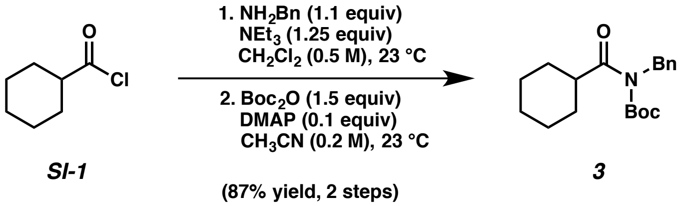
Supporting Information – Table of Contents

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. Reagents **SI-1**, **SI-2a**, **SI-2b, SI-3**, **SI-5**, **SI-7**, **SI-9, SI-11, SI-13**, **SI-16** and **SI-23** were purchased from Sigma-Aldrich. Reagents **SI-14**, **SI-17**, **SI-19**, **SI-21**, **rac-SI-21** and **rac-SI-23** were purchased from Combi-Blocks, Inc. Alcohols **4, 7** and **SI-28**, **SI-29**, **SI-30**, **SI-32**, **SI-33**, **SI-35**, **SI-36**, **SI-37**, **SI-38** were purchased from Sigma-Aldrich. Alcohols **SI-31** and **SI-34** were purchased from Combi-Blocks, Inc. Benzylamine and LHMDS were purchased from Sigma-Aldrich. EDC and HOBt were purchased from Chem-Impex International. DMAP and Boc_2O were purchased from Oakwood Products, Inc. Toluene was purified by distillation and taken through five freeze-pump-thaw cycles prior to use. Pivaloyl chloride (**SI-16**) and 1-hexanol (**7**) were distilled over CaH_2 prior to use. HNBnBoc was prepared by known literature conditions.[1] $Ni(cod)_2$ was obtained from Strem Chemicals. $Ni(cod)_2$ – paraffin capsule samples were obtained from TCI Chemicals (B5417). Terpyridine was obtained from Sigma-Aldrich. 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, ceric ammonium molybdate, iodine, vanillin, and potassium permanganate staining techniques. Silicycle Siliaflash P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^1H NMR spectra were recorded on Bruker spectrometers (at 500 MHz) and are reported relative to residual solvent signals. Data for ^1H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz), and integration. Data for ^{13}C NMR are reported in terms of chemical shift (at 125 MHz). 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 on Thermo Scientific™ Exactive Mass Spectrometer with DART ID-CUBE, Waters GST premier, and Waters LCT premier. Optical rotations were measured with a Rudolf Autopol III Automatic Polarimeter. Determination of enantiopurity was carried out on an Agilent 1260 HPLC (high performance liquid chromatography) and a Mettler Toledo SFC (supercritical fluid chromatography) using a Daicel ChiralPak OD-H columns and Daicel ChiralPak IA-H.

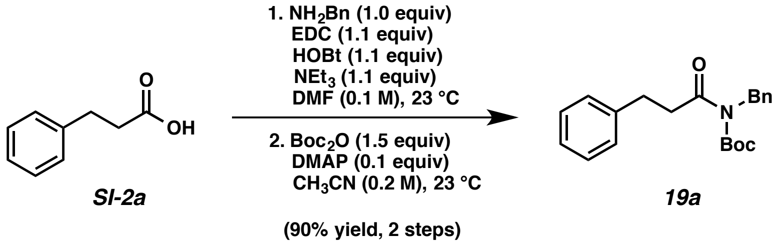
Experimental Procedure

A. Syntheses of Starting Materials



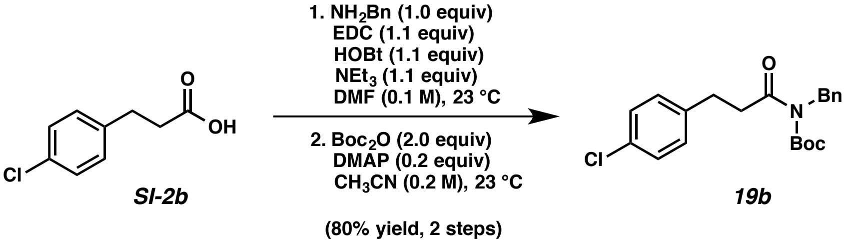
Amide 3. To a solution of acid chloride **SI-1** (2.0 g, 13.64 mmol, 1.0 equiv) and triethylamine (2.40 mL, 17.05 mmol, 1.25 equiv) in dichloromethane (20 mL) was added a solution of benzylamine (1.64 mL, 15.0 mmol, 1.1 equiv) in dichloromethane (7 mL, 0.5 M). The reaction mixture was stirred at 23 °C for 1 h, diluted with CH_2Cl_2 (30 mL), and then washed successively with 1.0 M HCl (25 mL) and brine (25 mL). The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude solid material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (84.3 mg, 0.69 mmol, 0.1 equiv), followed by acetonitrile (35 mL, 0.2 M). Boc_2O (2.26 g, 10.35 mmol, 1.5 equiv) was added in one portion. The reaction vessel was flushed with N_2, and then the reaction mixture was allowed to stir at 23 °C for 12 h. The reaction mixture was diluted with EtOAc (50 mL), and then washed with 1.0 M HCl (20 mL) and brine (20 mL). The organics were dried over Na_2SO_4 and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (20:1 Hexanes:EtOAc) to yield amide **3** (1.91 g, 87% yield, over two steps) as a white solid. Amide **3**: mp: 45.2–47.1 °C; R_f 0.50 (10:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30–7.20 (m, 5H), 4.88 (s, 2H), 3.43 (tt, J = 11.4, 3.2, 1H), 1.90–1.87 (m, 2H), 1.80–1.76 (m, 2H), 1.70–1.66 (m, 1H), 1.51–1.43 (m, 1H), 1.47 (s, 9H), 1.37–1.18 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 179.8, 153.3, 138.7, 128.4, 127.6, 127.1, 83.1, 47.8, 44.9, 30.0, 28.0, 26.1, 25.9; IR (film): 2925, 2857, 1732, 1697, 1460, 1216 cm^{-1}; HRMS-ESI (m/z) $[M + Na]^+$ calcd for $C_{19}H_{27}NO_3Na$ 340.1889; found 340.1881.



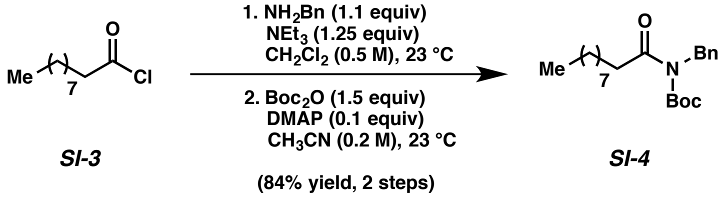
Amide 19a. To a mixture of carboxylic acid **SI-2a** (10.50 g, 70.0 mmol, 1.0 equiv), EDC (14.80 g, 77.0 mmol, 1.1 equiv), HOBt (10.4 g, 77.0 mmol, 1.1 equiv), triethylamine (10.7 mL, 77.0 mmol, 1.1 equiv), and DMF (700 mL, 0.1 M) was added benzylamine (8.24 g, 77.0 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 12 h, and then diluted with deionized water (500 mL) and EtOAc (500 mL). The layers were separated and the organic layer was washed successively with 1.0 M HCl (300 mL), saturated aqueous $NaHCO_3$ (300 mL), and brine (300 mL). The organics were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude solid material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (0.86 g, 7.0 mmol, 0.1 equiv) followed by acetonitrile (350 mL, 0.2 M). Boc_2O (22.92 g, 105.0 mmol, 1.5 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 12 h. The reaction mixture was diluted with EtOAc (50 mL) and then washed with saturated aqueous $NaHCO_3$ (2 X 100 mL) and brine (100 mL). The organics were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (20:1 Hexanes:EtOAc) to yield amide **19a** (21.50 g, 90% yield, over two steps) as a white solid. Amide **19a**: mp: 65.3–67.1 °C; R_f 0.60 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31–7.27 (m, 4H), 7.25–7.18 (m, 6H), 4.89 (s, 2H), 3.28 (t, *J* = 7.7 , 2H), 3.03 (t, *J* = 7.7, 2H), 1.39 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.5, 153.2, 141.3, 138.4, 128.7, 128.5, 128.4, 127.6, 127.2, 126.1, 83.3, 47.5, 40.1, 31.3, 28.0; IR (film): 3032, 2979, 1731, 1693, 1373, 1210 cm^{-1}; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{21}H_{25}NO_3Na$ 362.1732; found 362.1739.



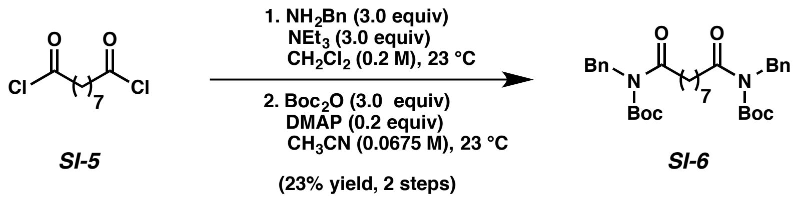
Amide 19b. To a mixture of carboxylic acid **SI-2b** (1.0 g, 5.42 mmol, 1.0 equiv), EDC (1.14 g, 5.96 mmol, 1.1 equiv), HOBt (805 mg, 5.96 mmol, 1.1 equiv), triethylamine (0.83 mL, 5.96 mmol, 1.1 equiv), and DMF (54 mL, 0.1 M) was added benzylamine (861 mL, 5.96 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 12 h, and then diluted with deionized water (50 mL) and EtOAc (50 mL). The aqueous layer was extracted with EtOAc (3 X 50 mL). The combined organic layers were washed with deionized water (3 X 100 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (132 mg, 1.08 mmol, 0.2 equiv), followed by acetonitrile (27 mL, 0.2 M). Boc_2O (2.4 g, 10.8 mmol, 2.0 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 15 h. The reaction was quenched by the addition of saturated aqueous $NaHCO_3$ (50 mL). The mixture was transferred to a separatory funnel with EtOAc (75 mL) and H_2O (50 mL). The aqueous layer was extracted with EtOAc (3 X 75 mL). The combined organic layers were dried over Na_2SO_4, filtered and then concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (19:1 Hexanes:EtOAc) to yield amide **19b** (1.6 g, 80% yield, over two steps) as a clear oil. Amide **19b**: R_f 0.59 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32–7.26 (m, 2H), 7.26–7.18 (m, 5H), 7.18–7.12 (m, 2H), 4.87 (s, 2H), 3.22 (app t, 2H), 2.96 (app t, 2H), 1.39 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.2, 153.2, 139.8, 138.3, 131.9, 130.1, 128.6, 128.4, 127.6, 127.3, 83.5, 47.5, 40.0, 30.7, 28.0; IR (film): 3034, 2977, 2929, 1731, 1696, 1368 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{21}H_{24}ClNO_3$ 374.15230; found 374.14960.



Amide SI-4. To a solution of acid chloride **SI-3** (2.0 g, 10.5 mmol, 1.0 equiv), triethylamine (1.82 mL, 13.13 mmol, 1.25 equiv), and dichloromethane (16 mL), was added a solution of benzylamine (1.26 mL, 11.55 mmol, 1.1 equiv) in dichloromethane (5 mL, 0.5 M). The reaction mixture was stirred at 23 °C for 1 h, diluted with CH_2Cl_2 (30 mL), and then washed successively with 1.0 M HCl (25 mL) and brine (25 mL). The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude solid material was used in the subsequent step without further purification.

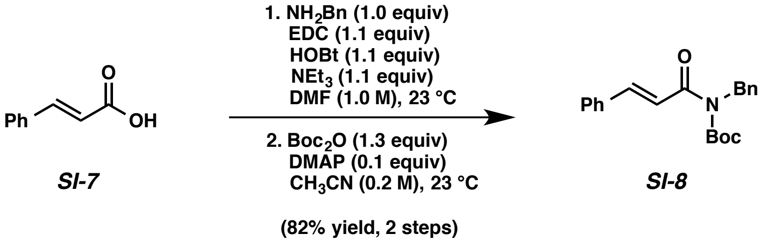
To a flask containing the crude material from the previous step was added DMAP (69.6 mg, 0.57 mmol, 0.1 equiv) followed by acetonitrile (28.5 mL, 0.2 M). Boc_2O (1.87 g, 8.6 mmol, 1.5 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 12 h. The reaction mixture was diluted with EtOAc (50 mL), and then washed with 1.0 M HCl (20 mL) and brine (20 mL). The organics were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (20:1 Hexanes:EtOAc) to yield amide **SI-4** (1.76 g, 84% yield, over two steps) as a clear oil. Amide **SI-4**: R_f 0.48 (10:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30–7.27 (m, 2H), 7.25–7.21 (m, 3H), 4.88 (s, 2H), 2.92 (t, *J* = 7.5, 2H), 1.69–1.63 (m, 2H), 1.41 (s, 9H), 1.34–1.26 (m, 12H), 0.89 (t, *J* = 7.0, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.4, 153.3, 138.6, 128.4, 127.8, 127.2, 83.2, 47.4, 38.5, 32.0, 29.6, 29.4, 29.3, 28.0, 25.4, 22.8, 14.2; IR (film): 2925, 2857, 1732, 1697, 1460, 1216 cm^{-1}; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{22}H_{35}NO_3Na$ 384.2515; found 384.2516.



Amide SI-6. To a solution of acid chloride **SI-5** (2.0 g, 8.9 mmol, 1.0 equiv), triethylamine (3.72 mL, 26.7 mmol, 3.0 equiv), and dichloromethane (80 mL), was added a solution of benzylamine

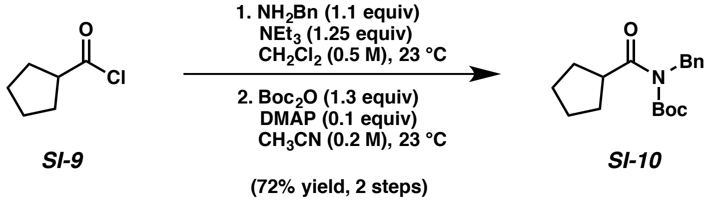
(2.92 mL, 26.7 mmol, 3.0 equiv) in dichloromethane (10 mL, 0.1 M). The reaction mixture was stirred at 23 °C for 4 h and then filtered over celite. The pad was eluted with CH_2Cl_2 (40 mL). The collected organics were washed successively with 1.0 M HCl (25 mL) and brine (25 mL). The organics were dried over Na_2SO_4, filtered and then concentrated under reduced pressure. The resulting crude solid material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (108.7 mg, 0.89 mmol, 0.1 equiv), followed by acetonitrile (44 mL, 0.2 M). Boc_2O (5.83 g, 26.7 mmol, 3.0 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 12 h. The reaction mixture was diluted with EtOAc (50 mL) and then washed with deionized water (3 X 50 mL). The organics were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (20:1 → 10:1 Hexanes:EtOAc) to yield amide **SI-6** as a white solid (1.16 g, 23% yield, over two steps). Amide **SI-6**: mp: 67.0–69.2 °C; R_f 0.33 (20:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30–7.27 (m, 4H), 7.25–7.20 (m, 6H), 4.88 (s, 4H), 2.90 (t, J = 7.6, 4H), 1.66–1.63 (m, 4H), 1.40 (s, 18H), 1.34–1.31 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.4, 153.3, 138.6, 128.4, 127.7, 127.2, 83.2, 47.4, 38.4, 29.4, 29.3, 28.0, 25.3; IR (film): 3703, 3668, 2977, 2862, 1732, 1695, 1458 cm^{-1}; HRMS-ESI (m/z) [M+Na]+ calcd for $C_{33}H_{46}N_2O_6Na$ 589.3254; found 589.3254.



Amide SI-8. To a mixture of carboxylic acid **SI-7** (3.0 g, 20.2 mmol, 1.0 equiv), EDC (3.5 g, 22.3 mmol, 1.1 equiv), HOBt (3.4 g, 22.3 mmol, 1.1 equiv) and triethylamine (3.1 mL, 22.3 mmol, 1.1 equiv) in DMF (202 mL, 1.0 M) was added benzylamine (2.4 mL, 22.3 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 17 h, and then diluted with deionized water (100 mL). The mixture was transferred to a separatory funnel with EtOAc (100 mL) and brine (100 mL). The aqueous layer was extracted with EtOAc (3 X 100 mL). The combined organic layers were washed with deionized water (3 X 100 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

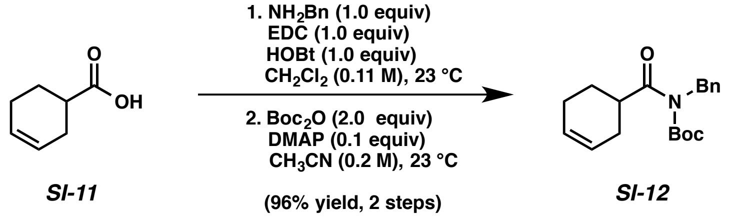
To a flask containing the crude material from the previous step was added DMAP (239 mg, 1.96 mmol, 0.1 equiv), followed by acetonitrile (98 mL, 0.2 M). Boc_2O (5.6 g, 25.5 mmol, 1.3 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 19 h. The reaction was quenched by the addition of saturated aqueous $NaHCO_3$ (75 mL). The mixture was transferred to a separatory funnel with EtOAc (100 mL) and H_2O (50 mL). The aqueous layer was extracted with EtOAc (3 X 100 mL). The combined organic layers were dried over Na_2SO_4, filtered and then concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (25:1 Hexanes:EtOAc) to yield amide **SI-8** (4.2 g, 82% yield, over two steps) as a white solid. Amide **SI-8**: mp: 89.1–92.9 ºC; R_f 0.46 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.78–7.71 (m, 1H), 7.60–7.55 (m, 2H), 7.55–7.49 (m, 1H), 7.41–7.34 (m, 3H), 7.33–7.28 (m, 4H), 7.26–7.21 (m, 1H), 4.97 (s, 2H), 1.43 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.0, 153.4, 143.7, 138.5, 135.3, 130.1, 128.9, 128.5, 128.3, 127.7, 127.3, 121.4, 83.5, 48.0, 28.1; IR (film): 2980, 2935, 1722, 1667, 1615, 1348 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{21}H_{24}NO_3$ 338.1756; found 338.1738.



SI-9 ***SI-10***

Amide SI-10. To a solution of acid chloride **SI-9** (2.0 g, 15.1 mmol, 1.0 equiv) and triethylamine (2.61 mL, 18.9 mmol, 1.25 equiv) in dichloromethane (15.1 mL), was added a solution of benzylamine (1.81 mL, 16.6 mmol, 1.1 equiv) in dichloromethane (15.1 mL, 0.5 M). The reaction mixture was stirred at 23 °C for 15 h. The mixture was diluted with EtOAc (50 mL), and then washed successively with 1.0 M HCl (50 mL) and brine (50 mL). The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

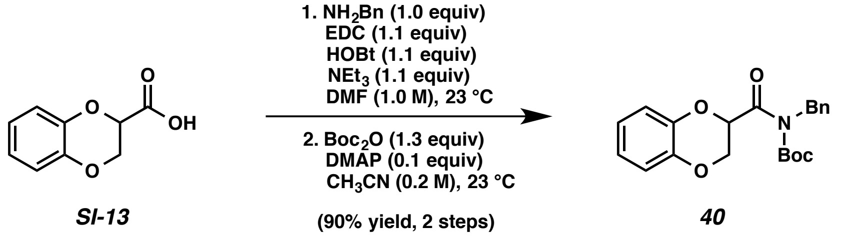
To a flask containing the crude material from the previous step was added DMAP (184.0 mg, 1.51 mmol, 0.1 equiv) followed by acetonitrile (76.0 mL, 0.2 M). Boc_2O (4.3 g, 19.6 mmol, 1.3 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 24 h. The reaction was quenched by the addition of saturated aqueous $NaHCO_3$ (30 mL), and then transferred to a separatory funnel with EtOAc (50 mL) and H_2O (50 mL). The aqueous layer was extracted with EtOAc (3 X 50 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (20:1 Hexanes:EtOAc) to yield amide **SI-10** (1.97 g, 72% yield, over two steps) as a clear oil. Amide **SI-10**: R_f 0.59 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32–7.27 (m, 2H), 7.25–7.20 (m, 3H), 4.88 (s, 2H), 3.83–3.72 (m, 1H), 2.01–1.88 (m, 2H), 1.87–1.77 (m, 2H), 1.77–1.66 (m, 2H), 1.64–1.53 (m, 2H), 1.40 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 179.9, 153.3, 138.7, 128.4, 127.5, 127.1, 83.1, 47.8, 45.7, 31.0, 28.0, 26.1; IR (film): 2955, 2867, 1730, 1687, 1453, 1213 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{18}H_{26}NO_3$, 304.1913; found 304.1895.



Amide SI-12. To a mixture of carboxylic acid **SI-11** (1.0 g, 7.9 mmol, 1.0 equiv), EDC (1.50 g, 7.9 mmol, 1.0 equiv), HOBt (1.1 g, 7.9 mmol, 1.0 equiv), and CH_2Cl_2 (79 mL, 0.1 M) was added benzylamine (0.86 mL, 7.9 mmol, 1.0 equiv). The resulting mixture was stirred at 23 °C for 16 h, and then diluted with CH_2Cl_2 (30 mL). The mixture was washed with 1M HCl (30 mL), saturated aqueous $NaHCO_3$ (30mL), and brine (30 mL). The organics were dried over Na_2SO_4, filtered and then concentrated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (56.7 mg, 0.46 mmol, 0.1 equiv), followed by acetonitrile (23 mL, 0.2 M). Boc_2O (2.03 g, 9.28 mmol, 2.0 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 12 h. The mixture was washed with 1.0 M HCl (5 mL) and brine (5 mL). After drying over Na_2SO_4, filtered and the organics were concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (10:1 Hexanes:EtOAc) to yield **SI-12** (1.40 g, 96% yield, over two steps) as a clear oil. Amide **SI-12**:

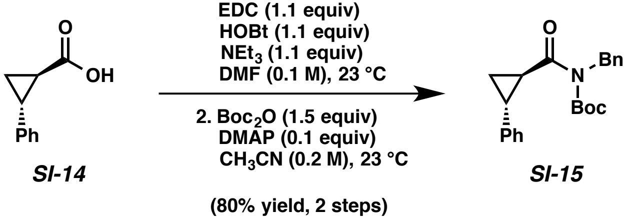
R_f 0.69 (5:1 Hexanes: EtOAc); ^{1}H NMR (500 MHz, CDCl3): δ 7.31–7.28 (m, 2H), 7.24–7.21 (m, 3H), 5.73–5.67 (m, 2H), 4.88 (d, *J* = 2.9, 2H), 3.66–3.60 (m, 1H), 2.32–2.23 (m, 2H), 2.14–2.10 (m, 2H), 1.98–1.94 (m, 1H), 1.75–1.67 (m, 1H), 1.40 (br s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ179.6, 153.2, 138.6, 128.4, 127.6, 127.2, 126.7, 125.7, 83.3, 47.8, 41.1, 28.6, 28.0, 26.3, 25.1; IR (film): 3030, 2980, 1730, 1690, 1438, 1368; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{19}H_{25}NO_3Na$ 338.1732; found 338.1725.



Amide 40. To a mixture of carboxylic acid **SI-13** (2.0 g, 11.1 mmol, 1.0 equiv), EDC (1.89 g, 12.2 mmol, 1.1 equiv), HOBt (1.87 g, 12.2 mmol, 1.1 equiv), and triethylamine (1.7 mL, 22.3 mmol, 1.1 equiv) in DMF (202 mL, 1.0 M) was added benzylamine (2.4 mL, 12.2 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 18 h, and then diluted with deionized water (100 mL). The mixture was transferred to a separatory funnel with EtOAc (75 mL) and brine (50 mL). The aqueous layer was extracted with EtOAc (3 X 75 mL). The combined organic layers were washed with deionized water (3 X 75 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

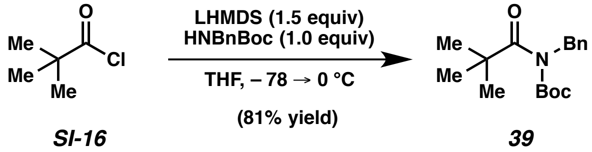
To a flask containing the crude material from the previous step was added DMAP (92 mg, 0.78 mmol, 0.1 equiv) followed by acetonitrile (39 mL, 0.2 M). Boc_2O (2.2 g, 10.1 mmol, 1.3 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 17 h. The reaction was quenched by the addition of saturated aqueous $NaHCO_3$ (75 mL), and then transferred to a separatory funnel with EtOAc (100 mL) and H_2O (50 mL). The layers were separated. The aqueous layer was extracted with EtOAc (3 X 100 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (29:1 → 15:1 Hexanes:EtOAc) to yield amide **40** (2.3 g, 90% yield, over two steps) as a white solid. Amide **40**: mp: 76.5–78.5 ºC; R_f 0.57 (5:1 Hexanes:EtOAc); ^{1}H NMR

(500 MHz, $CDCl_3$): δ 7.33–7.27 (m, 2H), 7.26–7.20 (m, 3H), 7.00–6.94 (m, 1H), 6.92–6.81 (m, 3H), 5.81–5.76 (m, 1H), 4.96–4.80 (m, 2H), 4.50–4.39 (m, 2H), 1.43 (s, 9H), ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.9, 152.9, 143.4, 143.3, 137.5, 128.6, 127.8, 127.5, 122.2, 121.6, 117.2, 117.15, 84.6, 73.9, 65.2, 48.1, 28.0; IR (film): 2980, 2932, 1730, 1710, 1595, 1493 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{21}H_{24}NO_5$ 370.1654; found 370.1591.

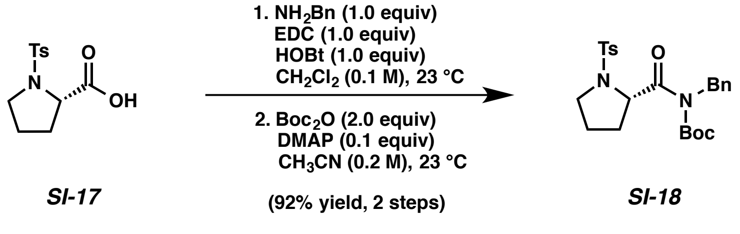


Amide SI-15: To a mixture of carboxylic acid **SI-14** (1.0 g, 6.17 mmol, 1.0 equiv), EDC (1.30 g, 6.79 mmol, 1.1 equiv), HOBt (917 mg, 6.79 mmol, 1.1 equiv), triethylamine (0.94 mL, 6.79 mmol, 1.1 equiv), and DMF (61.7 mL, 0.1 M) was added benzylamine (0.74 mL, 6.79 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 12 h, and then diluted with deionized water (50 mL). The mixture was transferred to a separatory funnel with EtOAc (50 mL) and brine (50 mL). The aqueous layer was extracted with EtOAc (3 X 50 mL). The combined organic layers were washed with deionized water (3 X 50 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude solid material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (75 mg, 0.62 mmol, 0.1 equiv) followed by acetonitrile (31 mL, 0.2 M). Boc_2O (1.75 g, 8.02 mmol, 1.5 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 12 h. The reaction mixture was diluted with EtOAc (50 mL) and then washed with saturated aqueous $NaHCO_3$ (2 X 50 mL) and brine (50 mL). The organics were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (29:1 → 20:1 Hexanes:EtOAc) to yield amide **SI-15** (1.29 g, 80% yield, over two steps) as a clear oil. Amide **SI-15**: R_f 0.50 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33–7.26 (m, 5H), 7.26–7.11 (m, 5H), 4.98–4.79 (m, 2H), 3.20–3.11 (m, 1H), 2.71–2.64 (m, 1H), 1.77–1.69 (m, 1H), 1.46–1.40 (m, 1H), 1.35 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.8, 153.6, 140.8, 138.5, 128.6, 128.5, 127.6, 127.2, 126.5, 83.5, 48.2, 28.4, 28.0, 26.9, 19.4; IR (film): 3035, 2977, 1730, 1677, 1373, 1149 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{22}H_{26}O_3$, 352.1913; found 352.1897; $[\alpha]^{26.5}_D$ 0.00 ° (*c* = 1.00, $CHCl_3$).



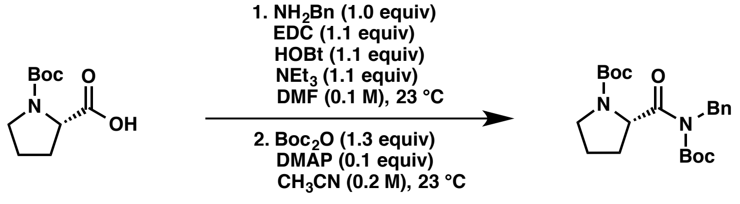
Amide 39: A solution of HNBnBoc (860 mg, 4.2 mmol, 1.0 equiv) in THF (4.2 mL, 1.0 M) was cooled to –78 °C. To this mixture, a solution of LHMDS (1.0 g, 6.2 mmol, 1.5 equiv) in THF (4.2 mL, 1.0 M) was added dropwise dropwise (500 µL/min) and left to stir at –78 °C for 1 h. Acid chloride **SI-16** (0.355 mL, 4.15 mmol, 1.0 equiv) was added dropwise (500 µL/min) and left to stir for 1 h. The reaction was warmed to 0 °C and left to stir for 1 h. The reaction was quenched with saturated aqueous $NaHCO_3$ (10 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 X 15 mL). The organics were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (25:1 Hexanes:EtOAc) to yield **39** (0.98 g, 81% yield) as a white solid. Amide **39**: mp: 48.3–48.8 °C; R_f 0.35 (5:1 Hexanes: EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33–7.26 (m, 4H), 7.26–7.20 (m, 1 H), 4.71 (s, 2H), 1.39 (s, 9H), 1.27 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 185.9, 154.0, 138.5, 128.5, 128.1, 127.4, 82.7, 50.7, 43.5, 28.2, 28.0; IR (film): 2969, 2874, 1733, 1691, 1366, 1148 cm^{-1}; HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{17}H_{25}NO_3$ 292.19127; found 292.19127.



Amide SI-18. To a mixture of carboxylic acid **SI-17** (0.50 g, 1.86 mmol, 1.0 equiv), EDC (0.36 g, 1.86 mmol, 1.0 equiv), HOBt (0.25 g, 1.86 mmol, 1.0 equiv), and CH_2Cl_2 (19 mL, 0.1 M) was added benzylamine (0.20 mL, 1.86 mmol, 1.0 equiv). The resulting mixture was stirred at 23 °C for 16 h, and then diluted with CH_2Cl_2 (30 mL). The mixture was washed with deionized water

(2 X 20 mL). The organics were dried over Na_2SO_4, filtered and then concentrated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (15.9 mg, 0.13 mmol, 0.1 equiv), followed by acetonitrile (6.5 mL, 0.2 M). Boc_2O (0.57 g, 2.6 mmol, 2.0 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 12 h. The mixture was washed with 1.0 M HCl (5 mL) and brine (5 mL). After drying over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (5:1 → 3:1 Hexanes:EtOAc) to yield **SI-18** (0.55 g, 92% yield, over two steps) as a white solid. Amide **SI-18**: mp: 106.6–107.8 °C; R_f 0.29 (5:1 Hexanes: EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.7 (d, J = 8.1, 2H), 7.32–7.28 (m, 3H), 7.25–7.22 (m, 3H), 5.49 (dd, J = 5.8, 3.0, 1H), 4.99 (d, J = 15, 1H), 4.79 (d, J = 14.8, 1H), 3.54–3.49 (m, 1H), 3.38–3.34 (m, 1H), 2.41 (br s, 3H), 2.24–2.15 (m, 1H), 1.99–1.90 (m, 2H), 1.79–1.73 (m, 1H), 1.41 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.4, 153.0, 143.4, 138.1, 135.9, 129.7, 128.5, 127.6, 127.5, 127.3, 83.9, 62.5, 48.6, 47.9, 31.7, 27.9, 24.4, 21.7; IR (film): 2977, 2932, 1732, 1692, 1453, 1369 cm^{-1}; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{24}H_{30}N_2O_5SNa$ 481.1773; found 481.1750; $[\alpha]^{23.5}_D$ –1042.0 ° (c = 1.00, CH_2Cl_2).

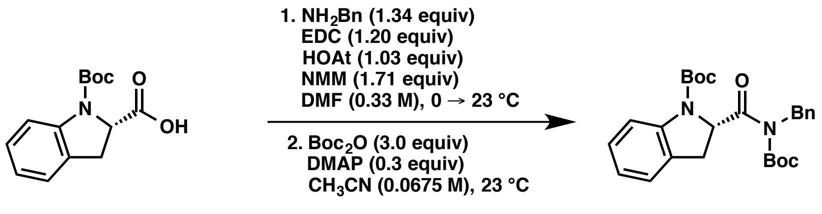


SI-19 (56% yield, 2 steps) *SI-20*

Amide SI-20. To a mixture of carboxylic acid **SI-19** (2.0 g, 9.3 mmol, 1.0 equiv), EDC (1.96 g, 10.23 mmol, 1.1 equiv), HOBt (1.30 g, 10.23 mmol, 1.1 equiv), triethylamine (1.4 mL, 10.23 mmol, 1.1 equiv), and DMF (93 mL, 0.1 M) was added benzylamine (1.12 mL, 10.23 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 16 h, and then diluted with deionized water (50 mL) and EtOAc (30 mL). The mixture was transferred to a separatory funnel with EtOAc (50 mL) and brine (50 mL). The aqueous layer was extracted with EtOAc (3 X 100 mL). The combined organic layers were washed with deionized water (3 X 50 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (113 mg, 0.93 mmol, 0.1 equiv), followed by acetonitrile (46.5 mL, 0.2 M). Boc$_2$O (2.64 g, 12.09 mmol, 1.3 equiv) was added in one portion and the reaction vessel was flushed with N$_2$. The reaction mixture was allowed to stir at 23 °C for 16 h and then quenched by addition of saturated aqueous NaHCO$_3$ (10 mL). The mixture was transferred to a separatory funnel with EtOAc (30 mL) and deionized water (30 mL). The layers were separated. The aqueous layer was extracted with EtOAc (3 X 50 mL). The combined organic layers were dried over Na$_2$SO$_4$, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (10:1 Hexanes:EtOAc) to yield **SI-20** (1.68 g, 56% yield, over two steps) as a white solid. Amide **SI-20**: mp: 81.1–81.8 °C, R*f* 0.34 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.32–7.26 (m, 3H), 7.25–7.18 (m, 2H), 5.31–5.2 (m, 1H), 5.09–4.70 (m, 2H), 3.68–3.55 (m, 1H), 3.53–3.39 (m, 1H), 2.41–2.28 (m, 1H), 2.03–1.80 (m, 3H), 1.50–1.30 (m, 18H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 176.3, 176.1, 154.5, 153.7, 152.9, 152.7, 138.3, 138.0, 128.3, 128.3, 127.8, 127.4, 127.3, 127.3, 126.9, 83.6, 83.4, 79.4, 79.3, 61.2, 60.9, 47.6, 47.6, 47.0, 46.7, 31.3, 30.6, 28.5, 28.3, 27.9, 27.9, 23.7, 23.1; IR (film): 2977, 2973, 1729, 1697, 1393, 1146 cm^{-1}; HRMS-ESI (m/z) HRMS-ESI (m/z) [M+Na]$^+$ calcd for $C_{22}H_{32}N_2O_5Na$ 427.2209; found 427.2195; $[\alpha]^{19.7}_{D}$ +54.0 ° (*c* = 1.00, CH$_2$Cl$_2$).

*Note: Amide **SI-20** was obtained as a mixture of rotamers. These data represent empirically observed chemical shifts and coupling constants from the ^{1}H and ^{13}C NMR spectra.*

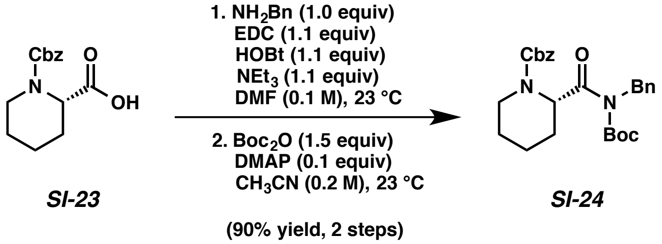


Amide SI-22. To a mixture of carboxylic acid **SI-21** (0.95 g, 3.6 mmol, 1.0 equiv), EDC (0.83 g, 4.32 mmol, 1.20 equiv), HOAt (0.50 g, 3.71 mmol, 1.03 equiv), and DMF (11 mL, 0.33 M) was added benzylamine (0.53 mL, 4.82 mmol, 1.34 equiv) and 4-methylmorpholine (NMM, 0.68 mL, 6.16 mmol, 1.71 equiv) at 0 °C. The resulting mixture was stirred at 0 ° C for 30 min and then at 23 °C for 16 h. The orange mixture was then diluted with deionized water (15 mL) and EtOAc

(30 mL). The layers were separated. The organic layer was washed with 1 M HCl (20 mL), saturated aqueous $NaHCO_3$ (20 mL), and brine (20 mL). The organic layer was dried over Na_2SO_4 and then concentrated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (132 mg, 1.08 mmol, 0.3 equiv), followed by acetonitrile (53 mL, 0.07 M). Boc_2O (2.40 g, 10.8 mmol, 3.0 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 16 h and then quenched by addition of saturated aqueous $NaHCO_3$ (10 mL). The mixture was transferred to a separatory funnel with EtOAc (50 mL) and deionized water (30 mL). The layers were separated. The organic layer was washed with 1 M HCl (30 mL), saturated aqueous $NaHCO_3$ (30 mL), and brine (30 mL). The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (10:1 Hexanes:EtOAc) to yield **SI-22** (1.09 g, 67% yield, over two steps) as a (viscous) colourless oil. Amide **SI-22**: R_f 0.33 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93– 7.52 (m, 1H), 7.30–7.15 (m, 6H), 7.10 (d, *J* = 7.3, 1H), 6.94–6.91 (m, 1H), 5.86–5.79 (m, 1H), 5.04–4.95 (m, 1H), 4.82–4.77 (m 1H), 3.70–3.62 (m, 1H), 3.06 (tt, *J* = 16.7, 3.8, 1H), 1.62–1.47 (m, 9H), 1.41–1.39 (m, 9H), ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.4, 174.2, 152.9, 152.8, 151.7, 143.4, 142.5, 138.2, 137.9, 129.2, 128.5, 128.4, 128.2, 128.18, 127.9, 127.7, 127.6, 127.5, 127.2, 124.8, 124.4, 122.4, 122.3, 114.6, 84.1, 83.7, 82.1, 80.8, 76.9, 62.7, 62.5, 47.7, 47.66, 33.8, 33.2, 28.6, 28.2, 28.0, 27.8; IR (film): 2980, 2935, 1730, 1712, 1608, 1485, 1396, 1363, 1318, 1253, 1213, 1146; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{26}H_{32}N_2O_5Na$ 475.2209; found 475.2209; $[\alpha]^{21.7}_D$ –150.0 ° (c = 1.00, CH_2Cl_2).

*Note: Amide **SI-22** was obtained as a mixture of rotamers. These data represent empirically observed chemical shifts and coupling constants from the ^{1}H and ^{13}C NMR spectra.*



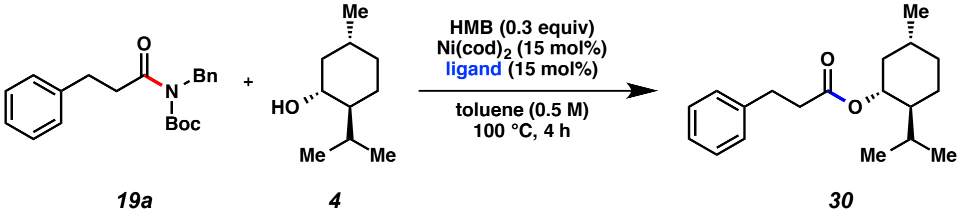
Amide **SI-24**: To a mixture of carboxylic acid **SI-23** (750 mg, 2.85 mmol, 1.0 equiv), EDC (600 mg, 3.13 mmol, 1.1 equiv), HOBt (422 mg, 3.13 mmol, 1.1 equiv), triethylamine (0.43 mL, 3.13

mmol, 1.1 equiv), and DMF (31 mL, 0.1 M) was added benzylamine (0.34 mL, 3.13 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 12 h, and then diluted with deionized water (50 mL) and EtOAc (50 mL). The layers were separated and the organic layer was washed successively with 1.0 M HCl (50 mL), saturated aqueous $NaHCO_3$ (50 mL), and brine (50 mL). The organics were dried over Na_2SO_4 and concentrated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (122 mg, 1.0 mmol, 0.1 equiv) followed by acetonitrile (53 mL, 0.2 M). Boc_2O (3.0 g, 13.78 mmol, 1.5 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 12 h. The reaction mixture was diluted with EtOAc (50 mL) and then washed with saturated aqueous $NaHCO_3$ (2 X 50 mL) and brine (50 mL). The organics were dried over Na_2SO_4 and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (5:1 Benzene:Hexanes → 1:1 Benzene:EtOAc) to yield amide **SI-24** (1.4 g, 90% yield, over two steps) as a off white solid. Amide **SI-24**: R_f 0.35 (5:1 Hexanes:EtOAc); ^1H NMR (500 MHz, $CDCl_3$): δ 7.40–7.26 (m, 5H), 7.26–7.12 (m, 5H), 5.95 (app d, 1H), 5.21–5.05 (m, 2H), 5.0–4.76 (m, 2H), 4.13–3.95 (m, 1H), 3.52–3.33 (m, 1H), 2.09–1.91 (m, 1H), 1.89–1.57 (m, 3H), 1.49–1.29 (m, 10H), 1.28–1.12 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.1, 176.0, 157.1, 156.4, 152.6, 152.6, 138.3, 138.1, 136.9, 136.8, 128.6, 128.50, 128.46, 128.0, 127.9, 127.8, 127.6, 127.5, 127.4, 127.3, 127.2, 83.8, 67.32, 67.26, 55.6, 55.4, 47.83, 47.75, 42.3, 42.2, 28.0, 27.9, 27.22, 27.17, 24.9, 24.7, 19.78, 19.65; IR (film): 2975, 2942, 1732, 1705, 1348, 1146 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{26}H_{33}N_2O_5$, 453.2390; found 453.23710; $[\alpha]^{23.1}_D$ +4.40 ° (c = 1.00, $CHCl_3$).

B. Reaction Discovery

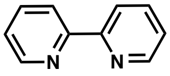
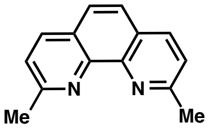
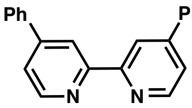
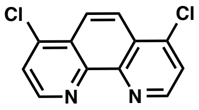
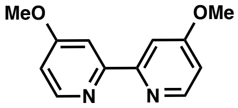
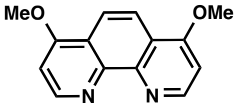
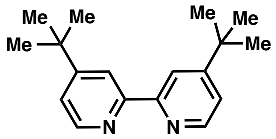
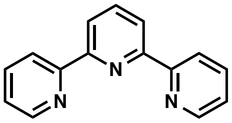
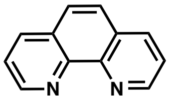
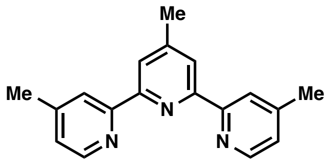
Representative procedure for pyridine-type ligand screening
(coupling of amide ***19a*** *and (–)-menthol (****4****) is used as an example).*



Ester 30. A 1-dram vial containing amide **19a** (50.0 mg, 0.15 mmol, 1.0 equiv), hexamethylbenzene (HMB) (7.3 mg, 0.045 mmol, 0.3 equiv), and a magnetic stir bar was charged with Ni(cod)$_2$ (6.2 mg, 0.0225 mmol, 15 mol%) and ligand (0.0225 mmol, 15 mol%) in a glove box. Subsequently, toluene (0.15 mL, 1.0 M) and then (–)-menthol (**4**) (29.3 mg, 0.19 mmol, 1.2 equiv) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred at 100 °C for 4 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were evaporated under reduced pressure, and the yield was determined by ^{1}H NMR analysis with hexamethylbenzene as an internal standard.

The representative procedure shown above depicts all of the results shown in Tables S1–S3.

Table S1. Evaluation of Pyridine-Type Ligands.[a]

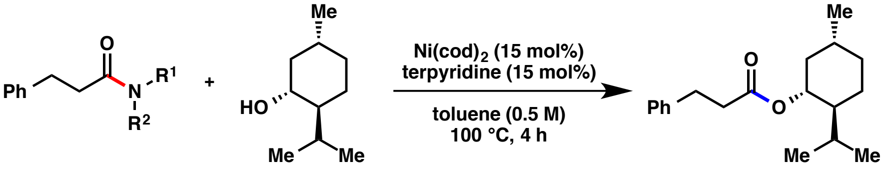
Entry	***Ligand***	***Yield of 30***	***Entry***	***Ligand***	***Yield of 30***
1		0%	6		0%
2		0%	7		0%
3		0%	8		37%
4		23%	9		60%
5		0%	10		17%

[a] Yields were determined using ^{1}H NMR analysis with hexamethylbenzene as an internal standard.

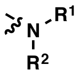
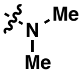
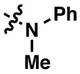
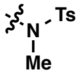
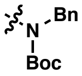
C. Survey of Amide Substrates

Compounds SI-25,[2] SI-26[3] and SI-27[4] were prepared by known literature procedure.

Table S2. Survey of Amide Substrates Under the Optimized Reaction Conditions.[a]



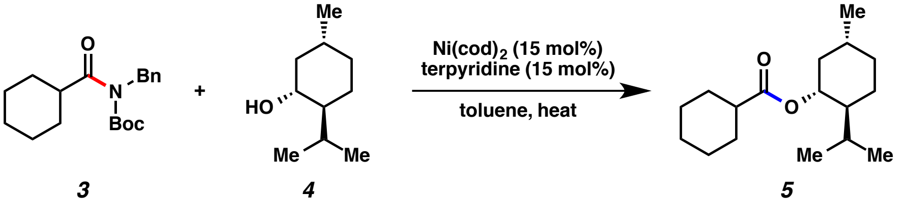
4 **30**

Entry			*Yield of 30*	*Remainder of the mass*
1		***SI-25***	0% yield	***SI-25***; 100%
2		***SI-26***	0% yield	***SI-26;*** 100%
3		***SI-27***	13% yield	***SI-27***; 87%
4		***19a***	60% yield	***19a***; 40%

[a] Yields were determined using ^{1}H NMR analysis with hexamethylbenzene as an internal standard.

D. Optimization of Reaction Conditions and Relevant Control Experiments

Table S3. Optimization of Reaction Conditions.[a]

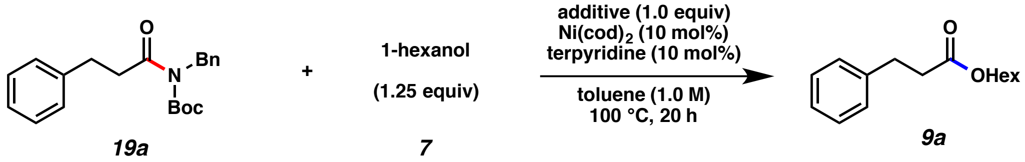


Reaction conditions	Experimental Results	
	5	**3**
Ni(cod)$_2$ (15 mol%), terpyridine (15 mol%), menthol (1.2 equiv), toluene (0.5 M), 80 °C, 20 h	24%	76%
Ni(cod)$_2$ (15 mol%), terpyridine (7.5 mol%), menthol (1.2 equiv), toluene (0.5 M), 80 °C, 20 h	31%	69%
Ni(cod)$_2$ (15 mol%), terpyridine (30 mol%), menthol (1.2 equiv), toluene (0.5 M), 80 °C, 20 h	31%	69%
Ni(cod)$_2$ (15 mol%), terpyridine (15 mol%), menthol (2.5 equiv), toluene (0.5 M), 80 °C, 20 h	30%	70%
Ni(cod)$_2$ (15 mol%), terpyridine (15 mol%), menthol (1.2 equiv), toluene (0.33 M), 80 °C, 20 h	19%	81%
Ni(cod)$_2$ (15 mol%), terpyridine (15 mol%), menthol (1.2 equiv), toluene (1.0 M), 80 °C, 20 h	35%	65%
Ni(cod)$_2$ (15 mol%), terpyridine (15 mol%), menthol (1.2 equiv), toluene (1.0 M), 100 °C, 20 h	56%	44%
Control experiments:		
Ni(cod)$_2$ (15 mol%), menthol (1.2 equiv), toluene (1.0 M), 100 °C, 20 h	0%	100%
Terpyridine (30 mol%), menthol (1.2 equiv), toluene (1.0 M), 100 °C, 20 h	0%	100%
Menthol (1.2 equiv), toluene (1.0 M), 100 °C, 20 h	0%	100%

[a] Yields were determined using ^{1}H NMR analysis with hexamethylbenzene as an internal standard.

E. Evaluation of Functional Group Compatibility in the Esterification Reaction

In order to provide further insight into functional group tolerance for the esterification reaction, a "robustness screen" was performed.[5] The esterification reaction of **19a** and **7** was performed, but doped with 1.0 equiv of various alkyl and aryl halides and nitriles to determine whether or not these functional grous would be compatible in the coupling reaction.

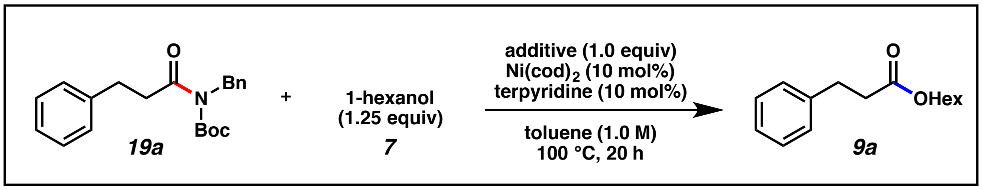


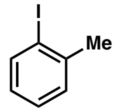
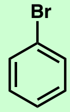
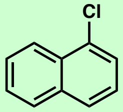
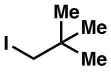
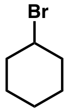
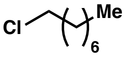
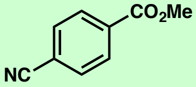
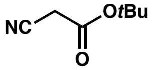
Representative Procedure for esterification reactions in the presence of additives from Table S4.

A 1-dram vial containing amide **19a** (67.9 mg, 0.20 mmol, 1.0 equiv) and a magnetic stir bar was charged with Ni(cod)$_2$ (5.5 mg, 0.020 mmol, 0.10 equiv) and terpyridine (4.7 mg, 0.020 mmol, 0.10 equiv) in a glove box. Subsequently, toluene (0.20 mL, 1.0 M) and then alcohol **7** (31.4 μL, 0.250 mmol, 1.25 equiv) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred at 100 °C for 20 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over 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 1,3,5-trimethoxybenzene as an internal standard. Ester **9a**: R_f 0.52 (20:1 Hexanes:EtOAc). Spectral data match those previously reported.[6]

Any modifications of the conditions shown in the representative procedure above are specified below in Table S4.

Table S4. Evaluation of Functional Group Compatibility in the Esterification Reaction.[a]

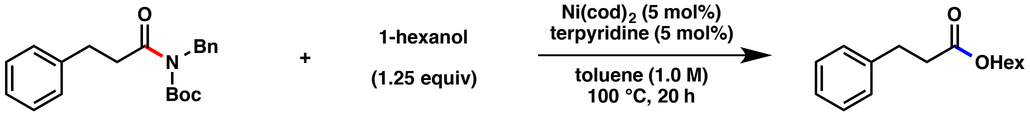


Entry	Additive	Yield of *9a* (%)	Additive Remaining (%)	*19a* Remaining (%)
1	None	90	-	10
2		31	95	69
3		87	61	8
4		90	96	14
5		23	35	74
6		23	75	77
7		45	32	55
8		91	90	8
9		0	100	100

[a] Conditions: Ni(cod)$_2$ (10 mol%), terpyridine (10 mol%), substrate (1.0 equiv), 1-hexanol (1.25 equiv), toluene (1.0 M), and additive (1.0 equiv) at 100 °C for 20 h. Yields of coupled product, remaining additive, and remaining starting material were determined by ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

F. Scope of Methodology

*Representative Procedure (coupling of **19a** and **7** is used as an example).*

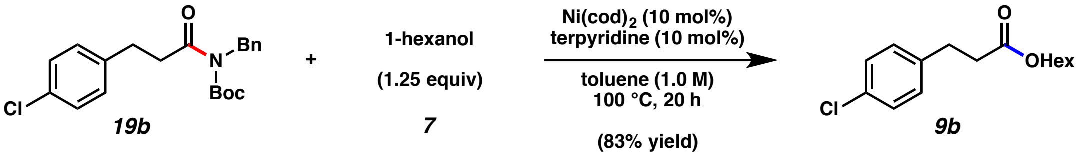


19a **7** (77% yield) **9a**

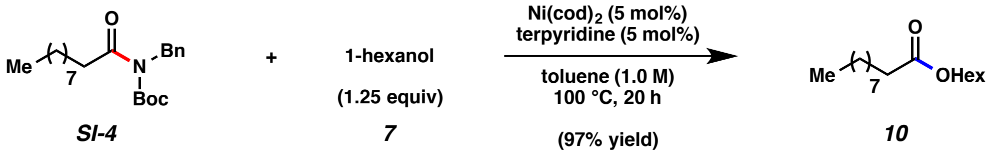
Ester 9a (Figure 2). A 1-dram vial containing amide **19a** (71.8 mg, 0.30 mmol, 1.0 equiv) and a magnetic stir bar was charged with Ni(cod)$_2$ (4.1 mg, 0.015 mmol, 5 mol%) and terpyridine (3.5 mg, 0.015 mmol, 5 mol%) in a glove box. Subsequently, toluene (0.30 mL, 1.0 M) and then alcohol **7** (38.3 μg, 0.38 mmol, 1.25 equiv) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred at 100 °C for 20 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure and the crude residue was purified by flash chromatography (50:1 Hexanes:EtOAc) generated ester **9a** (77% yield, average of two experiments) as a clear oil. Ester **9a**: R$_f$ 0.52 (20:1 Hexanes:EtOAc). Spectral data match those previously reported.[6]

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 Figures 2–4.

