

Base-promoted ring-closing carbonyl–allene metathesis for the synthesis of 2,4-disubstituted Pyrroles

Guolin Cheng,* Weiwei Lv, Lulu Xue

College of Materials Science & Engineering, Huaqiao University, Xiamen 361021, China

Table of Contents

1. General Information	S2
2. General Procedure for Synthesis of Preparation of <i>N</i>-propargyl β-enaminones 1a-v	S3
3. General Procedure for Synthesis of 2a-v	S3
4. Procedure for Gram-Scale Synthesis of 2a	S8
5. Procedure for Synthesis of 4 and 5	S8
6. ^1H and ^{13}C NMR Spectra	S10

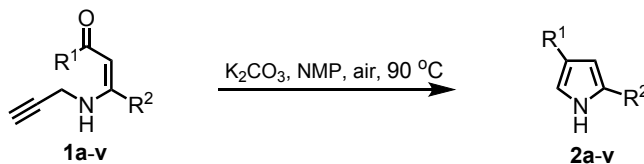
1. General Information

All reagents were used directly without further purification. Silica gel was purchased from Qing Dao Hai Yang Chemical Industry Co. ^1H and ^{13}C NMR spectra were measured on a 400 MHz Bruker spectrometer (^1H 400 MHz, ^{13}C 100 MHz) or a 500 MHz Bruker spectrometer (^1H 500 MHz, ^{13}C 125 MHz) using CDCl_3 or DMSO-D_6 as the solvent with tetramethylsilane (TMS) as the internal standard at room temperature. HRMS-ESI spectra were obtained on Agilent UPLC1290-QTOF6545. The products listed below were determined by ^1H , ^{13}C NMR. PE is petroleum ether (60–90 °C).

2. General Procedure for Synthesis of Preparation of *N*-propargyl β -enaminones 1a-v.¹

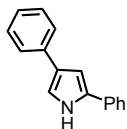
A mixture of propargylamine (1.1 g, 20 mmol), propynones (20 mmol), and CH₃OH (50 mL) was stirred at room temperature under air overnight. After propynones was exhausted completely (monitored by TLC), the solvent was evaporated and the residue was purified by chromatography (silica gel, 5% EtOAc in PE) to give **1**.

3. General Procedure for Synthesis of 2a-v



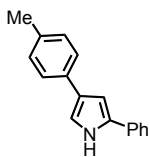
To a 15 mL Schlenk tube equipped with a magnetic stir bar were added K₂CO₃ (13.8 mg, 0.1 mmol), enaminones **1** (0.1 mmol), and NMP (1.0 mL). The solution was stirred at 90 °C under air atmosphere for 12 h. After the reaction finished, the reaction system was directly purified by column chromatography (ethyl acetate/PE = 1/50) to yield the desired products.

Spectroscopic Data for Products



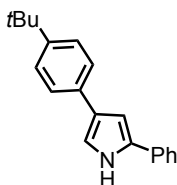
2,4-diphenyl-1*H*-pyrrole (**2a**)²

White solid (20.4 mg, 93% yield); m.p. 177-179 °C; ¹H NMR (400 MHz, DMSO) δ 11.44 (s, 1H), 7.69 (d, J = 7.8 Hz, 2H), 7.62 (d, J = 7.7 Hz, 2H), 7.35 (m, 5H), 7.15 (m, 2H), 6.96 (s, 1H); ¹³C NMR (100 MHz, DMSO) δ 136.2, 133.1, 132.7, 129.1, 129.0, 126.1, 125.5, 125.2, 124.9, 123.9, 117.0, 103.6.



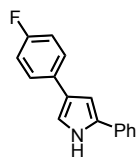
2-phenyl-4-(*p*-tolyl)-1*H*-pyrrole (**2b**)²

White solid (21.0 mg, 90% yield); m.p. 194-196 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.40 (s, 1H), 7.48 (dd, J = 17.2, 7.9 Hz, 4H), 7.38 (t, J = 7.6 Hz, 2H), 7.22 (d, J = 7.4 Hz, 1H), 7.17 (d, J = 7.8 Hz, 2H), 7.10 (s, 1H), 6.80 (s, 1H), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 135.2, 132.9, 132.5, 129.3, 128.9, 126.6, 126.4, 125.1, 123.8, 115.2, 103.9, 21.0.



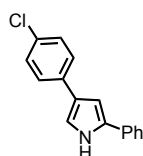
4-(4-(*tert*-butyl)phenyl)-2-phenyl-1*H*-pyrrole (**2c**)

White solid (17.6 mg, 64% yield); m.p. 165-167 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.35 (s, 1H), 7.53 – 7.45 (m, 4H), 7.41 – 7.33 (m, 4H), 7.21 (dd, *J* = 10.3, 3.7 Hz, 1H), 7.09 – 7.01 (m, 1H), 6.80 (d, *J* = 1.6 Hz, 1H), 1.34 (d, *J* = 2.7 Hz, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 148.6, 132.8, 132.7, 132.5, 128.9, 126.5, 126.3, 125.5, 124.9, 123.7, 115.4, 104.0, 34.4, 31.3; HRMS (ESI) calcd for C₂₀H₂₂N [M+H]⁺ 276.1747, found: 276.1754.



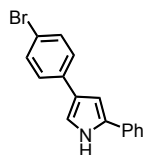
4-(4-fluorophenyl)-2-phenyl-1*H*-pyrrole (2d)²

White solid (22.3 mg, 94% yield); m.p. 191-194 °C; ¹H NMR (400 MHz, DMSO) δ 11.44 (s, 1H), 7.69 (d, *J* = 7.9 Hz, 2H), 7.64 (dd, *J* = 7.5, 5.7 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.32 (s, 1H), 7.22 – 7.12 (m, 3H), 6.93 (s, 1H); ¹³C NMR (100 MHz, DMSO) δ 160.7 (d, *J* = 241.1 Hz), 133.1, 132.8, 132.8, 129.2, 126.5 (d, *J* = 7.6 Hz), 126.2, 124.3, 123.9, 117.0, 115.7 (d, *J* = 21.2 Hz), 103.7; ¹⁹F NMR (376 MHz, DMSO) δ -118.2.



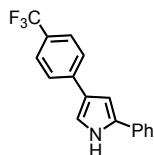
4-(4-chlorophenyl)-2-phenyl-1*H*-pyrrole (2e)²

White solid (21.0 mg, 83% yield); m.p. 206-208 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.44 (s, 1H), 7.48 (t, *J* = 7.4 Hz, 4H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.24 (t, *J* = 7.3 Hz, 1H), 7.10 (s, 1H), 6.77 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 134.0, 133.3, 132.2, 131.2, 128.9, 128.7, 126.6, 126.3, 125.5, 123.9, 115.6, 103.8.



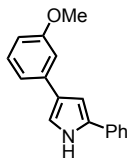
4-(4-bromophenyl)-2-phenyl-1*H*-pyrrole (2f)³

White solid (23.3 mg, 78% yield); m.p. 188-191 °C; ¹H NMR (400 MHz, DMSO) δ 11.51 (s, 1H), 7.68 (d, *J* = 7.9 Hz, 2H), 7.60 – 7.55 (m, 2H), 7.49 (d, *J* = 7.4 Hz, 2H), 7.42 – 7.35 (m, 3H), 7.18 (t, *J* = 7.3 Hz, 1H), 6.97 (s, 1H); ¹³C NMR (100 MHz, DMSO) δ 135.5, 132.9, 131.8, 129.1, 126.8, 126.2, 123.9, 123.9, 118.0, 117.5, 103.6.



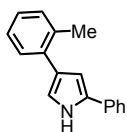
2-phenyl-4-(4-(trifluoromethyl)phenyl)-1*H*-pyrrole (2g)

White solid (20.1 mg, 70% yield); m.p. 201-208 °C; ¹H NMR (400 MHz, DMSO) δ 11.59 (s, 1H), 7.80 (d, *J* = 7.1 Hz, 2H), 7.65 (dd, *J* = 20.9, 7.2 Hz, 4H), 7.50 (s, 1H), 7.36 (t, *J* = 6.7 Hz, 2H), 7.18 (t, *J* = 6.6 Hz, 1H), 7.04 (s, 1H); ¹³C NMR (100 MHz, DMSO) δ 140.4, 133.3, 132.8, 129.2, 126.4, 125.9 (q, *J* = 4.4 Hz), 125.5, 125.1, 124.8 (q, *J* = 20.8 Hz), 124.0, 123.8, 118.6, 103.9; ¹⁹F NMR (376 MHz, DMSO) δ -60.49; HRMS (ESI) calcd for C₁₇H₁₃F₃N [M+H]⁺ 288.0995, found: 288.0996.



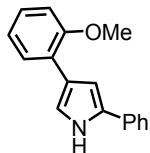
4-(4-methoxyphenyl)-2-phenyl-1H-pyrrole (2h)

White solid (19.9 mg, 80% yield); m.p. 117-118 °C ¹H NMR (400 MHz, CDCl₃) δ 8.44 (s, 1H), 7.50 (d, *J* = 7.8 Hz, 2H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.30 – 7.20 (m, 2H), 7.17 (d, *J* = 7.6 Hz, 1H), 7.11 (s, 2H), 6.84 – 6.72 (m, 2H), 3.85 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.9, 136.9, 133.0, 132.4, 129.6, 128.9, 126.5, 123.8, 117.8, 115.7, 111.1, 110.9, 104.0, 55.2; HRMS (ESI) calcd for C₁₇H₁₆NO [M+H]⁺ 250.1226, found: 250.1232.



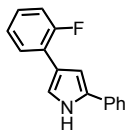
2-phenyl-4-(*o*-tolyl)-1H-pyrrole (2i)²

White solid (18.0 mg, 77% yield); m.p. 110-113 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.39 (s, 1H), 7.52 – 7.45 (m, 2H), 7.44 – 7.32 (m, 3H), 7.27 – 7.13 (m, 4H), 6.90 (dd, *J* = 2.4, 1.6 Hz, 1H), 6.68 (dd, *J* = 2.5, 1.6 Hz, 1H), 2.46 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 135.5, 135.2, 132.5, 131.8, 130.5, 129.1, 128.9, 126.3, 126.1, 126.0, 125.8, 123.7, 117.7, 106.9, 21.3.



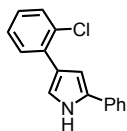
4-(2-methoxyphenyl)-2-phenyl-1H-pyrrole (2j)

Colorless oil (17.7 mg, 71% yield); ¹H NMR (400 MHz, CDCl₃) δ 8.44 (s, 1H), 7.59 (d, *J* = 7.6 Hz, 1H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.35 (t, *J* = 7.5 Hz, 3H), 7.24 – 7.14 (m, 2H), 7.02 – 6.89 (m, 3H), 3.89 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 156.1, 132.6, 131.8, 128.8, 127.8, 126.5, 126.2, 124.2, 123.8, 122.0, 120.7, 119.0, 111.1, 105.5, 55.3; HRMS (ESI) calcd for C₁₇H₁₆NO [M+H]⁺ 250.1226, found: 250.1233.



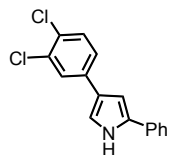
4-(2-fluorophenyl)-2-phenyl-1H-pyrrole (2k)

White solid (17.8 mg, 75% yield); m.p. 104-106 °C; ¹H NMR (400 MHz, DMSO) δ 11.58 (s, 1H), 7.74 – 7.68 (m, 3H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.32 (s, 1H), 7.23 – 7.19 (m, 4H), 7.01 (s, 1H); ¹³C NMR (100 MHz, DMSO) δ 159.3 (d, *J* = 245.3 Hz), 132.9, 132.4, 129.2, 128.1 (d, *J* = 4.9 Hz), 126.9 (d, *J* = 8.4 Hz), 126.4, 125.0 (d, *J* = 3.2 Hz), 124.0, 123.6 (d, *J* = 12.9 Hz), 119.5 (d, *J* = 9.7 Hz), 118.7, 116.3 (d, *J* = 22.3 Hz), 104.9 (d, *J* = 3.1 Hz); ¹⁹F NMR (376 MHz, CDCl₃) δ -115.08; HRMS (ESI) calcd for C₁₆H₁₃FN [M+H]⁺ 238.1027, found: 238.1035.



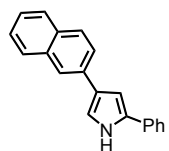
4-(2-chlorophenyl)-2-phenyl-1H-pyrrole (2l)

White solid (15.2 mg, 60% yield); m.p. 108-110 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.52 (s, 1H), 7.52 (t, *J* = 7.6 Hz, 3H), 7.46 – 7.35 (m, 3H), 7.30 – 7.20 (m, 3H), 7.15 (t, *J* = 7.5 Hz, 1H), 6.84 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 134.3, 132.3, 132.0, 131.7, 130.3, 130.1, 128.9, 126.9, 126.7, 126.1, 123.9, 123.3, 118.8, 106.7; HRMS (ESI) calcd for C₁₆H₁₃ClN [M+H]⁺ 254.0731, found: 254.0734.



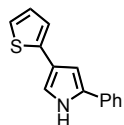
4-(3,4-dichlorophenyl)-2-phenyl-1H-pyrrole (2m)

White solid (20.1 mg, 70% yield); m.p. 123-124 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.50 (s, 1H), 7.63 (s, 1H), 7.50 (d, *J* = 8.0 Hz, 2H), 7.44 – 7.34 (m, 4H), 7.25 (t, *J* = 6.8 Hz, 1H), 7.11 (s, 1H), 6.75 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 135.7, 133.5, 132.5, 132.0, 130.4, 129.0, 129.0, 126.8, 126.7, 124.3, 124.3, 123.9, 115.9, 103.7; HRMS (ESI) calcd for C₁₆H₁₂Cl₂N [M+H]⁺ 288.0341, found: 288.0349.



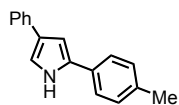
4-(naphthalen-2-yl)-2-phenyl-1H-pyrrole (2n)

White solid (22.1 mg, 82% yield); m.p. 229-230 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.49 (s, 1H), 7.99 (s, 1H), 7.82 (t, *J* = 8.9 Hz, 3H), 7.76 – 7.69 (m, 1H), 7.55 (d, *J* = 7.4 Hz, 2H), 7.47-7.38 (m, 4H), 7.26 (d, *J* = 5.2 Hz, 2H), 6.96 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 134.0, 133.3, 132.9, 132.4, 132.0, 128.9, 128.1, 127.6, 127.6, 126.6, 126.5, 126.0, 124.9, 124.5, 123.9, 122.6, 115.9, 104.1; HRMS (ESI) calcd for C₂₀H₁₆N [M+H]⁺ 270.1277, found: 270.1285.



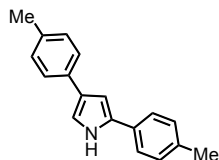
2-phenyl-4-(thiophen-2-yl)-1H-pyrrole (2o)

White solid (15.3 mg, 68% yield); m.p. 155-156 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.35 (s, 1H), 7.47 (d, *J* = 7.6 Hz, 2H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.23 (dd, *J* = 8.3, 6.4 Hz, 1H), 7.10 (dd, *J* = 9.5, 4.0 Hz, 2H), 7.01 (dd, *J* = 8.7, 4.8 Hz, 2H), 6.70 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 139.0, 132.9, 132.1, 128.9, 127.4, 126.6, 123.9, 121.9, 121.3, 120.5, 115.5, 104.5; HRMS (ESI) calcd for C₁₄H₁₂NS [M+H]⁺ 226.0685, found: 226.0688.



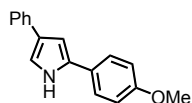
4-phenyl-2-(p-tolyl)-1H-pyrrole (2p)²

White solid (20.0 mg, 86% yield); m.p. 205-207 °C; ¹H NMR (400 MHz, DMSO) δ 11.35 (s, 1H), 7.58 (t, *J* = 8.3 Hz, 4H), 7.36 – 7.27 (m, 3H), 7.18 (d, *J* = 7.8 Hz, 2H), 7.12 (t, *J* = 7.1 Hz, 1H), 6.88 (s, 1H), 2.30 (s, 3H); ¹³C NMR (100 MHz, DMSO) δ 136.2, 135.2, 132.8, 130.4, 129.7, 128.9, 125.4, 125.0, 124.8, 123.8, 116.6, 103.0, 21.1.



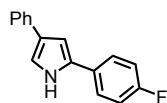
2,4-di-*p*-tolyl-1*H*-pyrrole(2q)³

White solid (15.6 mg, 63% yield); m.p. 203-204 °C ¹H NMR (400 MHz, DMSO) δ 11.29 (s, 1H), 7.56 (d, *J* = 7.7 Hz, 2H), 7.48 (d, *J* = 7.6 Hz, 2H), 7.23 (s, 1H), 7.17 (d, *J* = 7.8 Hz, 2H), 7.12 (d, *J* = 7.8 Hz, 2H), 6.84 (s, 1H), 2.29 (s, 3H), 2.28 (s, 3H); ¹³C NMR (100 MHz, DMSO) δ 135.2, 134.3, 133.4, 132.6, 130.5, 129.7, 129.5, 125.0, 124.7, 123.8, 116.1, 102.9, 21.1, 21.1.



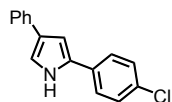
2-(4-methoxyphenyl)-4-phenyl-1*H*-pyrrole (2r)²

White solid (17.9 mg, 72% yield); m.p. 212-213 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.34 (s, 1H), 7.60 – 7.54 (m, 2H), 7.47 – 7.43 (m, 2H), 7.35 (t, *J* = 7.7 Hz, 2H), 7.19 (t, *J* = 7.4 Hz, 1H), 7.13 – 7.08 (m, 1H), 6.98 – 6.90 (m, 2H), 6.74 – 6.70 (m, 1H), 3.84 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 158.4, 135.6, 133.1, 128.6, 126.5, 125.6, 125.5, 125.3, 125.1, 114.8, 114.4, 103.0, 55.3.



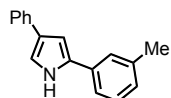
2-(4-fluorophenyl)-4-phenyl-1*H*-pyrrole (2s)²

White solid (18.5 mg, 78% yield); m.p. 174-176 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.33 (s, 1H), 7.55 (d, *J* = 7.1 Hz, 2H), 7.45 (s, 2H), 7.35 (t, *J* = 7.3 Hz, 2H), 7.20 (t, *J* = 7.2 Hz, 1H), 7.14 – 7.01 (m, 3H), 6.74 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 161.6 (d, *J* = 245.8 Hz), 135.4, 132.3, 128.9 (d, *J* = 3.3 Hz), 128.7, 126.6, 125.8, 125.5 (d, *J* = 7.9 Hz), 125.2, 115.9 (d, *J* = 21.8 Hz), 115.5, 103.9; ¹⁹F NMR (376 MHz, CDCl₃) δ -115.7.



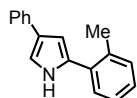
2-(4-chlorophenyl)-4-phenyl-1*H*-pyrrole (2t)²

White solid (17.2 mg, 68% yield); m.p. 186-187 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.39 (s, 1H), 7.55 (d, *J* = 7.3 Hz, 2H), 7.43 (d, *J* = 8.1 Hz, 2H), 7.35 (d, *J* = 7.8 Hz, 4H), 7.21 (t, *J* = 7.3 Hz, 1H), 7.14 (s, 1H), 6.80 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 135.2, 132.0, 131.9, 131.0, 129.1, 128.6, 126.8, 125.8, 125.2, 125.0, 115.8, 104.4.



4-phenyl-2-(*m*-tolyl)-1*H*-pyrrole (2u)⁴

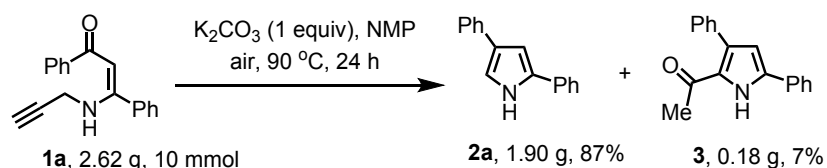
White solid (19.1 mg, 82% yield); m.p. 144-146 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.41 (s, 1H), 7.65 – 7.51 (m, 2H), 7.37 – 7.24 (m, 5H), 7.19 (t, *J* = 7.4 Hz, 1H), 7.15 – 7.09 (m, 1H), 7.05 (d, *J* = 7.1 Hz, 1H), 6.81 (dd, *J* = 2.5, 1.7 Hz, 1H), 2.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 138.5, 135.5, 133.2, 132.4, 128.8, 128.6, 127.3, 126.5, 125.6, 125.1, 124.6, 120.9, 115.3, 103.8, 21.5.



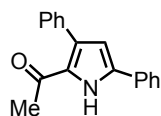
4-phenyl-2-(*o*-tolyl)-1H-pyrrole (**2v**)⁴

White solid (15.1 mg, 65% yield); m.p. 81-84 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.24 (s, 1H), 7.57 (d, *J* = 7.5 Hz, 2H), 7.36 (dt, *J* = 12.7, 4.7 Hz, 3H), 7.30 – 7.11 (m, 5H), 6.64 (s, 1H), 2.50 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 135.6, 135.1, 132.5, 132.3, 131.0, 128.6, 127.9, 127.0, 126.0, 125.8, 125.6, 125.1, 114.6, 106.8, 21.2.

4. Procedure for Gram-Scale Synthesis of **2a**



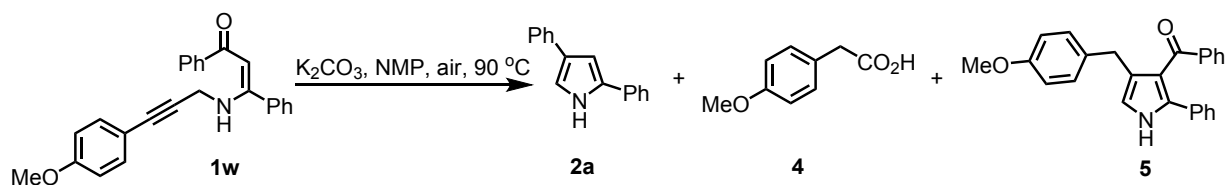
K₂CO₃ (1.38 g, 10 mmol) was added to a solution of enaminone **1a** (2.62 g, 10 mmol) in NMP (50 mL). The solution was stirred at 90 °C under air atmosphere for 24 h. After the reaction finished, the reaction system was quenched by water (200 mL), and extracted with Et₂O (5 x 20 mL). The combined Et₂O extracts were dried over Na₂SO₄ and concentrated. Then solvent was evaporated and the residue was purified by chromatography (ethyl acetate/PE = 1/50) to yield **2a** (1.9 g, 87%) and **3** (0.18 g, 7% yield).



1-(3,5-diphenyl-1H-pyrrol-2-yl)ethan-1-one (**3**)

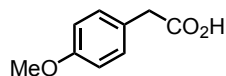
White solid; m.p. 90-91 °C; ¹H NMR (500 MHz, CDCl₃) δ 9.72 (s, 1H), 7.63 (d, *J* = 7.4 Hz, 2H), 7.48 – 7.30 (m, 8H), 6.56 (d, *J* = 3.0 Hz, 1H), 2.09 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 188.6, 136.2, 136.1, 134.3, 130.7, 129.7, 129.3, 129.1, 128.2, 128.2, 127.7, 125.0, 110.8, 27.5; HRMS (ESI) calcd for C₁₈H₁₆NO [M+H]⁺ 262.1226, found: 262.1227.

5. Procedure for Synthesis of **4** and **5**



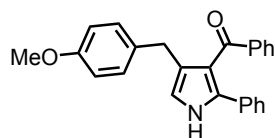
K₂CO₃ (27.6 mg, 0.2 mmol) and enaminone **1w** (73.4 mg, 0.2 mmol) were added in NMP (2.0 mL). The solution was stirred at 90 °C under air atmosphere for 24 h. After the reaction finished, the reaction system was quenched by

1M HCl (10 mL), and extracted with Et₂O (5 x 5 mL). The combined Et₂O extracts were dried over Na₂SO₄ and concentrated. Then solvent was evaporated and the residue was purified by chromatography (ethyl acetate/AcOH PE = 1/1/50 to 1/1/10) to yield **2a** (13.4 mg, 31%), **4** (8.0 mg, 24% yield), and **5** (36.7 mg, 50% yield).



2-(4-methoxyphenyl)acetic acid (**4**)

¹H NMR (500 MHz, CDCl₃) δ 11.50 (s, 1H), 7.18 (d, *J* = 8.4 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 3.77 (s, 3H), 3.56 (s, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 178.5, 158.7, 130.3, 125.2, 114.0, 55.2, 40.1.



