

Supporting Information

A Bifunctional Hypercrosslinked Polymer Valorised from Expired Pharmaceutical: Efficient Lewis Acid and Phase Transfer Catalyst

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1. Materials and General Methods

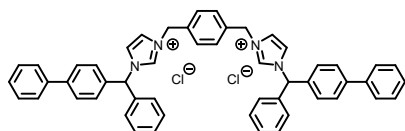
Unless specially indicated, all chemical reagents were purchased from commercial sources and were used as received without further purification. Fourier transform infrared spectroscopy (FT-IR) spectra were recorded on a Bruker INVENIO-R FT-IR spectrophotometer. Scanning electron microscopy (SEM) and was carried out on an VEGA3 TESCAN at 15 kV. N₂ sorption curves were obtained by Micromeritics ASAP 2420-4MP Plus automated sorption analyzer under 77 K. Thermogravimetric analysis (TGA) was performed using Pyris1 TGA in N₂ atmosphere by heating from room temperature to 600 °C. The X-ray photoelectron spectra (XPS) were obtained with the Thermo Scientific K-Alpha. ¹H and ¹³C NMR spectra of organic compounds were recorded on Bruker 400. Chemical shifts are expressed in ppm relative to Me₄Si in CDCl₃ or DMSO-d₆.

2. Experimental Section

Reclaim of bifonazole

To a flask containing 10 mL expired bifonazole solution was added 20 mL water. Then some white precipitate appeared. After filtering and drying, pure bifonazole was obtained.

Synthesis of Monomer B



2 mmol bifonazole and 1 mmol *p*-xylylene dichloride (PXC) and 5 mL CH₃CN were added into a round bottom flask. The solution was stirred at 60 °C for 72 h. During the

reaction, lots of white solid was precipitated. After reaction, the solid was recovered by filtration, washed three times with CH₃CN and EtOH, and dried at 70 °C under an air atmosphere. **Monomer B**: ¹H NMR (400 MHz, DMSO) δ 9.59 (s, 2H), 7.96 (t, *J* = 1.8 Hz, 2H), 7.87 – 7.81 (m, 2H), 7.76 (d, *J* = 8.4 Hz, 4H), 7.68 (d, *J* = 7.5 Hz, 4H), 7.55 – 7.43 (m, 14H), 7.43 – 7.31 (m, 12H), 5.50 (s, 4H). ¹³C NMR (101 MHz, DMSO) δ 141.14, 139.63, 137.61, 137.25, 136.68, 135.76, 129.68, 129.54, 129.53, 129.45, 129.31, 128.57, 128.39, 127.85, 127.24, 123.73, 123.23, 65.99, 52.14, 40.62, 40.41, 40.20, 39.99, 39.78, 39.58, 39.37.

Synthesis of HCP-B

The DCE was used instead of CH₃CN in the synthesis of Monomer B. After reaction, 3 mmol PXC and 3 mmol FeCl₃ were added into the reaction solution. Then the solution was heated to 90 °C for refluxing. After 24 h, the solid was recovered by filtration, then washed by HCl (3 N) and EtOH. Resulting material was obtained via Soxhlet extraction for 48 h using EtOH and drying in a vacuum oven at 60 °C overnight.

Synthesis of **HCP-E/C/M** was obtained according to the above procedure.

Three components reaction process

Add HCP-B (100 mg), 0.2 mmol indole or its derivatives, 0.4 mmol glycolaldehyde acetal, 0.4 mmol 1,3-dicarbonyl compounds to a V-type flask equipped with a magnetic stirring bar. The mixture was stirred at 60 °C for 8 hours. After the reaction, the solid was separated by centrifugation, washed several times with ethanol, and dried in a vacuum for later use. The product was isolated by preparative thin-layer chromatography using a mixture of ethyl acetate and petroleum ether as eluent.

Typical procedure for the synthesis of 7a

To a tube-type flask containing mixture of L-hydroxyproline **5a** (0.5 mmol) and benzaldehyde (1.0 mmol) in 2 mL DMSO was added HCP-B (15 mg). The tube reactor

equipped with triangular magnetic stirrer then reacted at 130 °C for 12 hours and the reaction was monitored by TLC. After reaction, reaction mixture allowed to cool to room temperature and was centrifuged. Then, the organic layer was washed by 100 mL water, and extracted three times using EA. After concentration, the product was isolated by preparative thin-layer chromatography using a mixture of ethyl acetate and petroleum ether as eluent. The NMR is one of the isomers.

Characterization of HCPs

Table S1 Difference of the HCPs.

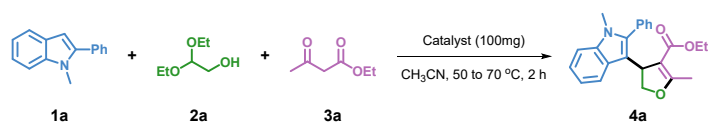
Sample	$S_{\text{BET}}^b / \text{m}^2 \text{ g}^{-1}$	Fe e (wt%)
HCP-B	125.4	5.62
HCP-C	596.0	0.38
HCP-M	25.2	1.51
HCP-E	404.8	1.02

Table S2 Difference of the fresh and cycled HCP-B.

Sample	$S_{\text{BET}}^b / \text{m}^2 \text{ g}^{-1}$	Total acid c /mmol g^{-1}	Peak temperature $^d / ^\circ\text{C}$	Fe e (wt%)
HCP-B	125.4	0.157	144.4; 265.8	5.62
The used a	15.1	0.178	116.1; 228.5	3.28

a The HCP-IL was performed in the three components reaction for 5 times. b BET surface area. c Measured by TPD-NH₃. d Temperature corresponding to desorption peak of NH₃. e Measured by atomic emission spectroscopy.

Table S3 Controlled experiments.



Entry ^a	Temp. (°C)	Yield (%) ^b	Active energy ^c
1	50	12	> 50 KJ/mol
2	60	23	
3	70	37	

^a Standard conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), **3a** (0.4 mmol), catalyst: HCP-B (100 mg), solvent (1 mL), 2 h. ^b Isolated yield. N.D. = No desired product. ^c Approximately calculated according to the Arrhenius equation.

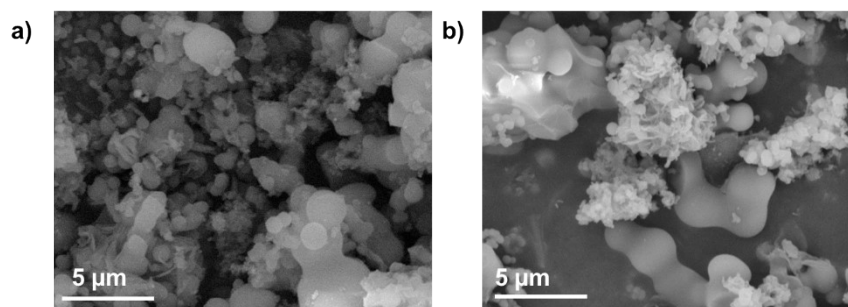


Fig S1 SEM image of HCP-B: a) fresh HCP-B; b) the used HCP-B for 5 cycles.

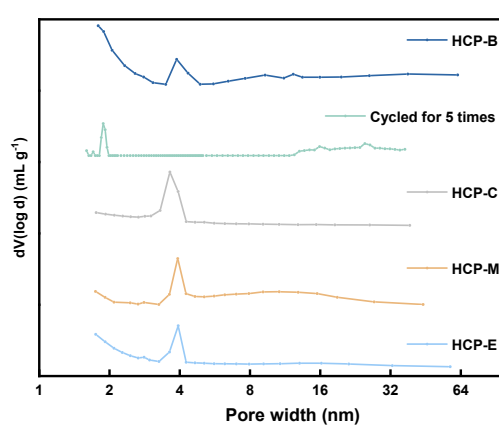


Fig S2 Pore size distributions of different polymers.

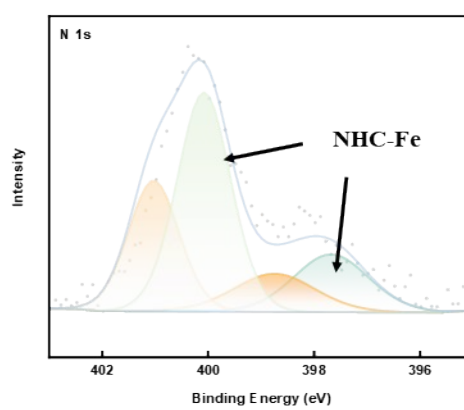


Fig S3 N 1s XPS of HCP-B.

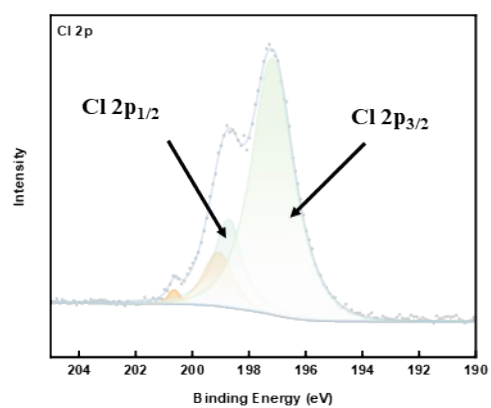


Fig S4 Cl 2p XPS of HCP-B.

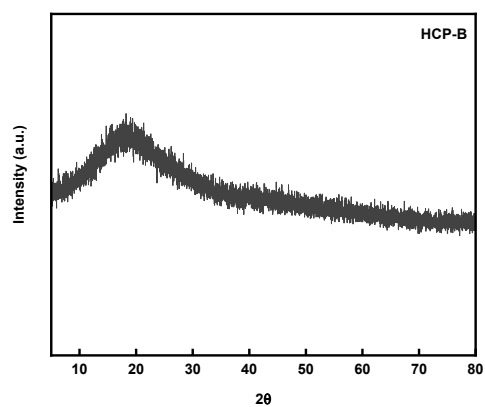
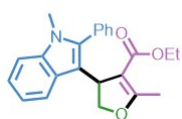
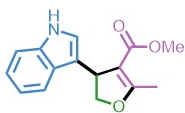


Fig S5 XRD of HCP-B.

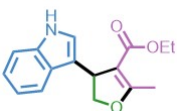
NMR data for some products



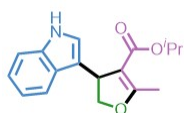
Ethyl 2-methyl-4-(1-methyl-2-phenyl-1*H*-indol-3-yl)-4,5-dihydrofuran-3-carboxylate (4a): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.41 – 7.55 (m, 6H), 7.33 (d, $J = 8.2$ Hz, 1H), 7.22 (d, $J = 8.4$ Hz, 1H), 7.09 (t, $J = 7.5$ Hz, 1H), 4.59 (d, $J = 4.6$ Hz, 2H), 4.53 – 4.37 (m, 1H), 3.97 (qd, $J = 7.1, 5.1$ Hz, 2H), 3.55 (s, 3H), 2.28 (s, 3H), 1.05 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.45, 165.92, 138.14, 137.47, 131.80, 131.01, 128.25, 128.13, 126.03, 121.55, 119.37, 119.29, 113.73, 109.42, 106.04, 77.23, 59.13, 39.56, 30.74, 14.48, 14.29.



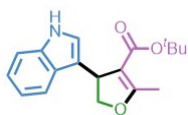
Methyl 4-(1*H*-indol-3-yl)-2-methyl-4,5-dihydrofuran-3-carboxylate (4b): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.06 (s, 1H), 7.55 (d, $J = 7.9$ Hz, 1H), 7.34 (d, $J = 8.1$ Hz, 1H), 7.18 (t, $J = 7.6$ Hz, 1H), 7.10 (t, $J = 7.6$ Hz, 1H), 6.98 (d, $J = 2.5$ Hz, 1H), 4.72 (dd, $J = 10.2, 8.7$ Hz, 1H), 4.61 (dd, $J = 10.1, 3.3$ Hz, 1H), 4.48 (dd, $J = 8.8, 4.4$ Hz, 1H), 3.57 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.93, 166.49, 136.71, 125.91, 121.98, 121.69, 119.43, 118.90, 118.27, 111.35, 105.97, 77.43, 50.76, 39.67, 14.39.



Ethyl 4-(1*H*-indol-3-yl)-2-methyl-4,5-dihydrofuran-3-carboxylate (4c): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.08 (s, 1H), 7.56 (d, $J = 7.9$ Hz, 1H), 7.34 (d, $J = 8.1$ Hz, 1H), 7.18 (t, $J = 7.6$ Hz, 1H), 7.09 (t, $J = 7.5$ Hz, 1H), 6.98 (d, $J = 2.5$ Hz, 1H), 4.72 (dd, $J = 10.1, 8.7$ Hz, 1H), 4.61 (dd, $J = 10.3, 3.2$ Hz, 1H), 4.49 (dd, $J = 8.7, 4.5$ Hz, 1H), 4.03 (qd, $J = 7.1, 1.8$ Hz, 2H), 2.33 (d, $J = 1.3$ Hz, 3H), 1.10 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.51, 166.06, 136.67, 125.95, 121.91, 121.78, 119.37, 118.97, 118.29, 111.31, 106.30, 77.30, 59.32, 39.76, 14.38, 14.24.



iso-Propyl 4-(1*H*-indol-3-yl)-2-methyl-4,5-dihydrofuran-3-carboxylate (4d): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.15 (s, 1H), 7.56 (d, $J = 7.9$ Hz, 1H), 7.32 (d, $J = 8.1$ Hz, 1H), 7.16 (t, $J = 7.5$ Hz, 1H), 7.08 (t, $J = 7.5$ Hz, 1H), 6.95 (d, $J = 2.4$ Hz, 1H), 4.91 (hept, $J = 6.3$ Hz, 1H), 4.71 (dd, $J = 10.3, 8.7$ Hz, 1H), 4.60 (dd, $J = 10.3, 4.6$ Hz, 1H), 4.48 (dd, $J = 8.7, 4.7$ Hz, 1H), 2.33 (s, 3H), 1.15 (d, $J = 6.2$ Hz, 3H), 0.95 (d, $J = 6.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.26, 165.69, 136.65, 126.00, 121.86, 121.82, 119.29, 118.99, 118.24, 111.30, 106.66, 77.24, 66.49, 39.81, 22.05, 21.75, 14.37.



tert-Butyl

4-(1*H*-indol-3-yl)-2-methyl-4,5-dihydrofuran-3-

carboxylate (4e): brown liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.06

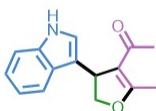
(s, 1H), 7.56 (d, $J = 7.9$ Hz, 1H), 7.34 (d, $J = 8.1$ Hz, 1H), 7.17 (t, $J =$

7.6 Hz, 1H), 7.08 (t, $J = 7.5$ Hz, 1H), 6.98 (d, $J = 2.4$ Hz, 1H), 4.69 (dd, $J = 10.3$, 8.7

Hz, 1H), 4.57 (dd, $J = 10.3$, 5.0 Hz, 1H), 4.43 (dd, $J = 8.7$, 5.0 Hz, 1H), 2.31 (s, 3H),

1.28 (s, 9H). ^{13}C NMR (101 MHz, CDCl_3) δ 167.64, 165.56, 136.67, 126.04, 121.83,

121.69, 119.28, 119.04, 118.46, 111.21, 107.41, 79.36, 77.01, 40.08, 28.27, 14.30.



1-(4-(1*H*-Indol-3-yl)-2-methyl-4,5-dihydrofuran-3-yl) ethan-1-one

(4f): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.14 (s, 1H), 7.55 (d,

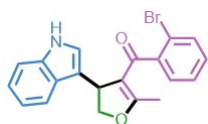
$J = 7.9$ Hz, 1H), 7.38 (d, $J = 8.2$ Hz, 1H), 7.25 – 7.17 (m, 1H), 7.16 –

7.08 (m, 1H), 7.00 (d, $J = 2.5$ Hz, 1H), 4.75 (dd, $J = 10.3$, 8.5 Hz, 1H), 4.67 (dd, $J =$

10.3, 4.5 Hz, 1H), 4.40 (dd, $J = 8.6$, 4.5 Hz, 1H), 2.38 (s, 3H), 2.01 (s, 3H). ^{13}C NMR

(101 MHz, CDCl_3) δ 195.84, 169.44, 136.83, 125.90, 122.33, 121.89, 119.67, 118.70,

118.14, 115.01, 111.48, 77.92, 40.12, 29.38, 15.14.



(4-(1*H*-Indol-3-yl)-2-methyl-4,5-dihydrofuran-3-yl)

(2-

bromophenyl) methanone (4g): yellow liquid. ^1H NMR (400 MHz,

CDCl_3) δ 8.02 (s, 1H), 7.53 – 7.44 (m, 2H), 7.31 (d, $J = 8.1$ Hz, 1H),

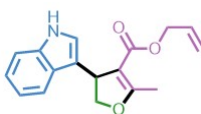
7.20 – 7.10 (m, 3H), 7.06 (t, $J = 7.5$ Hz, 1H), 6.95 – 6.88 (m, 1H), 6.86 (d, $J = 2.4$ Hz,

1H), 4.86 – 4.73 (m, 2H), 4.57 (dd, $J = 7.0$, 3.1 Hz, 1H), 1.96 (s, 3H). ^{13}C NMR (101

MHz, CDCl_3) δ 191.19, 171.78, 142.90, 136.70, 132.69, 130.11, 127.81, 127.18,

125.72, 122.20, 121.92, 119.37, 118.83, 118.43, 117.12, 116.51, 111.36, 77.70, 40.07,

14.86.



Allyl

4-(1*H*-indol-3-yl)-2-methyl-4,5-dihydrofuran-3-

carboxylate (4h): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.02

(s, 1H), 7.56 (d, $J = 7.9$ Hz, 1H), 7.35 (d, $J = 8.1$ Hz, 1H), 7.18 (t, J

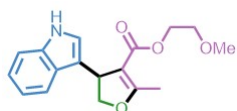
$= 7.6$ Hz, 1H), 7.10 (t, $J = 7.5$ Hz, 1H), 7.01 (d, $J = 2.5$ Hz, 1H), 5.77 (ddt, $J = 17.1$,

10.7, 5.4 Hz, 1H), 5.12 – 5.03 (m, 2H), 4.74 (dd, $J = 10.2$, 8.7 Hz, 1H), 4.64 (dd, $J =$

10.2, 4.3 Hz, 1H), 4.55 – 4.43 (m, 3H), 2.35 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ

169.14, 165.61, 136.69, 132.77, 125.90, 121.98, 121.76, 119.44, 118.96, 118.24,

117.03, 111.30, 105.97, 77.40, 64.00, 39.71, 14.45.



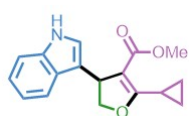
1-(4-(1*H*-Indol-3-yl)-2-methyl-4,5-dihydrofuran-3-yl)-3-

methoxypropan-1-one (4i): brown liquid. ^1H NMR (400 MHz,

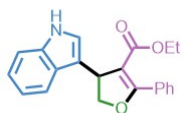
CDCl_3) δ 8.11 (s, 1H), 7.56 (d, $J = 7.9$ Hz, 1H), 7.33 (d, $J = 8.1$ Hz, 1H), 7.17 (t, $J =$

7.6 Hz, 1H), 7.09 (t, $J = 7.0$ Hz, 1H), 6.99 (d, $J = 2.4$ Hz, 1H), 4.73 (dd, $J = 10.2$, 8.6

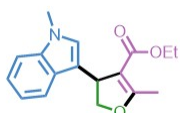
Hz, 1H), 4.67 – 4.60 (m, 1H), 4.49 (dd, $J = 8.6, 4.5$ Hz, 1H), 4.19 – 4.05 (m, 2H), 3.49 – 3.36 (m, 2H), 3.23 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 169.13, 165.86, 136.66, 125.95, 121.90, 121.86, 119.39, 118.93, 118.18, 111.30, 106.10, 77.45, 70.60, 62.39, 58.73, 39.66, 14.43.



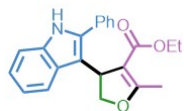
Methyl 2-cyclopropyl-4-(1H-indol-3-yl)-4,5-dihydrofuran-3-carboxylate (4j): white solid, mp 80-81 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.04 (s, 1H), 7.54 (d, $J = 7.9$ Hz, 1H), 7.34 (d, $J = 8.1$ Hz, 1H), 7.18 (t, $J = 7.6$ Hz, 1H), 7.10 (t, $J = 7.5$ Hz, 1H), 7.00 (d, $J = 2.4$ Hz, 1H), 4.61 (d, $J = 3.2$ Hz, 2H), 4.45 – 4.34 (m, 1H), 3.59 (s, 3H), 2.83 (tt, $J = 8.3, 5.0$ Hz, 1H), 1.30 – 1.13 (m, 1H), 1.09 – 1.01 (m, 1H), 0.98 – 0.90 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 172.50, 166.99, 136.73, 125.97, 121.93, 121.67, 119.38, 118.98, 118.47, 111.31, 105.17, 77.15, 50.69, 39.95, 9.36, 8.03, 7.28.



Ethyl 4-(1H-indol-3-yl)-2-phenyl-4,5-dihydrofuran-3-carboxylate (4k): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.04 (s, 1H), 7.89 – 7.82 (m, 2H), 7.67 (d, $J = 8.0$ Hz, 1H), 7.47 – 7.36 (m, 4H), 7.24 – 7.15 (m, 1H), 7.15 – 7.06 (m, 2H), 4.93 – 4.79 (m, 2H), 4.64 (dd, $J = 7.6, 3.3$ Hz, 1H), 3.98 (qt, $J = 8.1, 4.1$ Hz, 2H), 1.01 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.47, 165.20, 136.66, 130.48, 130.21, 129.56, 127.71, 125.97, 121.99, 121.93, 119.54, 119.10, 118.19, 111.34, 106.81, 76.87, 59.61, 41.46, 13.99.

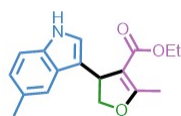


Ethyl 2-methyl-4-(1-methyl-1H-indol-3-yl)-4,5-dihydrofuran-3-carboxylate (4l): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.55 (d, $J = 7.9$ Hz, 1H), 7.27 (t, $J = 8.3$ Hz, 1H), 7.21 (t, $J = 7.6$ Hz, 1H), 7.08 (t, $J = 7.4$ Hz, 1H), 6.87 (s, 1H), 4.71 (dd, $J = 10.2, 8.7$ Hz, 1H), 4.59 (dd, $J = 10.5, 3.8$ Hz, 1H), 4.47 (dd, $J = 8.7, 4.4$ Hz, 1H), 4.03 (q, $J = 7.1$ Hz, 2H), 3.72 (s, 3H), 2.33 (s, 3H), 1.11 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.29, 166.01, 137.35, 126.47, 126.40, 121.47, 119.08, 118.83, 116.89, 109.34, 106.49, 77.57, 59.29, 39.61, 32.65, 14.37, 14.26.

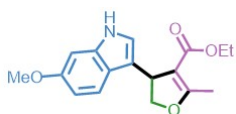


Ethyl 2-methyl-4-(2-phenyl-1H-indol-3-yl)-4,5-dihydrofuran-3-carboxylate (4m): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.03 (s, 1H), 7.77 – 7.70 (m, 2H), 7.53 (d, $J = 8.0$ Hz, 1H), 7.48 (t, $J = 7.6$ Hz, 2H), 7.39 (dd, $J = 13.4, 7.6$ Hz, 2H), 7.19 (t, $J = 7.6$ Hz, 1H), 7.09 (t, $J = 7.5$ Hz, 1H), 4.90 (dd, $J = 10.3, 7.1$ Hz, 1H), 4.67 (t, $J = 10.1$ Hz, 1H), 4.50 (dd, $J = 9.2, 6.4$ Hz, 1H), 3.97 (q, $J = 7.1$ Hz, 2H), 2.36 (s, 3H), 1.01 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.81, 165.98, 136.21, 135.12, 133.07, 128.86, 128.76, 127.91, 127.15, 122.11,

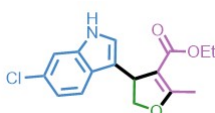
119.72, 119.67, 113.68, 110.92, 105.86, 77.23, 59.23, 39.27, 14.51, 14.20.



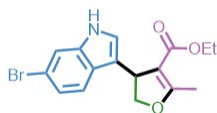
Ethyl 2-methyl-4-(5-methyl-1*H*-indol-3-yl)-4,5-dihydrofuran-3-carboxylate (4n): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.89 (s, 1H), 7.32 (s, 1H), 7.24 (d, $J = 8.4$ Hz, 1H), 7.01 (d, $J = 8.2$ Hz, 1H), 6.97 (d, $J = 2.4$ Hz, 1H), 4.72 (dd, $J = 10.2, 8.7$ Hz, 1H), 4.58 (dd, $J = 10.9, 4.2$ Hz, 1H), 4.48 (dd, $J = 8.7, 4.5$ Hz, 1H), 4.03 (q, $J = 7.1$ Hz, 2H), 2.45 (s, 3H), 2.33 (s, 3H), 1.10 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.36, 166.04, 134.96, 128.55, 126.17, 123.54, 121.79, 118.63, 117.98, 110.90, 106.26, 77.22, 59.26, 39.65, 21.61, 14.33, 14.23.



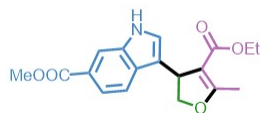
Ethyl 4-(6-methoxy-1*H*-indol-3-yl)-2-methyl-4,5-dihydrofuran-3-carboxylate (4o): brown liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.89 (s, 1H), 7.42 (d, $J = 8.7$ Hz, 1H), 6.90 (d, $J = 2.3$ Hz, 1H), 6.84 (d, $J = 2.2$ Hz, 1H), 6.77 (dd, $J = 8.7, 2.3$ Hz, 1H), 4.70 (dd, $J = 10.1, 8.7$ Hz, 1H), 4.56 (dd, $J = 10.3, 4.6$ Hz, 1H), 4.47 (dd, $J = 8.7, 4.6$ Hz, 1H), 4.03 (q, $J = 7.1$ Hz, 2H), 3.83 (s, 3H), 2.32 (s, 3H), 1.11 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.38, 166.01, 156.42, 137.42, 120.47, 120.37, 119.56, 118.33, 109.34, 106.37, 94.81, 77.23, 59.29, 55.67, 39.78, 14.35, 14.25.



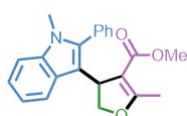
Ethyl 4-(6-chloro-1*H*-indol-3-yl)-2-methyl-4,5-dihydrofuran-3-carboxylate (4p): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.07 (s, 1H), 7.45 (d, $J = 8.5$ Hz, 1H), 7.32 (d, $J = 1.9$ Hz, 1H), 7.06 (dd, $J = 8.5, 1.9$ Hz, 1H), 6.99 (d, $J = 2.4$ Hz, 1H), 4.72 (dd, $J = 10.3, 8.9$ Hz, 1H), 4.58 (dd, $J = 9.5, 4.1$ Hz, 1H), 4.44 (dd, $J = 8.9, 4.7$ Hz, 1H), 4.03 (qt, $J = 7.2, 3.7$ Hz, 2H), 2.33 (s, 3H), 1.10 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.56, 165.89, 136.96, 127.91, 124.54, 122.36, 120.15, 119.81, 118.55, 111.21, 106.25, 77.21, 59.38, 39.64, 14.38, 14.24.



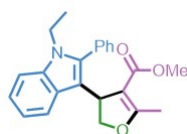
Ethyl 4-(6-bromo-1*H*-indol-3-yl)-2-methyl-4,5-dihydrofuran-3-carboxylate (4q): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.06 (s, 1H), 7.49 (d, $J = 1.8$ Hz, 1H), 7.41 (d, $J = 8.5$ Hz, 1H), 7.19 (dd, $J = 8.5, 1.7$ Hz, 1H), 6.98 (d, $J = 2.4$ Hz, 1H), 4.72 (dd, $J = 10.3, 8.9$ Hz, 1H), 4.58 (dd, $J = 10.2, 4.3$ Hz, 1H), 4.44 (dd, $J = 8.9, 4.7$ Hz, 1H), 4.02 (qt, $J = 6.9, 3.4$ Hz, 2H), 2.33 (s, 3H), 1.10 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.56, 165.87, 137.39, 124.84, 122.73, 122.31, 120.20, 118.62, 115.54, 114.20, 106.24, 77.20, 59.39, 39.62, 14.37, 14.24.



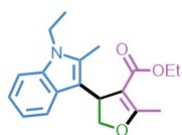
Methyl 3-(4-(ethoxycarbonyl)-5-methyl-2,3-dihydrofuran-3-yl)-1H-indole-6-carboxylate (4r): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 8.42 (s, 1H), 8.09 (d, $J = 1.5$ Hz, 1H), 7.78 (dd, $J = 8.4, 1.5$ Hz, 1H), 7.57 (d, $J = 8.4$ Hz, 1H), 7.17 (d, $J = 2.5$ Hz, 1H), 4.74 (dd, $J = 10.3, 8.9$ Hz, 1H), 4.62 (dd, $J = 10.8, 4.5$ Hz, 1H), 4.47 (dd, $J = 8.9, 4.6$ Hz, 1H), 4.03 (qt, $J = 7.1, 3.5$ Hz, 2H), 3.93 (s, 3H), 2.34 (s, 3H), 1.09 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.70, 168.15, 165.89, 135.89, 129.49, 125.19, 123.63, 120.48, 118.90, 118.52, 113.69, 106.26, 77.26, 59.40, 51.96, 39.59, 14.39, 14.23.



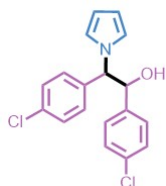
Methyl 2-methyl-4-(1-methyl-2-phenyl-1H-indol-3-yl)-4,5-dihydrofuran-3-carboxylate (4s): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.53 (d, $J = 7.9$ Hz, 2H), 7.48 (d, $J = 7.9$ Hz, 2H), 7.47 – 7.40 (m, 2H), 7.33 (d, $J = 8.1$ Hz, 1H), 7.27 – 7.21 (m, 1H), 7.14 – 7.06 (m, 1H), 4.60 (dd, $J = 10.5, 7.5$ Hz, 1H), 4.57 – 4.51 (m, 1H), 4.46 (dd, $J = 7.3, 4.5$ Hz, 1H), 3.54 (s, 3H), 3.50 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.67, 166.33, 138.21, 137.44, 131.86, 131.02, 128.28, 128.22, 125.90, 121.61, 119.36, 119.30, 113.71, 109.50, 105.86, 50.60, 39.51, 30.74, 14.45.



