Supplementary Material (ESI) for Chemical Communications

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An efficient synthesis of 2-bromo(chloro)-3-selenyl(sulfenyl)indoles via tandem reactions of 2-(*gem*-dibromovinyl)anilines with diselenides(disulfides)

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Table of Contents for Supporting Information

1. General considerations

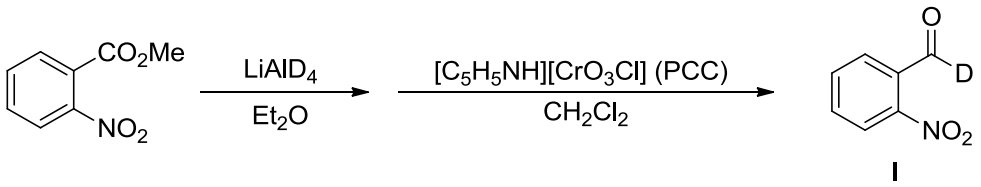
All the tandem reactions of *gem*-dihalovinyl substrate with diphenyldiselenide or diphenyldisulfide were carried out under an air atmosphere. ^{1}H NMR, ^{13}C NMR spectra were measured on a Bruker Avance NMR spectrometer (400 MHz or 100MHz, respectively) with $CDCl_3$ as solvent and recorded in ppm relative to internal tetramethylsilane standard. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, J, are reported in Hertz (Hz). High resolution mass spectroscopy data of the product were collected on a Waters Micromass GCT instrument.

Solvents, and general chemicals were purchased from commercial suppliers and used without further purification. All the *gem*-dihalovinyl substrates were synthesized according to the reported procedures in the literature (See: Y.-Q. Fang and M. Lautens, *J. Org. Chem.* 2008, **73**, 538).

2. Typical procedure for the tandem reaction

A sealable reaction tube equipped with a magnetic stirrer bar was charged with *gem*-dihalovinyl substrate (0.50 mmol), diphenyldiselenide or diphenyldisulfide (0.25 mmol), *t*-BuOLi (0.50 mmol), I_2 (0.050 mmol) and DMSO (2.0 mL). The rubber septum was then replaced by a Teflon-coated screw cap, and the reaction vessel placed in an oil bath at 110 °C. After stirring the mixture at this temperature for 12 h, it was cooled to room temperature and diluted with ethyl acetate, washed with water and brine, dried over by Mg_2SO_4. After the solvent was removed under reduced pressure, the residue was purified by column chromatography on silica gel to afford the corresponding product.

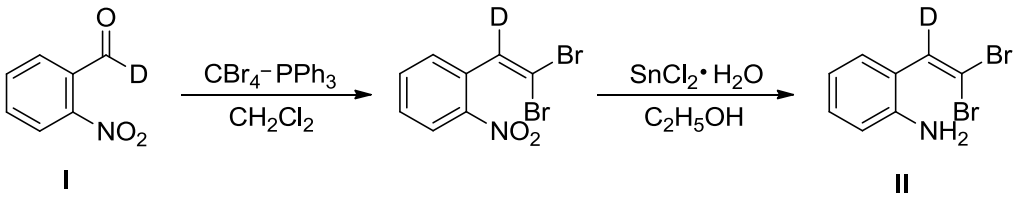
3. Typical synthetic procedure of 1a-D



I

To a suspension of methyl 2-nitrobenzoate (5.44 g, 30 mmol) in Et_2O (90 mL) was added $LiAlD_4$ (0.672 g, 16 mmol) in several portions at 0 ºC. After addition, the mixture was stirred at 0 ºC for another 2 h before quenched by pouring into HCl (1.0 mol/L, 100 mL). The resulting mixture was extracted with Et_2O (2×60 mL), washed with $NaHCO_3$ (aq. 20 mL), brine (20 mL), and dried over $MgSO_4$. The crude material was chromatographed with 25% EtOAc in hexanes to afford the benzyl alcohol as a brown solid (1.52 g, 33%), which was directly used in next step.

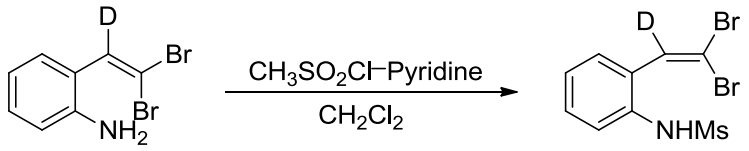
The benzyl alcohol (1.50 g) solution in DCM (30 mL) was added PCC (pyriddinium chlorochromate, 3.2 g), and the mixture was stirred at room temperature overnight. The resulting mixture was filtered through a celite pad. After the celite was washed with DCM (40 mL), the combined organic solvent was evaporated and chromatographed with 20% EtOAc in hexanes to give the deuterated 2-nitrobenzaldehyde-D (**I**) as a slightly yellow solid (1.19 g, 80%).



I II

To a solution of 2-nitrobenzaldehyde-D (**I**, 1.19 g, 7.76 mmol) and CBr_4 (3.86 g, 11.64 mmol) in DCM (40 mL) at 0 ºC was added dropwise a solution of PPh_3 (6.10 g, 23.28 mmol) in DCM (20 mL) by an addition funnel. The addition rate was controlled so that the internal temperature was at 1–5 ºC. After addition (about 1 h), the mixture was stirred for another 0.5 h before it was warmed to

room temperature, and stirred for an additional 1 h. The reaction mixture was filtered through a short plug of silica gel (12 g) and was washed with a copious amount of DCM until no product was found. Solvent was removed under reduced pressure to give a mixture of the desired intermediate and triphenylphosphine oxide. To the mixture was added EtOH (95%, 20 mL) and $SnCl_2 \cdot H_2O$ (8.76 g, 38.8 mmol). The suspension was heated at 100 ºC (reflux) for 45 min, and then cooled to room temperature. After most of the ethanol was removed under reduced pressure, H_2O (20 mL) and EtOAc (20 mL) were added. To the resulting mixture, solid K_2CO_3 was added carefully until pH > 10. The EtOAc layer was separated from the heterogeneous mixture, and the aqueous phase was extracted with EtOAc until it was free of the product (4×20 mL). The combined organic solution was washed with brine and dried over Na_2SO_4/K_2CO_3. Solvent was removed under reduced pressure and the residue was redissolved in Et_2O. The resulting precipitated $Ph_3P(O)$ was removed by filtration. The product was further purified by flash chromatography using 10% EtOAc in hexanes. The product (**II)** was obtained as a white solid (1.60 g, 74% yield over 2 steps).

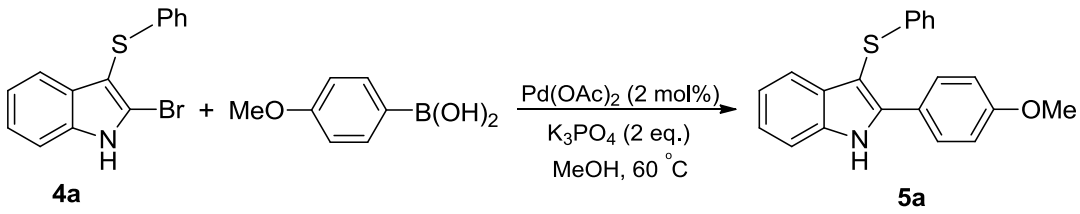


II **1a-D**

To a solution of **II** (1.38g, 5.0 mmol) and pyridine (0.8 mL, 10 mmol) in DCM (10 mL) was added dropwise MsCl (0.58 mL, 7.5 mmol) at 0 ºC. The mixture was warmed slowly to room temperature, stirred for an additional 12 h, and diluted with EtOAc (10 mL). The mixture was washed with $NaHSO_4$ (20%, 2×10 mL), $NaHCO_3$ (aq. 10 mL), brine (10 mL), and dried over anhydrous Na_2SO_4. The crude mixture was purified by column chromatography on silica gel to afford the product **1a**-D as a white solid (1.71 g, 96% yield). ^1H NMR ($CDCl_3$, 400 MHz) δ:

7.56–7.54 (m, 1H), 7.46–7.38 (m, 2H), 7.27–7.24 (m, 1H), 6.64 (s, 1H), 3.07 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 133.76, 130.00, 129.11, 125.79, 122.39, 95.60, 40.15. HRMS (EI) ($[M]^+$) Calcd. for $C_9H_8DBr_2NO_2S$: 353.8784, Found: 353.8788.

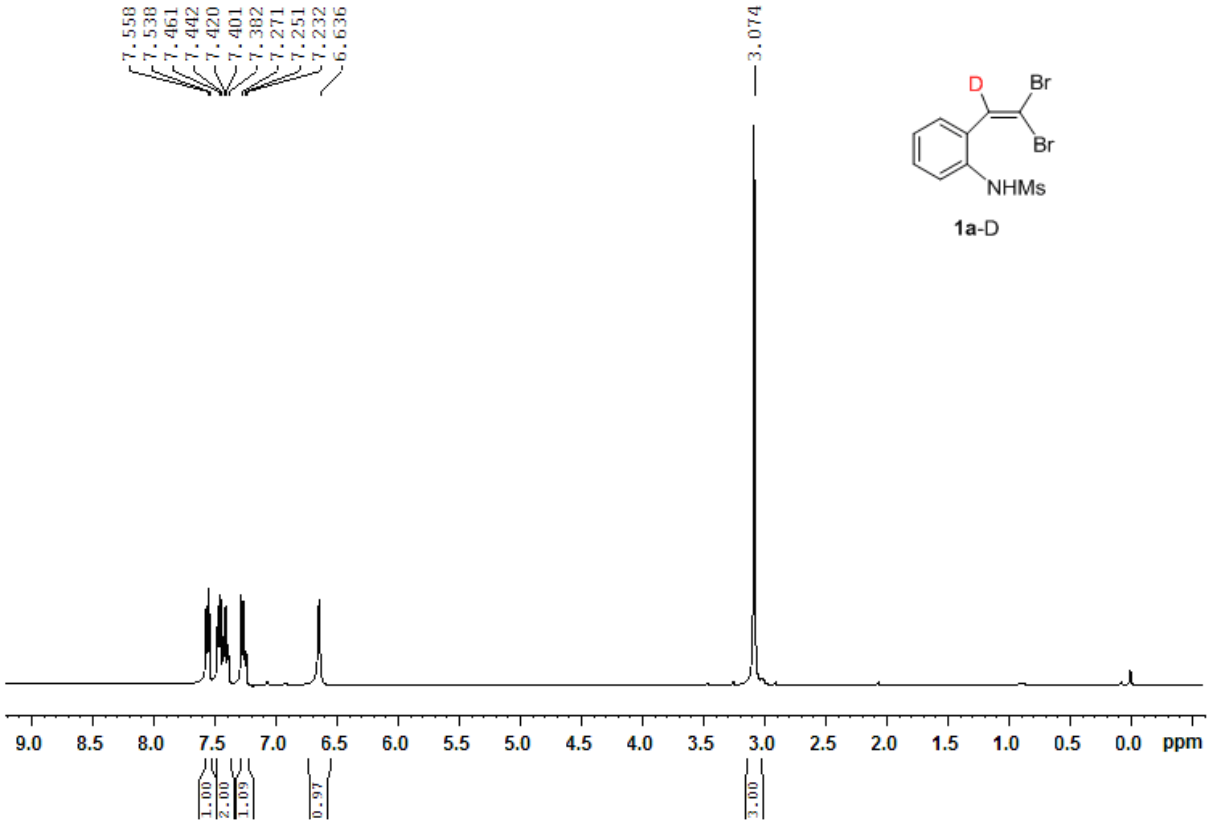
4. Suzuki reaction of 4a with 4-$MeOC_6H_4B(OH)_2$

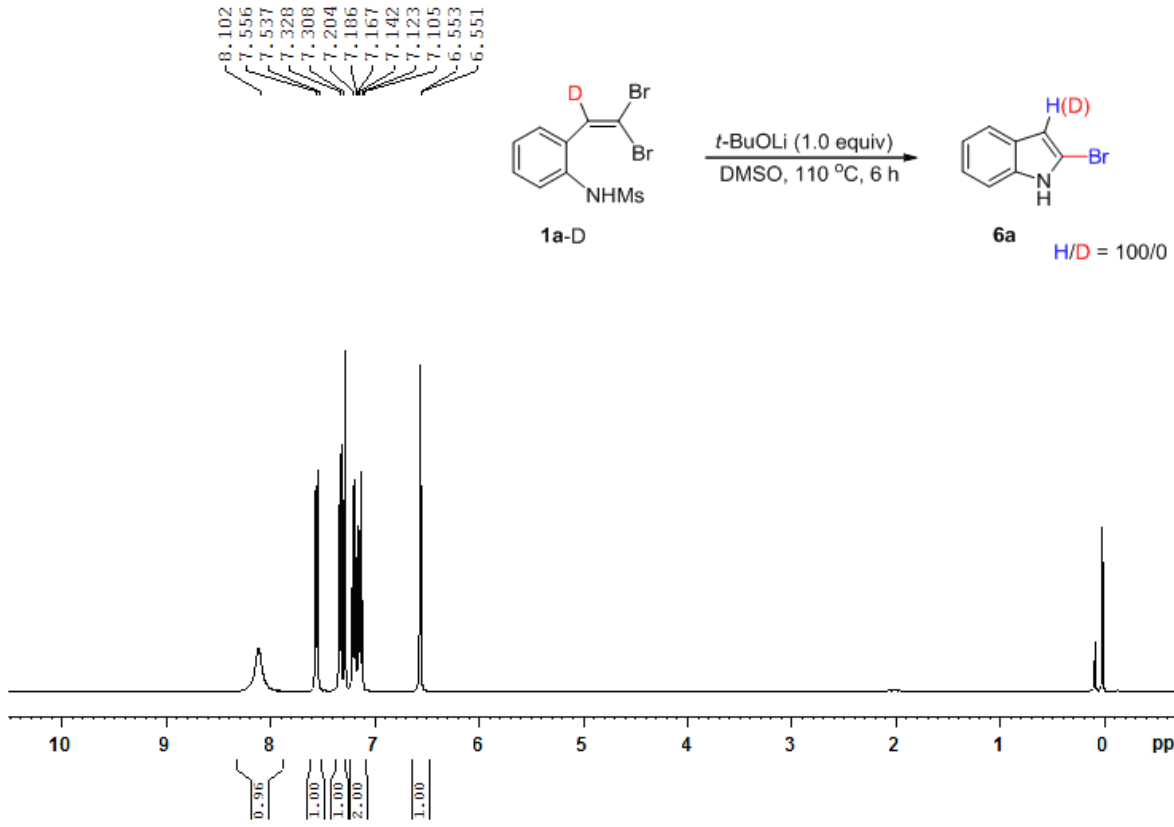


4a **5a**

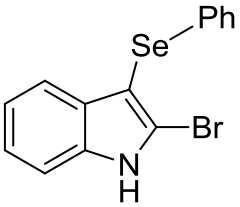
A mixture of **4a** (0.50 mmol), *p*-methoxyphenylboronic acid (0.75 mmol), $Pd(OAc)_2$ (0.0010 mmol), K_3PO_4 (1.0 mmol) in methanol (2.0 mL) was stirred at 60 °C for 10 h until complete consumption of starting material as monitored by TLC. The mixture was added to brine (10 mL) and extracted with diethyl ether (3×10 mL). The solvent was concentrated in vacuo and the product **5a** was isolated by chromatography on a silica gel column (152 mg, 92% yield).

5. The ^{1}H NMR spectra of isotope experiments



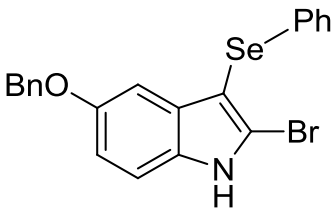


6. Characterization data for all products



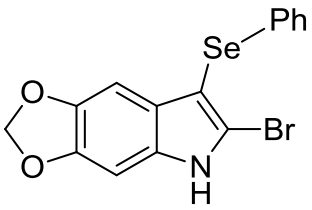
3a: Yellow solid, m.p. 90.5–91.2 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52 (s, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.30–7.23 (m, 3H), 7.21–7.13 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.45, 132.35, 130.56, 129.17, 129.03, 125.97, 123.13, 121.36, 120.17, 118.68, 110.59, 101.39. HRMS (EI) ($[M]^+$) Calcd. for $C_{14}H_{10}N^{79}Br^{74}Se$: 344.9221, Found: 344.9217.



3b: Yellow oil.

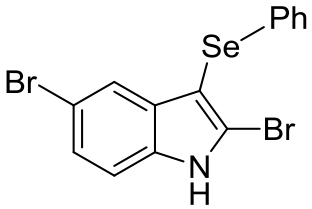
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.50 (s, 1H), 7.45–7.44 (m, 2H), 7.39–7.36 (m, 2H), 7.34–7.32 (m, 1H), 7.27–7.23 (m, 3H), 7.17–7.14 (m, 4H), 6.97–6.94 (m, 1H), 5.06 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.53, 137.24, 132.39, 131.62, 131.40, 129.25, 129.06, 128.49, 127.85, 127.67, 126.01, 118.77, 114.04, 111.52, 103.49, 101.22, 70.74. HRMS (EI) ($[M]^+$) Calcd. for $C_{21}H_{16}N^{74}SeOBr$: 450.9640, Found: 450.9636.



3c: Yellow solid, m.p. 109.5–110.1 °C.

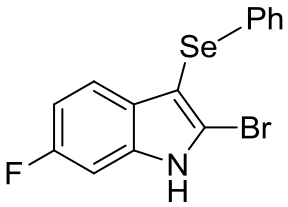
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (s, 1H), 7.27–7.14 (m, 5H), 6.96 (s, 1H), 6.80 (s, 1H), 5.94 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.68, 144.13, 132.40, 131.19, 129.14, 129.10, 126.03, 125.08,

115.21, 101.41, 100.89, 99.22, 91.80. HRMS (EI) ([M]$^+$) Calcd. for $C_{15}H_{10}NO_2{}^{79}Br^{74}Se$: 388.9120, Found: 388.9126.



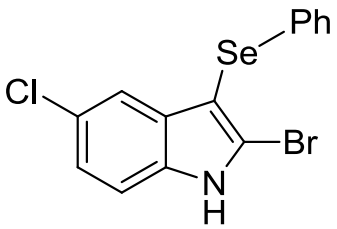
3d: White Solid. m.p. 127.5–127.7 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.58 (s, 1H), 7.74 (s, 1H), 7.32 (d, *J* = 8.7 Hz, 1H), 7.26 (d, *J* = 6.8 Hz, 2H), 7.22–7.18 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.13, 132.37, 131.96, 129.37, 129.21, 126.31, 126.19, 122.80, 120.11, 114.98, 112.10, 101.27. HRMS (EI) ([M]$^+$) Calcd. for $C_{14}H_9N^{74}SeBr_2$: 422.8326, Found: 422.8330.



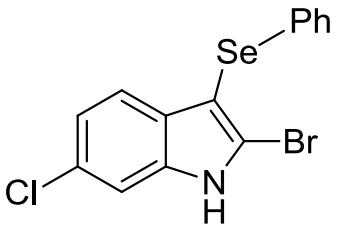
3e: White Solid. m.p. 133.2–133.4 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52 (s, 1H), 7.51–7.48 (m, 1H), 7.28–7.17 (m, 5H), 7.06–7.04 (m, 1H), 6.96–6.91 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.36 (d, *J* = 238.9 Hz), 136.19 (d, *J* = 12.5 Hz), 132.08, 129.39, 129.13, 127.12 (d, *J* = 1.1 Hz), 126.21, 121.22 (d, *J* = 9.9 Hz), 118.26 (d, *J* = 3.0 Hz), 110.16 (d, *J* = 24.3 Hz), 101.75, 97.32 (d, *J* = 26.7 Hz). HRMS (EI) ([M]$^+$) Calcd. for $C_{14}H_9N^{74}SeFBr$: 362.9127, Found: 362.9125.



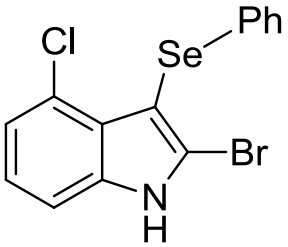
3f: Yellow solid, m.p. 124.5–124.8 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.60 (s, 1H), 7.57 (s, 1H), 7.28–7.17 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.86, 132.02, 131.81, 129.34, 129.20, 127.39, 126.28, 123.57, 120.25, 119.72, 111.78, 101.26. HRMS (EI) ($[M]^+$) Calcd. for $C_{14}H_9NCl^{79}Br^{74}Se$: 378.8832, Found: 378.8835.



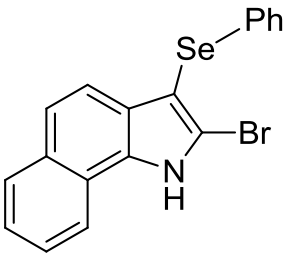
3g: Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.70 (s, 1H), 7.48 (d, *J* = 8.4 Hz, 1H), 7.36 (s, 1H), 7.27–7.12 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.67, 131.99, 129.37, 129.23, 129.20, 129.13, 126.22, 122.19, 121.16, 119.19, 110.61, 101.88. HRMS (EI) ($[M]^+$) Calcd. for $C_{14}H_9N^{74}SeClBr$: 378.8832, Found: 378.8827.



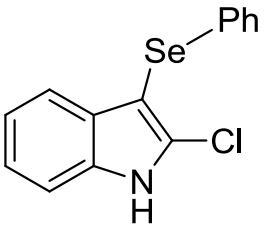
3h: White solid. m.p. 111.2–111.5 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.72 (s, 1H), 7.31–7.26 (m, 3H), 7.21–7.10 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.76, 134.22, 129.28, 129.01, 126.51, 126.18, 125.95, 123.40, 122.76, 121.82, 109.57, 100.26. HRMS (EI) ($[M]^+$) Calcd. for $C_{14}H_9N^{74}SeClBr$: 378.8832, Found: 378.8830.



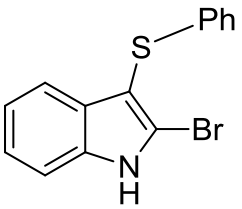
3i: White Solid. m.p. 133.9-134.2 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 9.23 (s, 1H), 7.97–7.93 (m, 2H), 7.68–7.66 (m, 1H), 7.58–7.55 (m, 2H), 7.51–7.47 (m, 1H), 7.30–7.27 (m, 2H), 7.19–7.14 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 132.55, 131.53, 130.72, 129.22, 129.09, 129.07, 127.09, 126.11, 126.03, 124.70, 122.29, 120.82, 119.73, 119.15, 115.38, 103.29. HRMS (EI) ($[M]^+$) Calcd. for $C_{18}H_{12}N^{74}SeBr$: 394.9378, Found: 394.9374.



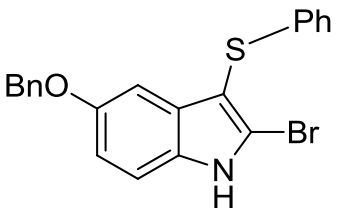
3j: Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.51 (s, 1H), 7.59 (d, *J* = 7.8 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.29–7.25 (m, 3H), 7.21–7.14 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.92, 132.37, 130.39, 130.11, 129.28, 129.08, 126.04, 123.26, 121.48, 120.18, 110.63, 97.41. HRMS (EI) ($[M]^+$) Calcd. for $C_{14}H_{10}N^{74}SeCl$: 300.9727, Found: 300.9726.



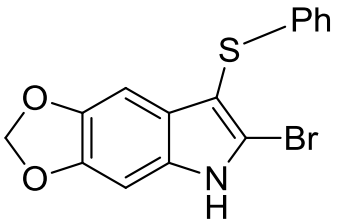
4a: Yellow solid, m.p. 102.4–103.1 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.47 (s, 1H), 7.62 (d, *J* = 7.9 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.30–7.26 (m, 1H), 7.24–7.16 (m, 5H), 7.15–7.11 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.41, 136.17, 129.47, 128.78, 126.27, 125.18, 123.22, 121.41, 119.24, 118.31, 110.80, 104.61. HRMS (EI) ($[M]^+$) Calcd. for $C_{14}H_{10}BrNS$: 302.9717, Found: 302.9721.



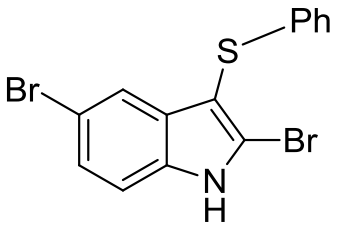
4b: White solid, m.p. 116.2–116.6 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (s, 1H), 7.46–7.44 (m, 2H), 7.40–7.31 (m, 3H), 7.24–7.20 (m, 3H), 7.15–7.10 (m, 4H), 6.99–6.96 (m, 1H), 5.05 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.54, 137.52, 137.16, 131.35, 130.40, 128.87, 128.53, 127.91, 127.73, 126.34, 125.25, 118.46, 114.16, 111.81, 104.36, 102.51, 70.76. HRMS (EI) ($[M]^+$) Calcd. for $C_{21}H_{16}NOSBr$: 409.0136, Found: 409.0139.



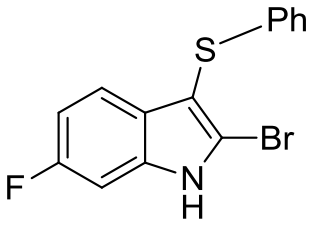
4c: Yellow solid, m.p. 123.1–123.6 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.39 (s, 1H), 7.22–7.19 (m, 2H), 7.12–7.10 (m, 3H), 6.95 (s, 1H), 6.81 (s, 1H), 5.95 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.80, 144.22, 137.45, 130.96, 128.83, 126.28, 125.22, 124.05, 114.71, 104.86, 100.93, 98.34, 91.98. HRMS (EI) ($[M]^+$) Calcd. for $C_{15}H_{10}NO_2BrS$: 346.9616, Found: 346.9613.