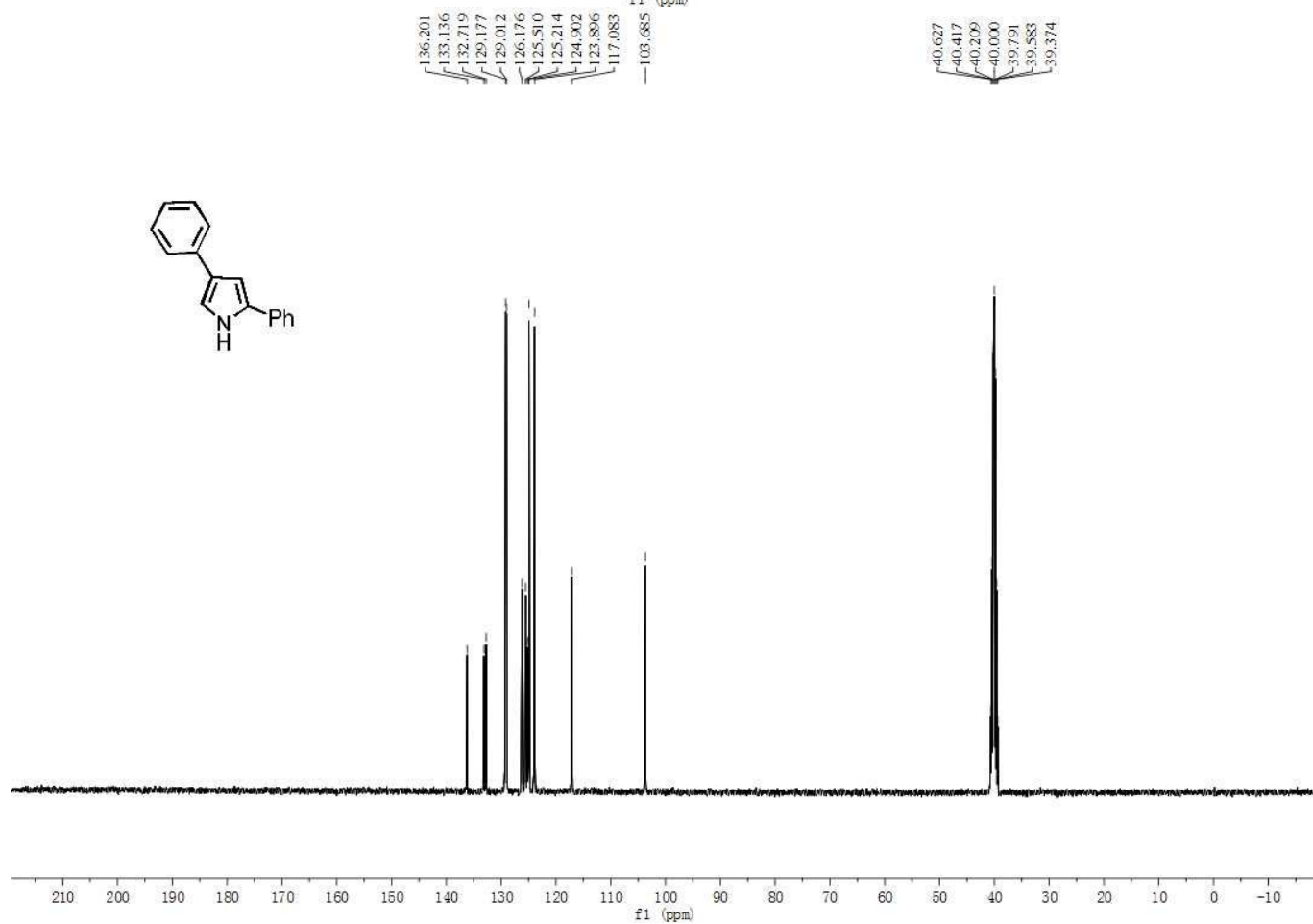
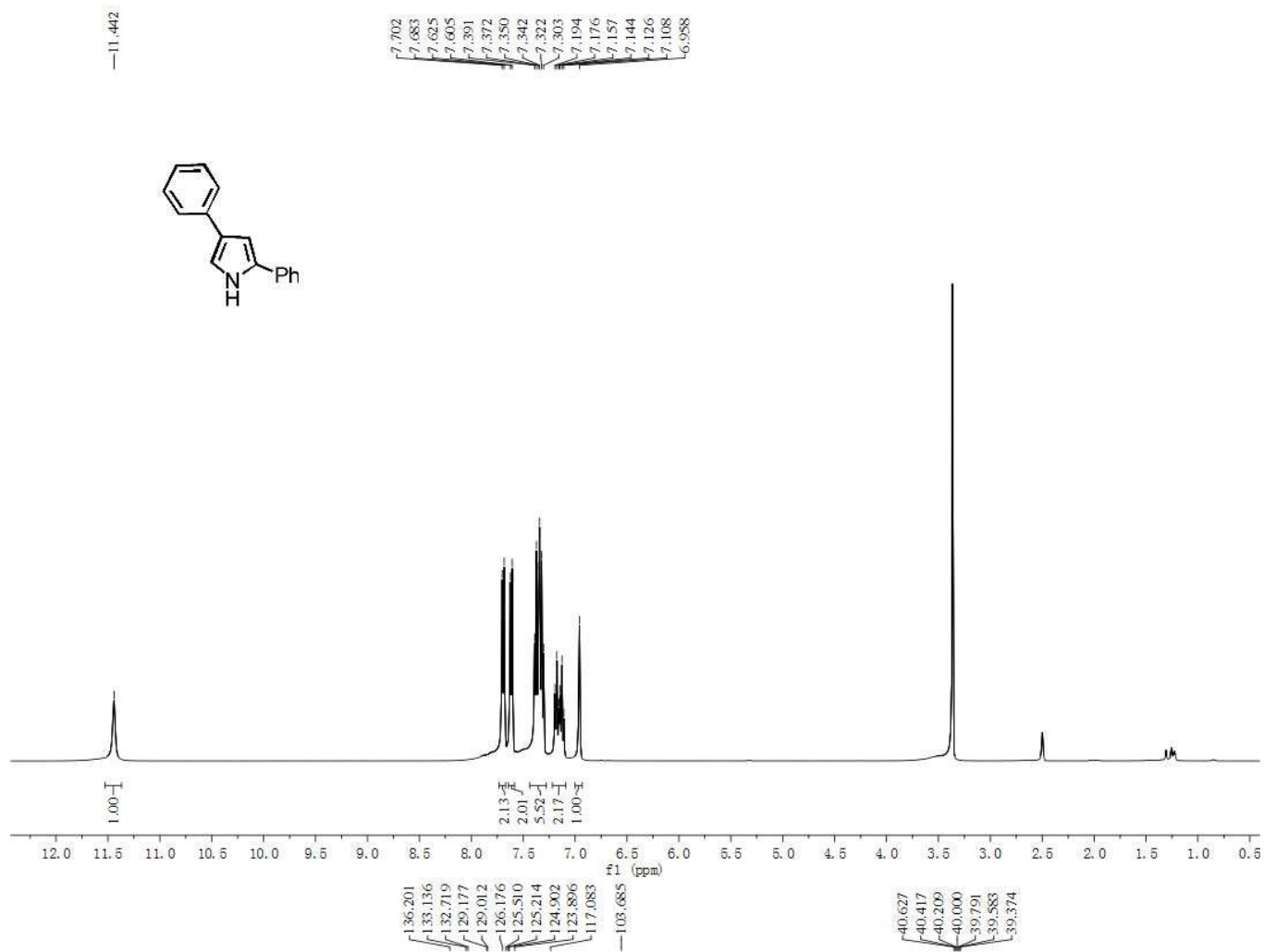
(4-(4-methoxybenzyl)-2-phenyl-1H-pyrrol-3-yl)(phenyl)methanone (**5**)

White solid; m.p. 72-75 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.61 (s, 1H), 7.59 (dd, *J* = 8.2, 1.1 Hz, 2H), 7.26 (t, *J* = 7.4, 7.4 Hz, 1H), 7.14 – 7.05 (m, 9H), 6.78 – 6.74 (m, 2H), 6.38 (d, *J* = 2.4 Hz, 1H), 3.89 (s, 2H), 3.74 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 194.8, 157.6, 139.3, 136.4, 133.4, 132.1, 131.7, 129.8, 129.7, 128.2, 128.0, 127.6, 127.3, 127.3, 119.6, 117.6, 113.6, 55.2, 31.5; HRMS (ESI) calcd for C₂₅H₂₂NO₂ [M+H]⁺ 368.1645, found: 368.1651.

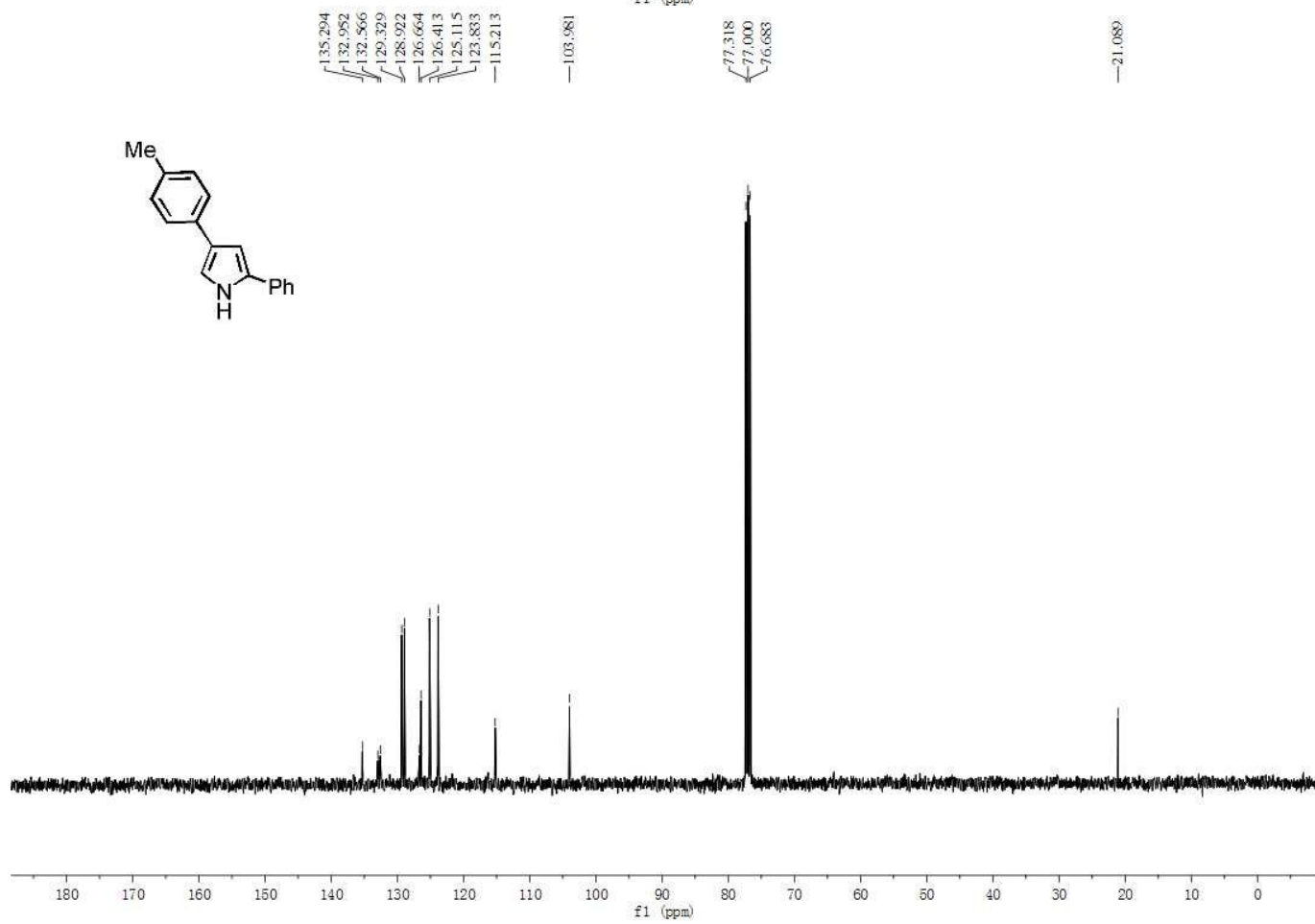
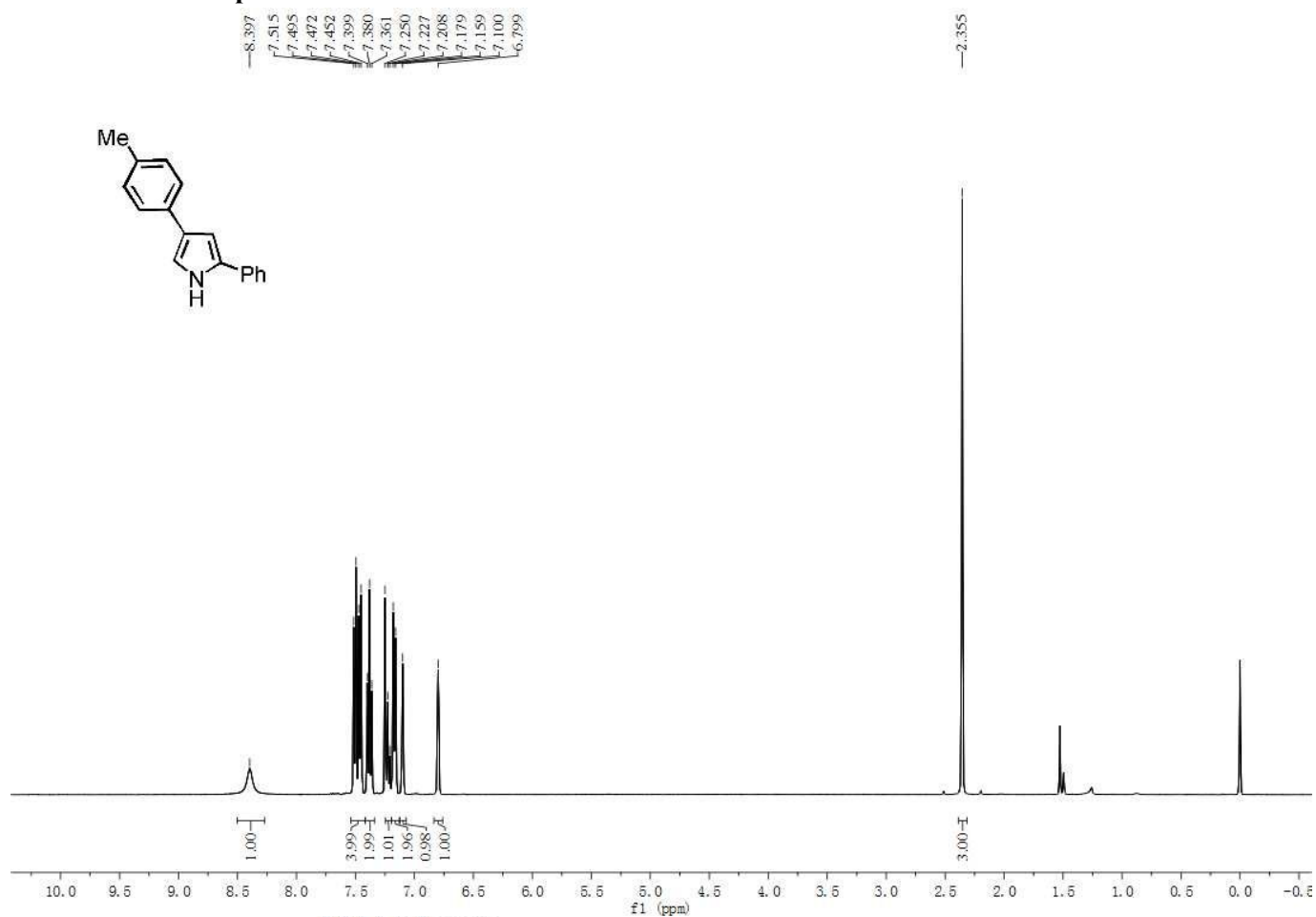
Reference:

1. K. Goutham, D. Ashok Kumar, S. Suresh, B. Sridhar, R. Narendar and G. V. Karunakar, *J. Org. Chem.* 2015, **80**, 11162.
2. F. Chen, T. Shen, Y. Cui and N. Jiao, *Org. Lett.*, 2012, **14**, 4926.
3. M. Adib, N. Ayashi, F. Heidari and P. Mirzaei, *Synlett*, 2016, **27**, 1738.
4. R. Umeda, T. Mashino and Y. Nishiyama, *Tetrahedron*, 2014, **70**, 4395.

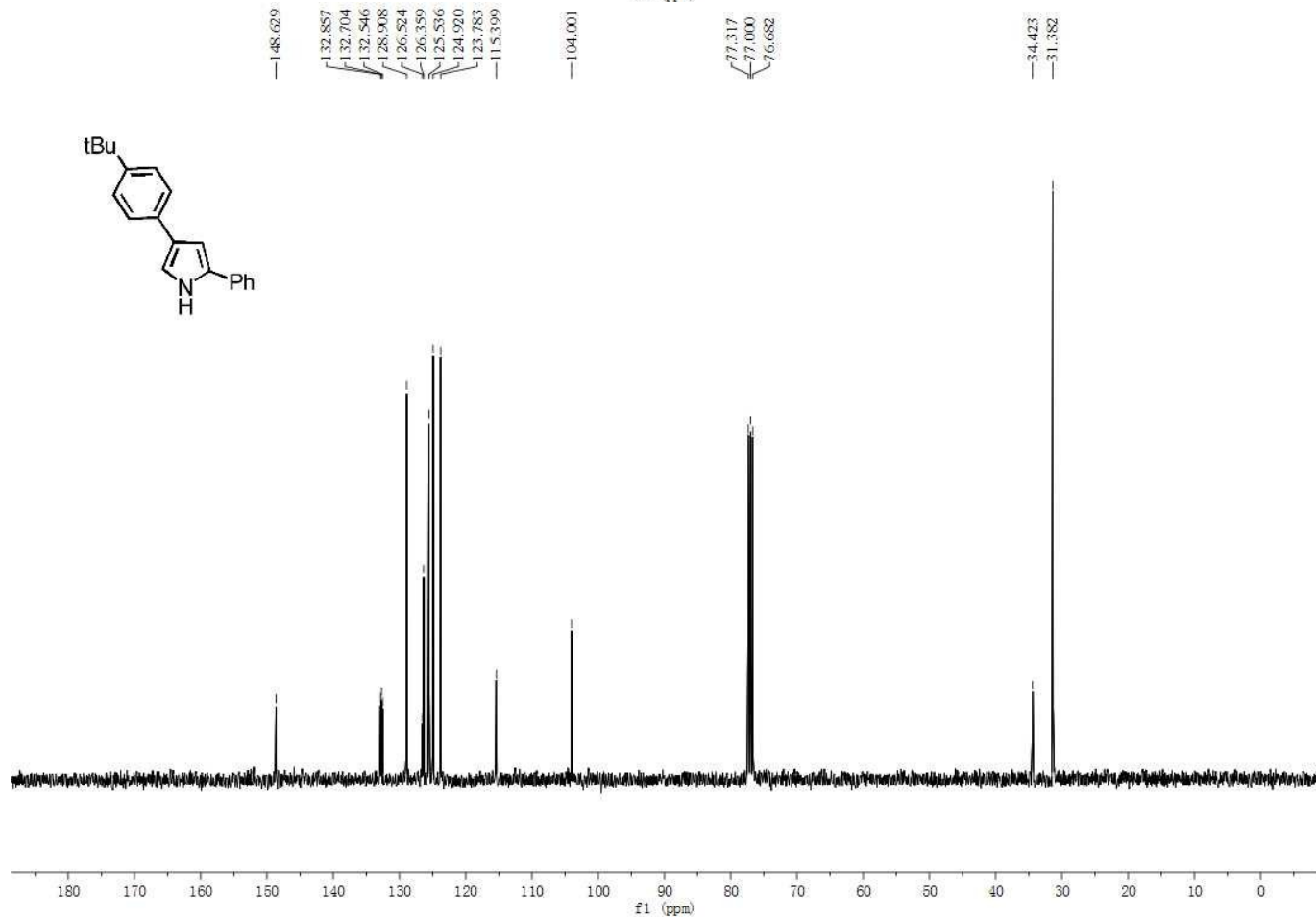
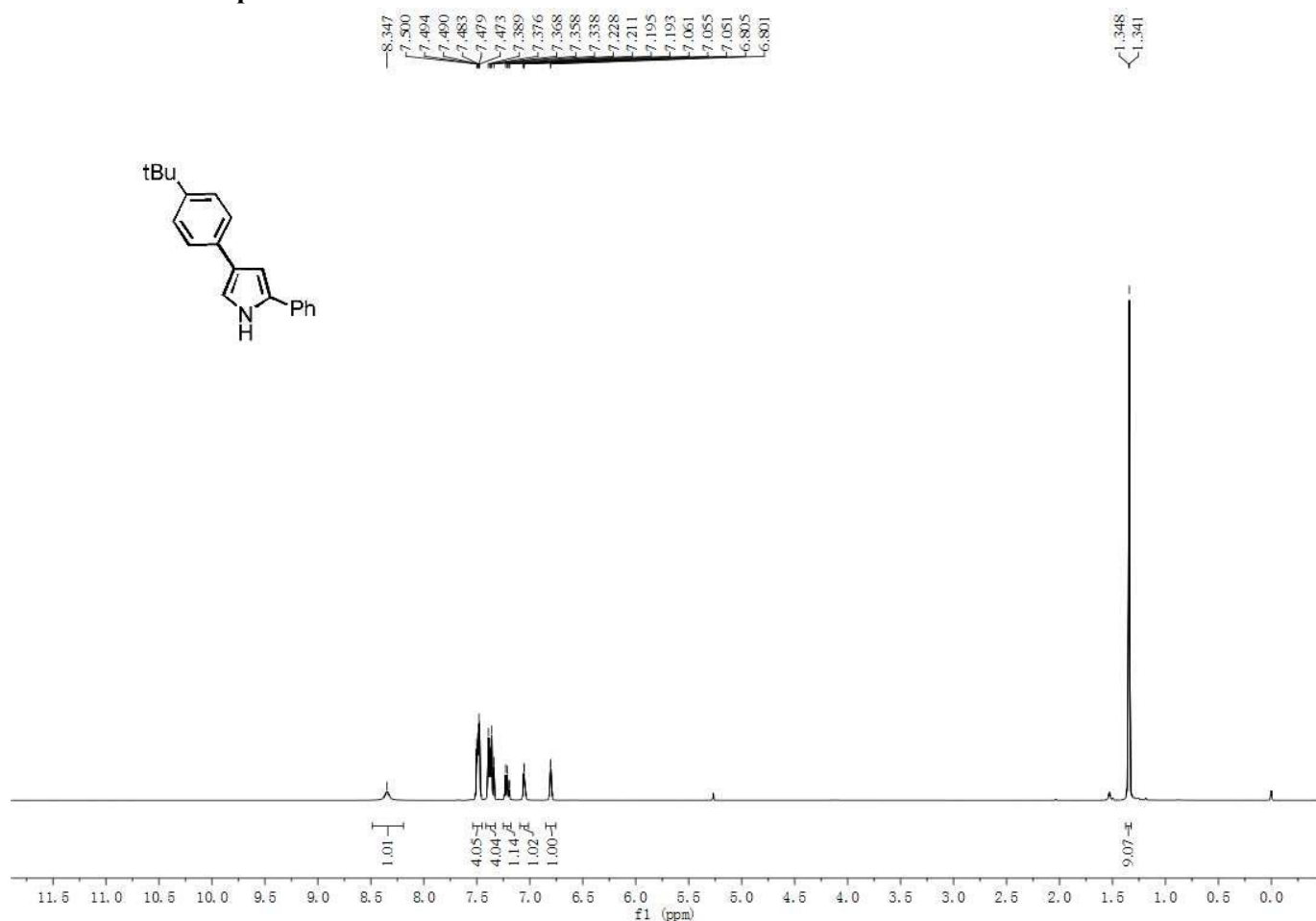
¹H and ¹³C NMR Spectra of 2a



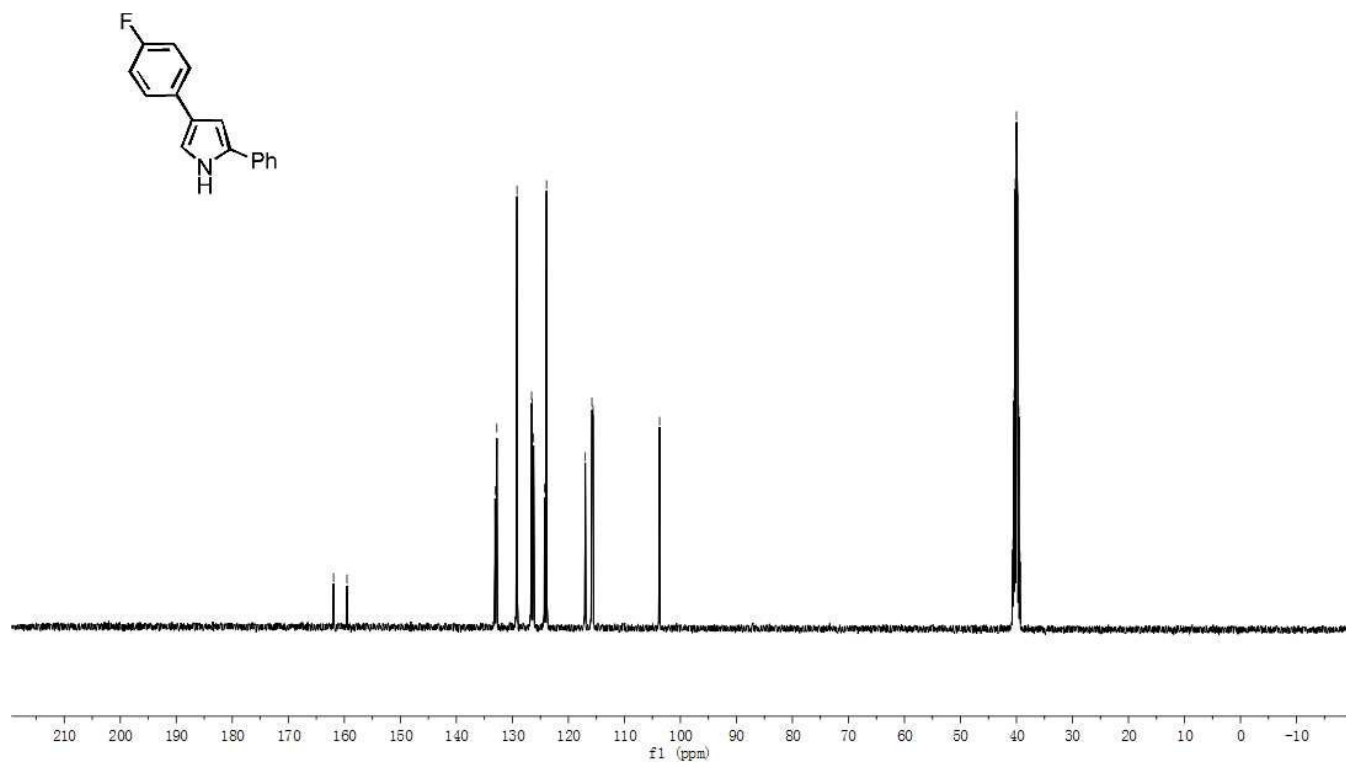
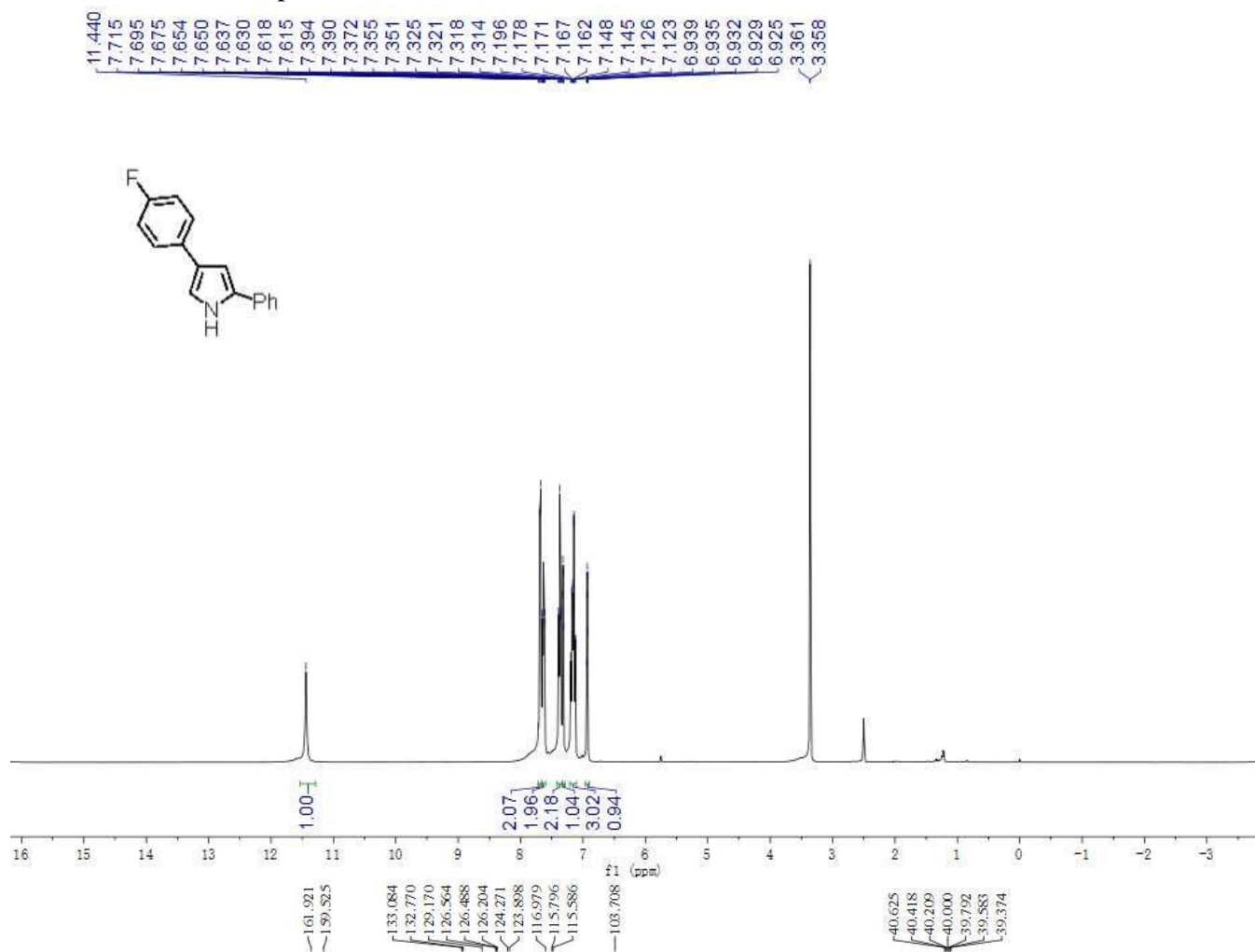
¹H and ¹³C NMR Spectra of 2b