Methyl 4-(1-ethyl-2-phenyl-1H-indol-3-yl)-2-methyl-4,5-dihydrofuran-3-carboxylate (4t): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.71 – 7.41 (m, 5H), 7.40 – 7.18 (m, 3H), 7.09 (t, $J = 7.5$ Hz, 1H), 4.59 (dd, $J = 12.6, 10.3$ Hz, 1H), 4.52 – 4.43 (m, 2H), 3.99 (q, $J = 7.2$ Hz, 2H), 3.50 (s, 3H), 2.25 (s, 3H), 1.20 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 168.63, 166.27, 137.71, 136.20, 132.15, 130.95, 128.26, 126.21, 121.43, 119.39, 119.23, 113.91, 109.67, 105.85, 77.24, 50.52, 39.44, 38.52, 15.35, 14.38.

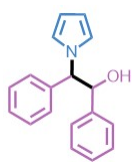


Ethyl 4-(1-ethyl-2-methyl-1H-indol-3-yl)-2-methyl-4,5-dihydrofuran-3-carboxylate (4u): yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.45 (d, $J = 7.9$ Hz, 1H), 7.29 – 7.23 (m, 1H), 7.17 – 7.08 (m, 1H), 7.07 – 6.98 (m, 1H), 4.76 – 4.66 (m, 1H), 4.60 (ddd, $J = 10.7, 7.4, 4.5$ Hz, 2H), 4.11 (q, $J = 7.2$ Hz, 2H), 3.96 (qd, $J = 7.1, 1.5$ Hz, 2H), 2.41 (s, 3H), 2.33 (s, 3H), 1.30 (t, $J = 7.2$ Hz, 3H), 1.08 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 167.72, 165.98, 135.73, 132.48, 126.08, 120.32, 118.77, 118.42, 112.29, 108.72, 106.45, 77.39, 77.07, 76.75, 76.31, 59.13, 39.29, 37.65, 15.31, 14.28, 10.12.

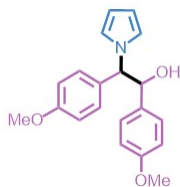


1,2-bis(4-chlorophenyl)-2-(1H-pyrrol-1-yl) ethanol (7a): yellow oil, dr: 52: 48. Two diastereoisomers were further isolated. ^1H NMR (400 MHz, CDCl_3) δ 7.30 (d, $J = 8.4$ Hz, 2H), 7.24 (d, $J = 2.3$ Hz, 2H), 7.22

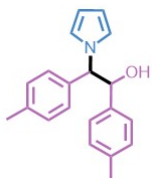
(d, $J = 2.4$ Hz, 2H), 7.07 (d, $J = 8.2$ Hz, 2H), 6.62 (t, $J = 2.2$ Hz, 2H), 6.05 (t, $J = 2.2$ Hz, 2H), 5.37 (d, $J = 6.9$ Hz, 1H), 5.06 (d, $J = 6.9$ Hz, 1H), 2.20 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.20, 135.53, 134.31, 133.99, 129.85, 128.85, 128.60, 127.55, 120.03, 108.67, 74.99, 68.59.



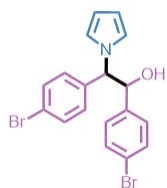
1,2-bisphenyl-2-(1H-pyrrol-1-yl) ethanol (7b): yellow oil, dr: 70: 30. ^1H NMR (400 MHz, CDCl_3) δ 7.34 – 7.29 (m, 2H), 7.23 – 7.17 (m, 6H), 7.08 (dd, $J = 6.8, 2.9$ Hz, 2H), 6.87 (t, $J = 2.2$ Hz, 2H), 6.20 (t, $J = 2.2$ Hz, 2H), 5.33 (dd, $J = 7.9, 2.3$ Hz, 1H), 5.17 (d, $J = 7.9$ Hz, 1H), 2.42 (d, $J = 2.8$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.01, 138.24, 128.49, 128.37, 128.33, 128.14, 127.58, 126.99, 120.60, 108.79, 76.34, 70.38.



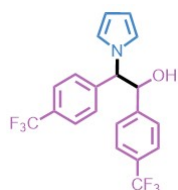
1,2-bis(4-methoxyphenyl)-2-(1H-pyrrol-1-yl) ethanol (7c): yellow oil, dr: 56: 44. ^1H NMR (400 MHz, CDCl_3) δ 7.26 (d, $J = 3.4$ Hz, 2H), 7.11 (d, $J = 6.9$ Hz, 2H), 6.87 (d, $J = 2.5$ Hz, 2H), 6.78 – 6.74 (m, 2H), 6.62 (t, $J = 2.4$ Hz, 2H), 6.02 (t, $J = 2.4$ Hz, 2H), 5.35 (d, $J = 7.3$ Hz, 1H), 5.08 (d, $J = 7.3$ Hz, 1H), 3.79 (s, 3H), 3.78 (s, 3H), 2.08 (d, $J = 3.4$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.49, 159.33, 133.05, 129.61, 129.47, 127.49, 120.00, 116.03, 114.81, 108.01, 75.38, 69.06, 55.29, 55.19.



1,2-bis(4-methylphenyl)-2-(1H-pyrrol-1-yl) ethanol (7d): yellow oil, dr: 71: 29. ^1H NMR (400 MHz, CDCl_3) δ 7.09 – 7.03 (m, 8H), 6.86 (t, $J = 2.2$ Hz, 2H), 6.19 (t, $J = 2.2$ Hz, 2H), 5.31 (d, $J = 7.9$ Hz, 1H), 5.15 (d, $J = 7.9$ Hz, 1H), 2.28 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 137.72, 137.52, 137.15, 135.34, 129.15 (2C), 129.02 (2C), 127.52 (2C), 126.90 (2C), 120.52 (2C), 108.62 (2C), 76.10, 69.94, 21.19, 21.08.

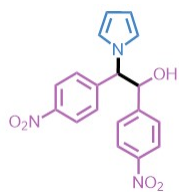


1,2-bis(4-bromophenyl)-2-(1H-pyrrol-1-yl) ethanol (7e): yellow oil, dr: 58: 42. ^1H NMR (400 MHz, CDCl_3) δ 7.36 (d, $J = 8.5$ Hz, 2H), 7.33 (d, $J = 8.4$ Hz, 2H), 7.00 (d, $J = 8.4$ Hz, 2H), 6.93 (d, $J = 8.7$ Hz, 2H), 6.81 (t, $J = 2.2$ Hz, 2H), 6.20 (t, $J = 2.2$ Hz, 2H), 5.22 (d, $J = 7.8$ Hz, 1H), 5.06 (d, $J = 7.6$ Hz, 1H), 2.51 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 138.70, 136.97, 131.76, 131.58, 129.17 (2C), 128.63 (2C), 122.28 (2C), 122.16 (2C), 120.47 (2C), 109.24 (2C), 75.55, 69.55.

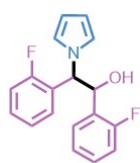


1,2-bis(4-trifluoromethylphenyl)-2-(1H-pyrrol-1-yl) ethanol (7f): yellow solid, dr: 33: 67. ^1H NMR (400 MHz, CDCl_3) δ 7.59 (d, $J = 8.2$ Hz, 4H), 7.43 (d, $J = 8.2$ Hz, 4H), 6.83 (t, $J = 2.2$ Hz, 2H), 6.22 (t, $J =$

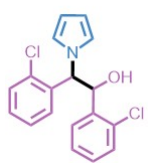
2.2 Hz, 2H), 5.41 (d, $J = 7.3$ Hz, 1H), 5.21 (d, $J = 7.3$ Hz, 1H), 2.61 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.48, 141.78, 130.59 (q, $J_{\text{CF}_3} = 32.6$ Hz), 130.48 (q, $J_{\text{CF}_3} = 32.6$ Hz), 127.81, 127.21, 125.61 (q, $J_{\text{CF}_3} = 3.92$ Hz), 125.42 (q, $J_{\text{CF}_3} = 3.91$ Hz), 123.93 (q, $J_{\text{CF}} = 273.3$ Hz), 123.82 (q, $J_{\text{CF}} = 273.3$ Hz), 120.65, 109.43, 75.58, 69.53. ^{19}F NMR (376 MHz, CDCl_3) δ -62.58, -62.70.



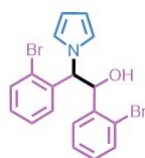
1,2-bis(4-nitrophenyl)-2-(1H-pyrrol-1-yl) ethanol (7g): black brown solid, dr: 67:33. ^1H NMR (400 MHz, CDCl_3) δ 8.12 (dd, $J = 8.9, 2.3$ Hz, 5H), 7.38 (d, $J = 8.6$ Hz, 3H), 6.83 (t, $J = 2.2$ Hz, 2H), 6.22 (t, $J = 2.2$ Hz, 2H), 5.57 (d, $J = 6.4$ Hz, 1H), 5.31 (d, $J = 6.4$ Hz, 1H), 2.68 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 147.83, 146.53, 144.84, 143.90, 128.31, 127.58, 123.94, 123.69, 120.78, 109.80, 75.22, 68.96.



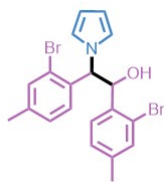
1,2-bis(2-fluorophenyl)-2-(1H-pyrrol-1-yl) ethanol (7h): yellow oil, dr: 40: 60. ^1H NMR (400 MHz, CDCl_3) δ 7.68 (td, $J = 7.5, 1.8$ Hz, 1H), 7.31 – 7.22 (m, 3H), 7.09 – 7.01 (m, 3H), 6.89 (d, $J = 9.0$ Hz, 1H), 6.73 (t, $J = 2.2$ Hz, 2H), 6.04 (t, $J = 2.2$ Hz, 2H), 5.74 – 5.71 (m, 2H), 2.37 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 160.86 (d, $J = 247.1$ Hz), 160.17 (d, $J = 246.2$ Hz), 129.98 (d, $J = 8.2$ Hz), 129.81 (d, $J = 8.7$ Hz), 129.52 (d, $J = 3.5$ Hz), 128.00 (d, $J = 3.9$ Hz), 127.48 (d, $J = 12.6$ Hz), 124.50 (d, $J = 13.4$ Hz), 124.35 (d, $J = 3.0$ Hz), 124.27 (d, $J = 3.5$ Hz), 120.24, 115.76 (d, $J = 23.0$ Hz), 115.35 (d, $J = 21.7$ Hz), 108.31, 70.12, 60.23. ^{19}F NMR (376 MHz, CDCl_3) δ -117.41 (dt, $J = 12.0, 6.4$ Hz), -118.45 (dt, $J = 11.5, 6.2$ Hz).



1,2-bis(2-chlorophenyl)-2-(1H-pyrrol-1-yl) ethanol (7i): yellow oil, dr: 54: 46. ^1H NMR (400 MHz, CDCl_3) δ 7.81 (dd, $J = 7.8, 1.7$ Hz, 1H), 7.18 (ddd, $J = 10.6, 7.7, 1.9$ Hz, 6H), 6.79 (t, $J = 2.2$ Hz, 2H), 6.15 (t, $J = 2.2$ Hz, 2H), 5.99 (d, $J = 6.3$ Hz, 1H), 5.89 (t, $J = 5.4$ Hz, 1H), 2.43 (d, $J = 4.7$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 137.35, 135.58, 133.97, 132.47, 130.16, 129.90, 129.62, 129.50, 129.37, 129.21, 128.37, 126.93, 121.00, 108.54, 71.84, 63.51.

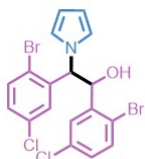


1,2-bis(2-bromophenyl)-2-(1H-pyrrol-1-yl) ethanol (7j): yellow oil, dr: 55: 45. ^1H NMR (400 MHz, CDCl_3) δ 7.90 (d, $J = 1.7$ Hz, 1H), 7.51 (d, $J = 3.5$ Hz, 1H), 7.36 – 7.31 (m, 2H), 7.25 (d, $J = 7.2$ Hz, 1H), 7.11 – 7.05 (m, 3H), 6.74 (t, $J = 2.2$ Hz, 2H), 6.14 (t, $J = 2.2$ Hz, 2H), 5.95 (d, $J = 6.3$ Hz, 1H), 5.84 (t, $J = 5.3$ Hz, 1H), 2.48 (d, $J = 5.1$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.00, 137.08, 133.33, 132.76, 130.57, 129.64, 129.53, 128.61, 127.74, 127.71, 126.02, 125.05, 120.97, 108.57, 74.27, 66.08.



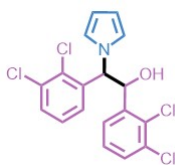
1,2-bis(2-bromo-4-methylphenyl)-2-(1H-pyrrol-1-yl) ethanol (7k):

yellow oil, dr: 54: 46. ^1H NMR (400 MHz, CDCl_3) δ 7.72 (d, $J = 3.8$ Hz, 1H), 7.36 – 7.27 (m, 4H), 7.19 (d, $J = 8.0$ Hz, 1H), 6.83 (t, $J = 2.2$ Hz, 2H), 6.08 (t, $J = 2.2$ Hz, 2H), 6.03 (d, $J = 5.4$ Hz, 1H), 5.82 (d, $J = 4.8$ Hz, 1H), 2.33 (d, $J = 5.0$ Hz, 1H), 2.28 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.92, 139.79, 135.94, 133.71, 133.38, 133.13, 129.87, 129.05, 128.61, 128.19, 125.84, 124.84, 120.47, 108.11, 74.83, 63.90, 20.78, 20.73.



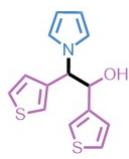
1,2-bis(2-bromo-5-chlorophenyl)-2-(1H-pyrrol-1-yl) ethanol (7l):

yellow oil, dr: 63: 37. ^1H NMR (400 MHz, CDCl_3) δ 7.85 (d, $J = 2.8$ Hz, 1H), 7.44 (d, $J = 5.3$ Hz, 1H), 7.37 (d, $J = 8.5$ Hz, 1H), 7.13 – 7.10 (m, 3H), 6.92 (t, $J = 2.3$ Hz, 2H), 6.17 (t, $J = 2.3$ Hz, 2H), 6.01 (d, $J = 4.2$ Hz, 1H), 5.81 (d, $J = 4.1$ Hz, 1H), 2.48 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.47, 136.67, 134.27, 133.91, 133.74, 133.71, 130.86, 129.89, 129.83, 128.79, 123.54, 120.56, 120.49, 108.77, 74.54, 63.30.



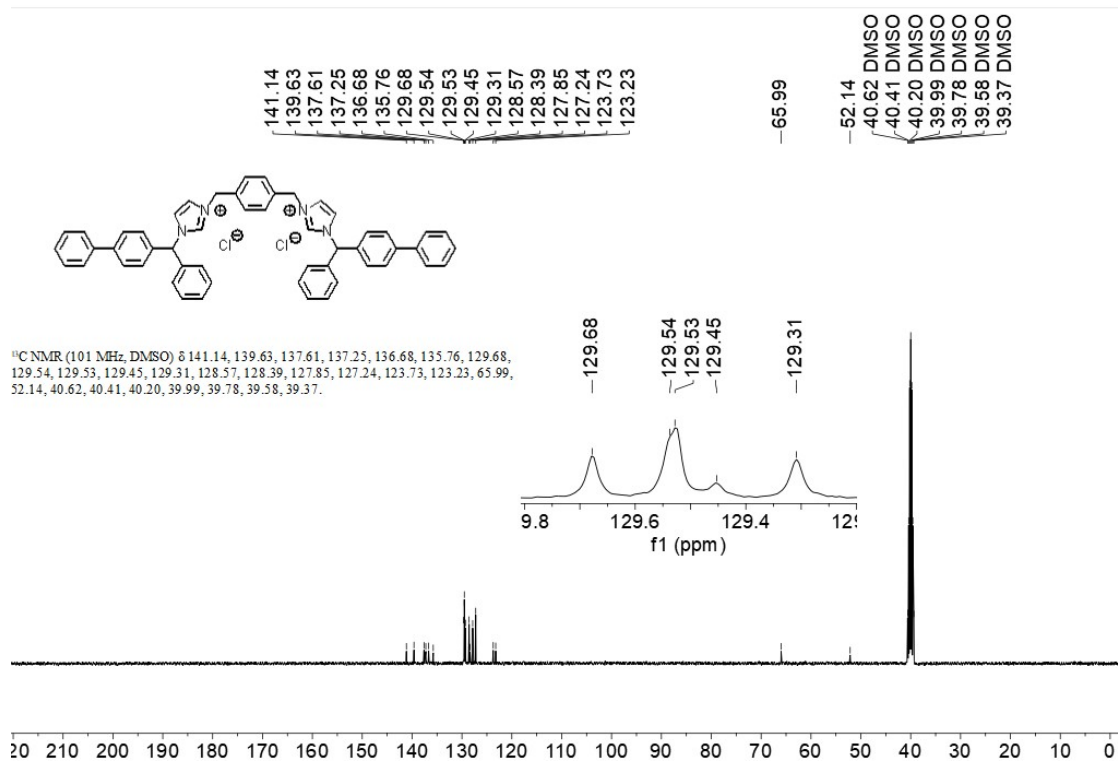
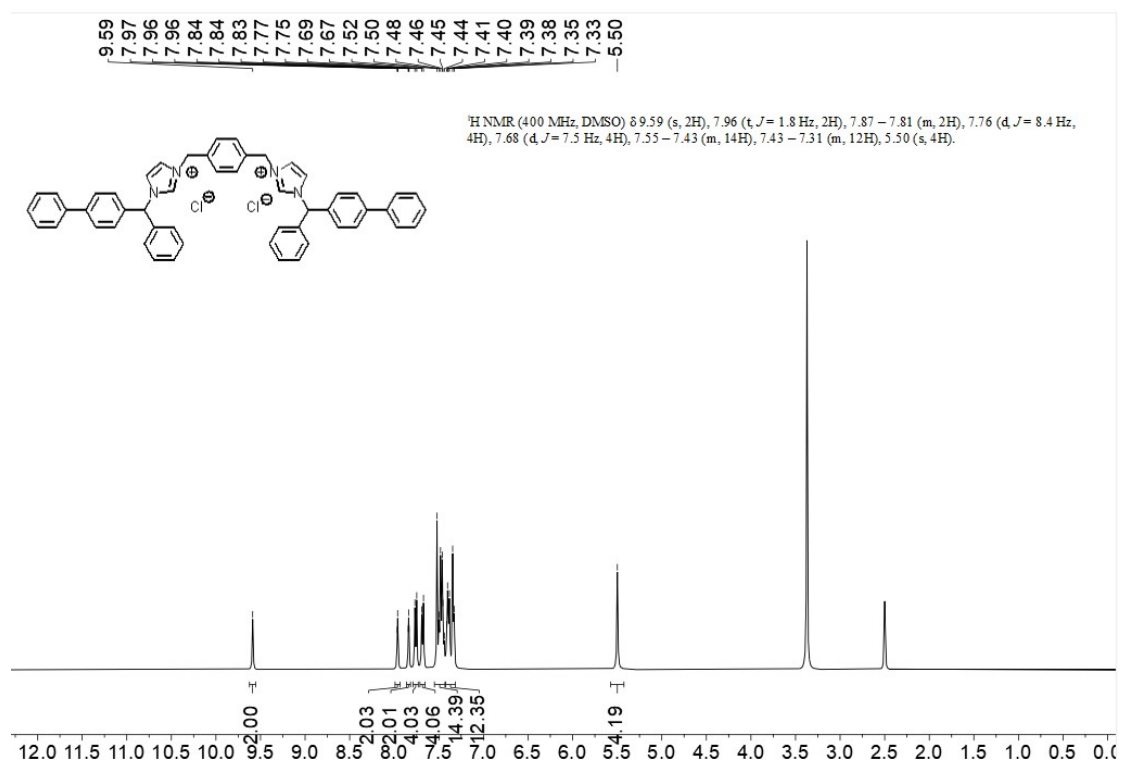
1,2-bis(2,3-dichlorophenyl)-2-(1H-pyrrol-1-yl) ethanol (7m):

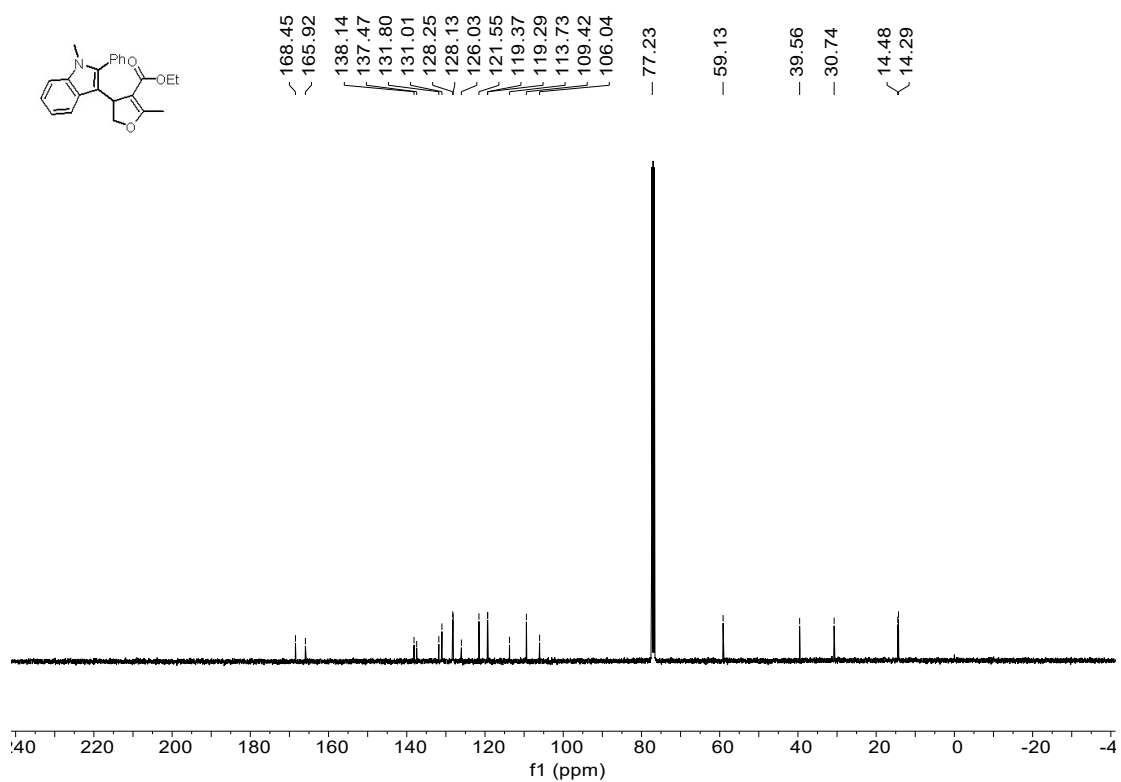
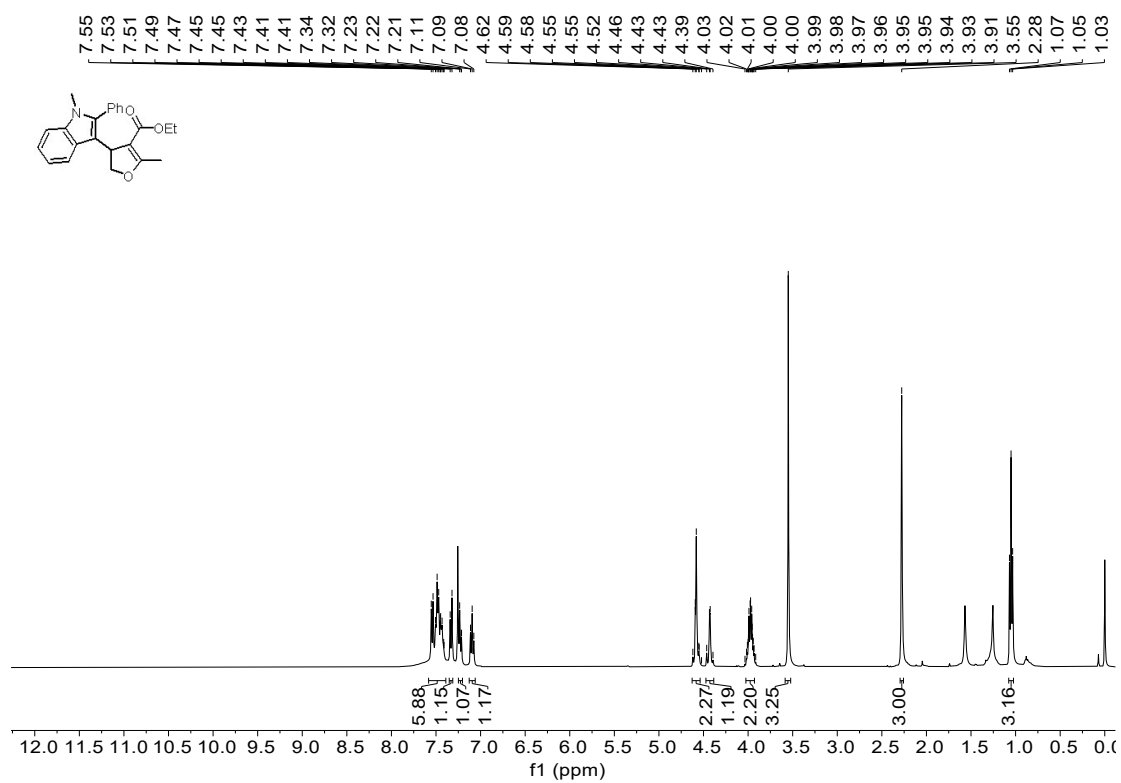
yellow oil, dr: 56: 44. ^1H NMR (400 MHz, CDCl_3) δ 7.78 – 7.74 (m, 1H), 7.44 – 7.39 (m, 2H), 7.22 (d, $J = 2.2$ Hz, 1H), 7.15 (t, $J = 7.8$ Hz, 1H), 7.09 (s, 1H), 6.86 (t, $J = 2.2$ Hz, 2H), 6.13 (t, $J = 2.2$ Hz, 3H), 5.97 (t, $J = 4.9$ Hz, 1H), 2.46 (d, $J = 4.3$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.63, 136.21, 133.25, 133.13, 130.48, 130.44, 130.24, 130.21, 128.45, 127.50, 126.99, 126.23, 120.56, 108.65, 73.14, 61.60.

