Supporting Information

Lawesson's Reagent-Promoted Deoxygenation of γ-Hydroxylactams or Succinimides for the Syntheses of Substituted Pyrroles

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I. General Information

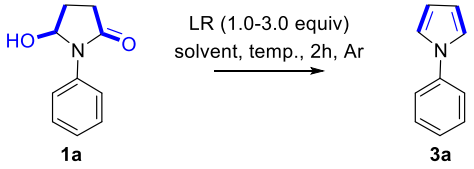
All reactions were carried out in a dry solvent under argon atmosphere, and all reagents were obtained from commercial suppliers and used without further purification, unless otherwise noted. All solvents were processed through the reference Purification of Laboratory Chemicals (Seventh Edition). External bath temperatures were used to record all reaction temperatures. Silica gel (300~400 mesh) and petroleum ether, EtOAc, CH_2Cl_2 and MeOH were used for product purification by flash column chromatography. NMR spectra were recorded on Bruker 300 MHz, 400 MHz or 600 MHz spectrometers. Proton chemical shifts are reported relative to internal standard TMS at δ 0.0 ppm or residual solvent peak ($CDCl_3$ at 7.26 ppm, methanol-d_4 at 3.31 ppm, acetone-d_6 at 2.05 ppm, DMSO-d_6 at 2.50 ppm). Carbon chemical shifts are reported relative to a residual solvent peak ($CDCl_3$ at 77.06 ppm, methanol-d_4 at 49.03 ppm, acetone-d_6 at 29.82 ppm, DMSO-d_6 at 39.53 ppm). The following abbreviations were used to designate multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet, br = broad. Fourier transform infrared spectra (FT-) were recorded on an Agilent Cary 630 FT-IR instrument. LC-MS spectra were recorded on an Agilent Technologies 1260II-MSD 6125 Quotation with an Agilent HC-C18(2) column (4.6 mm x 250 mm, film: 5 μm). High-resolution mass spectra (HRMS) were measured on a Brucker Daltonics Apex II 47e Specification (for HRMS).

II. Experimental Section and Characterization Data for Synthesized Compounds

i. Optimization of the reaction conditions.[a]

Procedure: A Schlenk tube was charged with a mixture of γ-hydroxylactam **1a** (0.1 mmol, 17.7 mg) and Lawesson's reagent in indicated solvent under an argon atmosphere. The vial was sealed and stirred vigorously at indicated temperature. After 2 h, the reaction was stopped by rapid cooling. Then the solvent was removed under reduced pressure, and the residue was directly subjected to column chromatography.

Table S1. Optimization of the reaction conditions.[a]



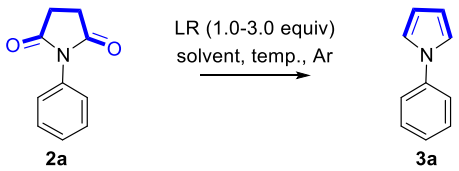
1a **3a**

Entry	Thiolation reagent (equiv)	Solvent	Temp. (°C)	Isolated yield
1	LR (1.0)	mesitylene	60	20%
2	LR (1.0)	toluene	60	45%
3	LR (1.0)	DCM	60	40%
4	LR (1.0)	DCE	60	32%
5	LR (1.0)	dioxane	60	35%
6	LR (1.0)	THF	60	37%
7	LR (1.0)	MeCN	60	18%
8	LR (1.0)	DMSO	60	N. D.[b]
9	LR (1.0)	MeOH	60	N. D.[b]
10	LR (1.0)	toluene	80	51%
11	LR (1.0)	toluene	90	54%
12	LR (1.0)	toluene	100	54%
13	LR (1.0)	toluene	110	62%
14	LR (0.5)	toluene	110	61%
15	LR (1.5)	toluene	110	71%
16	LR (2.0)	toluene	110	73%
17	LR (3.0)	toluene	110	21%
18	-	toluene	110	N. R.[c]
19	P_2S_5/HMDO[d]	toluene	110	N. D.[b]

[a.] Reaction conditions: **1a** (0.1 mmol), thiolation reagent (1.0 – 3.0 equiv), solvent (1.0 mL), temperature (60 – 110 °C), 2h, Ar. [b.] No product was detected. [c.] No reaction happened, and **1a** was recovered in 80% yield. [d.] P_2S_5 (0.3 equiv), HMDO (1.7 equiv). HMDO = hexamethyldisiloxane.

Procedure: A Schlenk tube was charged with a mixture of succinimide **2a** (0.1 mmol, 17.5 mg) and Lawesson's reagent in indicated solvent under an argon atmosphere. The vial was sealed and stirred vigorously at indicated temperature. After several hours, the reaction was stopped by rapid cooling. Then the solvent was removed under reduced pressure, and the residue was directly subjected to column chromatography.

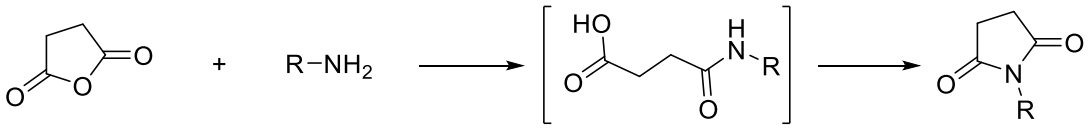
Table S2. Optimization of the reaction conditions.[a]



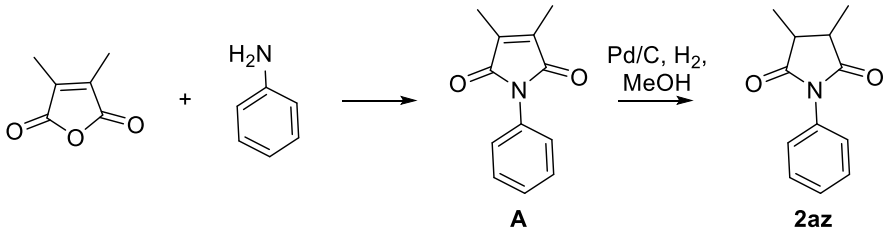
Entry	Thiolation reagent (equiv)	Solvent	Temp. (°C)	reaction time (h)	Isolated yield
1	LR (2.0)	toluene	110	2	34%
2	LR (2.0)	DCM	110	2	12%
3	LR (2.0)	DCE	110	2	10%
4	LR (2.0)	dioxane	110	2	N. D.[b]
5	LR (2.0)	THF	110	2	N. D.[b]
6	LR (2.0)	MeCN	110	2	9%
7	LR (2.0)	MeOH	110	2	N. D.[b]
8	LR (2.0)	toluene	120	2	29%
9	LR (2.0)	toluene	130	2	39%
10	LR (2.0)	toluene	140	2	31%
11	LR (0.5)	toluene	130	2	N. D.[b]
12	LR (1.0)	toluene	130	2	28%
13	LR (3.0)	toluene	130	2	14%
14	LR (2.0)	toluene	130	12	53%
15	LR (2.0)	toluene	130	24	71%
16	-	toluene	130	24	N. R.[c]
17	P_2S_5/HMDO[d]	toluene	130	24	N. D.[b]

[a.] Reaction conditions: **2a** (0.1 mmol), thiolation reagent (1.0 – 3.0 equiv), solvent (1.0 mL), temperature (110 – 140 °C), Ar. [b.] No product was detected. [c.] No reaction happened, and **2a** was recovered in 80% yield. [d.] P_2S_5 (0.3 equiv), HMDO (1.7 equiv). HMDO = hexamethyldisiloxane.

ii. Preparation and characterization data of succinimides.

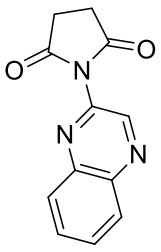


A solution of succinic anhydride (1.0 equiv) and amine (0.9 equiv) in toluene (0.2 M) was heated at reflux. After the disappearance of amine monitored by TLC, toluene was evaporated and acetyl chloride (0.2 M) was added. Then the reaction mixture was heated at reflux until the disappearance of the acid intermediate. The mixture was cooled and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel to give corresponding succinimides. All succinimides were prepared by following this procedure.



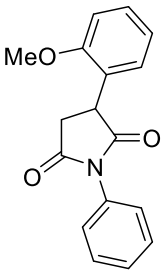
By using the above procedure, succinimide **A** was obtained. The subsequent hydrogenation gave hydrogenated succinimide **2az**. To be specific, **A** (1 equiv) and 10% Pd/C (0.5 equiv) were dissolved in anhyd MeOH (0.2 M) and the mixture was continually stirred under hydrogen atmosphere (hydrogen balloon) at rt. After completion of the reaction monitored by TLC, the mixture was filtered, and the filtrate was evaporated under reduced pressure. The residue was directly purified by silica gel column chromatography (PE : EA = 5 : 1) to give **2az**. Notably, **2y**, **2aj**, **2am, 2ao, and 2aq-2at** are unknown compounds and were characterized by NMR and LC-MS.

1-(quinoxalin-2-yl)pyrrolidine-2,5-dione (2y): eluent: PE : EA = 5 : 1, light yellow powder (400.6 mg, 54%). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.86 (s, 1H), 8.25 – 8.06 (m, 2H), 7.92 – 7.77 (m, 2H), 3.02 (s, 4H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 175.3, 143.1, 142.1, 141.5, 141.4, 131.1,

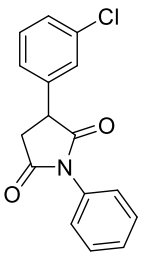


131.0, 129.4, 129.3, 28.9. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{12}H_9N_3NaO_2^+$ 250.0587, found 250.0577.

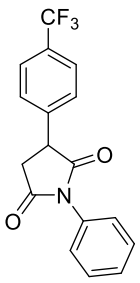
3-(2-methoxyphenyl)-1-phenylpyrrolidine-2,5-dione (2aj): eluent: PE : EA = 5 : 1, light yellow powder (440 mg, 52%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.52 – 7.46 (m, 2H), 7.43 – 7.36 (m, 1H), 7.36 – 7.28 (m, 3H), 7.26 – 7.20 (m, 1H), 6.99 – 6.88 (m, 2H), 4.05 (dd, *J* = 9.8, 5.2 Hz, 1H), 3.80 (s, 3H), 3.22 (dd, *J* = 18.3, 9.9 Hz, 1H), 2.89 (dd, *J* = 18.3, 5.2 Hz, 1H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 177.6, 175.9, 156.8, 132.4, 131.2, 129.5, 129.2, 128.5, 126.5, 126.0, 121.0, 111.1, 55.5, 44.3, 36.4. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{17}H_{15}NNaO_3^+$ 304.0944, found 304.0941.

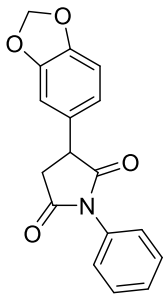


3-(3-chlorophenyl)-1-phenylpyrrolidine-2,5-dione (2am): eluent: PE : EA = 5 : 1, white powder (300 mg, 33%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.50 – 7.44 (m, 2H), 7.43 – 7.37 (m, 1H), 7.34 – 7.28 (m, 5H), 7.21 – 7.15 (m, 1H), 4.14 (dd, *J* = 9.6, 5.0 Hz, 1H), 3.33 (dd, *J* = 18.5, 9.7 Hz, 1H), 2.93 (dd, *J* = 18.5, 5.0 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 176.0, 174.7, 138.9, 135.0, 131.7, 130.5, 129.2, 128.8, 128.3, 127.8, 126.4, 125.6, 45.5, 36.9. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{16}H_{12}ClNNaO_2^+$ 308.0449, found 308.0455.

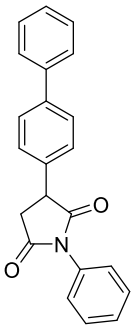


1-phenyl-3-(4-(trifluoromethyl)phenyl)pyrrolidine-2,5-dione (2aq): eluent: PE : EA = 5 : 1, light yellow powder (350 mg, 45%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.67 (d, *J* = 8.2 Hz, 2H), 7.52 – 7.39 (m, 5H), 7.35 – 7.30 (m, 2H), 4.27 (dd, *J* = 9.7, 5.1 Hz, 1H), 3.41 (dd, *J* = 18.5, 9.8 Hz, 1H), 3.00 (dd, *J* = 18.5, 5.1 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.9, 174.5, 141.0 – 140.8 (m), 131.7, 130.7, 130.4, 129.3, 128.9, 128.0, 126.4, 126.3 (q, *J* = 3.8 Hz), 45.7, 36.8. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{17}H_{12}F_3NNaO_2^+$ 342.0712, found 342.0717.

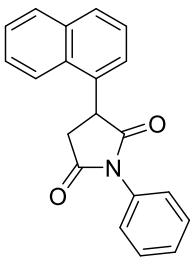


3-(benzo[d][1,3]dioxol-5-yl)-1-phenylpyrrolidine-2,5-dione (2ar): 

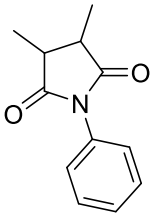
eluent: PE : EA = 5 : 1, white powder (600 mg, 65%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.48 – 7.28 (m, 5H), 6.83 – 6.67 (m, 3H), 5.90 (s, 2H), 4.00 (dd, *J* = 9.5, 4.8 Hz, 1H), 3.21 (dd, *J* = 18.5, 9.7 Hz, 1H), 2.82 (dd, *J* = 18.5, 4.8 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 176.6, 174.9, 148.0, 147.1, 131.8, 130.6, 128.9, 128.4, 126.3, 120.6, 108.4, 107.6, 101.1, 45.4, 37.0. **HRMS (ESI, m/z)** [M + Na]+ calcd for $C_{17}H_{13}NNaO_4^+$ 318.0737, found 318.0745.

3-([1,1'-biphenyl]-4-yl)-1-phenylpyrrolidine-2,5-dione (2as): 

eluent: PE : EA = 5 : 1, white powder (206 mg, 44%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.62 – 7.55 (m, 4H), 7.49 – 7.40 (m, 5H), 7.39 – 7.32 (m, 5H), 4.19 (dd, *J* = 9.7, 4.8 Hz, 1H), 3.35 (dd, *J* = 18.6, 9.7 Hz, 1H), 2.99 (dd, *J* = 18.5, 4.9 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 176.6, 175.1, 141.0, 140.3, 136.0, 131.9, 129.2, 128.8, 128.7, 127.9, 127.8, 127.5, 127.1, 126.5, 45.6, 37.1. **HRMS (ESI, m/z)** [M + Na]+ calcd for $C_{22}H_{17}NNaO_2^+$ 350.1151, found 350.1149.

3-(naphthalen-1-yl)-1-phenylpyrrolidine-2,5-dione (2at): 

eluent: PE : EA = 5 : 1, white powder (390 mg, 78%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.96 – 7.84 (m, 3H), 7.62 – 7.39 (m, 9H), 4.91 (dd, *J* = 9.8, 5.0 Hz, 1H), 3.53 (dd, *J* = 18.5, 9.8 Hz, 1H), 2.97 (dd, *J* = 18.5, 5.0 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 177.1, 175.0, 134.3, 133.8, 132.0, 131.2, 129.34, 129.26, 128.84, 128.78, 126.9, 126.5, 126.2, 125.6, 124.8, 122.7, 43.2, 37.7. **HRMS (ESI, m/z)** [M + Na]+ calcd for $C_{20}H_{15}NNaO_2^+$ 324.0995, found 324.0991.

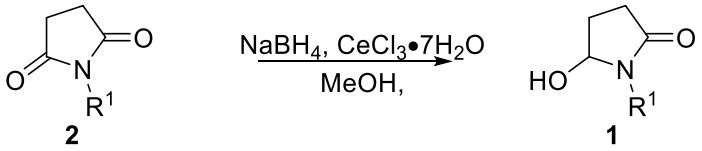
3,4-dimethyl-1-phenylpyrrolidine-2,5-dione (2ao): 

eluent: PE : EA = 5 : 1, white solid (504 mg, 76%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.49 – 7.42 (m, 2H), 7.40 – 7.34 (m, 1H), 7.31 – 7.25 (m, 2H), 3.14 – 3.04 (m, 2H), 1.34 – 1.28 (m, 6H). **^{13}C NMR (76 MHz, $CDCl_3$)** 179.4, 132.0,

129.1, 128.4, 126.4, 38.5, 11.7. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{12}H_{13}NNaO_2^+$ 226.0838, found 226.0845.

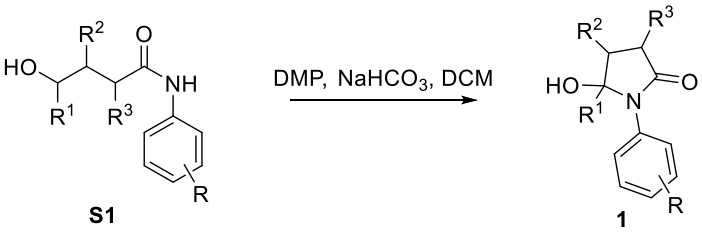
iii. Preparation and characterization data of *γ*-hydroxylactams.

Method A:



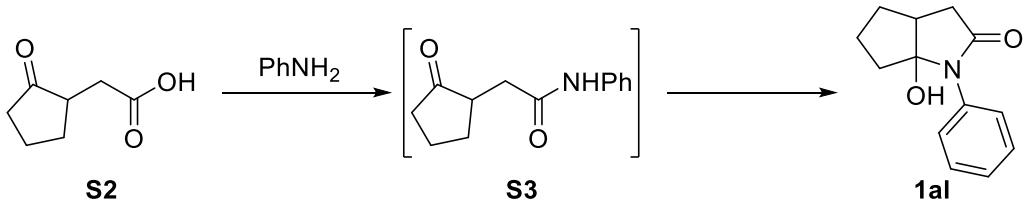
To a stirred solution of succinimide **2** and $CeCl_3•7H_2O$ (1.1 equiv) in MeOH (0.2 M) was added $NaBH_4$ (0.55 equiv) at 0 ^oC. After stirring for 20 min, the other batch of $NaBH_4$ (0.55 equiv) was added. After the completion of the reaction detected by TLC, the reaction was quenched by H_2O, extracted with ethyl acetate, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel to give corresponding *γ*-hydroxy lactams. **1x**, **1t**, and **1ai**, were synthesized by following this procedure.

Method B:

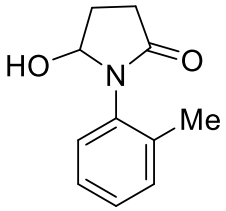


To a stirred mixture of **S1** and $NaHCO_3$ (3.0 equiv) in DCM (0.2M) was added DMP (1.2 equiv) at room temperature. After completion, the reaction was quenched by a 1:1 solution of $NaHCO_3$ and $Na_2S_2O_3$, extracted by DCM, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel to give corresponding *γ*-hydroxy lactams. **1a-1s, 1u-1w, 1y-1ac, 1ag-1ah, 1au, 1az-1aab** were synthesized by following this procedure.

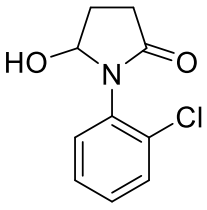
Method C:



To a stirred mixture of acid **S2** and aniline (1.0 equiv) in DCM (0.2 M) was added DCC (1.0 equiv) at rt under Ar atmosphere. After the completion, the reaction was quenched by saturated $NaHCO_3$ aqueous solution. After stirring for 1h in the presence of $NaHCO_3$, the reaction was extracted with DCM, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel to give corresponding *γ*-hydroxy lactams. **1ae-1af** and **1av-1ay** were synthesized by following this procedure. Compounds **1b**, **1c**, **1f**, **1j**, **1p-1q**, **1s-1y**, **1aa-1ac**, **1ag-1ah**, **1au**, **1aw** were unknown compounds and were characterized by NMR, IR and LC-MS.



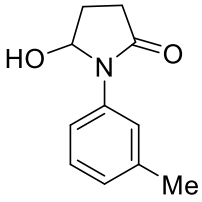
5-Hydroxy-1-(o-tolyl)pyrrolidin-2-one (1b): eluent: PE : EA = 1 : 2, colorless oil, yield: 74%. **^{1}H NMR (400 MHz, methanol-*d*$_4$)** δ 7.34 – 7.23 (m, 3H), 7.12 – 7.05 (m, 1H), 5.65 – 5.60 (m, 1H), 2.82 – 2.69 (m, 1H), 2.53 – 2.41 (m, 2H), 2.36 (s, 3H), 2.02 – 1.93 (m, 1H). **^{13}C NMR (101 MHz, methanol-*d*$_4$)** δ 176.8, 140.0, 138.6, 129.8, 128.3, 126.4, 122.8, 86.8, 30.8, 29.3, 21.5. **IR (KBr, *ν* / cm $^{-1}$)** 3284, 3029, 2932, 2876, 1657, 1588, 1530, 1493, 1456, 1292, 1258, 1057, 751. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{11}H_{13}NNaO_2^+$ 214.0838, found 214.0829.



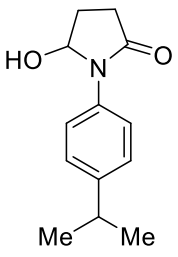
1-(2-Chlorophenyl)-5-hydroxypyrrolidin-2-one (1c): eluent: PE : EA = 1 : 2, white power, yield: 65%. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.48 – 7.41 (m, 1H), 7.31 – 7.21 (m, 3H), 5.42 – 5.33 (m, 1H), 4.31 – 4.14 (m, 1H), 2.68 – 2.56 (m, 1H), 2.50 – 2.30 (m, 2H), 2.06 – 1.92 (m, 1H). **^{13}C NMR (76 MHz, CDCl$_3$)** δ 174.9, 133.7, 132.5, 131.1, 130.1, 129.6, 127.6, 84.7, 28.8, 28.6. **IR (KBr, *ν* / cm $^{-1}$)** 3071, 2995, 2945, 1724, 1485, 1405, 1299, 1252,

1206, 1163, 1064, 1029, 1014. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{10}H_{10}ClNNaO_2^+$ 234.0292, found 234.0289.

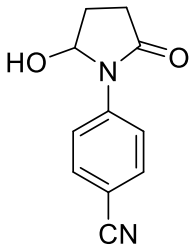
5-Hydroxy-1-(*m*-tolyl)pyrrolidin-2-one (1f): eluent: PE : EA = 1 : 2, colorless oil, yield: 65%. **^{1}H NMR (400 MHz, methanol-*d*$_4$)** δ 7.34 – 7.23 (m, 3H), 7.12 – 7.05 (m, 1H), 5.65 – 5.60 (m, 1H), 2.82 – 2.69 (m, 1H), 2.53 – 2.40 (m, 2H), 2.36 (s, 3H), 2.03 – 1.93 (m, 1H). **^{13}C NMR (101 MHz, methanol-*d*$_4$)** δ 176.8, 140.0, 138.6, 129.8, 128.3, 126.4, 122.8, 86.8, 30.8, 29.3, 21.5. **IR (KBr, *v* / cm $^{-1}$)** 3357, 2958, 1677, 1608, 1590, 1493, 1450, 1407, 1297, 1232, 1180, 1115, 1066, 988, 783, 697. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{11}H_{13}NNaO_2^+$ 214.0838, found 214.0845.



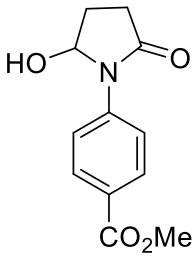
5-Hydroxy-1-(4-isopropylphenyl)pyrrolidin-2-one (1j): eluent: PE : EA = 1 : 2, colorless oil, yield: 62%. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.40 – 7.35 (m, 2H), 7.23 – 7.19 (m, 2H), 5.55 (d, *J* = 5.0 Hz, 1H), 4.08 (s, 1H), 2.88 (hept, *J* = 7.0 Hz, 1H), 2.76 – 2.64 (m, 1H), 2.43 – 2.29 (m, 2H), 2.02 – 1.93 (m, 1H), 1.22 (d, *J* = 6.9 Hz, 6H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 174.7, 147.1, 134.7, 127.1, 123.9, 85.4, 33.7, 29.7, 28.2, 24.0. **IR (KBr, *v* / cm $^{-1}$)** 3316, 2961, 2932, 2870, 1681, 1612, 1517, 1437, 1407, 1295, 1206, 1064, 837. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{13}H_{17}NNaO_2^+$ 242.1151, found 242.1152.



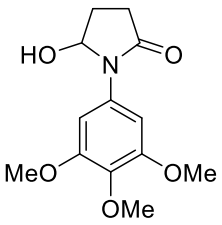
4-(2-Hydroxy-5-oxopyrrolidin-1-yl)benzonitrile (1p): eluent: PE : EA = 1 : 2, white solid, mp 105-106 °C, yield: 67%. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.76 (d, *J* = 8.7 Hz, 2H), 7.58 (d, *J* = 8.7 Hz, 2H), 5.68 (t, *J* = 5.9 Hz, 1H), 4.24 (d, *J* = 7.9 Hz, 1H), 2.84 – 2.70 (m, 1H), 2.52 – 2.34 (m, 2H), 2.10 – 2.00 (m, 1H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 174.8, 141.6, 132.9, 121.7, 118.6, 108.1, 84.6, 29.9, 28.2. **IR (KBr, *v* / cm $^{-1}$)** 3371, 2229, 1687, 1603, 1510, 1429, 1390, 1303, 1202, 1064, 842, 665. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{11}H_{10}N_2NaO_2^+$ 225.0634, found 225.0639.



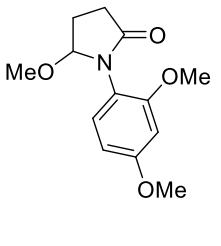
Methyl 4-(2-hydroxy-5-oxopyrrolidin-1-yl)benzoate (1q): eluent: PE : EA = 1 : 2, pale yellow solid, mp 130-131 °C, yield: 49%. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.02 – 7.96 (m, 2H), 7.71 – 7.65 (m, 2H), 5.73 (d, *J* = 5.4 Hz, 1H), 3.90 (s, 3H), 2.84 – 2.70 (m, 1H), 2.52 – 2.36 (m, 2H), 2.09 – 2.01 (m, 1H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 174.5, 166.7, 141.6, 130.6, 126.8, 121.4, 84.8, 52.2, 30.0, 28.3. **IR (KBr, *v* / cm $^{-1}$)** 3349, 2958, 1720, 1605, 1513, 1435, 1390, 1282, 1191, 1115, 772. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{12}H_{13}NNaO_4^+$ 258.0737, found 258.0741.



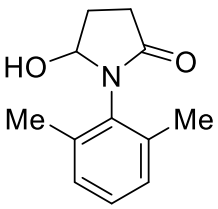
5-Hydroxy-1-(3,4,5-trimethoxyphenyl)pyrrolidin-2-one (1s): eluent: PE : EA = 1 : 2, colorless oil, yield: 68%. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.60 (s, 2H), 5.56 (dd, *J* = 6.0, 1.4 Hz, 1H), 3.80 – 3.71 (m, 9H), 2.76 – 2.64 (m, 1H), 2.47 – 2.32 (m, 2H), 2.07 – 1.95 (m, 1H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 174.6, 153.1, 135.9, 132.9, 102.3, 85.5, 60.8, 56.0, 29.7, 27.8. **IR (KBr, *v* / cm $^{-1}$)** 3347, 1669, 1590, 1503, 1451, 1121. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{13}H_{17}NNaO_5^+$ 290.0999, found 290.1006.



1-(2,4-dimethoxyphenyl)-5-methoxypyrrolidin-2-one (1t): eluent: DCM : MeOH = 20 : 1, white powder, yield: 85%. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.19 (d, *J* = 8.3 Hz, 1H), 7.73 (s, 1H), 6.50 – 6.41 (m, 2H), 3.84 (s, 3H), 3.78 (s, 3H), 3.70 (s, 3H), 2.78 – 2.66 (m, 4H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.3, 169.0, 156.3, 149.1, 121.1, 120.6, 103.6, 98.5, 55.6, 55.5, 51.8, 32.1, 29.2. **IR (KBr, *v* / cm $^{-1}$)** 2952, 1737, 1677, 1530, 1459, 1439, 1416, 1282, 1210, 1159, 1128, 1034. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{13}H_{17}NNaO_4^+$ 274.1050, found 274.1047.

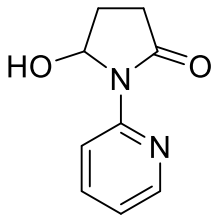


1-(2,6-Dimethylphenyl)-5-hydroxypyrrolidin-2-one (1u): eluent: PE : EA = 1 : 2, colorless oil, yield: 85%. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.17 – 7.00 (m, 3H), 5.29 – 5.21 (m, 1H), 3.99 – 3.84 (m, 1H), 2.67 – 2.55 (m, 1H), 2.43 – 2.23 (m, 2H), 2.20 (s, 3H), 2.08 (s,

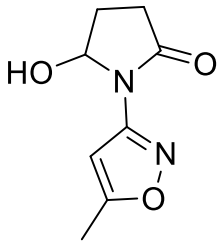


3H), 2.01 – 1.92 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 174.4, 138.4, 135.4, 133.7, 128.7, 128.4, 128.3, 85.0, 28.8, 28.5, 18.6, 17.9. **IR (KBr, *ν* / cm $^{-1}$)** 3330, 2960, 1720, 1600, 1585, 1491, 1450, 1285, 1232, 1180. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{12}H_{15}NNaO_2^+$ 228.0995, found 228.0990.

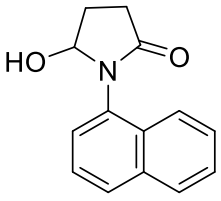
5-Hydroxy-1-(pyridin-2-yl)pyrrolidin-2-one (1v): eluent: PE : EA = 1 : 2, yellow powder, yield: 65%. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.18 (ddd, *J* = 5.0, 1.9, 0.8 Hz, 1H), 8.14 – 8.09 (m, 1H), 7.61 (ddd, *J* = 8.5, 7.4, 1.9 Hz, 1H), 6.96 (ddd, *J* = 7.3, 5.0, 1.0 Hz, 1H), 5.95 (d, *J* = 6.1 Hz, 1H), 2.83 – 2.69 (m, 1H), 2.48 – 2.37 (m, 1H), 2.29 – 2.09 (m, 1H), 2.04 – 1.93 (m, 1H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 174.1, 151.2, 146.9, 138.0, 119.7, 114.9, 83.3, 30.6, 25.2. **IR (KBr, *ν* / cm $^{-1}$)** 3472, 2932, 2958, 1713, 1590, 1474, 1437, 1392, 1303, 1213, 1066. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_9H_{10}N_2NaO_2^+$ 201.0634, found 201.0630.



5-Hydroxy-1-(5-methylisoxazol-3-yl)pyrrolidin-2-one (1w): eluent: DCM : MeOH = 20 : 1, colorless oil, yield: 57 %. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.71 – 6.65 (m, 1H), 5.82 (dd, *J* = 6.4, 1.6 Hz, 1H), 4.86 (s, 1H), 2.72 (ddd, *J* = 17.2, 9.5, 8.1 Hz, 1H), 2.44 – 2.26 (m, 5H), 2.09 – 1.99 (m, 1H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 173.6, 169.9, 157.0, 95.4, 82.1, 29.5, 26.6, 12.4. **IR (KBr, *ν* / cm $^{-1}$)** 3416, 3170, 2967, 1724, 1612, 1508, 1454, 1388, 1279, 1232, 1169, 1092, 1068, 986, 801. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_8H_{10}N_2NaO_3^+$ 205.0584, found 205.0592.

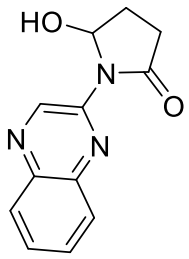


5-Hydroxy-1-(naphthalen-1-yl)pyrrolidin-2-one (1x): eluent: PE : EA = 1 : 2, white powder, yield: 65%. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.91 – 7.84 (m, 2H), 7.79 – 7.62 (m, 1H), 7.54 – 7.44 (m, 3H), 7.34 (d, *J* = 6.9 Hz, 1H), 5.51 – 5.36 (m, 1H), 3.19 (d, *J* = 4.5 Hz, 1H), 2.86 – 2.70 (m, 1H), 2.60 – 2.45 (m, 2H), 2.15 – 2.06 (m, 1H). **^{13}C NMR (76 MHz, DMSO-*d*$_6$)** δ 174.1, 134.3, 134.0, 130.5, 128.1, 127.9, 126.4, 126.2, 125.7, 123.6,

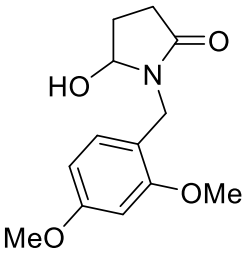


85.3, 29.1, 28.9. **IR (KBr, *ν* / cm $^{-1}$)** 3416, 1644, 1047, 1027, 997, 828, 766. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{14}H_{13}NNaO_2^+$ 250.0838, found 250.0844.

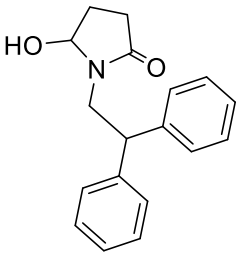
5-Hydroxy-1-(quinoxalin-2-yl)pyrrolidin-2-one (1y): eluent: PE : EA = 1 : 1, white solid, mp 141-142 ºC, yield: 55%. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.76 (s, 1H), 7.98 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.74 (dd, *J* = 8.1, 1.8 Hz, 1H), 7.65 – 7.55 (m, 2H), 6.15 (d, *J* = 5.9 Hz, 1H), 5.11 (s, 1H), 2.98 – 2.84 (m, 1H), 2.58 (ddd, *J* = 17.7, 9.8, 3.3 Hz, 1H), 2.46 – 2.32 (m, 1H), 2.22 – 2.11 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 174.6, 146.2, 139.8, 139.6, 139.5, 130.5, 129.0, 128.4, 127.5, 83.1, 30.6, 25.8. **IR (KBr, *ν* / cm $^{-1}$)** 3338, 2999, 2969, 1716, 1698, 1562, 1476, 1497, 1422, 1398, 1329, 1254, 1206, 1062, 1001, 988, 770. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{12}H_{11}N_3NaO_2^+$ 252.0743, found 252.0749.



1-(2,4-Dimethoxybenzyl)-5-hydroxypyrrolidin-2-one (1aa): eluent: PE : EA = 1 : 2, colorless oil, yield: 68%. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.17 (d, *J* = 8.7 Hz, 1H), 6.45 – 6.38 (m, 2H), 5.09 – 5.02 (m, 1H), 4.61 (d, *J* = 14.6 Hz, 1H), 4.55 – 4.44 (m, 1H), 4.22 (d, *J* = 14.6 Hz, 1H), 3.79 (s, 3H), 3.75 (s, 3H), 2.60 – 2.48 (m, 1H), 2.29 – 2.09 (m, 2H), 1.90 – 1.80 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 174.6, 160.4, 158.1, 131.2, 116.8, 104.4, 98.4, 82.4, 55.4, 55.2, 37.8, 29.0, 27.2. **IR (KBr, *ν* / cm $^{-1}$)** 3330, 2948, 2840, 1668, 1616, 1592, 1511, 1463, 1420, 1288, 1210, 1159, 1126, 1036, 835. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{13}H_{17}NNaO_4^+$ 274.1050, found 274.1045.

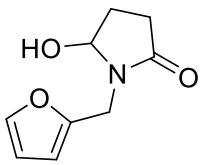


1-(2,2-Diphenylethyl)-5-hydroxypyrrolidin-2-one (1ab): eluent: PE : EA = 1 : 1, white powder, yield: 67 %. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.29 – 7.22 (m, 8H), 7.22 – 7.15 (m, 2H), 4.67 (d, *J* = 6.3 Hz, 1H), 4.41 (t, *J* = 8.3 Hz, 1H), 4.30 (s, 1H), 4.17 (dd, *J* = 13.7, 8.2 Hz, 1H), 3.68 (dd, *J* = 13.7, 8.4 Hz, 1H), 2.48 –

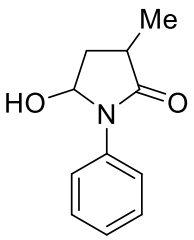


2.35 (m, 1H), 2.17 – 2.05 (m, 1H), 2.01 – 1.89 (m, 1H), 1.77 – 1.66 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.4, 142.3, 141.4, 128.6, 128.6, 128.1, 127.9, 126.8, 126.8, 83.3, 48.6, 44.4, 28.8, 28.1. **IR (KBr, *v* / cm $^{-1}$)** 3332, 3066, 3030, 2935, 1666, 1495, 1454, 1422, 1329, 1286, 1251, 1169, 1070, 956, 740, 703. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{18}H_{19}NNaO_2^+$ 304.1308, found 304.1301.

1-(Furan-2-ylmethyl)-5-hydroxypyrrolidin-2-one (1ac): eluent: PE : EA = 1 : 2, yellow oil, yield: 63%. **^{1}H NMR (400 MHz, acetone-*d*$_6$)** δ 7.49 – 7.42 (m, 1H), 6.39 – 6.23 (m, 2H), 5.28 (d, *J* = 7.2 Hz, 1H), 5.20 – 5.14 (m, 1H), 4.74 (d, *J* = 15.6 Hz, 1H), 4.15 (d, *J* = 15.6 Hz, 1H), 2.48 – 2.39 (m, 1H), 2.32 – 2.16 (m, 2H), 1.90 – 1.82 (m, 1H). **^{13}C NMR (101 MHz, acetone-*d*$_6$)** δ 174.2, 151.8, 143.0, 111.1, 108.5, 82.6, 36.3, 29.1, 28.6. **IR (KBr, *v* / cm $^{-1}$)** 3332, 2961, 1672, 1461, 1422, 1279, 1139, 1077, 1012. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_9H_{11}NNaO_3^+$ 204.0631, found 204.0638.

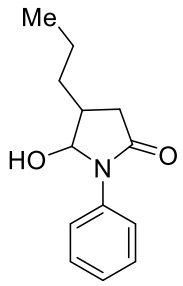


5-Hydroxy-3-methyl-1-phenylpyrrolidin-2-one (1ag): eluent: PE : EA = 3 : 1, colorless oil, yield: 82%. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.56 (d, *J* = 7.7 Hz, 2H), 7.40 (t, *J* = 7.9 Hz, 2H), 7.25 – 7.20 (m, 1H), 5.63 (t, *J* = 5.8 Hz, 1H), 2.98 – 2.86 (m, 1H), 2.73 (d, *J* = 5.9 Hz, 1H), 2.32 (dd, *J* = 13.4, 8.3 Hz, 1H), 2.06 – 1.95 (m, 1H), 1.28 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 176.9, 137.5, 129.2, 126.1, 123.0, 83.2, 37.2, 35.0, 16.1. **IR (KBr, *v* / cm $^{-1}$)** 3375, 2967, 2932, 1685, 1597, 1498, 1459, 1407, 1299, 1284, 1202, 1101, 1058, 1027, 760, 721, 693. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{11}H_{13}NNaO_2^+$ 214.0838, found 214.0844.

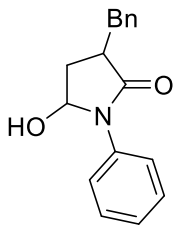


5-Hydroxy-1-phenyl-4-propylpyrrolidin-2-one (1ah): eluent: PE : EA = 1 : 2, colorless oil, yield: 55%. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.46 (d, *J* = 7.8 Hz, 2H), 7.35

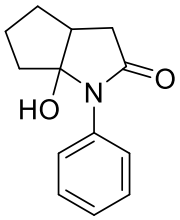
(t, *J* = 7.8 Hz, 2H), 7.21 (t, *J* = 7.4 Hz, 1H), 5.19 (s, 1H), 4.23 (s, 1H), 2.78 (dd, *J* = 17.2, 8.3 Hz, 1H), 2.19 – 2.02 (m, 2H), 1.60 – 1.46 (m, 1H), 1.40 – 1.28 (m, 3H), 0.92 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 174.2, 137.1, 129.0, 126.1, 123.5, 90.2, 40.7, 36.1, 35.4, 20.2, 14.0. **IR (KBr, *v* / cm $^{-1}$)** 3355, 2987, 2928, 1690, 1590, 1488, 1456, 1408, 1276, 1250, 1202. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{13}H_{17}NNaO_2^+$ 242.1151, found 242.1157.



3-benzyl-5-hydroxy-1-phenylpyrrolidin-2-one (1au): eluent: DCM : MeOH = 40: 1, white powder, yield: 75%. **^{1}H NMR (400 MHz, methanol-d_4)** δ 7.48 – 7.44 (m, 2H), 7.41 – 7.36 (m, 2H), 7.32 – 7.27 (m, 2H), 7.27 – 7.21 (m, 4H), 5.37 (dd, *J* = 6.2, 1.4 Hz, 1H), 3.25 – 3.15 (m, 2H), 2.81 – 2.73 (m, 1H), 2.21 – 2.11 (m, 1H), 2.03 – 1.95 (m, 1H). **^{13}C NMR (101 MHz, methanol-d_4)** δ 177.7, 140.2, 138.9, 130.2, 129.9, 129.6, 127.6, 127.4, 125.3, 84.7, 43.5, 37.6, 35.6. **IR (KBr, *v* / cm $^{-1}$)** 3375, 2943, 1677, 1597, 1498, 1407, 1288, 1215, 1118, 1083, 1051. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{17}H_{17}NNaO_2^+$ 290.1151, found 290.1150.



6a-Hydroxy-1-phenylhexahydrocyclopenta[b]pyrrol-2(1H)-one (1aw): eluent: PE : EA = 3:1, colorless oil, yield: 74%. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.46 – 7.33 (m, 4H), 7.32 – 7.26 (m, 1H), 4.09 (s, 1H), 2.85 (dd, *J* = 17.8, 9.4 Hz, 1H), 2.51 – 2.39 (m, 1H), 2.23 – 2.09 (m, 2H), 1.91 – 1.80 (m, 1H), 1.79 – 1.67 (m, 2H), 1.67 – 1.53 (m, 1H), 1.47 – 1.35 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.0, 136.3, 129.0, 127.2, 127.0, 103.0, 44.3, 37.4, 37.2, 34.0, 24.3. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{13}H_{15}NNaO_2^+$ 240.0995, found 240.0991.

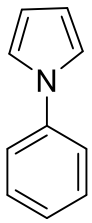


iv. Preparation and characterization data of pyrroles.

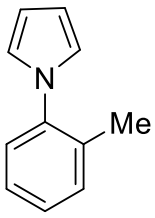
Procedure A (succinimides as the substrates): Unless otherwise noted, a Schlenk tube was charged with a mixture of succinimides **2** (0.1 mmol) and Lawesson's reagent (80.8 mg, 0.2 mmol) in toluene (1.0 mL) under an argon atmosphere. The vial was sealed and stirred vigorously at 130 °C. After stirring for 24 h, the reaction was cooled to room temperature, and the solvent was removed under reduced pressure. The residue was directly subjected to column chromatography. The yields were depicted in the main text.

Procedure B (*γ*-hydroxylactams as the substrates): Unless otherwise noted, a Schlenk tube was charged with a mixture of *γ*-hydroxylactams **1** (0.1 mmol) and Lawesson's reagent (80.8 mg, 0.2 mmol) in toluene (1.0 mL) under an argon atmosphere. The vial was sealed and stirred vigorously at 110 °C. After stirring for 2 h, the reaction was cooled to room temperature, and the solvent was removed under reduced pressure. The residue was directly subjected to column chromatography. The yields were depicted in parentheses.

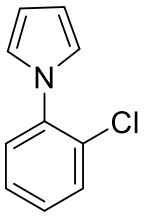
1-Phenyl-1H-pyrrole (3a):[1] eluent: PE : EA = 10 : 1, white solid (10.4 mg, 73%), mp 58.3-59.3 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.45 – 7.36 (m, 4H), 7.27 – 7.20 (m, 1H), 7.09 (t, *J* = 2.1 Hz, 2H), 6.35 (t, *J* = 2.1 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.8, 129.6, 125.6, 120.6, 119.3, 110.4. **IR (KBr, *ν* / cm $^{-1}$)** 3144, 1605, 1513, 1461, 1327, 1256, 1085, 759, 721, 690.



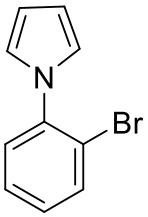
1-(*o*-Tolyl)-1H-pyrrole (3b):[1] eluent: PE : EA = 50 : 1, colorless oil (14.1 mg, 90%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.37 – 7.27 (m, 4H), 6.87 – 6.81 (m, 2H), 6.41 – 6.33 (m, 2H), 2.27 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.6, 133.8, 131.0, 127.5, 126.6, 126.5, 122.0, 108.7, 17.9. **IR (KBr, *ν* / cm $^{-1}$)** 3103, 3071, 3030, 2958, 2926, 1504, 1480, 1459, 1325, 1101, 1072, 1018, 926, 762, 727.



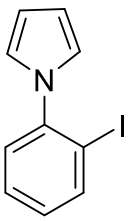
1-(2-Chlorophenyl)-1H-pyrrole (3c):[2] eluent: PE : EA = 50 : 1, white solid (14.5 mg, 82%), mp 86-87 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.53 – 7.49 (m, 1H), 7.38 – 7.32 (m, 2H), 7.32 – 7.27 (m, 1H), 6.92 (t, *J* = 2.1 Hz, 2H), 6.35 (t, *J* = 2.1 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 138.8, 130.7, 129.7, 128.2, 127.9, 127.6, 122.2, 109.3. **IR (KBr, *v* / cm $^{-1}$)** 3134, 2935, 1606, 1532, 1460, 1341, 1268.



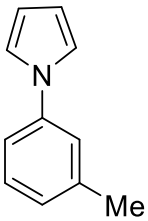
1-(2-Bromophenyl)-1H-pyrrole (3d):[3] eluent: PE : EA = 30 : 1, white solid, (19.4 mg, 88%), mp 73-75 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.69 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.41 – 7.31 (m, 2H), 7.26 – 7.20 (m, 1H), 6.88 (t, *J* = 2.1 Hz, 2H), 6.34 (t, *J* = 2.1 Hz, 2H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 140.4, 133.8, 128.8, 128.3, 128.2, 122.3, 119.9, 109.2. **IR (KBr, *v* / cm $^{-1}$)** 3130, 2930, 1601, 1573, 1502, 1431, 1339, 1256, 1078, 860, 771, 728, 679.



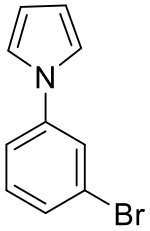
1-(2-Iodophenyl)-1H-pyrrole (3e):[3] eluent: PE : EA = 30 : 1, white solid (21.0 mg, 78%), mp 114-116 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.94 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.41 (td, *J* = 7.7, 1.4 Hz, 1H), 7.31 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.10 (td, *J* = 7.7, 1.7 Hz, 1H), 6.82 (t, *J* = 2.2 Hz, 2H), 6.34 (t, *J* = 2.2 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 144.0, 139.9, 129.4, 129.0, 128.1, 122.2, 109.1, 95.9. **IR (KBr, *v* / cm $^{-1}$)** 3050, 2987, 1586, 1498.



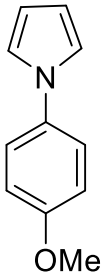
1-(*m*-Tolyl)-1H-pyrrole (3f): [1] eluent: PE : EA = 30 : 1, colorless oil (12.5 mg, 80%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.30 (t, *J* = 7.6 Hz, 1H), 7.23 – 7.17 (m, 2H), 7.10 – 7.04 (m, 3H), 6.34 (t, *J* = 2.1 Hz, 2H), 2.40 (s, 3H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 140.8, 139.6, 129.4, 126.5, 121.4, 119.4, 117.7, 110.2, 21.5. **IR (KBr, *v* / cm $^{-1}$)** 3100, 2984, 1597, 1576, 1490, 1466, 1331, 1312, 1052, 771, 709, 682.



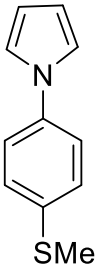
1-(3-Bromophenyl)-1H-pyrrole (3g):[4] eluent: PE : EA = 30 : 1, white solid (19.2 mg, 87%), mp 62-63 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.54 (t, *J* = 1.9 Hz, 1H), 7.38 – 7.22 (m, 3H), 7.05 (t, *J* = 2.2 Hz, 2H), 6.34 (t, *J* = 2.2 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 141.9, 130.8, 128.5, 123.5, 123.1, 119.2, 118.9, 111.1. **IR (KBr, *v* / cm $^{-1}$)** 3152, 3133, 3073, 1597, 1575, 1500, 1469, 1433, 1336, 1254, 1075, 1021, 993, 863, 775, 727, 678.



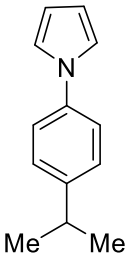
1-(4-Methoxyphenyl)-1H-pyrrole (3h): [1] eluent: PE : EA = 40 : 1, pale yellow oil (14.7 mg, 85%). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.32 (d, *J* = 8.5 Hz, 2H), 7.04 – 6.98 (m, 2H), 6.95 (d, *J* = 8.5 Hz, 2H), 6.41 – 6.25 (m, 2H), 3.84 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 157.7, 134.5, 122.2, 119.7, 114.6, 109.9, 55.6. **IR (KBr, *v* / cm $^{-1}$)** 3144, 3016, 2961, 2837, 1522, 1258, 1027, 824, 720.



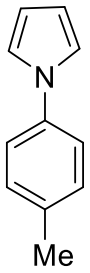
1-(4-(methylthio)phenyl)-1H-pyrrole(3i):[5] eluent: PE : EA = 30 : 1, white solid (12.7 mg, 67%). mp 112-113 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.32 (s, 4H), 7.06 (t, *J* = 2.2 Hz, 2H), 6.34 (t, *J* = 2.2 Hz, 2H), 2.51 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 138.3, 135.5, 128.1, 121.1, 119.3, 110.4, 16.5. **IR (KBr, *v* / cm $^{-1}$)** 3079, 2911, 1469, 1424, 1381, 1113, 1089, 1063, 1001, 969, 802.



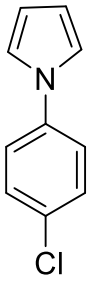
1-(4-Isopropylphenyl)- pyrrole (3j): [1] eluent: PE : EA = 30 : 1, white solid (15.0 mg, 81%), mp 67.0-67.9 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.34 – 7.30 (m, 2H), 7.30 – 7.26 (m, 2H), 7.07 (t, *J* = 2.2 Hz, 2H), 6.34 (t, *J* = 2.2 Hz, 2H), 2.94 (hept, *J* = 6.9 Hz, 1H), 1.28 (d, *J* = 6.9 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 146.4, 138.7, 127.5, 120.7, 119.5, 110.0, 33.6, 24.1. **IR (KBr, *v* / cm $^{-1}$)** 3446, 2963, 2926, 1638, 1525, 1461, 1329, 1077.



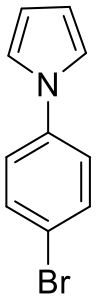
1-(*p*-Tolyl)-1H-pyrrole (3k):[6] eluent: PE : EA = 30 : 1, colorless oil (12.1 mg, 77%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.30 – 7.26 (m, 2H), 7.21 (d, *J* = 8.2 Hz, 2H), 7.05 (t, *J* = 2.2 Hz, 2H), 6.33 (t, *J* = 2.2 Hz, 2H), 2.37 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 138.5, 135.4, 130.1, 120.6, 119.4, 110.1, 20.9. **IR (KBr, *v* / cm $^{-1}$)** 3049, 2950, 1653, 1457, 1403, 1025, 814, 718.



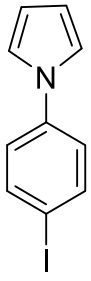
1-(4-Chlorophenyl)-1H-pyrrole (3l): [1] eluent: PE : EA = 30 : 1, white solid (16.4 mg, 93%), mp 87-89 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.42 – 7.37 (m, 2H), 7.36 – 7.30 (m, 2H), 7.05 (t, *J* = 2.1 Hz, 2H), 6.36 (t, *J* = 2.1 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 139.4, 131.1, 129.7, 121.7, 119.3, 110.9. **IR (KBr, *v* / cm $^{-1}$)** 3133, 3111, 2926, 1508, 1075, 826, 732.



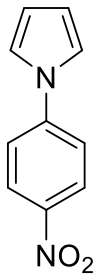
1-(4-Bromophenyl)-1H-pyrrole (3m): [1] eluent: PE : EA = 50 : 1, white solid (16.6 mg, 75%), mp 92-93 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 – 7.69 (m, 2H), 7.48 – 7.44 (m, 2H), 7.24 (t, *J* = 2.2 Hz, 2H), 6.55 (t, *J* = 2.2 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 139.8, 132.6, 122.0, 119.2, 118.7, 110.9. **IR (KBr, *v* / cm $^{-1}$)** 3100, 3064, 1582, 1489, 1406, 1102, 1051, 908, 812, 722.



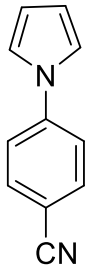
1-(4-Iodophenyl)-1H-pyrrole (3n):[7] eluent: PE : EA = 30 : 1, white solid (18.9 mg, 70%), mp 129-130 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 – 7.70 (m, 2H), 7.19 – 7.12 (m, 2H), 7.05 (t, *J* = 2.2 Hz, 2H), 6.36 (t, *J* = 2.2 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.4, 138.5, 122.2, 119.1, 111.0, 89.4. **IR (KBr, *v* / cm $^{-1}$)** 3149, 3130, 3100, 3059, 1589, 1493, 1405, 1328, 1246, 1189, 1119, 1072, 994, 920, 814.



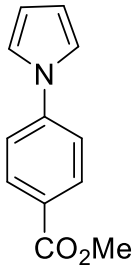
1-(4-Nitrophenyl)-pyrrole (3o): [1] eluent: PE : EA = 30 : 1, white solid (13.7 mg, 73%), mp 182-183 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.31 (d, *J* = 9.1 Hz, 2H), 7.52 (d, *J* = 9.1 Hz, 2H), 7.20 – 7.16 (m, 2H), 6.46 – 6.40 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 145.2, 144.7, 125.6, 119.4, 119.1, 112.6. **IR (KBr, *v* / cm $^{-1}$)** 1581, 1512, 1501, 1459, 1312, 1171, 1079, 1034, 987, 834.



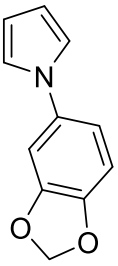
4-(1H-Pyrrol-1-yl)benzonitrile (3p): [2] eluent: PE : EA = 30 : 1, white solid (12.4 mg, 74%), mp 101-102 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 – 7.69 (m, 2H), 7.51 – 7.45 (m, 2H), 7.17 – 7.12 (m, 2H), 6.43 – 6.38 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 143.7, 133.9, 120.0, 118.9, 118.5, 112.2, 108.7. **IR (KBr, *v* / cm $^{-1}$)** 2218, 1620, 1510, 1455, 1330.



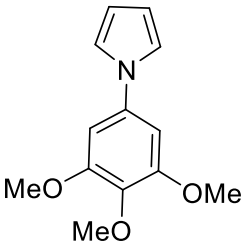
Methyl 4-(1H-pyrrol-1-yl)benzoate (3q): [8] eluent: PE : EA = 30 : 1, white solid (18.9 mg, 94%), mp 125-126 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.10 (d, *J* = 8.8 Hz, 2H), 7.44 (d, *J* = 8.7 Hz, 2H), 7.20 – 7.13 (m, 2H), 6.44 – 6.34 (m, 2H), 3.93 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 166.4, 144.0, 131.3, 126.9, 119.3, 119.0, 111.5, 52.2. **IR (KBr, *v* / cm $^{-1}$)** 1722, 1601, 1518, 1462, 1428, 1327, 1279, 1183, 1103, 837, 754, 709.



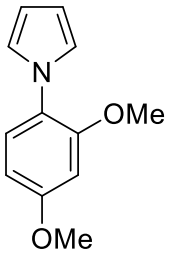
1-(benzo[d][1,3]dioxol-5-yl)-1H-pyrrole (3r):[9] eluent: PE : EA = 50 : 1, yellow powder (10.3 mg, 55%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.95 (t, *J* = 2.2 Hz, 2H), 6.86 (dd, *J* = 1.8, 0.8 Hz, 1H), 6.82 – 6.79 (m, 2H), 6.29 (t, *J* = 2.2 Hz, 2H), 5.96 (s, 2H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 148.3, 145.6, 135.7, 119.8, 114.0, 110.0, 108.4, 103.0, 101.6. **IR (KBr, *v* / cm $^{-1}$)** 3129, 2904, 1616, 1515, 1484, 1454, 1239, 1105, 1077, 1046, 936, 809.



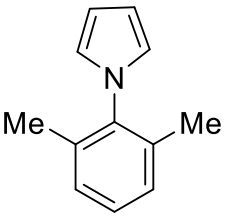
1-(3,4,5-Trimethoxyphenyl)-1H-pyrrole (3s): [10] eluent: PE : EA = 10 : 1, white solid (20.8 mg, 89%), mp 88-89 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.02 (t, *J* = 2.1 Hz, 2H), 6.60 (s, 2H), 6.34 (t, *J* = 2.1 Hz, 2H), 3.90 (s, 6H), 3.87 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 153.8, 137.2, 136.2, 119.8, 110.2, 98.9, 61.1, 56.3. **IR (KBr, *ν* / cm $^{-1}$)** 3000, 2943, 2833, 2846, 1599, 1511, 1467, 1426, 1258, 1234, 1129, 1075, 1006.



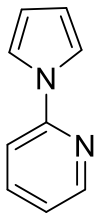
1-(2,4-dimethoxyphenyl)-1H-pyrrole (3t): eluent: PE : EA = 30 : 1, pale yellow solid (15.0 mg, 74%), mp 45-46 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.20 (d, *J* = 8.6 Hz, 1H), 6.90 (t, *J* = 2.2 Hz, 2H), 6.58 (d, *J* = 2.6 Hz, 1H), 6.51 (dd, *J* = 8.6, 2.6 Hz, 1H), 6.29 (t, *J* = 2.1 Hz, 2H), 3.84 (s, 3H), 3.80 (s, 3H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 159.4, 154.1, 126.7, 124.0, 122.3, 108.4, 104.2, 99.8, 55.8, 55.6. **IR (KBr, *ν* / cm $^{-1}$)** 2967, 2937, 1618, 1593, 1521, 1299, 1286, 1210, 1163, 1074, 1044. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{12}H_{13}NNaO_2^+$ 226.0838, found 226.0845.



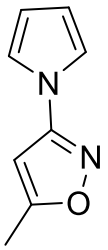
1-(2,6-Dimethylphenyl)-pyrrole (3u): [1] eluent: PE : EA = 30 : 1, pale yellow solid (7.7 mg, 45%), mp 45-46 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.14 – 7.07 (m, 1H), 7.06 – 7.00 (m, 2H), 6.52 (t, *J* = 2.1 Hz, 2H), 6.23 (t, *J* = 2.1 Hz, 2H), 1.95 (s, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 139.9, 136.3, 128.0, 127.9, 121.3, 108.5, 17.3. **IR (KBr, *ν* / cm $^{-1}$)** 3120, 3099, 2956, 2924, 1498, 1472, 1314, 1265, 1090, 783, 736.



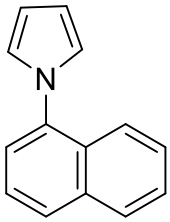
2-(1H-Pyrrol-1-yl)pyridine (3v):[11] eluent: PE : EA = 50 : 1, colorless oil (4.5 mg, 31%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.46 – 8.40 (m, 1H), 7.77 – 7.71 (m, 1H), 7.53 – 7.51 (m, 2H), 7.32 (dt, *J* = 8.4, 0.9 Hz, 1H), 7.13 – 7.08 (m, 1H), 6.38 – 6.34 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 148.7, 138.5, 120.2, 118.1, 111. 5, 111.3. **IR (KBr, *ν* / cm $^{-1}$)** 3140, 3103, 3065, 3018, 1862, 1710, 1591, 1486, 1440, 1392, 1331, 1250, 1152, 1061, 1015, 989, 927, 870, 780, 739, 612.



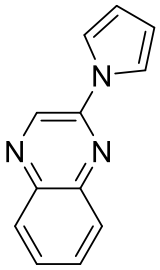
5-Methyl-3-(1H-pyrrol-1-yl)isoxazole (3w): eluent: PE : EA = 8 : 1, colorless oil. 9.6 mg, 65%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.17 (t, *J* = 2.2 Hz, 2H), 6.34 (t, *J* = 2.2 Hz, 2H), 6.11 (s, 1H), 2.45 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.1, 159.5, 119.0, 111.5, 94.1, 12.8. **IR (KBr, *ν* / cm $^{-1}$)** 2958, 2920, 2851, 1620, 1597, 1536, 1497, 1444, 1295, 1075, 1027, 755, 693. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_8H_8N_2NaO^+$ 171.0529, found 171.0531.



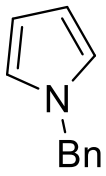
1-(Naphthalen-1-yl)-1H-pyrrole (3x): [1] eluent: PE : EA = 30 : 1, colorless oil (13.5 mg, 70%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.93 (d, *J* = 7.6 Hz, 1H), 7.88 (d, *J* = 8.1 Hz, 1H), 7.76 (d, *J* = 8.3 Hz, 1H), 7.56 – 7.45 (m, 4H), 7.01 (t, *J* = 2.1 Hz, 2H), 6.42 (t, *J* = 2.1 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 138.3, 134.3, 129.9, 128.1, 127.9, 127.0, 126.6, 125.3, 123.32, 123.30, 123.2, 109.1. **IR (KBr, *ν* / cm $^{-1}$)** 2924, 2857, 1589, 1511, 1489, 1459, 1400, 725.



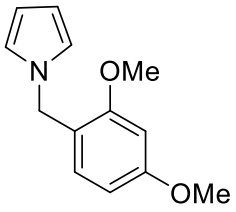
2-(1*H*-Pyrrol-1-yl)quinoxaline (3y): eluent: PE : EA = 8 : 1, pale brown solid (9.9 mg, 51%), mp 98-99 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.12 (s, 1H), 8.06 (d, *J* = 8.3 Hz, 1H), 7.99 (d, *J* = 8.3 Hz, 1H), 7.79 – 7.71 (m, 3H), 7.70 – 7.65 (m, 1H), 6.47 (t, *J* = 2.3 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 145.1, 141.0, 140.5, 136.6, 131.0, 129.2, 128.4, 128.4, 118.4, 112.8. **IR (KBr, *ν* / cm $^{-1}$)** 3137, 3107, 3056, 1571, 1534, 1500, 1465, 1357, 1299, 1122, 1070, 969, 926, 759, 732, 677. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{12}H_9N_3Na^+$ 218.0689, found 218.0691.



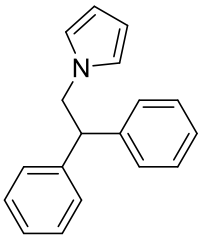
1-Benzyl-1H-pyrrole (3z): [6] eluent: PE : EA = 10 : 1, colorless oil (9.9 mg, 63%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.35 – 7.26 (m, 3H), 7.12 (d, *J* = 7.2 Hz, 2H), 6.70 (t, *J* = 2.0 Hz, 2H), 6.20 (t, *J* = 2.0 Hz, 2H), 5.08 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 138.2, 128.8, 127.7, 127.0, 121.2, 108.5, 53.4. **IR (KBr, *ν* / cm $^{-1}$)** 2922, 2853, 1500, 1456, 1288, 1090, 723.



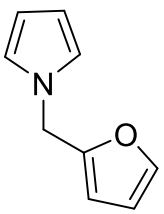
1-(2,4-Dimethoxybenzyl)-1H-pyrrole (3aa): [12] eluent: PE : EA = 30 : 1, colorless oil (14.9 mg, 69%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.81 (d, *J* = 8.3 Hz, 1H), 6.71 (t, *J* = 2.1 Hz, 2H), 6.46 (d, *J* = 2.3 Hz, 1H), 6.41 (dd, *J* = 8.3, 2.4 Hz, 1H), 6.15 (t, *J* = 2.1 Hz, 2H), 5.01 (s, 2H), 3.83 (s, 3H), 3.79 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 160.6, 157.8, 129.4, 121.1, 119.2, 107.9, 104.1, 98.4, 55.4, 55.4, 47.9. **IR (KBr, *ν* / cm $^{-1}$)** 3002, 2939, 2838, 1614, 1590, 1510, 1463, 1439, 1292, 1269, 1210, 1157, 1124, 1087, 1036.



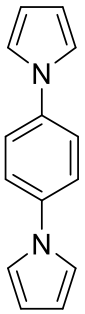
1-(2,2-Diphenylethyl)-1H-pyrrole (3ab): [13] eluent: PE : EA = 20 : 1, pale yellow oil (12.6 mg, 51%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.24 – 7.18 (m, 4H), 7.18 – 7.13 (m, 2H), 7.13 – 7.08 (m, 4H), 6.37 (t, *J* = 2.1 Hz, 2H), 5.95 (t, *J* = 2.1 Hz, 2H), 4.42 (d, *J* = 7.7 Hz, 2H), 4.27 (t, *J* = 7.7 Hz, 1H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 141.9, 128.7, 127.9, 126.9, 120.9, 107.9, 54.7, 53.4. **IR (KBr, *ν* / cm $^{-1}$)** 3062, 3029, 2926, 2853, 1601, 1497, 1452, 1284, 1090, 1068, 1034, 788, 725.

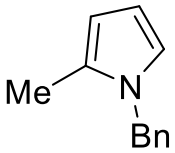


1-(Furan-2-ylmethyl)-1H-pyrrole (3ac):[2] eluent: PE : EA = 30 : 1, colorless oil (9.7 mg, 66%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.37 (dd, *J* = 1.8, 0.8 Hz, 1H), 6.71 (t, *J* = 2.1 Hz, 2H), 6.33 (dd, *J* = 3.2, 1.9 Hz, 1H), 6.26 – 6.23 (m, 1H), 6.17 (t, *J* = 2.1 Hz, 2H), 5.02 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 150.8, 142.7, 120.7, 110.4, 108.5, 108.1, 46.1.

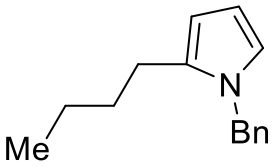


1,4-di(1H-pyrrol-1-yl)benzene (3ad):[14] eluent: PE : EA = 30 : 1, white powder (15.6 mg, 75%, this product was obtained only from the corresponding succinimide substrate). **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.45 (s, 4H), 7.10 (t, *J* = 2.2 Hz, 4H), 6.38 (t, *J* = 2.2 Hz, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 138.5, 121.6, 119.4, 110.7. **IR (KBr, *ν* / cm $^{-1}$)** 1655, 1478, 1403, 1135, 1124, 1092.

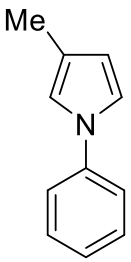


1-Benzyl-2-methyl-1H-pyrrole (3ae): eluent: PE : EA = 30 : 1, colorless oil (13.0 mg, 76%). 

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 2H), 7.26 – 7.22 (m, 1H), 6.99 (d, *J* = 7.4 Hz, 2H), 6.65 – 6.60 (m, 1H), 6.10 (t, *J* = 3.0 Hz, 1H), 5.96 – 5.91 (m, 1H), 5.02 (s, 2H), 2.14 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 138.5, 128.8, 128.7, 127.3, 126.4, 120.9, 107.1, 107.1, 50.4, 12.0. **IR (KBr, *ν* / cm $^{-1}$)** 2933, 2861, 1703, 1687, 1497, 1456, 1422, 1357, 1303, 1079, 1031. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{12}H_{13}NNa^+$ 194.0940, found 194.0943.

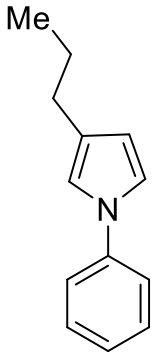
1-Benzyl-2-butyl-1H-pyrrole (3af):[15] eluent: PE : EA = 30 : 1, colorless oil (13.4 mg, 63%). 

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 2H), 7.26 – 7.21 (m, 1H), 6.98 (d, *J* = 7.6 Hz, 2H), 6.63 – 6.58 (m, 1H), 6.13 (t, *J* = 3.1 Hz, 1H), 5.97 – 5.93 (m, 1H), 5.03 (s, 2H), 2.44 (t, *J* = 7.8 Hz, 2H), 1.54 (p, *J* = 7.4 Hz, 2H), 1.33 (h, *J* = 7.3 Hz, 2H), 0.87 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 138.6, 133.7, 128.7, 127.3, 126.4, 120.8, 107.1, 105.9, 50.2, 31.0, 25.9, 22.5, 13.9. **IR (KBr, *ν* / cm $^{-1}$)** 2963, 2922, 2857, 1697, 1606, 1499, 1320, 1080, 757, 697.

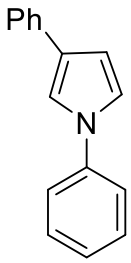
3-Methyl-1-phenyl-1H-pyrrole (3ag): [1] eluent: PE : EA = 50 : 1, white solid (9.1 mg, 58%), mp 49-51 °C. 

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.34 (m, 4H), 7.23 – 7.18 (m, 1H), 7.01 (t, *J* = 2.5 Hz, 1H), 6.90 – 6.87 (m, 1H), 6.21 – 6.17 (m, 1H), 2.18 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 129.5, 125.2, 121.2, 120.0, 119.0, 117.2, 112.0, 12.0. **IR (KBr, *ν* / cm $^{-1}$)** 3470, 3140, 2928, 2868, 1644, 1601, 1510, 1461, 1349, 1265, 1213, 1075, 1053.

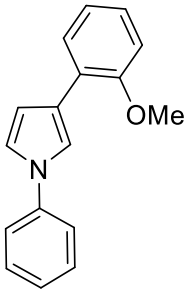
1-Phenyl-3-propyl-1H-pyrrole (3ah): eluent: PE : EA = 30 : 1, colorless oil (16.6 mg, 90%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.34 – 7.25 (m, 4H), 7.15 – 7.07 (m, 1H), 6.93 (t, *J* = 2.5 Hz, 1H), 6.80 (s, 1H), 6.12 (s, 1H), 2.41 (t, *J* = 7.6 Hz, 2H), 1.56 (h, *J* = 7.4 Hz, 2H), 0.90 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 140.8, 129.5, 126.8, 125.1, 119.9, 118.8, 116.4, 110.9, 29.3, 24.2, 14.1. **IR (KBr, *v* / cm $^{-1}$)** 2930, 2958, 2872, 1601, 1510, 1459, 1366, 1074, 1053, 755, 691. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{13}H_{15}NNa^+$ 208.1097, found 208.1099.



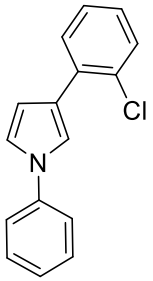
1,3-Diphenyl-1H-pyrrole (3ai):[16] eluent: PE : EA = 30 : 1, pale yellow solid (10.3 mg, 47%), mp 118-120 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.62 – 7.58 (m, 2H), 7.48 – 7.44 (m, 4H), 7.42 – 7.36 (m, 3H), 7.32 – 7.27 (m, 1H), 7.25 – 7.20 (m, 1H), 7.16 – 7.13 (m, 1H), 6.70 – 6.66 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.5, 135.3, 129.7, 128.7, 126.9, 125.9, 125.8, 125.2, 120.37, 120.35, 115.8, 108.7. **IR (KBr, *v* / cm $^{-1}$)** 3130, 3045, 2920, 2855, 1598, 1560, 1507, 1447, 1365, 1307, 1264, 1238, 1183, 1155, 1113, 1082, 1063, 1035, 932, 916, 780, 753, 691.