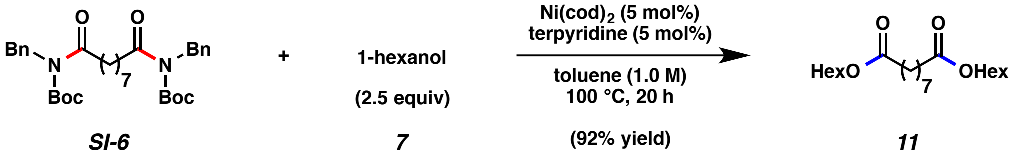
For each of the nickel-catalyzed reactions described herein, control experiments were performed concurrently where Ni(cod)$_2$ and both Ni(cod)$_2$ and terpyridine were omitted from the reactions. In all cases, these control experiments led to the recovery of the amide substrates with no detectable conversion to the corresponding esters.



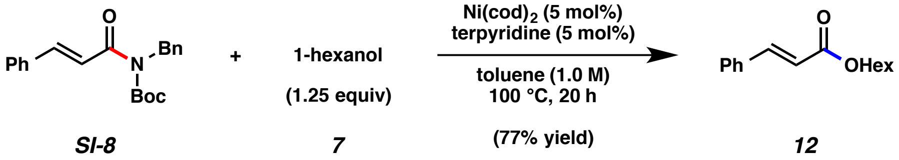
Ester 9b (Figure 2). Purification by preparative thin layer chromatography (5:1 → 9:1 Hexanes:EtOAc) generated ester **9b** (83% yield, yield determined via ^{1}H NMR internal standard 1,3,5–trimethoxybenzene) as a clear oil. Ester **9b**: R$_f$ 0.66 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): 7.32–7.20 (m, 2H), 7.19–7.09 (m, 2H), 4.07 (t, J = 6.7, 2H), 2.93 (app t, 2H), 2.62 (app t), 1.68–1.54 (m, 2H), 1.38–1.23 (m, 6H), 0.96–0.85 (m, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ cm^{-1} 172.9, 139.1, 132.1, 129.8, 128.7, 64.9, 35.9, 31.6,30.5, 28.7, 25.7, 22.7, 14.1; IR (film): 2957, 2930, 2860, 1727, 1493, 1455 cm^{-1}; HRMS-ESI (m/z) [M+H]$^+$ calcd for $C_{15}H_{21}ClO_2$ 269.13083; found 269.12854.



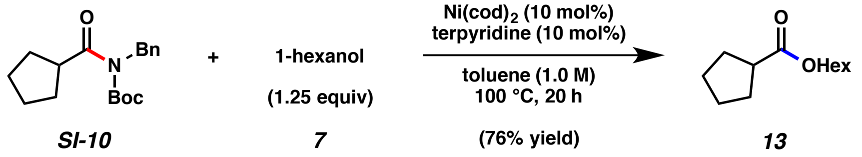
Ester 10 (Figure 2). Purification by flash chromatography (20:1 Hexanes:EtOAc) generated ester **10** (97% yield, average of two experiments) as a clear oil. Ester **10**: R$_f$ 0.47 (20:1 Hexanes:EtOAc). Spectral data match those previously reported.[7]



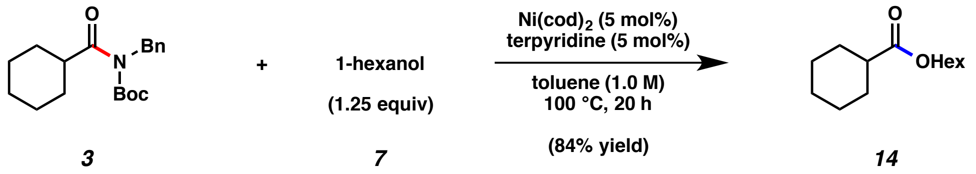
Ester 11 (Figure 2). Purification by preparative thin layer chromatography (20:1 Hexanes:EtOAc) generated ester **11** (92% yield, average of two experiments) as a clear oil. Ester **11**: R$_f$ 0.48 (20:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): 4.06 (t, *J* = 6.7, 4H), 2.29 (t, *J* = 7.6, 4H), 1.62–1.57 (m, 8H), 1.35–1.24 (m, 18H), 0.89 (t, *J* = 6.8, 6H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 174.1, 64.6, 34.5, 31.6, 29.1, 29.05, 28.8, 25.7, 25.1, 22.7, 14.1; IR (film): 2952, 2930, 1730, 1465, 1248, 1176, 1156 cm^{-1}; HRMS-ESI (m/z) [M+Na]$^+$ calcd for $C_{21}H_{40}O_4Na$ 379.2824; found 379.2826.



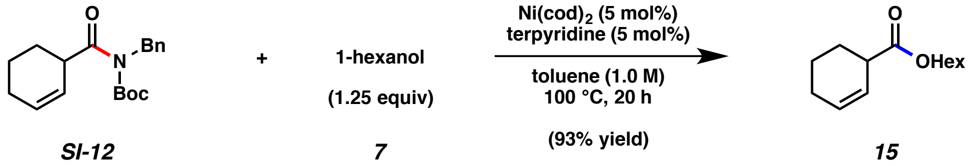
Ester 12 (Figure 2). Purification by flash chromatography (20:1 Hexanes:EtOAc) generated ester **12** (77% yield, average of two experiments) as a clear oil. Ester **12**: R$_f$ 0.58 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[8]



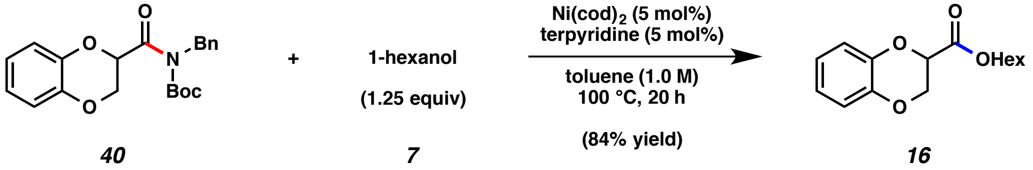
Ester 13 (Figure 2). Purification by flash chromatography (29:1 Hexanes:EtOAc) generated ester **13** (76% yield, average of two experiments) as a clear oil. Ester **13**: R$_f$ 0.41 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 4.05 (t, *J* = 6.7, 2H), 2.71 (quintet, J = 7.9, 8.2, 1H), 1.95–1.83 (m, 2H), 1.83–1.74 (m, 2H), 1.74–1.65 (m, 2H), 1.65–1.51 (m 4H), 1.42–1.22 (m, 6H), 0.95–0.83 (m, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 177.1, 64.6, 44.1, 31.6, 30.2, 28.8, 25.9, 25.7, 22.7, 14.1; IR (film): 2957, 2935, 2870, 1732, 1453 cm^{-1}; HRMS-ESI (*m/z*) [M+NH$_4$]$^+$ calcd for $C_{12}H_{22}O_2NH_4$, 216.1964; found 216.1958.



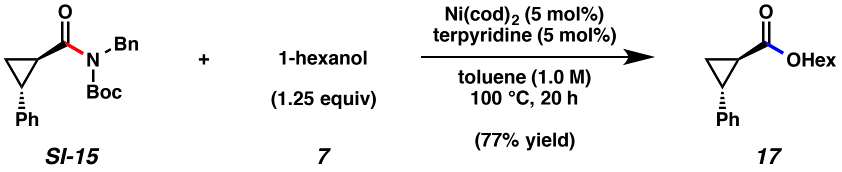
Ester 14 (Figure 2). Purification by flash chromatography (50:1 Hexanes:EtOAc) generated ester **14** (84% yield, average of two experiments) as a clear oil. Ester **14**: R*f* 0.47 (20:1 Hexanes:EtOAc). Spectral data match those previously reported.[9]



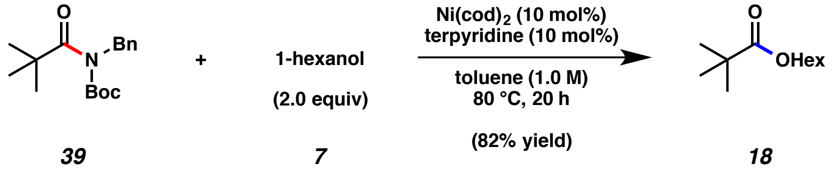
Ester 15 (Figure 2). Purification by flash chromatography (40:1 Hexanes:EtOAc) generated ester **15** (93% yield, average of two experiments) as a clear oil. Ester **15**: R*f* 0.58 (20:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): 5.67 (s, 2H), 4.08 (t, *J* = 6.7, 2H), 2.57–2.51 (m, 1H), 2.25–2.23 (m, 2H), 2.13–1.97 (m, 3H), 1.72–1.58 (m, 3H), 1.36–1.29 (m, 6H), 0.89 (t, *J* = 6.9, 3H) ^{13}C NMR (125 MHz, CDCl$_3$): δ 176.1, 126.8, 125.4, 64.6, 39.5, 31.6, 28.7, 27.6, 25.7, 25.2, 24.6, 22.7, 14.1; IR (film): 3027, 2932, 1730, 1690, 1458, 1151 cm^{-1}; HRMS-ESI (m/z) [M–H]$^{-}$ calcd for $C_{13}H_{21}O_2$ 209.1542; found 209.1535.



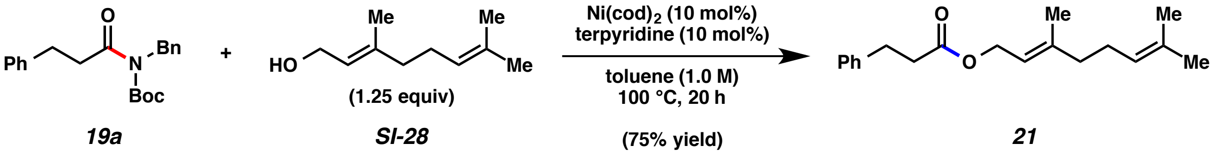
Ester 16 (Figure 2). Purification by flash chromatography (50:1 Hexanes:EtOAc) generated ester **16** (84% yield, average of two experiments) as a clear oil. Ester **16**: R*f* 0.56 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.02–6.97 (m, 1H), 6.91–6.83 (m, 3H), 4.85–4.81 (m, 1H), 4.44–4.33 (m, 2H), 4.26–4.14 (m, 2H), 1.66–1.57 (m, 2H), 1.33–1.21 (m, 6H), 0.91–0.85 (m, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 168.3, 143.1, 142.5, 122.3, 121.9, 117.5, 117.4, 72.2, 66.2, 65.1, 31.4, 28.6, 25.5, 22.6, 14.1; IR (film): 2957, 2927, 2860, 1760, 1735, 1598 cm^{-1}; HRMS-ESI (*m/z*) [M+Na]$^{+}$ calcd for $C_{15}H_{20}O_4Na$, 287.1259; found 287.1248.



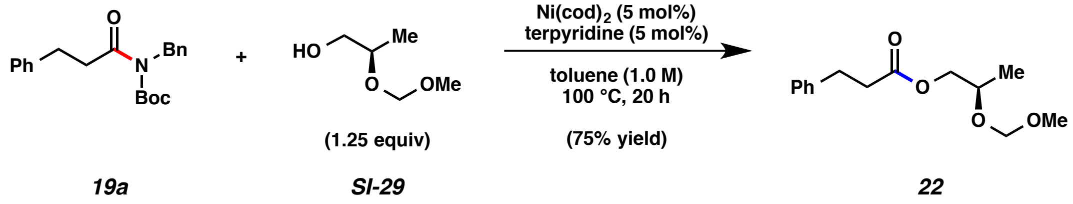
Ester 17 (Figure 2). Purification by preparative thin layer chromatography (2 X 8:1 Hexanes:EtOAc) generated ester **17** (77% yield, average of two experiments) as a clear oil. Ester **17**: R*f* 0.65 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32–7.26 (m, 2H), 7.23–7.18 (m, 1H), 7.13–7.07 (m, 2H), 4.11 (t, *J* = 6.8, 2H), 2.56–2.48 (m, 1H), 1.94–1.87 (m, 1H), 1.68–1.57 (m, 3H), 1.41–1.26 (m, 7H), 0.95–0.86 (app t, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.6, 140.3, 128.6, 126.6, 126.3, 65.1, 31.6, 28.8, 26.3, 25.7, 24.4, 22.7, 17.2, 14.2; IR (film): 2960, 2932, 2860, 1722, 1411, 1173 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{16}H_{21}O_3$, 261.1907; found 261.14917; $[\alpha]^{26.5}_D$ 0.00 ° (*c* = 1.00, $CHCl_3$).



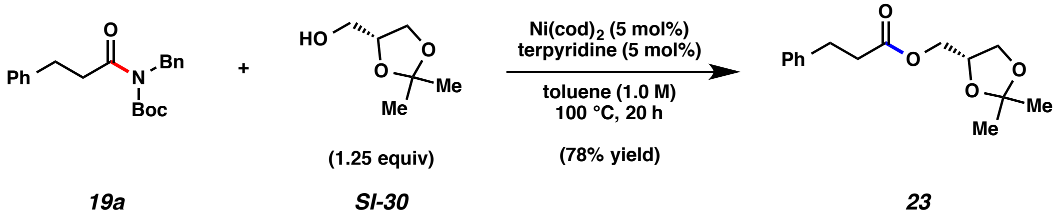
Ester 18 (Figure 2). Ester **18**: R*f* 0.82 (5:1 Hexanes:EtOAc). Spectral data of the crude mixture of ester **18** match those previously reported.[10]



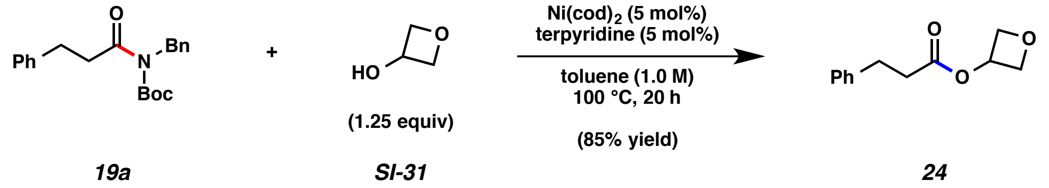
Ester 21 (Figure 3). Purification by flash chromatography (40:1 Hexanes:EtOAc) generated ester **21** (75% yield, average of two experiments) as a clear oil. Ester **21**: R*f* 0.44 (20:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30–7.27 (m, 2 H), 7.29–7.18 (m, 3H), 5.33 (td, *J* = 1.2, 1H), 5.10 (tt, *J* = 6.9, 1.3, 1H), 4.60 (d, *J* = 7.2, 2H), 2.97 (apt t, *J* = 8.2, 7.6, 2H), 2.64 (t, *J* = 8.1, 7.5, 2H), 2.12–2.02 (m, 4H), 1.70–1.69 (m, 6H), 1.60 (br s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.1, 142.4, 140.7, 131.99, 128.6, 128.4, 126.3, 123.9, 118.4, 61.5, 39.7, 36.1, 31.1, 26.4, 25.8, 17.8, 16.6; IR (film): 3068, 3026, 2967, 2857, 1734, 1452, 1379; HRMS-ESI (m/z) $[M–H]^-$ calcd for $C_{19}H_{25}O_2$ 285.1855; found 285.1869.



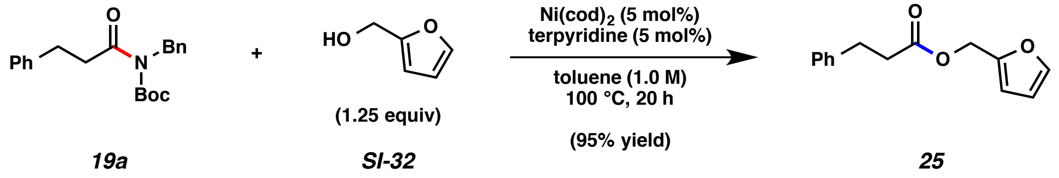
Ester 22 (Figure 3). Purification by flash chromatography (20:1 → 10:1 Hexanes:EtOAc) generated ester **22** (75% yield, average of two experiments) as a clear oil. Ester **22**: R_f 0.34 (10:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32–7.28 (m, 2H), 7.22–7.17 (3H), 4.68 (q, *J* = 6.9, 3.1, 2H), 4.12–4.02 (m, 2H), 3.96–3.86 (m, 1H), 3.36 (br s, 3H), 2.99 (t, *J* = 8.2, 7.4, 2H), 2.69 (apt t, *J* = 8.2, 7.3, 2H), 1.18 (d, *J* = 6.4, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.9, 140.6, 128.7, 128.4, 126.4, 95.3, 55.4, 35.9, 31.0, 17.3; IR (film): 3029, 2893, 2852, 1734, 1454, 1377; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{14}H_{20}O_4Na$ 275.1259; found 275.1263; $[\alpha]^{24.8}_D$ –980.0 ° (*c* = 1.00, CH_2Cl_2).



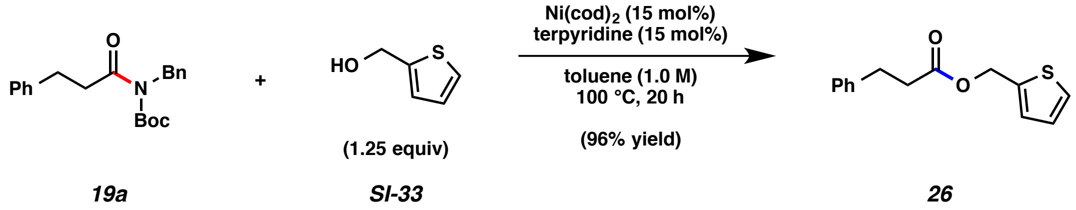
Ester 23 (Figure 3). Purification by flash chromatography (10:1 Hexanes:EtOAc) generated ester **23** (78% yield, average of two experiments) as a clear oil. Ester **23**: R_f 0.45 (10:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30–7.27 (m, 2H), 7.22–7.19 (m, 3H), 4.29–4.24 (m, 1H), 4.17 (dd, *J* = 6.8, 4.8, 1H), 4.10 (dd, *J* = 6.0, 5.5, 1H), 4.04 (dd, *J* = 6.4, 2.0, 1H); 3.69 (dd, *J* = 6.2, 2.3, 1H), 2.98 (t, *J* = 7.8, 2H), 2.69 (apt t, *J* = 8.0, 7.6, 2H), 1.42 (br s, 3H), 1.36 (br s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.8, 140.4, 128.7, 128.4, 126.5, 109.9, 73.7, 66.4, 64.9, 35.8, 31.0, 26.8, 25.5; IR (film): 3030, 2990, 1740, 1603, 1208, 1154; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{15}H_{20}O_4Na$ 287.1259; found 275.1252; $[\alpha]^{25.0}_D$ –990.0 ° (*c* = 1.00, CH_2Cl_2).



Ester 24 (Figure 3). Purification by flash chromatography (20:1 → 10:1 Hexanes:EtOAc) generated ester **24** (85% yield, average of two experiments) as a clear oil. Ester **24**: R$_f$ 0.26 (20:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.31–7.28 (m, 2H), 7.23–7.19 (m, 3H), 5.43–5.40 (m 1H), 4.87–4.84 (m, 2H), 4.59–4.56 (m, 2H), 2.99 (t, *J* =, 2H), 2.70 (apt t, *J* = 8.4, 7.8, 2H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 172.2, 140.1, 128.7, 128.4, 126.5, 77.6, 67.96, 335.6, 30.9; IR (film): 3085, 2882, 1731, 1500, 1455, 1359; HRMS-ESI (m/z) [M+Na]$^+$ calcd for $C_{12}H_{14}O_3Na$ 229.0841; found 229.0842.

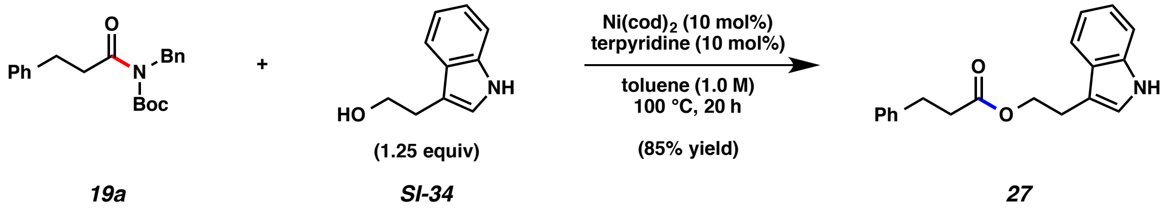


Ester 25 (Figure 3). Purification by flash chromatography (40:1 → 30:1 Hexanes:EtOAc) generated ester **25** (95% yield, average of two experiments) as a clear oil. Ester **25**: R$_f$ 0.45 (10:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.423–7.420 (m. 1H), 7.29–7.26 (2H), 7.21–7.17 (m, 3H), 6.39–6.36 (2H), 5.07 (br s, 2H), 2.97 (t, *J* = 7.7, 2H), 2.68 (t, *J* = 7.7 , 2H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 172.6, 149.6, 143.4, 140.5, 128.6, 128.4, 126.4, 110.8 110.7, 58.2, 35.9, 30.9; IR (film): 3029, 2935, 1733, 1497, 1369, 1145; HRMS-ESI (m/z) [M+Na]$^+$ calcd for $C_{14}H_{14}O_3Na$ 253.0841; found 253.0844.

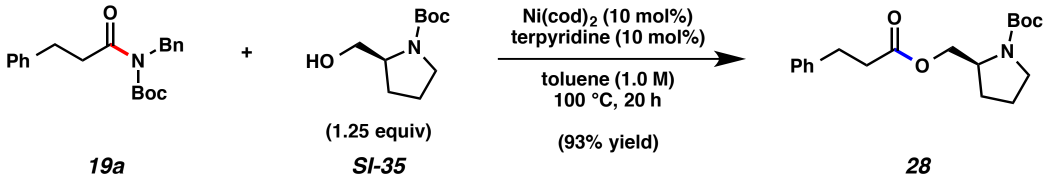


Ester 26 (Figure 3). Purification by flash chromatography (30:1 Hexanes:EtOAc) generated ester **26** (96% yield, average of two experiments) as a clear oil. Ester **26**: R$_f$ 0.65 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.34–7.30 (m, 1H), 7.30–7.25 (m, 2H), 7.22–

7.16 (m, 3H), 7.10–7.06 (m, 1H), 7.01–6.96 (m, 1H), 5.27 (s, 2H), 2.99–2.93 (m, 2H), 2.70–2.63 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.7, 140.5, 138.0, 128.6, 128.4, 128.35, 126.98, 126.96, 126.4, 60.6, 36.0, 31.0; IR (film): 3065, 2955, 1735, 1453, 1231, 1146 cm^{-1}; HRMS-ESI (*m/z*) $[M+NH_4]^+$ calcd for $C_{14}H_{14}O_2SNH_4$, 264.1058; found 264.1053.

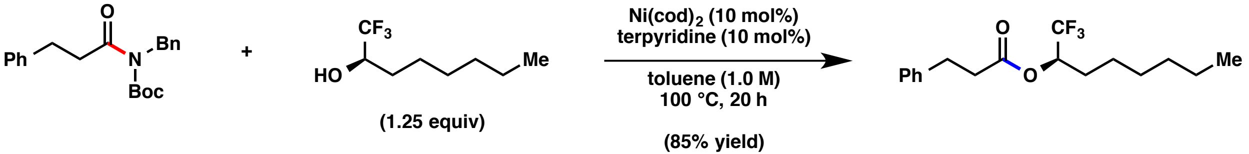


Ester 27 (Figure 3). Purification by flash chromatography (9:1 Hexanes:EtOAc) generated ester **27** (85% yield, average of two experiments) as a brown oil. Ester **27**: R$_f$ 0.18 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.99 (s, 1H), 7.66–7.60 (m, 1H), 7.40–7.34 (m, 1H), 7.31–7.27 (m, 2H) 7.24–7.17 (m, 4H), 7.17–7.12 (m, 1H), 7.00–6.96 (m, 1H), 4.37 (t, *J* = 7.1, 2H), 3.11–3.06 (m, 2H), 2.98–2.92 (m, 2H), 2.67–2.61 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.1, 140.7, 136.3, 128.6, 128.5, 127.6, 126.4, 122.3, 122.1, 119.6, 118.9, 112.2, 111.3, 64.7, 36.1, 31.1, 24.9; IR (film): 3409, 3025, 2920, 1715, 1458, 1161 cm^{-1}; HRMS-ESI (*m/z*) $[M+Na]^+$ calcd for $C_{19}H_{19}NO_2Na$, 316.1313; found 316.1304.



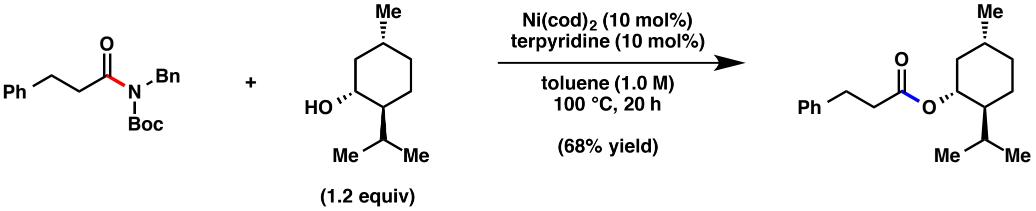
Ester 28 (Figure 3). Purification by flash chromatography (10:1 → 5:1 Hexanes:EtOAc) generated ester **28** (93% yield, average of two experiments) as a clear oil. Ester **28**: R$_f$ 0.17 (20:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30–7.27 (m, 2H), 7.21 (7.18, 3H), 4.17 (dd, *J* = 10.5, 6.9, 1H), 4.06–3.90 (m, 2H), 3.39–3.30 (m, 2H), 2.96 (t, *J* = 7.8, 2H), 2.65 (t, *J* = 7.7, 2H), 1.89–1.65 (m, 5H), (br s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.8, 154.5, 104.5, 128.6, 128.4, 126.4, 79.9, 64.9, 55.6, 46.8, 46.6, 36.0, 31.1, 28.8, 28.6, 27.9, 23.9, 23.1; IR (film): 2974, 1736, 1690, 1454, 1389, 1365, 1248, 1160, 1106, 699; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{19}H_{27}NO_4Na$ 356.1838; found 356.1821; $[\alpha]^{24.7}_D$ –1024.0 ° (*c* = 1.00, CH_2Cl_2).

*Note: Amide **28** was obtained as a mixture of rotamers. These data represent empirically observed chemical shifts and coupling constants from the ^{1}H NMR spectrum.*



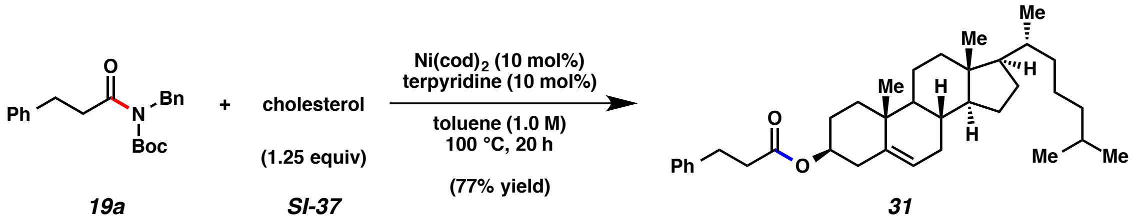
19a ***SI-36*** ***29***

Ester 29 (Figure 3). Purification by flash chromatography (30:1 Hexanes:EtOAc) generated ester **29** (85% yield, average of two experiments) as a clear oil. Ester **29**: R$_f$ 0.70 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.31–7.28 (m, 2H), 7.22–7.20 (m, 3H), 5.33–5.26 (m, 1H), 2.99 (t, J = 7.7, 2H), 2.78–2.68 (m, 2H), 1.76–1.63 (m, 2H), 1.28–1.24 (m, 6H), 0.89 (t, J = 6.8, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 171.6, 140.0, 128.7, 128.4, 126.6, 124.0 (quartet, J = 283.5, 562.4), 69.7 (quartet, J = 31.8, 64.8), 35.5, 31.3, 30.9, 27.86, 27.85, 24.2, 22.4, 14.0; ^{19}F NMR (282 MHz, CDCl$_3$): δ –77.2 (d, J = 6.9); IR (film): 2957, 2867, 1755, 1278, 1176, 752 cm^{-1}; HRMS-ESI (m/z) $[M+NH_4]^+$ calcd for $C_{16}H_{21}F_3O_2NH_4$, 320.1837; found 320.1839; $[\alpha]^{19.8}_{D}$ +8.80 ° (c = 1.00, CHCl$_3$).

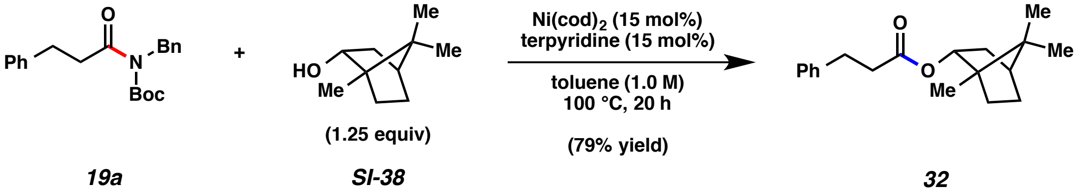


19a ***4*** ***30***

Ester 30 (Figure 3). Purification by flash chromatography (40:1 Hexanes:EtOAc) generated ester **30** (68% yield, average of two experiments) as a clear oil. Ester **30**: R$_f$ 0.40 (20:1 Hexanes:EtOAc). Spectral data match those previously reported.[11]

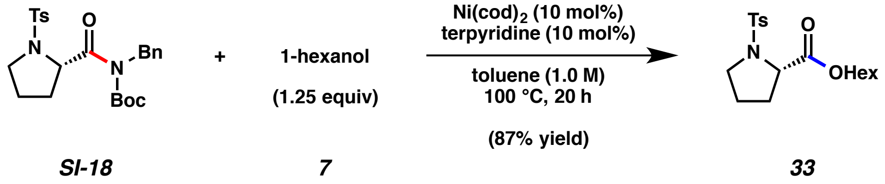


Ester 31 (Figure 3). Purification by flash chromatography (40:1 Hexanes:EtOAc) generated ester **31** (77% yield, average of two experiments) as a white solid. Ester **31**: R*f* 0.40 (20:1 Hexanes:EtOAc). Spectral data match those previously reported.[12]

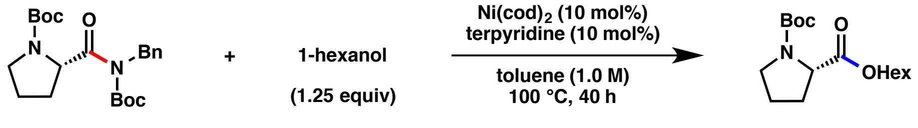


Ester 32 (Figure 3). Purification by flash chromatography (40:1 Hexanes:EtOAc) generated ester **32** (79% yield, average of two experiments) as a clear oil. Ester **32**: R*f* 0.57 (20:1 Hexanes:EtOAc). Spectral data match those previously reported.[13]

G. Coupling with Amino Acid Derivatives



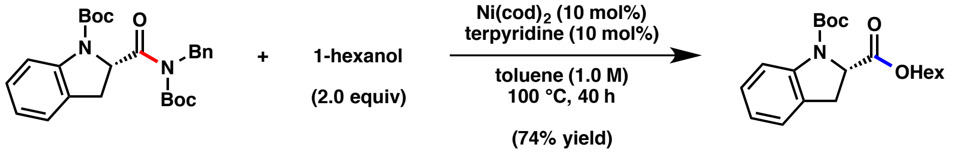
Ester 33 (Figure 4). Purification by flash chromatography (10:1 → 5:1 Hexanes:EtOAc) generated ester **33** (87% yield, average of two experiments) as a clear oil. Ester **33**: R*f* 0.29 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.76 (d, *J* = 8.1, 2H), 7.31 (d, *J* = 8.1, 2H), 4.31–4.28 (m, 1H), 4.13–4.04 (m, 2H), 3.49–3.45 (m, 1H), 3.34–3.29 (m, 1H), 2.42 (s, 3H), 2.06–1.91 (m, 3H), 1.79–1.73 (m, 1H), 1.64–1.58 (m, 2H), 1.35–1.30 (m, 6H), 0.90 (apt t, *J* = 7.0, 5.6, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 172.3, 143.6, 135.6, 129.7, 127.6, 65.6, 60.6, 48.5, 31.5, 31.1, 28.6, 25.6, 24.8, 22.6, 21.7, 14.1; IR (film): 2957, 2875, 1749, 1598, 1455, 1094 cm^{-1}; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{18}H_{27}NO_4SNa$ 376.1559; found 376.1563; $[\alpha]^{23.1}_{D}$ –72.80 ° (*c* = 1.00, CHCl$_3$).



SI-20 **7** ***34***

Ester 34 (Figure 4). Purification via flash chromatography (30:1 → 20:1 → 15:1 Hexanes:EtOAc) yielded ester **34** (74% yield, average of two experiments) as a clear liquid**.** Ester **34**: R_f 0.4 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.38–4.19 (m, 1H), 4.19–4.03 (m, 2H), 3.61–3.31 (m, 2H), 2.30–2.11 (m, 1H), 2.02–1.80 (m, 3H), 1.69–1.58 (m, 2H), 1.50–1.39 (9H), 1.38–1.25 (m, 6H), 0.93–0.83 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.5, 173.2, 171.3, 170.8, 169.3, 154.5, 154.0, 147.6, 146.8, 128.8, 127.6, 79.9, 67.5, 66.0, 65.9, 65.2, 62.6, 60.9, 59.3, 59.2, 59.0, 50.6, 49.2, 46.7, 46.4, 46.3, 31.5, 31.5, 31.4, 41.1, 30.4, 30.1, 29.8, 28.7, 28.6, 28.5, 25.7, 25.6, 25.5, 24.4, 24.0, 23.7, 23.6, 22.6, 14.1; IR (film): 2962, 2934, 2872, 2860, 1742, 1699, 1460, 1393, 1363, 1161, 1123; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{16}H_{29}NO_4Na$ 322.1993; found 322.1985; $[\alpha]^{27.4}_{D}$ –43.20 ° (c = 1.00, $CHCl_3$).

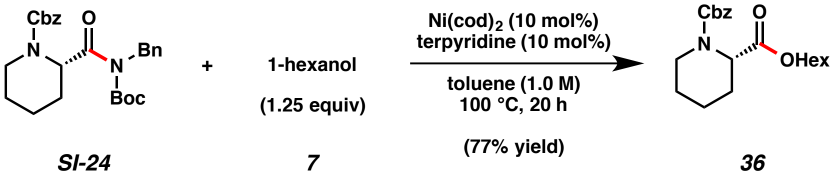
*Note: Ester **34** was obtained as a mixture of rotamers. These data represent empirically observed chemical shifts and coupling constants from the ^{1}H and ^{13}C NMR spectra.*



SI-22 **7** ***35***

Ester 35 (Figure 4). Purification via flash chromatography (30:1 Hexanes:EtOAc) yielded ester **35** (74% yield, average of two experiments) as a clear liquid**.** Ester **35**: R_f 0.33 (20:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.90–7.48 (m, 1H), 7.20–7.17 (m, 1H), 7.10 (d, J = 7.2, 1H), 6.95 (t, J = 7.2, 1H), 4.91–4.82 (m, 1H), 4.13 (br s, 2H), 3.53 (apt t, J = 15.5, 12.0, 1H), 3.11 (dd, J = 16.6, 4.1, 1H), 1.63–1.49 (m, 11H), 1.32–1.27 (m, 6H), 0.88 (t, J = 6.9, 6.5, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.1, 152.7, 151.7, 142.7, 141.8, 128.9, 124.8, 124.4, 114.7, 82.3, 81.3, 60.5, 60.3, 32.1, 31.4, 28.6, 28.3, 25.5, 22.6, 14.0; IR (film): 2955, 2930, 2865, 1750, 1710, 1483, 1383, 1273, 1253, 1164, 1146, 1044, 1019; HRMS-ESI (m/z) $[M+Na]^+$ calcd for $C_{20}H_{29}NO_4Na$ 370.1994; found 370.1988; $[\alpha]^{21.5}_{D}$ –34.0 ° (c = 1.00, CH_2Cl_2).

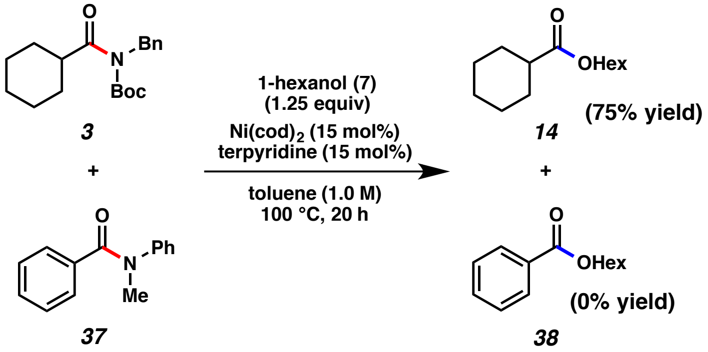
*Note: Ester **35** was obtained as a mixture of rotamers. These data represent empirically observed chemical shifts and coupling constants from the ^{1}H and ^{13}C NMR spectra*



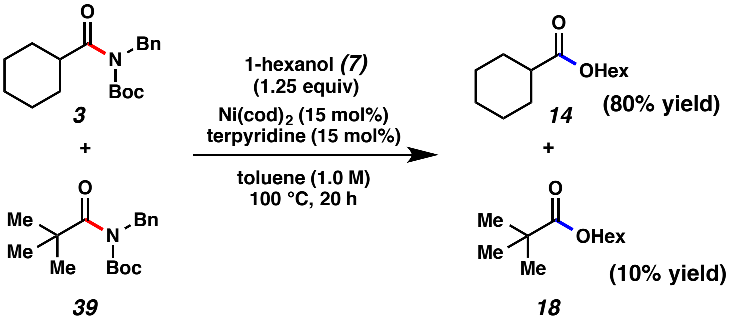
Ester 36 (Figure 4). Purification via flash chromatography (3 X 10:1 Hexanes:EtOAc) yielded ester **36** (77% yield, average of two experiments) as a clear liquid. Ester **36**: R$_f$ 0.47 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.41–7.27 (m, 5H), 5.20–5.07 (m, 2H), 4.98–4.78 (m, 1H), 4.19–3.99 (m, 3H), 3.13–2.88 (m, 1H), 2.31–2.15 (m, 1H), 1.78–1.60 (m, 4H), 1.59–1.36 (m, 2H), 1.36–1.17 (m, 7H), 0.94–0.82 (app t, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 171.9, 171.8, 156.7, 156.1, 136.9, 128.6, 128.1, 127.9, 67.4, 67.3, 65.4, 54.8, 54.6, 42.0, 42.0, 31.5, 28.7, 28.1, 27.0, 26.9, 25.7, 24.9, 24.7, 22.6, 20.9, 20.88, 20.76, 14.1; IR (film): 2932, 2857, 1740, 1702, 1416 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{20}H_{28}NO_5$, 362.19675; found 362.19515; $[\alpha]^{25.3}_{D}$ –36.30 ° (*c* = 1.00, CHCl$_3$).

*Note: Ester **36** was obtained as a mixture of rotamers. These data represent empirically observed chemical shifts and coupling constants from the ^{1}H and ^{13}C NMR spectra.*

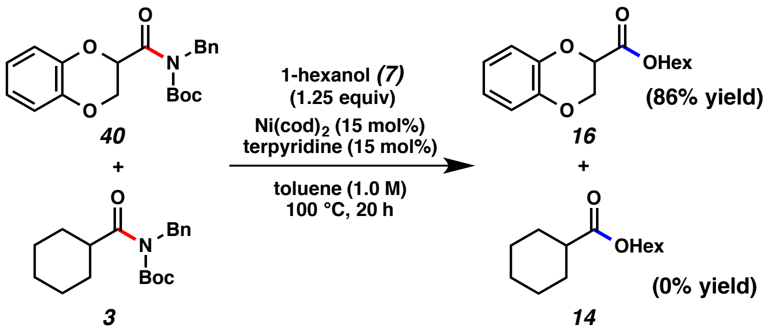
H. Competition Experiments



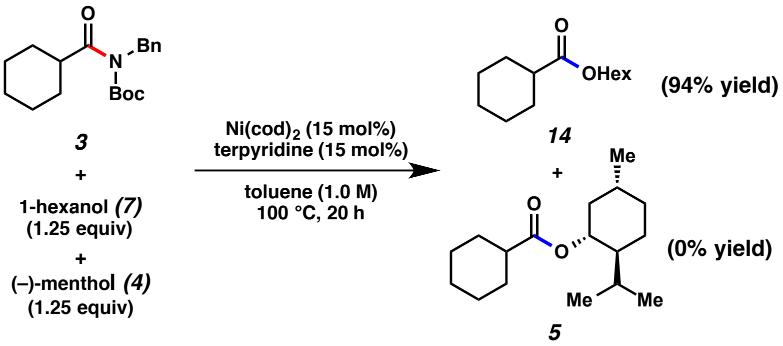
Aliphatic vs. Aromatic Substrate (Figure 5). A 1-dram vial containing amide **3** (47.6 mg, 0.150 mmol, 1.0 equiv), **37**[14] (31.7 mg, 0.150 mmol, 1.0 equiv), 1,3,5–trimethoxybenzene (7.6 mg, 0.045 mmol, 0.3 equiv) and a magnetic stir bar was charged with Ni(cod)$_2$ (6.2 mg, 0.023 mmol, 15 mol%) and terpyridine (5.4 mg, 0.023 mmol, 15 mol%) in a glove box. Subsequently, toluene (0.150 mL, 1.0 M) and then alcohol **7** (23.4 µL, 0.188 mmol, 1.25 equiv) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred at 100 °C for 20 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure. Spectral data match those previously reported for ester **14** (see page 25).



Tertiary vs. Quaternary α Carbon (Figure 5). A 1-dram vial containing amide **3** (47.6 mg, 0.150 mmol, 1.0 equiv), **39** (43.7 mg, 0.150 mmol, 1.0 equiv), 1,3,5–trimethoxybenzene (7.6 mg, 0.045 mmol, 0.3 equiv) and a magnetic stir bar was charged with Ni(cod)$_2$ (6.2 mg, 0.023 mmol, 15 mol%) and terpyridine (5.4 mg, 0.023 mmol, 15 mol%) in a glove box. Subsequently, toluene (0.150 mL, 1.0 M) and then alcohol **7** (23.4 µL, 0.188 mmol, 1.25 equiv) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred at 100 °C for 20 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure. Spectral data match those previously reported for ester **14** (see page 25) and ester **18** (see page 26).

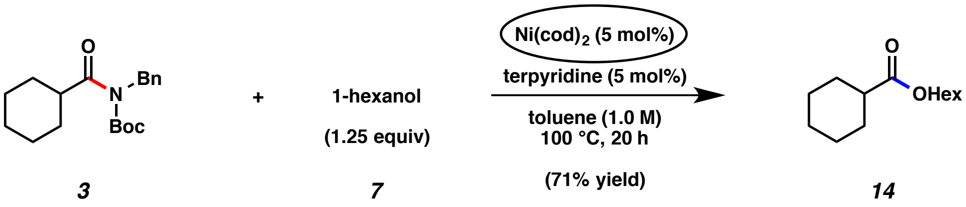


Tertiary vs. Tertiary with Heteroatom (Figure 5). A 1-dram vial containing amide **40** (55.4 mg, 0.150 mmol, 1.0 equiv), **3** (47.6 mg, 0.150 mmol, 1.0 equiv), 1,3,5–trimethoxybenzene (7.6 mg, 0.045 mmol, 0.3 equiv) and a magnetic stir bar was charged with Ni(cod)$_2$ (6.2 mg, 0.023 mmol, 15 mol%) and terpyridine (5.4 mg, 0.023 mmol, 15 mol%) in a glove box. Subsequently, toluene (0.150 mL, 1.0 M) and then alcohol **7** (23.4 µL, 0.188 mmol, 1.25 equiv) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred at 100 °C for 20 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure. Spectral data match those previously reported for ester **16** (see page 25).



Primary vs. Secondary Alcohol (Figure 5). A 1-dram vial containing amide **3** (63.5 mg, 0.200 mmol, 1.0 equiv), (–)-menthol **4** (39.1 mg, 0.250 mmol, 1.25 equiv), 1,3,5–trimethoxybenzene (10.1 mg, 0.060 mmol, 0.3 equiv) and a magnetic stir bar was charged with $Ni(cod)_2$ (8.3 mg, 0.030 mmol, 15 mol%) and terpyridine (7.0 mg, 0.030 mmol, 15 mol%) in a glove box. Subsequently, toluene (0.200 mL, 1.0 M) and then alcohol **7** (31.4 µL, 0.250 mmol, 1.25 equiv) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred at 100 °C for 20 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure. Spectral data match those previously reported for ester **14** (see page 25).

I. Protocol for Benchtop Delivery of $Ni(cod)_2$

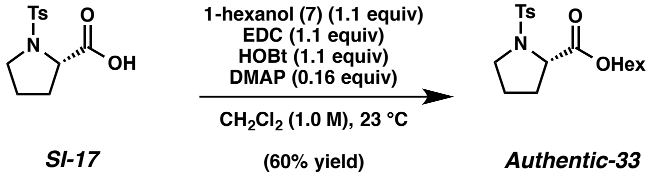


Ester 14. A 1-dram vial containing a magnetic stir bar was flame-dried under reduced pressure, and then allowed to cool under N_2. The vial was charged with amide substrate **3** (317 mg, 1.0 mmol, 1.0 equiv), 1-hexanol (**7**, 0.157 mL, 1.25 mmol, 1.25 equiv),

terpyridine (11.7 mg, 0.05 mmol, 0.05 equiv), and a paraffin capsule containing Ni(cod)$_2$ (13.8 mg, 0.05 mmol, 0.05 equiv). The vial was flushed with N$_2$, and subsequently toluene (1.0 mL, 1.0 M) that had been sparged with N$_2$ for 20 min was added. The vial was capped with a Teflon-line screw cap under a flow of N$_2$ and the reaction mixture was stirred at 100 °C for 20 h. After removing the vial from heat, the reaction mixture was transferred to a 100 mL round bottom flask containing 3.0 g of silica gel with hexanes (10.0 mL) and CH$_2$Cl$_2$ (10.0 mL). The mixture was adsorbed onto the silica gel under reduced pressure and filtered over a plug of silica gel (100 mL of hexanes eluent to remove paraffin, then 100 mL 4:1 Hexanes:EtOAc eluent). The volatiles were removed under reduced pressure, and the crude residue was purified by flash chromatography (49:1 Hexanes:EtOAc) to yield ester **14** (71% yield) as a clear oil. Spectral data matched those previously reported.[9]

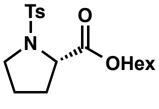
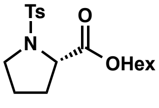
Verification of Enantiopurity

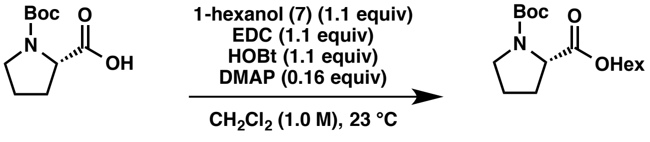
A. Optical Rotation Analysis
I. Synthesis of Optically Pure Ester



Ester Authentic-33: The esterification of **Authentic-33** was carried out following the protocol for achieving the esterification without enantiomeric excess loss.[15] To a mixture of carboxylic acid **SI-17** (100 mg, 0.371 mmol, 1.0 equiv), EDC (78 mg, 0.408 mmol, 1.1 equiv), HOBt (55 mg, 0.408 mmol, 1.1 equiv), DMAP (7 mg, 0.060 mmol, 0.16 equiv), and CH$_2$Cl$_2$ (0.37 mL, 1.0 M) was added 1-hexanol (**7**) (52 μL, 0.408 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 16 h, and then diluted with deionized water (4 mL) and EtOAc (5 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 X 5 mL). The organics were dried over Na$_2$SO$_4$ and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (15:1 → 5:1 Hexanes:EtOAc) to yield ester **Authentic-33** (82 mg, 60% yield) as a clear oil. Spectral data matched those reported in the Experimental Procedure (page S31). $[\alpha]^{23.1}_{D}$ –72.80 ° (c = 1.00, CHCl$_3$).

Table S5: Optical Rotation Data for **33**.

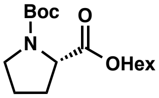
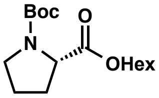
Compound	Optical Rotation	% Optical Purity
 ***Authentic-33***	–72.80 °	X
 ***33***	–73.00 °	>99% ee



SI-19 ***Authentic-34***

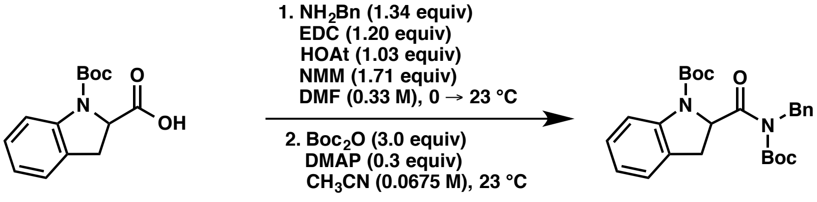
Ester Authentic-34: The esterification of **Authentic-34** was carried out following the protocol for achieving the esterification without enantiomeric excess loss.[15] To a mixture of carboxylic acid **SI-19** (300 mg, 1.39 mmol, 1.0 equiv), EDC (294 mg, 1.53 mmol, 1.1 equiv), HOBt (207 mg, 1.53 mmol, 1.1 equiv), DMAP (19 mg, 0.153 mmol, 0.16 equiv), and CH_2Cl_2 (1.39 mL, 1.0 M) was added 1-hexanol (**7**) (209 µL, 1.67 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 16 h, and then diluted with deionized water (8 mL) and EtOAc (10 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 X 10 mL). The organics were dried over Na_2SO_4 and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (15:1 Hexanes:EtOAc) to yield ester **Authentic-34** (82 mg, 60% yield) as a clear oil. Spectral data matched those reported in the Experimental Procedure (page S32). $[\alpha]^{27.4}_{D}$ –43.20 ° (c = 1.00, $CHCl_3$).

Table S6: Optical Rotation Data for **34**.

Compound	Optical Rotation	% Optical Purity
 ***Authentic-34***	–43.20 °	X
 ***34***	–41.20 °	95% ee

B. Chiral SFC/HPLC Assays

I. Syntheses of Racemic Compounds



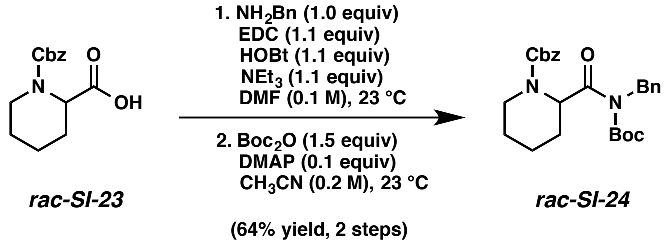
rac-SI-21

(94% yield, 2 steps)

rac-SI-22

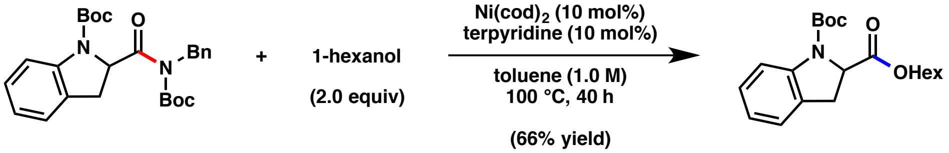
Amide rac-SI-22 (Figure 4). rac-SI-22 was prepared using the procedure described earlier to synthesize (–)-**SI-21** (see page S14), but using racemic carboxylic acid **rac-SI-21**. To a mixture of carboxylic acid **rac-SI-21** (0.95 g, 3.6 mmol, 1.0 equiv), EDC (0.83 g, 4.32 mmol, 1.20 equiv), HOBt (0.50 g, 3.71 mmol, 1.03 equiv), and DMF (11 mL, 0.33 M) was added benzylamine (0.53 mL, 4.82 mmol, 1.34 equiv) and 4-methylmorpholine (NMM, 0.68 mL, 6.16 mmol, 1.71 equiv) at 0 °C. The resulting mixture was stirred at 0 °C for 30 min and then at 23 °C for 16 h. The orange mixture was then diluted with deionized water (15 mL) and EtOAc (30 mL). The layers were separated. The organic layer was washed with 1 M HCl (20 mL), saturated aqueous $NaHCO_3$ (20 mL), and brine (20 mL). The organic layer was dried over Na_2SO_4 and then concentrated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (132.0 mg, 1.08 mmol, 0.3 equiv), followed by acetonitrile (53 mL, 0.0675 M). Boc_2O (2.40 g, 10.8 mmol, 3.0 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 16 h and then quenched by addition of saturated aqueous $NaHCO_3$ (10 mL). The mixture was transferred to a separatory funnel with EtOAc (50 mL) and deionized water (30 mL). The layers were separated. The organic layer was washed with 1 M HCl (30 mL), saturated aqueous $NaHCO_3$ (30 mL), and brine (30 mL). The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (10:1 Hexanes:EtOAc) to yield **rac-SI-22** (1.53 g, 94% yield, over two steps) as a white solid. Spectral data matched those reported in the Experimental Procedure (page S14).



