Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

$Zn(OTf)_2$ Catalyzed Ugi-type Reaction of 3-(2-Isocyanoethyl)indoles with Indole-Derived Ketimines: Rapid Access to Polycyclic Spiroindolines

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1) General Information

Reactions were monitored by thin layer chromatography using UV light to visualize the course of reaction. Purification of reaction products was carried out by flash chromatography on silica gel. Chemical yields refer to pure isolated substances. ^{1}H, ^{19}F and ^{13}C NMR spectra were obtained using a Bruker DPX-300 or DPX-500 spectrometer. Chemical shifts are reported in ppm from $CDCl_3$ with the solvent resonance as the internal standard. The following abbreviations were used to designate chemical shift multiplicities: s=singlet,d=doublet, t = triplet, q=quartet, h=heptet, m=multiplet, br=broad.

Anhydrous solvents such as CH_2Cl_2, $ClCH_2CH_2Cl$, CH_3CN, THF, toluene, EtOAc, acetone, EtOH as well as metal salts were purchased from Energy Chemical. Unless otherwise stated, all purchased reagents were used without further purification. All reactions involving air- or moisture-sensitive compounds were carried out under nitrogen atmosphere in dried Schlenk tube. The 3-(2-isocyanoethyl)indoles[1], 2-arylindoles[2], 2-aryl-3,3-difluoro-3*H*-indoles[3] and 2-aryl-3*H*-indol-3-ones[4] were prepared using the literature procedures.

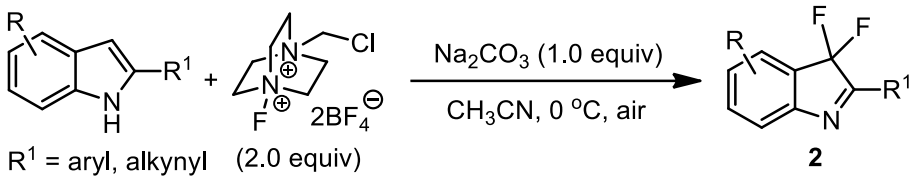
[1] (a) Zhao, X.; Liu, X.; Mei, H.; Guo, J.; Lin, L.; Feng, X. *Angew. Chem. Int. Ed.* **2015**, *54*, 4032-4035. (b) Saya, J. M.; Oppelaar, B.; Cioc, R. C.; van der Heijden, G.; Vande Velde, C. M. L.; Orru, R. V. A.; Ruijter, E. *Chem. Commun.* **2016**, *52*, 12482-12485.

[2] Yang, S.-D.; Sun, C.-L.; Fang, Z.; Li, B.-J.; Li, Y.-Z.; Shi, Z.-J. *Angew. Chem. Int. Ed.* **2008**, *47*, 1473-1476.

[3] (a) Lin,R.; Ding, S.; Shi, Z.; Jiao, N. *Org. Lett.* **2011**, *13*, 4498-4501. (b) Preciado, S.; Vicente-García, E.; Llabrés, S.; Luque, F. J.; Lavilla, R. *Angew. Chem. Int. Ed.* **2012**, *51*, 6874-6877.

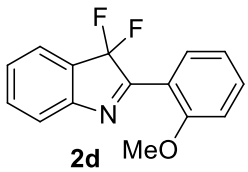
[4] Yamashita, M.; Nishizono, Y.; Himekawa, S.; Iida, A. *Tetrahedron* **2016**, *72*, 4123-4131.

2. General Procedure and Spectral Data of 3,3-difluoro-3*H*-indoles 2[3]



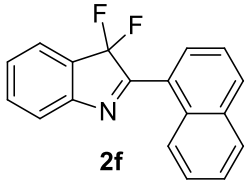
R[1] = aryl, alkynyl

To a solution of indole (1 mmol) in CH_3CN (15 mL) were added Na_2CO_3 (1 g) and Selectfluor (2.2 mmol) at 0 °C. The mixture was stirred at this temperature for hours till the full consumption of indole. When the reaction was complete, CH_2Cl_2 (100 mL) was added and the mixture washed with H_2O (3×20mL) and brine (3×15mL). The organic extracts were dried over Na_2SO_4, and the solvent was concentrated in vacuo. The resulting residue was directly subjected to column chromatography using petroleum ether/EtOAc (from 10:1-3:1) as the eluent to afford the desired product **2**. **The spectral data of some new compounds are provided below**.



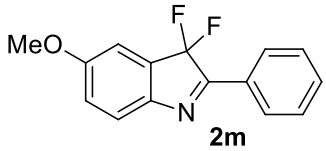
2d

Compound **2d** was obtained in 86% yield as a yellow solid (m.p. 45-48 °C). ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.95 (s, 3H), 7.04-7.10 (m, 2H), 7.27 (t, *J* = 5.0 Hz, 1H), 7.46-7.51 (m, 2H), 7.53 (d, *J* = 5.0 Hz, 1H), 7.59 (d, *J* = 5.0 Hz, 1H), 8.08 (d, *J* = 5.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.99 (t, *J* = 24 Hz), 159.66, 152.67 (t, *J* = 10 Hz), 133.30, 132.94, 131.05 (t, *J* = 3.8 Hz), 128.42 (t, *J* = 25 Hz), 127.31, 123.34 (t, *J* = 256 Hz), 122.60, 121.99, 120.47, 118.45 (t, *J* =2.5 Hz), 112.11, 55.98; ^{19}F NMR (471 MHz, $CDCl_3$): δ -116.80 (m, 2F); IR: (KBr) ν_{max} 2940, 1599, 1579, 1461, 1271, 1078, 757; HRMS (EI): Exact mass calcd for $C_{15}H_{12}F_2NO$ $[M]^+$: 260.0881, Found: 260.0879.



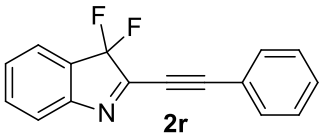
2f

Compound **2f** was obtained in 71% yield as a yellow solid (m.p. 85-87 °C). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35 (t, *J* = 10 Hz, 1H), 7.55-7.64 (m, 4H), 7.69-7.74 (m, 2H), 7.94 (d, *J* = 10 Hz, 1H), 8.05 (d, *J* = 10 Hz, 1H), 8.39 (d, *J* = 10 Hz, 1H), 9.65 (d, *J* = 10 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.46 (t, *J* = 23 Hz), 152.78 (t, *J* = 10 Hz), 134.21, 133.30, 133.23, 131.80, 130.53 (t, *J* = 3.8 Hz), 128.86, 128.25, 128.12 (t, *J* = 25 Hz), 127.59, 126.99, 126.45, 125.09 (t, *J* = 2.5 Hz), 124.76, 124.04 (t, *J* =256 Hz), 122.90, 122.19; ^{19}F NMR (471 MHz, $CDCl_3$): δ -112.72 (s, 2F); IR: (KBr) ν_{max} 2925, 1619, 1541, 1509, 1460, 1252, 1061, 765; HRMS (EI): Exact mass calcd for $C_{18}H_{12}F_2N$ $[M]^+$: 280.0932, Found: 280.0939.



2m

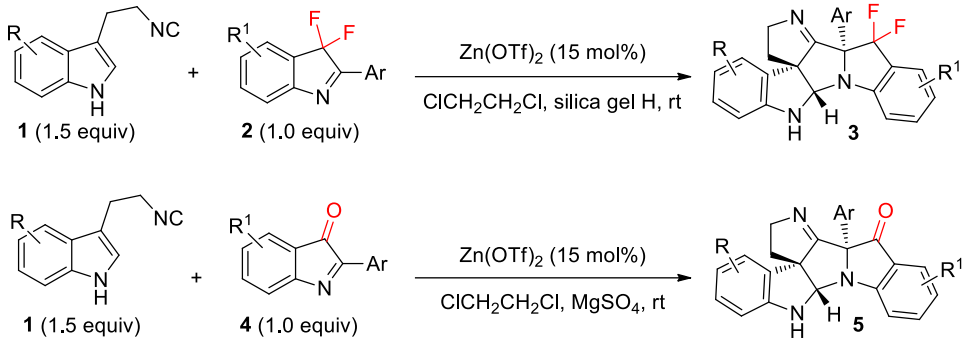
Compound **2m** was obtained in 77% yield as a yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.86 (s, 3H), 6.98 (dd, *J* = 10, 5.0 Hz, 1H), 7.15 (d, *J* = 5.0 Hz, 1H), 7.46 (d, *J* = 5.0 Hz, 1H), 7.49-7.56 (m, 3H), 8.17 (d, *J* = 5.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 167.52 (t, *J* = 24 Hz), 159.51, 145.60 (t, *J* = 8.8 Hz), 132.00, 130.41 (t, *J* = 24 Hz), 129.15 (t, *J* = 2.5 Hz), 128.83, 128.10, 122.92 (t, *J* = 256 Hz), 122.60, 116.98, 110.20, 55.87; ^{19}F NMR (471 MHz, $CDCl_3$): δ -116.02 (s, 2F); IR: (KBr) ν_{max} 2967, 1606, 1551, 1478, 1364, 1265, 1066, 696; HRMS (EI): Exact mass calcd for $C_{15}H_{12}F_2NO$ $[M]^+$: 260.0881, Found: 260.0875.



2r

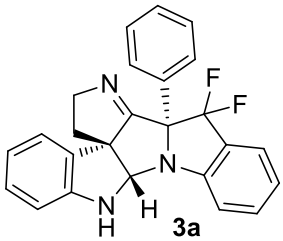
Compound **2r** was obtained in 68% yield as a yellow solid (m.p. 55-56 ^{o}C). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32-7.36 (m, 1H), 7.40-7.43 (m, 2H), 7.45-7.49 (m, 1H), 7.50-7.54 (m, 3H), 7.66-7.68 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.34 (t, *J* = 28 Hz), 152.66 (t, *J* = 8.8 Hz), 133.39, 132.81, 130.68, 128.60, 128.46, 127.35 (t, *J* = 24 Hz), 123.40, 122.55, 120.50, 120.46 (t, *J* = 255 Hz), 103.34, 80.76 (t, *J* = 2.5 Hz); ^{19}F NMR (471 MHz, $CDCl_3$): δ -122.40 (s, 2F); IR: (KBr) ν_{max} 2923, 2193, 1548, 1458, 1260, 1081, 1063, 756; HRMS (EI): Exact mass calcd for $C_{16}H_{10}F_2N$ $[M]^+$: 254.0776, Found: 254.0775.

3. General Procedure and Spectral Data of Products 3 and 5

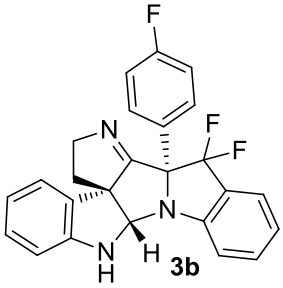


General procedure for the synthesis of products 3: To a 10.0 mL Schlenk tube were successively added $Zn(OTf)_2$ (8.1 mg, 0.0225 mmol), 3-(2-isocyanoethyl)indoles **1** (0.225 mmol), 2-aryl-3,3-difluoro-3H-indoles **2** (0.15 mmol), silica gel H (30 mg) and anhydrous $ClCH_2CH_2Cl$ (1.0 mL). The reaction mixture was stirred vigorously at room temperature till almost full consumption of 2-aryl-3,3-difluoro-3H-indoles **2** as monitored by TLC (36-72 h). Then the reaction mixture was directly subjected to column chromatography using CH_2Cl_2/EtOAc (from 10:1-5:1) as the eluent to afford the desired product **3**.

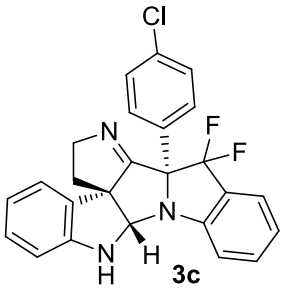
General procedure for the synthesis of products 5: To a 10.0 mL Schlenk tube were successively added $Zn(OTf)_2$ (8.1 mg, 0.0225 mmol), 3-(2-isocyanoethyl)indoles **1** (0.225 mmol), 2-aryl-3H-indol-3-ones **4** (0.15 mmol), anhydrous $MgSO_4$ (30 mg) and anhydrous $ClCH_2CH_2Cl$ (1.0 mL). The reaction mixture was stirred vigorously at room temperature till almost full consumption of 2-aryl-3H-indol-3-ones **4** as monitored by TLC (36-48 h). Then the reaction mixture was directly subjected to column chromatography using CH_2Cl_2/EtOAc (from 10:1-5:1) as the eluent to afford the desired product **5**.



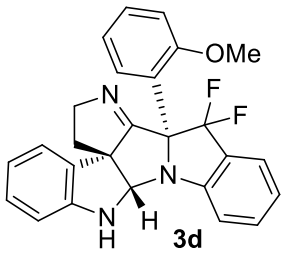
The reaction was run at rt for 36 h, affording product **3a** in 67% yield as a pale yellow solid (m.p. 153-158 °C). ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.21-2.31 (m, 2H), 4.41-4.52 (m, 2H), 4.61 (brs, 1H), 4.96 (s, 1H), 6.36-6.41 (m, 2H), 6.63 (d, *J* = 10 Hz, 1H), 6.79 (t, *J* = 10 Hz, 1H), 7.01-7.07 (m, 5H), 7.41-7.44 (m, 2H), 7.46-7.50 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.77 (d, *J* = 3.8 Hz), 151.30 (dd, *J* = 7.6, 5.8 Hz), 148.18, 134.05 (d, *J* = 6.3 Hz), 133.20, 130.15, 128.59, 127.88, 127.51, 126.32, 124.68, 123.62 (dd, *J* = 259, 243 Hz), 122.66, 122.24 (dd, *J* = 26, 25 Hz), 122.10, 119.49, 112.12, 109.24, 84.69, 74.46 (dd, *J* = 30, 20 Hz), 71.31, 66.90, 39.15; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.35 (d, *J* = 254 Hz, 1F), -96.26 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3422, 2930, 1665, 1608, 1469, 1258, 1089, 745; HRMS (EI): Exact mass calcd for $C_{25}H_{19}F_2N_3$ $[M]^+$: 399.1547, Found: 399.1550.



The reaction was run at rt for 36 h, affording product **3b** in 61% yield as a yellow solid (m.p. 136-138 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.20-2.31 (m, 2H), 4.40-4.52 (m, 2H), 4.61 (brs, 1H), 4.96 (s, 1H), 6.41-6.44 (m, 2H), 6.63 (d, *J* = 10 Hz, 1H), 6.68-6.72 (m, 2H), 6.83 (t, *J* = 10 Hz, 1H), 7.02-7.08 (m, 2H), 7.38-7.41 (m, 2H), 7.46-7.50 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.68 (d, *J* = 2.5 Hz), 163.27, 161.31, 151.15 (dd, *J* = 7.5, 6.3 Hz), 148.04, 133.29, 130.06, 129.89 (dd, *J* = 5.0, 2.5 Hz), 128.76, 128.26 (d, *J* = 8.8 Hz), 124.71, 123.52 (dd, *J* = 258, 241 Hz), 122.60, 122.27, 122.11 (dd, *J* = 27, 26 Hz), 119.66, 114.45 (d, *J* = 23 Hz), 112.16, 109.27, 84.68, 76.89 (dd, *J* = 30, 20 Hz), 71.29, 66.96, 39.08; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.17 (d, *J* = 254 Hz, 1F), -96.69 (d, *J* = 254 Hz, 1F), -114.52 (s, 1F); IR: (KBr) ν_{max} 3420, 2949, 1665, 1608, 1470, 1233, 1091, 823; HRMS (EI): Exact mass calcd for $C_{25}H_{18}F_3N_3$ $[M]^+$: 417.1453, Found: 417.1457.

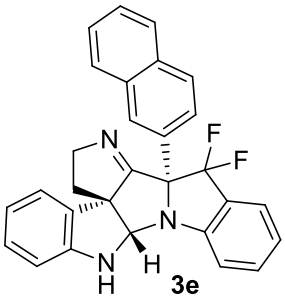


The reaction was run at rt for 36 h, affording product **3c** in 52% yield as a yellow solid (m.p. 196-198 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.21-2.31 (m, 2H), 4.41-4.52 (m, 2H), 4.62 (brs, 1H), 4.95 (s, 1H), 6.42-6.45 (m, 2H), 6.62 (d, *J* = 5 Hz, 1H), 6.86 (t, *J* = 5 Hz, 1H), 6.97-6.99 (m, 2H), 7.03 (d, *J* = 5 Hz, 1H), 7.06 (t, *J* = 5 Hz, 1H), 7.34-7.36 (m, 2H), 7.46-7.51 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.54 (d, *J* = 2.5 Hz), 151.14 (dd, *J* = 7.5, 6.3 Hz), 148.03, 133.72, 133.34, 132.83 (d, *J* = 6.3 Hz), 129.89, 128.79, 127.81, 127.70, 124.71, 123.53 (dd, *J* = 259, 243 Hz), 122.63, 122.33, 122.08 (dd, *J* = 27, 25 Hz), 119.75, 112.20, 109.35, 84.69, 76.93 (dd, *J* = 30, 20 Hz), 71.33, 66.98, 38.95; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.29 (d, *J* = 254 Hz, 1F), -96.01 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3433, 2989, 1621, 1588, 1455, 1188, 1012, 802; HRMS (EI): Exact mass calcd for $C_{25}H_{18}ClF_2N_3$ $[M]^+$: 433.1157, Found: 433.1152.



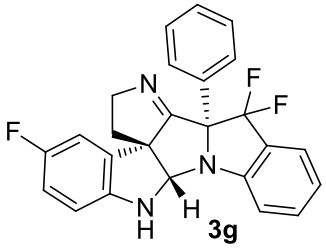
The reaction was run at rt for 36 h, affording product **3d** in 69% yield as a pale yellow solid (m.p. 115-117 ^{o}C). ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.19-2.31 (m, 2H), 3.77 (s, 3H), 4.33-4.45 (m, 2H), 4.62 (s, 1H), 4.93 (s, 1H), 6.36 (d, *J* = 10 Hz, 1H), 6.40 (t, *J* = 10 Hz, 1H), 6.53 (td, *J* = 10, 5.0 Hz, 1H), 6.58 (d, *J* = 10 Hz, 1H), 6.68 (d, *J* = 10 Hz, 1H), 6.78 (td, *J* = 10, 5.0 Hz, 1H), 6.96-7.02 (m, 3H), 7.43-7.47 (m, 2H), 7.55 (d, *J* = 5.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 177.23, 157.82, 150.58 (t,

J = 6.3 Hz), 148.58, 132.92, 130.61, 129.52, 128.59, 128.28, 124.72, 123.62 (dd, *J* = 260, 240 Hz), 122.51 (dd, *J* = 30, 28 Hz), 121.94, 121.63, 119.61, 119.11, 111.42, 110.82, 108.94, 84.45, 76.36 (dd, *J* = 31, 21 Hz), 70.59, 66.02, 55.52, 39.51; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.78 (d, *J* = 254 Hz, 1F), -97.37 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3421, 2931, 1661, 1607, 1483, 1256, 1086, 746; HRMS (EI): Exact mass calcd for $C_{26}H_{22}F_2N_3O$ $[M]^+$: 430.1725, Found: 430.1734.



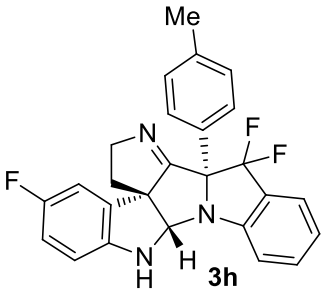
3e

The reaction was run at rt for 36 h, affording product **3e** in 70% yield as a yellow solid (m.p. 155-160 °C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.23-2.32 (m, 2H), 4.44-4.56 (m, 2H), 4.67 (brs, 1H), 5.00 (s, 1H), 6.10 (t, *J* = 5 Hz, 1H), 6.31 (d, *J* = 5 Hz, 1H), 6.47 (t, *J* = 5 Hz, 1H), 6.56 (d, *J* = 5 Hz, 1H), 7.05-7.09 (m, 2H), 7.32-7.38 (m, 2H), 7.48-7.59 (m, 5H), 7.64-7.65 (m, 1H), 7.82 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.80 (d, *J* = 3.8 Hz), 151.34 (dd, *J* = 7.5, 6.3 Hz), 148.10, 133.27, 132.80, 132.47, 131.52 (d, *J* = 5 Hz), 130.13, 128.40, 127.83, 127.31, 127.26, 126.06, 125.83, 125.57, 124.74, 123.92, 123.77 (dd, *J* = 259, 243 Hz), 122.34, 122.29 (dd, *J* = 27, 25 Hz), 122.18, 119.34, 112.19, 109.04, 84.77, 77.63 (dd, *J* = 30, 20 Hz), 71.37, 66.96, 38.97; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.02 (d, *J* = 254 Hz, 1F), -95.89 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3411, 2901, 1644, 1608, 1485, 1007, 802; HRMS (EI): Exact mass calcd for $C_{29}H_{21}F_2N_3$ $[M]^+$: 449.1704, Found: 449.1707.



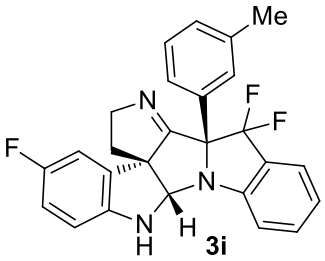
3g

The reaction was run at rt for 36 h, affording product **3g** in 75% yield as a pale yellow solid (m.p. 198-200 °C). ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.21-2.29 (m, 2H), 4.43-4.46 (m, 2H), 4.50 (brs, 1H), 4.96 (s, 1H), 6.31-6.34 (m, 2H), 6.50 (td, *J* = 10, 5 Hz, 1H), 7.02 (d, *J* = 10 Hz, 1H), 7.05-7.08 (m, 4H), 7.42-7.44 (m, 2H), 7.46-7.50 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.10 (d, *J* = 2.5 Hz), 157.93, 156.05, 151.16 (dd, *J* = 7.5, 5 Hz), 144.38 (d, *J* = 1.3 Hz), 133.90 (d, *J* = 6.3 Hz), 133.26, 131.49 (d, *J* = 7.5 Hz), 128.12, 127.66, 126.35, 124.71, 123.53 (d, *J* = 258, 243 Hz), 122.24, 122.19 (dd, *J* = 28, 25 Hz), 114.95 (d, *J* = 24 Hz), 112.09, 109.86 (d, *J* = 6.3 Hz), 109.73 (d, *J* = 8.8 Hz), 85.39, 77.41 (dd, *J* = 30, 20 Hz), 71.54, 66.88, 38.78; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.47 (d, *J* = 254 Hz, 1F), -96.05 (d, *J* = 254 Hz, 1F), -125.11 (s, 1F); IR: (KBr) ν_{max} 3223, 2930, 1668, 1611, 1486, 1183, 1068, 747; HRMS (EI): Exact mass calcd for $C_{25}H_{18}F_3N_3$ $[M]^+$: 417.1453, Found: 417.1449.



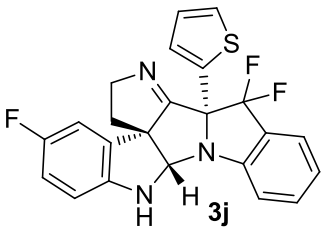
3h

The reaction was run at rt for 36 h, affording product **3h** in 89% yield as a yellow solid (m.p. 200-202 °C). ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.18 (s, 3H), 2.24-2.27 (m, 2H), 4.41-4.46 (m, 2H), 4.48 (brs, 1H), 4.95 (s, 1H), 6.29-6.33 (m, 2H), 6.51 (td, *J* = 9.0, 3.0 Hz, 1H), 6.84-6.87 (m, 2H), 7.00-7.08 (m, 2H), 7.27-7.30 (m, 2H), 7.45-7.50 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.22 (d, *J* = 3.8 Hz), 157.98, 156.09, 151.20 (dd, *J* = 7.5, 6.3 Hz), 144.41, 137.77, 133.18, 131.62 (d, *J* = 7.5 Hz), 130.73 (d, *J* = 5 Hz), 128.33, 126.32, 124.69, 123.50 (dd, *J* = 259, 243 Hz), 122.30 (dd, *J* = 26, 25 Hz), 122.15, 114.66 (d, *J* = 24 Hz), 112.07, 109.85 (d, *J* = 10 Hz), 109.72 (d, *J* = 6.3 Hz), 85.37, 77.33 (dd, *J* = 31, 21 Hz), 71.51, 66.83, 38.80, 20.90; ^{19}F NMR (282 MHz, $CDCl_3$): δ -82.60 (d, *J* = 254 Hz, 1F), -96.69 (d, *J* = 254 Hz, 1F), -125.29 (s, 1F); IR: (KBr) ν_{max} 3298, 2942, 1632, 1596, 1475, 1142, 1045, 842; HRMS (EI): Exact mass calcd for $C_{26}H_{20}F_3N_3$ $[M]^+$: 431.1609, Found: 431.1614.



3i

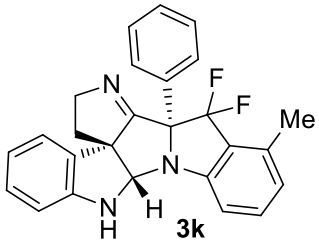
The reaction was run at rt for 36 h, affording product **3i** in 79% yield as a yellow solid (m.p. 207-210 °C). ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.15 (s, 3H), 2.20-2.30 (m, 2H), 4.43-4.46 (m, 2H), 4.49 (brs, 1H), 4.95 (s, 1H), 6.30-6.34 (m, 2H), 6.50 (td, *J* = 10, 5.0 Hz, 1H), 6.86 (d, *J* = 10 Hz, 1H), 6.97 (t, *J* = 10 Hz, 1H), 7.02 (d, *J* = 5 Hz, 1H), 7.06 (t, *J* = 10 Hz, 1H), 7.17 (s, 1H), 7.24-7.26 (m, 1H), 7.47-7.50 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.16 (d, *J* = 3.8 Hz), 157.96, 156.08, 151.19 (dd, *J* = 7.5, 6 Hz), 144.53, 133.70 (d, *J* = 6.3 Hz), 133.23, 131.67 (d, *J* = 7.5 Hz), 128.79, 127.62, 127.02, 124.72, 123.54, 123.40 (dd, *J* = 260, 243 Hz), 122.18, 122.16 (dd, *J* = 26, 25 Hz), 114.88 (d, *J* = 24 Hz), 112.06, 109.76 (d, *J* = 24 Hz), 109.53 (d, *J* = 8.8 Hz), 85.46, 77.50 (dd, *J* = 30, 20 Hz), 71.47, 66.86, 38.68, 21.14; ^{19}F NMR (471 MHz, $CDCl_3$): δ -81.75 (d, *J* = 254 Hz, 1F), -96.80 (d, *J* = 254 Hz, 1F), -125.28 (s, 1F); IR: (KBr) ν_{max} 3402, 2914, 1654, 1605, 1563, 1461, 1081, 762; HRMS (EI): Exact mass calcd for $C_{26}H_{20}F_3N_3$ $[M]^+$: 431.1609, Found: 431.1604.



3j

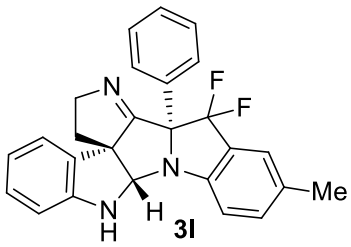
The reaction was run at rt for 72 h, affording product **3j** in 30% yield as a yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.18-2.35 (m, 2H), 4.44-4.49 (m, 2H), 4.55 (brs, 1H), 4.94 (s, 1H), 6.37-6.41 (m, 1H), 6.47 (dd, *J* = 9, 3 Hz, 1H), 6.60 (td, *J* = 9, 3 Hz, 1H), 6.70-7.73 (m, 1H), 6.94-7.02 (m, 3H), 7.09 (t, *J* = 9 Hz, 1H), 7.48-7.52 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 178.68, 158.07, 156.19, 150.80 (dd, *J* = 7.4, 5.2 Hz),

144.53, 138.36, 133.35, 131.82 (d, *J* = 10 Hz), 127.09, 126.77, 125.96, 124.82, 123.08 (dd, *J* = 273, 245 Hz), 122.73, 122.27 (dd, *J* = 27, 24 Hz), 115.10 (d, *J* = 24 Hz), 112.37, 110.20 (d, *J* = 8.8 Hz), 109.77 (d, *J* = 24 Hz), 85.63, 71.73, 66.92, 39.08; ^{19}F NMR (282 MHz, $CDCl_3$): δ -85.09 (d, *J* = 251 Hz, 1F), -97.77 (d, *J* = 251 Hz, 1F), -124.91 (s, 1F); IR: (KBr) ν_{max} 3415, 2952, 1670, 1596, 1552, 1431, 1087, 753; HRMS (EI): Exact mass calcd for $C_{23}H_{16}F_3N_3S$ $[M]^+$: 423.1017, Found: 423.1021.



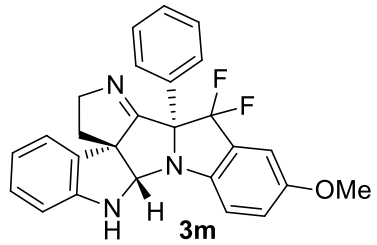
3k

The reaction was run at rt for 48 h, affording product **3k** in 50% yield as a yellow solid (m.p. 194-197 °C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.22-2.30 (m, 2H), 2.39 (s, 3H), 4.42-4.53 (m, 2H), 4.60 (brs, 1H), 4.94 (s, 1H), 6.37 (t, *J* = 10 Hz, 1H), 6.41 (d, *J* = 10 Hz, 1H), 6.62 (d, *J* = 5 Hz, 1H), 6.77-6.84 (m, 3H), 7.00-7.03 (m, 3H), 7.35 (t, *J* = 10 Hz, 1H), 7.40-7.43 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 180.01 (d, *J* = 3.8 Hz), 151.70 (dd, *J* = 7.5, 5 Hz), 148.22, 137.64, 134.37 (d, *J* = 6.3 Hz), 132.98, 130.02, 128.55, 127.79, 127.49, 126.24, 124.86 (dd, *J* = 259, 243 Hz), 123.86, 122.70, 120.02 (dd, *J* = 25, 24 Hz), 119.47, 109.44, 109.27, 84.64, 77.10 (dd, *J* = 30, 20 Hz), 71.29, 66.93, 39.07, 16.98; ^{19}F NMR (471 MHz, $CDCl_3$): δ -84.39 (d, *J* = 254 Hz, 1F), -95.05 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3425, 2933, 1660, 1601, 1484, 1262, 1101, 735; HRMS (EI): Exact mass calcd for $C_{26}H_{21}F_2N_3$ $[M]^+$: 413.1704, Found: 413.1708.

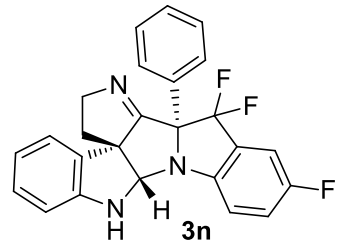


3l

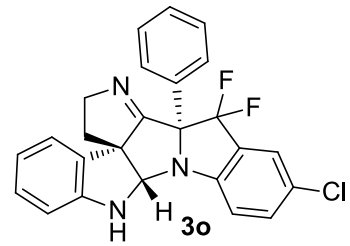
The reaction was run at rt for 36 h, affording product **3l** in 69% yield as a yellow solid (m.p. 176-179 °C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.17-2.30 (m, 2H), 2.33 (s, 3H), 4.40-4.52 (m, 2H), 4.59 (brs, 1H), 4.92 (s, 1H), 6.36-6.40 (m, 2H), 6.63 (d, *J* = 5 Hz, 1H), 6.78 (td, *J* = 10, 5 Hz, 1H), 6.94 (d, *J* = 5 Hz, 1H), 7.00-7.03 (m, 3H), 7.27-7.30 (m, 2H), 7.41-7.43 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 180.02 (d, *J* = 2.5 Hz), 149.18 (dd, *J* = 7.5, 6.3 Hz), 148.25, 134.14 (d, *J* = 6 Hz), 133.98, 131.90, 130.20, 128.56, 127.83, 127.48, 126.37, 124.68, 123.69 (dd, *J* = 259 , 243 Hz), 122.65, 122.20 (dd, *J* = 26, 24 Hz), 119.44, 112.06, 109.23, 84.86, 77.72 (dd, *J* = 30, 20 Hz), 71.24, 66.90, 39.19, 20.72; ^{19}F NMR (471 MHz, $CDCl_3$): δ -81.93 (d, *J* = 254 Hz, 1F), -97.38 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3422, 2928, 1679, 1608, 1496, 1072, 740; HRMS (EI): Exact mass calcd for $C_{26}H_{21}F_2N_3$ $[M]^+$: 413.1704, Found: 413.1700.



The reaction was run at rt for 36 h, affording product **3m** in 49% yield as a pale yellow solid (m.p. 180-182 ^{o}C). ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.19-2.30 (m, 2H), 3.79 (s, 3H), 4.40-4.52 (m, 2H), 4.61 (brs, 1H), 4.89 (s, 1H), 6.36-6.40 (m, 2H), 6.63 (d, *J* = 10 Hz, 1H), 6.78 (td, *J* = 10, 5.0 Hz, 1H), 6.95-6.99 (m, 2H), 7.01-7.03 (m, 3H), 7.06-7.08 (m, 1H), 7.43-7.44 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 180.13 (d, *J* = 2.5 Hz), 155.54, 148.26, 145.16 (dd, *J* = 8.8, 6.3 Hz), 134.09 (d, *J* = 6.3 Hz), 130.25, 128.59, 127.88, 127.50, 126.45, 123.67 (dd, *J* = 259, 244 Hz), 122.74 (dd, *J* = 26, 24 Hz), 122.65, 121.00, 119.48, 113.46, 109.28, 107.95, 85.11 (d, *J* = 1.3 Hz), 78.02 (dd, *J* = 31, 20 Hz), 71.28 (d, *J* = 1.3 Hz), 66.96, 55.90, 39.24; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.24 (d, *J* = 254 Hz, 1F), -97.80 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3396, 2893, 1645, 1614, 1468, 1307, 1070, 788; HRMS (EI): Exact mass calcd for $C_{26}H_{22}F_2N_3O$ $[M]^+$: 430.1725, Found: 430.1734.

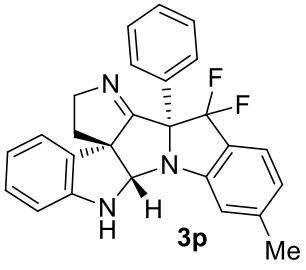


The reaction was run at rt for 36 h, affording product **3n** in 54% yield as a yellow solid (m.p. 149-154 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.21-2.32 (m, 2H), 4.42-4.53 (m, 2H), 4.60 (brs, 1H), 4.91 (s, 1H), 6.37-6.40 (m, 2H), 6.63 (d, *J* = 5 Hz, 1H), 6.79 (td, *J* = 10, 5 Hz, 1H), 6.97-7.00 (m, 1H), 7.01-7.04 (m, 3H), 7.15-7.22 (m, 2H), 7.40-7.42 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.64 (d, *J* = 2.5 Hz), 158.29 (d, *J* = 240 Hz), 148.08, 147.43 (dd, *J* = 7.5, 6.3 Hz), 133.62 (d, *J* = 6 Hz), 130.03, 128.66, 128.04, 127.57, 126.32, 123.23 (dd, *J* = 32, 25 Hz), 122.98 (dd, *J* = 259, 241 Hz), 122.63, 120.46 (d, *J* = 24 Hz), 119.58, 113.27 (d, *J* = 7.5 Hz), 111.39 (d, *J* = 24 Hz), 109.28, 85.05, 78.01 (dd, *J* = 30, 20 Hz), 71.32, 66.92, 39.24; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.68 (d, *J* = 254 Hz, 1F), -97.34 (d, *J* = 254 Hz, 1F), -120.82 (s, 1F); IR: (KBr) ν_{max} 3256, 2950, 1678, 1608, 1491, 1271, 1182, 1070, 743; HRMS (EI): Exact mass calcd for $C_{25}H_{18}F_3N_3$ $[M]^+$: 417.1453, Found: 417.1456.

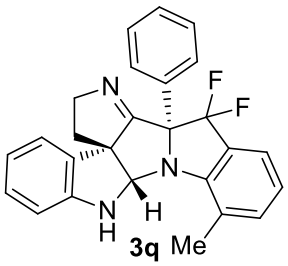


The reaction was run at rt for 36 h, affording product **3o** in 64% yield as a yellow solid (m.p. 105-107 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.20-2.32 (m, 2H), 4.41-4.53 (m, 2H), 4.60 (brs, 1H), 4.93 (s, 1H), 6.37-6.40 (m, 2H), 6.63 (d, *J* = 10 Hz, 1H), 6.79 (td, *J* = 10, 1 Hz, 1H), 6.97 (d, *J* = 10 Hz, 1H), 7.01-7.04 (m, 3H), 7.39-7.40 (m, 2H), 7.43-7.45 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.37 (d, *J* = 3.8 Hz), 149.82 (dd, *J* = 7.5, 6.3 Hz), 148.02, 133.46 (d, *J* = 5 Hz), 133.30, 129.98, 128.70, 128.10, 127.61, 127.06, 126.31, 124.75, 123.77 (dd, *J* = 27, 25 Hz), 122.81 (dd, *J* = 259, 243

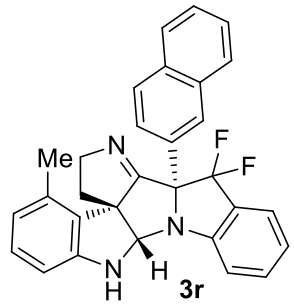
Hz), 122.65, 119.63, 113.33, 109.27, 84.79, 77.87 (dd, *J* = 30, 20 Hz), 71.37, 66.90, 39.28; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.84 (d, *J* = 254 Hz, 1F), -97.15 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3382, 2945, 1658, 1597, 1475, 1296, 743; HRMS (EI): Exact mass calcd for $C_{25}H_{18}ClF_2N_3$ $[M]^+$: 433.1157, Found: 433.1155.



The reaction was run at rt for 36 h, affording product **3p** in 63% yield as a yellow solid (m.p. 102-103 ^oC); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.21-2.30 (m, 2H), 2.43 (s, 3H), 4.41-4.52 (m, 2H), 4.60 (brs, 1H), 4.94 (s, 1H), 6.37 (t, *J* = 10 Hz, 1H), 6.41 (d, *J* = 5 Hz, 1H), 6.62 (d, *J* = 10 Hz, 1H), 6.79 (t, *J* = 10 Hz, 1H), 6.84-6.87 (m, 2H), 7.00-7.02 (m, 3H), 7.34 (d, *J* = 5 Hz, 1H), 7.39-7.43 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.85 (d, *J* = 3.8 Hz), 151.59 (dd, *J* = 7.5, 6.3 Hz), 148.21, 143.91, 134.29 (d, *J* = 6.2 Hz), 130.22, 128.56, 127.80, 127.48, 126.30, 124.34, 123.68 (dd, *J* = 258 , 243 Hz), 123.18, 122.68, 119.55 (dd, *J* = 28, 25 Hz), 119.48, 112.64, 109.28, 84.55, 77.64 (dd, *J* = 30, 20 Hz), 71.33, 66.90, 39.09, 22.04; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.12 (d, *J* = 254 Hz, 1F), -95.45 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3293, 2912, 1667, 1601, 1432, 1297, 1040, 751; HRMS (EI): Exact mass calcd for $C_{26}H_{21}F_2N_3$ $[M]^+$: 413.1704, Found: 413.1706.

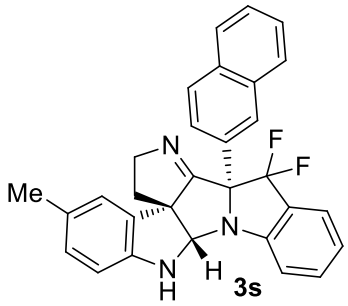


The reaction was run at rt for 48 h, affording product **3q** in 44% yield as a yellow solid (m.p. 126-128 ^oC); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.24-2.27 (m, 2H), 2.58 (s, 3H), 4.44-4.53 (m, 2H), 4.75 (brs, 1H), 4.98 (s, 1H), 6.37 (t, *J* = 10 Hz, 1H), 6.49 (d, *J* = 10 Hz, 1H), 6.59 (d, *J* = 5 Hz, 1H), 6.82 (t, *J* = 10 Hz, 1H), 6.99-7.03 (m, 4H), 7.30-7.33 (m, 2H), 7.44-7.46 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 180.02 (d, *J* = 3.8 Hz), 149.94 (dd, *J* = 7.5, 6.3 Hz), 147.93, 135.50, 134.61 (d, *J* = 5 Hz), 130.36, 128.58, 127.73, 127.54, 126.10, 123.44 (dd, *J* = 259 , 240 Hz), 123.02, 122.85 (dd, *J* = 27, 24 Hz), 122.77, 122.64, 122.20, 119.65, 109.33, 83.18, 77.12 (dd, *J* = 30, 20 Hz), 70.93, 66.90, 39.20, 19.26; ^{19}F NMR (471 MHz, $CDCl_3$): δ -80.75 (d, *J* = 254 Hz, 1F), -94.91 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3409, 2927, 1668, 1609, 1482, 1259, 1461, 1089, 738; HRMS (EI): Exact mass calcd for $C_{26}H_{21}F_2N_3$ $[M]^+$: 413.1704, Found: 413.1691.



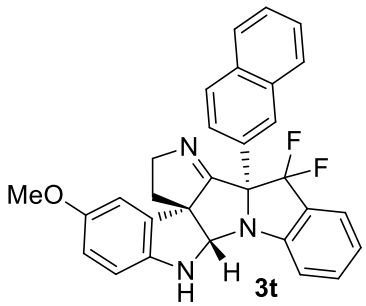
3r

The reaction was run at rt for 36 h, affording product **3r** in 64% yield as a yellow solid (m.p. 124-126 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.76 (s, 3H), 2.14-2.19 (m, 1H), 2.26-2.32 (m, 1H), 4.44-4.50 (m, 1H), 4.56-4.63 (m, 1H), 4.72 (brs, 1H), 4.79 (s, 1H), 5.74 (d, *J* = 10 Hz, 1H), 6.35 (d, *J* = 5 Hz, 1H), 6.43 (t, *J* = 10 Hz, 1H), 7.06-7.09 (m, 2H), 7.32-7.38 (m, 2H), 7.49-7.53 (m, 4H), 7.57-7.58 (m, 1H), 7.64-7.66 (m, 1H), 7.84 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 175.36 (d, *J* = 3.8 Hz), 151.67 (dd, *J* = 7.5, 6.3 Hz), 148.83, 133.29, 133.23, 132.87, 132.58, 131.70 (d, *J* = 6.3 Hz), 128.76, 128.64, 127.81, 127.36, 127.29, 125.72, 125.58, 124.87, 124.66, 123.98 (dd, *J* = 259, 241 Hz), 123.30, 122.45 (dd, *J* = 27, 25 Hz), 122.27, 121.58, 112.50, 107.19, 87.08, 77.69 (dd, *J* = 30, 20 Hz), 70.93, 66.72, 35.11, 17.63; ^{19}F NMR (471 MHz, $CDCl_3$): δ -79.72 (d, *J* = 254 Hz, 1F), -95.79 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3300, 2925, 1671, 1608, 1480, 1265, 1090, 808; HRMS (EI): Exact mass calcd for $C_{30}H_{23}F_2N_3$ $[M]^+$: 463.1860, Found: 463.1858.



3s

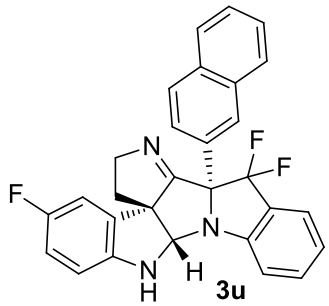
The reaction was run at rt for 36 h, affording product **3s** in 68% yield as a yellow solid (m.p. 142-146 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.60 (s, 3H), 2.25-2.27 (m, 2H), 4.44-4.55 (m, 3H), 4.94 (s, 1H), 6.20-6.21 (m, 2H), 6.26 (s, 1H), 7.05-7.08 (m, 2H), 7.32-7.38 (m, 2H), 7.48-7.56 (m, 4H), 7.58 (d, *J* = 10 Hz, 1H), 7.65 (d, *J* = 10 Hz, 1H), 7.76 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.76 (d, *J* = 3.8 Hz), 151.51 (dd, *J* = 7.6, 5.8 Hz), 145.93, 133.29, 132.76, 132.43, 131.80 (d, *J* = 6.3 Hz), 130.48, 129.05, 128.83, 127.82, 127.20, 127.14, 126.24, 125.83, 125.49, 124.75, 123.96, 123.78 (dd, *J* = 260, 243 Hz), 123.14, 122.30 (dd, *J* = 27, 25 Hz), 122.15, 112.17, 109.25, 85.40, 77.69 (dd, *J* = 31, 21 Hz), 71.57, 67.03, 38.29, 20.03; ^{19}F NMR (471 MHz, $CDCl_3$): δ -81.92 (d, *J* = 254 Hz, 1F), -95.22 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3421, 2930, 1665, 1610, 1487, 1255, 1073, 766; HRMS (EI): Exact mass calcd for $C_{30}H_{23}F_2N_3$ $[M]^+$: 463.1860, Found: 463.1866.



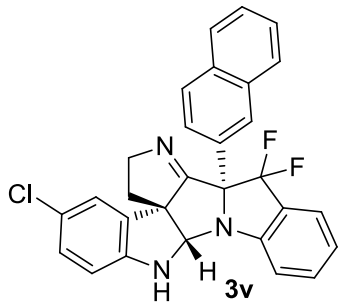
3t

The reaction was run at rt for 36 h, affording product **3t** in 71% yield as a yellow solid (m.p. 159-164 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.26-2.28 (m, 2H), 3.11 (s, 3H), 4.47-4.50 (m, 3H), 4.95 (s, 1H), 6.03 (dd, *J* = 10, 5 Hz, 1H), 6.06 (d, *J* = 5 Hz, 1H), 6.28 (d, *J* = 10 Hz, 1H), 7.06-7.09 (m, 2H), 7.33-7.38 (m, 2H), 7.48-7.53 (m, 2H), 7.56-7.61 (m, 3H), 7.65-7.68 (m, 1H), 7.80 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.47 (d, *J* = 2.5 Hz), 153.69, 151.45 (dd, *J* = 7.5, 5

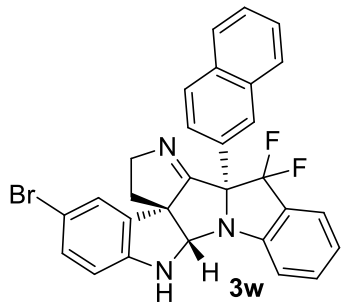
Hz), 142.35, 133.32, 132.79, 132.46, 131.83 (d, *J* = 5 Hz), 131.72, 127.85, 127.33, 127.24, 126.10, 125.89, 125.58, 124.72, 123.96, 123.79 (dd, *J* = 260, 244 Hz), 122.29 (dd, *J* = 27, 25 Hz), 122.19, 114.29, 112.20, 110.14, 109.56, 85.77, 77.62 (dd, *J* = 31, 21 Hz), 71.93, 67.03, 55.96, 38.14; ^{19}F NMR (471 MHz, $CDCl_3$): δ -82.40 (d, *J* = 254 Hz, 1F), -94.46 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3385, 2927, 1669, 1611, 1492, 1208, 811, 751; HRMS (EI): Exact mass calcd for $C_{30}H_{23}F_2N_3O$ $[M]^+$: 479.1809, Found: 479.1808.



The reaction was run at rt for 36 h, affording product **3u** in 86% yield as a yellow solid (m.p. 223-225 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.23-2.32 (m, 2H), 4.47-4.50 (m, 2H), 4.54 (s, 1H), 5.01 (s, 1H), 6.15 (td, *J* = 10, 5 Hz, 1H), 6.19 (dd, *J* = 10, 5 Hz, 1H), 6.28 (dd, *J* = 10, 5 Hz, 1H), 7.07-7.10 (m, 2H), 7.35-7.41 (m, 2H), 7.48-7.53 (m, 2H), 7.58-7.61 (m, 3H), 7.68 (d, *J* = 5 Hz, 1H), 7.83 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.12 (d, *J* = 2.5 Hz), 157.72, 155.84, 151.18 (dd, *J* = 7.5, 6.3 Hz), 144.31, 133.32, 132.85, 132.44, 131.35 (dd, *J* = 29, 7.5 Hz), 127.71, 127.50, 127.35, 126.21, 126.08, 125.81, 124.77, 123.88, 123.66 (dd, *J* = 260, 244 Hz), 122.31, 122.22 (dd, *J* = 27, 25 Hz), 114.71 (d, *J* = 24 Hz), 112.15, 109.57, 109.43 (d, *J* = 15 Hz), 85.37, 77.64 (dd, *J* = 31, 21 Hz), 71.54, 66.94, 38.72; ^{19}F NMR (471 MHz, $CDCl_3$): δ -81.93 (d, *J* = 254 Hz, 1F), -96.32 (d, *J* = 250 Hz, 1F), -125.20 (s, 1F); IR: (KBr) ν_{max} 3416, 2912, 1684, 1607, 1466, 1108, 782; HRMS (EI): Exact mass calcd for $C_{29}H_{20}F_3N_3$ $[M]^+$: 467.1609, Found: 467.1603.

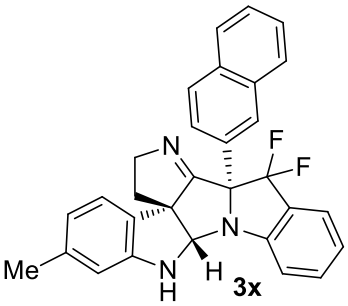


The reaction was run at rt for 36 h, affording product **3v** in 78% yield as a yellow solid (m.p. 244-247 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.21-2.32 (m, 2H), 4.44-4.55 (m, 2H), 4.62 (s, 1H), 5.01 (s, 1H), 6.13 (d, *J* = 10 Hz, 1H), 6.34 (dd, *J* = 10, 5 Hz, 1H), 6.51 (d, *J* = 5 Hz, 1H), 7.05-7.10 (m, 2H), 7.36-7.42 (m, 2H), 7.50-7.56 (m, 3H), 7.59-7.62 (m, 2H), 7.69 (d, *J* = 5 Hz, 1H), 7.79 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 178.99 (d, *J* = 3.8 Hz), 151.08 (dd, *J* = 7.5, 6.3 Hz), 146.86, 133.31, 132.90, 132.41, 131.78, 131.03 (d, *J* = 5 Hz), 128.09, 127.76, 127.47, 127.35, 126.50, 126.15, 125.83, 124.83, 123.93, 123.80, 123.52 (dd, *J* = 260, 243 Hz), 122.56, 122.35, 122.19 (dd, *J* = 27, 25 Hz), 112.08, 109.66, 84.96, 77.76 (dd, *J* = 31, 21 Hz), 71.12, 67.00, 38.68; ^{19}F NMR (471 MHz, $CDCl_3$): δ -80.93 (d, *J* = 259 Hz, 1F), -97.90 (d, *J* = 259 Hz, 1F); IR: (KBr) ν_{max} 3482, 2935, 1667, 1598, 1432, 1204, 741; HRMS (EI): Exact mass calcd for $C_{29}H_{20}ClF_2N_3$ $[M]^+$: 483.1314, Found: 483.1310.



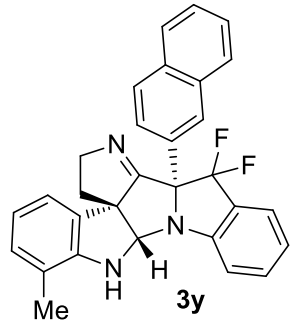
3w

The reaction was run at rt for 36 h, affording product **3w** in 52% yield as a yellow solid (m.p. 125-128 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.21-2.32 (m, 2H), 4.44-4.54 (m, 2H), 4.62 (brs, 1H), 5.00 (s, 1H), 6.09 (d, *J* = 5 Hz, 1H), 6.47 (dd, *J* = 10, 5 Hz, 1H), 6.63 (d, *J* = 5 Hz, 1H), 7.05-7.10 (m, 2H), 7.36-7.43 (m, 2H), 7.50-7.56 (m, 3H), 7.59-7.62 (m, 2H), 7.69 (d, *J* = 10 Hz, 1H), 7.78 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 178.96 (d, *J* = 3.8 Hz), 151.07 (dd, *J* = 7.5, 6.3 Hz), 147.31, 133.31, 132.92, 132.41, 132.23, 131.02 (d, *J* = 5 Hz), 130.92, 127.81, 127.47, 127.38, 126.55, 126.16, 125.84, 125.47, 124.85, 123.92, 123.49 (dd, *J* = 260, 243 Hz), 122.36, 122.19 (dd, *J* = 27, 25 Hz), 112.07, 110.79, 110.14, 84.88, 77.79 (dd, *J* = 31, 21 Hz), 71.03, 67.02, 38.61; ^{19}F NMR (471 MHz, $CDCl_3$): δ -80.78 (d, *J* = 259 Hz, 1F), -98.11 (d, *J* = 259 Hz, 1F); IR: (KBr) ν_{max} 3223, 2930, 1668, 1469, 1308, 1091, 747; HRMS (EI): Exact mass calcd for $C_{29}H_{20}BrF_2N_3$ $[M]^+$: 527.0809, Found: 527.0804.



3x

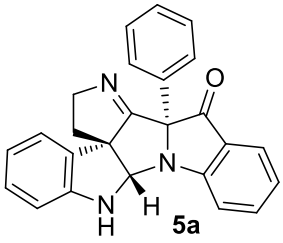
The reaction was run at rt for 36 h, affording product **3x** in 53% yield as a yellow solid (m.p. 130-133 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.69 (s, 3H), 2.21-2.28 (m, 2H), 4.41-4.52 (m, 2H), 4.58 (brs, 1H), 4.98 (s, 1H), 5.85 (d, *J* = 10 Hz, 1H), 6.10 (s, 1H), 6.38 (d, *J* = 5 Hz, 1H), 7.05-7.08 (m, 2H), 7.34-7.37 (m, 2H), 7.48-7.52 (m, 2H), 7.55-7.58 (m, 3H), 7.65-7.67 (m, 1H), 7.77 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.98 (d, *J* = 3.8 Hz), 151.35 (dd, *J* = 7.5, 6.3 Hz), 148.47, 138.77, 133.25, 132.74, 132.44, 131.64 (d, *J* = 5 Hz), 127.78, 127.44, 127.25, 127.24, 126.11, 125.75, 125.50, 124.73, 123.97, 123.74 (dd, *J* = 260, 244 Hz), 122.28 (dd, *J* = 27, 25 Hz), 122.13, 122.04, 120.22, 112.18, 109.81, 85.04, 77.74 (dd, *J* = 31, 20 Hz), 71.06, 66.92, 38.64, 20.82; ^{19}F NMR (471 MHz, $CDCl_3$): δ -81.53 (d, *J* = 254 Hz, 1F), -96.34 (d, *J* = 250 Hz, 1F); IR: (KBr) ν_{max} 3289, 2971, 1665, 1608, 1508, 1470, 1063, 742; HRMS (EI): Exact mass calcd for $C_{30}H_{23}F_2N_3$ $[M]^+$: 463.1860, Found: 463.1856.



3y

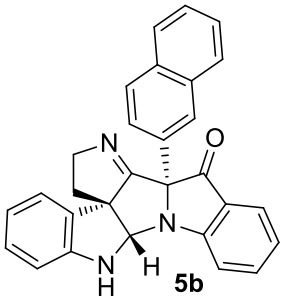
The reaction was run at rt for 36 h, affording product **3y** in 51% yield as a yellow solid (m.p. 141-142 ^{o}C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.92 (s, 3H), 2.20-2.26 (m, 1H), 2.30-2.34 (m, 1H), 4.41-4.55 (m, 3H), 5.06 (s, 1H), 6.05 (t, *J* = 10 Hz, 1H), 6.24 (d, *J* = 5 Hz, 1H), 6.45 (d, *J* = 10 Hz, 1H), 7.07 (t, *J* = 10 Hz, 1H), 7.12 (d, *J* = 5 Hz, 1H), 7.32-7.38 (m, 2H), 7.49-7.57 (m, 4H), 7.64 (d, *J* = 10 Hz, 2H), 7.82 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 179.91 (d, *J* = 2.5 Hz), 151.21 (dd, *J* = 7.5, 5 Hz), 146.79, 133.22, 132.85, 132.50, 131.26 (d, *J* = 5 Hz), 129.55,

129.05, 127.70, 127.25, 127.03, 125.96, 125.85, 125.57, 124.74, 123.95, 123.57 (dd, *J* = 260, 244 Hz), 122.13, 122.09 (dd, *J* = 27, 25 Hz), 119.66, 119.55, 118.27, 112.16, 84.41, 77.78 (dd, *J* = 31, 20 Hz), 71.49, 66.90, 39.07, 16.42; ^{19}F NMR (471 MHz, $CDCl_3$): δ -79.93 (d, *J* = 254 Hz, 1F), -99.45 (d, *J* = 254 Hz, 1F); IR: (KBr) ν_{max} 3415, 2927, 1665, 1609, 1495, 1468, 1259, 1091, 752; HRMS (EI): Exact mass calcd for $C_{30}H_{23}F_2N_3$ $[M]^+$: 463.1860, Found: 463.1857.



