

Supporting Information

Metal-Free oxidative Trifluoromethylation of Indoles with $\text{CF}_3\text{SO}_2\text{Na}$

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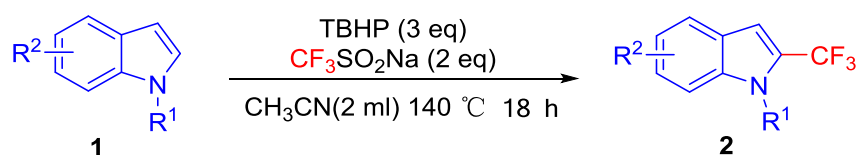
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1. General

The raw materials and reagents were purchased from various commercial sources and were used without further purification. The reactions were developed at 140 °C. Silica gel (300-400 mesh) was used for column chromatography. Analytical thin-layer chromatography (TLC) was visualized by UV-light. ¹H NMR, ¹³C NMR and ¹⁹F NMR spectra were mostly operated on a 400 MHz, 101 MHz and 376 MHz spectrometer (¹H NMR: CDCl₃ at 7.26 ppm; ¹³C NMR: CDCl₃ at 77.0 ppm). All chemical shift values, δ , and coupling constants, J , are quoted in ppm and Hz, respectively.

2. General Procedure for Synthesis



Indoles **1** (0.3 mmol), CF₃SO₂Na (0.6 mmol, 2 eq), TBHP (0.9 mmol, 3 eq) and CH₃CN (2 mL) were added into a sealed Pyrex test tube stirred at 140 °C under air. The reaction was stopped about 18 h and cooled to room temperature. Then, the crude product was purified by column chromatography on silica gel using ethyl acetate(EA) and petroleum ether(PE) as the developing solvent to give the products **2**.

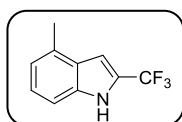
3. Data of NMR for the Compounds

2-(trifluoromethyl)-1H-indole(2a):¹ Pale-yellow solid: mp 106-107 °C, 61% yield, Known compound, ¹H NMR (400 MHz, CDCl₃) δ 8.37 (s, 1H), 7.68 (d, J = 8.0 Hz, 1H), 7.40 (d, J = 8.3 Hz, 1H), 7.32 (t, J = 7.6 Hz, 1H), 7.20 (dd, J = 14.6, 6.8 Hz, 1H), 6.92 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ -60.54 (s). ¹³C NMR (101 MHz, CDCl₃) δ 136.13, 126.60, 125.73 (q, J = 38.8 Hz), 124.77, 122.08, 121.26 (q, J = 268.5 Hz), 121.13, 111.69, 104.27 (q, J = 3.3 Hz).

1-methyl-2-(trifluoromethyl)-1H-indole(2b):² colorless liquid, 56% yield, Known compound, ¹H NMR (500 MHz, CDCl₃) δ 7.65 (d, J = 7.9 Hz, 1H), 7.34 (d, J = 2.8 Hz, 1H), 7.19-7.14 (m, 1H), 6.91 (s, 1H), 3.80 (s, 3H). ¹⁹F NMR (376 MHz, CDCl₃) δ -59.53. ¹³C NMR (126 MHz, CDCl₃) δ 138.49, 127.17 (q, J = 37.6 Hz), 125.64, 124.36, 122.57, 121.50 (q, J = 267.0 Hz), 122.20, 120.63, 109.77, 104.24 (q, J = 4.4 Hz), 30.58.

3-methyl-2-(trifluoromethyl)-1H-indole(2c):¹ yellow solid: mp 70-71 °C, 73% yield, Known compound, ¹H NMR (400 MHz, CDCl₃) δ 8.21 (s, 1H), 7.68 (d, J = 8.0 Hz, 1H), 7.46 – 7.30 (m, 2H), 7.25 (dd, J = 17.2, 9.7 Hz, 1H), 2.49 (d, J = 1.2 Hz, 3H). ¹⁹F NMR (376 MHz, CDCl₃) δ = -58.63. ¹³C NMR (101 MHz, CDCl₃) δ 135.19, 128.05, 124.74, 122.12 (q, J = 269.7 Hz), 121.53 (q, J = 36.9 Hz), 120.36 120.07, 114.05 (q, J = 2.9 Hz), 111.54, 8.29.

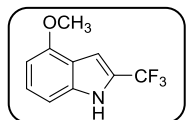
4-methyl-2-(trifluoromethyl)-1H-indole(2d): brown liquid, 76% yield, Known compound,



^1H NMR (400 MHz, CDCl_3) δ 8.29 (s, 1H), 7.13 (d, J = 4.2 Hz, 2H), 6.89 (dd, J = 10.6, 6.7 Hz, 2H), 2.46 (s, 3H). ^{19}F NMR (376 MHz, CDCl_3) δ -60.41. ^{13}C NMR (101 MHz, CDCl_3) δ 135.94, 131.78, 126.72, 125.10(q, J = 39.2 Hz), 124.90, 121.35(q, J = 268.5 Hz), 121.12, 109.21, 102.81(q, J = 3.4 Hz), 18.53. HRMS (ESI)

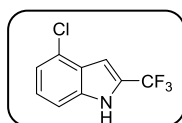
m/z calculated for $[\text{C}_{10}\text{H}_9\text{F}_3\text{N}]^+$ 200.0682; Found: 200.0689.

4-methoxy-2-(trifluoromethyl)-1H-indole(2e):³ white solid: mp 74-75 °C, 79% yield,



Known compound, ^1H NMR (400 MHz, CDCl_3) δ 8.24 (s, 1H), 7.11 (td, J_1 = 8.2, J = 3.2 Hz, 1H), 6.95 (d, J = 1.0 Hz, 1H), 6.81 (d, J = 8.3 Hz, 1H), 6.44 (dd, J = 7.8, 3.3 Hz, 1H), 3.81 (d, J = 2.7 Hz, 3H). ^{19}F NMR (376 MHz, CDCl_3) δ -60.35. ^{13}C NMR (101 MHz, CDCl_3) δ 154.17, 137.54, 125.75, 124.24(d, J = 38.8 Hz), 121.26(d, J = 268.4 Hz), 117.78, 104.68, 101.85(d, J = 3.5 Hz), 100.41, 55.31.

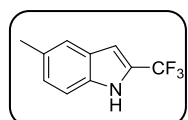
4-chloro-2-(trifluoromethyl)-1H-indole(2f): reddish brown liquid, 51% yield, Known



compound, ^1H NMR (400 MHz, CDCl_3) δ 8.55 (s, 1H), 7.32 (d, J = 7.8 Hz, 1H), 7.26 – 7.18 (m, 2H), 7.05 (s, 1H). ^{19}F NMR (376 MHz, CDCl_3) δ -60.74. ^{13}C NMR (101 MHz, CDCl_3) δ 136.66, 127.41, 126.23 (q, J = 40.4 Hz), 125.74, 125.37, 120.90 (q, J = 268.9 Hz), 120.86, 110.36, 102.92 (q, J = 3.0 Hz). HRMS (ESI) m/z

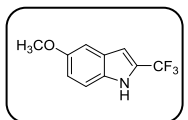
calculated for $[\text{C}_9\text{H}_5\text{ClF}_3\text{Na}]^+$ 241.9955; Found: 241.9962.

5-methyl-2-(trifluoromethyl)-1H-indole(2g):⁴ white solid: mp 122-123 °C, 61% yield,



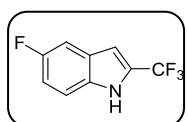
Known compound, ^1H NMR (400 MHz, CDCl_3) δ 8.30 (s, 1H), 7.46 (s, 1H), 7.31 (d, J = 8.4 Hz, 1H), 7.15 (d, J = 8.4 Hz, 1H), 6.84 (s, 1H), 2.45 (s, 3H). ^{19}F NMR (376 MHz, CDCl_3) δ = -60.52. ^{13}C NMR (101 MHz, CDCl_3) δ 134.49, 130.49, 126.89, 126.56, 125.71 (q, J = 39.4 Hz), 121.47, 121.30 (q, J = 268.6 Hz), 111.32, 103.77 (q, J = 3.3 Hz), 21.36.

5-methoxy-2-(trifluoromethyl)-1H-indole(2h):¹ yellow wax, 56% yield, Known compound,



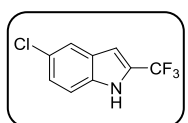
^1H NMR (400 MHz, CDCl_3) δ 8.43 (s, 1H), 7.35 (d, J = 8.8 Hz, 1H), 7.15 (s, 1H), 7.05 (d, J = 8.7 Hz, 1H), 6.90 (s, 1H), 3.91 (s, 3H). ^{19}F NMR (376 MHz, CDCl_3) δ = -60.51. ^{13}C NMR (101 MHz, CDCl_3) δ 154.90, 131.30, 127.09, 126.18 (q, J = 39.0 Hz), 121.20 (q, J = 268.7 Hz), 115.79, 112.56, 103.89 (q, J = 3.2 Hz), 102.80, 55.76.

5-fluoro-2-(trifluoromethyl)-1H-indole(2i):⁴ yellow liquid, 68% yield, Known compound,



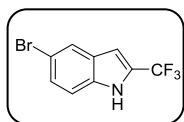
^1H NMR (400 MHz, CDCl_3) δ 8.46 (s, 1H), 7.38-7.30 (m, 2H), 7.09 (td, J = 9.1, 2.4 Hz, 1H), 6.88 (s, 1H). ^{19}F NMR (376 MHz, CDCl_3) δ -60.78, -122.56(td, J_1 = 11.3 Hz, J_2 = 3.8 Hz). ^{13}C NMR (101 MHz, CDCl_3) δ 158.35(d, J = 237.9 Hz), 132.66, 127.30(q, J = 39.4 Hz), 127.03(d, J = 10.1 Hz), 120.91(q, J = 268.7 Hz), 113.7(d, J = 27.3 Hz), 112.64(d, J = 9.1 Hz), 106.67(d, J = 24.2 Hz), 104.22(dq, J = 3.0 Hz).

5-chloro-2-(trifluoromethyl)-1H-indole(2j):⁵ yellow liquid, 58% yield, Known compound,

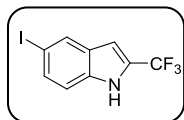


^1H NMR (400 MHz, CDCl_3) δ 8.52 (s, 1H), 7.65 (d, J = 1.3 Hz, 1H), 7.35 (d, J = 8.8 Hz, 1H), 7.28 (dd, J = 8.8, 1.9 Hz, 1H), 6.87 (s, 1H). ^{19}F NMR (376 MHz, CDCl_3) δ -60.79. ^{13}C NMR (101 MHz, CDCl_3) δ 134.43, 127.59, 127.24, 126.85, 125.32, 121.40, 120.89 (q, J = 268.9 Hz), 112.82, 103.79 (q, J = 3.2 Hz).

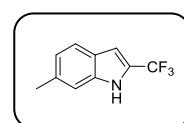
5-bromo-2-(trifluoromethyl)-1H-indole(2k):⁵ reddish brown solid: mp 52-53 °C, 54% yield, Known compound, ¹H NMR (400 MHz, CDCl₃) δ 8.2 (s, 1H), 7.81 (s, 1H), 7.40 (d, J = 7.0 Hz, 1H), 7.28 (dd, J = 14.2, 9.0 Hz, 1H), 6.86 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ = -60.77. ¹³C NMR (101 MHz, CDCl₃) δ 134.68, 128.23, 127.84, 126.86 (q, J = 39.3 Hz), 124.55, 120.84 (q, J = 269.1 Hz), 114.29, 113.19, 103.68 (q, J = 3.3 Hz).



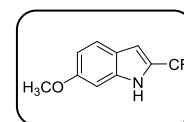
5-iodo-2-(trifluoromethyl)-1H-indole(2l):⁶ brown solid: mp 173-174 °C, 59% yield, Known compound, ¹H NMR (400 MHz, CDCl₃) δ = 8.42 (s, 1H), 8.02 (s, 1H), 7.56 (s, 1H), 7.22 – 7.16 (m, 1H), 6.83 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ = -60.72. ¹³C NMR (101 MHz, CDCl₃) δ 135.11, 133.19, 130.89, 129.02, 126.46 (q, J = 39.1 Hz), 120.80 (q, J = 269.0 Hz), 113.61, 103.33 (q, J = 3.1 Hz), 84.46.



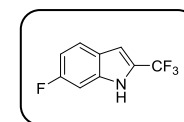
6-methyl-2-(trifluoromethyl)-1H-indole(2m): white solid: mp 106-107 °C, 75% yield, Known compound, ¹H NMR (400 MHz, CDCl₃) δ 8.25 (s, 1H), 7.55 (d, J = 8.2 Hz, 1H), 7.18 (s, 1H), 7.02 (d, J = 8.2 Hz, 1H), 6.86 (s, 1H), 2.47 (s, 3H). ¹⁹F NMR (376 MHz, CDCl₃) δ -60.45. ¹³C NMR (101 MHz, CDCl₃) δ 136.65, 134.90, 125.03 (q, J = 39.0 Hz), 124.42, 123.09, 122.51 (q, J = 268.4 Hz), 121.63, 111.41, 104.15 (q, J = 3.5 Hz), 21.81. HRMS (ESI) m/z calculated for [C₁₀H₉F₃N]⁺ 200.0682; Found: 200.0687.



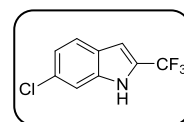
6-methoxy-2-(trifluoromethyl)-1H-indole(2n):³ pale-yellow solid: mp 89-90 °C, 60% yield, Known compound, ¹H NMR (400 MHz, CDCl₃) δ 8.36 (s, 1H), 7.53 (d, J = 8.7 Hz, 1H), 6.88 – 6.80 (m, 3H), 3.83 (s, 3H). ¹⁹F NMR (376 MHz, CDCl₃) δ -60.35. ¹³C NMR (101 MHz, CDCl₃) δ 158.20, 137.17, 124.46 (q, J = 40.8 Hz), 122.75, 121.32 (q, J = 268.1 Hz), 120.80, 111.88, 104.32 (q, J = 3.4 Hz), 94.31, 55.54.



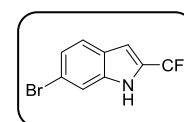
6-fluoro-2-(trifluoromethyl)-1H-indole(2o):¹ reddish brown liquid, 72% yield, Known compound, ¹H NMR (400 MHz, CDCl₃) δ 8.54 (s, 1H), 7.65 (dd, J = 8.6, 5.3 Hz, 1H), 7.15 (d, J = 9.2 Hz, 1H), 7.02 (t, J = 8.5 Hz, 1H), 6.96 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ -60.69, -116.72 (td, J₁ = 9.4 Hz, J₂ = 5.3 Hz). ¹³C NMR (101 MHz, CDCl₃) δ 161.23 (d, J = 242.5 Hz), 136.22 (d, J = 13.8 Hz), 126.39 (d, J = 3.1 Hz), 123.24, 123.14, 121.00 (q, J = 268.5 Hz), 110.49 (d, J = 25.1 Hz), 104.37 (q, J = 3.6 Hz), 97.91 (dq, J = 2.7 Hz).



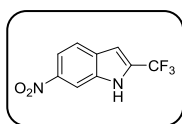
6-chloro-2-(trifluoromethyl)-1H-indole(2p):¹ reddish brown liquid, 61% yield, Known compound, ¹H NMR (400 MHz, CDCl₃) δ 8.40 (s, 1H), 7.58 (d, J = 8.5 Hz, 1H), 7.41 (s, 1H), 7.16 (dd, J = 8.5, 1.7 Hz, 1H), 6.90 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ -60.73. ¹³C NMR (101 MHz, CDCl₃) δ 136.42, 130.73, 126.41 (q, J = 39.2 Hz), 125.13, 123.01, 122.15, 120.94 (q, J = 269.0 Hz), 111.61, 104.32 (q, J = 3.4 Hz).



6-bromo-2-(trifluoromethyl)-1H-indole(2q): yellow liquid, 58% yield, Known compound, ¹H NMR (400 MHz, CDCl₃) δ 8.35 (s, 1H), 7.51 – 7.40 (m, 2H), 7.22 (d, J = 8.5 Hz, 1H), 6.81 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ -60.72 (s). ¹³C NMR (101 MHz, CDCl₃) δ 136.78, 126.29 (q, J = 39.3 Hz), 125.42, 124.68, 123.30, 120.90 (q, J = 269.1 Hz), 118.35, 114.64, 104.35 (q, J = 3.2 Hz). HRMS (ESI) m/z calculated for [C₉H₅BrF₃NNa]⁺ 285.9450; Found: 285.9455.



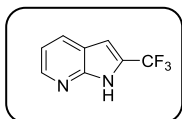
6-nitro-2-(trifluoromethyl)-1H-indole(2r):⁷ bright yellow solid: mp 139-140 °C, 51% yield,



Known compound, ¹H NMR (400 MHz, CDCl₃) δ 9.30 (s, 1H), 8.46 (s, 1H), 8.10 (d, *J* = 8.7 Hz, 1H), 7.79 (d, *J* = 8.8 Hz, 1H), 7.04 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ -61.27. ¹³C NMR (126 MHz, CDCl₃) δ 145.10, 134.70, 131.19, 131.07 (q, *J* = 42.7 Hz), 122.45, 120.70 (q, *J* = 193.9 Hz), 116.22, 108.92, 104.29 (q, *J* = 3.3

Hz).

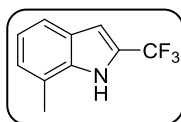
2-(trifluoromethyl)-1H-pyrrolo[2,3-b]pyridine(2s):⁸ brown yellow solid: mp 143-144 °C,



64% yield, Known compound, ¹H NMR (400 MHz, CDCl₃) δ 14.35 (s, 1H), 8.47 (d, *J* = 3.6 Hz, 1H), 8.07 (d, *J* = 7.8 Hz, 1H), 7.22 – 7.18 (m, 1H), 6.88 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ -61.18. ¹³C NMR (101 MHz, CDCl₃) δ 148.53, 144.48, 131.43, 127.41 (q, *J* = 39.3 Hz), 121.36 (q, *J* = 245.4 Hz), 119.82, 116.87,

101.32 (q, *J* = 4.0 Hz).

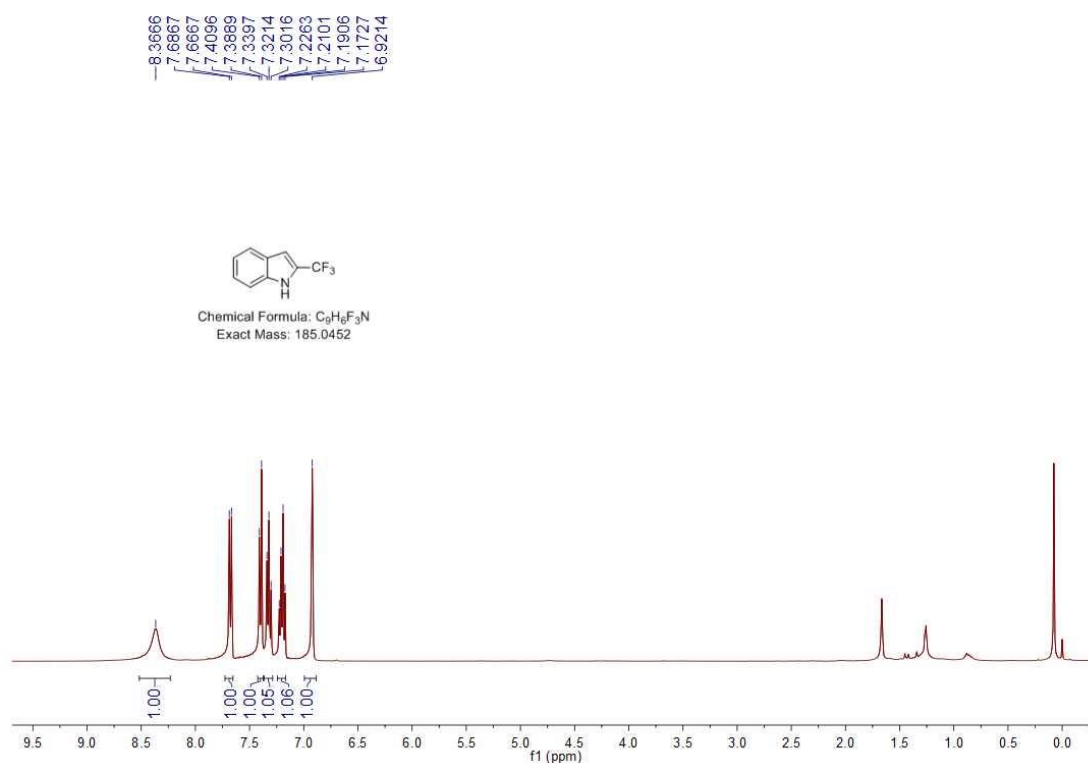
7-methyl-2-(trifluoromethyl)-1H-indole(2t):³ reddish-brown liquid, 57% yield, Known