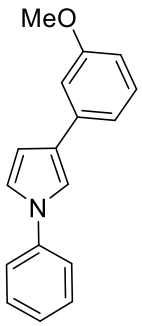
3-(2-methoxyphenyl)-1-phenyl-1H-pyrrole (3aj): eluent: PE : EA = 30 : 1, white powder (13.4 mg, 54%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.64 (t, *J* = 2.0 Hz, 1H), 7.58 (dd, *J* = 7.6, 1.8 Hz, 1H), 7.46 – 7.39 (m, 4H), 7.26 – 7.16 (m, 2H), 7.11 (t, *J* = 2.6 Hz, 1H), 7.01 – 6.94 (m, 2H), 6.75 (dd, *J* = 3.0, 1.7 Hz, 1H), 3.91 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 156.2, 140.8, 129.5, 127.9, 126.7, 125.6, 124.1, 122.4, 120.8, 120.4, 119.17, 119.15, 111.2, 110.3, 55.4. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{17}H_{15}NNaO^+$ 272.1046, found 272.1033.



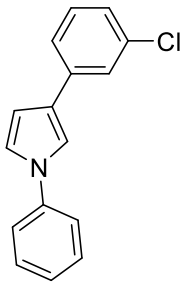
3-(2-chlorophenyl)-1-phenyl-1H-pyrrole (3ak): eluent: PE : EA = 50 : 1 to 20 : 1, pale yellow powder (11.3 mg, 45%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.55 – 7.51 (m, 2H), 7.46 – 7.42 (m, 5H), 7.29 – 7.23 (m, 2H), 7.18 – 7.11 (m, 2H), 6.69 (dd, *J* = 3.0, 1.8 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.5, 134.1, 131.8, 130.4, 130.2, 129.7, 127.0, 126.9, 125.9, 123.6, 120.5, 119.3, 119.2, 111.4. **IR (KBr, *ν* / cm $^{-1}$)** 3079, 1597, 1510, 1437, 1351, 1077, 1057, 1033, 751, 691. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{16}H_{12}ClNNa^+$ 276.0550, found 276.0557.



3-(3-methoxyphenyl)-1-phenyl-1H-pyrrole (3al): eluent: PE : EA = 20 : 1, pale yellow powder (16.4 mg, 66%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.45 – 7.41 (m, 4H), 7.37 (t, *J* = 2.0 Hz, 1H), 7.30 – 7.22 (m, 2H), 7.19 – 7.16 (m, 1H), 7.13 – 7.09 (m, 2H), 6.78 – 6.74 (m, 1H), 6.64 (dd, *J* = 2.9, 1.8 Hz, 1H), 3.84 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 160.0, 140.5, 136.8, 129.7, 129.6, 126.8, 125.8, 120.4, 120.3, 117.9, 116.0, 111.2, 111.0, 108.9, 55.2. **IR (KBr, *ν* / cm $^{-1}$)** 1599, 1508, 1370, 1213, 1047, 759, 691. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{17}H_{15}NNaO^+$ 272.1046, found 272.1038.

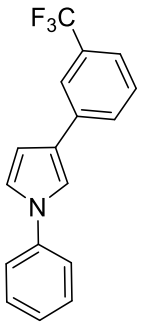


3-(3-chlorophenyl)-1-phenyl-1H-pyrrole (3am): eluent: PE : EA = 50 : 1 to 20 : 1, pale yellow powder (16.4 mg, 65%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.57 (t, *J* = 1.7 Hz, 1H), 7.49 – 7.42 (m, 5H), 7.39 (t, *J* = 1.9 Hz, 1H), 7.32 – 7.26 (m, 2H), 7.20 – 7.16 (m, 1H), 7.13 (t, *J* = 2.6 Hz, 1H), 6.64 (dd, *J* = 2.7, 1.9 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.4, 137.3, 134.6, 129.9, 129.7, 126.1, 125.7, 125.6, 125.2, 123.2, 120.7, 120.5, 116.3, 108.7. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{16}H_{12}ClNNa^+$ 276.0550, found 276.0555.

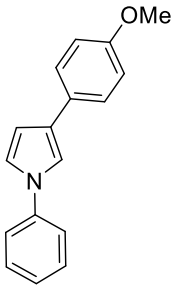


1-phenyl-3-(3-(trifluoromethyl)phenyl)-1H-pyrrole (3an): eluent: PE : EA = 50 : 1 to 20 : 1, pale yellow powder (19.5 mg, 68%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.82 (s, 1H), 7.76 – 7.72 (m, 1H), 7.50 – 7.43 (m, 7H), 7.34 – 7.28 (m, 1H), 7.17 – 7.14 (m,

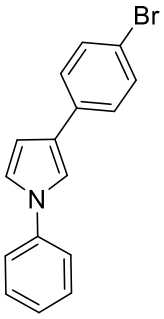
1H), 6.69 (dd, *J* = 3.0, 1.8 Hz, 1H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 140.3, 136.2, 131.2, 130.9, 129.7, 129.1, 128.6 – 127.8 (m), 126.2, 125.5, 122.3 (q, *J* = 3.8 Hz), 121.8 (q, *J* = 3.8 Hz), 120.8, 120.5, 116.4, 108.6. **IR (KBr, *v* / cm $^{-1}$)** 1329, 1174, 1109, 1075, 770, 697. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{17}H_{12}F_3NNa^+$ 310.0814, found 310.0812.



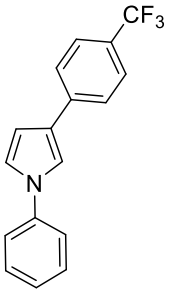
3-(4-methoxyphenyl)-1-phenyl-1H-pyrrole (3ao):[17] eluent: PE : EA = 50 : 1 to 20 : 1, white solid, mp115−118 °C (22.1 mg, 89%). **^{1}H NMR (600 MHz, CDCl$_3$)** δ 7.49 (d, *J* = 8.6 Hz, 2H), 7.45 – 7.41 (m, 4H), 7.30 (t, *J* = 2.0 Hz, 1H), 7.26 – 7.23 (m, 1H), 7.10 (t, *J* = 2.4 Hz, 1H), 6.91 (d, *J* = 8.6 Hz, 2H), 6.60 – 6.58 (m, 1H), 3.82 (s, 3H). **^{13}C NMR (151 MHz, CDCl$_3$)** δ 158.0, 140.6, 129.6, 128.2, 126.7, 126.4, 125.7, 120.3, 120.2, 115.1, 114.2, 108.7, 55.4. **IR (KBr, *v* / cm $^{-1}$)** 3040, 2815, 1602, 1507, 1477, 1263.



3-(4-bromophenyl)-1-phenyl-1H-pyrrole (3ap):[17] eluent: PE : EA = 50 : 1 to 20 : 1, yellow solid, mp 114-116 °C, (27.3 mg, 92%). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.47 – 7.37 (m, 8H), 7.33 (s, 1H), 7.28 – 7.21 (m, 1H), 7.08 (t, *J* = 2.4 Hz, 1H), 6.59 (s, 1H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 140.4, 134.3, 131.7, 129.7, 126.7, 126.0, 125.7, 120.6, 120.4, 119.3, 116.0, 108.6.

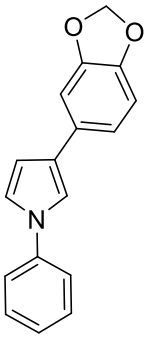


1-phenyl-3-(4-(trifluoromethyl)phenyl)-1H-pyrrole (3aq): eluent: PE : EA = 50 : 1 to 20 : 1, pale yellow powder (26.1 mg, 91%). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.67 (d, *J* = 8.3 Hz, 2H), 7.61 (d, *J* = 8.3 Hz, 2H), 7.50 – 7.43 (m, 5H), 7.34 – 7.28 (m, 1H), 7.15 (t, *J* = 2.6 Hz, 1H), 6.71 – 6.67 (m, 1H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 140.4, 139.0, 129.7, 126.2, 125.7 (q, *J* = 3.8 Hz), 125.5, 125.1, 120.9, 120.6, 116.8,

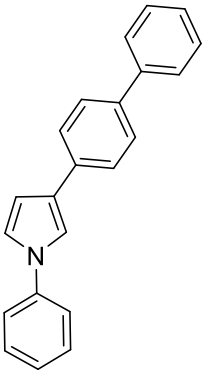


108.8. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{17}H_{12}F_3NNa^+$ 310.0814, found 310.0818.

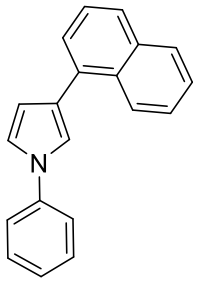
3-(benzo[d][1,3]dioxol-5-yl)-1-phenyl-1H-pyrrole (3ar): eluent: PE : EA = 20 : 1, pale yellow powder (24.2 mg, 92%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.45 – 7.38 (m, 4H), 7.28 – 7.21 (m, 2H), 7.09 – 7.07 (m, 1H), 7.06 – 7.02 (m, 2H), 6.84 – 6.79 (m, 1H), 6.55 (dd, *J* = 2.9, 1.8 Hz, 1H), 5.94 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 148.0, 145.8, 140.5, 129.8, 129.6, 126.8, 125.7, 120.3, 120.2, 118.4, 115.3, 108.7, 108.6, 106.0, 100.9. **IR (KBr, *ν* / cm $^{-1}$)** 1601, 1506, 1482, 1450, 1256, 1228, 1038, 936, 770, 755, 691. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{17}H_{13}NNaO_2^+$ 286.0838, found 286.0844.



3-([1,1'-biphenyl]-4-yl)-1-phenyl-1H-pyrrole (3as): eluent: PE : EA = 50 : 1 to 20 : 1, pale yellow powder (23.0 mg, 78%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.43 – 8.38 (m, 1H), 7.92 – 7.86 (m, 1H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.55 (dd, *J* = 7.1, 1.4 Hz, 1H), 7.50 – 7.44 (m, 7H), 7.34 – 7.32 (m, 1H), 7.30 – 7.24 (m, 1H), 7.24 – 7.21 (m, 1H), 6.65 (dd, *J* = 2.8, 1.7 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.6, 134.2, 134.0, 131.9, 129.7, 128.4, 126.8, 126.5, 126.2, 125.9, 125.8, 125.7, 125.6, 125.5, 120.4, 119.4, 118.4, 112.6. **IR (KBr, *ν* / cm $^{-1}$)** 1653, 1625, 751, 688. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{22}H_{17}NNa^+$ 318.1253, found 318.1250.

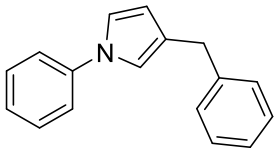


3-(naphthalen-1-yl)-1-phenyl-1H-pyrrole (3at): eluent: PE : EA = 50 : 1 to 20 : 1, pale yellow powder (13.9 mg, 52%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.43 – 8.38 (m, 1H), 7.92 – 7.86 (m, 1H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.55 (dd, *J* = 7.1, 1.4 Hz, 1H), 7.50 – 7.44 (m, 7H), 7.34 – 7.32 (m, 1H), 7.30 – 7.24 (m, 1H), 7.24 – 7.21 (m, 1H), 6.65 (dd, *J* = 2.8, 1.7 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.6, 134.2, 134.0, 131.9, 129.7, 128.4, 126.8, 126.5, 126.2, 125.9, 125.8, 125.7, 125.6, 125.5, 120.4, 119.4, 118.4,

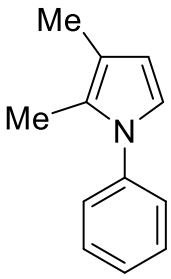


112.6. **IR (KBr, *v* / cm $^{-1}$)** 1508, 1355, 1159, 1053, 781, 757. **HRMS (ESI, m/z)** [M + Na]$^{+}$ calcd for $C_{20}H_{15}NNa^{+}$ 292.1097, found 292.1091.

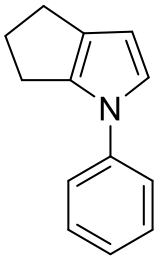
3-benzyl-1-phenyl-1H-pyrrole (3au):[17] eluent: PE : EA = 50 : 1, yellow solid, mp 82-83 °C (17.4 mg, 75%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.41 – 7.33 (m, 4H), 7.31 – 7.28 (m, 4H), 7.23 – 7.18 (m, 2H), 7.03 (t, *J* = 2.5 Hz, 1H), 6.85 (s, 1H), 6.22 – 6.16 (m, 1H), 3.89 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 141.9, 140.7, 129.5, 128.7, 128.4, 125.9, 125.6, 125.3, 120.1, 119.3, 117.3, 111.3, 33.6. **IR (KBr, *v* / cm $^{-1}$)** 2920, 1601, 1510, 1357, 1325, 1228, 1074, 1051, 757, 691.



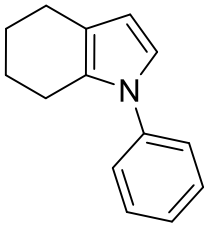
2,3-Dimethyl-1-phenyl-1H-pyrrole (3av):[18] eluent: PE : EA = 30 : 1, colorless oil (11.8 mg, 69%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.48 – 7.41 (m, 2H), 7.37 – 7.27 (m, 3H), 6.73 (d, *J* = 2.8 Hz, 1H), 6.12 (d, *J* = 2.8 Hz, 1H), 2.15 (s, 3H), 2.12 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.7, 129.0, 126.6, 125.7, 125.2, 119.9, 116.2, 109.8, 11.5, 10.6. **IR (KBr, *v* / cm $^{-1}$)** 2922, 2861, 1689, 1599, 1500, 1385, 1182, 1124.



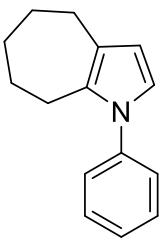
1-Phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (3aw):[19] eluent: PE : EA = 30 : 1, yellow oil (14.3 mg, 78%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.43 – 7.34 (m, 4H), 7.24 – 7.18 (m, 1H), 6.93 (d, *J* = 2.8 Hz, 1H), 6.11 (d, *J* = 2.8 Hz, 1H), 2.88 (t, *J* = 6.9 Hz, 2H), 2.68 (t, *J* = 7.0 Hz, 2H), 2.50 – 2.42 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.8, 137.2, 129.4, 129.0, 125.1, 122.3, 120.9, 105.3, 29.3, 26.7, 25.4. **IR (KBr, *v* / cm $^{-1}$)** 2928, 2855, 1685, 1599, 1500, 1456, 1381, 1264, 759, 695.



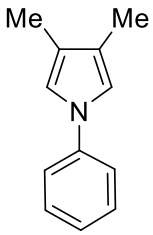
1-Phenyl-4,5,6,7-tetrahydro-1H-indole (3ax):[19] eluent: PE : EA = 30 : 1, yellow oil (15.1 mg, 77%). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.44 – 7.37 (m, 2H), 7.33 – 7.24 (m, 3H), 6.77 (d, *J* = 2.6 Hz, 1H), 6.10 (d, *J* = 2.6 Hz, 1H), 2.63 – 2.53 (m, 4H), 1.82 – 1.75 (m, 4H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 140.2, 129.1, 128.1, 126.1, 124.5, 119.8, 119.0, 108.1, 23.6, 23.5, 23.4, 23.3. **IR (KBr, *v* / cm $^{-1}$)** 1603, 1502, 1308, 692.



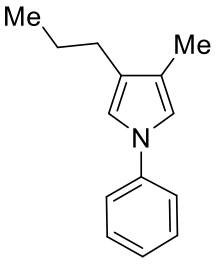
1-Phenyl-1,4,5,6,7,8-hexahydrocyclohepta[b]pyrrole (3ay):[19] eluent: PE : EA = 30 : 1, yellow oil (11.8 mg, 56%). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.44 – 7.39 (m, 2H), 7.34 – 7.29 (m, 1H), 7.26 – 7.22 (m, 2H), 6.58 (d, *J* = 2.7 Hz, 1H), 6.05 (d, *J* = 2.7 Hz, 1H), 2.67 – 2.61 (m, 4H), 1.85 – 1.78 (m, 2H), 1.75 – 1.67 (m, 2H), 1.65 – 1.58 (m, 2H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 140.5, 131.8, 128.9, 126.6, 126.2, 123.7, 118.9, 109.6, 32.3, 28.9, 28.6, 27.8, 27.0. **IR (KBr, *v* / cm $^{-1}$)** 2922, 2879, 1600, 1510, 1298, 687.



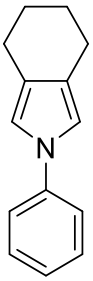
3,4-dimethyl-1-phenyl-1H-pyrrole (3az):[20] eluent: PE : EA = 60 : 1, light yellow solid, mp 62-63 °C, (15.2 mg, 89%). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.37 – 7.32 (m, 2H), 7.31 – 7.27 (m, 2H), 7.16 – 7.10 (m, 1H), 6.82 (s, 2H), 2.07 (s, 6H). **^{13}C NMR (76 MHz, CDCl$_3$)** δ 140.7, 129.4, 124.6, 120.8, 119.4, 116.7, 10.2. **IR (KBr, *v* / cm $^{-1}$)** 2933, 1599, 1502, 1463, 1239, 1154, 1124, 1092.



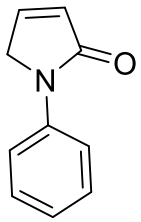
3-Methyl-1-phenyl-4-propyl-1H-pyrrole (3aaa): eluent: PE : EA = 30 : 1, colorless oil (14.9 mg, 75%). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.44 – 7.32 (m, 4H), 7.22 – 7.16 (m, 1H), 6.91 – 6.83 (m, 2H), 2.50 – 2.42 (m, 2H), 2.15 – 2.09 (m, 3H), 1.71 – 1.60 (m, 2H), 1.04 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (151 MHz, CDCl$_3$)** δ 140.8, 129.4, 126.0, 124.6, 120.3, 119.4, 116.8, 116.2, 27.7, 23.4, 14.2, 10.3. **IR (KBr, *v* / cm $^{-1}$)** 2987, 2837, 1598, 1457, 1430, 1377, 758, 749, 677. **HRMS (ESI, m/z)** [M + Na]$^+$ calcd for $C_{14}H_{17}NNa^+$ 222.1253, found 222.1255.



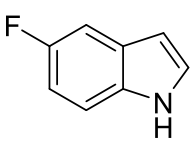
2-Phenyl-4,5,6,7-tetrahydro-2H-isoindole (3aab): eluent: PE : EA = 30 : 1, white solid (14.9 mg, 76%), mp 103-104 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.42 – 7.30 (m, 4H), 7.21 – 7.14 (m, 1H), 6.80 (s, 2H), 2.68 – 2.61 (m, 4H), 1.81 – 1.73 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 141.0, 129.5, 124.8, 122.2, 119.9, 114.6, 24.1, 22.1. **IR (KBr, *v* / cm $^{-1}$)** 2922, 2851, 1593, 1461, 1437, 1383, 766, 751, 688. **HRMS (ESI, m/z)** $[M + Na]^+$ calcd for $C_{14}H_{15}NNa^+$ 220.1097, found 220.1099.



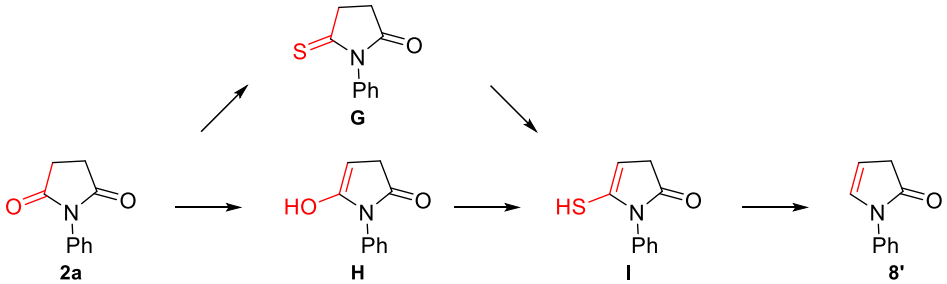
1-phenyl-1,5-dihydro-2H-pyrrol-2-one (10):[21] brown solid, mp 88-90 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 – 7.67 (m, 2H), 7.40 – 7.34 (m, 2H), 7.20 – 7.09 (m, 2H), 6.27 (dt, *J* = 6.1, 1.9 Hz, 1H), 4.44 (t, *J* = 1.9 Hz, 2H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 170.2, 142.2, 139.1, 129.2, 129.1, 124.2, 118.9, 53.2.



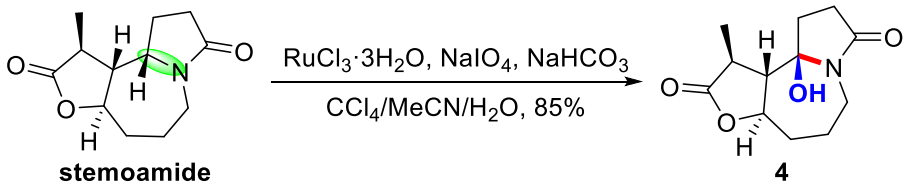
5-Fluoro-1H-indole (7):[22] eluent: PE : EA = 6 : 1, colorless crystalline powder (6.0 mg, 45%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.15 (s, 1H), 7.34 – 7.27 (m, 2H), 7.26 – 7.24 (m, 1H), 6.95 (td, *J* = 9.1, 2.5 Hz, 1H), 6.56 – 6.49 (m, 1H). **^{13}C NMR (76 MHz, $CDCl_3$)** δ 157.9 (d, *J* = 234.0 Hz), 132.3, 128.1 (d, *J* = 10.7 Hz), 126.0, 111.6 (d, *J* = 9.8 Hz), 110.4 (d, *J* = 26.5 Hz), 105.4 (d, *J* = 23.4 Hz), 102.7 (d, *J* = 4.6 Hz). **IR (KBr, *v* / cm $^{-1}$)** 3427, 3097, 3075, 1627, 1482, 1454, 1420, 1342, 1325, 1241, 1211, 1137, 1118, 1090.



v. Mechanism

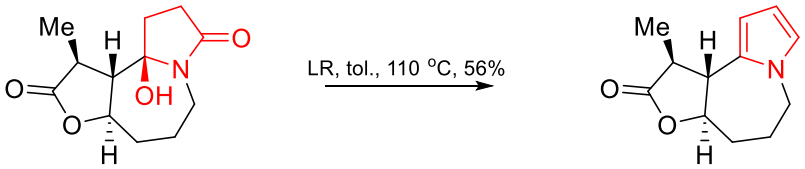


vi. Syntheses of the key intermediate of *Stemona* alkaloids



stemoamide　　4

$NaHCO_3$ (0.76 g, 9 mmol, 5.0 eq), $NaIO_4$ (0.77 g, 3.6 mmol, 2.0 eq), and $RuCl_3·3H_2O$ (94 mg, 0.36 mmol, 0.2 eq) were added to a solution of stemoamide (0.4 g, 1.8 mmol, 1.0 eq) in CCl_4: MeCN: H_2O (95 mL, 2:2:3) at rt. After stirring for 3 h, the reaction was quenched with aqueous saturated $Na_2S_2O_3$. The mixture was extracted with DCM/MeOH = 15:1 (10 × 150 mL) and the combined organic phases were washed with brine (200 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuum. The residue was purified by flash chromatography on silica gel (eluent: ether / EtOAc = 2:3 to DCM/MeOH = 10:1) to give compound **4** (0.37 g, 85%) as a white solid. **^{1}H NMR (400 MHz, methanol-*d*$_4$)** δ 4.13 – 3.98 (m, 1H), 3.92 – 3.77 (m, 1H), 3.08 – 2.97 (m, 1H), 2.88 (dq, *J* = 11.8, 7.0 Hz, 1H), 2.75 – 2.58 (m, 1H), 2.49 (dd, *J* = 12.0, 10.0 Hz, 1H), 2.38 – 2.32 (m, 1H), 2.32 – 2.20 (m, 2H), 2.06 – 1.87 (m, 1H), 1.89 – 1.78 (m, 1H), 1.63 (tdd, *J* = 12.6, 10.9, 4.5 Hz, 1H), 1.56 – 1.40 (m, 1H), 1.33 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR (101 MHz, methanol-*d*$_4$)** δ 179.0, 175.5, 90.3, 78.2, 58.7, 37.3, 37.1, 34.3, 28.9, 28.1, 24.7, 14.1. **IR (KBr, *ν* / cm^{-1})** 3623, 3451, 2927, 1780, 1643, 1423, 1212, 1194, 998. **HRMS (ESI, m/z):** $[M + Na]^+$ calcd for $[C_{12}H_{17}NO_4Na]^+$ 262.1050, found 262.1055.



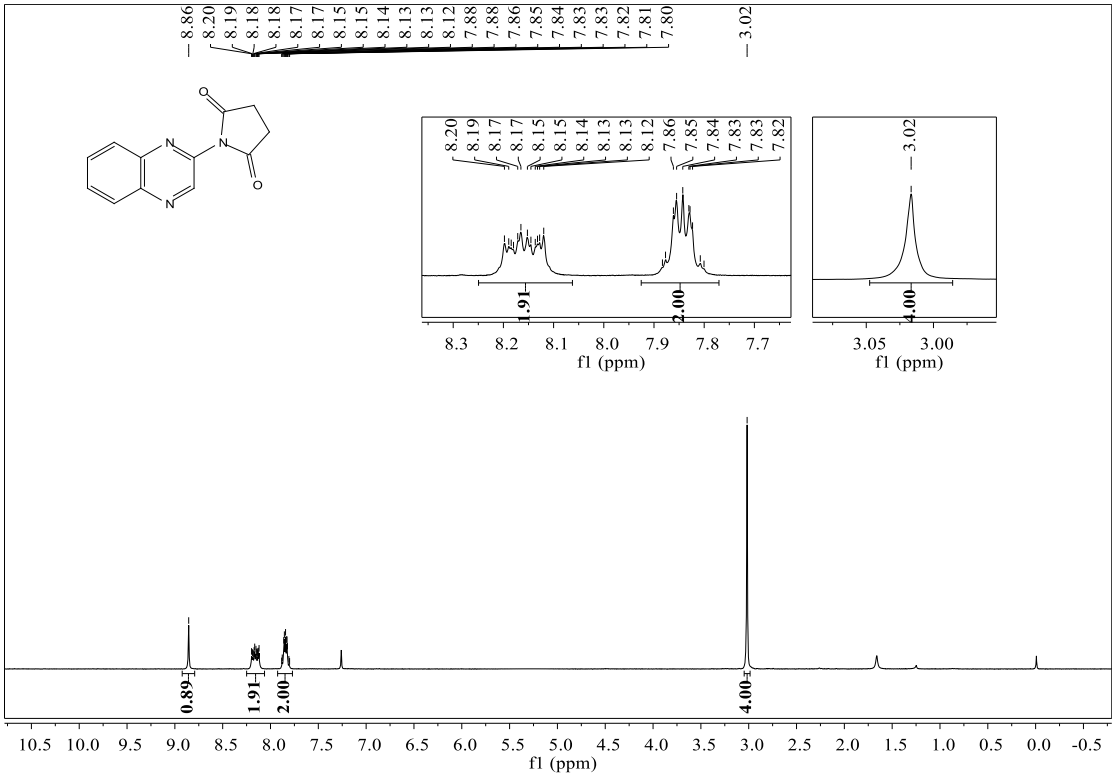
4　　5

A Schlenk tube was charged with a mixture of compound **4** (316.0 mg, 1.32 mmol) and Lawesson's reagent (534 mg, 1.32 mmol, 1.0 equiv) in toluene (6.6 mL) under an argon atmosphere. The vial was sealed and stirred vigorously at 110 °C. After stirring for 2h, the reaction was cooled to room temperature, and the solvent was removed under reduced pressure. The residue was directly subjected to column chromatography (eluent: PE : EA = 3 : 1) on silica gel to give pure **5** as a colorless oil (151.5 mg, 56%). **$[\alpha]_D^{23.6}$** = -210.00 (*c* 0.1 in $CHCl_3$) **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.66 – 6.60 (m, 1H), 6.05

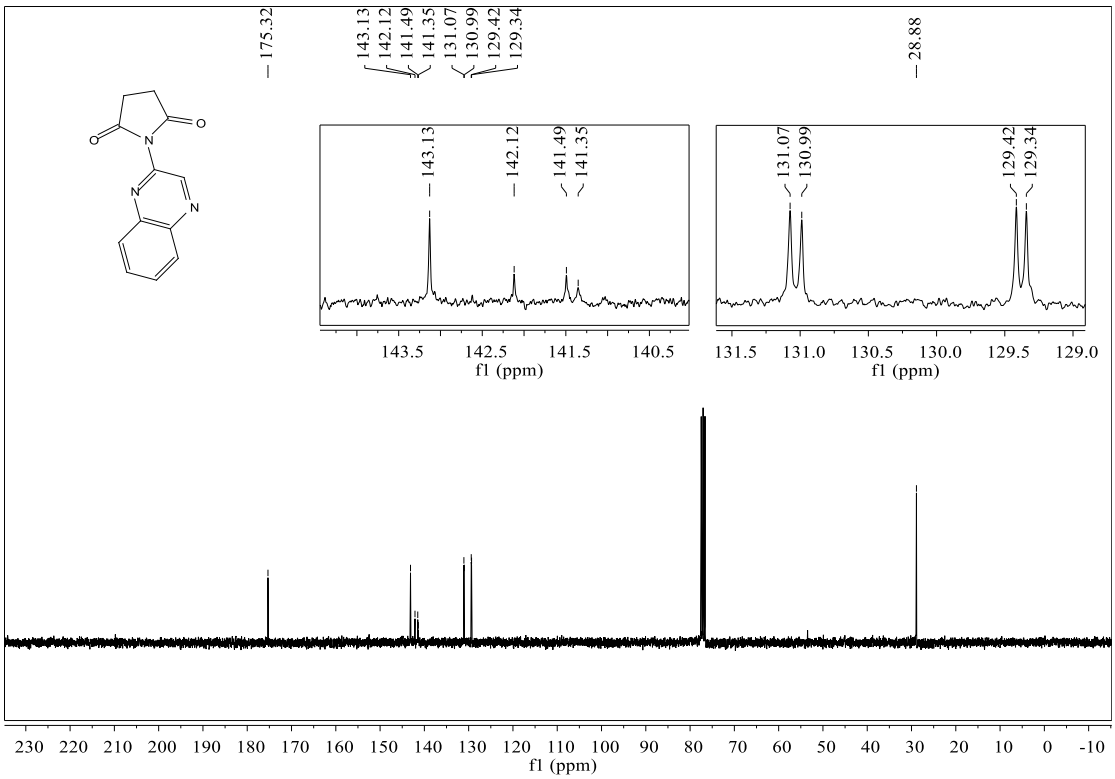
(t, *J* = 3.1 Hz, 1H), 5.99 – 5.94 (m, 1H), 4.12 (dd, *J* = 14.4, 5.2 Hz, 1H), 3.94 – 3.82 (m, 2H), 3.07 – 2.92 (m, 2H), 2.58 – 2.48 (m, 1H), 2.17 – 2.06 (m, 1H), 1.82 – 1.61 (m, 2H), 1.43 (d, *J* = 6.4 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 178.3, 128.6, 122.6, 106.5, 105.0, 81.6, 49.3, 49.1, 39.6, 34.1, 26.2, 13.8. **IR (KBr, *ν* / cm $^{-1}$)** 2937, 1774, 1489, 1454, 1323, 1221, 1202, 1169, 1146, 1013, 937, 723. **LC-MS (ESI, m/z)** cacl $C_{12}H_{15}NNaO_2$ 228.0995, found 228.1.

III. Copies of ^{1}H and ^{13}C NMR Spectra

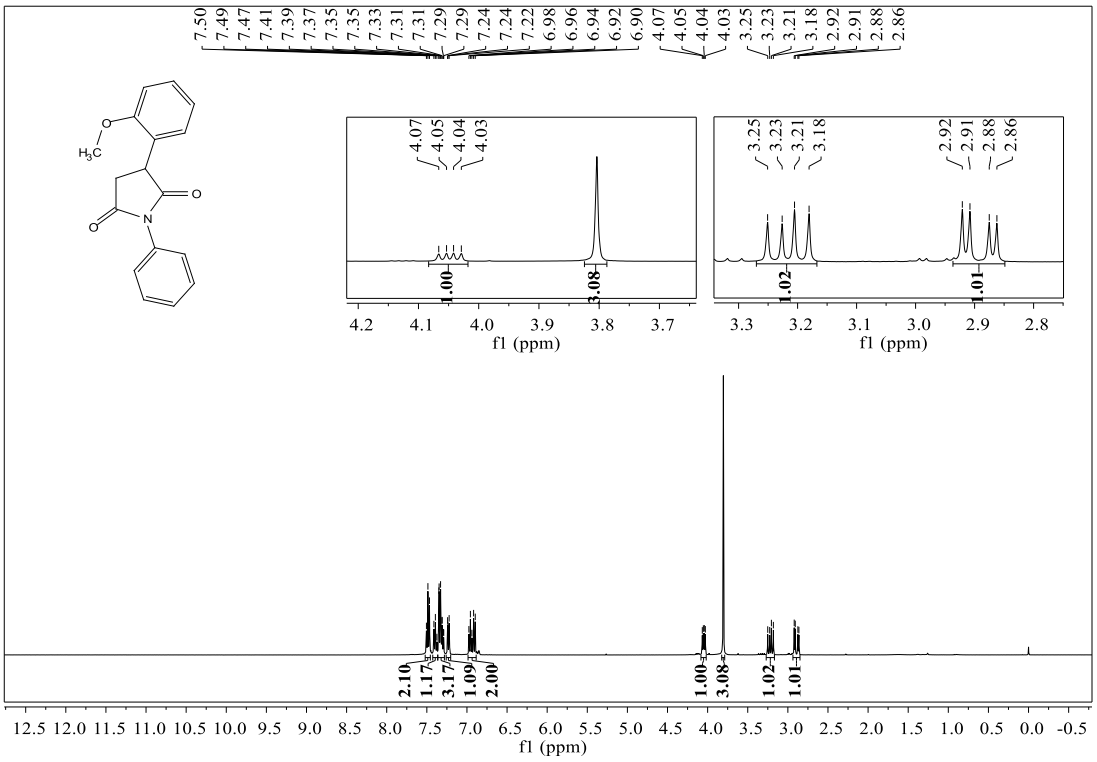
^{1}H NMR spectrum of compound 2y (300 MHz, $CDCl_3$)



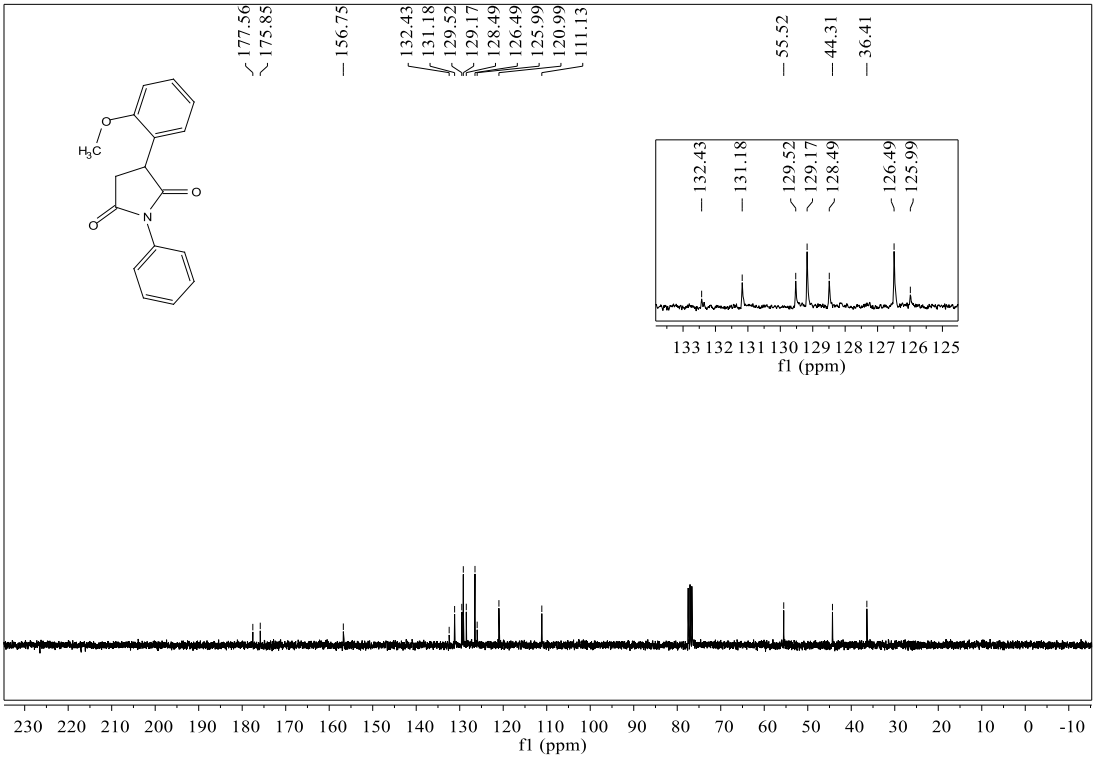
^{13}C NMR spectrum of compound 2y (76 MHz, $CDCl_3$)



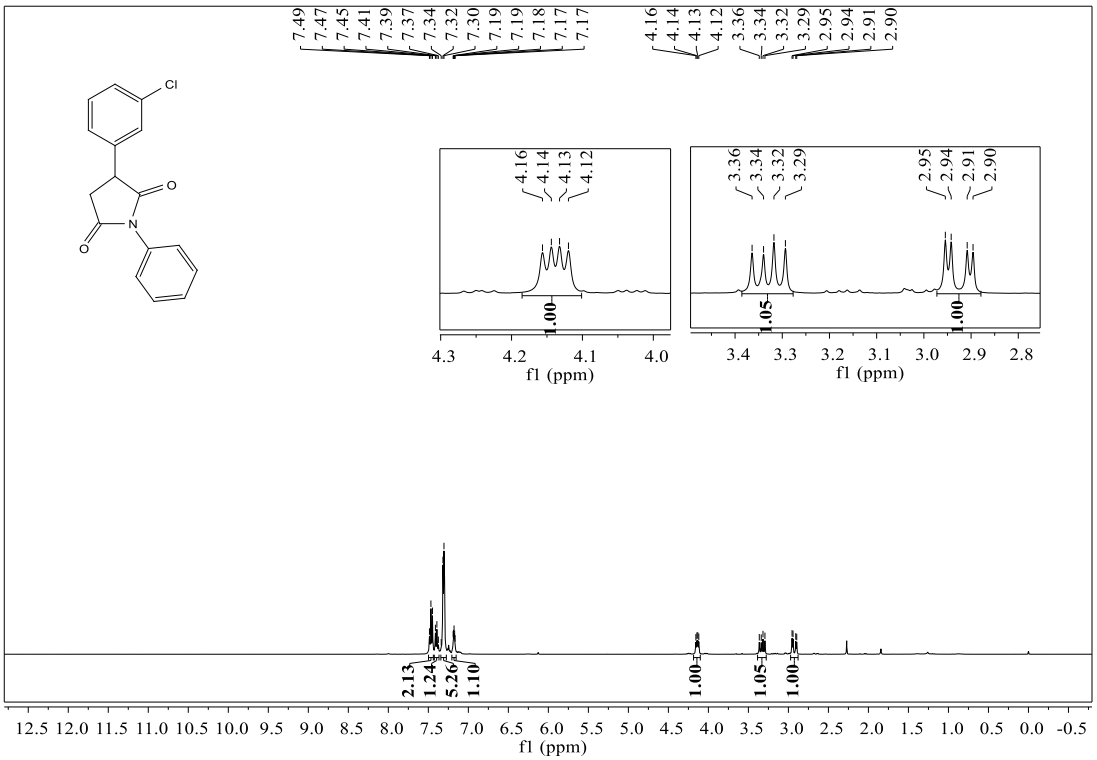
^{1}H NMR spectrum of compound 2aj (400 MHz, $CDCl_3$)



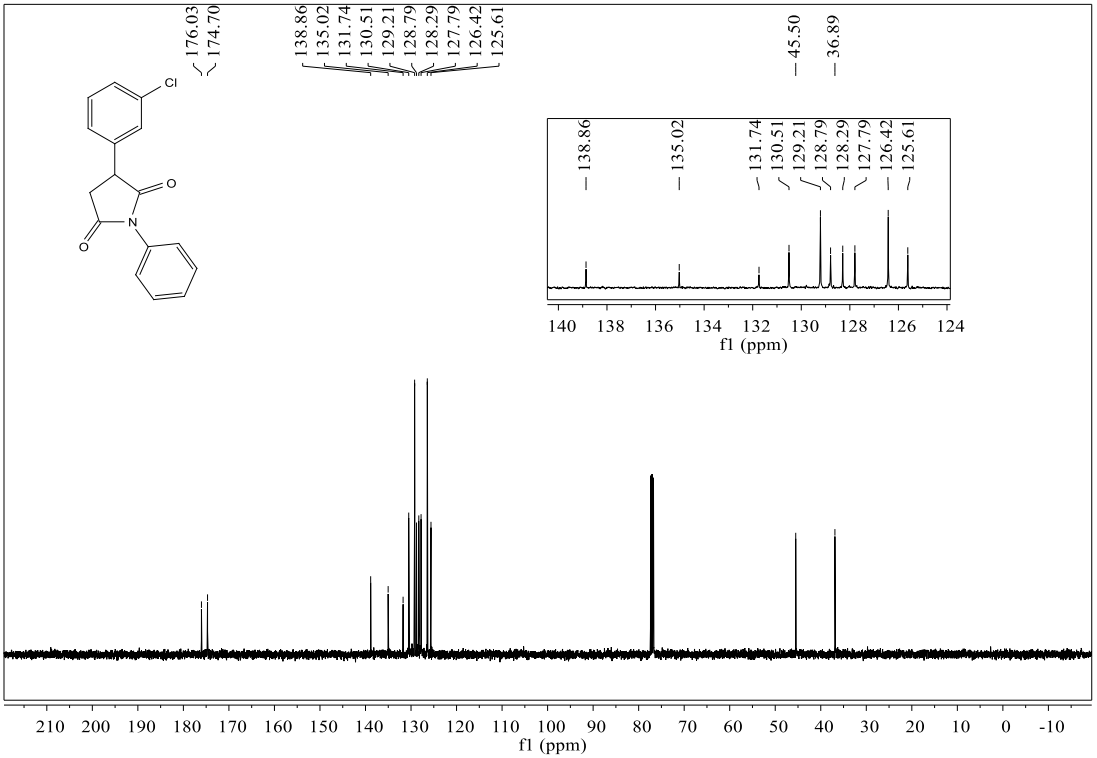
^{13}C NMR spectrum of compound 2aj (101 MHz, $CDCl_3$)



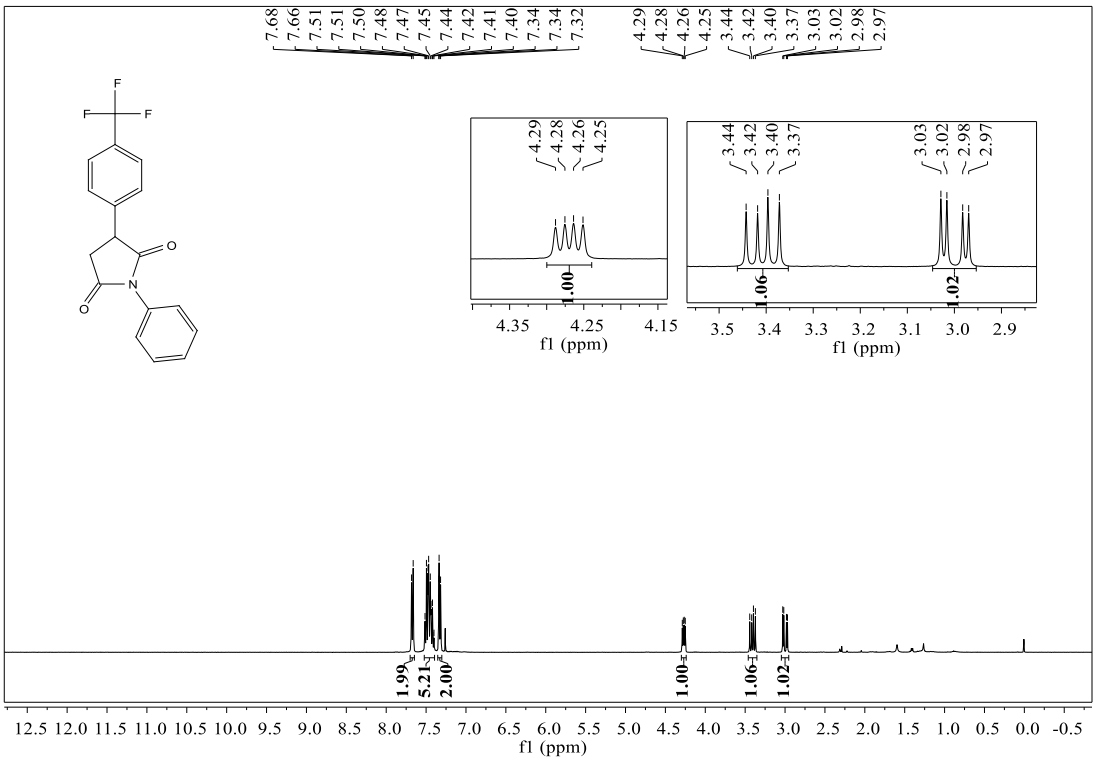
^{1}H NMR spectrum of compound 2am (400 MHz, $CDCl_3$)



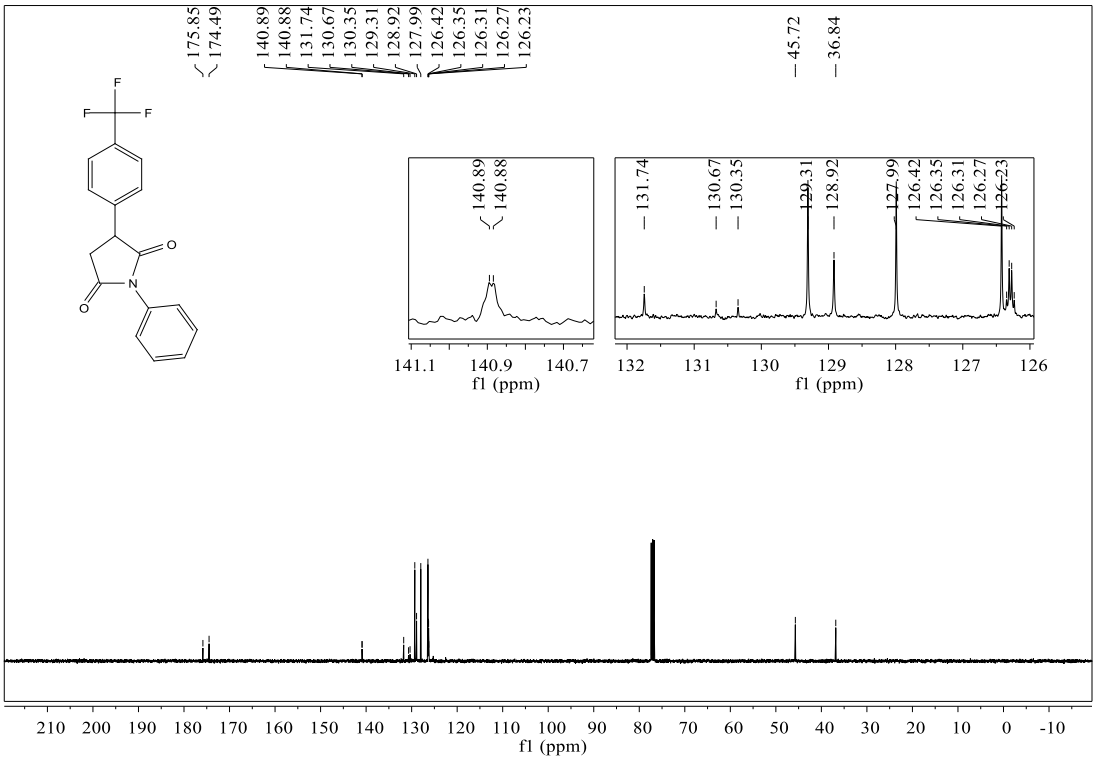
^{13}C NMR spectrum of compound 2am (101 MHz, $CDCl_3$)



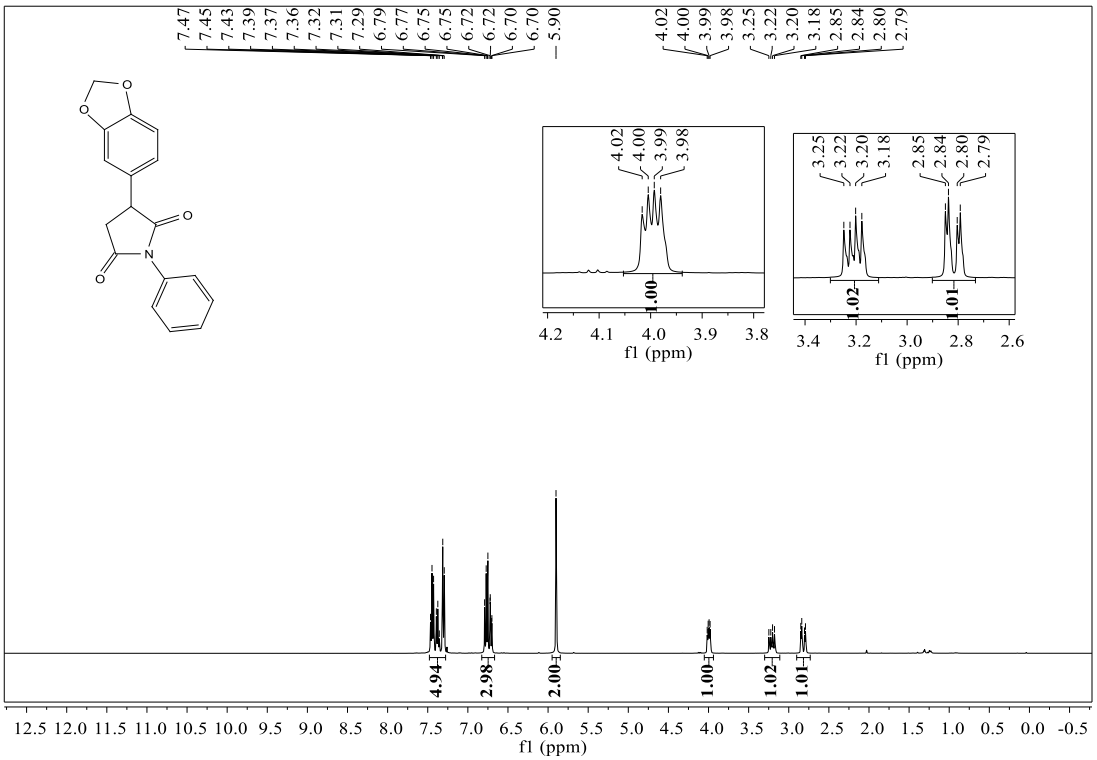
^{1}H NMR spectrum of compound 3aq (400 MHz, $CDCl_3$)



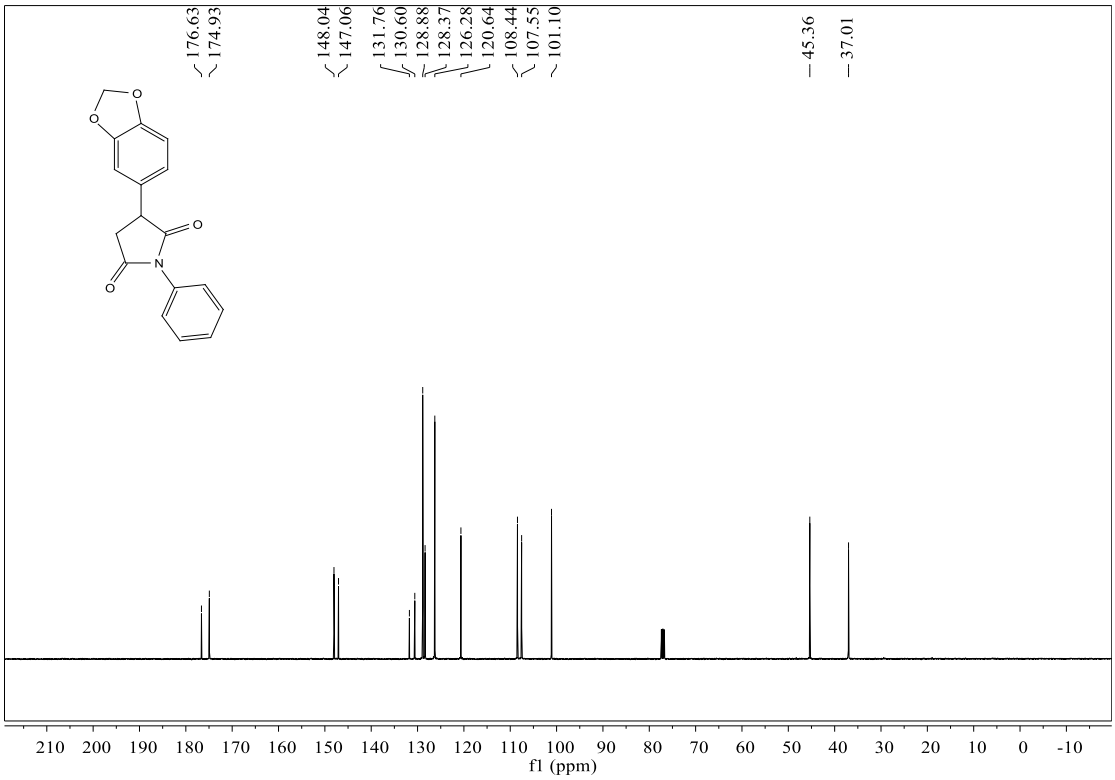
^{13}C NMR spectrum of compound 3aq (101 MHz, $CDCl_3$)



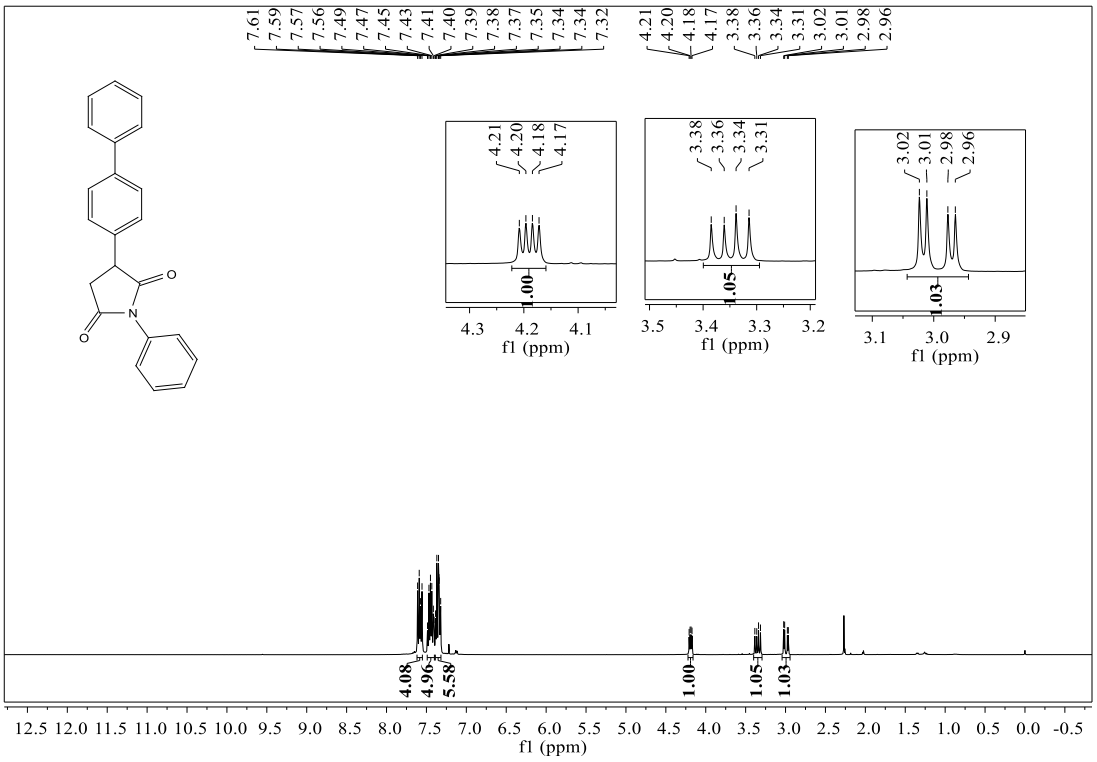
^{1}H NMR spectrum of compound 3ar (400 MHz, $CDCl_3$)



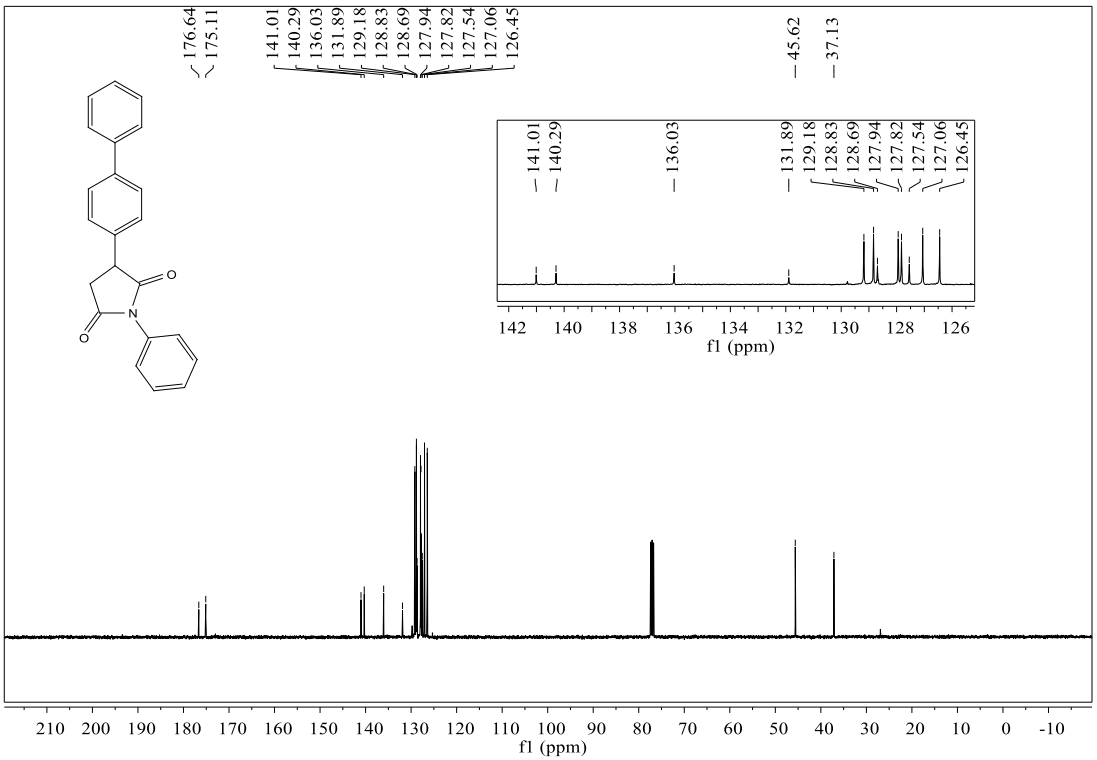
^{13}C NMR spectrum of compound 3ar (101 MHz, $CDCl_3$)



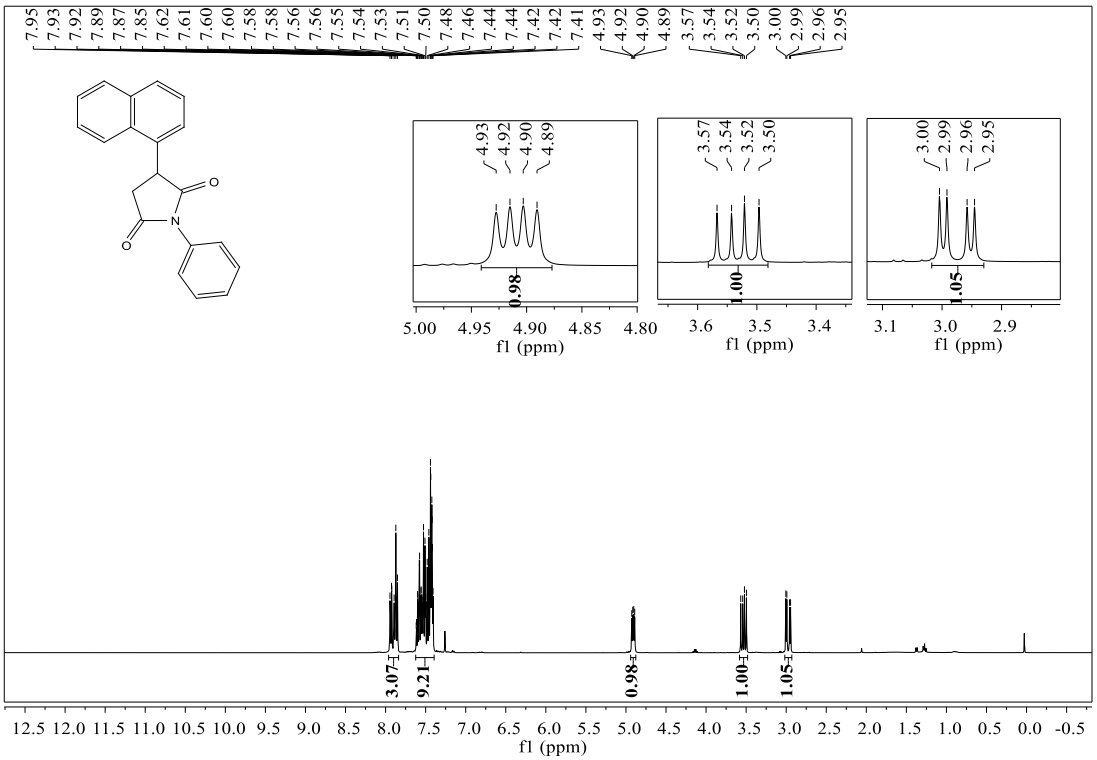
^{1}H NMR spectrum of compound 2as (400 MHz, $CDCl_3$)



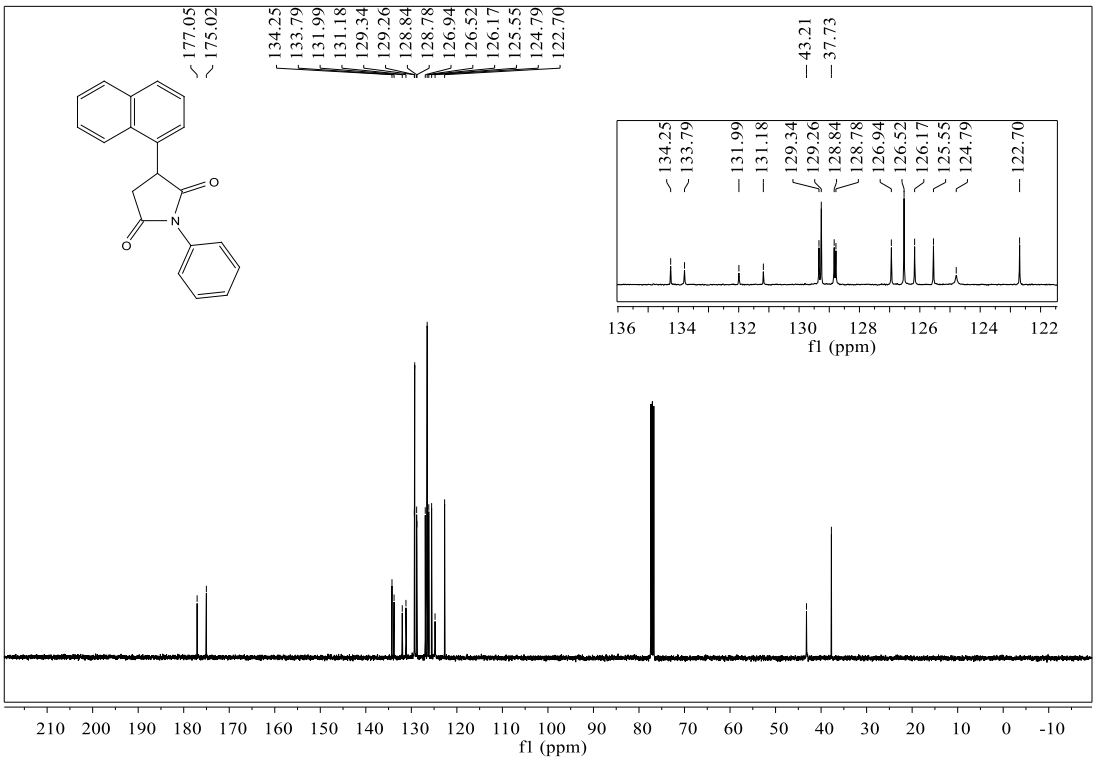
^{13}C NMR spectrum of compound 2as (101 MHz, $CDCl_3$)



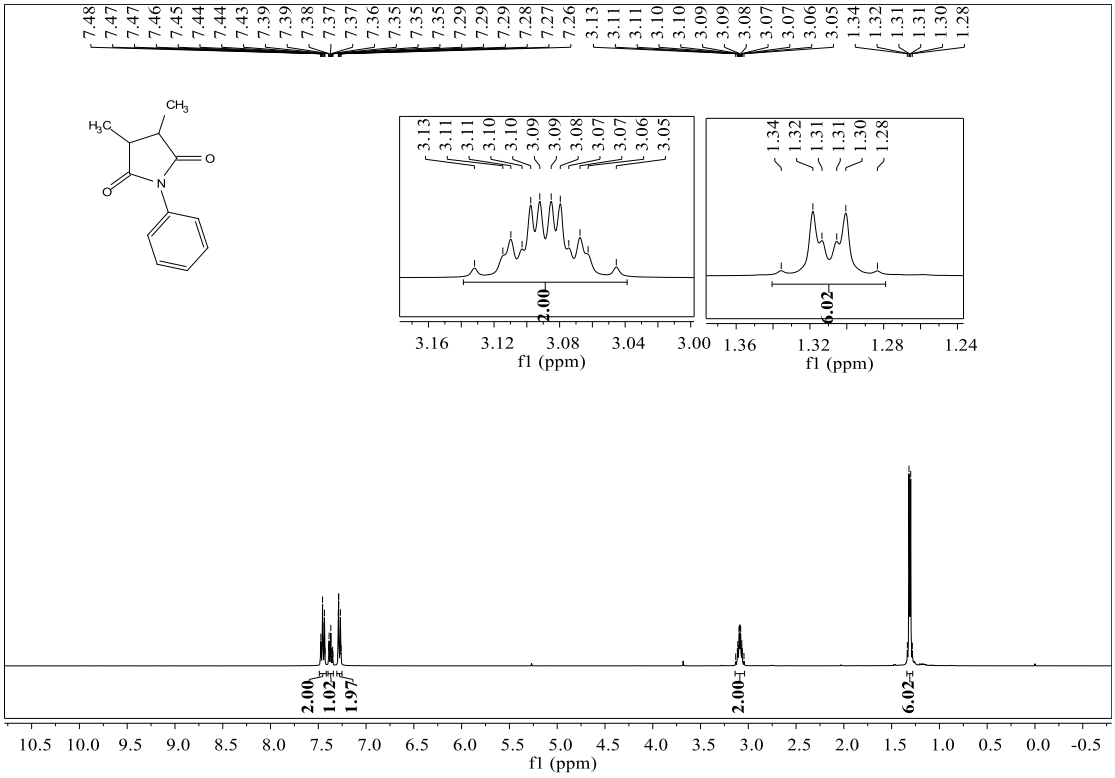
^{1}H NMR spectrum of compound 2at (400 MHz, $CDCl_3$)



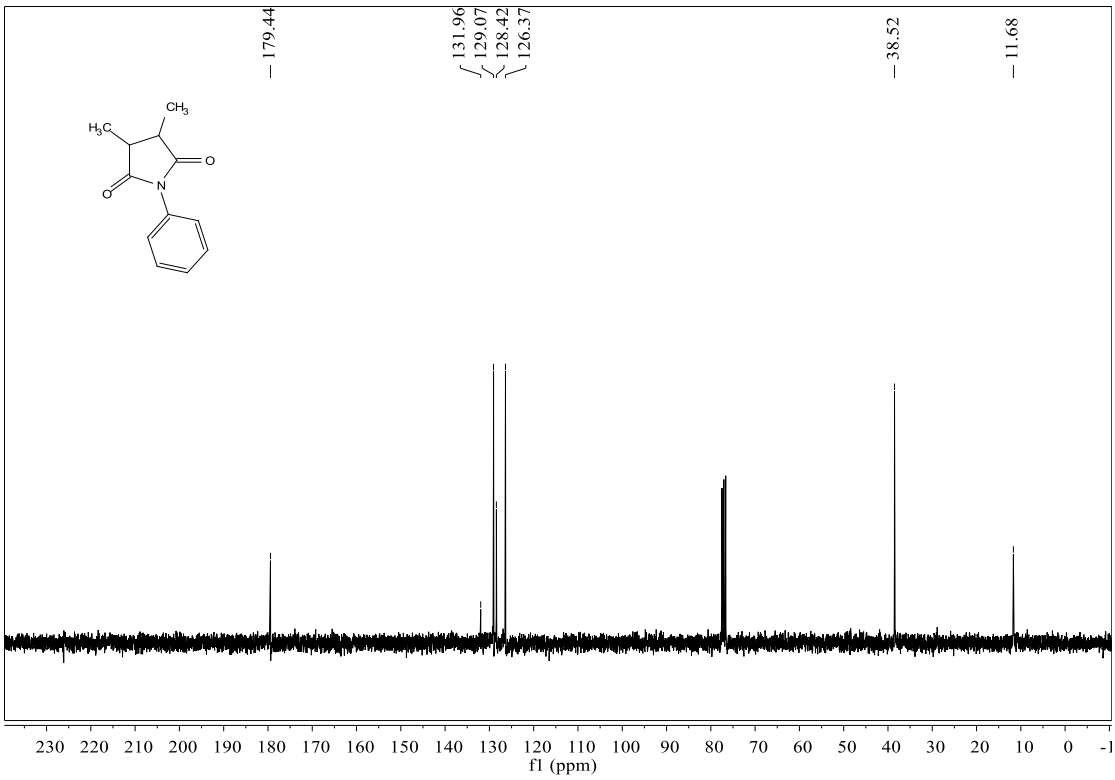
^{13}C NMR spectrum of compound 2at (101 MHz, $CDCl_3$)



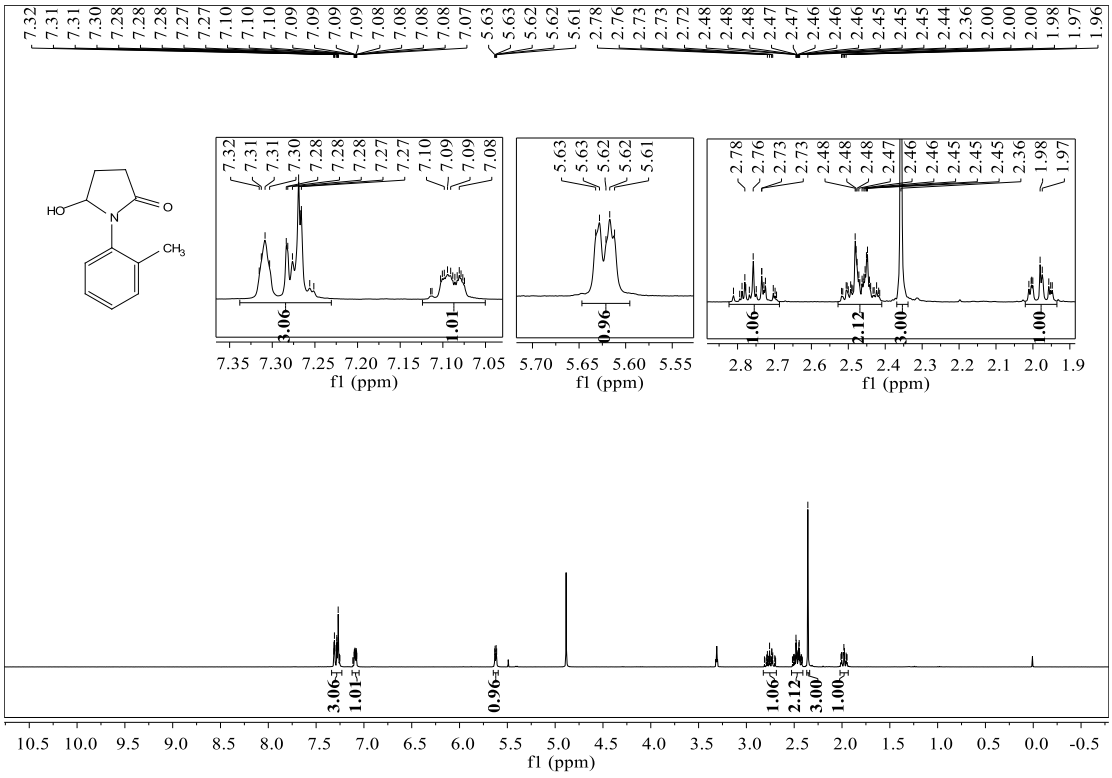
^{1}H NMR spectrum of compound 2az (400 MHz, $CDCl_3$)