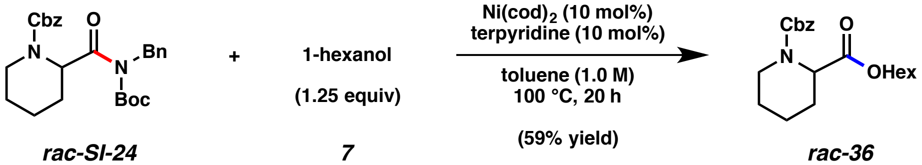
Amide rac-SI-24 (Figure 4). rac-SI-24 was prepared using the procedure described earlier to synthesize (–)-**SI-23** (see page S15), but using racemic carboxylic acid **rac-SI-51**. To a mixture of carboxylic acid **rac-SI-23** (1.0 g, 3.8 mmol, 1.0 equiv), EDC (0.80 g, 4.18 mmol, 1.1 equiv), HOBt (0.56 g, 4.18 mmol, 1.1 equiv), and DMF (38 mL, 0.1 M) was added benzylamine (0.46 mL, 4.18 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 16 h, and then diluted with deionized water (50 mL) and EtOAc (50 mL). The layers were separated and the organic layer was washed successively with 1.0 M HCl (50 mL), saturated aqueous $NaHCO_3$ (50 mL), and brine (50 mL). The organics were dried over Na_2SO_4 and concentrated under reduced pressure. The resulting crude solid material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (46 mg, 0.38 mmol, 0.1 equiv) followed by acetonitrile (38 mL, 0.1 M). Boc_2O (1.1 g, 4.94 mmol, 1.53 equiv) was added in one portion and the reaction vessel was flushed with N_2. The reaction mixture was allowed to stir at 23 °C for 16 h. The reaction mixture was diluted with EtOAc (50 mL) and then washed with saturated aqueous $NaHCO_3$ (2 X 50 mL) and brine (50 mL). The organics were dried over Na_2SO_4 and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (19:1 → 15:1 → 8:1 Hexanes:EtOAc) to yield amide **rac-SI-24** (1.1 g, 64% yield, over two steps) as a off white solid. Spectral data matched those reported in the Experimental Procedure (page S15).



rac-SI-22 ***7*** ***rac-35***

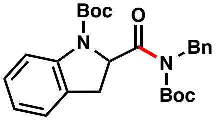
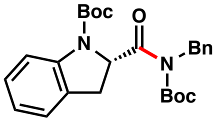
Ester rac-35. A 1-dram vial containing amide **rac-SI-22** (136.0 mg, 0.30 mmol, 1.0 equiv) and a magnetic stir bar was charged with Ni(cod)$_2$ (8.25 mg, 0.030 mmol, 10 mol%) and terpyridine (6.99 mg, 0.030 mmol, 10 mol%) in a glove box. Subsequently, toluene (0.30 mL, 1.0 M) and then 1-hexanol (75.4 μL, 0.60 mmol, 2.0 equiv) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred at 100 °C for 40 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure and the crude residue was purified by flash chromatography (30:1 Hexanes:EtOAc) generated ester **rac-35** (66% yield) as a clear oil.

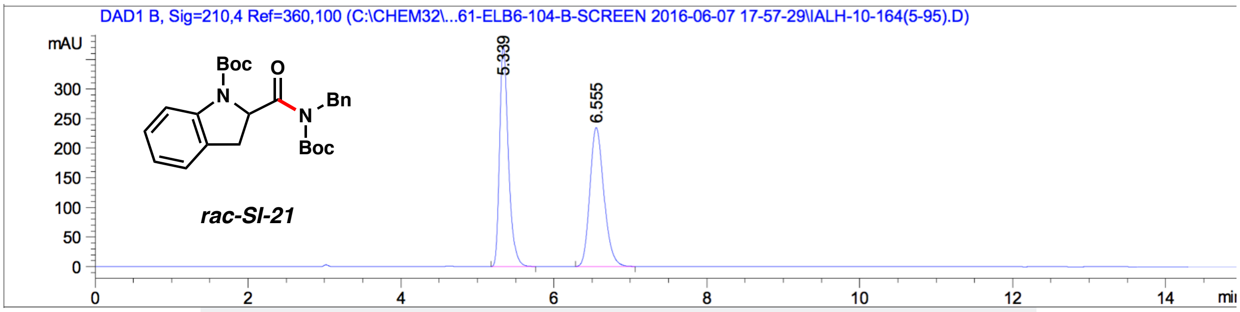


rac-SI-24 ***7*** ***rac-36***

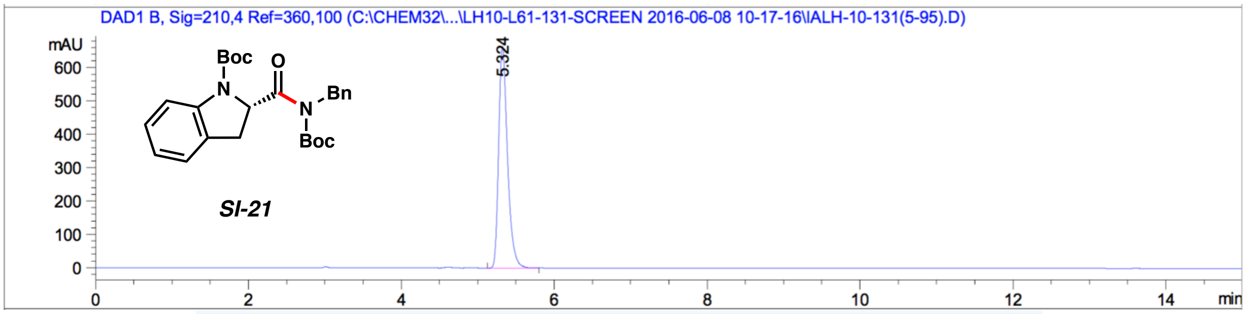
Ester rac-36. A 1-dram vial containing amide **rac-SI-24** (90.5 mg, 0.20 mmol, 1.0 equiv) and a magnetic stir bar was charged with Ni(cod)$_2$ (5.5 mg, 0.020 mmol, 10 mol%) and terpyridine (4.7 mg, 0.020 mmol, 10 mol%) in a glove box. Subsequently, toluene (0.20 mL, 1.0 M) and then 1-hexanol (31.4 μL, 0.25 mmol, 1.25 equiv) were added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred at 100 °C for 20 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure and the crude residue was purified by preparative thin layer chromatography (8:1 Hexanes:EtOAc) generated ester **rac-36** (59% yield) as a clear oil.

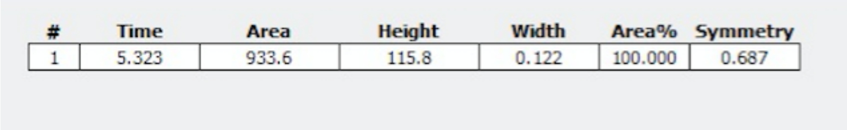
II. Chiral Assays

Compound	Method Column/Temp.	Solvent	Method Flow Rate	Retention Times (min)	Enantiomeric Ratio (er)
 ***rac-SI-21***	Daicel ChiralPak IA-H/23 °C	5% isopropanol in hexanes	1 mL/min	5.34/6.55	50:50
 ***rac-35***	Daicel ChiralPak IA-H/23 °C	5% isopropanol in hexanes	1 mL/min	5.32	100:0

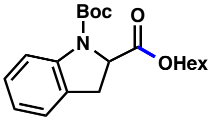
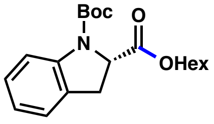


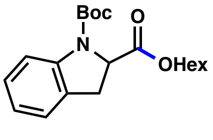
#	Time	Area	Height	Width	Area%	Symmetry
1	5.339	520.3	64.8	0.1217	50.013	0.69
2	6.555	520.1	40.7	0.1973	49.987	0.797



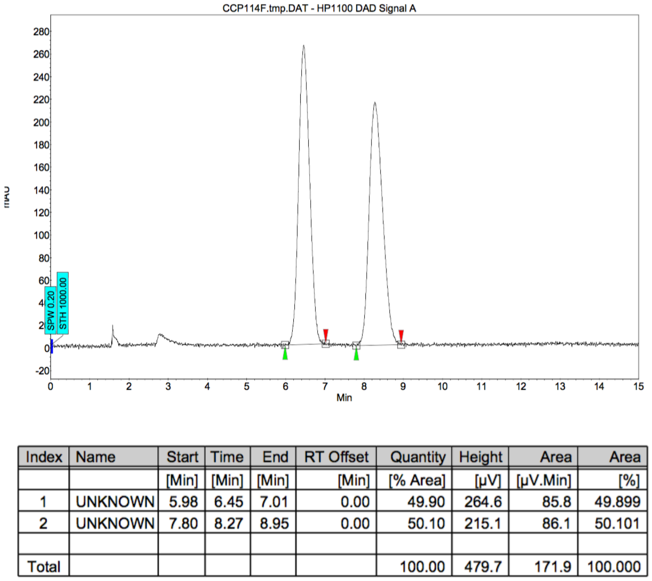


#	Time	Area	Height	Width	Area%	Symmetry
1	5.323	933.6	115.8	0.122	100.000	0.687

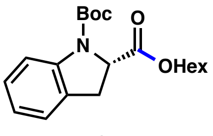
Compound	Method Column/Temp.	Solvent	Method Flow Rate	Retention Times	Enantiomeric Ratio (er)
 ***rac*-35**	Daicel ChiralPak OD-H/35 °C	5% MeCN in CO_2	2 mL/min	6.45/8.27	50:50
 ***36***	Daicel ChiralPak OD-H/35 °C	5% MeCN in CO_2	2 mL/min	6.96/8.98	98.2:1.8



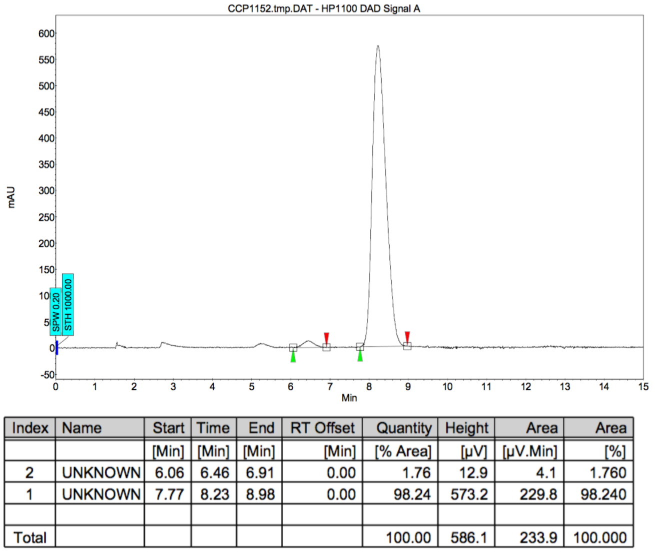
***rac*-35**



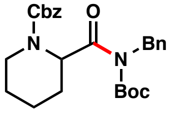
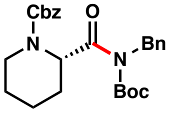
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.98	6.45	7.01	0.00	49.90	264.6	85.8	49.899
2	UNKNOWN	7.80	8.27	8.95	0.00	50.10	215.1	86.1	50.101
Total						100.00	479.7	171.9	100.000

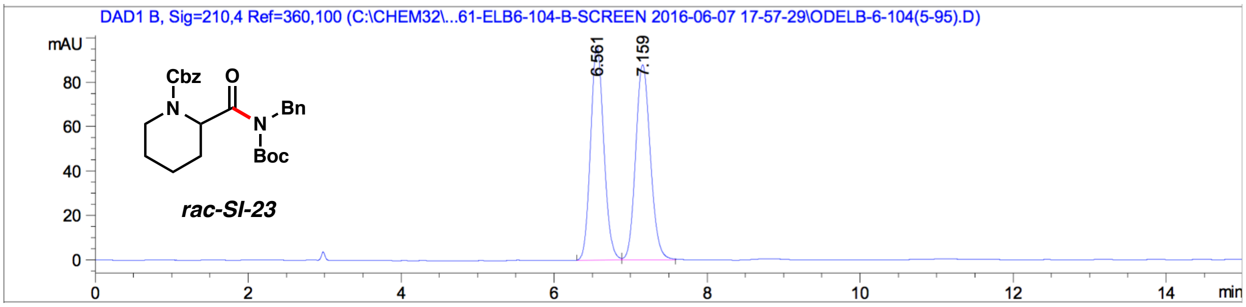


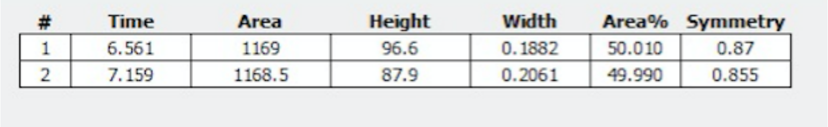
35



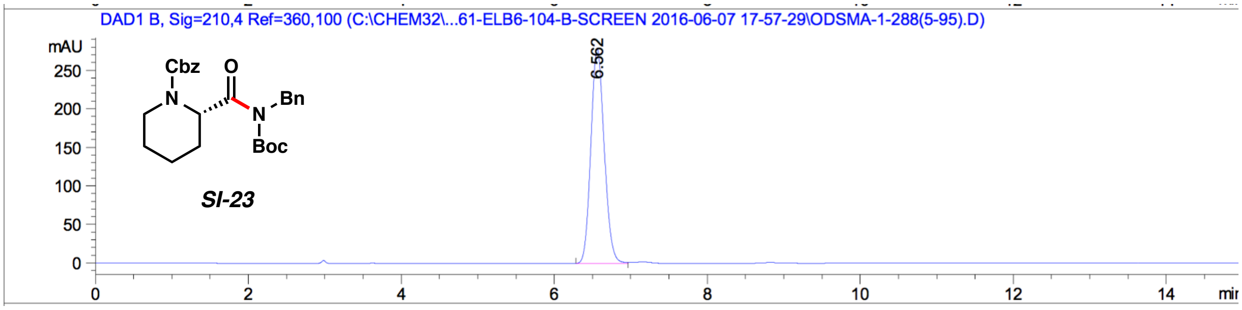
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
2	UNKNOWN	6.06	6.46	6.91	0.00	1.76	12.9	4.1	1.760
1	UNKNOWN	7.77	8.23	8.98	0.00	98.24	573.2	229.8	98.240
Total						100.00	586.1	233.9	100.000

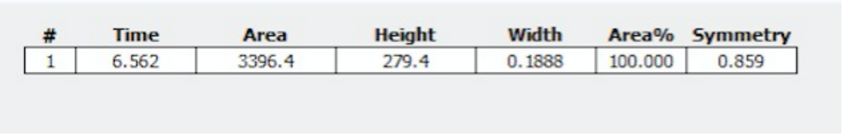
Compound	Method Column/Temp.	Solvent	Method Flow Rate	Retention Times	Enantiomeric Ratio (er)
 ***rac-SI-23***	Daicel ChiralPak OD-H/23 °C	5% isopropanol in hexanes	1 mL/min	6.56/7.16	50:50
 ***SI-23***	Daicel ChiralPak OD-H/23 °C	5% isopropanol in hexanes	1 mL/min	6.56	100:0



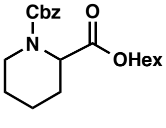
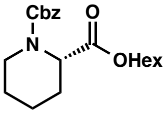


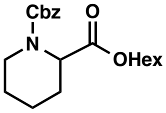
#	Time	Area	Height	Width	Area%	Symmetry
1	6.561	1169	96.6	0.1882	50.010	0.87
2	7.159	1168.5	87.9	0.2061	49.990	0.855



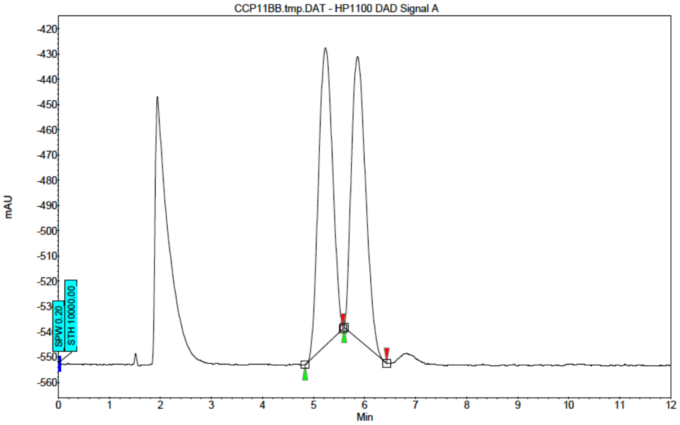


#	Time	Area	Height	Width	Area%	Symmetry
1	6.562	3396.4	279.4	0.1888	100.000	0.859

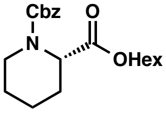
Compound	**Method Column/Temp.**	**Solvent**	**Method Flow Rate**	**Retention Times**	**Enantiomeric Ratio (er)**
 ***rac-36***	Daicel ChiralPak OD-H/35 °C	5% MeCN in CO_2	1 mL/min	4.83/5.60	51:49
 ***36***	Daicel ChiralPak OD-H/35 °C	5% MeCN in CO_2	1 mL/min	4.98	98:2



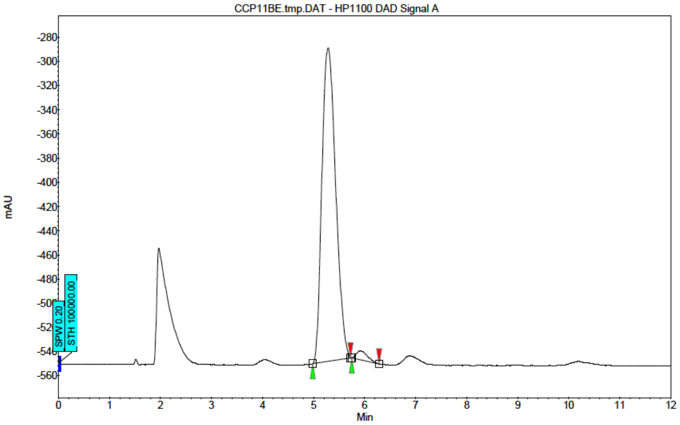
rac-36



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	4.83	5.23	5.58	0.00	50.88	117.9	37.6	50.876
2	UNKNOWN	5.60	5.87	6.43	0.00	49.12	111.7	36.3	49.124
Total						100.00	229.6	73.9	100.000



36



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	4.98	5.28	5.73	0.00	97.72	259.3	81.1	97.723
2	UNKNOWN	5.75	5.94	6.28	0.00	2.28	7.4	1.9	2.277
Total						100.00	266.7	83.0	100.000

References

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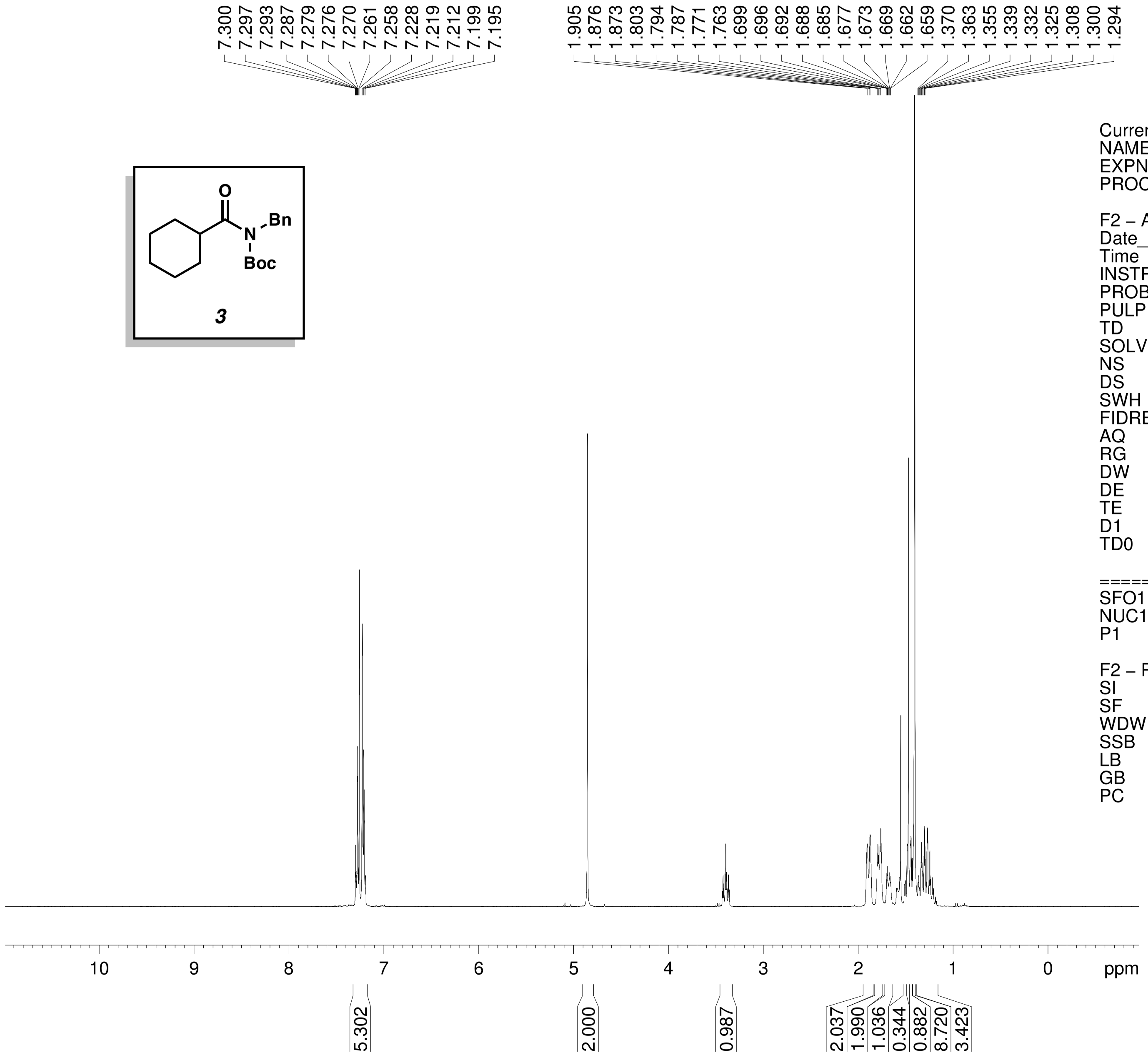
^{1}H NMR Spectra



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EXPNO              130
PROCNO               1

F2 - Acquisition Parameters
Date_         20151105
Time             10.06
INSTRUM          av400
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               52882
SOLVENT          CDCl3
NS                  16
DS                   0
SWH         8012.820 Hz
FIDRES      0.151523 Hz
AQ        3.2998867 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

F2 - Processing parameters
SI               65536
SF       400.1300184 MHz
WDW                 EM
SSB                  0
LB             0.30 Hz
GB                   0
PC                1.00
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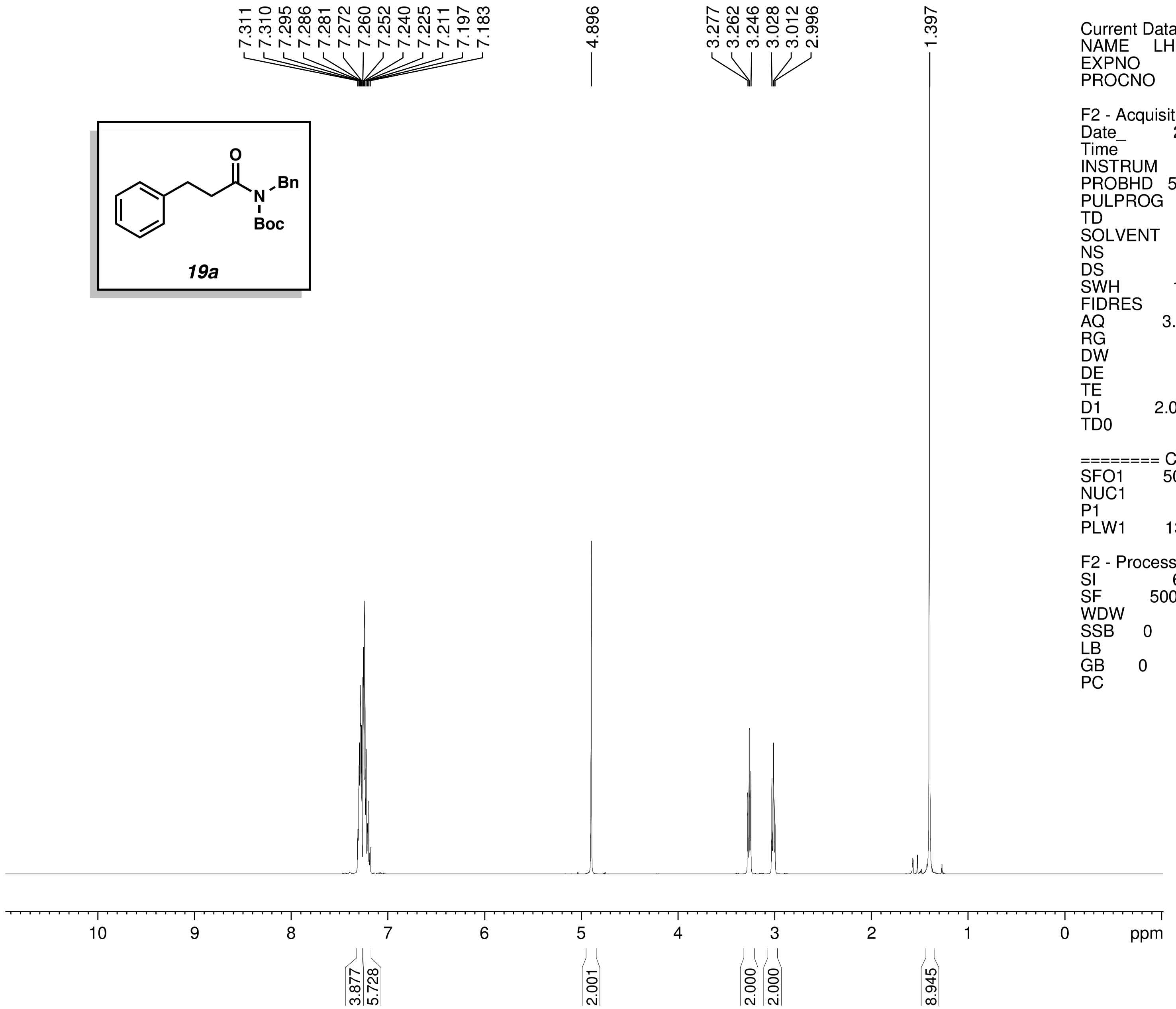


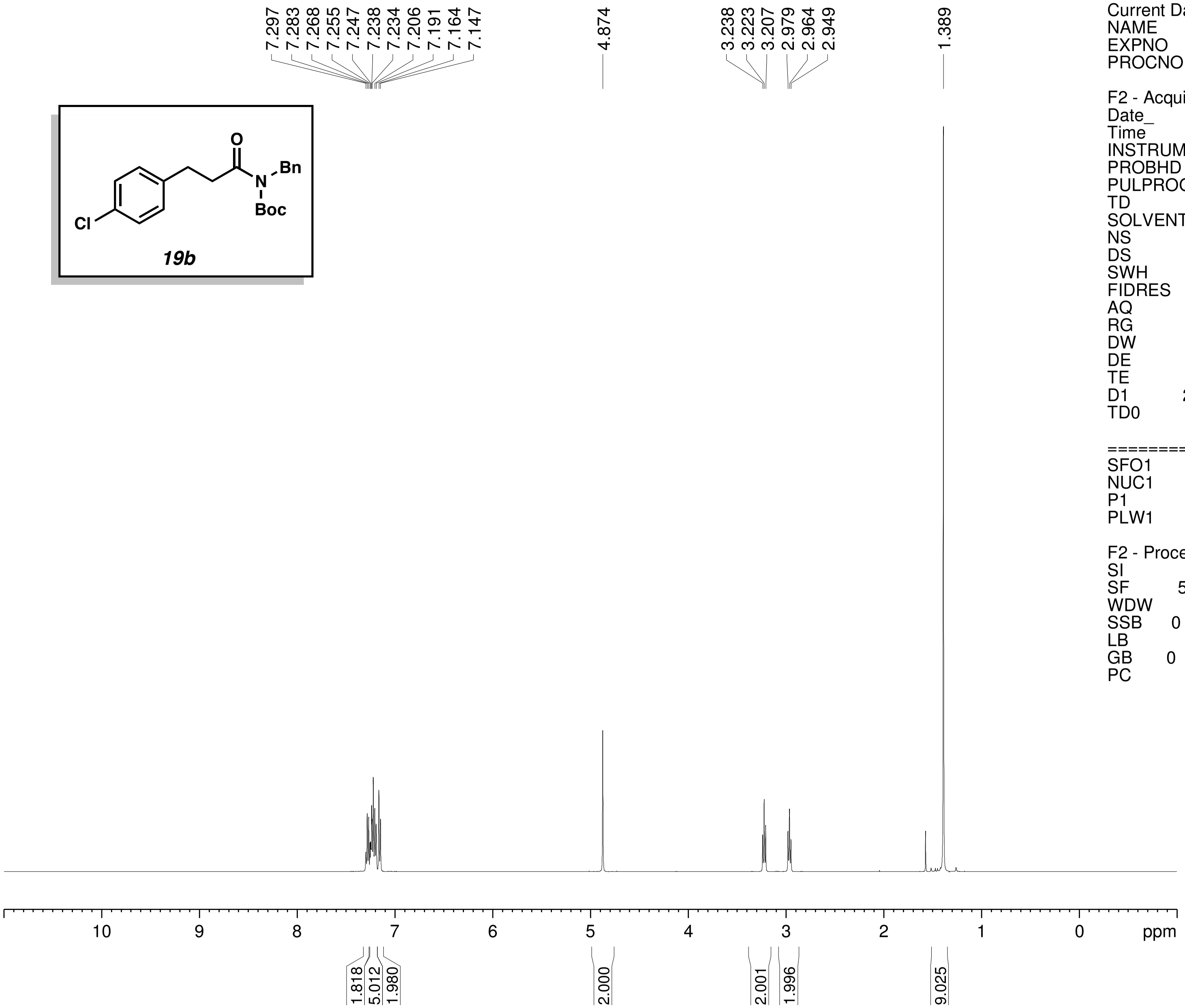
Current Data Parameters
NAME LH-9-44-proton
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160127
Time 13.30
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 14.67
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

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

Purified Product, 1H NMR



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EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
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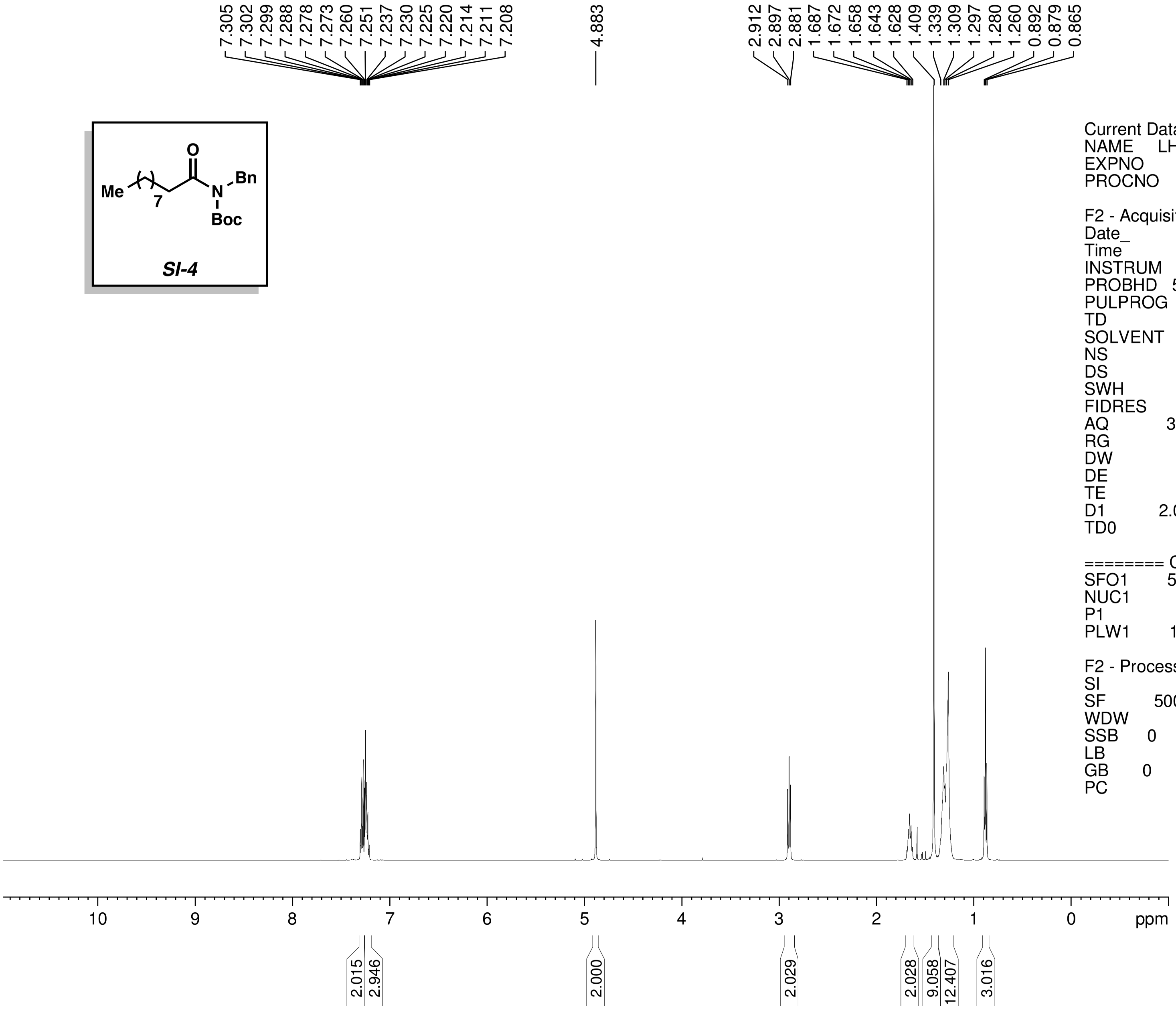
Time             9.29
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                  8
DS                  0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ        3.2767999 sec
RG              12.14
DW             50.000 usec
DE              10.00 usec
TE              298.0 K
D1        2.00000000 sec
TD0                 1

======== CHANNEL f1 =====
SFO1      500.1330008 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
```



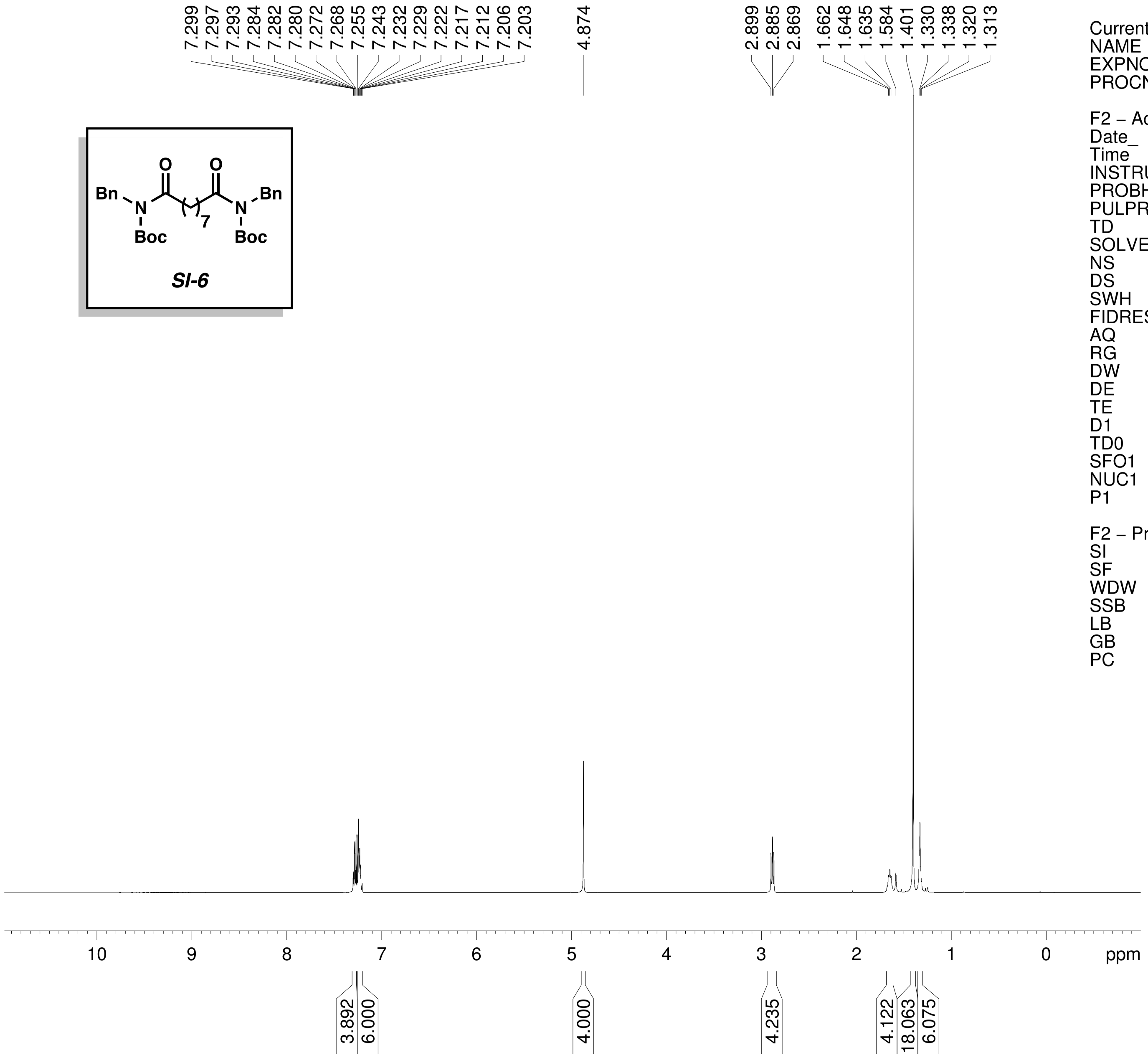
SI-4

Current Data Parameters
NAME LH-9-66-proton
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160129
Time 13.09
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 10.71
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

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

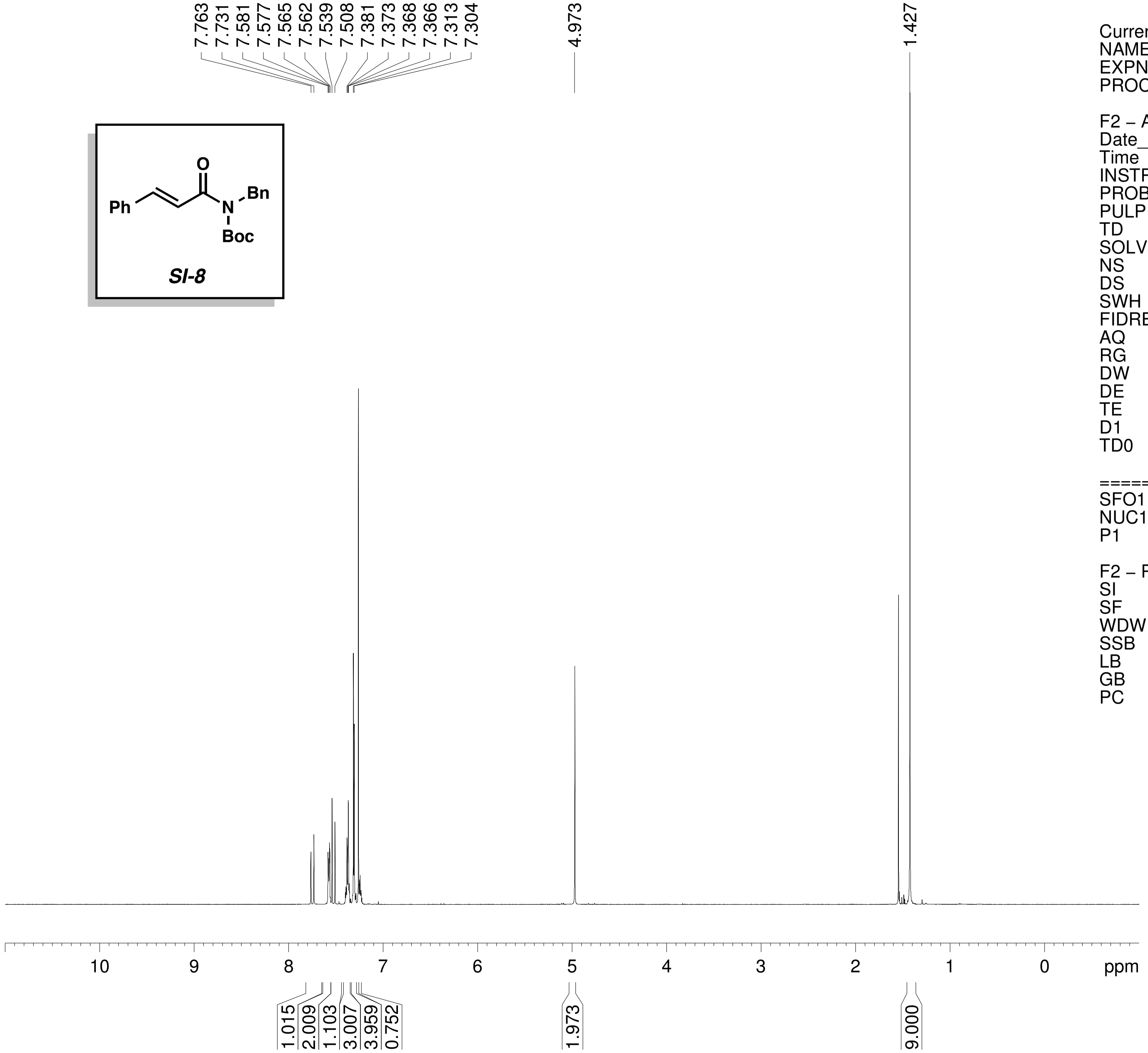


SI-6

```
Current Data Parameters
NAME          LH-9-255
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_         20160321
Time           19.10 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  21
DS                   0
SWH         10000.000 Hz
FIDRES       0.305176 Hz
AQ          3.2768500 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

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



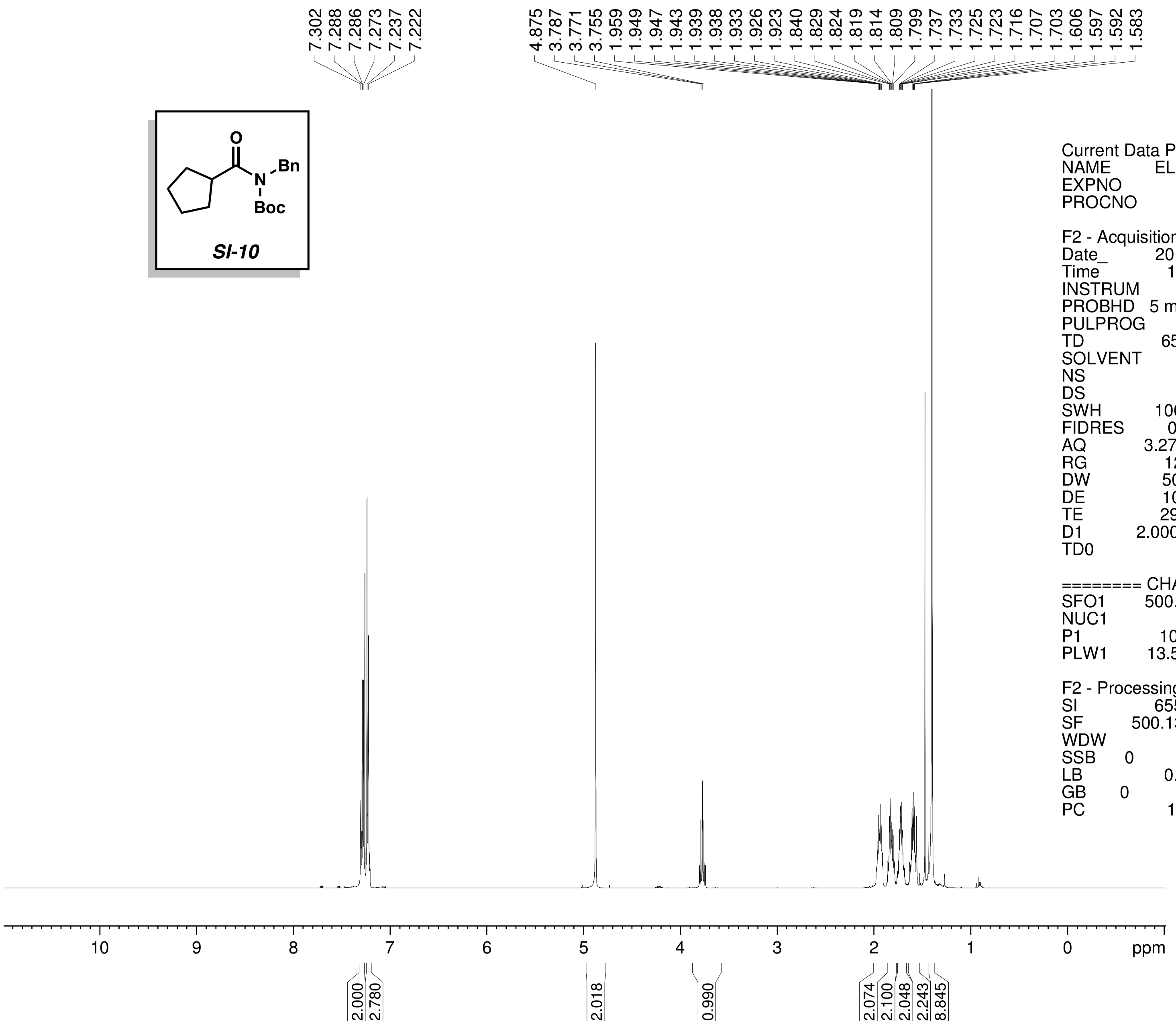
SI-8

```
Current Data Parameters
NAME        ELB-5-102p
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_         20160120
Time             16.13
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          3.2768500 sec
RG               19.06
DW              50.000 usec
DE               10.00 usec
TE               298.0 K
D1         2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       500.1330008 MHz
NUC1                1H
P1               10.00 usec

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



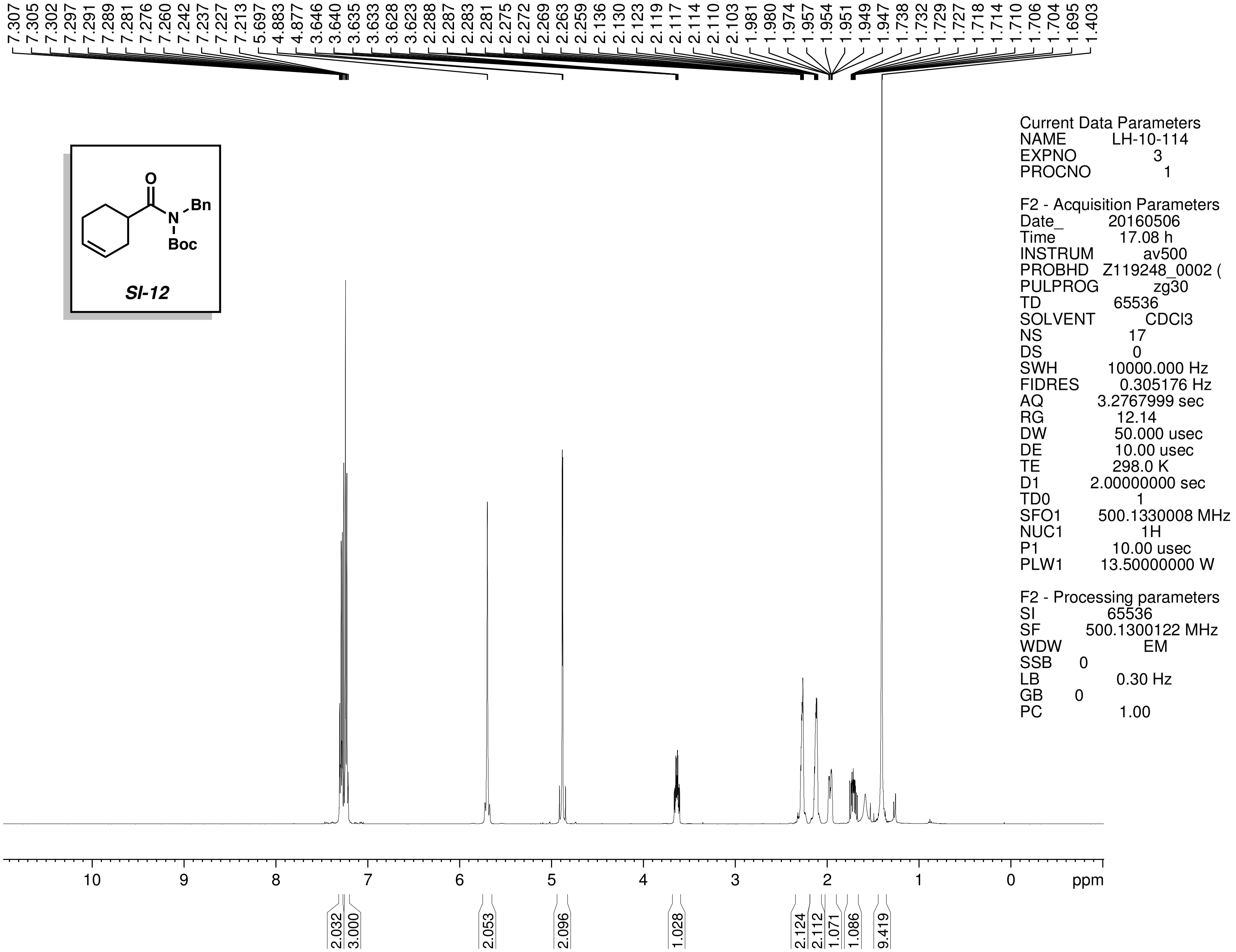
SI-10

```
Current Data Parameters
NAME        ELB-5-117p
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20160211
Time            10.23
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      500.1330008 MHz
NUC1                1H
P1              10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI              65536
SF        500.1300126 MHz
WDW                EM
SSB      0
LB               0.30 Hz
GB       0
PC               1.00
```

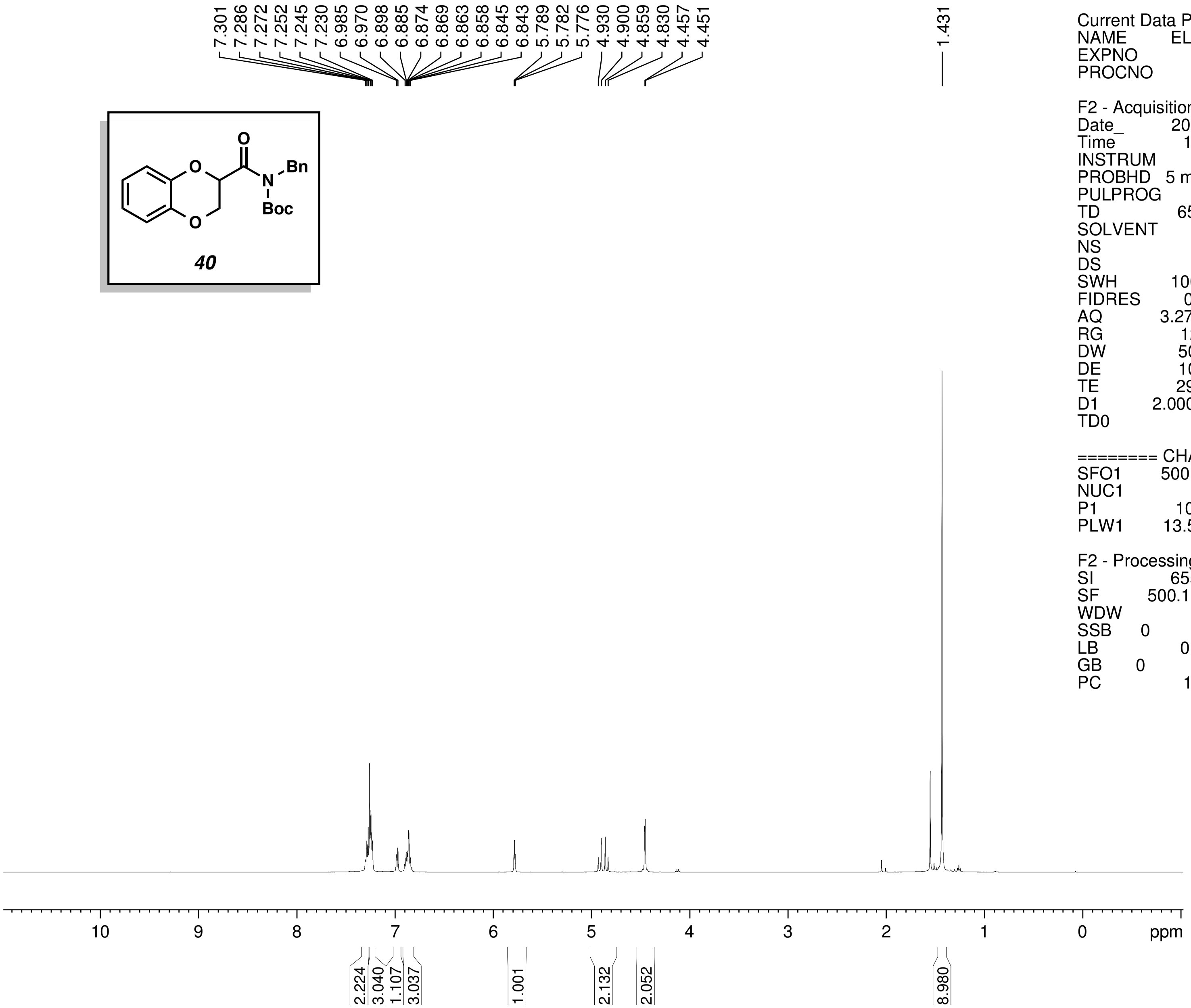


SI-12

