

Supporting Information

Enantioselective Synthesis of Trifluoromethyl Substituted Piperidines with Multiple Stereogenic Centers *via* Hydrogenation of Pyridinium Chlorides

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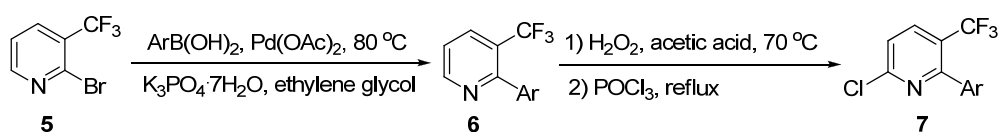
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1. General and materials:

General: All reactions were carried out under an atmosphere of nitrogen using standard Schlenk techniques, unless otherwise noted. ^1H NMR and ^{13}C NMR spectra were recorded at room temperature in CDCl_3 on 400 MHz instrument with tetramethylsilane (TMS) as internal standard. Enantiomeric excess was determined by HPLC analysis, using chiral column described below in detail. Optical rotations were measured by polarimeter. Flash column chromatography was performed on silica gel (200-300 mesh). All reactions were monitored by TLC analysis.

Materials: Commercially available reagents were used throughout without further purification other than those detailed below. The solvents for asymmetric hydrogenation reaction were purchased without further purification.

2. Synthesis of 6-alkyl-2-aryl-3-(trifluoromethyl)pyridines:



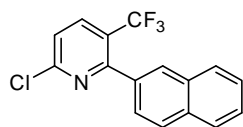
General procedure for the synthesis of 6-chloro-2-aryl-3-(trifluoromethyl)pyridines:

Procedure one: A mixture of 2-bromo-3-(trifluoromethyl)pyridine **5** (10.0 mmol), arylboronic acid (15.0 mmol), Pd(OAc)₂ (0.5 mol%, 11.2 mg), K₃PO₄·7H₂O (20.0 mmol, 6.768 g) and ethylene glycol (20.0 mL) was stirred at 80 °C for indicated time. The mixture was added to brine (30 mL). The mixture was extracted with diethyl ether (3×20 mL). The extracts were combined, dried over sodium sulfate, and evaporated under reduced pressure. The residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **6**.^[1]

Procedure two: Hydrogen peroxide (30%, 1.4 mL, 13 mmol) was added into the solution of the 2-aryl-3-(trifluoromethyl)pyridine **6** (10.0 mmol) in 10 mL of acetic acid. The reaction mixture was stirred at 70 °C for 72 h. The solvent was evaporated under vacuum, and the residue was basified with aqueous solution of sodium carbonate until pH = 9. The resulting mixture was extracted with chloroform (3×20 mL). The organic phase were combined and dried over anhydrous sodium sulfate, filtered and evaporated under vacuum. The residue was purified by flash chromatography on silica gel (dichloromethane/methanol = 15/1).^[2]

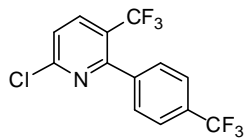
Procedure three: 2-aryl-3-(trifluoromethyl)pyridine *N*-oxide (5.0 mmol) is taken up in excess phosphoryl chloride (10.0 mL). The mixture is refluxed for 4 h, cooled, poured into cold water (50.0 mL), basified with 10% aqueous ammonia solution (20 mL), and extracted with chloroform (2×50 mL). The organic phase were combined and dried over anhydrous sodium sulfate, filtered and evaporated under vacuum. The residue was purified by flash chromatography on silica gel (dichloromethane/methanol = 20/1) to yield the products **7a-7h**.^[3] The 6-chloro-2-aryl-3-(trifluoromethyl) pyridines **7a-7d**, and **7f** are the known compounds.^[4]

6-Chloro-2-(naphthalen-2-yl)-3-(trifluoromethyl)pyridine (7e): 54% yield, a yellow oil, R_f = 0.60 (petroleum ether/ethyl acetate = 20/1); ^1H NMR (400 MHz, CDCl_3) δ 7.89-7.84 (m, 2H), 7.80-7.73 (m, 3H), 7.51 (dd, J = 8.5, 1.3 Hz, 1H), 7.44-7.35 (m, 2H), 7.26 (d, J = 8.3 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.2 (d, J = 2.0 Hz), 153.6, 137.6 (q, J = 4.0 Hz), 135.2, 133.5, 132.7, 128.7 (d, J = 2.0 Hz), 128.7, 127.9, 127.8, 126.6, 126.1 (d, J = 1.0 Hz), 124.9, 123.9 (q, J = 32.0 Hz), 122.6, 122.1; ^{19}F NMR (376 MHz, CDCl_3) δ -56.8; HRMS (ESI) m/z Calculated for

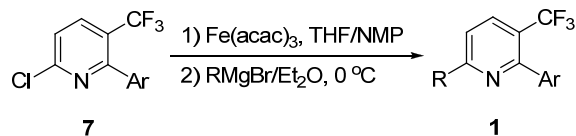
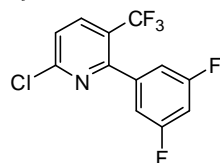


C₁₆H₁₀ClF₃N [M+H]⁺ 308.0448, found 308.0447.

6-Chloro-3-(trifluoromethyl)-2-(4-(trifluoromethyl)phenyl)pyridine (7g): 68% yield, white solid, mp = 61-62 °C, R_f = 0.70 (petroleum ether/ ethyl acetate = 20/1); ¹H NMR (400 MHz, CDCl₃) δ 8.06 (d, *J* = 8.4 Hz, 1H), 7.73 (d, *J* = 8.2 Hz, 2H), 7.64 (d, *J* = 8.1 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 157.8, 154.0, 141.3, 137.8 (q, *J* = 5.0 Hz), 131.6 (d, *J* = 33.0 Hz), 129.4 (q, *J* = 2.0 Hz), 128.7 (d, *J* = 77.0 Hz), 125.3 (q, *J* = 33.0 Hz), 124.2 (q, *J* = 33.0 Hz), 123.5, 122.3 (q, *J* = 79.0 Hz); ¹⁹F NMR (376 MHz, CDCl₃) δ -57.0, -62.9; HRMS (ESI) *m/z* Calculated for C₁₃H₇ClF₆N [M+H]⁺ 326.0166, found 326.0165.

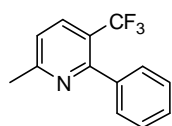


6-Chloro-2-(3,5-difluorophenyl)-3-(trifluoromethyl)pyridine (7h): 58% yield, yellow oil, R_f = 0.59 (petroleum ether/ ethyl acetate = 20/1); ¹H NMR (400 MHz, CDCl₃) δ 8.05 (d, *J* = 8.4 Hz, 1H), 7.52 (d, *J* = 8.4 Hz, 1H), 7.06 (d, *J* = 5.8 Hz, 2H), 7.00-6.92 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 162.6 (dd, *J* = 248.0, 13.0 Hz), 156.5, 153.9, 140.4, 137.7 (q, *J* = 4.8 Hz), 123.9 (q, *J* = 33.0 Hz), 123.6, 112.2 (d, *J* = 27.0 Hz), 105.2, 104.8 (d, *J* = 25.0 Hz); ¹⁹F NMR (376 MHz, CDCl₃) δ -57.2, -109.2; HRMS (ESI) *m/z* Calculated for C₁₂H₆ClF₅N [M+H]⁺ 294.0103, found 294.0100.

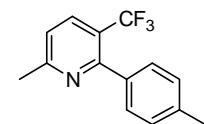


General procedure for synthesis of 6-alkyl-2-aryl-3-(trifluoromethyl)pyridines: RMgBr (3.6 mmol, 1.2 equiv.) was added to a solution of the corresponding 6-chloro-2-aryl-3-(trifluoromethyl)pyridine **7** (3.0 mmol) and Fe(acac)₃ (10 mol%) in THF/NMP (10.0 mL/mmol) cooled to 0 °C. The reaction mixture was stirred at 0 °C for 15 min and then quenched with brine. After extraction with dichloromethane, the combined organic layer was dried over anhydrous sodium sulfate, filtered and concentrated. The resulting crude product was purified by chromatography on silica gel (dichloromethane /methanol = 20/1) to afford the products **1**.^[5]

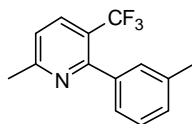
6-Methyl-2-phenyl-3-(trifluoromethyl)pyridine (1a): 95% yield, yellow oil, R_f = 0.30 (petroleum ether/ethyl acetate = 20/1); ¹H NMR (400 MHz, CDCl₃) δ 7.92 (d, *J* = 8.1 Hz, 1H), 7.53-7.45 (m, 2H), 7.43 (dt, *J* = 4.3, 2.7 Hz, 3H), 7.23 (d, *J* = 8.0 Hz, 1H), 2.65 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 161.4, 157.9 (d, *J* = 2.0 Hz), 139.5, 134.9 (q, *J* = 5.0 Hz), 128.6, 128.0, 125.3, 122.2 (q, *J* = 32.0 Hz), 121.3, 119.9, 24.7; ¹⁹F NMR (376 MHz, CDCl₃) δ -57.0; HRMS (ESI) *m/z* Calculated for C₁₃H₁₁F₃N [M+H]⁺ 238.0838, found 238.0842.



6-Methyl-2-*p*-tolyl-3-(trifluoromethyl)pyridine (1b): 80% yield, yellow oil, R_f = 0.34 (petroleum ether/ethyl acetate = 30/1); ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 8.1 Hz, 1H), 7.39 (d, *J* = 7.6 Hz, 2H), 7.24 (t, *J* = 8.1 Hz, 3H), 2.66 (s, 3H), 2.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 161.3, 158.0, 138.5, 136.7, 134.9 (q, *J* = 5.0 Hz), 128.7, 128.6 (d, *J* = 1.0 Hz), 125.4, 122.0 (q, *J* = 32.0 Hz), 121.1, 24.6, 21.3; ¹⁹F NMR (376 MHz, CDCl₃) δ -57.0; HRMS (ESI) *m/z* Calculated for C₁₄H₁₃F₃N [M+H]⁺ 252.0995, found 252.1004.

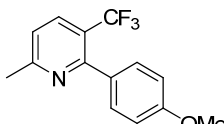


6-Methyl-2-*m*-tolyl-3-(trifluoromethyl)pyridine (1c): 74% yield, yellow oil, R_f = 0.33 (petroleum ether/ethyl acetate = 30/1); ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 8.1 Hz, 1H),



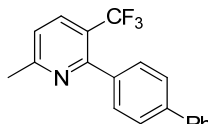
7.33-7.23 (m, 5H), 2.67 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.3 (d, $J = 1.0$ Hz), 158.2 (d, $J = 2.0$ Hz), 139.4, 137.6, 134.8 (q, $J = 5.0$ Hz), 129.4, 129.3 (d, $J = 1.0$ Hz), 127.8, 125.7 (d, $J = 2.0$ Hz), 122.6, 122.1 (q, $J = 32.0$ Hz), 121.2, 24.6, 21.4; ^{19}F NMR (376 MHz, CDCl_3) δ -57.0; HRMS (ESI) m/z Calculated for $\text{C}_{14}\text{H}_{13}\text{F}_3\text{N}$ $[\text{M}+\text{H}]^+$ 252.0995, found 252.0994.

2-(4-Methoxyphenyl)-6-methyl-3-(trifluoromethyl)pyridine (1d): 90% yield, yellow oil, $R_f = 0.20$ (petroleum ether/ethyl acetate = 20/1); ^1H NMR (400 MHz, CDCl_3) δ 7.91 (d, $J = 8.1$ Hz, 1H), 7.45 (d, $J = 8.6$ Hz, 2H), 7.21 (d, $J = 8.1$ Hz, 1H), 7.04-6.87 (m, 2H), 3.85 (s, 3H), 2.65 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.3, 160.1, 157.6, 135.0 (q, $J = 5.0$ Hz), 132.1, 130.1 (d, $J = 2.0$ Hz), 125.4, 121.9 (q, $J = 32.0$ Hz), 120.9, 113.5, 55.3, 24.7; ^{19}F NMR (376 MHz, CDCl_3) δ -57.0; HRMS (ESI) m/z Calculated for $\text{C}_{14}\text{H}_{13}\text{F}_3\text{NO}$ $[\text{M}+\text{H}]^+$ 268.0944, found 268.0953.



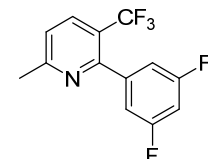
6-Methyl-2-(naphthalen-2-yl)-3-(trifluoromethyl)pyridine (1e): 77% yield, yellow oil, $R_f = 0.40$ (petroleum ether/ethyl acetate = 30/1); ^1H NMR (400 MHz, CDCl_3) δ 8.00-7.90 (m, 5H), 7.63 (d, $J = 8.4$ Hz, 1H), 7.54-7.52 (m, 2H), 7.29 (d, $J = 8.1$ Hz, 1H), 2.70 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.5, 157.9, 136.9, 135.0 (q, $J = 5.0$ Hz), 133.3, 132.9, 128.5, 128.2 (d, $J = 2.0$ Hz), 127.7 (d, $J = 3.0$ Hz), 126.6, 126.3 (d, $J = 8.0$ Hz), 125.4, 122.9, 122.4 (q, $J = 32.0$ Hz), 121.9, 121.4, 24.7; ^{19}F NMR (376 MHz, CDCl_3) δ -56.9; HRMS (ESI) m/z Calculated for $\text{C}_{17}\text{H}_{13}\text{F}_3\text{N}$ $[\text{M}+\text{H}]^+$ 288.0995, found 288.0998.

2-(Biphenyl-4-yl)-6-methyl-3-(trifluoromethyl)pyridine (1f): 82% yield, yellow oil, $R_f = 0.26$ (petroleum ether/ethyl acetate = 30/1); ^1H NMR (400 MHz, CDCl_3) δ 7.95 (d, $J = 8.1$ Hz, 1H), 7.72-7.61 (m, 4H), 7.58 (dd, $J = 7.9, 4.0$ Hz, 2H), 7.45 (td, $J = 7.6, 1.7$ Hz, 2H), 7.40-7.31 (m, 1H), 7.25 (d, $J = 8.1$ Hz, 1H), 2.67 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.5, 157.6, 141.6, 140.8, 138.5, 135.0 (q, $J = 48.0$ Hz), 129.1, 128.8, 127.5, 127.2, 126.8, 125.4, 122.3 (q, $J = 32.0$ Hz), 121.3, 24.7; ^{19}F NMR (376 MHz, CDCl_3) δ -56.9; HRMS (ESI) m/z Calculated for $\text{C}_{19}\text{H}_{15}\text{F}_3\text{N}$ $[\text{M}+\text{H}]^+$ 314.1151, found 314.1155.

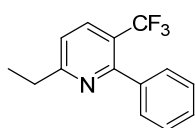


6-Methyl-3-(trifluoromethyl)-2-(4-(trifluoromethyl)phenyl)pyridine (1g): 83% yield, yellow oil, $R_f = 0.50$ (petroleum ether/ethyl acetate = 20/1); ^1H NMR (400 MHz, CDCl_3) δ 7.97 (d, $J = 8.2$ Hz, 1H), 7.71 (d, $J = 8.1$ Hz, 2H), 7.60 (d, $J = 8.1$ Hz, 2H), 7.31 (d, $J = 8.1$ Hz, 1H), 2.67 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.8, 156.4, 142.9, 135.0 (q, $J = 5.0$ Hz), 130.8 (q, $J = 32.0$ Hz), 129.1 (d, $J = 16.0$ Hz), 125.4, 125.0 (q, $J = 4.0$ Hz), 122.7, 122.4 (q, $J = 12.0$ Hz), 122.0, 24.6; ^{19}F NMR (376 MHz, CDCl_3) δ -57.0, -62.8; HRMS (ESI) m/z Calculated for $\text{C}_{14}\text{H}_{10}\text{F}_6\text{N}$ $[\text{M}+\text{H}]^+$ 306.0712, found 306.0727.

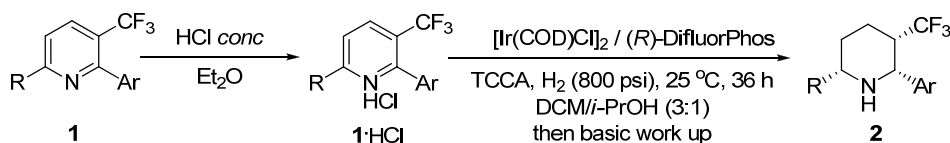
2-(3,5-Difluorophenyl)-6-methyl-3-(trifluoromethyl)pyridine (1h): 77% yield, yellow oil, $R_f = 0.60$ (petroleum ether/ethyl acetate = 30/1); ^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, $J = 8.2$ Hz, 1H), 7.31 (d, $J = 8.1$ Hz, 1H), 7.03 (dd, $J = 7.8, 1.9$ Hz, 2H), 6.89 (tt, $J = 8.9, 2.3$ Hz, 1H), 2.67 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.5 (dd, $J = 247.0, 13.0$ Hz), 161.8, 155.3, 142.2 (t, $J = 9.6$ Hz), 135.1 (q, $J = 4.9$ Hz), 123.6 (q, $J = 272.0$ Hz), 122.3 (q, $J = 32.0$ Hz), 122.2, 112.2 (m, 1C), 104.2 (t, $J = 25.0$ Hz), 24.6. ^{19}F NMR (376 MHz, CDCl_3) δ -57.2, -109.9; HRMS (ESI) m/z Calculated for $\text{C}_{13}\text{H}_9\text{F}_5\text{N}$ $[\text{M}+\text{H}]^+$ 274.0650, found 274.0642.



6-Ethyl-2-phenyl-3-(trifluoromethyl)pyridine (1i): 73% yield, yellow oil, $R_f = 0.50$ (petroleum ether/ethyl acetate = 20/1); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.94 (d, $J = 8.2$ Hz, 1H), 7.50 (d, $J = 4.7$ Hz, 2H), 7.46-7.38 (m, 3H), 7.24 (d, $J = 8.1$ Hz, 1H), 2.92 (q, $J = 7.6$ Hz, 2H), 1.34 (td, $J = 7.6, 0.6$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.5, 157.9 (d, $J = 2.0$ Hz), 139.7, 135.1 (q, $J = 5.0$ Hz), 128.7 (d, $J = 2.0$ Hz), 128.6, 127.9, 125.4, 122.2 (q, $J = 32.0$ Hz) 112.0, 31.5, 13.7; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -56.9; HRMS (ESI) m/z Calculated for $\text{C}_{14}\text{H}_{13}\text{F}_3\text{N}$ $[\text{M}+\text{H}]^+$ 252.0995, found 252.1000.



3. General procedure for asymmetric hydrogenation of 6-alkyl-2-aryl-3-(trifluoromethyl)pyridinium salts 1·HCl

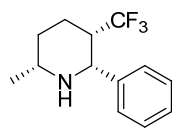


To a stirred solution of the substituted 6-alkyl-2-aryl-3-(trifluoromethyl)pyridine **1** (0.50 g, 2.4 mmol) in ether (10 mL) was added 1.0 mL of HCl *conc.* (or 2 *N* diethylether solution) at room temperature. A white solid formed immediately, and the reaction mixture was stirred at room temperature for around 30 min. All volatiles were removed under reduced pressure to give the corresponding 6-alkyl-2-aryl-3-(trifluoromethyl)pyridineium salt **1·HCl** as a white solid.

In a nitrogen-filled glove box, a mixture of $[\text{Ir}(\text{cod})\text{Cl}]_2$ (2.1 mg, 0.0031 mmol) and (*R*)-DifluorPhos (4.7 mg, 0.0069 mmol) in dichloromethane/isopropanol (3:1, 1.0 mL) was stirred at room temperature for 15-20 min, the mixture was transferred by a syringe to a stainless steel autoclave, in which substrate **1·HCl** (0.20 mmol) and TCCA (2.9 mg, 0.0125 mmol) had been placed beforehand. Dichloromethane/isopropanol (3:1, 2.0 mL) was then added to the mixture. The hydrogenation was performed at 25 °C under 800 psi of hydrogen for 36 h. After carefully releasing the hydrogen, triethylamine (56 μL , 0.40 mmol) was added and the mixture was stirred for 30 min. The organic layer was separated and extracted with dichloromethane twice, and the combined organic extracts were dried over sodium sulfate and concentrated in *vacuo*. Purification was performed on a silica gel column eluted with petroleum ether/ ethyl acetate to give the desired product **2**.

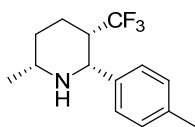
A mixture of benzoyl chloride (42 mg, 0.30 mmol) and triethylamine (56 μL , 0.40 mmol) and **2** dissolved in 3 mL of dichloromethane was stirred at room temperature for 30 min. After concentrating in *vacuo*, the resulting precipitate was directly purified by column chromatography on silica gel using hexanes/ethyl acetate to give the corresponding *N*-4-benzoyl derivatives. The enantiomeric excesses were then determined by chiral HPLC.

(2*R*,3*S*,6*R*)-6-Methyl-2-phenyl-3-(trifluoromethyl)piperidine (2a): 95% yield, pale oil, $R_f = 0.70$ (petroleum ether /ethyl acetate = 1/1), 90% ee, $[\alpha]_D^{20} = + 54.0$ (*c* 0.50, CHCl_3); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.36-7.31 (m, 4H), 7.29-7.23 (m, 1H), 4.11 (s, 1H), 2.99-2.76 (m, 1H), 2.65-2.44 (m, 1H), 2.36-2.17 (m, 1H), 1.99-1.70 (m, 1H), 1.65-1.30 (m, 3H), 1.19 (d, $J = 6.3$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 141.6, 128.2, 127.5 (q, $J = 282.0$ Hz), 127.1, 126.4, 61.3, 53.1, 42.6 (q, $J = 2.0$ Hz), 29.4, 25.5 (q, $J = 3.0$ Hz), 22.8; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -59.2; Enantiomeric excess was determined by HPLC for the corresponding benzamide (OJ-H, elute: Hexanes/*i*-PrOH = 90/10,

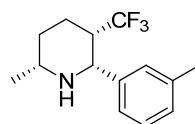


detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 10.6$ min (maj), $t_2 = 15.3$ min; HRMS (ESI) m/z Calculated for $C_{13}H_{17}F_3N$ $[M+H]^+$ 244.1308, found 244.1305.

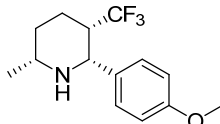
(2R,3S,6R)-6-Methyl-2-*p*-tolyl-3-(trifluoromethyl)piperidine (2b): 95% yield, pale oil, $R_f = 0.60$ (petroleum ether /ethyl acetate = 1/1), 89% ee, $[\alpha]_D^{20} = +44.8$ (c 0.54, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 7.24 (dd, $J = 9.4, 4.5$ Hz, 2H), 7.13 (d, $J = 6.4$ Hz, 2H), 4.09 (s, 1H), 2.85 (s, 1H), 2.52 (s, 1H), 2.30 (dd, $J = 20.0, 8.1$ Hz, 4H), 1.82 (t, $J = 14.2$ Hz, 1H), 1.55 (s, 2H), 1.46-1.31 (m, 1H), 1.19 (dd, $J = 6.2, 2.6$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.6, 136.6, 128.8, 127.5 (q, $J = 282.0$ Hz), 126.3, 61.1, 53.2, 42.6 (q, $J = 22.0$ Hz), 29.4, 25.4 (q, $J = 2.0$ Hz), 22.8, 21.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -59.2; Enantiomeric excess was determined by HPLC for the corresponding benzamide (AD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 11.4$ min, $t_2 = 13.0$ min (maj); HRMS (ESI) m/z Calculated for $C_{14}H_{19}F_3N$ $[M+H]^+$ 258.1464, found 258.1463.



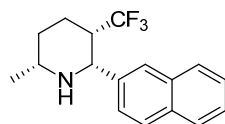
(2R,3S,6R)-6-Methyl-2-*m*-tolyl-3-(trifluoromethyl)piperidine (2c): 84% yield, pale oil, $R_f = 0.61$ (petroleum ether /ethyl acetate = 1/1), 88% ee, $[\alpha]_D^{20} = +50.4$ (c 0.54, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 7.23-7.05 (m, 4H), 4.10 (s, 1H), 2.86 (s, 1H), 2.56-2.53 (m, 1H), 2.35 (s, 3H), 2.31-2.27 (m, 1H), 1.83 (t, $J = 14.2$ Hz, 1H), 1.57 (s, 2H), 1.46-1.31 (m, 1H), 1.19 (d, $J = 6.2, 3H$); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.5, 137.7, 131.7, 128.0, 127.8, 127.5 (q, $J = 282.0$ Hz), 127.0, 61.3, 53.2, 42.6 (q, $J = 22.0$ Hz), 29.5, 25.5 (q, $J = 3.0$ Hz), 22.8, 21.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -59.2; Enantiomeric excess was determined by HPLC for the corresponding benzamide (AD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 7.6$ min, $t_2 = 8.8$ min (maj); HRMS (ESI) m/z Calculated for $C_{14}H_{19}F_3N$ $[M+H]^+$ 258.1464, found 258.1464.



(2R,3S,6R)-2-(4-Methoxyphenyl)-6-methyl-3-(trifluoromethyl)piperidine (2d): 94% yield, pale oil, $R_f = 0.50$ (petroleum ether /ethyl acetate = 1/1), 88% ee, $[\alpha]_D^{20} = +55.5$ (c 0.64, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 7.26 (d, $J = 8.7$ Hz, 2H), 6.85 (d, $J = 8.7$ Hz, 2H), 4.07 (s, 1H), 3.79 (s, 3H), 2.85 (s, 1H), 2.48 (dd, $J = 9.6, 4.5$ Hz, 1H), 2.27 (ddd, $J = 5.9, 3.5, 1.7$ Hz, 1H), 1.88-1.75 (m, 1H), 1.55 (d, $J = 11.9$ Hz, 2H), 1.37 (d, $J = 13.6$ Hz, 1H), 1.18 (d, $J = 6.3$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7, 133.8, 127.5 (q, $J = 282.0$ Hz), 127.4, 113.6, 60.8, 55.2, 53.2, 42.6 (q, $J = 22.0$ Hz), 29.4, 25.4 (q, $J = 2.0$ Hz), 22.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -59.2; Enantiomeric excess was determined by HPLC for the corresponding benzamide (AD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 13.7$ min, $t_2 = 18.5$ min (maj); HRMS (ESI) m/z Calculated for $C_{14}H_{19}F_3NO$ $[M+H]^+$ 274.1413, found 274.1418.

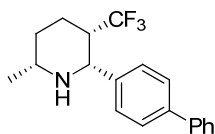


(2R,3S,6R)-6-Methyl-2-(naphthalen-2-yl)-3-(trifluoromethyl)piperidine (2e): 93% yield, pale oil, $R_f = 0.65$ (petroleum ether /ethyl acetate = 1/1), 89% ee, $[\alpha]_D^{20} = +75.3$ (c 0.68, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 7.82 (dd, $J = 13.2, 7.7$ Hz, 4H), 7.45 (dd, $J = 11.5, 5.6$ Hz, 3H), 4.29 (s, 1H), 2.93 (s, 1H), 2.70-2.67 (m, 1H), 2.33 (d, $J = 14.4$ Hz, 1H), 1.90 (t, $J = 13.7$ Hz, 1H), 1.72-1.56 (m, 2H), 1.53-1.27 (m, 1H), 1.26 (d, $J = 6.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 133.4, 132.7, 128.9, 128.0, 127.6 (d, $J = 7.5$ Hz), 127.5 (q, $J = 282.0$ Hz), 126.0, 125.7, 125.0, 124.7, 61.3, 53.2, 42.5 (q, $J = 22.0$ Hz), 29.5, 25.5 (q, $J = 2.0$ Hz), 22.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -59.2; Enantiomeric excess was determined by HPLC for the corresponding benzamide (AD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 =$

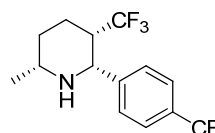


13.0 min, $t_2 = 16.8$ min (maj); HRMS (ESI) m/z Calculated for $C_{17}H_{18}F_3N$ $[M+H]^+$ 294.1464, found 294.1472.

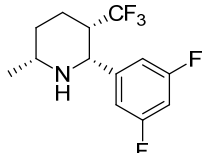
(2*R*,3*S*,6*R*)-2-(Biphenyl-4-yl)-6-methyl-3-(trifluoromethyl)piperidine (2f): 90% yield, pale solid, mp = 135-136 °C, $R_f = 0.62$ (petroleum ether /ethyl acetate = 1/1), 87% ee, $[\alpha]_D^{20} = +55.5$ (c 0.70, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 7.60-7.53 (m, 4H), 7.43-7.40 (m, 4H), 7.32 (t, $J = 7.3$ Hz, 1H), 4.16 (s, 1H), 2.87 (s, 1H), 2.63-2.49 (m, 1H), 2.36-2.24 (m, 1H), 1.85 (t, $J = 14.1$ Hz, 1H), 1.67-1.53 (m, 2H), 1.42 (t, $J = 12.4$ Hz, 1H), 1.20 (d, $J = 6.3$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.9, 140.7, 140.0, 128.8, 128.7, 127.5 (q, $J = 282.0$ Hz) 127.2, 127.0, 126.9, 61.1, 53.2, 42.6 (q, $J = 22.0$ Hz), 29.4, 25.4 (q, $J = 2.0$ Hz), 22.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -59.1; Enantiomeric excess was determined by HPLC for the corresponding benzamide (AD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 13.0$ min, $t_2 = 18.3$ min (maj); HRMS (ESI) m/z Calculated for $C_{19}H_{20}F_3N$ $[M+H]^+$ 320.1621, found 320.1620.



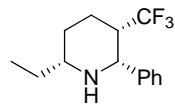
(2*R*,3*S*,6*R*)-6-Methyl-3-(trifluoromethyl)-2-(4-(trifluoromethyl)phenyl)piperidine (2g): 90% yield, pale oil, $R_f = 0.48$ (petroleum ether /ethyl acetate = 1/1), 86% ee, $[\alpha]_D^{20} = +57.8$ (c 0.58, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, $J = 8.3$ Hz, 2H), 7.48 (d, $J = 8.2$ Hz, 2H), 4.18 (s, 1H), 2.90-2.85 (m, 1H), 2.57-2.54 (m, 1H), 2.34-2.28 (m, 1H), 1.94-1.77 (m, 1H), 1.65-1.57 (m, 2H), 1.47-1.31 (m, 1H), 1.20 (d, $J = 6.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.8 (d, $J = 1.0$ Hz), 129.4 (t, $J = 32.0$ Hz), 127.4 (q, $J = 281.0$ Hz), 127.0 (d, $J = 1.0$ Hz), 125.3 (q, $J = 4.0$ Hz), 124.3 (q, $J = 271.0$ Hz), 61.0, 53.2, 42.6 (q, $J = 22.0$ Hz), 29.4, 25.5 (q, $J = 3.0$ Hz), 22.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ -59.2, -62.5; Enantiomeric excess was determined by HPLC for the corresponding benzamide (AD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 7.5$ min, $t_2 = 14.0$ min (maj); HRMS (ESI) m/z Calculated for $C_{14}H_{16}F_6N$ $[M+H]^+$ 312.1187, found 312.1179.



(2*R*,3*S*,6*R*)-2-(3,5-Difluorophenyl)-6-methyl-3-(trifluoromethyl)piperidine (2h): 72% yield, pale oil, $R_f = 0.61$ (petroleum ether /ethyl acetate = 1/1), 87% ee, $[\alpha]_D^{20} = +62.5$ (c 0.36, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 6.95-6.87 (m, 2H), 6.71-6.66 (m, 1H), 4.10 (d, $J = 1.1$ Hz, 1H), 2.86-2.82 (m, 1H), 2.54-2.49 (m, 1H), 2.37-2.23 (m, 1H), 1.92-1.73 (m, 1H), 1.63-1.52 (m, 1H), 1.44-1.35 (m, 2H), 1.19 (d, $J = 6.3$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0 (dd, $J = 246.0, 13.0$ Hz), 145.8 (t, $J = 9.0$ Hz), 127.2 (q, $J = 282.0$ Hz), 109.4 (dd, $J = 18.0, 7.0$ Hz), 102.5 (t, $J = 25.0$ Hz), 60.4, 52.9, 42.4 (q, $J = 3.0$ Hz), 29.2, 25.2 (q, $J = 3.0$ Hz), 22.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ -59.4, 110.2; Enantiomeric excess was determined by HPLC for the corresponding benzamide (OD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 3.9$ min (maj), $t_2 = 4.7$ min; HRMS (ESI) m/z Calculated for $C_{13}H_{15}F_5N$ $[M+H]^+$ 280.1119, found 280.1124.

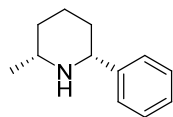


(2*R*,3*S*,6*R*)-6-Ethyl-2-phenyl-3-(trifluoromethyl)piperidine (2i): 82% yield, pale oil, $R_f = 0.64$ (petroleum ether /ethyl acetate = 1/1), 87% ee, $[\alpha]_D^{20} = +52.1$ (c 0.48, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 7.38-7.30 (m, 4H), 7.28-7.21 (m, 1H), 4.11 (s, 1H), 2.65-2.52 (m, 2H), 2.32-2.27 (m, 1H), 1.85-1.39 (m, 5H), 0.96 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.7, 128.2, 127.5 (q, $J = 282.0$ Hz), 127.1, 126.4 (d, $J = 1.0$ Hz), 61.3, 59.1, 43.1 (q, $J = 22.0$ Hz), 29.8, 27.1, 25.4 (q, $J = 2.0$ Hz), 10.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ -59.3; Enantiomeric excess was determined by HPLC for



the corresponding benzamide (AD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 7.5$ min, $t_2 = 8.8$ min (maj); HRMS (ESI) m/z Calculated for $C_{14}H_{19}F_3N$ $[M+H]^+$ 258.1464, found 258.1473.

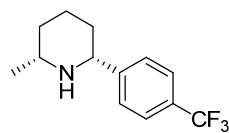
(2*R*,6*R*)-2-Methyl-6-phenylpiperidine (4a): 89% conv., pale oil, $R_f = 0.16$ (Dichloromethane/MeOH = 15/1), 78% ee, $[\alpha]_D^{20} = +38.0$ (c 0.20, $CHCl_3$), Lit:^[6] ((+)-(2*R*, 6*R*):



$[\alpha]_D^{20} = +22.17$ (c 0.69, EtOH)^[6a]; (-)-(2*S*, 6*S*): $[\alpha]_D^{25} = -22.4$ (c 0.80, $CHCl_3$) (for an ee of 80%)^[6b]; 1H NMR (400 MHz, $CDCl_3$) δ 7.39-7.32 (m, 2H), 7.30 (dd, $J = 8.1, 6.7$ Hz, 2H), 7.24 (dd, $J = 4.9, 3.6$ Hz, 1H), 3.66 (dd, $J = 10.7, 2.4$

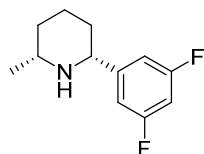
Hz, 1H), 2.84-2.77 (m, 1H), 1.93-1.80 (m, 2H), 1.80-1.69 (m, 1H), 1.64 (dd, $J = 8.5, 4.5$ Hz, 1H), 1.55-1.41 (m, 2H), 1.18-1.15 (m, 1H), 1.11 (d, $J = 6.3$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.5, 128.5, 127.2, 126.9, 62.7, 53.4, 34.3, 34.0, 25.5, 23.2; Enantiomeric excess was determined by HPLC for the corresponding benzamide (OJ-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 11.2$ min, $t_2 = 17.2$ min (maj).

(2*R*,6*R*)-2-Methyl-6-(4-(trifluoromethyl)phenyl)piperidine (4b): 83% conv., pale oil, $R_f = 0.10$ (ethyl acetate), 79% ee, $[\alpha]_D^{20} = +17.4$ (c 0.46, $CHCl_3$), Lit:^[6b] ((-)-(2*S*, 6*S*): $[\alpha]_D^{25} = -15.3$ (c 0.77, $CHCl_3$) (for an ee of 55%)^[6b]; 1H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, $J = 8.3$ Hz, 2H), 7.50 (d, $J = 8.2$ Hz, 2H), 3.73 (dd, $J = 10.9, 2.3$ Hz, 1H),



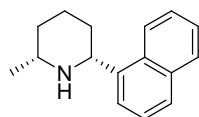
2.86-2.78 (m, 1H), 1.95-1.62 (m, 4H), 1.59-1.35 (m, 2H), 1.24-1.15 (m, 1H), 1.12 (d, $J = 6.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.6, 129.4 (q, $J = 32.0$ Hz), 127.3, 125.4 (q, $J = 4.0$ Hz), 124.4 (q, $J = 270.0$ Hz), 62.2, 53.2, 34.6, 33.8, 25.4, 23.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.4; Enantiomeric excess was determined by HPLC for the corresponding benzamide (OG, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 8.3$ min, $t_2 = 10.7$ min (maj).

(2*R*,6*R*)-2-(3,5-Difluorophenyl)-6-methylpiperidine (4c): [CAS: 1341965-51-2]; 93% conv., pale oil, $R_f = 0.30$ (ethyl acetate), 79% ee, $[\alpha]_D^{20} = +27.1$ (c 0.42, $CHCl_3$); 1H NMR (400



MHz, $CDCl_3$) δ 6.99-6.85 (m, 2H), 6.68-6.63 (m, 1H), 3.64 (dd, $J = 11.0, 2.1$ Hz, 1H), 2.82-2.75 (m, 1H), 1.94-1.58 (m, 4H), 1.56-1.28 (m, 2H), 1.20-1.07 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2 (dd, $J = 246.0, 13.0$ Hz), 109.7 (d, $J = 6.0$), 109.5 (d, $J = 6.0$), 102.3 (t, $J = 25.4$ Hz), 61.8 (t, $J = 2.0$ Hz), 53.1, 34.5, 33.8, 25.3, 23.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ -110.3; Enantiomeric excess was determined by HPLC for the corresponding benzamide (OJ-H, elute: Hexanes/*i*-PrOH = 95/5, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 11.4$ min, $t_2 = 13.1$ min (maj).

(2*R*,6*R*)-2-Methyl-6-(naphthalen-1-yl)piperidine (4d): [CAS: 1488821-66-4]; >95% conv., pale oil, $R_f = 0.15$ (ethyl acetate), 64% ee, $[\alpha]_D^{20} = +21.7$ (c 0.48, $CHCl_3$); 1H NMR (400



MHz, $CDCl_3$) δ 8.17 (d, $J = 8.3$ Hz, 1H), 7.88 (d, $J = 8.0$ Hz, 1H), 7.76 (d, $J = 7.7$ Hz, 2H), 7.59-7.40 (m, 3H), 4.48 (d, $J = 10.2$ Hz, 1H), 3.02-2.97 (m, 1H), 2.20-1.89 (m, 3H), 1.83-1.52 (m, 3H), 1.37-1.23 (m, 1H), 1.19 (d, $J = 6.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.0, 134.0, 131.0, 129.2, 127.5, 126.0, 126.0, 125.5, 123.3, 123.1, 57.9, 53.9, 34.3, 33.6, 25.8, 23.3; Enantiomeric excess was determined by HPLC for the corresponding benzamide (OD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 220 nm, flow rate: 1.0 mL/min), 30 °C, $t_1 = 8.3$ min (maj), $t_2 = 10.0$ min.

4. The determination of the absolute configuration of **2f**

The absolute configuration of hydrogenation product 2-(biphenyl-4-yl)-6-methyl-3-(trifluoromethyl)piperidine **2f** [87% ee, $[\alpha]_D^{20} = +55.5$ (c 0.70, CHCl_3)] was determined by X-ray diffraction analysis by recrystallization from mixture solvent of dichloromethane/*n*-hexane to upgrade ee to >99%. The configurations of the other chiral products are assigned by analogy. CCDC 1009006 contains the structure and supplementary crystallographic data for the structure of (2*R*,3*S*,6*R*)-2-(biphenyl-4-yl)-6-methyl-3-(trifluoromethyl)piperidine **2f**. These data can be obtained free of charge *via* www.ccdc.com.ac.uk/data_request/cif from the Cambridge Crystallographic Data Centre.

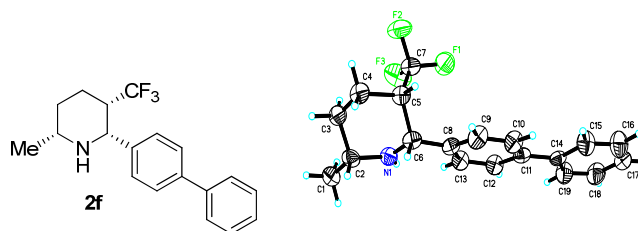


Figure 1. The X-ray structure of (2*R*,3*S*,6*R*)-2-(biphenyl-4-yl)-6-methyl-3-(trifluoromethyl)piperidine **2f**.