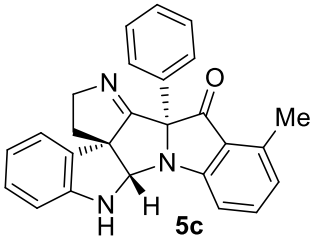
5a

The reaction was run at rt for 48 h, affording product **5a** in 65% yield as a yellow foamy solid (m.p. 138-141 °C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.29-2.38 (m, 2H), 4.34-4.39 (m, 1H), 4.43-4.49 (m, 1H), 4.73 (brs, 1H), 4.98 (s, 1H), 6.44 (t, *J* = 10 Hz, 1H), 6.52 (d, *J* = 10 Hz, 1H), 6.69 (d, *J* = 5 Hz, 1H), 6.86 (t, *J* = 10 Hz, 1H), 6.99-7.07 (m, 4H), 7.19 (d, *J* = 10 Hz, 1H), 7.43-7.45 (m, 2H), 7.61-7.65 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 194.95, 178.36, 162.08, 148.14, 137.43, 135.15, 129.50, 128.84, 127.85, 127.55, 125.97, 125.12, 122.69, 122.15, 121.19, 119.82, 113.35, 109.50, 84.75, 77.31, 73.32, 66.12, 38.99; IR: (KBr) ν_{max} 3365, 2931, 1711, 1663, 1605, 1472, 1320, 1153, 739; HRMS (EI): Exact mass calcd for $C_{25}H_{19}N_3O$ $[M]^+$: 377.1528, Found: 377.1525.



5b

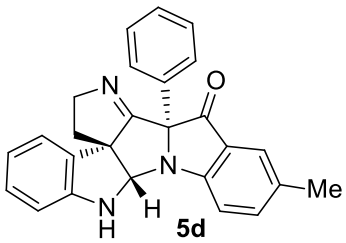
The reaction was run at rt for 48 h, affording product **5b** in 54% yield as a yellow foamy solid (m.p. 155-157 °C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.31-2.40 (m, 2H), 4.38-4.43 (m, 1H), 4.47-4.54 (m, 1H), 4.82 (brs, 1H), 5.03 (s, 1H), 6.24 (td, *J* = 10, 5 Hz, 1H), 6.47 (d, *J* = 5 Hz, 1H), 6.62-6.67 (m, 2H), 7.06 (t, *J* = 10 Hz, 1H), 7.24 (d, *J* = 10 Hz, 1H), 7.31-7.35 (m, 2H), 7.54 (d, *J* = 5 Hz, 1H), 7.59-7.68 (m, 5H), 7.85 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 195.07, 178.36, 162.15, 148.09, 137.49, 132.76, 132.69, 132.68, 129.45, 128.83, 127.85, 127.76, 127.31, 126.00, 125.64, 125.55, 124.56, 122.93, 122.47, 122.23, 121.28, 119.75, 113.45, 109.38, 84.86, 77.44, 73.41, 66.17, 38.97; IR: (KBr) ν_{max} 3367, 2926, 1714, 1666, 1604, 1473, 1321, 1156, 744; HRMS (EI): Exact mass calcd for $C_{29}H_{21}N_3O$ $[M]^+$: 427.1685, Found: 427.1677.



5c

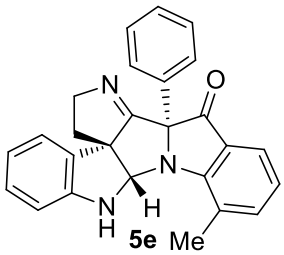
The reaction was run at rt for 36 h, affording product **5c** in 56% yield as a yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.28-2.37 (m, 2H), 2.52 (s, 3H), 4.35-4.40 (m, 1H), 4.43-4.49 (m, 1H), 4.71 (brs, 1H), 4.96 (s, 1H), 6.42 (t, *J* = 5 Hz, 1H), 6.52 (d, *J* = 5 Hz, 1H), 6.68 (d, *J* = 5 Hz, 1H), 6.79 (d, *J* = 5 Hz, 1H), 6.85 (t, *J* = 5 Hz, 1H), 6.98-7.05 (m, 4H), 7.43-7.49 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): 195.60, 178.65, 162.71, 148.19, 141.56, 136.73, 135.53, 129.59, 128.80, 127.82, 127.48, 125.08, 123.84,

122.72, 119.80, 118.94, 110.54, 109.50, 84.86, 76.95, 73.10, 66.14, 38.93, 18.10; IR: (KBr) ν_{max} 3391, 2928, 1704, 1665, 1596, 1485, 1314, 1029, 746; HRMS (EI): Exact mass calcd for $C_{26}H_{21}N_3O$ $[M]^+$: 391.1685, Found: 391.1682.



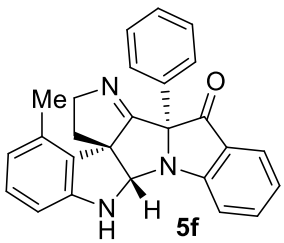
5d

The reaction was run at rt for 36 h, affording product **5d** in 61% yield as a yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.28-2.32 (m, 2H), 2.33 (s, 3H), 4.33-4.38 (m, 1H), 4.42-4.49 (m, 1H), 4.71 (brs, 1H), 4.93 (s, 1H), 6.43 (t, *J* = 5 Hz, 1H), 6.52 (d, *J* = 5 Hz, 1H), 6.68 (d, *J* = 10 Hz, 1H), 6.85 (td, *J* = 10, 5 Hz, 1H), 6.98-7.05 (m, 3H), 7.10 (d, *J* = 10 Hz, 1H), 7.41-7.47 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): 194.99, 178.53, 160.43, 148.20, 138.64, 135.33, 131.95, 129.57, 128.80, 127.80, 127.46, 125.42, 125.15, 122.69, 121.20, 119.77, 113.23, 109.50, 84.89, 77.68, 73.28, 66.15, 39.01, 20.56; IR: (KBr) ν_{max} 3390, 2925, 1714, 1667, 1620, 1487, 1340, 1045, 745; HRMS (EI): Exact mass calcd for $C_{26}H_{21}N_3O$ $[M]^+$: 391.1685, Found: 391.1686.



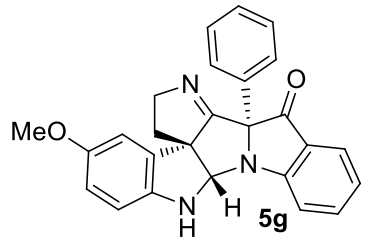
5e

The reaction was run at rt for 36 h, affording product **5e** in 37% yield as a yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.27-2.37 (m, 2H), 2.65 (s, 3H), 4.36-4.40 (m, 1H), 4.43-4.49 (m, 1H), 4.83 (brs, 1H), 5.04 (s, 1H), 6.44 (t, *J* = 10 Hz, 1H), 6.57 (d, *J* = 10 Hz, 1H), 6.68 (d, *J* = 10 Hz, 1H), 6.88 (td, *J* = 10, 5 Hz, 1H), 6.98-7.05 (m, 4H), 7.45-7.50 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): 195.13, 178.48, 161.16, 147.93, 139.30, 135.35, 129.71, 128.84, 127.80, 127.46, 125.08, 124.41, 123.55, 122.74, 122.66, 121.92, 119.91, 109.52, 83.12, 77.36, 72.79, 66.07, 39.24, 18.95; IR: (KBr) ν_{max} 3413, 2929, 1713, 1667, 1602, 1486, 1317, 1052, 739; HRMS (EI): Exact mass calcd for $C_{26}H_{21}N_3O$ $[M]^+$: 391.1685, Found: 391.1682.

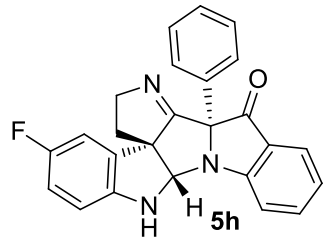


5f

The reaction was run at rt for 48 h, affording product **5f** in 40% yield as a yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.84 (s, 3H), 2.17-2.22 (m, 1H), 2.33-2.39 (m, 1H), 4.35-4.41 (m, 1H), 4.48-4.53 (m, 1H), 4.75 (brs, 1H), 4.78 (s, 1H), 6.16 (d, *J* = 5 Hz, 1H), 6.49 (d, *J* = 5 Hz, 1H), 6.79 (t, *J* = 5 Hz, 1H), 7.00-7.07 (m, 4H), 7.18 (d, *J* = 10 Hz, 1H), 7.43-7.47 (m, 2H), 7.62-7.65 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 195.36, 174.68, 162.27, 148.79, 137.41, 134.80, 133.70, 129.00, 128.08, 127.76, 127.48, 125.92, 124.71, 122.26, 121.94, 121.73, 113.61, 107.59, 86.57, 77.21, 72.79, 65.94, 35.04, 17.52; IR: (KBr) ν_{max} 3375, 2923, 1715, 1668, 1606, 1473, 1322, 1051, 744; HRMS (EI): Exact mass calcd for $C_{26}H_{21}N_3O$ $[M]^+$: 391.1685, Found: 391.1682.

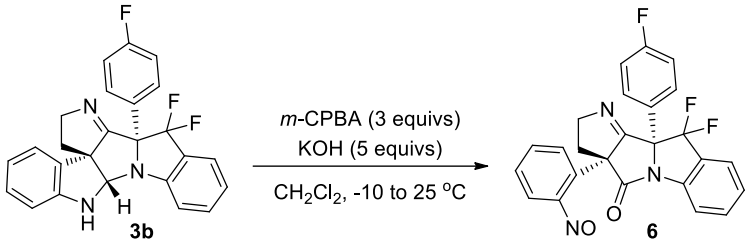


The reaction was run at rt for 48 h, affording product **5g** in 44% yield as a yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.27-2.37 (m, 2H), 3.52 (s, 3H), 4.34-4.47 (m, 2H), 4.52 (brs, 1H), 4.94 (s, 1H), 6.22 (d, *J* = 2.5 Hz, 1H), 6.44-6.49 (m, 2H), 7.00-7.06 (m, 4H), 7.19 (d, *J* = 5 Hz, 1H), 7.43-7.45 (m, 2H), 7.61-7.65 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 194.91, 178.10, 162.19, 154.21, 142.29, 137.44, 135.30, 131.04, 127.86, 127.54, 125.95, 125.14, 122.19, 121.28, 115.00, 113.39, 110.66, 109.08, 85.74, 77.31, 73.84, 66.17, 56.21, 38.35; IR: (KBr) ν_{max} 3382, 2929, 1715, 1665, 1605, 1492, 1321, 1042, 754; HRMS (EI): Exact mass calcd for $C_{26}H_{21}N_3O_2$ $[M]^+$: 407.1634, Found: 407.1636.



The reaction was run at rt for 48 h, affording product **5h** in 60% yield as a yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.28-2.38 (m, 2H), 4.34-4.46 (m, 2H), 4.66 (brs, 1H), 4.99 (s, 1H), 6.38 (dd, *J* = 10, 5 Hz, 1H), 6.44 (dd, *J* = 10, 5 Hz, 1H), 6.56 (td, *J* = 10, 5 Hz, 1H), 7.02-7.08 (m, 4H), 7.18 (d, *J* = 10 Hz, 1H), 7.44-7.46 (m, 2H), 7.61-7.65 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 194.67, 177.72, 161.93, 158.15, 156.26, 144.35, 137.48, 134.96, 130.89 (d, *J* = 7.5 Hz), 128.00, 127.80, 126.01, 125.13, 122.30, 121.17, 115.25 (d, *J* = 24 Hz), 113.33, 110.08 (d, *J* = 7.5 Hz), 109.81 (d, *J* = 25 Hz), 85.43, 77.20, 73.49, 66.12, 38.69; ^{19}F NMR (471 MHz, $CDCl_3$): δ -124.63 (s, 1F); IR: (KBr) ν_{max} 3398, 2925, 1712, 1665, 1622, 1482, 1260, 740; HRMS (EI): Exact mass calcd for $C_{25}H_{18}FN_3O$ $[M]^+$: 395.1434, Found: 395.1440.

4. Transformation of compound 3b



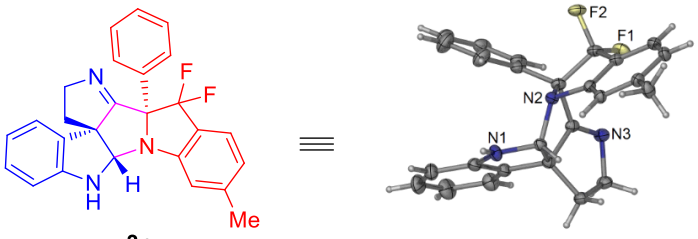
To a dried 10.0 mL Schlenk tube were successively added compound **3b** (41.7 mg, 0.1 mmol), *m*-CPBA (51.6 mg, 0.3 mmol), CH_2Cl_2 (2.0 mL) and potassium hydroxide (28 mg, 0.5 mmol) at -10 °C. The resulting reaction mixture was then stirred at 25 °C for 3 h till full consumption of compound **3b** as monitored by TLC. The crude reaction mixture was directly purified by column chromatography using CH_2Cl_2/EtOAc (from 10:1) as the eluent to provide the corresponding product **6** in 27% yield as a yellow oil (12.1 mg).

Data of compound 6: ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.69-2.75 (m, 1H), 2.91-3.02 (m, 1H), 4.12-4.23 (m, 1H), 4.42-4.50 (m, 1H), 5.70 (dd, *J* = 9, 1.5 Hz, 1H), 6.62-6.68 (m, 2H), 7.05-7.10 (m, 3H), 7.31 (t, *J* = 9 Hz, 1H), 7.44 (td, *J* = 9, 3 Hz, 1H), 7.51 (d, *J* = 9 Hz, 1H), 7.60-7.65 (m, 2H), 7.89 (d, *J* = 9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): 170.86, 170.18, 163.55, 161.57, 161.54, 141.62, 139.18 (dd, *J* = 6.5, 5.1 Hz), 135.52, 133.17 (d, *J* = 2.5 Hz), 128.77, 128.49, 127.85, 127.12, 127.11, 126.66 (dd, *J* = 28, 25 Hz), 126.55 (d, *J* = 1.3 Hz), 124.61, 122.34 (dd, *J* = 260, 253 Hz), 117.68 (d, *J* = 1.3 Hz), 115.13 (d, *J* = 23 Hz), 104,97, 77.16 (dd, *J* = 23, 20 Hz), 70.13, 64.09, 37.53; ^{19}F NMR (282 MHz, $CDCl_3$): δ -79.87 (d, *J* = 240 Hz, 1F), -111.33 (d, *J* = 240 Hz, 1F), -112.41 (s, 1F); IR: (KBr) ν_{max} 2943, 1690, 1645, 1594, 1432, 1002, 744; HRMS (ESI): Exact mass calcd for $C_{25}H_{16}F_3N_3O_2$ $[M+H]^+$: 448.1267, Found: 448.1265.

5. X-Ray crystallographic data for compounds 3p, 5a and 6

Compound 3p

Data intensity of **3p** was collected using a Bruker 'Bruker APEX-II CCD' diffractometer at 100(10) K. Data collection and reduction were done by using Olex2 and the structure was solved with the ShelXS structure solution program using direct methods and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for non-H atoms using SHELX-97. Hydrogen atoms were added at their geometrically idea positions and refined isotropically. CCDC deposition number: 1830099 (**3p**).



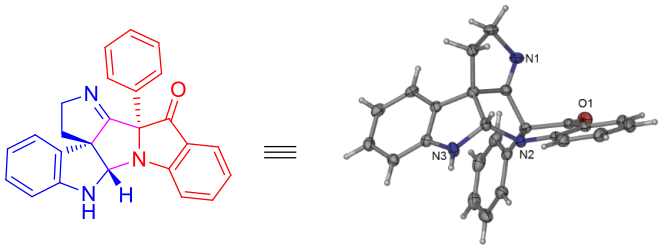
3p X-ray structure of product **3p**

Crystal data

Empirical formula	$C_{26}H_{21}F_2N_3$
Formula weight	413.46
Temperature/K	100.00(10)
Crystal system	tetragonal
Space group	$I4_1/a$
a/Å	18.76710(10)
b/Å	18.76710(10)
c/Å	24.2076(3)
α/	90
β/	90
γ/	90
Volume/Å^3	8526.01(14)
Z	16
ρ_{calc}g/cm^3	1.288
μ/mm^{-1}	0.726
F(000)	3456.0
Crystal size/mm^3	0.49×0.32×0.21
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.426 to 134.116
Index ranges	$-20 \le h \le 22$, $-22 \le k \le 22$, $-23 \le l \le 28$
Reflections collected	44385
Independent reflections	3816 [R_{int} = 0.0852, R_{sigma} = 0.0320]
Data/restraints/parameters	3816/0/281
Goodness-of-fit on F^2	1.041
Final R indexes [I>=2σ (I)]	R_1 = 0.0395, wR_2 = 0.0980
Final R indexes [all data]	R_1 = 0.0426, wR_2 = 0.1000
Largest diff. peak/hole / e Å^{-3}	0.27/-0.26

Compound 5a

Data intensity of **5a** was collected using a Bruker 'Bruker APEX-II CCD' diffractometer at 100(10) K. Data collection and reduction were done by using Olex2 and the structure was solved with the ShelXS structure solution program using direct methods and refined by full-matrix least-squares on F ^{2} with anisotropic displacement parameters for non-H atoms using SHELX-97. Hydrogen atoms were added at their geometrically idea positions and refined isotropically. CCDC deposition number: 1830098 (**5a**).



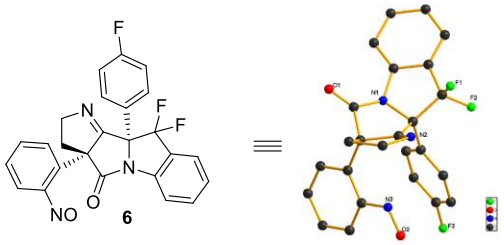
5a X-ray structure of product **5a**

Crystal data.

Identification code	exp_234
Empirical formula	$C_{25}H_{19}N_3O$
Formula weight	377.43
Temperature/K	100.01(10)
Crystal system	triclinic
Space group	P-1
a/Å	8.2339(2)
b/Å	14.7918(4)
c/Å	17.1794(5)
α/	101.330(2)
β/	95.203(2)
γ/	106.126(2)
Volume/Å^3	1947.28(9)
Z	2
ρ_{calc}g/cm^3	1.287
μ/mm^{-1}	0.633
F(000)	792.0
Crystal size/mm^3	0.45 × 0.38 × 0.34
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.216 to 134.14
Index ranges	-9 ≤ h ≤ 5, -17 ≤ k ≤ 17, -19 ≤ l ≤ 20
Reflections collected	19522
Independent reflections	6920 [R_{int} = 0.0399, R_{sigma} = 0.0407]
Data/restraints/parameters	6920/0/527
Goodness-of-fit on F^2	1.030
Final R indexes [I>=2σ (I)]	R_1 = 0.0407, wR_2 = 0.0982
Final R indexes [all data]	R_1 = 0.0445, wR_2 = 0.1005
Largest diff. peak/hole / e Å^{-3}	0.38/-0.31

Compound 6

Data intensity of **6** was collected using a Bruker 'Bruker APEX-II CCD' diffractometer at 100(10) K. Data collection and reduction were done by using Olex2 and the structure was solved with the ShelXS structure solution program using direct methods and refined by full-matrix least-squares on F ^{2} with anisotropic displacement parameters for non-H atoms using SHELX-97. CCDC deposition number: 1830573 (**6**).



X-ray structure of compound **6**

Datablock: 1

Bond precision: C-C = 0.0022 A Wavelength=1.54178

Cell:	a=19.5242(3)	b=8.4475(1)	c=25.5820(4)
	alpha=90	beta=107.178(2)	gamma=90
Temperature:	100 K		

	Calculated	Reported
Volume	4031.04(11)	4031.04(11)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C25 H16 F3 N3 O2	C25 H16 F3 N3 O2
Sum formula	C25 H16 F3 N3 O2	C25 H16 F3 N3 O2
Mr	447.41	447.41
Dx,g cm-3	1.474	1.474
Z	8	8
Mu (mm-1)	0.968	0.968
F000	1840.0	1840.0
F000'	1846.58	
h,k,lmax	24,10,31	24,10,31
Nref	8222	8222
Tmin,Tmax	0.785,0.824	0.785,0.824
Tmin'	0.785	

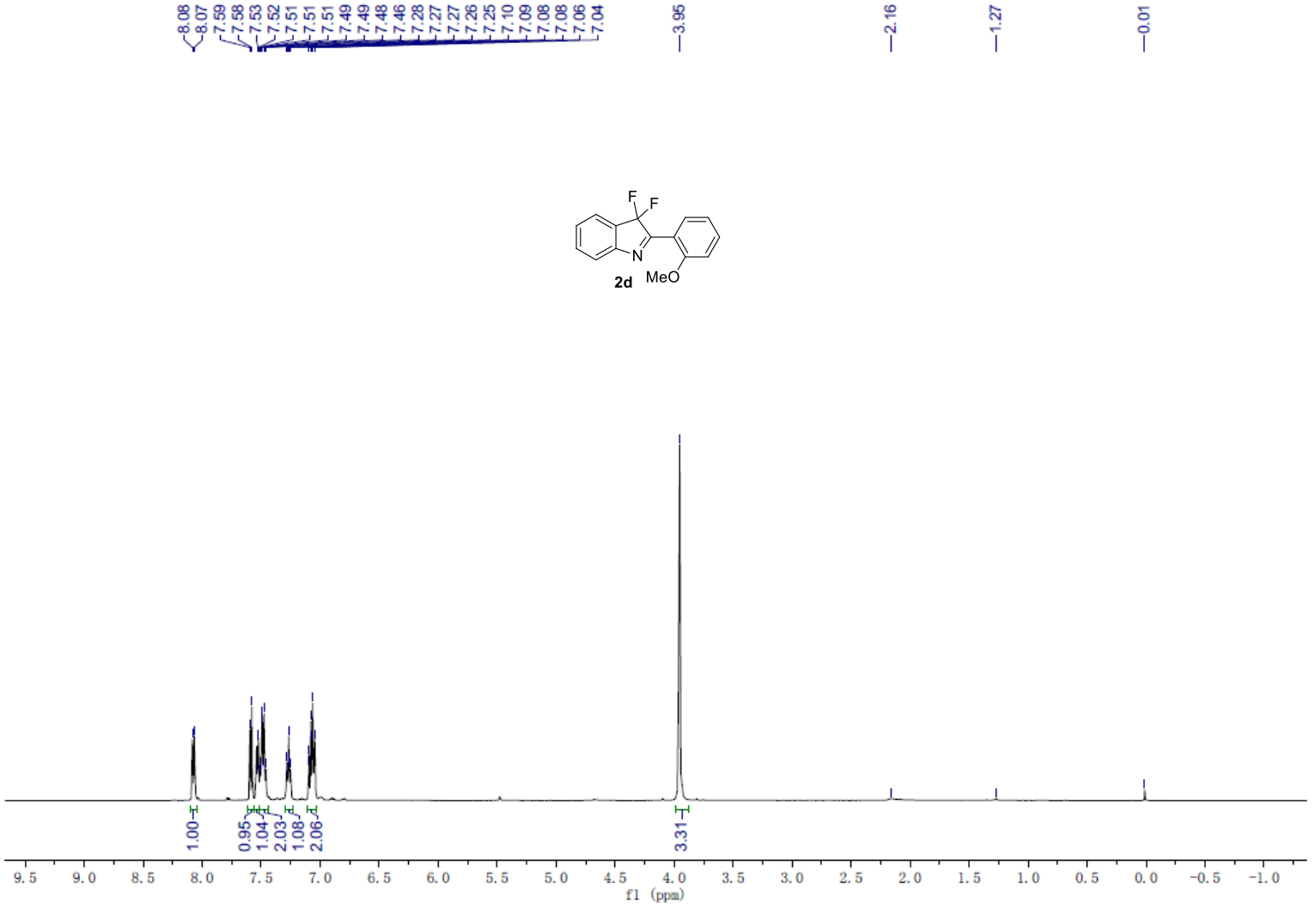
Correction method= # Reported T Limits: Tmin=0.785 Tmax=0.824
AbsCorr = MULTI-SCAN

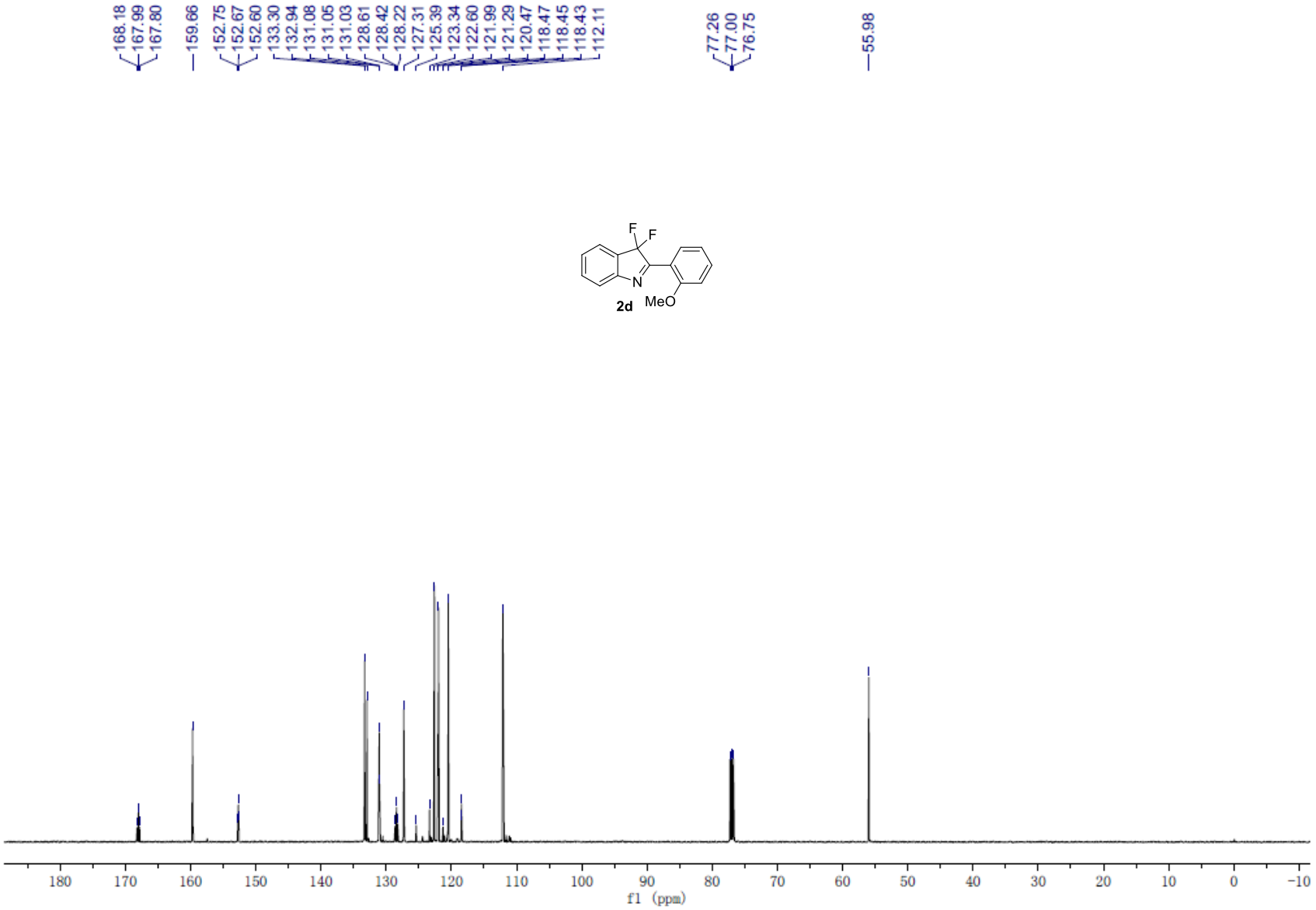
Data completeness= 1.000 Theta(max)= 74.361

R(reflections)= 0.0494(7388) wR2(reflections)= 0.1456(8129)

S = 1.057 Npar= 595

6. Copies of ^{1}H NMR, ^{19}F NMR and ^{13}C NMR Spectra

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131.03
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127.31
125.39
123.34
122.60
121.99
121.29
120.47
118.47
118.45
118.43
112.11
77.26
77.00
76.75
55.98
F
F
N
MeO
2d
180
170
160
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100
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80
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60
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40
30
20
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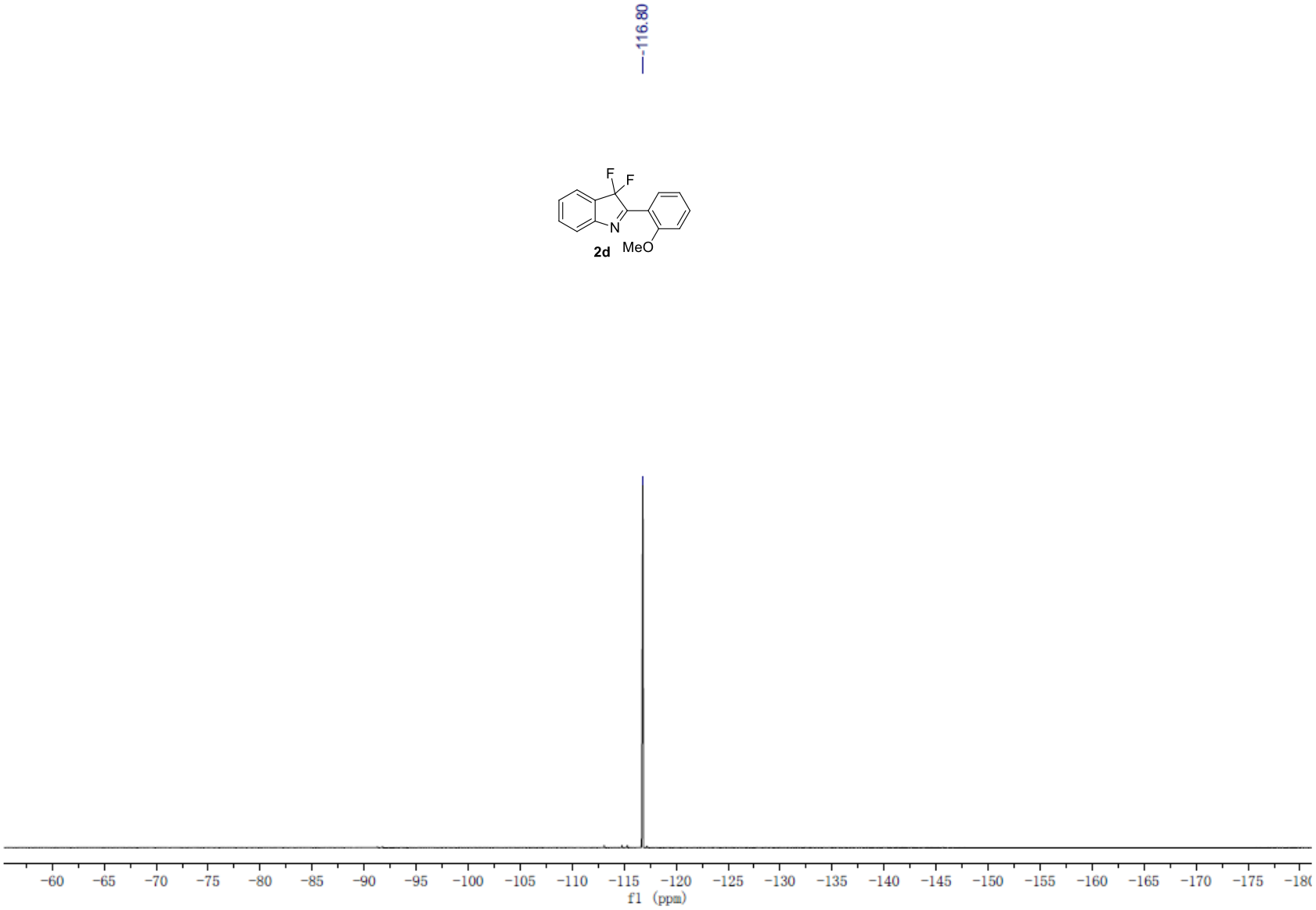
−10
f1 (ppm)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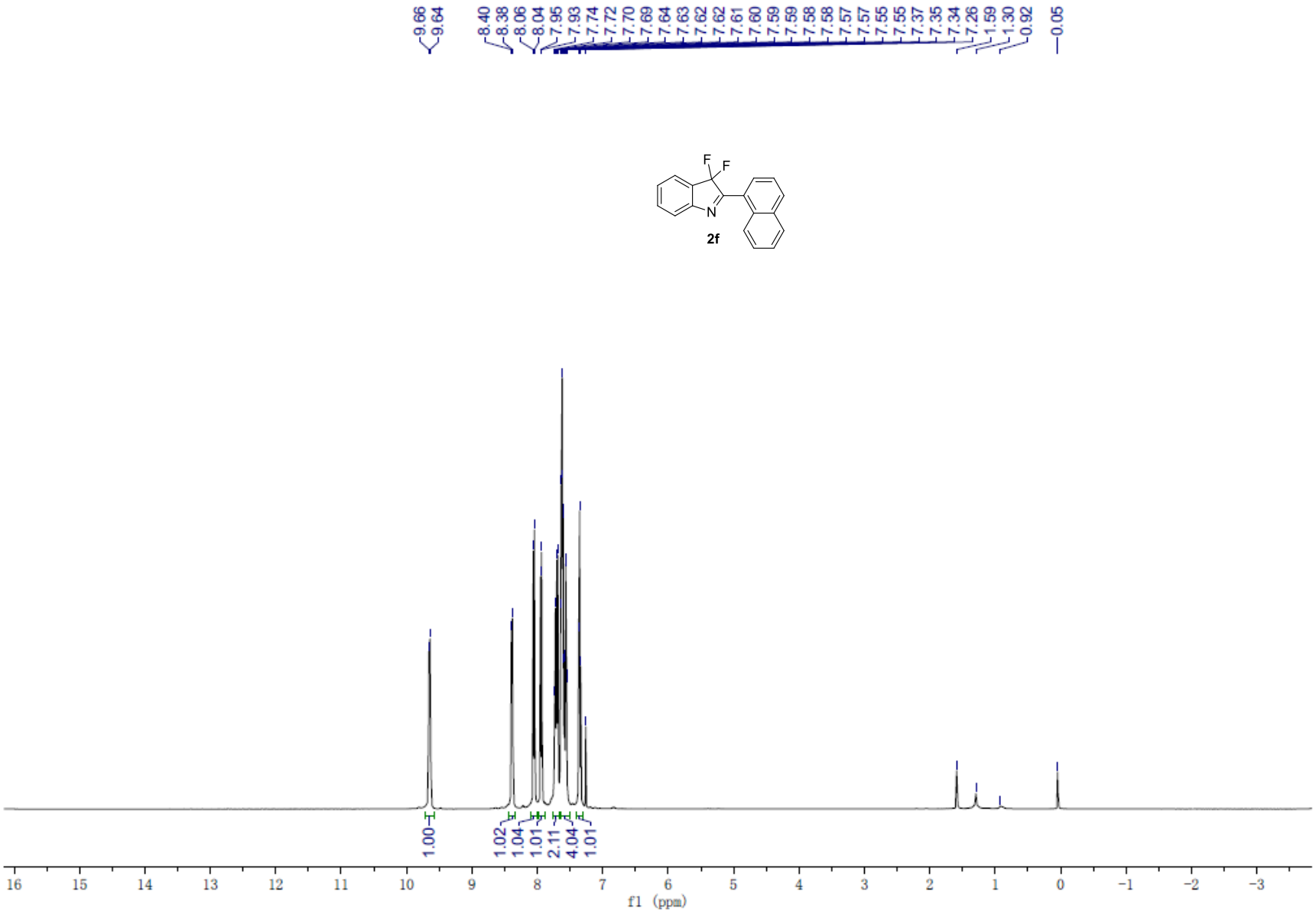
-170
-175
f1 (ppm)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7.37
7.35
7.34
7.26
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1.30
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0.05
F
F
N
2f
1.00
1.02
1.04
1.01
2.11
4.04
1.01
16
15
14
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5
4
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1
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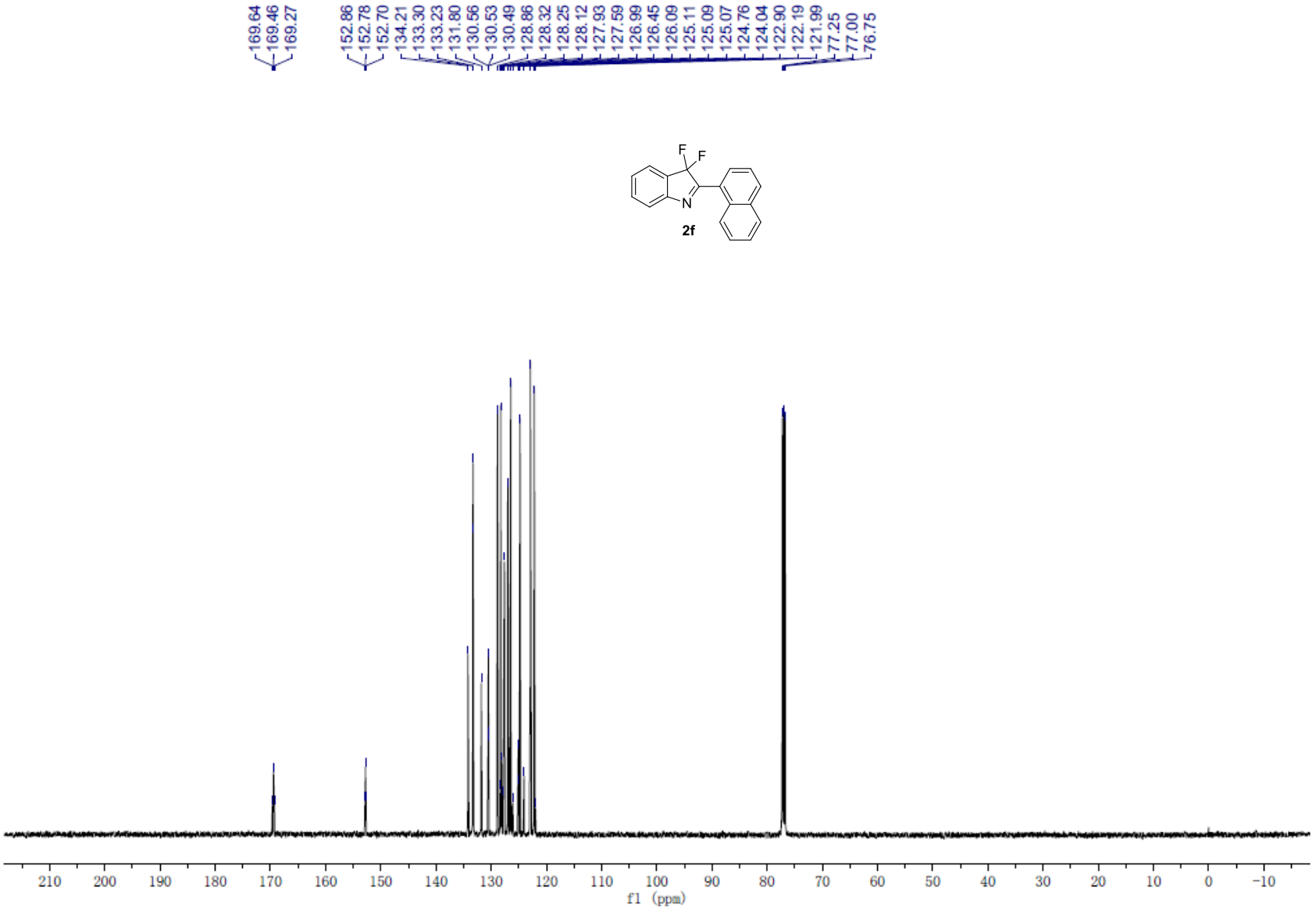
f1 (ppm)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126.99
126.45
126.09
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125.07
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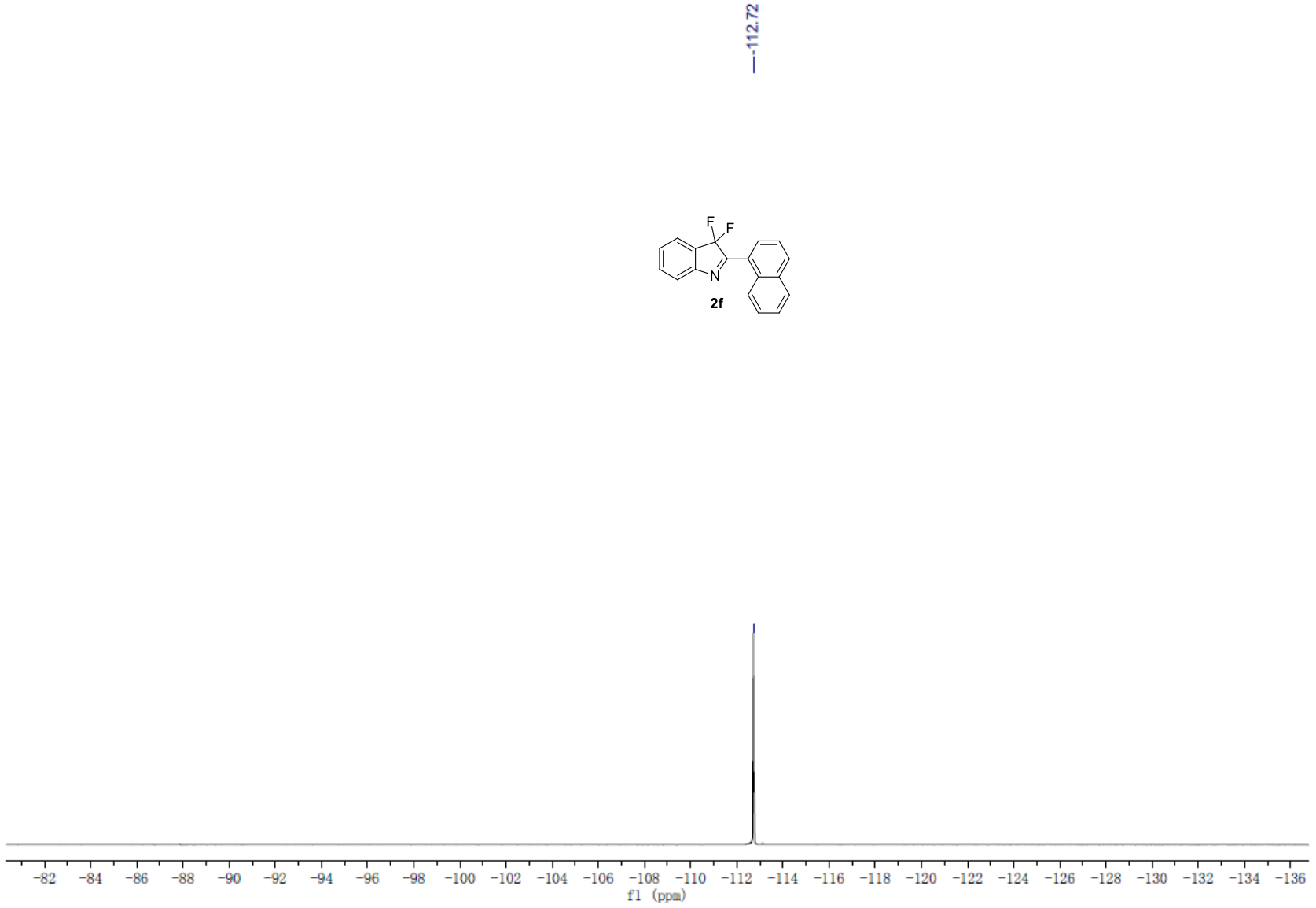
f1 (ppm)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F
N
2f
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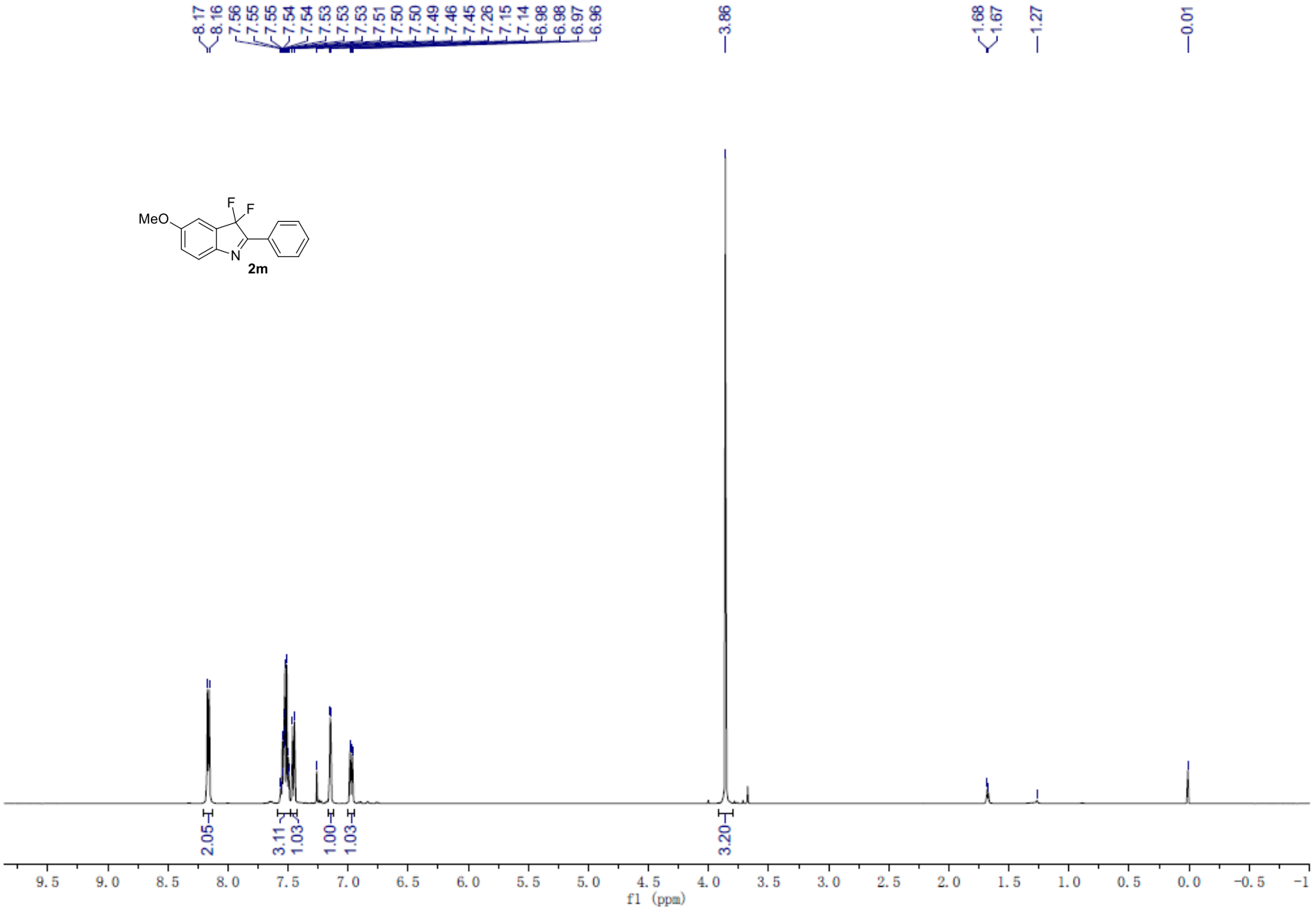
f1 (ppm)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6.98
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MeO
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N
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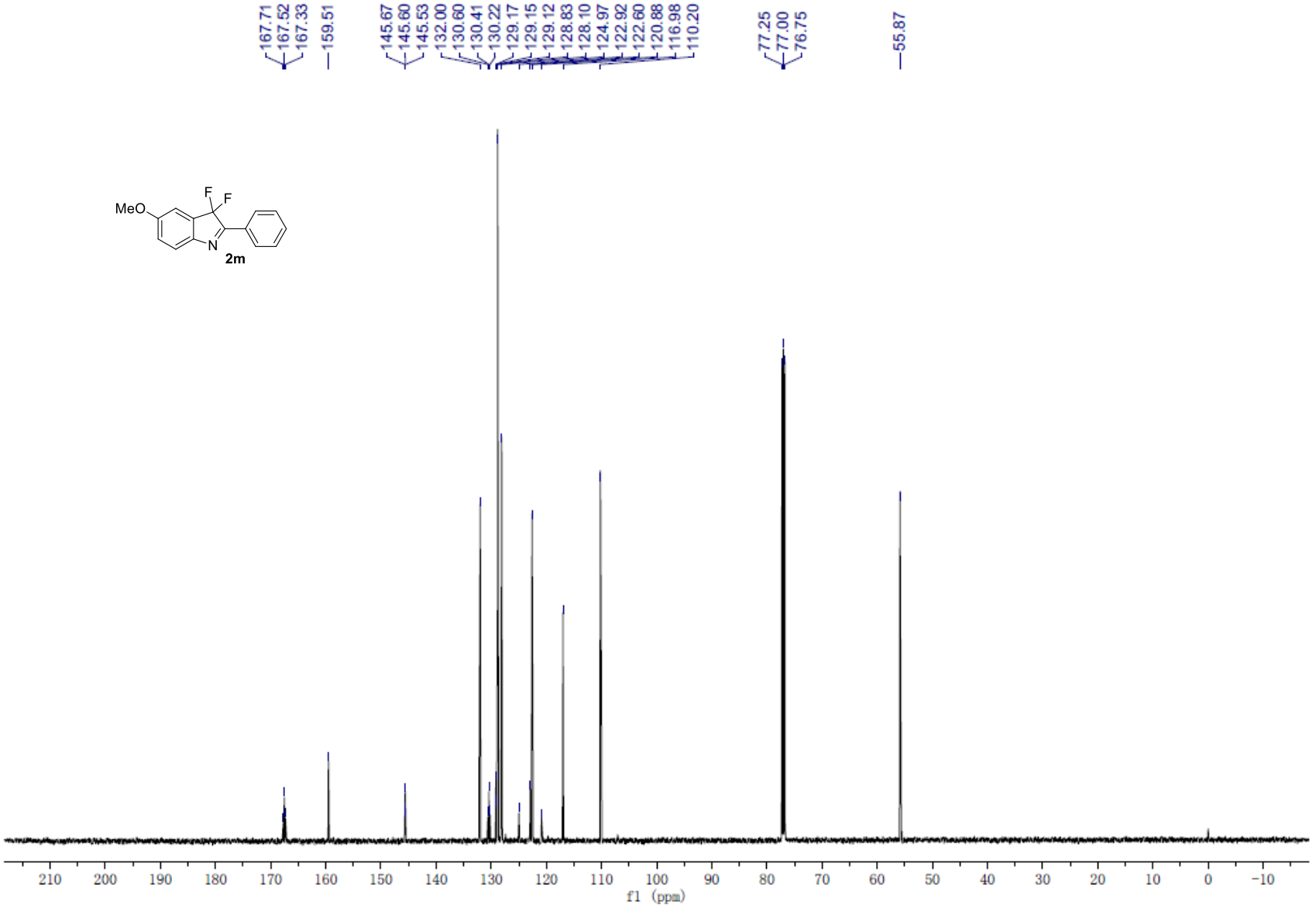
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128.83
128.10
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122.92
122.60
120.88
116.98
110.20
77.25
77.00
76.75
55.87
MeO
F
F
N
2m
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200
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180
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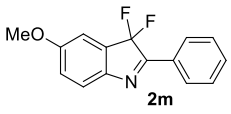
f1 (ppm)


F
F
MeO
N

2m

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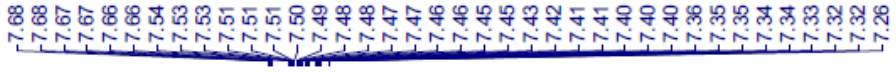
f1 (ppm)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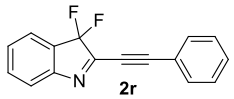
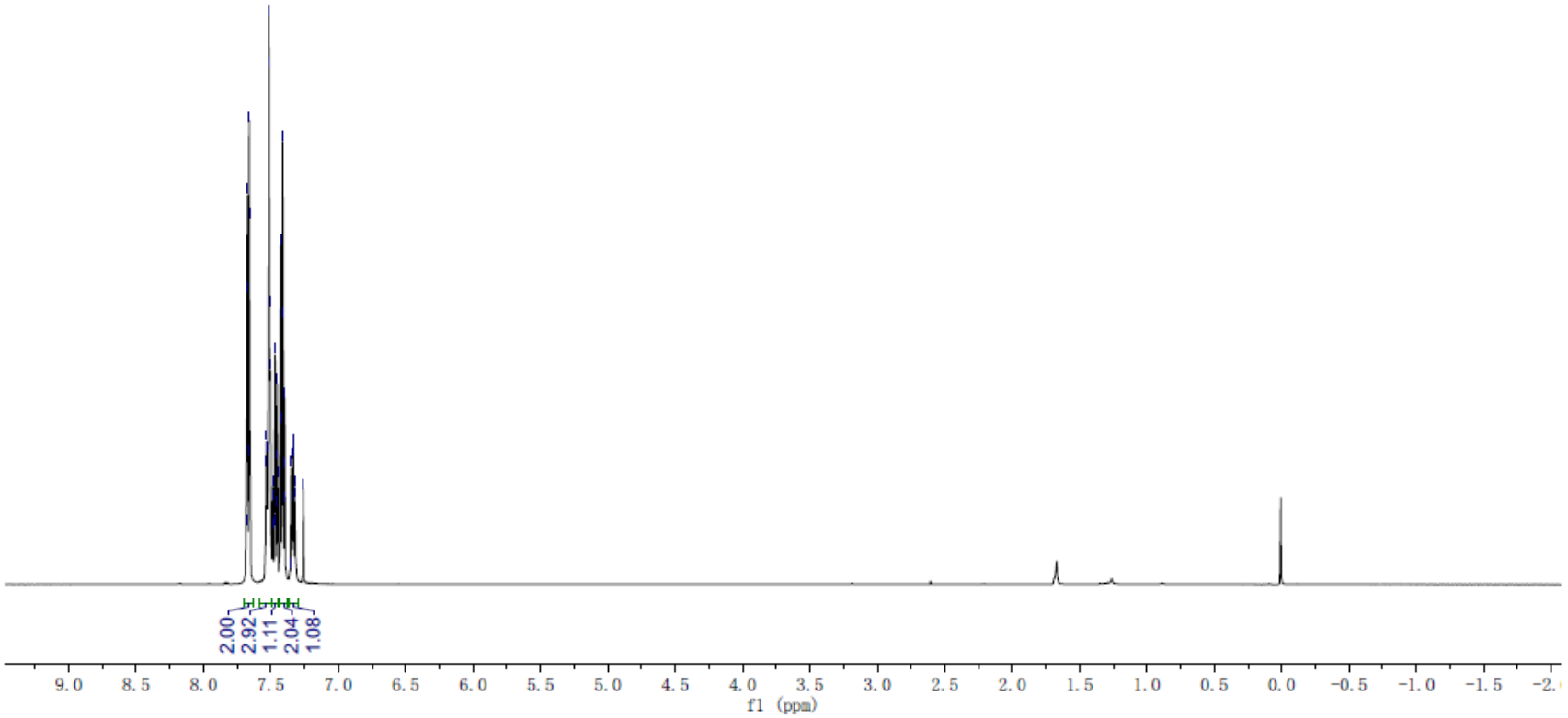
7.26