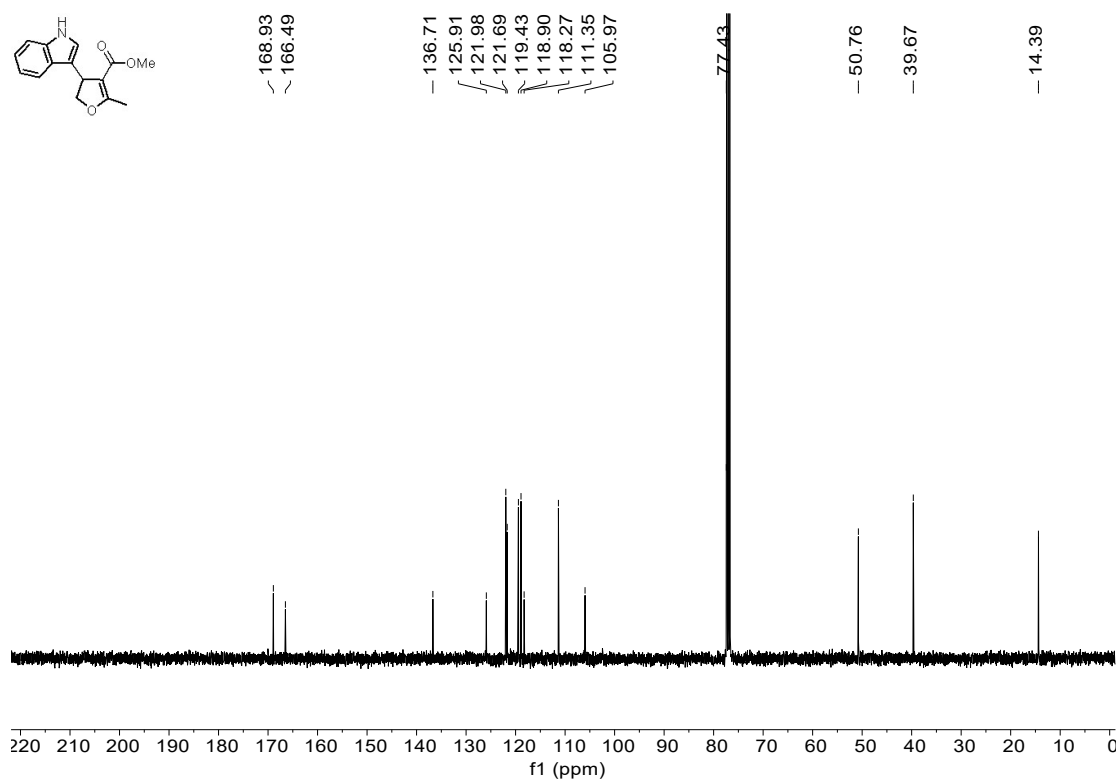
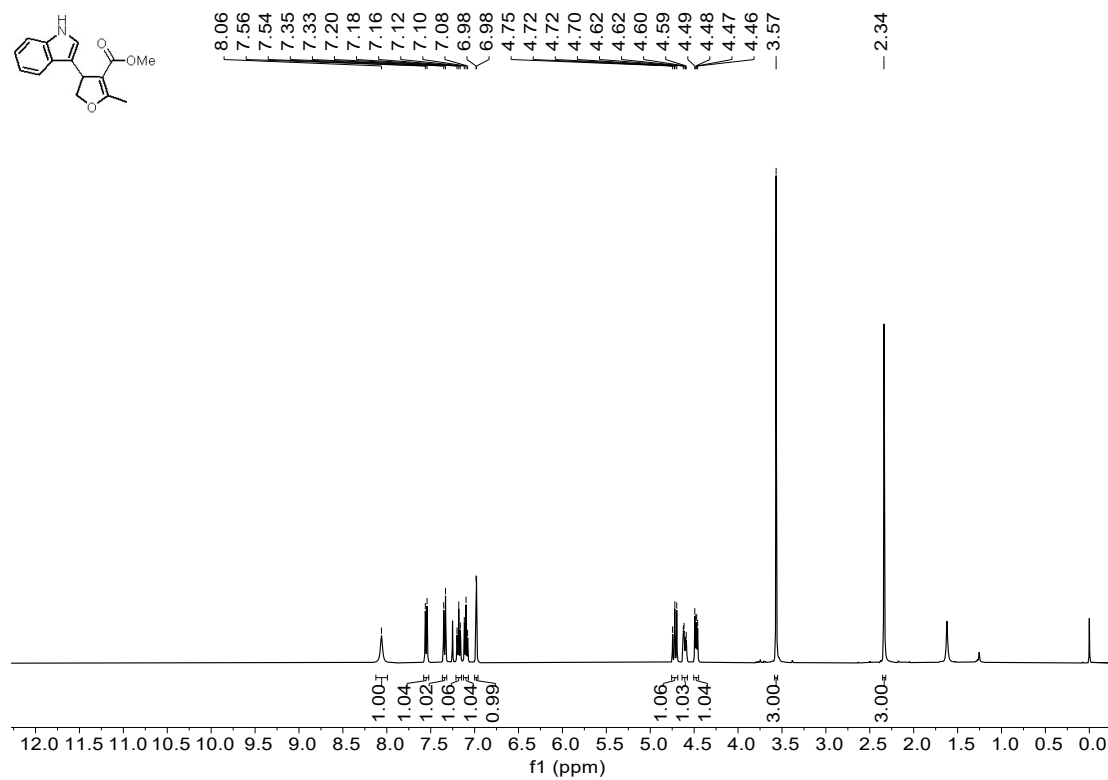


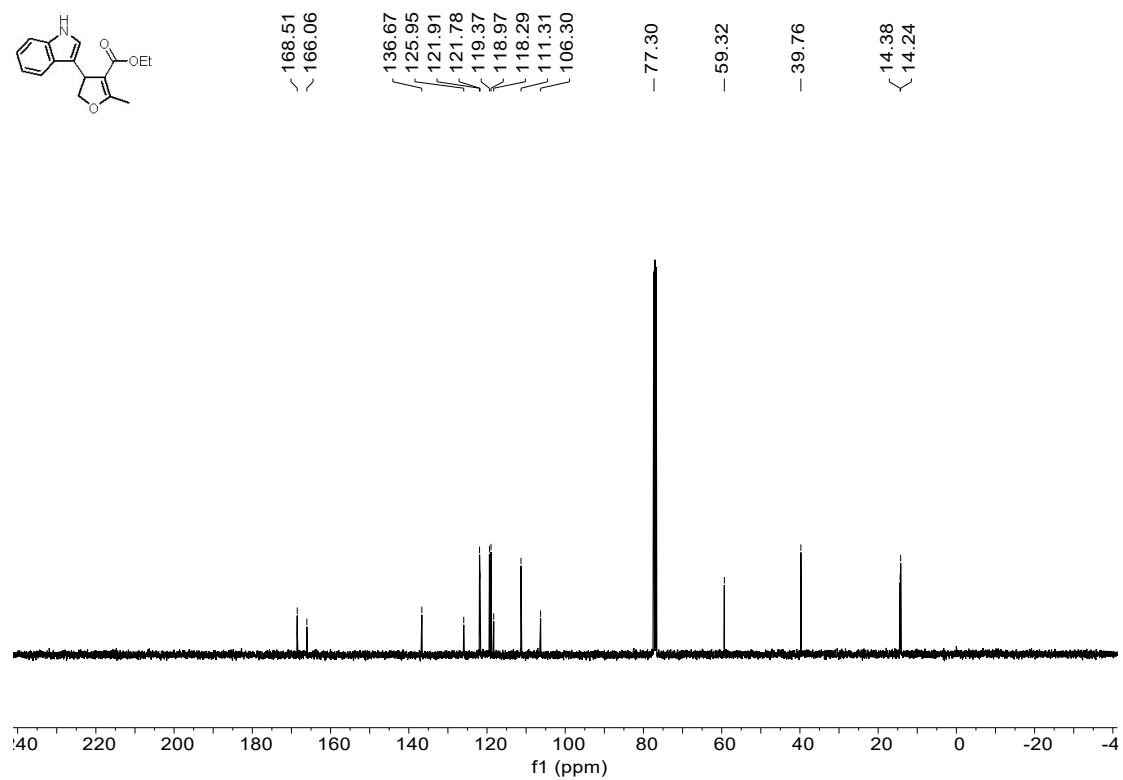
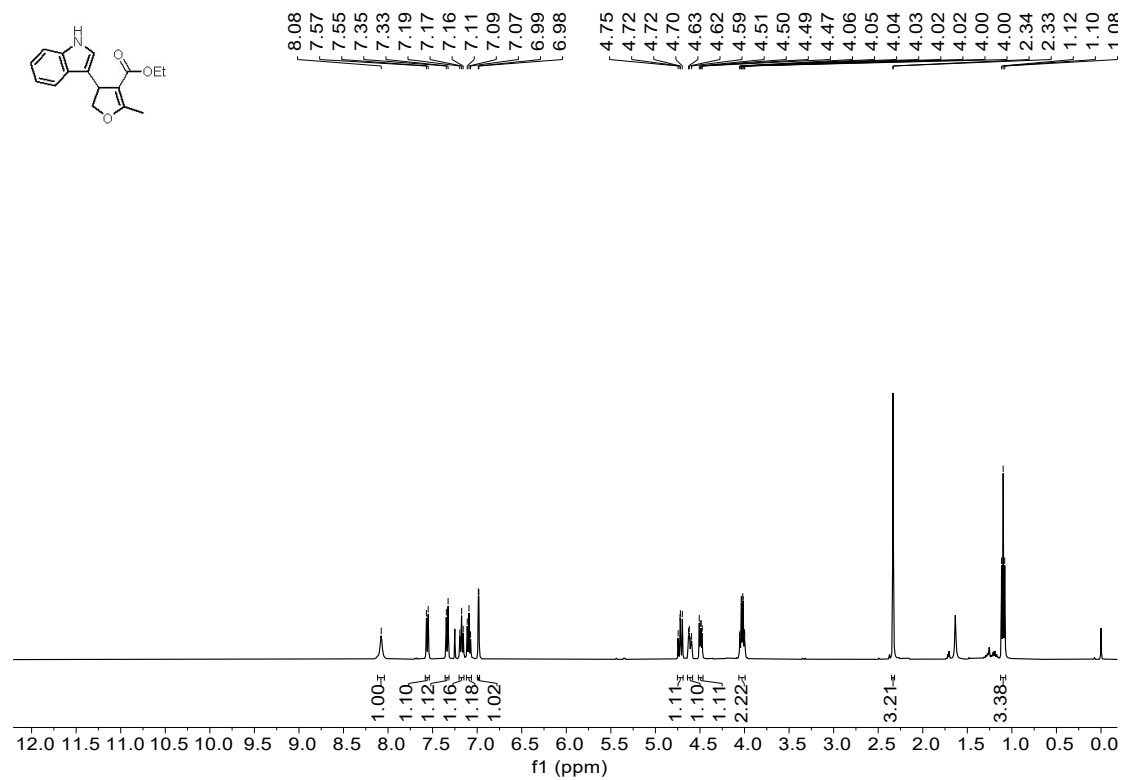
1,2-bis(thiophen-3-yl)-2-(1H-pyrrol-1-yl) ethanol (7n): yellow oil, dr:

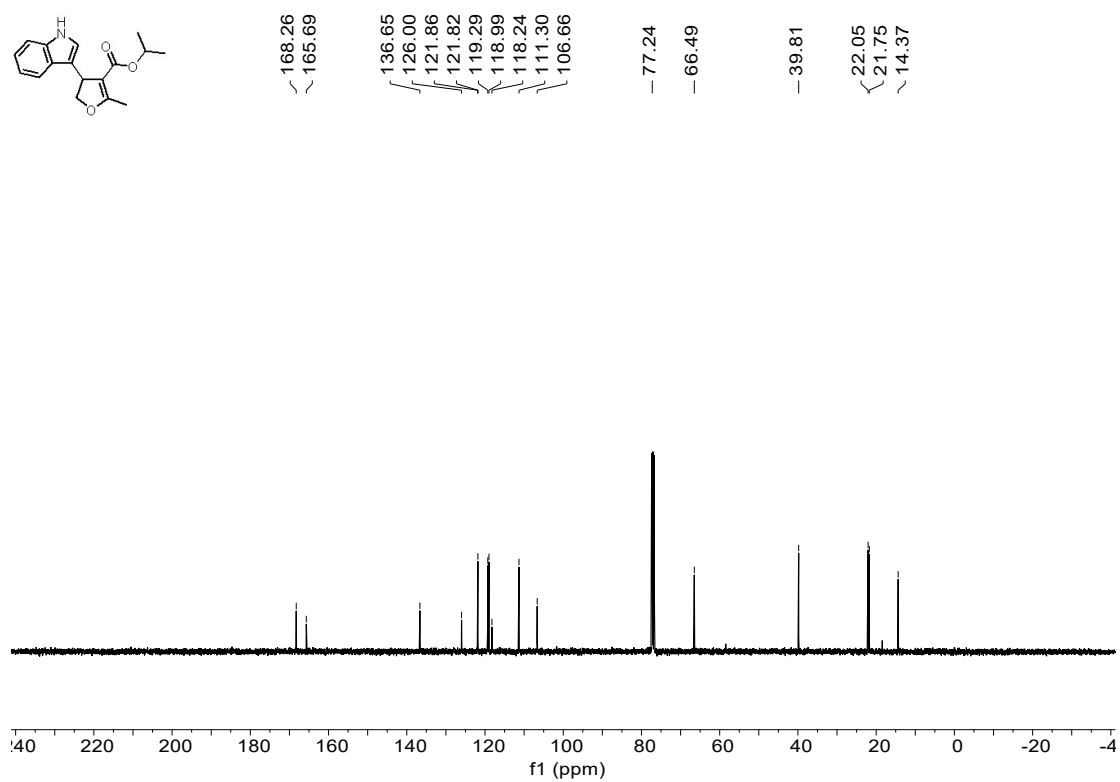
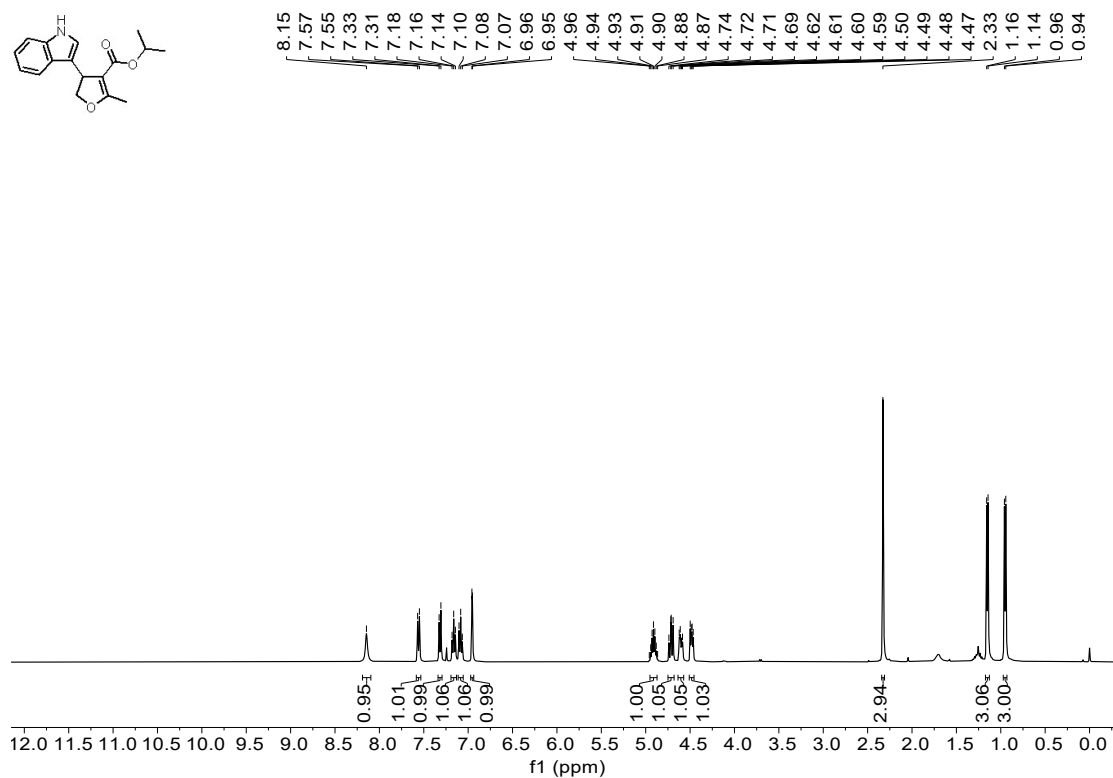
58: 42. ^1H NMR (400 MHz, CDCl_3) δ 7.31 – 7.25 (m, 2H), 7.23 (d, $J = 3.1$ Hz, 1H), 7.04 – 6.97 (m, 2H), 6.78 (d, $J = 5.1$ Hz, 1H), 6.65 (t, $J = 2.2$ Hz, 2H), 6.08 (t, $J = 2.2$ Hz, 2H), 5.37 (d, $J = 6.9$ Hz, 1H), 5.28 (t, $J = 7.6$ Hz, 1H), 2.33 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 141.99, 138.16, 127.62, 126.22, 126.03, 125.52, 124.08, 122.11, 120.25, 108.30, 73.29, 64.86.

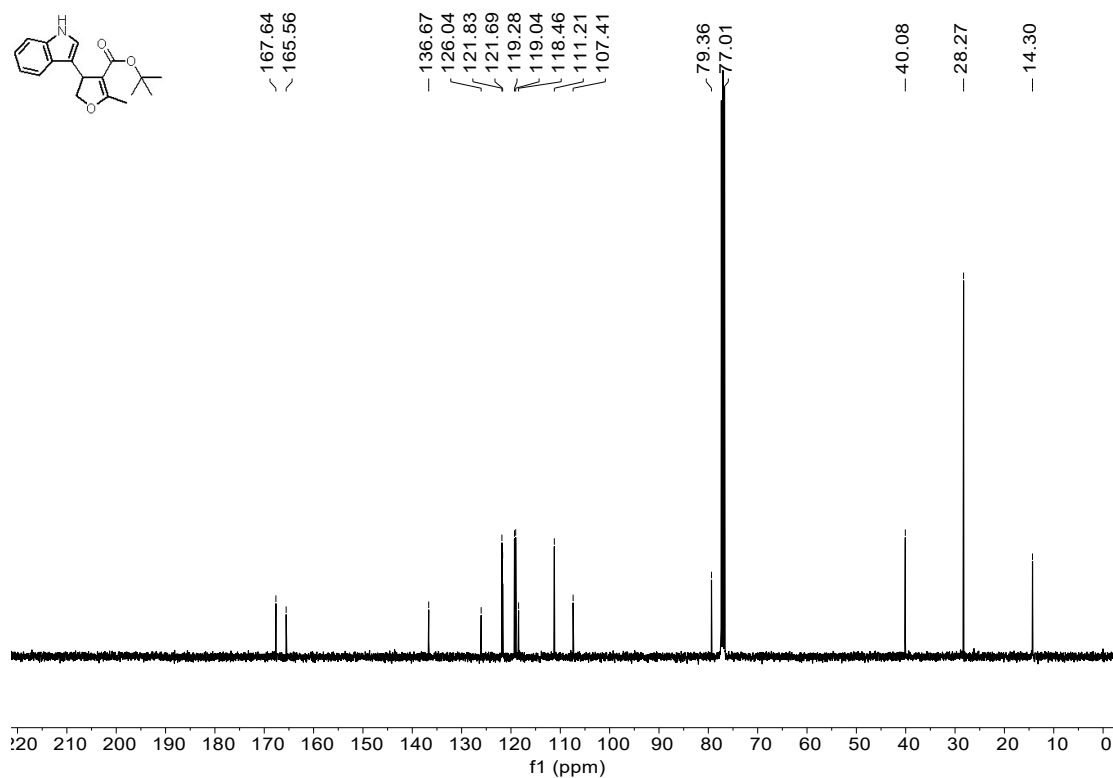
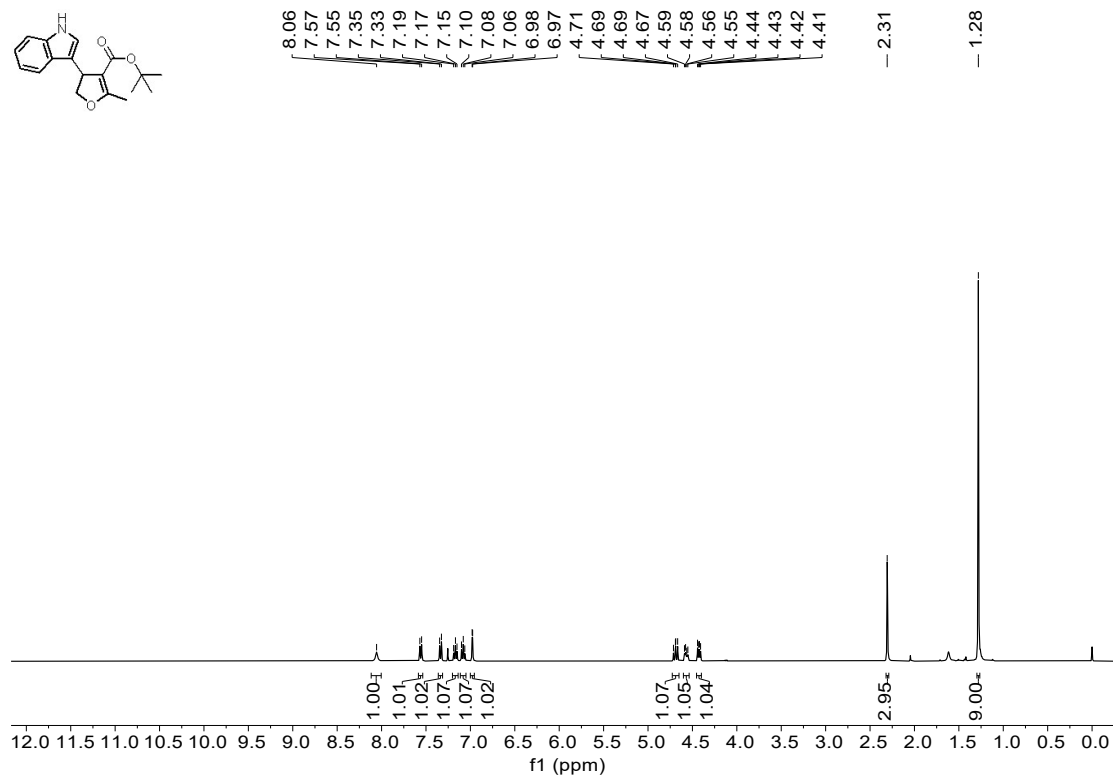


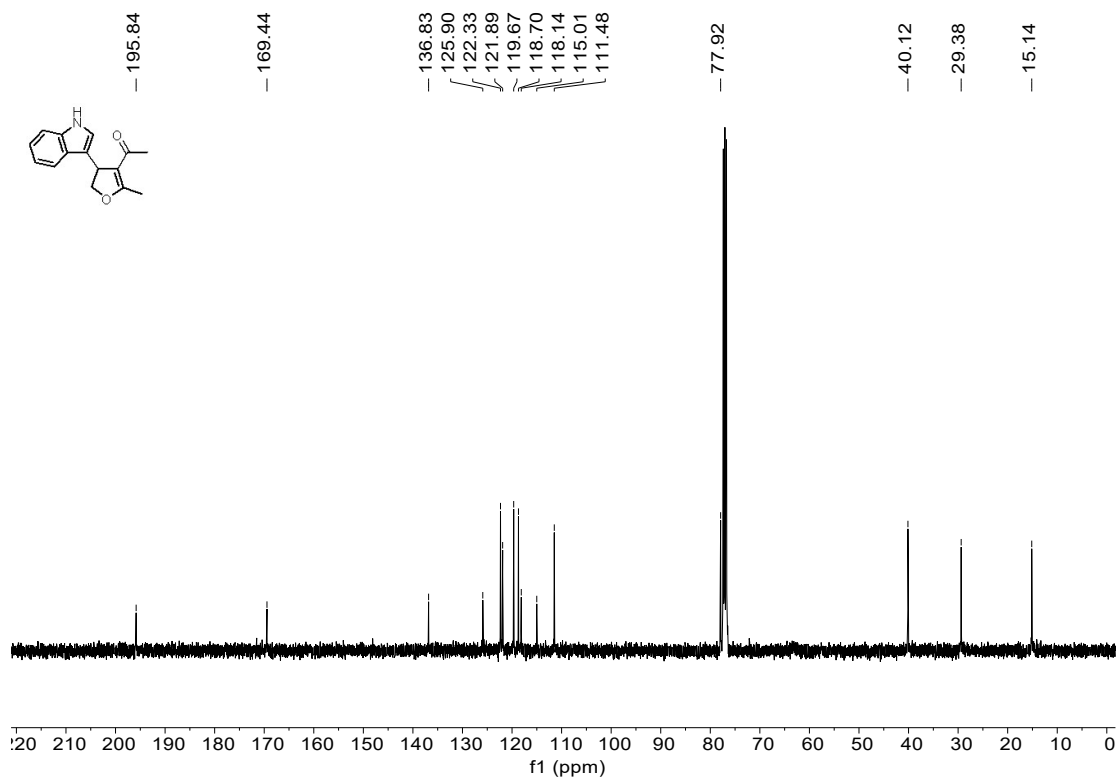
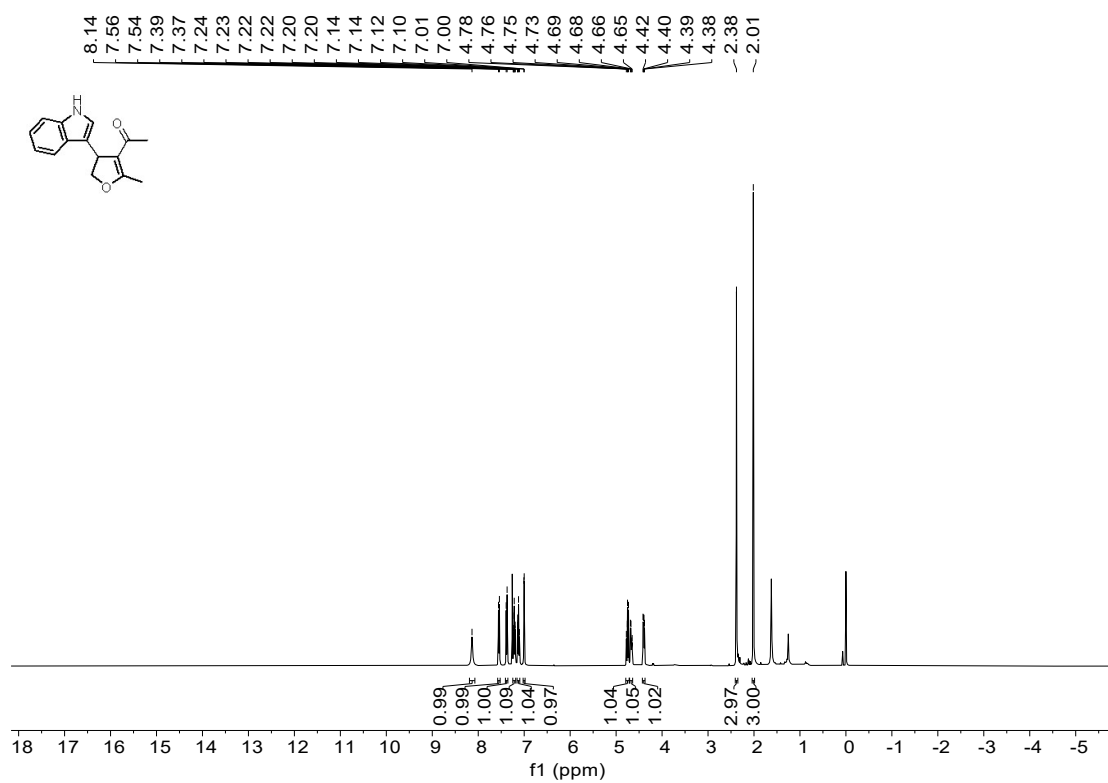


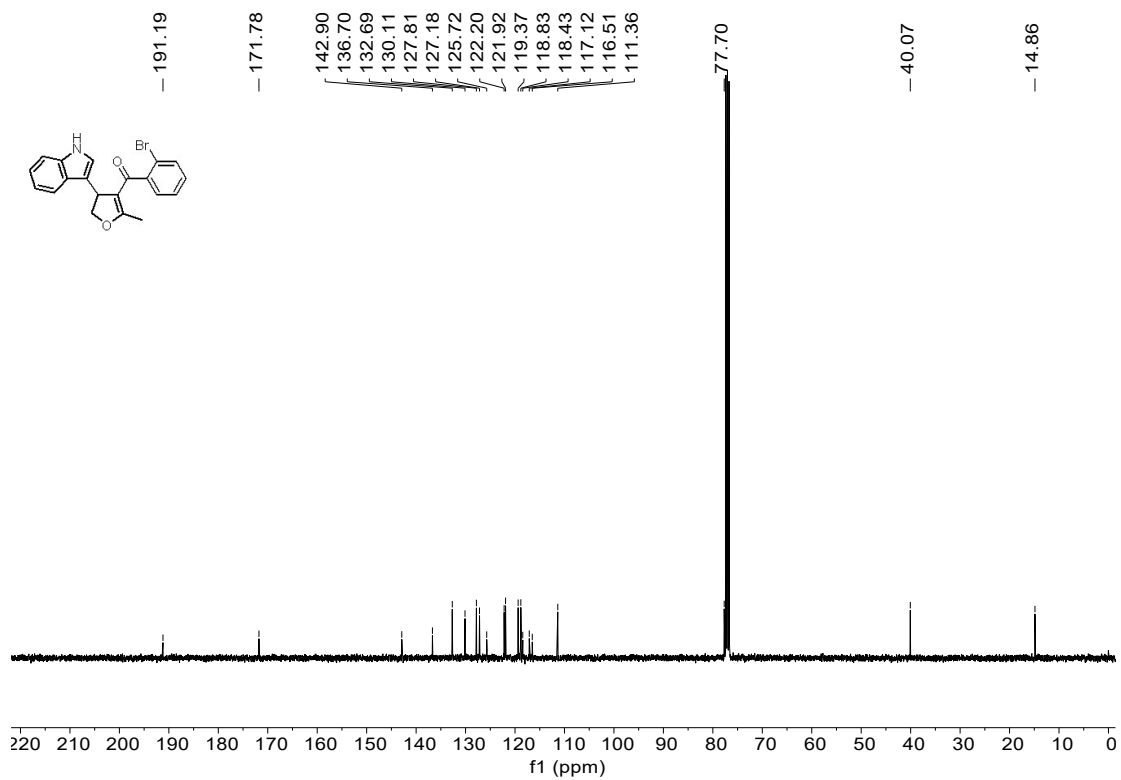
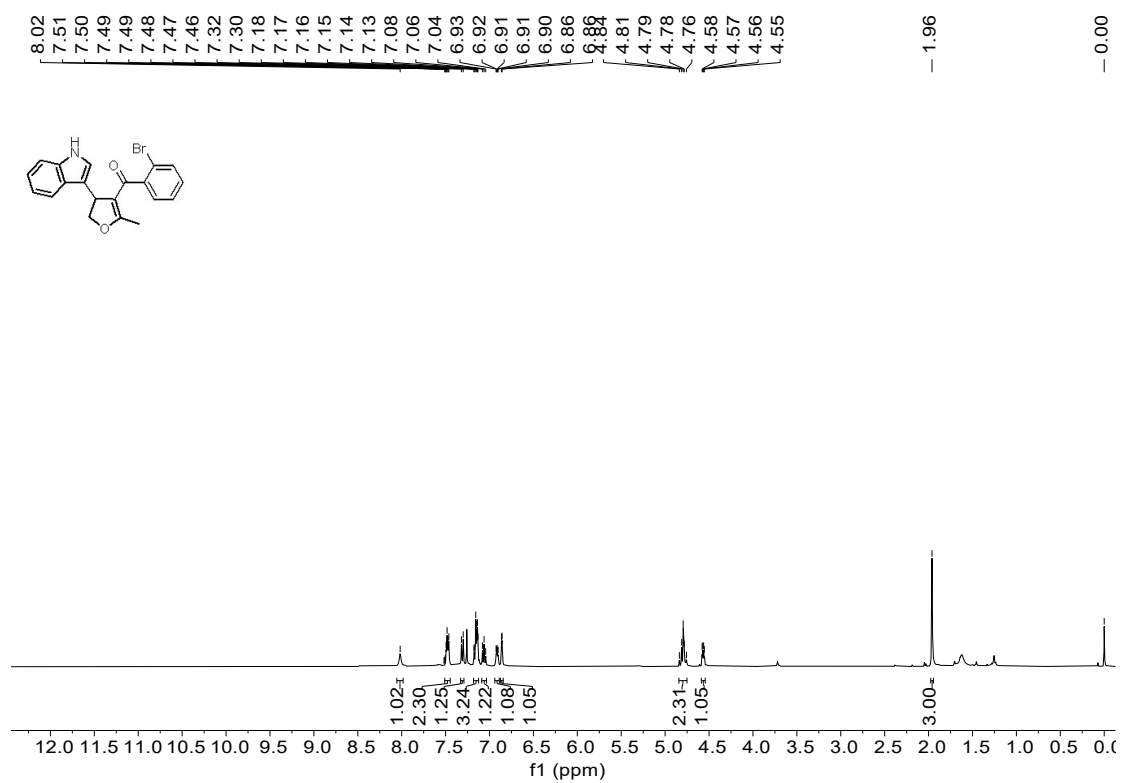