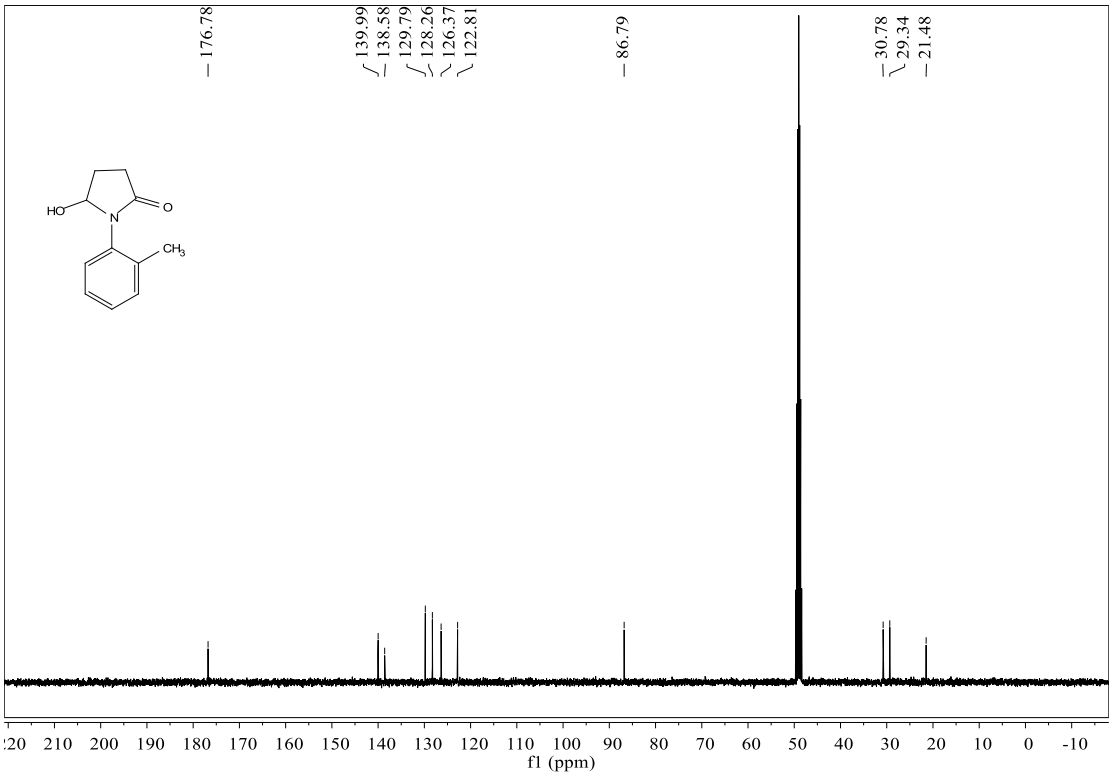
^{13}C NMR spectrum of compound 2az (76 MHz, $CDCl_3$)



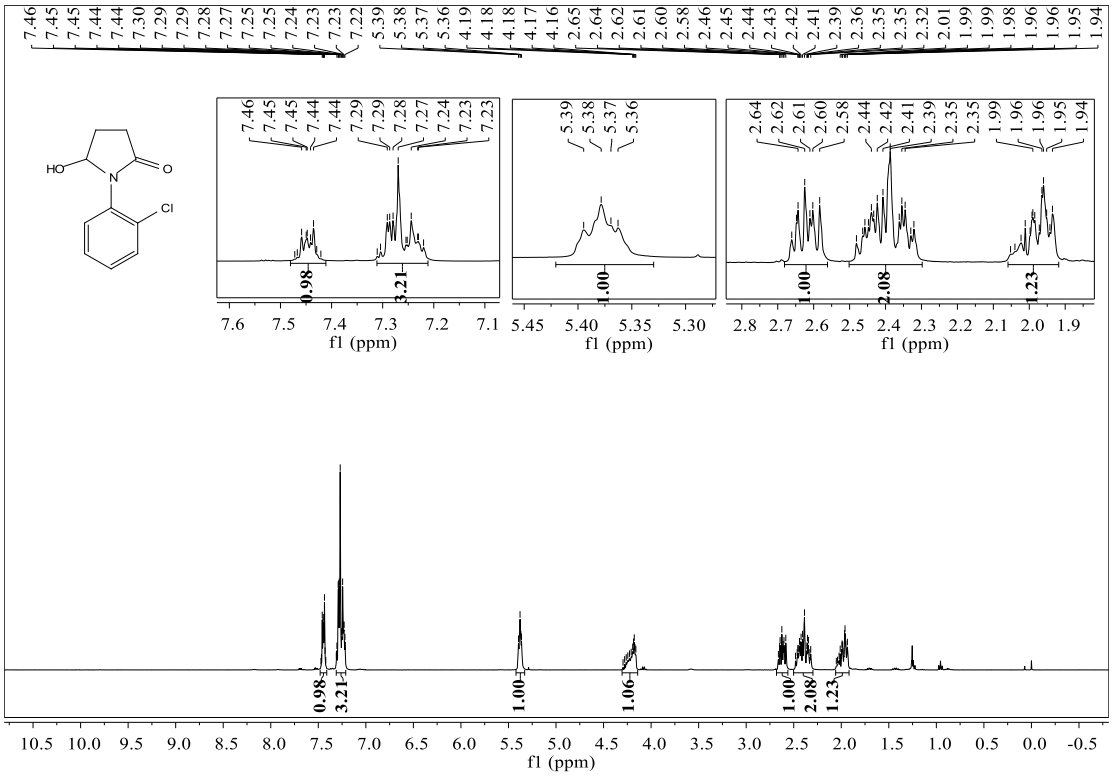
^{1}H NMR spectrum of compound 1b (400 MHz, methanol-d_4)



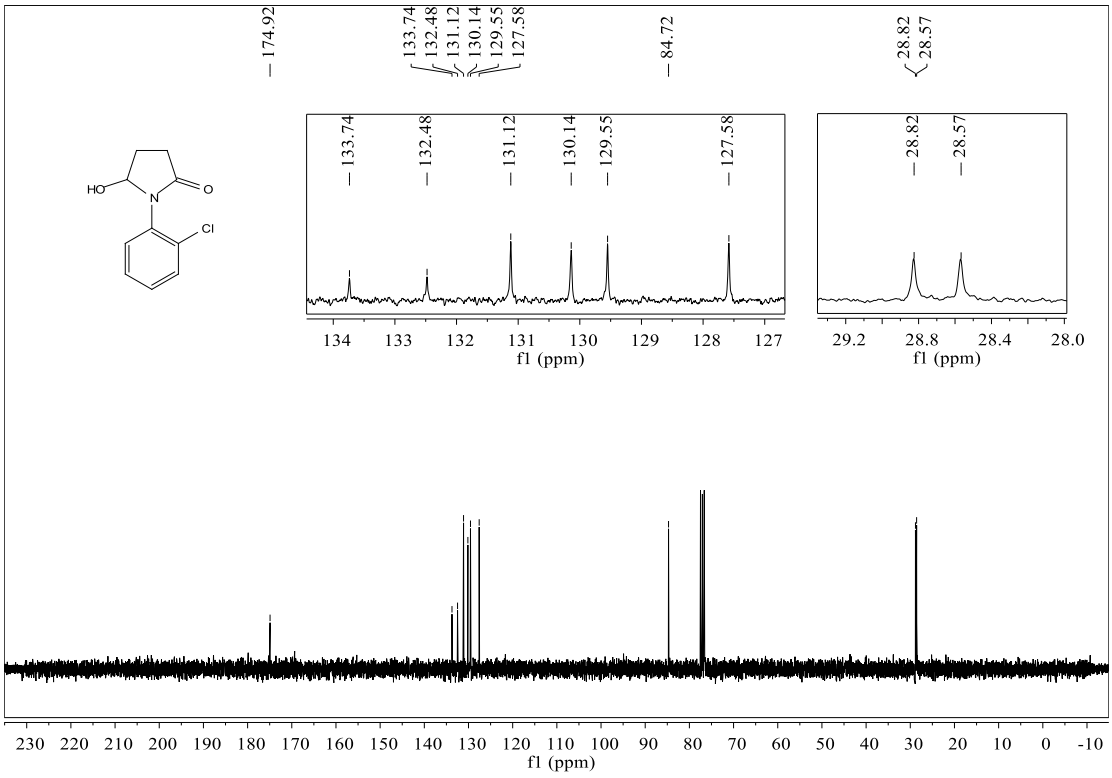
^{13}C NMR spectrum of compound 1b (101 MHz, methanol-d_4)



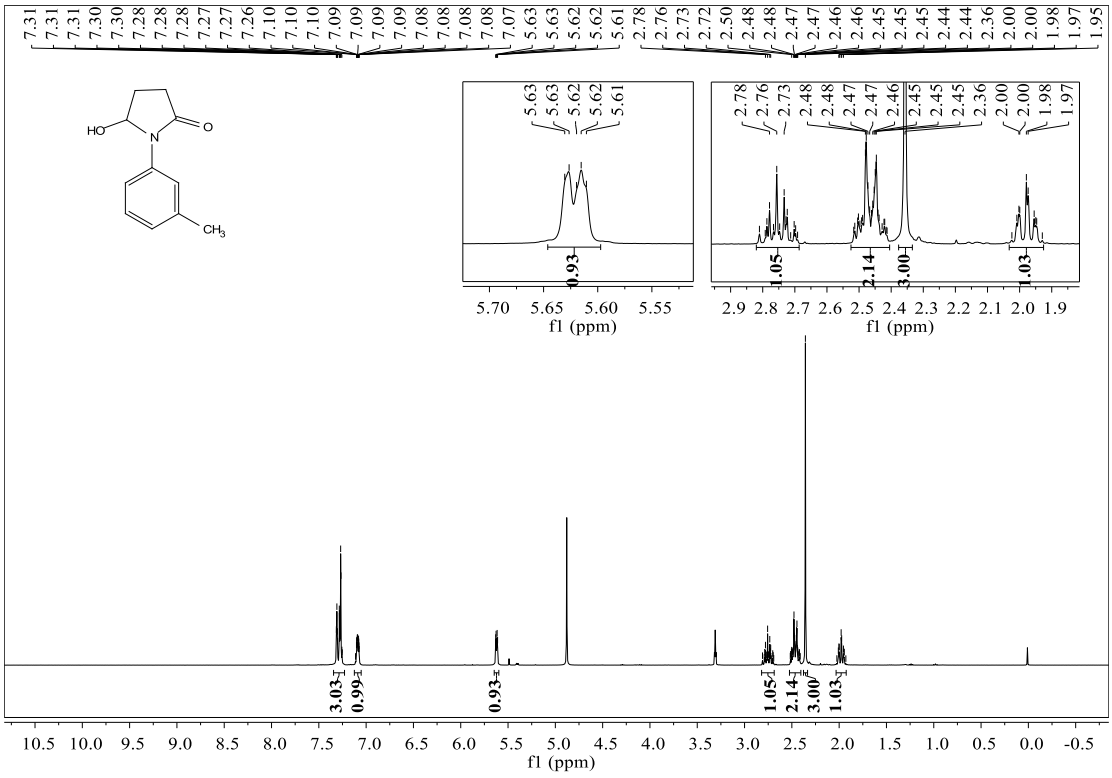
^{1}H NMR spectrum of compound 1c (400 MHz, $CDCl_3$)



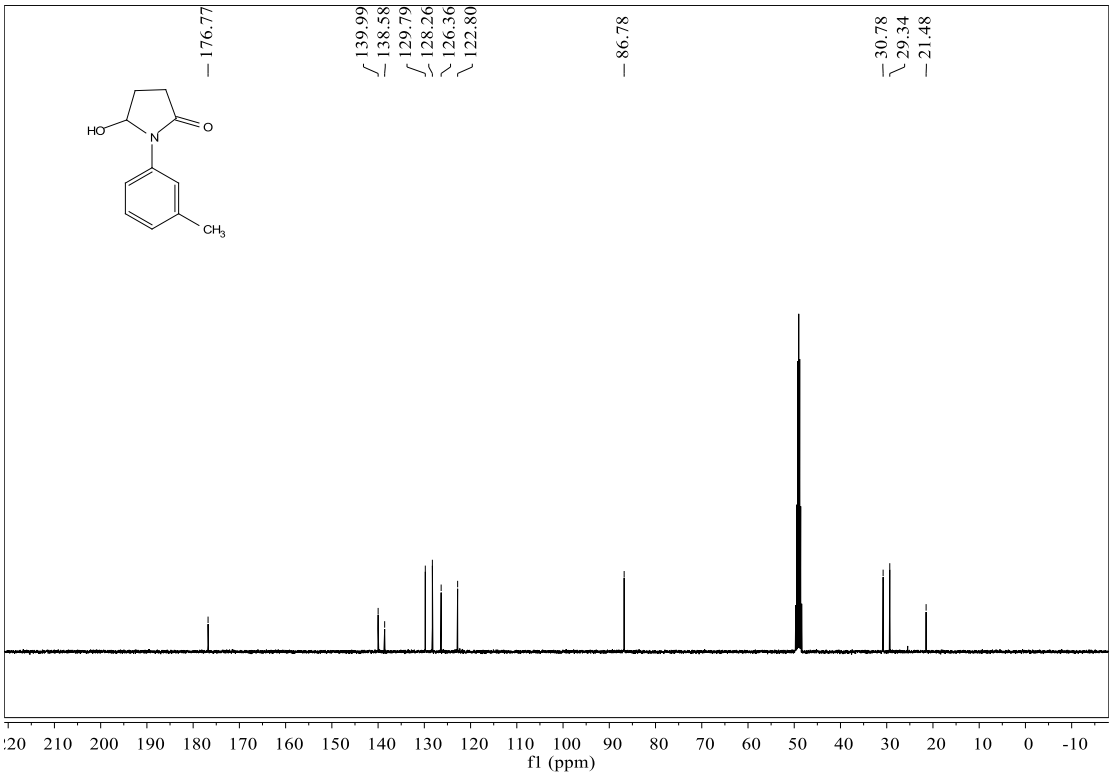
^{13}C NMR spectrum of compound 1c (76 MHz, $CDCl_3$)



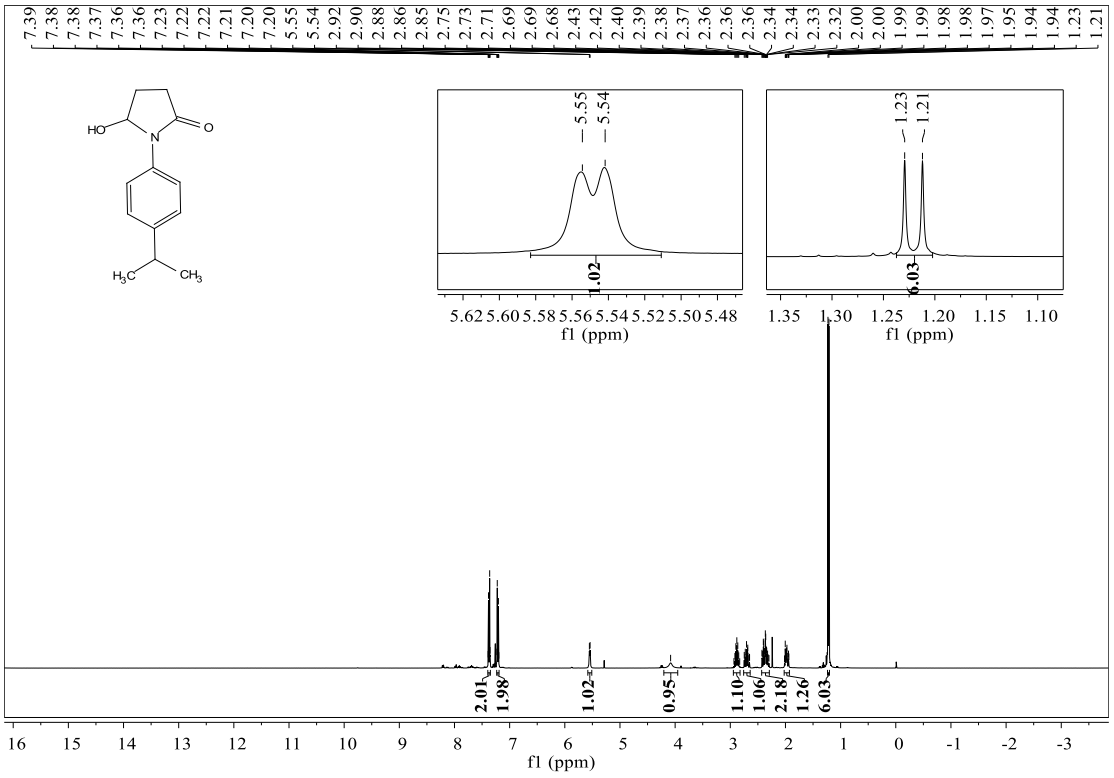
^{1}H NMR spectrum of compound 1f (400 MHz, methanol-d_4)



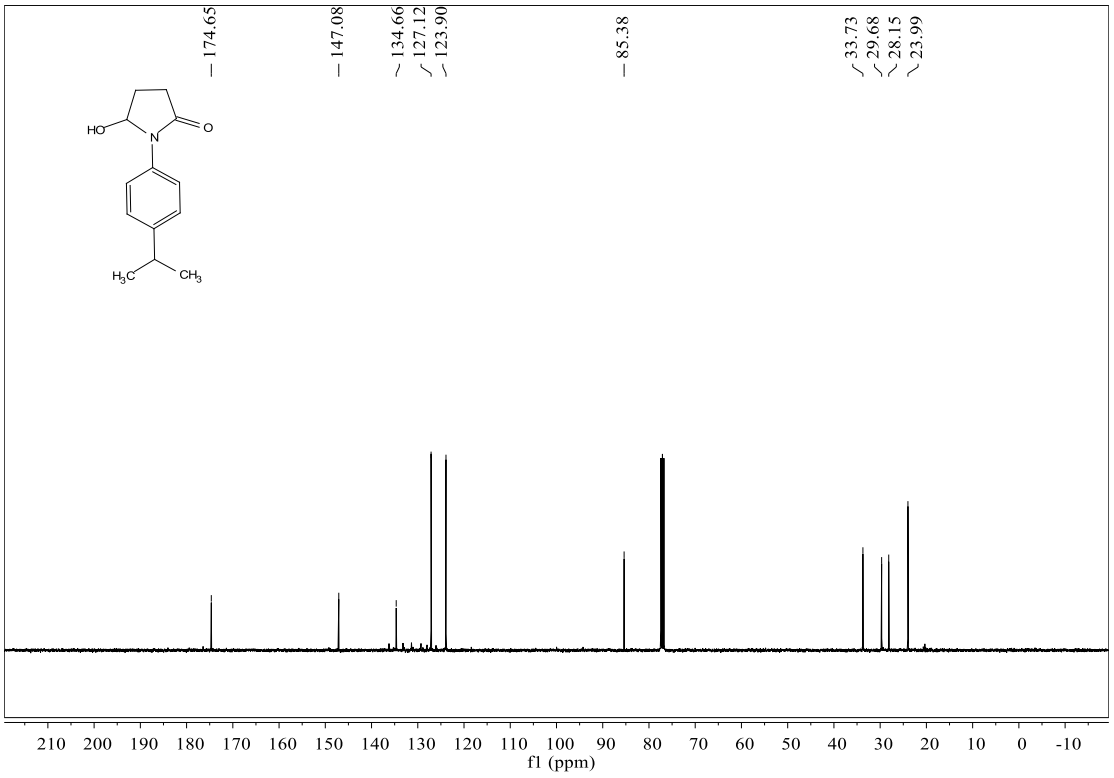
^{13}C NMR spectrum of compound 1f (101 MHz, methanol-d_4)



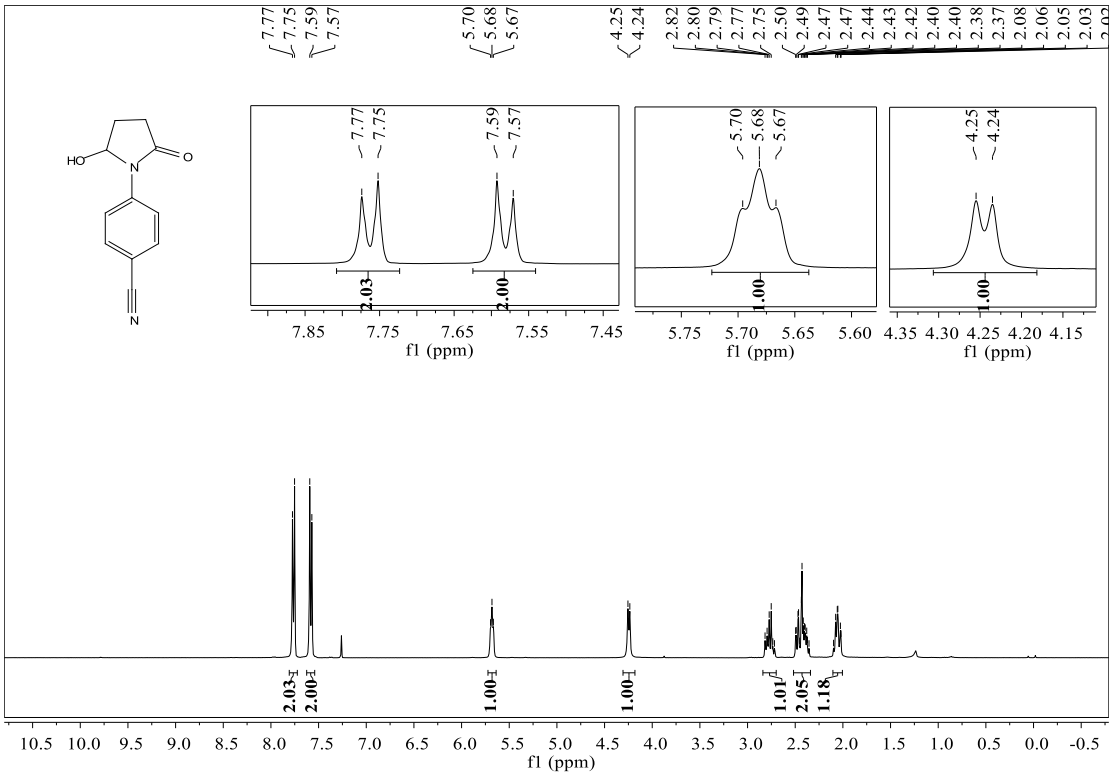
^{1}H NMR spectrum of compound 1j (400 MHz, $CDCl_3$)



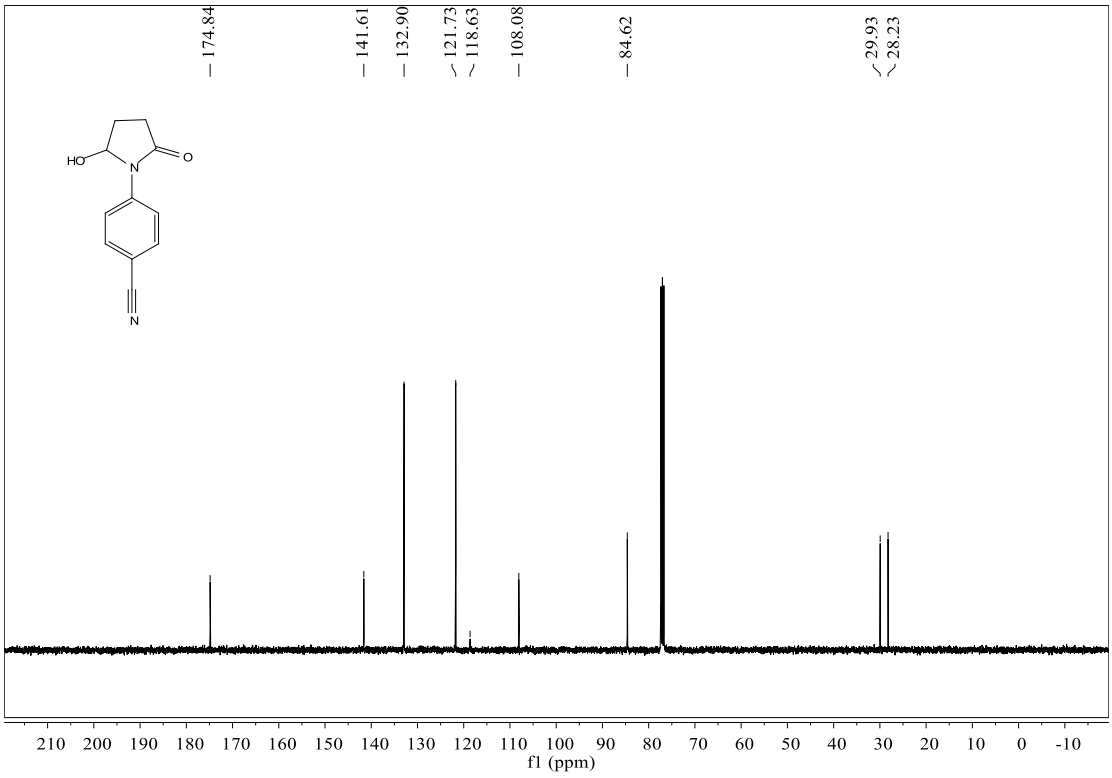
^{13}C NMR spectrum of compound 1j (101 MHz, $CDCl_3$)



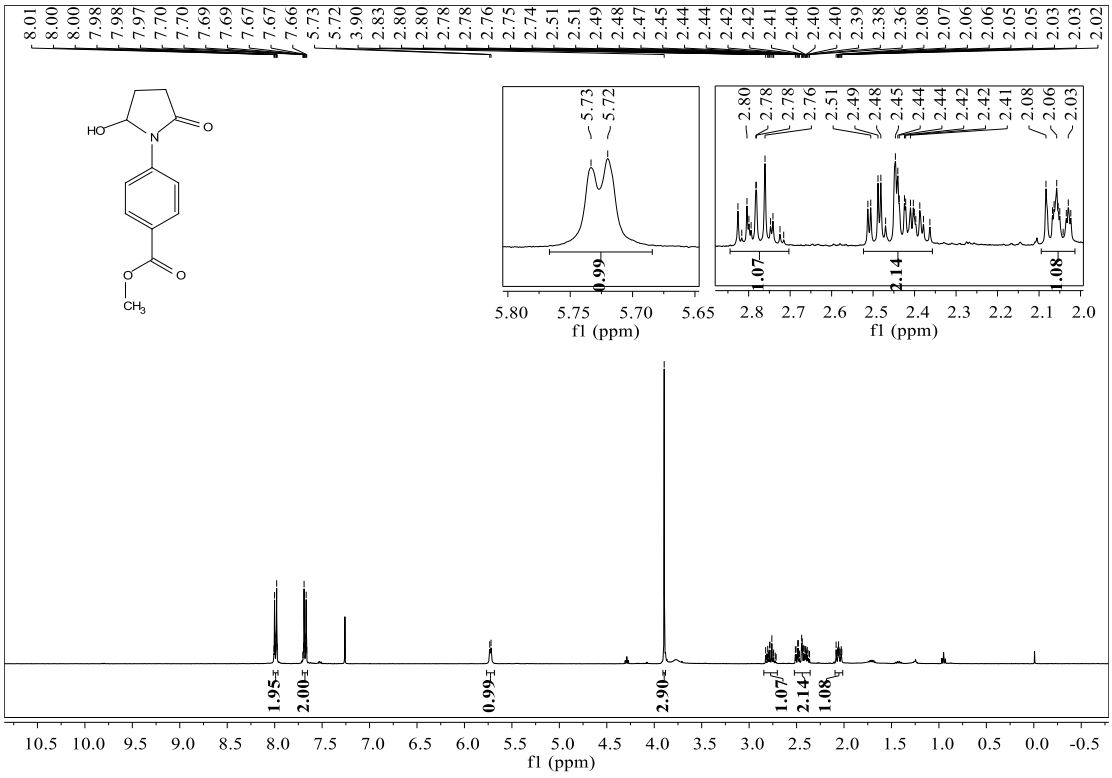
^{1}H NMR spectrum of compound 1p (400 MHz, $CDCl_3$)



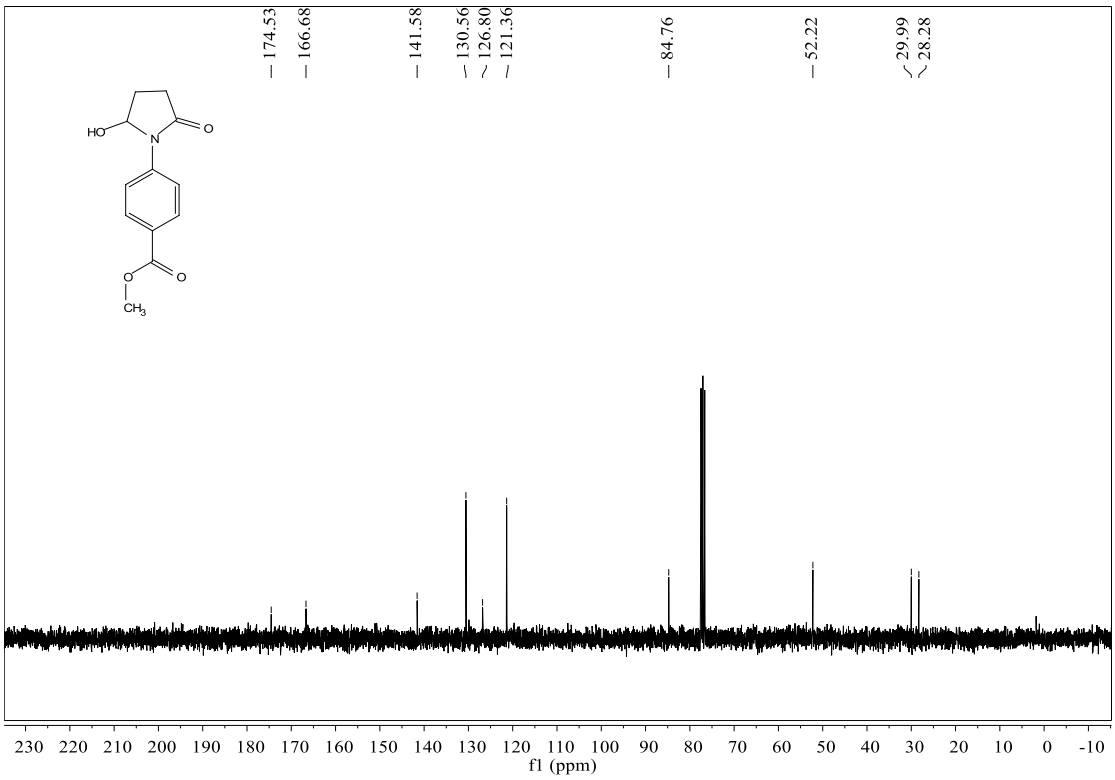
^{13}C NMR spectrum of compound 1p (101 MHz, $CDCl_3$)



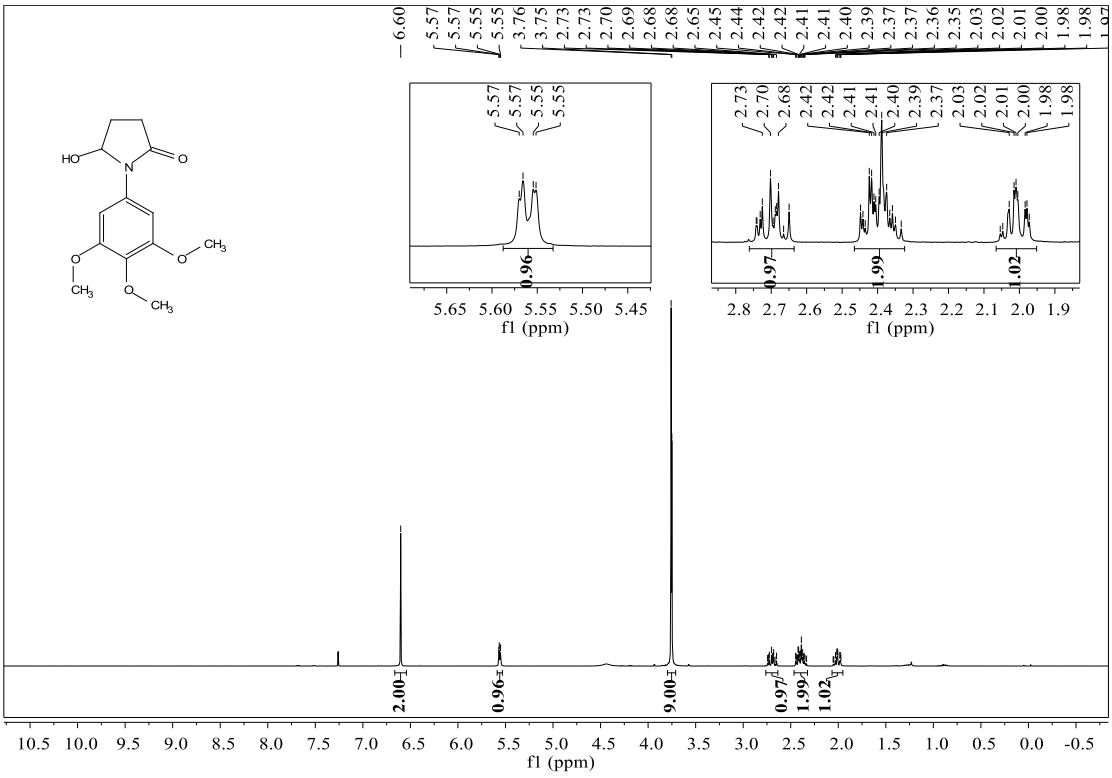
^{1}H NMR spectrum of compound 1q (400 MHz, $CDCl_3$)



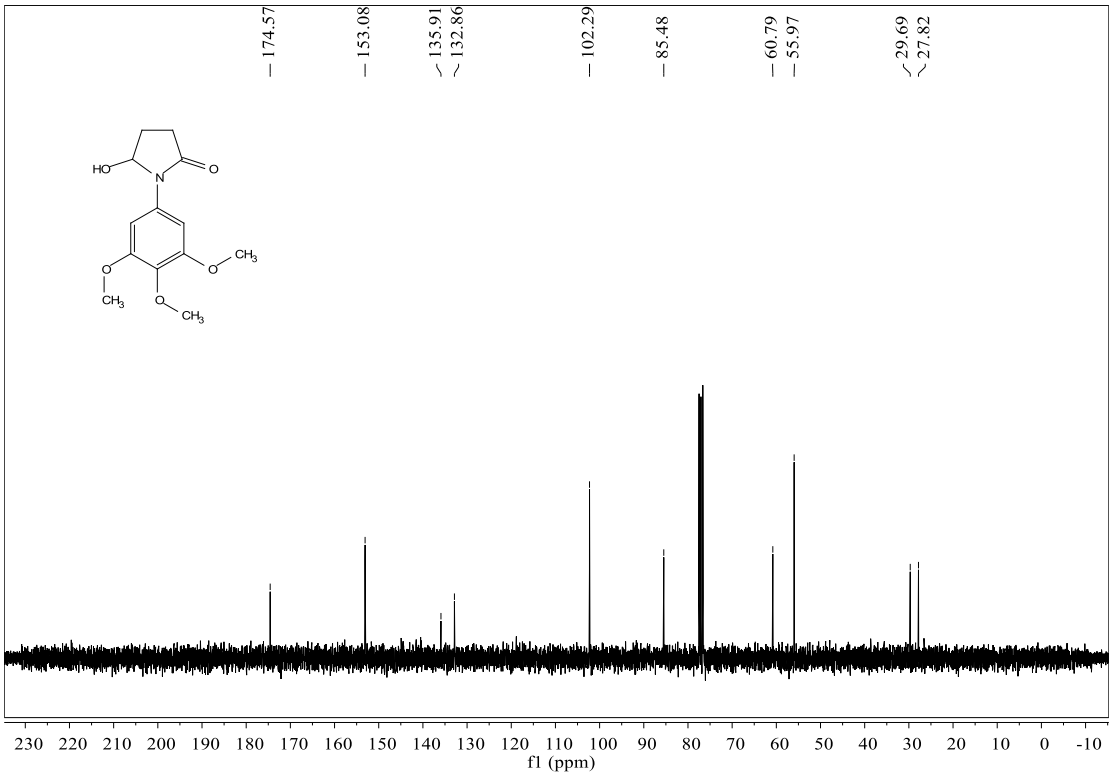
^{13}C NMR spectrum of compound 1q (76 MHz, $CDCl_3$)



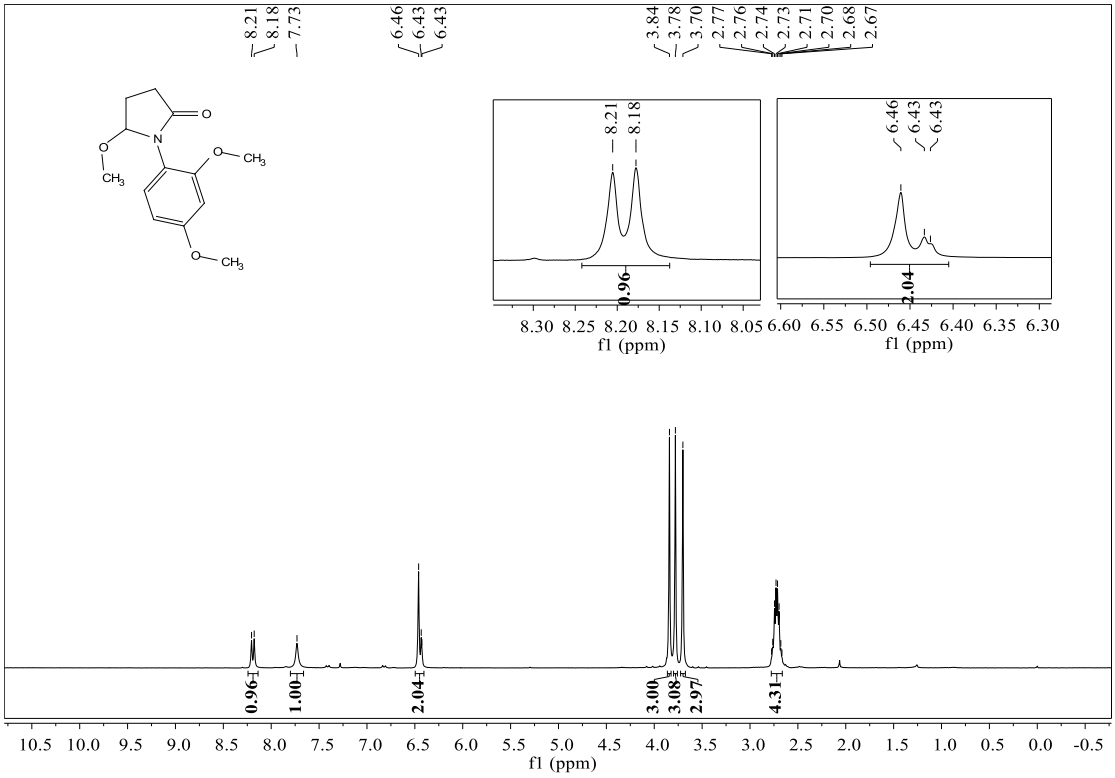
^{1}H NMR spectrum of compound 1s (400 MHz, $CDCl_3$)



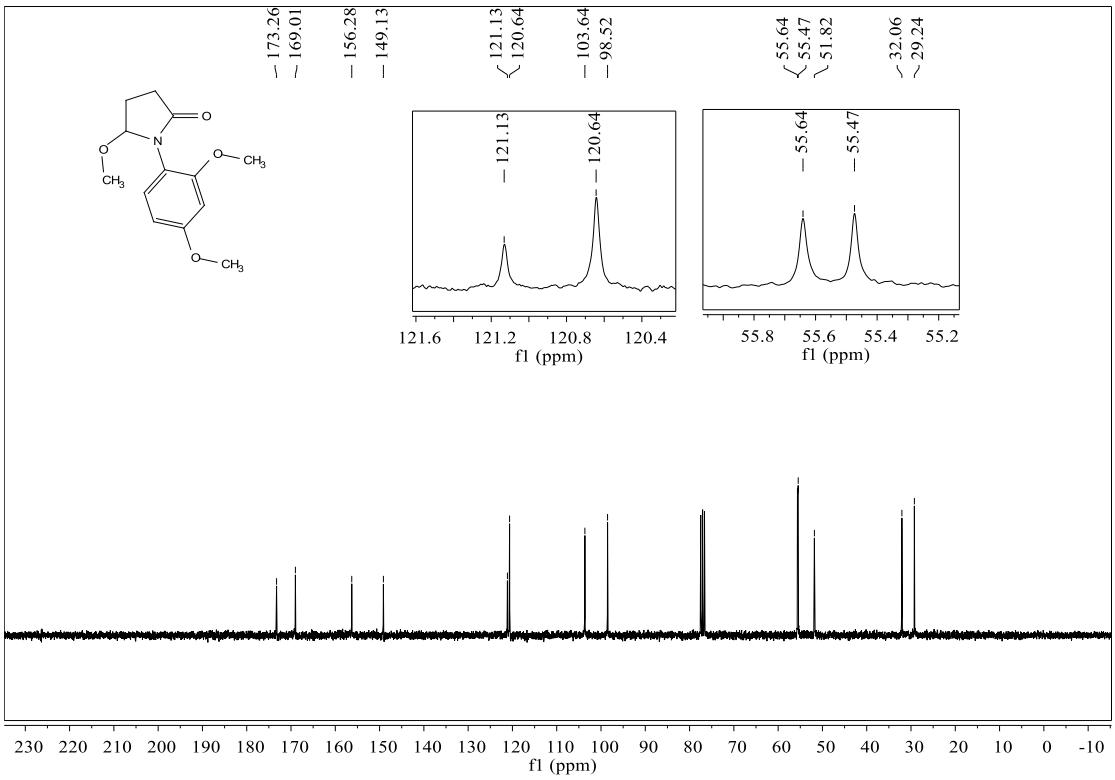
^{13}C NMR spectrum of compound 1s (76 MHz, $CDCl_3$)



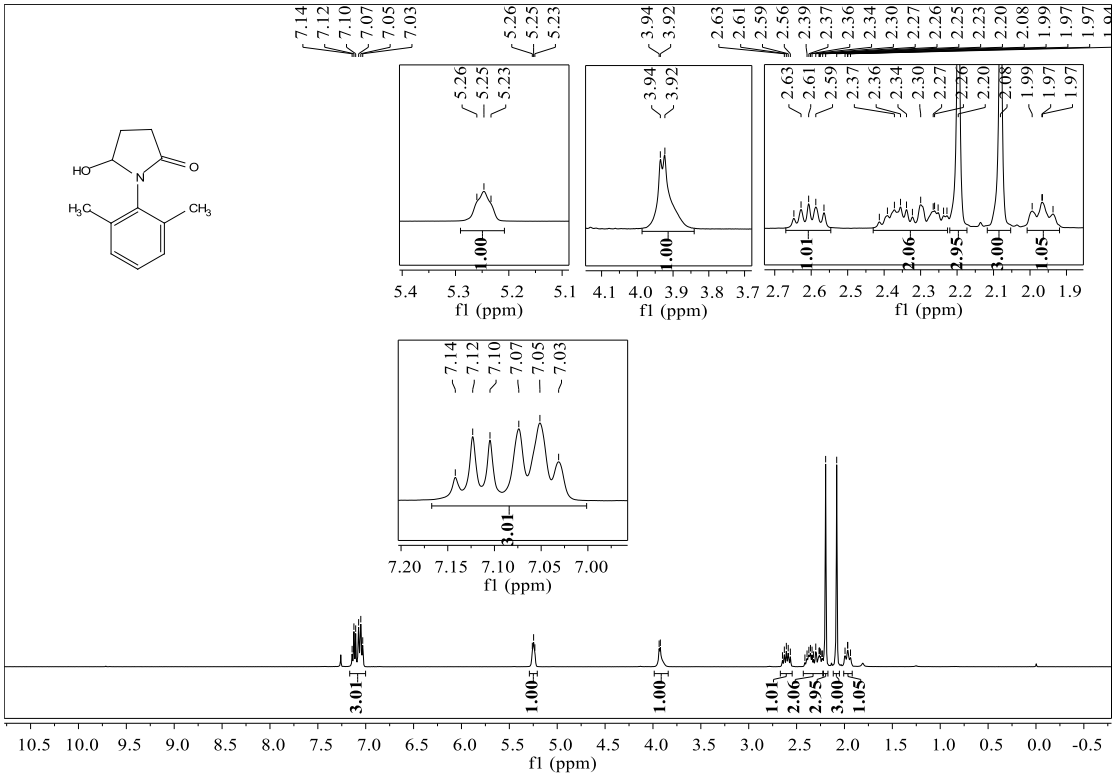
^{1}H NMR spectrum of compound 1t (300 MHz, $CDCl_3$)



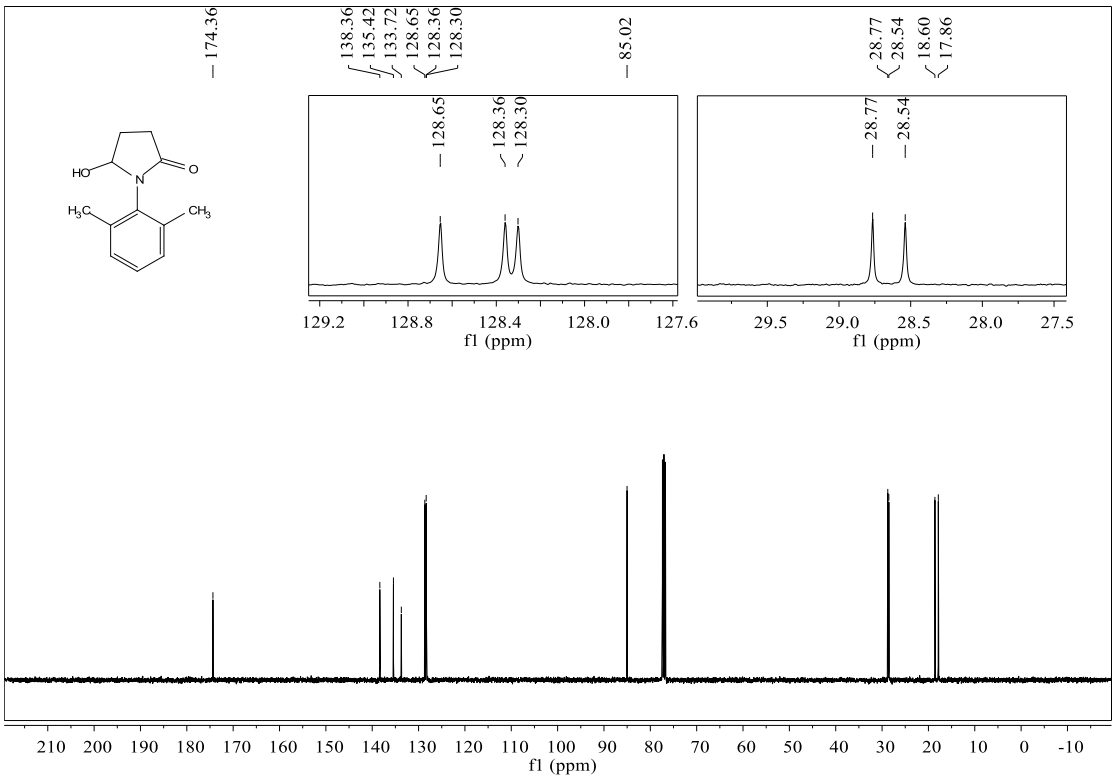
^{13}C NMR spectrum of compound 1t (76 MHz, $CDCl_3$)



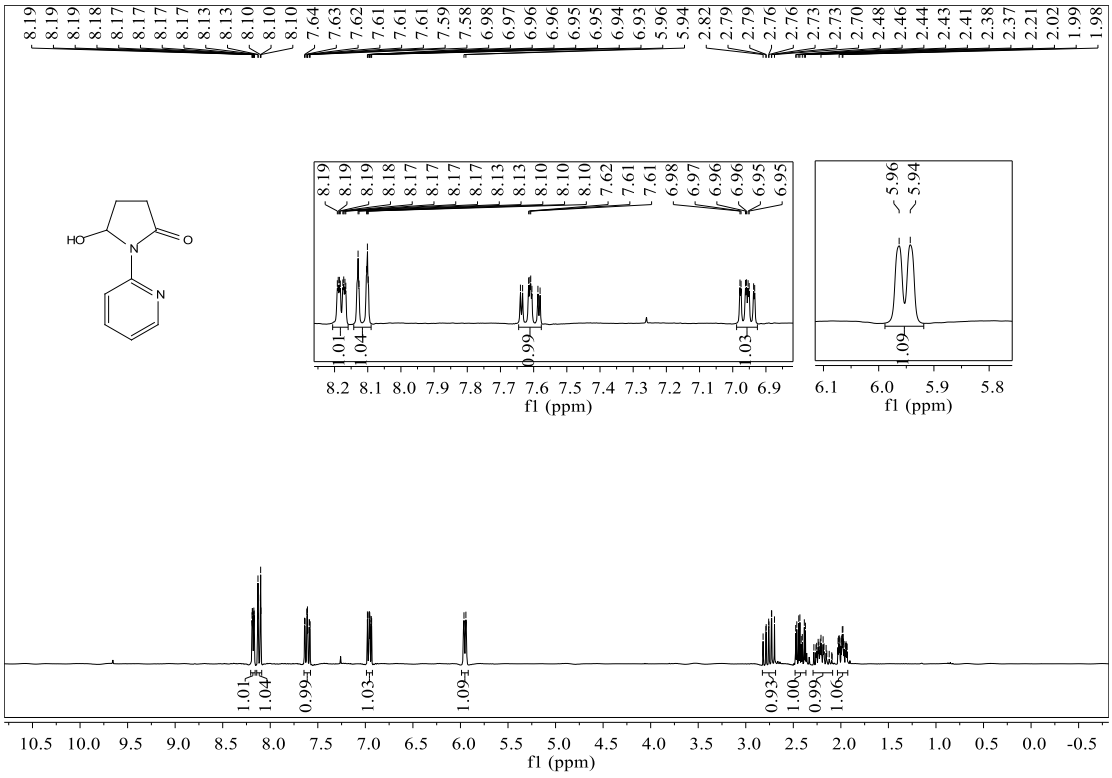
^{1}H NMR spectrum of compound 1u (400 MHz, $CDCl_3$)



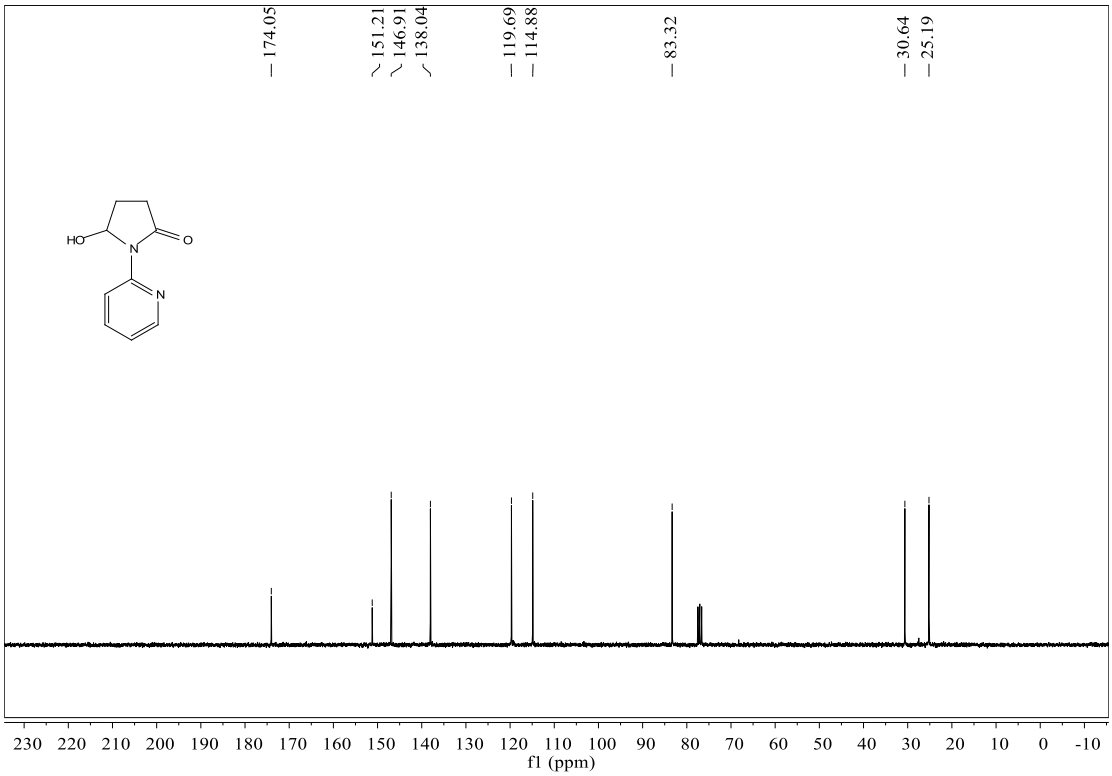
^{13}C NMR spectrum of compound 1u (101 MHz, $CDCl_3$)



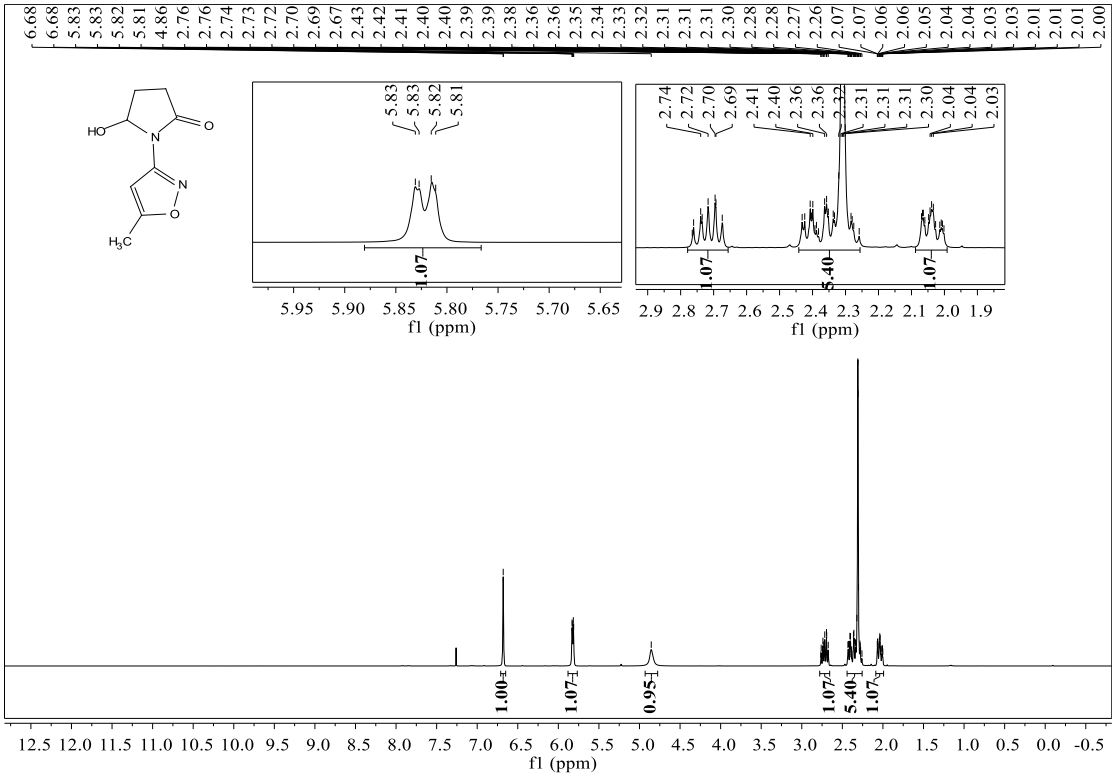
^{1}H NMR spectrum of compound 1v (300 MHz, $CDCl_3$)



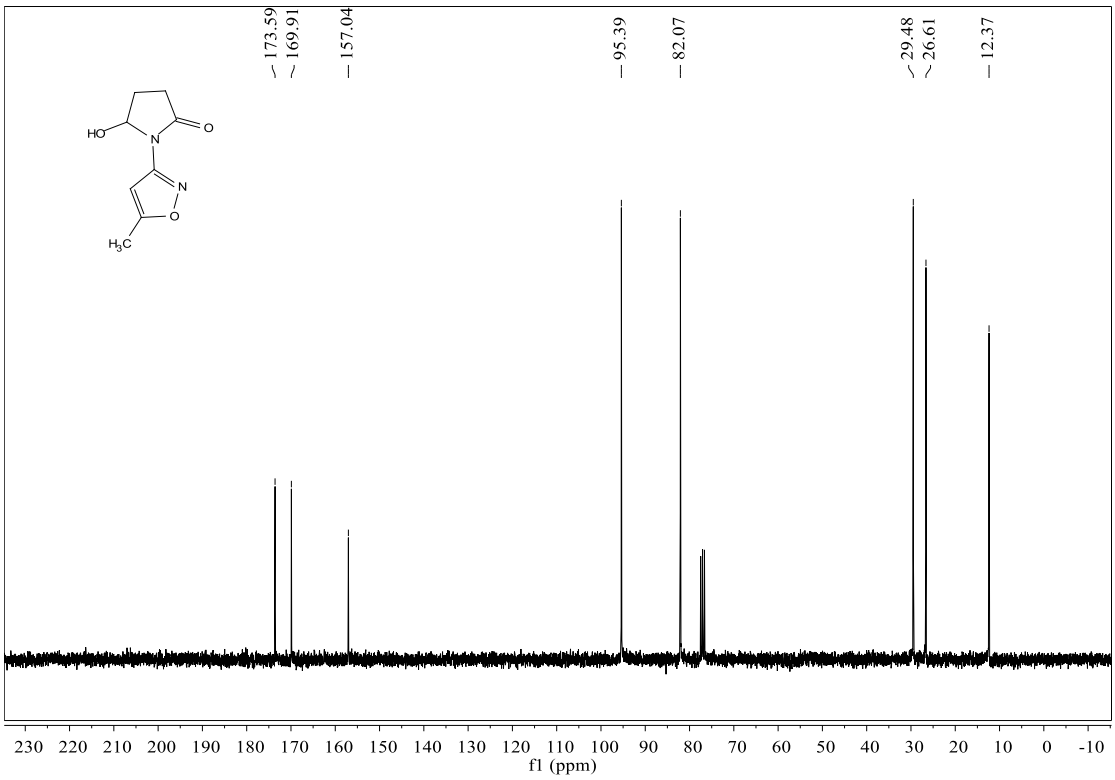
^{13}C NMR spectrum of compound 1v (76 MHz, $CDCl_3$)



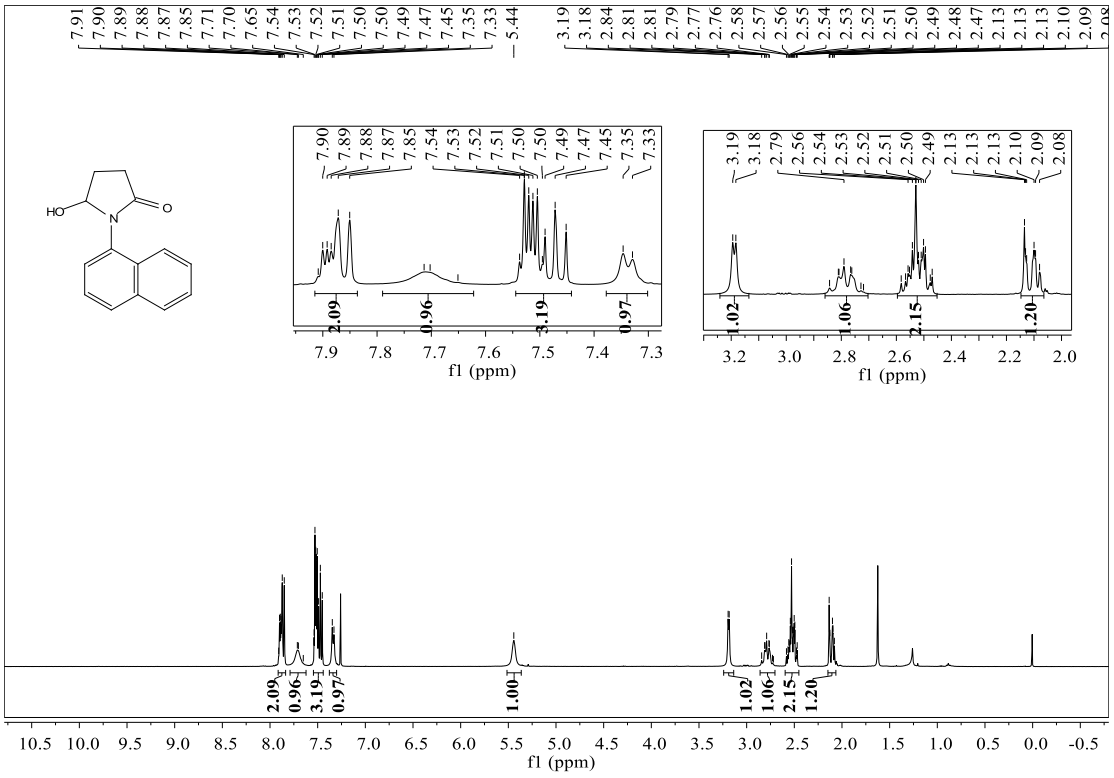
^{1}H NMR spectrum of compound 1w (400 MHz, $CDCl_3$)



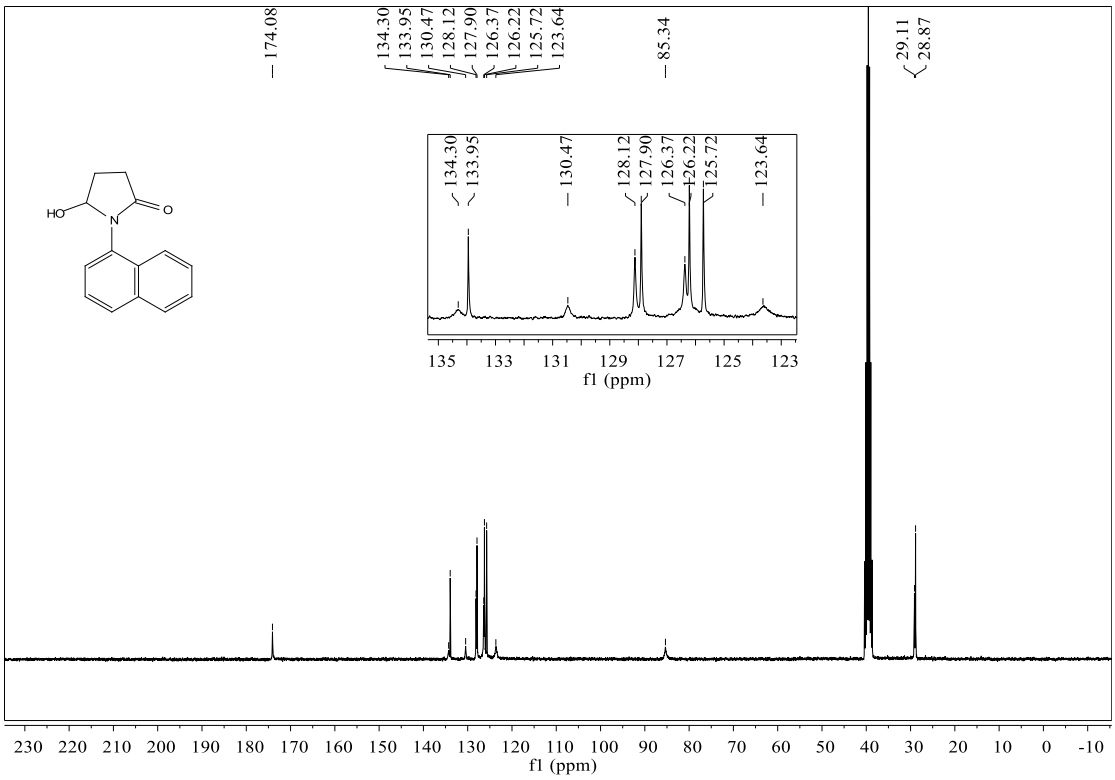
^{13}C NMR spectrum of compound 1w (76 MHz, $CDCl_3$)



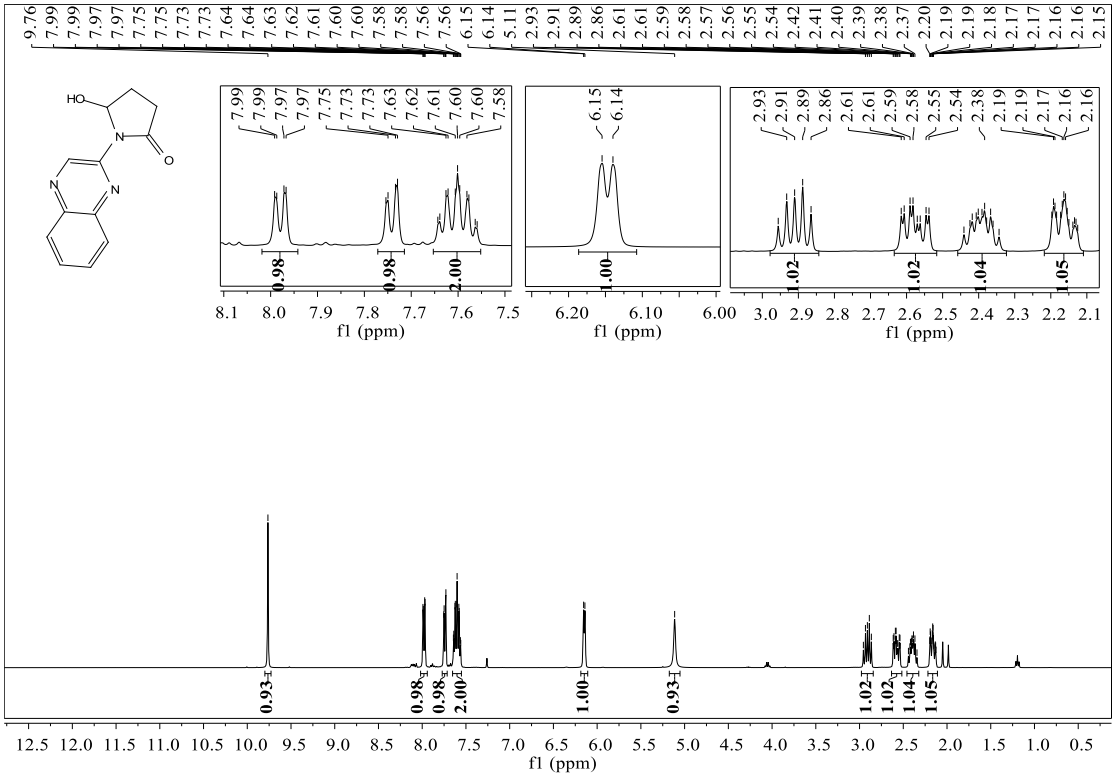
^{1}H NMR spectrum of compound 1x (400 MHz, $CDCl_3$)



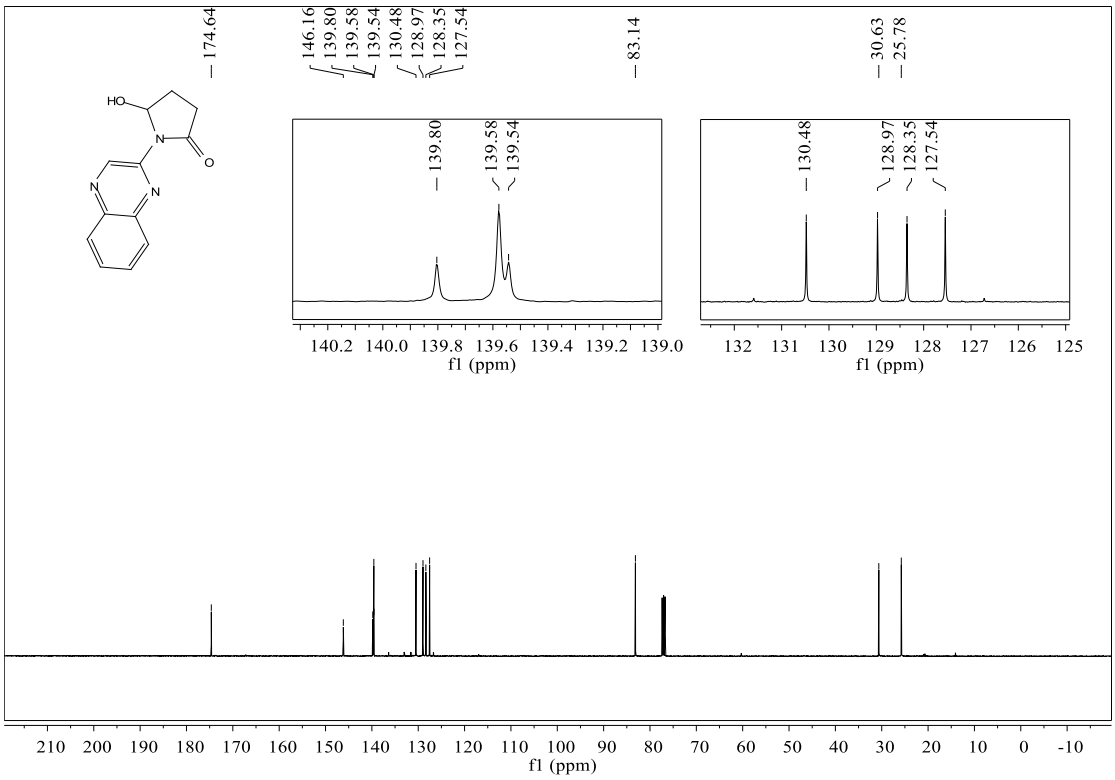
^{13}C NMR spectrum of compound 1x (76 MHz, DMSO-d_6)



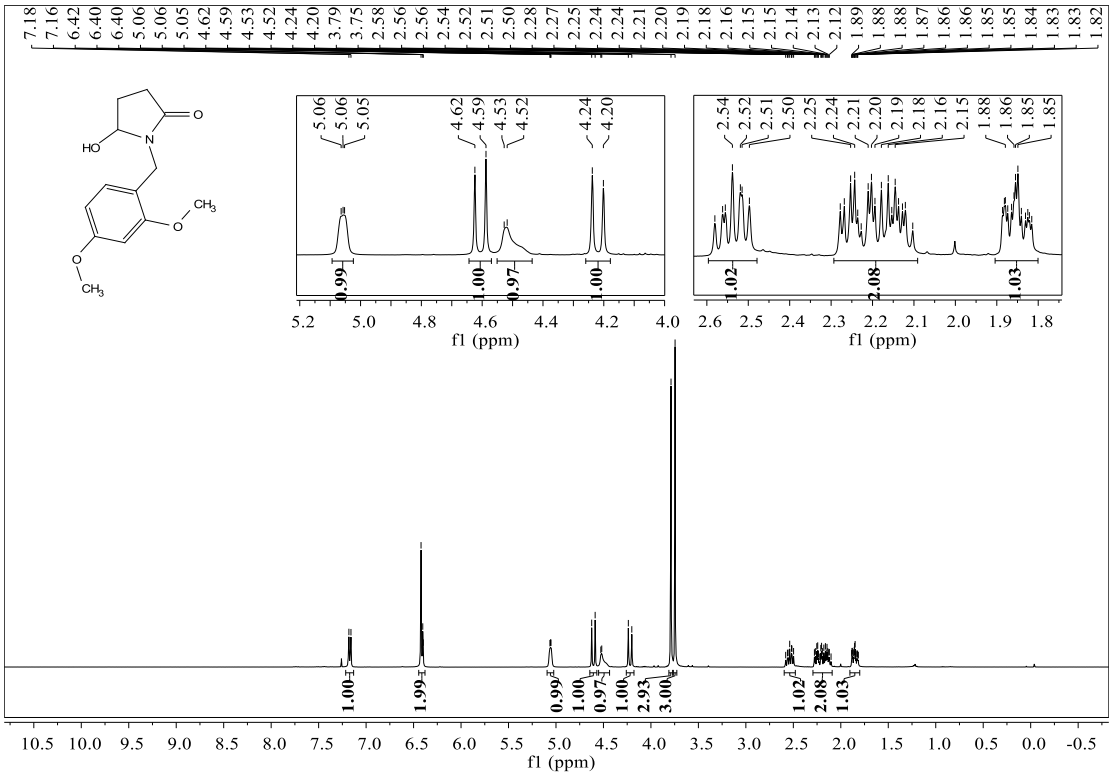
^{1}H NMR spectrum of compound 1y (400 MHz, $CDCl_3$)