F
F
N

2r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4.0
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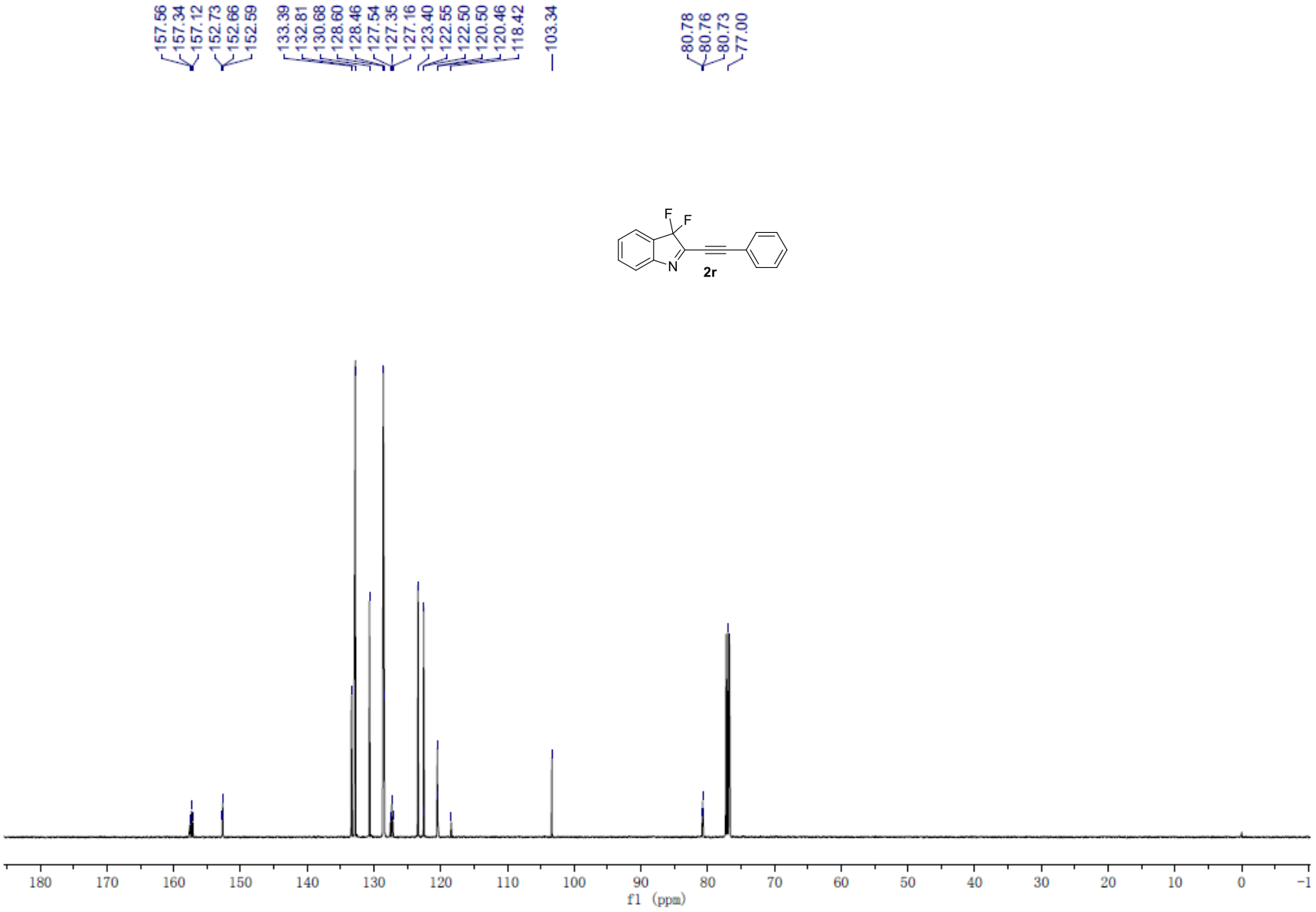
f1 (ppm)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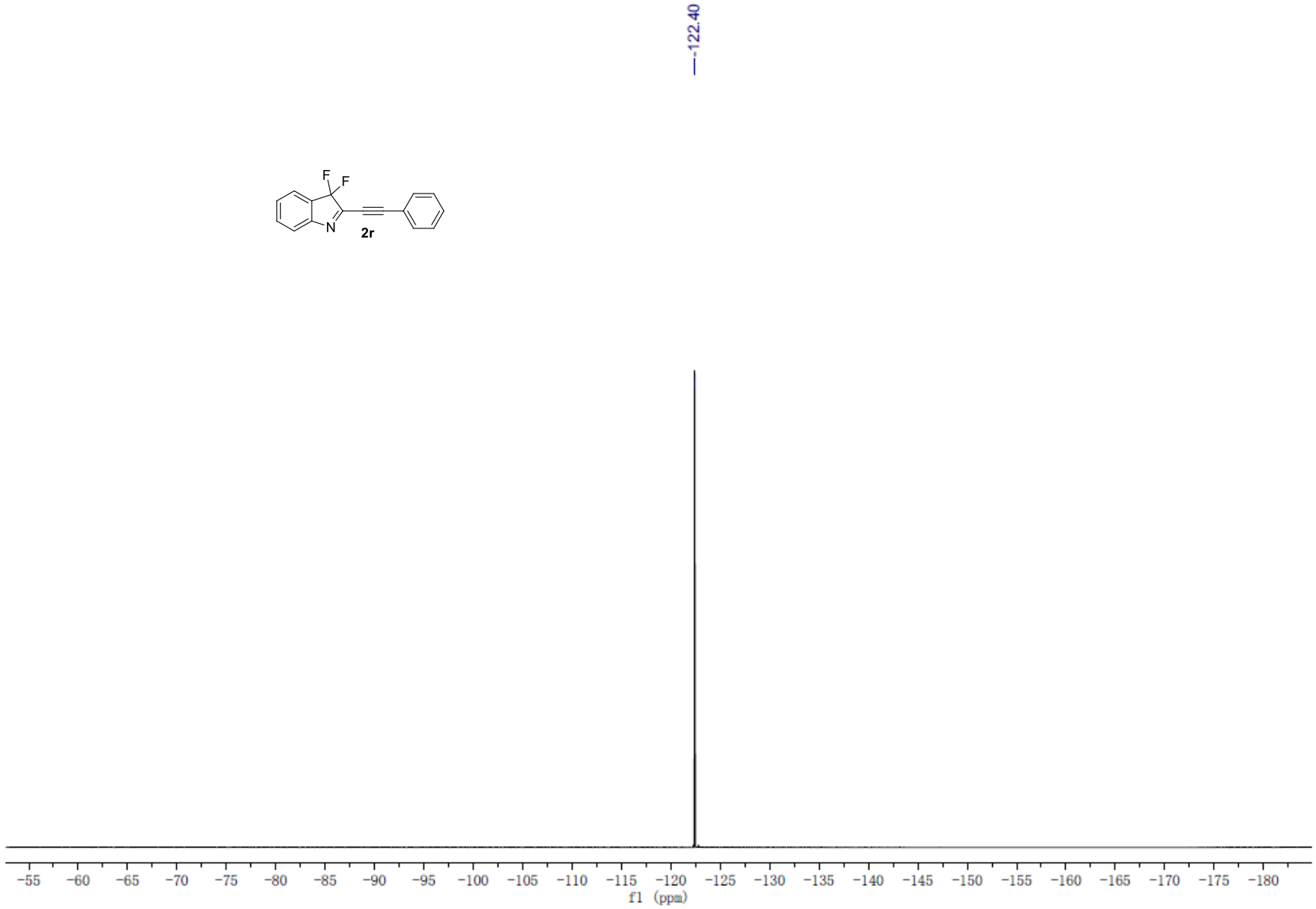
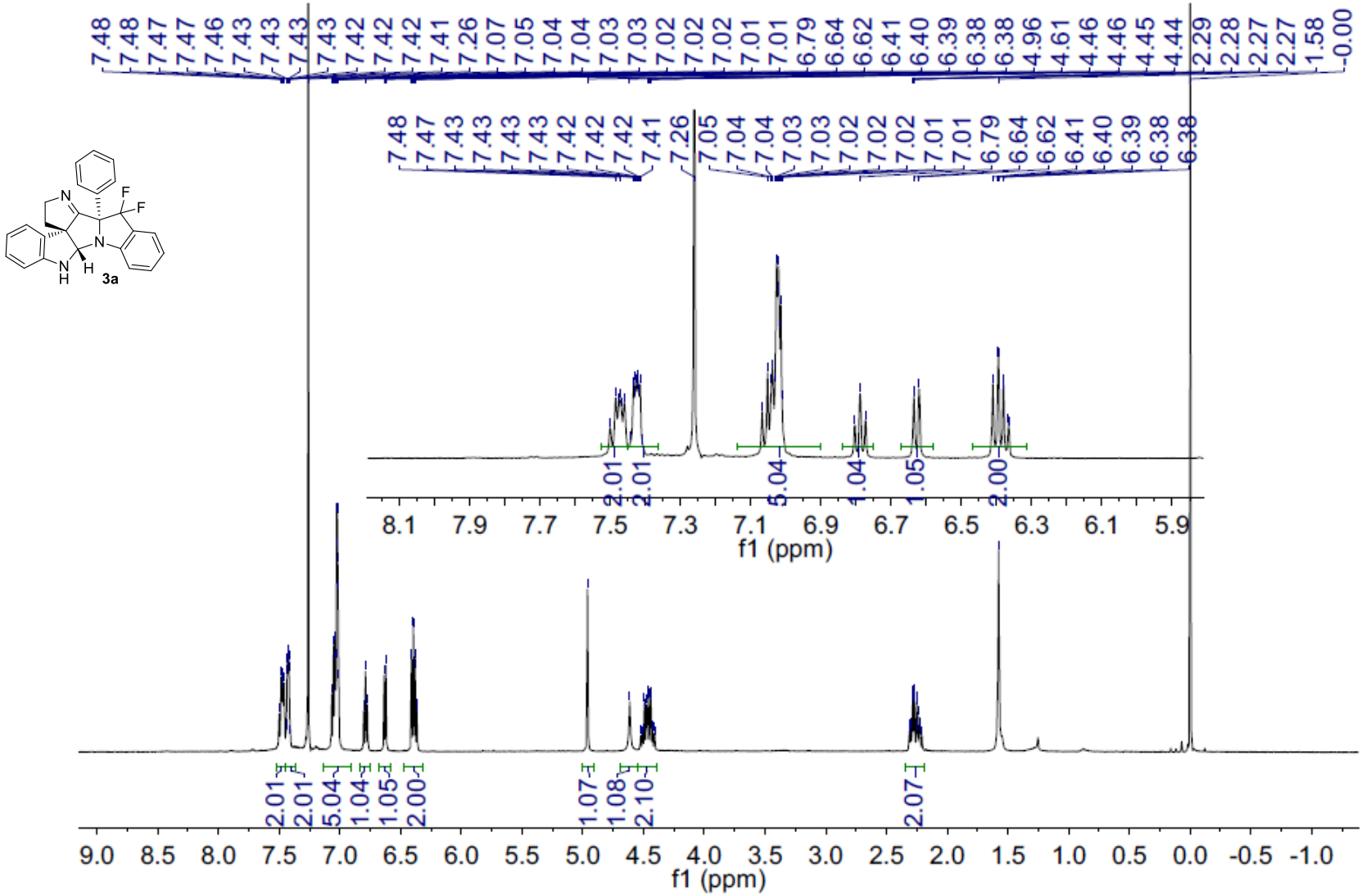
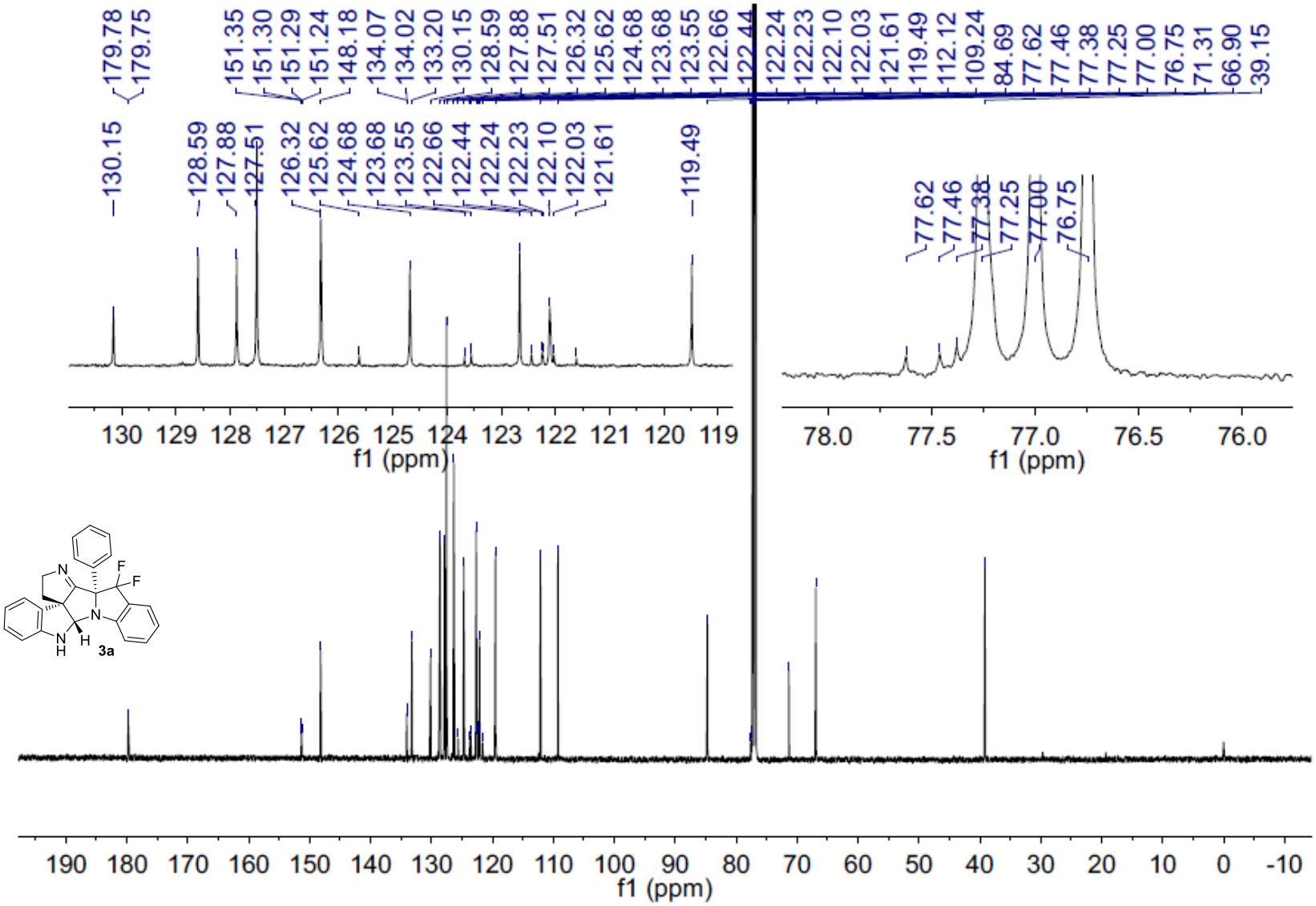
127.54
127.35
127.16
123.40
122.55
122.50
120.50
120.46
118.42
103.34
80.78
80.76
80.73
77.00
F
F
N
2r
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−1
f1 (ppm)


-122.40
F
F
N
2r
-55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155 -160 -165 -170 -175 -180
f1 (ppm)


7.48 7.48 7.47 7.47 7.46 7.43 7.43 7.43 7.43 7.42 7.42 7.42 7.41 7.26 7.07 7.05 7.04 7.04 7.03 7.03 7.02 7.02 7.02 7.01 7.01 6.79 6.64 6.62 6.41 6.40 6.39 6.38 6.38 4.96 4.61 4.46 4.46 4.45 4.44 2.29 2.28 2.27 2.27 1.58 -0.00
F
F
N
N
H
H
3a
2.01 2.01 5.04 1.04 1.05 2.00 1.07 1.08 2.10 2.07
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)
8.1 7.9 7.7 7.5 7.3 7.1 6.9 6.7 6.5 6.3 6.1 5.9
f1 (ppm)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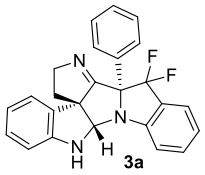
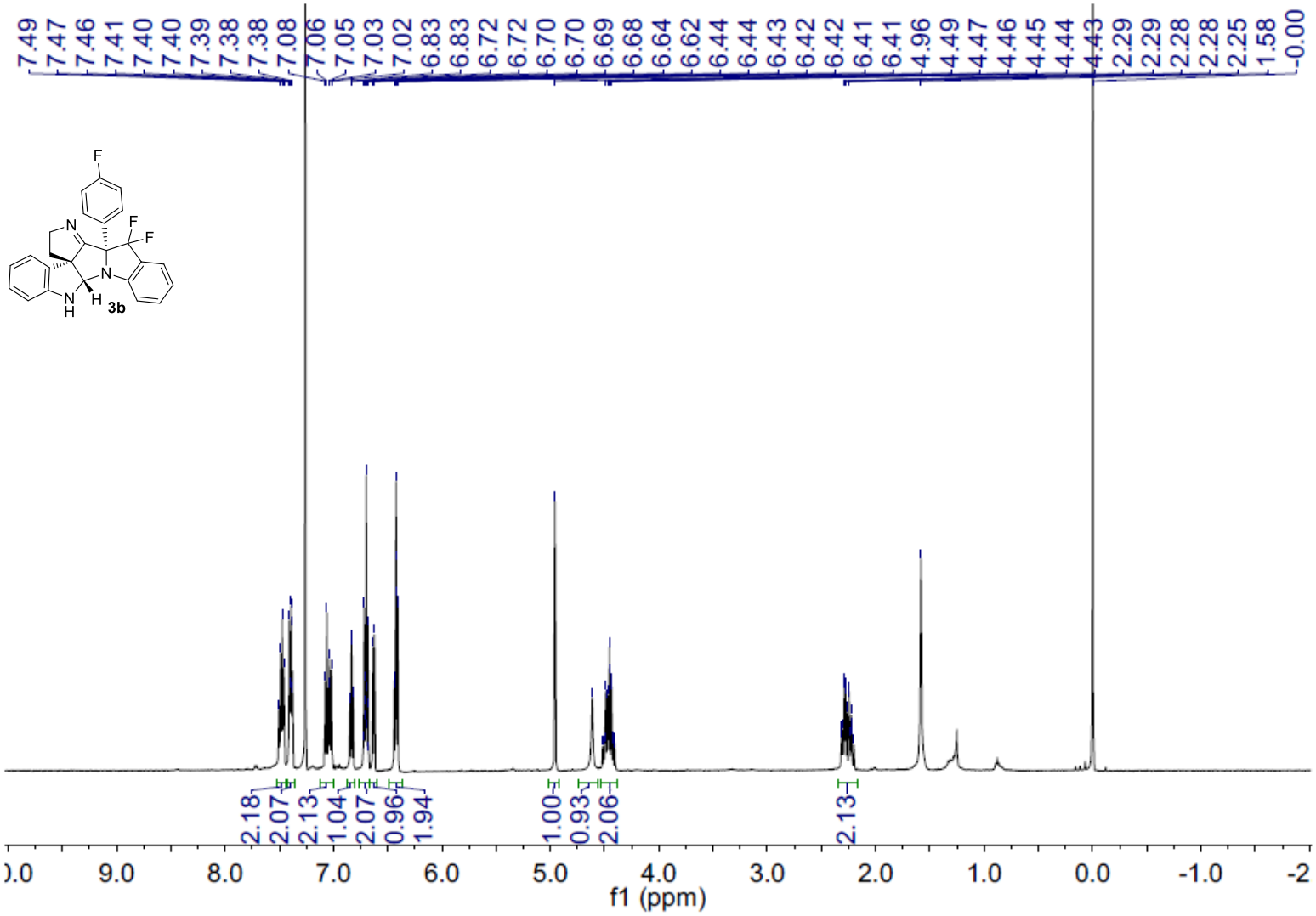
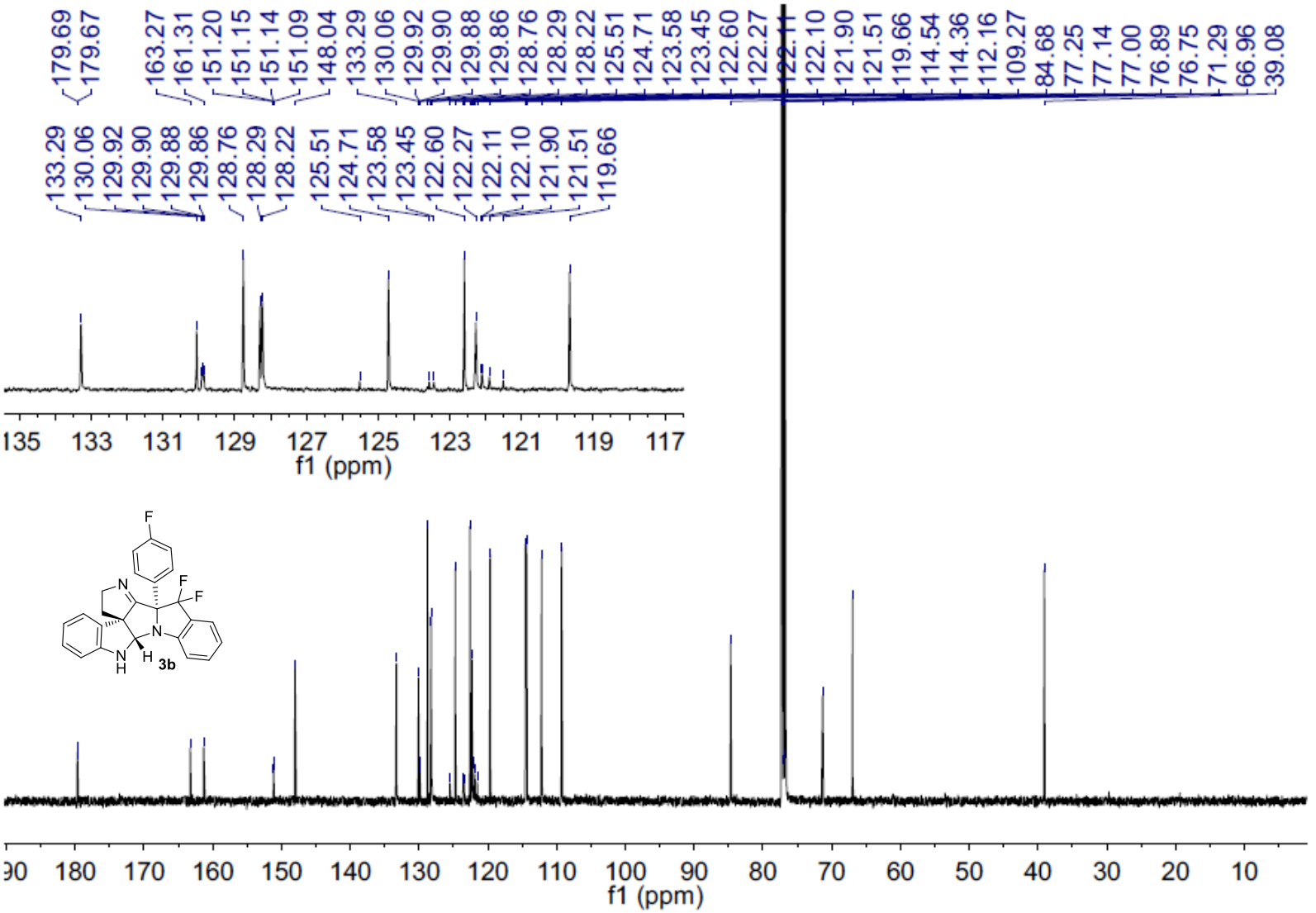
179.75
151.35
151.30
151.29
151.24
148.18
134.07
134.02
133.20
130.15
128.59
127.88
127.51
126.32
125.62
124.68
123.68
123.55
122.66
122.44
122.24
122.23
122.10
122.03
121.61
119.49
112.12
109.24
84.69
77.62
77.46
77.38
77.25
77.00
76.75
71.31
66.90
39.15
f1 (ppm)
f1 (ppm)
f1 (ppm)
3a

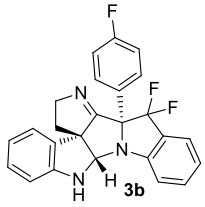

N
F
F
N
N
H
H

3a

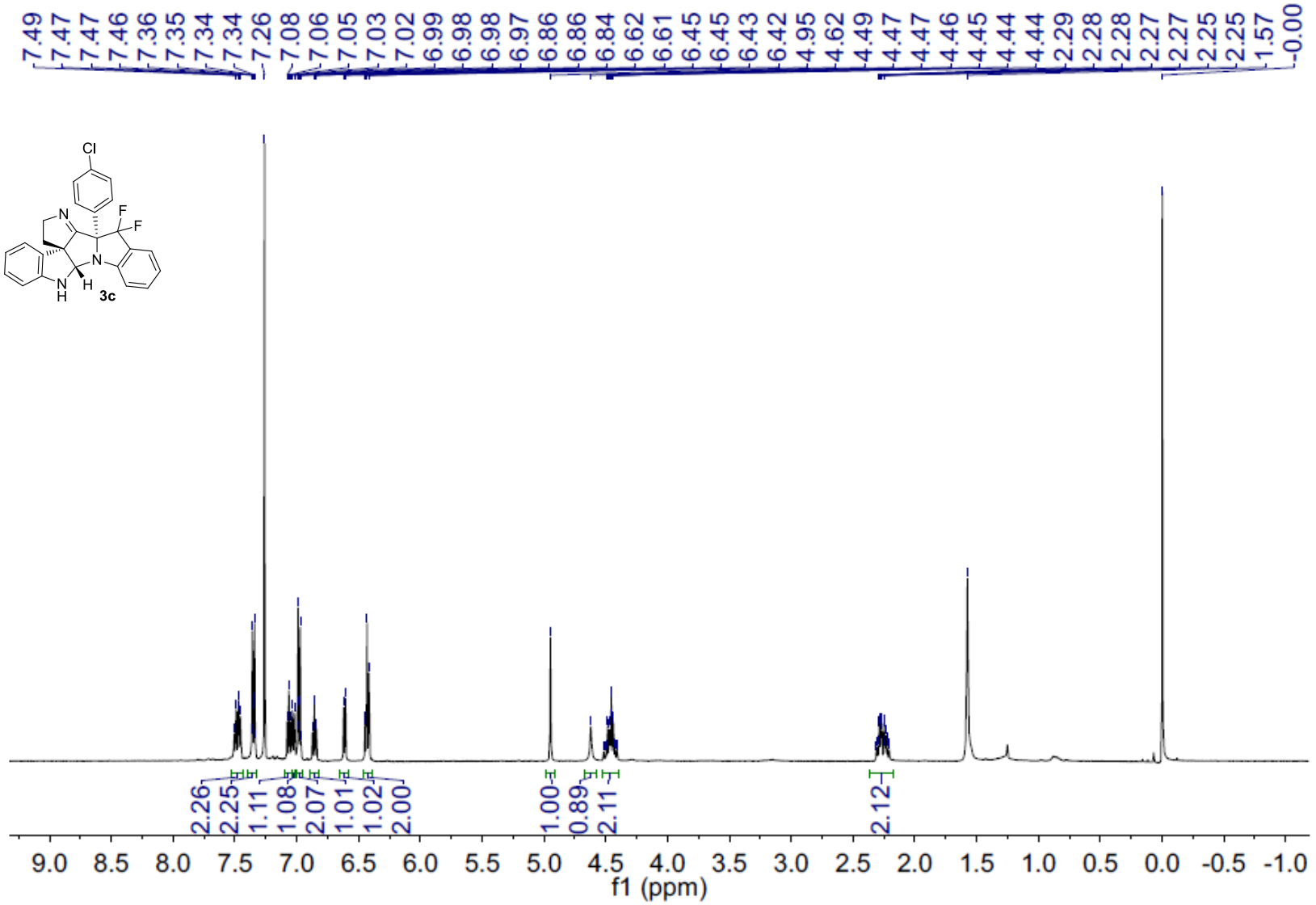
-82.08
-82.62
-95.99
-96.53
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)

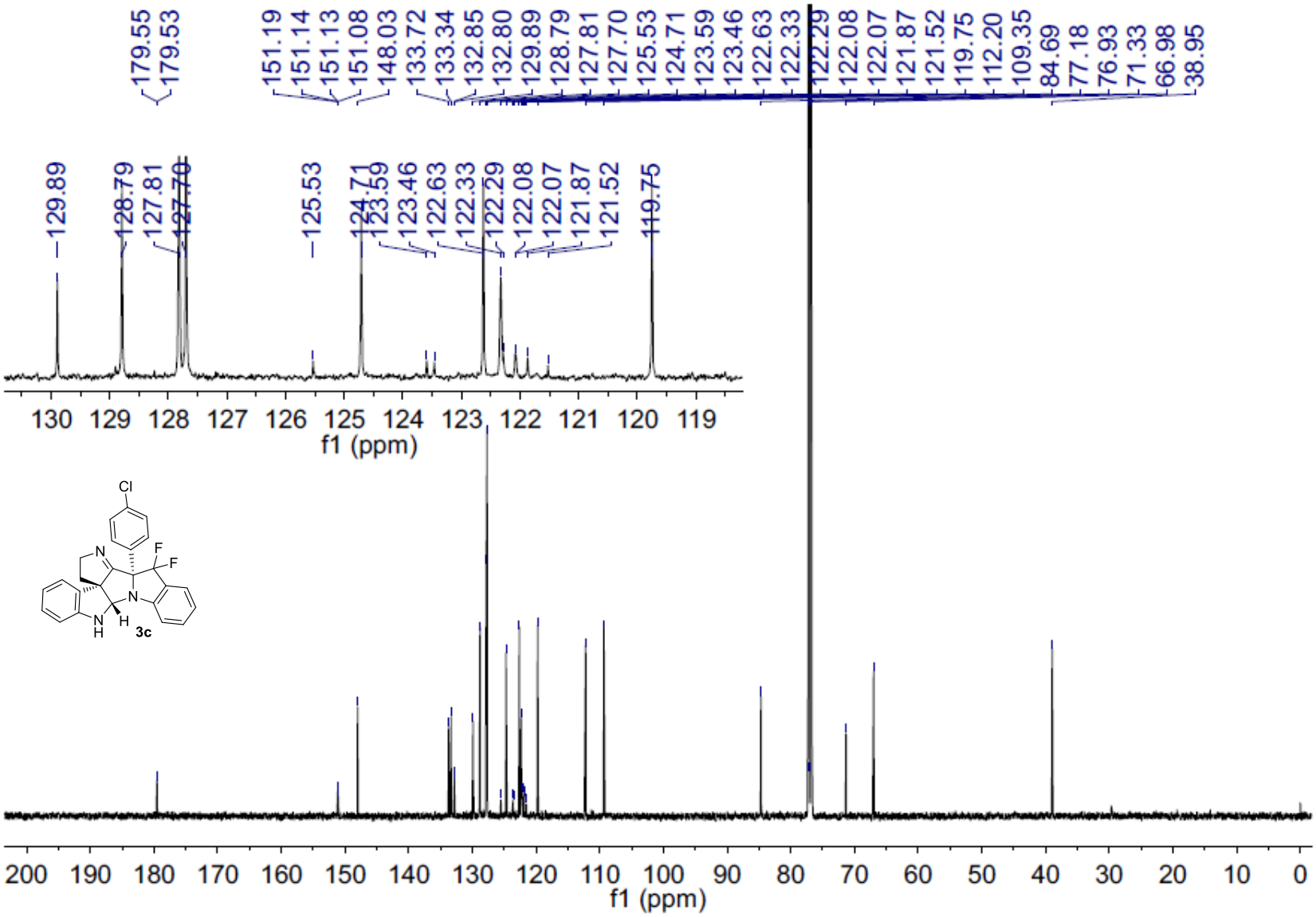

7.49
7.47
7.46
7.41
7.40
7.40
7.39
7.38
7.38
7.08
7.06
7.05
7.03
7.02
6.83
6.83
6.72
6.72
6.70
6.70
6.69
6.68
6.64
6.62
6.44
6.44
6.43
6.42
6.42
6.41
6.41
4.96
4.49
4.47
4.46
4.45
4.44
4.43
2.29
2.29
2.28
2.28
2.25
1.58
0.00
F
N
F
F
N
N
H
H
3b
2.18
2.07
2.13
1.04
2.07
0.96
1.94
1.00
0.93
2.06
2.13
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2
f1 (ppm)

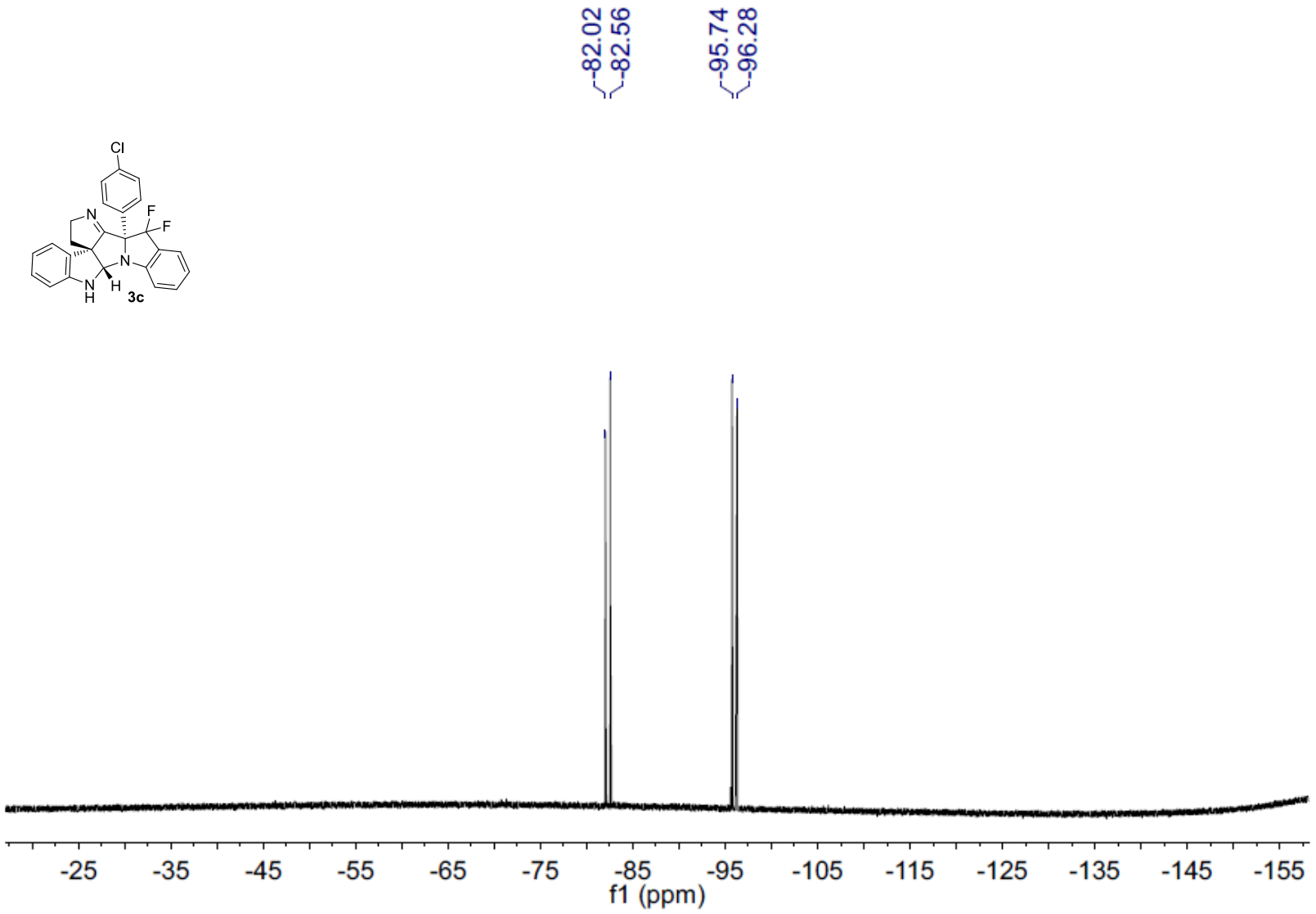

179.69
179.67
163.27
161.31
151.20
151.15
151.14
151.09
148.04
133.29
130.06
129.92
129.90
129.88
129.86
128.76
128.29
128.22
125.51
124.71
123.58
123.45
122.60
122.27
122.11
122.10
121.90
121.51
119.66
114.54
114.36
112.16
109.27
84.68
77.25
77.14
77.00
76.89
76.75
71.29
66.96
39.08
f1 (ppm)
F
N
F
F
N
H
N
H
3b

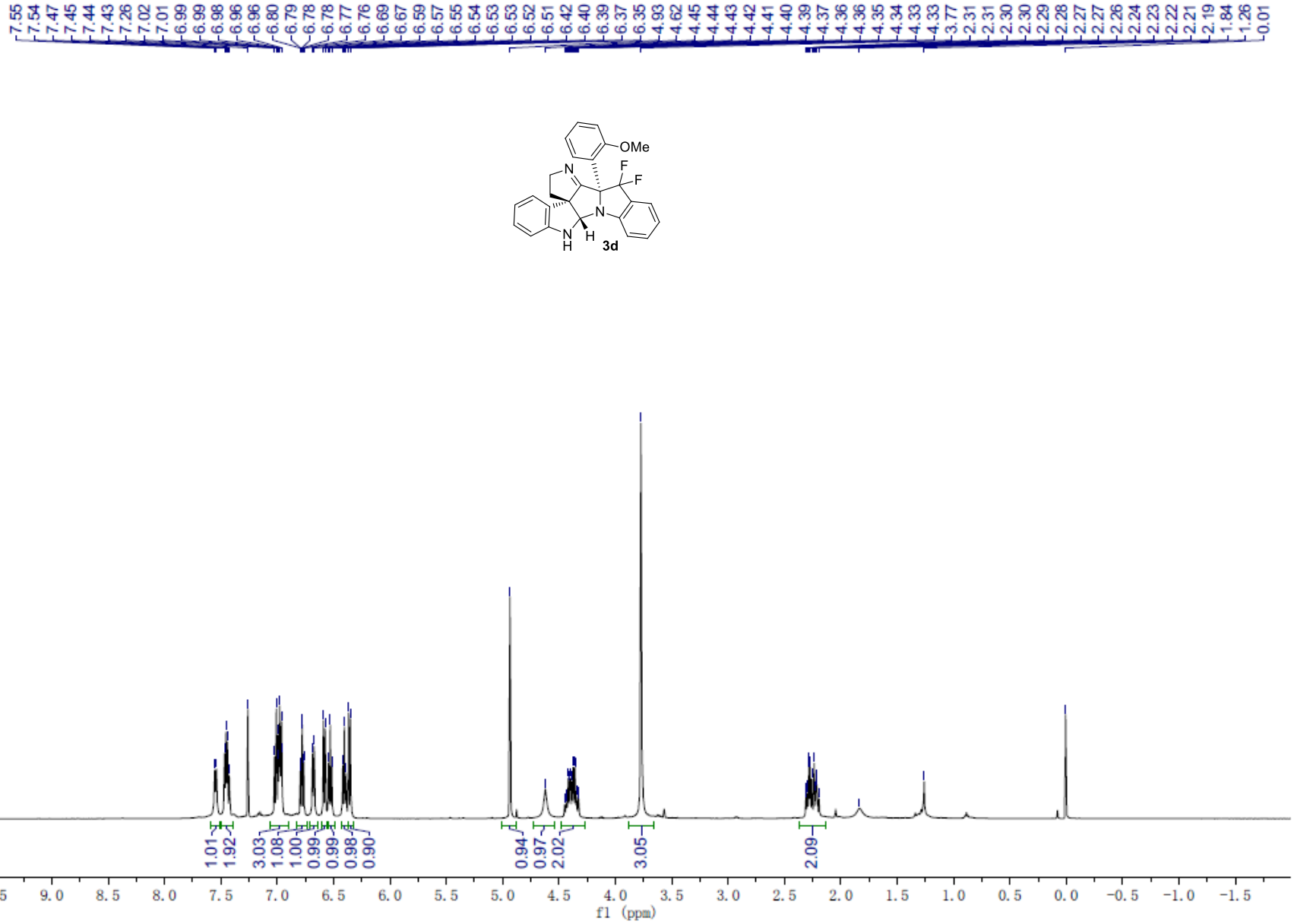

F
N
F
F
N
N
H
H
3b

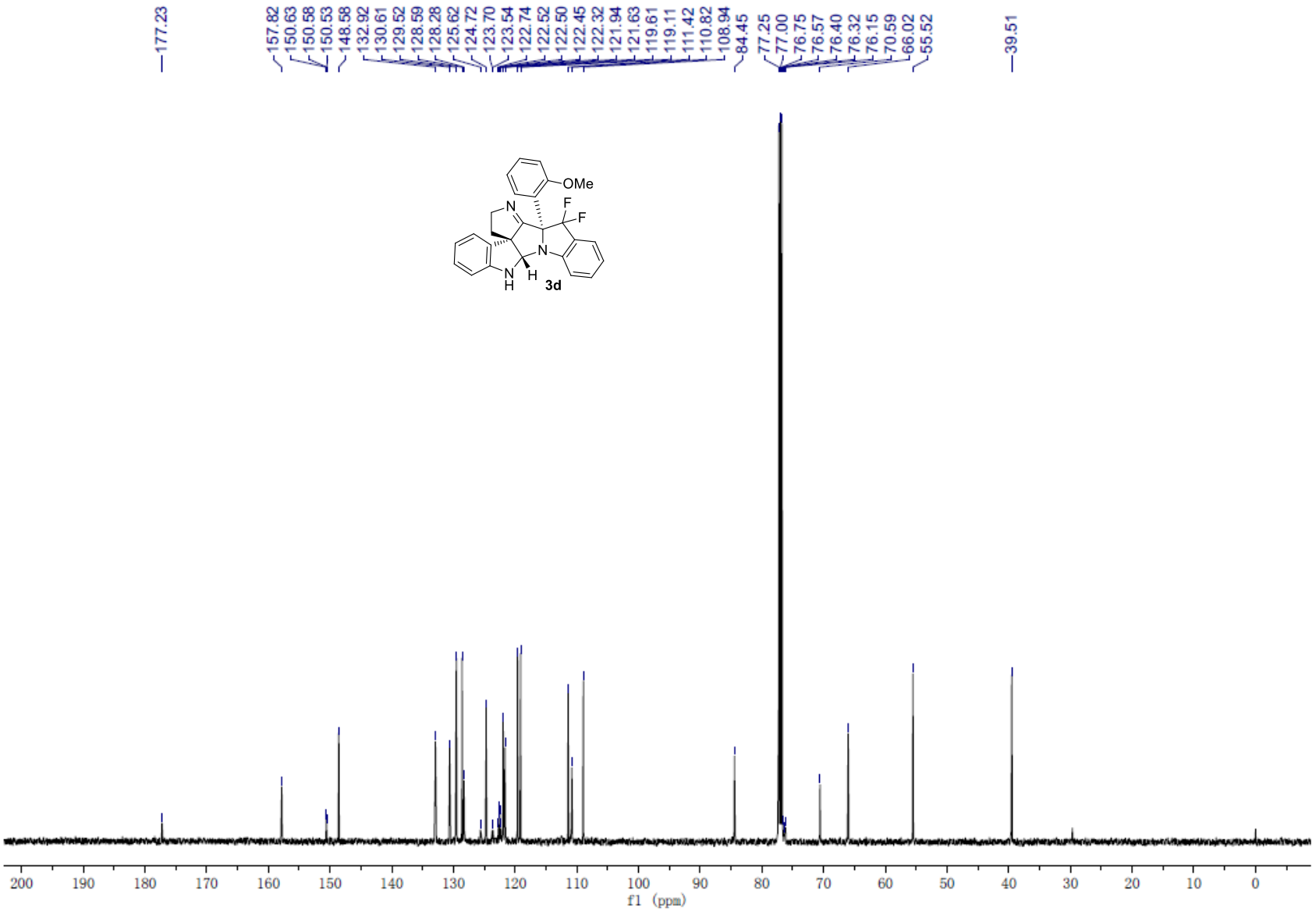
-81.90
-82.44
-96.42
-96.95
-114.52
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

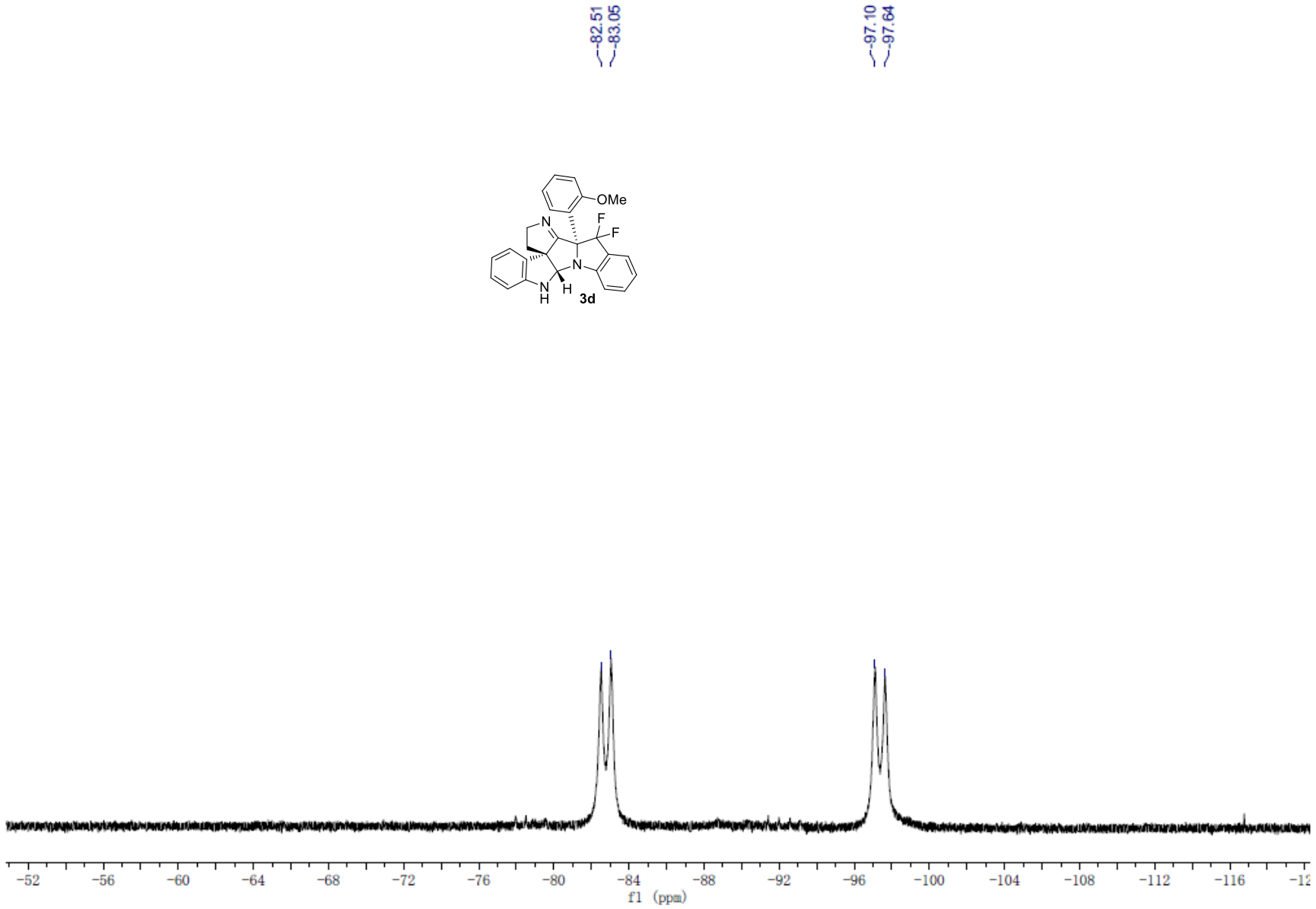

7.49
7.47
7.47
7.46
7.36
7.35
7.34
7.34
7.26
7.08
7.06
7.05
7.03
7.02
6.99
6.98
6.98
6.97
6.86
6.86
6.84
6.62
6.61
6.45
6.45
6.43
6.42
4.95
4.62
4.49
4.47
4.47
4.46
4.45
4.44
4.44
2.29
2.28
2.28
2.27
2.27
2.25
2.25
1.57
-0.00
Cl
F
F
N
N
H
N
H
3c
2.26
2.25
1.11
1.08
2.07
1.01
1.02
2.00
1.00
0.89
2.11
2.12
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

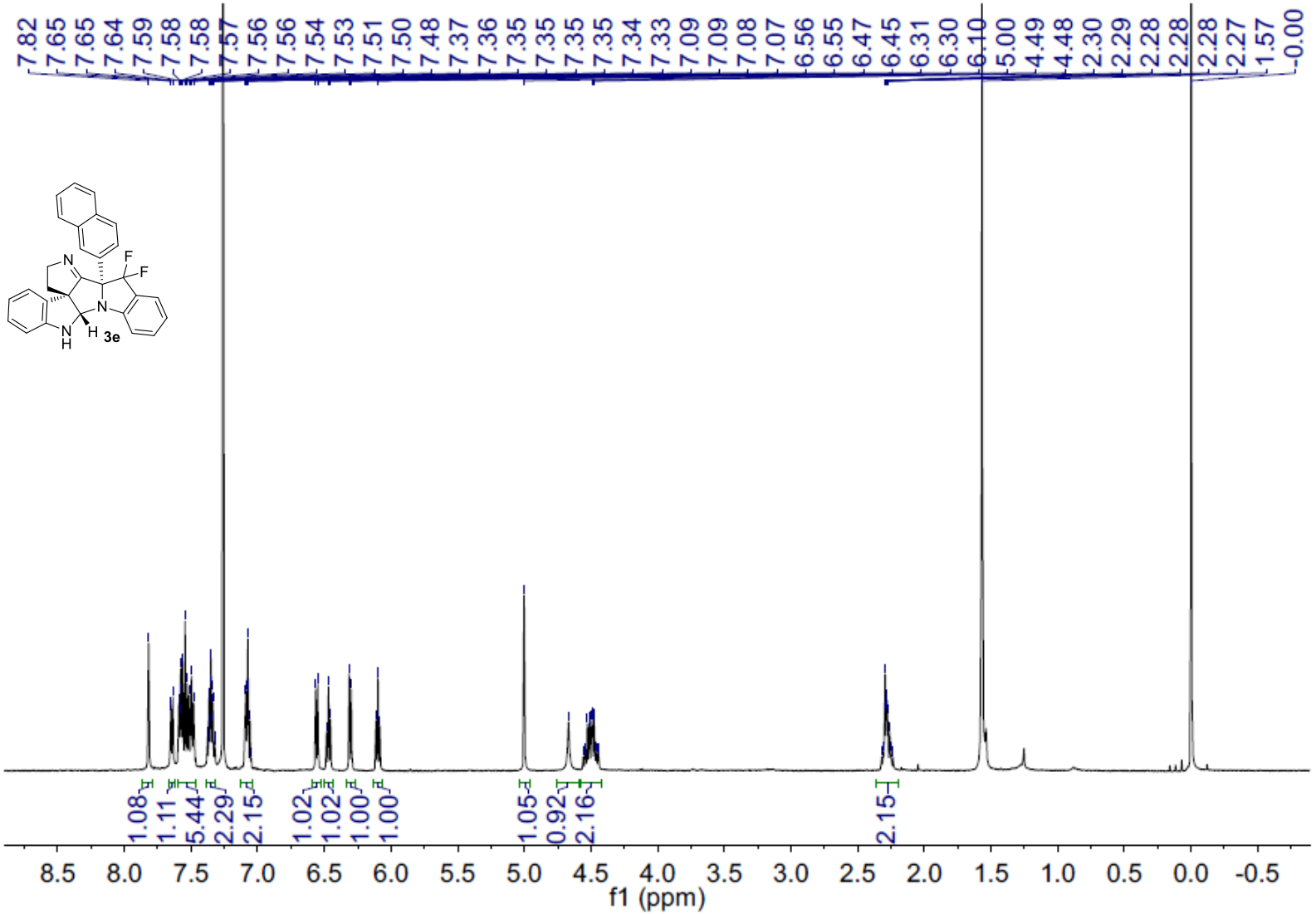

179.55
179.53
151.19
151.14
151.13
151.08
148.03
133.72
133.34
132.85
132.80
129.89
128.79
127.81
127.70
125.53
124.71
123.59
123.46
122.63
122.33
122.29
122.08
122.07
121.87
121.52
119.75
112.20
109.35
84.69
77.18
76.93
71.33
66.98
38.95
f1 (ppm)
Cl
F
F
N
N
H
N
H
3c

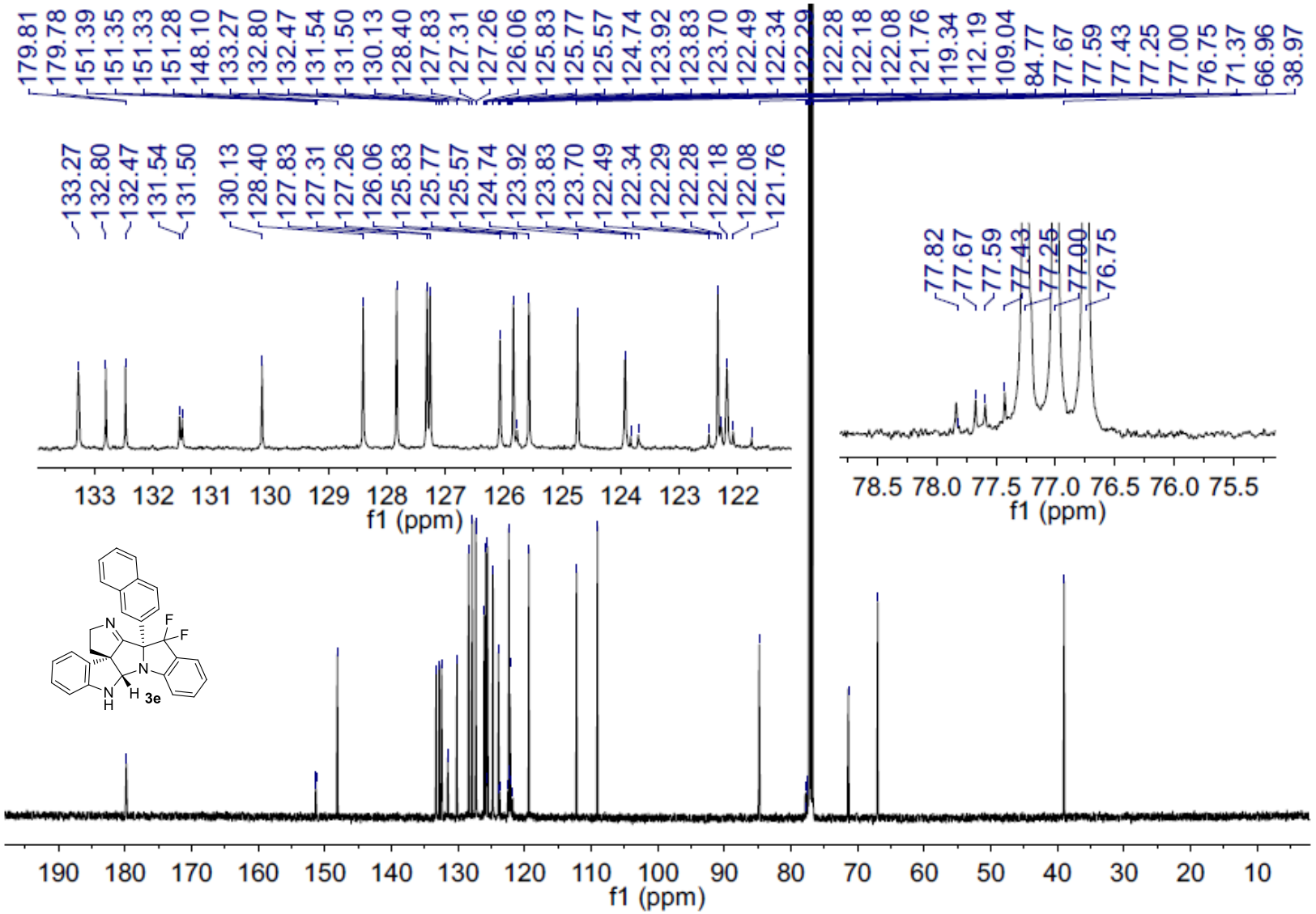

-82.02
-82.56
-95.74
-96.28
Cl
F
F
N
N
H
N
H
3c
-25
-35
-45
-55
-65
-75
-85
-95
-105
-115
-125
-135
-145
-155
f1 (ppm)


7.55 7.54 7.47 7.45 7.44 7.43 7.26 7.02 7.01 6.99 6.99 6.98 6.96 6.96 6.80 6.79 6.78 6.78 6.77 6.76 6.69 6.67 6.59 6.57 6.55 6.54 6.53 6.53 6.52 6.51 6.42 6.40 6.39 6.37 6.35 4.93 4.62 4.45 4.44 4.43 4.42 4.41 4.40 4.39 4.37 4.36 4.36 4.35 4.34 4.33 4.33 3.77 2.31 2.31 2.30 2.30 2.29 2.28 2.27 2.27 2.26 2.24 2.23 2.22 2.21 2.19 1.84 1.26 0.01
OMe
F
F
N
N
H
N
H
3d
1.01 1.92 3.03 1.08 1.00 0.99 0.99 0.98 0.90 0.94 0.97 2.02 3.05 2.09
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


177.23
157.82
150.63
150.58
150.53
148.58
132.92
130.61
129.52
128.59
128.28
125.62
124.72
123.70
123.54
122.74
122.52
122.50
122.45
122.32
121.94
121.63
119.61
119.11
111.42
110.82
108.94
84.45
77.25
77.00
76.75
76.57
76.40
76.32
76.15
70.59
66.02
55.52
39.51
OMe
F
F
N
N
H
N
H
3d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-82.51
-83.05
-97.10
-97.64
OMe
F
F
N
N
H
N
H
3d
-52
-56
-60
-64
-68
-72
-76
-80
-84
-88
-92
-96
-100
-104
-108
-112
-116
f1 (ppm)