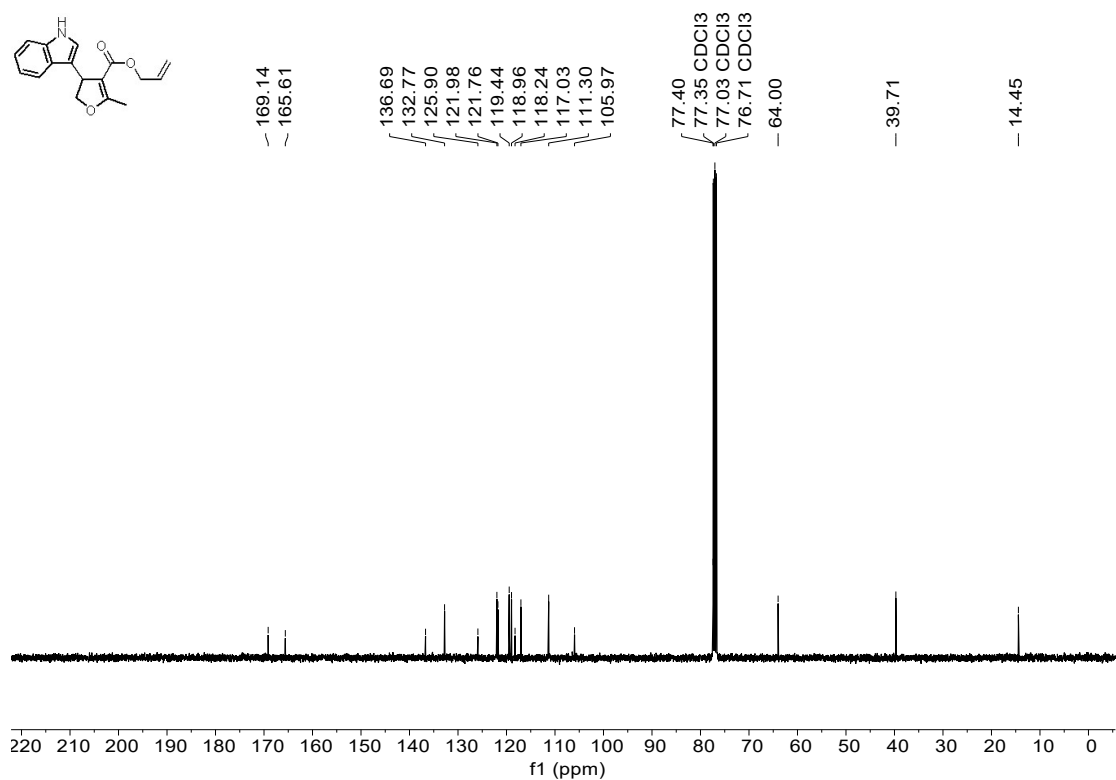
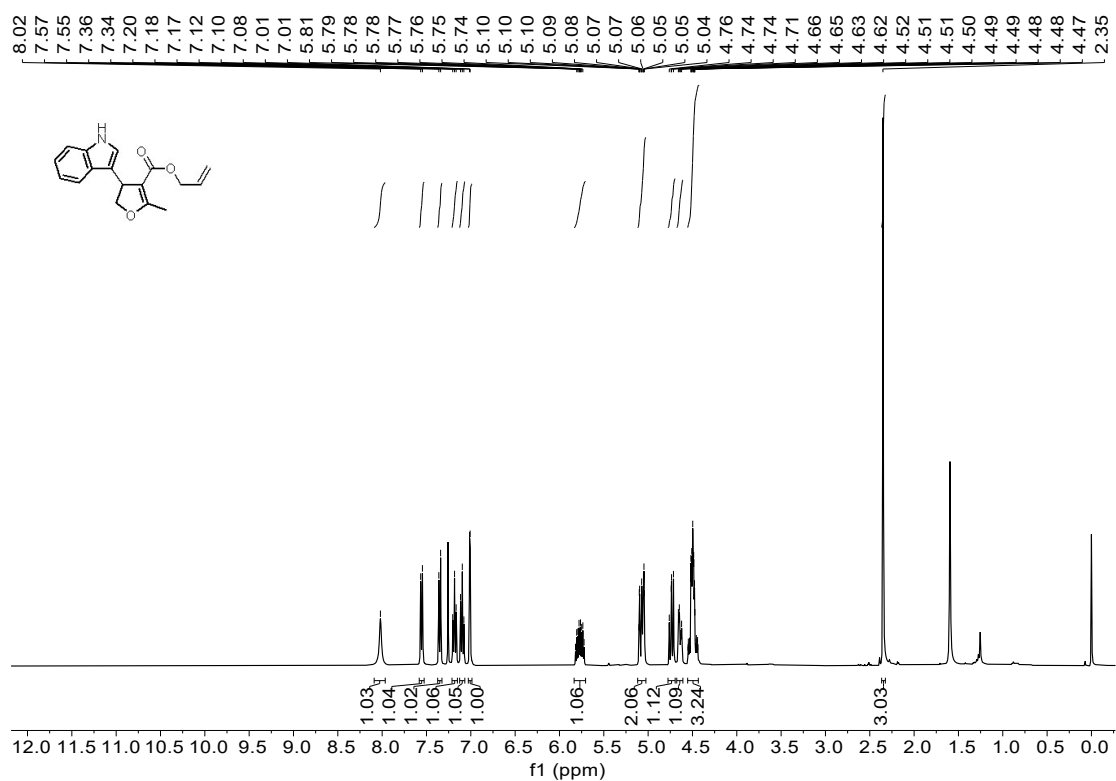


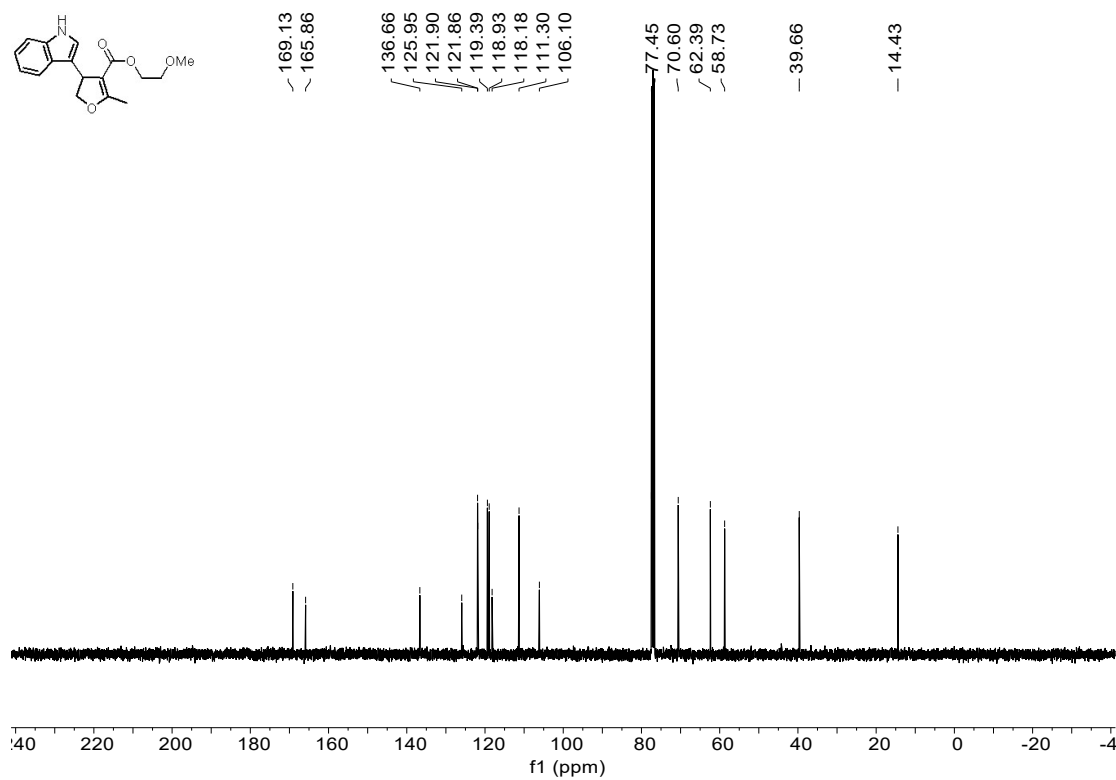
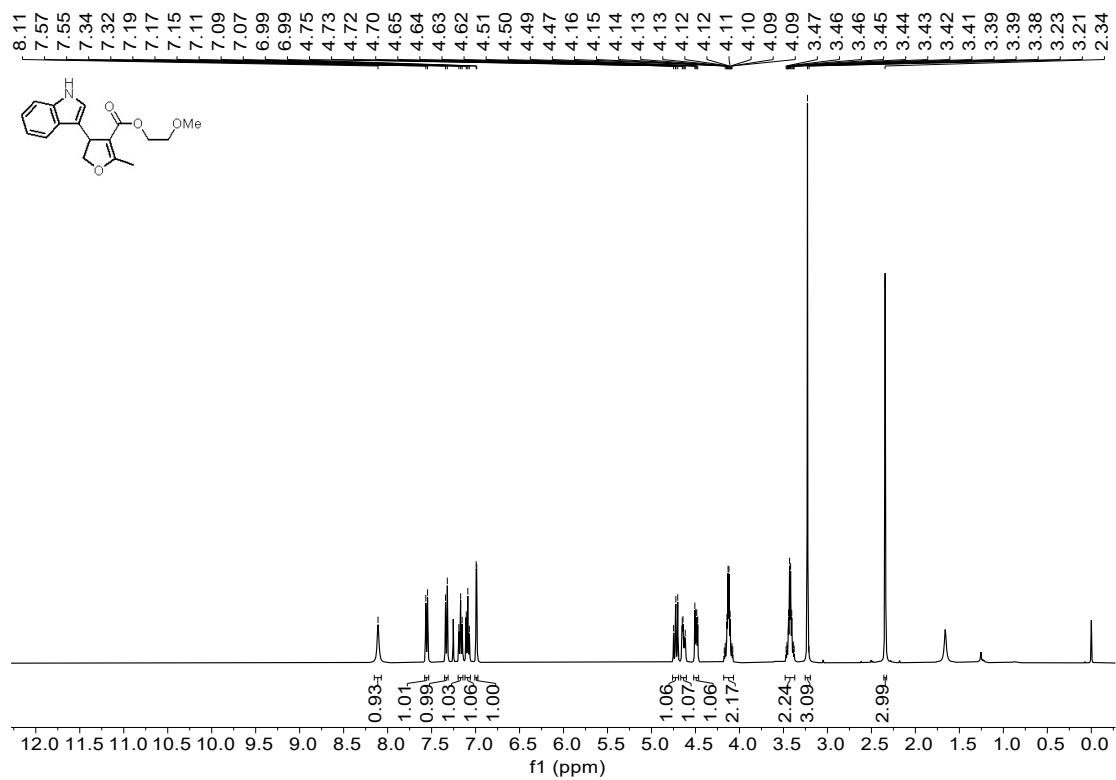


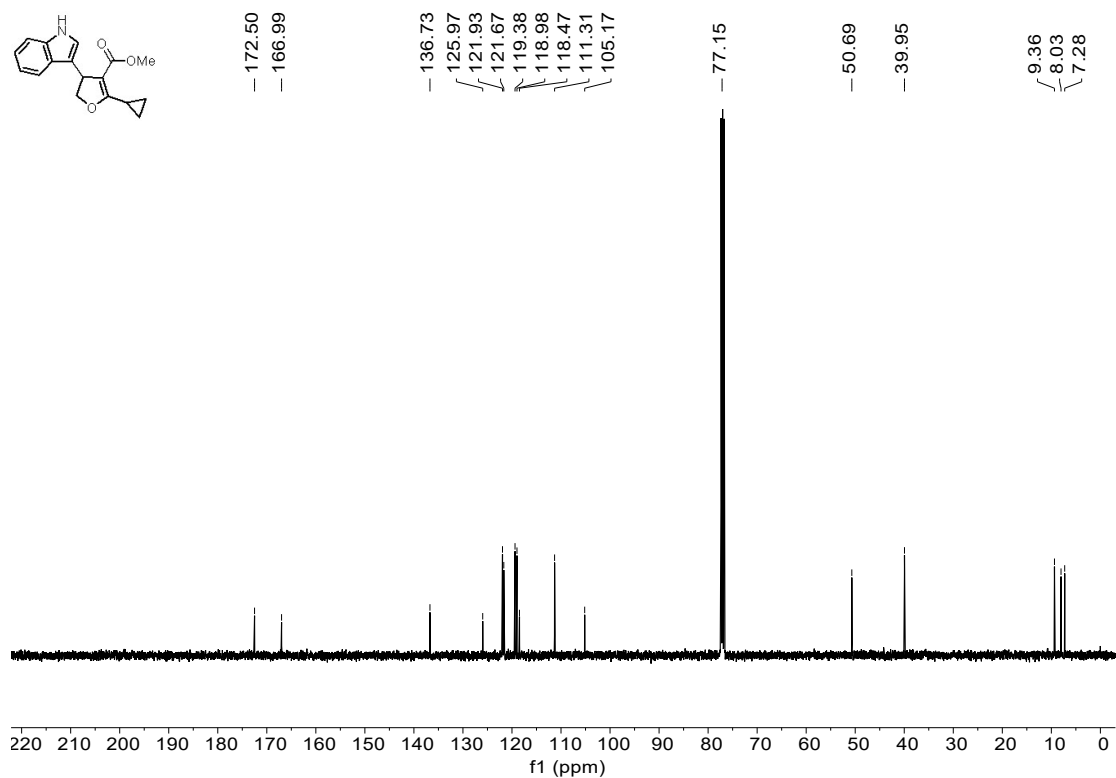
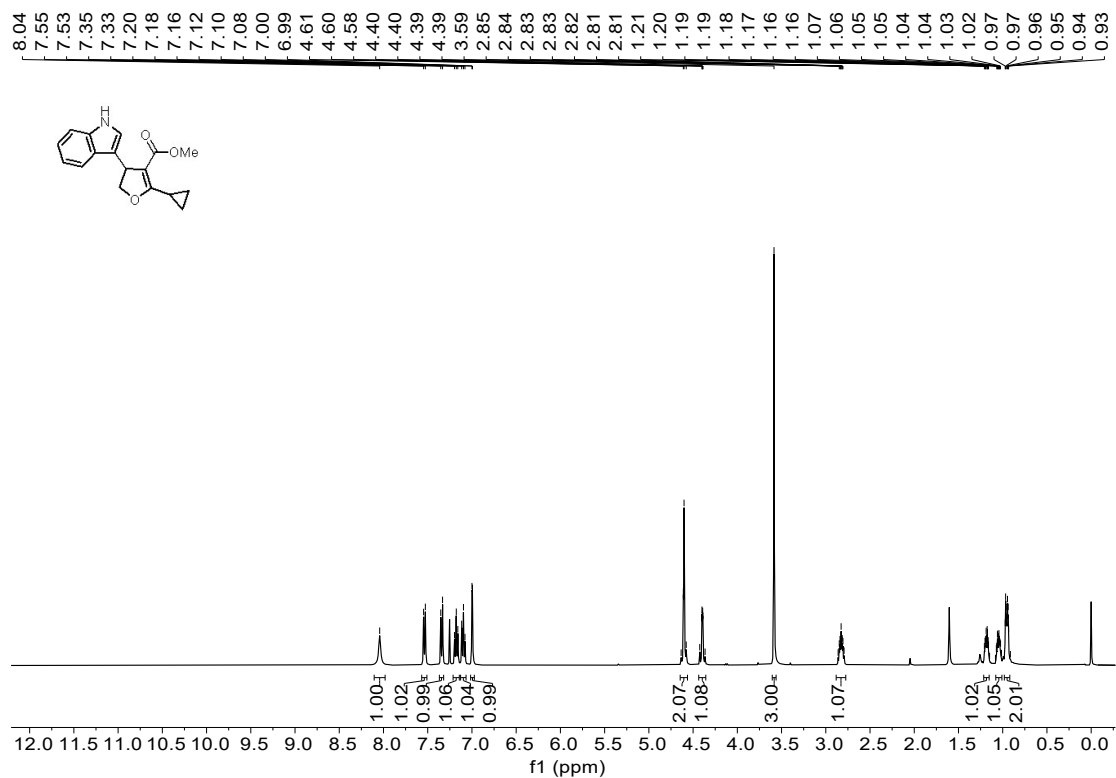


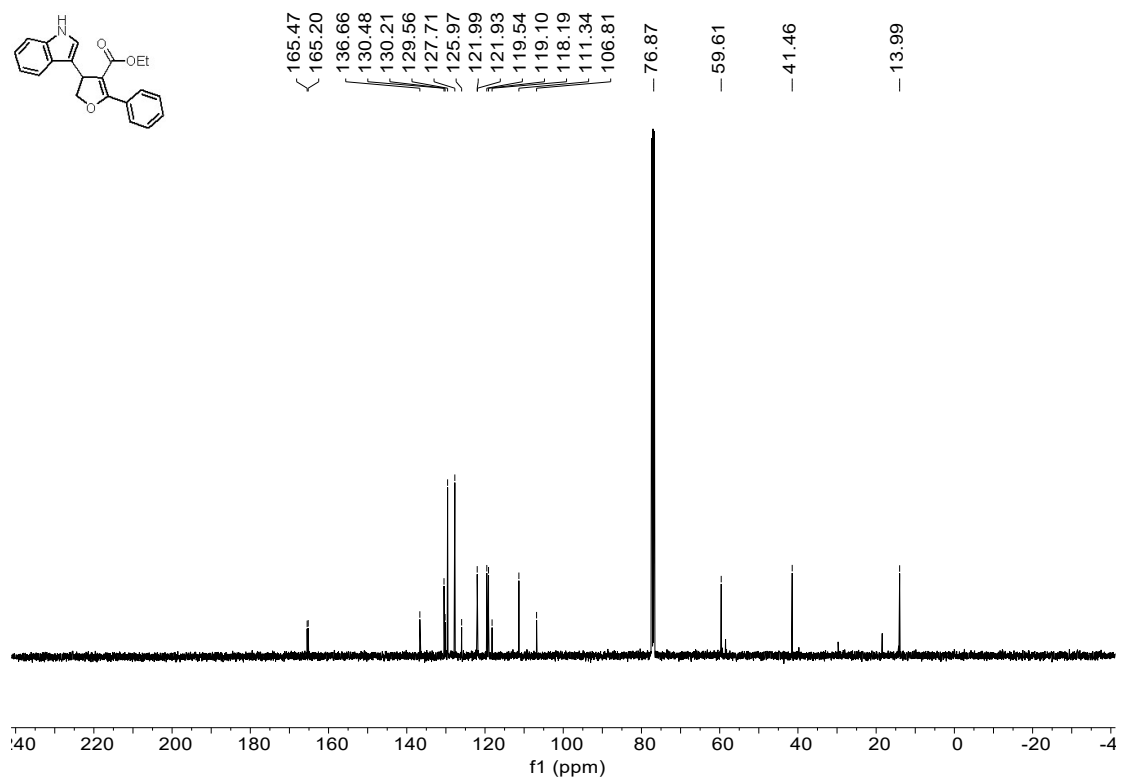
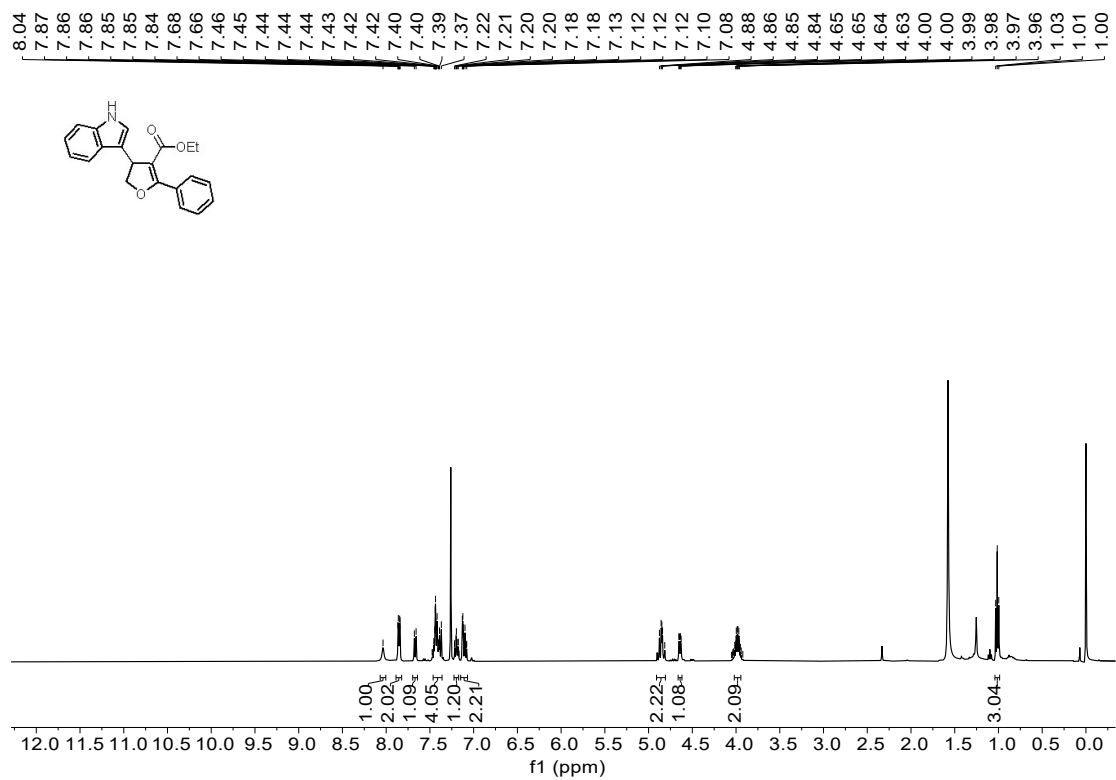


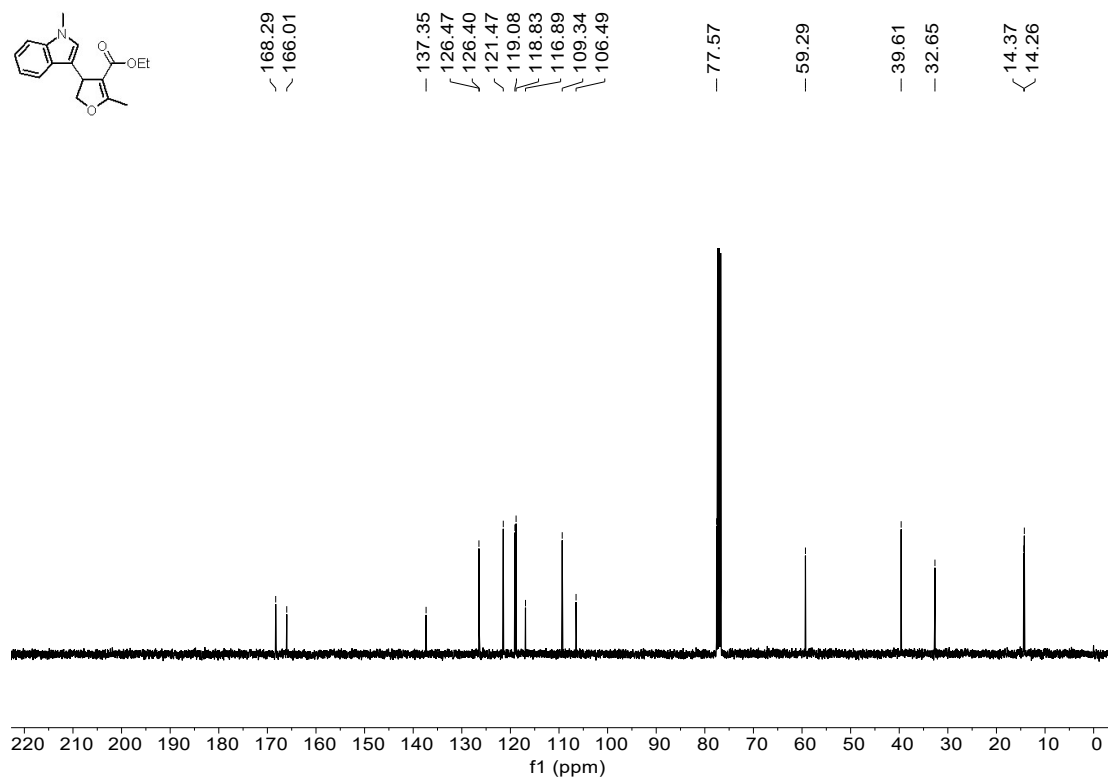
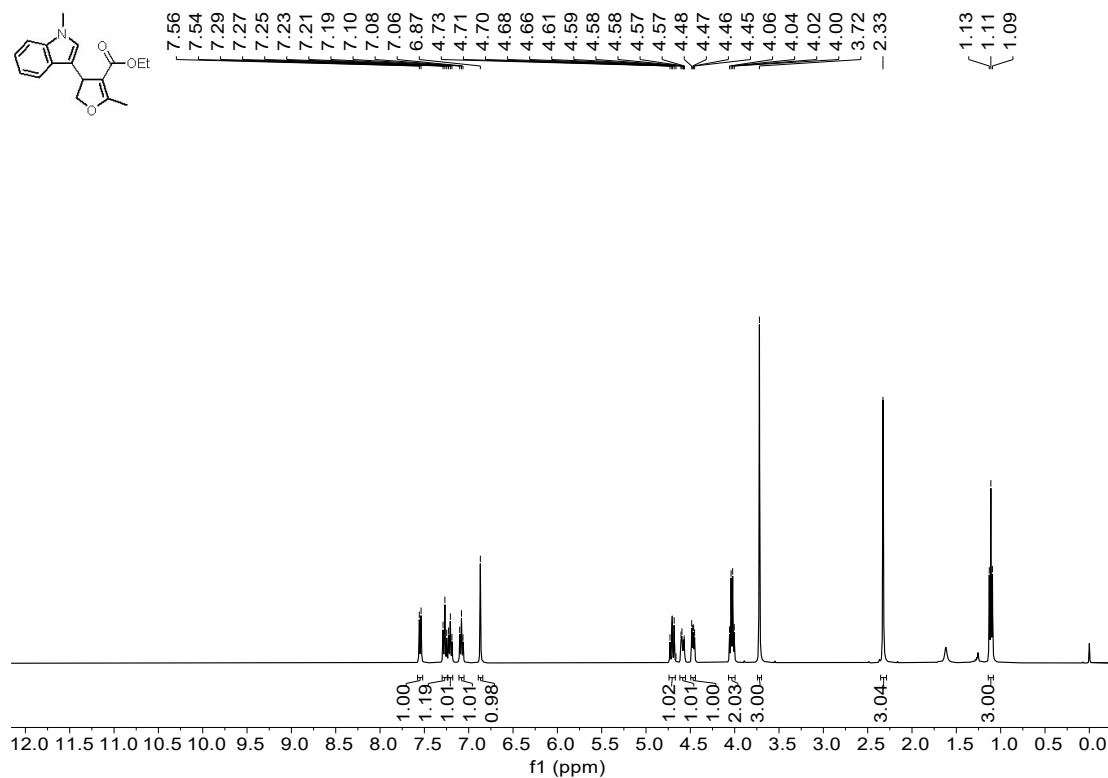


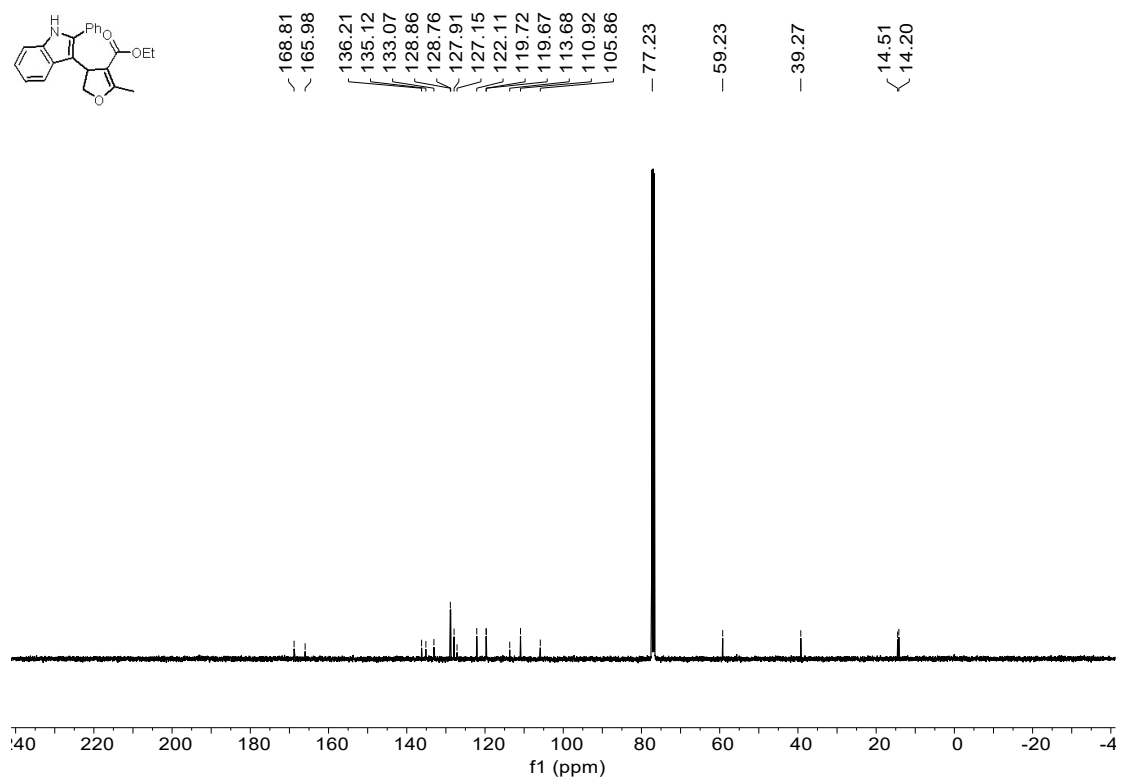
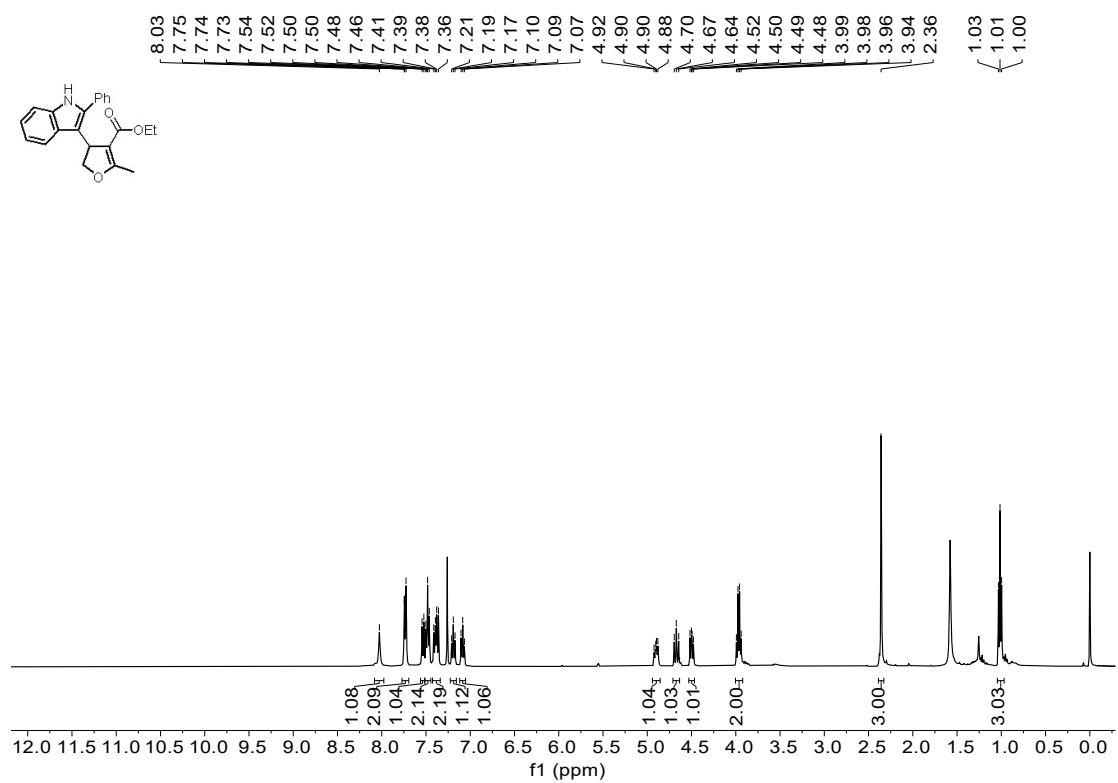