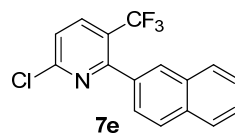
5. References

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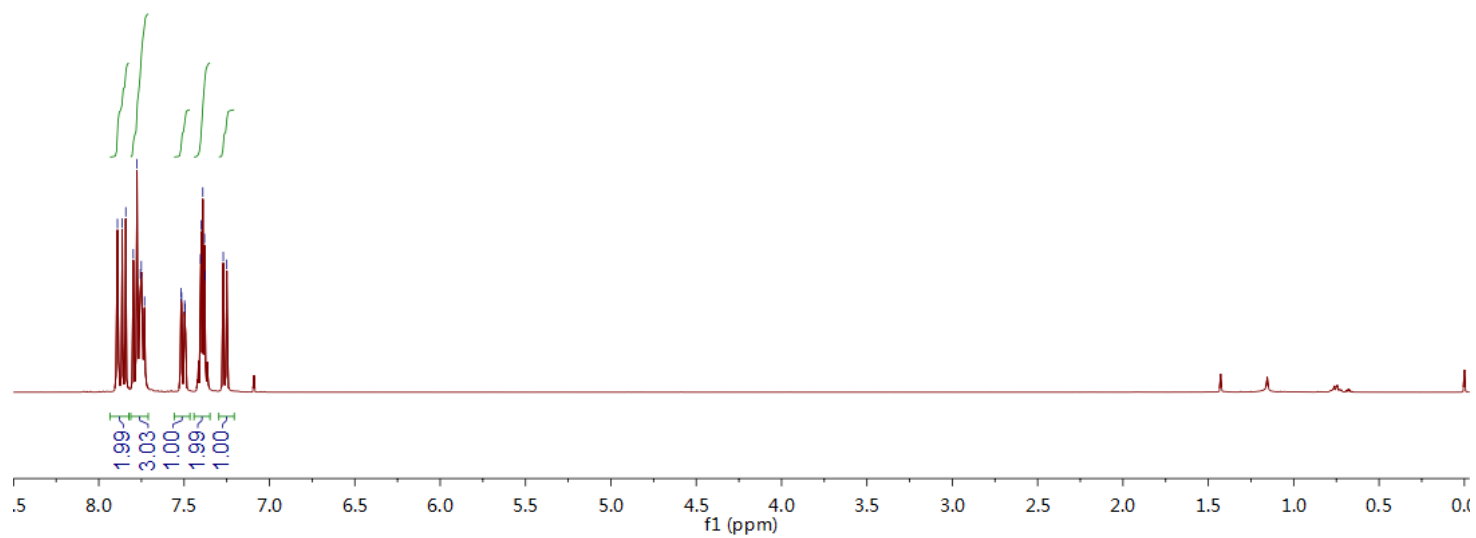
6.1 Copy of NMR for trifluoromethyl pyridines

7.8904
7.8625
7.8415
7.7979
7.7760
7.7560
7.7506
7.7319
7.7180
7.5148
7.4968
7.4937
7.4039
7.3995
7.3902
7.3804
7.3768
7.2706
7.2499

¹H NMR MC-9-44A in CDCl₃

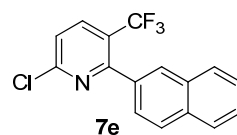
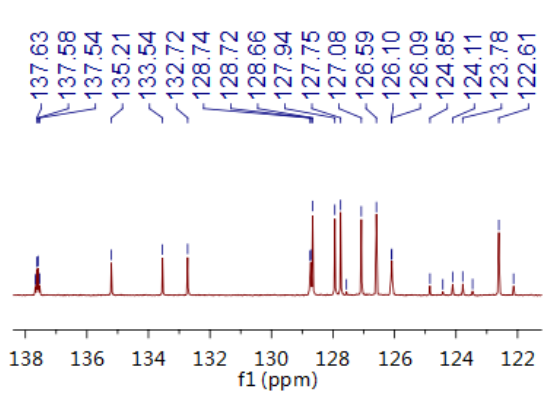


¹H NMR (400 MHz, CDCl₃)

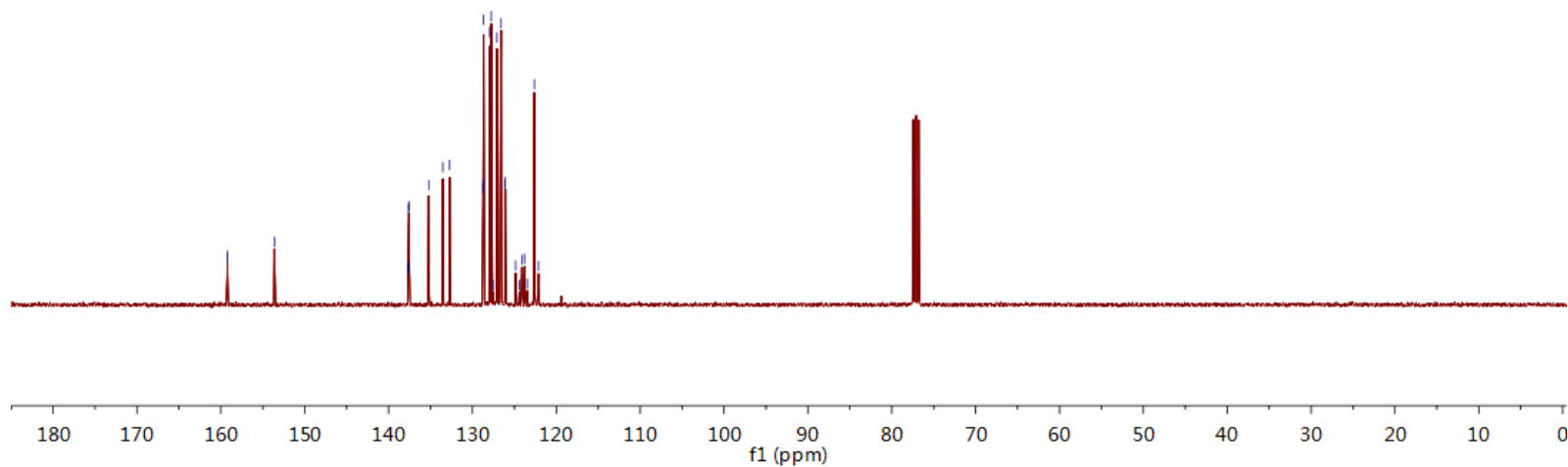




¹³C NMR MC-9-44A in CDCl₃

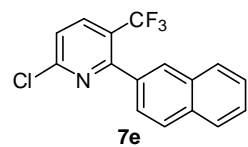


¹³C NMR (100 MHz, CDCl₃)

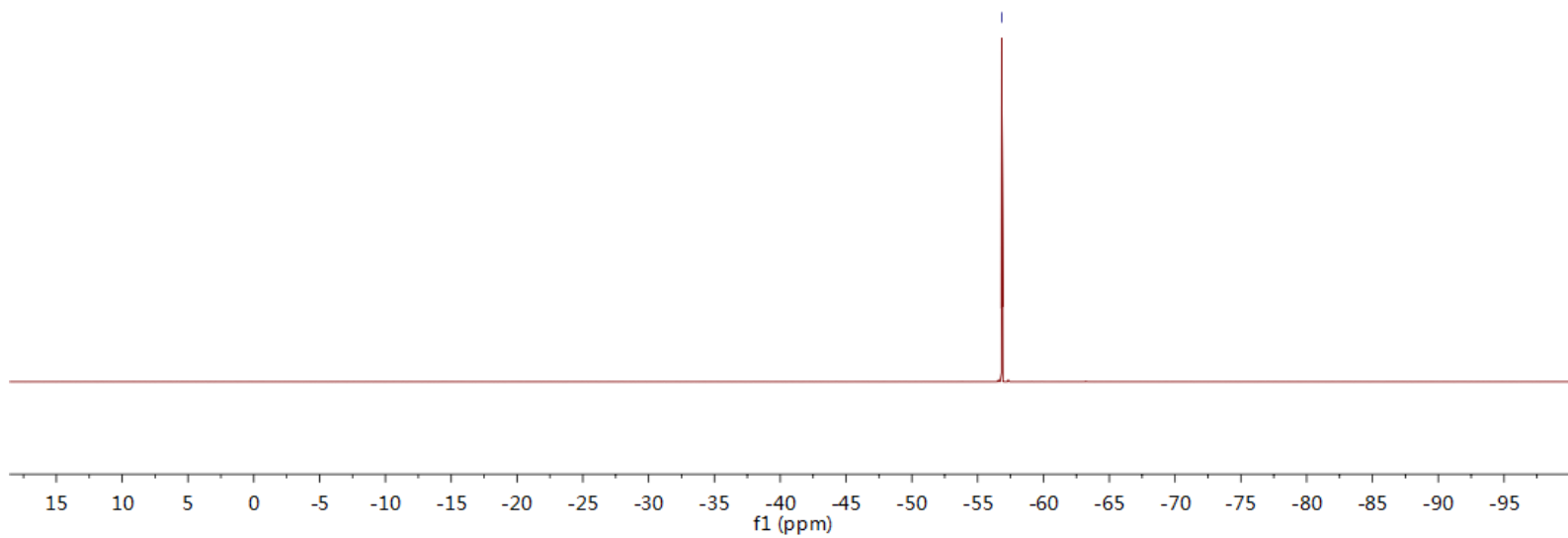


¹⁹F NMR MC-9-44A in CDCl₃

—56.8216

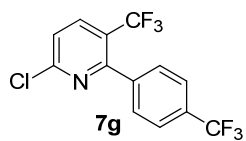


¹⁹F NMR (376 MHz, CDCl₃)

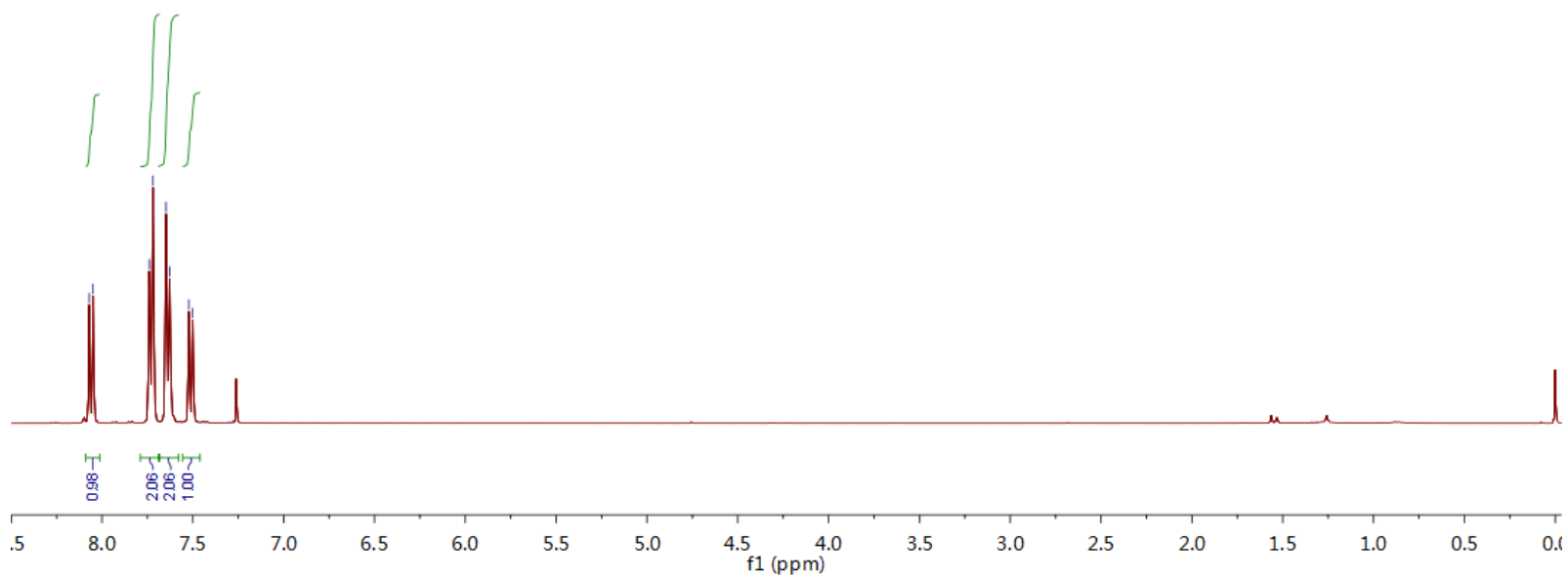


8.0705
8.0496
7.7400
7.7195
7.6475
7.6272
7.5217
7.5008

¹H NMR MC-9-83 in CDCl₃

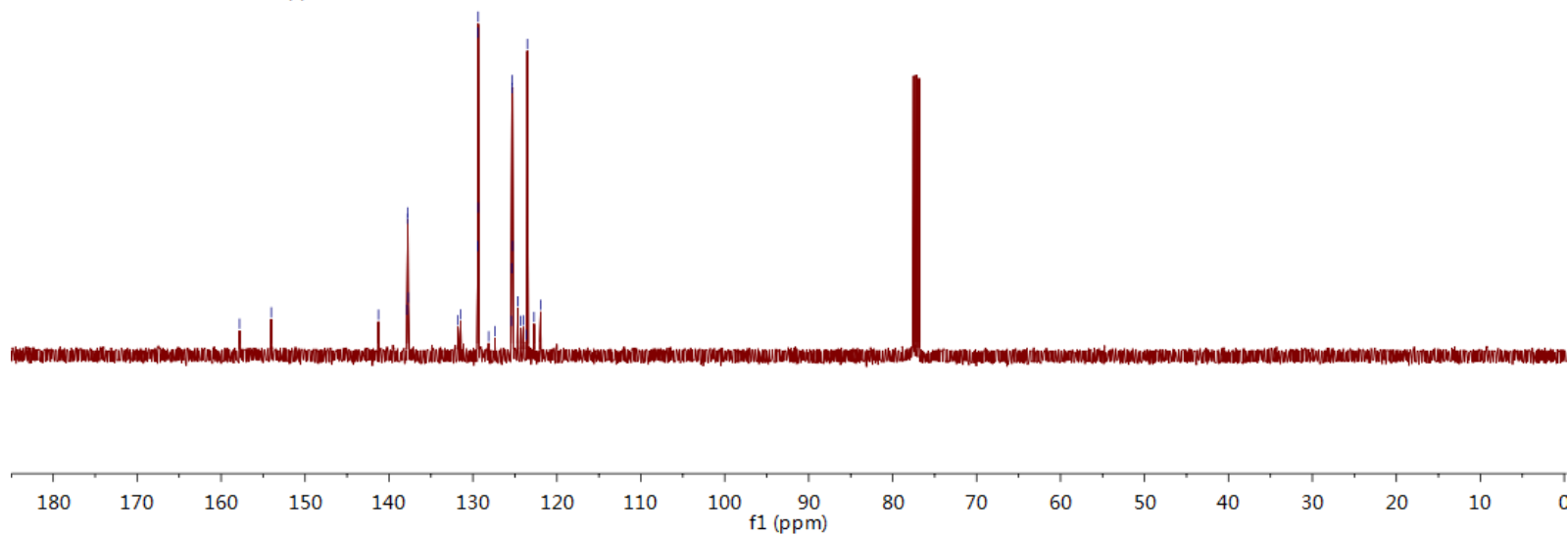
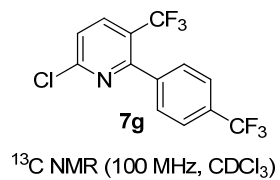
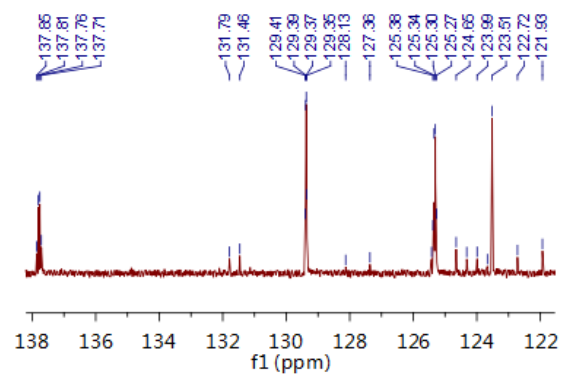


¹H NMR (400 MHz, CDCl₃)



157.80
 154.03
 141.25
 137.85
 137.81
 137.76
 137.71
 131.79
 131.46
 129.41
 129.39
 129.37
 128.35
 128.13
 127.36
 125.38
 125.34
 125.30
 125.27
 124.65
 123.90
 123.51
 122.72
 121.93
 127.98
 126.42
 125.36
 125.34
 125.30
 125.27
 124.65
 124.32
 123.90
 123.66
 123.51
 122.72
 121.93

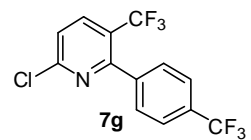
¹³C NMR MC-9-83 in CDCl₃



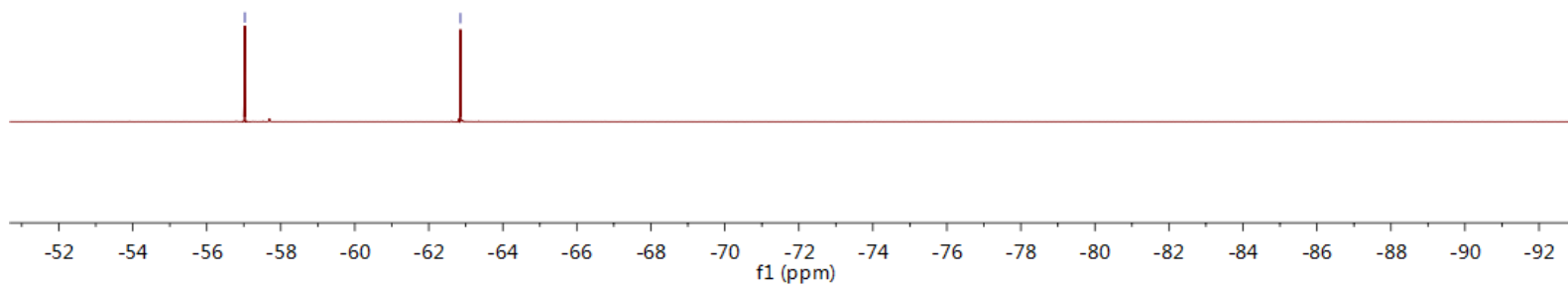
^{19}F NMR MC-9-83 in CDCl_3

-57.0291

-62.8558

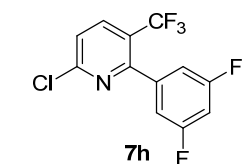


^{19}F NMR (376 MHz, CDCl_3)

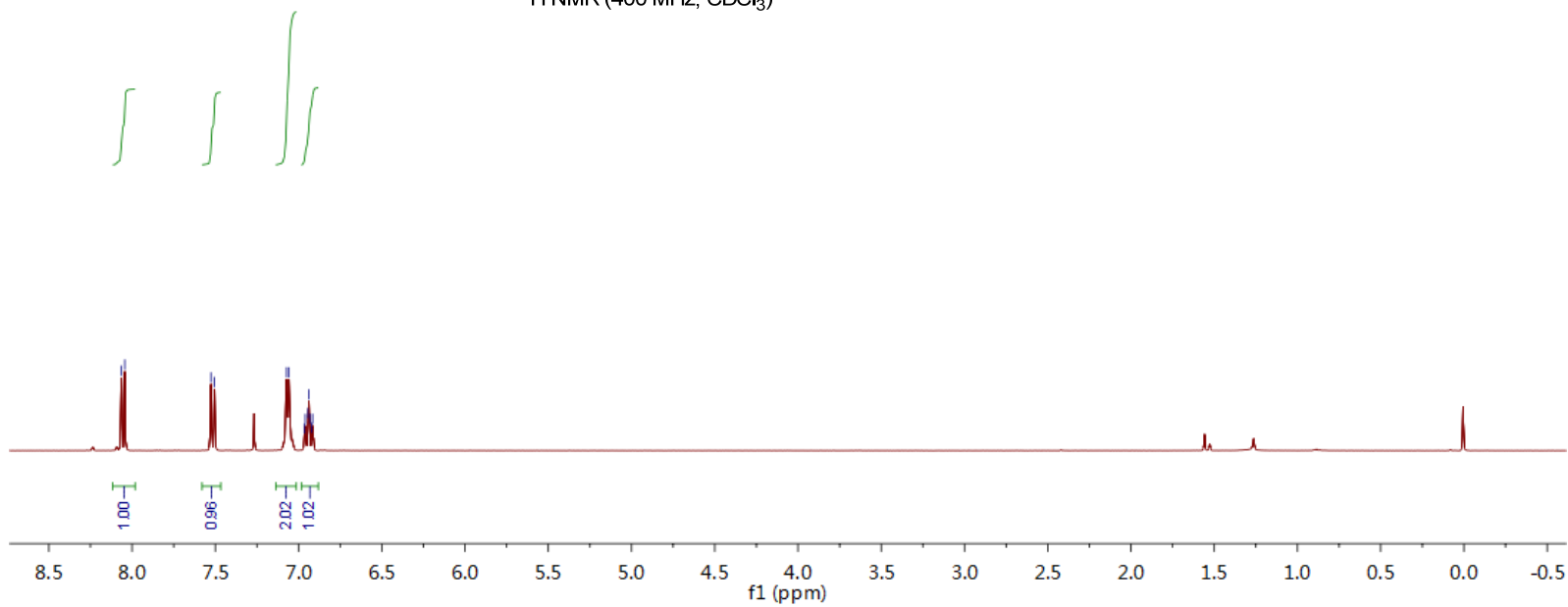


8.0641
8.0432
7.5262
7.5053
7.0720
7.0574
6.9654
6.9598
6.9433
6.9377
6.9321
6.9212
6.9156

¹H NMR MC-9-34B in CDCl₃



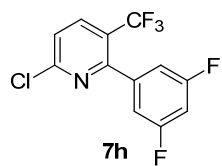
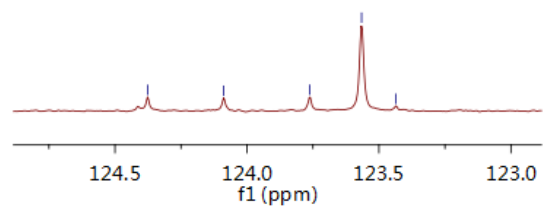
¹H NMR (400 MHz, CDCl₃)



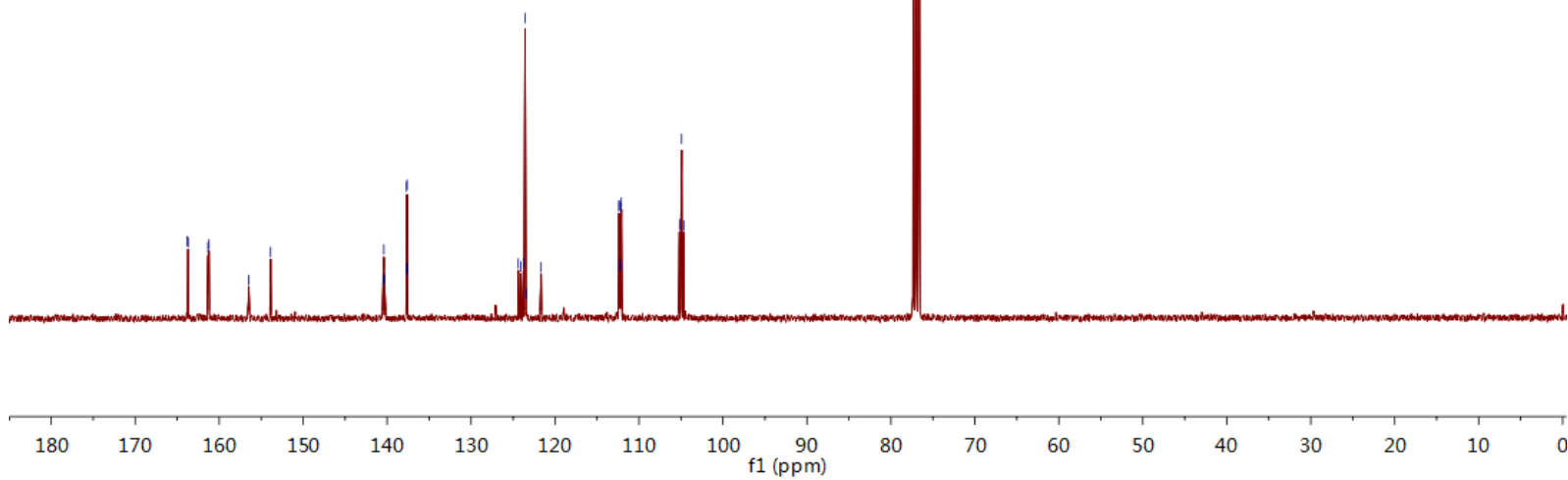
163.82
163.69
161.34
161.21
156.48
153.88
140.39
137.73
137.68
137.63
137.59
124.38
124.09
123.76
123.57
112.37
105.18
104.93
104.68

¹³C NMR MC-9-34B in CDCl₃

124.38
124.09
123.76
123.57
123.44



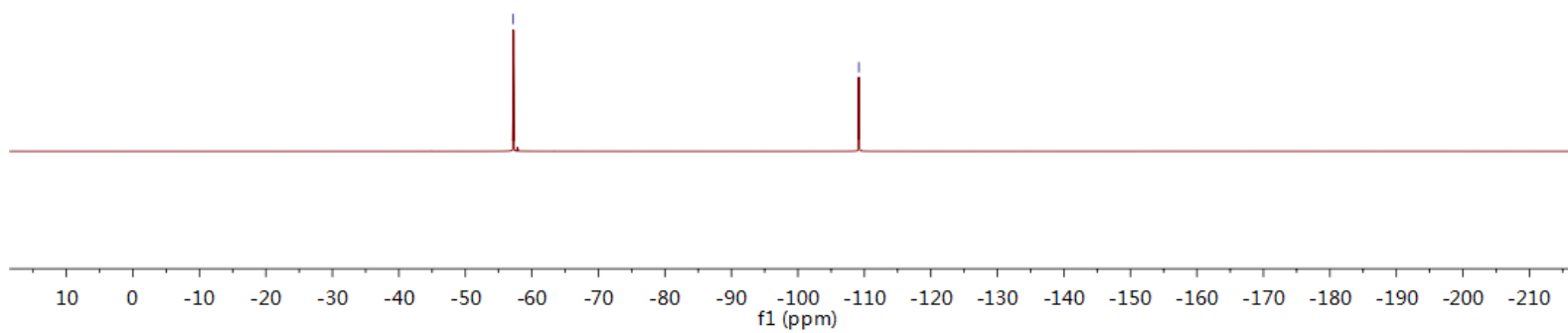
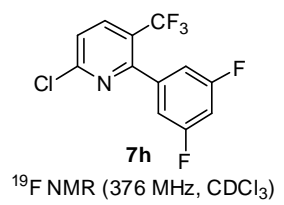
¹³C NMR (100 MHz, CDCl₃)



¹⁹F NMR MC-9-34B in CDCl₃

---57.1891

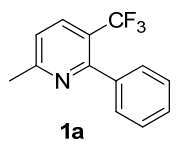
---109.1754



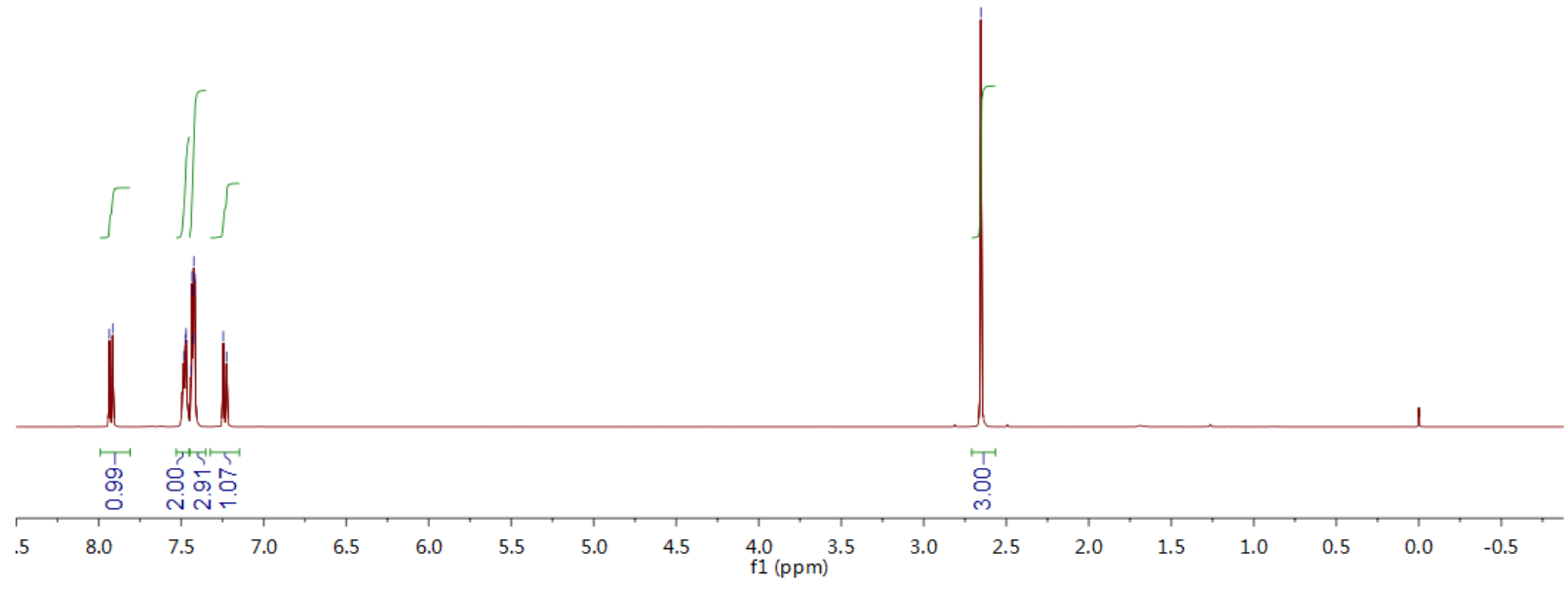
7.9343
7.9139
7.4834
7.4744
7.4690
7.4402
7.4331
7.4310
7.4269
7.4227
7.4162
7.2440
7.2240

2.6536

¹H NMR MC-9-36A in CDCl₃

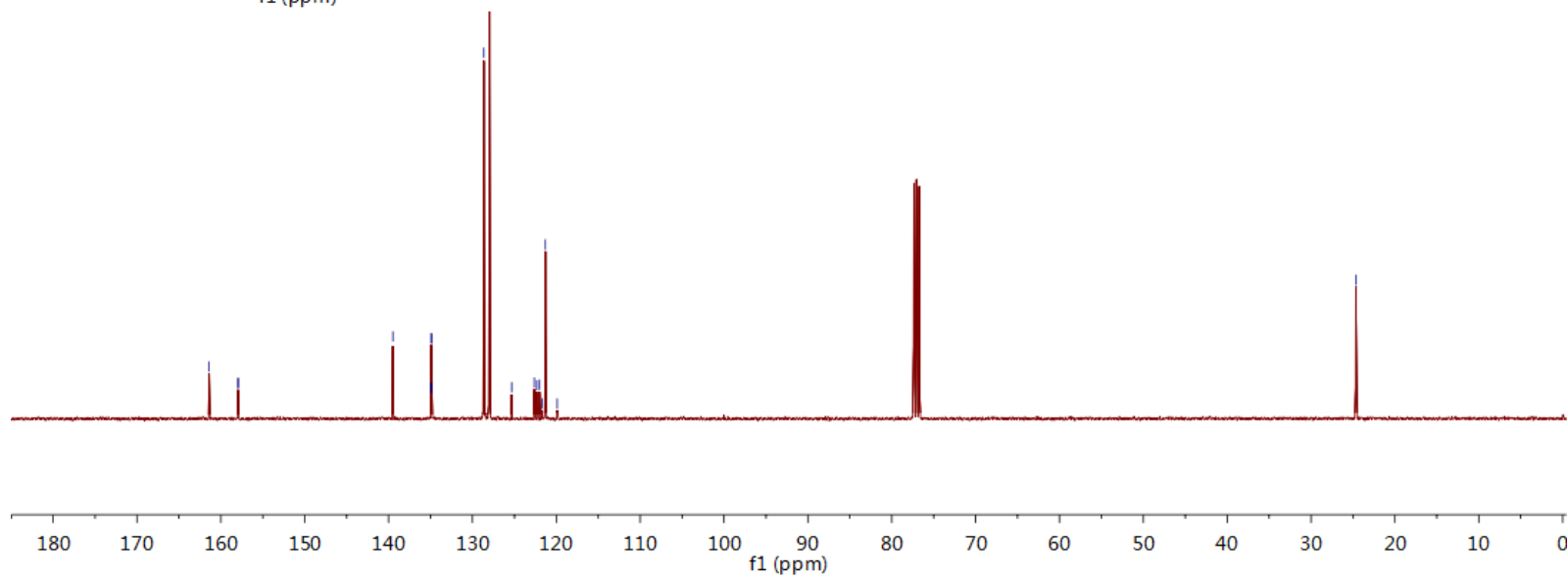
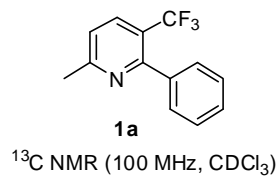
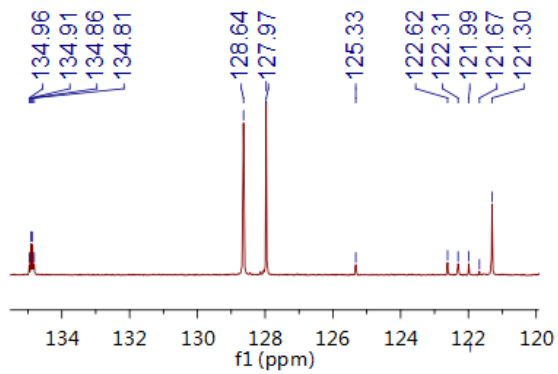


¹H NMR (400 MHz, CDCl₃)



161.41
157.95
157.93
139.49
134.96
134.91
134.86
134.81
128.64
127.97
125.33
122.62
122.31
121.99
121.67
121.30
119.90

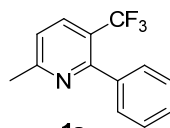
¹³C NMR MC-9-36A in CDCl₃



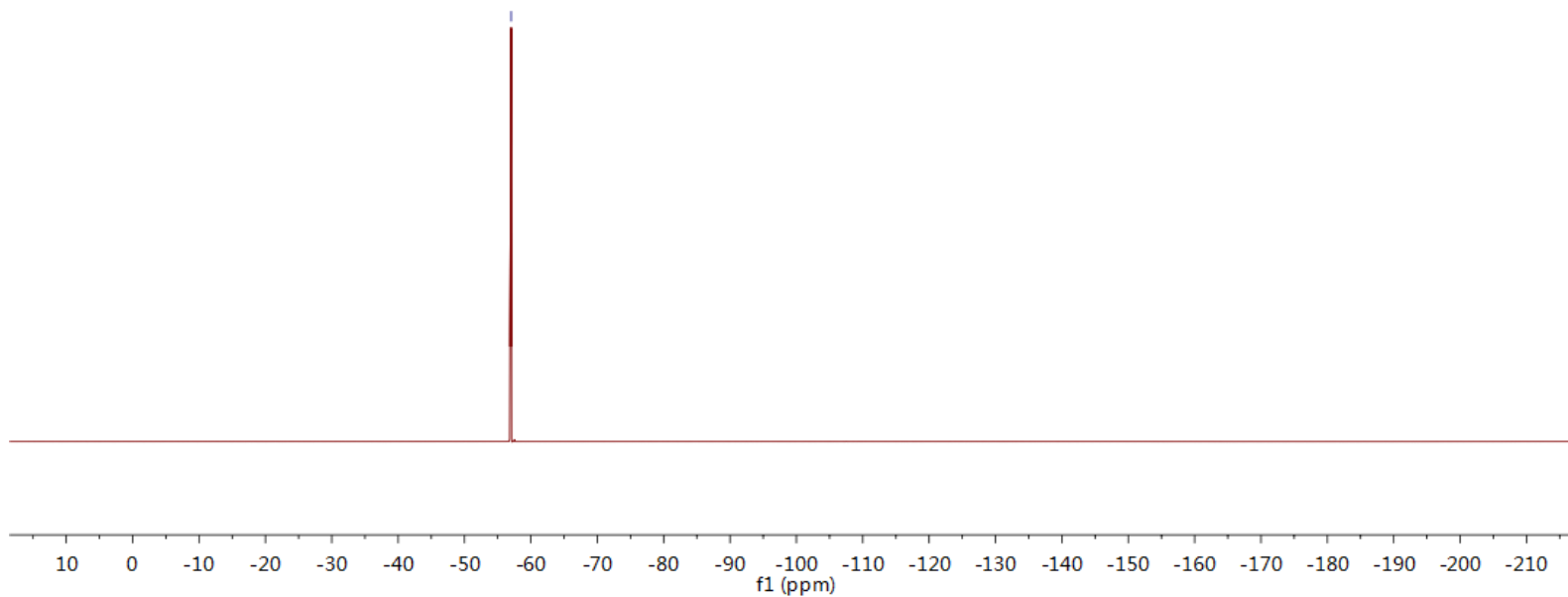
—24.65

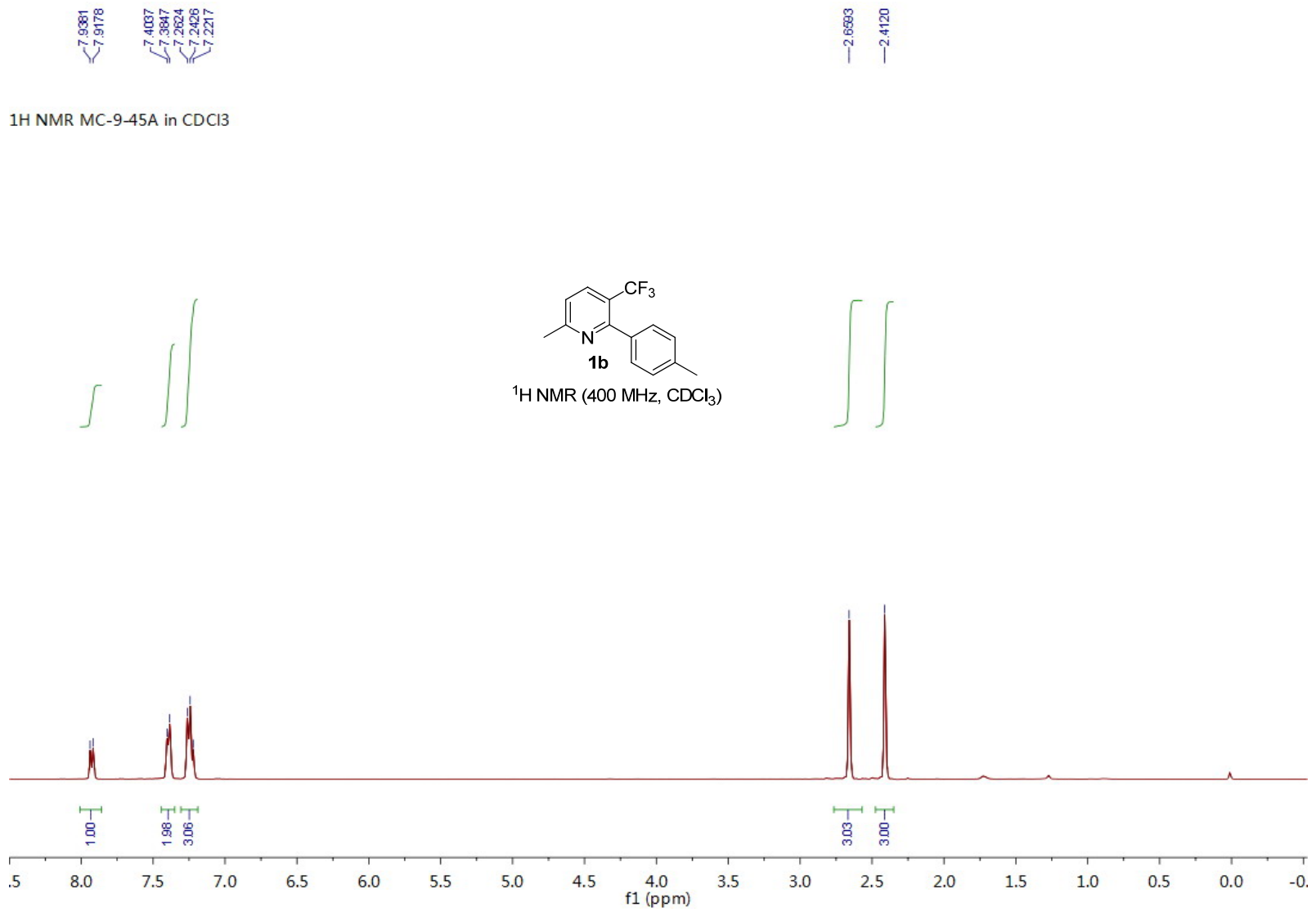
¹⁹F NMR MC-9-36A in CDCl₃

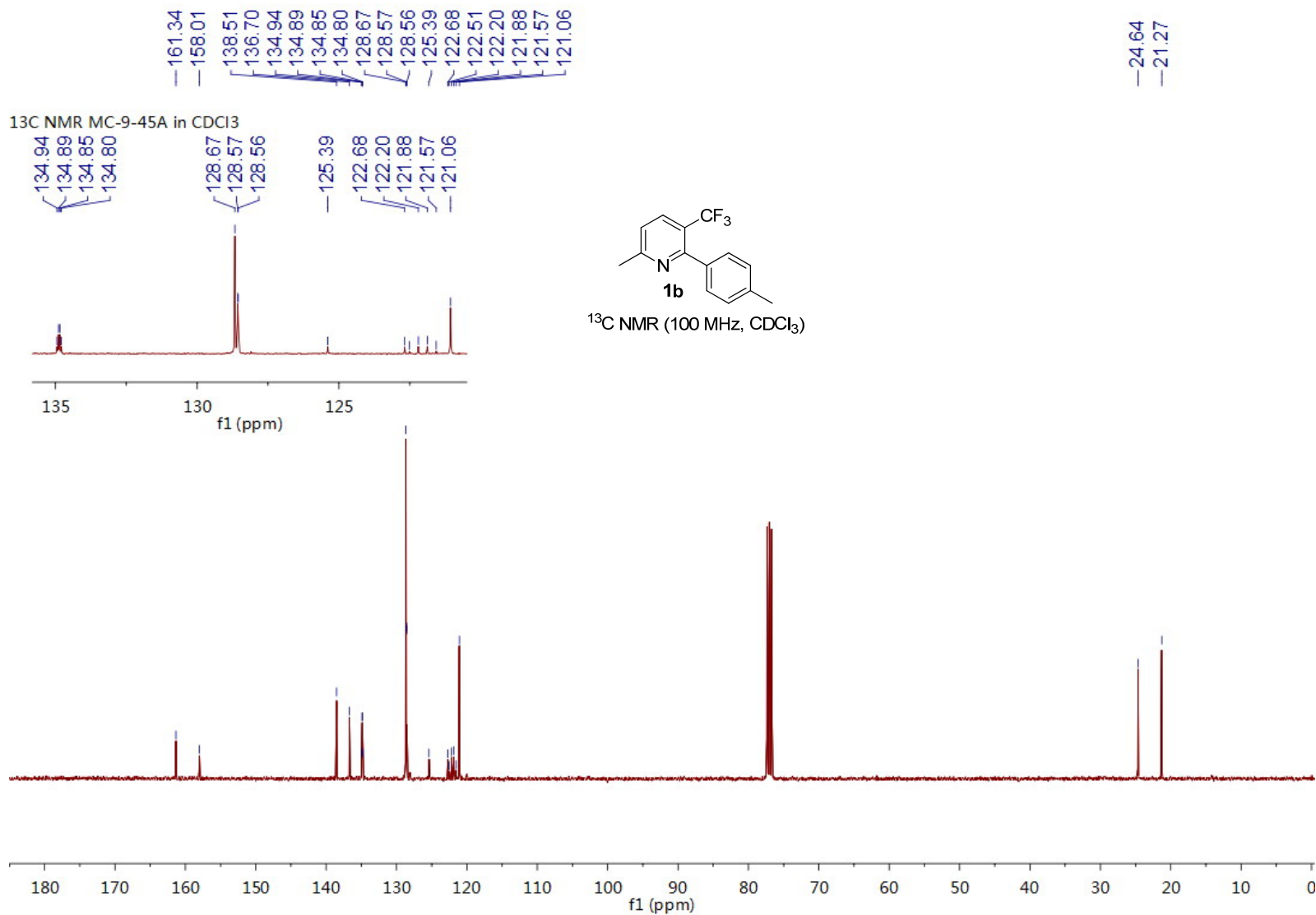
-57.0290



¹⁹F NMR (376 MHz, CDCl₃)