7.82
7.65
7.65
7.64
7.59
7.58
7.58
7.57
7.56
7.56
7.54
7.53
7.51
7.50
7.48
7.37
7.36
7.35
7.35
7.35
7.35
7.34
7.33
7.09
7.09
7.08
7.07
6.56
6.55
6.47
6.45
6.31
6.30
6.10
5.00
4.49
4.48
2.30
2.29
2.28
2.28
2.28
2.27
1.57
0.00
N
F
F
N
N
H
H
3e
1.08
1.11
5.44
2.29
2.15
1.02
1.02
1.00
1.00
1.05
0.92
2.16
2.15
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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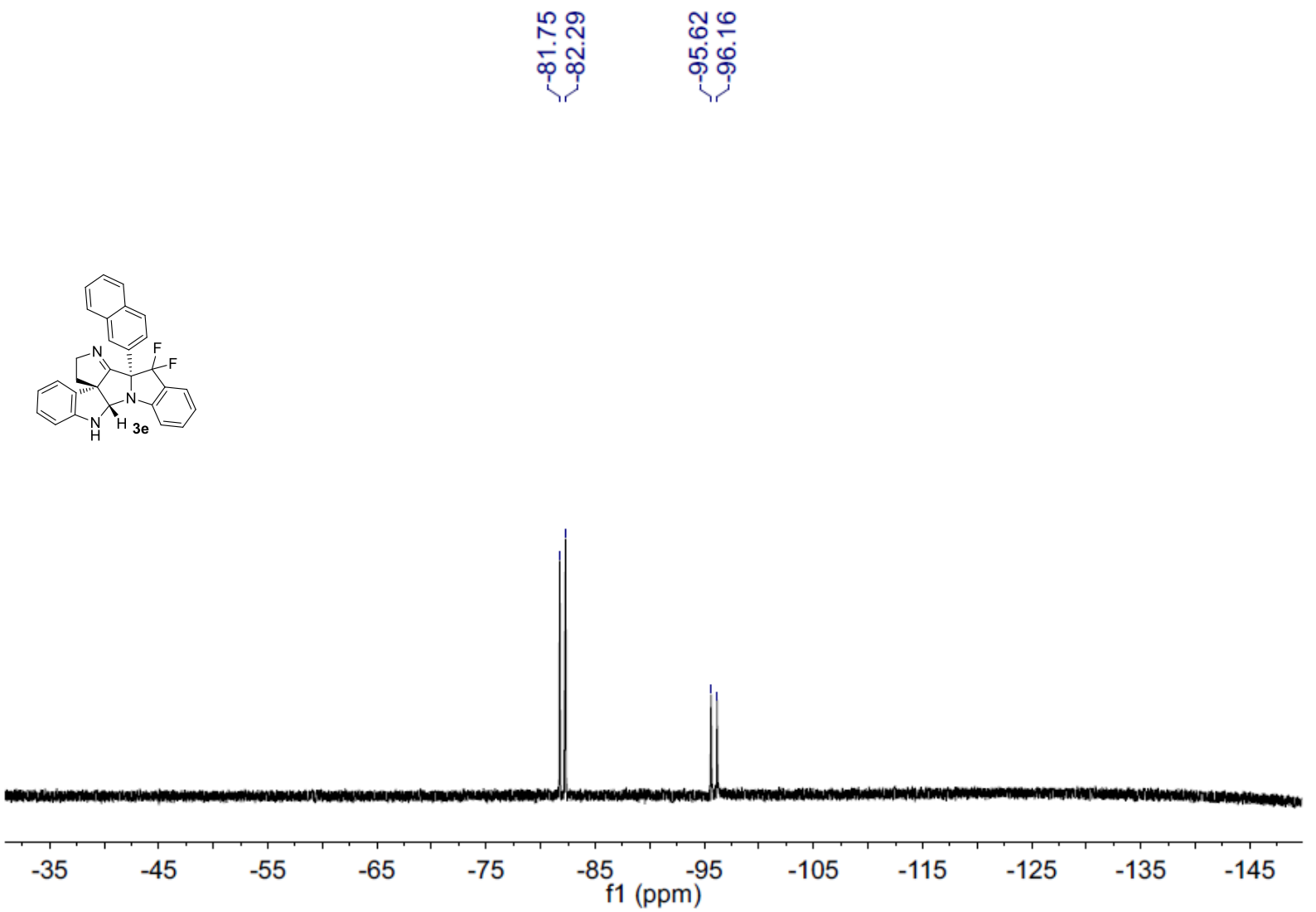
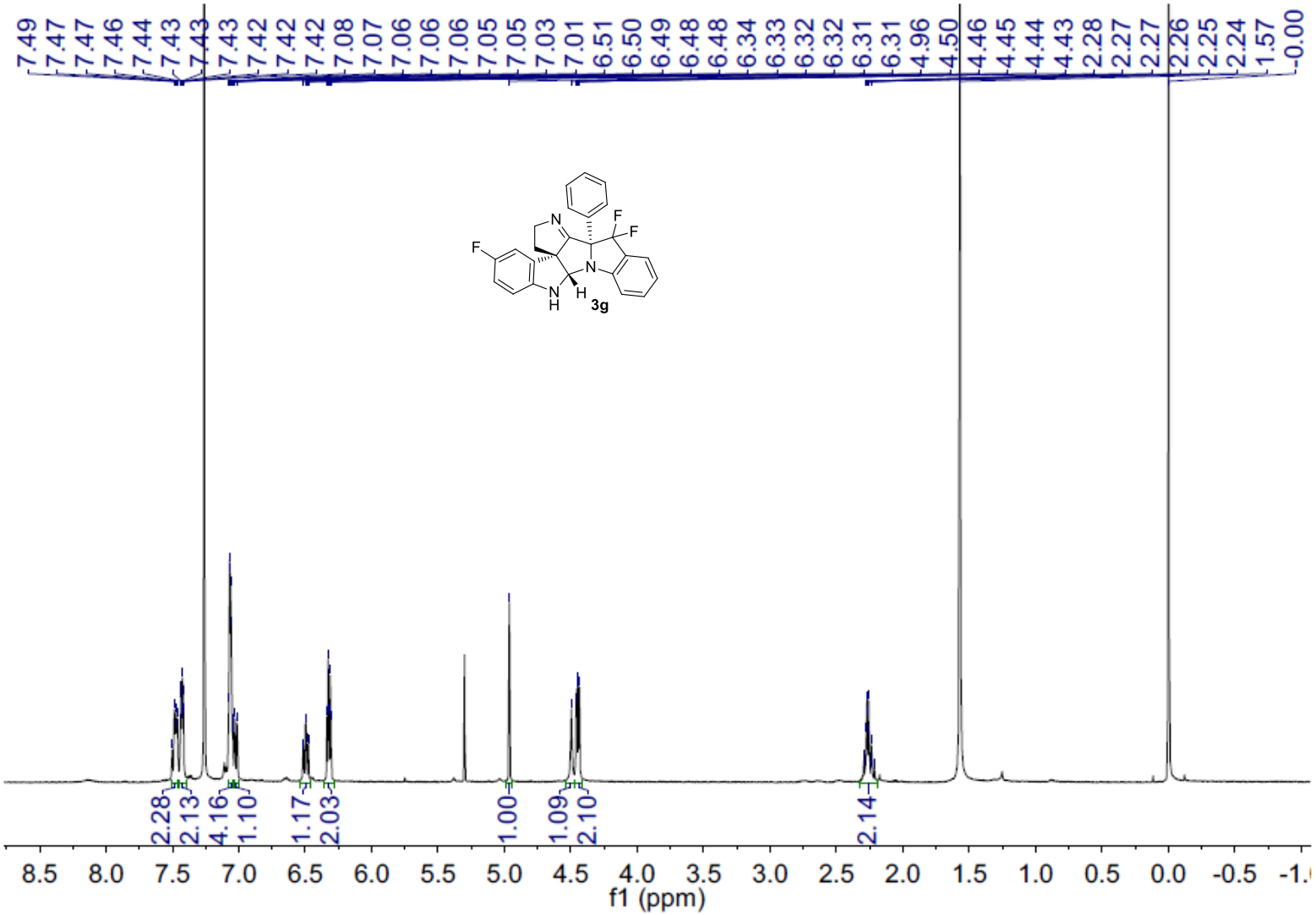
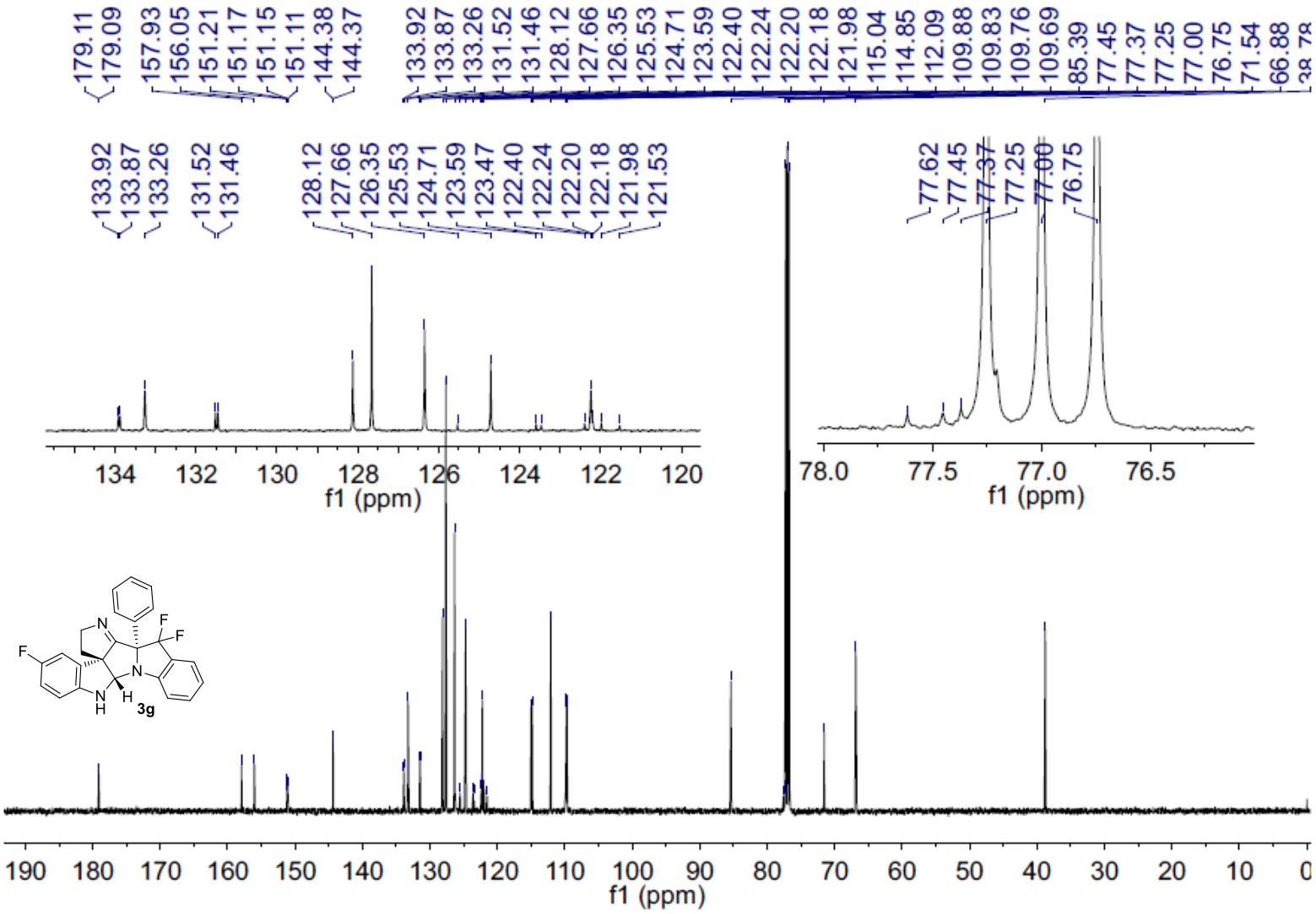
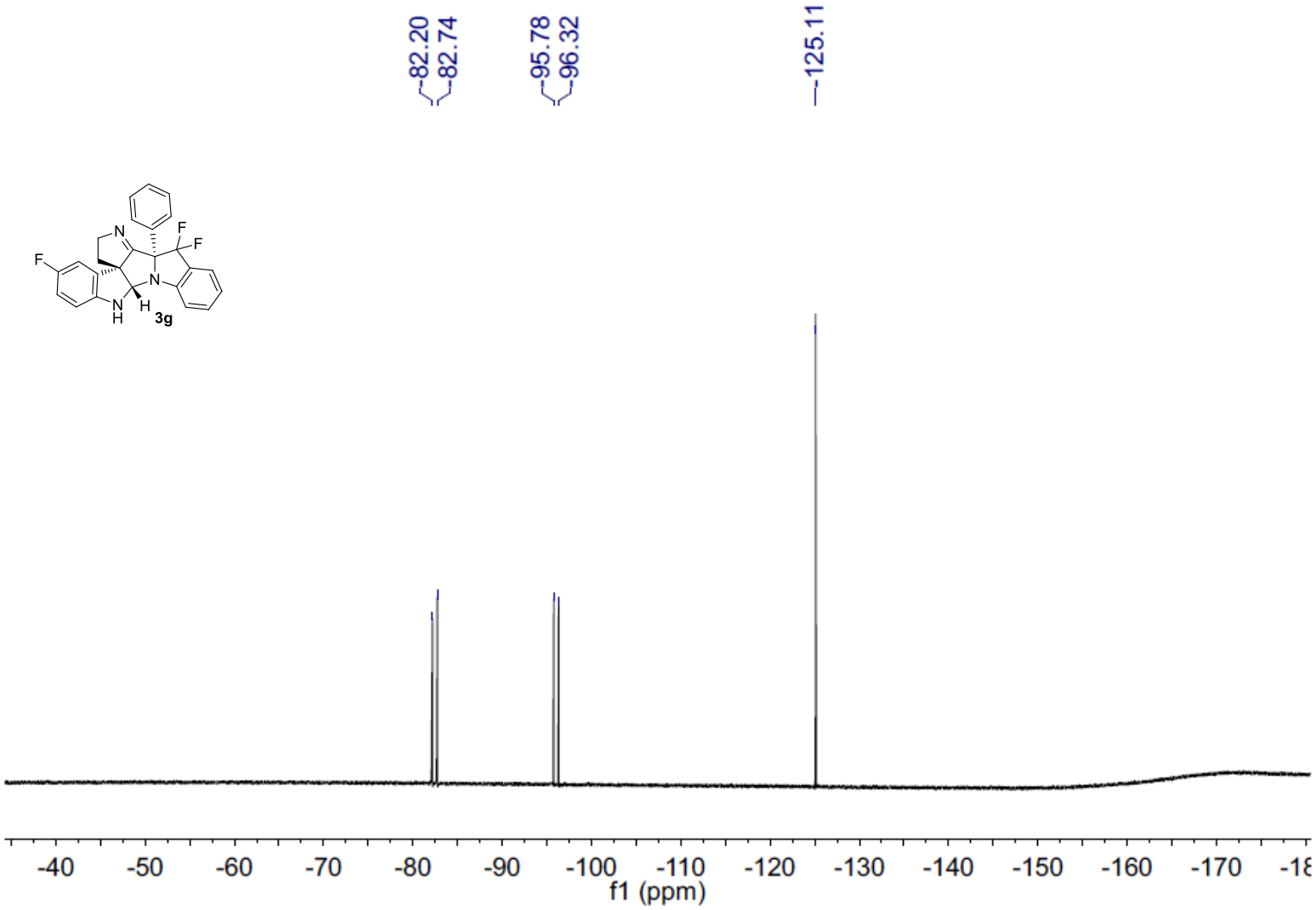
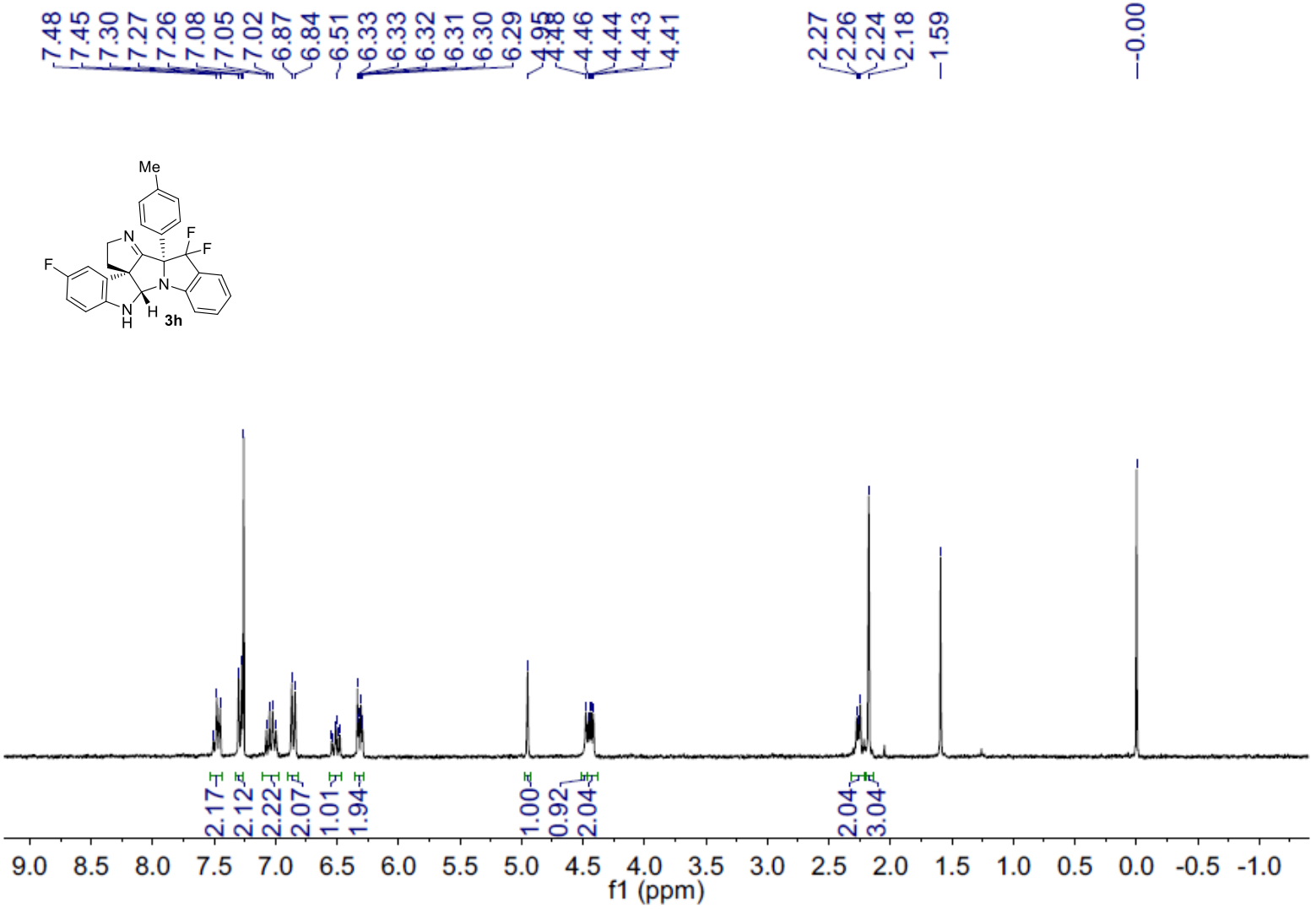
179.78
151.39
151.35
151.33
151.28
148.10
133.27
132.80
132.47
131.54
131.50
130.13
128.40
127.83
127.31
127.26
126.06
125.83
125.77
125.57
124.74
123.92
123.83
123.70
122.49
122.34
122.29
122.28
122.18
122.08
121.76
119.34
112.19
109.04
84.77
77.67
77.59
77.43
77.25
77.00
76.75
71.37
66.96
38.97
f1 (ppm)
78.5 78.0 77.5 77.0 76.5 76.0 75.5
f1 (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)
3e

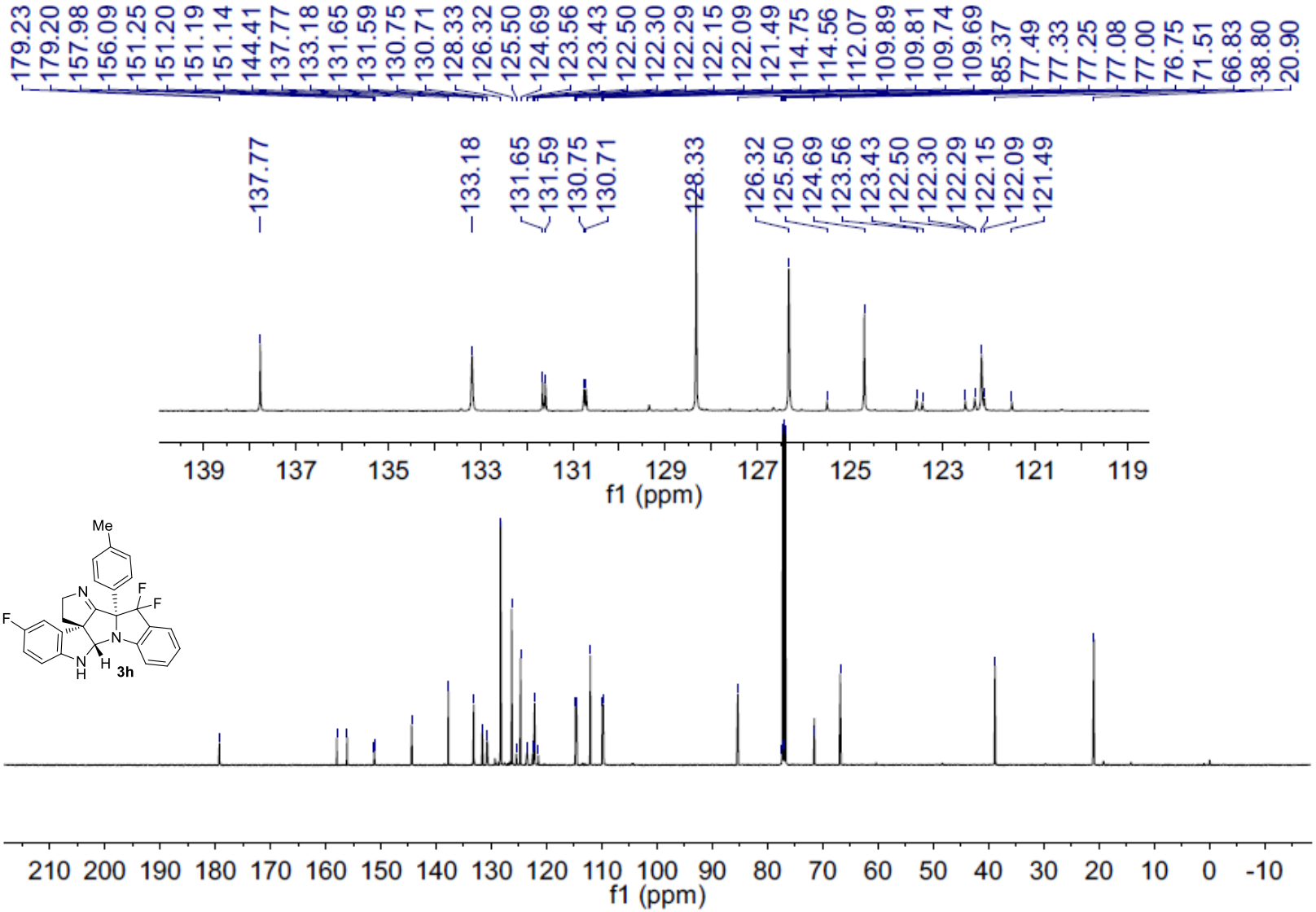

-81.75
-82.29
-95.62
-96.16
F
F
N
N
H
N
H
3e
-35
-45
-55
-65
-75
-85
-95
-105
-115
-125
-135
-145
f1 (ppm)


7.49
7.47
7.47
7.46
7.44
7.43
7.43
7.43
7.42
7.42
7.42
7.08
7.07
7.06
7.06
7.06
7.05
7.05
7.03
7.01
6.51
6.50
6.49
6.48
6.48
6.34
6.33
6.32
6.32
6.31
6.31
4.96
4.50
4.46
4.45
4.44
4.43
2.28
2.27
2.27
2.26
2.25
2.24
1.57
-0.00
F
F
F
N
N
N
H
H
3g
2.28
2.13
4.16
1.10
1.17
2.03
1.00
1.09
2.10
2.14
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


179.11
179.09
157.93
156.05
151.21
151.17
151.15
151.11
144.38
144.37
133.92
133.87
133.26
131.52
131.46
128.12
127.66
126.35
125.53
124.71
123.59
122.40
122.24
122.20
122.18
121.98
115.04
114.85
112.09
109.88
109.83
109.76
109.69
85.39
77.45
77.37
77.25
77.00
76.75
71.54
66.88
f1 (ppm)
3g


-82.20
-82.74
-95.78
-96.32
-125.11
F
F
F
N
N
H
N
H
3g
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
f1 (ppm)


7.48
7.45
7.30
7.27
7.26
7.08
7.05
7.02
6.87
6.84
6.51
6.33
6.33
6.32
6.31
6.30
6.29
4.46
4.44
4.43
4.41
2.27
2.26
2.24
2.18
1.59
0.00
Me
F
F
F
N
N
H
N
H
3h
2.17
2.12
2.22
2.07
1.01
1.94
1.00
0.92
2.04
2.04
3.04
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


179.23
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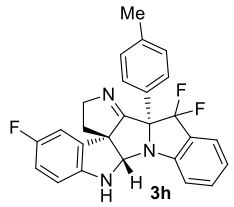
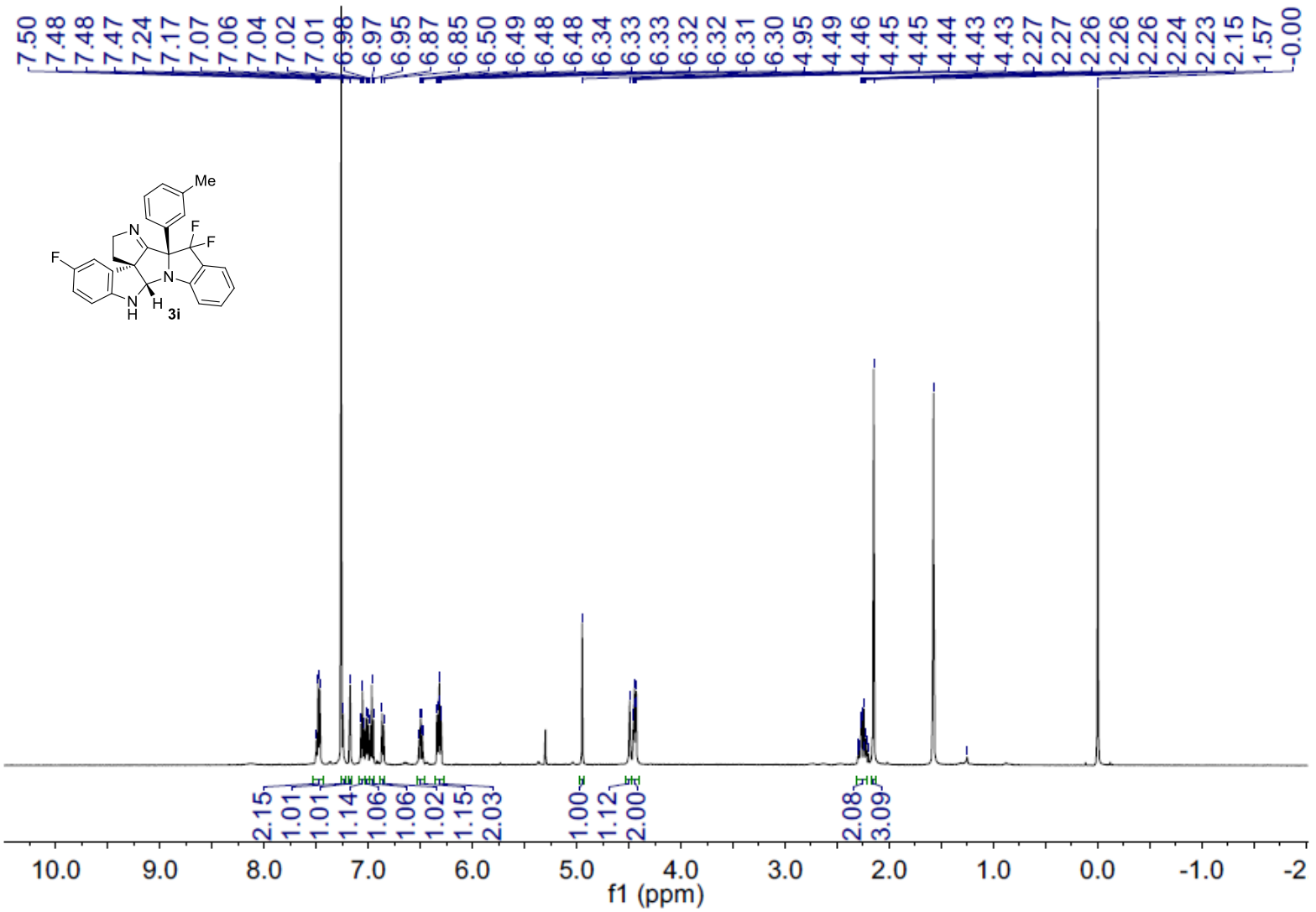
151.25
151.20
151.19
151.14
144.41
137.77
133.18
131.65
131.59
130.75
130.71
128.33
126.32
125.50
124.69
123.56
123.43
122.50
122.30
122.29
122.15
122.09
121.49
114.75
114.56
112.07
109.89
109.81
109.74
109.69
85.37
77.49
77.33
77.25
77.08
77.00
76.75
71.51
66.83
38.80
20.90
137.77
133.18
131.65
131.59
130.75
130.71
128.33
126.32
125.50
124.69
123.56
123.43
122.50
122.30
122.29
122.15
122.09
121.49
139 137 135 133 131 129 127 125 123 121 119
f1 (ppm)
Me
F
F
F
N
N
H
N
H
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Me
F
F
F
N
N
H
N
H
3h

-82.15
-83.05
-96.24
-97.14
-125.29
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)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7.48
7.48
7.47
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7.17
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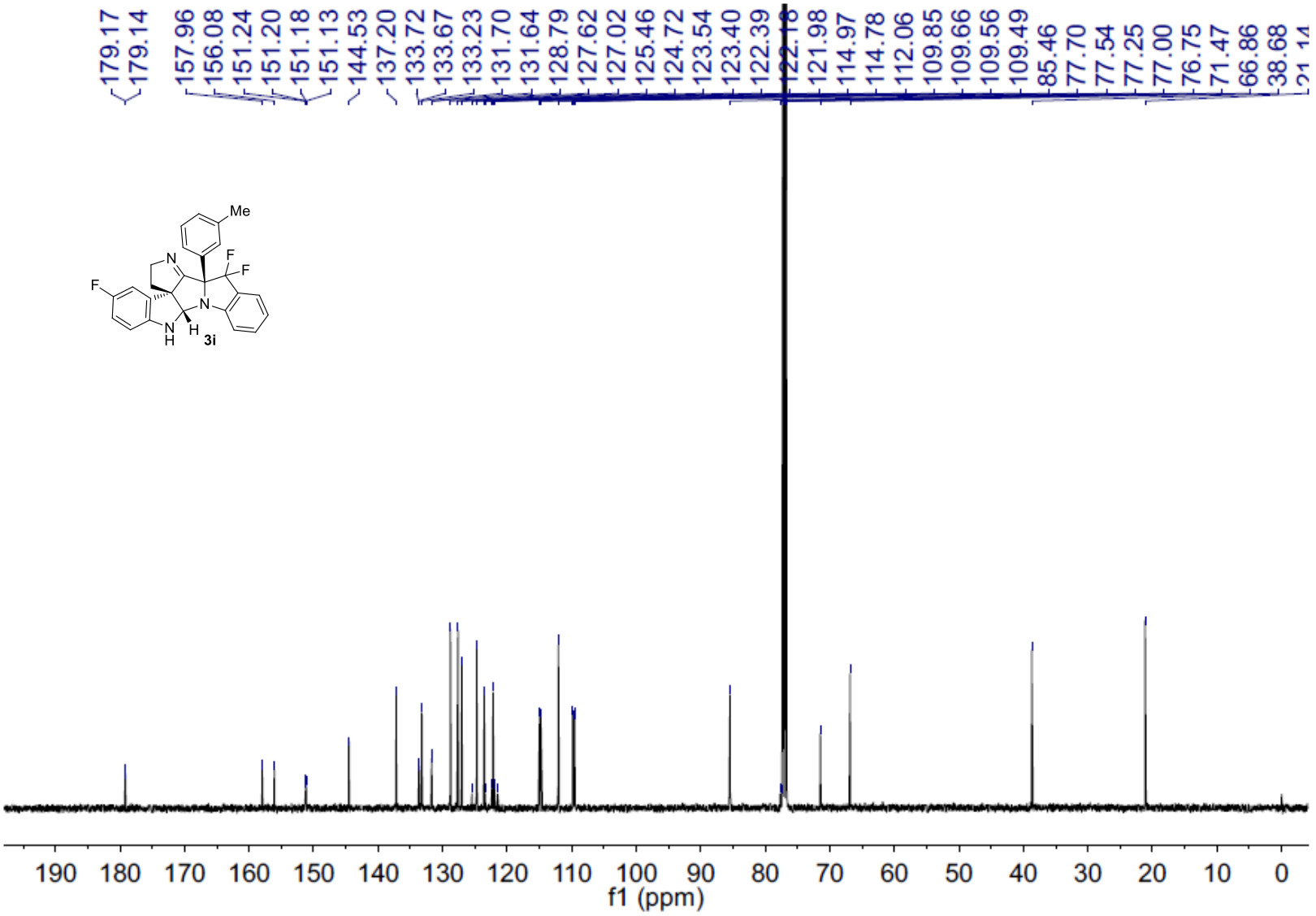
7.06
7.04
7.02
7.01
6.98
6.97
6.95
6.87
6.85
6.50
6.49
6.48
6.48
6.34
6.33
6.33
6.32
6.32
6.31
6.30
4.95
4.49
4.46
4.45
4.45
4.44
4.43
4.43
2.27
2.27
2.26
2.26
2.26
2.24
2.23
2.15
1.57
-0.00
Me
F
F
F
N
N
H
N
H
3i
2.15
1.01
1.01
1.14
1.06
1.06
1.02
1.15
2.03
1.00
1.12
2.00
2.08
3.09
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2
f1 (ppm)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157.96
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151.13
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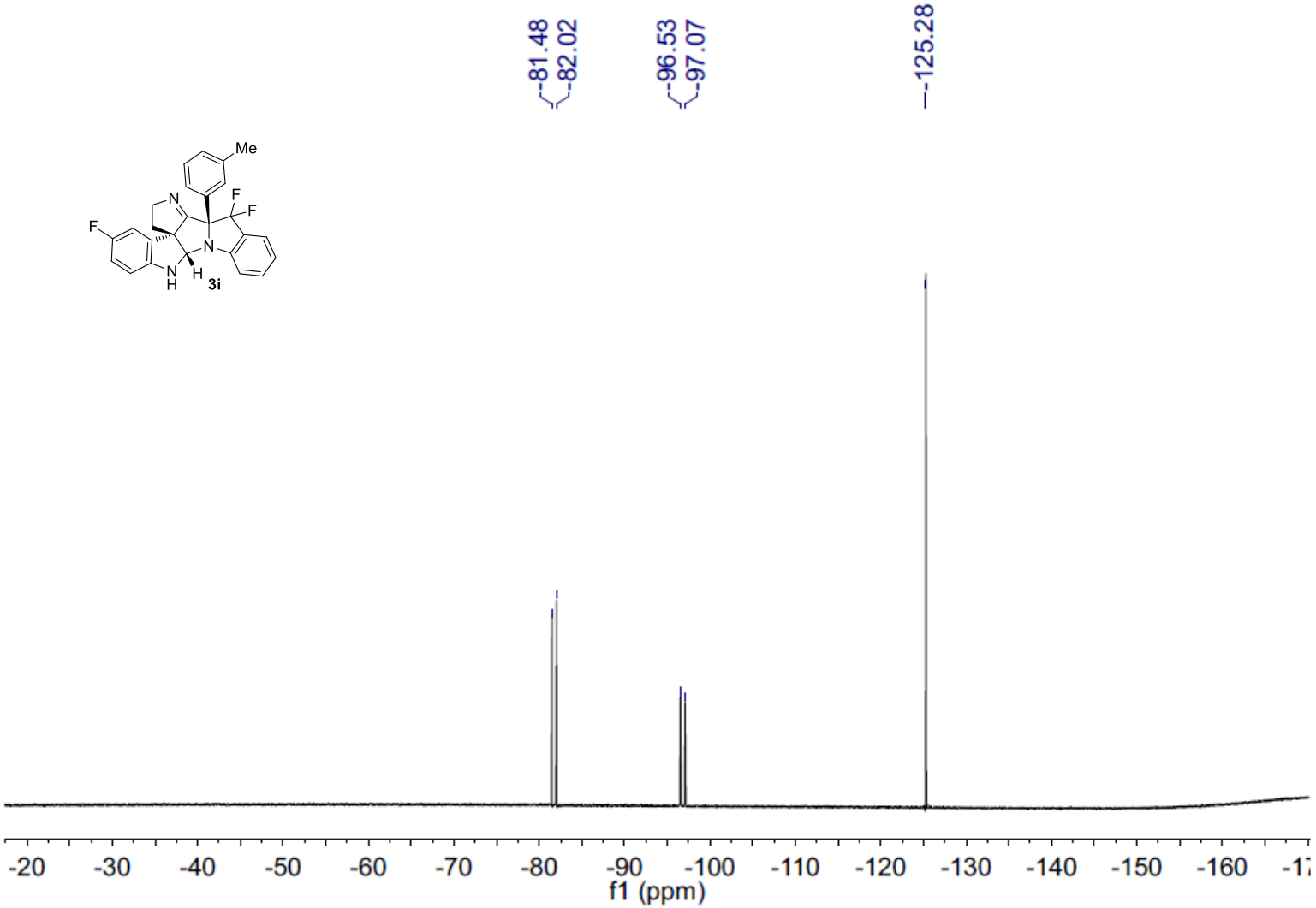
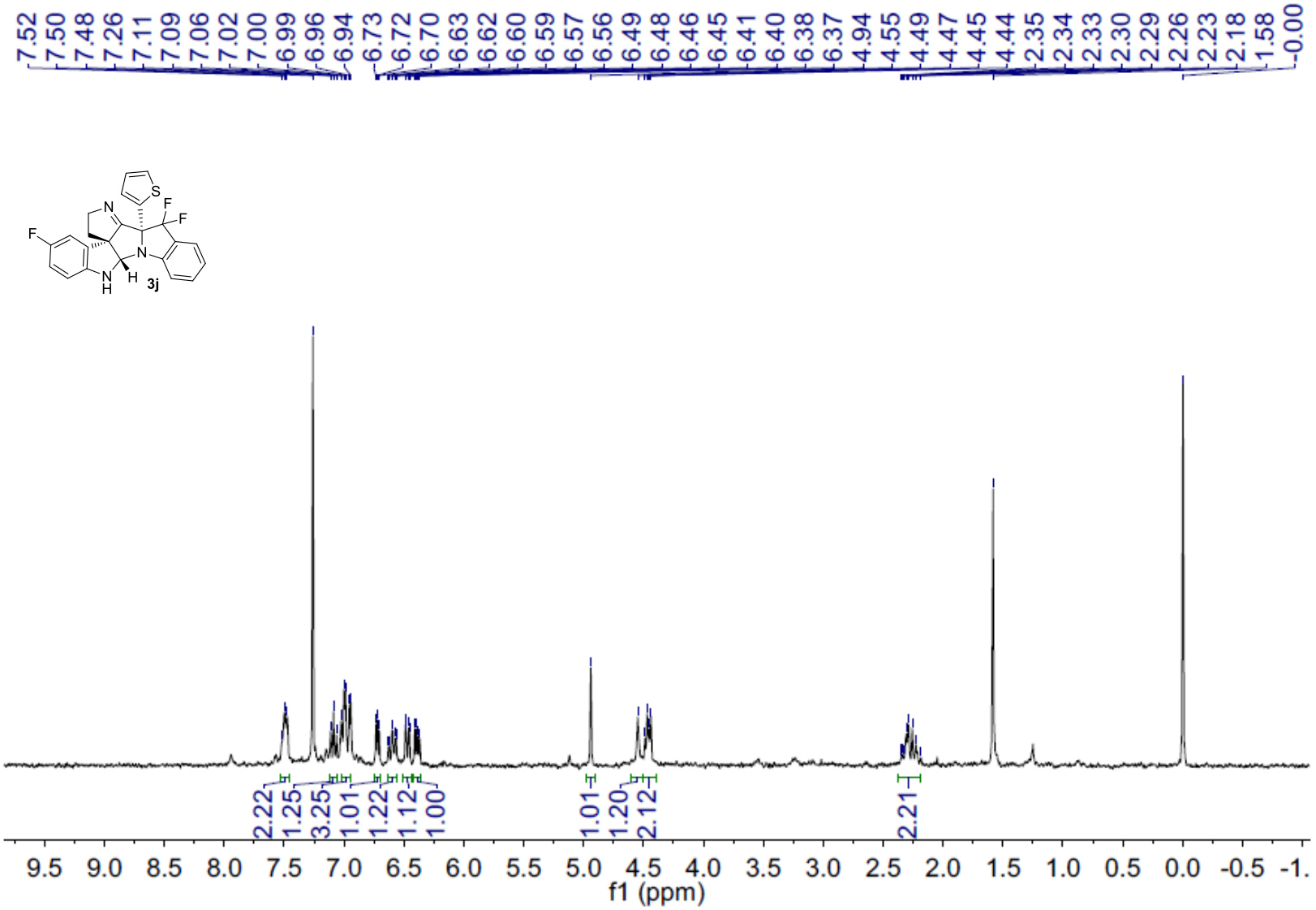
137.20
133.72
133.67
133.23
131.70
131.64
128.79
127.62
127.02
125.46
124.72
123.54
123.40
122.39
122.18
121.98
114.97
114.78
112.06
109.85
109.66
109.56
109.49
85.46
77.70
77.54
77.25
77.00
76.75
71.47
66.86
38.68
Me
F
F
F
N
N
N
H
H
3i
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-81.48
-82.02
-96.53
-97.07
-125.28
Me
F
F
F
N
N
H
H
3i
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)


7.52
7.50
7.48
7.26
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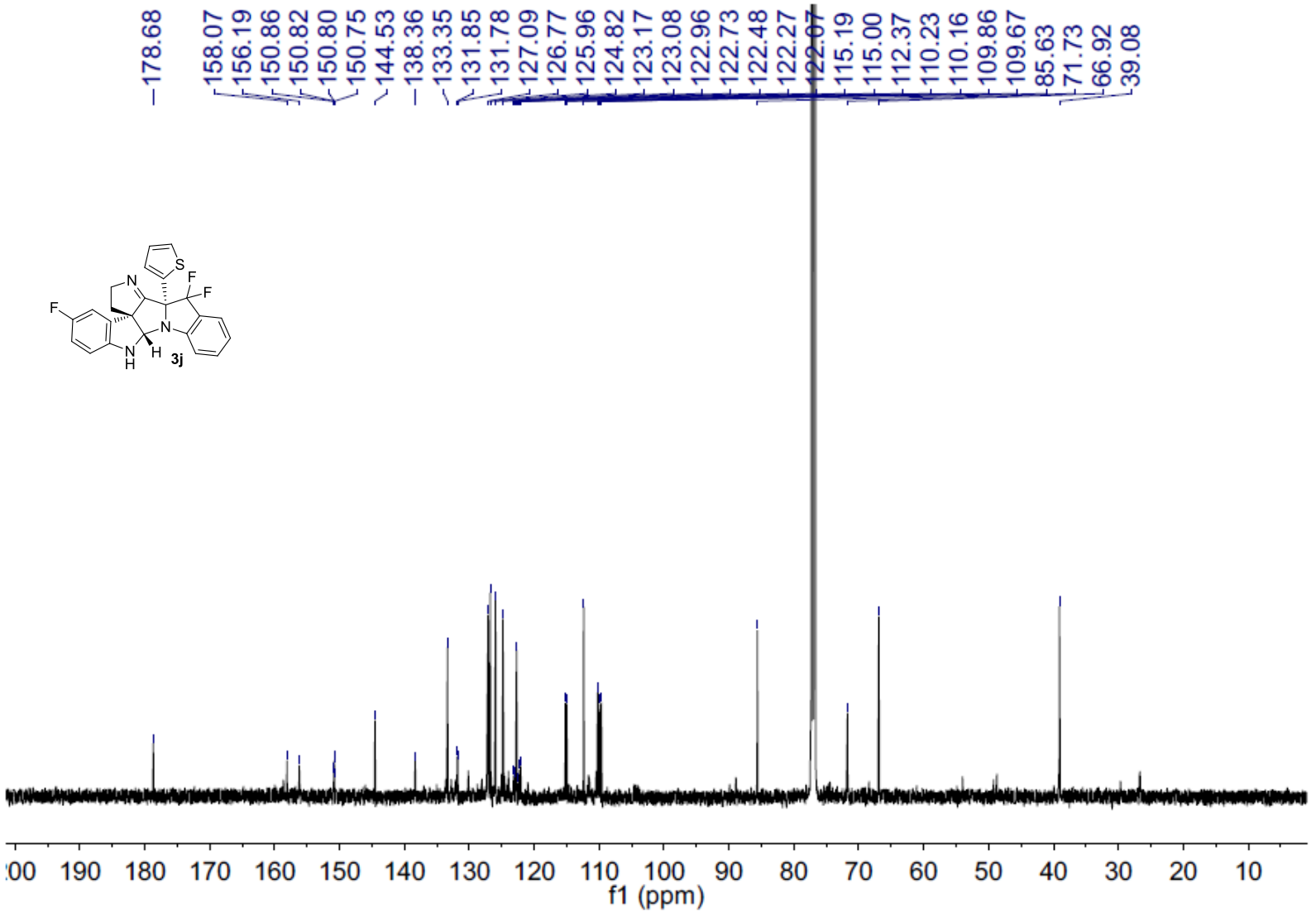
7.09
7.06
7.02
7.00
6.99
6.96
6.94
6.73
6.72
6.70
6.63
6.62
6.60
6.59
6.57
6.56
6.49
6.48
6.46
6.45
6.41
6.40
6.38
6.37
4.94
4.55
4.49
4.47
4.45
4.44
2.35
2.34
2.33
2.30
2.29
2.26
2.23
2.18
1.58
-0.00
3j
2.22
1.25
3.25
1.01
1.22
1.12
1.00
1.01
1.20
2.12
2.21
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)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158.07
156.19
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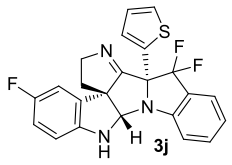
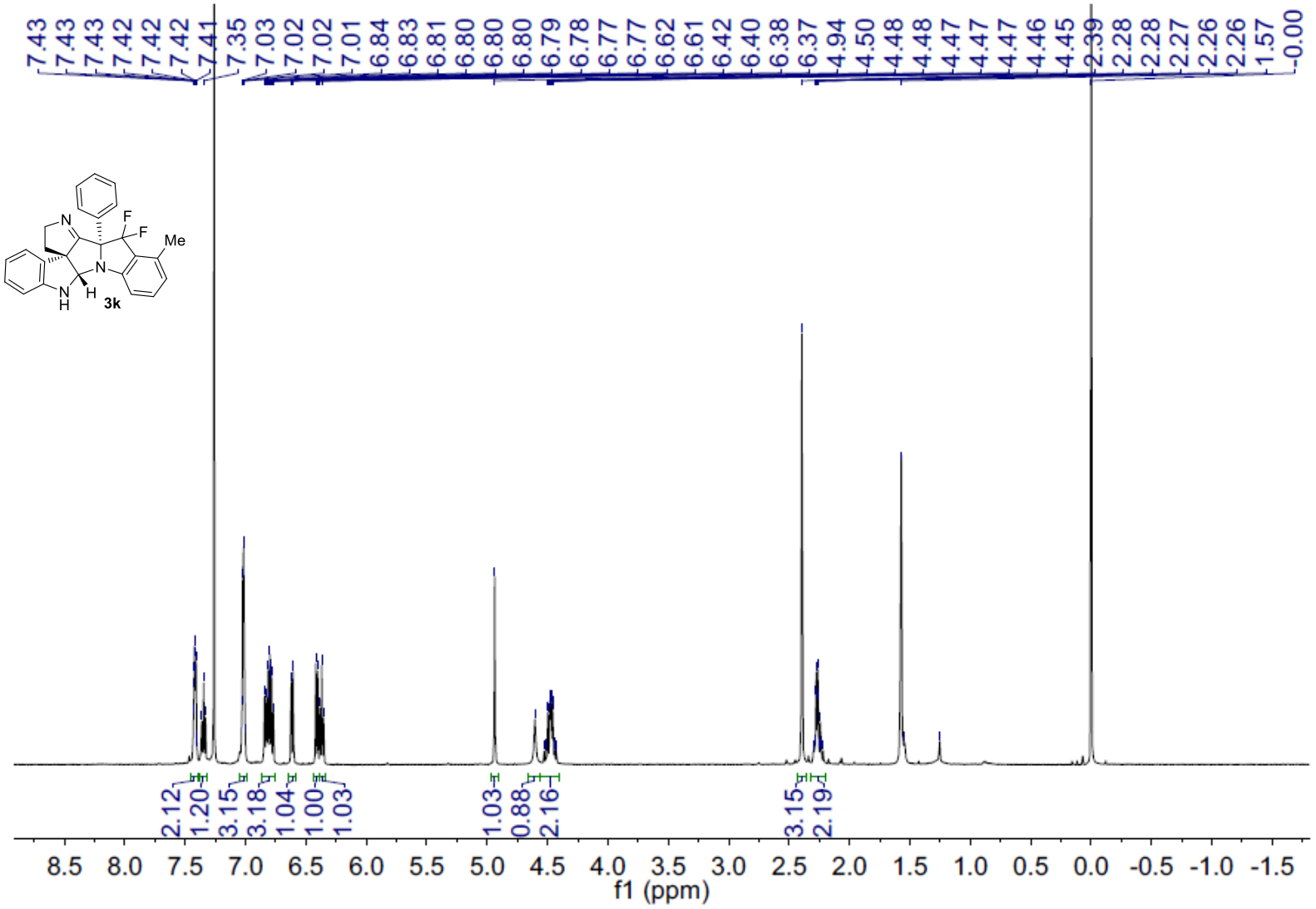
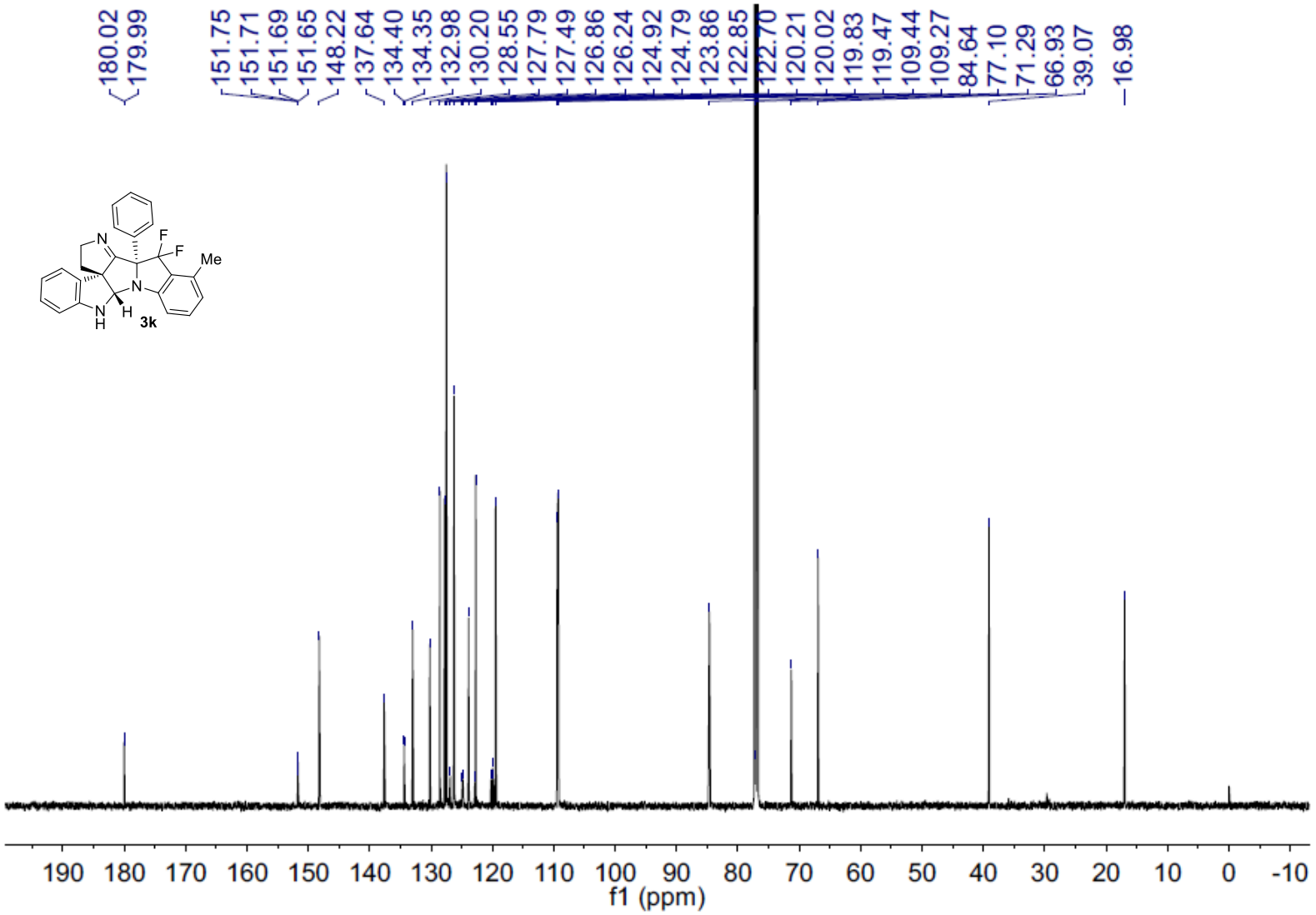
150.82
150.80
150.75
144.53
138.36
133.35
131.85
131.78
127.09
126.77
125.96
124.82
123.17
123.08
122.96
122.73
122.48
122.27
122.07
115.19
115.00
112.37
110.23
110.16
109.86
109.67
85.63
71.73
66.92
39.08
F
S
F
F
N
N
N
H
H
3j
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


S
F
F
N
F
N
H
N
H
3j

-84.64
-85.53
-97.32
-98.21
-124.91
50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -15
f1 (ppm)