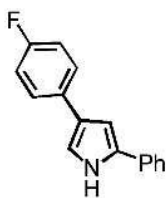


¹H and ¹³C NMR Spectra of 2c

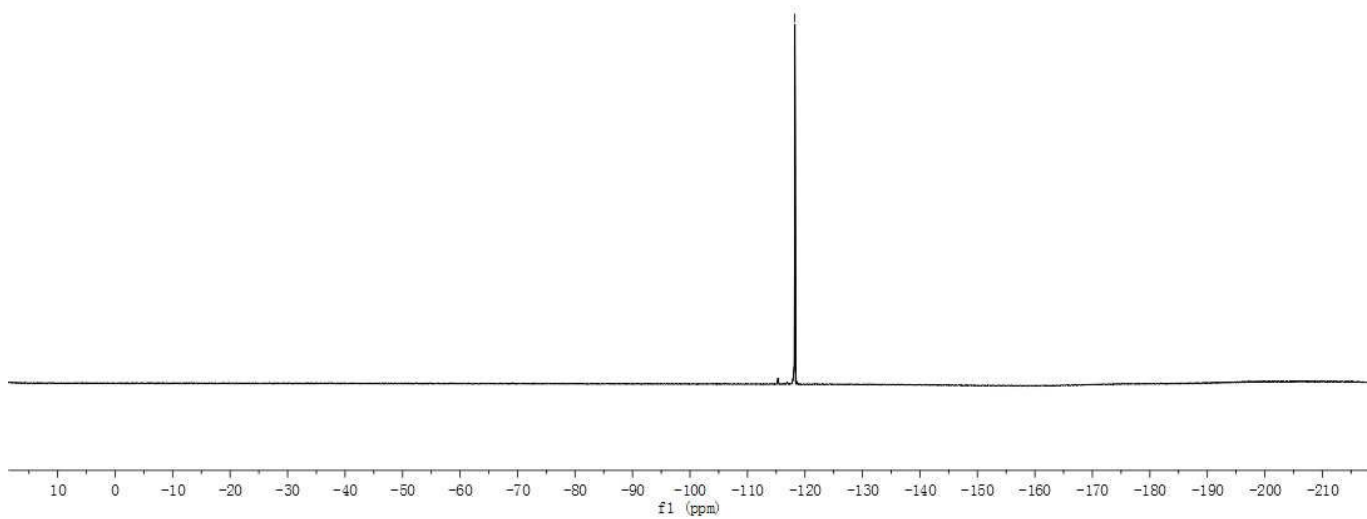


¹H, ¹³C and ¹⁹F NMR Spectra of 2d



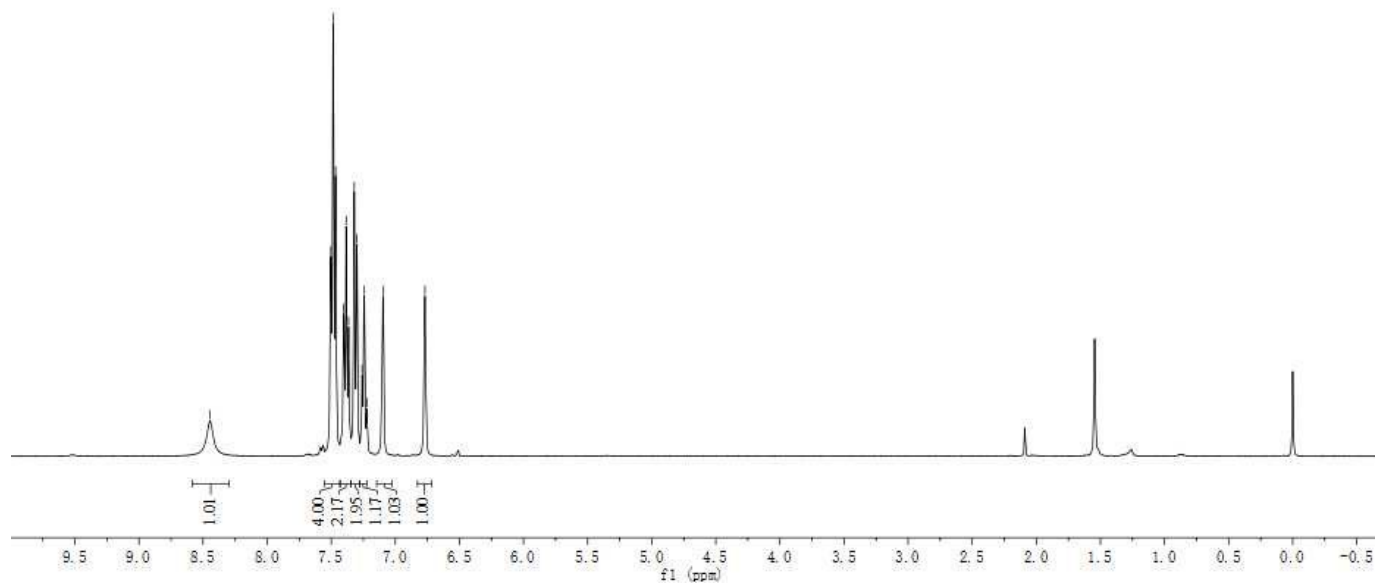
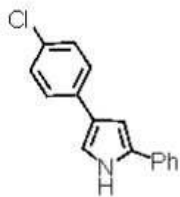


118.201
118.225
118.239
118.251
118.259
118.276

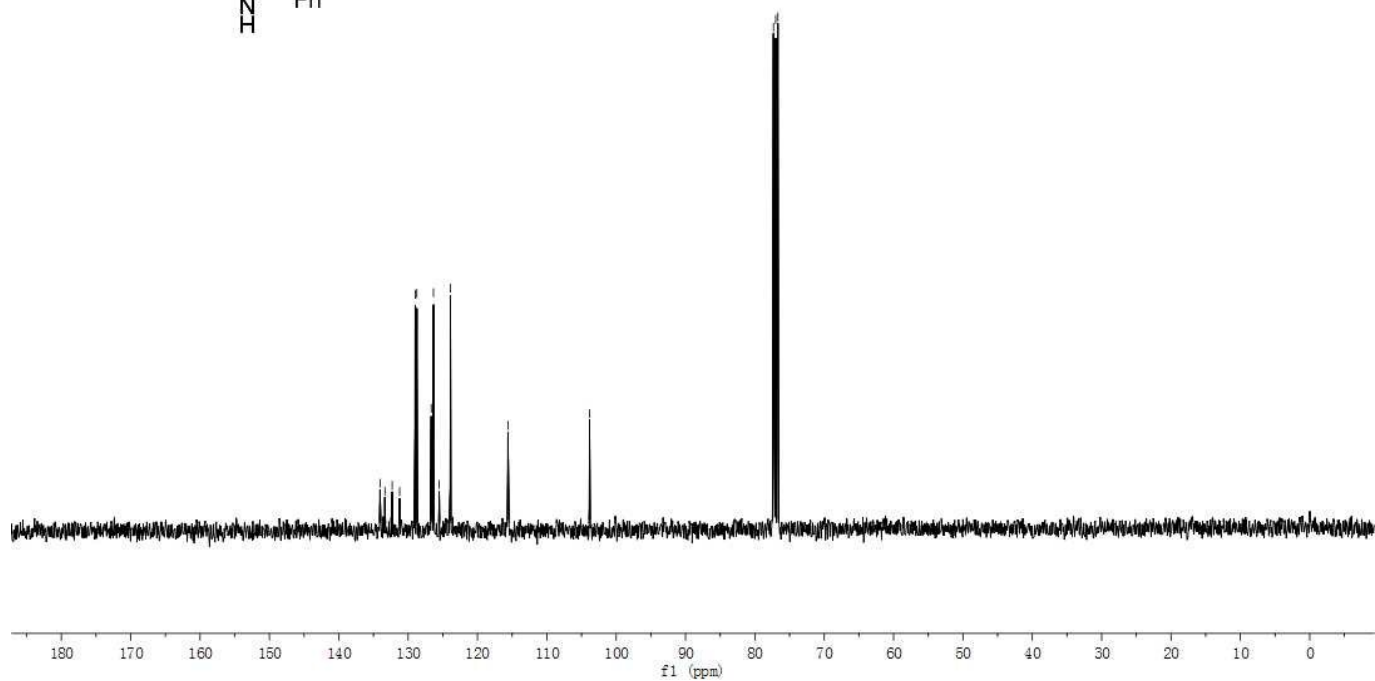
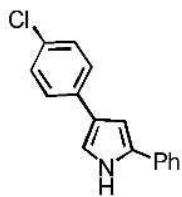


¹H and ¹³C NMR Spectra of 2e

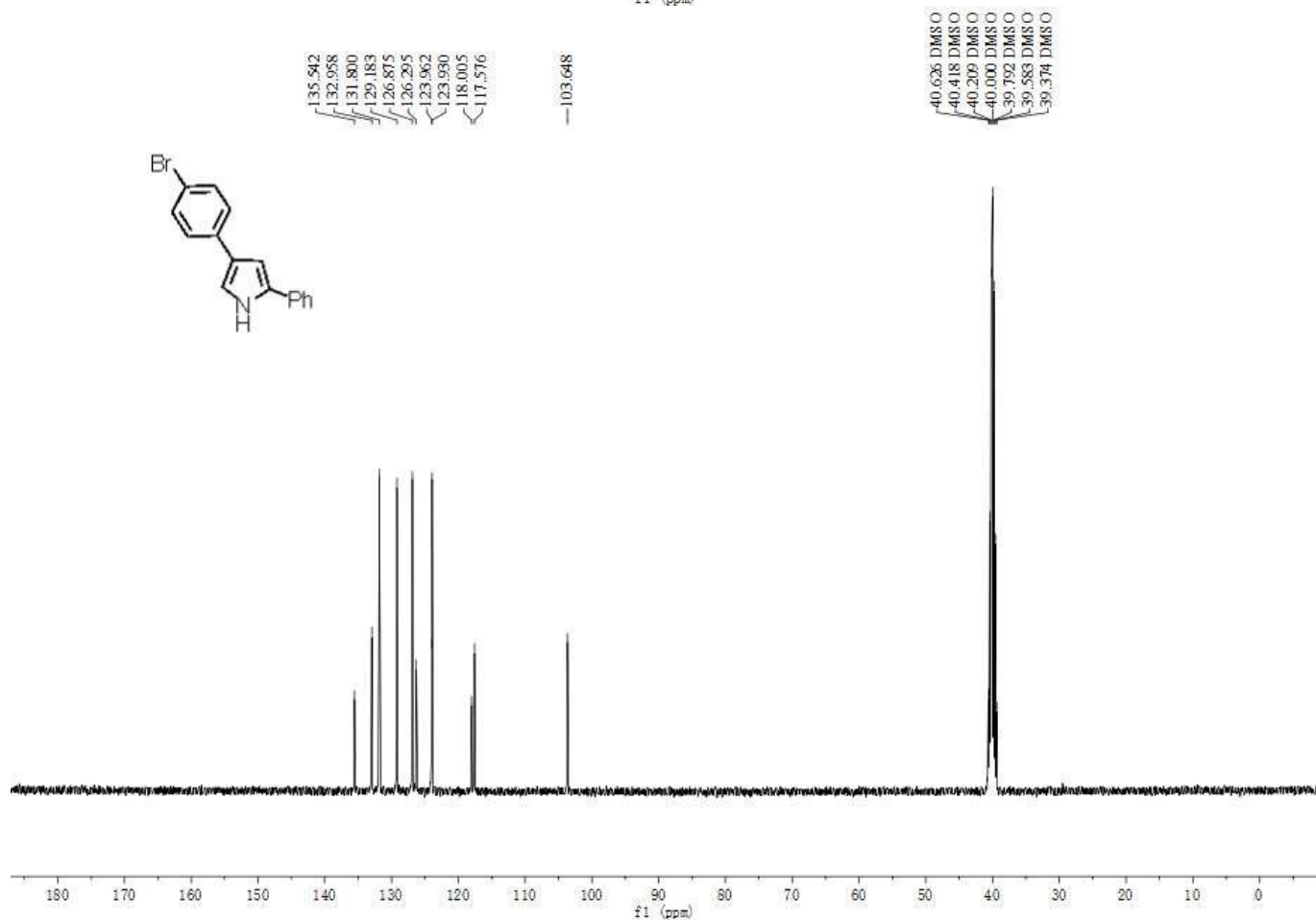
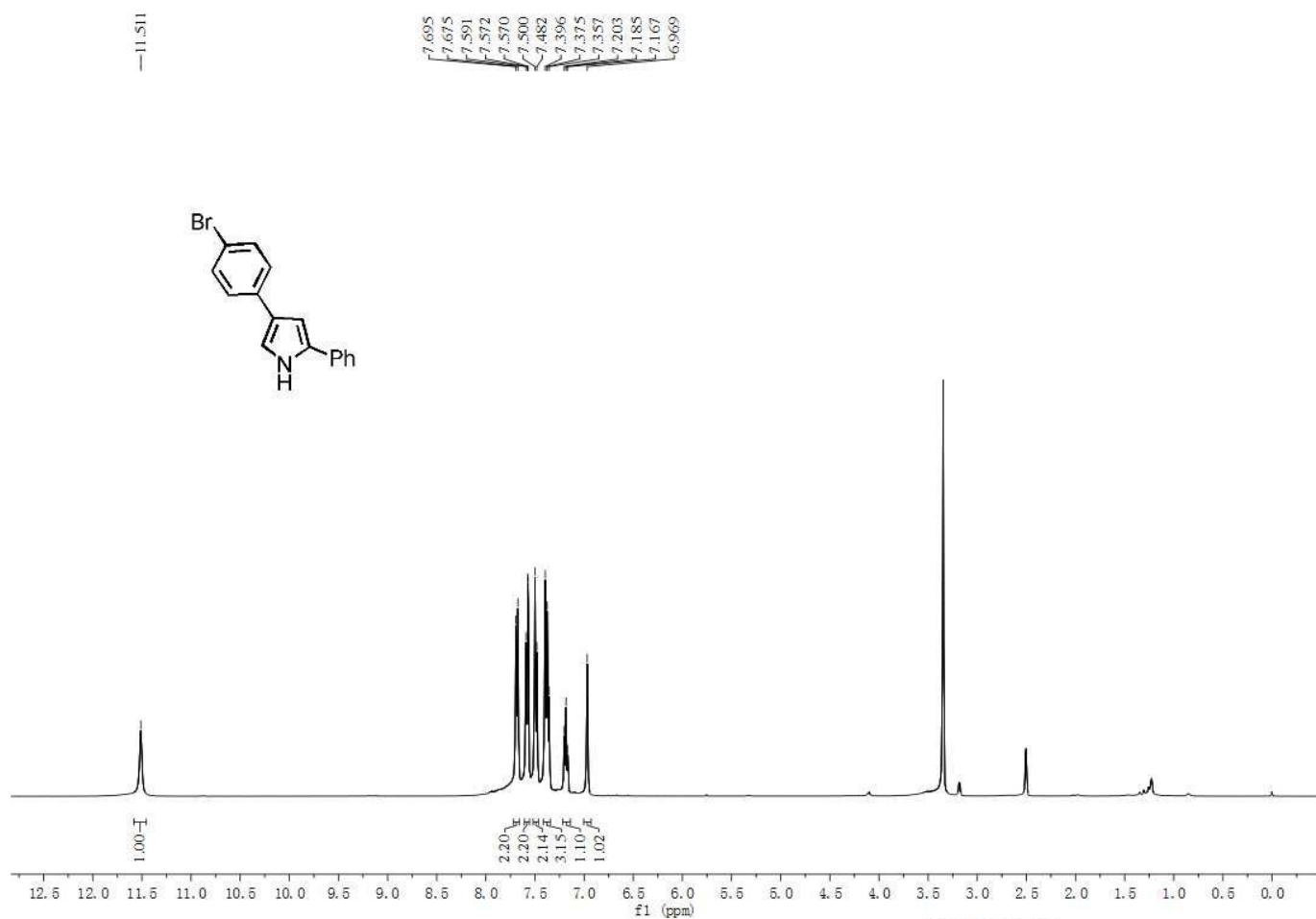
8.444
7.503
7.486
7.466
7.403
7.384
7.365
7.320
7.299
7.259
7.245
7.223
7.097
6.769



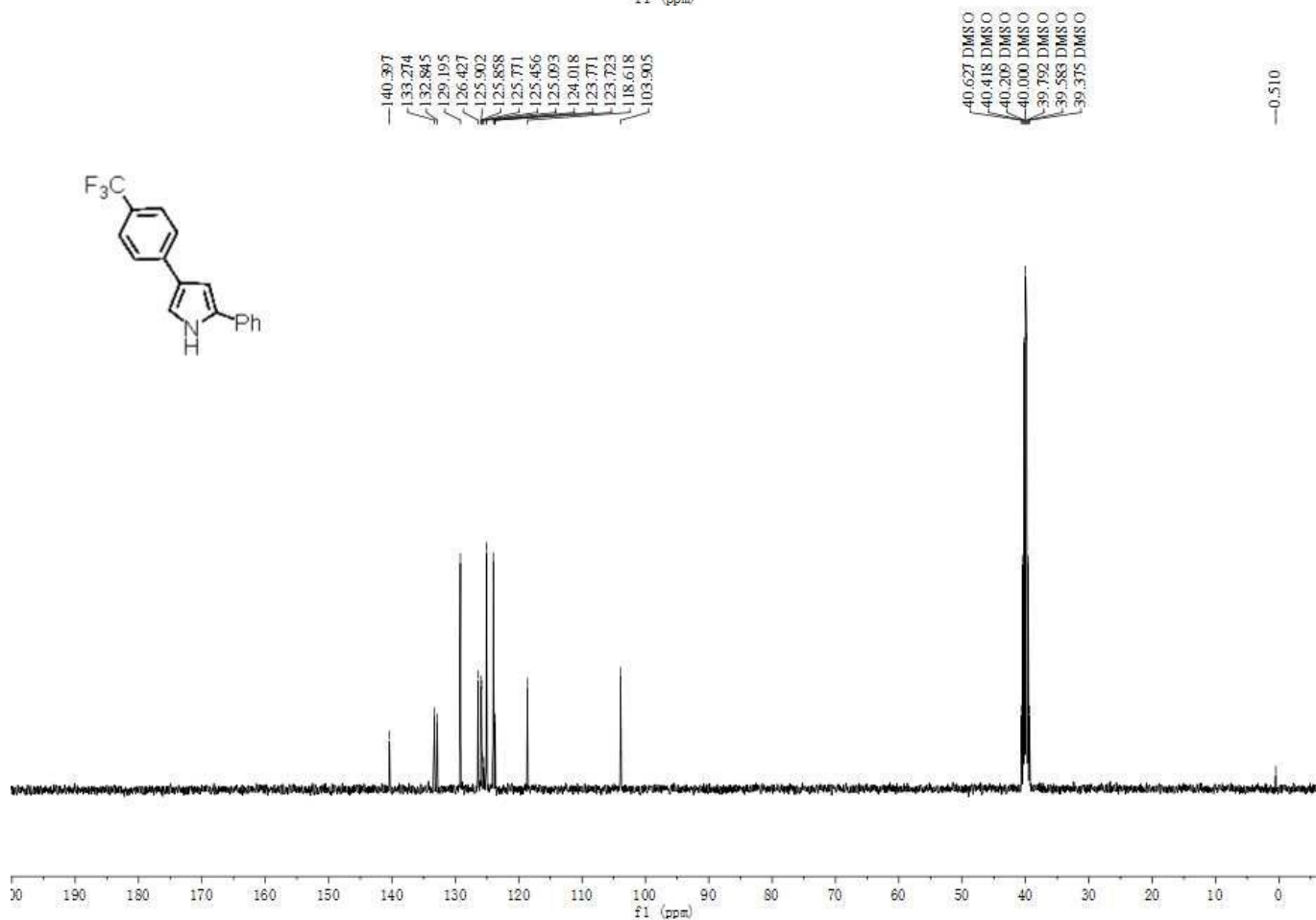
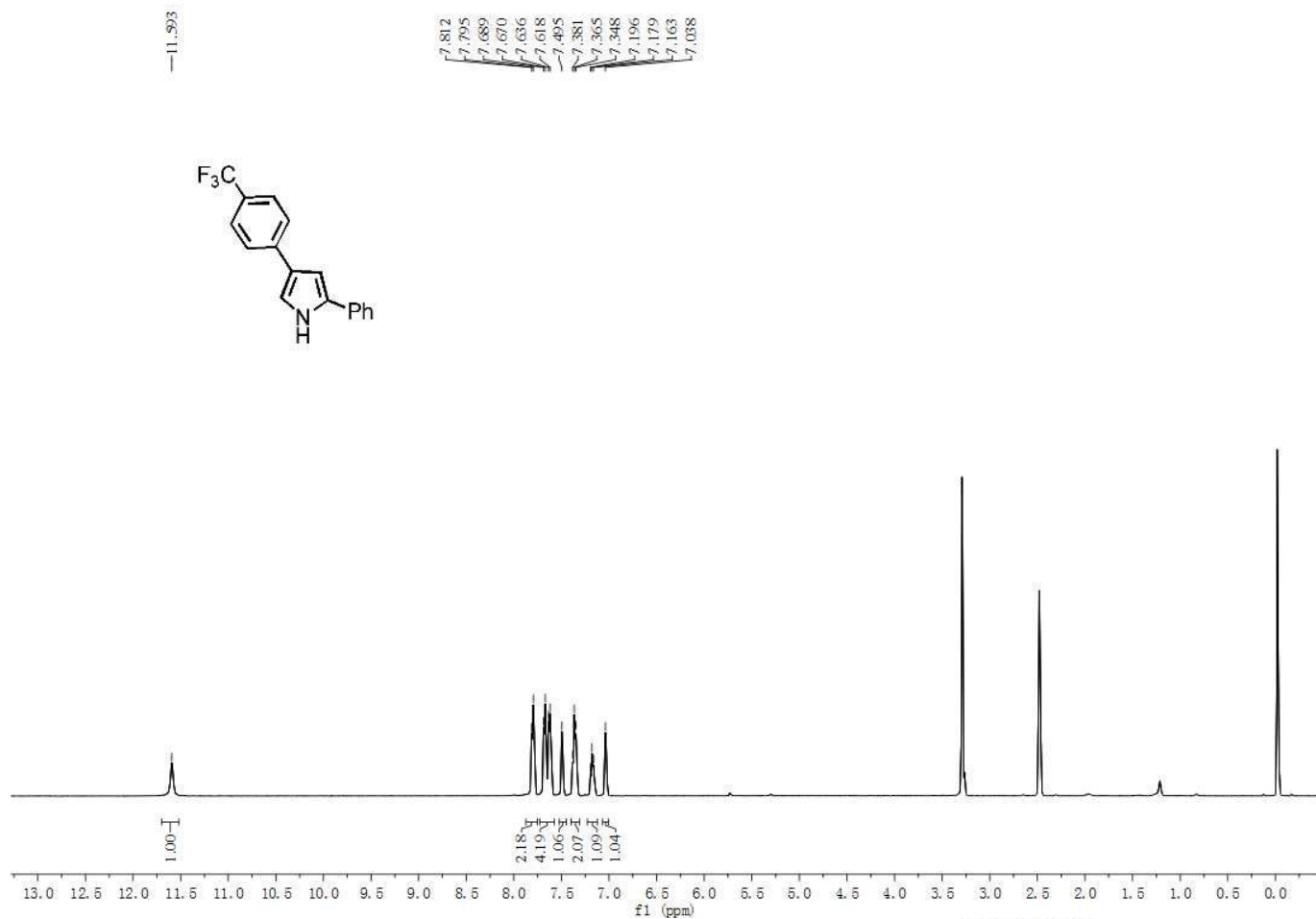
134.049
133.343
132.292
131.217
128.981
128.739
126.663
126.341
125.515
123.895
115.606
103.835
77.318
77.000
76.682

