SI-3** (767 mg, 31% yield) as a colorless oil. Ester **SI-3**: R_f 0.63 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.83 (dd, J = 7.7, 1.3, 1H), 7.40 (td, J = 7.5, 1.5, 1H), 7.23 (t, J = 7.5, 1H), 7.20 (d, J = 7.7, 1H), 3.89 (s, 3H), 3.78 (s, 2H) 1.76 (s, 3H), 1.71 (s, 3H), 1.57 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.7, 142.1, 131.9, 130.6, 130.3, 129.2, 127.1, 125.73, 125.66, 52.0, 37.7, 20.73, 20.69, 18.6; IR (film): 2916, 1720, 1433, 1262, 1246, 1121, 1076 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{19}O_7^+$, 219.13796; found 219.13784.



SI-3 (91% yield, 3 steps) ***5***

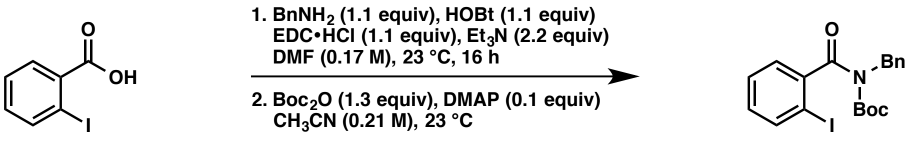
Imide 5. To a solution of ester **SI-3** (767 mg, 3.51 mmol, 1.0 equiv) in THF (6.6 mL) was added a solution of NaOH (703 mg, 17.6 mmol, 5.0 equiv) in H_2O (3.3 mL). The reaction was heated to 90 °C and stirred for 12 h. After cooling to room temperature, the reaction mixture was poured into deionized water (25 mL) and diluted with EtOAc (25 mL). The layers were separated and the aqueous layer was acidified to pH ~4 with 1 N HCl (15 mL) and extracted with EtOAc (3 x 25 mL). The organic layers were combined, washed with deionized water (300 mL), dried over $MgSO_4$, and concentrated under reduced pressure to afford the corresponding carboxylic acid, which was used in the subsequent step without further purification.

To a solution of the crude carboxylic acid, HOBt (591 mg, 3.86 mmol, 1.1 equiv from **SI-3**), and EDC•HCl (740 mg, 3.86 mmol, 1.1 equiv from **SI-3**) in DMF (21 mL) was added benzylamine (0.42 mL, 3.86 mmol, 1.1 equiv from **SI-3**) and triethylamine (0.5 mL, 3.86 mmol, 1.1 equiv from **SI-3**). After stirring for 7 h, the reaction mixture was poured into deionized water (100 mL) and diluted with EtOAc (50 mL). The layers were separated and the aqueous layer was extracted with EtOAc (2 x 50 mL). The organic layers were combined, washed with deionized water (100 mL), dried over $MgSO_4$, and concentrated under reduced pressure to afford the corresponding amide, which was used in the subsequent step without further purification.

To a solution of the crude amide in CH_3CN (17 mL) was added DMAP (43 mg, 0.351 mmol, 0.1 equiv from **SI-3**) and Boc_2O (996 mg, 4.56 mmol, 1.3 equiv from **SI-3**). After stirring for 7 h, the reaction mixture was concentrated under reduced pressure and purified by flash chromatography (99:1 Hexanes:EtOAc) to yield imide **5** (1.26 g, 91% yield, 3 steps) as a white solid. Imide **5**: mp: 65–67 °C; R_f 0.60 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43 (d, *J* = 7.9, 2H), 7.34 (d, *J* = 7.4, 2H), 7.31–7.25 (m, 2H), 7.16 (t, *J* = 7.4, 1H), 7.11 (d, *J* = 7.9, 2H), 5.03 (s, 2H), 3.43 (s, 2H), 1.75 (s, 3H), 1.70 (s, 3H), 1.58 (s, 3H), 1.11 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.6, 153.0, 138.7, 138.0, 137.7, 129.5, 128.6, 128.40, 128.36, 127.5, 127.2, 125.9, 125.44, 125.37, 83.4, 48.0, 36.8, 27.5, 20.76, 20.75, 18.8; IR (film): 2981, 2922, 1728, 1670, 1368, 1333, 1229, 1138 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{25}H_{32}NO_3^+$, 394.23767; found 394.23462.

A.2. Syntheses of Halo-Imide Reductive Coupling Partners

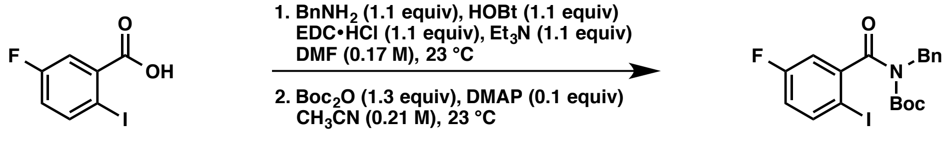
Representative Procedure (synthesis of imide SI-5 is used as an example).



SI-4 (93% yield, 2 steps) *SI-5*

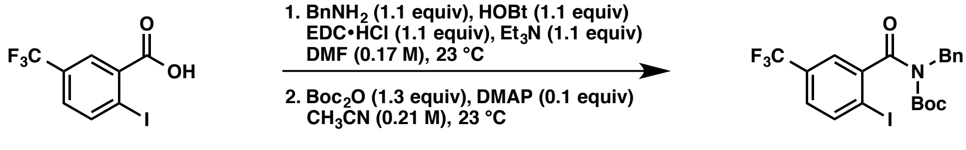
Iodo-imide SI-5. To a mixture of 2-iodo-benzoic acid (**SI-4**) (10.0 g, 40.4 mmol, 1.0 equiv), EDC•HCl (8.5 g, 44 mmol, 1.1 equiv), HOBt (6.0 g, 44 mmol, 1.1 equiv) and triethylamine (6.2 mL, 88 mmol, 2.2 equiv) in DMF (238 mL) was added benzylamine (5.0 mL, 44 mmol, 1.1 equiv). The resulting mixture was stirred for 16 h, and then diluted with deionized water (100 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 100 mL). The combined organic layers were washed with 0.1 N HCl (100 mL), sat. aq. $NaHCO_3$ (100 mL), and brine (100 mL), dried over Na_2SO_4, and filtered. Concentration under reduced pressure afforded the crude amide, which was used in the subsequent step without further purification.

To the vessel containing the crude amide was added DMAP (0.5 g, 4 mmol, 0.1 equiv from **SI-4**), followed by acetonitrile (192 mL). Boc_2O (11.5 g, 52.5 mmol, 1.3 equiv from **SI-4**) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir for 16 h. The reaction was concentrated under reduced pressure and the resulting crude residue was purified by flash chromatography (9:1 Hexanes:EtOAc) to yield iodo-imide **SI-5** (16.4 g, 93% yield, 2 steps) as a white solid. Iodo-imide **SI-5**: mp: 100–101 ºC; R_f 0.54 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.79 (d, *J* = 8.0, 1H), 7.47 (d, *J* = 8.0, 2H), 7.37–7.32 (m, 3H), 7.30–7.27 (m, 1H), 7.18–7.15 (m, 1H), 7.10–7.05 (m, 1H), 5.05 (s, 2H), 1.14 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.7, 152.1, 144.6, 139.2, 137.5, 130.3, 128.6, 128.5, 127.9, 127.6, 127.0, 91.7, 83.9, 48.0, 27.6; IR (film): 2979, 1731, 1668, 1228, 741 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{19}H_{21}INO_3^+$, 438.05606; found 438.05536.



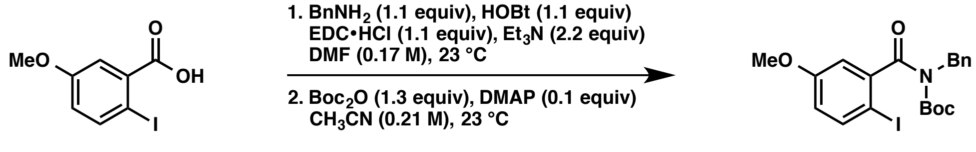
SI-6 (72% yield, 2 steps) *SI-7*

Iodo-imide SI-7. Following the representative procedure with 2-iodo-5-fluorobenzoic acid (**SI-6**) (2.0 g, 7.52 mmol), purification by flash chromatography (99:1 Pentane:Et$_2$O → 19:1 Pentane:Et$_2$O) afforded iodo-imide **SI-7** (2.46 g, 72% yield, 2 steps) as a white solid. Iodo-imide **SI-7**: mp: 61–63 °C; R$_f$ 0.60 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.72 (dd, *J* = 8.7, 5.2, 1H), 7.46 (d, *J* = 7.3, 2H), 7.34 (tt, *J* = 7.1, 1.4, 2H), 7.28 (tt, *J* = 7.3, 1.4, 1H), 6.93 (dd, *J* = 8.5, 3.1, 1H), 6.83 (dt, *J* = 8.5, 3.1, 1H), 5.04 (s, 2H), 1.20 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 170.3 (d, *J* = 2.2), 162.7 (d, *J* = 246), 151.8, 146.2 (d, *J* = 7.2), 140.6 (d, *J* = 7.7), 137.2, 128.63, 128.56, 127.7, 117.7 (d, *J* = 22), 114.7 (d, *J* = 24), 84.6 (d, *J* = 3.6), 84.3, 48.0, 27.6; ^{19}F NMR (376 MHz, CDCl$_3$): δ –113.7, (s, 1F); IR (film): 2981, 1736, 1671, 1369, 1350, 1331, 1232, 1149 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{19}H_{20}FINO_3^+$ 456.04664; found 456.04664.



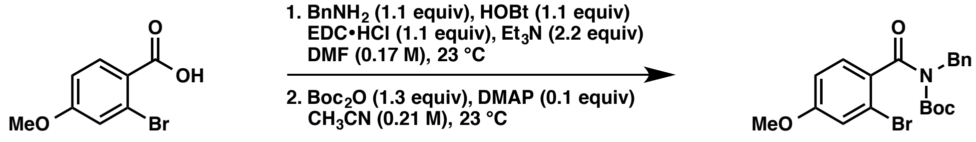
SI-8 (88% yield, 2 steps) *SI-9*

Iodo-imide SI-9. Following the representative procedure with 2-iodo-5-(trifluoromethyl)benzoic acid (**SI-8**) (1.98 g, 6.27 mmol), purification by flash chromatography (99:1 Pentane:Et$_2$O → 19:1 Pentane:Et$_2$O) afforded iodo-imide **SI-9** (2.79 g, 88% yield, 2 steps) as an off-white solid. Iodo-imide **SI-9**: mp: 60–62 °C; R$_f$ 0.70 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.93 (d, *J* = 8.3, 1H), 7.48 (d, *J* = 7.3, 2H), 7.41 (d, *J* = 2.0, 1H), 7.35 (tt, *J* = 7.6, 1.5, 2H), 7.33–7.28 (m, 2H), 5.07 (s, 2H), 1.15 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 170.4, 151.7, 145.5, 139.8, 137.1, 130.8 (q, *J* = 33), 128.7, 128.6, 127.8, 126.5 (q, *J* = 3.6), 123.68 (q, *J* = 273), 123.67 (q, *J* = 3.8), 95.8, 84.4, 48.0, 27.5; ^{19}F NMR (376 MHz, CDCl$_3$): δ –63.0, (s, 3F); IR (film): 2982, 1737, 1669, 1317, 1225, 1126, 1079 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{20}H_{20}F_3INO_3^+$ 506.04345; found 506.04387.



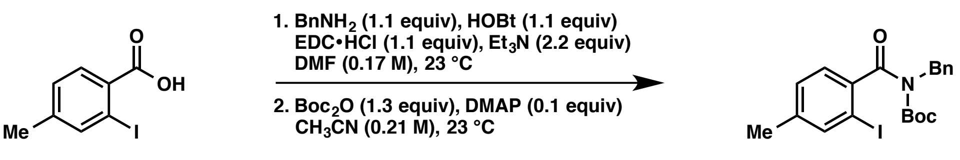
SI-10 (87% yield, 2 steps) *SI-11*

Iodo-imide SI-11. Following the representative procedure with 2-iodo-5-methoxybenzoic acid (**SI-10**) (1.0 g, 3.6 mmol), 2-iodo-imide **SI-11** (1.5 g, 87% yield, 2 steps) was obtained as a colorless oil. Iodo-imide **SI-11**: R*f* 0.24 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.62 (d, *J* = 8.8, 1H), 7.48–7.45 (m, 2H), 7.36–7.32 (m, 2H), 7.30–7.26 (m, 1H), 6.73 (d, *J* = 3.1, 1H), 6.66 (dd, *J* = 8.7, 3.1, 1H), 5.04 (s, 2H), 3.76, (s, 3H), 1.17 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.3, 159.6, 151.9, 145.1, 139.7, 137.3, 128.5, 128.4, 127.4, 116.8, 112.8, 83.7, 80.0, 55.5, 47.9, 27.4; IR (film): 2979, 1735, 1466, 1144, 848, 699 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{20}H_{23}INO_4^+$, 468.06663; found 468.06671.



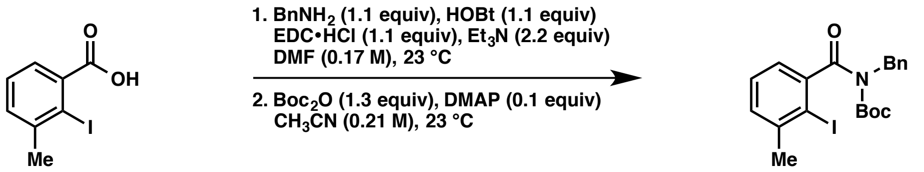
SI-12 (quantitative yield, 2 steps) *SI-13*

Bromo-imide SI-13. Following the representative procedure with 2-bromo-4-methoxybenzoic acid (**SI-12**) (0.46 g, 2.0 mmol), 2-bromo-imide **SI-13** (0.86 g, quantitative yield, 2 steps) was obtained as a yellow oil. Bromo-imide **SI-13**: R*f* 0.25 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.46–7.44 (m, 2H), 7.35–7.31 (m, 2H), 7.32–7.29 (m, 1H), 7.22 (d, *J* = 8.7, 1H), 7.07 (d, *J* = 2.5, 1H), 6.86 (dd, *J* = 8.7, 2.5, 1H), 5.02 (s, 2H), 3.81, (s, 3H), 1.19 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.5, 160.8, 152.5, 137.7, 132.7, 129.3, 128.5 (4 carbons), 127.5, 119.8, 118.11, 113.2, 83.5, 55.8, 48.2, 27.6; IR (film): 2979, 1731, 1599, 1227, 848, 558 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{20}H_{23}BrNO_4^+$, 420.08050; found 420.08082.



SI-14 **(92% yield, 2 steps)** ***SI-15***

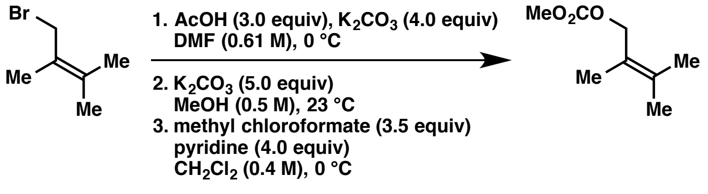
Iodo-imide SI-15. Following the representative procedure with 2-iodo-4-methylbenzoic acid (**SI-14**) (2.0 g, 7.6 mmol), iodo-imide **SI-15** (3.3 g, 92% yield, 2 steps) was obtained as a colorless oil. Iodo-imide **SI-15**: R*f* 0.34 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.65 (d, *J* = 7.8, 1H), 7.48–7.45 (m, 2H), 7.35–7.31 (m, 2H), 7.29–7.25 (m, 1H), 7.00 (d, *J* = 2.1, 1H), 6.89 (ddd, *J* = 8.1, 2.1, 0.8, 1H), 5.04 (s, 2H), 2.29 (s, 3H), 1.15 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 171.7, 152.1, 144.2, 138.8, 138.0, 137.4, 131.2, 128.5, 128.4, 127.8, 127.4, 87.4, 83.6, 47.9, 27.4, 20.8; IR (film): 2979, 1731, 1668, 1141, 848, 698 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{20}H_{23}INO_3^+$, 452.07171; found 452.07080.



SI-16 **(85% yield, 2 steps)** ***SI-17***

Iodo-imide SI-17. Following the representative procedure with 2-iodo-3-methylbenzoic acid (**SI-16**) (3.0 g, 12 mmol), iodo-imide **SI-17** (4.4 g, 85% yield, 2 steps) was obtained as a white solid. Iodo-imide **SI-17**: mp: 87–89 ºC; R*f* 0.62 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.48 (d, *J* = 7.8, 2H), 7.36–7.32 (m, 2H), 7.29–7.26 (m, 1H), 7.24–7.20 (m, 2H), 6.90 (dd, *J* = 6.7, 2.4, 1H), 5.07 (s, 2H), 2.46 (s, 3H), 1.11 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 171.9, 152.0, 145.7, 142.2, 137.4,129.5, 128.42, 128.36, 127.9, 127.4, 123.7, 98.5, 83.6, 47.7, 28.8, 27.4; IR (film): 2979, 1732, 1338, 1145, 849, 699 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{20}H_{23}INO_3^+$, 452.07171; found 452.07177.

A.3. Syntheses of Carbonate Reductive Coupling Partners

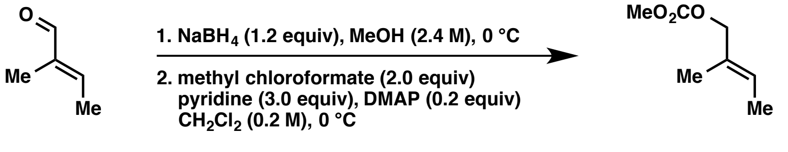


SI-2 ***SI-18***

Carbonate SI-18. To a suspension of K_2CO_3 (39.0 g, 0.282 mol, 4.0 equiv) in DMF (116 mL) was added AcOH (12 mL, 0.212 mol, 3.0 equiv). The mixture was cooled to 0 °C. After stirring for 5 min, bromide **SI-2**[4] (11.5 g, 0.0705 mol, 1.0 equiv) was added. After stirring vigorously at 0 °C for 2 h, the reaction mixture was poured into deionized water (300 mL) and diluted with Et_2O (150 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2 x 150 mL). The organic layers were combined, washed with deionized water (300 mL), dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature) to afford the corresponding acetate, which was used in the subsequent step without further purification.

To a solution of the crude acetate in MeOH (141 mL) was added K_2CO_3 (48.7 g, 0.353 mol, 4.0 equiv from **SI-2**). After stirring vigorously for 12 h, the reaction mixture was poured into deionized water (300 mL) and diluted with Et_2O (150 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2 x 150 mL). The organic layers were combined, washed with deionized water (300 mL), dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature) to afford the corresponding alcohol, which was used in the subsequent step without further purification.

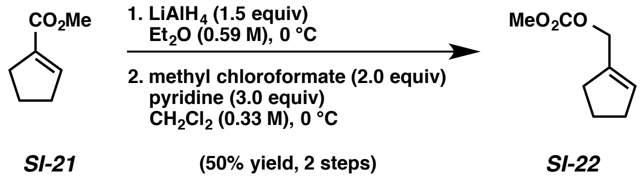
To a solution of the crude alcohol in CH_2Cl_2 (176 mL) was added pyridine (17.0 mL, 0.212 mol, 3.0 equiv from **SI-2**). The reaction was cooled to 0 °C. After stirring for 5 min, methyl chloroformate (11 mL, 0.141 mol, 2.0 equiv from **SI-2**) was added dropwise over 1 min. The reaction was stirred for 6 h, and allowed to warm to room temperature, at which point additional pyridine (8.50 mL, 0.141 mol, 1.0 equiv from **SI-2**) and methyl chloroformate (5.5 ml, 0.106 mol, 1.5 equiv from **SI-2**) was added. After stirring for an additional 12 h, the reaction mixture was poured into brine (200 mL) and diluted with Et_2O (150 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2 x 150 mL). The organic layers were combined, washed with 1 N HCl (300 mL), dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature). The crude mixture was purified via flash chromatography (99:1 Pentane:Et_2O → 15:1 Pentane:Et_2O) to afford carbonate **SI-18** (4.62 g, 41% yield, 3 steps) as a colorless oil. Carbonate **SI-18**: R_f 0.61 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.63 (s, 2H), 3.74 (s, 3H), 1.75–1.73 (m, 3H), 1.70–1.68 (m, 3H), 1.67 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.1, 132.8, 122.4, 69.3, 54.7, 20.9, 20.3, 16.7; IR (film): 2988, 2919, 1744, 1442, 1246 cm^{-1}; HRMS–ESI (*m/z*) $[M + Na]^+$ calcd for $C_8H_{14}NaO_3^+$, 181.0841; found 181.0843.



SI-19 **(80% yield, 2 steps)** ***SI-20***

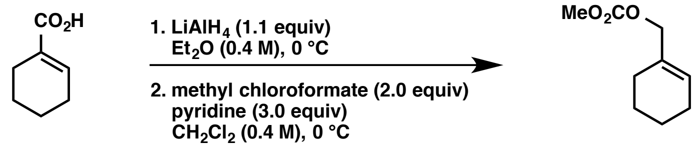
Carbonate SI-20. To a solution of tiglic aldehyde (**SI-19**) (3.0 g, 36 mmol, 1.0 equiv) in MeOH (15 mL) at 0 °C was added $NaBH_4$ (1.6 g, 43 mmol, 1.2 equiv) in 10 portions over 5 min at 0 °C. After 3 h of stirring at room temperature, the mixture was poured into deionized water (50 mL) and diluted with CH_2Cl_2 (50 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 50 mL). The organic layers were combined, washed with brine (50 mL), dried over $MgSO_4$, and concentrated under reduced pressure to afford the corresponding alcohol, which was used in the subsequent step without further purification.

To a solution of the crude alcohol in CH_2Cl_2 (180 mL) was added pyridine (2.57 mL, 31.9 mmol, 3.0 equiv from **SI-19**) and DMAP (0.86 g, 7.1 mmol, 0.2 equiv). The reaction was cooled to 0 °C. After stirring for 5 min, methyl chloroformate (0.87 mL, 11.3 mmol, 2.0 equiv from **SI-19**) was added dropwise over 20 min. The reaction was allowed to warm to room temperature. After stirring for 1 h, the reaction mixture was poured into brine (50 mL) and diluted with CH_2Cl_2 (50 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 50 mL). The organic layers were combined, washed with 1 N HCl (50 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude mixture was purified via flash chromatography (9:1 Pentane:Et_2O) to afford carbonate **SI-20** (4.6 g, 80% yield, 2 steps) as a colorless oil. Carbonate **SI-20**: R_f 0.61 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.59 (q, *J* = 7.0, 1H), 4.51 (s, 2H), 3.77 (s, 3H), 1.67 (s, 3H), 1.63 (d, *J* = 7.0, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.0, 130.4, 125.2, 74.0, 54.8, 13.7, 13.4; IR (film): 2957, 1745, 1442, 1250, 935, 792 cm^{-1}; HRMS–ESI (m/z) [M + Na]$^+$ calcd for $C_8H_{14}O_3Na$ 181.0843; found 181.0841.



Carbonate SI-22. To a solution of methyl ester **SI-21** (1.26 g, 10.0 mmol, 1.0 equiv) in Et_2O (17 mL) at 0 °C was added $LiAlH_4$ (570 mg, 15.0 mmol, 1.5 equiv) at 0 °C. After stirring for 4 h, deionized water (3 mL) was added dropwise over 5 min at 0 °C. The resulting heterogeneous mixture was filtered through a plug of celite® (50 mL of Et_2O eluent) and the filtrate was diluted with deionized water (50 mL). The layers were separated and the aqueous layer was extracted with Et_2O (3 x 50 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature) to afford the corresponding alcohol, which was used in the subsequent step without further purification.

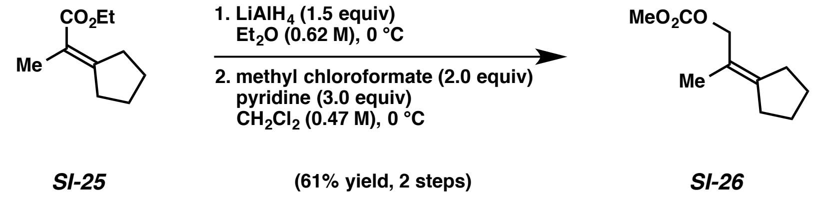
To a solution of the crude alcohol in CH_2Cl_2 (30 mL) was added pyridine (2.50 mL, 30.0 mmol, 3.0 equiv from **SI-21**). The reaction was cooled to 0 °C. After stirring for 5 min, methyl chloroformate (1.55 mL, 20.0 mmol, 2.0 equiv from **SI-21**) was added dropwise over 1 min. The reaction was allowed to warm to room temperature. After stirring for 15 h, the reaction mixture was poured into brine (150 mL) and diluted with CH_2Cl_2 (50 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 x 50 mL). The organic layers were combined, washed with 1 N HCl (50 mL), dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature). The crude mixture was purified via flash chromatography (98:2 Hexanes:Et_2O) to afford carbonate **SI-22** (792 mg, 50% yield, 2 steps) as a colorless oil. Carbonate **SI-22**: R_f 0.48 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.72 (s, 1H), 4.69 (s, 2H), 3.79 (s, 3H), 2.38–2.31 (m, 4H), 1.92 (quint, J = 7.7, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.0, 138.6, 129.6, 66.9, 54.9, 32.9, 32.6, 23.4; IR (film): 2960, 2918, 2848, 1750, 1447, 1263, 949 cm^{-1}; HRMS–ESI (*m/z*) [M + Na]$^+$ calcd for $C_8H_{12}O_3Na$, 179.0684; found 179.0677.



SI-23 ***SI-24***

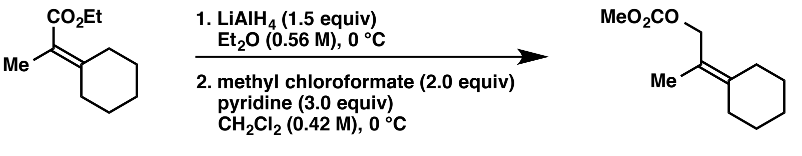
Carbonate SI-24. To a solution of carboxylic acid **SI-23** (2.00 g, 16.5 mmol, 1.0 equiv) in Et_2O (40 mL) at 0 °C was added $LiAlH_4$ (18.2 mL of a 1.0 M solution in Et_2O, 18.2 mmol, 1.1 equiv) dropwise over 5 min at 0 °C. After stirring for 15 min, deionized water (5 mL) was added dropwise at 0 °C. The resulting heterogeneous mixture was filtered through a plug of celite® (25 mL of Et_2O eluent) and the filtrate was diluted with deionized water (50 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2 x 50 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature) to afford the corresponding alcohol, which was used in the subsequent step without further purification.

To a solution of the crude alcohol in CH_2Cl_2 (40 mL) was added pyridine (2.55 mL, 49.5 mmol, 3.0 equiv from **SI-23**). The reaction was cooled to 0 °C. After stirring for 5 min, methyl chloroformate (2.6 mL, 33.0 mmol, 2.0 equiv from **SI-23**) was added dropwise over 1 min. The reaction was allowed to warm to room temperature. After stirring for 4 h, the reaction mixture was poured into brine (50 mL) and diluted with CH_2Cl_2 (50 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 50 mL). The organic layers were combined, washed with 1 N HCl (50 mL), dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature). The crude mixture was purified via flash chromatography (99:1 Pentane:Et_2O → 49:1 Pentane:Et_2O) to afford carbonate **SI-24** (2.00 g, 71% yield, 2 steps) as a colorless oil. Carbonate **SI-24**: R_f 0.86 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.78 (m, 1H), 4.49 (s, 2H), 3.78 (s, 3H), 2.02 (m, 4H), 1.64 (m, 2H), 1.57 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.0, 132.5, 127.5, 72.7, 54.8, 25.9, 25.1, 22.4, 22.2; IR (film): 2930, 1744, 1441, 1250 cm^{-1}; HRMS–ESI (*m/z*) $[M + Na]^+$ calcd for $C_9H_{14}NaO_3^+$, 193.0841; found 193.0839.



Carbonate SI-26. To a solution of known ester **SI-25**[5] (3.15 g, 18.7 mmol, 1.0 equiv) in Et_2O (30 mL) at 0 °C was added $LiAlH_4$ (1.07 g, 28.1 mmol, 1.5 equiv) at 0 °C. After stirring for 4 h, deionized water (3 mL) was added dropwise over 5 min at 0 °C. The resulting heterogeneous mixture was filtered through a plug of celite® (25 mL of Et_2O eluent) and the filtrate was diluted with deionized water (25 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2 x 50 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature) to afford the corresponding alcohol, which was used in the subsequent step without further purification.

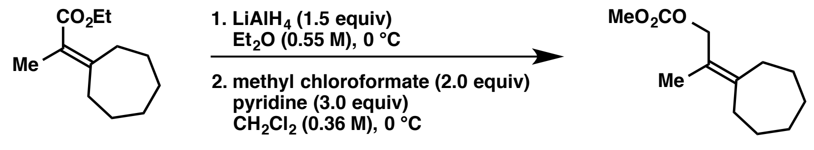
To a solution of the crude alcohol in CH_2Cl_2 (40 mL) was added pyridine (4.65 mL, 56.1 mmol, 3.0 equiv from **SI-25**). The reaction was cooled to 0 °C. After stirring for 5 min, methyl chloroformate (2.9 mL, 37.4 mmol, 2.0 equiv from **SI-25**) was added dropwise over 1 min. The reaction was allowed to warm to room temperature. After stirring for 15 h, the reaction mixture was poured into brine (100 mL) and diluted with Et_2O (50 mL). The layers were separated and the aqueous layer was extracted with Et_2O (3 x 50 mL). The organic layers were combined, washed with 1 N HCl (25 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude mixture was purified via flash chromatography (95:5 Hexanes:Et_2O) to afford carbonate **SI-26** (2.09 g, 61% yield, 2 steps) as a colorless oil. Carbonate **SI-26**: R_f 0.45 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.63 (s, 2H), 3.78 (s, 3H), 2.33 (s, 2H), 2.22 (s, 2H), 1.70–1.63 (m, 7H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.2, 145.0, 119.6, 70.6, 54.8, 31.3, 30.4, 27.0, 26.4, 17.2; IR (film): 2956, 2867, 1748, 1442, 1373, 1256, 942 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{10}H_{17}O_3^+$, 185.11722; found 185.11700.



SI-27 **(54% yield, 2 steps)** ***SI-28***

Carbonate SI-28. To a solution of known ester **SI-27**[3] (3.08 g, 16.9 mmol, 1.0 equiv) in Et_2O (30 mL) at 0 °C was added $LiAlH_4$ (965 mg, 25.4 mmol, 1.5 equiv). After stirring for 1 h, deionized water (3 mL) was added dropwise over 5 min at 0 °C. The resulting heterogeneous mixture was filtered through a plug of celite® (25 mL of Et_2O eluent) and the filtrate was diluted with deionized water (25 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2 x 50 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature) to afford the corresponding alcohol, which was used in the subsequent step without further purification.

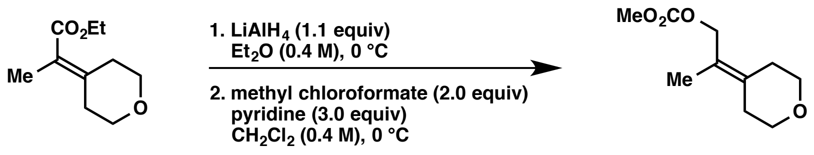
To a solution of the crude alcohol in CH_2Cl_2 (40 mL) was added pyridine (4.21 mL, 50.7 mmol, 3.0 equiv from **SI-27**). The reaction was cooled to 0 °C. After stirring for 5 min, methyl chloroformate (2.6 mL, 33.8 mmol, 2.0 equiv from **SI-27**) was added dropwise over 1 min. The reaction was allowed to warm to room temperature. After stirring for 15 h, the reaction mixture was poured into brine (100 mL) and diluted with Et_2O (50 mL). The layers were separated and the aqueous layer was extracted with Et_2O (3 x 50 mL). The organic layers were combined, washed with 1 N HCl (25 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude mixture was purified via flash chromatography (95:5 Hexanes:Et_2O) to afford carbonate **SI-28** (1.83 g, 54% yield, 2 steps) as a colorless oil. Carbonate **SI-28**: R_f 0.45 (9:1 Hexanes:EtOAc); ^1H NMR (500 MHz, $CDCl_3$): δ 4.68 (s, 2H), 3.77 (s, 3H), 2.28–2.22 (m, 2H), 2.21–2.15 (m, 2H), 1.73 (s, 3H), 1.60–1.48 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.2, 141.4, 119.1, 69.0, 54.8, 31.0, 30.7, 28.4, 27.9, 26.8, 16.5; IR (film): 2925, 2854, 1744, 1443, 1373, 1247, 937 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{11}H_{19}O_3^+$, 199.13287; found 199.13247.



SI-29 **(64% yield, 2 steps)** ***SI-30***

Carbonate SI-30. To a solution of known ester **SI-29**[6] (2.15 g, 10.9 mmol, 1.0 equiv) in Et_2O (20 mL) at 0 °C was added $LiAlH_4$ (626 mg, 16.4 mmol, 1.5 equiv). After stirring for 1 h, deionized water (3 mL) was added dropwise over 5 min at 0 °C. The resulting heterogeneous mixture was filtered through a plug of celite® (25 mL of Et_2O eluent) and the filtrate was diluted with deionized water (25 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2 x 50 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature) to afford the corresponding alcohol, which was used in the subsequent step without further purification.

To a solution of the crude alcohol in CH_2Cl_2 (30 mL) was added pyridine (2.80 mL, 32.9 mmol, 3.0 equiv from **SI-29**). The reaction was cooled to 0 °C. After stirring for 5 min, methyl chloroformate (1.7 mL, 21.9 mmol, 2.0 equiv from **SI-29**) was added dropwise over 1 min. The reaction was allowed to warm to room temperature. After stirring for 15 h, the reaction mixture was poured into brine (100 mL) and diluted with Et_2O (50 mL). The layers were separated and the aqueous layer was extracted with Et_2O (3 x 50 mL). The organic layers were combined, washed with 1 N HCl (25 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude mixture was purified via flash chromatography (95:5 Hexanes:Et_2O) to afford carbonate **SI-30** (1.49 g, 64% yield, 2 steps) as a colorless oil. Carbonate **SI-30**: R_f 0.52 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.67 (s, 2H), 3.77 (s, 3H), 2.33 (t, J = 6.0, 2H), 2.26 (t, J = 6.0, 2H), 1.71 (s, 3H), 1.60–1.53 (m, 4H), 1.51–1.44 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.1, 142.2, 122.4, 69.1, 54.6, 32.3, 31.2, 28.9, 28.6, 28.2, 26.8, 16.4; IR (film): 2922, 2854, 1748, 1443, 1374, 1256, 936 cm^{-1}; HRMS–ESI (*m/z*) $[M + Na]^+$ calcd for $C_{12}H_{20}O_3Na^+$, 235.1310; found 235.1301.

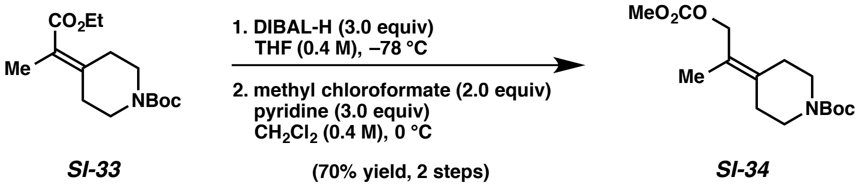


SI-31 **(71% yield, 2 steps)** ***SI-32***

Carbonate SI-32. To a solution of ester **SI-31**[7] (1.47 g, 8.0 mmol, 1.0 equiv) in Et_2O (20 mL) at 0 °C was added $LiAlH_4$ (8.8 mL of a 1.0 M solution in Et_2O, 8.8 mmol, 1.1 equiv) dropwise over 5 min. After stirring for 1 h, deionized water (1 mL) was added dropwise over 5 min at 0 °C. The resulting heterogeneous mixture was filtered through a plug of celite® (25 mL of Et_2O eluent) and the filtrate was diluted with deionized water (25 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2 x 50 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated under reduced pressure (~100 mbar, at room temperature) to afford the corresponding alcohol, which was used in the subsequent step without further purification.

To a solution of the crude alcohol in CH_2Cl_2 (20 mL) was added pyridine (1.93 mL, 24.0 mmol, 3.0 equiv from **SI-31**). The reaction was cooled to 0 °C. After stirring for 5 min, methyl chloroformate (1.24 mL, 16.0 mmol, 2.0 equiv from **SI-31**) was added dropwise over 1 min. The reaction was allowed to warm to room temperature. After stirring for 12 h, the reaction mixture was poured into brine (25 mL) and diluted with Et_2O (25 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2 x 50 mL). The organic layers were combined, washed with 1 N HCl (25 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude mixture was purified via flash chromatography (9:1 Hexanes:EtOAc → 5:1 Hexanes:EtOAc) to afford carbonate **SI-32** (1.14 g, 71% yield, 2 steps) as a colorless oil. Carbonate **SI-32**: R_f 0.73 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.66 (s, 2H), 3.78 (s, 3H), 3.67 (app ddd, *J* = 13.2, 7.7, 5.5, 4H), 2.39 (t, *J* = 5.5, 2H), 2.32 (t, *J* = 5.5, 2H) 1.75 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.1, 135.6, 121.5, 68.9–68.2 (2 carbons), 54.9, 31.3 & 31.1 (1 carbon), 16.3; IR (film): 2958, 2847, 1743, 1442, 1251 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{10}H_{17}O_7^+$, 201.11214; found 201.11059.

*Note: The data for carbonate **SI-32** represents empirically observed chemical shifts from the ^{13}C NMR spectrum, presumably due to the oxygen-containing heterocycle.*



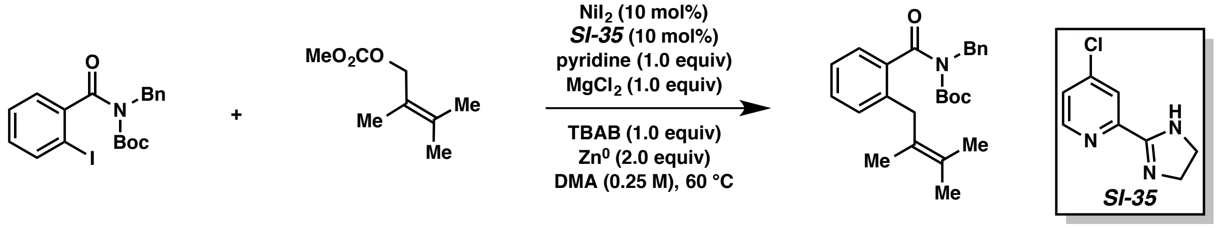
Carbonate SI-34. To a solution of ester **SI-33**[8] (1.60 g, 5.65 mmol, 1.0 equiv) in THF (14 mL) at 0 °C was added DIBAL-H (11.3 mL of a 1.0 M solution in THF, 11.3 mmol, 2.0 equiv) dropwise over 5 min. The reaction was stirred for 3 h, and allowed to warm to room temperature, at which point additional DIBAL-H (5.65 mL of a 1.0 M solution in THF, 5.65 mmol, 1.0 equiv) was added. After stirring for an additional hour, the reaction mixture was poured into water (50 mL) and diluted with Et_2O (50 mL). The resulting heterogeneous mixture was filtered through a plug of celite® (100 mL of Et_2O eluent). The layers of the resulting filtrate were separated and the aqueous layer was extracted with Et_2O (1 x 50 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated under reduced pressure to afford the corresponding alcohol, which was used in the subsequent step without further purification.

To a solution of the crude alcohol in CH_2Cl_2 (14 mL) was added pyridine (2.57 mL, 31.9 mmol, 3.0 equiv from **SI-33**). The reaction was cooled to 0 °C. After stirring for 5 min, methyl chloroformate (0.87 mL, 11.3 mmol, 2.0 equiv from **SI-33**) was added dropwise over 1 min. The reaction was allowed to warm to room temperature. After stirring for 1 h, the reaction mixture was poured into brine (50 mL) and diluted with CH_2Cl_2 (50 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 50 mL). The organic layers were combined, washed with 1 N HCl (50 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude mixture was purified via flash chromatography (9:1 Hexanes:EtOAc) to afford carbonate **SI-34** (1.18 g, 70% yield, 2 steps) as a colorless oil. Carbonate **SI-34**: R_f 0.78 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.66 (s, 2H), 3.77 (s, 3H), 3.40 (app q, J = 6.8, 4H), 2.35 (t, J = 5.6, 2H), 2.28 (t, J = 5.6, 2H) 1.75 (s, 3H), 1.45 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.0, 154.9, 136.2, 122.3, 79.6, 68.3, 54.9, 29.9, 29.5, 28.6, 16.5; IR (film): 2974, 1745, 1691, 1441, 1420, 1365, 1250, 1228, 1164 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{26}NO_5^+$, 300.18055; found 300.17829.

A.4. Reductive Cross Coupling of Imides and Carbonates

Representative Procedure (synthesis of imide 5 is used as an example).

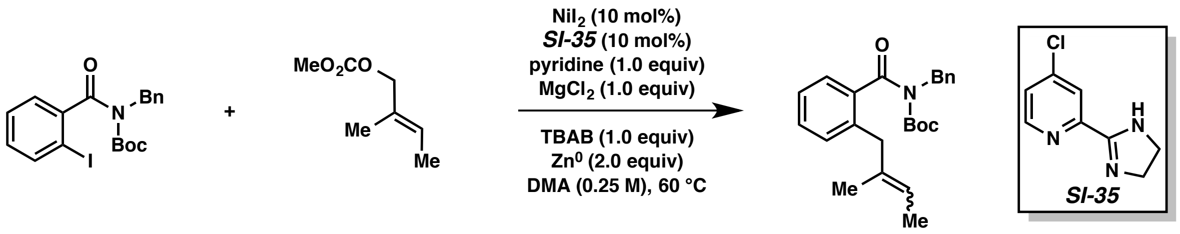
Reductive couplings were performed using a modification of the procedure reported by Gong and co-workers for the coupling of aryl bromides with substituted allylic acetates.[1]



SI-5 ***SI-18*** **(47% yield)** ***5***

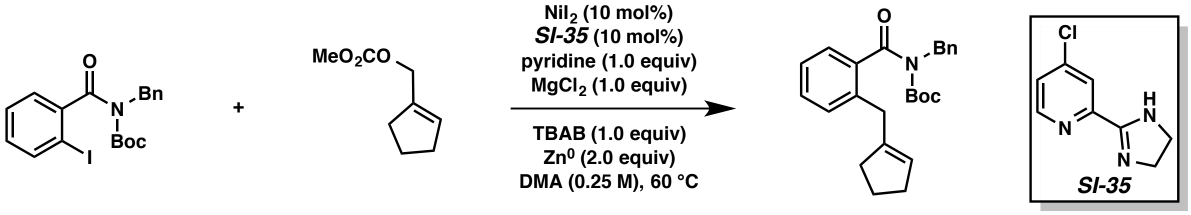
Imide 5. A scintillation vial containing imide **SI-5** (219 mg, 0.50 mmol, 1.0 equiv), ligand **SI-35** (9.1 mg, 0.050 mmol, 10 mol%), and a magnetic stir bar was sequentially charged with NiI_2 (15.6 mg, 0.050 mmol, 10 mol%), $MgCl_2$ (47.6 mg, 0.50 mmol, 1.0 equiv), TBAB (161 mg, 0.50 mmol, 1.0 equiv) and Zn^0 (65.4 mg, 1.0 mmol, 2.0 equiv) in the glovebox. The vial was removed from the glovebox, at which point DMA (2.0 mL), pyridine (40 µL, 0.5 mmol, 1.0 equiv), and carbonate **SI-18** (158 mg, 1.0 mmol, 2.0 equiv) were added. The vial was quickly sealed with a teflon-lined screw cap, and stirred at 60 °C for 14 h. After cooling to room temperature, the mixture was passed through a column of silica gel and flushed (5:2 Hexanes:EtOAc) until TLC indicated the desired product had eluted. The volatiles were removed under reduced pressure and the crude mixture was further purified by flash chromatography (99:1 Hexanes:EtOAc) to yield imide **5** (92 mg, 47% yield) as a white solid. Spectral data matched what is reported in Section A.1.

Any modifications of the conditions shown in the representative procedures above are specified in the following schemes.



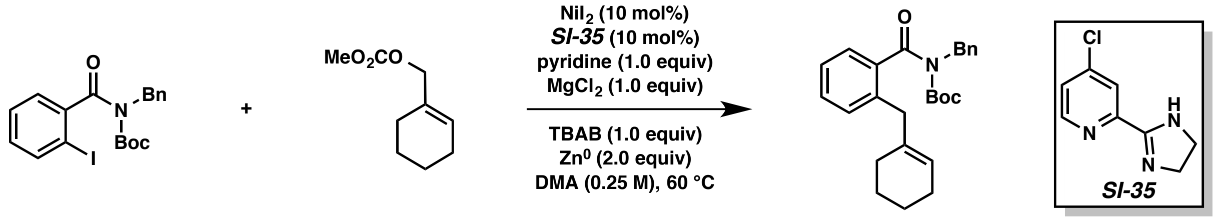
SI-5 ***SI-20*** **(41% yield, ratio E:Z (7:1))** ***SI-36***

Imide SI-36. Following the representative procedure with iodo-imide **SI-5** (218 mg, 0.5 mmol, 1.0 equiv), purification by flash chromatography (9:1 Hexanes:EtOAc) yielded **SI-36** as an inseparable mixture of olefin isomers (79 mg, 41% yield, 7:1 isomer ratio E:Z) and as a colorless oil. Configurational isomers of imide **SI-36** were analyzed as a mixture: R*f* 0.62 (4:1 Hexanes:EtOAc); Major (*E*)-isomer **SI-36** ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45–7.41 (m, 2H), 7.36–7.32 (m, 2H), 7.31–7.26 (m, 1H), 7.21–7.12 (m, 2H), 5.24 (q, *J* = 6.8, 1H), 5.01 (s, 2H), 3.34 (s, 2H), 1.57 (d, *J* = 6.8, 3H), 1.53 (s, 3H), 1.10 (s, 9H); Minor (*Z*)-isomer **SI-36** ^{1}H NMR (500 MHz, $CDCl_3$): (20 of 29 signals observed) δ 5.45 (q, *J* = 6.8, 1H), 5.07–5.03 (m, 1H), 4.90–4.85 (m, 1H), 3.41 (s, 2H), 1.63 (d, *J* = 6.8, 3H), 1.61 (s, 3H), 1.11 (s, 9H); Major (*E*)-isomer **SI-36** ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.5, 153.0, 138.6, 138.1, 137.5, 134.2, 130.0, 129.4, 127.5, 126.4, 125.8, 121.7, 83.4, 48.1, 42.8, 27.6, 16.0, 13.7; Minor (*Z*)-isomer **SI-36** ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.6, 153.1, 138.7, 138.0, 137.0, 133.8, 129.7, 129.6, 127.6, 126.1, 125.7, 121.9, 83.5, 48.1, 34.2, 27.7, 23.8, 13.8; IR (film): 2979, 1726, 1669, 1228, 1137, 739 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{24}H_{29}NO_3$ 380.22202; found 380.22186.



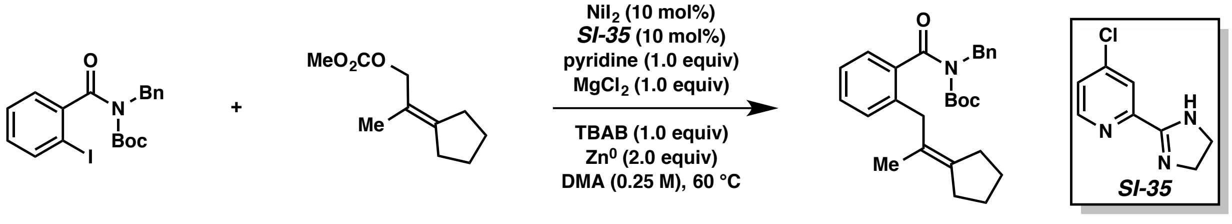
SI-5 ***SI-22*** **(21% yield)** ***SI-37***

Imide SI-37. Following the representative procedure with iodo-imide **SI-5** (874 mg, 2.0 mmol, 1.0 equiv), imide **SI-37** (165 mg, 21% yield) was obtained as a colorless oil. Imide **SI-37**: R*f* 0.48 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43 (d, *J* = 7.4, 2H), 7.35–7.24 (m, 4H), 7.22–7.13 (m, 3H), 5.32 (s, 1H), 5.01 (s, 2H), 3.45 (s, 2H), 2.32–2.24 (m, 2H), 2.20–2.14 (m, 2H), 1.83 (quint, *J* = 7.7, 2H), 1.10 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.4, 152.9, 142.6, 138.2, 138.0, 137.3, 130.1, 129.4, 128.5, 128.4, 127.5, 126.7, 126.4, 125.7, 83.3, 48.1, 35.1, 34.9, 32.6, 27.5, 23.6; IR (film): 2933, 1728, 1671, 1369, 1334, 1229, 1139 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{25}H_{30}NO_3^+$, 392.22202; found 392.21992.



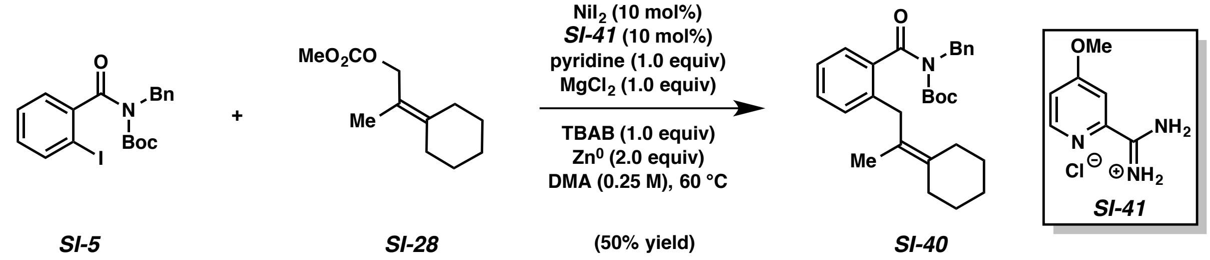
SI-5 **SI-24** (41% yield) **SI-38**

Imide SI-38. Following the representative procedure with iodo-imide **SI-5** (1.09 g, 2.5 mmol, 1.0 equiv), imide **SI-38** (417 mg, 41% yield) was obtained as a colorless oil. Imide **SI-38**: R$_f$ 0.66 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.43 (d, *J* = 7.1, 2H), 7.33 (t, *J* = 7.6, 2H), 7.31–7.24 (m, 2H), 7.23–7.13 (m, 3H), 5.43 (m, 1H), 5.01 (s, 2H), 3.31 (s, 2H), 1.98 (s, 2H), 1.83 (s, 2H), 1.55 (m, 4H), 1.11 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 172.5, 152.9, 138.4, 138.0, 137.4, 136.2, 129.9, 129.4, 128.6, 128.4, 127.5, 126.4, 125.7, 124.2, 83.3, 48.1, 41.4, 28.3, 27.5, 25.5, 23.0, 22.4; IR (film): 2929, 1728, 1671, 1334, 1230, 1140 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{26}H_{32}NO_3^+$, 406.23767; found 406.23493.

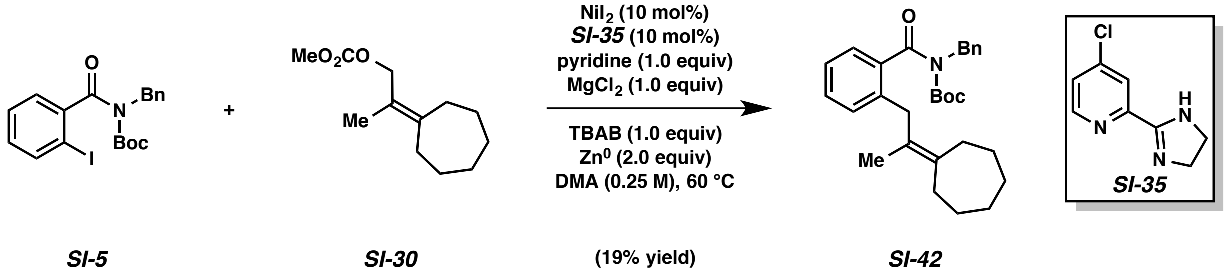


SI-5 **SI-26** (50% yield) **SI-39**

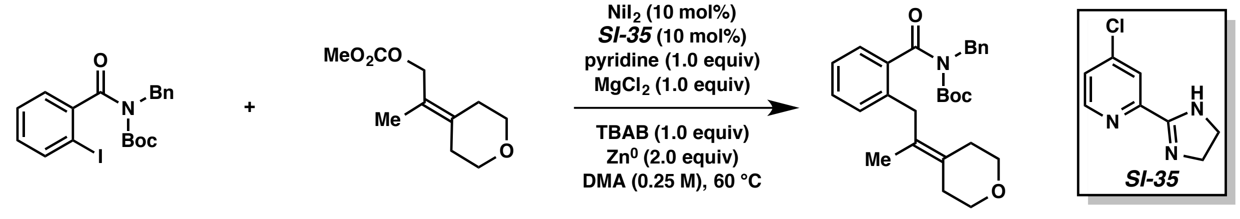
Imide SI-39. Following the representative procedure with iodo-imide **SI-5** (874 mg, 2.0 mmol), imide **SI-39** (418 mg, 50% yield) was obtained as a colorless oil. Imide **SI-39**: R$_f$ 0.45 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.44 (d, *J* = 7.7, 2H), 7.33 (t, *J* = 7.2, 2H), 7.31–7.25 (m, 2H), 7.18–7.10 (m, 3H), 5.03 (s, 2H), 3.39 (s, 2H), 2.25 (m, 4H), 1.67 (m, 4H), 1.55 (m, 3H), 1.10 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 172.6, 153.0, 139.7, 138.7, 138.0, 137.5, 129.5, 128.59, 128.57, 128.4, 127.5, 126.1, 125.5, 122.9, 83.4, 48.0, 38.1, 31.0, 30.9, 27.5, 27.2, 27.0, 19.3 ; IR (film): 2938, 1729, 1672, 1335, 1229, 1139 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{27}H_{34}NO_3^+$, 420.25332; found 420.25300.



Imide SI-40. Following the representative procedure with iodo-imide **SI-5** (0.44 g, 1.0 mmol, 1.0 equiv), imide **SI-40** (0.22 g, 50% yield) was obtained as a colorless oil. Imide **SI-40**: R_f 0.45 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.45–7.42 (m, 2H), 7.46–7.43 (m, 2H), 7.36–7.32 (m, 2H), 7.18–7.10 (m, 3H), 5.04 (s, 2H), 3.44 (s, 2H), 2.25 (t, *J* = 5.8, 2H), 2.17 (t, *J* = 5.8, 2H), 1.60 (s, 3H), 1.59–1.54 (m, 4H), 1.52–1.46 (m, 2H), 1.10 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 172.6, 153.0, 138.7, 138.0, 137.8, 135.9, 129.5, 128.6, 128.4, 128.3, 127.5, 125.9, 125.4, 121.8, 83.4, 48.0, 36.2, 30.84, 30.78, 28.5, 28.3, 27.5, 27.1, 18.4; IR (film): 2922, 1728, 1368, 1137, 740, 672 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{28}H_{36}NO_3^+$, 434.26897; found 434.26866.



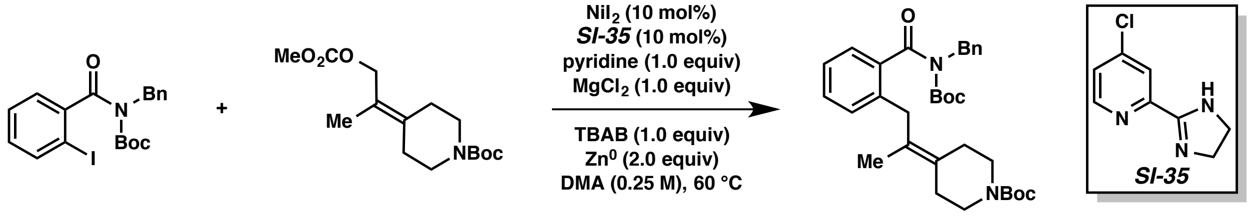
Imide SI-42. Following the representative procedure with iodo-imide **SI-5** (874 mg, 2.0 mmol, 1.0 equiv), purification by flash chromatography (199:1 Hexanes:EtOAc) yielded imide **SI-42** (172 mg, 19% yield) was obtained as a colorless oil. Imide **SI-42**: R_f 0.50 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.44 (d, *J* = 7.8, 2H), 7.33 (t, *J* = 7.2, 2H), 7.31–7.25 (m, 2H), 7.18–7.10 (m, 3H), 5.04 (s, 2H), 3.42 (s, 2H), 2.31 (t, *J* = 5.9, 2H), 2.25 (t, *J* = 5.9, 2H), 1.65–1.59 (m, 2H), 1.58 (s, 3H), 1.57–1.47 (m, 6H), 1.11 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 172.6, 153.0, 138.7, 138.0, 137.7, 137.0, 129.5, 128.6, 128.4, 128.2, 127.5, 126.0, 125.4, 125.3, 83.4, 48.0, 36.4, 32.0, 31.8, 29.4, 29.0, 28.1, 27.8, 27.5, 18.6; IR (film): 2921, 1730, 1673, 1335, 1229, 1138 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{29}H_{38}NO_3^+$, 448.28462; found 448.28241.



SI-5 ***SI-32*** **(46% yield)** ***SI-43***

Imide SI-43. After following the representative procedure with iodo-imide **SI-5** (1.16 g, 2.66 mmol, 1.0 equiv), the crude reaction mixture was vigorously stirred with 1:1 1 N NaOH/THF (26 mL) solution for 4 h to remove residual carbonate **SI-32**. Purification by flash chromatography (49:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) afforded imide **SI-43** (536 mg, 46% yield) as a colorless oil. Imide **SI-43**: R_f 0.49 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43 (d, *J* = 7.7, 2H), 7.34 (t, *J* = 7.7, 2H), 7.29 (dt, *J* = 7.7, 1.3, 2H), 7.17 (t, *J* = 7.7, 1H), 7.13 (t, *J* = 8.0, 2H), 5.03 (s, 2H), 3.71 (d, *J* = 5.5, 2H), 3.64 (d, *J* = 5.5, 2H), 3.45 (s, 2H), 2.38 (d, *J* = 5.5, 2H), 2.30 (d, *J* = 5.5, 2H), 1.63 (s, 3H), 1.11 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.5, 153.0, 138.6, 137.9, 137.3, 130.3, 129.6, 128.6, 128.4, 128.2, 127.6, 126.0, 125.6, 124.5, 83.5, 69.2 & 69.1 (1 carbon), 48.1, 35.9, 31.3 & 31.2 (1 carbon), 27.6, 18.3; IR (film): 2962, 2845, 1728, 1669, 1369, 1333, 1228, 1137, 1101 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{27}H_{34}NO_4^+$, 436.24824; found 436.24824.

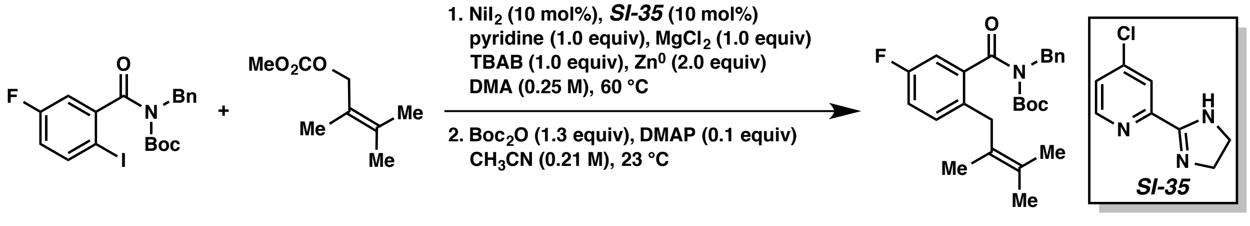
*Note: The data for imide **SI-43** represents empirically observed chemical shifts from the ^{13}C NMR spectrum, presumably due to the oxygen-containing heterocycle.*



SI-5 *SI-34* (47% yield) *SI-44*

Imide SI-44. After following the representative procedure with iodo-imide **SI-5** (1.08 g, 2.47 mmol, 1.0 equiv), the crude reaction mixture was vigorously stirred with 2:1 1 N NaOH/THF (30 mL) solution for 22 h to remove residual carbonate **SI-34**. Purification by flash chromatography (199:1 Benzene:EtOAc → 24:1 Benzene:EtOAc) afforded imide **SI-44** (622 mg, 47% yield) as a colorless oil. Imide **SI-44**: R_f 0.49 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$):): δ 7.44 (d, J = 7.9, 2H), 7.34 (tt, J = 7.6, 1.4, 2H), 7.32–7.27 (m, 2H), 7.16 (td, J = 7.6, 1.0, 1H), 7.11 (td, J = 7.4, 1.3, 2H), 5.03 (s, 2H), 3.45 (s, 4H), 3.37 (s, 2H), 2.34 (t, J = 5.6, 2H), 2.27 (s, 2H), 1.63 (s, 3H), 1.47 (s, 9H) 1.11 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.5, 155.1, 153.0, 138.6, 137.9, 137.2, 130.9, 129.6, 128.6, 128.4, 128.2, 127.6, 126.0, 125.7, 125.3, 83.5, 79.5, 48.1, 45.0 & 44.4 (1 carbon), 36.2, 29.8 & 29.7 (1 carbon), 28.6, 27.6, 18.5; IR (film): 2976, 1730, 1692, 1672, 1367, 1231, 1164, 1141 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{32}H_{43}N_2O_5^+$, 535.31665; found 535.31392.

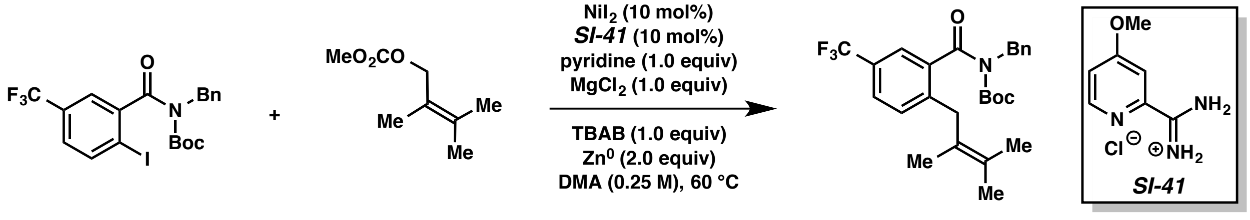
*Note: The data for imide **SI-44** represents empirically observed chemical shifts from the ^{13}C NMR spectrum, presumably due to the nitrogen-containing heterocycle.*



SI-7 *SI-18* (17% yield, 2 steps) *SI-45*

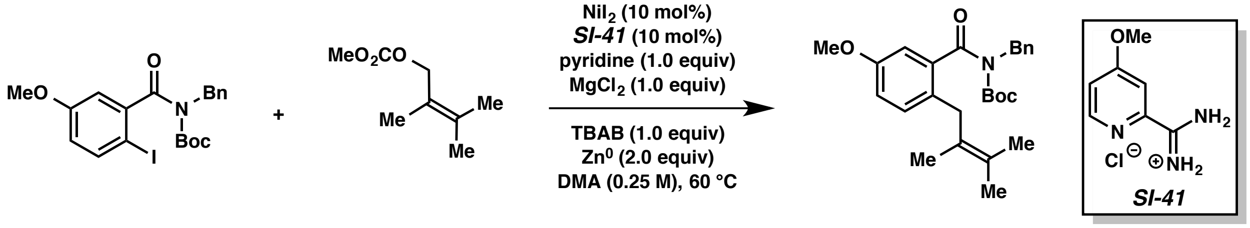
Imide SI-45. Following the representative procedure with iodo-imide **SI-7** (1.82g, 4.0 mmol) led to appreciable amounts of des-Boc coupled product. As such, the mixture was re-subjected to the general conditions used to install a boc group described in the synthesis of **5** (Section A.1).

Purification by flash chromatography (99:1 Pentane:Et$_2$O → 49:1 Pentane:Et$_2$O) afforded imide **SI-45** (275 mg, 17% yield, 2 steps) as an off-white oil. Imide **SI-45**: R$_f$ 0.64 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.42 (d, *J* = 7.8, 2H), 7.34 (tt, *J* = 7.2, 1.4, 2H), 7.28 (tt, *J* = 7.4, 1.4, 1H), 7.06 (dd, *J* = 8.6, 5.6, 1H), 6.98 (dt, *J* = 8.4, 2.7, 1H), 6.84 (dd, *J* = 8.6, 2.7, 1H) 5.02 (s, 2H), 3.34 (s, 2H), 1.74 (s, 3H), 1.68 (s, 3H), 1.56 (s, 3H), 1.16 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 171.2 (d, *J* = 2.4), 160.7 (d, *J* = 246), 152.6, 139.9 (d, *J* = 6.8), 137.7, 133.0 (d, *J* = 3.3), 130.0 (d, *J* = 7.6), 128.6, 128.3, 127.6, 127.5, 125.2, 116.1 (d, *J* = 21), 113.0 (d, *J* = 23), 83.8, 48.0, 36.1, 27.6, 20.8, 20.7, 18.7; ^{19}F NMR (376 MHz, CDCl$_3$): δ –117.9, (s, 1F); IR (film): 2982, 2920, 1734, 1673, 1369, 1331, 1229, 1149 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{25}H_{31}FNO_3^+$, 412.22825; found 412.22781.



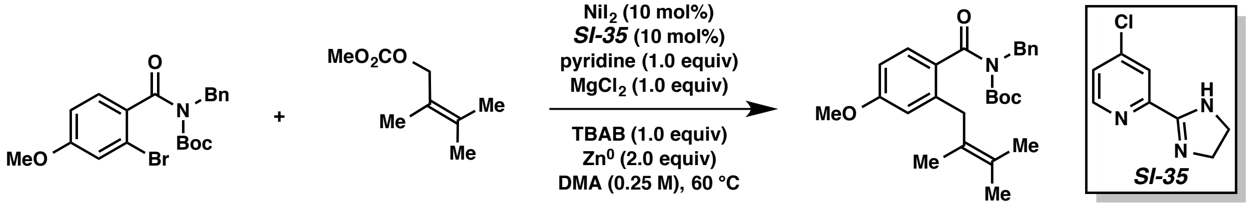
SI-9 ***SI-18*** **(41% yield)** ***SI-46***

Imide SI-46. Following the representative procedure with iodo-imide **SI-9** (455 mg, 0.9 mmol), purification by flash chromatography (199:1 Benzene:EtOAc) yielded imide **SI-46** (169 mg, 41% yield) as a yellow oil. Imide **SI-46**: R$_f$ 0.75 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.54 (dd, *J* = 8.2, 1.4, 1H), 7.43 (d, *J* = 8.0, 2H), 7.37 (d, *J* = 1.5, 1H), 7.35 (tt, *J* = 7.8, 1.4, 2H), 7.29 (tt, *J* = 7.4, 1.3, 1H), 7.24 (d, *J* = 8.1, 1H) 5.05 (s, 2H), 3.44 (s, 2H), 1.76 (s, 3H), 1.69 (s, 3H), 1.58 (s, 3H), 1.12 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 171.2, 152.4, 141.7, 139.3, 137.6, 128.9, 128.7, 128.4, 128.3, 128.2, 127.7, 125.9 (q, *J* = 3.7), 124.4, 124.1 (q, *J* = 274), 122.8 (q, *J* = 3.7), 84.0, 48.1, 36.9, 27.5, 20.78, 20.75, 18.9; ^{19}F NMR (376 MHz, CDCl$_3$): δ –62.4, (s, 3F); IR (film): 2983, 1736, 1672, 1318, 1141, 1123 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{26}H_{31}F_3NO_3^+$, 462.22505; found 462.22240.



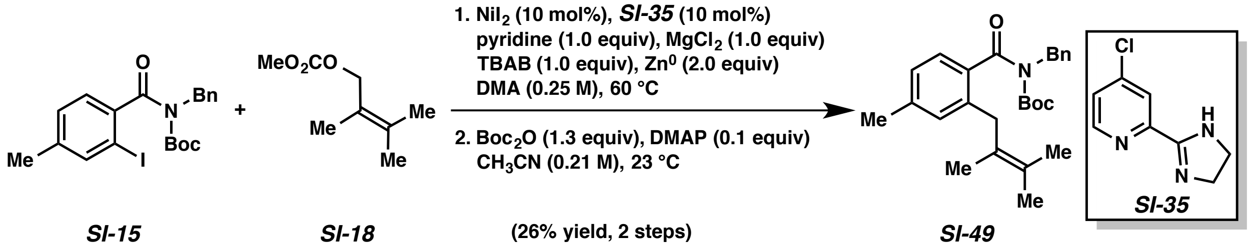
SI-11 **SI-18** (21% yield) **SI-47**

Imide SI-46. Following the representative procedure with iodo-imide **SI-11** (0.42 g, 0.9 mmol), purification by flash chromatography (99:1 Hexanes:EtOAc) yielded imide **SI-47** (79 mg, 21% yield) as a colorless oil. Imide **SI-47**: R_f 0.52 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45–7.42 (m, 2H), 7.36–7.31 (m, 2H), 7.29–7.25 (m, 1H), 7.00 (d, *J* = 8.6, 1H), 6.84 (dd, *J* = 8.6, 2.8, 1H), 6.66 (d, *J* = 2.8, 1H), 5.02 (s, 2H), 3.74 (s, 3H), 3.33 (s, 2H), 1.73 (s, 3H), 1.69 (s, 3H), 1.56 (s, 3H), 1.14 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.4, 157.5, 153.0, 139.4, 138.0, 129.60, 129.57, 128.7, 128.5, 127.6, 127.0, 125.8, 115.3, 111.5, 83.5, 55.6, 48.1, 36.0, 27.7, 20.84, 20.81, 18.8; IR (film): 2980, 1730, 1672, 1142, 1039, 851 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{26}H_{34}NO_4^+$, 424.24824; found 424.24803.