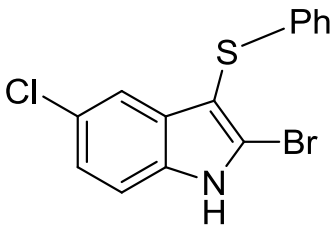
4d: White solid, m.p. 108.8–109.3 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.58 (s, 1H), 7.73 (s, 1H), 7.33 (d, J = 8.6 Hz, 1H), 7.24–7.20 (m, 3H), 7.14–7.11 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.97, 134.86, 131.30, 128.97, 126.44, 126.32, 125.52, 121.87, 119.73, 115.09, 112.30, 104.72. HRMS (EI) ($[M]^+$) Calcd. for $C_{14}H_9NSBr_2$: 380.8822, Found: 380.8824.



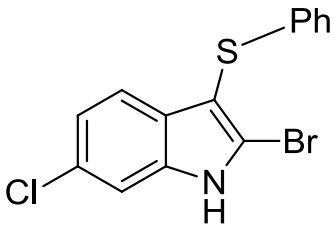
4e: White solid. m.p. 98.2–98.5 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.59 (s, 1H), 7.50–7.47 (m, 1H), 7.23–7.19 (m, 2H), 7.14–7.10 (m, 3H), 7.07–7.05 (m, 1H), 6.96–6.91 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.37 (d, *J* = 239.1 Hz), 137.16, 136.02 (d, *J* = 11.5 Hz), 128.89, 126.47, 126.01 (d, *J* = 1.1 Hz), 125.42, 120.32 (d, *J* = 9.9 Hz), 117.89 (d, *J* = 2.8 Hz), 110.23 (d, *J* = 24.3 Hz), 105.17, 97.60 (d, *J* = 26.7 Hz). HRMS (EI) ($[M]^+$) Calcd. for $C_{14}H_9NSFBr$: 320.9623, Found: 320.9619.



4f: Yellow solid, m.p. 132.8–133.0 °C.

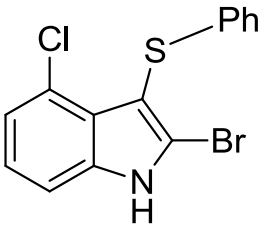
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (s, 1H), 7.57 (s, 1H), 7.28–7.19 (m, 4H), 7.14–7.11 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.04, 134.61, 130.74, 128.95, 127.49, 126.40, 125.48, 123.68, 119.88, 118.79, 111.99, 104.64. HRMS (EI) ($[M]^+$) Calcd. for $C_{14}H_9NSClBr$: 336.9328, Found: 336.9323.



4g: Yellow oil.

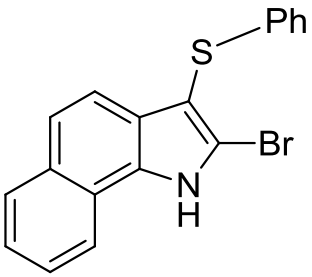
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.64 (s, 1H), 7.47 (d, *J* = 8.4 Hz, 1H), 7.36 (s, 1H), 7.23–7.10 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.03, 136.44, 129.32, 128.89, 128.14, 126.48, 125.45, 122.27,

120.27, 118.80, 110.85, 105.39. HRMS (EI) ([M]$^+$) Calcd. for $C_{14}H_9NSClBr$: 336.9328, Found: 336.9331.



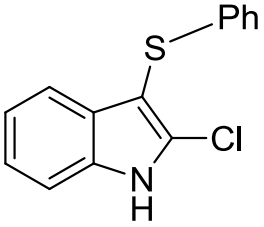
4h: White solid, m.p. 125.3–125.5 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.86 (s, 1H), 7.27–7.20 (m, 3H), 7.14–7.09 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.12, 137.58, 128.79, 126.22, 126.21, 125.65, 125.12, 123.70, 122.88, 121.34, 109.73, 104.49. HRMS (EI) ([M]$^+$) Calcd. for $C_{14}H_9NSClBr$: 336.9328, Found: 336.9327.



4i: White solid, m.p. 128.7–129.0 °C.

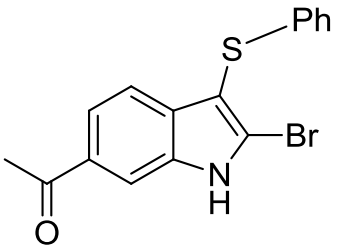
^{1}H NMR (400 MHz, $CDCl_3$): δ 9.26 (s, 1H), 7.99–7.93 (m, 2H), 7.67–7.65 (m, 1H), 7.60–7.56 (m, 2H), 7.51–7.48 (m, 1H), 7.22–7.14 (m, 4H), 7.12–7.08 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.65, 131.30, 130.75, 129.09, 128.83, 126.35, 126.16, 125.23, 124.78, 122.36, 120.90, 119.09, 118.73, 114.94, 106.67. HRMS (EI) ([M]$^+$) Calcd. for $C_{18}H_{12}NSBr$: 352.9874, Found: 352.9875.



4j: Yellow oil.

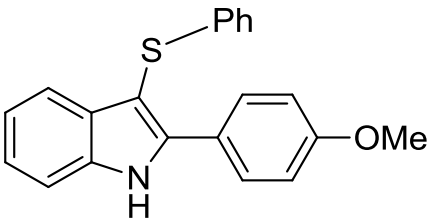
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (s, 1H), 7.58 (d, J = 7.8 Hz, 1H), 7.36 (d, J = 8.0 Hz, 1H), 7.29–7.26 (m, 1H), 7.22–7.18 (m, 3H), 7.15–7.09 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.51,

134.54, 129.91, 129.41, 128.84, 126.37, 125.24, 123.35, 121.55, 119.32, 110.83, 101.02. HRMS (EI) ($[M]^+$) Calcd. for $C_{14}H_{10}NSCl$: 259.0222, Found: 259.0223.



4k: White solid, m.p. 175.7–175.9 °C.

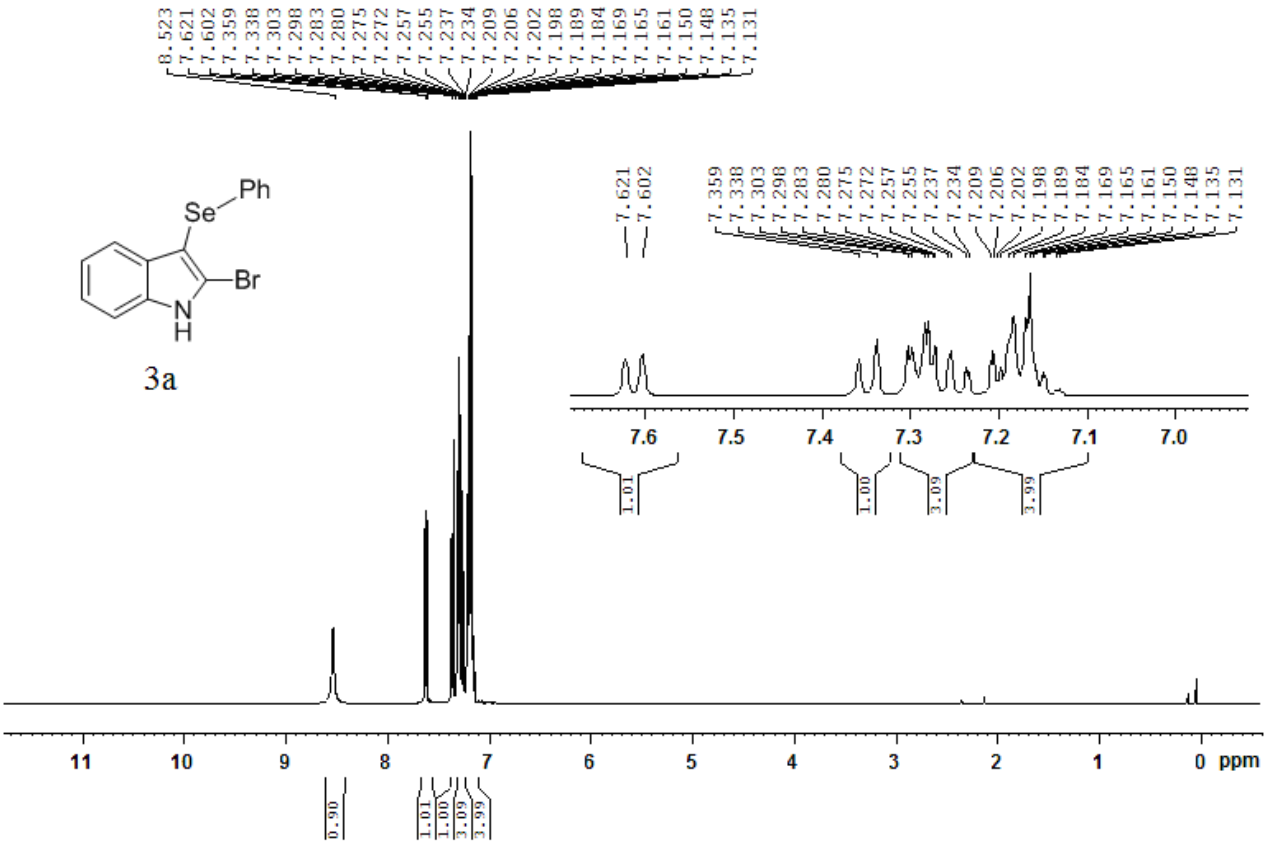
^{1}H NMR (400 MHz, $CDCl_3$): δ 9.15 (s, 1H), 8.10 (s, 1H), 7.78 (d, *J* = 8.3 Hz, 1H), 7.62 (d, *J* = 8.4 Hz, 1H), 7.22–7.18 (m, 2H), 7.13–7.11 (m, 3H), 2.68 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.06, 136.91, 135.93, 133.40, 132.53, 128.90, 126.60, 125.53, 122.41, 121.91, 119.10, 111.47, 105.89, 26.86. HRMS (EI) ($[M]^+$) Calcd. for $C_{16}H_{12}NOSBr$: 344.9823, Found: 344.9828.

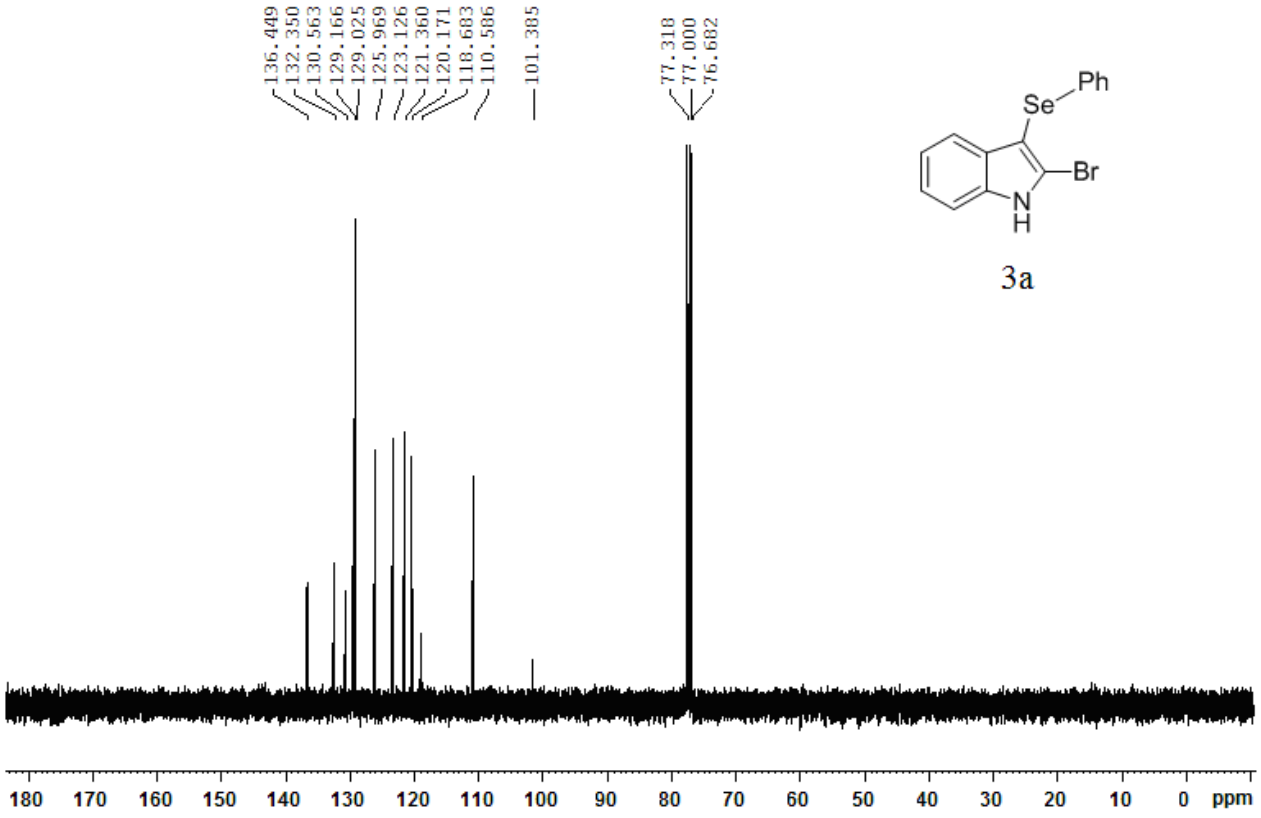


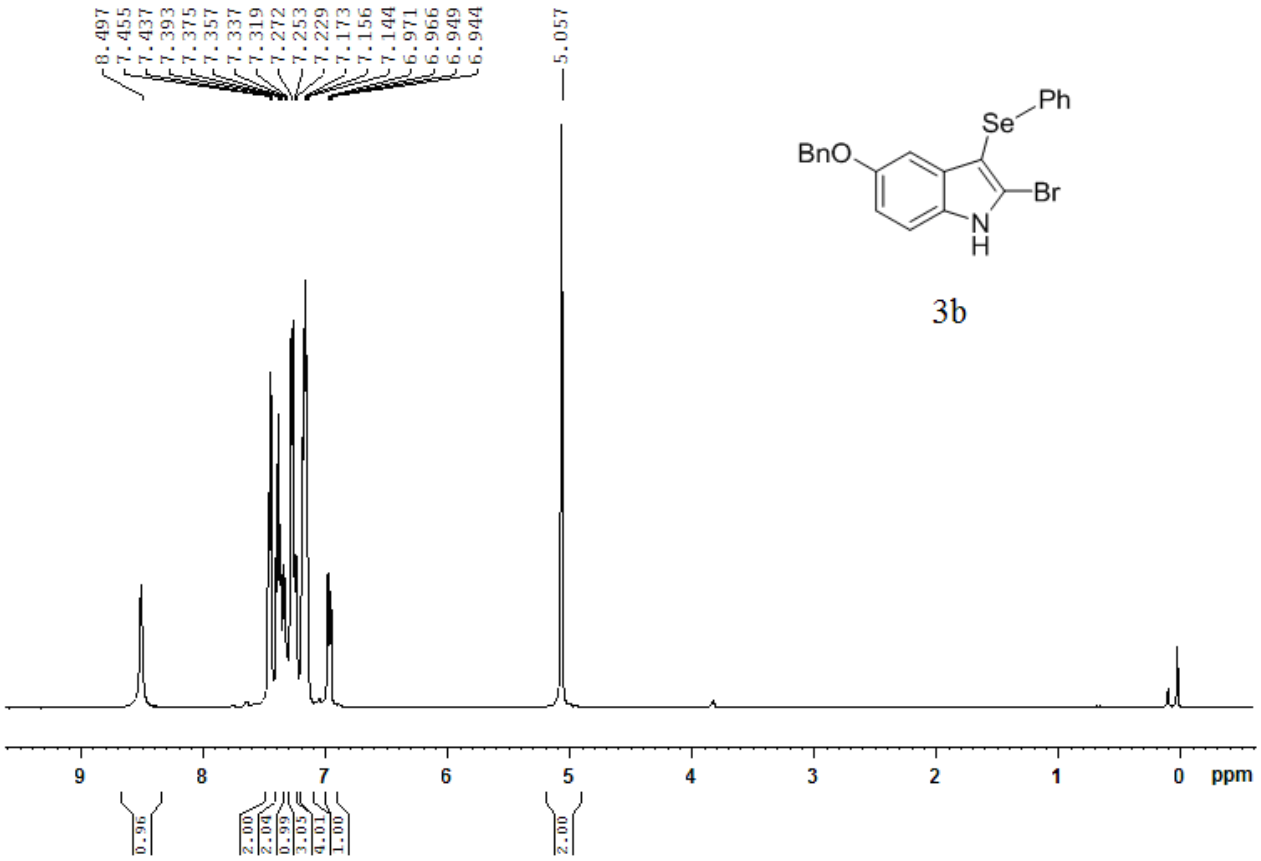
5a: Colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.51 (s, 1H), 7.71 (d, *J* = 8.7 Hz, 2H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.29–7.25 (m, 1H), 7.20–7.16 (m, 3H), 7.13–7.11 (m, 2H), 7.08–7.05 (m, 1H), 6.98 (d, *J* = 8.7 Hz, 2H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.98, 149.65, 142.20, 139.47, 135.76, 131.32, 129.45, 128.82, 125.51, 124.58, 123.96, 123.04, 121.06, 119.72, 114.26, 111.07, 55.35. HRMS (EI) ($[M]^+$) Calcd. for $C_{21}H_{17}NOS$: 331.1031, Found: 331.1029.

7. ^{1}H, ^{13}C NMR and HRMS spectra



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7.393
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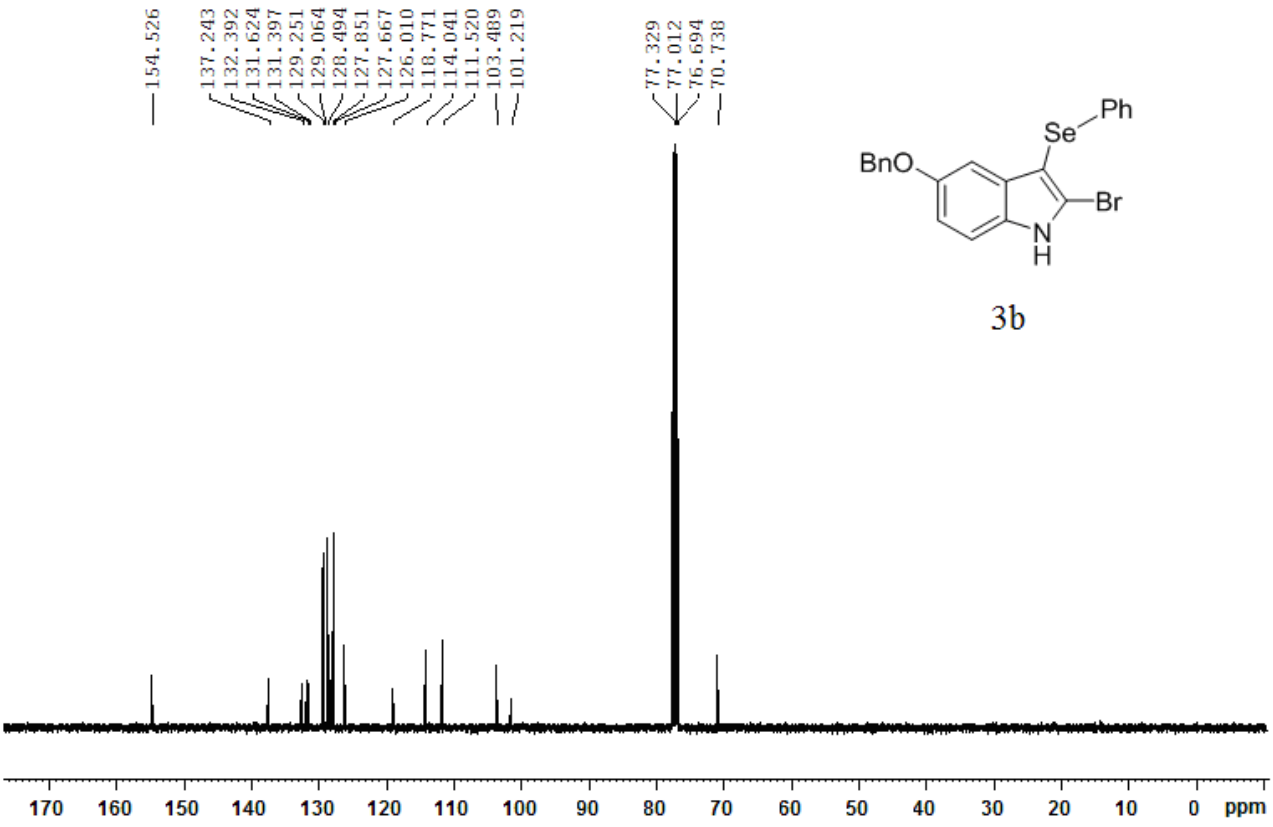
7.357
7.337
7.319
7.272
7.253
7.229
7.173
7.156
7.144
6.971
6.966
6.949
6.944
5.057
Ph
Se
BnO
Br
N
H
9
8
7
6
5
4
3
2
1
0
ppm
0.96
2.00
2.04
0.99
3.05
4.01
1.00
2.00

3b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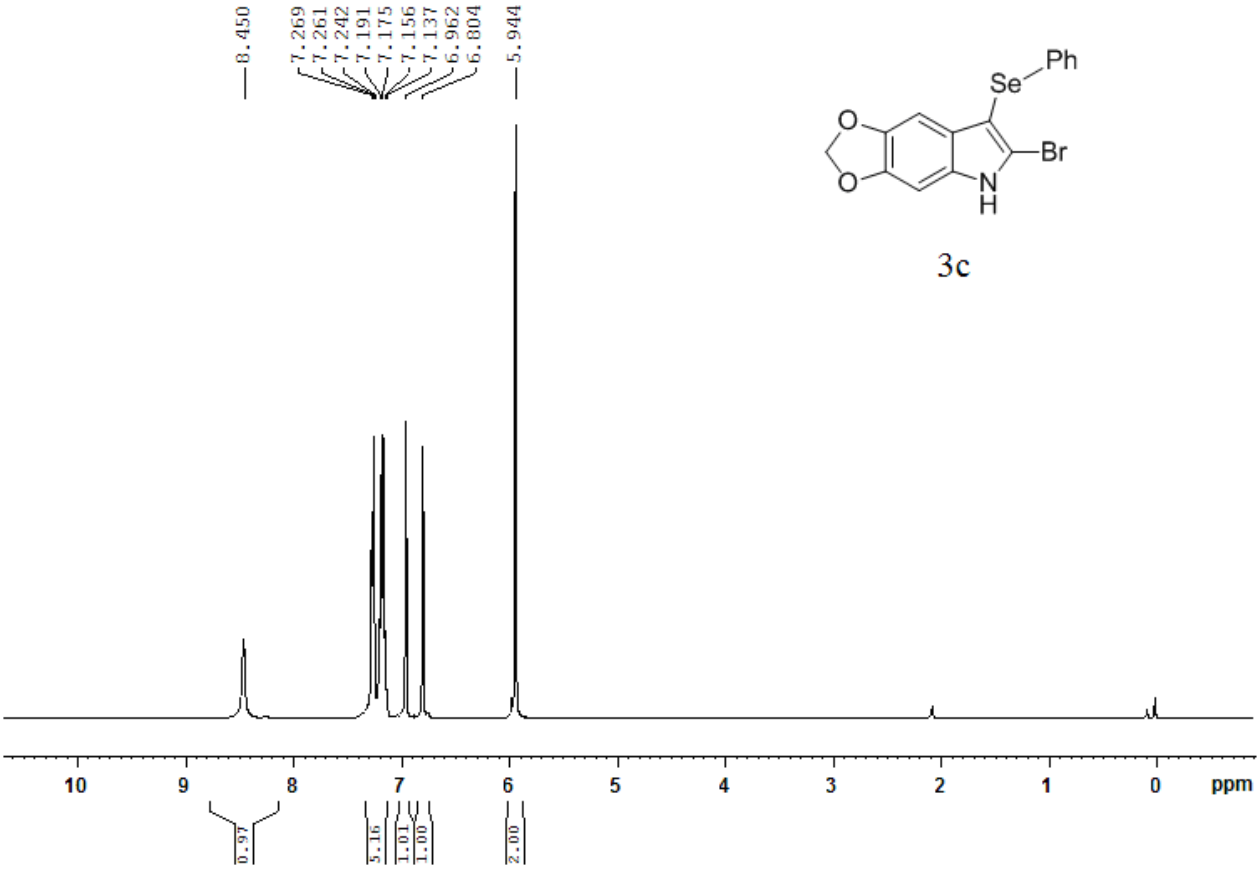
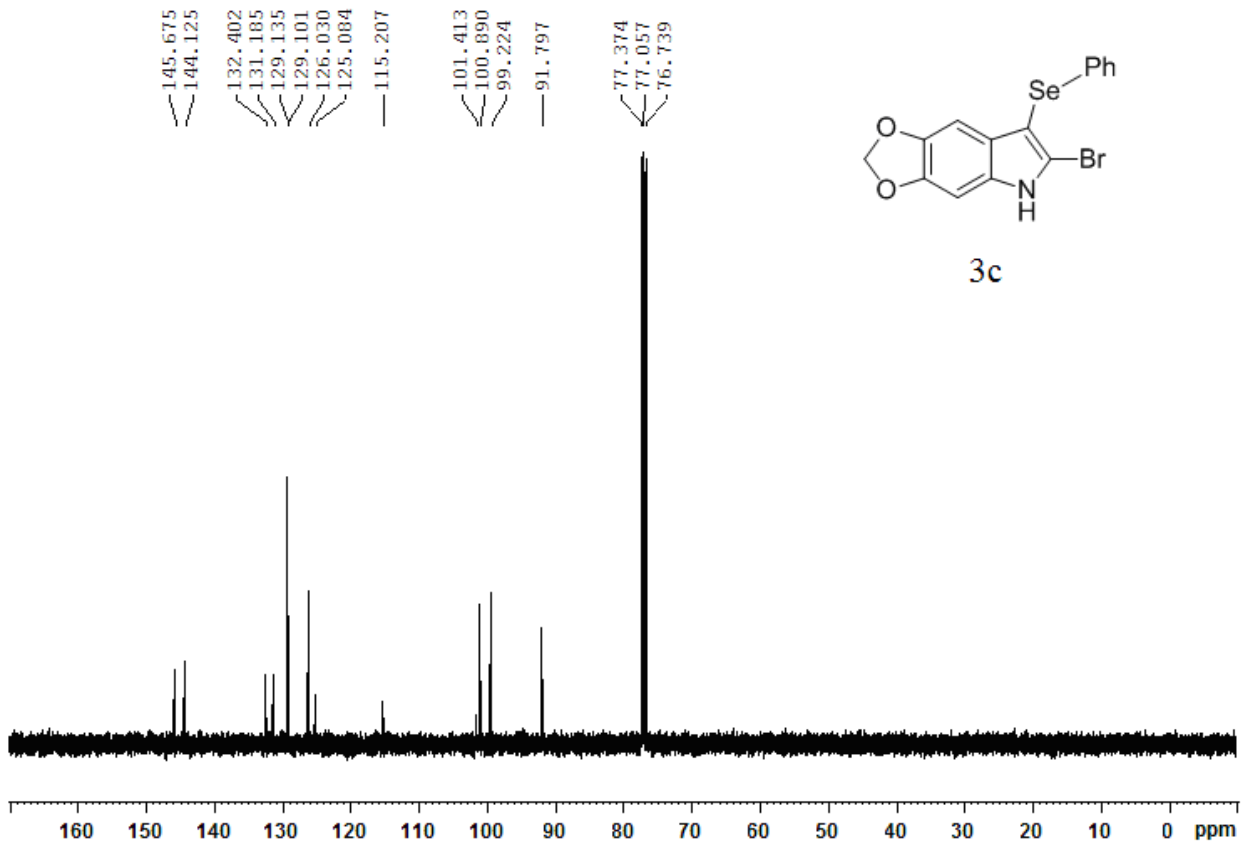
132.392
131.624
131.397
129.251
129.064
128.494
127.851
127.667
126.010
118.771
114.041
111.520
103.489
101.219
77.329
77.012
76.694
70.738
Ph
Se
BnO
Br
N
H
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

