Supporting Information for

Visible-Light-Induced, Graphene Oxide-Promoted C3-chalcogenylation of Indoles Strategy under Transition-Metal-Free Conditions

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CONTENTS

1. General Methods

^{1}H and ^{13}C NMR spectra were recorded on a Bruker spectrometers at 400 and 101 MHz, respectively. Mass spectra were recorded with Bruker Dalton Esquire 3000 plus LC-MS apparatus. Elemental analysis were carried out on a Perkin-Elmer 240B instrument. Silica gel (300-400 mesh) was used for flash column chromatography, eluting (unless otherwise stated) with an ethyl acetate/petroleum ether (PE) (60-90 °C) mixture.

Raman spectra were collected with a Horiba Jobin Y von-Labram HR UV-Visible-NIR Raman Microscope Spectrometer, using a 632 nm laser. The spectra were the average of 10 scans at a resolution of 2 cm^{-1} between 1000-2000 cm^{-1} Raman Shift.

2. Characterization of GO

GO was prepared by graphite oxidation using the Hummers and Offeman method and subsequent exfoliation. Further details and GO characterization have been previously reported.

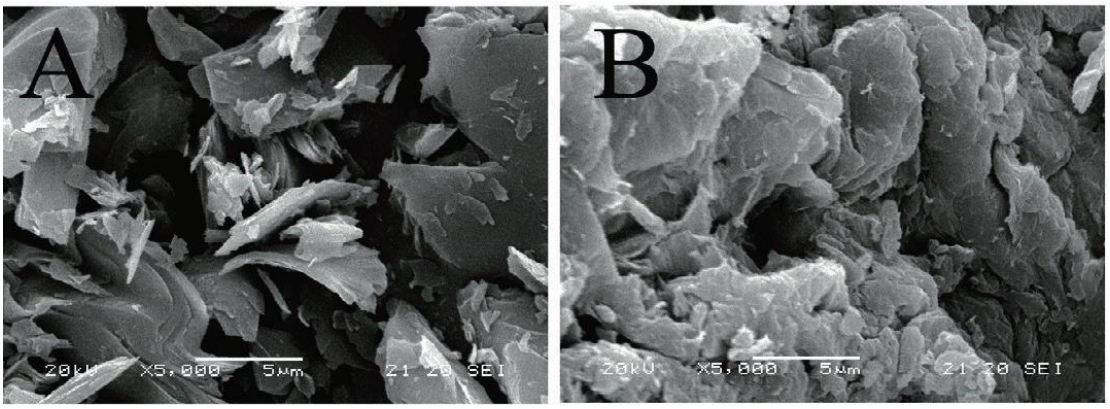


Figure S1. (A) SEM image of graphite. (B) SEM image of GO.

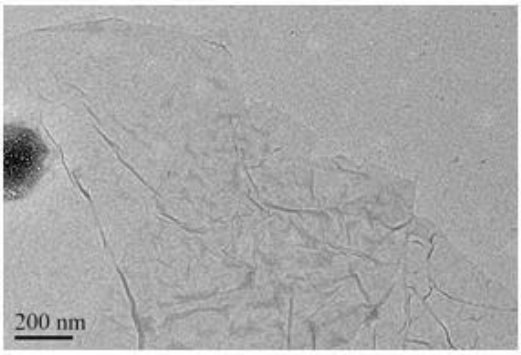


Figure S2. TEM image of graphite.

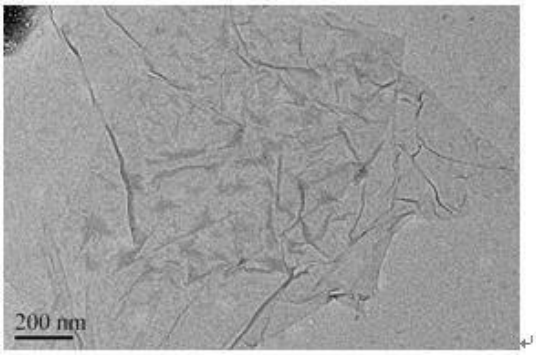


Figure S3. TEM image of GO.

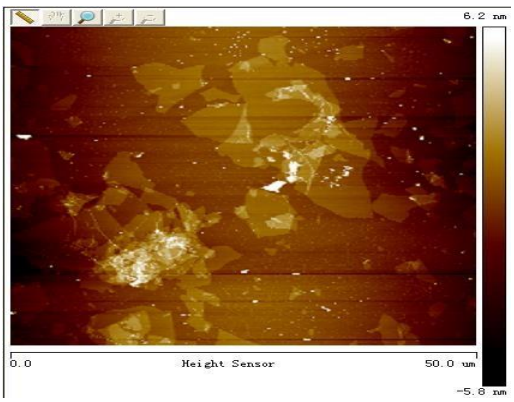


Figure S4. AFM image of GO.

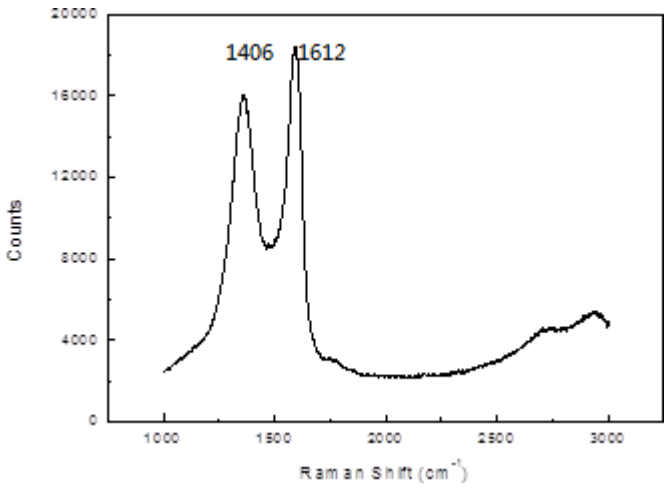


Figure S5. Raman image of GO (I_D/I_G ratio = 0.87).

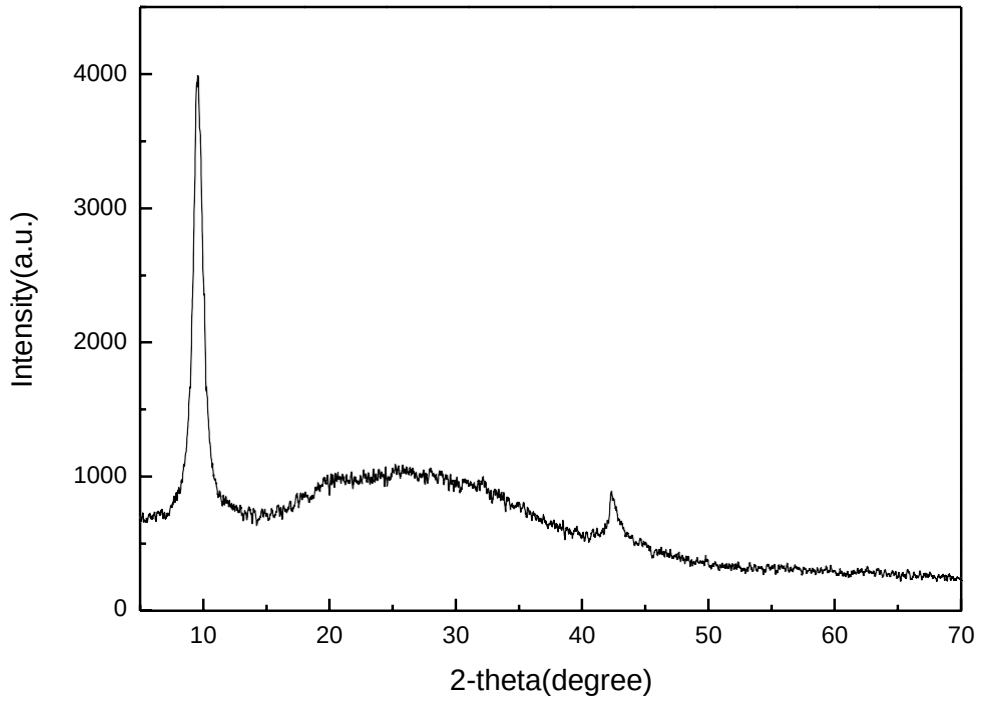
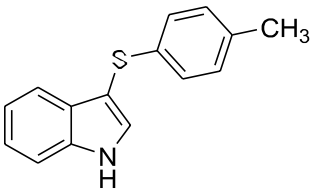


Figure S6. XRD image of GO.

3. General Procedure and Spectroscopic Data of the Products 6

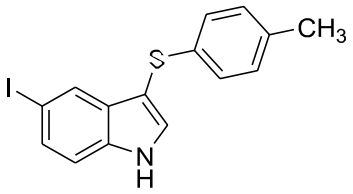
In a 10 mL Schlenk tube, indole (0.3 mmol), GO (17.6 mg), and thiol (0.36 mmol) were stirred in DCE (1 mL) for 12 h at room temperature under an air atmosphere irradiated by blue LEDs. The reaction mixture was concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (eluent: EtOAc/PE = 1:10) to yield the corresponding product **6**.

3-(*p*-Tolylthio)-1*H*-indole (**6aa**)



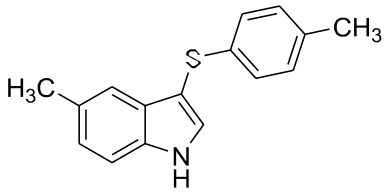
Yellow amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37 (s, 1H), 7.63 (d, J = 7.9 Hz, 1H), 7.46 (d, J = 2.6 Hz, 1H), 7.43 (d, J = 8.1 Hz, 1H), 7.26 (dt, J = 1.0, 8.1 Hz, 1H, Ar-H), 7.18 (t, J = 7.1 Hz, 1H), 7.05 (d, J = 8.3 Hz, 2H), 6.99 (d, J = 8.3 Hz, 2H), 2.26 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.5, 135.5, 134.7, 130.4, 129.5, 129.1, 126.3, 123.0, 120.8, 119.7, 111.5, 103.6, 20.8. MS (ESI): 240 ($M+H^+$, 100). These assignments matched with those previously published.[1]

5-Iodo-3-(*p*-tolylthio)-1*H*-indole (**6ba**)



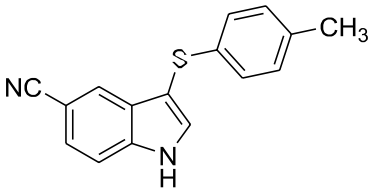
Brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.51 (s, 1H, NH), 7.99 (d, J = 1.5 Hz, 1H, Ar-H), 7.53 (dd, J = 8.5, 1.5 Hz, 1H, Ar-H), 7.43 (d, J = 1.5 Hz, 1H, Ar-H), 7.21 (d, J = 8.5 Hz, 1H, Ar-H), 7.06-7.01 (m, 4H, Ar-H), 2.29 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 135.6, 135.1, 134.9, 131.7, 131.4, 131.3, 129.6, 128.4, 126.3, 113.6, 102.8, 84.6, 20.9. MS (ESI): 366 ($M+H^+$, 100). These assignments matched with those previously published.[2]

5-Methyl-3-(*p*-tolylthio)-1*H*-indole (**6ca**)



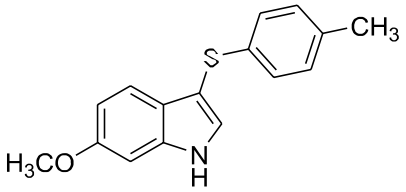
Yellow amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19 (s, 1H, NH), 7.55 (d, J = 0.7 Hz, 1H, Ar-H), 7.40 (d, J = 2.6 Hz, 1H, Ar-H), 7.34 (d, J = 8.2 Hz, 1H, Ar-H), 7.21-7.13 (m, 3H, Ar-H), 7.08 (d, J = 8.2 Hz, 2H, Ar-H), 2.52 (s, 3H, CH_3), 2.36 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 135.9, 134.9, 134.7, 130.94, 130.4, 129.7, 129.5, 126.2, 124.7, 119.2, 111.5, 102.4, 21.6, 21.0. MS (ESI): 254 ($M+H^+$, 100). These assignments matched with those previously published.[3]

3-(*p*-Tolylthio)-1*H*-indole-5-carbonitrile (**6da**)



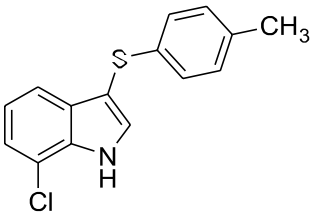
Yellow amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.20 (s, 1H, NH), 7.99 (d, J = 1.6 Hz, 1H, Ar-H), 7.81 (s, 1H, Ar-H), 7.66 (d, J = 8.4 Hz, 1H, Ar-H), 7.53 (d, J = 8.4 Hz, 1H, Ar-H), 7.04 (d, J = 8.0 Hz, 2H, Ar-H), 6.99 (d, J = 8.0 Hz, 2H, Ar-H), 2.20 (s, 3H, CH_3). ^{13}C NMR (101 MHz, DMSO-d_6): δ 139.1, 135.3, 135.2, 134.9, 130.1, 129.0, 126.9, 125.4, 124.1, 120.7, 114.3, 102.8, 102.5, 20.9. MS (ESI): 265 (M+H$^+$, 100). These assignments matched with those previously published.[3]

6-Methoxy-3-(*p*-tolylthio)-1*H*-indole (**6ea**)



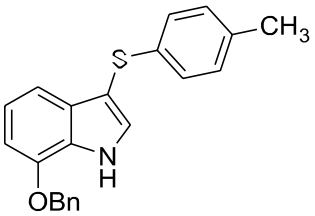
Reddish brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.33 (s, 1H, NH), 7.51 (d, J = 8.6 Hz, 1H, Ar-H), 7.34 (d, J = 2.2 Hz, 1H, Ar-H), 7.08 (d, J = 8.2 Hz, 2H, Ar-H), 7.02 (d, J = 8.2 Hz, 2H, Ar-H), 6.90 (d, J = 2.2 Hz, 1H, Ar-H), 6.86 (dd, J = 8.6, 2.2 Hz, 1H, Ar-H), 3.87 (s, 3H, OCH_3), 2.29 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 157.2, 137.3, 135.6, 134.7, 129.5, 129.3, 126.3, 123.3, 120.3, 110.8, 103.4, 95.2, 55.7, 20.9. MS (ESI): 270 (M+H$^+$, 100). These assignments matched with those previously published.[4]

7-Chloro-3-(*p*-tolylthio)-1*H*-indole (**6fa**)



Red amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.65 (s, 1H, NH), 7.55 (dt, J = 1.0, 7.8 Hz, 1H, Ar-H), 7.54 (d, J = 2.6 Hz, 1H, Ar-H), 7.29 (dd, J = 7.8, 1.0 Hz, 1H, Ar-H), 7.12 (t, J = 7.8 Hz, 1H, Ar-H), 7.08 (d, J = 8.2 Hz, 2H, Ar-H), 7.03 (d, J = 8.2 Hz, 2H, Ar-H), 2.30 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 135.0, 134.9, 133.8, 130.9, 130.6, 129.6 , 126.6, 122.4, 121.6, 118.4, 117.0, 105.2, 20.9. MS (ESI): 274 (M+H$^+$, 30), 276 (M+H$^+$, 100). Anal calcd for $C_{15}H_{12}ClNS$: C, 65.81; H, 4.42; N, 5.12; S, 11.71. Found C, 65.46; H, 4.81; N, 4.83; S, 11.62.

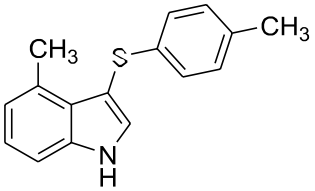
7-(Benzyloxy)-3-(*p*-tolylthio)-1*H*-indole (**6ga**)



Reddish brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.71 (s, 1H, NH), 7.52 (d, J = 7.1 Hz, 2H, Ar-H), 7.49-7.40 (m, 4H, Ar-H), 7.27 (d, J = 8.4 Hz, 1H, Ar-H), 7.09 (t, J = 7.8 Hz, 1H, Ar-H), 7.07 (d, J = 8.0 Hz, 2H, Ar-H), 7.01 (d, J = 8.0 Hz, 2H, Ar-H), 6.81 (d, J = 7.8 Hz, 1H, Ar-H), 5.24 (s, 2H, OCH_2), 2.28 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 145.6, 136.9, 135.7, 134.6, 130.8, 130.1, 129.5, 128.7, 128.3, 128.0, 127.2, 126.3, 121.2, 112.6, 104.0 , 103.7, 70.4,

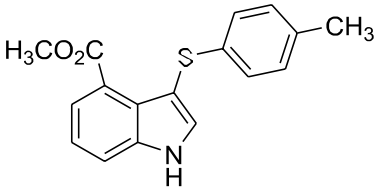
20.9. MS (ESI): 346 ($M+H^+$, 100). Anal calcd for $C_{22}H_{19}NOS$: C, 76.49; H, 5.54; N, 4.05; S, 9.28. Found C, 76.35; H, 5.47; N, 4.33; S, 8.95.

4-Methyl-3-(*p*-tolylthio)-1*H*-indole (**6ha**)



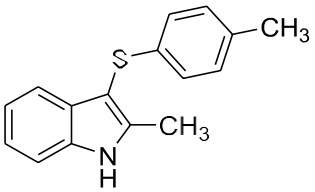
Reddish brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (s, 1H, NH), 7.43 (d, J = 2.6 Hz, 1H, Ar-H), 7.29 (d, J = 8.1 Hz, 1H, Ar-H), 7.17 (t, J = 8.1 Hz, 1H, Ar-H), 7.06-6.98 (m, 4H, Ar-H), 6.92 (d, J = 7.1 Hz, 1H), 2.70 (s, 3H, CH_3), 2.29 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 137.9, 137.0, 134.3, 132.2, 131.7, 129.6, 127.0, 125.5, 123.1, 122.4, 109.4, 102.9, 20.9, 18.7. MS (ESI): 254 ($M+H^+$, 100). These assignments matched with those previously published.[3]

Methyl 3-(*p*-tolylthio)-1*H*-indole-4-carboxylate (**6ia**)



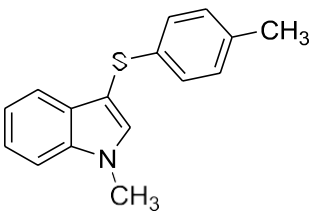
Brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.19 (s, 1H, NH), 7.51 (d, J = 2.6 Hz, 1H, Ar-H), 7.49 (d, J = 1.0 Hz, 1H, Ar-H), 7.38 (d, J = 2.6 Hz, 1H, Ar-H), 7.24 (t, J = 7.8 Hz, 1H, Ar-H), 6.98 (s, 4H, Ar-H), 3.68 (s, 3H), 2.30 (d, J = 37.7 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 169.6, 137.6, 136.4, 134.6, 133.4, 129.4, 126.2, 125.4, 125.3, 122.1, 122.0, 115.1, 103.1, 51.9, 20.8. MS (ESI): 298 ($M+H^+$, 100). Anal calcd for $C_{17}H_{15}NO_2S$: C, 68.66; H, 5.08; N, 4.71; S, 10.78. Found C, 68.80; H, 5.26; N, 4.64; S, 10.57.

2-Methyl-3-(*p*-tolylthio)-1*H*-indole (**6ja**)



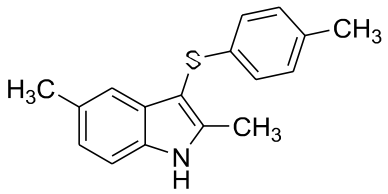
Reddish brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26 (s, 1H, NH), 7.58 (d, J = 7.8 Hz, 1H, Ar-H), 7.36 (dt, J = 1.0, 7.8 Hz, 1H, Ar-H), 7.22 (dt, J = 1,0, 7.8 Hz, 1H, Ar-H), 7.15 (dt, J = 1.0, 7.8 Hz, 1H, Ar-H), 6.99 (s, 4H, Ar-H), 2.54 (s, 3H, CH_3), 2.27 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.0, 135.7, 135.5, 134.4, 130.4, 129.5, 125.8 , 122.1, 120.7, 119.0, 110.7, 99.9, 20.9, 12.2. MS (ESI): 254 ($M+H^+$, 100). These assignments matched with those previously published.[3]

1-Methyl-3-(*p*-tolylthio)-1*H*-indole (**6ka**)



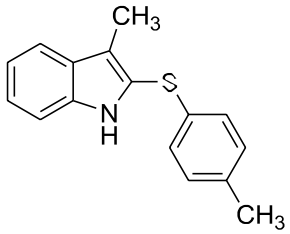
Reddish brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, J = 8.2 Hz, 1H, Ar-H), 7.45 (d, J = 8.2 Hz, 1H, Ar-H), 7.39 (dt, J = 1.0, 7.0 Hz, 1H, Ar-H), 7.37 (s, 1H, Ar-H), 7.28 (dt, J = 1.0, 7.0 Hz, 1H, Ar-H), 7.15 (d, J = 8.2 Hz, 2H, Ar-H), 7.07 (d, J = 8.2 Hz, 1H, Ar-H), 3.86 (s, 3H, NCH_3), 2.35 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 137.6, 136.1, 134.9, 134.6, 123.0, 129.6, 126.3, 126.2, 122.6, 120.5, 119.8, 109.8, 101.3, 33.1, 21.0. MS (ESI): 254 ($M+H^+$, 100). These assignments matched with those previously published.[2]

2,5-Dimethyl-3-(*p*-tolylthio)-1*H*-indole (**6la**)



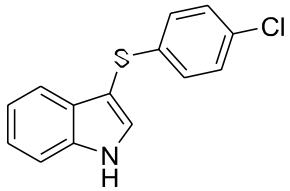
Reddish brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (s, 1H, NH), 7.46 (s, 1H, Ar-H), 7.25 (d, J = 8.2 Hz, 1H, Ar-H), 7.10 (dd, J = 8.2, 1.2 Hz, 1H, Ar-H), 7.46 (s, 4H, Ar-H), 2.51 (s, 3H, CH_3), 2.49 (s, 3H, CH_3), 2.34 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.3, 136.0, 134.3, 133.8, 130.7, 130.1, 129.6, 125.7, 123.7, 118.7, 110.5, 99.0, 21.5, 20.9, 12.1. MS (ESI): 268 ($M+H^+$, 100). These assignments matched with those previously published.[3]

3-Methyl-2-(*p*-tolylthio)-1*H*-indole (**6ma**)



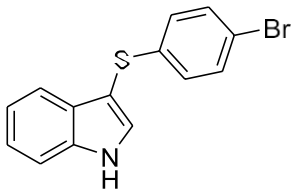
Reddish brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (s, 1H, NH), 7.71 (d, J = 7.9 Hz, 1H, Ar-H), 7.33 (d, J = 3.6 Hz, 2H, Ar-H), 7.26 (m, 1H, Ar-H), 7.12 (d, J = 8.3 Hz, 2H, Ar-H), 7.09 (d, J = 8.3 Hz, 2H, Ar-H), 2.51 (s, 3H, CH_3), 2.38 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.9, 135.9, 133.5, 130.0, 128.6, 127.1, 123.4, 122.3, 119.7, 119.5, 119.4, 111.0, 21.0, 9.6. MS (ESI): 254 ($M+H^+$, 100). These assignments matched with those previously published.[3]

3-((4-Chlorophenyl)thio)-1*H*-indole (**6ab)**



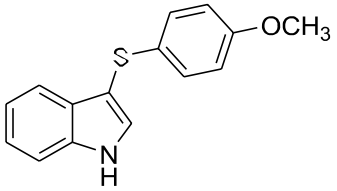
Light yellow amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.47 (s, 1H, NH), 7.58 (d, J = 8.0 Hz, 1H, Ar-H), 7.49 (d, J = 2.6 Hz, 1H, Ar-H), 7.45 (d, J = 8.2 Hz, 1H, Ar-H), 7.29 (dt, J = 1.0, 8.0 Hz, 1H, Ar-H), 7.18 (dt, J = 1.0, 8.0 Hz, 1H, Ar-H), 7.12 (d, J = 8.7 Hz, 2H, Ar-H), 7.02 (d, J = 8.7 Hz, 2H, Ar-H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 137.8, 136.5, 130.6, 130.5, 128.7, 128.6, 127.1, 123.1, 121.0, 119.4, 111.6, 102.4. MS (ESI): 260 ($M+H^+$, 30), 262 ($M+H^+$, 100). These assignments matched with those previously published.[2]

3-((4-Bromophenyl)thio)-1*H*-indole (**6ac**)



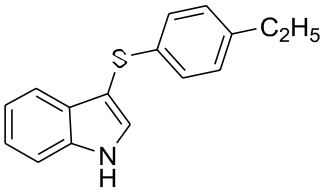
Brown amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.76 (s, 1H, NH), 7.80 (d, J = 2.7 Hz, 1H, Ar-H), 7.51 (d, J = 8.1 Hz, 1H, Ar-H), 7.41 – 7.35 (m, 3H, Ar-H), 7.20 (dt, J = 1.1, 8.1 Hz, 1H, Ar-H), 7.08 (dt, J = 1.1, 8.1 Hz, 1H, Ar-H), 6.96 (dt, J = 2.7, 8.6 Hz, 2H, Ar-H). ^{13}C NMR (101 MHz, DMSO-d_6): δ 139.5, 137.3, 133.1, 132.1, 128.9, 127.7, 122.7, 120.7, 118.6, 118.0, 112.9, 99.1. MS (ESI): 304 (M+H$^+$, 100), 306 (M+H$^+$, 100). These assignments matched with those previously published.[2]

3-((4-Methoxyphenyl)thio)-1*H*-indole (**6ad**)



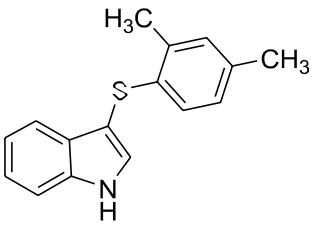
Brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.38 (s, 1H, NH), 7.63 (d, J = 8.0 Hz, 1H, Ar-H), 7.46 (d, J = 2.6 Hz, 1H, Ar-H), 7.41 (d, J = 8.0 Hz, 1H, Ar-H), 7.25 (dt, J = 1.0, 8.0 Hz, 1H, Ar-H), 7.17 (dt, J = 1.0, 8.0 Hz, 1H, Ar-H), 7.13 (d, J = 8.9 Hz, 2H, Ar-H), 6.74 (d, J = 8.9 Hz, 2H, Ar-H), 3.73 (s, 3H, OCH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 157.8, 136.5, 123.0, 129.5, 129.0, 128.6, 122.9, 120.8, 119.7, 114.5, 111.5, 104.7, 55.3. MS (ESI): 256 (M+H$^+$, 100). These assignments matched with those previously published.[1]

3-((4-Ethylphenyl)thio)-1*H*-indole (**6ae**)



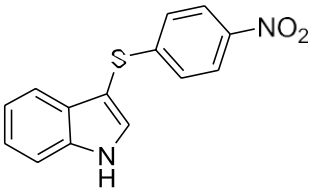
Reddish brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.34 (s, 1H, NH), 7.71 (d, J = 8.0 Hz, 1H, Ar-H), 7.45 (d, J = 2.3 Hz, 1H, Ar-H), 7.44 (d, J = 8.0 Hz, 1H, Ar-H), 7.32 (dt, J = 1.0, 8.0 Hz, 1H, Ar-H), 7.23 (t, J = 7.5 Hz, 1H, Ar-H), 7.13 (d, J = 8.2 Hz, 2H, Ar-H), 7.06 (d, J = 8.2 Hz, 2H, Ar-H), 2.61 (q, J = 7.6 Hz, 2H, CH_2), 1.23 (t, J = 7.6 Hz, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.3,136.5, 135.9, 130.7, 129.2, 128.5, 126.3, 123.1, 120.9, 119.7, 111.8, 103.2, 28.4, 15.7. MS (ESI): 254 (M+H$^+$, 100). Anal calcd for $C_{16}H_{15}NS$: C, 75.85; H, 5.97; N, 5.53; S, 12.65. Found C, 75.59; H, 5.63; N, 5.71; S, 12.32.

3-((2,4-Dimethylphenyl)thio)-1*H*-indole (**6af**)



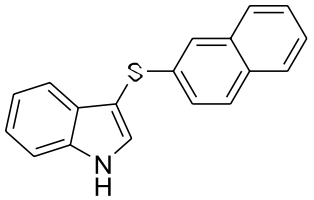
Tawny amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.28 (s, 1H, NH), 7.71 (d, J = 8.0 Hz, 1H, Ar-H), 7.45 (d, J = 8.0 Hz, 1H, Ar-H), 7.41 (d, J = 2.6 Hz, 1H, Ar-H), 7.36 (t, J = 7.7 Hz, 1H, Ar-H), 7.27 (t, J = 7.7 Hz, 1H, Ar-H), 7.09 (s, 1H, Ar-H), 6.83 (d, J = 8.0 Hz, 1H, Ar-H), 6.78 (d, J = 8.0 Hz, 1H, Ar-H), 2.59 (s, 3H, CH_3), 2.34 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.6, 134.7, 134.6, 134.4, 130.9, 130.5, 129.3, 127.1, 126.1, 123.0, 120.8, 119.7, 111.6, 103.0, 20.7, 19.9. MS (ESI): 254 ($M+H^+$, 100). Anal calcd for $C_{16}H_{15}NS$: C, 75.85; H, 5.97; N, 5.53; S, 12.65. Found C, 76.04; H, 5.83; N, 5.66; S, 12.35.