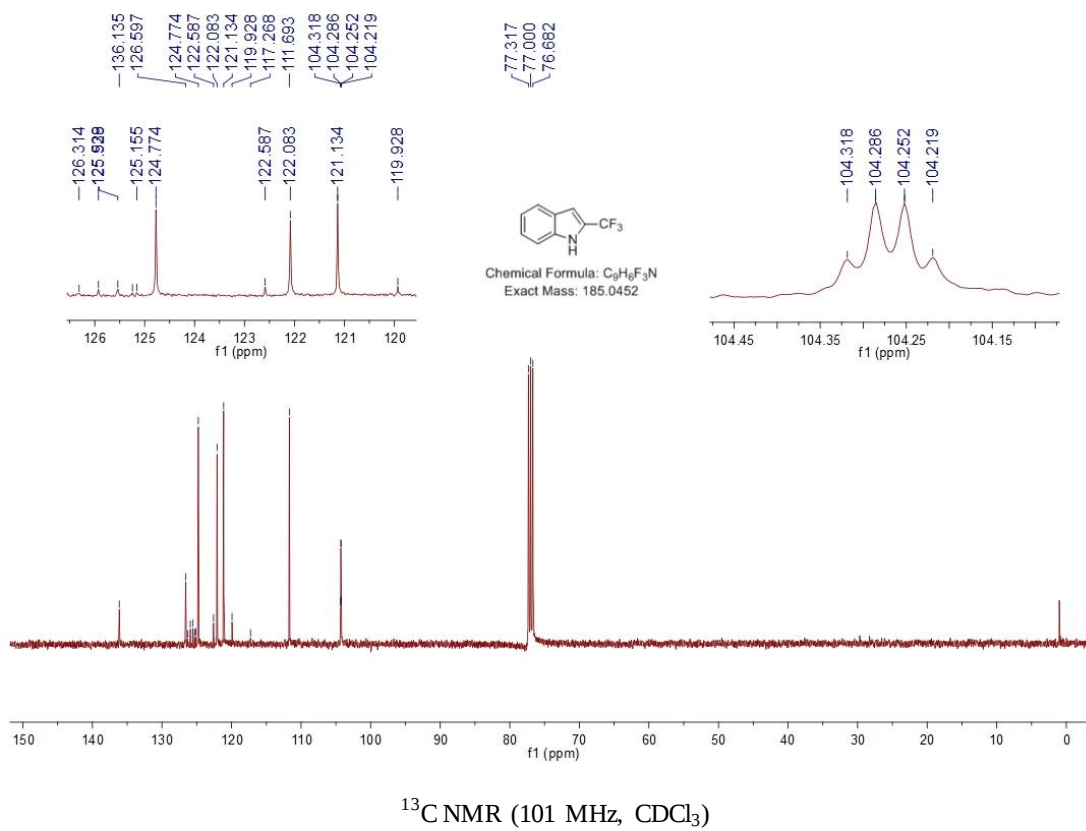
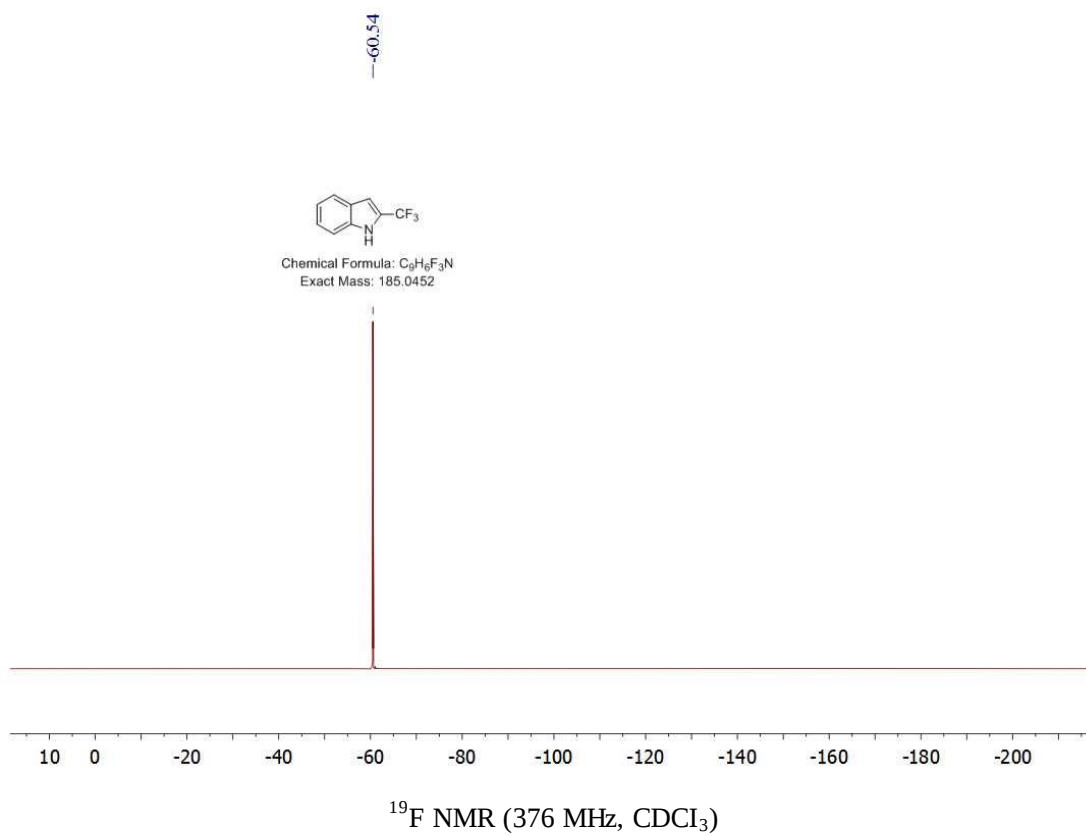
compound, ¹H NMR (400 MHz, CDCl₃) δ 8.21 (s, 1H), 7.45 – 7.41 (m, 1H), 7.05 – 7.00 (m, 2H), 6.84 (s, 1H), 2.40 (s, 3H). ¹⁹F NMR (376 MHz, CDCl₃) δ -60.39 (s). ¹³C NMR (101 MHz, CDCl₃) δ 135.96, 126.19, 125.45 (q, *J* = 39.0 Hz) 125.09, 121.35, 121.39 (q, *J* = 268.7 Hz), 121.00, 119.70, 104.79 (q, *J* = 3.3 Hz), 16.49.

4. NMR Spectra for the Compounds

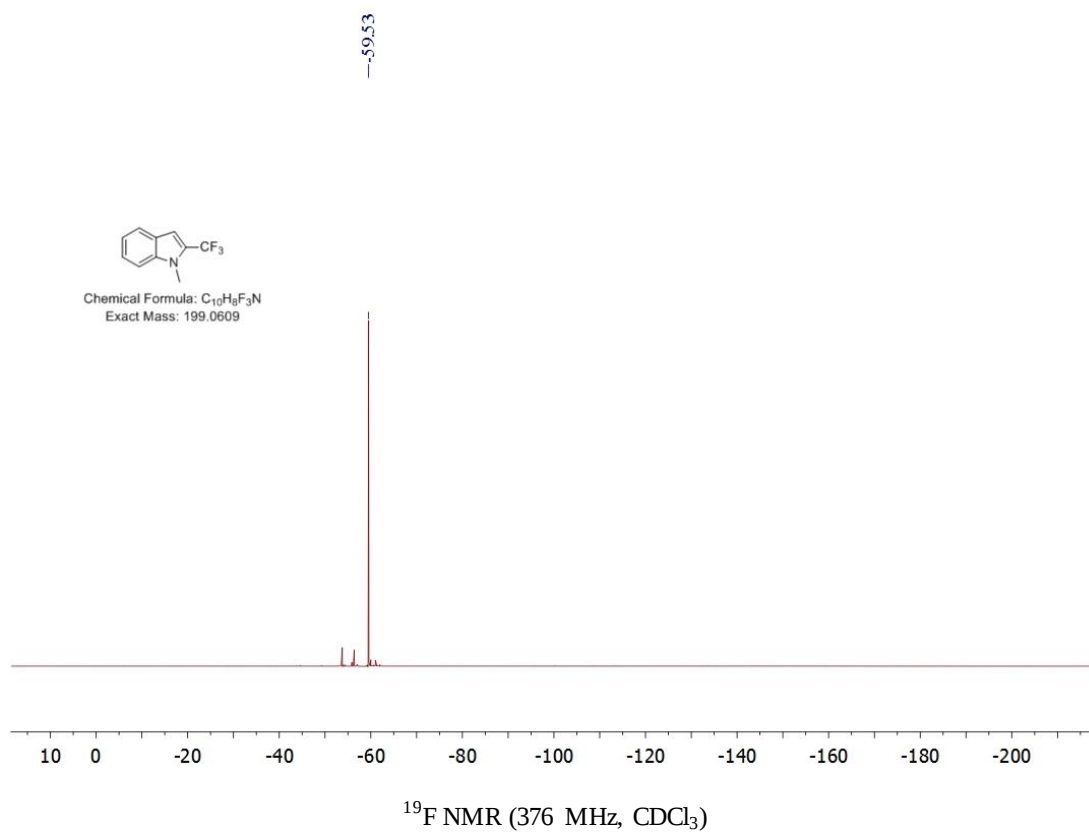
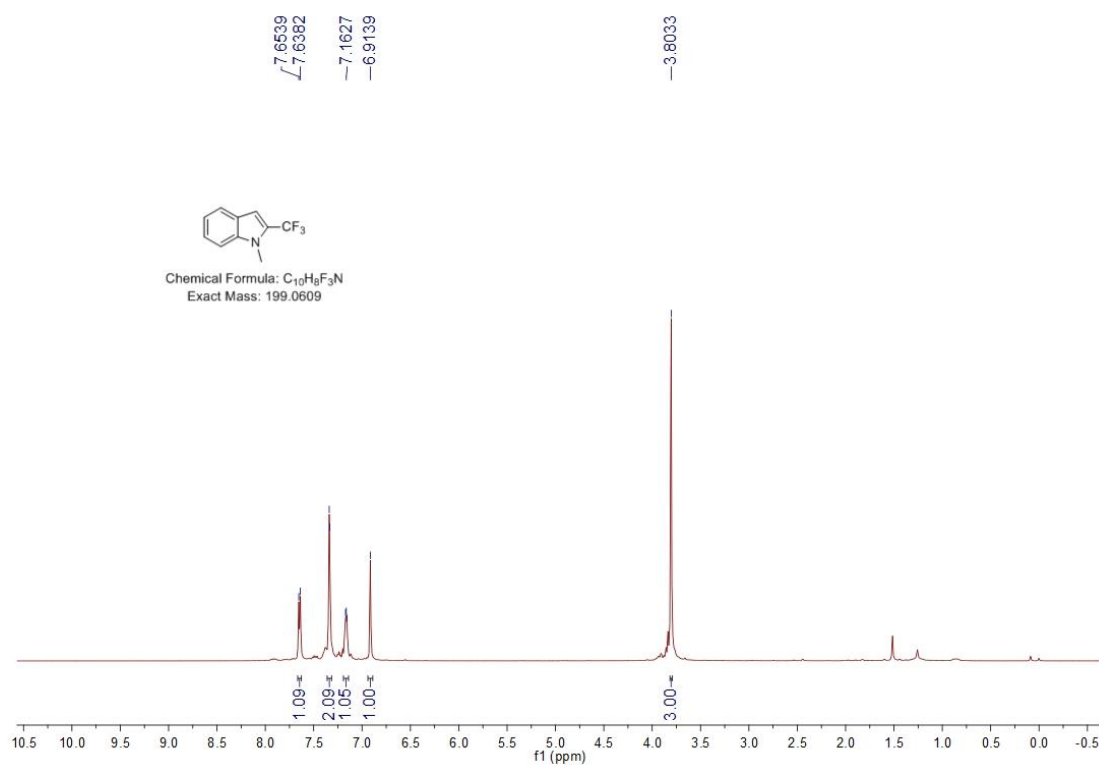
2-(trifluoromethyl)-1H-indole(2a)

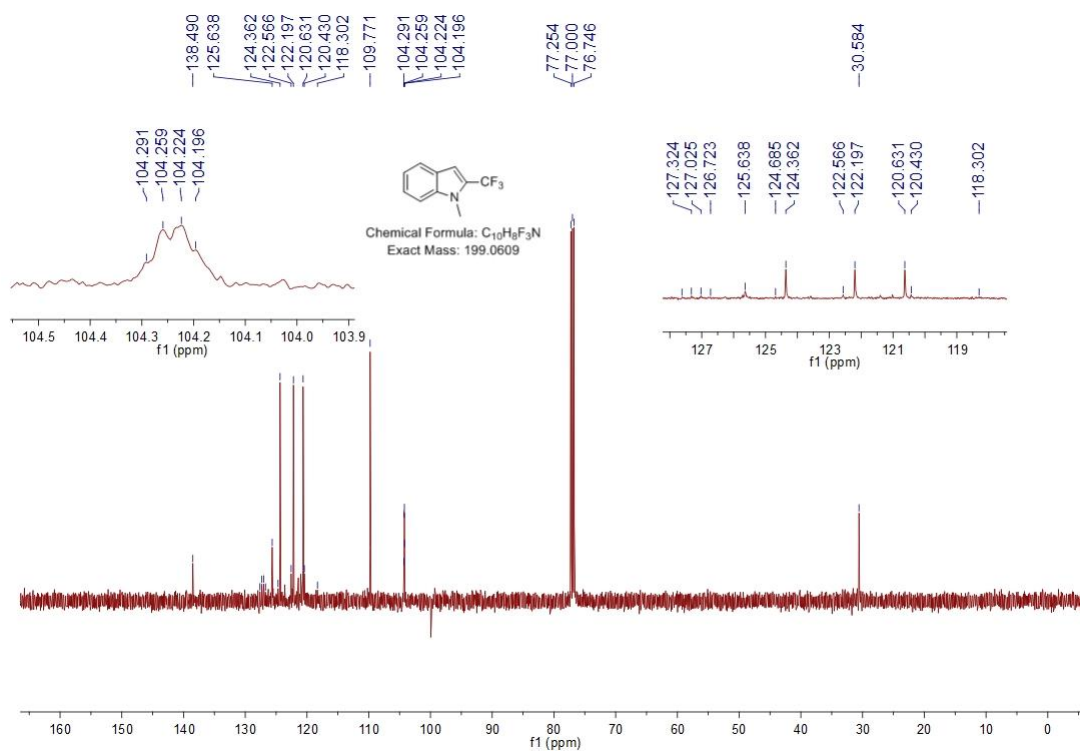


¹H NMR (400 MHz, CDCl₃)



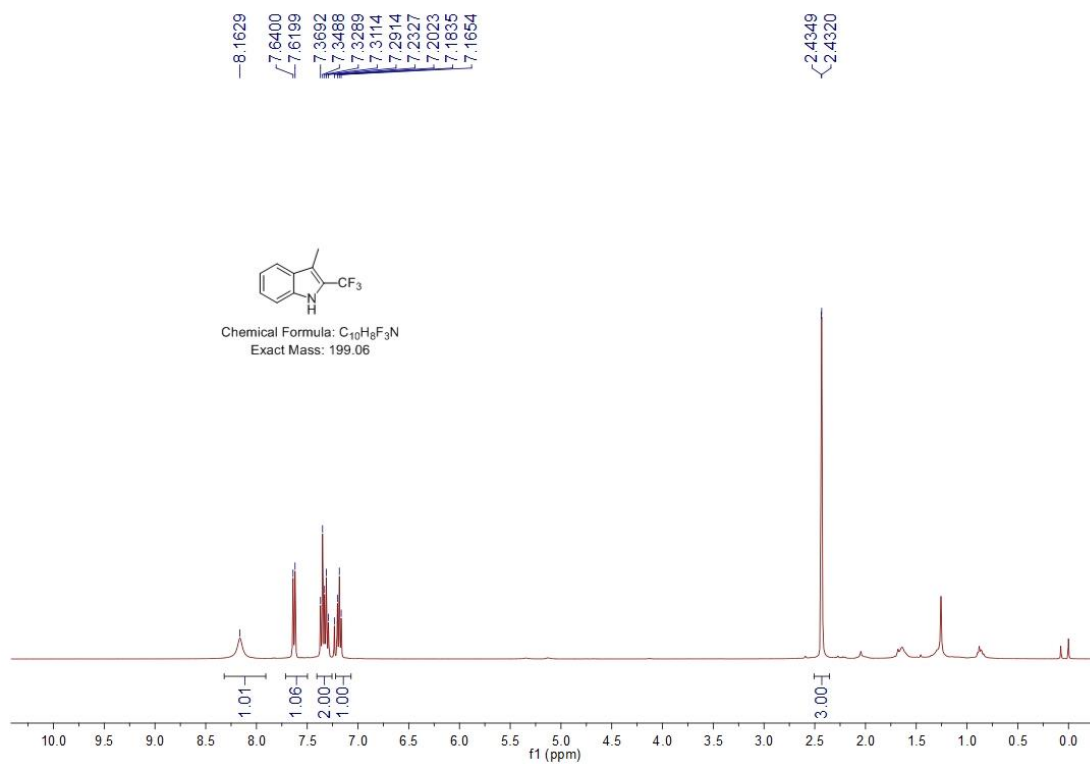
1-methyl-2-(trifluoromethyl)-1H-indole(2b)



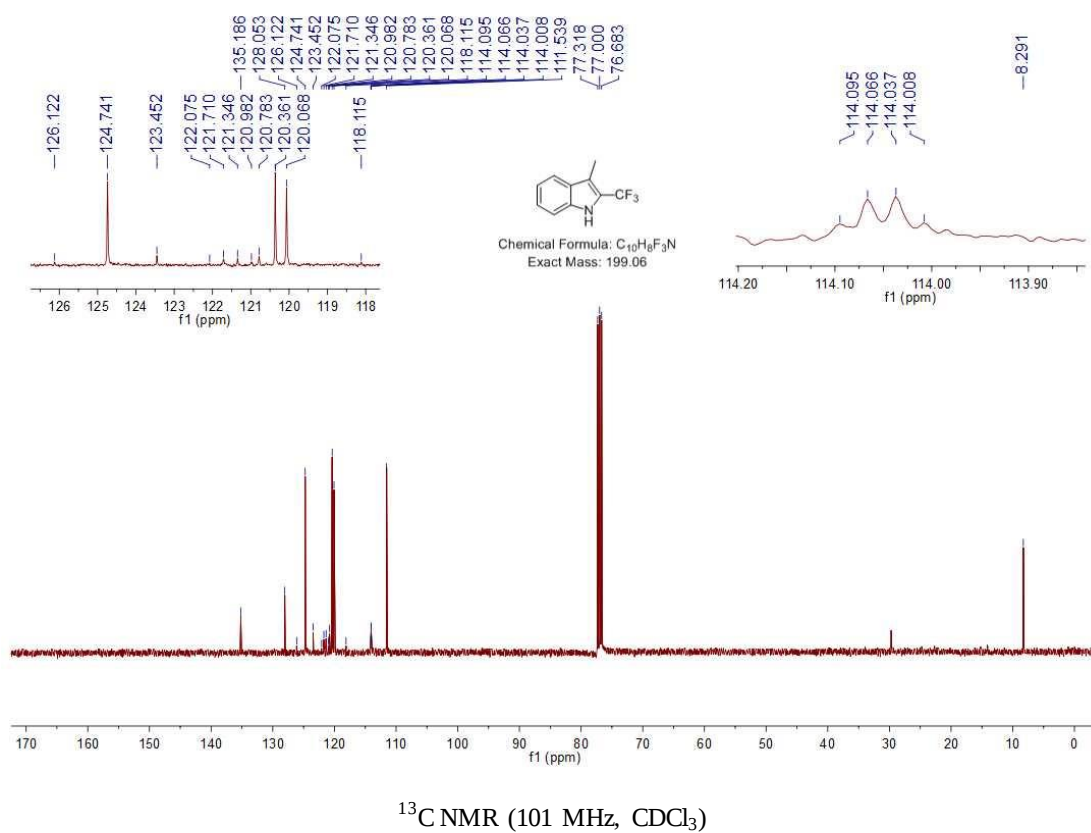
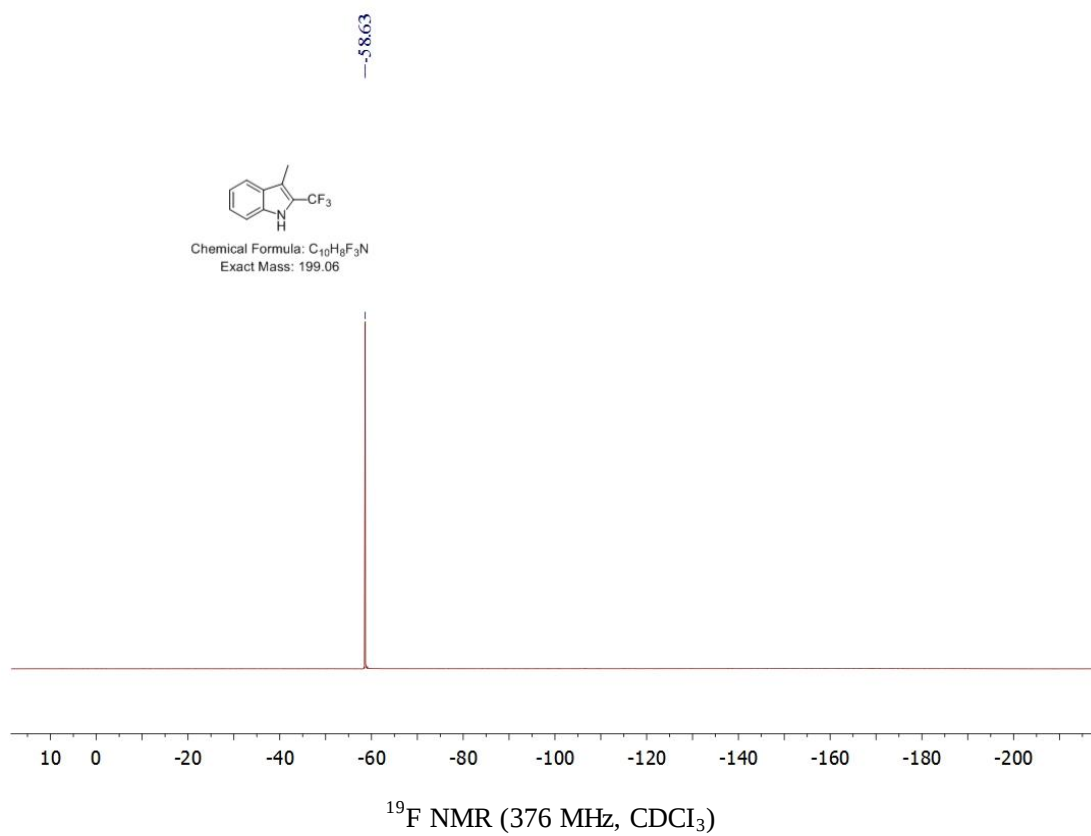


^{13}C NMR (126 MHz, $CDCl_3$)