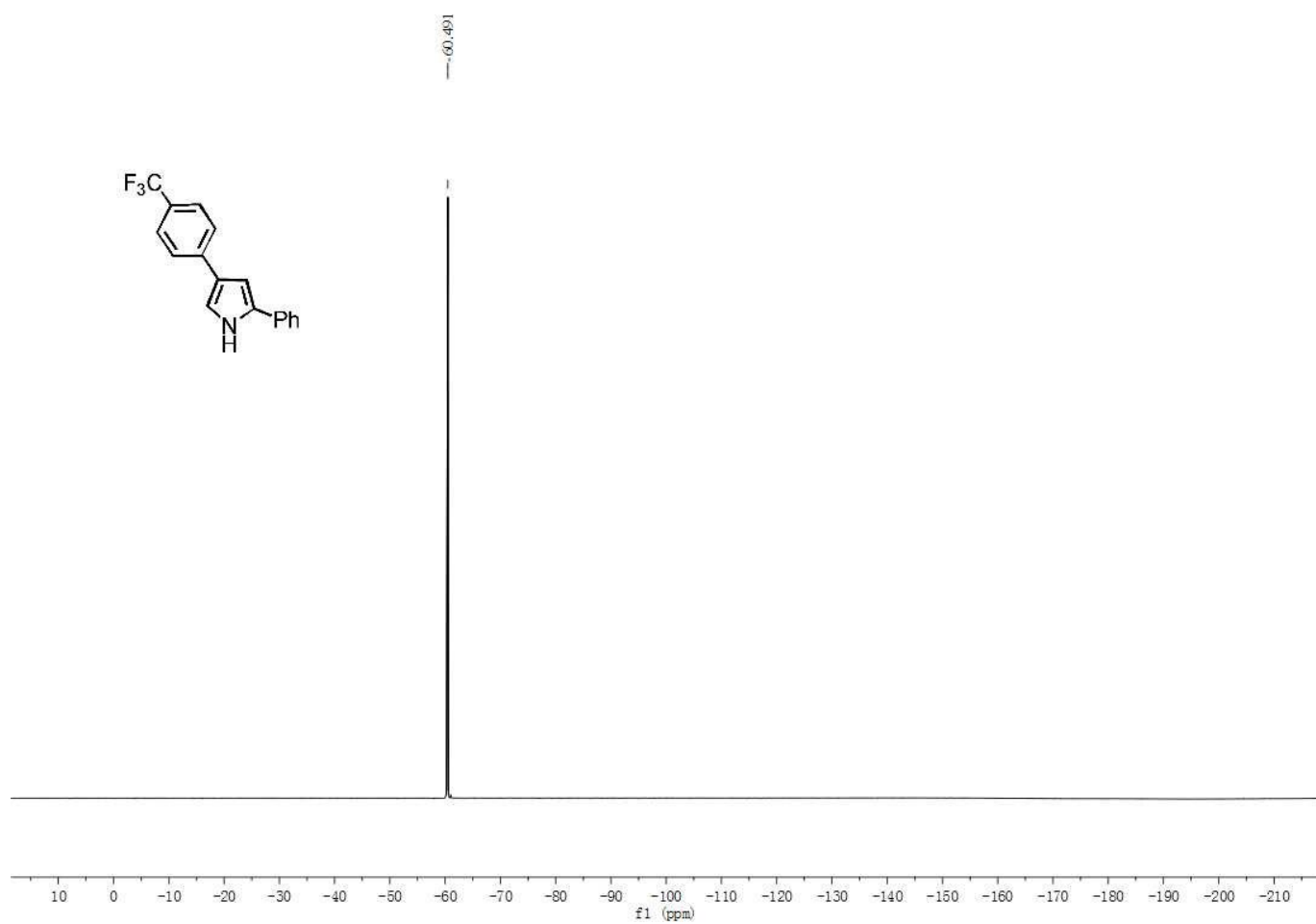
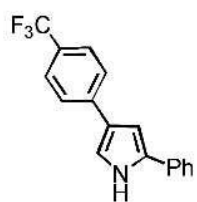


¹H and ¹³C NMR Spectra of 2f

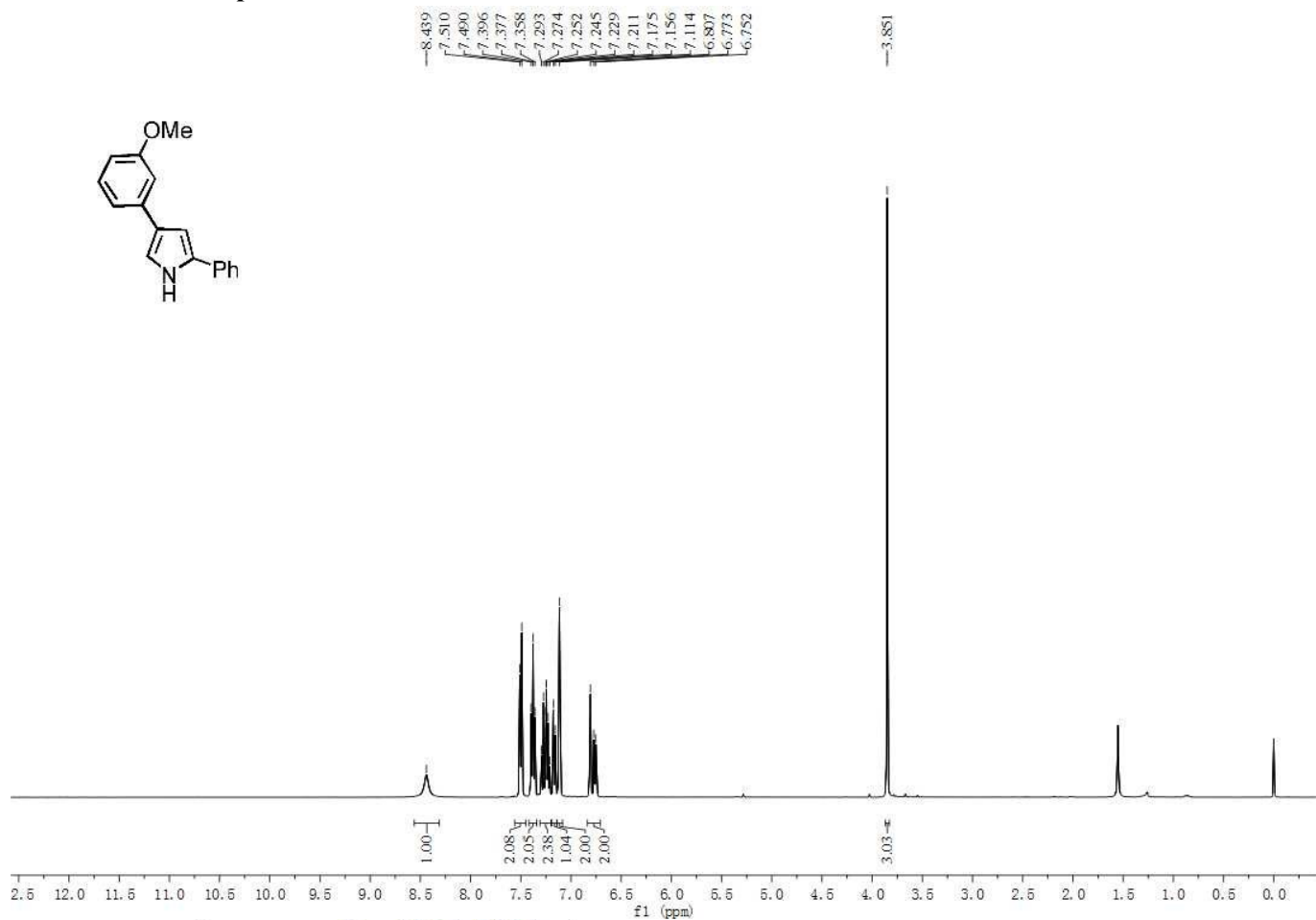


¹H, ¹³C and ¹⁹F NMR Spectra of 2g

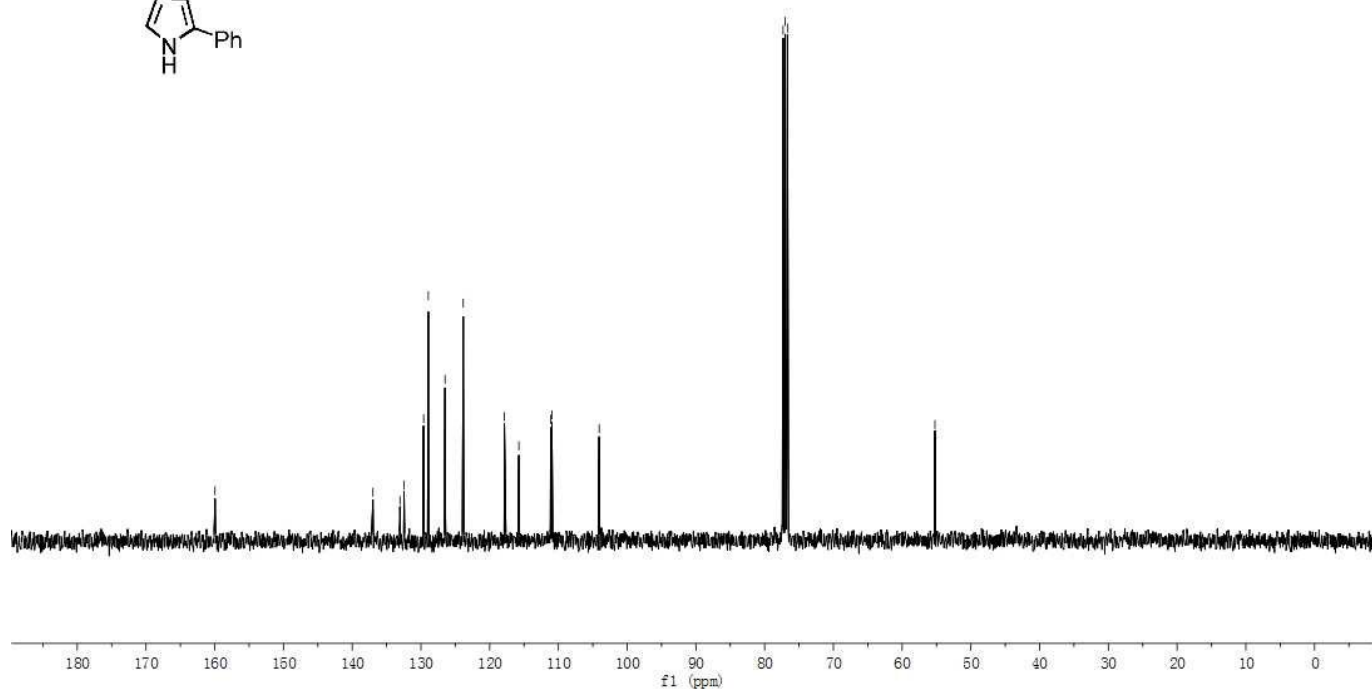
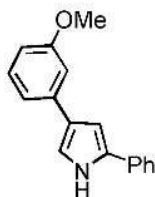
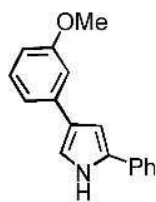




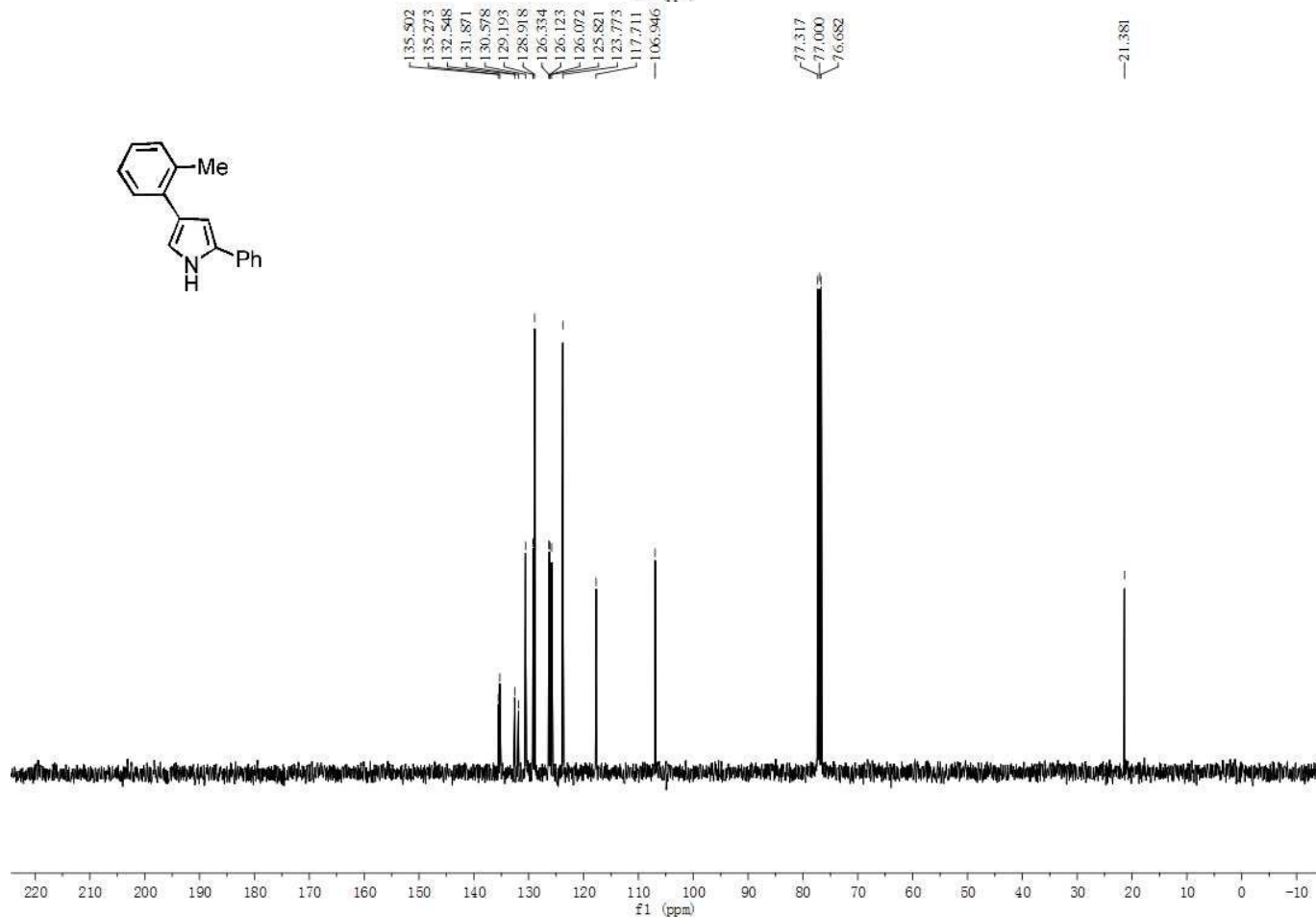
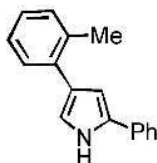
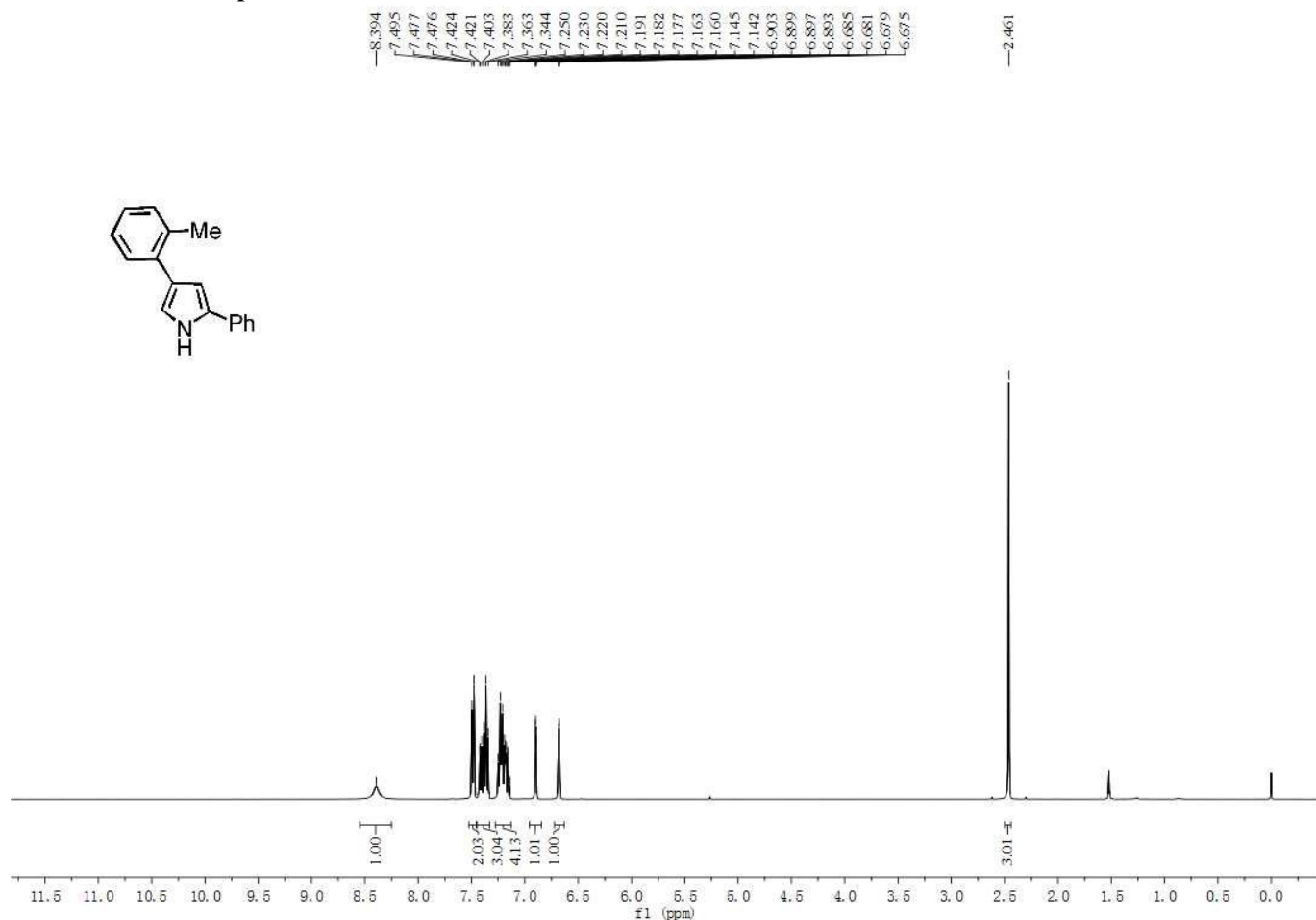
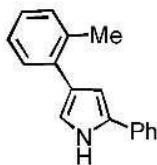
¹H and ¹³C NMR Spectra of 2h



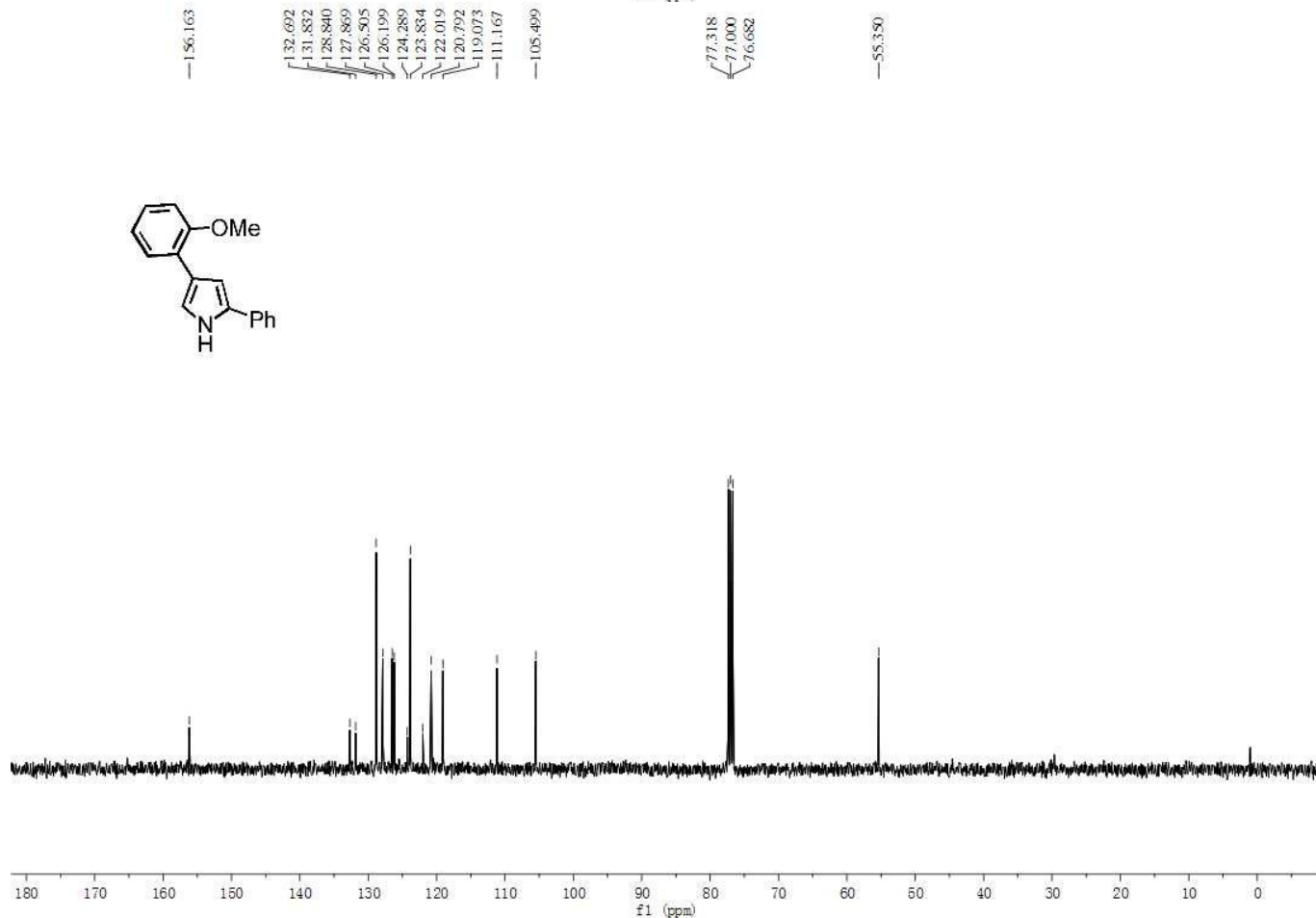
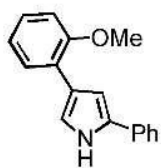
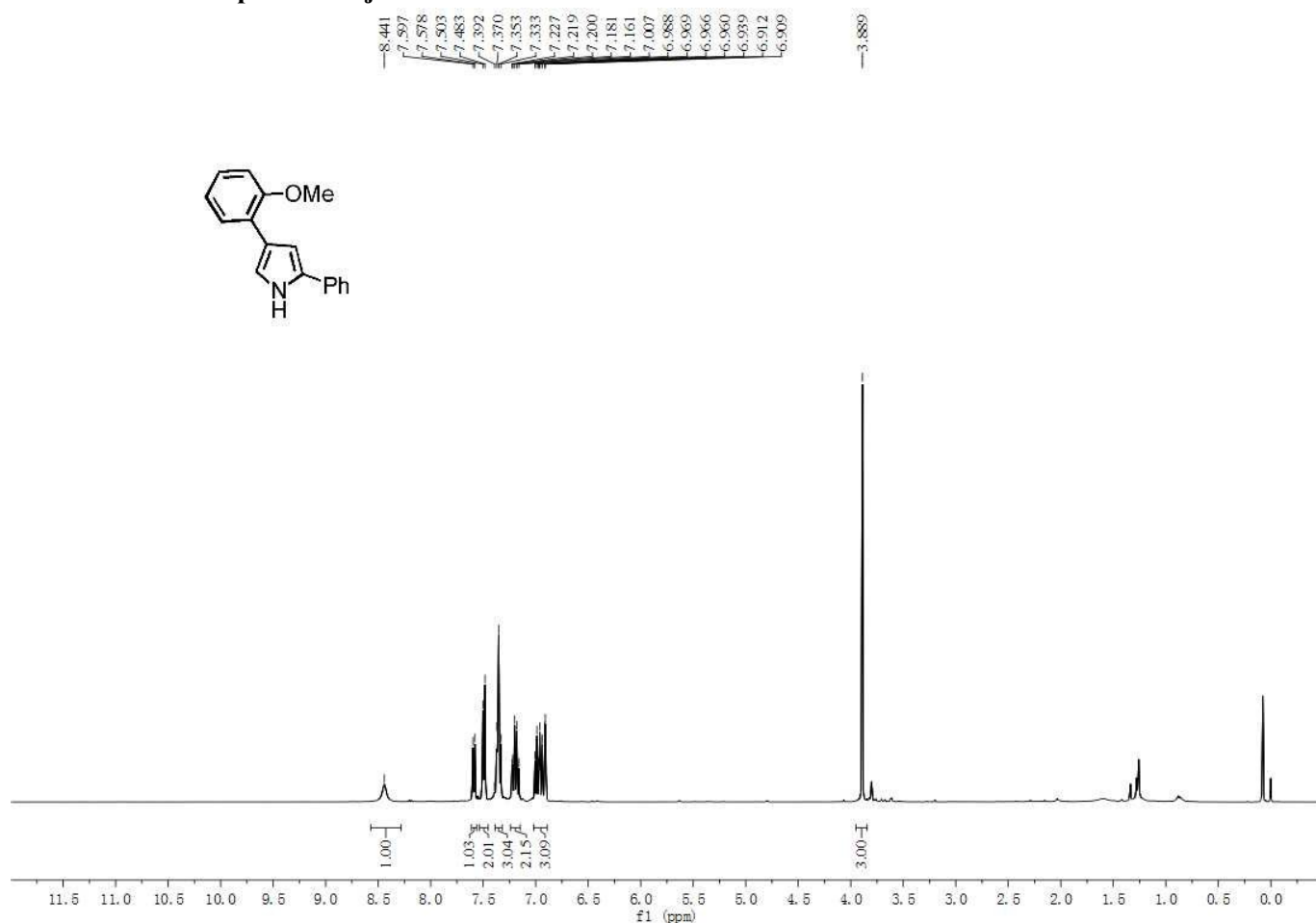
8.439
7.510
7.490
7.396
7.377
7.358
7.293
7.274
7.252
7.245
7.229
7.211
7.175
7.156
7.114
6.807
6.773
6.752