3-((4-Nitrophenyl)thio)-1*H*-indole (**6ag**)



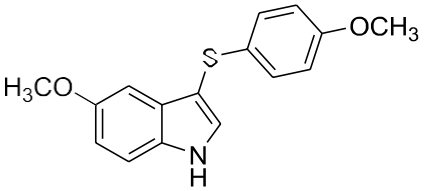
Reddish brown amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.86 (s, 1H, NH), 8.02 (d, J = 9.0 Hz, 2H, Ar-H), 7.55 (t, J = 7.7 Hz, 1H, Ar-H), 7.54 (d, J = 2.6 Hz, 1H, Ar-H), 7.53 (d, J = 8.1 Hz, 1H, Ar-H), 7.34 (t, J = 8.1 Hz, 1H, Ar-H), 7.22 (t, J = 7.7 Hz, 1H, Ar-H), 7.15 (d, J = 9.0 Hz, 2H, Ar-H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 150.0, 144.9, 136.7, 131.4, 128.5, 125.2, 123.9, 123.5, 121.4, 119.2, 112.1, 100.1. MS (ESI): 271 ($M+H^+$, 100). These assignments matched with those previously published.[5]

3-(Naphthalen-2-ylthio)-1*H*-indole (**6ah**)



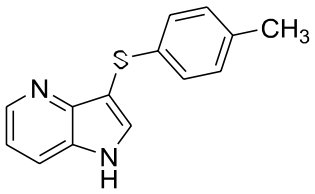
Reddish brown amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.74 (s, 1H, NH), 7.83 (d, J = 2.5 Hz, 1H, Ar-H), 7.77 (d, J = 8.6 Hz, 1H, Ar-H), 7.73 (d, J = 8.7 Hz, 1H, Ar-H), 7.61 (d, J = 7.5 Hz, 1H, Ar-H), 7.50 (d, J = 2.6 Hz, 1H, Ar-H), 7.48 (s, 1H, Ar-H), 7.40 – 7.33 (m, 3H, Ar-H), 7.20 (dd, J = 2.6 Hz, 1H, Ar-H), 7.17 (t, J = 2.6 Hz, 1H, Ar-H), 7.03 (t, J = 7.5 Hz, 1H, Ar-H). ^{13}C NMR (101 MHz, DMSO-d_6): δ 137.3, 137.2, 133.7, 133.0, 131.3, 129.1, 128.8, 128.1, 127.1, 127.0, 125.7, 124.9, 123.3, 122.6, 120.6, 118.8, 112.9, 99.8. MS (ESI): 276 ($M+H^+$, 100). These assignments matched with those previously published.[3]

5-Methoxy-3-((4-methoxyphenyl)thio)-1*H*-indole (**6ai**).



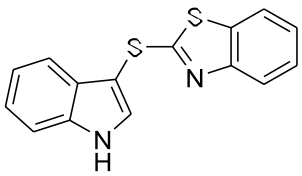
Red amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.42 (s, 1H), 7.40 (d, J = 2.6 Hz, 1H), 7.28 (d, J = 8.8 Hz, 1H), 7.17 (dt, J = 2.6, 8.8 Hz, 2H), 7.13 (d, J = 2.4 Hz, 1H), 6.95 (dd, J = 8.8, 2.4 Hz, 1H), 6.80 (dt, J = 2.6, 8.8 Hz, 2H), 3.84 (s, 3H), 3.76 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 157.8, 155.0, 131.5, 131.0, 129.9, 129.8, 128.3, 114.6, 113.4, 112.5, 103.7, 104.0, 55.9, 55.4. These assignments matched with those previously published.[6]

3-(*p*-Tolylthio)-1*H*-pyrrolo[3,2-*b*]pyridine (**6aj**).



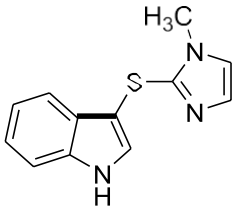
Light yellow amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.86 (s, 1H, NH), 8.36 (d, J = 4.0 Hz, 1H, Ar-H), 8.00 (d, J = 2.6 Hz, 1H, Ar-H), 7.86 (d, J = 8.1 Hz, 1H, Ar-H), 7.19 (dd, J = 8.1, 4.5 Hz, 1H, Ar-H), 7.00 (d, J = 7.2 Hz, 2H, Ar-H), 6.96 (d, J = 7.2 Hz, 2H, Ar-H), 2.19 (s, 3H, CH_3). ^{13}C NMR (101 MHz, DMSO-d_6): δ 146.0, 143.8, 136.2, 135.9, 134.5, 129.8, 129.7, 126.5, 120.0, 117.7, 101.6, 20.9. MS (ESI): 241 ($M+H^+$, 100). Anal calcd for $C_{14}H_{12}N_2S$: C, 69.97; H, 5.03; N, 11.66; S, 13.34. Found C, 70.21; H, 5.37; N, 11.31; S, 13.15.

2-((1*H*-indol-3-yl)thio)benzo[*d*]thiazole (**6ak**)



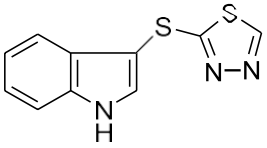
Brown amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.03 (s, 1H, NH), 8.04 (d, J = 2.8 Hz, 1H, Ar-H), 7.82 (dd, J = 2.8, 1.8 Hz, 1H, Ar-H), 7.80 (dd, J = 2.1, 1.0 Hz, 1H, Ar-H), 7.57 (d, J = 7.8 Hz, 1H, Ar-H), 7.56 (d, J = 7.8 Hz, 1H, Ar-H), 7.41 (dt, J = 1.2, 8.4 Hz, 1H, Ar-H), 7.30-7.23 (m, 2H, Ar-H), 7.15 (dt, J = 1.0, 7.1 Hz, 1H, Ar-H). ^{13}C NMR (101 MHz, DMSO-d_6): δ 173.8, 154.6, 137.2, 135.4, 134.4, 128.4, 126.6, 124.4, 123.1, 122.1, 121.6, 121.3, 118.5, 113.1, 97.7. MS (ESI): 283 ($M+H^+$, 100). These assignments matched with those previously published.[7]

3-((1-Methyl-1*H*-imidazol-2-yl)thio)-1*H*-indole (**6al**)



Yellow amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.51 (s, 1H, NH), 7.69 (s, 1H, Ar-H), 7.60 (d, J = 7.6 Hz, 1H, Ar-H), 7.39 (d, J = 7.6 Hz, 1H, Ar-H), 7.16 (s, 1H, Ar-H), 7.10 (t, J = 7.1 Hz, 1H, Ar-H), 7.04 (t, J = 7.1 Hz, 1H, Ar-H), 6.85 (s, 1H, Ar-H), 3.66 (s, 3H, CH_3). ^{13}C NMR (101 MHz, DMSO-d_6): δ 140.0, 136.7, 131.0, 128.9, 128.5, 124.0, 122.4, 120.3, 119.1, 112.5, 100.6, 34.0. MS (ESI): 230 ($M+H^+$, 100). Anal calcd for $C_{12}H_{11}N_3S$: C, 62.86; H, 4.84; N, 18.33; S, 13.98. Found C, 63.10; H, 5.07; N, 18.05; S, 13.61.

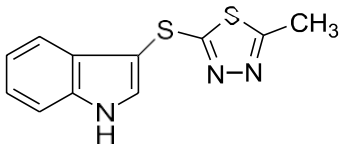
2-((1*H*-indol-3-yl)thio)-1,3,4-thiadiazole (**6am**)



Yellow amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.98 (s, 1H, NH), 9.29 (s, 1H, Ar-H), 8.00 (d, J = 2.8 Hz, 1H, Ar-H), 7.56 (d, J = 8.0 Hz, 1H, Ar-H), 7.53 (d, J = 7.9 Hz, 1H, Ar-H), 7.25 (dt, J = 1.0, 8.0 Hz, 1H, Ar-H), 7.16 (d, J = 7.9 Hz, 1H, Ar-H). ^{13}C NMR (101 MHz,

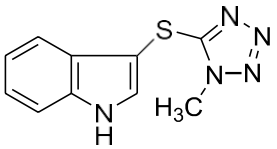
DMSO-d_6): δ 173.0, 154.3, 137.2, 133.6, 127.8, 123.2, 121.3, 118.4, 113.2, 98.6. MS (ESI): 234 ($M+H^+$, 100). Anal calcd for $C_{10}H_7N_3S_2$: C, 51.48; H, 3.02; N, 18.01; S, 27.48. Found C, 51.83; H, 3.39; N, 17.85; S, 27.17.

2-((1*H*-indol-3-yl)thio)-5-methyl-1,3,4-thiadiazole (**6an**)



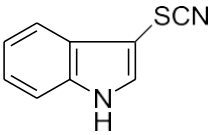
Yellow amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.95 (s, 1H, NH), 7.96 (d, J = 2.7 Hz, 1H, Ar-H), 7.55 (d, J = 2.7 Hz, 1H, Ar-H), 7.53 (d, J = 2.7 Hz, 1H, Ar-H), 7.24 (d, J = 7.5 Hz, 1H, Ar-H), 7.16 (d, J = 7.5 Hz, 1H, Ar-H), 2.50 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 172.4, 165.7, 137.1, 133.5, 128.0, 123.1, 121.3, 118.4, 113.1, 98.8, 15.6. MS (ESI): 248 ($M+H^+$, 100). Anal calcd for $C_{11}H_9N_3S_2$: C, 53.42; H, 3.67; N, 16.99; S, 25.92. Found C, 53.76; H, 3.92; N, 16.84; S, 25.59.

3-((1-Methyl-1*H*-tetrazol-5-yl)thio)-1*H*-indole (**6ao**)



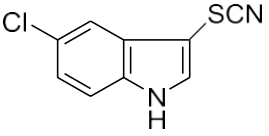
Pink amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.85 (s, 1H, NH), 7.94 (d, J = 2.7 Hz, 1H, Ar-H), 7.54 (d, J = 8.7 Hz, 1H, Ar-H), 7.51 (d, J = 8.7 Hz, 1H, Ar-H), 7.22 (d, J = 7.2 Hz, 1H, Ar-H), 7.13 (d, J = 7.2 Hz, 1H, Ar-H), 4.03 (s, 3H, NCH_3). ^{13}C NMR (101 MHz, DMSO-d_6): δ 153.8, 136.9, 133.5, 128.8, 122.9, 121.0, 118.6, 112.9, 94.5, 34.5. MS (ESI): 232 ($M+H^+$, 100). Anal calcd for $C_{10}H_9N_5S$: C, 51.93; H, 3.92; N, 30.28; S, 13.86. Found C, 52.20; H, 4.28; N, 29.91; S, 13.93.

3-Thiocyanato-1*H*-indole (**7a**)



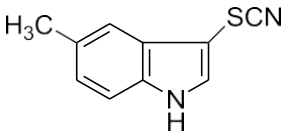
White amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.76 (s, 1H, NH), 7.83 (dd, J = 5.9, 3.1 Hz, 1H, Ar-H), 7.52 (d, J = 2.8 Hz, 1H, Ar-H), 7.45 (dt, J = 5.9, 3.1 Hz, 1H, Ar-H), 7.35 (t, J = 3.1 Hz, 1H, Ar-H), 7.33 (t, J = 3.1 Hz, 1H, Ar-H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.0, 131.0, 127.7, 123.9, 121.9, 118.8, 112.1, 111.9, 92.3. MS (ESI): 175 ($M+H^+$, 100). These assignments matched with those previously published.[8]

5-Chloro-3-thiocyanato-1-*H*-indole (**7b**).



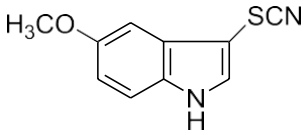
Yellow amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.20 (s, 1H, NH), 8.05 (s, 1H, Ar-H), 7.66 (s, 1H, Ar-H), 7.55 (d, J = 7.7 Hz, 1H, Ar-H), 7.26 (d, J = 7.7 Hz, 1H, Ar-H). ^{13}C NMR (101 MHz, DMSO-d_6): δ 135.3, 135.2, 129.1, 126.4, 123.5, 117.4, 115.0, 112.5, 89.9. MS (ESI): 209 ($M+H^+$, 100), 211 ($M+H^+$, 32). These assignments matched with those previously published.[9]

5-Methyl-3-thiocyanato-1-*H*-indole (**7c**).



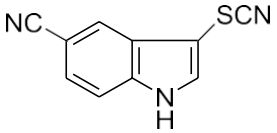
White amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.00 (s, 1H, NH), 7.94 (d, J = 2.9 Hz, 1H, Ar-H), 7.35 (d, J = 8.2 Hz, 1H, Ar-H), 7.12 (t, J = 8.2 Hz, 1H, Ar-H), 6.93 (d, J = 7.1 Hz, 1H, Ar-H), 2.85 (s, 3H, CH_3). ^{13}C NMR (101 MHz, DMSO-d_6): δ 137.2, 134.6, 129.9, 125.7, 123.4, 123.0, 114.3, 111.3, 89.8, 19.2. MS (ESI): 189 ($M+H^+$, 100). These assignments matched with those previously published.[10]

5-Methoxy-3-thiocyanato-1*H*-indole (**7d**)



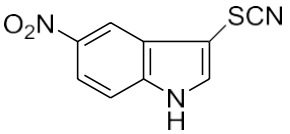
White amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (s, 1H, NH), 7.67 (d, J = 8.7 Hz, 1H, Ar-H), 7.40 (d, J = 2.7 Hz, 1H, Ar-H), 6.97 (dd, J = 8.7, 2.1 Hz, 1H, Ar-H), 6.90 (d, J = 2.1 Hz, 1H, Ar-H), 3.86 (s, 3H, OCH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 157.7, 136.9, 129.8, 121.8, 119.5, 112.1, 111.9, 95.2, 92.3, 55.7. MS (ESI): 205 ($M+H^+$, 100). These assignments matched with those previously published.[8]

3-Thiocyanato-1-*H*-indole-5-carbonitrile (**7e**).



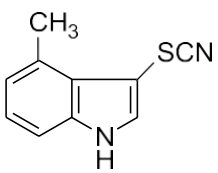
Yellow amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.52 (s, 1H, NH), 8.23 (d, J = 2.6 Hz, 1H, Ar-H), 8.21 (s, 1H, Ar-H), 7.71 (d, J = 8.5 Hz, 1H, Ar-H), 7.64 (dd, J = 8.6, 2.6 Hz, 1H, Ar-H). ^{13}C NMR (101 MHz, DMSO-d_6): δ 143.4, 141.1, 132.5, 130.9, 128.6, 125.1, 119.5, 117.2, 108.7, 96.8. MS (ESI): 200 ($M+H^+$, 100). These assignments matched with those previously published.[9]

5-Nitro-3-thiocyanato-1-*H*-indole (**7f**).



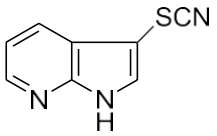
Yellow amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.63 (s, 1H, NH), 8.51 (d, J = 1.8 Hz, 1H, Ar-H), 8.27 (s, 1H, Ar-H), 8.12 (dd, J = 9.0, 1.8 Hz, 1H, Ar-H), 7.70 (d, J = 9.0 Hz, 1H, Ar-H). ^{13}C NMR (101 MHz, DMSO-d_6): δ 142.6, 140.0, 137.5, 127.3, 118.6, 114.8, 114.1, 112.4, 93.6. MS (ESI): 220 ($M+H^+$, 100). These assignments matched with those previously published.[9]

4-Methyl-3-thiocyanato-1-*H*-indole (**7g**).



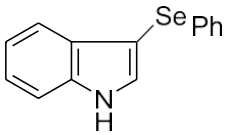
White amorphous solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.91 (s, 1H, NH), 7.92 (d, J = 2.9 Hz, 1H, Ar-H), 7.46 (s, 1H, Ar-H), 7.43 (d, J = 8.3 Hz, 1H, Ar-H), 7.10 (dd, J = 8.3, 1.1 Hz, 1H, Ar-H), 2.45 (s, 3H, CH_3). ^{13}C NMR (101 MHz, DMSO-d_6): δ 135.1, 133.5, 130.5, 128.2, 125.0, 117.7, 113.0, 112.8, 89.0, 21.6. MS (ESI): 189 ($M+H^+$, 100). These assignments matched with those previously published.[9]

3-Thiocyanato-1*H*-pyrrolo[2,3-*b*]pyridine (**7h**)



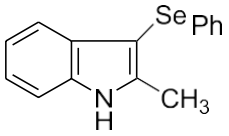
White amorphous solid. ^{1}H NMR (400 MHz, DMSO): δ 12.62 (s, 1H, NH), 8.40 (d, J = 4.5 Hz, 1H, Ar-H), 8.18 (s, 1H, Ar-H), 8.13 (d, J = 7.8 Hz, 1H, Ar-H), 7.31 (dd, J = 7.8, 4.7 Hz, 1H, Ar-H). ^{13}C NMR (101 MHz, DMSO): δ 148.8, 145.0, 134.4, 127.0, 120.3, 117.8, 112.6, 89.5. MS (ESI): 176 ($M+H^+$, 100). These assignments matched with those previously published.[11]

3-(Phenylselanyl)-1*H*-indole (**9a**)



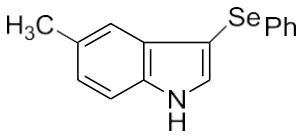
Yellow amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (s, 1H, NH), 7.69 (d, J = 7.9 Hz, 1H, Ar-H), 7.49 (d, J = 2.5 Hz, 1H, Ar-H), 7.46 (d, J = 8.2 Hz, 1H, Ar-H), 7.33-7.27 (m, 3H, Ar-H), 7.24-7.12 (m, 4H, Ar-H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.4, 133.9, 131.3, 130.0, 129.0, 128.7, 125.6, 123.0, 120.9, 120.4, 111.4, 98.2. MS (ESI): 274 ($M+H^+$, 100). These assignments matched with those previously published.[12]

2-Methyl-3-(phenylselanyl)-1*H*-indole (**9b**)



Yellow amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20 (s, 1H, NH), 7.64 (d, J = 7.7 Hz, 1H, Ar-H), 7.36 (d, J = 7.9 Hz, 1H, Ar-H), 7.26 (dd, J = 3.5, 1.4 Hz, 1H, Ar-H), 7.23 (dd, J = 3.5, 1.4 Hz, 2H, Ar-H), 7.20 (d, J = 4.1 Hz, 1H, Ar-H), 7.19-7.12 (m, 3H, Ar-H), 2.56 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.0, 135.8, 134.0, 131.3, 129.0, 128.4, 125.5, 122.2, 120.7, 119.8, 110.6, 96.2, 13.2. MS (ESI): 288 ($M+H^+$, 100). These assignments matched with those previously published.[13]

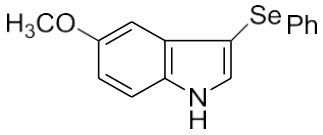
5-Methyl-3-(phenylselanyl)-1*H*-indole (**9c**)



Yellow amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.30 (s, 1H, NH), 7.51 (d, J = 0.5 Hz, 1H, Ar-H), 7.44 (d, J = 2.5 Hz, 1H, Ar-H), 7.35 (d, J = 8.3 Hz, 1H, Ar-H), 7.31-7.28 (m, 2H, Ar-H), 7.22-7.14 (m, 4H, Ar-H), 2.49 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 134.7, 134.1, 131.5,

130.4, 130.3, 129.0, 128.6, 125.6, 124.7, 119.9, 111.1, 97.4, 21.5. MS (ESI): 288 ($M+H^+$, 100). These assignments matched with those previously published.[14]

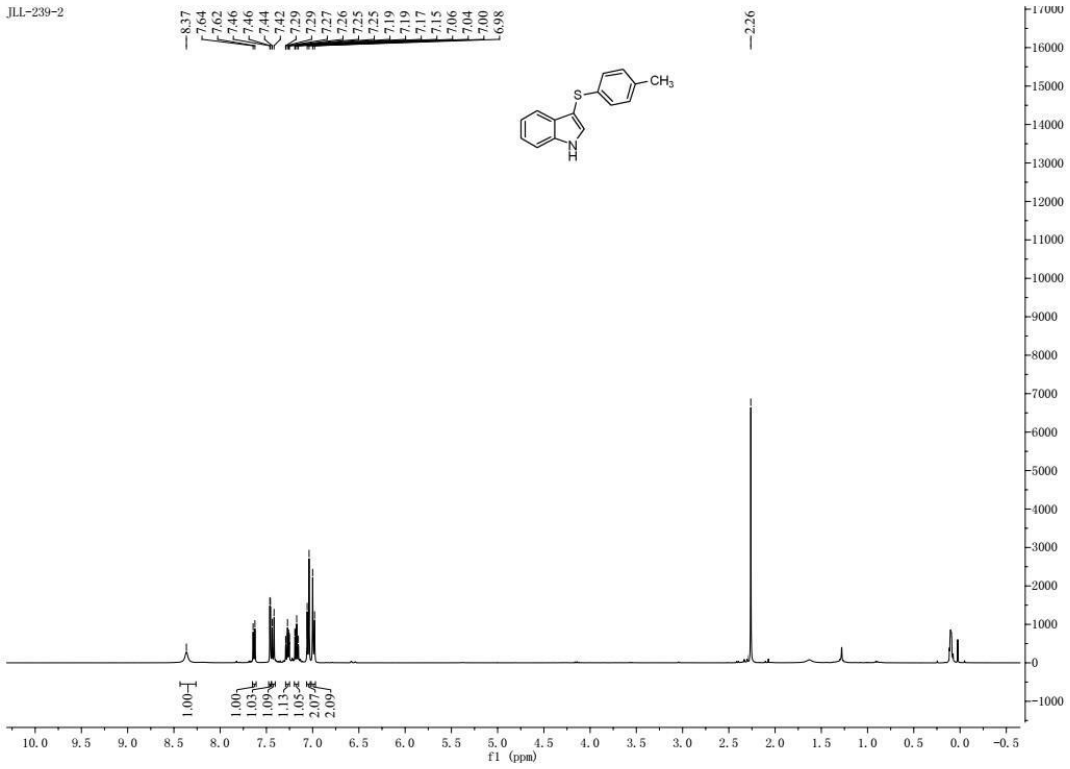
5-Methoxy-3-(phenylselanyl)-1*H*-indole (**9d**)

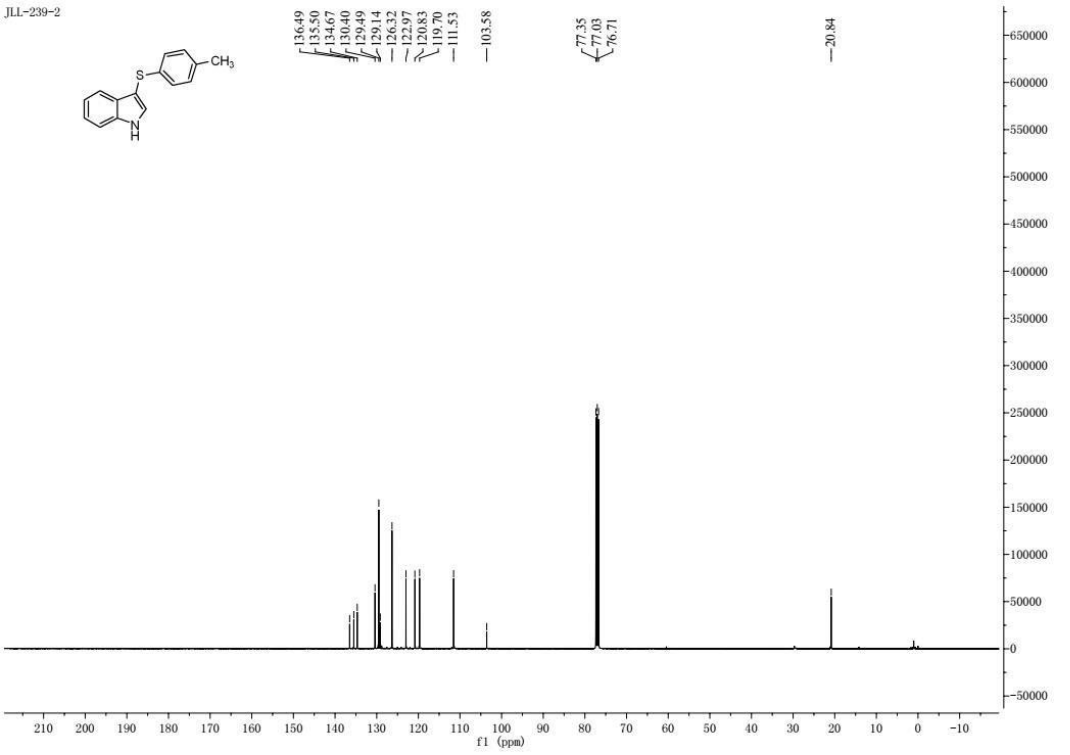


Yellow amorphous solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.42 (s, 1H, NH), 7.44 (d, J = 2.5 Hz, 1H, Ar-H), 7.33 (d, J = 8.8 Hz, 1H, Ar-H), 7.30 (dd, J = 8.2, 1.5 Hz, 1H, Ar-H), 7.29 (s, 1H, Ar-H), 7.21-7.13 (m, 4H, Ar-H), 6.97 (dd, J = 8.8, 2.5 Hz, 1H, Ar-H), 3.85 (s, 3H, OCH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 155.1, 134.0, 132.0, 131.4, 130.8, 129.1, 128.5, 125.6, 113.5, 112.4, 101.6, 97.5, 55.9. MS (ESI): 304 ($M+H^+$, 100). These assignments matched with those previously published.[13]

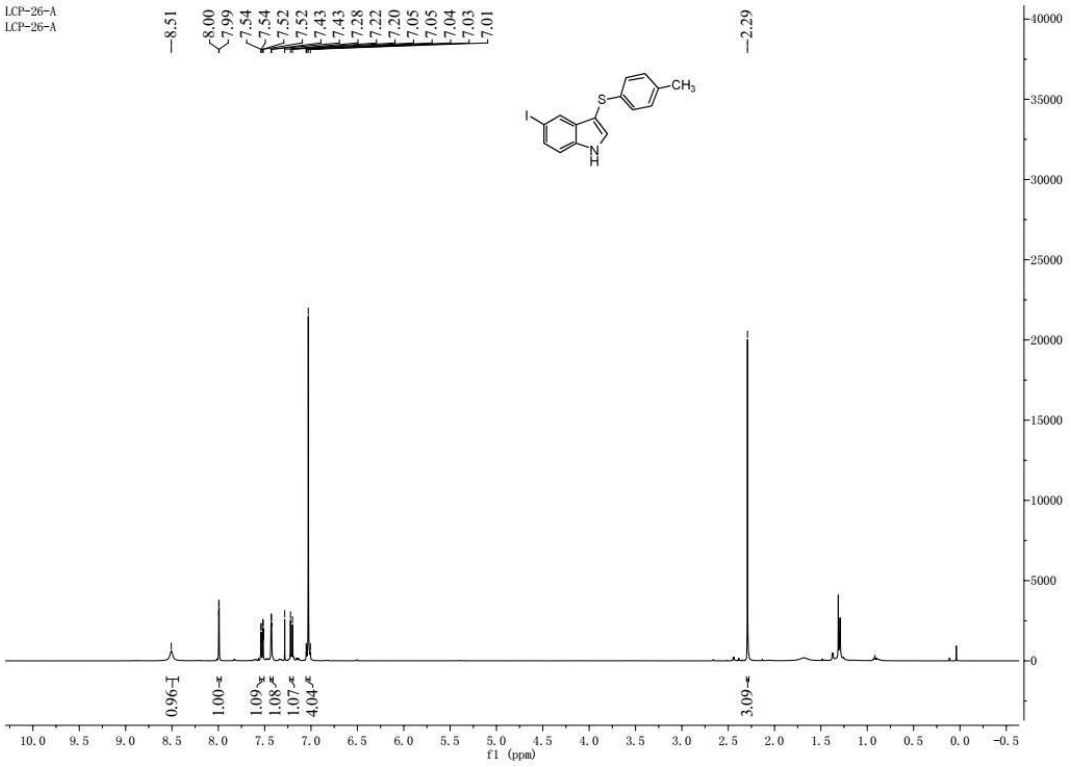
4. Copies of ^{1}H and ^{13}C Spectra

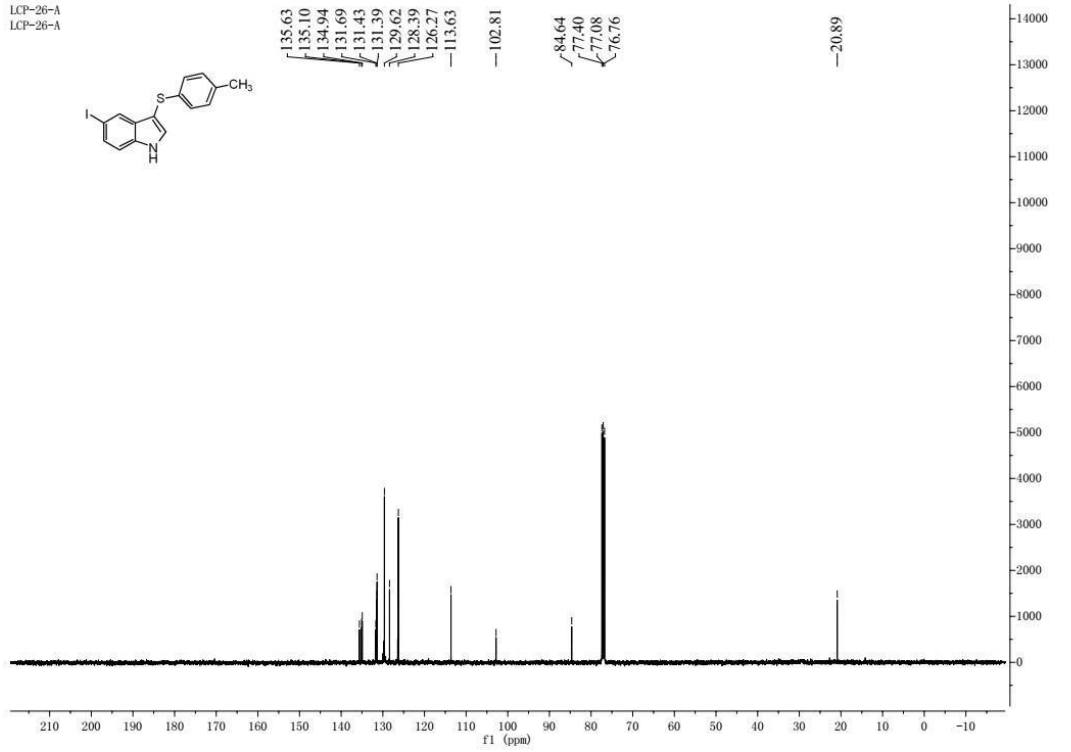
^{1}H and ^{13}C NMR Spectra for **6aa**