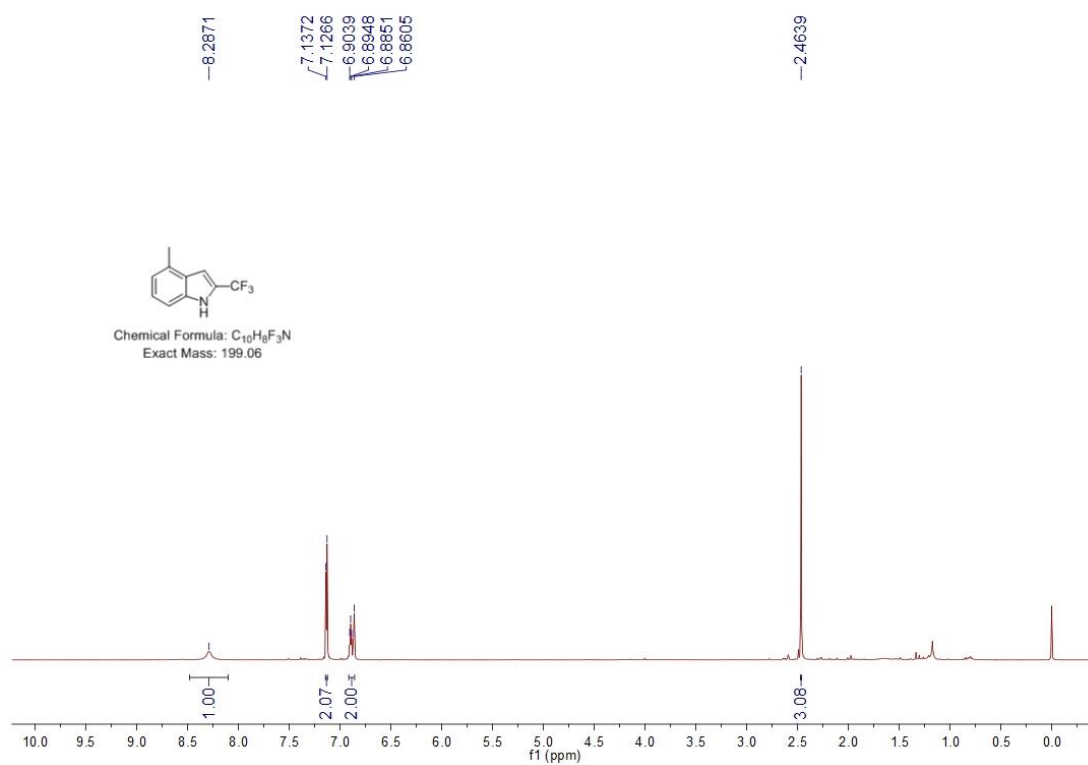
3-methyl-2-(trifluoromethyl)-1H-indole(2c)



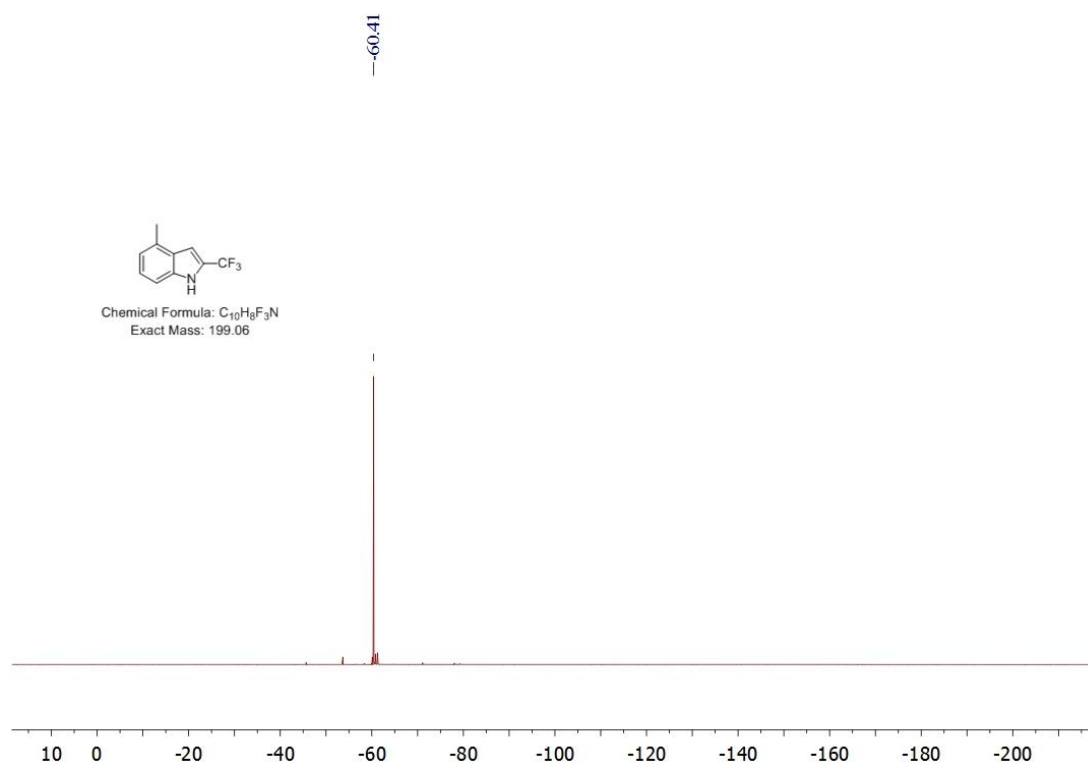
1H NMR (400 MHz, $CDCl_3$)



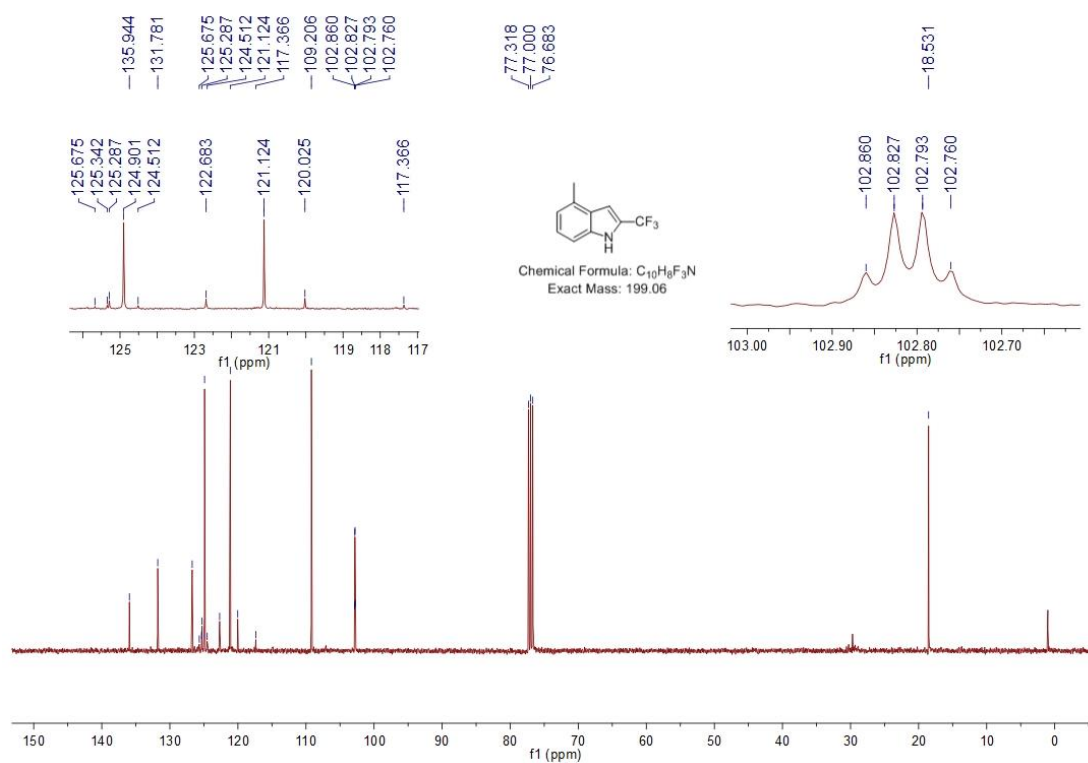
4-methyl-2-(trifluoromethyl)-1H-indole(2d)



1H NMR (400 MHz, $CDCl_3$)

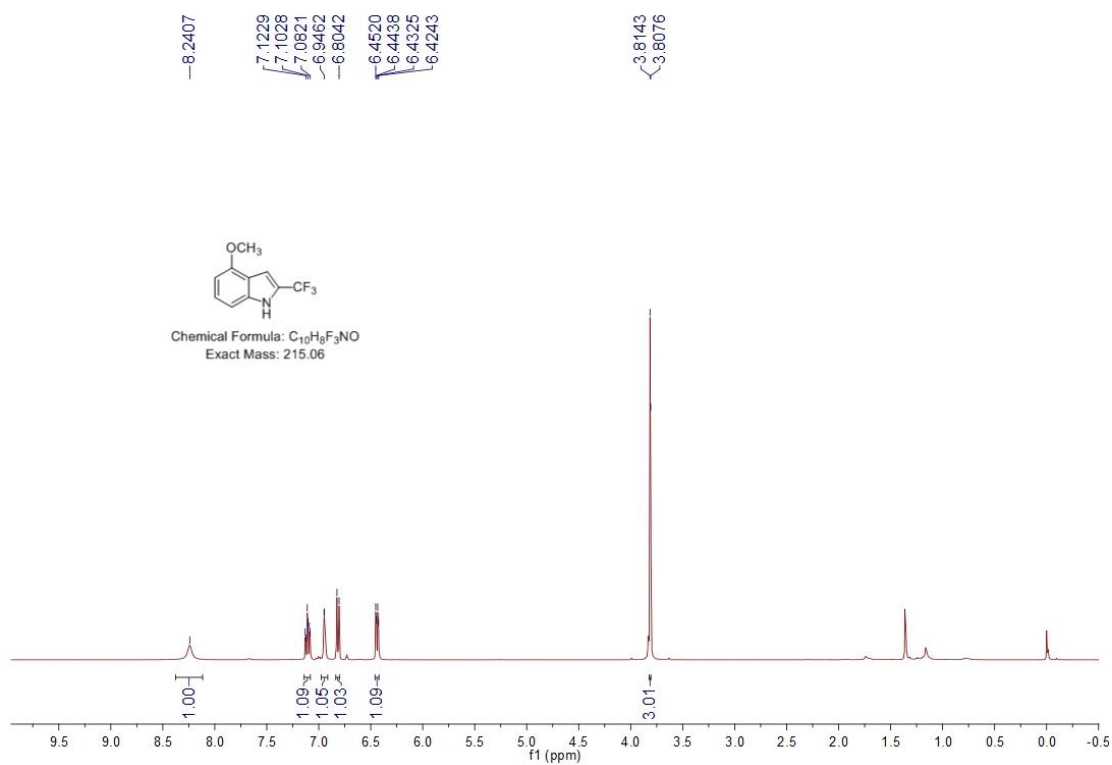


^{19}F NMR (376 MHz, $CDCl_3$)

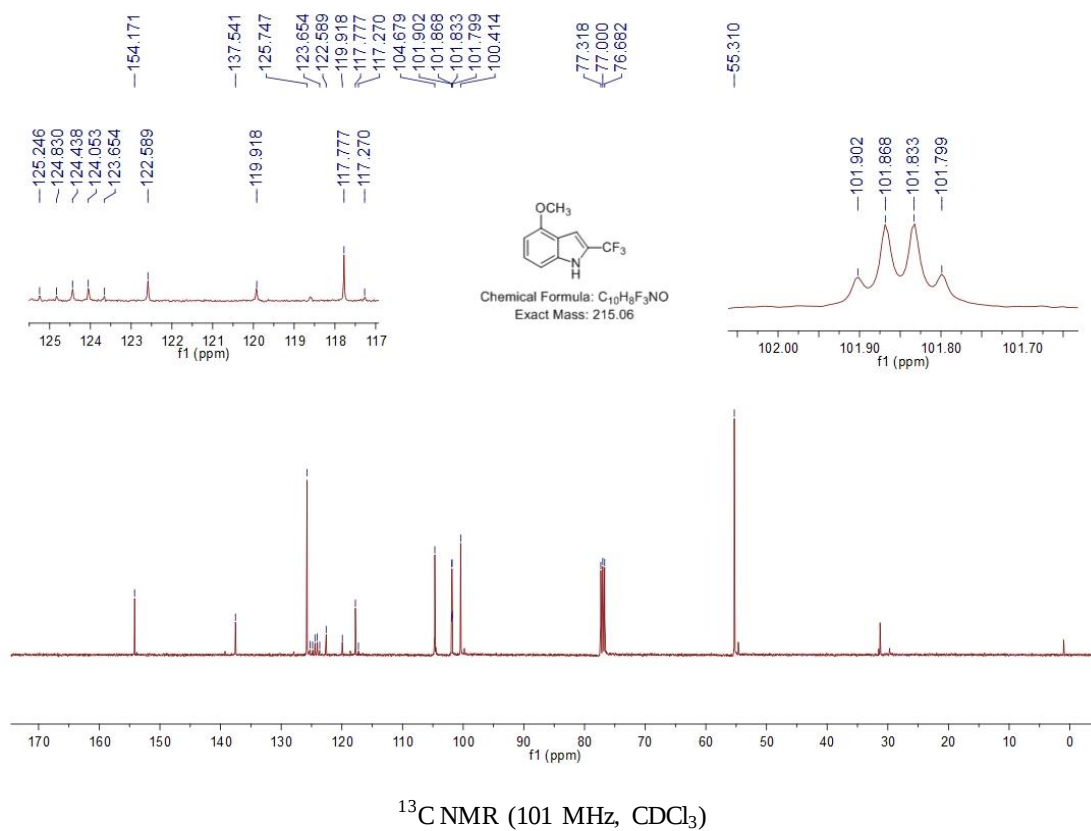
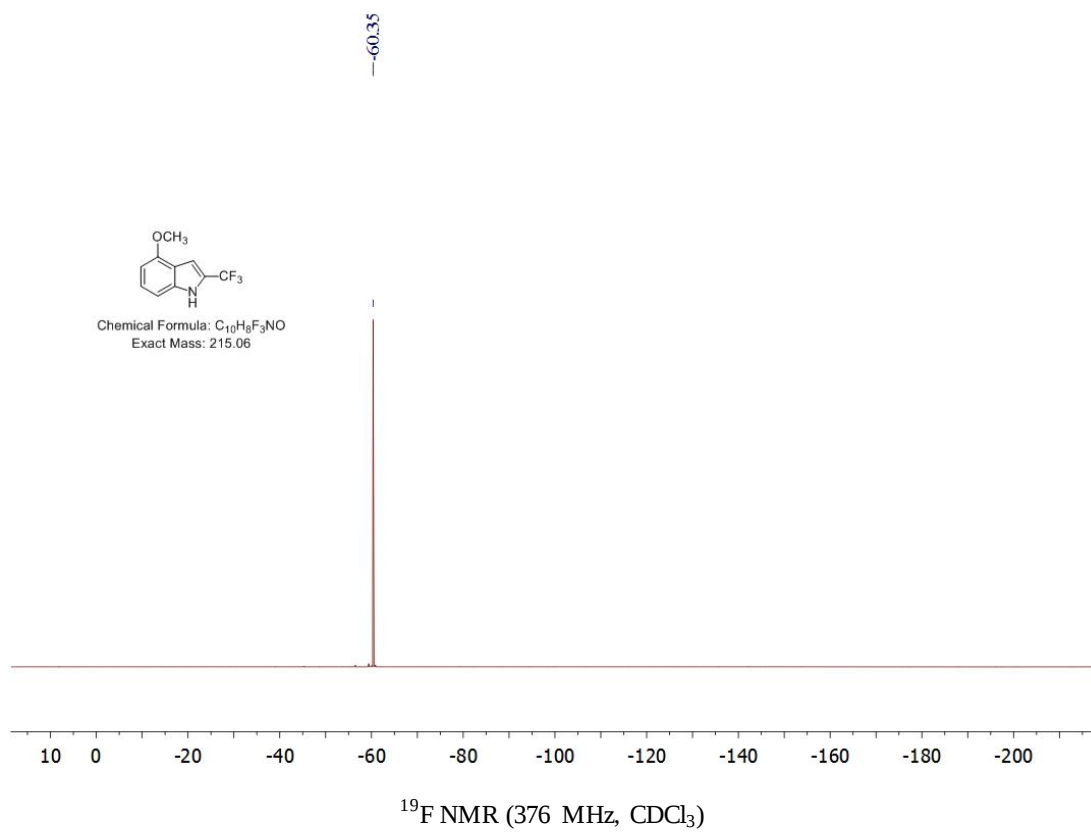


^{13}C NMR (101 MHz, $CDCl_3$)

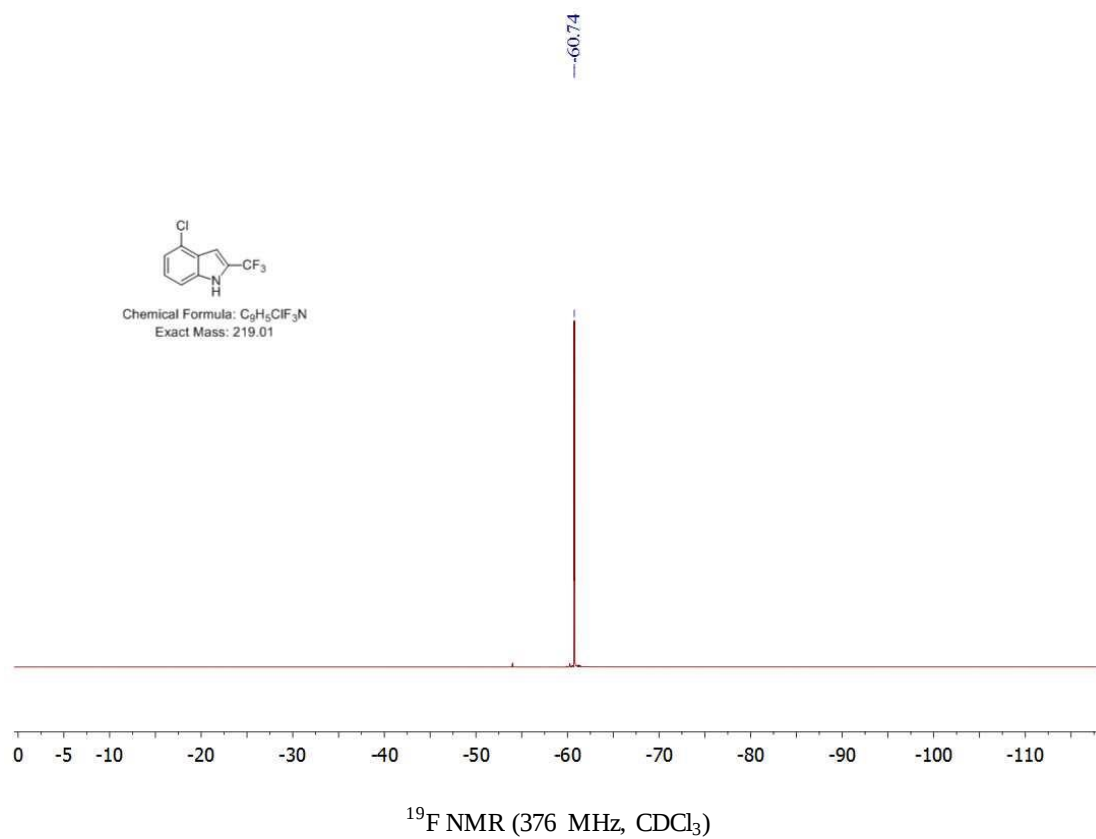
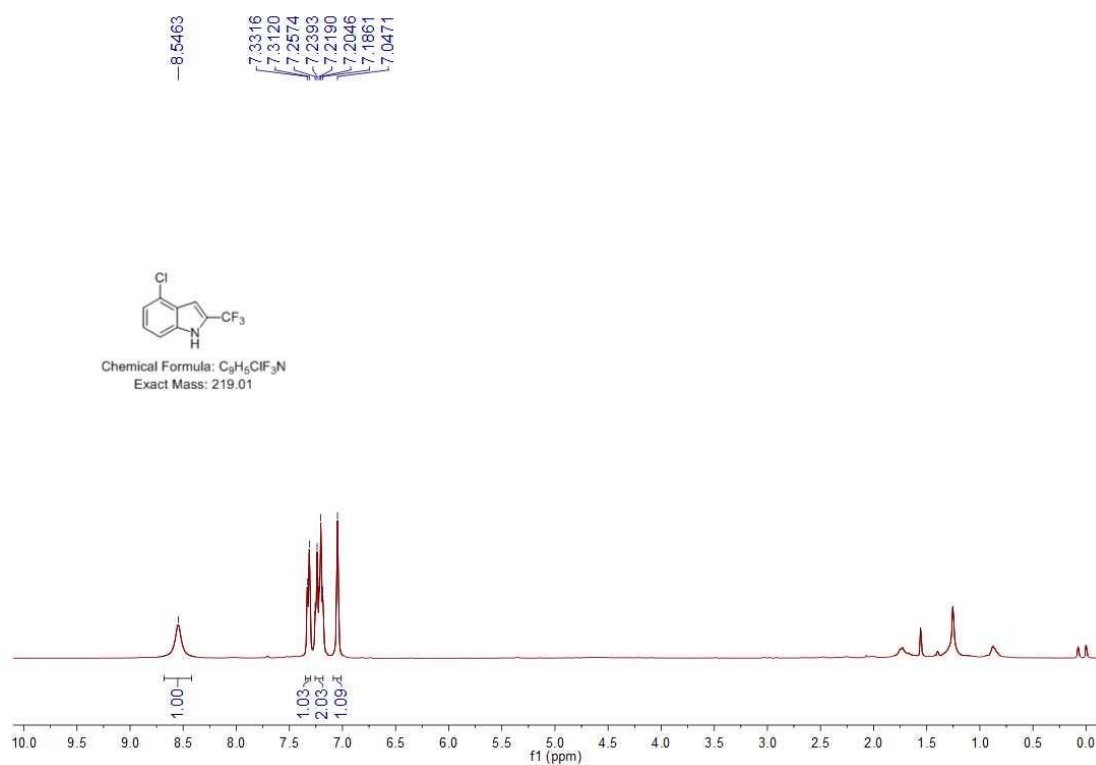
4-methoxy-2-(trifluoromethyl)-1H-indole(2e)