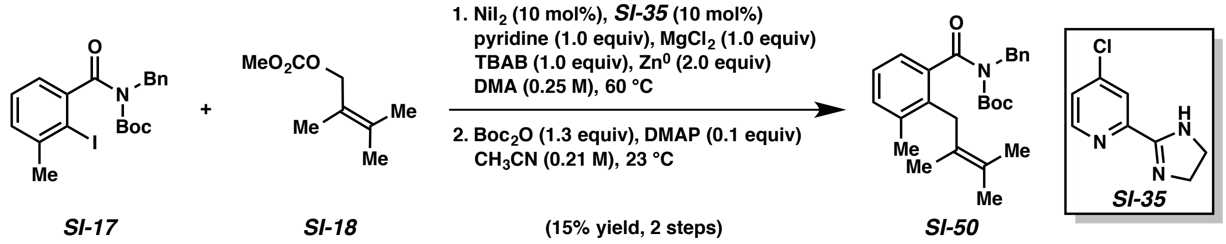


SI-13 **SI-18** (28% yield) **SI-48**

Imide SI-48: Following the representative procedure with bromo-imide **SI-13** (0.84 g, 2.0 mmol), purification by flash chromatography (98:2 Hexanes:EtOAc) yielded imide **SI-48** (0.24 g, 28% yield) as a colorless oil. Imide **SI-48**: R_f 0.40 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.44–7.42 (m, 2H), 7.35–7.32 (m, 2H), 7.28–7.24 (m, 1H), 7.13–7.10 (m, 1H), 6.69–6.66 (m, 2H), 5.00 (s, 2H), 3.78 (s, 3H), 3.47 (s, 2H), 1.75 (s, 3H), 1.70 (s, 3H), 1.59 (s, 3H), 1.15 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): (20 of 21 signals observed) δ 172.5, 160.9, 153.3, 140.7, 138.1, 131.0, 128.6, 128.34, 128.30, 127.4, 125.3, 114.7, 109.7, 83.0, 55.4, 48.4, 37.0, 27.7, 20.8, 18.9; IR (film): 2979, 1726, 1328, 1227, 966, 626 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{26}H_{34}NO_4^+$, 424.24824; found 424.24858.



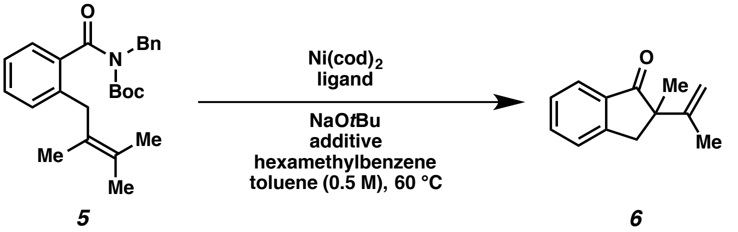
Imide SI-49. Following the representative procedure with iodo-imide **SI-15** (2.1 g, 4.5 mmol) led to appreciable amounts of des-Boc coupled product. As such, the mixture was re-subjected to the general conditions used to install a boc group described in the synthesis of **5** (Section A.1). Purification by flash chromatography (9:1 Hexanes:EtOAc) afforded imide **SI-49** (0.48 g, 26% yield, 2 steps) as a colorless oil. Imide **SI-49**: R_f 0.51 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42–7.39 (m, 2H), 7.33–7.29 (m, 2H), 7.27–7.22 (m, 1H), 7.01 (d, J = 7.7, 1H), 6.94 (d, J = 7.7, 1H), 6.88 (s, 1H), 5.00 (s, 2H), 3.41 (s, 2H), 2.30 (s, 3H), 1.73 (s, 3H), 1.69 (s, 3H), 1.56 (s, 3H), 1.11 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.6, 153.0, 139.4, 137.9, 137.7, 135.5, 129.0, 128.4, 128.1, 127.2, 126.7, 126.1, 125.9, 125.4, 83.0, 47.9, 36.6, 27.4, 21.5, 20.6, 18.6; IR (film): 2980, 1728, 1670, 1229, 1142, 969 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{26}H_{33}NO_3^+$, 408.25332; found 408.25332.



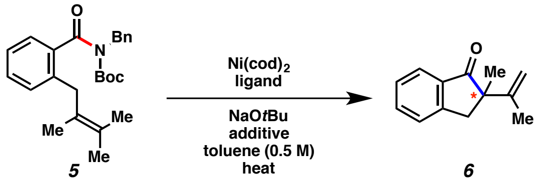
Imide SI-50. Following the representative procedure with iodo-imide **SI-17** (0.93 g, 2.0 mmol) led to appreciable amounts of des-Boc coupled product. As such, the mixture was re-subjected to the general conditions used to install a boc group described in the synthesis of **5** (Section A.1). Purification by flash chromatography (9:1 Hexanes:EtOAc) afforded imide **SI-50** (102 mg, 15% yield, 2 steps) as a colorless oil. Imide **SI-50**: R_f 0.57 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42–7.39 (m, 2H), 7.34–7.30 (m, 2H), 7.28–7.24 (m, 1H), 7.13 (d, J = 8.0, 1H), 7.06 (t, J = 8.0, 1H), 6.95 (d, J = 8.0, 1H), 5.00 (s, 2H), 3.43 (br s, 2H), 2.21 (s, 3H), 1.74 (s, 3H), 1.67 (s, 3H), 1.39 (s, 3H), 1.10 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.1, 153.0, 139.6, 138.3, 138.2, 136.5, 131.2, 128.6, 128.4, 127.5, 125.7, 125.3, 125.1, 123.8, 83.8, 48.0, 34.8, 27.6, 21.1, 20.7, 19.8, 16.9; IR (film): 2979, 1729, 1674, 1368, 1144, 698 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{26}H_{34}NO_3^+$, 408.25332; found 408.25311.

B. Initial Evaluation of Ligand Effects and Reaction Conditions

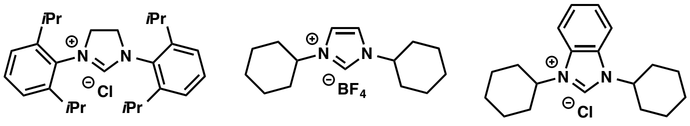
Representative Procedure for the Nickel-Catalyzed Heck Cyclization of Imides



Indanone 6 (Table 1). A dram vial containing imide **5** (39.3 mg, 0.10 mmol, 1.0 equiv), hexamethylbenzene, and a magnetic stir bar was sequentially charged with the appropriate ligand, Ni(cod)$_2$, and NaO*t*-Bu in a glovebox. Subsequently, toluene (0.20 mL) and the additive (when applicable) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, wrapped with Teflon tape, and stirred at the appropriate temperature for 24 h. After cooling to room temperature, the mixture was diluted with Hexanes (1.0 mL) and filtered through a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure, and the yield was determined by ^{1}H NMR analysis with hexamethylbenzene as the internal standard.



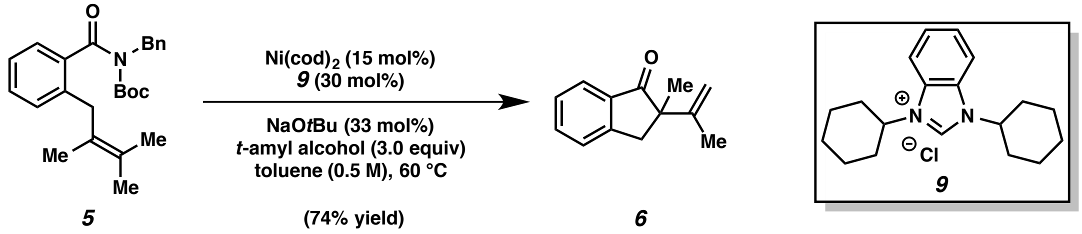
Entry	*Ni(cod)$_2$ (loading)*	*Ligand (loading)*	*Additive*	*Temp.*	*Yield^b*
1	20 mol%	*7* (40 mol%)	none	100 °C	0%
2	20 mol%	*8* (40 mol%)	none	100 °C	24%
3	20 mol%	*9* (40 mol%)	none	100 °C	76%
4	20 mol%	*9* (20 mol%)	none	100 °C	67%
5	10 mol%	*9* (20 mol%)	none	100 °C	51%
6	15 mol%	*9* (30 mol%)	none	100 °C	91%
7	15 mol%	*9* (30 mol%)	*t*-amyl alcohol^c	60 °C	95%



SIPr•HCl (7)　*ICy•HBF$_4$ (8)*　*Benz-ICy•HCl (9)*

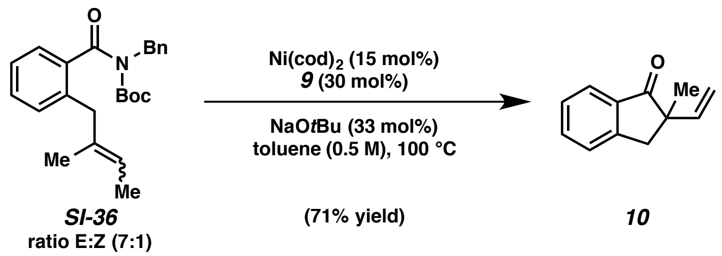
C. Scope of Methodology

Representative Procedure for the Nickel-Catalyzed Heck Cyclization of Imides (synthesis of indanone 6 is used as an example).

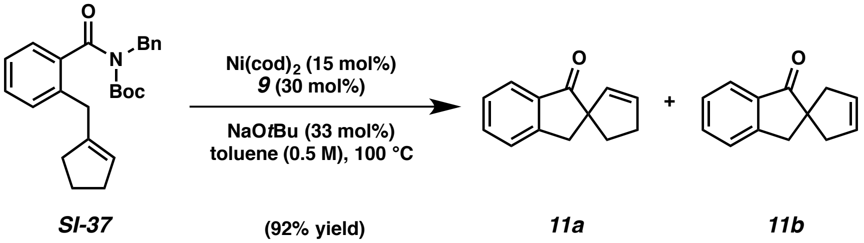


Indanone 6 (Table 2). A dram vial containing imide **5** (39.3 mg, 0.10 mmol, 1.0 equiv) and a magnetic stir bar was sequentially charged with **9** (9.6 mg, 0.030 mmol, 30 mol%), Ni(cod)$_2$ (4.1 mg, 0.015 mmol, 15 mol%), and NaO*t*-Bu (3.2 mg, 0.033 mmol, 33 mol%) in a glovebox. Subsequently, toluene (0.20 mL) and then *t*-amyl alcohol (32 µL, 0.30 mmol, 3.0 equiv) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glovebox, wrapped with Teflon tape and stirred at 60 °C for 24 h. After cooling to room temperature, the mixture was diluted with hexanes (1.0 mL) and filtered through a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure. ^{1}H NMR analysis of the crude reaction mixture indicated a 95% yield (average of two experiments) of ketone **6** relative to a hexamethylbenzene external standard. Purification by preparative thin-layer chromatography (3:1 Hexanes:EtOAc) afforded indanone **6** (74% yield, average of two experiments) as a colorless oil. The diminished isolated yields of **6** can be attributed to the volatility of the neat compound. Indanone **6**: R_f 0.59 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.78 (d, *J* = 7.7, 1H), 7.61 (td, *J* = 7.5, 1.1, 1H), 7.45 (dt, *J* = 7.7, 0.9, 1H), 7.38 (td, *J* = 7.5, 0.7, 1H), 4.95 (m, 2H), 3.33 (d, *J* = 17.4, 1H), 2.95 (d, *J* = 17.4, 1H), 1.65 (s, 3H), 1.38 (s, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 209.2, 152.7, 145.9, 135.9, 135.0, 127.6, 126.5, 124.6, 112.1, 54.5, 41.3, 22.7, 19.9; IR (film): 2966, 2928, 1710, 1604, 1464, 1277 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{13}H_{15}O$, 187.11174; found 187.11130.

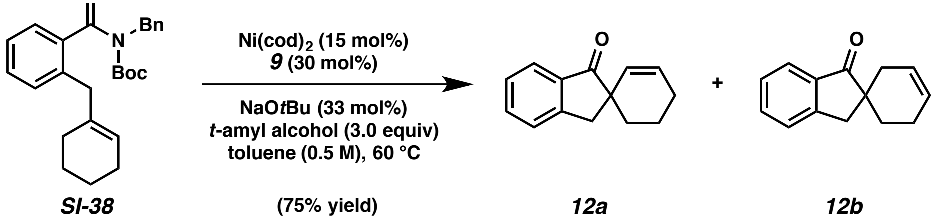
Any modifications of the conditions shown in the representative procedure above are specified in the following schemes, which depict all of the results shown in Table 2 and Figure 2.



Indanone 10 (Table 2). Purification by preparative thin-layer chromatography (9:1 Hexanes:EtOAc) afforded indanone **10** (71% yield, average of two experiments) as a colorless oil. Indanone **10**: R_f 0.59 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77 (d, *J* = 7.8, 1H), 7.60 (td, *J* = 7.5, 1.2, 1H), 7.45 (dt, *J* = 7.7, 0.9, 1H), 7.38 (t, *J* = 7.8, 1H), 5.95 (dd, *J* = 7.5, 10.6, 1H), 5.20–5.11 (m, 2H), 3.32 (d, *J* = 17.0, 1H), 3.02 (d, *J* = 17.0, 1H), 1.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 208.0, 152.0, 140.6, 135.2, 134.9, 127.5, 126.4, 124.6, 113.9, 52.3, 40.7, 23.2; IR (film): 2966, 1714, 1465, 1279, 738 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{12}H_{13}O^+$, 173.09609; found 173.09618.

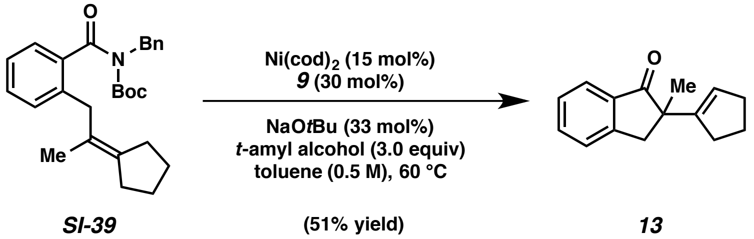


Indanone 11 (Table 2). Purification by preparative thin-layer chromatography (95:5 Benzene:CH_3CN) afforded indanones **11a** and **11b** (92% combined yield, average of two experiments) as a ~1:1 mixture of olefin isomers. Iterative purification by preparative thin-layer chromatography (Benzene) afforded analytical samples of indanones **11a** and **11b** as colorless oils. Spectral data match those previously reported.[9]

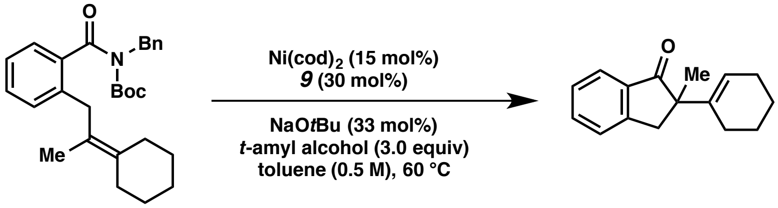


Indanone 12 (Table 2). Purification by preparative thin-layer chromatography (19:1 Benzene:CH_3CN) afforded indanones **12a** and **12b** (75% combined yield, average of two

experiments) as a ~1:1 mixture of olefin isomers. Iterative purification by preparative thin-layer chromatography (Benzene) afforded analytical samples of **12a** and **12b** as colorless oils. Indanone **12a**: R*f* 0.47 (benzene); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77 (d, *J* = 7.6, 1H) 7.59 (td, *J* = 7.6, 1.1, 1H), 7.43 (d, *J* = 7.6, 1H), 7.37 (t, *J* = 7.6, 1H), 6.00 (ddd, *J* = 7.5, 4.2, 3.3, 1H), 5.45 (d, *J* = 9.9, 1H), 3.14 (d, *J* = 17.1, 1H), 3.07 (d, *J* = 17.1, 1H), 2.22–2.13 (m, 1H), 2.13–2.04 (m, 1H), 2.04–1.96 (m, 1H), 1.90 (td, *J* = 11.6, 2.7, 1H), 1.69–1.57 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 210.1, 152.6, 135.9, 135.0, 130.2, 128.3, 127.7, 126.7, 124.7, 51.4, 42.6, 32.8, 24.6, 19.5; IR (film): 3019, 2930, 1712, 1605, 1464, 1282 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{15}O^+$, 199.11174; found 199.11026. Indanone **12b**: R*f* 0.50 (benzene); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77 (d, *J* = 7.7, 1H) 7.59 (td, *J* = 7.6, 0.9, 1H), 7.44 (d, *J* = 7.5, 1H), 7.38 (t, *J* = 7.5, 1H), 5.79 (m, 2H), 3.06 (d, *J* = 17.3, 1H), 2.93 (d, *J* = 17.3, 1H), 2.48 (dquint *J* = 17.7, 2.5, 1H), 2.30–2.14 (m, 2H), 1.91 (ddd, *J* = 17.5, 11.1, 6.6, 1H), 1.79 (dt, *J* = 17.7, 2.5, 1H), 1.50 (dt, *J* = 17.5, 2.7, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 211.3, 153.0, 136.0, 135.0, 127.6, 126.81, 126.76, 125.3, 124.5, 48.5, 39.2, 34.1, 28.7, 22.8; IR (film): 3026, 2925, 2840, 1712, 1608, 1284 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{15}O^+$, 199.11174; found 199.11028.

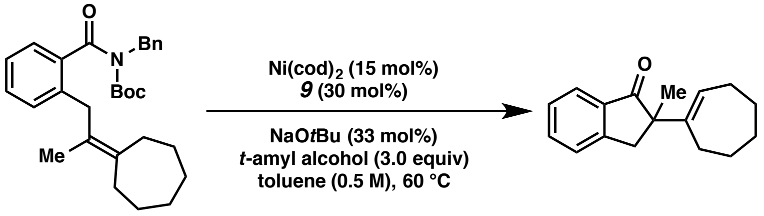


Indanone 13 (Table 2). Purification by preparative thin-layer chromatography (97:3 Benzene:CH_3CN) afforded indanone **13** (51% yield, average of two experiments) as a colorless oil. Indanone **13**: R*f* 0.25 (9:1 Hexane:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77 (d, *J* = 7.7, 1H), 7.60 (dt, *J* = 7.5, 1.2, 1H), 7.44 (td, *J* = 7.7, 0.9, 1H), 7.37 (dt, *J* = 7.5, 0.9, 1H), 5.57 (quint, *J* = 2.2, 1H), 3.32 (d, *J* = 17.2, 1H), 2.97 (d, *J* = 17.2, 1H), 2.34–2.20 (m, 3H), 2.15–2.06 (m, 1H), 1.88–1.78 (m, 2H), 1.41 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 208.9, 152.7, 145.5, 135.8, 135.0, 127.6, 126.6, 125.7, 124.7, 51.6, 41.4, 32.4, 32.2, 23.6, 22.9; IR (film): 2956, 2929, 2846, 1713, 1608, 1464, 1280 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{15}H_{17}O^+$, 213.12739; found 213.12580.



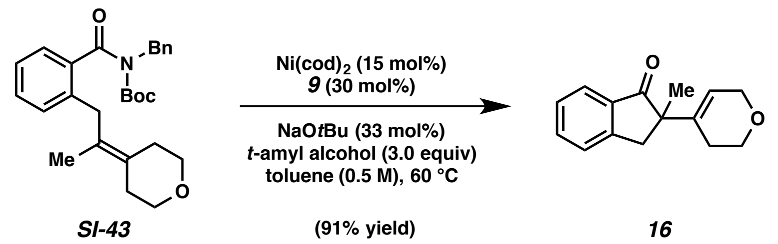
SI-40 (96% yield) *14*

Indanone 14 (Table 2). Purification by preparative thin-layer chromatography (9:1 Benzene:CH_3CN) afforded indanone **14** (96% yield, average of two experiments) as a colorless oil. Indanone **14**: R_f 0.48 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.76 (d, *J* = 7.6, 1H), 7.59 (td, *J* = 7.6, 1.2, 1H), 7.43 (dt, *J* = 7.6, 0.9, 1H), 7.39–7.34 (m, 1H), 5.65 (d, *J* = 17.6, 1H), 3.29 (d, *J* = 17.4, 1H), 2.91 (d, *J* = 17.4, 1H), 2.13–1.99 (m, 2H), 1.92–1.83 (m, 1H), 1.79–1.70 (m, 1H), 1.63–1.50 (m, 4H), 1.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 210.2, 153.1, 138.5, 136.3, 134.9, 127.5, 126.6, 124.5, 122.4, 54.6, 41.7, 25.5 (two carbons), 23.1, 22.32, 22.28; IR (film): 2928, 1712, 1464, 1153, 736 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{16}H_{19}O^+$, 227.14304; found 227.14236.

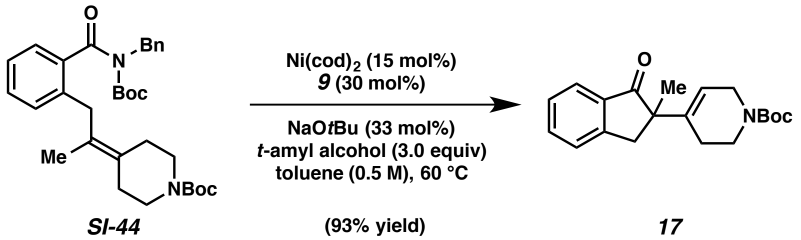


SI-42 (80% yield) *15*

Indanone 15 (Table 2). Purification by preparative thin-layer chromatography (97:3 Benzene:CH_3CN) afforded indanone **15** (80% yield, average of two experiments) as a colorless oil. Indanone **15**: R_f 0.48 (9:1 Hexane:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.78 (d, *J* = 7.7, 1H), 7.60 (dt, *J* = 7.5, 1.2, 1H), 7.44 (td, *J* = 7.7, 0.9, 1H), 7.37 (dt, *J* = 7.5, 0.9, 1H), 5.86 (t, *J* = 6.8, 1H), 3.26 (d, *J* = 17.4, 1H), 2.89 (d, *J* = 17.4, 1H), 2.22–2.10 (m, 2H), 1.98 (ddd, *J* = 14.9, 9.5, 1.7, 1H), 1.89 (ddd, *J* = 14.9, 8.9, 1.7, 1H), 1.78–1.64 (m, 2H), 1.55–1.45 (m, 2H) 1.45–1.30 (m, 2H), 1.29 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 210.0, 153.1, 145.0, 136.4, 134.9. 127.5, 127.4, 126.7, 124.6, 55.9, 40.9, 33.0, 31.1, 28.5, 27.4, 26.9, 22.7; IR (film): 2920, 2947, 1710, 1607, 1463, 1440, 1750 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{17}H_{21}O^+$, 241.15869; found 241.15748.

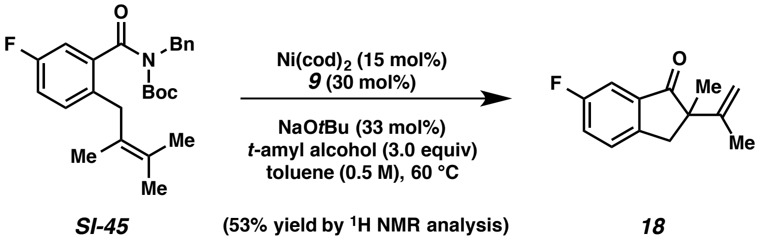


Indanone 16 (Table 2). Purification by preparative thin-layer chromatography (2:1 Hexanes:EtOAc) afforded indanone **16** (91% yield, average of two experiments) as a colorless oil. Indanone **16**: R_f 0.34 (3:1 Hexanes:EtOAc); ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.77 (d, *J* = 7.8, 1H), 7.61 (td, *J* = 7.6, 1.3, 1H), 7.45 (dt, *J* = 7.8, 1.0, 1H), 7.39 (t, *J* = 7.6, 1H), 5.65 (app sext, *J* = 1.4, 1H), 4.19 (app tquint, *J* = 16.4, 2.6, 2H), 3.73 (td, *J* = 5.0, 1.1, 2H), 3.34 (d, *J* = 17.3, 1H), 2.95 (d, *J* = 17.3, 1H), 2.12 (m, 1H), 1.86 (m, 1H), 1.37 (s, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 209.0, 152.8, 136.4, 136.0, 135.2, 127.8, 126.6, 124.7, 121.4, 65.9, 64.3, 54.0, 41.0, 25.6, 22.1; IR (film): 2961, 2927, 2850, 1709, 1606, 1464, 1277, 1127 cm^{-1}; HRMS–APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{17}O_2^+$, 229.12231; found 229.12094.

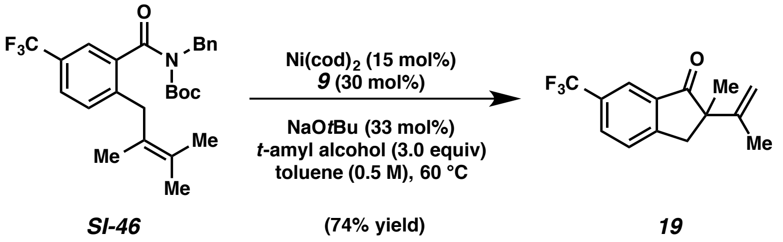


Indanone 17 (Table 2). Purification by preparative thin-layer chromatography (2:1 Hexanes:EtOAc) afforded indanone **17** (93% yield, average of two experiments) as a colorless oil. Indanone **17**: R_f 0.37 (3:1 Hexanes:EtOAc); ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.77 (d *J* = 7.6, 1H) 7.61 (td, *J* = 7.6, 1.1, 1H), 7.45 (d, *J* = 7.7, 1H), 7.39 (t, *J* = 7.7, 1H), 5.61 (br s, 1H), 3.93 (s, 2H), 3.43 (t, *J* = 4.7, 2H), 3.30 (d, *J* = 17.4, 1H), 2.95 (d, *J* = 17.4, 1H), 2.06 (m, 1H), 1.87 (m, 1H), 1.45 (s, 9H), 1.36 (s, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 209.1, 154.9, 152.7, 137.5, 135.9, 135.2, 127.8, 126.6, 124.7, 119.6, 79.7, 54.1, 43.5, 41.0, 39.7, 28.6, 25.7, 22.3; IR (film): 2975, 2931, 1695, 1419, 1365, 1241, 1171 cm^{-1}; HRMS–ESI (*m/z*) $[M + Na]^+$ calcd for $C_{20}H_{25}NNaO_3^+$, 350.1732; found 350.1736.

*Note: The data for indanone **17** represents empirically observed chemical shifts from the ^{13}C NMR spectrum, presumably due to the nitrogen-containing heterocycle.*

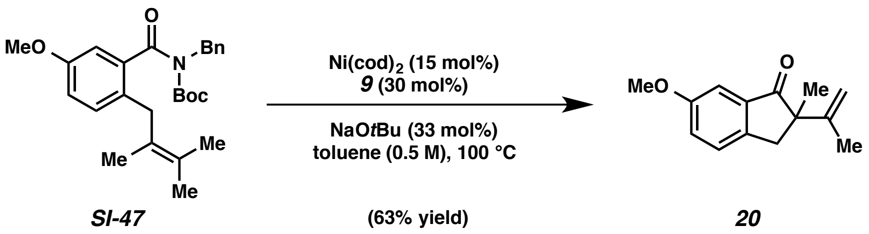


Indanone 18 (Figure 2). ^{1}H NMR analysis of the crude reaction mixture indicated a 53% yield (average of two experiments) of ketone **18** relative to a hexamethylbenzene external standard. Purification by preparative thin-layer chromatography (97:3 Benzene: CH_3CN) afforded an analytical sample of **18** as an off-white oil. Diminished isolated yields (i.e. <50%) for **18** were observed and can be attributed to the volatility of the neat compound. Indanone **18**: R_f 0.59 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.44–7.39 (m, 2H), 7.32 (td, J = 8.6, 2.4, 1H), 4.94 (m, 2H), 3.29 (d, J = 17.8, 1H), 2.91 (d, J = 17.8, 1H), 1.65 (q, J = 0.7, 3H), 1.38 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 208.3 (d, J = 2.6), 162.5 (d, J = 248), 148.1 (d, J = 2.1), 145.6, 137.7 (d, J = 7.0), 128.0 (d, J = 7.9), 122.8 (d, J = 24), 112.4, 110.4 (d, J = 22), 55.7, 40.8, 22.7, 20.0; ^{19}F NMR (376 MHz, $CDCl_3$): δ –114.3 (s, 1F); IR (film): 2970, 2929, 1712, 1484, 1447, 1263 cm^{-1}; HRMS–APCI (m/z) $[M + H]^+$ calcd for $C_{13}H_{14}FO^+$, 205.10232; found 205.10239.

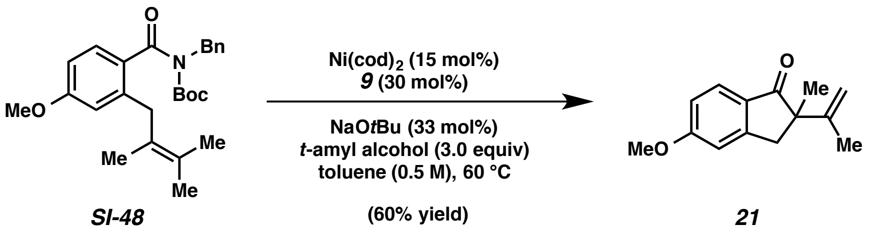


Indanone 19 (Figure 2). Purification by preparative thin-layer chromatography (4:1 Hexanes:EtOAc) afforded indanone **19** (74% yield, average of two experiments) as a yellow oil. Indanone **19**: R_f 0.69 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.05 (s, 1H), 7.32 (dd, J = 7.9, 1.3, 1H), 7.59 (d, J = 7.9, 1H), 4.97 (quint, J = 1.1, 1H), 4.95 (s, 1H), 3.40 (d, J = 18.0, 1H), 3.02 (d, J = 18.0, 1H), 1.67 (q, J = 0.7, 3H), 1.40 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.9, 156.0, 145.2, 136.4, 131.5 (q, J = 3.5), 130.6 (q, J = 33), 127.3, 123.9 (q, J =

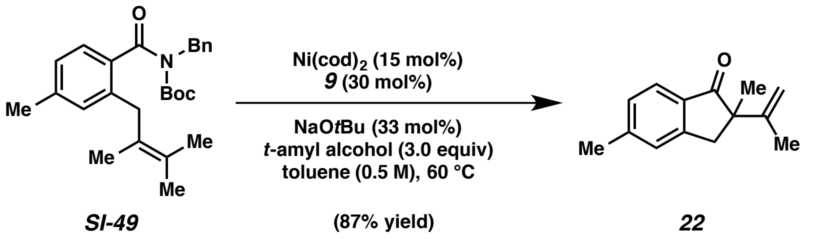
273), 122.0 (d, $J = 3.9$), 112.8, 55.2, 41.4, 22.7, 20.0; ^{19}F NMR (376 MHz, $CDCl_3$): δ –62.5 (s, 3F); IR (film): 2972, 2936, 1722, 1625, 1332, 1257, 1184, 1128 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{14}F_3O^+$, 255.09913; found 255.09744.



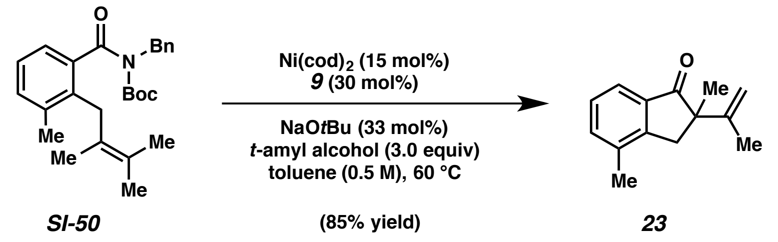
Indanone 20 (Figure 2). Purification by preparative thin-layer chromatography (99:1 Benzene:CH_3CN) afforded indanone **20** (63% yield, average of two experiments) as a white crystalline solid. Indanone **20**: mp: 41–43 ºC; R$_f$ 0.32 (10:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33 (d, $J = 8.0$, 1H), 7.23–7.18 (m, 2H), 4.95 (m, 2H), 3.84 (s, 3H), 3.24 (d, $J = 17.4$, 1H), 2.87 (d, $J = 17.4$, 1H), 1.64 (s, 3H), 1.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 209.2, 159.5, 145.9, 145.5, 137.0, 127.2, 124.5, 112.0, 105.5, 55.6, 40.6, 22.7, 19.8; IR (film): 2964, 1707, 1275, 1280, 894 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{18}O_2^+$, 217.12231; found 217.12123.



Indanone 21 (Figure 2). Purification by preparative thin-layer chromatography (9:1 Hexanes:Et_3N) afforded indanone **21** (60% yield, average of two experiments). Spectral data match those previously reported.[10]

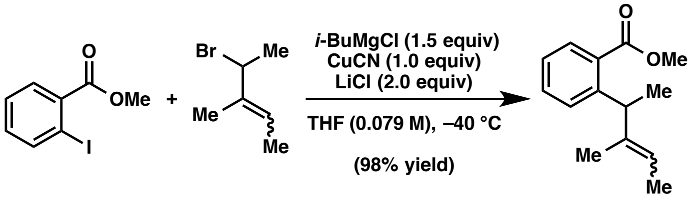


Indanone 22 (Figure 2). Purification by preparative thin-layer chromatography (9:1 Hexanes:EtOAc) afforded indanone **22** (87% yield, average of two experiments) as a colorless oil. Indanone **22**: R_f 0.43 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.67 (d, *J* = 7.8, 1H), 7.25–7.23 (m, 1H), 7.20–7.18 (m, 1H), 4.95–4.93 (m, 2H), 3.27 (d, *J* = 17.6, 1H), 2.89 (d, *J* = 17.4, 1H), 2.44 (s, 3H), 1.63 (s, 3H), 1.36 (s, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 208.8, 153.3, 146.3, 146.1, 133.7, 128.9, 126.9, 124.5, 112.0, 54.7, 41.2, 22.8, 22.2, 19.9; IR (film): 2965, 1705, 1608, 1322, 585 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{17}O^+$, 201.12739; found 201.12719.



Indanone 23 (Figure 2). Purification by preparative thin-layer chromatography (9:1 Hexanes:EtOAc) afforded indanone **23** (85% yield, average of two experiments) as a colorless oil. Indanone **23**: R_f 0.38 (10:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.63 (d, *J* = 7.4, 1H), 7.43 (d, *J* = 7.4, 1H), 7.31 (t, *J* = 7.4, 1H), 4.97–4.94 (m, 2H), 3.21 (d, *J* = 17.5, 1H), 2.84 (d, *J* = 17.4, 1H), 2.35 (s, 3H), 1.65 (s, 3H), 1.39 (s, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 209.5, 151.6, 146.0, 135.67, 135.66, 135.4, 127.8, 122.0, 112.0, 54.5, 40.3, 22.8, 19.9, 17.8; IR (film): 2966, 1708, 1591, 1268, 893 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{17}O^+$, 201.12739; found 201.12701.

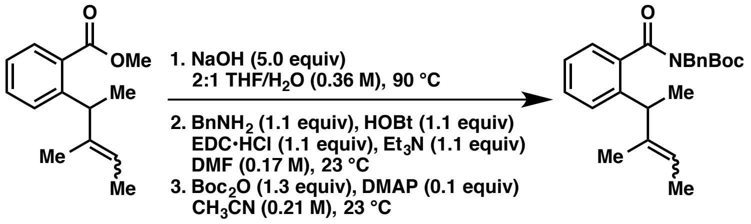
D. Diastereoselective Heck Cyclization



SI-1 ***SI-51*** ***SI-52***
ratio E:Z (4:1)

Ester SI-52. Following a modification of the general procedure reported by Querolle and co-workers,[3] a flask containing a stir bar was charged CuCN (680 mg, 7.50 mmol, 1.0 equiv) and LiCl (650 mg, 15.0 mmol, 2.0 equiv) in the glovebox. The flask was removed from the glovebox, and the solids were suspended in THF (25 mL). The resulting mixture was stirred vigorously until a completely dissolved solution of CuCN•2LiCl was formed. In a separate flask containing a solution of methyl-2-iodobenzoate (**SI-1**) (1.97 g, 7.50 mmol, 1.0 equiv) in THF (70 mL) at –40 °C was added *i*-BuMgCl (5.60 mL of a 2.0 M solution in THF, 11.3 mmol, 1.5 equiv) dropwise over 1 min at –40 °C. After this mixture was stirred at –40 °C for 1 h, the solution of CuCN•2LiCl was added via cannula. The combined mixture was stirred at –40 °C for an additional 15 min, at which point known bromide **SI-51**[11] (2.43 g, 15.0 mmol, 2.0 equiv) was added dropwise over 1 min. After stirring at –40 °C for an additional hour, the reaction was poured into 9:1 sat. aq. NH_4Cl:NH_4OH (100 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 75 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated under reduced pressure. The crude mixture was purified via flash chromatography (97:3 Hexanes:EtOAc) to afford ester **SI-52** (1.61 g, 98% yield) as an inseparable mixture of olefin isomers and as a colorless oil (4:1 mixture of alkene isomers). Configurational isomers of ester **SI-52** were analyzed as a mixture. Ester **SI-52**: R_f 0.55 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): Major (*E*)-isomer **SI-52** δ 7.71 (dd, *J* = 7.7, 1.5, 1H), 7.40 (dt, *J* = 7.8, 1.6, 1H), 7.30 (dd, *J* = 8.0, 1.24, 1H), 7.22 (dt, *J* = 7.7, 1.6, 1H), 5.38 (tq, *J* = 6.7, 1.6, 1H), 4.23 (q, *J* = 7.1, 1H), 3.88 (s, 3H), 1.63 (dt, *J* = 6.8, 1.2, 3H), 1.45 (br s, 3H), 1.33 (d, *J* = 7.0, 3H); Minor (*Z*)-isomer **SI-52** δ 7.60 (dd, *J* = 7.8, 1.3, 1H), 7.45–7.38 (m, 2H), 7.23 (dt, *J* = 7.6, 1.7, 1H), 5.25 (tq, *J* = 6.9, 0.8, 1H), 4.78 (q, *J* = 7.2, 1H), 3.84 (s, 3H), 1.61 (q, *J* = 1.5, 3H), 1.40 (quint, *J* = 1.5, 3H), 1.35 (d, *J* = 7.3, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): (27 of 28 signals observed) δ 169.4, 169.1, 146.9, 145.1, 139.3, 138.9, 131.7, 131.6, 131.1, 130.9, 129.8, 129.4, 127.8, 127.6, 125.8, 120.3, 118.3, 52.3, 52.1, 42.7, 35.3, 20.3, 20.2, 18.3, 16.0, 13.6, 13.3;

IR (film): 2968, 1721, 1601, 1576, 1485, 1446, 1371 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{19}O_2^+$, 219.13796; found 219.13794.



SI-52
ratio E:Z (4:1)

(68% yield, 3 steps)

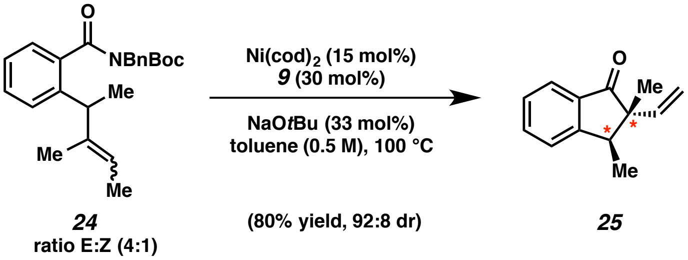
24
ratio E:Z (4:1)

Imide 24. To a solution of ester **SI-52** (1.61 g, 7.40 mmol, 1.0 equiv) in THF (35 mL) was added a solution of NaOH (1.48 g, 37.0 mmol, 5.0 equiv) in H_2O (35 mL). The reaction was heated to 90 °C and stirred for 12 h. After cooling to room temperature, the reaction mixture was poured into deionized water (25 mL) and diluted with EtOAc (25 mL). The layers were separated and the aqueous layer was acidified to pH ~2 with 1 N HCl (100 mL) and extracted with EtOAc (3 x 50 mL). The organic layers were combined, washed with deionized water (300 mL), dried over $MgSO_4$, and concentrated under reduced pressure to afford the corresponding carboxylic acid, which was used in the subsequent step without further purification.

To a solution of the crude carboxylic acid, HOBt (1.08 g, 7.80 mmol, 1.1 equiv from **SI-52**), and EDC•HCl (1.53 g, 7.80 mmol, 1.1 equiv from **SI-52**) in DMF (40 mL) was added benzylamine (0.90 mL, 7.80 mmol, 1.1 equiv from **SI-52**) and triethylamine (1.17 mL, 7.80 mmol, 1.1 equiv from **SI-52**). After stirring for 15 h, the reaction mixture was poured into deionized water (300 mL) and diluted with EtOAc (50 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 50 mL). The organic layers were combined, washed with deionized water (100 mL), dried over $MgSO_4$, and concentrated under reduced pressure to afford the corresponding amide, which was used in the subsequent step without further purification.

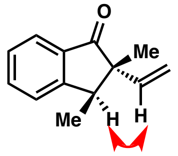
To a solution of the crude amide in CH_3CN (45 mL) was added DMAP (82 mg, 0.65 mmol, 0.1 equiv) and Boc_2O (1.85 g, 8.50 mmol, 1.3 equiv from **SI-52**). After stirring for 15 h, the reaction mixture was concentrated under reduced pressure and purified by flash chromatography (99:1 Hexanes:EtOAc) to yield imide **24** (1.97 g, 68% yield, 3 steps) as an inseparable mixture of olefin isomers and as a colorless oil (4:1 mixture of alkene isomers).

Configurational isomers of imide **24** were analyzed as a mixture. Imide **24**: R$_f$ 0.52 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): Major (*E*)-isomer **24** δ 7.43 (d, *J* = 7.5, 2H), 7.37–7.31 (m, 3H), 7.29–7.22 (m, 2H), 7.16 (dt, *J* = 7.7, 1.4, 1H), 7.12–7.08 (m, 1H), 5.36 (tq, *J* = 6.8, 1.3, 1H), 5.06 (s, 2H), 3.67 (q, *J* = 6.9, 1H), 1.60 (d, *J* = 6.7, 3H), 1.48 (s, 3H), 1.30 (d, *J* = 7.0, 3H), 1.13 (s, 9H); Minor (*E*)-isomer **24** δ 7.45–7.37 (m, 2H), 7.37–7.31 (m, 3H), 7.29–7.22 (m, 2H), 7.18 (dt, *J* = 7.5, 1.3, 1H), 7.12–7.08 (m, 1H), 5.22 (br s, 1H), 4.99 (br s, 2H), 4.34 (br s, 1H), 1.57 (d, *J* = 6.9, 3H), 1.53 (t, *J* = 1.4, 3H), 1.34 (d, *J* = 7.2, 3H), 1.11 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 172.4, 152.8, 142.8, 141.3, 138.9, 138.6, 138.1, 138.04, 138.02, 129.4, 129.0, 128.5, 128.3, 127.5, 127.4, 126.3, 126.2, 125.4, 120.0, 118.6, 83.5, 48.0, 43.2, 27.7, 27.6, 20.2, 18.7, 15.5, 13.5, 13.3; IR (film): 2973, 1728, 1670, 1456, 1369, 1335, 1228 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{25}H_{32}NO_3^+$, 394.23767; found 394.23814.



Indanone 25 (Figure 3). Following the representative procedure described in Section C, purification by preparative thin-layer chromatography (98:2 Benzene:CH$_3$CN) afforded indanone **25** (80% yield, 92:8 dr, average of two experiments) as a colorless oil. Indanone **25**: R$_f$ 0.48 (9:1 Hexane:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.75 (d, *J* = 7.7, 1H), 7.62 (dt, *J* = 7.7, 1.2, 1H), 7.49 (dd, *J* = 7.8, 0.9, 1H), 7.38 (tt, *J* = 7.5, 0.9, 1H), 5.94 (dd, *J* = 17.4, 10.6, 1H), 5.23–5.15 (m, 2H), 3.40 (q, *J* = 7.5, 1H), 1.32 (d, *J* = 7.4, 3H), 1.19 (s, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 208.4, 157.2, 141.0, 135.1, 134.5, 127.8, 125.1, 124.5, 114.6, 56.2, 43.1, 18.5, 15.1; IR (film): 2972, 1712, 1606, 1466, 1328, 1285, 1226 cm^{-1}; HRMS–APCI (*m/z*) [M + H]$^+$ calcd for $C_{13}H_{15}O^+$, 187.11174; found 187.11180.

*The stereochemistry of indanone **25** was verified by NOESY (500 MHz, CDCl$_3$), as the following correlation was observed:*



References

[1] Cui, X.; Wang, S.; Zhang, Y.; Deng, W.; Qian, Q.; Gong, H. *Org. Biomol. Chem.* **2013**, *11*, 3094–3097.

[2] Hansen, E. C.; Pedro, D. J.; Wotal, A. C.; Gower, N. J.; Nelson, J. D.; Caron, S.; Weix, D. J. *Nat. Chem.* **2016**, *8*, 1226–1230.

[3] Querolle, O.; Dubois, J.; Thoret, S.; Roussi, F.; Guéritte, F.; Guénard, D. *J. Med. Chem.* **2004**, *47*, 5937–5944.

[4] Clennan, E. L.; Chen, X. *J. Am. Chem. Soc.* **1989**, *111*, 5787–5792.

[5] Tanaka, K.; Uneme, H.; Ono, N.; Kaji, A. *Chem. Lett.* **1979**, *9*, 1039–1040.

[6] Friese, A.; Hell-Momeni, K.; Zundorf, I.; Winckler, T.; Dingermann, T.; Dannhardt, G. *J. Med. Chem*. **2002**, *45*, 1535–1542.

[7] Shimano, M.; Kamei, N.; Tanaka, T.; Harada, T.; Haino, M.; Okuyama, A.; Arakawa, Y. Murakami, Y. Reverse Hydroxamic Acid Derivatives. Eur. Pat. Appl. 1431285. June 23rd, 2004.

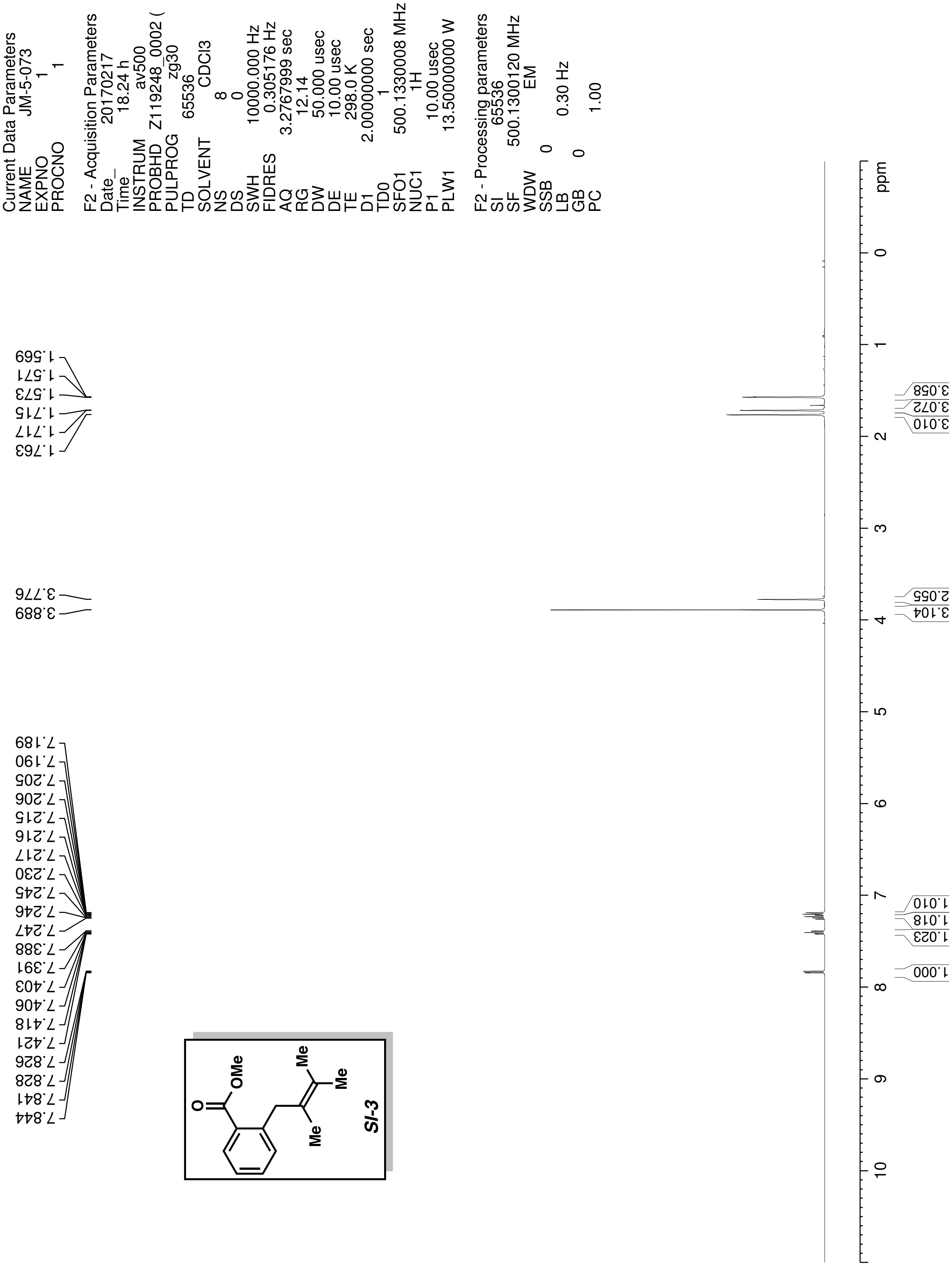
[8] Sato, K.; Sugimoto, H.; Rikimaru, K.; Imoto, H.; Kamaura, M.; Negoro, N.; Tsujihata, Y.; Miyashita, H.; Odani, T.; Murata, T. *Bioorg. Med. Chem.* **2014**, *22*, 1649–1666.

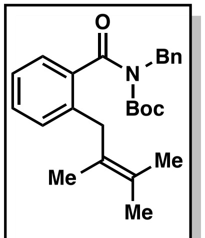
[9] (a) Mori, M.; Kaneta, N.; Isono, N.; Shibasaki, M. *J. Organomet. Chem*. **1993**, *455*, 255–260. (b) Kotha, S.; Mandal, K.; Tiwari, A.; Mobin, S. M. *Chem. Eur. J.* **2006**, *12*, 8024–8038.

[10] Hayashi, T.; Tang, J.; Kato, K. *Org. Lett.* **1999**, *1*, 1487–1489.

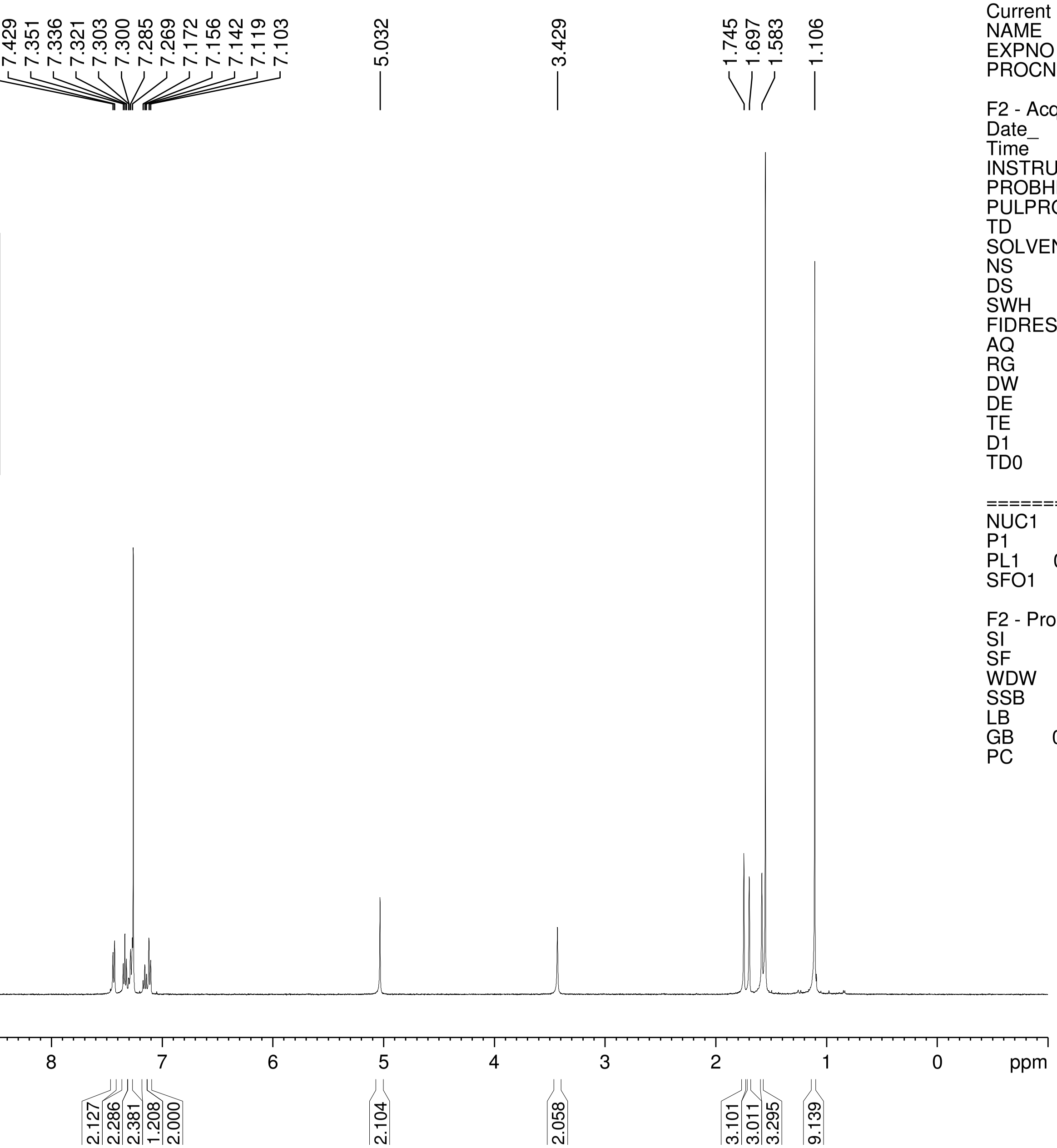
[11] Van Zyl. C. M.; McKeeby, J. L.; Van Dort, P. C.; Larson, E. J.; Silver, M. E. *Inorg. Chim. Acta*. **1987**, *133*, 289–294.

^{1}H NMR Spectra:

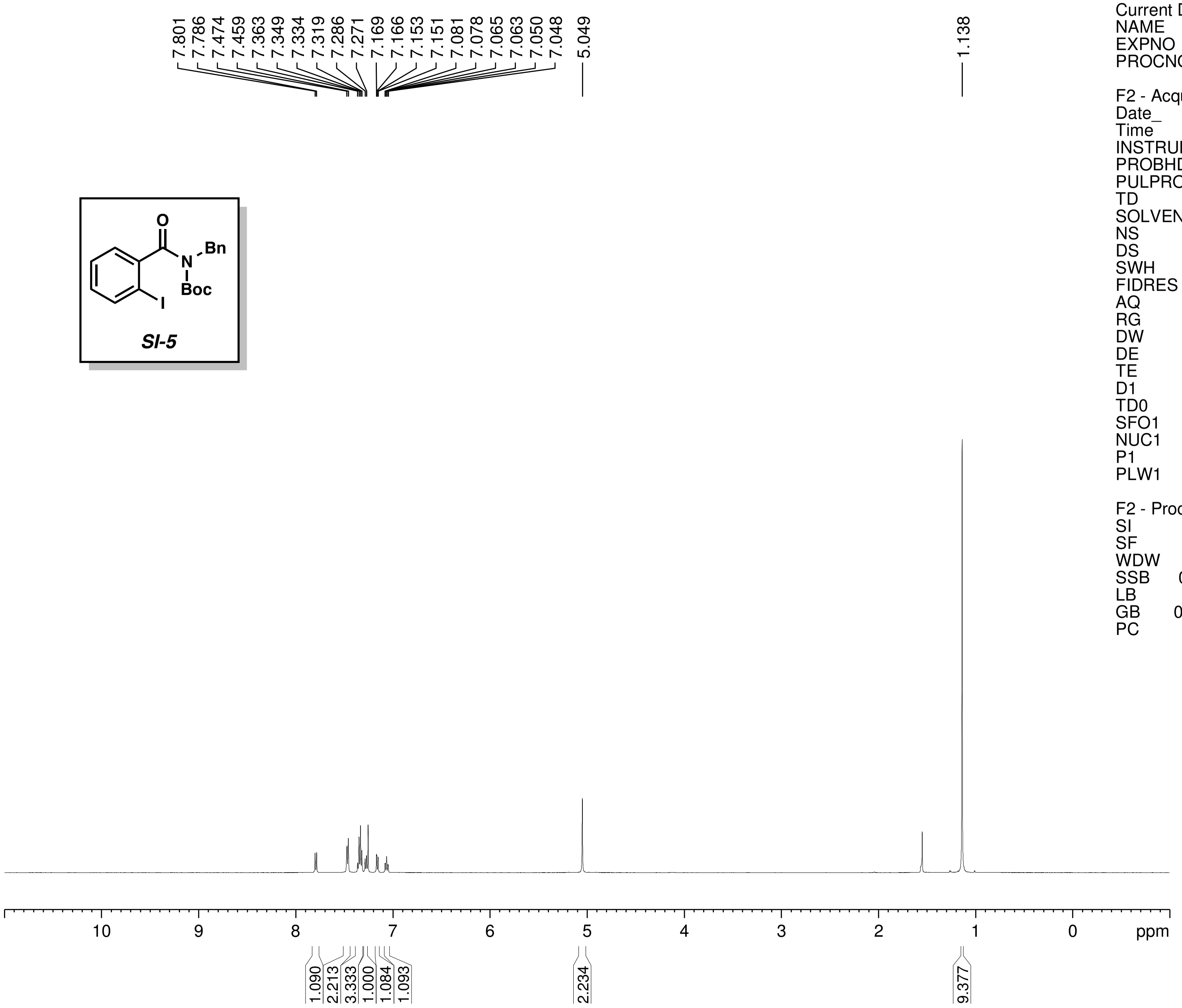

Current Data Parameters
NAME JM-5-073
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170217
Time 18.24 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.569
1.571
1.573
1.715
1.717
1.763
3.776
3.889
7.189
7.190
7.205
7.206
7.215
7.216
7.217
7.230
7.245
7.246
7.247
7.388
7.391
7.403
7.406
7.418
7.421
7.826
7.828
7.841
7.844
3.058
3.072
3.010
2.055
3.104
1.010
1.018
1.023
1.000
ppm
0
1
2
3
4
5
6
7
8
9
10
O
OMe
Me
Me
Me
SI-3



5



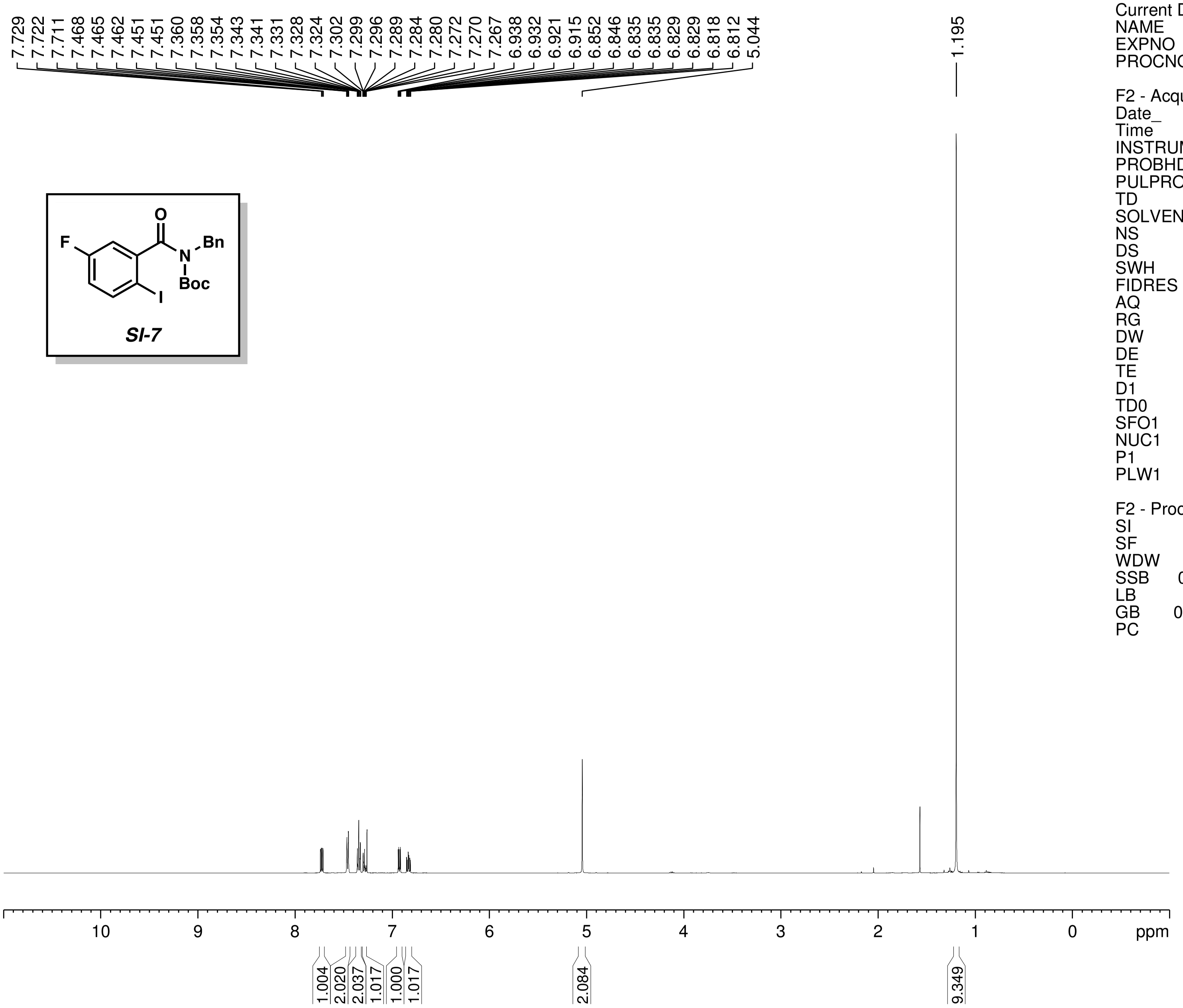
```
Current Data Parameters
NAME          JM-5-022
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_        20170112
Time            10.47
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         3.2767999 sec
RG                181
DW             50.000 usec
DE              6.00 usec
TE              297.1 K
D1         2.00000000 sec
TD0                 1

======== CHANNEL f1 ======
NUC1               1H
P1              13.30 usec
PL1      0 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
```



Current Data Parameters
NAME JM-4-296
EXPNO 100
PROCNO 1

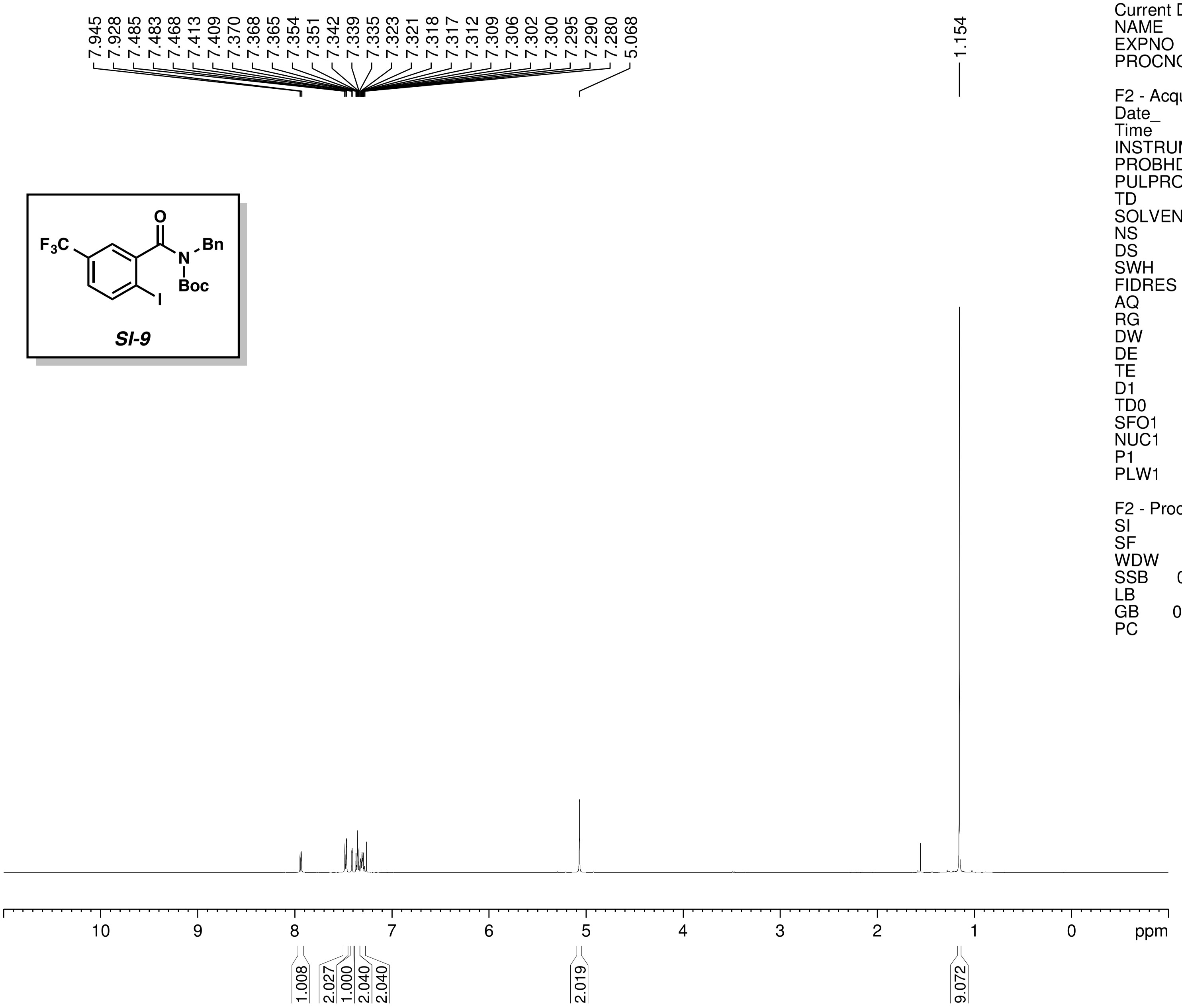
F2 - Acquisition Parameters
Date_ 20170115
Time 17.12 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



SI-7

```
Current Data Parameters
NAME          JM-5-054
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20170217
Time           18.30 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   0
SWH        10000.000 Hz
FIDRES      0.305176 Hz
AQ        3.2767999 sec
RG               23.34
DW          50.000 usec
DE          10.00 usec
TE             298.0 K
D1      2.00000000 sec
TD0                  1
SFO1   500.1330008 MHz
NUC1                1H
P1          10.00 usec
PLW1     13.50000000 W

F2 - Processing parameters
SI               65536
SF     500.1300121 MHz
WDW                 EM
SSB      0
LB             0.30 Hz
GB       0
PC                1.00
```

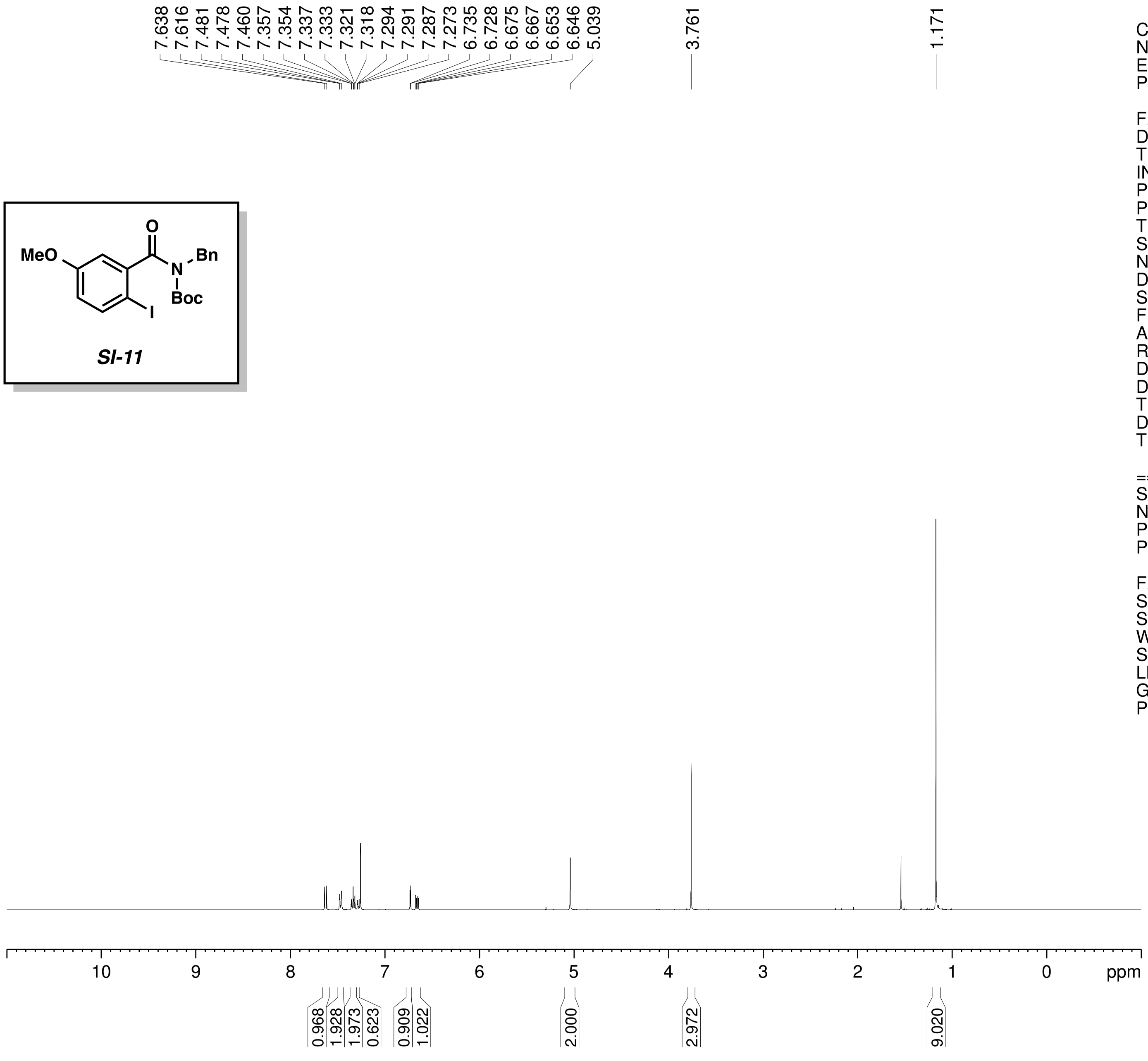


SI-9

Current Data Parameters
NAME JM-5-037
EXPNO 100
PROCNO 1

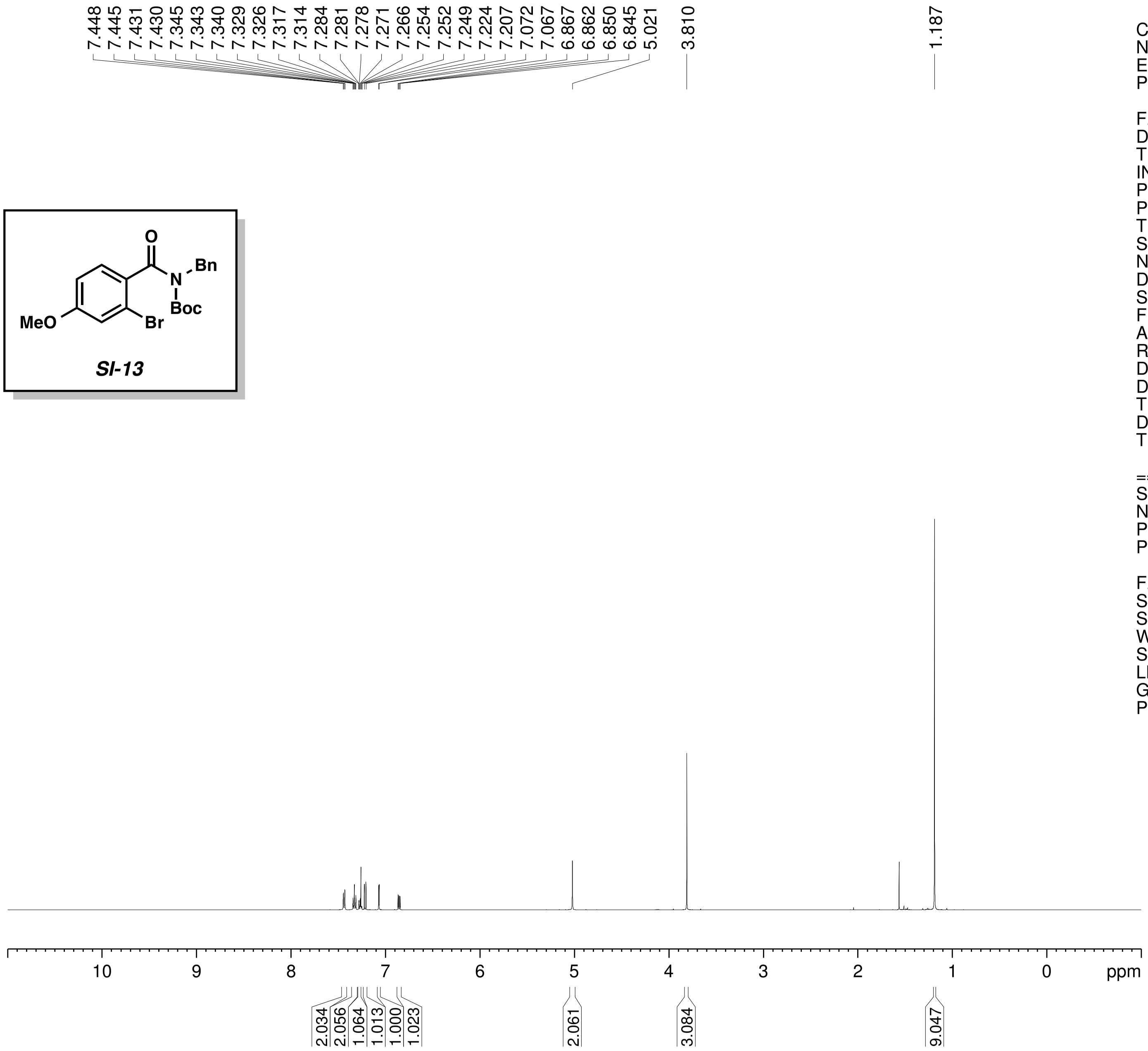
F2 - Acquisition Parameters
Date_ 20170130
Time 18.16 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