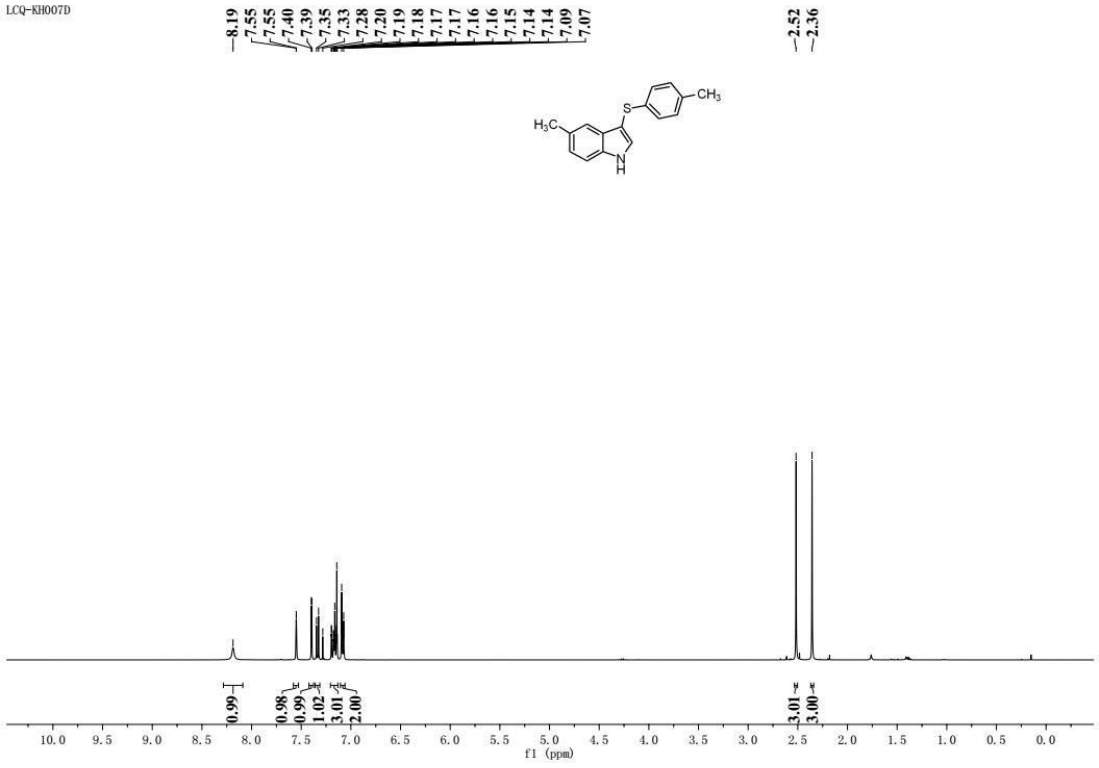


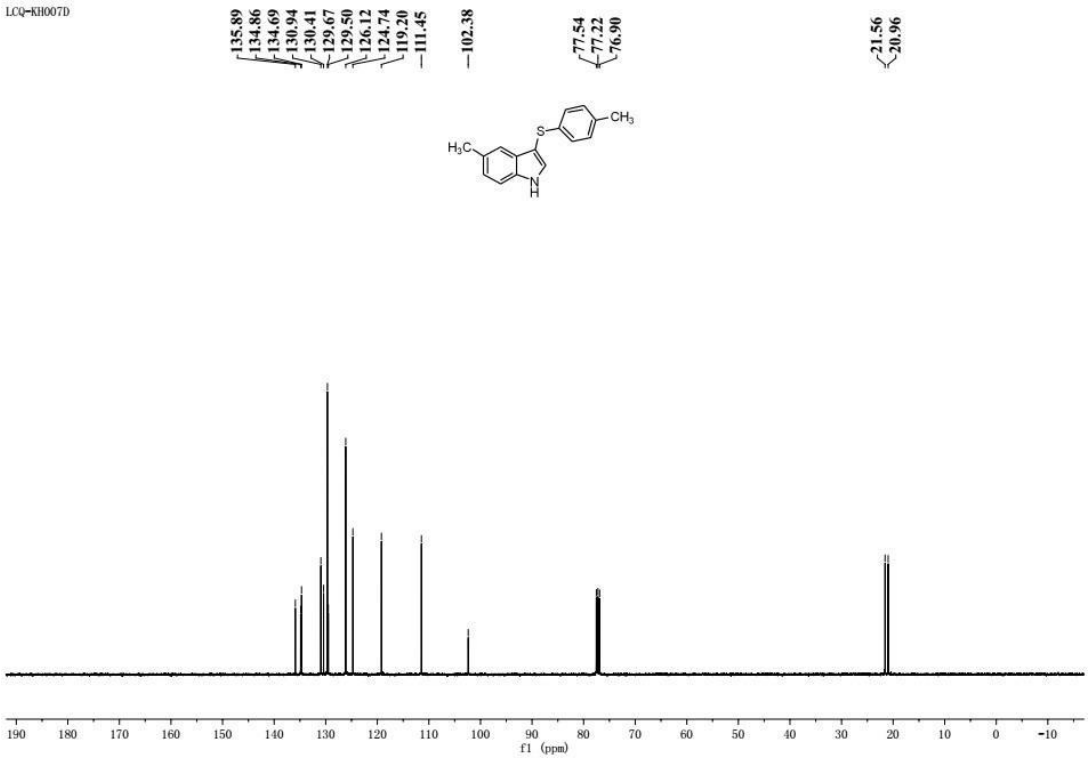
^{1}H and ^{13}C NMR Spectra for **6ba**



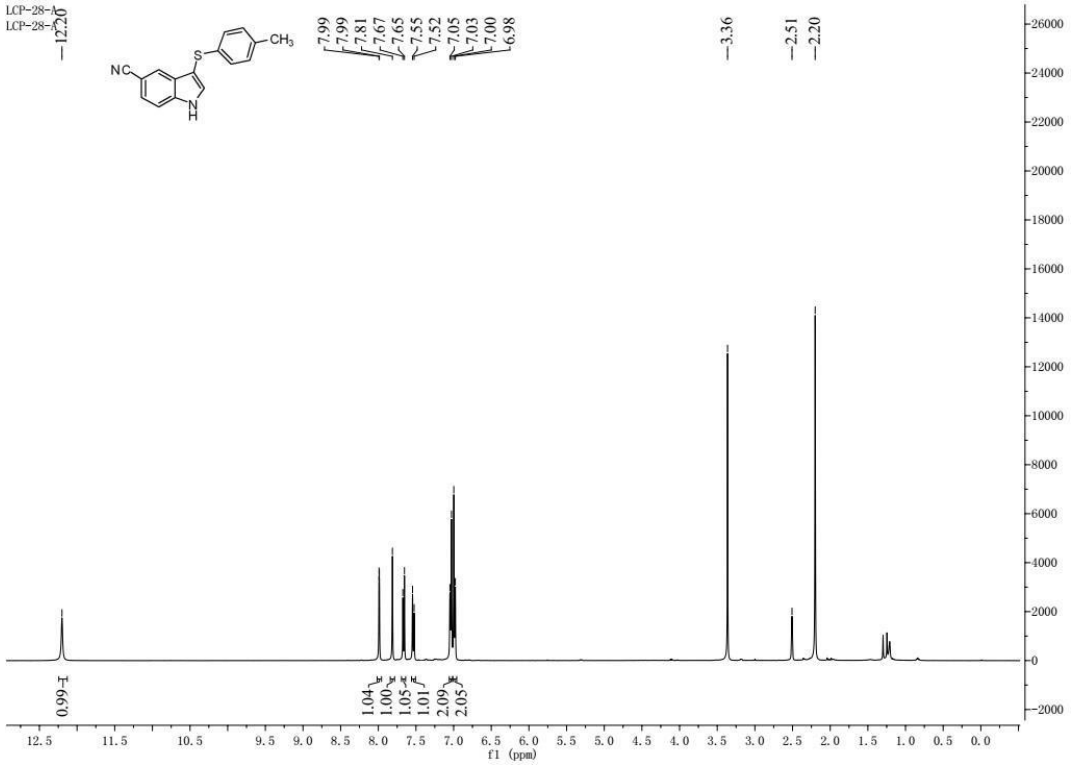


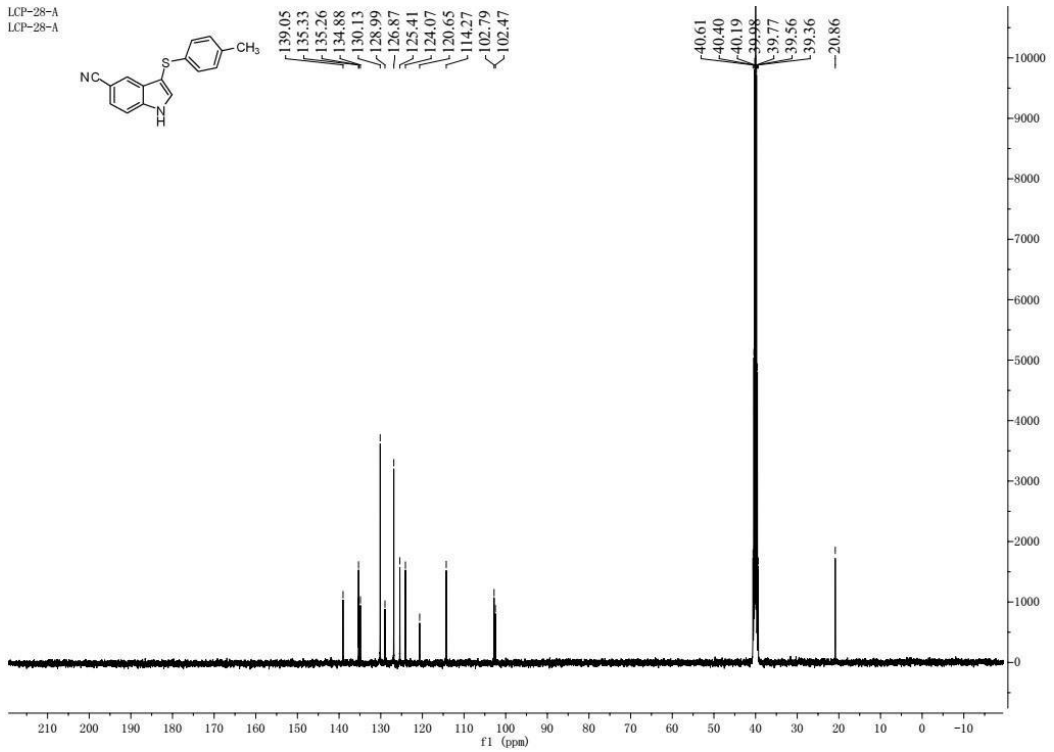
^{1}H and ^{13}C NMR Spectra for **6ca**



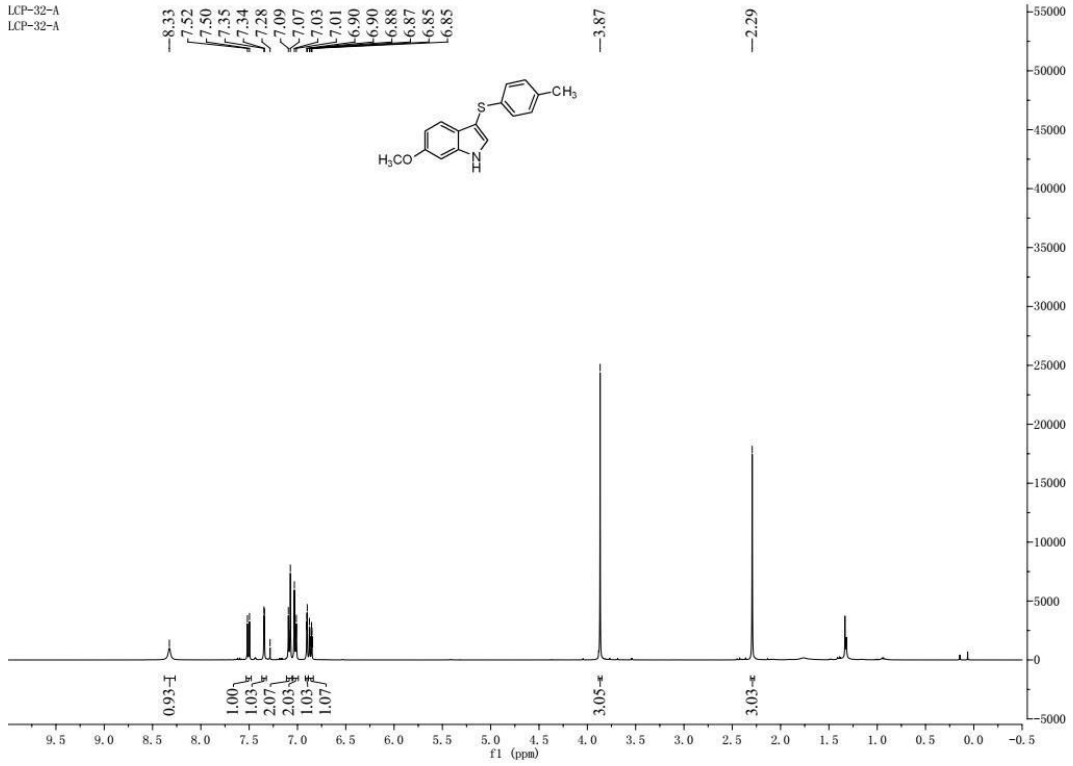


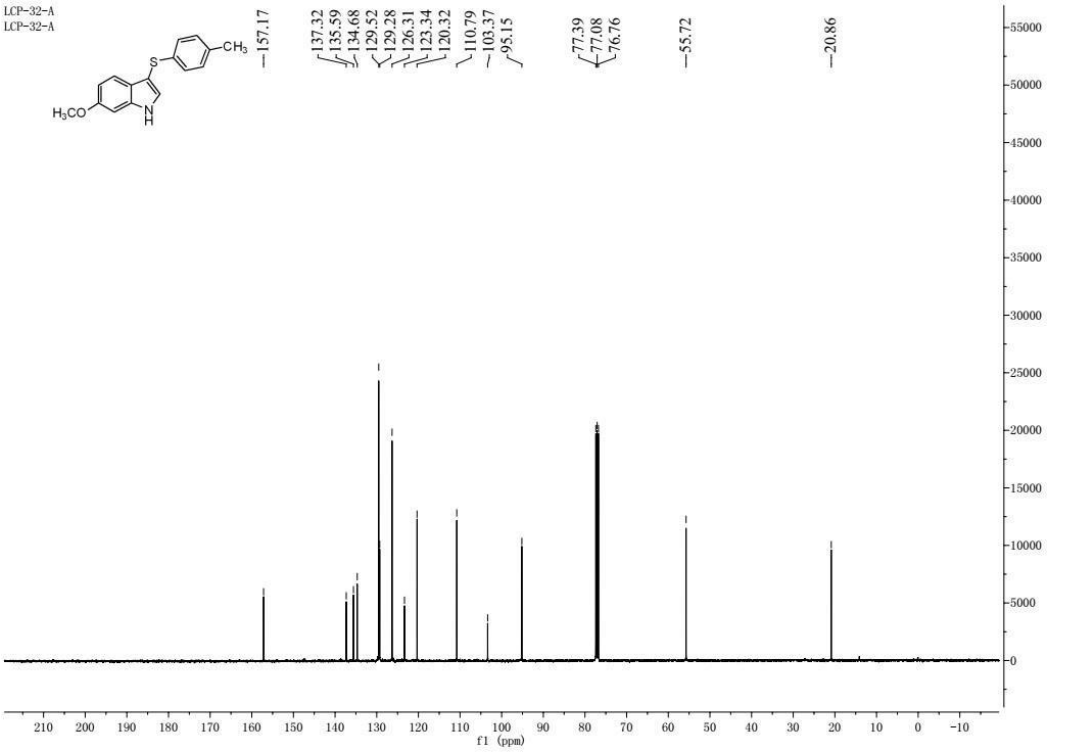
^{1}H and ^{13}C NMR Spectra for **6da**



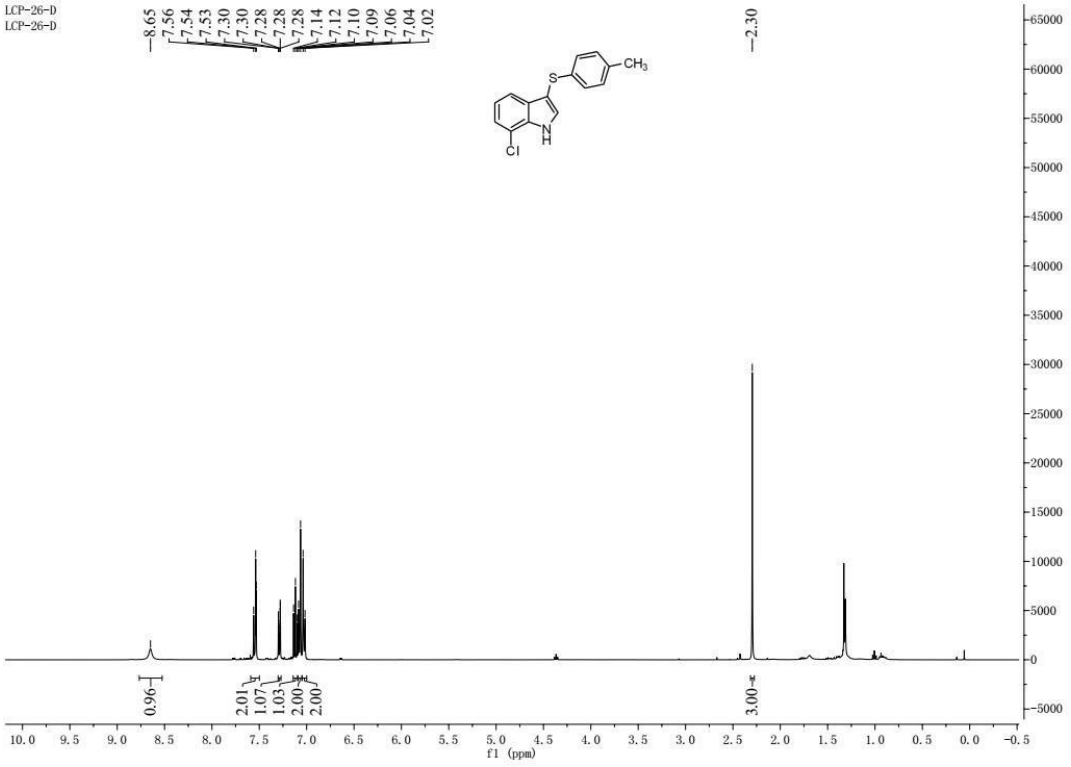


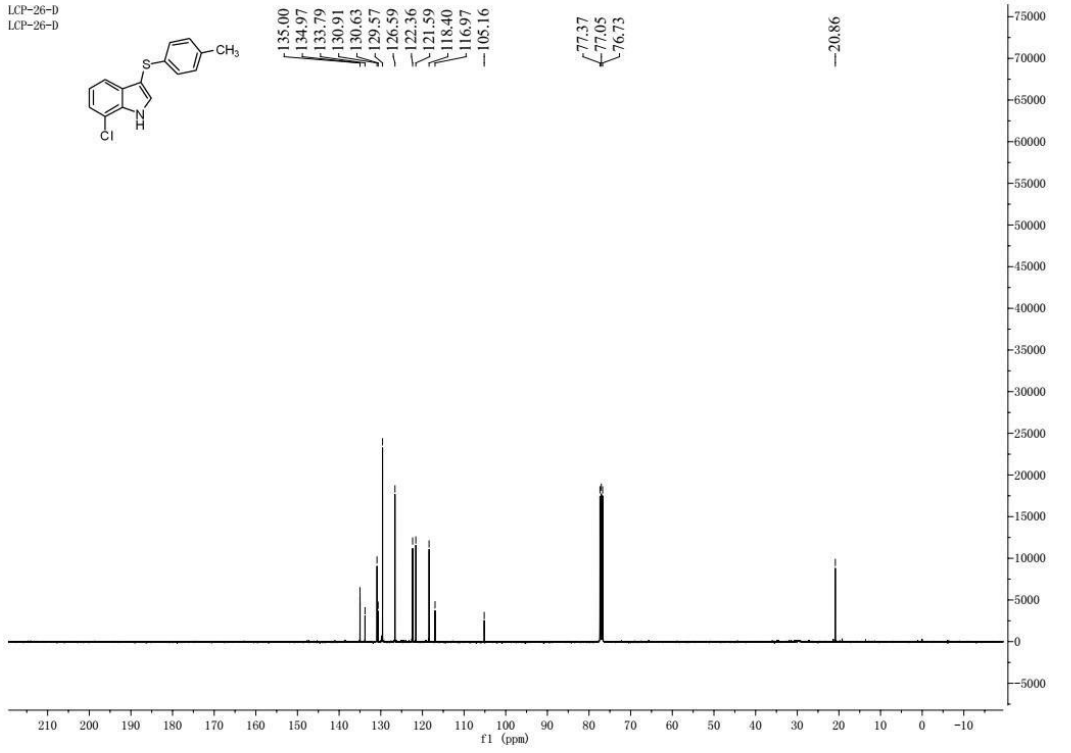
^{1}H and ^{13}C NMR Spectra for **6ea**



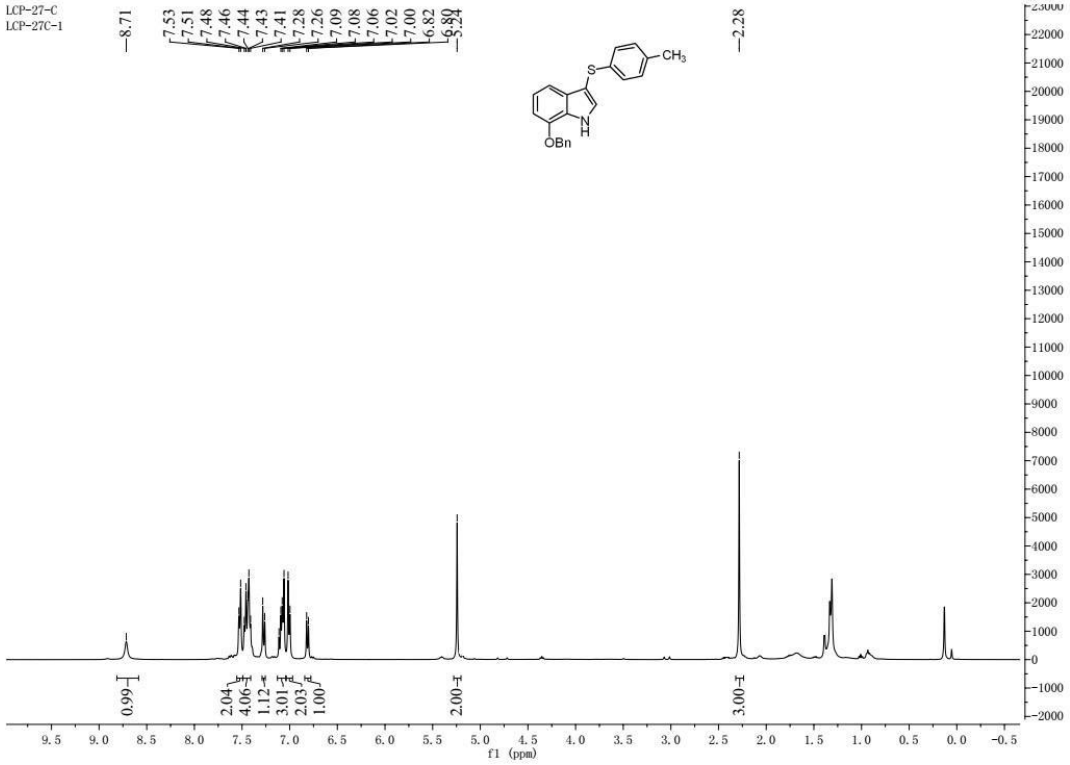


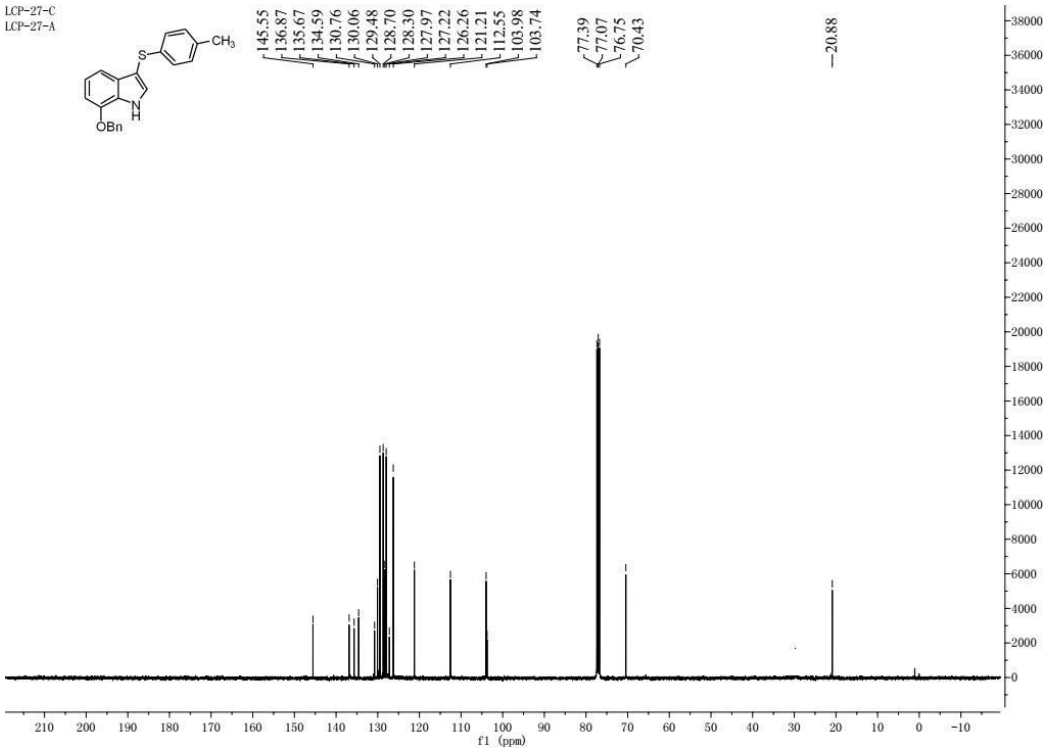
^{1}H and ^{13}C NMR Spectra for **6fa**



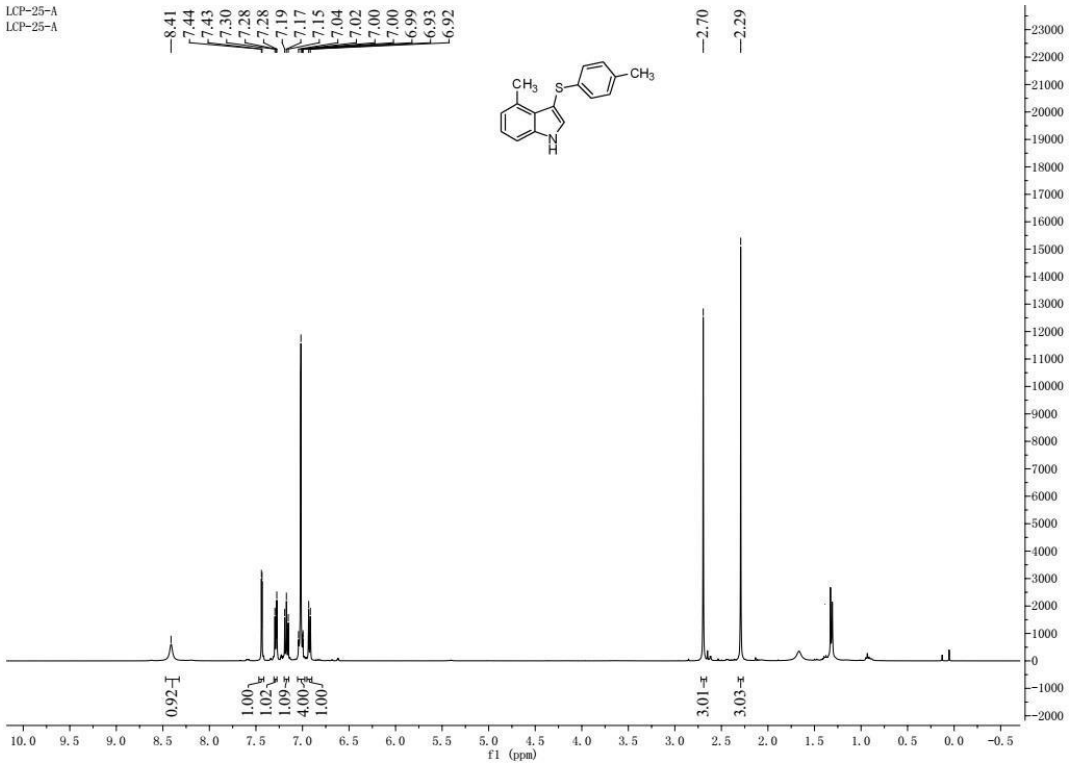


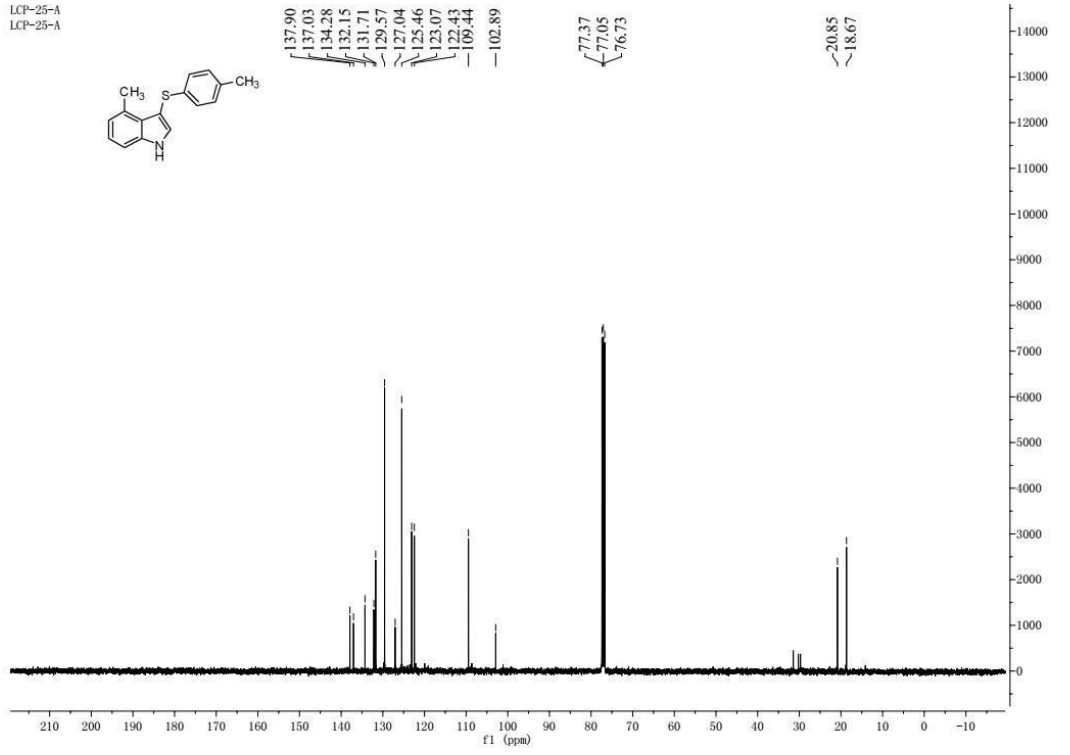
^{1}H and ^{13}C NMR Spectra for **6ga**