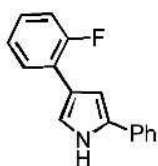
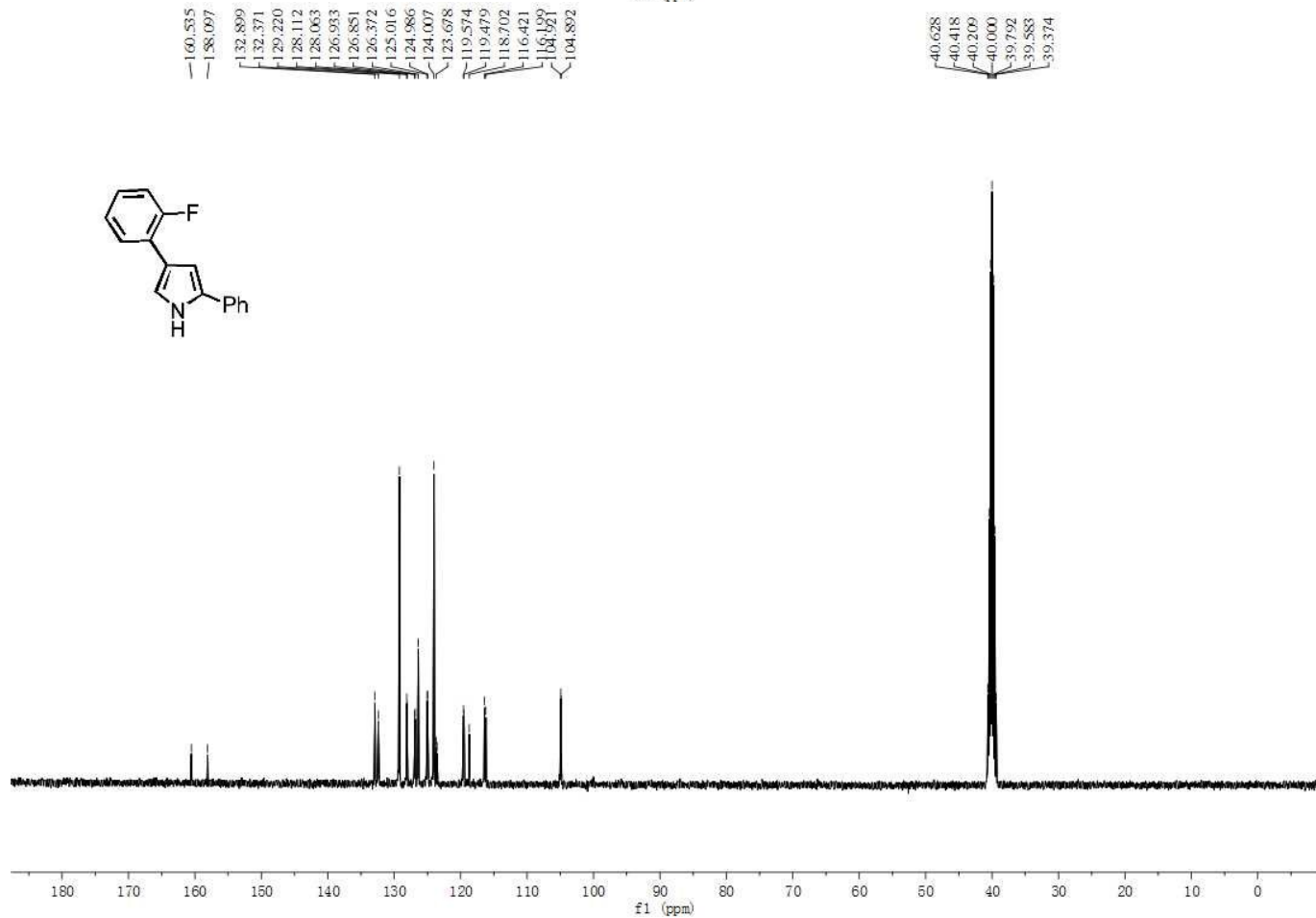
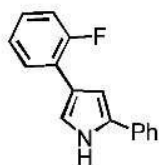
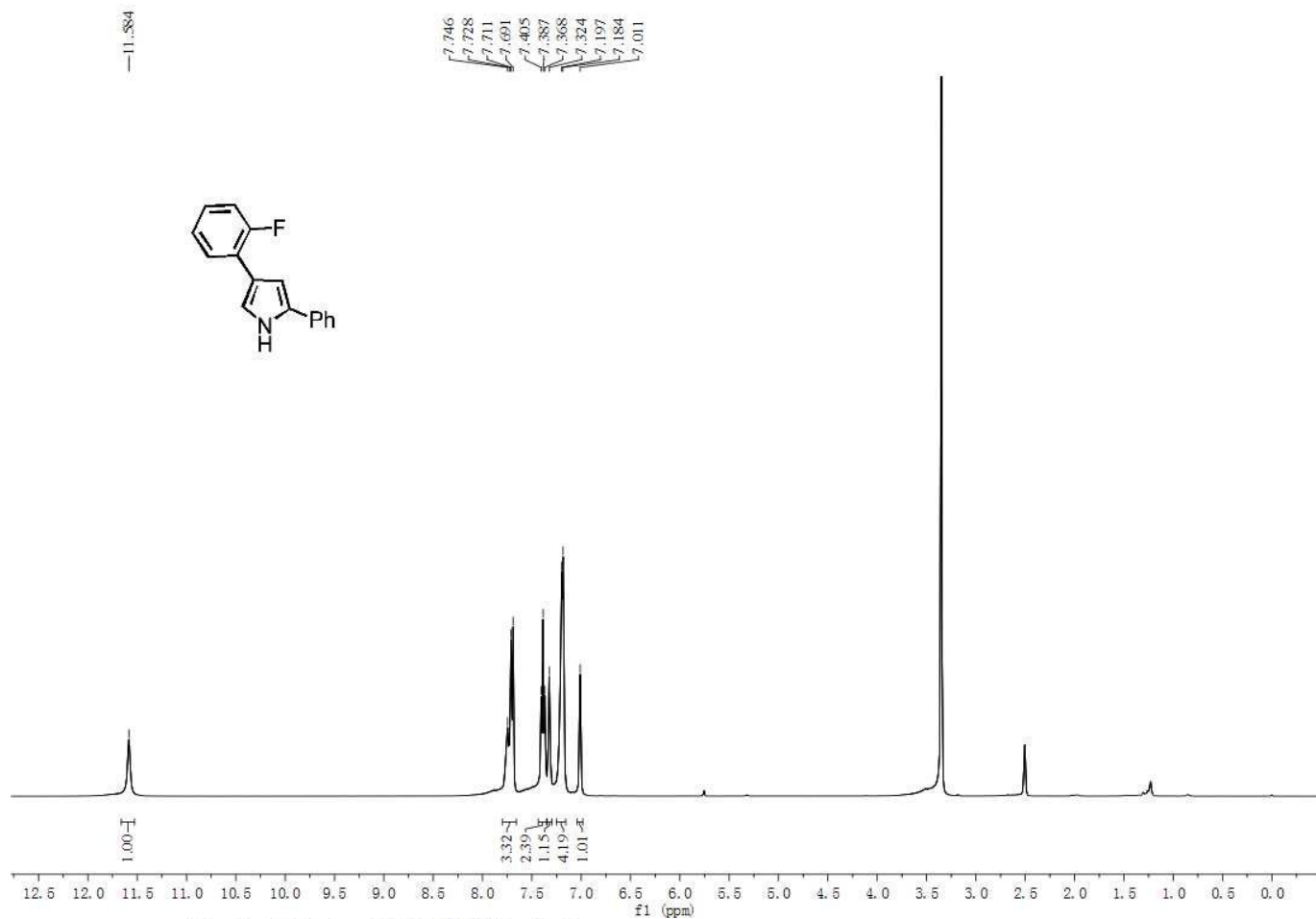
¹H and ¹³C NMR Spectra of 2i

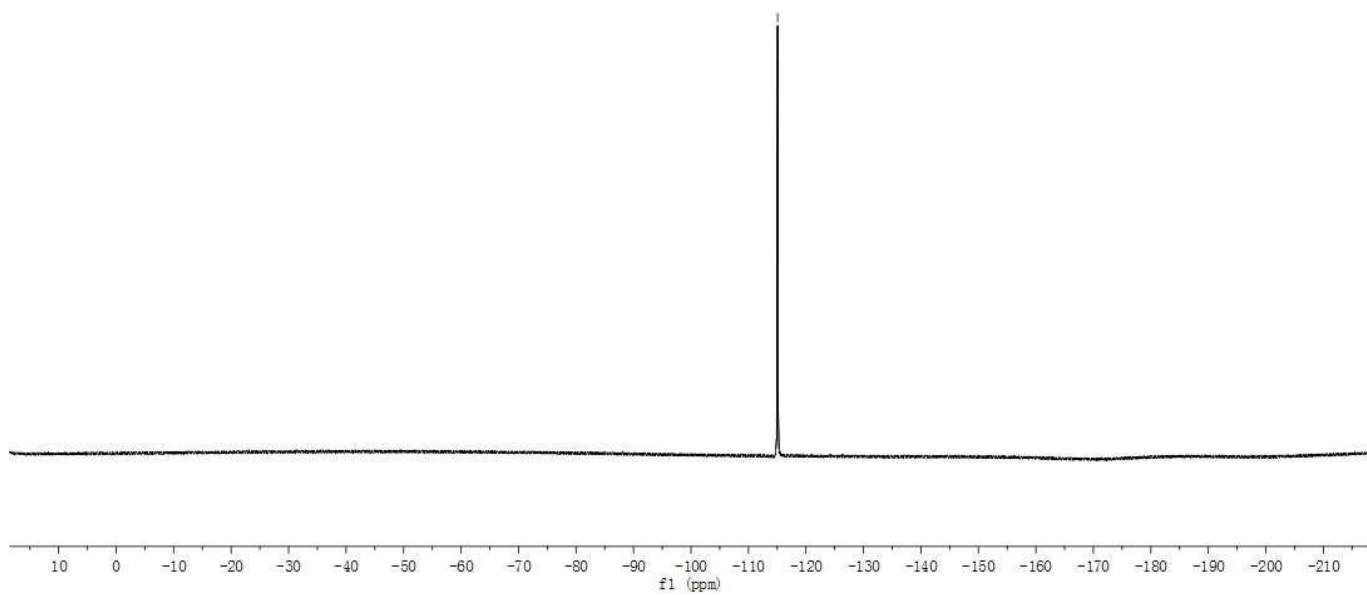
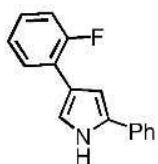


¹H and ¹³C NMR Spectra of 2j



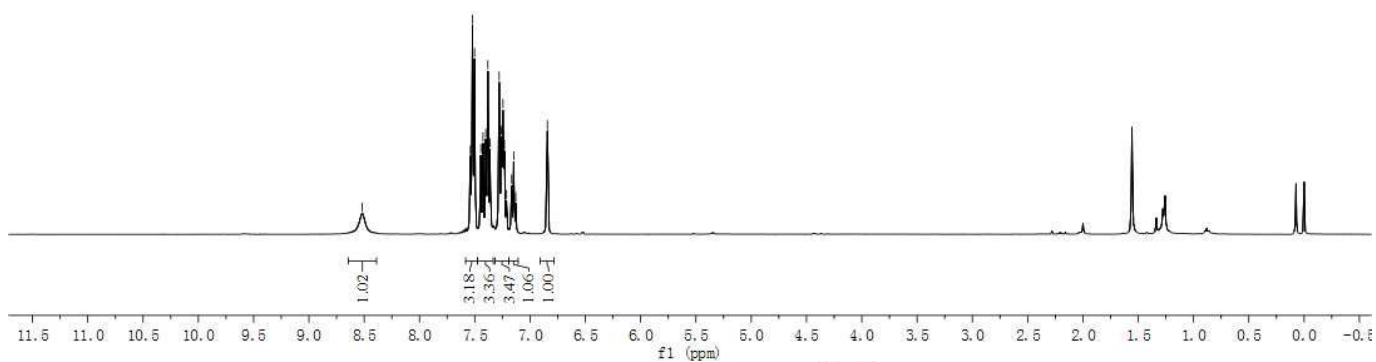
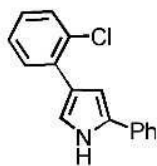
¹H, ¹³C and ¹⁹F NMR Spectra of 2k



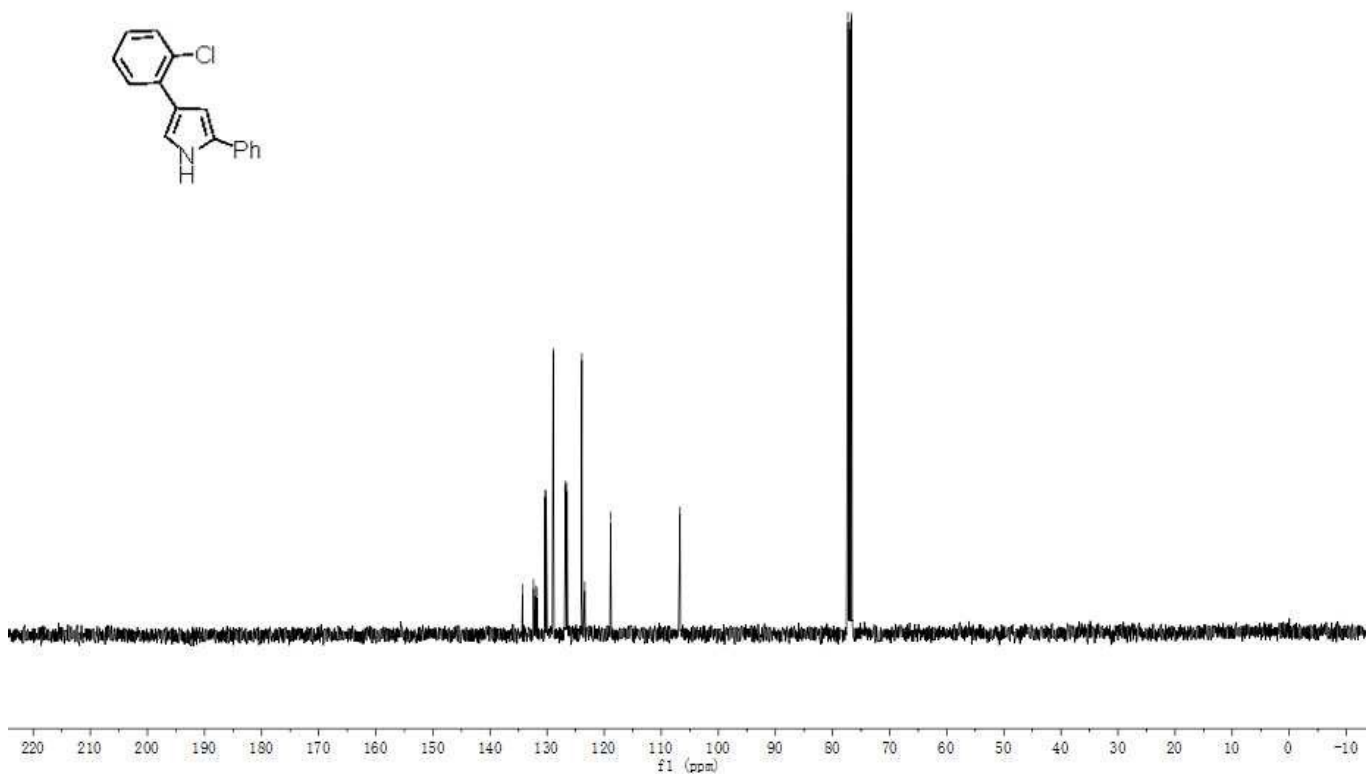
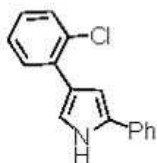


¹H and ¹³C NMR Spectra of 2l

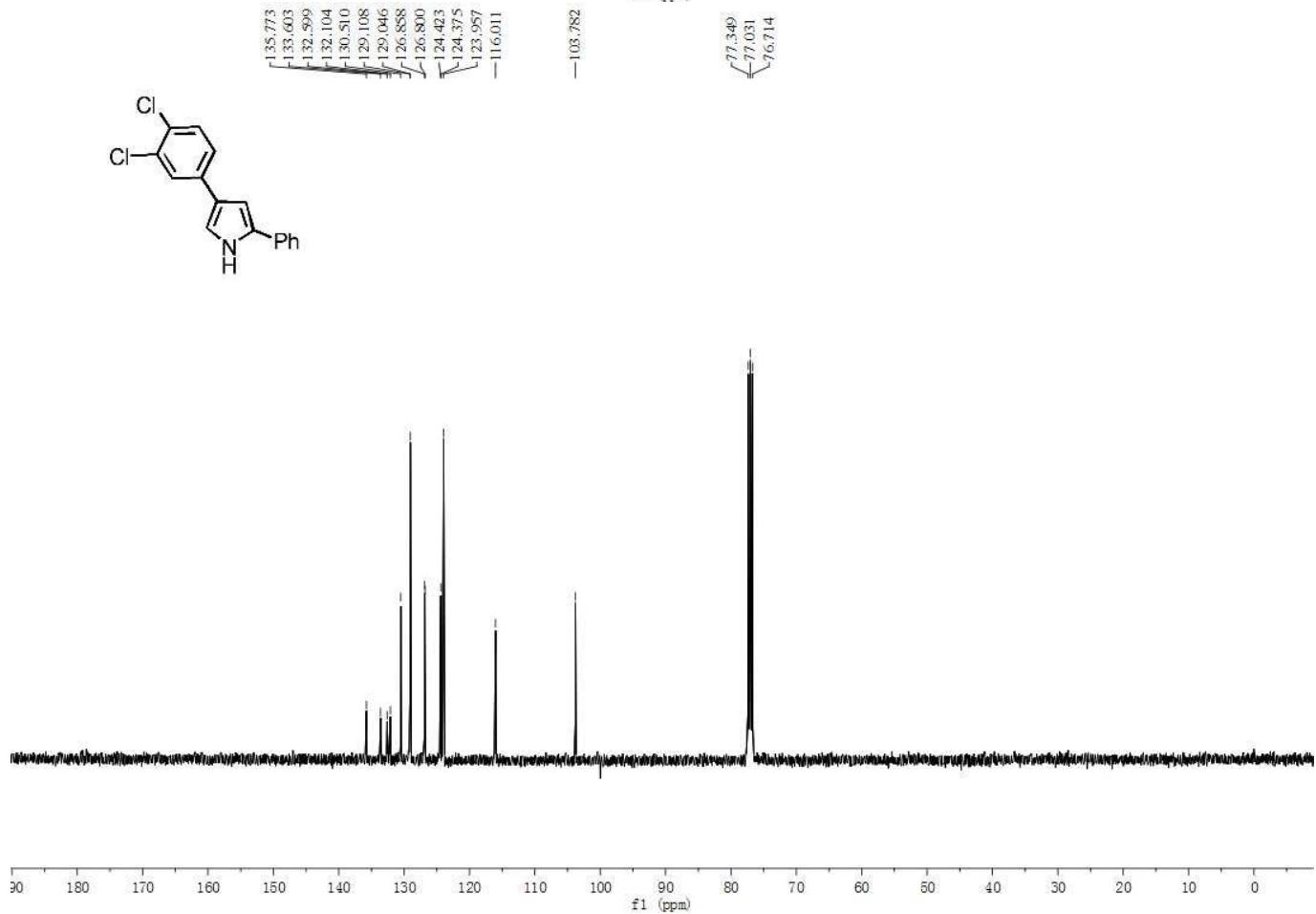
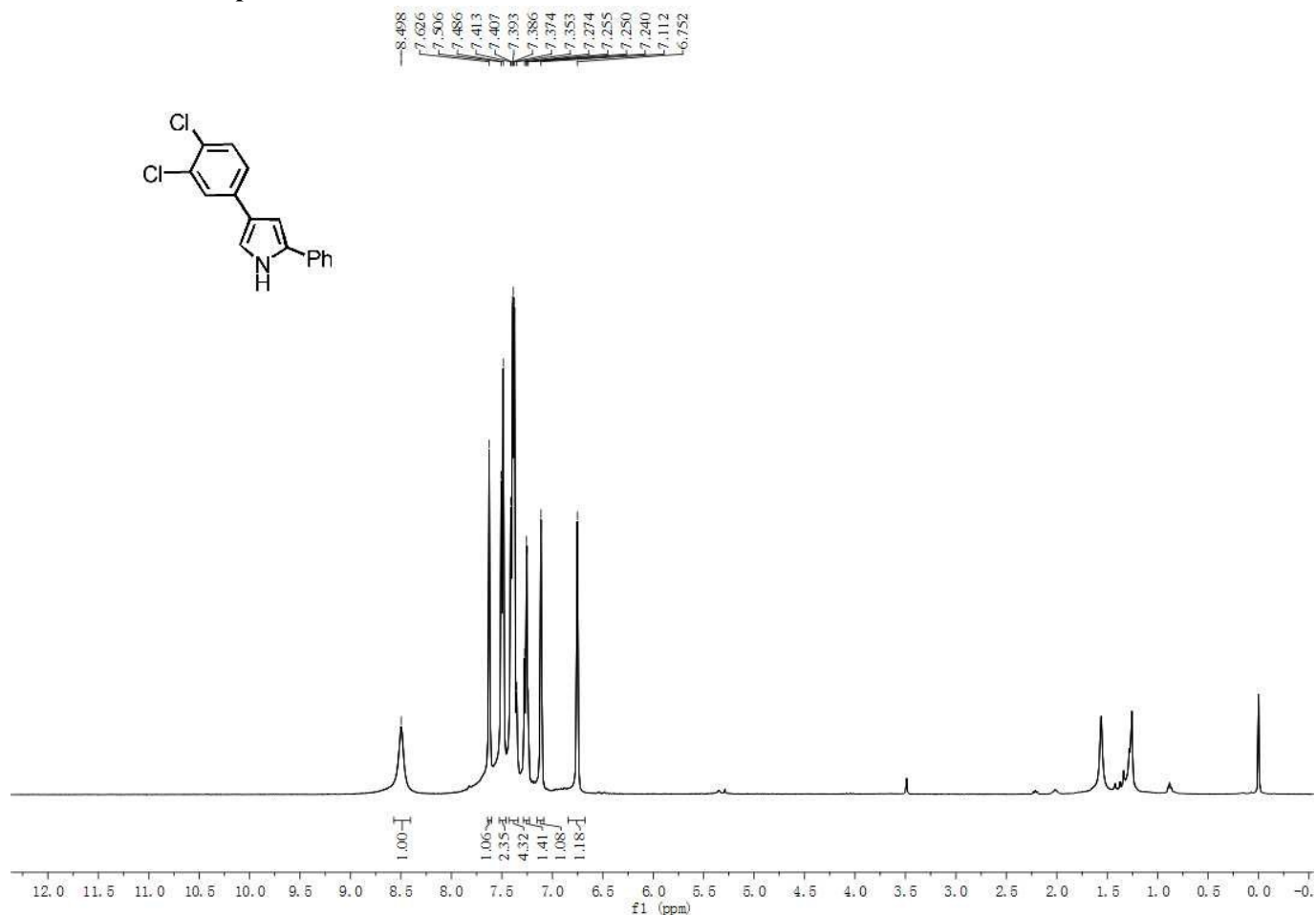
8.518
7.540
7.502
7.445
7.425
7.400
7.382
7.363
7.279
7.260
7.245
7.232
7.213
7.167
7.148
7.130
6.843



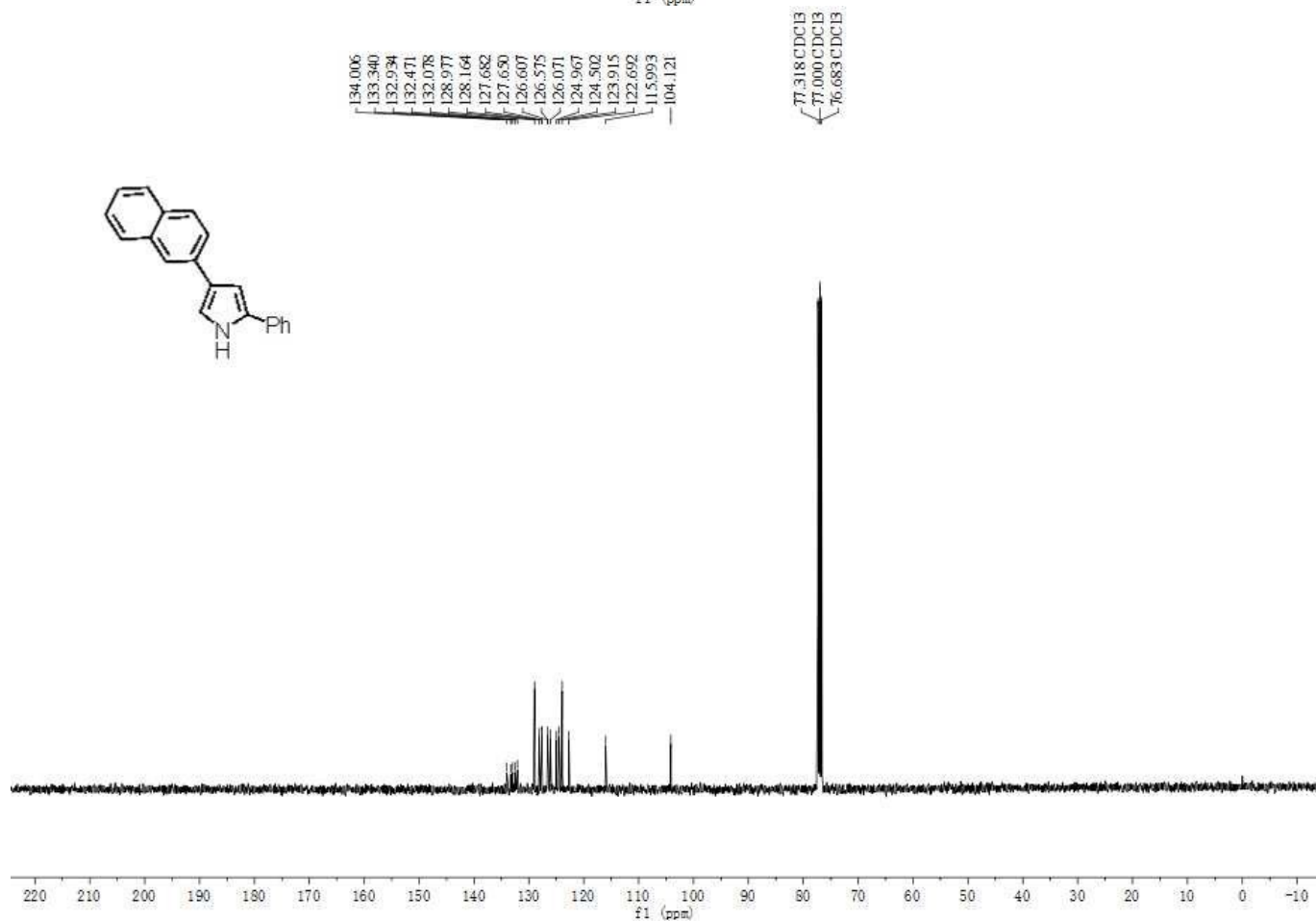
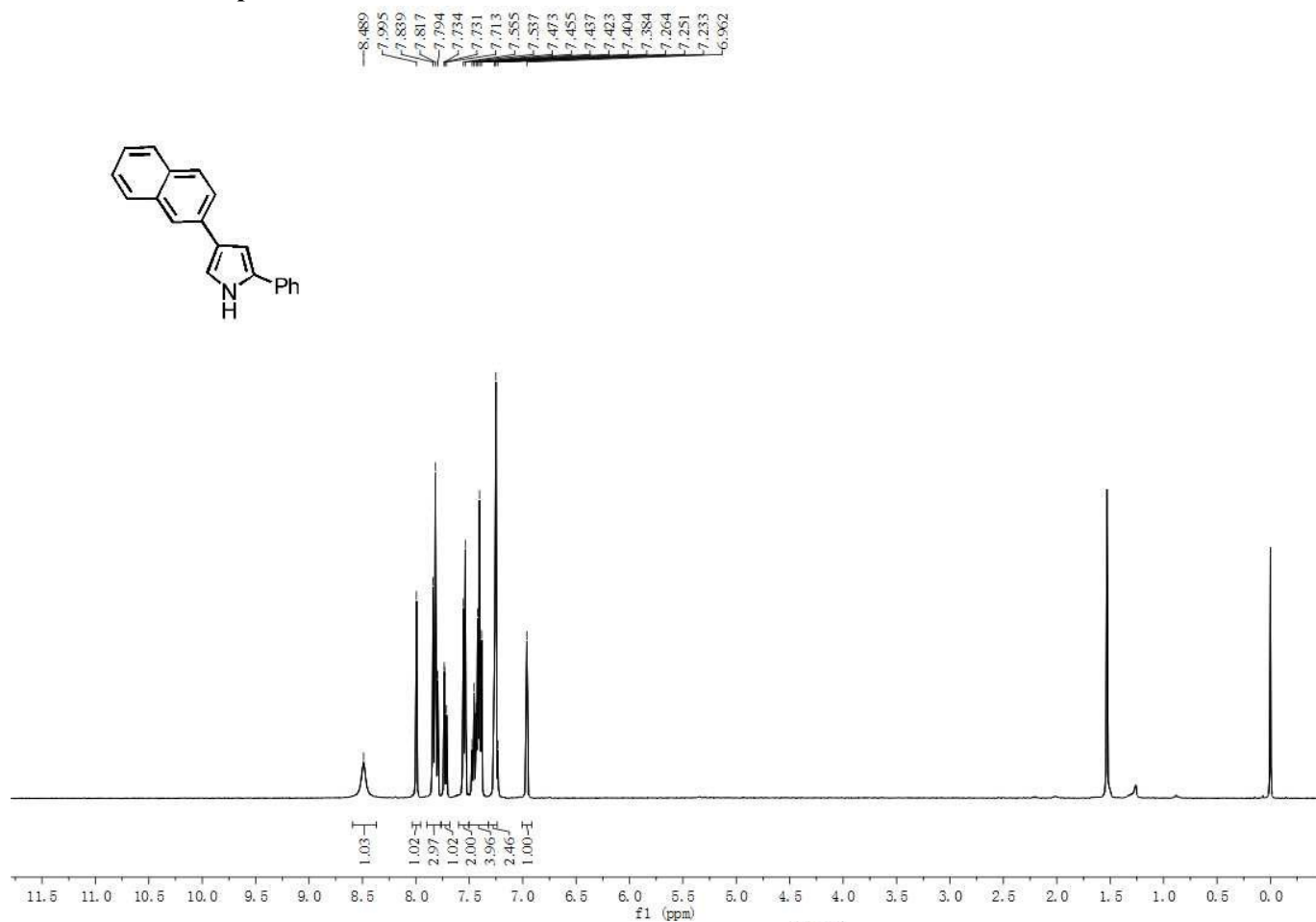
134.318
132.383
132.049
131.795
130.334
130.146
128.934
126.895
126.790
126.505
123.902
123.393
118.871
106.711
77.318 CDCl₃
77.000 CDCl₃
76.683 CDCl₃