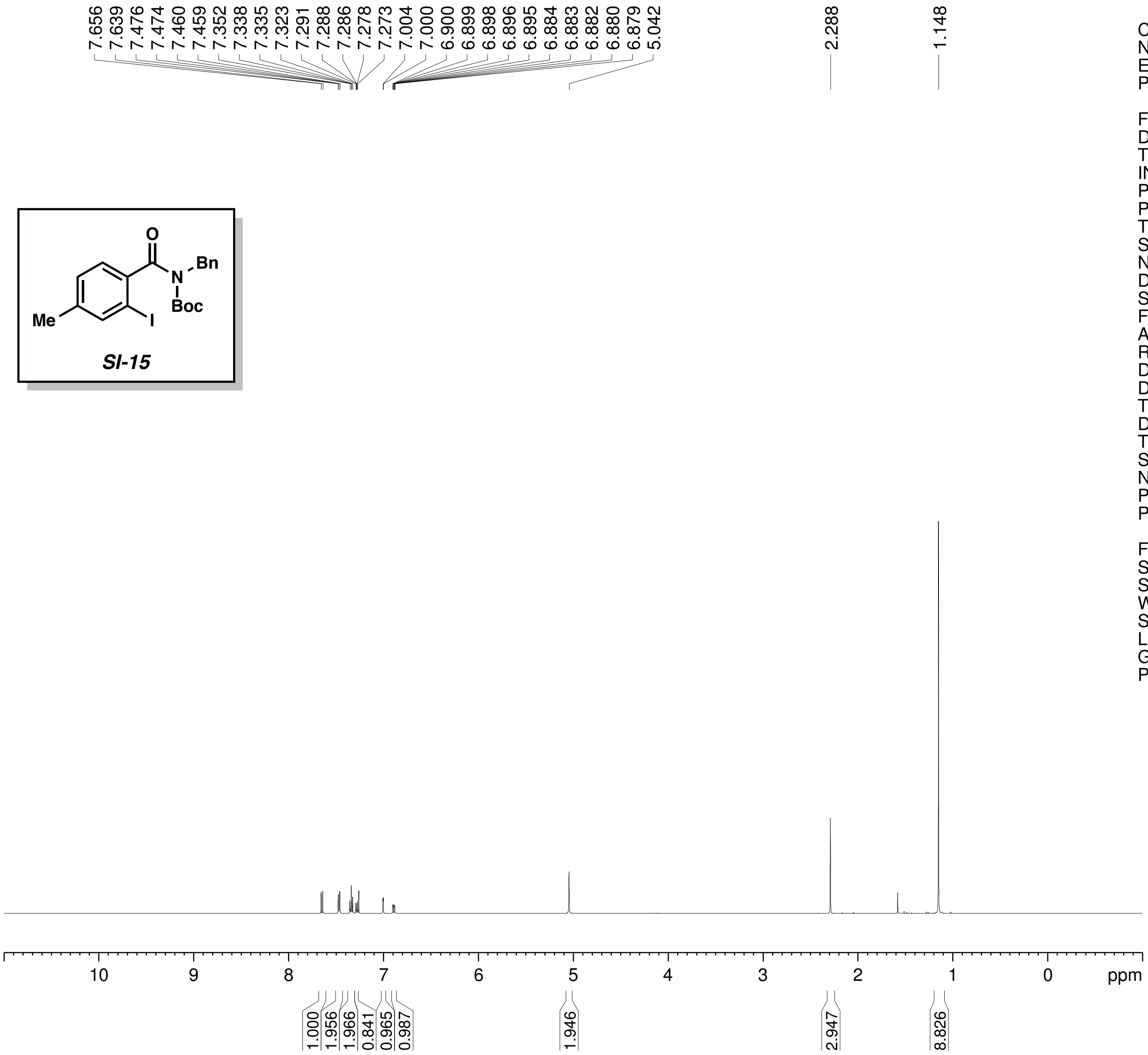
SI-11

```
Current Data Parameters
NAME          JM-5-033
EXPNO               90
PROCNO               1

F2 - Acquisition Parameters
Date_        20170129
Time            16.35
INSTRUM         av400
PROBHD   5 mm PABBO BB/
PULPROG          zg30
TD              52882
SOLVENT         CDCl3
NS                  8
DS                  0
SWH       8012.820 Hz
FIDRES     0.151523 Hz
AQ       3.2998369 sec
RG             155.85
DW         62.400 usec
DE           6.50 usec
TE            299.0 K
D1     2.00000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1    400.1324008 MHz
NUC1               1H
P1           15.00 usec
PLW1     13.00000000 W

F2 - Processing parameters
SI              65536
SF      400.1300183 MHz
WDW                EM
SSB      0
LB            0.30 Hz
GB     0
PC               1.00
```



SI-13

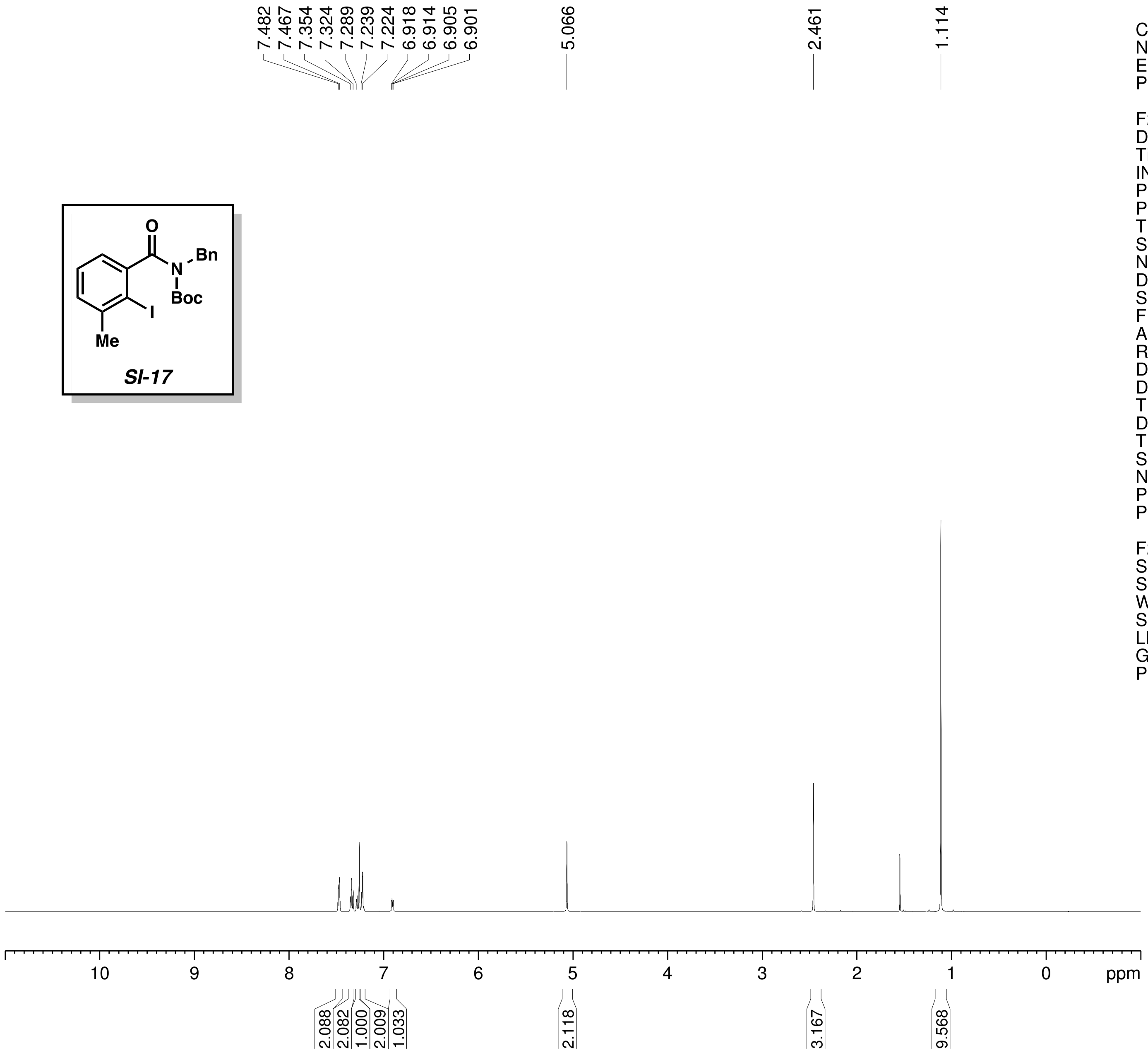
```
Current Data Parameters
NAME        JMM-7-68-1
EXPNO              1
PROCNO           999

F2 - Acquisition Parameters
Date_        20170207
Time            19.46
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 14
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ        3.2767999 sec
RG              12.14
DW            50.000 usec
DE             10.00 usec
TE              298.0 K
D1       2.00000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1     500.1340010 MHz
NUC1               1H
P1             10.00 usec
PLW1     13.50000000 W

F2 - Processing parameters
SI              65536
SF       500.1300123 MHz
WDW                EM
SSB      0
LB               0.30 Hz
GB       0
PC               1.00
```



SI-15

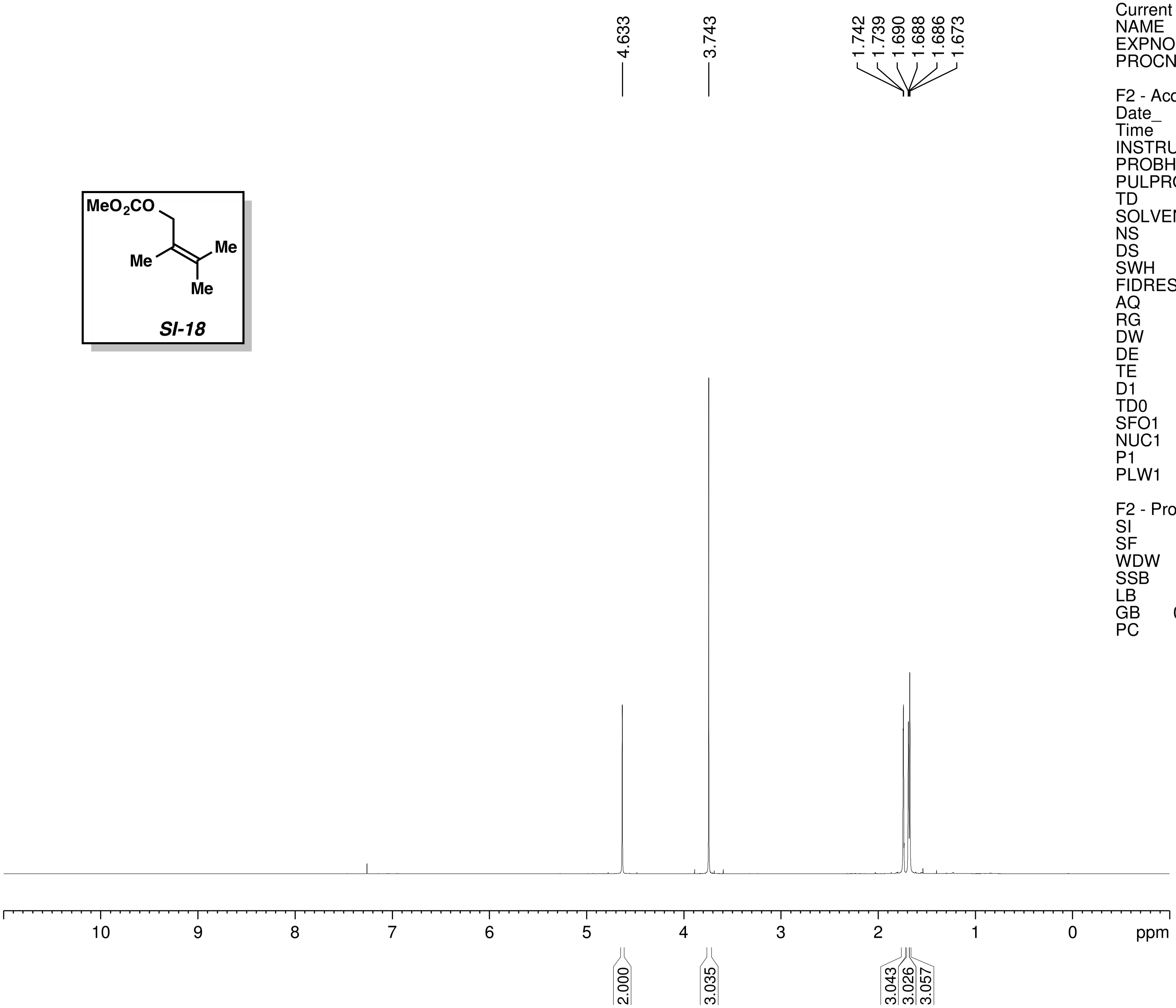
```
Current Data Parameters
NAME     sr-1-116.500.11
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_          20170210
Time             9.04 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH          10000.000 Hz
FIDRES        0.305176 Hz
AQ           3.2767999 sec
RG                12.14
DW               50.000 usec
DE                10.00 usec
TE                298.0 K
D1          2.00000000 sec
TD0                   1
SFO1       500.1330008 MHz
NUC1                 1H
P1                10.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI                65536
SF          500.1300122 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB      0
PC                 1.00
```

SI-17



```
Current Data Parameters
NAME          sr-1-088
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_        20170124
Time             9.10 h
INSTRUM         av500
PROBHD   Z119248_0002 (
PULPROG          zg30
TD             65536
SOLVENT         CDCl3
NS                  8
DS                  0
SWH        10000.000 Hz
FIDRES      0.305176 Hz
AQ         3.2767999 sec
RG             12.14
DW            50.000 usec
DE             10.00 usec
TE             298.0 K
D1        2.00000000 sec
TD0                 1
SFO1     500.1330008 MHz
NUC1               1H
P1             10.00 usec
PLW1     13.50000000 W

F2 - Processing parameters
SI             65536
SF       500.1300130 MHz
WDW                EM
SSB      0
LB              0.30 Hz
GB      0
PC              1.00
```

SI-18

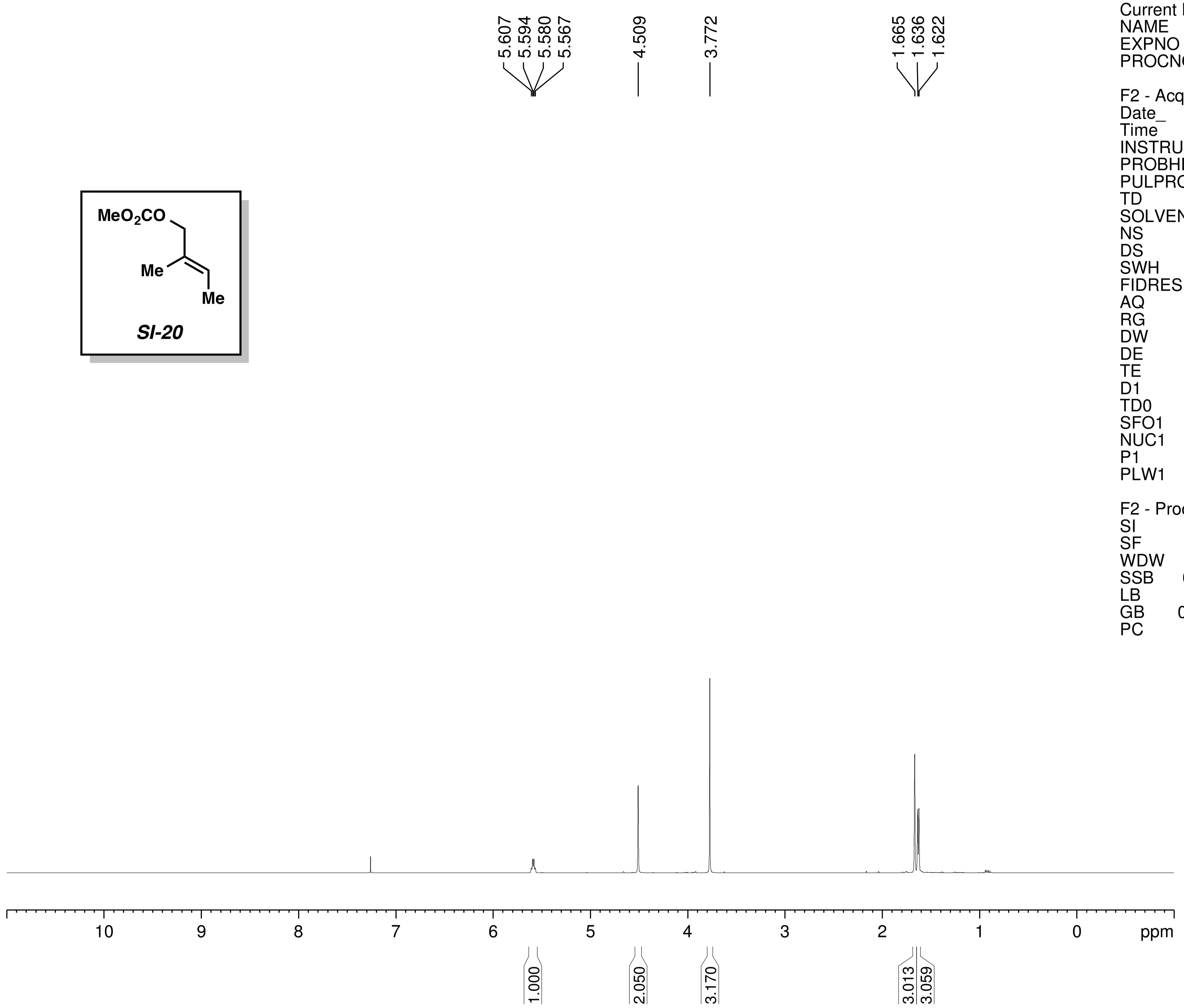


Current Data Parameters
NAME sr-1-035
EXPNO 1
PROCNO 1

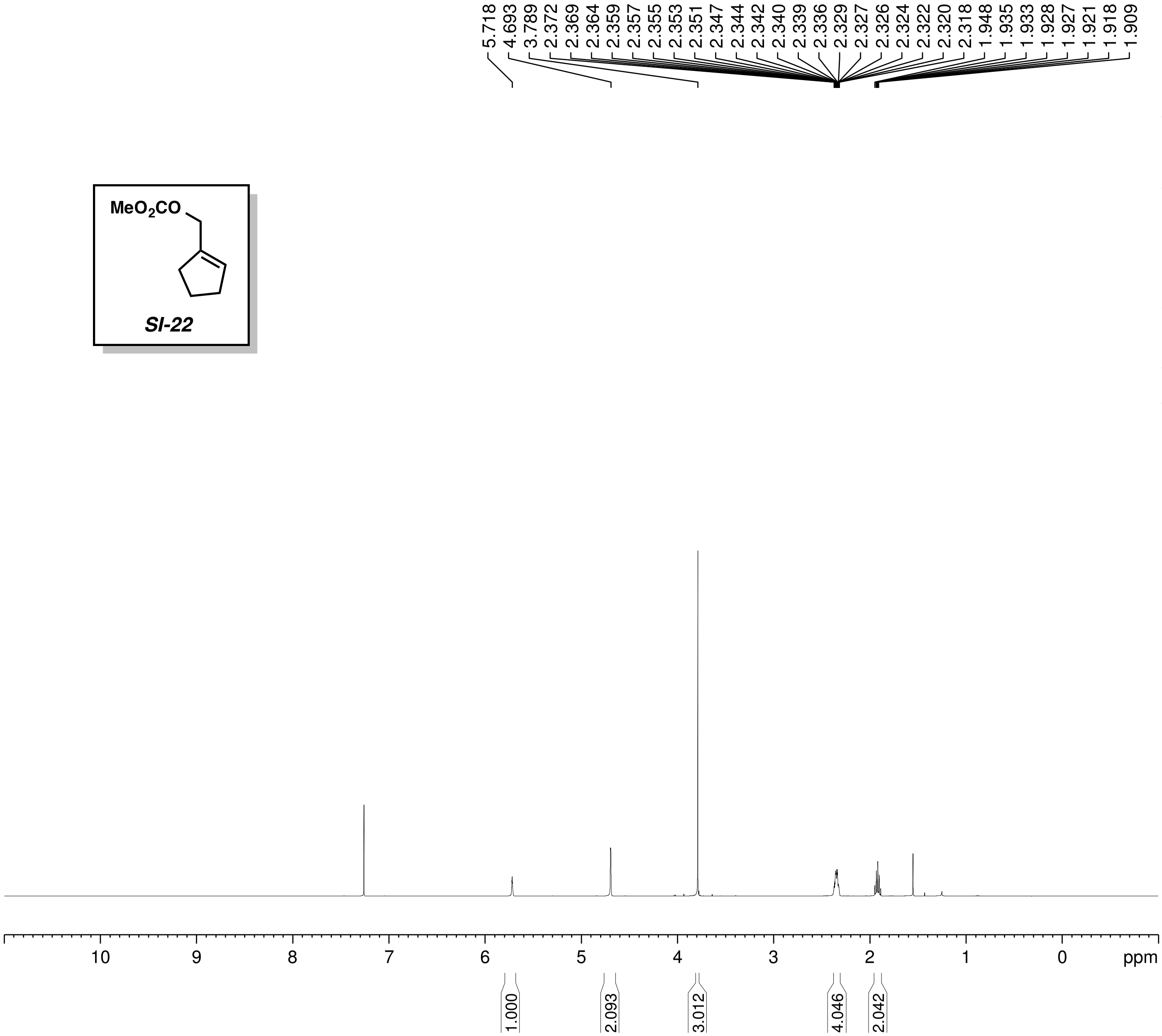
F2 - Acquisition Parameters
Date_ 20170109
Time 17.47 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 7.89
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

SI-20



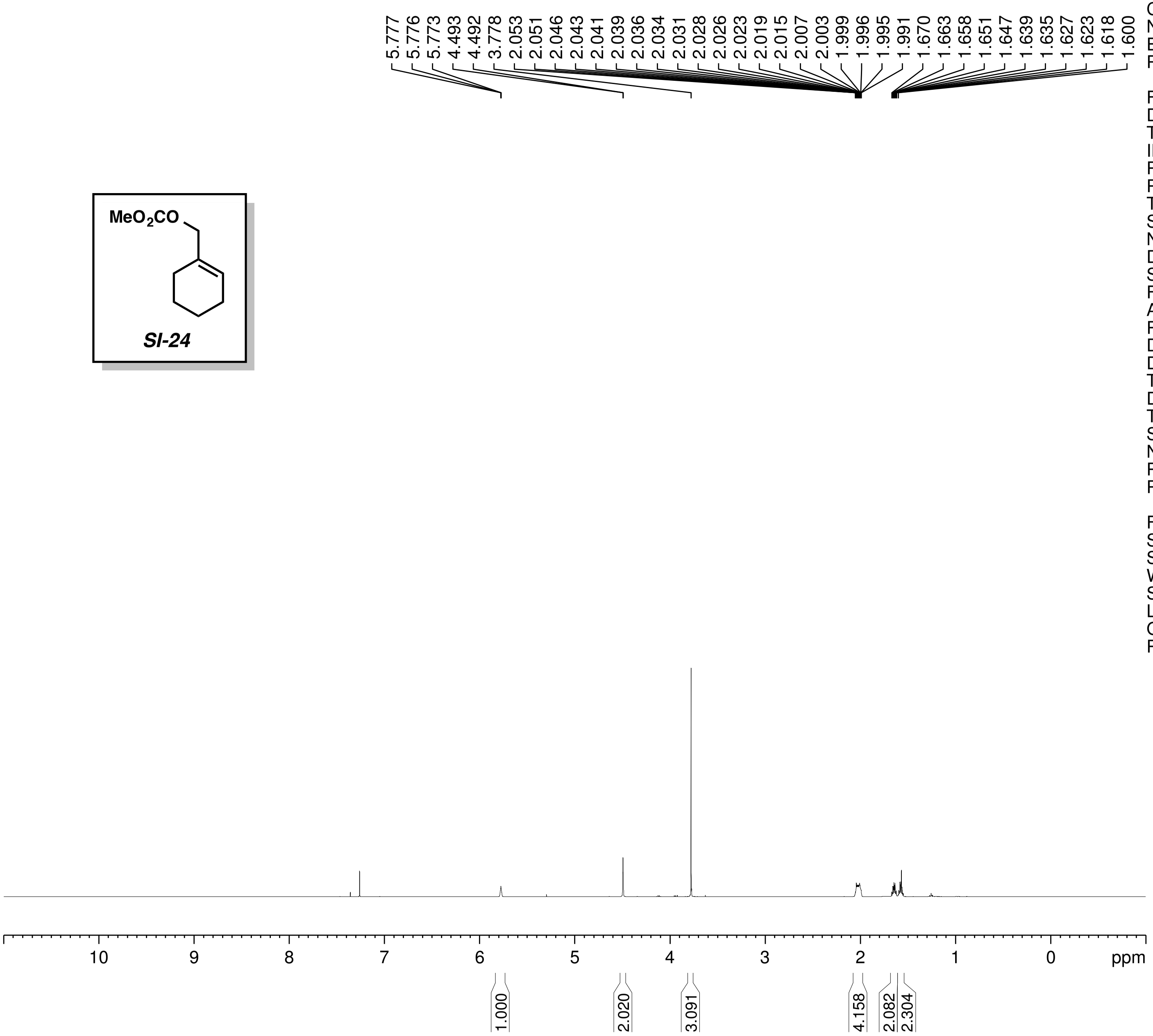
```
Current Data Parameters
NAME          sr-1-065
EXPNO              100
PROCNO               1

F2 - Acquisition Parameters
Date_          20170109
Time            17.54 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG            zg30
TD              65536
SOLVENT           CDCl3
NS                  8
DS                  0
SWH          10000.000 Hz
FIDRES        0.305176 Hz
AQ           3.2767999 sec
RG               19.06
DW              50.000 usec
DE               10.00 usec
TE               298.0 K
D1          2.00000000 sec
TD0                 1
SFO1       500.1330008 MHz
NUC1                1H
P1               10.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI              65536
SF         500.1300121 MHz
WDW                EM
SSB      0
LB                0.30 Hz
GB      0
PC                1.00
```



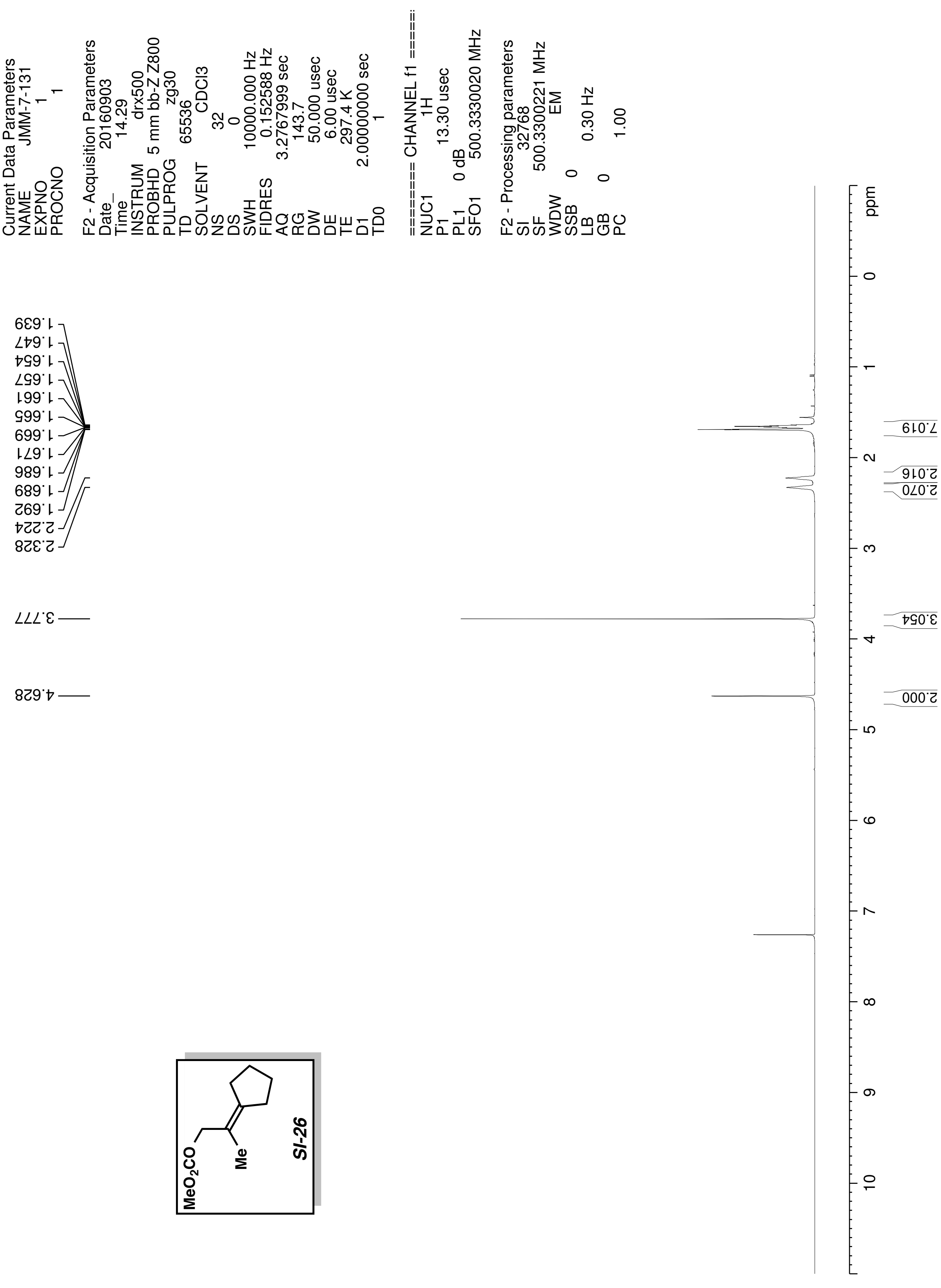
SI-22

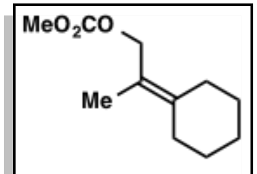
```
Current Data Parameters
NAME      JMM-7-216char
EXPNO              1
PROCNO           999

F2 - Acquisition Parameters
Date_        20170108
Time            10.07
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD             65536
SOLVENT         CDCl3
NS                26
DS                 0
SWH       10000.000 Hz
FIDRES     0.152588 Hz
AQ       3.2767999 sec
RG              21.37
DW         50.000 usec
DE          10.00 usec
TE            298.0 K
D1      2.00000000 sec
TD0                1

======== CHANNEL f1 =====
SFO1     500.1340010 MHz
NUC1               1H
P1          10.00 usec
PLW1     13.50000000 W

F2 - Processing parameters
SI             65536
SF       500.1300124 MHz
WDW               EM
SSB     0
LB             0.30 Hz
GB      0
PC              1.00
```

SI-24

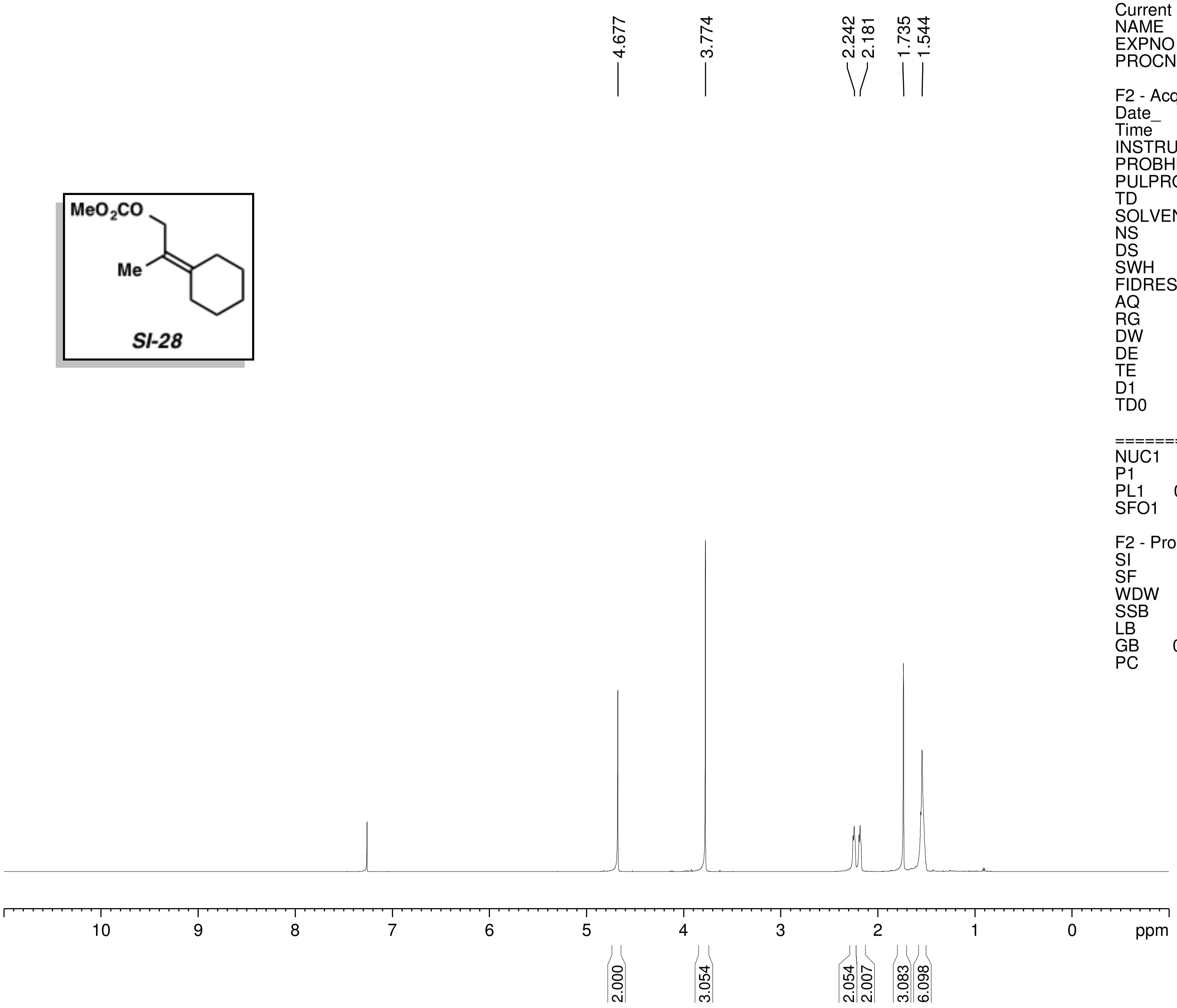


```
Current Data Parameters
NAME          JM-4-293
EXPNO              100
PROCNO               1

F2 - Acquisition Parameters
Date_         20161219
Time            18.20 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   0
SWH        10000.000 Hz
FIDRES      0.305176 Hz
AQ        3.2767999 sec
RG               12.14
DW          50.000 usec
DE           10.00 usec
TE              298.0 K
D1       2.00000000 sec
TD0                  1
SFO1    500.1330008 MHz
NUC1                1H
P1           10.00 usec
PLW1     13.50000000 W

F2 - Processing parameters
SI               65536
SF      500.1300121 MHz
WDW                 EM
SSB      0
LB             0.30 Hz
GB       0
PC                1.00
```


SI-26
Current Data Parameters
NAME JMM-7-131
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160903
Time 14.29
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 297.4 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300221 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.639
1.647
1.654
1.657
1.661
1.665
1.669
1.671
1.686
1.689
1.692
2.224
2.328
3.777
4.628
7.019
2.016
2.070
3.054
2.000
ppm
0
1
2
3
4
5
6
7
8
9
10
MeO$_2$CO
Me

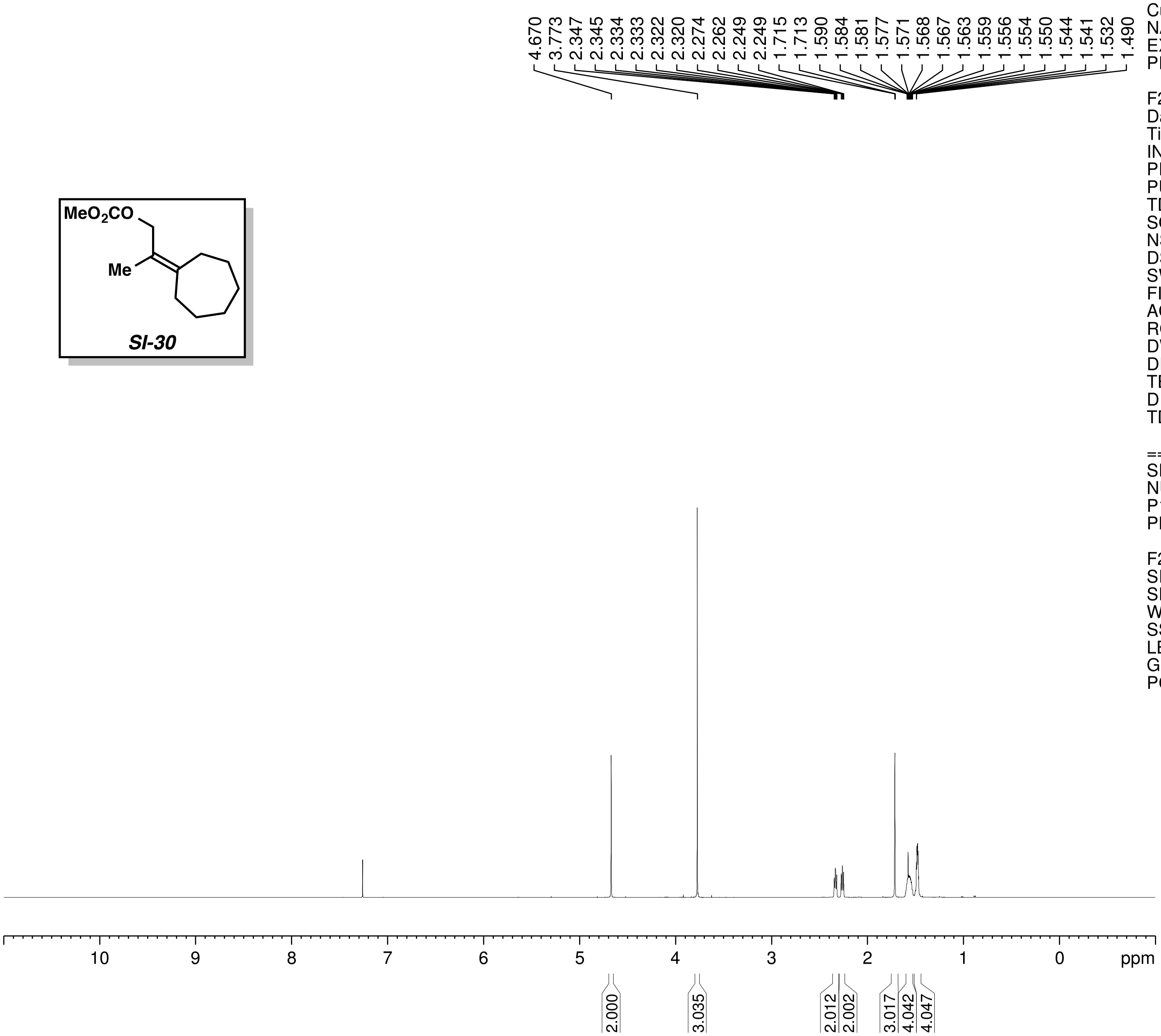


SI-28



```
Current Data Parameters
NAME        JMM-7-132
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_        20160903
Time            14.36
INSTRUM        drx500
PROBHD   5 mm bb-Z Z800
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 32
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ         3.2767999 sec
RG                128
DW            50.000 usec
DE             6.00 usec
TE              297.4 K
D1       2.00000000 sec
TD0                 1

======== CHANNEL f1 =====
NUC1               1H
P1             13.30 usec
PL1     0 dB
SFO1      500.3330020 MHz

F2 - Processing parameters
SI              32768
SF       500.3300217 MHz
WDW                EM
SSB     0
LB              0.30 Hz
GB      0
PC               1.00
```

SI-30

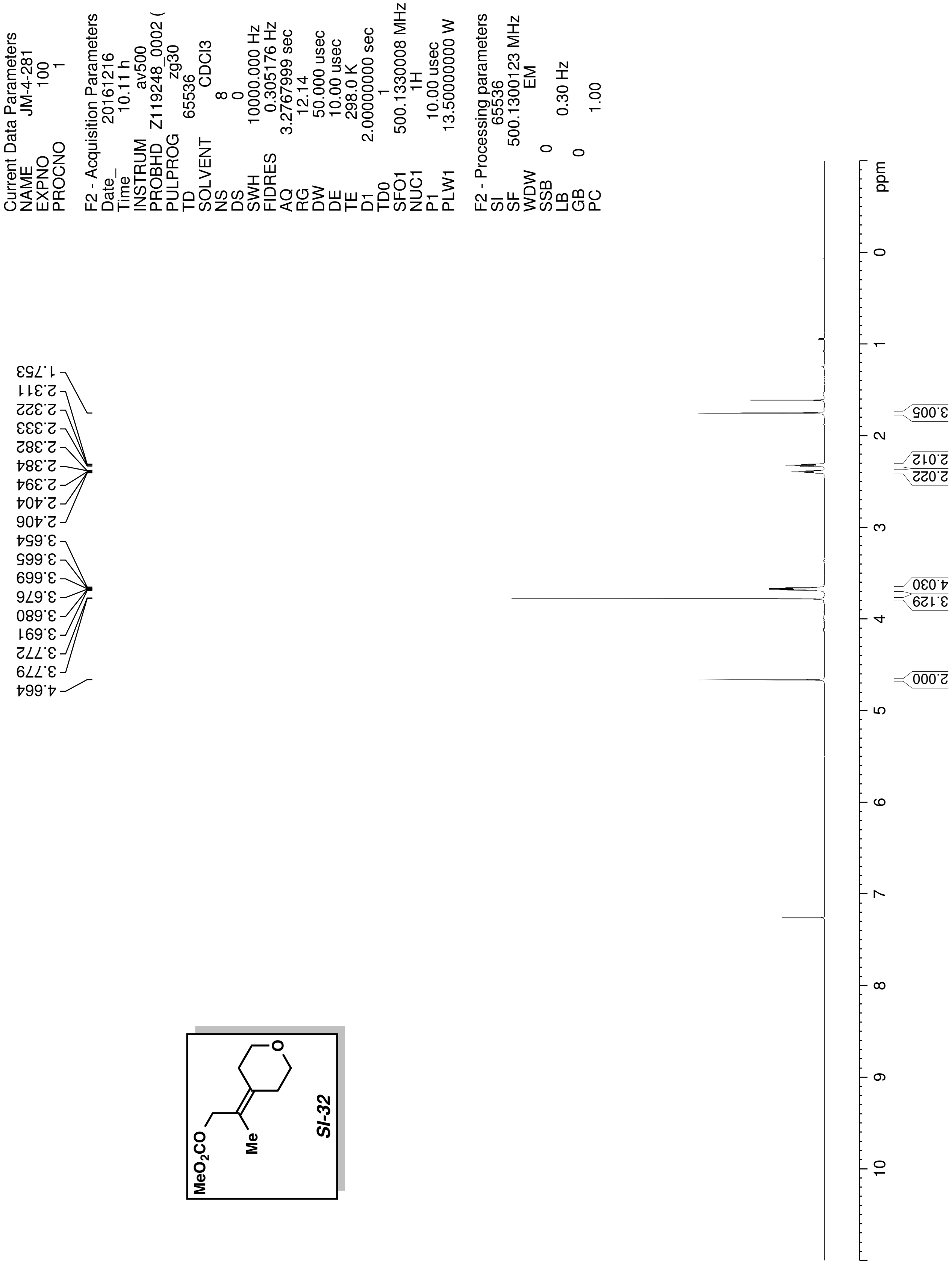


Current Data Parameters
NAME JMM-7-226
EXPNO 1
PROCNO 999

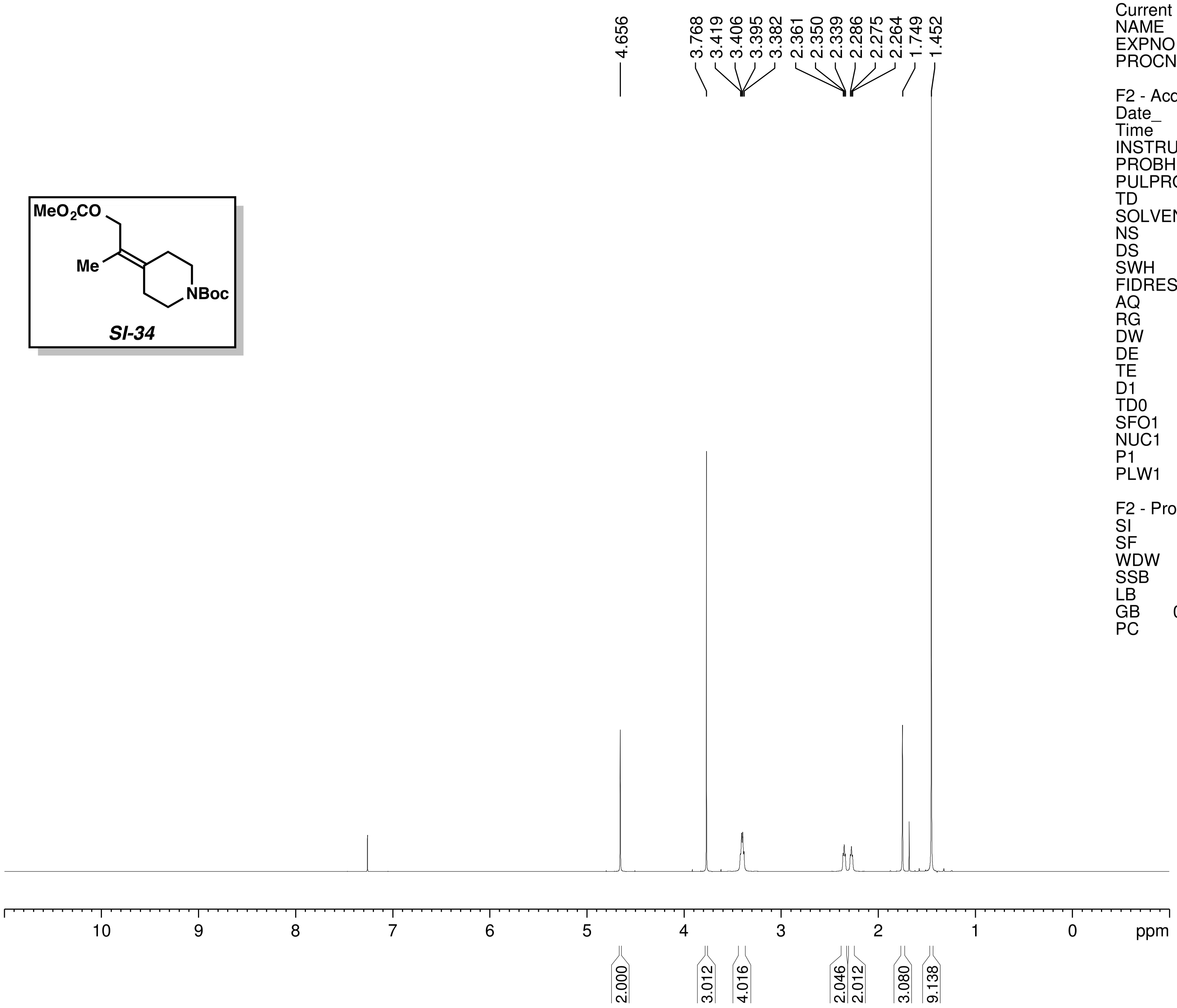
F2 - Acquisition Parameters
Date_ 20170116
Time 10.44
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ======
SFO1 500.1340010 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


Current Data Parameters
NAME JM-4-281
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161216
Time 10.11 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.753
2.311
2.322
2.333
2.382
2.384
2.394
2.404
2.406
3.654
3.665
3.669
3.676
3.680
3.691
3.772
3.779
4.664
3.005
2.012
2.022
4.030
3.129
2.000
ppm
0
1
2
3
4
5
6
7
8
9
10
MeO$_2$CO
Me
O
SI-32

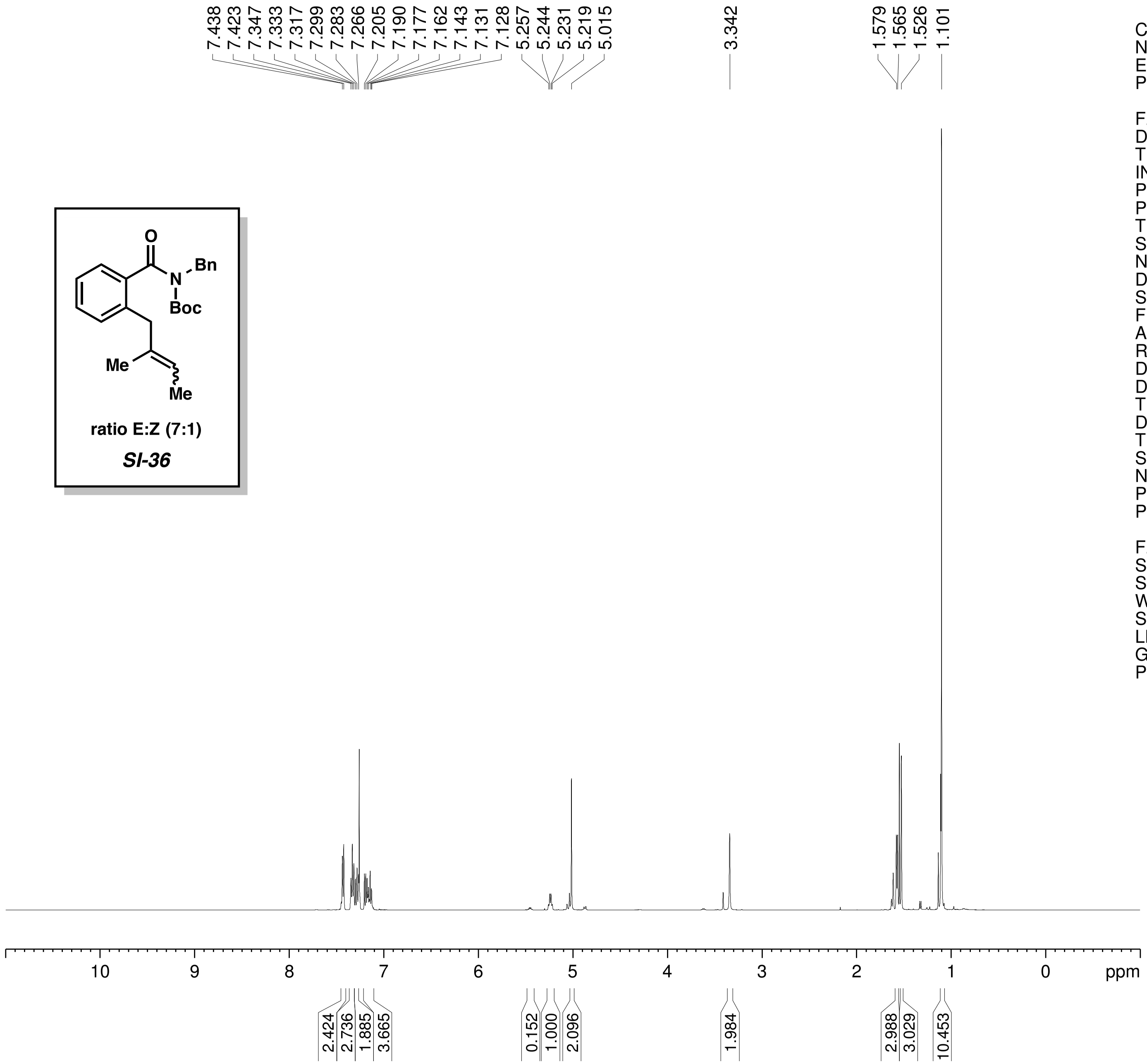
SI-34



```
Current Data Parameters
NAME          JM-4-287
EXPNO              100
PROCNO               1

F2 - Acquisition Parameters
Date_         20161216
Time            10.21 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   0
SWH        10000.000 Hz
FIDRES      0.305176 Hz
AQ         3.2767999 sec
RG               21.37
DW            50.000 usec
DE            10.00 usec
TE             298.0 K
D1       2.00000000 sec
TD0                  1
SFO1    500.1330008 MHz
NUC1                1H
P1            10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI               65536
SF      500.1300121 MHz
WDW                 EM
SSB      0
LB             0.30 Hz
GB       0
PC                1.00
```

ratio E:Z (7:1)

SI-36



Current Data Parameters

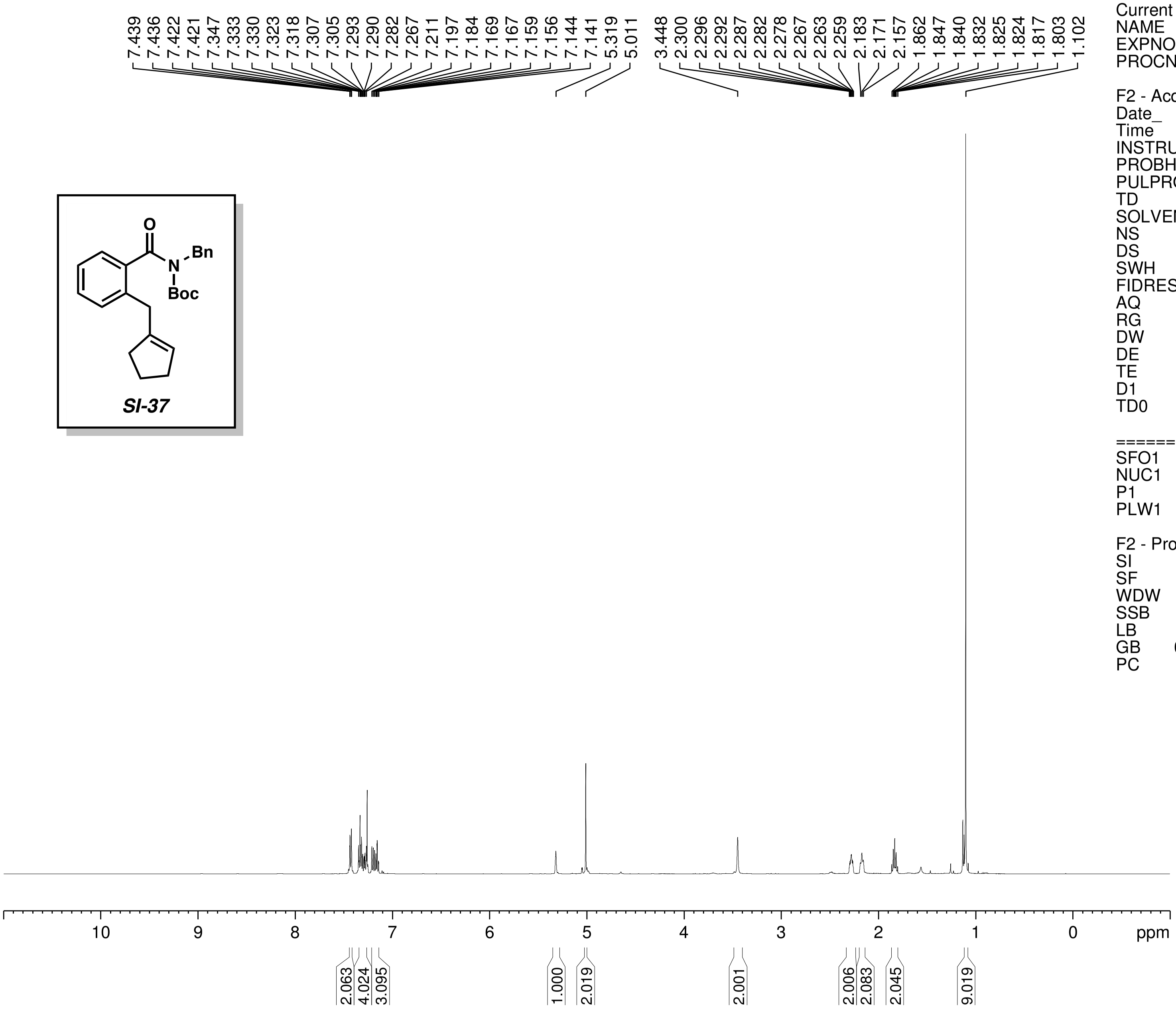
NAME	sr-1-099.500
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20170130
Time	9.03 h
INSTRUM	av500
PROBHD	Z119248_0002 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.305176 Hz
AQ	3.2767999 sec
RG	12.14
DW	50.000 usec
DE	10.00 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
SFO1	500.1330008 MHz
NUC1	1H
P1	10.00 usec
PLW1	13.50000000 W

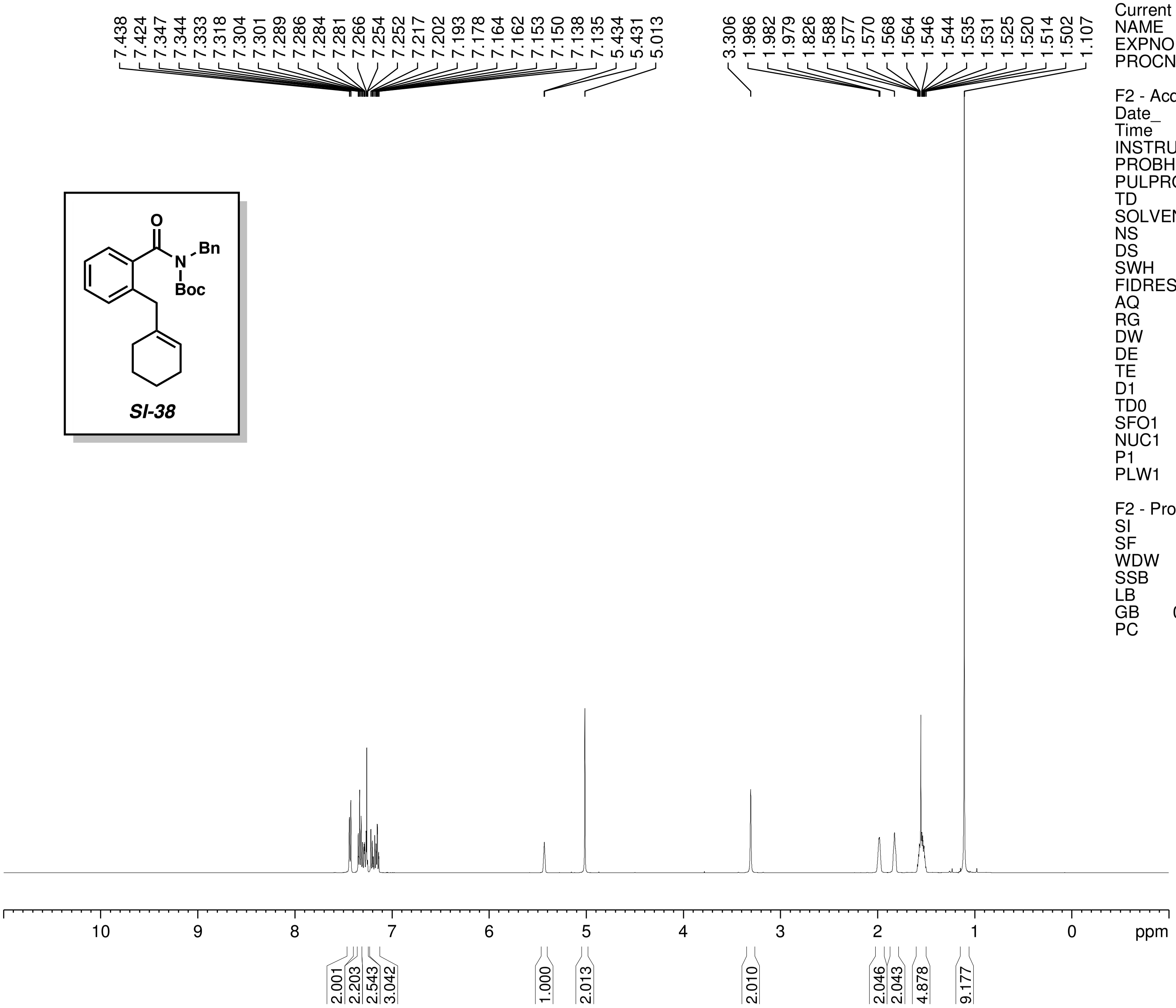
F2 - Processing parameters

SI	65536
SF	500.1300124 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



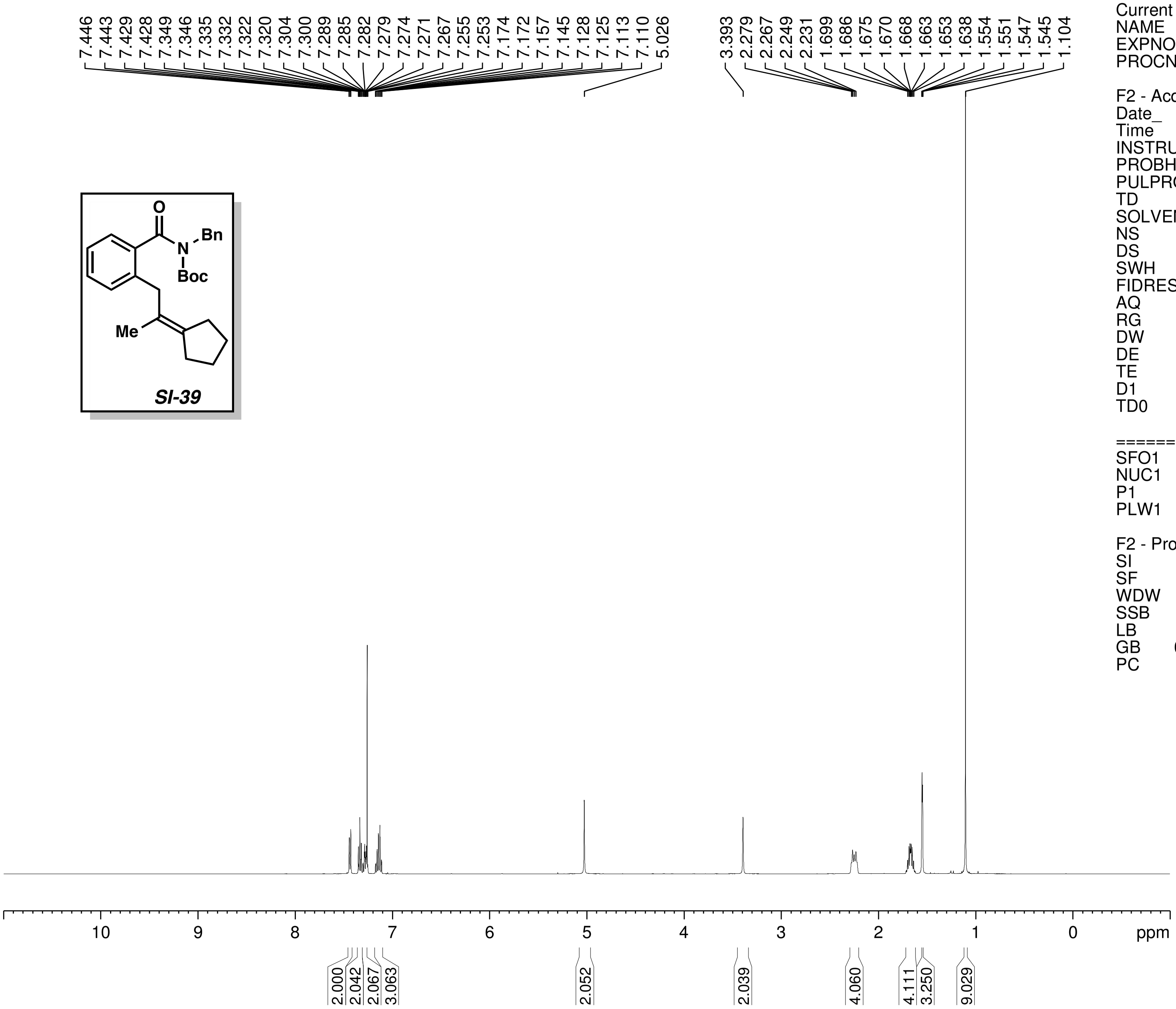
SI-37

```
Current Data Parameters
NAME     JMM-7-221-char
EXPNO              1
PROCNO           999

F2 - Acquisition Parameters
Date_        20170131
Time            10.10
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD             65536
SOLVENT         CDCl3
NS                30
DS                 0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ         3.2767999 sec
RG             12.14
DW            50.000 usec
DE            10.00 usec
TE            298.0 K
D1        2.00000000 sec
TD0                1

======== CHANNEL f1 =====
SFO1      500.1340010 MHz
NUC1               1H
P1             10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI             65536
SF        500.1300121 MHz
WDW               EM
SSB      0
LB              0.30 Hz
GB     0
PC              1.00
```

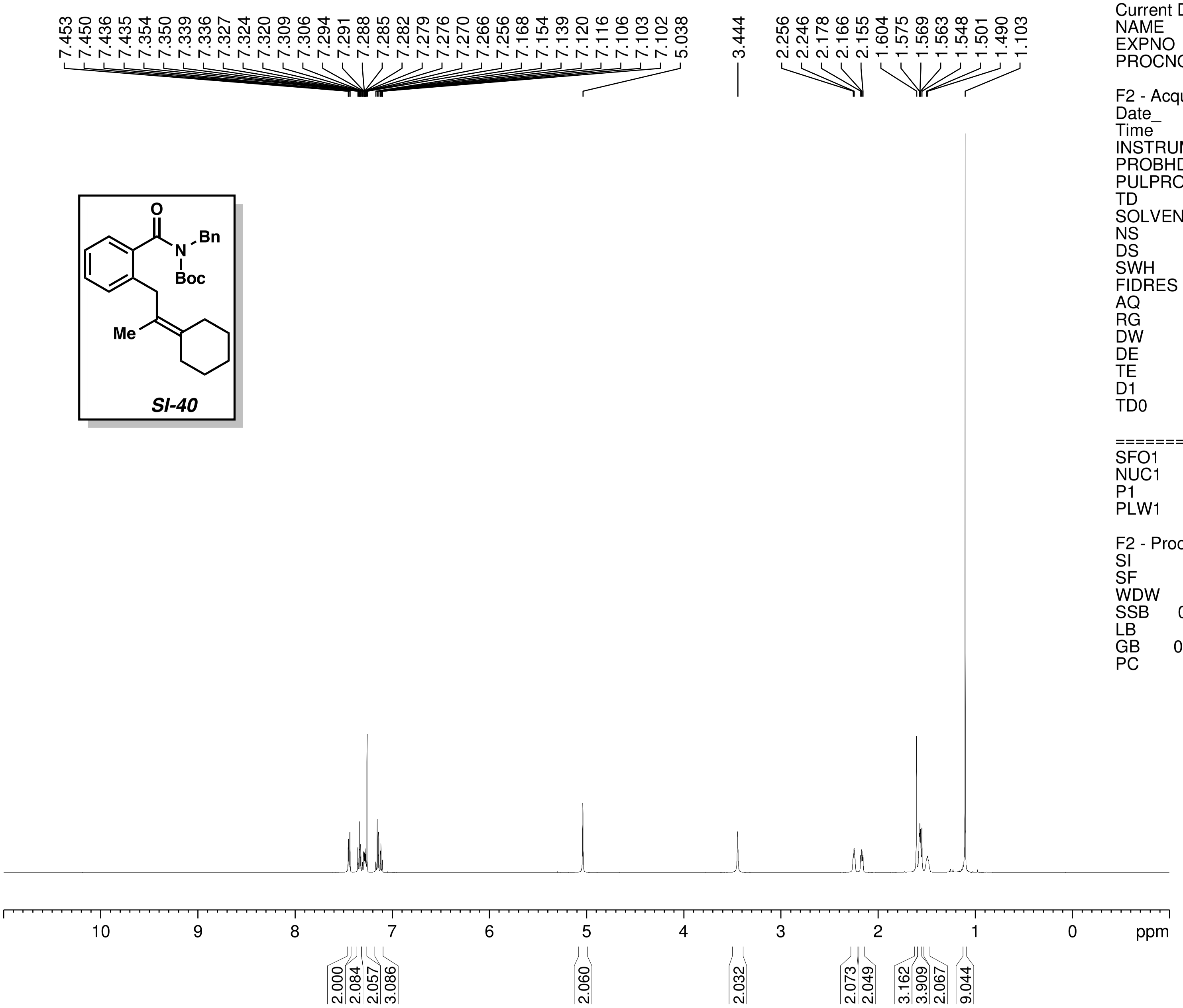


SI-38

```
Current Data Parameters
NAME          JM-4-295
EXPNO              100
PROCNO               1

F2 - Acquisition Parameters
Date_         20161218
Time            10.25 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD              65536
SOLVENT          CDCl3
NS                   8
DS                   0
SWH         10000.000 Hz
FIDRES       0.305176 Hz
AQ          3.2767999 sec
RG              12.14
DW             50.000 usec
DE             10.00 usec
TE             298.0 K
D1         2.00000000 sec
TD0                 1
SFO1      500.1330008 MHz
NUC1               1H
P1             10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI              65536
SF        500.1300121 MHz
WDW                EM
SSB      0
LB               0.30 Hz
GB       0
PC               1.00
```