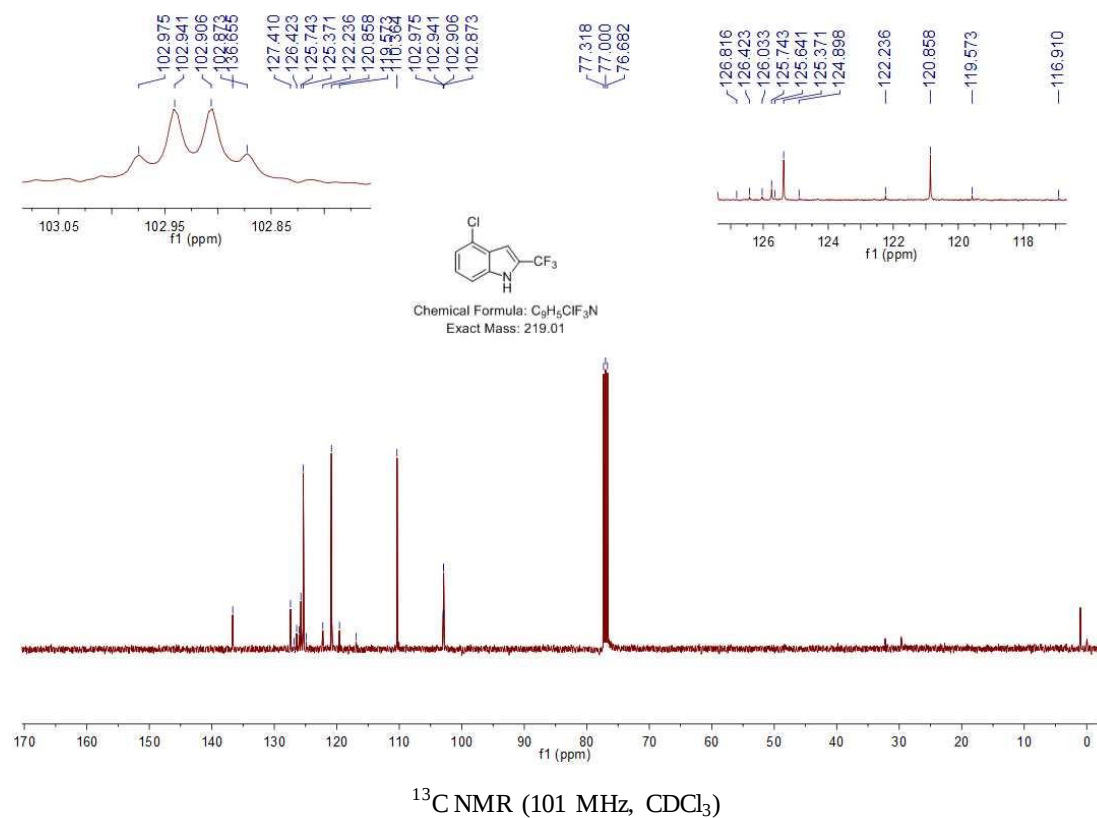


1H NMR (400 MHz, $CDCl_3$)

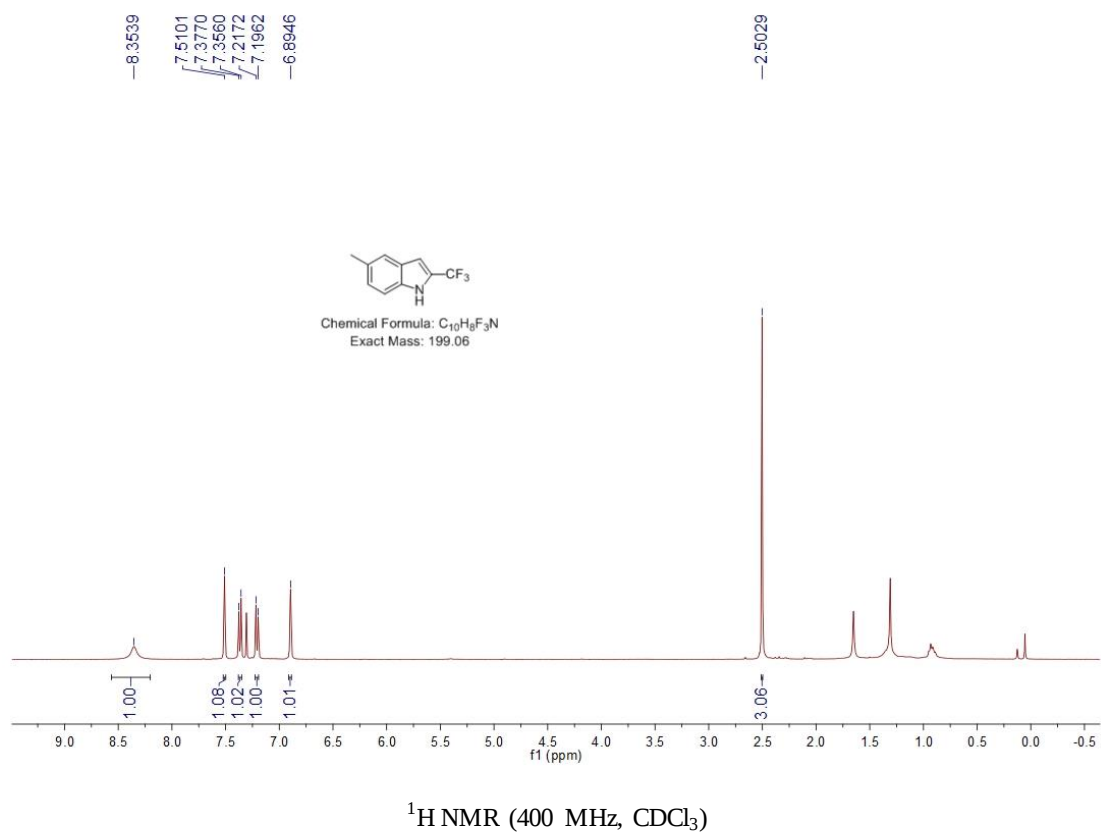


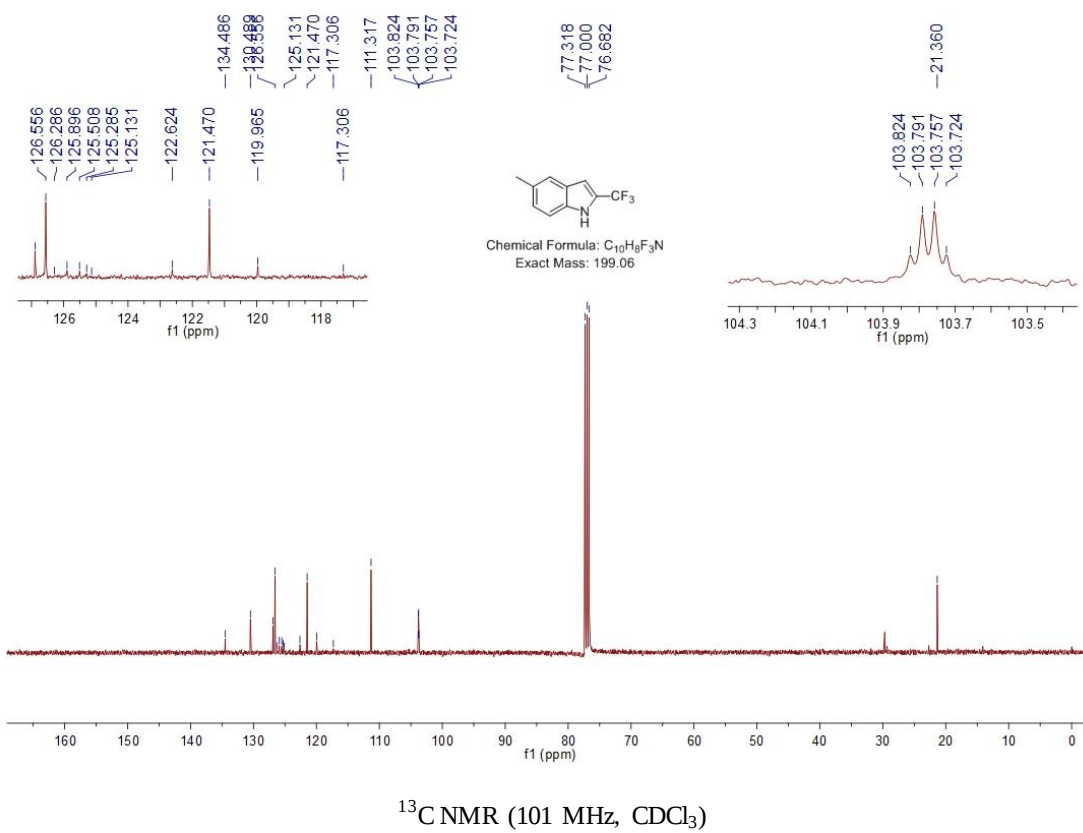
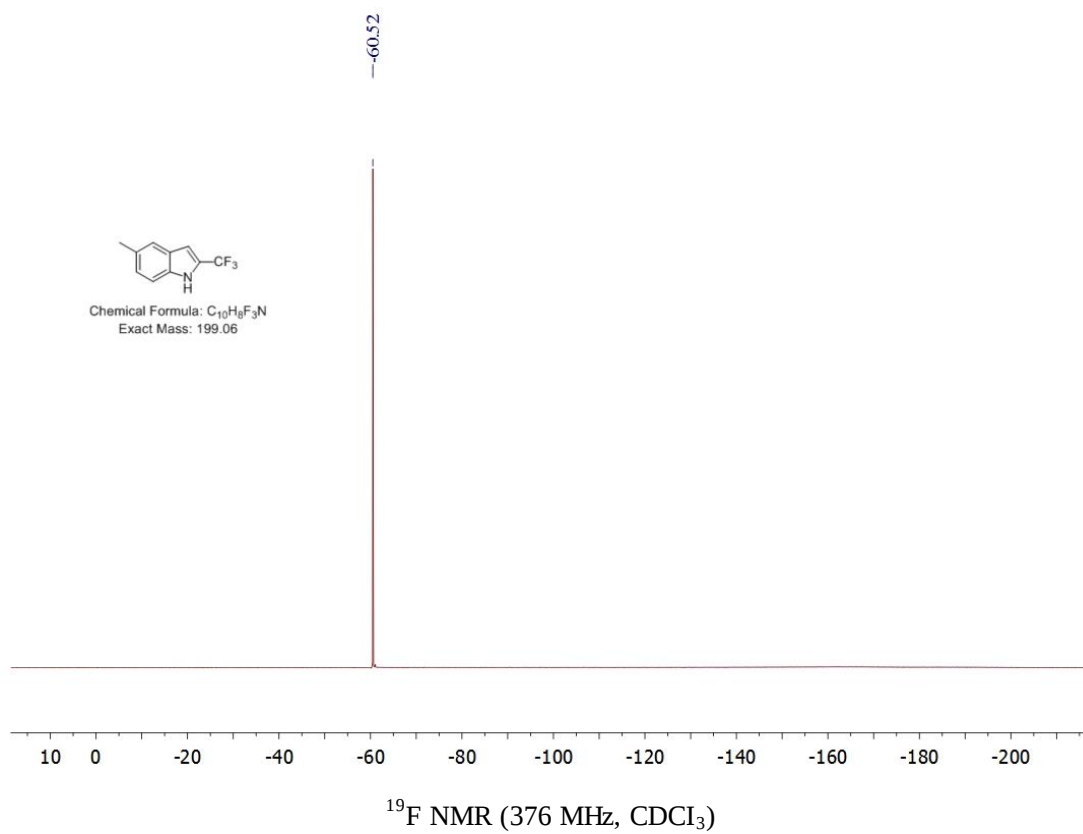
4-chloro-2-(trifluoromethyl)-1H-indole(2f)



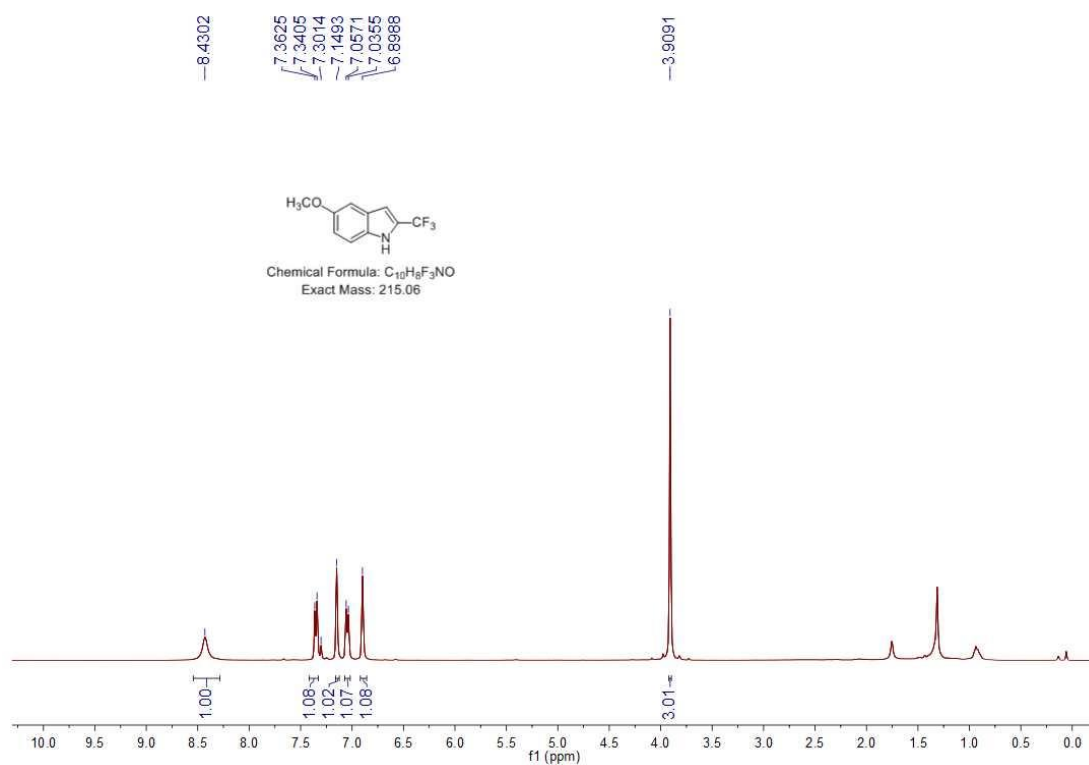


5-methyl-2-(trifluoromethyl)-1H-indole(2g)

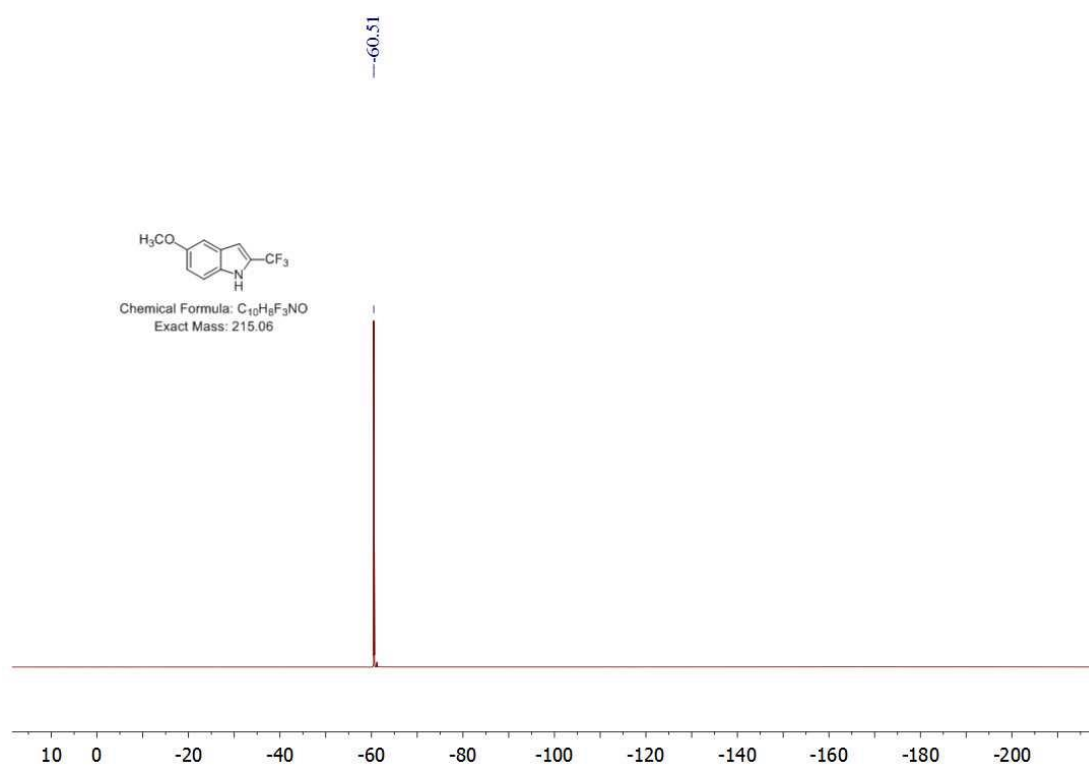




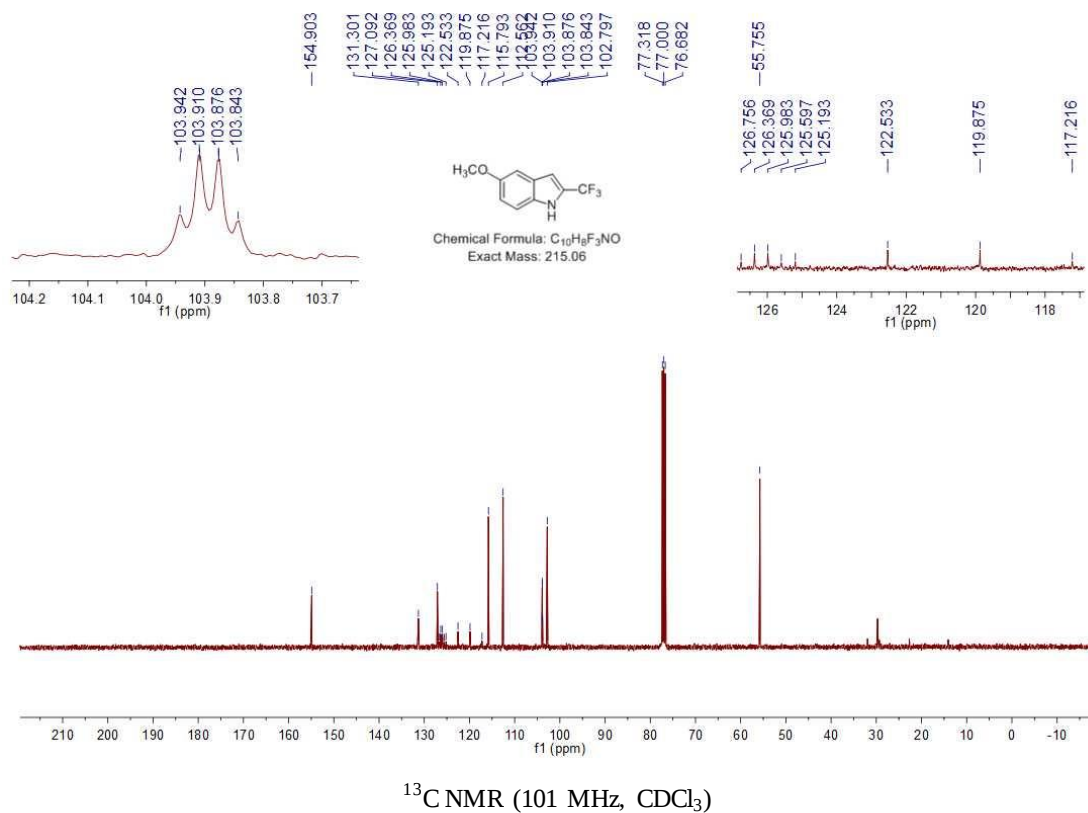
5-methoxy-2-(trifluoromethyl)-1H-indole(2h)



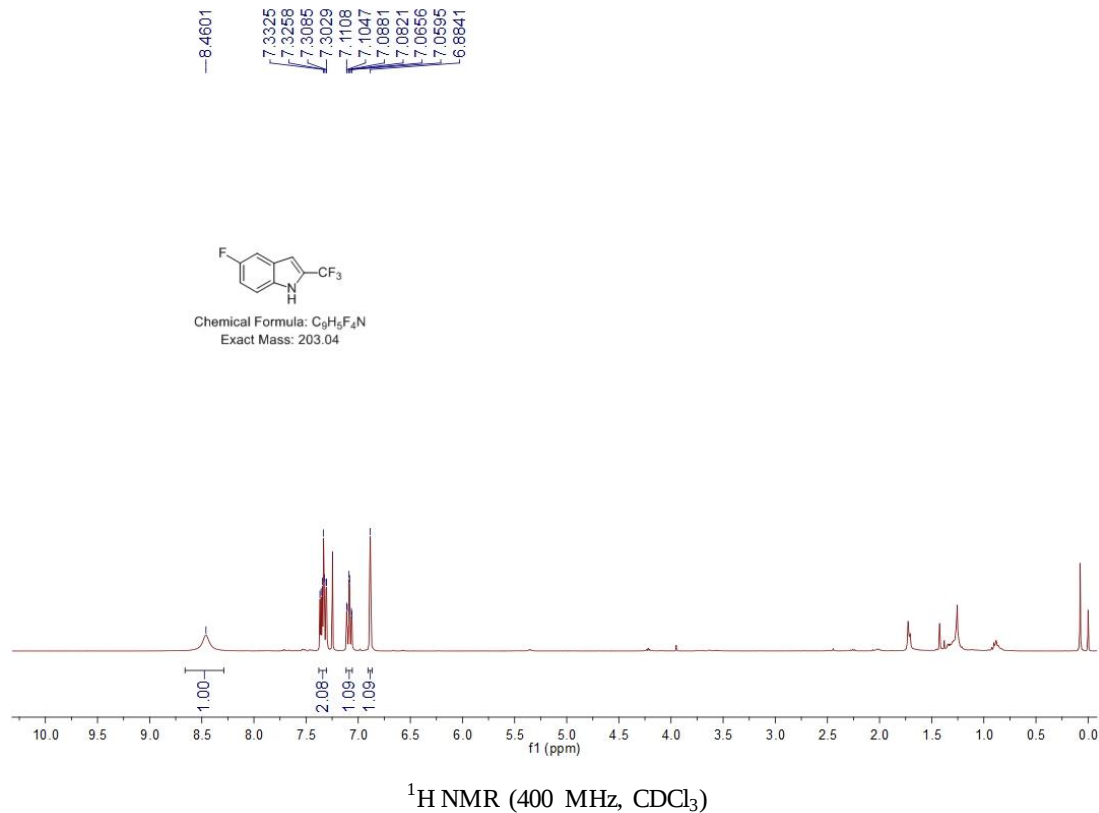
¹H NMR (400 MHz, CDCl₃)