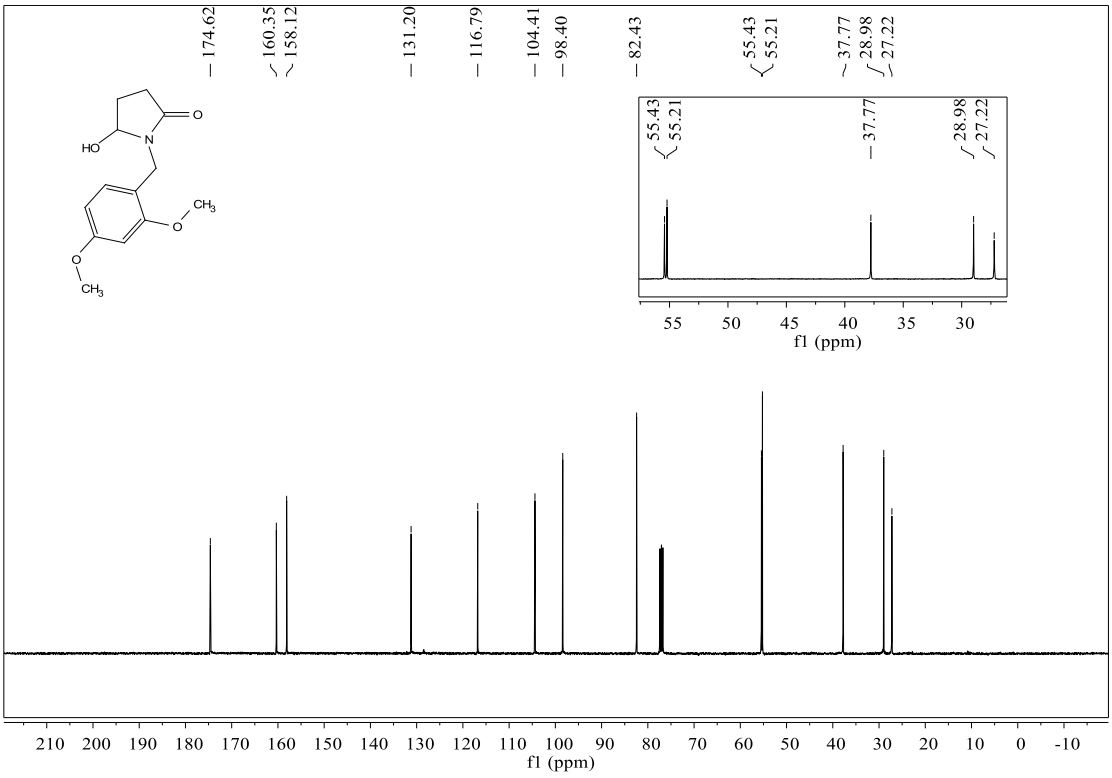
^{13}C NMR spectrum of compound 1y (101 MHz, $CDCl_3$)



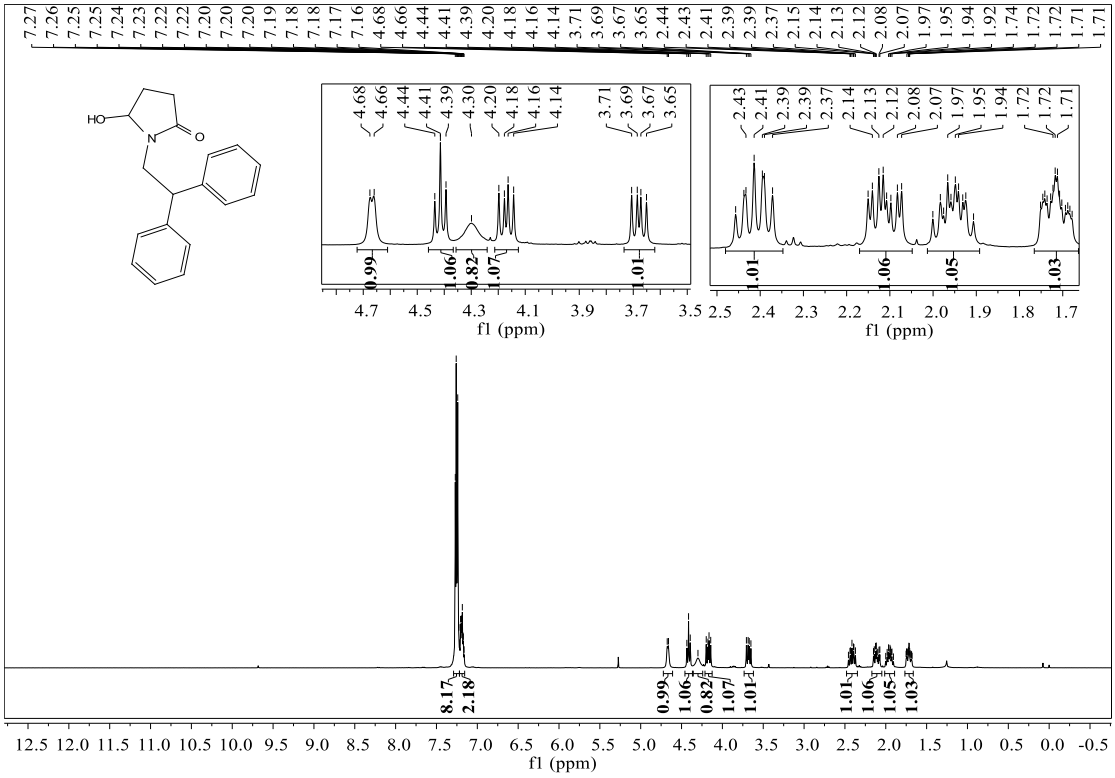
^{1}H NMR spectrum of compound 1aa (400 MHz, $CDCl_3$)



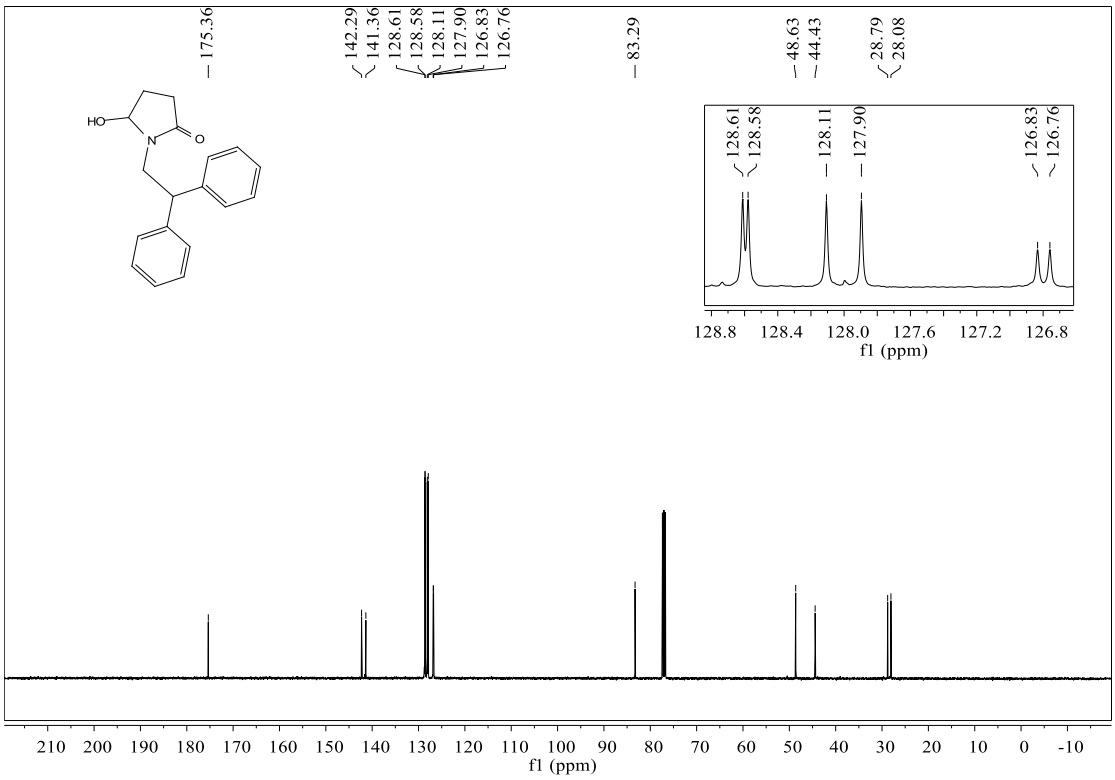
^{13}C NMR spectrum of compound 1aa (101 MHz, $CDCl_3$)



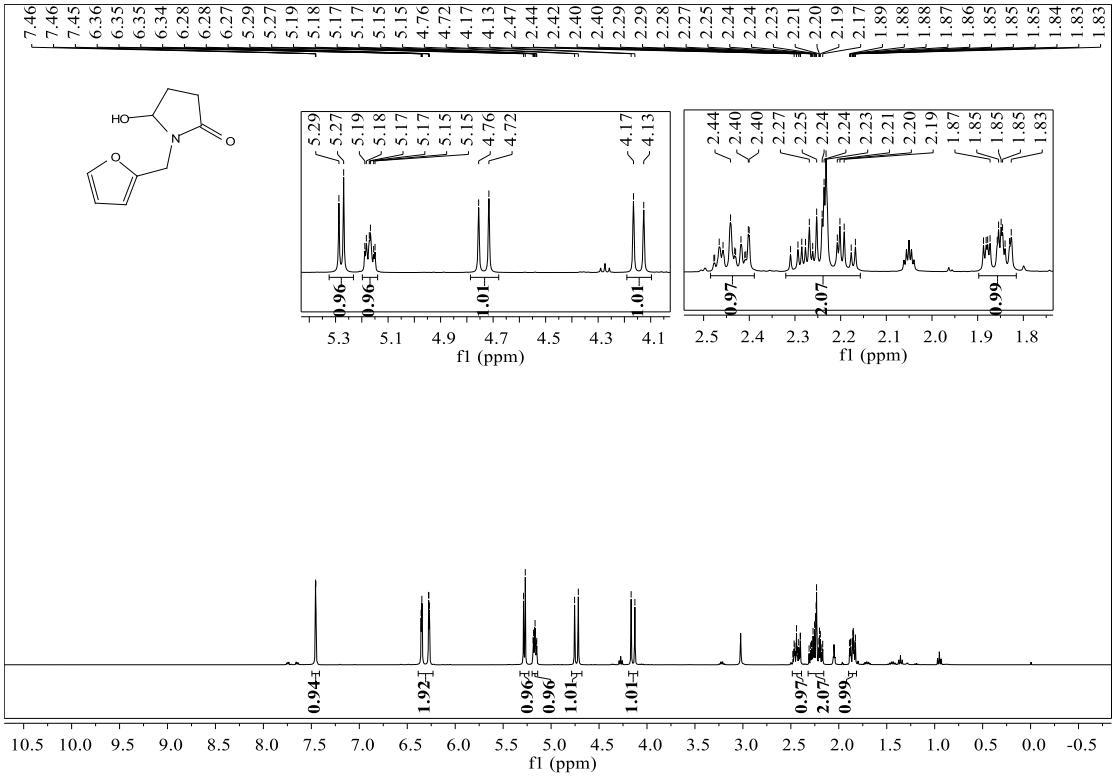
^{1}H NMR spectrum of compound 1ab (400 MHz, $CDCl_3$)



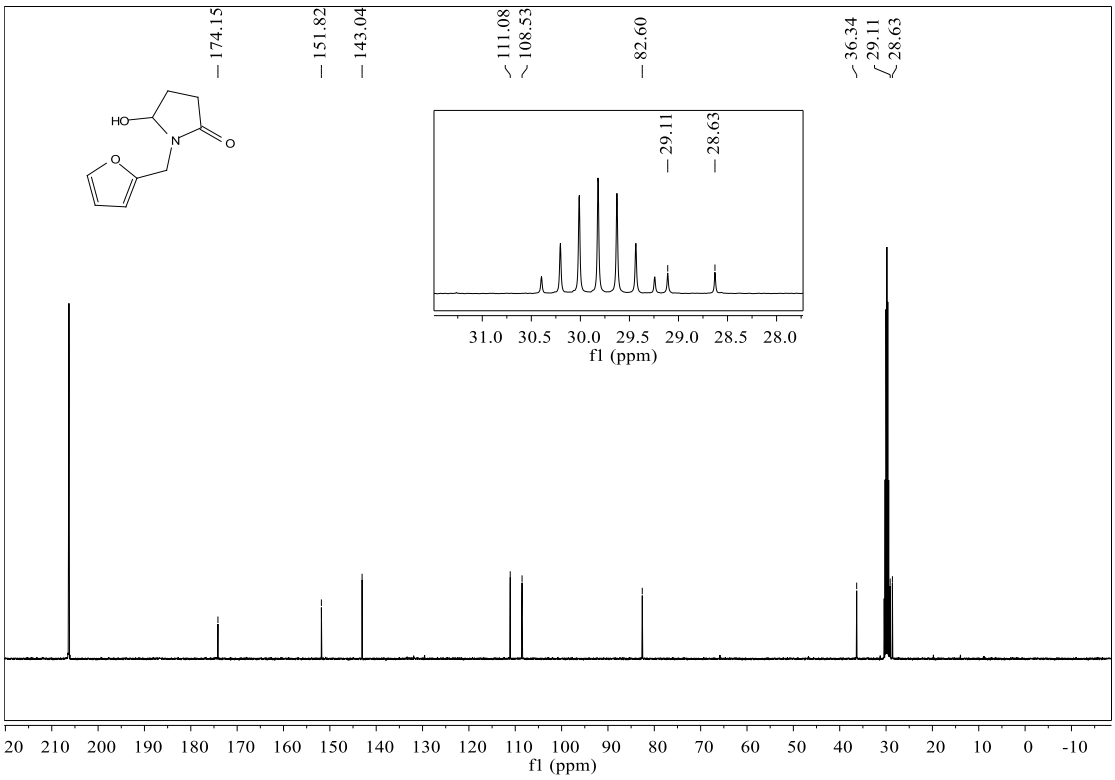
^{13}C NMR spectrum of compound 1ab (101 MHz, $CDCl_3$)



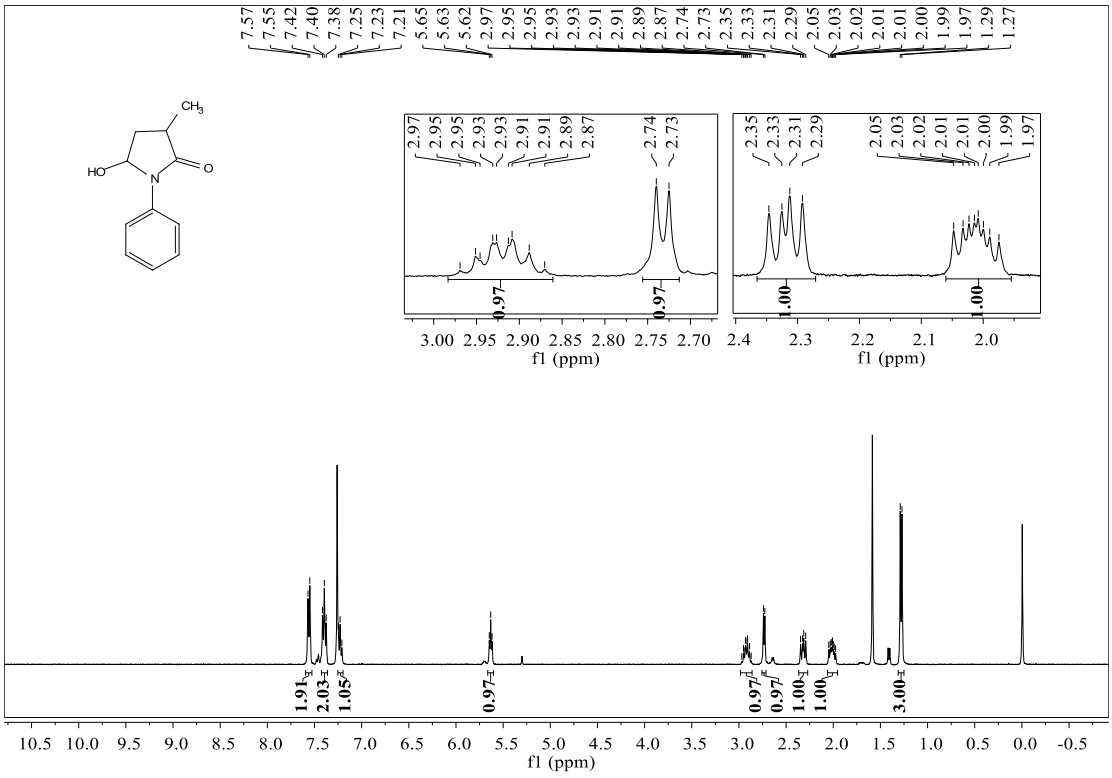
^{1}H NMR spectrum of compound 1ac (400 MHz, acetone-d_6)



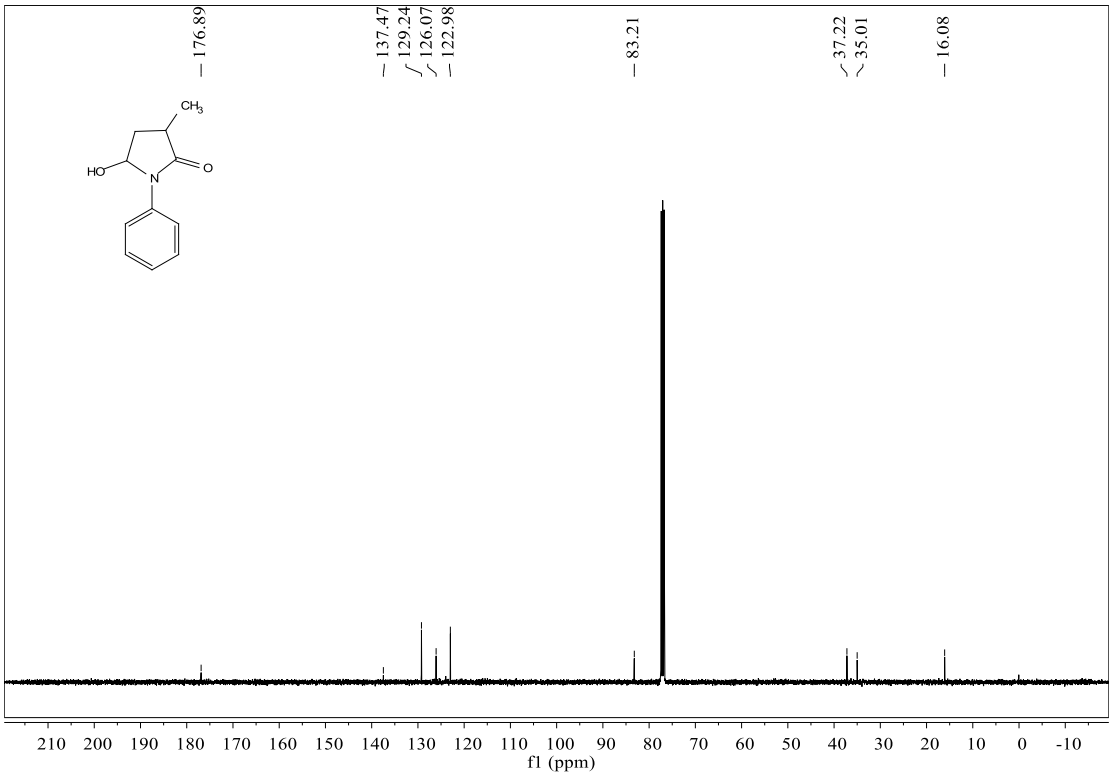
^{13}C NMR spectrum of compound 1ac (101 MHz, acetone-d_6)



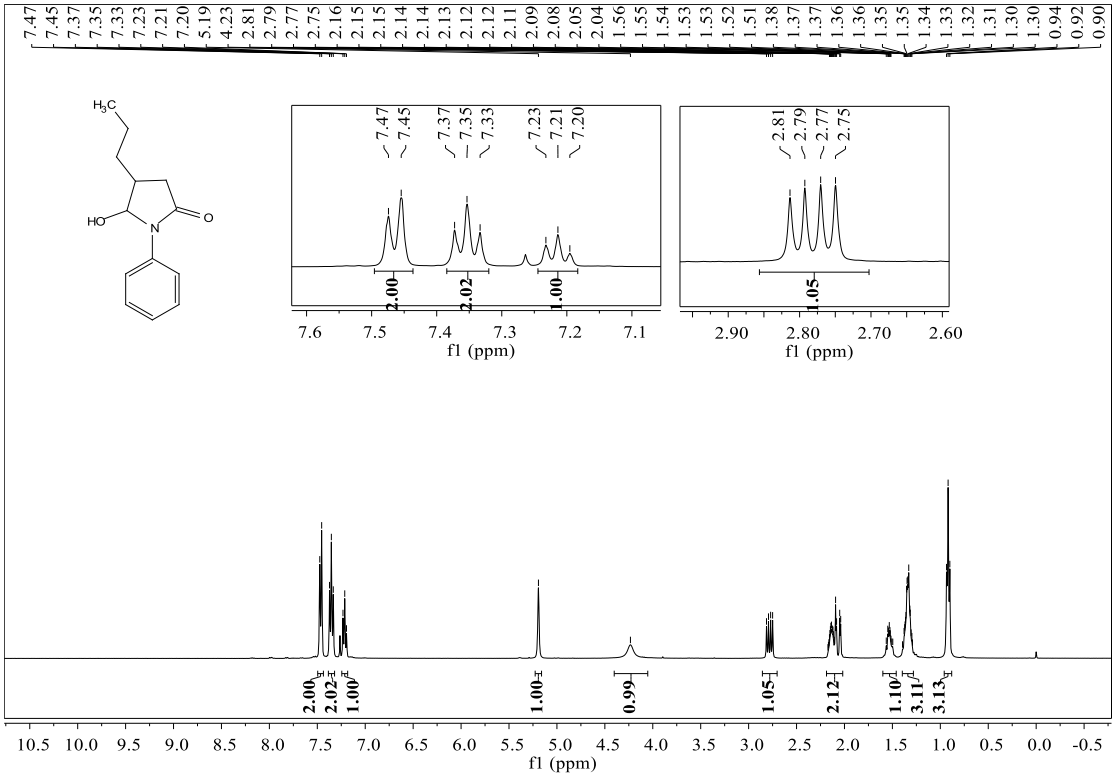
^{1}H NMR spectrum of compound 1ag (400 MHz, $CDCl_3$)



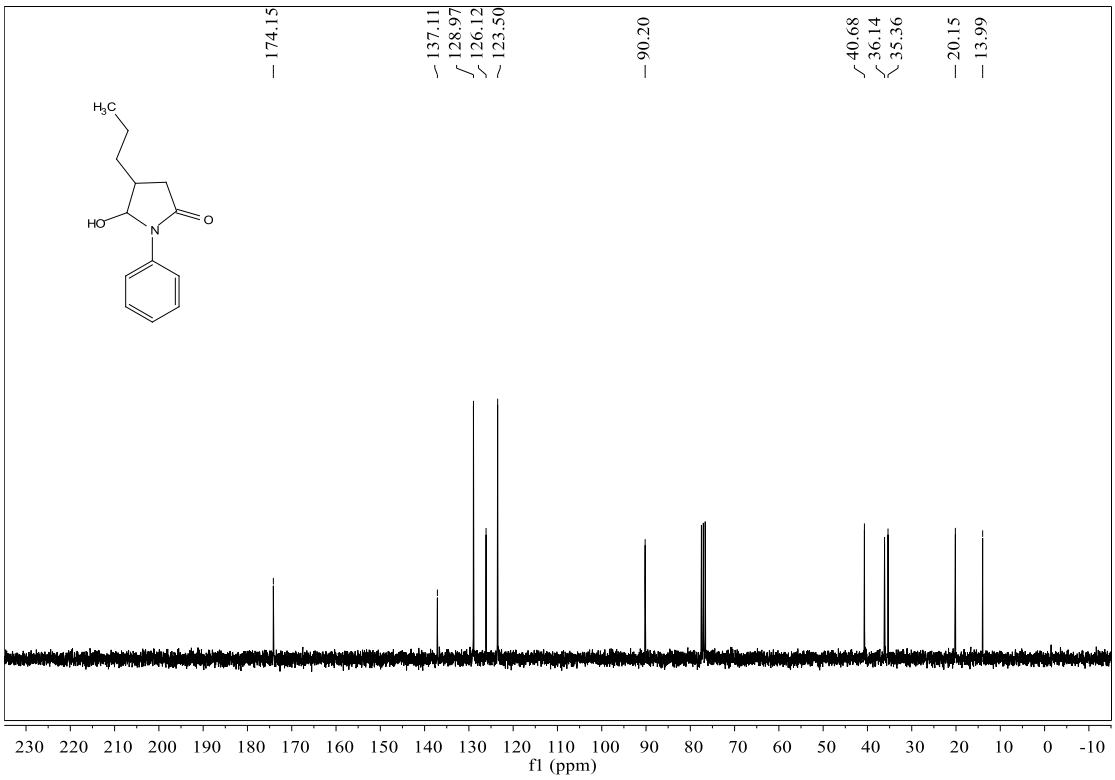
^{13}C NMR spectrum of compound 1ag (101 MHz, $CDCl_3$)



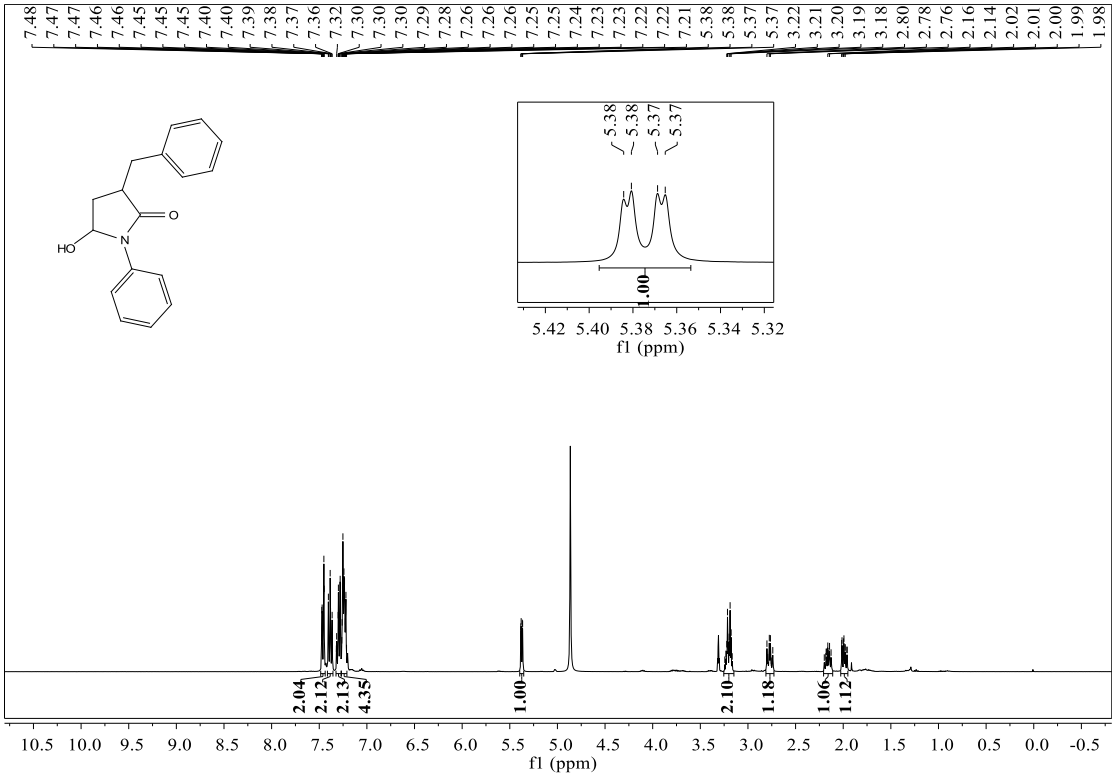
^{1}H NMR spectrum of compound 1ah (400 MHz, $CDCl_3$)



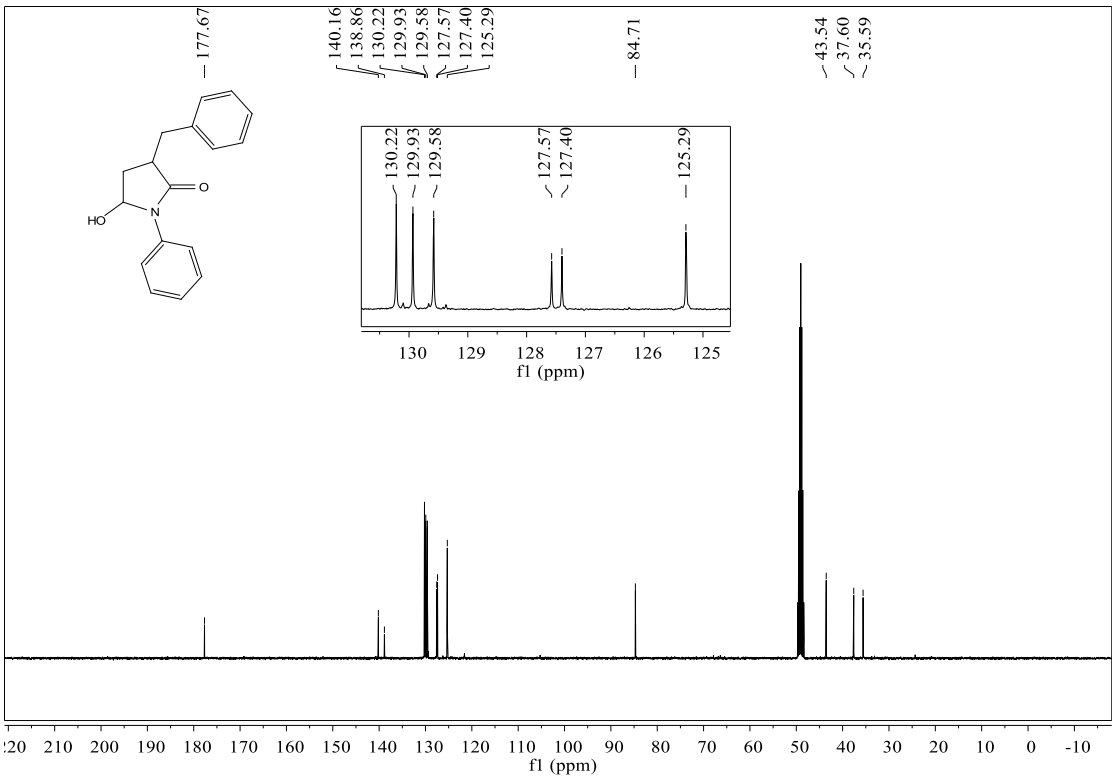
^{13}C NMR spectrum of compound 1ah (76 MHz, $CDCl_3$)



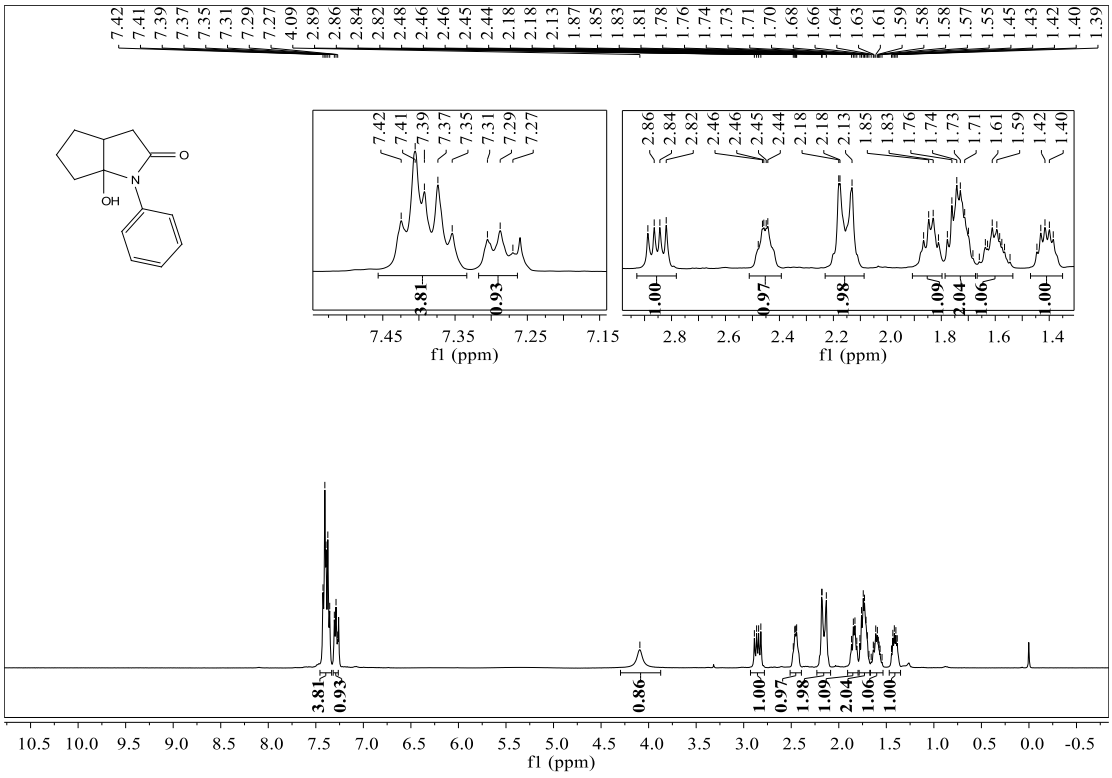
^{1}H NMR spectrum of compound 1au (400 MHz, methanol-d_4)



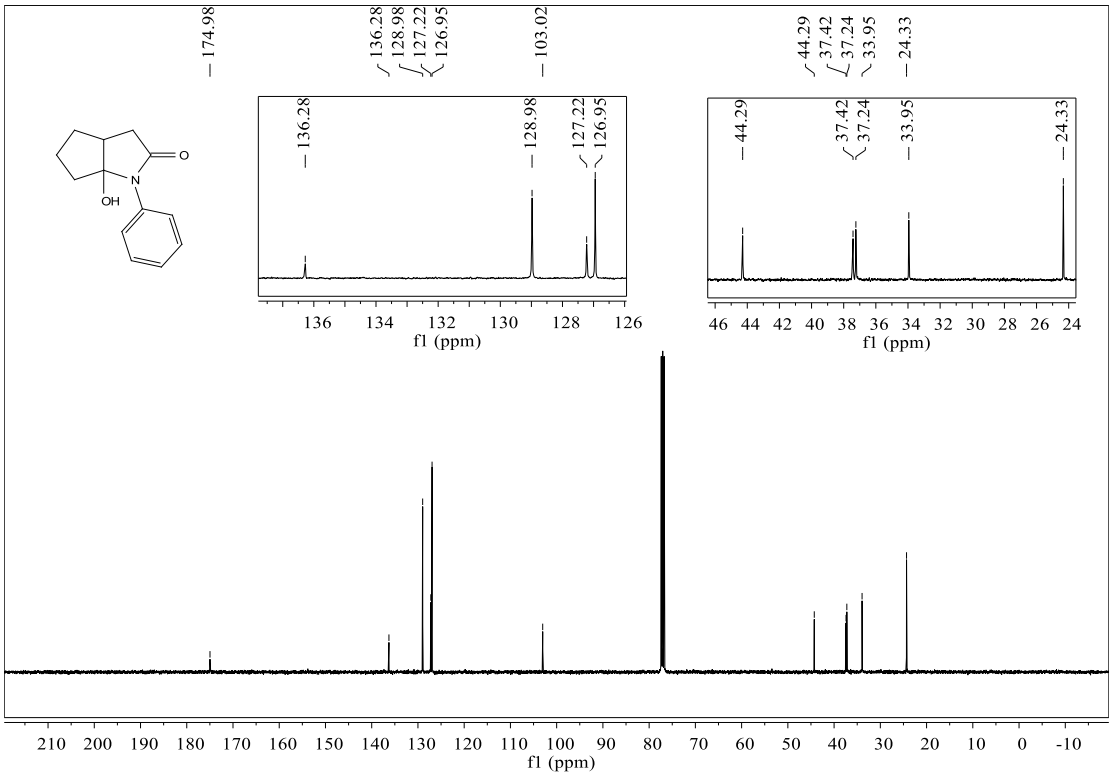
^{13}C NMR spectrum of compound 1au (101 MHz, methanol-d_4)



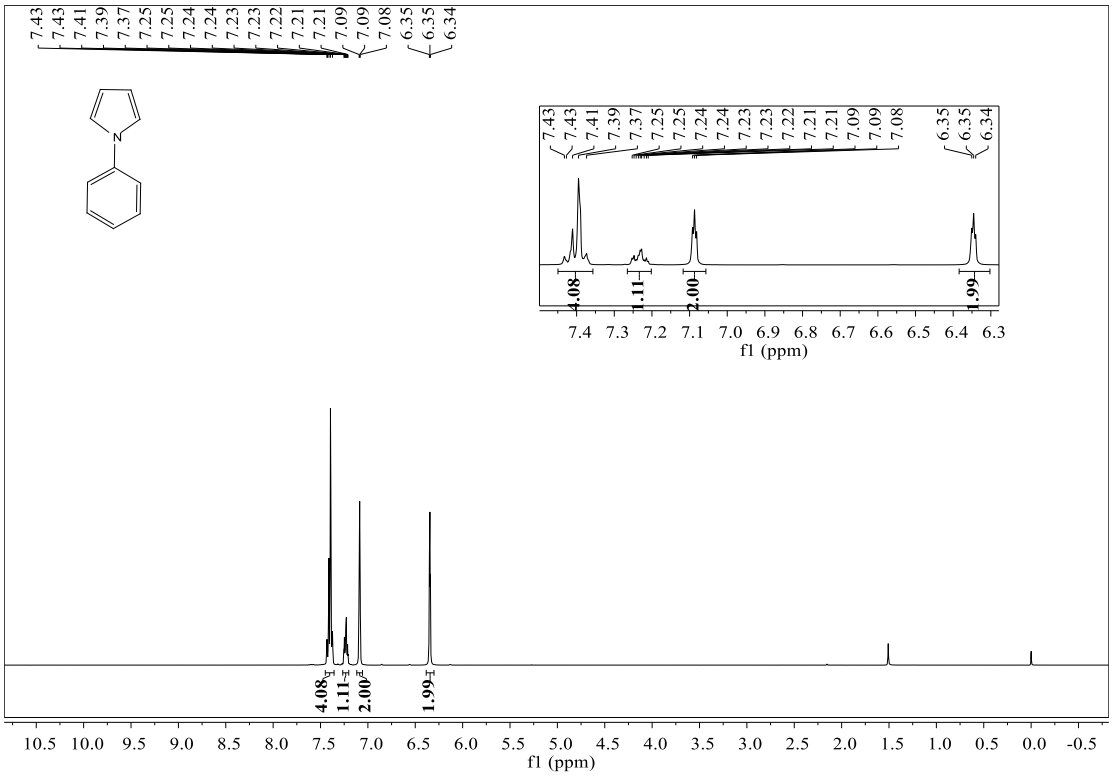
^{1}H NMR spectrum of compound 1aw (400 MHz, $CDCl_3$)



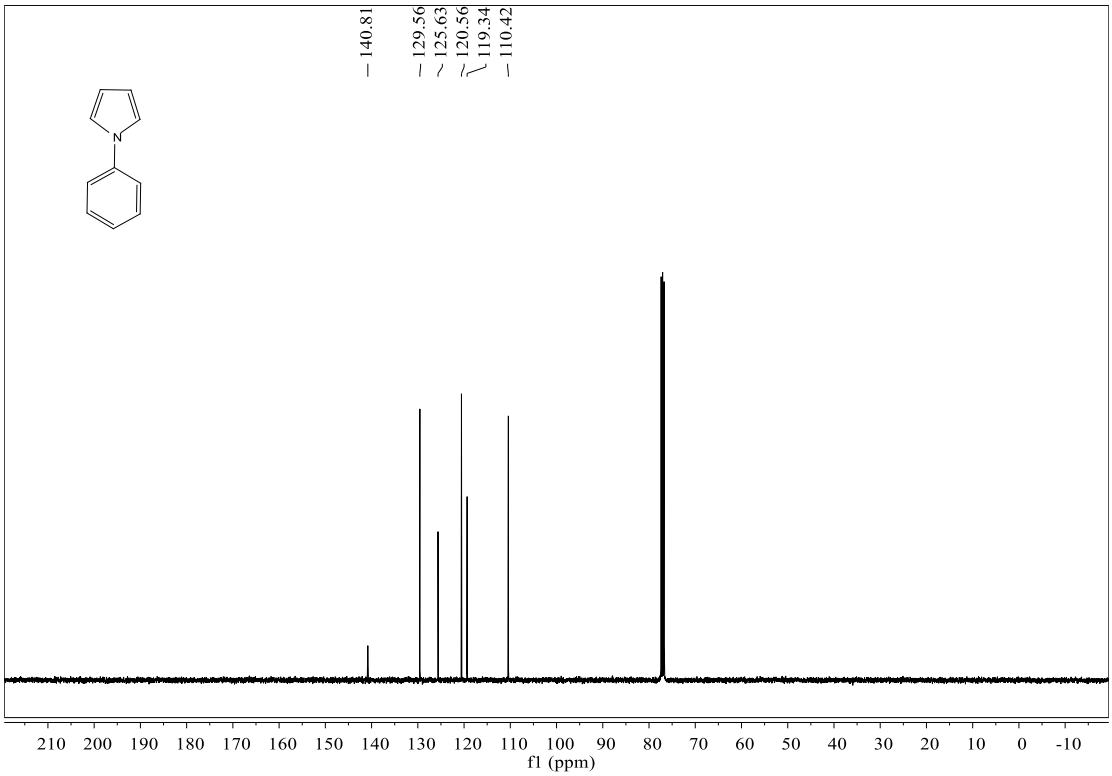
^{13}C NMR spectrum of compound 1aw (101 MHz, $CDCl_3$)



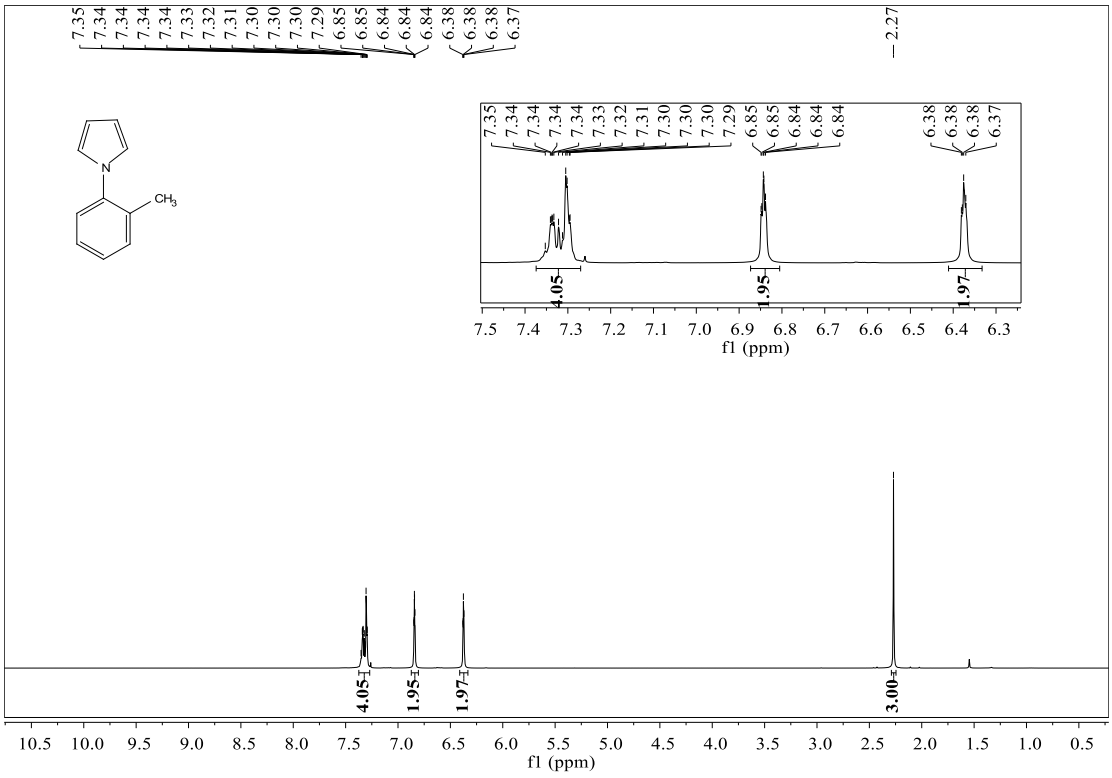
^{1}H NMR spectrum of compound 3a (400 MHz, $CDCl_3$)



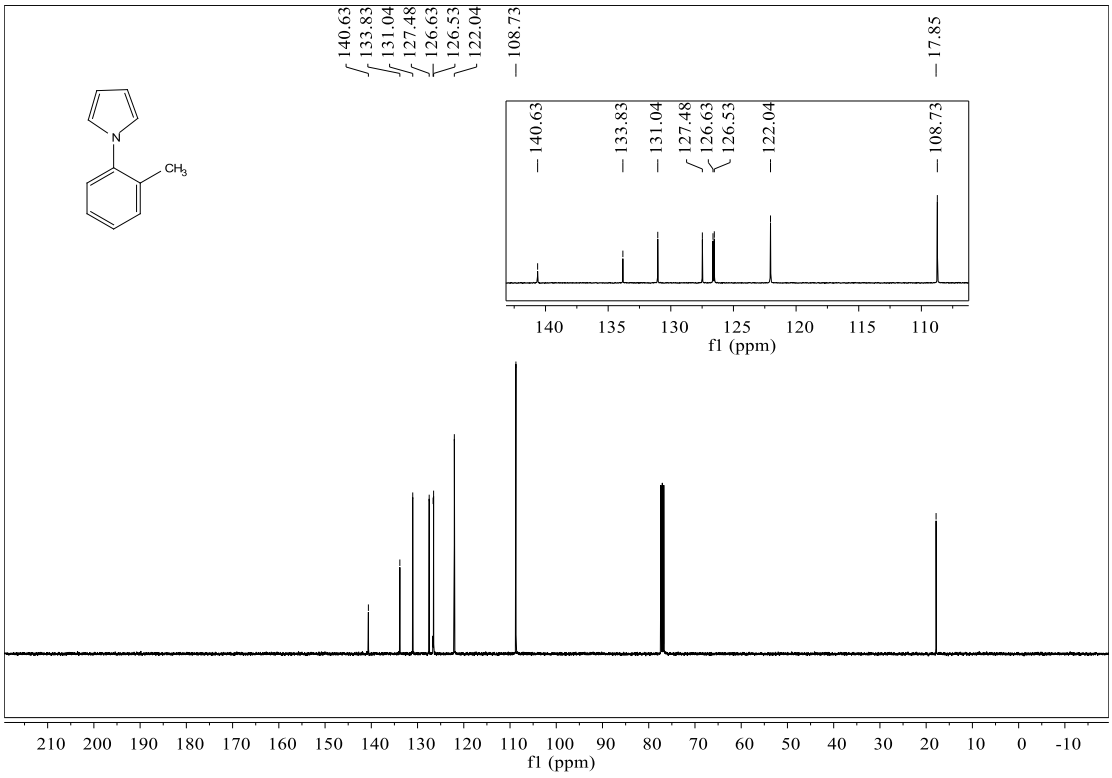
^{13}C NMR spectrum of compound 3a (101 MHz, $CDCl_3$)



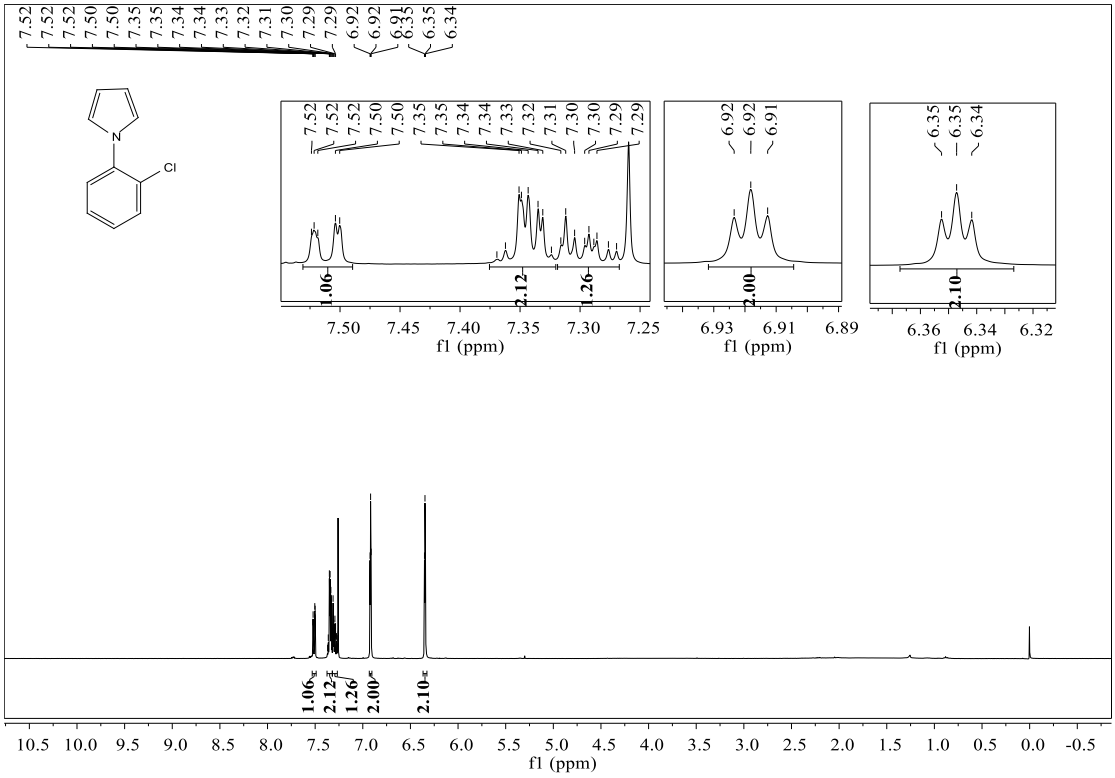
^{1}H NMR spectrum of compound 3b (400 MHz, $CDCl_3$)



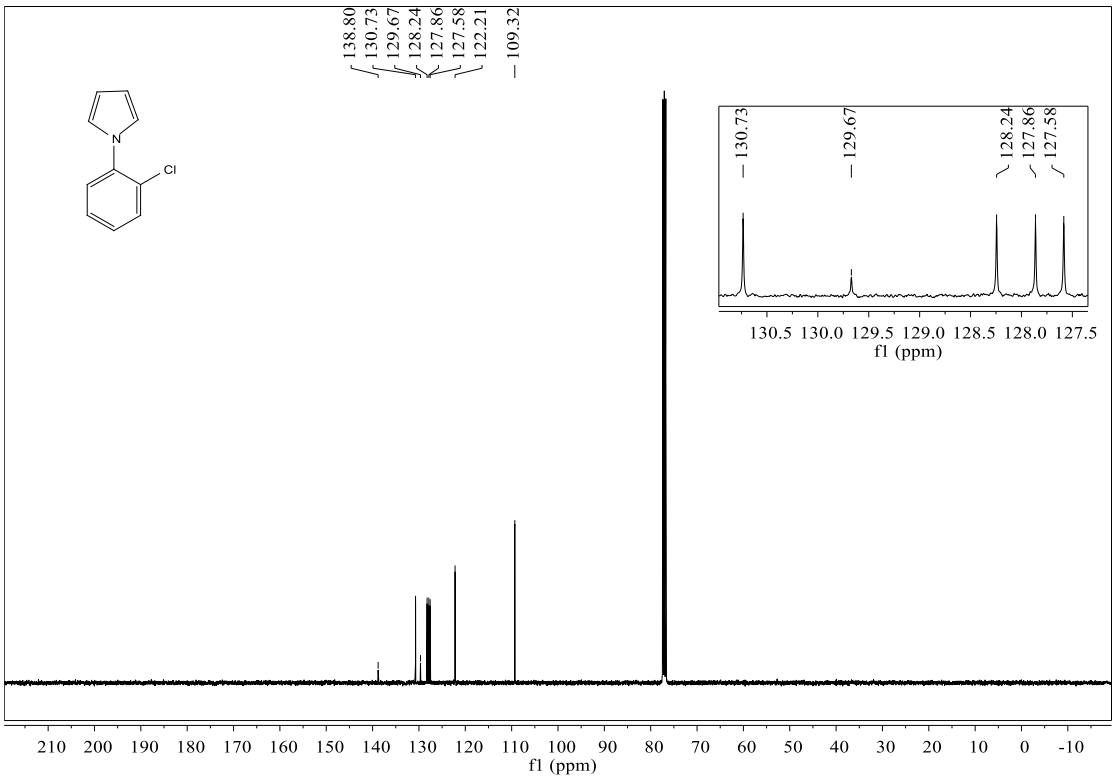
^{13}C NMR spectrum of compound 3b (101 MHz, $CDCl_3$)



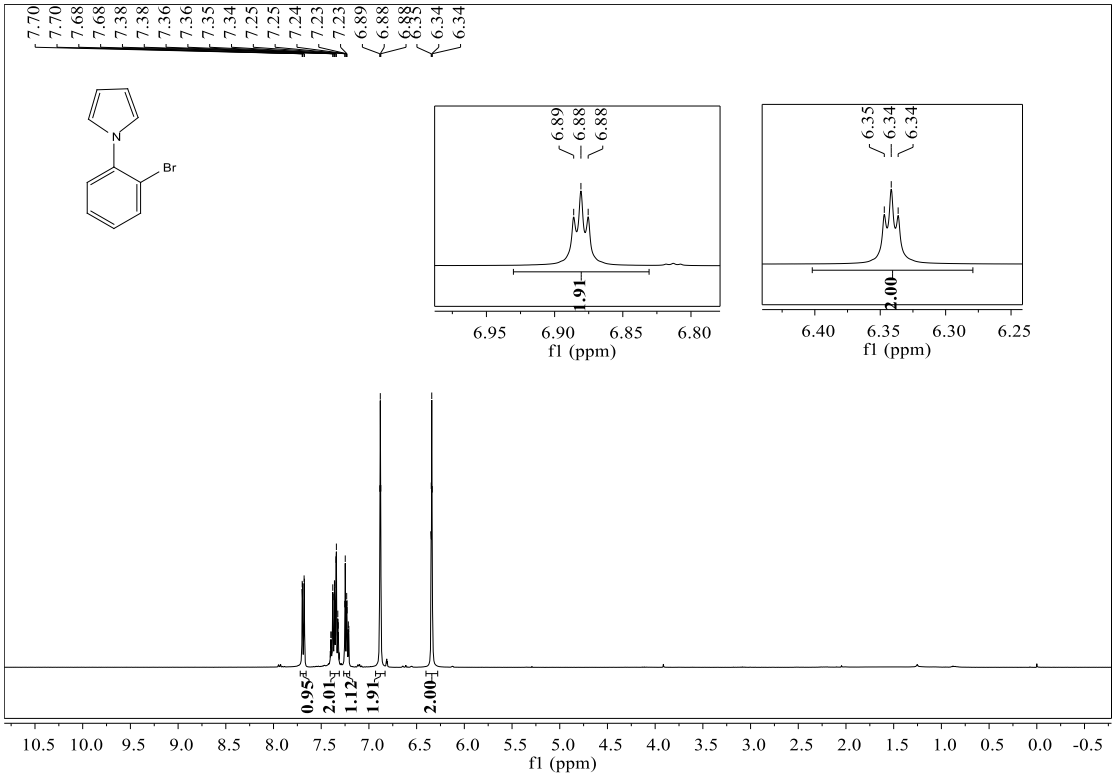
^{1}H NMR spectrum of compound 3c (400 MHz, $CDCl_3$)



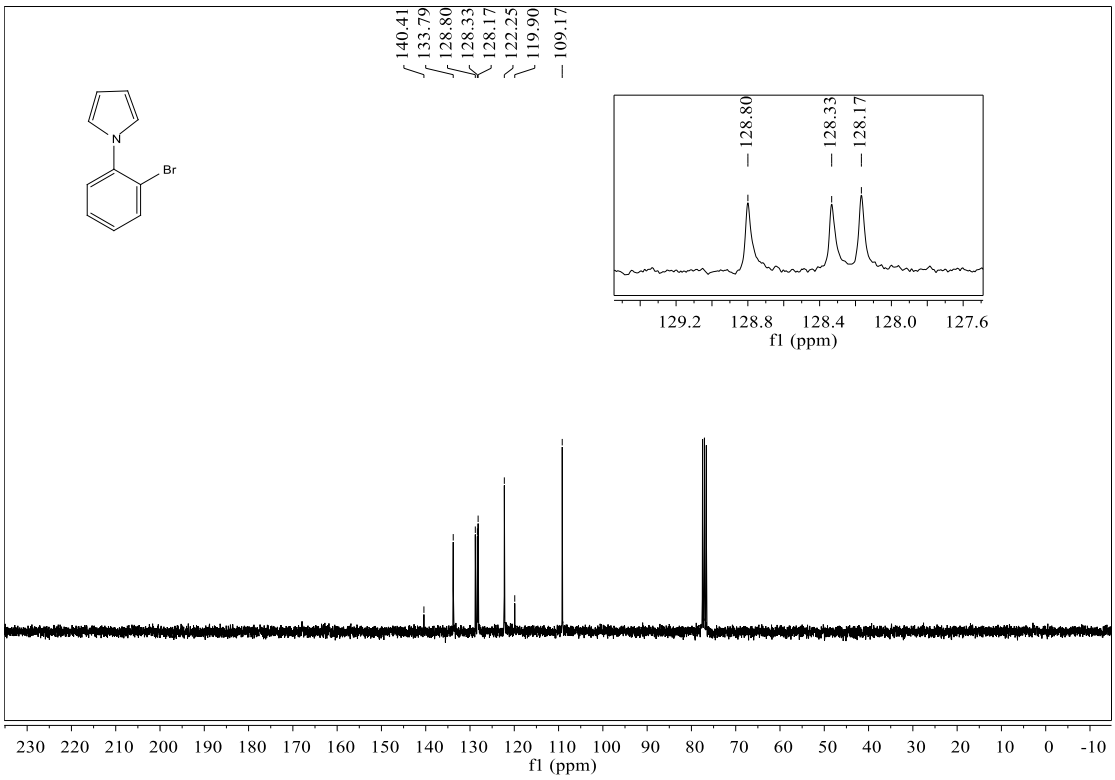
^{13}C NMR spectrum of compound 3c (101 MHz, $CDCl_3$)



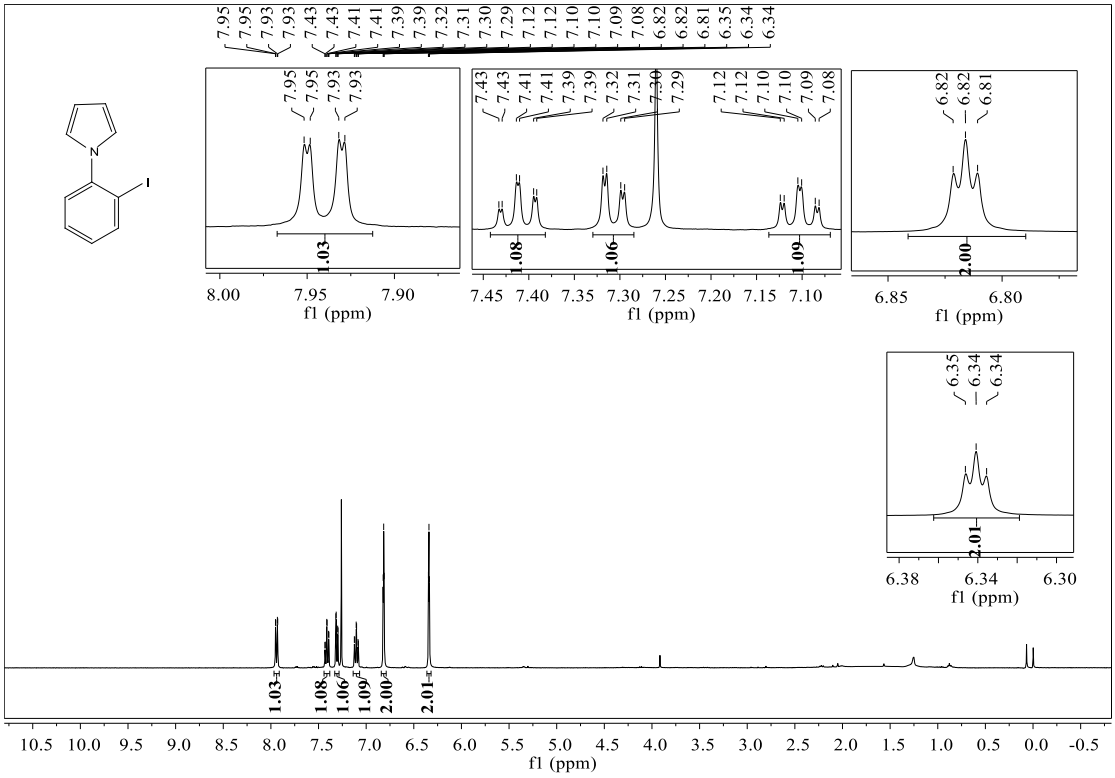
^{1}H NMR spectrum of compound 3d (400 MHz, $CDCl_3$)



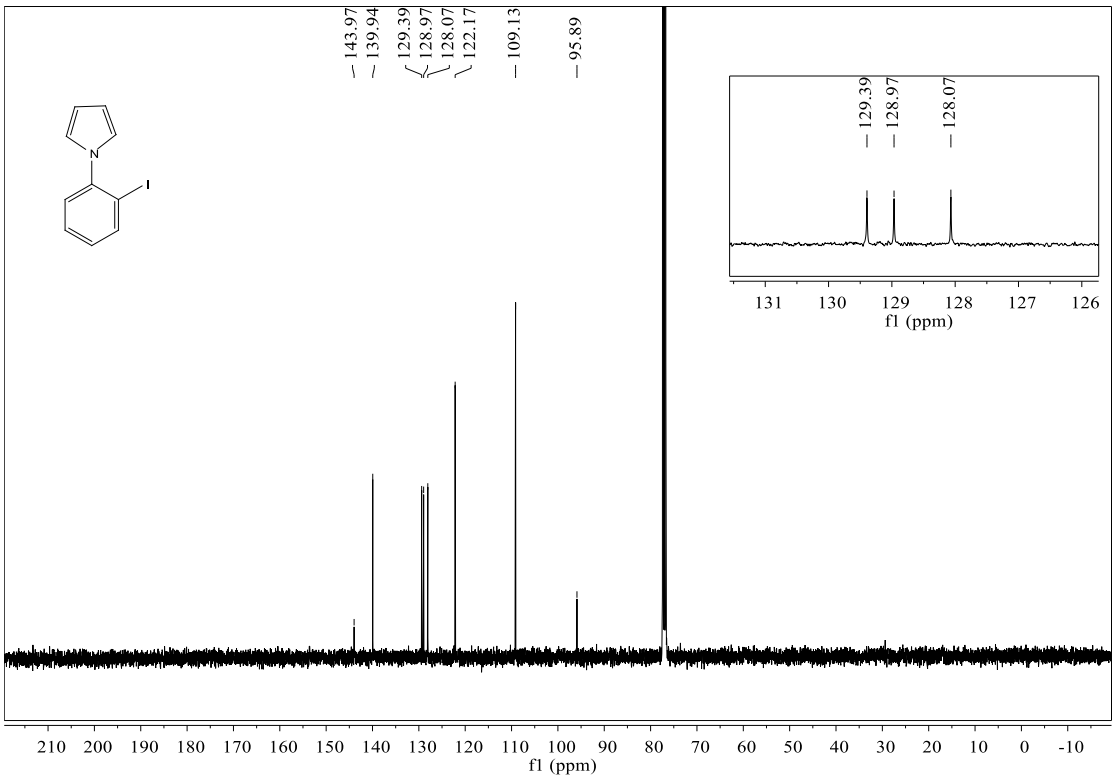
^{13}C NMR spectrum of compound 3d (76 MHz, $CDCl_3$)



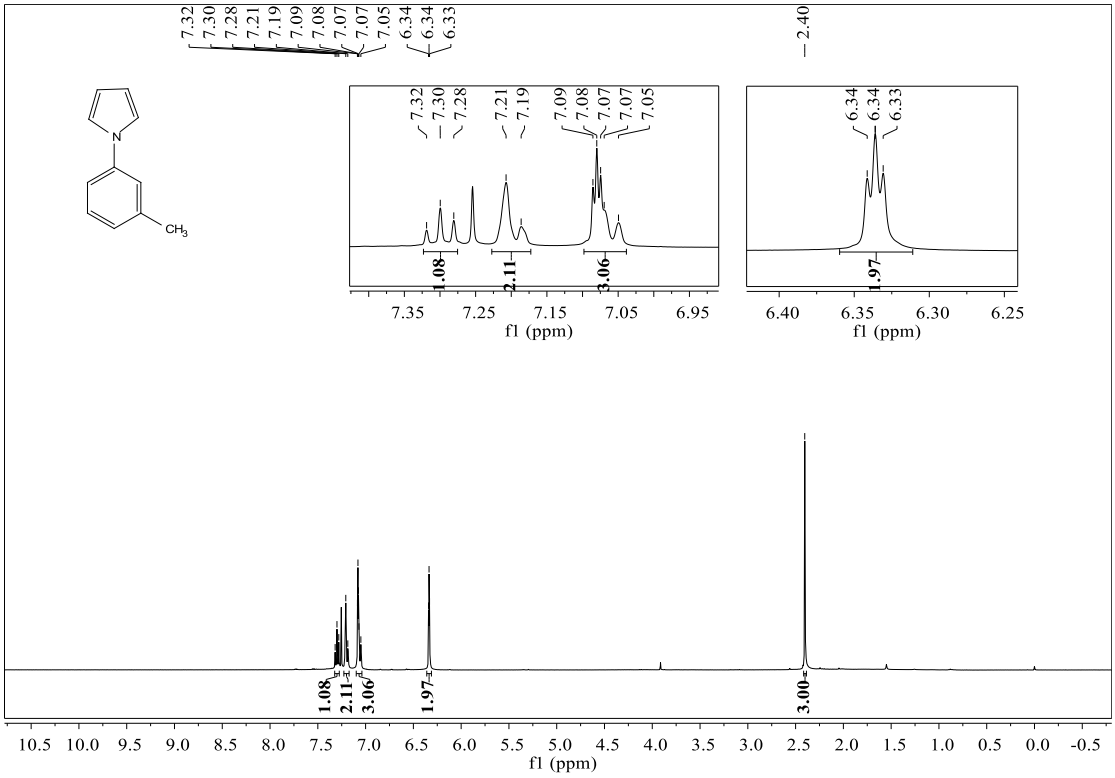
^{1}H NMR spectrum of compound 3e (400 MHz, $CDCl_3$)



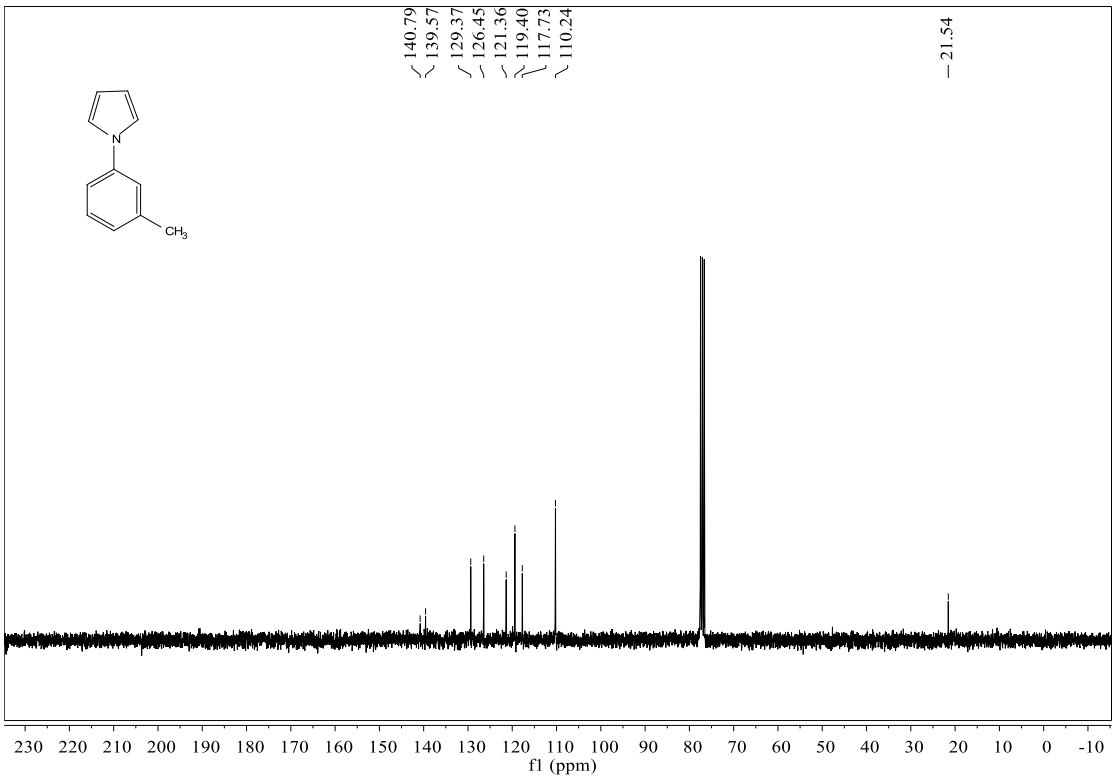
^{13}C NMR spectrum of compound 3e (101 MHz, $CDCl_3$)