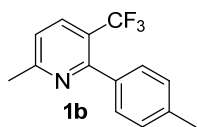




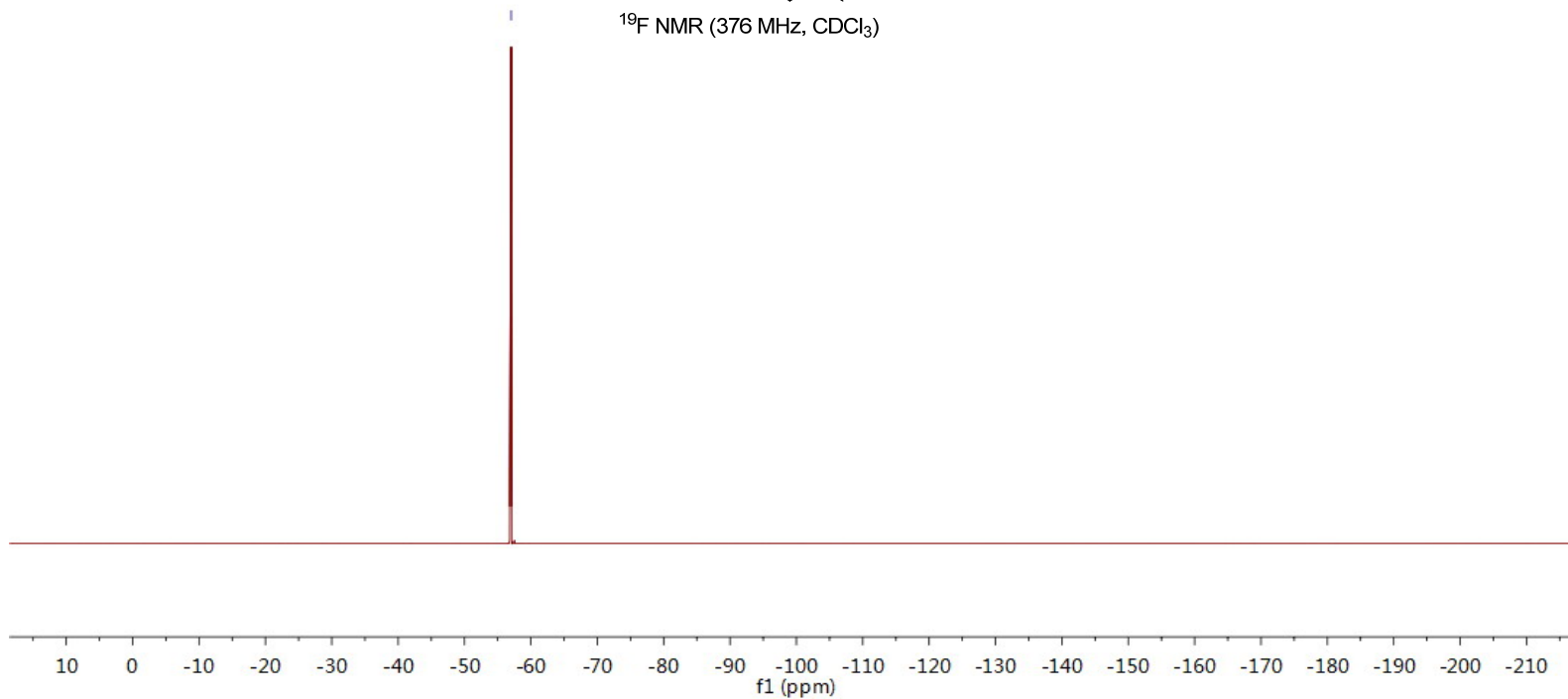


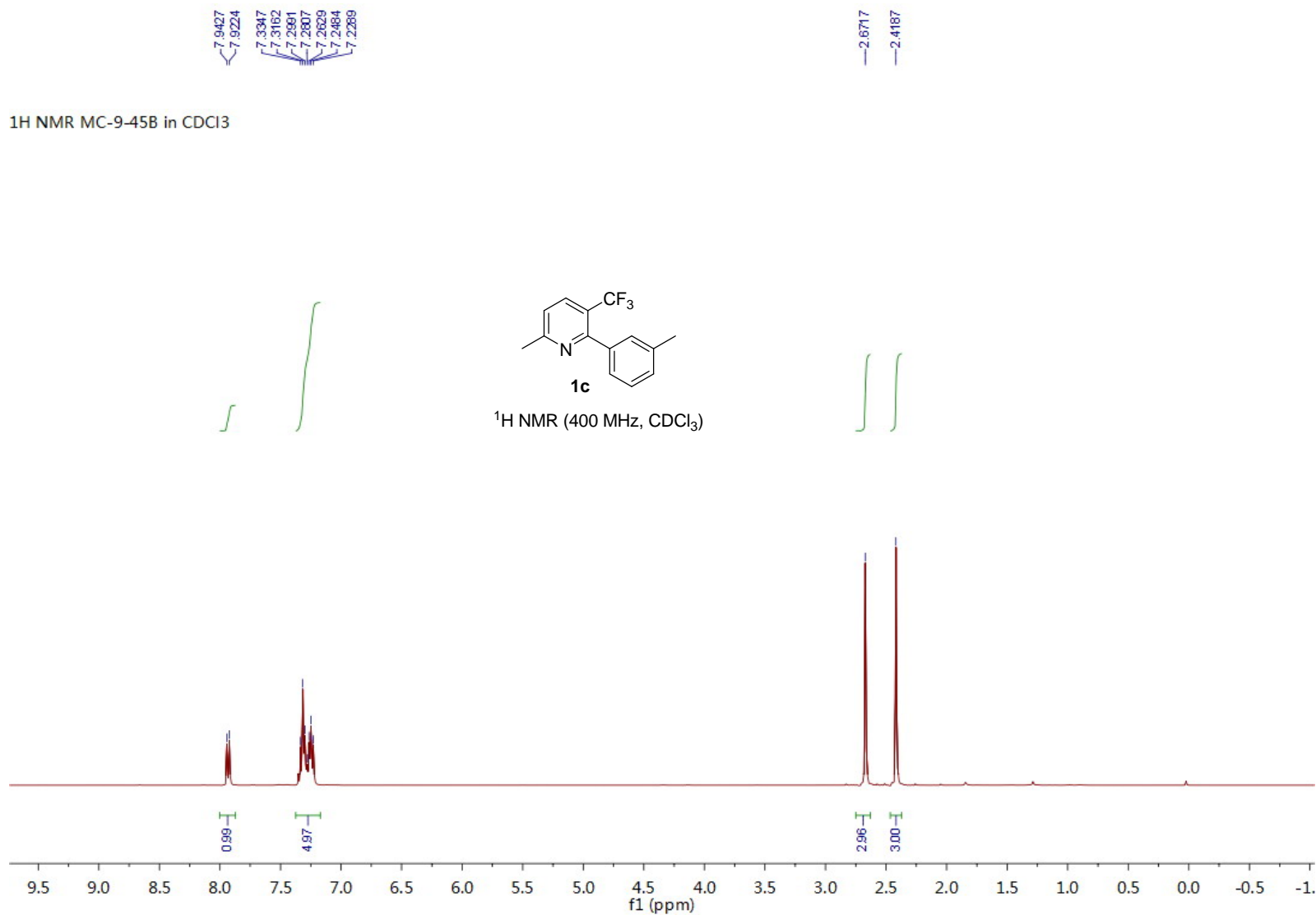
19F NMR MC-9-45A in CDCl3

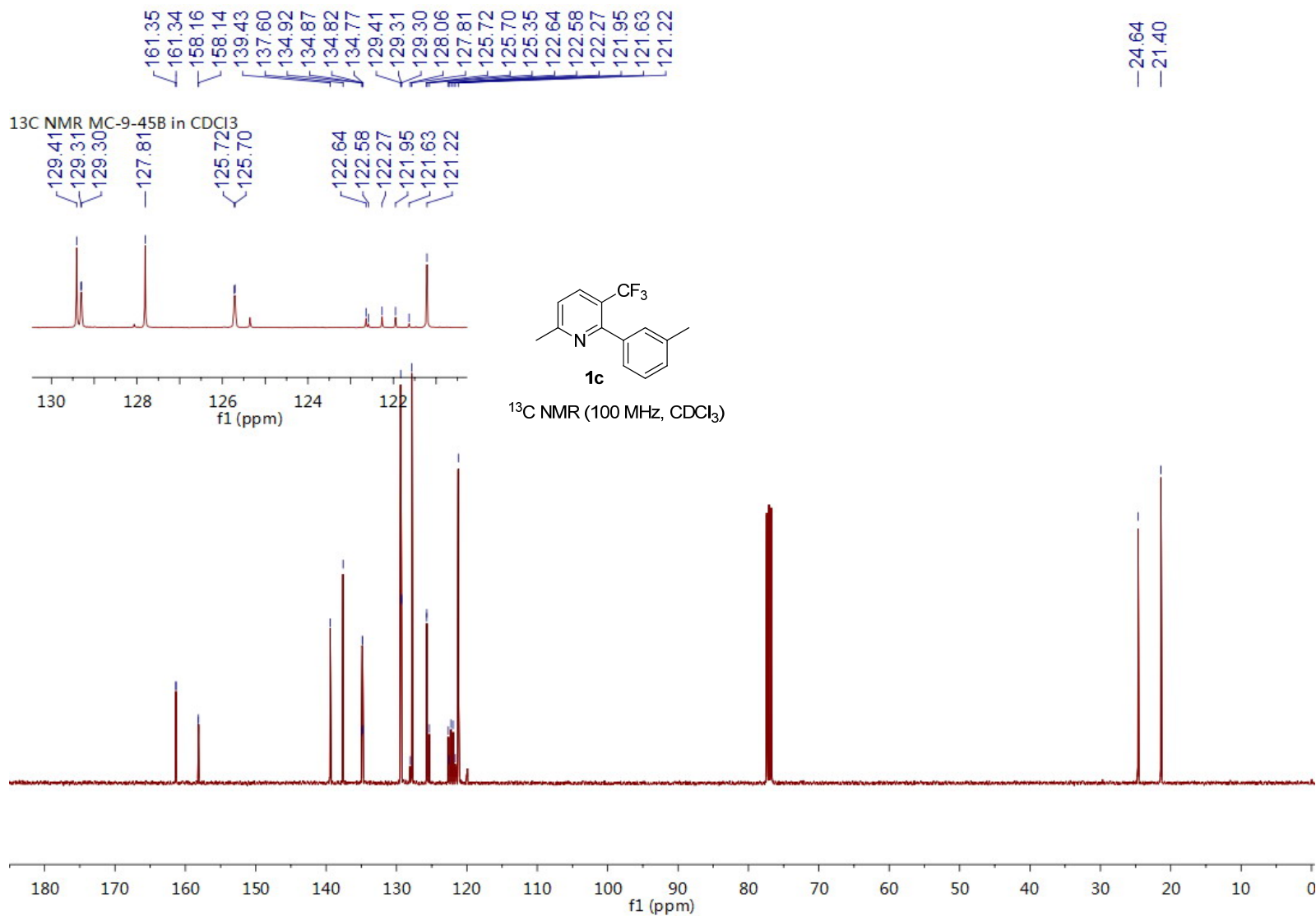
--57.0317



¹⁹F NMR (376 MHz, CDCl₃)

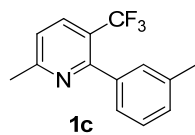




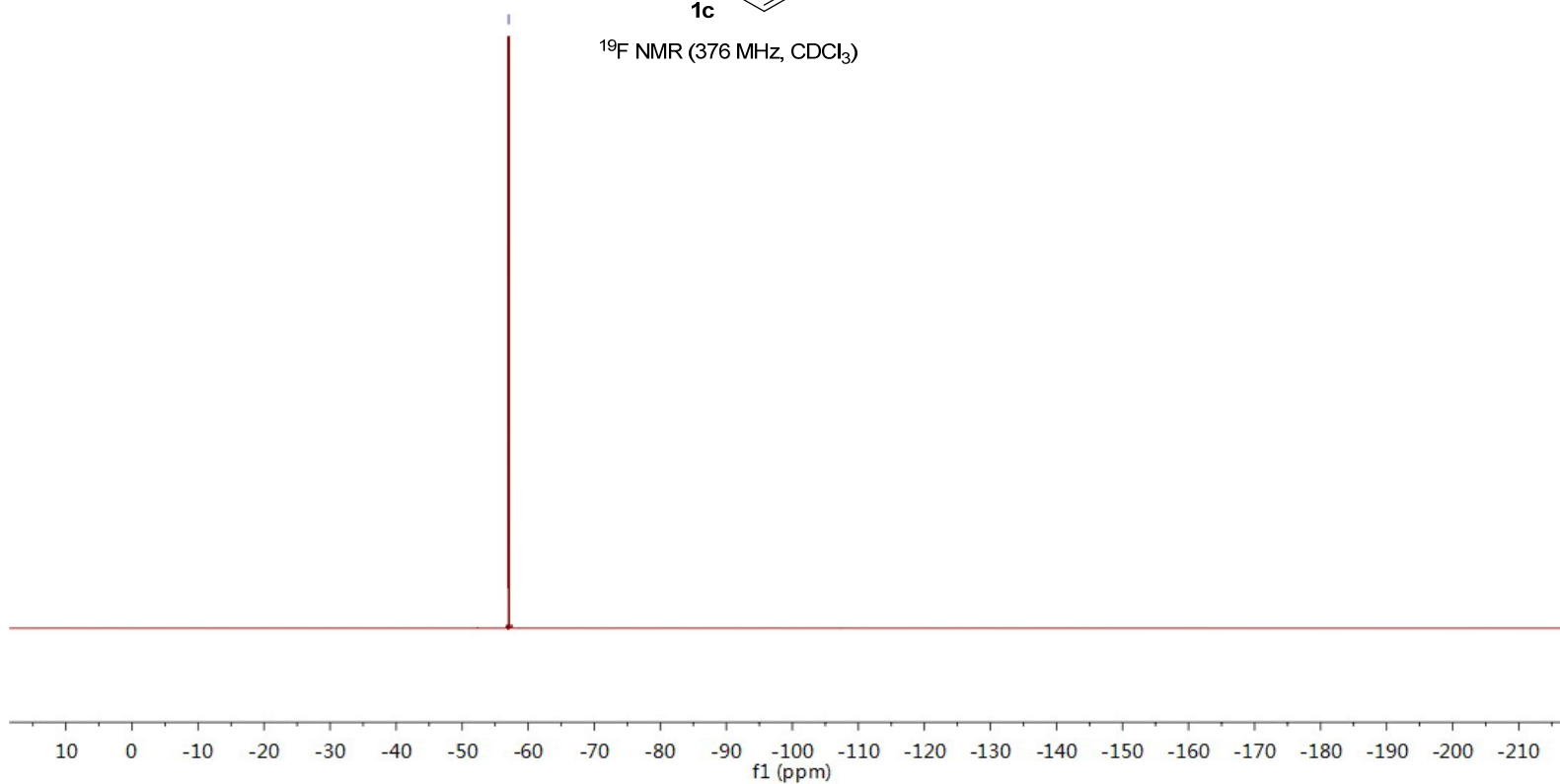


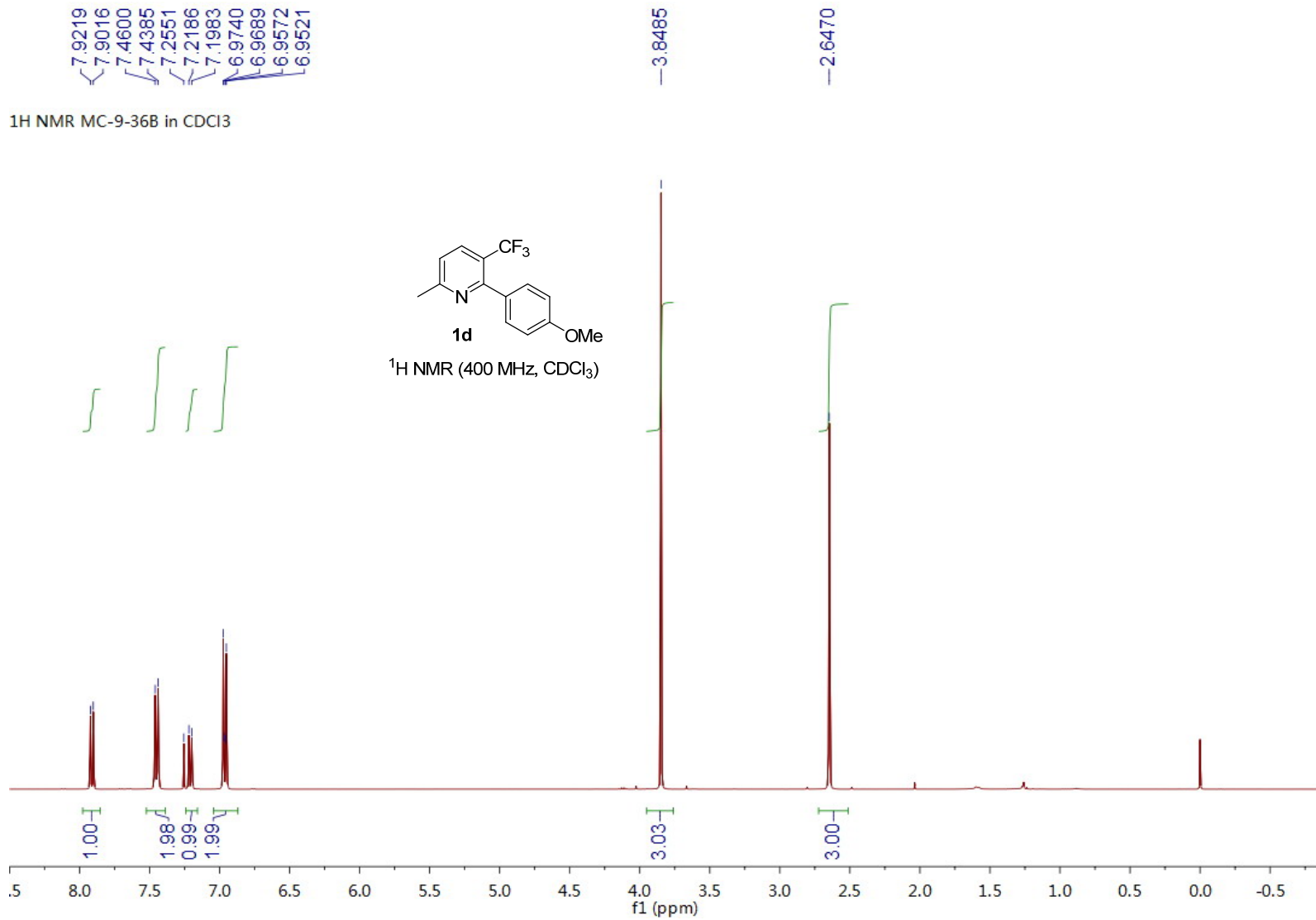
¹⁹F NMR MC-9-45B in CDCl₃

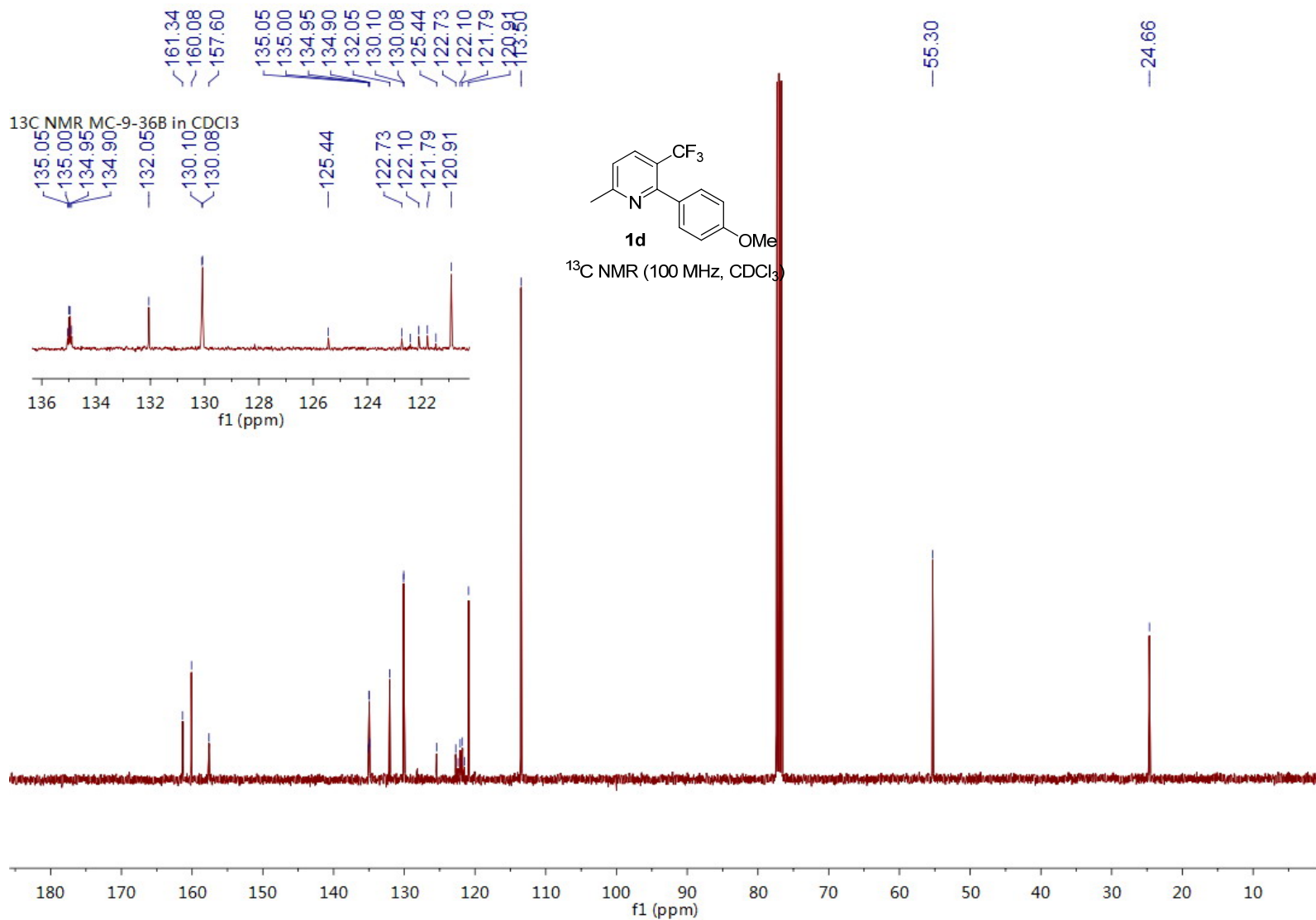
-57.0472



¹⁹F NMR (376 MHz, CDCl₃)

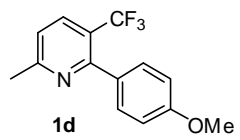




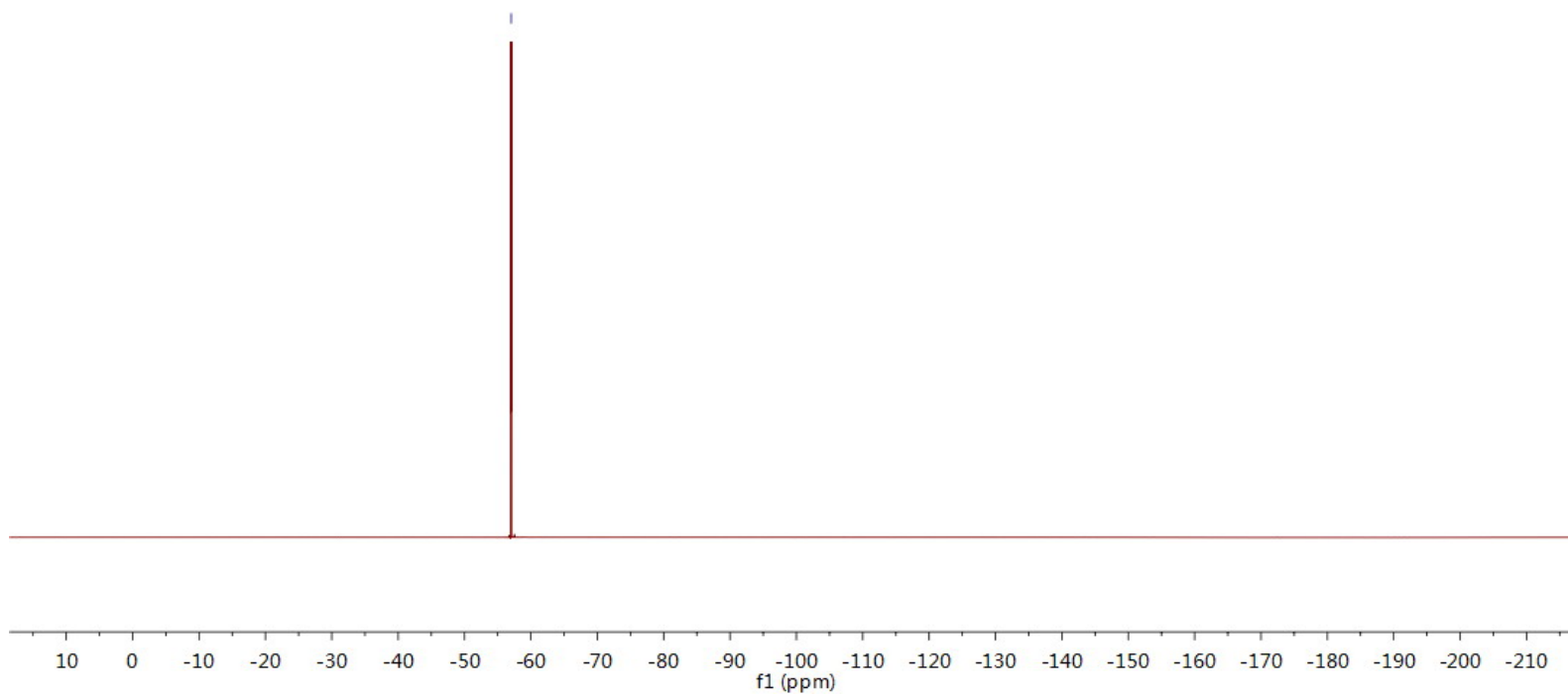


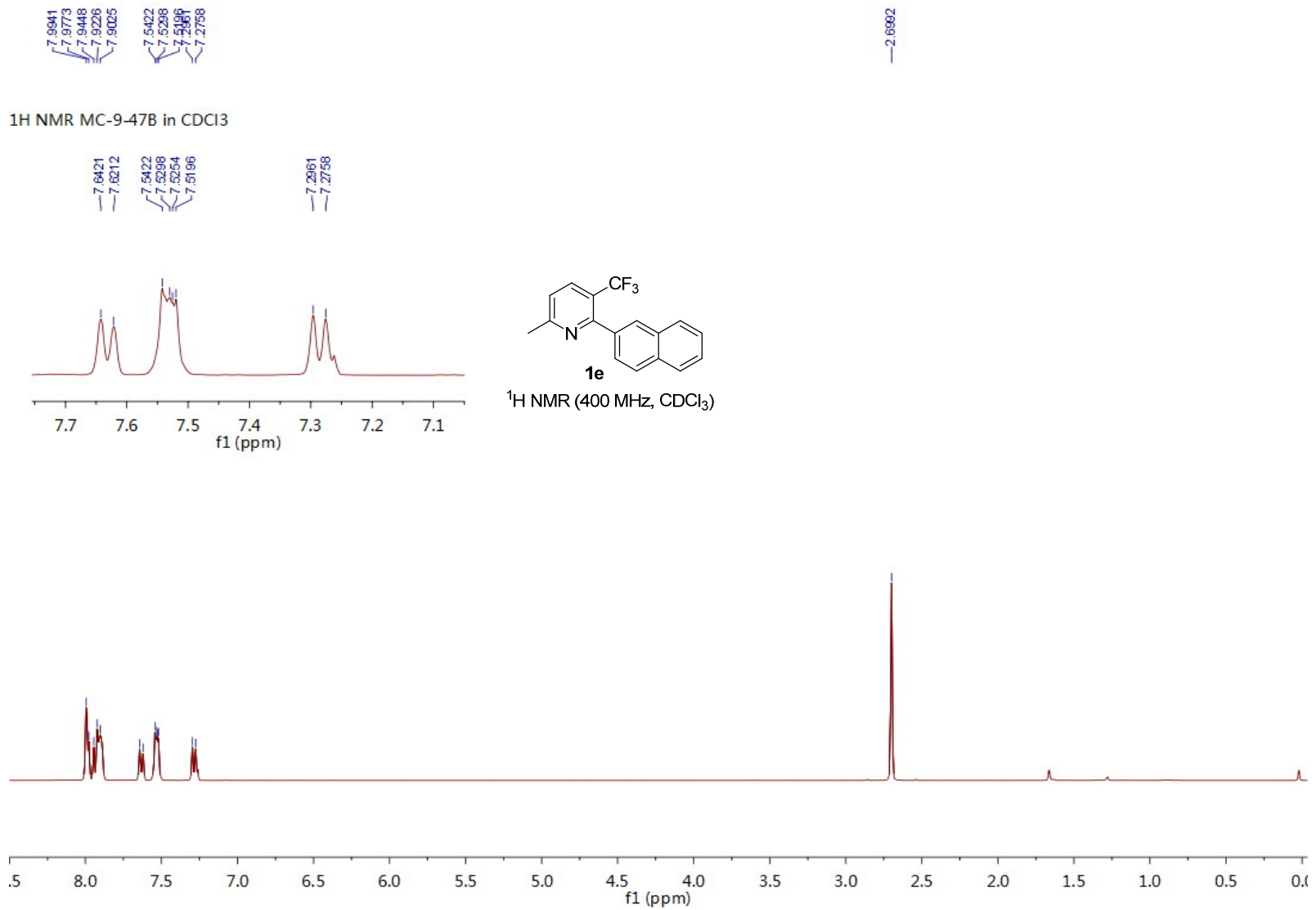
¹⁹F NMR MC-9-36B in CDCl₃

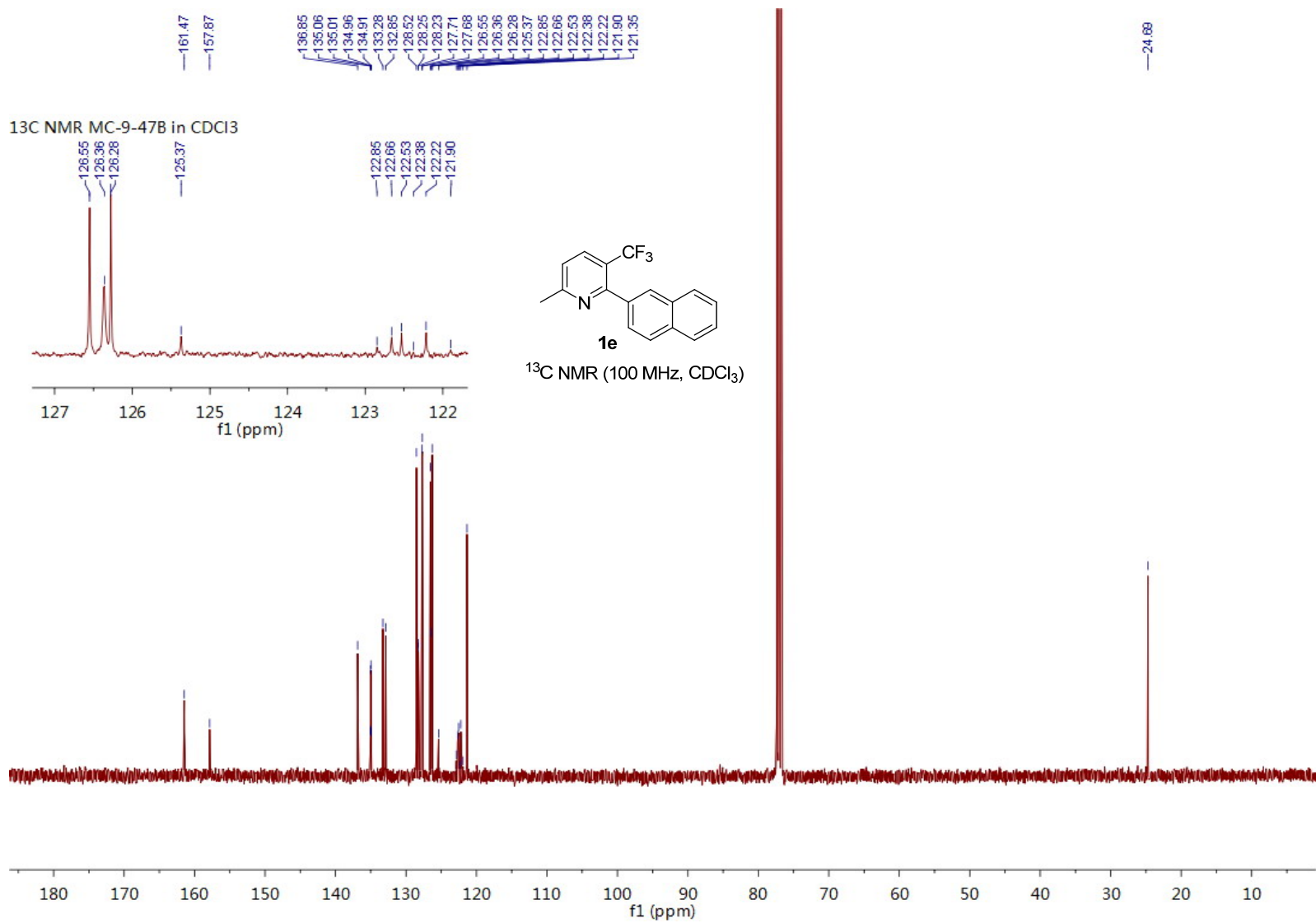
--57.0355



¹⁹F NMR (376 MHz, CDCl₃)

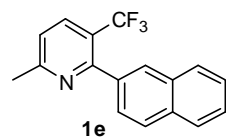




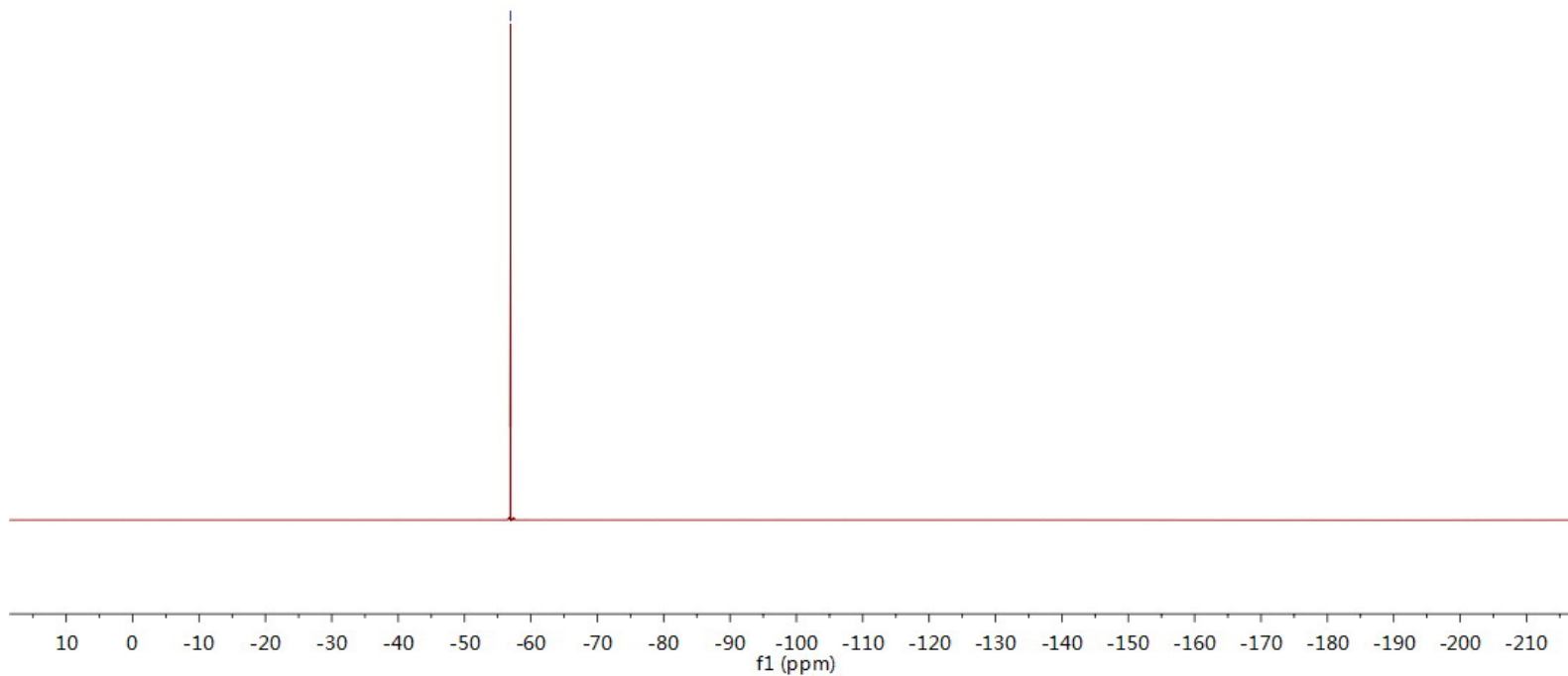


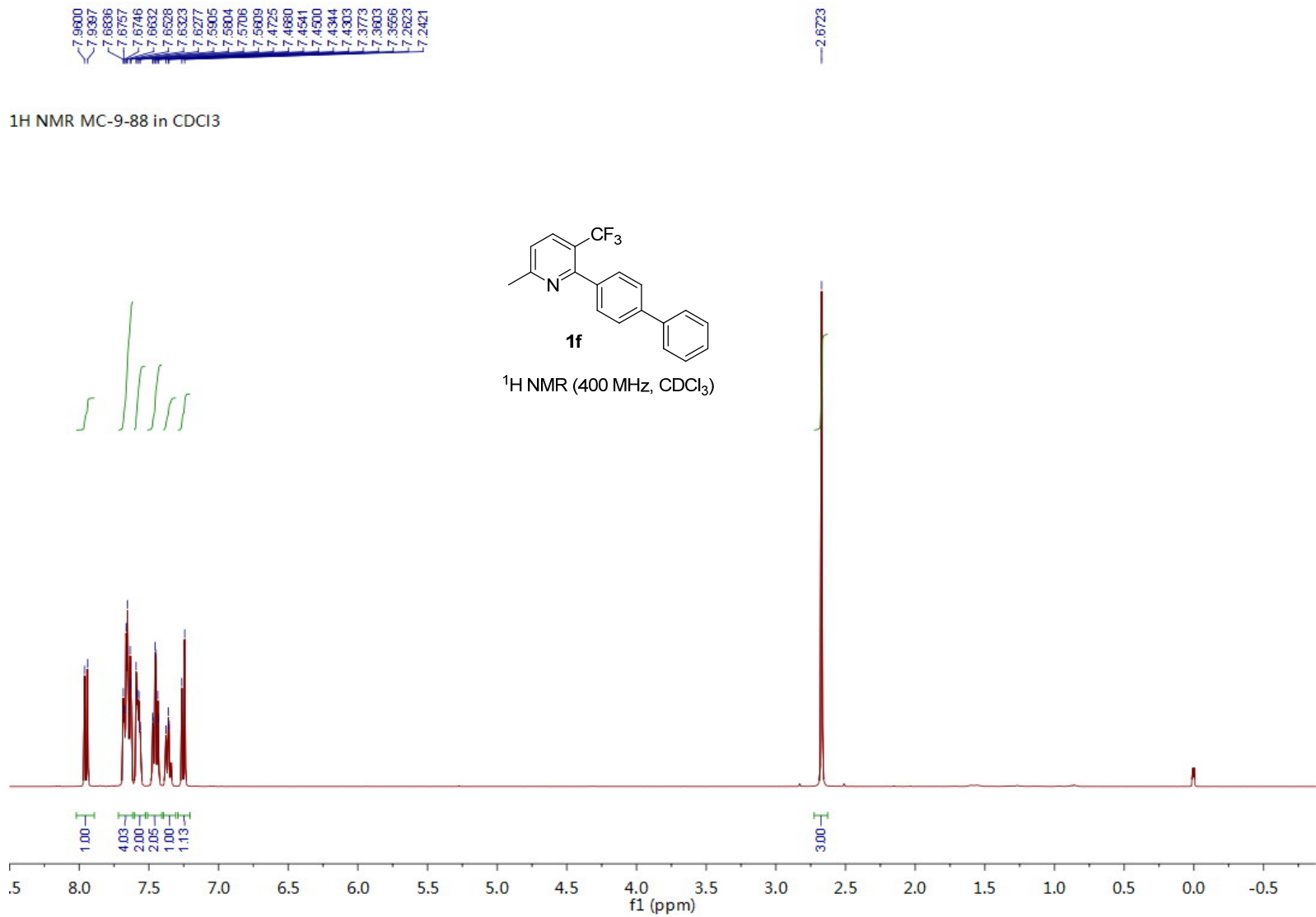
19F NMR MC-9-47B in CDCl3

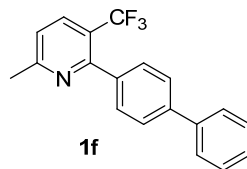
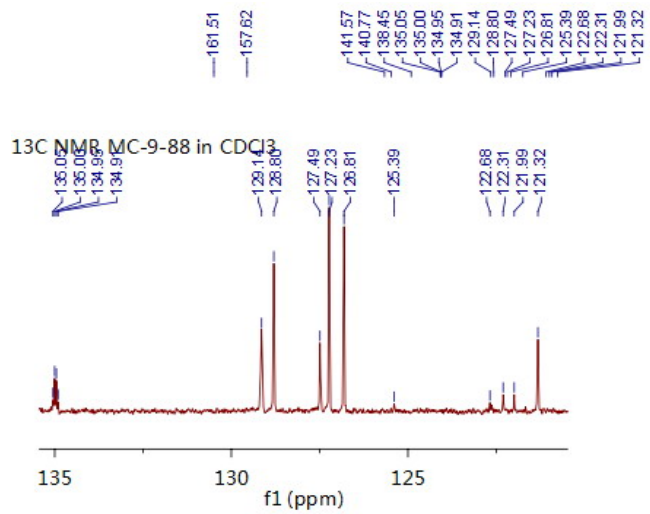
—56.9329