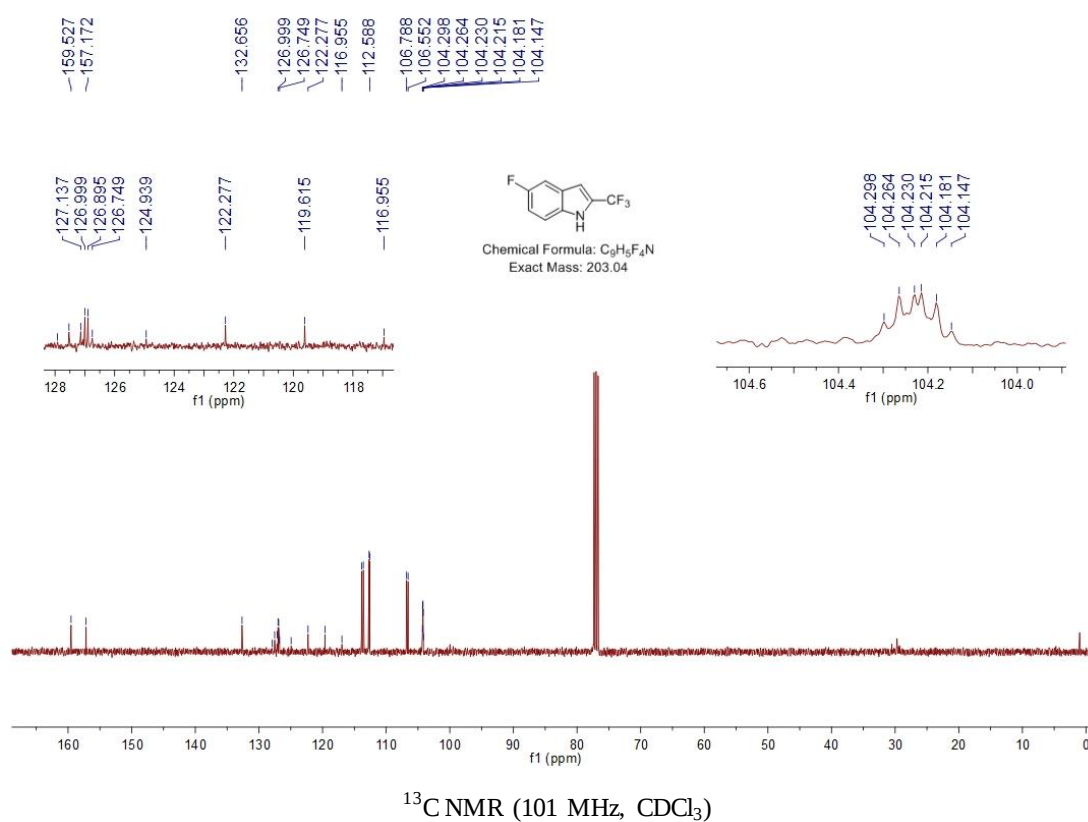
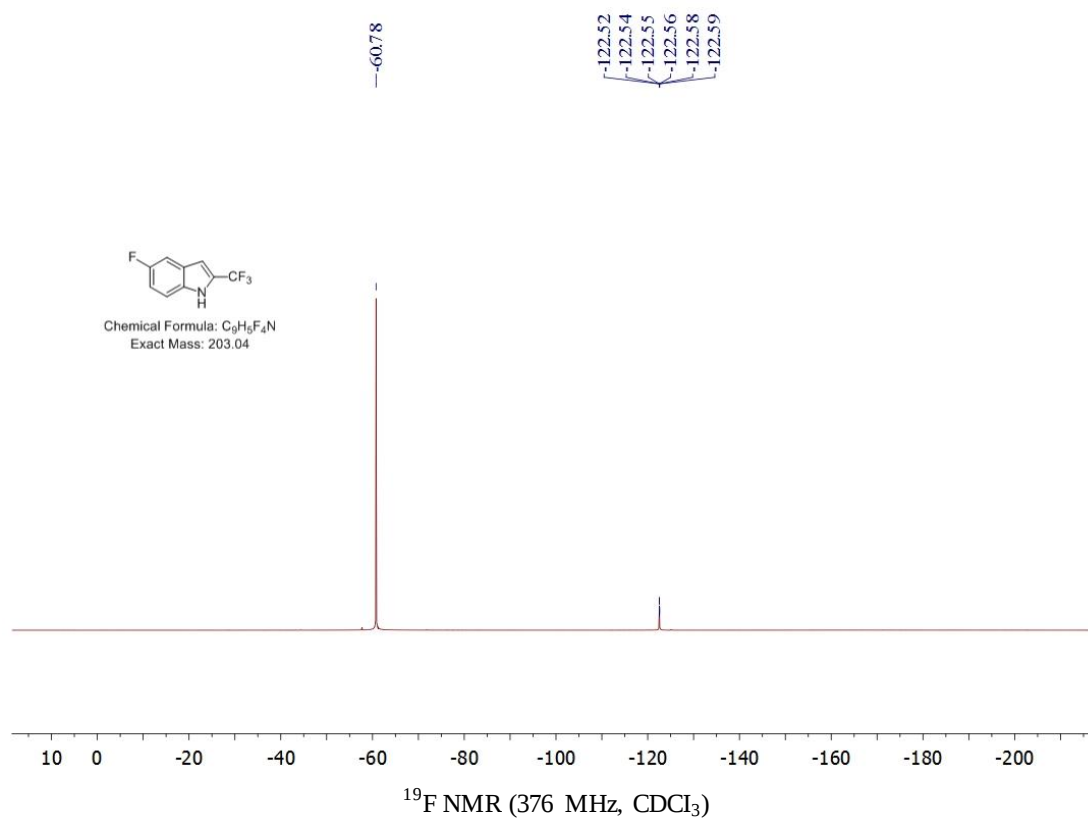


¹⁹F NMR (376 MHz, CDCl₃)



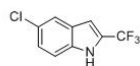
5-fluoro-2-(trifluoromethyl)-1H-indole(2i)



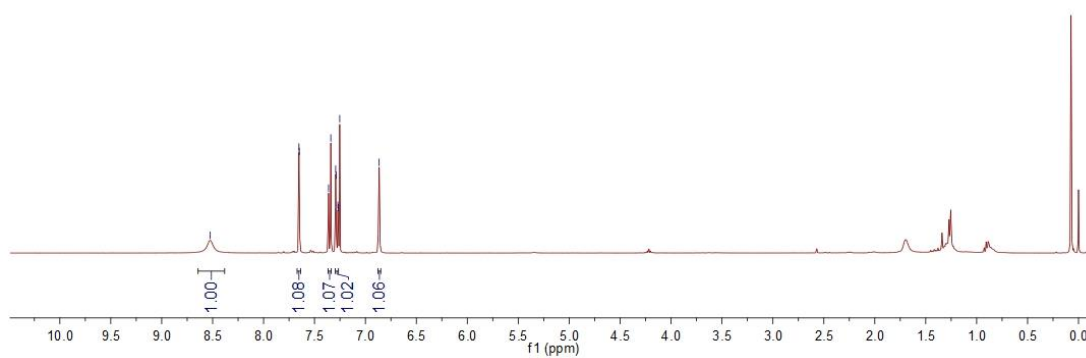


5-chloro-2-(trifluoromethyl)-1H-indole(2j)

δ 8.5236
 δ 7.6526
 δ 7.6493
 δ 7.2878
 δ 7.2525
 δ 6.8655

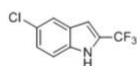


Chemical Formula: $C_9H_5ClF_3N$
 Exact Mass: 219.0063

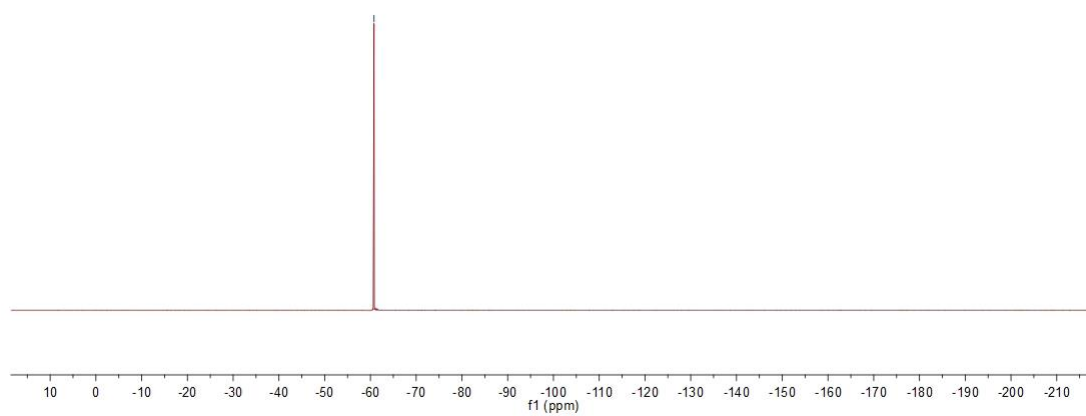


1H NMR (400 MHz, $CDCl_3$)

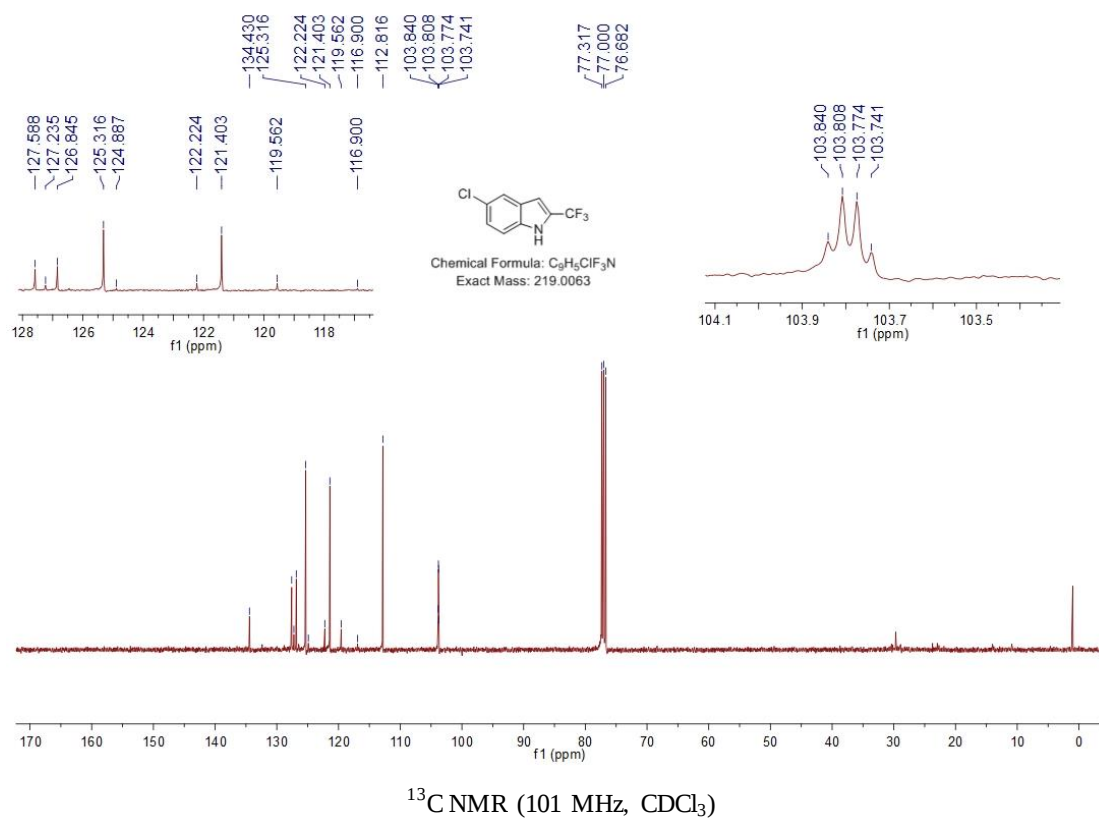
δ -60.79



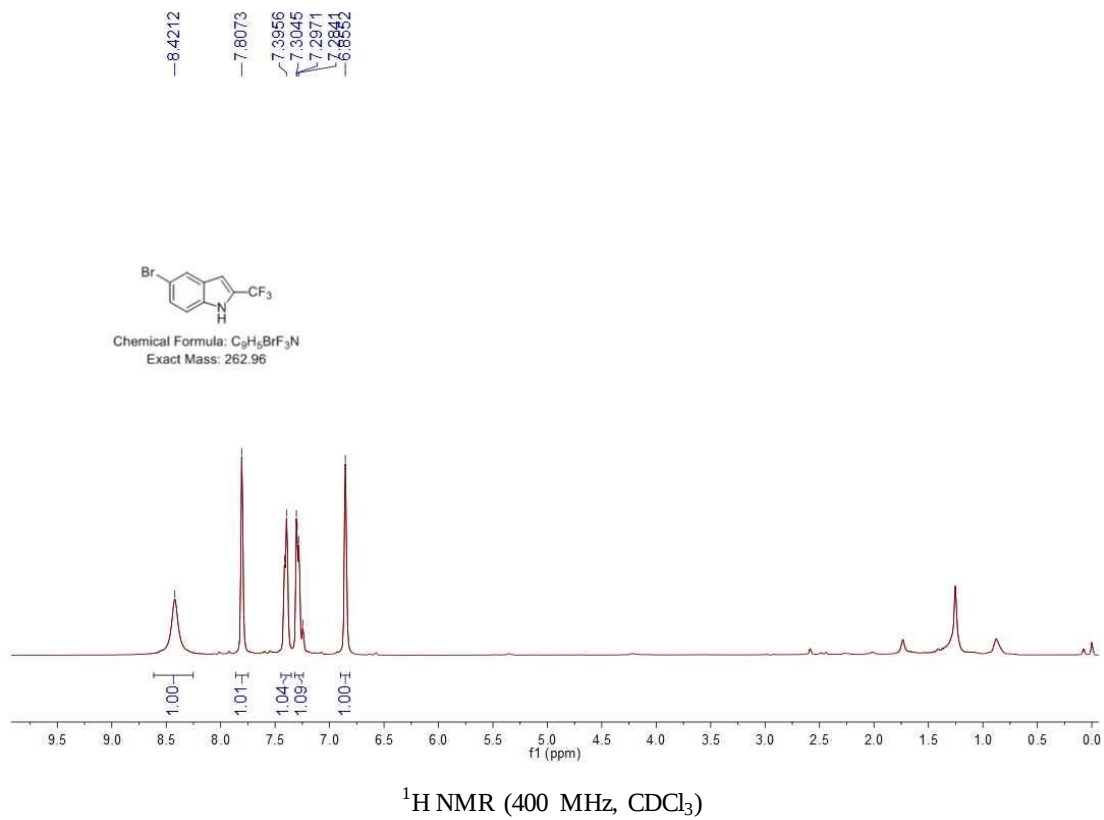
Chemical Formula: $C_9H_5ClF_3N$
 Exact Mass: 219.0063

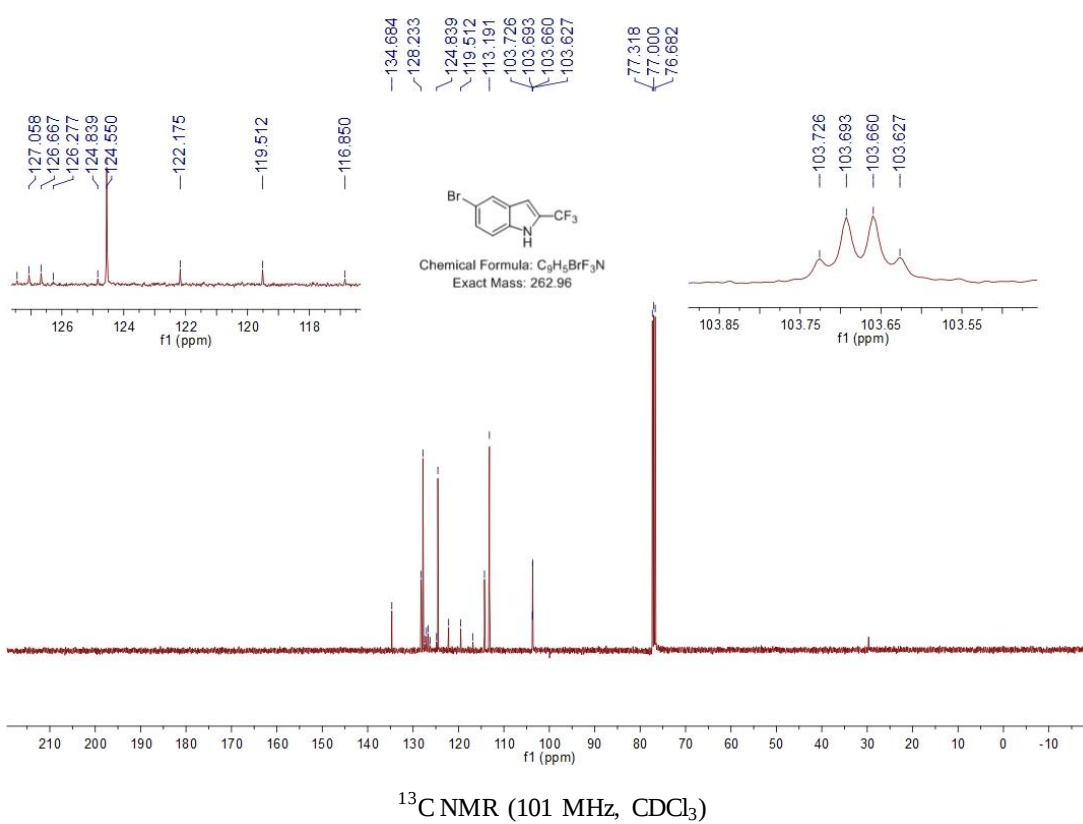
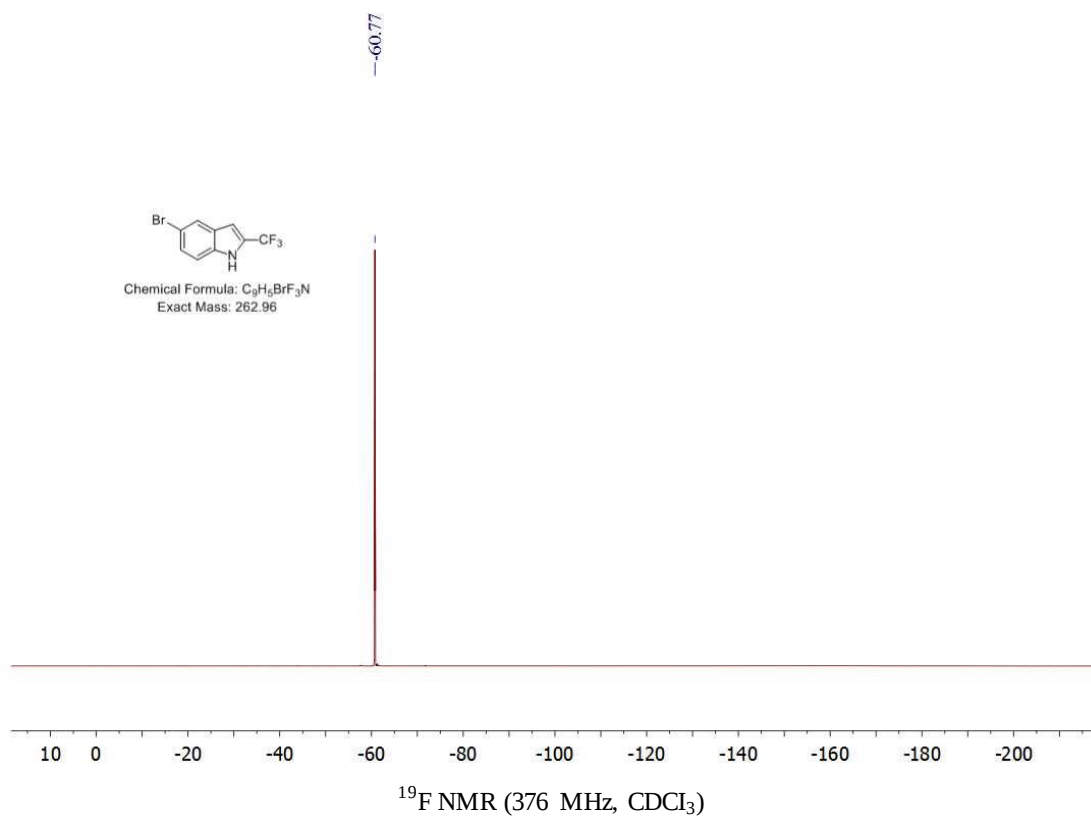


^{19}F NMR (376 MHz, $CDCl_3$)



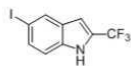
5-bromo-2-(trifluoromethyl)-1H-indole(2k)



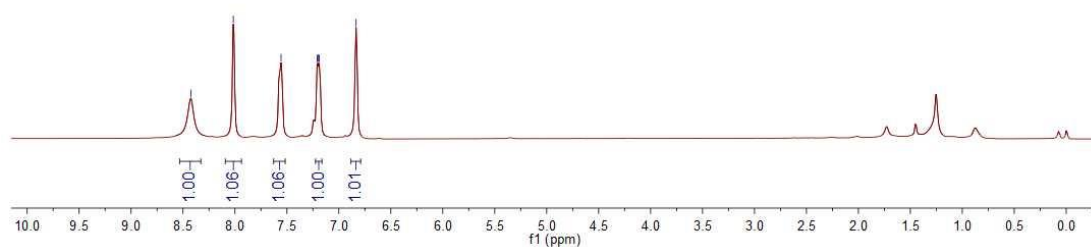


5-iodo-2-(trifluoromethyl)-1H-indole (2l)

8.4247
8.0163
7.5566
7.2093
7.1897
6.8347

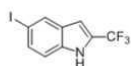


Chemical Formula: $C_9H_5F_3IN$
Exact Mass: 310.94

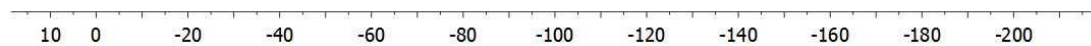


1H NMR (400 MHz, $CDCl_3$)