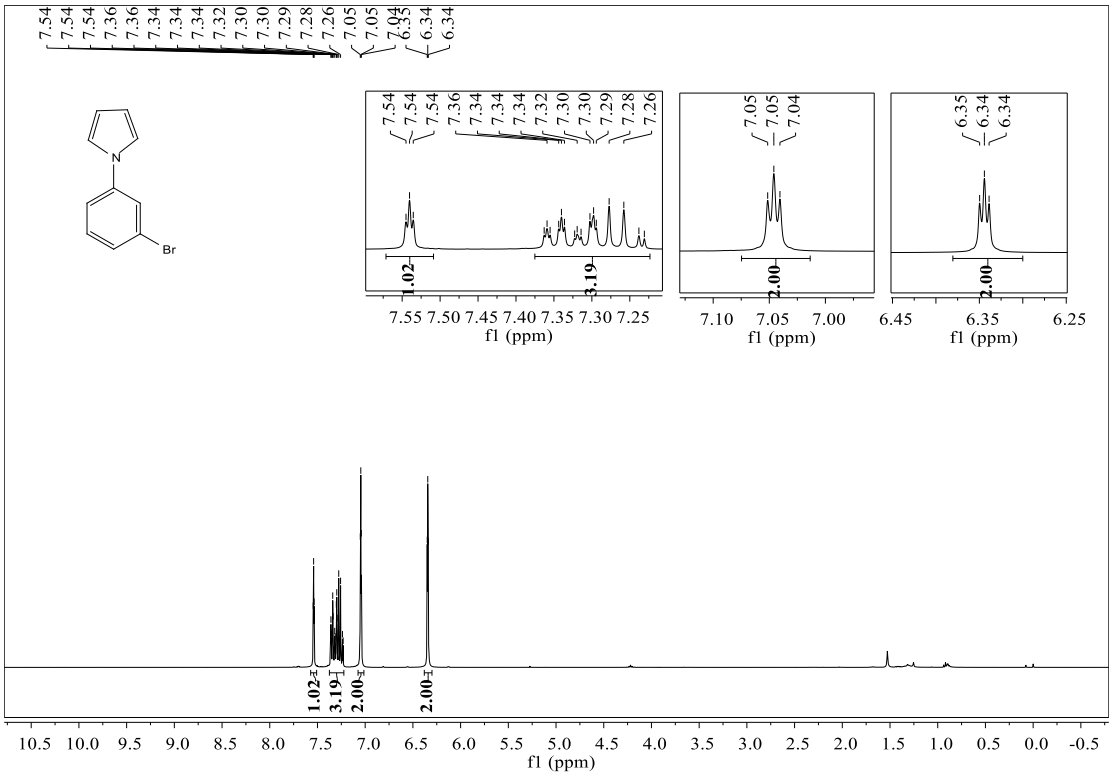
^{1}H NMR spectrum of compound 3f (400 MHz, $CDCl_3$)



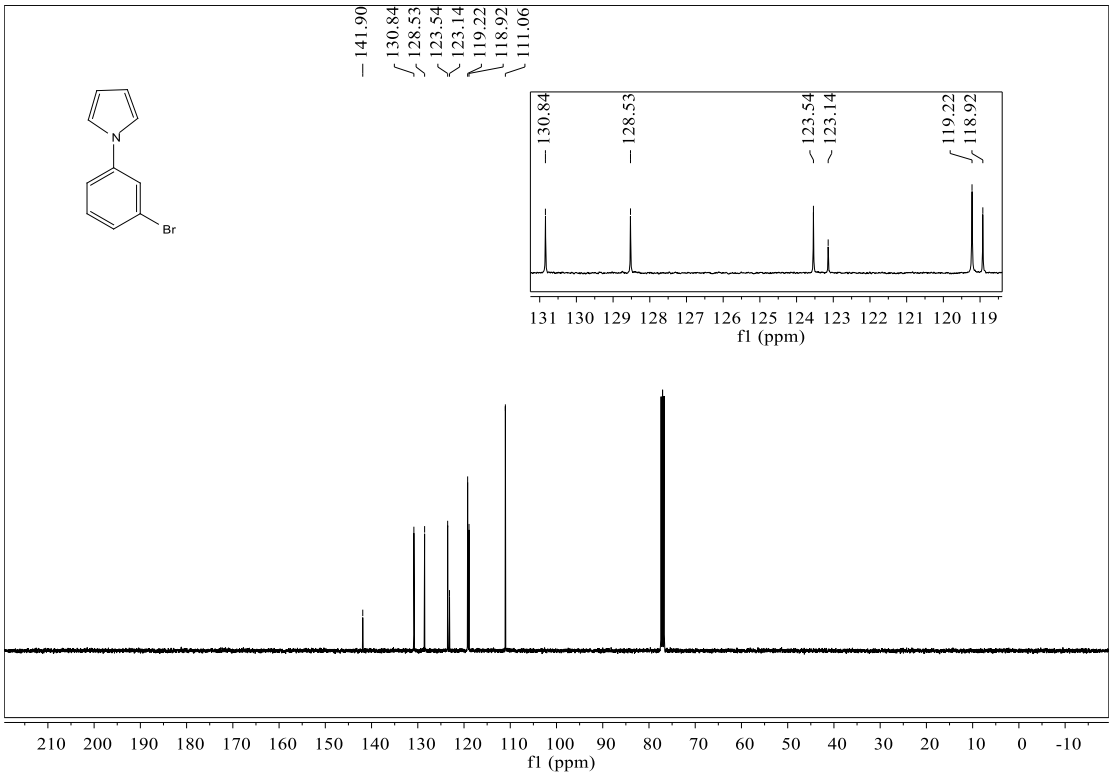
^{13}C NMR spectrum of compound 3f (76 MHz, $CDCl_3$)



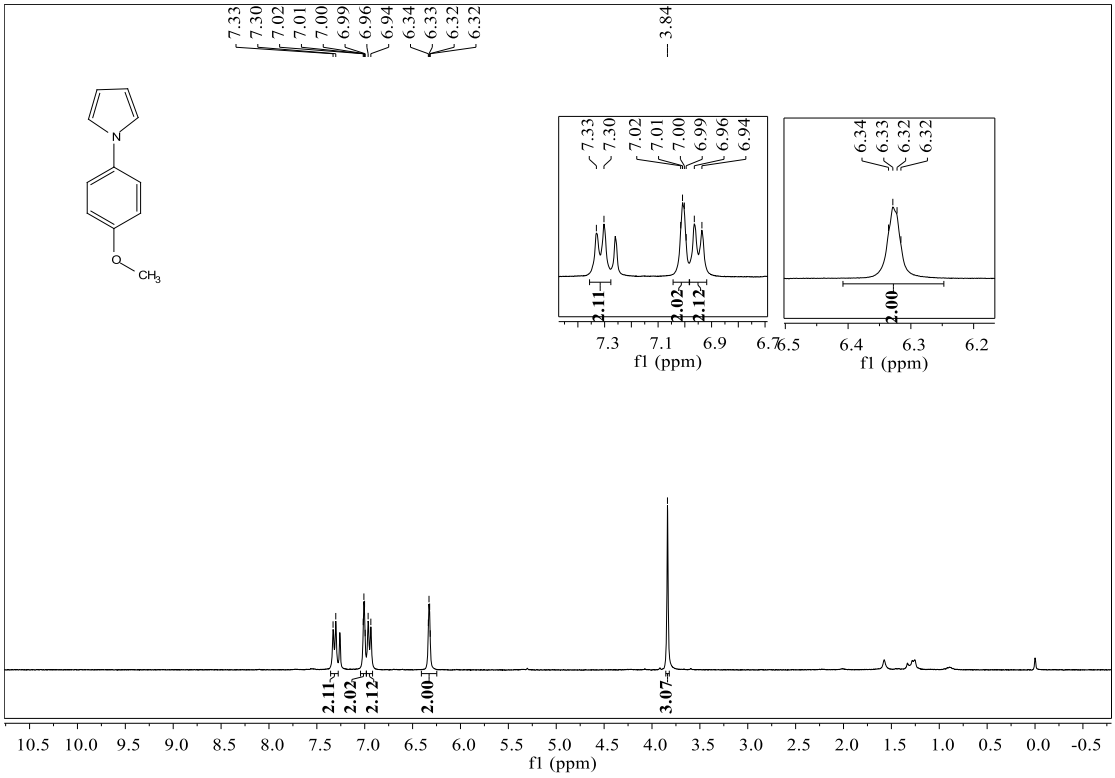
^{1}H NMR spectrum of compound 3g (400 MHz, $CDCl_3$)



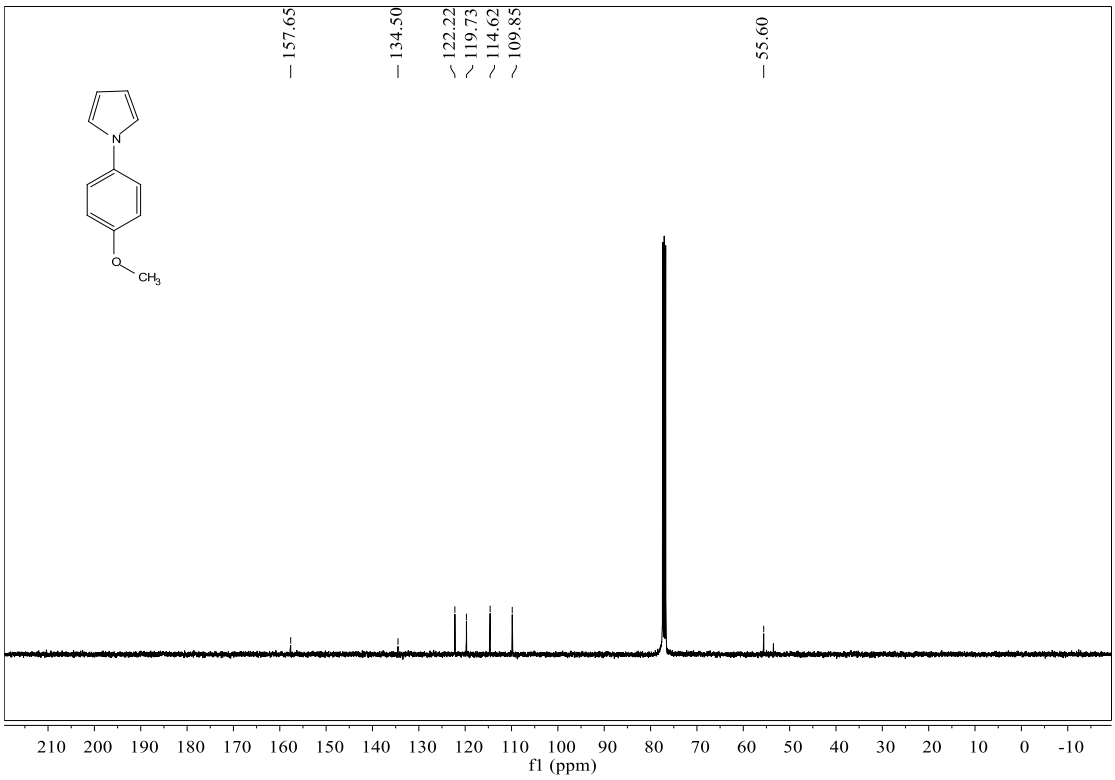
^{13}C NMR spectrum of compound 3g (101 MHz, $CDCl_3$)



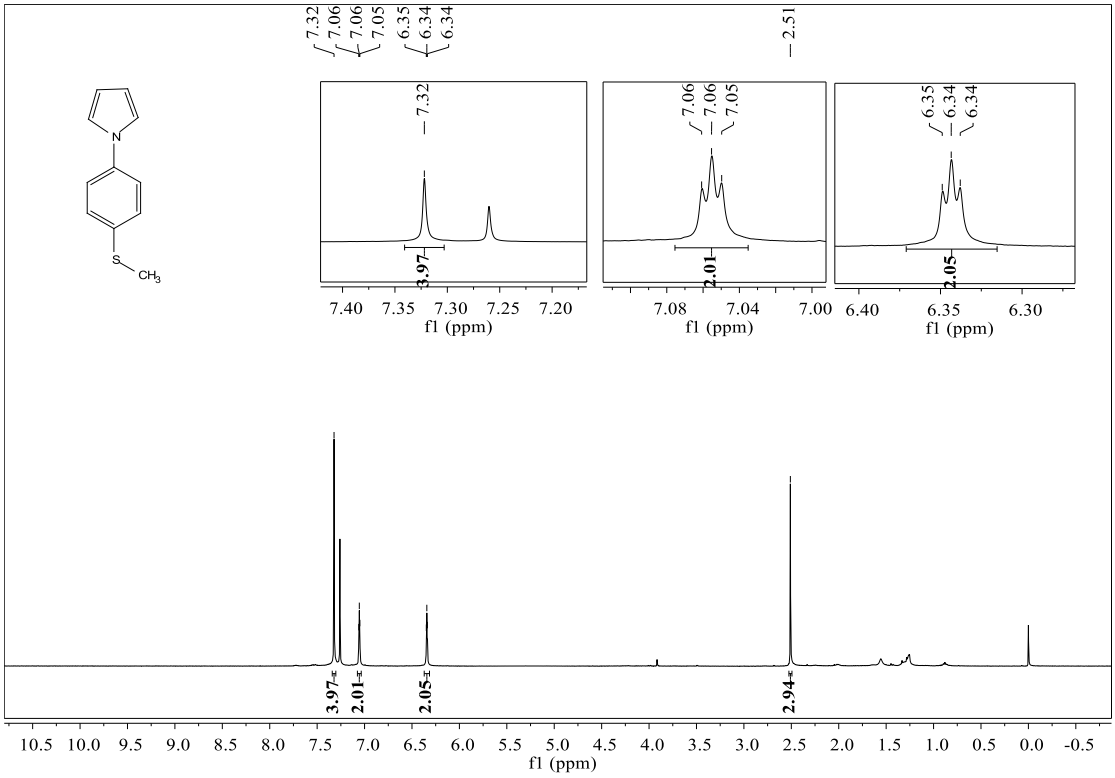
^{1}H NMR spectrum of compound 3h (300 MHz, $CDCl_3$)



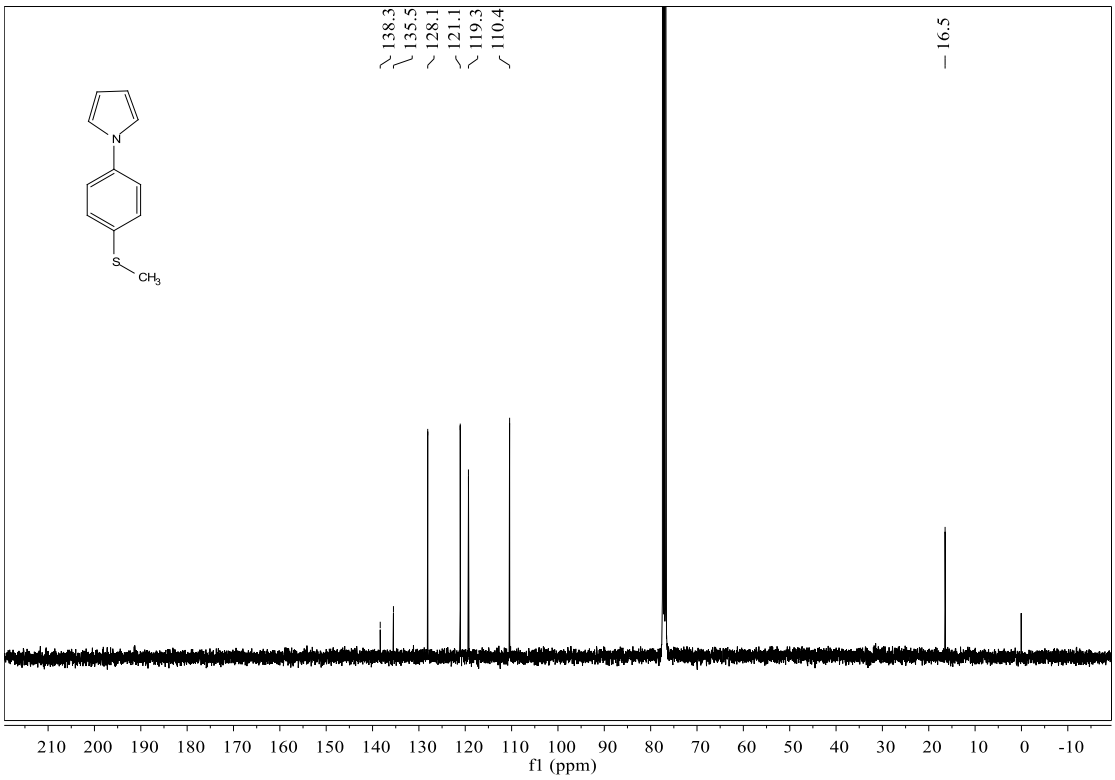
^{13}C NMR spectrum of compound 3h (101 MHz, $CDCl_3$)



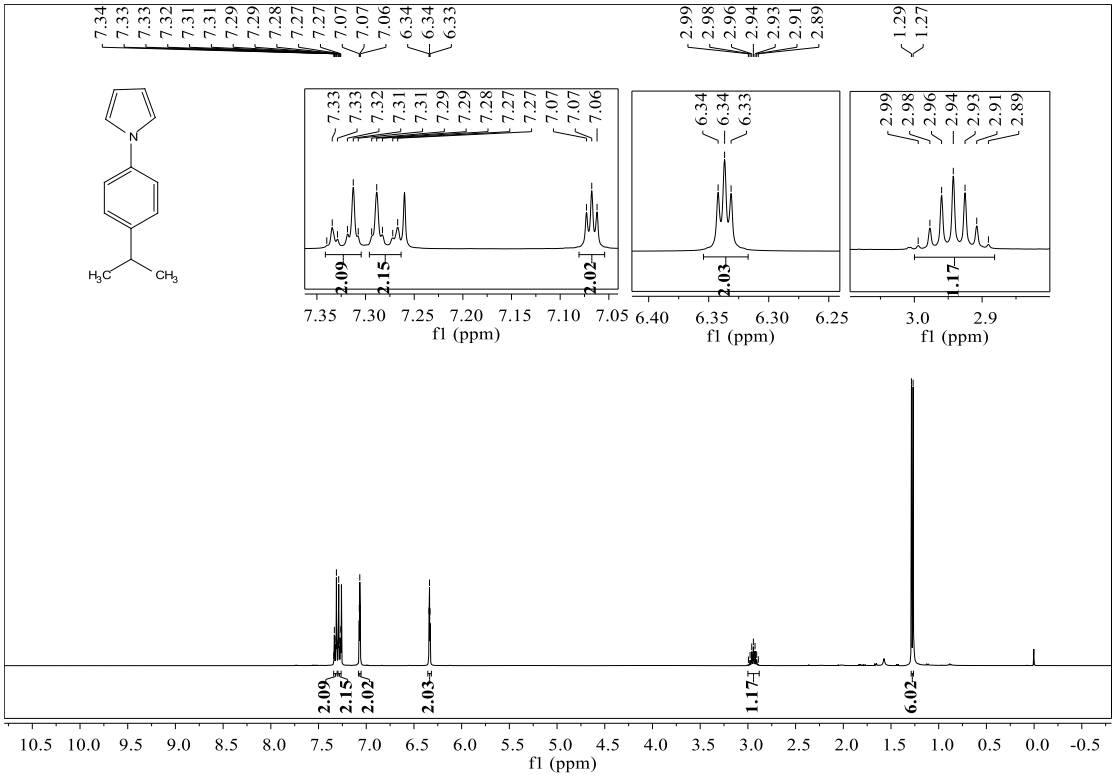
^{1}H NMR spectrum of compound 3i (400 MHz, $CDCl_3$)



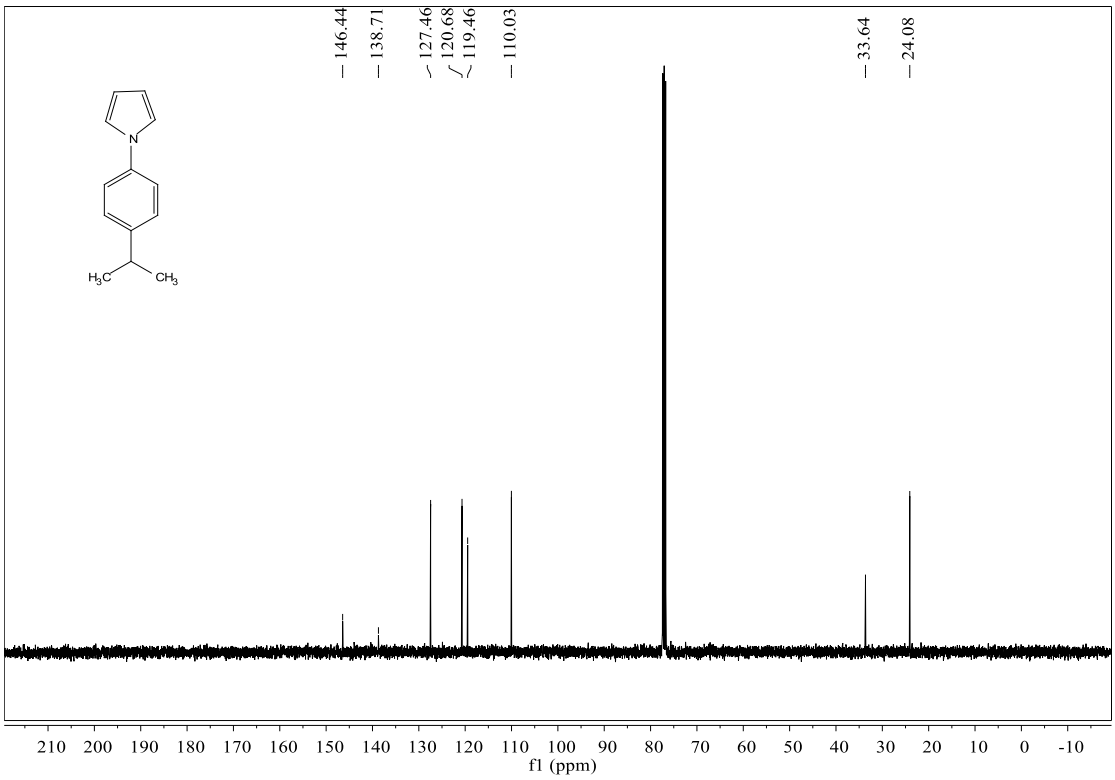
^{13}C NMR spectrum of compound 3i (101 MHz, $CDCl_3$)



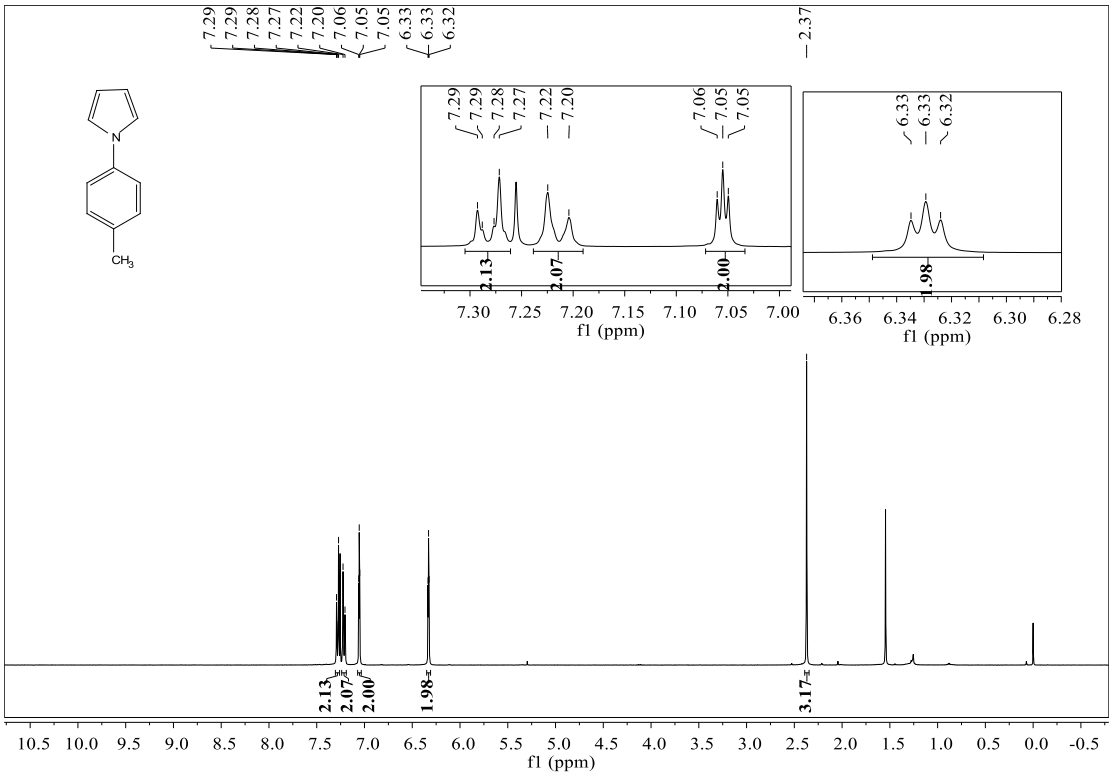
^{1}H NMR spectrum of compound 3j (400 MHz, $CDCl_3$)



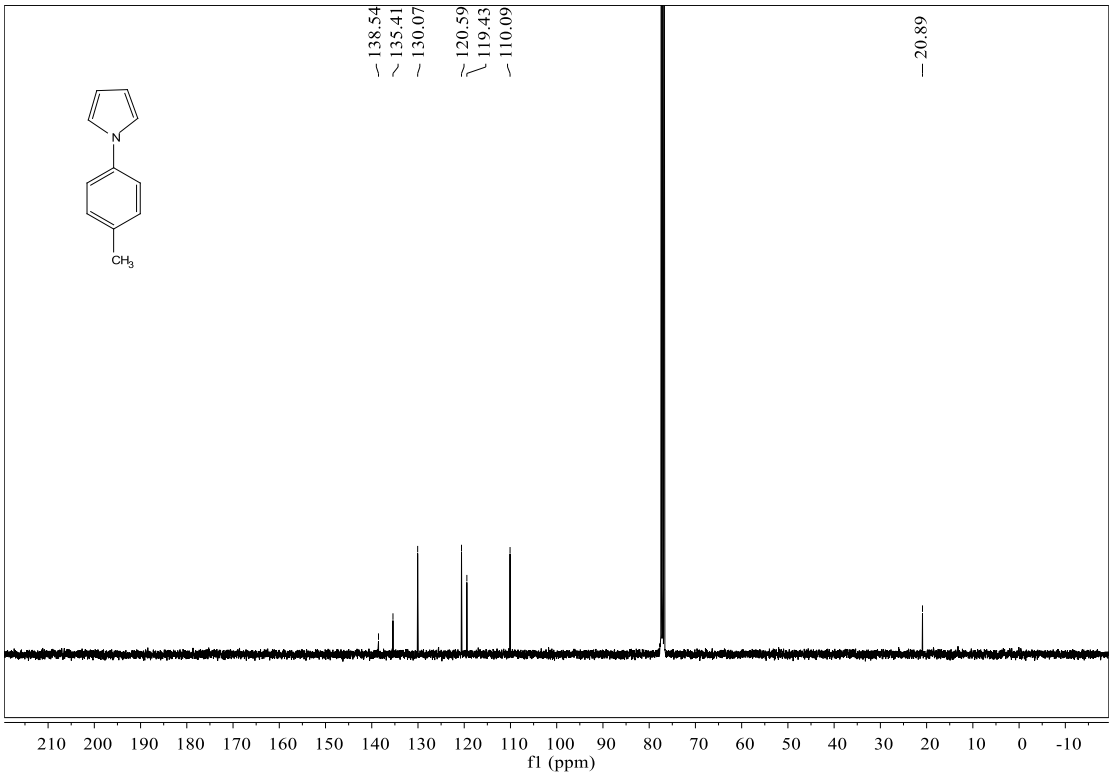
^{13}C NMR spectrum of compound 3j (101 MHz, $CDCl_3$)



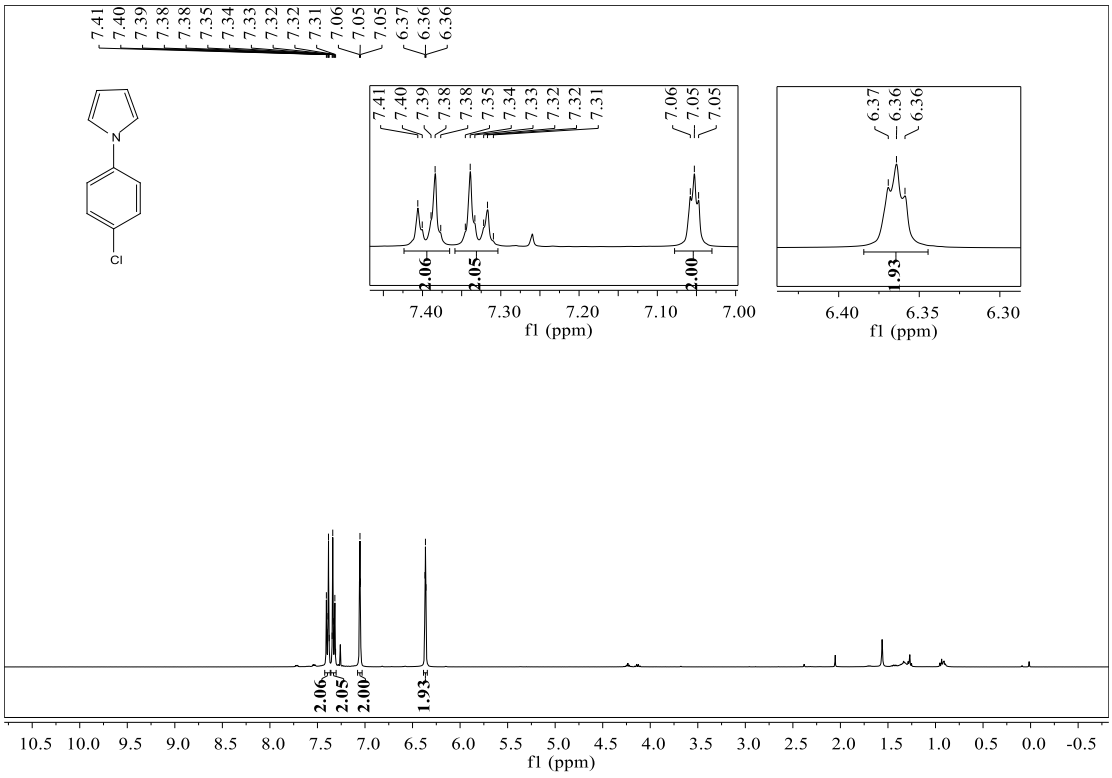
^{1}H NMR spectrum of compound 3k (400 MHz, $CDCl_3$)



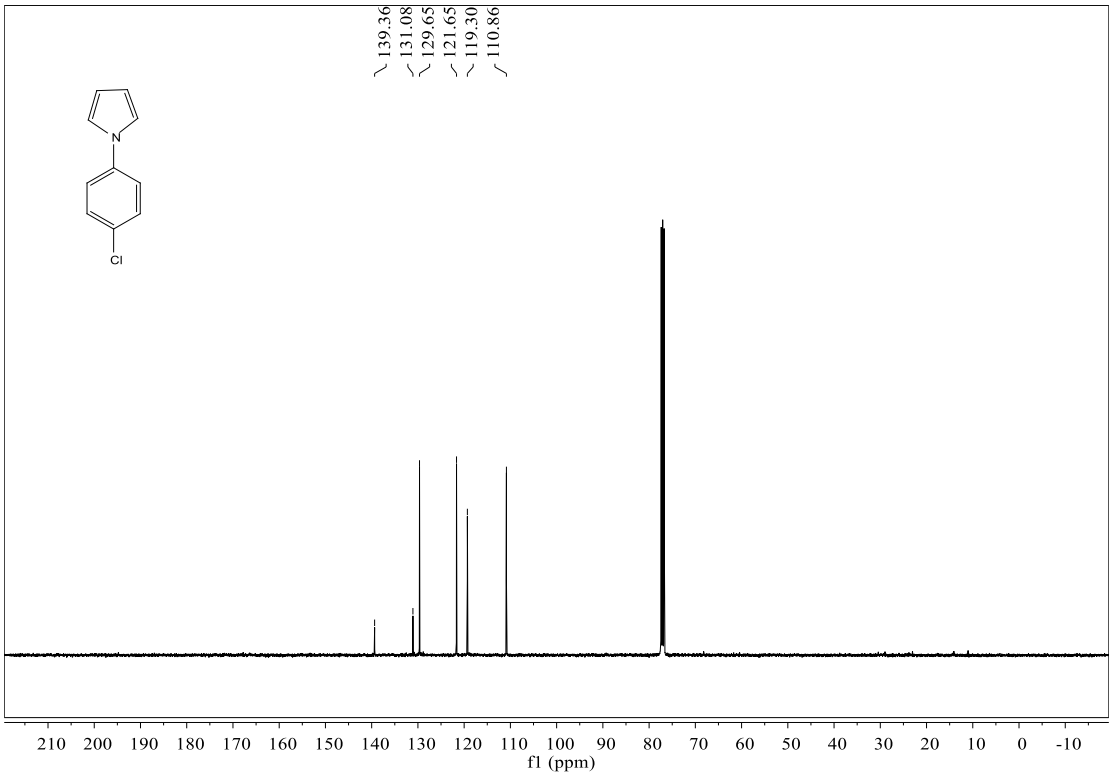
^{13}C NMR spectrum of compound 3k (101 MHz, $CDCl_3$)



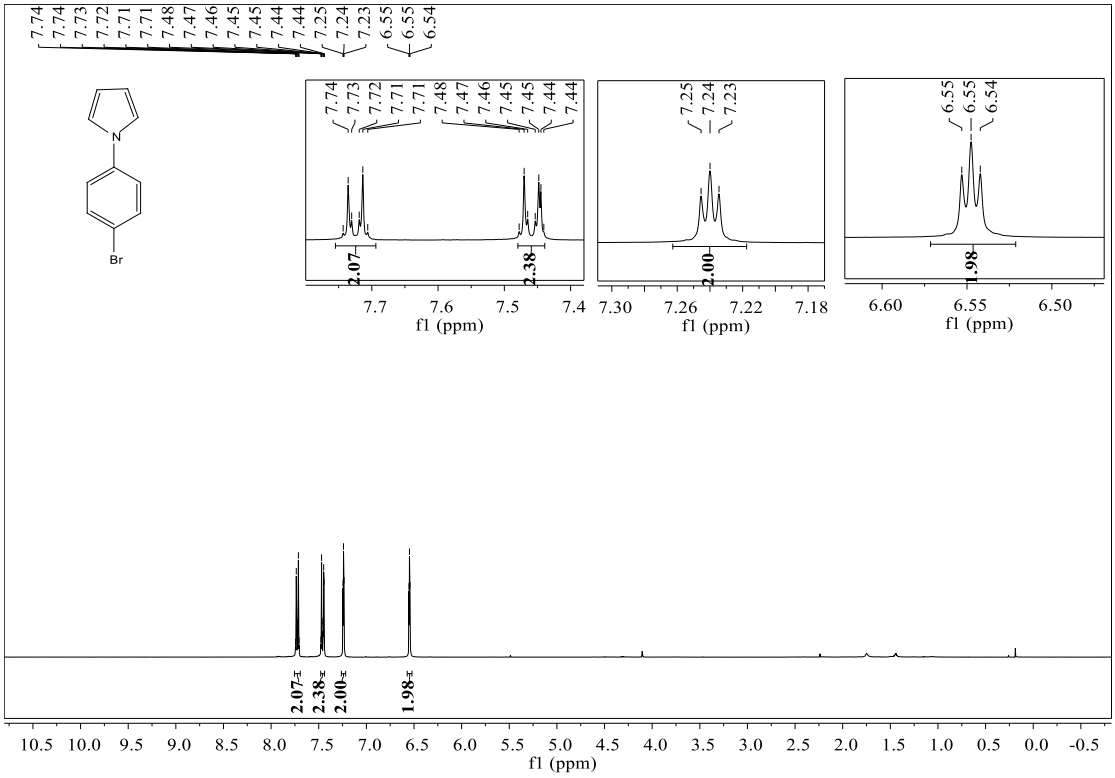
^{1}H NMR spectrum of compound 3l (400 MHz, $CDCl_3$)



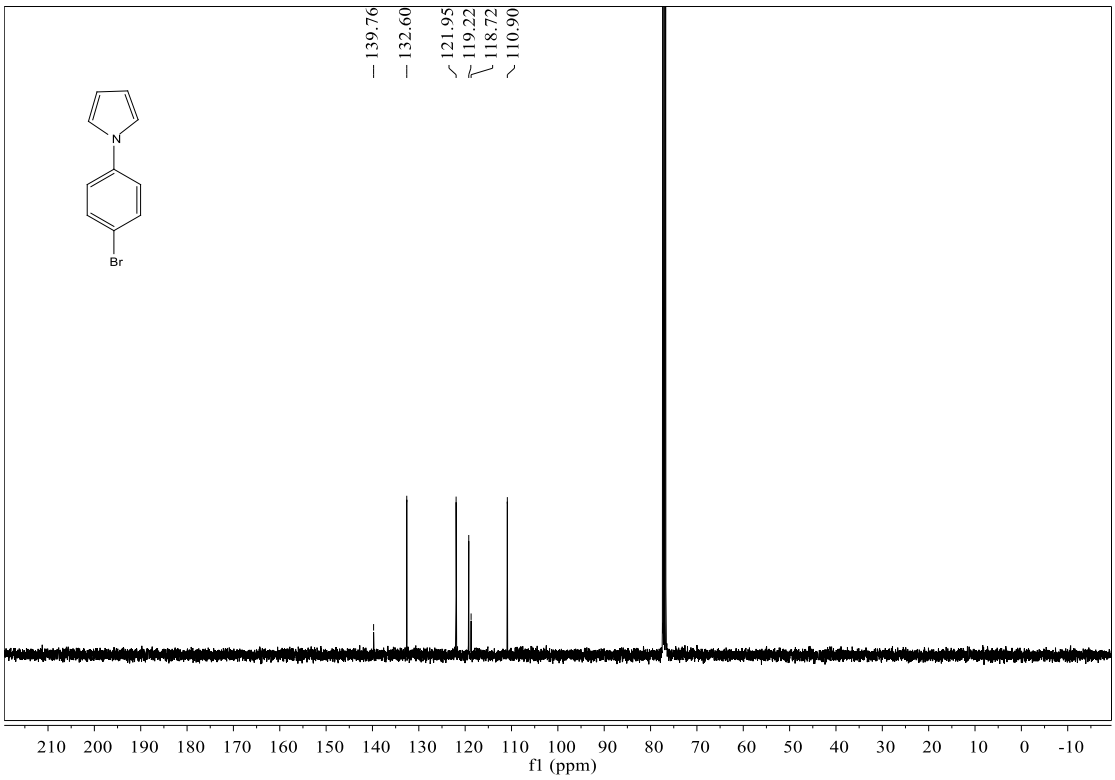
^{13}C NMR spectrum of compound 3l (101 MHz, $CDCl_3$)



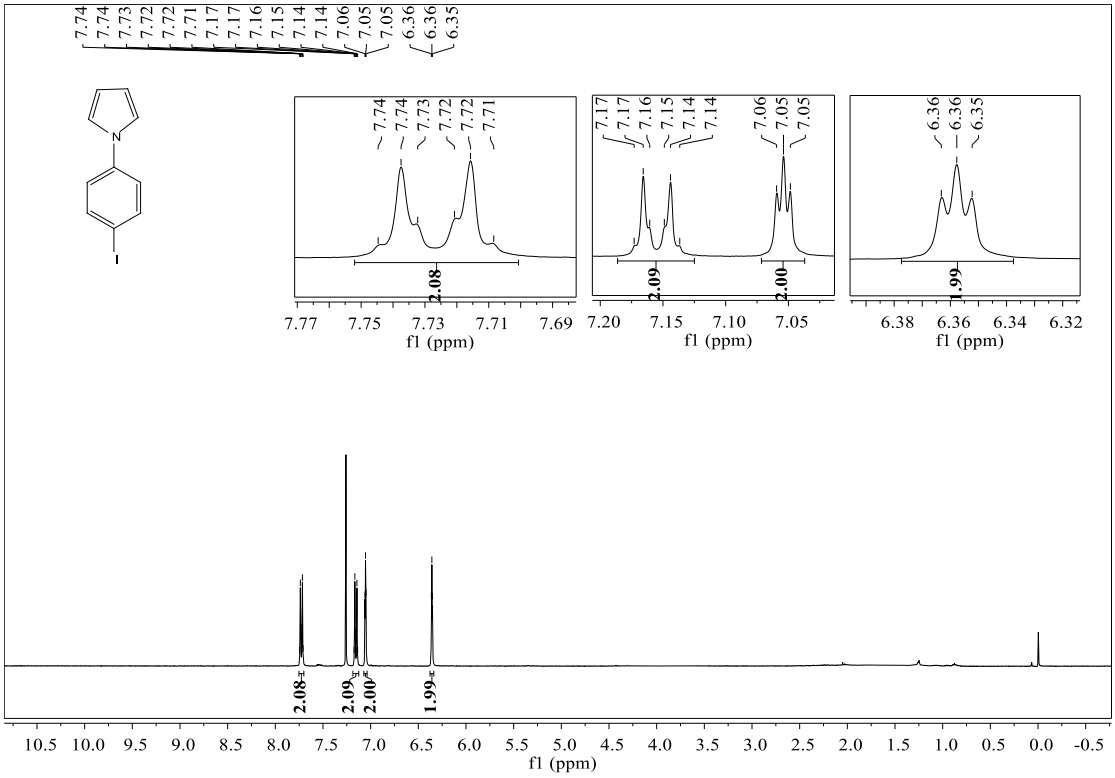
^{1}H NMR spectrum of compound 3m (400 MHz, $CDCl_3$)



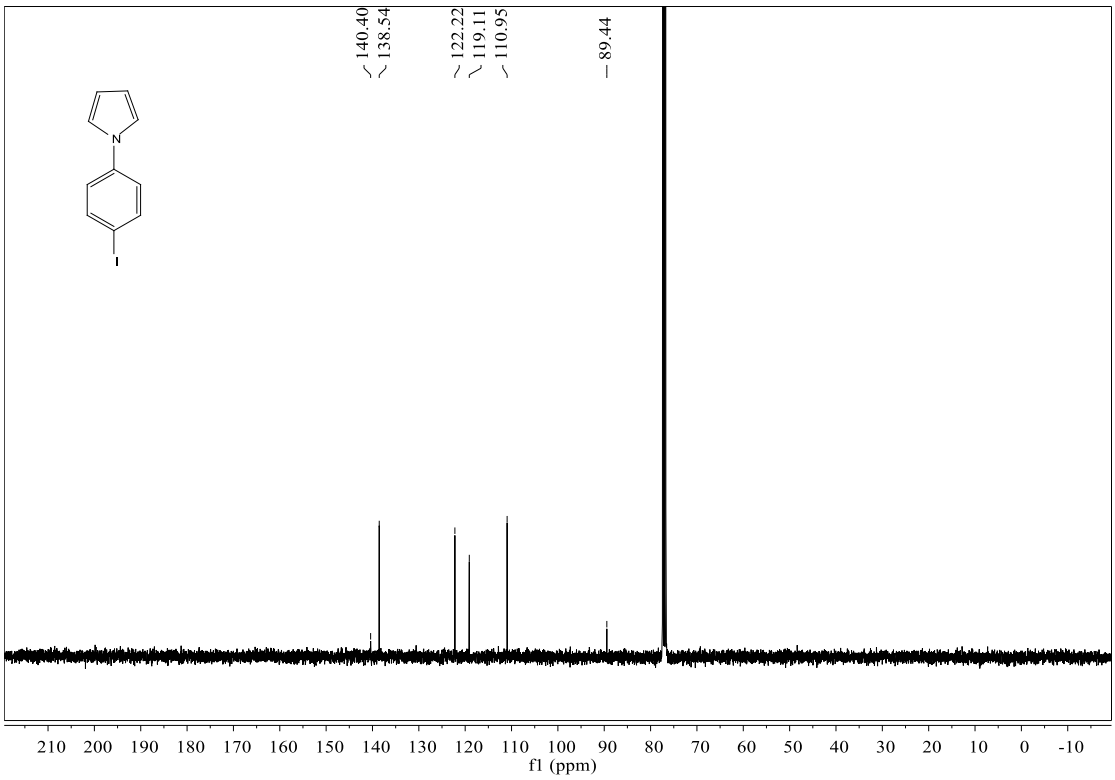
^{13}C NMR spectrum of compound 3m (101 MHz, $CDCl_3$)



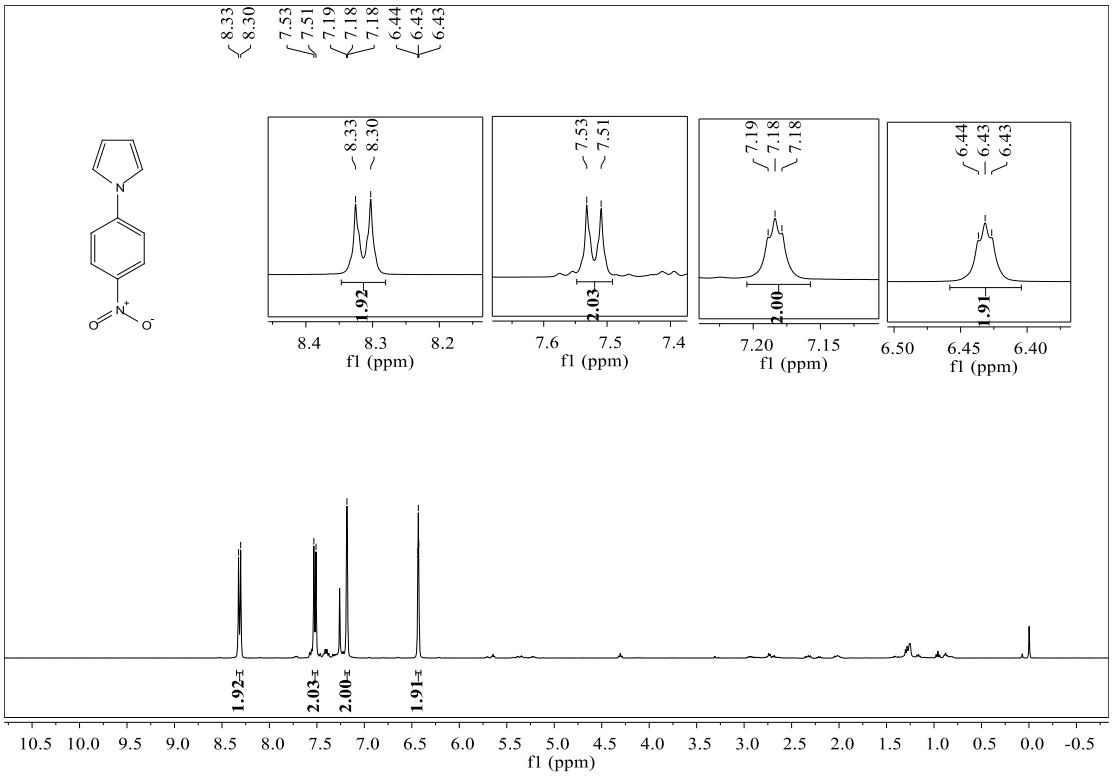
^{1}H NMR spectrum of compound 3n (400 MHz, $CDCl_3$)



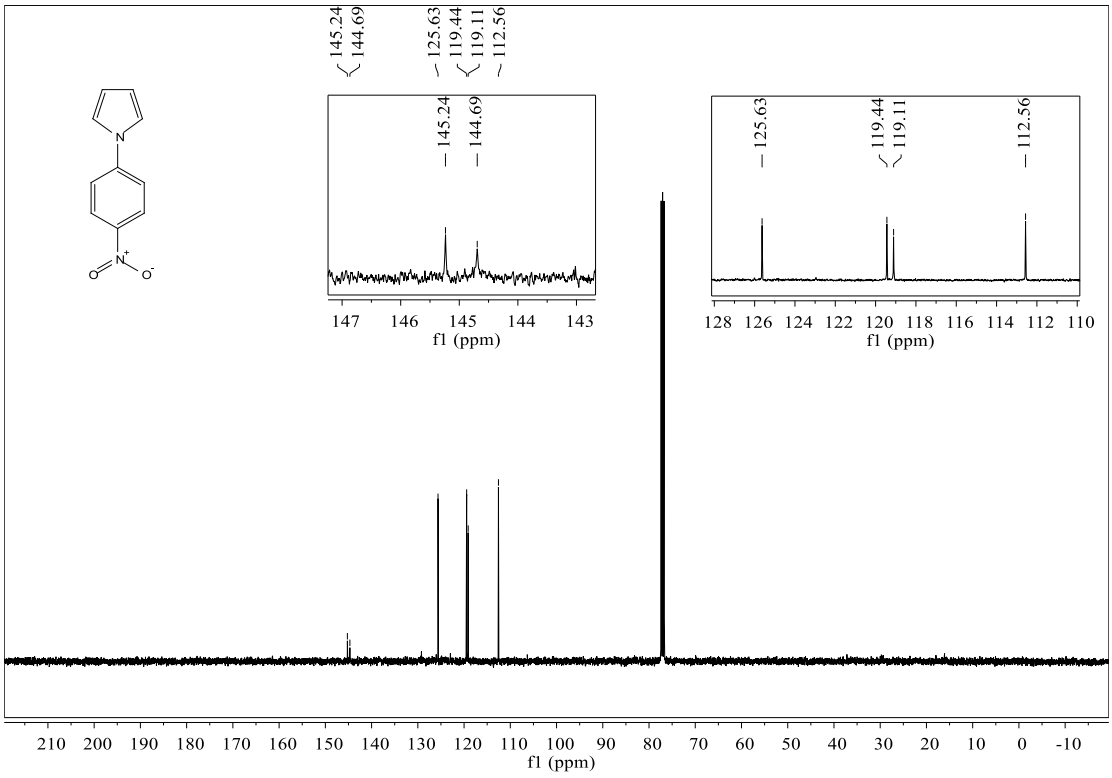
^{13}C NMR spectrum of compound 3n (101 MHz, $CDCl_3$)



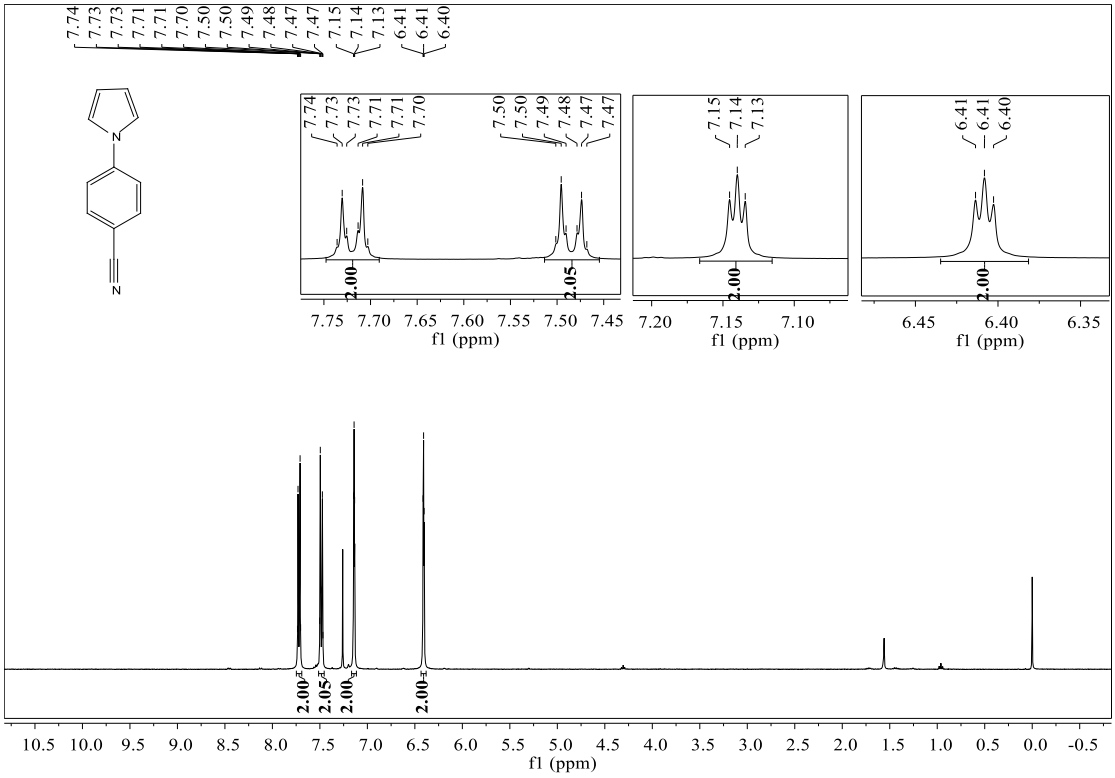
^{1}H NMR spectrum of compound 3o (400 MHz, $CDCl_3$)



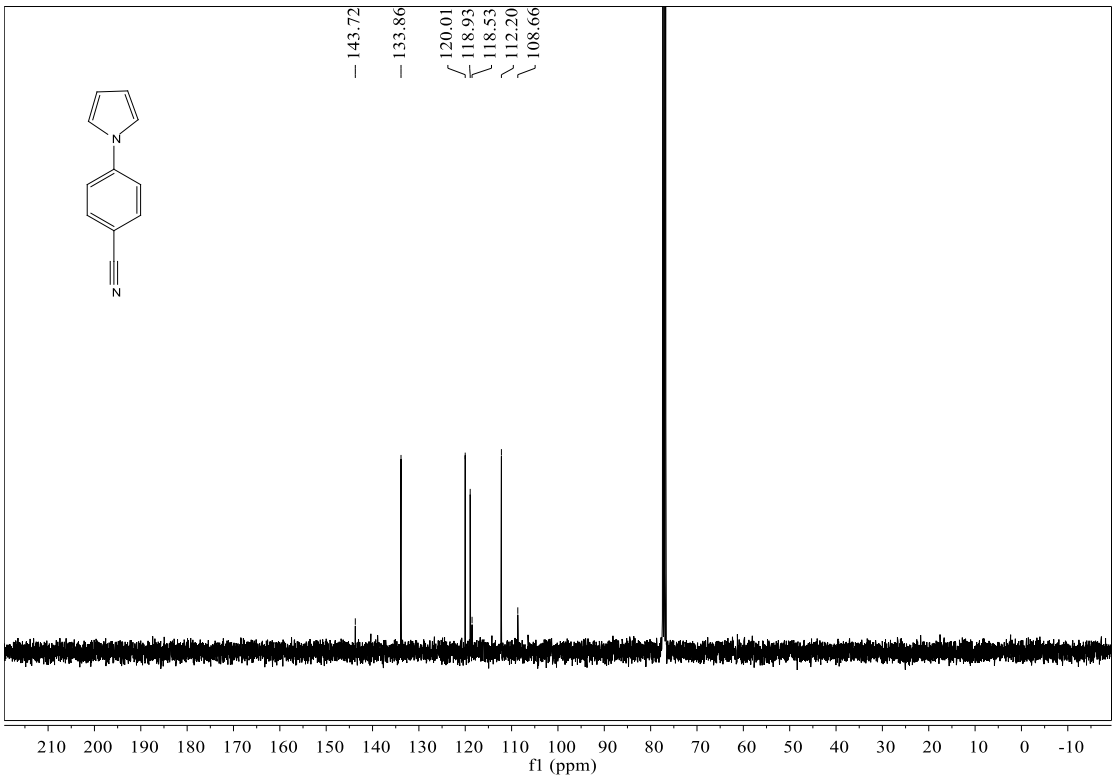
^{13}C NMR spectrum of compound 3o (101 MHz, $CDCl_3$)



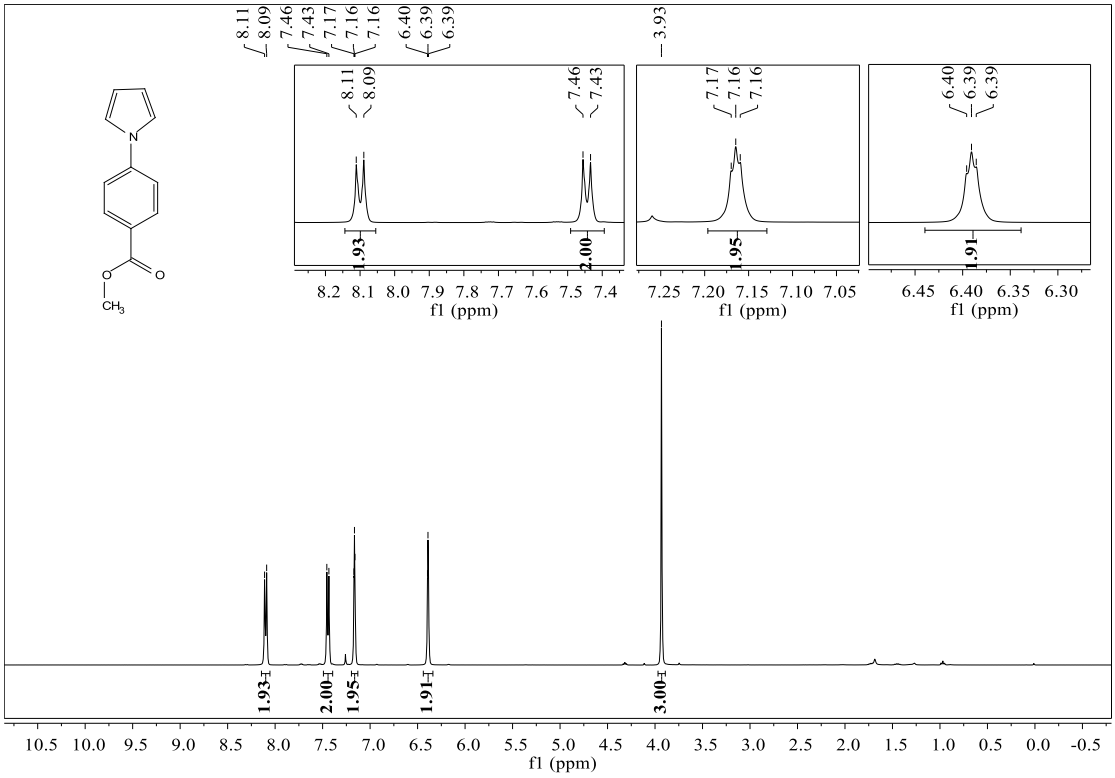
^{1}H NMR spectrum of compound 3p (400 MHz, $CDCl_3$)



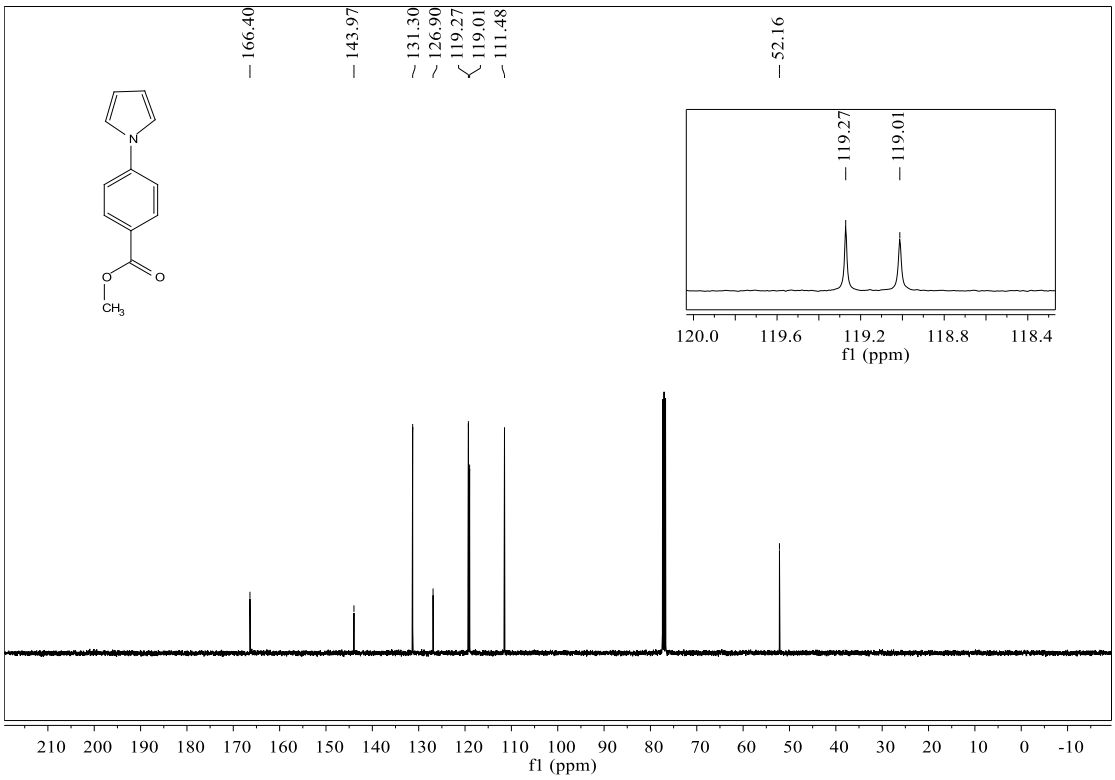
^{13}C NMR spectrum of compound 3p (101 MHz, $CDCl_3$)



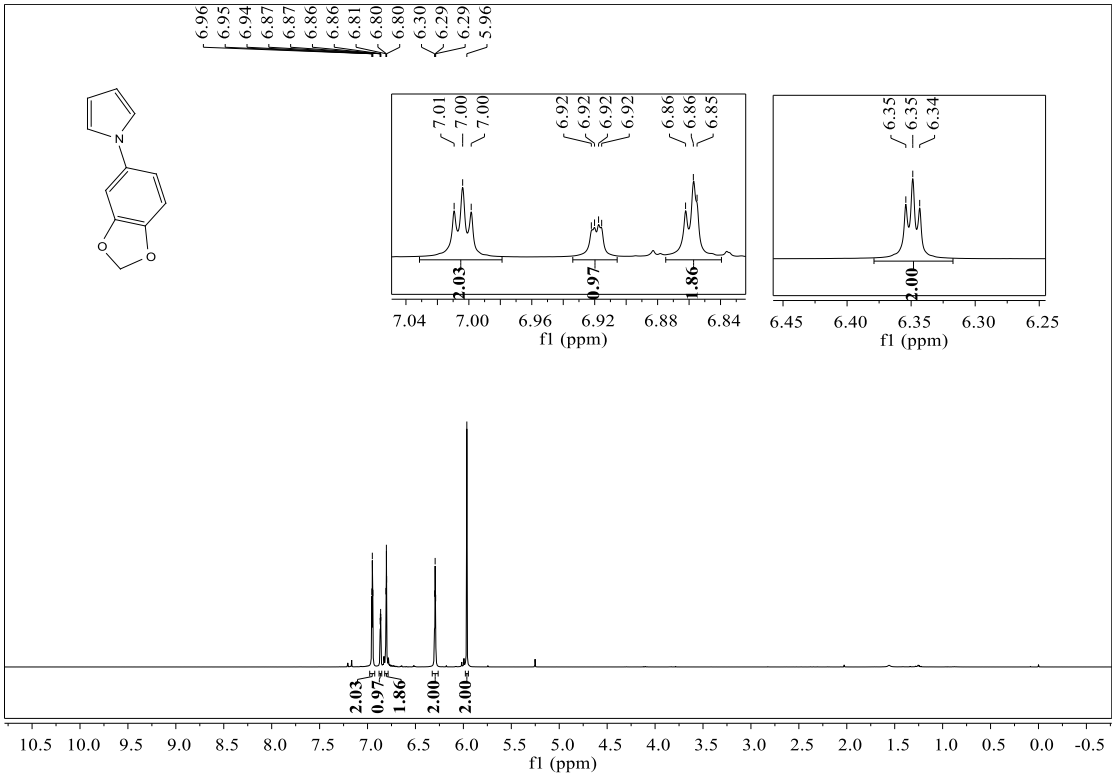
^{1}H NMR spectrum of compound 3q (400 MHz, $CDCl_3$)



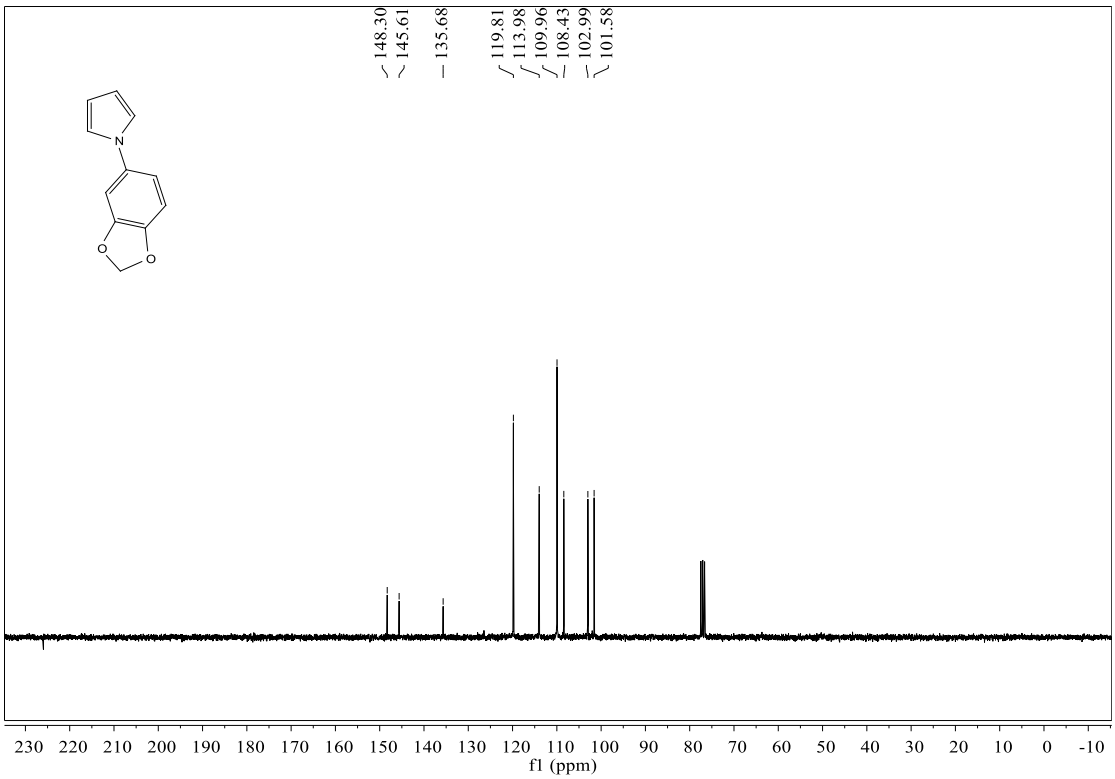
^{13}C NMR spectrum of compound 3q (101 MHz, $CDCl_3$)



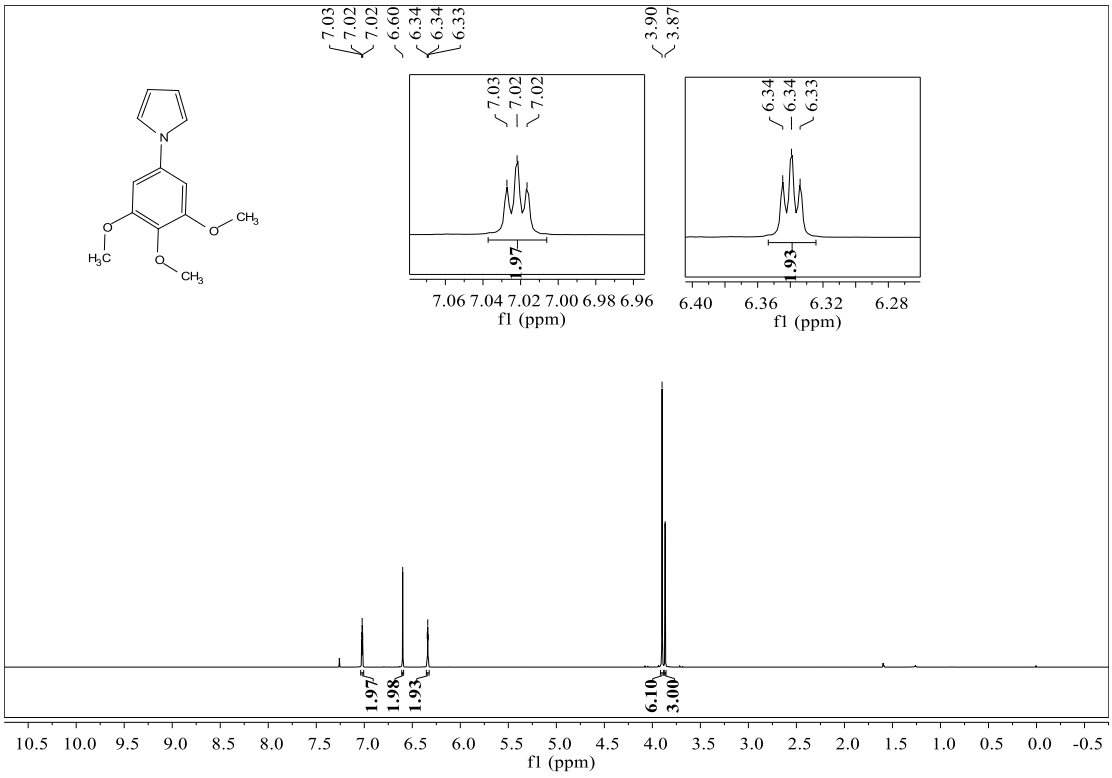
^{1}H NMR spectrum of compound 3r (400 MHz, $CDCl_3$)



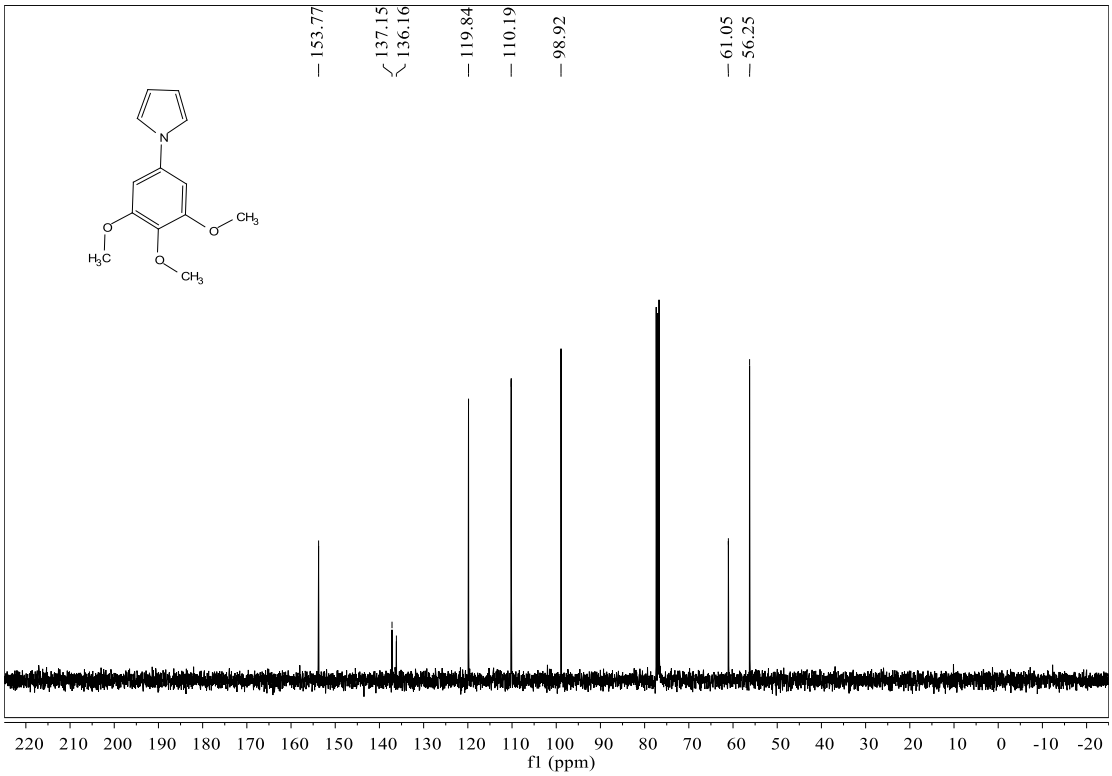
^{13}C NMR spectrum of compound 3r (76 MHz, $CDCl_3$)