7.43
7.43
7.43
7.42
7.42
7.42
7.41
7.35
7.03
7.02
7.02
7.01
6.84
6.83
6.81
6.80
6.80
6.80
6.79
6.78
6.77
6.77
6.62
6.61
6.42
6.40
6.38
6.37
4.94
4.50
4.48
4.48
4.47
4.47
4.47
4.46
4.45
2.39
2.28
2.28
2.27
2.26
2.26
1.57
-0.00
N
F
F
Me
N
N
H
H
3k
2.12
1.20
3.15
3.18
1.04
1.00
1.03
1.03
0.88
2.16
3.15
2.19
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)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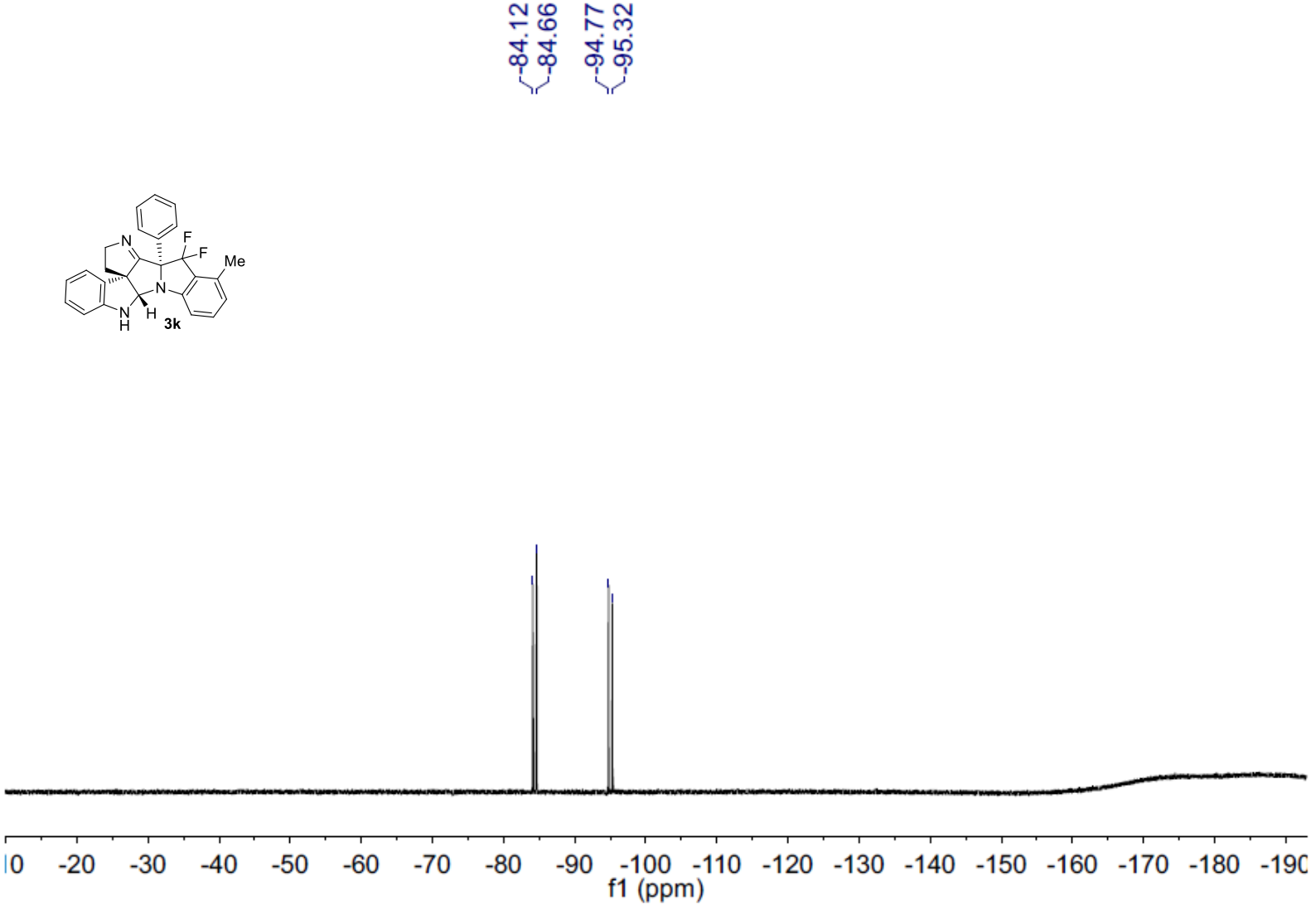
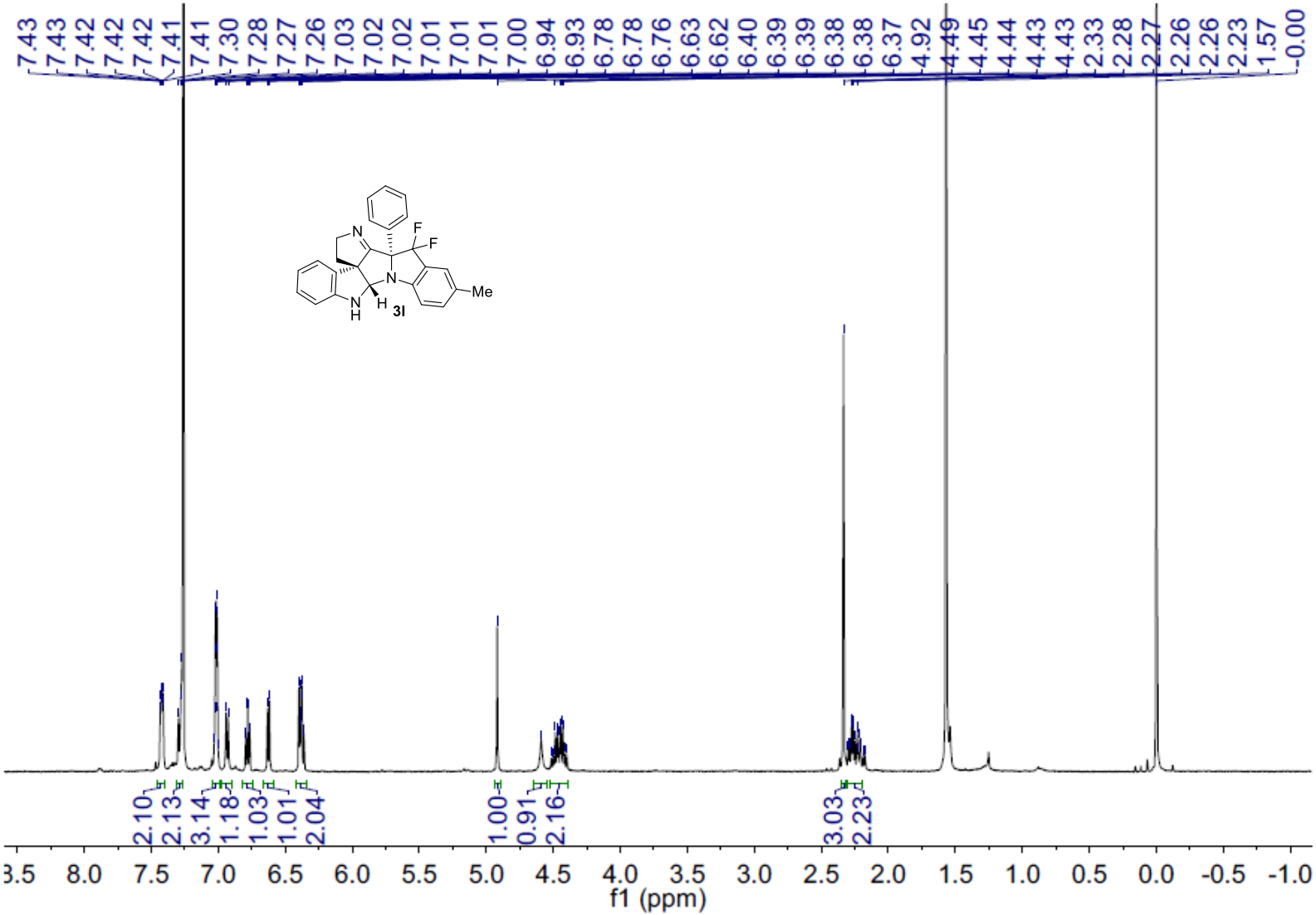
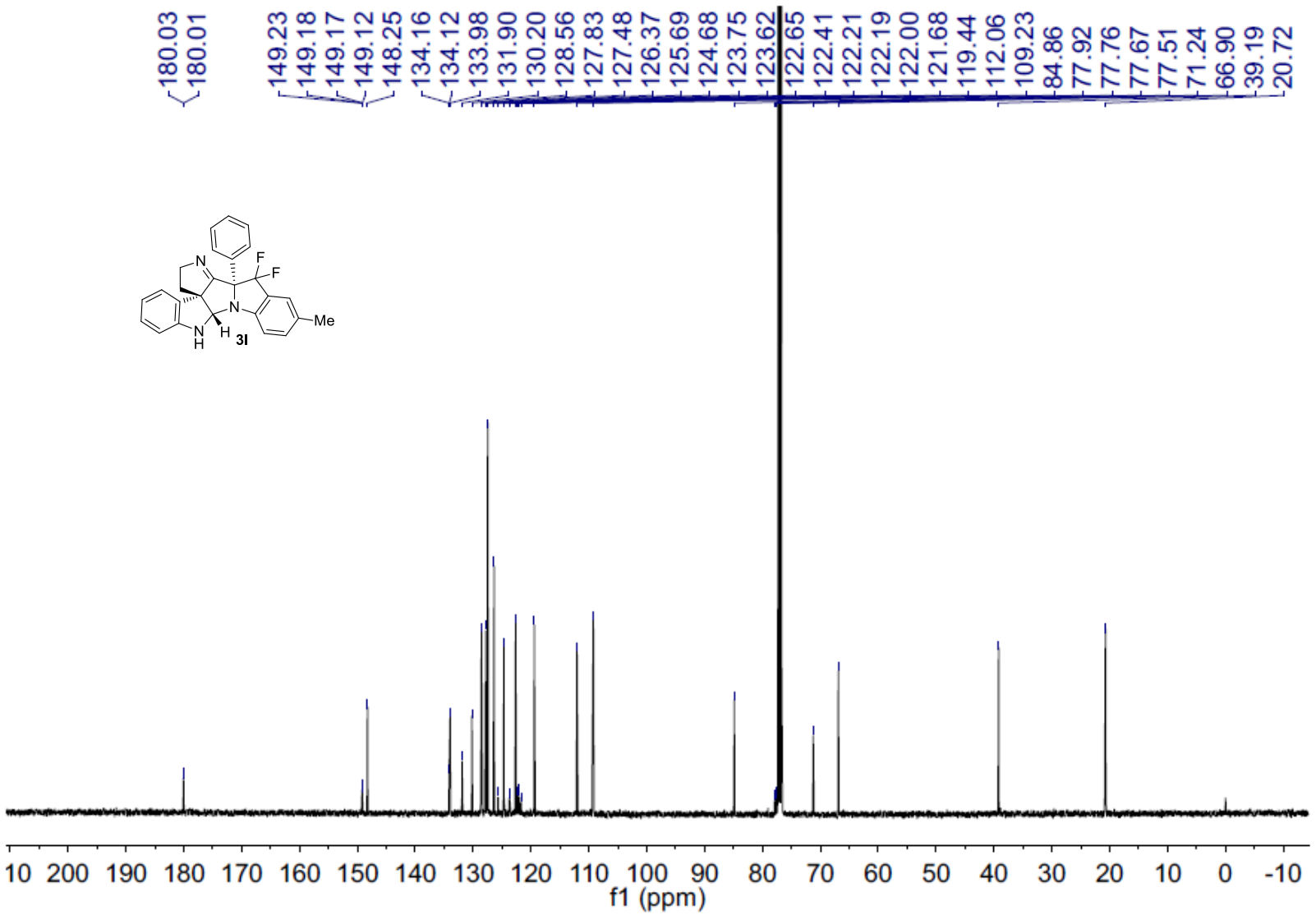
179.99
151.75
151.71
151.69
151.65
148.22
137.64
134.40
134.35
132.98
130.20
128.55
127.79
127.49
126.86
126.24
124.92
124.79
123.86
122.85
122.70
120.21
120.02
119.83
119.47
109.44
109.27
84.64
77.10
71.29
66.93
39.07
16.98
N
F
F
Me
N
N
H
H
3k
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-84.12
-84.66
-94.77
-95.32
N
F
F
Me
N
N
H
H
3k
0
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)


7.43
7.43
7.42
7.42
7.42
7.41
7.41
7.30
7.28
7.27
7.26
7.03
7.02
7.02
7.01
7.01
7.01
7.00
6.94
6.93
6.78
6.78
6.76
6.63
6.62
6.40
6.39
6.39
6.38
6.38
6.37
4.92
4.49
4.45
4.44
4.43
4.43
2.33
2.28
2.27
2.26
2.26
2.23
1.57
-0.00
N
F
F
N
N
H
H
Me
3l
2.10
2.13
3.14
1.18
1.03
1.01
2.04
1.00
0.91
2.16
3.03
2.23
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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180.01
149.23
149.18
149.17
149.12
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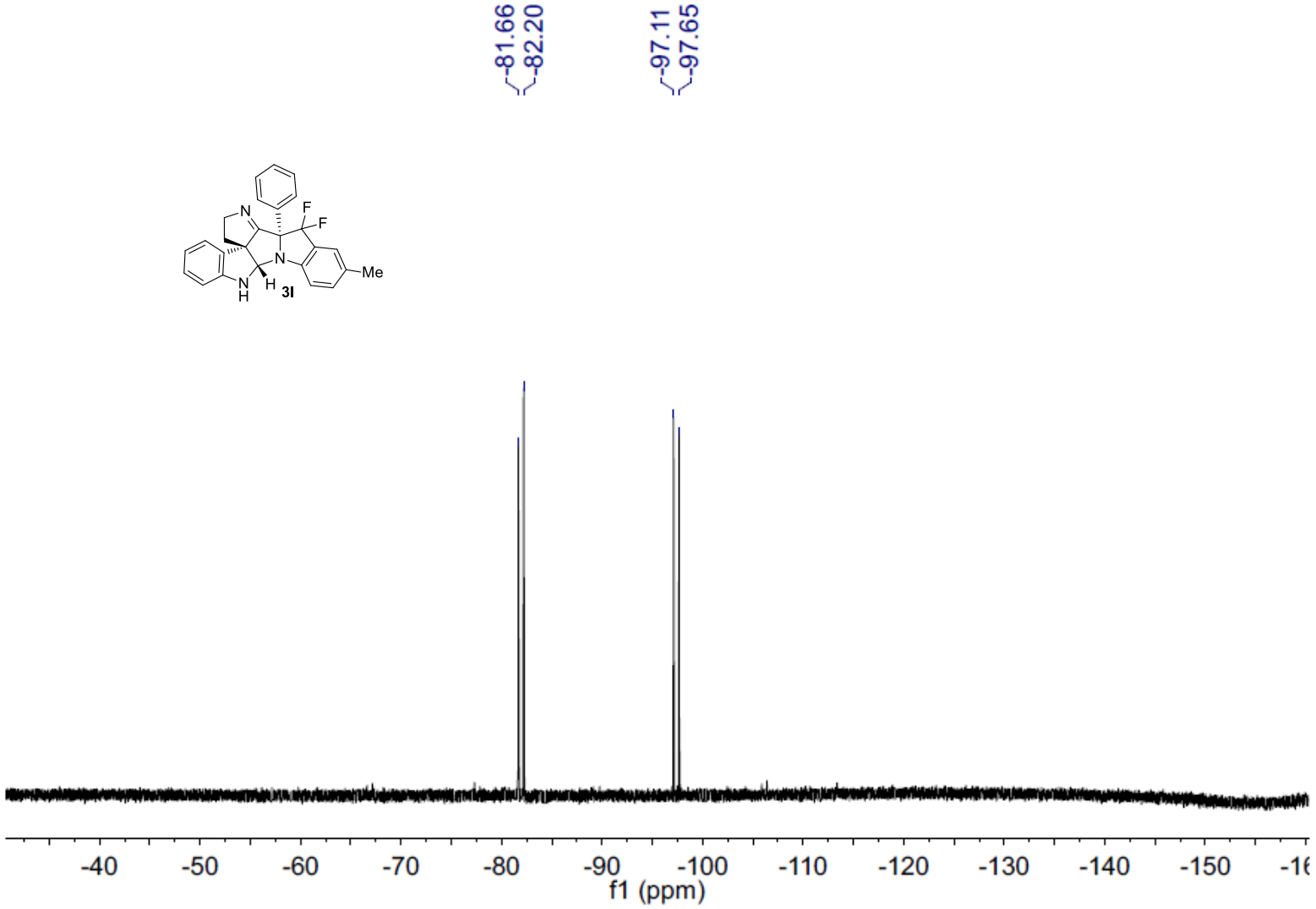
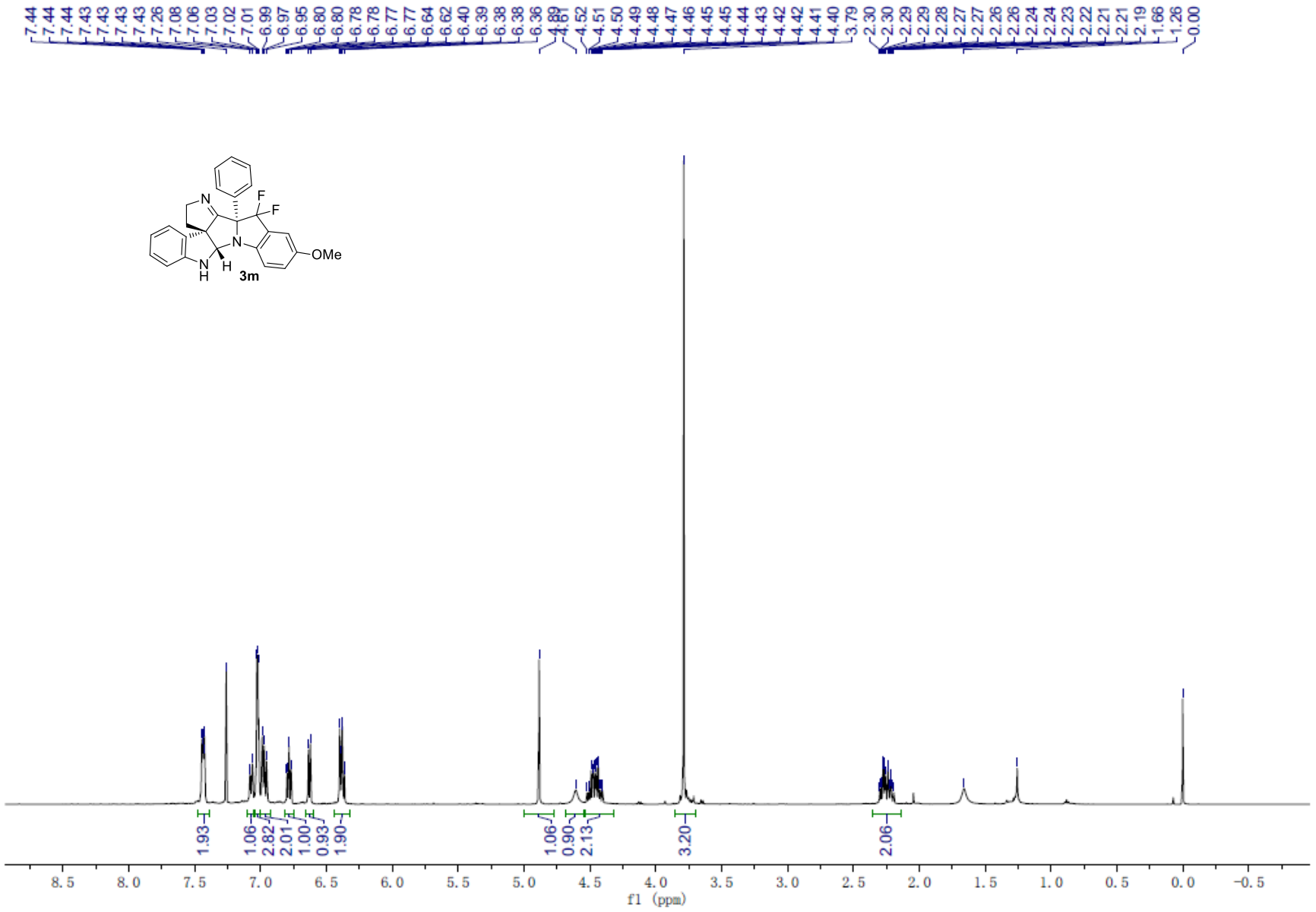
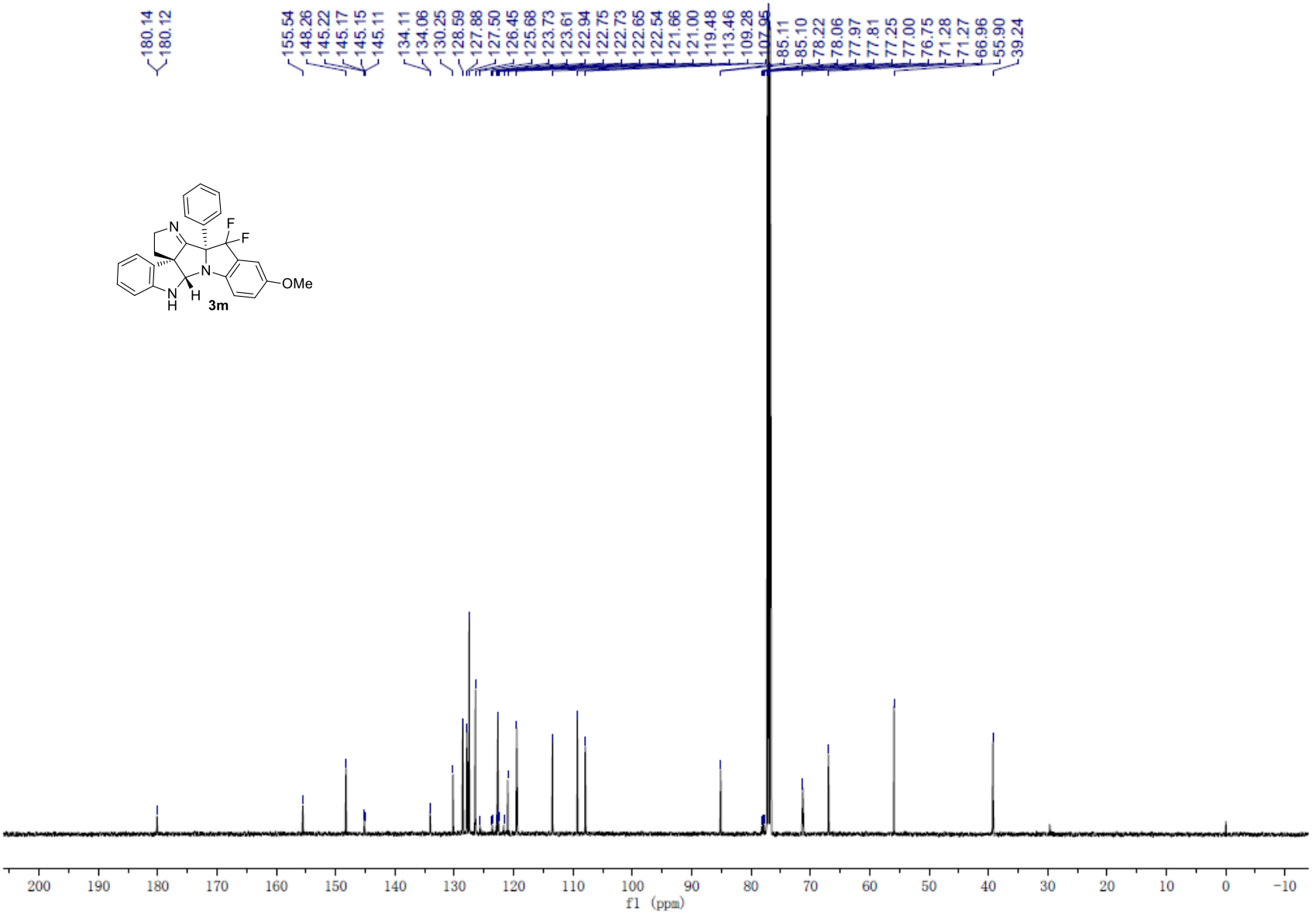
134.16
134.12
133.98
131.90
130.20
128.56
127.83
127.48
126.37
125.69
124.68
123.75
123.62
122.65
122.41
122.21
122.19
122.00
121.68
119.44
112.06
109.23
84.86
77.92
77.76
77.67
77.51
71.24
66.90
39.19
20.72
N
F
F
N
N
H
H
Me
3l
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-81.66
-82.20
-97.11
-97.65
F
F
N
N
H
N
H
Me
3l
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)


7.44 7.44 7.44 7.43 7.43 7.43 7.43 7.26 7.08 7.06 7.03 7.02 7.01 6.99 6.97 6.95 6.80 6.80 6.78 6.78 6.77 6.77 6.64 6.62 6.40 6.39 6.38 6.38 6.36 4.89 4.61 4.52 4.51 4.50 4.49 4.48 4.47 4.46 4.45 4.45 4.44 4.43 4.42 4.42 4.41 4.40 3.79 2.30 2.30 2.29 2.29 2.28 2.27 2.27 2.26 2.26 2.24 2.24 2.23 2.22 2.21 2.21 2.19 1.66 1.26 0.00
F
F
N
N
H
N
H
OMe
3m
1.93
1.06
2.82
2.01
1.00
0.93
1.90
1.06
0.90
2.13
3.20
2.06
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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148.26
145.22
145.17
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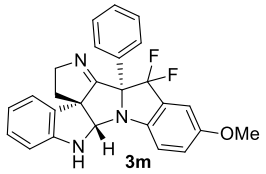
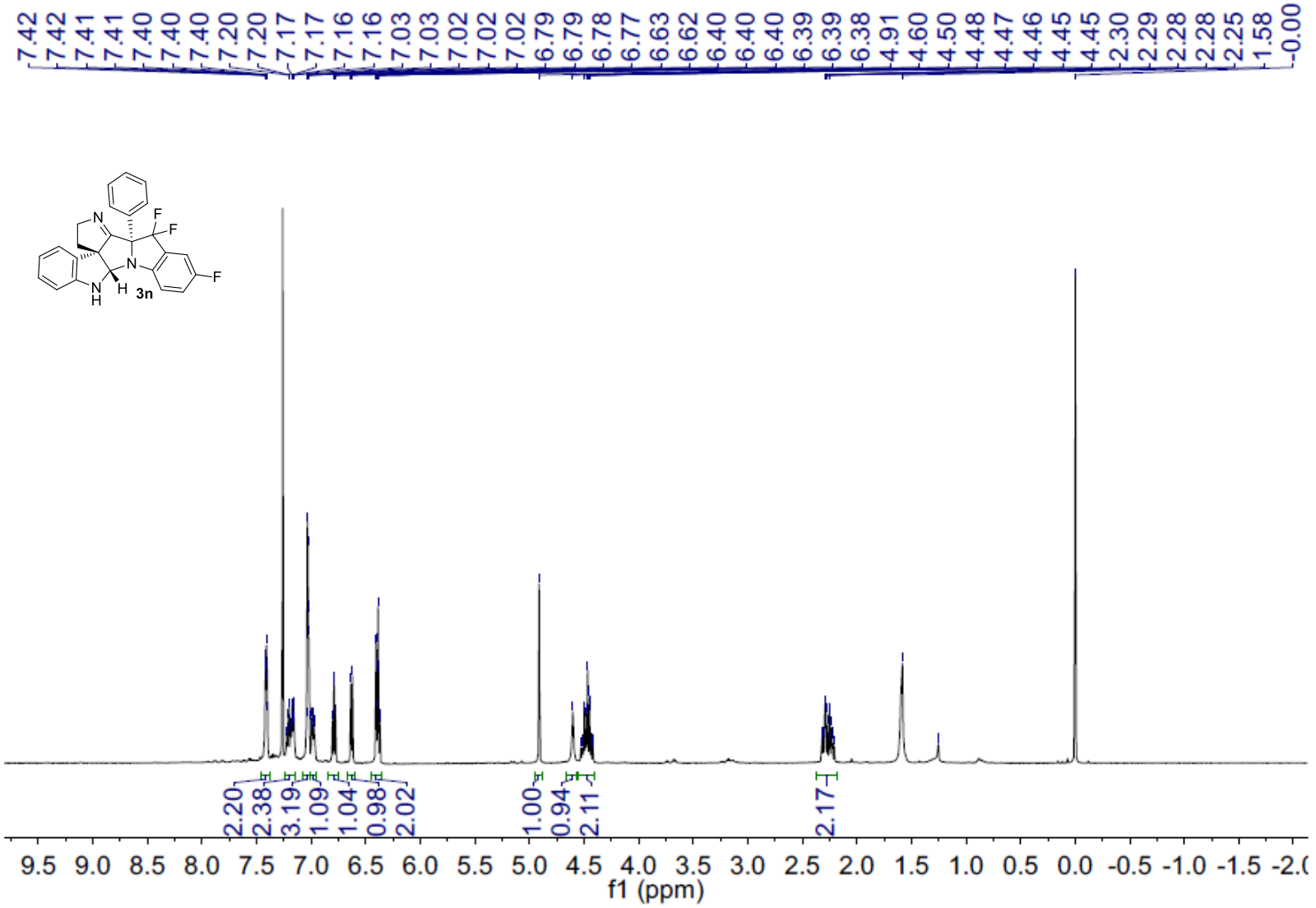
145.11
134.11
134.06
130.25
128.59
127.88
127.50
126.45
125.68
123.73
123.61
122.94
122.75
122.73
122.65
122.54
121.66
121.00
119.48
113.46
109.28
107.95
85.11
85.10
78.22
78.06
77.97
77.81
77.25
77.00
76.75
71.28
71.27
66.96
55.90
39.24
F
F
N
N
H
N
H
OMe
3m
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


F
F
N
N
H
H
OMe

3m

-81.97
-82.51
-97.53
-98.07
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)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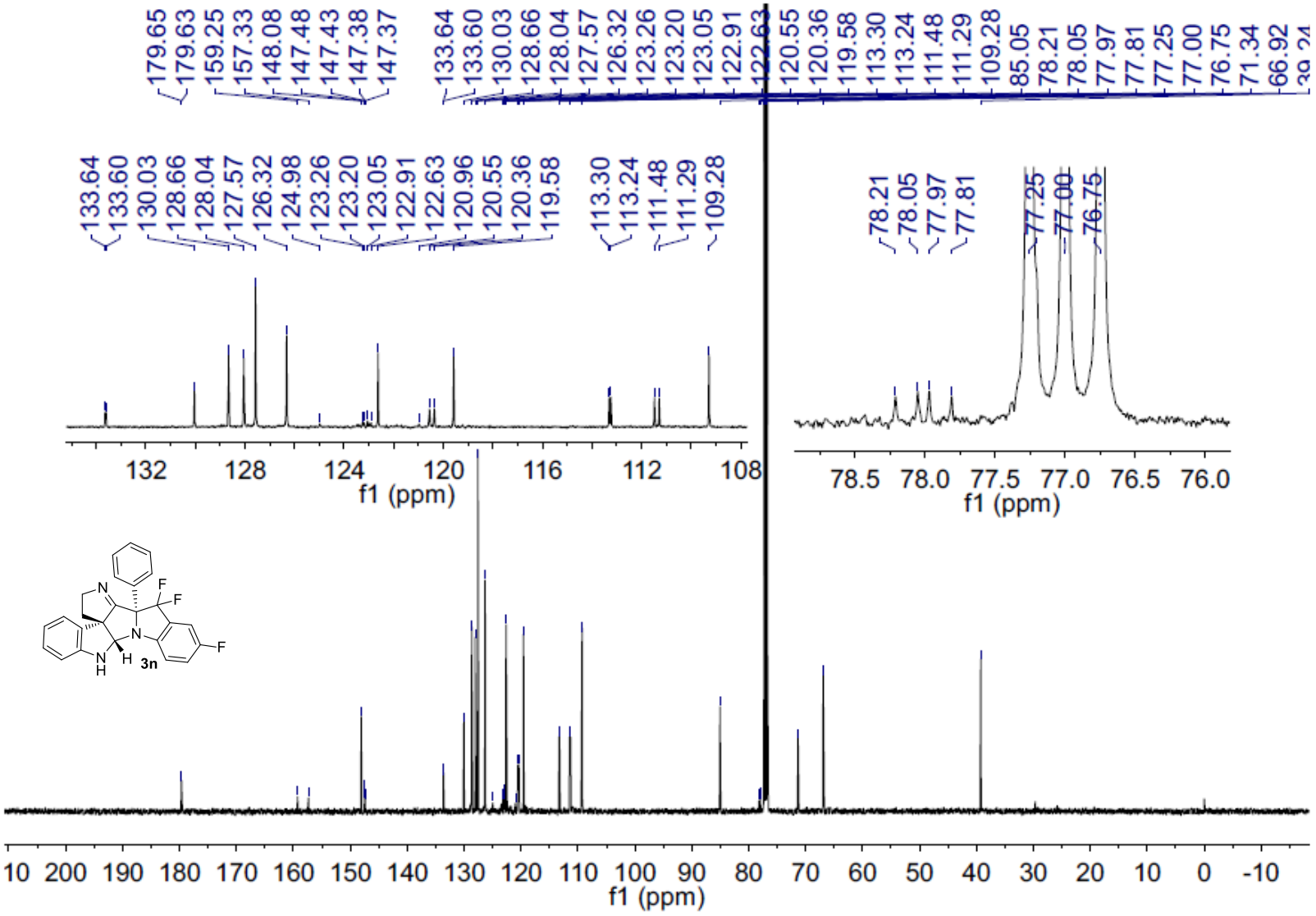
7.40
7.20
7.20
7.17
7.17
7.16
7.16
7.03
7.03
7.02
7.02
7.02
6.79
6.79
6.78
6.77
6.63
6.62
6.40
6.40
6.40
6.39
6.39
6.38
4.91
4.60
4.50
4.48
4.47
4.46
4.45
4.45
2.30
2.29
2.28
2.28
2.25
1.58
-0.00
F
F
F
N
N
H
N
H
3n
2.20
2.38
3.19
1.09
1.04
0.98
2.02
1.00
0.94
2.11
2.17
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.(
f1 (ppm)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147.38
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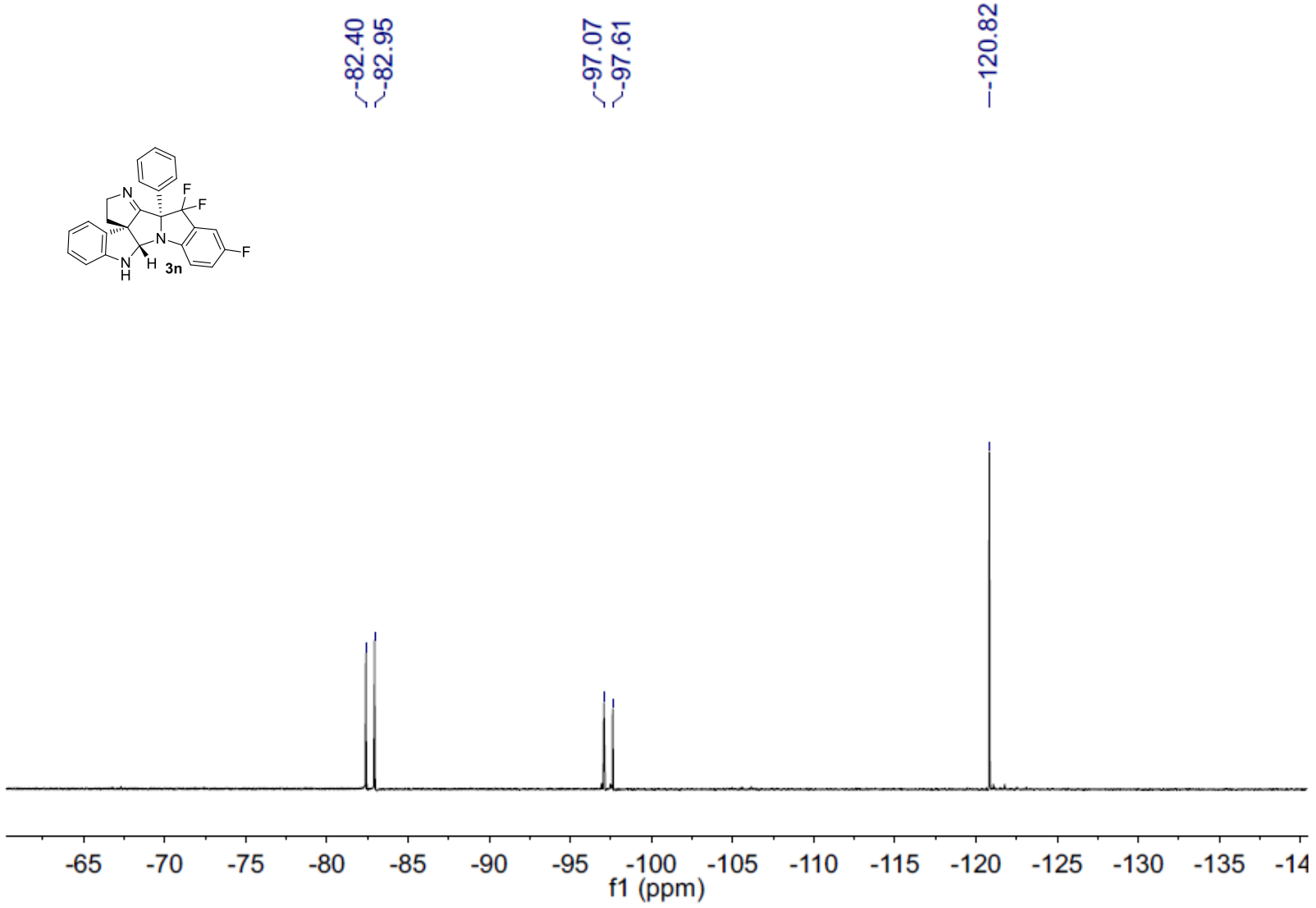
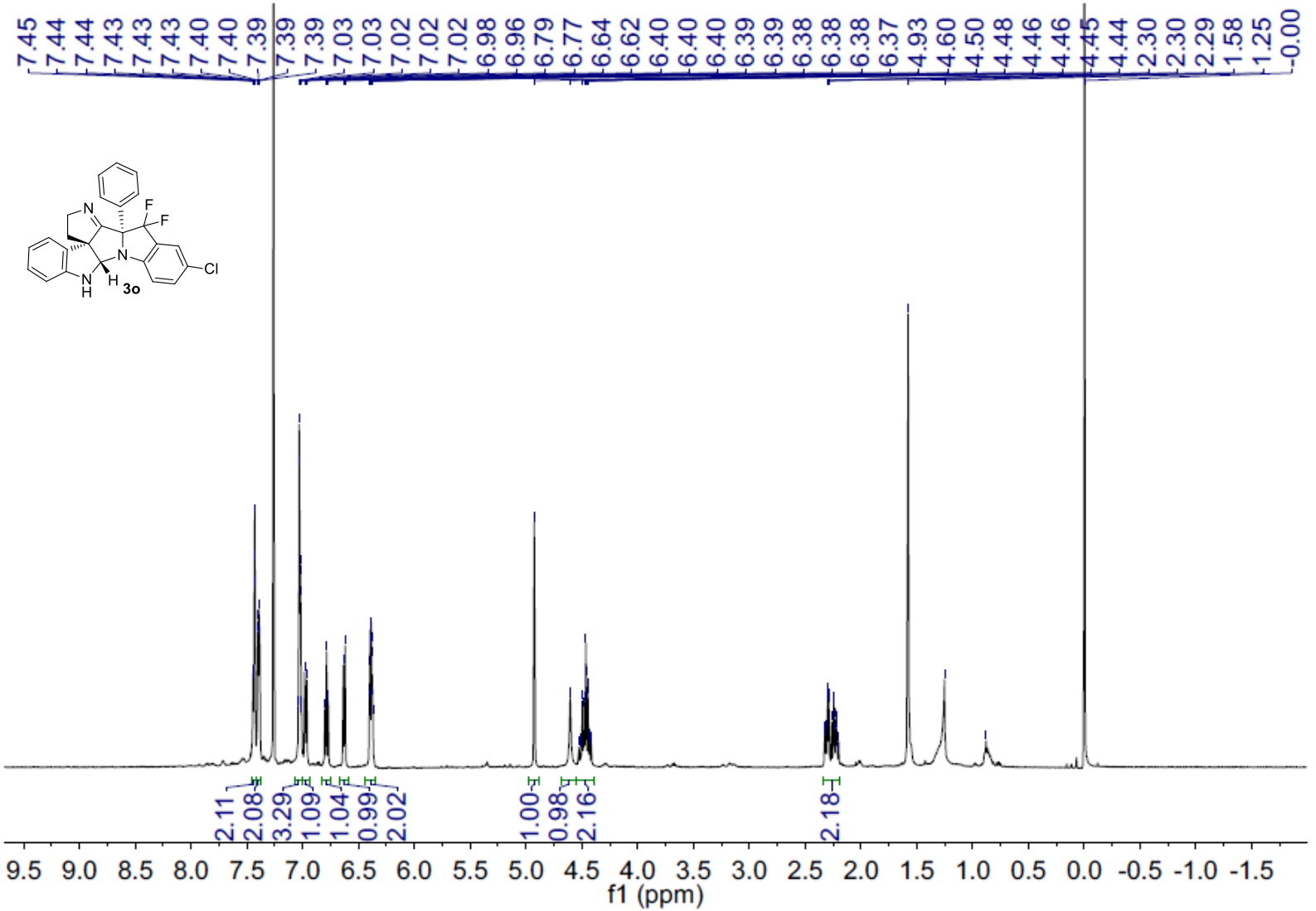
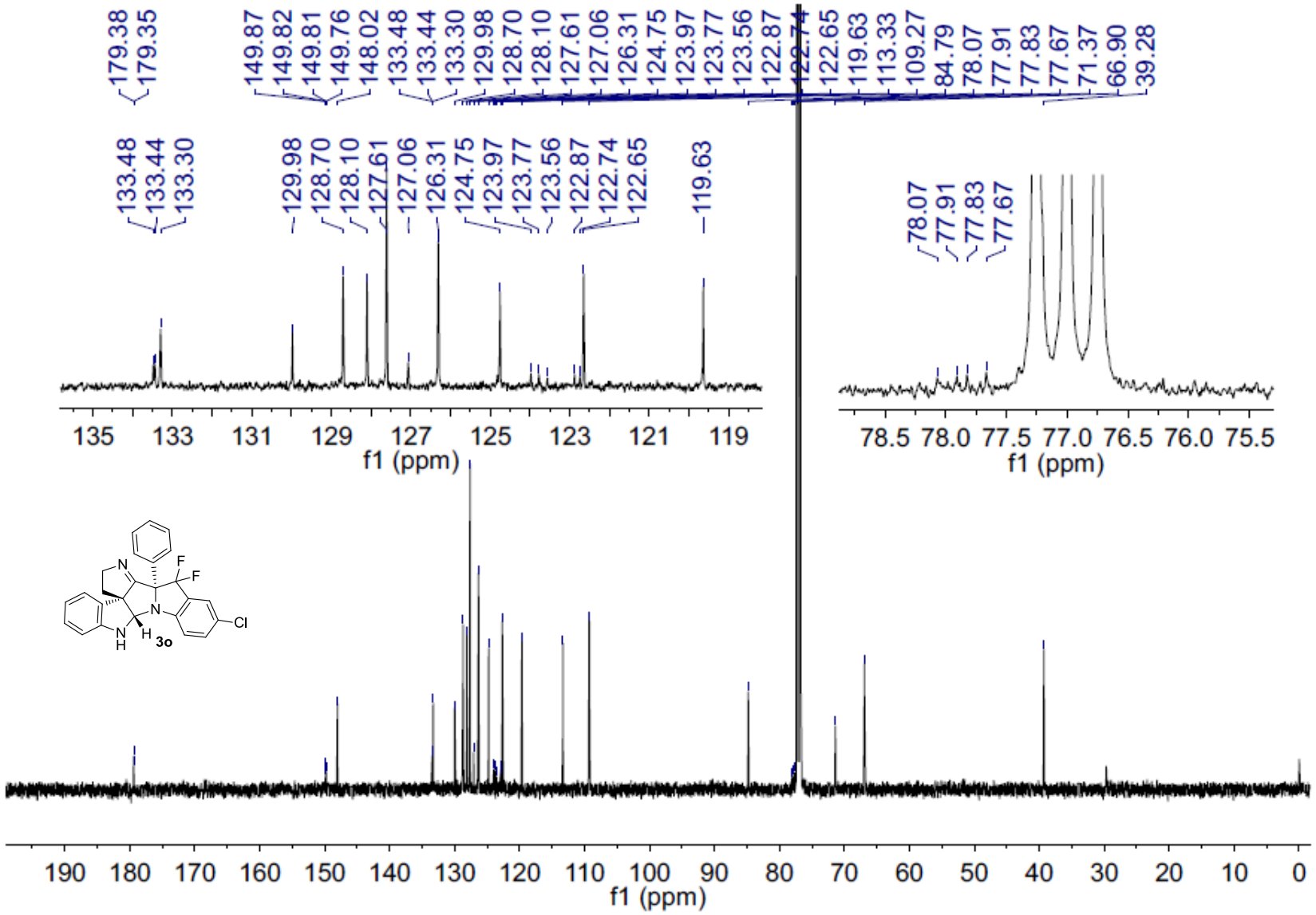
133.64
133.60
130.03
128.66
128.04
127.57
126.32
123.26
123.20
123.05
122.91
122.63
120.55
120.36
119.58
113.30
113.24
111.48
111.29
109.28
85.05
78.21
78.05
77.97
77.81
77.25
77.00
76.75
71.34
66.92
f1 (ppm)
3n


-82.40
-82.95
-97.07
-97.61
-120.82
F
F
F
N
N
H
N
H
3n
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
-135
-14
f1 (ppm)


7.45 7.44 7.44 7.43 7.43 7.43 7.40 7.40 7.39 7.39 7.39 7.03 7.03 7.02 7.02 7.02 6.98 6.96 6.79 6.77 6.64 6.62 6.40 6.40 6.40 6.39 6.39 6.38 6.38 6.38 6.37 4.93 4.60 4.50 4.48 4.46 4.46 4.45 4.44 2.30 2.30 2.29 1.58 1.25 -0.00
F
F
N
N
H
N
H
Cl
3o
2.11 2.08 3.29 1.09 1.04 0.99 2.02 1.00 0.98 2.16 2.18
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)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149.76
148.02
133.48
133.44
133.30
129.98
128.70
128.10
127.61
127.06
126.31
124.75
123.97
123.77
123.56
122.87
122.74
122.65
119.63
113.33
109.27
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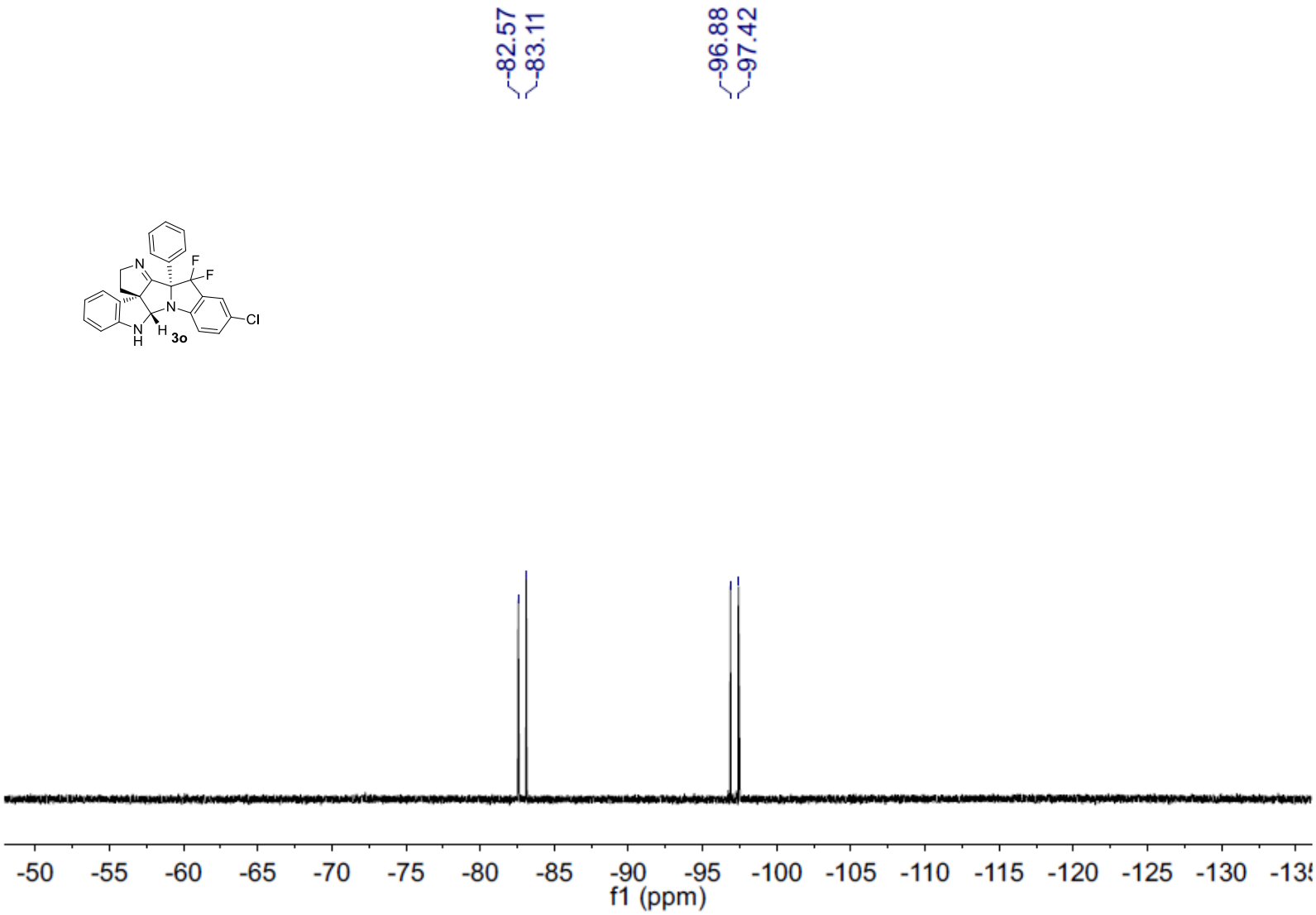
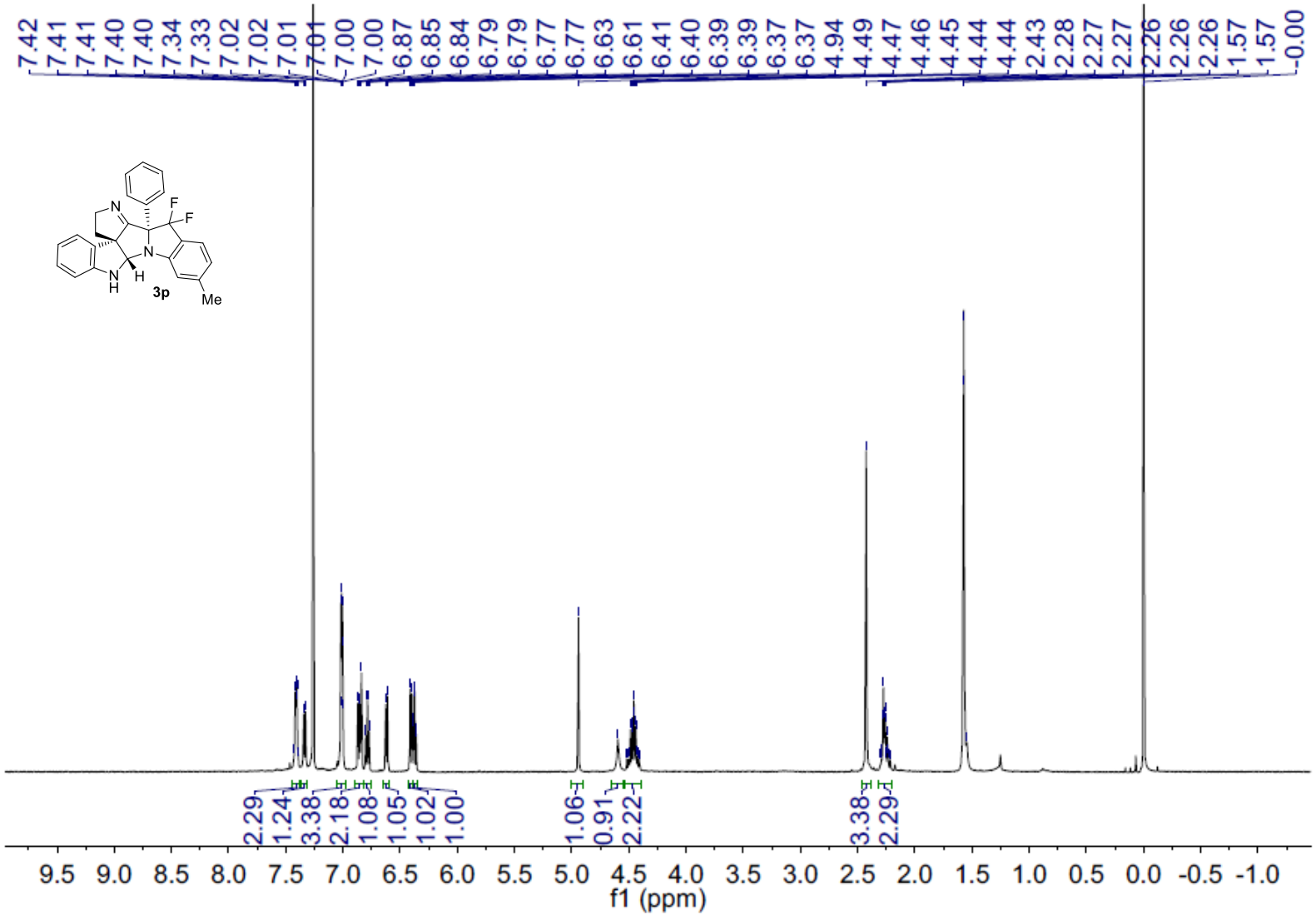
78.07
77.91
77.83
77.67
71.37
66.90
39.28
f1 (ppm)
3o