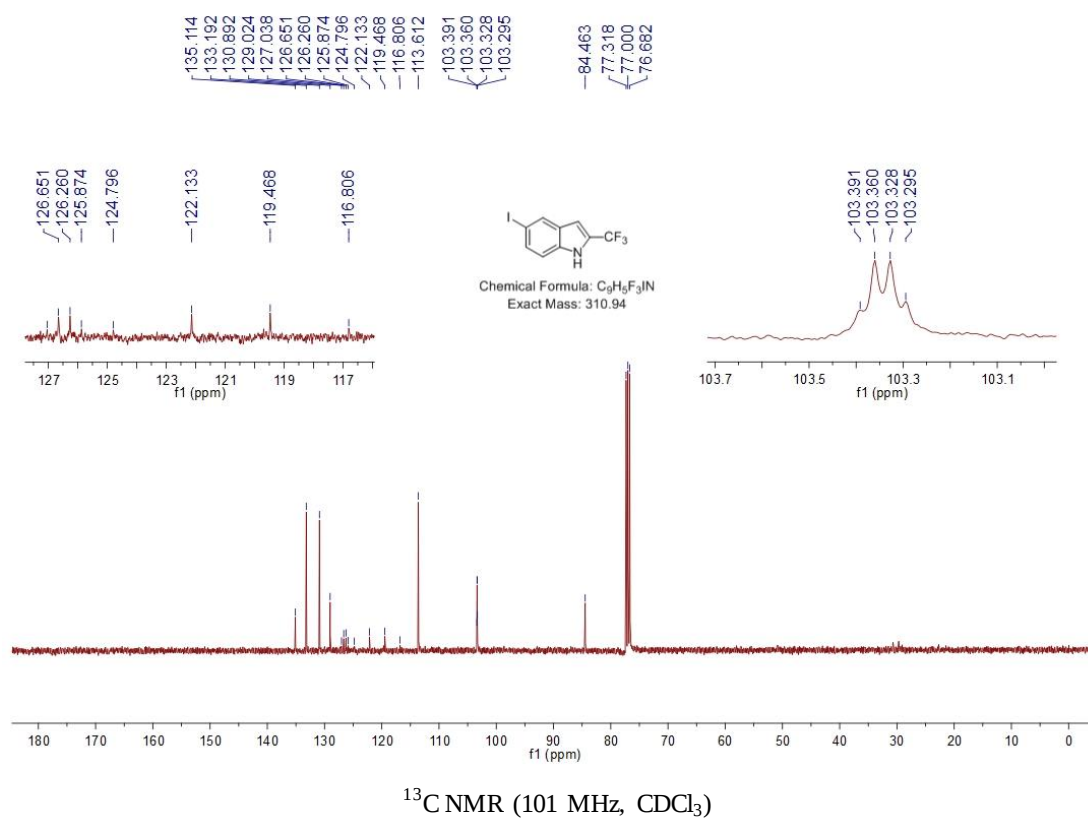
-60.72



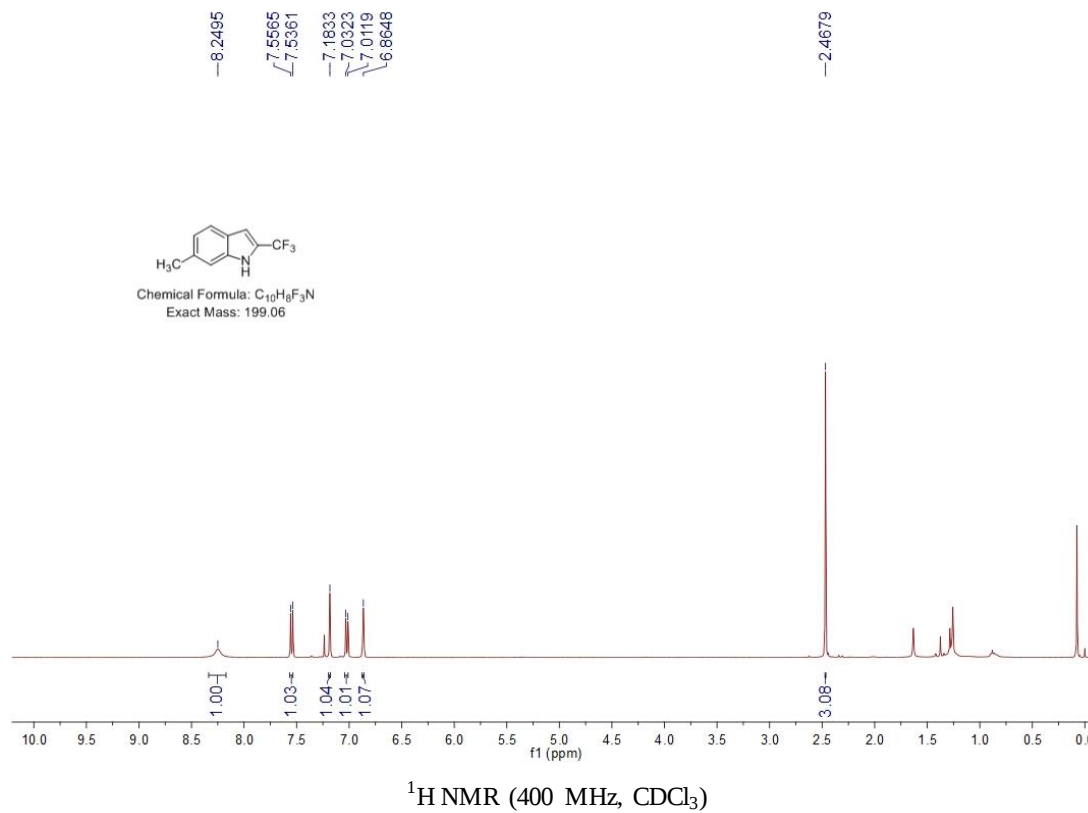
Chemical Formula: $C_9H_5F_3IN$
Exact Mass: 310.94

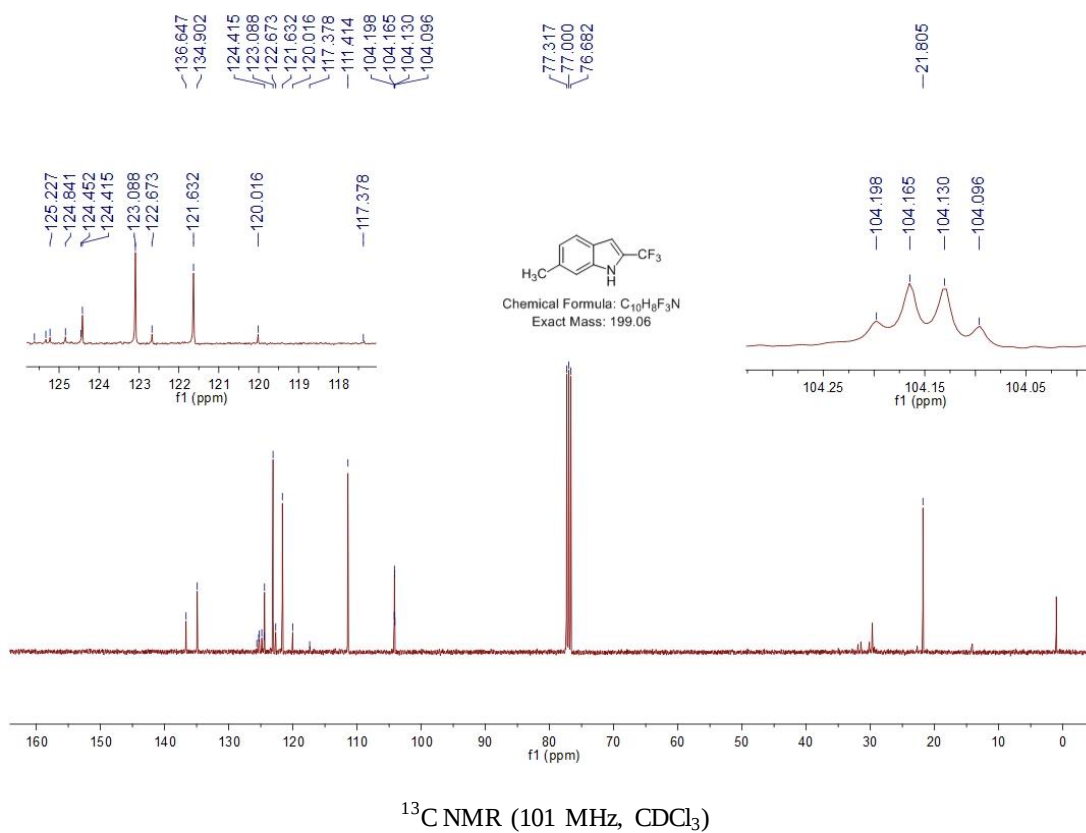
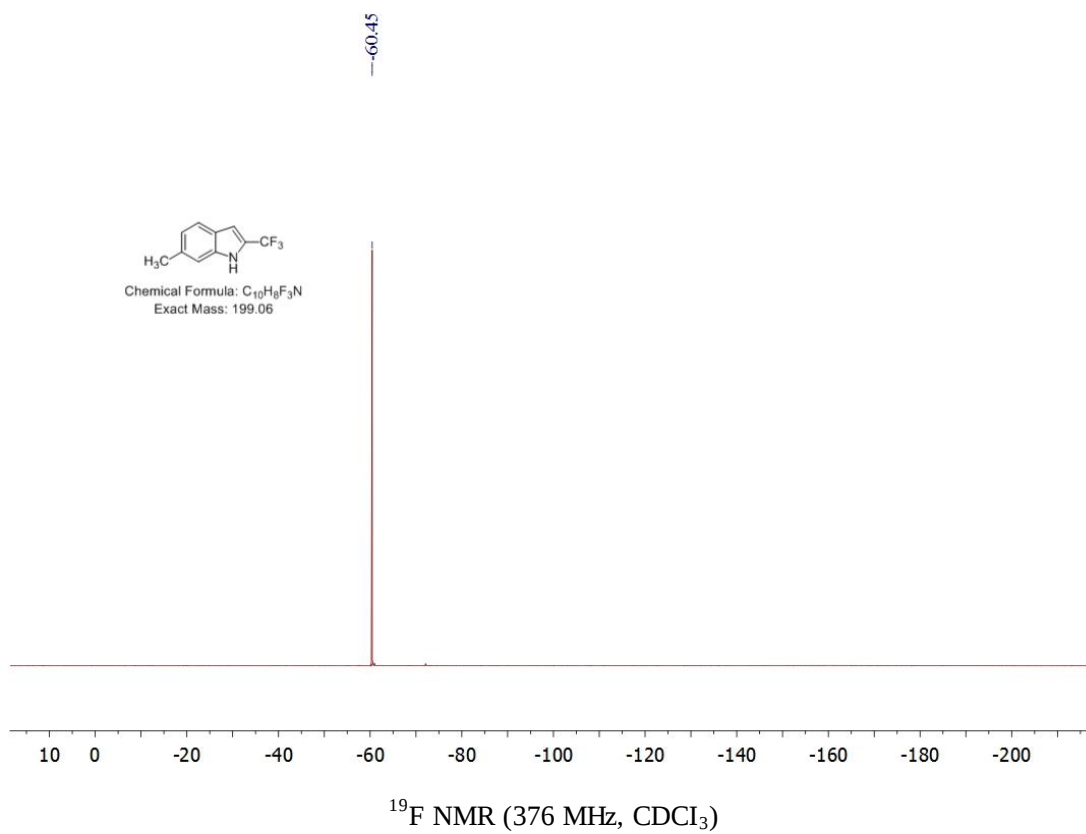


^{19}F NMR (376 MHz, $CDCl_3$)

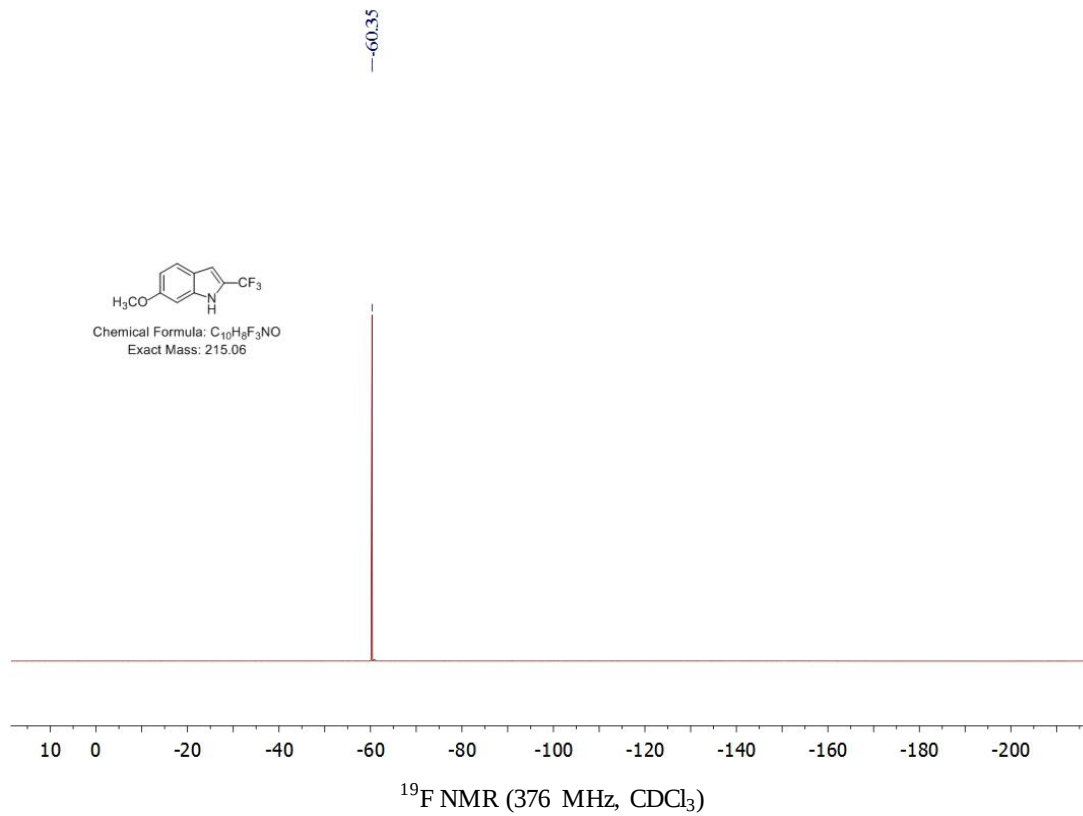
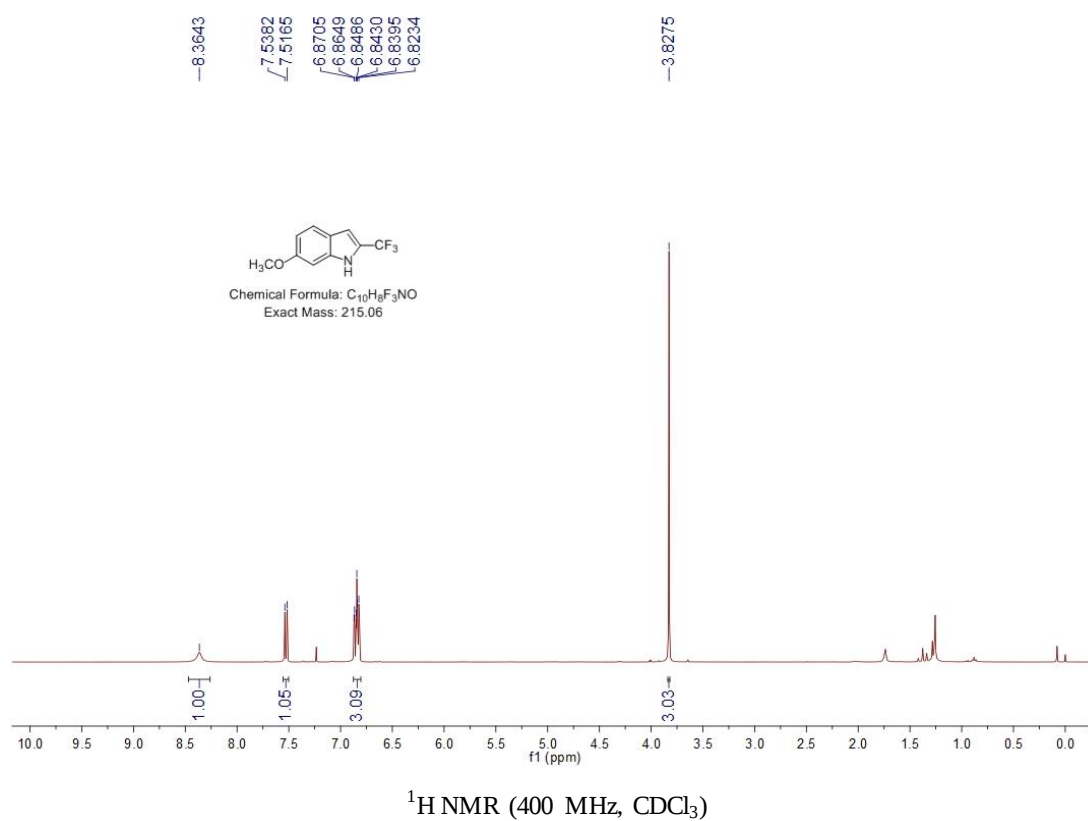


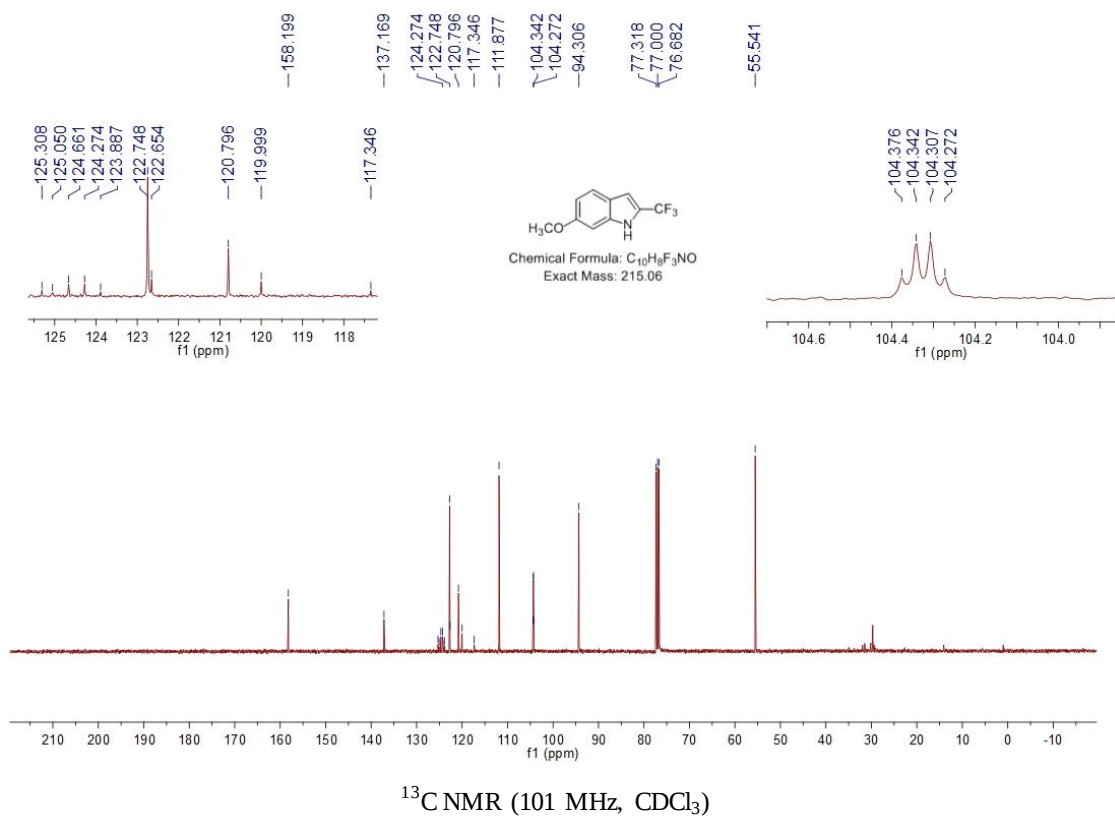
6-methyl-2-(trifluoromethyl)-1H-indole(2m)