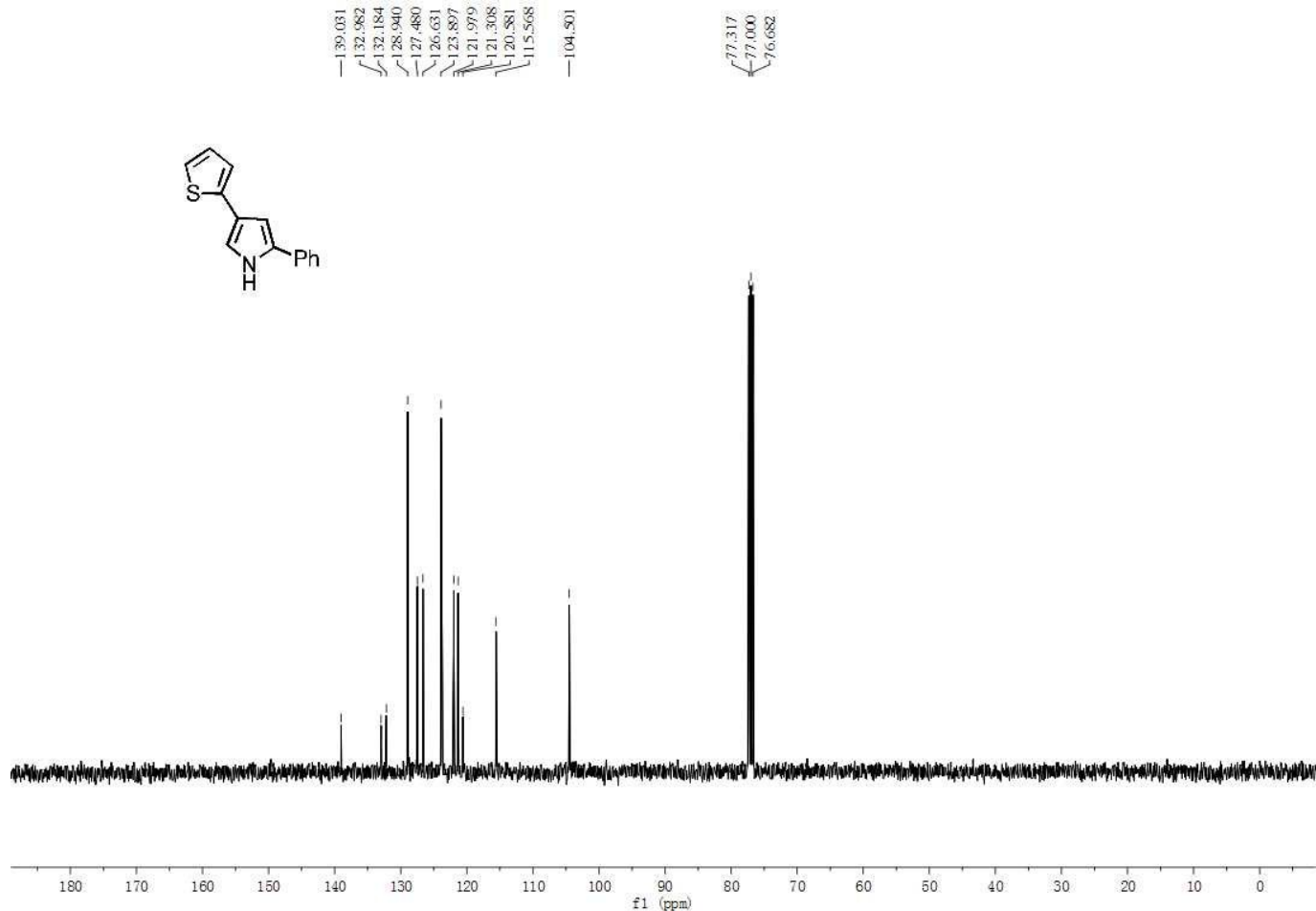
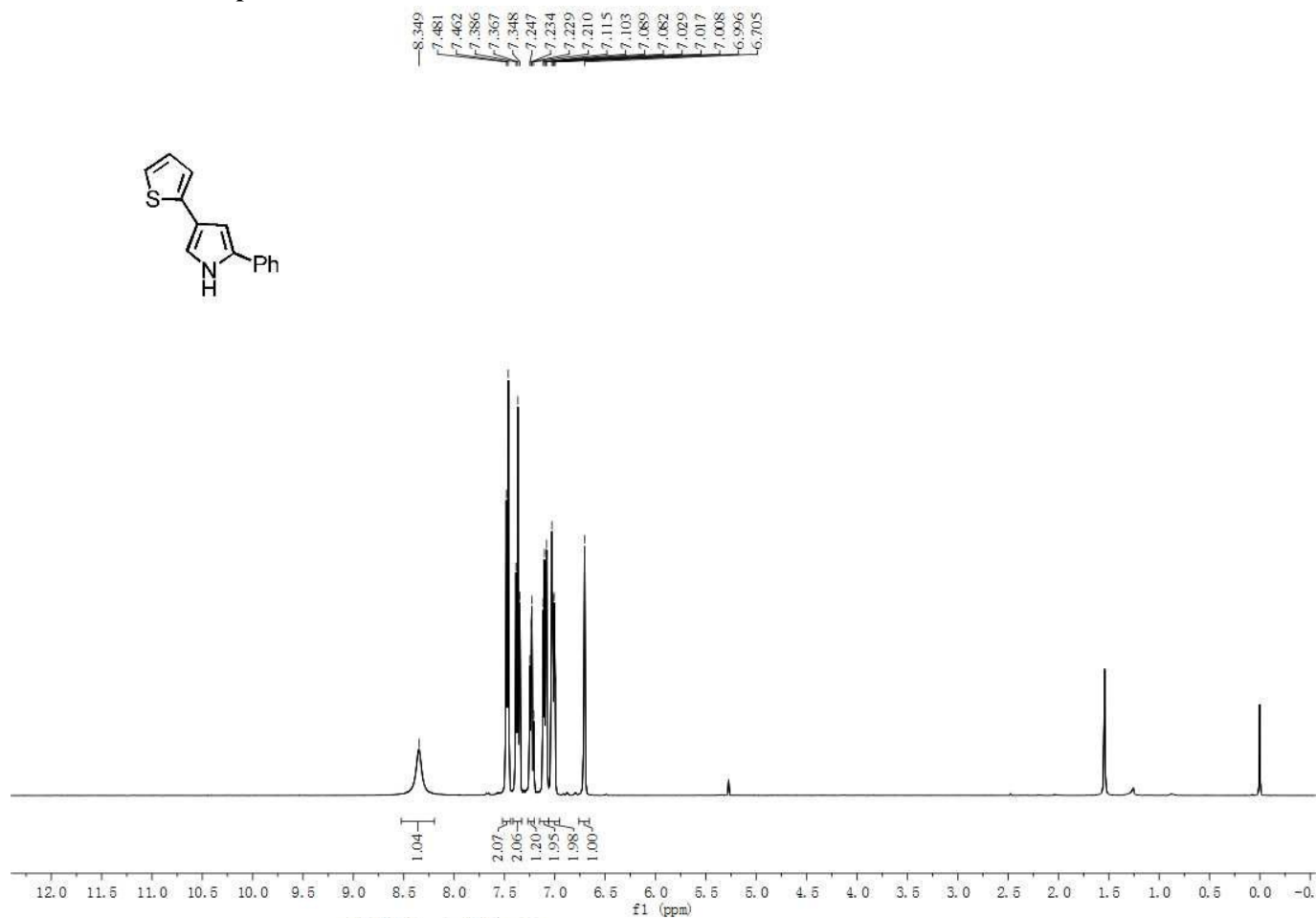
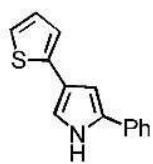
¹H and ¹³C NMR Spectra of 2m



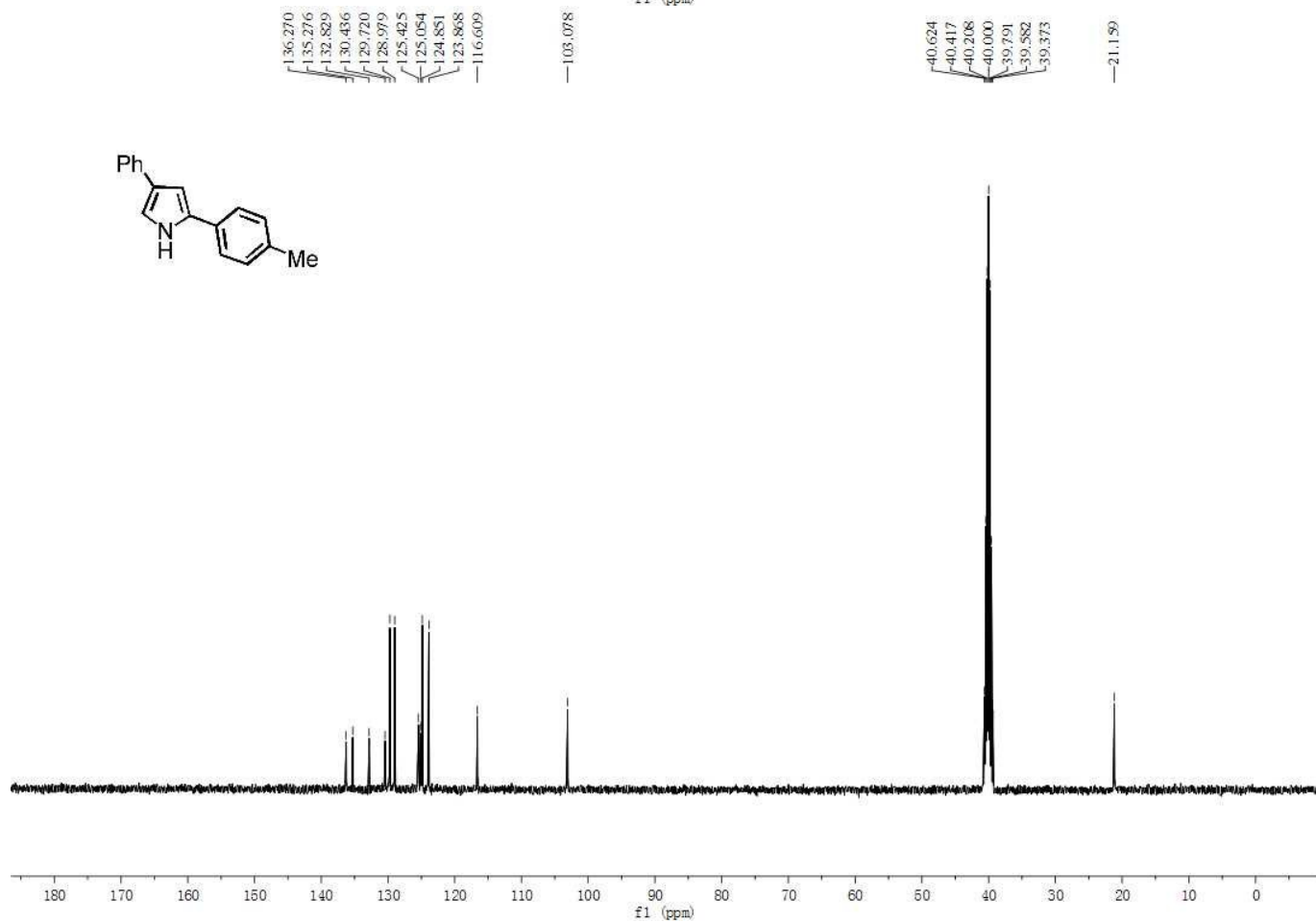
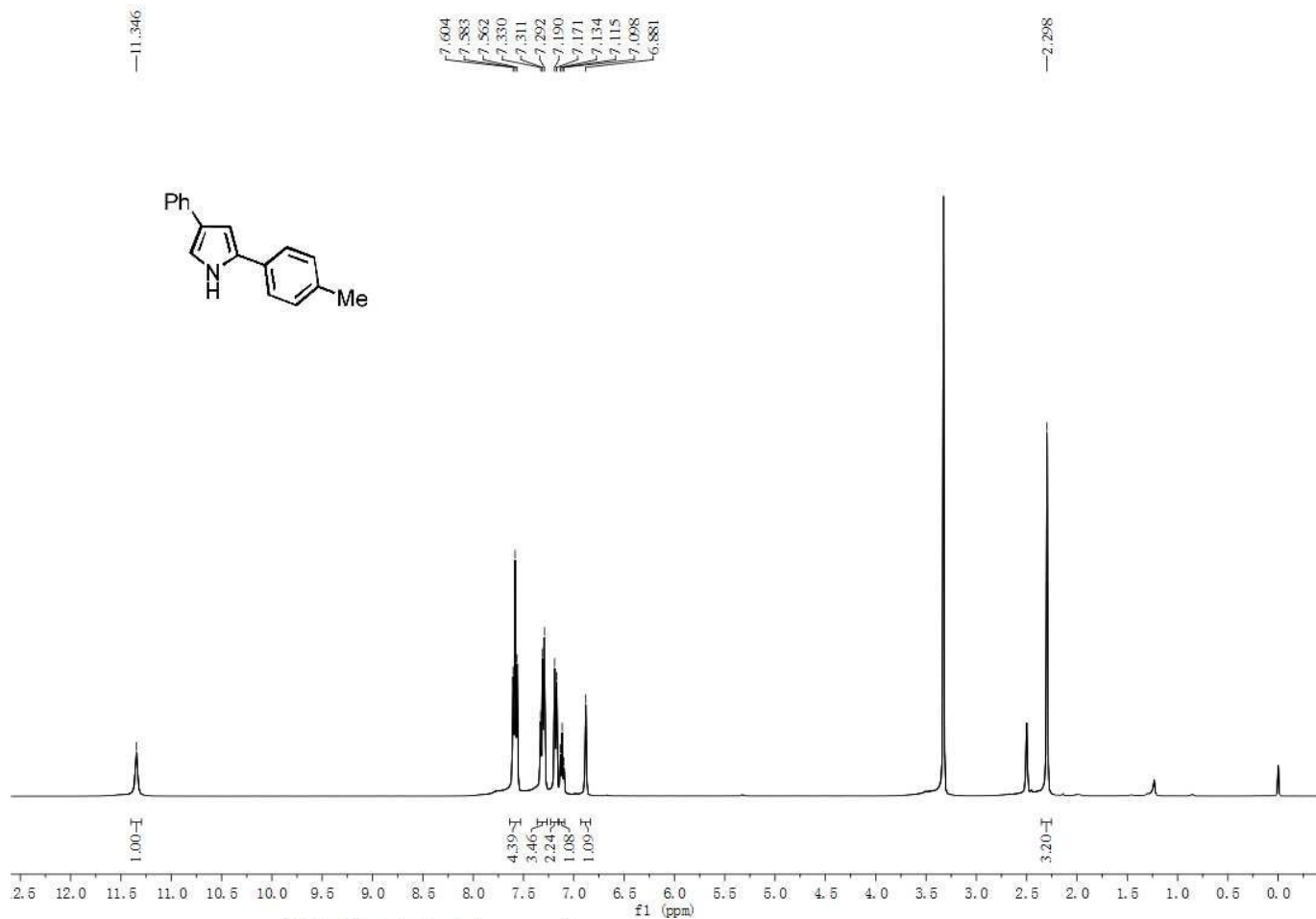
^1H and ^{13}C NMR Spectra of 2n



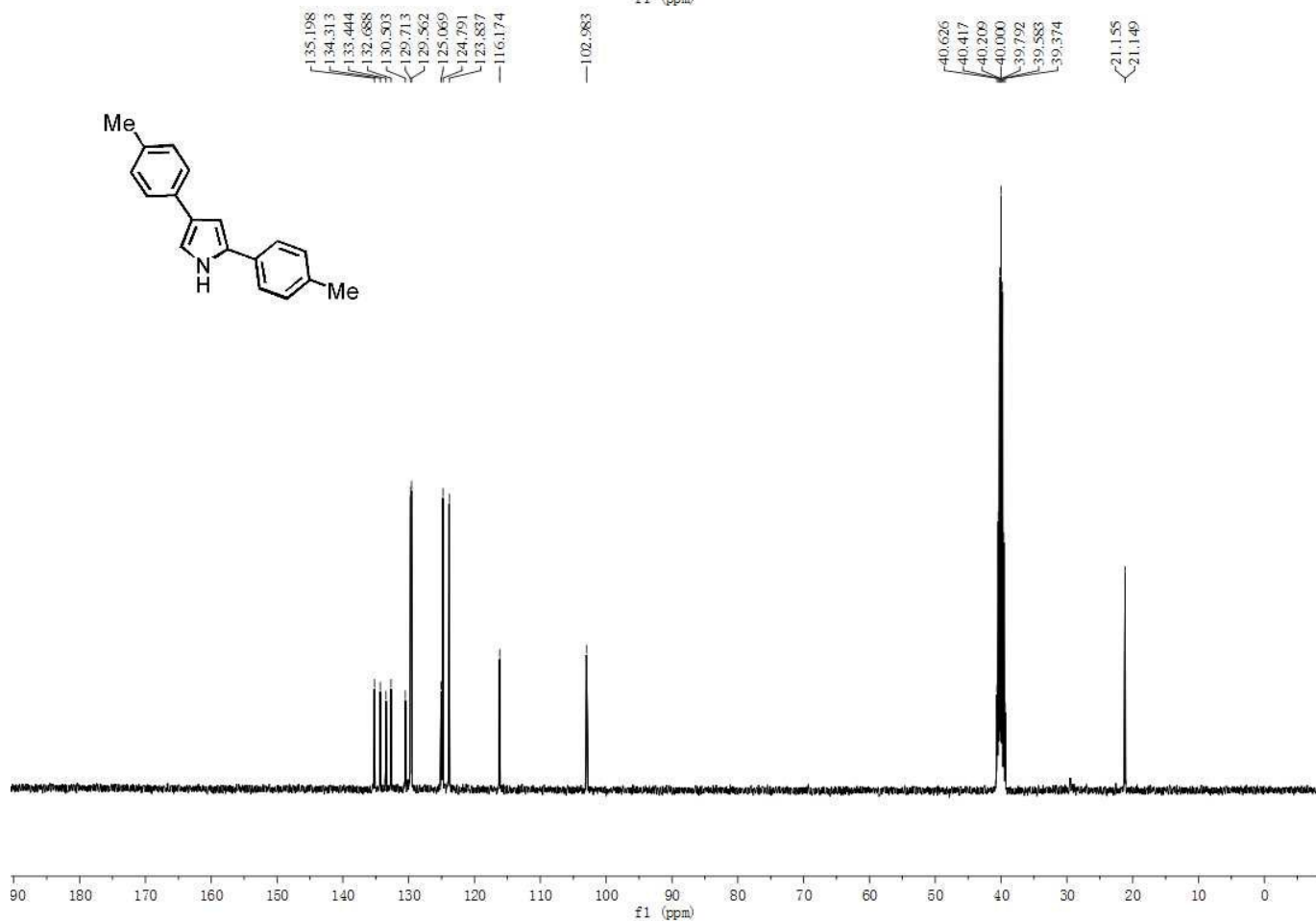
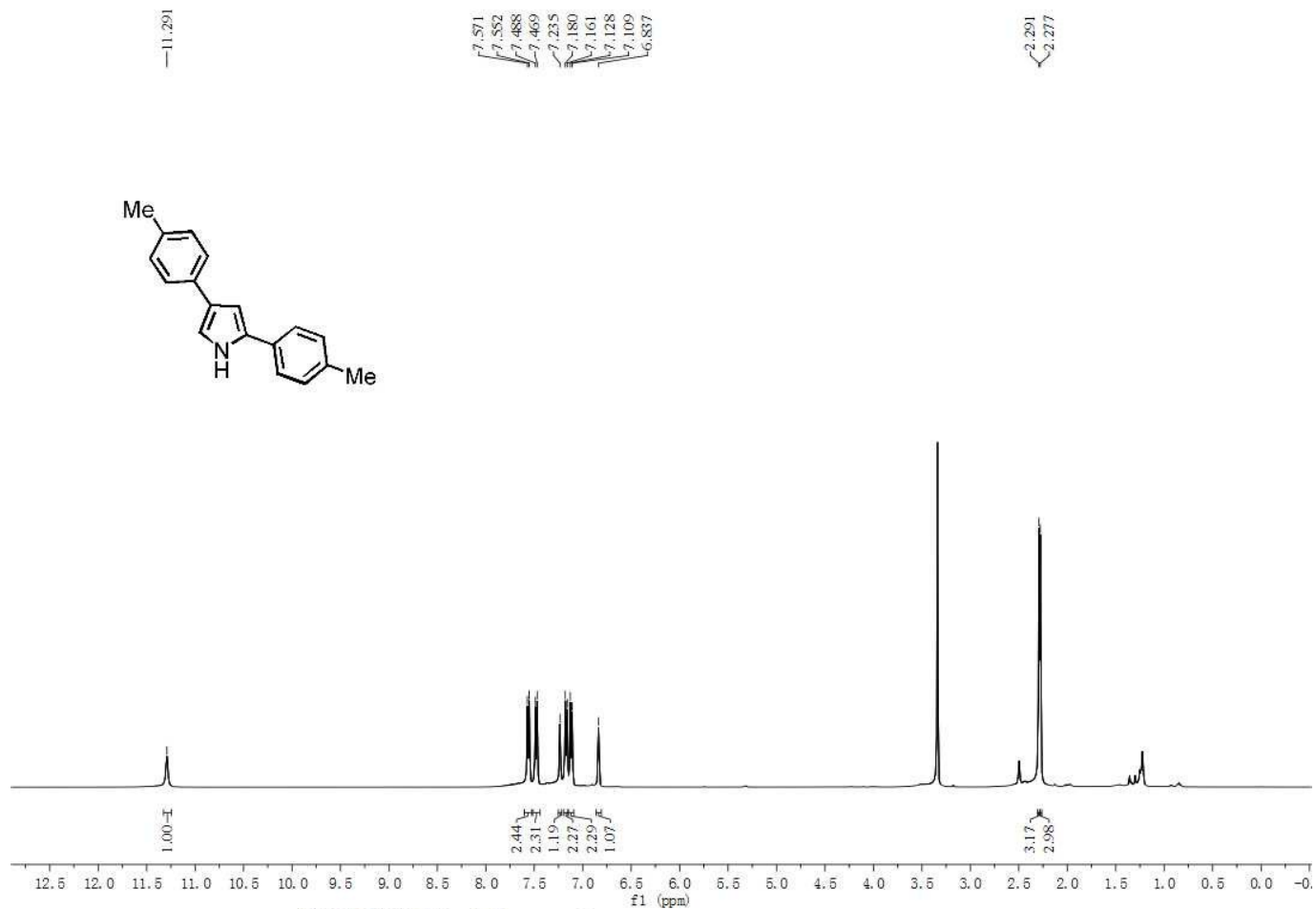
¹H and ¹³C NMR Spectra of 2o