```
Current Data Parameters
NAME          LH-10-114
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20160506
Time            17.08 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   17
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
```



40

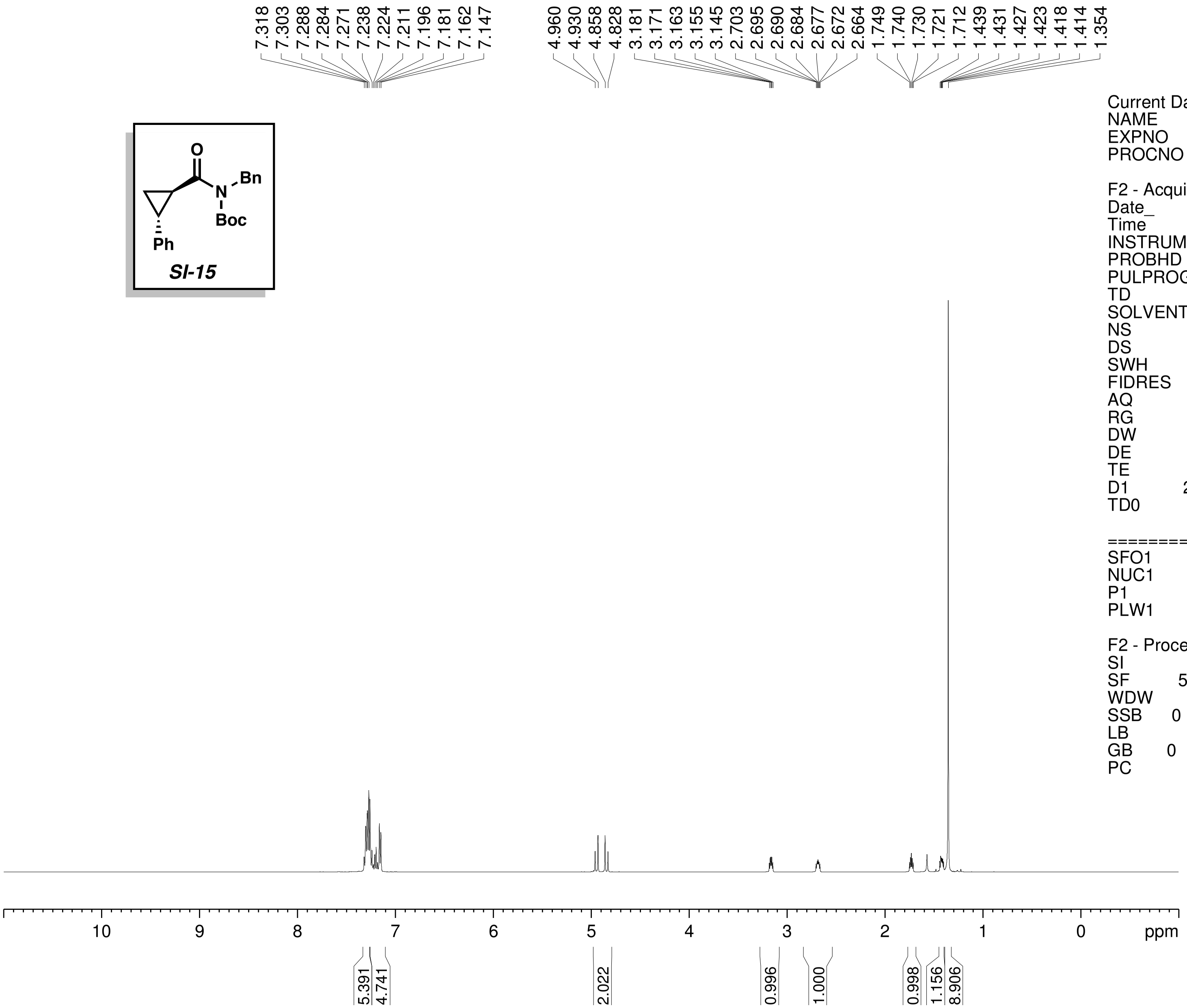
```
Current Data Parameters
NAME        ELB-5-106p
EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
Date_        20160121
Time            11.05
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      500.1330008 MHz
NUC1              1H
P1            10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI             65536
SF        500.1300129 MHz
WDW               EM
SSB     0
LB              0.30 Hz
GB      0
PC              1.00
```

Purified Product, 1H NMR



SI-15

Current Data Parameters
NAME ELB-6-127p
EXPNO 1
PROCNO 1

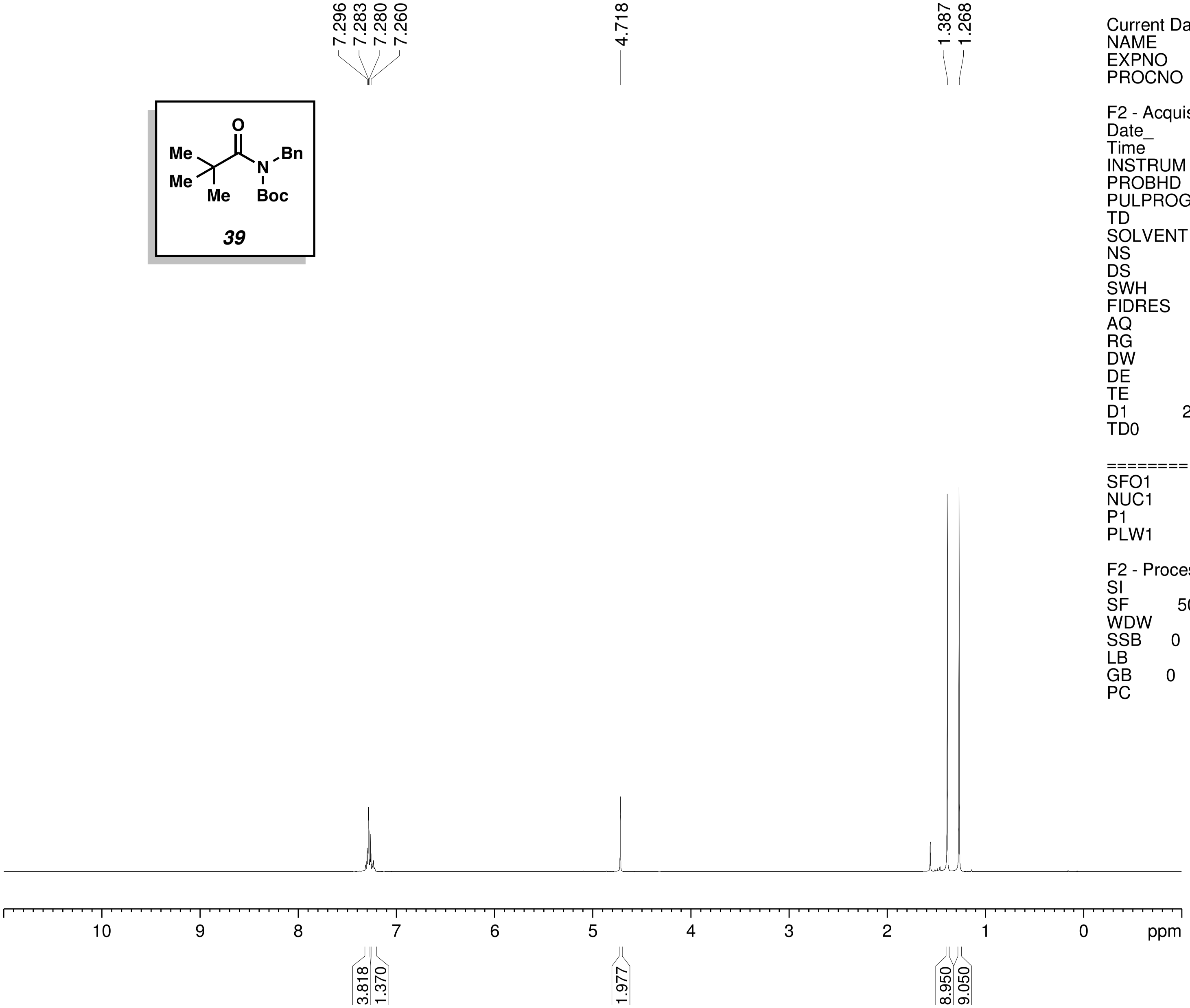
F2 - Acquisition Parameters
Date_ 20160614
Time 18.13
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 23.34
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 =====
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Purified Product, 1H NMR

39



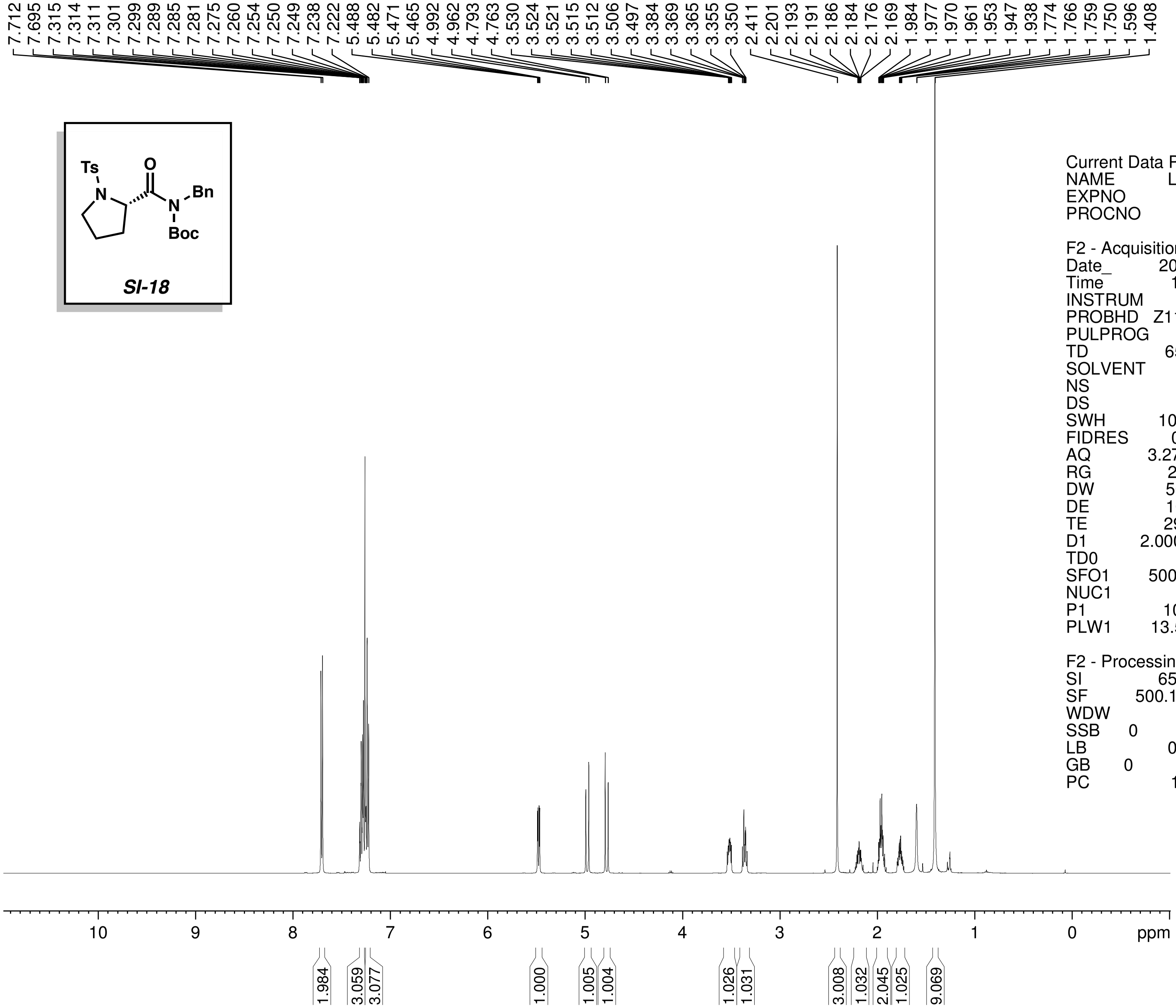
Current Data Parameters
NAME ELB-6-089p
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160607
Time 17.09
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 19.06
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 =====
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

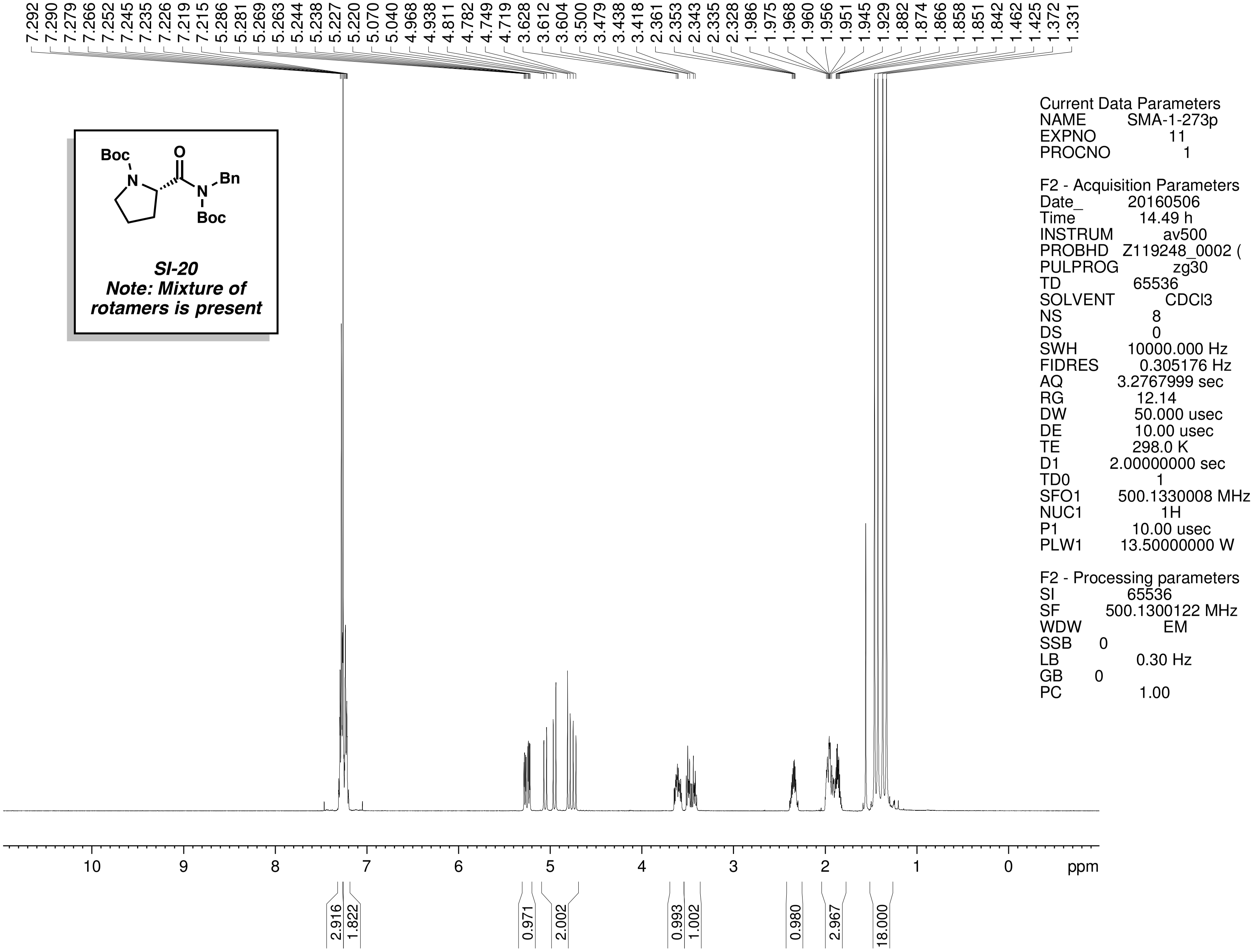
SI-18



Current Data Parameters
NAME LH-9-121
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160321
Time 19.25 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 18
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 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

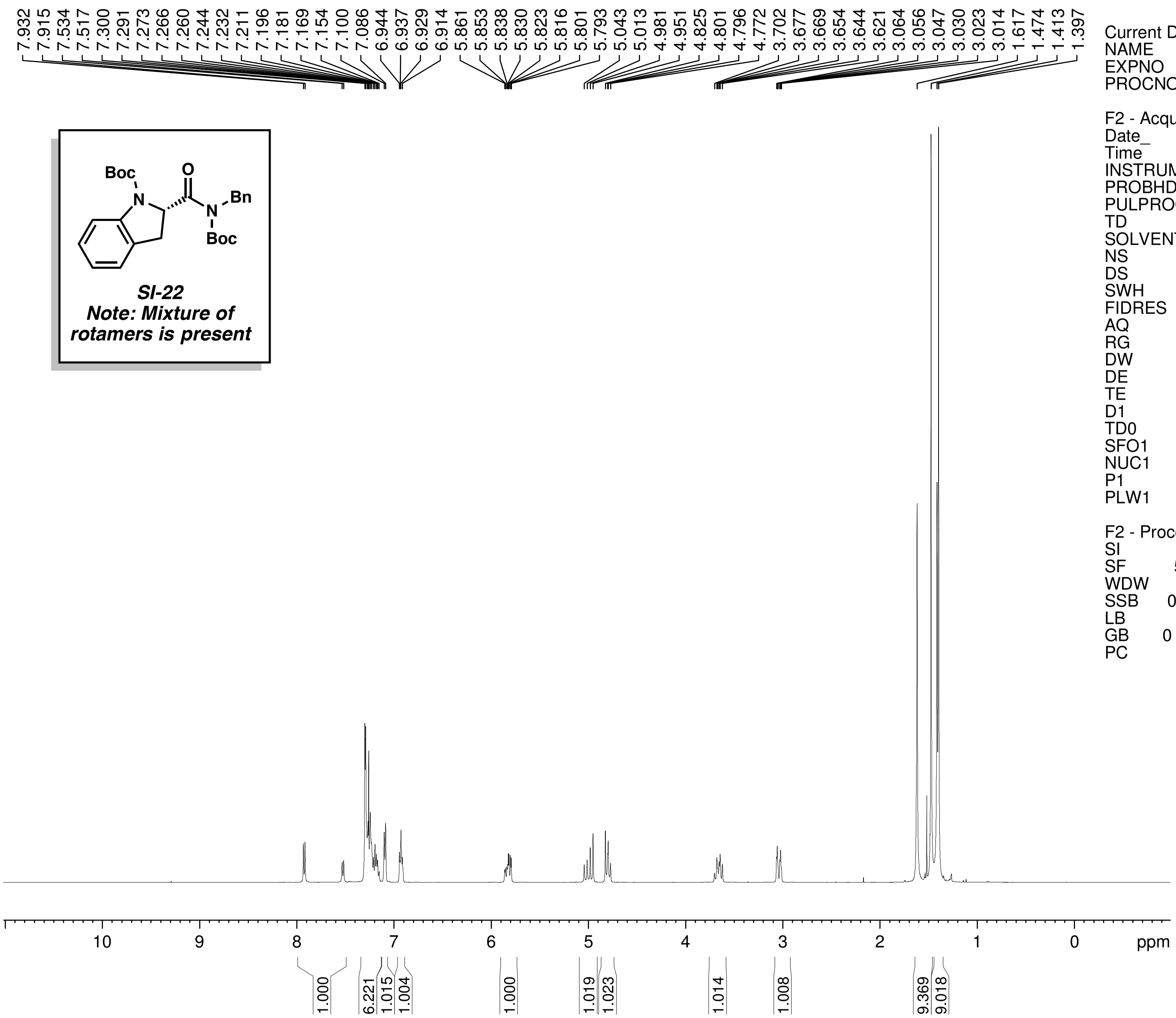


SI-20
Note: Mixture of rotamers is present

```
Current Data Parameters
NAME        SMA-1-273p
EXPNO             11
PROCNO             1

F2 - Acquisition Parameters
Date_        20160506
Time            14.49 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-22
Note: Mixture of rotamers is present

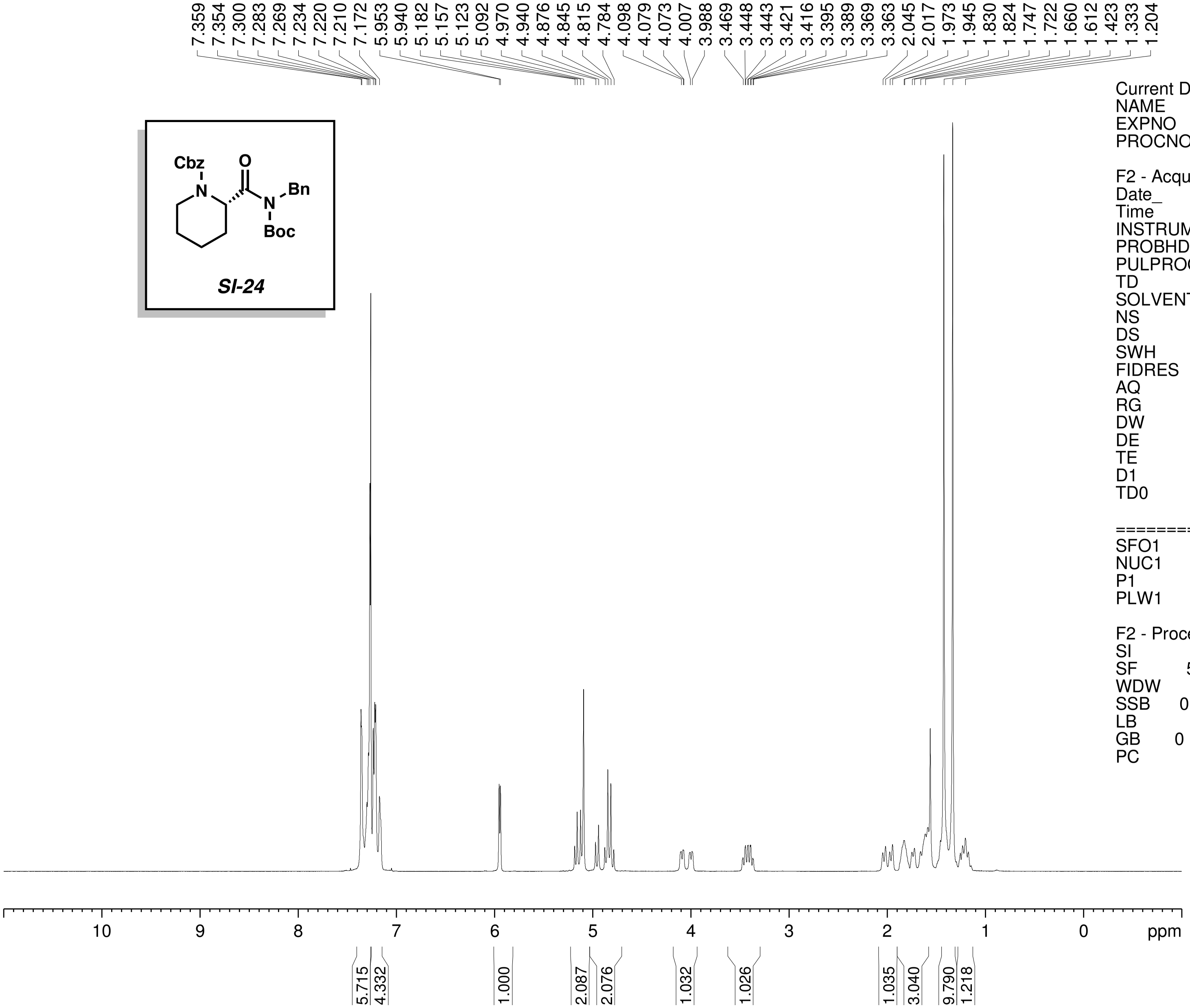
Current Data Parameters
NAME LH-10-131c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160530
Time 10.07 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 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

SI-24

Purified Product, 1H NMR

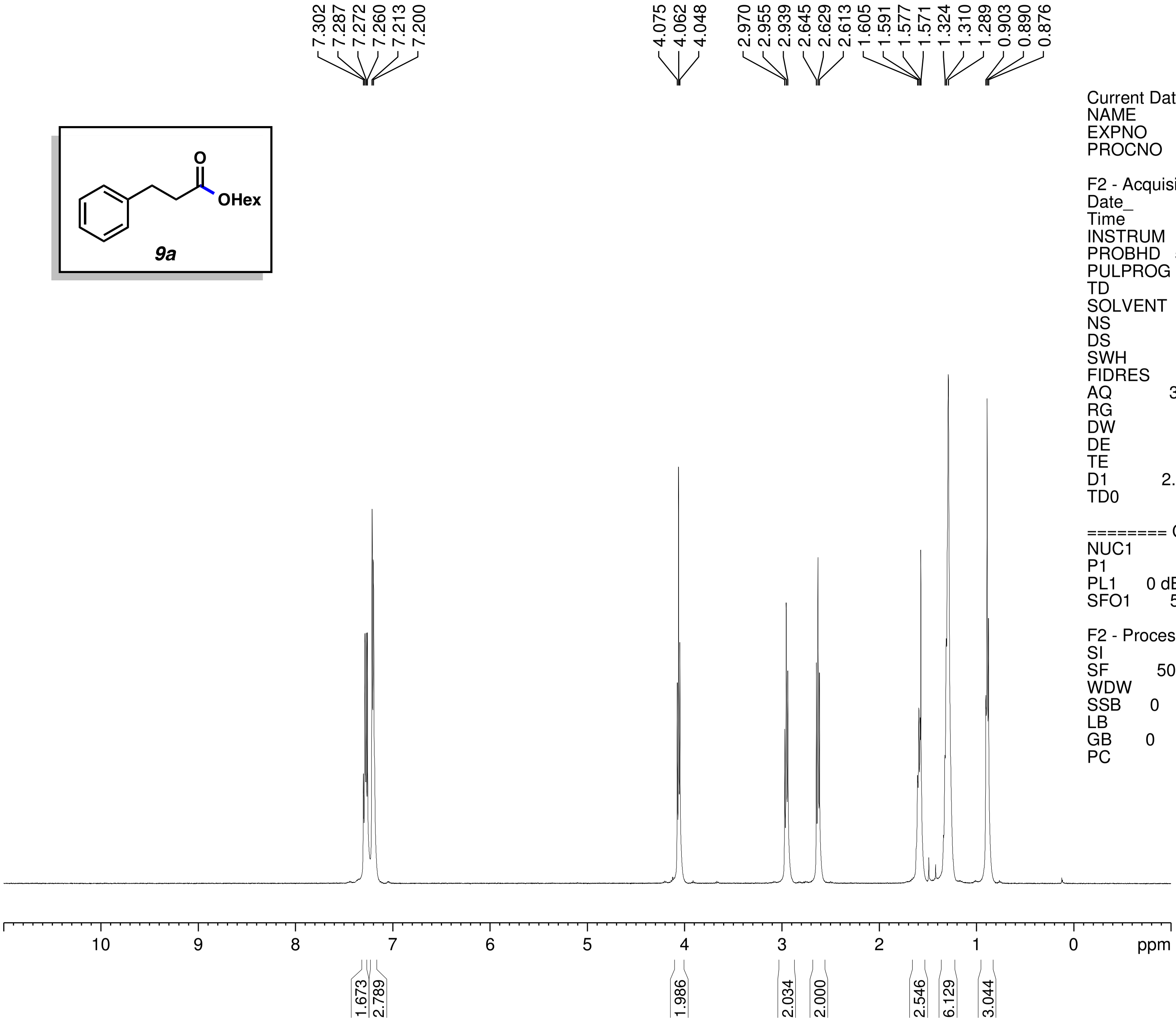


```
Current Data Parameters
NAME        ELB-6-104p
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20160609
Time           13.47
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      500.1330008 MHz
NUC1              1H
P1             10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI             65536
SF        500.1300126 MHz
WDW               EM
SSB    0
LB              0.30 Hz
GB     0
PC              1.00
```



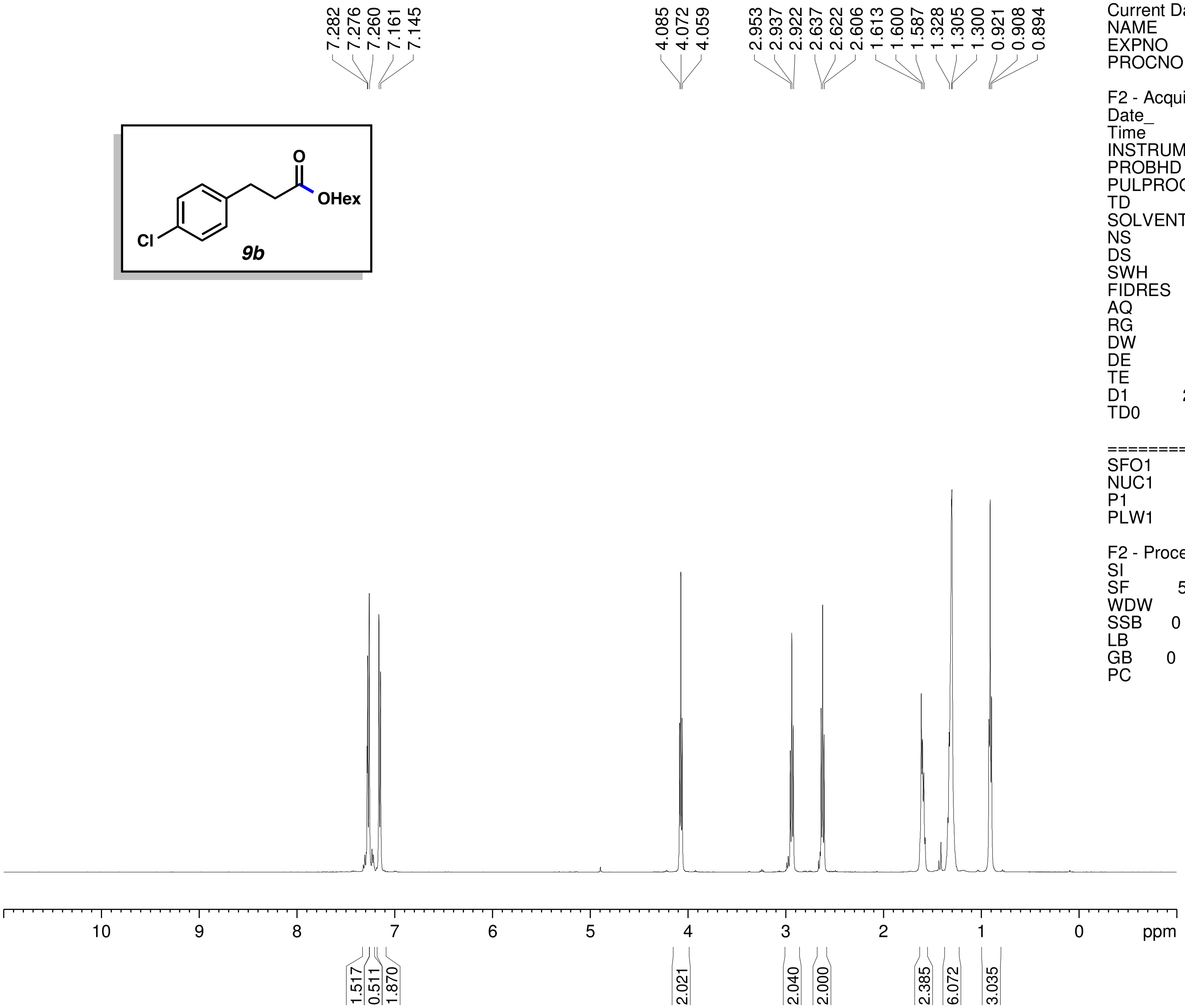
Current Data Parameters
NAME LH-9-274
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160118
Time 10.53
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 28
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 90.5
DW 50.000 usec
DE 6.00 usec
TE 296.7 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 500.3300222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

9b



Purified Product, 1H NMR

```
Current Data Parameters
NAME        ELB-6-280p
EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
Date_        20160918
Time           14.35
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      500.1330008 MHz
NUC1             1H
P1             10.00 usec
PLW1      13.50000000 W

F2 - Processing parameters
SI            65536
SF        500.1300011 MHz
WDW              EM
SSB     0
LB             0.30 Hz
GB      0
PC             1.00
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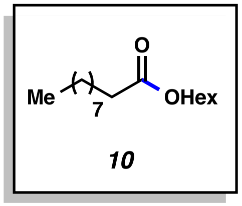
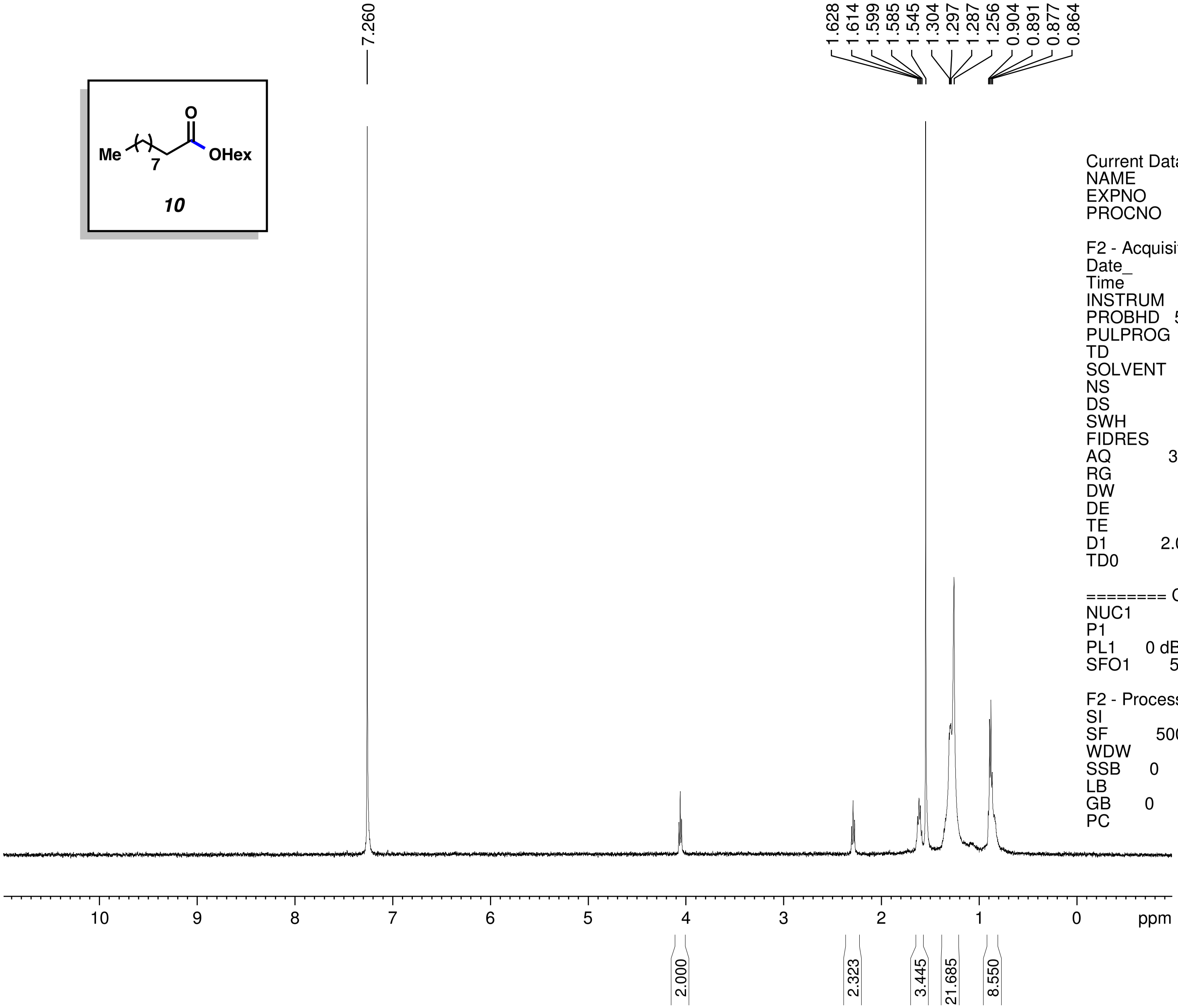
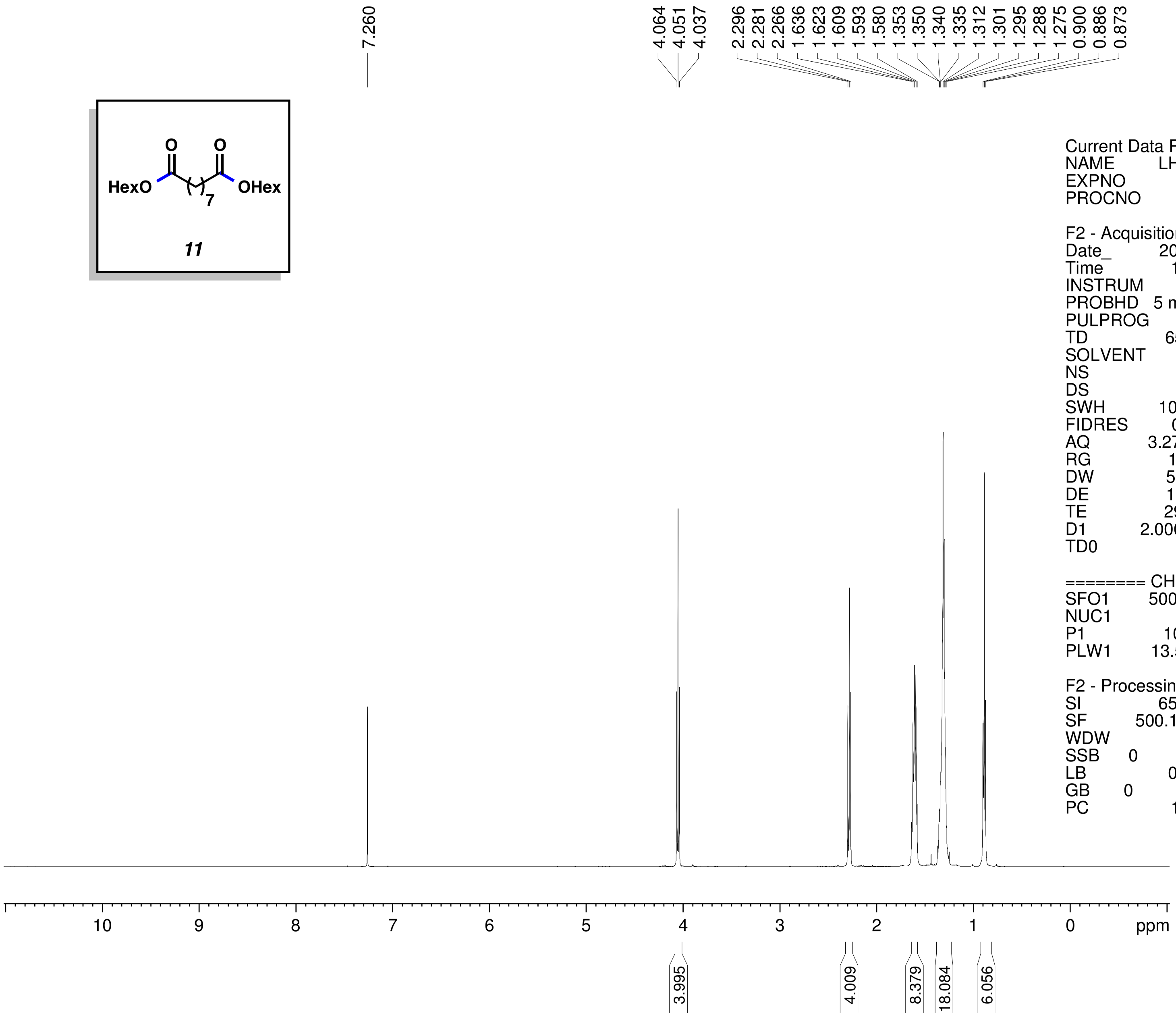


10



```
Current Data Parameters
NAME        LH-9-277-1
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20160119
Time           18.34
INSTRUM        drx500
PROBHD   5 mm bb-Z Z800
PULPROG          zg30
TD             65536
SOLVENT         CDCl3
NS                 9
DS                 0
SWH        10000.000 Hz
FIDRES      0.152588 Hz
AQ         3.2767999 sec
RG               181
DW            50.000 usec
DE              6.00 usec
TE             296.6 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        500.3300222 MHz
WDW               EM
SSB     0
LB              0.30 Hz
GB      0
PC              1.00
```

11

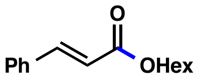


Current Data Parameters
NAME LH-9-288-4
EXPNO 1
PROCNO 1

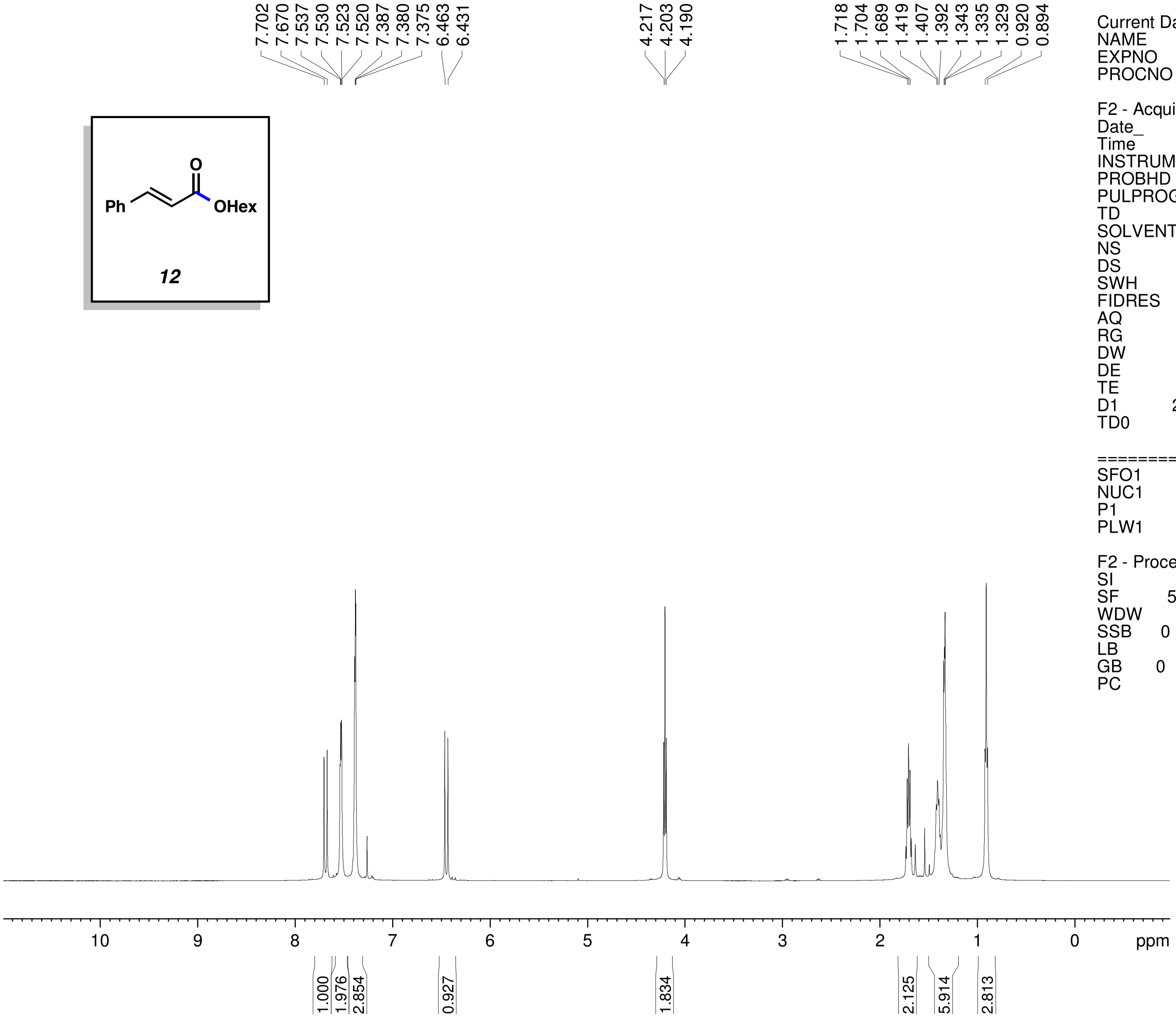
F2 - Acquisition Parameters
Date_ 20160129
Time 15.19
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 =====
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

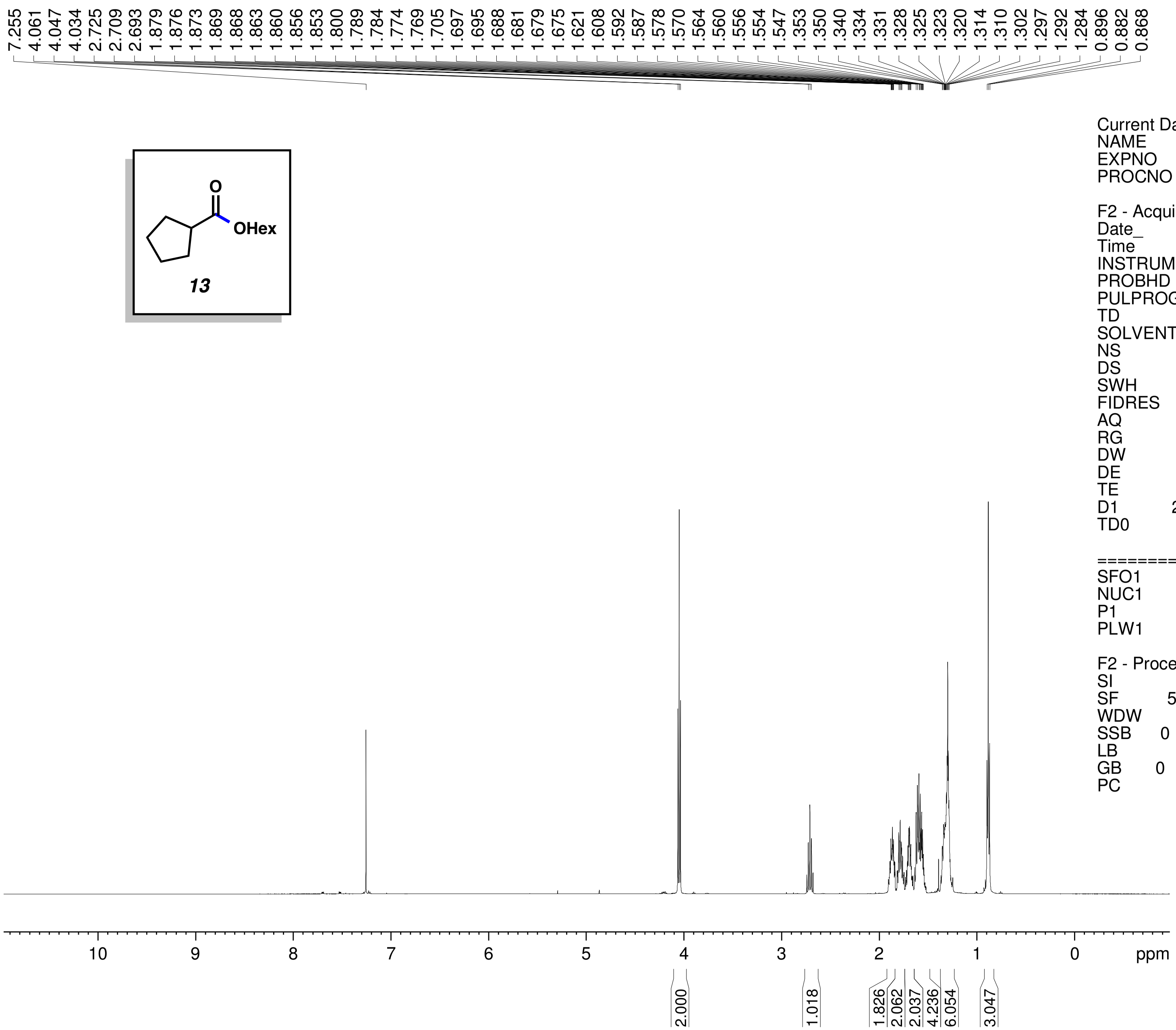
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



12

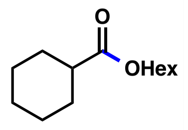


```
Current Data Parameters
NAME         ELB-5-149p
EXPNO               3
PROCNO              1

F2 - Acquisition Parameters
Date_         20160508
Time            10.38
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       500.1330008 MHz
NUC1                1H
P1              10.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI             65536
SF         500.1300128 MHz
WDW                EM
SSB     0
LB               0.30 Hz
GB      0
PC               1.00
```

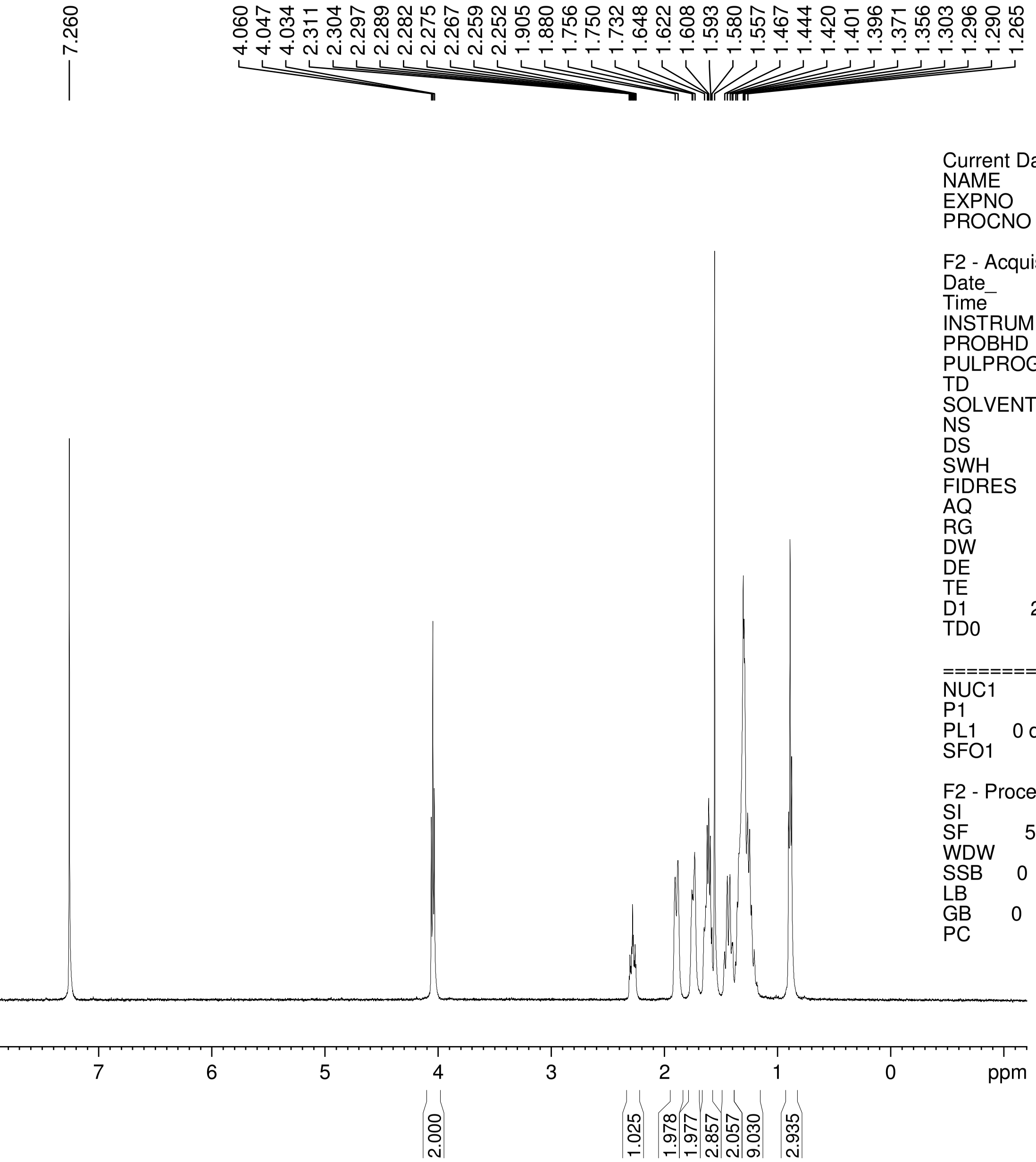


13

Current Data Parameters	
NAME	ELB-5-202p
EXPNO	9
PROCNO	1
F2 - Acquisition Parameters	
Date_	20160509
Time	12.26
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	500.1330008 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

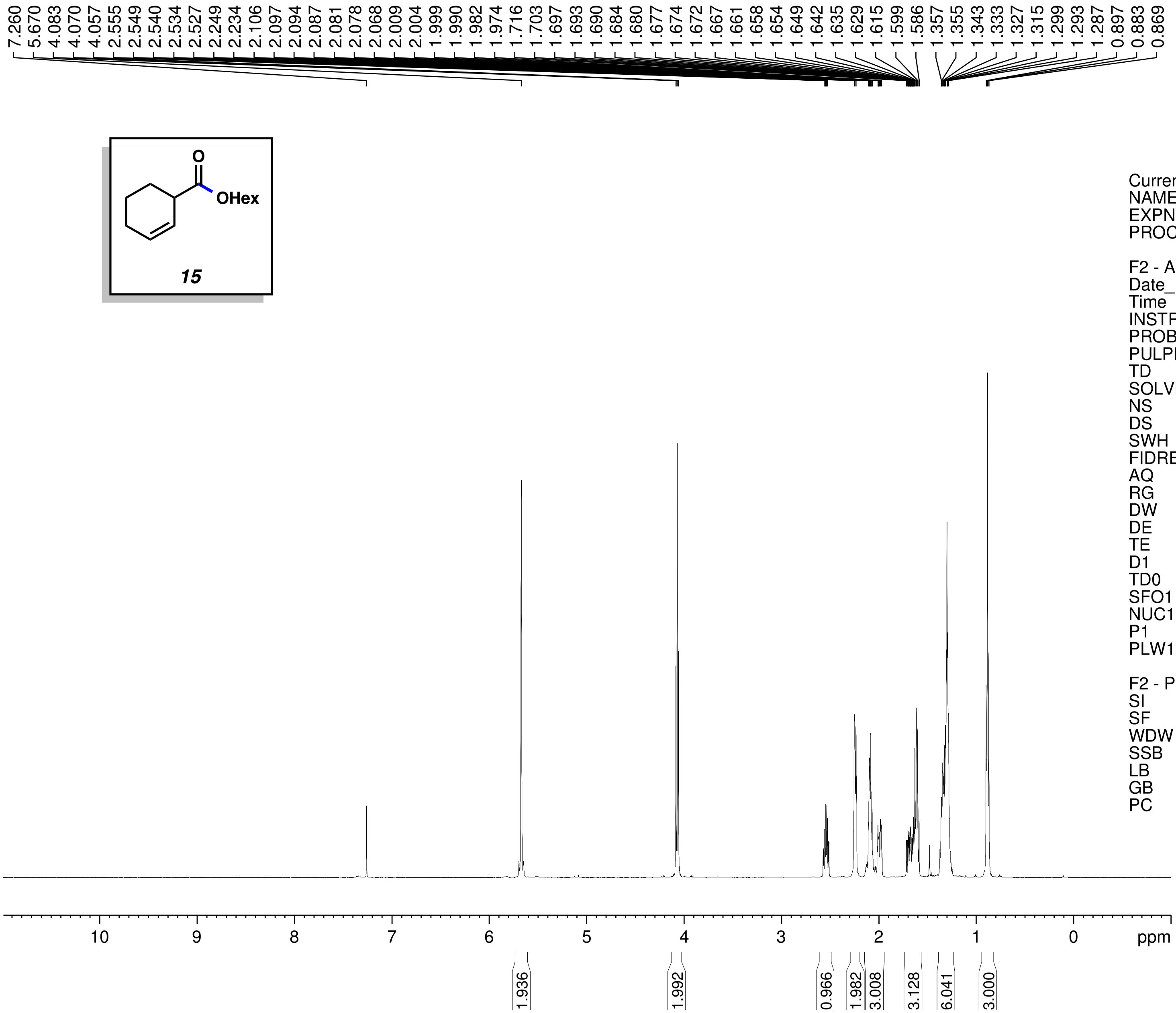


14