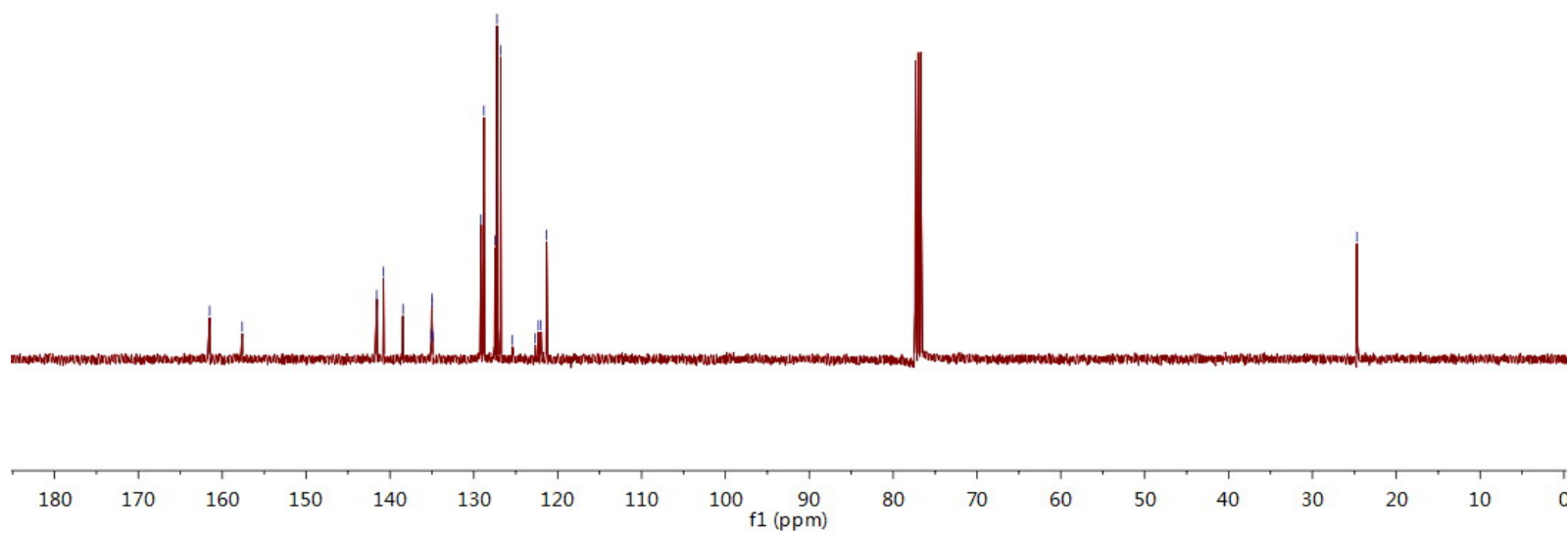
¹⁹F NMR (376 MHz, CDCl₃)





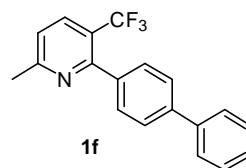


¹³C NMR (100 MHz, CDCl₃)

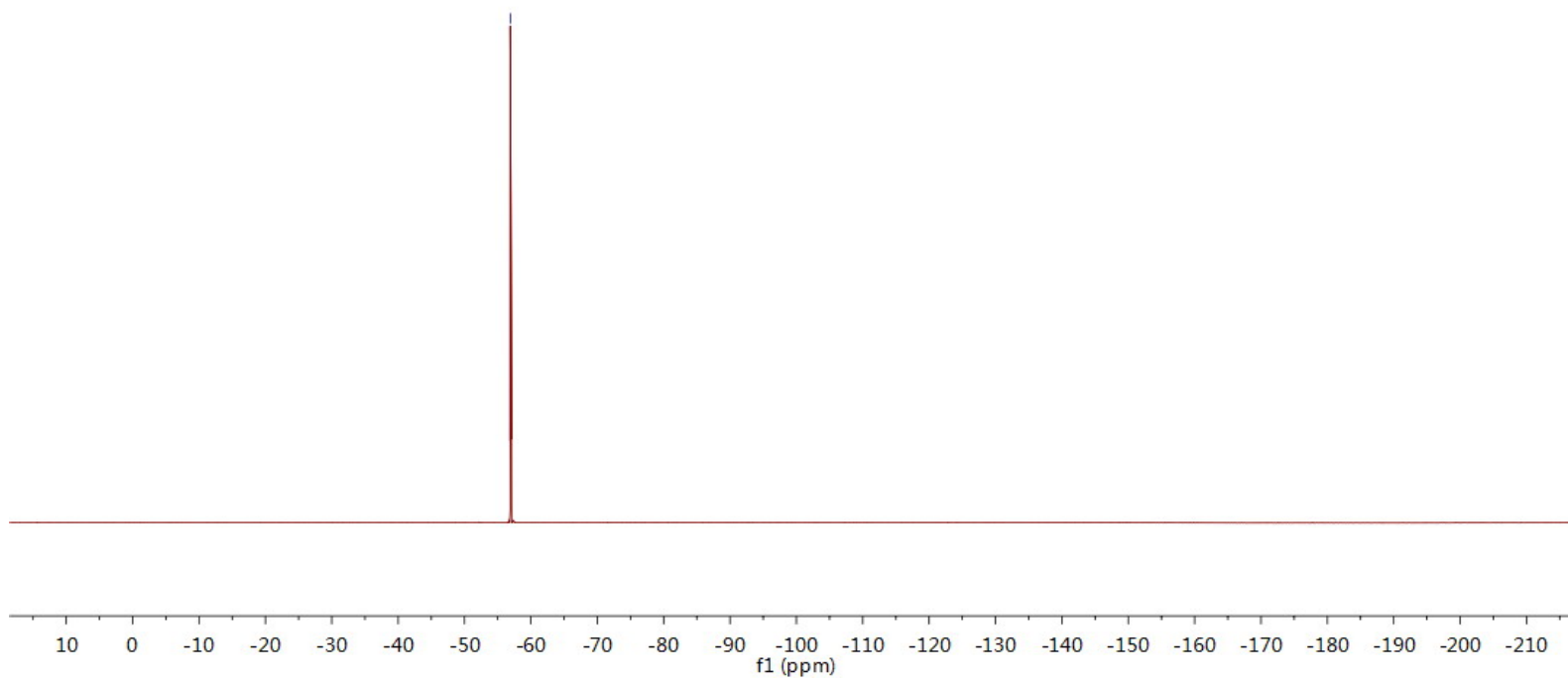


¹⁹F NMR MC-9-88 in CDCl₃

—56.8919



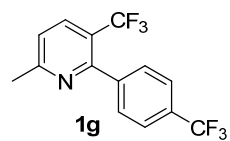
¹⁹F NMR (376 MHz, CDCl₃)



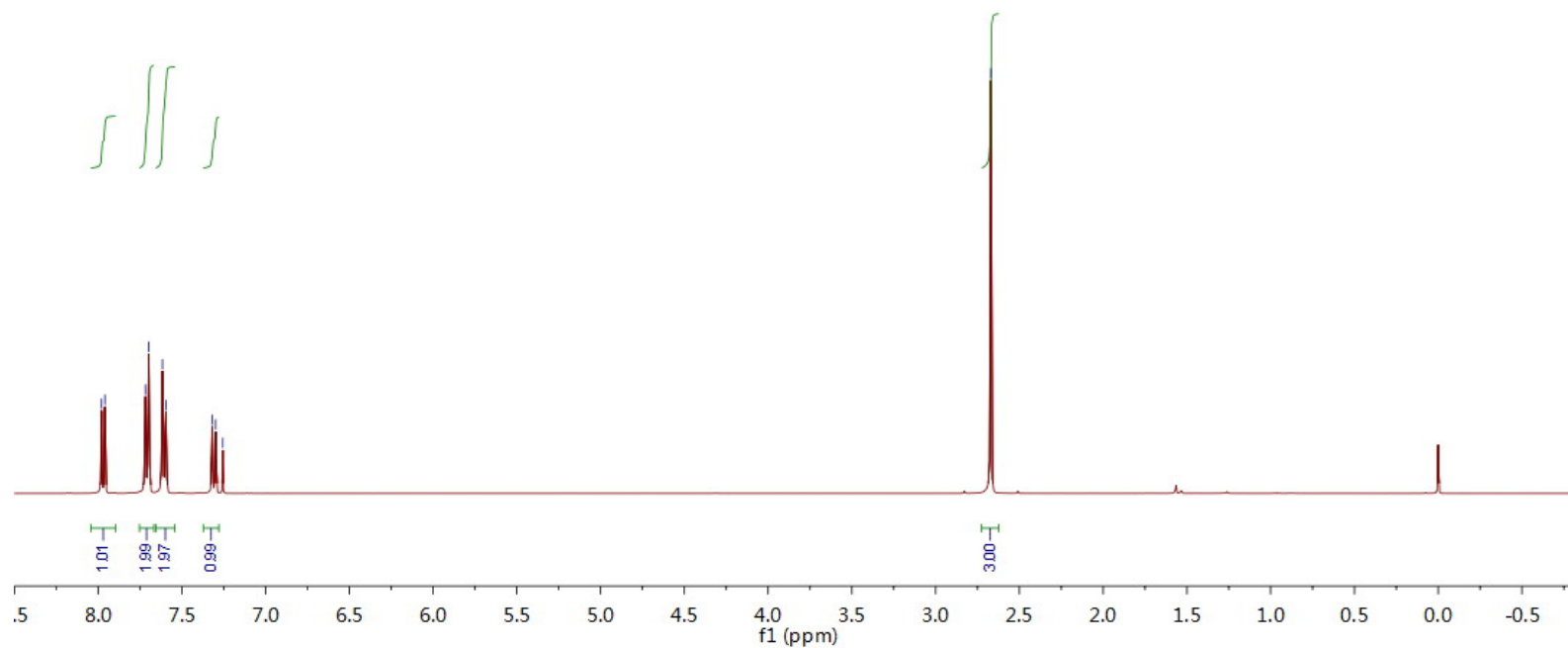
7.9783
7.9589
7.7173
7.6970
7.6163
7.2979
7.2565

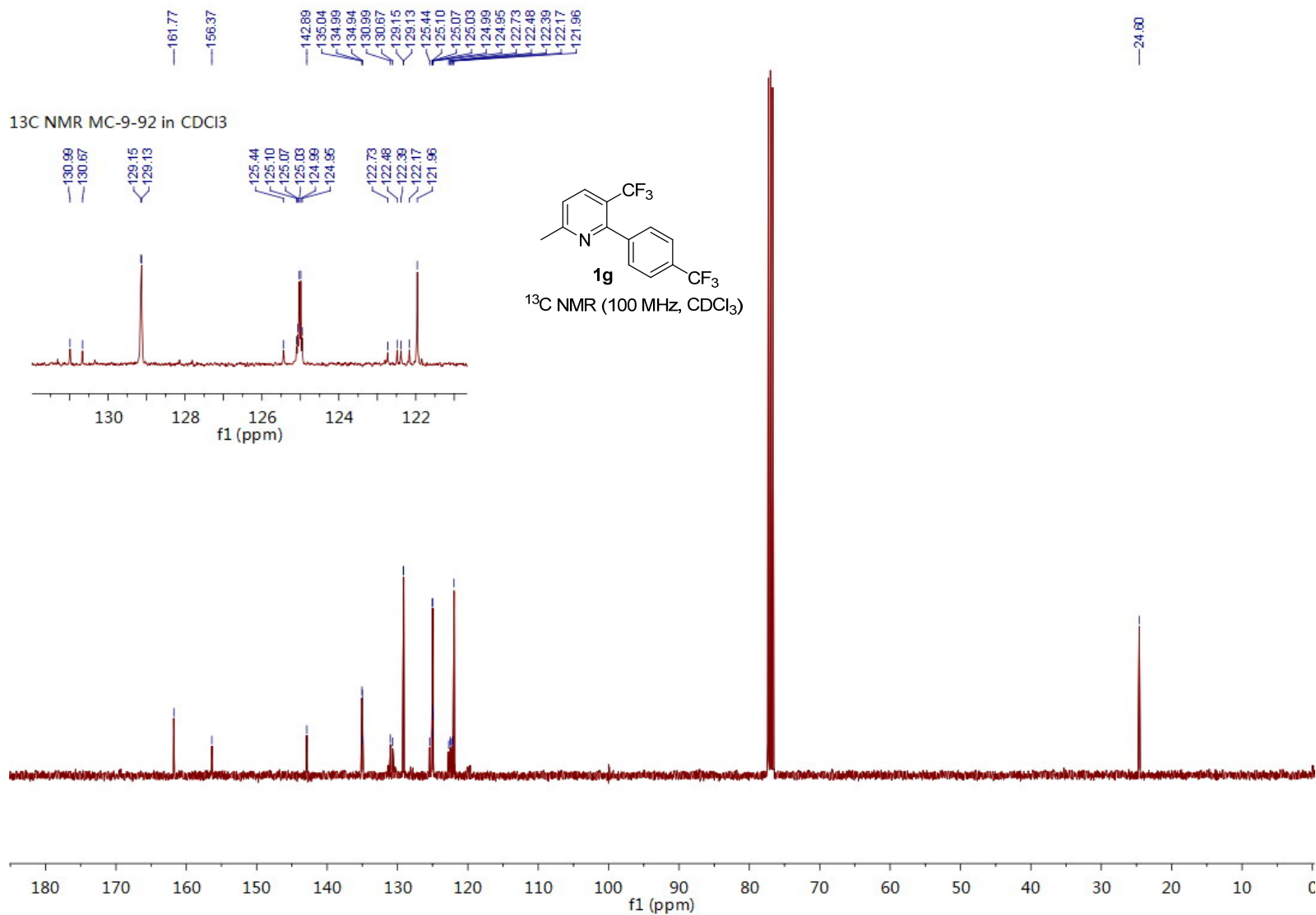
2.6689

¹H NMR MC-9-92 in CDCl₃



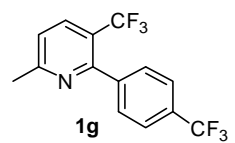
¹H NMR (400 MHz, CDCl₃)



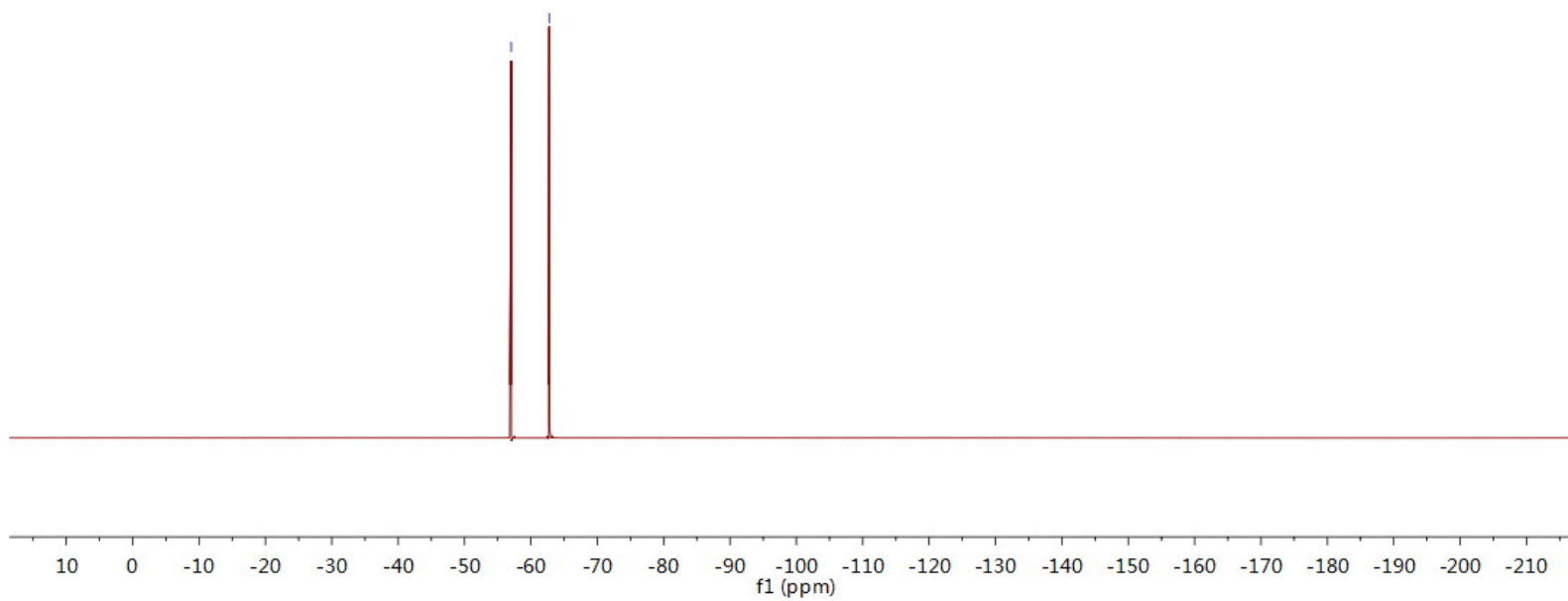


¹⁹F NMR MC-9-92 in CDCl₃

—57.0345
—62.7643



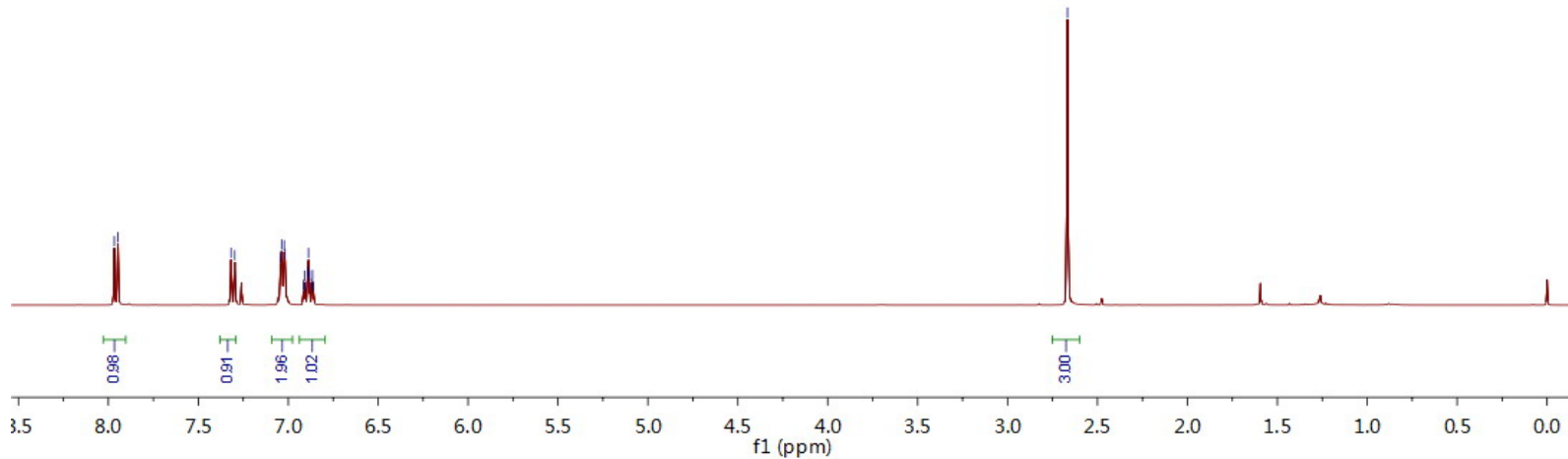
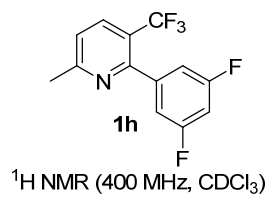
¹⁹F NMR (376 MHz, CDCl₃)

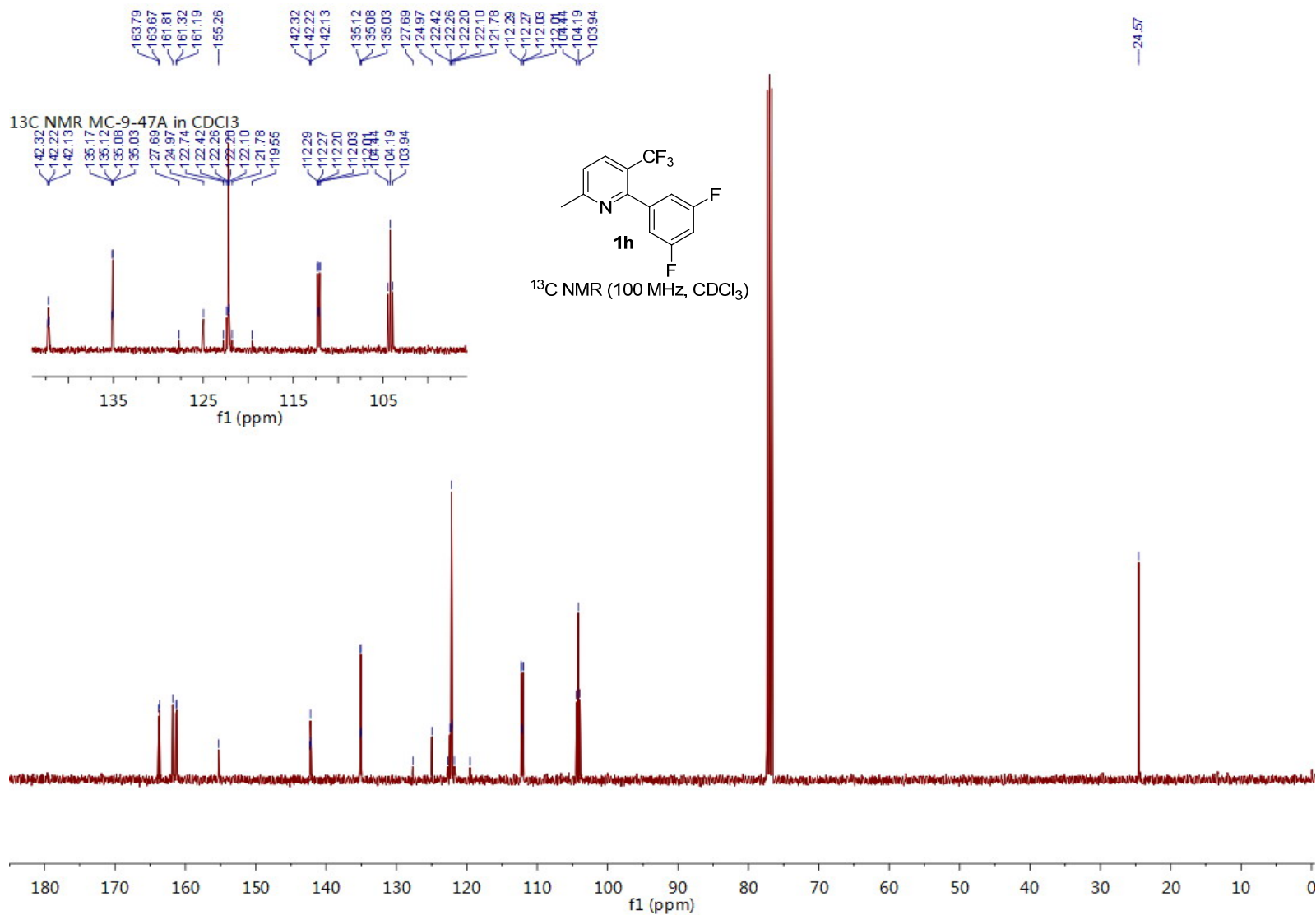


7.9669
7.9465
7.3176
7.2972
7.0417
7.0370
7.0222
7.0176
6.9150
6.9092
6.9034
6.8927
6.8869
6.8811
6.8704
6.8646
6.8587

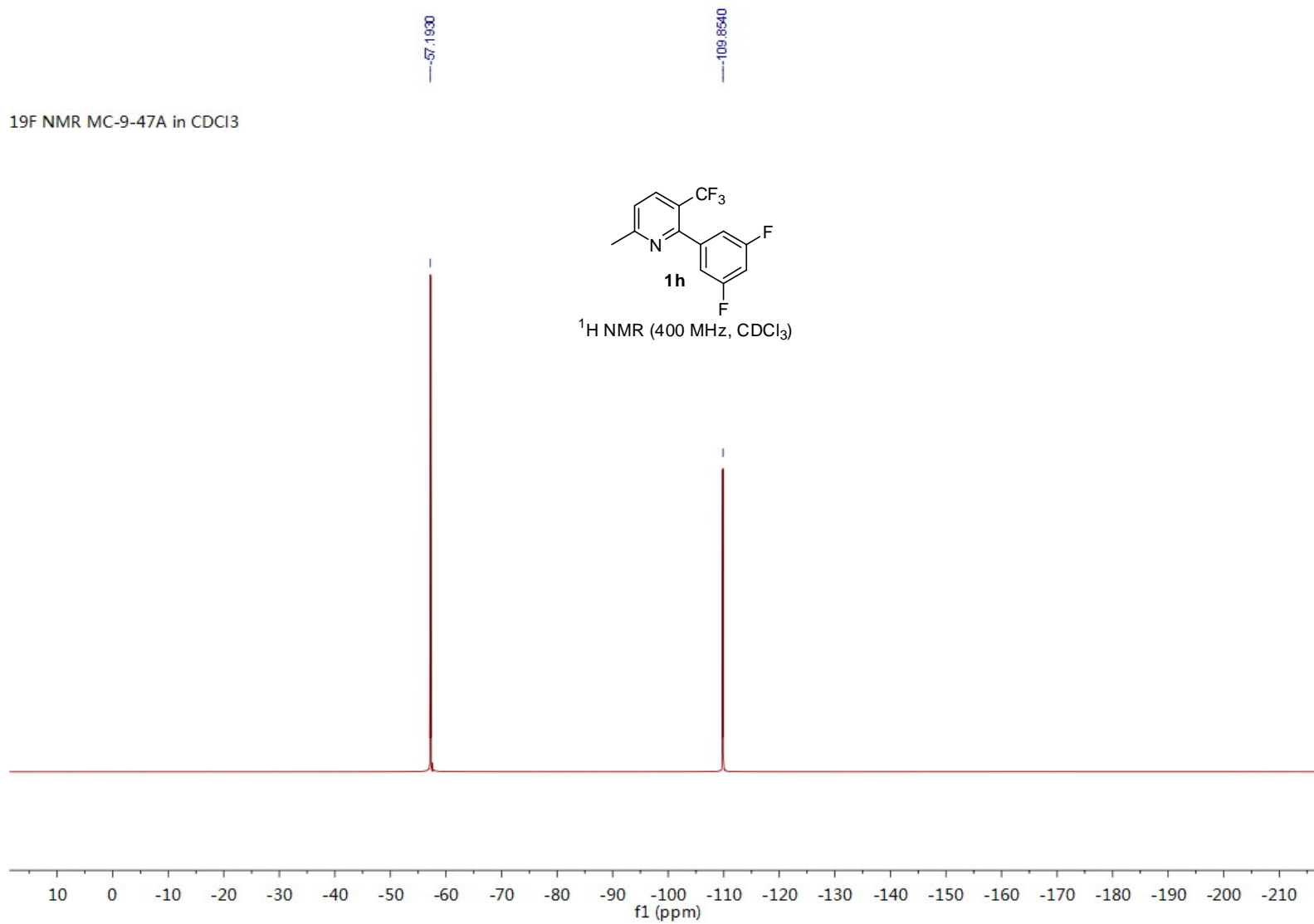
2.6669

¹H NMR MC-9-47A in CDCl₃





19F NMR MC-9-47A in CDCl3

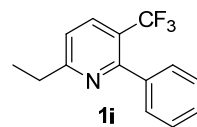


7.9652
7.9348
7.5064
7.4946
7.4410
7.4383
7.4298
7.4275
7.4202
7.4150
7.4134
7.2473
7.2271

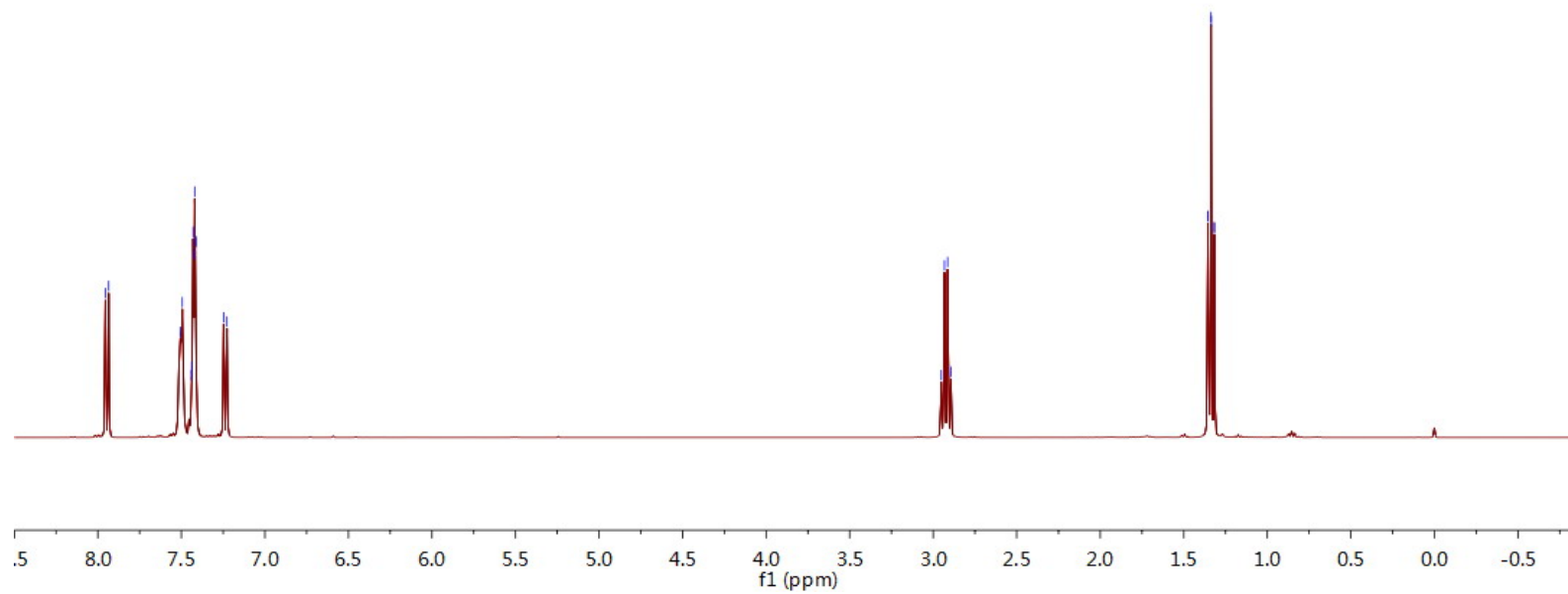
2.9514
2.9324
2.9134
2.8943

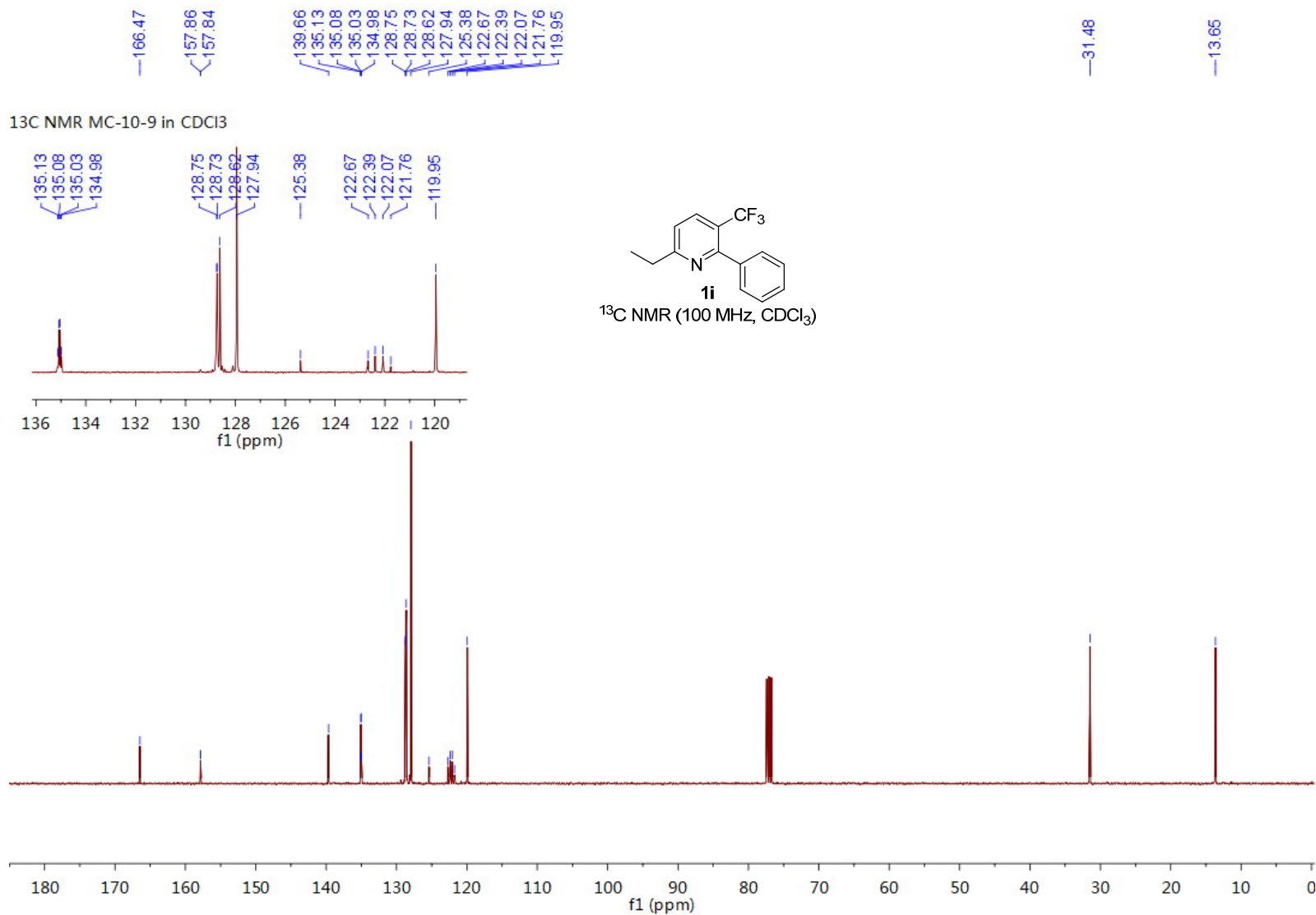
1.3657
1.3543
1.3368
1.3352
1.3176
1.3162

¹H NMR MC-10-9 in CDCl₃



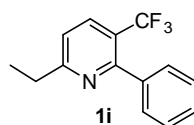
¹H NMR (400 MHz, CDCl₃)





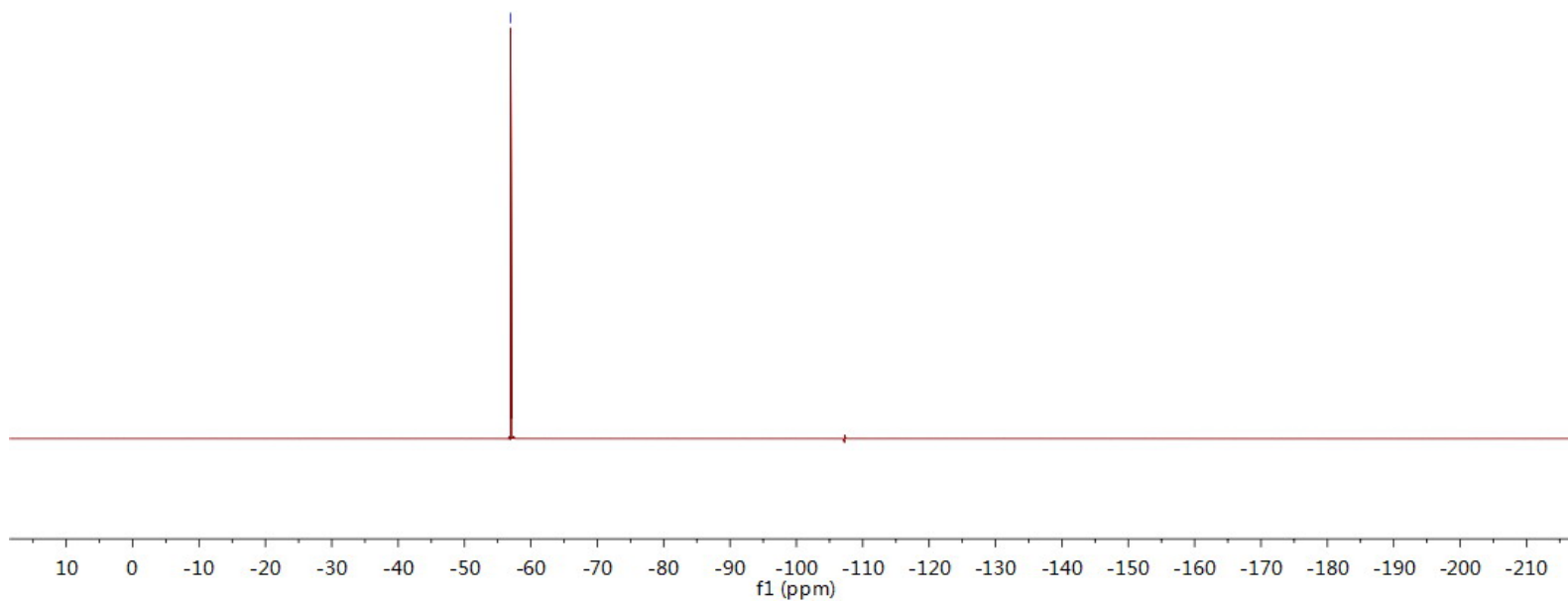
19F NMR MC-10-9 in CDCl3

—56.9314

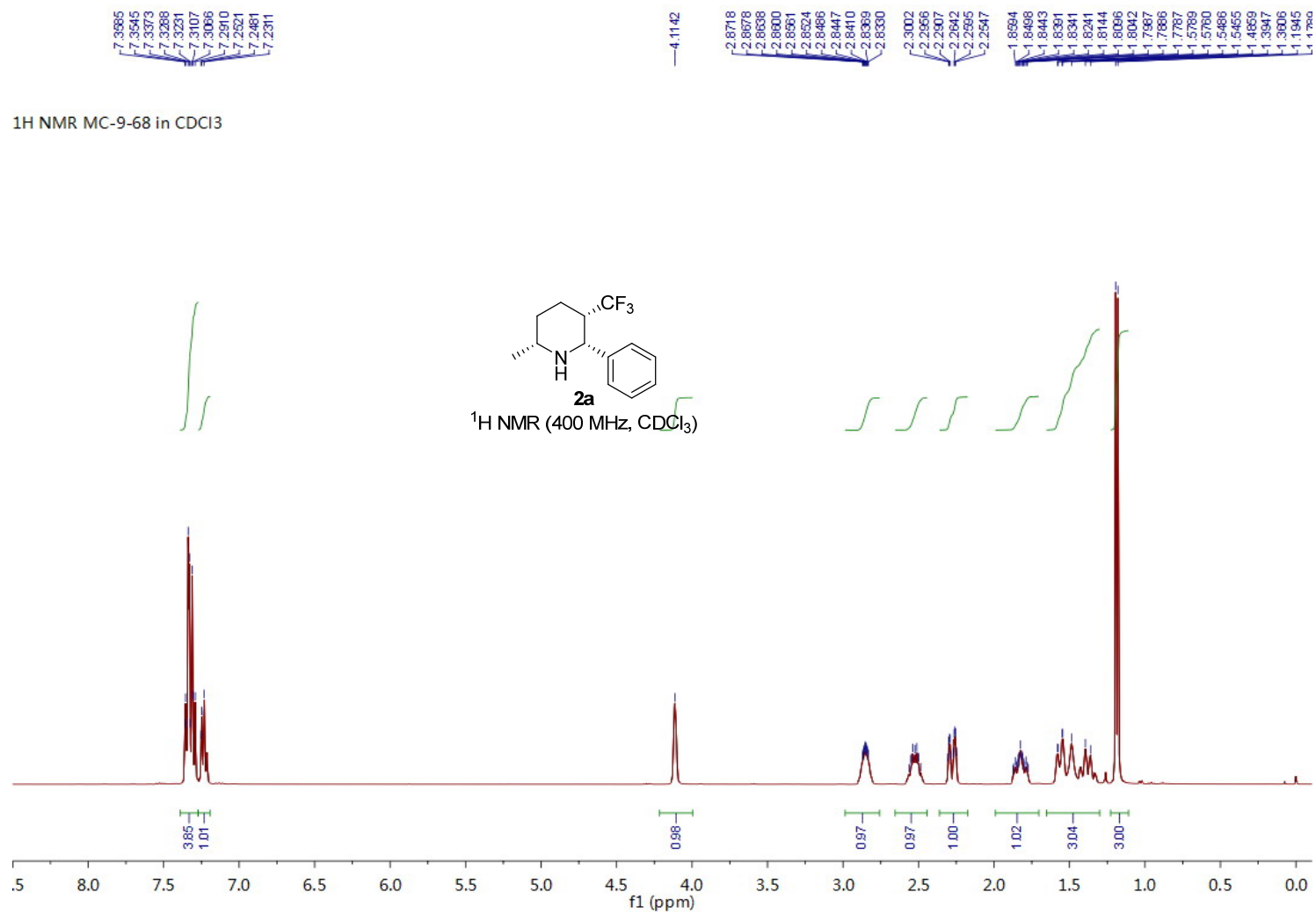


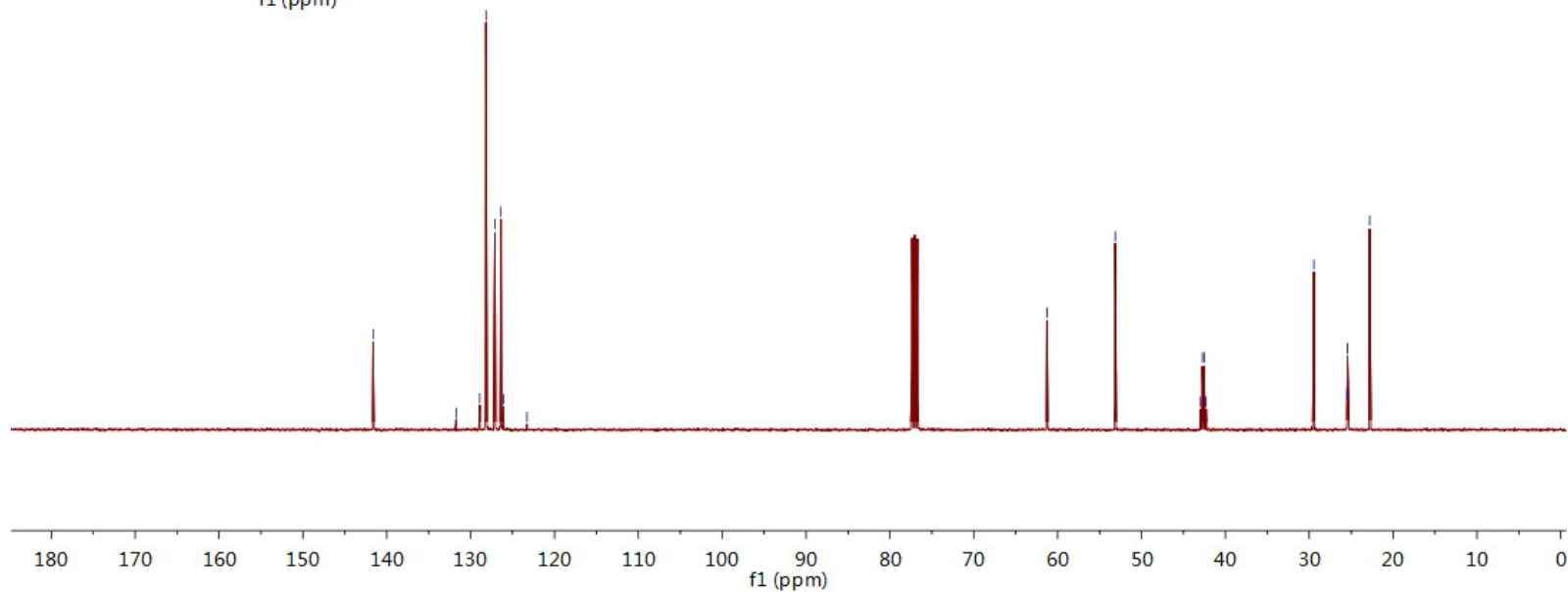
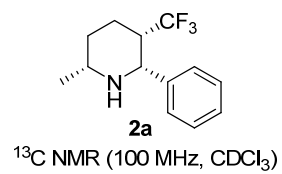
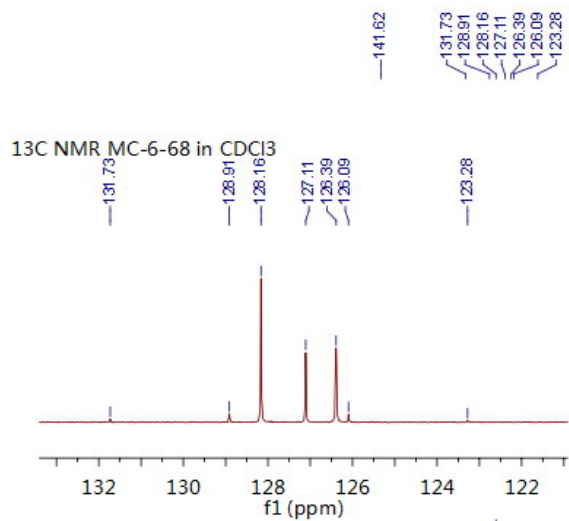
1i