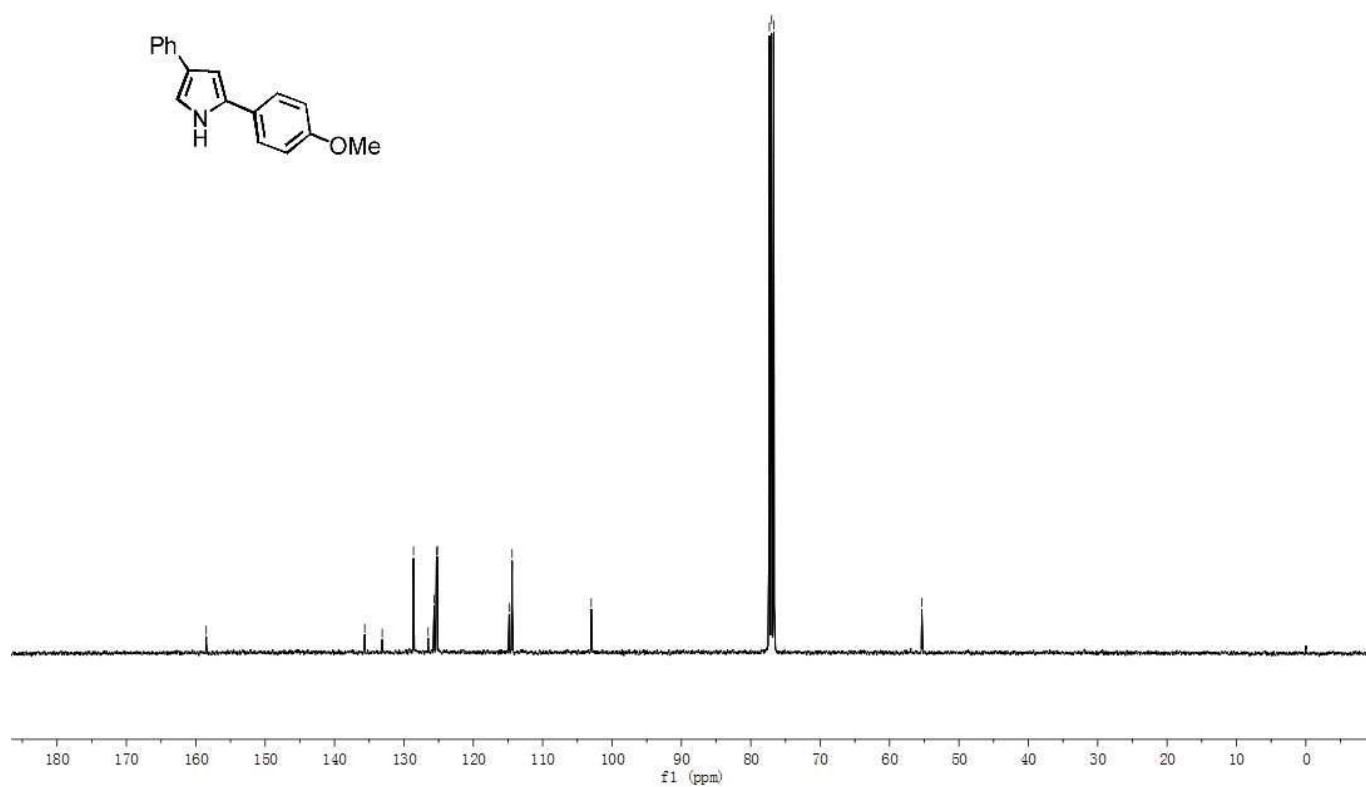
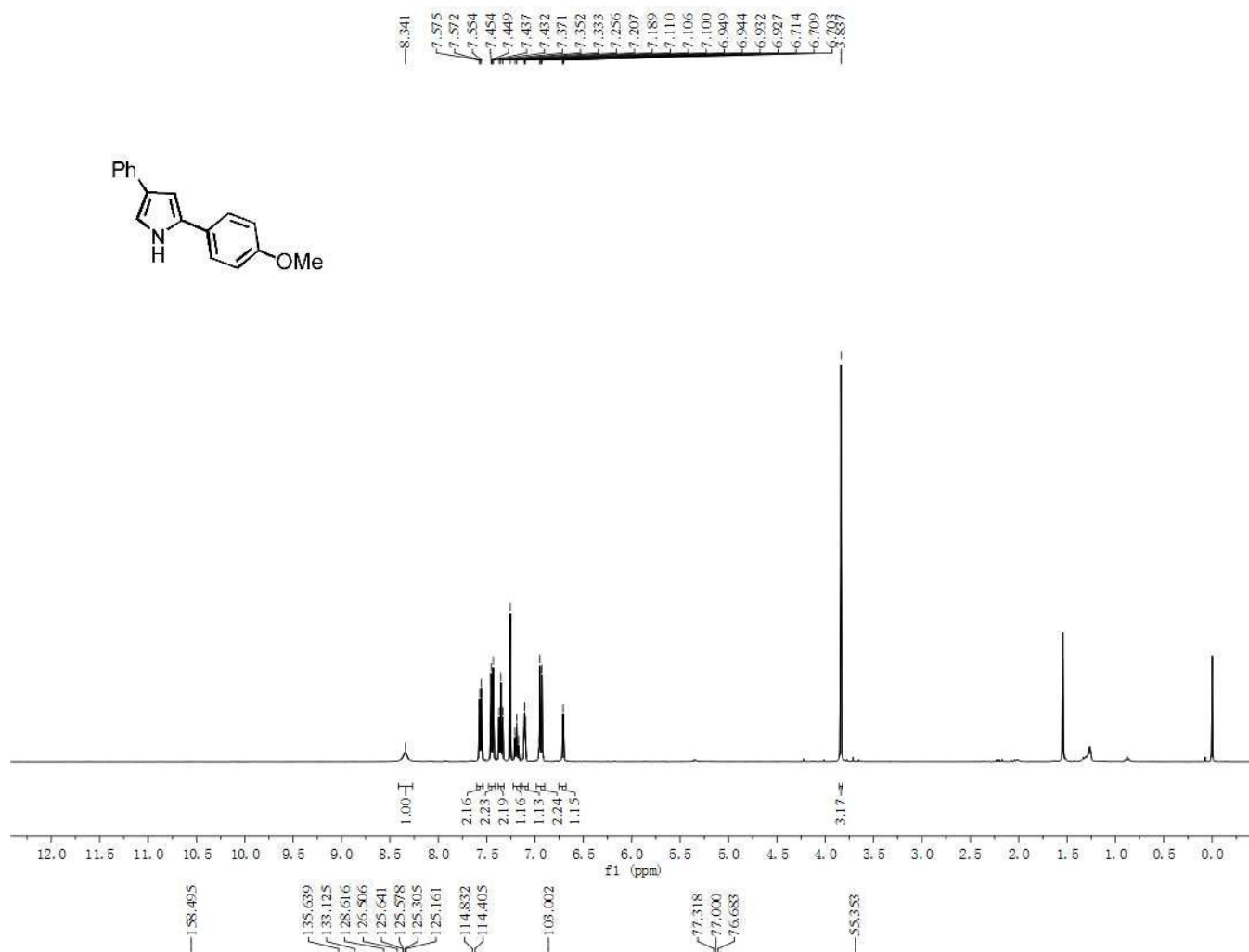
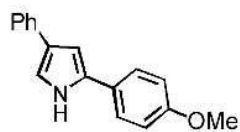
¹H and ¹³C NMR Spectra of 2p



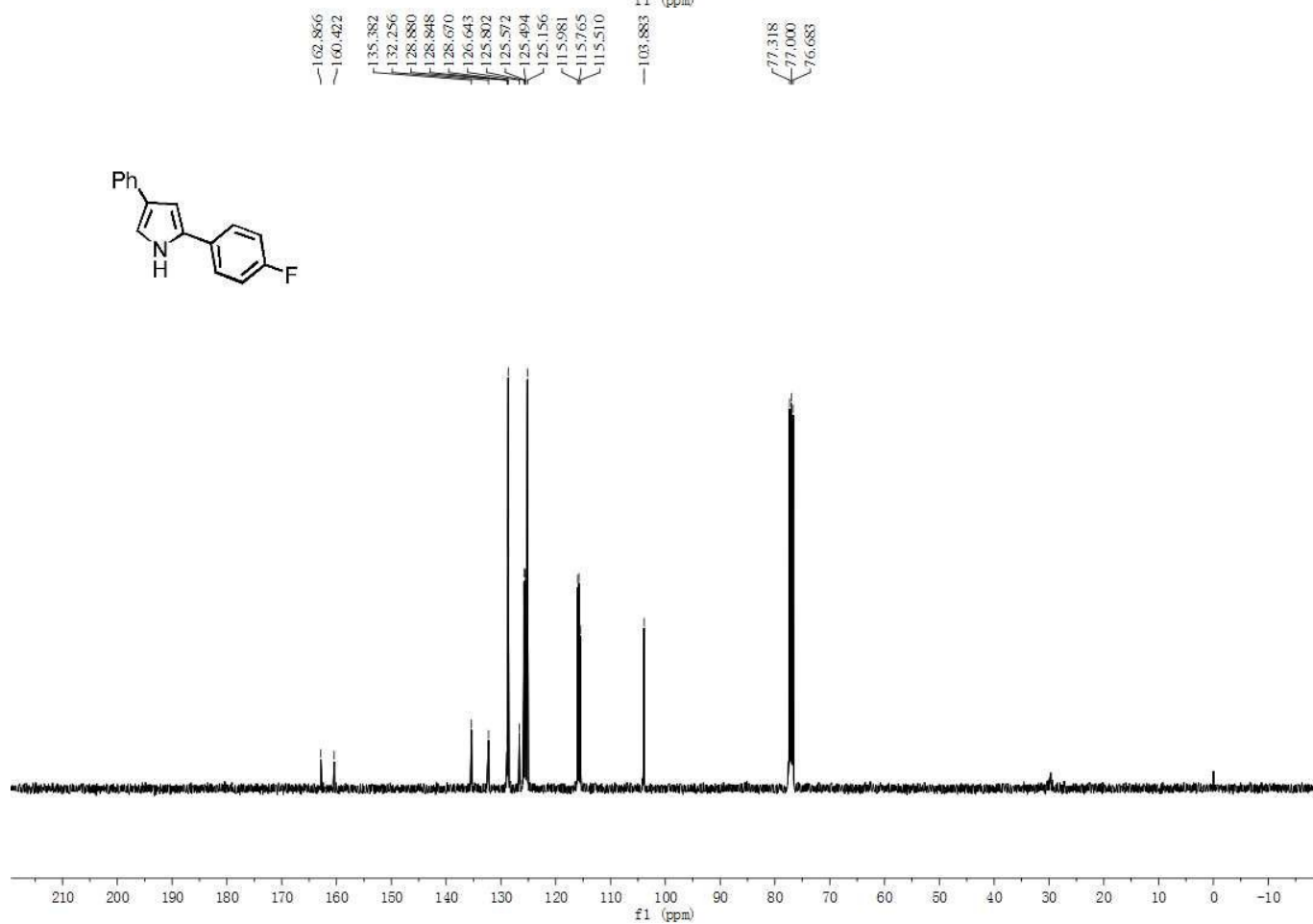
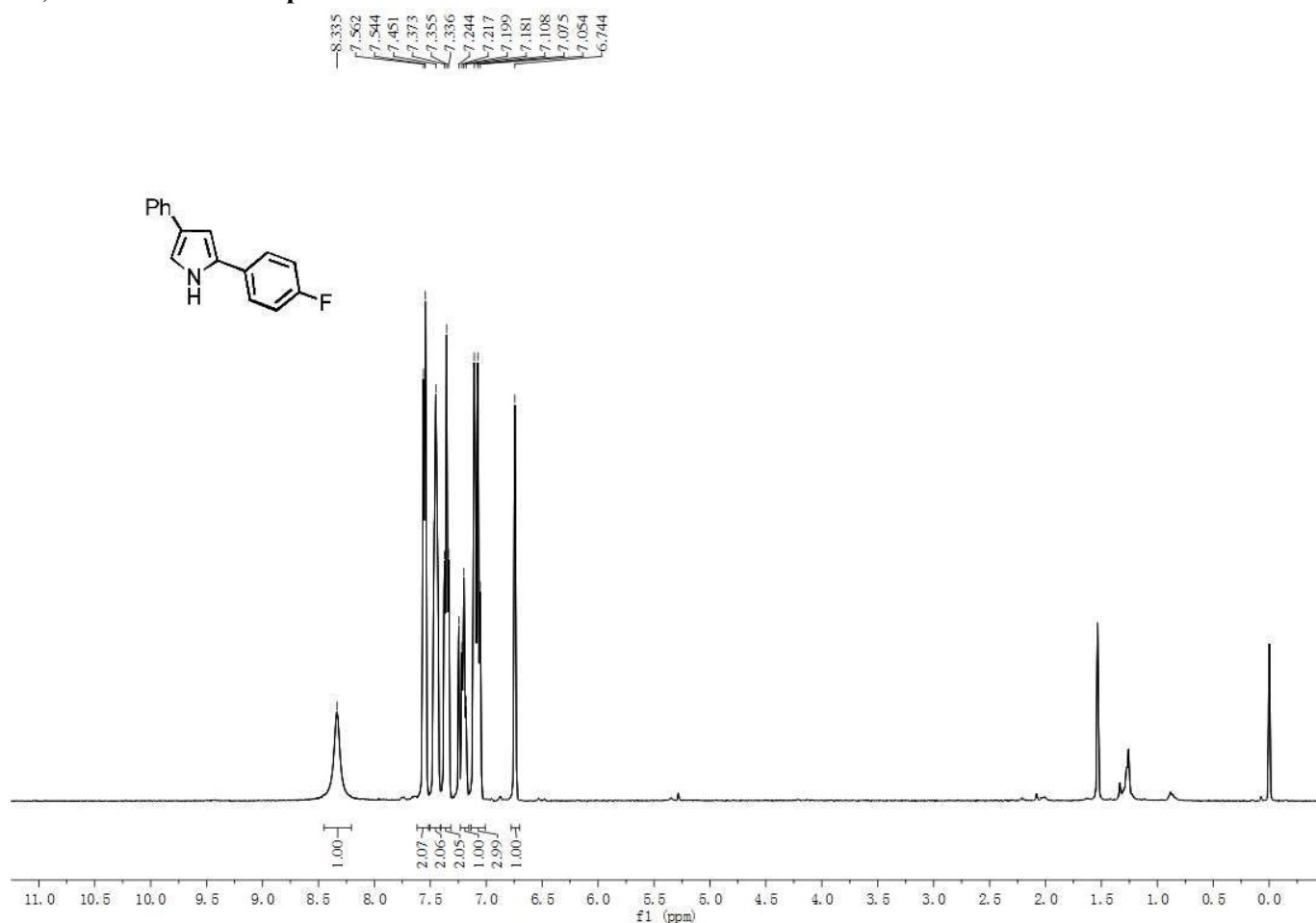
¹H and ¹³C NMR Spectra of 2q



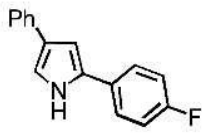
¹H and ¹³C NMR Spectra of 2r



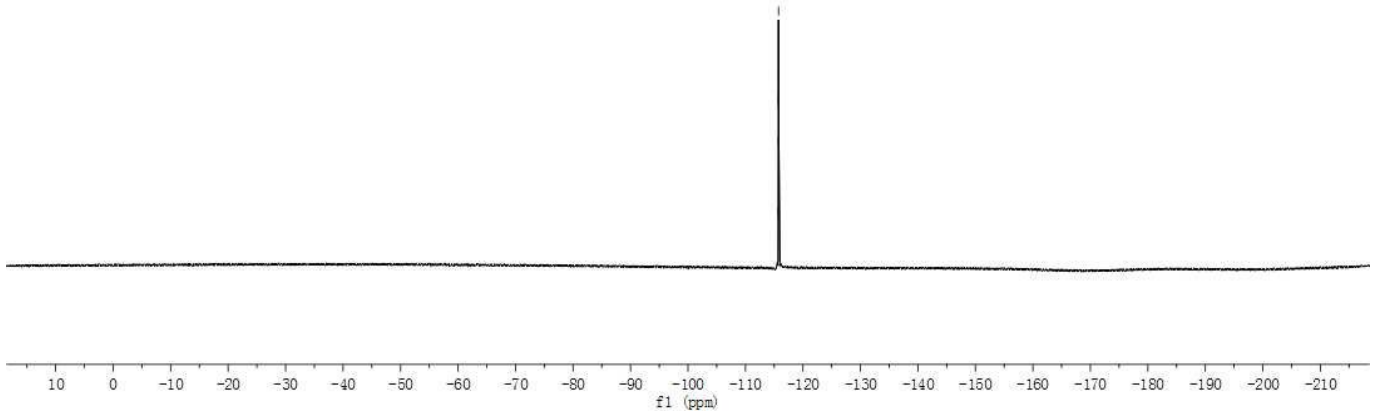
¹H, ¹³C and ¹⁹F NMR Spectra of 2s



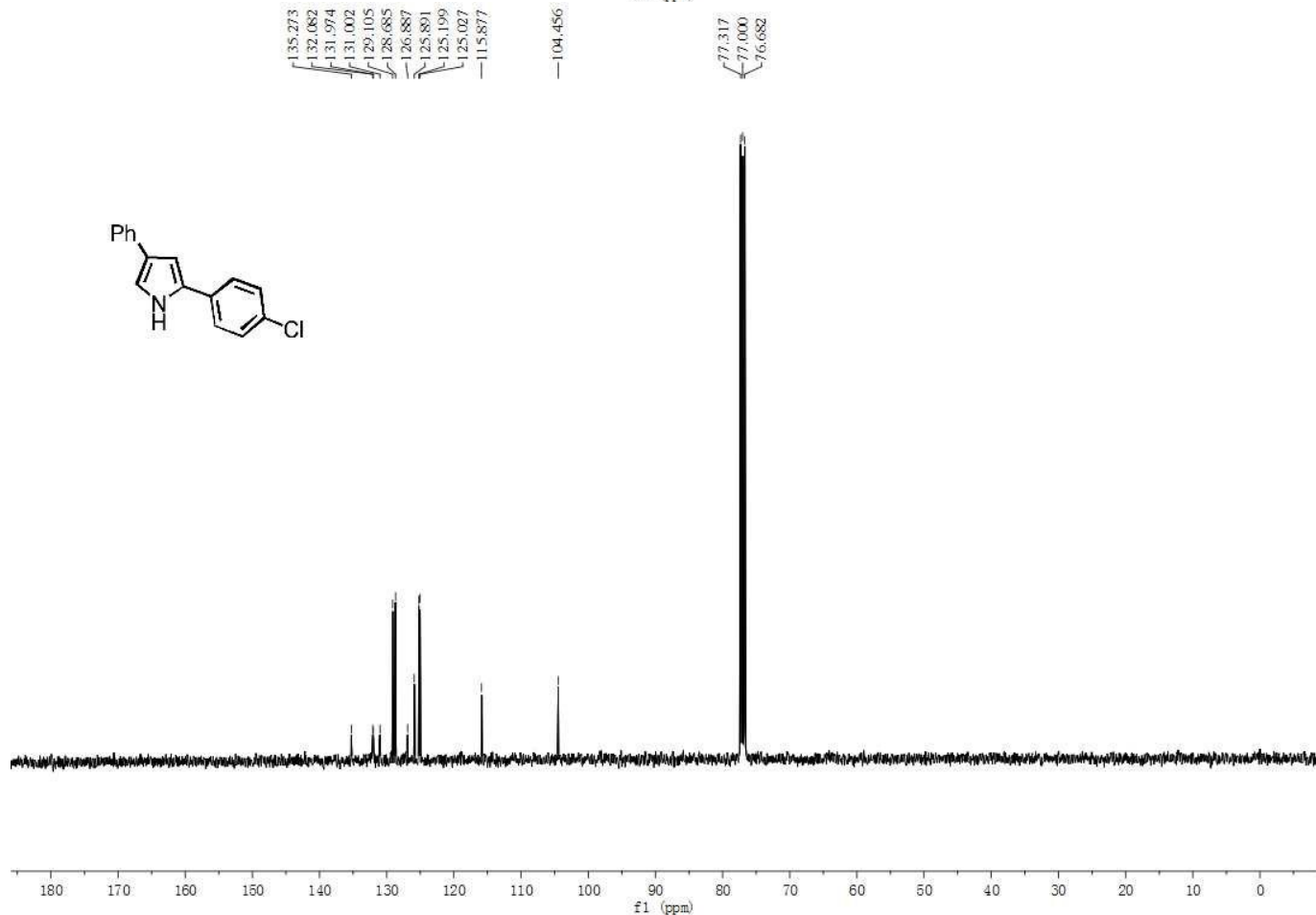
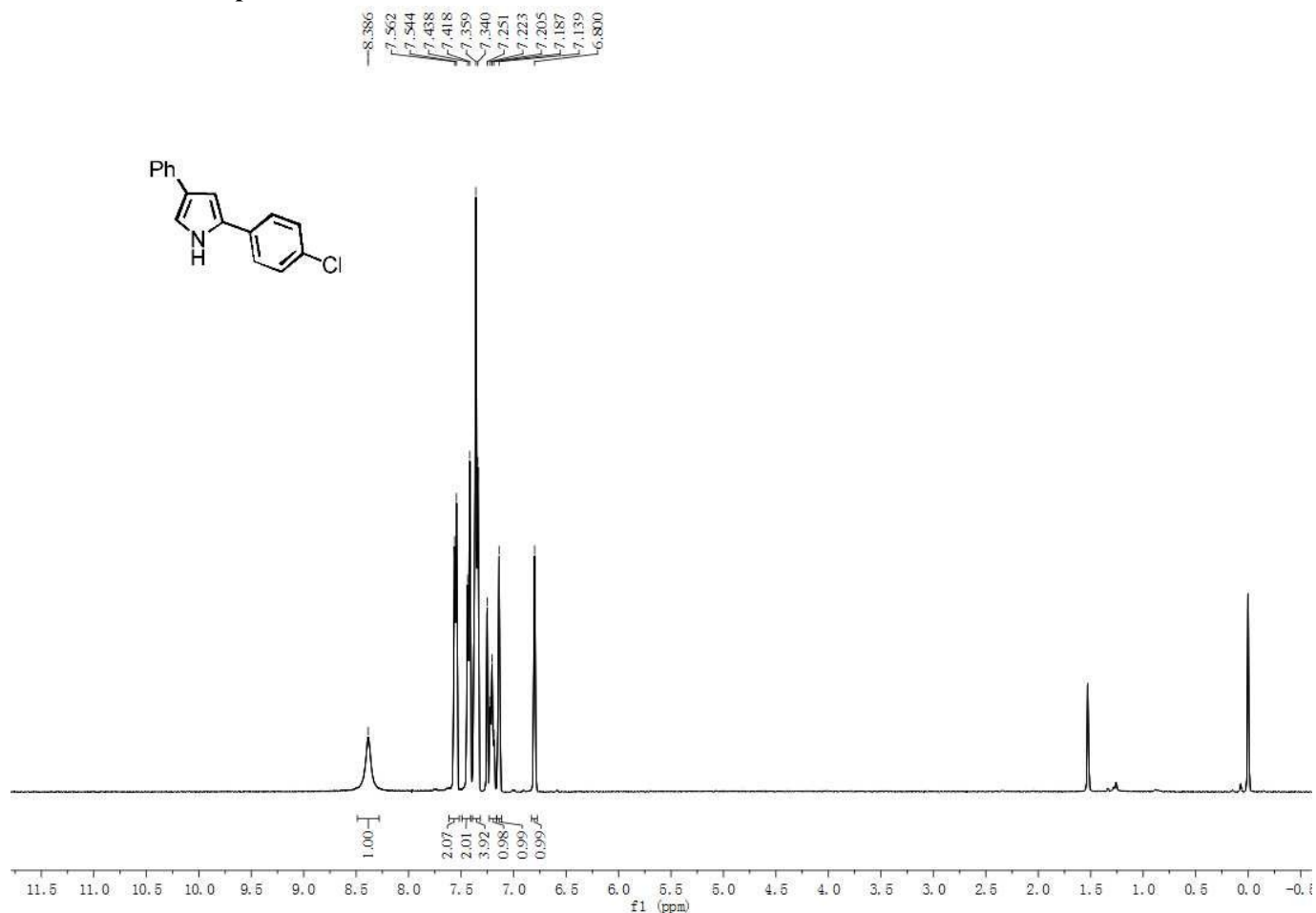
115.712
115.726
115.735
115.748
115.762
115.770
115.784