```
Current Data Parameters
NAME          LH-9-278
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20160119
Time             18.39
INSTRUM         drx500
PROBHD   5 mm bb-Z Z800
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   0
SWH       10000.000 Hz
FIDRES     0.152588 Hz
AQ       3.2767999 sec
RG               161.3
DW          50.000 usec
DE            6.00 usec
TE             296.6 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      500.3300222 MHz
WDW                 EM
SSB     0
LB             0.30 Hz
GB      0
PC                1.00
```

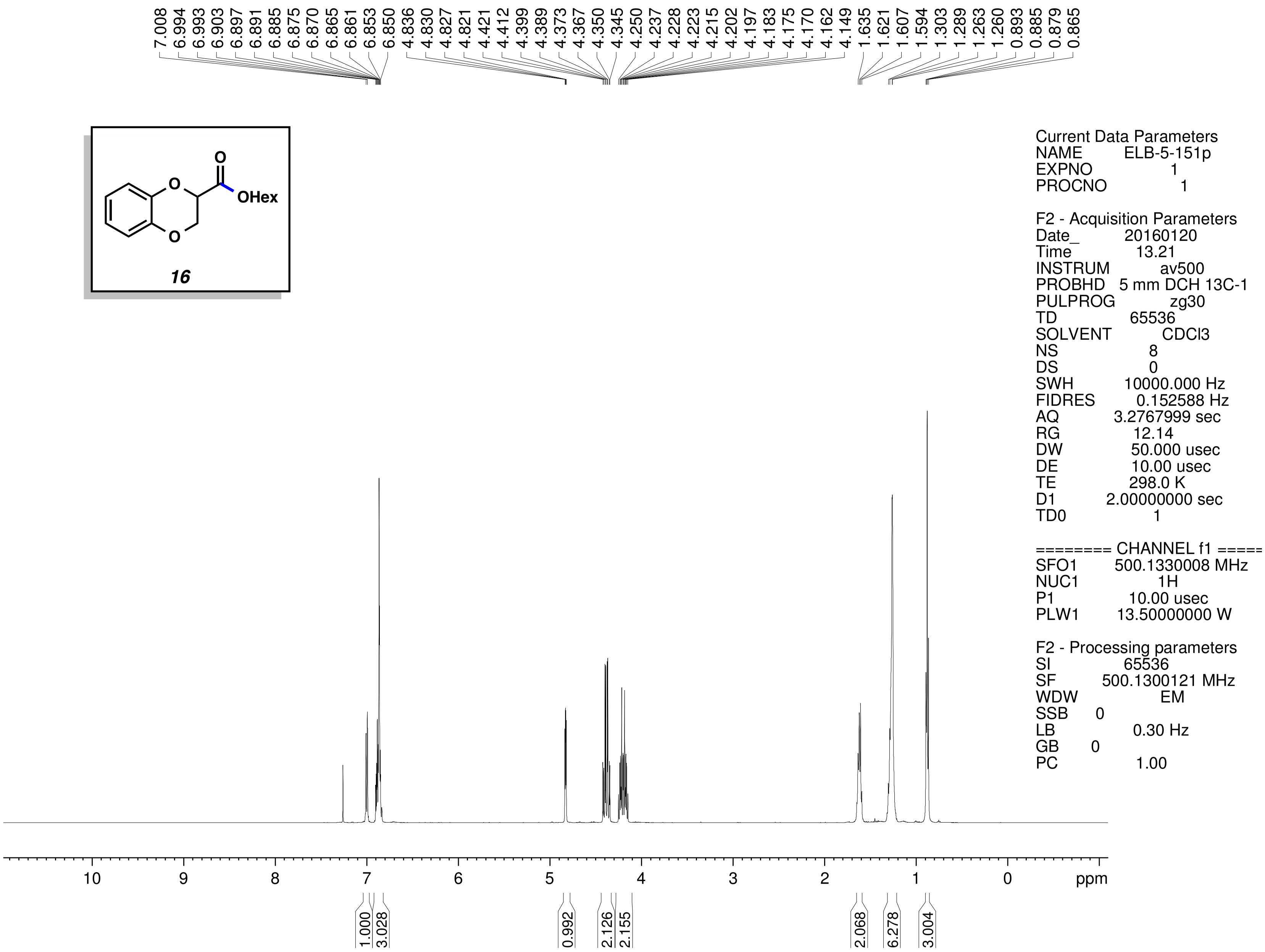
15



Current Data Parameters
NAME LH-10-124
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160504
Time 19.26 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 17
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

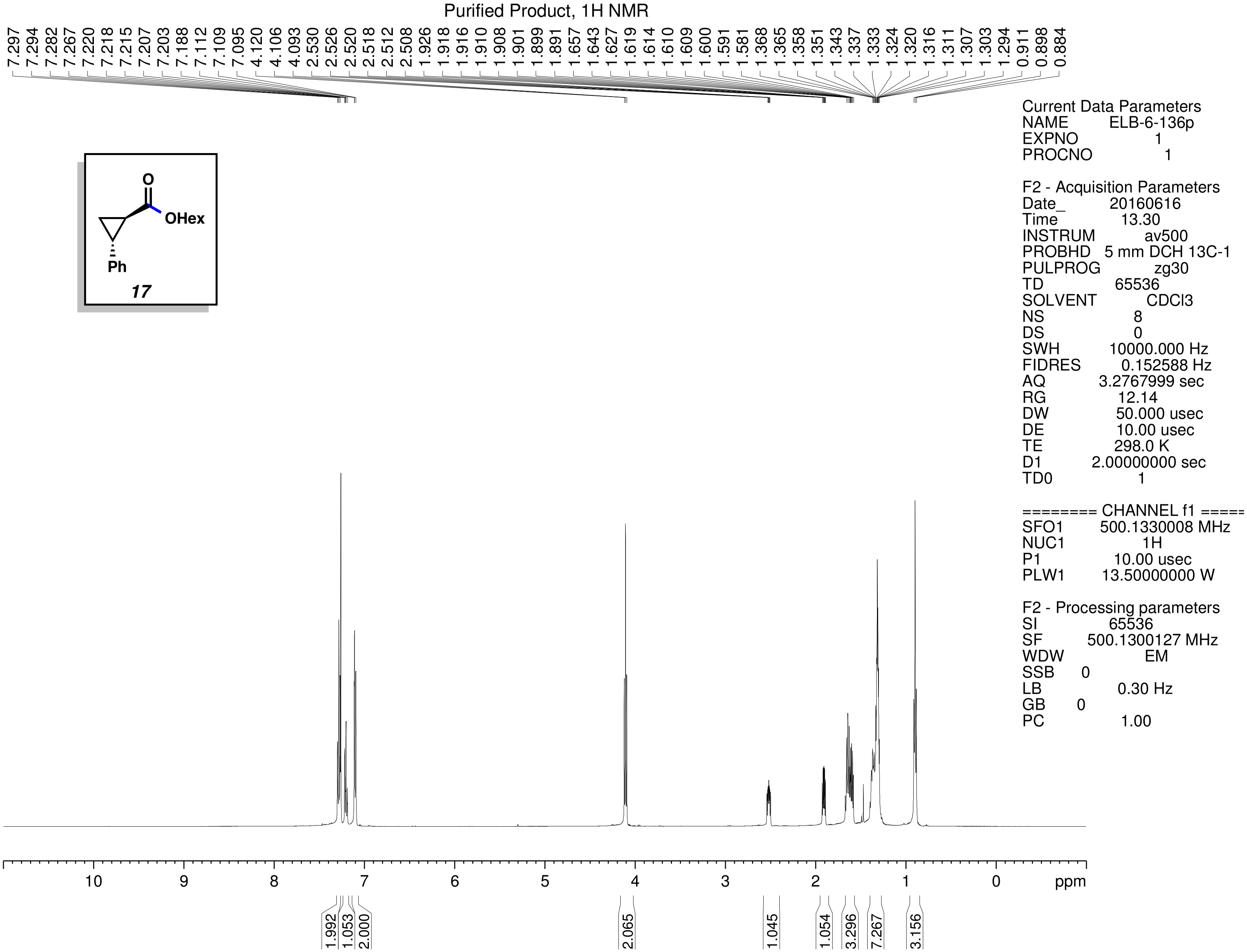
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



16

```
Current Data Parameters
NAME        ELB-5-151p
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20160120
Time            13.21
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      500.1330008 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
```

Purified Product, 1H NMR



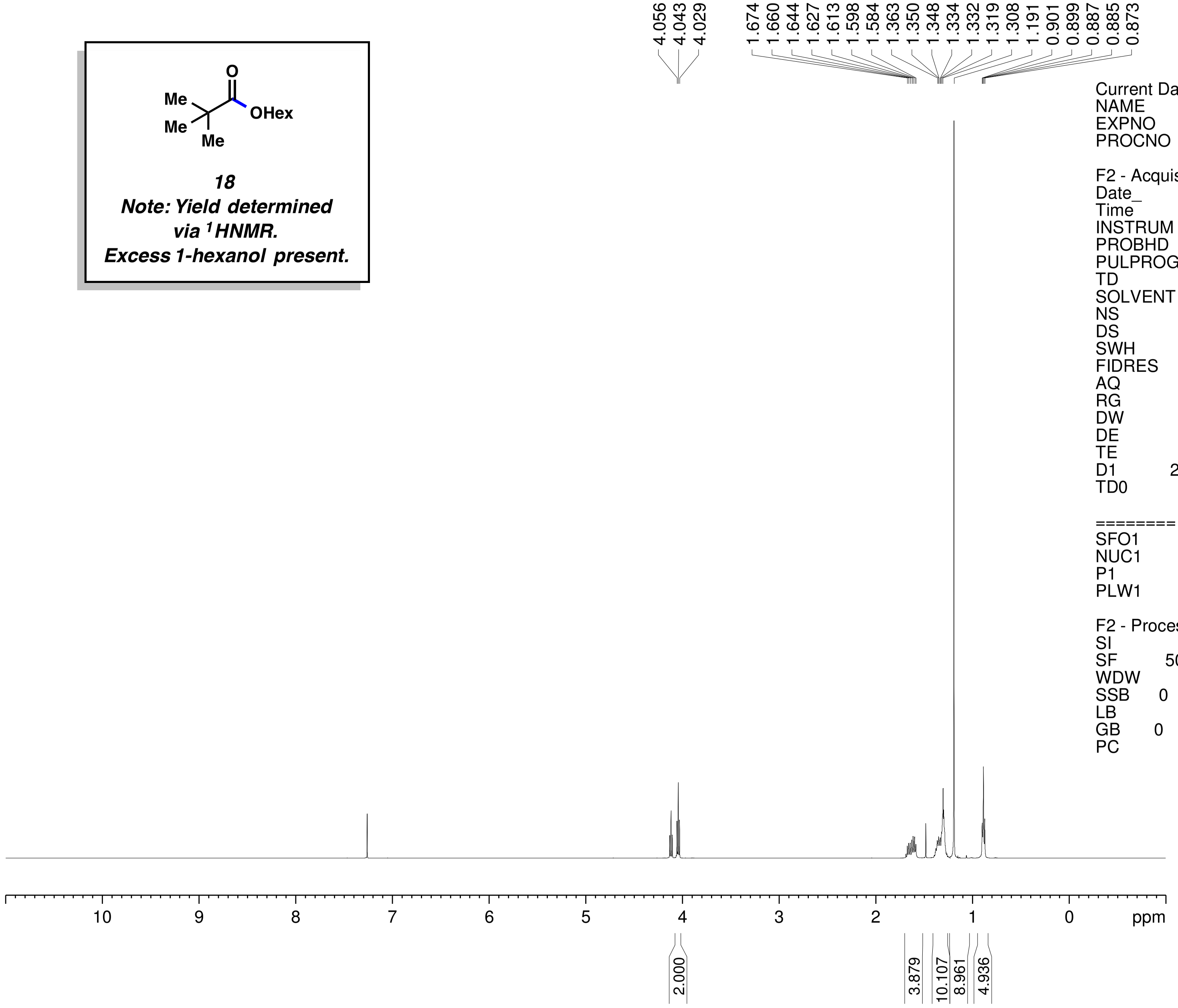
17

Current Data Parameters
NAME ELB-6-136p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160616
Time 13.30
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 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



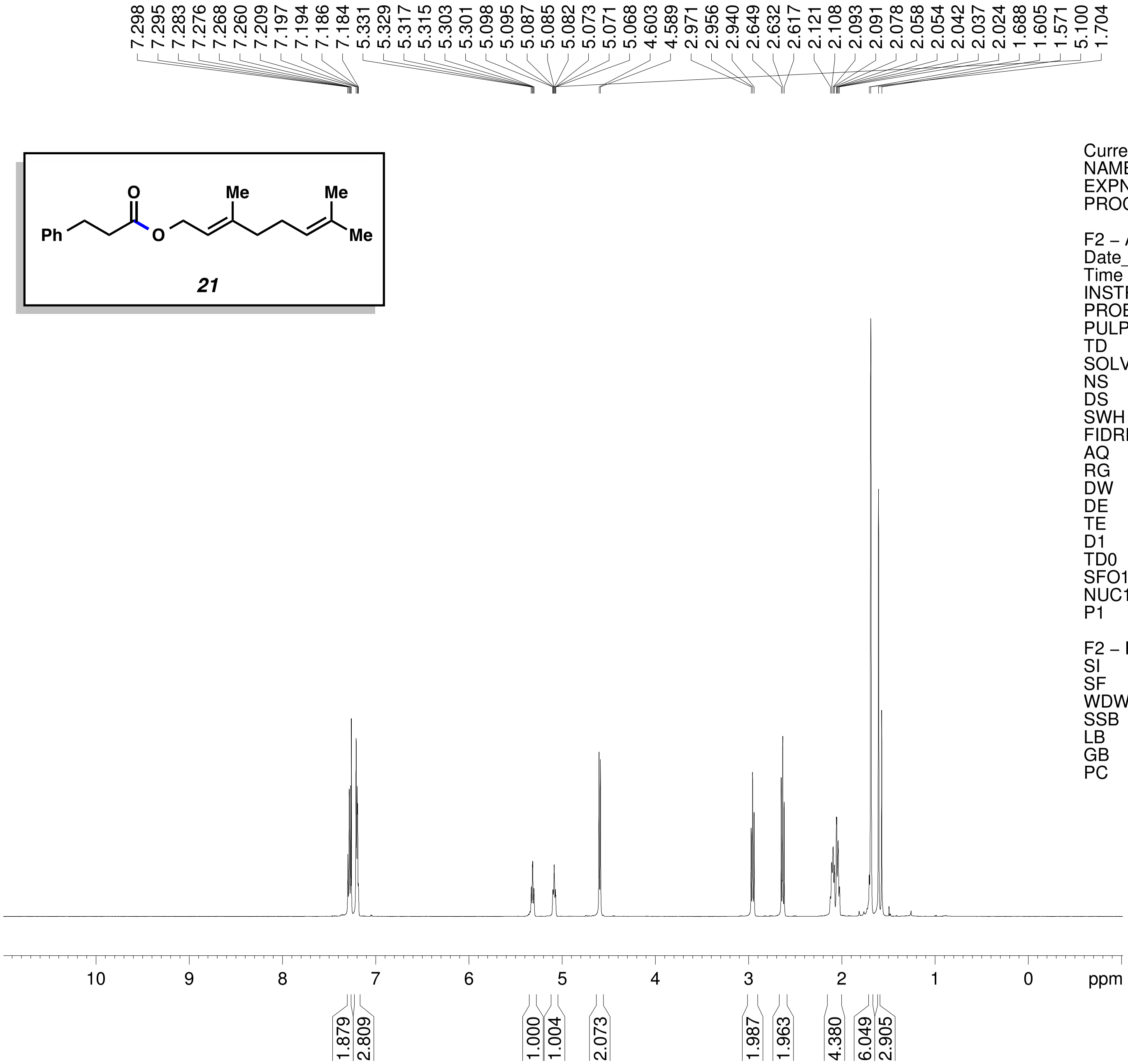
18

Note: Yield determined via ^{1}HNMR.

Excess 1-hexanol present.

Purified Product, 1H NMR

```
Current Data Parameters
NAME        ELB-6-105p
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20160608
Time             9.33
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      500.1330008 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
```

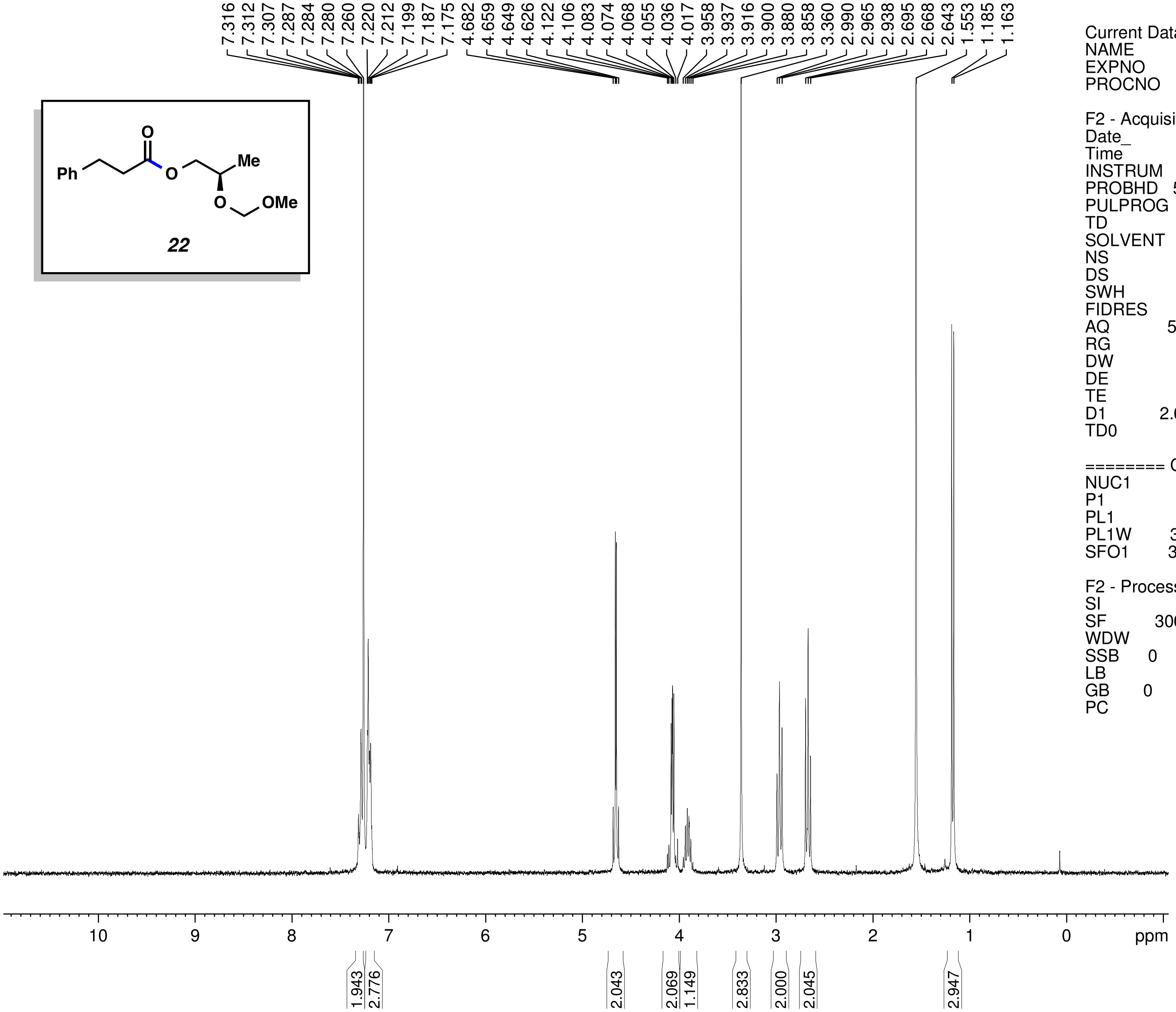


21

Current Data Parameters
NAME LH−10−70
EXPNO 1
PROCNO 1

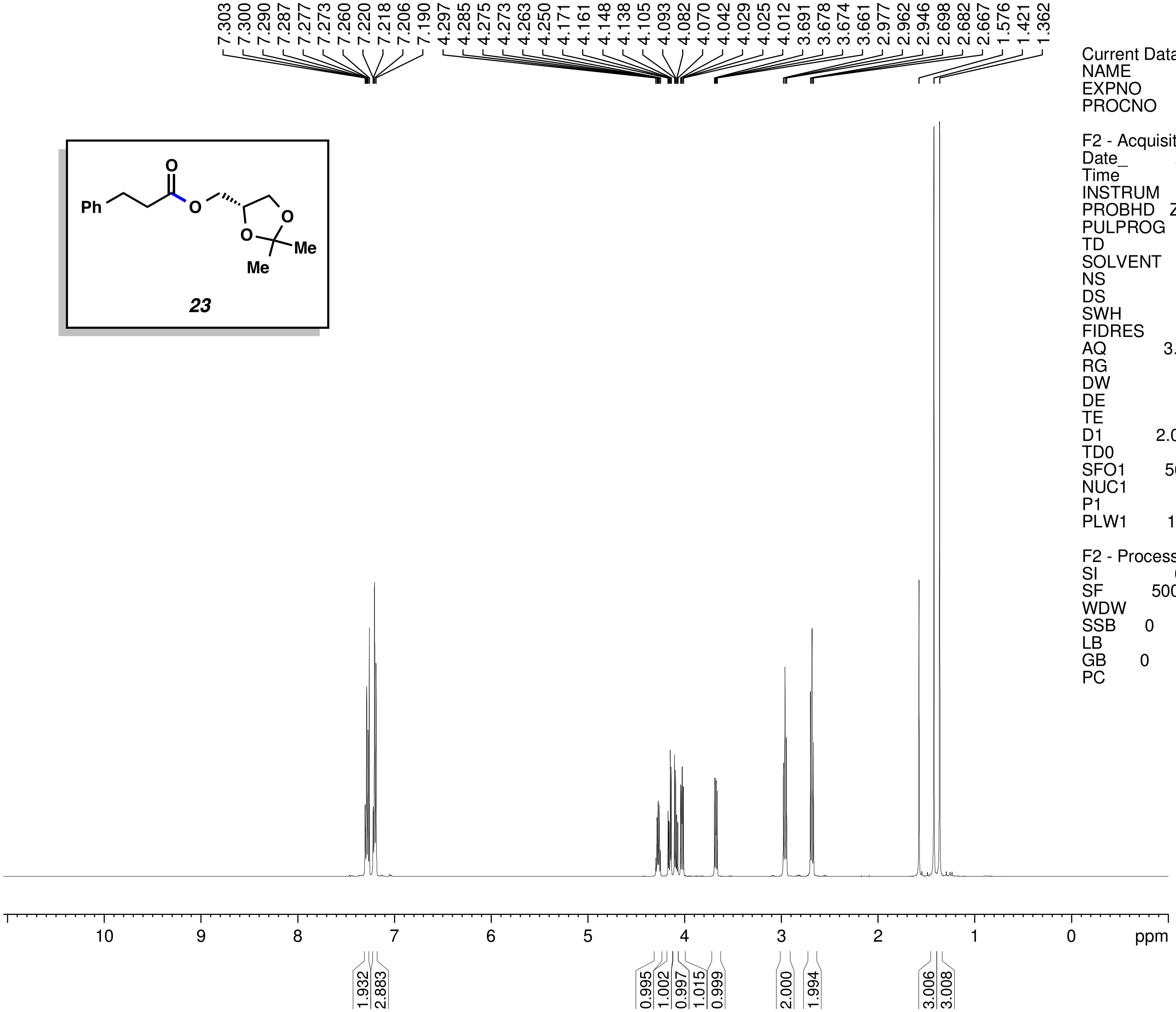
F2 − Acquisition Parameters
Date_ 20160328
Time 9.29 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2768500 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec

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



22

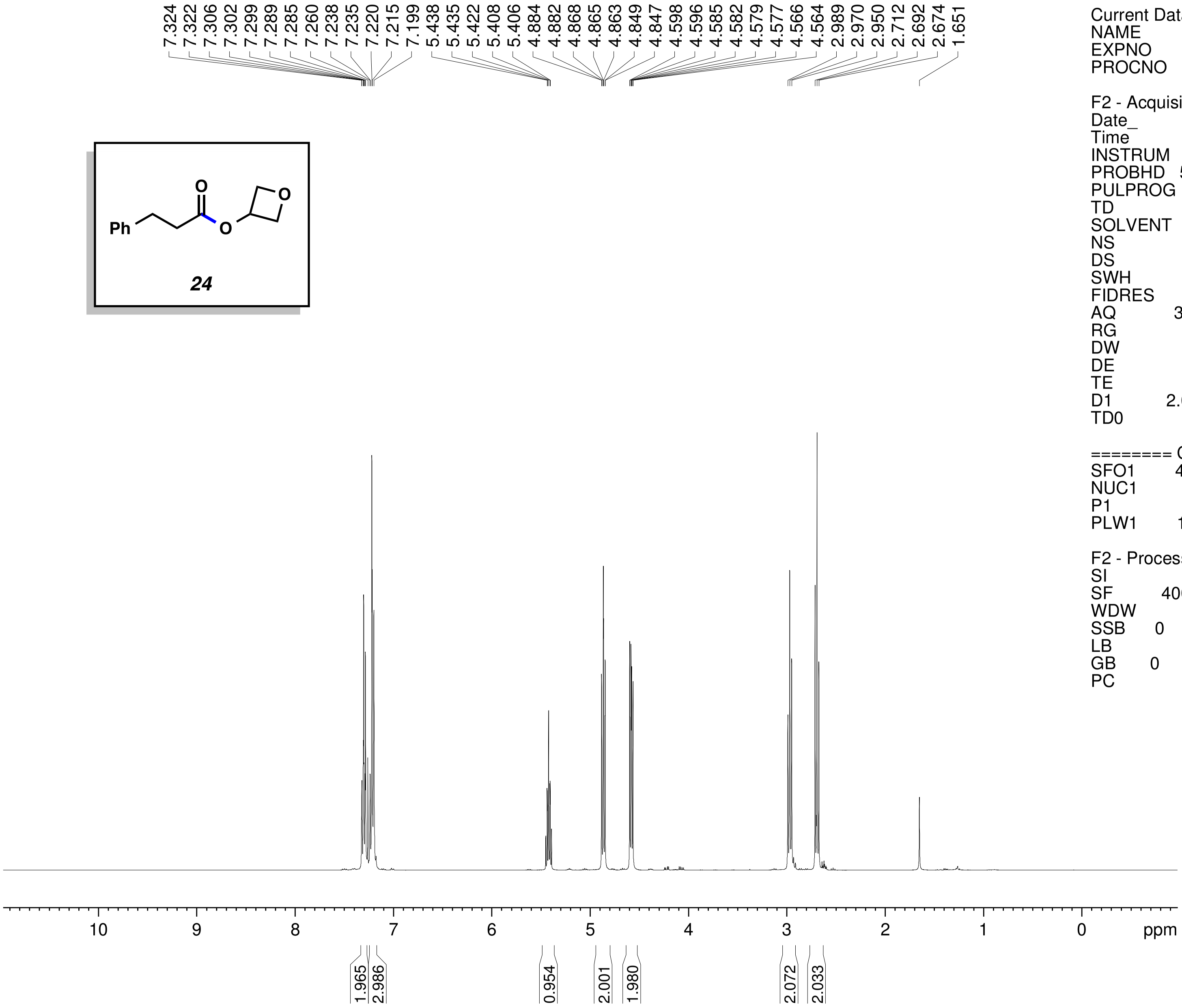
```
Current Data Parameters
NAME          LH-10-83
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20160406
Time             16.48
INSTRUM          av300
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  23
DS                   0
SWH        5995.204 Hz
FIDRES     0.091480 Hz
AQ        5.4657025 sec
RG               574.7
DW          83.400 usec
DE            6.00 usec
TE             298.0 K
D1      2.00000000 sec
TD0                  1

======== CHANNEL f1 =====
NUC1                1H
P1          11.10 usec
PL1           -6.00 dB
PL1W    37.09999847 W
SFO1    300.1318008 MHz

F2 - Processing parameters
SI               65536
SF      300.1300124 MHz
WDW                 EM
SSB      0
LB             0.30 Hz
GB       0
PC                1.40
```



Current Data Parameters
NAME LH-10-92
EXPNO 2
PROCNO 1

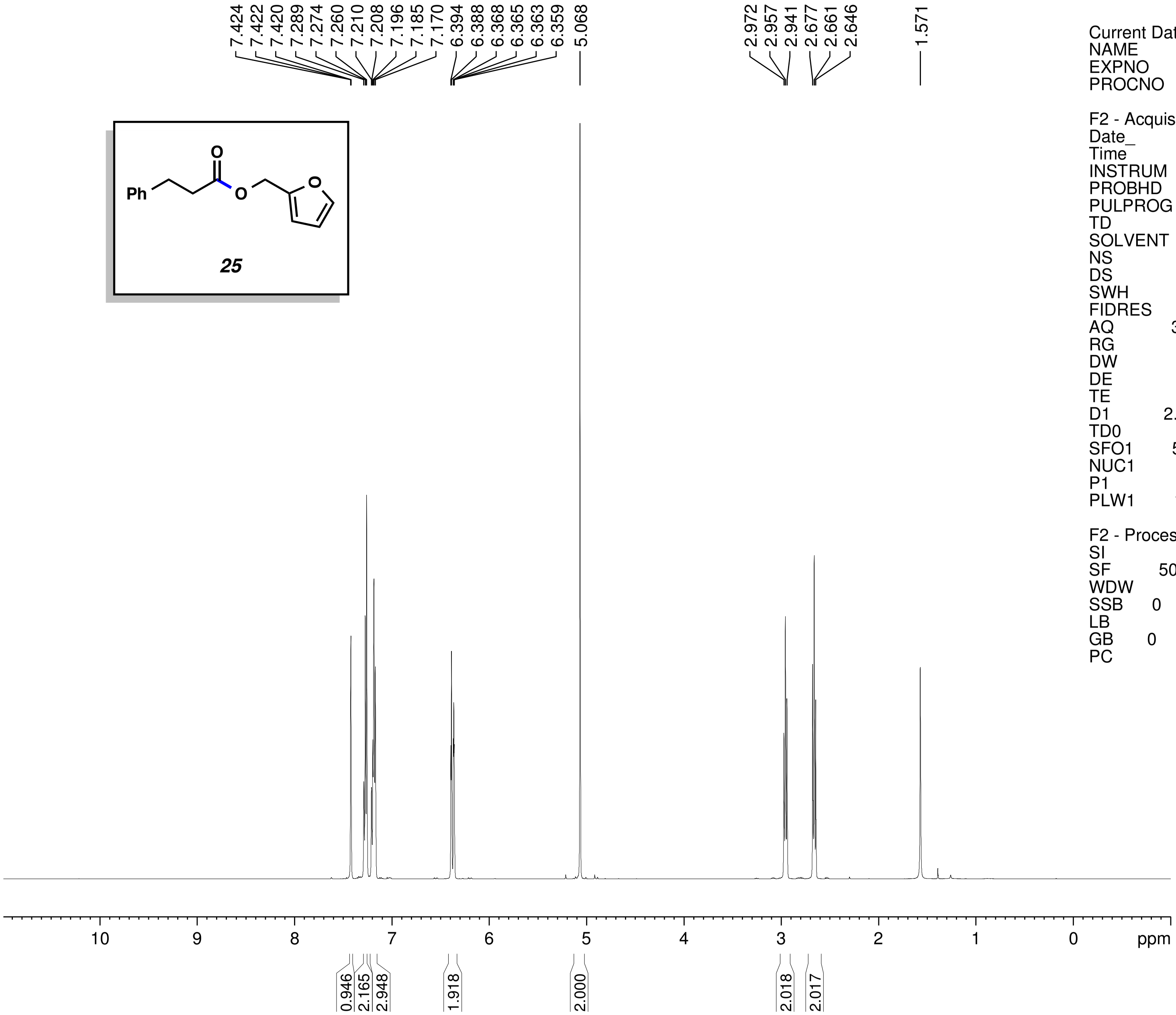
F2 - Acquisition Parameters
Date_ 20160411
Time 14.08 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

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



24

```
Current Data Parameters
NAME          LH-10-40
EXPNO              670
PROCNO               1

F2 - Acquisition Parameters
Date_          20160310
Time              19.13
INSTRUM           av400
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                52882
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           8012.820 Hz
FIDRES        0.151523 Hz
AQ           3.2998369 sec
RG                83.63
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
```

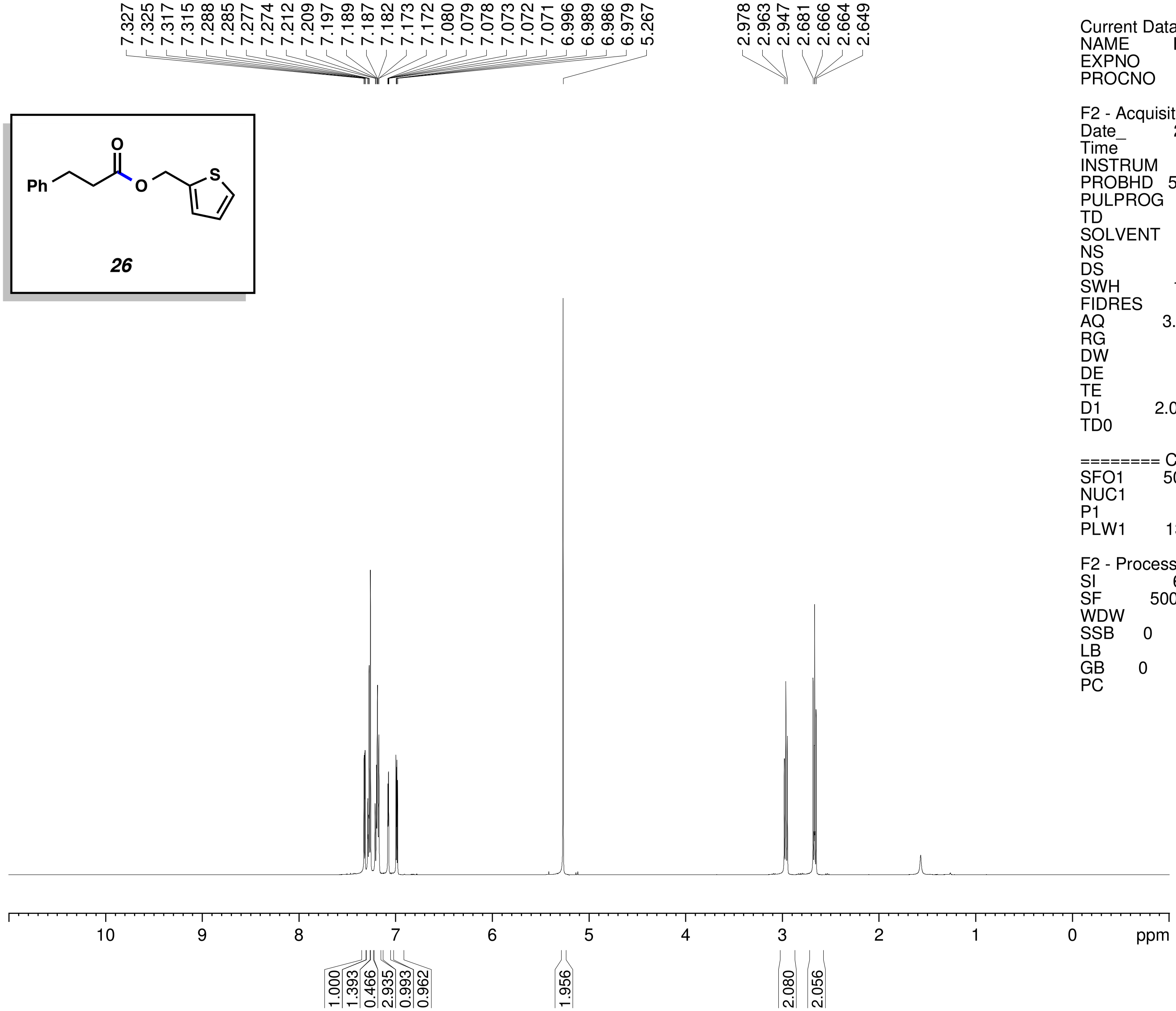


25

Current Data Parameters
NAME LH-10-81A
EXPNO 1
PROCNO 1

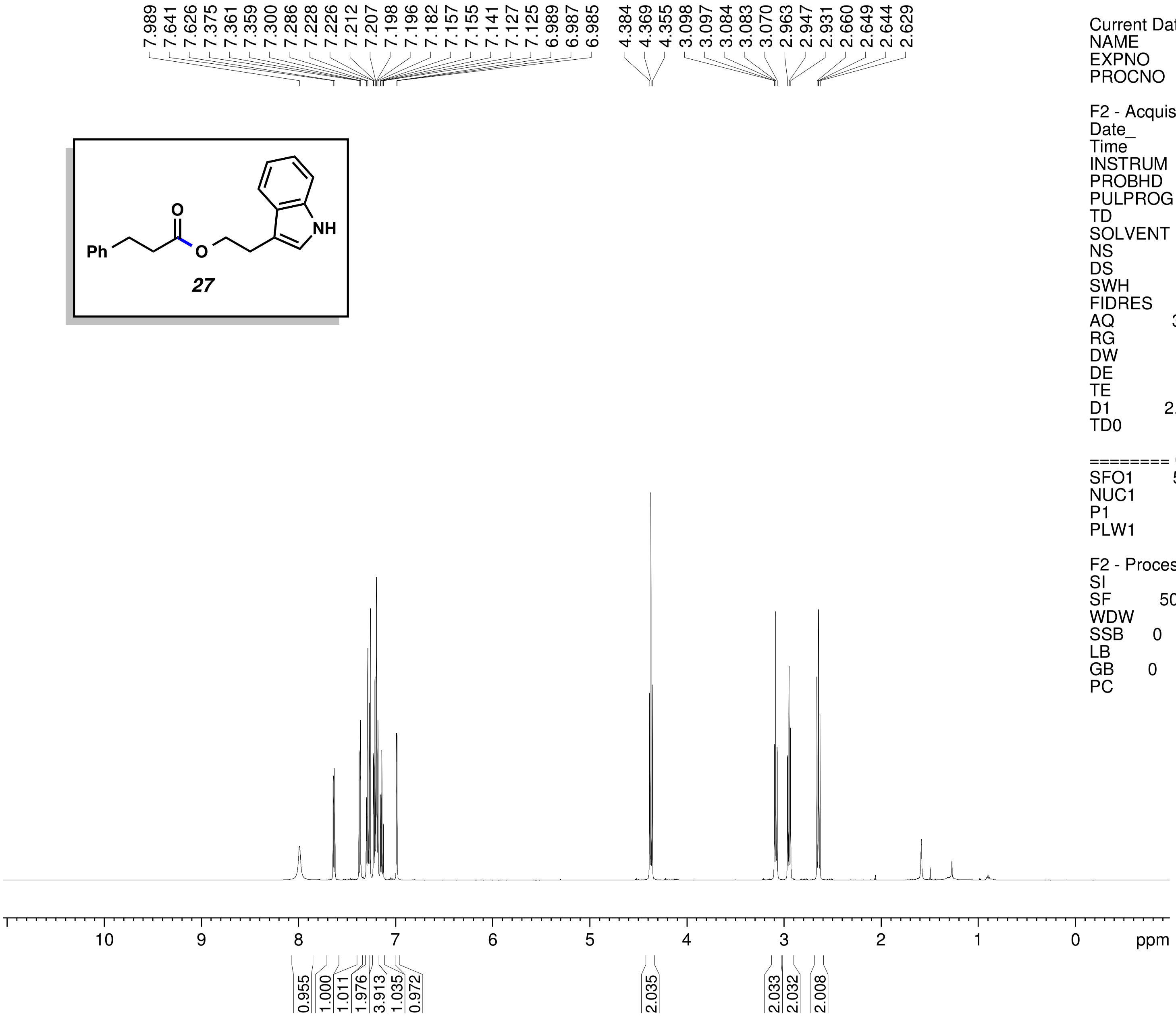
F2 - Acquisition Parameters
Date_ 20160406
Time 10.30 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 23
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 19.06
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

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



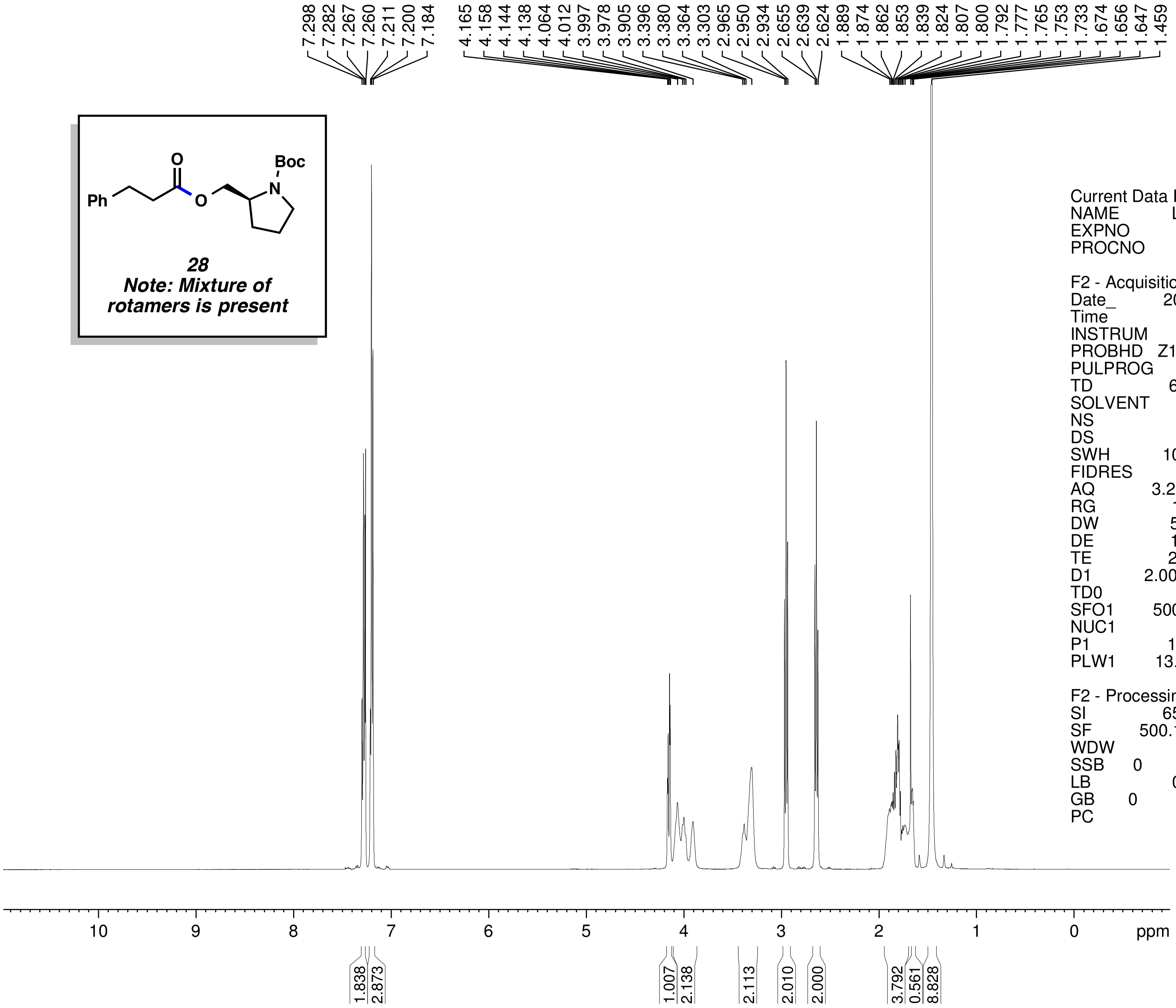
26

```
Current Data Parameters
NAME          ELB-5-273p
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_          20160503
Time              16.37
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     500.1330008 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
```



27

```
Current Data Parameters
NAME          ELB-5-294p
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20160425
Time              11.07
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       500.1330008 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
```

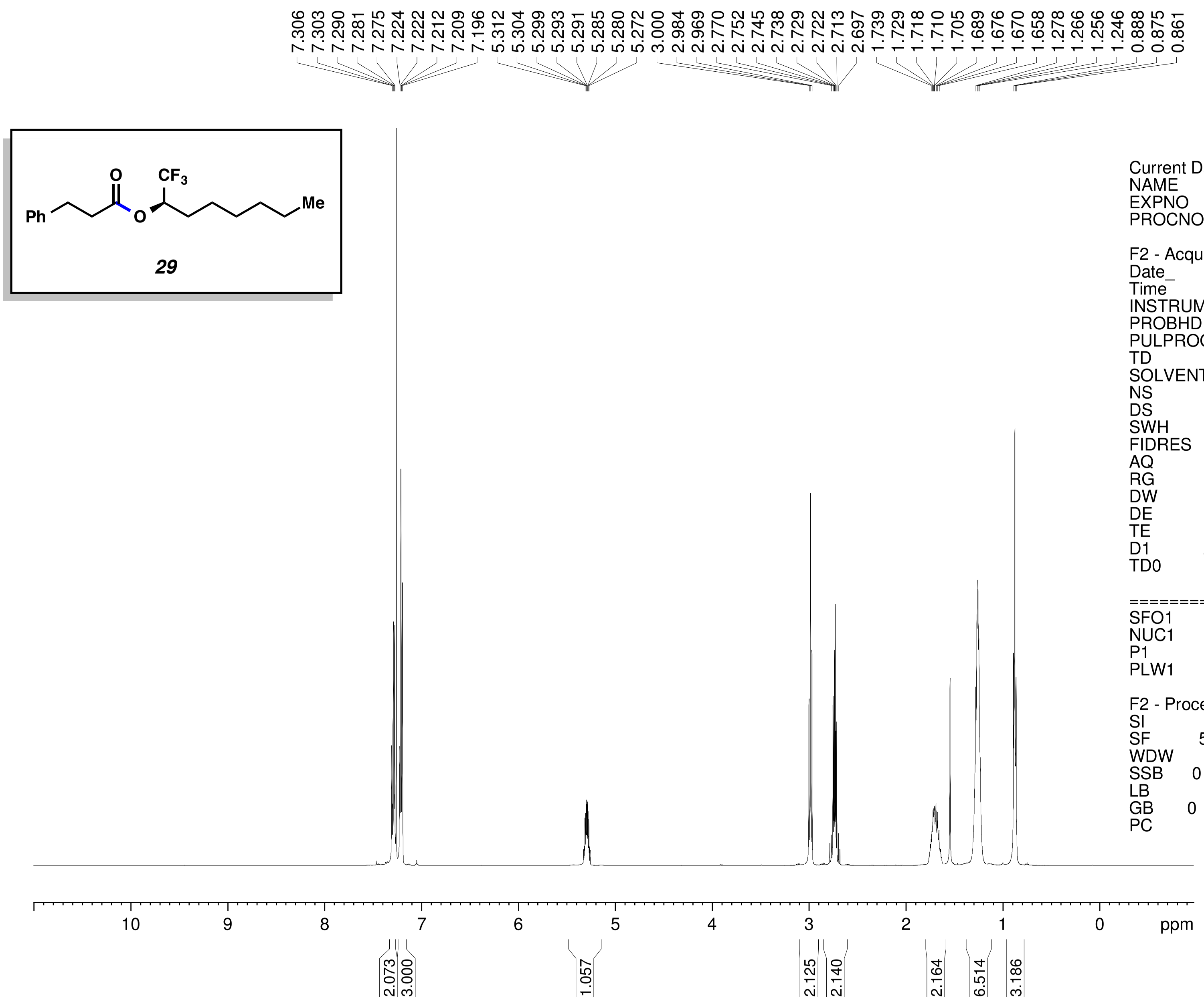


28
Note: Mixture of rotamers is present

Current Data Parameters
NAME LH-10-78
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160406
Time 10.04 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 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



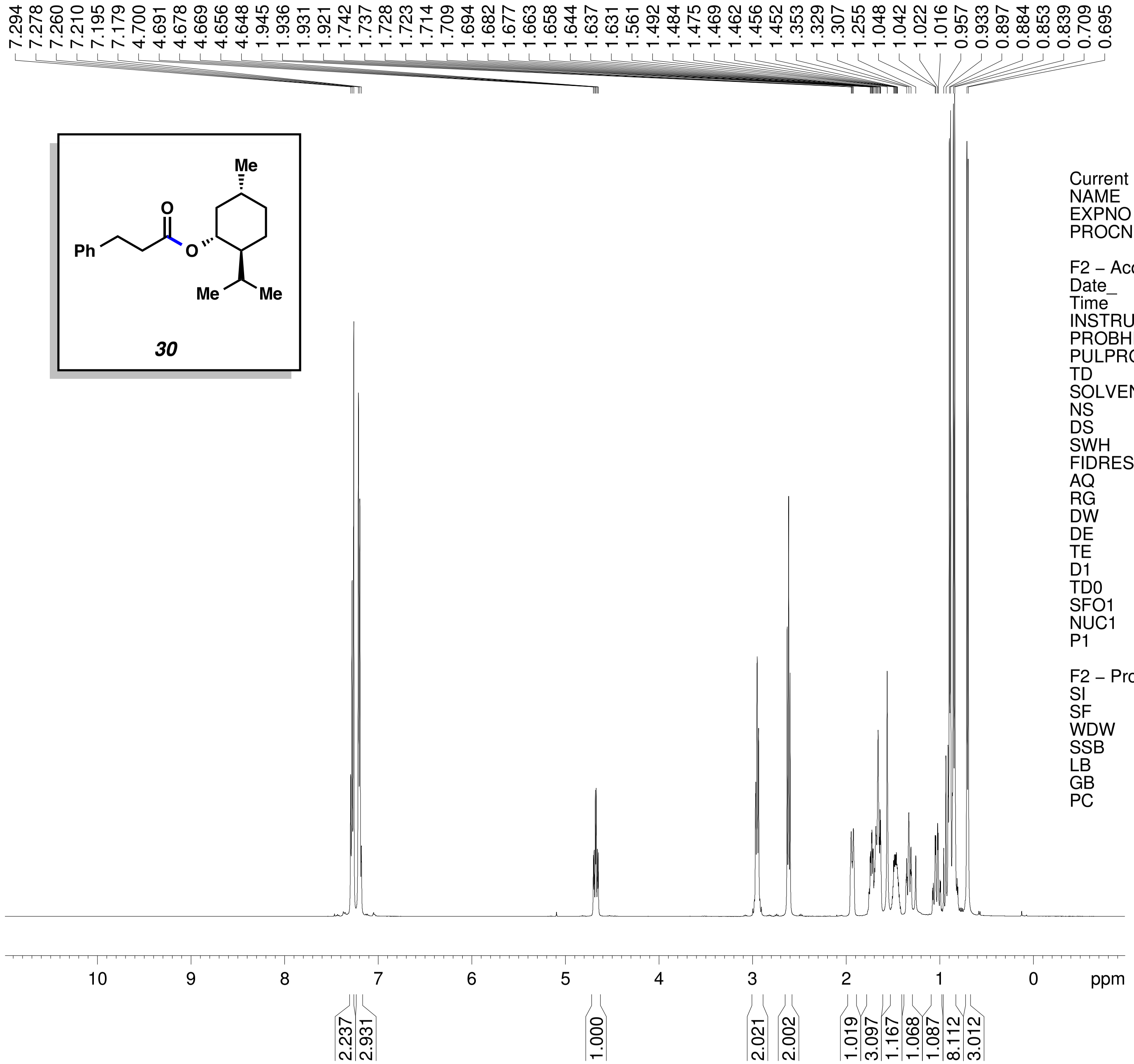
29

Current Data Parameters
NAME ELB-5-271p
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160412
Time 15.39
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 21.37
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 =====
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

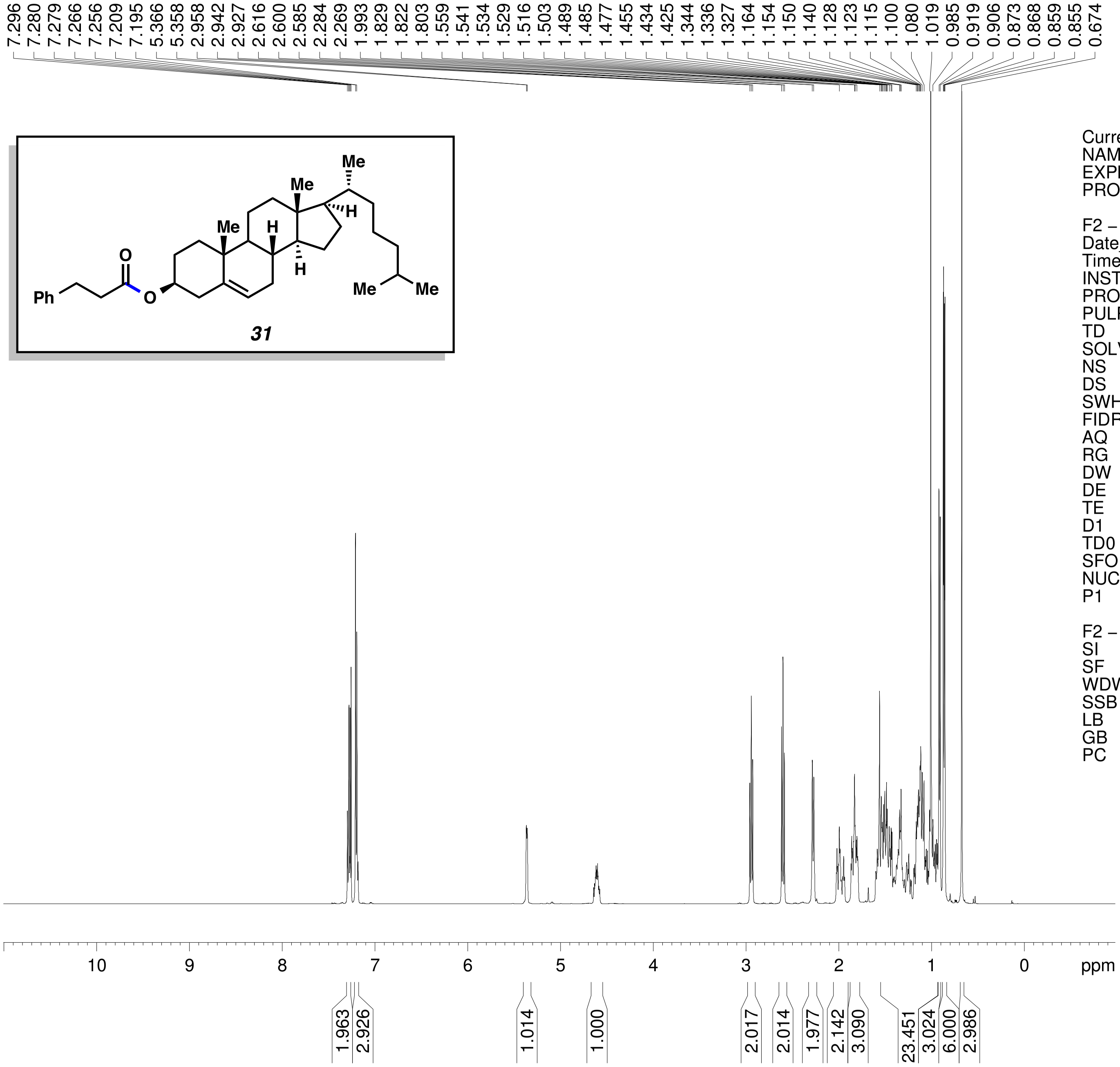
F2 - Processing parameters
SI 65536
SF 500.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



30