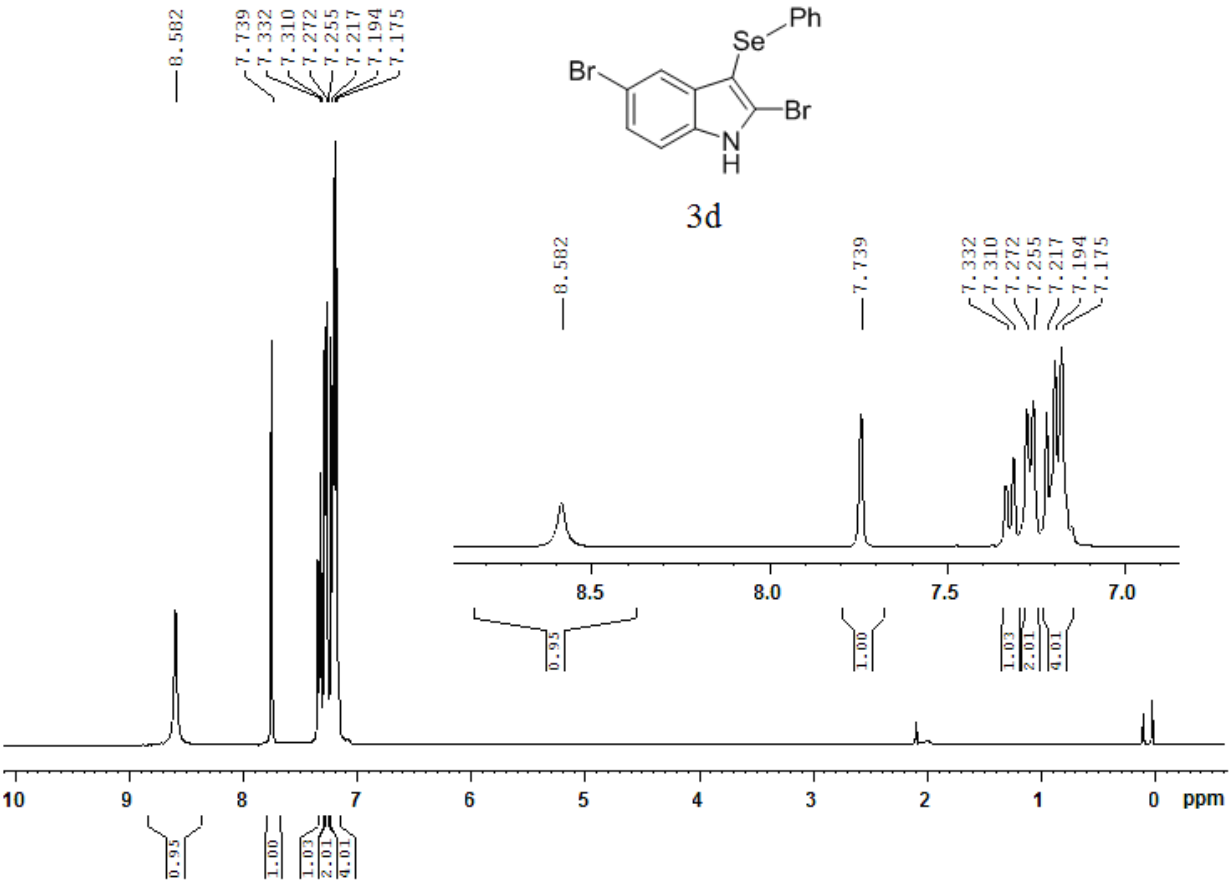
3b



3c



3c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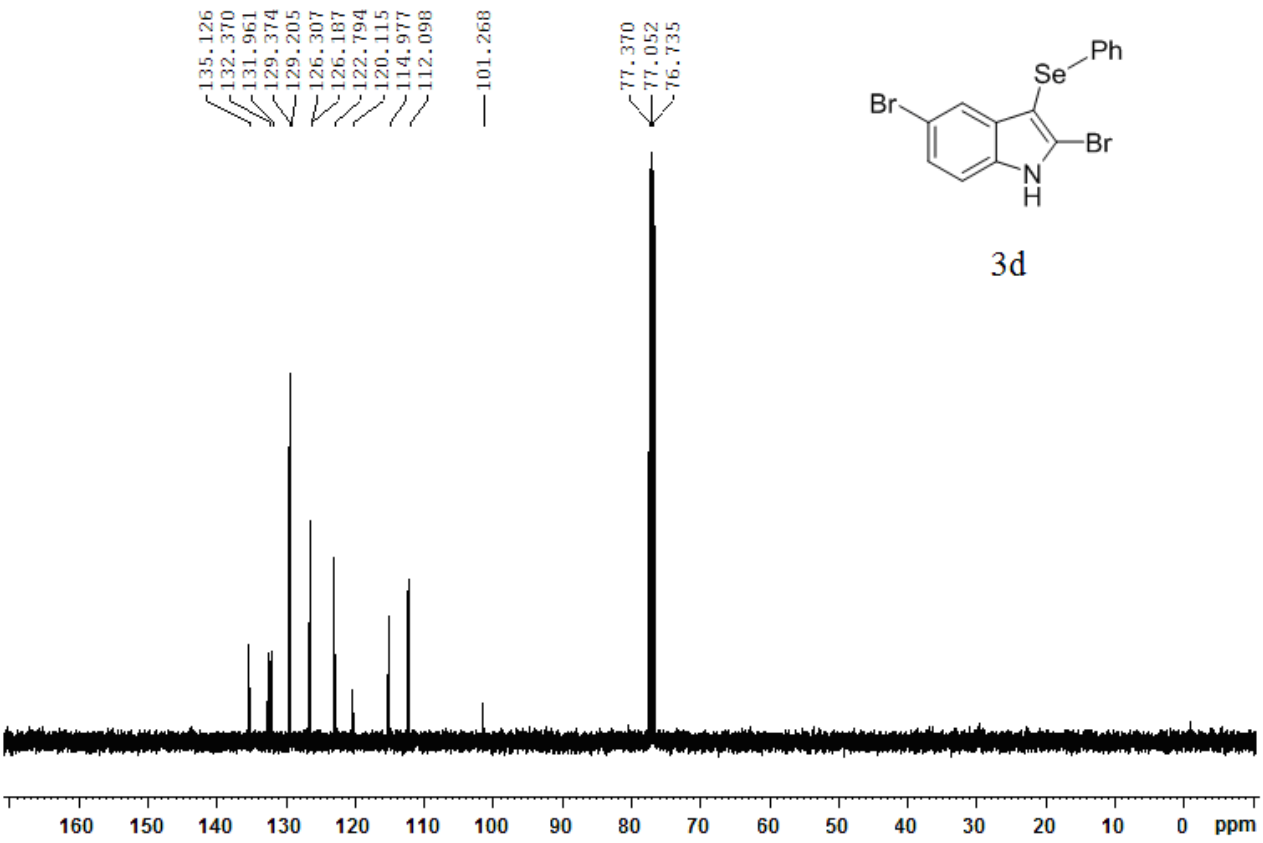
7.332
7.310
7.272
7.255
7.217
7.194
7.175
Br
Se
Ph
Br
N
H
3d
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
1.00
1.03
2.01
4.01


135.126
132.370
131.961
129.374
129.205
126.307
126.187
122.794
120.115
114.977
112.098
101.268
77.370
77.052
76.735
Br
Se
Ph
Br
N
H
3d
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150
140
130
120
110
100
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80
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60
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20
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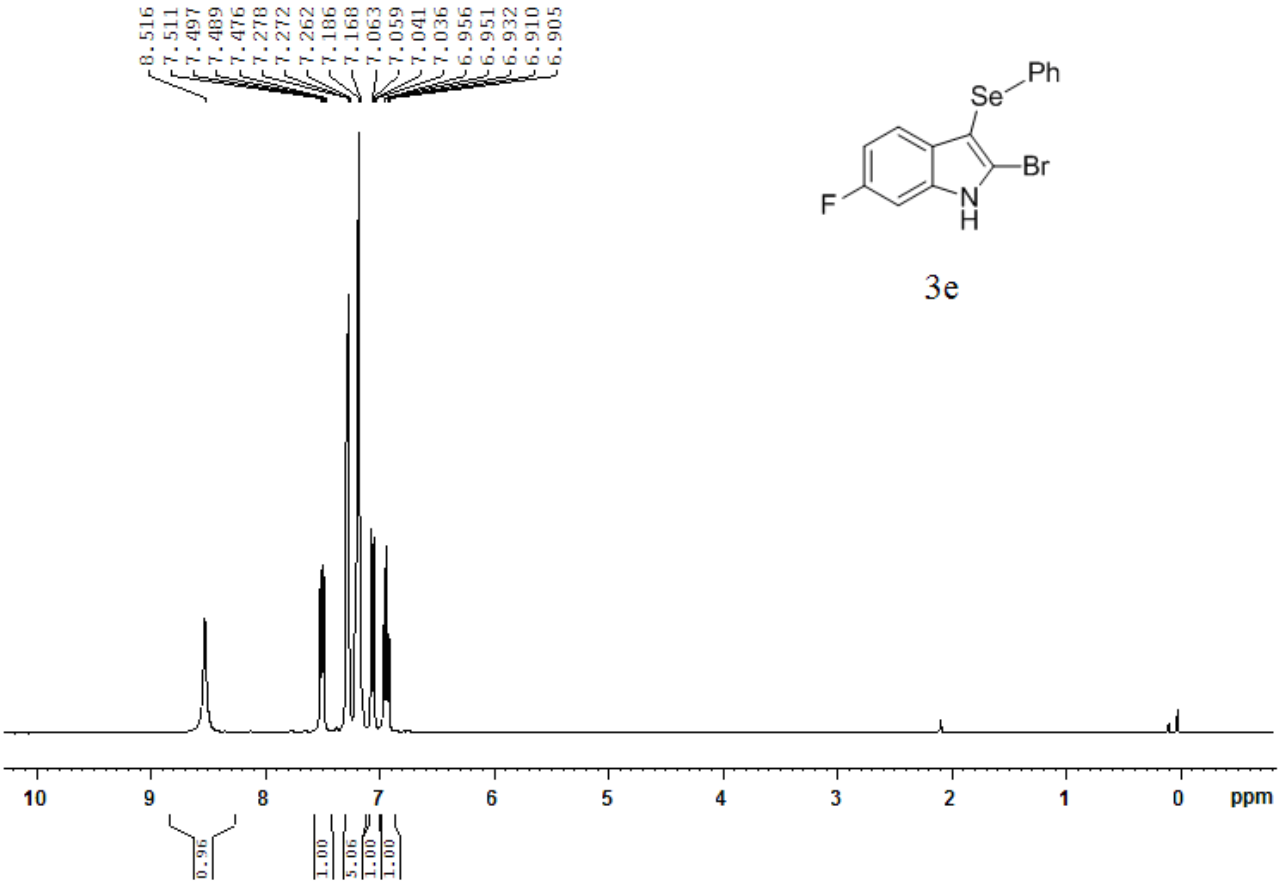
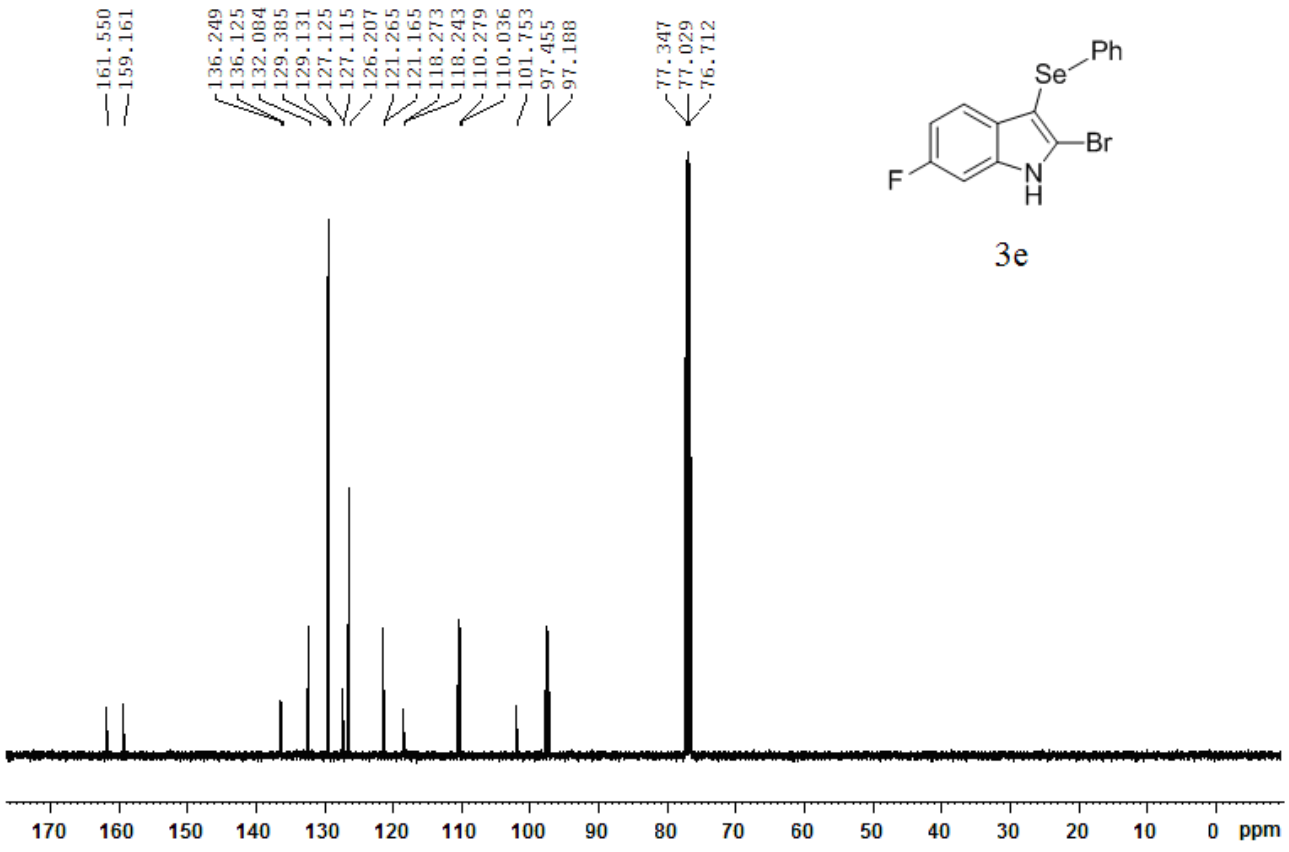
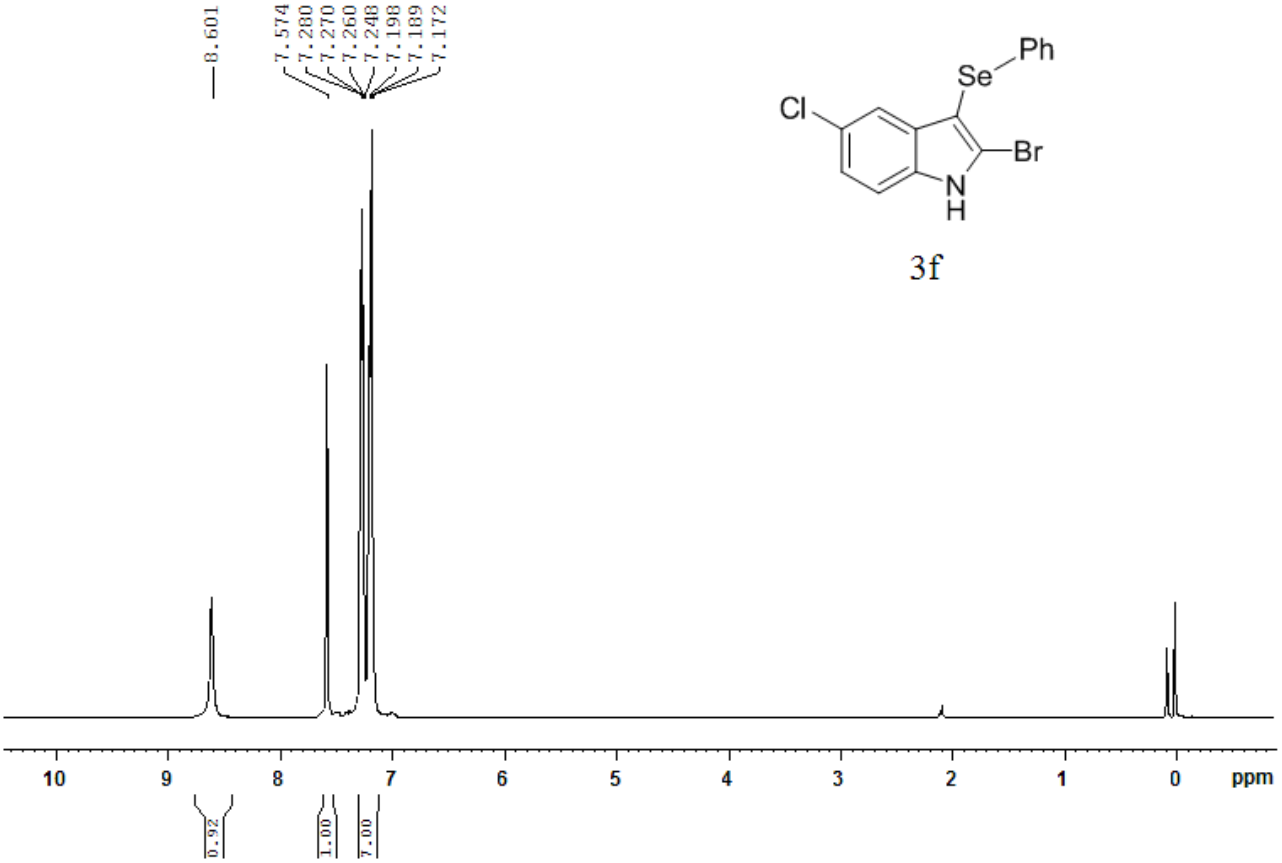
0
ppm