¹H NMR (400 MHz, CDCl₃)



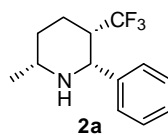
6.2 Copy of NMR for trifluoromethyl piperidines



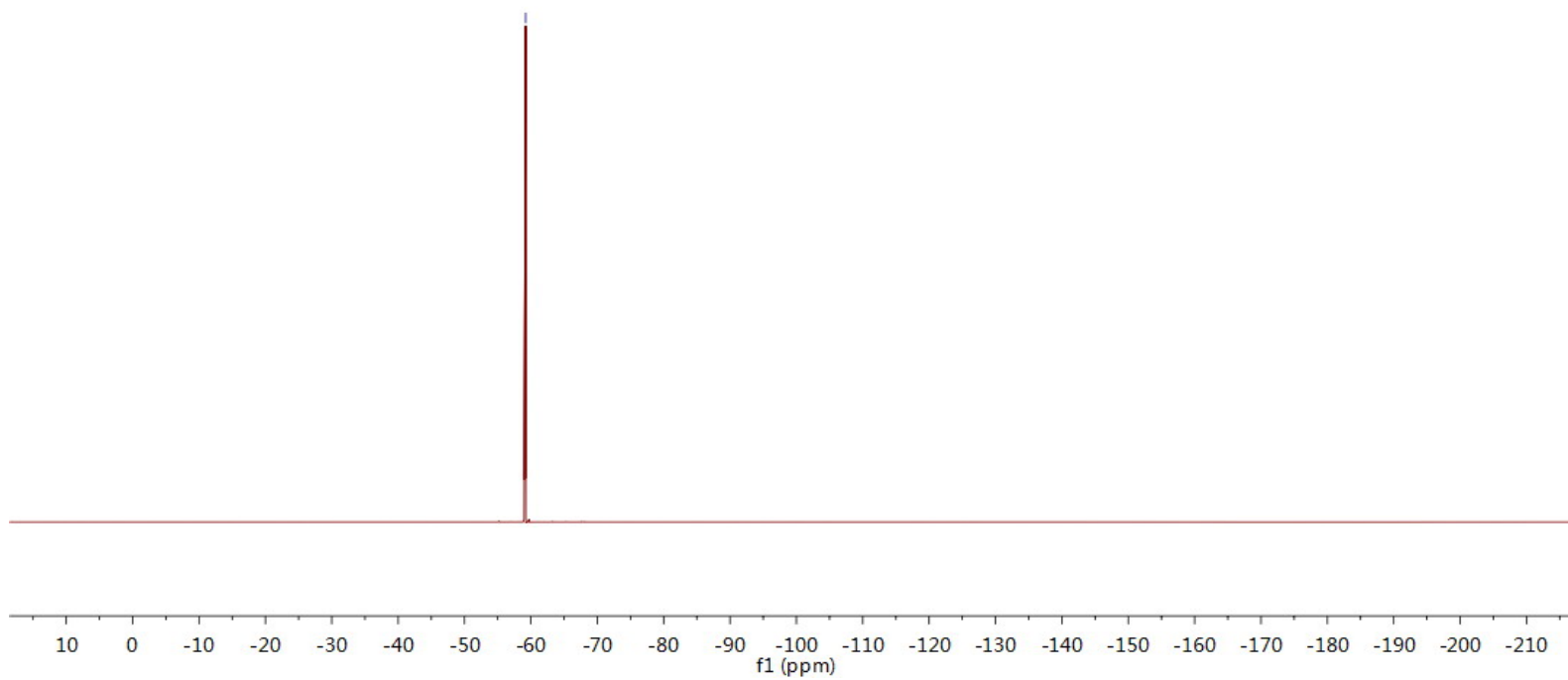


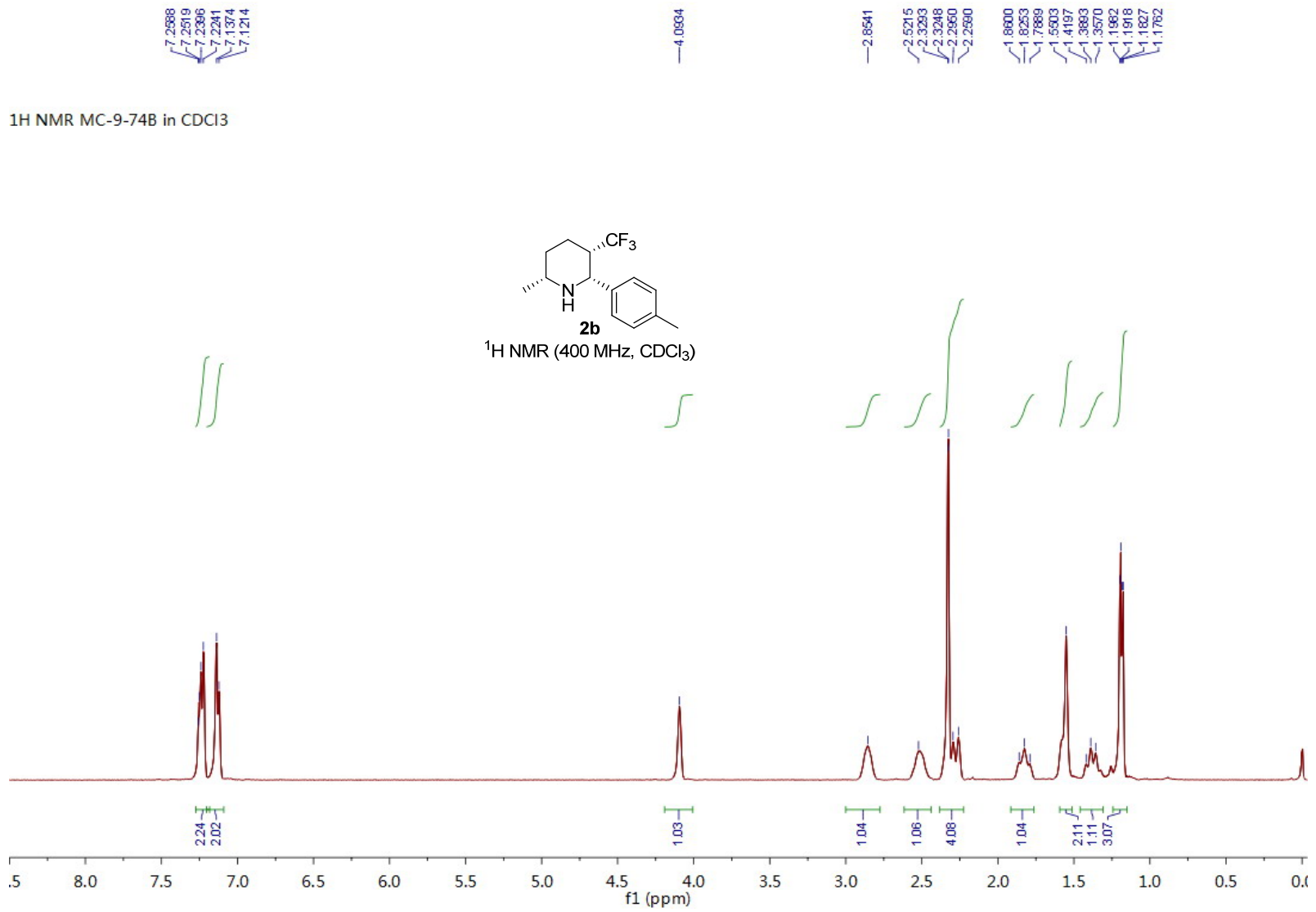
59.2108

19F NMR MC-9-68 in CDCl3

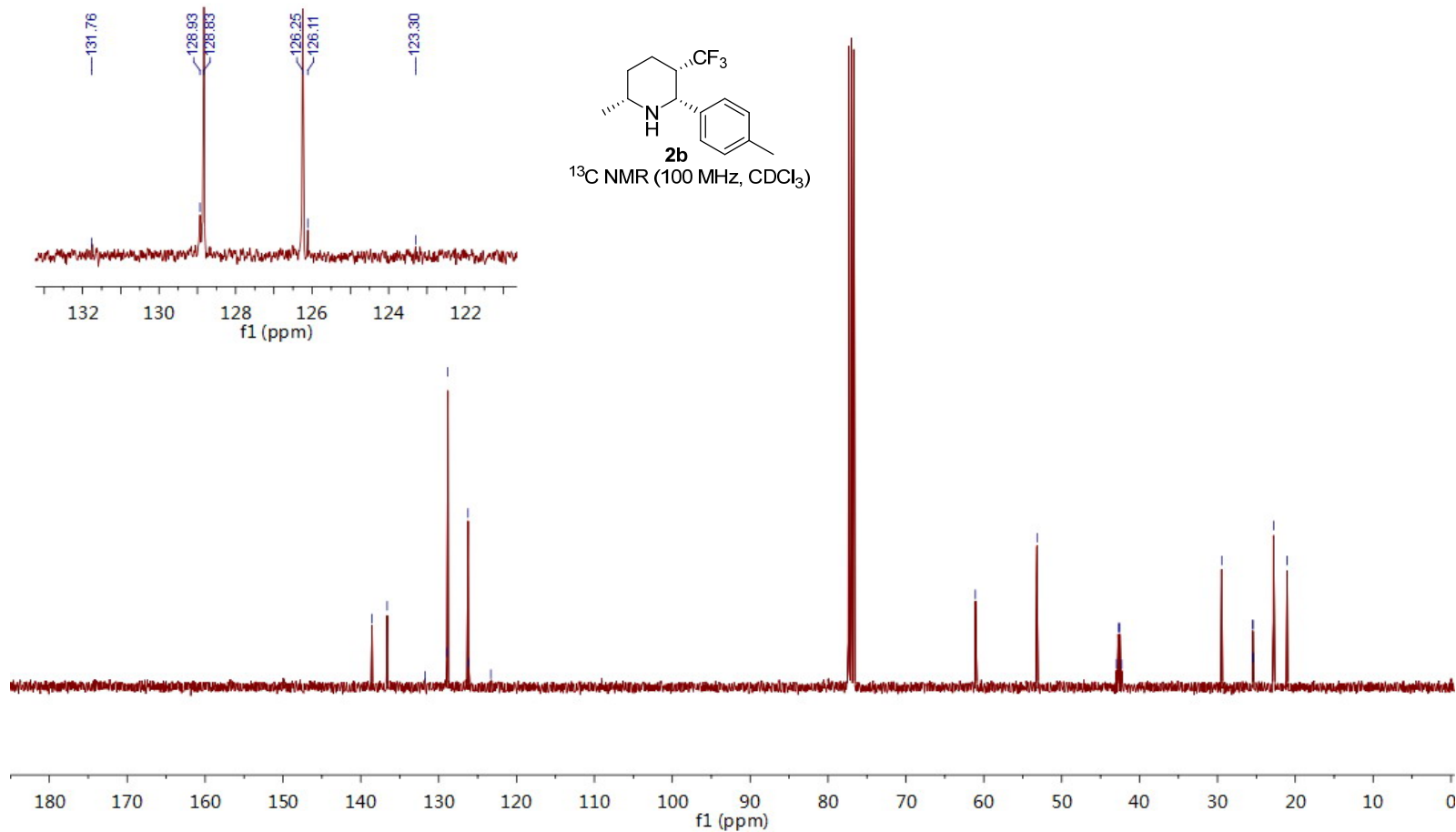


¹³C NMR (100 MHz, CDCl₃)



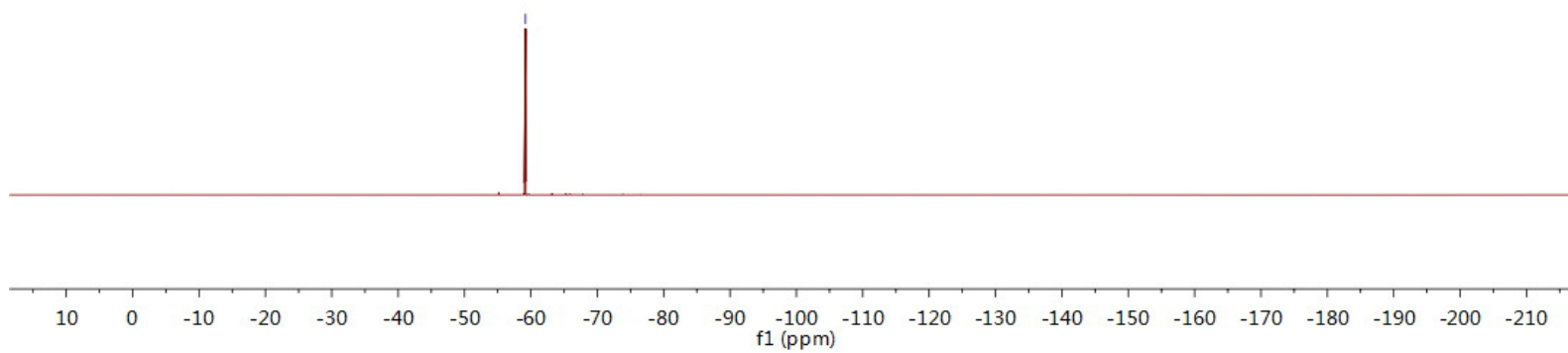
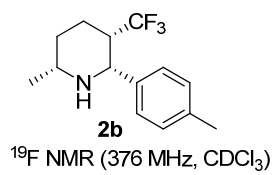


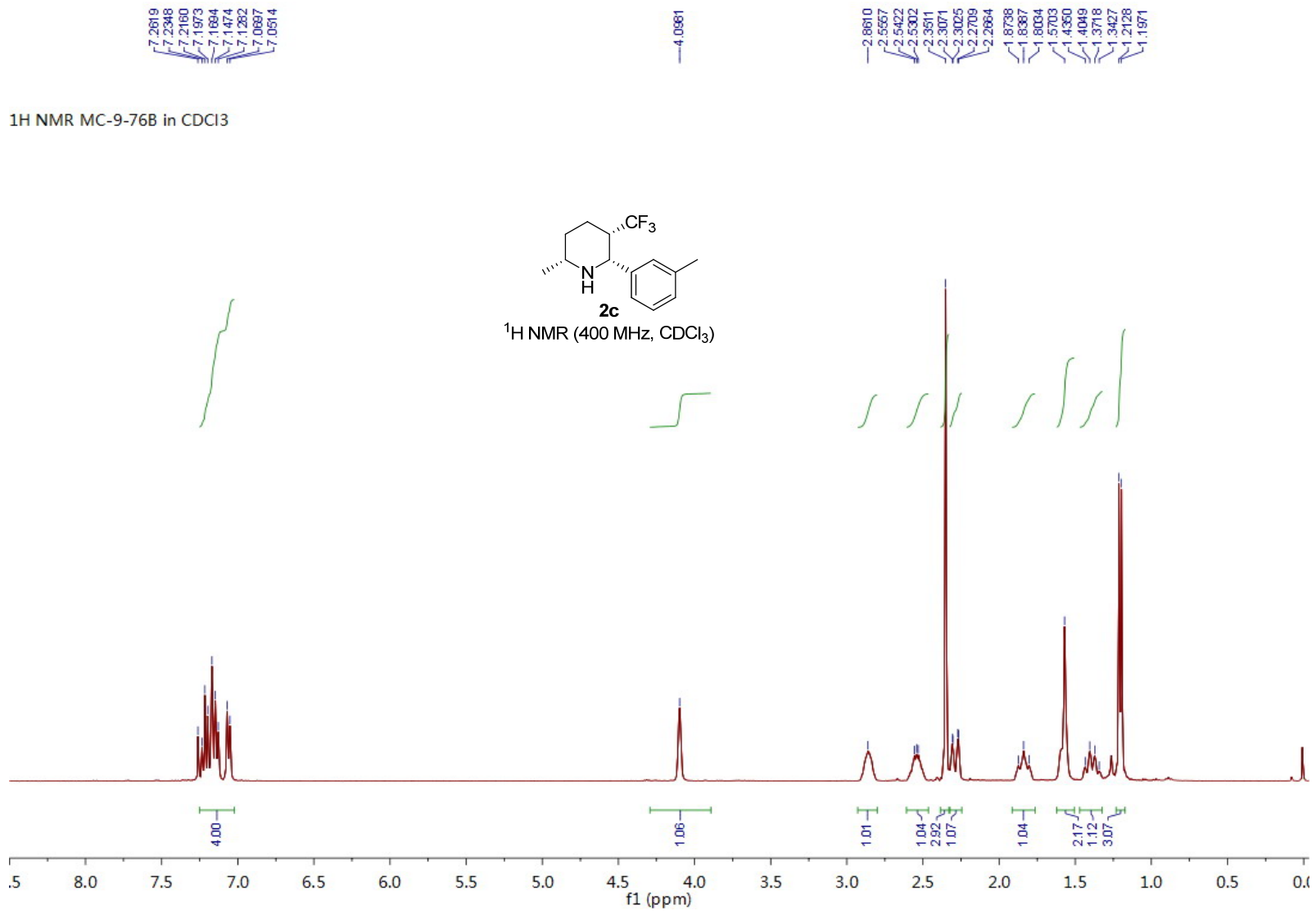
¹³C NMR MC-9-74B in CDCl₃

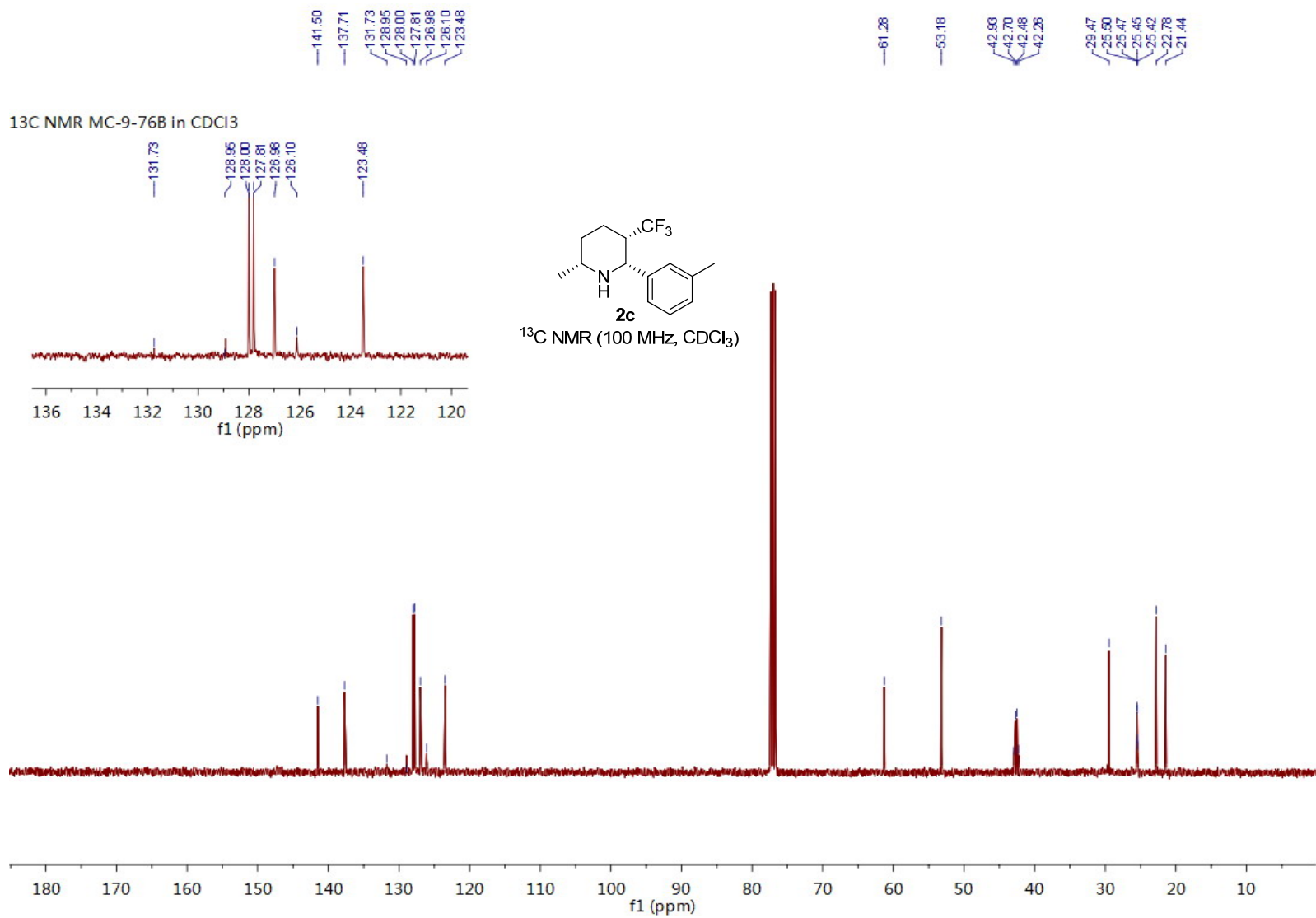


¹⁹F NMR MC-9-74B in CDCl₃

-59.1803

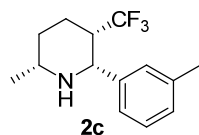




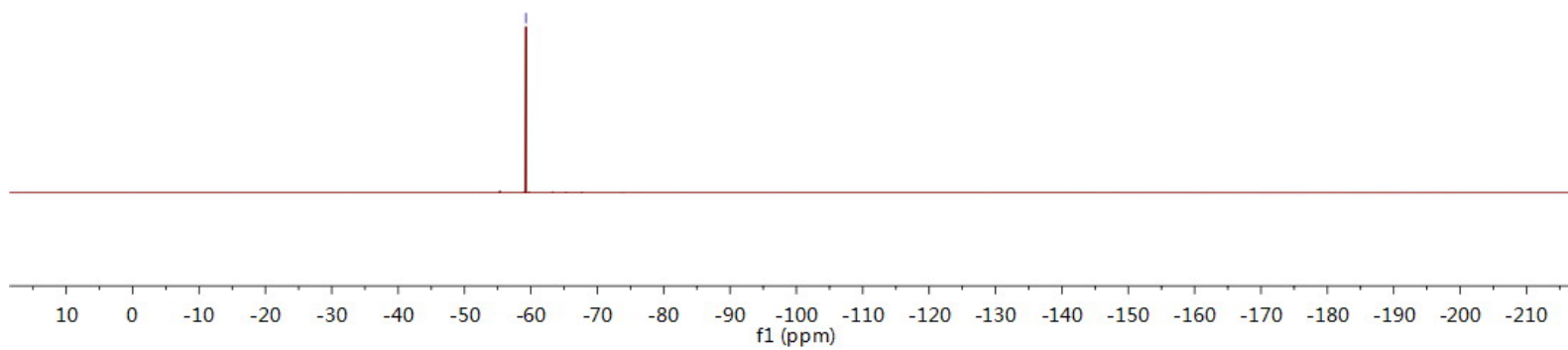


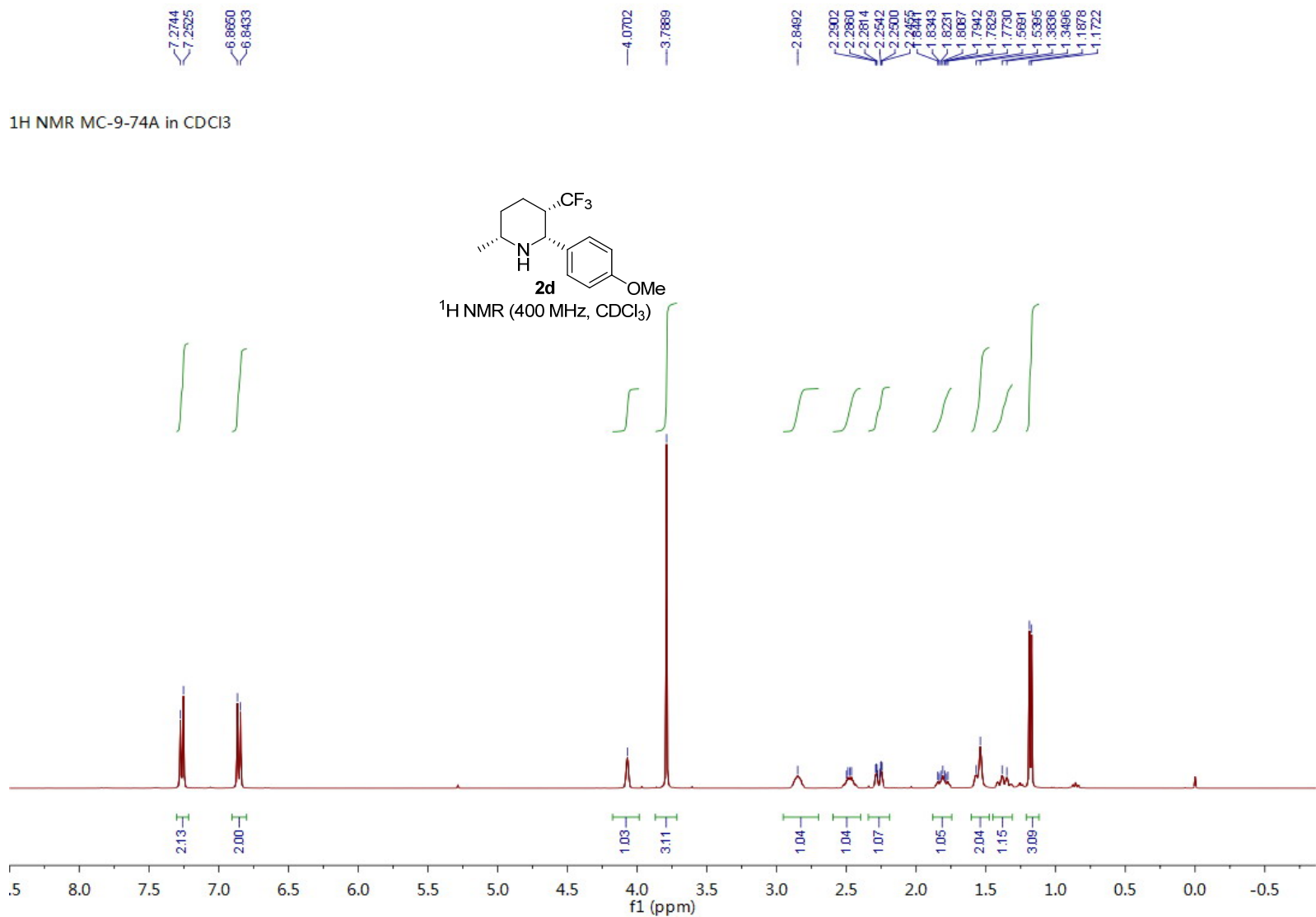
59.2472

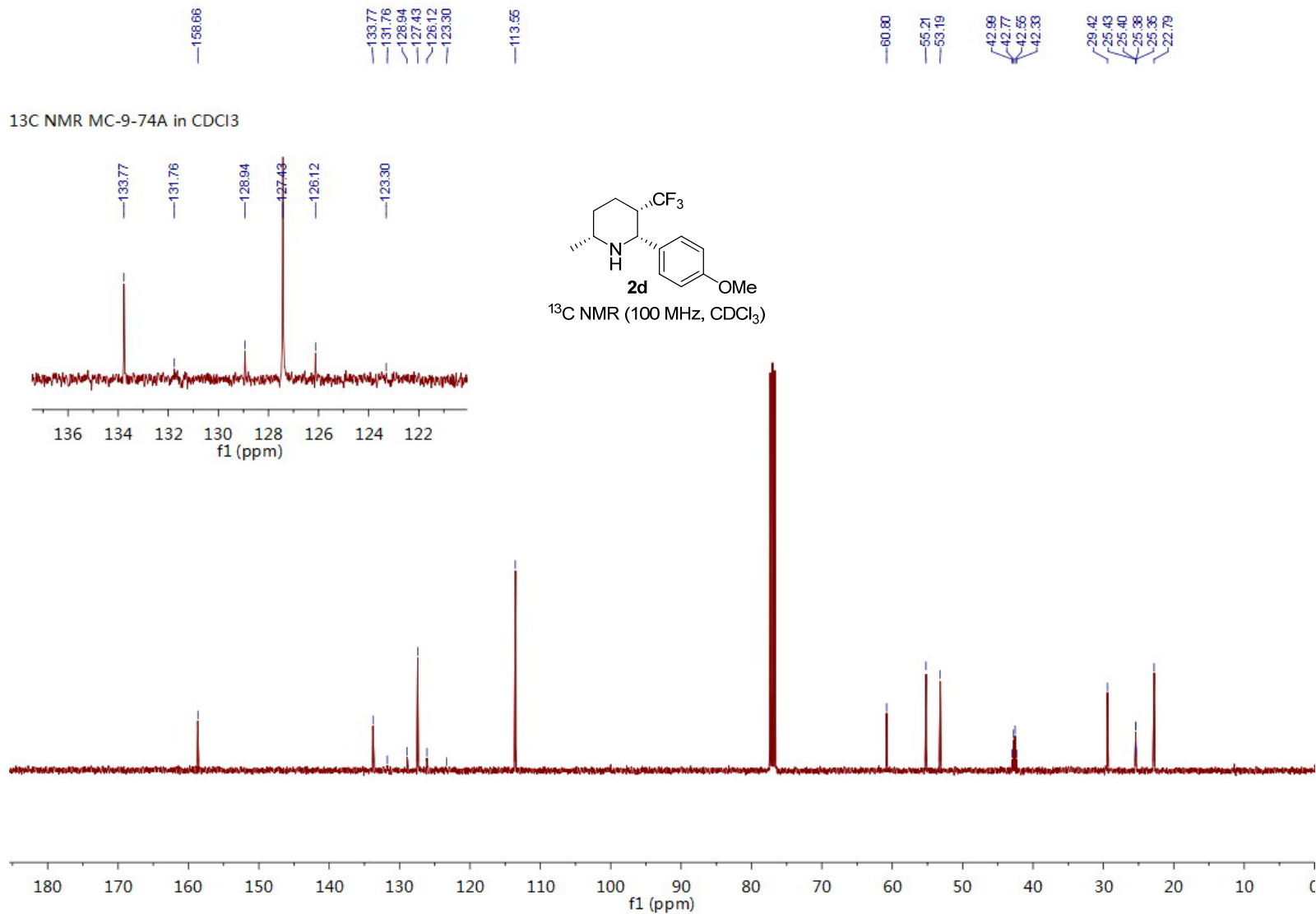
¹⁹F NMR MC-9-76B in CDCl₃



¹⁹F NMR (376 MHz, CDCl₃)

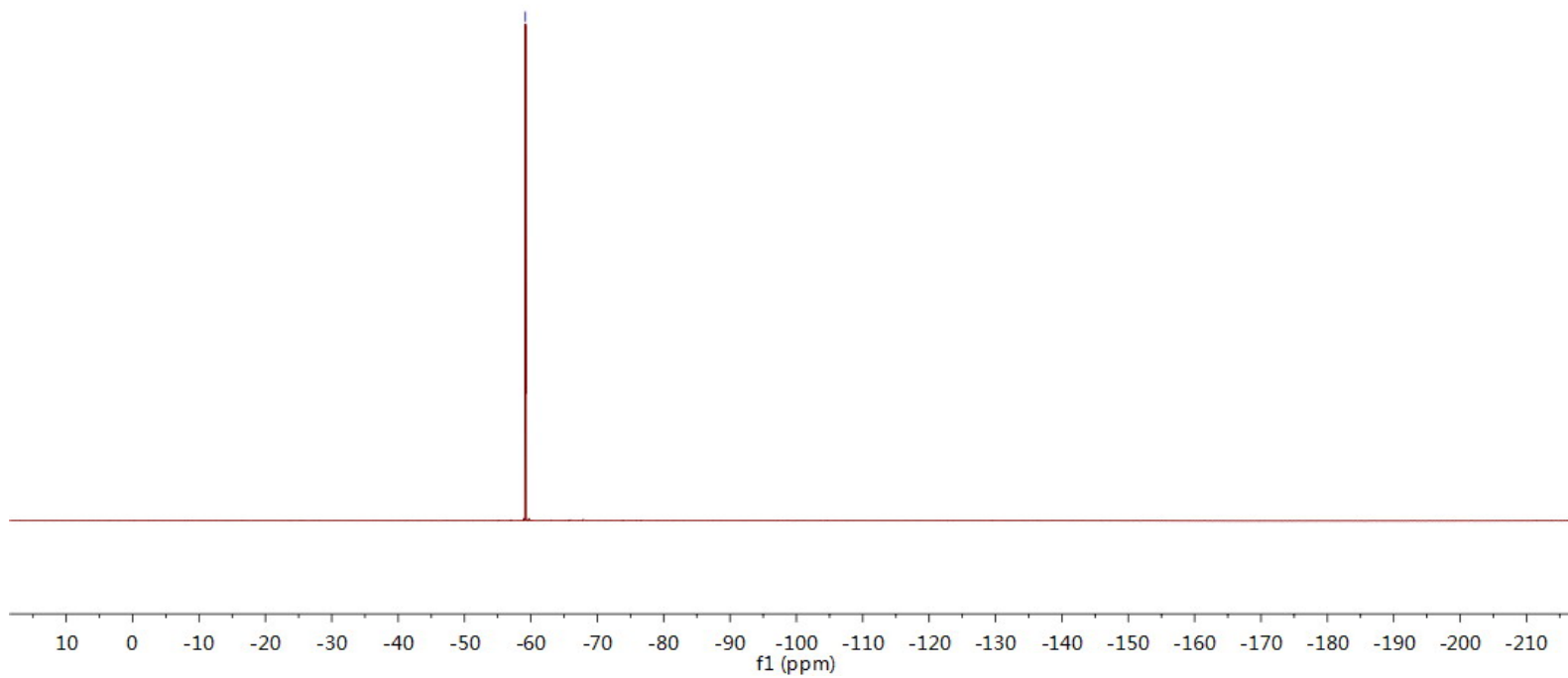
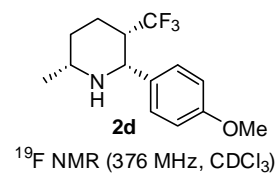


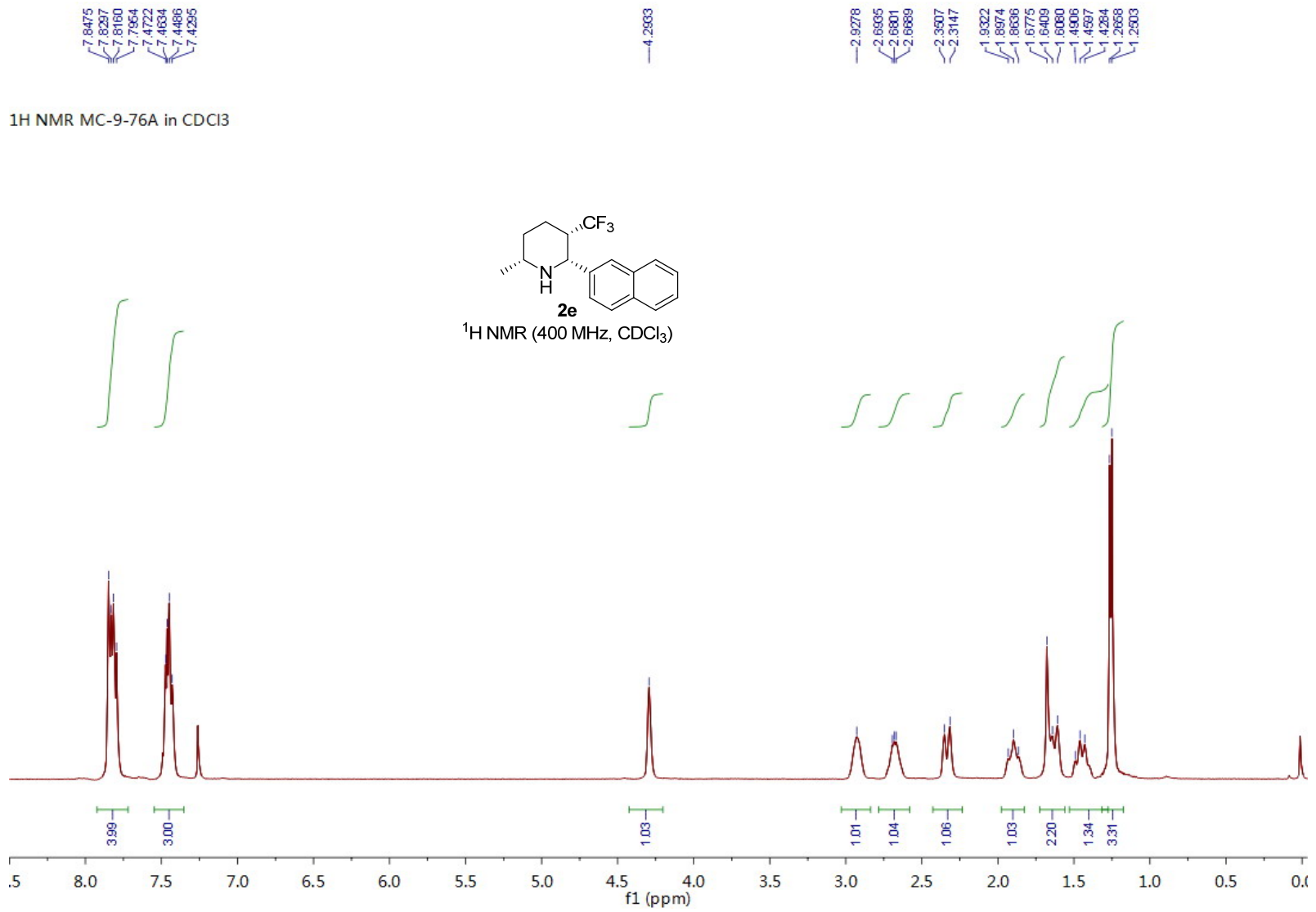


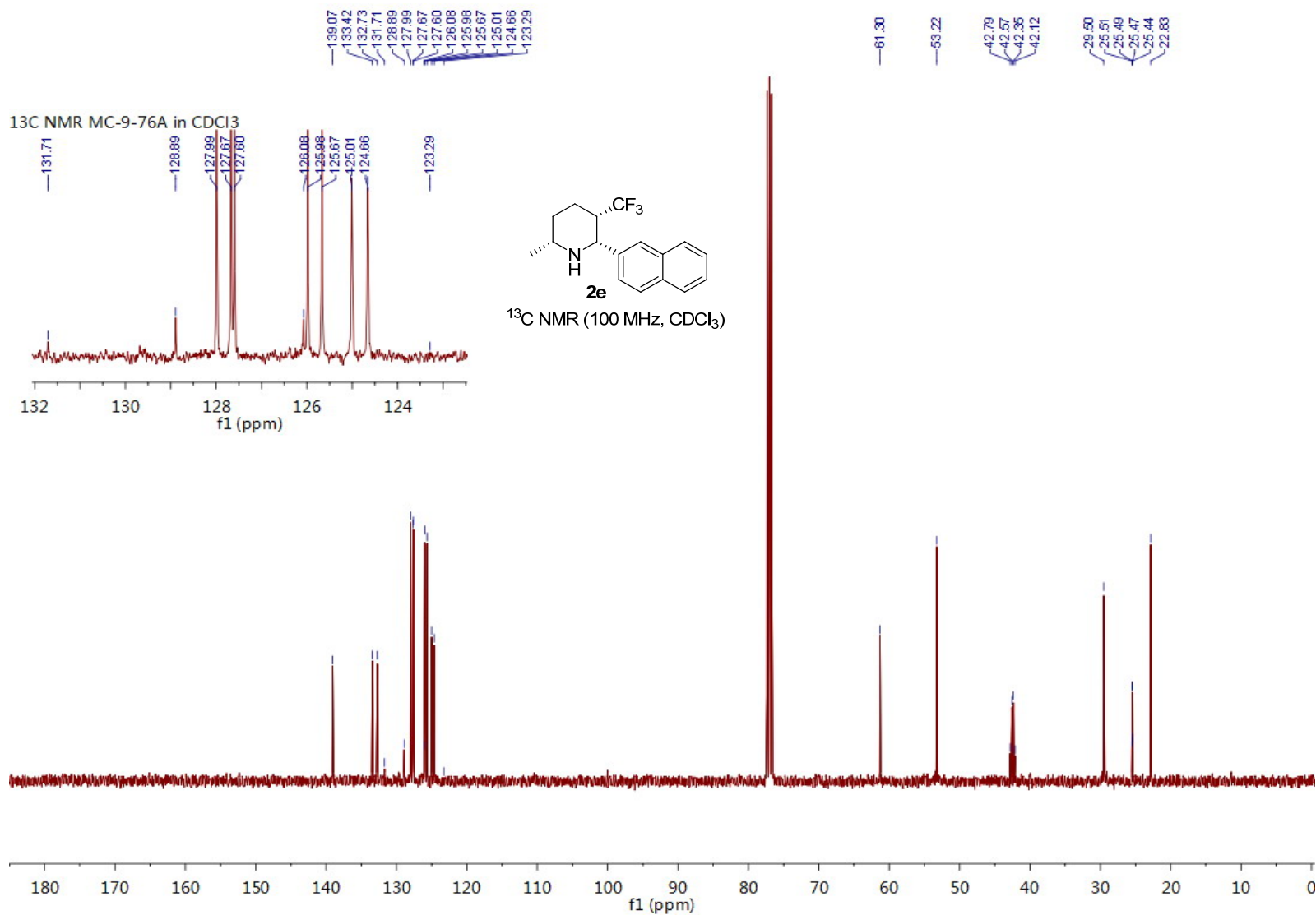


¹⁹F NMR MC-9-74A in CDCl₃

-59.1672

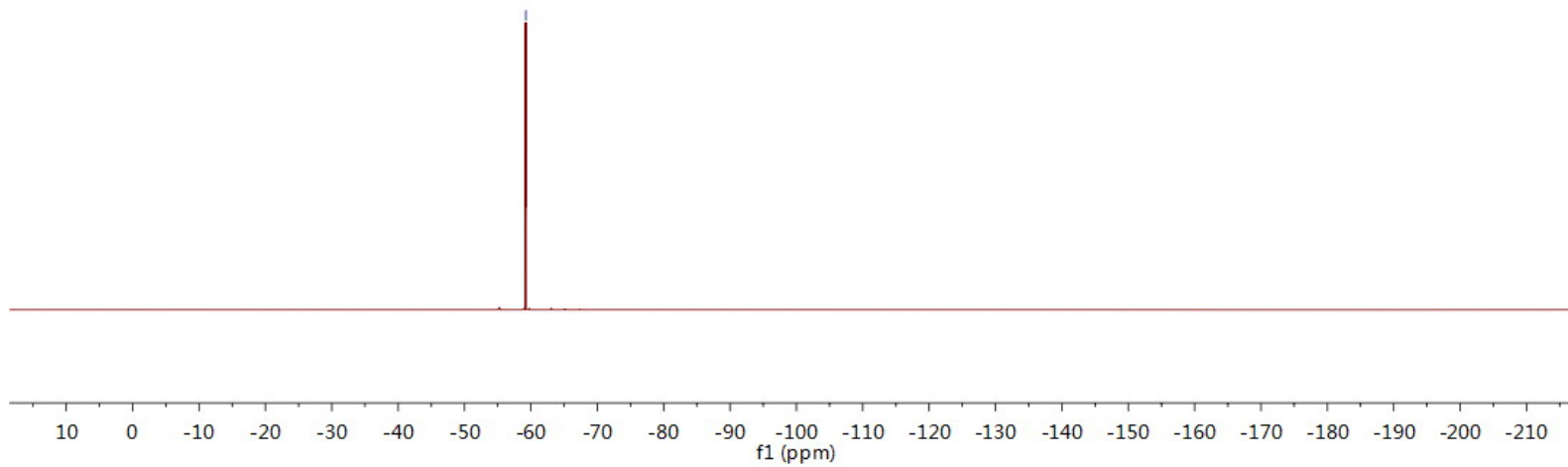
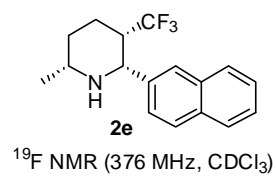