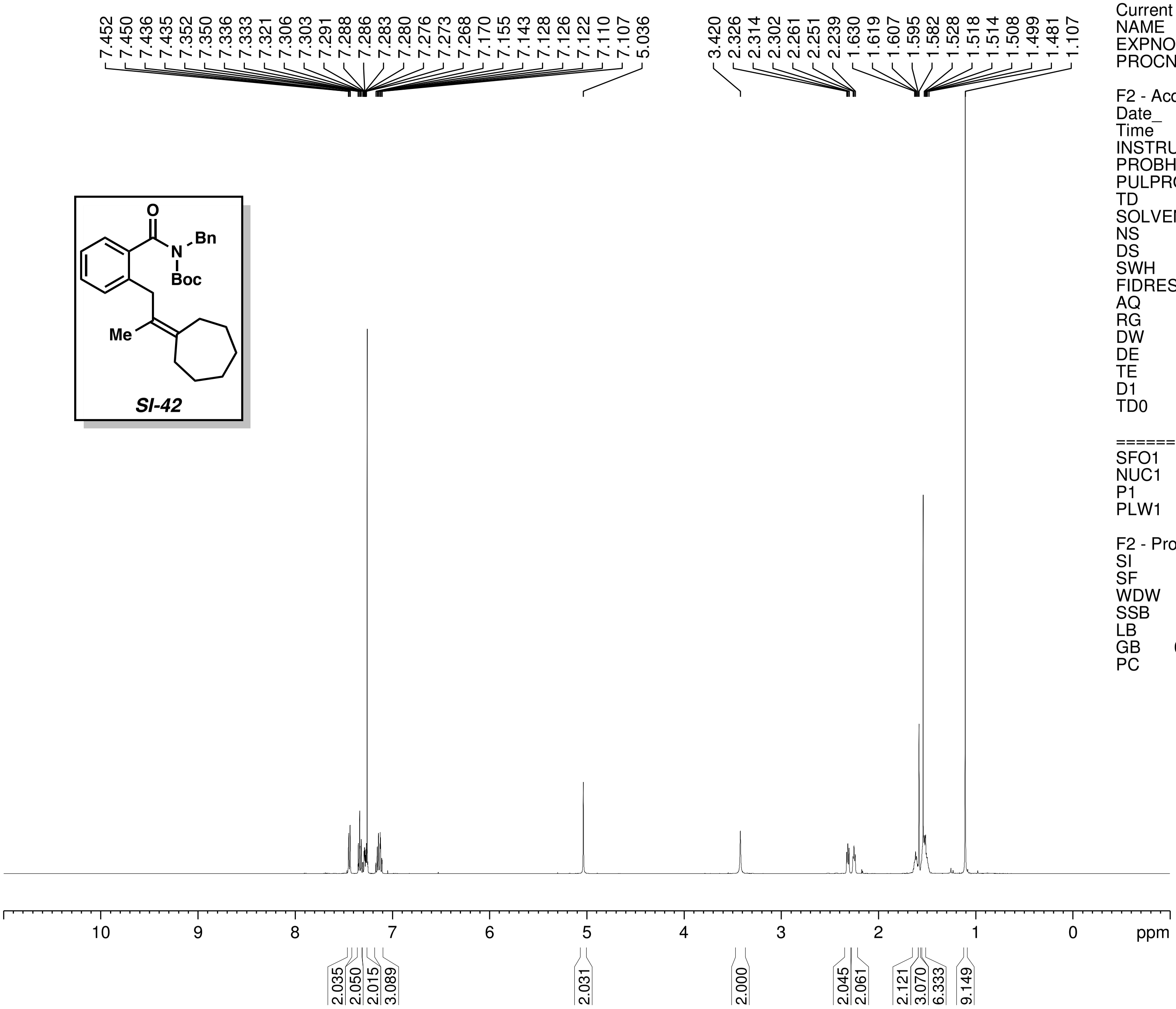


```
Current Data Parameters
NAME       JMM-7-134(c)
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20160907
Time             9.33
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 12
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ       3.2767999 sec
RG              12.14
DW            50.000 usec
DE             10.00 usec
TE              298.0 K
D1       2.00000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1      500.1340010 MHz
NUC1               1H
P1              10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI              65536
SF        500.1300123 MHz
WDW                EM
SSB     0
LB              0.30 Hz
GB      0
PC               1.00
```



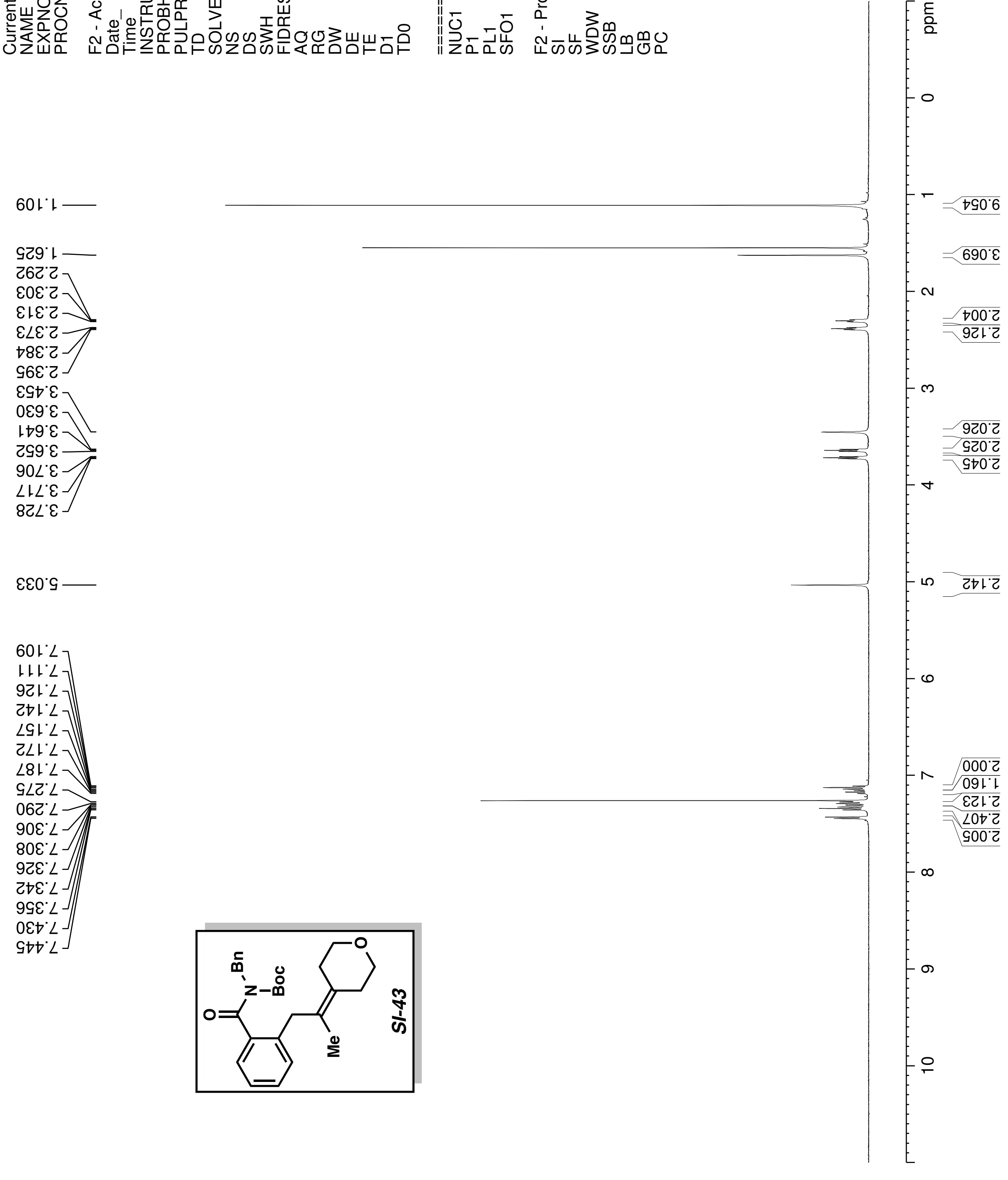
SI-40

```
Current Data Parameters
NAME        JMM-7-135(c)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_        20160907
Time             9.42
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 21
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ         3.2767999 sec
RG              26.59
DW             50.000 usec
DE              10.00 usec
TE              298.0 K
D1         2.00000000 sec
TD0                 1

======== CHANNEL f1 ======
SFO1       500.1340010 MHz
NUC1                1H
P1               10.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI              65536
SF         500.1300124 MHz
WDW                EM
SSB     0
LB               0.30 Hz
GB      0
PC               1.00
```



SI-42

```
Current Data Parameters
NAME     JMM-7-223-char
EXPNO              1
PROCNO           999

F2 - Acquisition Parameters
Date_        20170117
Time            21.03
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                  8
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ        3.2767999 sec
RG              12.14
DW             50.000 usec
DE             10.00 usec
TE              298.0 K
D1       2.00000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1      500.1340010 MHz
NUC1               1H
P1              10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI              65536
SF       500.1300123 MHz
WDW                EM
SSB      0
LB              0.30 Hz
GB       0
PC              1.00
```



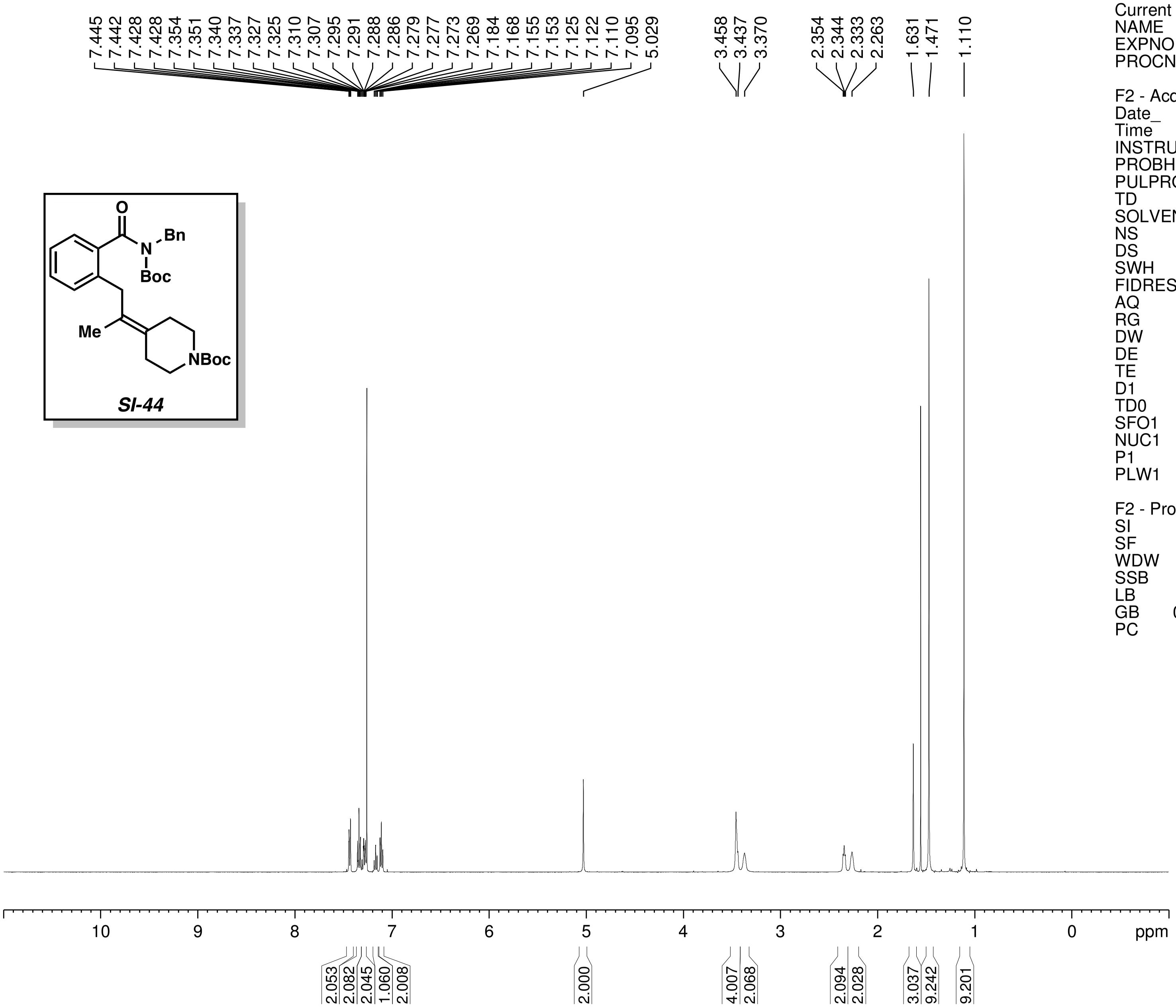
SI-43

Current Data Parameters
NAME JM-4-288
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161205
Time 9.53
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 3.2767999 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 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz

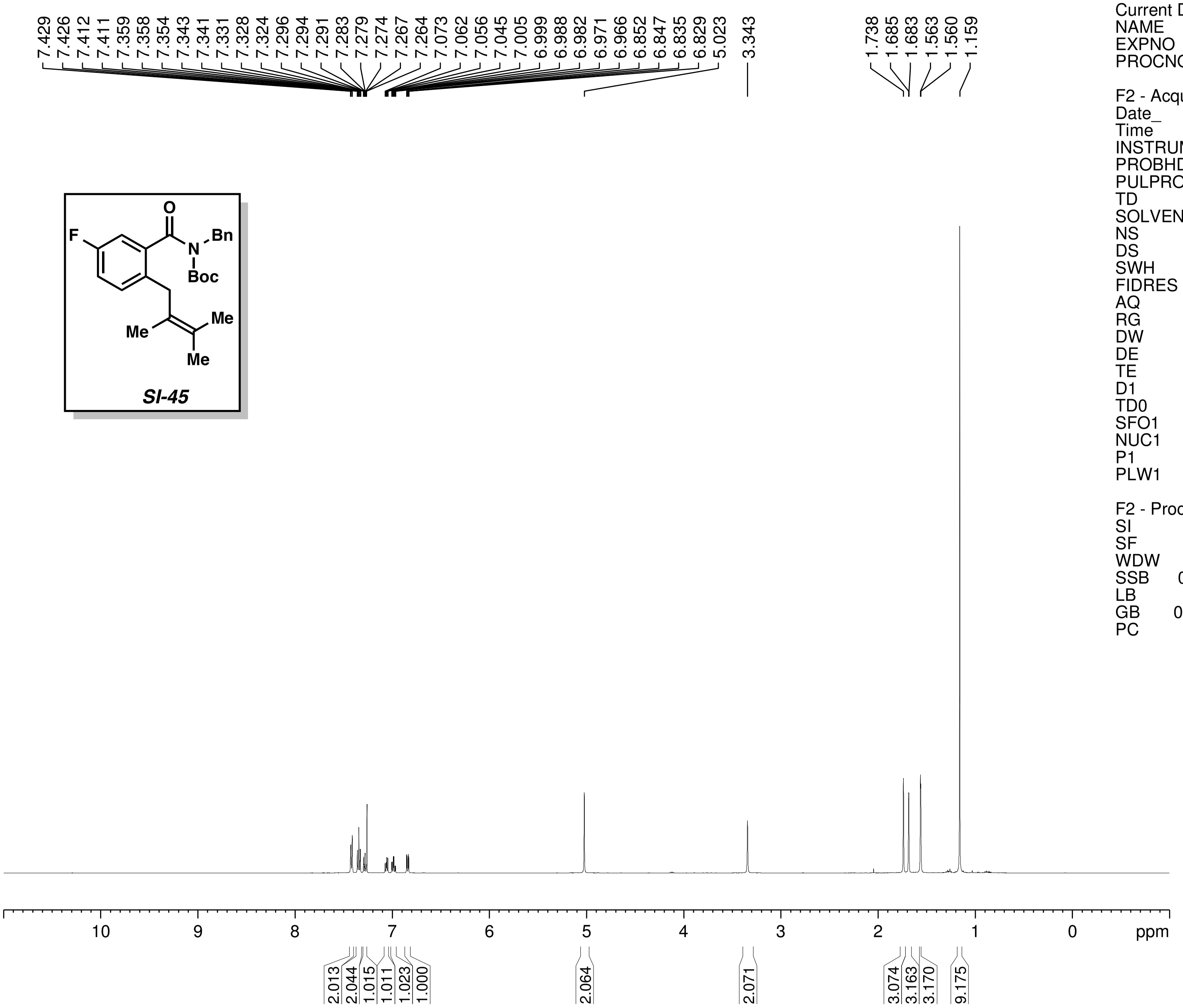
F2 - Processing parameters
SI 32768
SF 500.3300221 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME JM-4-290
EXPNO 100
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161218
Time 14.30 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

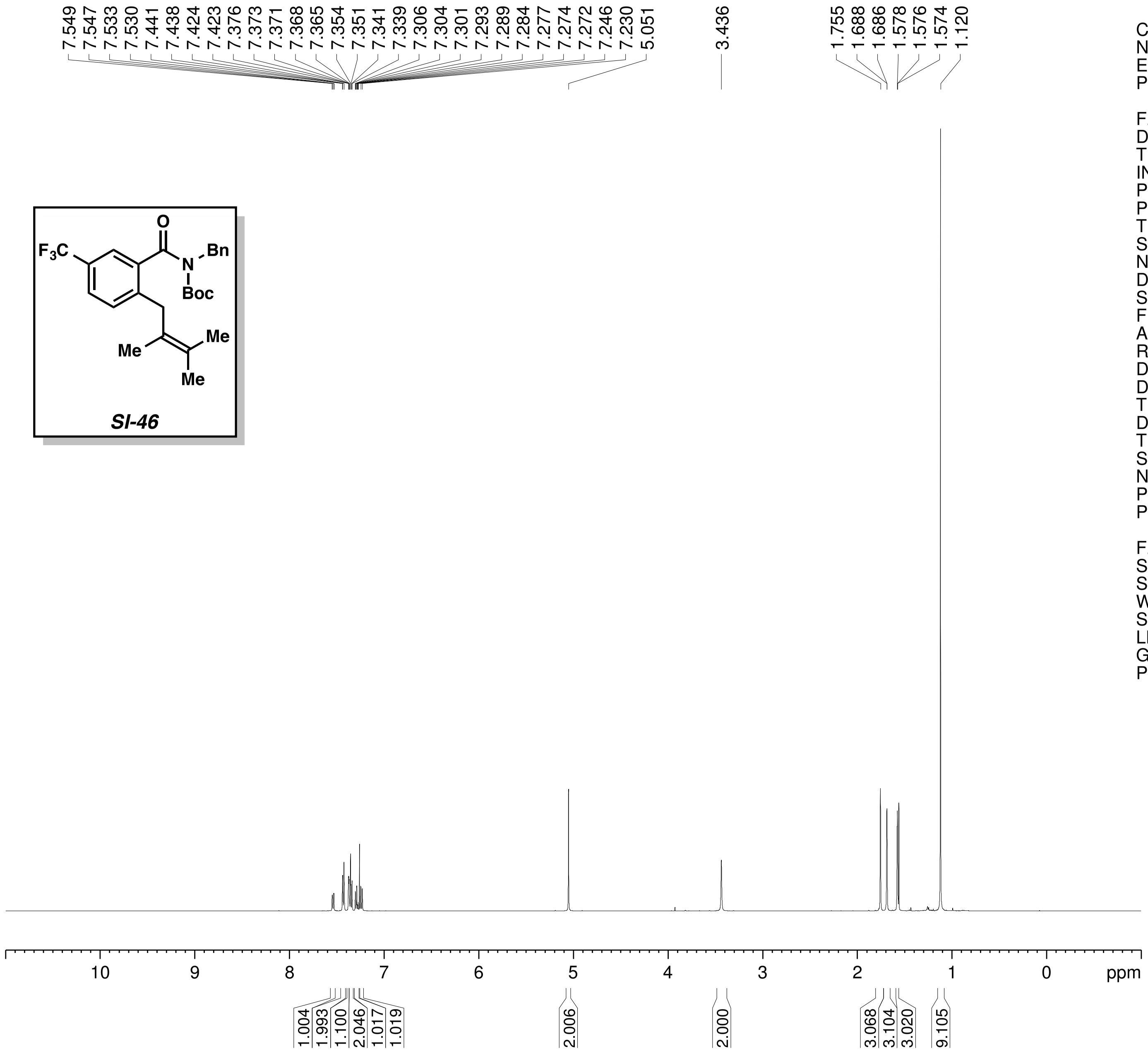


Current Data Parameters
NAME JM-5-067
EXPNO 100
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170217
Time 18.03 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

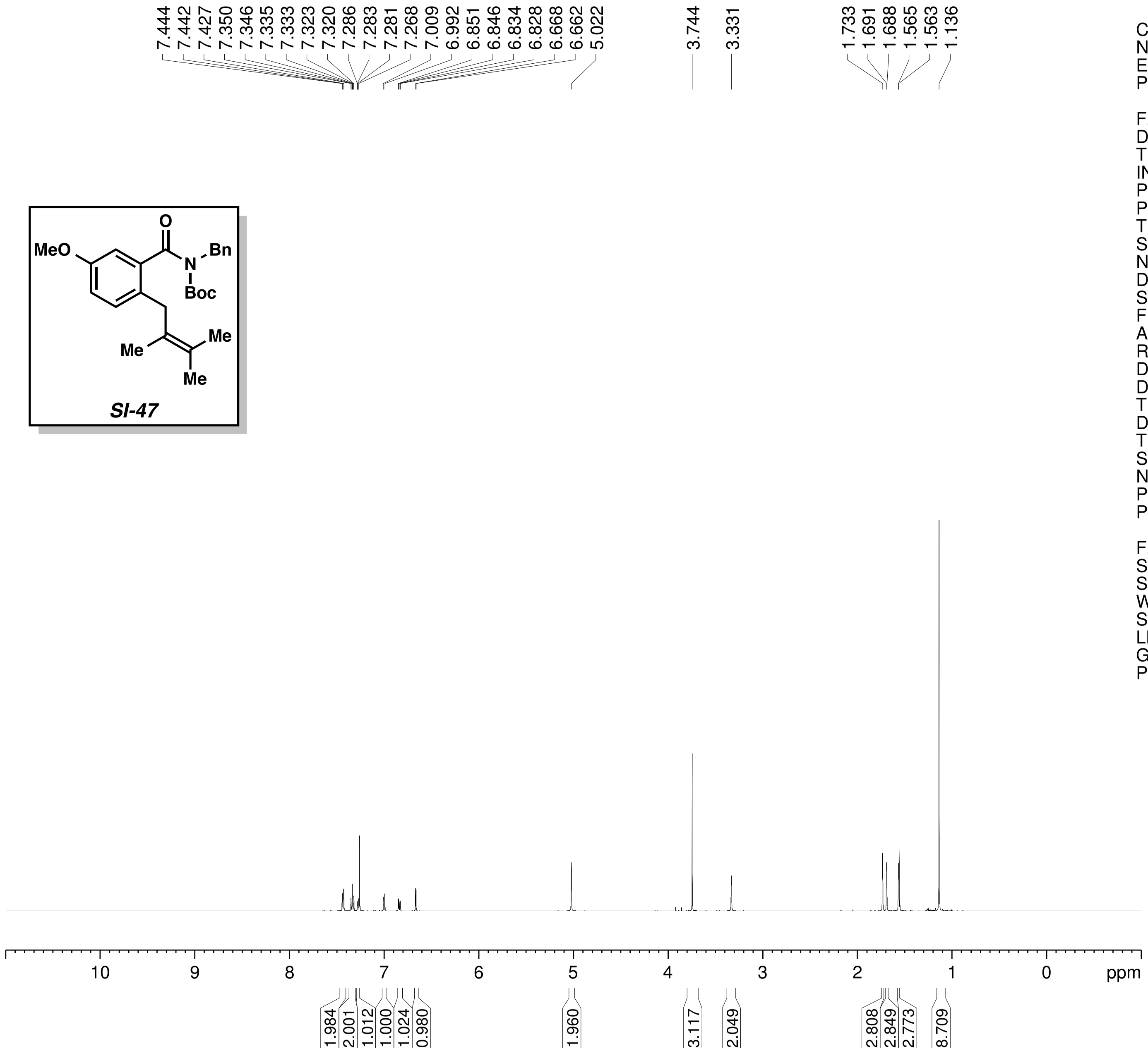
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

SI-46



```
Current Data Parameters
NAME          JM-5-055
EXPNO              200
PROCNO               1

F2 - Acquisition Parameters
Date_         20170206
Time           19.08 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   0
SWH         10000.000 Hz
FIDRES       0.305176 Hz
AQ          3.2767999 sec
RG              12.14
DW             50.000 usec
DE              10.00 usec
TE              298.0 K
D1         2.00000000 sec
TD0                  1
SFO1      500.1330008 MHz
NUC1                1H
P1              10.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI               65536
SF        500.1300121 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```

SI-47



Current Data Parameters

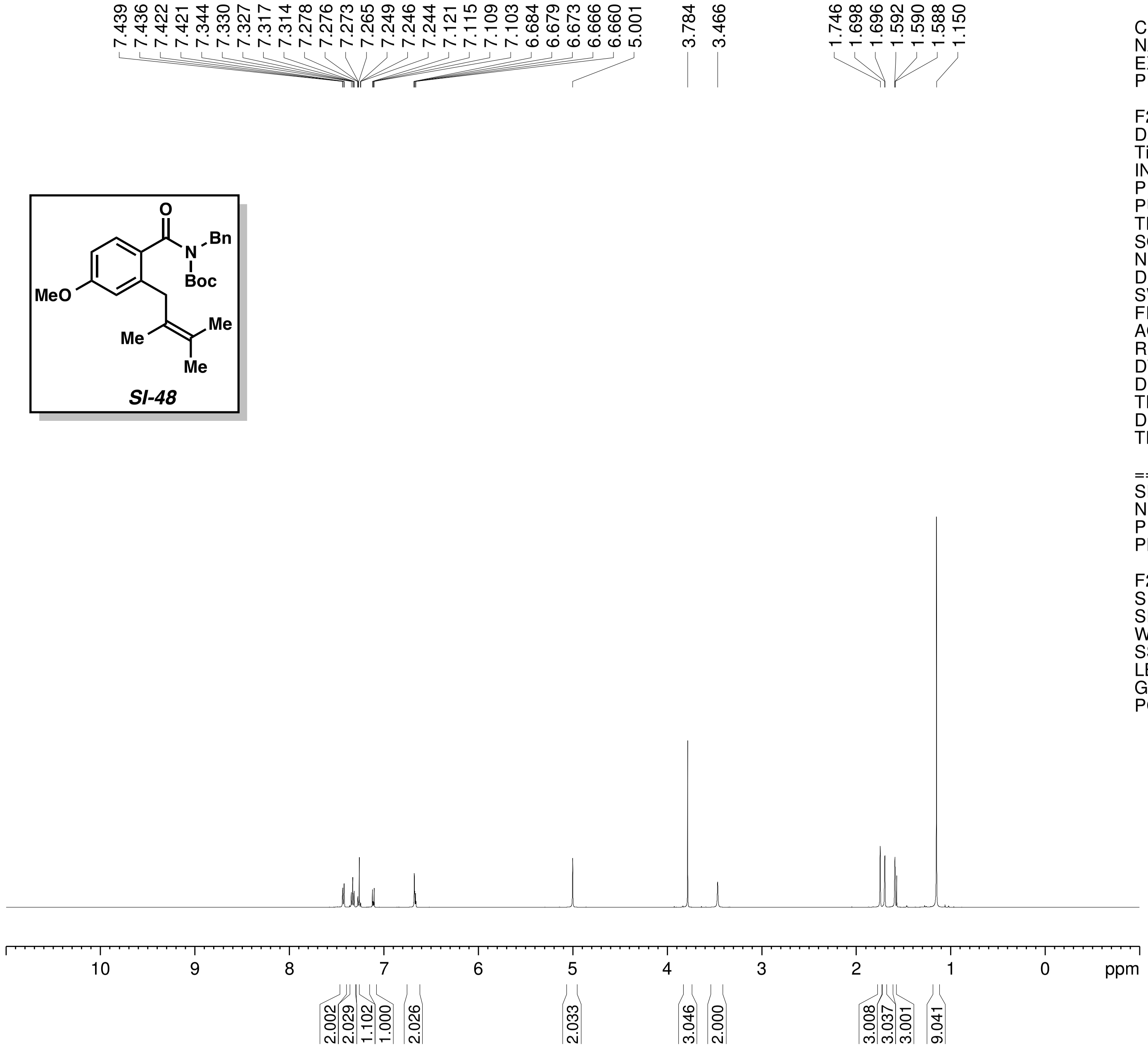
NAME	JMM-7-71.500
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20170131
Time	9.27 h
INSTRUM	av500
PROBHD	Z119248_0002 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.305176 Hz
AQ	3.2767999 sec
RG	37.5
DW	50.000 usec
DE	10.00 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
SFO1	500.1330008 MHz
NUC1	1H
P1	10.00 usec
PLW1	13.50000000 W

F2 - Processing parameters

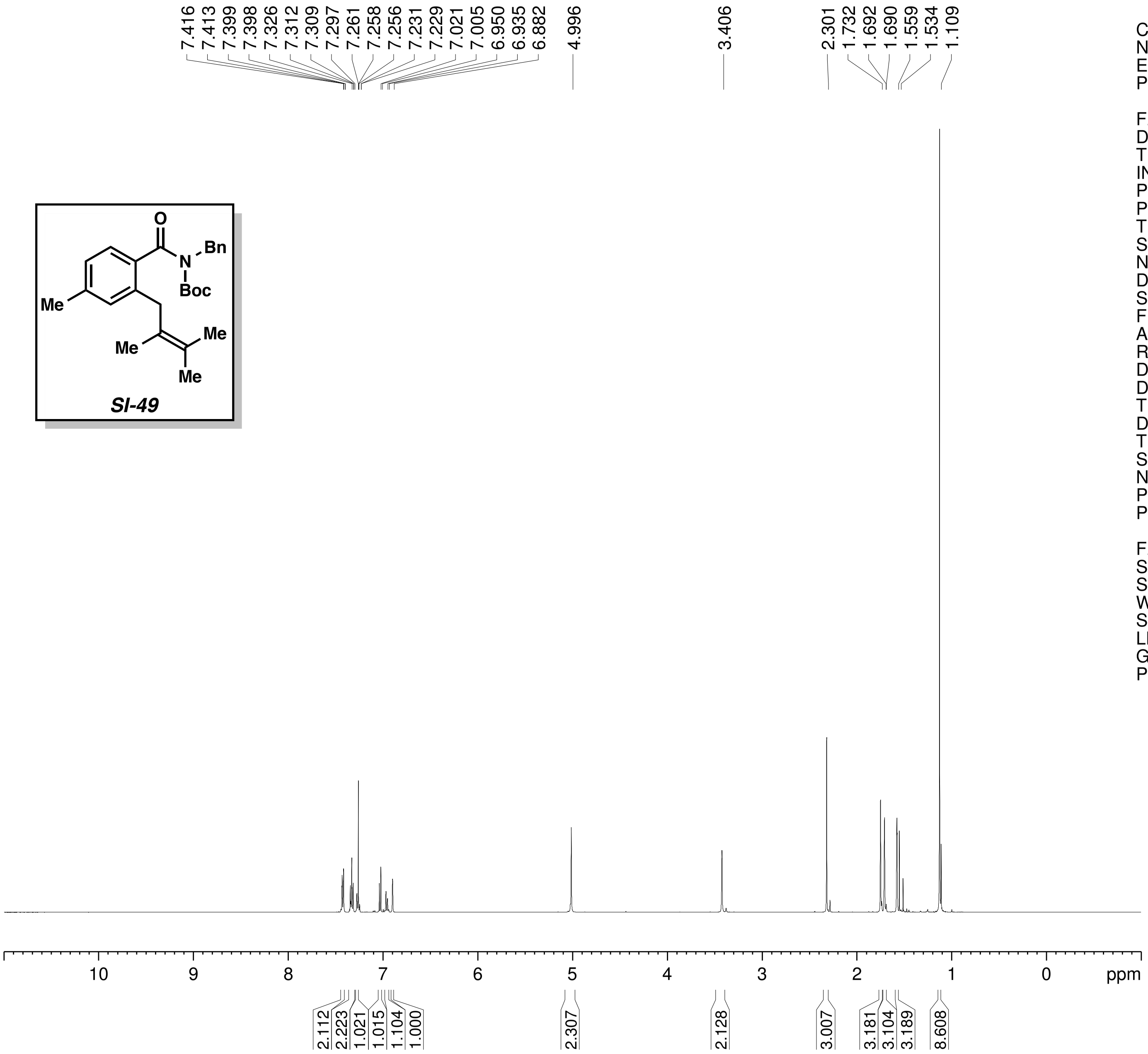
SI	65536
SF	500.1300119 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



SI-48

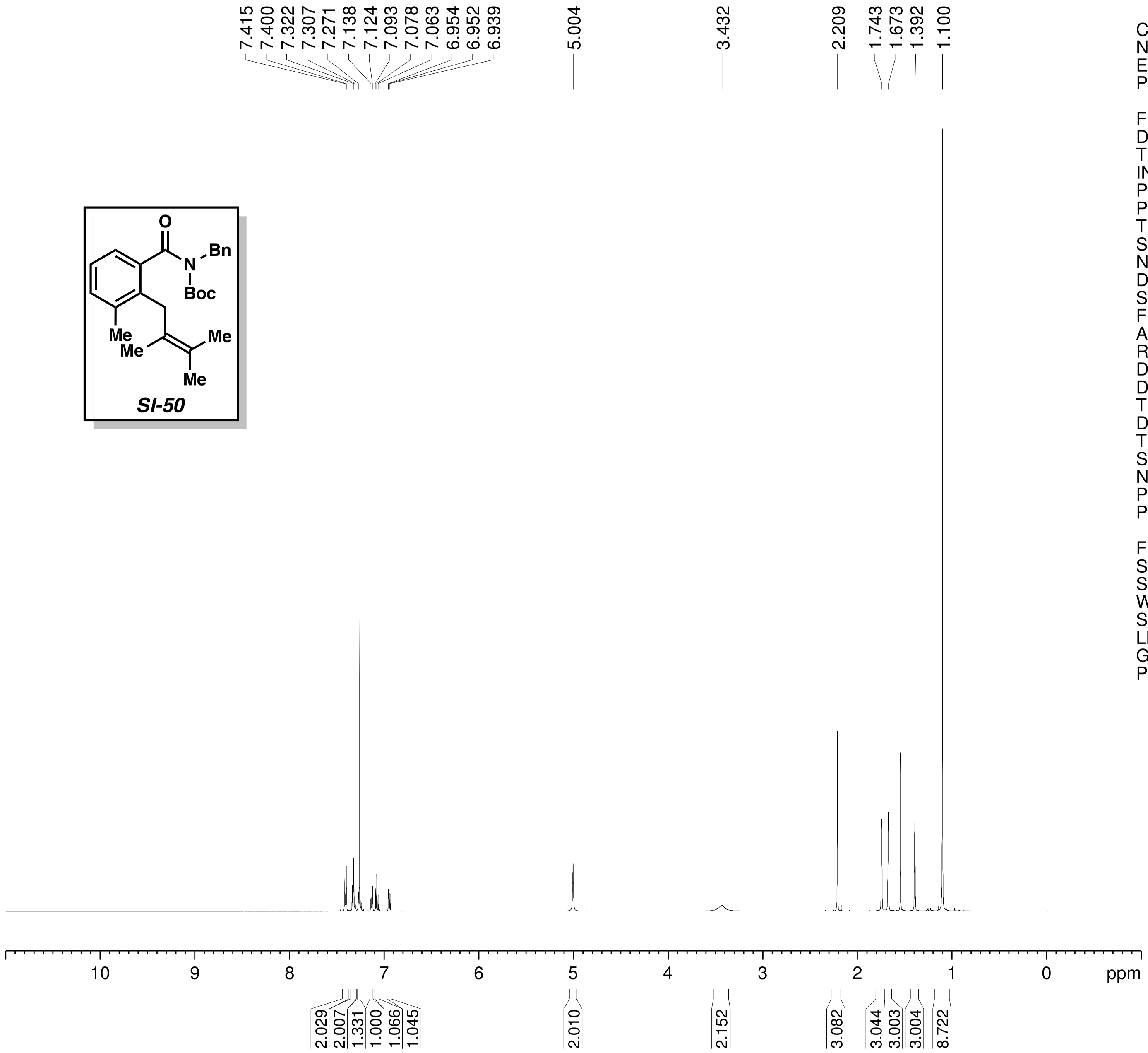
```
Current Data Parameters
NAME     JMM-7-252-char
EXPNO              1
PROCNO           999

F2 - Acquisition Parameters
Date_        20170215
Time            14.15
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 26
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ         3.2767999 sec
RG              12.14
DW          50.000 usec
DE          10.00 usec
TE            298.0 K
D1       2.00000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1      500.1340010 MHz
NUC1               1H
P1           10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI              65536
SF        500.1300124 MHz
WDW                EM
SSB     0
LB            0.30 Hz
GB      0
PC               1.00
```

SI-49



```
Current Data Parameters
NAME      sr-1-129.500
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20170217
Time           11.03 h
INSTRUM         av500
PROBHD   Z119248_0002 (
PULPROG          zg30
TD            65536
SOLVENT          CDCl3
NS                8
DS                0
SWH         10000.000 Hz
FIDRES       0.305176 Hz
AQ          3.2767999 sec
RG            12.14
DW            50.000 usec
DE            10.00 usec
TE            298.0 K
D1         2.00000000 sec
TD0               1
SFO1       500.1330008 MHz
NUC1              1H
P1            10.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI            65536
SF         500.1300123 MHz
WDW              EM
SSB     0
LB              0.30 Hz
GB      0
PC              1.00
```

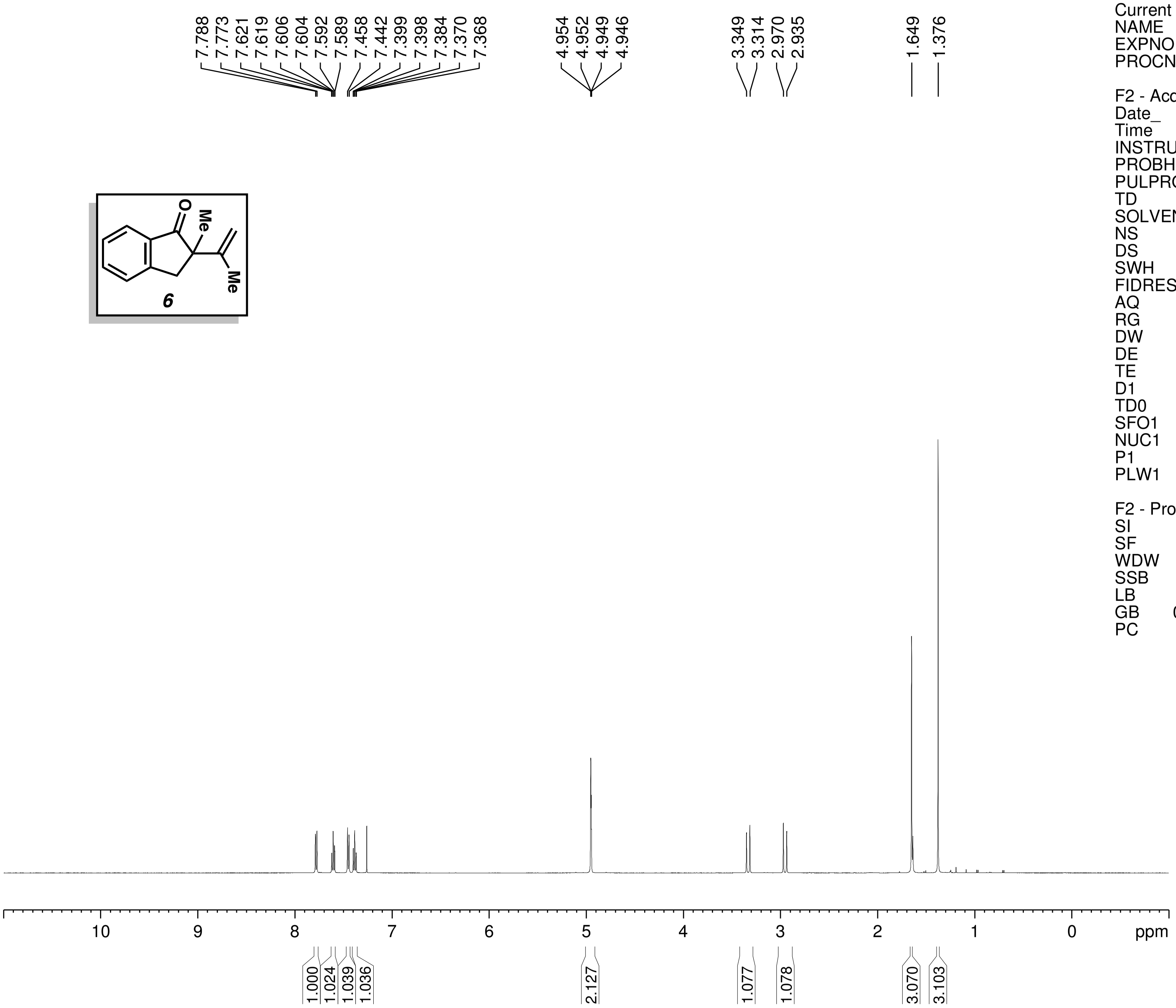
SI-50



Current Data Parameters
NAME sr-1-101.500
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170131
Time 9.08 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 23.34
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

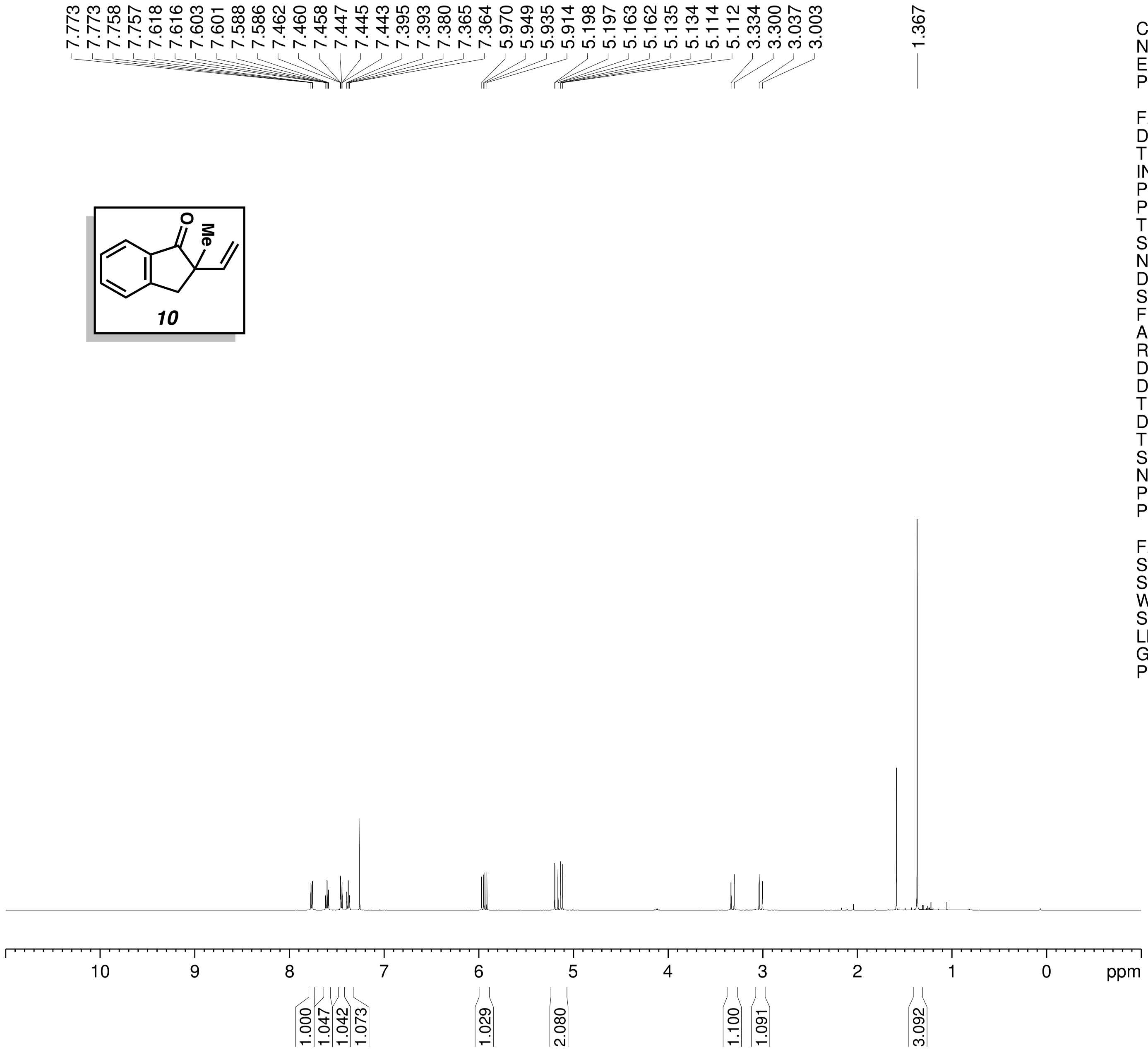
F2 - Processing parameters
SI 65536
SF 500.1300133 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



6

```
Current Data Parameters
NAME          JM-5-025
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_         20170115
Time            13.48 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   0
SWH         10000.000 Hz
FIDRES       0.305176 Hz
AQ          3.2767999 sec
RG               12.14
DW              50.000 usec
DE               10.00 usec
TE               298.0 K
D1          2.00000000 sec
TD0                  1
SFO1       500.1330008 MHz
NUC1                1H
P1               10.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI               65536
SF         500.1300123 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```

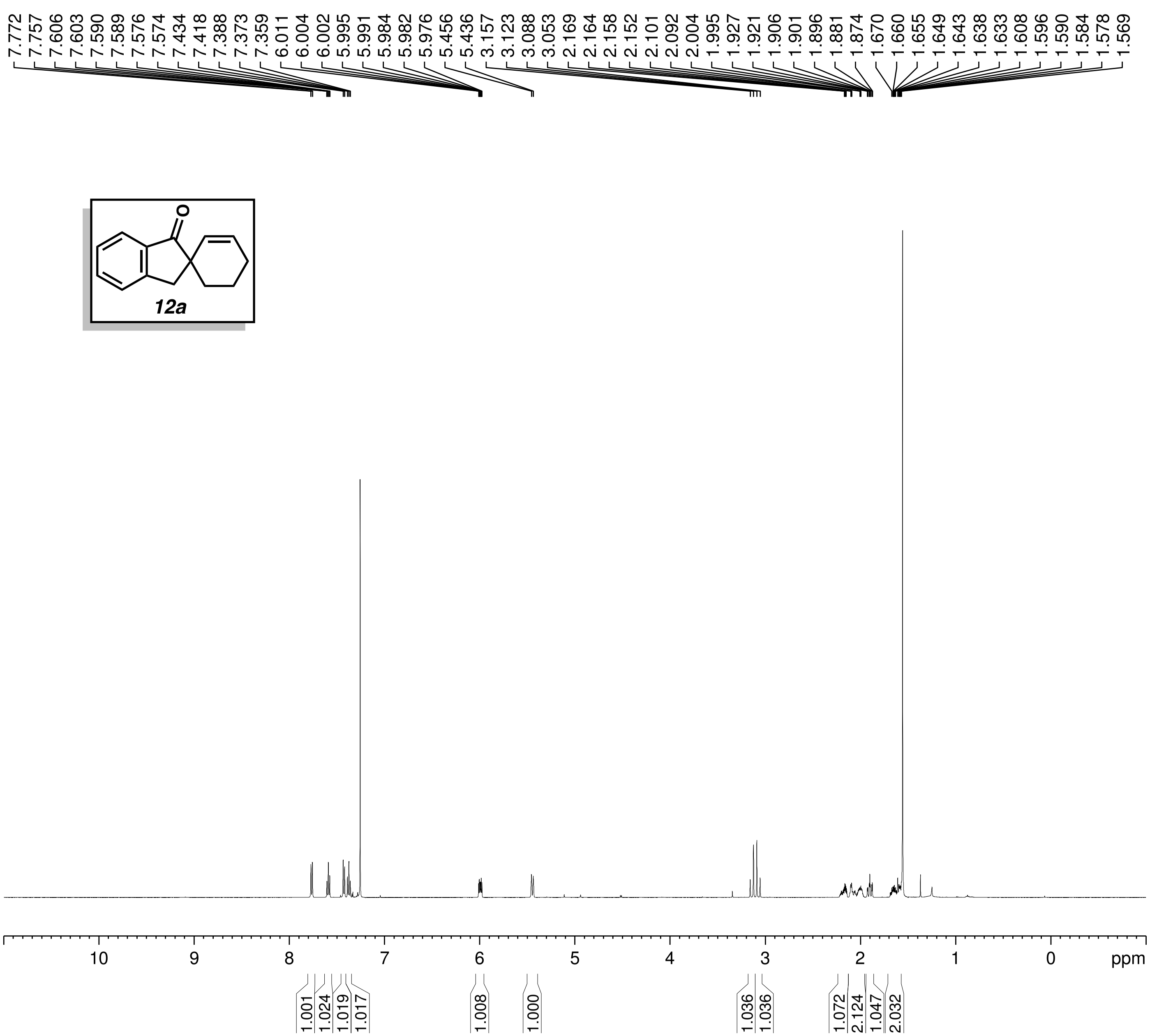
10



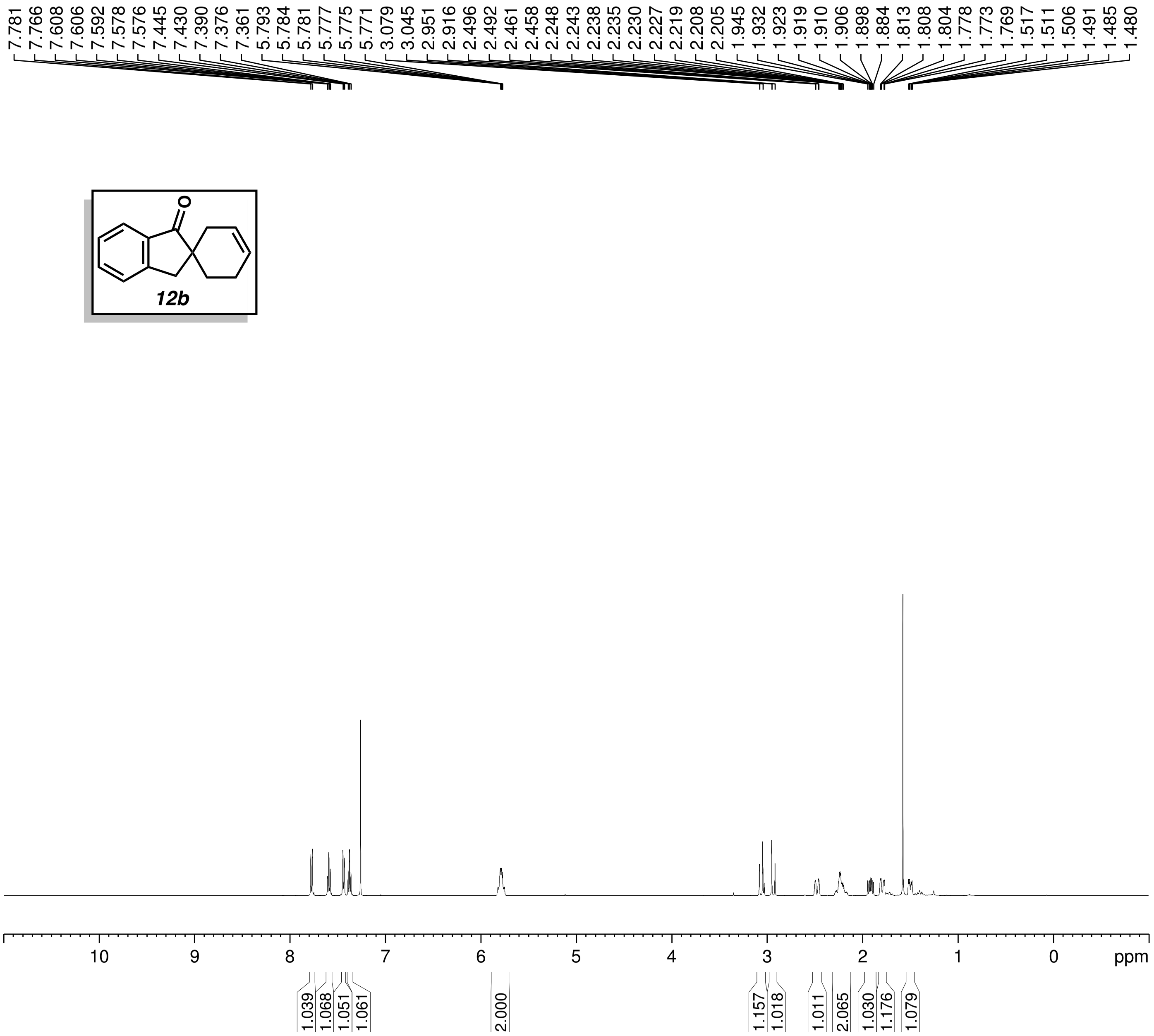
Current Data Parameters
NAME sr-1-108.500.6
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170207
Time 10.13 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 26.59
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300133 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

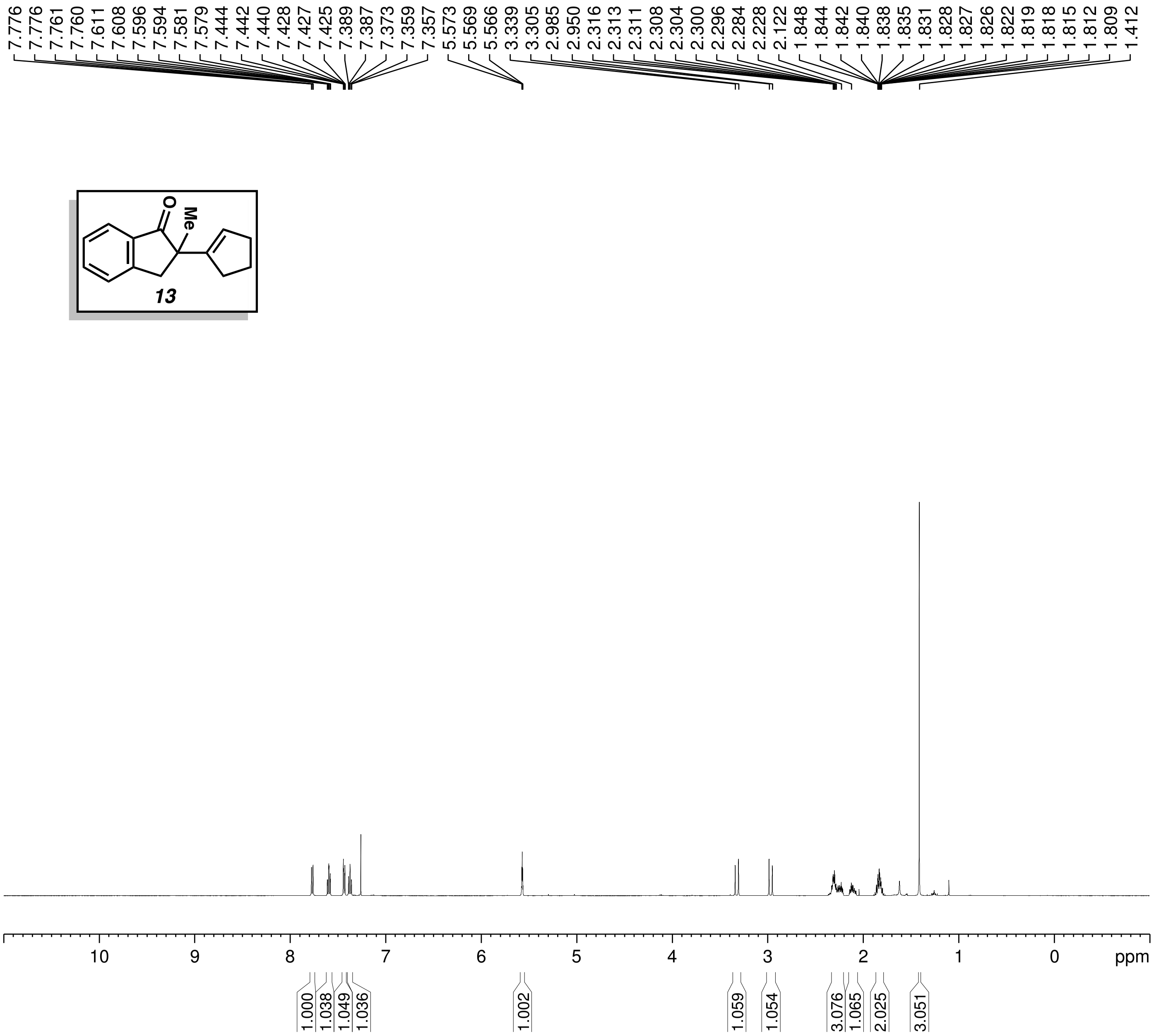

12a

```
Current Data Parameters
NAME          JM-5-019
EXPNO              400
PROCNO               1

F2 - Acquisition Parameters
Date_         20170109
Time           18.27 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   0
SWH        10000.000 Hz
FIDRES      0.305176 Hz
AQ        3.2767999 sec
RG               12.14
DW          50.000 usec
DE          10.00 usec
TE             298.0 K
D1      2.00000000 sec
TD0                  1
SFO1    500.1330008 MHz
NUC1                1H
P1          10.00 usec
PLW1     13.50000000 W

F2 - Processing parameters
SI               65536
SF      500.1300146 MHz
WDW                 EM
SSB      0
LB             0.30 Hz
GB       0
PC                1.00
```

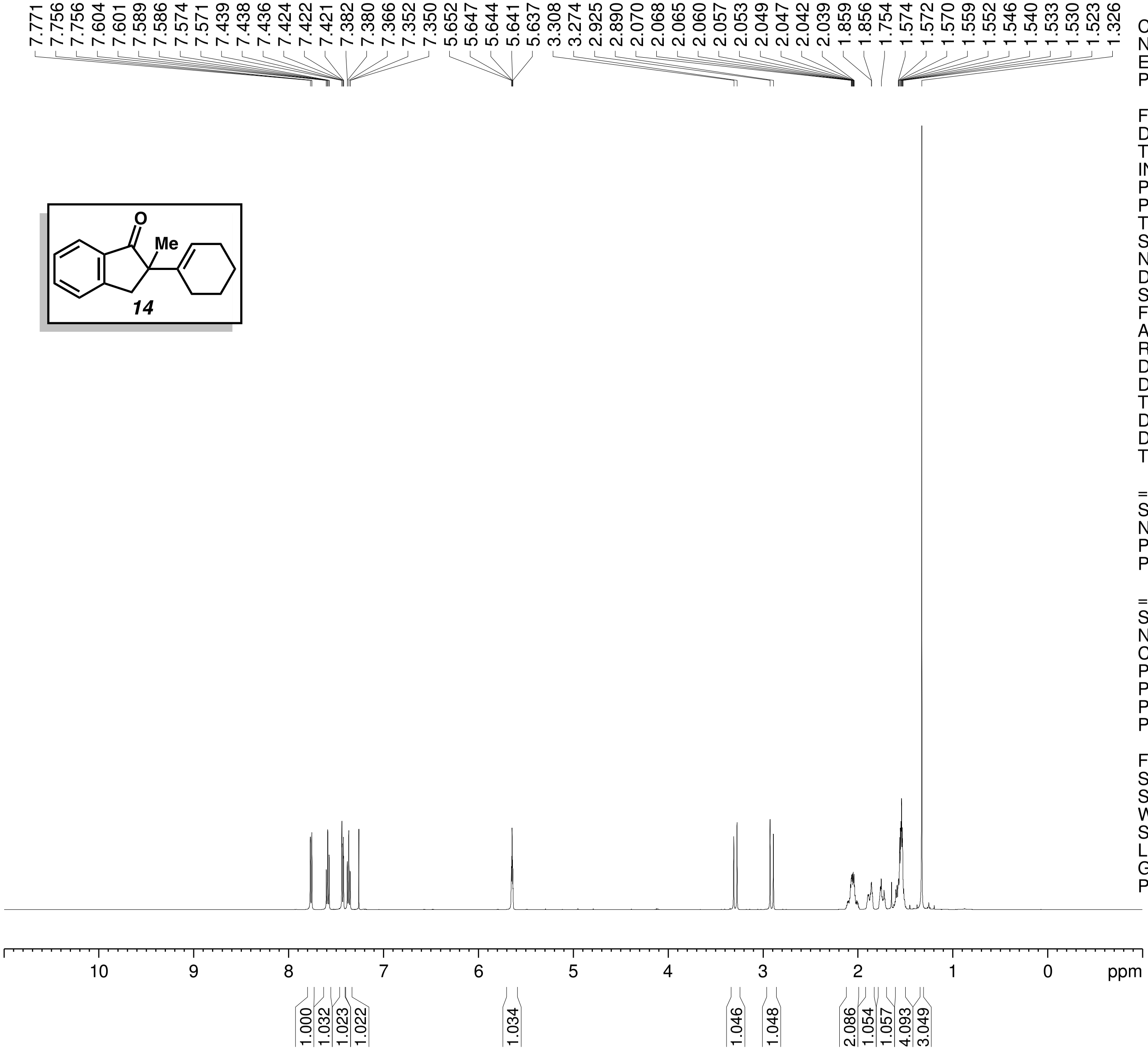


12b

```
Current Data Parameters
NAME          JM-5-019
EXPNO              300
PROCNO               1

F2 - Acquisition Parameters
Date_         20170109
Time            18.22 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   0
SWH         10000.000 Hz
FIDRES       0.305176 Hz
AQ          3.2767999 sec
RG               12.14
DW              50.000 usec
DE               10.00 usec
TE               298.0 K
D1          2.00000000 sec
TD0                  1
SFO1       500.1330008 MHz
NUC1                1H
P1               10.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI               65536
SF         500.1300122 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```

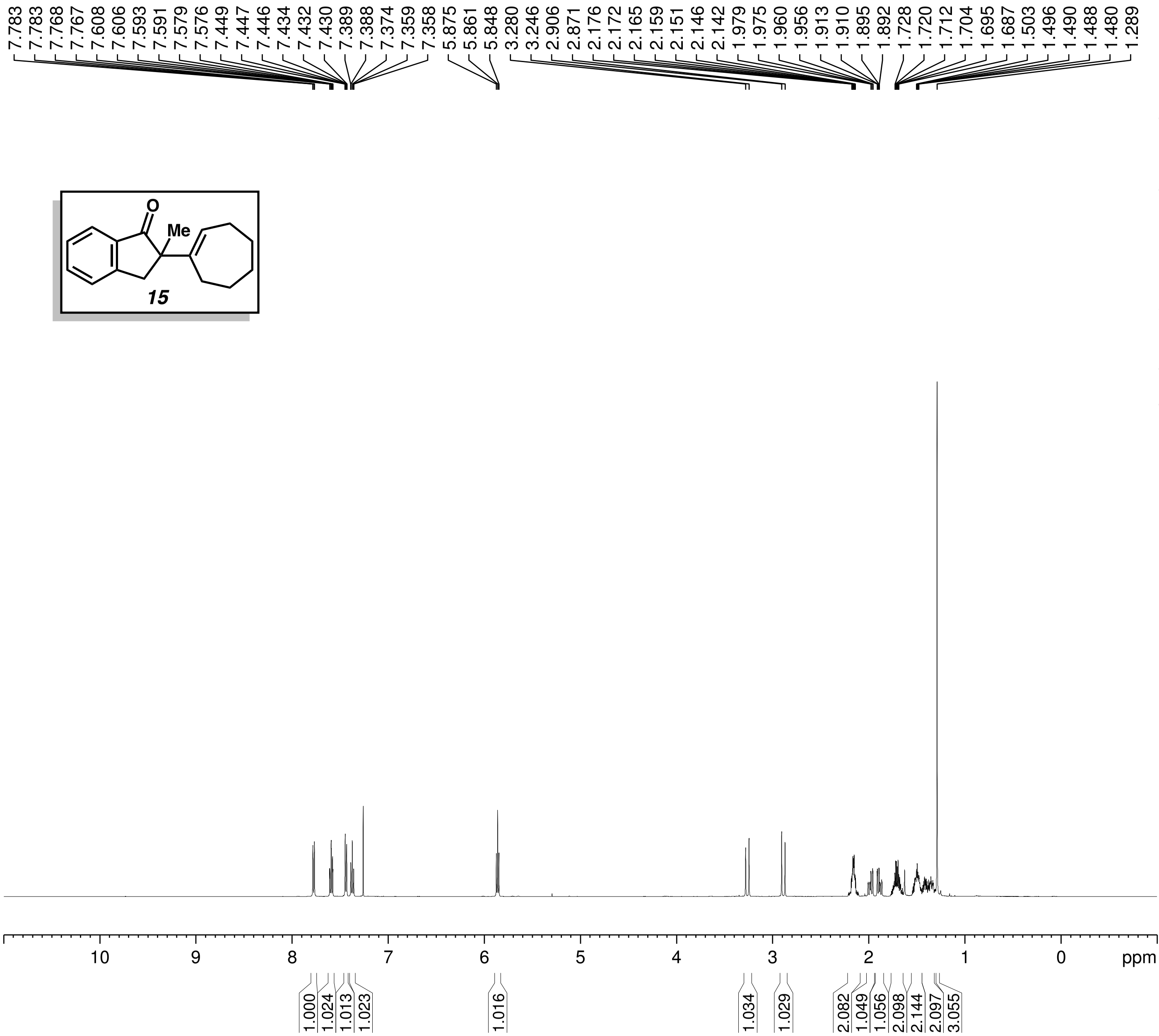


```
Current Data Parameters
NAME      JMM-7-212(A1)
EXPNO              1
PROCNO           999

F2 - Acquisition Parameters
Date_        20161216
Time             9.07
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 13
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ         3.2767999 sec
RG              12.14
DW            50.000 usec
DE             10.00 usec
TE             298.0 K
D1         2.00000000 sec
TD0                 1

======== CHANNEL f1 ======
SFO1       500.1320005 MHz
NUC1               1H
P1             10.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI              65536
SF         500.1300122 MHz
WDW                EM
SSB     0
LB               0.30 Hz
GB      0
PC               1.00
```



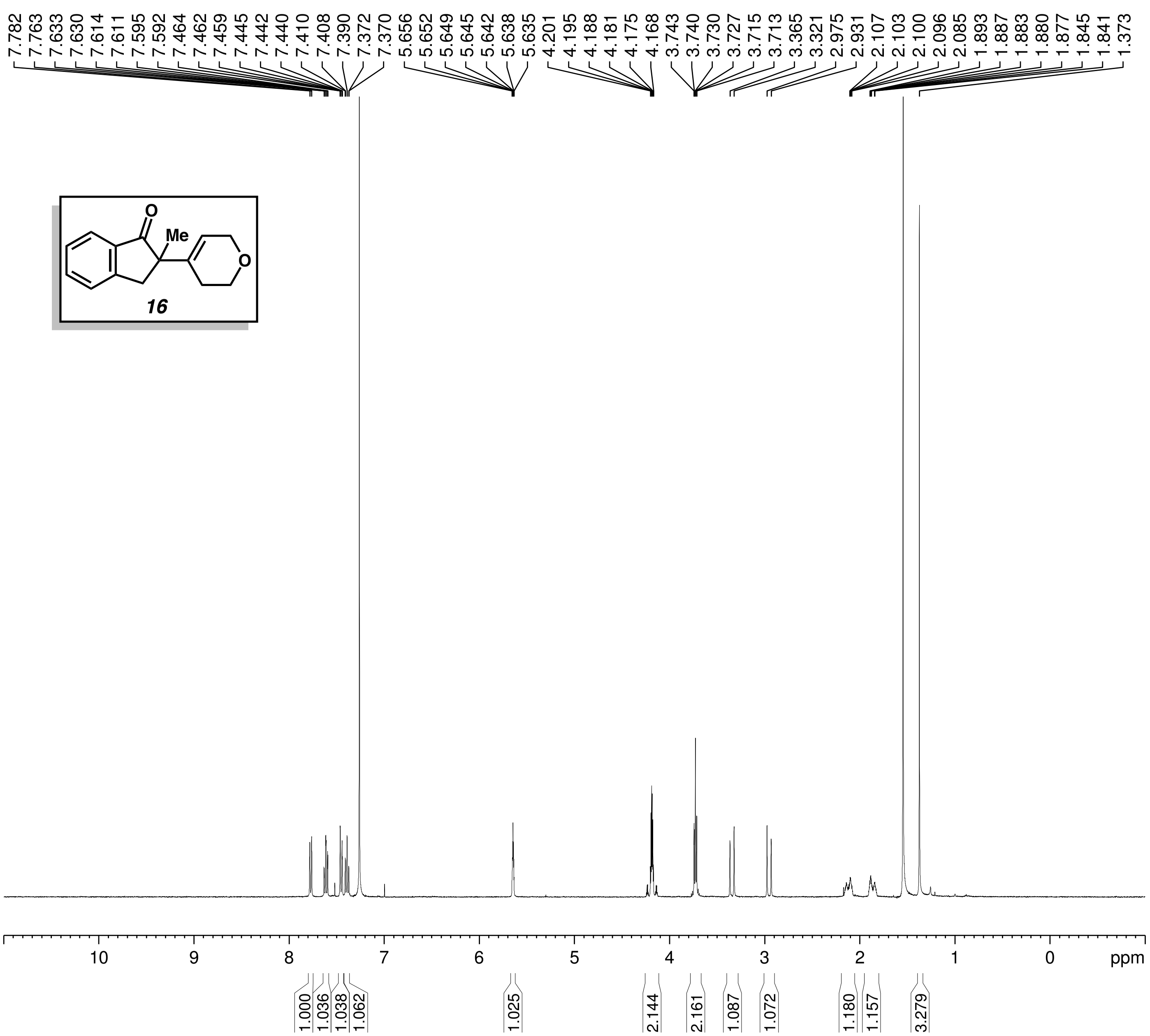
14