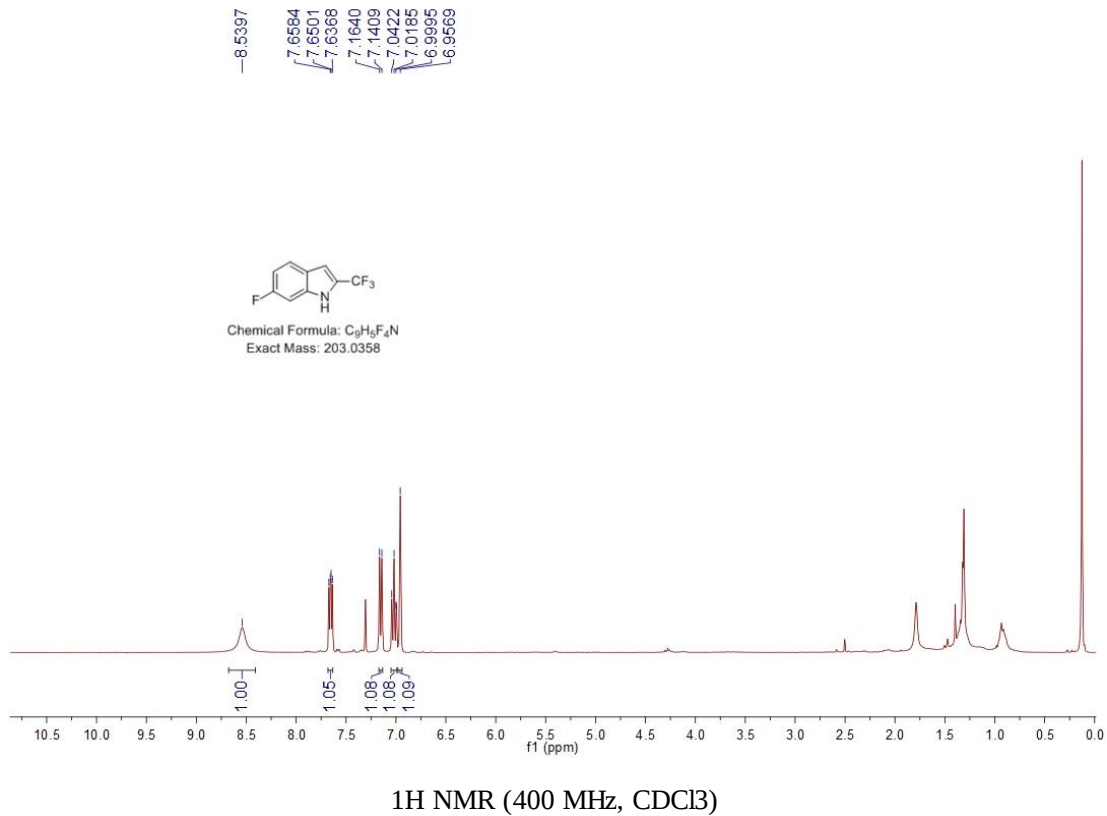


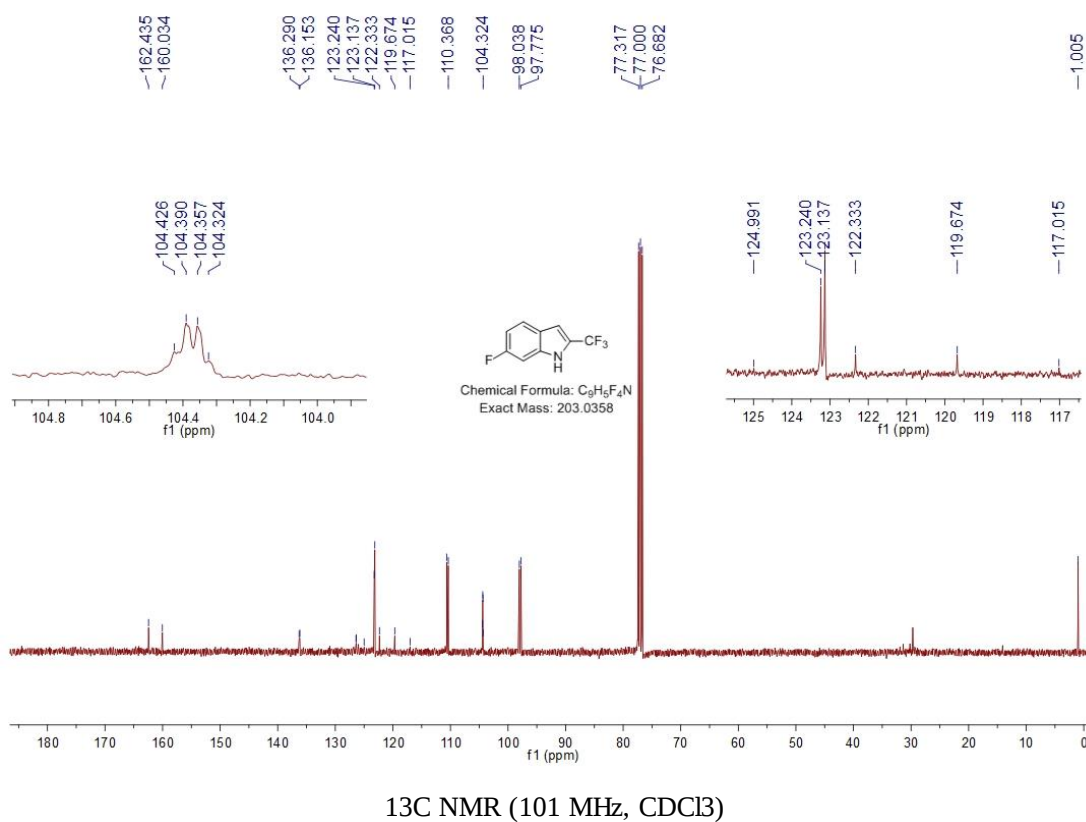
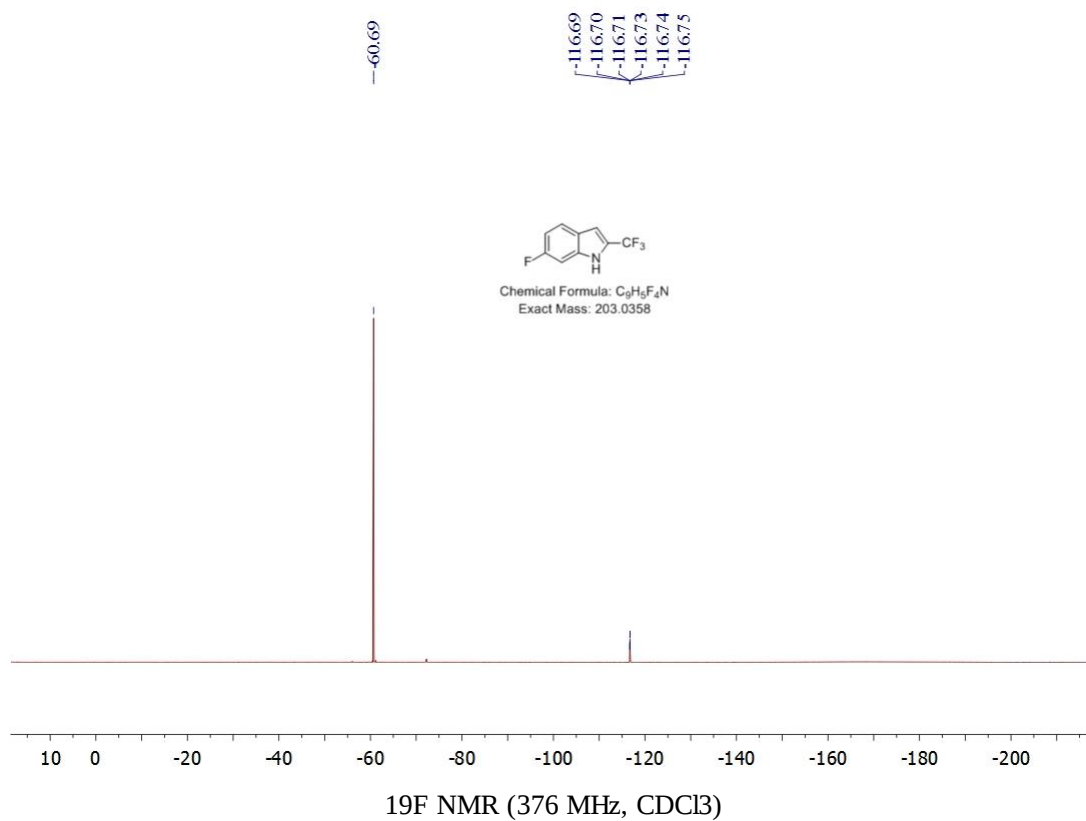
6-methoxy-2-(trifluoromethyl)-1*H*-indole(2n)





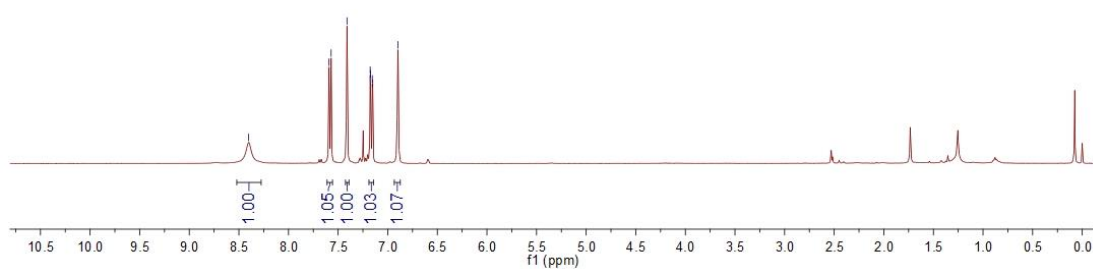
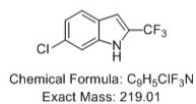
6-fluoro-2-(trifluoromethyl)-1H-indole(2o)





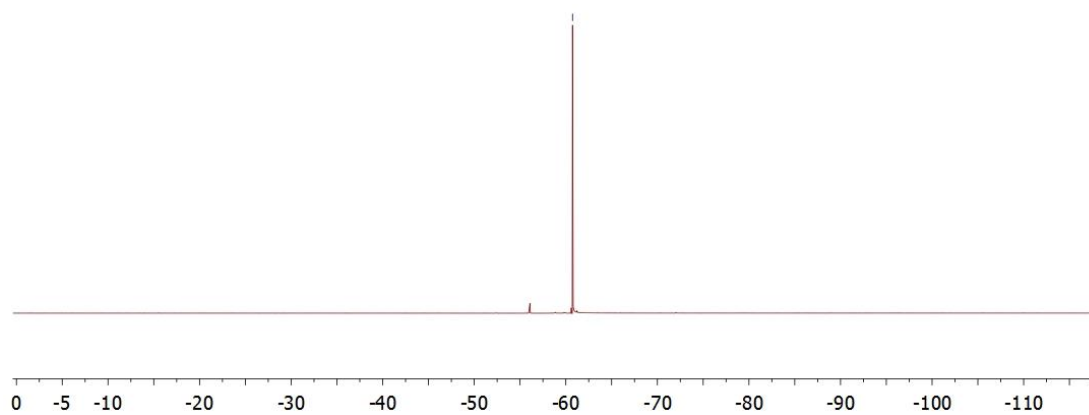
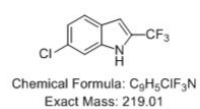
6-chloro-2-(trifluoromethyl)-1H-indole(2p)

8.4015
7.5915
7.5702
7.4105
7.1725
6.8970

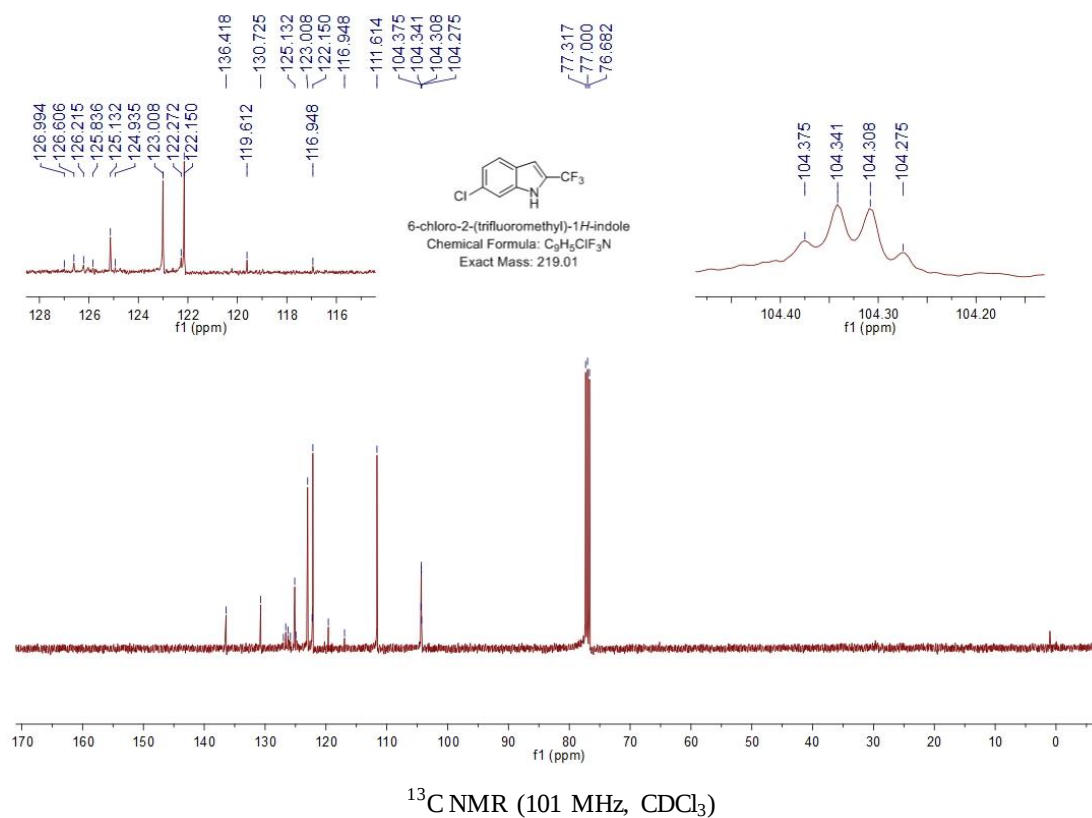


¹H NMR (400 MHz, CDCl₃)

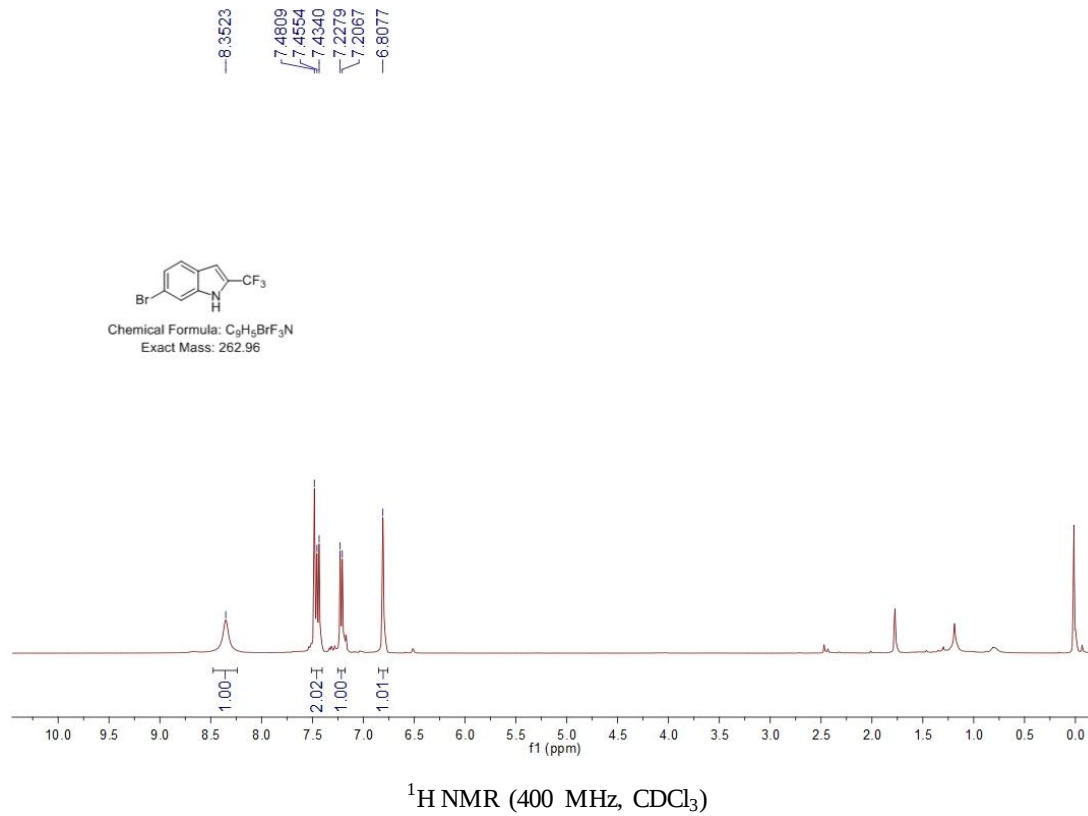
-60.73

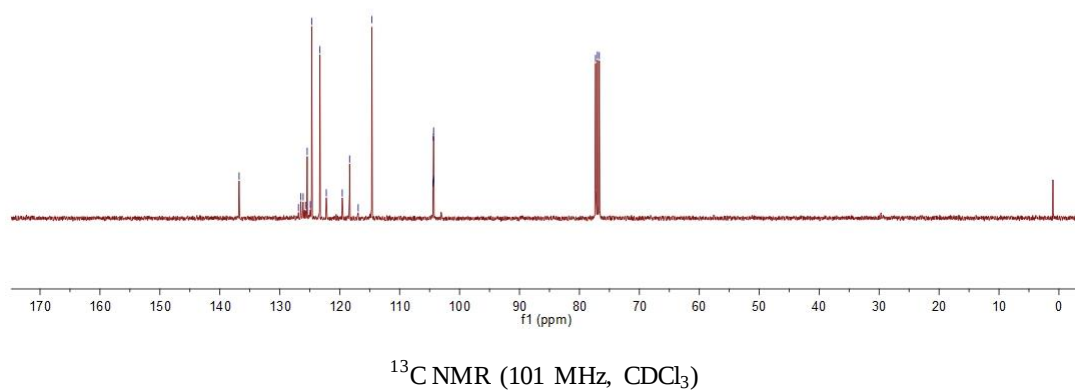
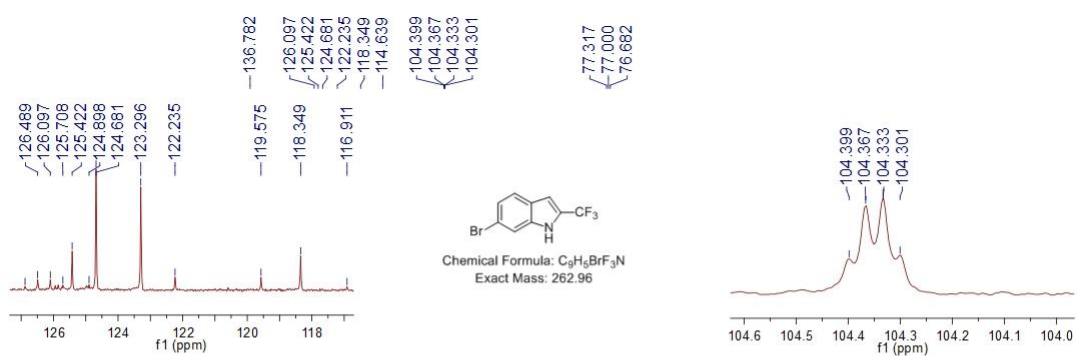
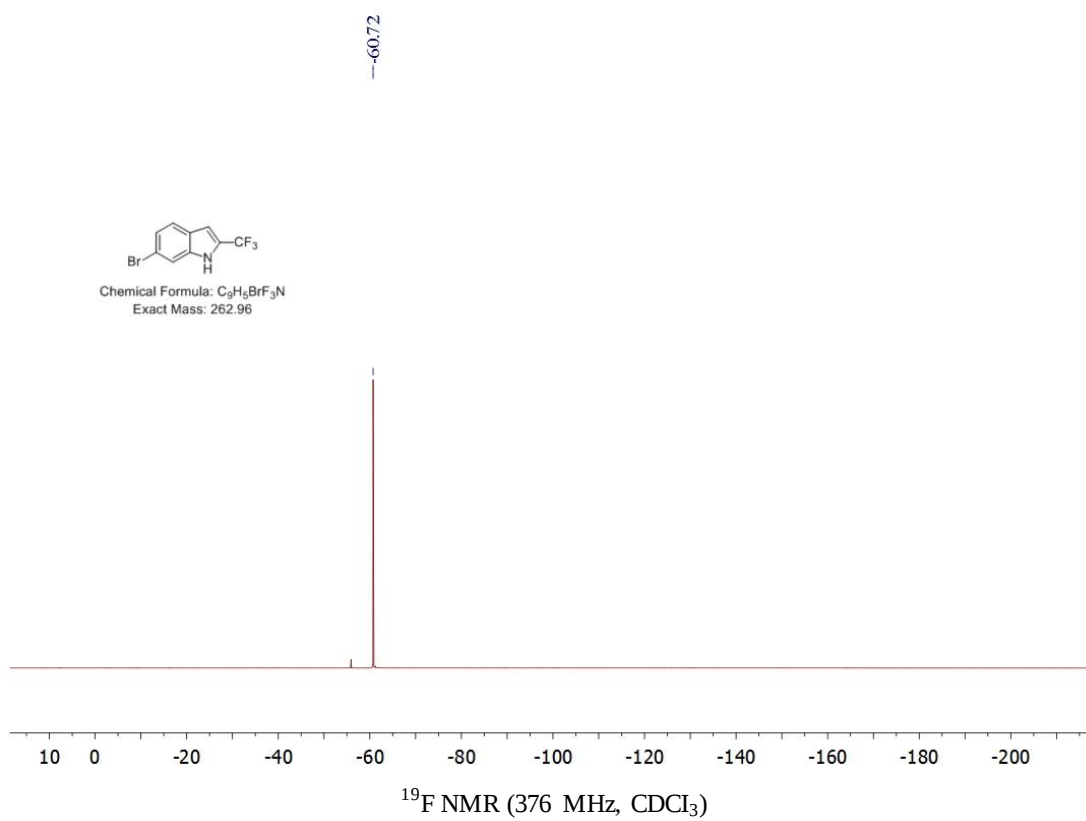


¹⁹F NMR (376 MHz, CDCl₃)



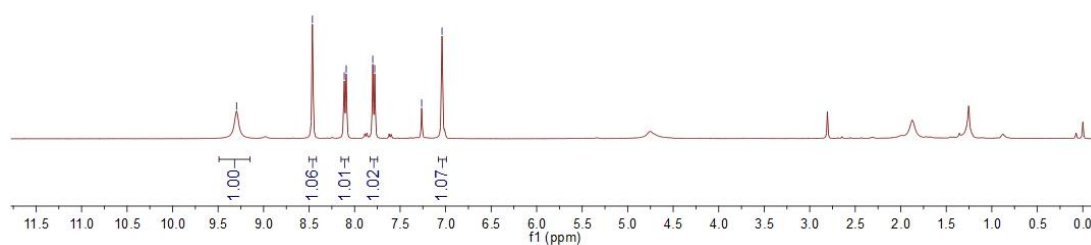
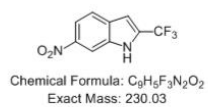
6-bromo-2-(trifluoromethyl)-1H-indole(2q)





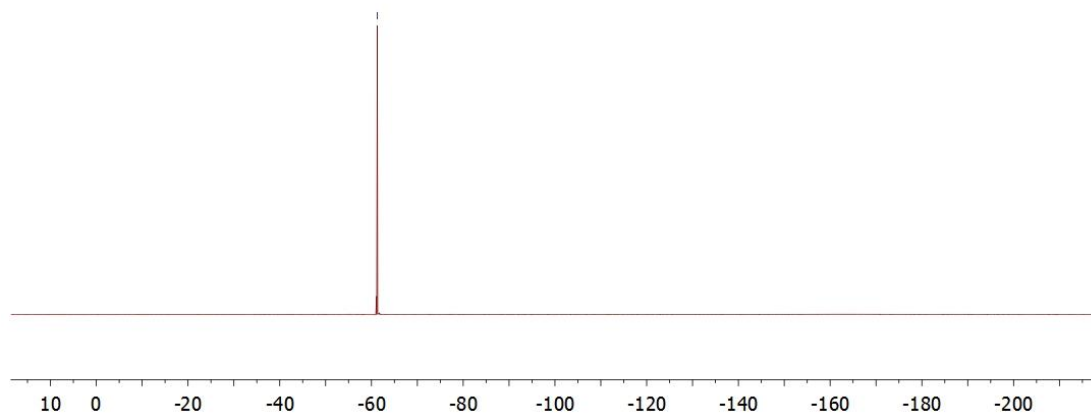
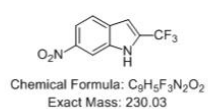
6-nitro-2-(trifluoromethyl)-1*H*-indole(2r)