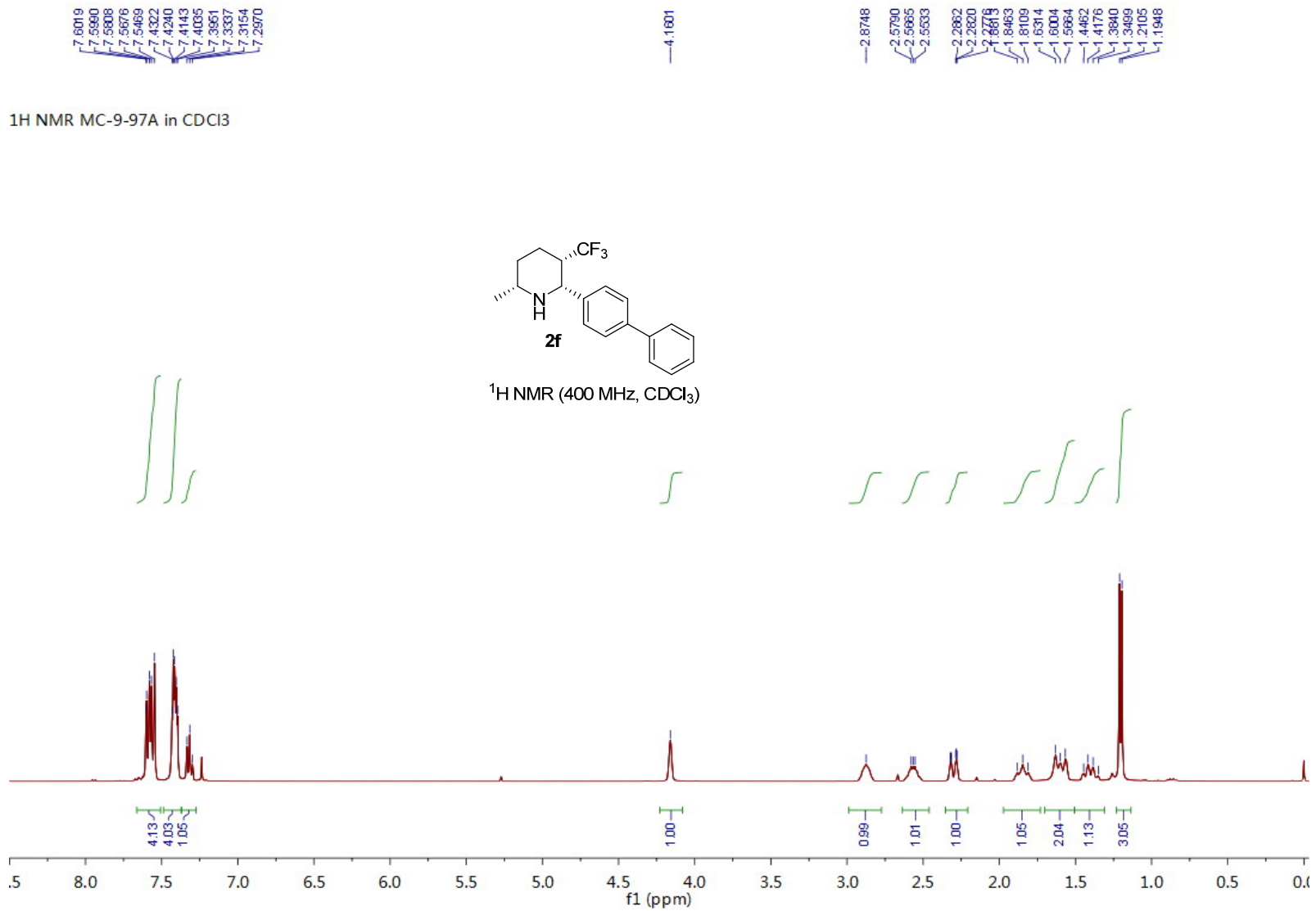


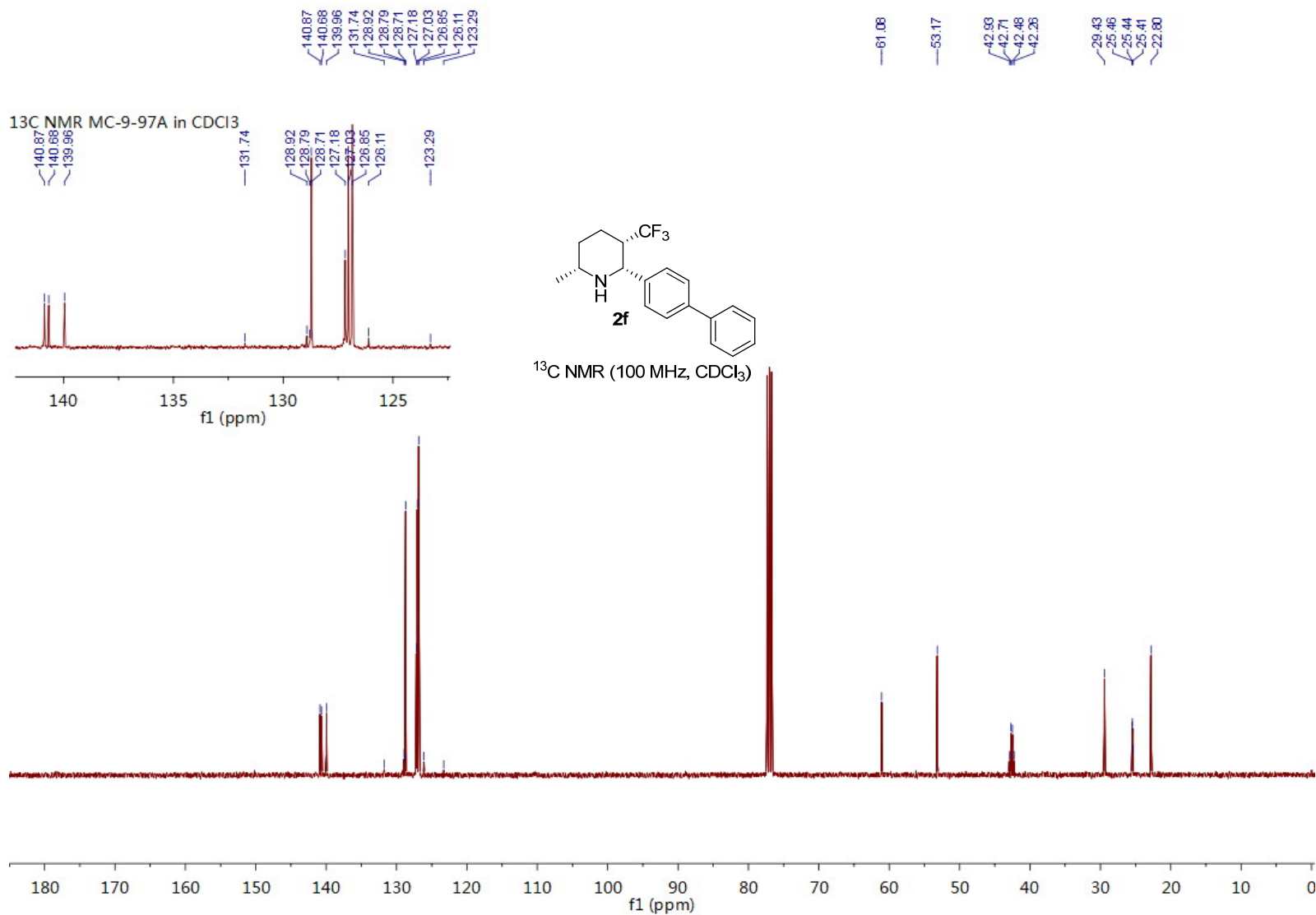


19F NMR MC-9-76A in CDCl3

-59.2342

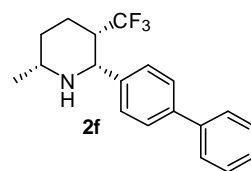




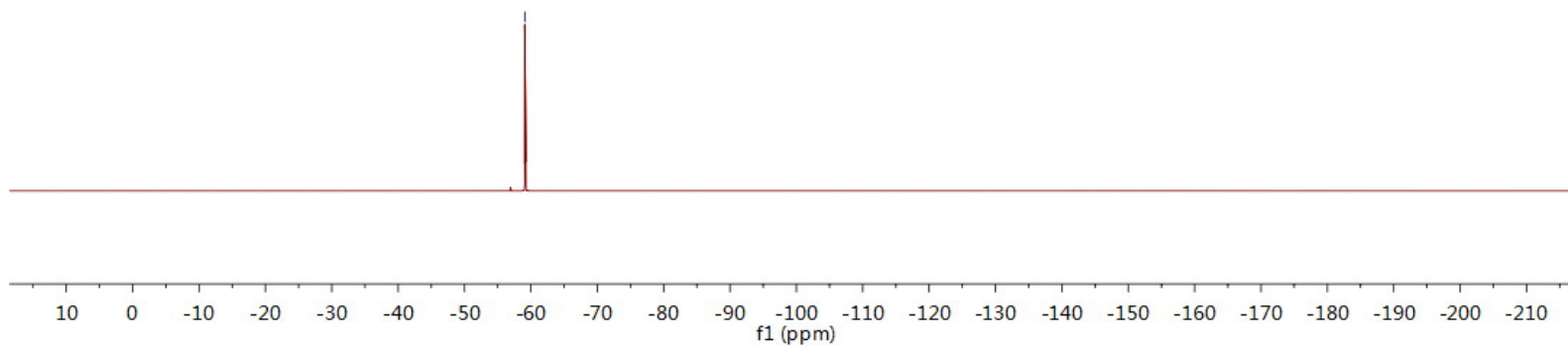


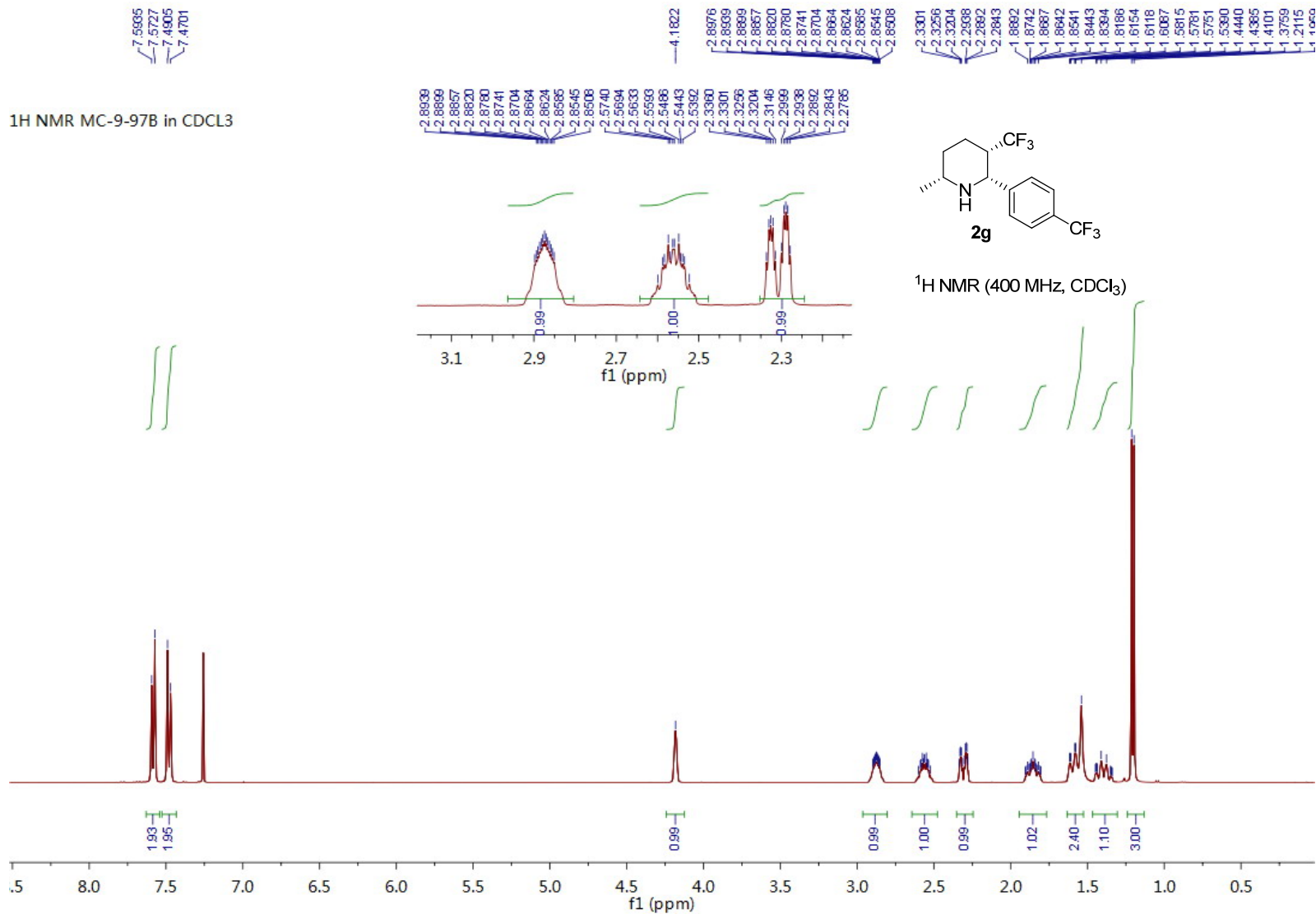
19F NMR MC-9-97A in CDCl3

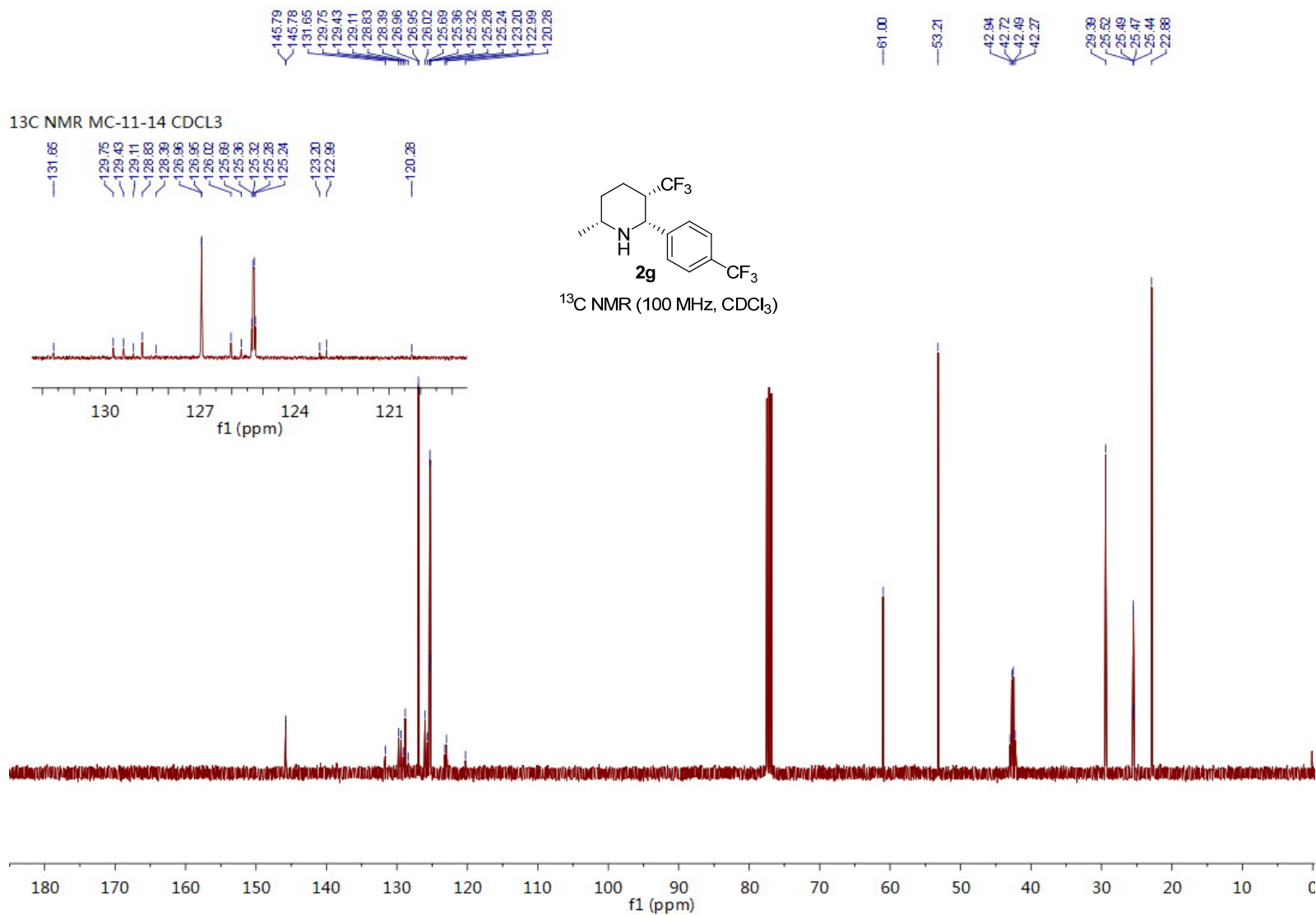
—59.0770



¹⁹F NMR (376 MHz, CDCl₃)



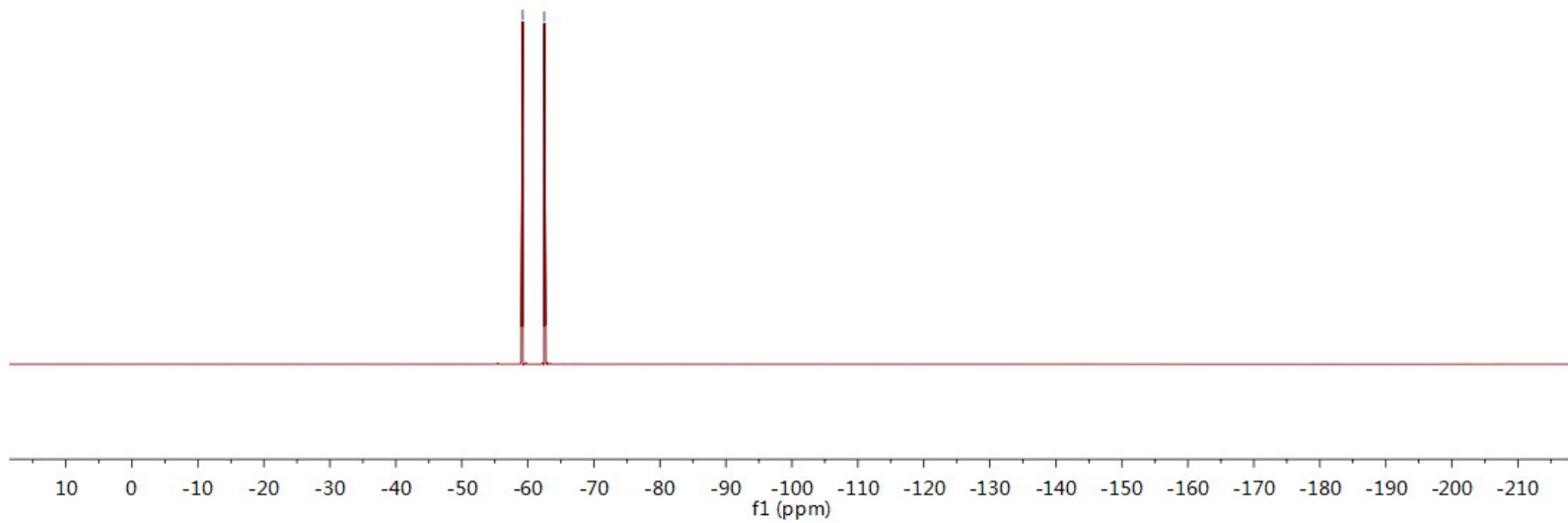
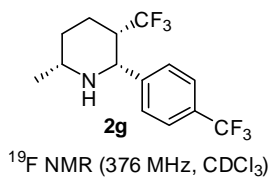




}

-59.2071
-62.4801

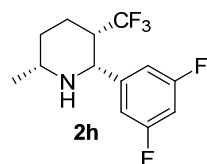
¹⁹F NMR MC-9-97B in CDCl₃



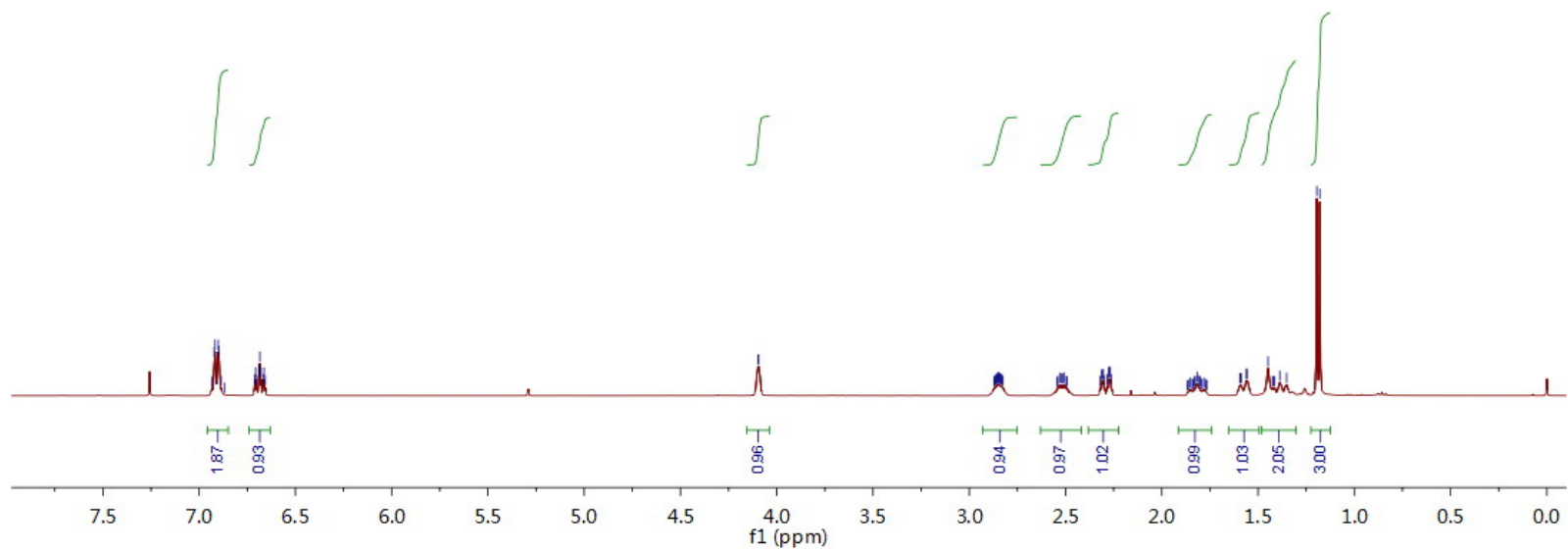
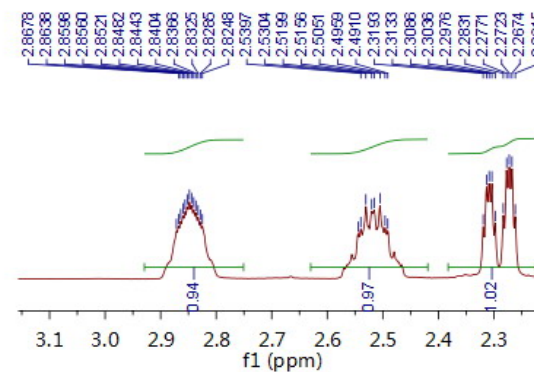
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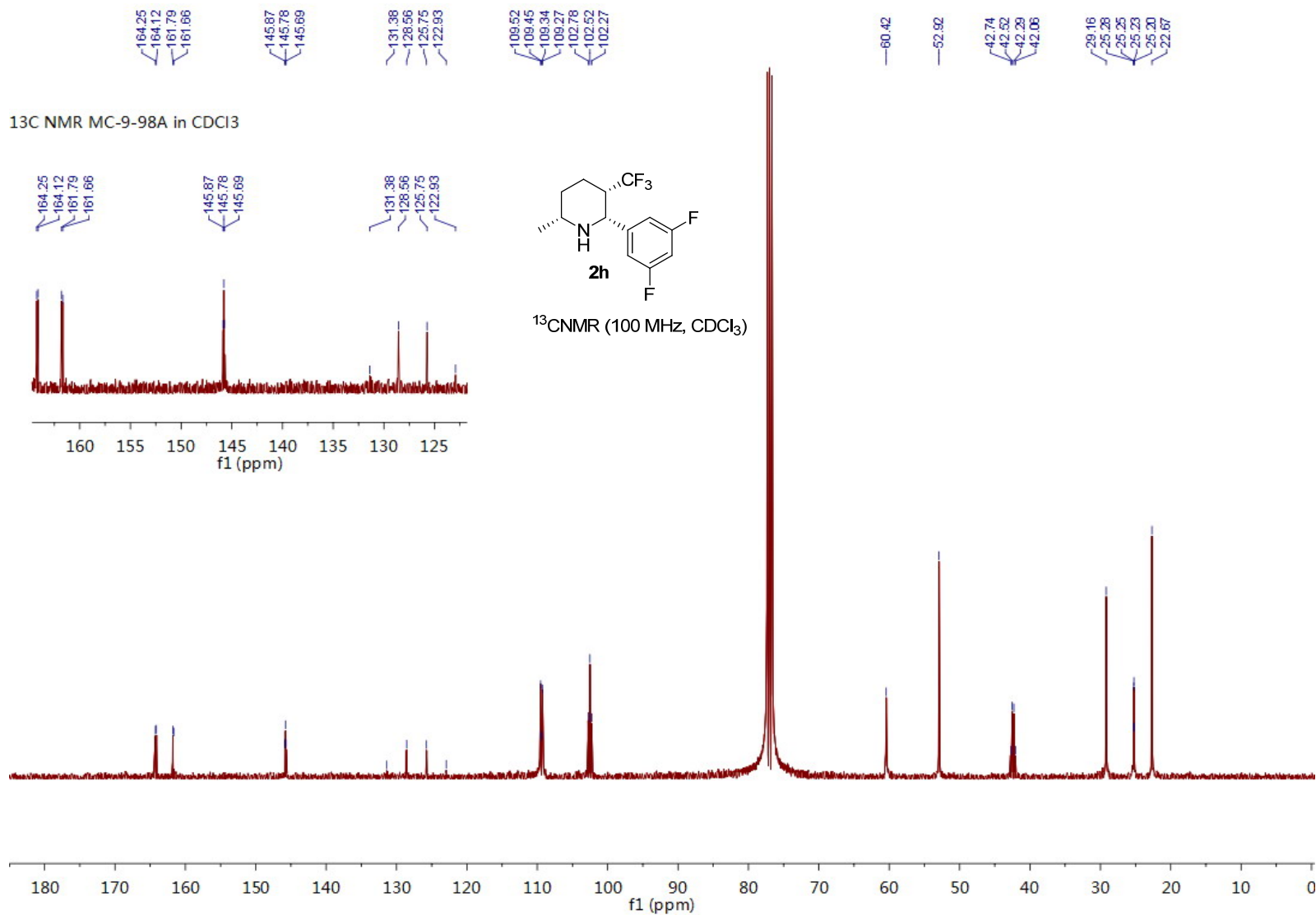
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¹H NMR MC-9-98A in CDCl₃

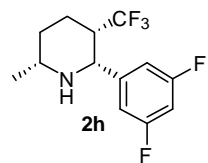


¹H NMR (400 MHz, CDCl₃)

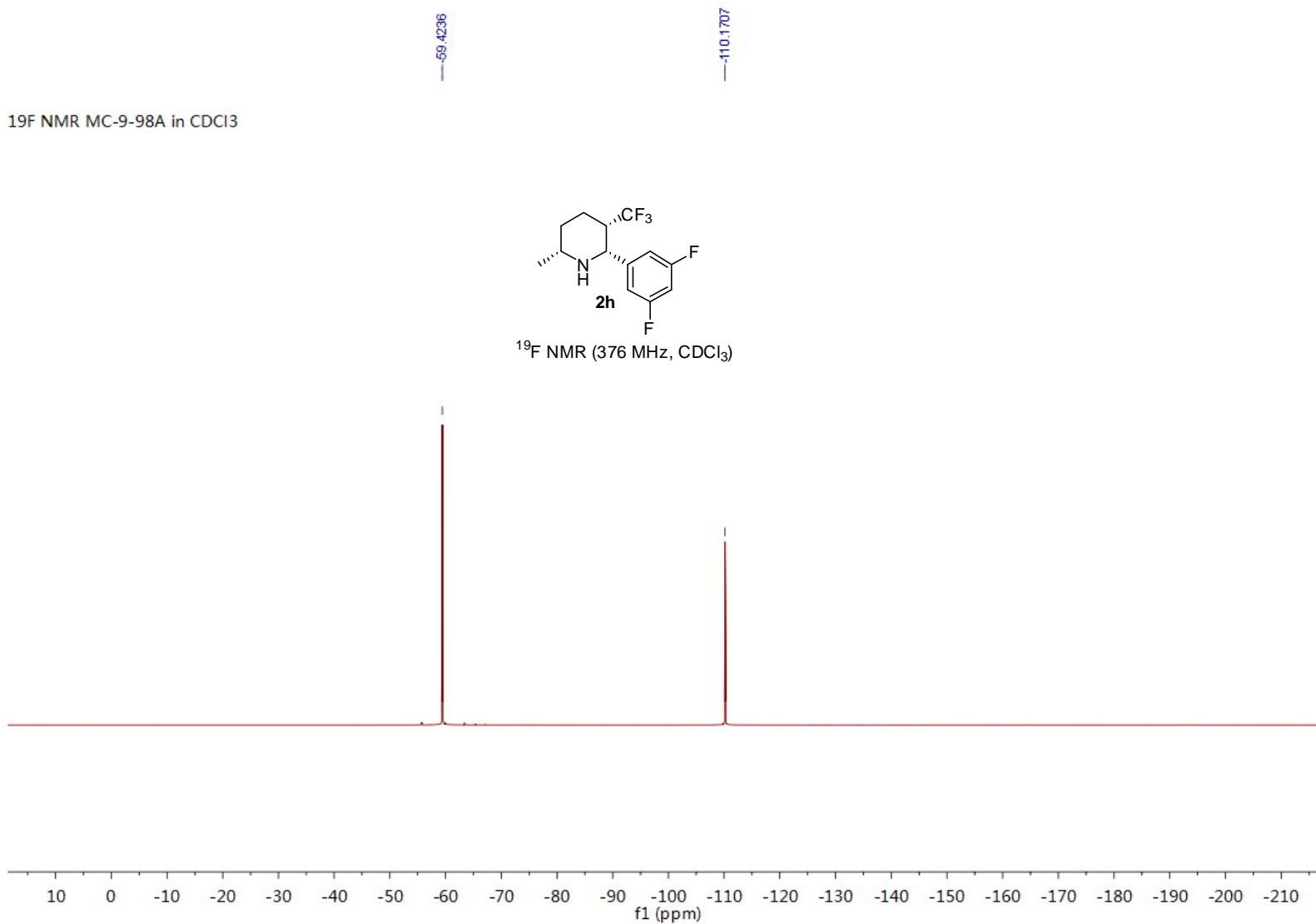


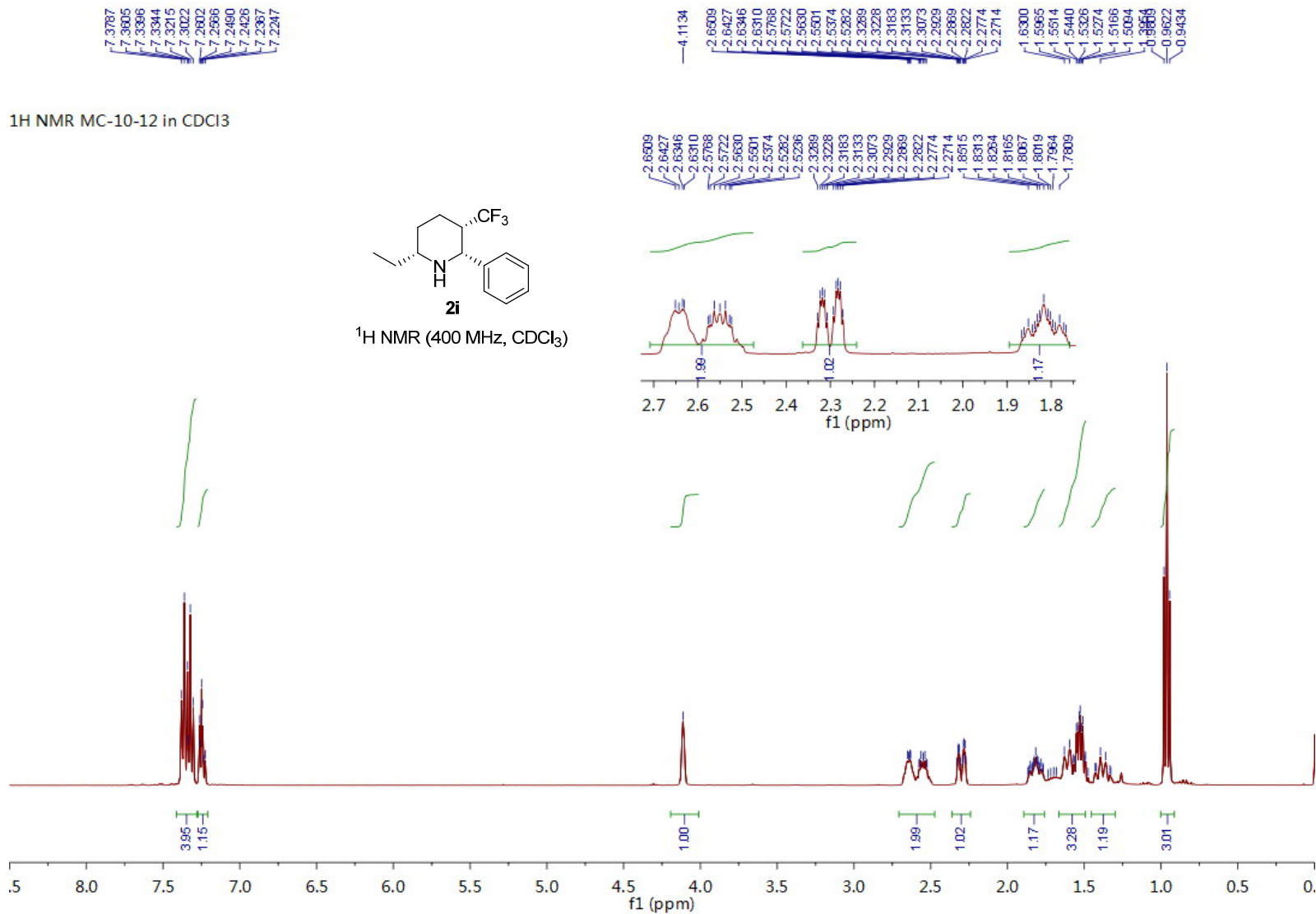


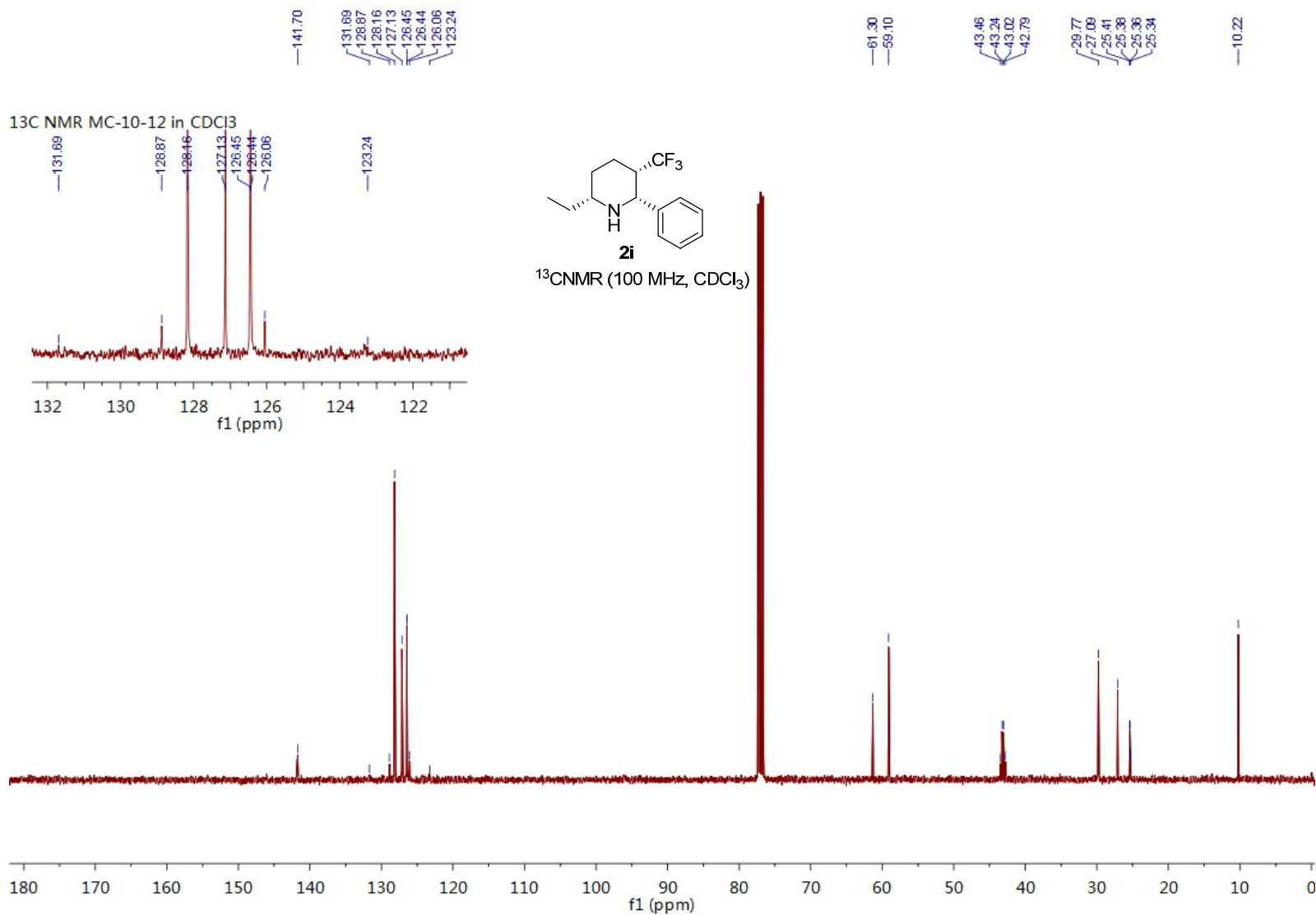
¹⁹F NMR MC-9-98A in CDCl₃



¹⁹F NMR (376 MHz, CDCl₃)