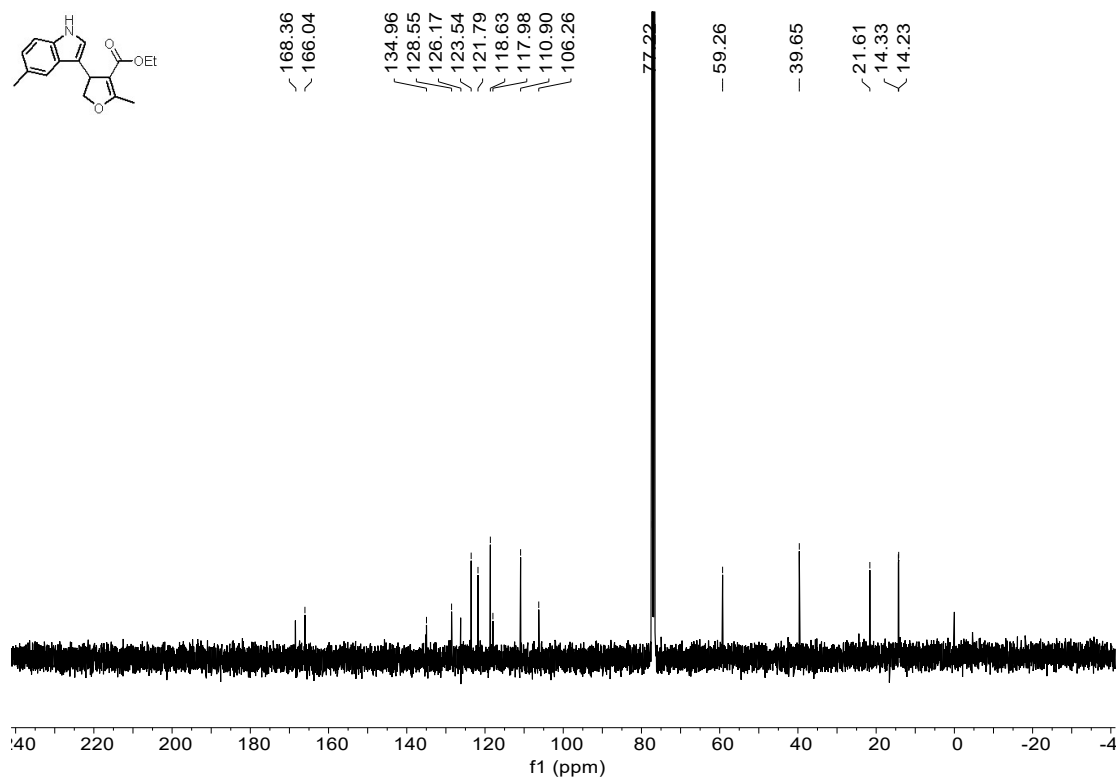
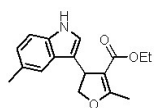
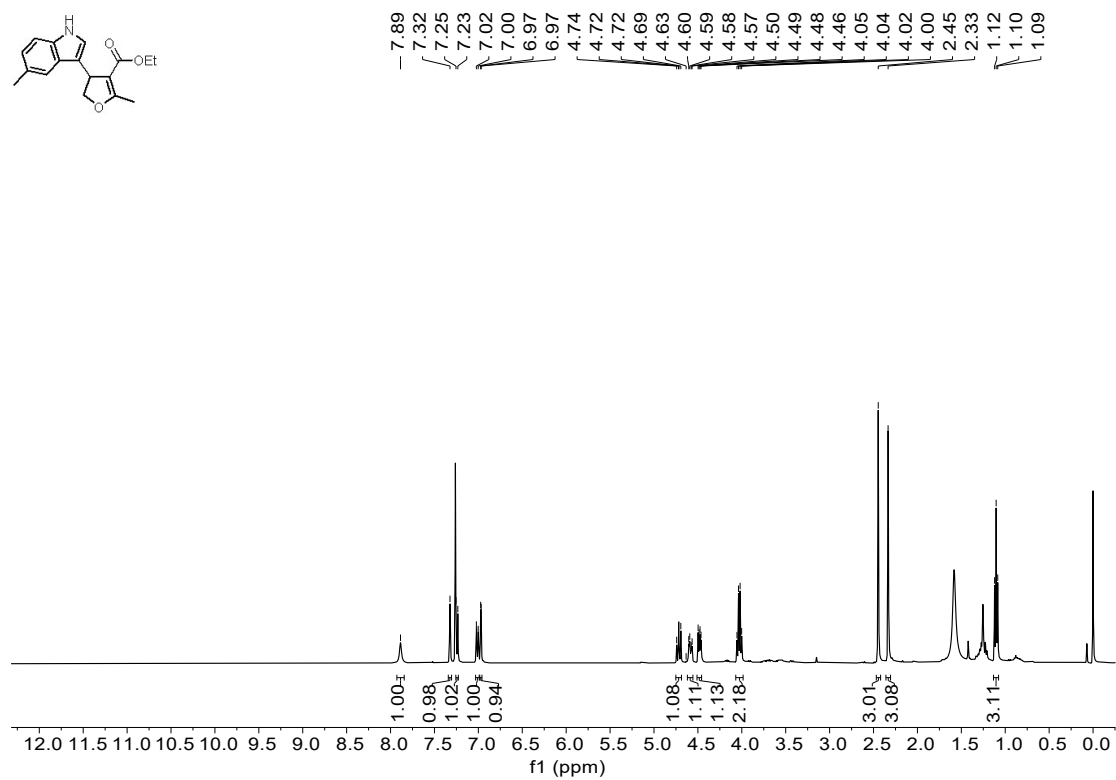
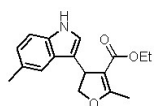


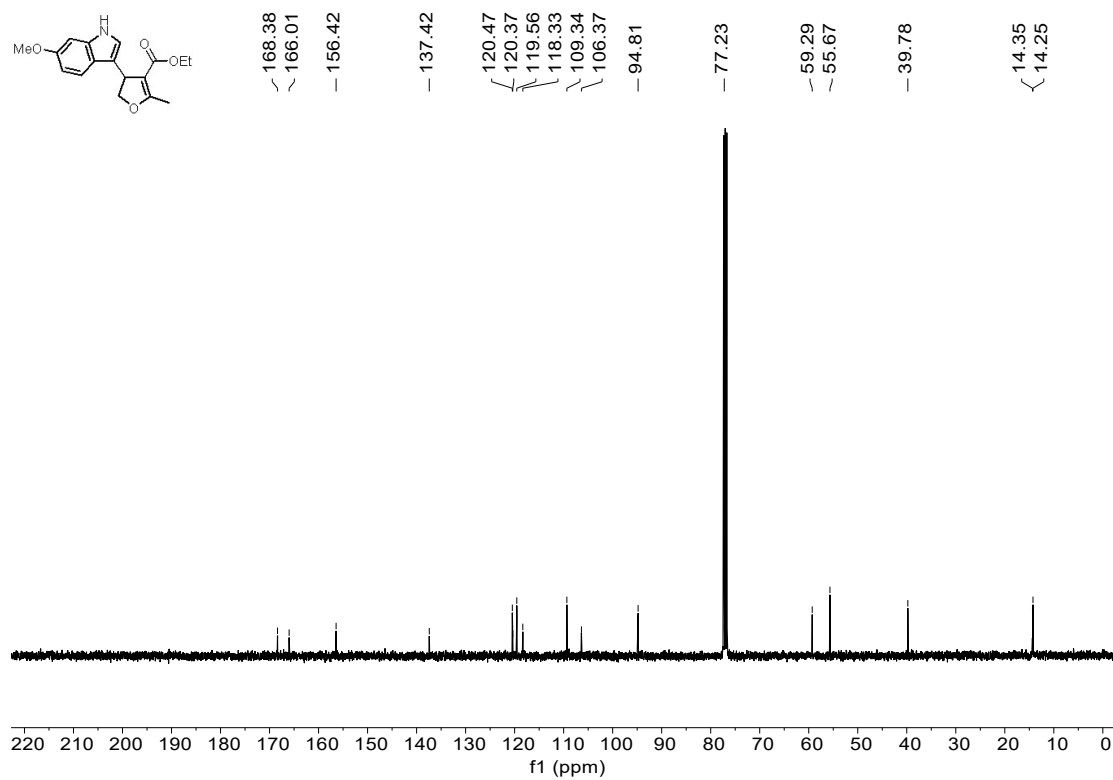
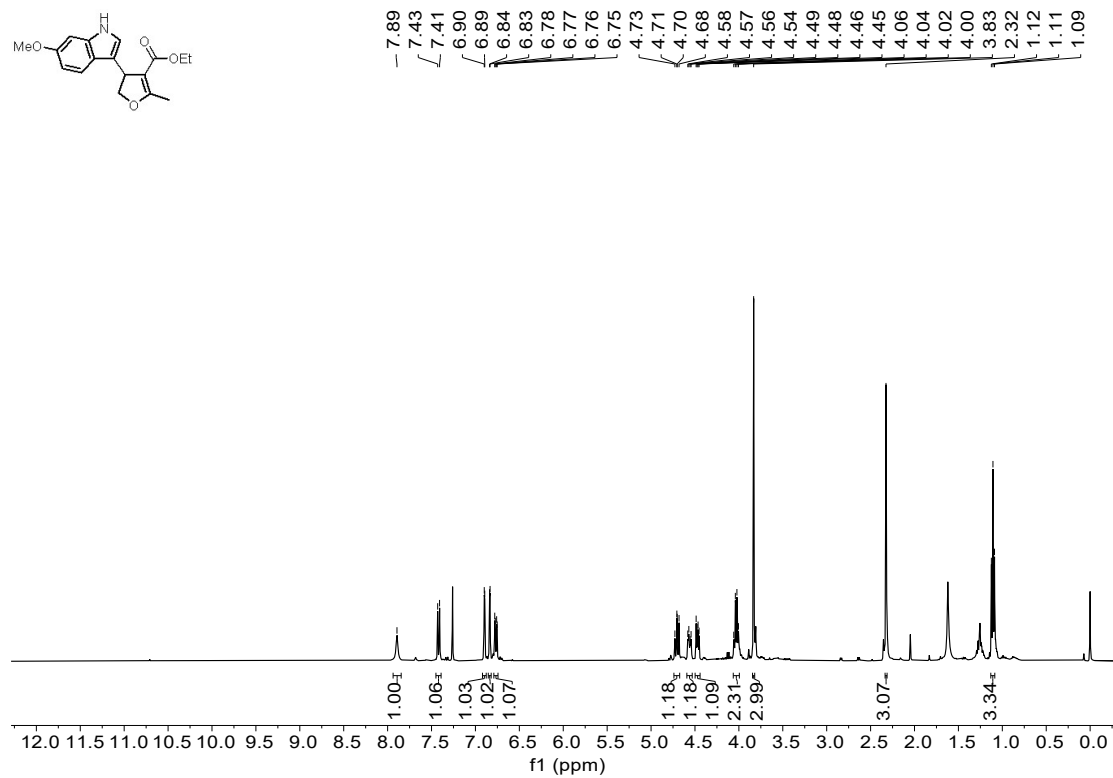


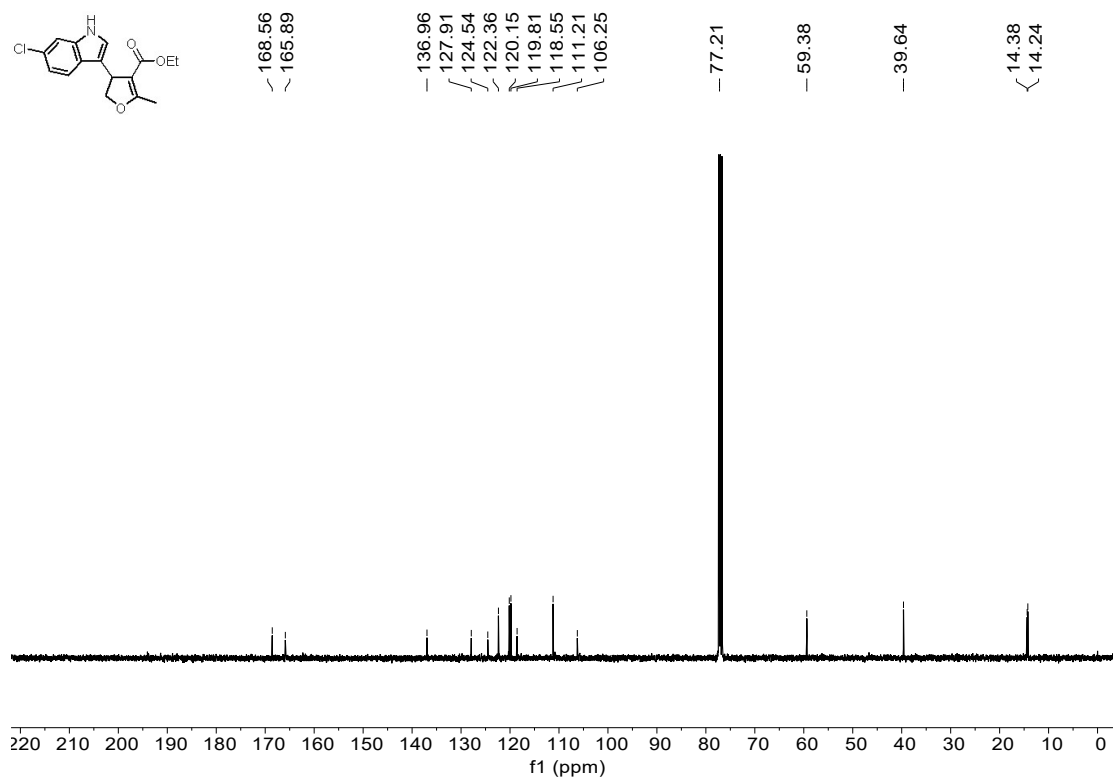
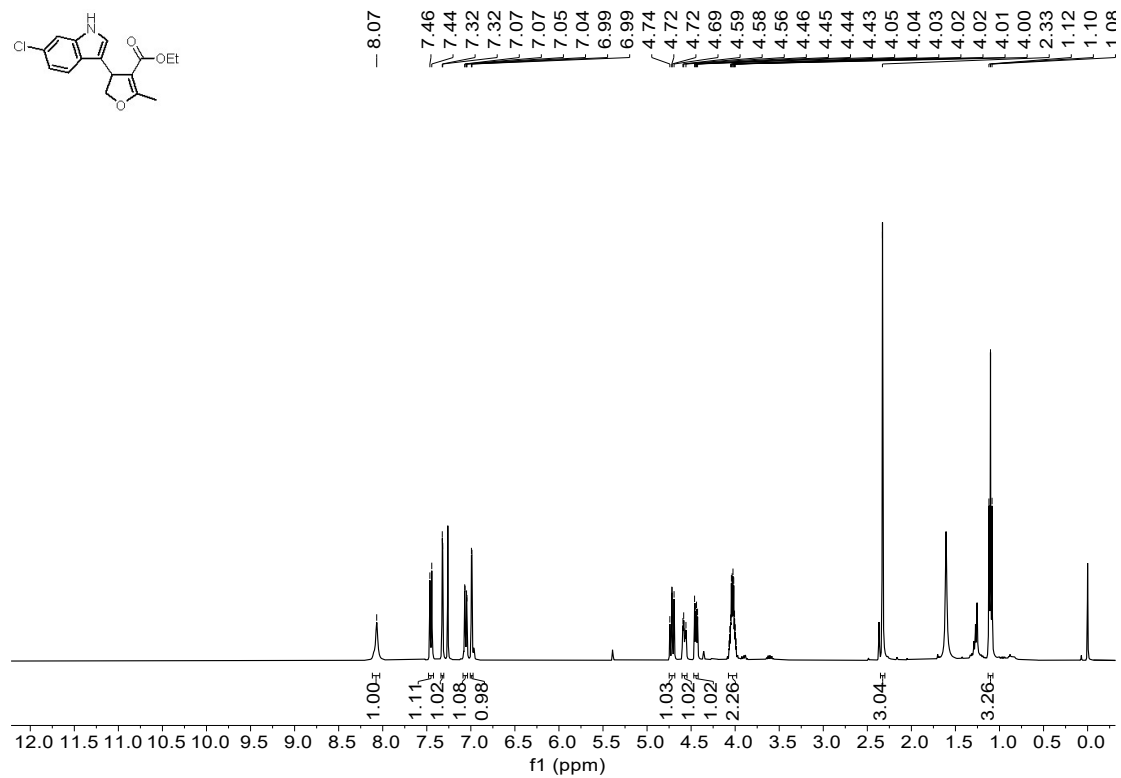


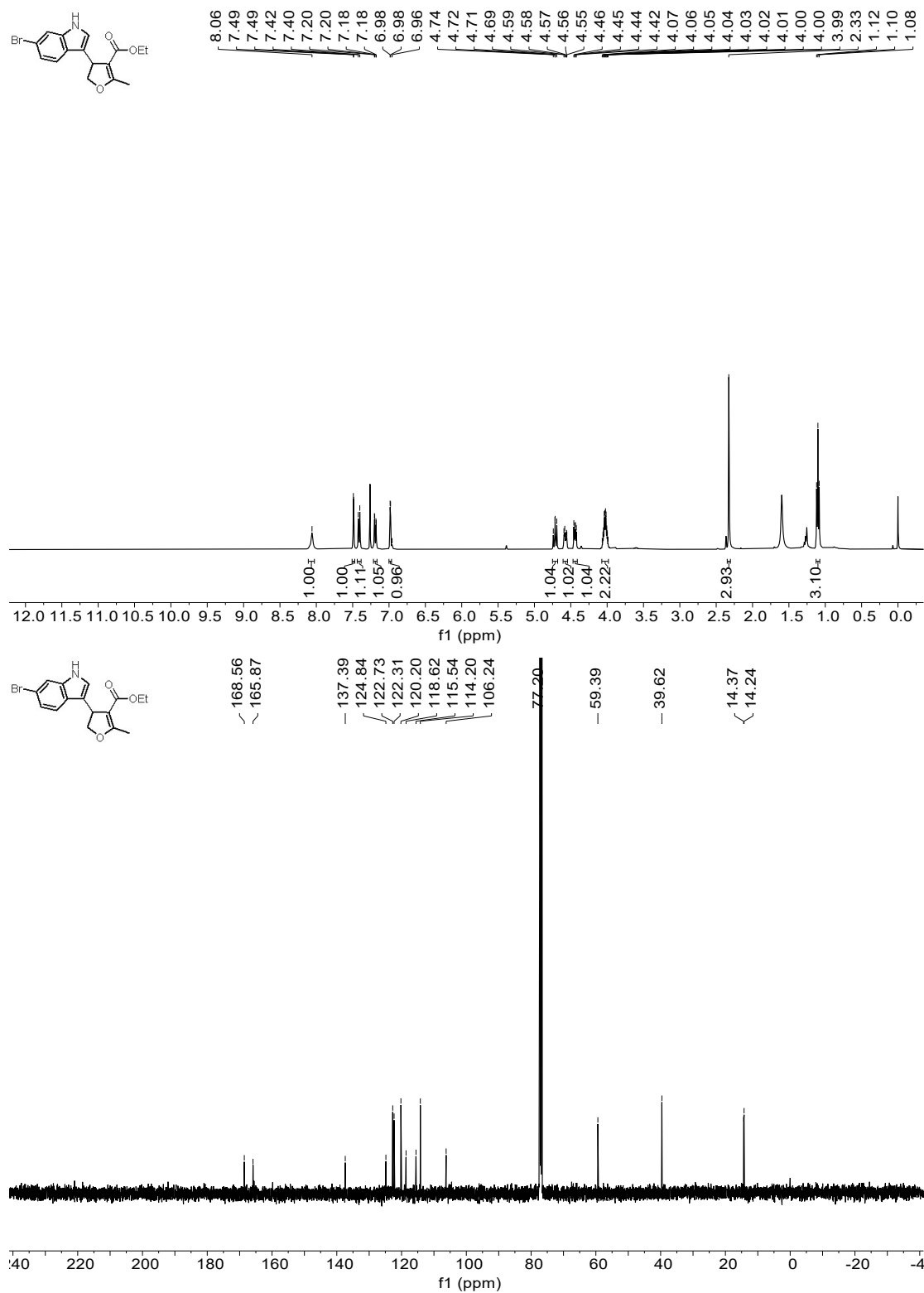


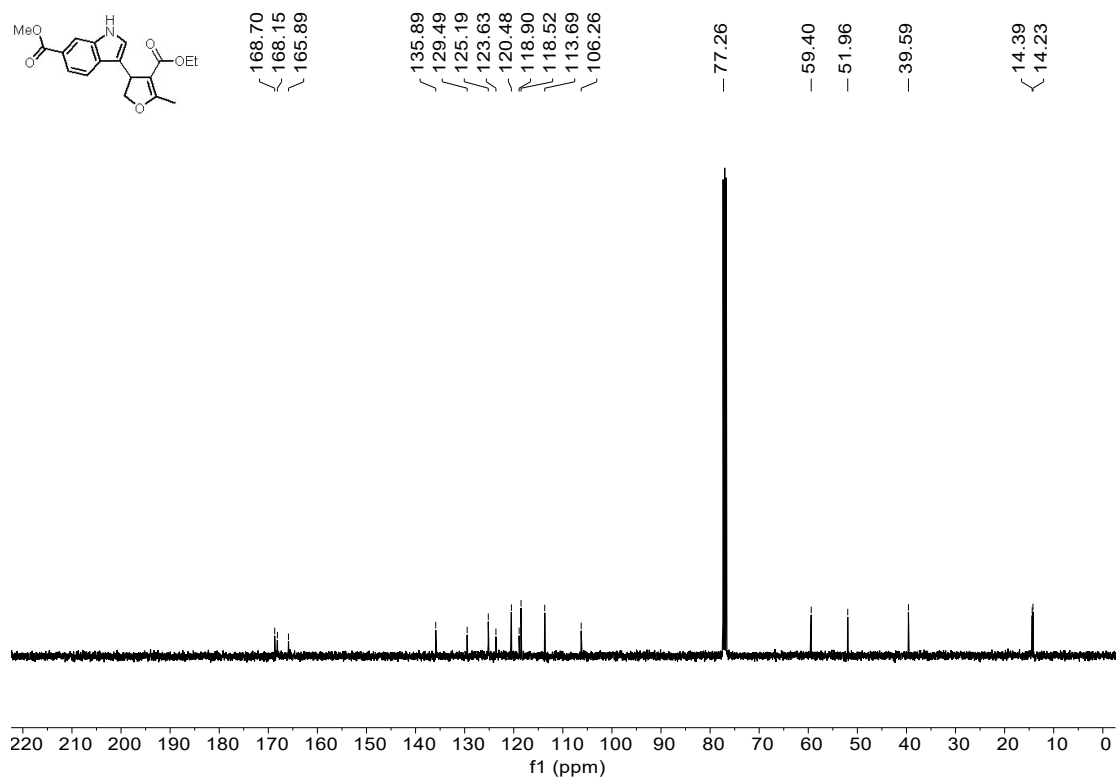
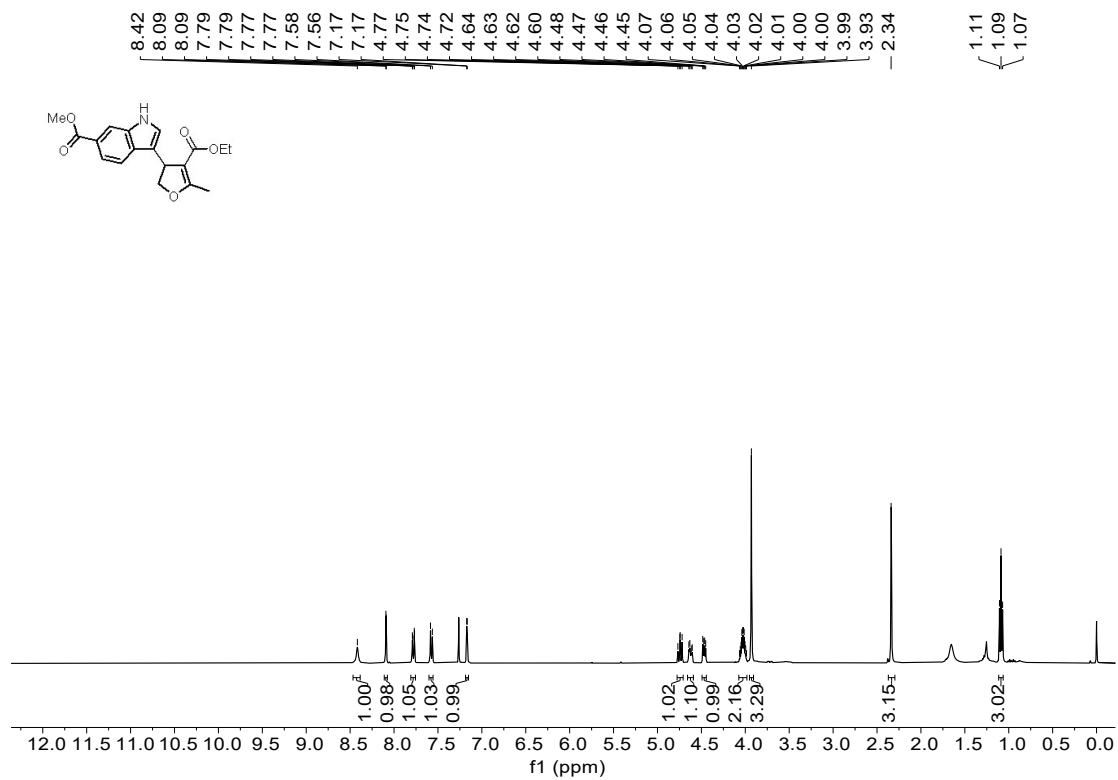


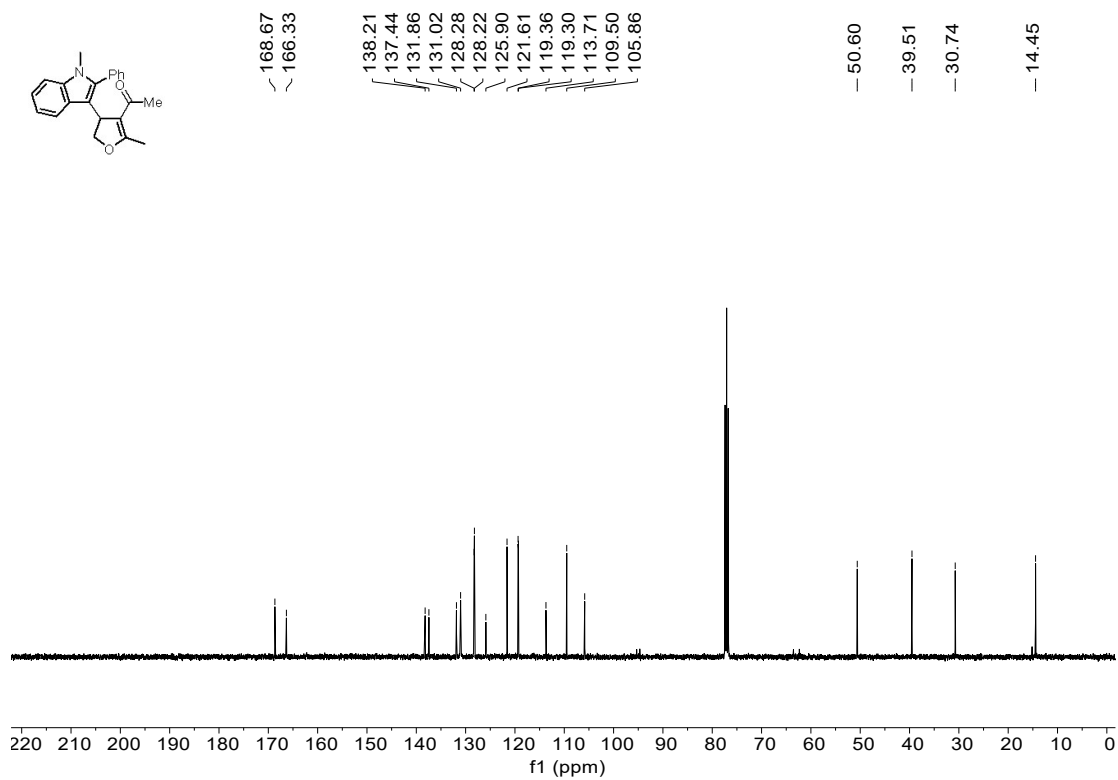
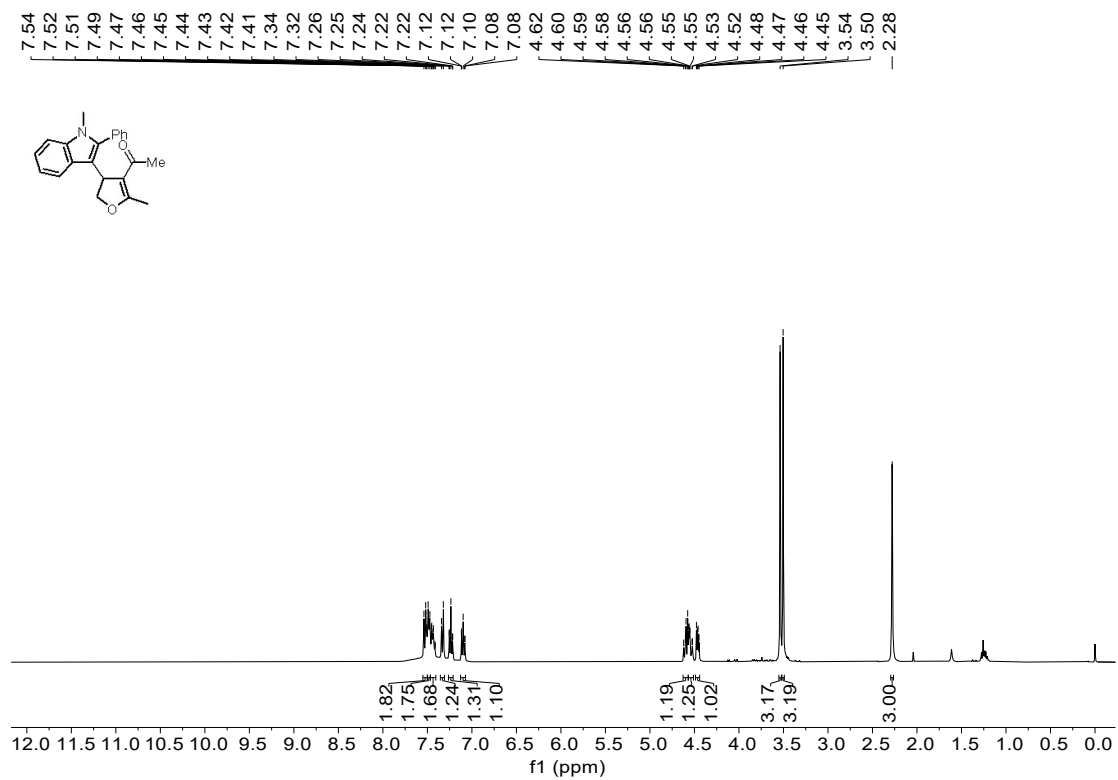