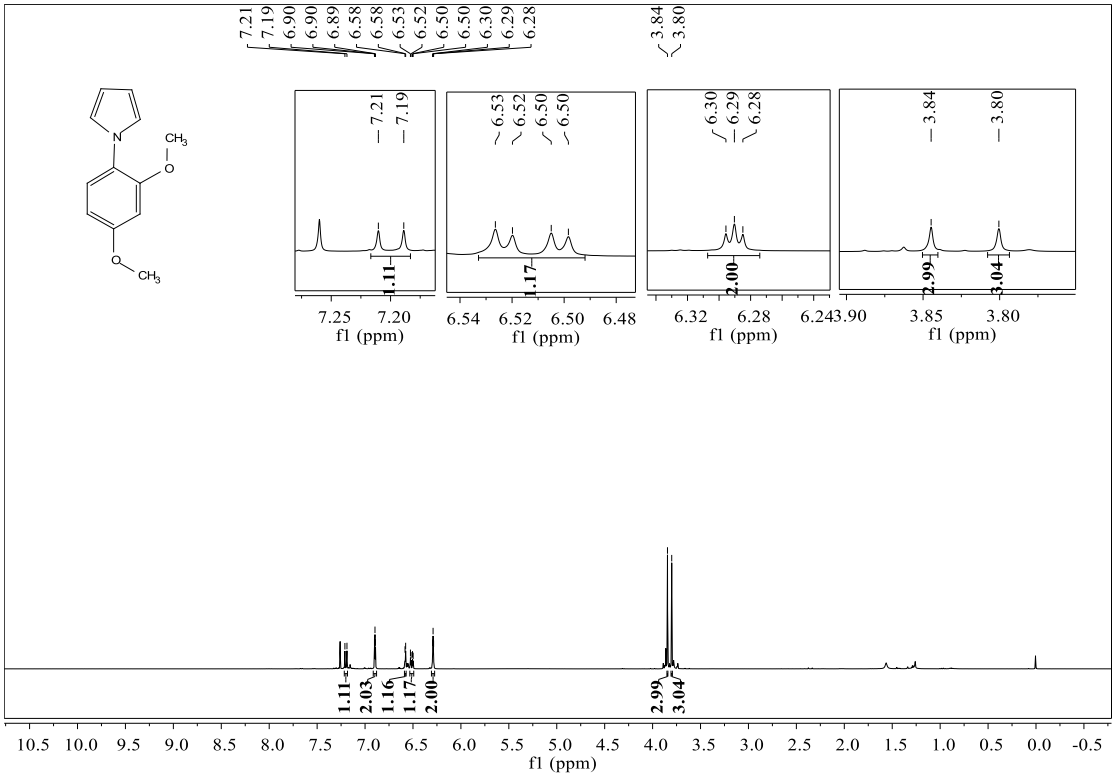
^{1}H NMR spectrum of compound 3s (400 MHz, $CDCl_3$)



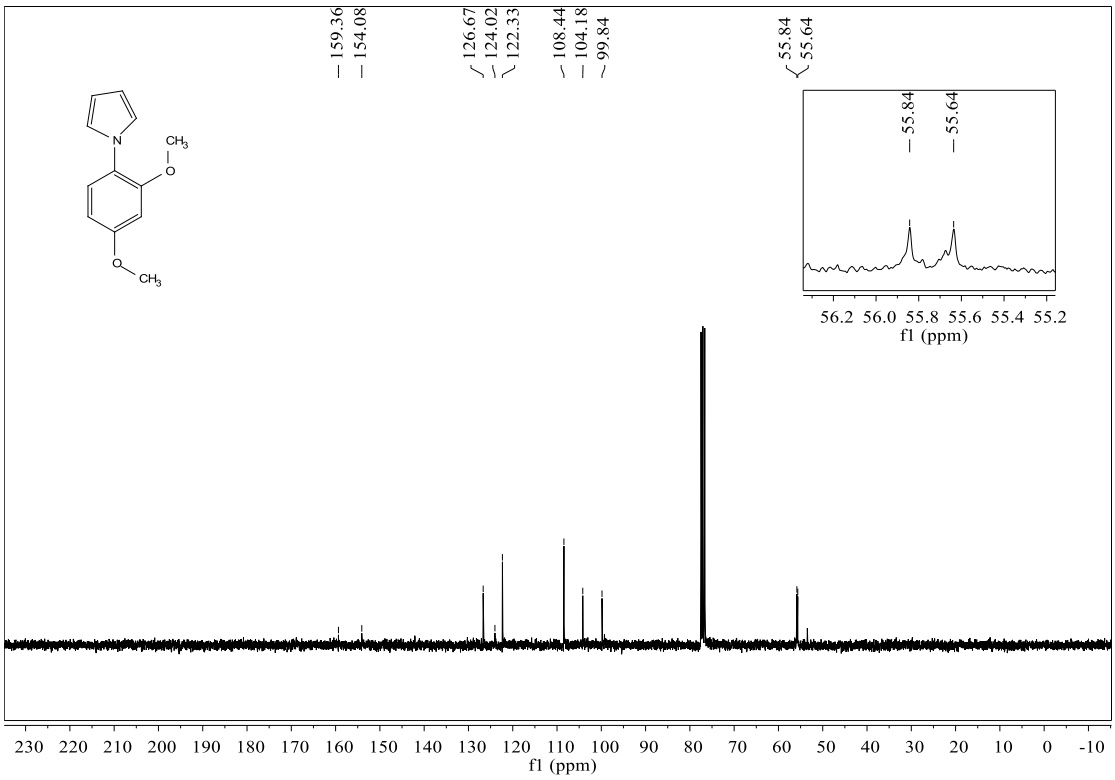
^{13}C NMR spectrum of compound 3s (101 MHz, $CDCl_3$)



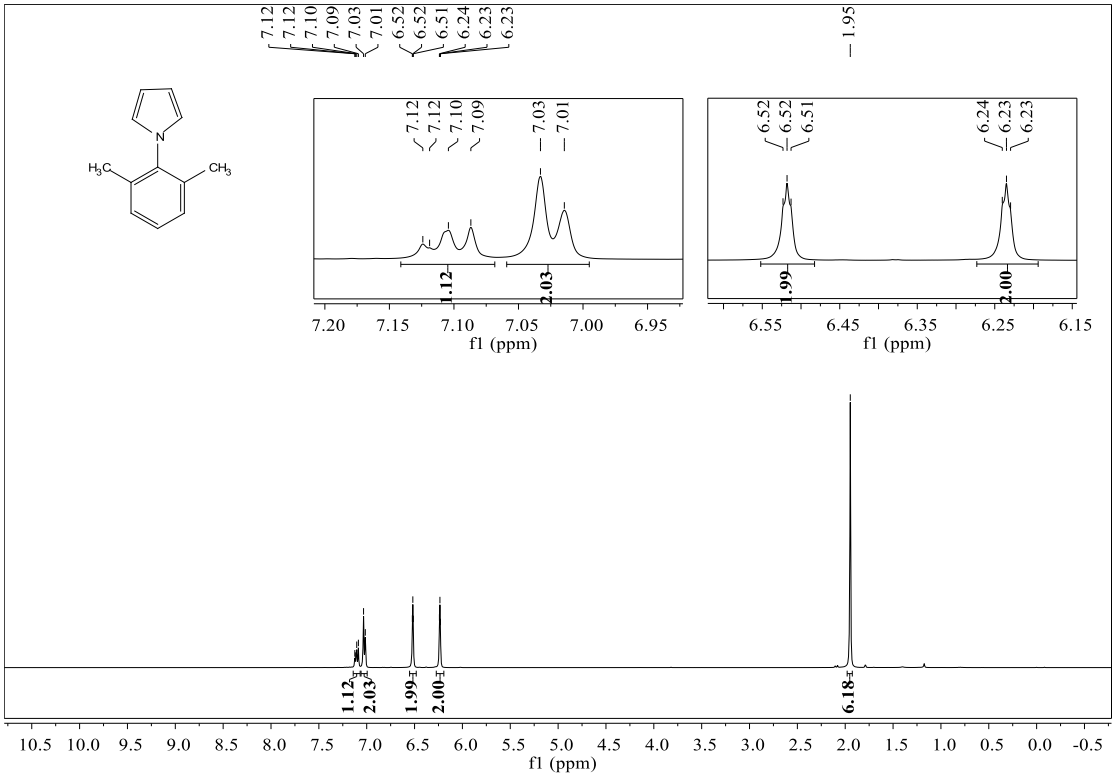
^{1}H NMR spectrum of compound 3t (400 MHz, $CDCl_3$)



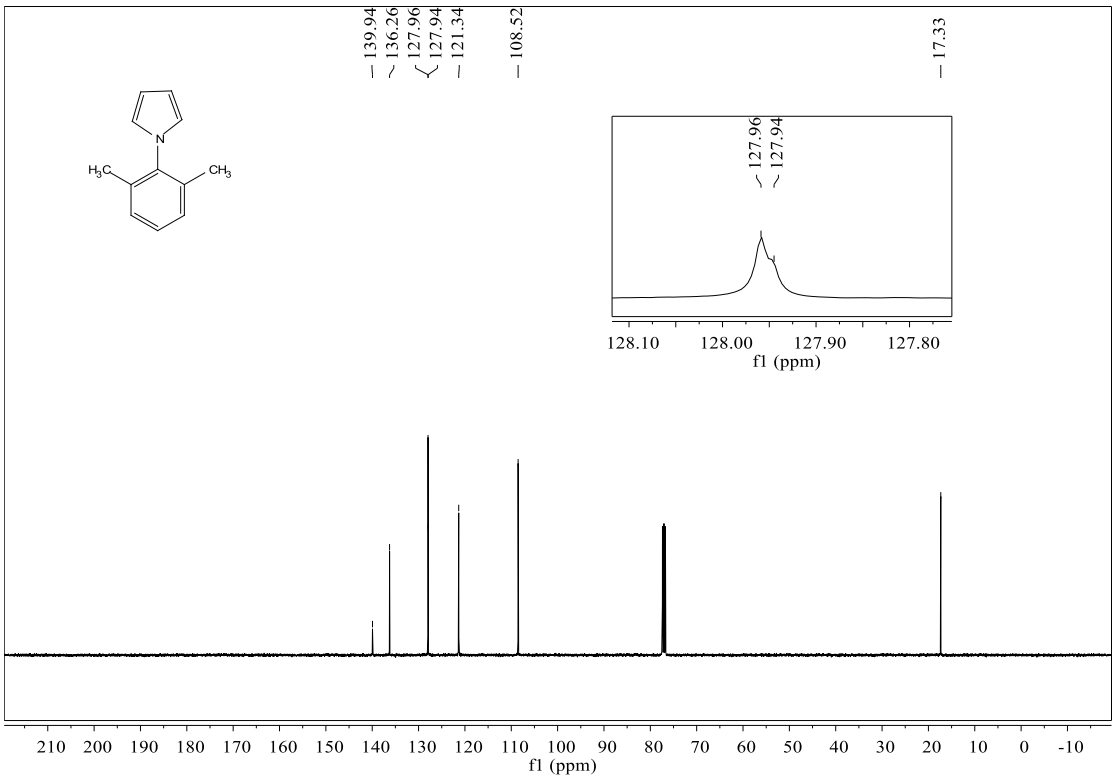
^{13}C NMR spectrum of compound 3t (76 MHz, $CDCl_3$)



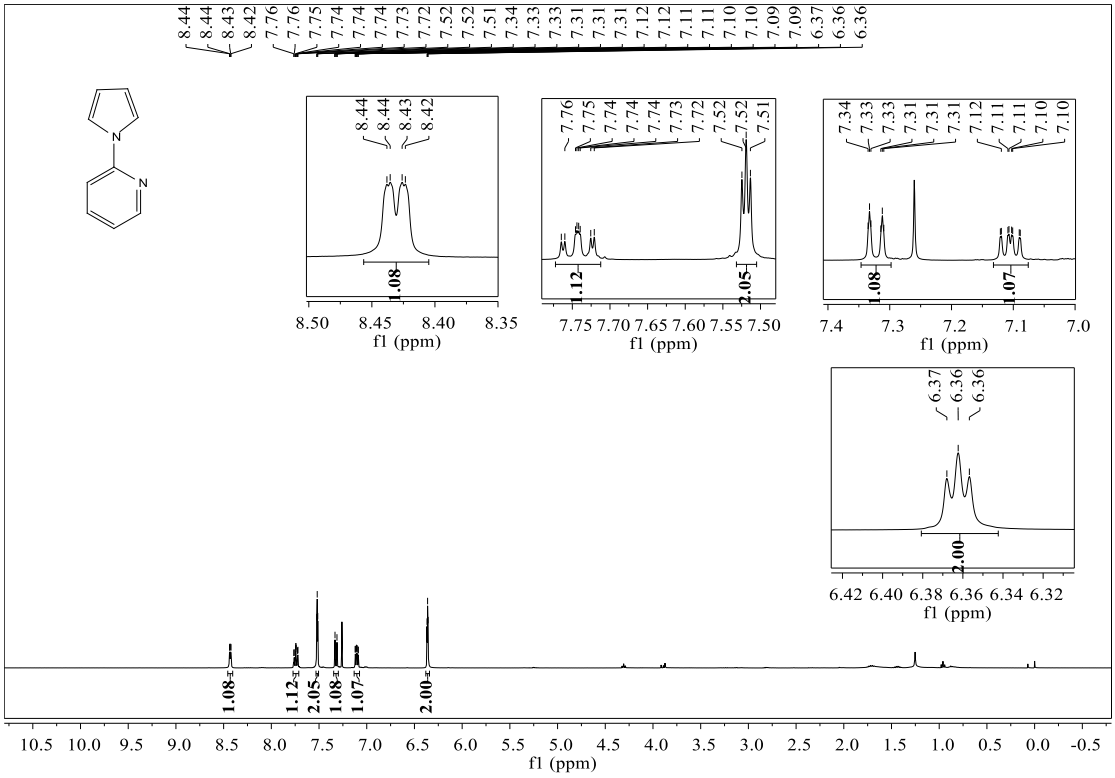
^{1}H NMR spectrum of compound 3u (400 MHz, $CDCl_3$)



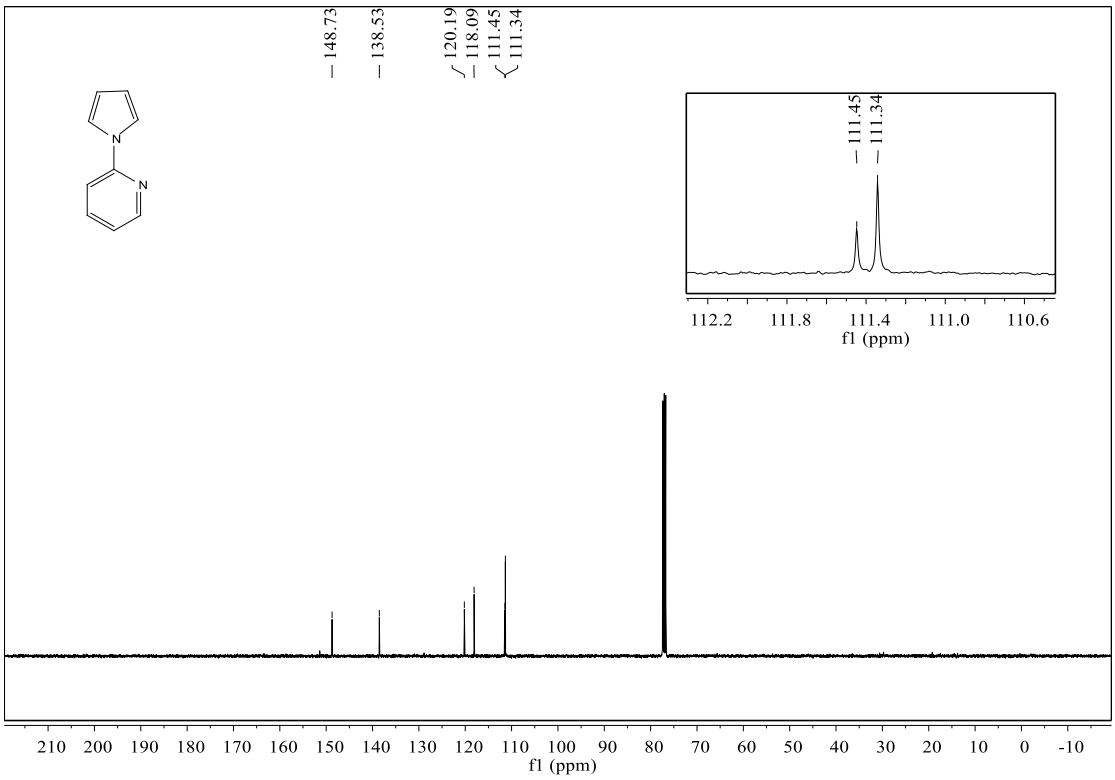
^{13}C NMR spectrum of compound 3u (101 MHz, $CDCl_3$)



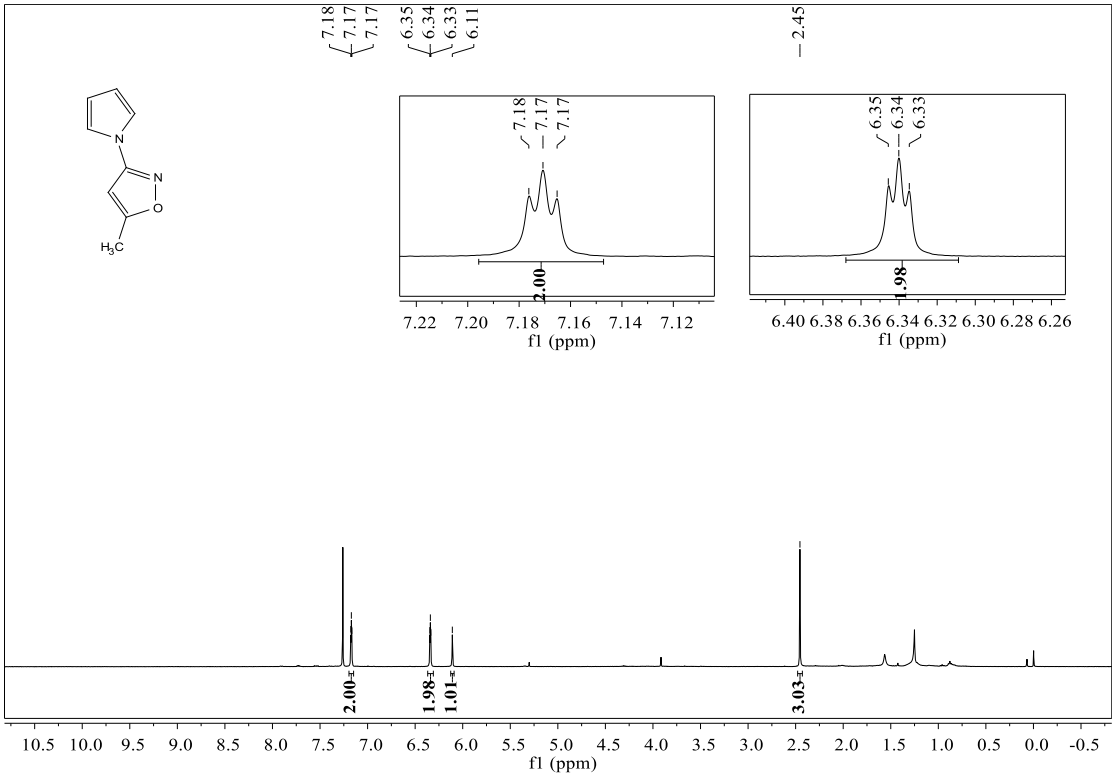
^{1}H NMR spectrum of compound 3v (400 MHz, $CDCl_3$)



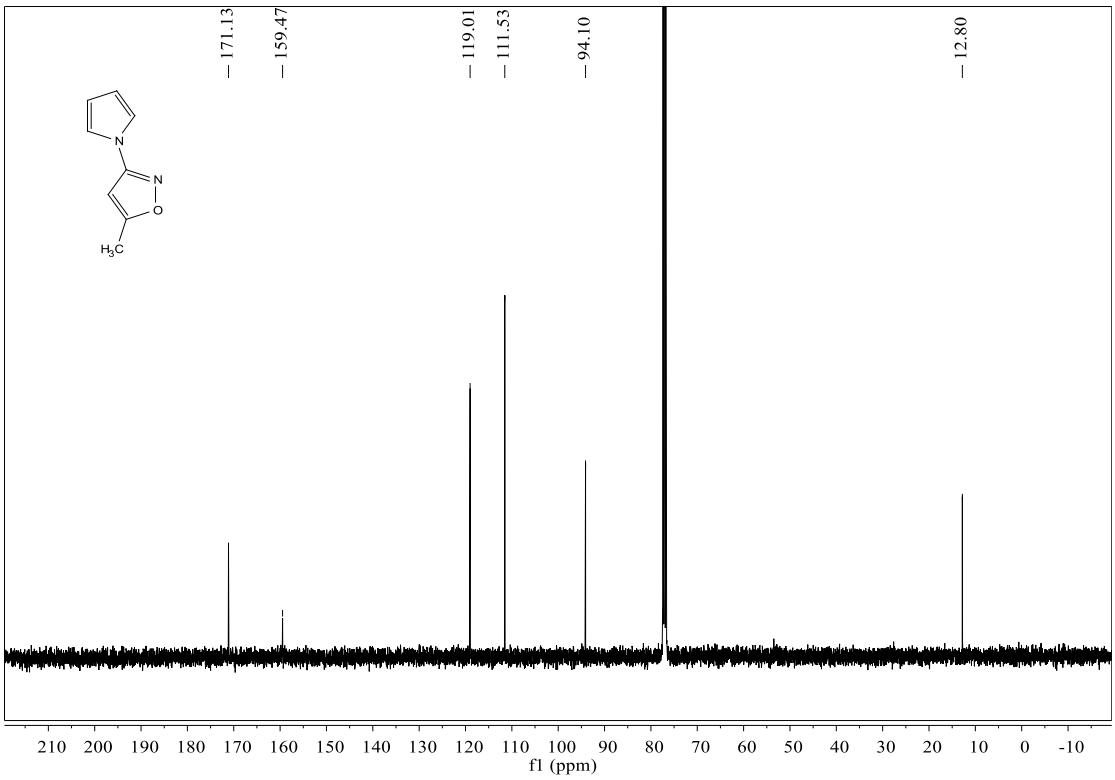
^{13}C NMR spectrum of compound 3v (101 MHz, $CDCl_3$)



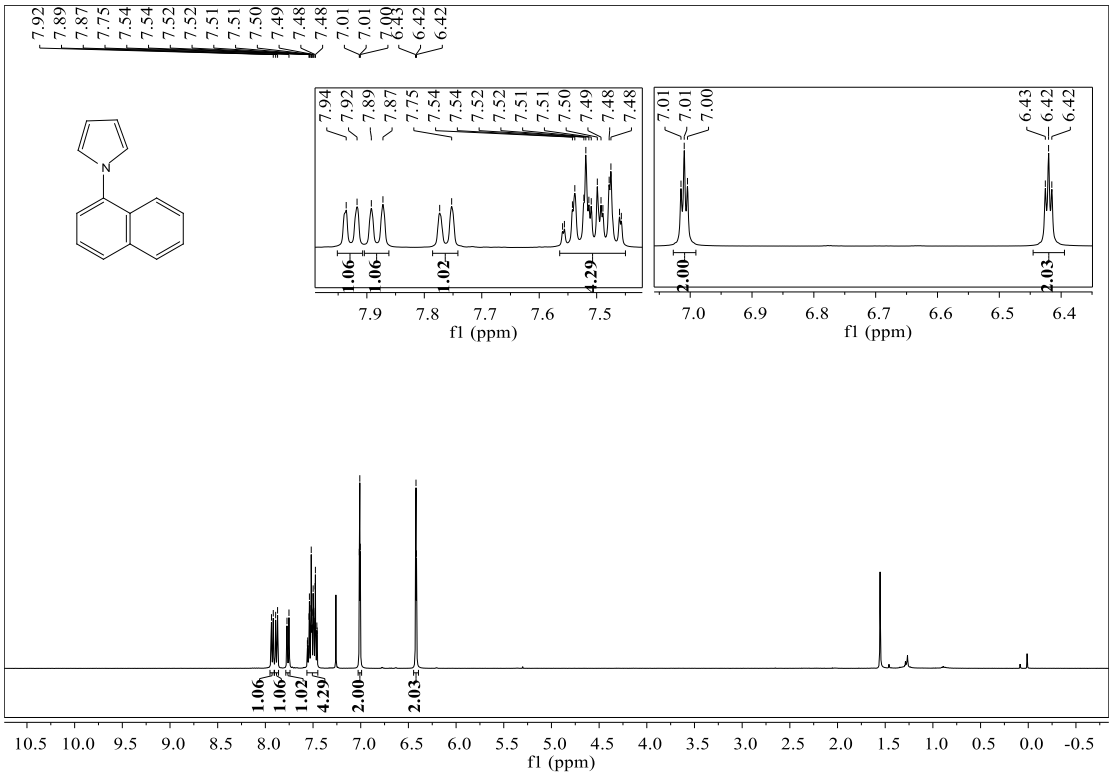
^{1}H NMR spectrum of compound 3w (400 MHz, $CDCl_3$)



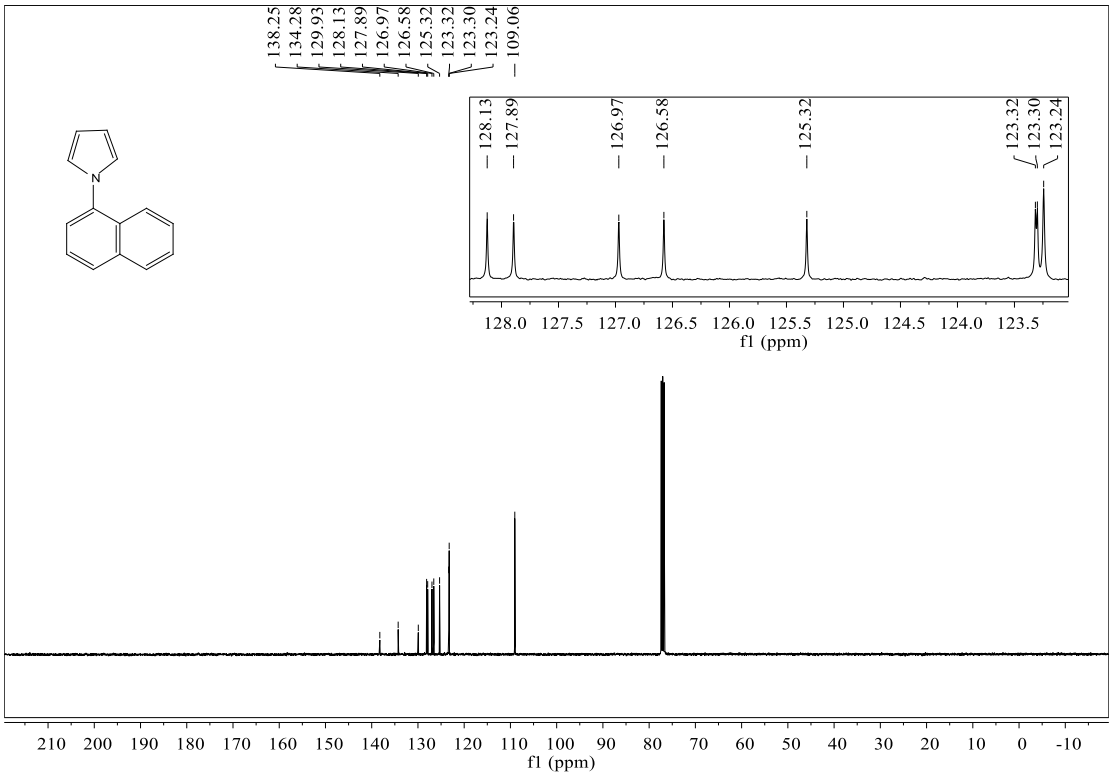
^{13}C NMR spectrum of compound 3w (101 MHz, $CDCl_3$)



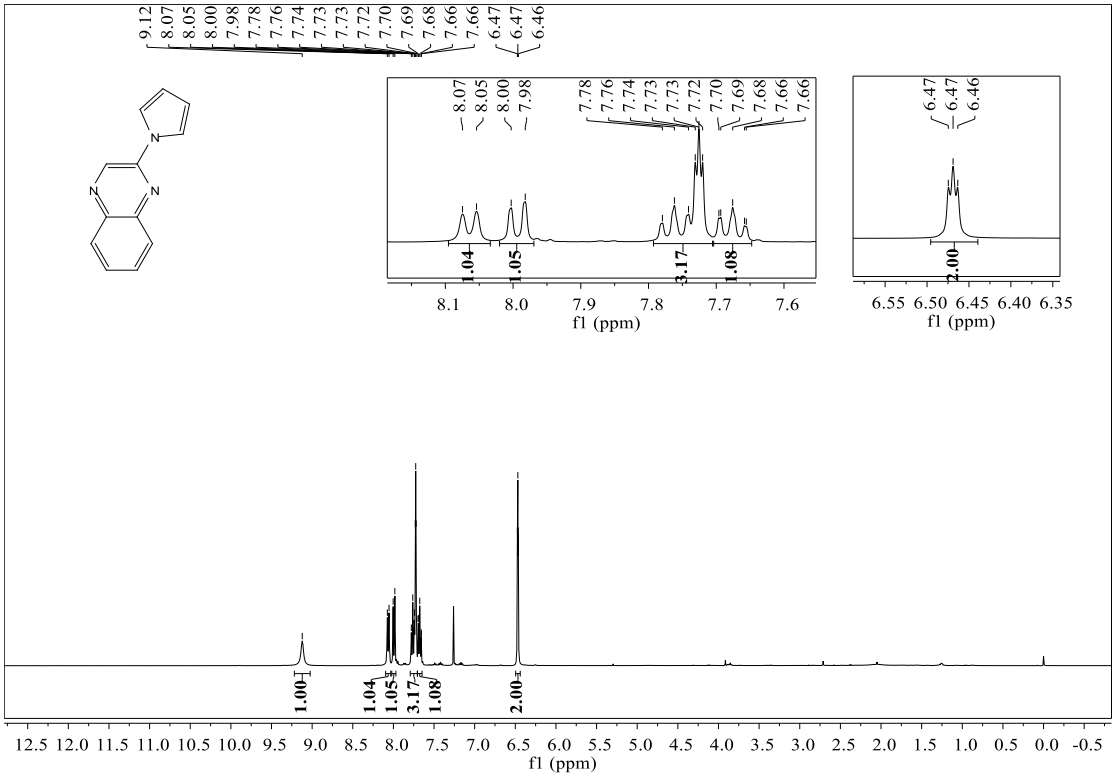
^{1}H NMR spectrum of compound 3x (400 MHz, $CDCl_3$)



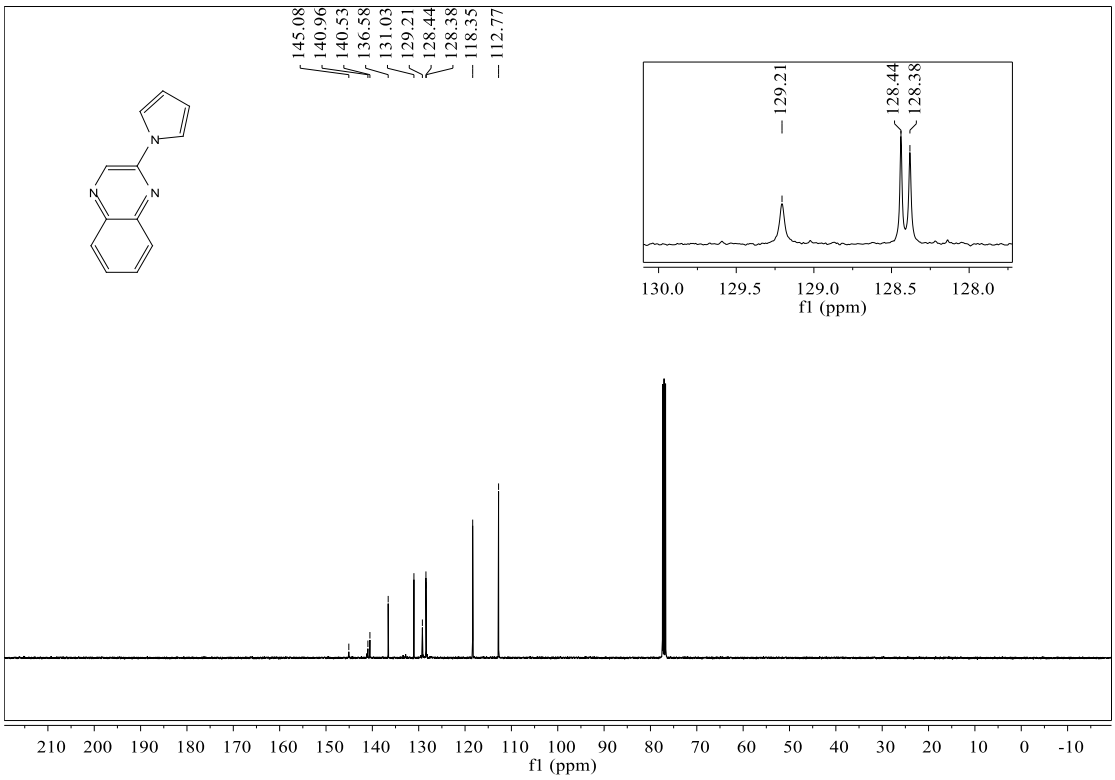
^{13}C NMR spectrum of compound 3x (101 MHz, $CDCl_3$)



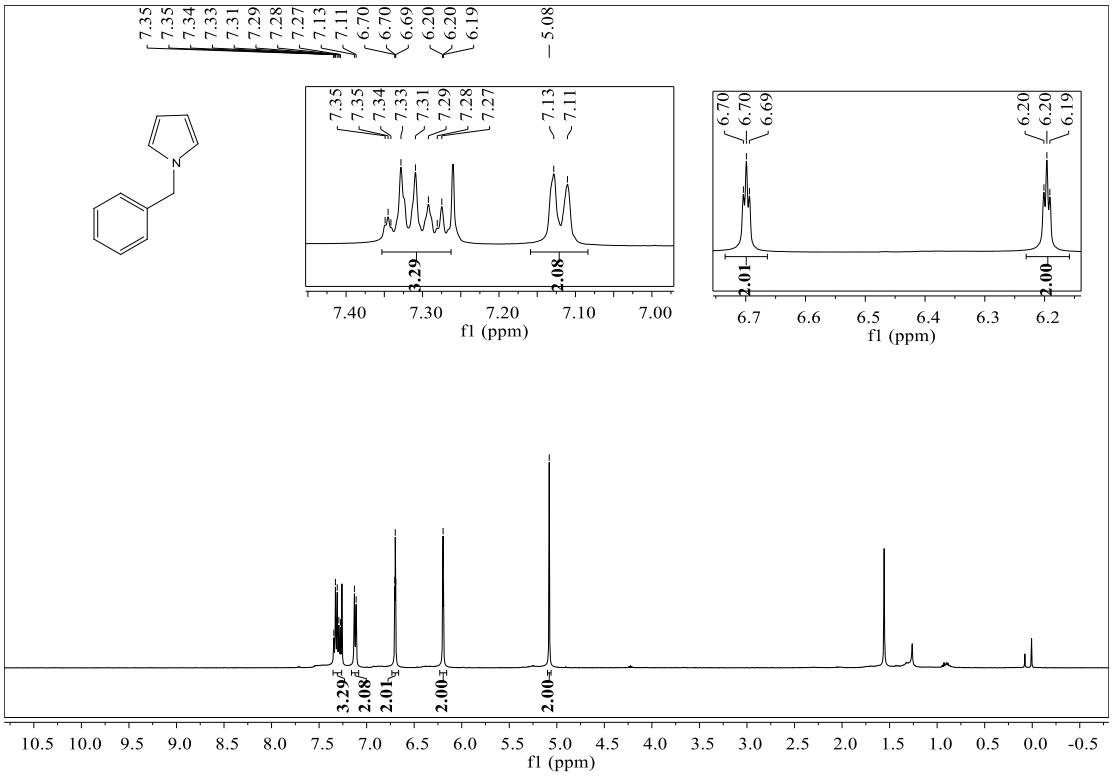
^{1}H NMR spectrum of compound 3y (400 MHz, $CDCl_3$)



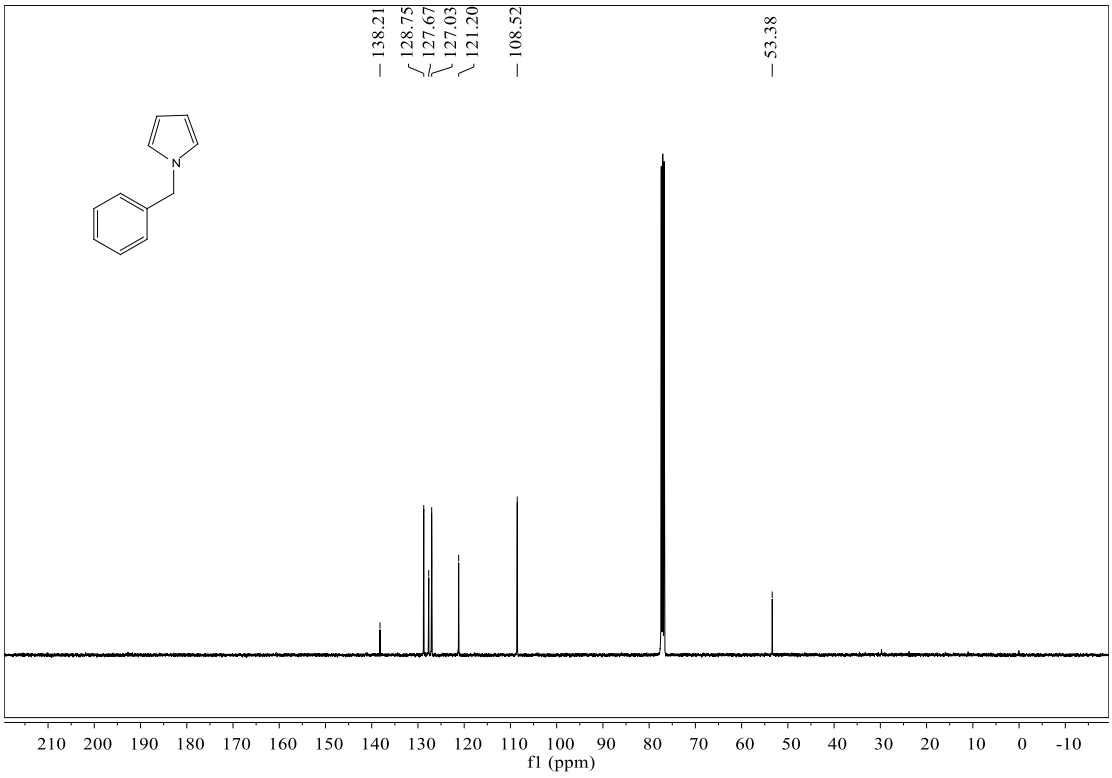
^{13}C NMR spectrum of compound 3y (101 MHz, $CDCl_3$)



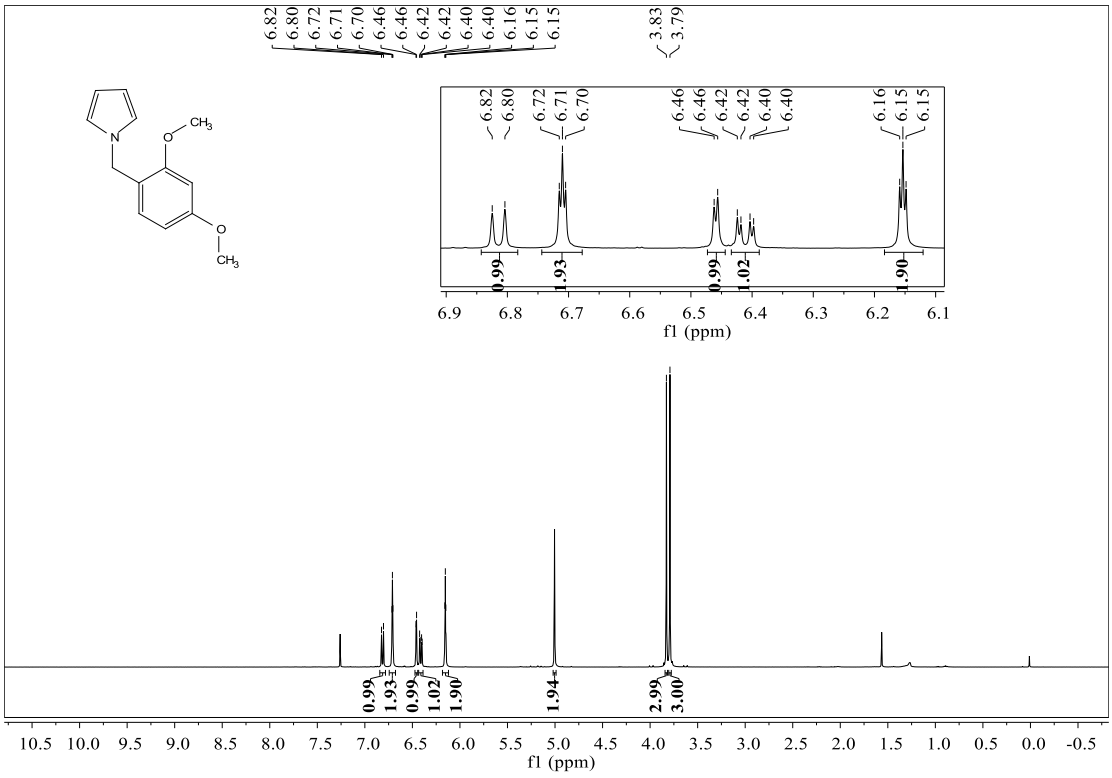
^{1}H NMR spectrum of compound 3z (400 MHz, $CDCl_3$)



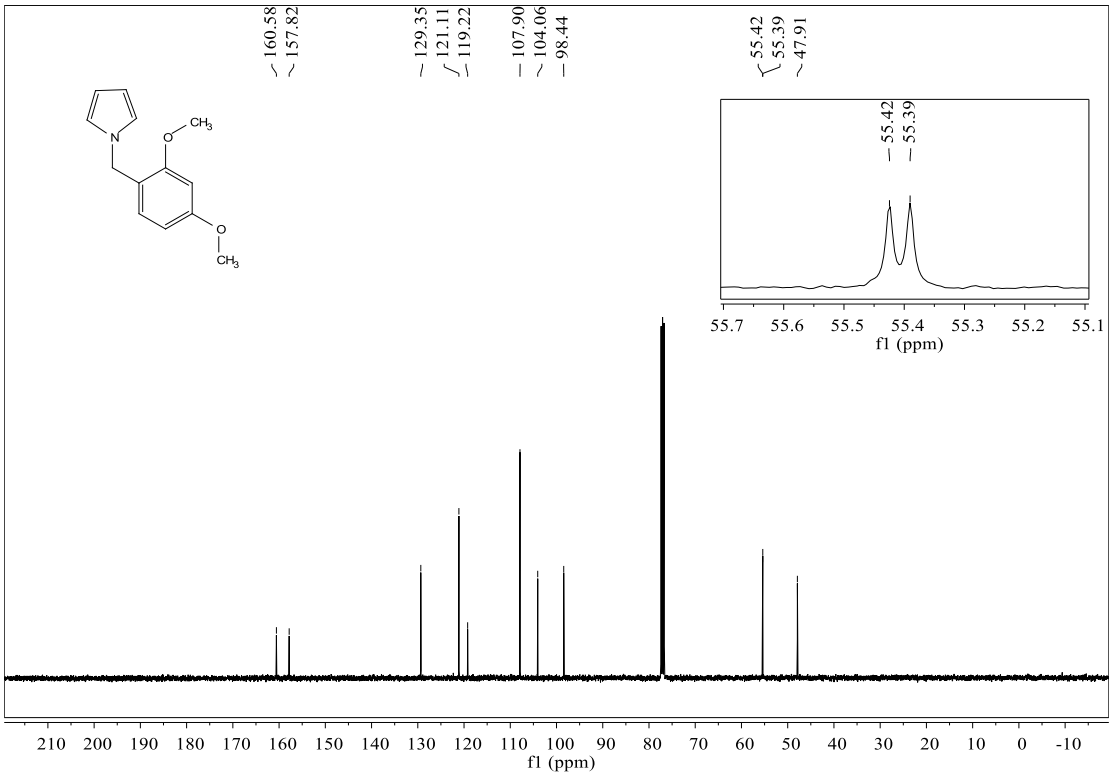
^{13}C NMR spectrum of compound 3z (101 MHz, $CDCl_3$)



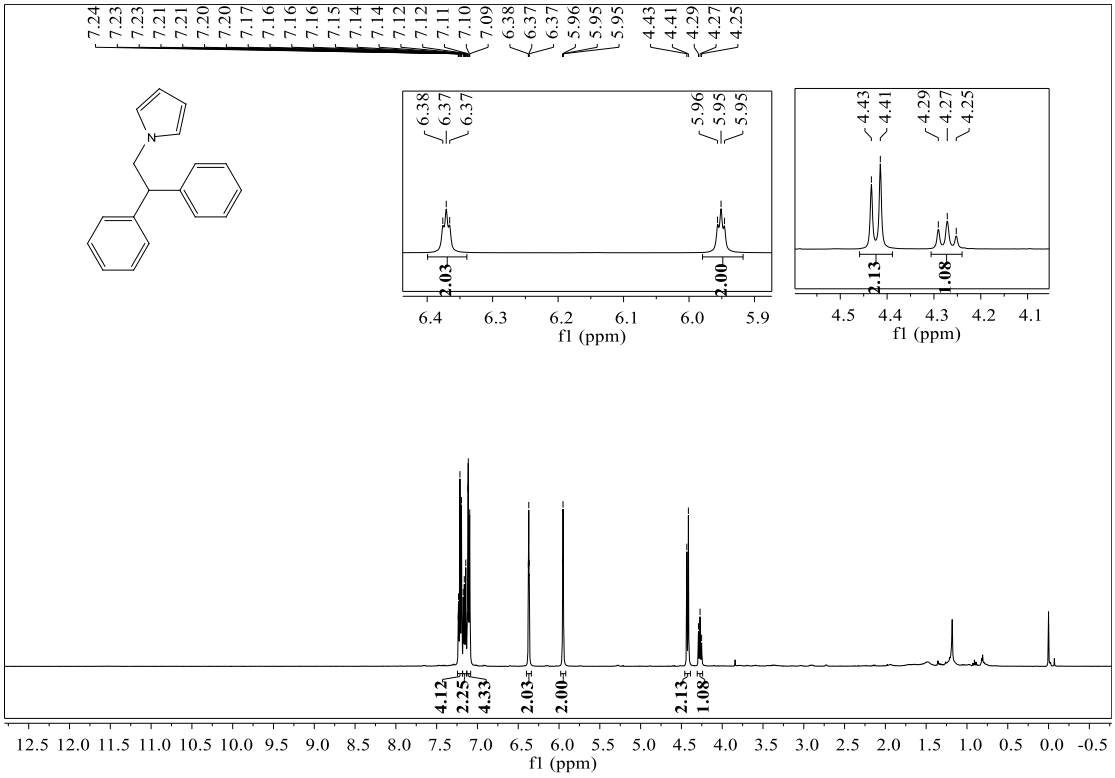
^{1}H NMR spectrum of compound 3aa (400 MHz, $CDCl_3$)



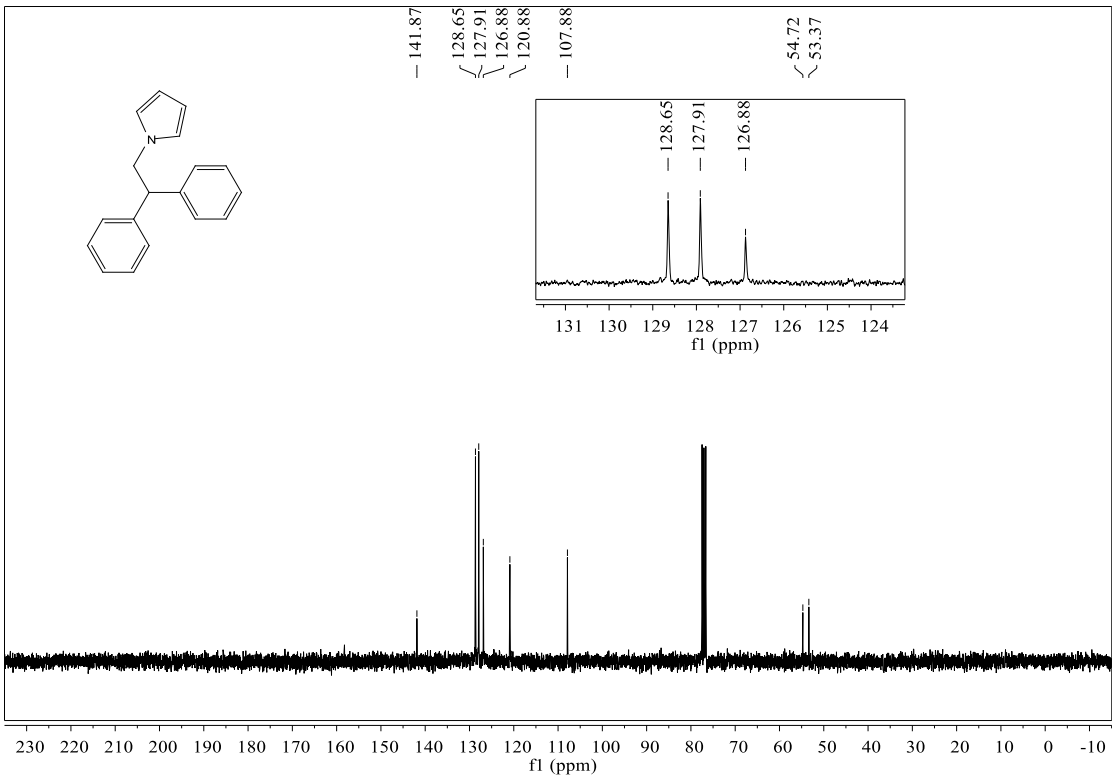
^{13}C NMR spectrum of compound 3aa (101 MHz, $CDCl_3$)



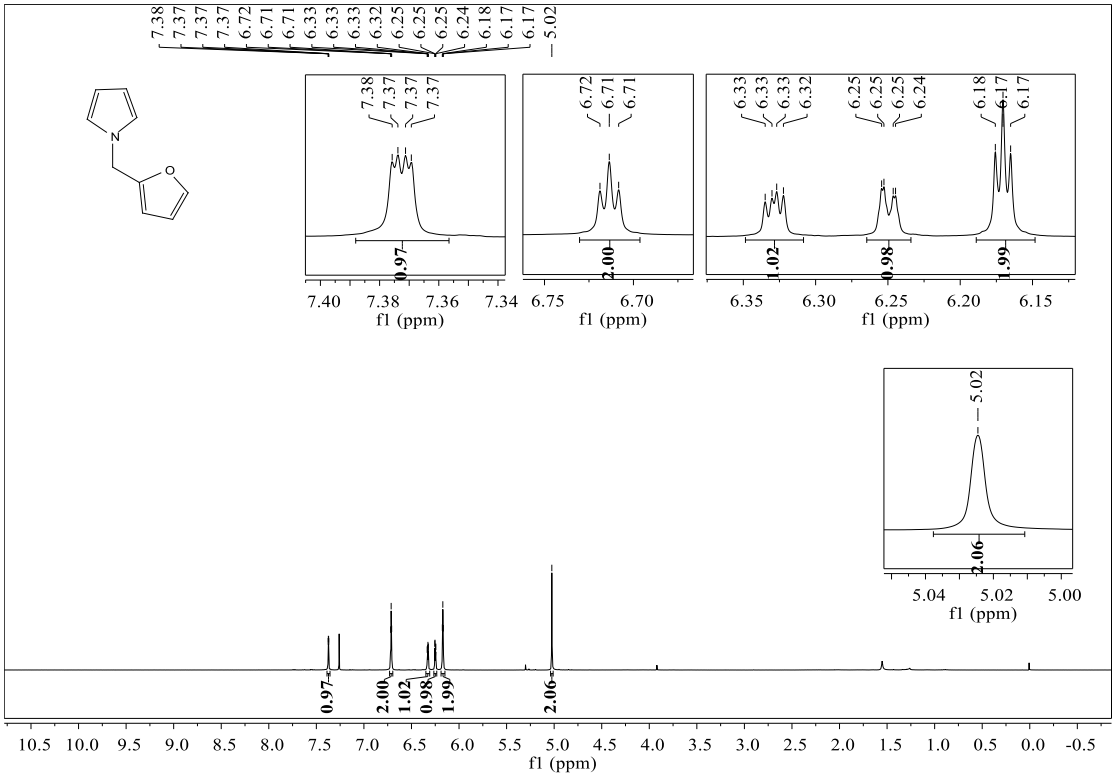
^{1}H NMR spectrum of compound 3ab (400 MHz, $CDCl_3$)



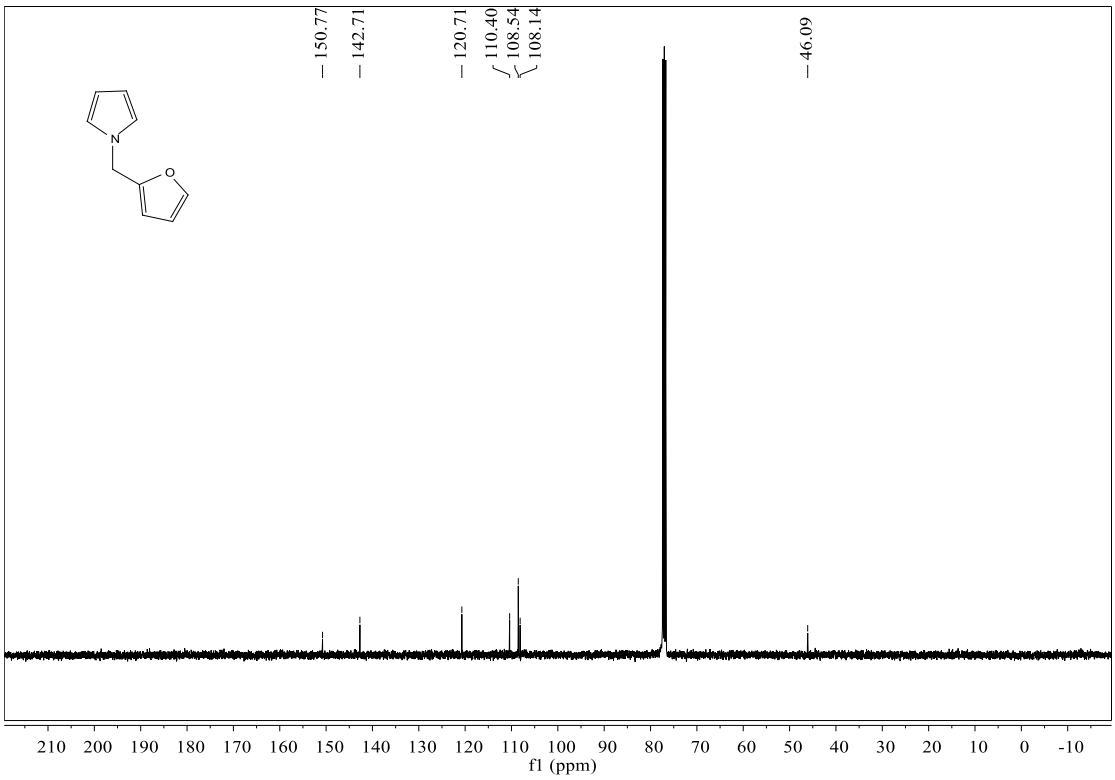
^{13}C NMR spectrum of compound 3ab (76 MHz, $CDCl_3$)



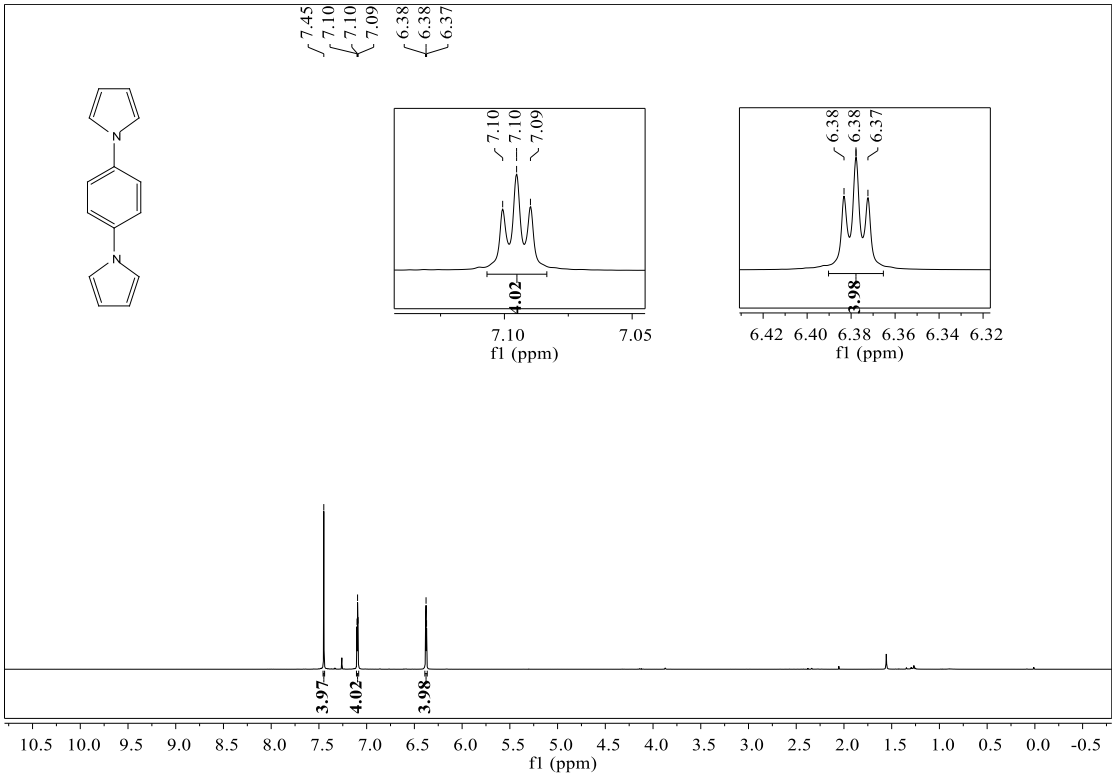
^{1}H NMR spectrum of compound 3ac (400 MHz, $CDCl_3$)



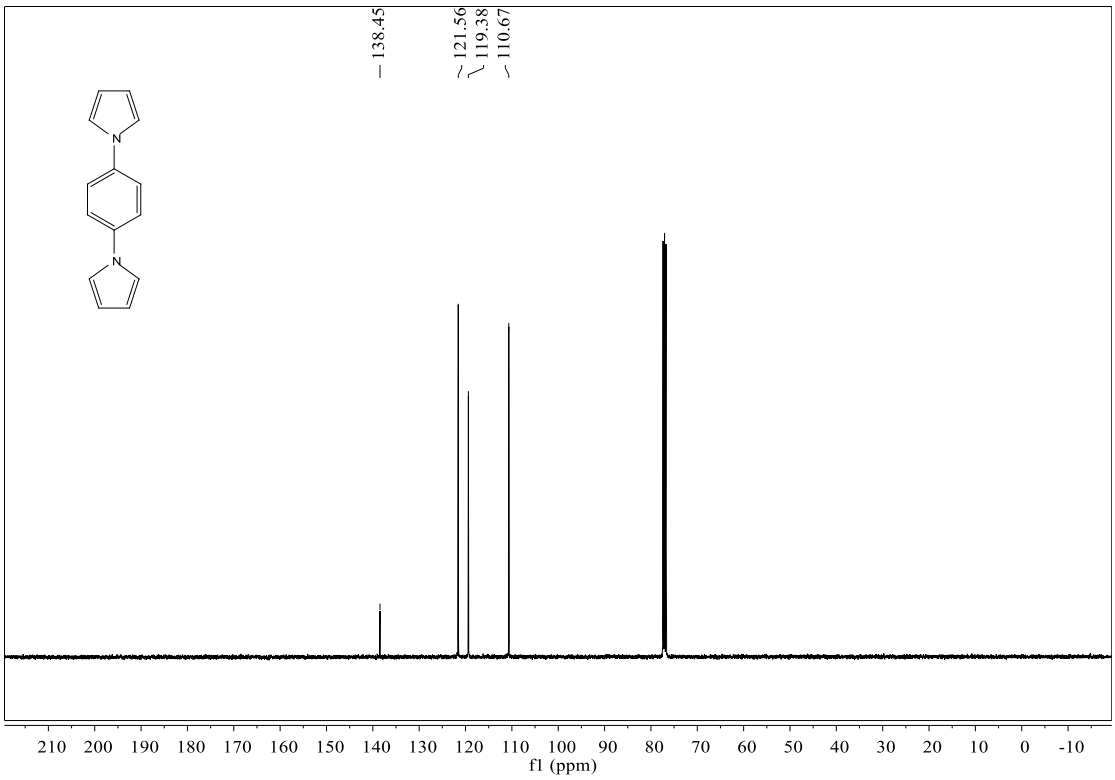
^{13}C NMR spectrum of compound 3ac (101 MHz, $CDCl_3$)



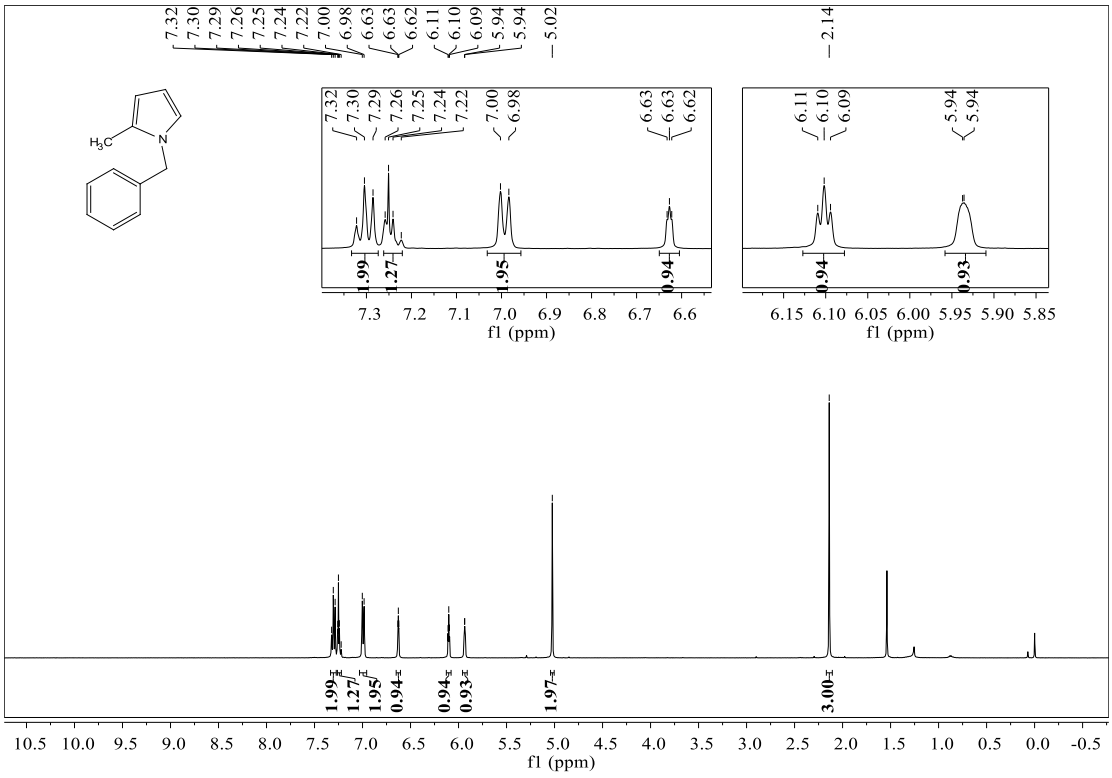
^{1}H NMR spectrum of compound 3ad (600 MHz, $CDCl_3$)



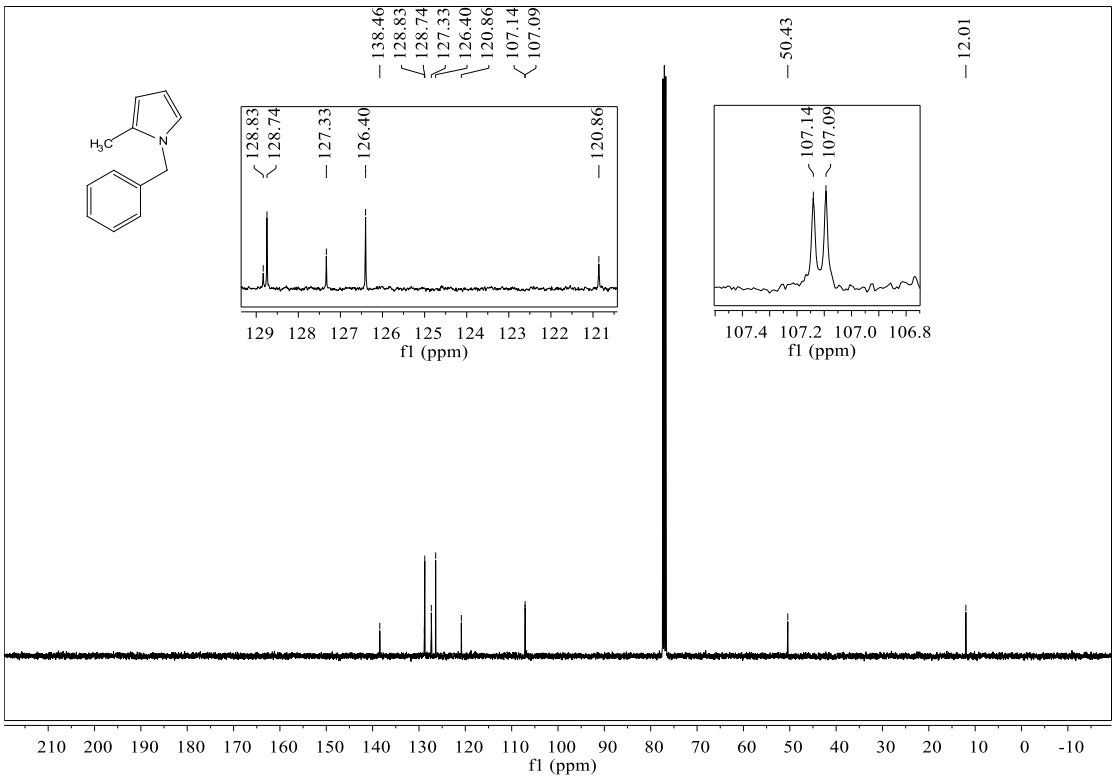
^{13}C NMR spectrum of compound 3ad (101 MHz, $CDCl_3$)



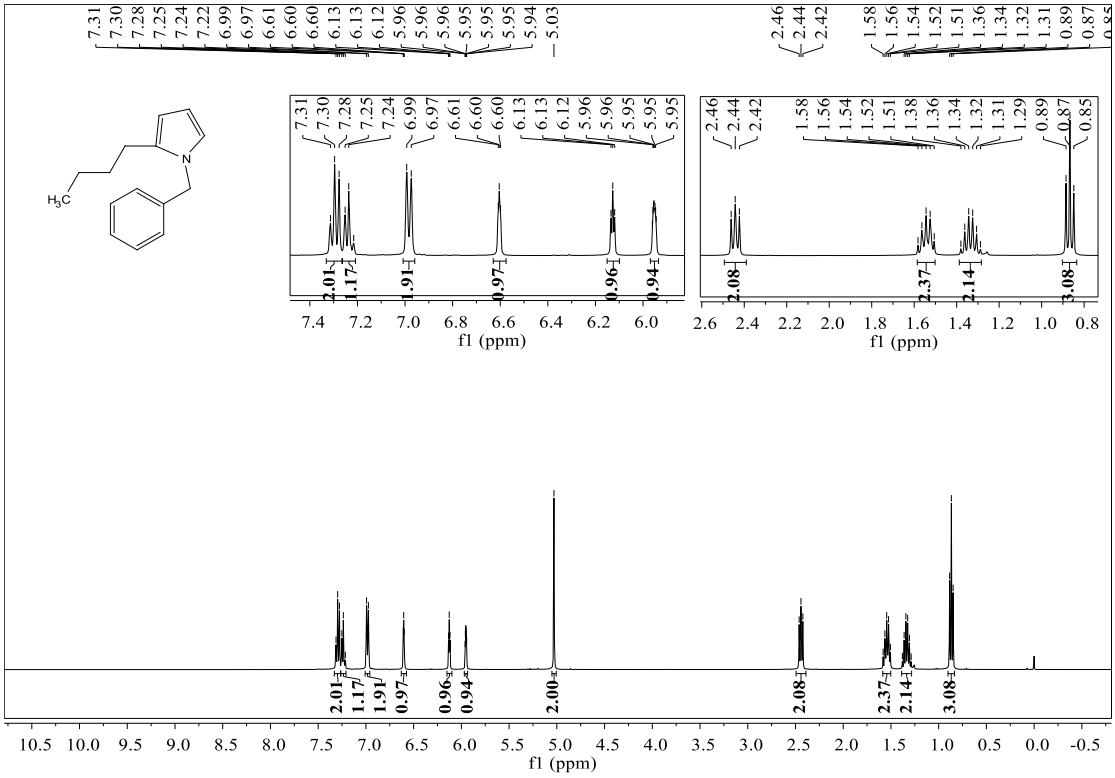
^{1}H NMR spectrum of compound 3ae (400 MHz, $CDCl_3$)



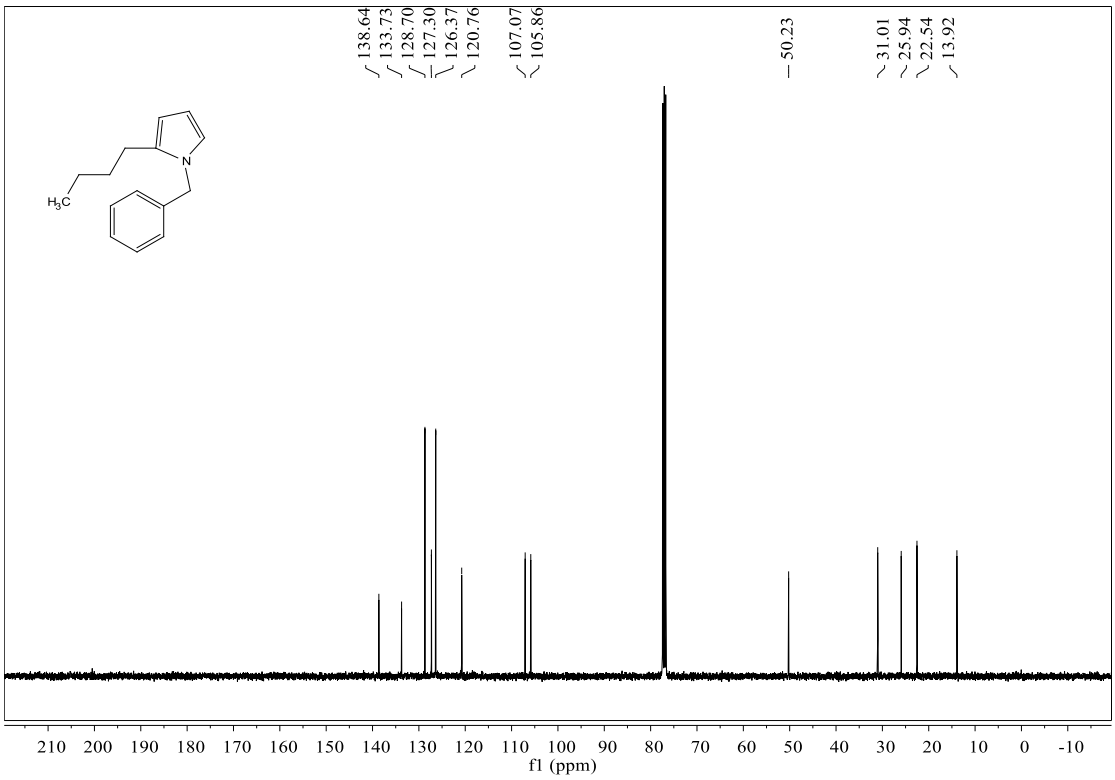
^{13}C NMR spectrum of compound 3ae (101 MHz, $CDCl_3$)