-82.57
-83.11
-96.88
-97.42
F
F
N
N
H
N
H
Cl
3o
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
f1 (ppm)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7.00
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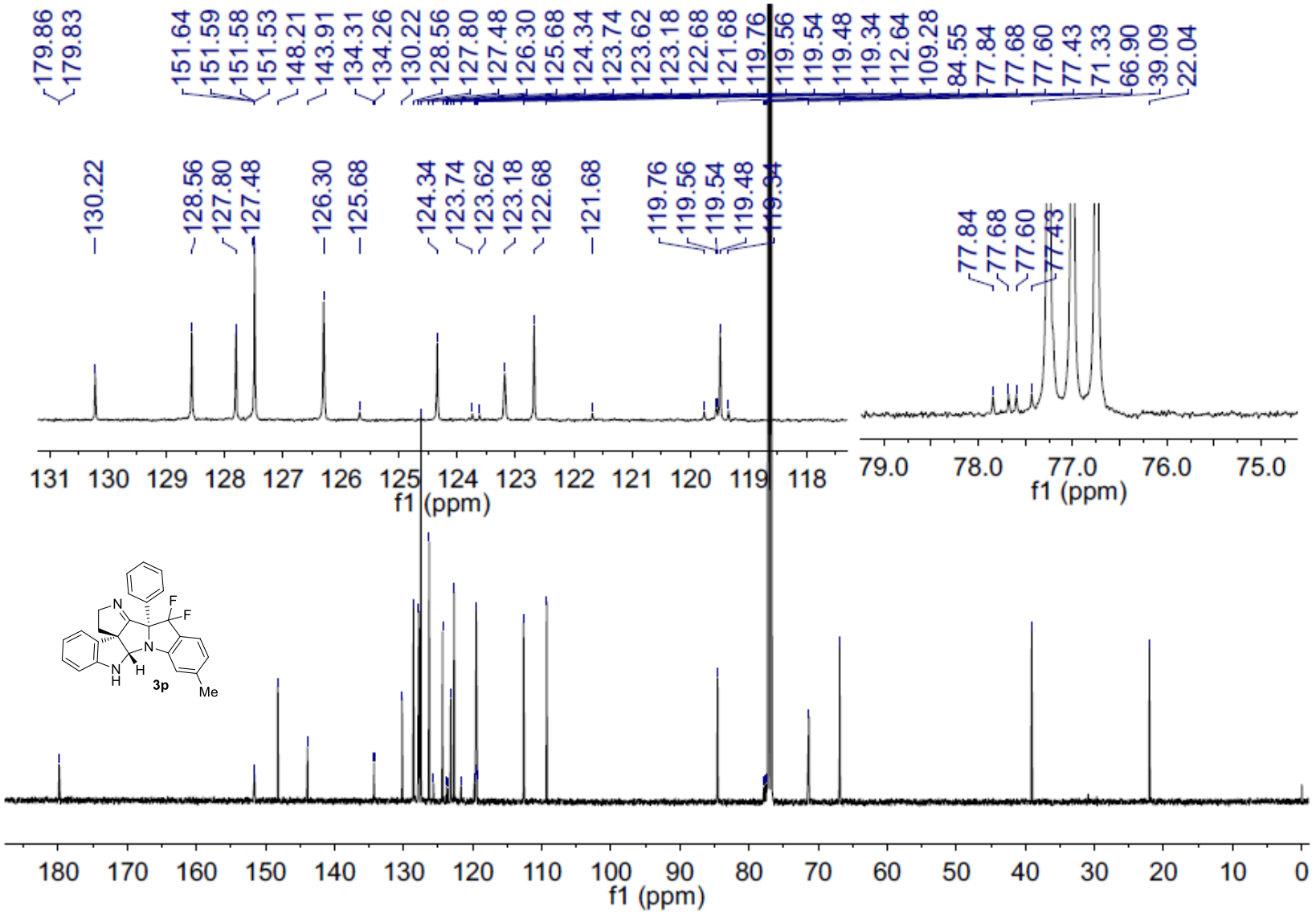
6.85
6.84
6.79
6.79
6.77
6.77
6.63
6.61
6.41
6.40
6.39
6.39
6.37
6.37
4.94
4.49
4.47
4.46
4.45
4.44
4.44
2.43
2.28
2.27
2.27
2.26
2.26
2.26
1.57
1.57
0.00
F
F
N
N
H
N
H
3p
Me
2.29
1.24
3.38
2.18
1.08
1.05
1.02
1.00
1.06
0.91
2.22
3.38
2.29
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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143.91
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134.26
130.22
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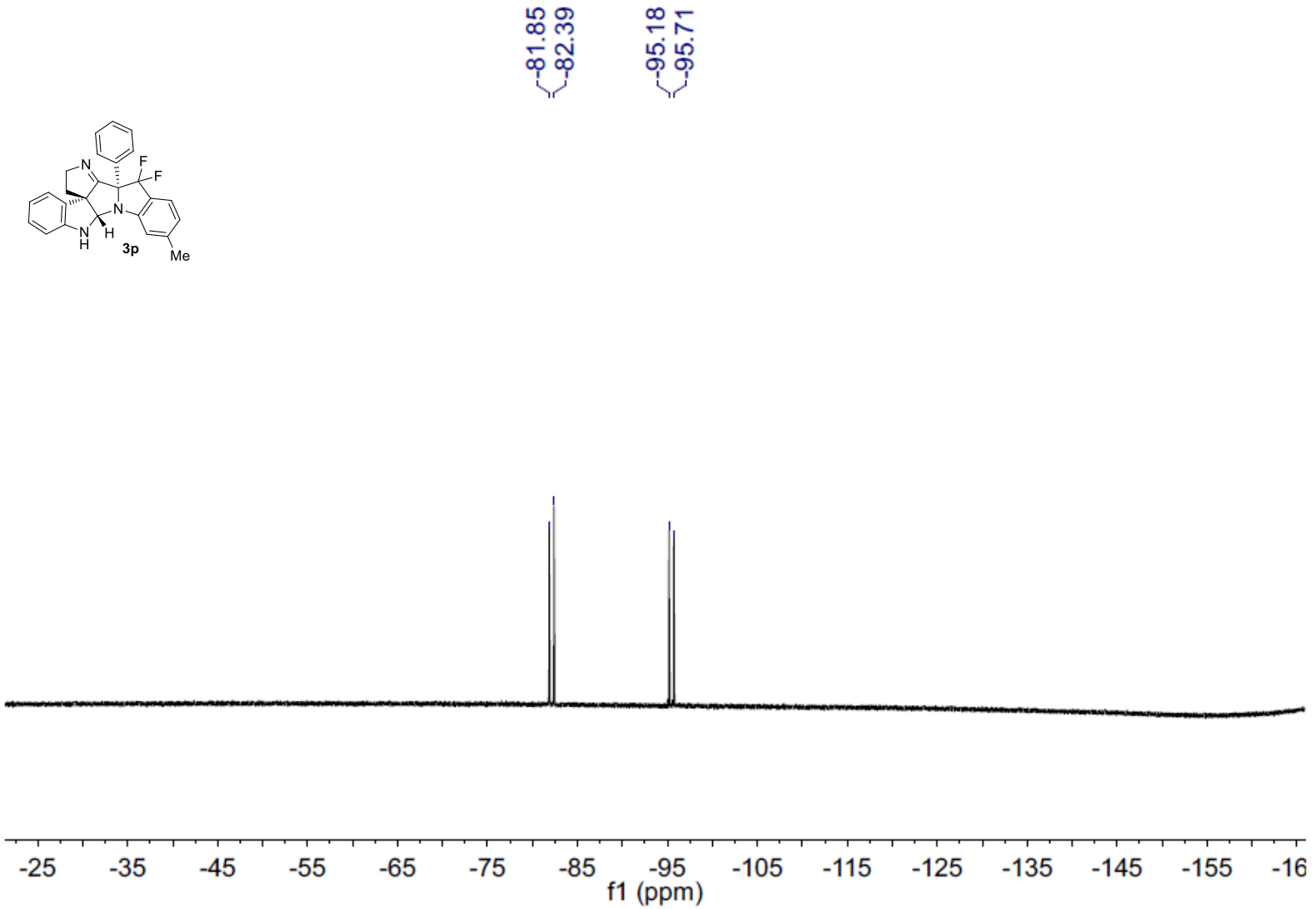
127.80
127.48
126.30
125.68
124.34
123.74
123.62
123.18
122.68
121.68
119.76
119.56
119.54
119.48
119.34
77.84
77.68
77.60
77.43
71.33
66.90
39.09
22.04
f1 (ppm)
3p
Me
F
F
N
H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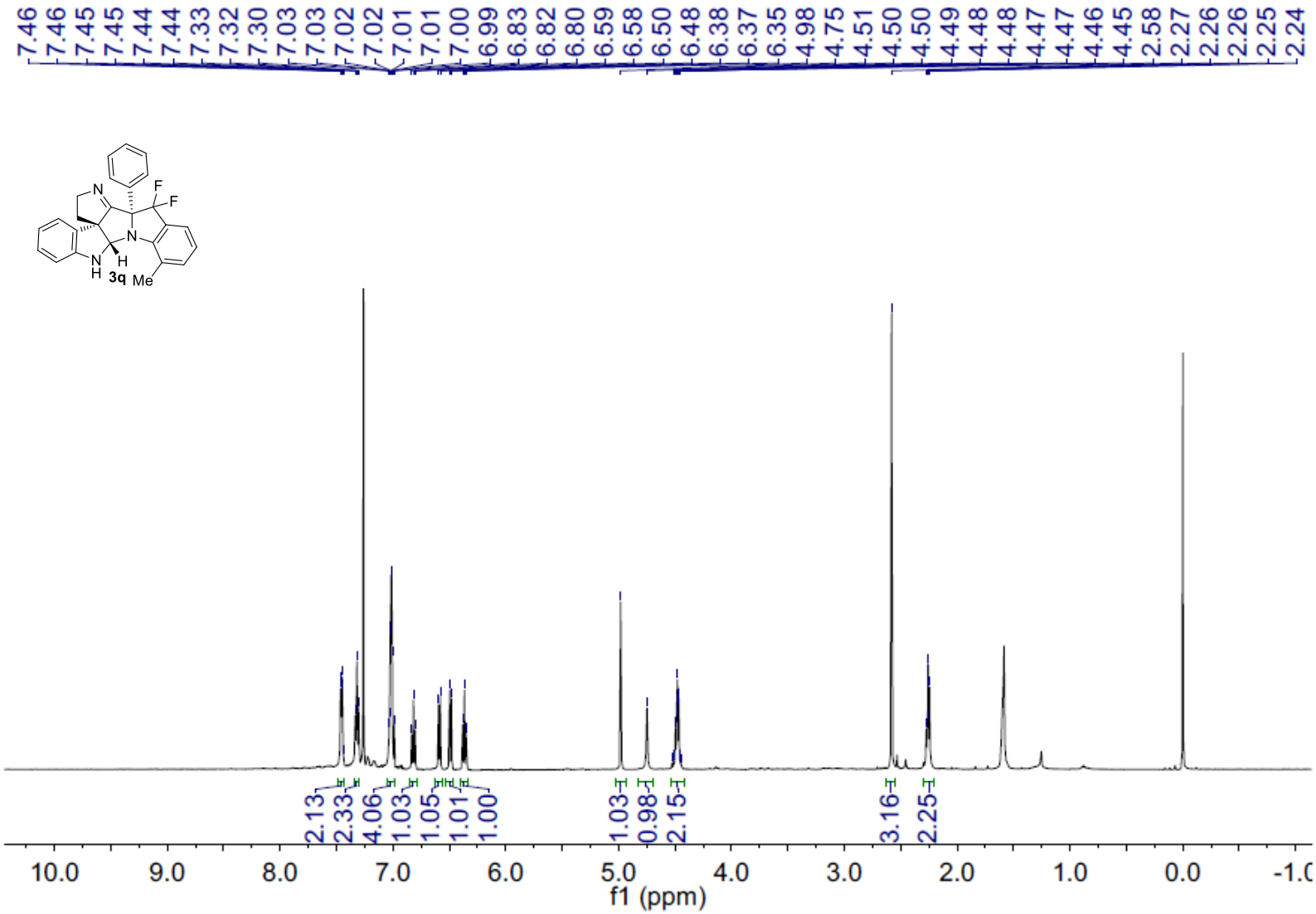
-95.18
-95.71
N
F
F
N
N
H
H
3p
Me
-25
-35
-45
-55
-65
-75
-85
-95
-105
-115
-125
-135
-145
-155
-16
f1 (ppm)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7.03
7.02
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7.01
7.01
7.00
6.99
6.83
6.82
6.80
6.59
6.58
6.50
6.48
6.38
6.37
6.35
4.98
4.75
4.51
4.50
4.50
4.49
4.48
4.48
4.47
4.47
4.46
4.45
2.58
2.27
2.26
2.26
2.25
2.24
N
F
F
N
N
H
H
3q
Me
2.13
2.33
4.06
1.03
1.05
1.01
1.00
1.03
0.98
2.15
3.16
2.25
10.0
9.0
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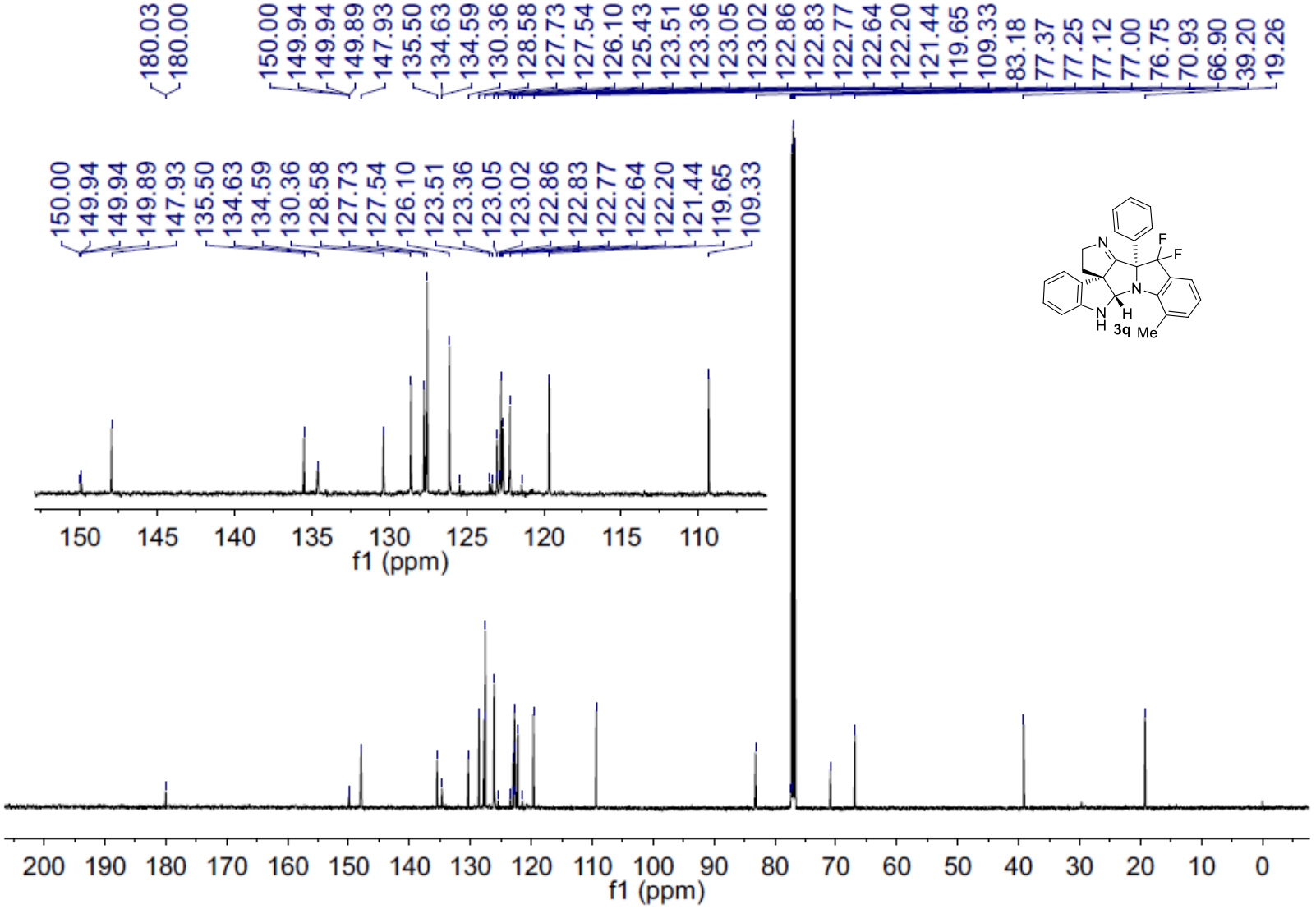
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)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147.93
135.50
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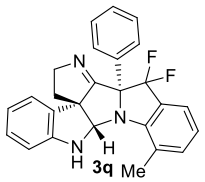
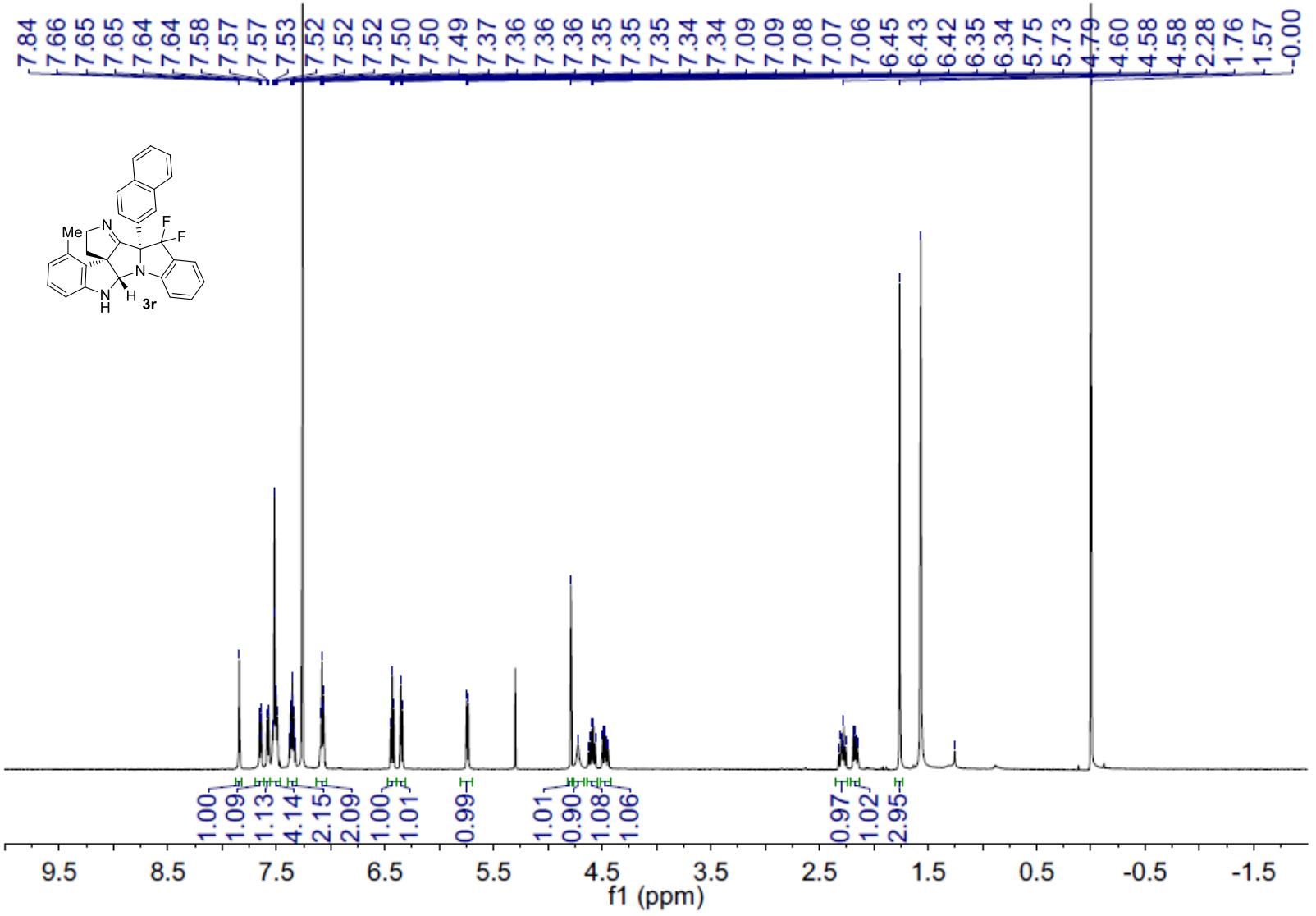
134.59
130.36
128.58
127.73
127.54
126.10
125.43
123.51
123.36
123.05
123.02
122.86
122.83
122.77
122.64
122.20
121.44
119.65
109.33
83.18
77.37
77.25
77.12
77.00
76.75
70.93
66.90
39.20
19.26
F
F
N
N
H
N
H
3q Me
f1 (ppm)
f1 (ppm)


N
F
F
N
N
H
H
3q Me

-80.48
-81.02
-94.64
-95.18
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)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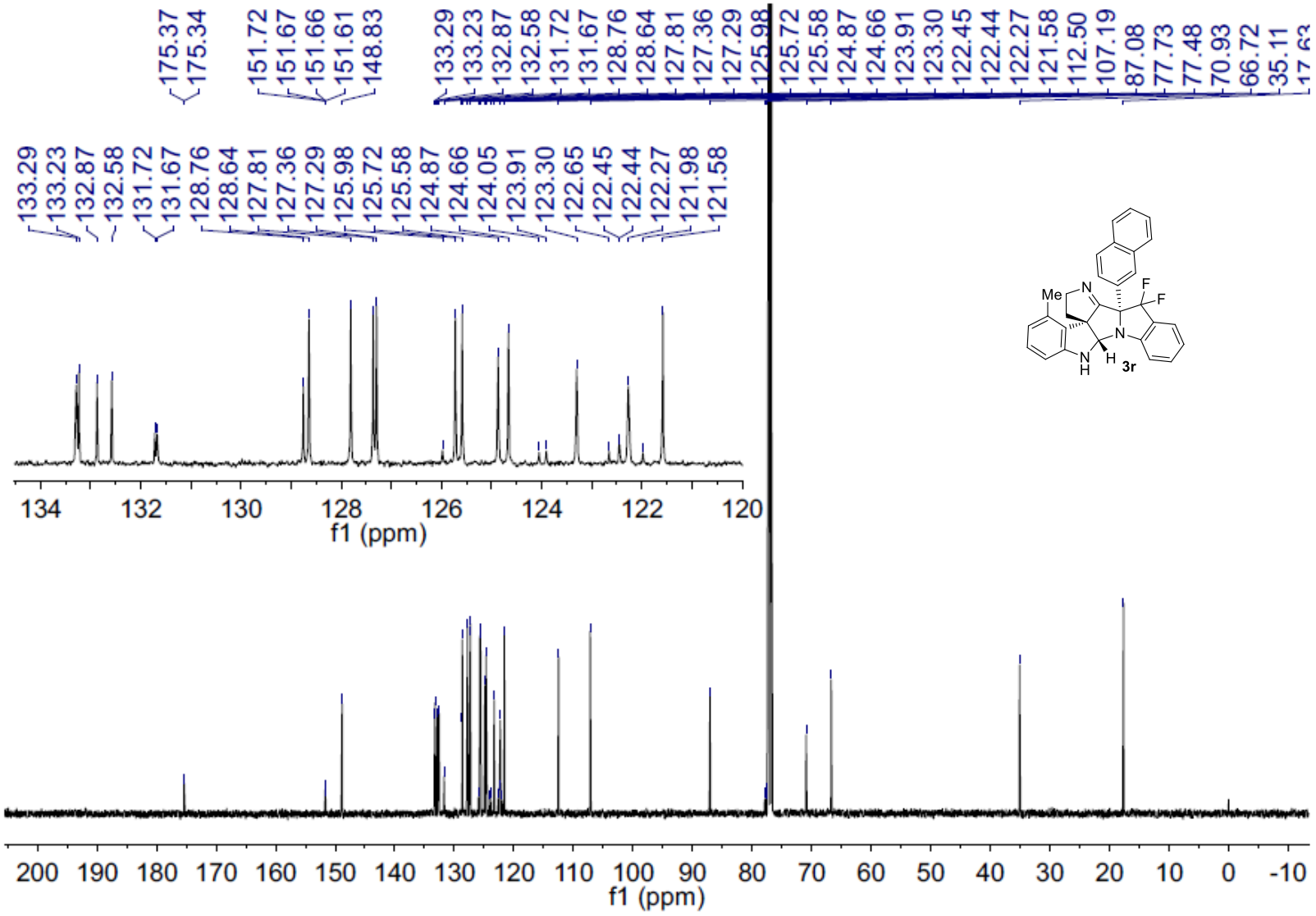
7.66
7.65
7.65
7.64
7.64
7.58
7.57
7.57
7.53
7.52
7.52
7.52
7.50
7.50
7.49
7.37
7.36
7.36
7.36
7.35
7.35
7.35
7.34
7.34
7.09
7.09
7.08
7.07
7.06
6.45
6.43
6.42
6.35
6.34
5.75
5.73
4.79
4.60
4.58
4.58
2.28
1.76
1.57
-0.00
Me
N
F
F
N
N
H
H
3r
1.00
1.09
1.13
4.14
2.15
2.09
1.00
1.01
0.99
1.01
0.90
1.08
1.06
0.97
1.02
2.95
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)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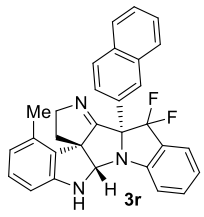
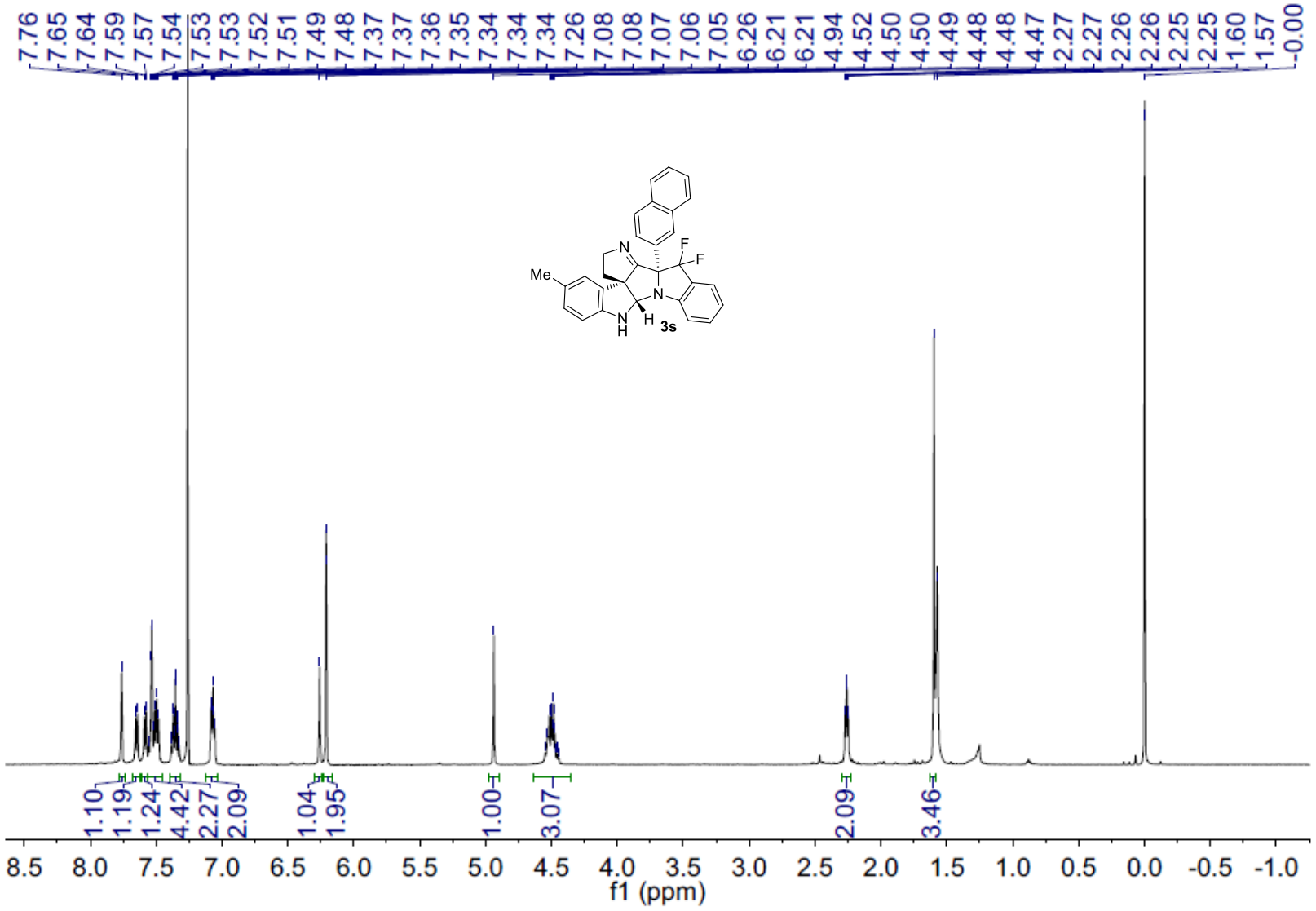
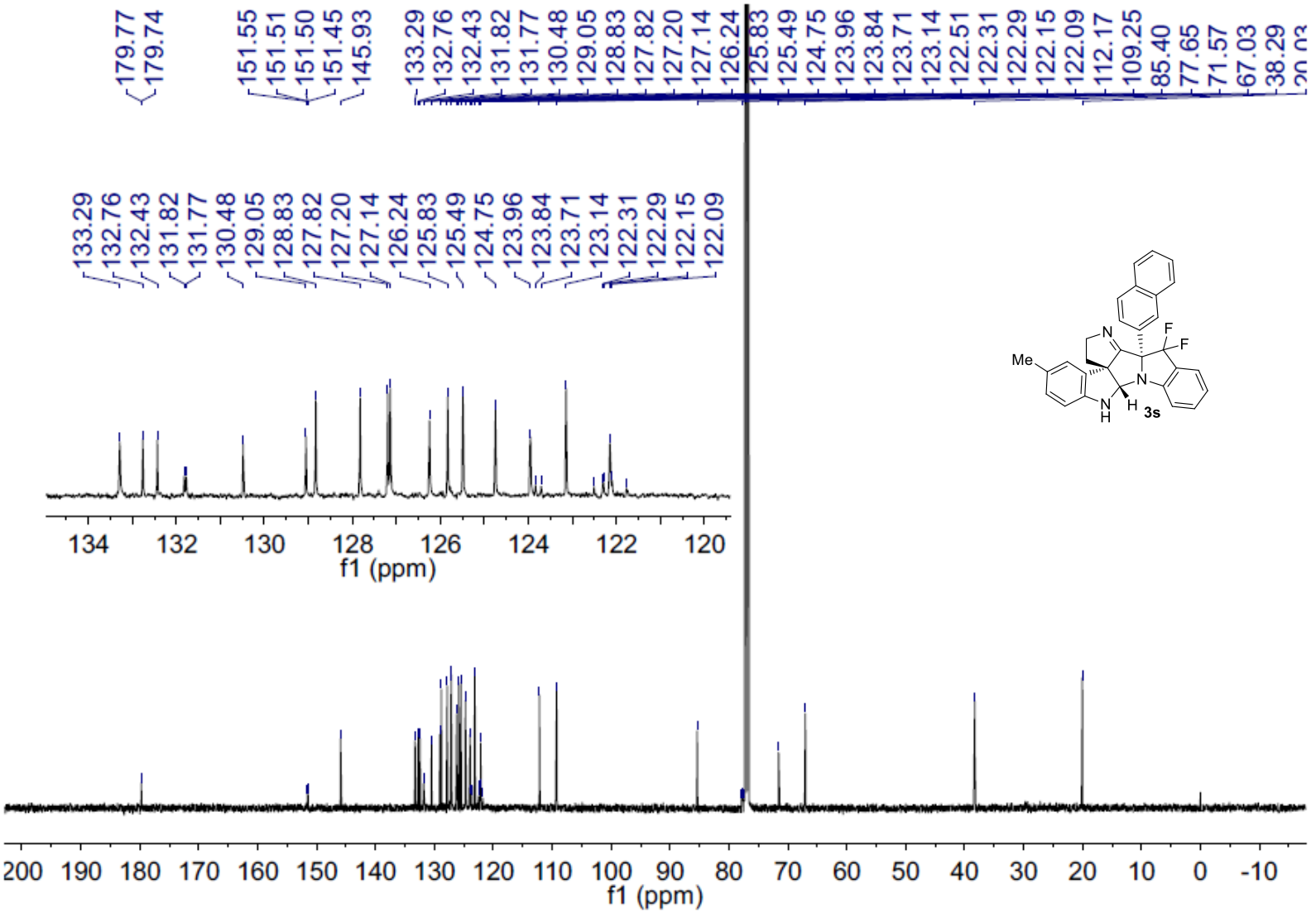
175.34
151.72
151.67
151.66
151.61
148.83
133.29
133.23
132.87
132.58
131.72
131.67
128.76
128.64
127.81
127.36
127.29
125.98
125.72
125.58
124.87
124.66
124.05
123.91
123.30
122.65
122.45
122.44
122.27
121.98
121.58
Me
N
F
F
N
N
H
3r
134
132
130
128
126
124
122
120
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


Me
N
F
F
N
N
H
H
3r

-79.45
-79.99
-95.52
-96.06
-50
-54
-58
-62
-66
-70
-74
-78
-82
-86
-90
-94
-98
-102
-106
-110
-114
-11
f1 (ppm)


7.76
7.65
7.64
7.59
7.57
7.54
7.53
7.53
7.52
7.51
7.49
7.48
7.37
7.37
7.36
7.35
7.34
7.34
7.34
7.26
7.08
7.08
7.07
7.06
7.05
6.26
6.21
6.21
4.94
4.52
4.50
4.50
4.49
4.48
4.48
4.47
2.27
2.27
2.26
2.26
2.25
2.25
1.60
1.57
-0.00
Me
N
F
F
N
N
H
H
3s
1.10
1.19
1.24
4.42
2.27
2.09
1.04
1.95
1.00
3.07
2.09
3.46
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


179.77
179.74
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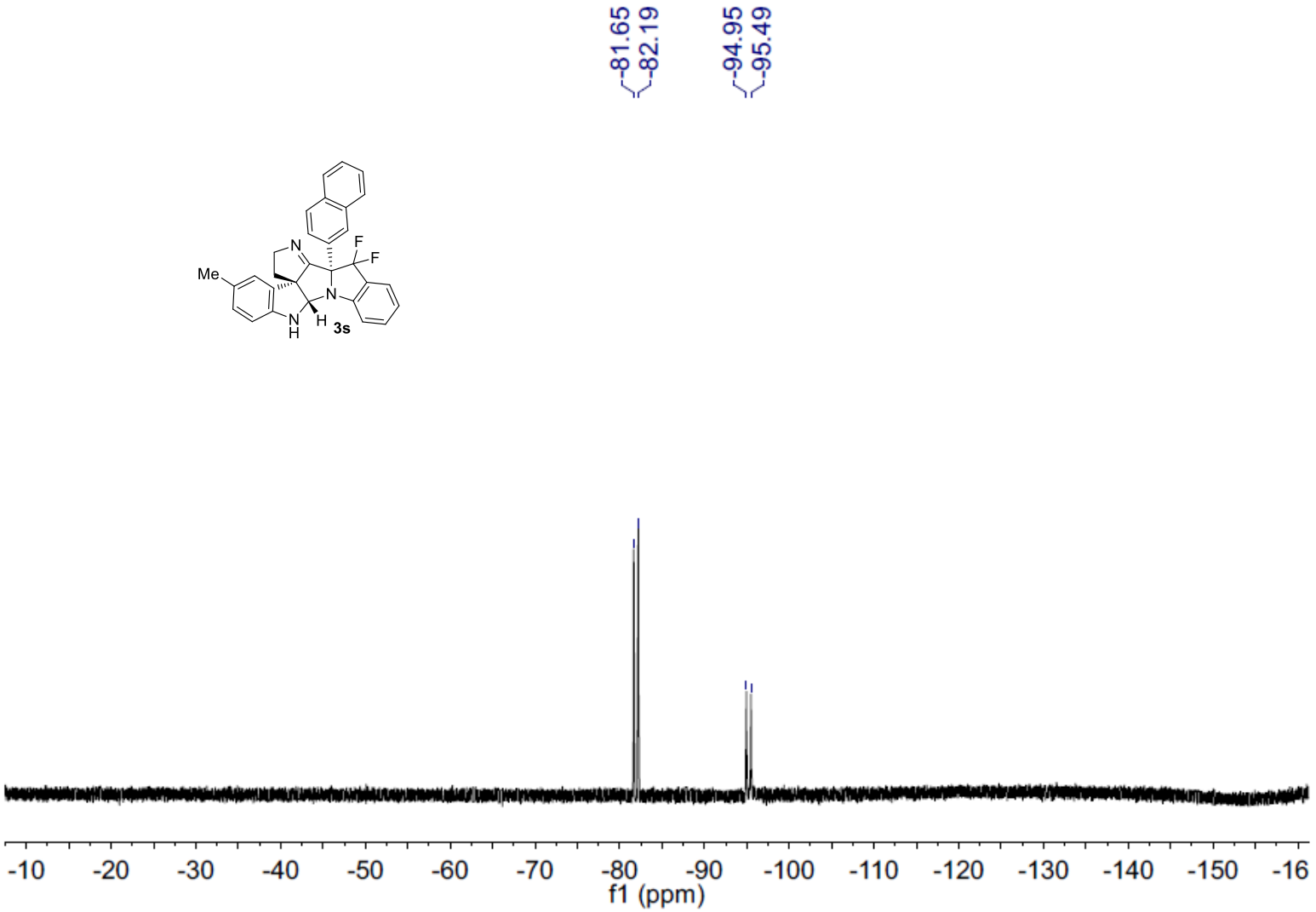
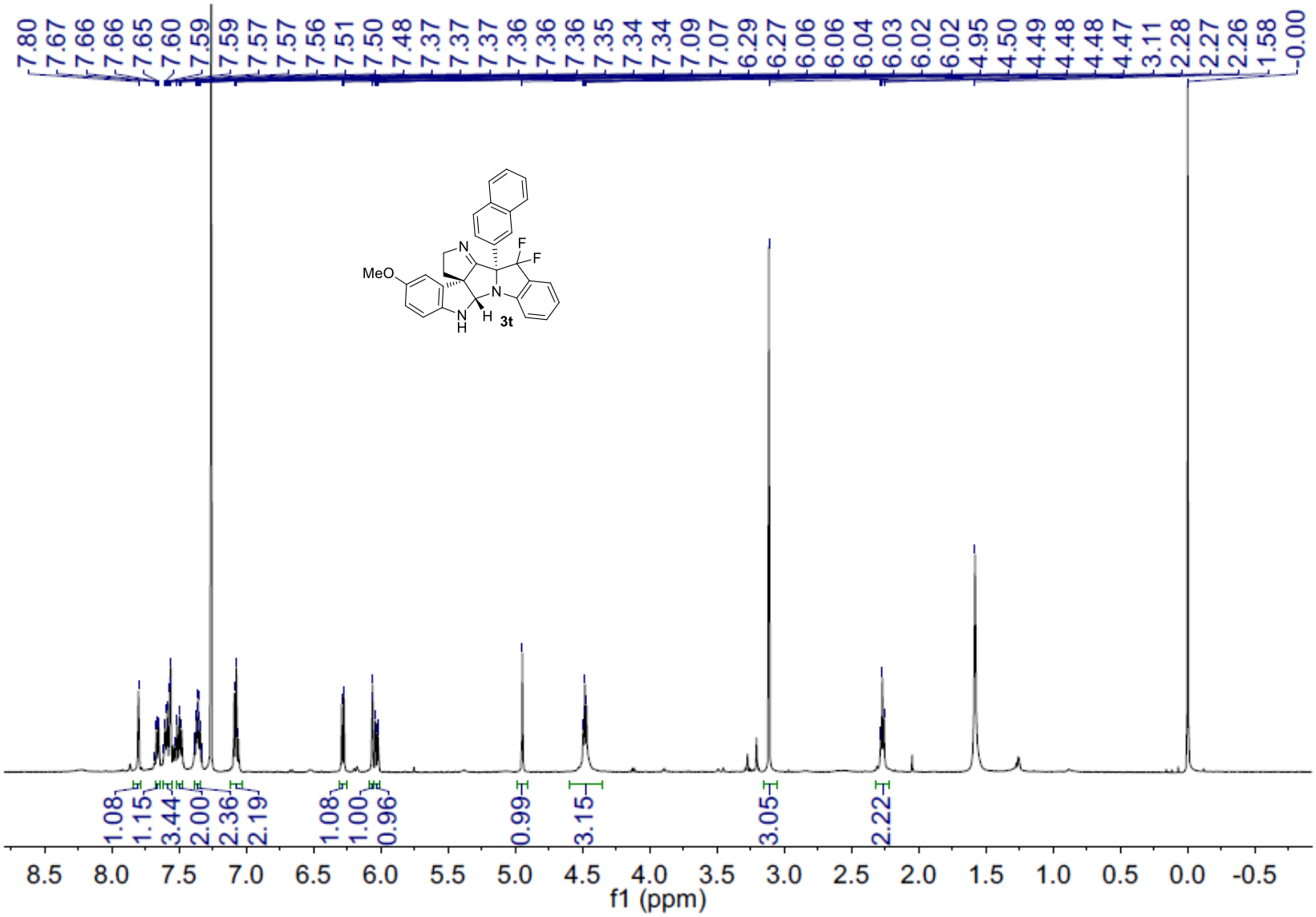
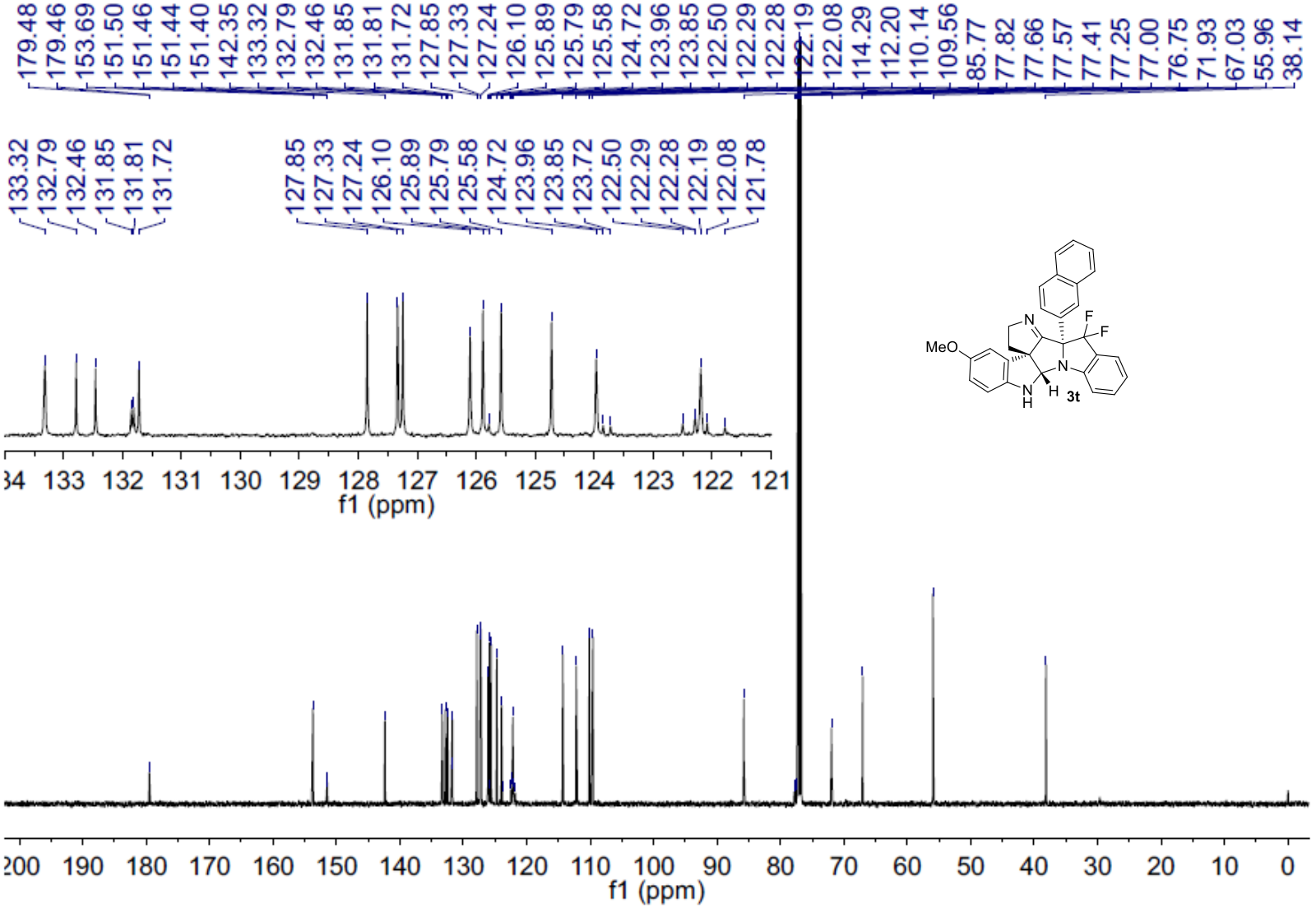
151.51
151.50
151.45
145.93
133.29
132.76
132.43
131.82
131.77
130.48
129.05
128.83
127.82
127.20
127.14
126.24
125.83
125.49
124.75
123.96
123.84
123.71
123.14
122.31
122.29
122.15
122.09
f1 (ppm)
Me
F
F
N
N
H
H
3s


-81.65
-82.19
-94.95
-95.49
Me
F
F
N
N
H
N
H
3s
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-16
f1 (ppm)


7.80 7.67 7.66 7.66 7.65 7.60 7.59 7.59 7.57 7.57 7.56 7.51 7.50 7.48 7.37 7.37 7.37 7.36 7.36 7.36 7.35 7.34 7.34 7.09 7.07 6.29 6.27 6.06 6.06 6.04 6.03 6.02 6.02 4.95 4.50 4.49 4.48 4.48 4.47 3.11 2.28 2.27 2.26 1.58 0.00
MeO
N
F
F
N
N
H
H
3t
1.08 1.15 3.44 2.00 2.36 2.19 1.08 1.00 0.96 0.99 3.15 3.05 2.22
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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153.69
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151.46
151.44
151.40
142.35
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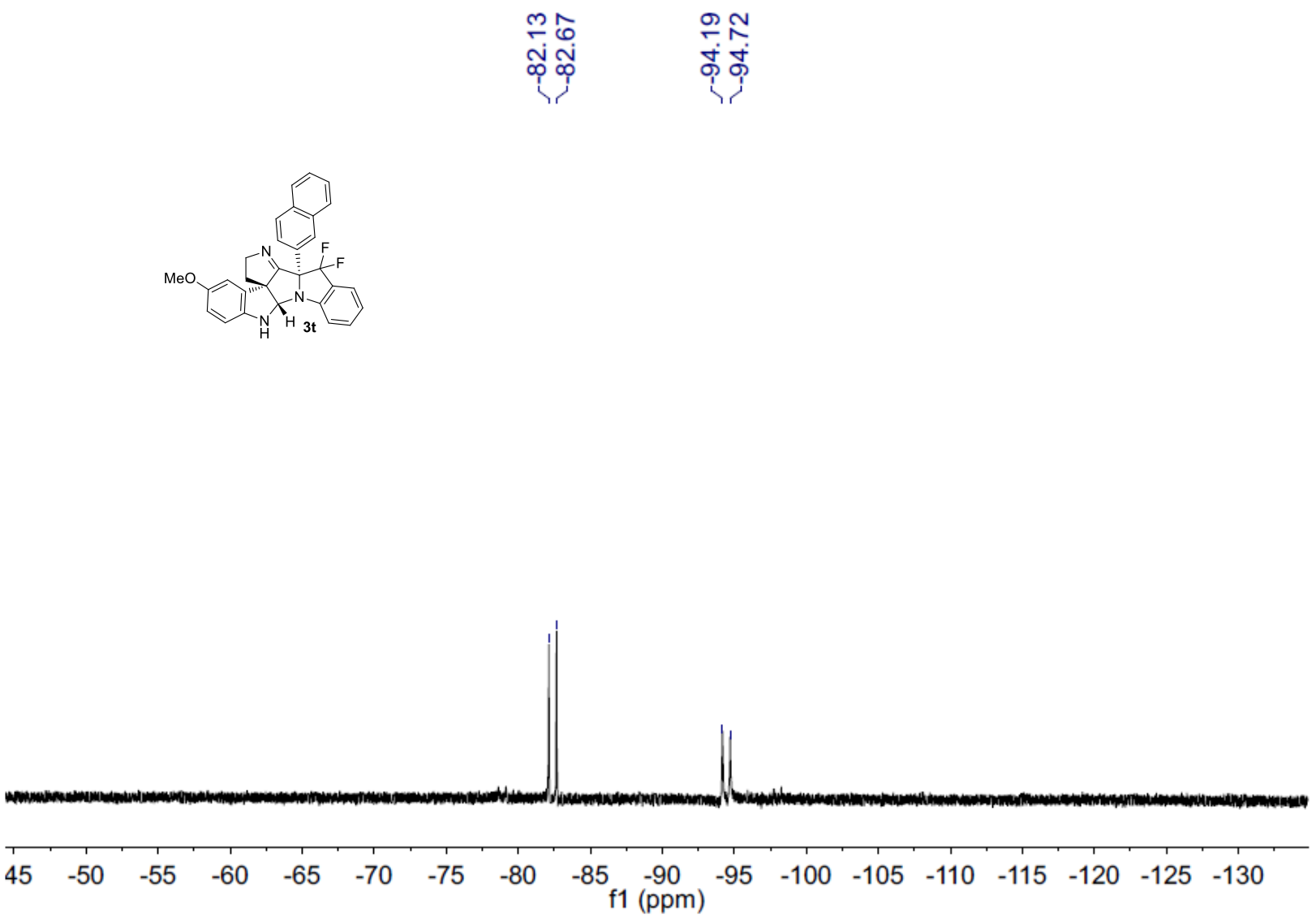
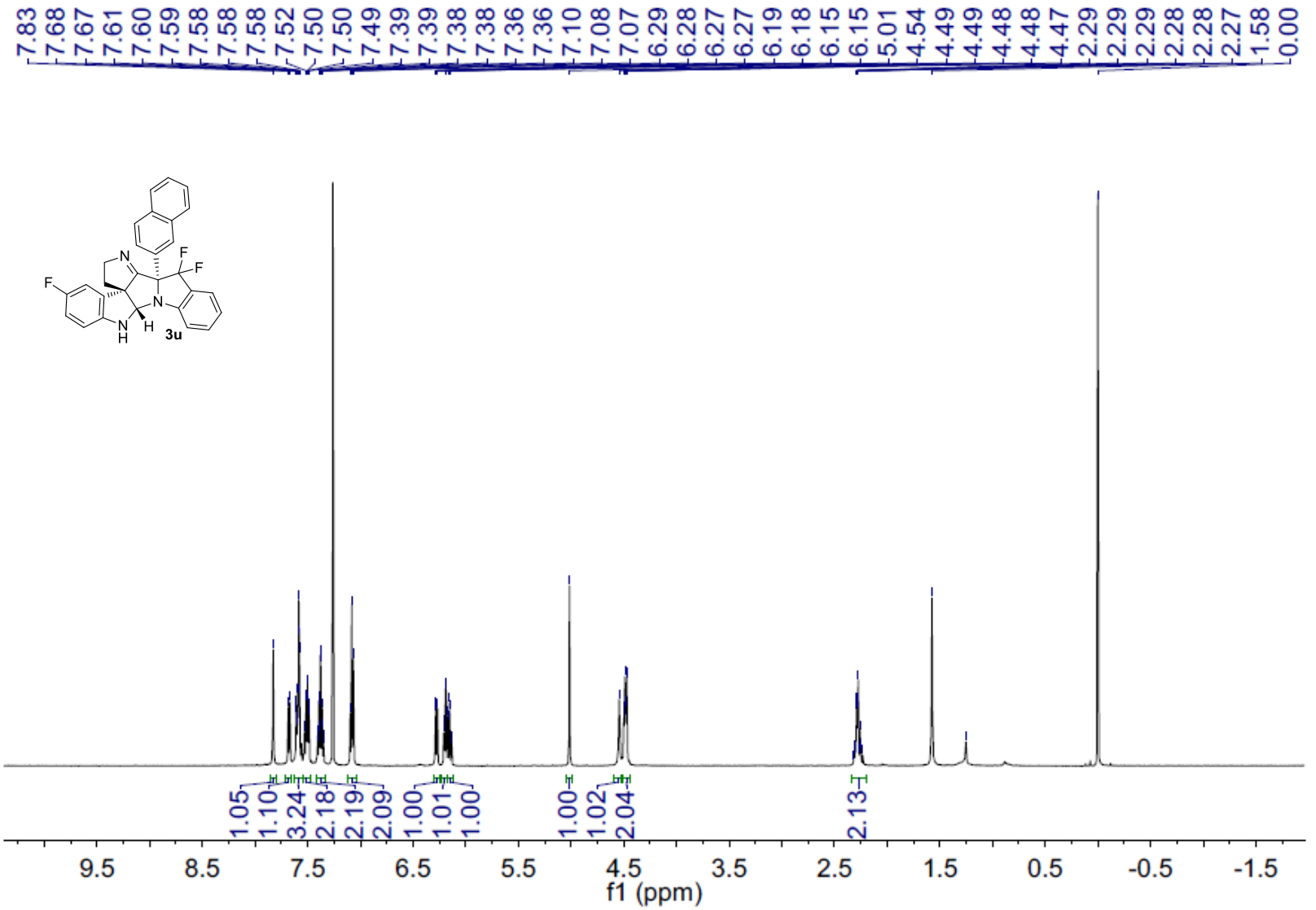
132.79
132.46
131.85
131.81
131.72
127.85
127.33
127.24
126.10
125.89
125.79
125.58
124.72
123.96
123.85
123.72
122.50
122.29
122.28
122.19
122.08
121.78
114.29
112.20
110.14
109.56
85.77
77.82
77.66
77.57
77.41
77.25
77.00
76.75
71.93
67.03
55.96
38.14
MeO
F
F
N
N
H
H
3t
f1 (ppm)
f1 (ppm)


-82.13
-82.67
-94.19
-94.72
MeO
F
F
N
N
H
N
H
3t
45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
f1 (ppm)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7.50
7.50
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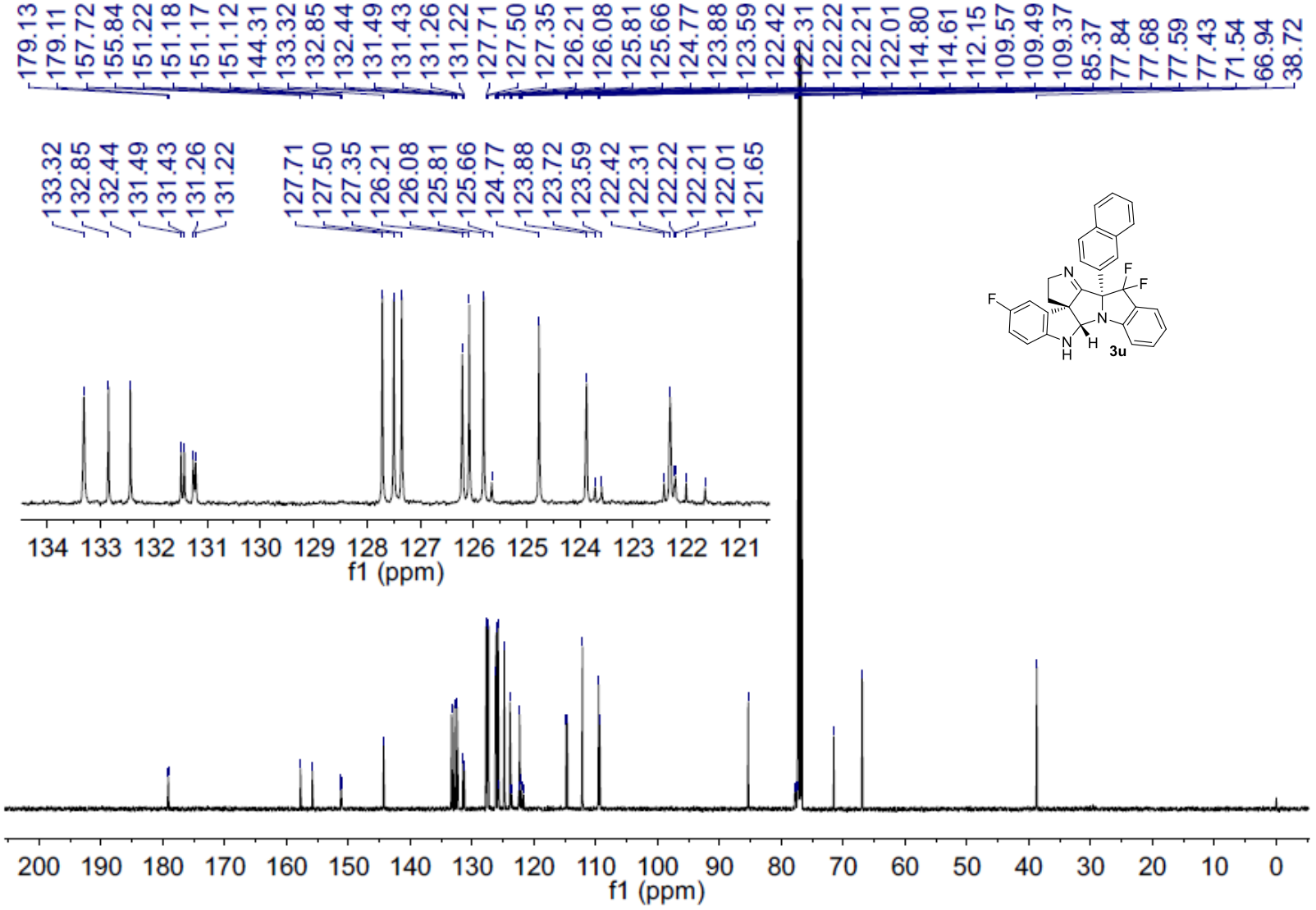
7.39
7.39
7.38
7.38
7.36
7.36
7.10
7.08
7.07
6.29
6.28
6.27
6.27
6.19
6.18
6.15
6.15
5.01
4.54
4.49
4.49
4.48
4.48
4.47
2.29
2.29
2.29
2.28
2.28
2.27
1.58
0.00
F
F
F
N
N
H
N
H
3u
1.05
1.10
3.24
2.18
2.19
2.09
1.00
1.01
1.00
1.00
1.02
2.04
2.13
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)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151.18
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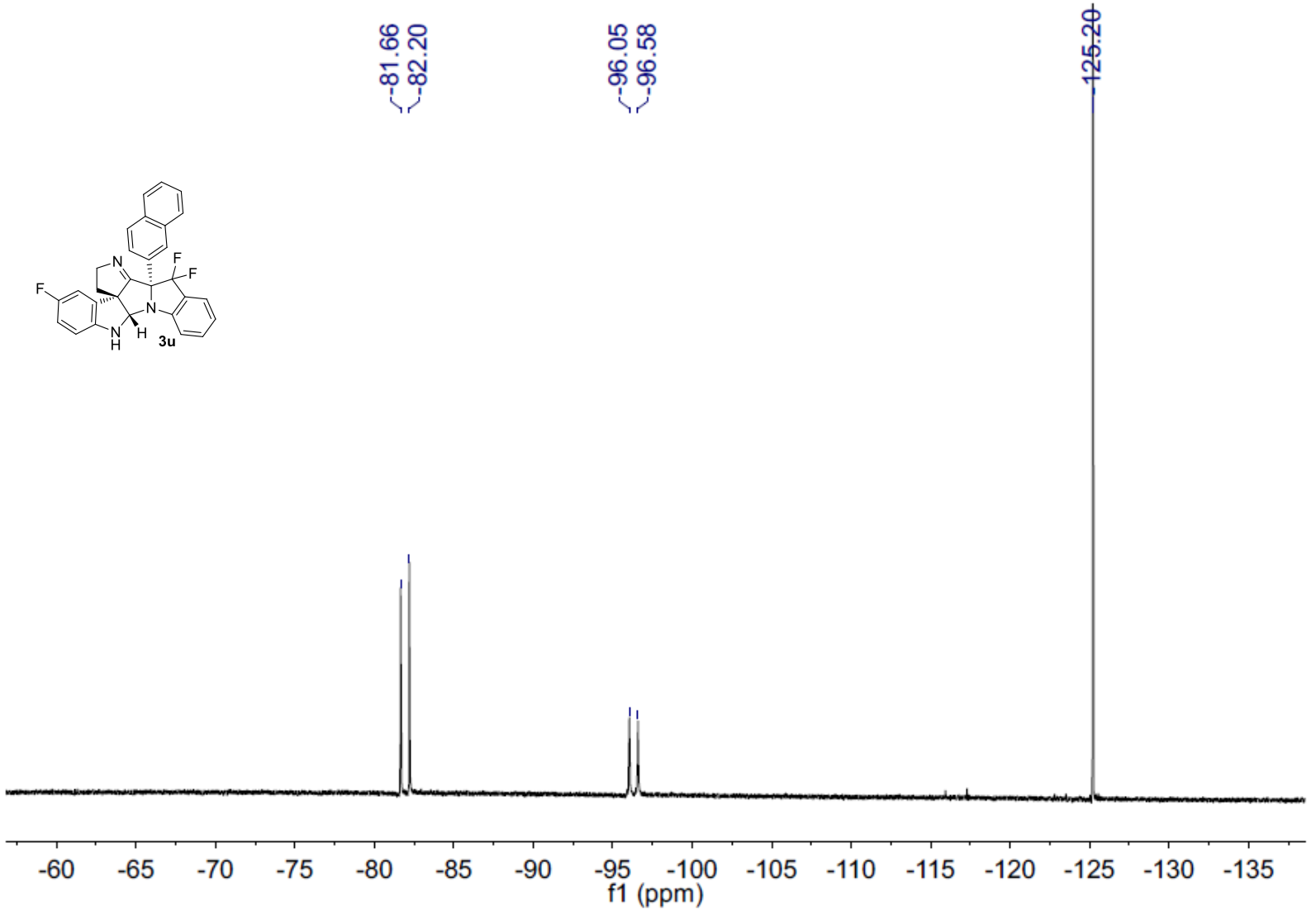
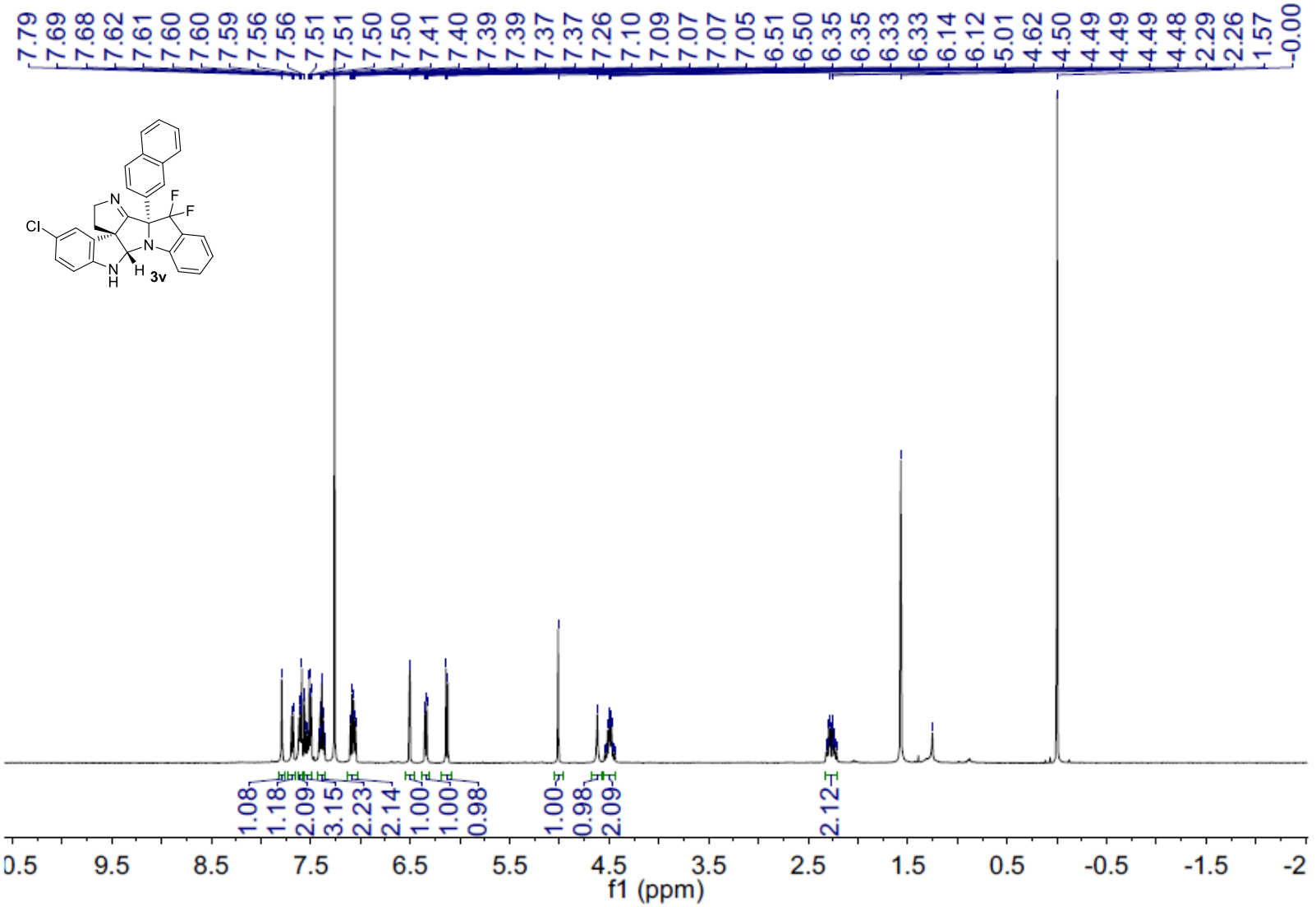
151.12
144.31
133.32
132.85
132.44
131.49
131.43
131.26
131.22
127.71
127.50
127.35
126.21
126.08
125.81
125.66
124.77
123.88
123.59
122.42
122.31
122.22
122.21
122.01
114.80
114.61
112.15
109.57
109.49
109.37
85.37
77.84
77.68
77.59
77.43
71.54
66.94
38.72
133.32
132.85
132.44
131.49
131.43
131.26
131.22
127.71
127.50
127.35
126.21
126.08
125.81
125.66
124.77
123.88
123.72
123.59
122.42
122.31
122.22
122.21
122.01
121.65
134 133 132 131 130 129 128 127 126 125 124 123 122 121
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
F
N
F
F
N
N
H
H
3u