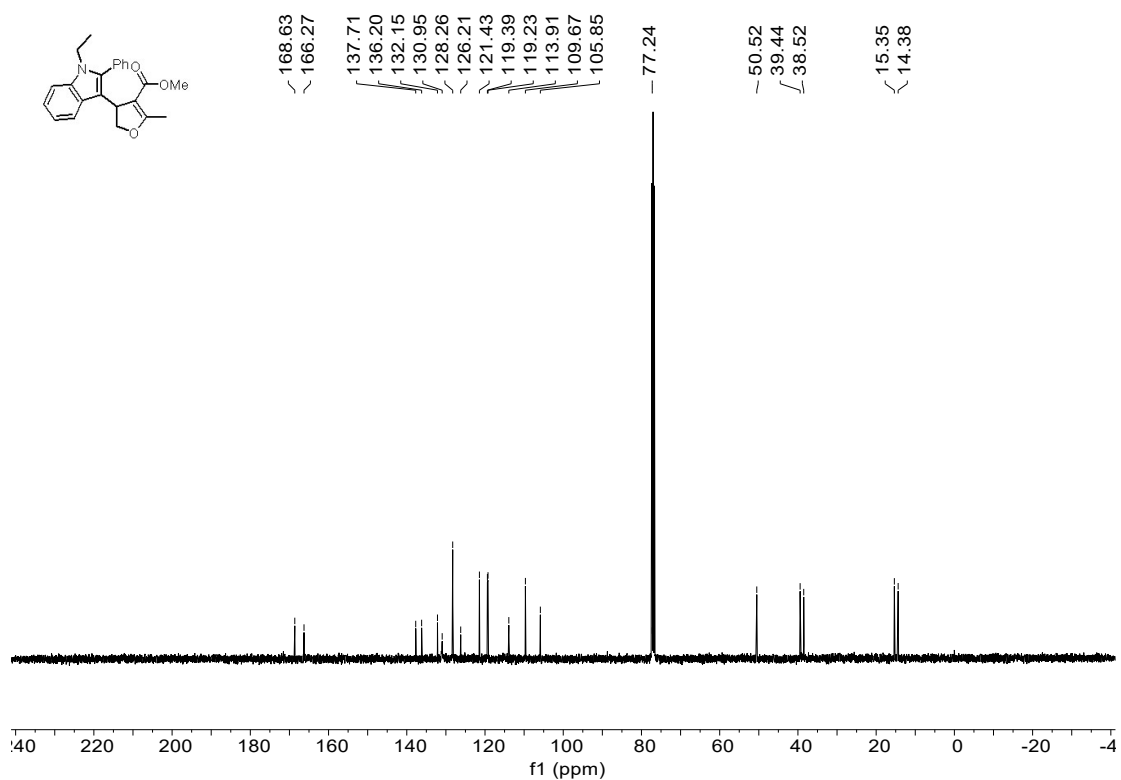
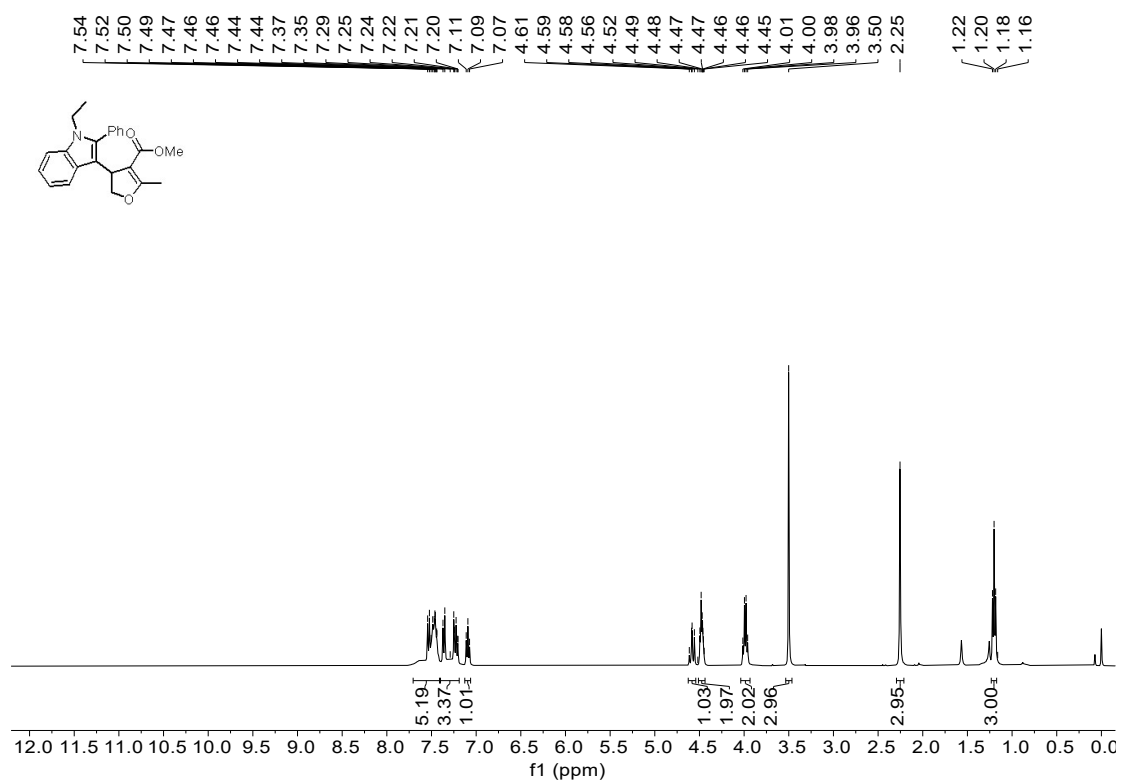


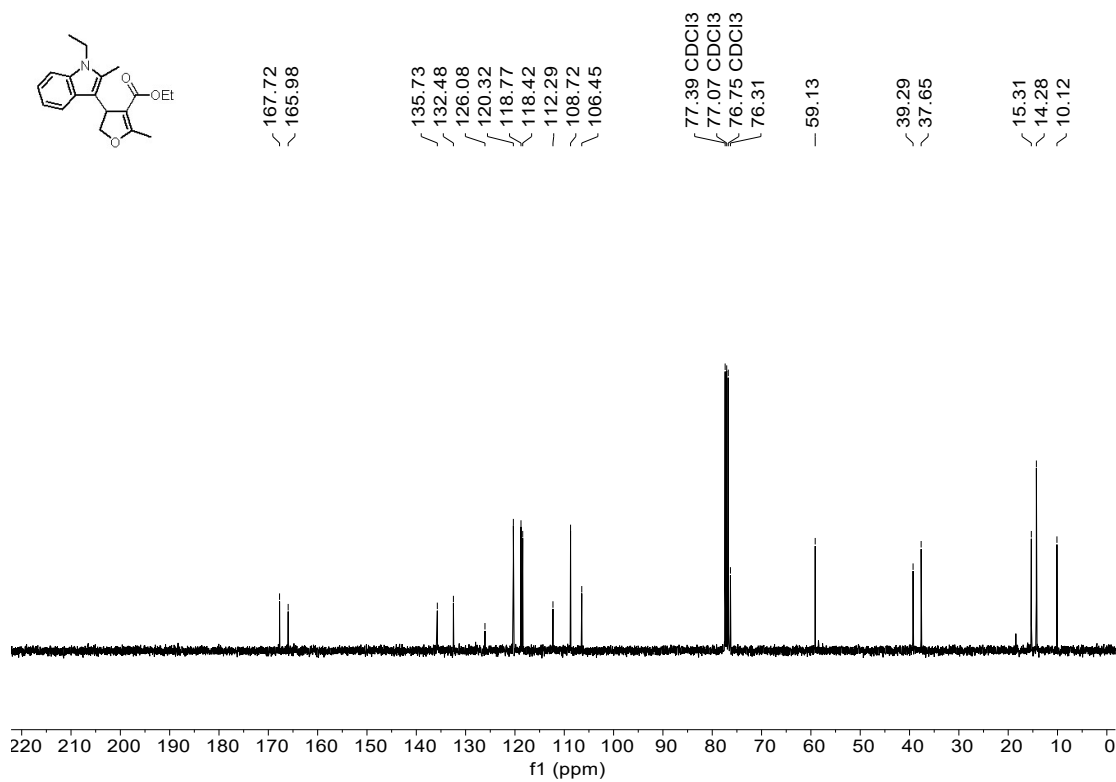
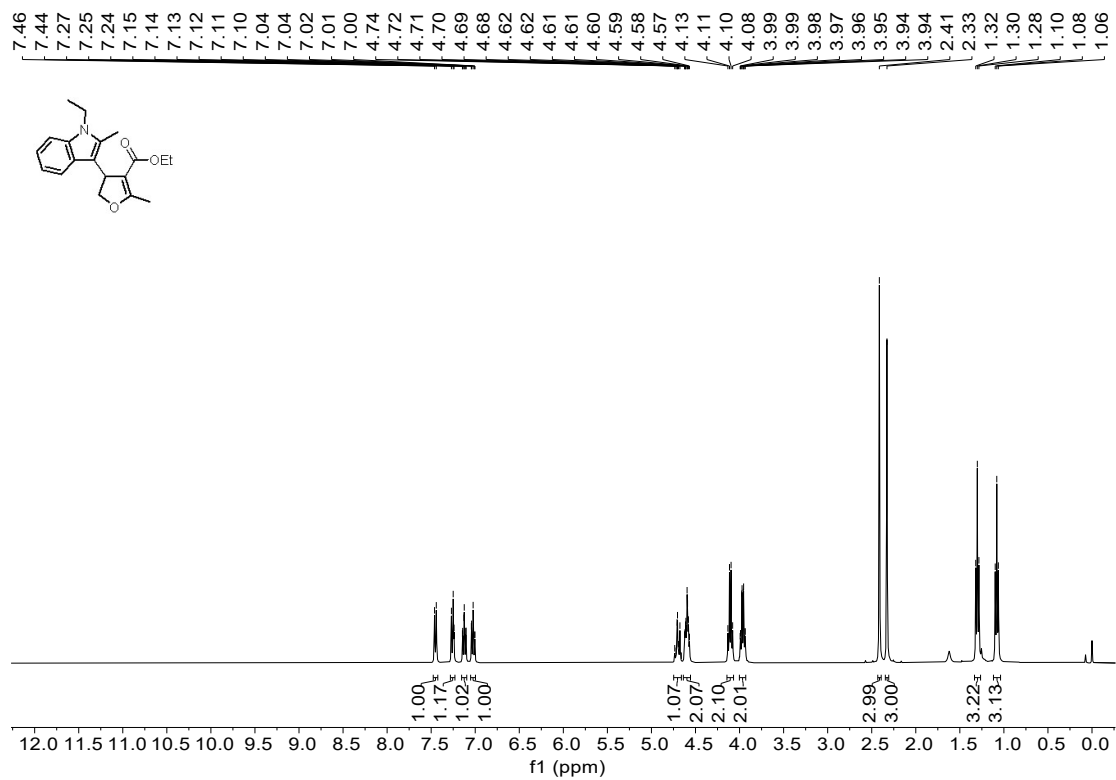


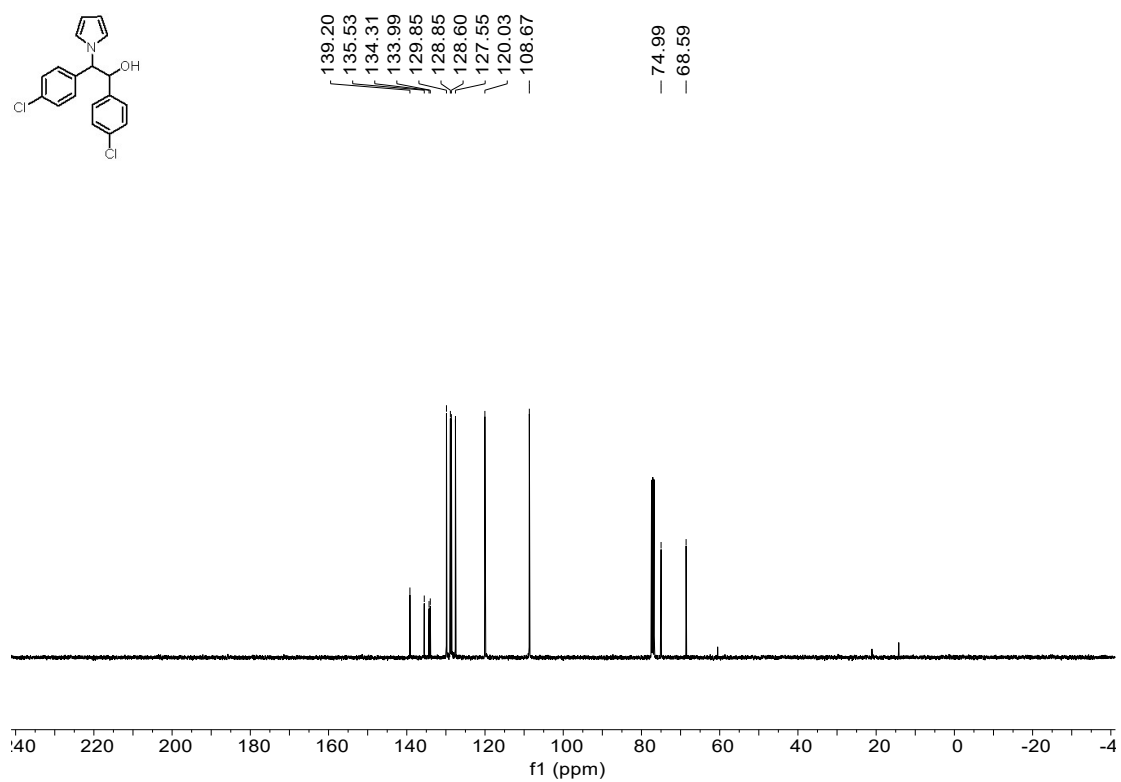
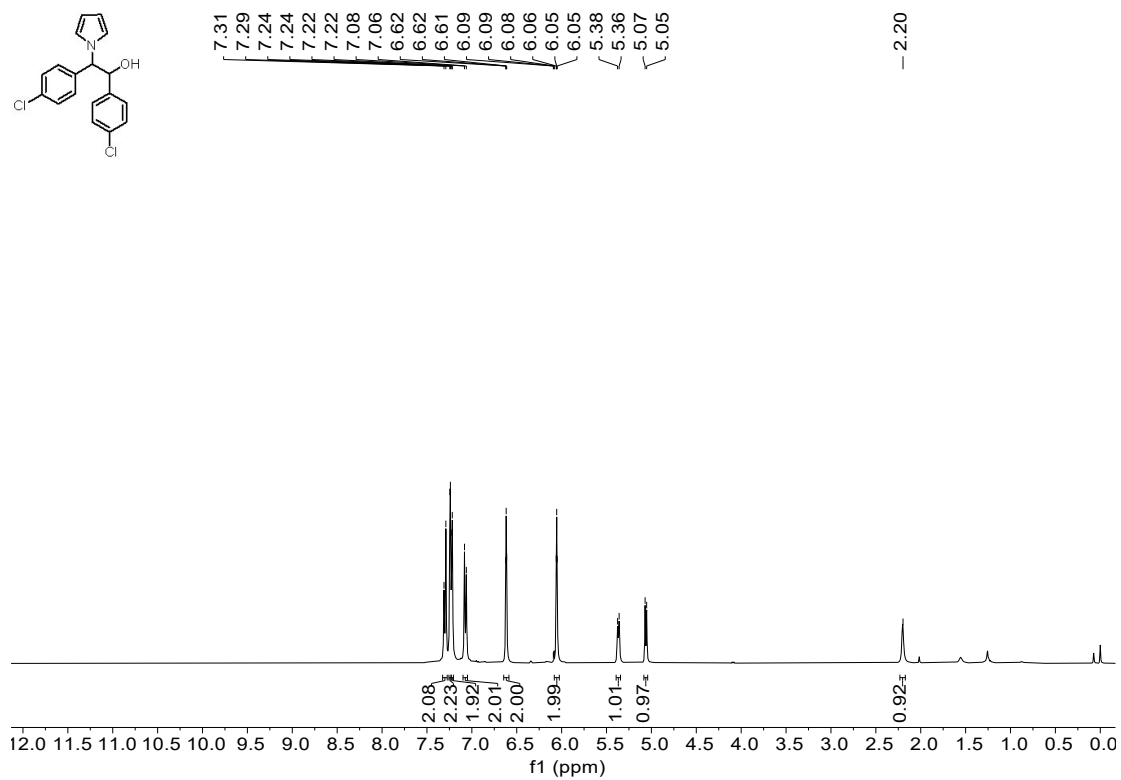


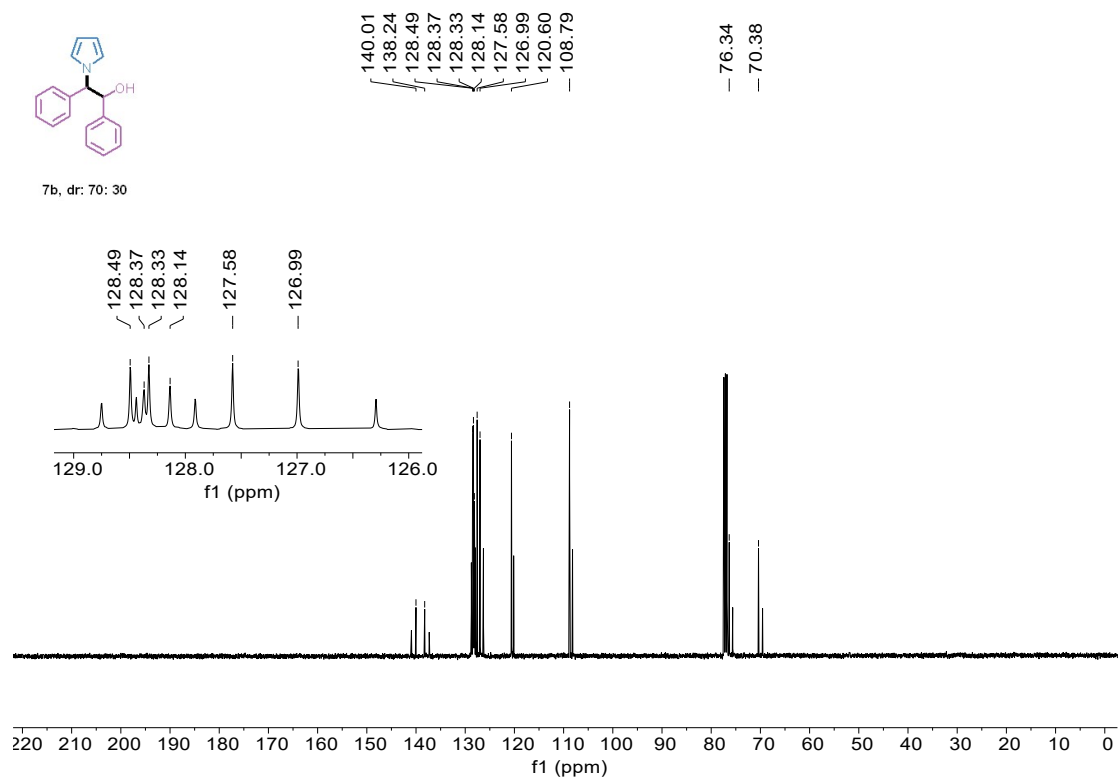
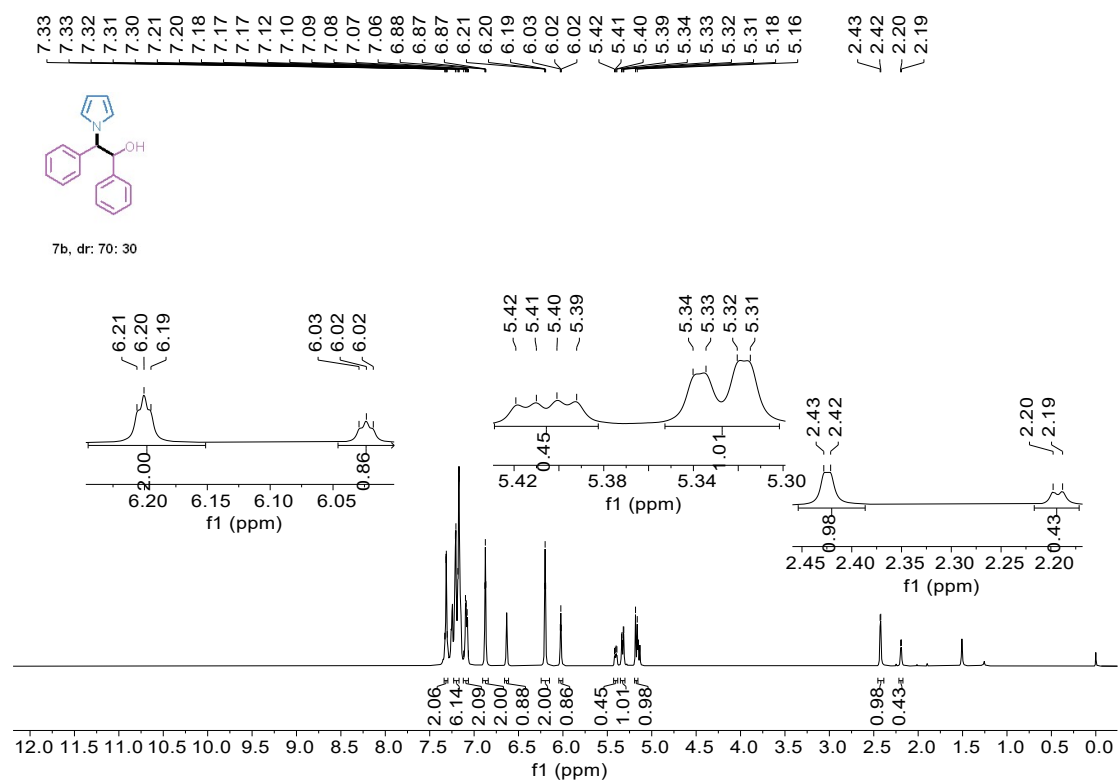


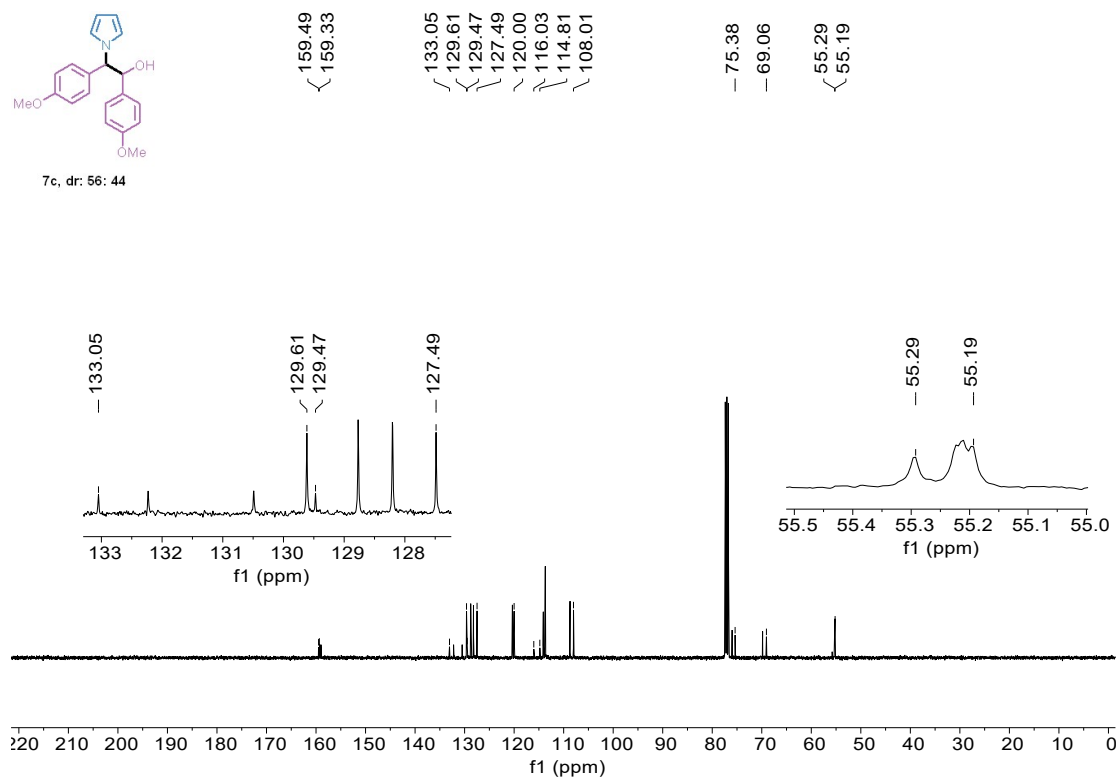
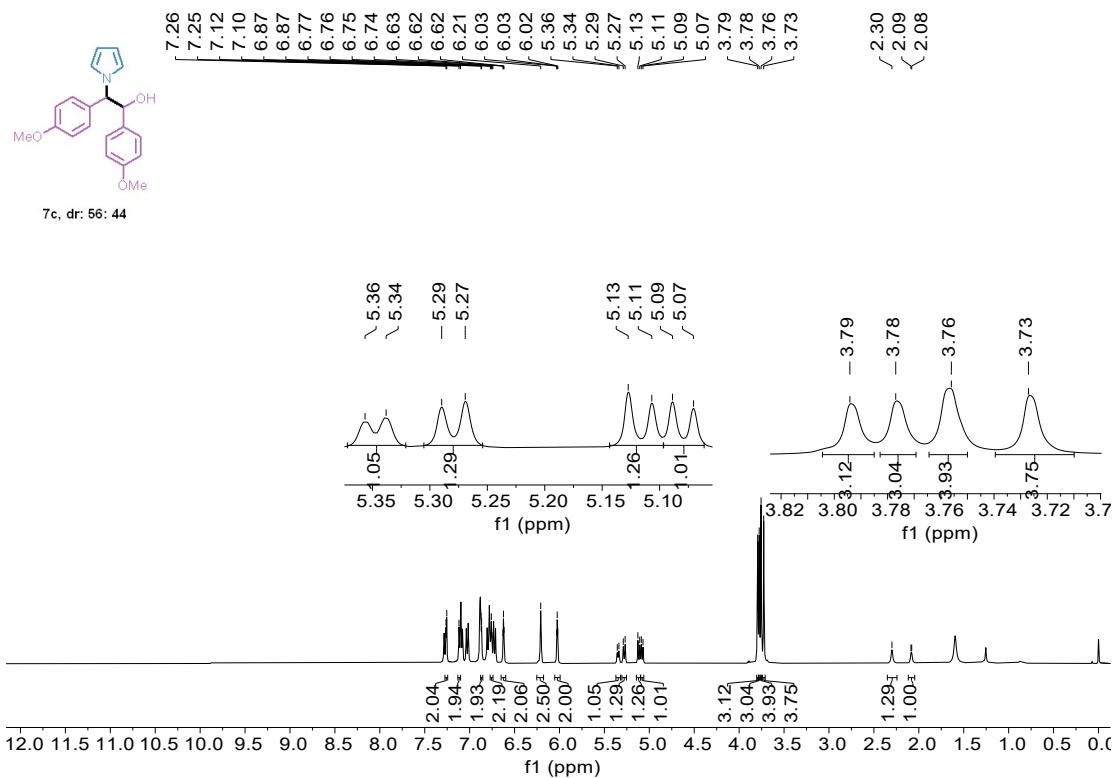