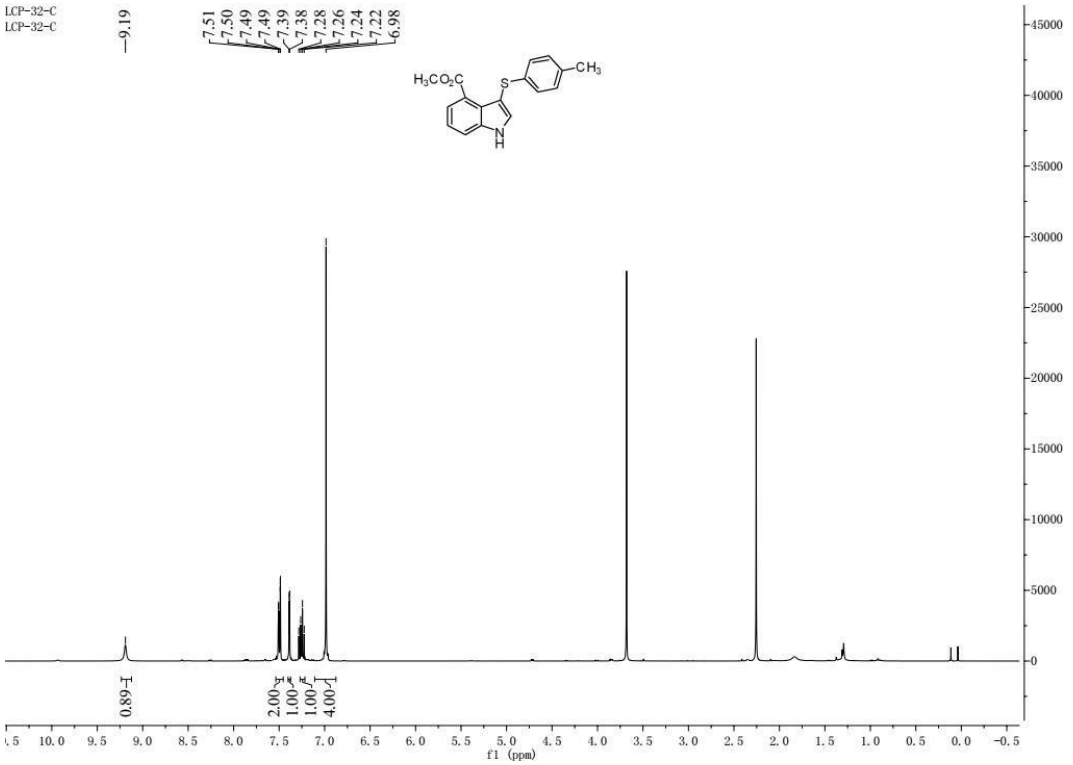


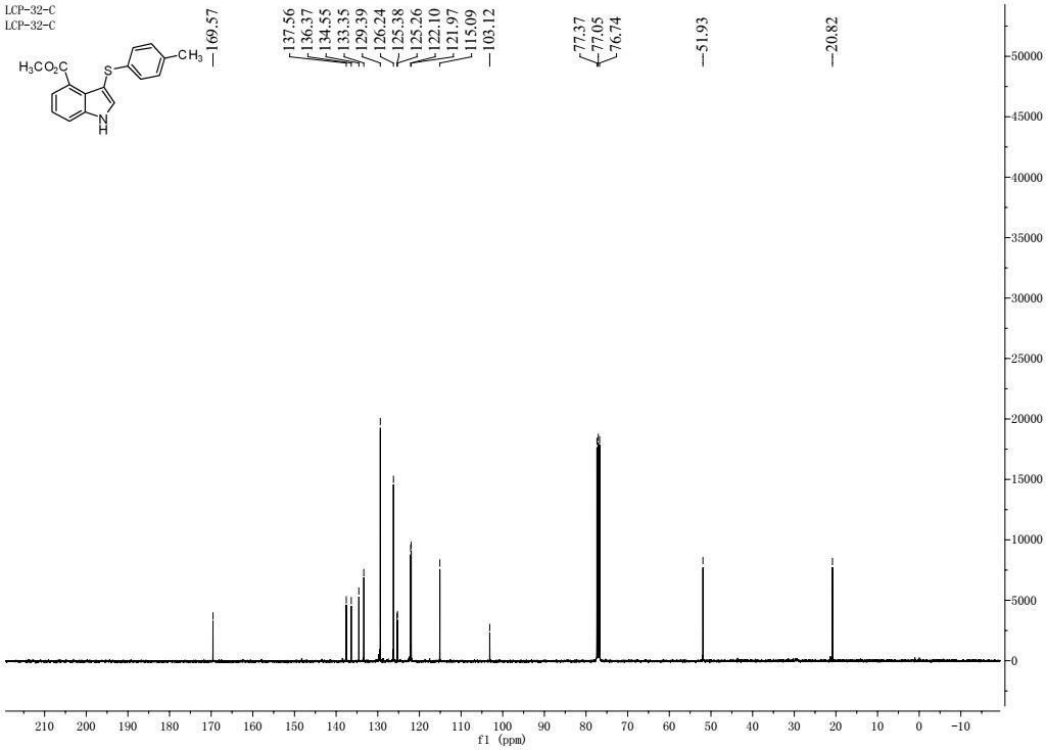
^{1}H and ^{13}C NMR Spectra for **6ha**



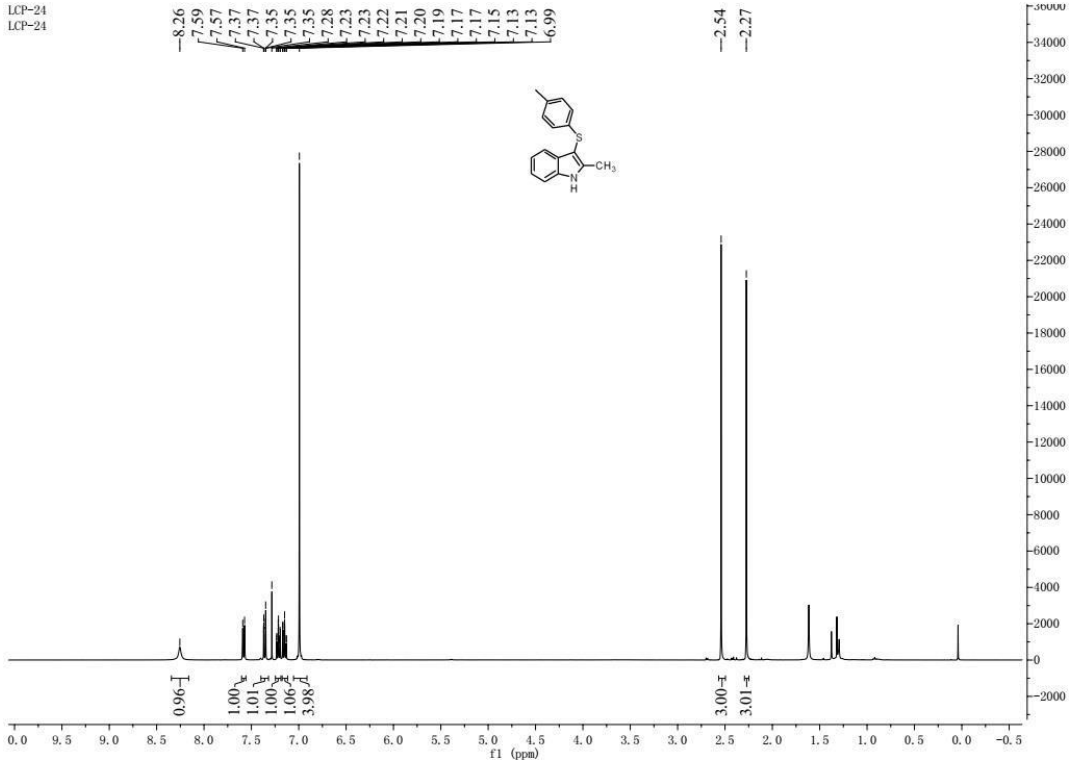


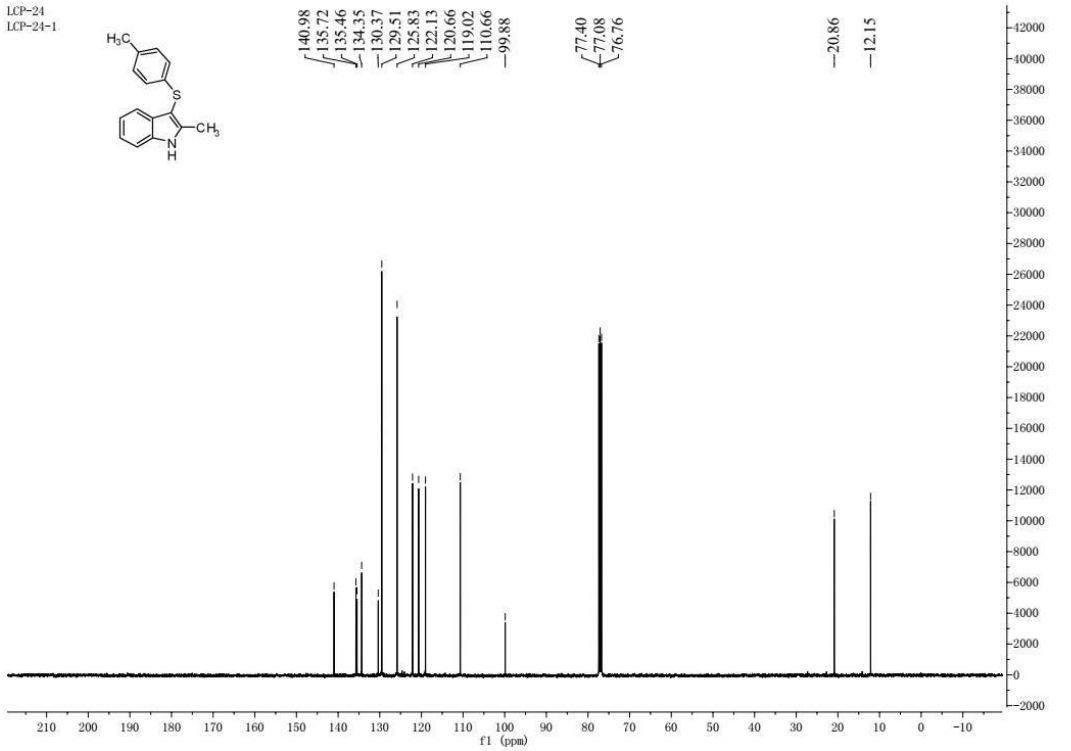
^{1}H and ^{13}C NMR Spectra for **6ia**



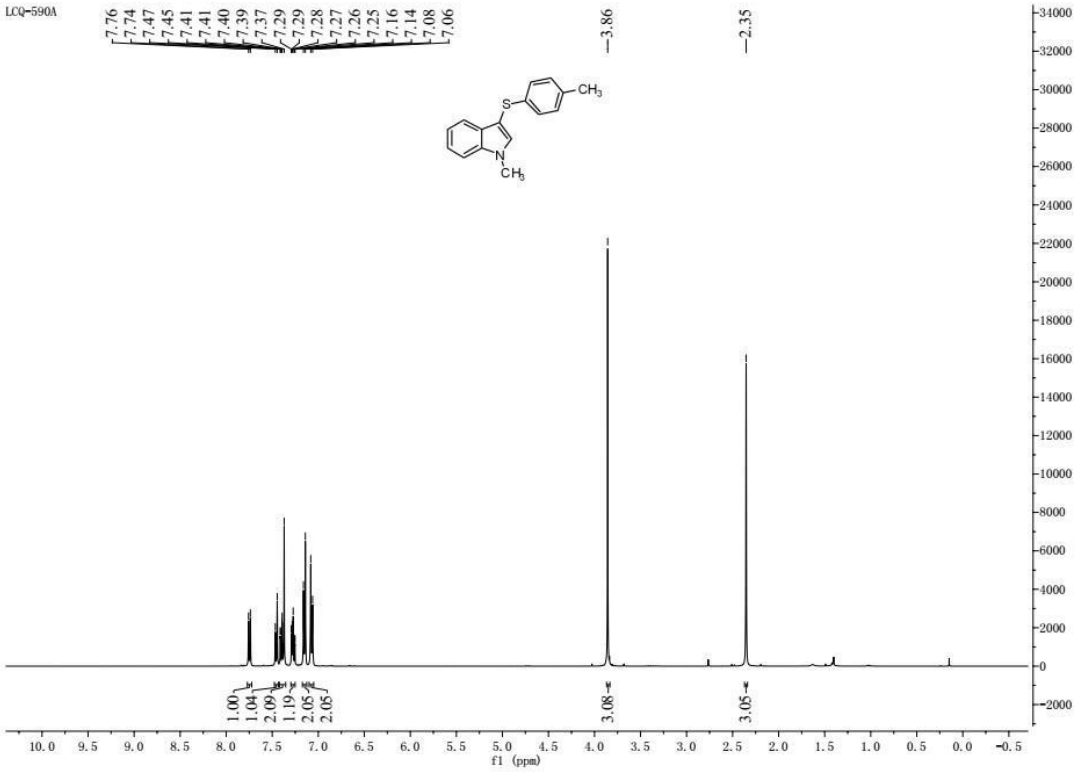


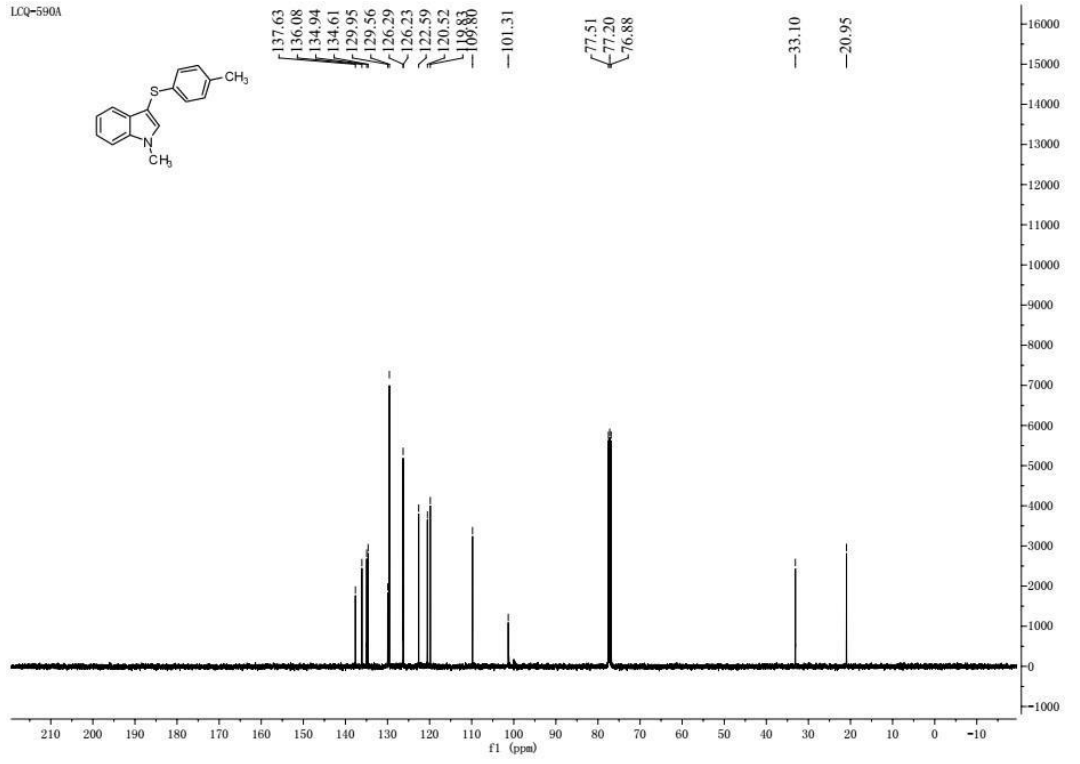
^{1}H and ^{13}C NMR Spectra for **6ja**



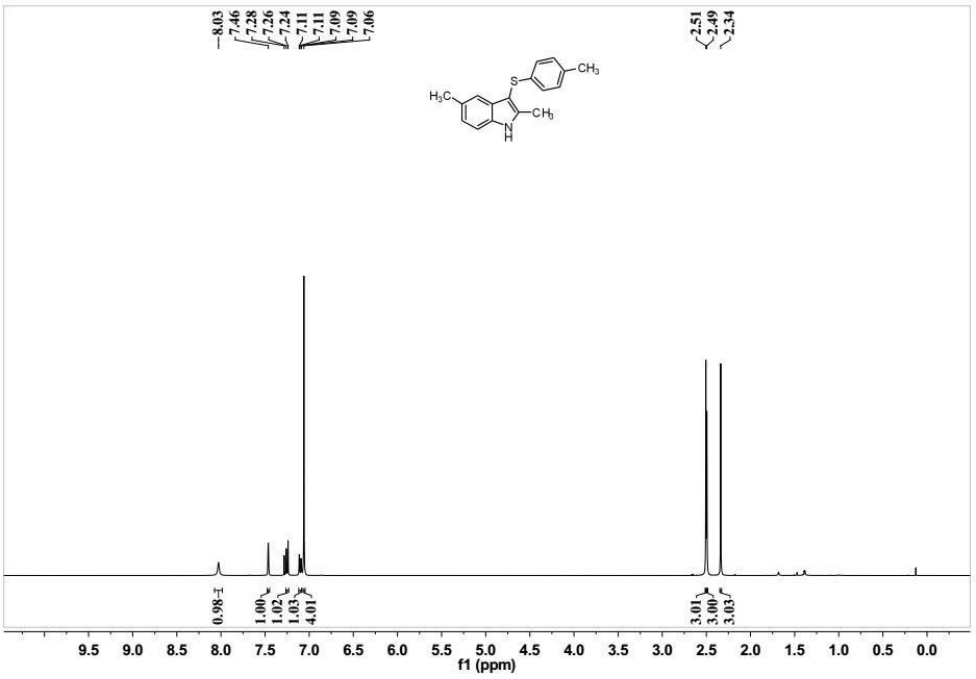


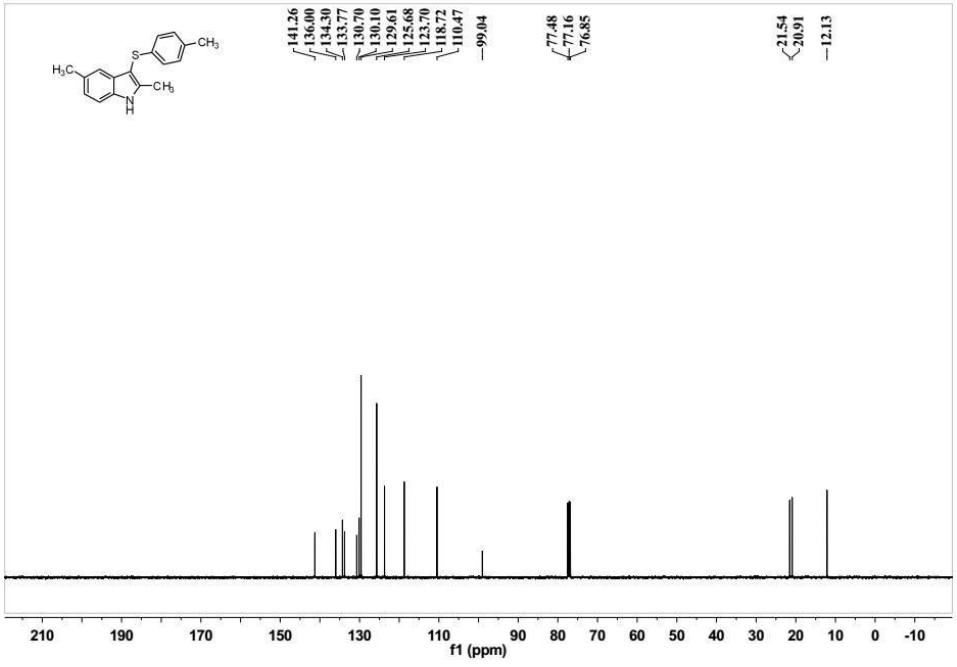
^{1}H and ^{13}C NMR Spectra for **6ka**



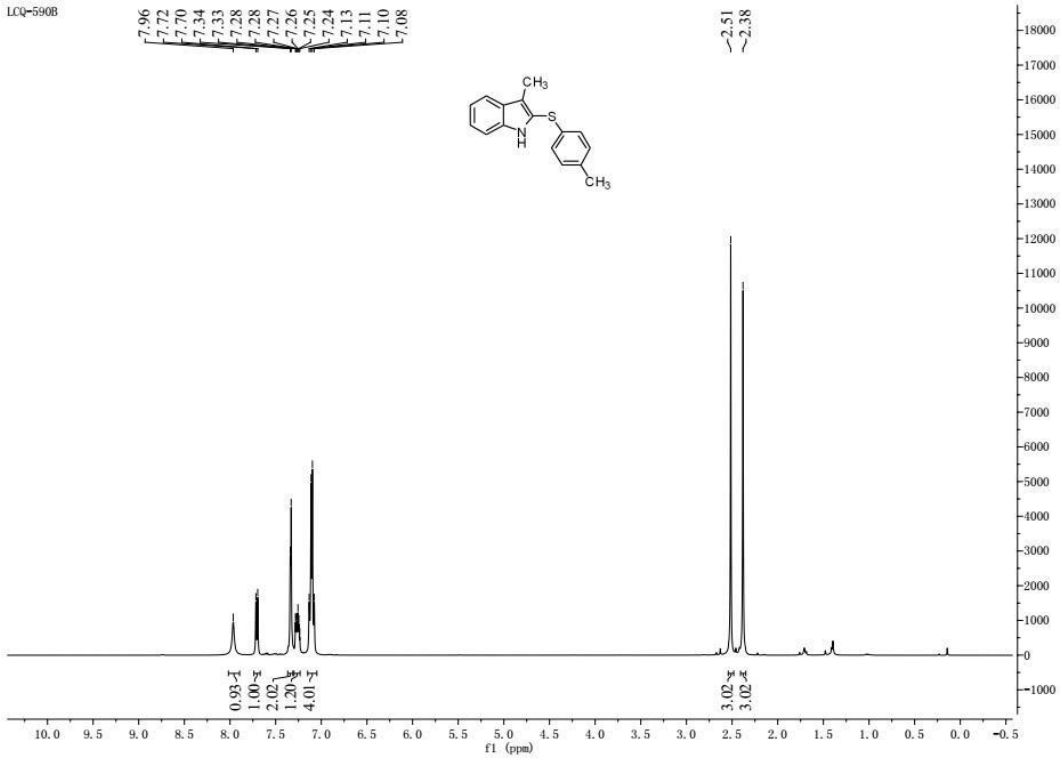


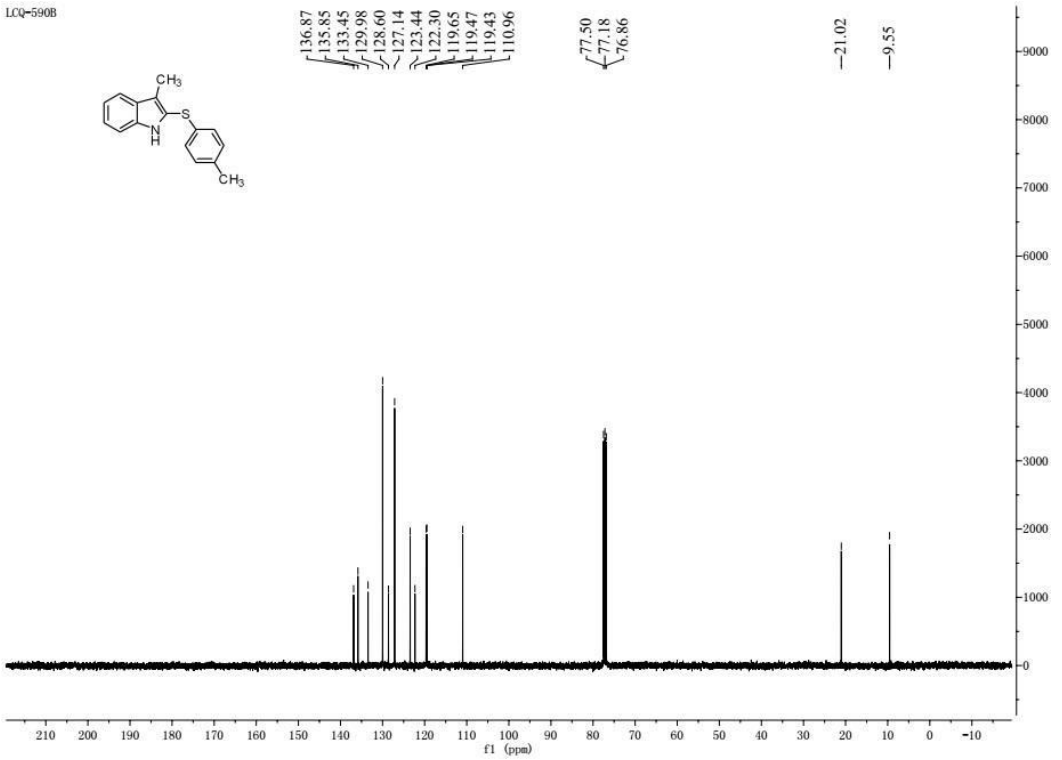
^{1}H and ^{13}C NMR Spectra for **6la**



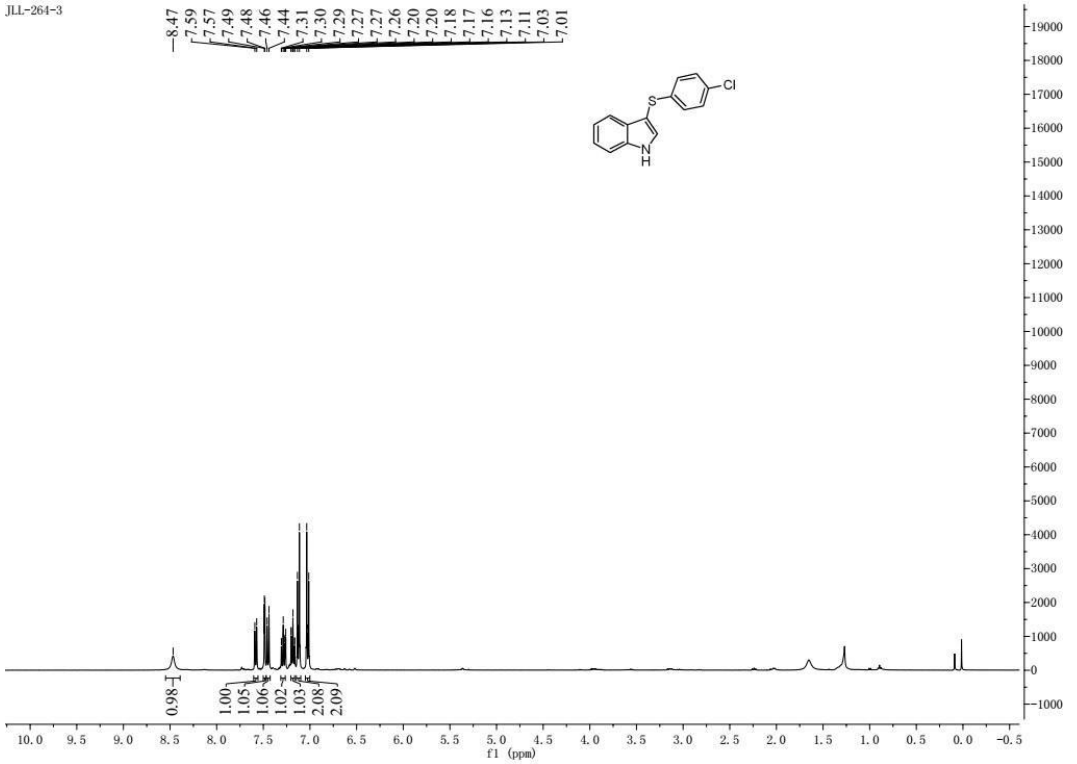


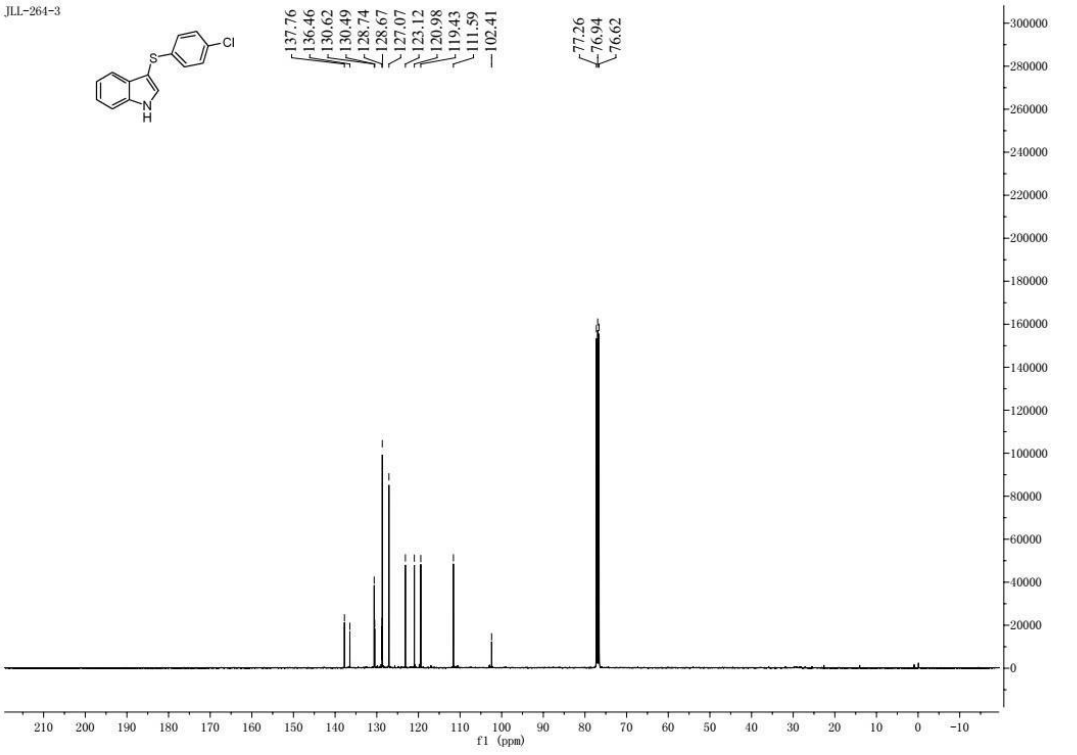
^{1}H and ^{13}C NMR Spectra for **6ma**