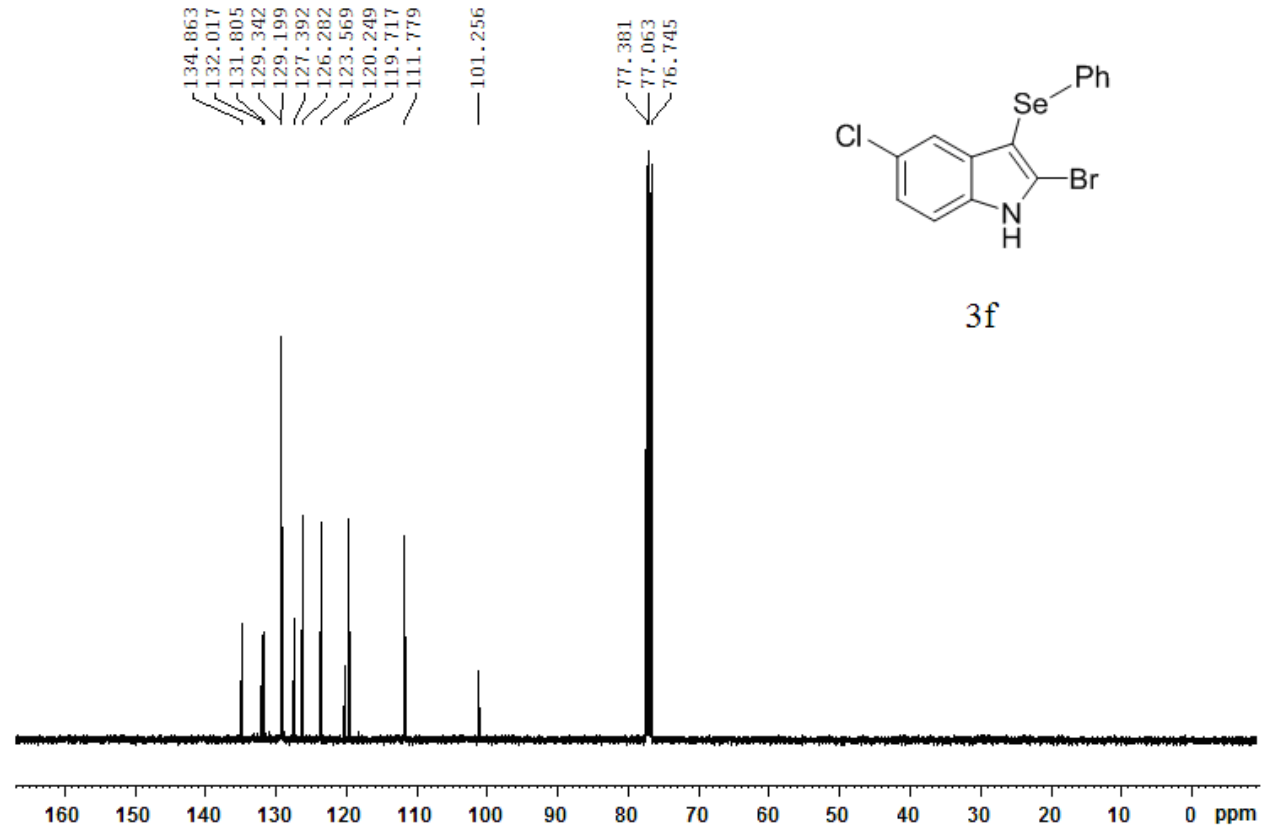


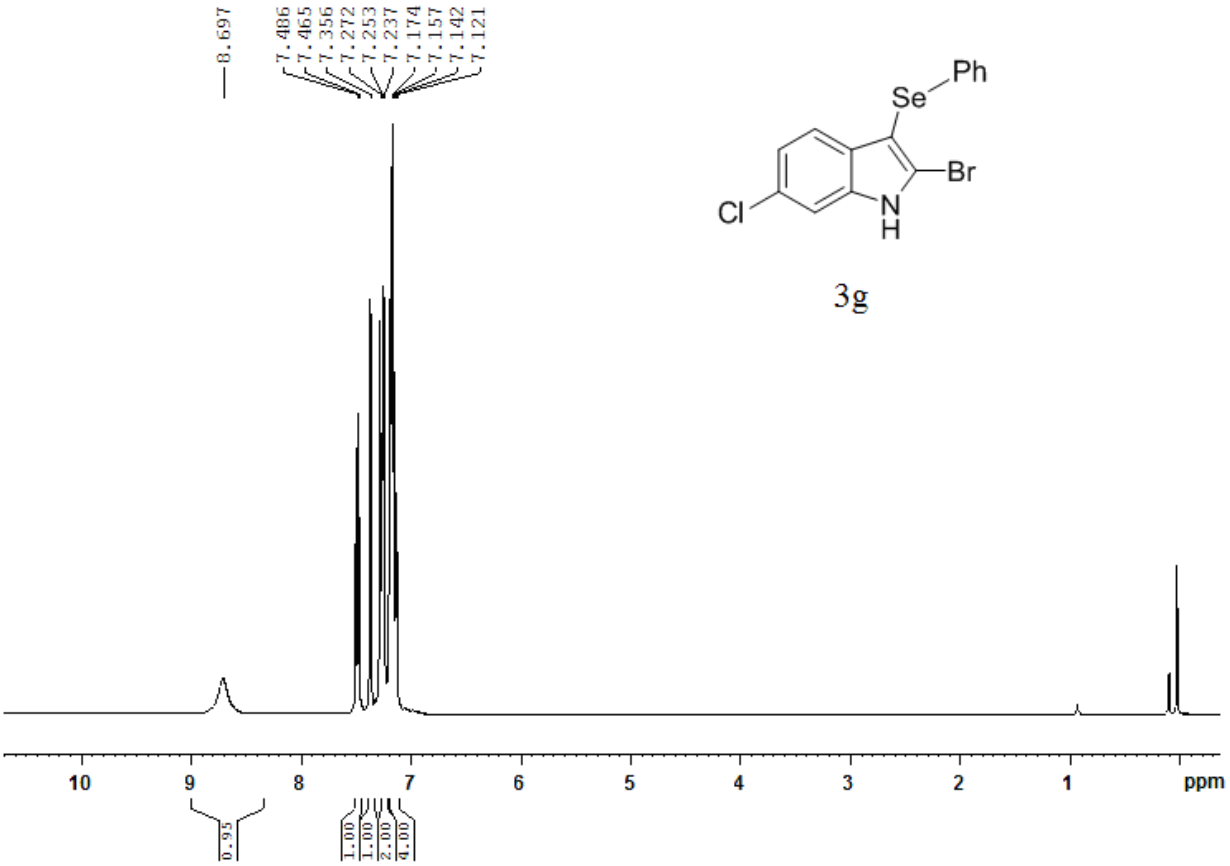
3e



3e



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Ph
Se
Br
Cl
N
H
10
9
8
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6
5
4
3
2
1
ppm
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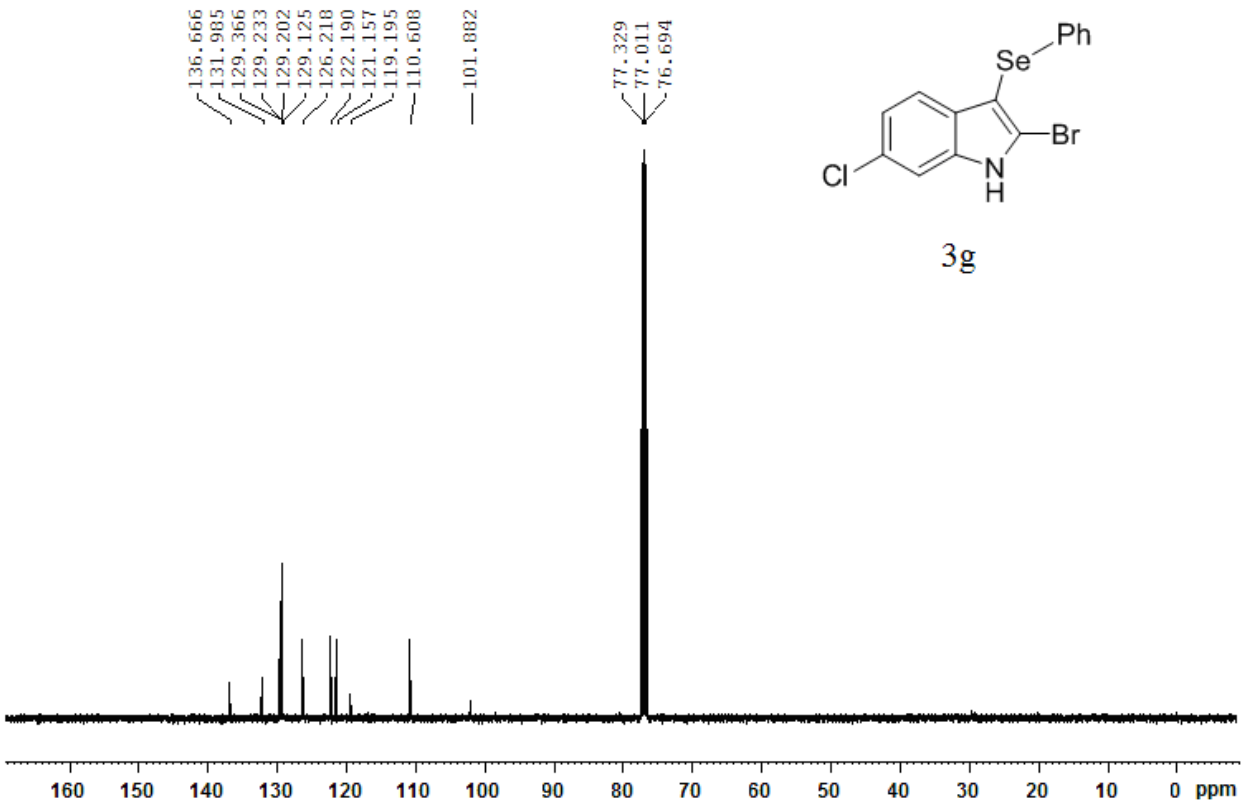
1.00
1.00
2.00
4.00

3g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122.190
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101.882
77.329
77.011
76.694
Ph
Se
Br
Cl
N
H
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150
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130
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100
90
80
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50
40
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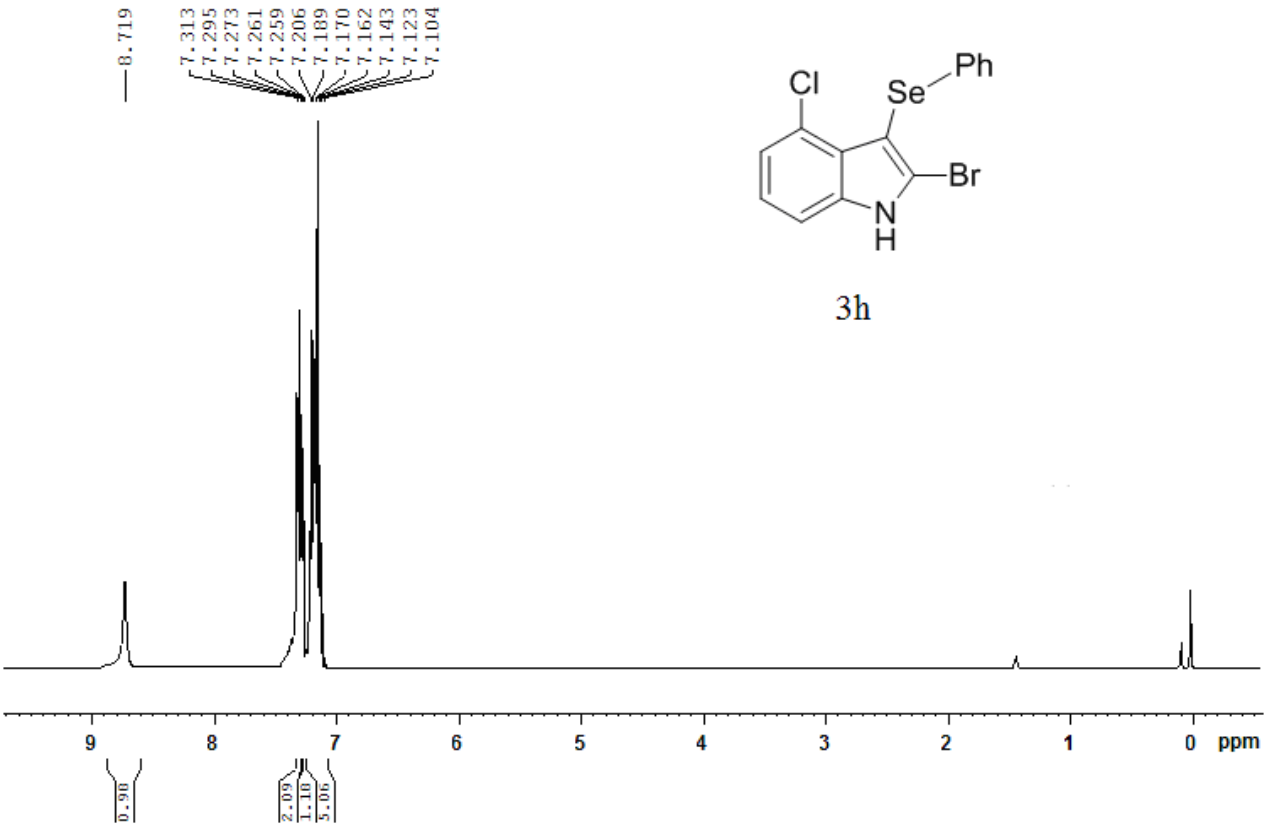
20
10
0
ppm

3g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7.259
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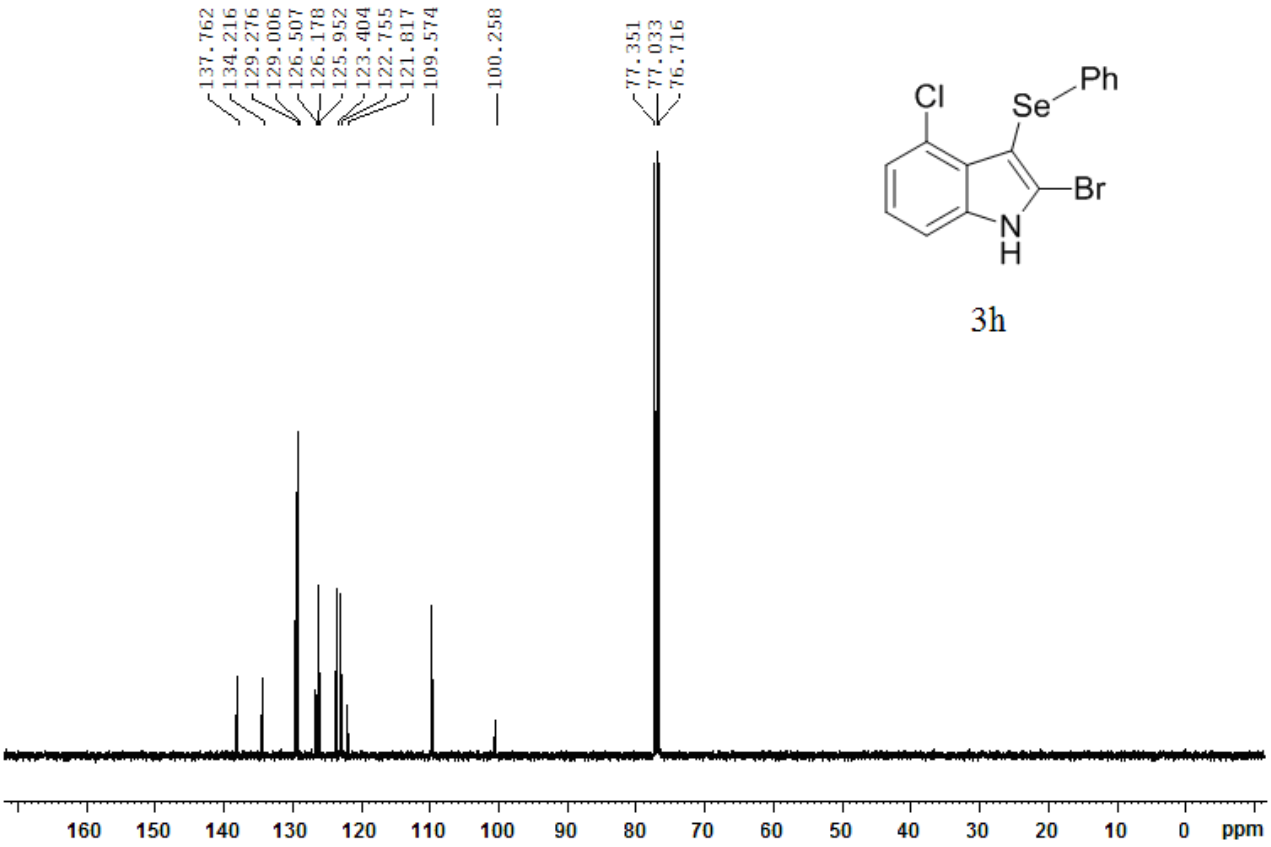
7.189
7.170
7.162
7.143
7.123
7.104
Cl
Se
Ph
Br
N
H
9
8
7
6
5
4
3
2
1
0
ppm
0.90
2.09
1.10
5.06

3h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121.817
109.574
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77.033
76.716
Cl
Se
Ph
Br
N
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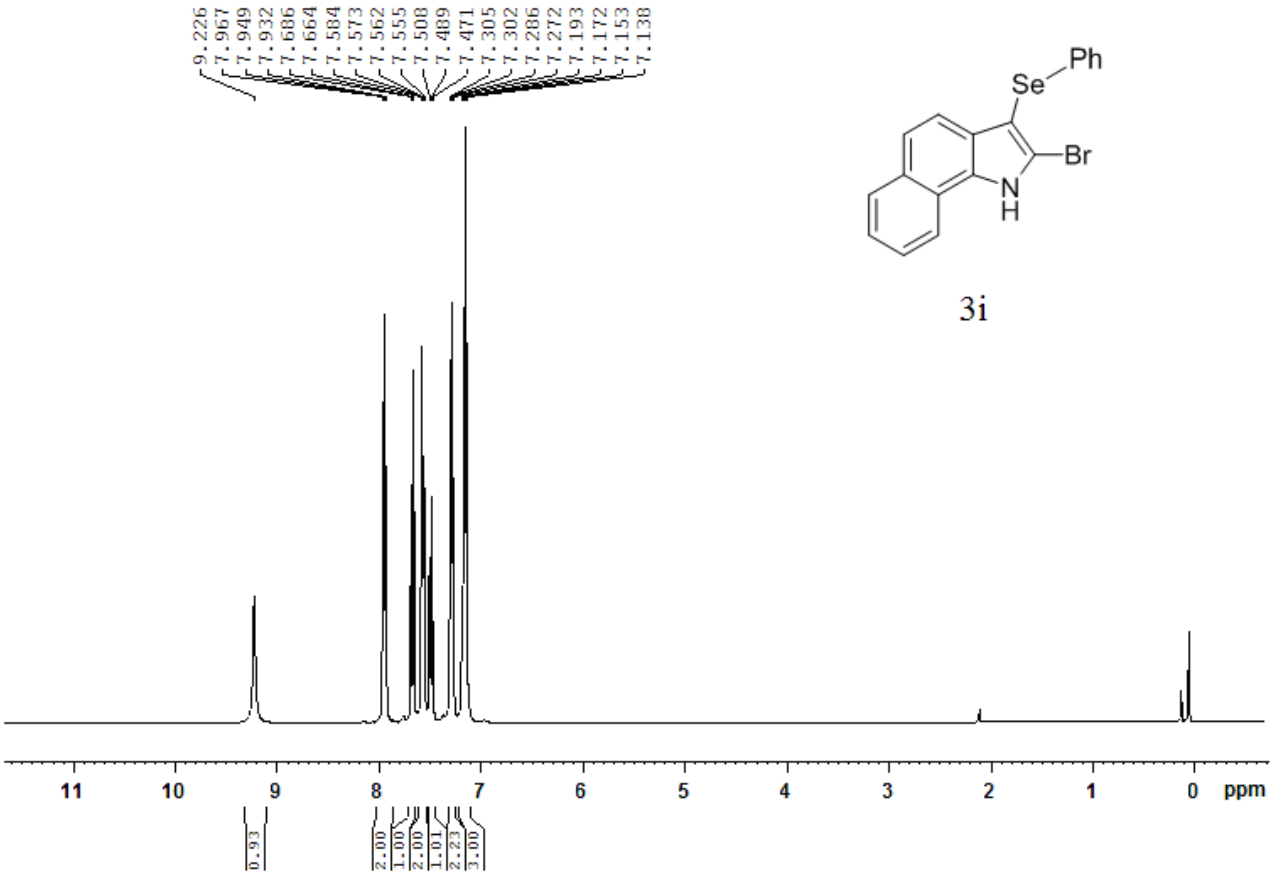
50
40
30
20
10
0
ppm

3h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7.664
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7.562
7.555
7.508
7.489
7.471
7.305
7.302
7.286
7.272
7.193
7.172
7.153
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Se
Br
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10
9
8
7
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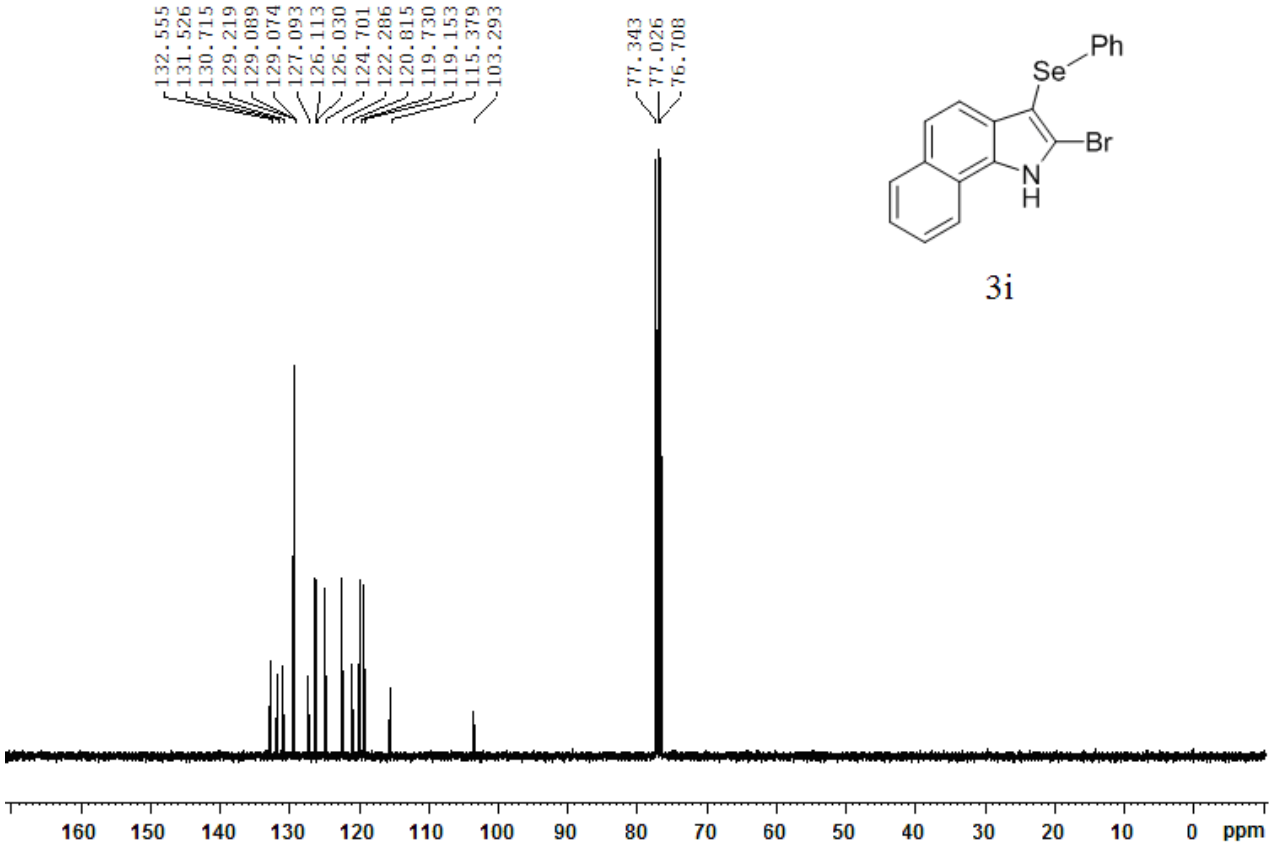
2.23
3.00

3i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129.074
127.093
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124.701
122.286
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119.730
119.153
115.379
103.293
77.343
77.026
76.708
Ph
Se
Br
N
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120
110
100
90
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50
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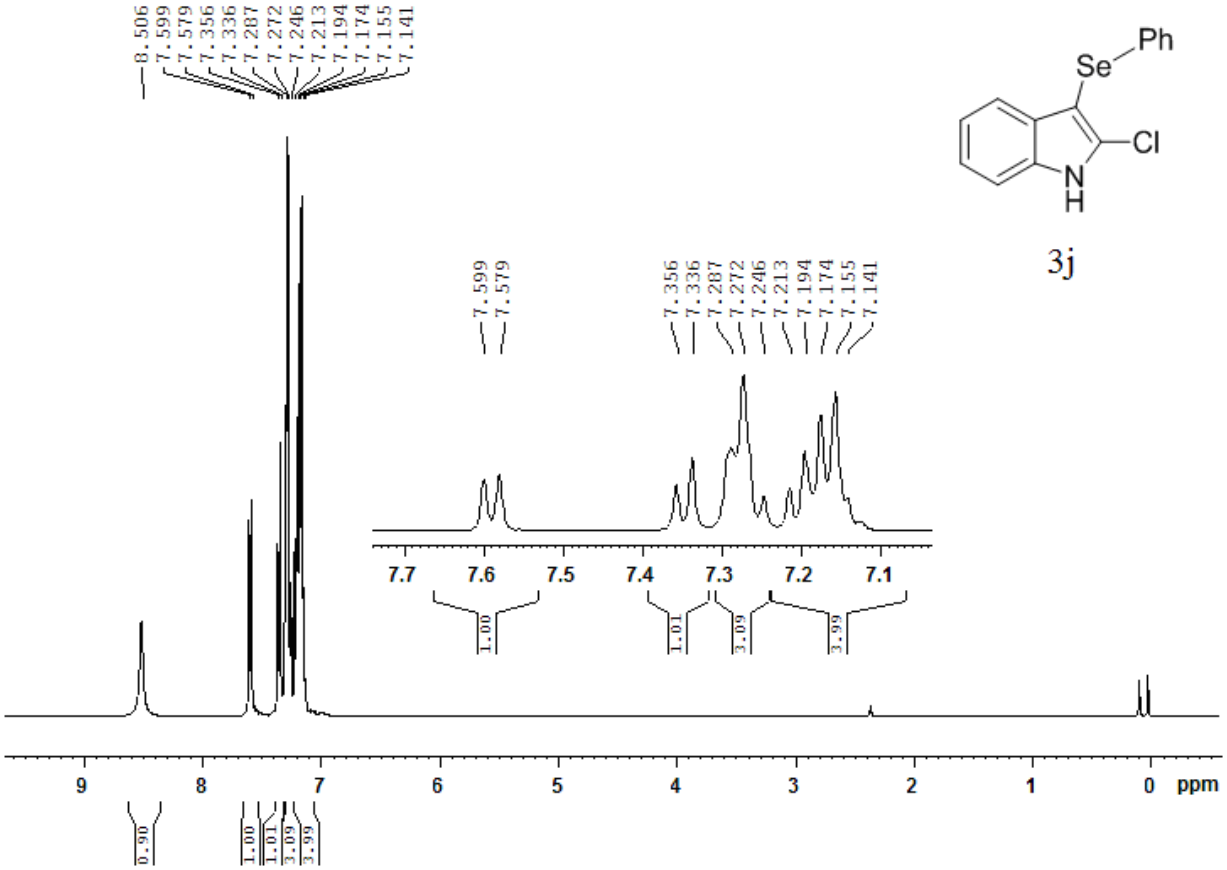
20
10
0
ppm

3i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7.336
7.287
7.272
7.246
7.213
7.194
7.174
7.155
7.141
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Se
Cl
N
H
3j
9
8
7
6
5
4
3
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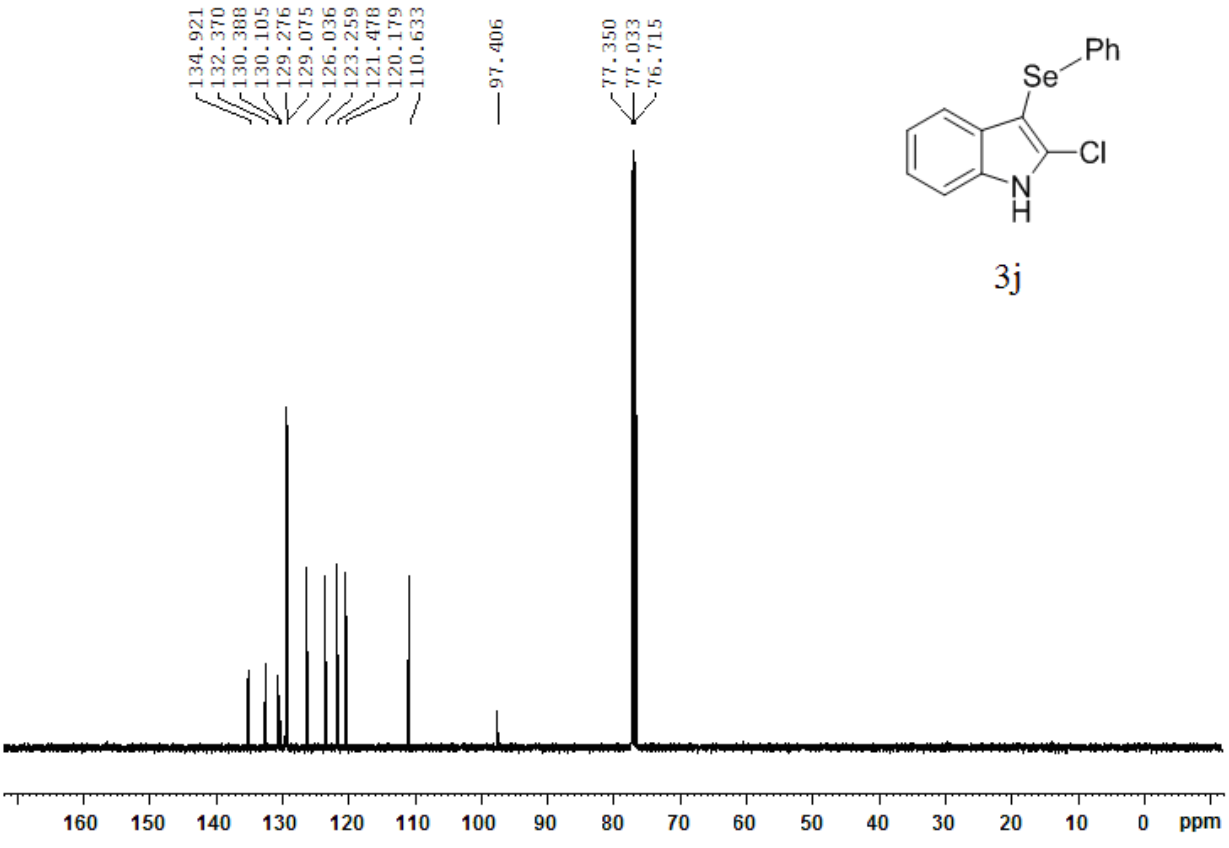
1
0
ppm