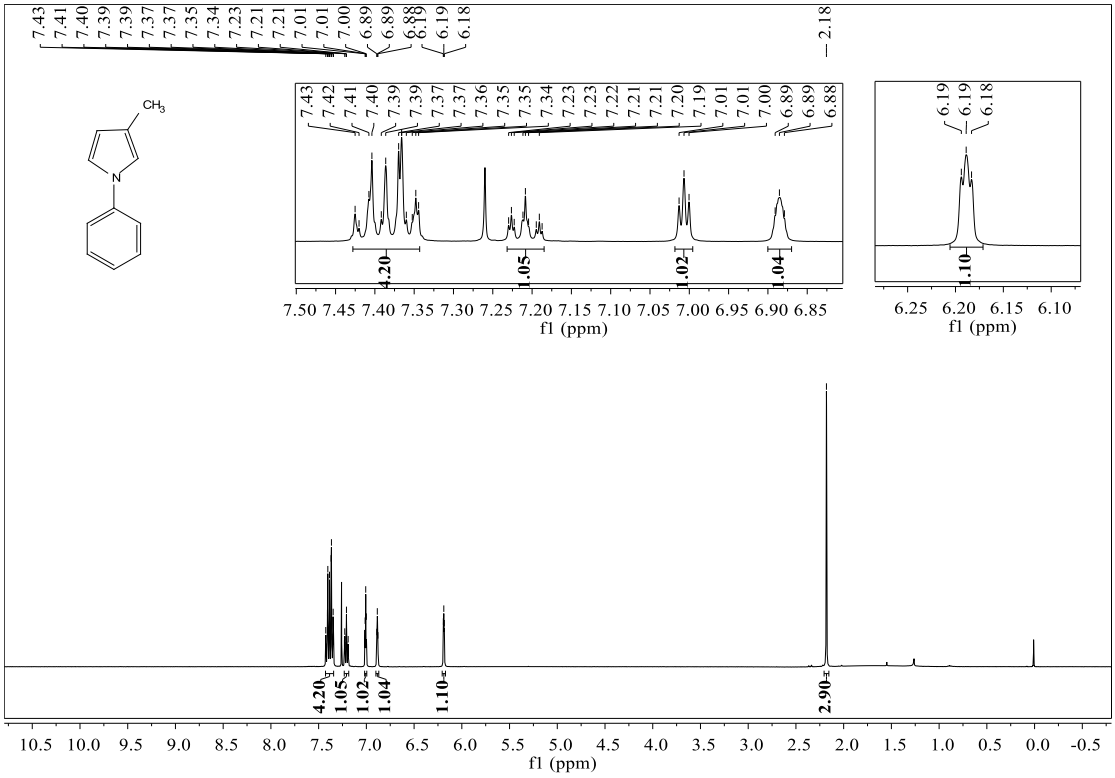
^{1}H NMR spectrum of compound 3af (400 MHz, $CDCl_3$)



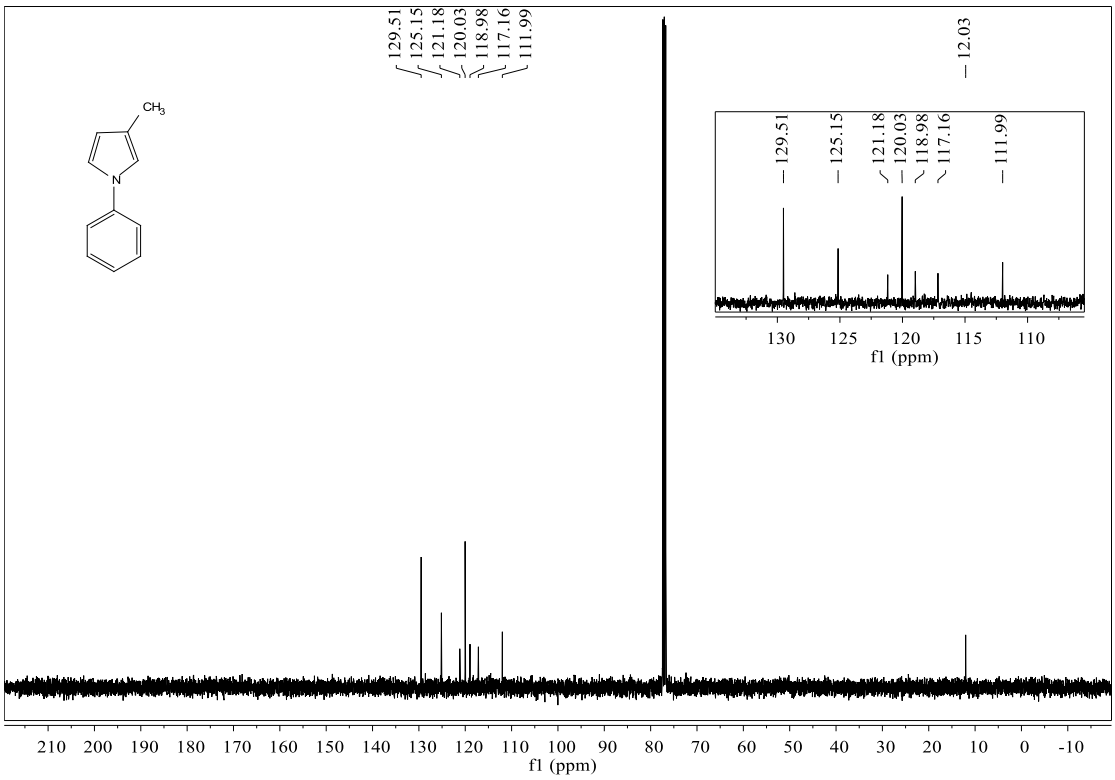
^{13}C NMR spectrum of compound 3af (101 MHz, $CDCl_3$)



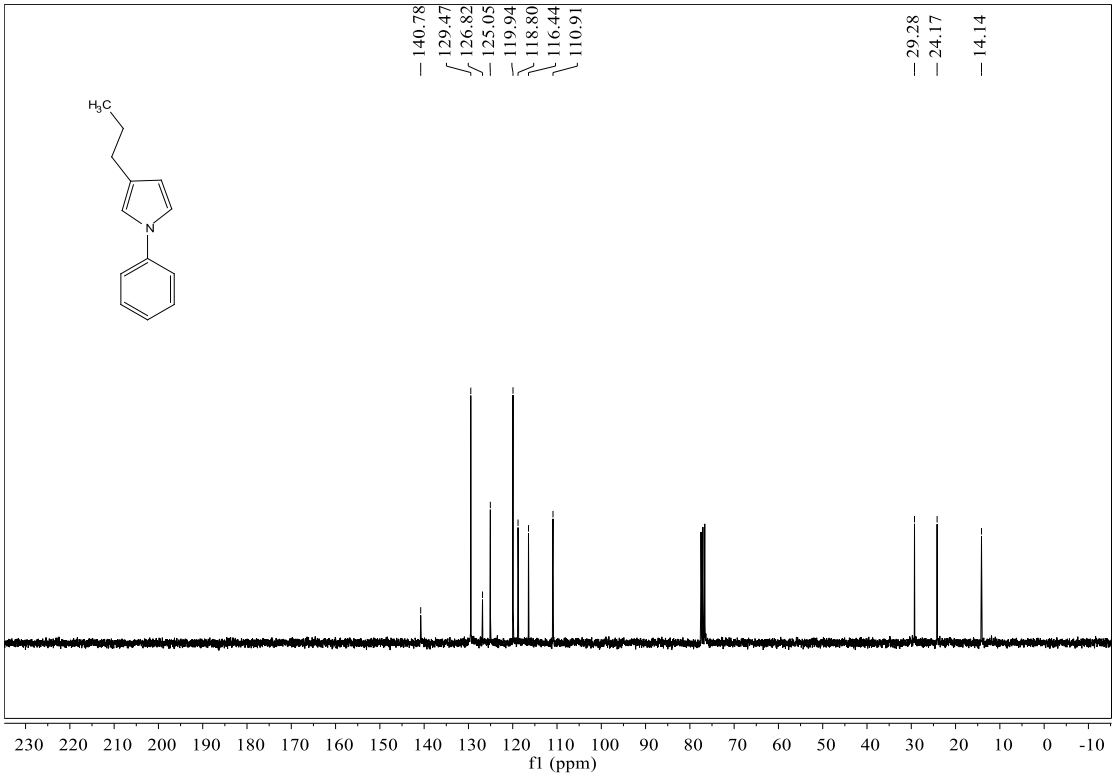
^{1}H NMR spectrum of compound 3ag (400 MHz, $CDCl_3$)



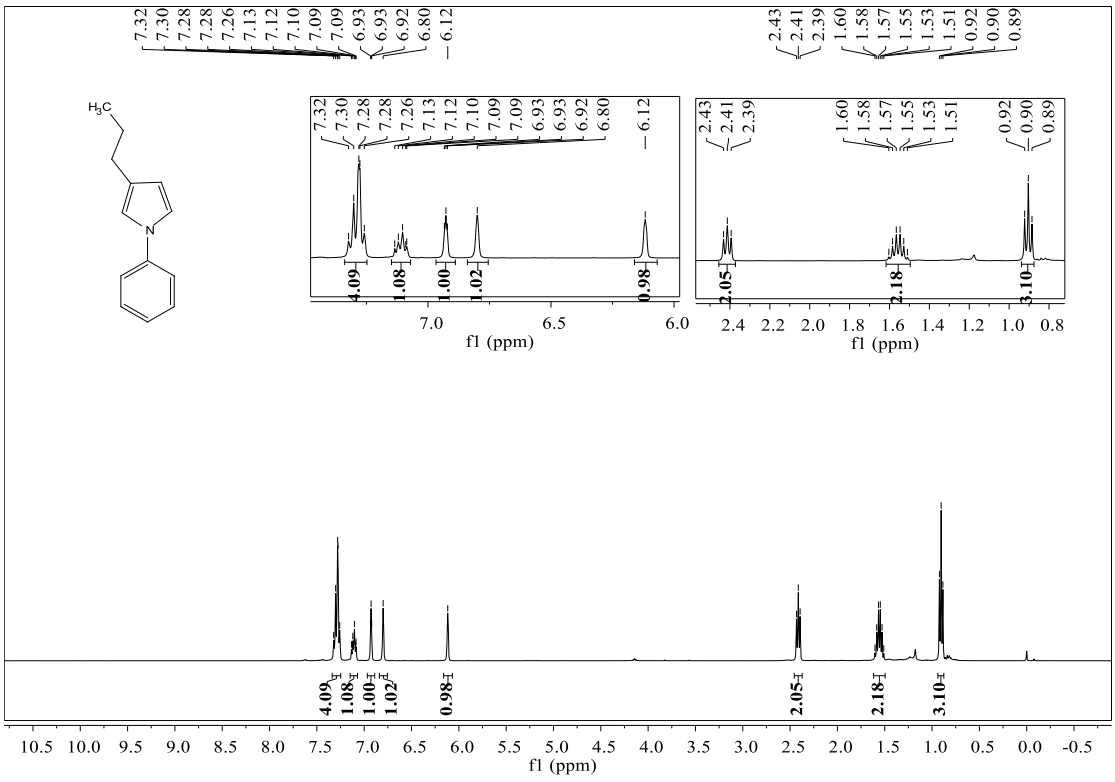
^{13}C NMR spectrum of compound 3ag (101 MHz, $CDCl_3$)



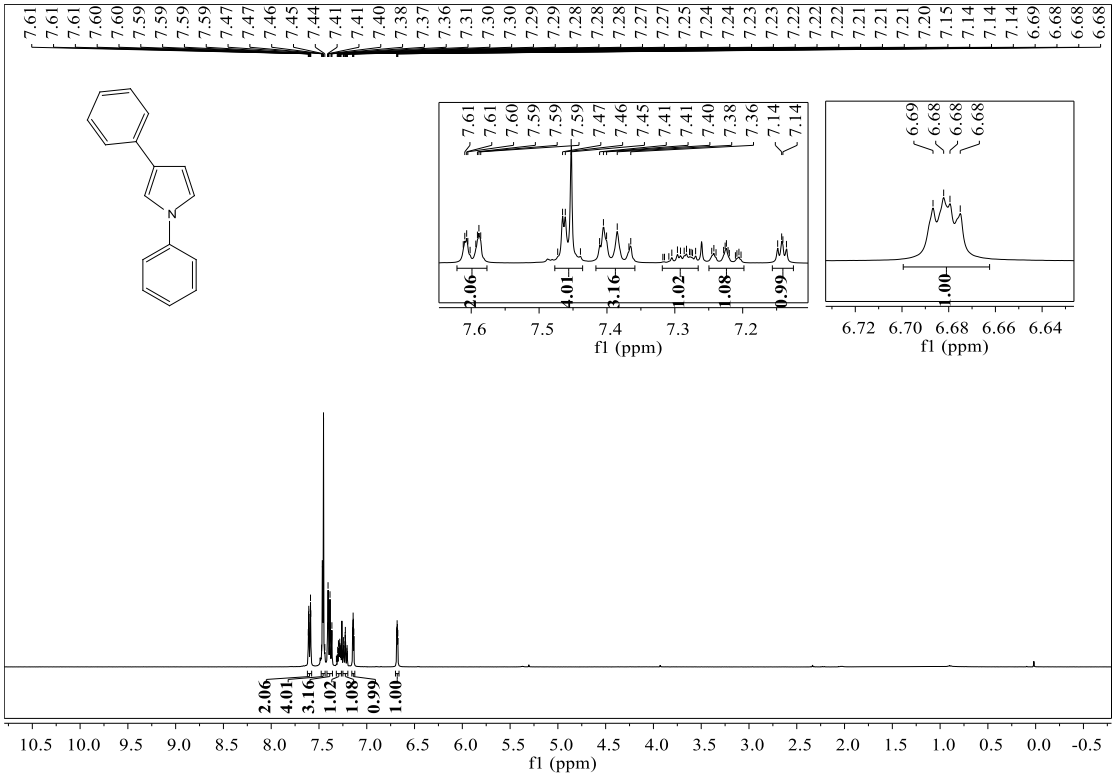
^{1}H NMR spectrum of compound 3ah (400 MHz, $CDCl_3$)



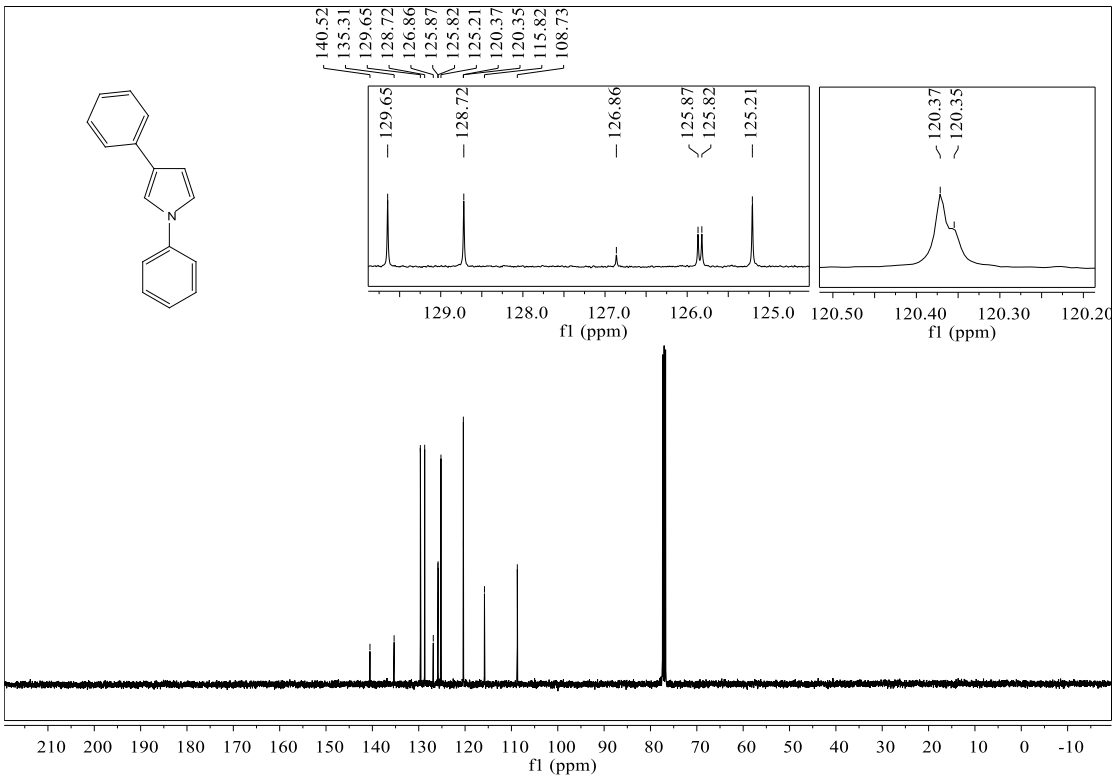
^{13}C NMR spectrum of compound 3ah (76 MHz, $CDCl_3$)



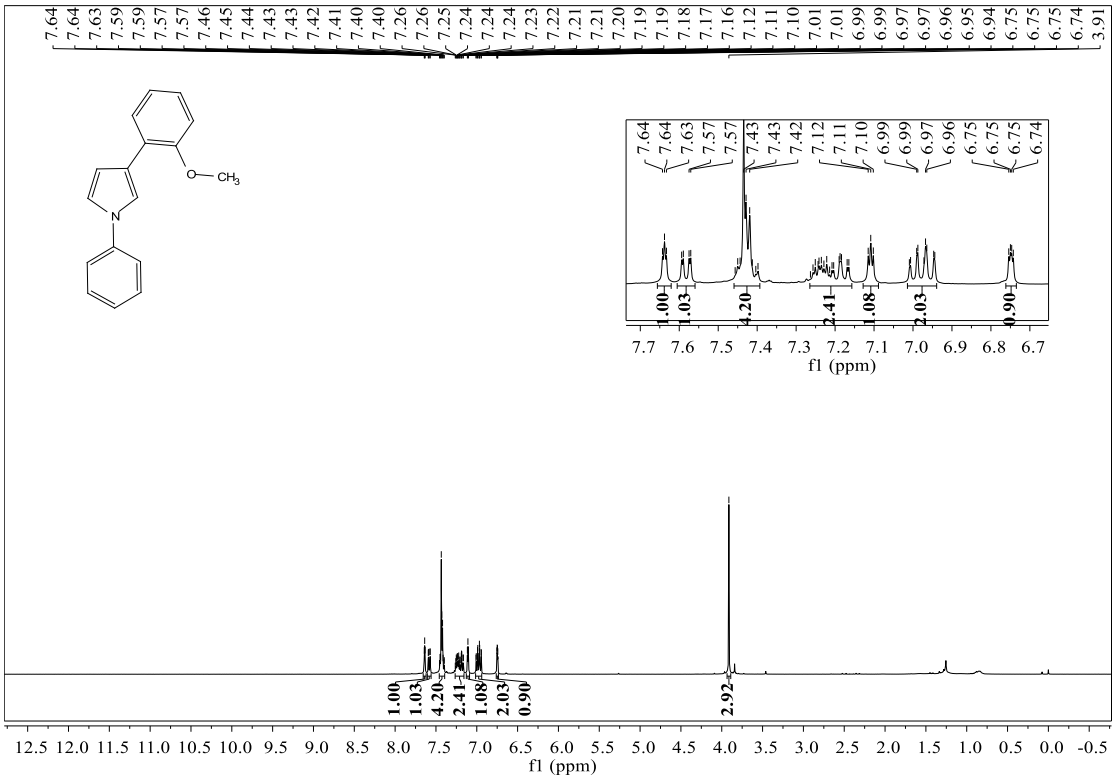
^{1}H NMR spectrum of compound 3ai (400 MHz, $CDCl_3$)



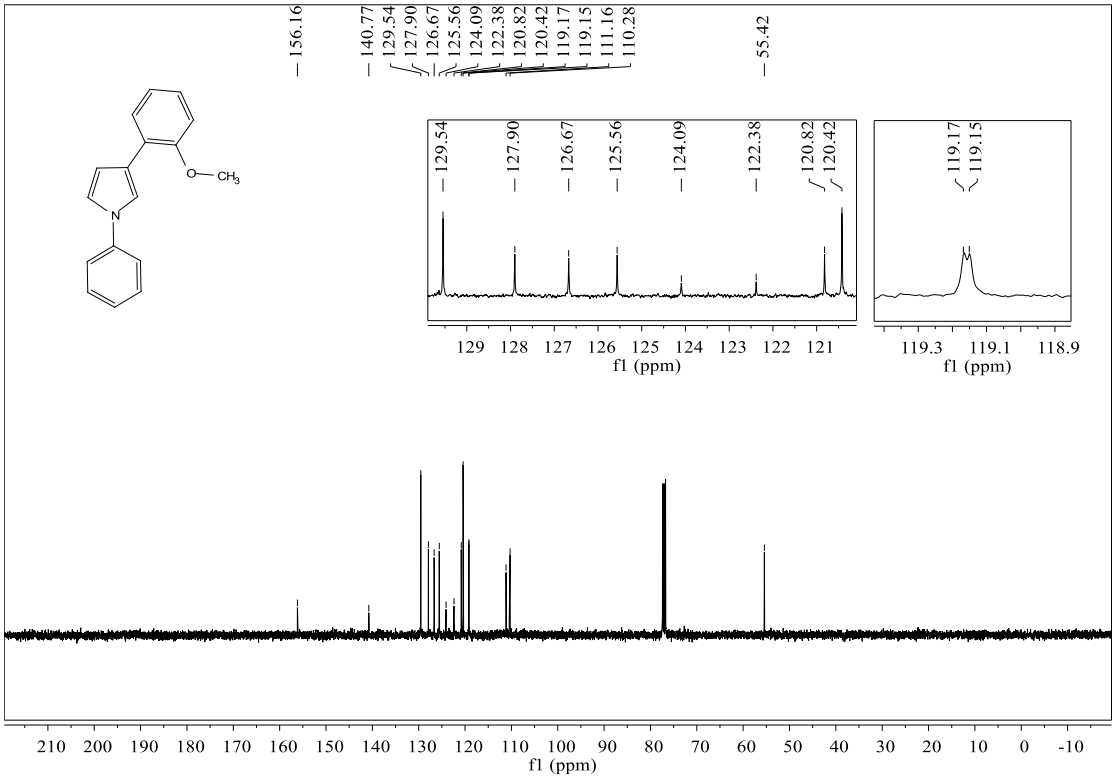
^{13}C NMR spectrum of compound 3ai (101 MHz, $CDCl_3$)



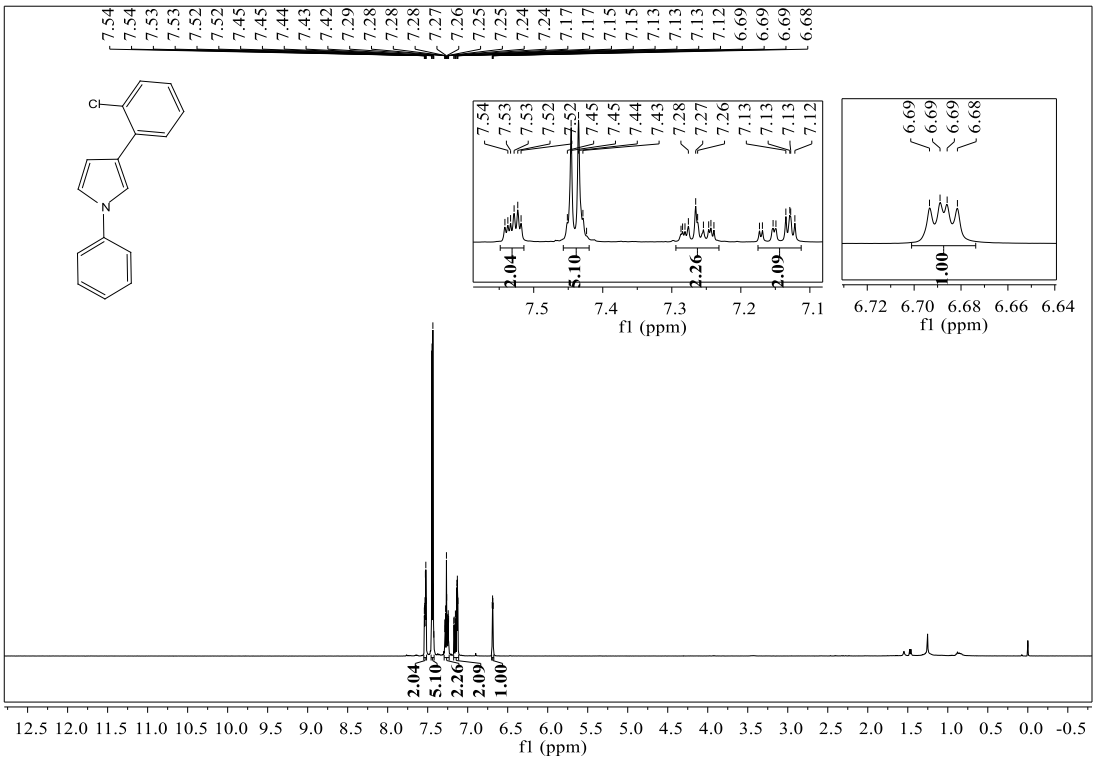
^{1}H NMR spectrum of compound 3aj (400 MHz, $CDCl_3$)



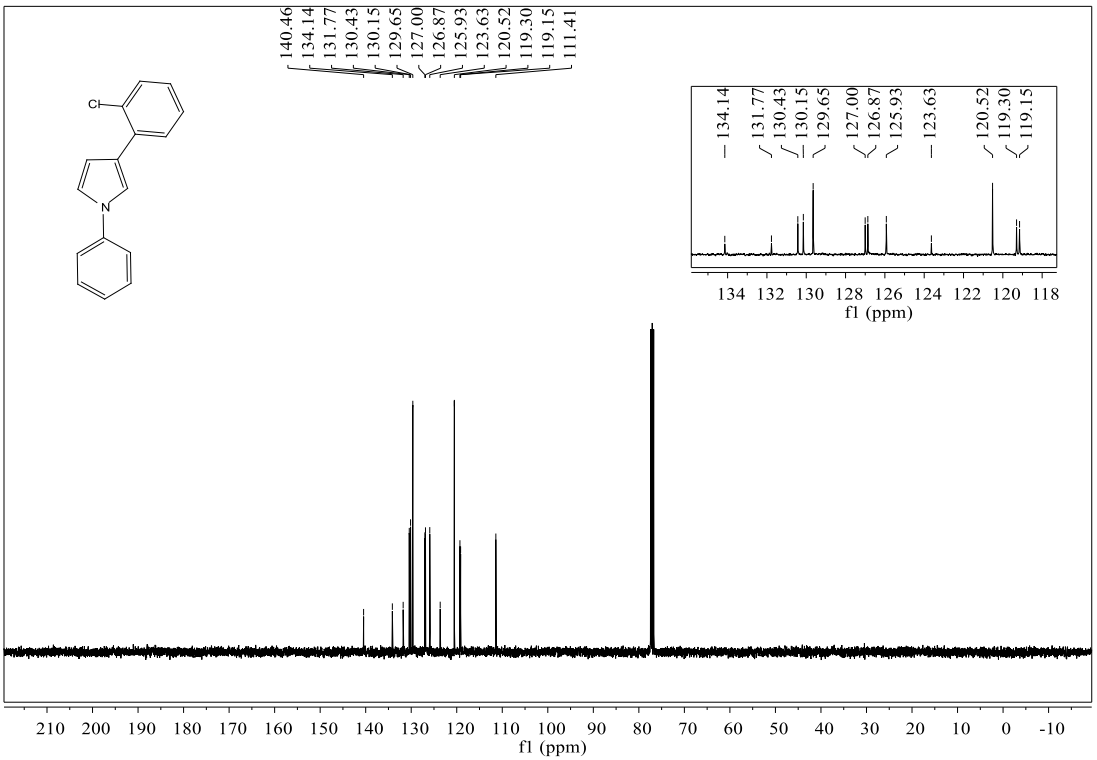
^{13}C NMR spectrum of compound 3aj (101 MHz, $CDCl_3$)



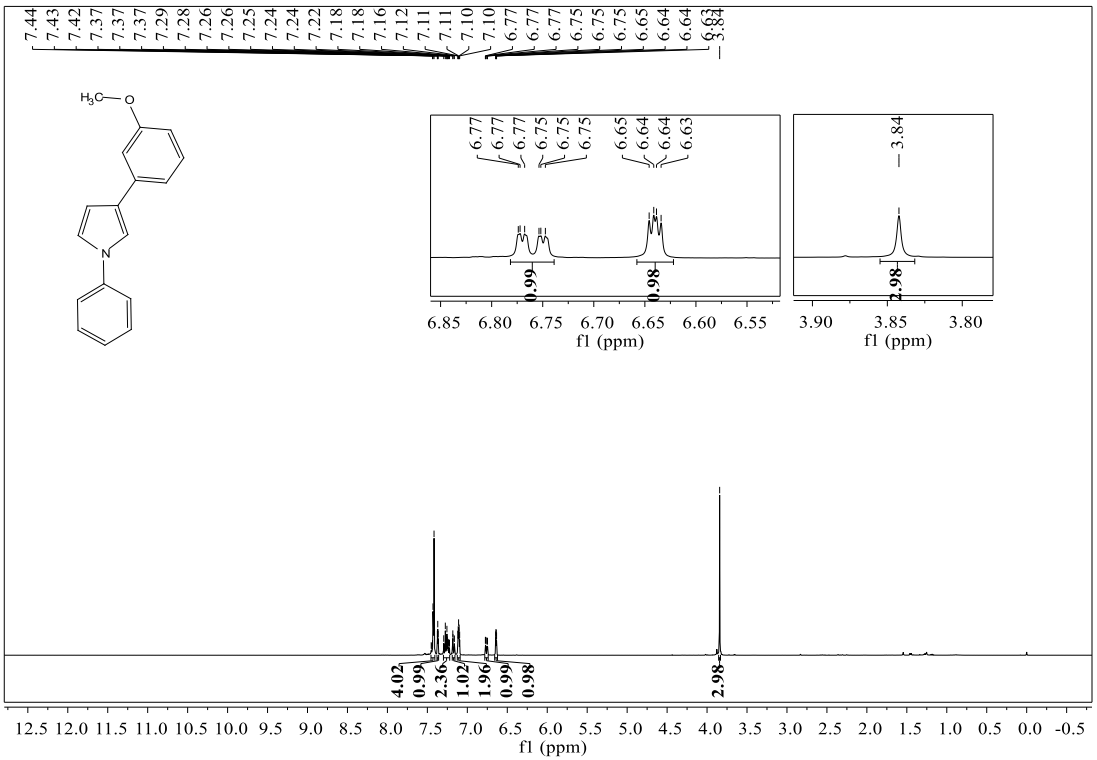
^{1}H NMR spectrum of compound 3ak (400 MHz, $CDCl_3$)



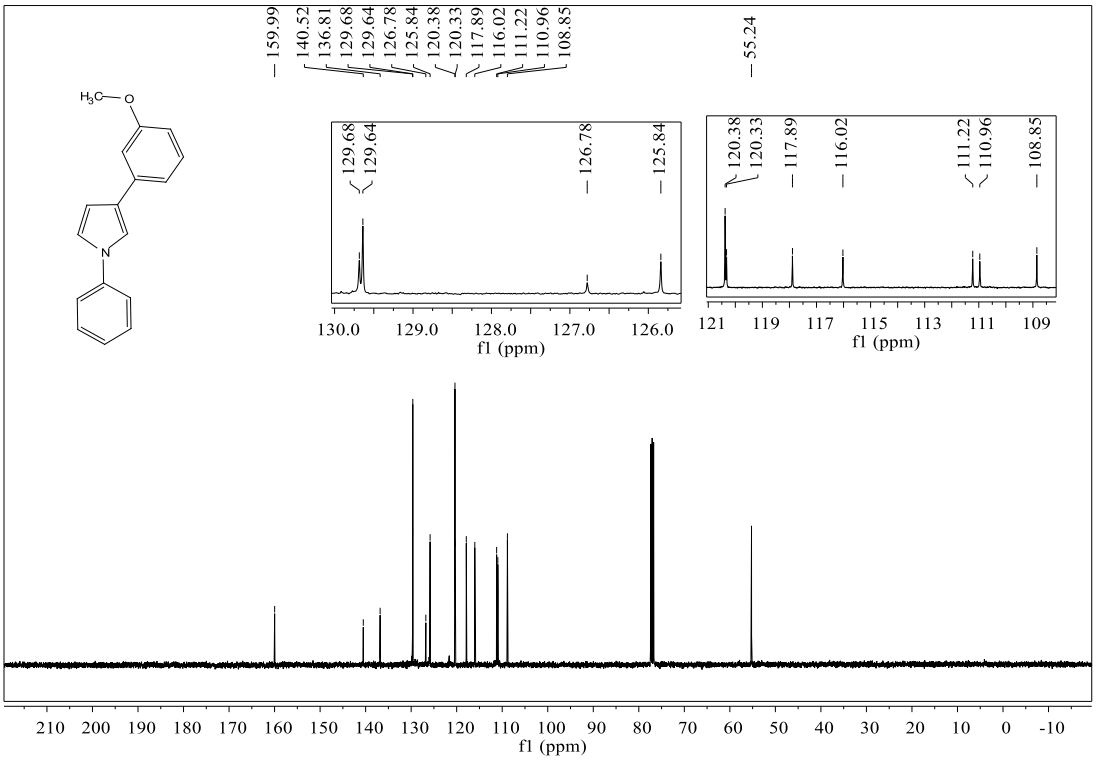
^{13}C NMR spectrum of compound 3ak (101 MHz, $CDCl_3$)



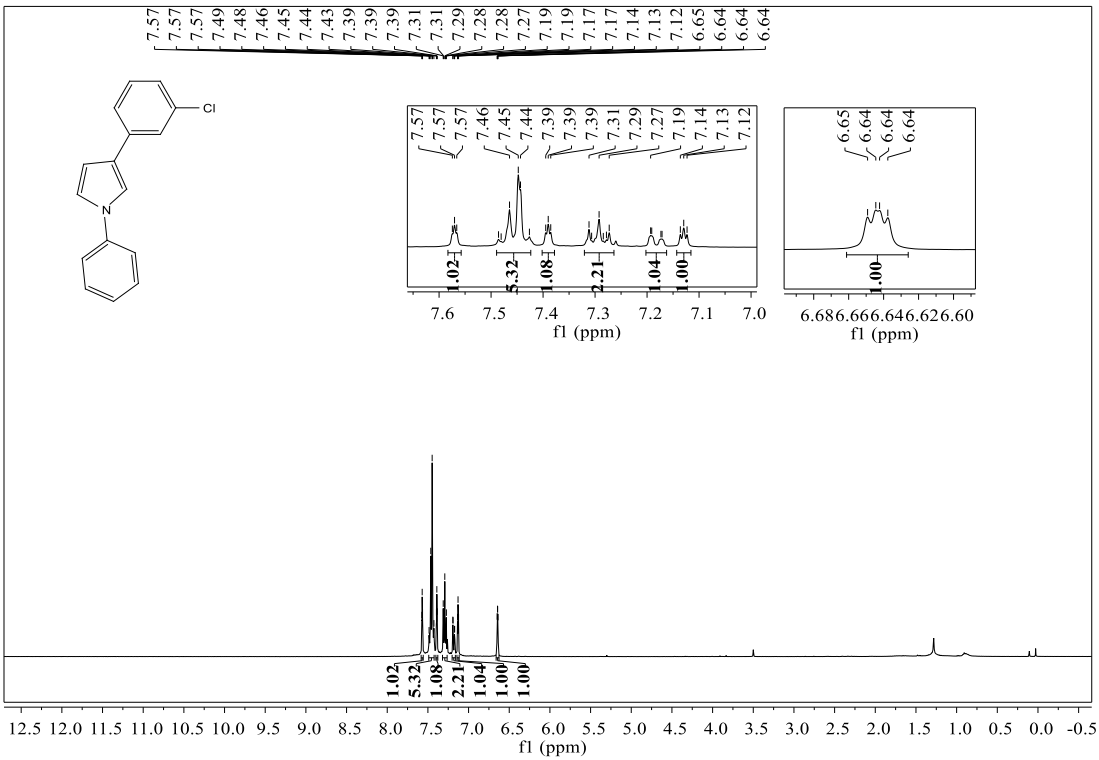
^{1}H NMR spectrum of compound 3al (400 MHz, $CDCl_3$)



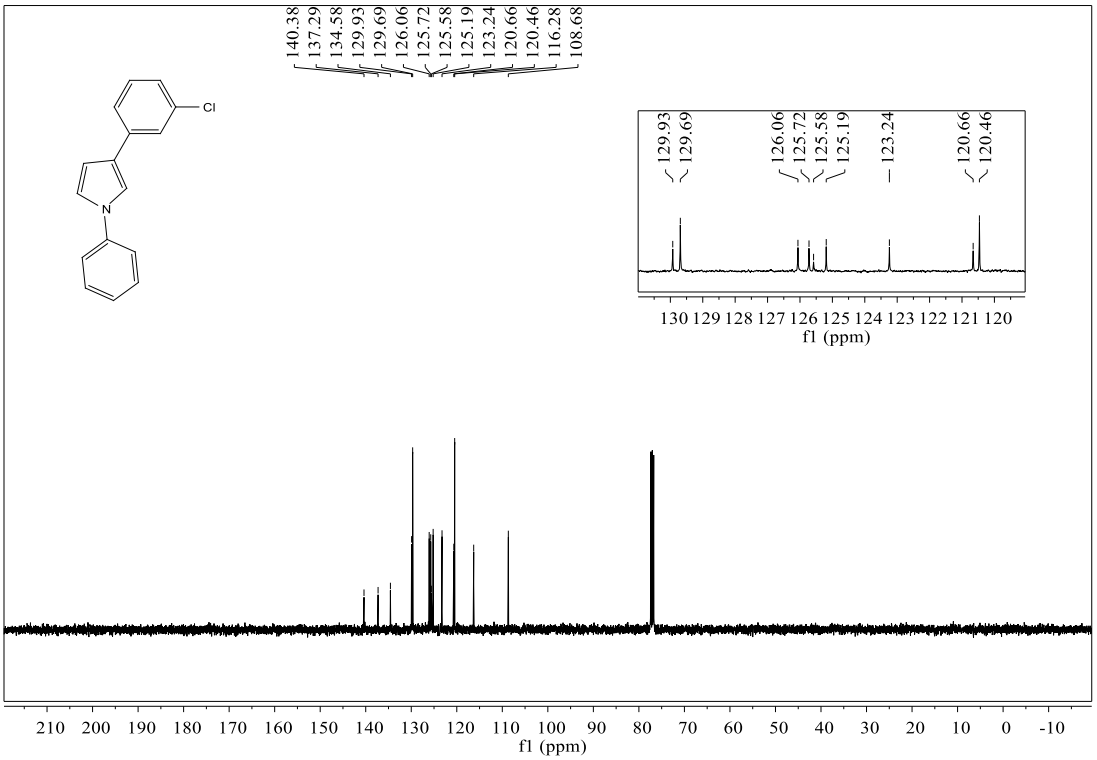
^{13}C NMR spectrum of compound 3al (101 MHz, $CDCl_3$)



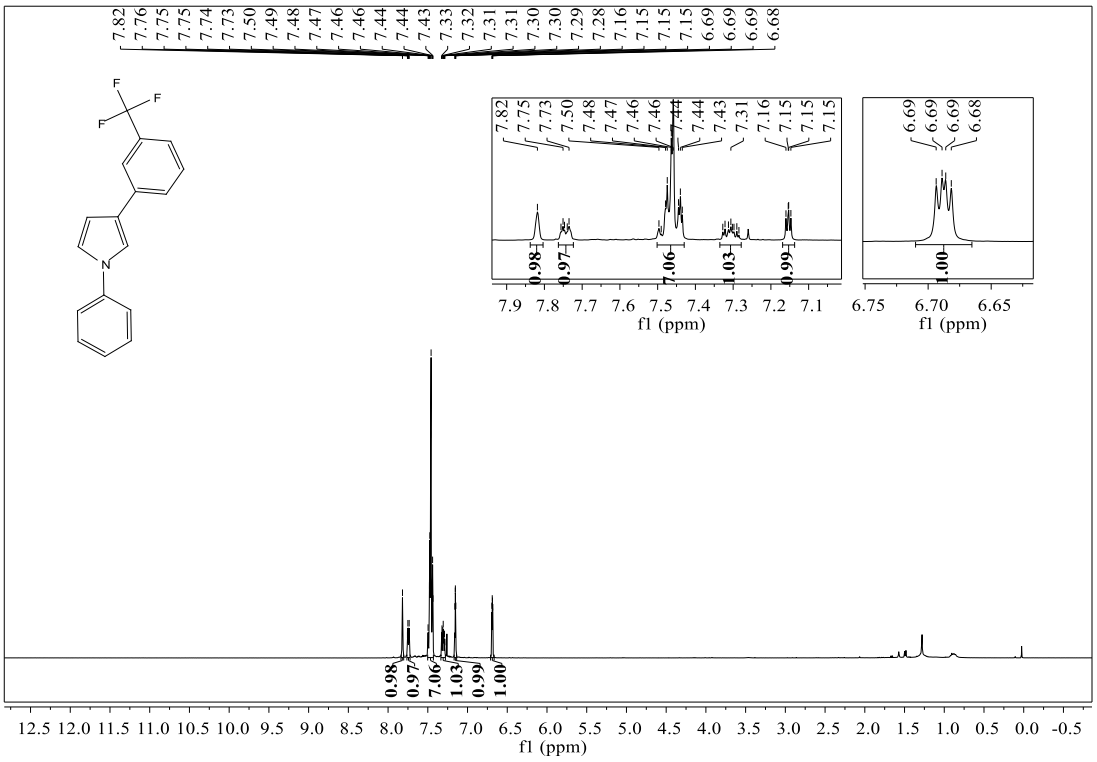
^{1}H NMR spectrum of compound 3am (400 MHz, $CDCl_3$)



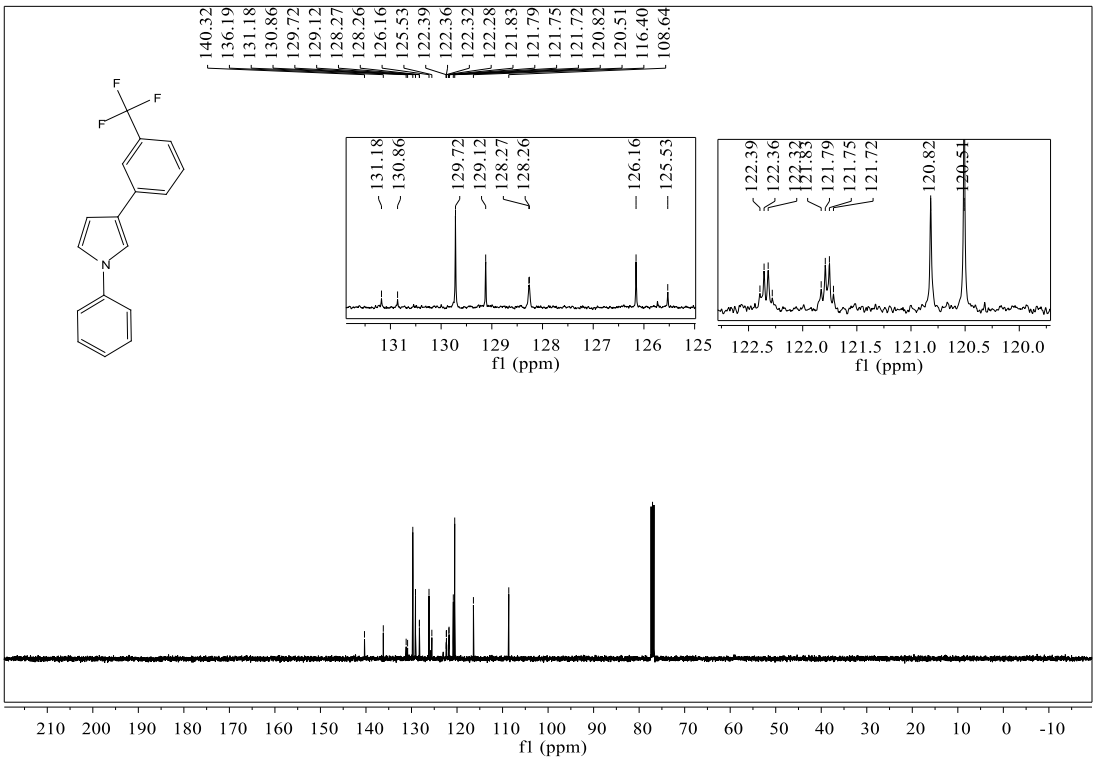
^{13}C NMR spectrum of compound 3am (101 MHz, $CDCl_3$)



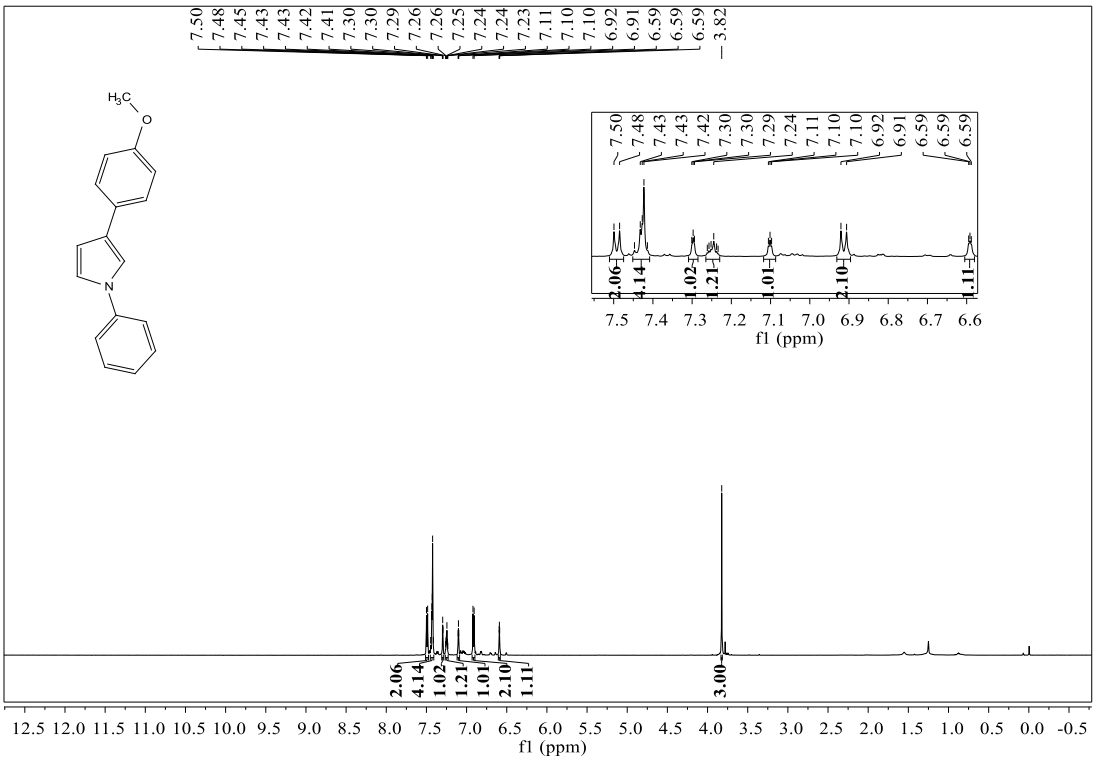
^{1}H NMR spectrum of compound 3an (400 MHz, $CDCl_3$)



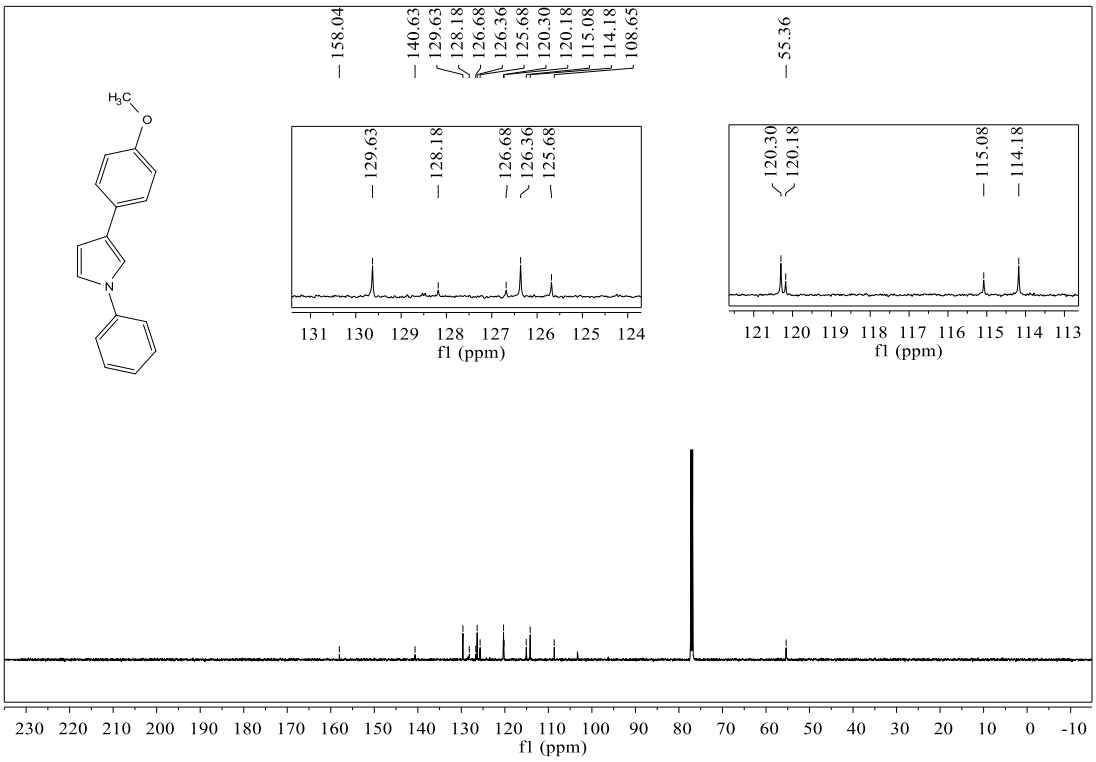
^{13}C NMR spectrum of compound 3an (101 MHz, $CDCl_3$)



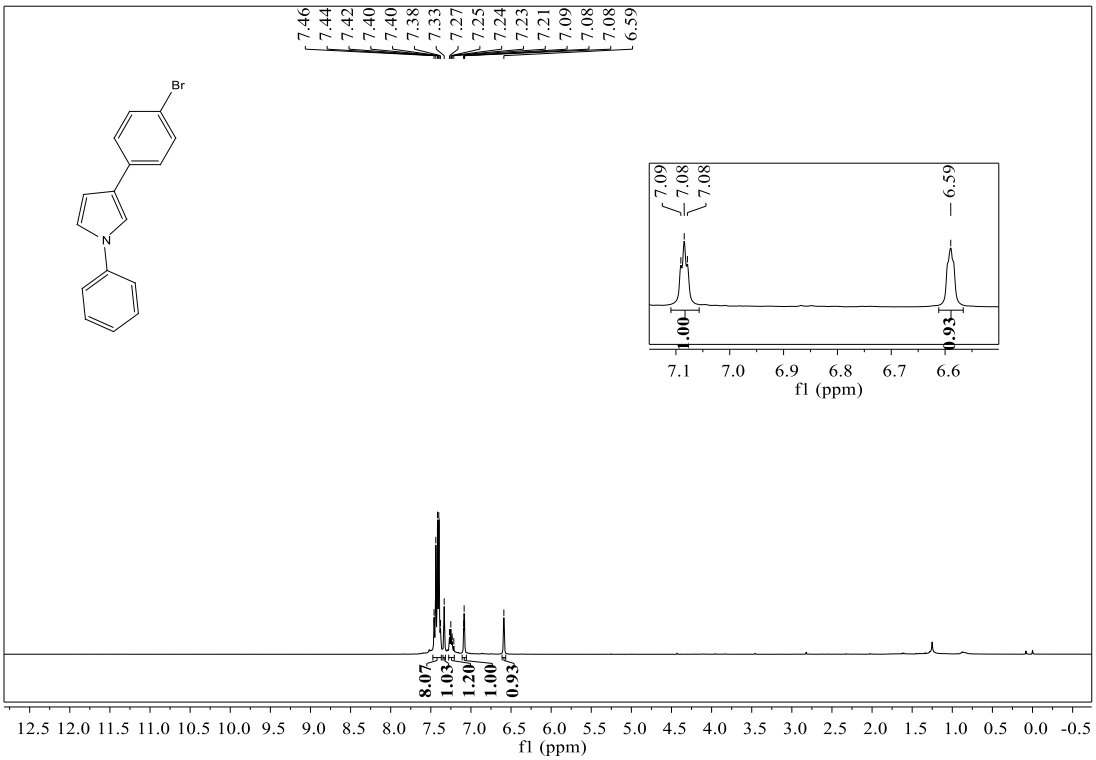
^{1}H NMR spectrum of compound 3ao (600 MHz, $CDCl_3$)



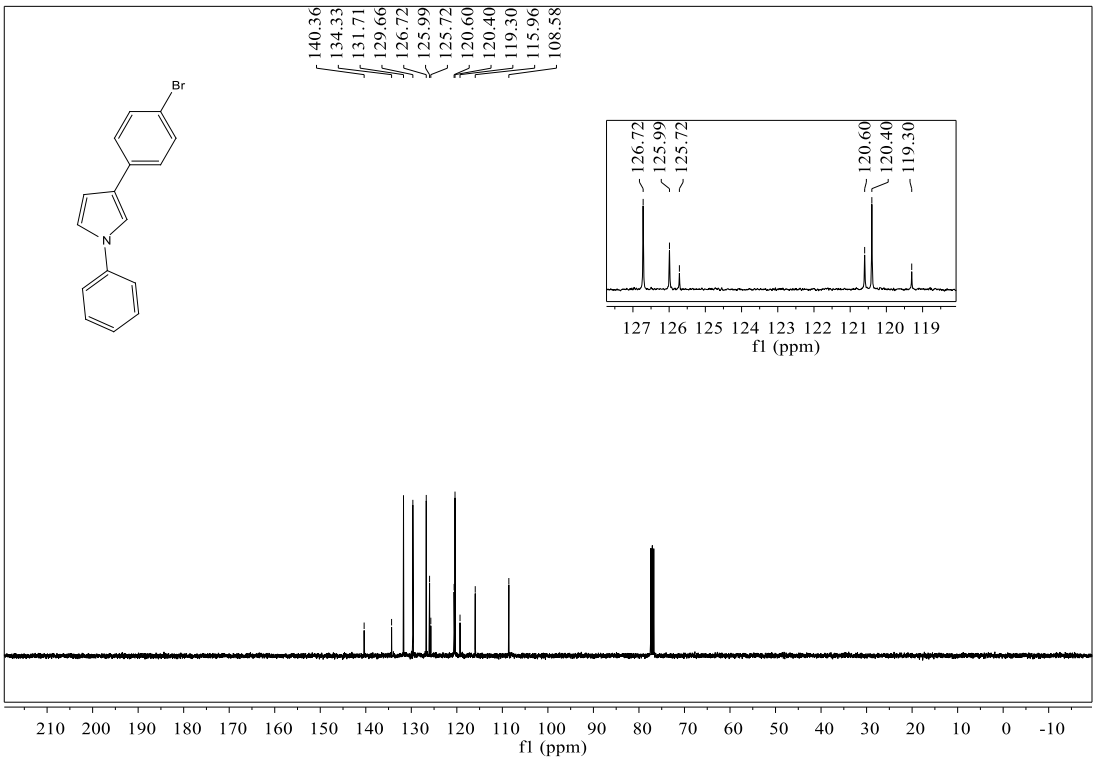
^{13}C NMR spectrum of compound 3ao (151 MHz, $CDCl_3$)



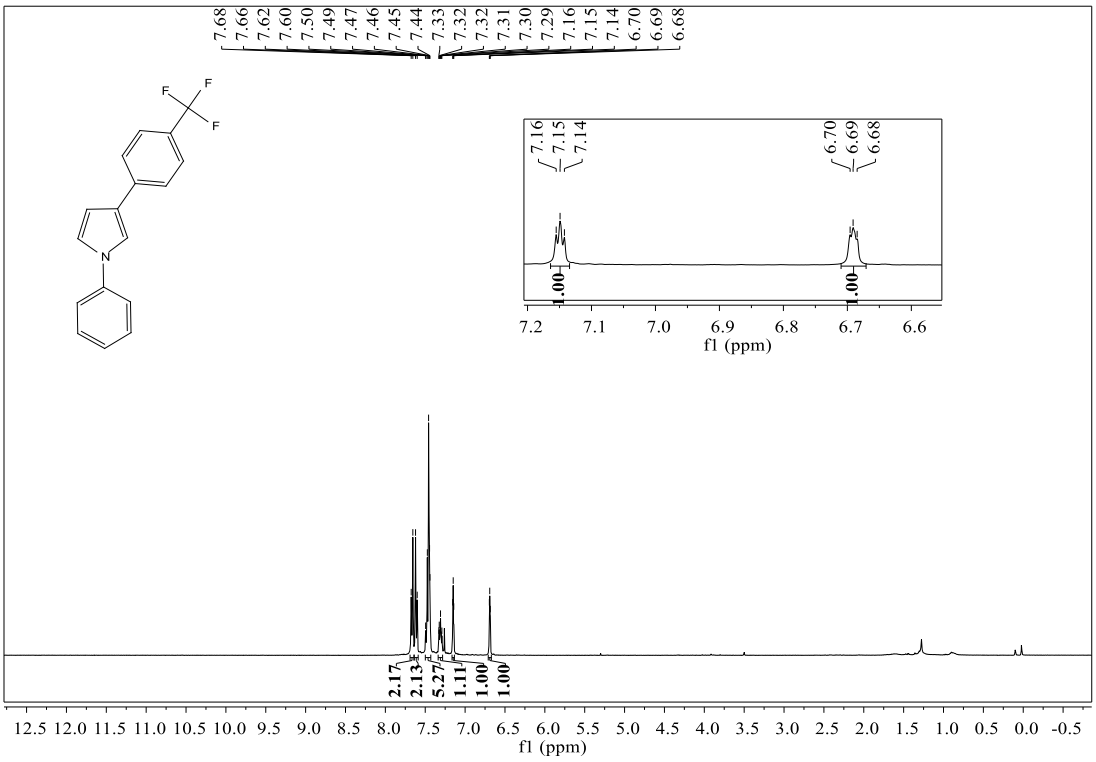
^{1}H NMR spectrum of compound 3ap (400 MHz, $CDCl_3$)



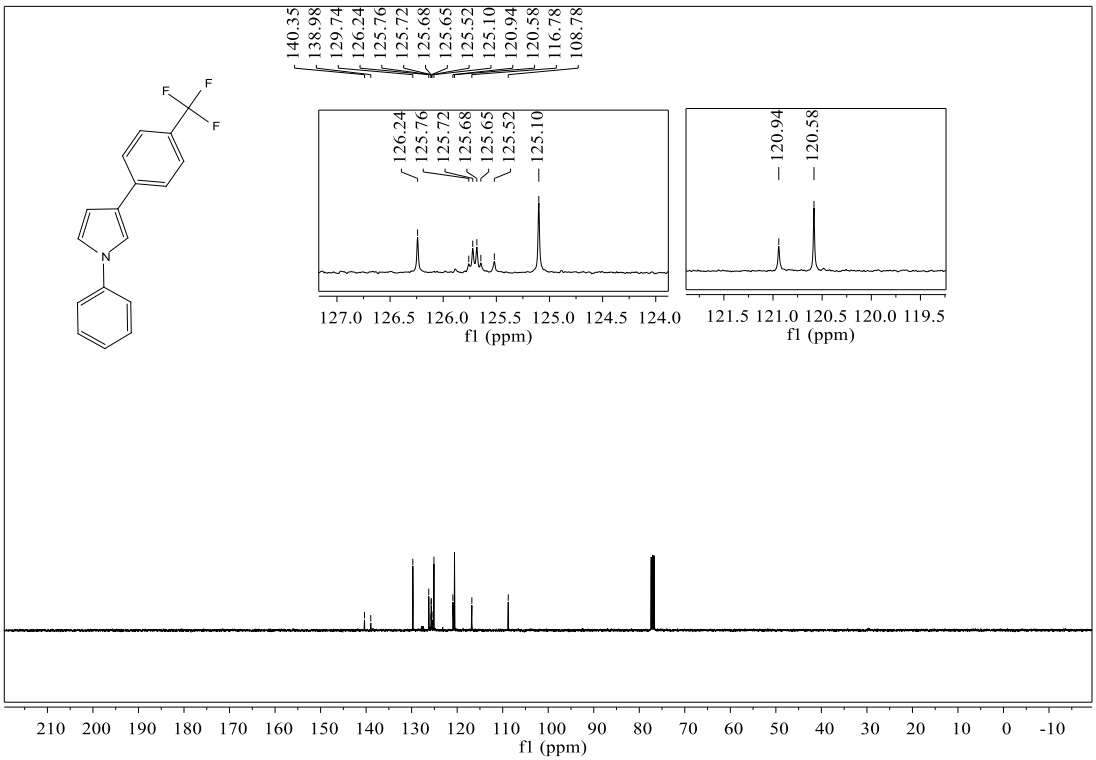
^{13}C NMR spectrum of compound 3ap (101 MHz, $CDCl_3$)



^{1}H NMR spectrum of compound 3aq (400 MHz, $CDCl_3$)



^{13}C NMR spectrum of compound 3aq (101 MHz, $CDCl_3$)



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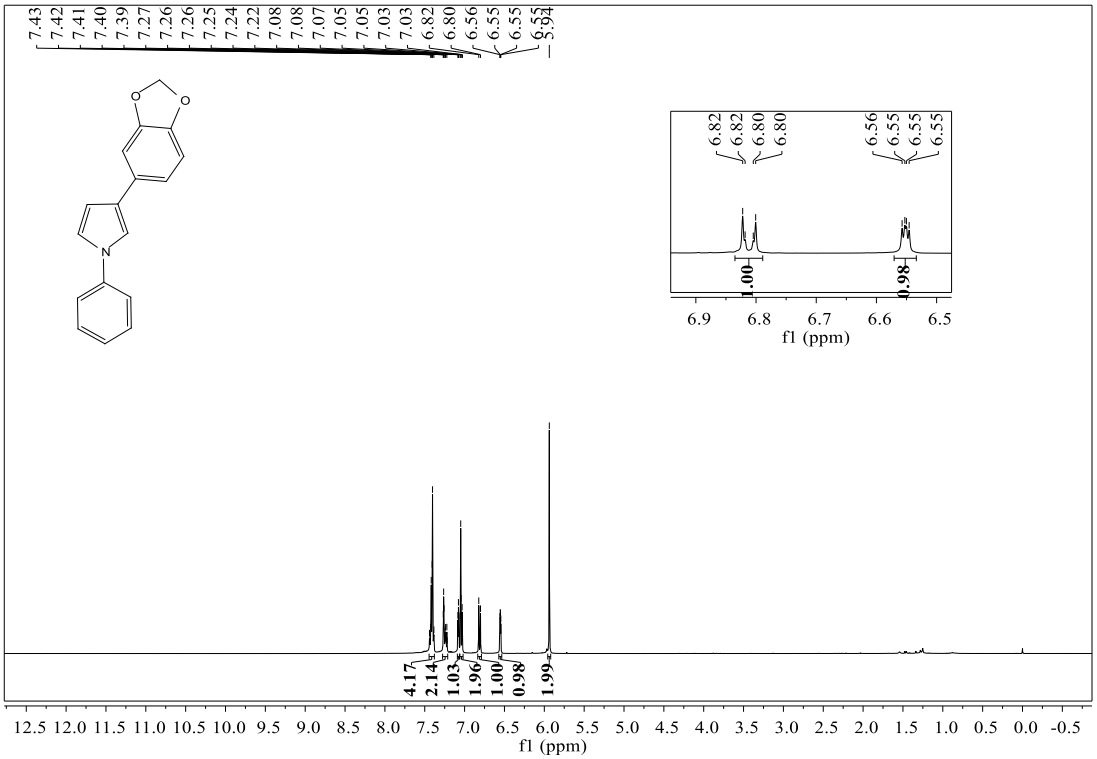
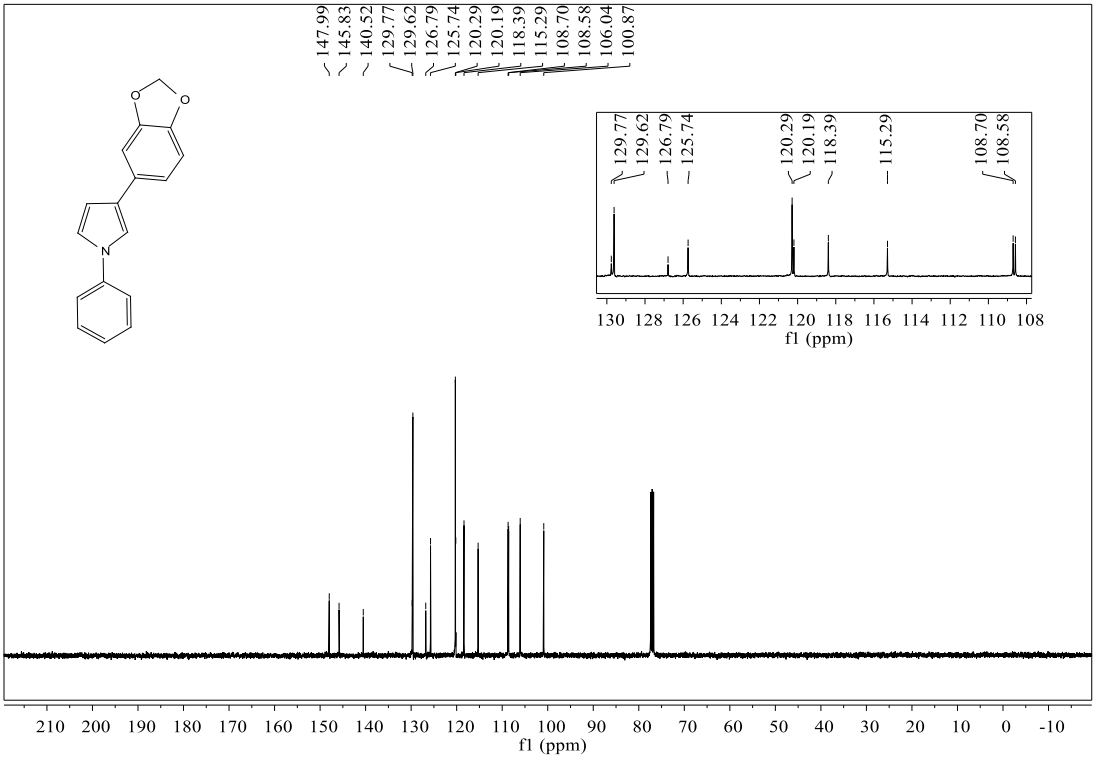


^{13}C NMR spectrum of compound 3ar (101 MHz, $CDCl_3$)



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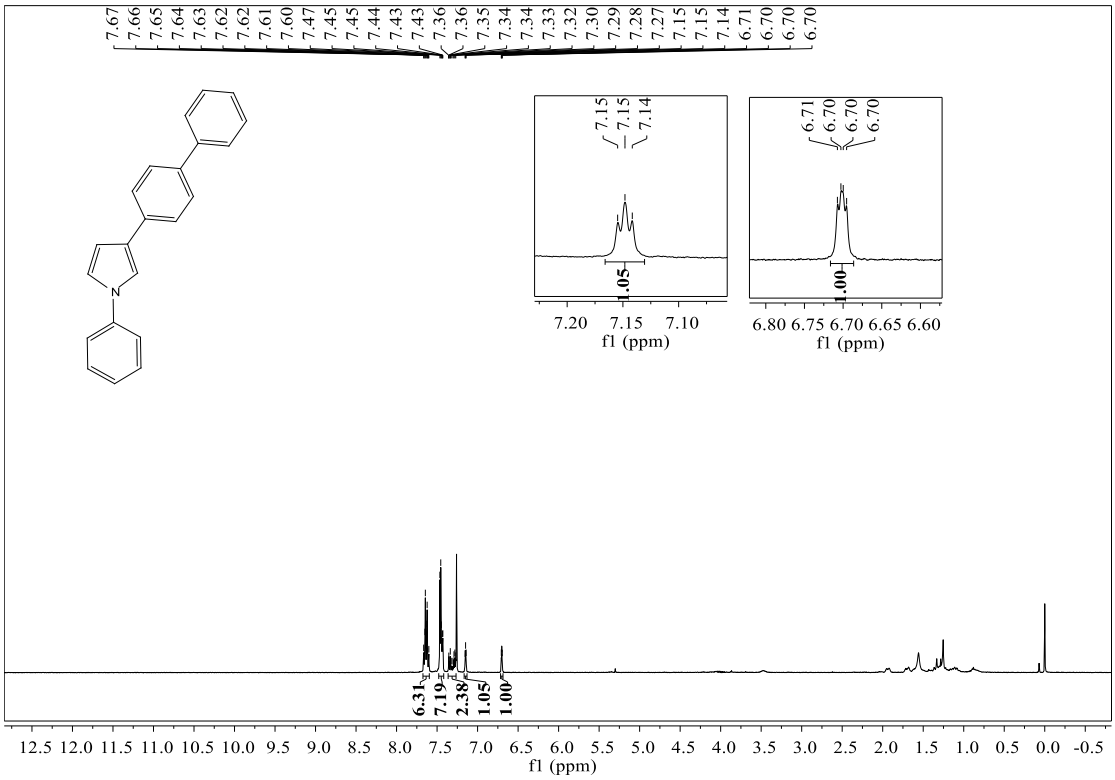

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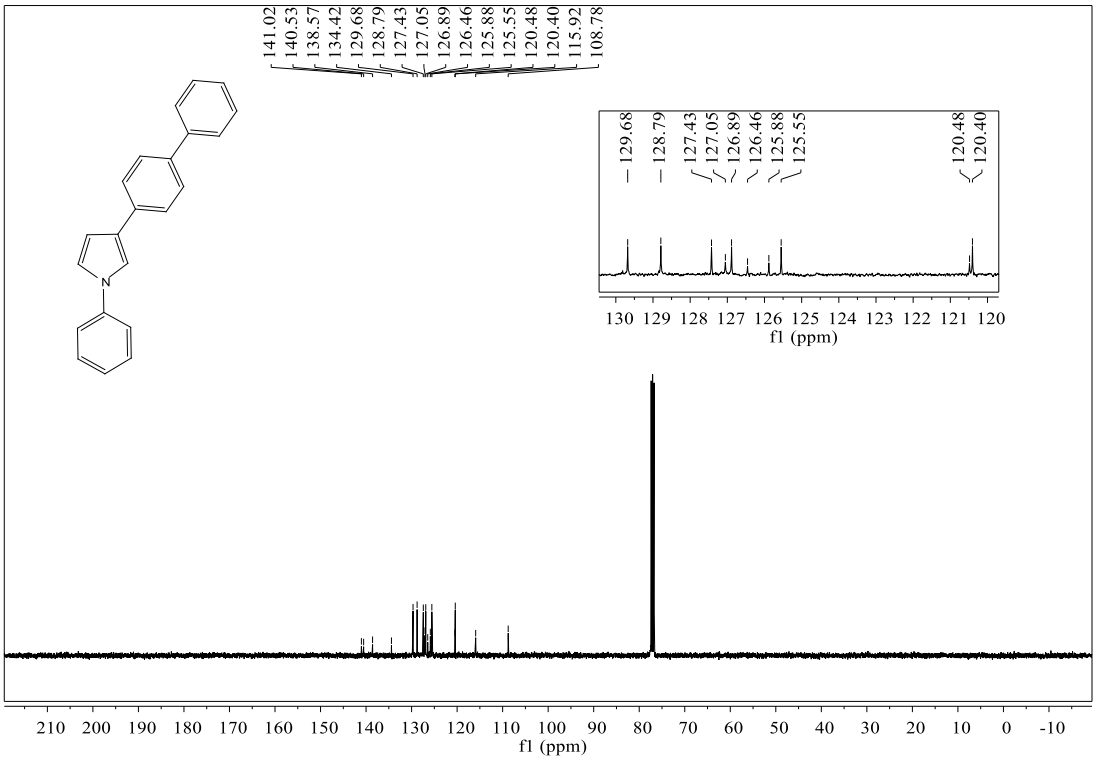