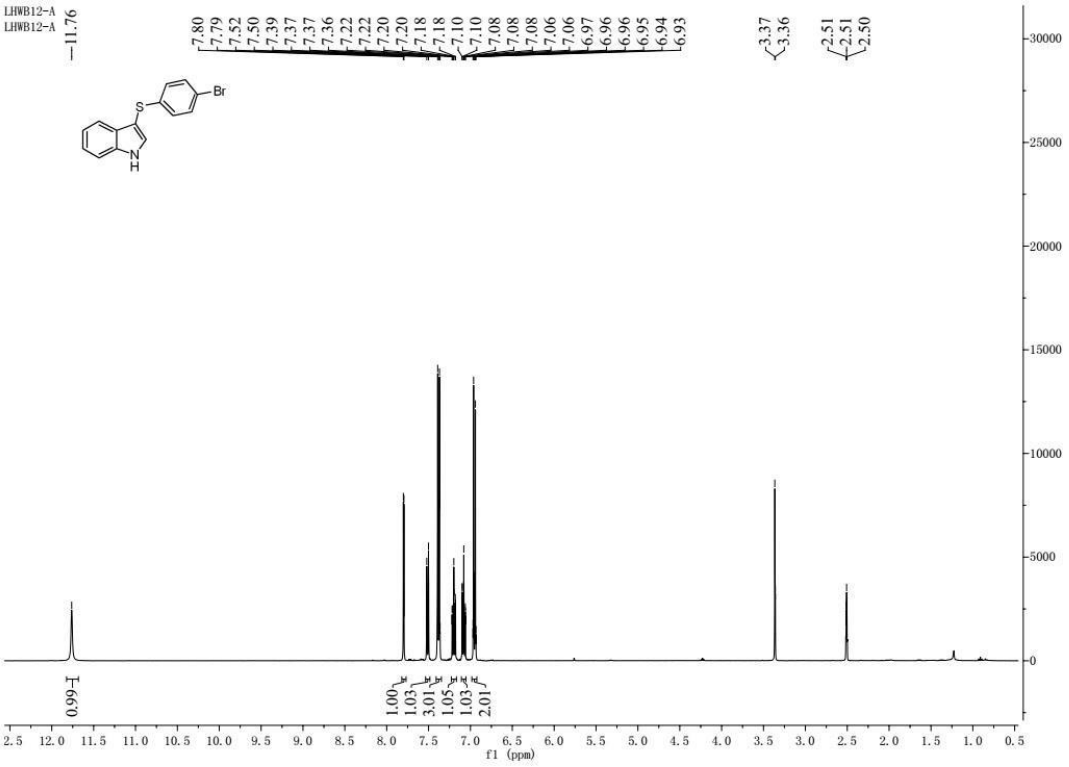


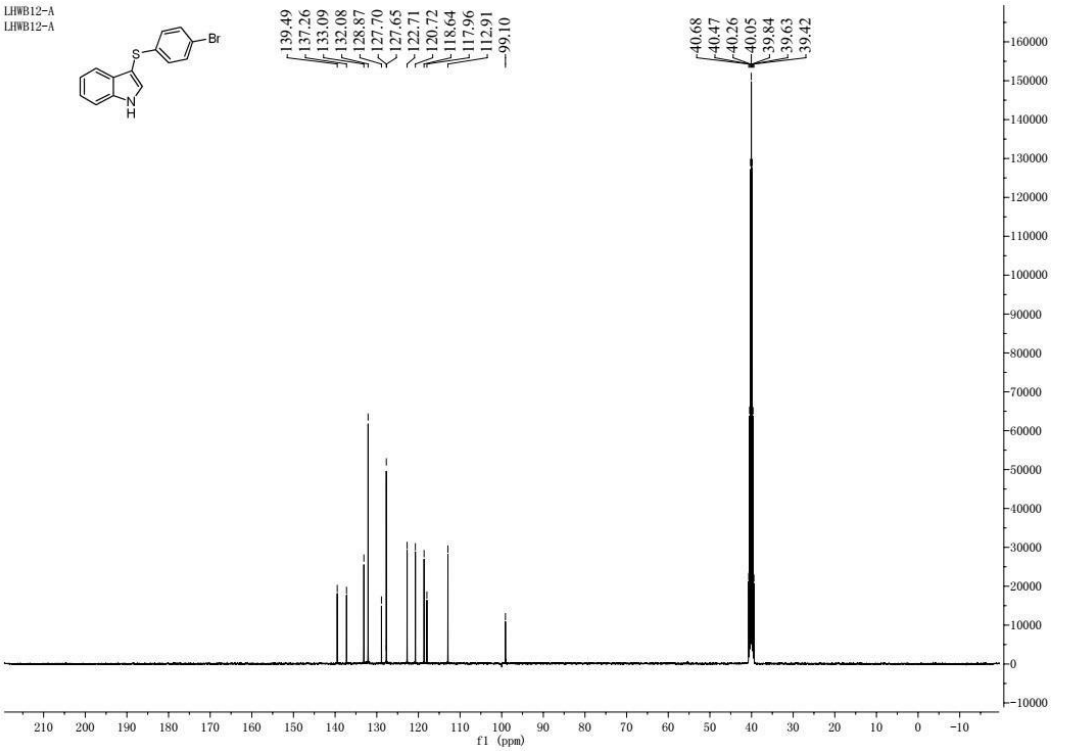
^{1}H and ^{13}C NMR Spectra for **6ab**



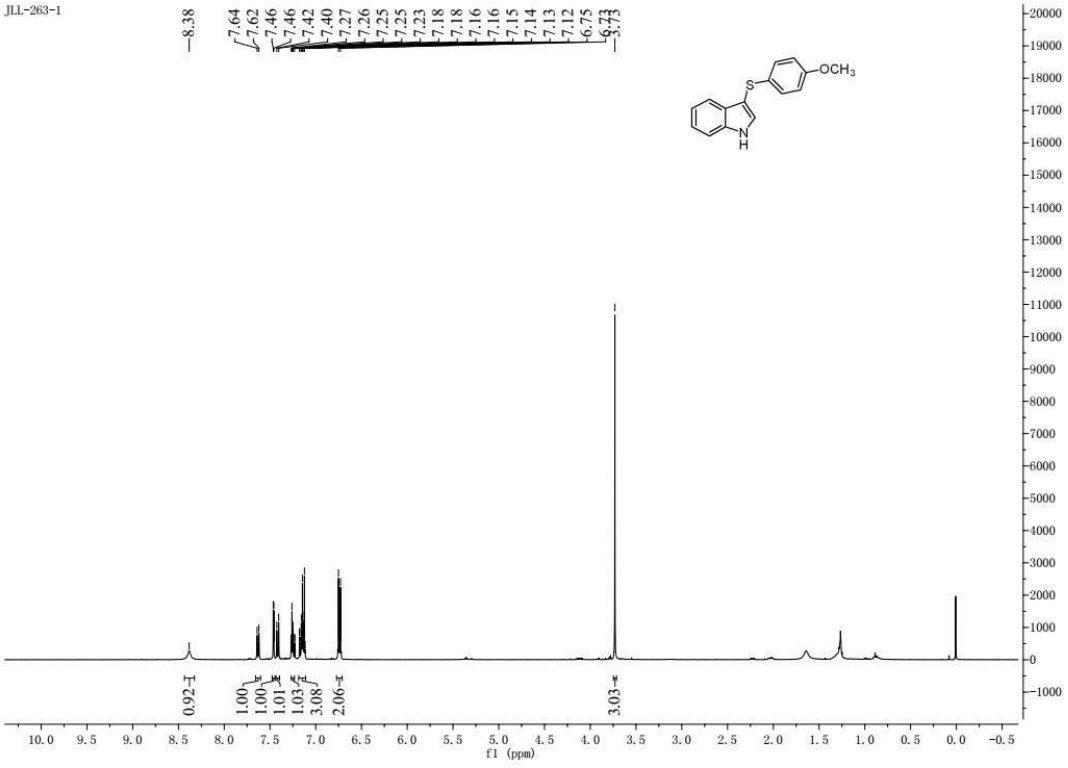


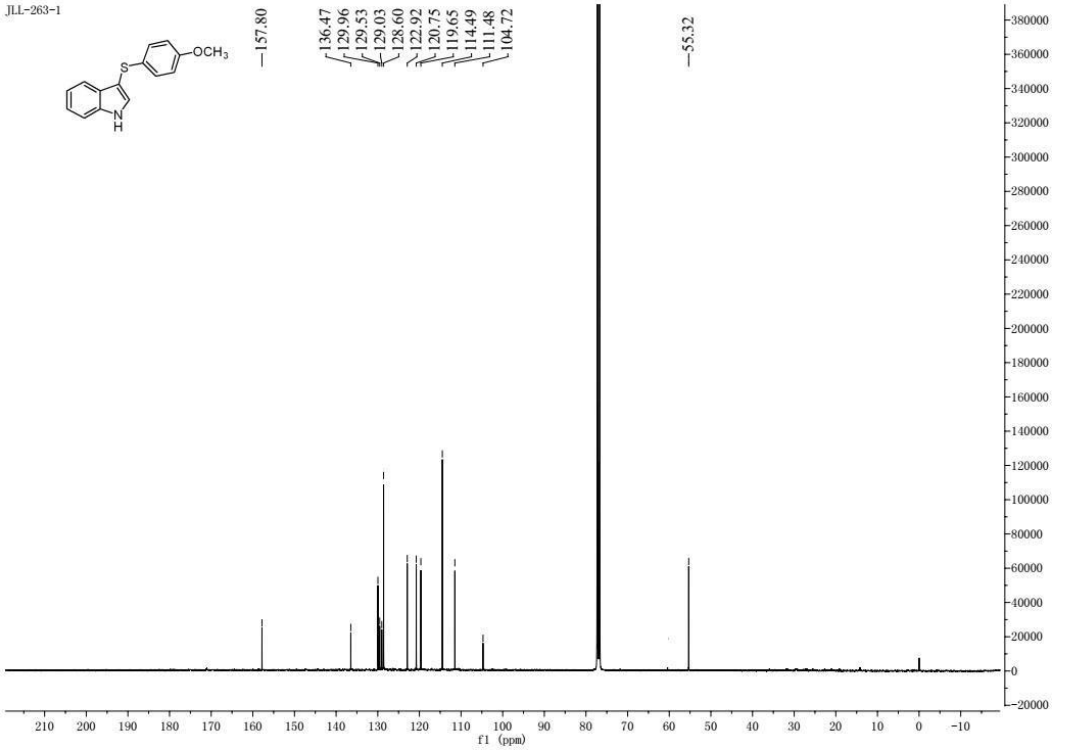
^{1}H and ^{13}C NMR Spectra for **6ac**



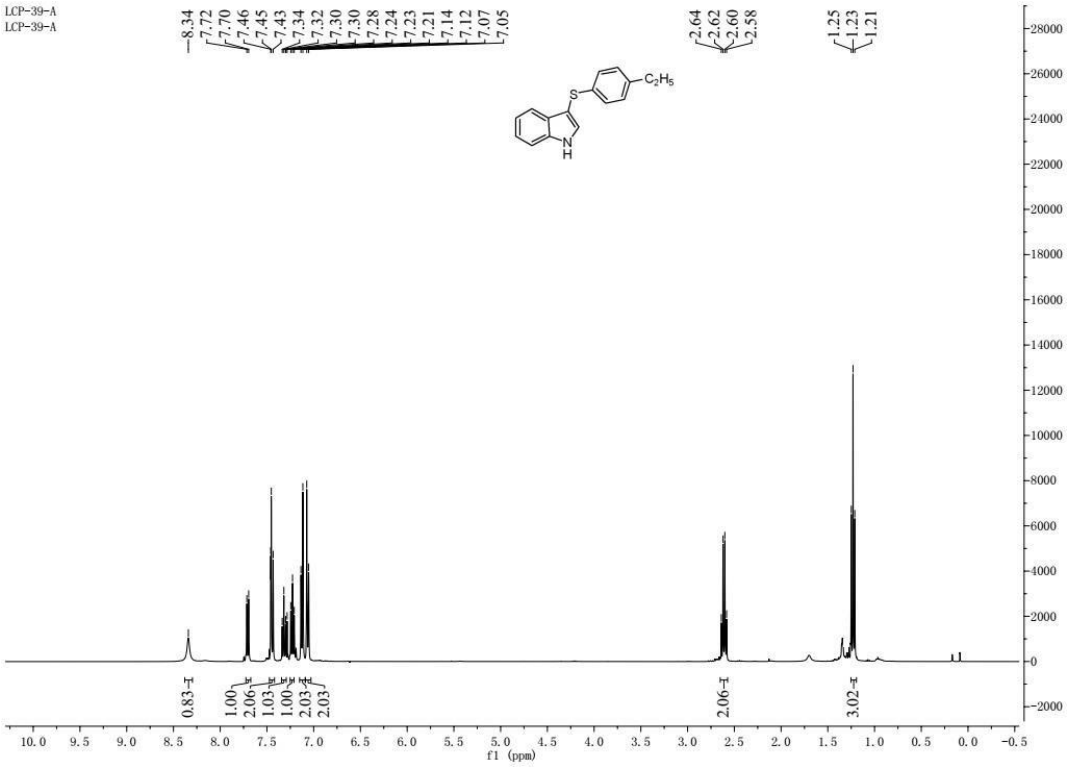


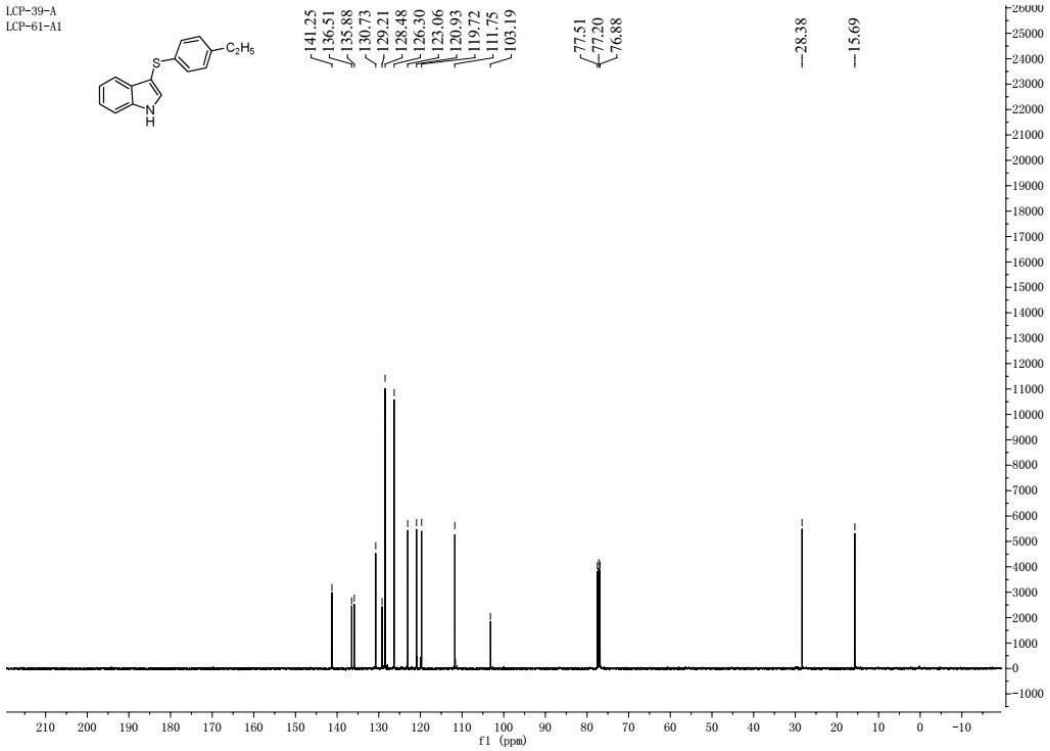
^{1}H and ^{13}C NMR Spectra for **6ad**



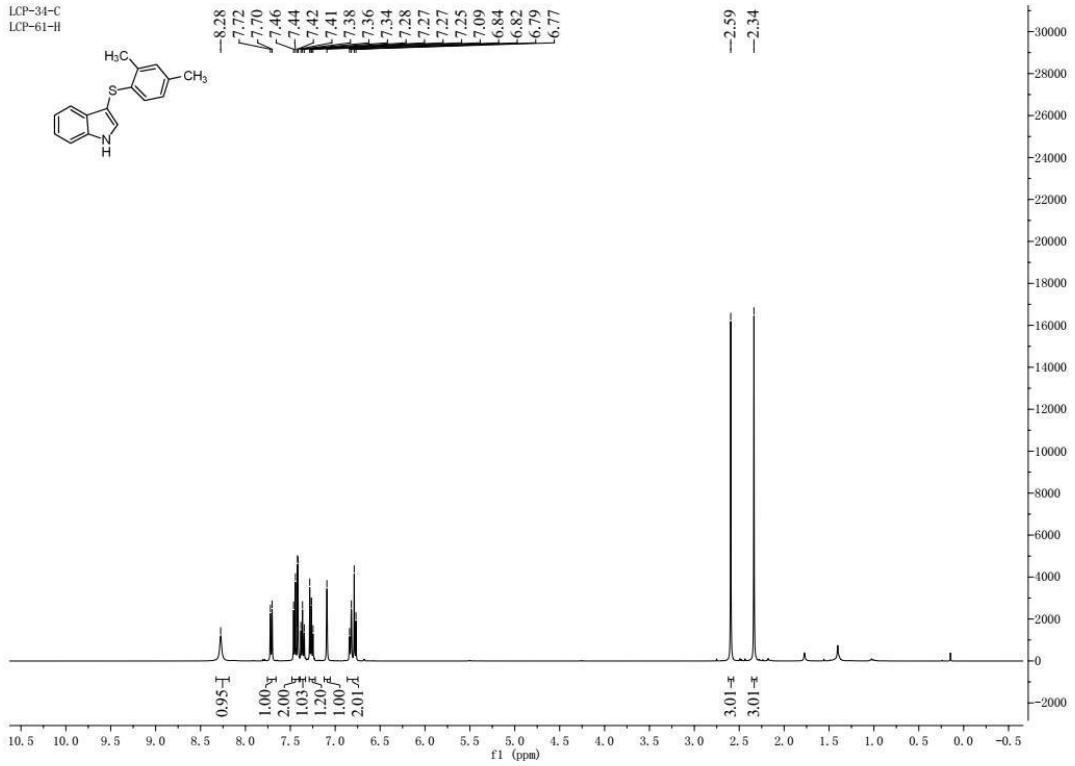


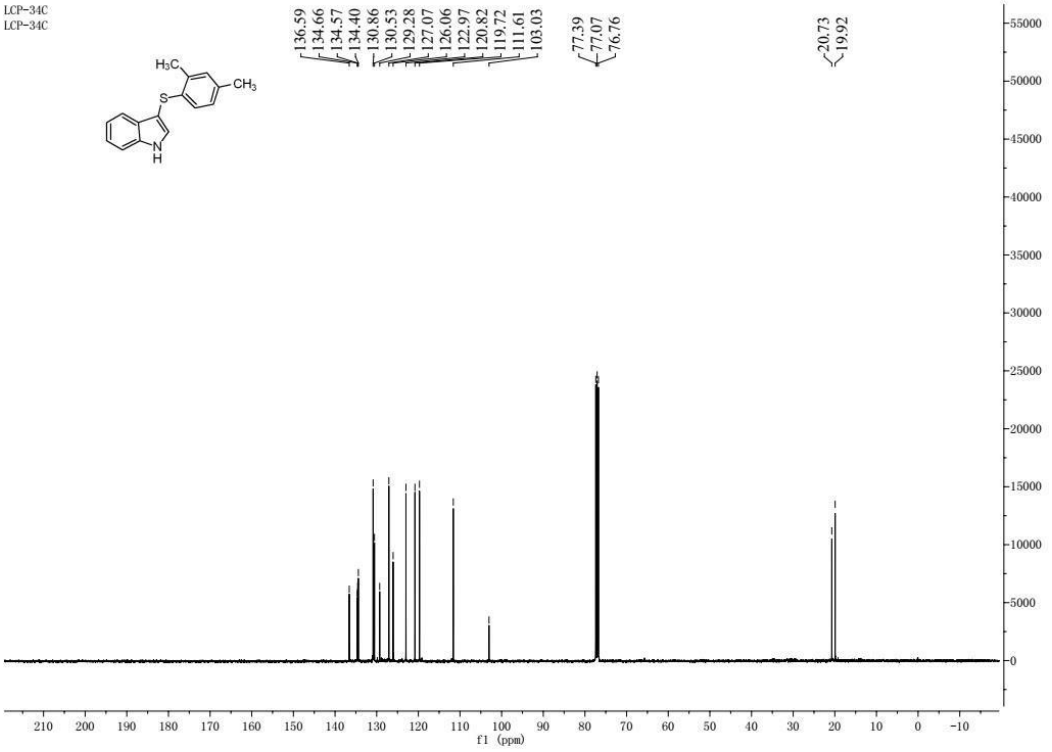
^{1}H and ^{13}C NMR Spectra for **6ae**



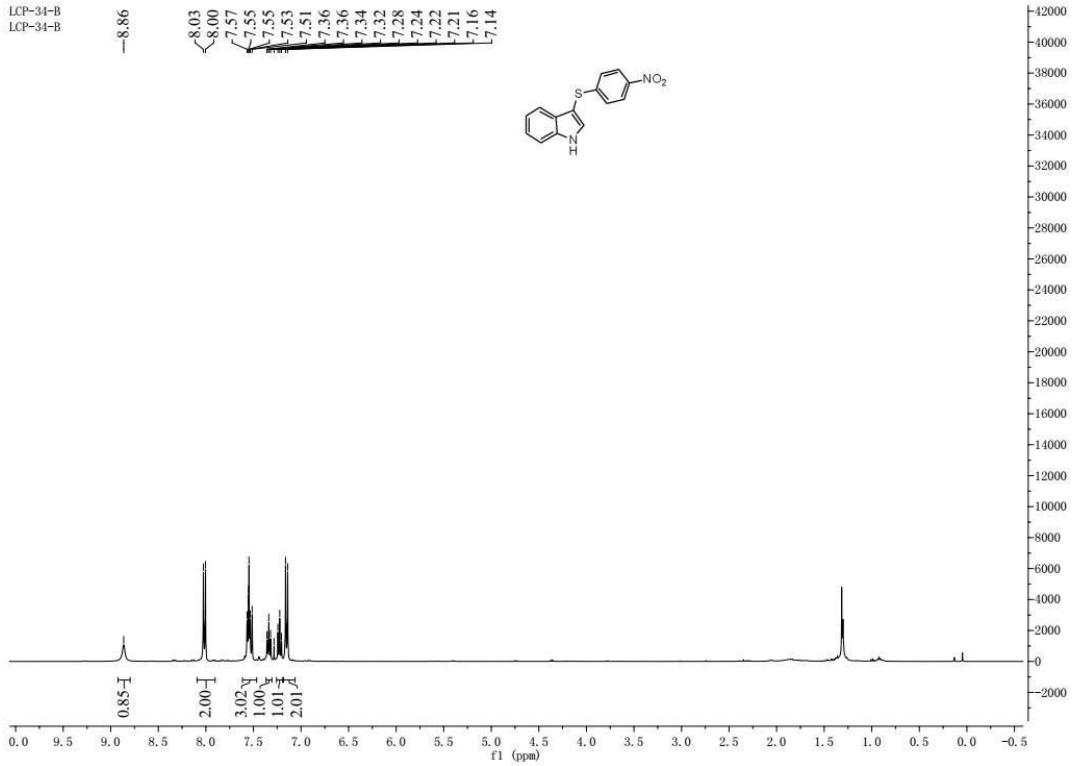


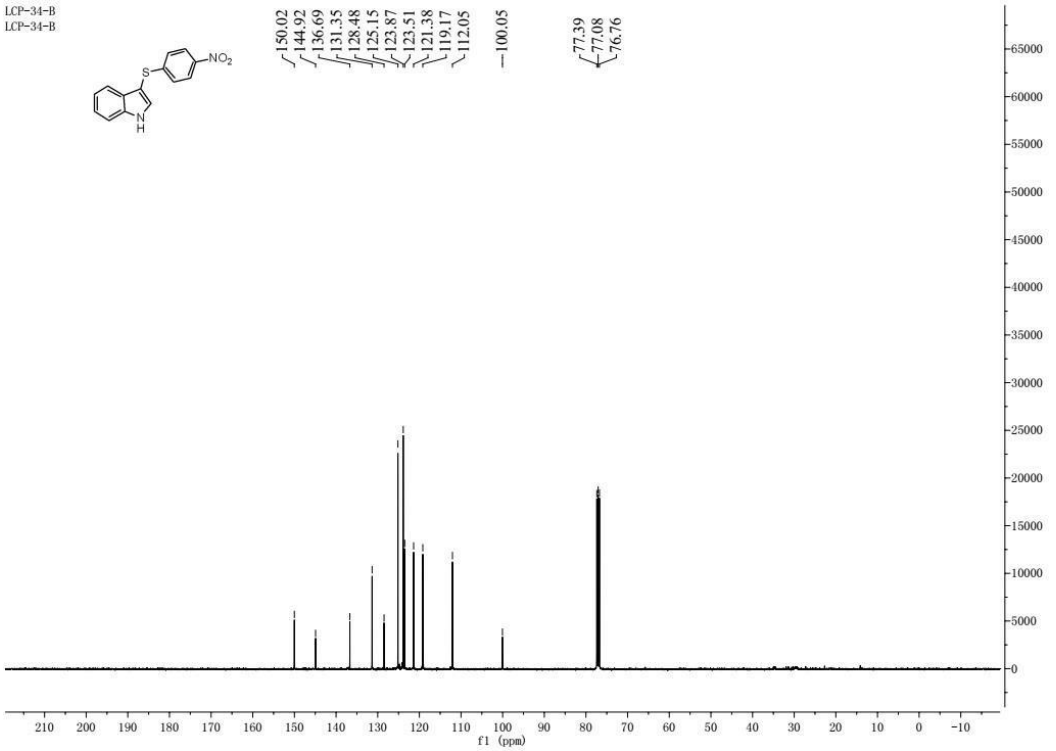
^{1}H and ^{13}C NMR Spectra for **6af**