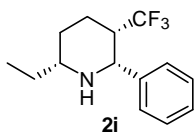




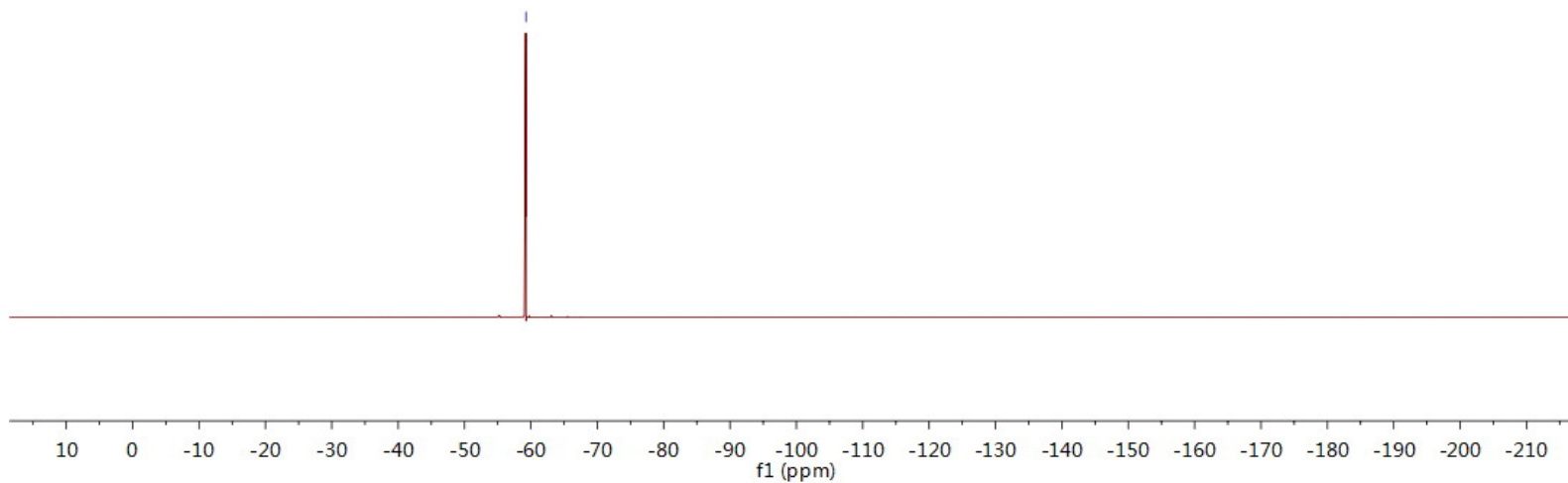


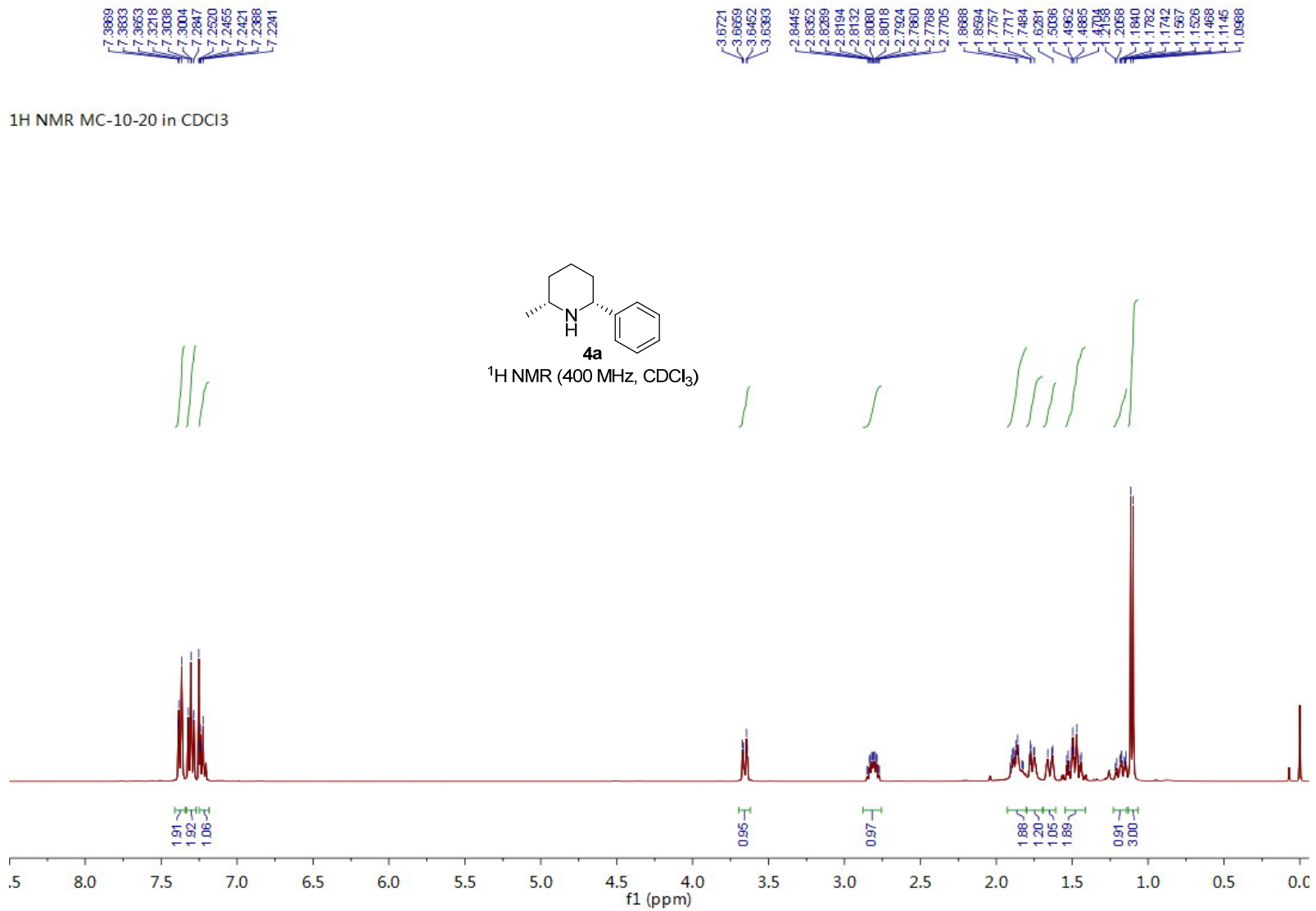
19F NMR MC-10-12 in CDCl3

1827.69

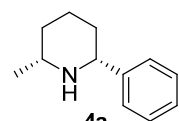


¹⁹F NMR (376 MHz, CDCl₃)

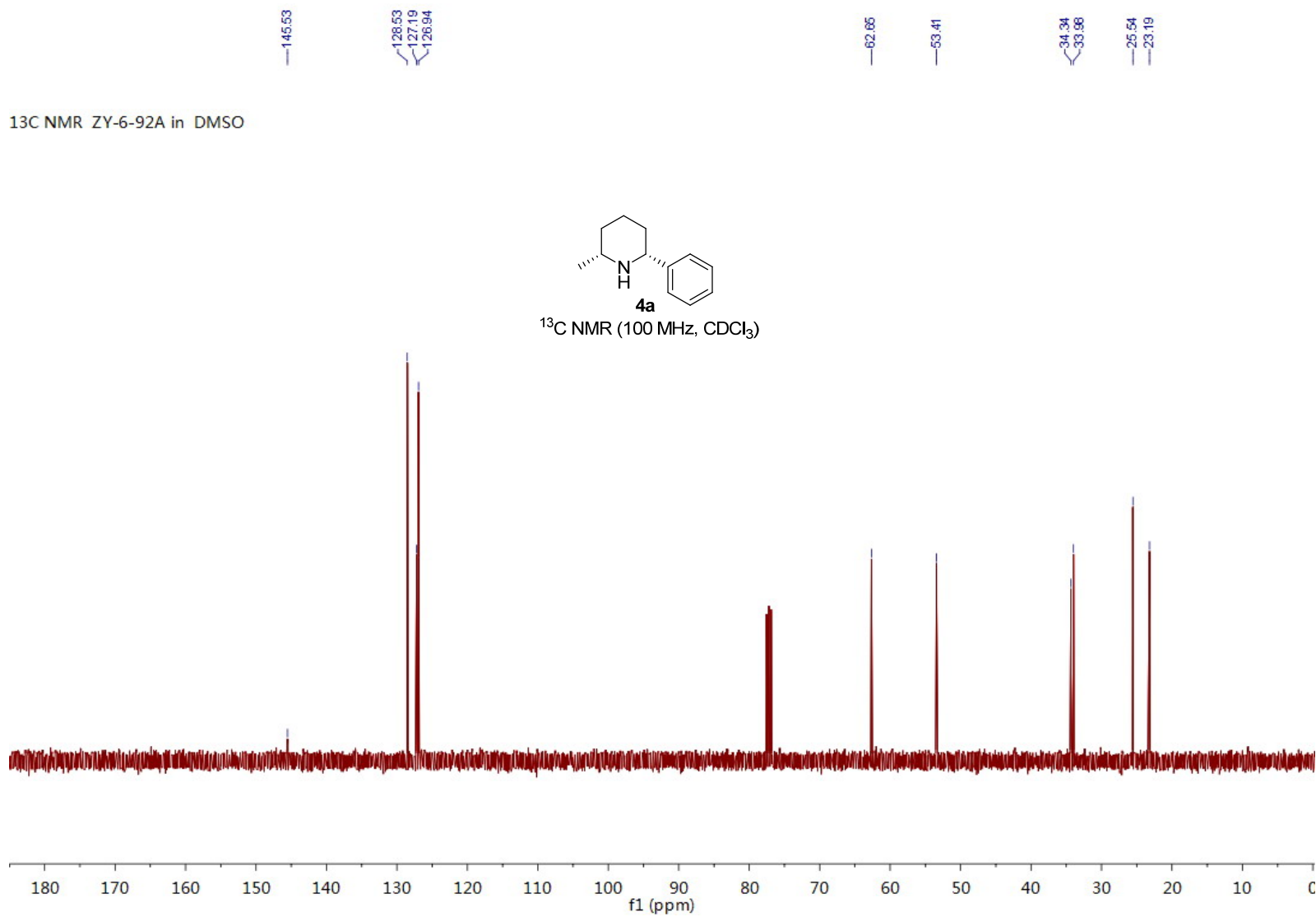




¹³C NMR ZY-6-92A in DMSO



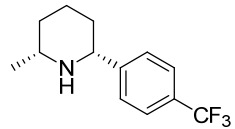
¹³C NMR (100 MHz, CDCl₃)



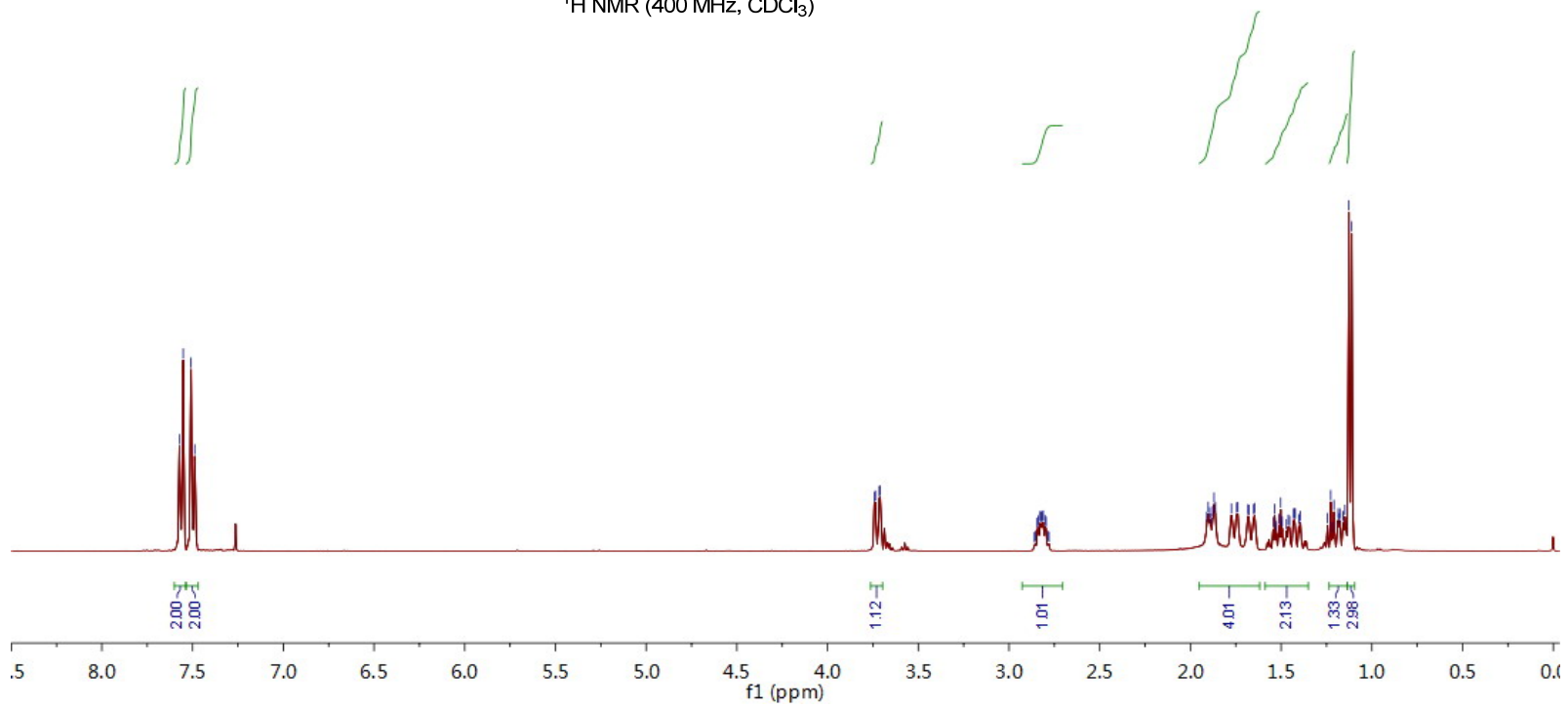
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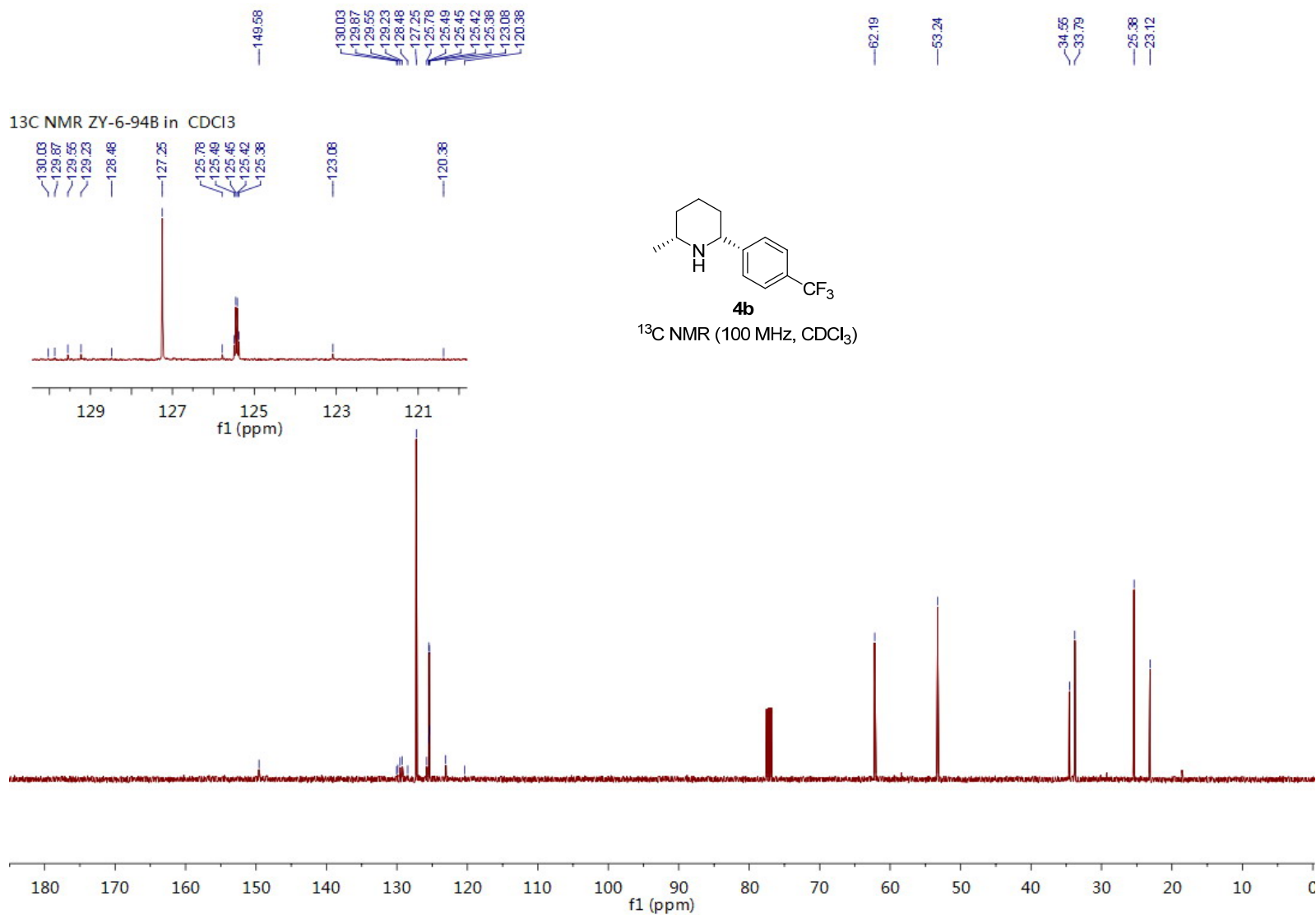
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¹H NMR ZY-6-94B in CDCl₃



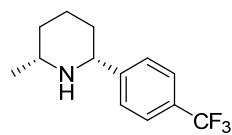
¹H NMR (400 MHz, CDCl₃)





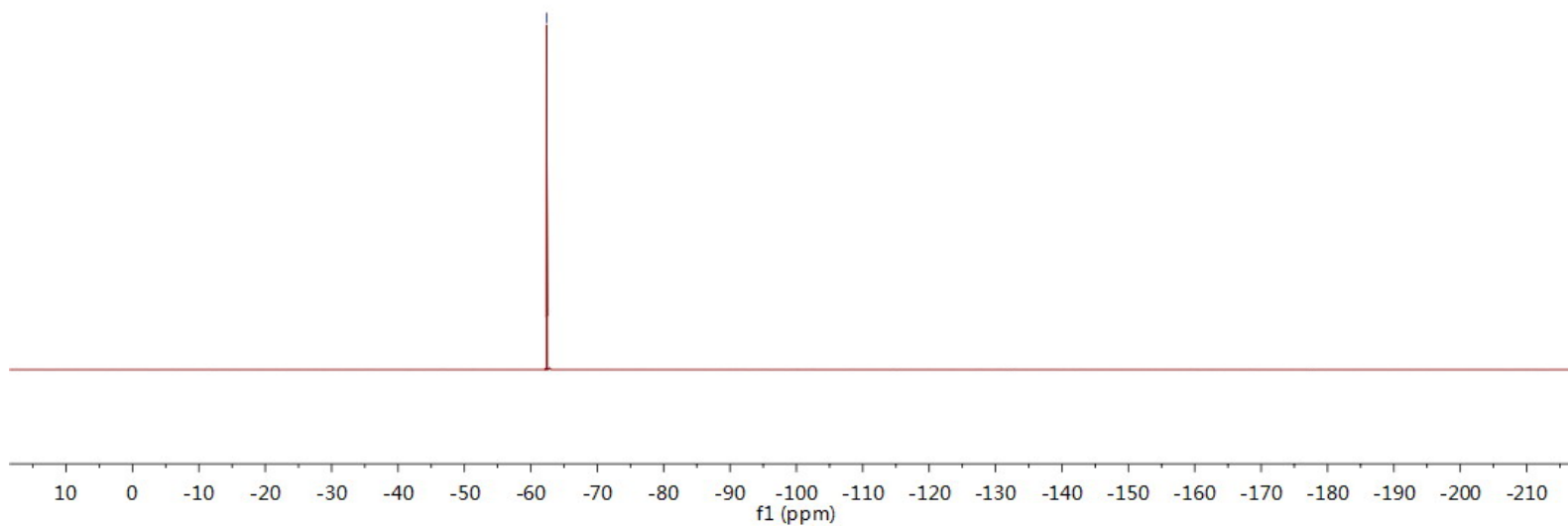
¹⁹F NMR ZY-6-94B in CDCl₃

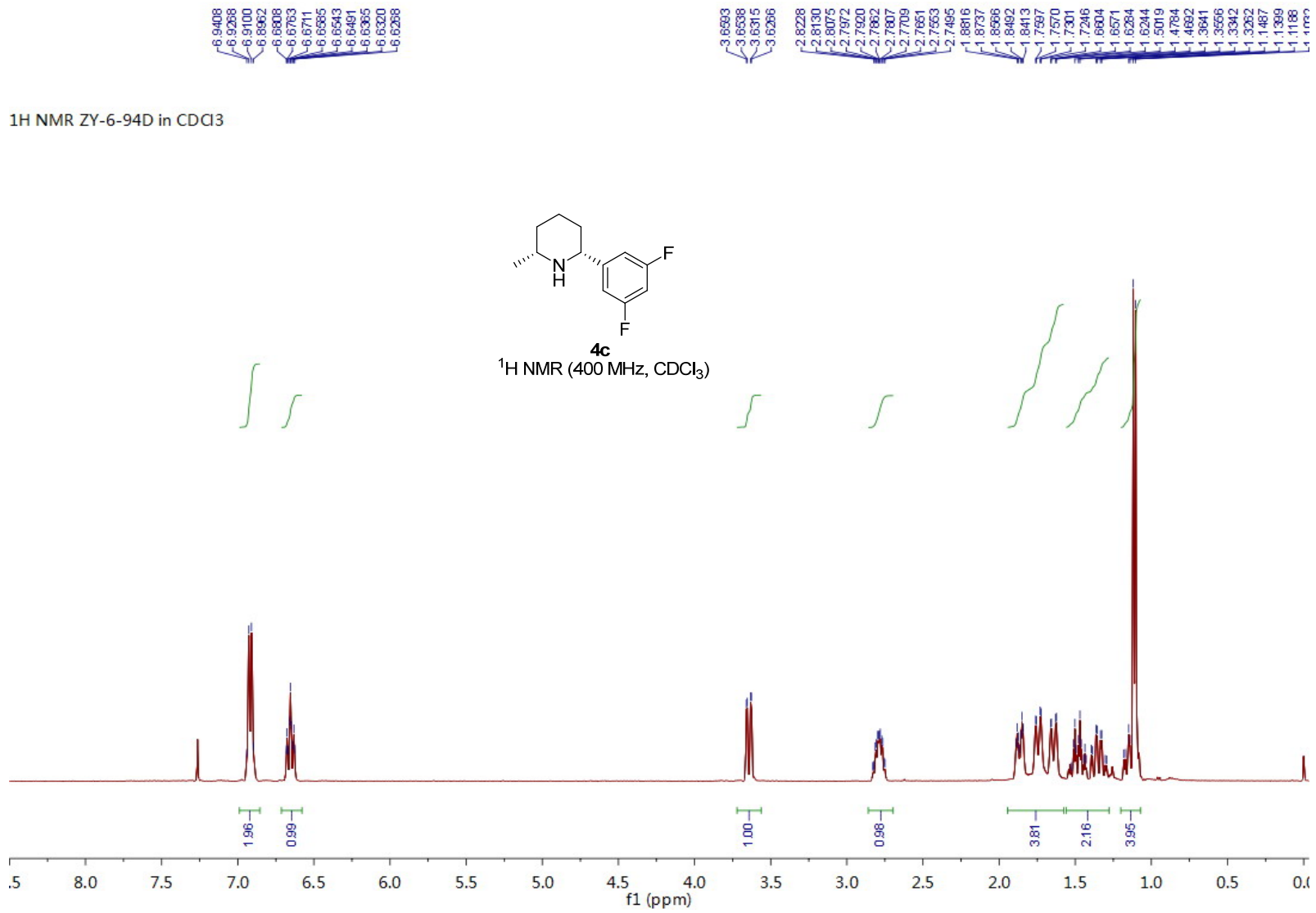
—62.3628

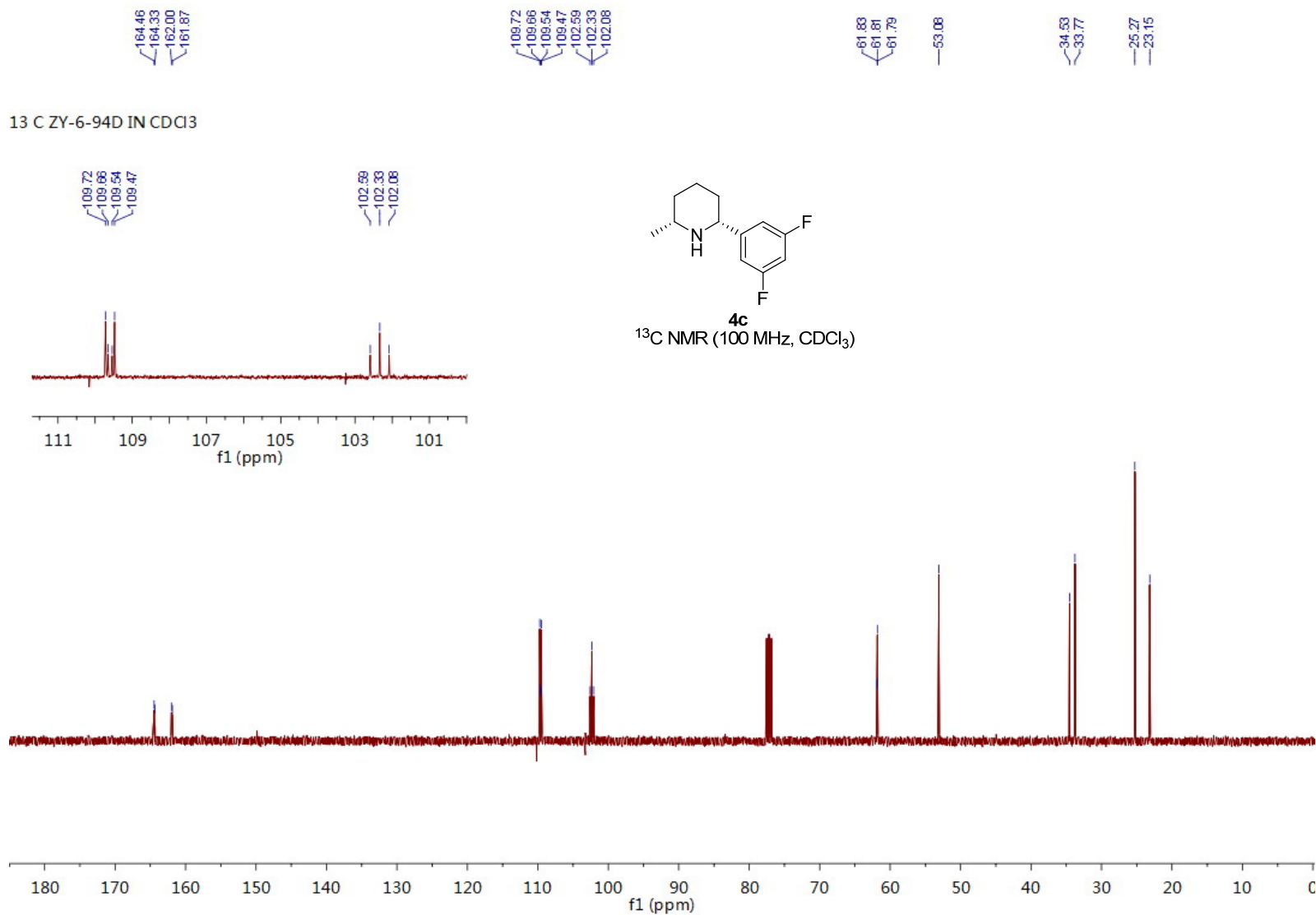


4b

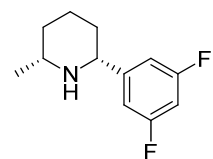
¹⁹F NMR (376 MHz, CDCl₃)





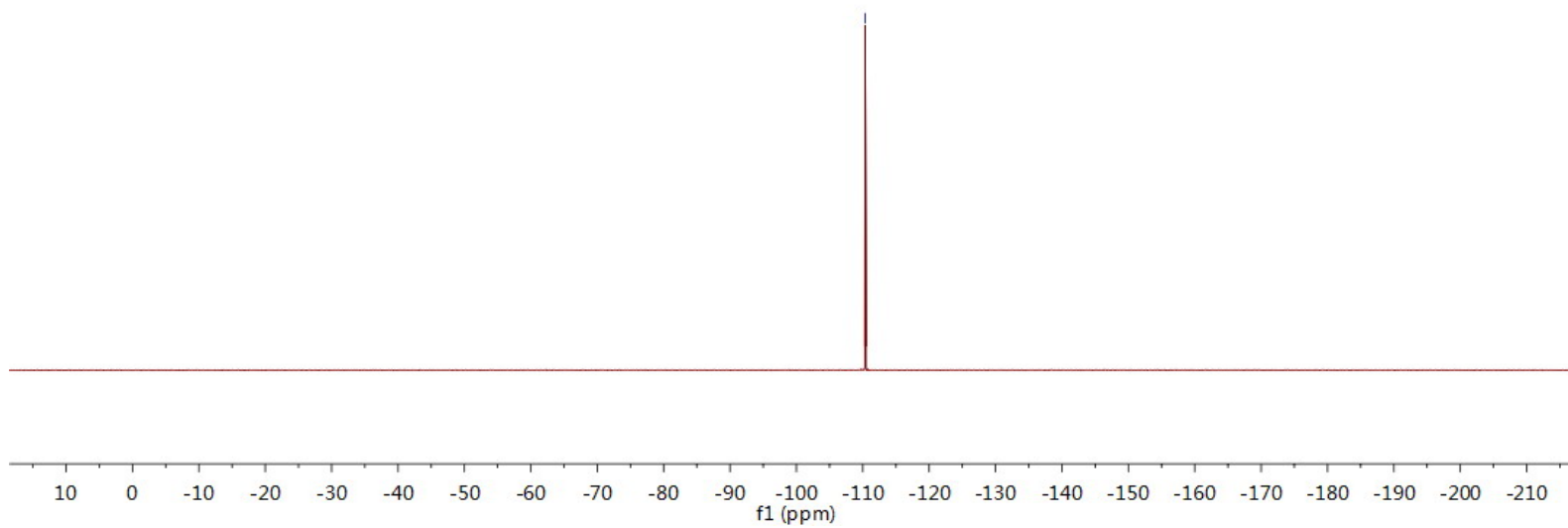


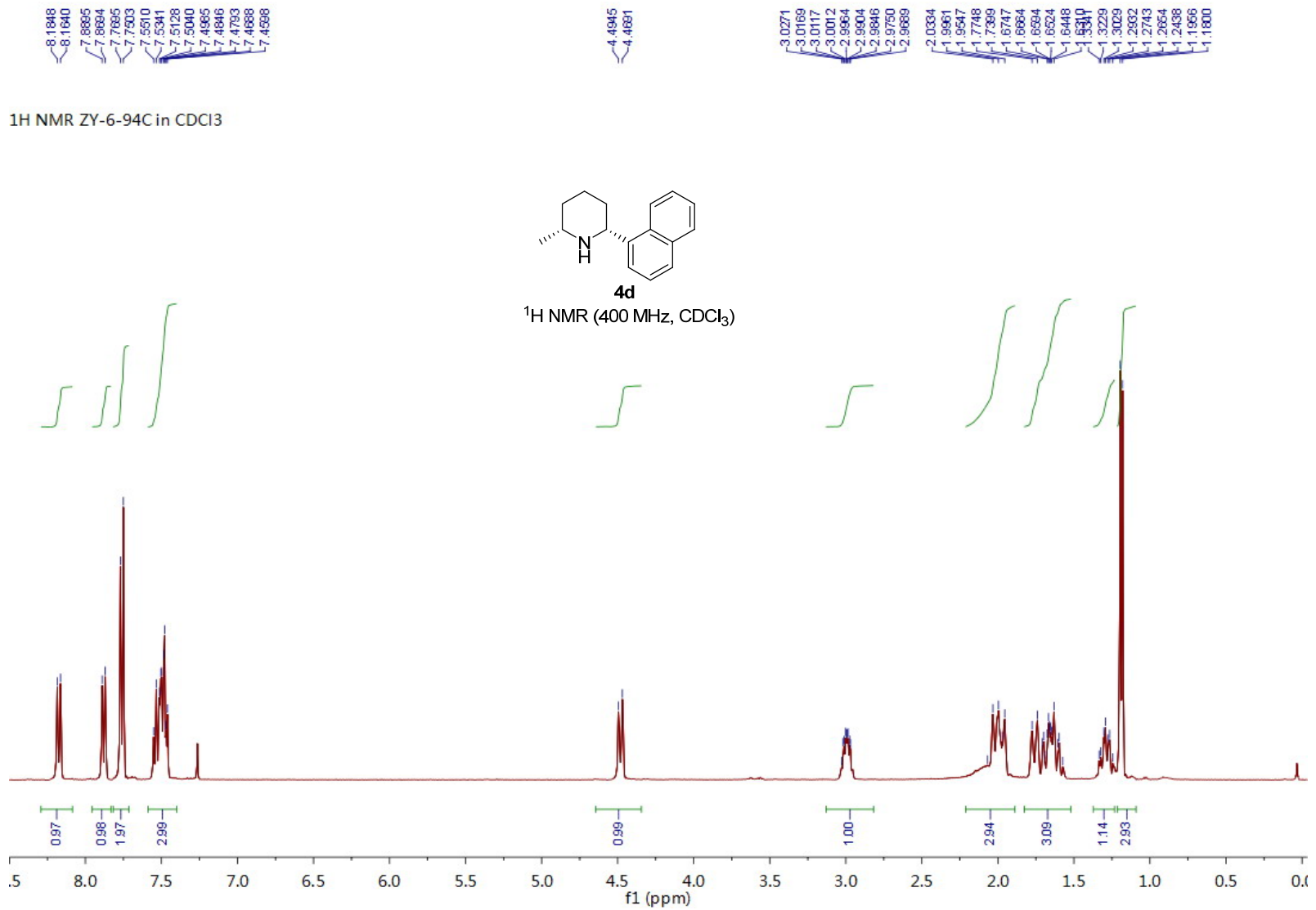
¹⁹F NMR ZY-6-94D in CDCl₃



4c
¹⁹F NMR (376 MHz, CDCl₃)

-110.3668





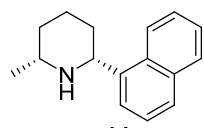
141.03
134.03
131.04
129.18
127.50
126.01
125.97
125.50
123.26
123.07

57.89
53.85

34.26
33.58

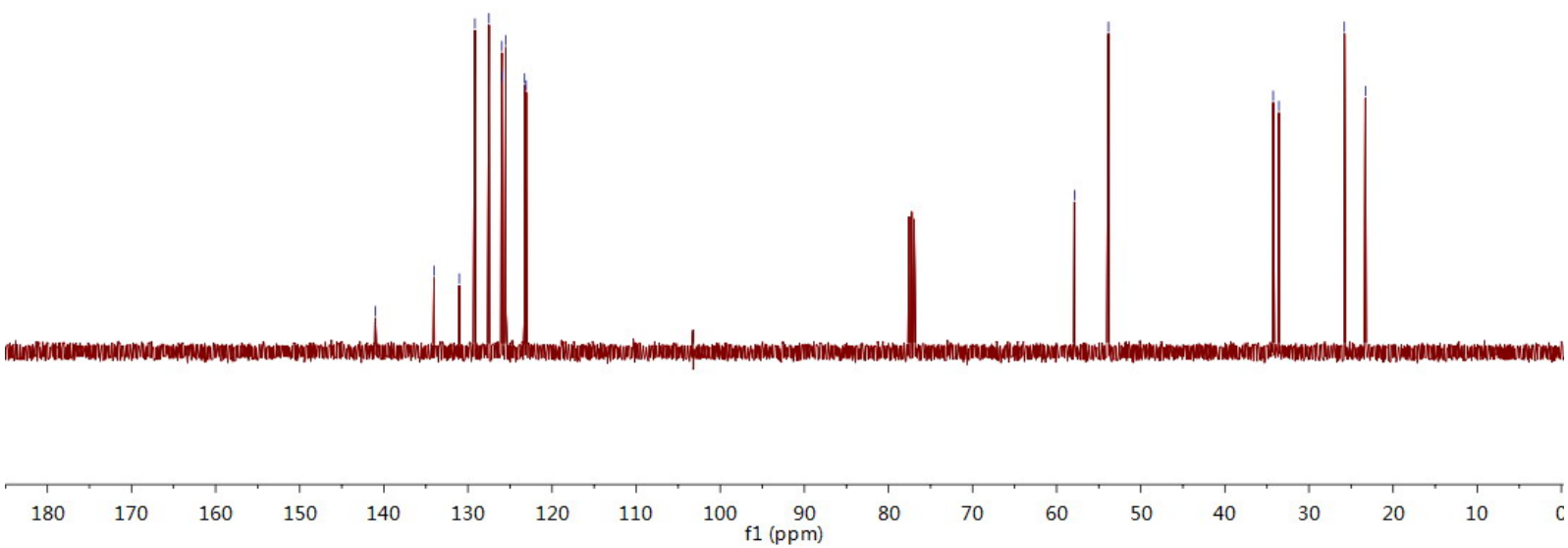
25.80
23.30

¹³C NMR ZY-6-94C in CDCl₃



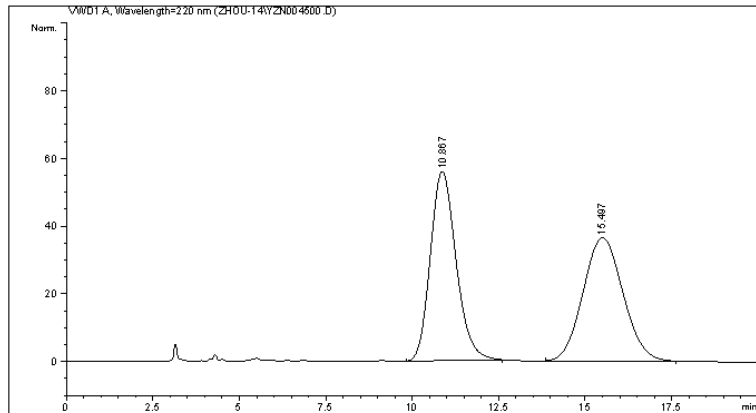
4d

¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-14\YZN004500.D
 Sample Name: HC-9-22B(+)

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 2/20/2014 4:48:53 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 2/20/2014 4:45:39 PM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 8/19/2014 9:15:31 AM by Z
 (modified after loading)
 Sample Info : 0J-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

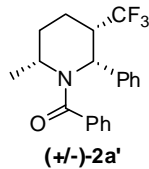
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	Height [mAU]	Area %
1	10.867	BB	0.8002	2896.43481	55.84499	49.6685
2	15.497	BB	1.2362	2935.10034	36.40302	50.3315

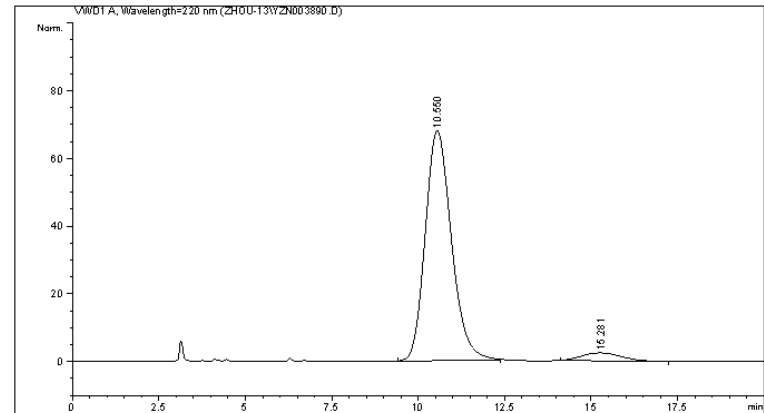
Totals : 5831.53516 92.24801

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN003890.D
 Sample Name: HC-9-58

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 11/5/2013 8:10:19 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 11/5/2013 8:08:05 PM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 8/19/2014 9:15:31 AM by Z
 (modified after loading)
 Sample Info : 0J-H, H/i-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

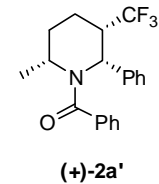
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	Height [mAU]	Area %
1	10.550	BB	0.8145	3575.78101	67.82823	95.1091
2	15.281	BB	0.9337	183.88016	2.32304	4.8909

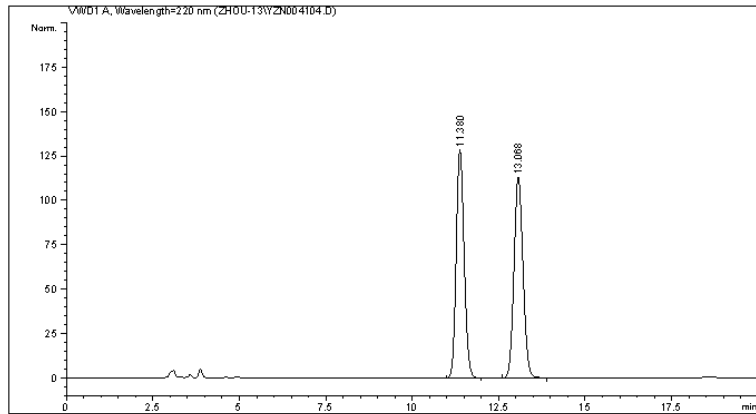
Totals : 3759.66116 70.15127

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004104.D
 Sample Name: HC-9-74E+

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 12/2/2013 10:29:28 AM
 Acq. Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 12/2/2013 10:26:24 AM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 8/19/2014 9:24:36 AM by Z
 (modified after loading)
 Sample Info : AD-H, H/i-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

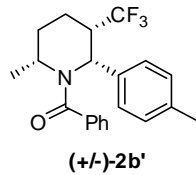
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.380	BB	0.2470	2046.13550	128.45103	49.8448
2	13.068	BB	0.2822	2058.87915	113.13163	50.1552