```
Current Data Parameters
NAME     JMM-7-198(Cchar)
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20161204
Time            15.48
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD            65536
SOLVENT          C6D6
NS                164
DS                  2
SWH        50000.000 Hz
FIDRES      0.762939 Hz
AQ        0.6553600 sec
RG             204.54
DW            10.000 usec
DE            100.00 usec
TE              298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1      125.7640769 MHz
NUC1              13C
P1              9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 =====
SFO2      500.1330008 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI              65536
SF        500.1300123 MHz
WDW                EM
SSB     0
LB               0.30 Hz
GB      0
PC               1.00
```



15

```
Current Data Parameters
NAME     JMM-7-225-char
EXPNO              1
PROCNO           999

F2 - Acquisition Parameters
Date_        20170126
Time            13.13
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 15
DS                  0
SWH       10000.000 Hz
FIDRES     0.152588 Hz
AQ       3.2767999 sec
RG              12.14
DW         50.000 usec
DE          10.00 usec
TE            298.0 K
D1     2.00000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1     500.1340010 MHz
NUC1               1H
P1          10.00 usec
PLW1    13.50000000 W

F2 - Processing parameters
SI              65536
SF       500.1300121 MHz
WDW                EM
SSB     0
LB            0.30 Hz
GB      0
PC               1.00
```



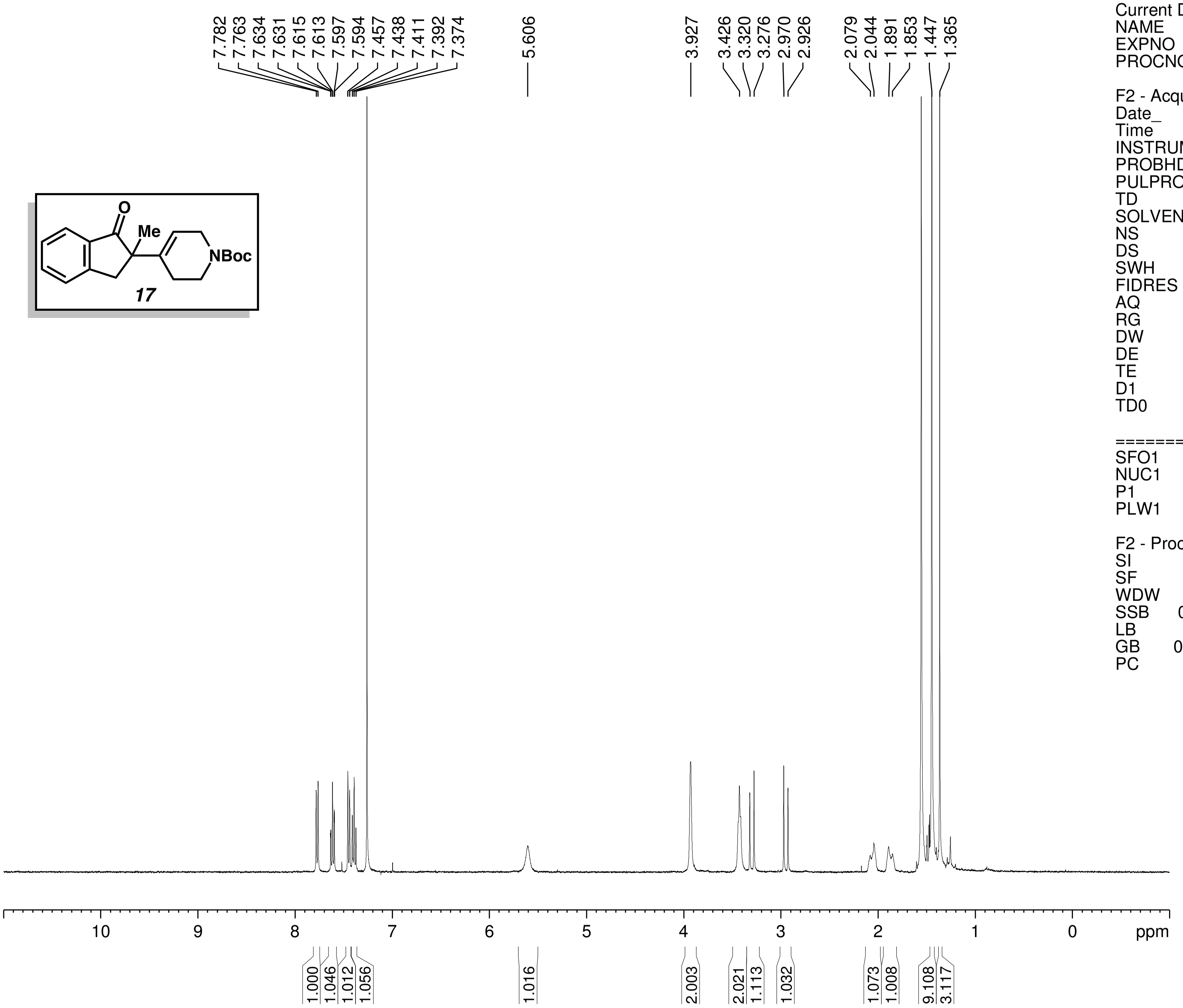
16

Current Data Parameters
NAME JM-4-292
EXPNO 450
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161206
Time 21.19
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 96
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 189.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ======
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

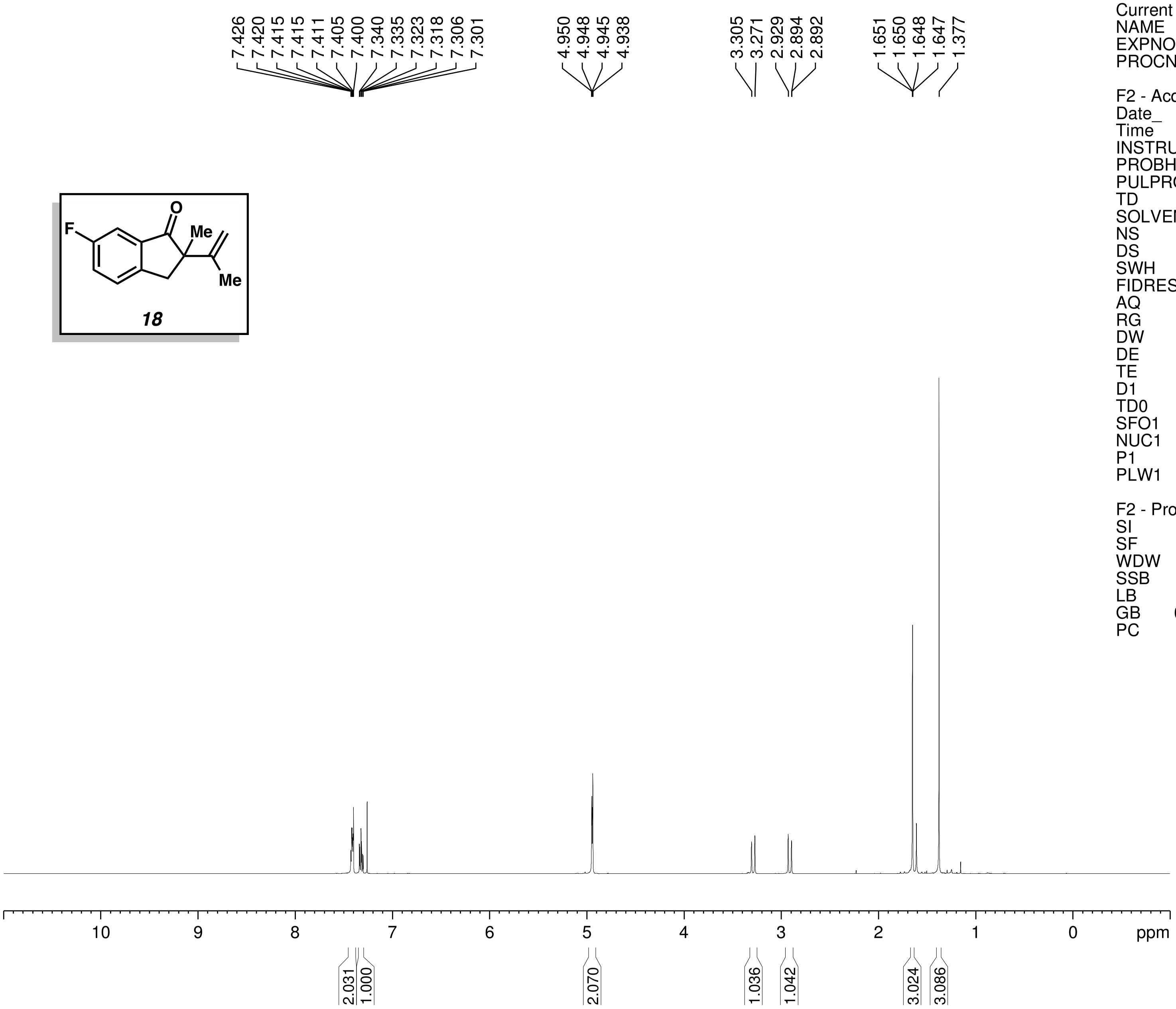


Current Data Parameters
NAME JM-5-032
EXPNO 180
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170120
Time 12.11
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 189.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ======
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300176 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

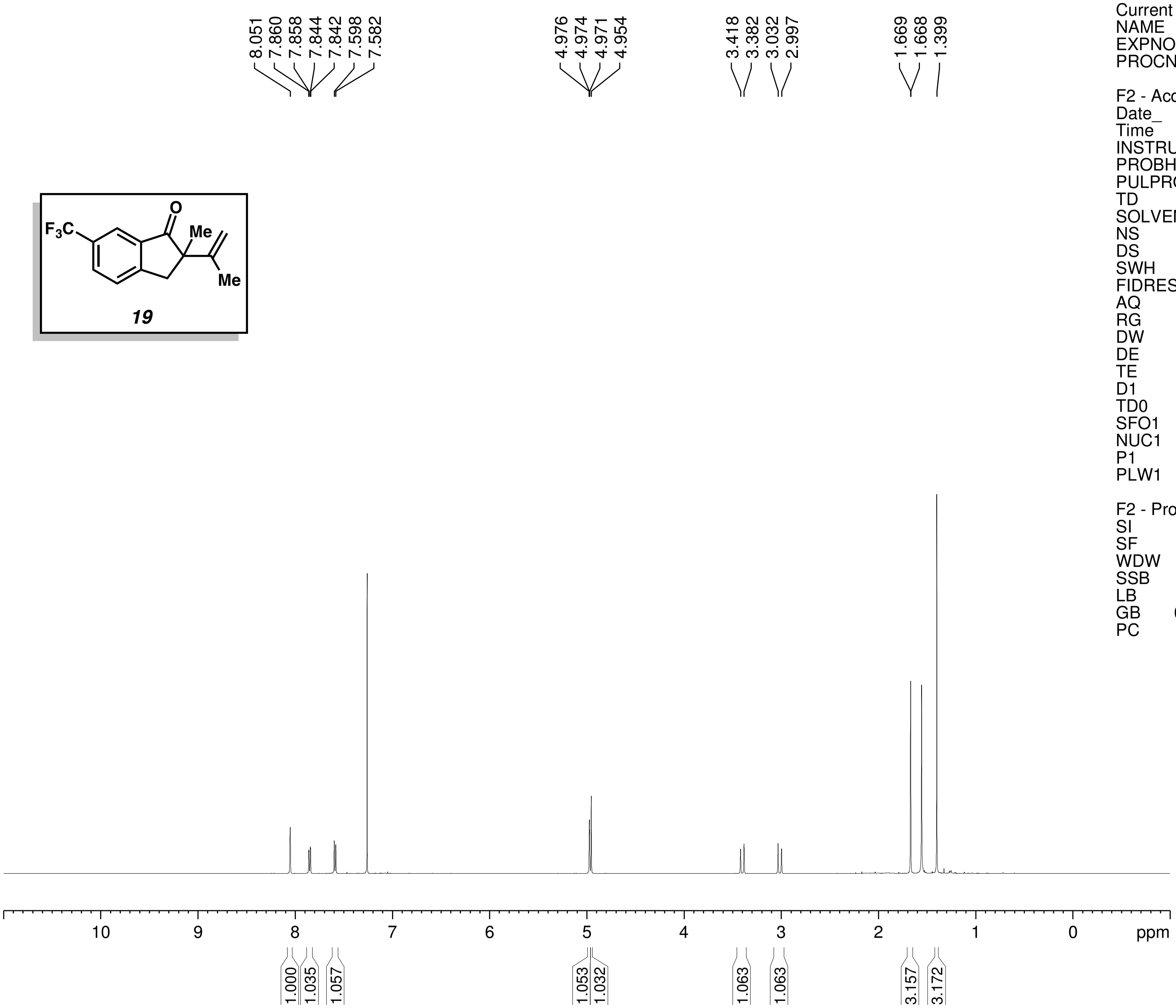


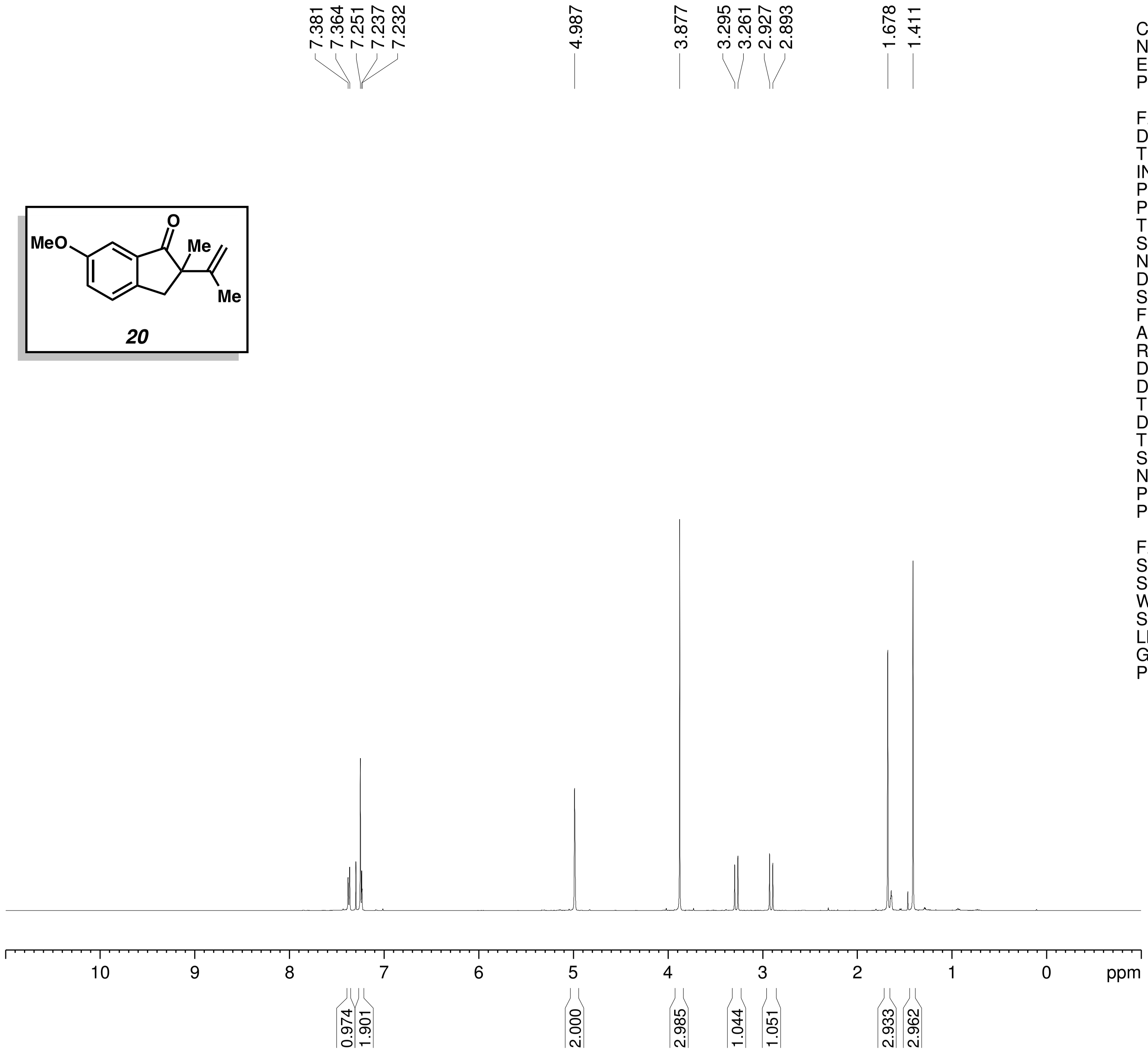
18

```
Current Data Parameters
NAME          JM-5-067
EXPNO              200
PROCNO               1

F2 - Acquisition Parameters
Date_         20170217
Time            18.15 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD             65536
SOLVENT          CDCl3
NS                   8
DS                   0
SWH        10000.000 Hz
FIDRES      0.305176 Hz
AQ         3.2767999 sec
RG              21.37
DW             50.000 usec
DE             10.00 usec
TE             298.0 K
D1        2.00000000 sec
TD0                  1
SFO1     500.1330008 MHz
NUC1                1H
P1             10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI            65536
SF       500.1300125 MHz
WDW              EM
SSB     0
LB             0.30 Hz
GB      0
PC             1.00
```

19

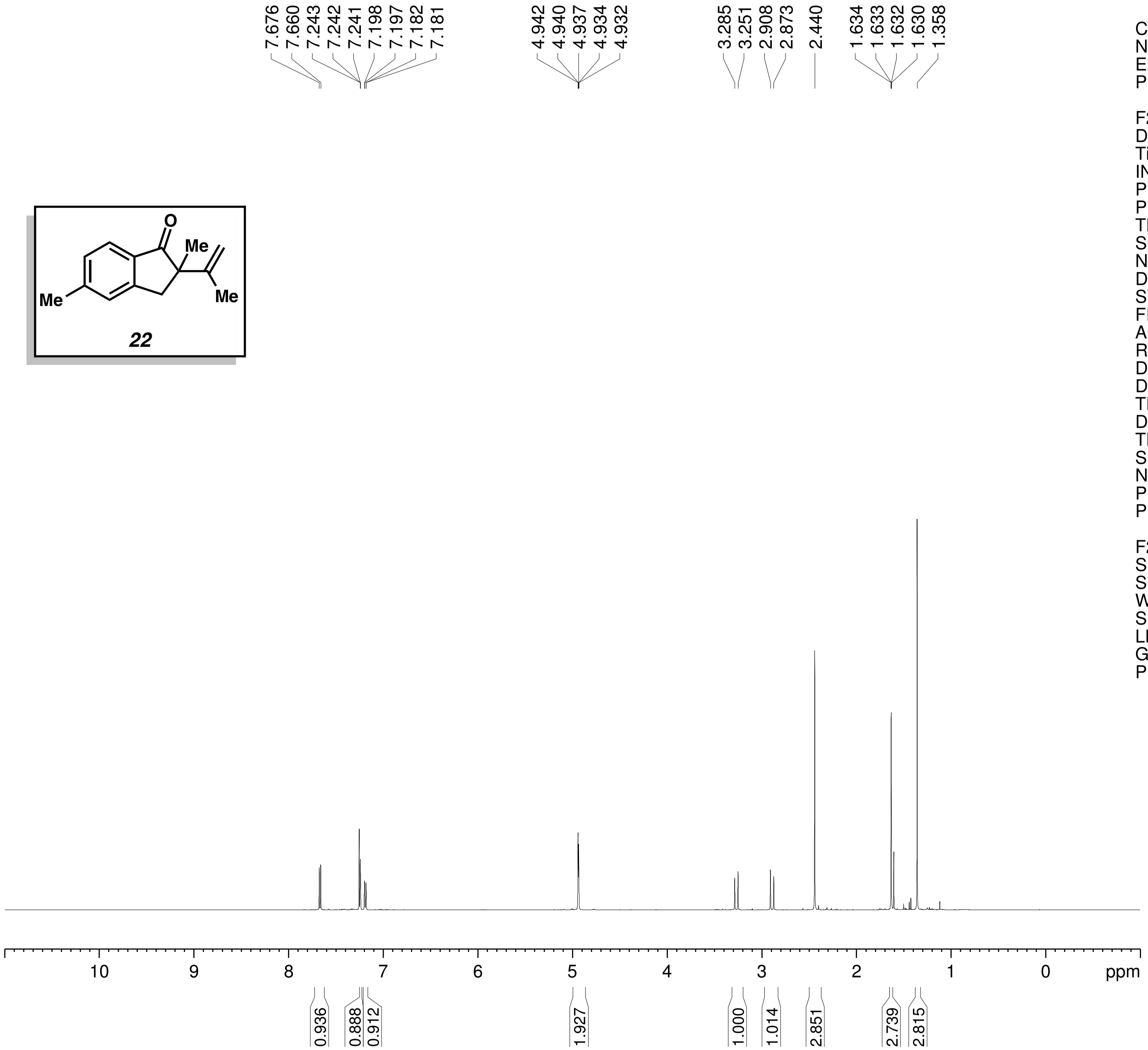
```
Current Data Parameters
NAME          JM-5-057
EXPNO              100
PROCNO               1

F2 - Acquisition Parameters
Date_          20170206
Time            19.24 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH          10000.000 Hz
FIDRES        0.305176 Hz
AQ           3.2767999 sec
RG                26.59
DW               50.000 usec
DE                10.00 usec
TE                298.0 K
D1           2.00000000 sec
TD0                   1
SFO1       500.1330008 MHz
NUC1                 1H
P1                10.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                65536
SF         500.1300123 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```





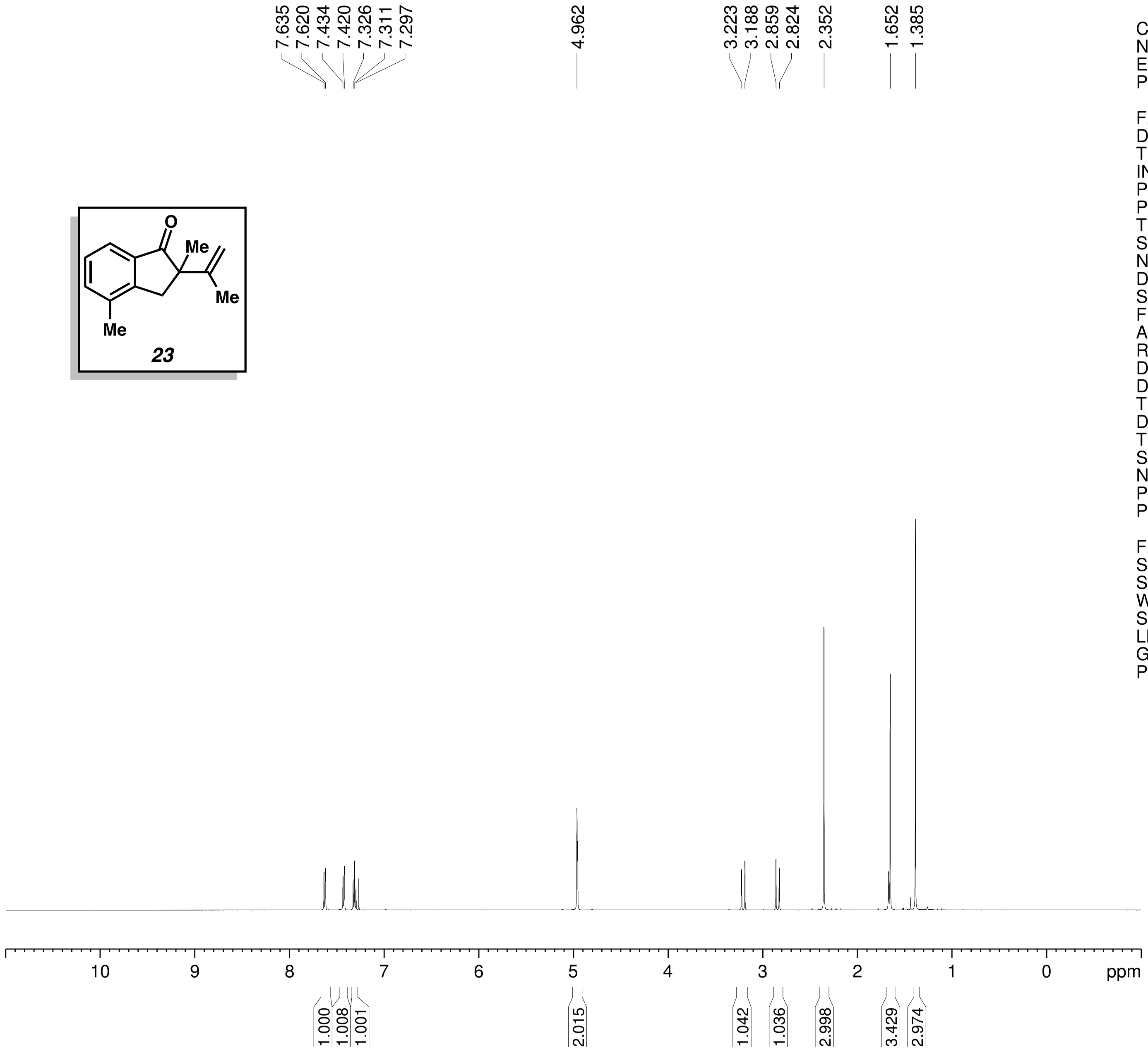
20

```
Current Data Parameters
NAME       sr-1-105.500
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20170203
Time             9.00 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH        10000.000 Hz
FIDRES      0.305176 Hz
AQ         3.2767999 sec
RG                12.14
DW           50.000 usec
DE            10.00 usec
TE              298.0 K
D1        2.00000000 sec
TD0                   1
SFO1     500.1330008 MHz
NUC1                 1H
P1            10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI                65536
SF       500.1299930 MHz
WDW                  EM
SSB     0
LB              0.30 Hz
GB      0
PC                 1.00
```



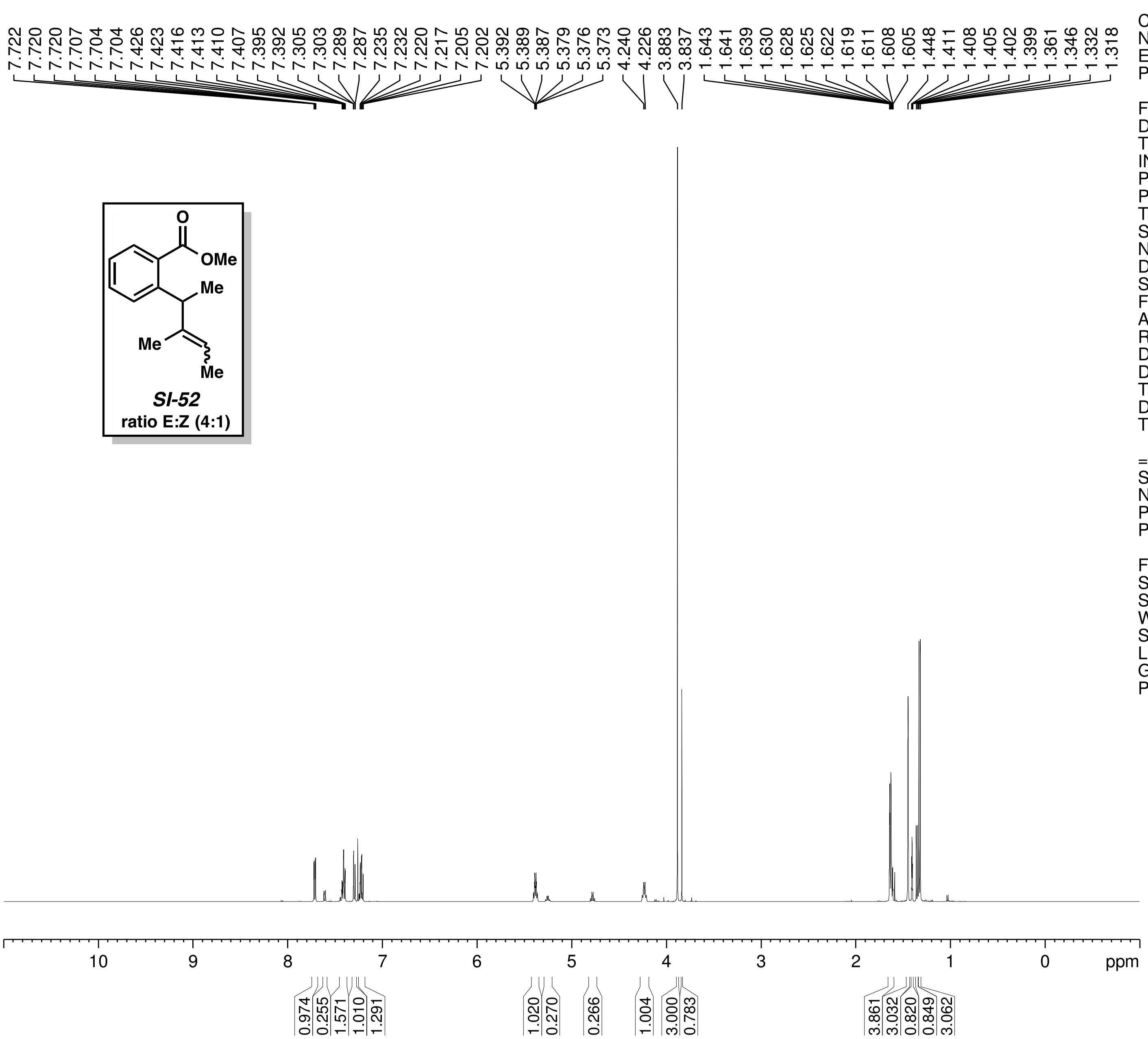
22

```
Current Data Parameters
NAME       sr-1-131.500
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20170217
Time            11.12 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH          10000.000 Hz
FIDRES        0.305176 Hz
AQ          3.2767999 sec
RG                23.34
DW               50.000 usec
DE                10.00 usec
TE                298.0 K
D1          2.00000000 sec
TD0                   1
SFO1       500.1330008 MHz
NUC1                 1H
P1                10.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                65536
SF         500.1300147 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```



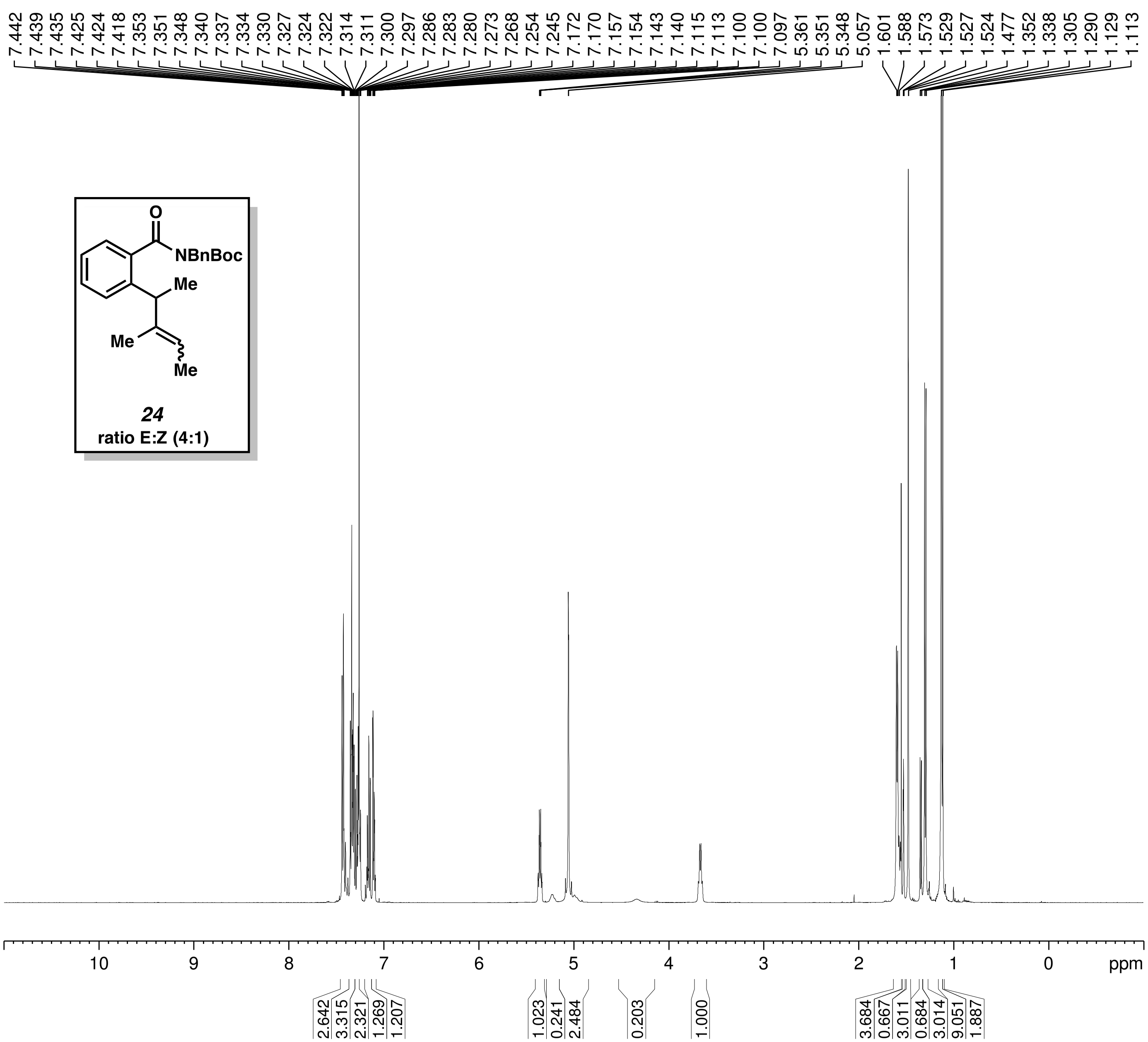
23

```
Current Data Parameters
NAME     sr-1-112.500.11
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_         20170208
Time            16.10 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD             65536
SOLVENT          CDCl3
NS                 8
DS                 0
SWH         10000.000 Hz
FIDRES       0.305176 Hz
AQ          3.2767999 sec
RG              12.14
DW             50.000 usec
DE              10.00 usec
TE              298.0 K
D1         2.00000000 sec
TD0                1
SFO1      500.1330008 MHz
NUC1              1H
P1              10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI            65536
SF        500.1300087 MHz
WDW               EM
SSB      0
LB              0.30 Hz
GB     0
PC              1.00
```



SI-52
ratio E:Z (4:1)

```
Current Data Parameters
NAME     JMM-7-244-char
EXPNO              1
PROCNO           999

F2 - Acquisition Parameters
Date_       20170215
Time           16.30
INSTRUM        av500
PROBHD   5 mm DCH 13C-1
PULPROG         zg30
TD             65536
SOLVENT        CDCl3
NS                29
DS                 0
SWH       10000.000 Hz
FIDRES     0.152588 Hz
AQ        3.2767999 sec
RG             12.14
DW            50.000 usec
DE             10.00 usec
TE             298.0 K
D1        2.00000000 sec
TD0                1

======== CHANNEL f1 =====
SFO1      500.1340010 MHz
NUC1              1H
P1             10.00 usec
PLW1     13.50000000 W

F2 - Processing parameters
SI             65536
SF        500.1300123 MHz
WDW               EM
SSB     0
LB              0.30 Hz
GB      0
PC              1.00
```



24
ratio E:Z (4:1)

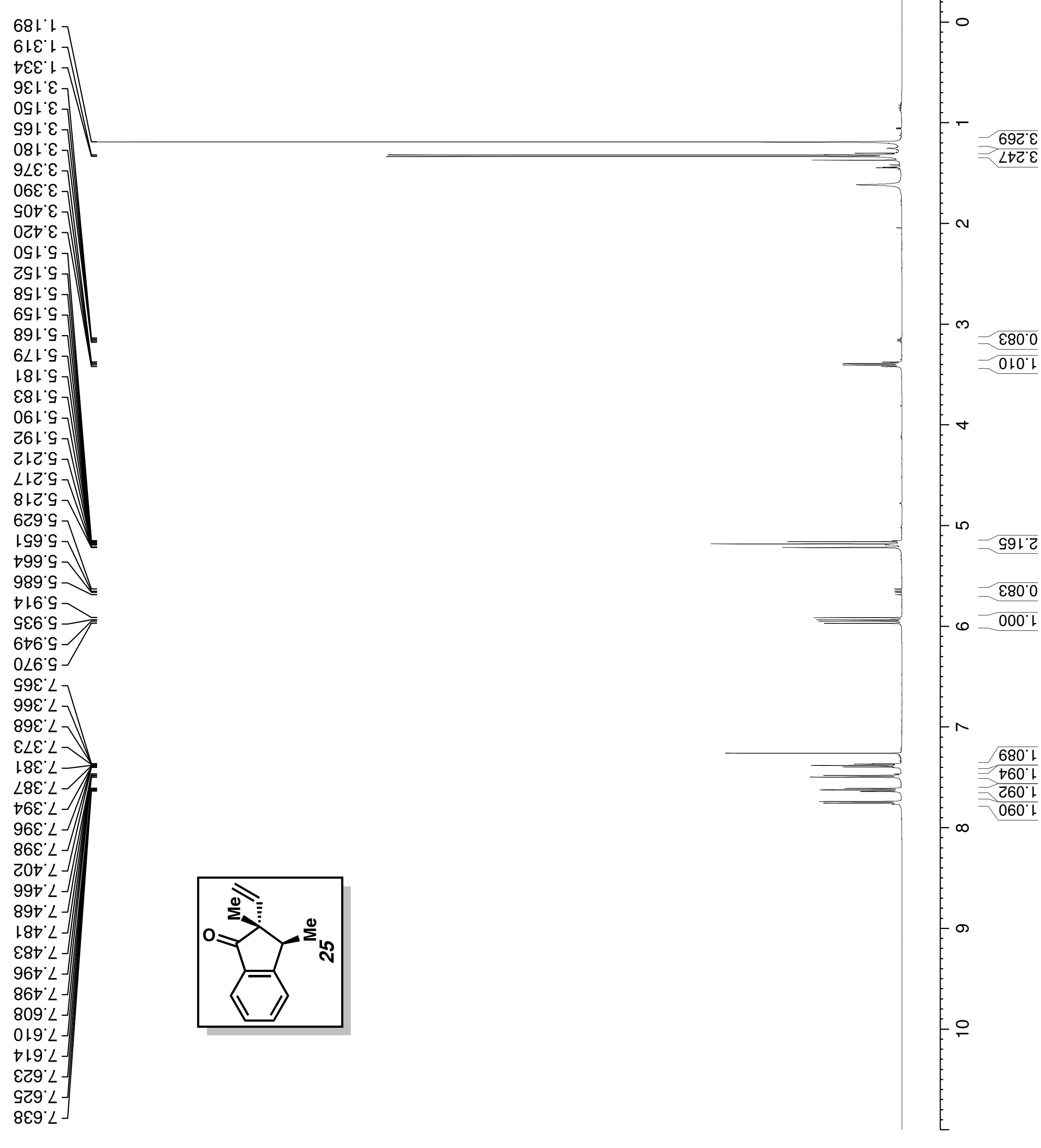
```
Current Data Parameters
NAME     JMM-7-247--char
EXPNO              1
PROCNO           999

F2 - Acquisition Parameters
Date_        20170215
Time            16.12
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 17
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ         3.2767999 sec
RG             12.14
DW            50.000 usec
DE             10.00 usec
TE             298.0 K
D1        2.00000000 sec
TD0                1

======== CHANNEL f1 =====
SFO1      500.1340010 MHz
NUC1               1H
P1             10.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI              65536
SF        500.1300121 MHz
WDW                EM
SSB      0
LB               0.30 Hz
GB      0
PC               1.00
```

Current Data Parameters
NAME JMM-7-249-char
EXPNO 1
PROCNO 999

F2 - Acquisition Parameters
Date_ 20170207
Time 19.59
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 30
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 23.34
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

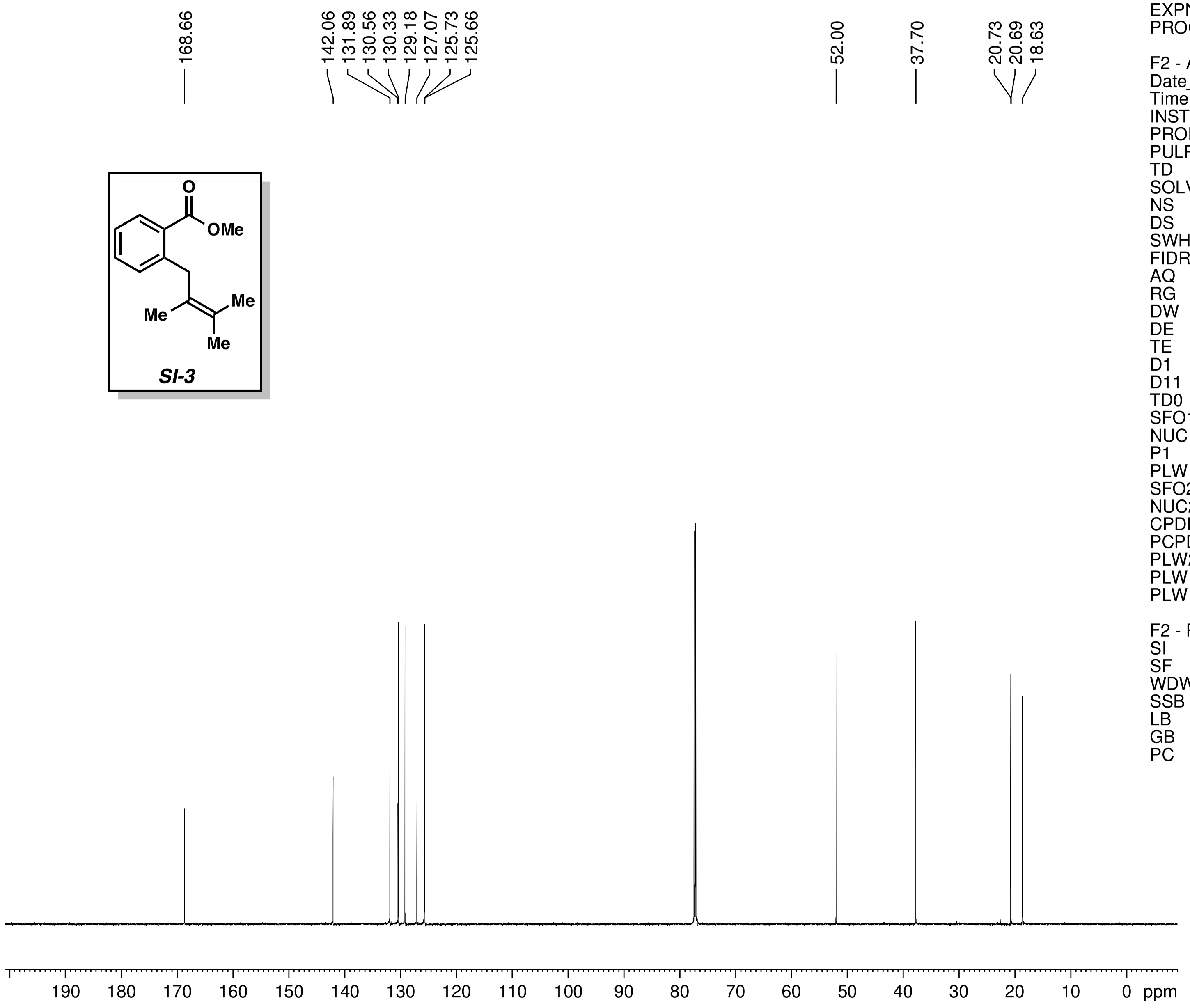
======== CHANNEL f1 ========
SFO1 500.1340010 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



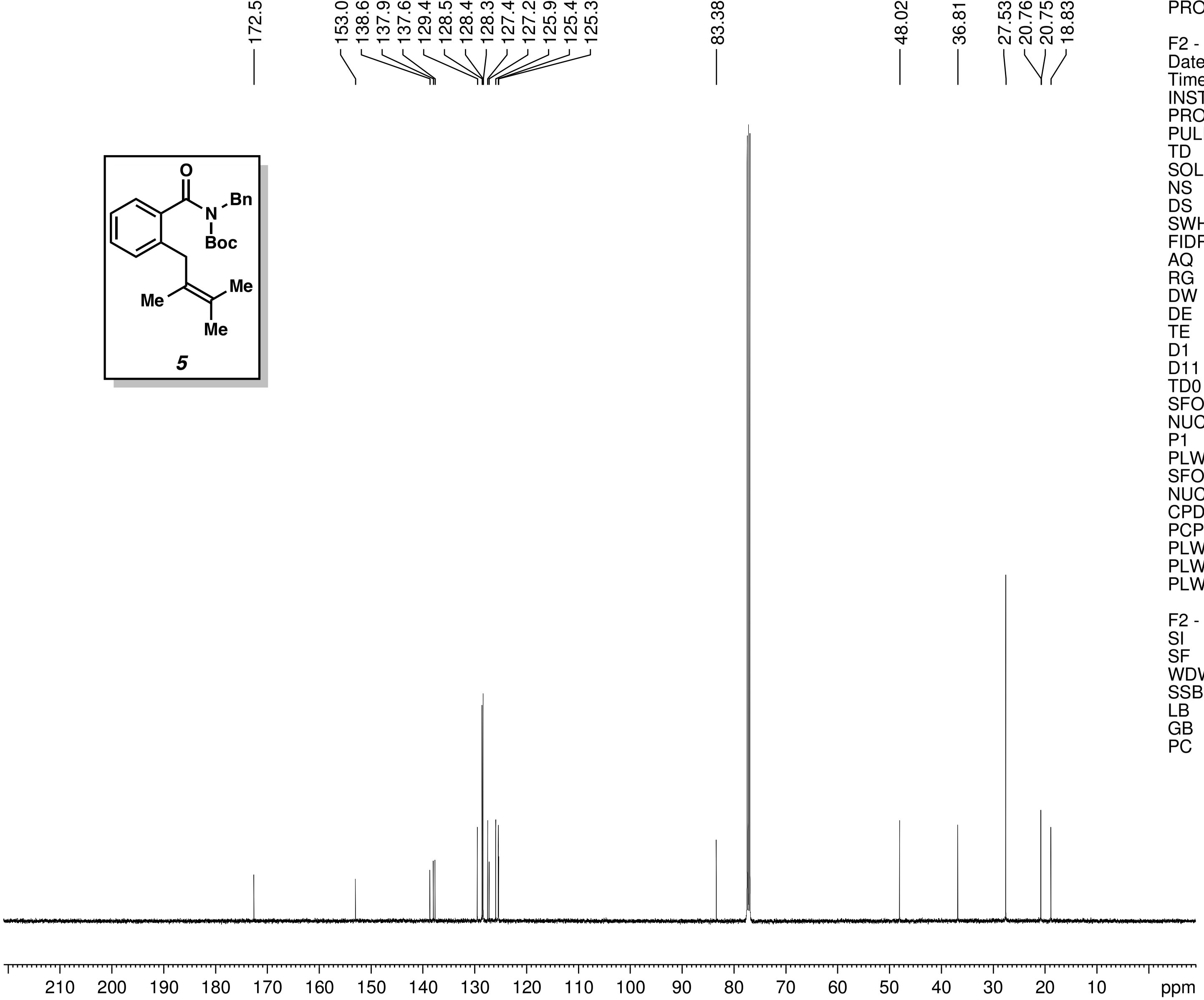
25

^{13}C NMR Spectra:

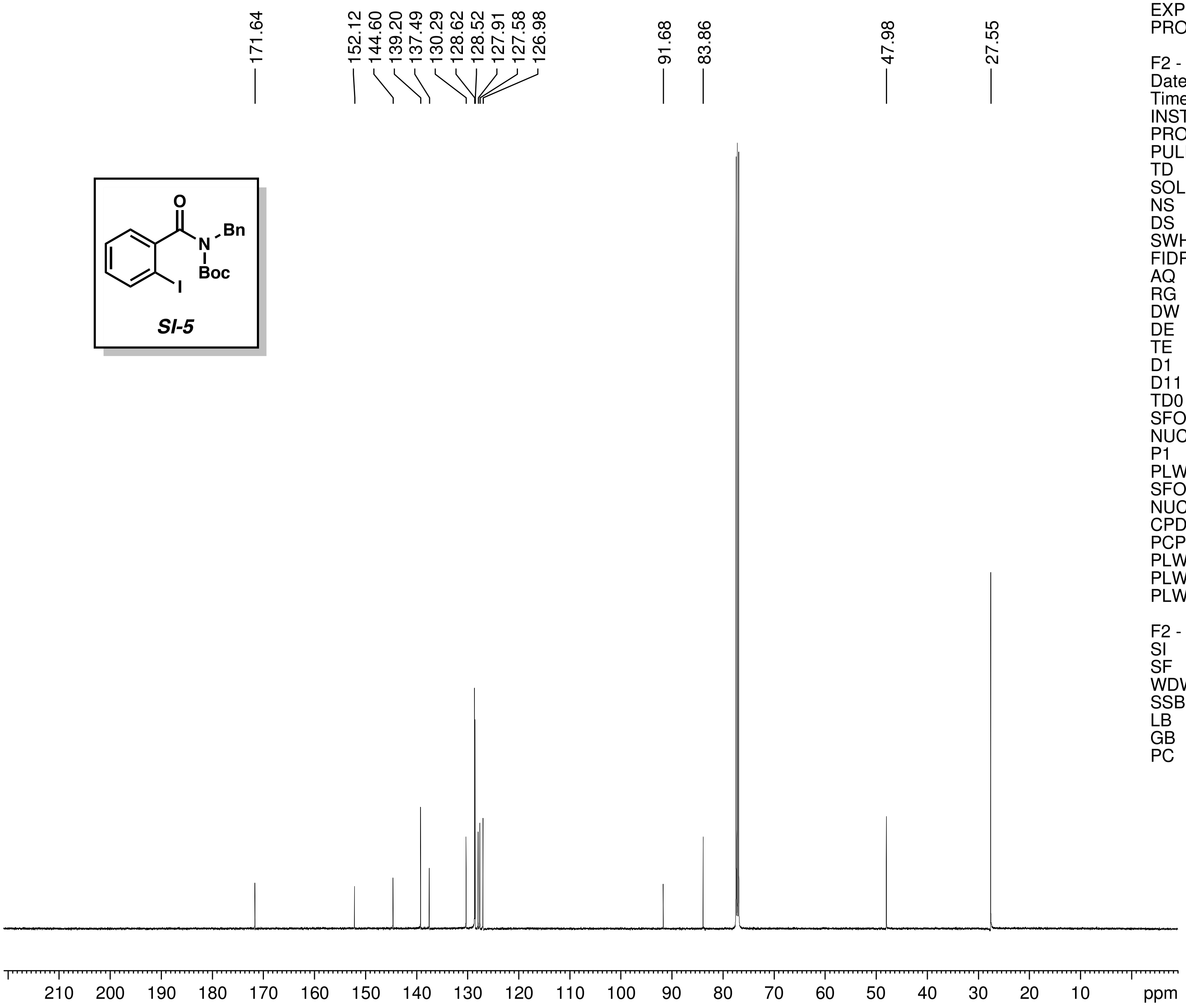


SI-3

```
Current Data Parameters
NAME          JM-5-073
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_         20170217
Time            18.26 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   2
SWH         31250.000 Hz
FIDRES       0.953674 Hz
AQ          1.0485760 sec
RG              204.54
DW              16.000 usec
DE               18.00 usec
TE               298.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1
SFO1       125.7722511 MHz
NUC1               13C
P1               10.50 usec
PLW1       23.00000000 W
SFO2       500.1330008 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2       13.50000000 W
PLW12       0.21094000 W
PLW13       0.10610000 W

F2 - Processing parameters
SI              131072
SF         125.7577764 MHz
WDW                 EM
SSB     0
LB                1.00 Hz
GB      0
PC                1.40
```

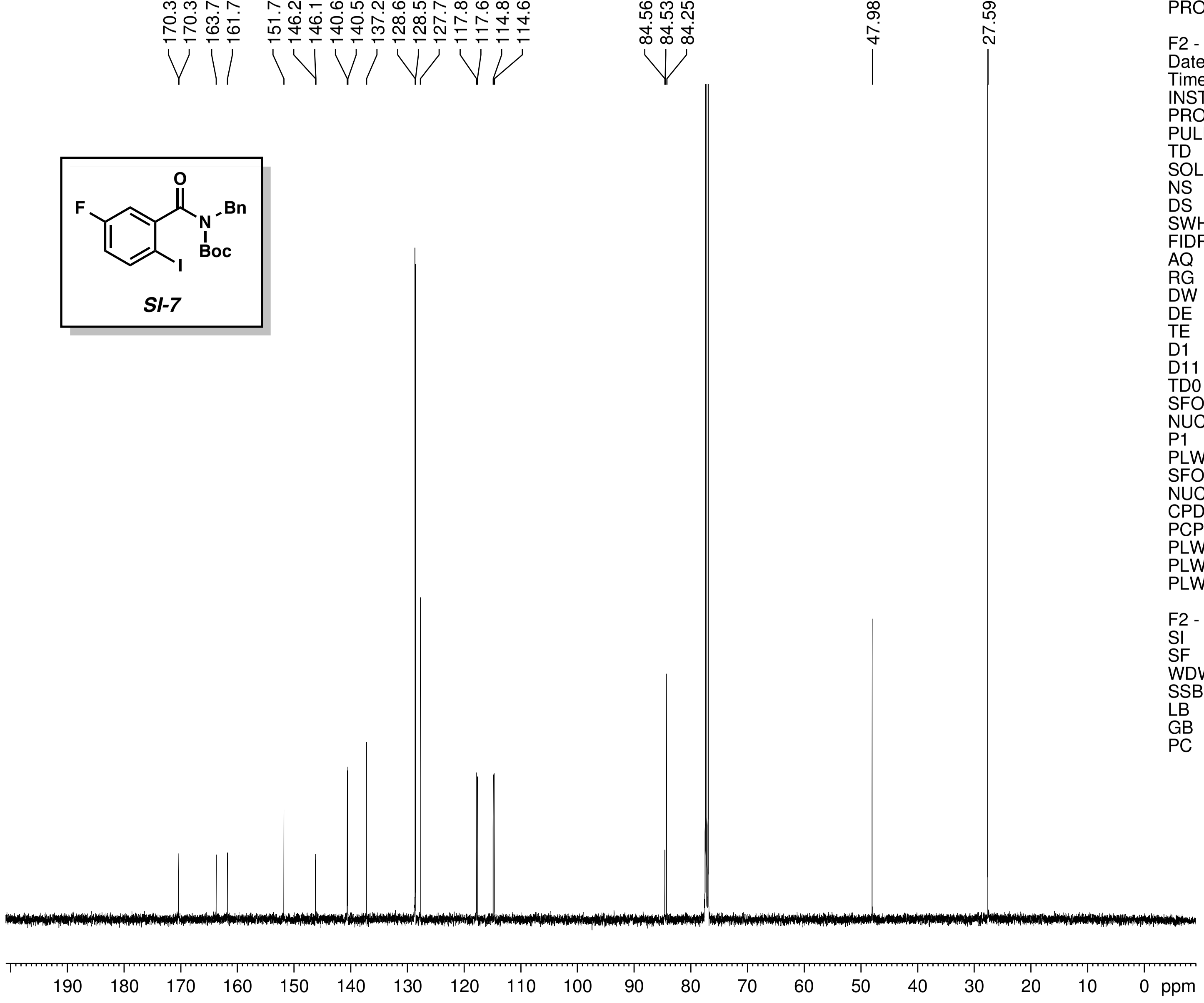


```
Current Data Parameters
NAME          SR-1-017
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_         20170109
Time           18.04 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                 17
DS                  2
SWH       31250.000 Hz
FIDRES     0.953674 Hz
AQ       1.0485760 sec
RG             204.54
DW         16.000 usec
DE          18.00 usec
TE             298.0 K
D1      2.00000000 sec
D11     0.03000000 sec
TD0                 1
SFO1    125.7722511 MHz
NUC1              13C
P1          10.50 usec
PLW1     23.00000000 W
SFO2    500.1330008 MHz
NUC2               1H
CPDPRG[2      waltz16
PCPD2       80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI             131072
SF      125.7577730 MHz
WDW                EM
SSB     0
LB            1.00 Hz
GB      0
PC               1.40
```



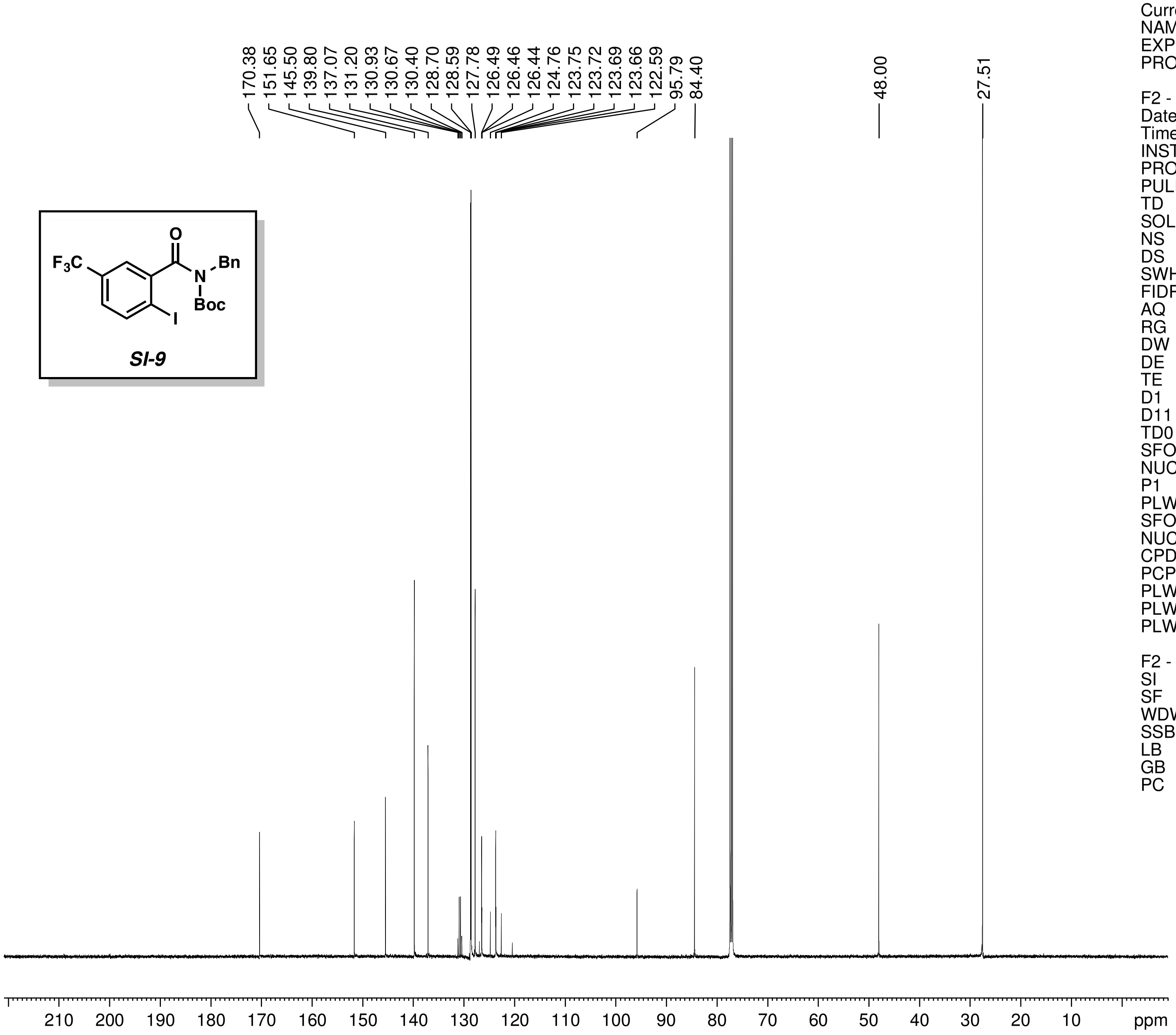
SI-5

```
Current Data Parameters
NAME          JM-4-296
EXPNO              101
PROCNO               1

F2 - Acquisition Parameters
Date_         20170115
Time           17.23 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD            65536
SOLVENT          CDCl3
NS               144
DS                 2
SWH          31250.000 Hz
FIDRES        0.953674 Hz
AQ          1.0485760 sec
RG             204.54
DW             16.000 usec
DE              18.00 usec
TE              298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                 1
SFO1      125.7722511 MHz
NUC1              13C
P1              10.50 usec
PLW1      23.00000000 W
SFO2      500.1330008 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2           80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.10610000 W

F2 - Processing parameters
SI             131072
SF        125.7577727 MHz
WDW                EM
SSB     0
LB               1.00 Hz
GB      0
PC               1.40
```



SI-7

```
Current Data Parameters
NAME          JM-5-054
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_         20170217
Time           18.33 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                   8
DS                   2
SWH       31250.000 Hz
FIDRES     0.953674 Hz
AQ       1.0485760 sec
RG              204.54
DW          16.000 usec
DE           18.00 usec
TE             298.0 K
D1      2.00000000 sec
D11     0.03000000 sec
TD0                  1
SFO1    125.7722511 MHz
NUC1               13C
P1           10.50 usec
PLW1     23.00000000 W
SFO2    500.1330008 MHz
NUC2                1H
CPDPRG[2       waltz16
PCPD2        80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI              131072
SF      125.7577738 MHz
WDW                 EM
SSB     0
LB             1.00 Hz
GB      0
PC                1.40
```



Current Data Parameters

NAME	JM-5-037
EXPNO	101
PROCNO	1

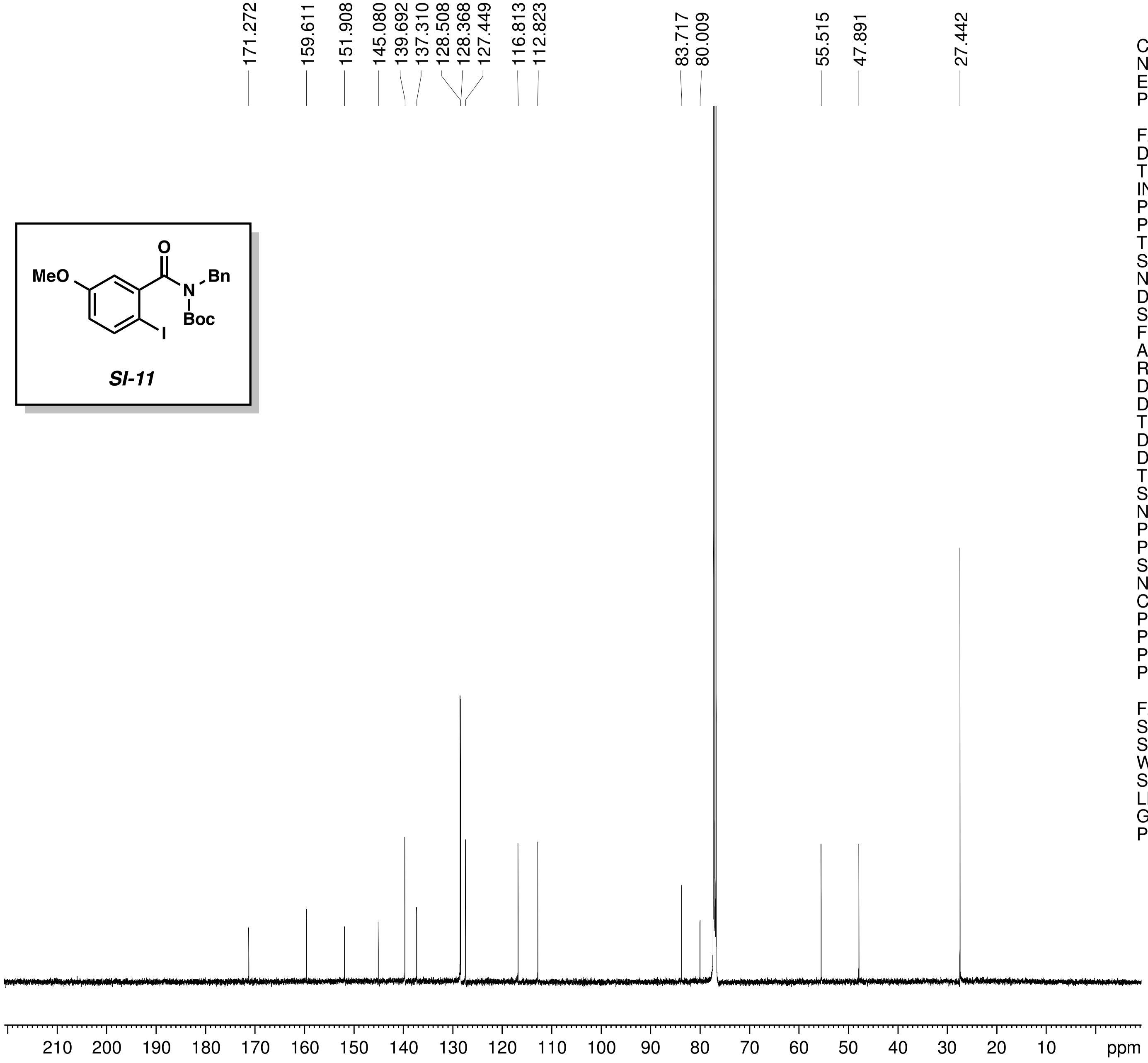
F2 - Acquisition Parameters

Date_	20170130
Time	18.24 h
INSTRUM	av500
PROBHD	Z119248_0002 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	104
DS	2
SWH	31250.000 Hz
FIDRES	0.953674 Hz
AQ	1.0485760 sec
RG	204.54
DW	16.000 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	125.7722511 MHz
NUC1	13C
P1	10.50 usec
PLW1	23.00000000 W
SFO2	500.1330008 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	80.00 usec
PLW2	13.50000000 W
PLW12	0.21094000 W
PLW13	0.10610000 W

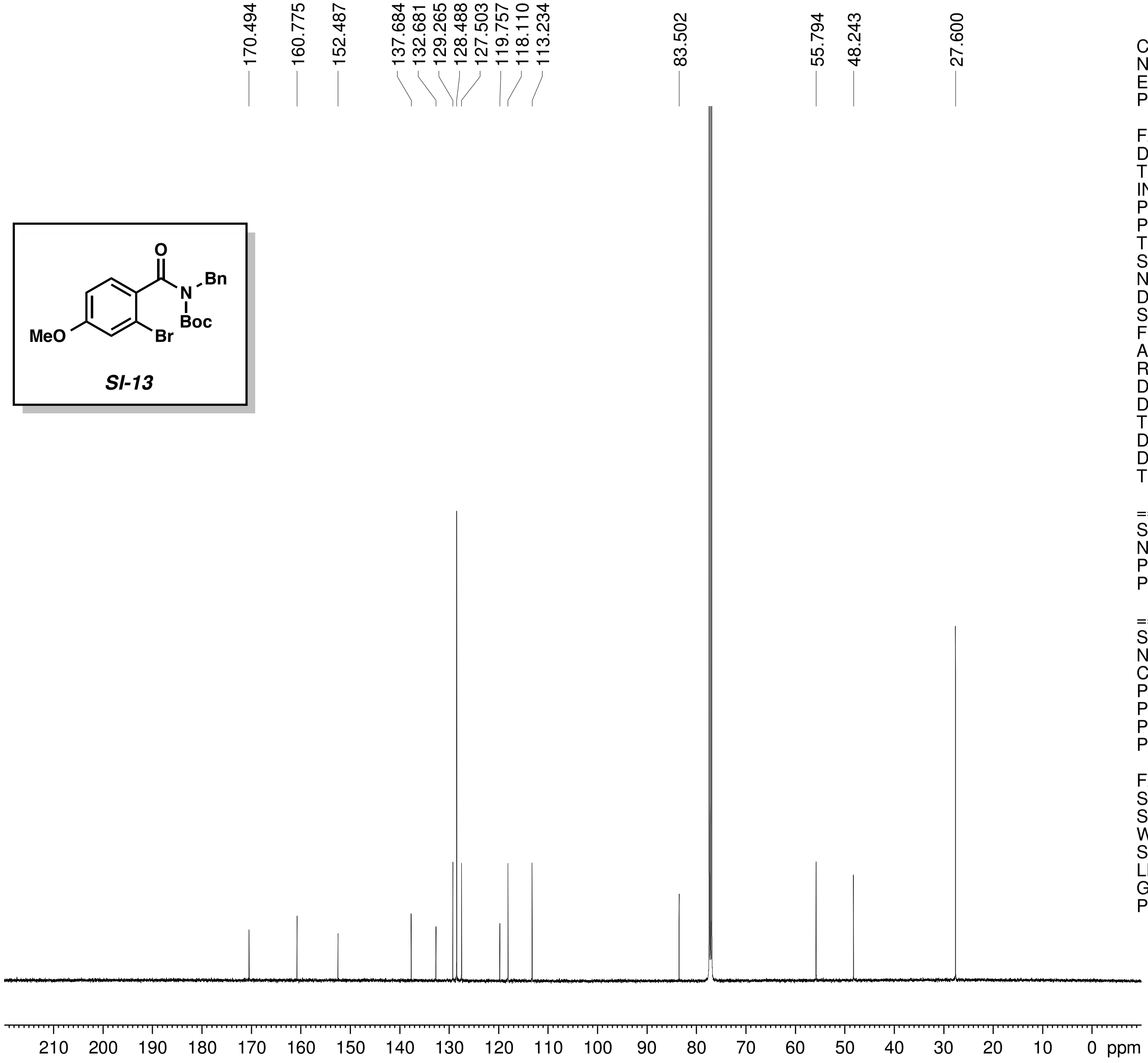
F2 - Processing parameters

SI	131072
SF	125.7577732 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

SI-11

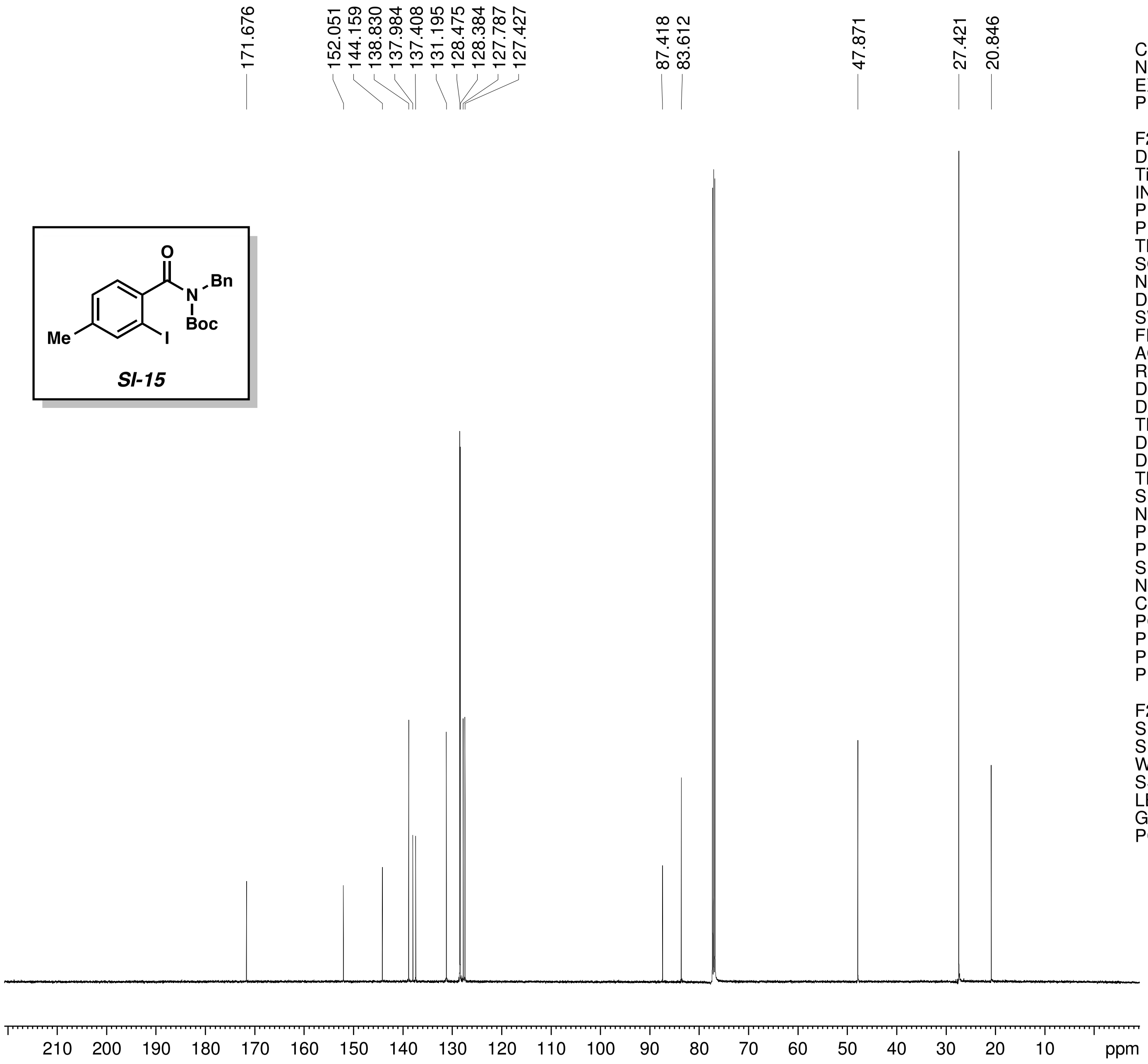