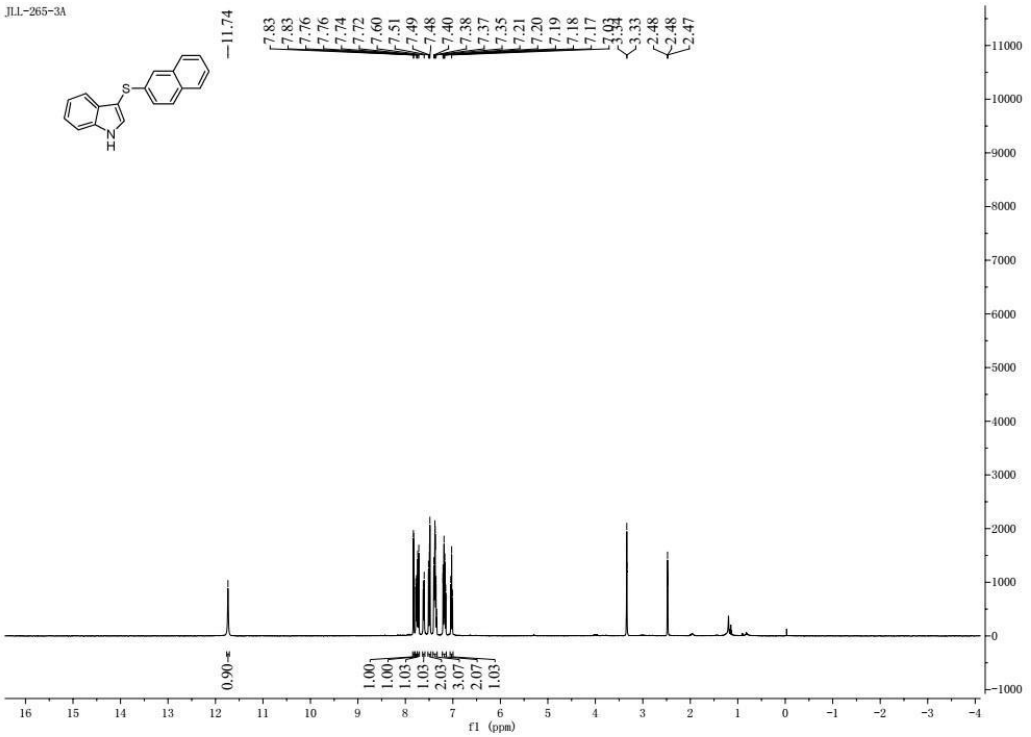


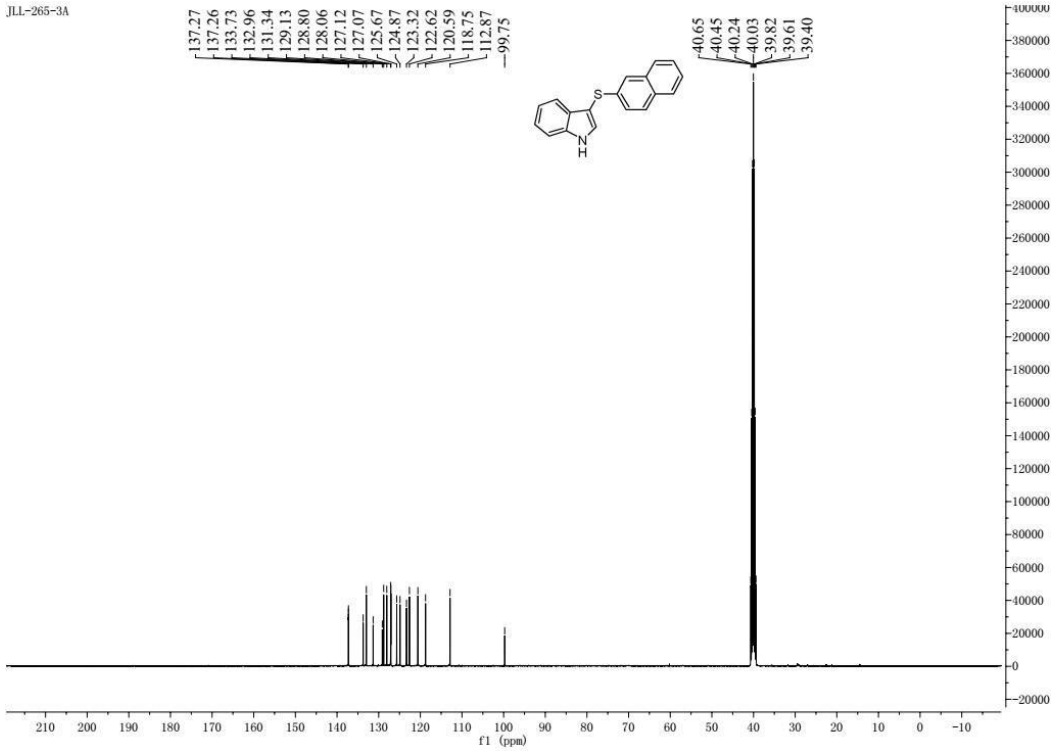
^{1}H and ^{13}C NMR Spectra for **6ag**





^{1}H and ^{13}C NMR Spectra for **6ah**





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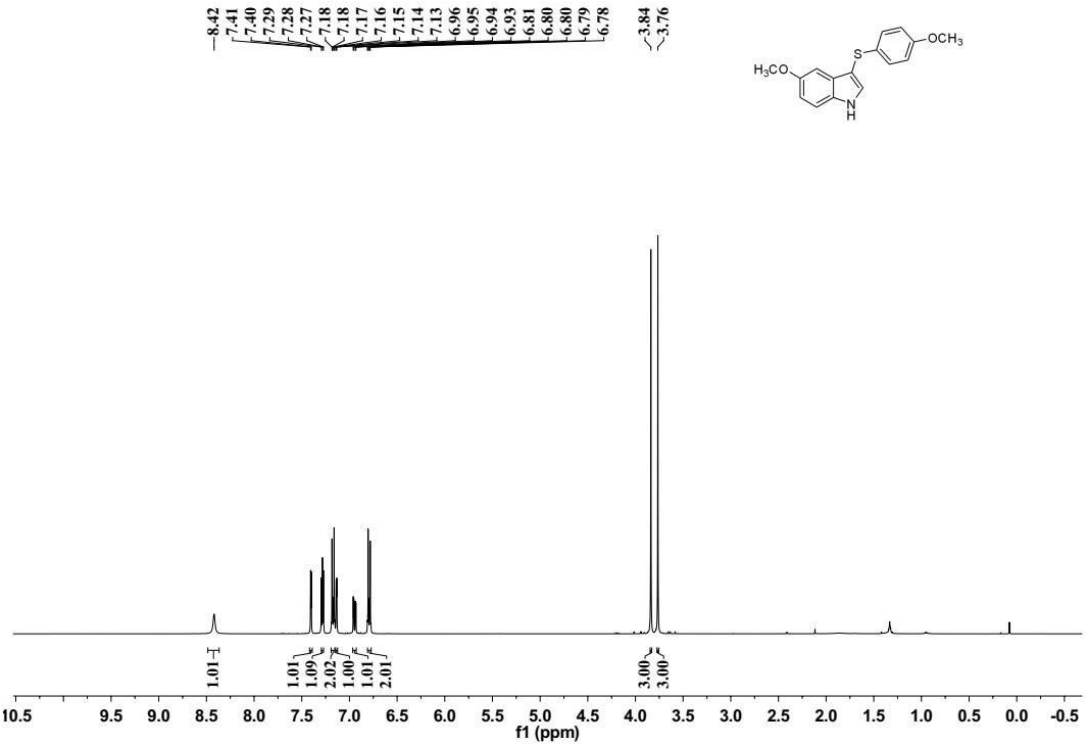
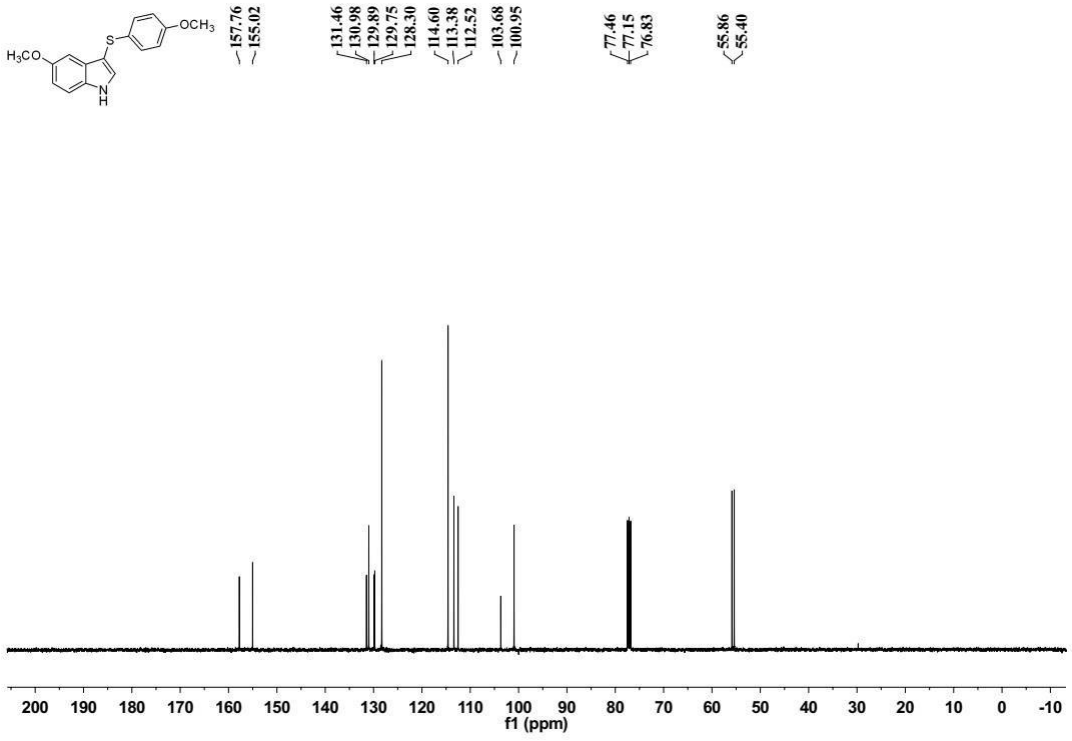
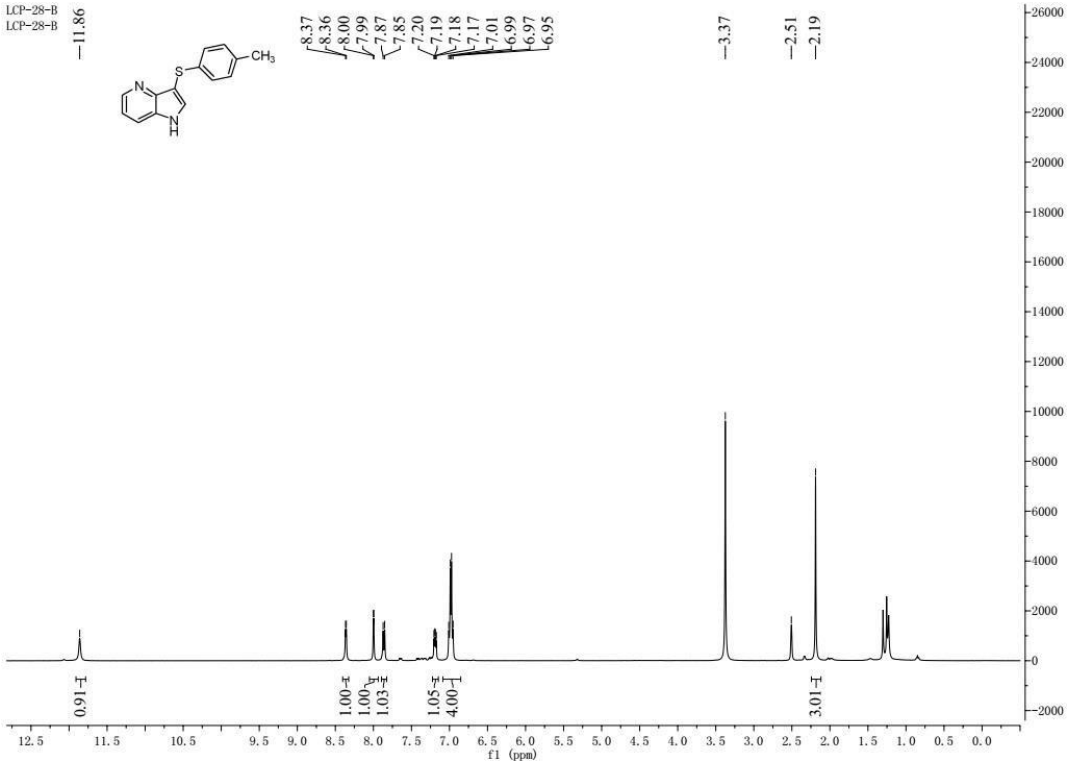
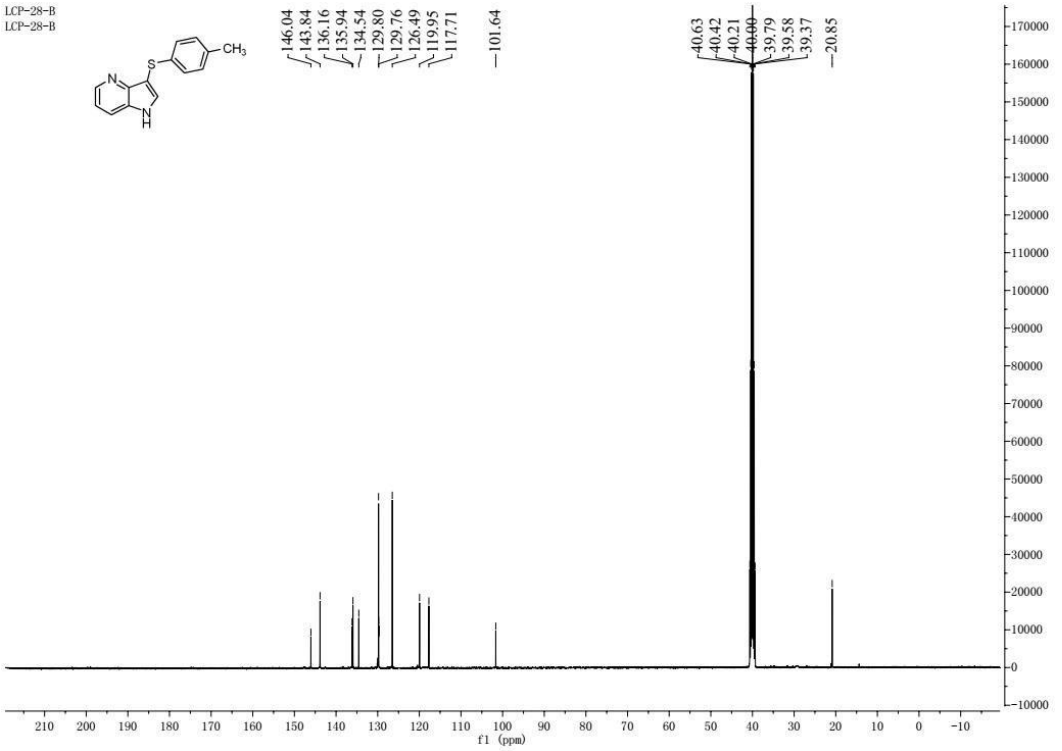


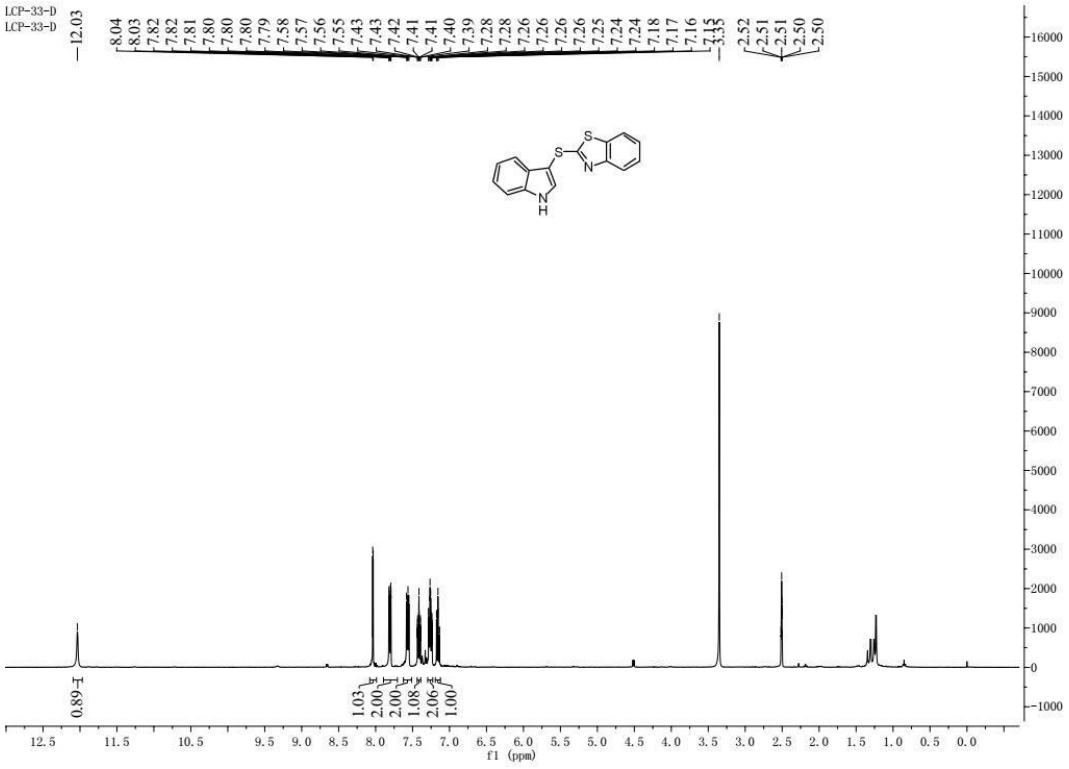


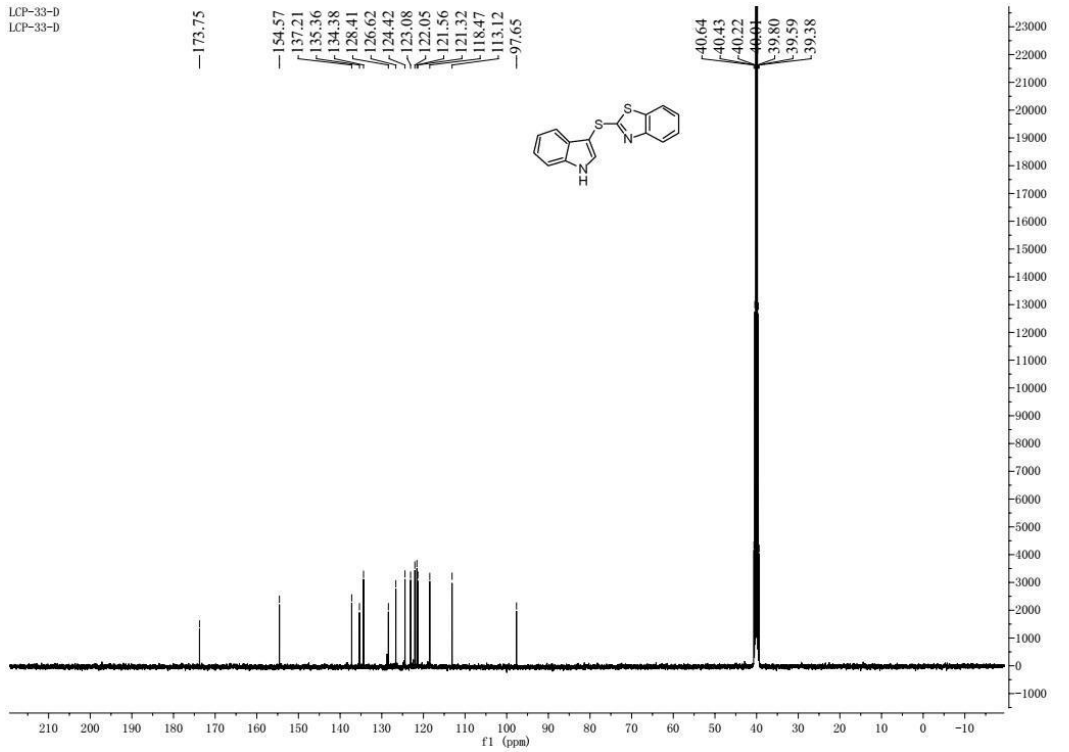
^{1}H and ^{13}C NMR Spectra for **6aj**



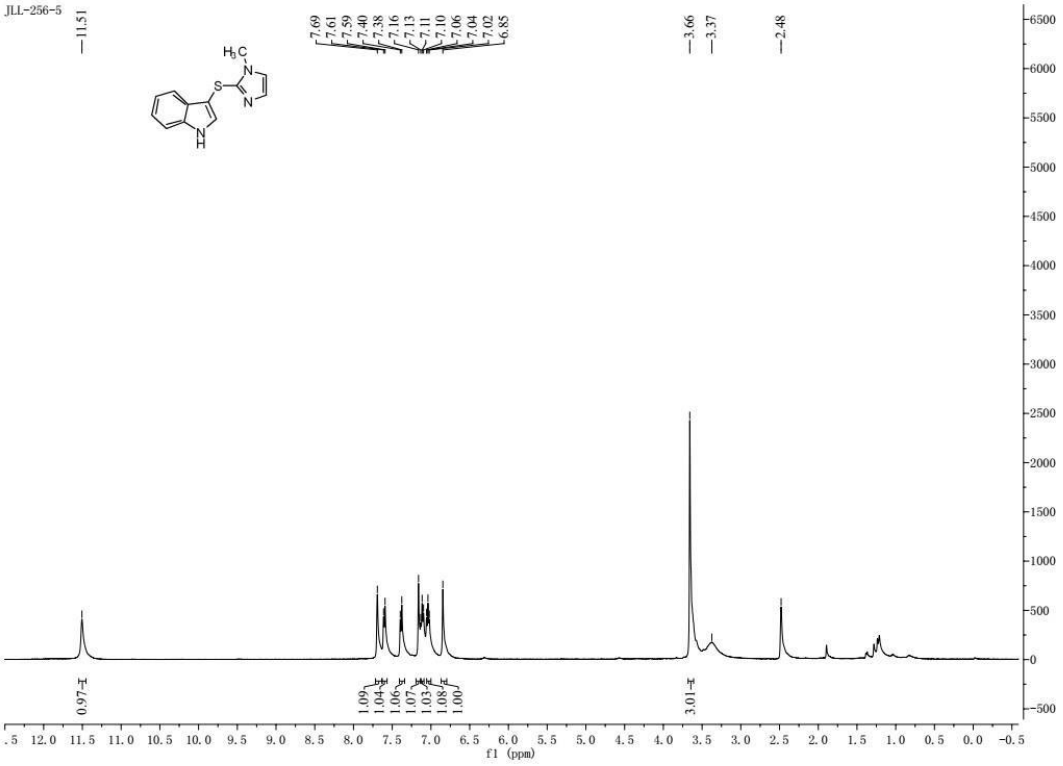


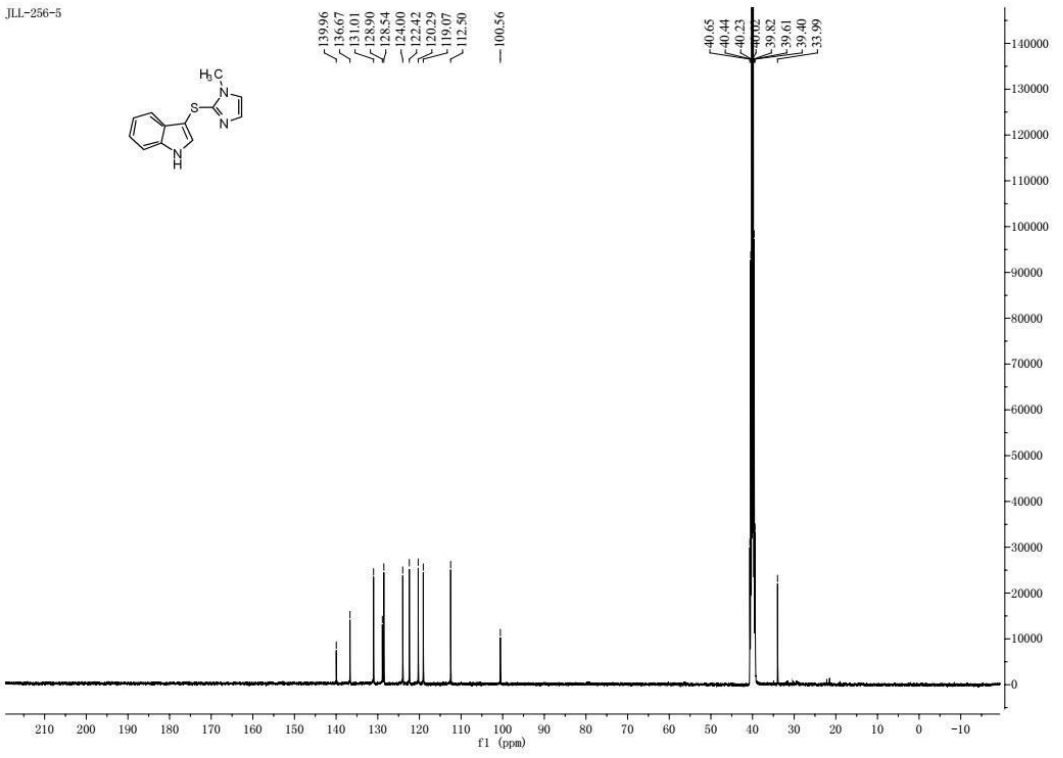
^{1}H and ^{13}C NMR Spectra for **6ak**





^{1}H and ^{13}C NMR Spectra for **6al**





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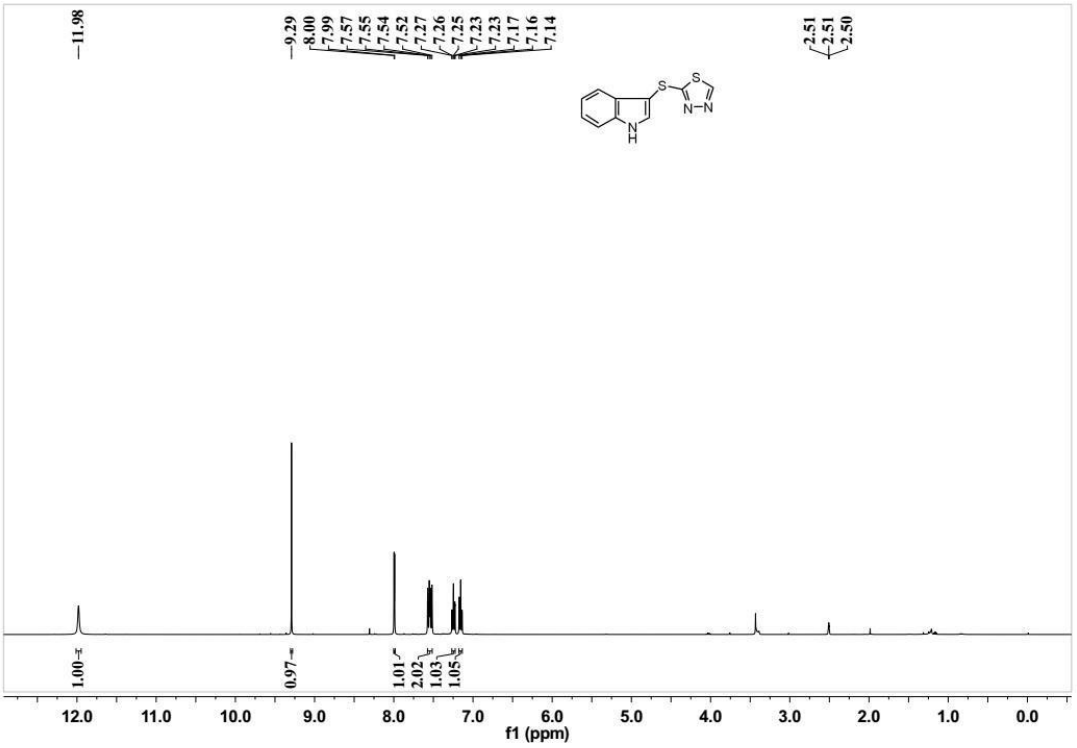