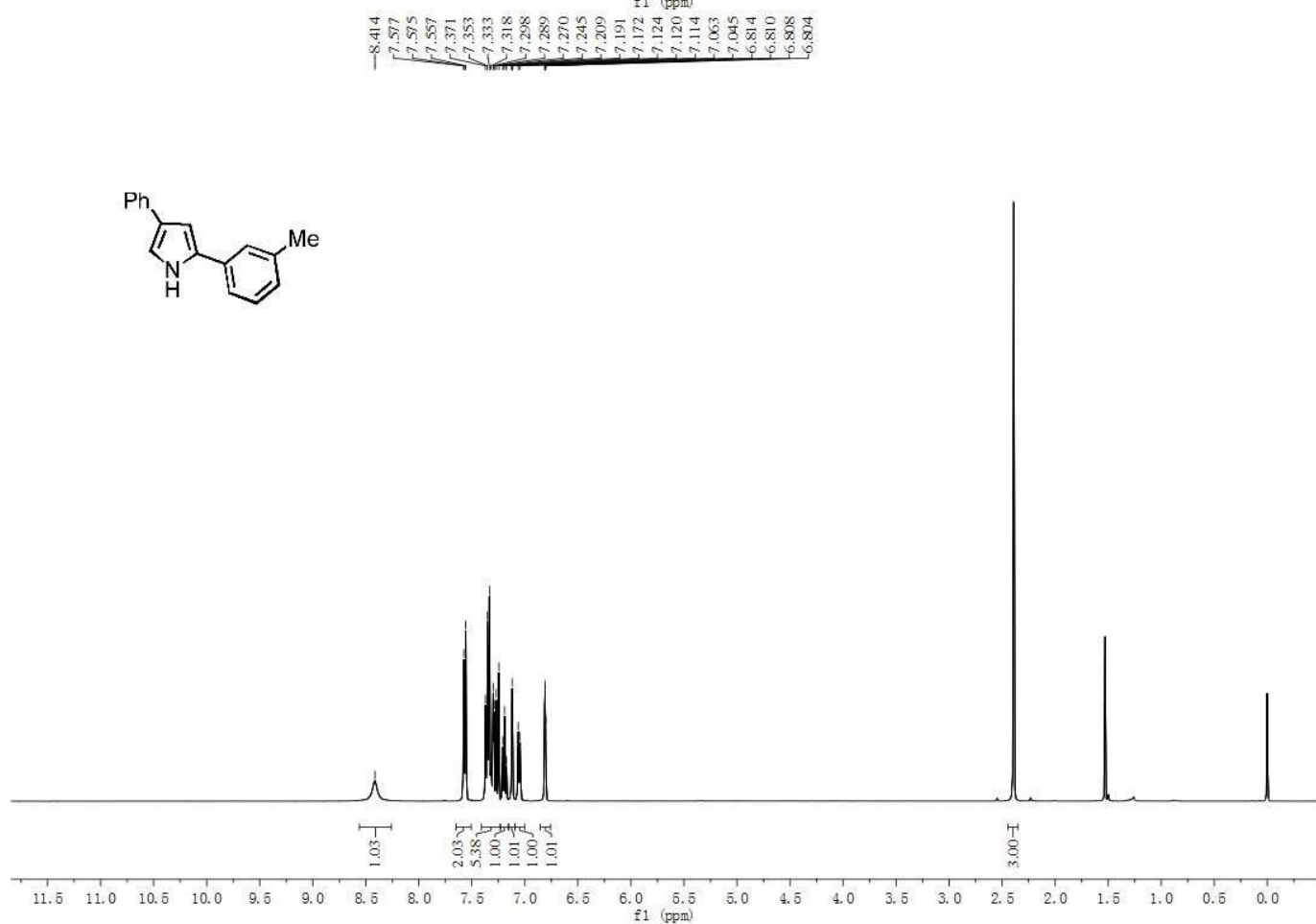
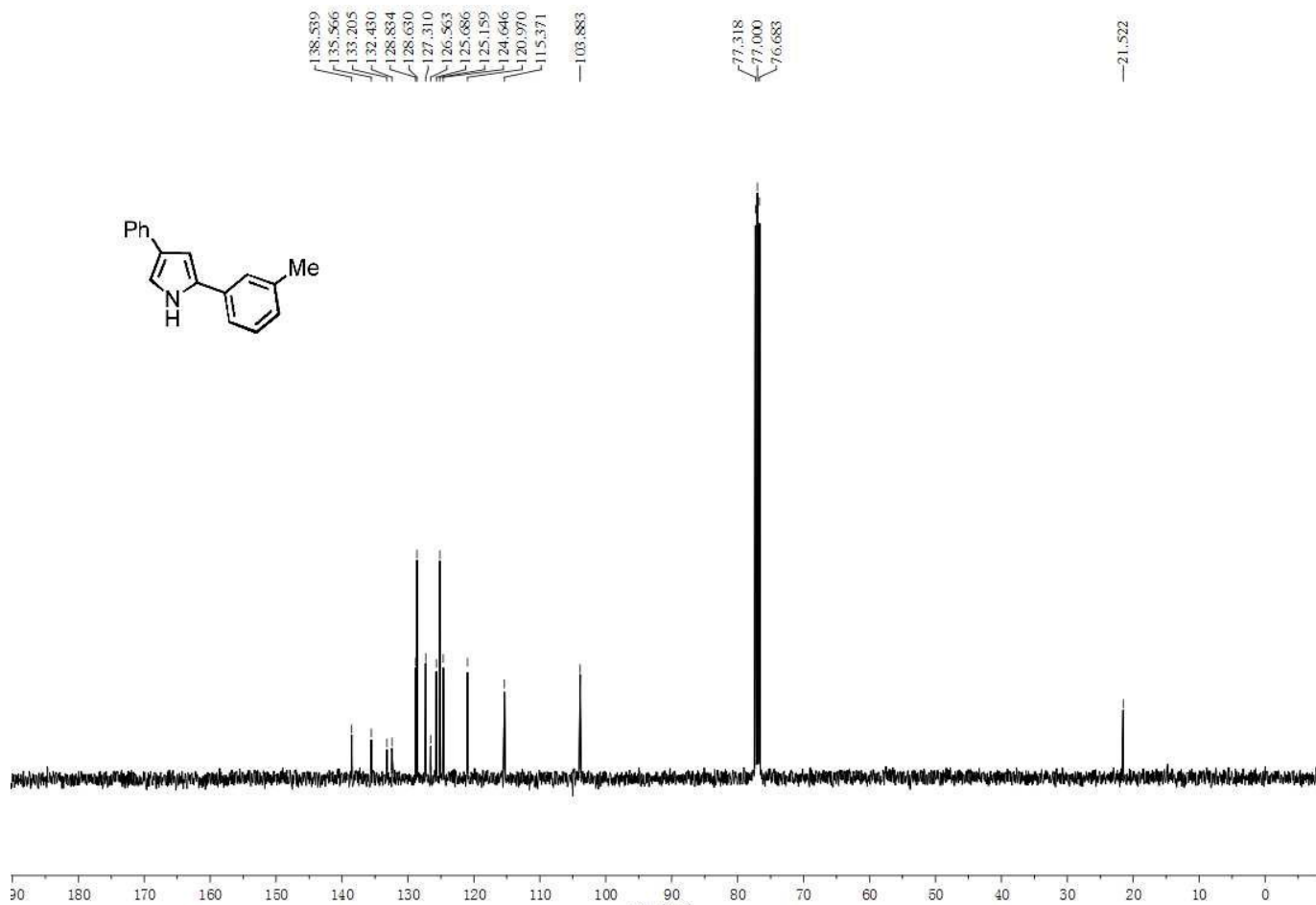
A (m)
-115.75



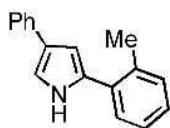
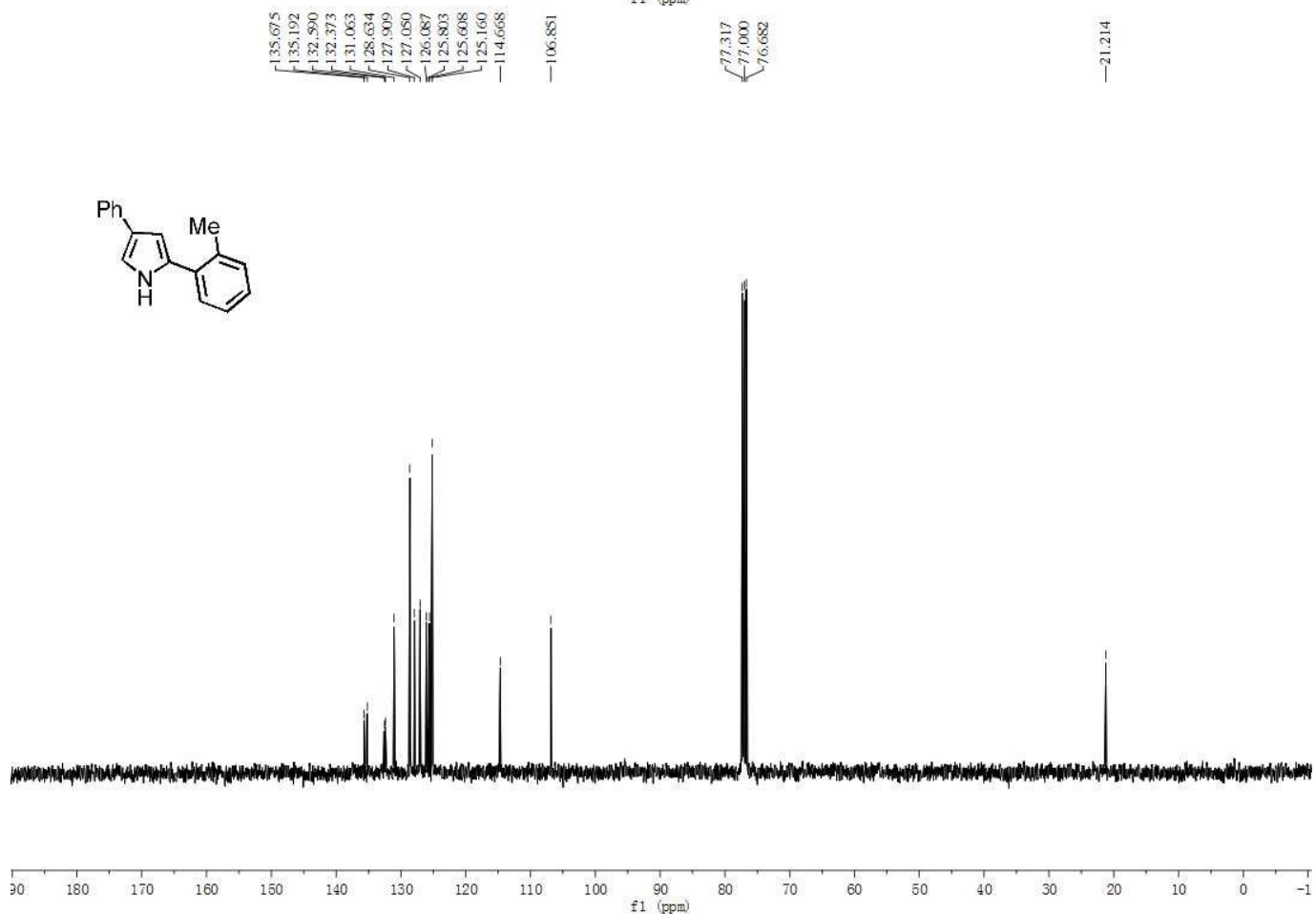
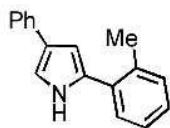
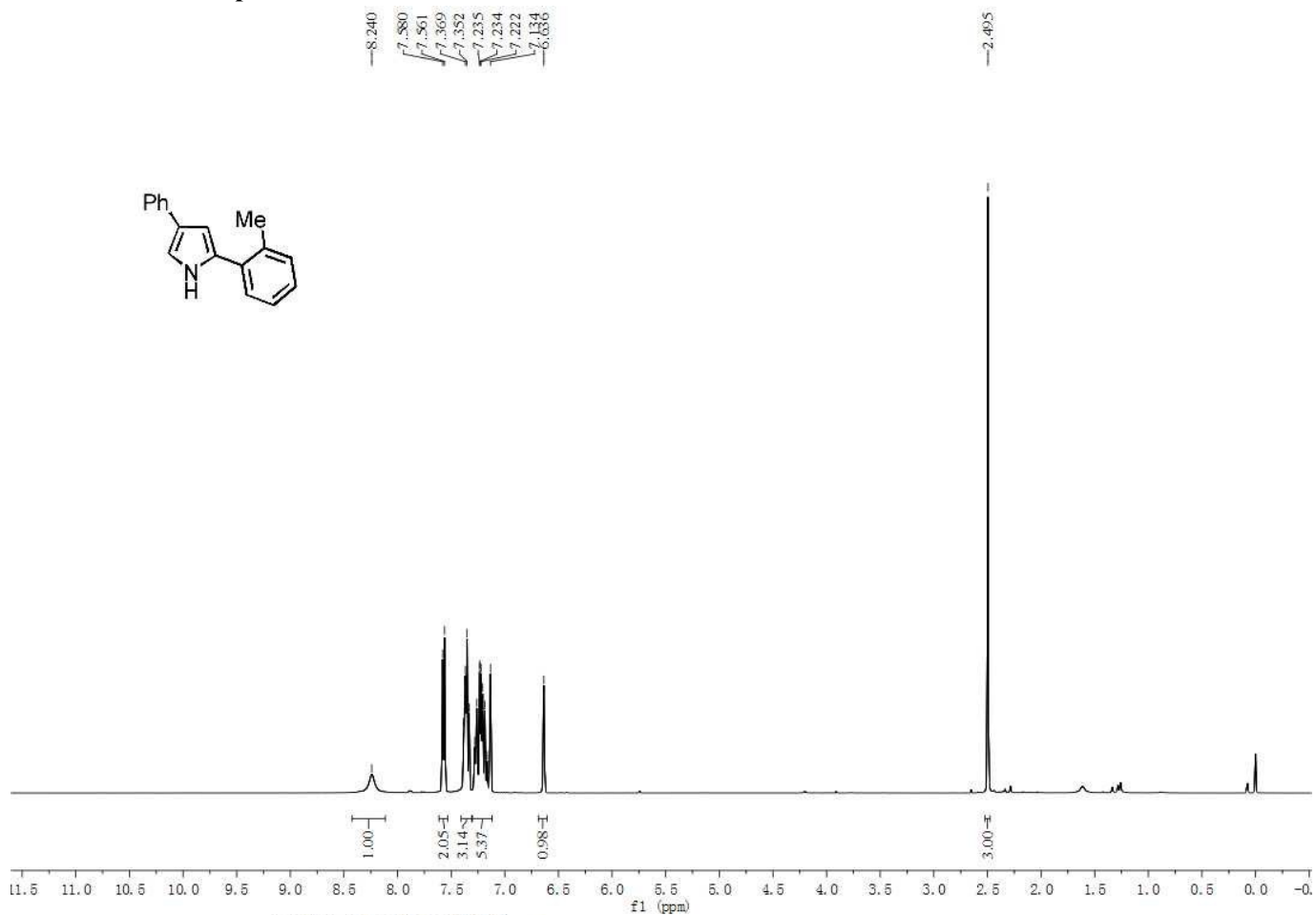
¹H and ¹³C NMR Spectra of 2t



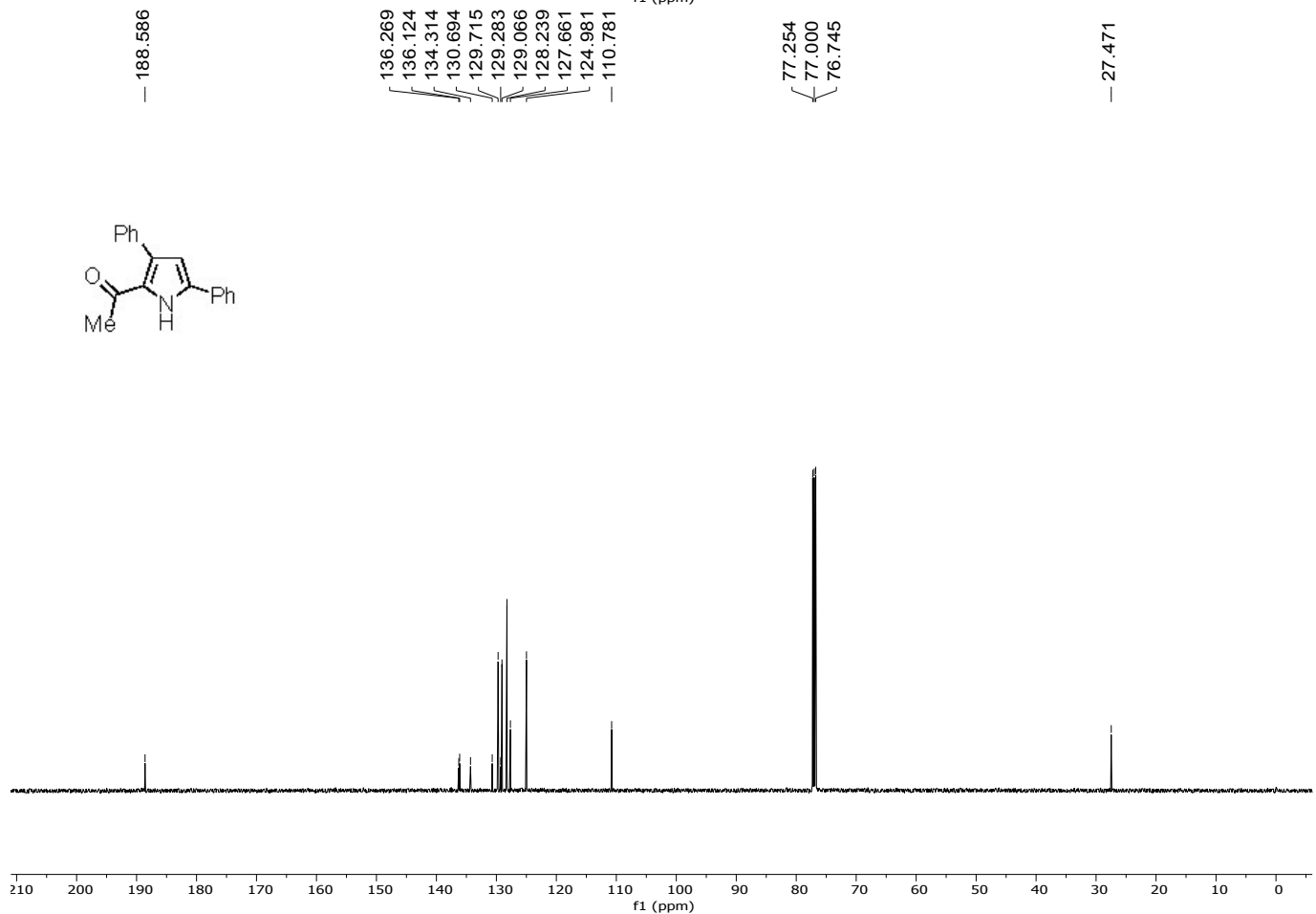
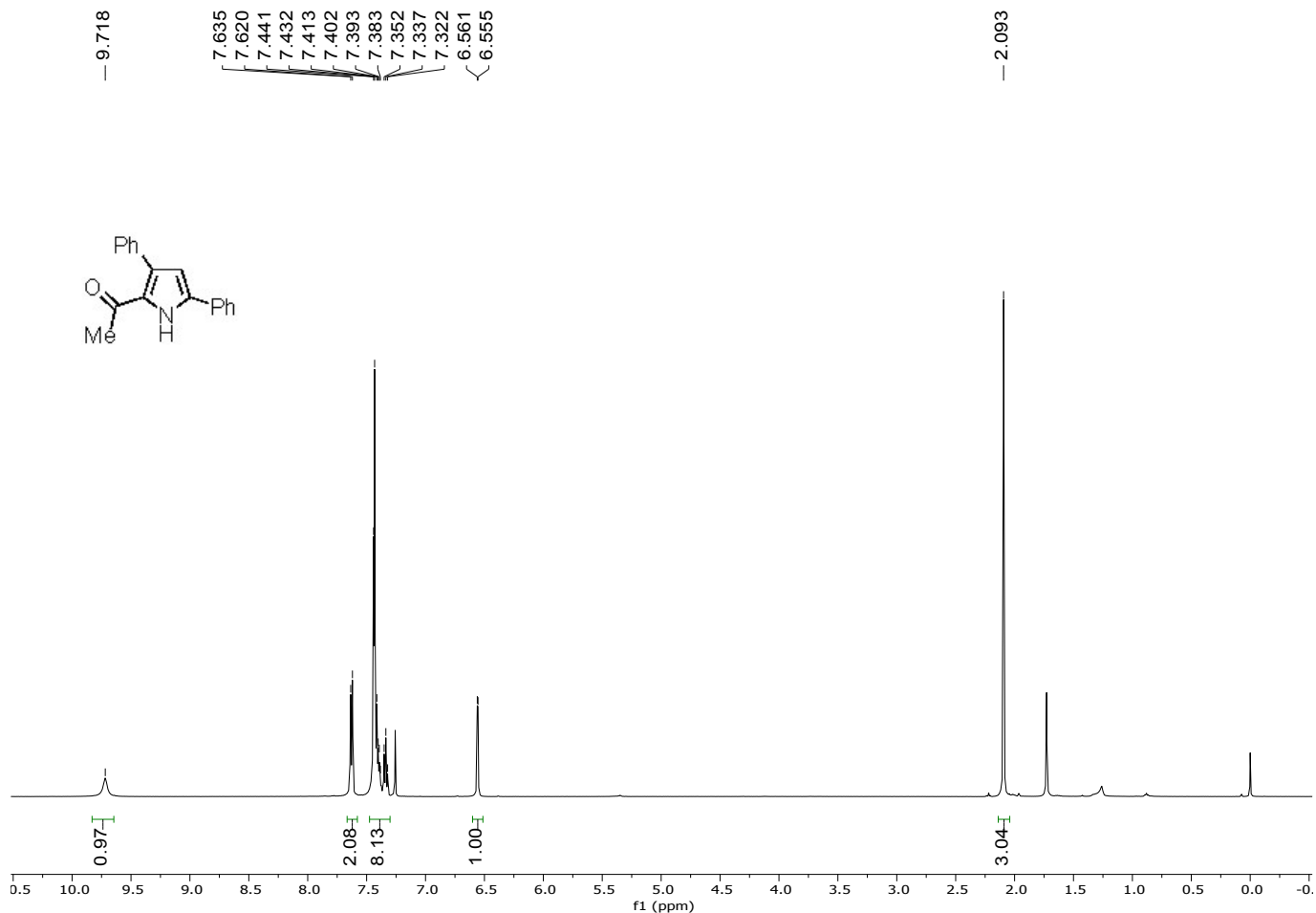
¹H and ¹³C NMR Spectra of 2u



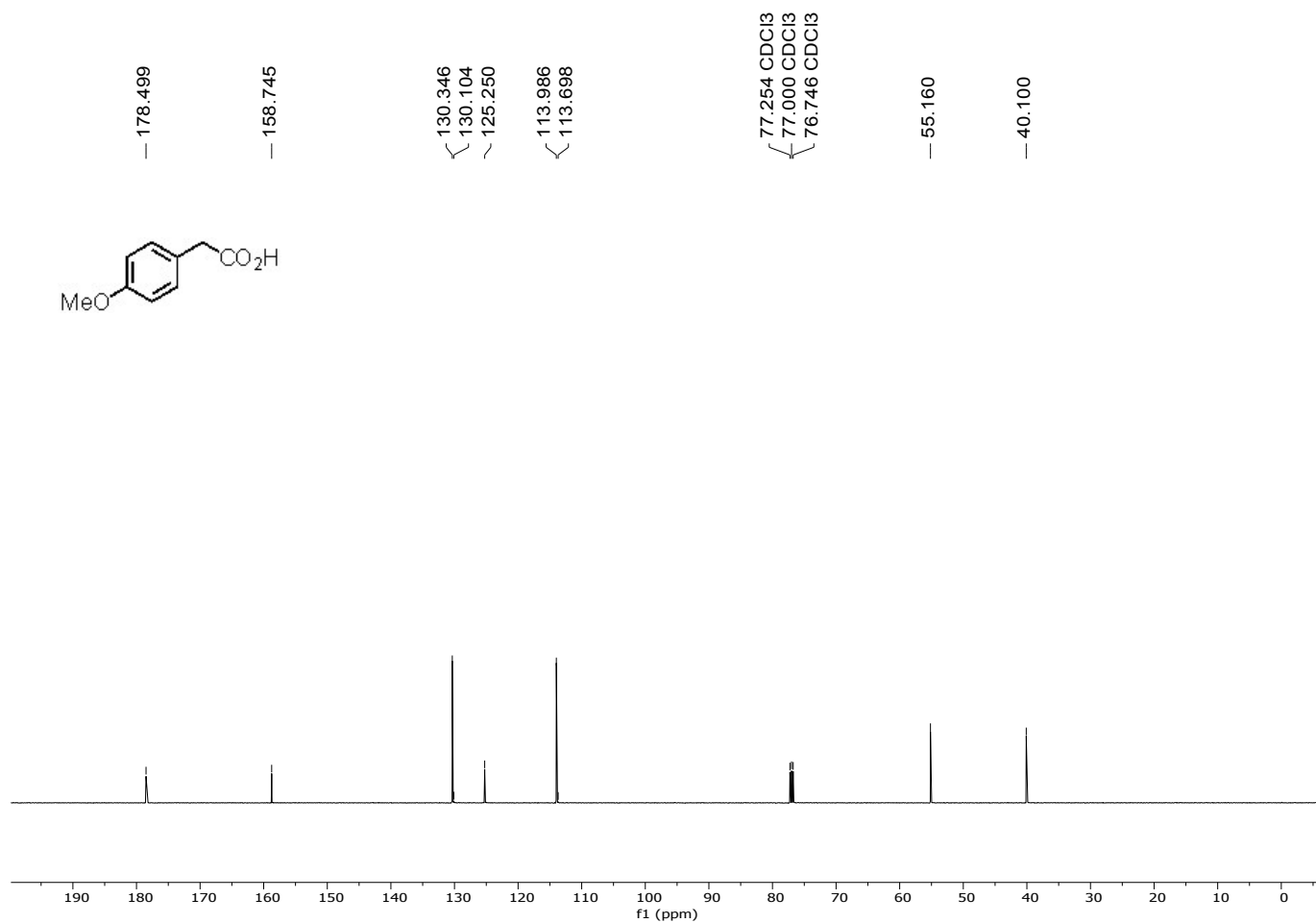
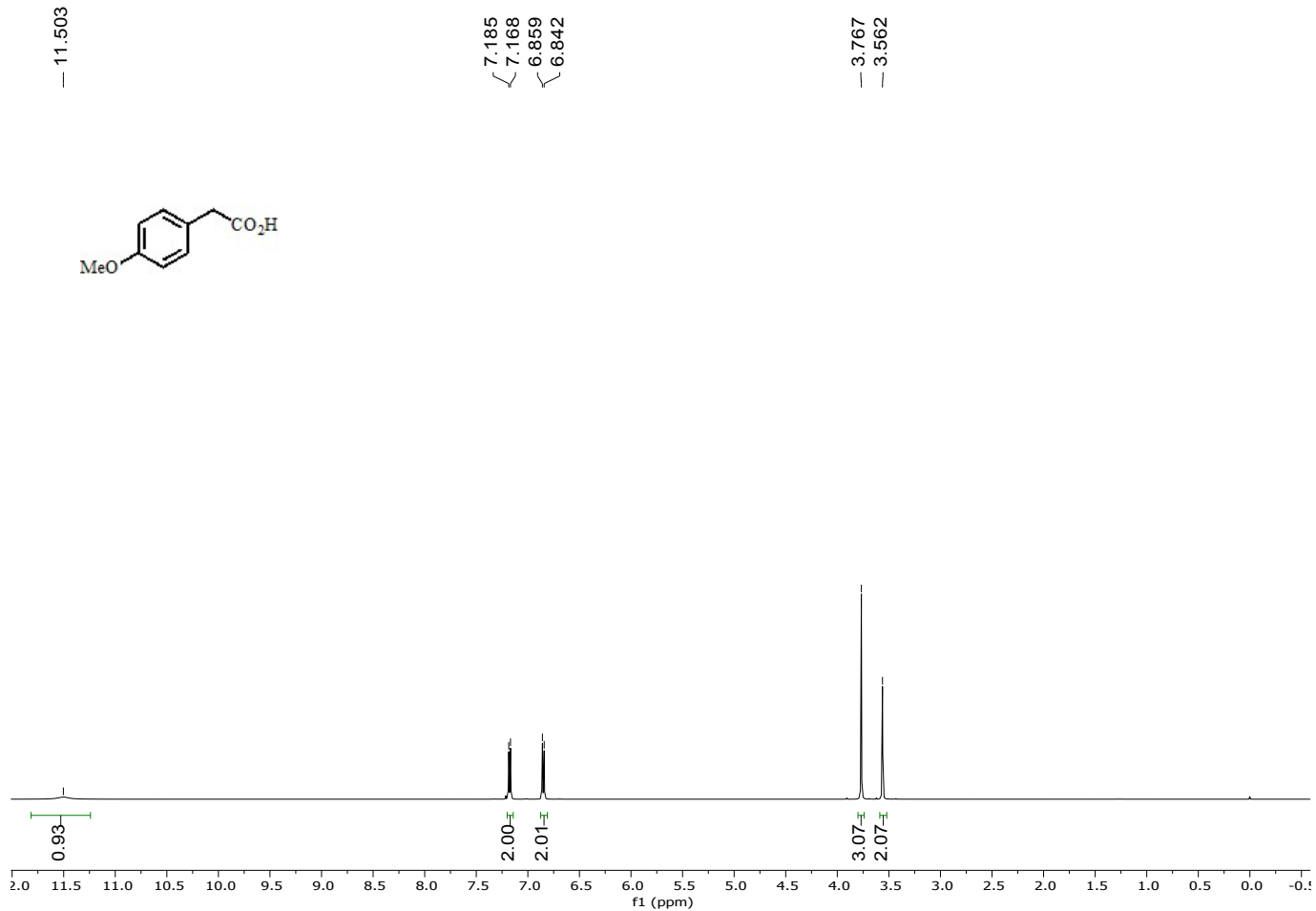
¹H and ¹³C NMR Spectra of 2v



¹H and ¹³C NMR Spectra of 3e



¹H and ¹³C NMR Spectra of 4



¹H and ¹³C NMR Spectra of 5