134.921
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130.105
129.276
129.075
126.036
123.259
121.478
120.179
110.633
97.406
77.350
77.033
76.715
Ph
Se
Cl
N
H
3j
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140
130
120
110
100
90
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60
50
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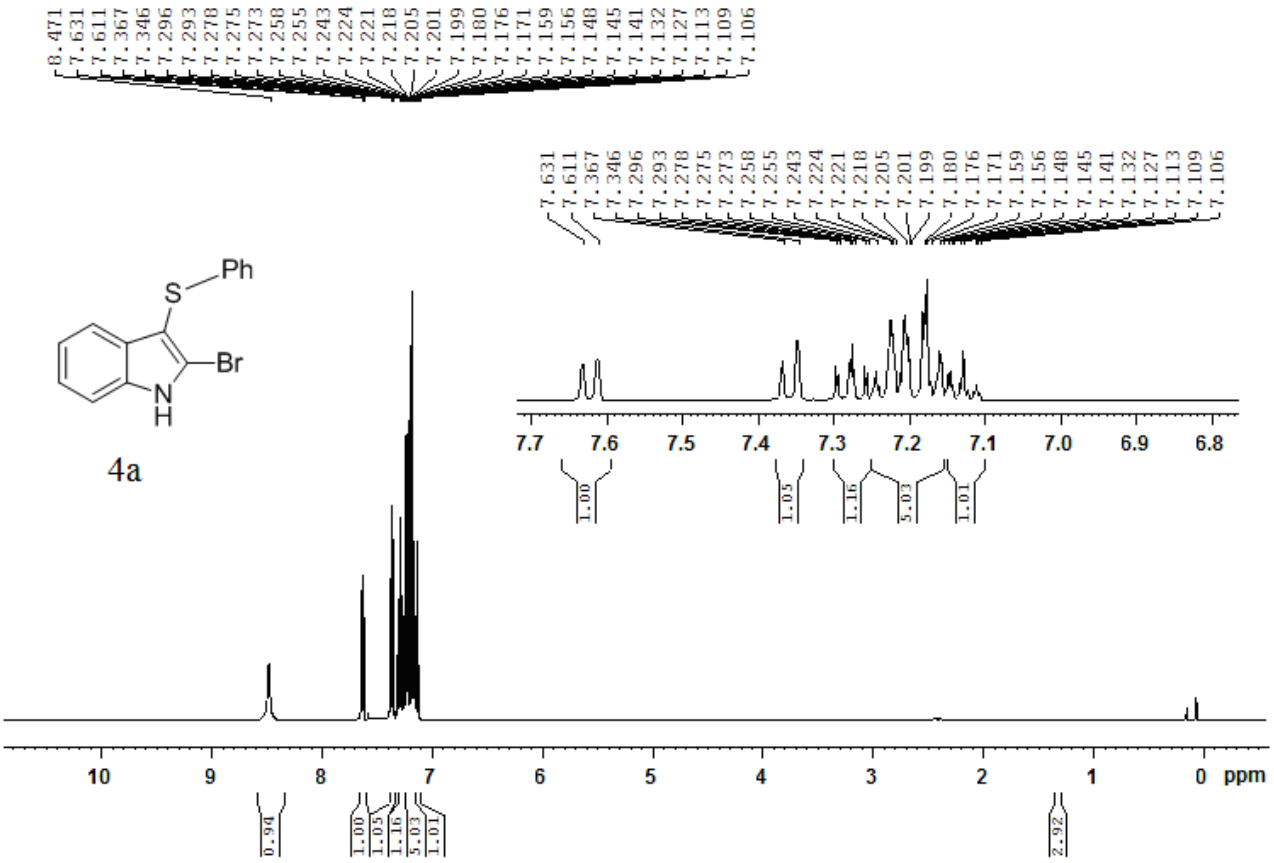
30
20
10
0
ppm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S
Br
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8
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ppm
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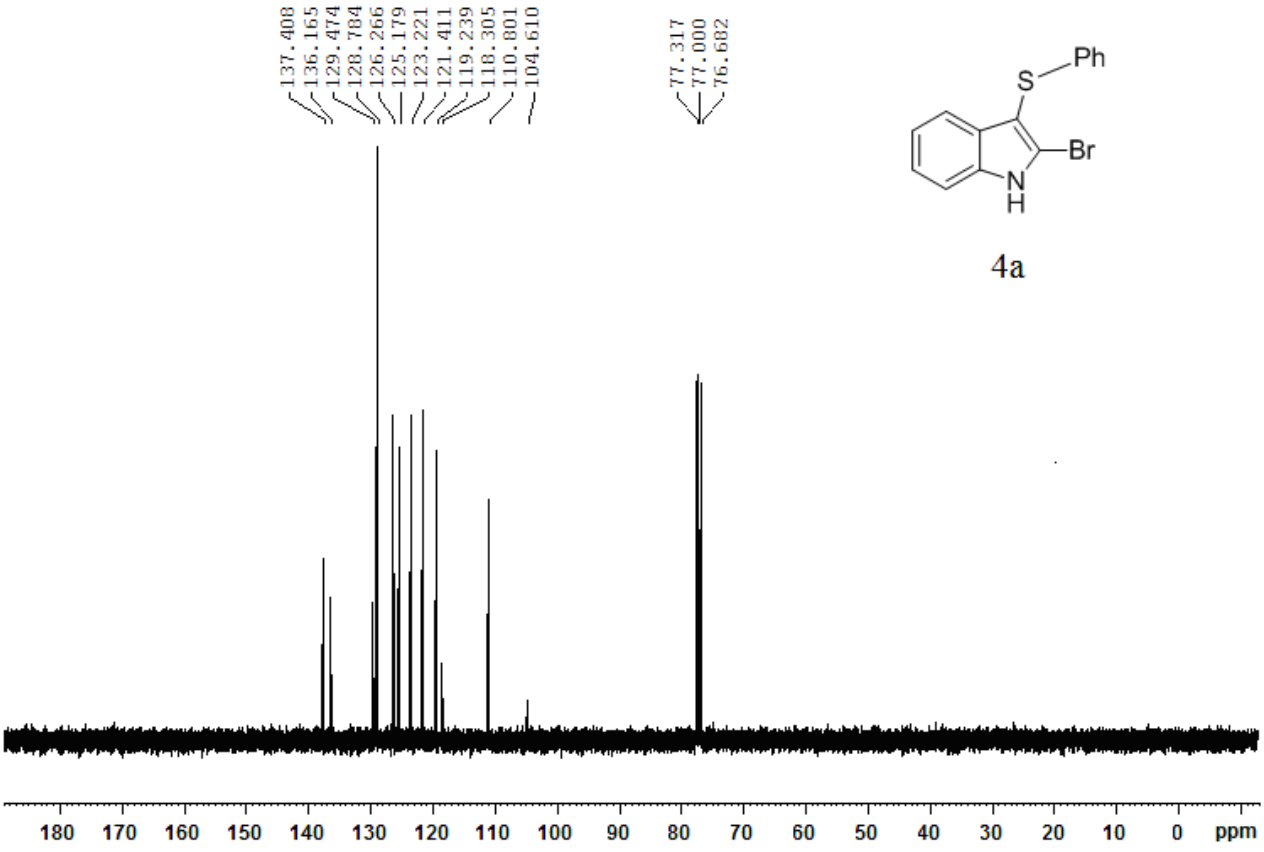
2.92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104.610
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77.000
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Ph
S
Br
N
H
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140
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120
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60
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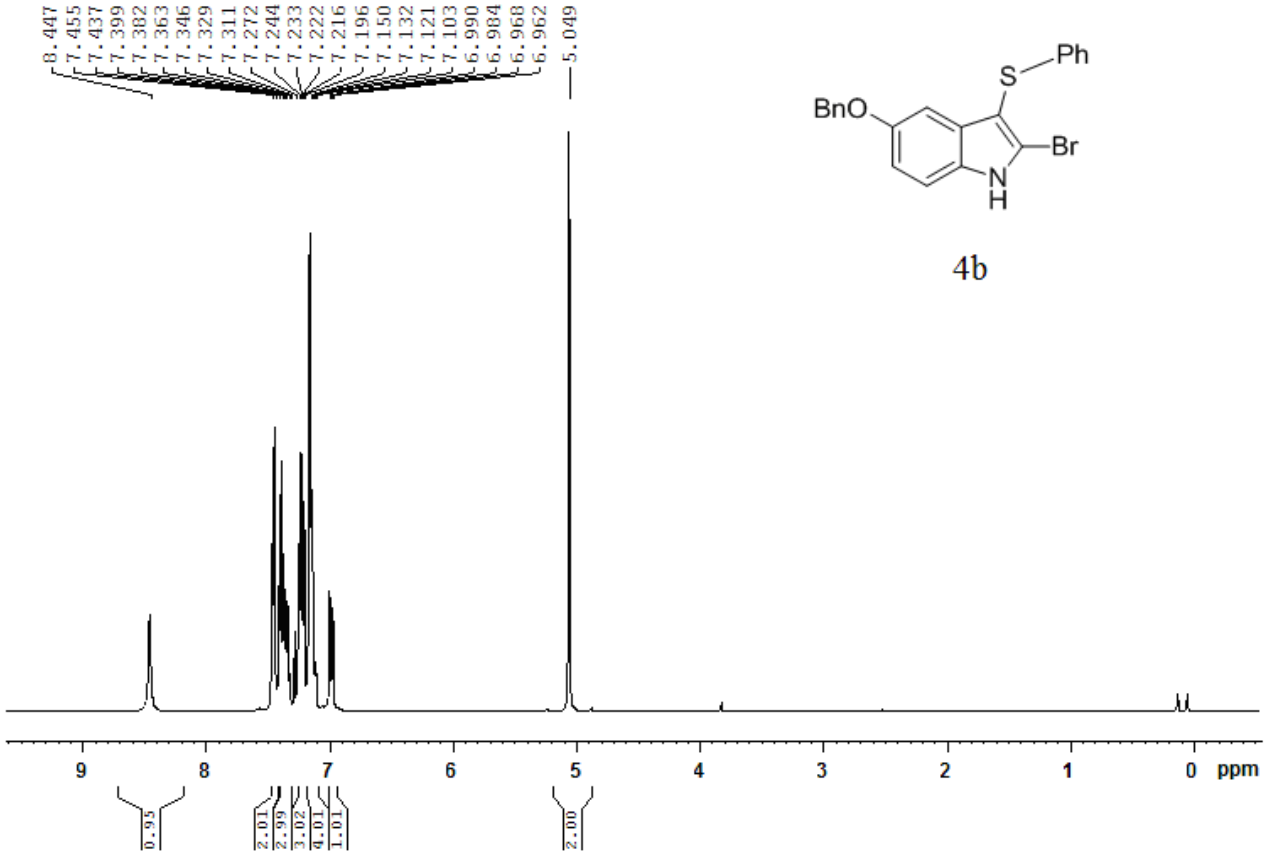
0
ppm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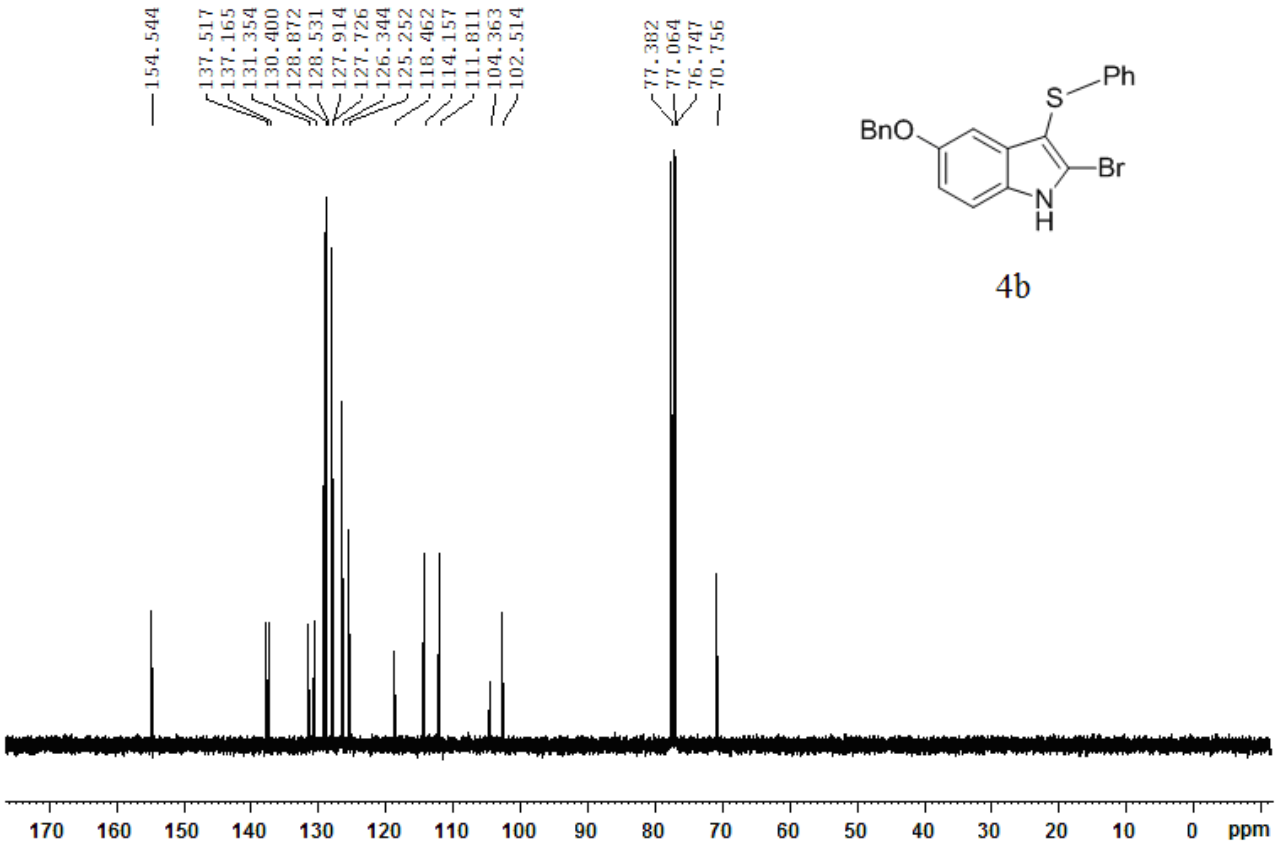
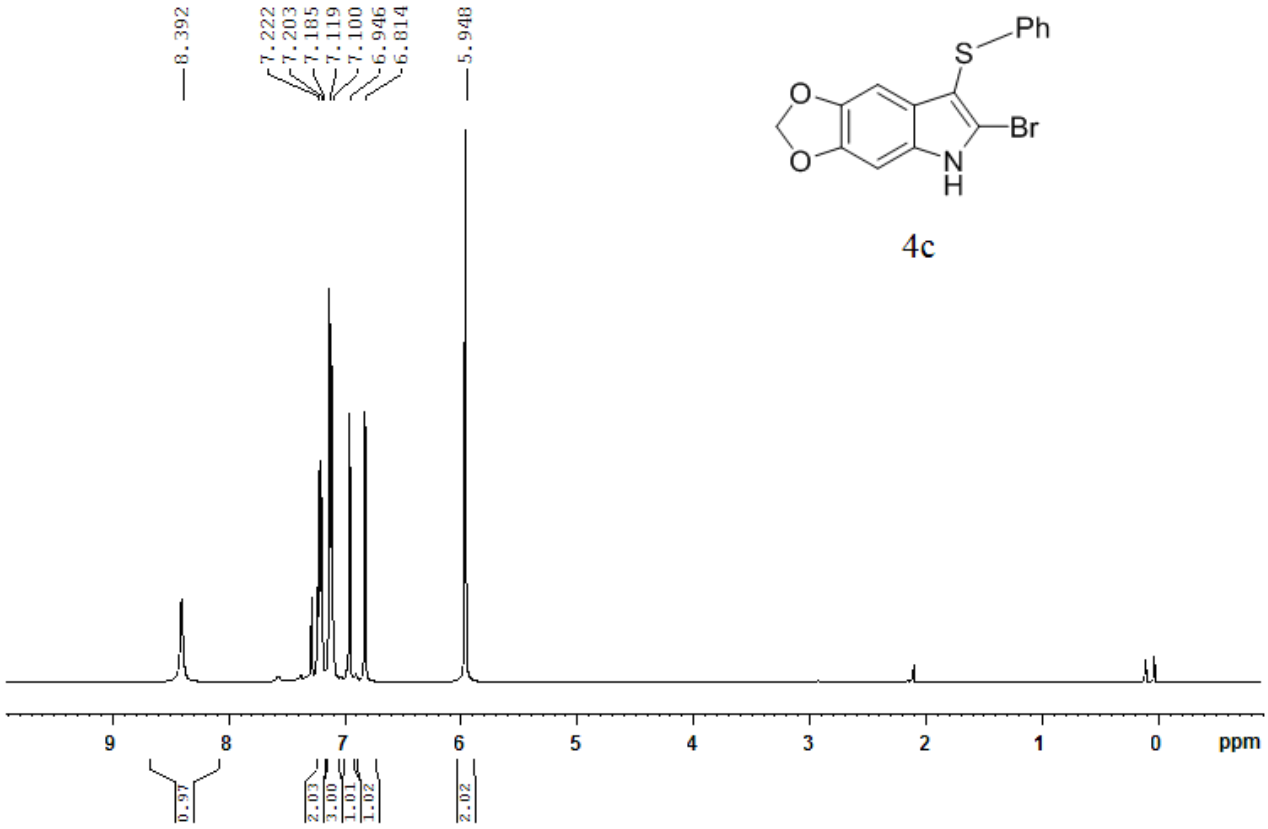
7.272
7.244
7.233
7.222
7.216
7.196
7.150
7.132
7.121
7.103
6.990
6.984
6.968
6.962
5.049
BnO
S
Ph
Br
N
H
9
8
7
6
5
4
3
2
1
0
ppm
0.95
2.01
2.99
3.02
4.01
1.01
2.00

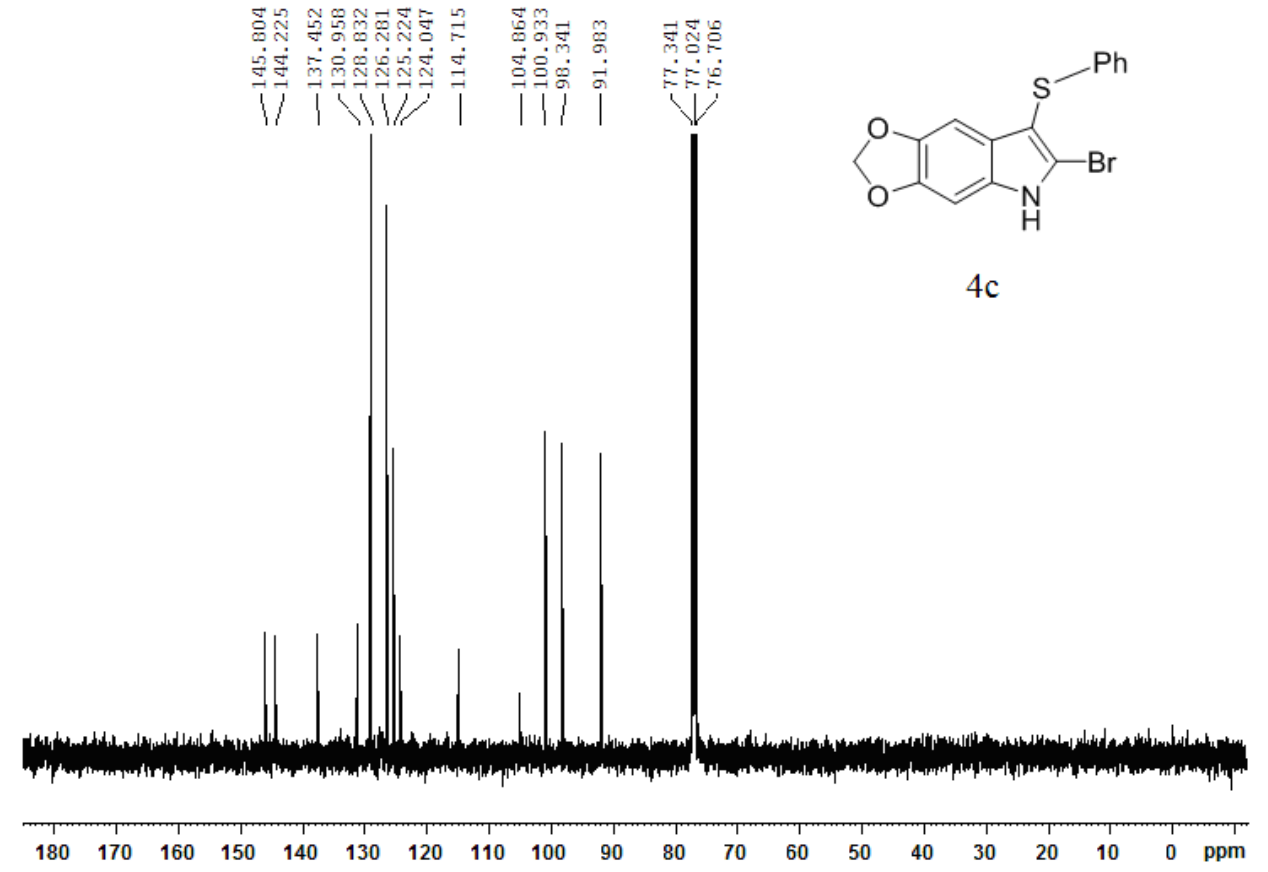
4b


154.544
137.517
137.165
131.354
130.400
128.872
128.531
127.914
127.726
126.344
125.252
118.462
114.157
111.811
104.363
102.514
77.382
77.064
76.747
70.756
BnO
S
Ph
Br
N
H
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

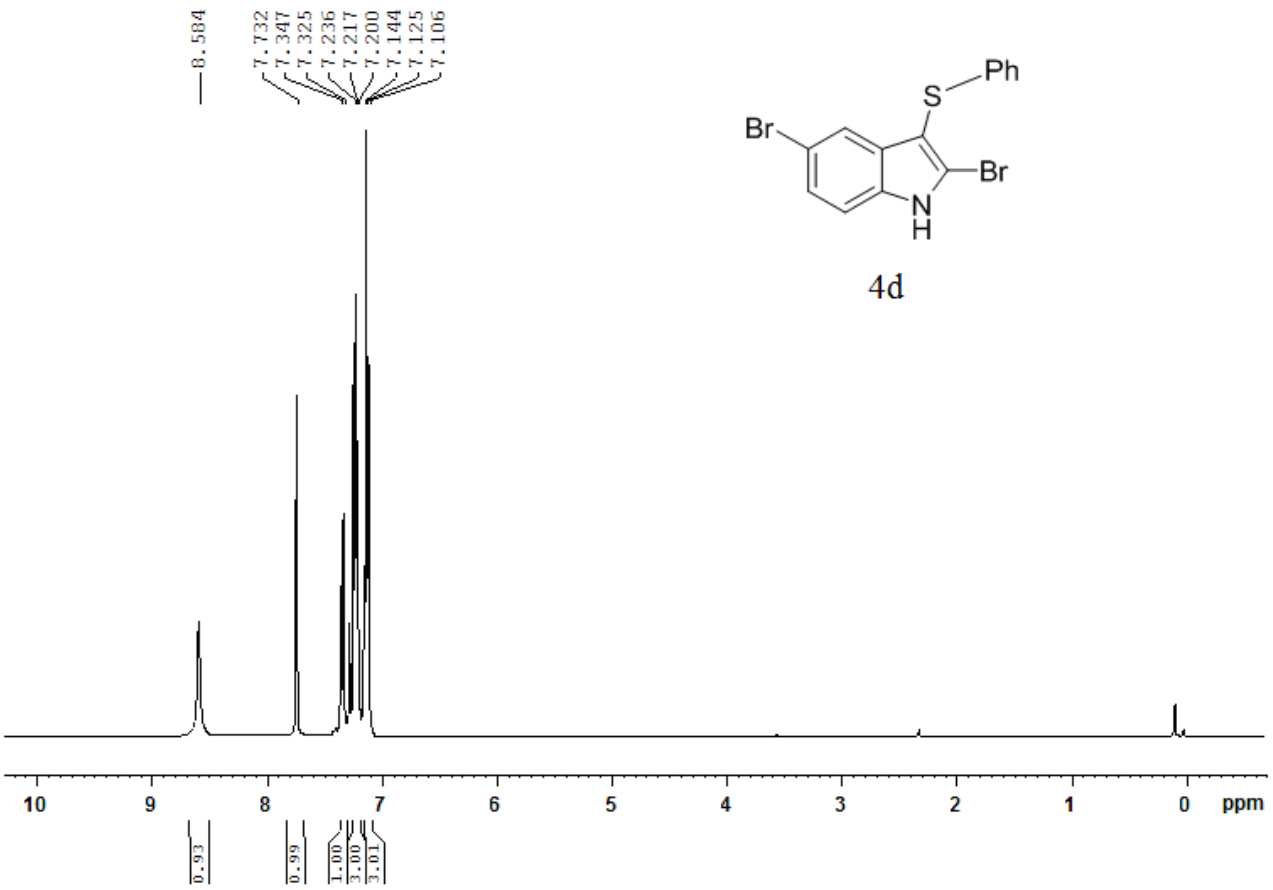
4b


8.392
7.222
7.203
7.185
7.119
7.100
6.946
6.814
5.948
Ph
S
Br
O
O
N
H
9
8
7
6
5
4
3
2
1
0
ppm
0.97
2.03
3.00
1.01
1.02
2.02