9.2969
8.4633
8.0923
7.8007
7.7787
7.2638
7.0399

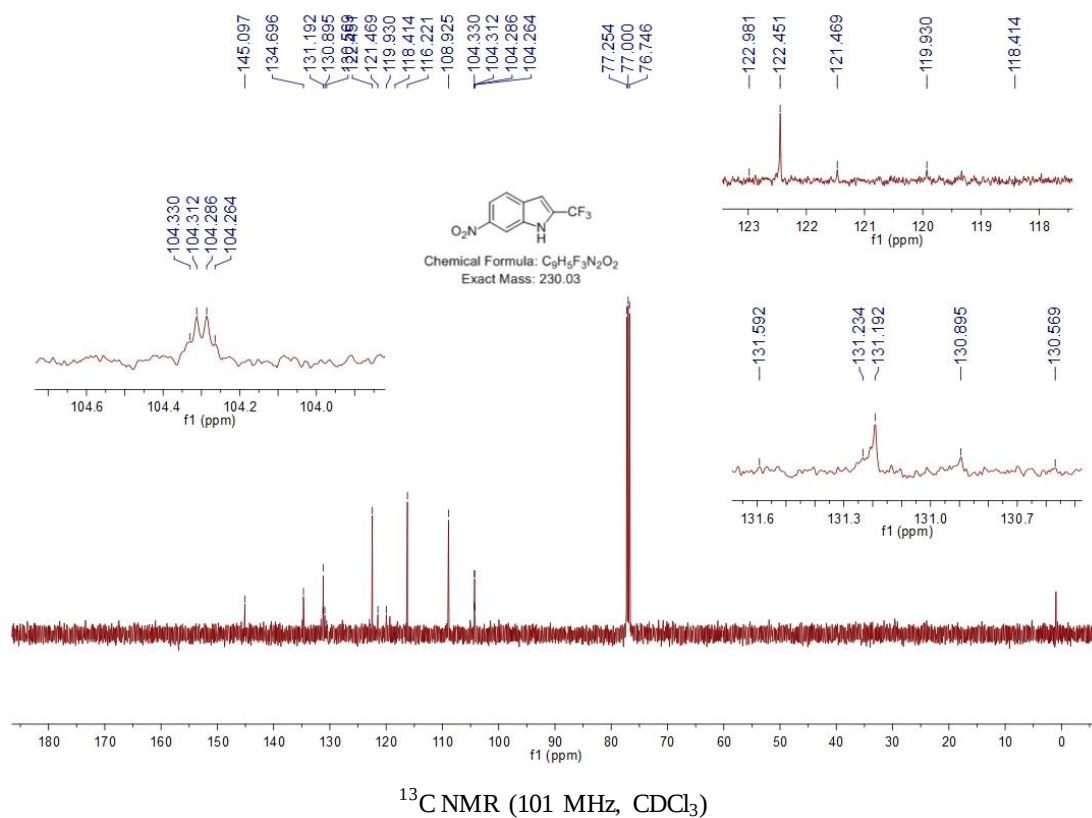


¹H NMR (400 MHz, CDCl₃)

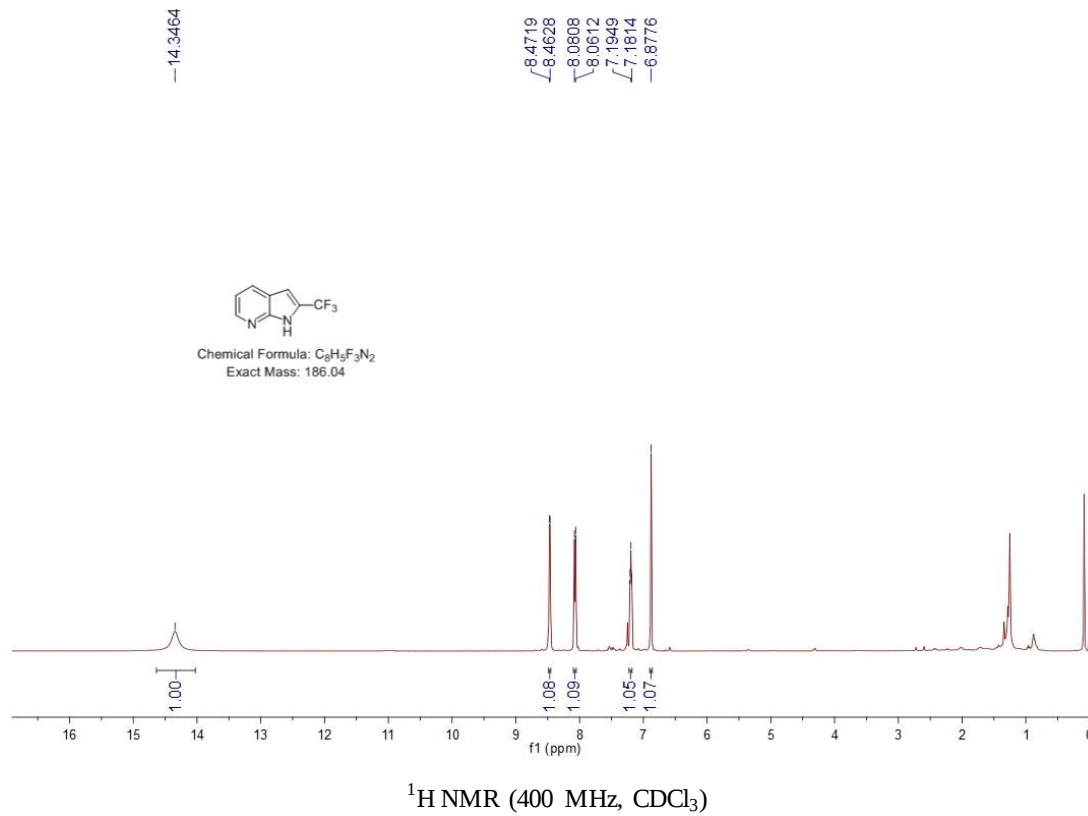
-61.27

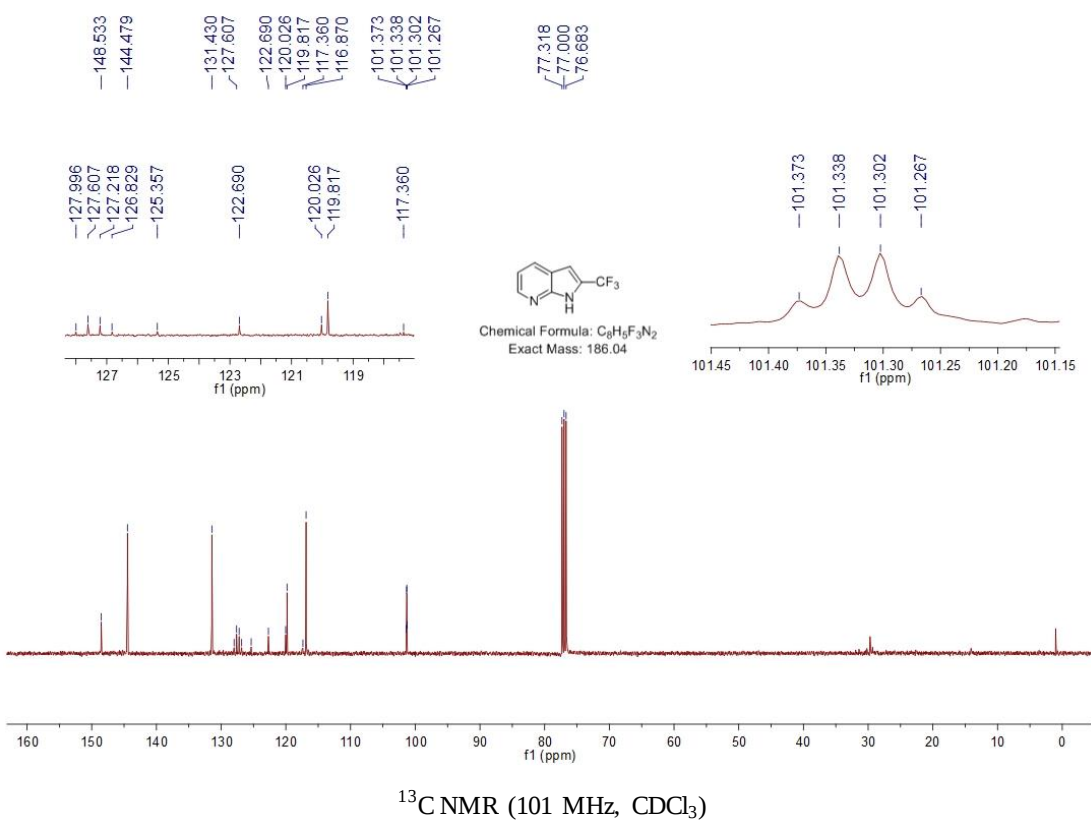
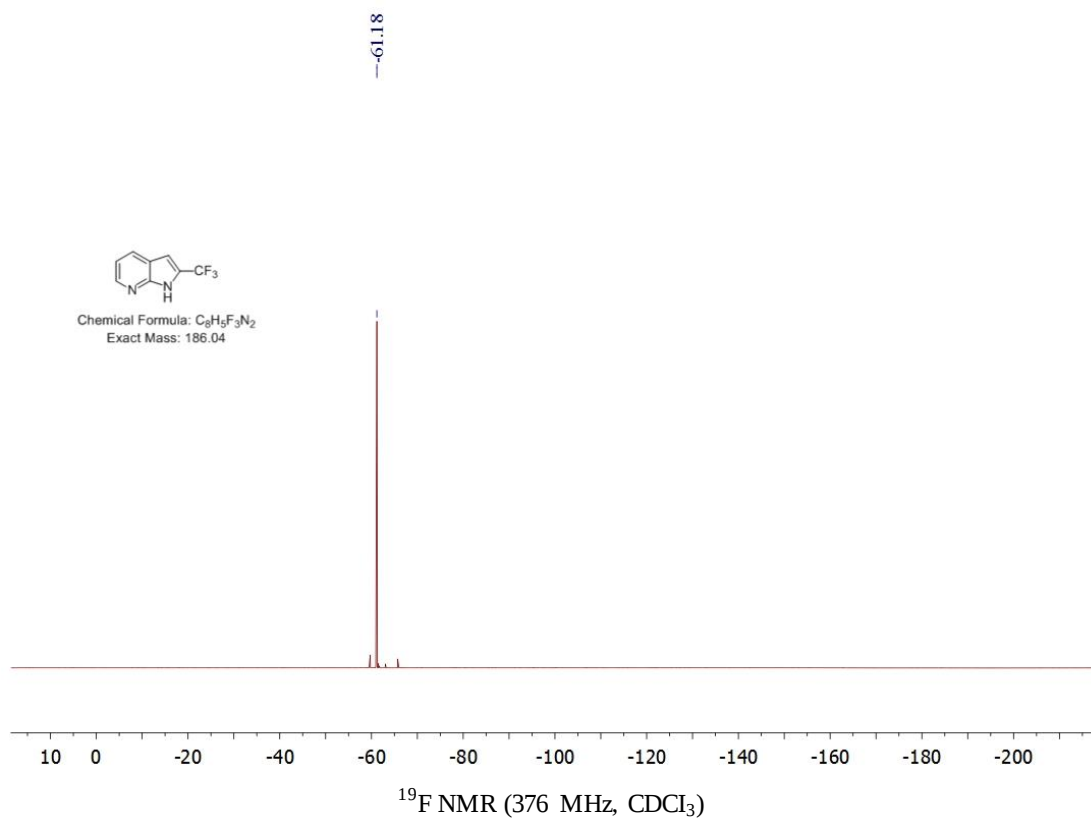


¹⁹F NMR (376 MHz, CDCl₃)

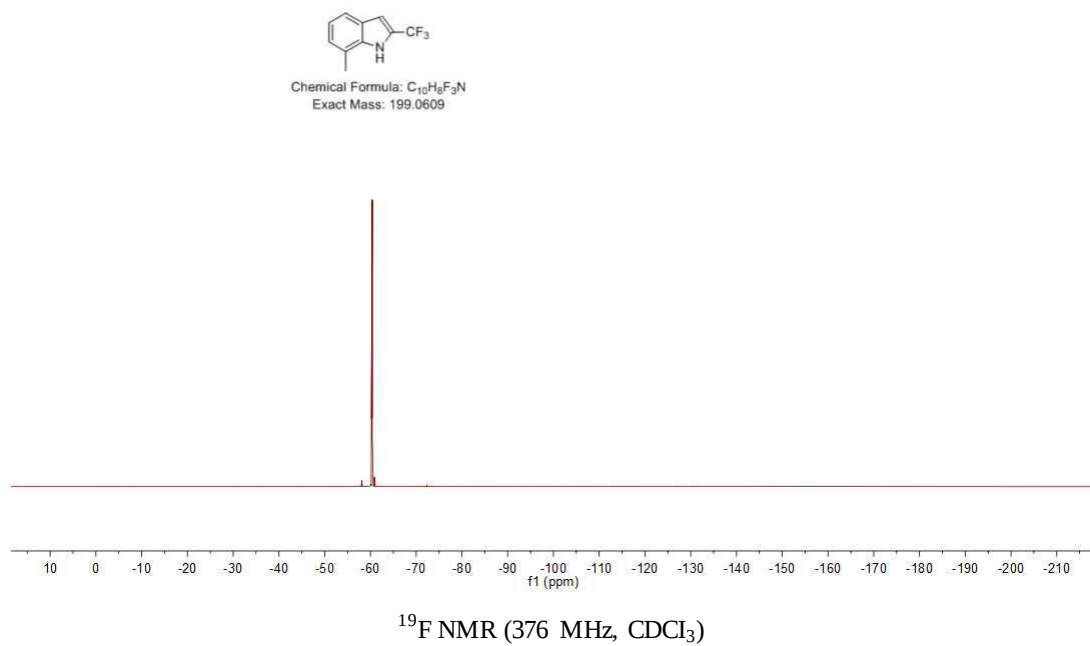
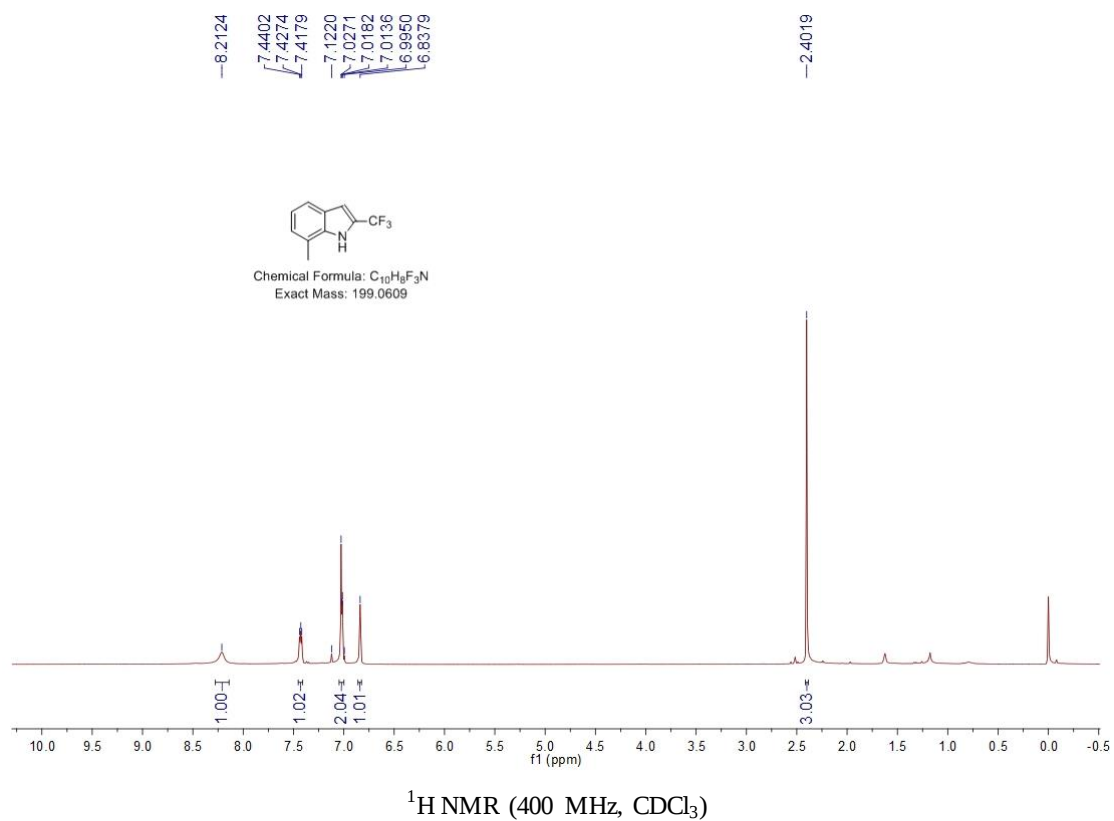


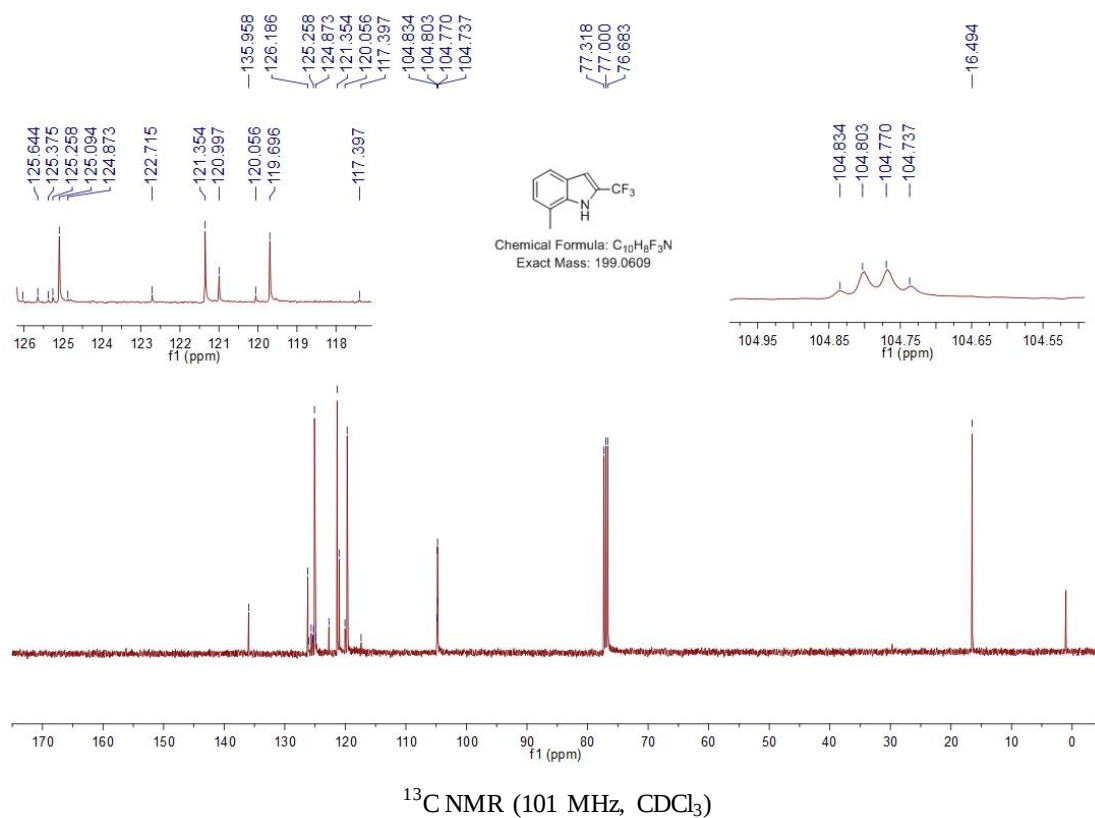
2-(trifluoromethyl)-1H-pyrrolo[2,3-b]pyridine(2s)



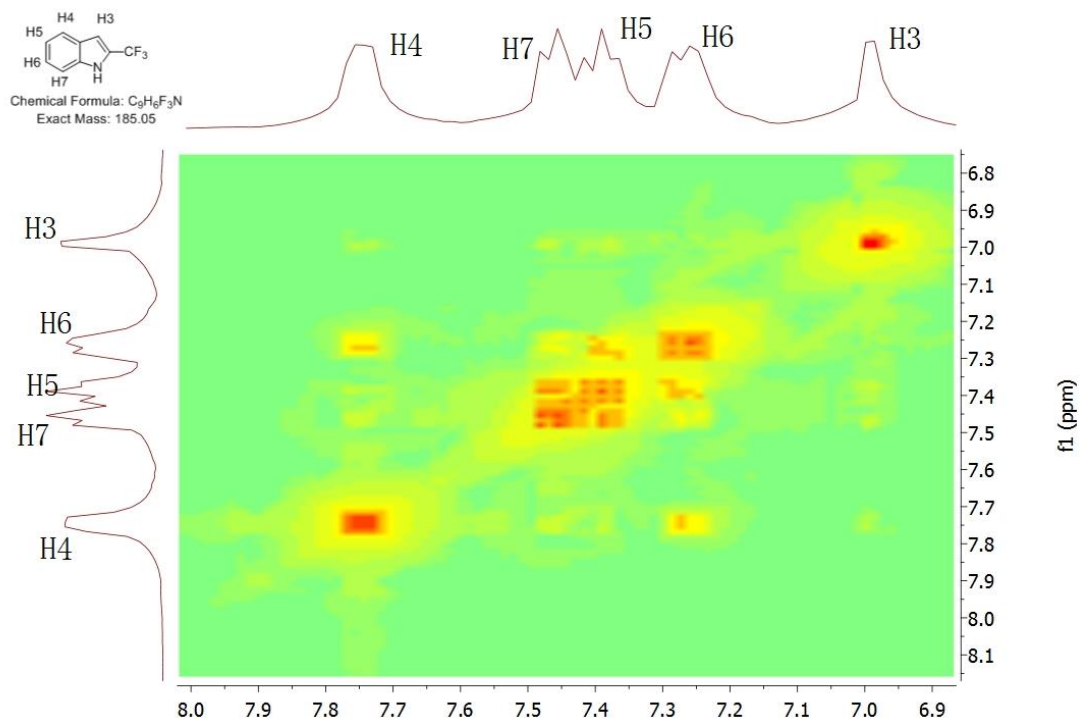


7-methyl-2-(trifluoromethyl)-1H-indole(2t)





5. 2D NMR of 2-Trifluoromethylindole



6. Referance

1. Z. Wang, F. Ge, W. Wan, H. Jiang and J. Hao, *J. Fluorine Chem.*, 2007, **128**, 1143.
2. M. S. Wiehn, E. V. Vinogradova and A. Togni, *J. Fluorine Chem.*, 2010, **131**, 951.
3. J. Pedroni and N Cramer, *Org. Lett.*, 2016, **18**, 1932.
4. T. Kino, Y. Nagase, Y. Horino and T. Yamakawa, *J. Mol. Catal. A Chem.*, 2008, **282**, 34.
5. R. A. Cormanich, C. A. Nunes and M. P. Freitas, *Quim Nova*, 2012, **35**, 1157.
6. I. Abdiaj, C. Bottecchia, J. Alcazar and T. Noël, *Synthesis*, 2017, **49**, 4978.
7. Z. Wang and Q. Ma, *J. Heterocyclic Chem.*, 2015, **52**, 1893.
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