-81.66
-82.20
-96.05
-96.58
-125.20
F
F
F
N
N
H
N
H
3u
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
-135
f1 (ppm)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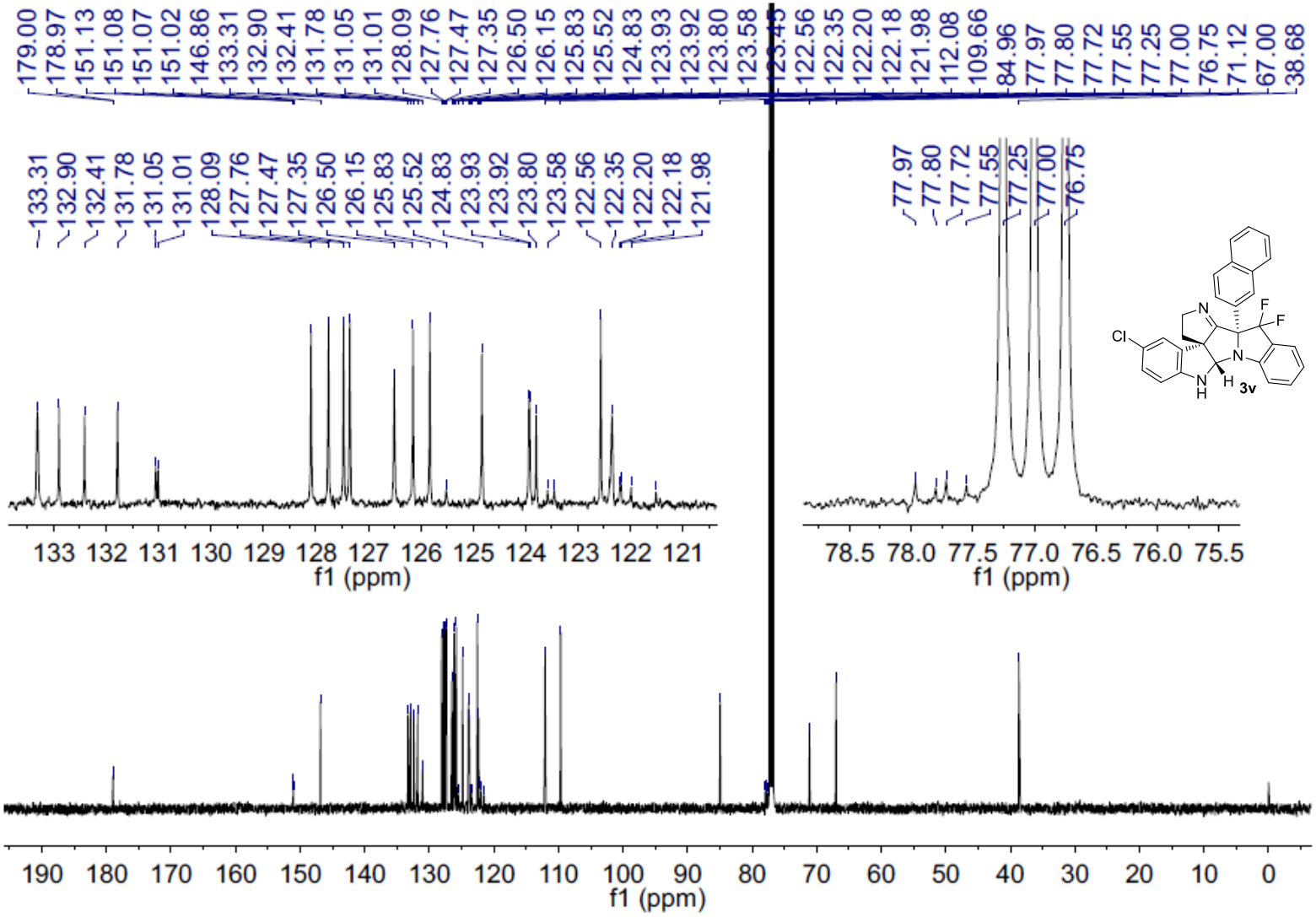
7.60
7.60
7.59
7.56
7.56
7.51
7.51
7.50
7.50
7.41
7.40
7.39
7.39
7.37
7.37
7.26
7.10
7.09
7.07
7.07
7.05
6.51
6.50
6.35
6.35
6.33
6.33
6.14
6.12
5.01
4.62
4.50
4.49
4.49
4.49
4.48
2.29
2.26
1.57
-0.00
Cl
N
F
F
N
N
H
H
3v
1.08
1.18
2.09
3.15
2.23
2.14
1.00
1.00
0.98
1.00
0.98
2.09
2.12
0.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
-2
f1 (ppm)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151.07
151.02
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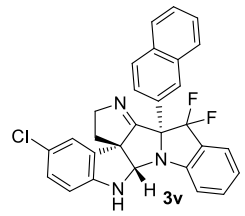
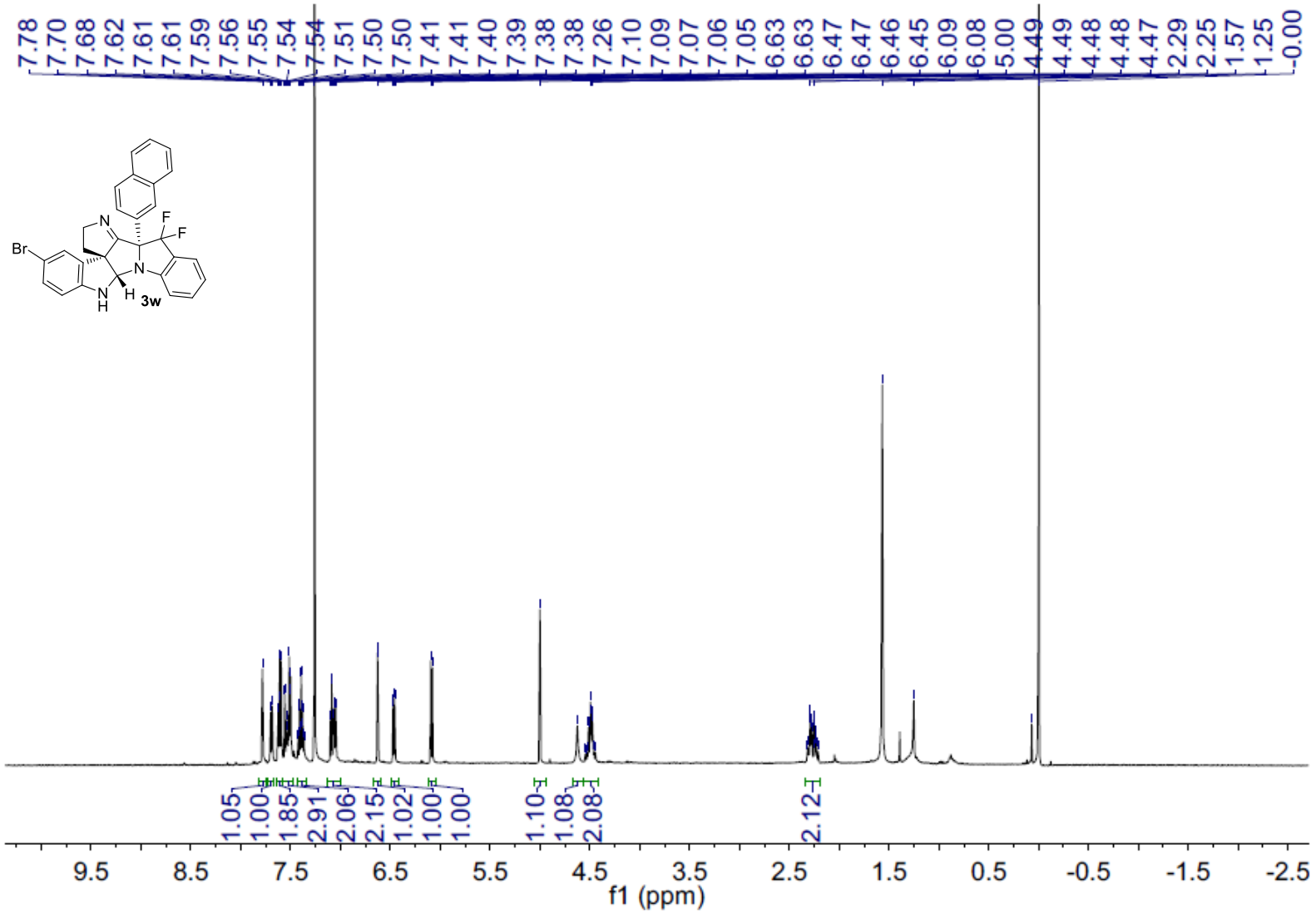
133.31
132.90
132.41
131.78
131.05
131.01
128.09
127.76
127.47
127.35
126.50
126.15
125.83
125.52
124.83
123.93
123.92
123.80
123.58
123.45
122.56
122.35
122.20
122.18
121.98
112.08
109.66
84.96
77.97
77.80
77.72
77.55
77.25
77.00
76.75
71.12
67.00
38.68
133 132 131 130 129 128 127 126 125 124 123 122 121
f1 (ppm)
78.5 78.0 77.5 77.0 76.5 76.0 75.5
f1 (ppm)
Cl
F
F
N
N
H
H
3v
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Cl
N
F
F
N
H
N
H
3v

-80.65
-81.20
-97.62
-98.17
35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140
f1 (ppm)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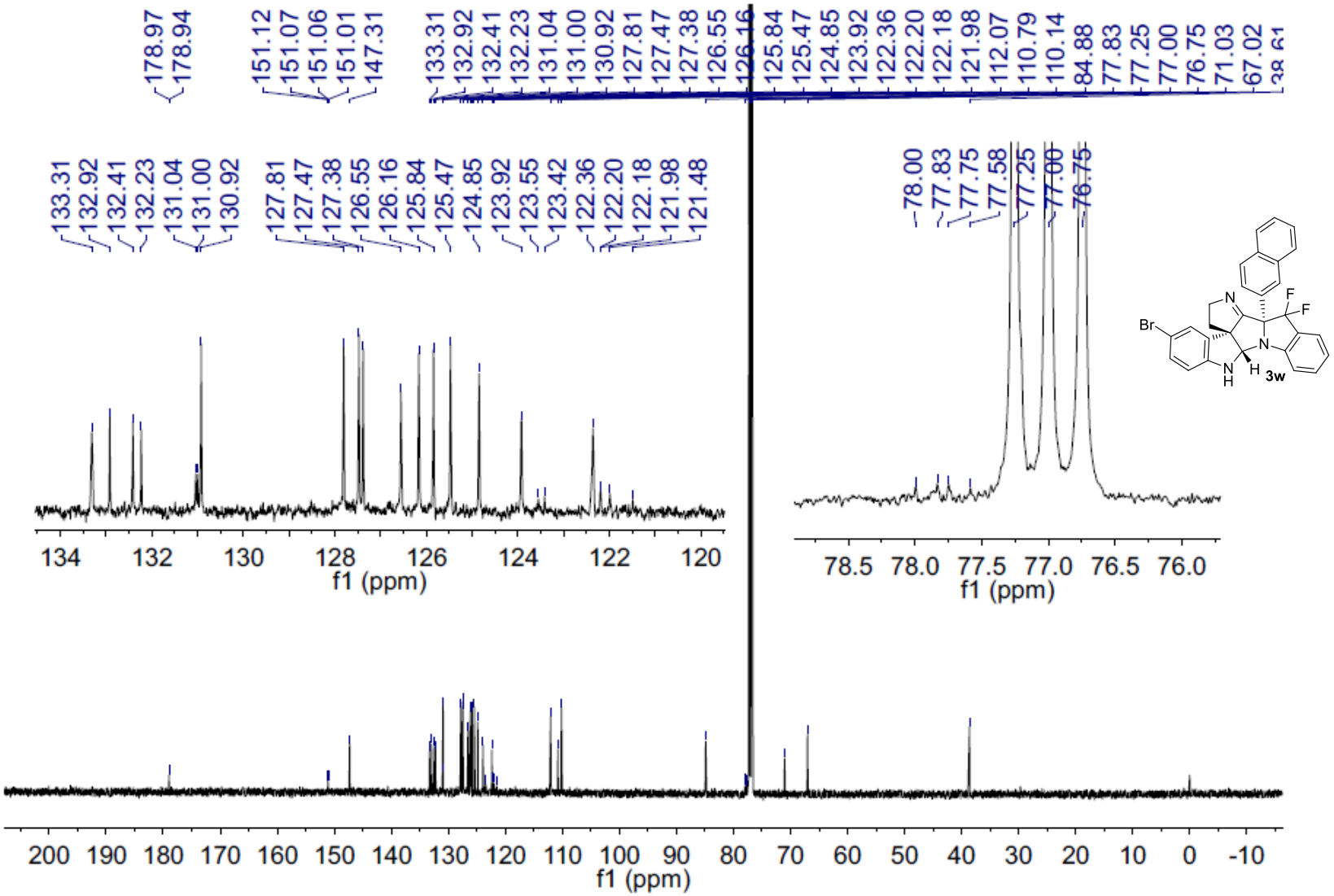
7.50
7.41
7.41
7.40
7.39
7.38
7.38
7.26
7.10
7.09
7.07
7.06
7.05
6.63
6.63
6.47
6.47
6.46
6.45
6.09
6.08
5.00
4.49
4.49
4.48
4.48
4.47
2.29
2.25
1.57
1.25
0.00
Br
N
F
F
N
N
H
H
3w
1.05
1.00
1.85
2.91
2.06
2.15
1.02
1.00
1.00
1.10
1.08
2.08
2.12
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
-2.5
f1 (ppm)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132.41
132.23
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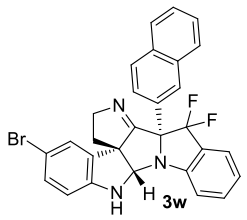
131.00
130.92
127.81
127.47
127.38
126.55
126.16
125.84
125.47
124.85
123.92
123.55
123.42
122.36
122.20
122.18
121.98
121.48
78.00
77.83
77.75
77.58
77.25
77.00
76.75
Br
F
F
N
N
H
N
H
3w
134
132
130
128
126
124
122
120
f1 (ppm)
78.5
78.0
77.5
77.0
76.5
76.0
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


Br
N
F
F
N
N
H
H
3w

-80.50
-81.05
-97.83
-98.38
-25
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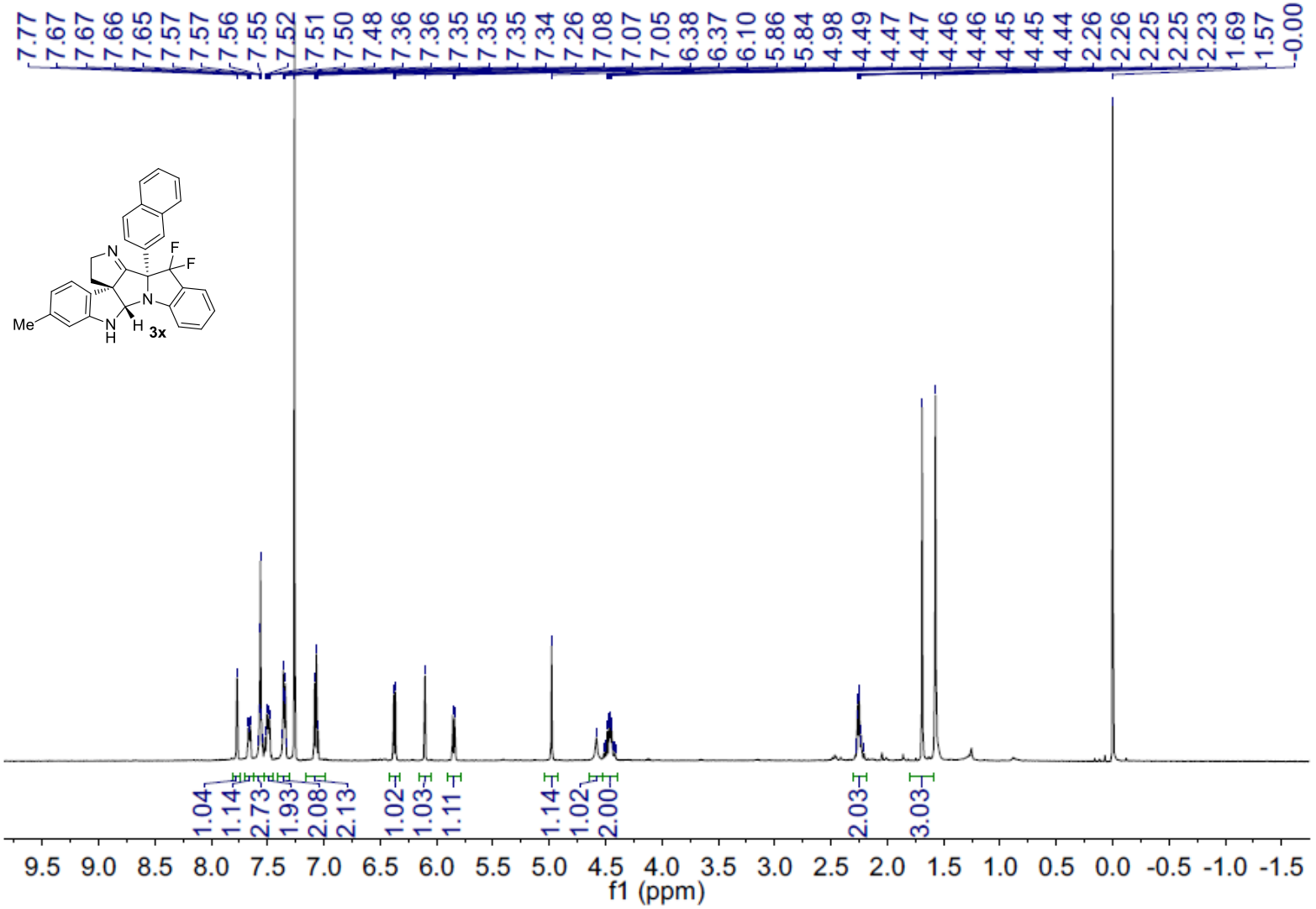
-45
-55
-65
-75
-85
-95
-105
-115
-125
-135
-145
-155
f1 (ppm)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7.52
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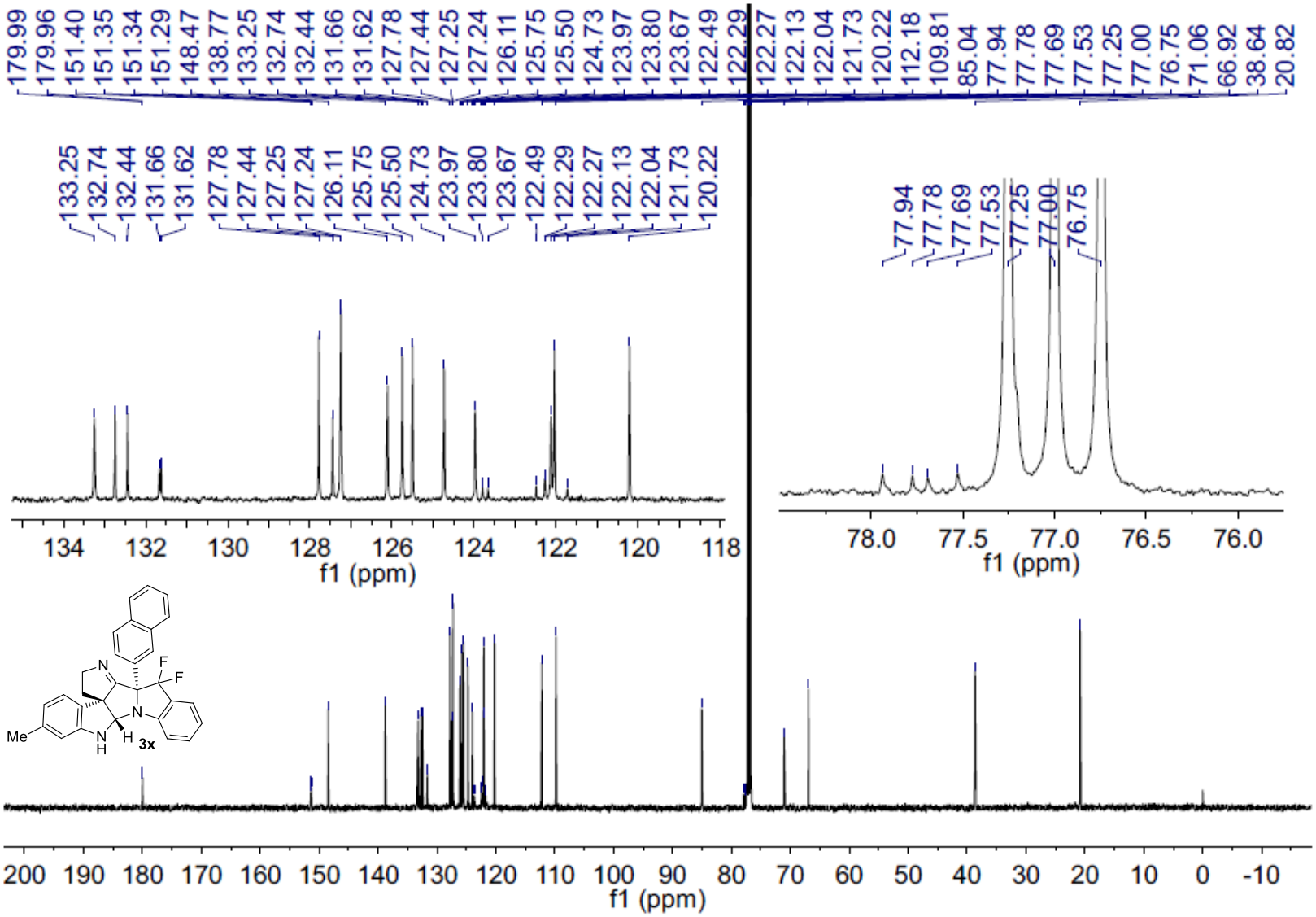
7.50
7.48
7.36
7.36
7.35
7.35
7.35
7.34
7.26
7.08
7.07
7.05
6.38
6.37
6.10
5.86
5.84
4.98
4.49
4.47
4.47
4.46
4.46
4.45
4.45
4.44
2.26
2.26
2.25
2.25
2.23
1.69
1.57
-0.00
Me
N
H
3x
F
F
N
1.04
1.14
2.73
1.93
2.08
2.13
1.02
1.03
1.11
1.14
1.02
2.00
2.03
3.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)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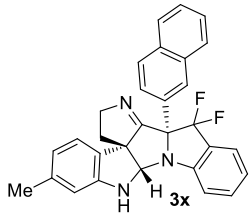
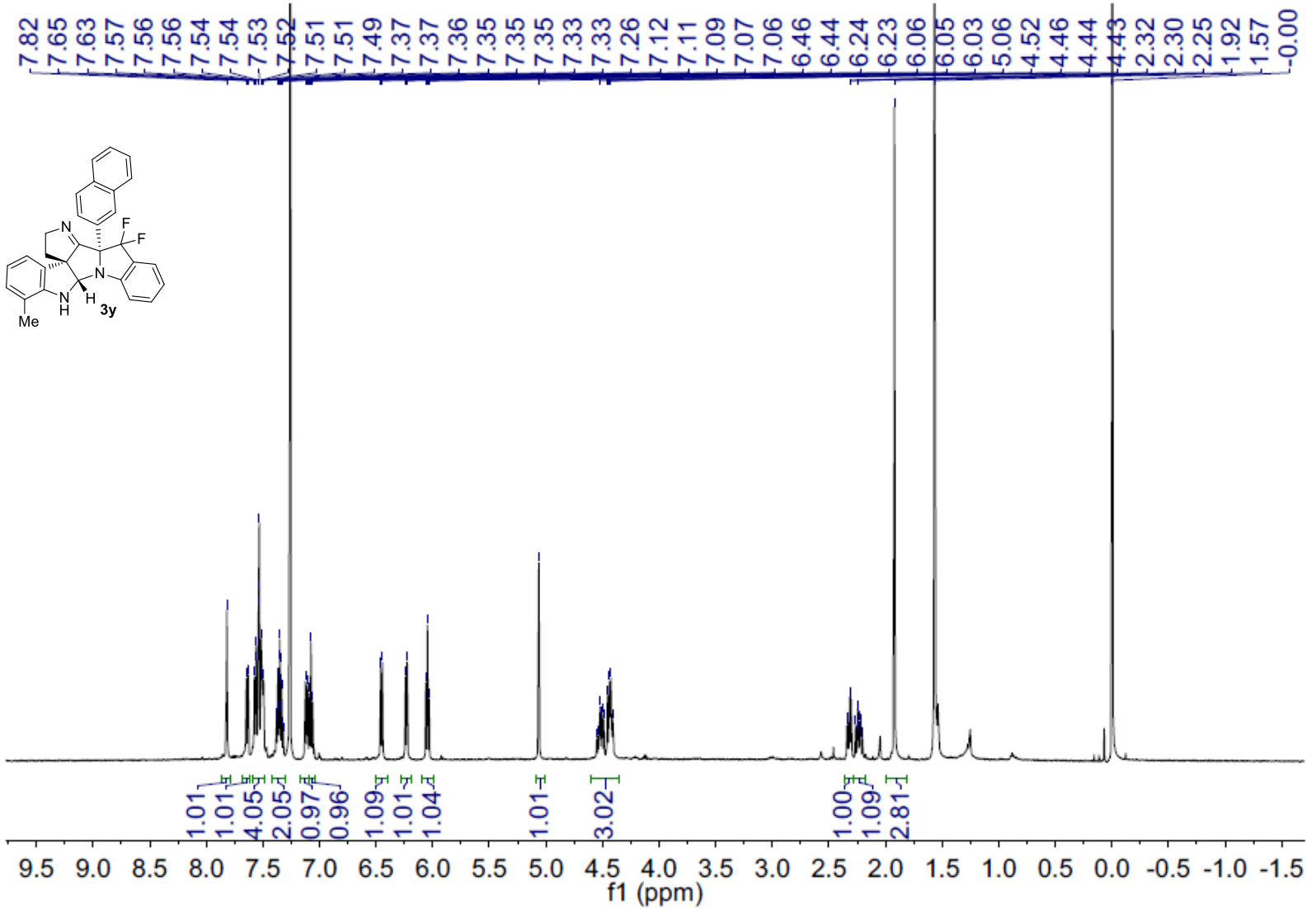
131.62
127.78
127.44
127.25
127.24
126.11
125.75
125.50
124.73
123.97
123.80
123.67
122.49
122.29
122.27
122.13
122.04
121.73
120.22
112.18
109.81
85.04
77.94
77.78
77.69
77.53
77.25
77.00
76.75
71.06
66.92
38.64
20.82
f1 (ppm)
f1 (ppm)
f1 (ppm)
Me
N
H
3x
F
F


Me
N
H
H
N
F
F
3x

-81.26
-81.80
-96.07
-96.60
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
f1 (ppm)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7.54
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7.52
7.51
7.51
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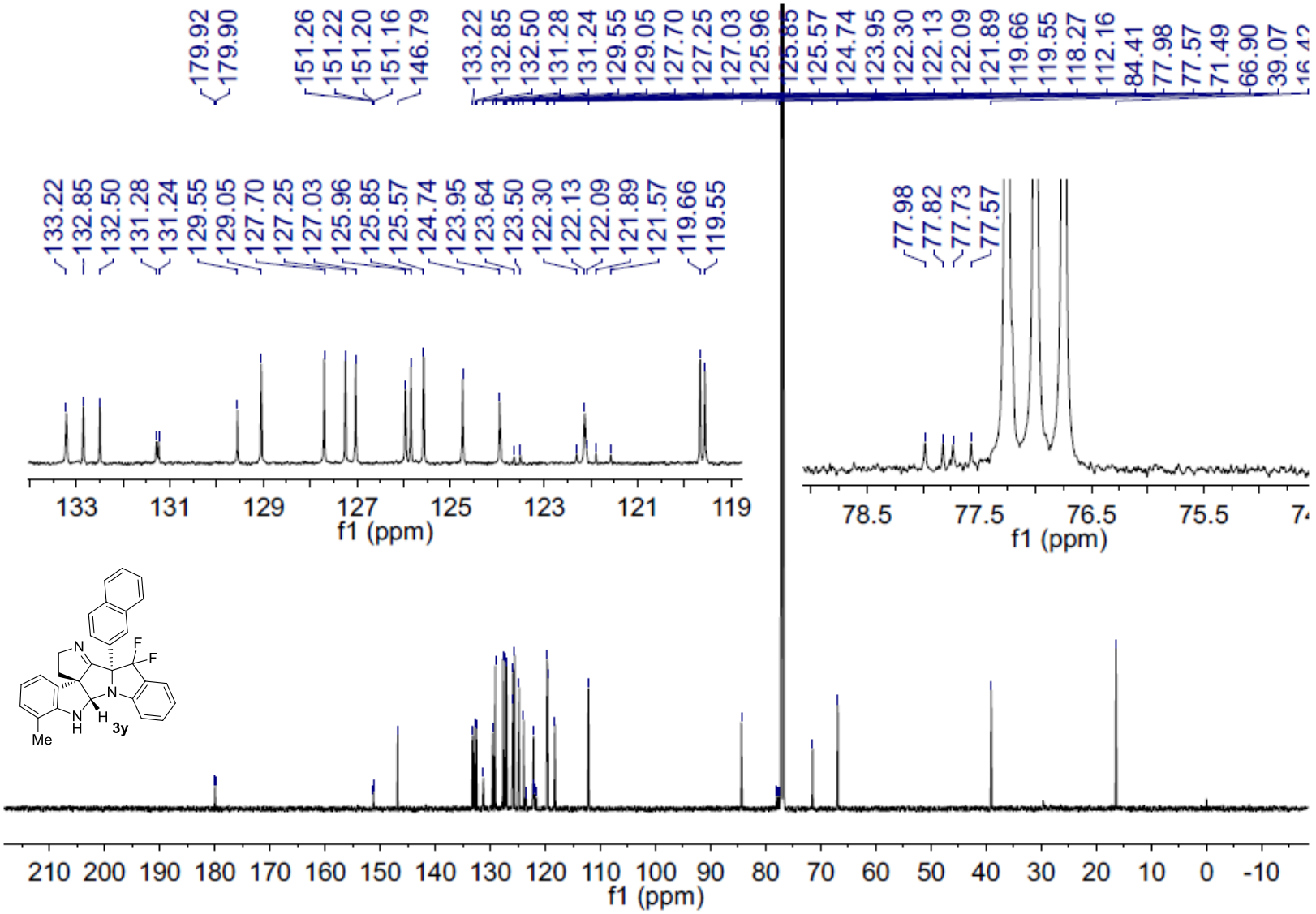
7.37
7.37
7.36
7.35
7.35
7.35
7.33
7.33
7.26
7.12
7.11
7.09
7.07
7.06
6.46
6.44
6.24
6.23
6.06
6.05
6.03
5.06
4.52
4.46
4.44
4.43
2.32
2.30
2.25
1.92
1.57
0.00
F
F
N
N
H
N
H
Me
3y
1.01
1.01
4.05
2.05
0.97
0.96
1.09
1.01
1.04
1.01
3.02
1.00
1.09
2.81
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)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179.90
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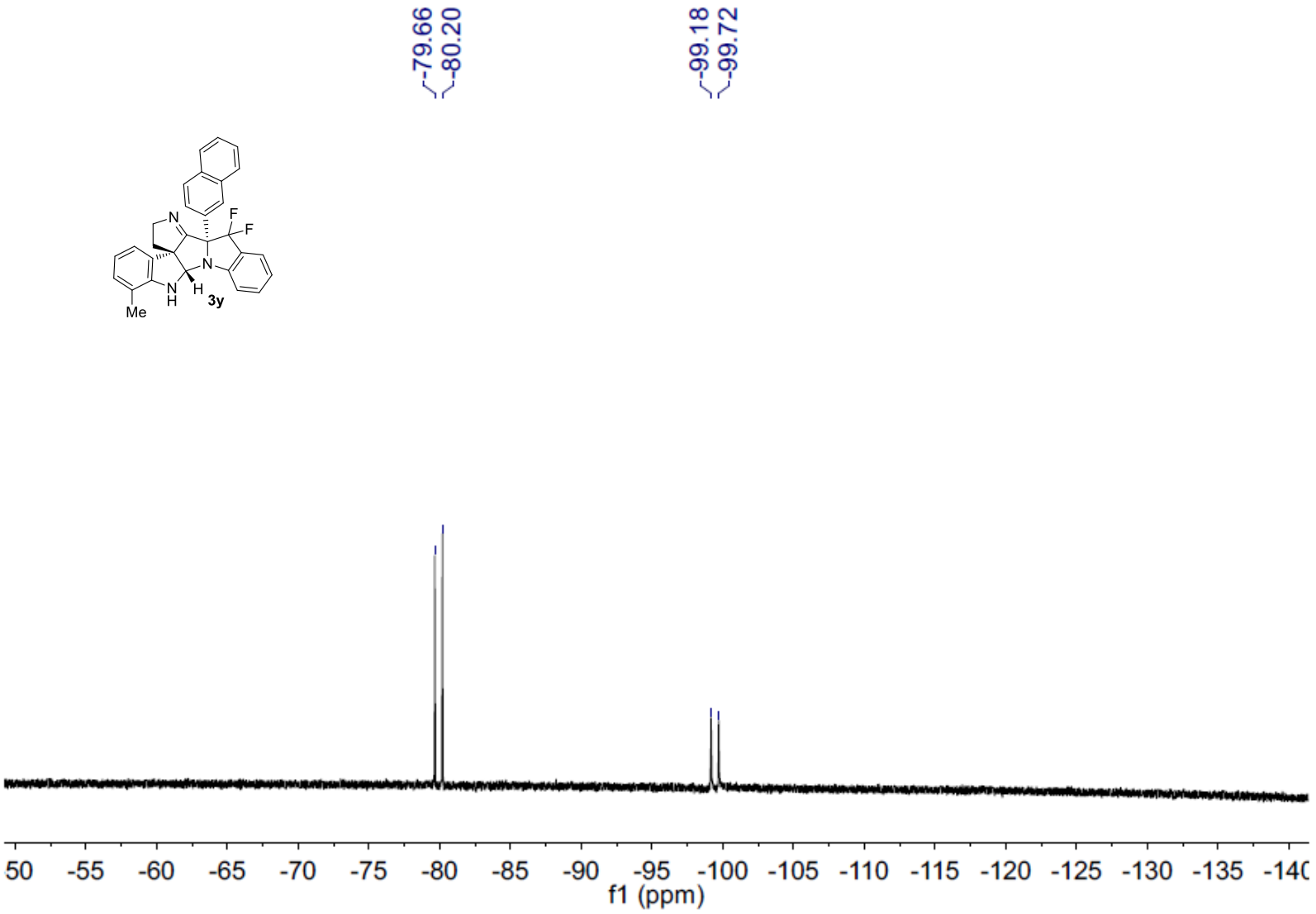
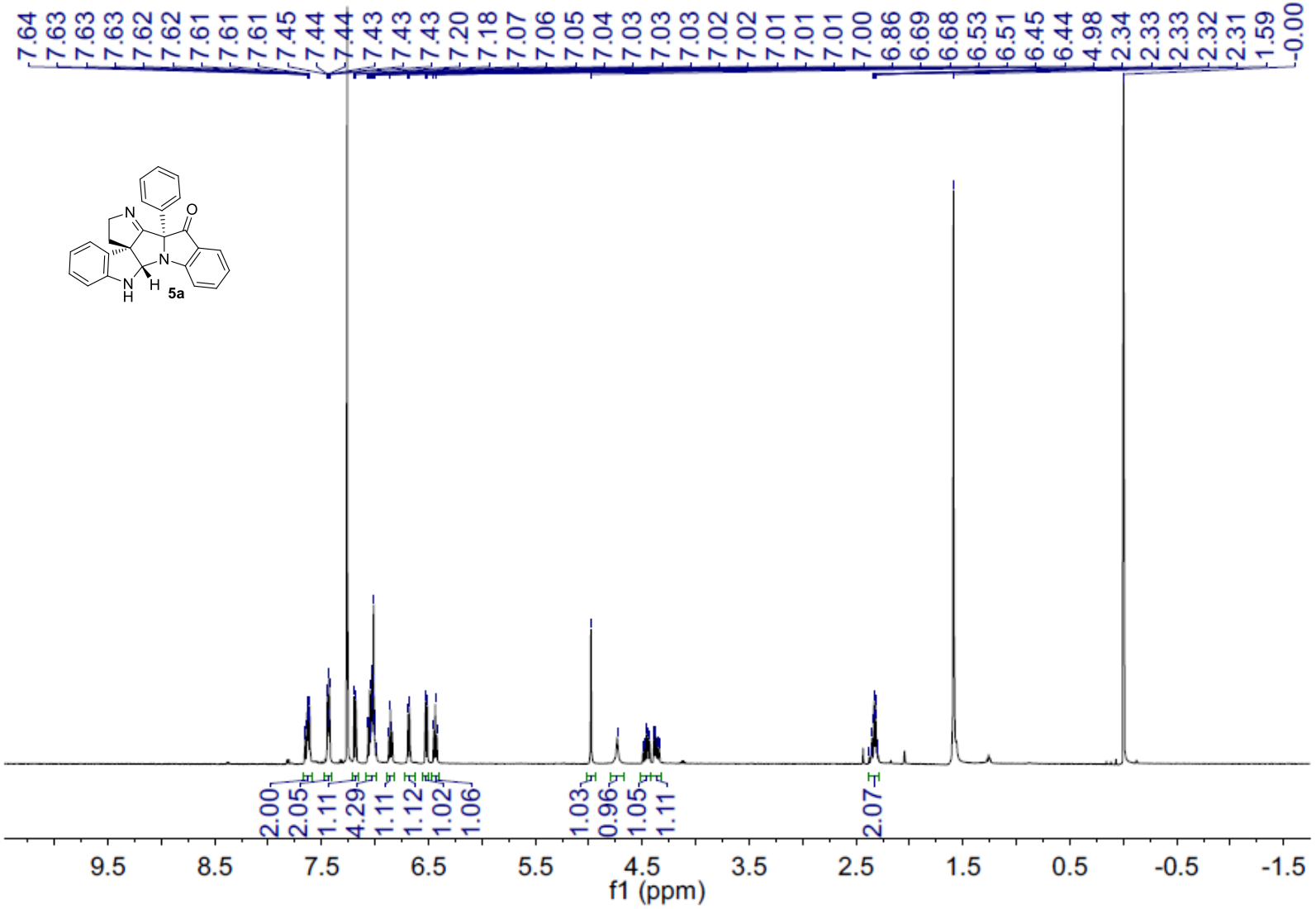
151.22
151.20
151.16
146.79
133.22
132.85
132.50
131.28
131.24
129.55
129.05
127.70
127.25
127.03
125.96
125.85
125.57
124.74
123.95
123.64
123.50
122.30
122.13
122.09
121.89
121.57
119.66
119.55
77.98
77.82
77.73
77.57
f1 (ppm)
f1 (ppm)
f1 (ppm)
3y
Me