```
Current Data Parameters
NAME           LH-10-62
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160324
Time              19.21 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   15
DS                    0
SWH          10000.000 Hz
FIDRES         0.305176 Hz
AQ            3.2768500 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

F2 - Processing parameters
SI               131072
SF          500.1300123 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

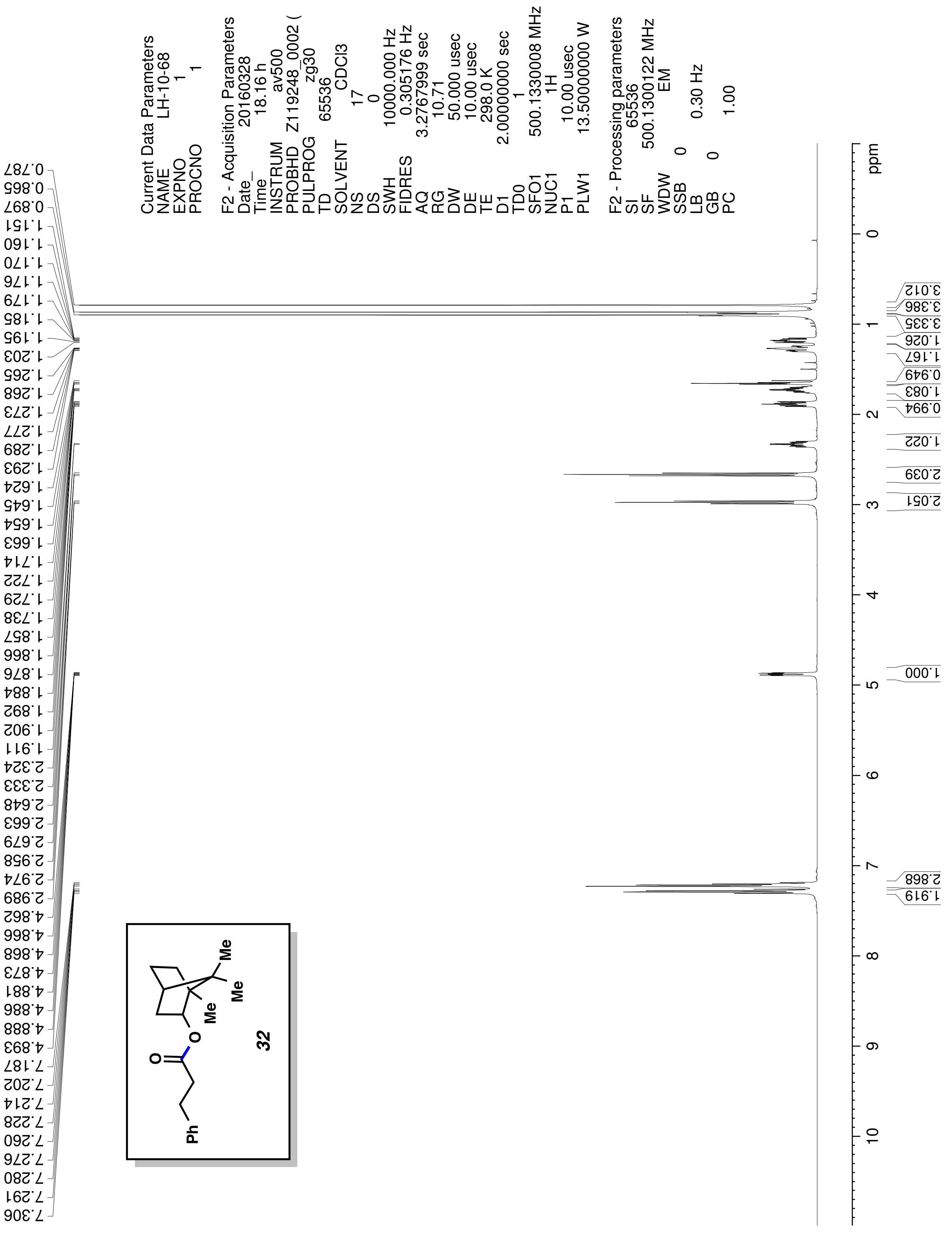
31



Current Data Parameters
NAME LH–10–64
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20160324
Time 19.37 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 18
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2768500 sec
RG 30.07
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

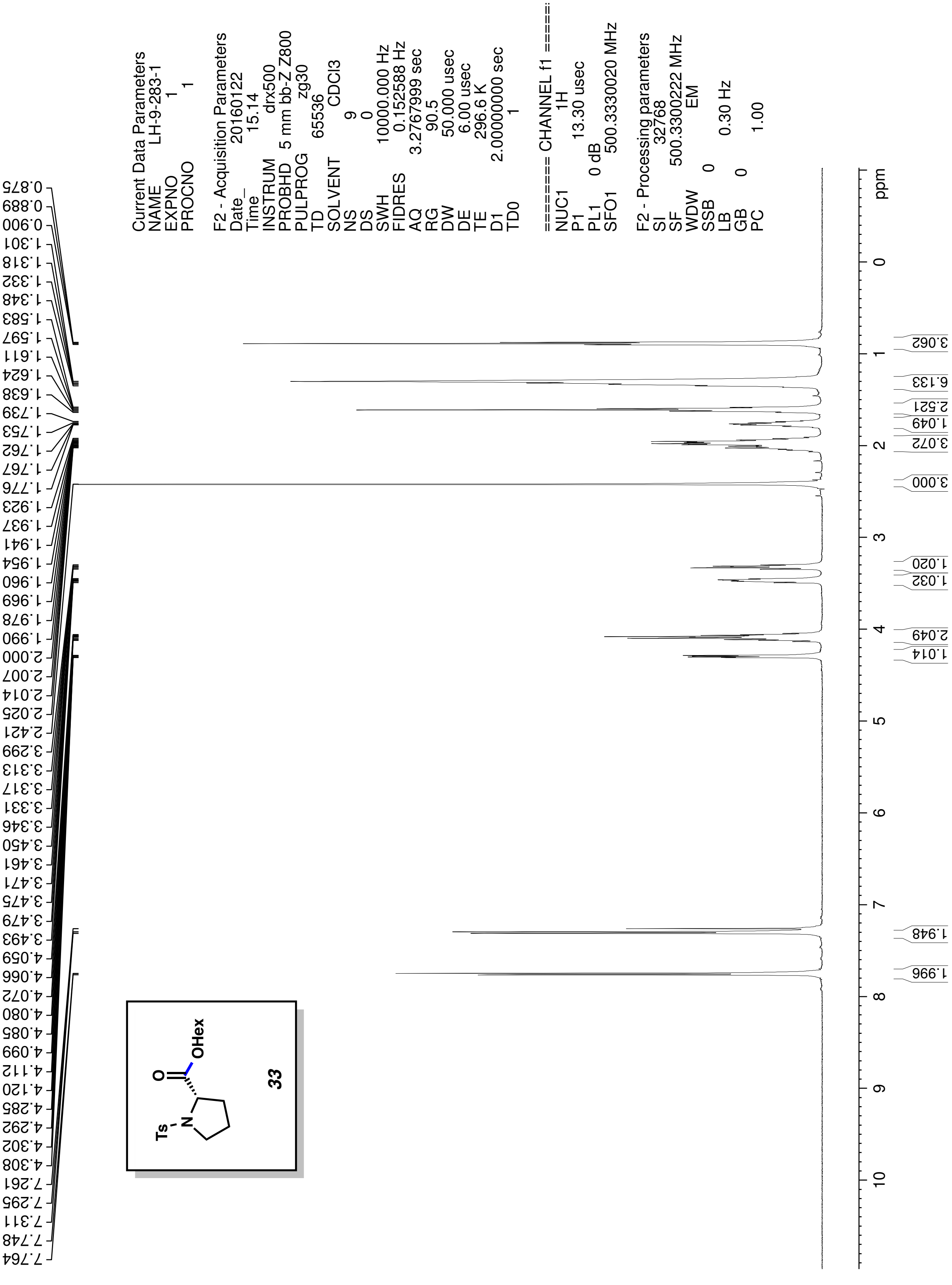
F2 – Processing parameters
SI 65536
SF 500.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

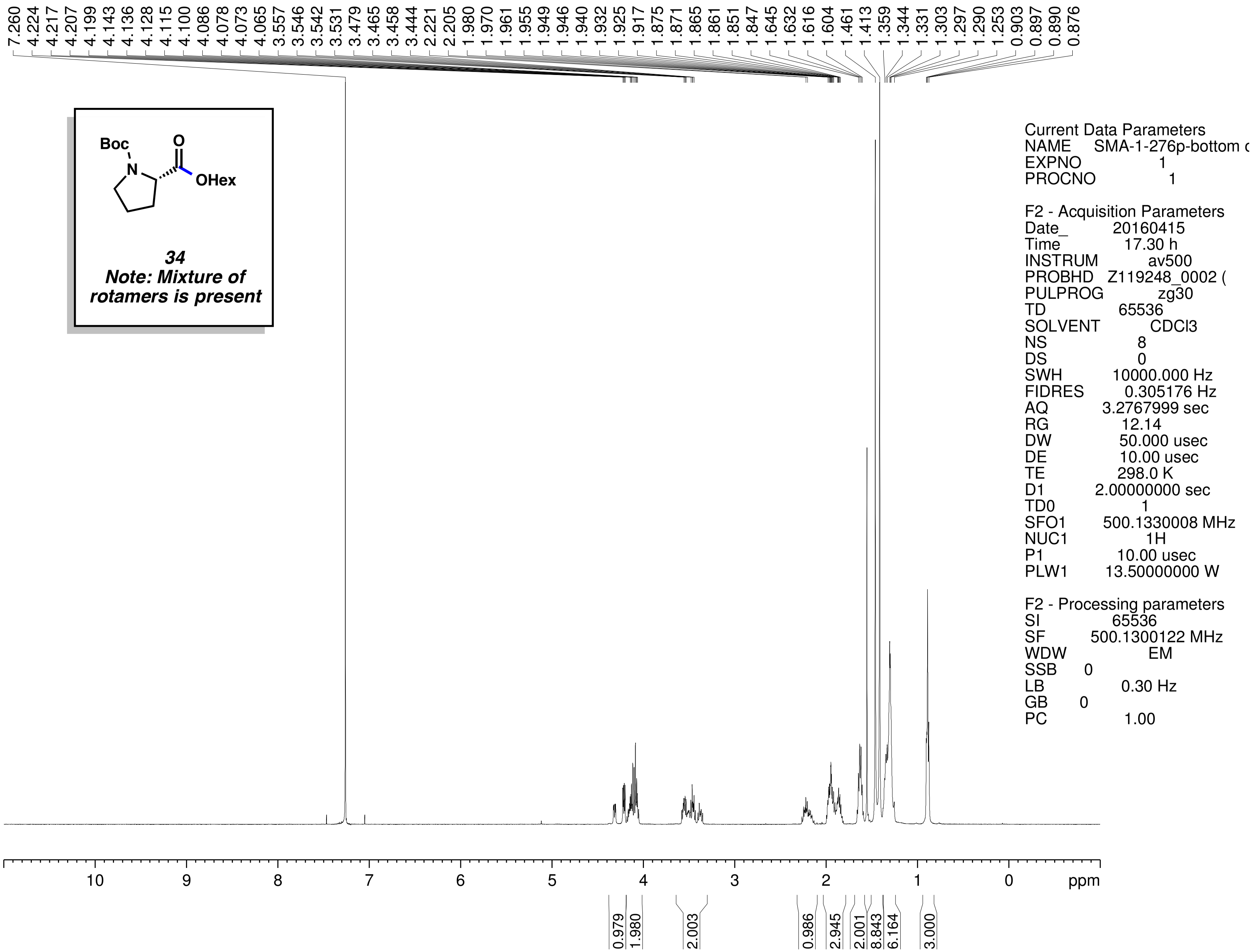


Current Data Parameters
NAME LH-10-68
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160328
Time 18.16 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 17
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 10.71
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


0.875
0.889
0.900
1.301
1.318
1.332
1.348
1.583
1.597
1.611
1.624
1.638
1.739
1.753
1.762
1.767
1.776
1.923
1.937
1.941
1.954
1.960
1.969
1.978
1.990
2.000
2.007
2.014
2.025
2.421
3.299
3.313
3.317
3.331
3.346
3.450
3.461
3.471
3.475
3.479
3.493
4.059
4.066
4.072
4.080
4.085
4.099
4.112
4.120
4.285
4.292
4.302
4.308
7.261
7.295
7.311
7.748
7.764
Current Data Parameters
NAME LH-9-283-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160122
Time 15.14
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 90.5
DW 50.000 usec
DE 6.00 usec
TE 296.6 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 500.3300222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ts
N
O
OHex
33
3.062
6.133
2.521
1.049
3.072
3.000
1.020
1.032
2.049
1.014
1.948
1.996
ppm
0
1
2
3
4
5
6
7
8
9
10

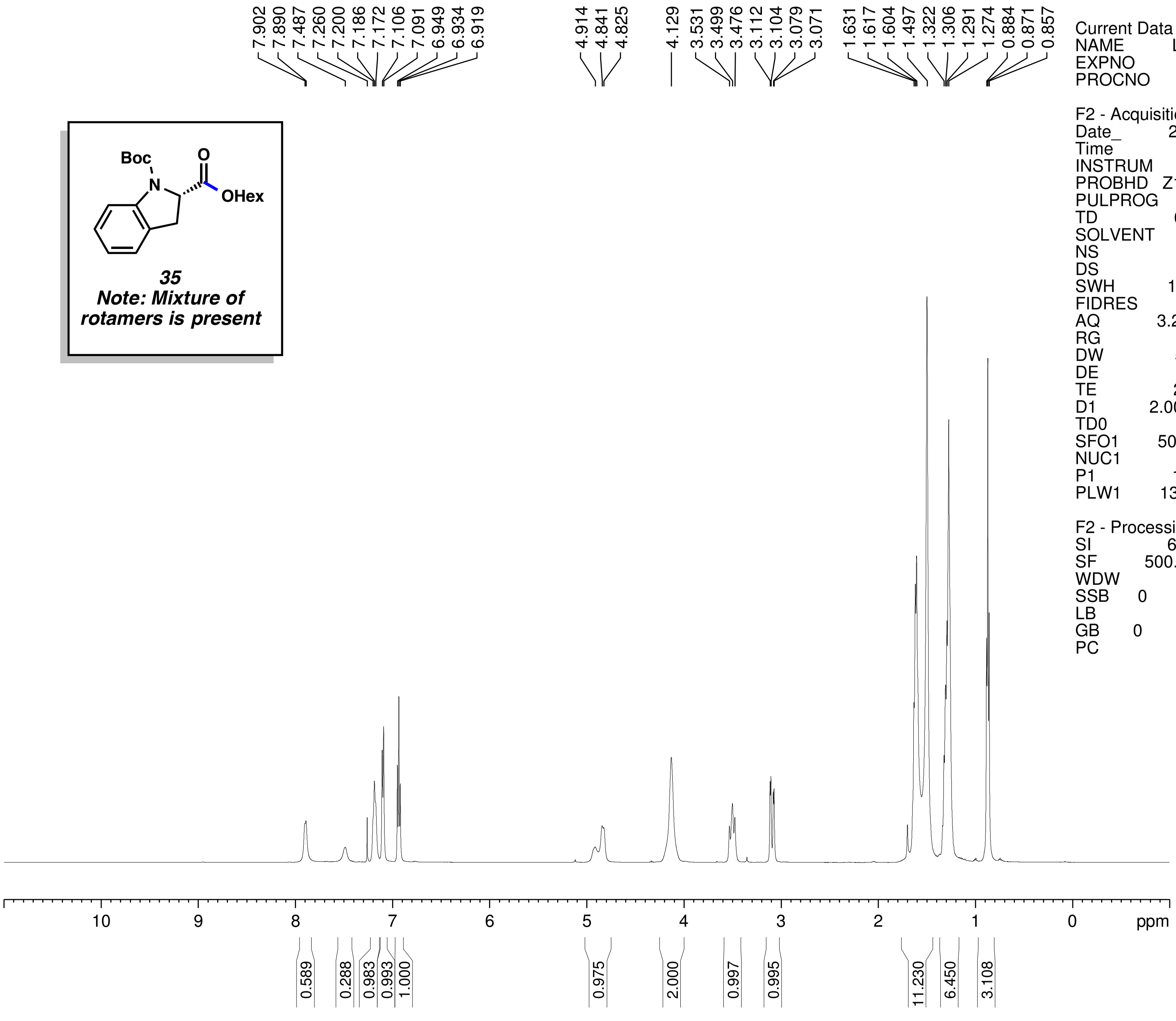


34

Note: Mixture of rotamers is present

```
Current Data Parameters
NAME     SMA-1-276p-bottom c
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20160415
Time            17.30 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
```

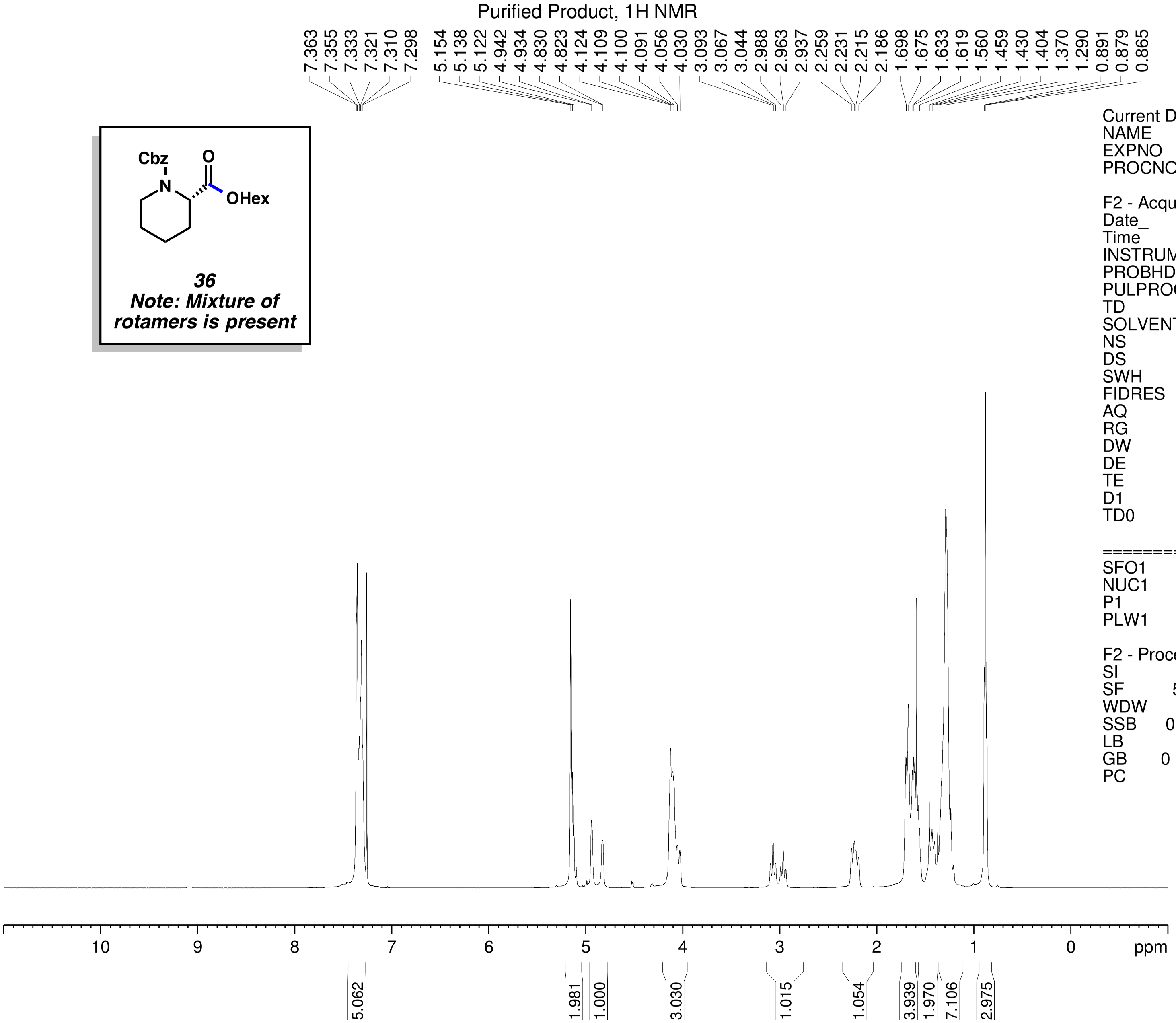
35
Note: Mixture of rotamers is present



```
Current Data Parameters
NAME          LH-10-149
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160527
Time            13.06 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   11
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          500.1300116 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

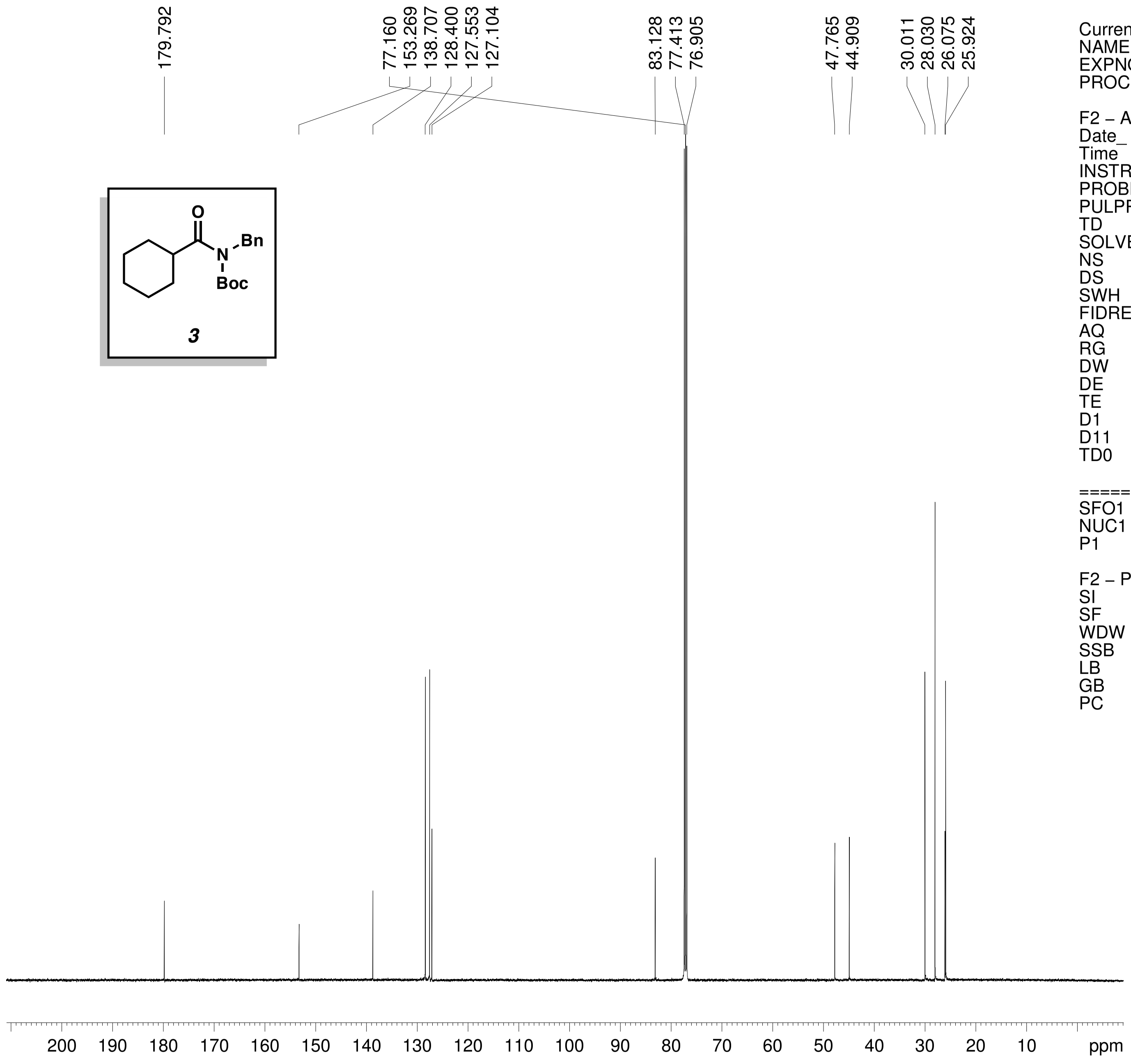
36

Note: Mixture of rotamers is present

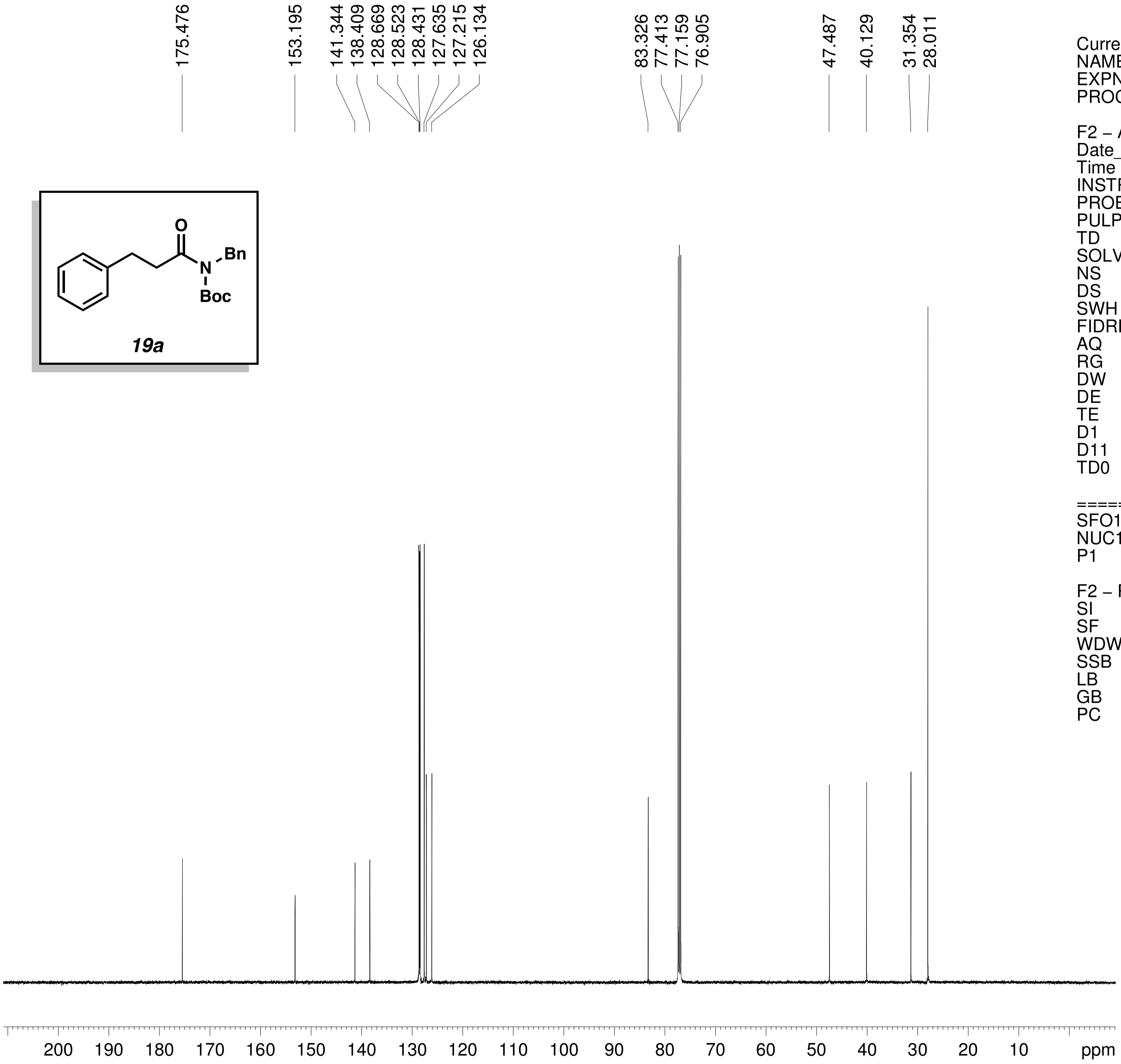

Purified Product, 1H NMR

```
Current Data Parameters
NAME        ELB-6-107p
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_        20160609
Time            13.58
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              21.37
DW             50.000 usec
DE             10.00 usec
TE             298.0 K
D1        2.00000000 sec
TD0                 1

======== CHANNEL f1 =====
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
```

^{13}C NMR Spectra

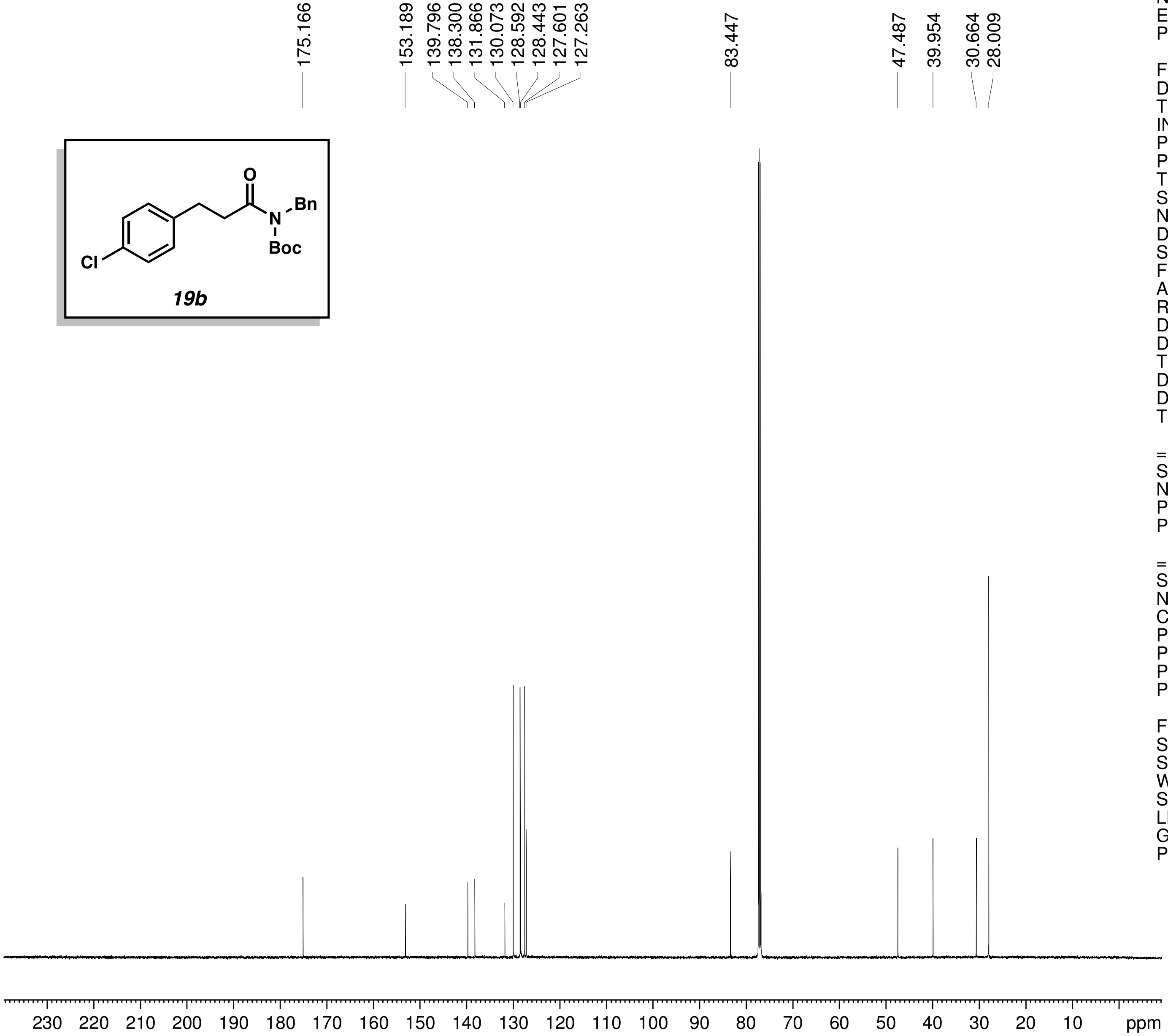


```
Current Data Parameters
NAME     LH-9-135-carbon
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_       20160127
Time           13.19
INSTRUM        av500
PROBHD   5 mm DCH 13C-1
PULPROG       zgpg30
TD             65536
SOLVENT        CDCl3
NS                98
DS                 2
SWH        31250.000 Hz
FIDRES      0.476837 Hz
AQ         1.0486259 sec
RG             14.67
DW            16.000 usec
DE             18.00 usec
TE             298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0                1

======== CHANNEL f1 ========
SFO1     125.7722511 MHz
NUC1             13C
P1              9.63 usec

F2 - Processing parameters
SI             131072
SF        125.7577726 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```


19a

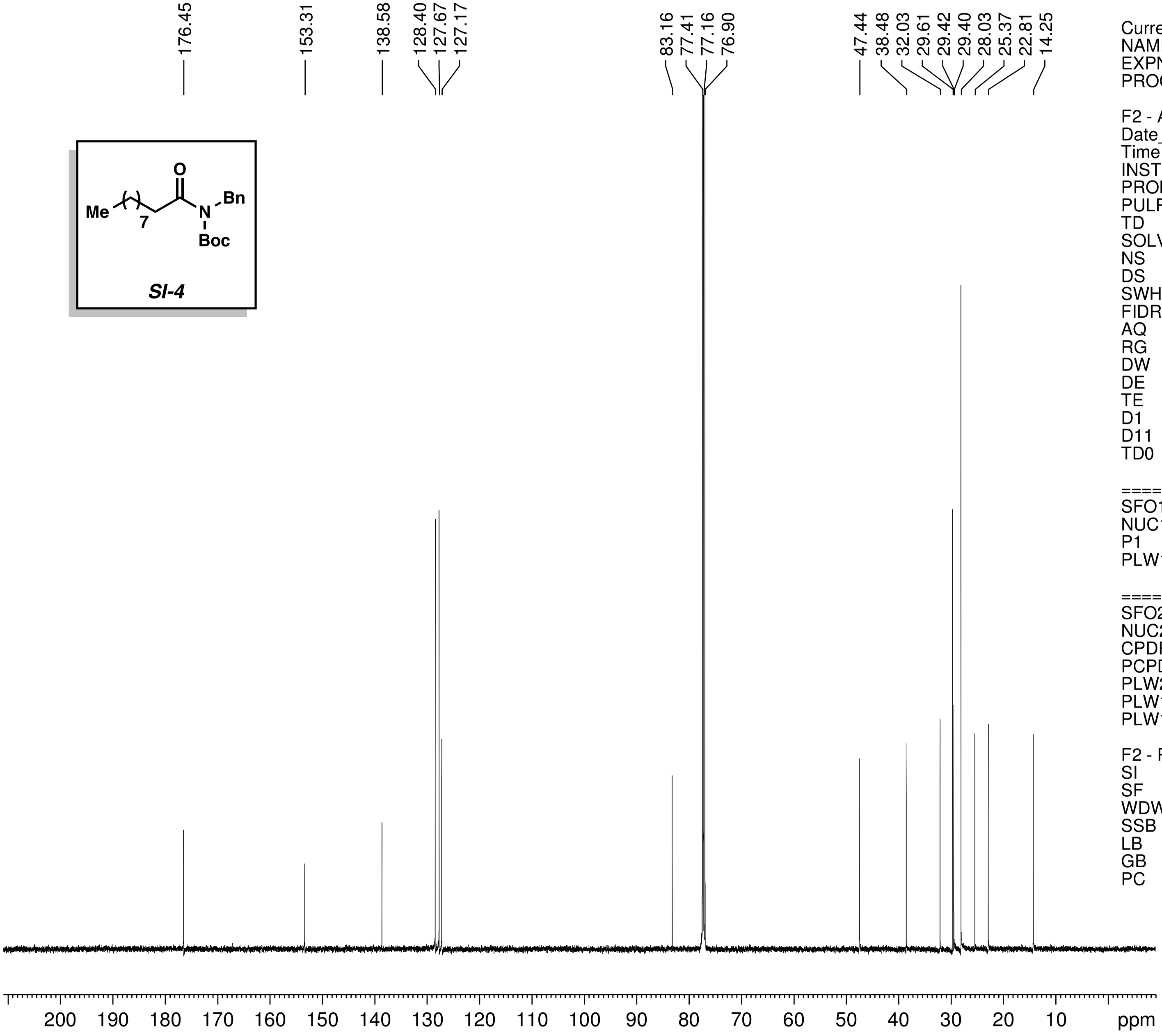
```
Current Data Parameters
NAME     LH-9-44-carbon
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_         20160127
Time             13.10
INSTRUM          av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                  67
DS                   2
SWH         31250.000 Hz
FIDRES       0.476837 Hz
AQ          1.0486259 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

======== CHANNEL f1 ========
SFO1       125.7722511 MHz
NUC1              13C
P1               9.63 usec

F2 - Processing parameters
SI             131072
SF        125.7577749 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

Purified Product, 13C NMR



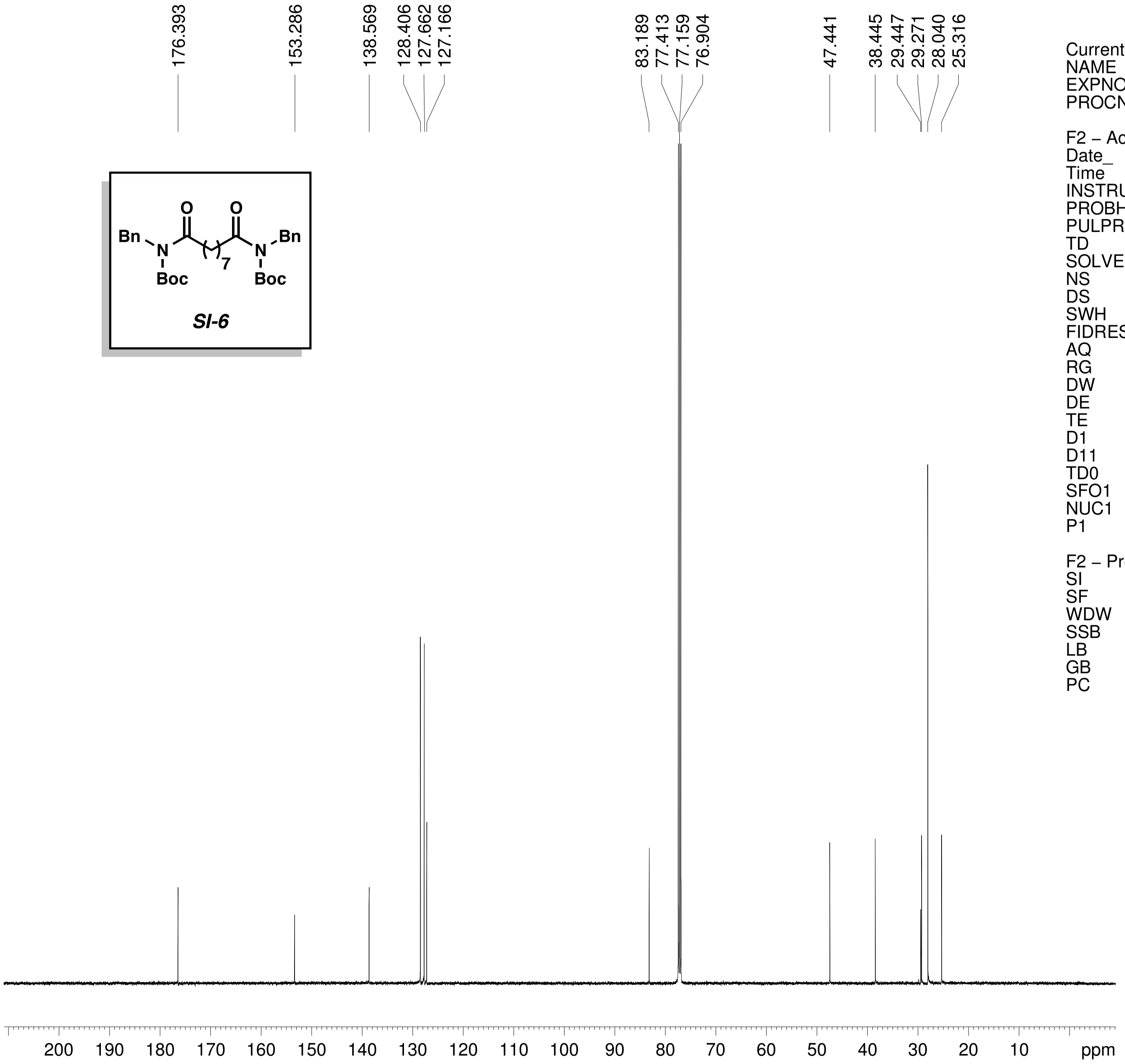
19b

```
Current Data Parameters
NAME        ELB-6-277p
EXPNO              4
PROCNO             1

F2 - Acquisition Parameters
Date_        20160918
Time            9.35
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD            65536
SOLVENT         CDCl3
NS                34
DS                 2
SWH         31250.000 Hz
FIDRES       0.476837 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

======== CHANNEL f1 =====
SFO1       125.7722511 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         125.7577743 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```



SI-4

```
Current Data Parameters
NAME     LH-9-66-carbon
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20160129
Time            13.12
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD             65536
SOLVENT          CDCl3
NS                82
DS                 2
SWH        31250.000 Hz
FIDRES      0.476837 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

======== CHANNEL f1 =====
SFO1      125.7722511 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
```

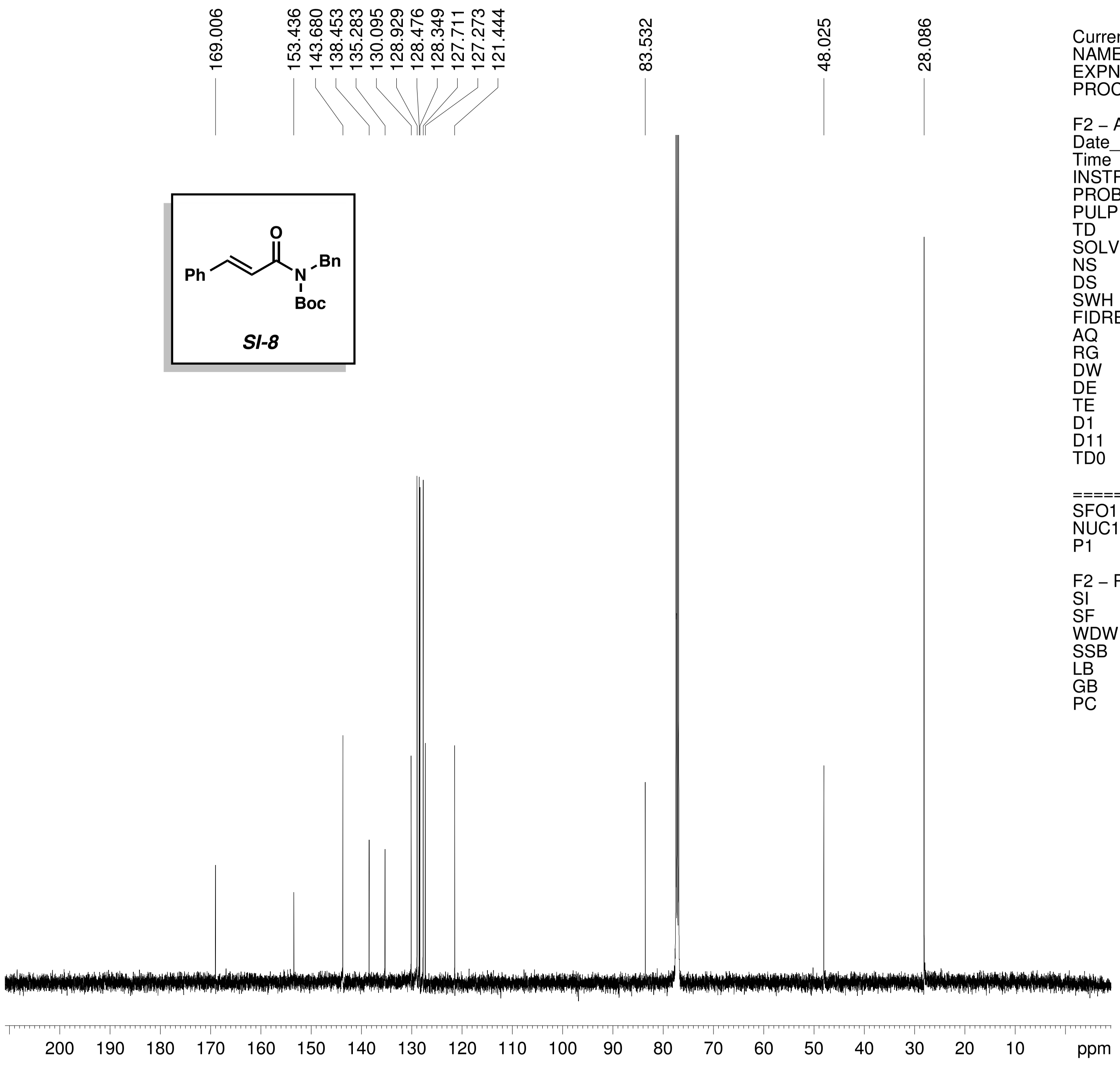


SI-6

Current Data Parameters
NAME LH–9–255
EXPNO 4
PROCNO 1

F2 – Acquisition Parameters
Date_ 20160321
Time 19.19 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 127
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0486259 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 9.63 usec

F2 – Processing parameters
SI 131072
SF 125.7577733 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



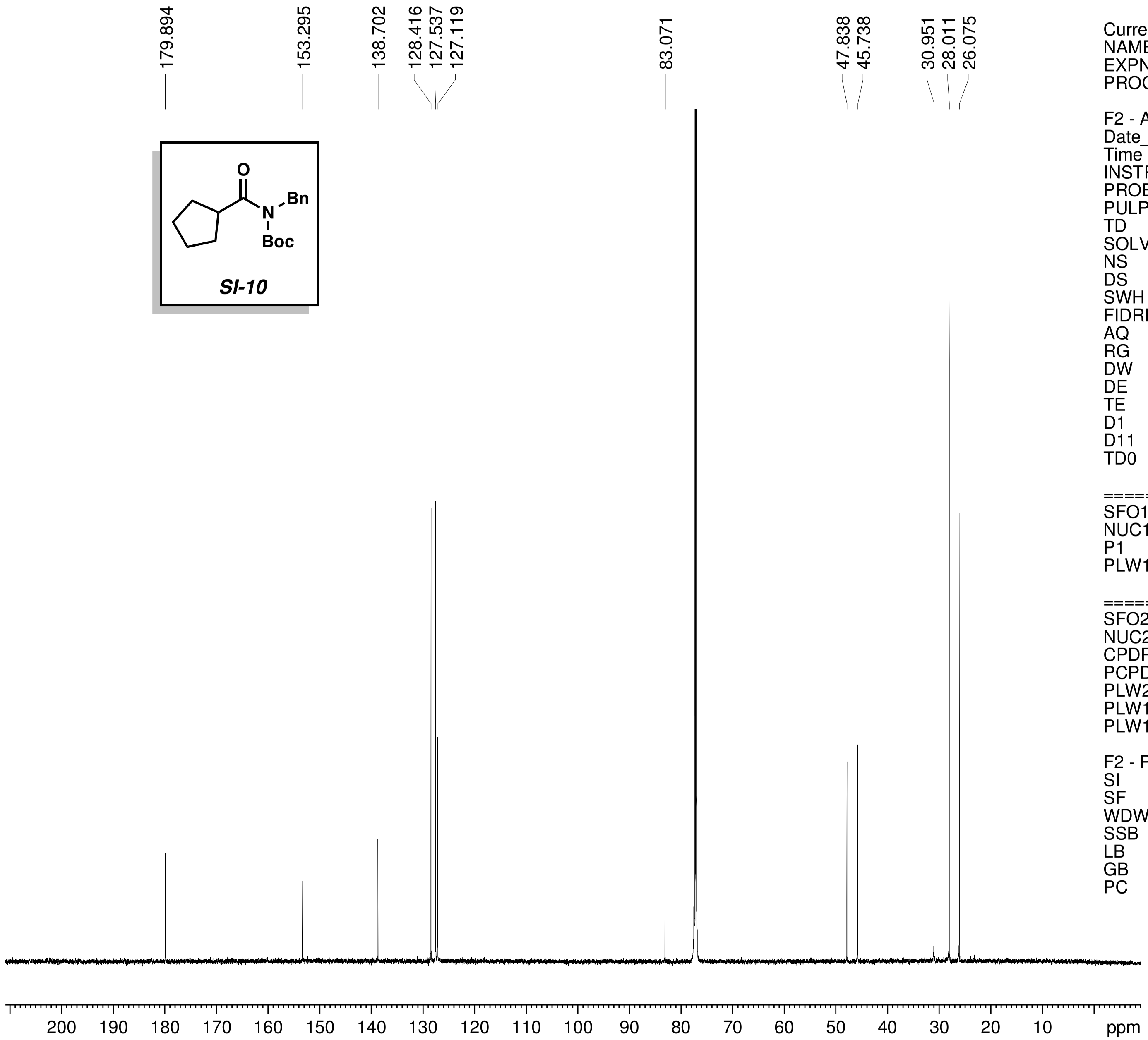
SI-8

Current Data Parameters
NAME ELB-5-102p
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160120
Time 16.18
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 54
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 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

======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec

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



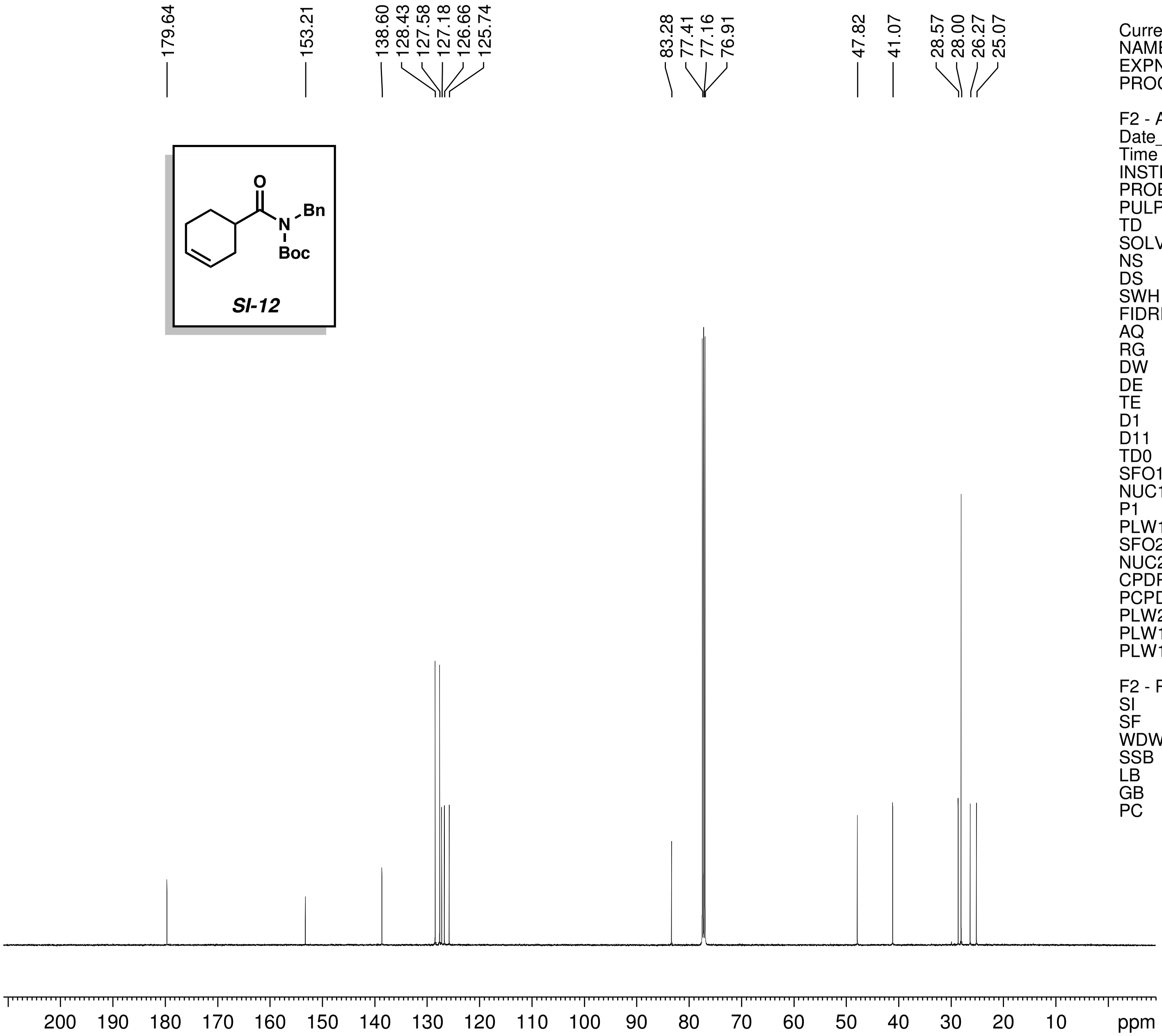
Current Data Parameters
NAME ELB-5-117p
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160412
Time 11.28
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 52
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 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