```
Current Data Parameters
NAME     JM-5-033.500.2
EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
Date_        20170130
Time             9.36 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG         zgpg30
TD             65536
SOLVENT          CDCl3
NS               128
DS                 2
SWH        31250.000 Hz
FIDRES      0.953674 Hz
AQ         1.0485760 sec
RG            204.54
DW            16.000 usec
DE             18.00 usec
TE             298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1
SFO1      125.7722511 MHz
NUC1             13C
P1             10.50 usec
PLW1      23.00000000 W
SFO2      500.1330008 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.10610000 W

F2 - Processing parameters
SI            131072
SF        125.7577916 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```

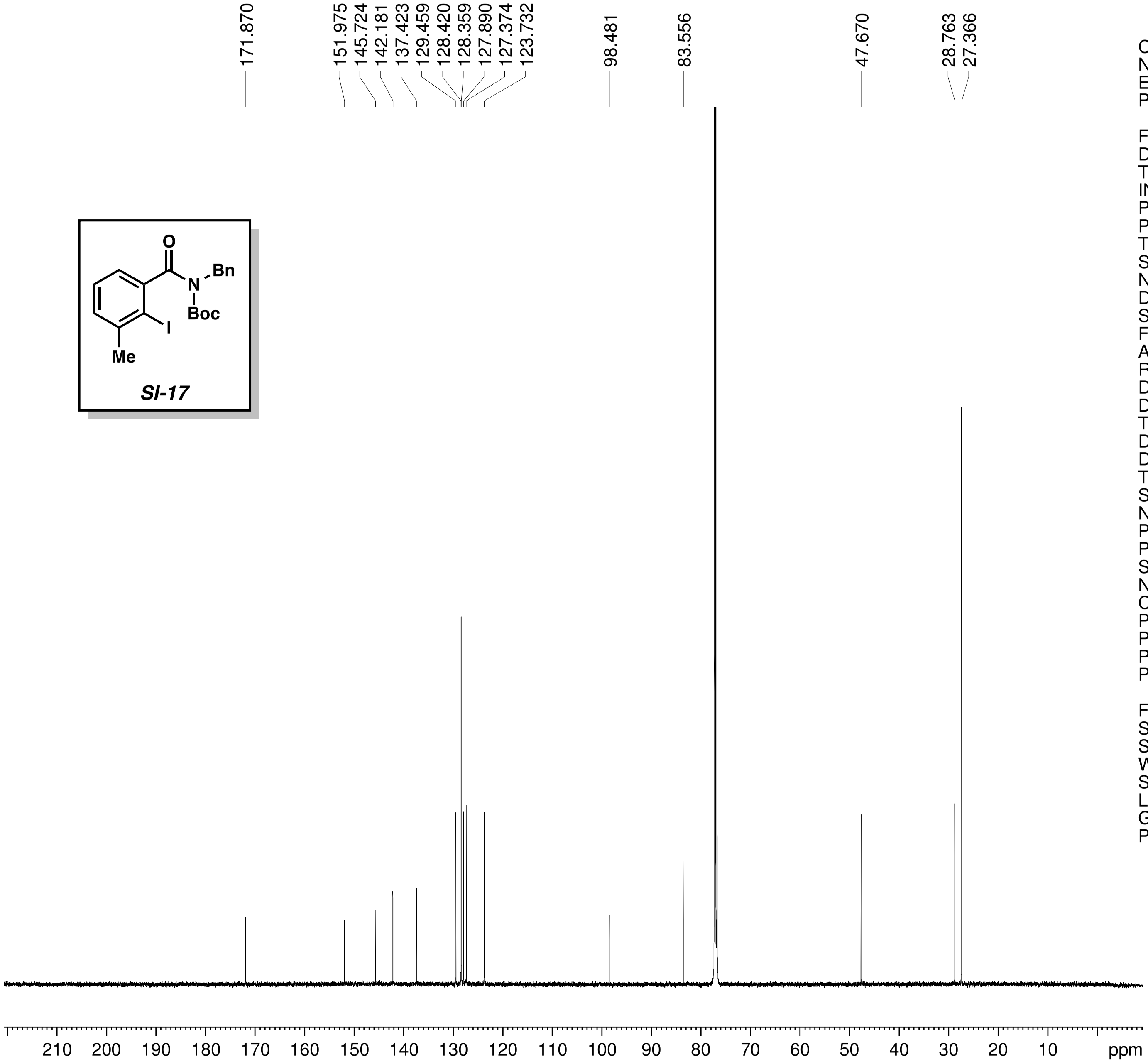


SI-13

```
Current Data Parameters
NAME        JMM-7-68-1
EXPNO              2
PROCNO           999

F2 - Acquisition Parameters
Date_        20170207
Time            19.50
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD             65536
SOLVENT         CDCl3
NS               106
DS                 2
SWH        50000.000 Hz
FIDRES      0.762939 Hz
AQ         0.6553600 sec
RG            204.54
DW            10.000 usec
DE             18.00 usec
TE             298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1

======== CHANNEL f1 =====
SFO1      125.7766527 MHz
NUC1             13C
P1              9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 =====
SFO2      500.1330008 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2           80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI            131072
SF        125.7577729 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```



```
Current Data Parameters
NAME     sr-1-116.500.11
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20170210
Time             9.09 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG          zgpg30
TD            65536
SOLVENT          CDCl3
NS               64
DS                2
SWH         31250.000 Hz
FIDRES       0.953674 Hz
AQ         1.0485760 sec
RG            204.54
DW            16.000 usec
DE             18.00 usec
TE             298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1
SFO1     125.7722511 MHz
NUC1             13C
P1             10.50 usec
PLW1     23.00000000 W
SFO2     500.1330008 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI           131072
SF        125.7577887 MHz
WDW               EM
SSB      0
LB              1.00 Hz
GB       0
PC              1.40
```



SI-17

Current Data Parameters
NAME sr-1-088
EXPNO 2
PROCNO 1

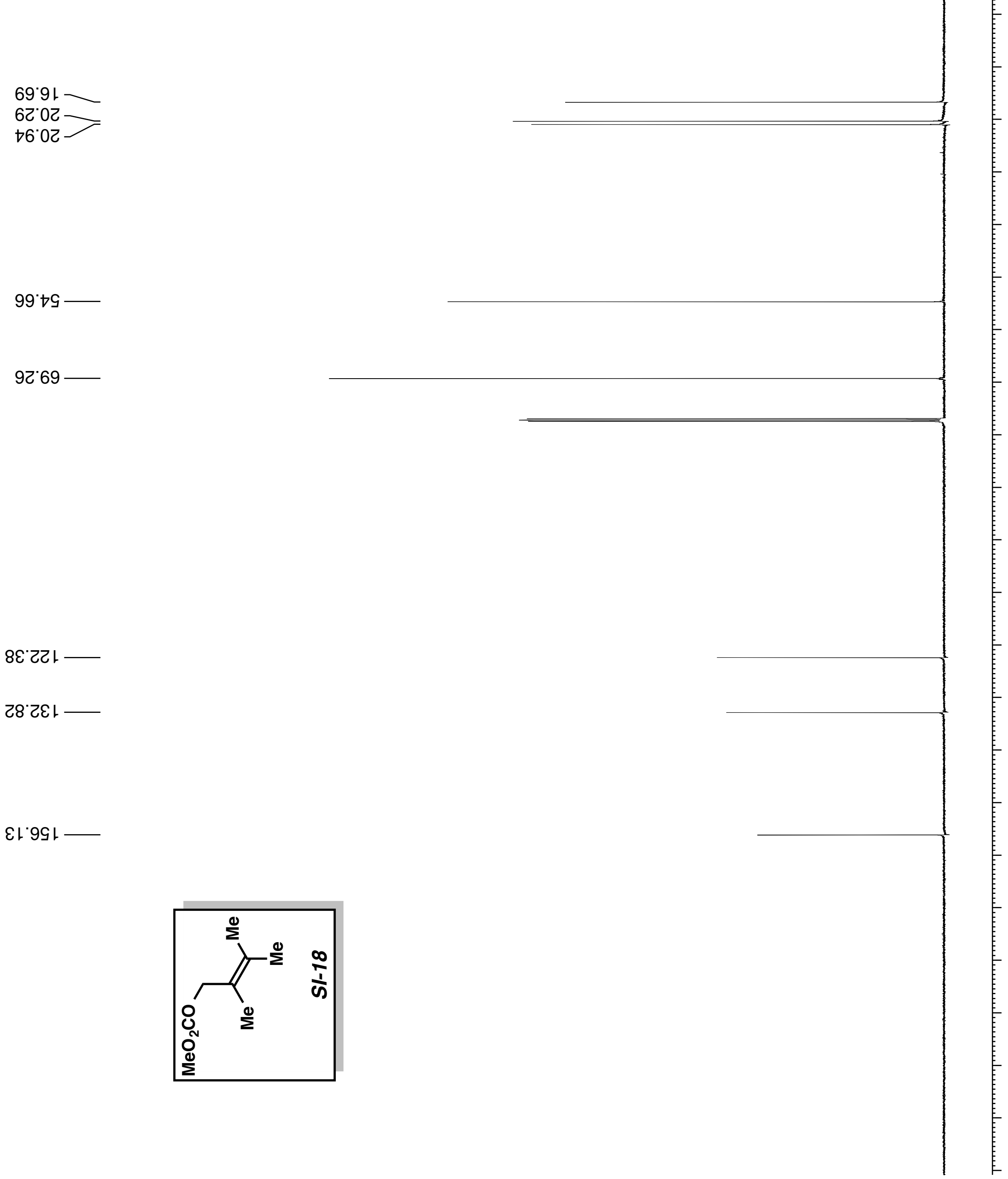
F2 - Acquisition Parameters
Date_ 20170124
Time 9.23 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 194
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W

F2 - Processing parameters
SI 131072
SF 125.7577907 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

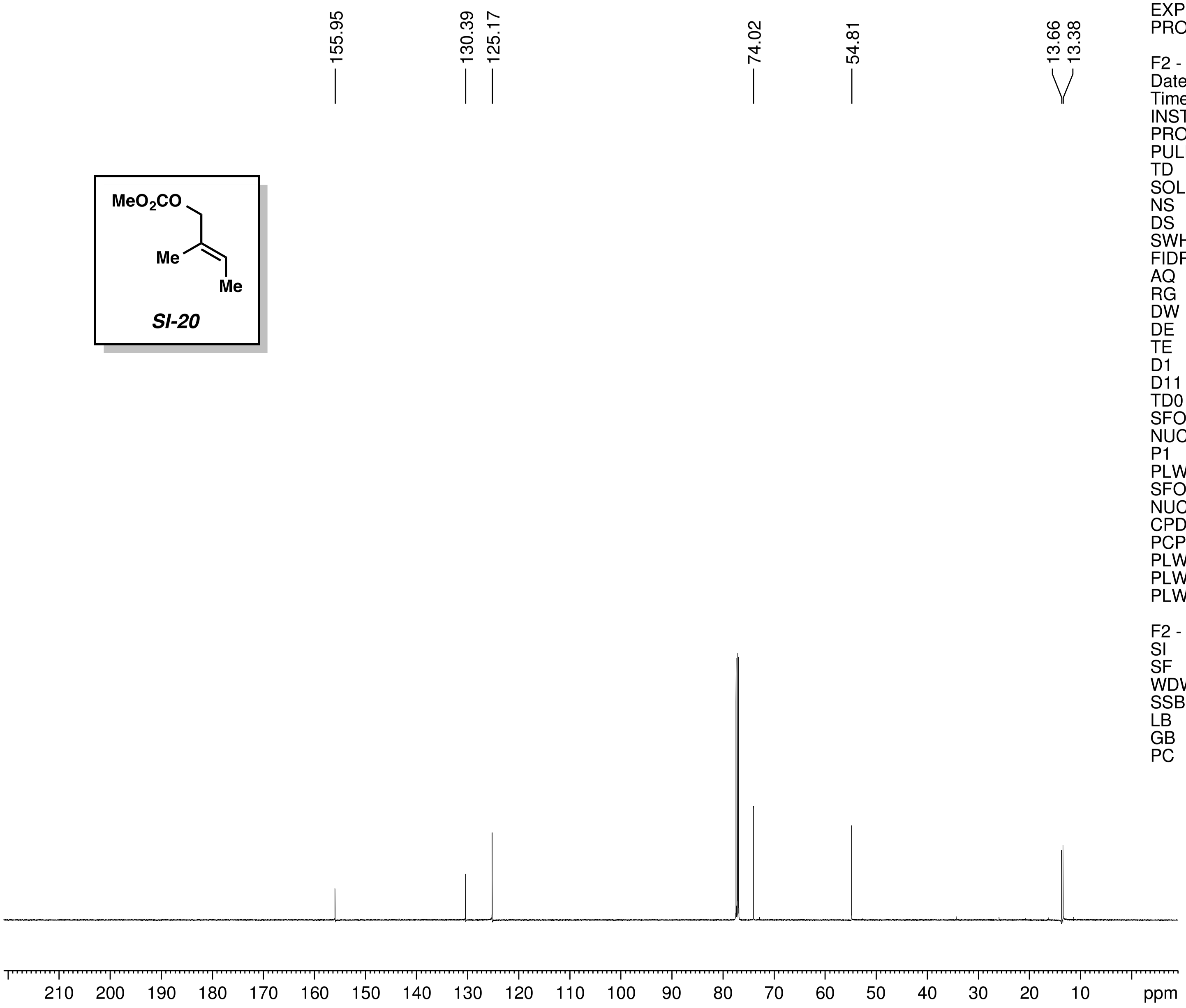
Current Data Parameters
NAME sr-1-035
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170109
Time 17.49 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W

F2 - Processing parameters
SI 131072
SF 125.7577774 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



SI-18



SI-20

Current Data Parameters

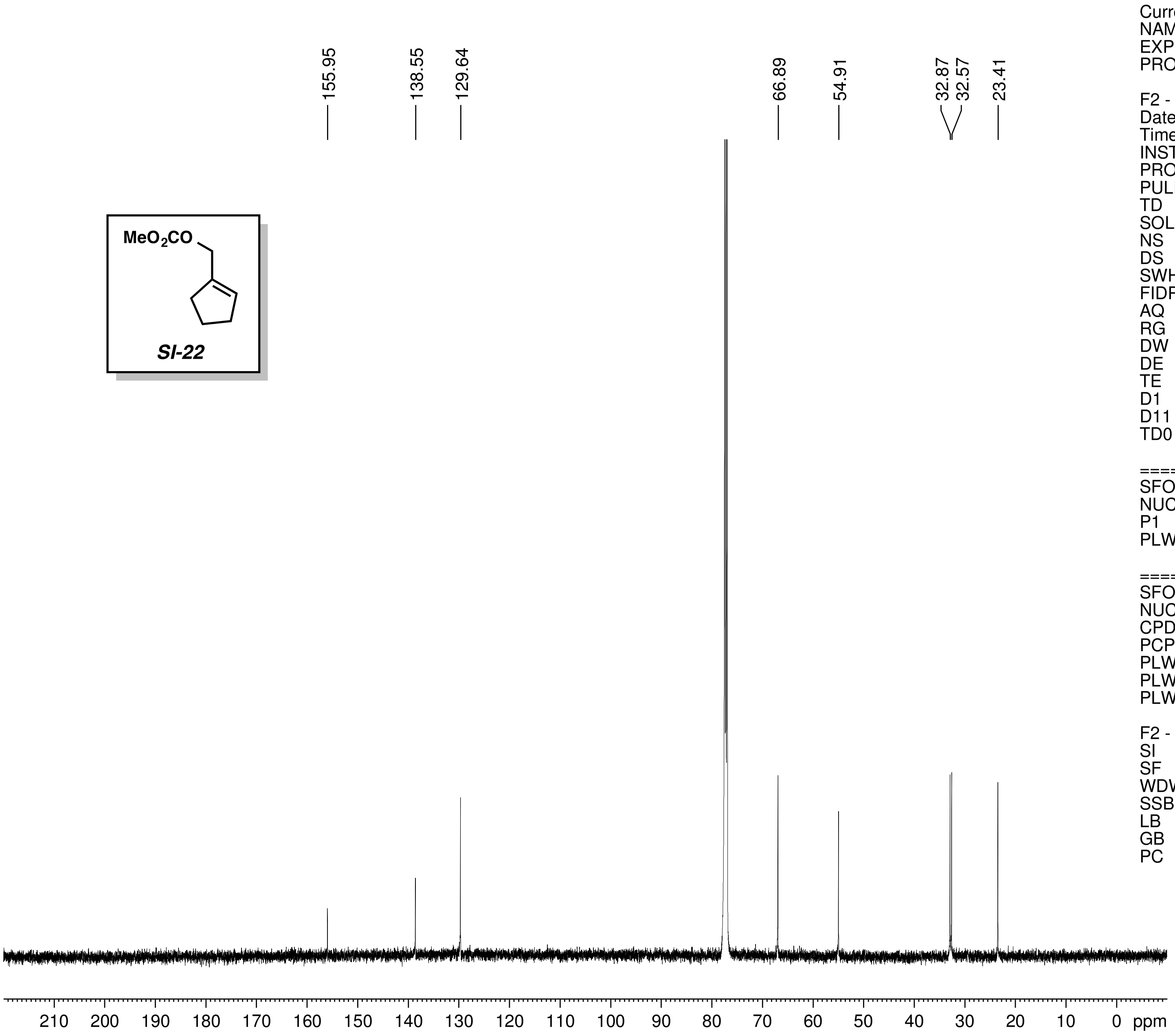
NAME	sr-1-065
EXPNO	101
PROCNO	1

F2 - Acquisition Parameters

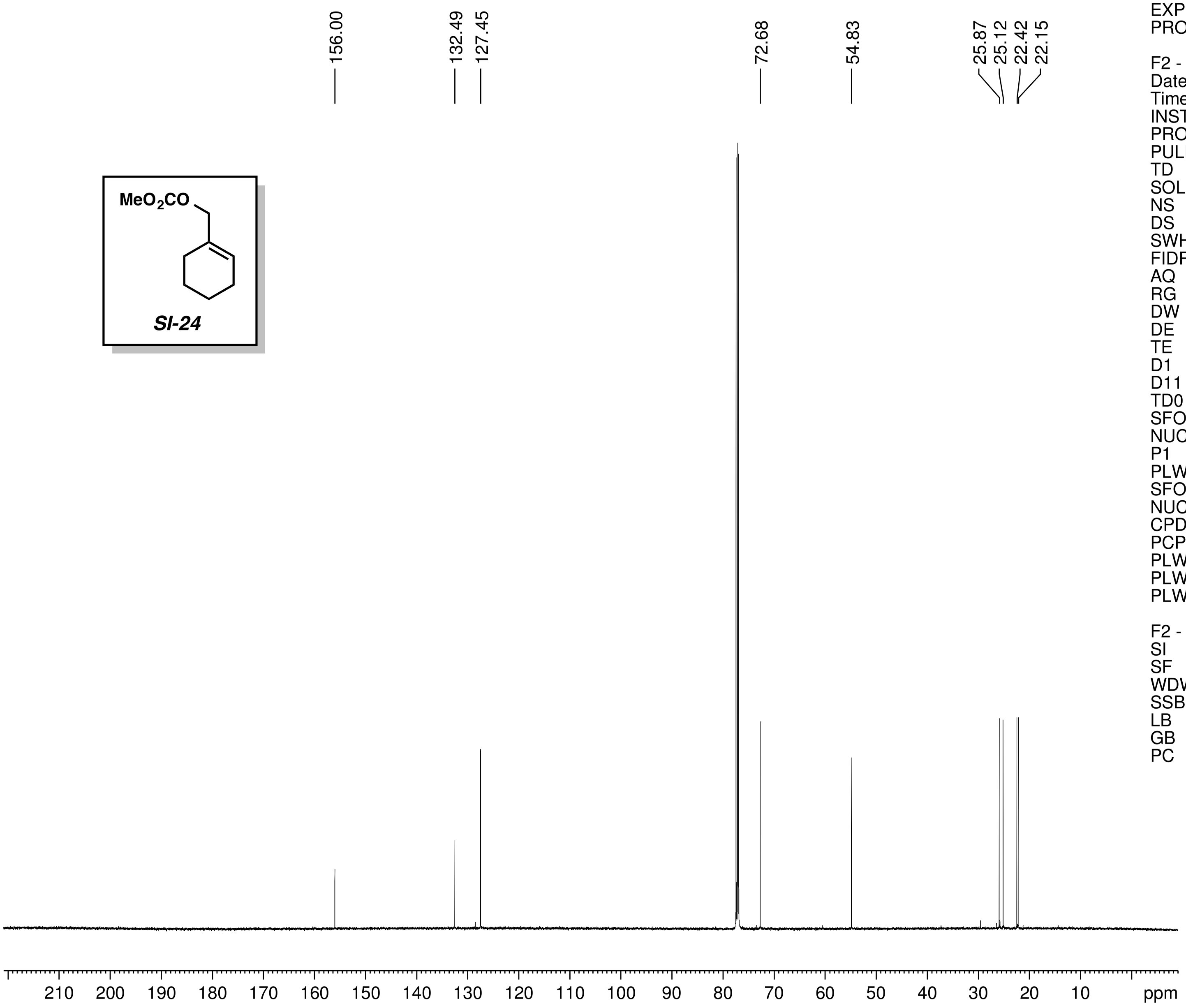
Date_	20170109
Time	17.56 h
INSTRUM	av500
PROBHD	Z119248_0002 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	31250.000 Hz
FIDRES	0.953674 Hz
AQ	1.0485760 sec
RG	204.54
DW	16.000 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	125.7722511 MHz
NUC1	13C
P1	10.50 usec
PLW1	23.00000000 W
SFO2	500.1330008 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	80.00 usec
PLW2	13.50000000 W
PLW12	0.21094000 W
PLW13	0.10610000 W

F2 - Processing parameters

SI	131072
SF	125.7577729 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



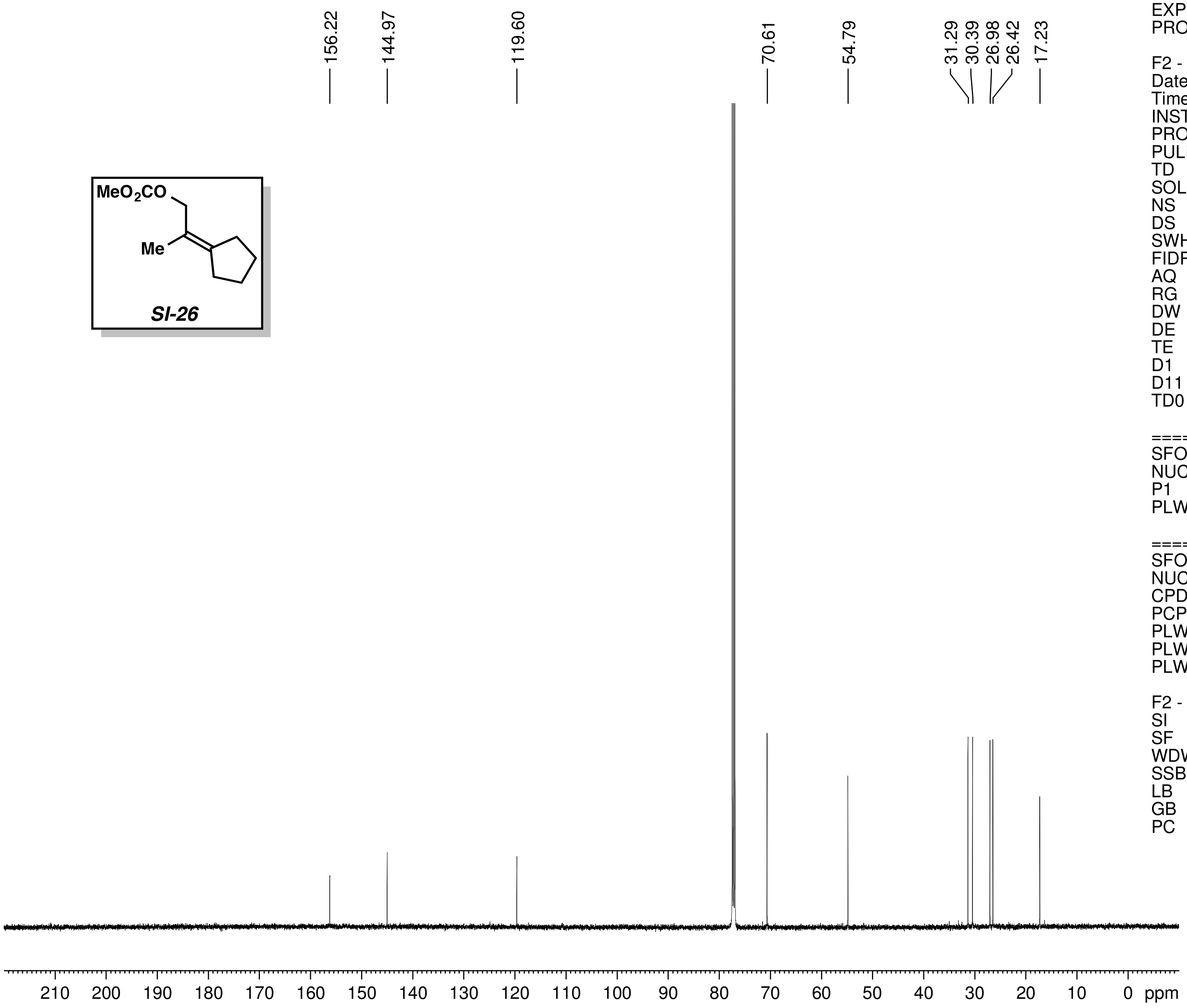
```
Current Data Parameters
NAME      JMM-7-216char
EXPNO              2
PROCNO           999

F2 - Acquisition Parameters
Date_        20170108
Time           10.11
INSTRUM        av500
PROBHD   5 mm DCH 13C-1
PULPROG       zgpg30
TD           65536
SOLVENT         CDCl3
NS               115
DS                 2
SWH        50000.000 Hz
FIDRES      0.762939 Hz
AQ         0.6553600 sec
RG            204.54
DW            10.000 usec
DE             18.00 usec
TE             298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1

======== CHANNEL f1 =====
SFO1      125.7766527 MHz
NUC1             13C
P1              9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 =====
SFO2      500.1330008 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI           131072
SF        125.7577718 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```



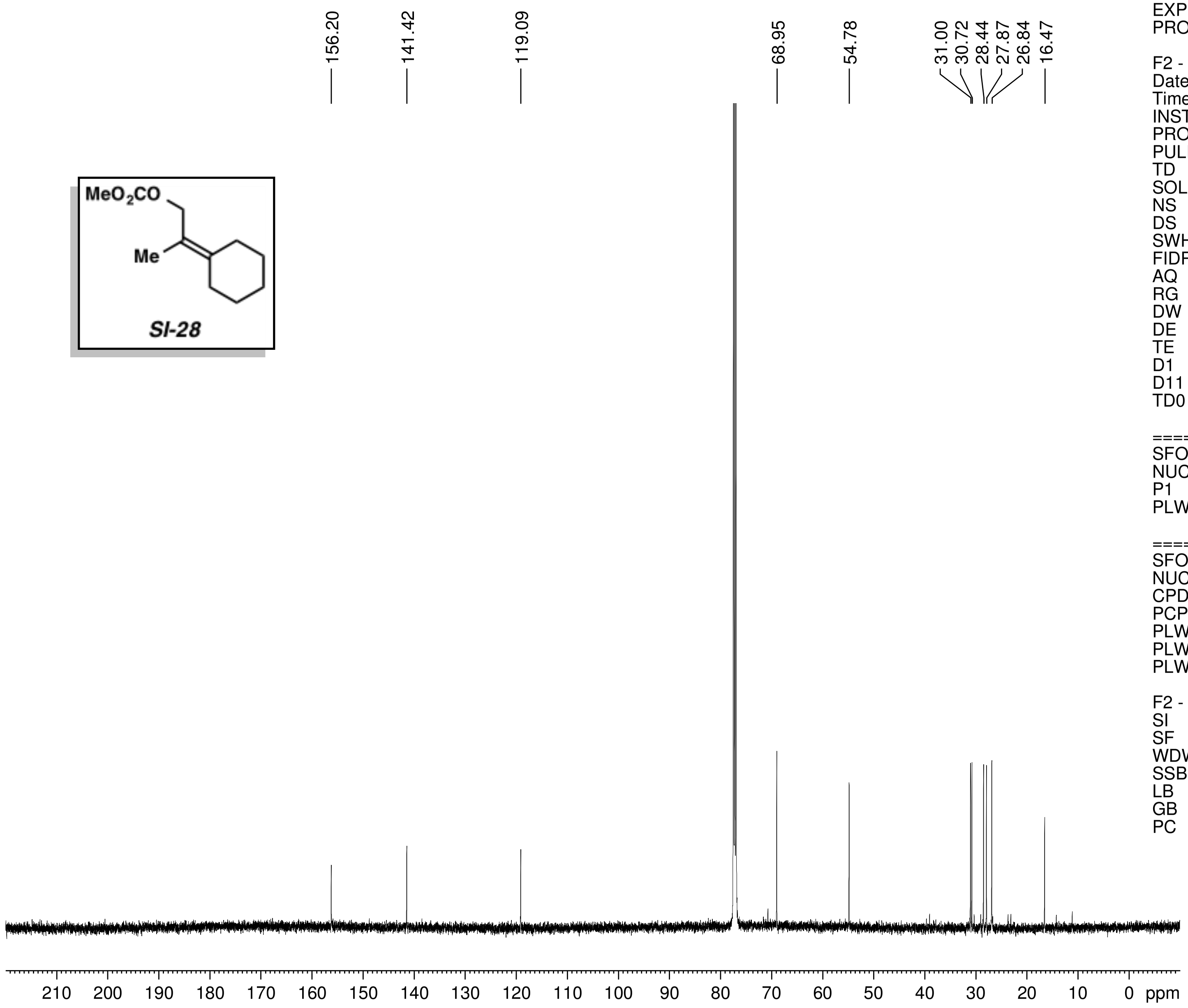
SI-24

```
Current Data Parameters
NAME          JM-4-293
EXPNO              101
PROCNO               1

F2 - Acquisition Parameters
Date_         20161219
Time           18.26 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD             65536
SOLVENT          CDCl3
NS                  48
DS                   2
SWH        31250.000 Hz
FIDRES      0.953674 Hz
AQ        1.0485760 sec
RG              204.54
DW          16.000 usec
DE           18.00 usec
TE             298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0                  1
SFO1    125.7722511 MHz
NUC1               13C
P1           10.50 usec
PLW1     23.00000000 W
SFO2    500.1330008 MHz
NUC2                1H
CPDPRG[2       waltz16
PCPD2        80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI              131072
SF      125.7577724 MHz
WDW                 EM
SSB      0
LB             1.00 Hz
GB       0
PC                1.40
```

SI-26

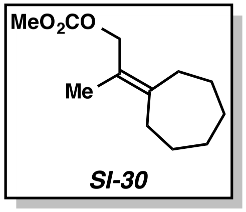


```
Current Data Parameters
NAME       JMM-7-192-3
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20161123
Time            14.09
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD             65536
SOLVENT         CDCl3
NS                67
DS                 2
SWH        43859.648 Hz
FIDRES      0.669245 Hz
AQ         0.7471104 sec
RG            204.54
DW            11.400 usec
DE             18.00 usec
TE             298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1

======== CHANNEL f1 =====
SFO1     125.7728799 MHz
NUC1              13C
P1               9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 =====
SFO2     500.1330008 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2           80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI            131072
SF       125.7577719 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```

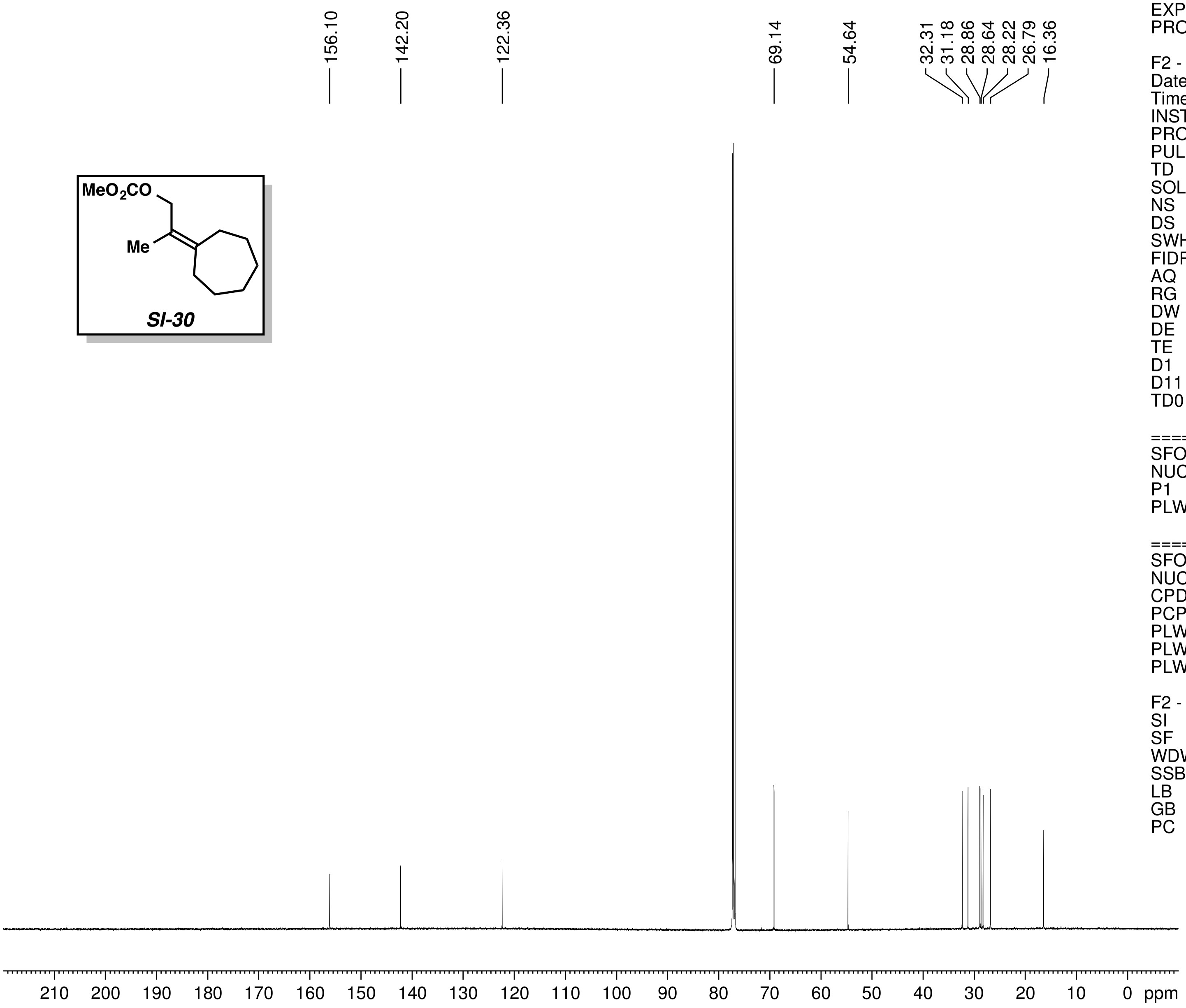


SI-28

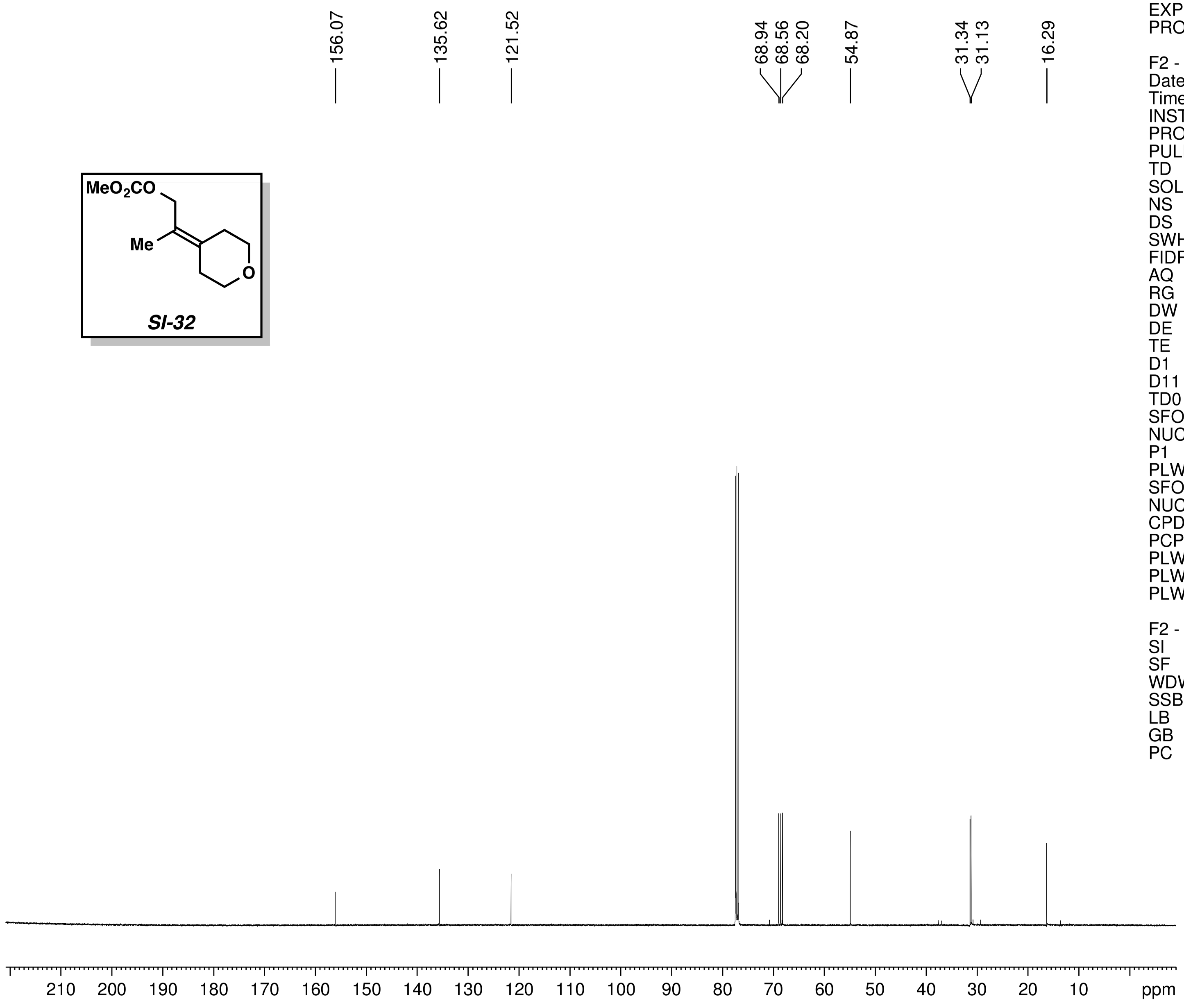
```
Current Data Parameters
NAME        JMM-7-193-1
EXPNO               2
PROCNO              1

F2 - Acquisition Parameters
Date_         20161123
Time           12.24
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD            65536
SOLVENT         CDCl3
NS               106
DS                 2
SWH        43859.648 Hz
FIDRES      0.669245 Hz
AQ         0.7471104 sec
RG            204.54
DW            11.400 usec
DE            100.00 usec
TE             298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1

======== CHANNEL f1 =====
SFO1      125.7728799 MHz
NUC1             13C
P1              9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 =====
SFO2      500.1330008 MHz
NUC2              1H
CPDPRG[2        waltz16
PCPD2           80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI            131072
SF        125.7577713 MHz
WDW               EM
SSB     0
LB             1.00 Hz
GB      0
PC              1.40
```



SI-30

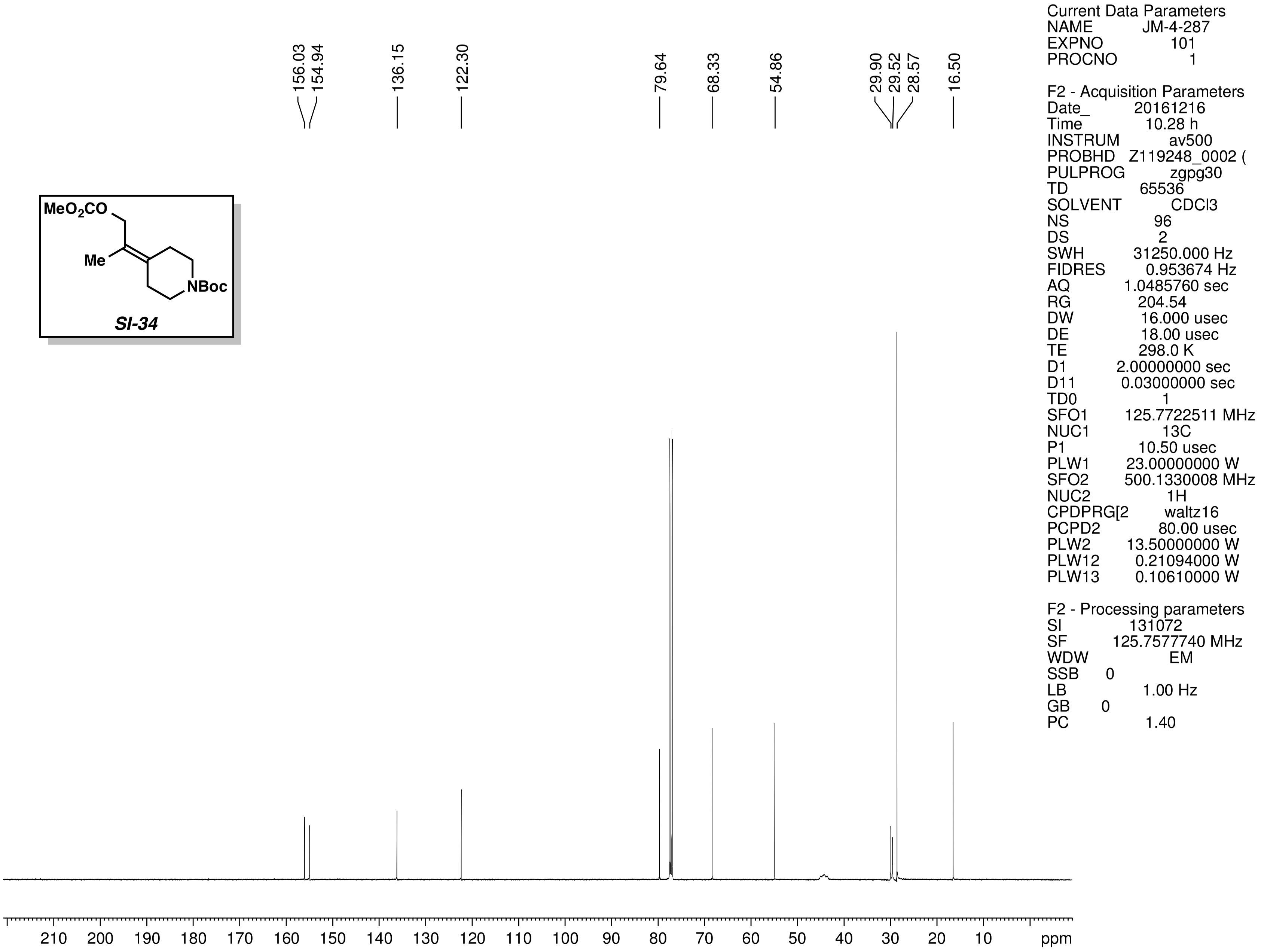


```
Current Data Parameters
NAME         JMM-7-226
EXPNO              2
PROCNO           999

F2 - Acquisition Parameters
Date_       20170116
Time           10.51
INSTRUM        av500
PROBHD   5 mm DCH 13C-1
PULPROG       zgpg30
TD           65536
SOLVENT        CDCl3
NS             103
DS               2
SWH        50000.000 Hz
FIDRES      0.762939 Hz
AQ        0.6553600 sec
RG           204.54
DW           10.000 usec
DE           100.00 usec
TE           298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0              1

======== CHANNEL f1 =====
SFO1      125.7766527 MHz
NUC1             13C
P1             9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 =====
SFO2      500.1330008 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI           131072
SF        125.7577892 MHz
WDW              EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```



SI-32

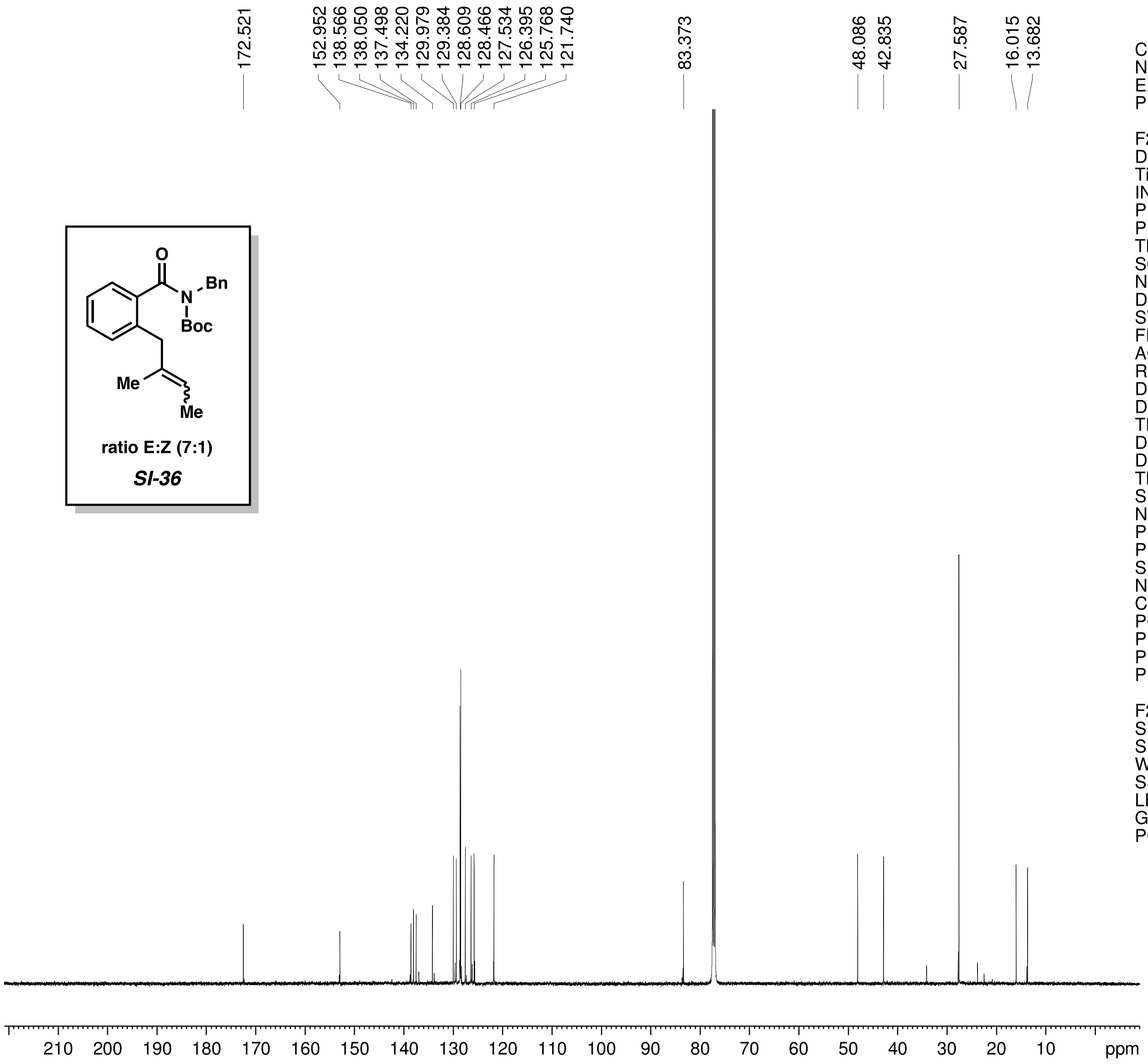
```
Current Data Parameters
NAME          JM-4-281
EXPNO              101
PROCNO               1

F2 - Acquisition Parameters
Date_         20161216
Time           10.39 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD            65536
SOLVENT          CDCl3
NS                48
DS                 2
SWH         31250.000 Hz
FIDRES       0.953674 Hz
AQ         1.0485760 sec
RG            204.54
DW            16.000 usec
DE             18.00 usec
TE             298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0                1
SFO1     125.7722511 MHz
NUC1             13C
P1             10.50 usec
PLW1      23.00000000 W
SFO2     500.1330008 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.10610000 W

F2 - Processing parameters
SI            131072
SF       125.7577729 MHz
WDW               EM
SSB    0
LB              1.00 Hz
GB     0
PC              1.40
```


MeO2CO
Me
NBoc
156.03
154.94
136.15
122.30
79.64
68.33
54.86
29.90
29.52
28.57
16.50
Current Data Parameters
NAME JM-4-287
EXPNO 101
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161216
Time 10.28 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 96
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577740 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

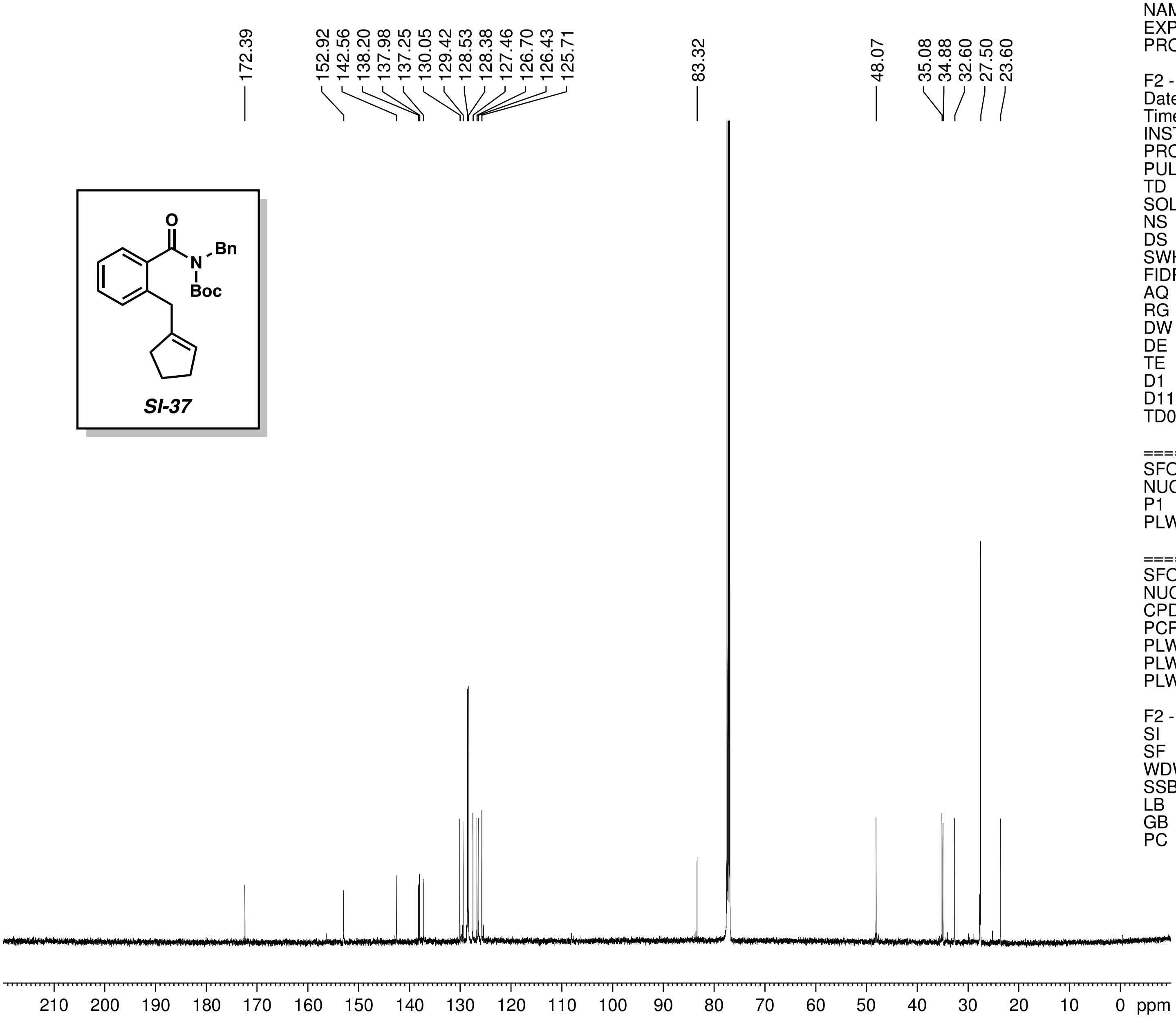
SI-34

ratio E:Z (7:1)

SI-36

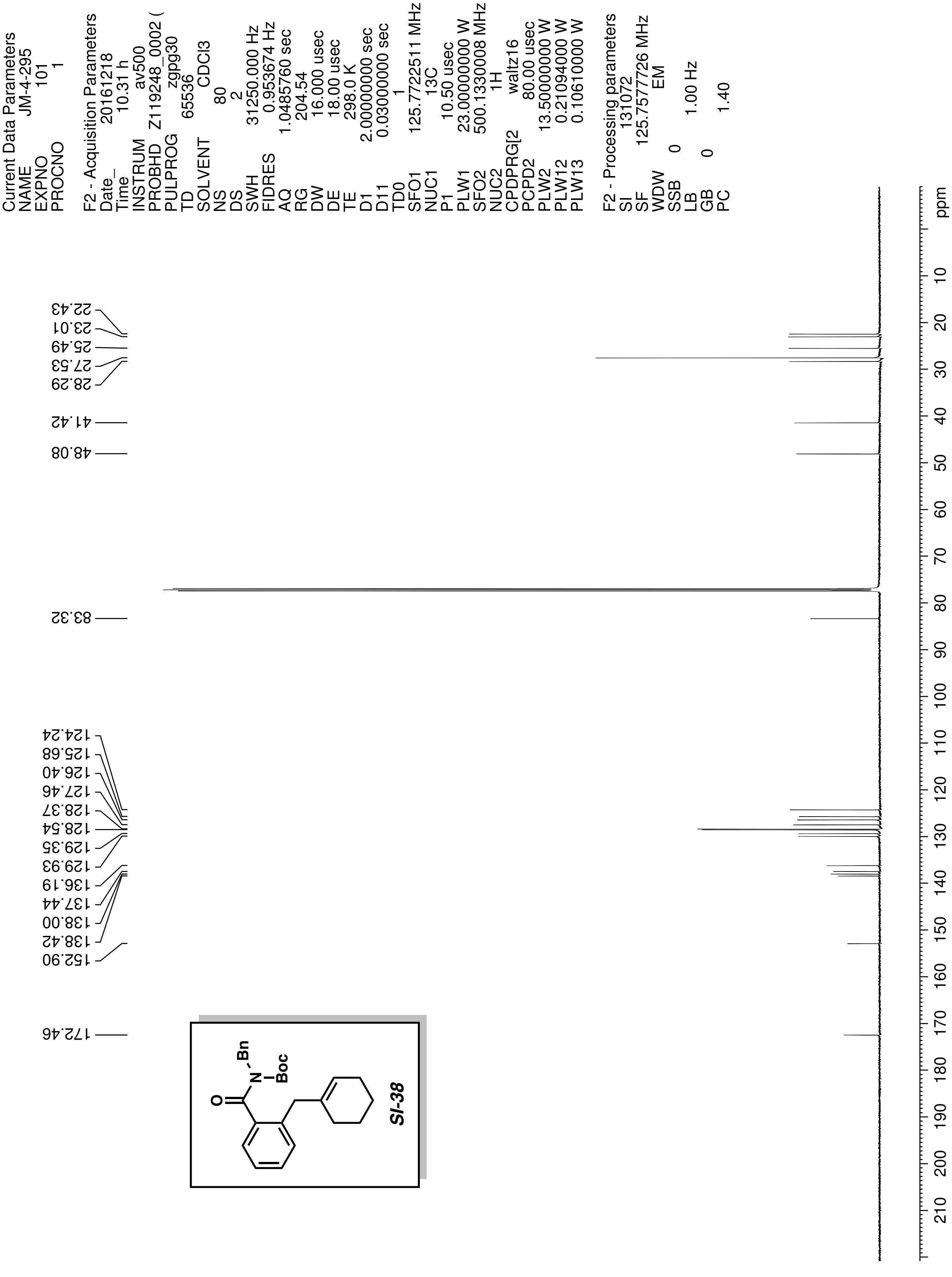


```
Current Data Parameters
NAME       sr-1-099.500
EXPNO               4
PROCNO              1

F2 - Acquisition Parameters
Date_         20170130
Time              9.21 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG         zgpg30
TD              65536
SOLVENT          CDCl3
NS                256
DS                  2
SWH          31250.000 Hz
FIDRES        0.953674 Hz
AQ          1.0485760 sec
RG              204.54
DW              16.000 usec
DE               18.00 usec
TE               298.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                 1
SFO1       125.7722511 MHz
NUC1               13C
P1               10.50 usec
PLW1       23.00000000 W
SFO2       500.1330008 MHz
NUC2                1H
CPDPRG[2       waltz16
PCPD2            80.00 usec
PLW2       13.50000000 W
PLW12       0.21094000 W
PLW13       0.10610000 W

F2 - Processing parameters
SI              131072
SF         125.7577640 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```

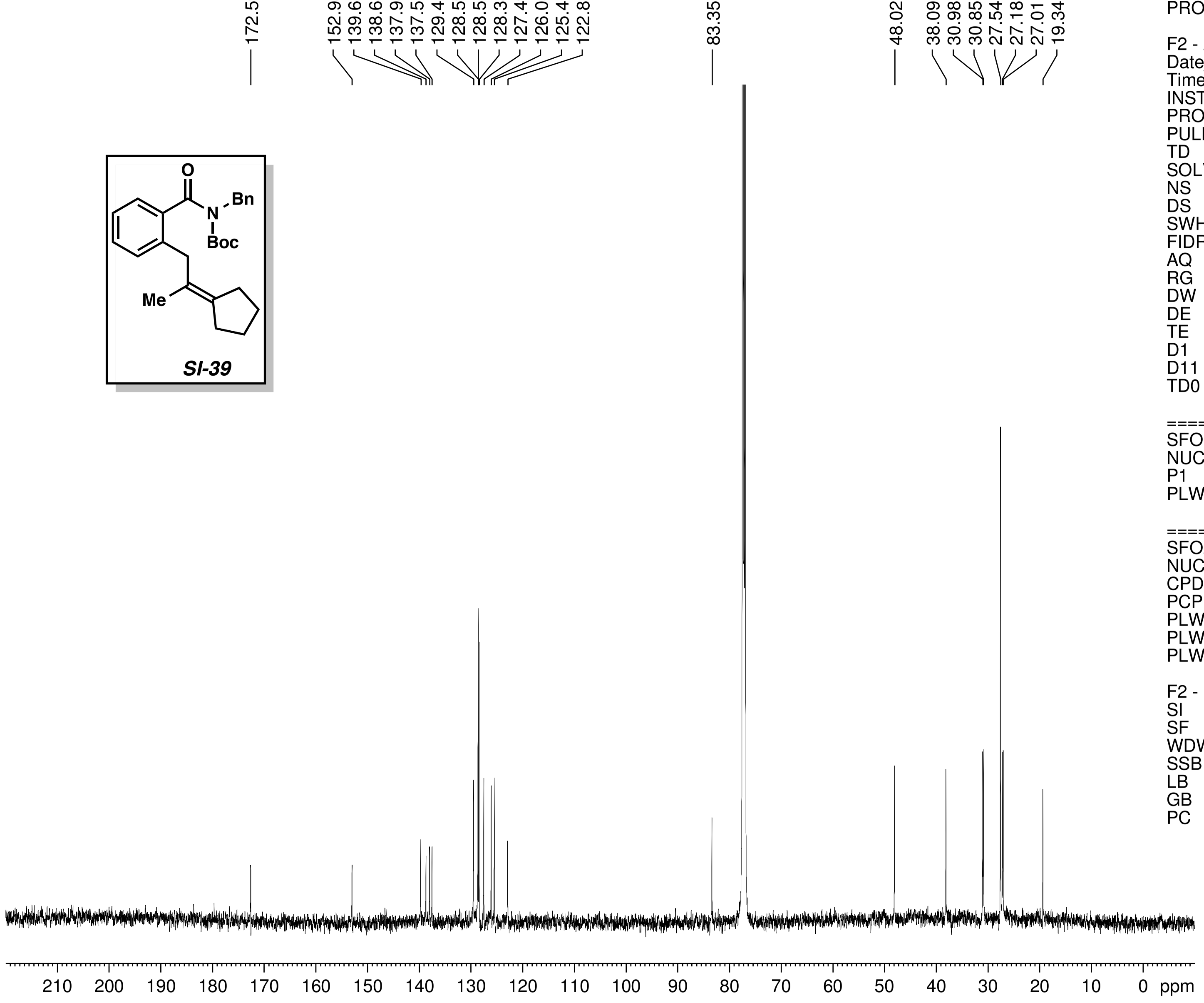


SI-37

```
Current Data Parameters
NAME     JMM-7-221-char
EXPNO              2
PROCNO           999

F2 - Acquisition Parameters
Date_        20170131
Time            10.14
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG       zgpg30
TD          65536
SOLVENT         CDCl3
NS              86
DS               2
SWH         50000.000 Hz
FIDRES       0.762939 Hz
AQ         0.6553600 sec
RG           204.54
DW            10.000 usec
DE            18.00 usec
TE            298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0              1

======== CHANNEL f1 =====
SFO1      125.7766527 MHz
NUC1            13C
P1             9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 =====
SFO2      500.1330008 MHz
NUC2             1H
CPDPRG[2      waltz16
PCPD2          80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI          131072
SF        125.7577730 MHz
WDW              EM
SSB     0
LB             1.00 Hz
GB      0
PC             1.40
```


SI-38

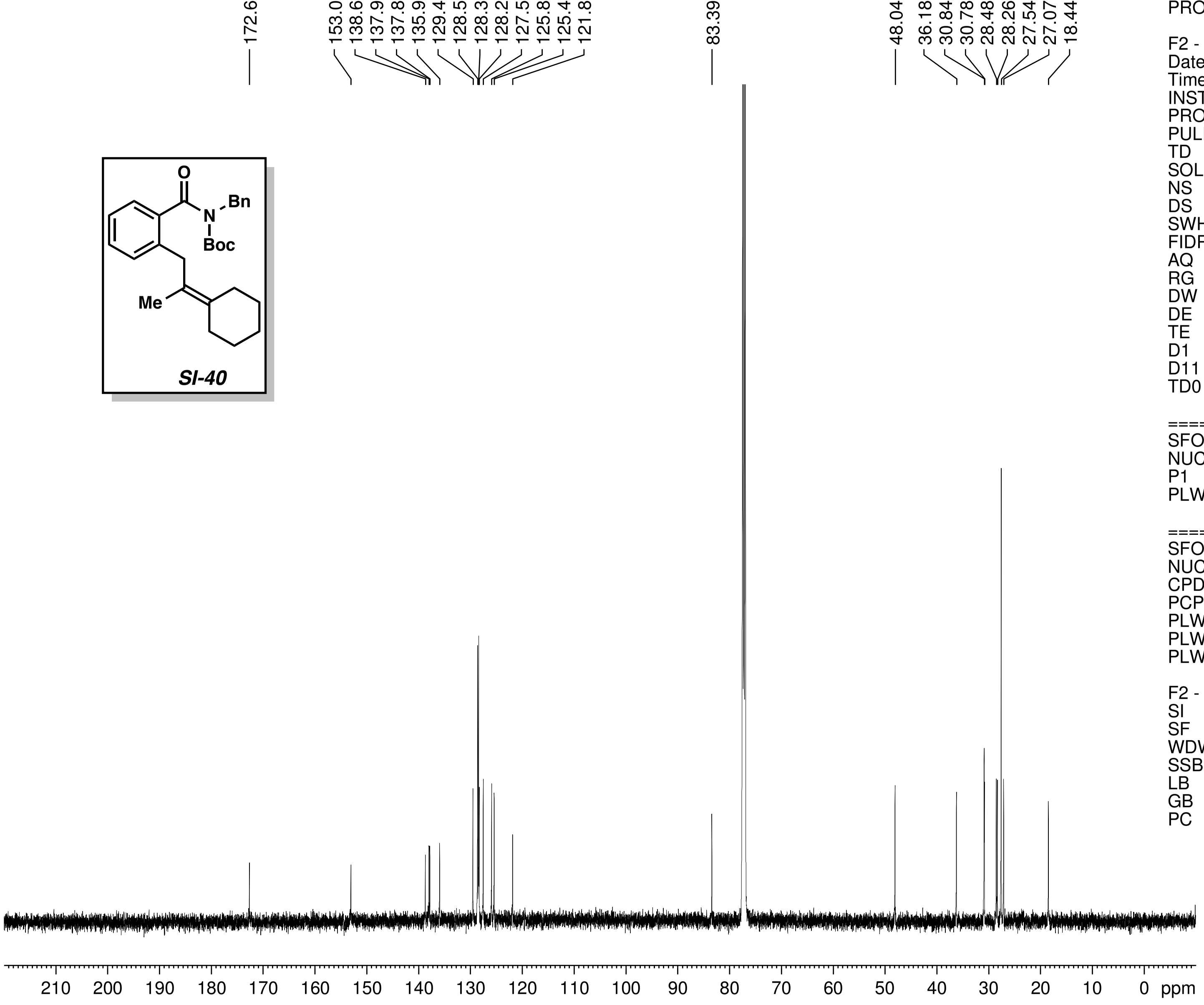
Current Data Parameters
NAME JM-4-295
EXPNO 101
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161218
Time 10.31 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 80
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577726 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
22.43
23.01
25.49
27.53
28.29
41.42
48.08
83.32
124.24
125.68
126.40
127.46
128.37
128.54
129.35
129.93
136.19
137.44
138.00
138.42
152.90
172.46
O
N
Bn
Boc
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210



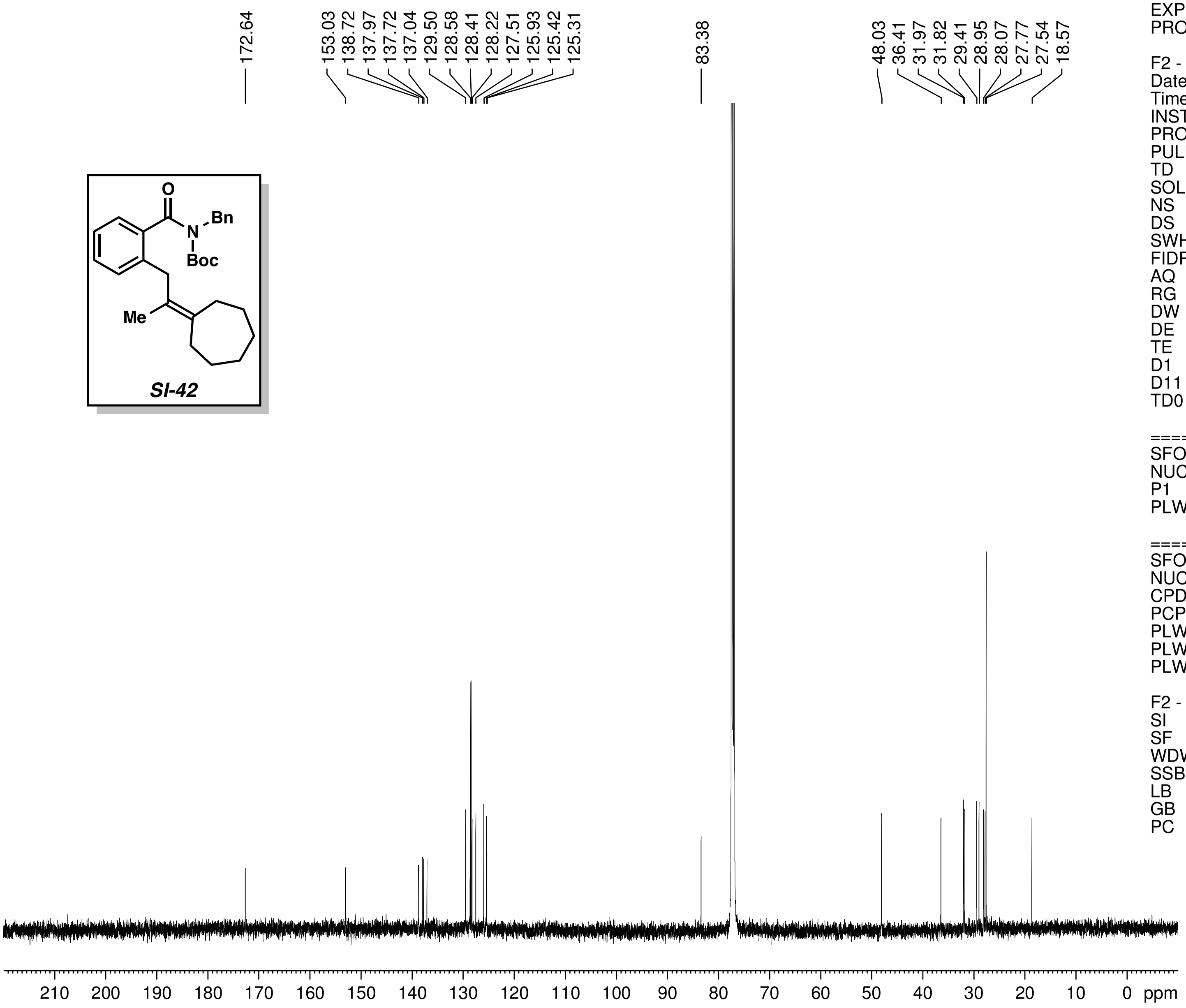
SI-39