-79.66
-80.20
-99.18
-99.72
F
F
N
N
H
N
H
Me
3y
50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
-135
-140
f1 (ppm)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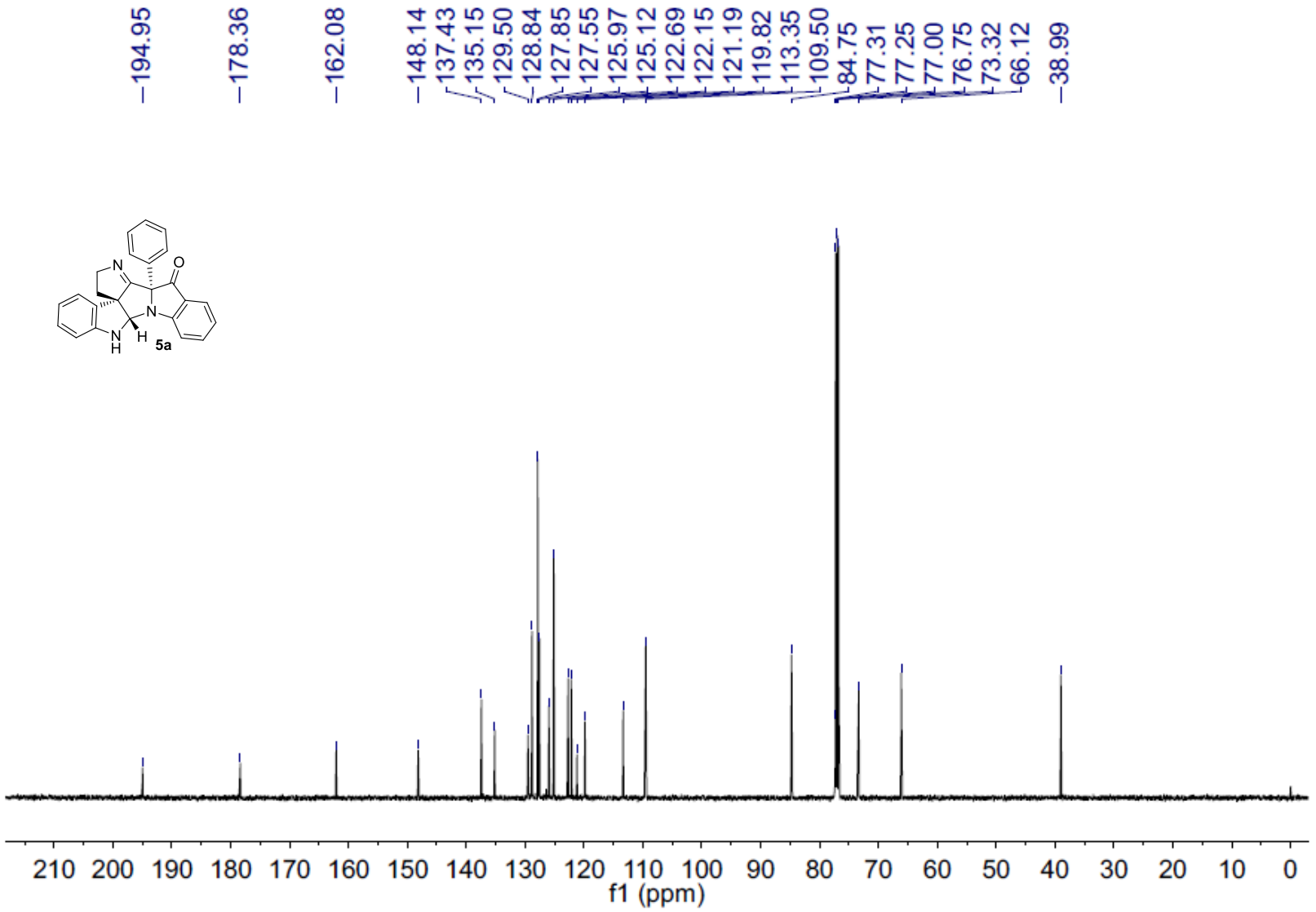
N
O
N
N
H
H
5a
2.00 2.05 1.11 4.29 1.11 1.12 1.02 1.06 1.03 0.96 1.05 1.11 2.07
9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5 -1.5
f1 (ppm)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148.14
137.43
135.15
129.50
128.84
127.85
127.55
125.97
125.12
122.69
122.15
121.19
119.82
113.35
109.50
84.75
77.31
77.25
77.00
76.75
73.32
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38.99
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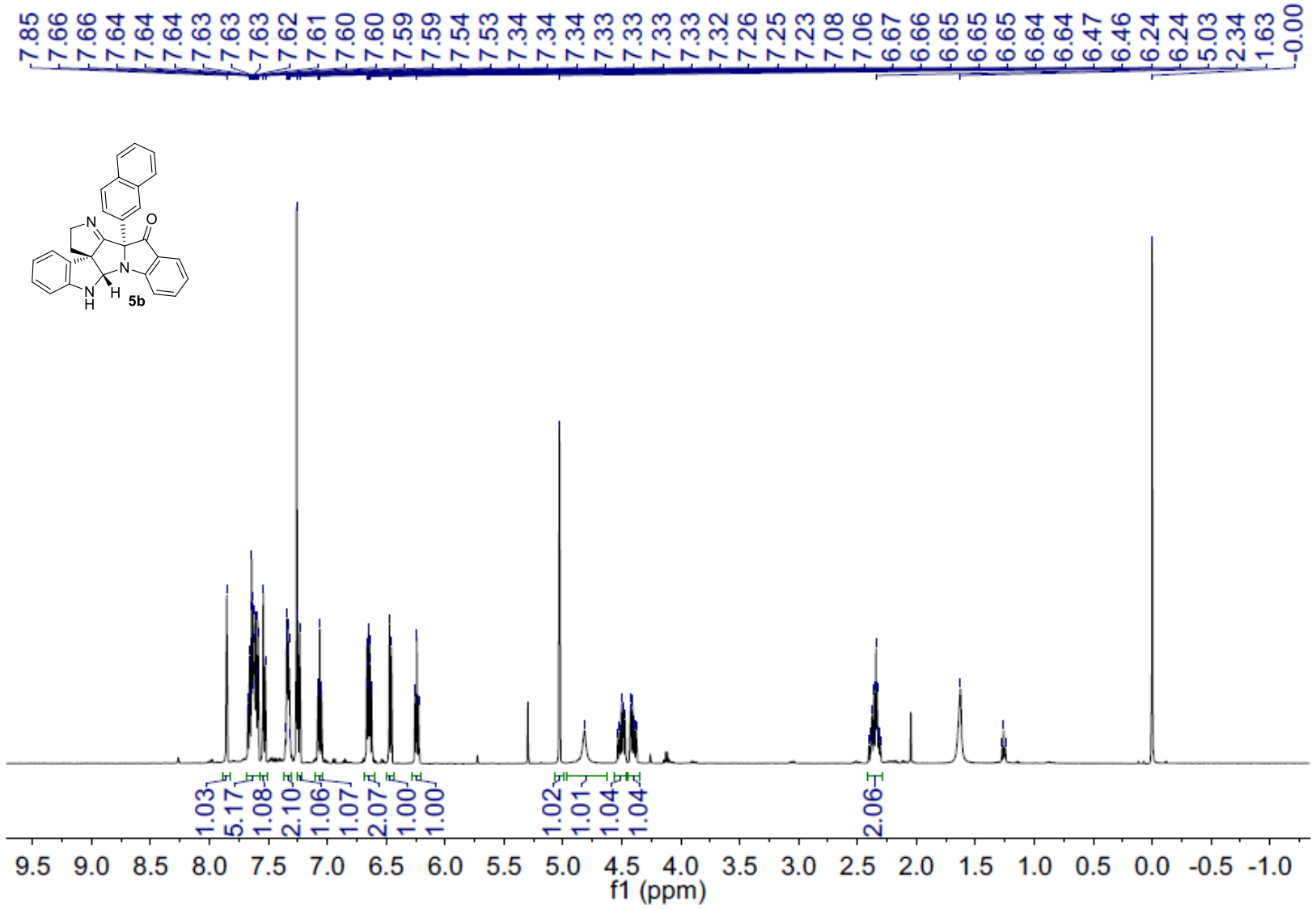
O
N
N
H
H
5a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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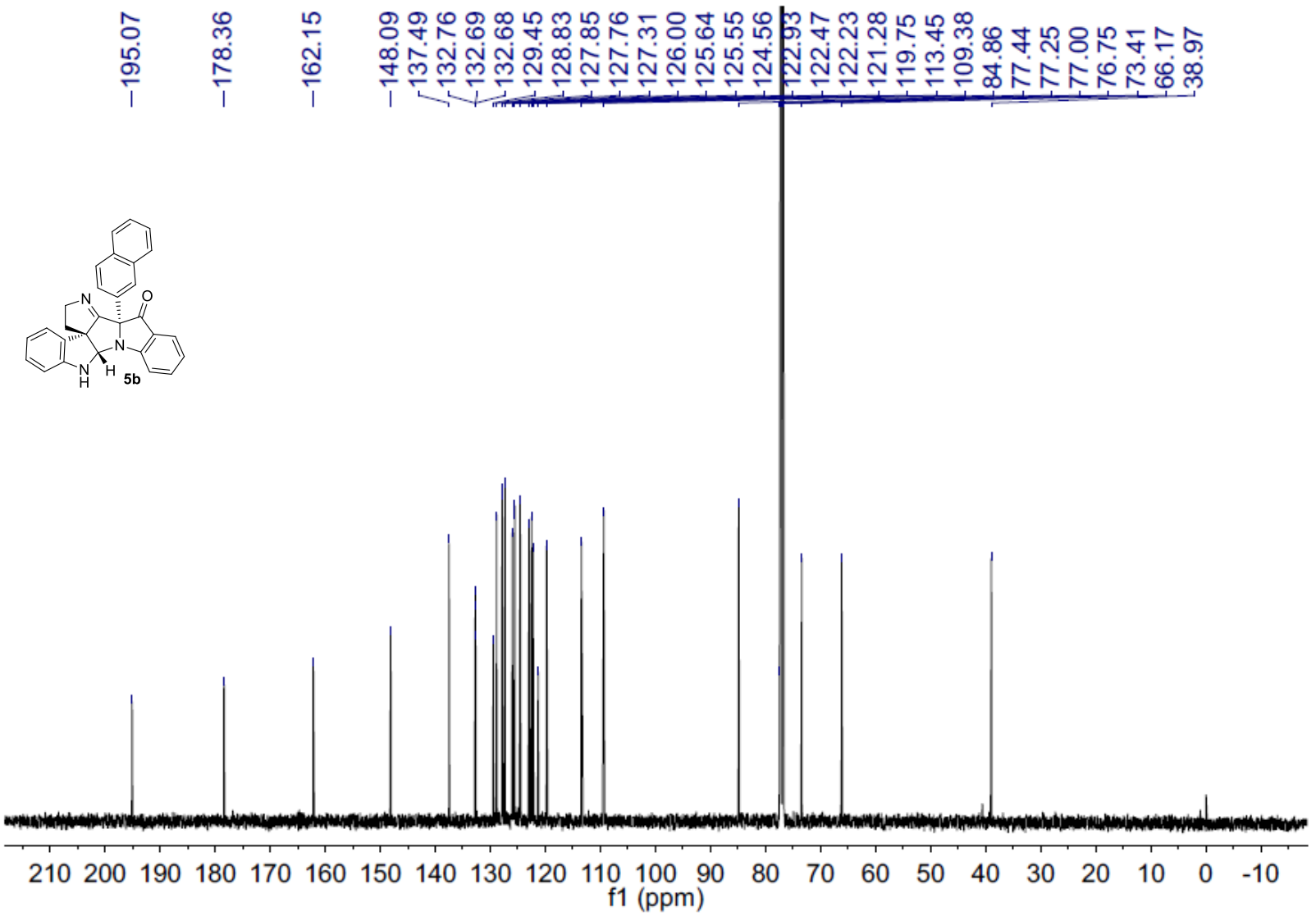
O
N
N
H
N
H
5b
1.03 5.17 1.08 2.10 1.06 1.07 2.07 1.00 1.00 1.02 1.01 1.04 1.04 2.06
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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127.85
127.76
127.31
126.00
125.64
125.55
124.56
122.93
122.47
122.23
121.28
119.75
113.45
109.38
84.86
77.44
77.25
77.00
76.75
73.41
66.17
38.97
N
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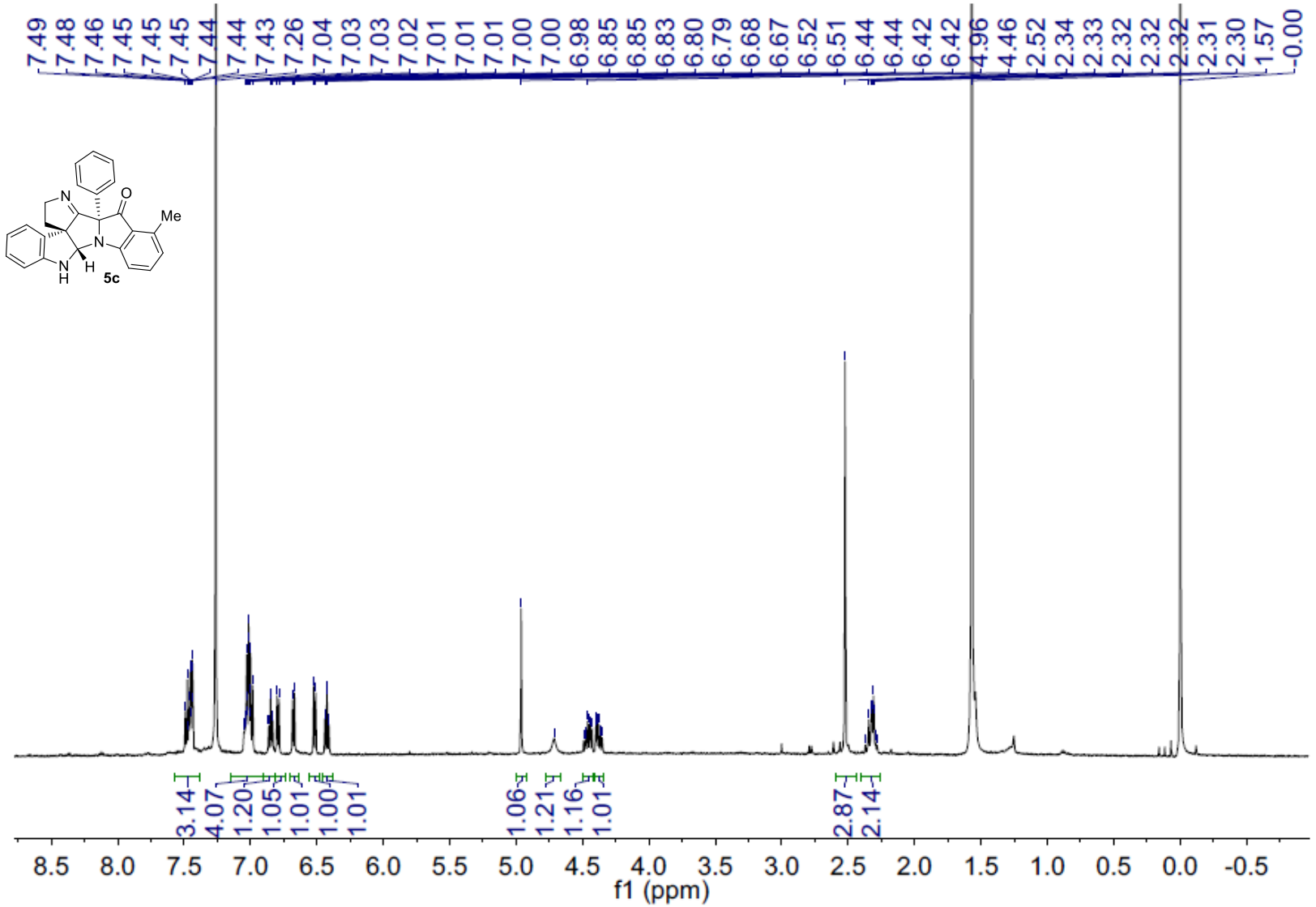
N
N
H
H
5b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.45
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7.44
7.43
7.26
7.04
7.03
7.03
7.02
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7.01
7.01
7.00
7.00
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6.85
6.83
6.80
6.79
6.68
6.67
6.52
6.51
6.44
6.44
6.42
6.42
4.96
4.46
2.52
2.34
2.33
2.32
2.32
2.32
2.31
2.30
1.57
-0.00
N
O
Me
N
N
H
H
5c
3.14
4.07
1.20
1.05
1.01
1.00
1.01
1.06
1.21
1.16
1.01
2.87
2.14
8.5
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7.5
7.0
6.5
6.0
5.5
5.0
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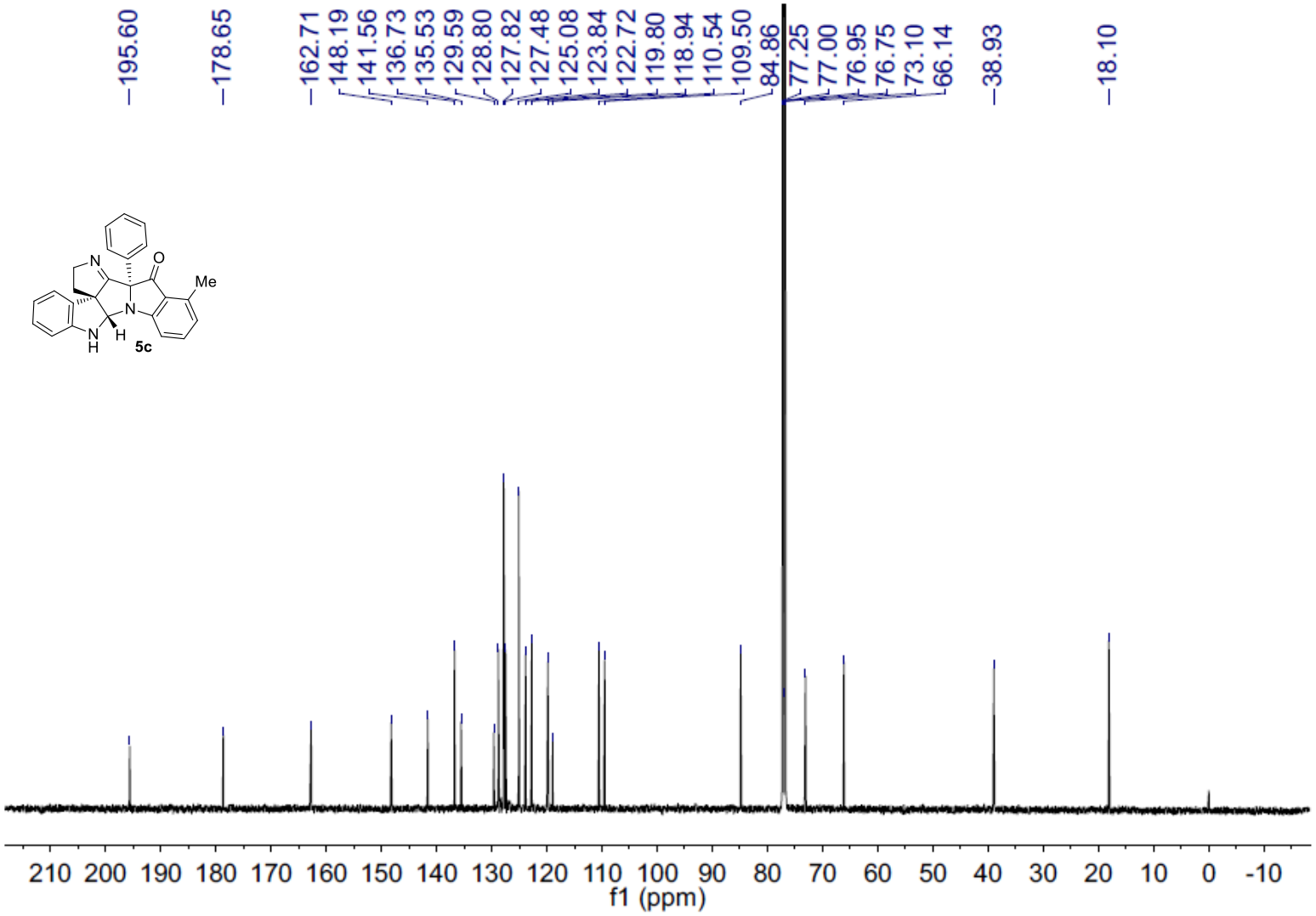
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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127.82
127.48
125.08
123.84
122.72
119.80
118.94
110.54
109.50
84.86
77.25
77.00
76.95
76.75
73.10
66.14
38.93
18.10
Me
O
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N
H
N
H
5c
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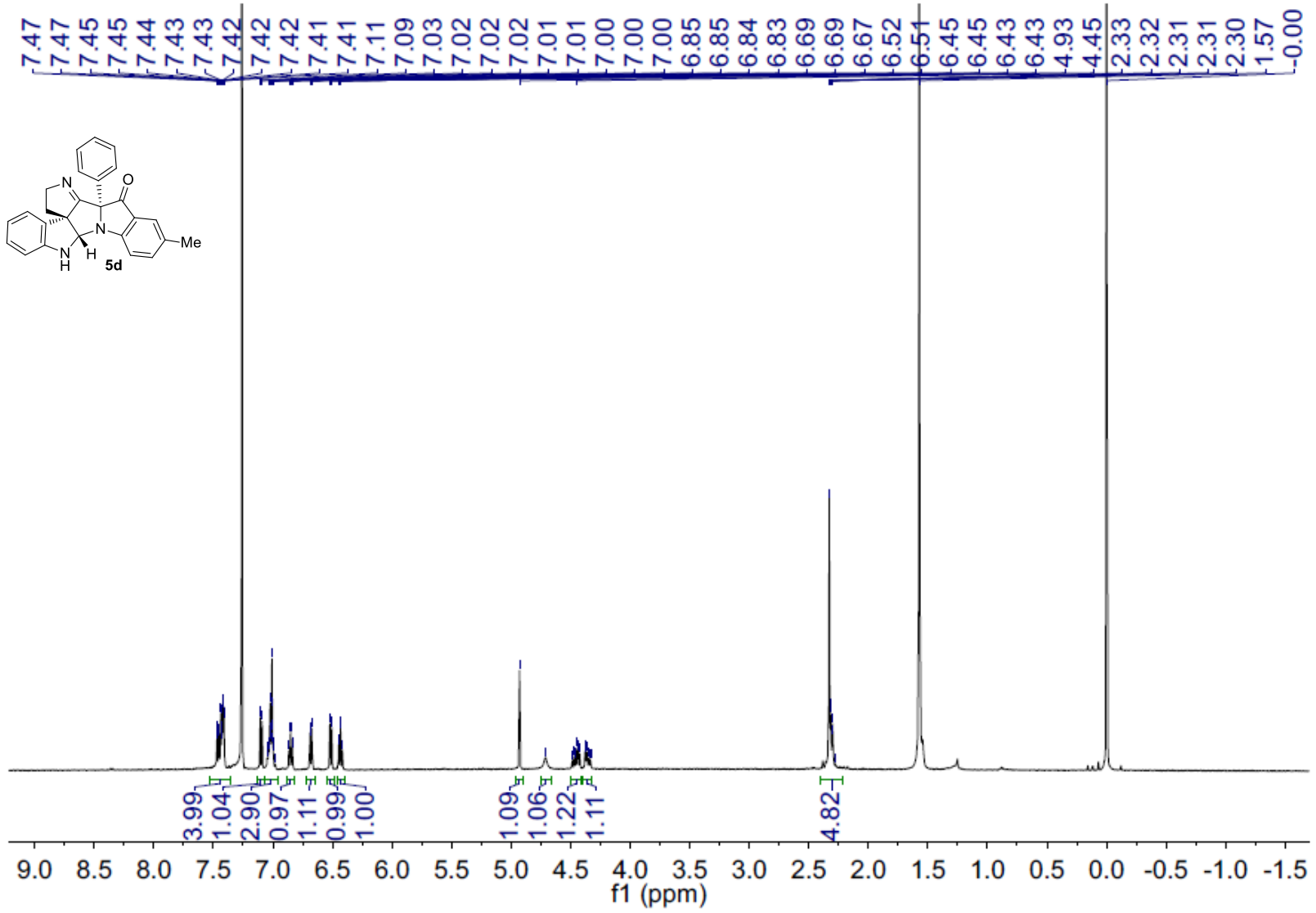
f1 (ppm)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7.02
7.02
7.02
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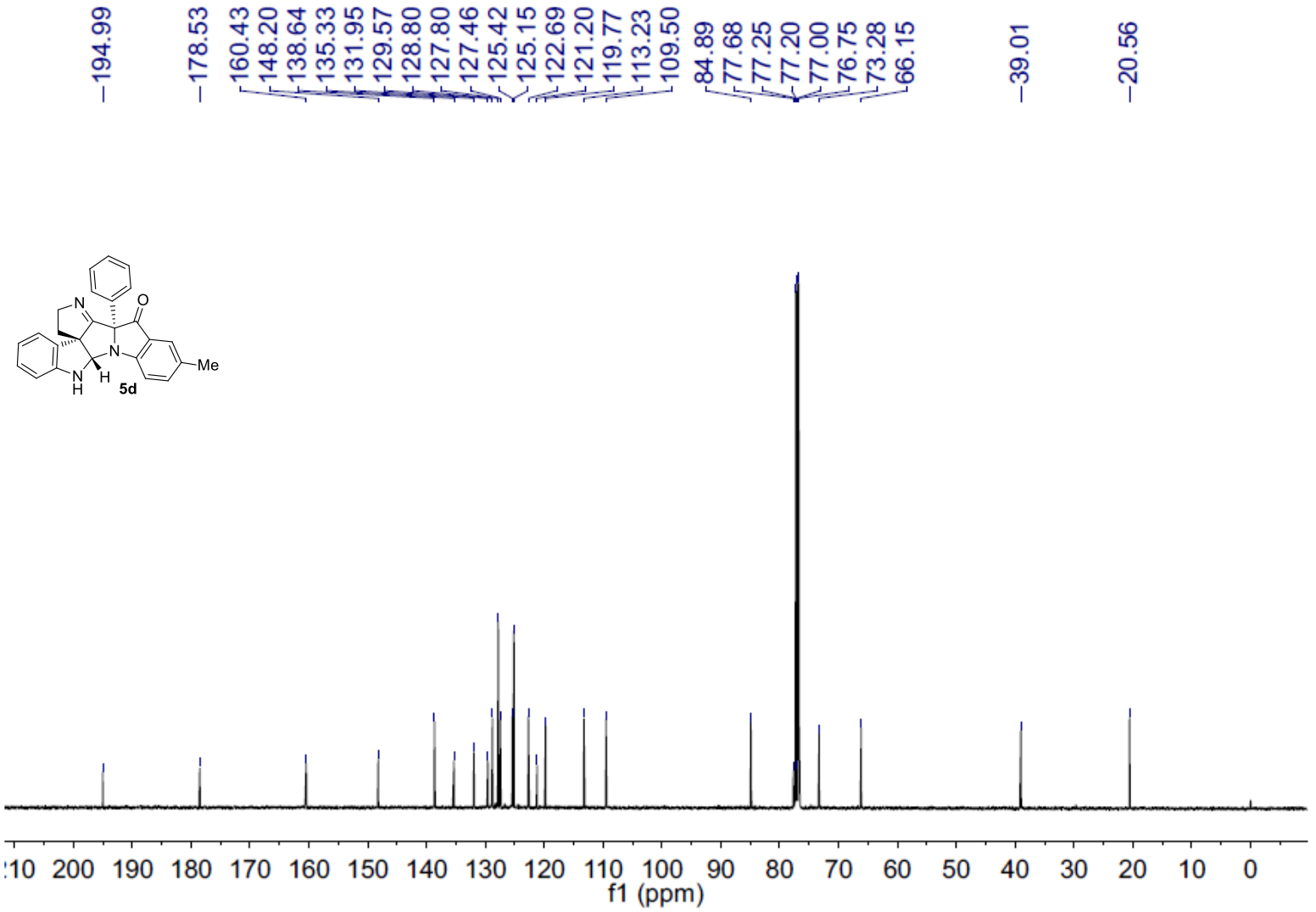
2.30
1.57
-0.00
N
O
N
N
H
H
Me
5d
3.99
1.04
2.90
0.97
1.11
0.99
1.00
1.09
1.06
1.22
1.11
4.82
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)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122.69
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113.23
109.50
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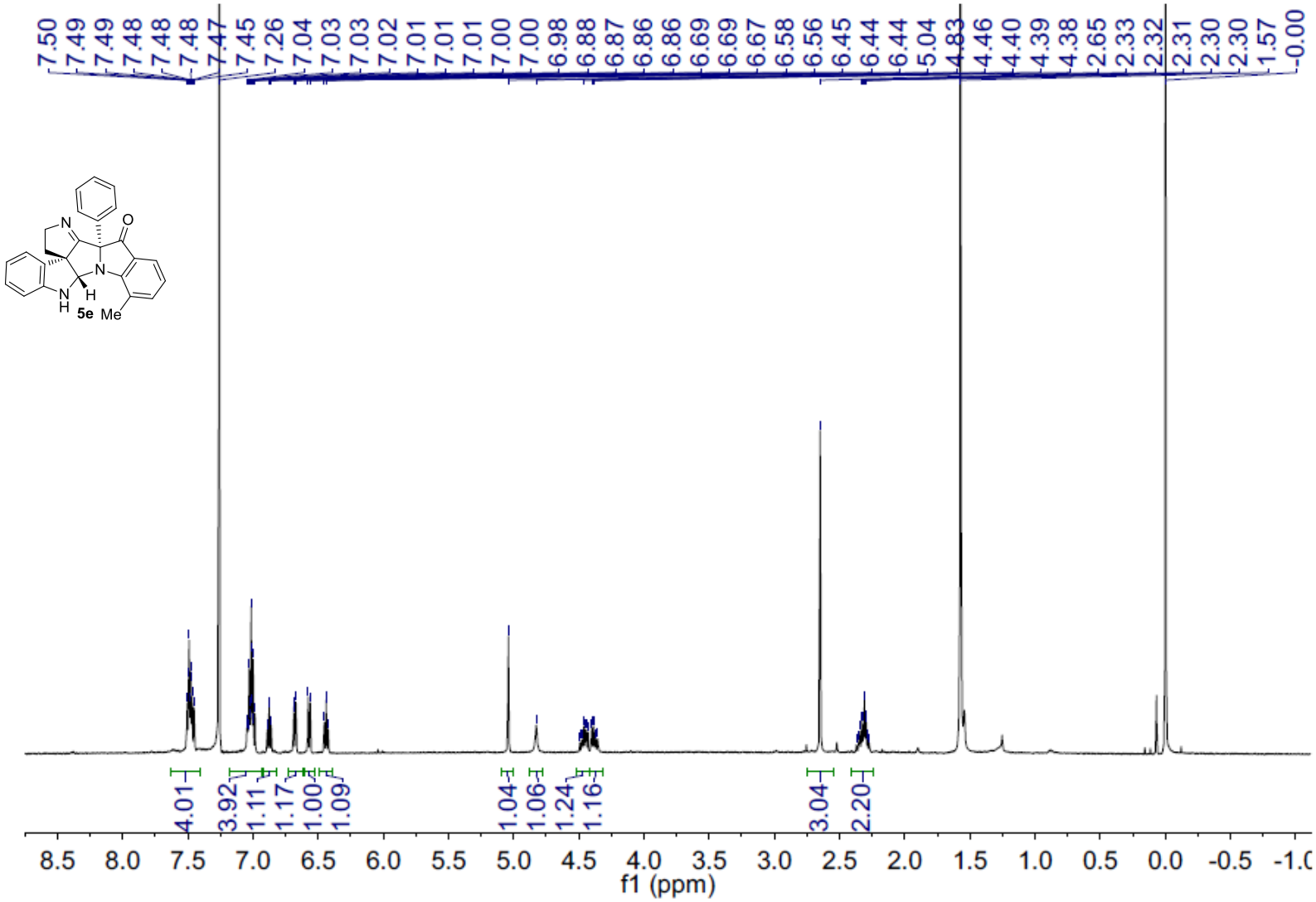
39.01
20.56
N
O
N
N
H
H
Me
5d
f1 (ppm)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7.03
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6.44
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2.33
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2.31
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2.30
1.57
-0.00
O
N
N
H
H
5e Me
4.01
3.92
1.11
1.17
1.00
1.09
1.04
1.06
1.24
1.16
3.04
2.20
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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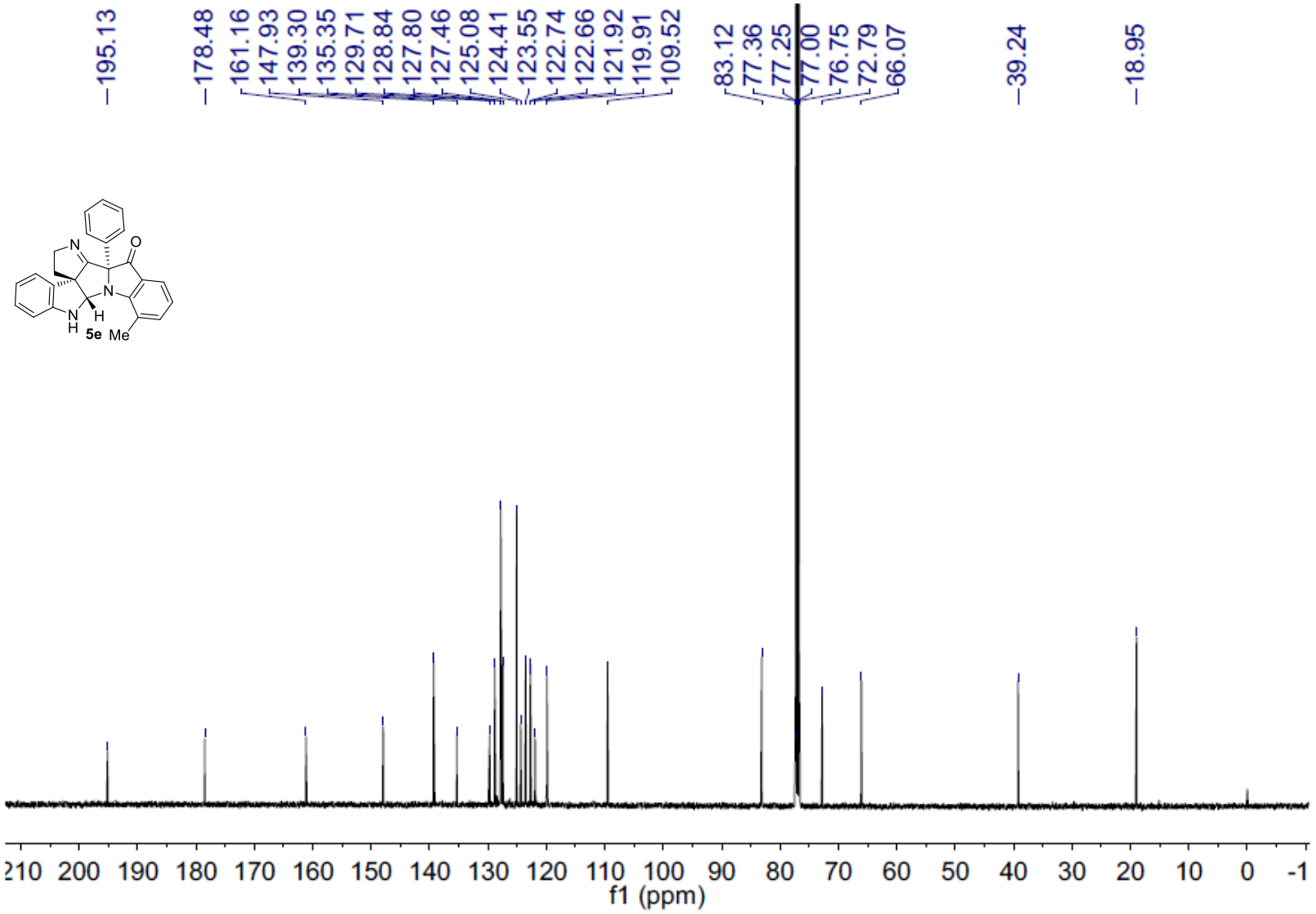
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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122.74
122.66
121.92
119.91
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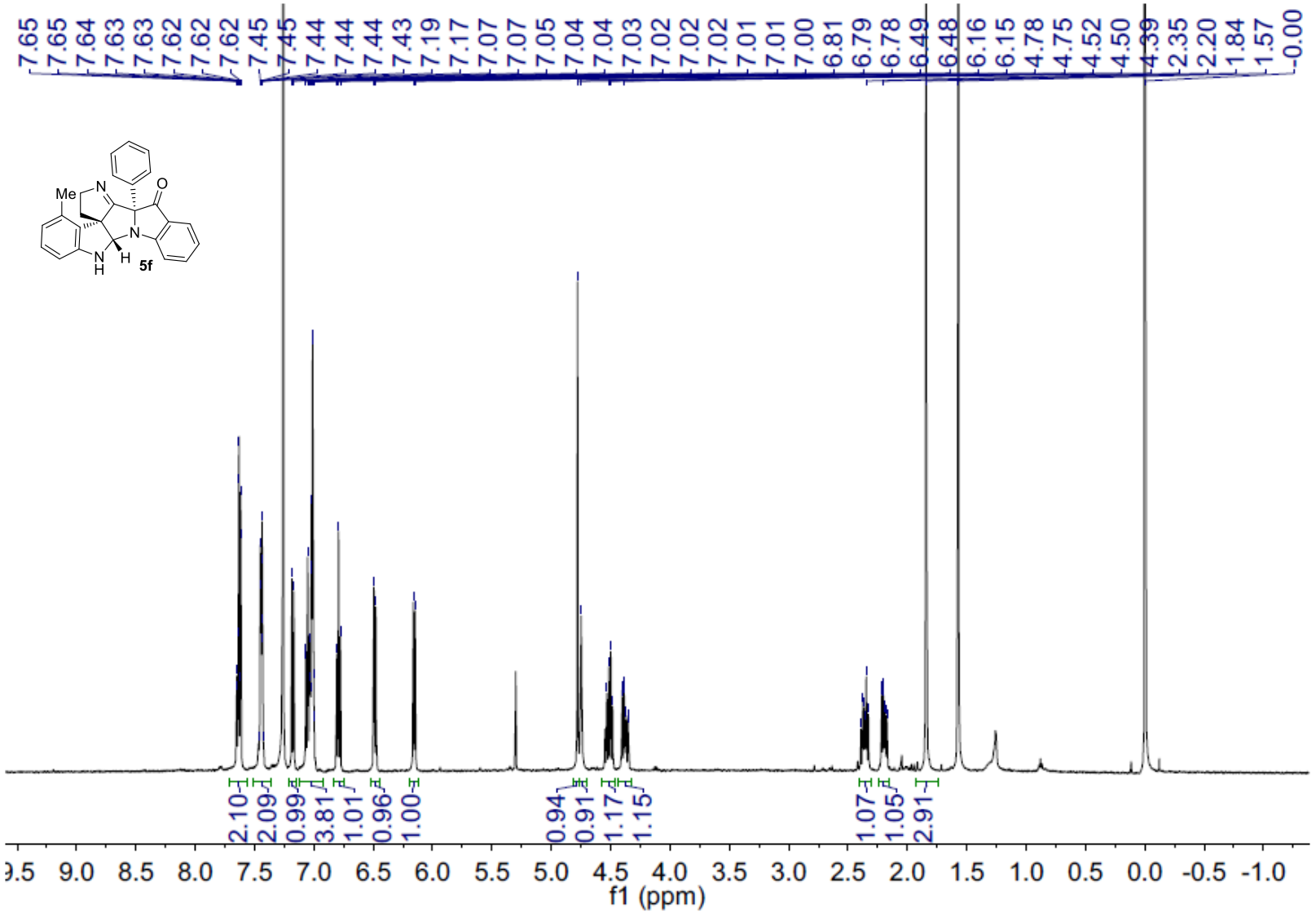
77.00
76.75
72.79
66.07
39.24
18.95
5e Me
f1 (ppm)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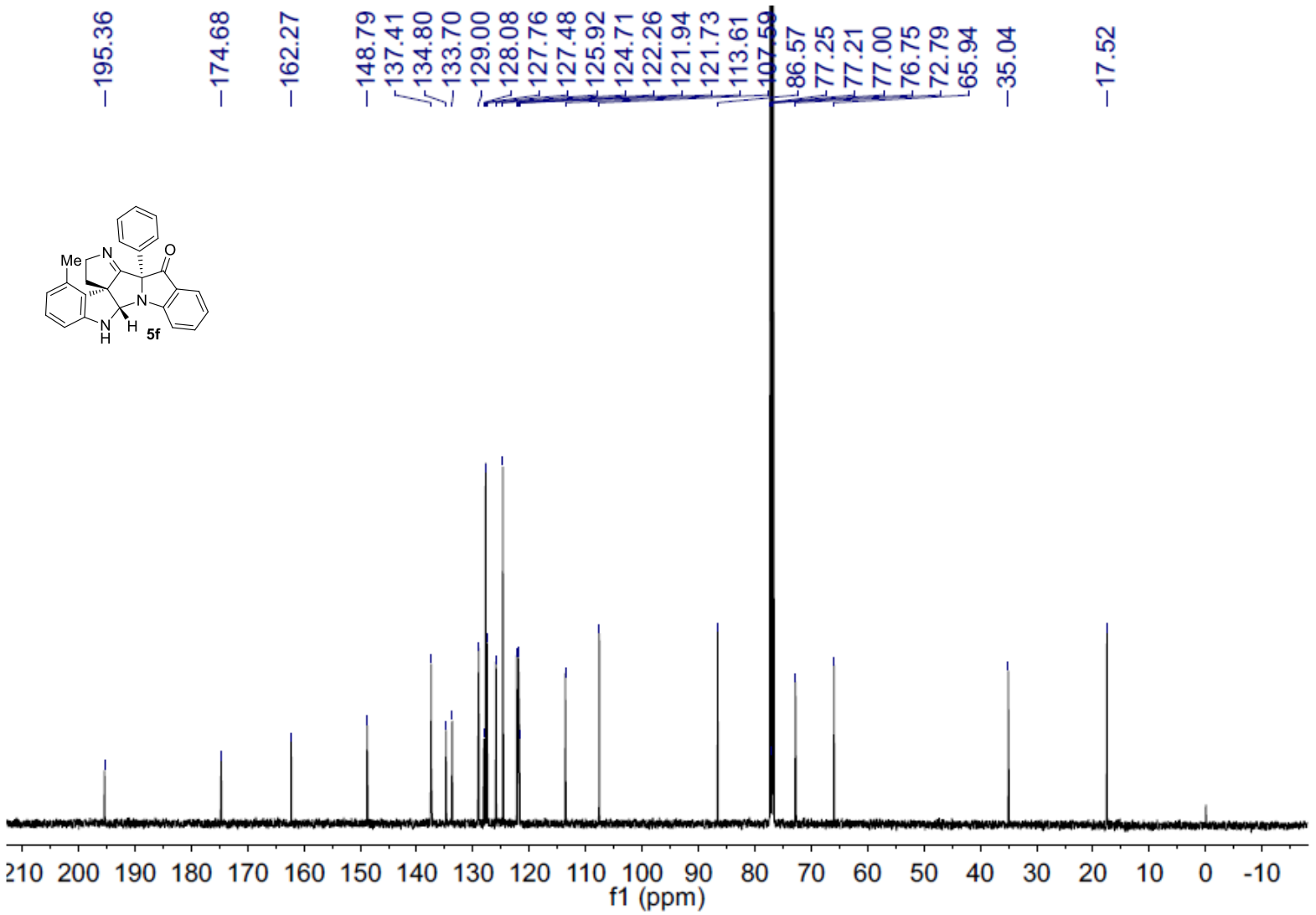
Me
N
O
N
N
H
H
5f
2.10 2.09 0.99 3.81 1.01 0.96 1.00 0.94 0.91 1.17 1.15 1.07 1.05 2.91
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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124.71
122.26
121.94
121.73
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107.59
86.57
77.25
77.21
77.00
76.75
72.79
65.94
35.04
17.52
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N
H
H
5f
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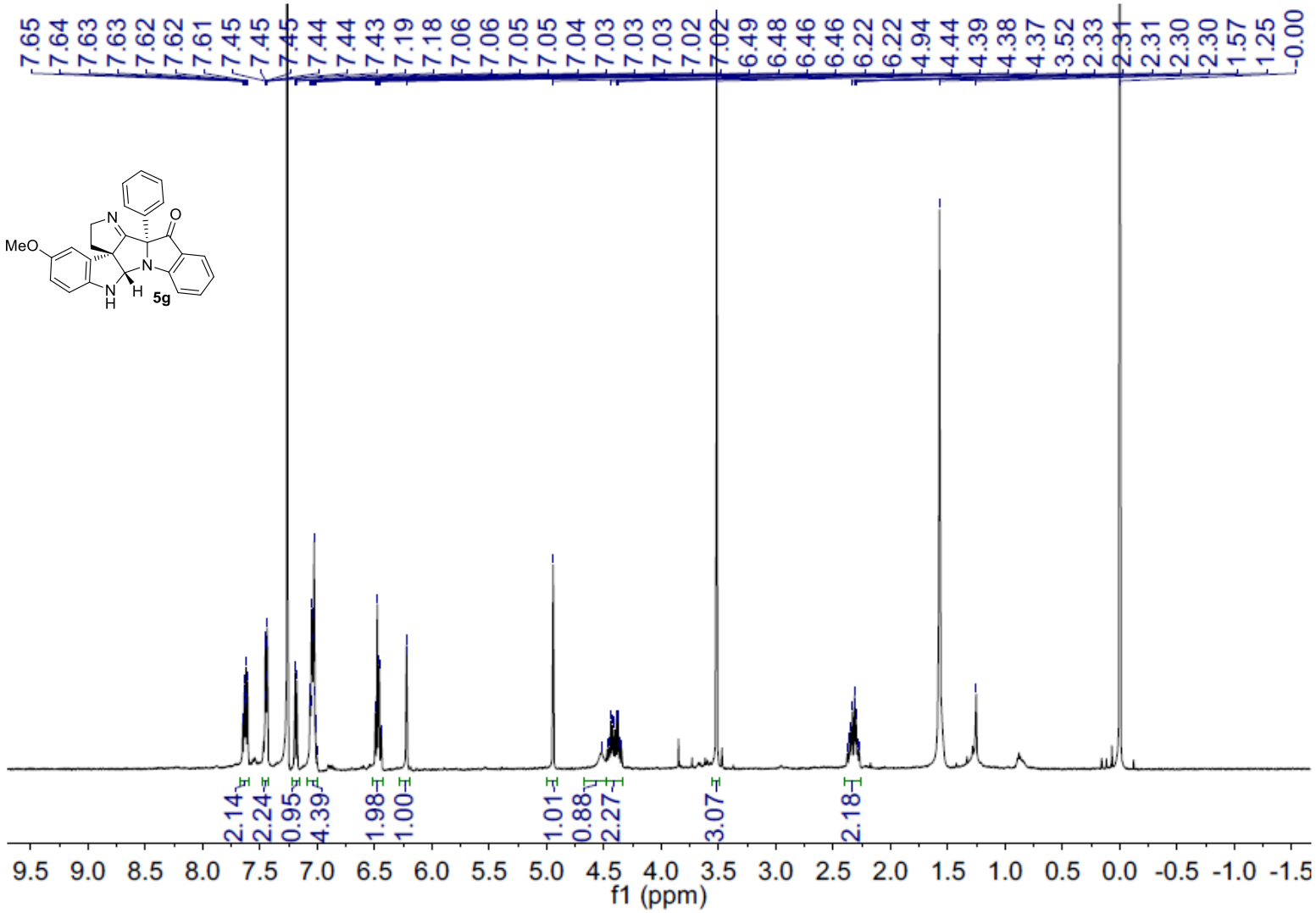
f1 (ppm)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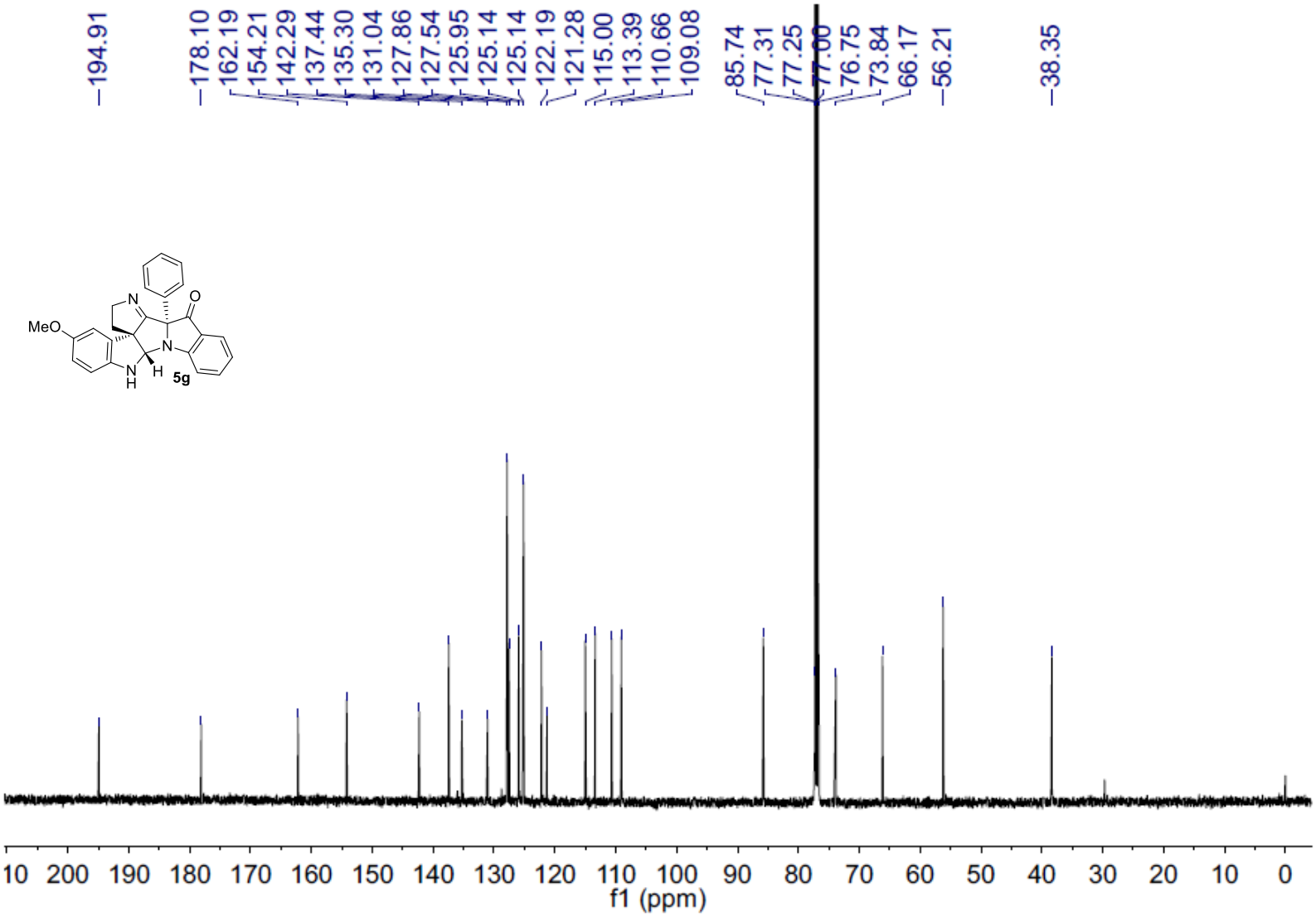
7.43
7.19
7.18
7.06
7.06
7.05
7.05
7.04
7.03
7.03
7.03
7.02
7.02
6.49
6.48
6.46
6.46
6.22
6.22
4.94
4.44
4.39
4.38
4.37
3.52
2.33
2.31
2.31
2.30
2.30
1.57
1.25
0.00
MeO
N
O
N
N
H
H
5g
2.14
2.24
0.95
4.39
1.98
1.00
1.01
0.88
2.27
3.07
2.18
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)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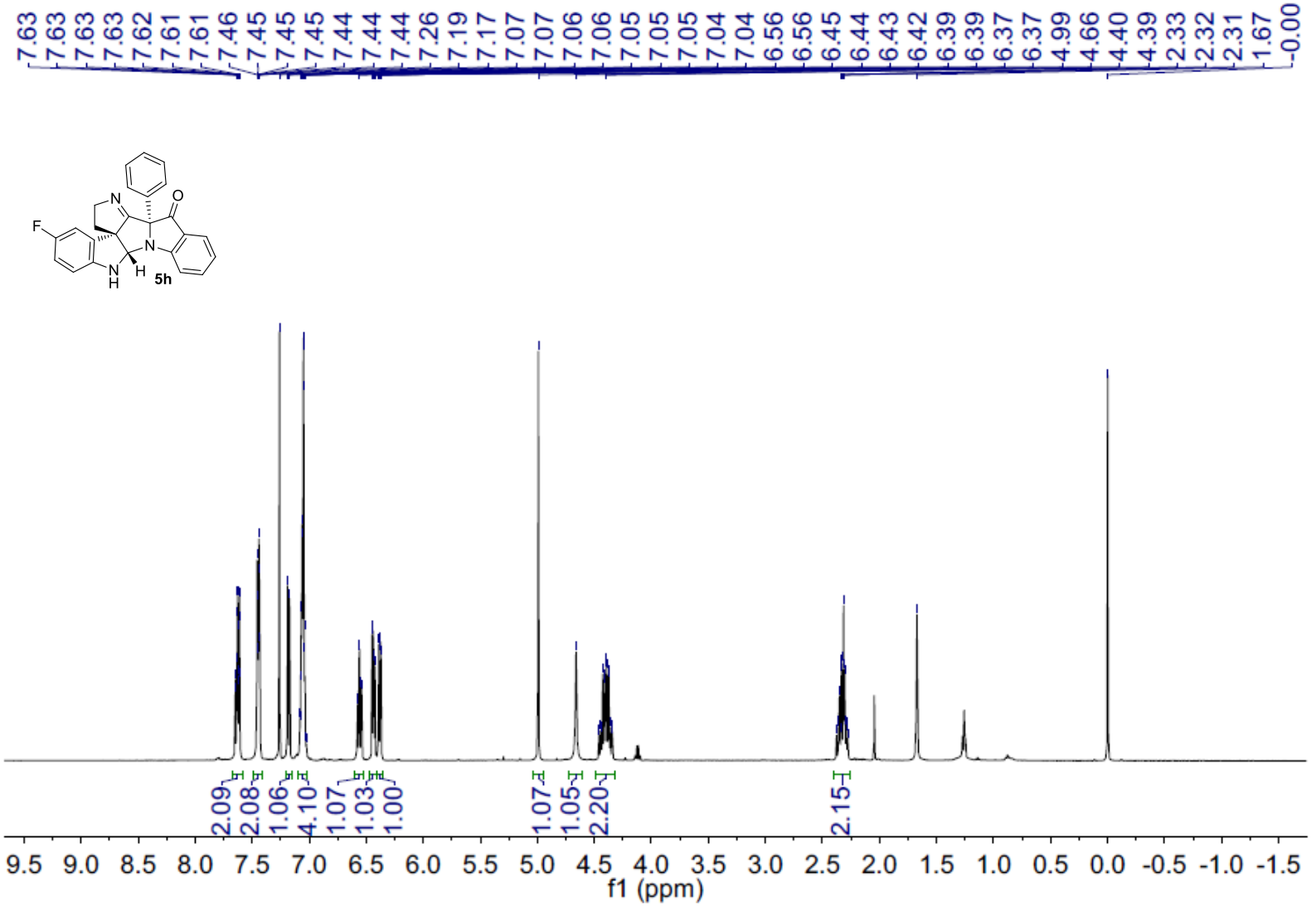
162.19
154.21
142.29
137.44
135.30
131.04
127.86
127.54
125.95
125.14
125.14
122.19
121.28
115.00
113.39
110.66
109.08
85.74
77.31
77.25
77.00
76.75
73.84
66.17
56.21
38.35
MeO
N
O
N
N
H
H
5g
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.61
7.61
7.46
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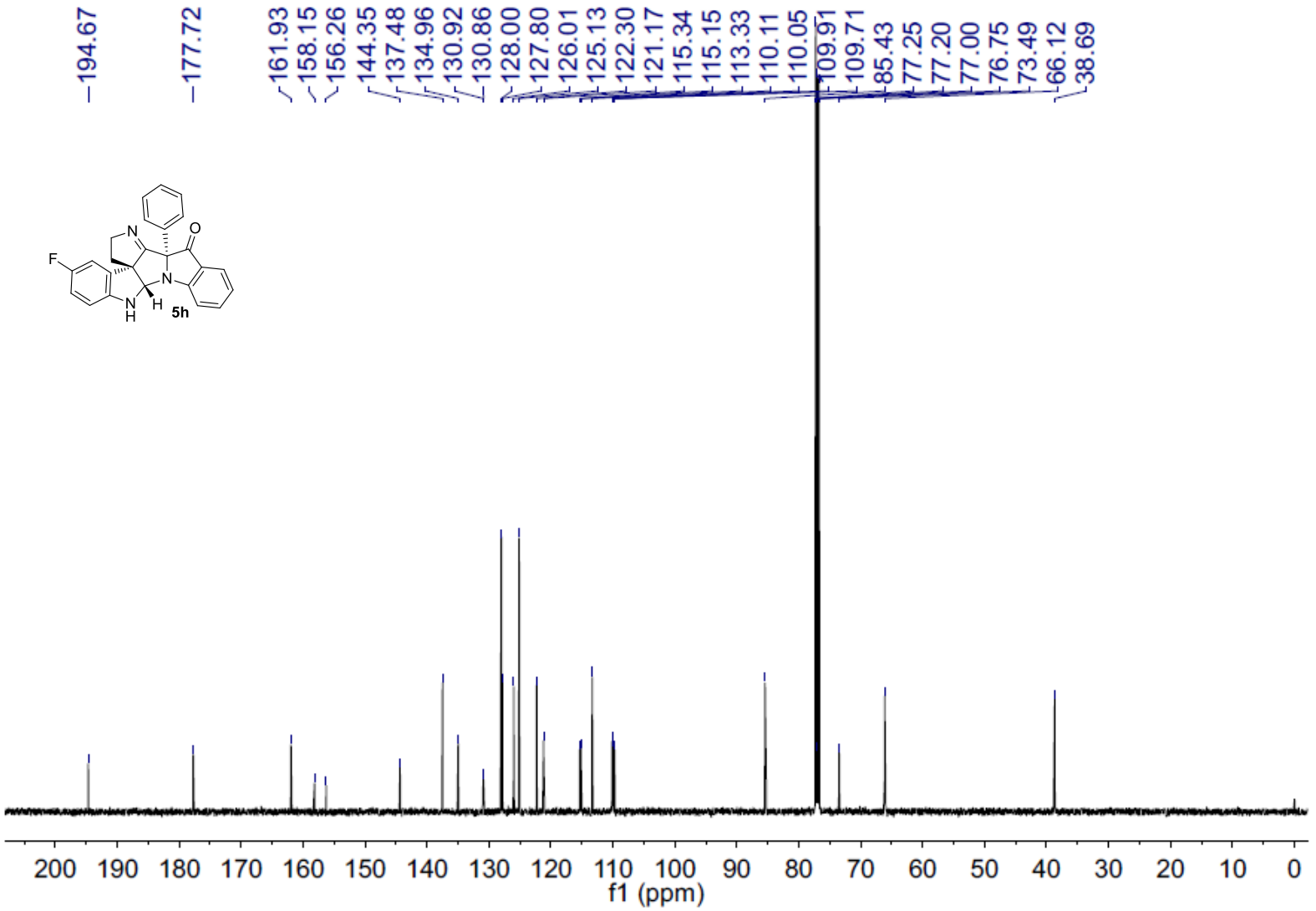
7.44
7.44
7.44
7.26
7.19
7.17
7.07
7.07
7.06
7.06
7.05
7.05
7.05
7.04
7.04
6.56
6.56
6.45
6.44
6.43
6.42
6.39
6.39
6.37
6.37
4.99
4.66
4.40
4.39
2.33
2.32
2.31
1.67
-0.00
F
N
O
N
H
N
H
5h
2.09
2.08
1.06
4.10
1.07
1.03
1.00
1.07
1.05
2.20
2.15
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)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144.35
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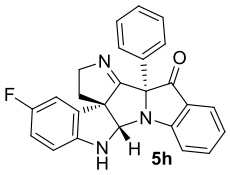
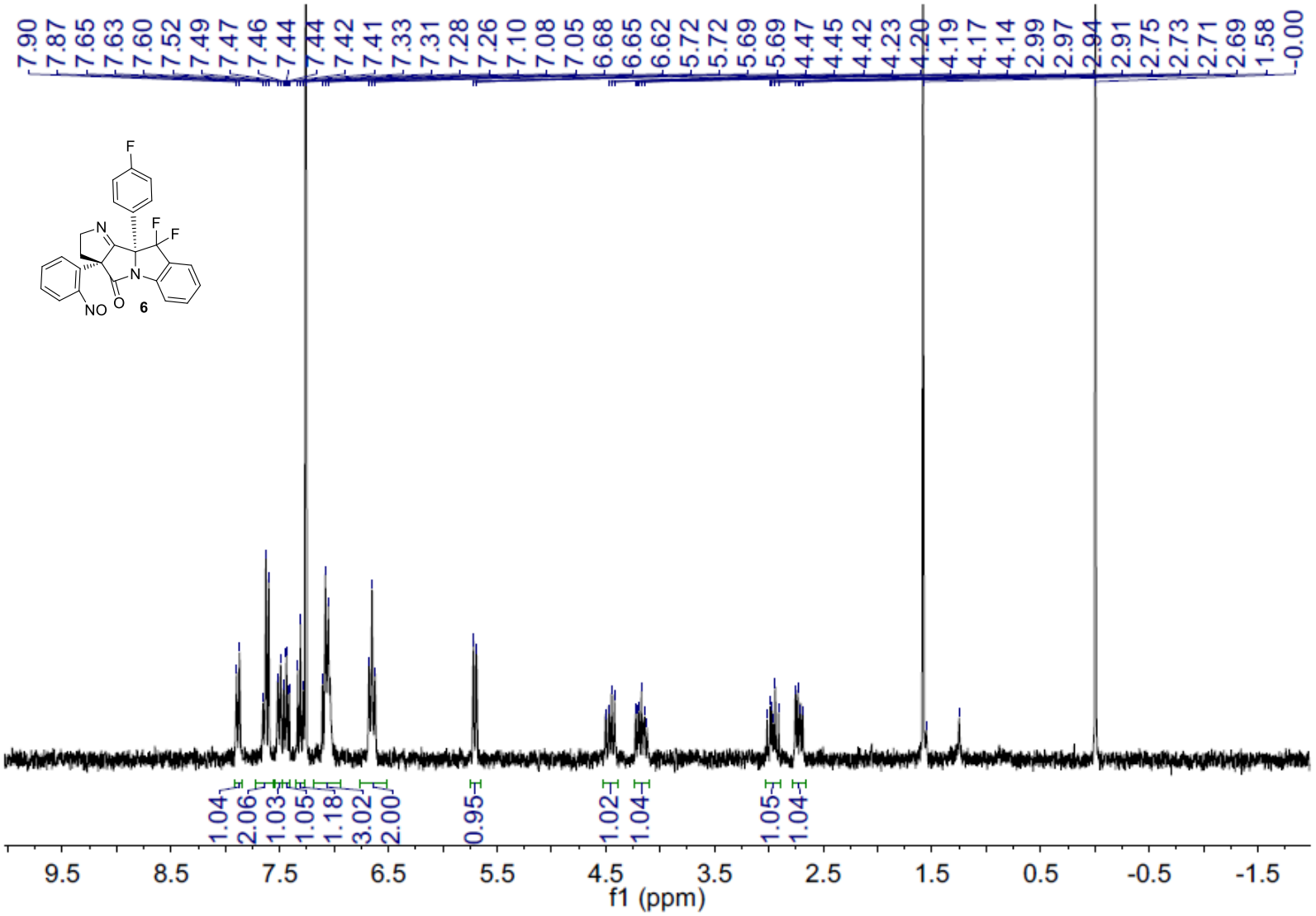
130.92
130.86
128.00
127.80
126.01
125.13
122.30
121.17
115.34
115.15
113.33
110.11
110.05
109.91
109.71
85.43
77.25
77.20
77.00
76.75
73.49
66.12
38.69
F
N
O
N
N
H
H
5h
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


F
N
O
N
N
H
H
5h

-124.63
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)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7.63
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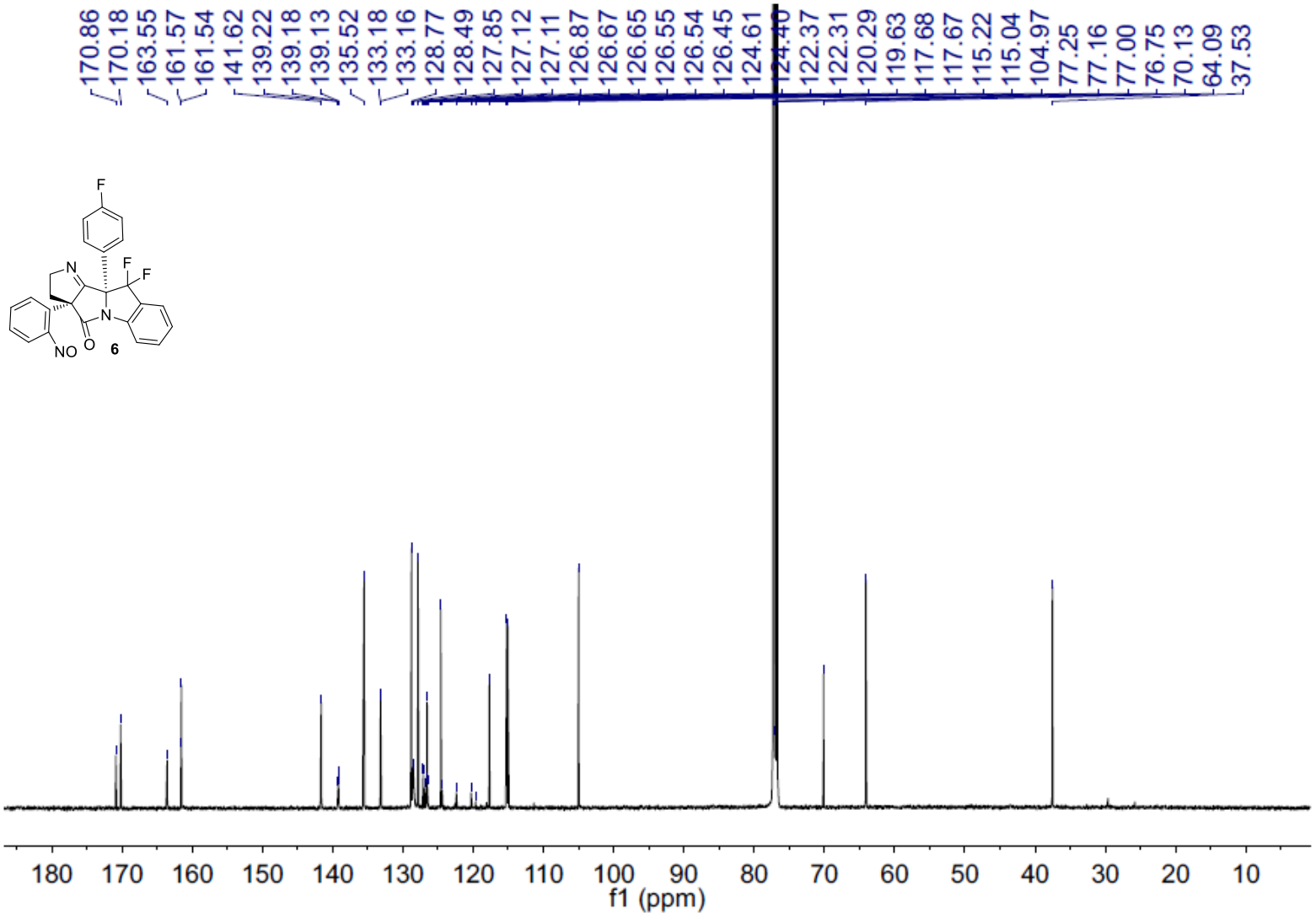
7.49
7.47
7.46
7.44
7.44
7.42
7.41
7.33
7.31
7.28
7.26
7.10
7.08
7.05
6.68
6.65
6.62
5.72
5.72
5.69
5.69
4.47
4.45
4.42
4.23
4.20
4.19
4.17
4.14
2.99
2.97
2.94
2.91
2.75
2.73
2.71
2.69
1.58
-0.00
F
F
F
N
N
O
NO
6
1.04
2.06
1.03
1.05
1.18
3.02
2.00
0.95
1.02
1.04
1.05
1.04
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)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161.57
161.54
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139.18
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135.52
133.18
133.16
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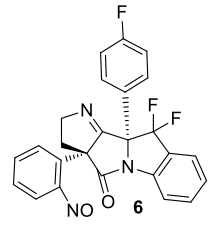
128.49
127.85
127.12
127.11
126.87
126.67
126.65
126.55
126.54
126.45
124.61
124.40
122.37
122.31
120.29
119.63
117.68
117.67
115.22
115.04
104.97
77.25
77.16
77.00
76.75
70.13
64.09
37.53
F
N
F
F
N
O
NO
6
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


F
N
F
F
N
O
NO
6

79.44
80.29
110.90
111.75
112.41
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
f1 (ppm)