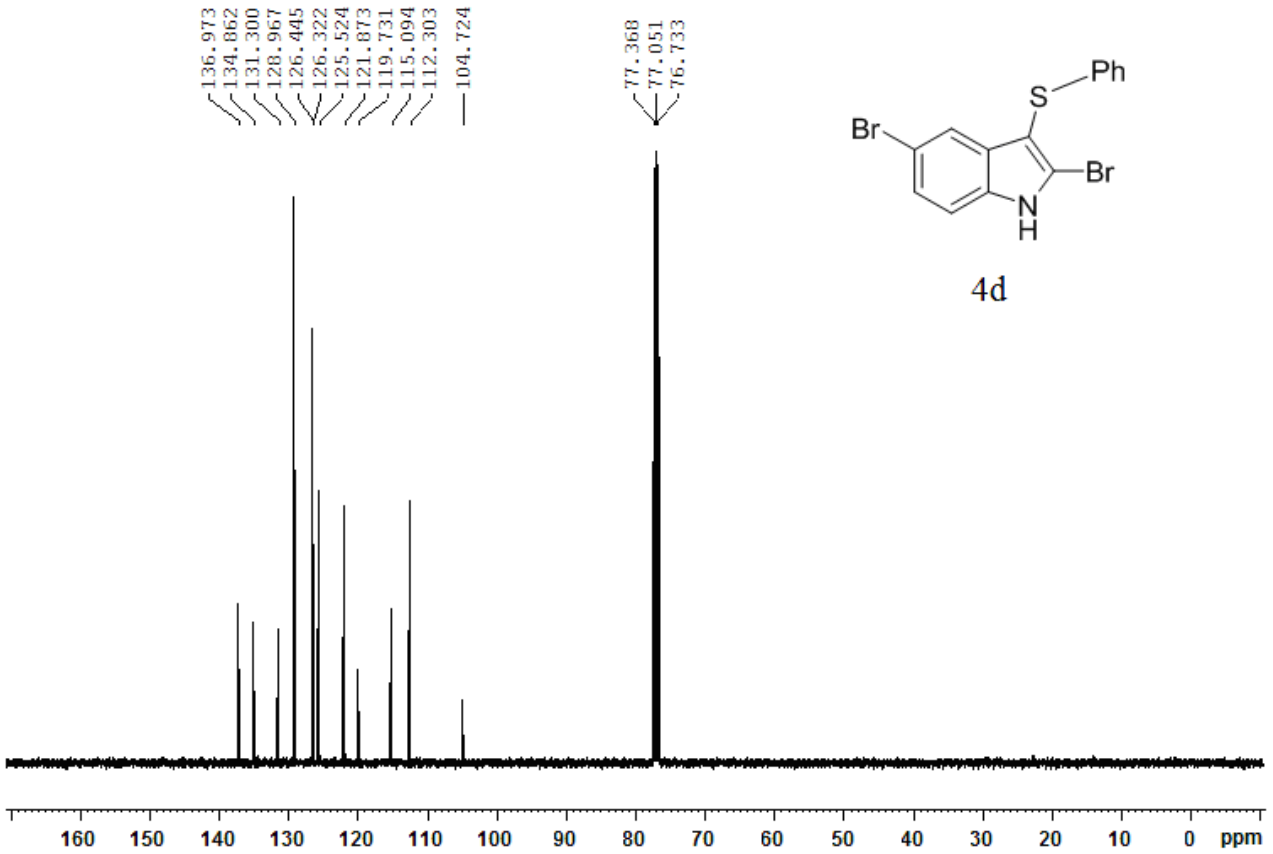
4c


145.804
144.225
137.452
130.958
128.832
126.281
125.224
124.047
114.715
104.864
100.933
98.341
91.983
77.341
77.024
76.706
Ph
S
Br
O
O
N
H
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

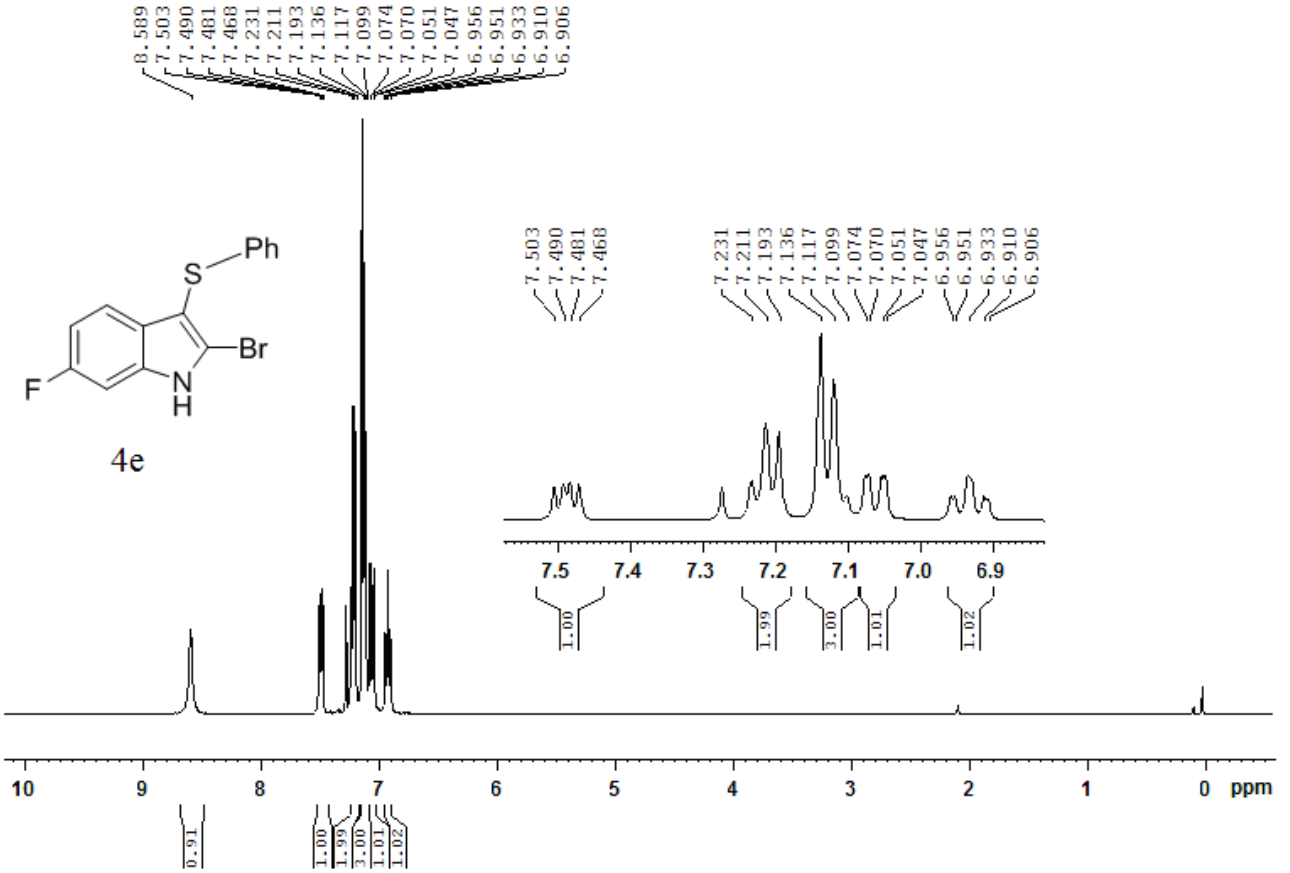
4c

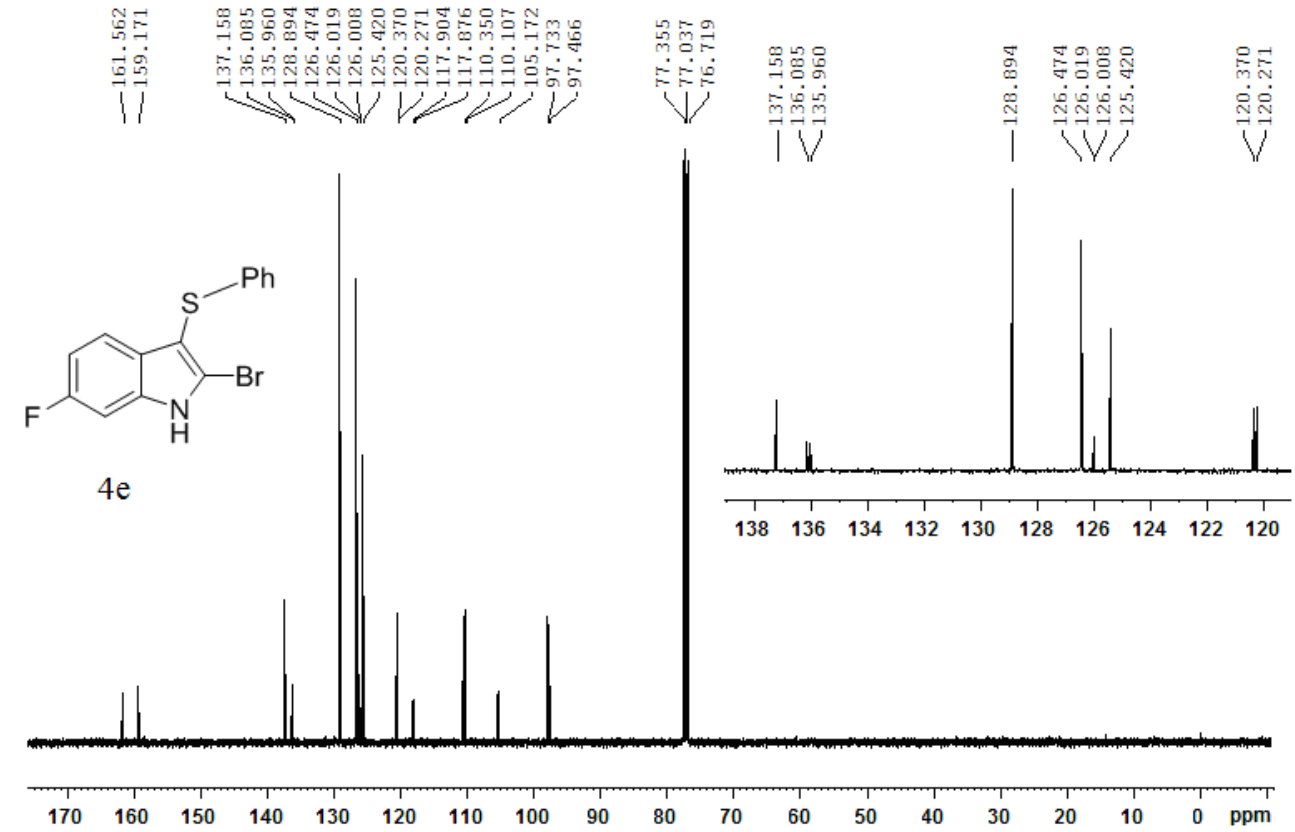

8.584
7.732
7.347
7.325
7.236
7.217
7.200
7.144
7.125
7.106
Ph
S
Br
Br
N
H
10
9
8
7
6
5
4
3
2
1
0
ppm
0.93
0.99
1.00
3.00
3.01

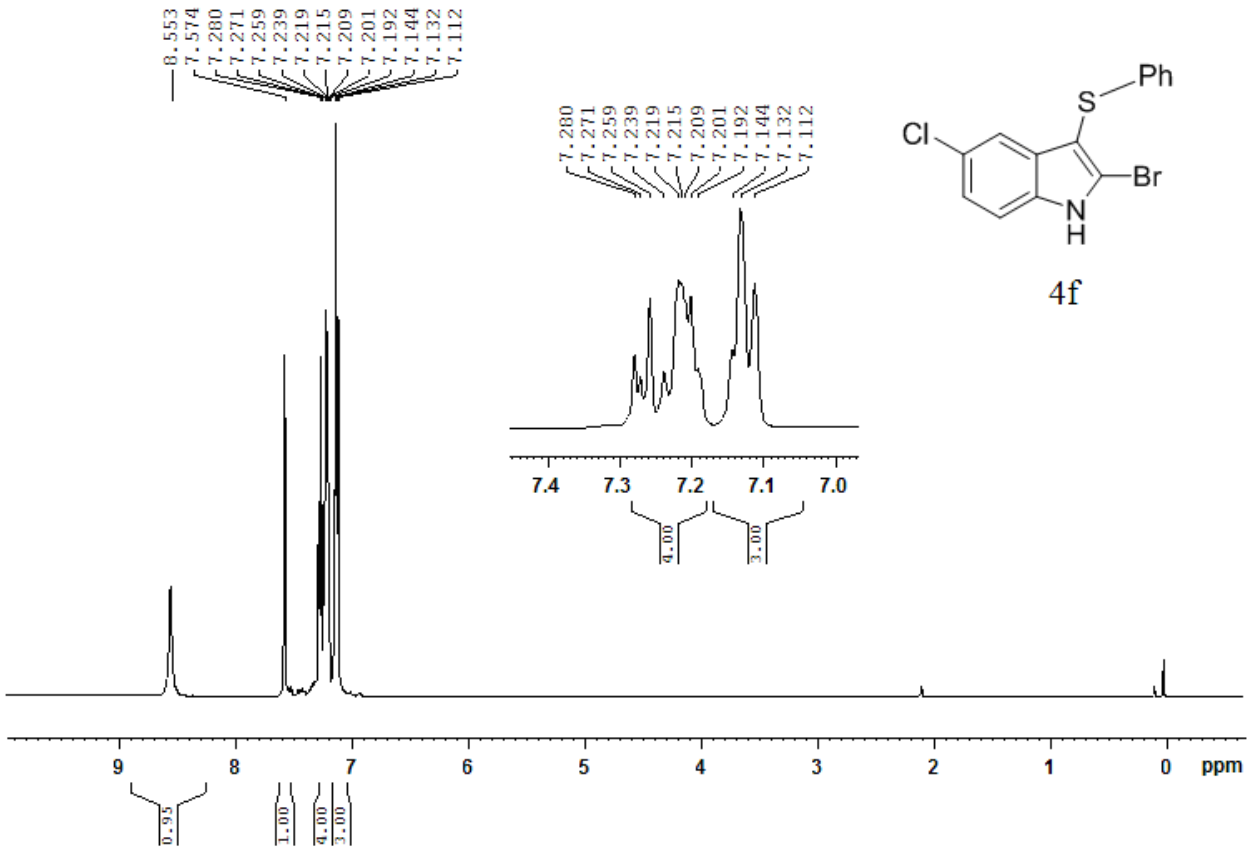
4d

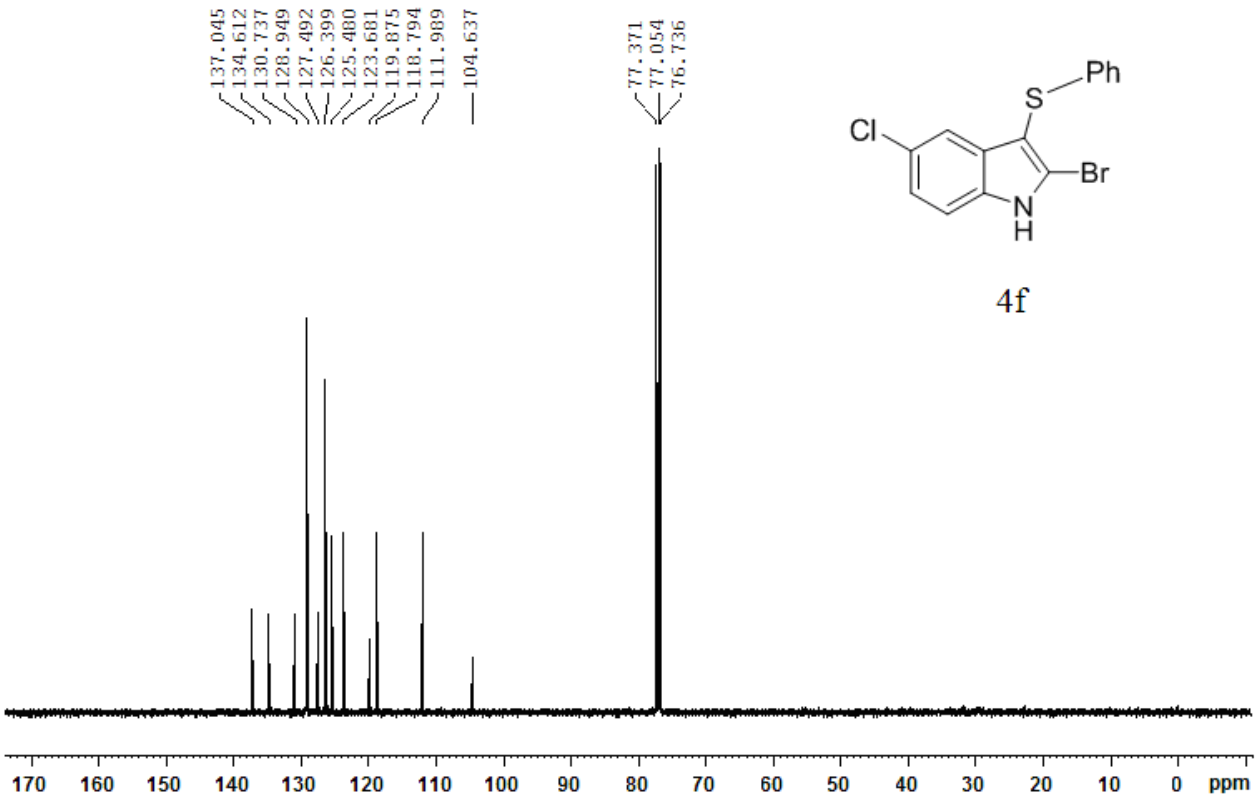

136.973
134.862
131.300
128.967
126.445
126.322
125.524
121.873
119.731
115.094
112.303
104.724
77.368
77.051
76.733
Ph
S
Br
Br
N
H
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

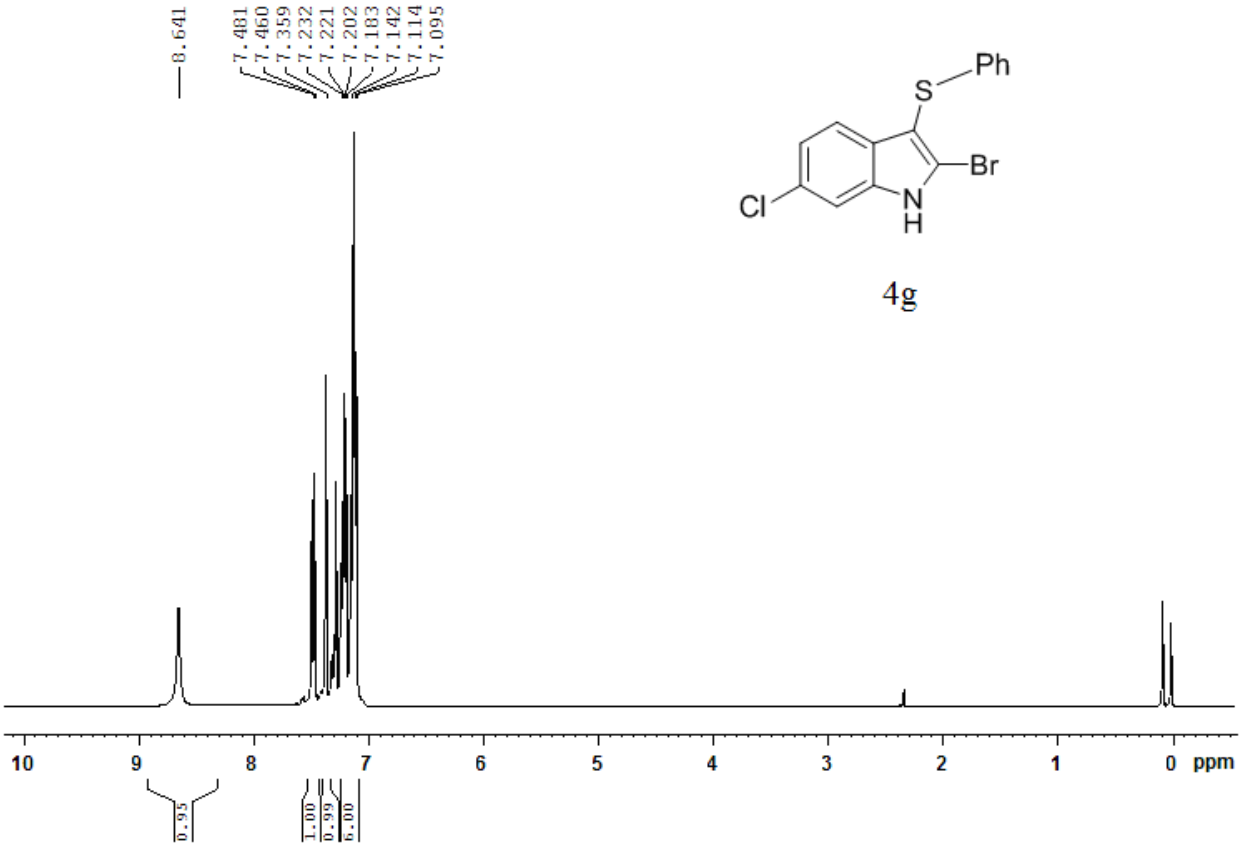
4d


8.589
7.503
7.490
7.481
7.468
7.231
7.211
7.193
7.136
7.117
7.099
7.074
7.070
7.051
7.047
6.956
6.951
6.933
6.910
6.906
Ph
S
Br
F
N
H
4e
10
9
8
7
6
5
4
3
2
1
0
ppm
0.91
1.00
1.99
3.00
1.01
1.02

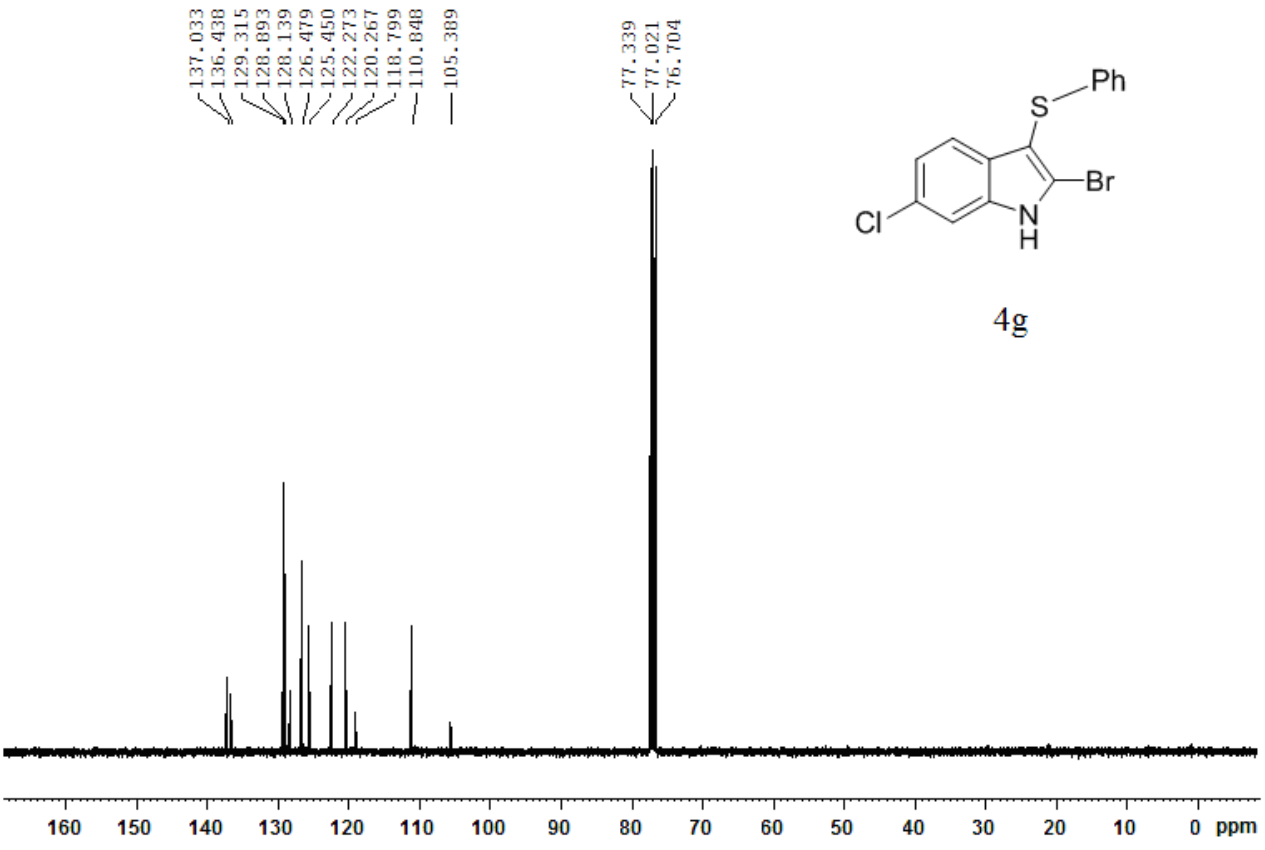

161.562
159.171
137.158
136.085
135.960
128.894
126.474
126.019
126.008
125.420
120.370
120.271
117.904
117.876
110.350
110.107
105.172
97.733
97.466
77.355
77.037
76.719
Ph
S
Br
F
N
H
4e
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.553
7.574
7.280
7.271
7.259
7.239
7.219
7.215
7.209
7.201
7.192
7.144
7.132
7.112
7.4
7.3
7.2
7.1
7.0
4.00
3.00
Ph
S
Cl
Br
N
H
4f
9
8
7
6
5
4
3
2
1
0
ppm
0.93
1.00
4.00
3.00