```
Current Data Parameters
NAME       JMM-7-134(c)
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20160907
Time             9.36
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD           65536
SOLVENT         CDCl3
NS               71
DS                2
SWH        43859.648 Hz
FIDRES      0.669245 Hz
AQ         0.7471104 sec
RG           204.54
DW            11.400 usec
DE           100.00 usec
TE            298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0               1

======== CHANNEL f1 =====
SFO1      125.7741375 MHz
NUC1             13C
P1              9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 =====
SFO2      500.1330008 MHz
NUC2              1H
CPDPRG[2        waltz16
PCPD2          80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI            131072
SF        125.7577720 MHz
WDW               EM
SSB     0
LB              3.00 Hz
GB      0
PC              1.40
```

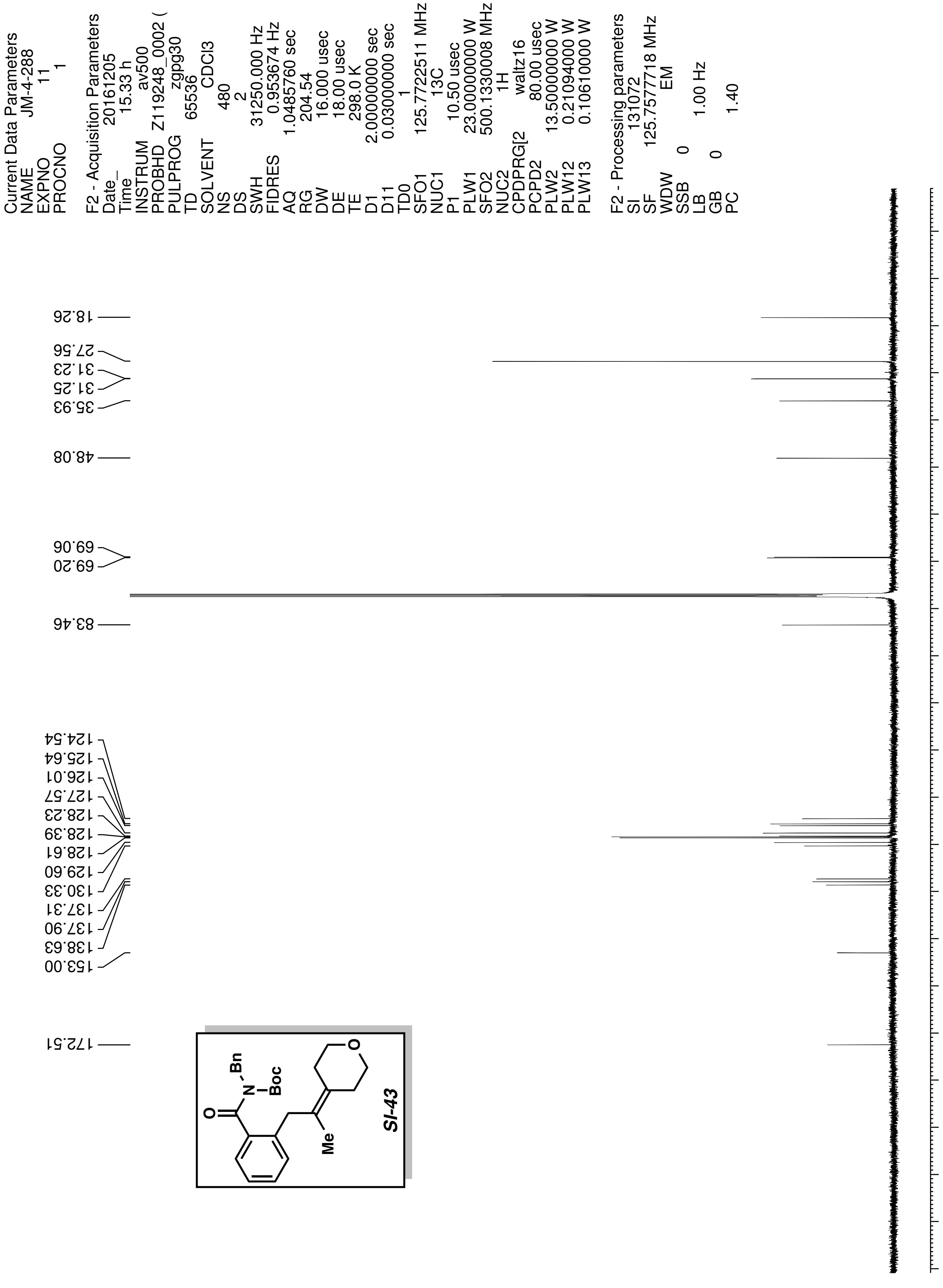
SI-40

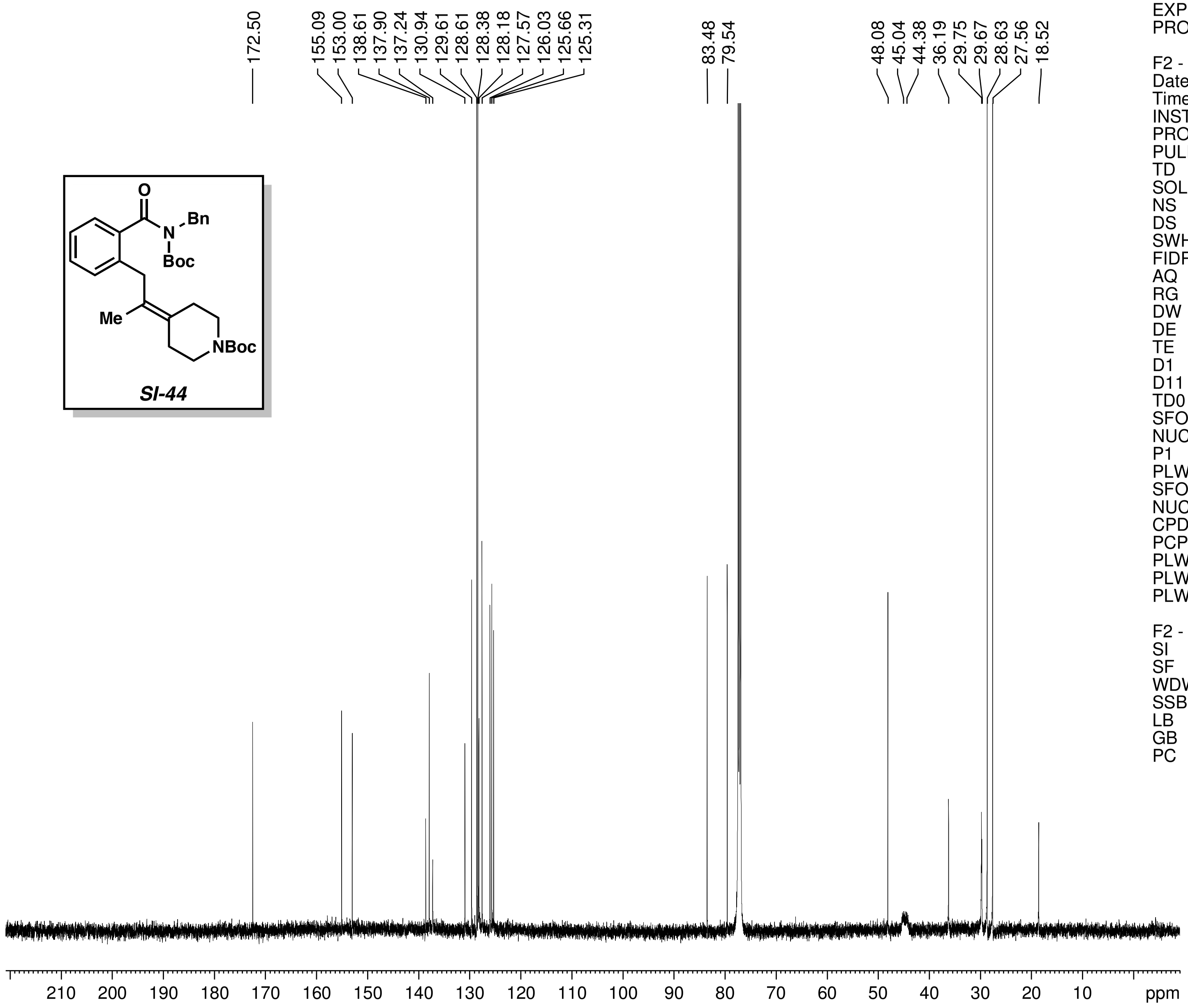


```
Current Data Parameters
NAME       JMM-7-135(c)
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_         20160907
Time             9.46
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD            65536
SOLVENT         CDCl3
NS               61
DS                2
SWH        43859.648 Hz
FIDRES      0.669245 Hz
AQ        0.7471104 sec
RG           204.54
DW           11.400 usec
DE           18.00 usec
TE           298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0               1

======== CHANNEL f1 =====
SFO1      125.7741375 MHz
NUC1             13C
P1              9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 =====
SFO2      500.1330008 MHz
NUC2              1H
CPDPRG[2        waltz16
PCPD2          80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI            131072
SF        125.7577721 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```



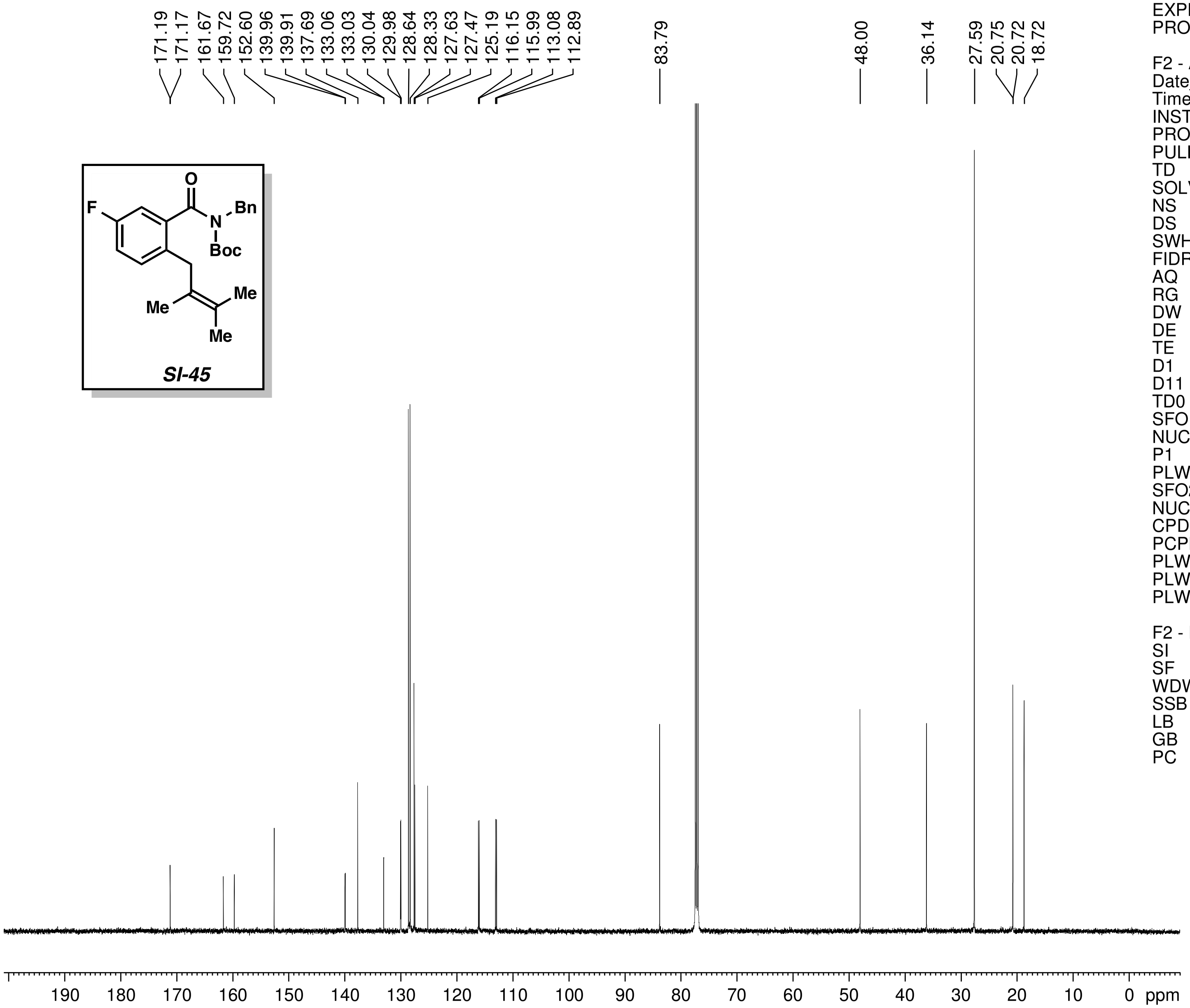
```
Current Data Parameters
NAME     JMM-7-223-char
EXPNO              2
PROCNO           999

F2 - Acquisition Parameters
Date_        20170117
Time            21.07
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD              65536
SOLVENT         CDCl3
NS                565
DS                  2
SWH        50000.000 Hz
FIDRES      0.762939 Hz
AQ        0.6553600 sec
RG             204.54
DW           10.000 usec
DE            18.00 usec
TE             298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1     125.7766527 MHz
NUC1                13C
P1             9.63 usec
PLW1     23.00000000 W

======== CHANNEL f2 =====
SFO2     500.1330008 MHz
NUC2                 1H
CPDPRG[2        waltz16
PCPD2         80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.13500001 W

F2 - Processing parameters
SI             131072
SF     125.7577714 MHz
WDW                EM
SSB     0
LB            1.00 Hz
GB      0
PC              1.40
```


Current Data Parameters
NAME JM-4-288
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161205
Time 15.33 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 480
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577718 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
18.26
27.56
31.23
31.25
35.93
48.08
69.06
69.20
83.46
124.54
125.64
126.01
127.57
128.23
128.39
128.61
129.60
130.33
137.31
137.90
138.63
153.00
172.51
O
N
Bn
Boc
Me
O
SI-43
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210

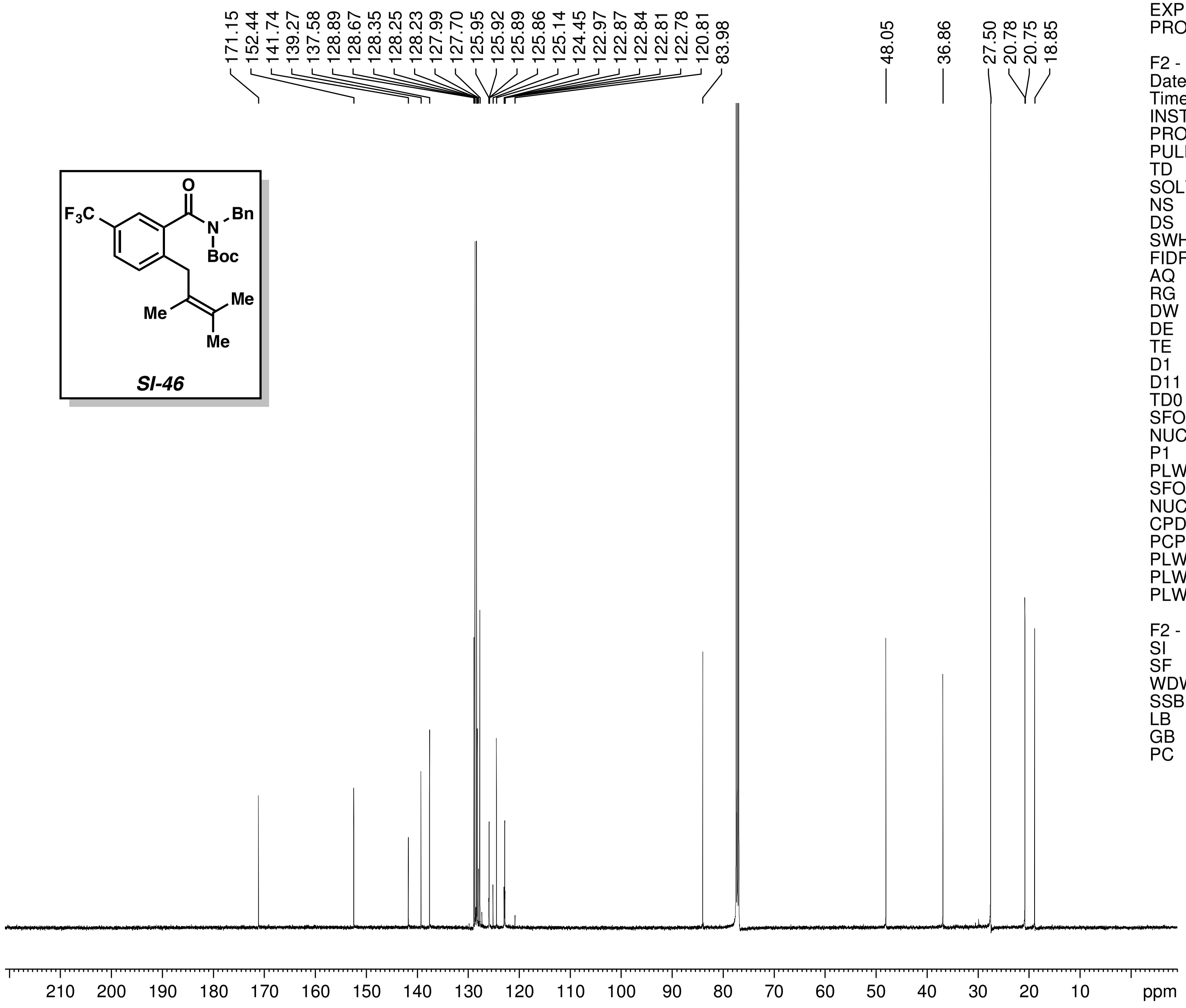


SI-44

```
Current Data Parameters
NAME          JM-4-290
EXPNO              101
PROCNO               1

F2 - Acquisition Parameters
Date_         20161218
Time           14.58 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                 480
DS                   2
SWH        31250.000 Hz
FIDRES      0.953674 Hz
AQ        1.0485760 sec
RG              204.54
DW          16.000 usec
DE           18.00 usec
TE               298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0                  1
SFO1    125.7722511 MHz
NUC1               13C
P1           10.50 usec
PLW1     23.00000000 W
SFO2    500.1330008 MHz
NUC2                1H
CPDPRG[2       waltz16
PCPD2        80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI              131072
SF      125.7577718 MHz
WDW                 EM
SSB      0
LB             1.00 Hz
GB       0
PC                1.40
```



```
Current Data Parameters
NAME          JM-5-067
EXPNO              101
PROCNO               1

F2 - Acquisition Parameters
Date_         20170217
Time            18.09 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG         zgpg30
TD              65536
SOLVENT          CDCl3
NS                  80
DS                   2
SWH          31250.000 Hz
FIDRES        0.953674 Hz
AQ           1.0485760 sec
RG              204.54
DW              16.000 usec
DE               18.00 usec
TE               298.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1
SFO1       125.7722511 MHz
NUC1               13C
P1               10.50 usec
PLW1       23.00000000 W
SFO2       500.1330008 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2       13.50000000 W
PLW12       0.21094000 W
PLW13       0.10610000 W

F2 - Processing parameters
SI              131072
SF         125.7577725 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



SI-46

Current Data Parameters

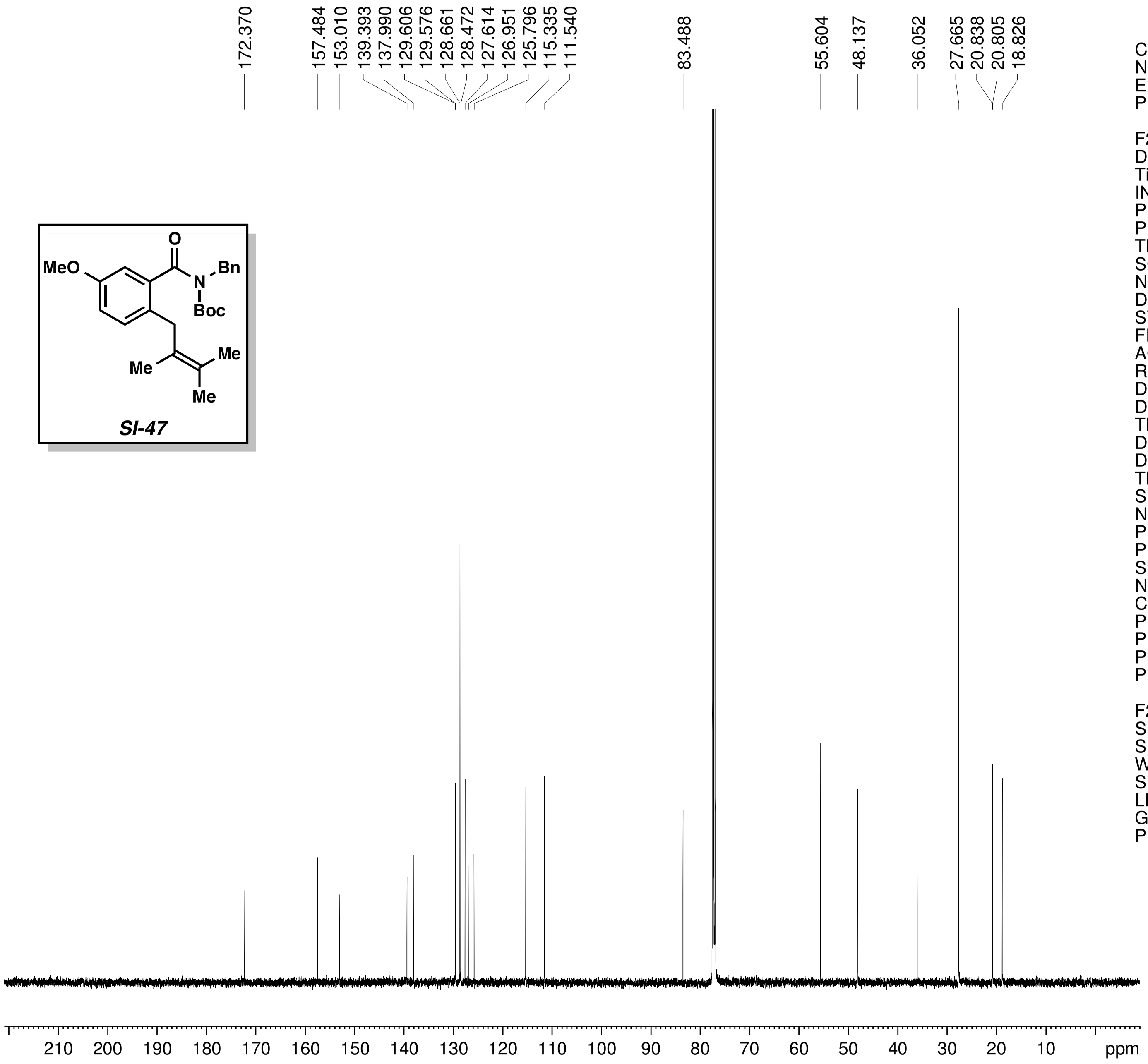
NAME	JM-5-055
EXPNO	201
PROCNO	1

F2 - Acquisition Parameters

Date_	20170206
Time	19.20 h
INSTRUM	av500
PROBHD	Z119248_0002 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	192
DS	2
SWH	31250.000 Hz
FIDRES	0.953674 Hz
AQ	1.0485760 sec
RG	204.54
DW	16.000 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	125.7722511 MHz
NUC1	13C
P1	10.50 usec
PLW1	23.00000000 W
SFO2	500.1330008 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	80.00 usec
PLW2	13.50000000 W
PLW12	0.21094000 W
PLW13	0.10610000 W

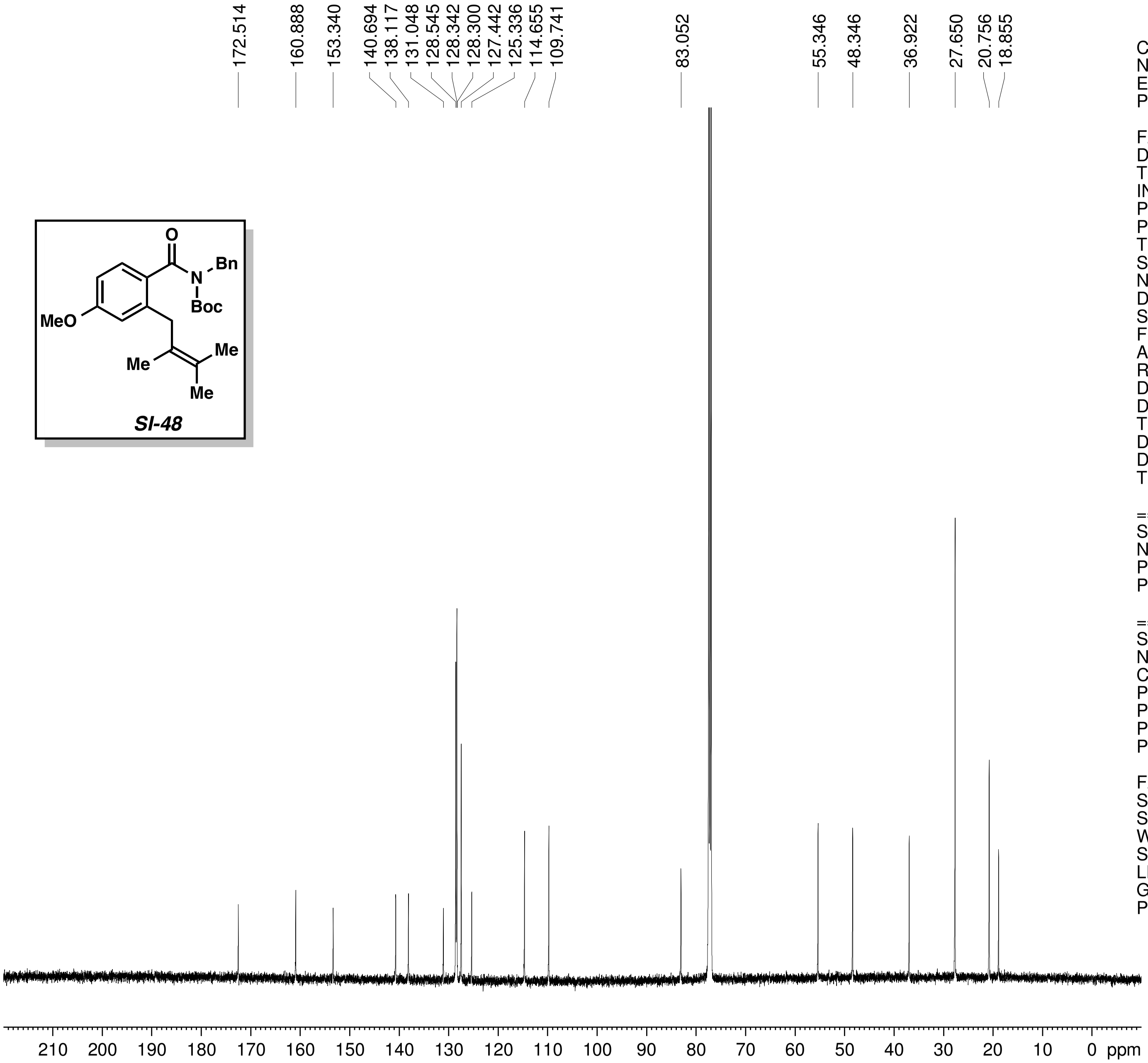
F2 - Processing parameters

SI	131072
SF	125.7577723 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



SI-47

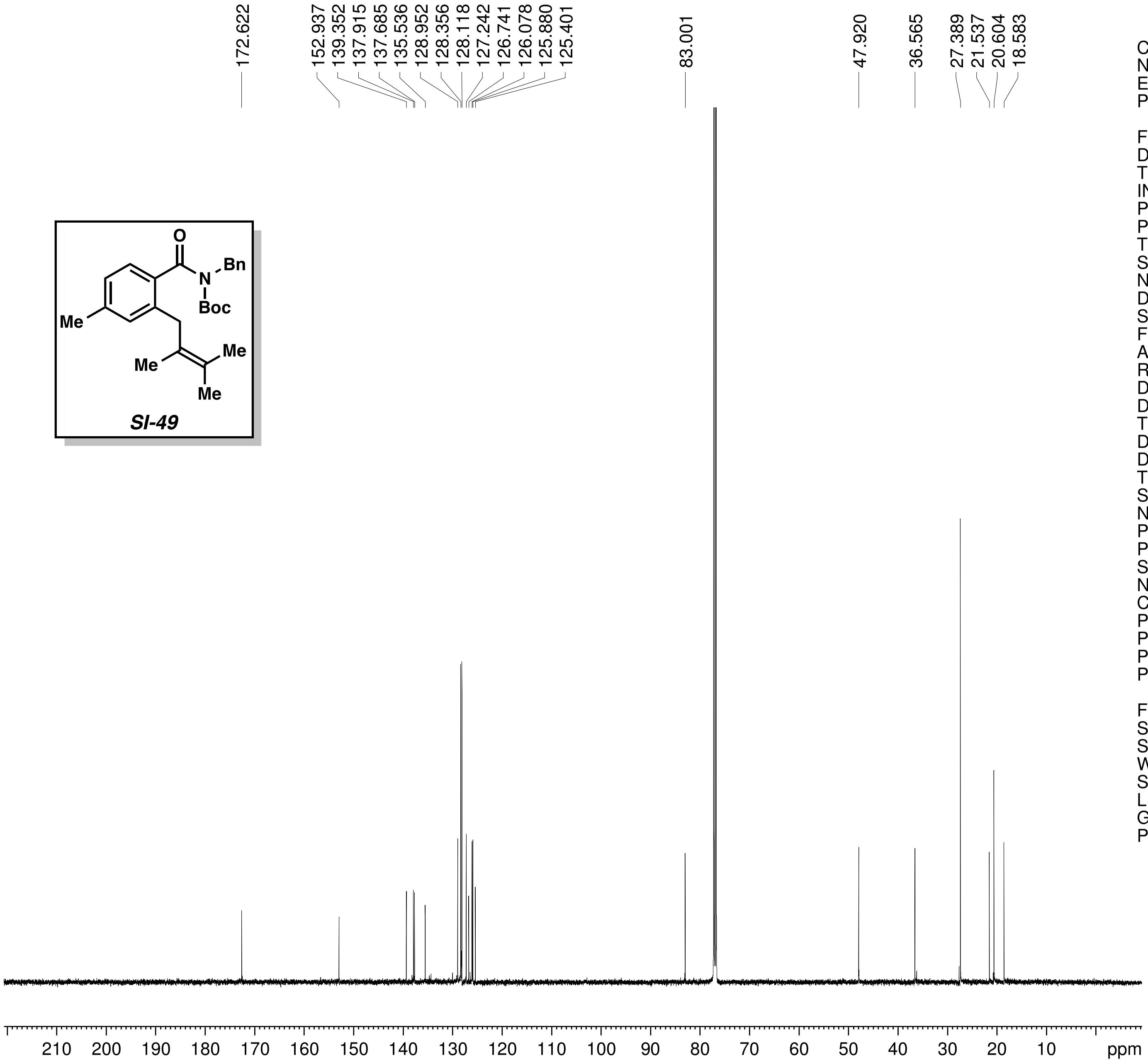
```
Current Data Parameters
NAME       JMM-7-71.500
EXPNO               2
PROCNO              1

F2 - Acquisition Parameters
Date_          20170131
Time             9.25 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG         zgpg30
TD              65536
SOLVENT          CDCl3
NS                 69
DS                  2
SWH         31250.000 Hz
FIDRES       0.953674 Hz
AQ         1.0485760 sec
RG             204.54
DW             16.000 usec
DE              18.00 usec
TE              298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                 1
SFO1      125.7722511 MHz
NUC1              13C
P1              10.50 usec
PLW1      23.00000000 W
SFO2      500.1330008 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2           80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.10610000 W

F2 - Processing parameters
SI             131072
SF        125.7577617 MHz
WDW                EM
SSB     0
LB               1.00 Hz
GB      0
PC               1.40
```



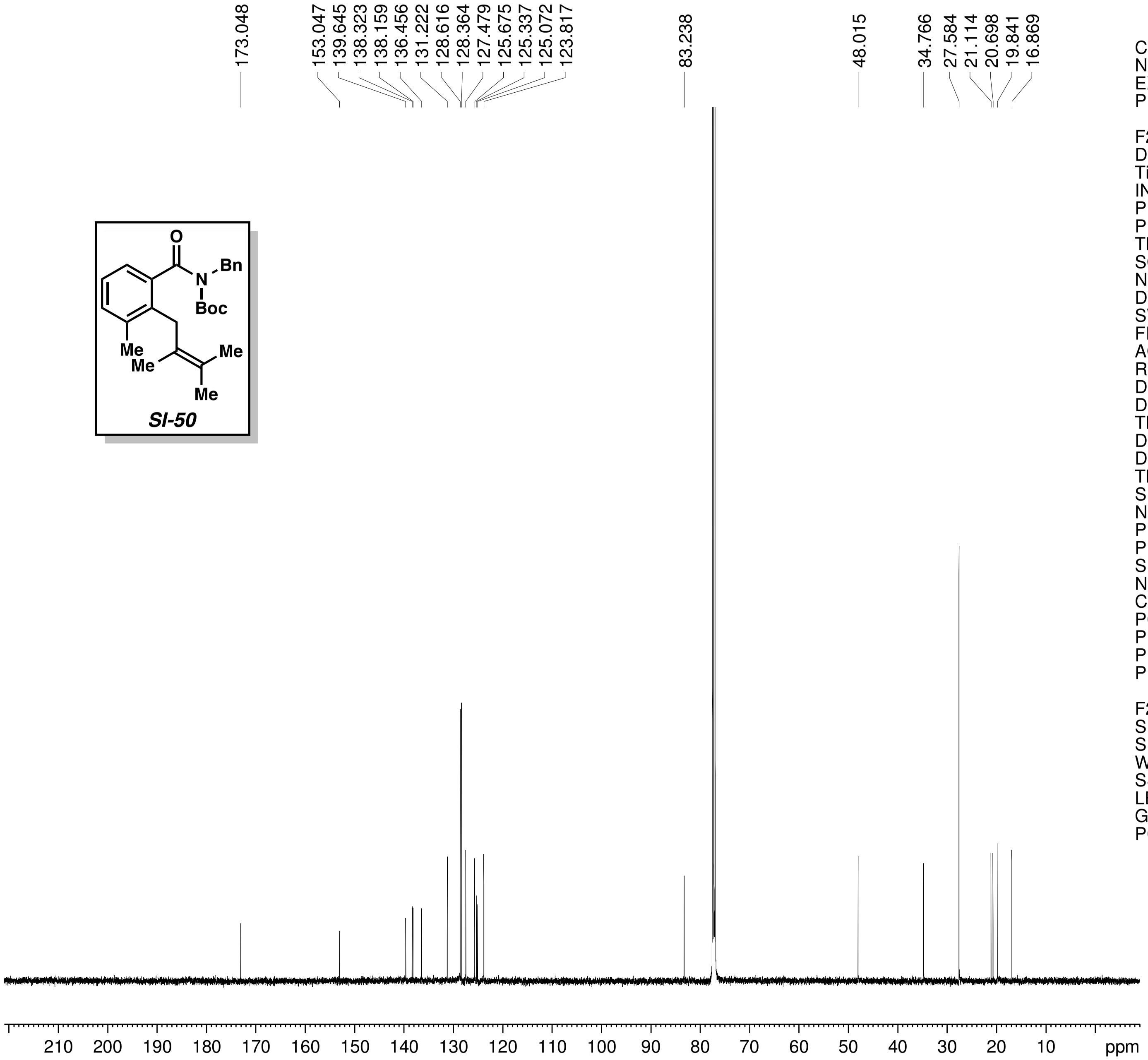
SI-48

```
Current Data Parameters
NAME     JMM-7-252-char
EXPNO              2
PROCNO           999

F2 - Acquisition Parameters
Date_        20170215
Time            14.19
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD              65536
SOLVENT         CDCl3
NS                 90
DS                  2
SWH        50000.000 Hz
FIDRES      0.762939 Hz
AQ         0.6553600 sec
RG             204.54
DW             10.000 usec
DE              18.00 usec
TE              298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1      125.7766527 MHz
NUC1              13C
P1               9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 =====
SFO2      500.1330008 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2           80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI            131072
SF        125.7577732 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```

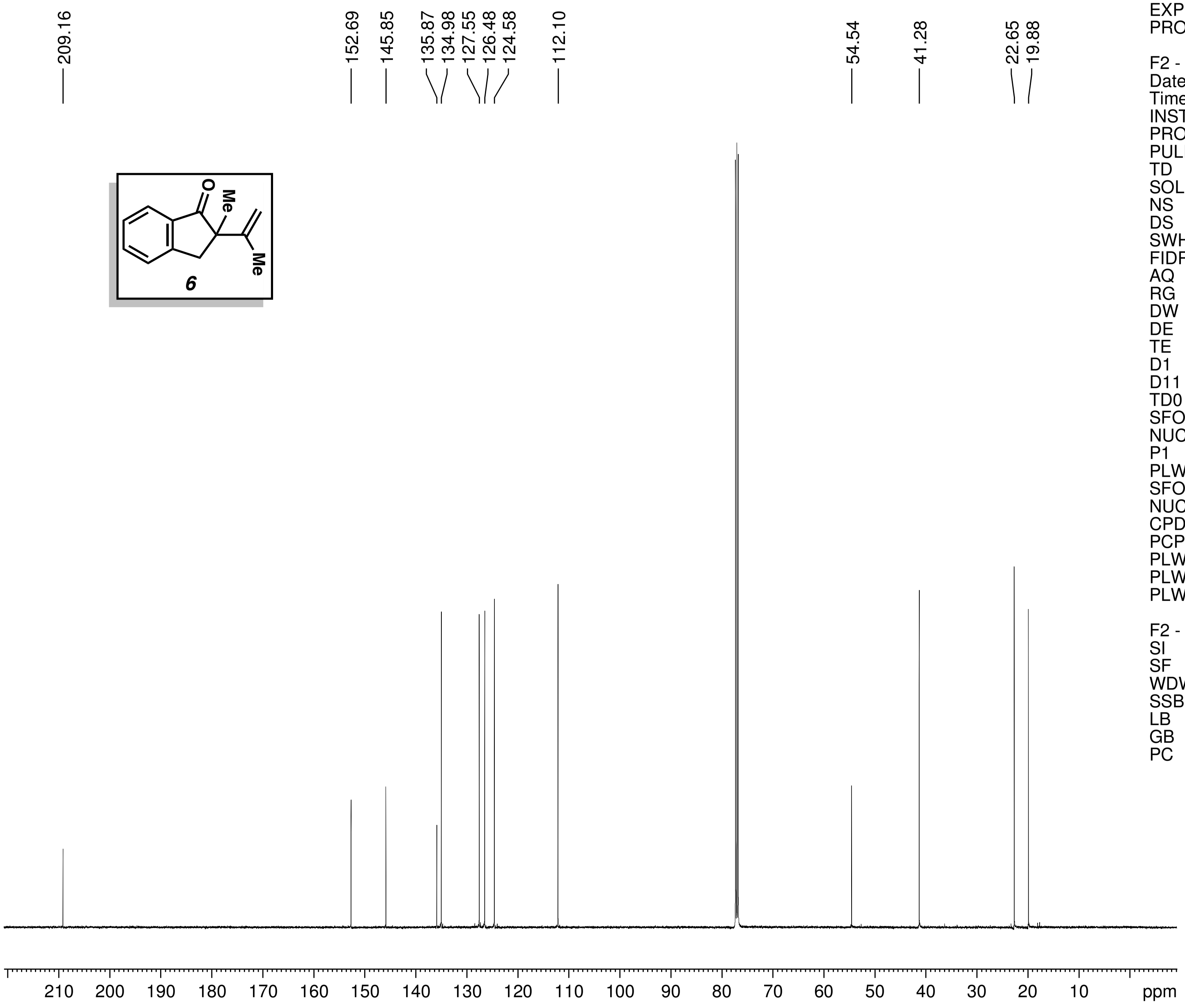
SI-49



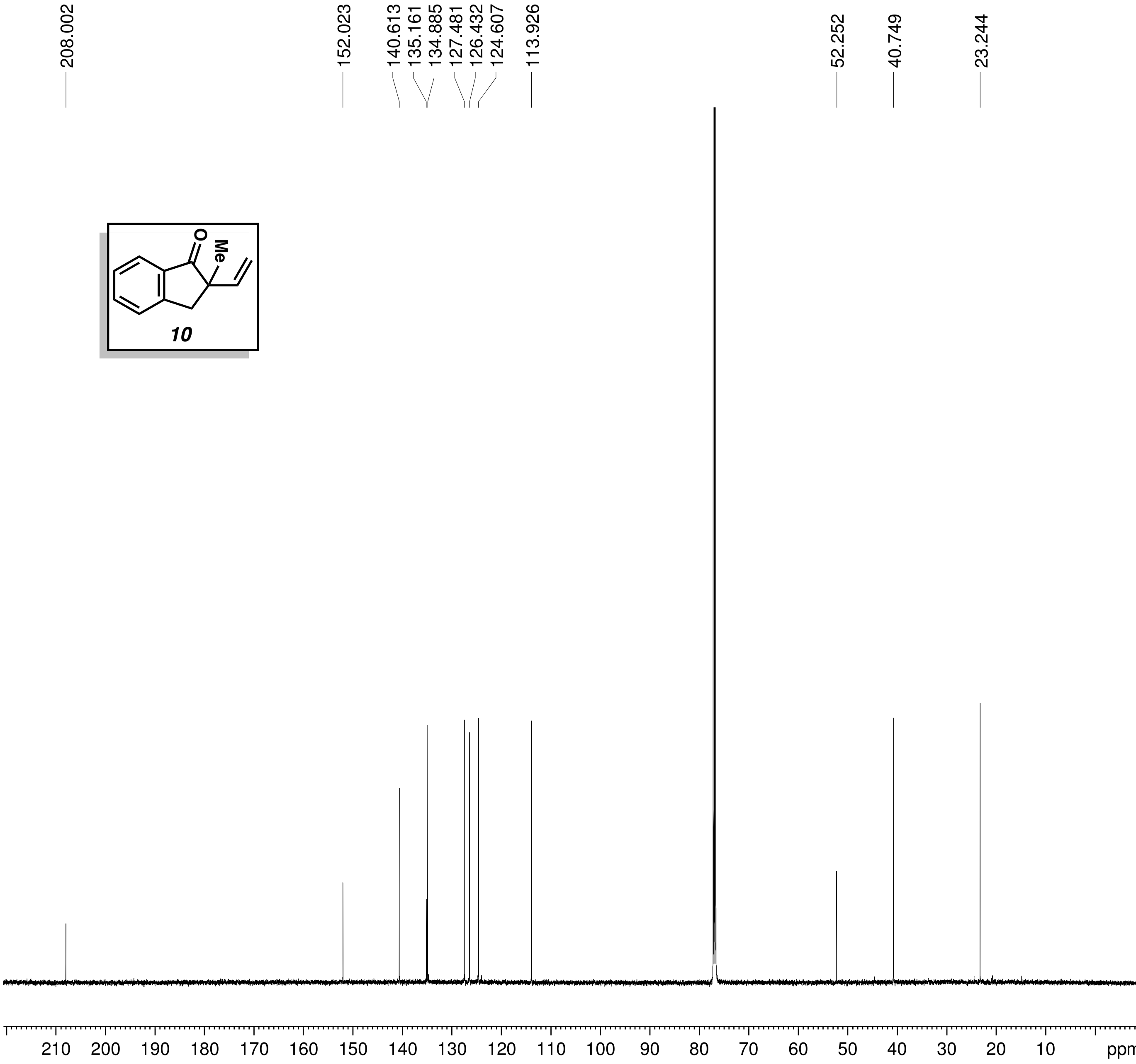
```
Current Data Parameters
NAME       sr-1-129.500
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20170217
Time             11.07 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG          zgpg30
TD               65536
SOLVENT           CDCl3
NS                   44
DS                    2
SWH          31250.000 Hz
FIDRES        0.953674 Hz
AQ           1.0485760 sec
RG              204.54
DW              16.000 usec
DE               18.00 usec
TE               298.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                   1
SFO1       125.7722511 MHz
NUC1                13C
P1               10.50 usec
PLW1       23.00000000 W
SFO2       500.1330008 MHz
NUC2                 1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2       13.50000000 W
PLW12       0.21094000 W
PLW13       0.10610000 W

F2 - Processing parameters
SI              131072
SF         125.7577961 MHz
WDW                  EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



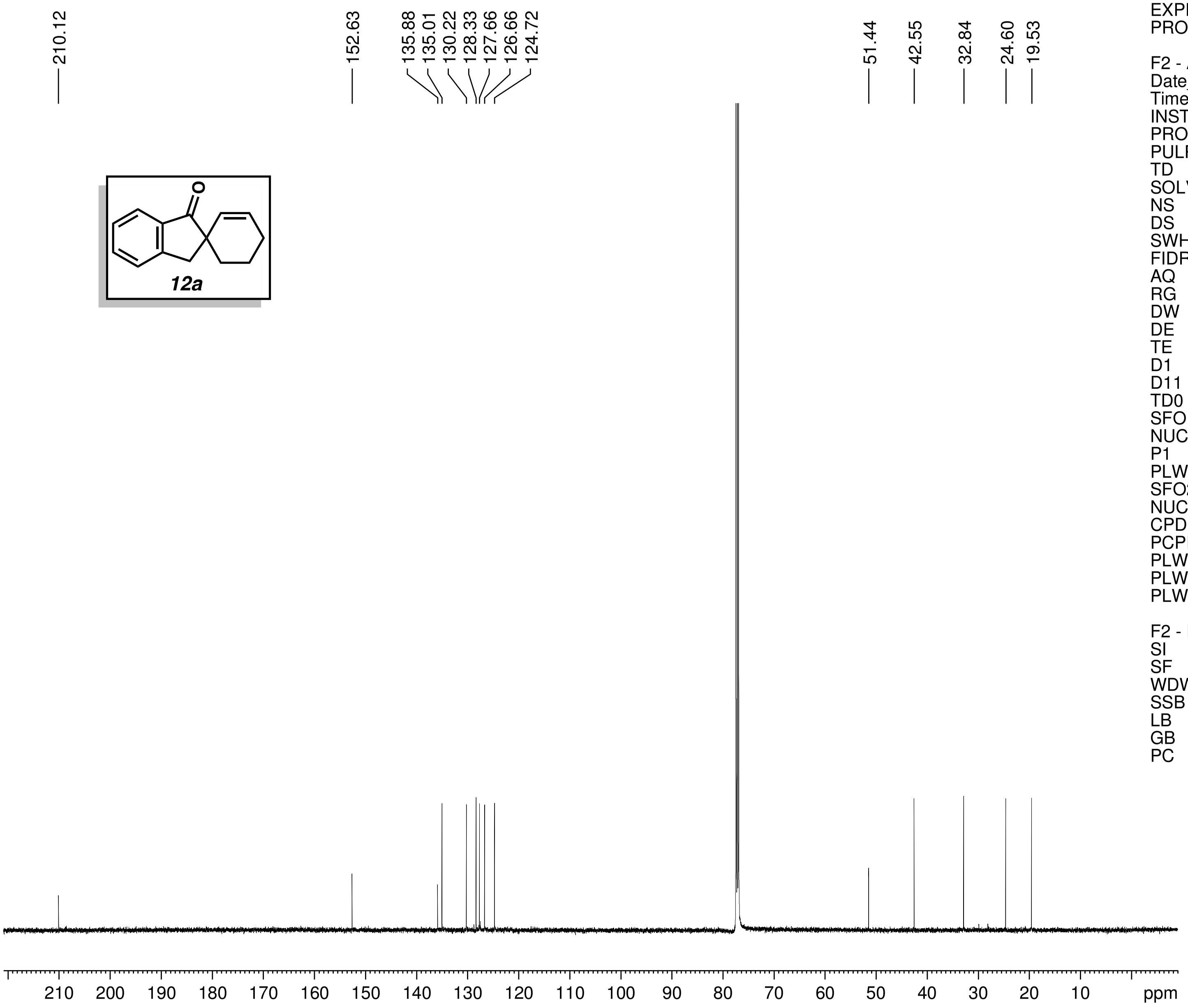
```
Current Data Parameters
NAME       sr-1-101.500
EXPNO              4
PROCNO             1

F2 - Acquisition Parameters
Date_        20170131
Time           9.17 h
INSTRUM        av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD            65536
SOLVENT        CDCl3
NS              160
DS                2
SWH       31250.000 Hz
FIDRES     0.953674 Hz
AQ        1.0485760 sec
RG           204.54
DW           16.000 usec
DE            18.00 usec
TE            298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0               1
SFO1    125.7722511 MHz
NUC1            13C
P1            10.50 usec
PLW1    23.00000000 W
SFO2    500.1330008 MHz
NUC2             1H
CPDPRG[2      waltz16
PCPD2         80.00 usec
PLW2    13.50000000 W
PLW12    0.21094000 W
PLW13    0.10610000 W

F2 - Processing parameters
SI           131072
SF      125.7577617 MHz
WDW              EM
SSB     0
LB             1.00 Hz
GB      0
PC             1.40
```



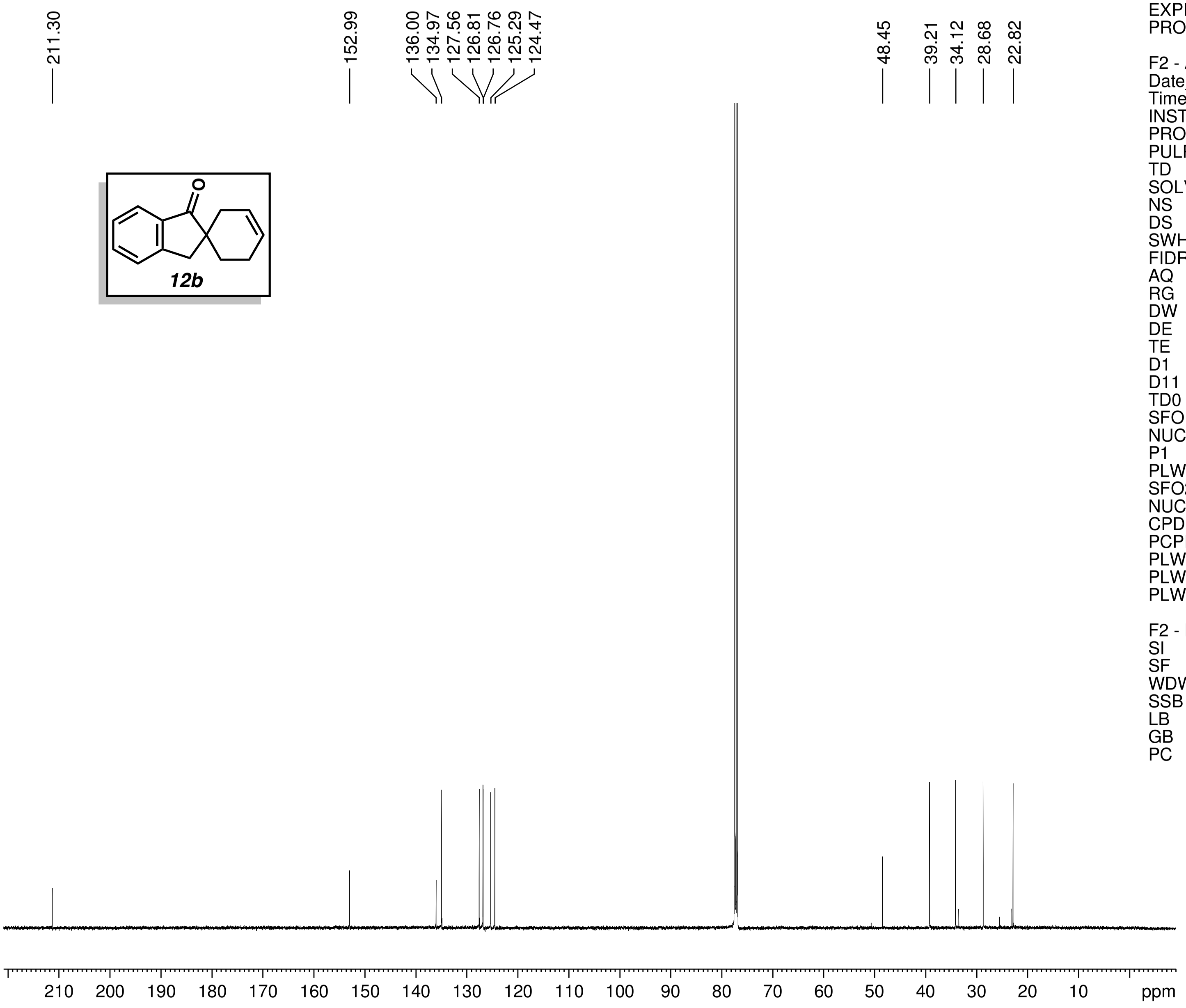
```
Current Data Parameters
NAME          JM-5-025
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_         20170115
Time             13.55 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD            65536
SOLVENT         CDCl3
NS                96
DS                 2
SWH        31250.000 Hz
FIDRES      0.953674 Hz
AQ         1.0485760 sec
RG            204.54
DW            16.000 usec
DE             18.00 usec
TE             298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1
SFO1     125.7722511 MHz
NUC1             13C
P1             10.50 usec
PLW1     23.00000000 W
SFO2     500.1330008 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI            131072
SF       125.7577892 MHz
WDW               EM
SSB      0
LB              1.00 Hz
GB       0
PC              1.40
```



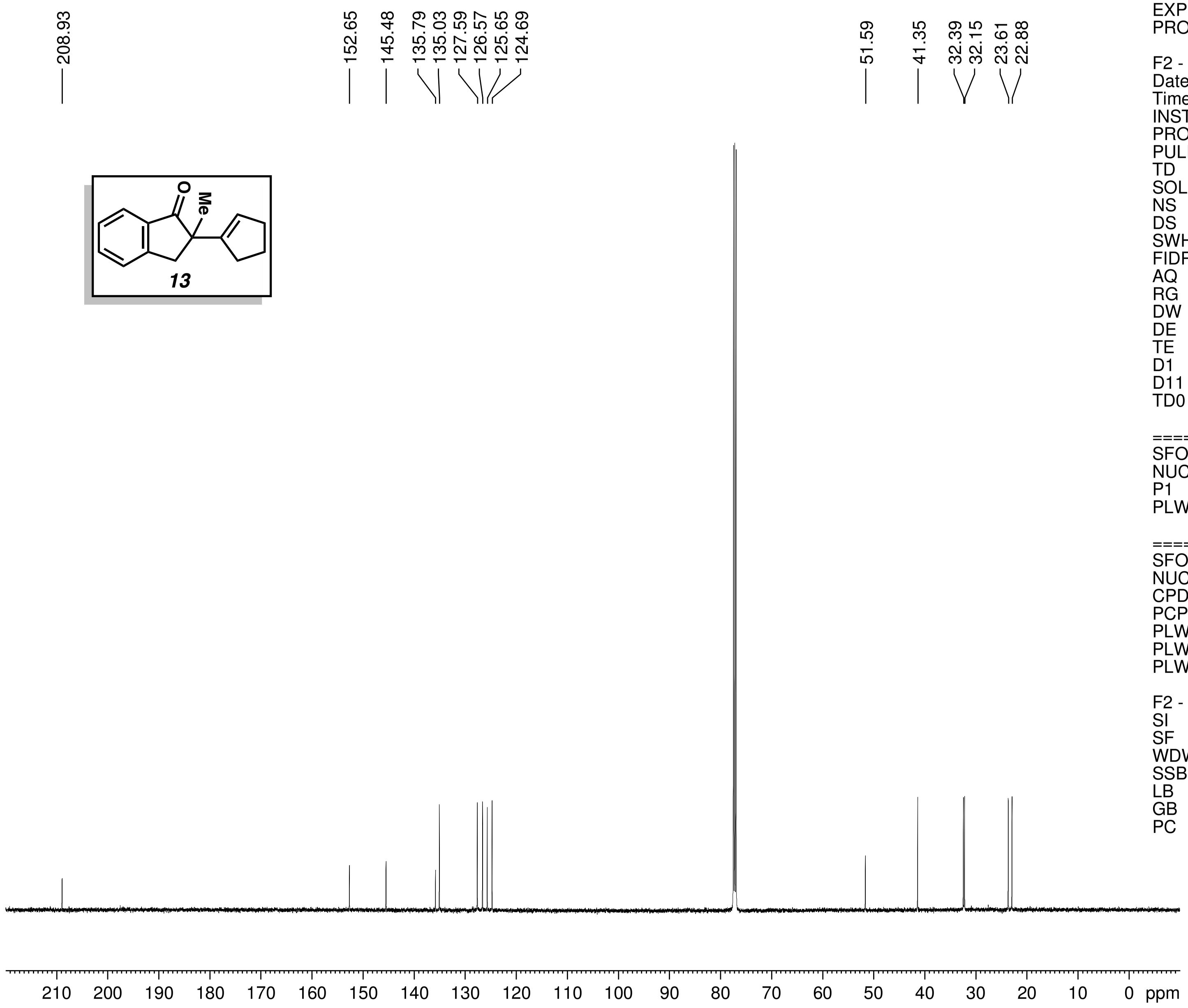
```
Current Data Parameters
NAME    sr-1-108.500.6
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20170207
Time           10.18 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD            65536
SOLVENT          CDCl3
NS                62
DS                 2
SWH        31250.000 Hz
FIDRES      0.953674 Hz
AQ        1.0485760 sec
RG            204.54
DW            16.000 usec
DE             18.00 usec
TE             298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1
SFO1     125.7722511 MHz
NUC1             13C
P1             10.50 usec
PLW1     23.00000000 W
SFO2     500.1330008 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI           131072
SF       125.7578023 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```



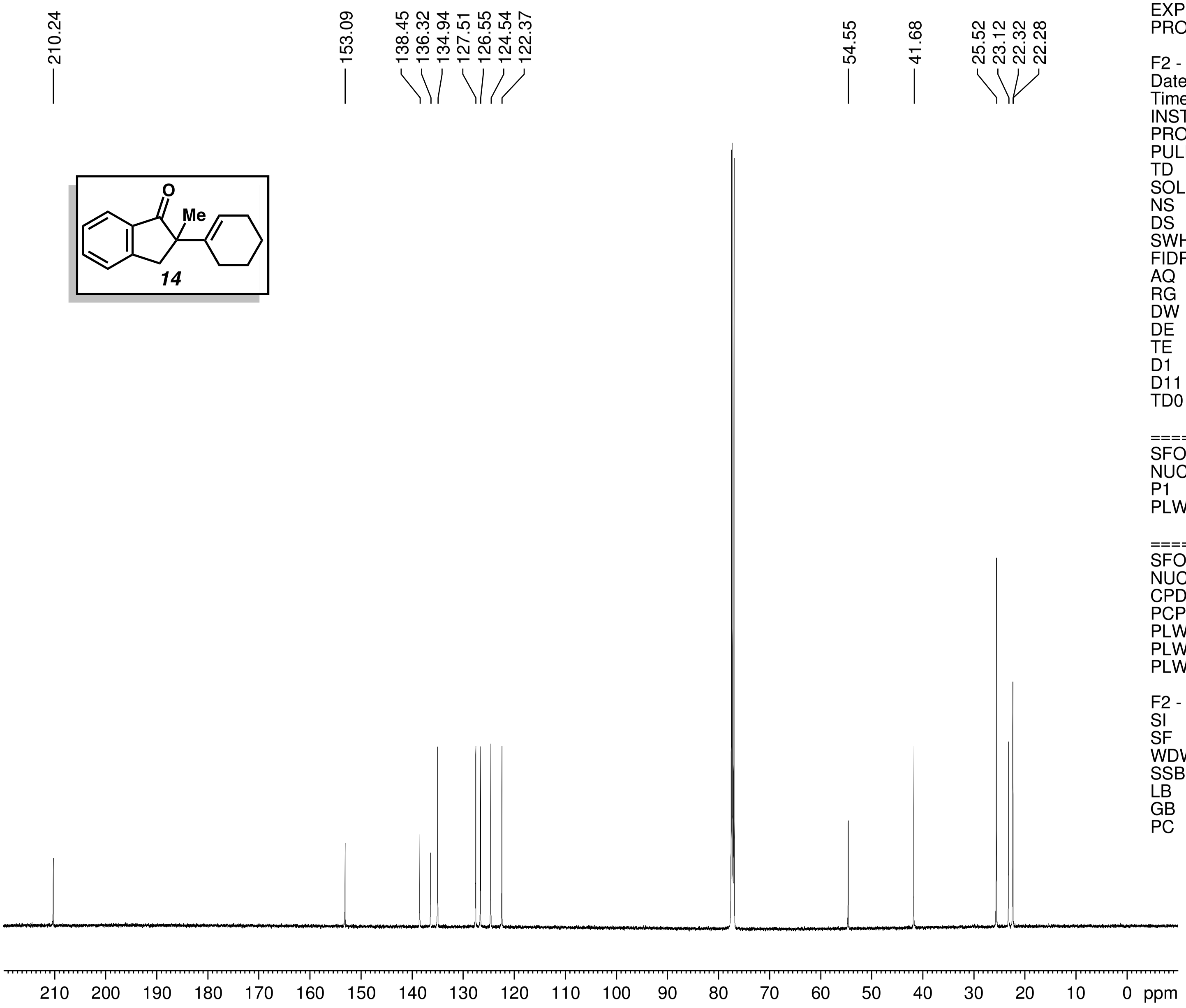
```
Current Data Parameters
NAME          JM-5-019
EXPNO              401
PROCNO               1

F2 - Acquisition Parameters
Date_         20170110
Time            9.50 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD             65536
SOLVENT          CDCl3
NS                 399
DS                   2
SWH       31250.000 Hz
FIDRES     0.953674 Hz
AQ       1.0485760 sec
RG              204.54
DW          16.000 usec
DE           18.00 usec
TE             298.0 K
D1      2.00000000 sec
D11     0.03000000 sec
TD0                  1
SFO1    125.7722511 MHz
NUC1               13C
P1           10.50 usec
PLW1     23.00000000 W
SFO2    500.1330008 MHz
NUC2                1H
CPDPRG[2       waltz16
PCPD2        80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI              131072
SF      125.7577718 MHz
WDW                 EM
SSB     0
LB             1.00 Hz
GB      0
PC                1.40
```



```
Current Data Parameters
NAME          JM-5-019
EXPNO              301
PROCNO               1

F2 - Acquisition Parameters
Date_         20170110
Time           10.03 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                162
DS                  2
SWH         31250.000 Hz
FIDRES       0.953674 Hz
AQ          1.0485760 sec
RG             204.54
DW             16.000 usec
DE              18.00 usec
TE              298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                 1
SFO1      125.7722511 MHz
NUC1              13C
P1              10.50 usec
PLW1      23.00000000 W
SFO2      500.1330008 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2           80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.10610000 W

F2 - Processing parameters
SI             131072
SF        125.7577722 MHz
WDW                EM
SSB      0
LB              1.00 Hz
GB       0
PC               1.40
```

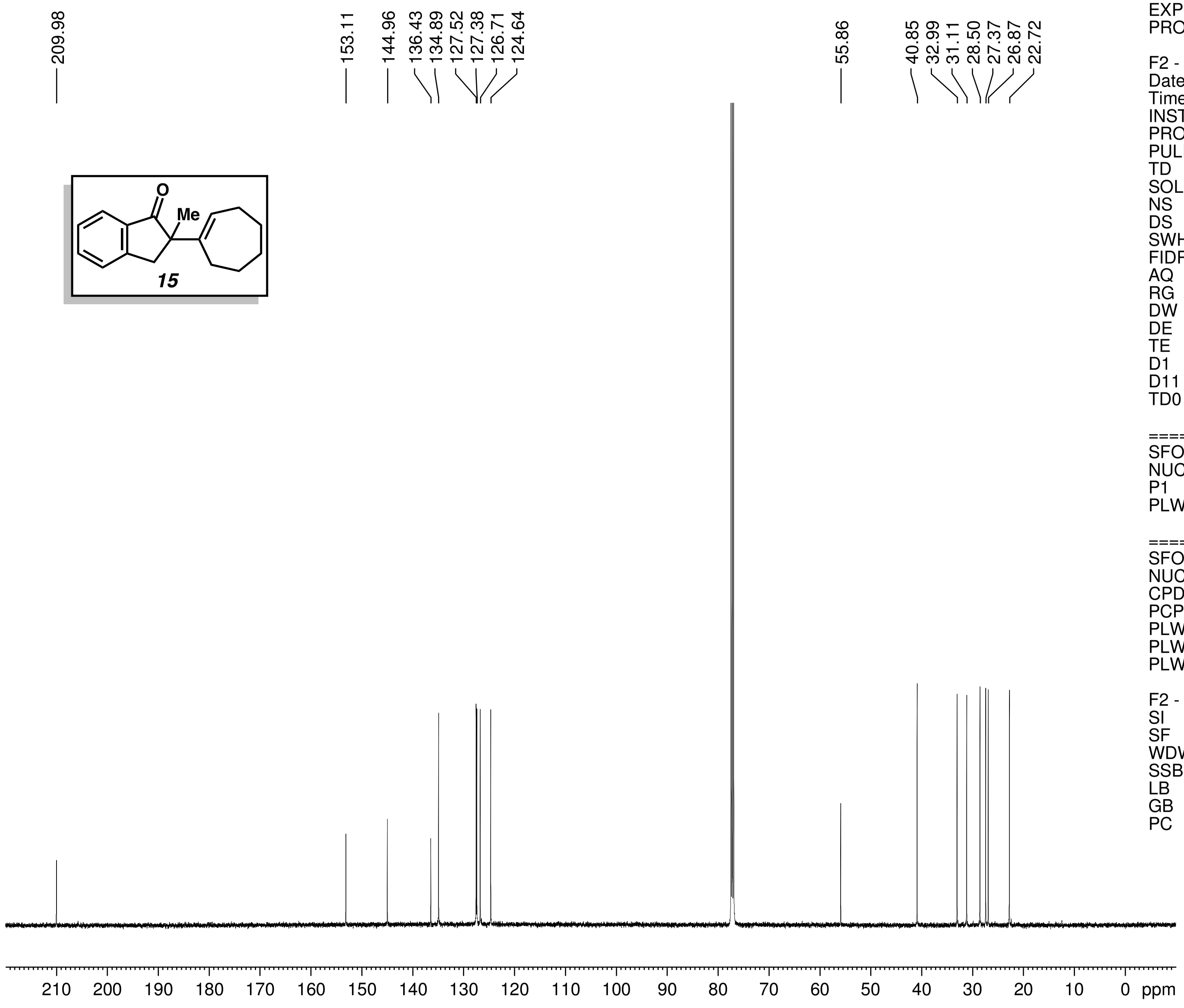


```
Current Data Parameters
NAME      JMM-7-212(A1)
EXPNO             2
PROCNO          999

F2 - Acquisition Parameters
Date_        20161216
Time            9.10
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD            65536
SOLVENT         CDCl3
NS               40
DS                2
SWH       50000.000 Hz
FIDRES     0.762939 Hz
AQ        0.6553600 sec
RG           204.54
DW           10.000 usec
DE            18.00 usec
TE            298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0               1

======== CHANNEL f1 =====
SFO1     125.7728799 MHz
NUC1            13C
P1             9.63 usec
PLW1     23.00000000 W

======== CHANNEL f2 =====
SFO2     500.1330008 MHz
NUC2             1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.13500001 W

F2 - Processing parameters
SI           131072
SF       125.7577731 MHz
WDW              EM
SSB    0
LB             1.00 Hz
GB     0
PC             1.40
```

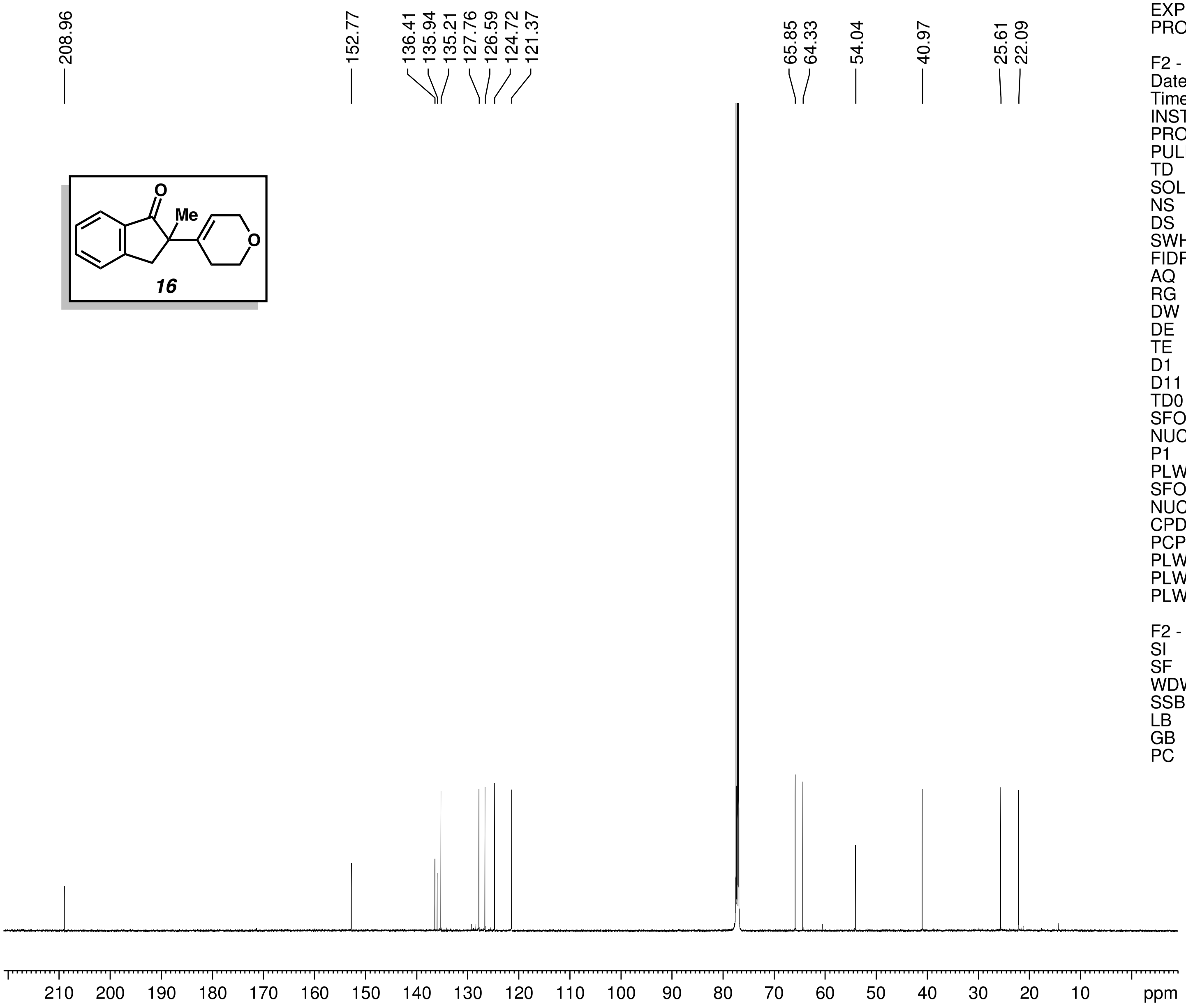


14

```
Current Data Parameters
NAME     JMM-7-198(Cchar)
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20161204
Time          14.22
INSTRUM          av500
PROBHD   5 mm DCH 13C-1
PULPROG         zgpg30
TD            65536
SOLVENT          CDCl3
NS               136
DS                 2
SWH          50000.000 Hz
FIDRES        0.762939 Hz
AQ          0.6553600 sec
RG             204.54
DW             10.000 usec
DE            100.00 usec
TE             298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                1

======== CHANNEL f1 =====
SFO1       125.7634481 MHz
NUC1              13C
P1               9.63 usec
PLW1       23.00000000 W

======== CHANNEL f2 =====
SFO2       500.1330008 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2           80.00 usec
PLW2       13.50000000 W
PLW12       0.21094000 W
PLW13       0.13500001 W

F2 - Processing parameters
SI             131072
SF         125.7577744 MHz
WDW                EM
SSB      0
LB               1.00 Hz
GB       0
PC               1.40
```