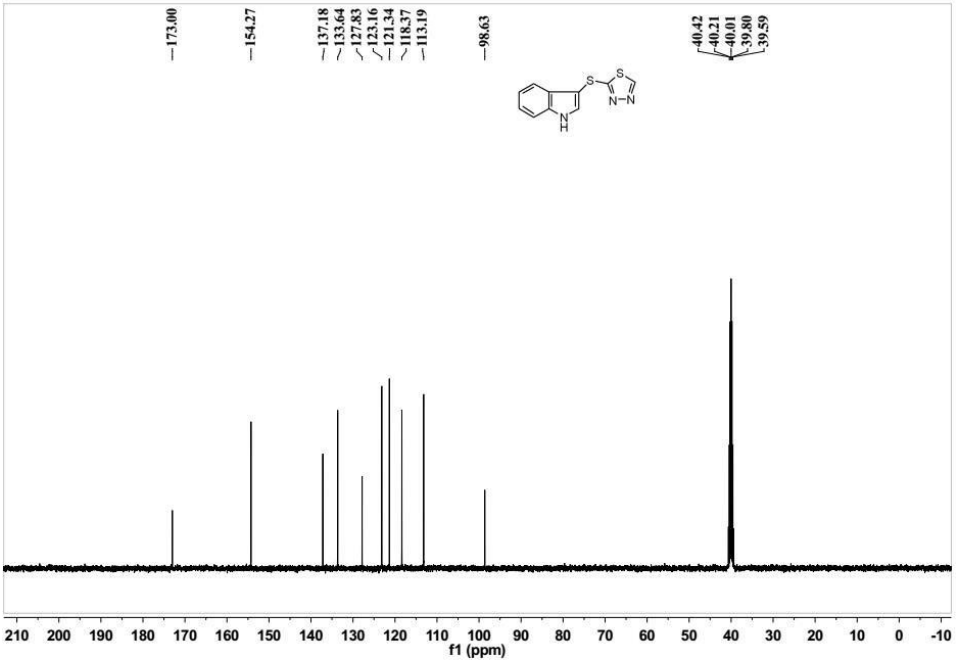

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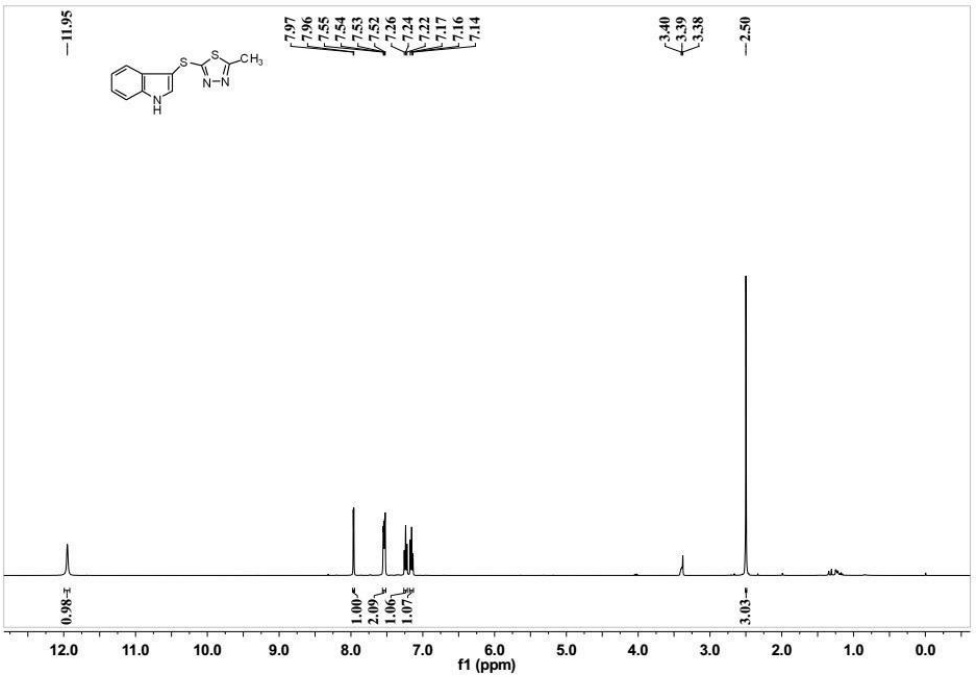
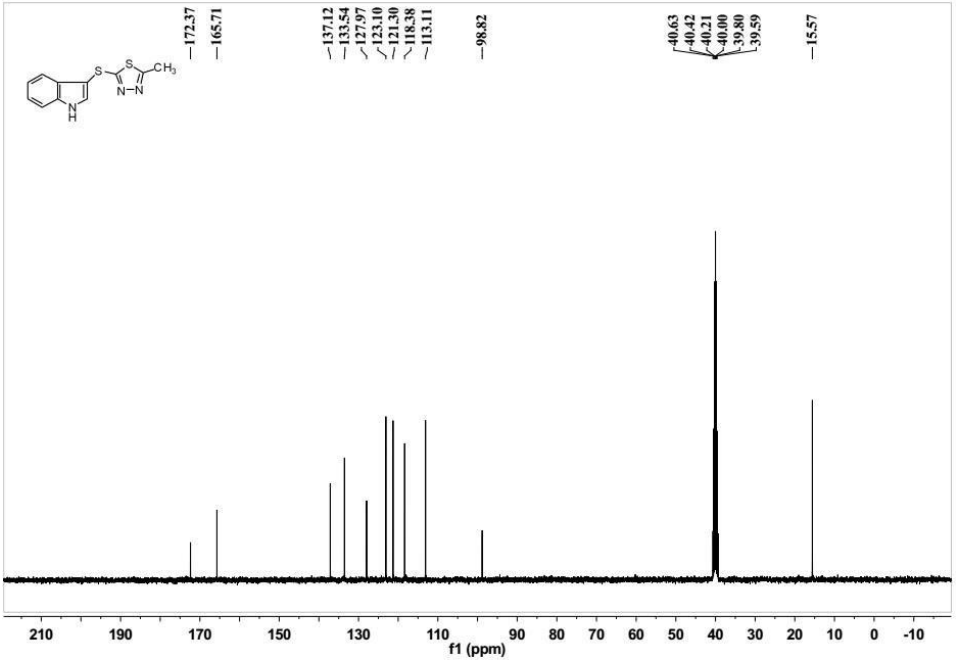
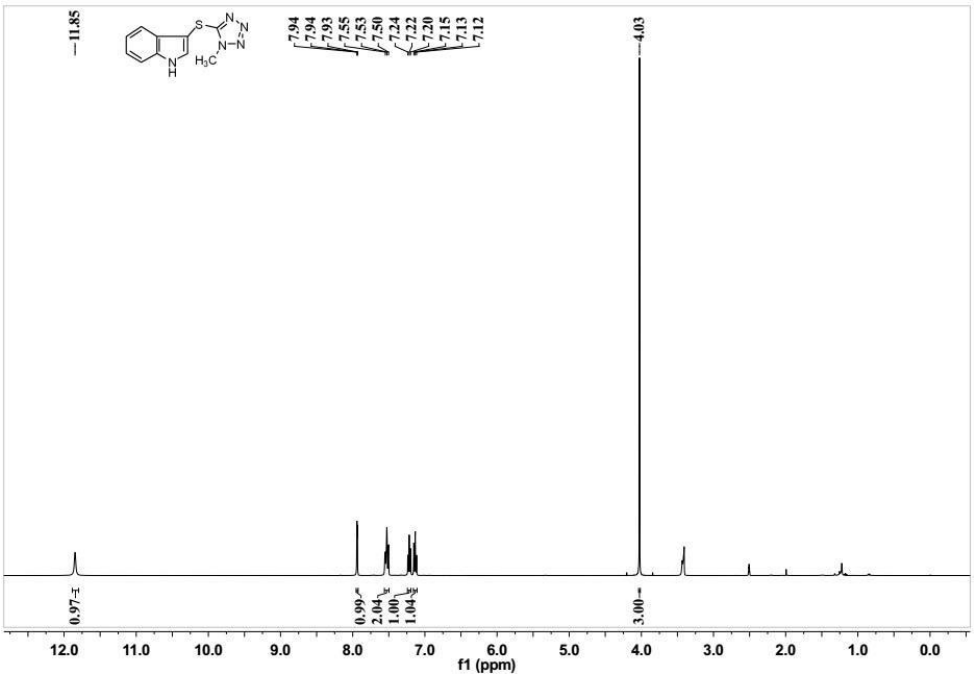
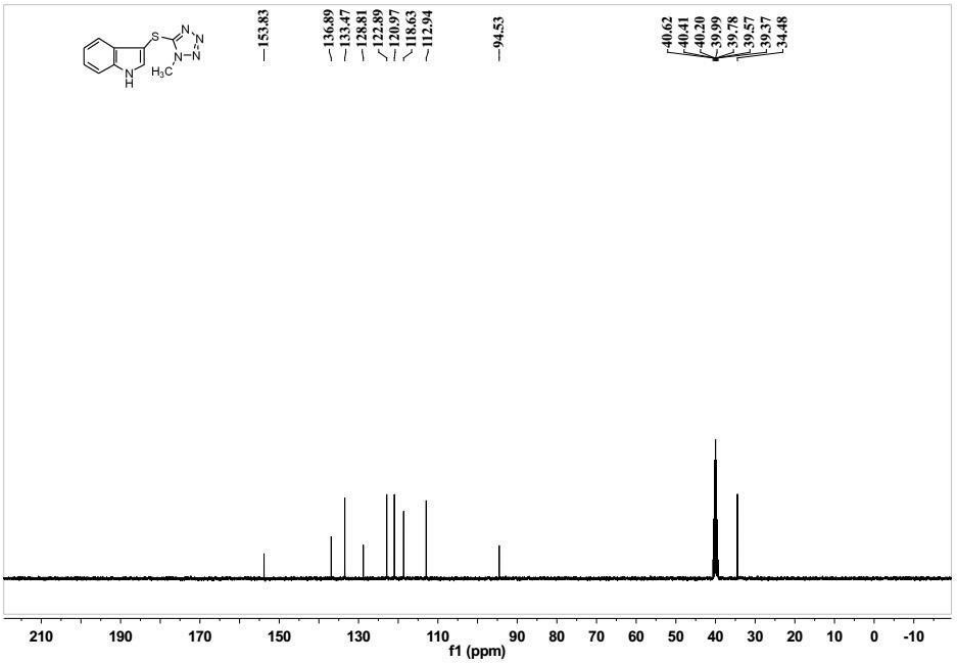


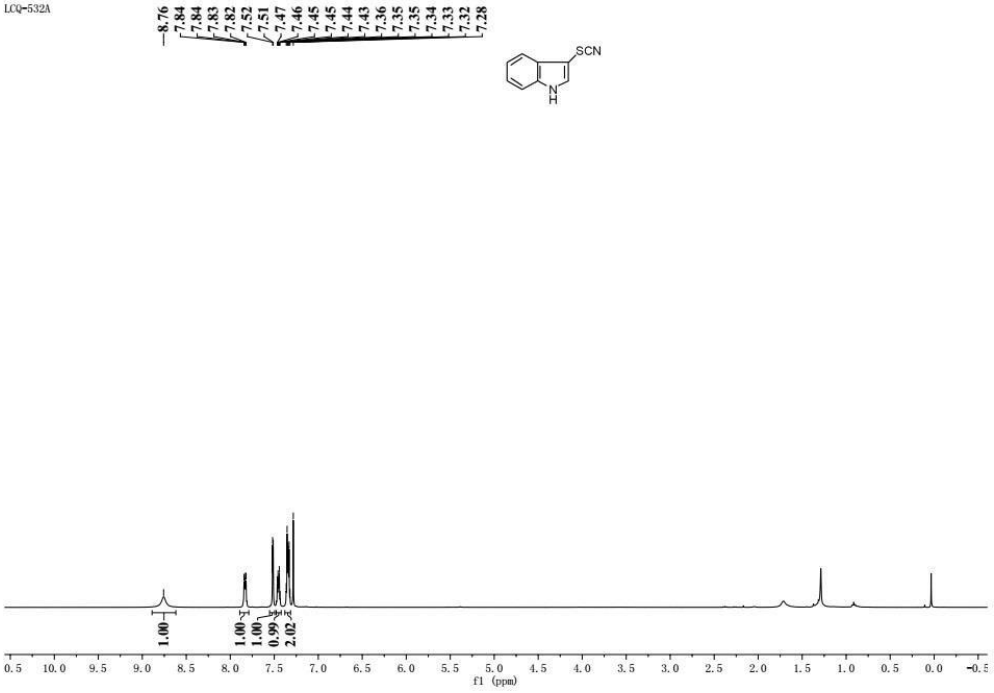


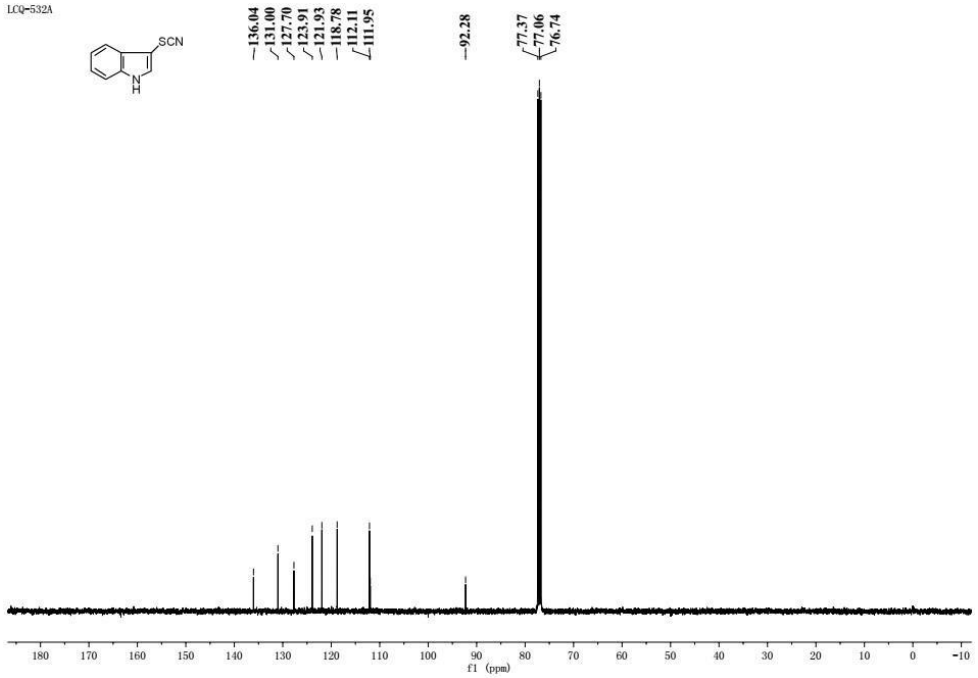
^{1}H and ^{13}C NMR Spectra for **6ao**



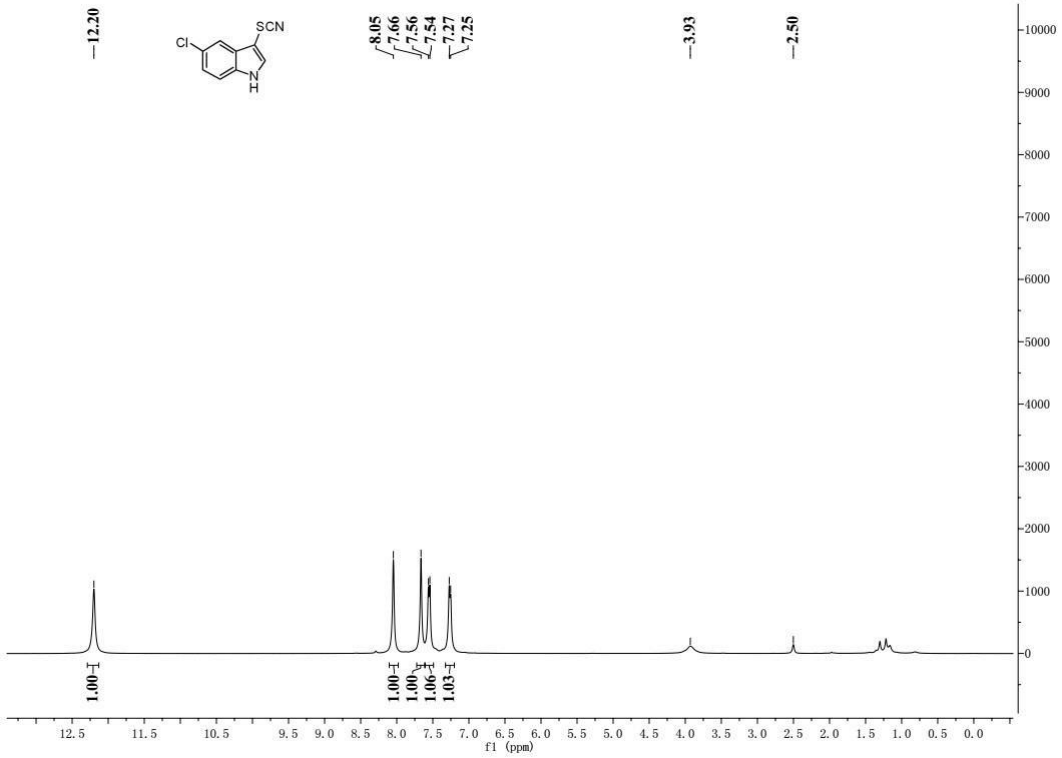


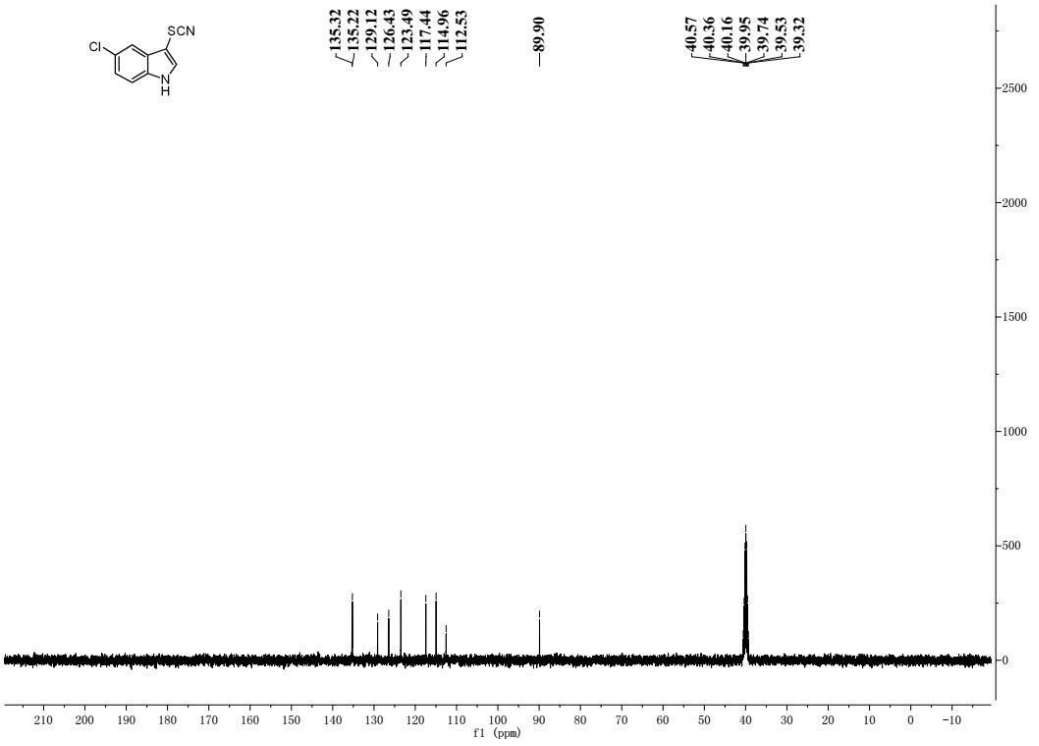
^{1}H and ^{13}C NMR Spectra for **7a**





^{1}H and ^{13}C NMR Spectra for **7b**





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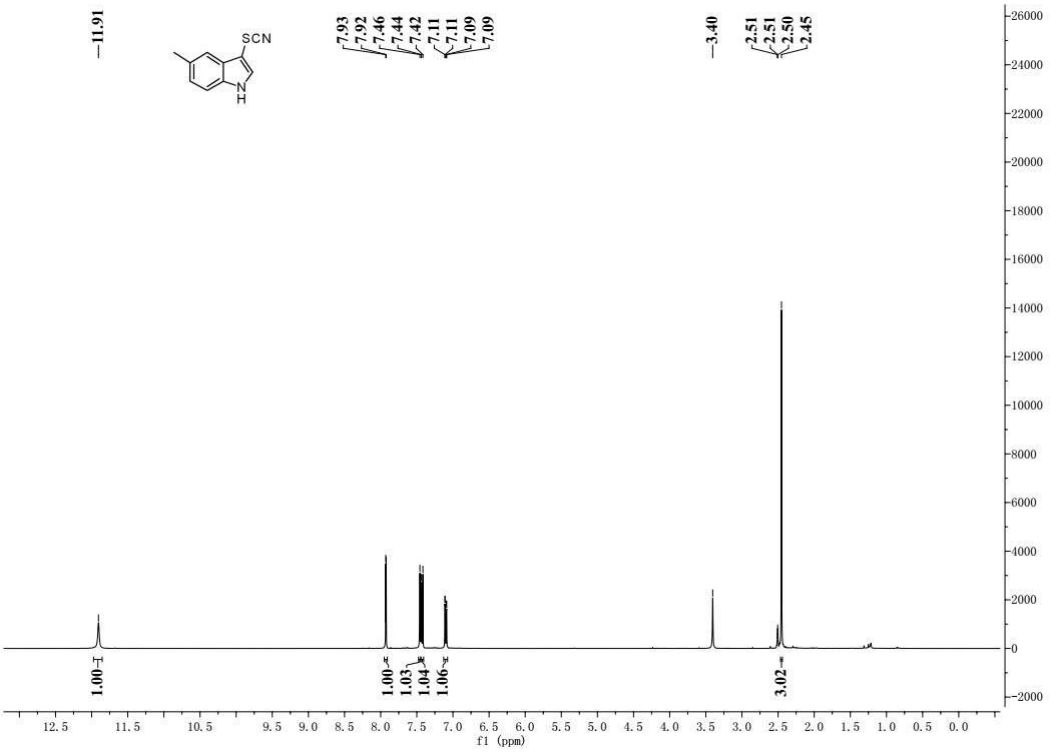
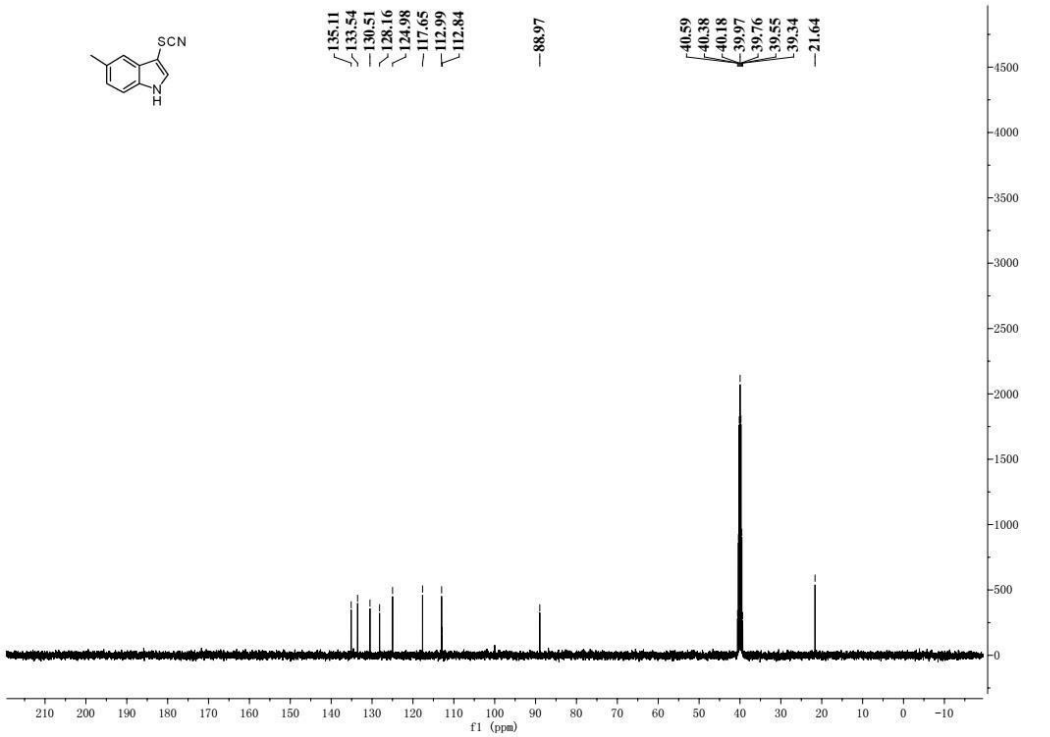
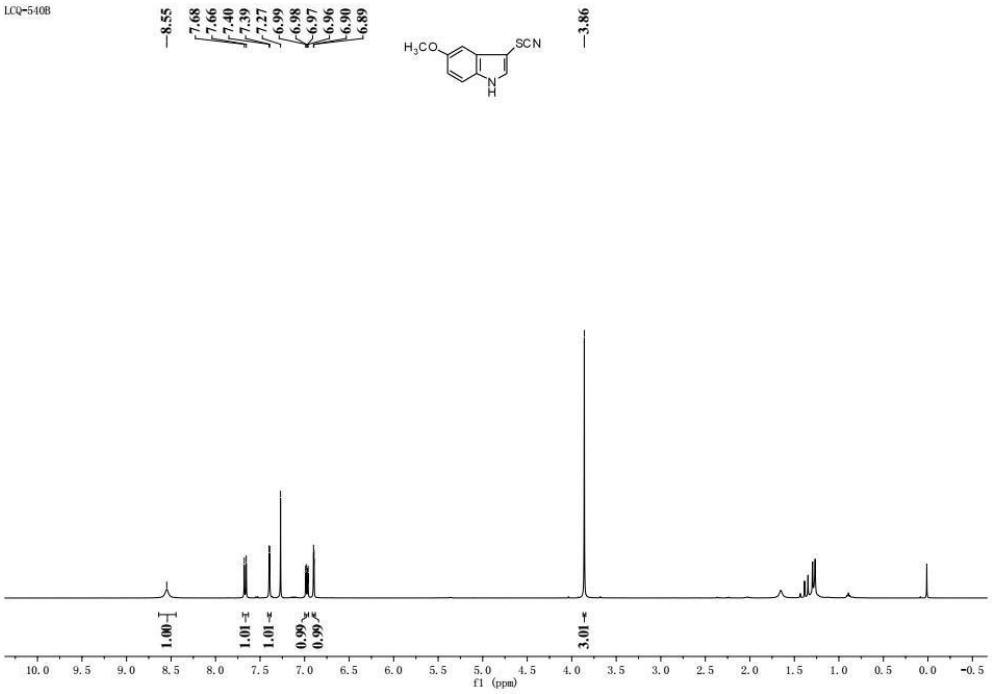
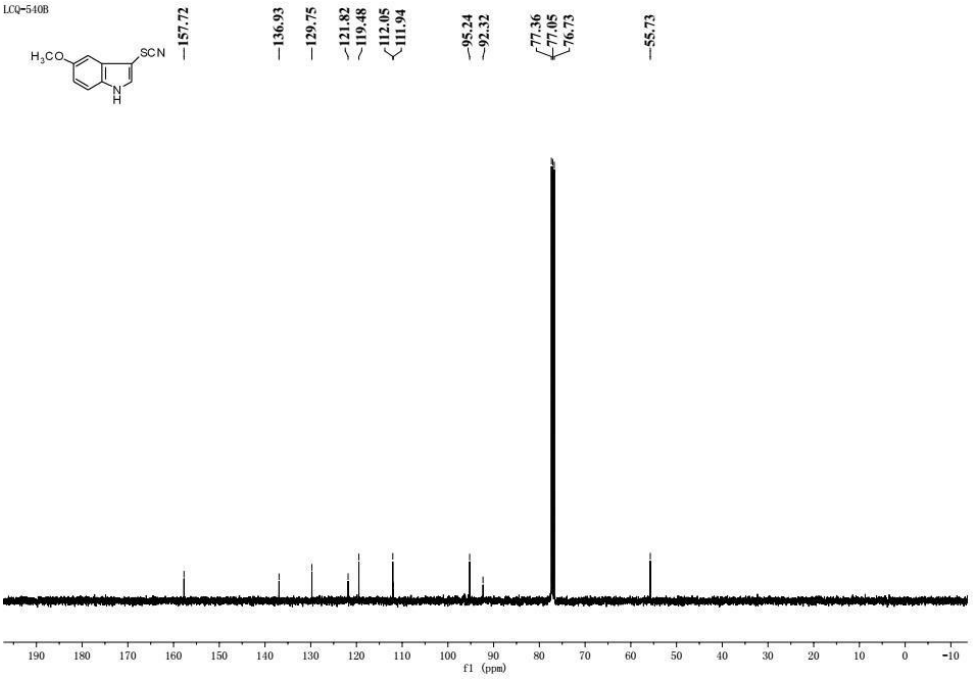