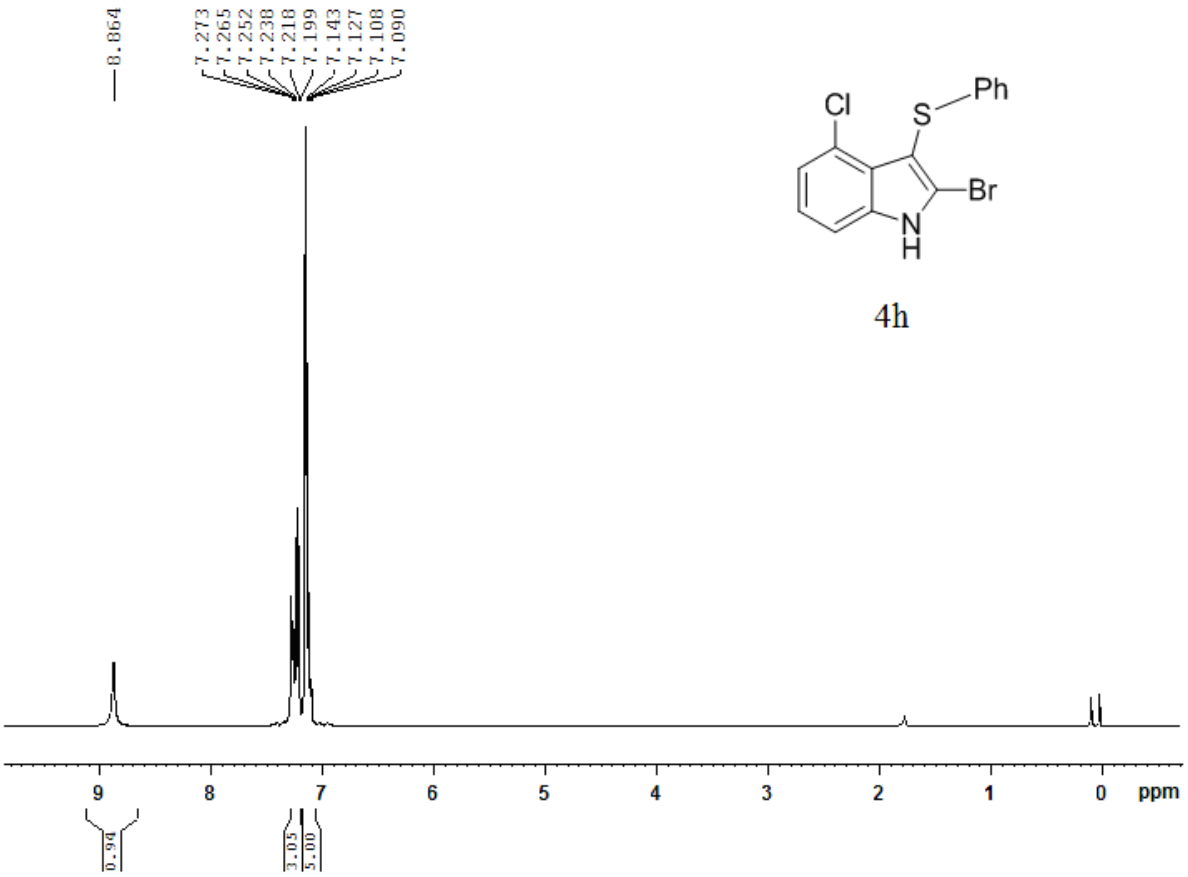

137.045
134.612
130.737
128.949
127.492
126.399
125.480
123.681
119.875
118.794
111.989
104.637
77.371
77.054
76.736
Ph
S
Cl
Br
N
H
4f
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.641
7.481
7.460
7.359
7.232
7.221
7.202
7.183
7.142
7.114
7.095
Ph
S
Br
Cl
N
H
10
9
8
7
6
5
4
3
2
1
0 ppm
0.95
1.00
0.99
6.00

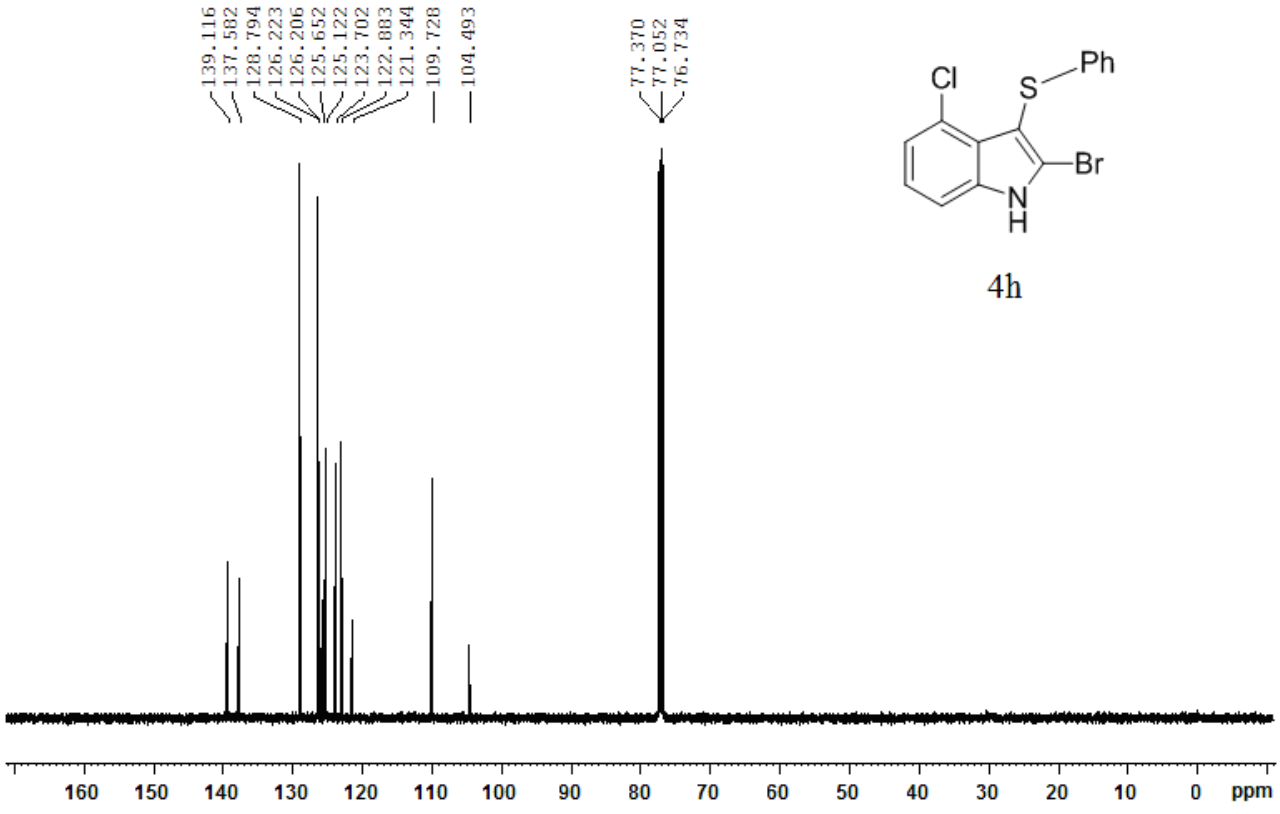
4g


137.033
136.438
129.315
128.893
128.139
126.479
125.450
122.273
120.267
118.799
110.848
105.389
77.339
77.021
76.704
Ph
S
Br
Cl
N
H
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

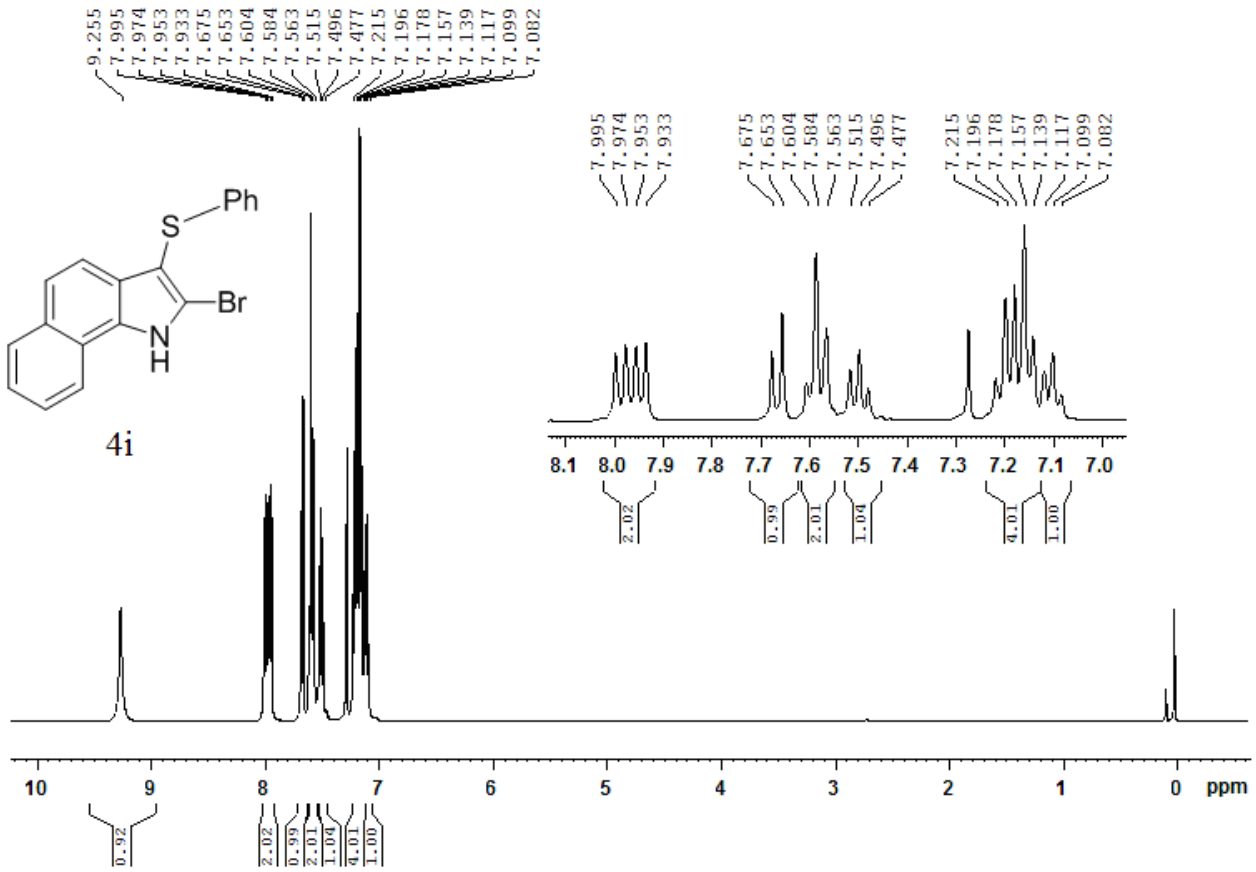
4g

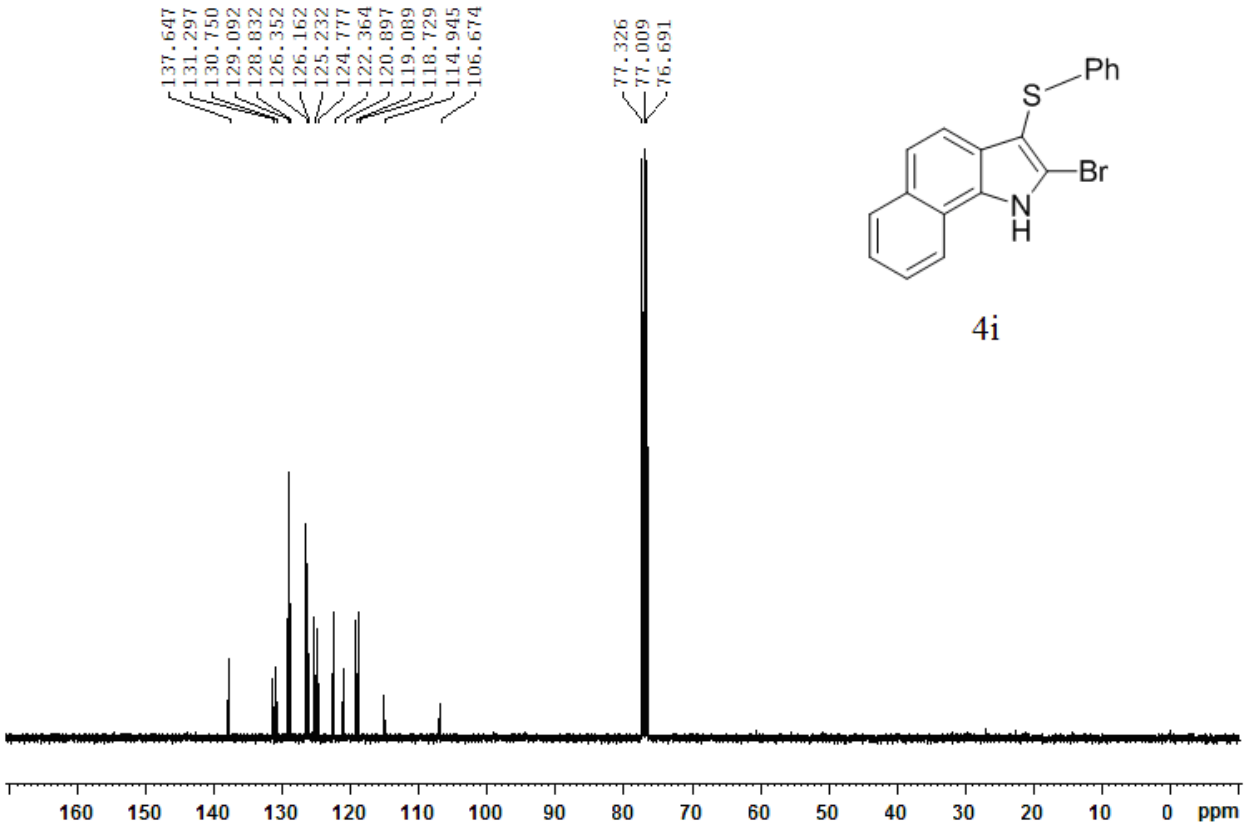

8.864
7.273
7.265
7.252
7.238
7.218
7.199
7.143
7.127
7.108
7.090
Cl
S
Ph
Br
N
H
9
8
7
6
5
4
3
2
1
0
ppm
0.94
3.05
5.00

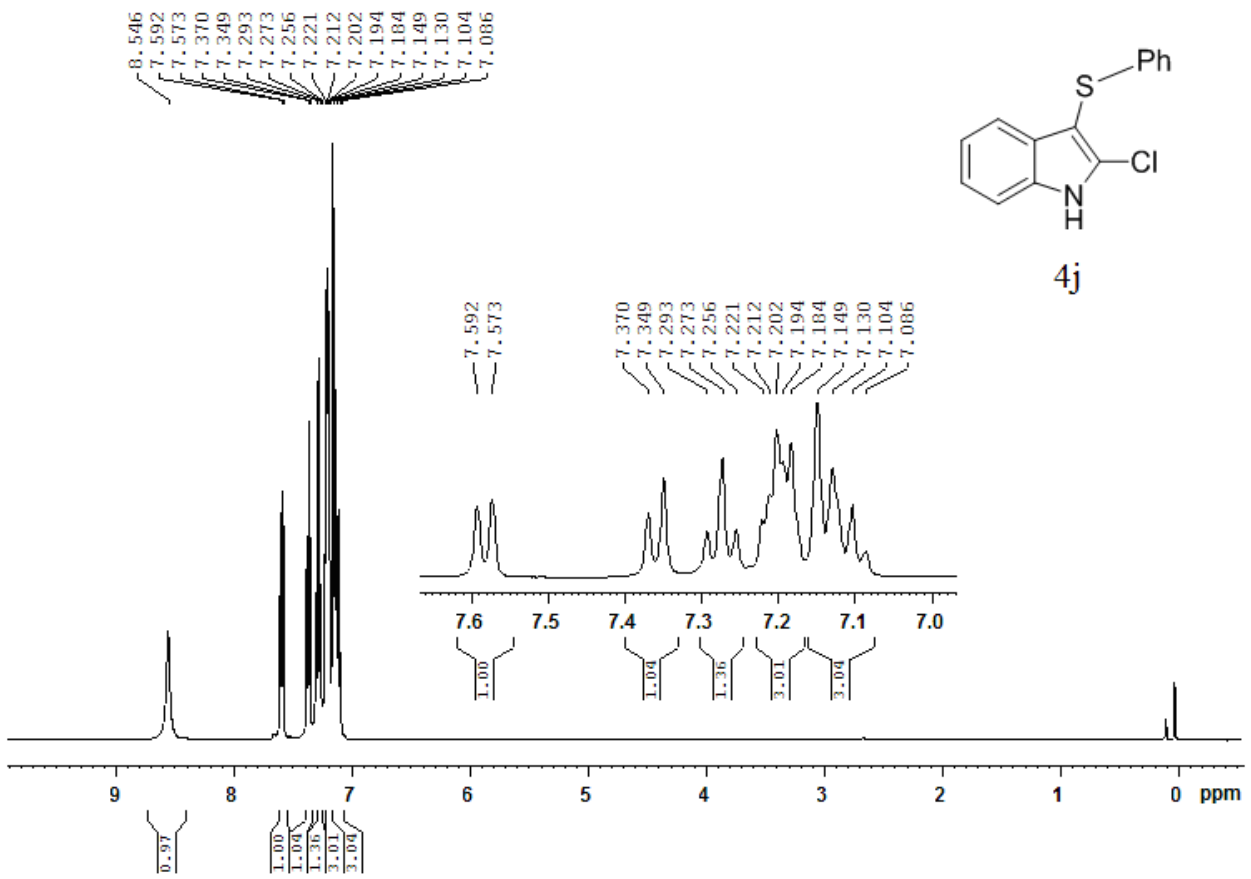
4h

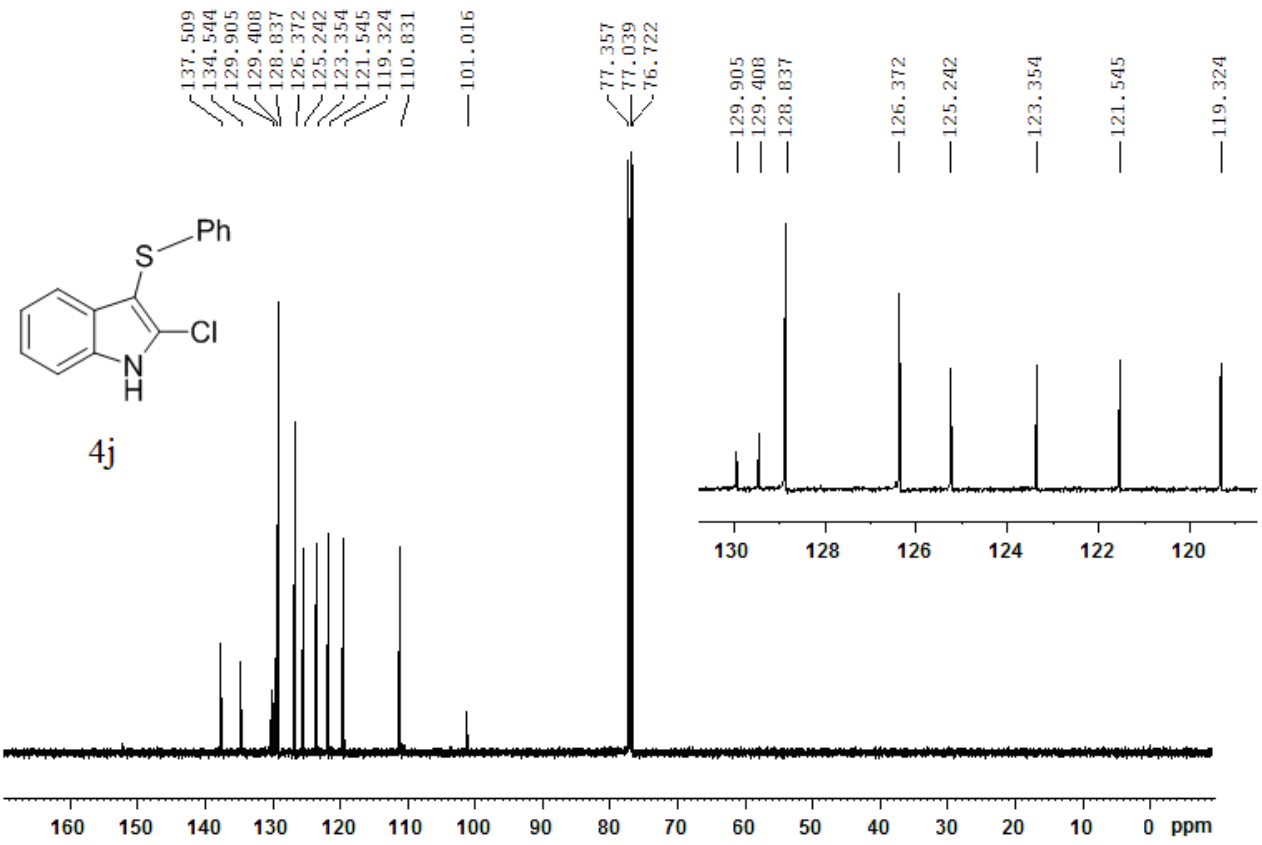

139.116
137.582
128.794
126.223
126.206
125.652
125.122
123.702
122.883
121.344
109.728
104.493
77.370
77.052
76.734
Cl
S
Ph
Br
N
H
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

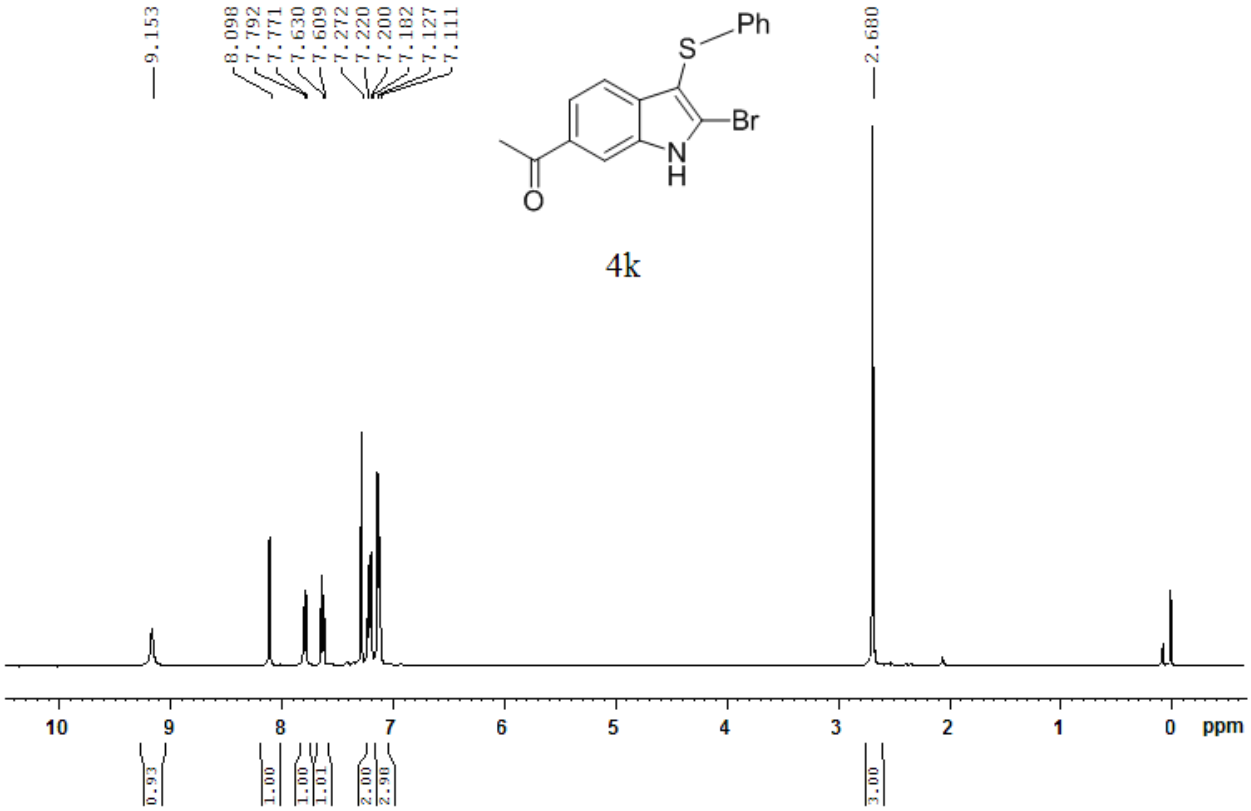
4h


9.255
7.995
7.974
7.953
7.933
7.675
7.653
7.604
7.584
7.563
7.515
7.496
7.477
7.215
7.196
7.178
7.157
7.139
7.117
7.099
7.082
Ph
S
Br
N
H
4i
10 9 8 7 6 5 4 3 2 1 0 ppm
0.92
2.02
0.99
2.01
1.04
4.01
1.00