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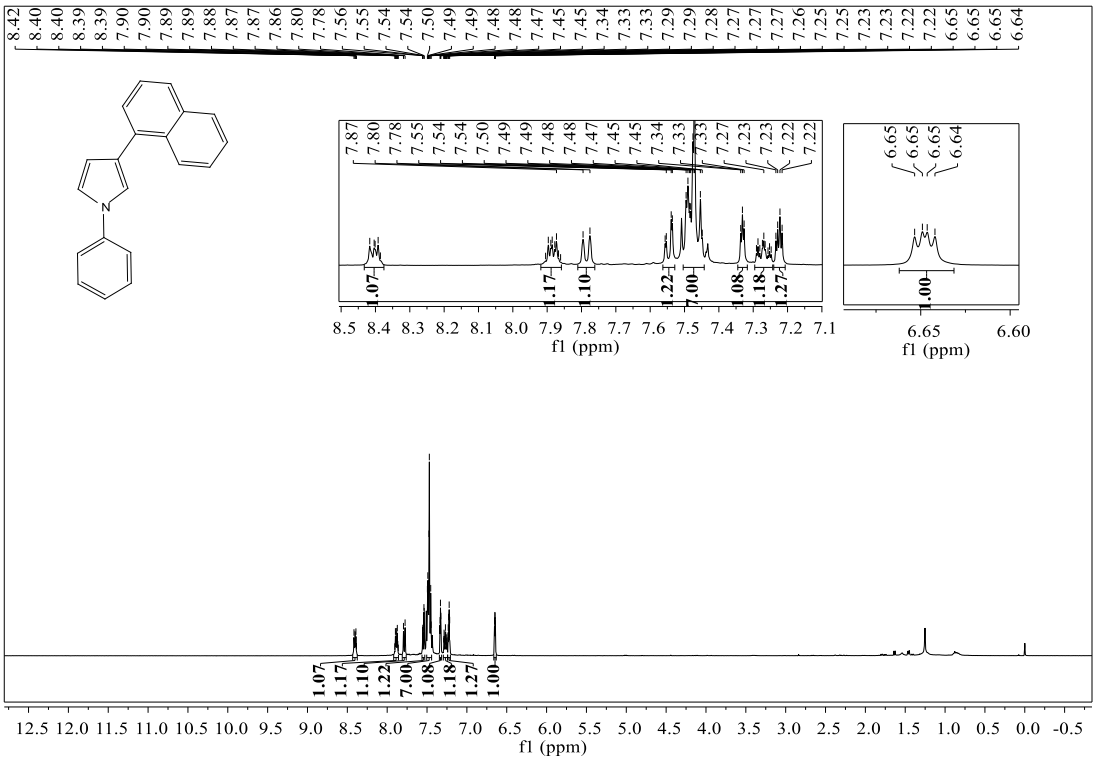
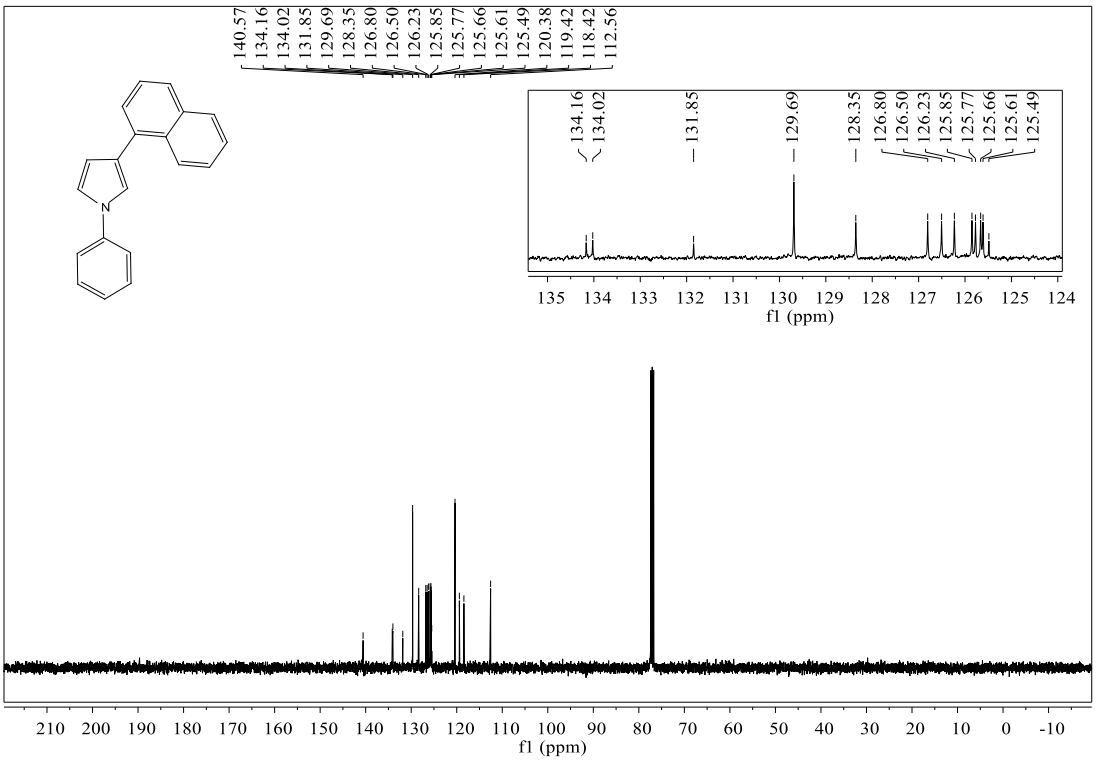
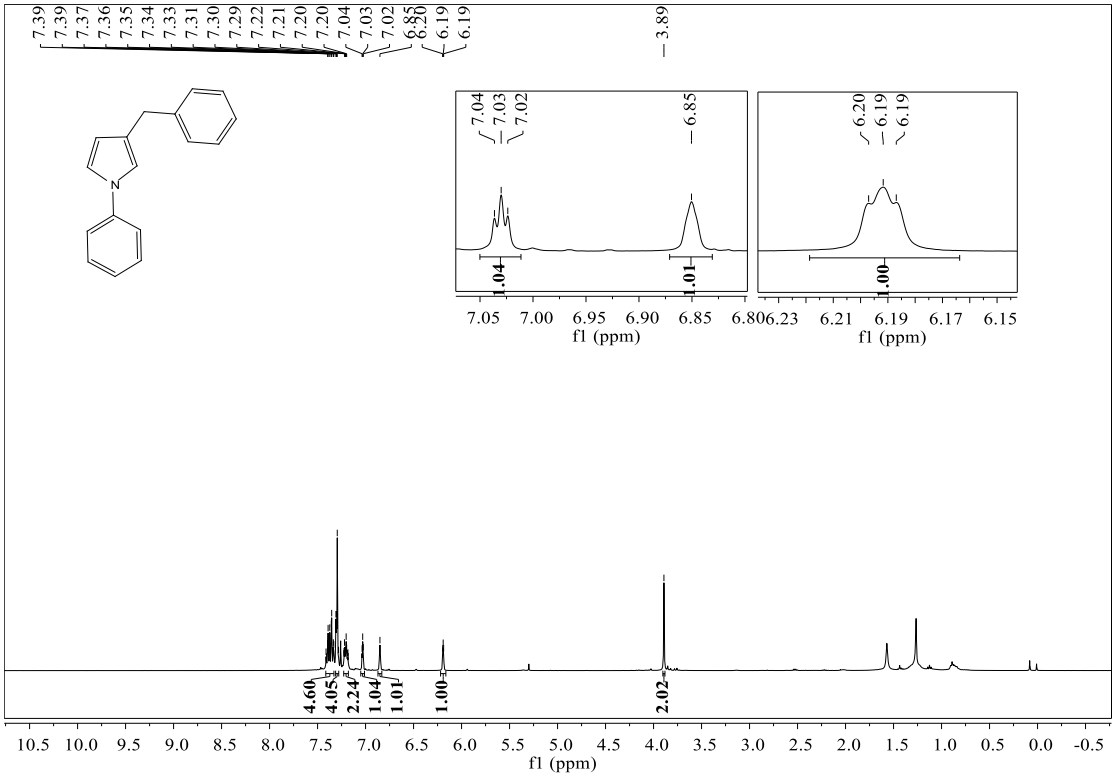


^{13}C NMR spectrum of compound 3at (101 MHz, $CDCl_3$)



^{1}H NMR spectrum of compound 3au (400 MHz, $CDCl_3$)



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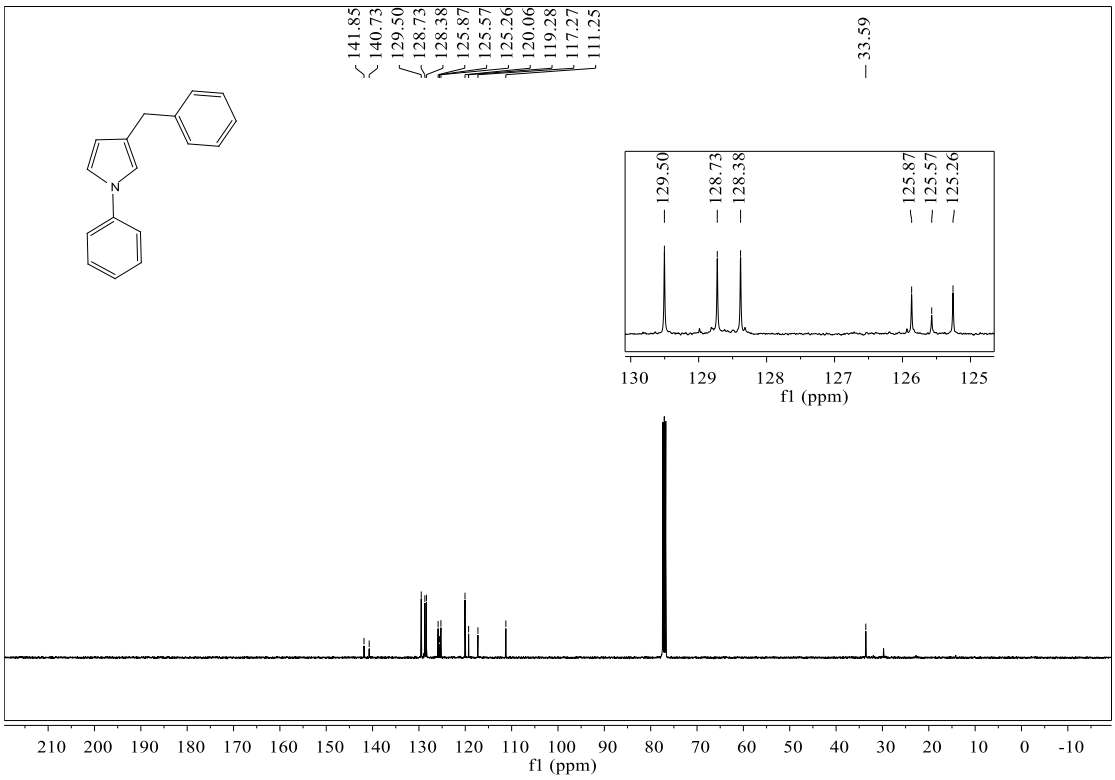

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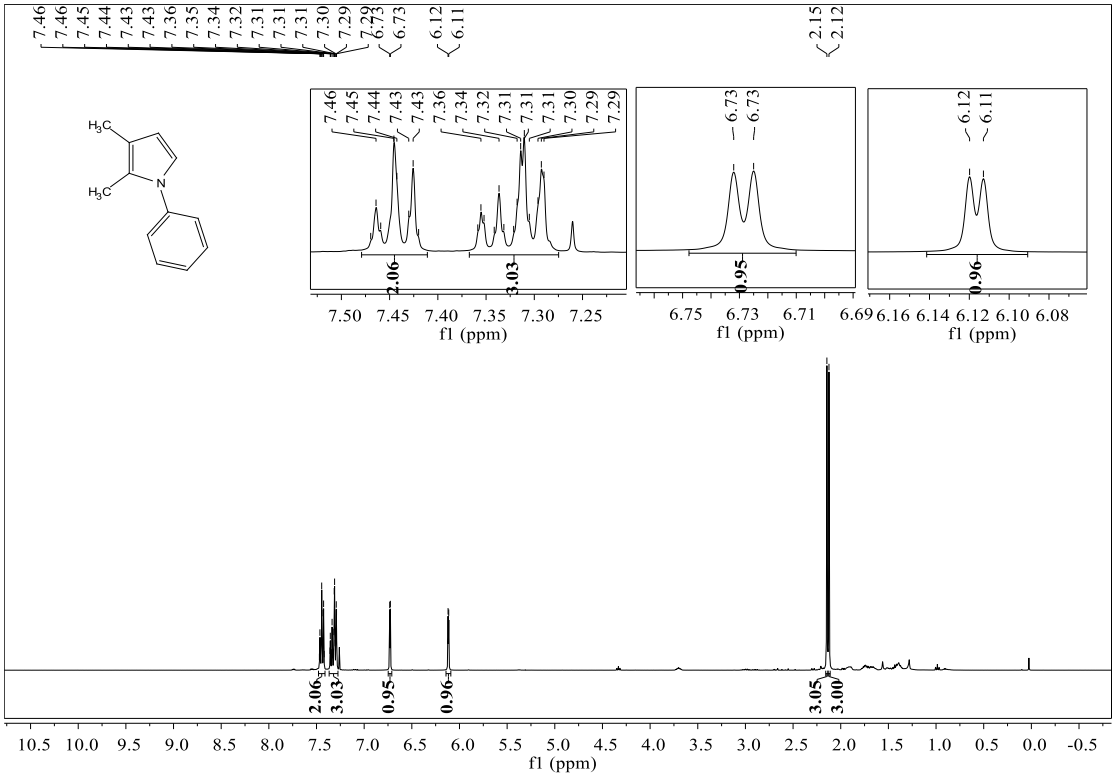

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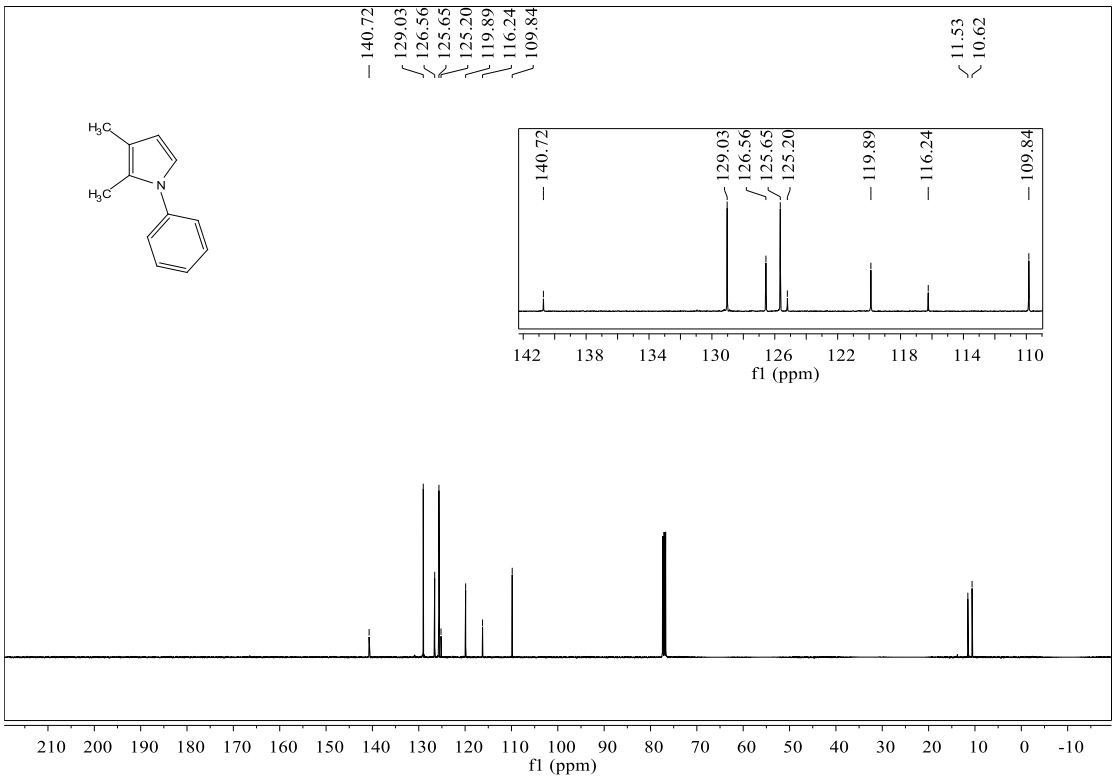
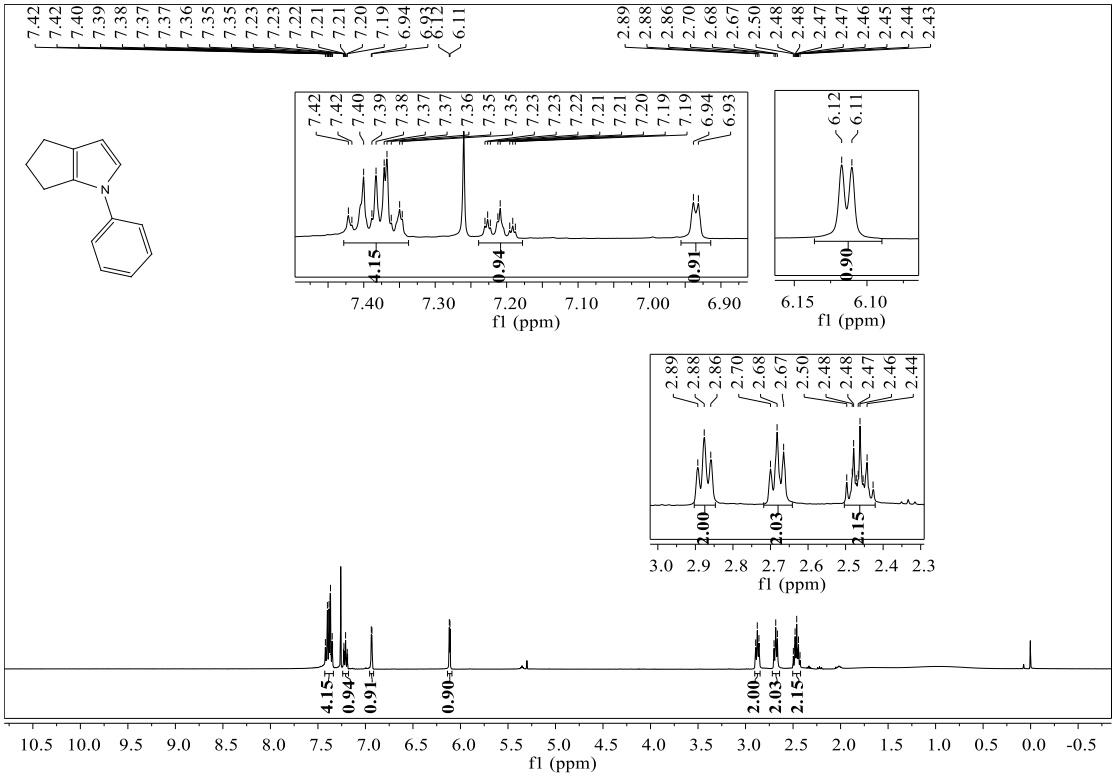
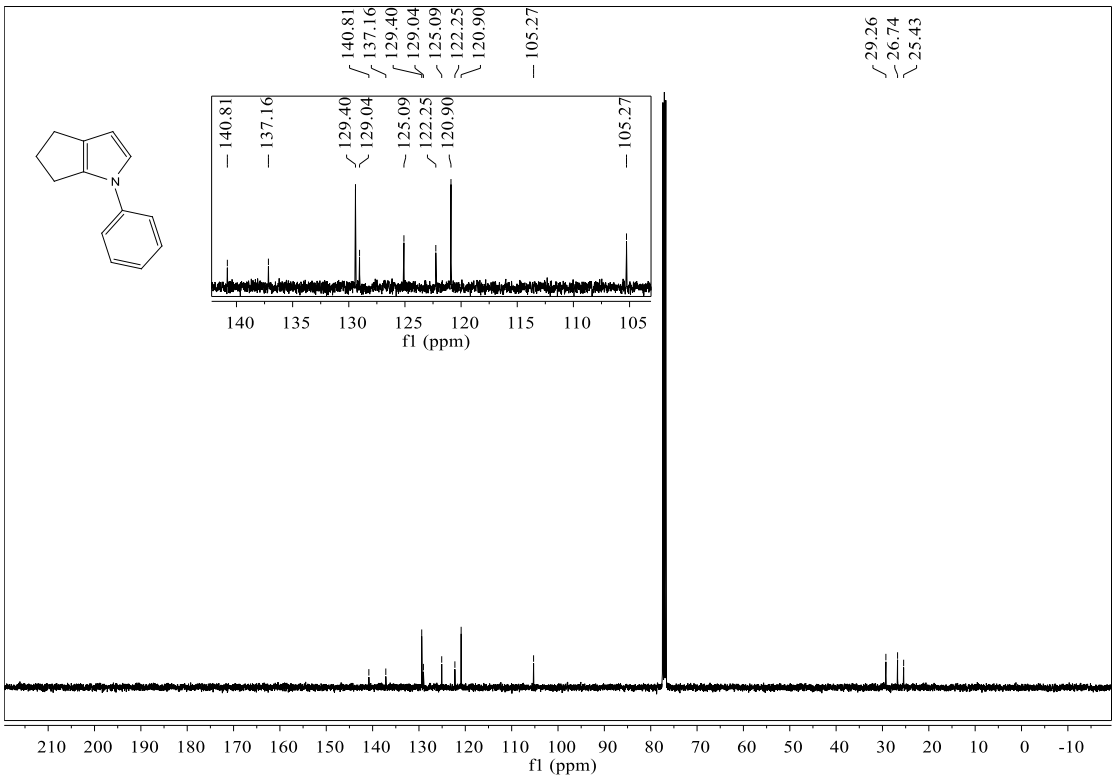


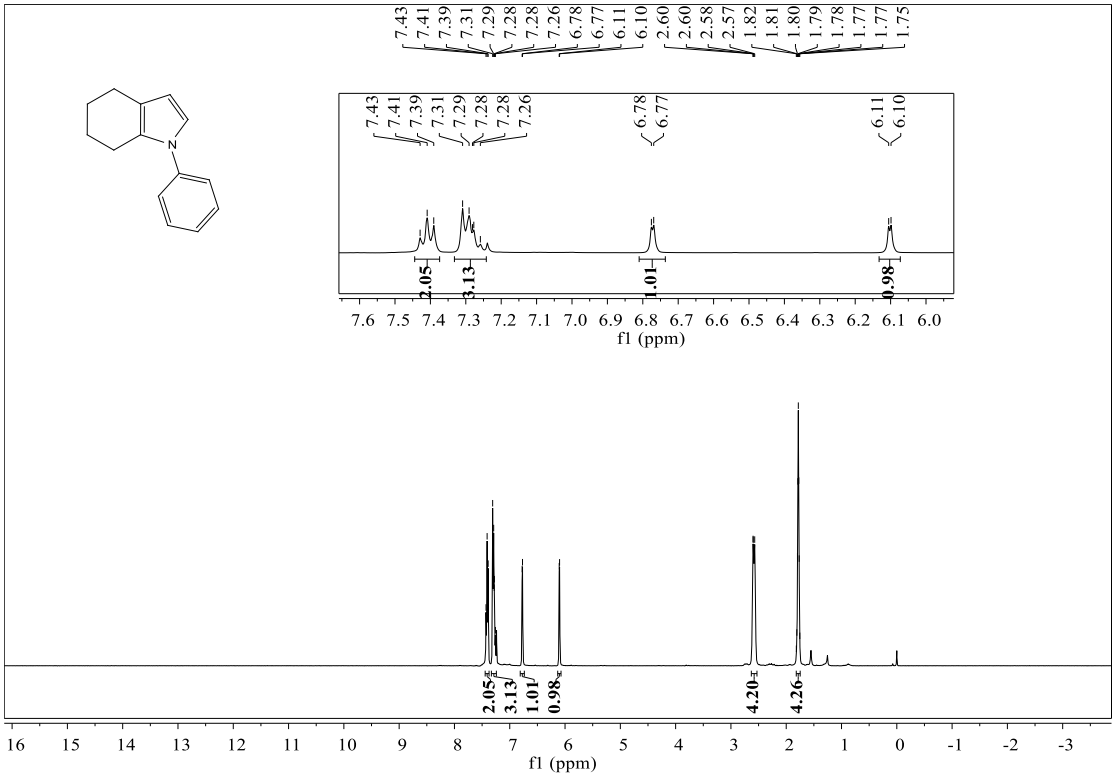
^{1}H NMR spectrum of compound 3aw (400 MHz, $CDCl_3$)



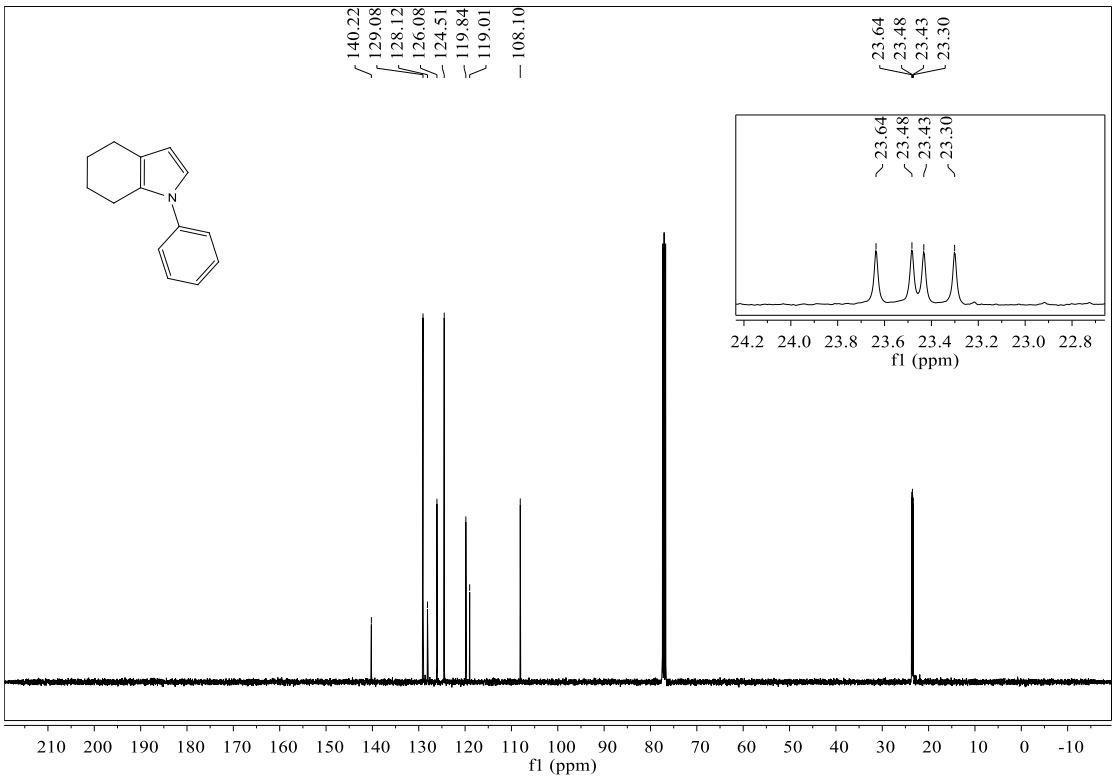
^{13}C NMR spectrum of compound 3aw (101 MHz, $CDCl_3$)



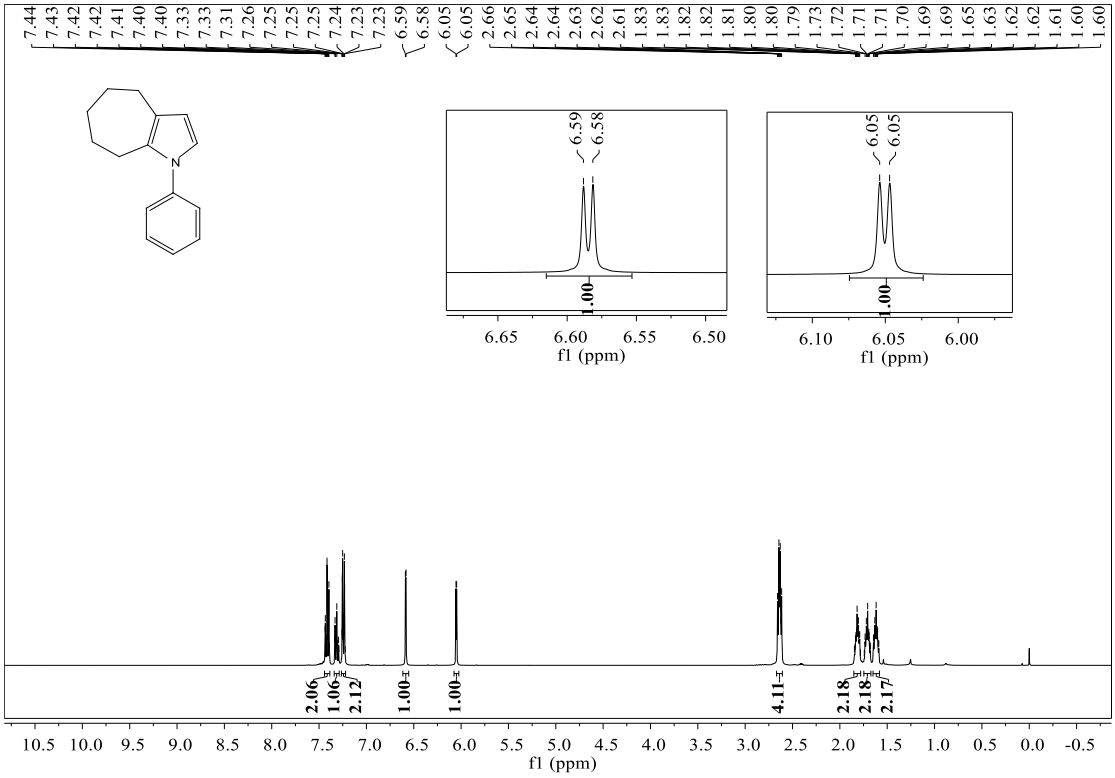
^{1}H NMR spectrum of compound 3ax (400 MHz, $CDCl_3$)



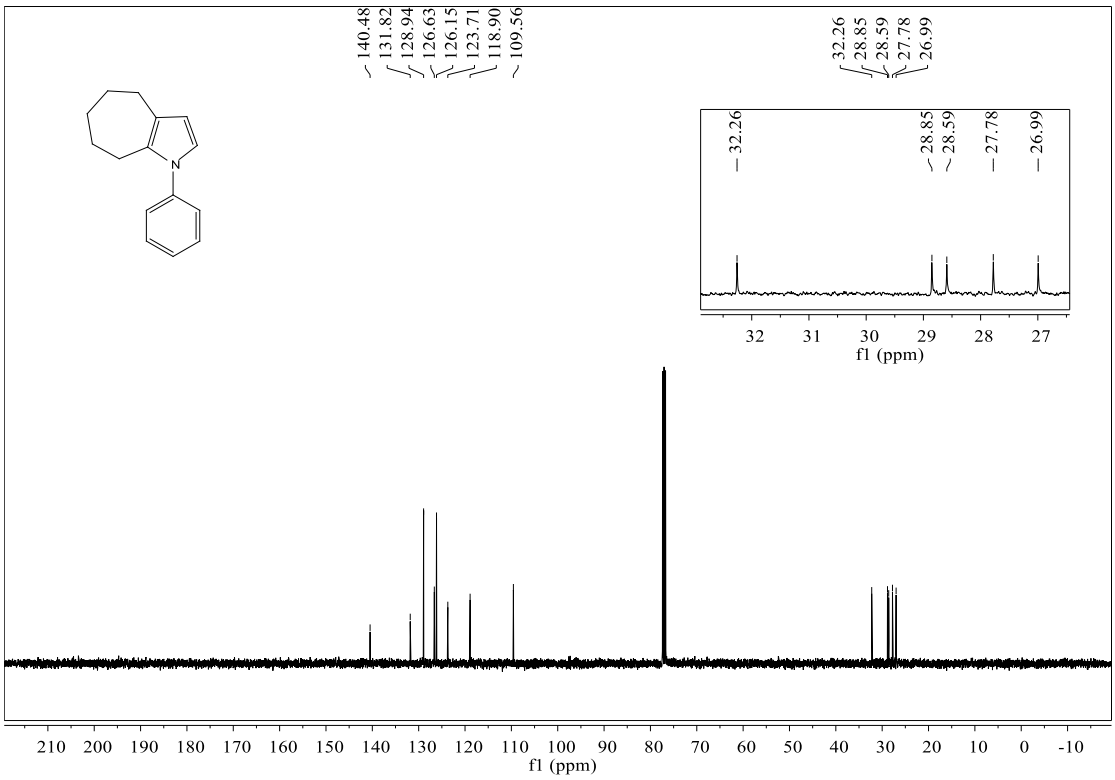
^{13}C NMR spectrum of compound 3ax (101 MHz, $CDCl_3$)



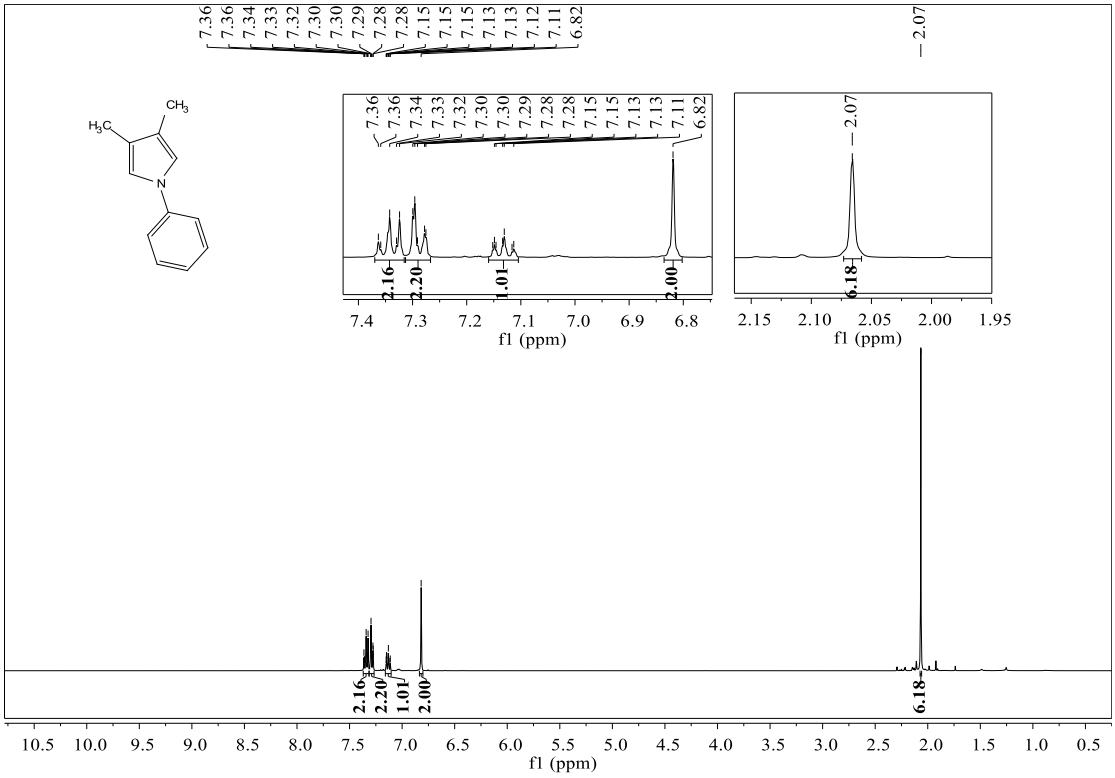
^{1}H NMR spectrum of compound 3ay (400 MHz, $CDCl_3$)



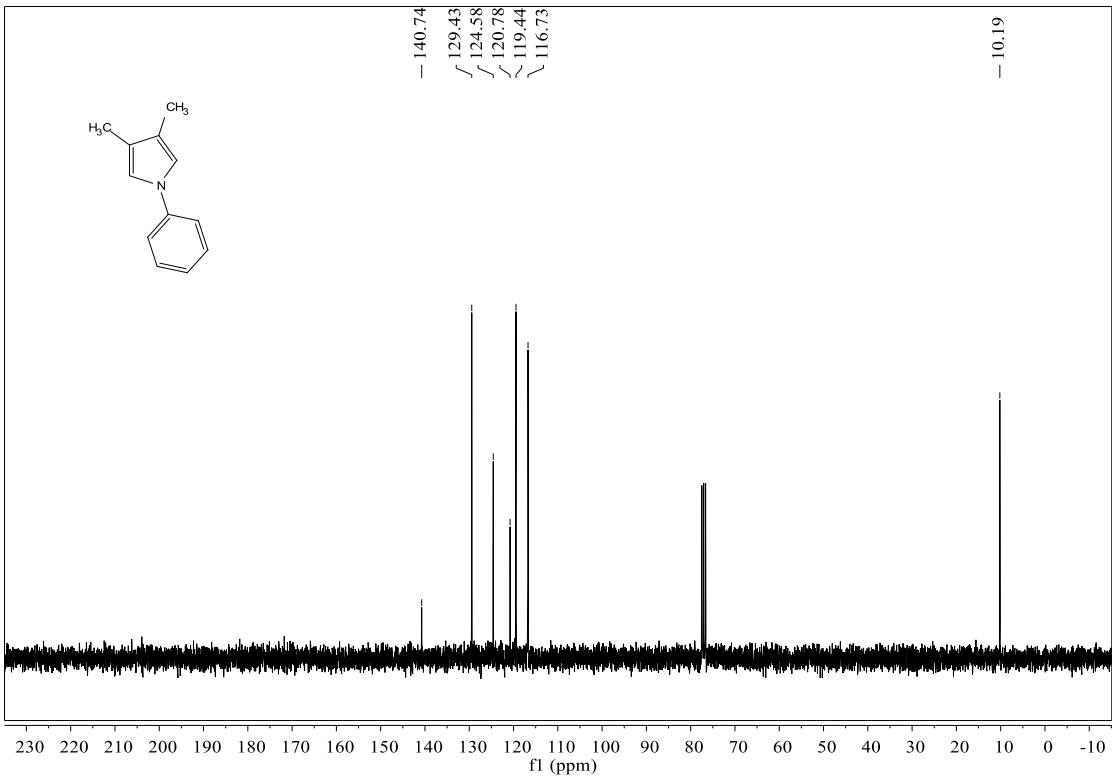
^{13}C NMR spectrum of compound 3ay (101 MHz, $CDCl_3$)



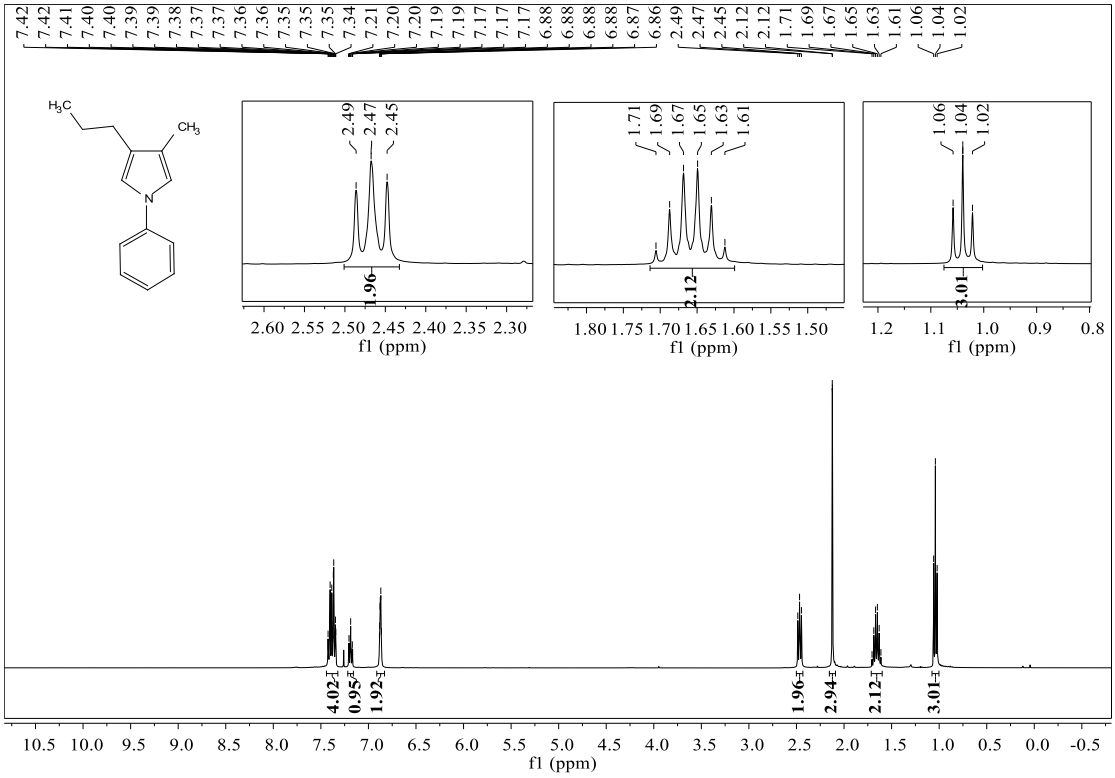
^{1}H NMR spectrum of compound 3az (400 MHz, $CDCl_3$)



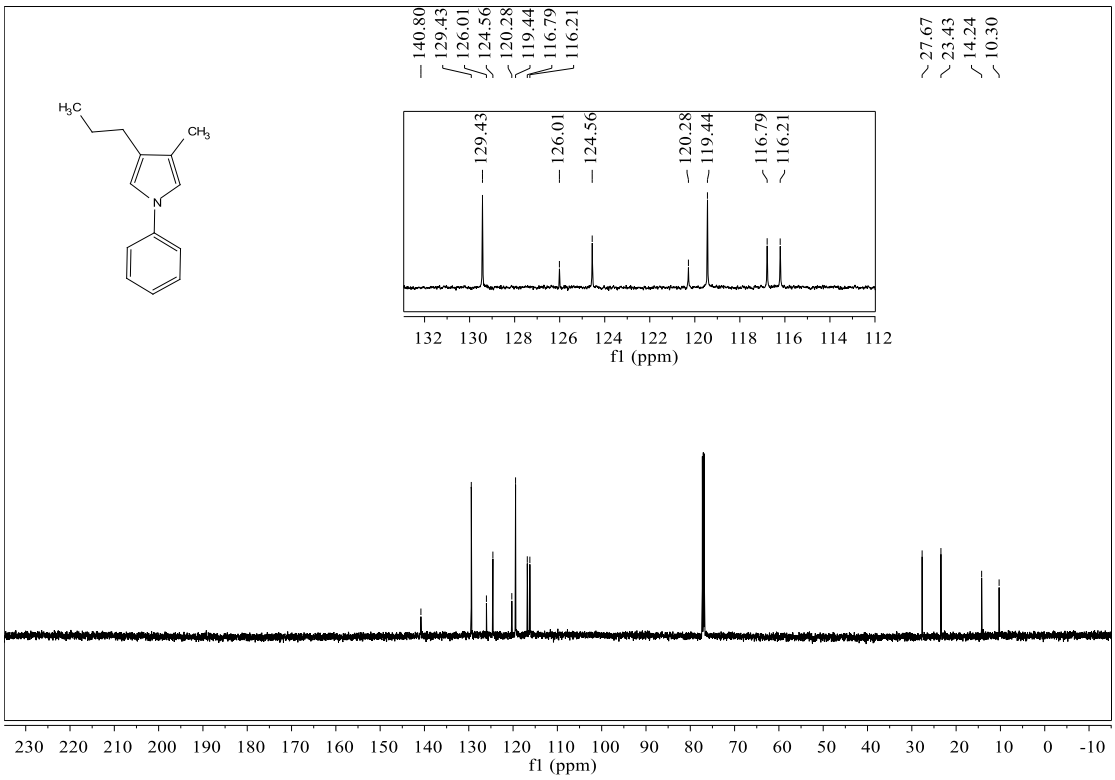
^{13}C NMR spectrum of compound 3az (76 MHz, $CDCl_3$)



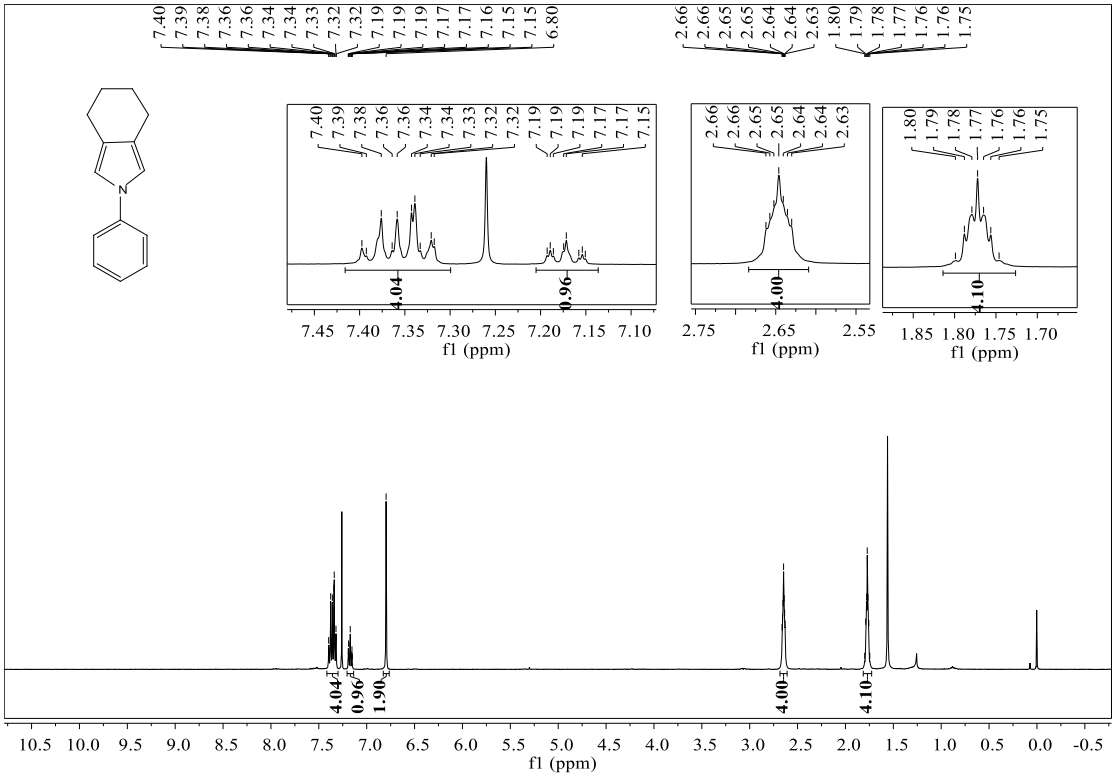
^{1}H NMR spectrum of compound 3aaa (400 MHz, $CDCl_3$)



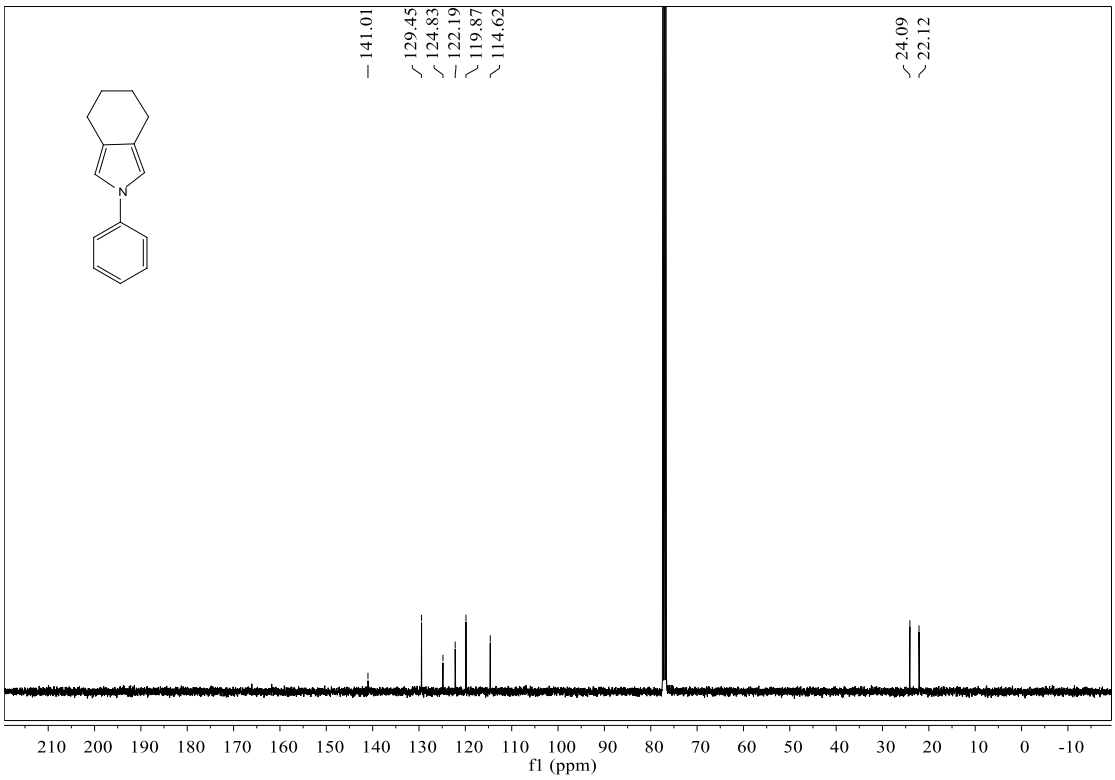
^{13}C NMR spectrum of compound 3aaa (151 MHz, $CDCl_3$)



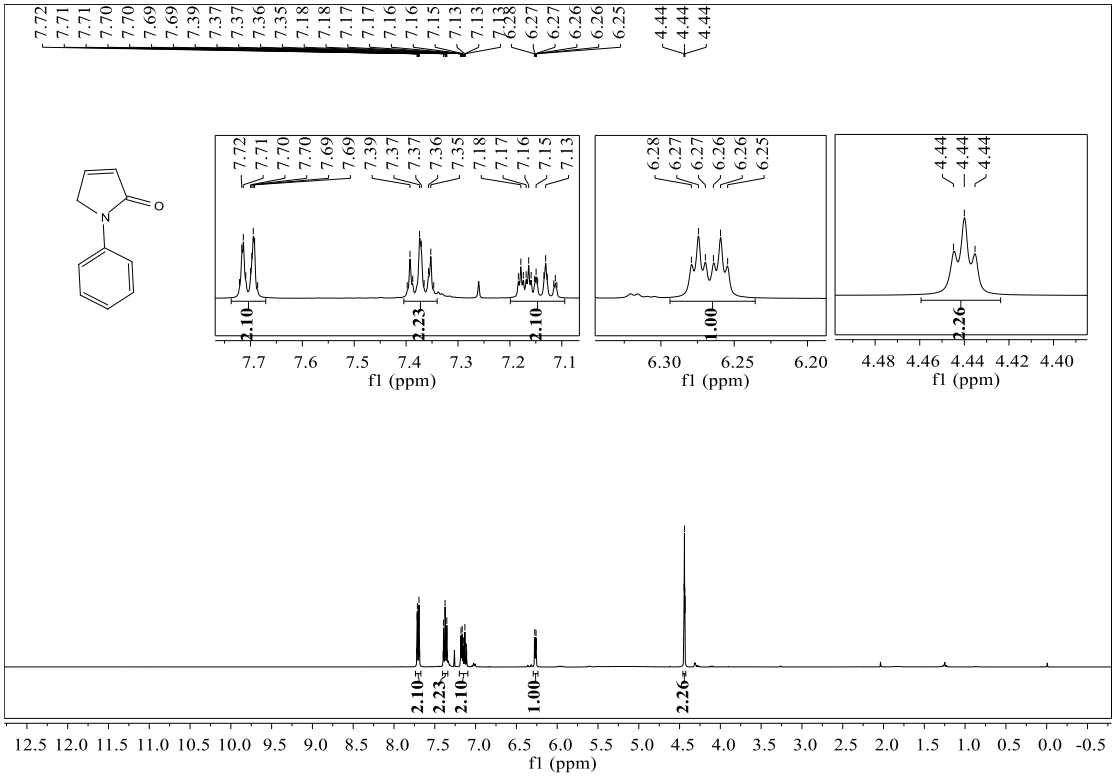
^{1}H NMR spectrum of compound 3aab (400 MHz, $CDCl_3$)



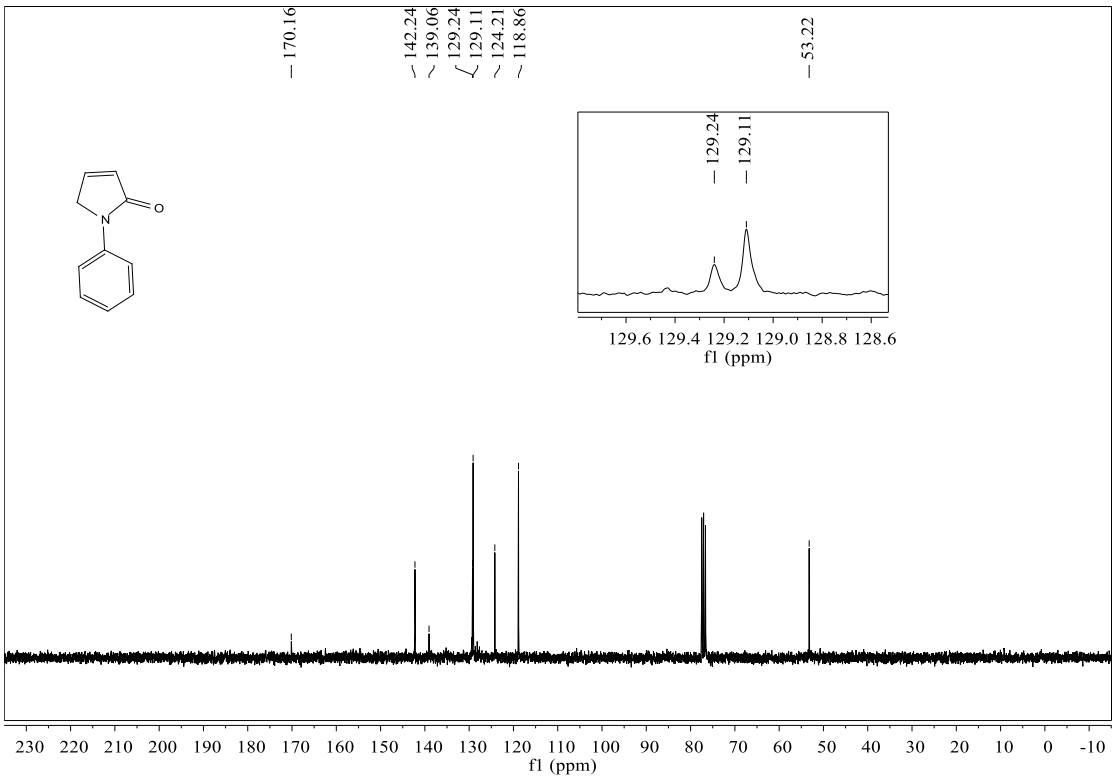
^{13}C NMR spectrum of compound 3aab (101 MHz, $CDCl_3$)



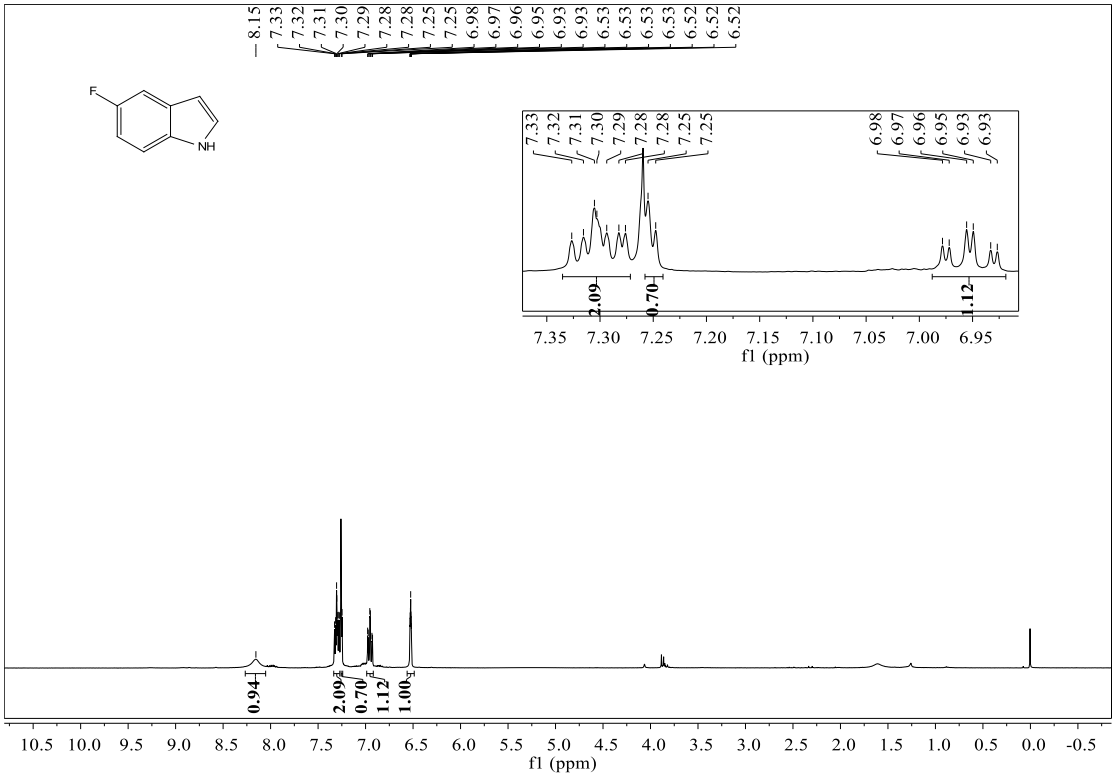
^{1}H NMR spectrum of compound 10 (400 MHz, $CDCl_3$)



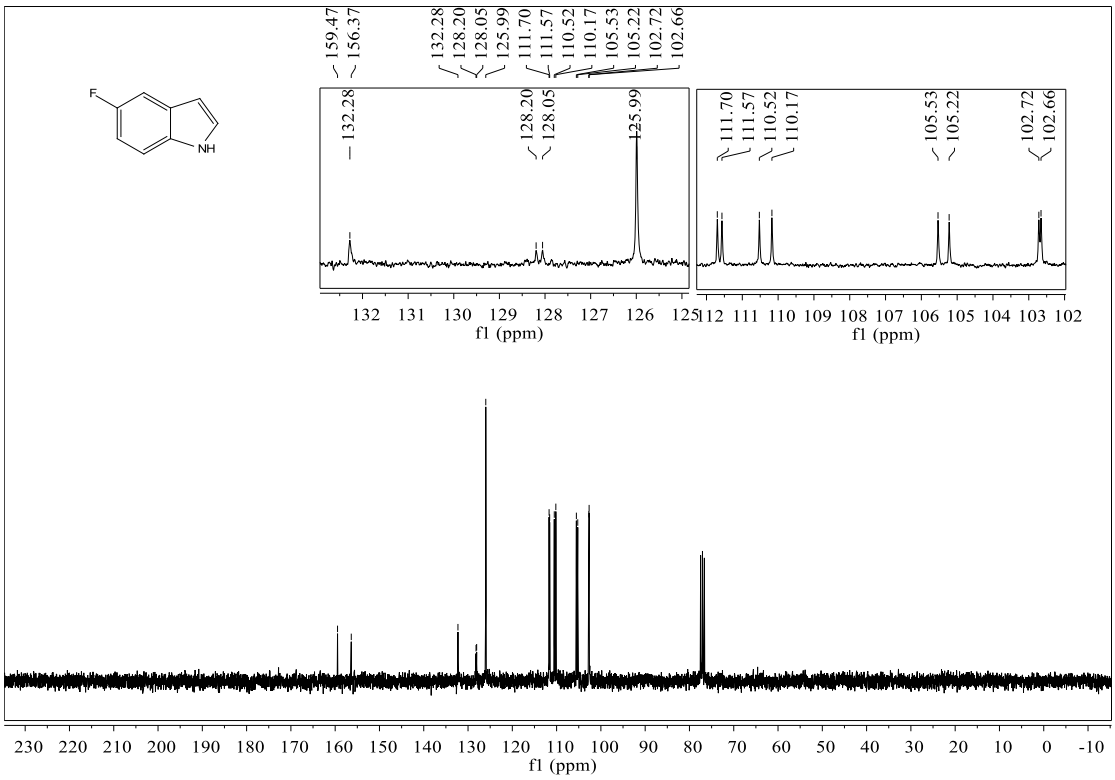
^{13}C NMR spectrum of compound 10 (76 MHz, $CDCl_3$)



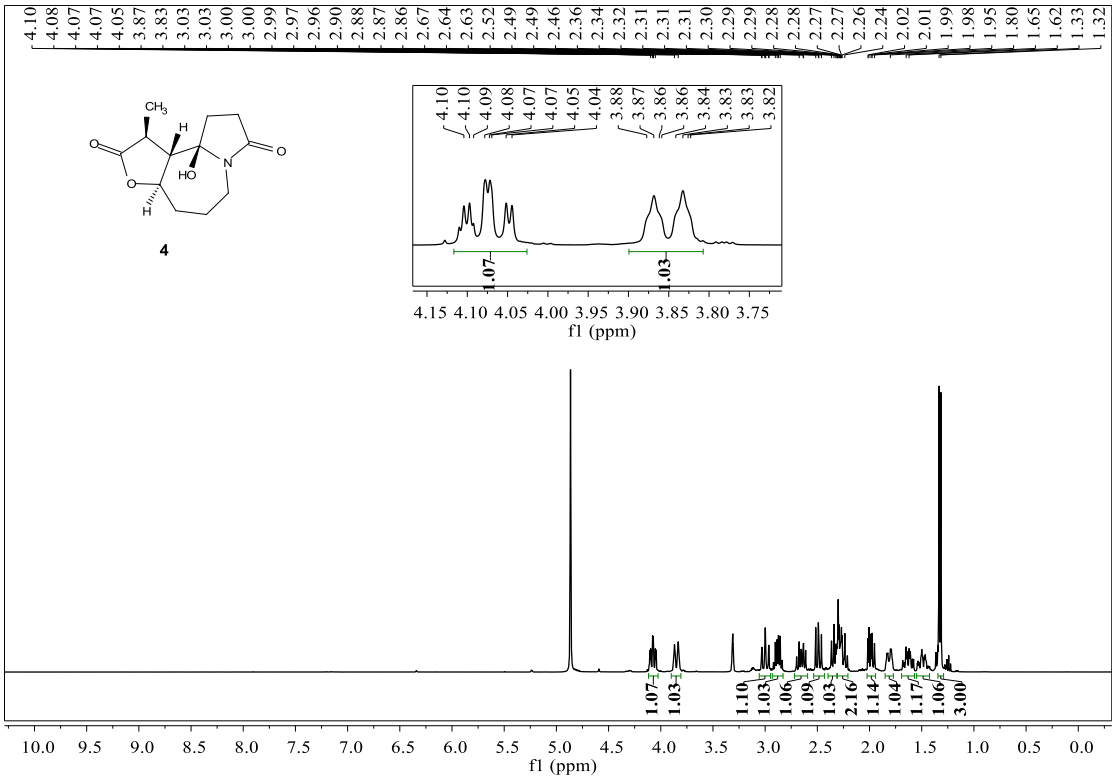
^{1}H NMR spectrum of compound 7 (400 MHz, $CDCl_3$)



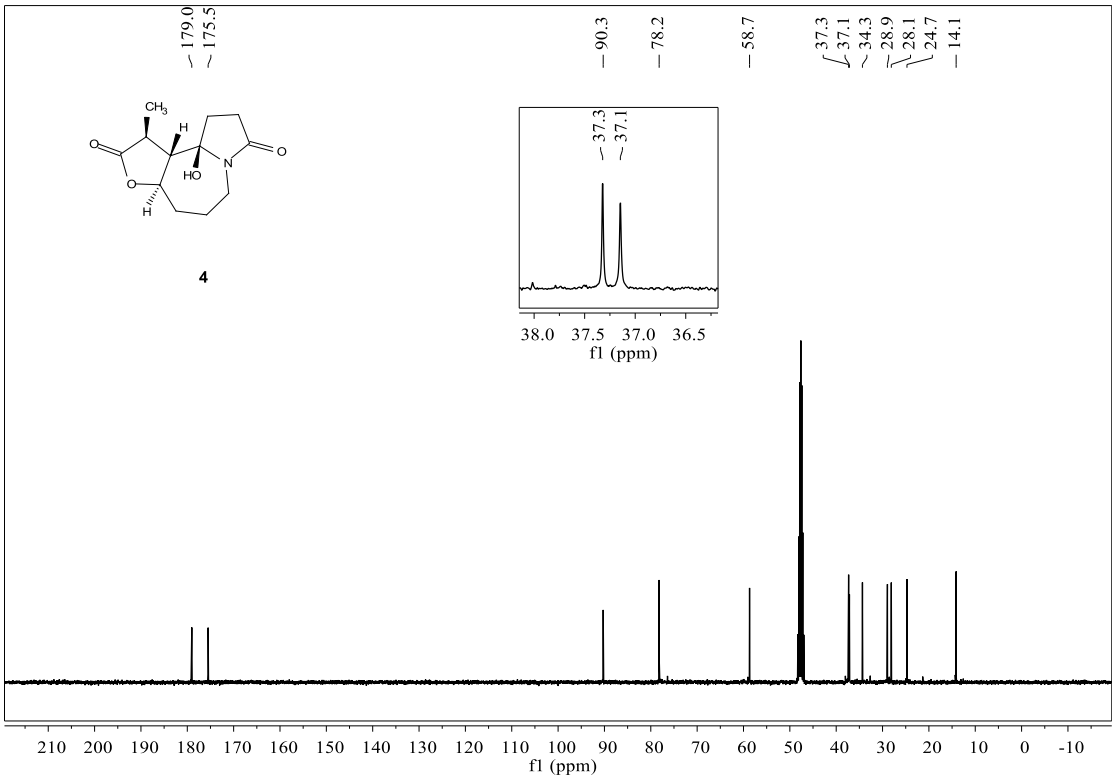
^{13}C NMR spectrum of compound 7 (76 MHz, $CDCl_3$)



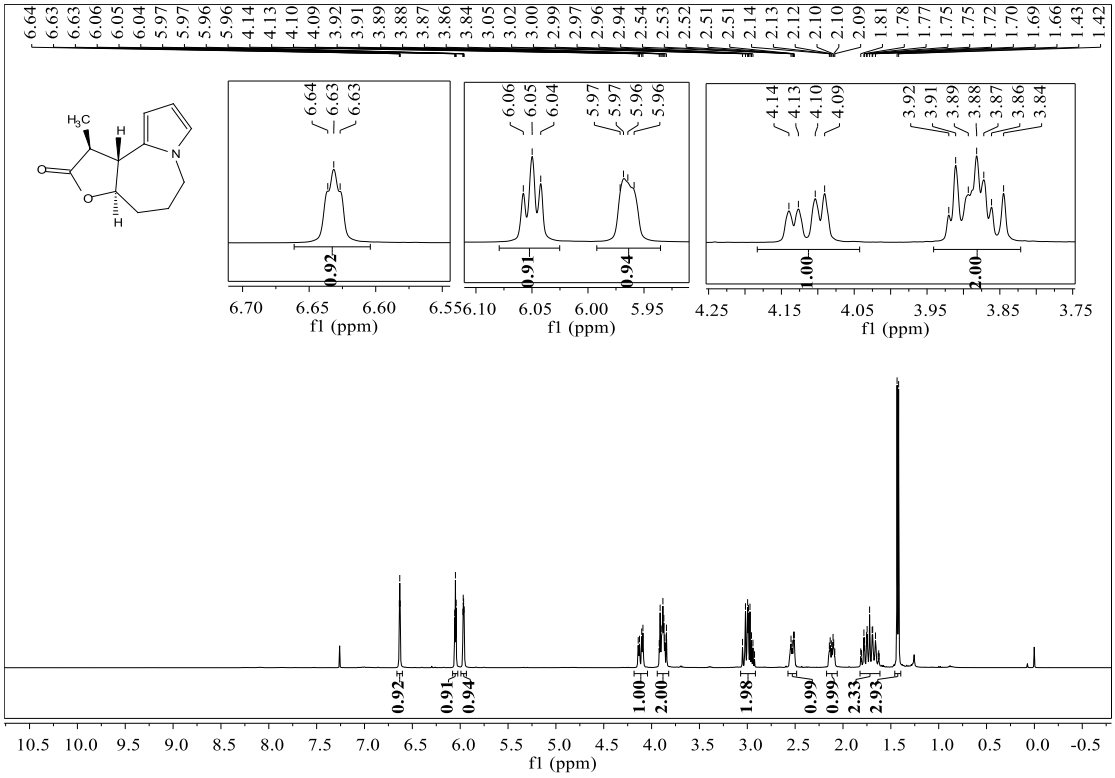
^{1}H NMR spectrum of compound 4 (400 MHz, methanol-d_4)



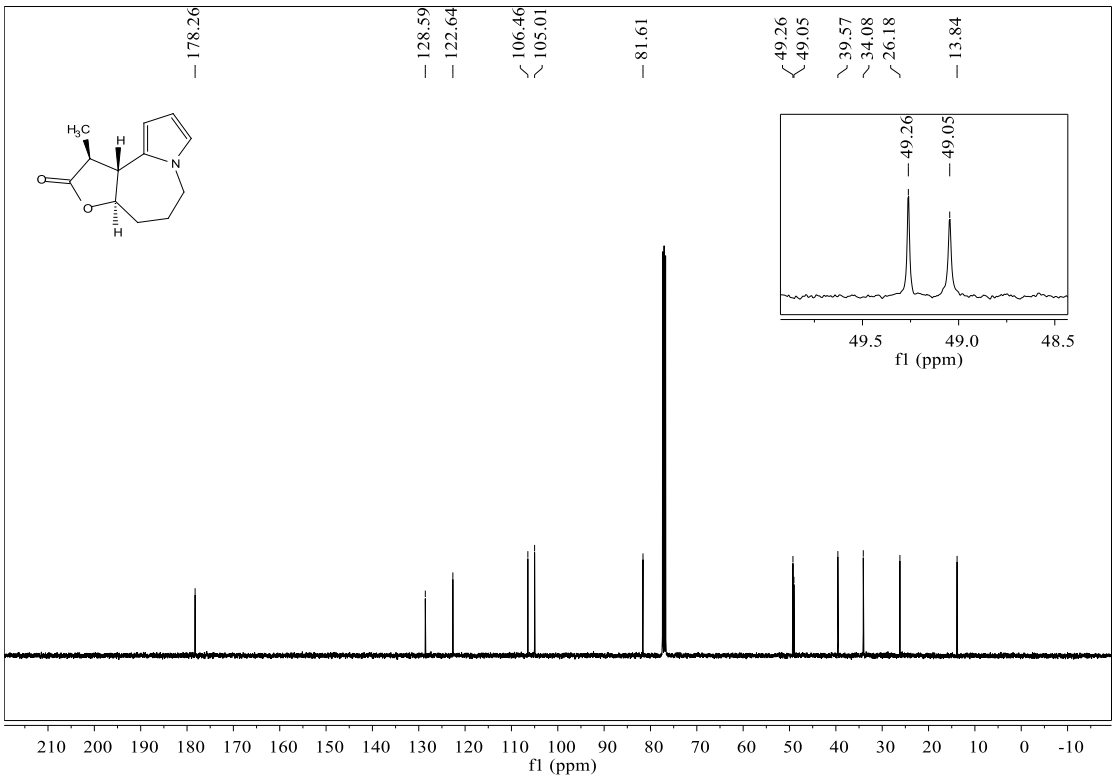
^{13}C NMR spectrum of compound 4 (101 MHz, methanol-d_4)



^{1}H NMR spectrum of compound 5 (400 MHz, $CDCl_3$)



^{13}C NMR spectrum of compound 5 (101 MHz, $CDCl_3$)



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