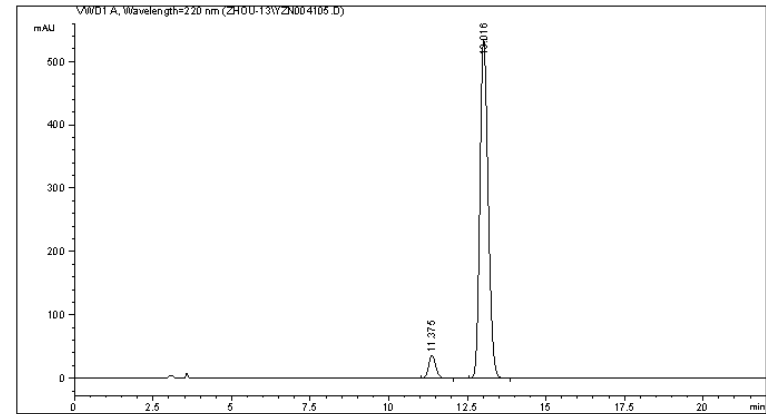
Totals : 4105.01465 241.58266

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004105.D
 Sample Name: HC-9-74E

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 12/2/2013 10:57:06 AM
 Acq. Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 12/2/2013 10:54:55 AM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 7/11/2014 3:35:13 PM by Z
 (modified after loading)
 Sample Info : AD-H, H/i-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

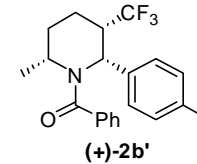
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.375	BB	0.2469	584.09735	36.68600	5.6356
2	13.016	BB	0.2849	9780.35449	534.22443	94.3644

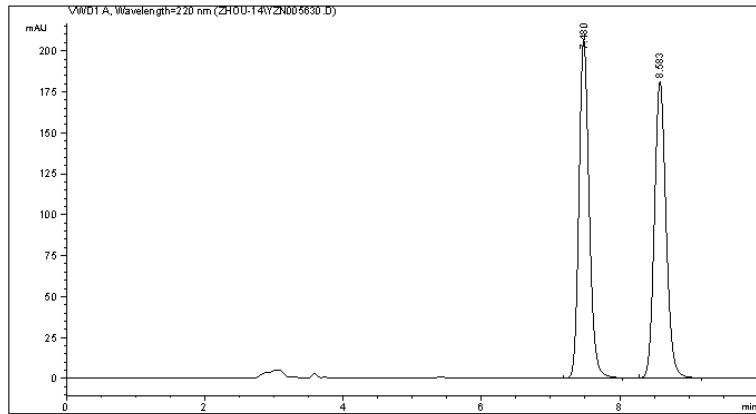
Totals : 1.03645e4 570.91042

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 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-14\YZN005630.D
 Sample Name: MC-9-76B+

=====
 Acq. Operator : Z
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 7/22/2014 2:08:32 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 7/22/2014 2:04:54 PM by Z
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 7/22/2014 3:09:32 PM by Z
 (modified after loading)
 Sample Info : AD-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

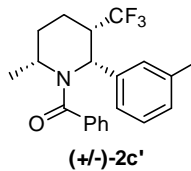
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.480	BB	0.1572	2111.59448	206.49776	49.9787
2	8.583	BB	0.1800	2113.39648	180.87971	50.0213

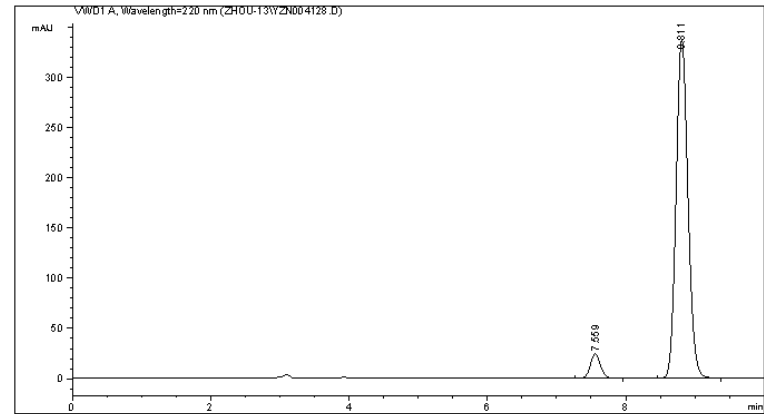
Totals : 4224.99097 387.37747

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 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004128.D
 Sample Name: MC-7-76B

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 12/3/2013 4:18:57 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 12/3/2013 4:16:05 PM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 7/11/2014 3:41:44 PM by Z
 (modified after loading)
 Sample Info : AD-H, H/i-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

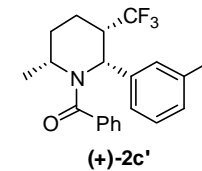
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.559	BB	0.1585	253.18146	24.47709	5.8976
2	8.811	BB	0.1863	4039.81006	337.31100	94.1024

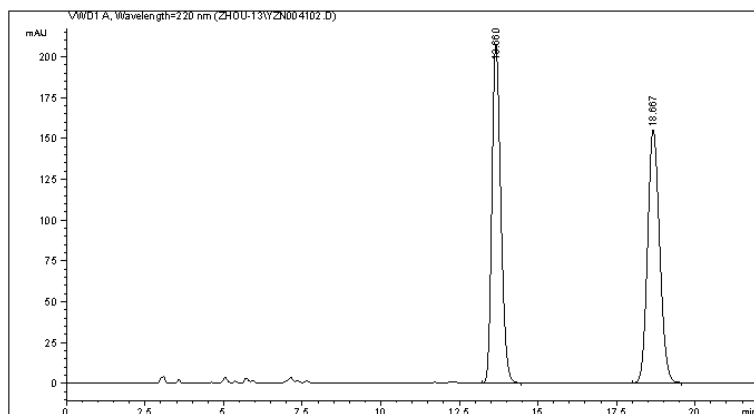
Totals : 4292.99152 361.78810

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 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004102.D
 Sample Name: HC-9-74A--

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 12/2/2013 9:31:31 AM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 12/2/2013 9:30:17 AM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 7/11/2014 3:35:13 PM by Z
 (modified after loading)
 Sample Info : AD-H, H/i-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

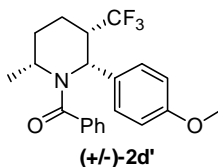
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.660	BB	0.3058	4080.51929	206.94737	49.7482
2	18.667	BB	0.4141	4121.82764	154.77292	50.2518

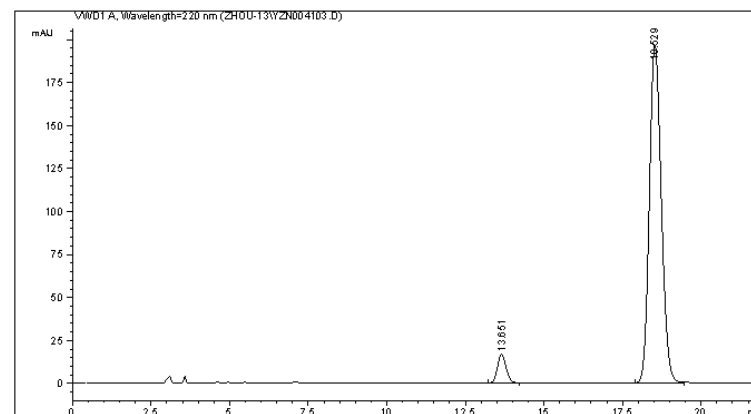
Totals : 8202.34692 361.72029

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004103.D
 Sample Name: HC-9-74A

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 12/2/2013 10:03:38 AM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 12/2/2013 10:02:01 AM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 7/11/2014 3:35:13 PM by Z
 (modified after loading)
 Sample Info : AD-H, H/i-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

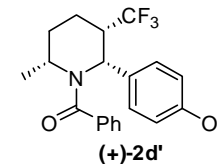
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.651	BB	0.2991	321.29056	16.67215	5.8571
2	18.529	BB	0.4086	5164.19824	196.44943	94.1429

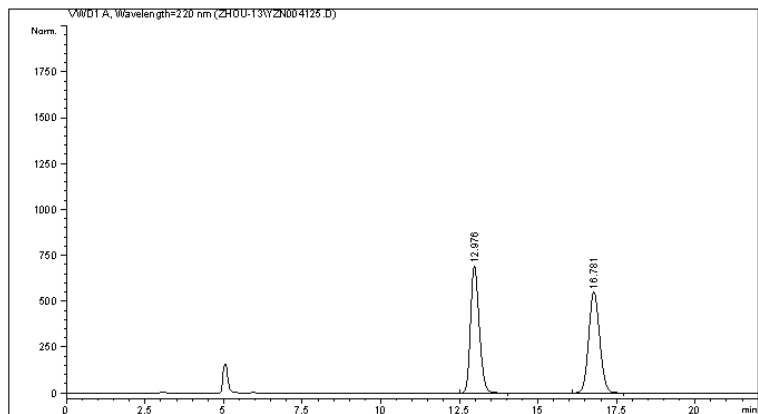
Totals : 5485.48880 213.12159

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 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004125.D
 Sample Name: MC-7-76A+

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 12/3/2013 3:05:00 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 12/3/2013 2:48:45 PM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 8/19/2014 10:04:46 AM by Z
 (modified after loading)
 Sample Info : AD-H, H/i-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

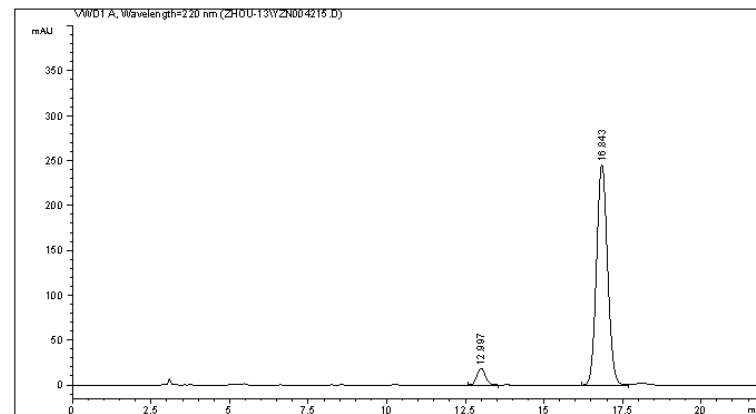
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.976	BB	0.2970	1.32771e4	690.87012	49.9083
2	16.781	VB	0.3779	1.33259e4	548.90643	50.0917

Totals : 2.66030e4 1239.77655

=====
 *** End of Report ***

Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004215.D
 Sample Name: MC-9-81A

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 12/11/2013 3:28:56 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 12/11/2013 3:25:08 PM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 7/11/2014 4:24:07 PM by Z
 (modified after loading)
 Sample Info : AD-H, H/i-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

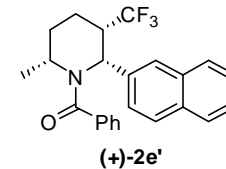
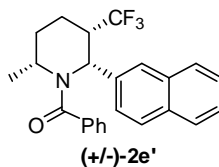
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.997	BB	0.2948	355.76962	18.81668	5.6055
2	16.843	BB	0.3791	5991.01758	245.72809	94.3945

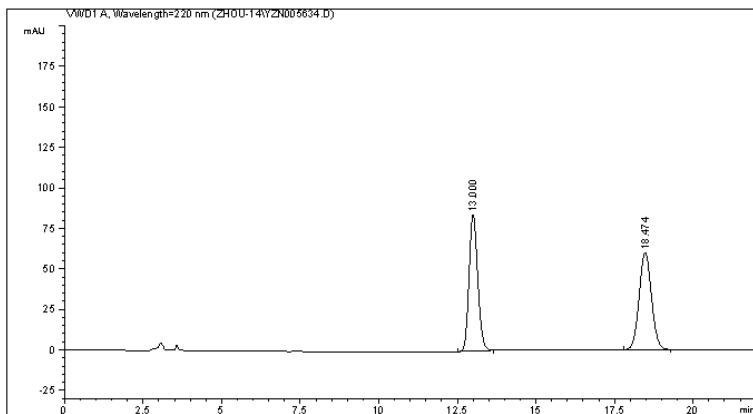
Totals : 6346.78720 264.54477

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-14\YZN005634.D
 Sample Name: MC-9-97A+-

=====
 Acq. Operator : Z
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 7/22/2014 5:42:07 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 7/22/2014 5:35:20 PM by Z
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 7/22/2014 6:10:08 PM by Z
 (modified after loading)
 Sample Info : AD-H , H/i-PrOH = 90/10, 1.0mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

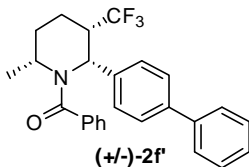
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	13.000	BB	0.2986	1622.31836	84.37788	49.8869
2	18.474	BB	0.4235	1629.67590	59.93539	50.1131

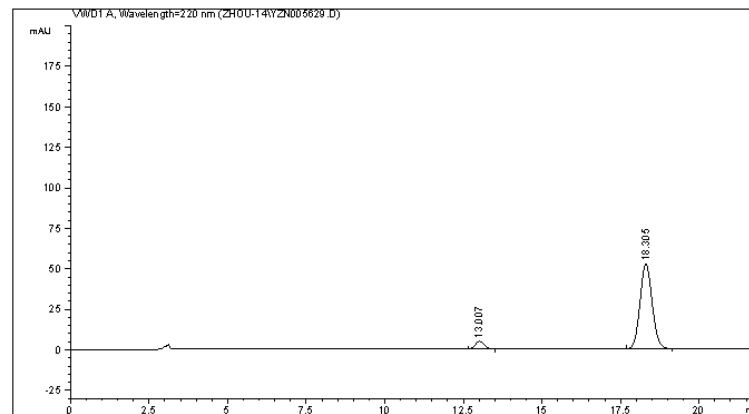
Totals : 3251.99426 144.31327

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-14\YZN005629.D
 Sample Name: MC-9-97a

=====
 Acq. Operator : Z
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 7/22/2014 1:38:02 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 7/22/2014 1:36:40 PM by Z
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 7/22/2014 6:10:08 PM by Z
 (modified after loading)
 Sample Info : AD-H , H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

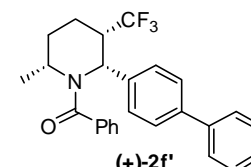
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	13.007	BB	0.2988	90.31953	4.72265	6.0443
2	18.305	BB	0.4155	1403.97449	52.48493	93.9557

Totals : 1494.29401 57.20758

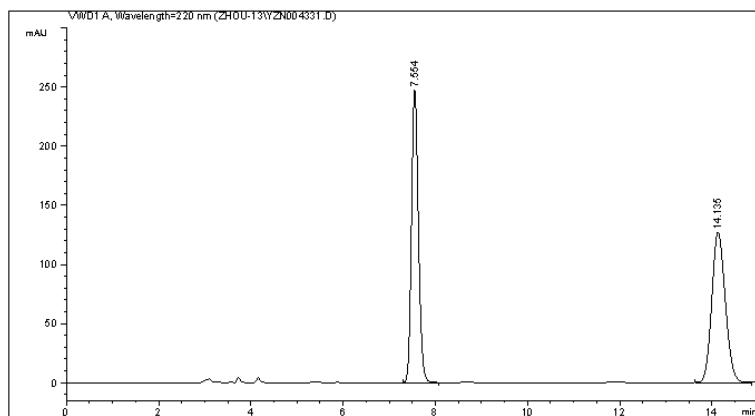
=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004331.D
 Sample Name: MC-9-97B+-

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=====
Acq. Operator   : B
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 12/31/2013 10:25:27 AM
Acq. Method    : C:\CHEM32\1\METHODS\DEF.LC.M
Last changed   : 12/31/2013 10:24:16 AM by B
                (modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF.LC.M
Last changed   : 7/11/2014 4:35:58 PM by Z
                (modified after loading)
Sample Info    : AD-H, H/i-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220 nm
=====
  
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 Area Percent Report
 =====

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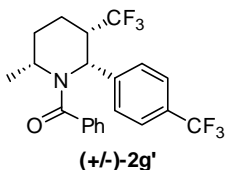
Sorted By      : Signal
Multiplier:    : 1.0000
Dilution:      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	7.554	BB	0.1624	2580.61621	247.43379	49.9413
2	14.135	BB	0.3177	2586.68237	126.99229	50.0587

Totals : 5167.29858 374.42609

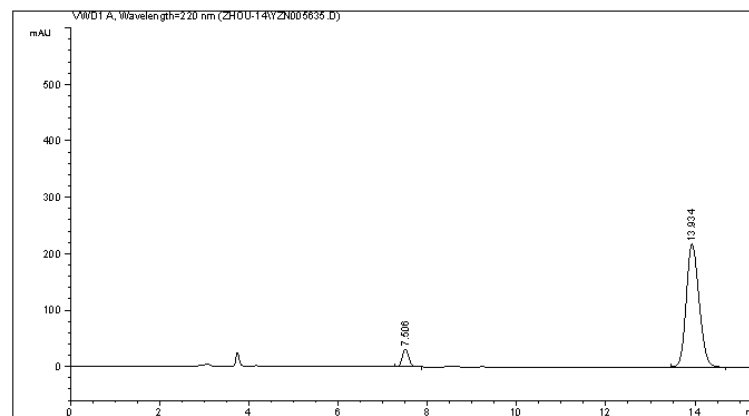
=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-14\YZN005635.D
 Sample Name: MC-9-97B

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=====
Acq. Operator   : Z
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 7/22/2014 6:08:19 PM
Acq. Method    : C:\CHEM32\1\METHODS\DEF.LC.M
Last changed   : 7/22/2014 6:04:17 PM by Z
                (modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF.LC.M
Last changed   : 7/22/2014 6:25:12 PM by Z
                (modified after loading)
Sample Info    : AD-H, H/i-PrOH = 90/10, 1.0mL/min, 30 oC, 220 nm
=====
  
```



=====
 Area Percent Report
 =====

```

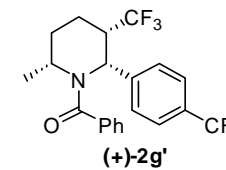
Sorted By      : Signal
Multiplier:    : 1.0000
Dilution:      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	7.506	BB	0.1596	315.96140	30.64944	6.8026
2	13.934	BB	0.3098	4328.75244	217.05875	93.1974

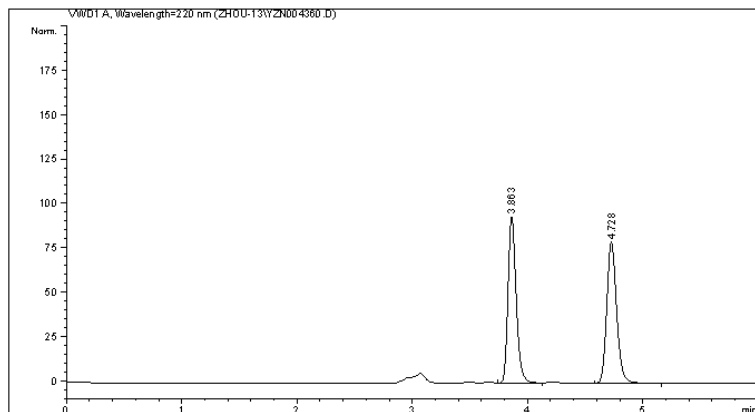
Totals : 4644.71384 247.70819

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004360.D
 Sample Name: MC-9-98A

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 1/3/2014 4:41:03 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 1/3/2014 4:36:18 PM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 8/19/2014 10:20:46 AM by Z
 (modified after loading)
 Sample Info : AD-H, H/1-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220nm



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 Area Percent Report
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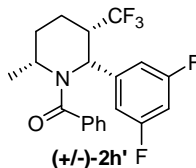
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.863	VV	0.0768	466.23618	94.05981	49.5955
2	4.728	VB	0.0913	473.84210	79.62459	50.4045

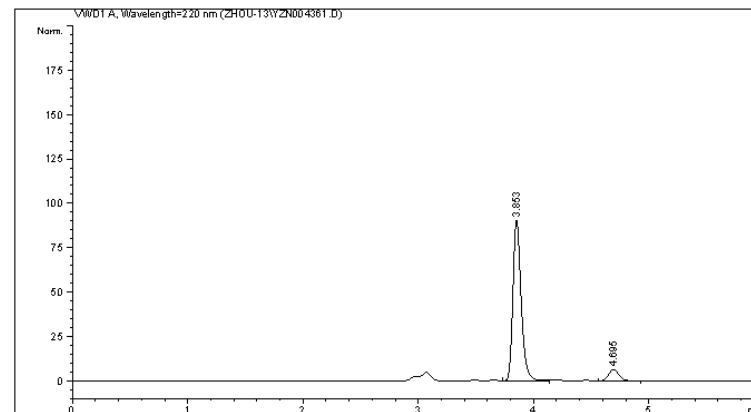
Totals : 940.07828 173.68440

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004361.D
 Sample Name: MC-9-98A(SHOU)

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 1/3/2014 4:50:17 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 1/3/2014 4:47:46 PM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 8/19/2014 10:20:46 AM by Z
 (modified after loading)
 Sample Info : AD-H, H/1-PrOH/ = 90/10, 1.0 mL/min, 30 oC, 220nm



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 Area Percent Report
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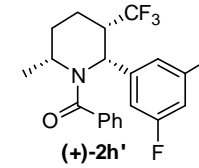
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.853	VB	0.0773	453.13580	90.61848	91.8844
2	4.695	VB	0.0932	40.02260	6.54235	8.1156

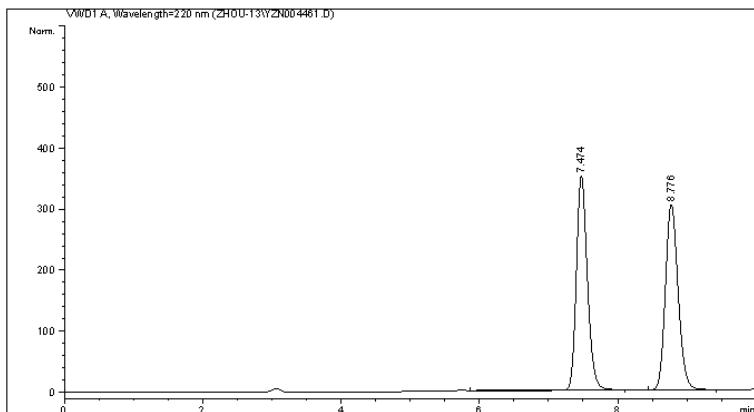
Totals : 493.15840 97.16082

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004461.D
 Sample Name: NC-10-12B+

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 1/20/2014 5:00:26 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 1/20/2014 4:45:22 PM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 8/19/2014 10:28:34 AM by Z
 (modified after loading)
 Sample Info : AD-H H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 220 nm



=====
 Area Percent Report
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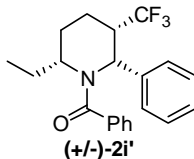
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height %s	Area [mAU]	Area %
1	7.474	VB	0.1727	3967.80762	350.98093	50.6577	
2	8.776	BB	0.1966	3864.78101	303.54080	49.3423	

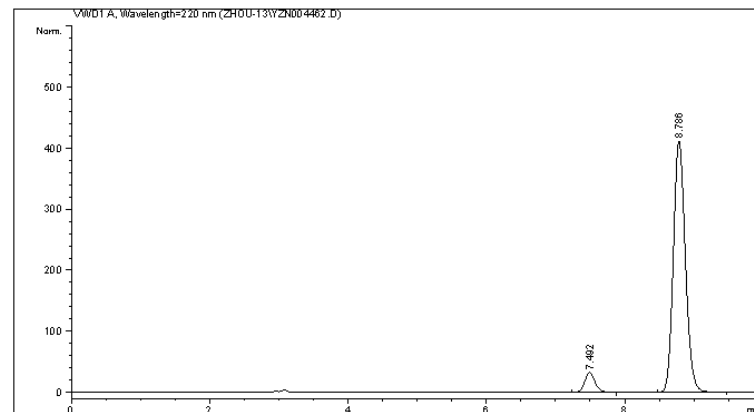
Totals : 7832.58862 654.52173

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004462.D
 Sample Name: NC-10-12A

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 1/20/2014 5:15:26 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 1/20/2014 5:13:37 PM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 8/19/2014 10:29:35 AM by Z
 (modified after loading)
 Sample Info : AD-H H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 220 nm



=====
 Area Percent Report
 =====

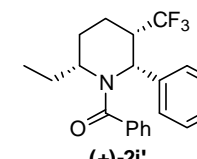
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height %s	Area [mAU]	Area %
1	7.492	BB	0.1584	332.55539	32.18368	6.2797	
2	8.786	BB	0.1875	4963.17773	410.99475	93.7203	

Totals : 5295.73312 443.17843

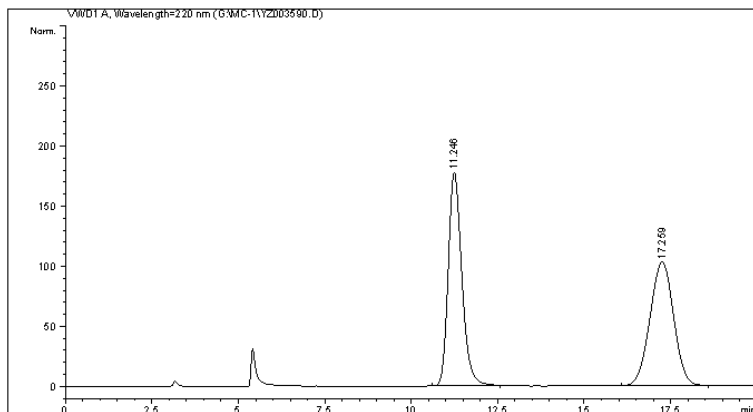
=====
 *** End of Report ***



Data File G:\MC-1\YZ003590.D
 Sample Name: ZY-6-66D (A)

```

=====
Acq. Operator   : ZX
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 12/20/2012 12:13:32 PM
Acq. Method    : C:\HPCHEM\1\METHODS\SW.M
Last changed   : 12/20/2012 11:47:21 AM by ZX
                (modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF.LC.M
Last changed   : 8/19/2014 11:00:53 AM by Z
                (modified after loading)
Sample Info    : 0J-H, H/1-PrOH = 90/10, 1.0 mL/min, 30 oC, 220 nm
=====
  
```



Area Percent Report

```

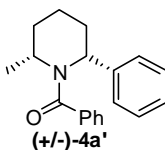
=====
Sorted By      : Signal
Multiplier:    : 1.0000
Dilution:      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.246	BB	0.4182	4860.72607	177.65092	49.7067
2	17.259	BB	0.7466	4918.08936	102.95752	50.2933

Totals : 9778.81543 280.60844

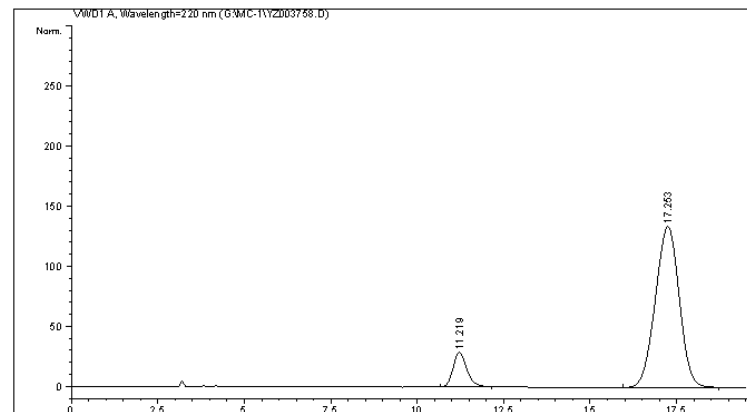
*** End of Report ***



Data File G:\MC-1\YZ003758.D
 Sample Name: ZY-6-78A

```

=====
Acq. Operator   : WH
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 1/17/2013 1:49:30 AM
Acq. Method    : C:\HPCHEM\1\METHODS\SW.M
Last changed   : 1/17/2013 1:45:57 AM by WH
                (modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF.LC.M
Last changed   : 8/19/2014 11:00:53 AM by Z
                (modified after loading)
Sample Info    : 0J-H, H/1-PrOH = 90/10, 1.0 mL/min, 30 oC, 220 nm
=====
  
```



Area Percent Report

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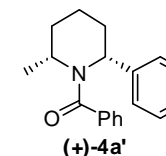
=====
Sorted By      : Signal
Multiplier:    : 1.0000
Dilution:      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.219	BB	0.4229	784.72314	28.52110	10.8397
2	17.253	BB	0.7467	6454.58789	134.05344	89.1603

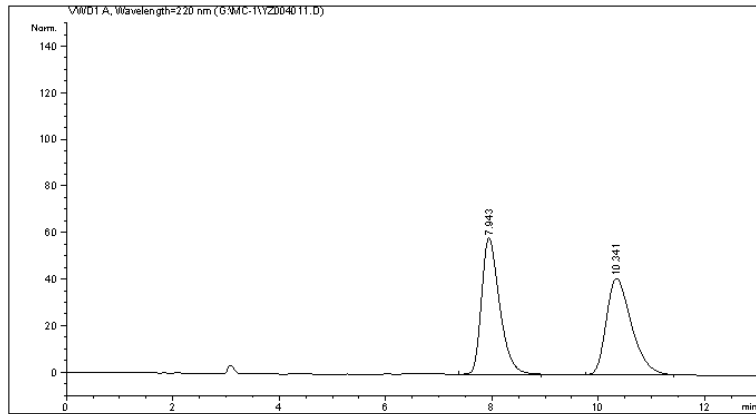
Totals : 7239.31104 162.57453

*** End of Report ***



Data File G:\MC-1\YZ004011.D
 Sample Name: ZY-6-94B(+/-)

=====
 Acq. Operator : WH
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 3/14/2013 8:11:53 AM
 Acq. Method : C:\HPCHEM\1\METHODS\SW.M
 Last changed : 3/14/2013 8:10:18 AM by WH
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 8/19/2014 11:02:36 AM by Z
 (modified after loading)
 Sample Info : 0G, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
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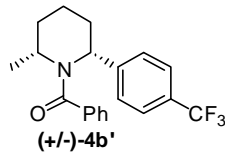
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]	Area %
1	7.943	BB	0.3595	1379.01208	50.4349	58.48378	50.4349
2	10.341	BB	0.5002	1355.22925	49.5651	41.35931	49.5651

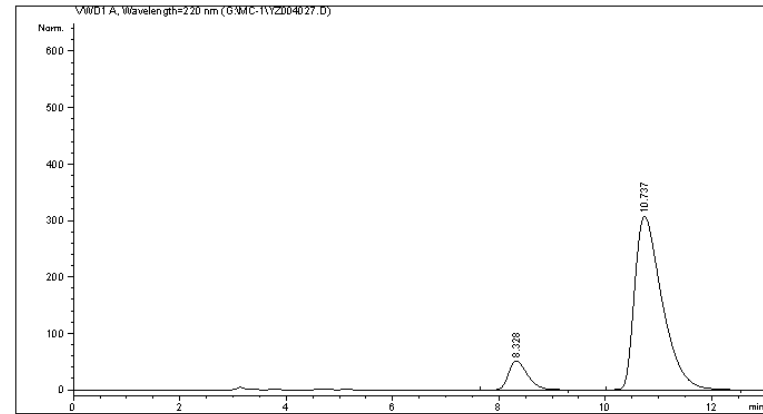
Totals : 2734.24133 99.84309

=====
 *** End of Report ***



Data File G:\MC-1\YZ004027.D
 Sample Name: ZY-6-95B

=====
 Acq. Operator : WH
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 3/16/2013 3:13:49 AM
 Acq. Method : C:\HPCHEM\1\METHODS\SW.M
 Last changed : 3/16/2013 3:05:14 AM by WH
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 8/19/2014 11:03:05 AM by Z
 (modified after loading)
 Sample Info : 0G, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
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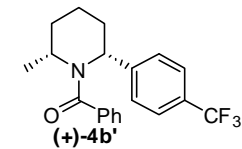
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]	Area %
1	8.328	VB	0.3797	1302.94641	51.99115	51.99115	10.5523
2	10.737	BB	0.5496	1.10446e4	307.82346	307.82346	89.4477

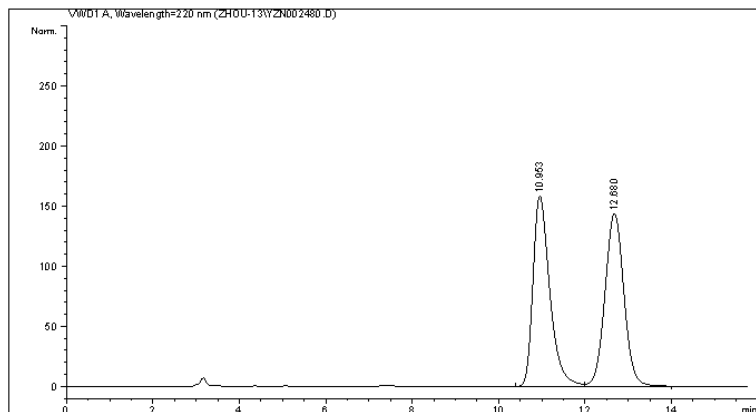
Totals : 1.23475e4 359.81461

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN002480.D
 Sample Name: ZY-6-94D(±)

=====
 Acq. Operator : WH
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 3/13/2013 11:06:04 AM
 Acq. Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 3/13/2013 10:57:01 AM by WH
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 8/19/2014 11:06:51 AM by Z
 (modified after loading)
 Sample Info : 0J-H, H/i-PrOH = 95/5, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
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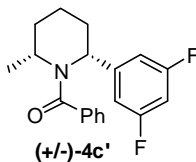
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.953	BB	0.4117	4290.16162	158.61508	49.5040
2	12.680	VB	0.4711	4376.13330	143.96098	50.4960

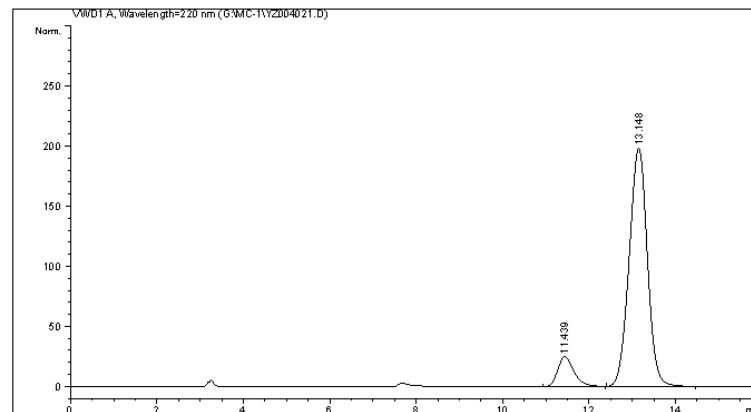
Totals : 8666.29492 302.57607

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 *** End of Report ***



Data File G:\MC-1\YZ004021.D
 Sample Name: ZY-6-95D

=====
 Acq. Operator : WH
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 3/16/2013 1:49:45 AM
 Acq. Method : C:\HPCHEM\1\METHODS\SW.M
 Last changed : 3/16/2013 1:30:34 AM by WH
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 8/19/2014 11:06:51 AM by Z
 (modified after loading)
 Sample Info : 0J-H, H/i-PrOH = 95/5, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

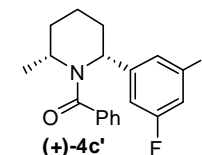
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.439	BB	0.4097	670.86322	24.95699	10.3279
2	13.148	BB	0.4539	5824.77051	198.83044	89.6721

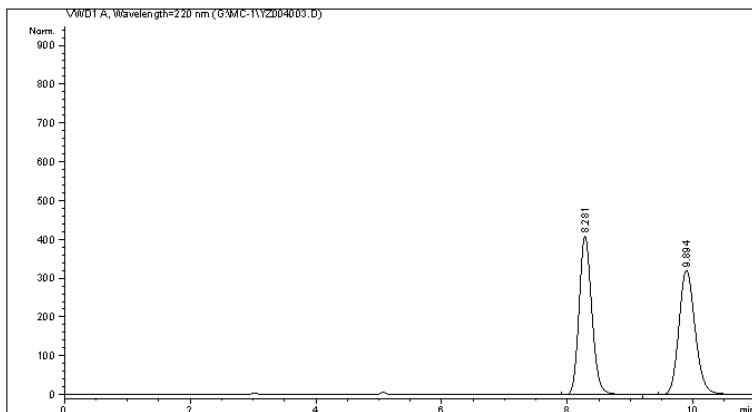
Totals : 6495.63373 223.78744

=====
 *** End of Report ***



Data File G:\MC-1\YZ004003.D
 Sample Name: ZY-6-94C(+/-)

=====
 Acq. Operator : WH
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 3/14/2013 3:08:36 AM
 Acq. Method : C:\HPCHEM\1\METHODS\SW.M
 Last changed : 3/14/2013 3:06:37 AM by WH
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 8/19/2014 11:05:37 AM by Z
 (modified after loading)
 Sample Info : OD-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
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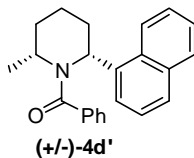
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.281	BB	0.2186	5766.77051	408.44028	50.0429
2	9.894	BB	0.2764	5756.89063	320.75250	49.9571

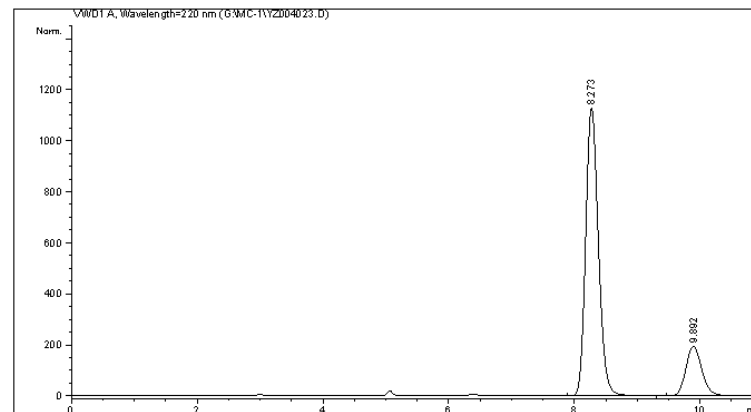
Totals : 1.15237e4 729.19278

=====
 *** End of Report ***



Data File G:\MC-1\YZ004023.D
 Sample Name: ZY-6-95C

=====
 Acq. Operator : WH
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 3/16/2013 2:25:33 AM
 Acq. Method : C:\HPCHEM\1\METHODS\SW.M
 Last changed : 3/16/2013 2:09:09 AM by WH
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF LC.M
 Last changed : 8/19/2014 11:04:48 AM by Z
 (modified after loading)
 Sample Info : OD-H, H/i-PrOH = 95/5, 1.0 mL/min, 30 oC, 220 nm



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 Area Percent Report
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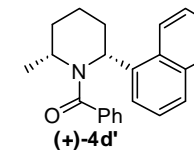
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.273	VB	0.2180	1.58589e4	1127.39514	82.0934
2	9.892	BB	0.2761	3459.23022	194.30327	17.9066

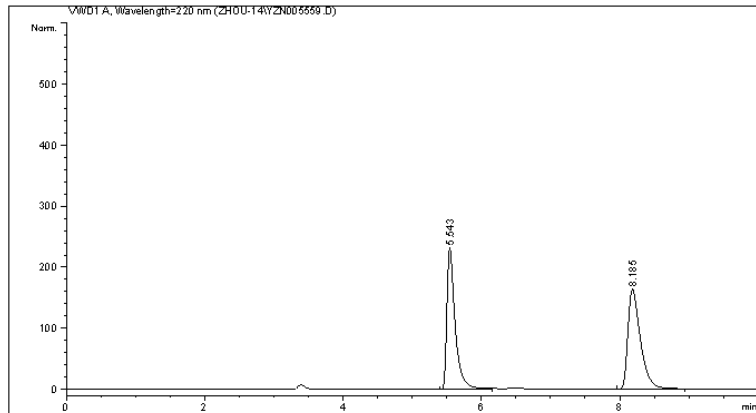
Totals : 1.93182e4 1321.69841

=====
 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-14\YZN005559.D
 Sample Name: MC-10-4A+

=====
 Acq. Operator : Z
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 7/17/2014 1:39:15 PM
 Acq. Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 7/17/2014 1:29:16 PM by Z
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 8/19/2014 10:29:35 AM by Z
 (modified after loading)
 Sample Info : OD-H, H/i-PrOH = 95/5, 0.9 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

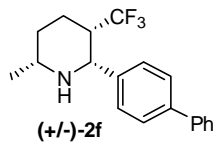
Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.543	VB	0.1213	1909.90576	230.73236	48.3532
2	8.185	BB	0.1885	2039.99951	162.72169	51.6468

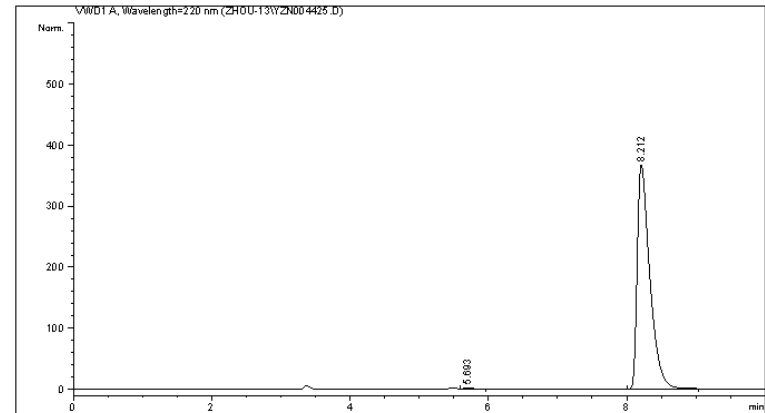
Totals : 3949.90527 393.45406

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 *** End of Report ***



Data File C:\CHEM32\1\DATA\ZHOU-13\YZN004425.D
 Sample Name: MC-104B2

=====
 Acq. Operator : B
 Acq. Instrument : Instrument 1 Location : Vial 1
 Injection Date : 1/13/2014 9:50:42 AM
 Acq. Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 1/13/2014 9:48:38 AM by B
 (modified after loading)
 Analysis Method : C:\CHEM32\1\METHODS\DEF.LC.M
 Last changed : 8/19/2014 10:29:35 AM by Z
 (modified after loading)
 Sample Info : OD-H, H/i-PrOH/ = 95/5, 0.9 mL/min, 30 oC, 220 nm



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 Area Percent Report
 =====

Sorted By : Signal
 Multiplier: : 1.0000
 Dilution: : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.693	VB	0.1367	20.57336	2.26167	0.4422
2	8.212	BB	0.1894	4632.18896	367.08691	99.5578

Totals : 4652.76232 369.34858

=====
 *** End of Report ***