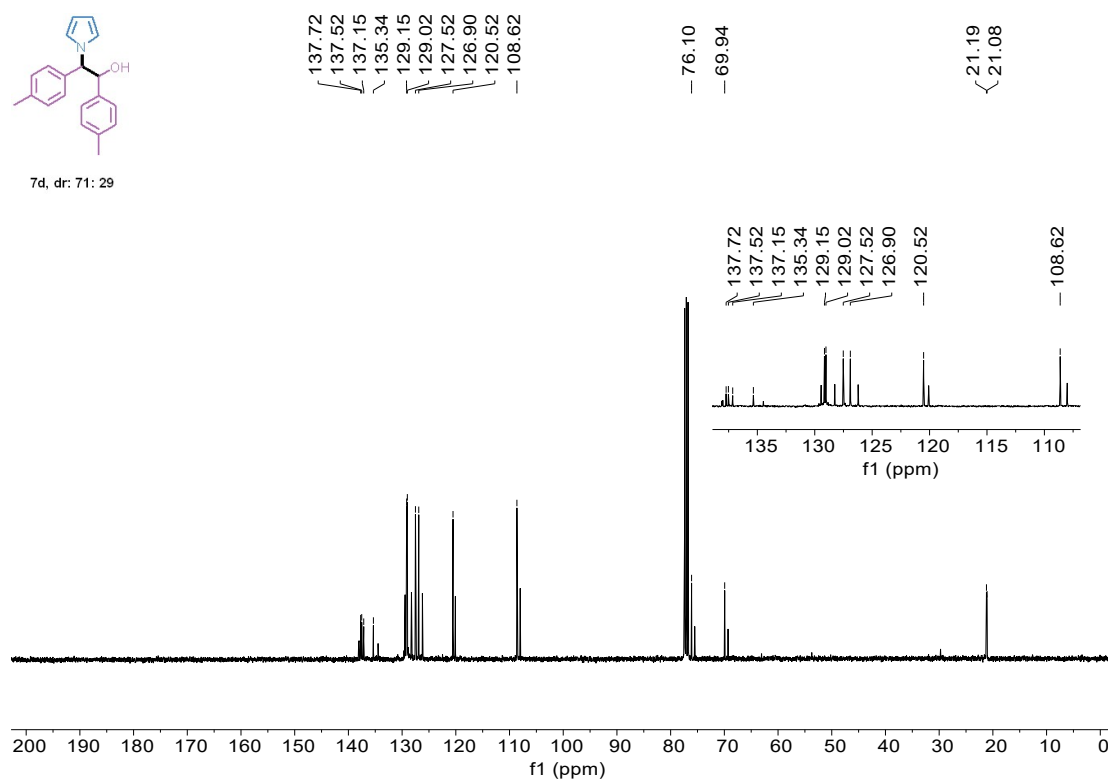
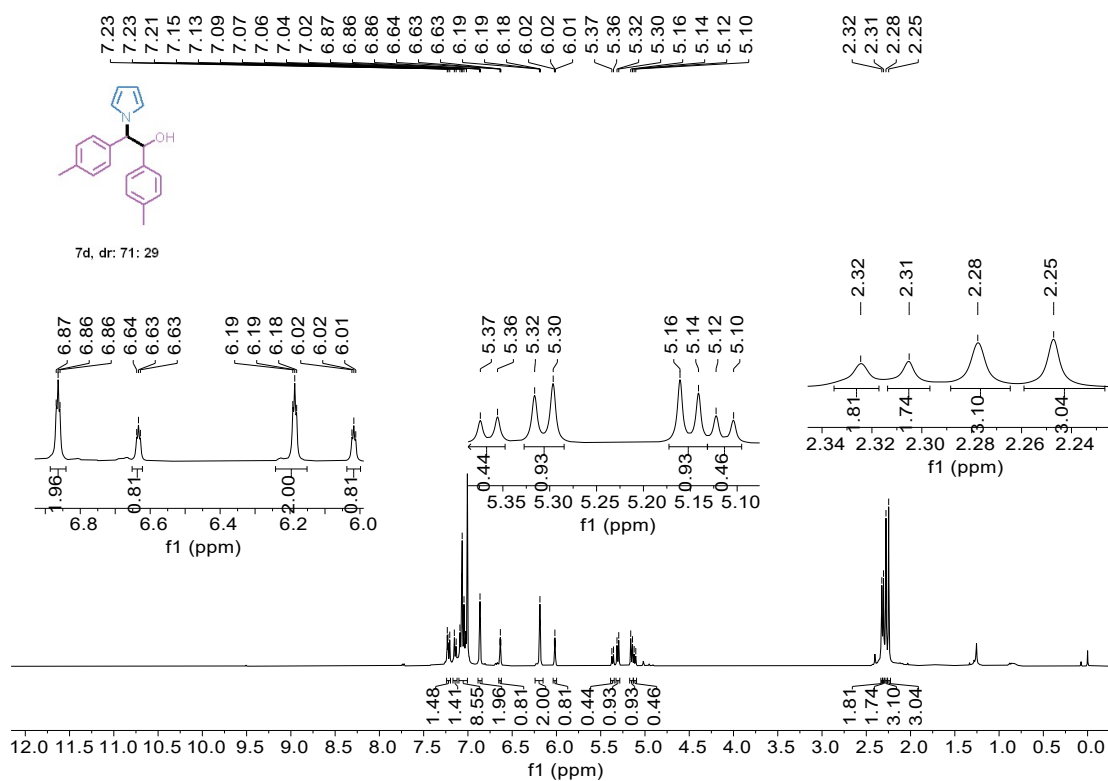


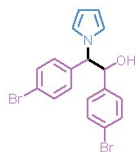
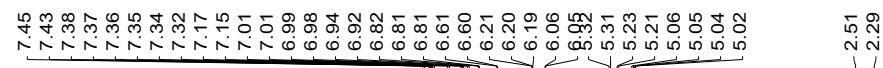




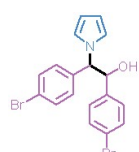
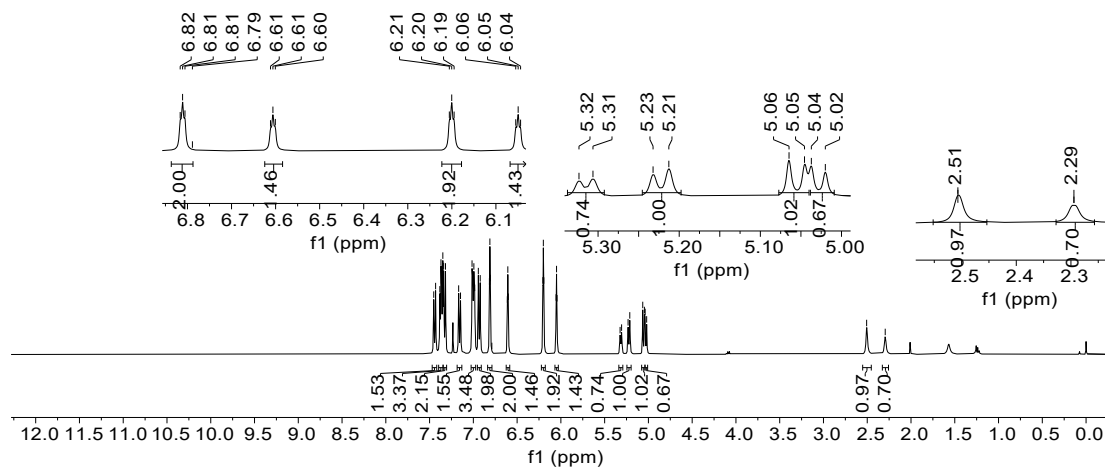




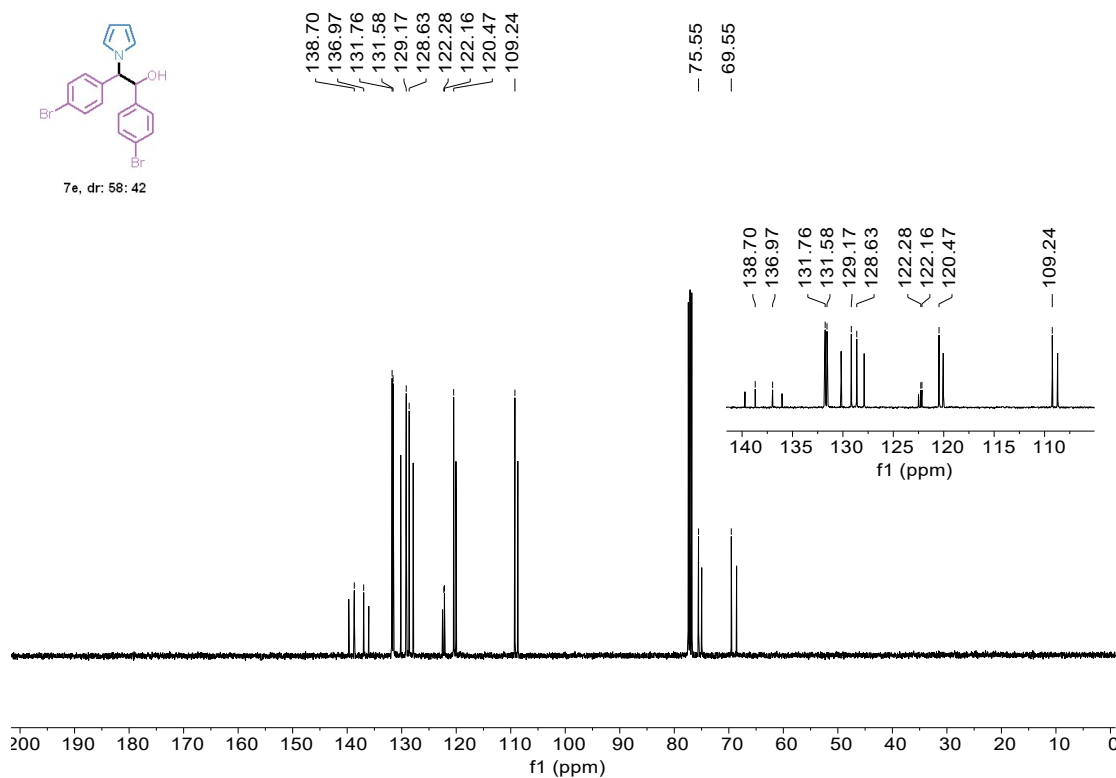


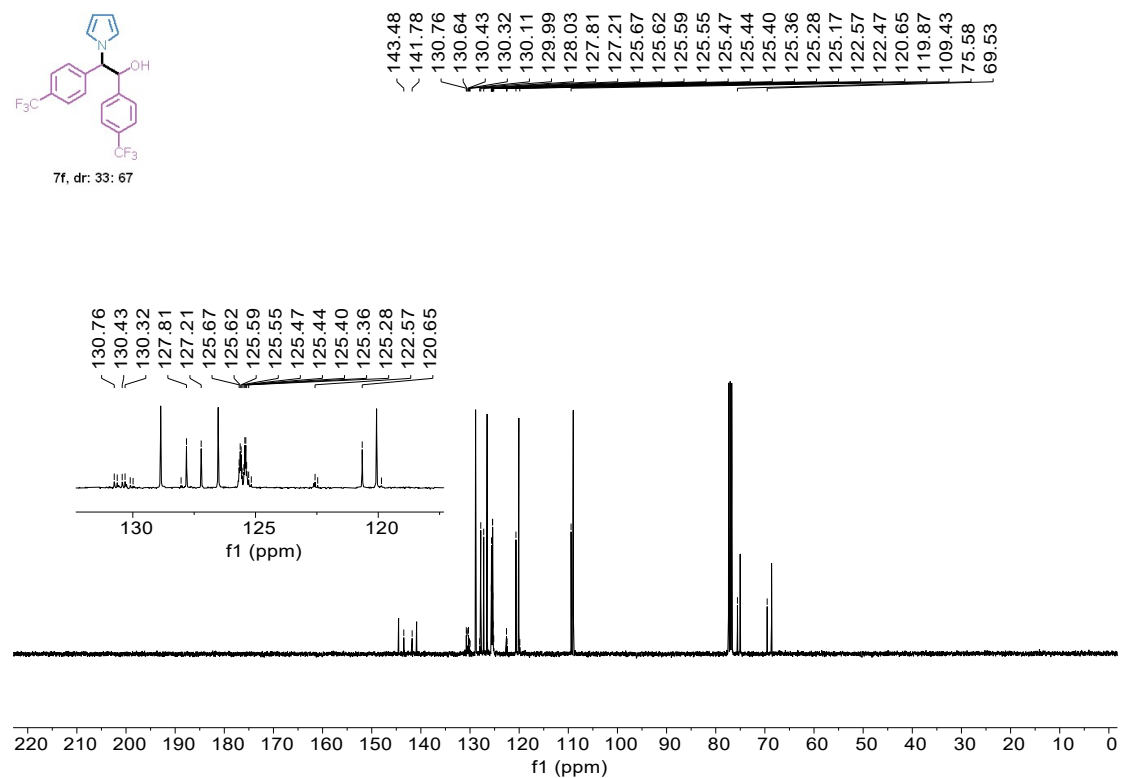
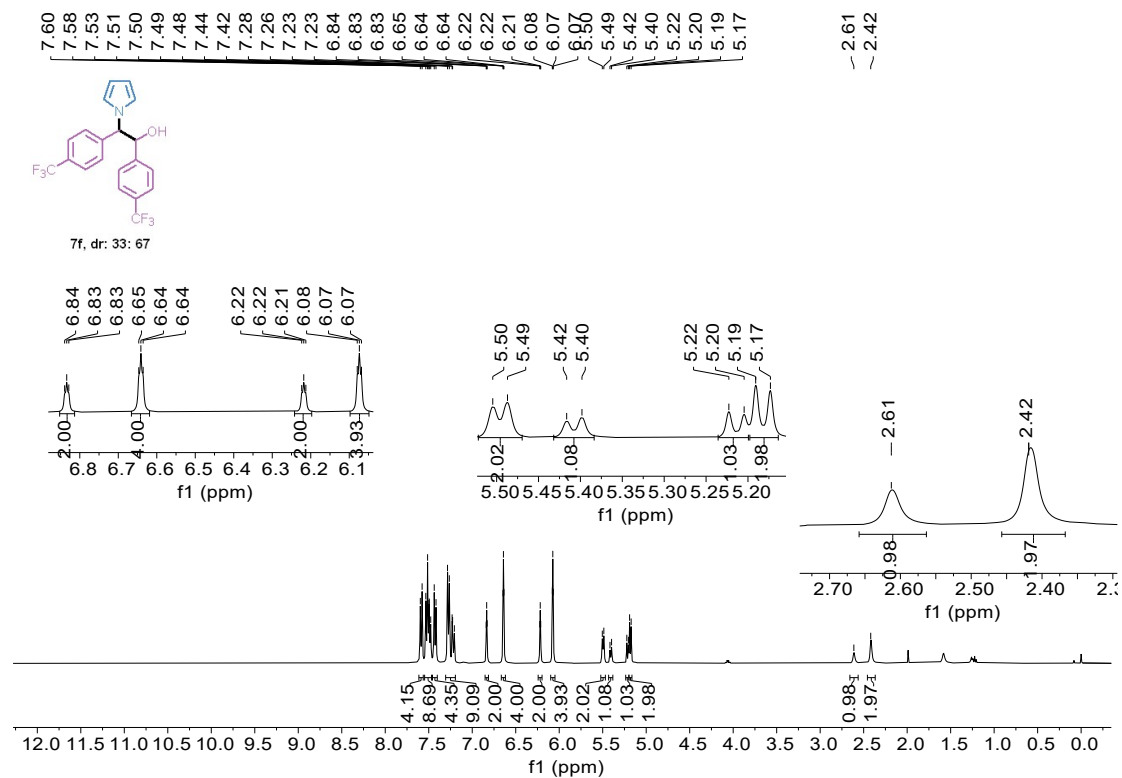


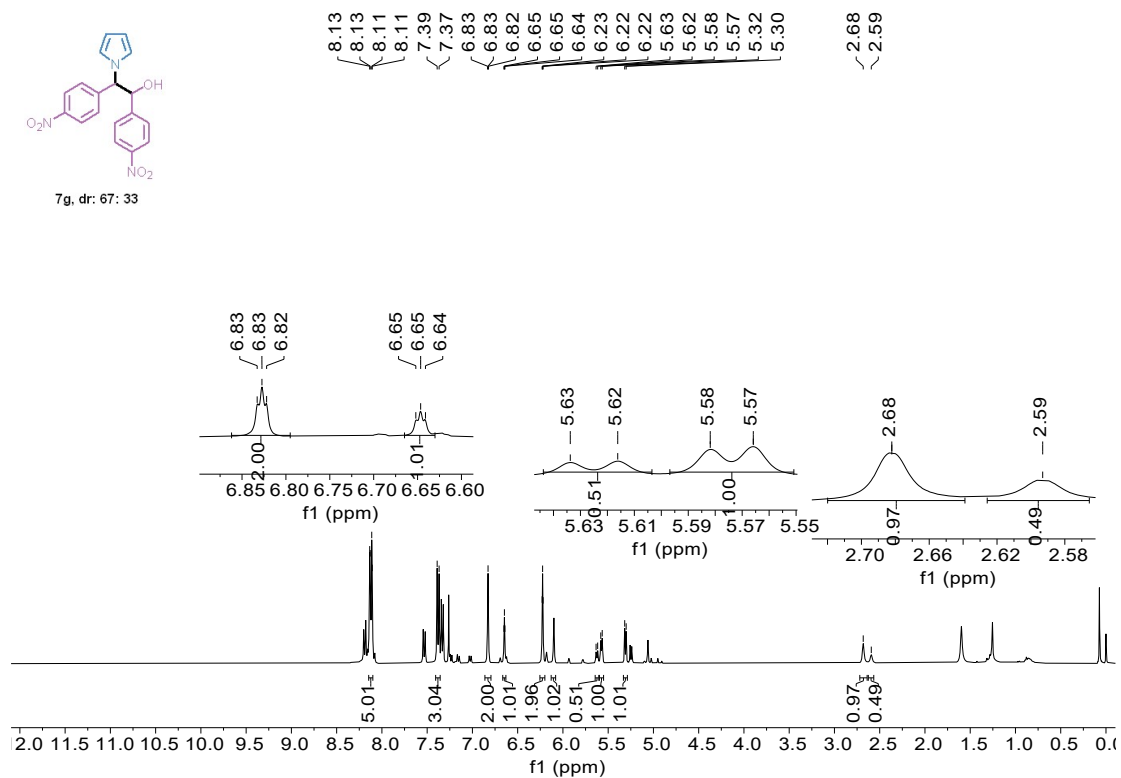
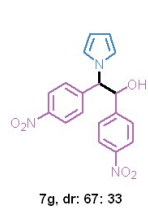
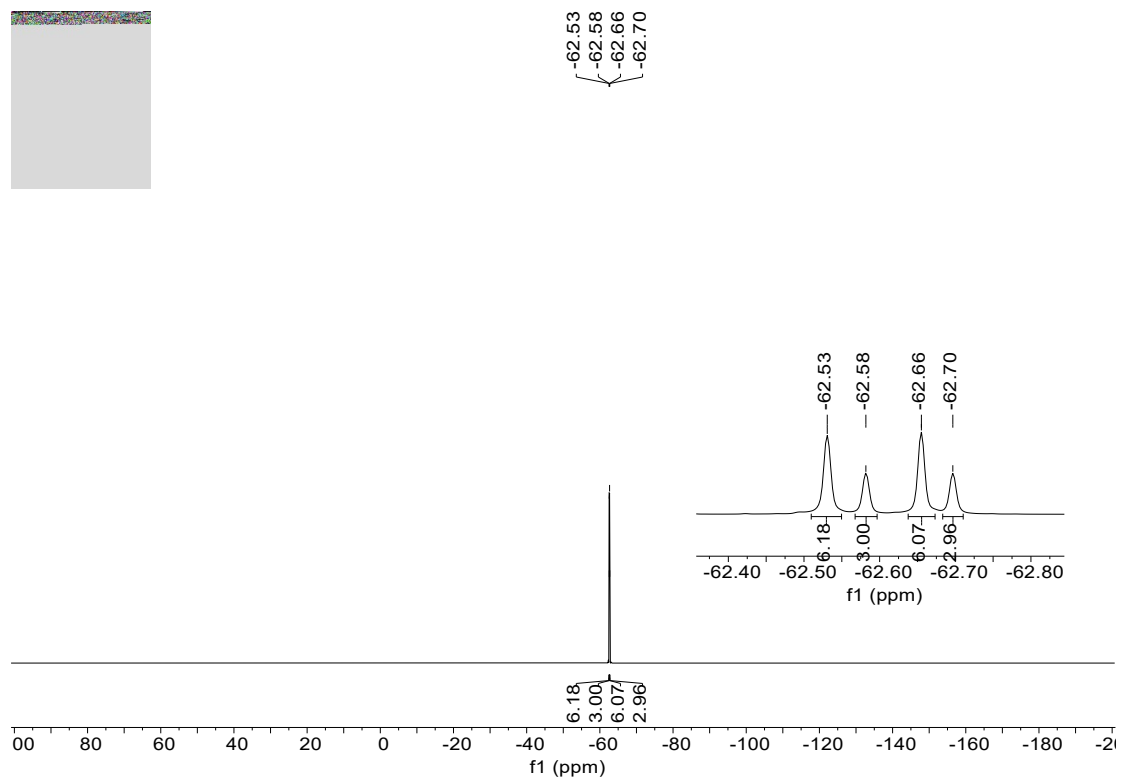
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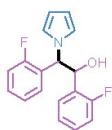
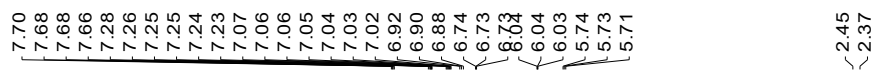
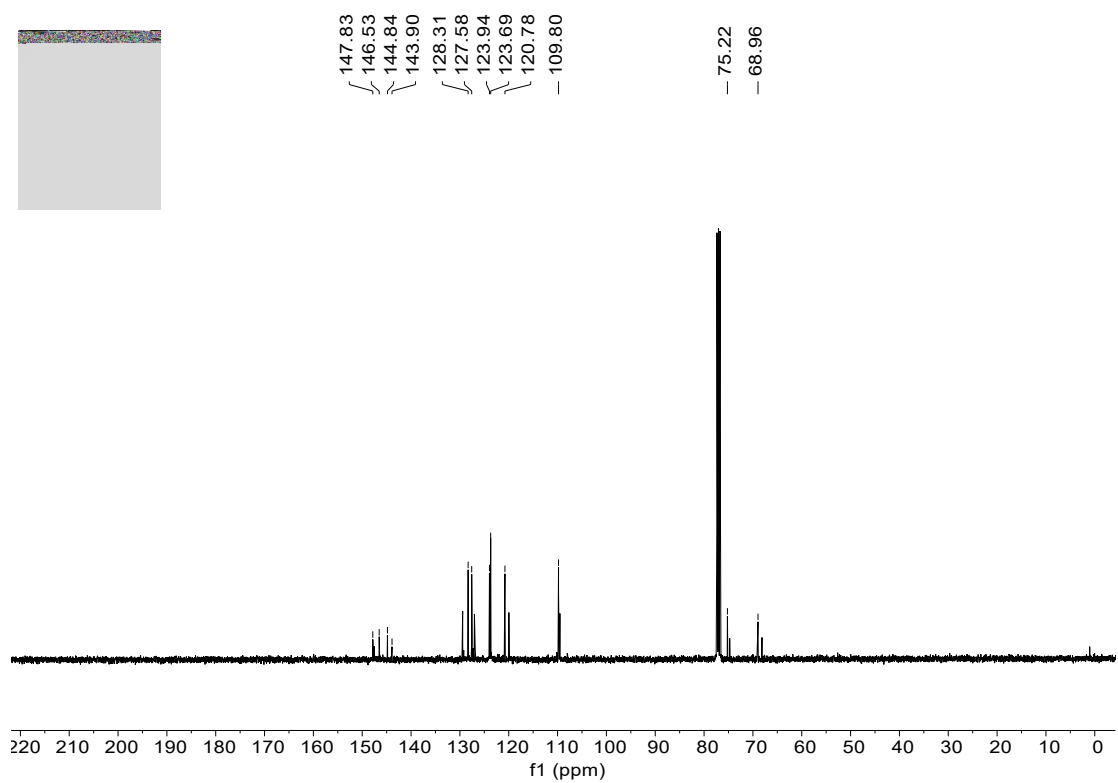


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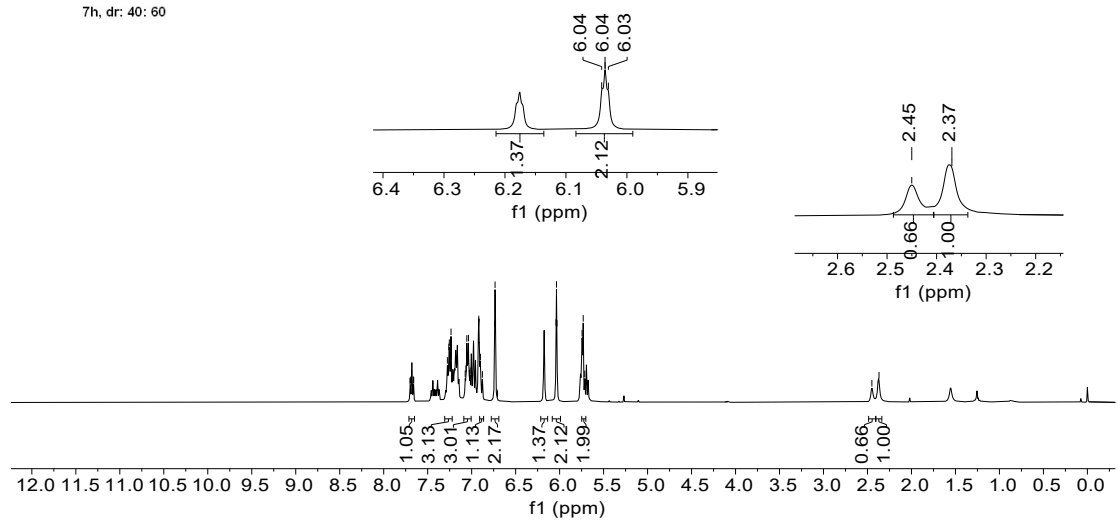


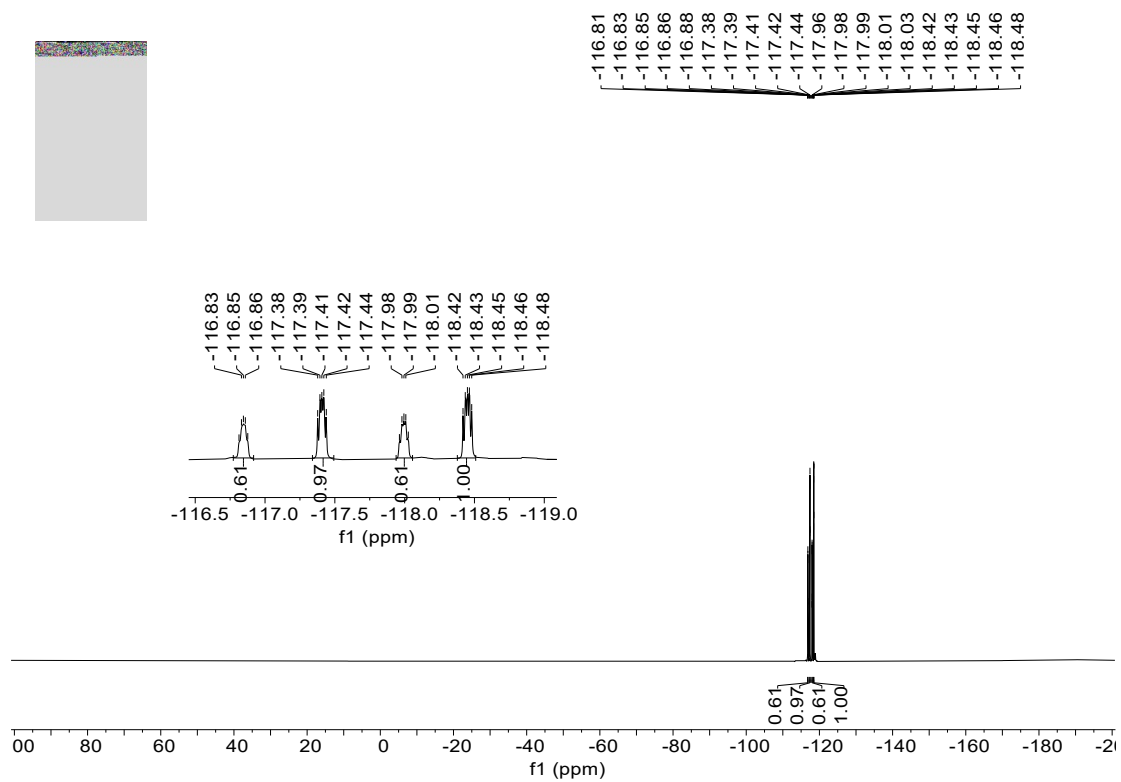
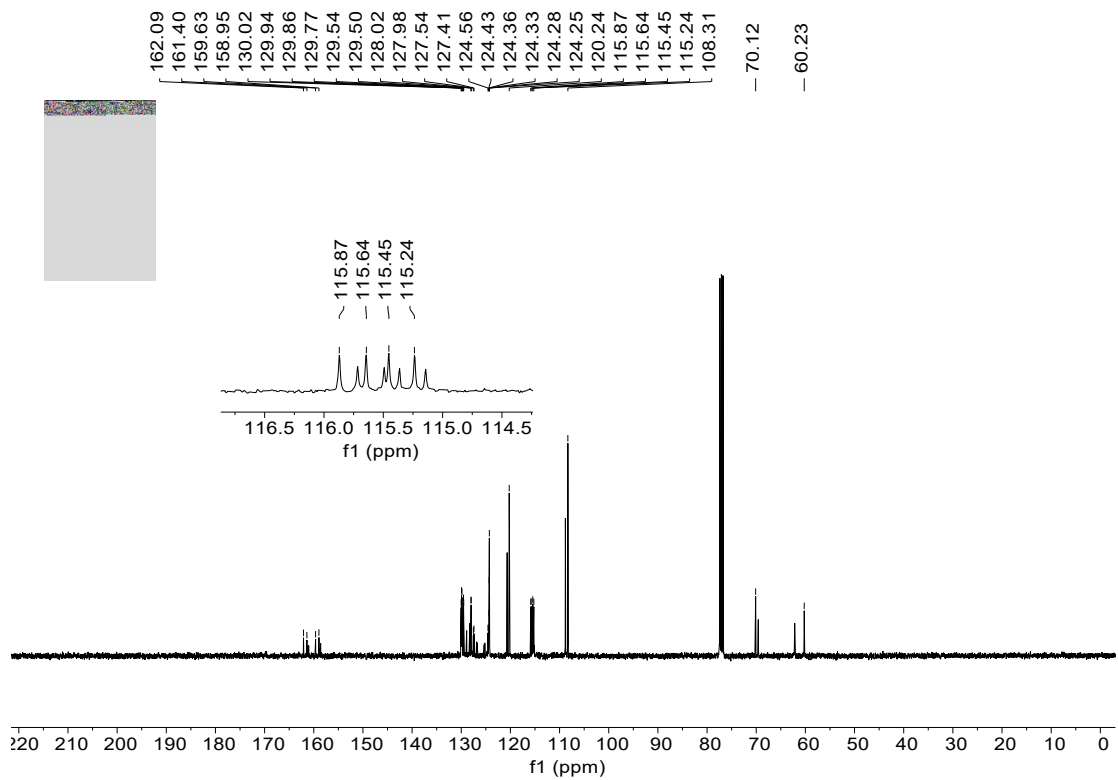