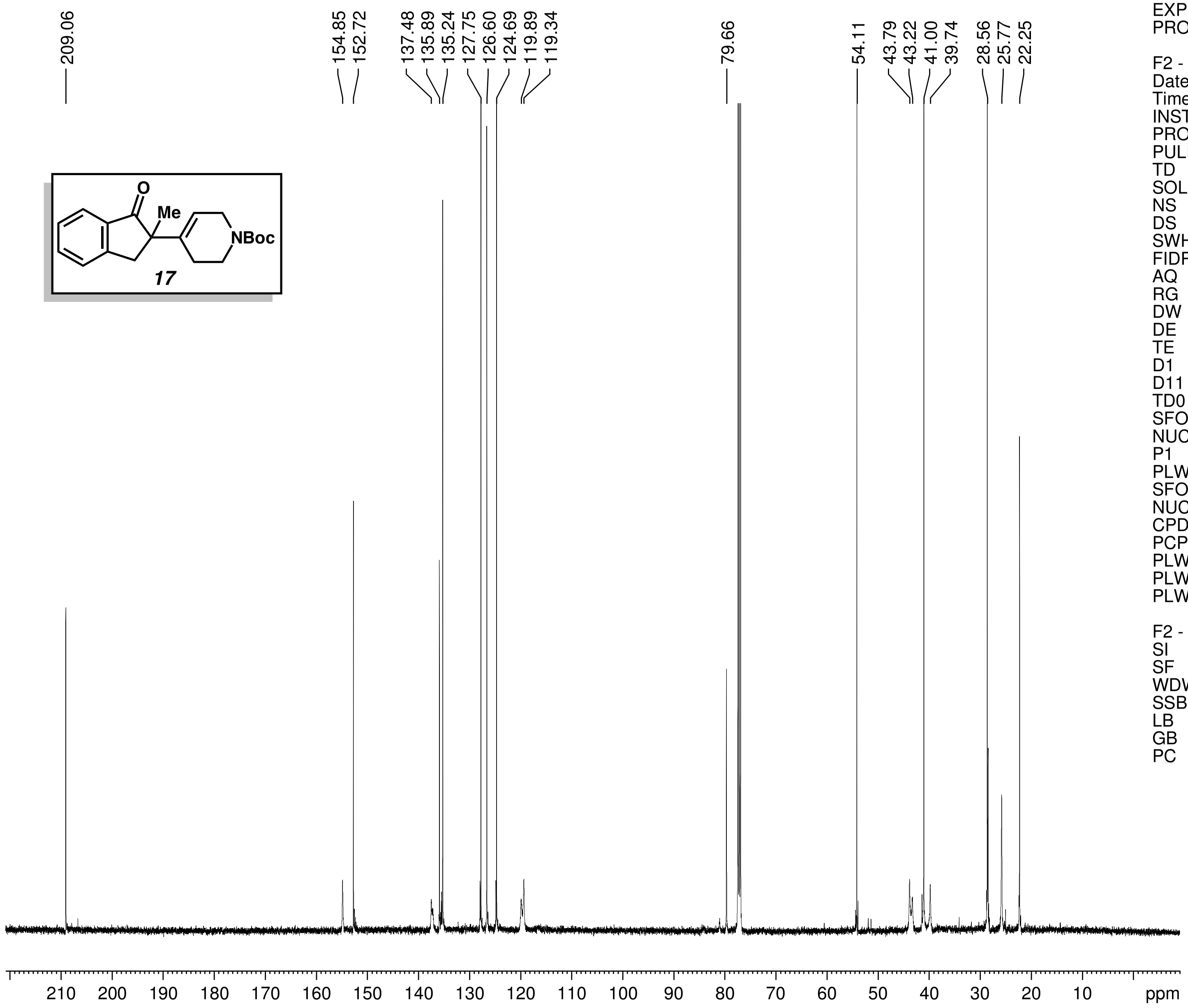
```
Current Data Parameters
NAME     JMM-7-225-char
EXPNO              2
PROCNO           999

F2 - Acquisition Parameters
Date_         20170126
Time             13.18
INSTRUM          av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                 89
DS                  2
SWH        50000.000 Hz
FIDRES      0.762939 Hz
AQ        0.6553600 sec
RG             204.54
DW          10.000 usec
DE           18.00 usec
TE             298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1     125.7766527 MHz
NUC1              13C
P1             9.63 usec
PLW1     23.00000000 W

======== CHANNEL f2 =====
SFO2     500.1330008 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2         80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.13500001 W

F2 - Processing parameters
SI            131072
SF       125.7577735 MHz
WDW               EM
SSB     0
LB             1.00 Hz
GB      0
PC              1.40
```



Current Data Parameters
NAME JM-5-017
EXPNO 101
PROCNO 1

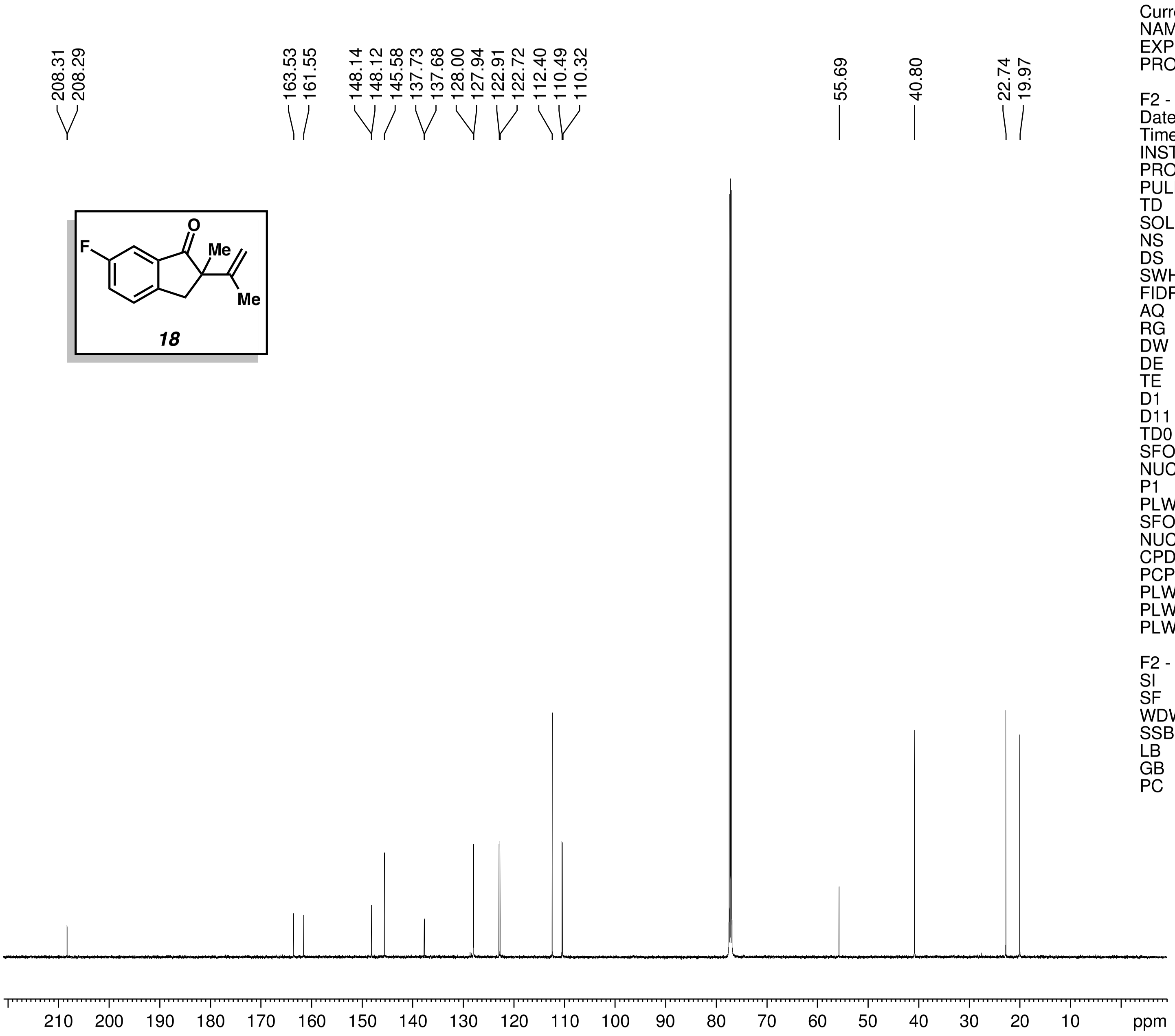
F2 - Acquisition Parameters
Date_ 20161216
Time 11.32 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 960
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W

F2 - Processing parameters
SI 131072
SF 125.7577719 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



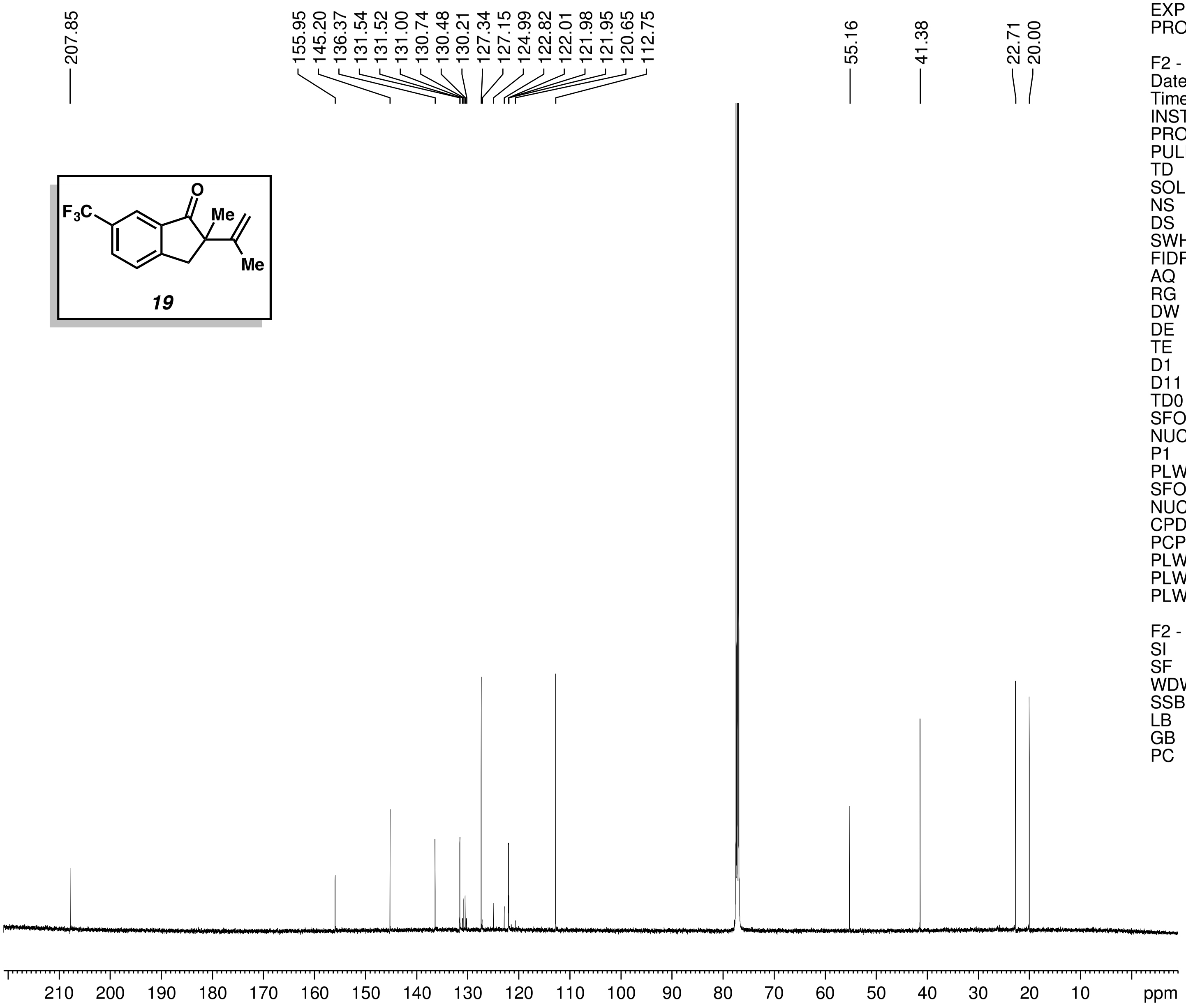
17

```
Current Data Parameters
NAME        JM-5-021-2D
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_         20170106
Time           14.26 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD             65536
SOLVENT          CDCl3
NS               129
DS                 2
SWH        31250.000 Hz
FIDRES      0.953674 Hz
AQ         1.0485760 sec
RG            204.54
DW            16.000 usec
DE            18.00 usec
TE            298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1
SFO1     125.7722511 MHz
NUC1             13C
P1            10.50 usec
PLW1     23.00000000 W
SFO2     500.1330008 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI            131072
SF       125.7577758 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```



18

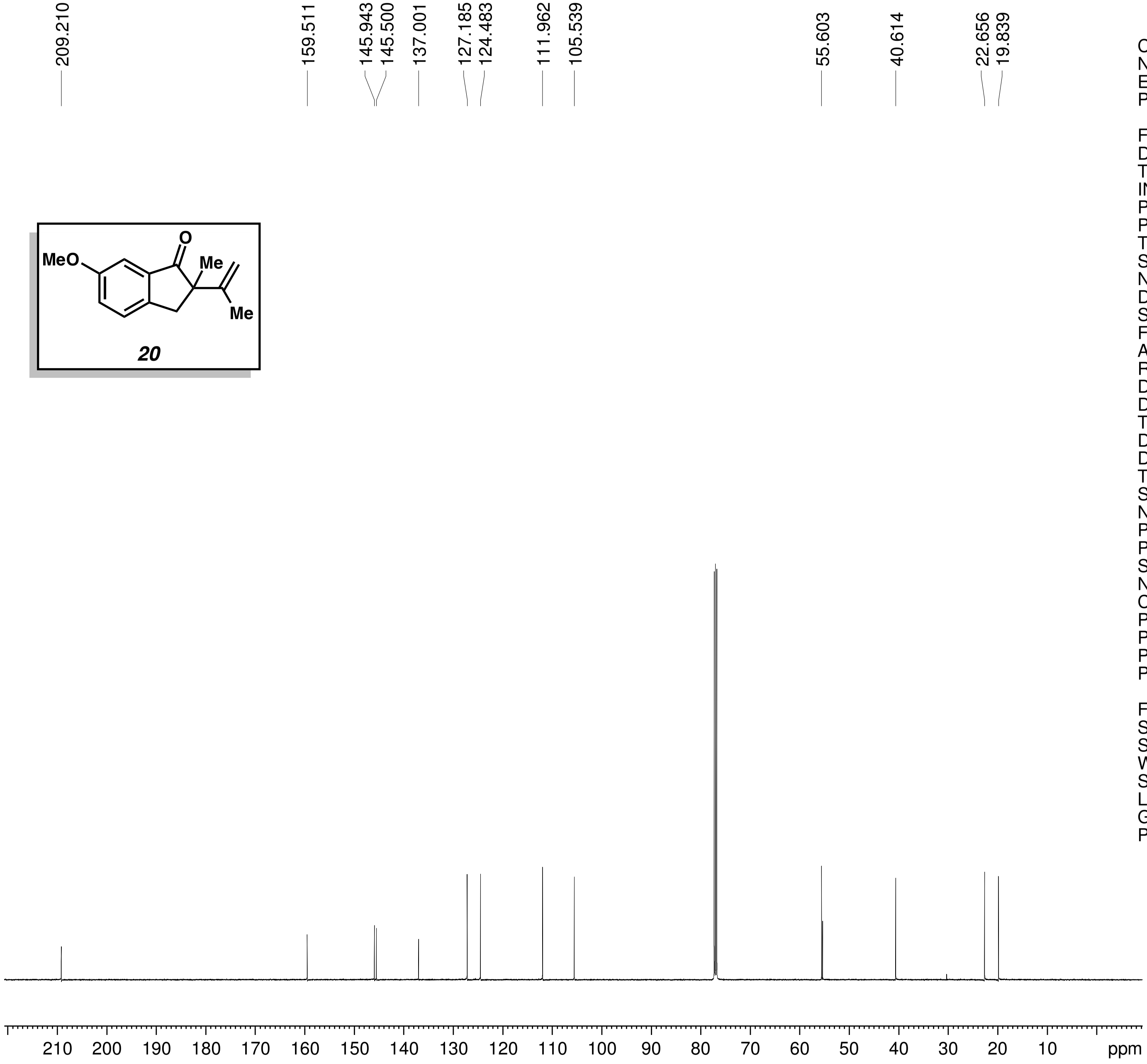
```
Current Data Parameters
NAME          JM-5-067
EXPNO              201
PROCNO               1

F2 - Acquisition Parameters
Date_         20170217
Time           18.20 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD             65536
SOLVENT          CDCl3
NS                 48
DS                  2
SWH        31250.000 Hz
FIDRES      0.953674 Hz
AQ         1.0485760 sec
RG            204.54
DW            16.000 usec
DE             18.00 usec
TE             298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1
SFO1     125.7722511 MHz
NUC1             13C
P1             10.50 usec
PLW1     23.00000000 W
SFO2     500.1330008 MHz
NUC2              1H
CPDPRG[2      waltz16
PCPD2          80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI            131072
SF       125.7577729 MHz
WDW               EM
SSB      0
LB              1.00 Hz
GB       0
PC              1.40
```



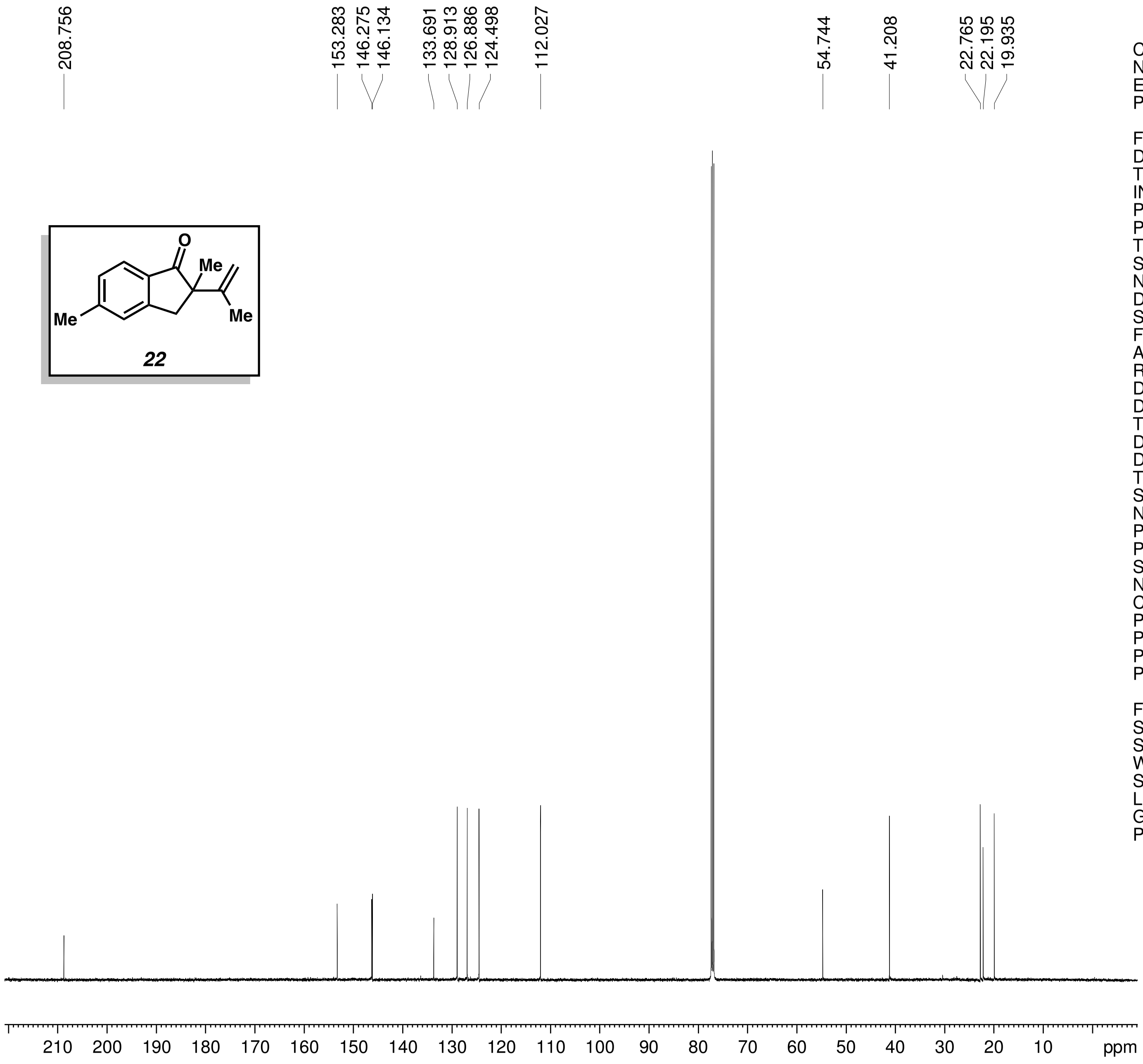
19

```
Current Data Parameters
NAME          JM-5-057
EXPNO              101
PROCNO               1

F2 - Acquisition Parameters
Date_         20170206
Time           20.04 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                730
DS                  2
SWH        31250.000 Hz
FIDRES      0.953674 Hz
AQ         1.0485760 sec
RG             204.54
DW             16.000 usec
DE              18.00 usec
TE              298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                 1
SFO1     125.7722511 MHz
NUC1              13C
P1              10.50 usec
PLW1      23.00000000 W
SFO2     500.1330008 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2           80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.10610000 W

F2 - Processing parameters
SI             131072
SF       125.7577716 MHz
WDW                EM
SSB      0
LB               1.00 Hz
GB       0
PC               1.40
```

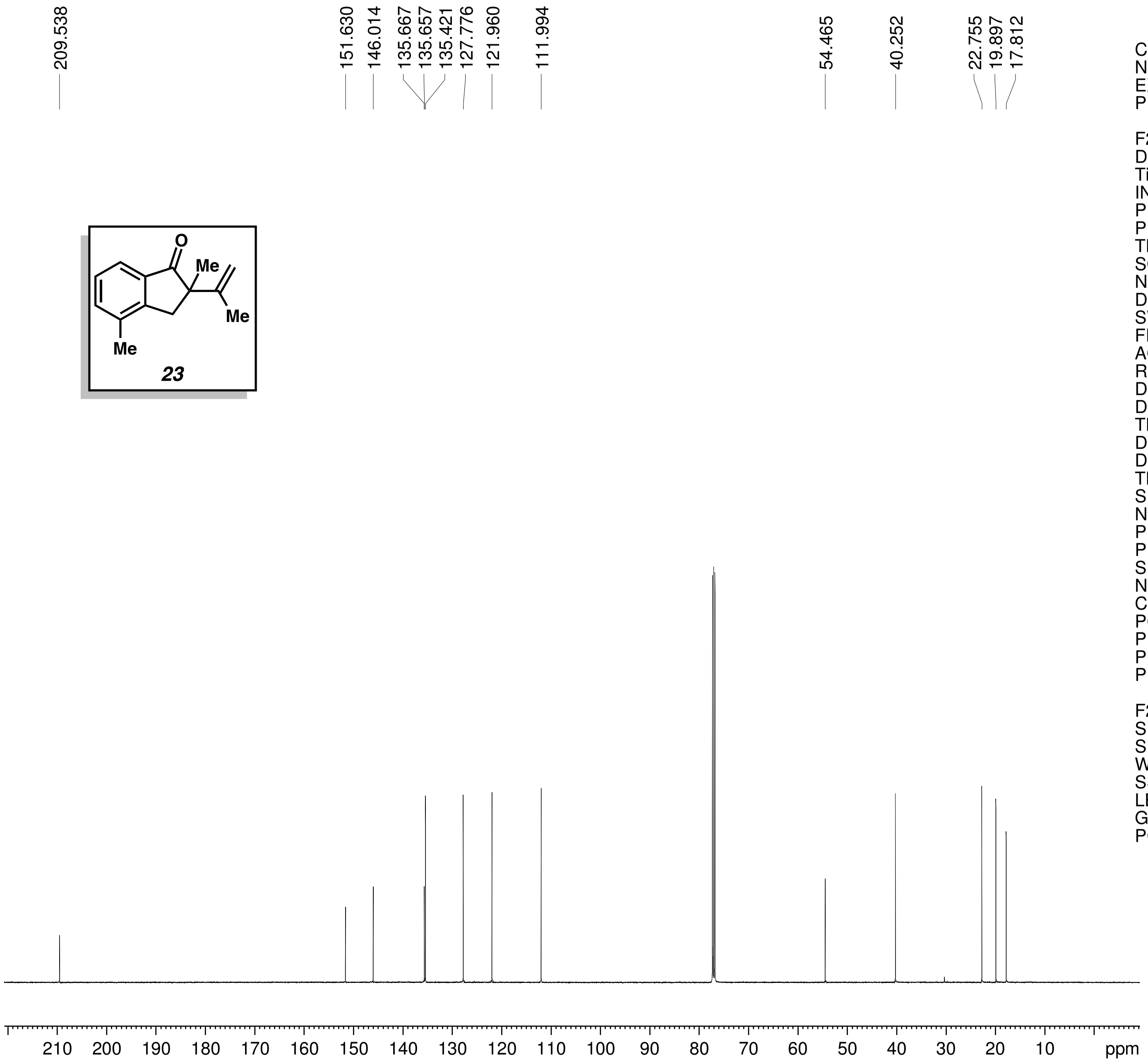
20



```
Current Data Parameters
NAME       sr-1-105.500
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_         20170203
Time            9.22 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG         zgpg30
TD              65536
SOLVENT          CDCl3
NS                 66
DS                  2
SWH         31250.000 Hz
FIDRES       0.953674 Hz
AQ          1.0485760 sec
RG             204.54
DW             16.000 usec
DE              18.00 usec
TE              298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                 1
SFO1      125.7722511 MHz
NUC1              13C
P1              10.50 usec
PLW1      23.00000000 W
SFO2      500.1330008 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2           80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.10610000 W

F2 - Processing parameters
SI             131072
SF        125.7577904 MHz
WDW                EM
SSB      0
LB               1.00 Hz
GB       0
PC               1.40
```



```
Current Data Parameters
NAME      sr-1-131.500
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_         20170217
Time            11.16 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD            65536
SOLVENT         CDCl3
NS               36
DS                2
SWH        31250.000 Hz
FIDRES      0.953674 Hz
AQ         1.0485760 sec
RG            204.54
DW            16.000 usec
DE            18.00 usec
TE            298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0               1
SFO1     125.7722511 MHz
NUC1             13C
P1            10.50 usec
PLW1     23.00000000 W
SFO2     500.1330008 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2          80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.10610000 W

F2 - Processing parameters
SI           131072
SF       125.7577795 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```



23

Current Data Parameters

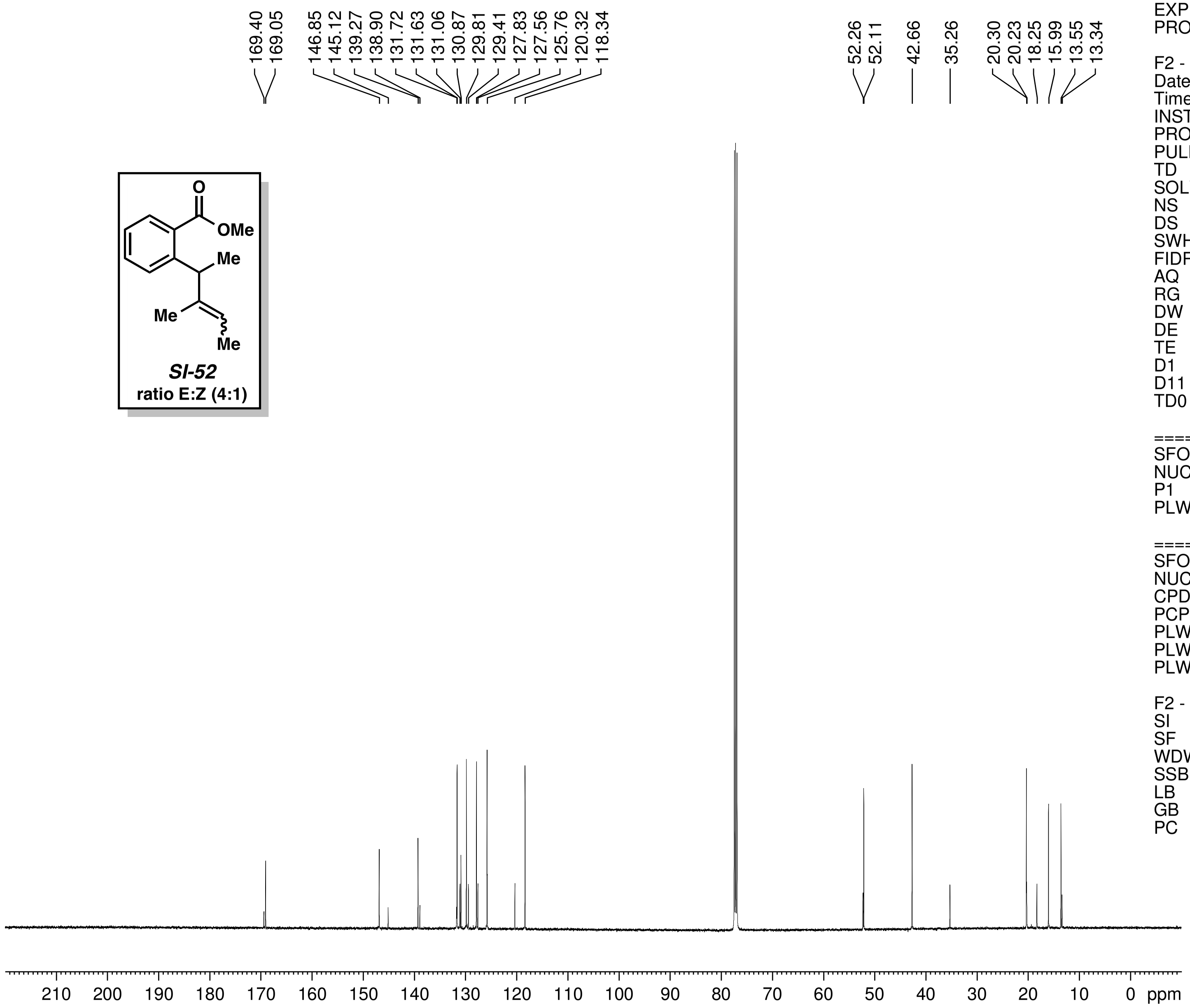
NAME	sr-1-112.500.11
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20170208
Time	16.16 h
INSTRUM	av500
PROBHD	Z119248_0002 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	81
DS	2
SWH	31250.000 Hz
FIDRES	0.953674 Hz
AQ	1.0485760 sec
RG	204.54
DW	16.000 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	125.7722511 MHz
NUC1	13C
P1	10.50 usec
PLW1	23.00000000 W
SFO2	500.1330008 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	80.00 usec
PLW2	13.50000000 W
PLW12	0.21094000 W
PLW13	0.10610000 W

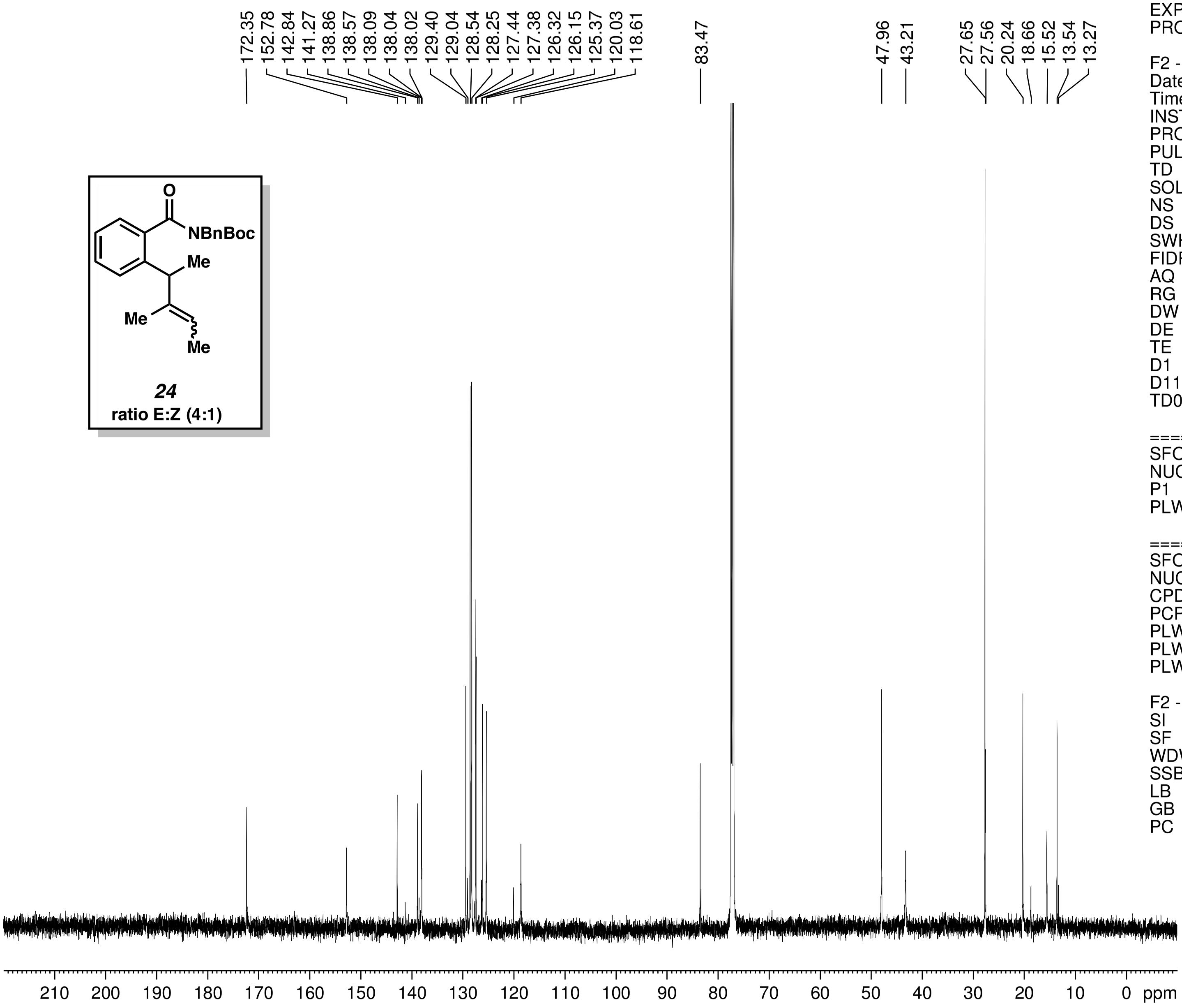
F2 - Processing parameters

SI	131072
SF	125.7577887 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

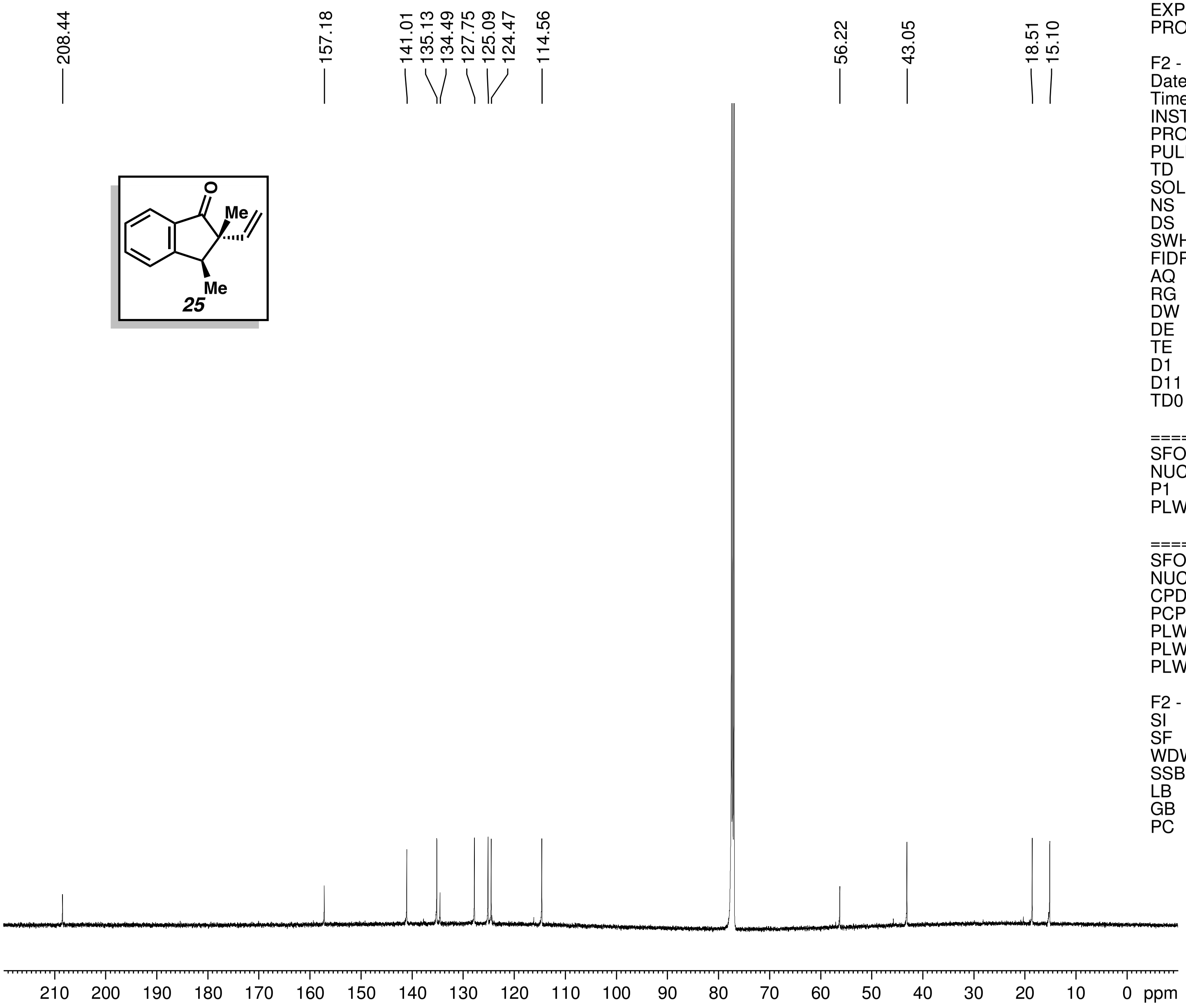


SI-52
ratio E:Z (4:1)

```
Current Data Parameters
NAME     JMM-7-244-char
EXPNO              2
PROCNO           999

F2 - Acquisition Parameters
Date_         20170215
Time            16.37
INSTRUM          av500
PROBHD   5 mm DCH 13C-1
PULPROG         zgpg30
TD              65536
SOLVENT          CDCl3
NS                166
DS                  2
SWH       50000.000 Hz
FIDRES     0.762939 Hz
AQ       0.6553600 sec
RG              204.54
DW          10.000 usec
DE         100.00 usec
TE             298.0 K
D1      2.00000000 sec
D11     0.03000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1    125.7766527 MHz
NUC1              13C
P1            9.63 usec
PLW1    23.00000000 W

======== CHANNEL f2 =====
SFO2    500.1330008 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2         80.00 usec
PLW2    13.50000000 W
PLW12    0.21094000 W
PLW13    0.13500001 W

F2 - Processing parameters
SI             131072
SF      125.7577734 MHz
WDW                EM
SSB     0
LB            1.00 Hz
GB      0
PC               1.40
```



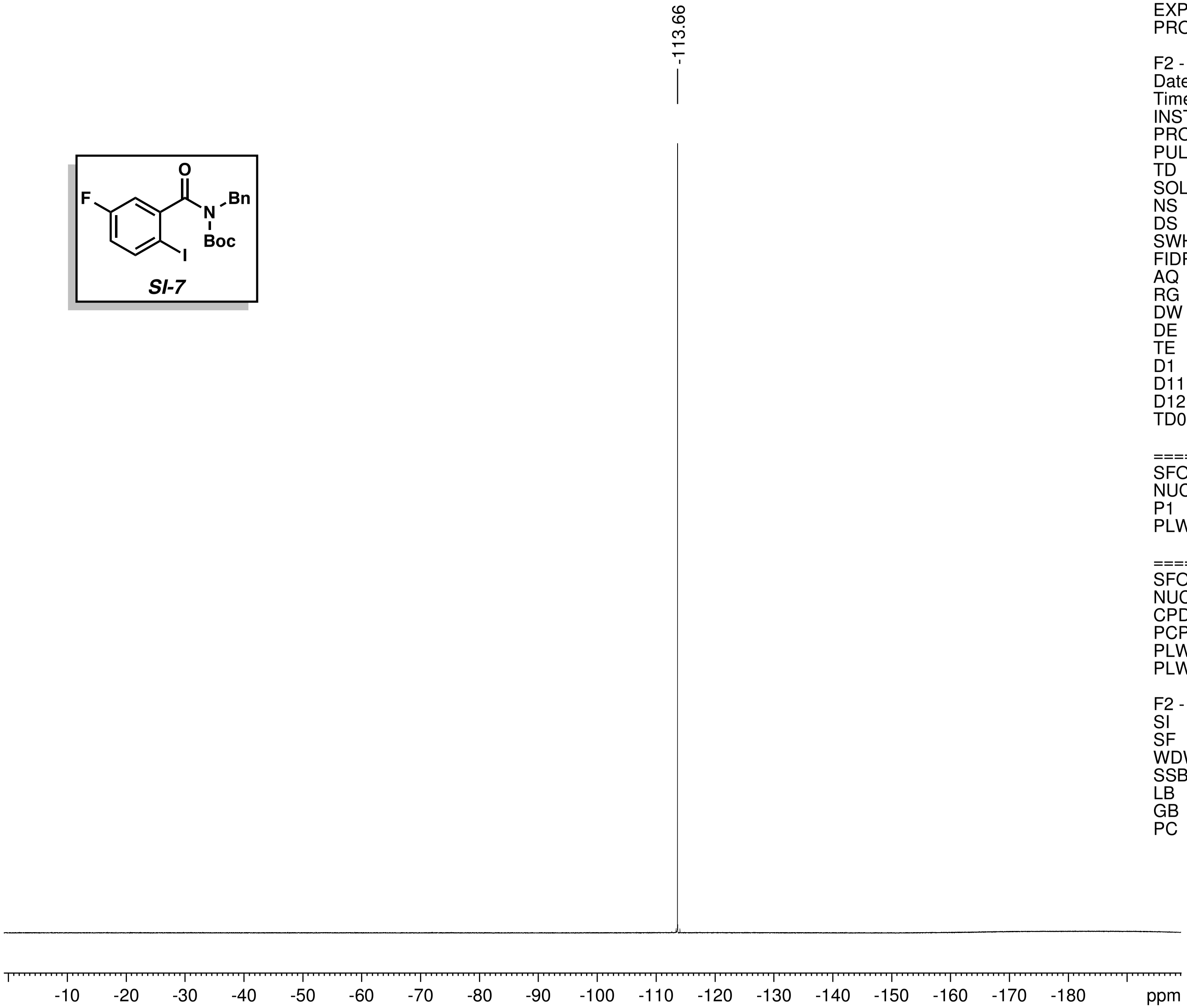
```
Current Data Parameters
NAME     JMM-7-247--char
EXPNO              2
PROCNO           999

F2 - Acquisition Parameters
Date_         20170215
Time             16.16
INSTRUM          av500
PROBHD   5 mm DCH 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 118
DS                   2
SWH       50000.000 Hz
FIDRES     0.762939 Hz
AQ       0.6553600 sec
RG              204.54
DW          10.000 usec
DE           18.00 usec
TE             298.0 K
D1      2.00000000 sec
D11     0.03000000 sec
TD0                  1

======== CHANNEL f1 =====
SFO1    125.7766527 MHz
NUC1               13C
P1             9.63 usec
PLW1     23.00000000 W

======== CHANNEL f2 =====
SFO2    500.1330008 MHz
NUC2                1H
CPDPRG[2       waltz16
PCPD2         80.00 usec
PLW2     13.50000000 W
PLW12     0.21094000 W
PLW13     0.13500001 W

F2 - Processing parameters
SI              131072
SF      125.7577725 MHz
WDW                 EM
SSB    0
LB             1.00 Hz
GB     0
PC                1.40
```



```
Current Data Parameters
NAME     JMM-7-249-char
EXPNO              2
PROCNO           999

F2 - Acquisition Parameters
Date_         20170207
Time             20.08
INSTRUM          av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD             65536
SOLVENT          CDCl3
NS               147
DS                 2
SWH          50000.000 Hz
FIDRES        0.762939 Hz
AQ          0.6553600 sec
RG             204.54
DW              10.000 usec
DE             100.00 usec
TE              298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                 1

======== CHANNEL f1 ====
SFO1      125.7766527 MHz
NUC1              13C
P1               9.63 usec
PLW1      23.00000000 W

======== CHANNEL f2 ====
SFO2      500.1330008 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            80.00 usec
PLW2      13.50000000 W
PLW12      0.21094000 W
PLW13      0.13500001 W

F2 - Processing parameters
SI             131072
SF        125.7577729 MHz
WDW                EM
SSB     0
LB               1.00 Hz
GB      0
PC               1.40
```

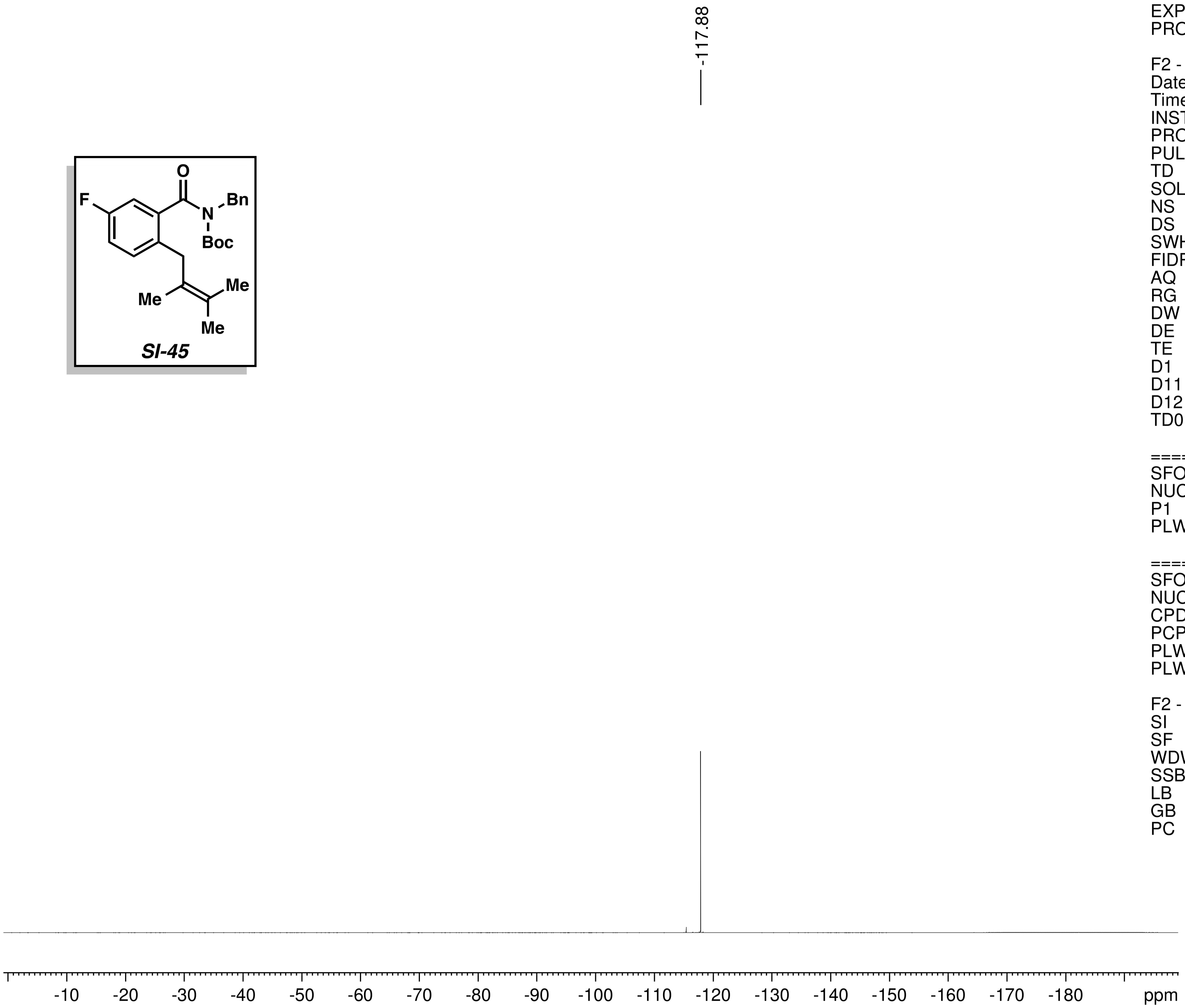
^{19}F NMR Spectra:

SI-7

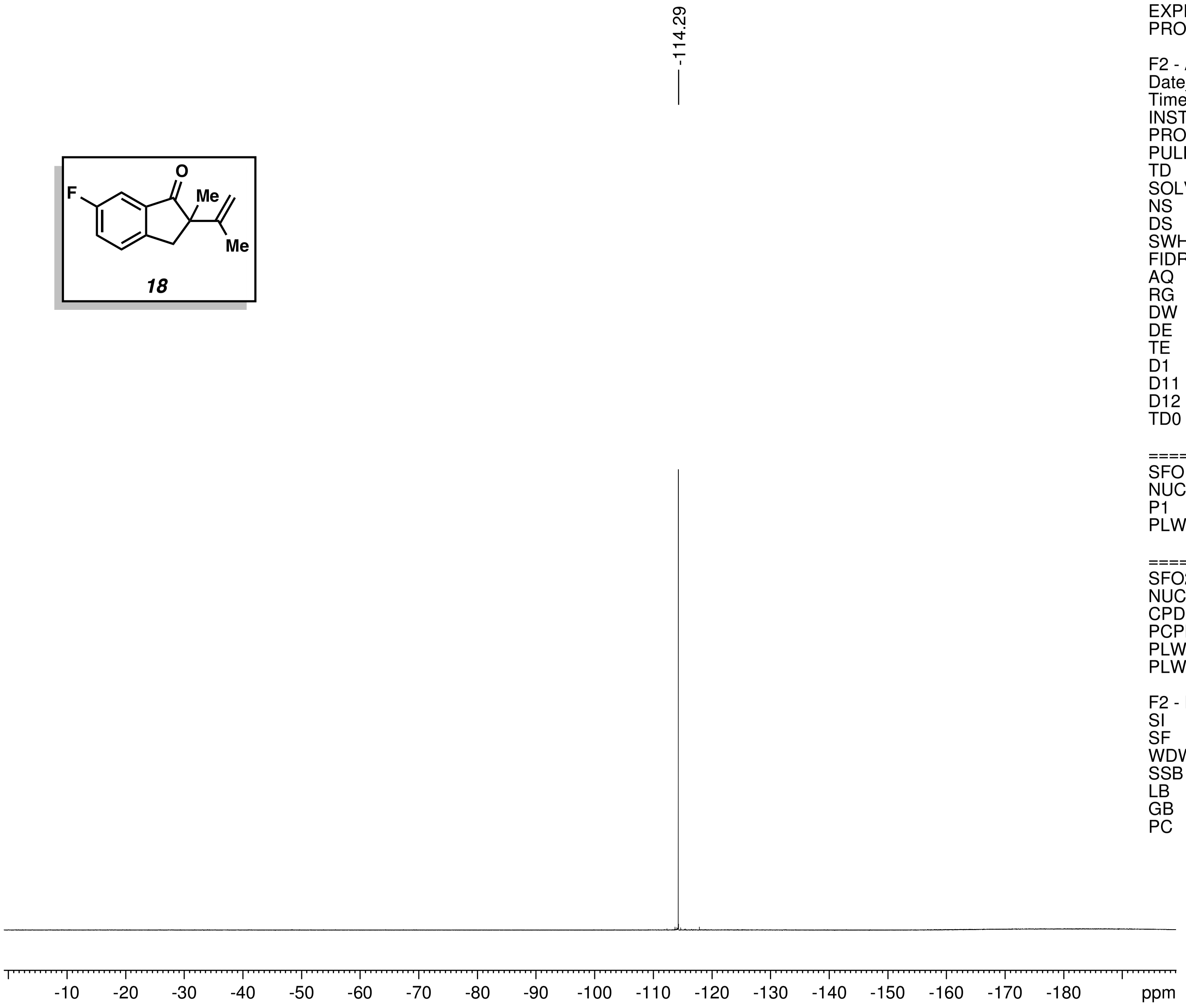


```
Current Data Parameters
NAME          JM-5-054
EXPNO              130
PROCNO               1

F2 - Acquisition Parameters
Date_         20170313
Time             13.33
INSTRUM          av400
PROBHD   5 mm PABBO BB/
PULPROG      zgfhigqn.2
TD              262144
SOLVENT          CDCl3
NS                  48
DS                   0
SWH         150000.000 Hz
FIDRES        0.572205 Hz
AQ           0.8738133 sec
RG              189.85
DW               3.333 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 =====
SFO1       376.4983660 MHz
NUC1               19F
P1               14.50 usec
PLW1       17.00000000 W

======== CHANNEL f2 =====
SFO2       400.1324008 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2            90.00 usec
PLW2       13.00000000 W
PLW12       0.36111000 W

F2 - Processing parameters
SI              262144
SF         376.4983660 MHz
WDW                 EM
SSB     0
LB               1.00 Hz
GB      0
PC                1.00
```

SI-45

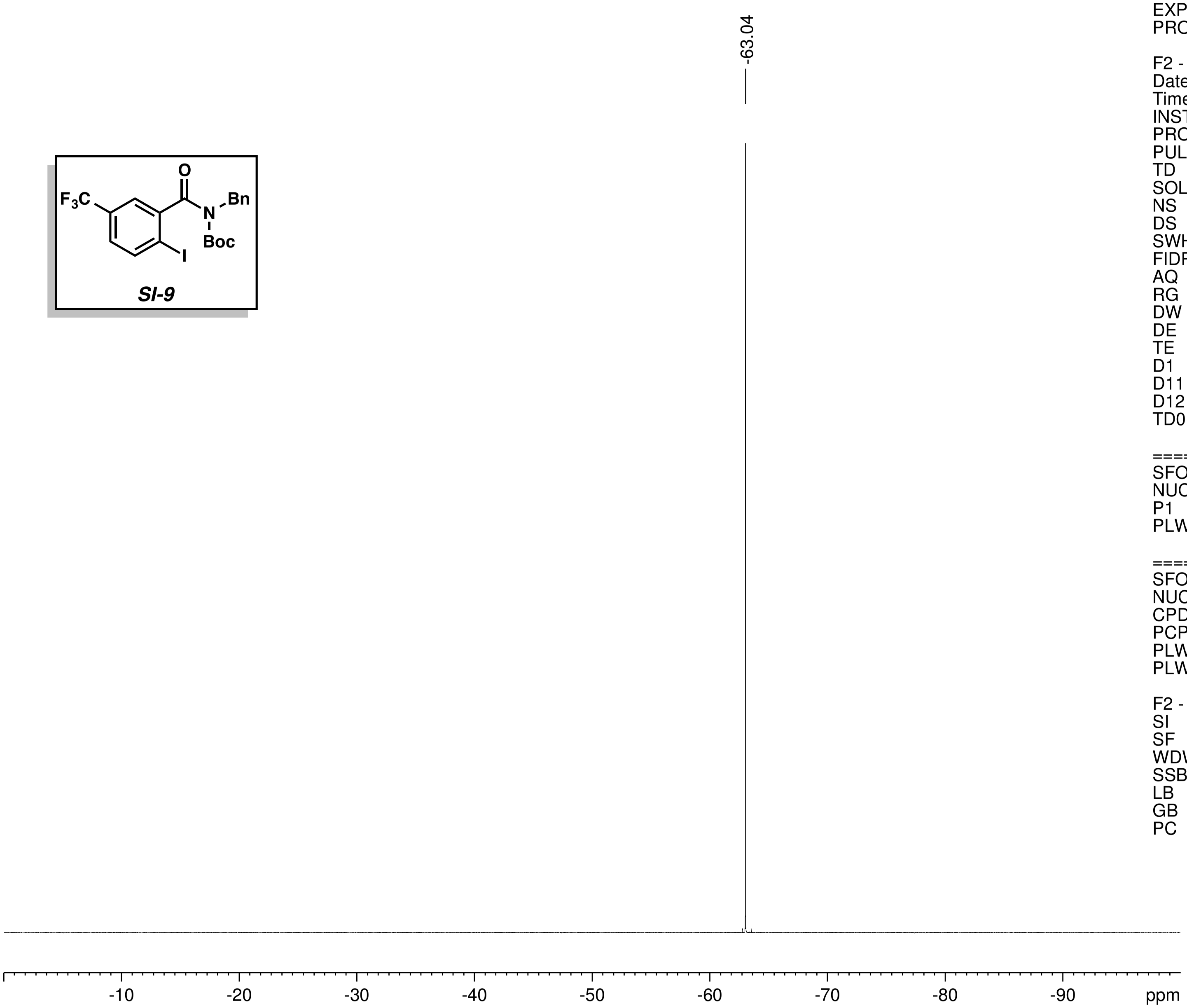


```
Current Data Parameters
NAME          JM-5-067
EXPNO              140
PROCNO               1

F2 - Acquisition Parameters
Date_         20170313
Time             13.39
INSTRUM          av400
PROBHD   5 mm PABBO BB/
PULPROG     zgfhigqn.2
TD              262144
SOLVENT          CDCl3
NS                  48
DS                   0
SWH        150000.000 Hz
FIDRES       0.572205 Hz
AQ         0.8738133 sec
RG              189.85
DW             3.333 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 =====
SFO1     376.4983660 MHz
NUC1               19F
P1              14.50 usec
PLW1       17.00000000 W

======== CHANNEL f2 =====
SFO2     400.1324008 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2          90.00 usec
PLW2       13.00000000 W
PLW12       0.36111000 W

F2 - Processing parameters
SI              262144
SF       376.4983660 MHz
WDW                 EM
SSB     0
LB             1.00 Hz
GB      0
PC                1.00
```

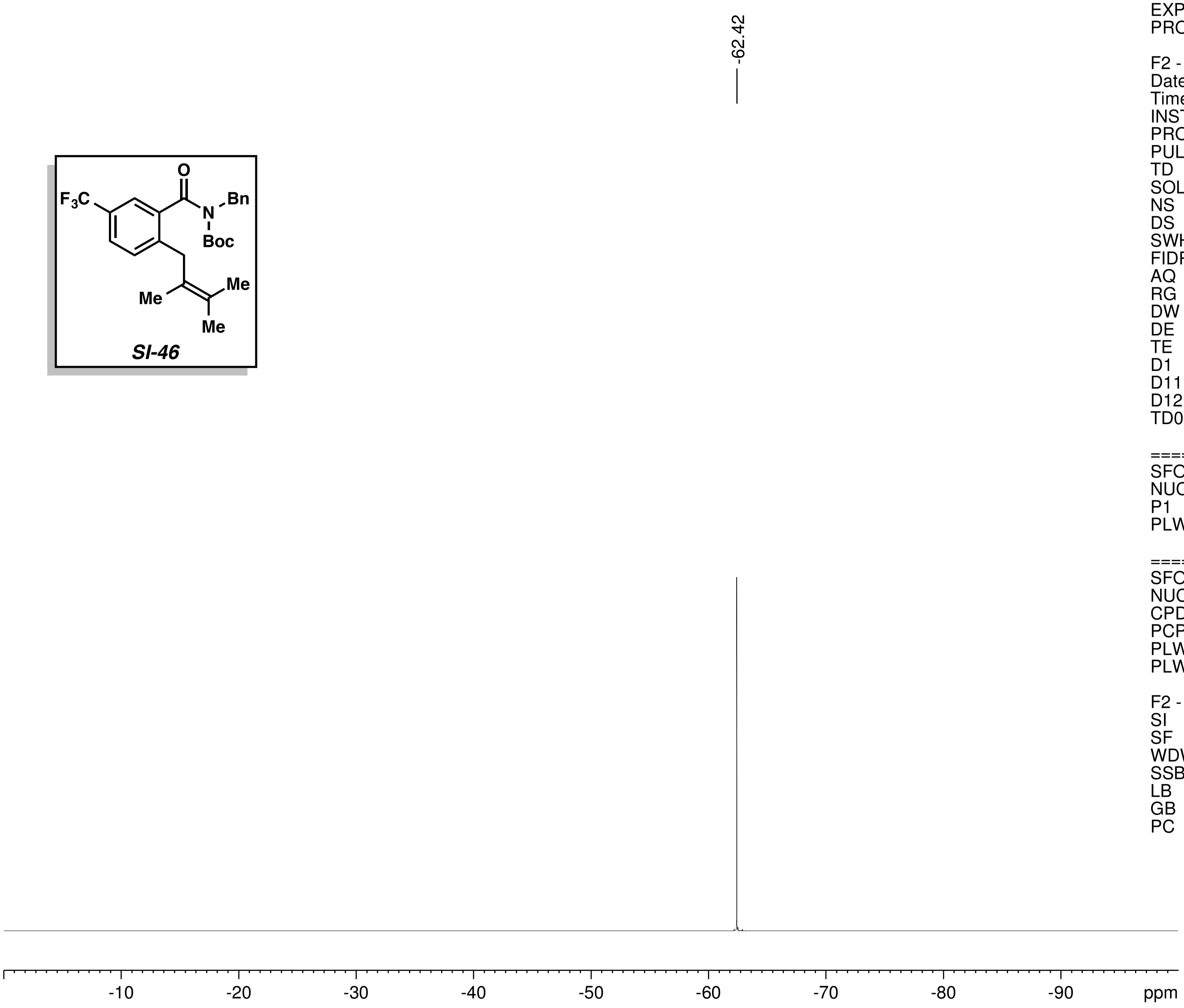


18

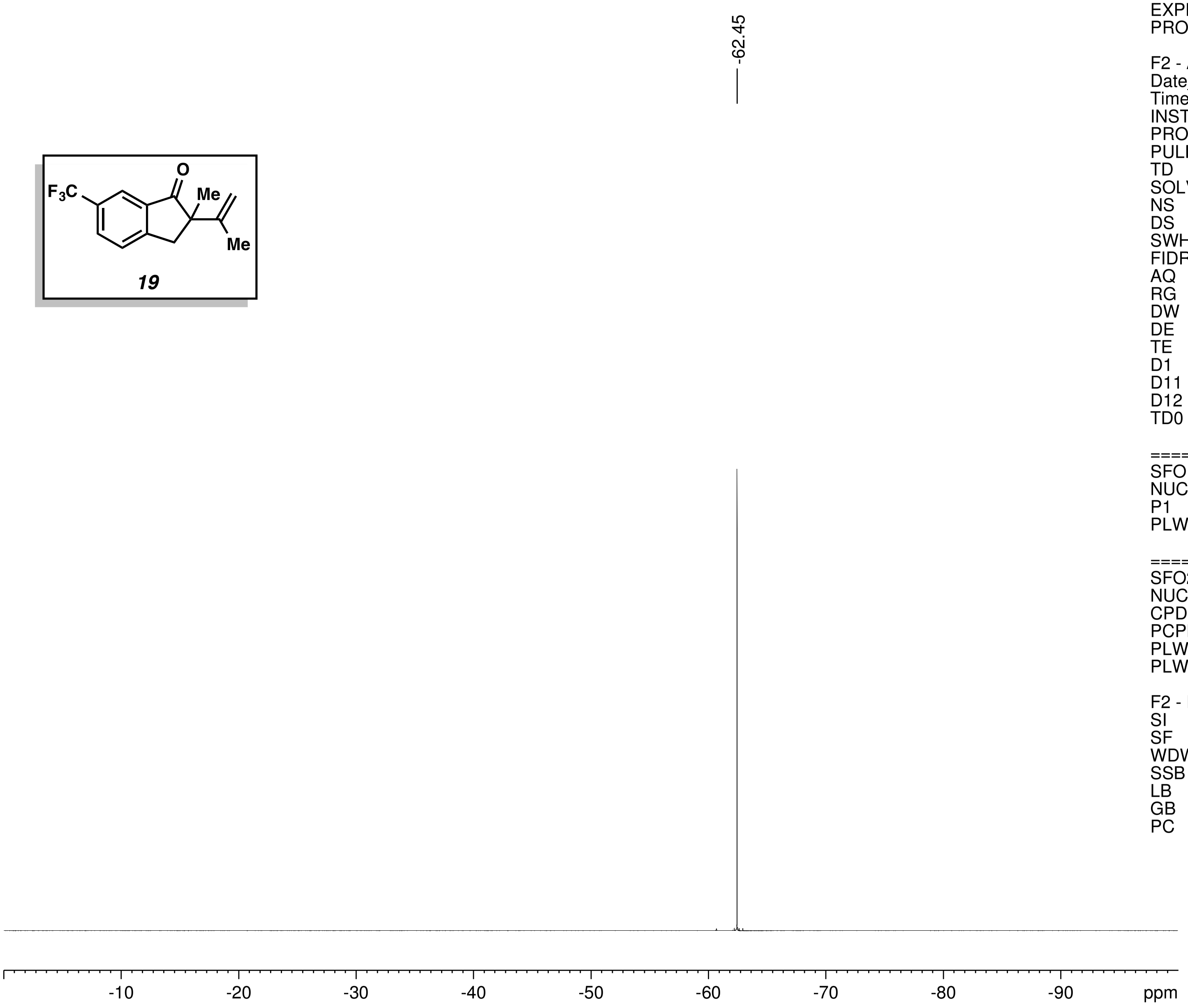
```
Current Data Parameters
NAME          JM-5-067
EXPNO              150
PROCNO               1

F2 - Acquisition Parameters
Date_         20170313
Time             13.45
INSTRUM          av400
PROBHD   5 mm PABBO BB/
PULPROG     zgfhigqn.2
TD              262144
SOLVENT          CDCl3
NS                  48
DS                   0
SWH        150000.000 Hz
FIDRES       0.572205 Hz
AQ          0.8738133 sec
RG              189.85
DW               3.333 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 =====
SFO1      376.4983660 MHz
NUC1               19F
P1               14.50 usec
PLW1       17.00000000 W

======== CHANNEL f2 =====
SFO2      400.1324008 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2       13.00000000 W
PLW12       0.36111000 W

F2 - Processing parameters
SI              262144
SF        376.4983660 MHz
WDW                 EM
SSB     0
LB                1.00 Hz
GB      0
PC                1.00
```



```
Current Data Parameters
NAME          JM-5-037
EXPNO              160
PROCNO               1

F2 - Acquisition Parameters
Date_         20170313
Time             13.51
INSTRUM          av400
PROBHD   5 mm PABBO BB/
PULPROG     zgfhigqn.2
TD              262144
SOLVENT          CDCl3
NS                  48
DS                   0
SWH        150000.000 Hz
FIDRES        0.572205 Hz
AQ           0.8738133 sec
RG              189.85
DW               3.333 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 =====
SFO1       376.4983660 MHz
NUC1               19F
P1               14.50 usec
PLW1       17.00000000 W

======== CHANNEL f2 =====
SFO2       400.1324008 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2       13.00000000 W
PLW12       0.36111000 W

F2 - Processing parameters
SI              262144
SF         376.4983660 MHz
WDW                 EM
SSB     0
LB                1.00 Hz
GB      0
PC                1.00
```



```
Current Data Parameters
NAME          JM-5-055
EXPNO              170
PROCNO               1

F2 - Acquisition Parameters
Date_         20170313
Time             13.57
INSTRUM          av400
PROBHD   5 mm PABBO BB/
PULPROG     zgfhigqn.2
TD              262144
SOLVENT          CDCl3
NS                  48
DS                   0
SWH        150000.000 Hz
FIDRES        0.572205 Hz
AQ          0.8738133 sec
RG              189.85
DW               3.333 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 =====
SFO1      376.4983660 MHz
NUC1               19F
P1               14.50 usec
PLW1       17.00000000 W

======== CHANNEL f2 =====
SFO2      400.1324008 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2            90.00 usec
PLW2       13.00000000 W
PLW12       0.36111000 W

F2 - Processing parameters
SI              262144
SF        376.4983660 MHz
WDW                 EM
SSB     0
LB                1.00 Hz
GB      0
PC                1.00
```



```
Current Data Parameters
NAME          JM-5-057
EXPNO              180
PROCNO               1

F2 - Acquisition Parameters
Date_         20170313
Time             14.03
INSTRUM          av400
PROBHD   5 mm PABBO BB/
PULPROG     zgfhigqn.2
TD             262144
SOLVENT          CDCl3
NS                  48
DS                   0
SWH        150000.000 Hz
FIDRES       0.572205 Hz
AQ          0.8738133 sec
RG              189.85
DW               3.333 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 =====
SFO1       376.4983660 MHz
NUC1               19F
P1               14.50 usec
PLW1       17.00000000 W

======== CHANNEL f2 =====
SFO2       400.1324008 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2       13.00000000 W
PLW12       0.36111000 W

F2 - Processing parameters
SI             262144
SF         376.4983660 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.00
```