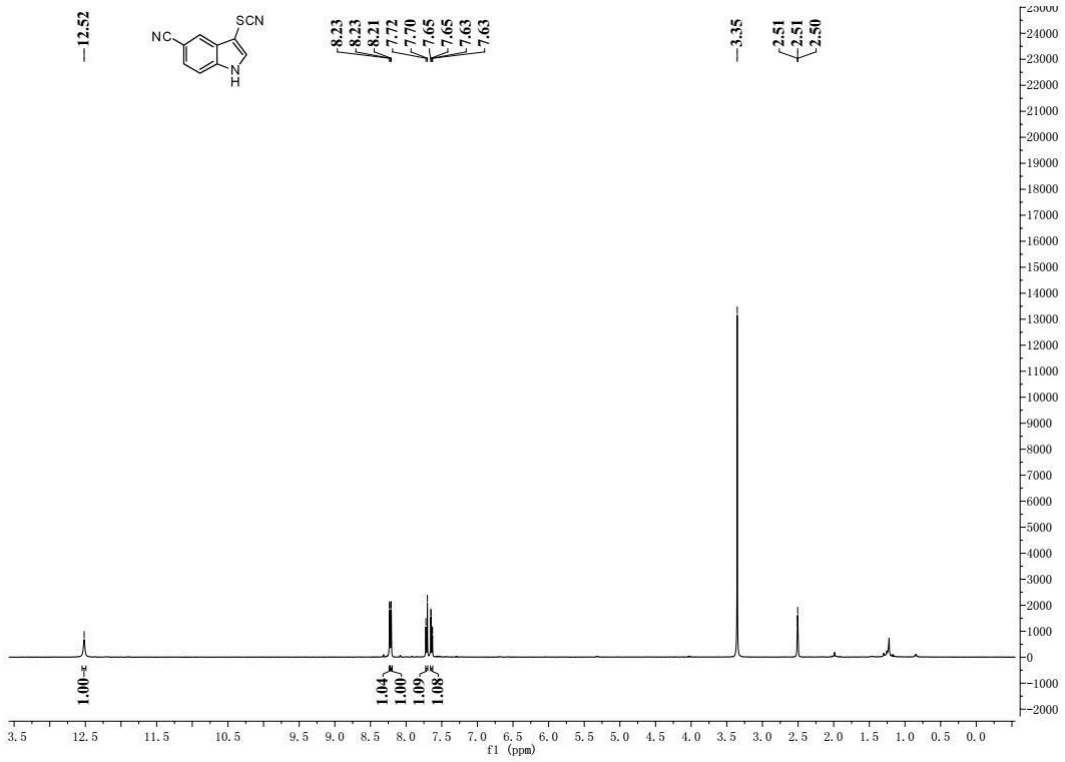


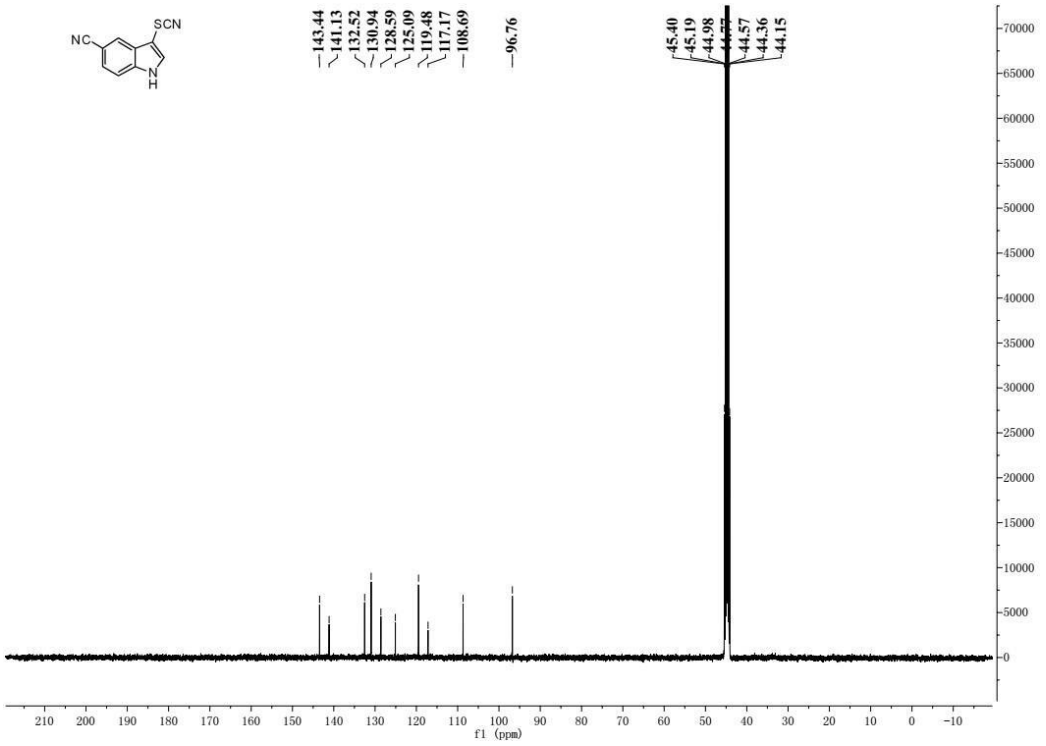
^{1}H and ^{13}C NMR Spectra for **7d**



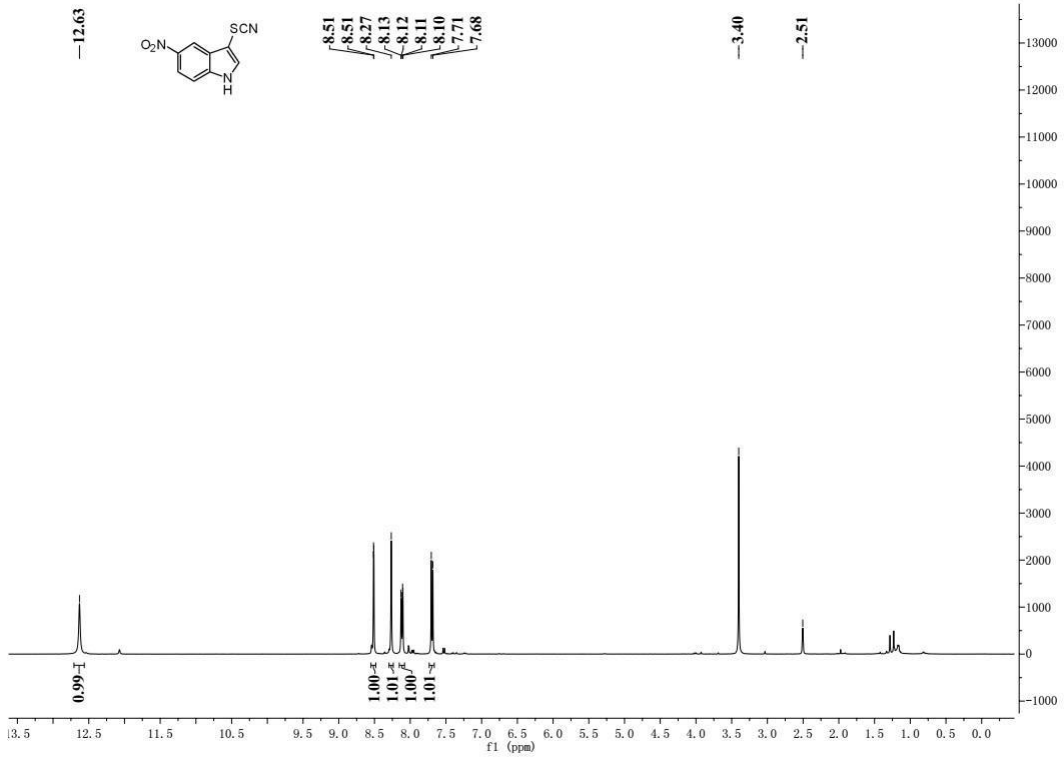


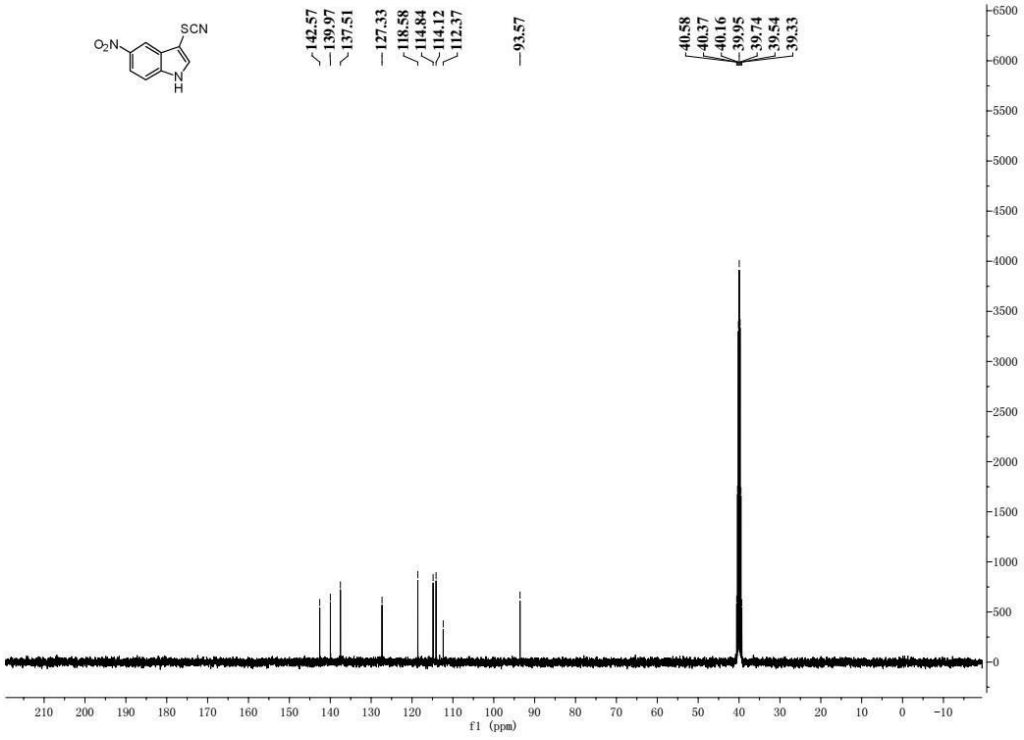
^{1}H and ^{13}C NMR Spectra for **7e**



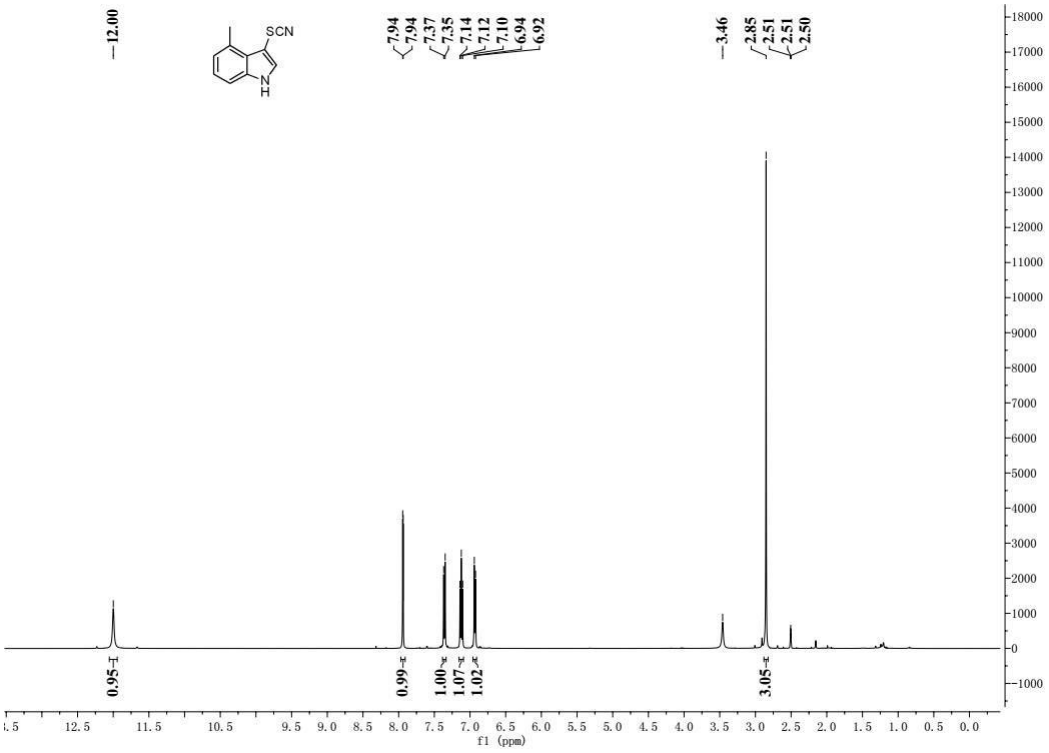


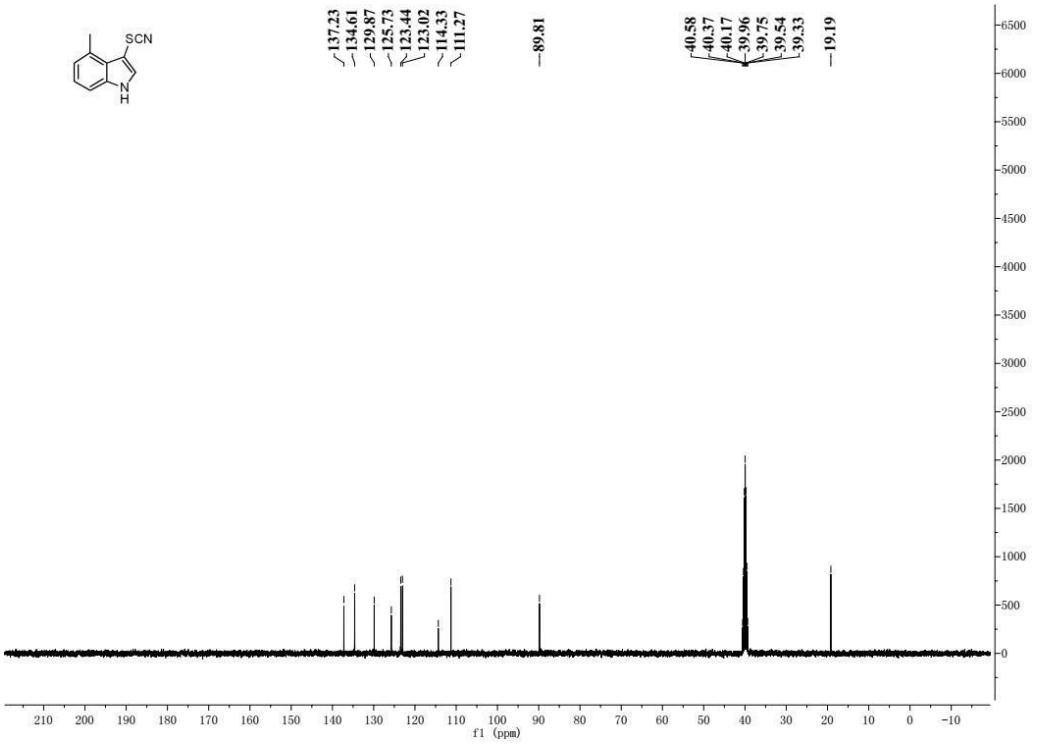
^{1}H and ^{13}C NMR Spectra for **7f**



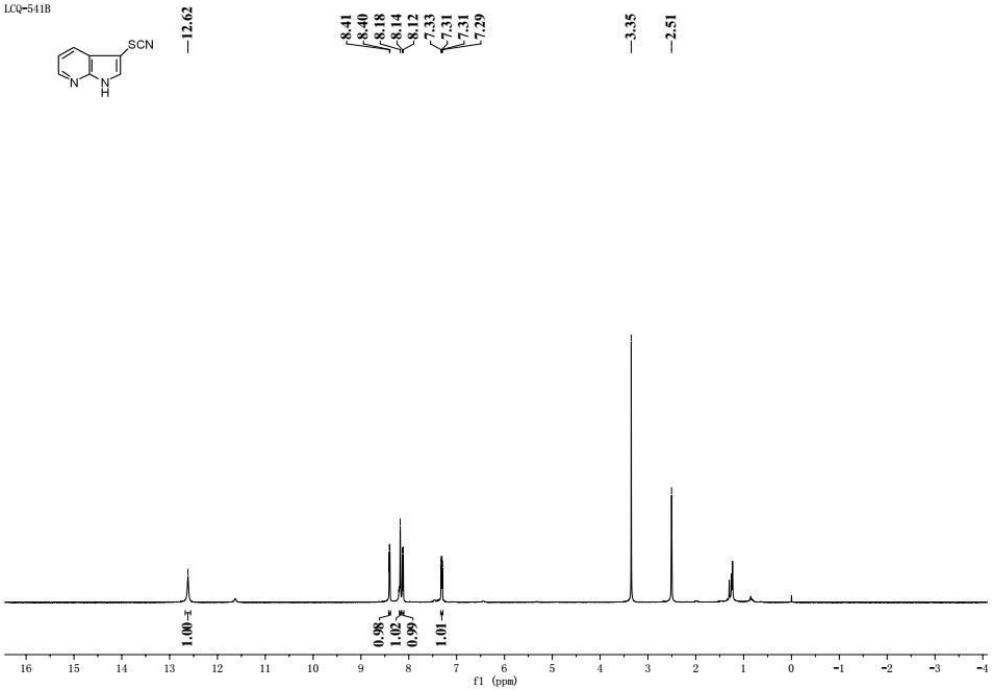


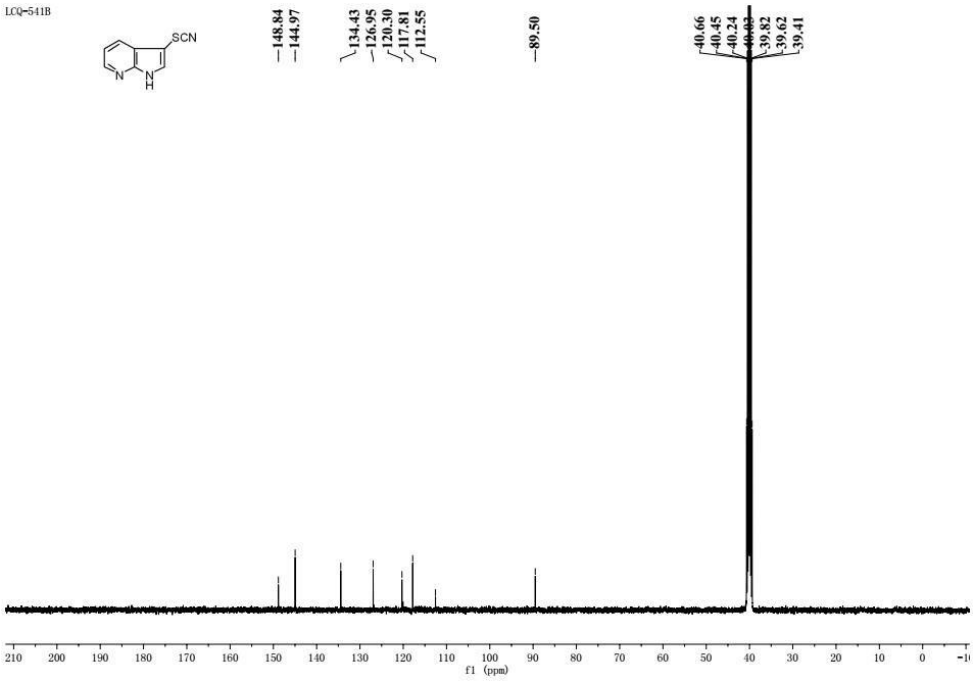
^{1}H and ^{13}C NMR Spectra for **7g**



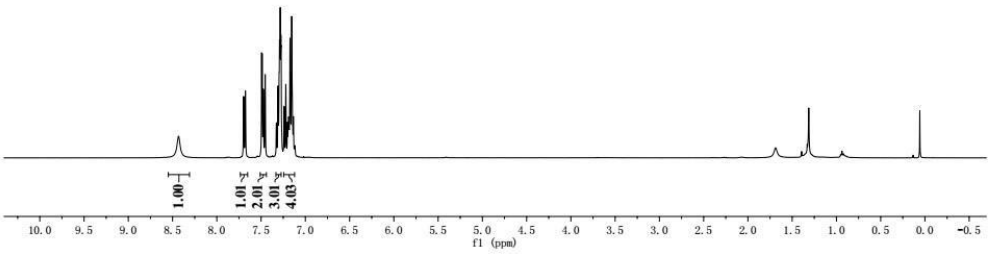


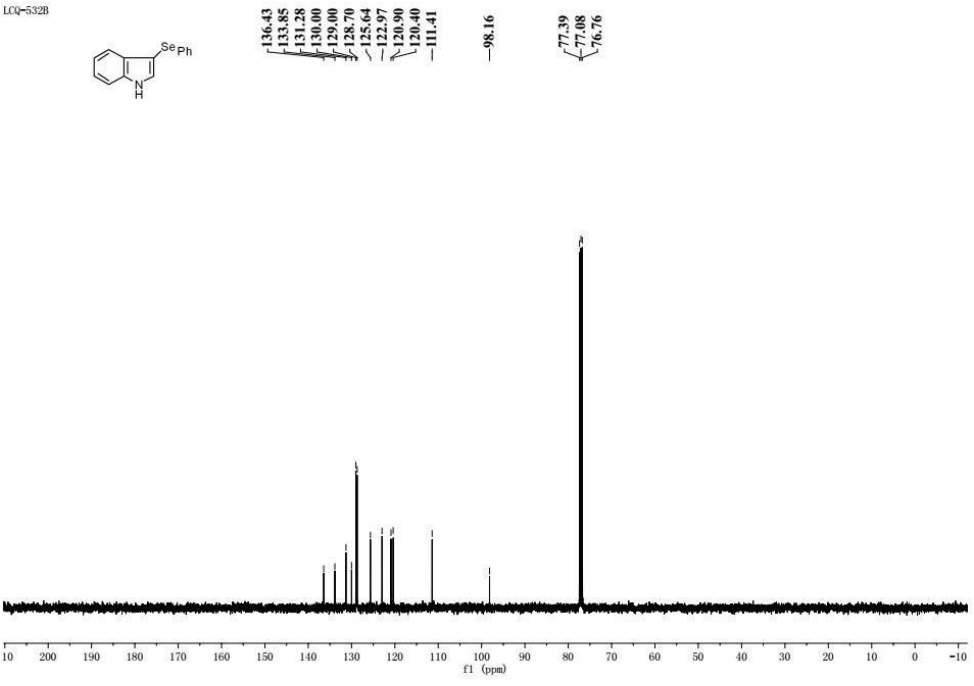
^{1}H and ^{13}C NMR Spectra for **7h**



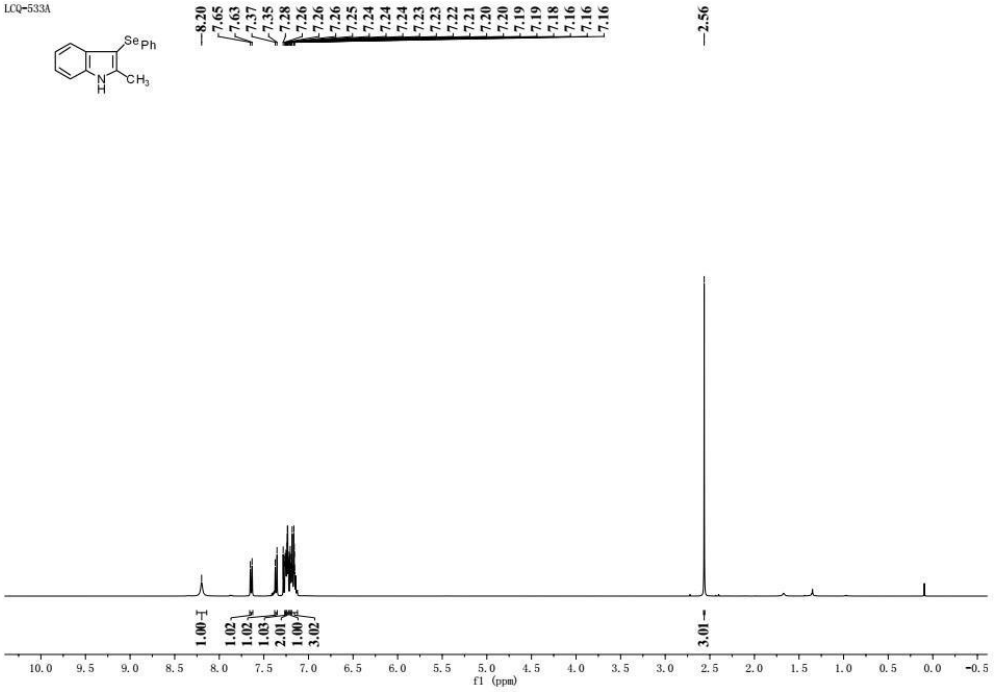


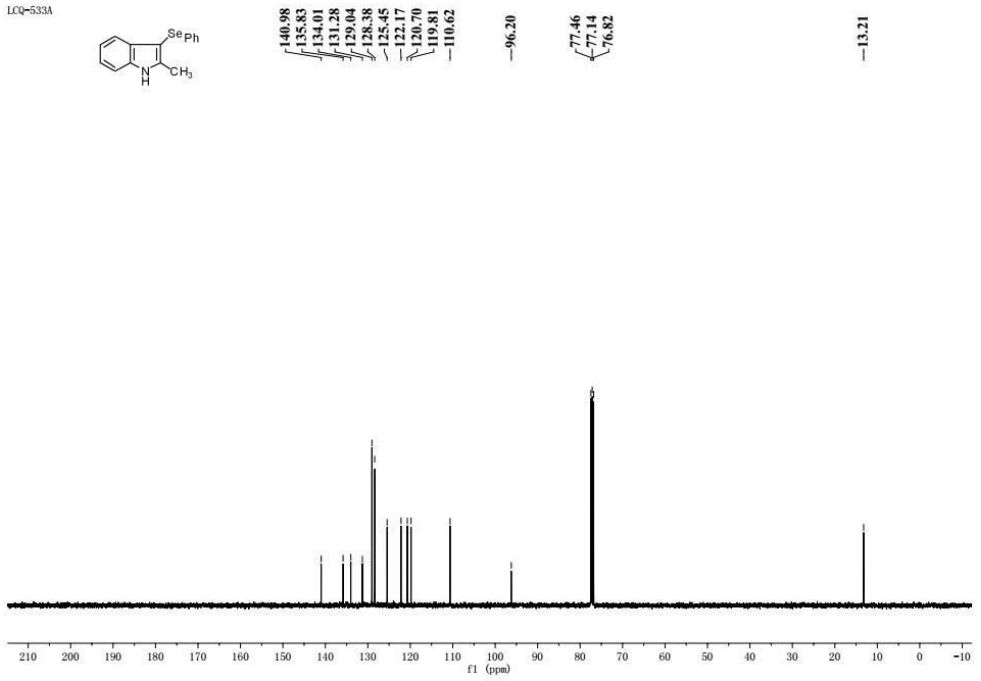
^{1}H and ^{13}C NMR Spectra for **9a**



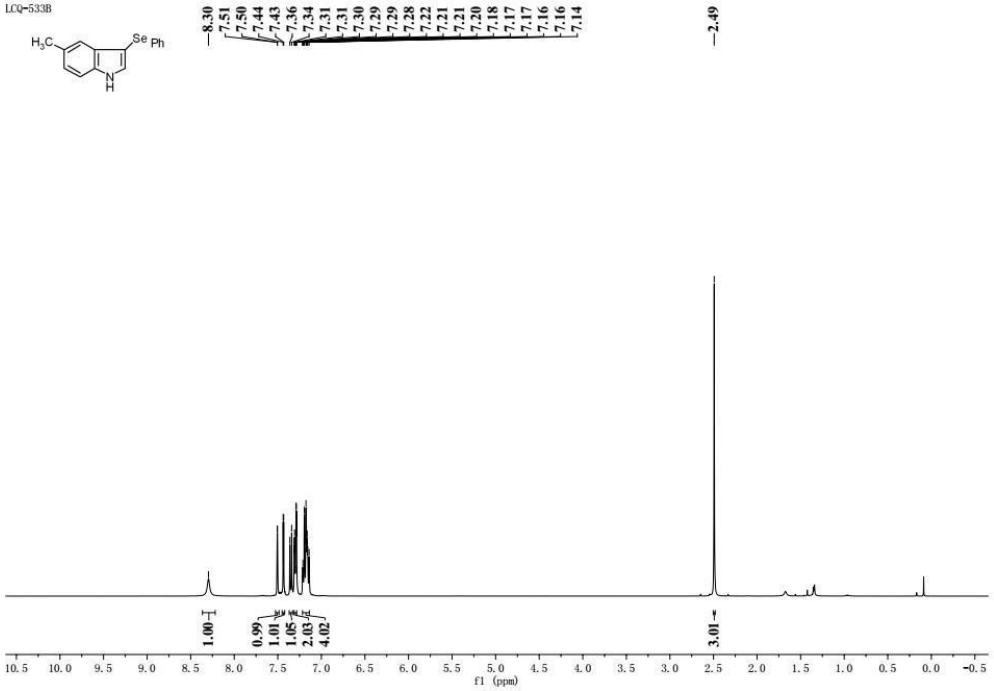


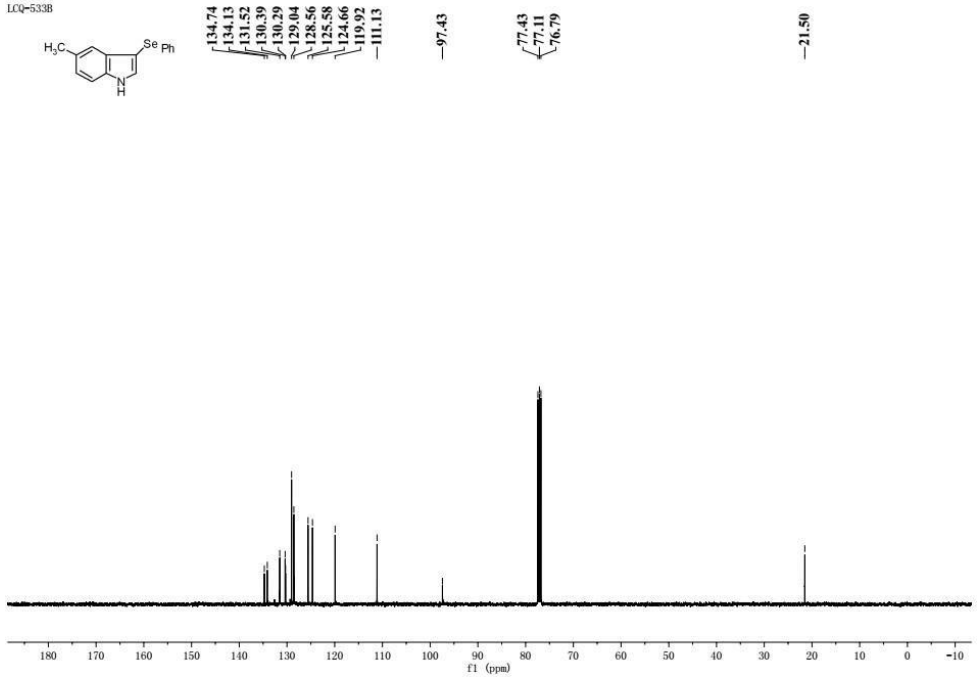
^{1}H and ^{13}C NMR Spectra for **9b**





^{1}H and ^{13}C NMR Spectra for **9c**





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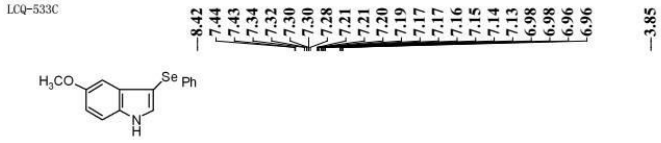

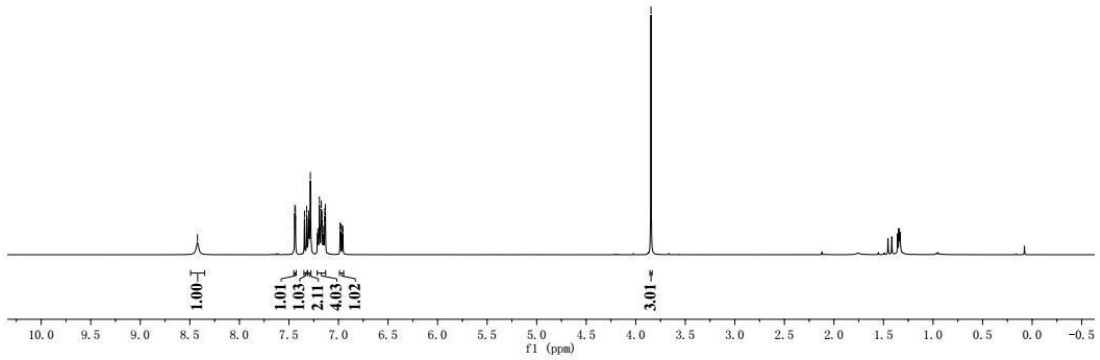

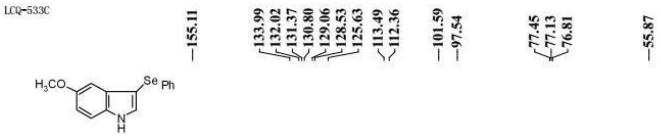

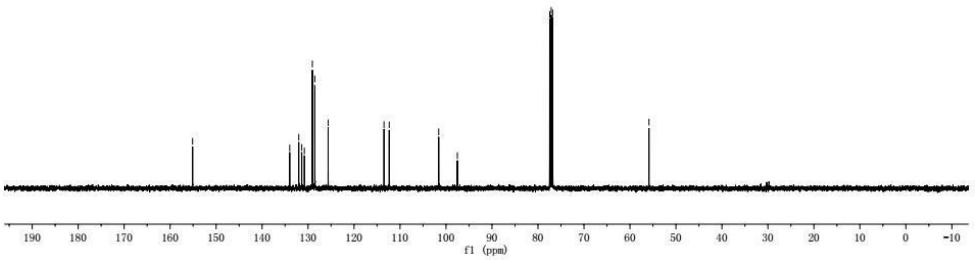

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