======== CHANNEL f1 =====
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

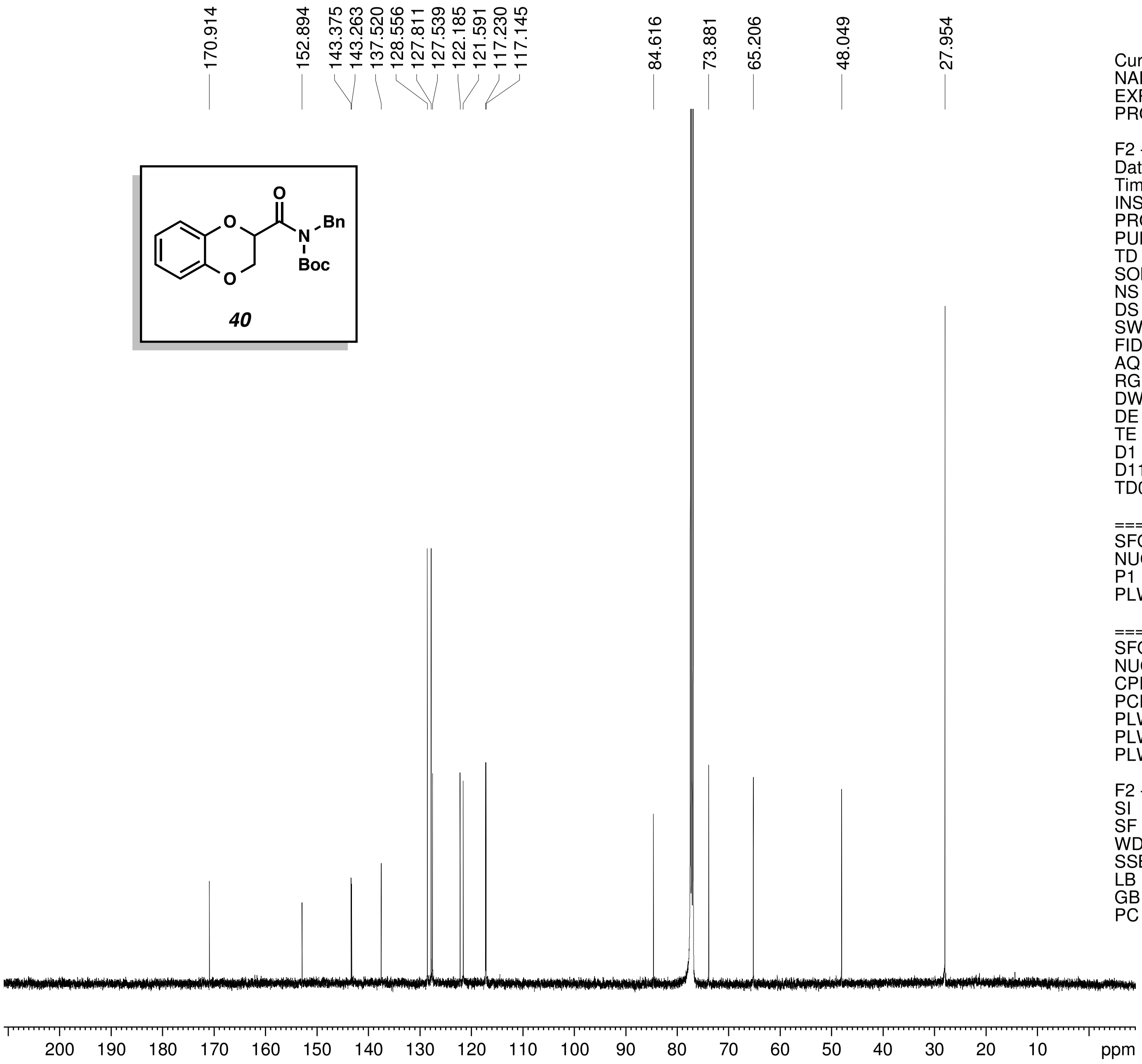
======== CHANNEL f2 =====
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

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



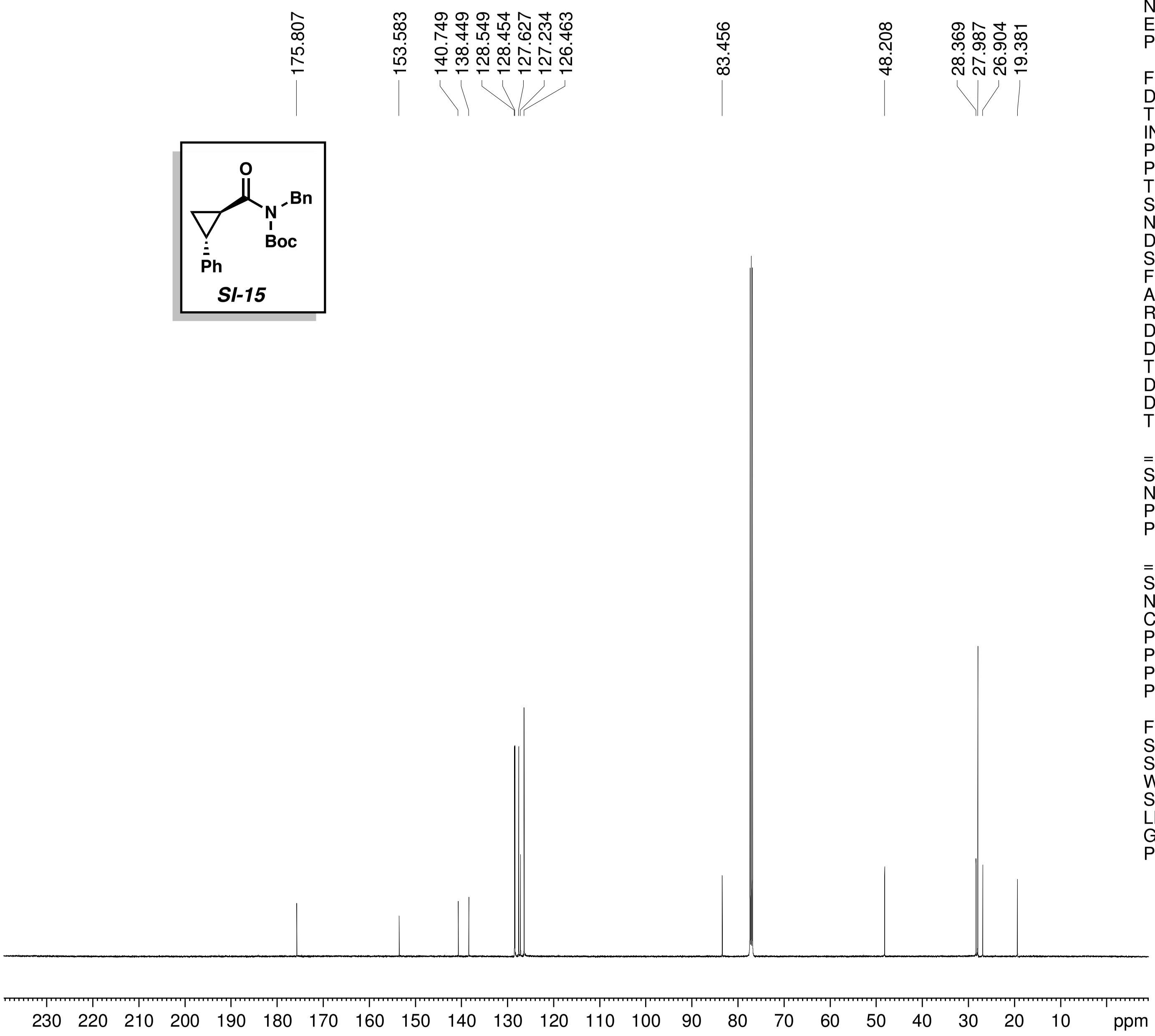
SI-12

```
Current Data Parameters
NAME        LH-10-114
EXPNO               4
PROCNO              1

F2 - Acquisition Parameters
Date_         20160506
Time           17.15 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD            65536
SOLVENT          CDCl3
NS               111
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               9.63 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      0.13500001 W

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



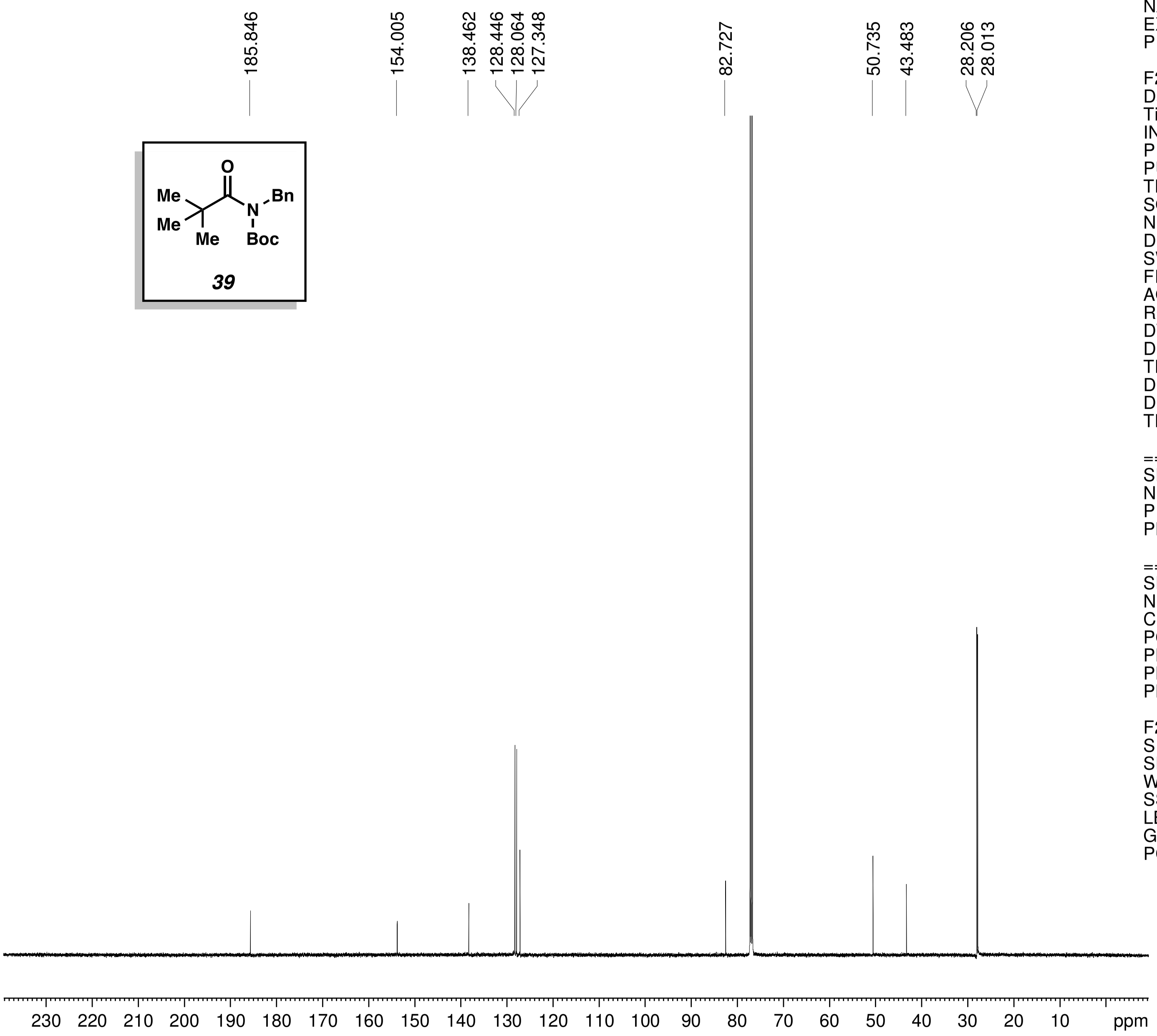
```
Current Data Parameters
NAME        ELB-5-106p
EXPNO              4
PROCNO             1

F2 - Acquisition Parameters
Date_         20160121
Time            11.14
INSTRUM          av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD             65536
SOLVENT          CDCl3
NS               104
DS                 2
SWH        31250.000 Hz
FIDRES      0.476837 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

======== CHANNEL f1 =====
SFO1      125.7722511 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       125.7577707 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```

Purified Product, 13C NMR

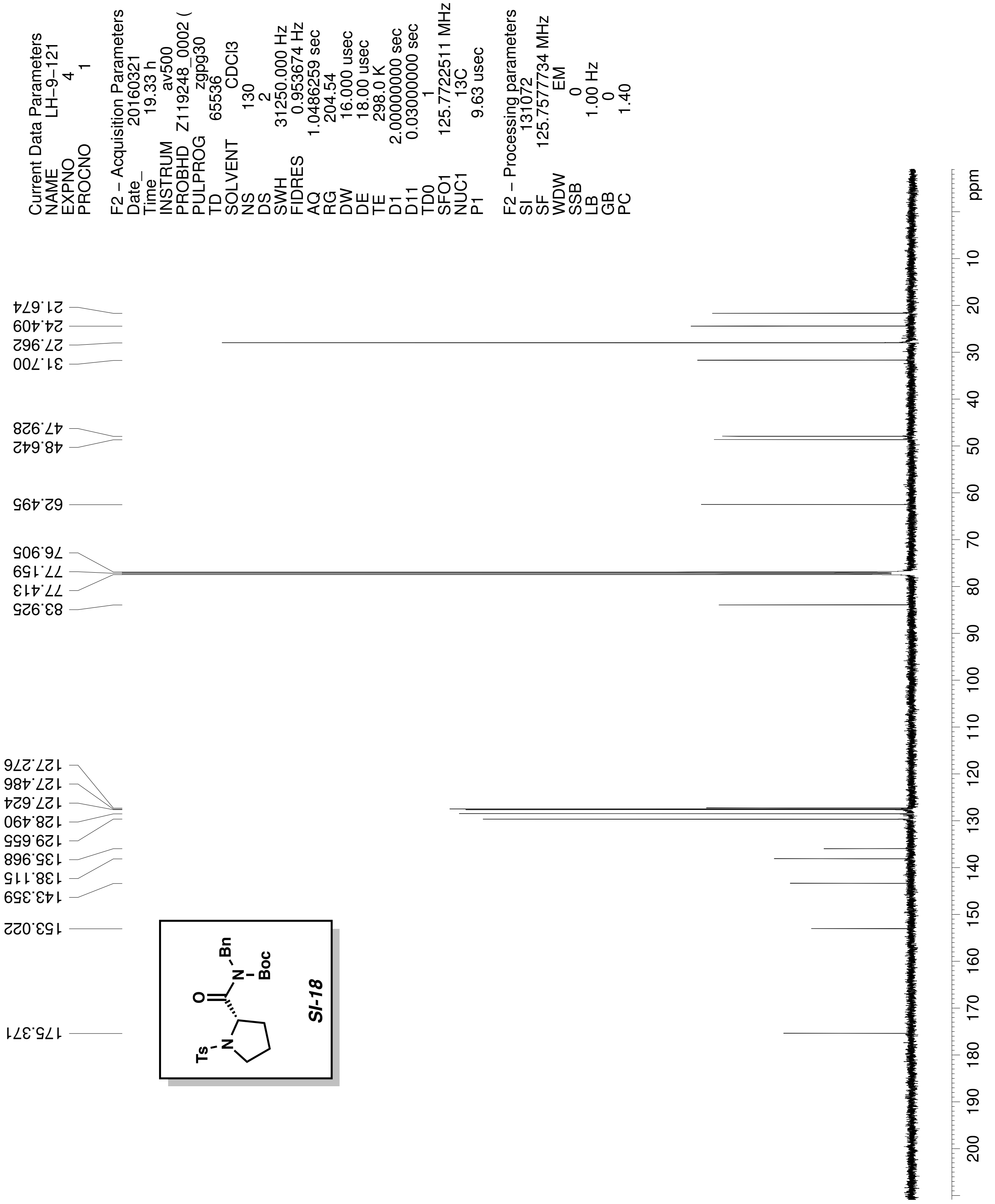


```
Current Data Parameters
NAME          ELB-6-127p
EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
Date_         20160615
Time            14.14
INSTRUM          av500
PROBHD   5 mm DCH 13C-1
PULPROG         zgpg30
TD             65536
SOLVENT          CDCl3
NS               108
DS                 2
SWH         31250.000 Hz
FIDRES       0.476837 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

======== CHANNEL f1 =====
SFO1      125.7722511 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        125.7577736 MHz
WDW              EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```

Purified Product, 13C NMR

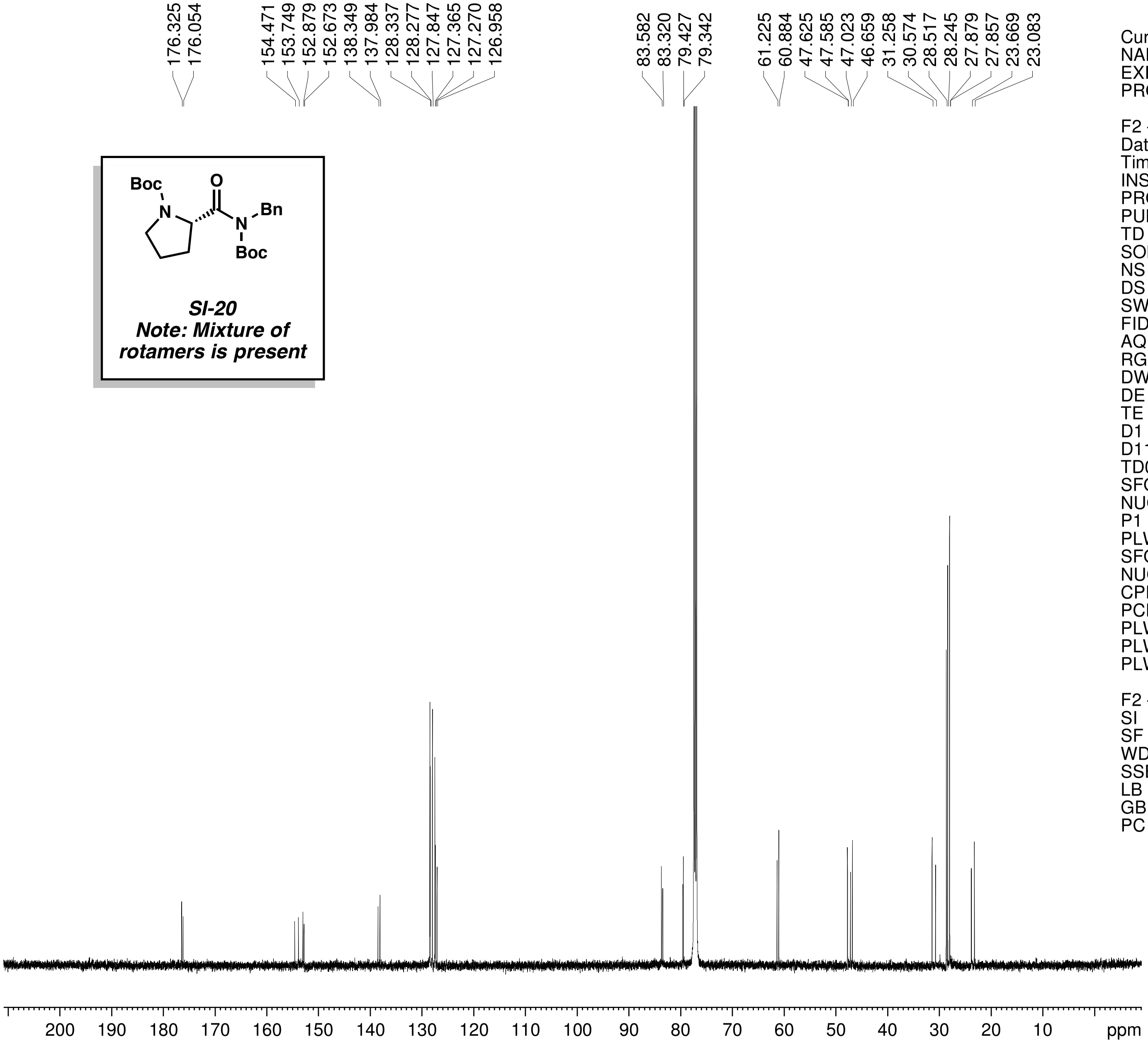


```
Current Data Parameters
NAME        ELB-6-089p
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_         20160607
Time            14.38
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG         zgpg30
TD             65536
SOLVENT          CDCl3
NS                 38
DS                  2
SWH          31250.000 Hz
FIDRES        0.476837 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

======== CHANNEL f1 =====
SFO1       125.7722511 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               1.00
```


Current Data Parameters
NAME LH-9-121
EXPNO 4
PROCNO 1
F2 – Acquisition Parameters
Date_ 20160321
Time 19.33 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 130
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0486259 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 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577734 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.674
24.409
27.962
31.700
47.928
48.642
62.495
76.905
77.159
77.413
83.925
127.276
127.486
127.624
128.490
129.655
135.968
138.115
143.359
153.022
175.371
Ts
N
O
N
Bn
Boc
SI-18
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200

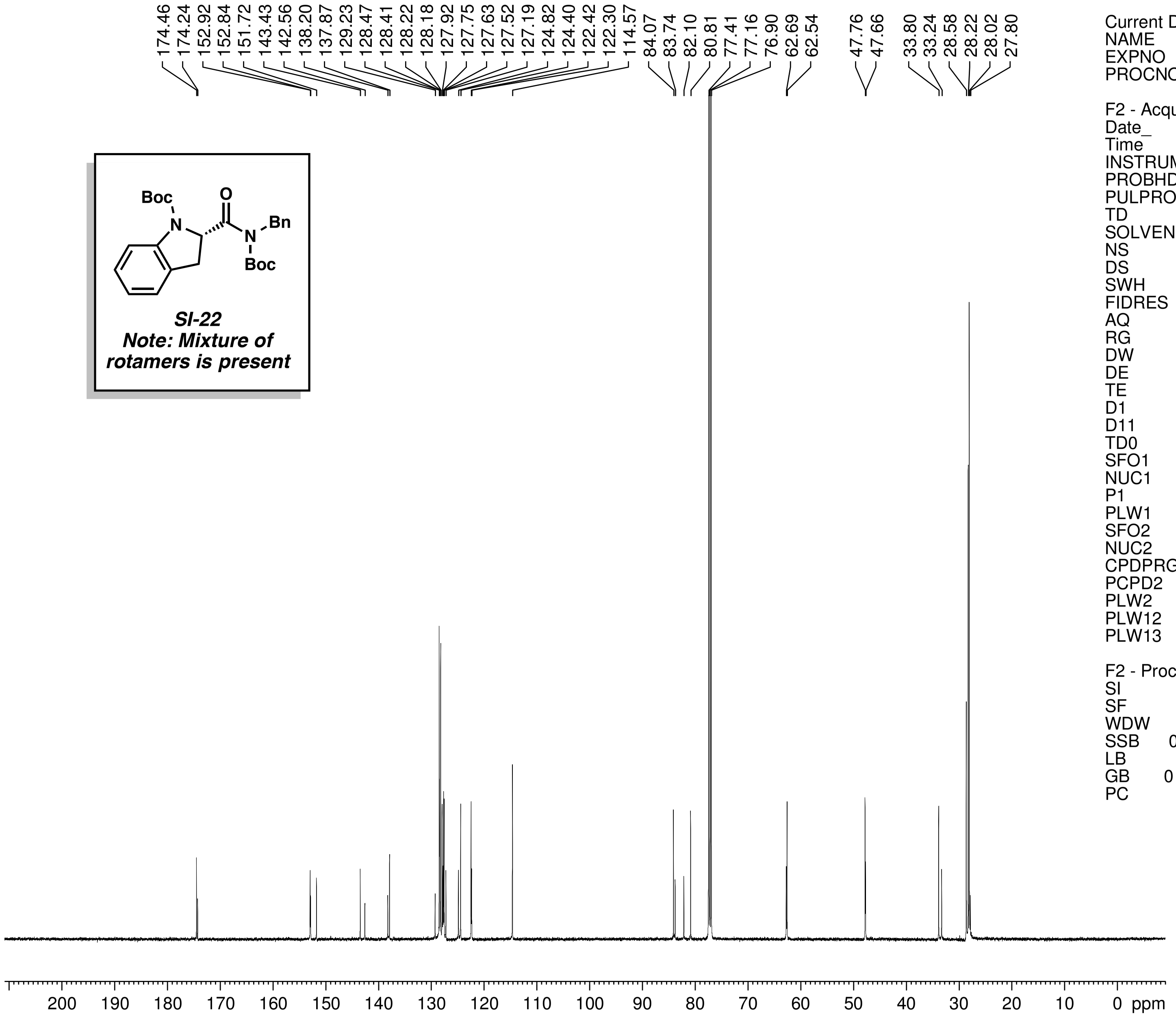
SI-20

Note: Mixture of rotamers is present

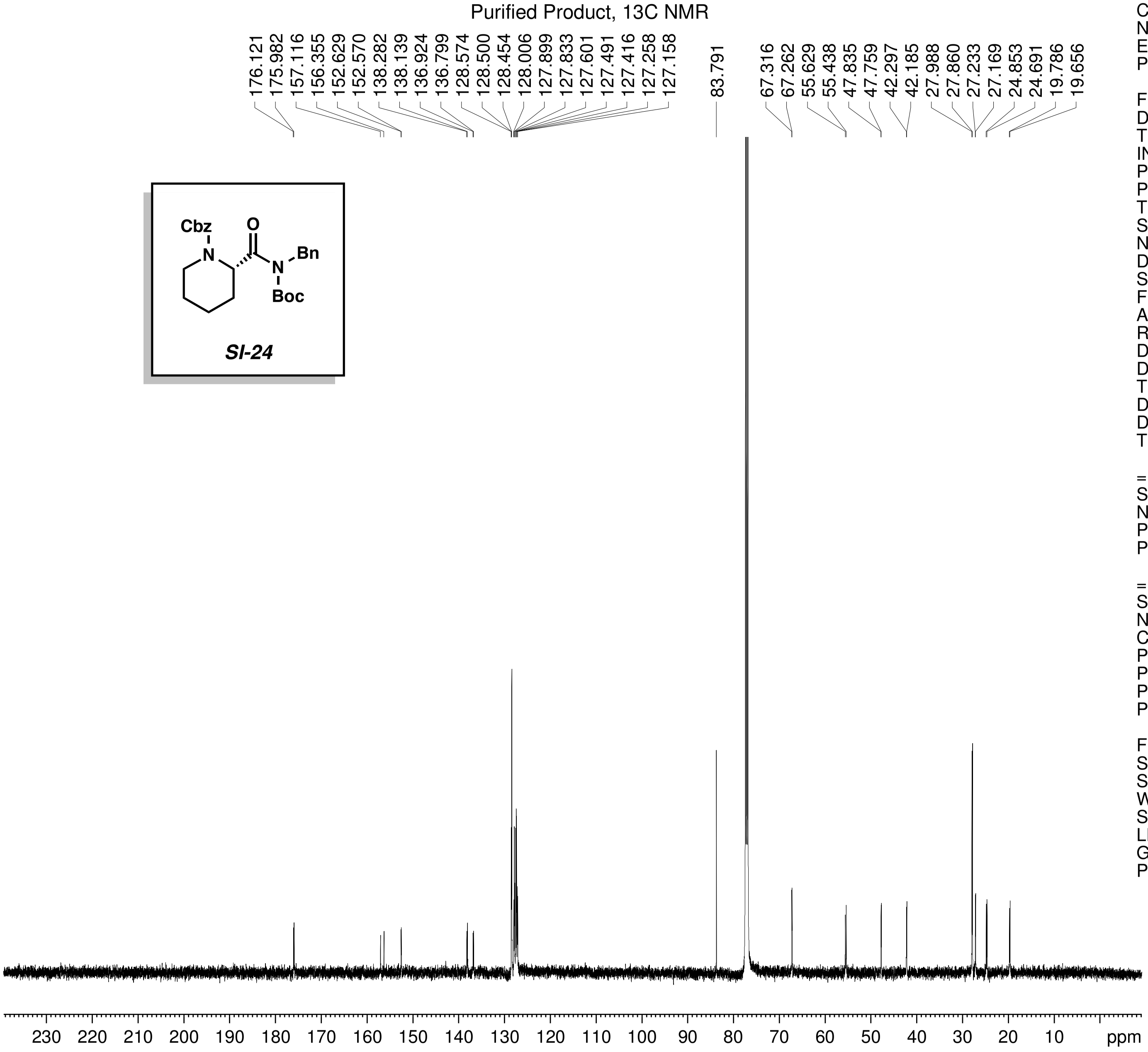


```
Current Data Parameters
NAME          SMA-1-273p
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160506
Time              8.39 h
INSTRUM            av500
PROBHD   Z119248_0002 (
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   434
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              9.63 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       125.7577717 MHz
WDW                   EM
SSB      0
LB               1.00 Hz
GB       0
PC                  1.40
```

SI-22
Note: Mixture of rotamers is present

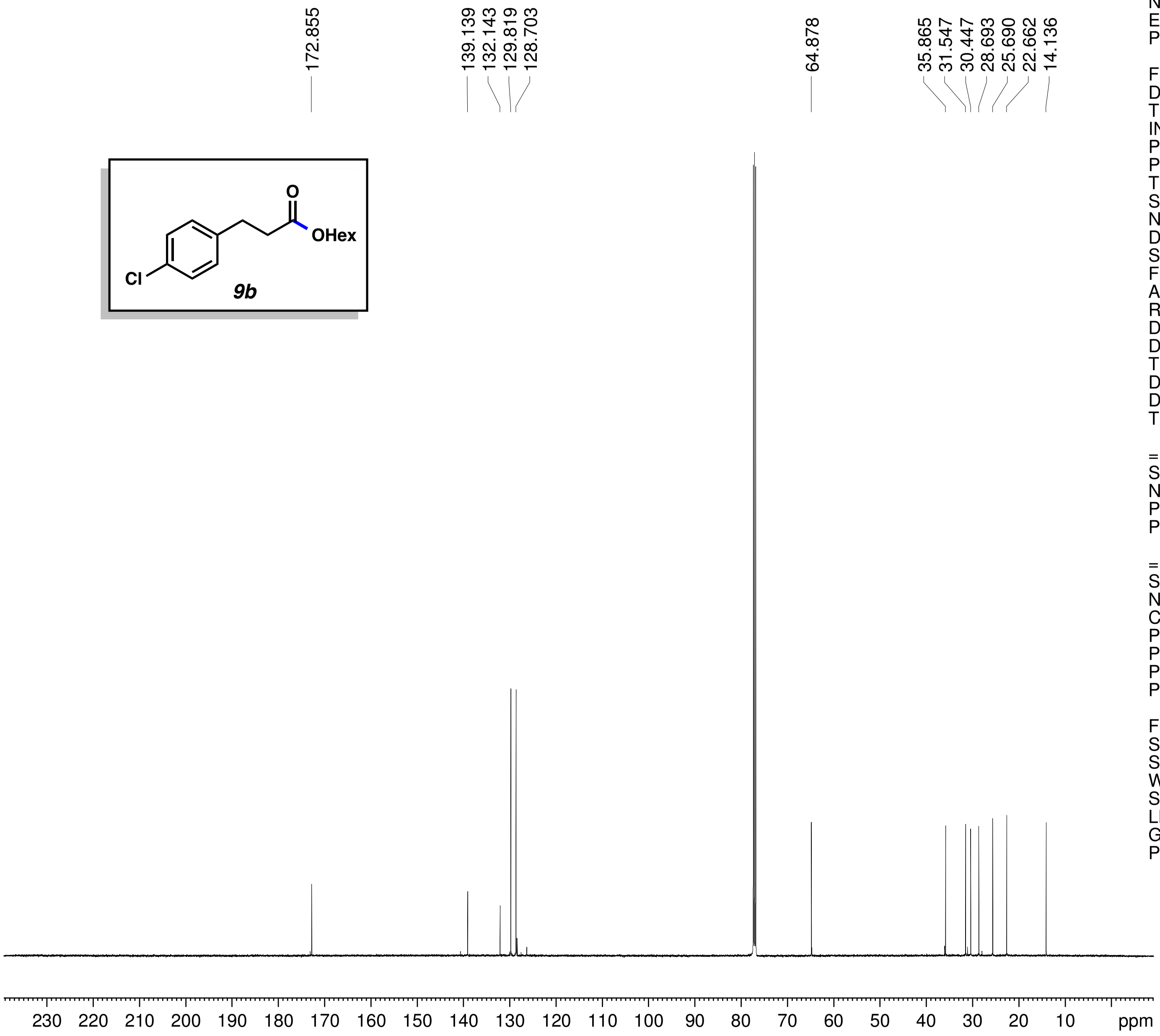


```
Current Data Parameters
NAME       LH-10-131c
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20160530
Time          10.15 h
INSTRUM         av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD              65536
SOLVENT         CDCl3
NS                108
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           9.63 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     0.13500001 W

F2 - Processing parameters
SI             131072
SF      125.7577767 MHz
WDW                EM
SSB     0
LB            1.00 Hz
GB      0
PC               1.40
```



```
Current Data Parameters
NAME        ELB-6-104p
EXPNO               3
PROCNO              1

F2 - Acquisition Parameters
Date_        20160610
Time           15.19
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD             65536
SOLVENT         CDCl3
NS               108
DS                 2
SWH        31250.000 Hz
FIDRES      0.476837 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

======== CHANNEL f1 =====
SFO1      125.7722511 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        125.7577728 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```

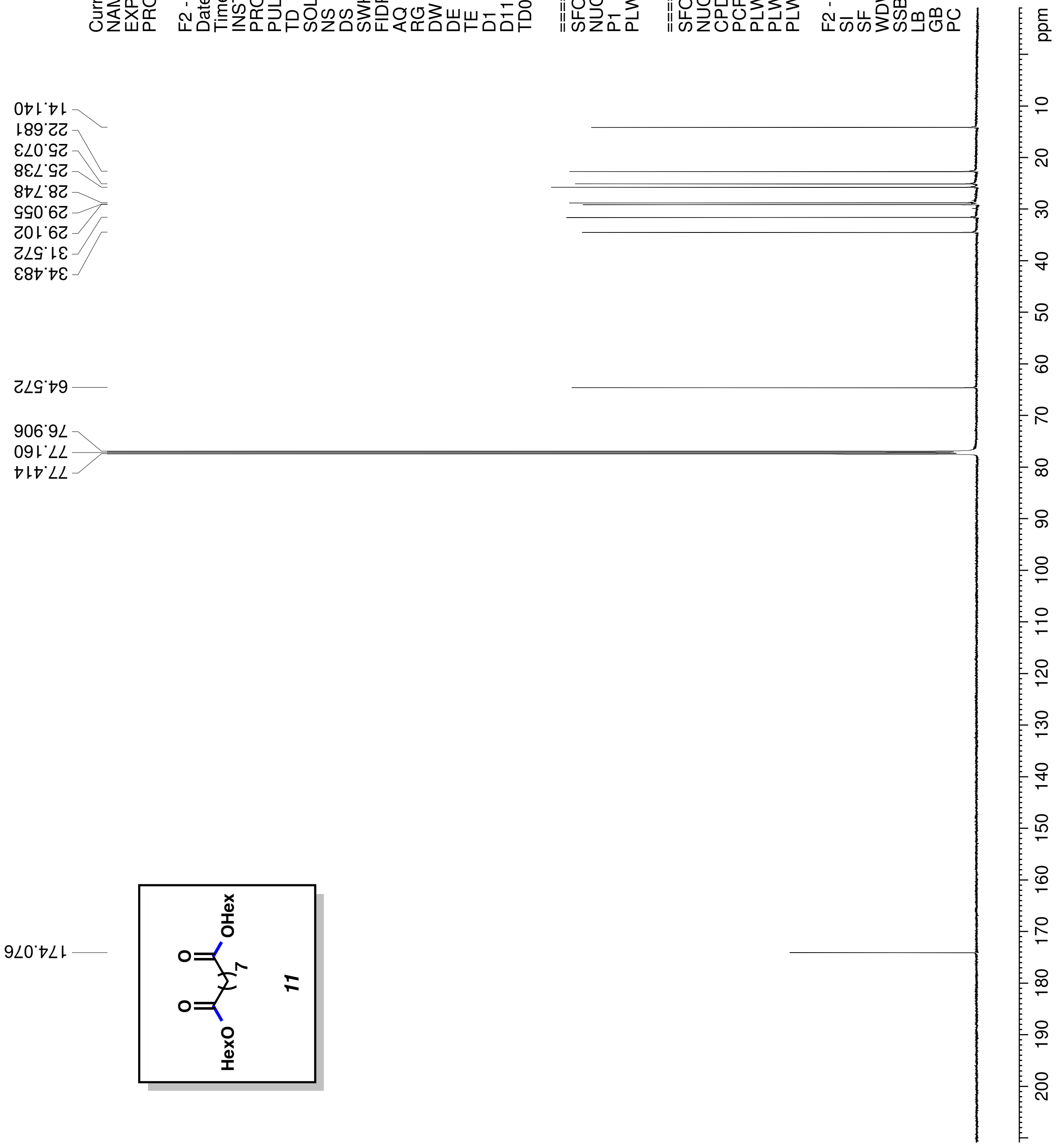
Purified Product, 13C NMR



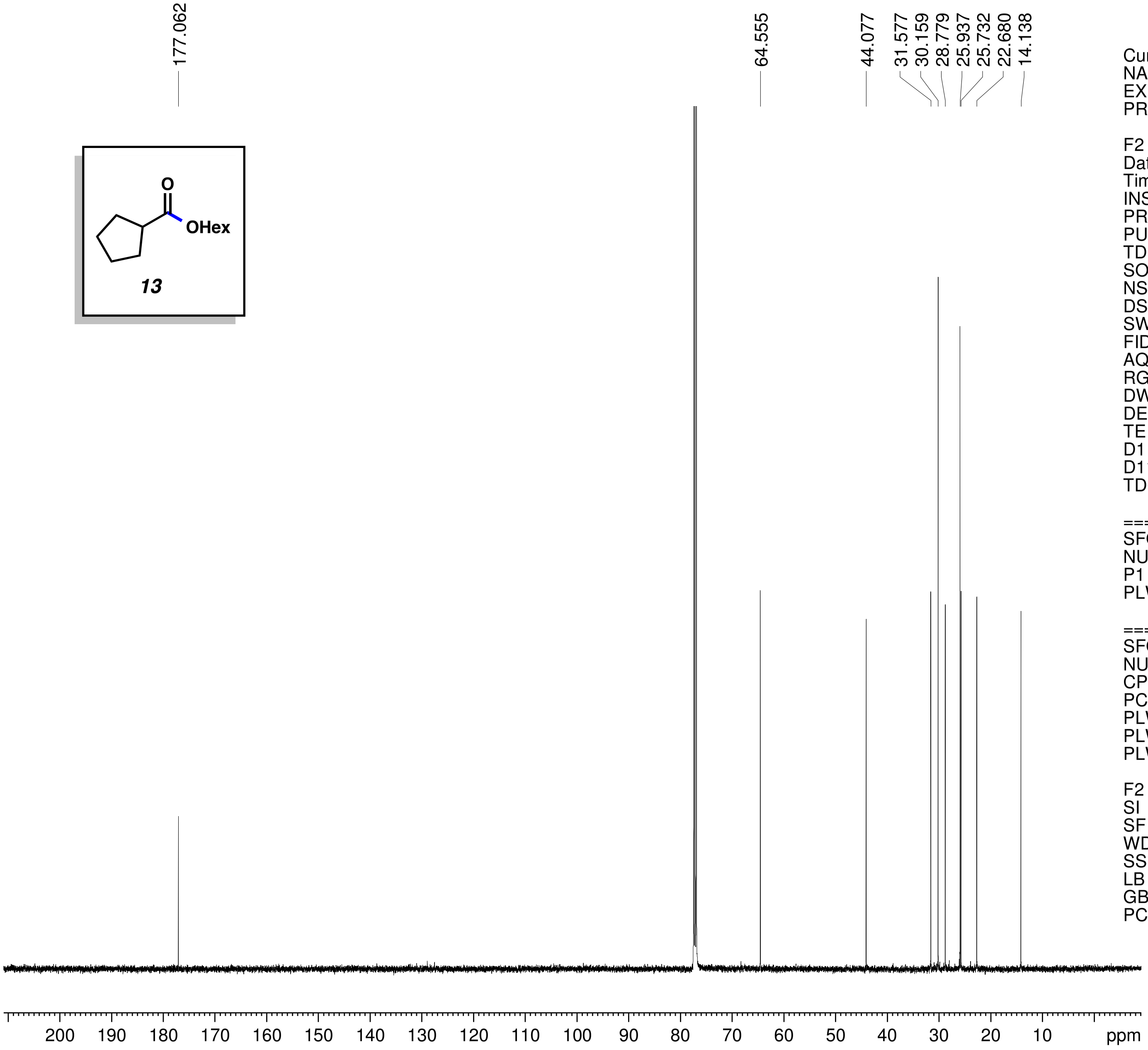
9b

```
Current Data Parameters
NAME         ELB-6-280p
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
Date_          20160918
Time              14.41
INSTRUM           av500
PROBHD   5 mm DCH 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   56
DS                    2
SWH        31250.000 Hz
FIDRES      0.476837 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

======== CHANNEL f1 =====
SFO1     125.7722511 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
```

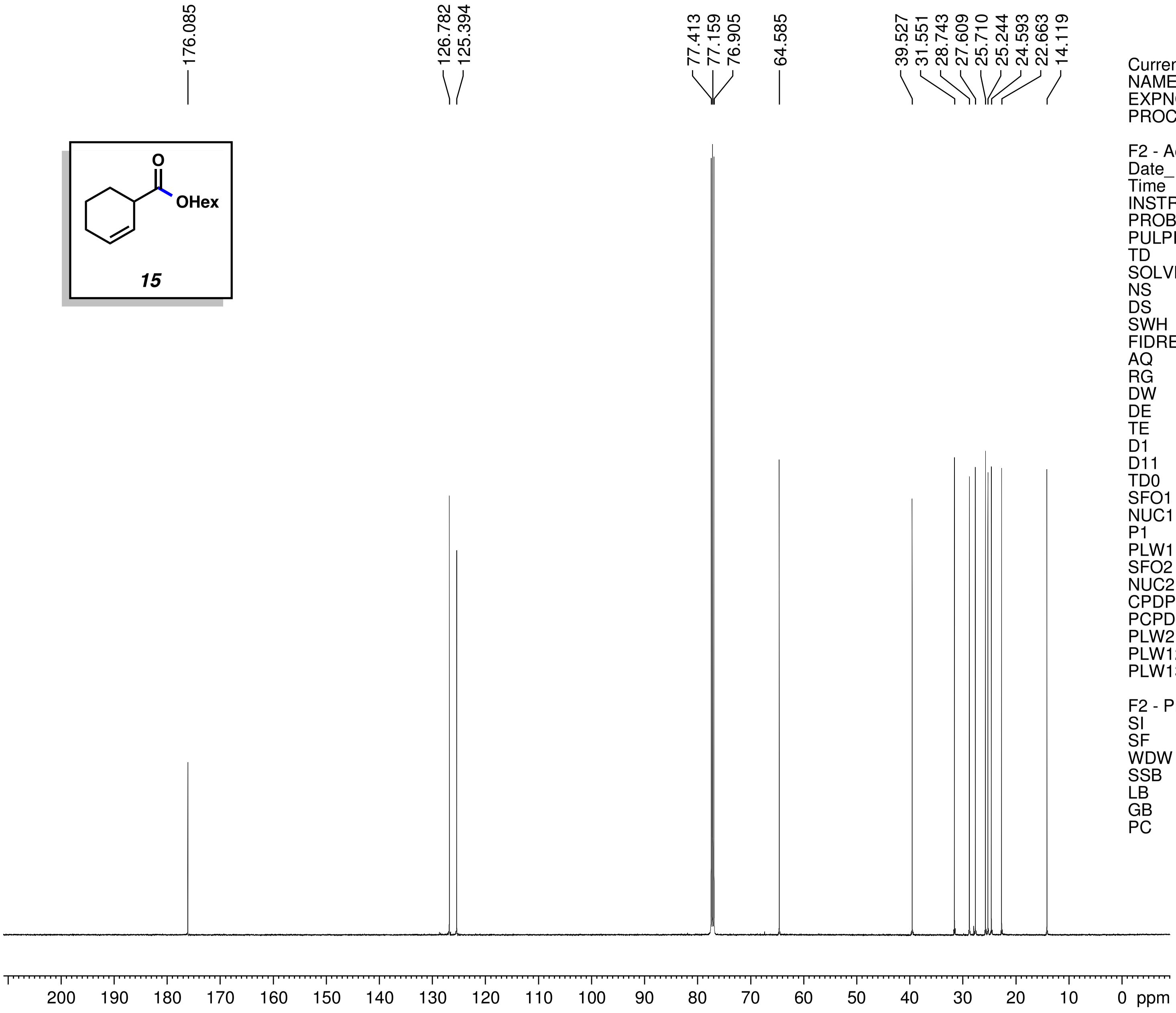
```
Current Data Parameters
NAME     LH-9-288-4-carbon
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_       20160131
Time           19.04
INSTRUM        av500
PROBHD   5 mm DCH 13C-1
PULPROG       zgpg30
TD            65536
SOLVENT        CDCl3
NS               268
DS                 2
SWH       31250.000 Hz
FIDRES     0.476837 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

======== CHANNEL f1 ========
SFO1     125.7722511 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
```



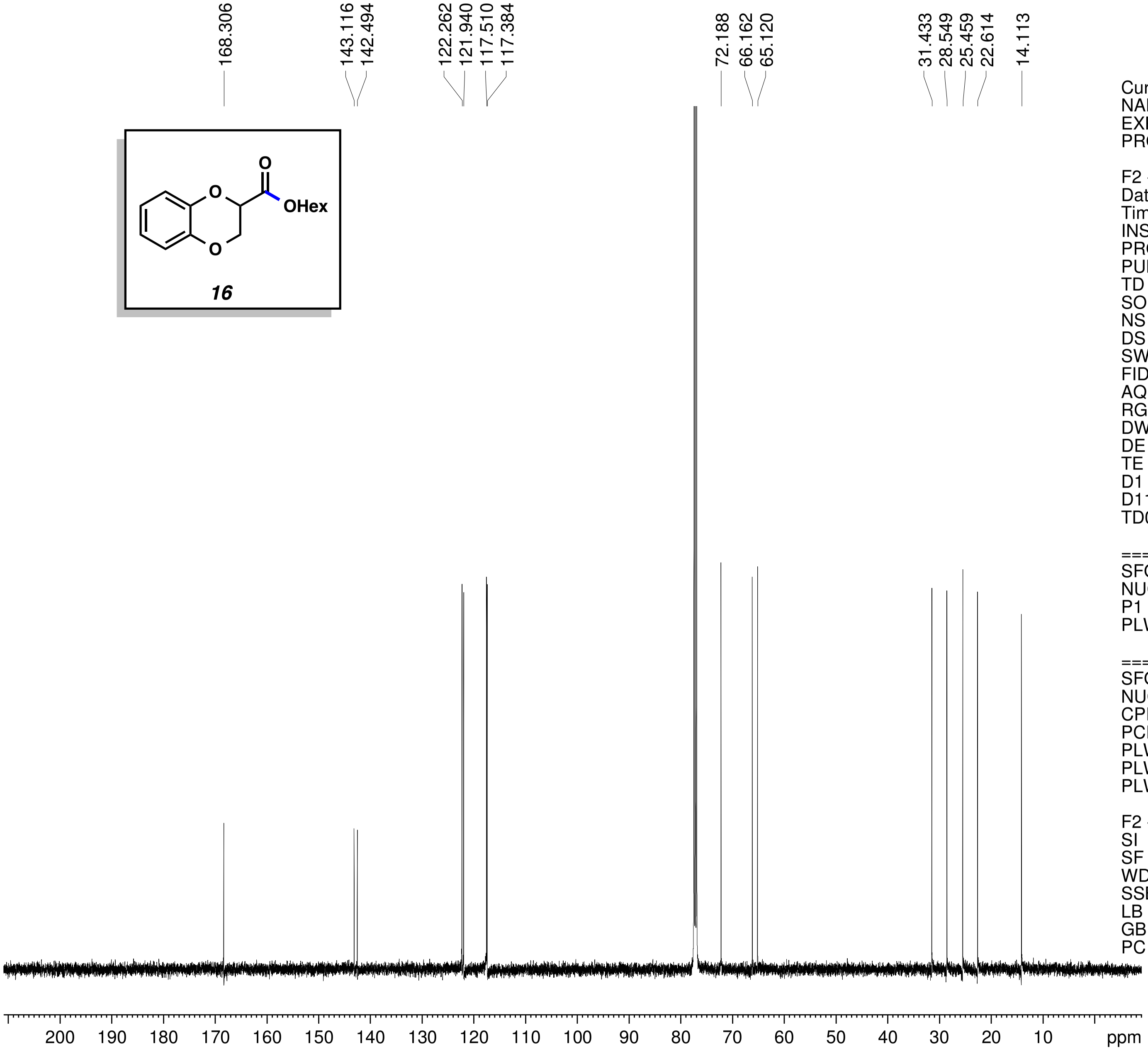
13



```
Current Data Parameters
NAME         ELB-5-202p
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20160509
Time              12.29
INSTRUM           av500
PROBHD   5 mm DCH 13C-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   35
DS                    2
SWH          31250.000 Hz
FIDRES        0.476837 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

======== CHANNEL f1 =====
SFO1      125.7722511 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
```

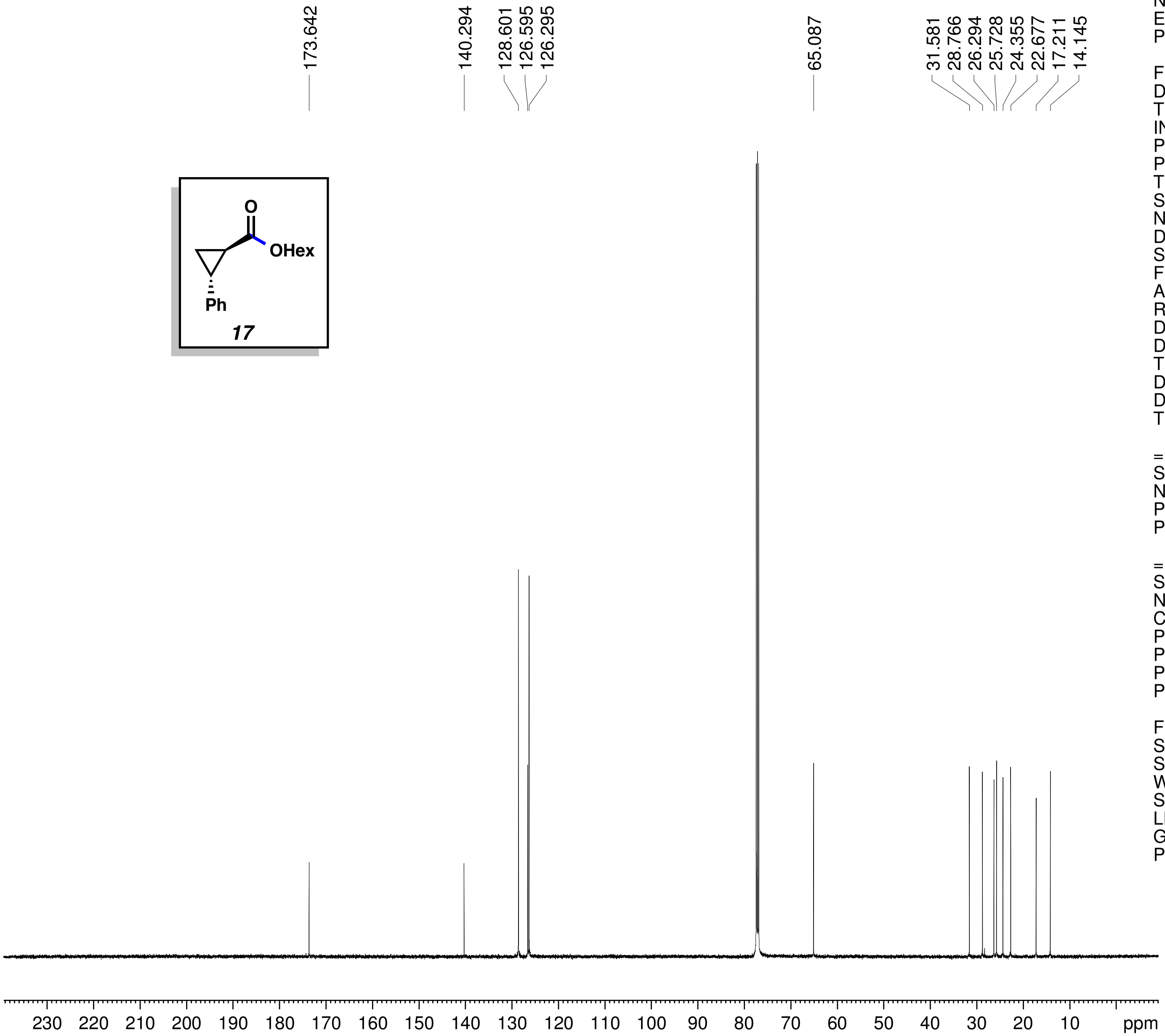


```
Current Data Parameters
NAME          LH-10-124
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20160504
Time              19.38 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 142
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                9.63 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       0.13500001 W

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

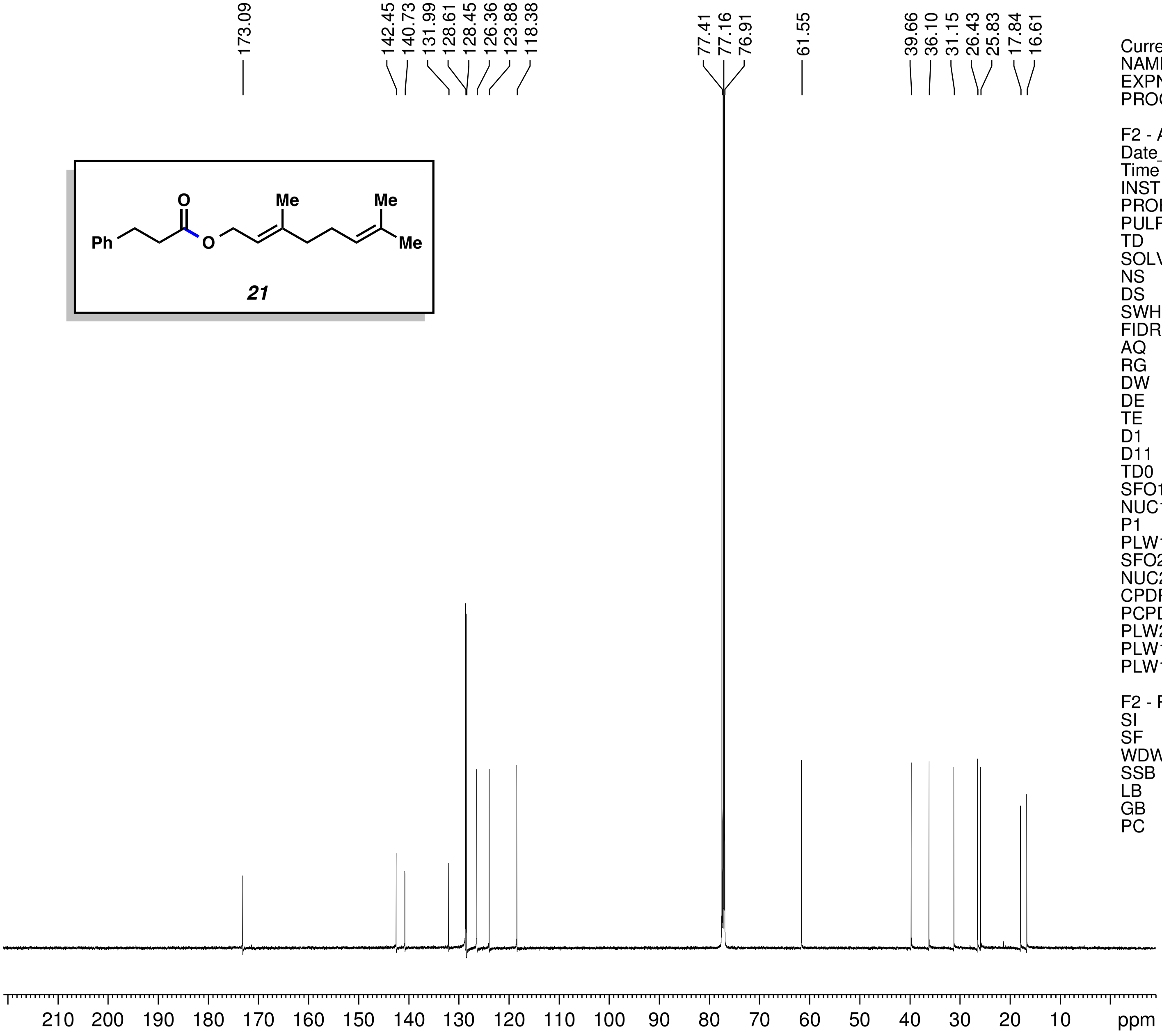