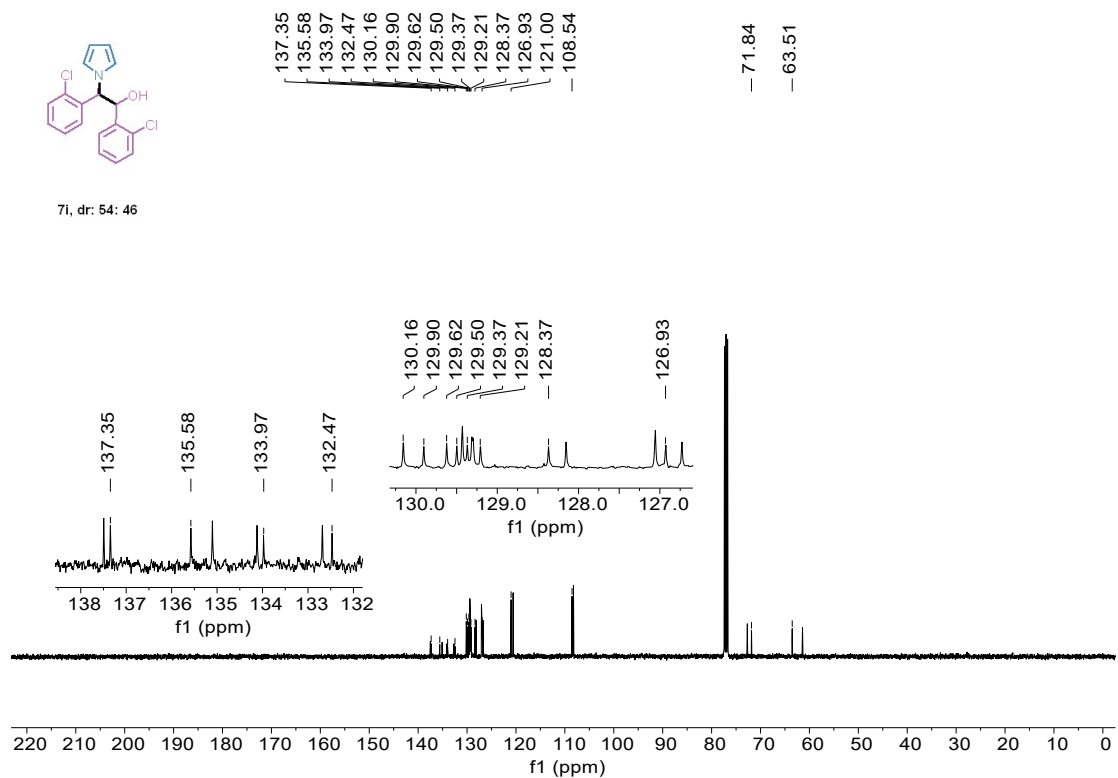
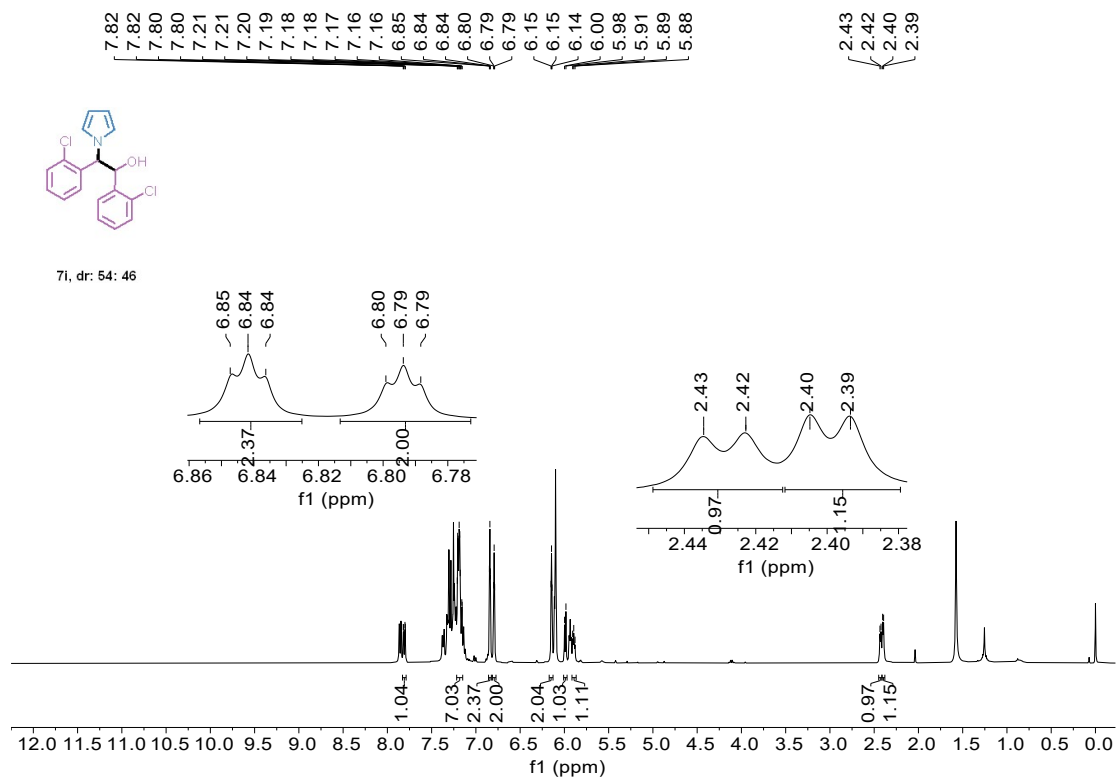


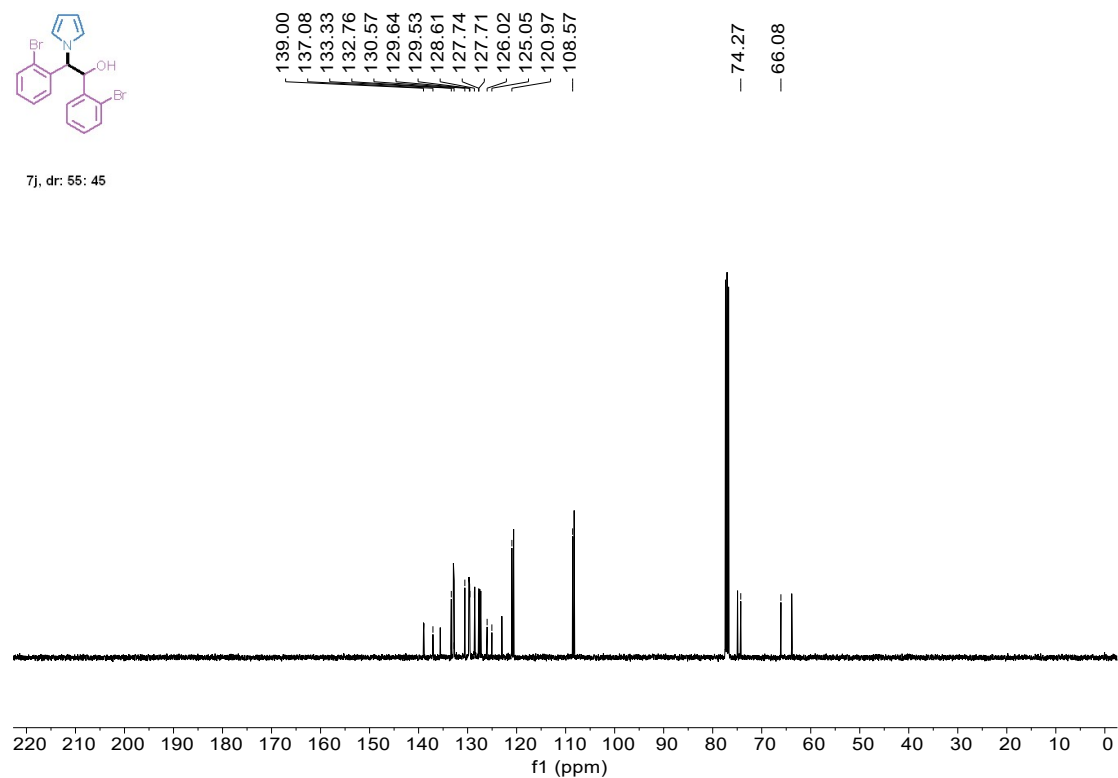
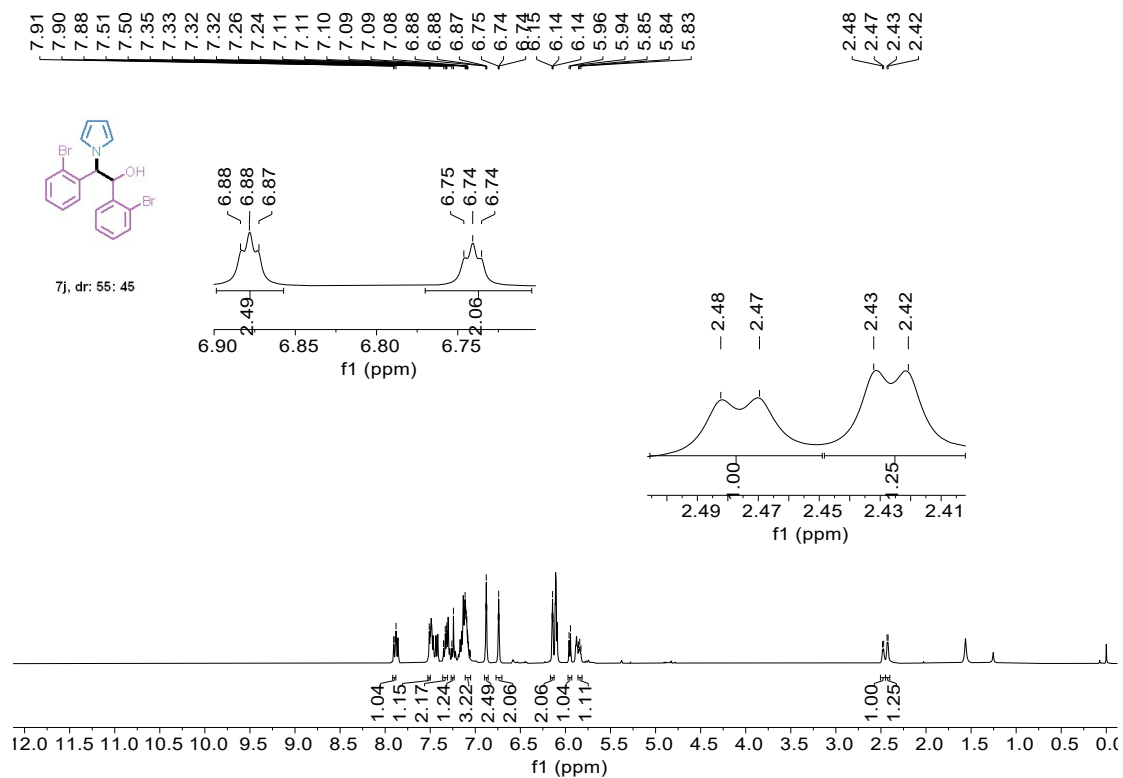


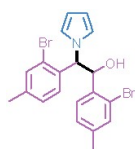
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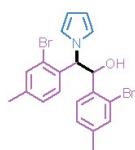
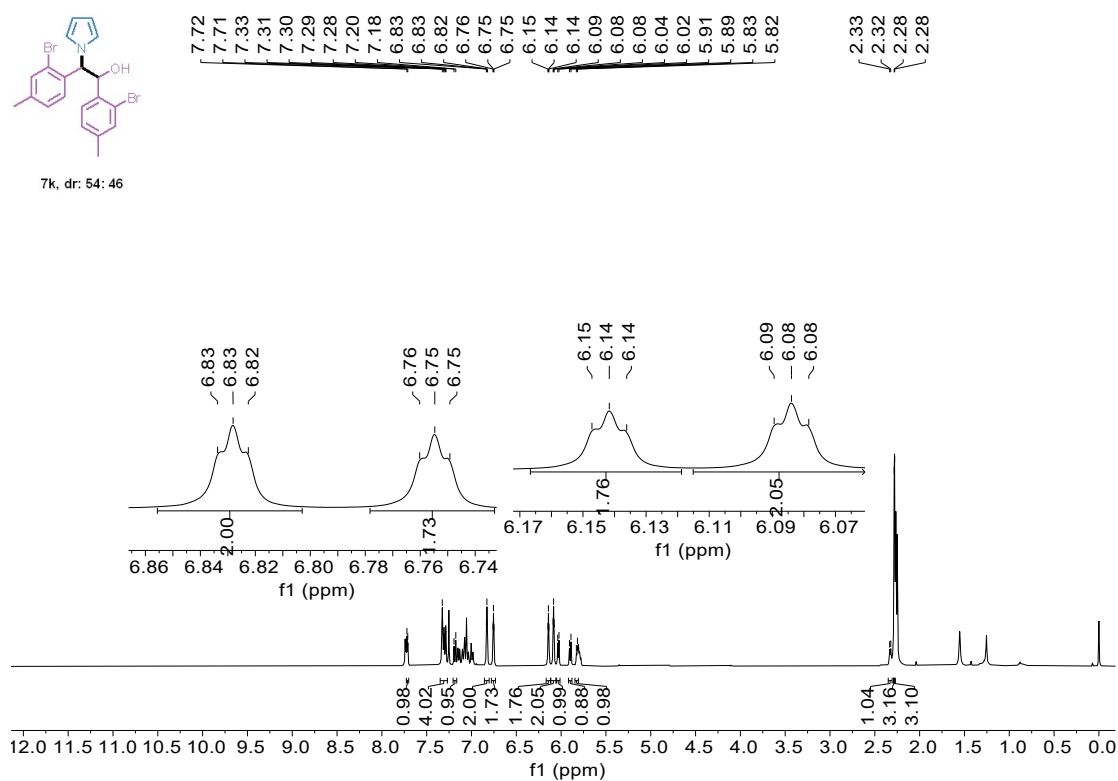




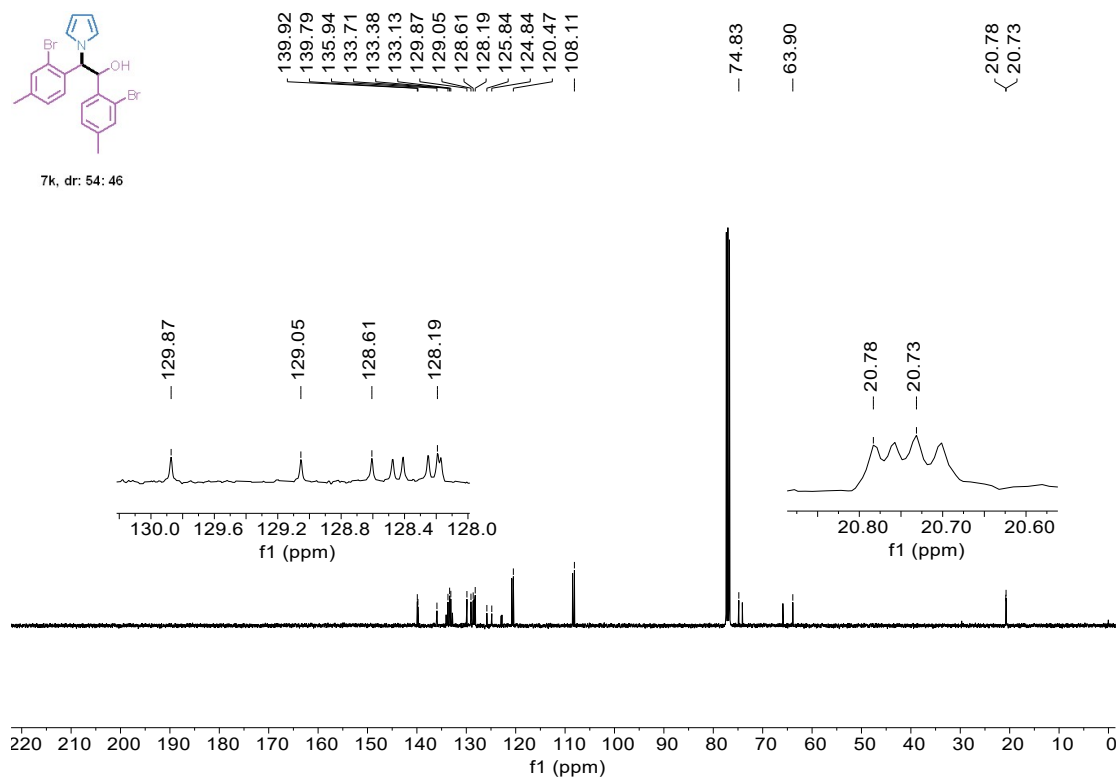


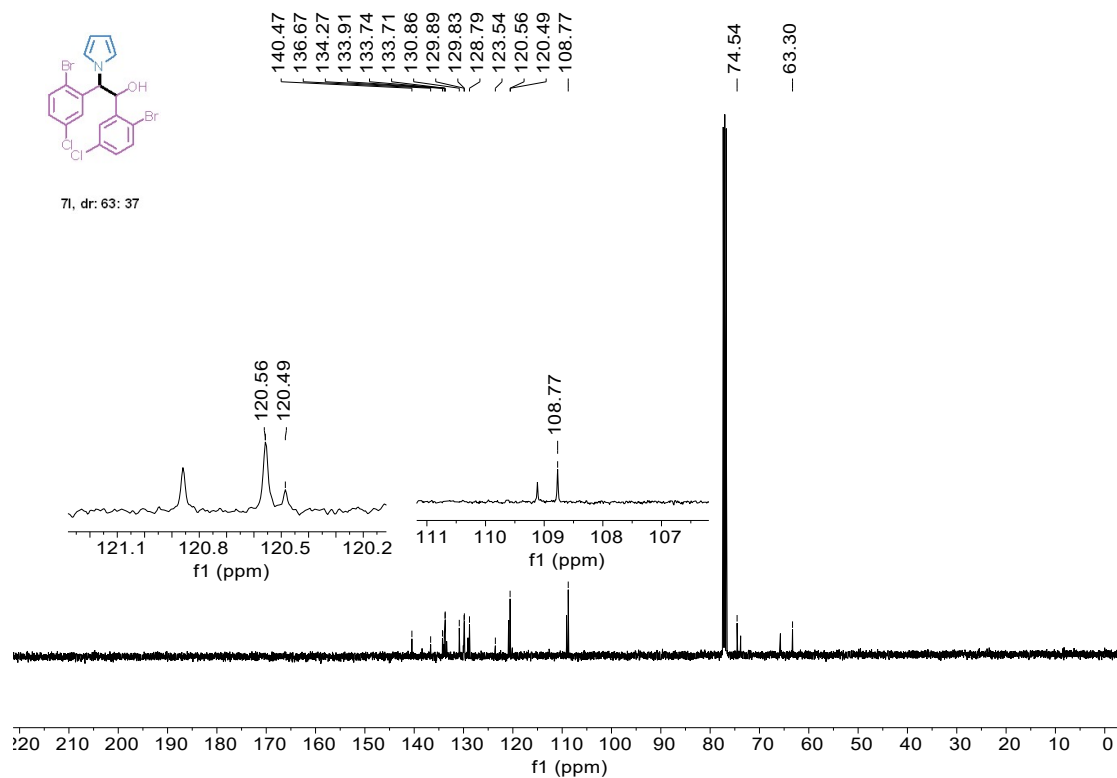
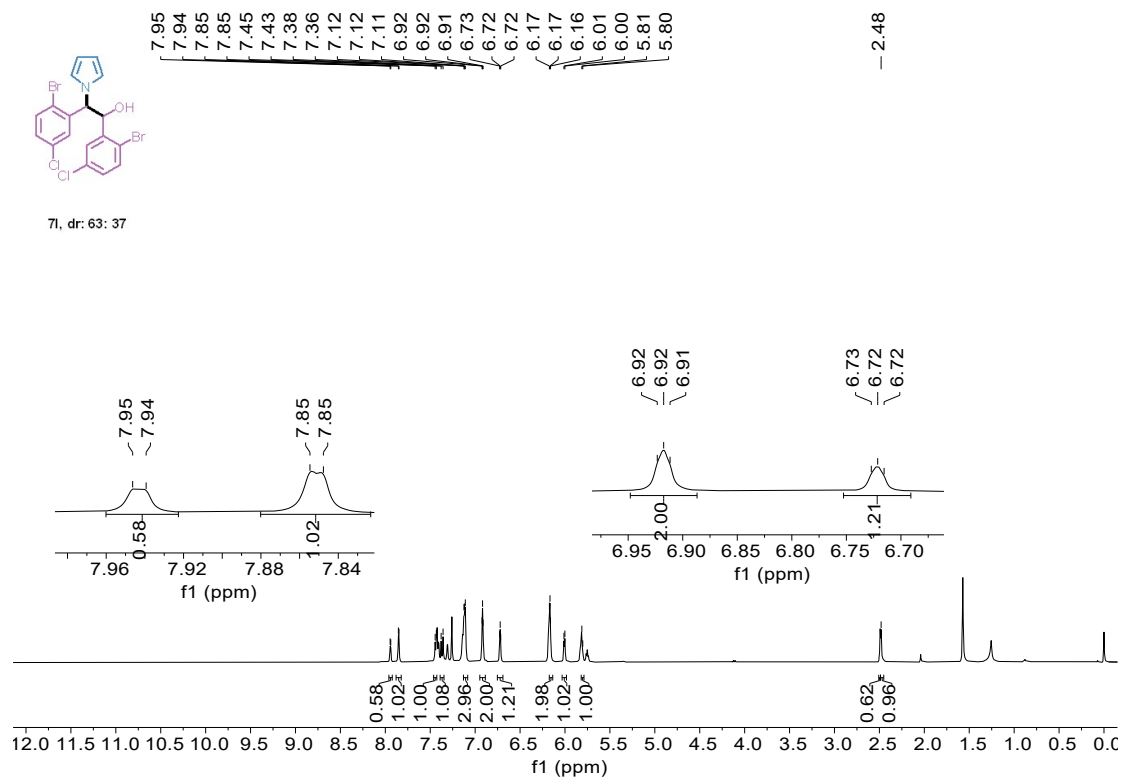


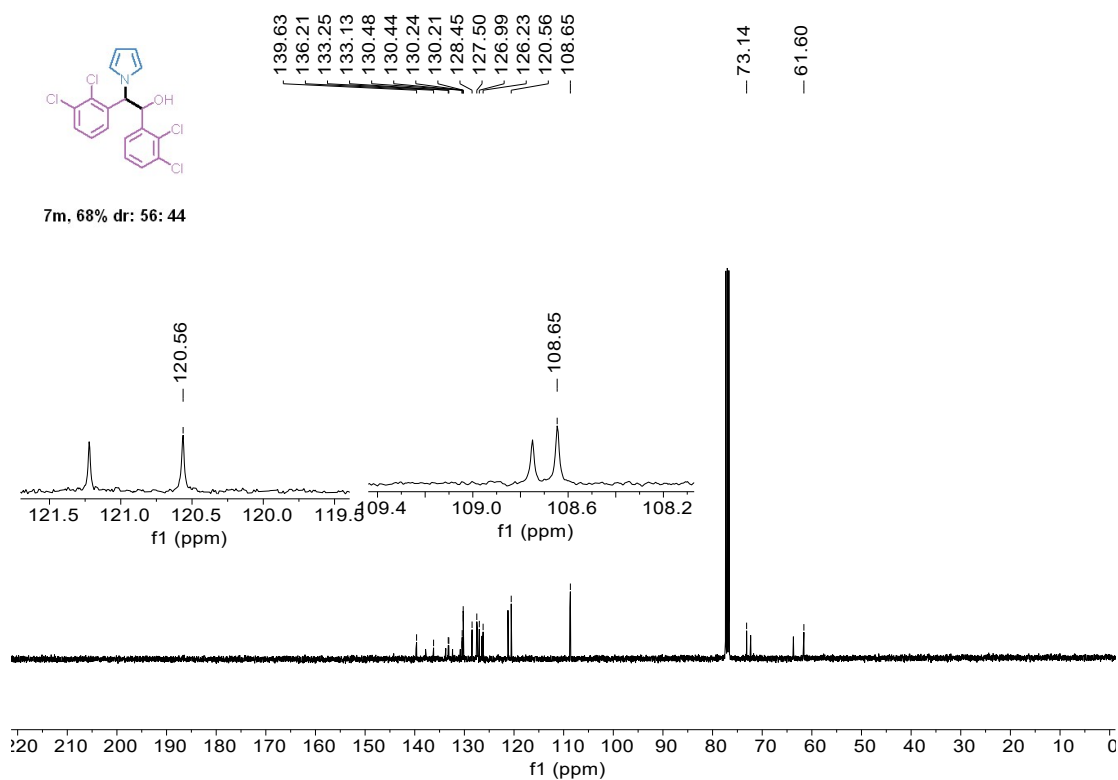
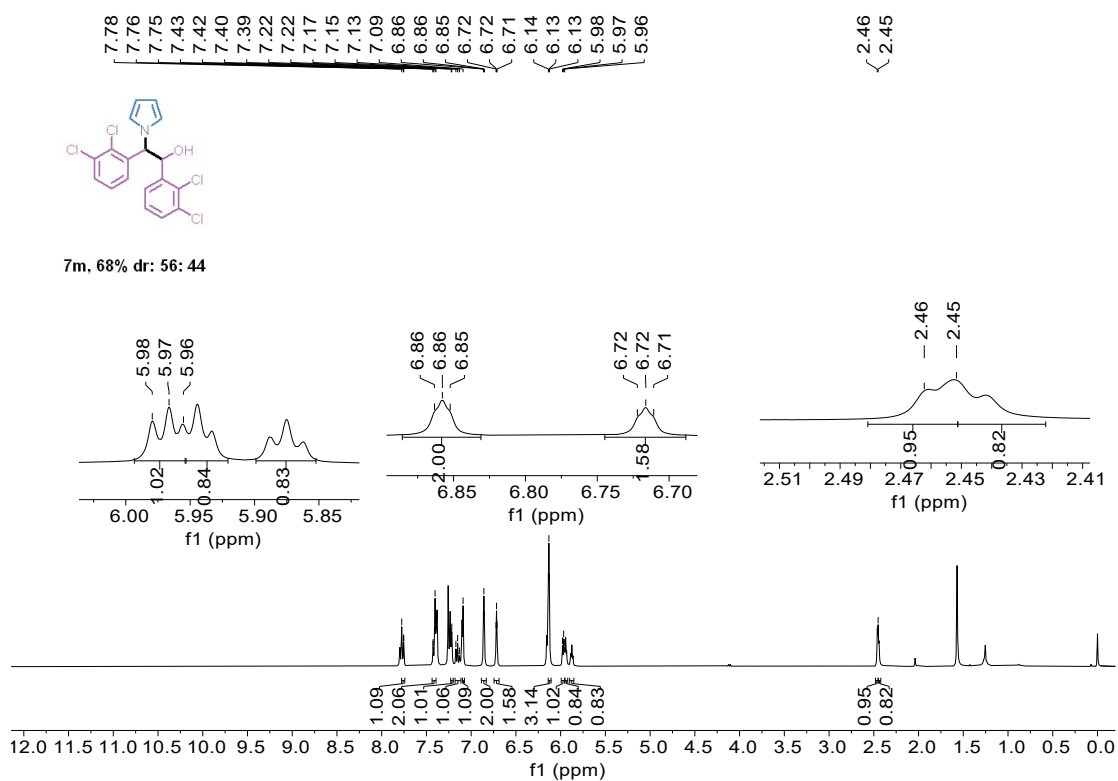
7k, dr: 54:46

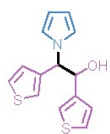


7k, dr: 54:46



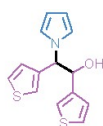
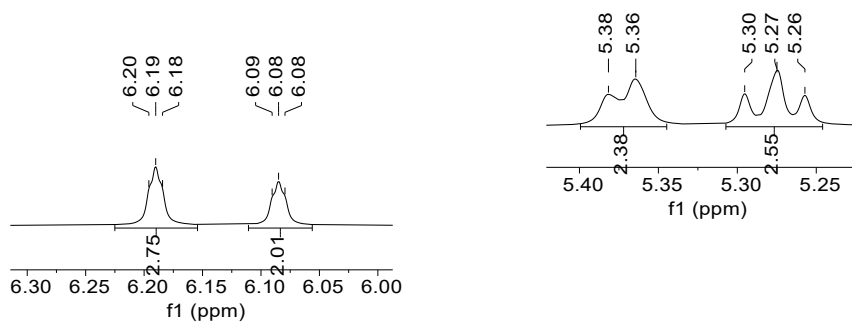






^1H NMR (400 MHz, CDCl_3) δ 7.31 – 7.25 (m, 2H), 7.23 (d, J = 3.1 Hz, 1H), 7.04 – 6.97 (m, 2H), 6.78 (d, J = 5.1 Hz, 1H), 6.65 (t, J = 2.2 Hz, 2H), 6.08 (t, J = 2.2 Hz, 2H), 5.37 (d, J = 6.9 Hz, 1H), 5.28 (t, J = 7.6 Hz, 1H), 2.33 (s, 1H).

7n, 27%, dr: 58: 42



^{13}C NMR (400 MHz, CDCl_3) δ 141.99, 138.16, 127.62, 126.22, 126.03, 125.52, 124.08, 122.11, 120.25, 108.30, 73.29, 64.86.

7n, 27%, dr: 58: 42