```
Current Data Parameters
NAME         ELB-5-151p
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20160120
Time              16.04
INSTRUM           av500
PROBHD   5 mm DCH 13C-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   30
DS                    2
SWH          31250.000 Hz
FIDRES        0.476837 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

======== CHANNEL f1 =====
SFO1       125.7722511 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         125.7577724 MHz
WDW                  EM
SSB     0
LB                 1.00 Hz
GB      0
PC                 1.40
```

Purified Product, 13C NMR



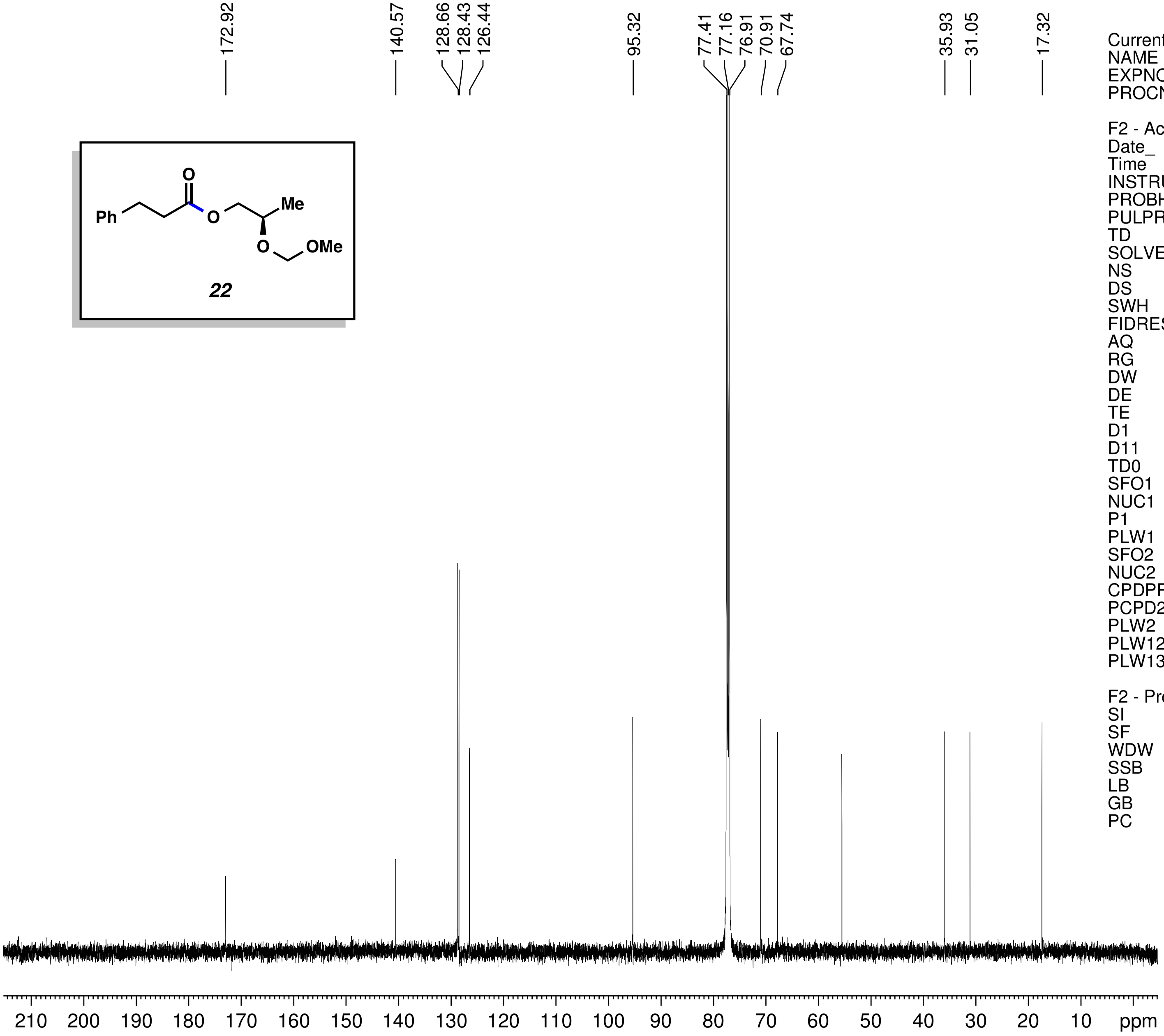
```
Current Data Parameters
NAME         ELB-6-136p
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_         20160616
Time             13.35
INSTRUM          av500
PROBHD   5 mm DCH 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT           CDCl3
NS                  32
DS                   2
SWH         31250.000 Hz
FIDRES       0.476837 Hz
AQ          1.0485760 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

======== CHANNEL f1 =====
SFO1       125.7722511 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        125.7577736 MHz
WDW                EM
SSB     0
LB               1.00 Hz
GB      0
PC               1.40
```



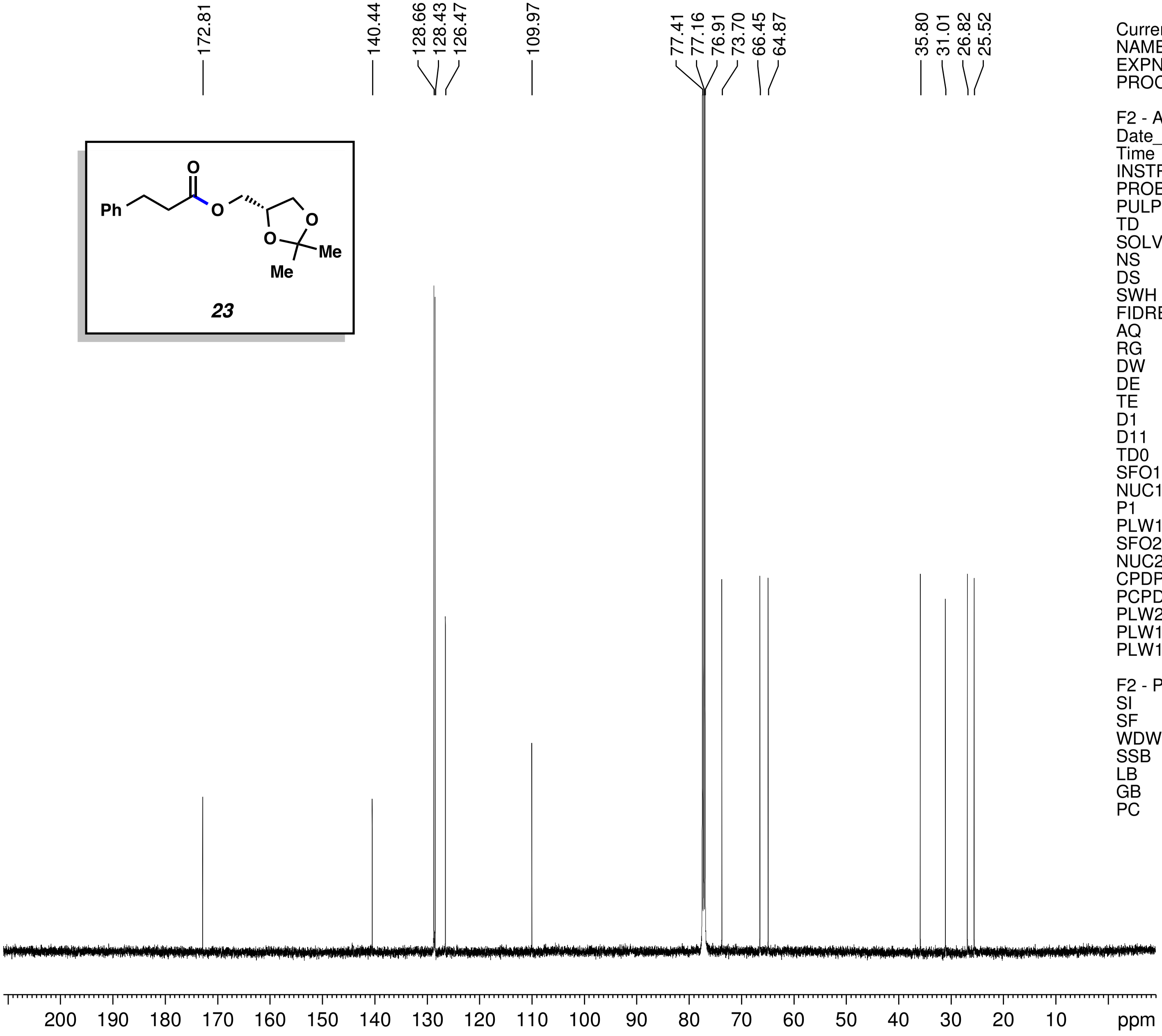
21

```
Current Data Parameters
NAME          LH-10-70
EXPNO               2
PROCNO              1

F2 - Acquisition Parameters
Date_          20160328
Time              9.38 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              9.63 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     0.13500001 W

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

22



```
Current Data Parameters
NAME          LH-10-83
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_         20160406
Time            19.32 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 293
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            9.63 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     0.13500001 W

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



23

Current Data Parameters

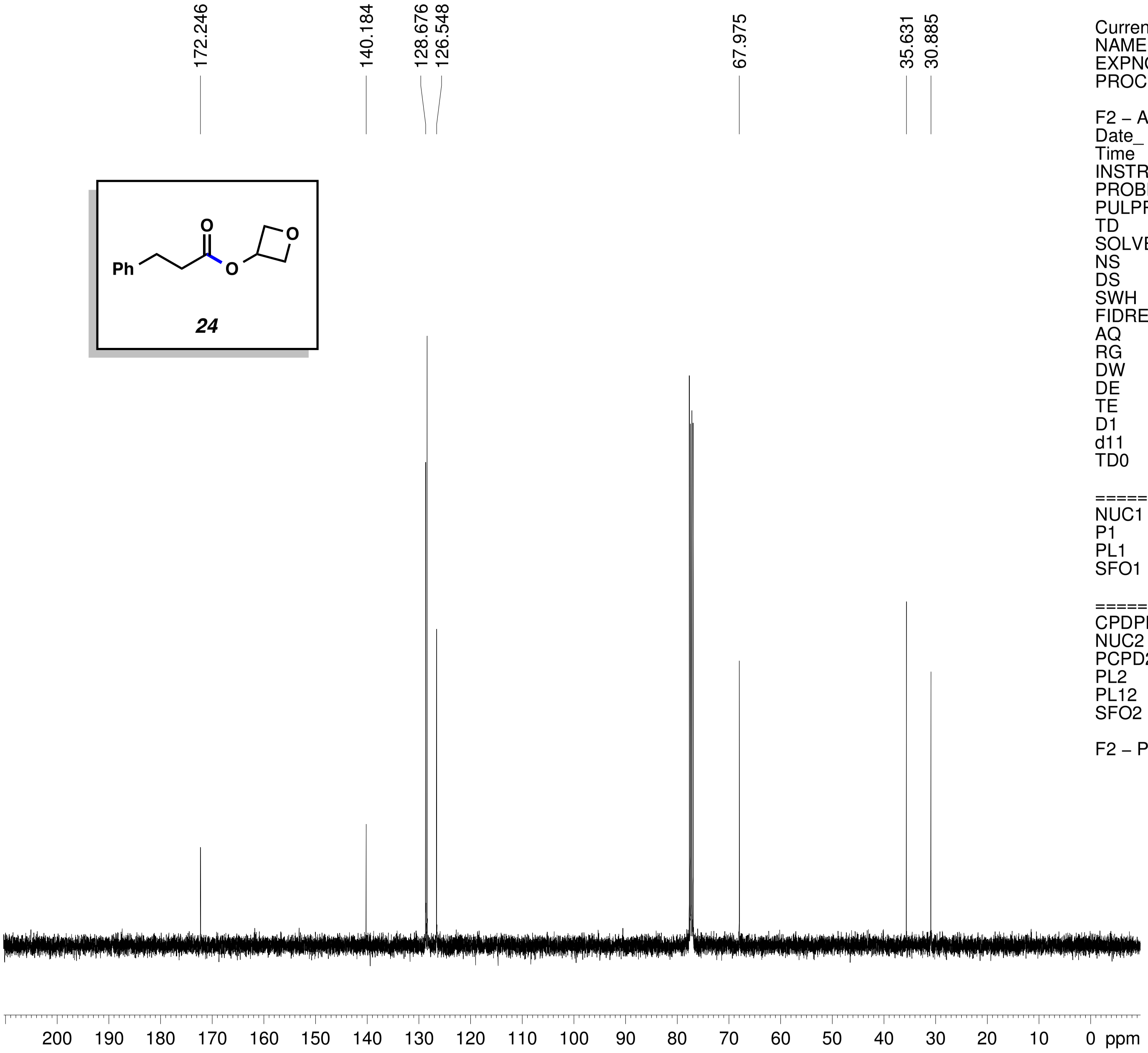
NAME	LH-10-92
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

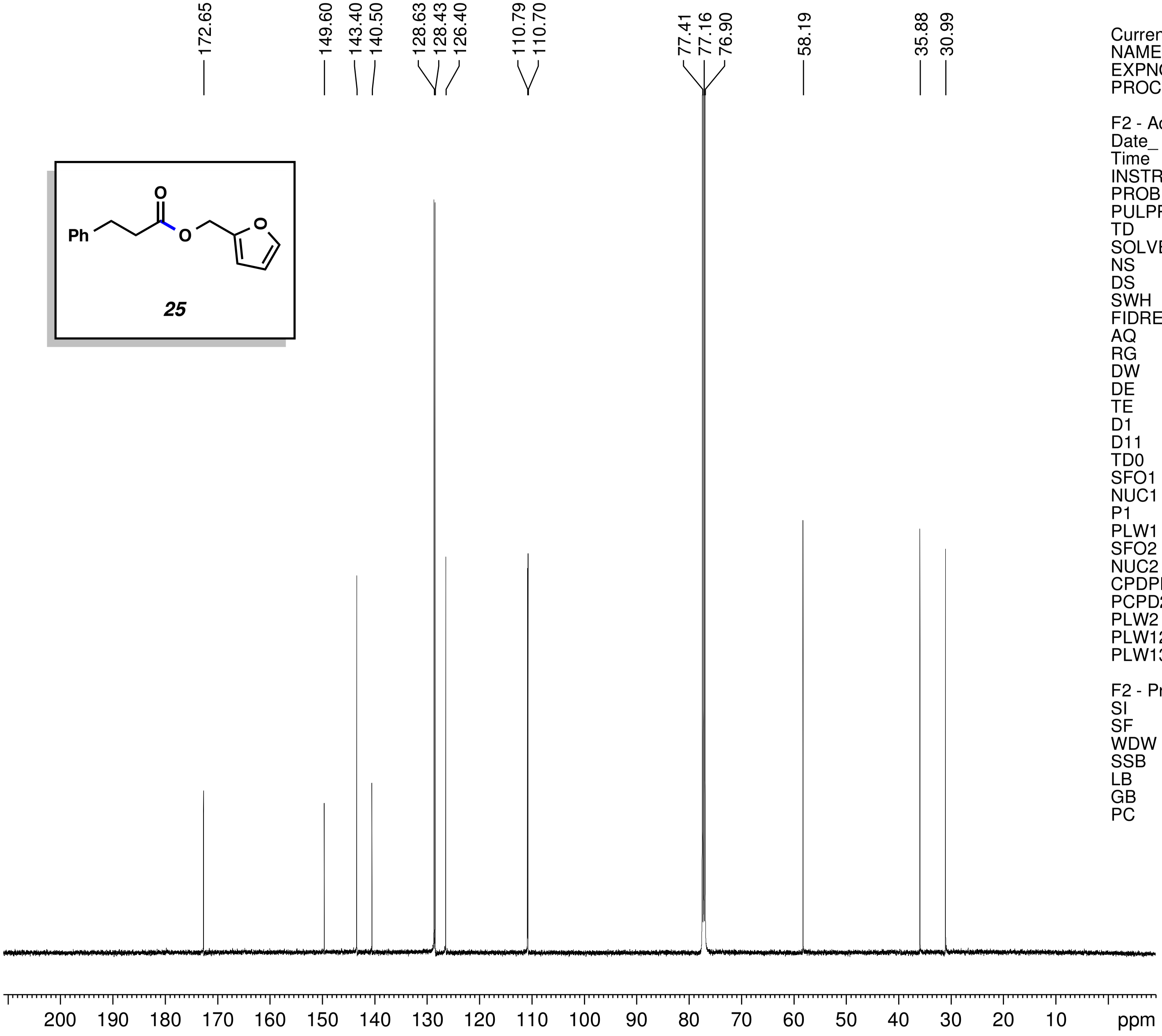
Date_	20160411
Time	14.19 h
INSTRUM	av500
PROBHD	Z119248_0002 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	186
DS	2
SWH	31250.000 Hz
FIDRES	0.953674 Hz
AQ	1.0485760 sec
RG	204.54
DW	16.000 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	125.7722511 MHz
NUC1	13C
P1	9.63 usec
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	0.13500001 W

F2 - Processing parameters

SI	131072
SF	125.7577728 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



```
Current Data Parameters
NAME          LH-10-40
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_         20160310
Time             18.43
INSTRUM         drx500
PROBHD   5 mm bb-Z Z800
PULPROG        zgdc30
TD               65536
SOLVENT          CDCl3
NS                 170
DS                   0
SWH        32679.738 Hz
FIDRES      0.498653 Hz
AQ        1.0027661 sec
RG                4096
DW          15.300 usec
DE            6.00 usec
TE               297.0 K
D1       2.00000000 sec
d11      0.03000000 sec
TD0                  1

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

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

F2 - Processing parameters
```



25

Current Data Parameters

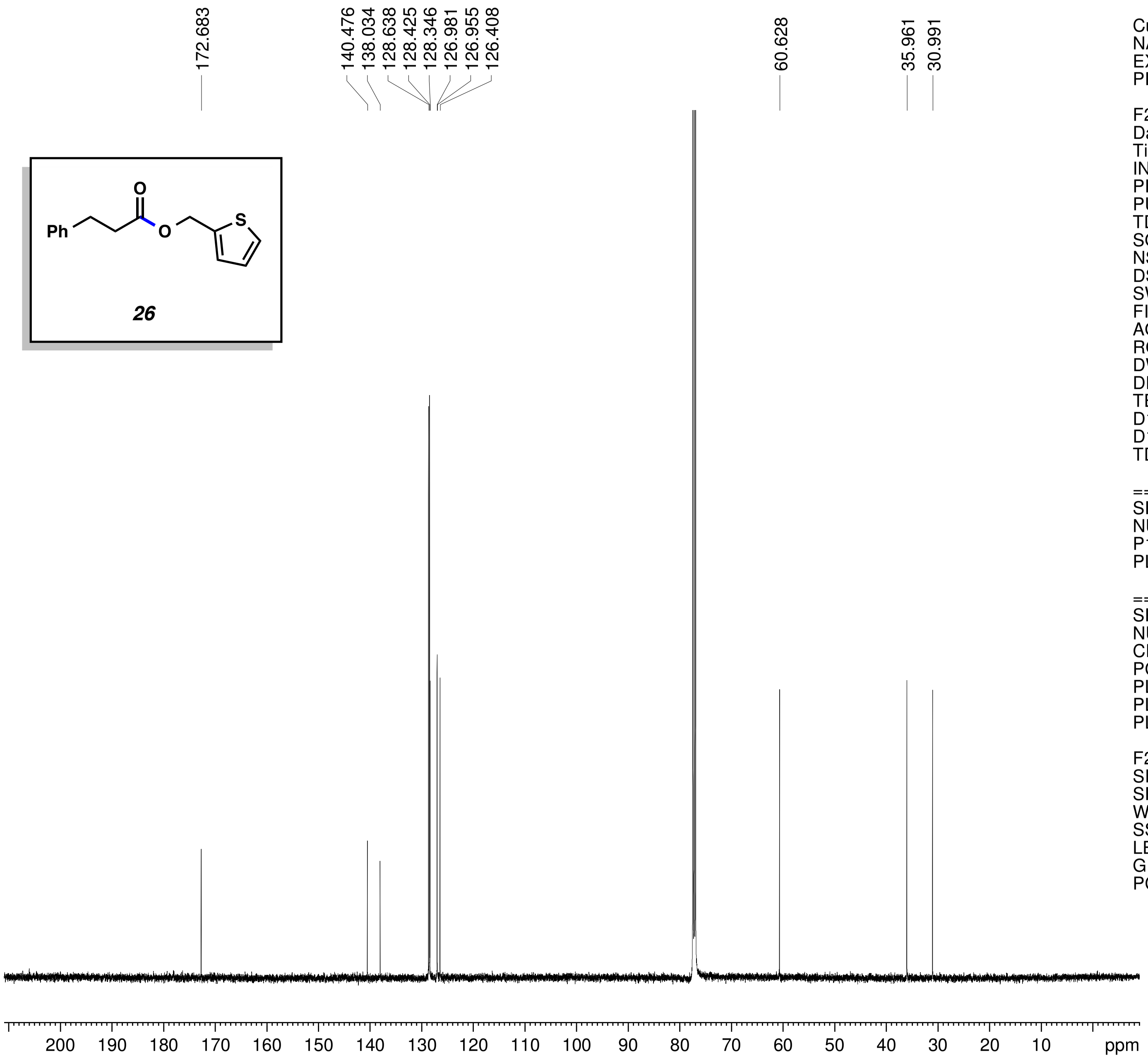
NAME	LH-10-81A
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20160406
Time	10.38 h
INSTRUM	av500
PROBHD	Z119248_0002 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	133
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	9.63 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	0.13500001 W

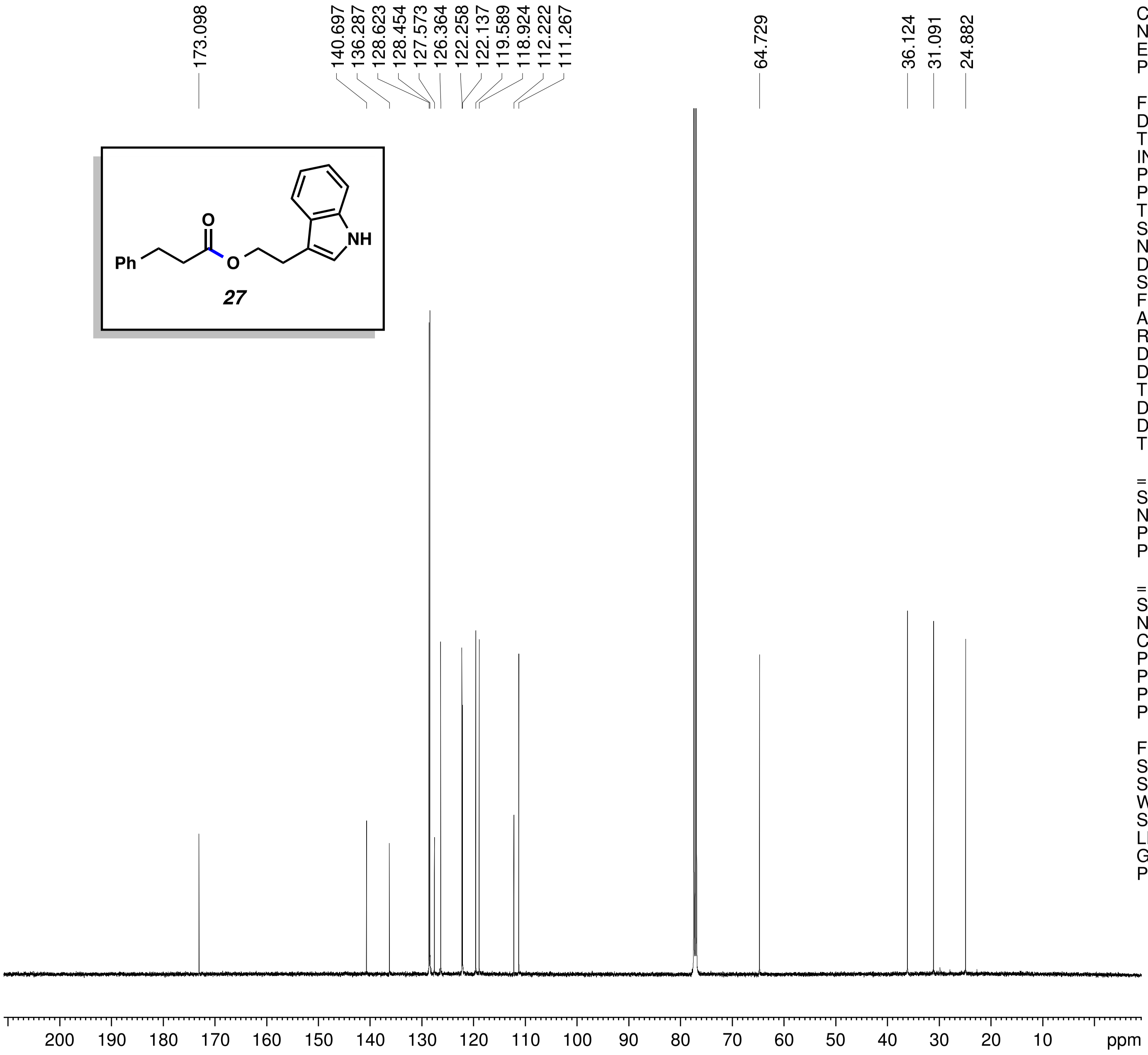
F2 - Processing parameters

SI	131072
SF	125.7577731 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

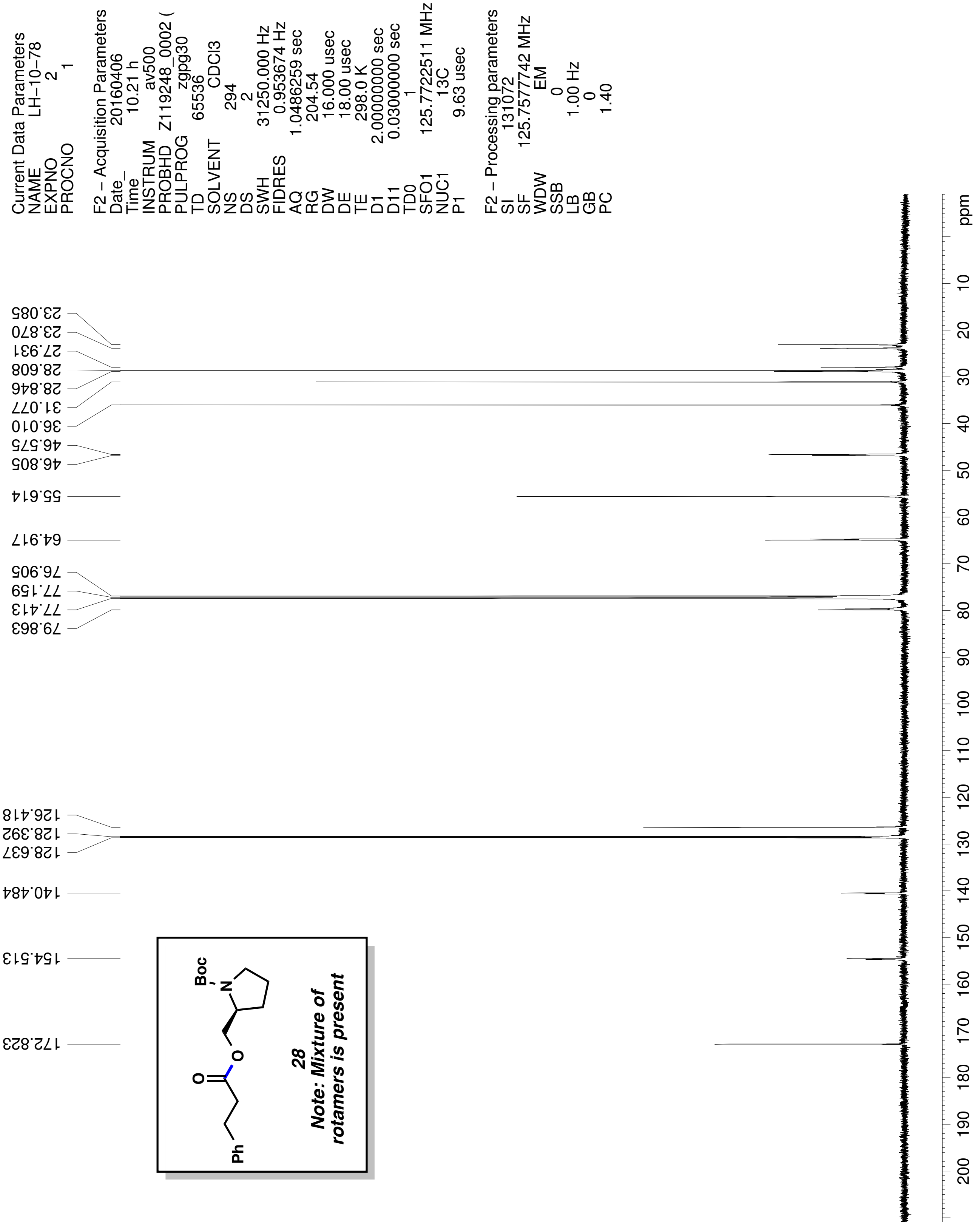


26

```
Current Data Parameters
NAME         ELB-5-273p
EXPNO              5
PROCNO             1

F2 - Acquisition Parameters
Date_        20160503
Time            16.43
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD            65536
SOLVENT         CDCl3
NS                40
DS                 2
SWH        31250.000 Hz
FIDRES      0.476837 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

======== CHANNEL f1 =====
SFO1      125.7722511 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        125.7577737 MHz
WDW               EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```



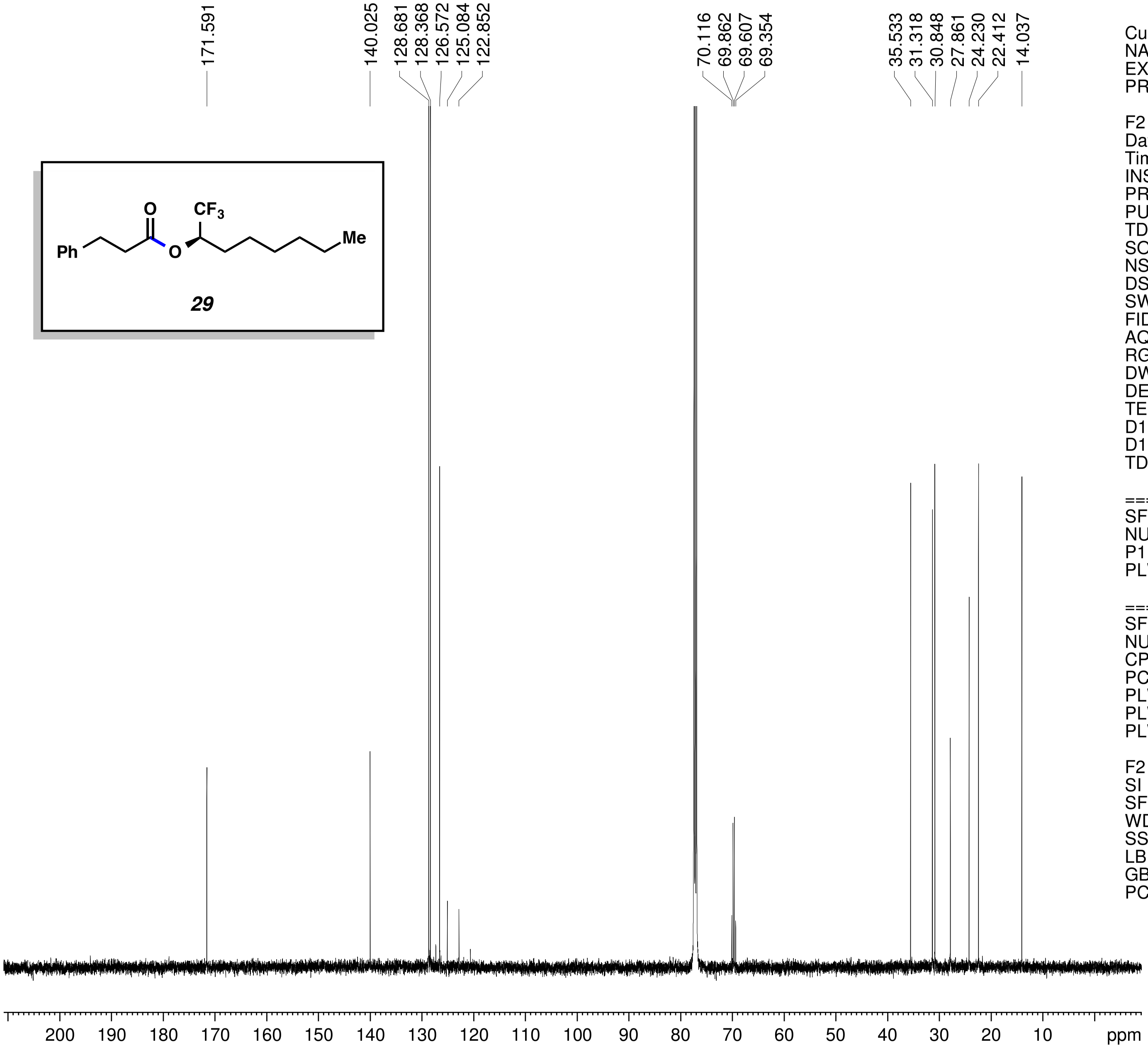
```
Current Data Parameters
NAME         ELB-5-294p
EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
Date_         20160425
Time            11.15
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG        zgpg30
TD             65536
SOLVENT          CDCl3
NS               108
DS                 2
SWH         31250.000 Hz
FIDRES       0.476837 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

======== CHANNEL f1 =====
SFO1      125.7722511 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        125.7577749 MHz
WDW               EM
SSB     0
LB               1.00 Hz
GB      0
PC               1.40
```



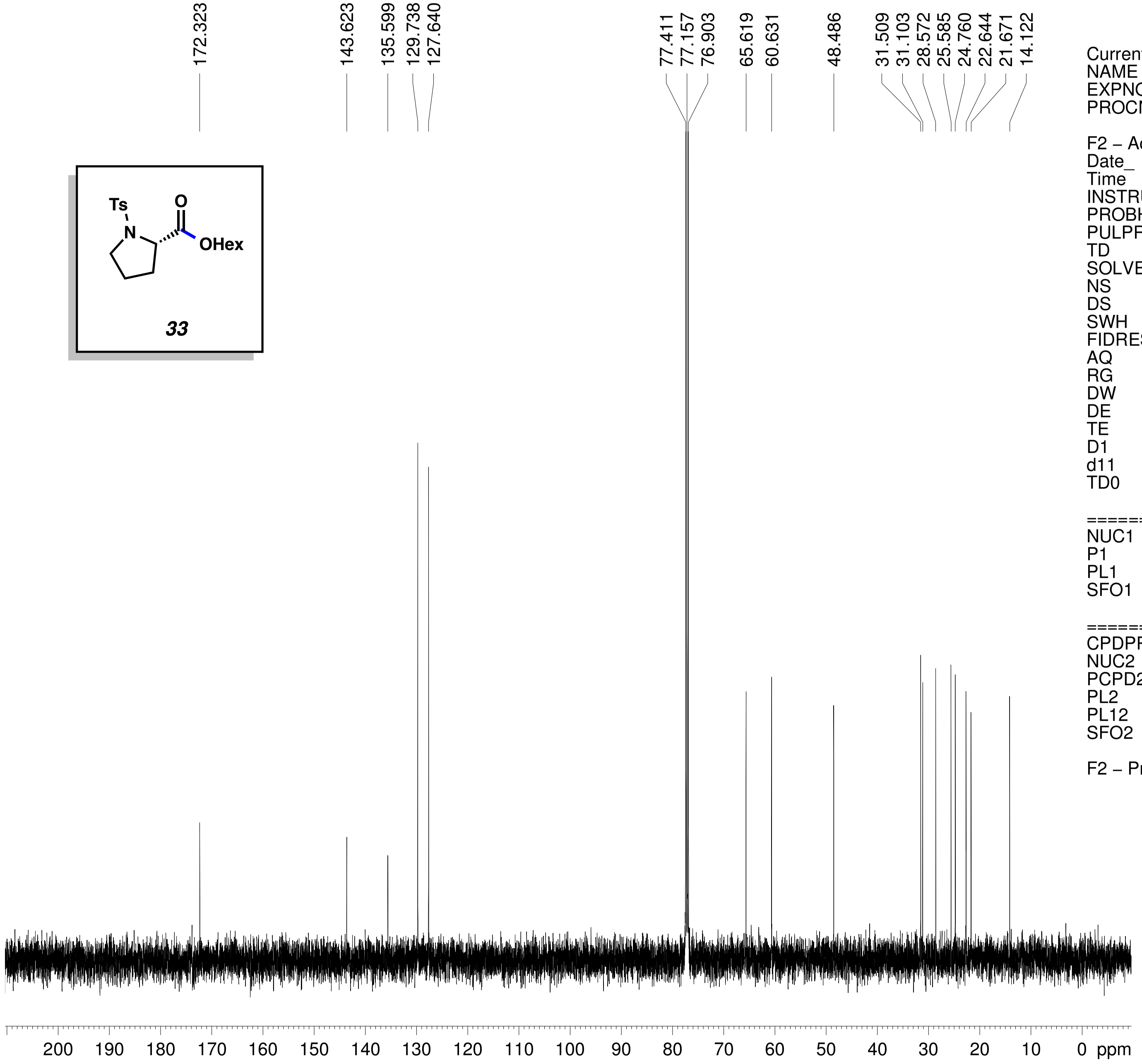
28

Note: Mixture of rotamers is present

```
Current Data Parameters
NAME          LH-10-78
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_         20160406
Time           10.21 h
INSTRUM          av500
PROBHD   Z119248_0002 (
PULPROG        zgpg30
TD              65536
SOLVENT         CDCl3
NS                294
DS                  2
SWH         31250.000 Hz
FIDRES       0.953674 Hz
AQ          1.0486259 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               9.63 usec

F2 - Processing parameters
SI             131072
SF        125.7577742 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```



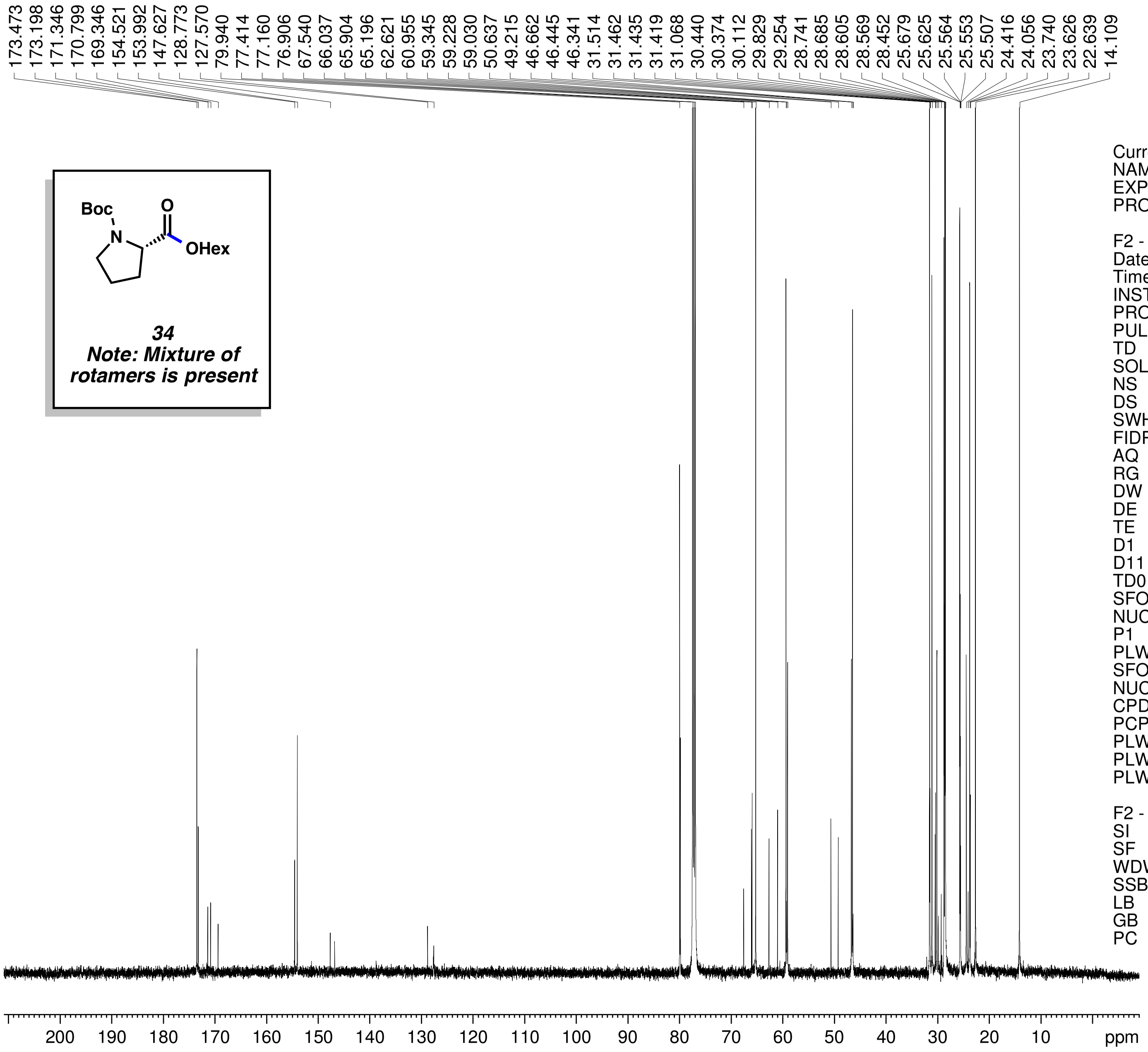
```
Current Data Parameters
NAME         ELB-5-271p
EXPNO                 9
PROCNO                1

F2 - Acquisition Parameters
Date_          20160412
Time              15.47
INSTRUM           av500
PROBHD   5 mm DCH 13C-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  108
DS                    2
SWH        31250.000 Hz
FIDRES      0.476837 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

======== CHANNEL f1 =====
SFO1     125.7722511 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       125.7577728 MHz
WDW                  EM
SSB      0
LB              1.00 Hz
GB       0
PC                 1.40
```



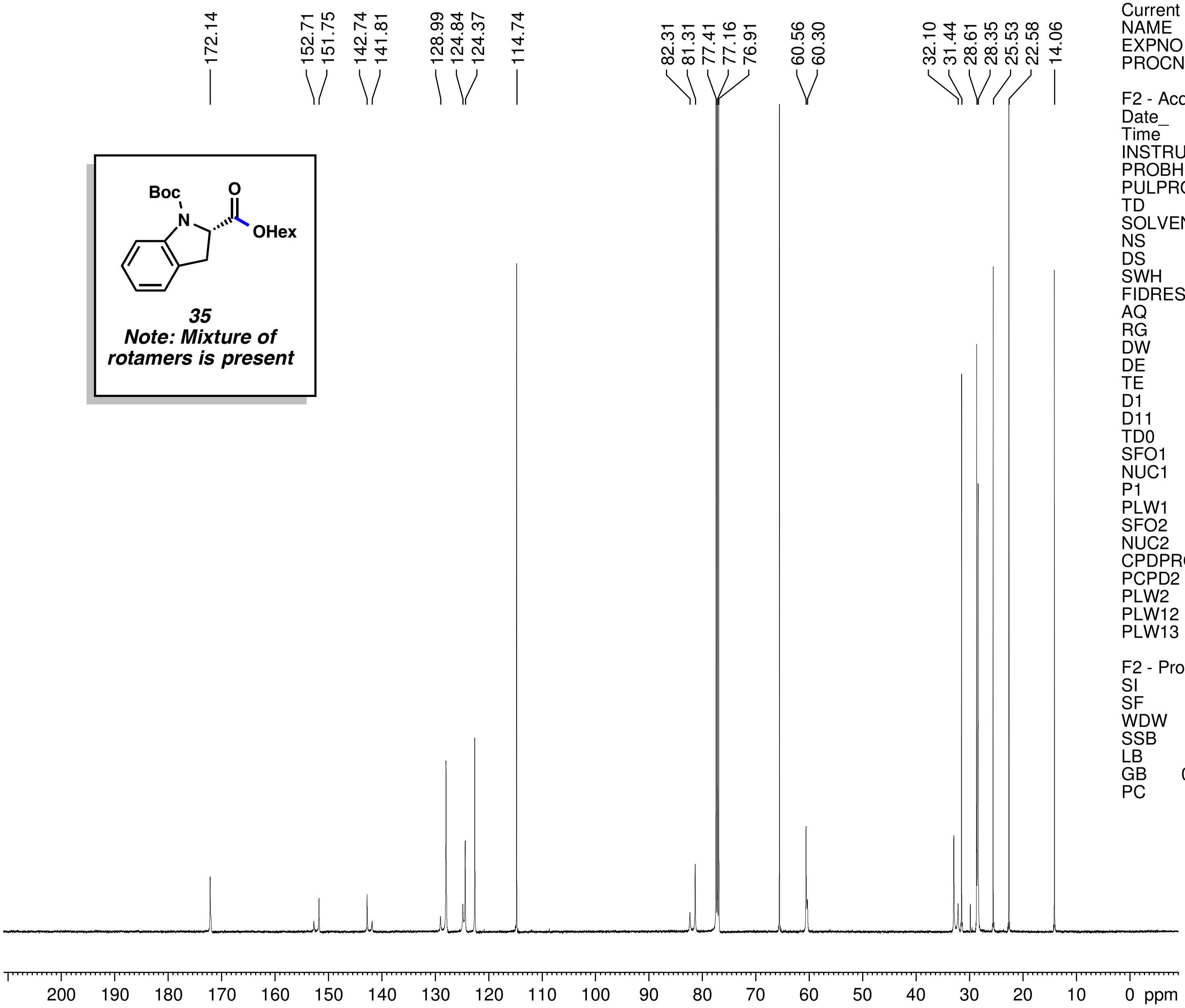
33

```
Current Data Parameters
NAME        LH-9-283-1
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_       20160122
Time           15.31
INSTRUM       drx500
PROBHD   5 mm bb-Z Z800
PULPROG       zgdc30
TD           65536
SOLVENT        CDCl3
NS               101
DS                 0
SWH        32679.738 Hz
FIDRES      0.498653 Hz
AQ         1.0027661 sec
RG              4096
DW            15.300 usec
DE              6.00 usec
TE             296.8 K
D1        2.00000000 sec
d11       0.03000000 sec
TD0                1

======== CHANNEL f1 ========
NUC1             13C
P1              6.50 usec
PL1             0.00 dB
SFO1     125.8231939 MHz

======== CHANNEL f2 ========
CPDPRG2       waltz16
NUC2              1H
PCPD2         100.00 usec
PL2             0.00 dB
PL12           17.52 dB
SFO2     500.3320013 MHz

F2 - Processing parameters
```

34
Note: Mixture of rotamers is present



```
Current Data Parameters
NAME         SMA-1-277p
EXPNO               131
PROCNO                1

F2 - Acquisition Parameters
Date_          20160506
Time             9.10 h
INSTRUM           av500
PROBHD   Z119248_0002 (
PULPROG          zgpg30
TD               65536
SOLVENT           CDCl3
NS                 356
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              9.63 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      0.13500001 W

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



35

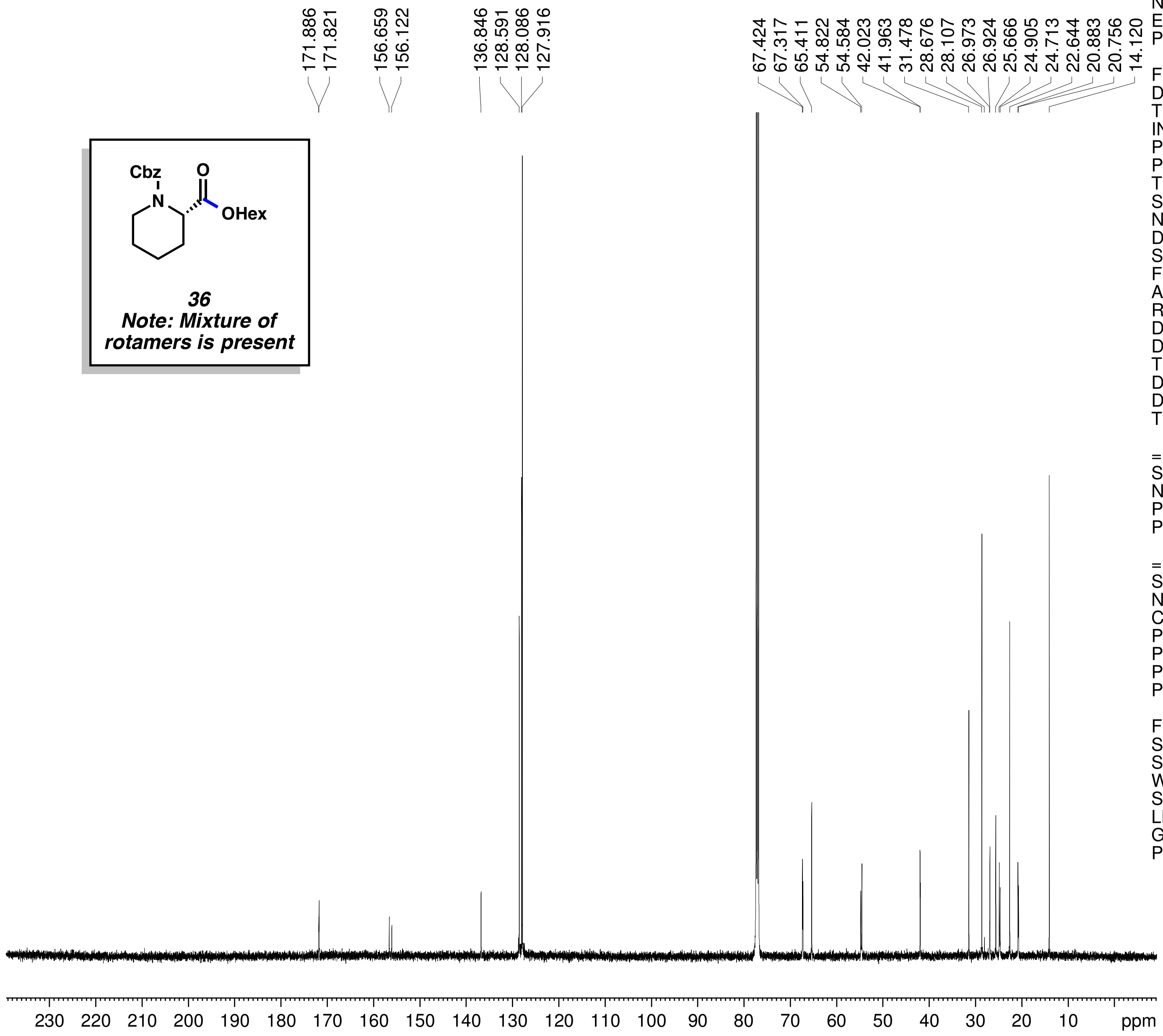
Note: Mixture of rotamers is present

Current Data Parameters
NAME LH-10-149
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160527
Time 13.20 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 232
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 9.63 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 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577779 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Purified Product, 13C NMR



36

Note: Mixture of rotamers is present

```
Current Data Parameters
NAME        ELB-6-107p
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20160620
Time            15.16
INSTRUM         av500
PROBHD   5 mm DCH 13C-1
PULPROG       zgpg30
TD             65536
SOLVENT         CDCl3
NS               108
DS                 2
SWH       31250.000 Hz
FIDRES     0.476837 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

======== CHANNEL f1 =====
SFO1      125.7722511 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        125.7577728 MHz
WDW              EM
SSB     0
LB             1.00 Hz
GB      0
PC             1.40
```
]