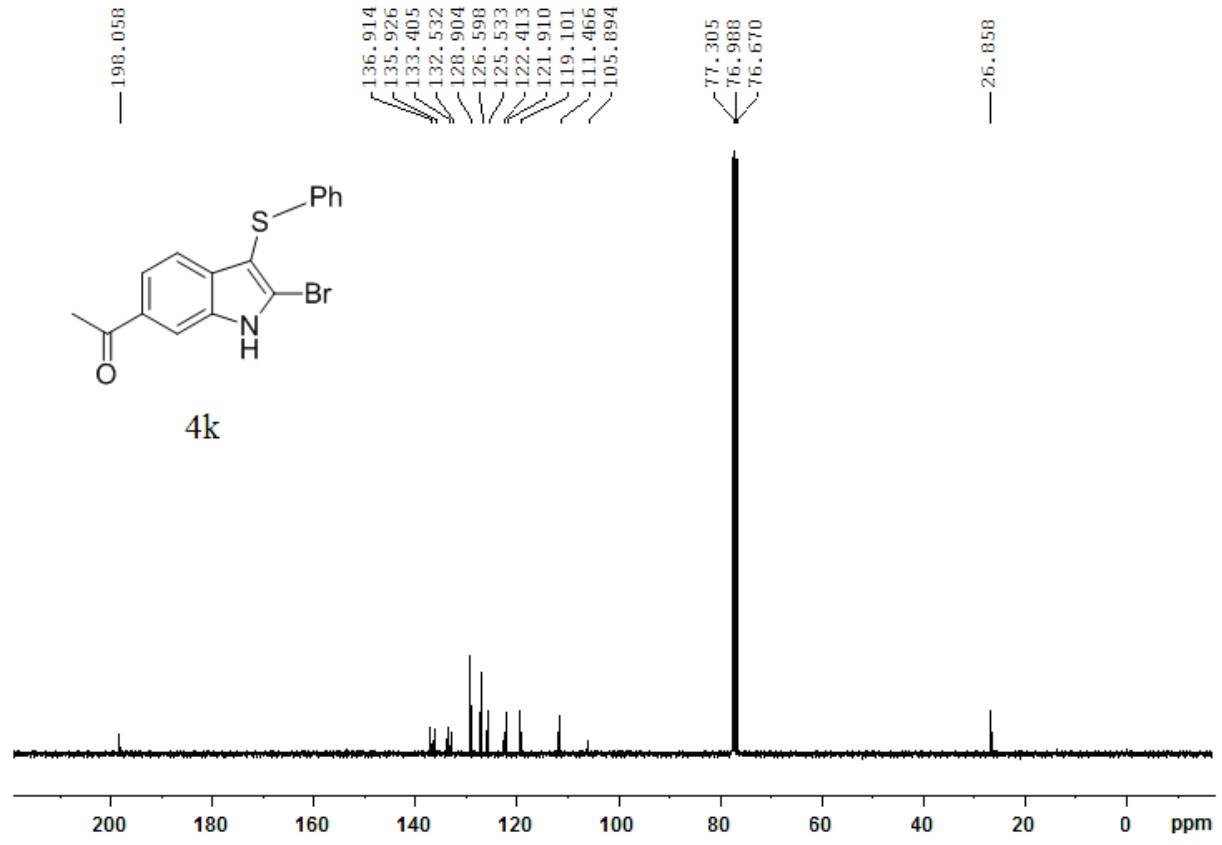

137.647
131.297
130.750
129.092
128.832
126.352
126.162
125.232
124.777
122.364
120.897
119.089
118.729
114.945
106.674
77.326
77.009
76.691
Ph
S
Br
N
H
4i
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.546
7.592
7.573
7.370
7.349
7.293
7.273
7.256
7.221
7.212
7.202
7.194
7.184
7.149
7.130
7.104
7.086
Ph
S
Cl
N
H
4j
9
8
7
6
5
4
3
2
1
0 ppm
0.97
1.00
1.04
1.36
3.01
3.04
7.6
7.5
7.4
7.3
7.2
7.1
7.0

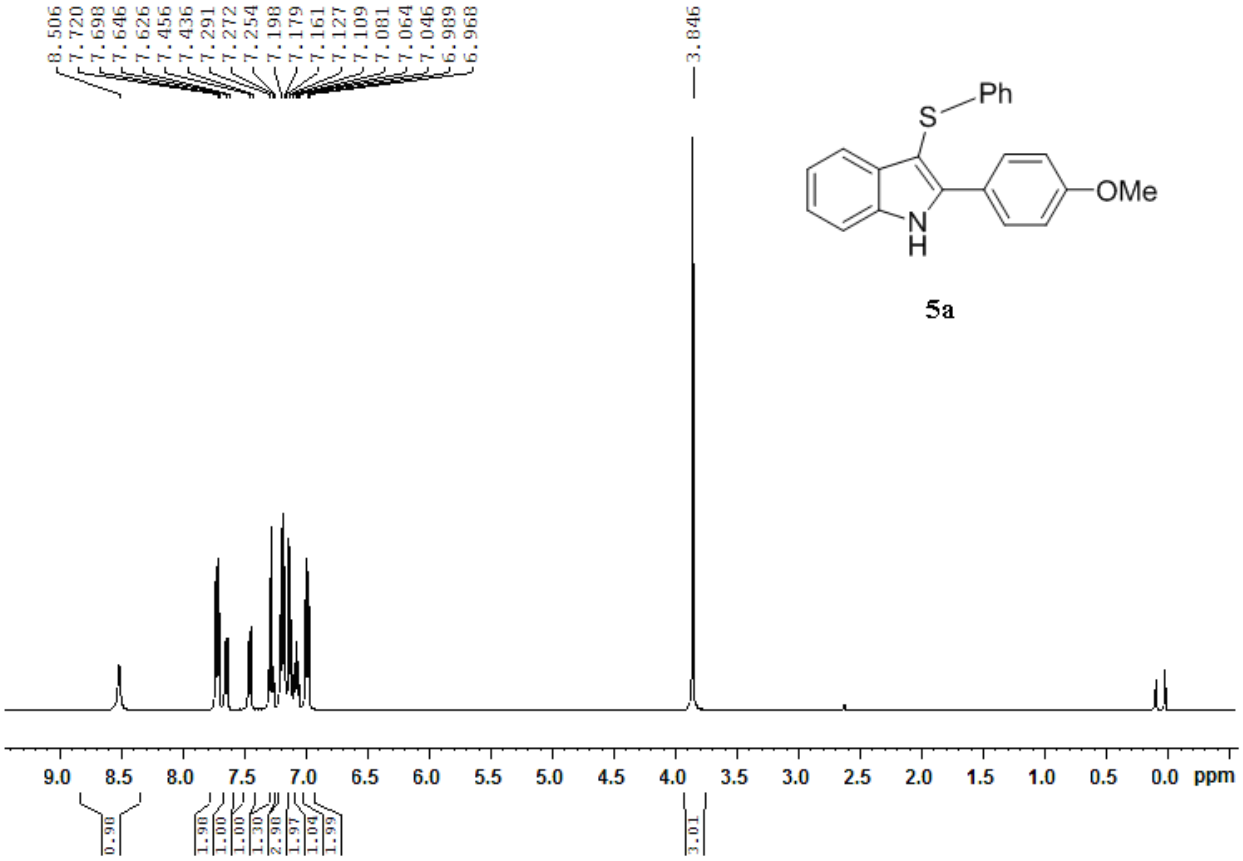

137.509
134.544
129.905
129.408
128.837
126.372
125.242
123.354
121.545
119.324
110.831
101.016
77.357
77.039
76.722
Ph
S
Cl
N
H
4j
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm
130
128
126
124
122
120


9.153
8.098
7.792
7.771
7.630
7.609
7.272
7.220
7.200
7.182
7.127
7.111
2.680
Ph
S
Br
N
H
O
10
9
8
7
6
5
4
3
2
1
0
ppm
0.93
1.00
1.00
1.01
2.00
2.90
3.00

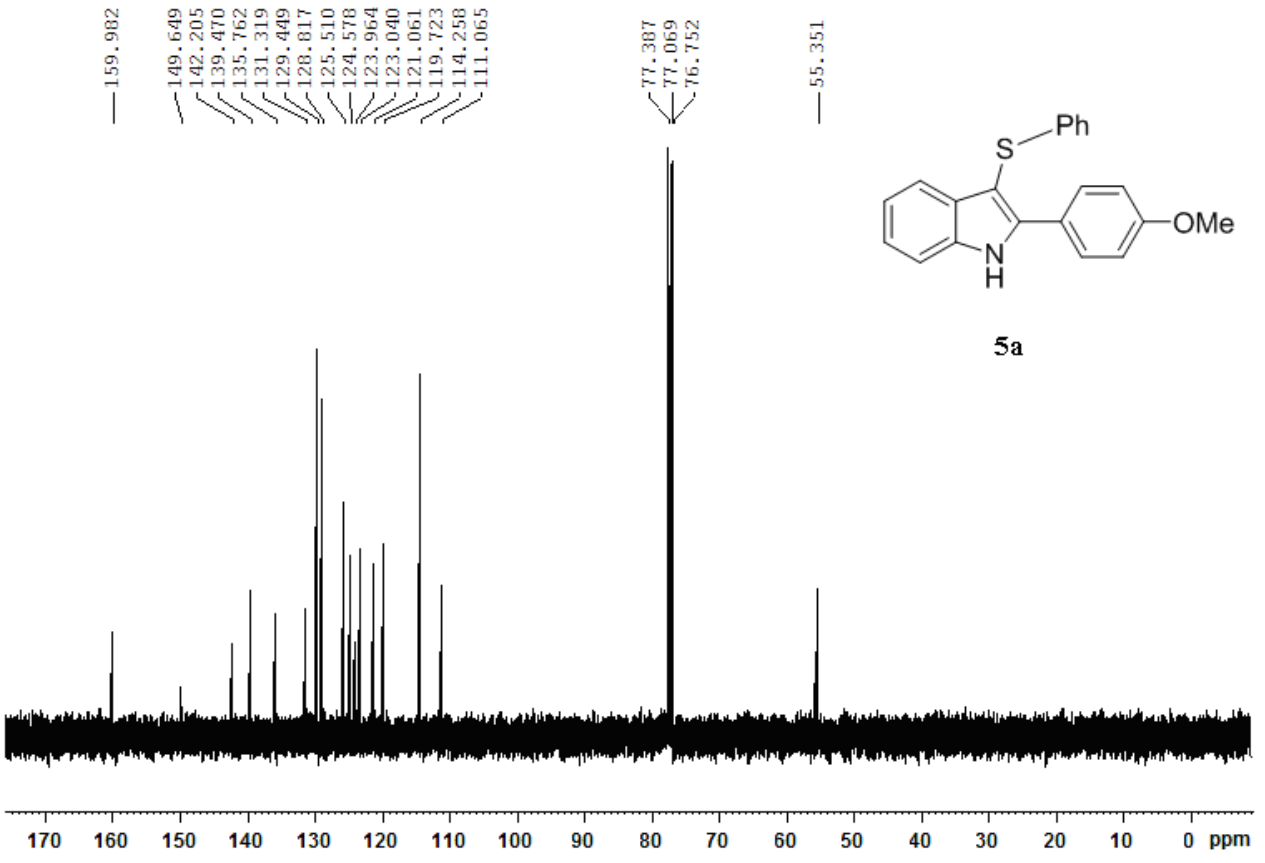
4k


198.058
136.914
135.926
133.405
132.532
128.904
126.598
125.533
122.413
121.910
119.101
111.466
105.894
77.305
76.988
76.670
26.858
Ph
S
Br
N
H
O
200
180
160
140
120
100
80
60
40
20
0
ppm

4k


8.506
7.720
7.698
7.646
7.626
7.456
7.436
7.291
7.272
7.254
7.198
7.179
7.161
7.127
7.109
7.081
7.064
7.046
6.989
6.968
3.846
Ph
S
OMe
N
H
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
1.98
1.00
1.00
1.30
2.98
1.97
1.04
1.99
3.01

5a

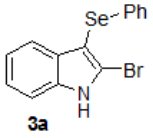

159.982
149.649
142.205
139.470
135.762
131.319
129.449
128.817
125.510
124.578
123.964
123.040
121.061
119.723
114.258
111.065
77.387
77.069
76.752
55.351
Ph
S
OMe
N
H
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

5a

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-01-OS0165
Sample Serial Number: HBSF-Lj8-S2
Operator: Li
Date: 2012/03/18
Elemental Composition Report
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Monoisotopic Mass, Odd and Even Electron Ions
710 formula(e) evaluated with 8 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-15 H: 0-80 N: 0-1 79Br: 0-1 81Br: 0-1 74Se: 0-1 76Se: 0-1 77Se: 0-1 78Se: 0-1 80Se: 0-1
82Se: 0-1



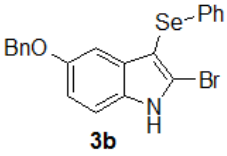
3a

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
344.9217	344.9221	-0.4	-1.2	11.0	5546031.0	C14 H10 N 79Br 74Se
	344.9212	0.5	1.4	1.5	5546030.5	C7 H20 N 74Se 76Se 77Se
	344.9223	-0.6	-1.7	-1.5	5546029.0	C7 H22 81Br 78Se 80Se
	344.9224	-0.7	-2.0	-1.5	5546030.5	C6 H21 N 79Br 77Se 82Se
	344.9226	-0.9	-2.6	12.5	5546030.5	C15 H11 76Se 78Se
	344.9204	1.3	3.8	12.5	5546030.5	C14 H10 N 76Se 77Se
	344.9233	-1.6	-4.6	1.5	5546030.5	C8 H21 74Se 76Se 78Se
	344.9201	1.6	4.6	-1.5	5546030.5	C6 H21 N 81Br 77Se 80Se

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-06-OS0463
Sample Serial Number: HBSF-LiJ06-S35
Operator: Li
Date: 2012/06/01



3b

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
493 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-4 O: 0-4 Br: 0-1 74Se: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
450.9636	450.9633	0.3	0.7	26.0	2773312.8	C28 H6 N O Br
	450.9640	-0.4	-0.9	15.0	2773330.2	C21 H16 N O Br 74Se
	450.9627	0.9	2.0	15.5	2773300.5	C19 H14 N4 Br 74Se
	450.9619	1.7	3.8	26.5	2773302.3	C26 H4 N4 Br
	450.9616	2.0	4.4	30.5	2773308.5	C31 H5 74Se

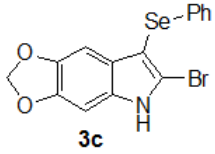
Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

Instrument: Waters Micromass GCT Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-T12-01-OS0169

Sample Serial Number: HBSF-Lj8-S6

Operator: Li



3c

Date: 2012/03/19
Elemental Composition Report
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%
Monoisotopic Mass, Odd and Even Electron Ions
1193 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)

Minimum:		2.0	5.0	-1.5		
Maximum:				50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
388.9126	388.9127	-0.1	-0.2	14.5	1	C16 H10 N O 77Se 80Se
	388.9124	0.2	0.5	13.5	3	C16 H11 O2 76Se 78Se
	388.9120	0.6	1.6	12.0	2	C15 H10 N O2 79Br 74Se
	388.9119	0.7	1.9	13.0	4	C16 H10 N O 79Br 78Se
	388.9117	0.9	2.2	13.0	5	C16 H10 N O 81Br 76Se

Shanghai Mass Spectrometry Center
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Chinese Academic of Sciences
High Resolution MS Data Report

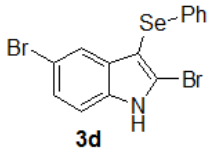
Instrument: Waters Micromass GCT Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-T12-04-OS0312

Sample Serial Number: HBSF-LiA22-S12

Operator: Li

Date: 2012/04/26



3d

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
642 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
422.8330	422.8326	0.4	0.8	11.0	1	C14 H9 N Br2 74Se
	422.8339	-0.9	-2.2	8.5	2	C11 H7 O6 Cl Br 74Se

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

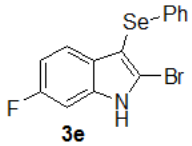
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-04-OS0380

Sample Serial Number: HBSF-LiM6-S31

Operator: Li

Date: 2012/05/18



3e

Elemental Composition Report
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
1064 formula(e) evaluated with 10 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-1 F: 0-1 Br: 0-1 74Se: 0-1 76Se: 0-1 77Se: 0-1 78Se: 0-1 80Se: 0-1
82Se: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
362.9125	362.9127	-0.2	-0.6	11.0	2773035.0	C14 H9 N F Br 74Se
	362.9123	0.2	0.6	3.0	2773309.3	C9 H20 76Se 77Se 82Se
	362.9129	-0.4	-1.1	-1.5	2773310.5	C6 H20 N F Br 77Se 82Se
	362.9121	0.4	1.1	15.5	2773270.8	C17 H9 74Se 76Se
	362.9132	-0.7	-1.9	12.5	2773296.0	C15 H10 F 76Se 78Se
	362.9117	0.8	2.2	1.5	2773307.8	C7 H19 N F 74Se 76Se 77Se
	362.9133	-0.8	-2.2	12.5	2773292.8	C16 H10 Br 82Se
	362.9139	-1.4	-3.9	1.5	2773296.5	C8 H20 F 74Se 76Se 78Se
	362.9140	-1.5	-4.1	1.5	2773296.0	C9 H20 Br 74Se 82Se
	362.9110	1.5	4.1	12.5	2773309.0	C14 H9 N F 76Se 77Se

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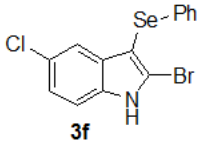
High Resolution MS Data Report

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-01-OS0167
Sample Serial Number: HBSF-Lj8-S4
Operator: Li
Date: 2012/03/18
Elemental Composition Report
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off



3f

Monoisotopic Mass, Odd and Even Electron Ions
1569 formula(e) evaluated with 15 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-20 H: 0-80 N: 0-1 Cl: 0-1 79Br: 0-1 81Br: 0-1 74Se: 0-1 76Se: 0-1 77Se: 0-1 78Se: 0-1
80Se: 0-1 82Se: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
378.8835	378.8836	-0.1	-0.3	12.5	2773087.8	C15 H10 Cl 76Se 78Se
	378.8834	0.1	0.3	18.0	2773097.5	C18 H6 77Se 80Se
	378.8834	0.1	0.3	-1.5	2773097.8	C6 H20 N Cl 79Br 77Se 82Se
	378.8833	0.2	0.5	-1.5	2773091.8	C7 H21 Cl 81Br 78Se 80Se
	378.8833	0.2	0.5	5.5	2773084.3	C11 H16 79Br 74Se 78Se
	378.8832	0.3	0.8	5.5	2773084.3	C11 H16 81Br 74Se 76Se
	378.8832	0.3	0.8	11.0	2773023.8	C14 H9 N Cl 79Br 74Se
	378.8841	-0.6	-1.6	7.0	2773096.3	C11 H16 74Se 77Se 80Se
	378.8843	-0.8	-2.1	1.5	2773086.0	C8 H20 Cl 74Se 76Se 78Se
	378.8826	0.9	2.4	16.5	2773084.3	C18 H6 79Br 78Se
	378.8844	-0.9	-2.4	2.5	2773095.8	C10 H17 79Br 81Br 82Se
	378.8824	1.1	2.9	16.5	2773084.3	C18 H6 81Br 76Se
	378.8822	1.3	3.4	1.5	2773095.5	C7 H19 N Cl 74Se 76Se 77Se

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High Resolution MS Data Report

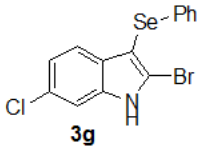
Instrument: Waters Micromass GCT Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-T*12-04-OS*0320

Sample Serial Number: HBSF-LiA22-S20

Operator: Li

Date: 2012/04/26



3g

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
135 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
378.8827	378.8827	0.0	0.1	15.5	1	C17 H6 O Br 74Se
	378.8832	-0.5	-1.2	11.0	2	C14 H9 N Cl Br 74Se

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

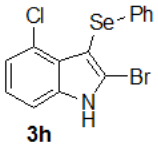
Instrument: Waters Micromass GCT Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-T*12-04-OS*0319

Sample Serial Number: HBSF-LiA22-S19

Operator: Li

Date: 2012/04/26



3h

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
135 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
378.8830	378.8832	-0.2	-0.4	11.0	2	C14 H9 N Cl Br 74Se
	378.8827	0.3	0.8	15.5	1	C17 H6 O Br 74Se

Shanghai Mass Spectrometry Center
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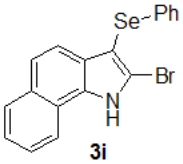
High Resolution MS Data Report

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-04-OS0382

Sample Serial Number: HBSF-LiM6-S33

Operator: Li



3i

Date: 2012/05/18
Elemental Composition Report
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Monoisotopic Mass, Odd and Even Electron Ions
1237 formula(e) evaluated with 14 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-1 F: 0-1 Br: 0-1 74Se: 0-1 76Se: 0-1 77Se: 0-1 78Se: 0-1 80Se: 0-1
82Se: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
394.9374	394.9375	-0.1	-0.3	2.0	2773045.5	C10 H23 F 74Se 77Se 82Se
	394.9371	0.3	0.8	25.0	2773041.3	C25 H2 N Br
	394.9378	-0.4	-1.0	14.0	2773013.8	C18 H12 N Br 74Se
	394.9379	-0.5	-1.3	1.5	2773045.0	C9 H23 N F 76Se 77Se 78Se
	394.9368	0.6	1.5	4.5	2773044.3	C11 H22 N 74Se 76Se 77Se
	394.9380	-0.6	-1.5	1.5	2773046.5	C10 H23 N Br 77Se 82Se
	394.9367	0.7	1.8	13.0	2773046.5	C17 H13 F 77Se 82Se
	394.9382	-0.8	-2.0	15.5	2773038.5	C19 H13 76Se 78Se
	394.9365	0.9	2.3	25.5	2773014.5	C25 H2 F 74Se
	394.9361	1.3	3.3	15.5	2773045.3	C18 H12 N 76Se 77Se
	394.9388	-1.4	-3.5	11.0	2773036.0	C16 H13 N F Br 78Se
	394.9390	-1.6	-4.1	4.5	2773036.8	C12 H23 74Se 76Se 78Se
	394.9357	1.7	4.3	0.5	2773037.3	C10 H23 F Br 74Se 80Se
	394.9355	1.9	4.8	3.0	2773041.8	C10 H23 N 76Se 80Se 82Se

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Chinese Academic of Sciences
High Resolution MS Data Report

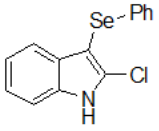
Instrument: Waters Micromass GCT Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-T12-04-OS0316

Sample Serial Number: HBSF-LiA22-S16

Operator: Li

Date: 2012/04/26



3j

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
190 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
300.9726	300.9727	-0.1	-0.2	11.0	3	C14 H10 N Cl 74Se
	300.9722	0.4	1.4	15.5	2	C17 H7 O 74Se
	300.9719	0.7	2.2	22.0	1	C21 N Cl

Shanghai Mass Spectrometry Center
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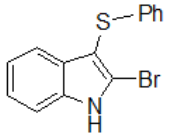
Chinese Academic of Sciences
High Resolution MS Data Report

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T11-12-OS0935
Sample Serial Number: HBSF-D28-S24
Operator: Li
Date: 2011/12/15
Elemental Composition Report
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Monoisotopic Mass, Odd and Even Electron Ions
445 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-4 O: 0-6 S: 0-1 Br: 0-1



4a

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
302.9721	302.9717	0.4	1.3	10.0	2775312.5	C14 H10 N S Br
	302.9729	-0.8	-2.6	6.5	2775214.3	C8 H8 N4 O4 Br
	302.9712	0.9	3.0	12.5	2775362.8	C12 H3 N2 O6 S

Shanghai Mass Spectrometry Center
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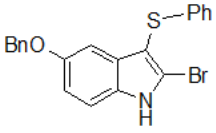
Chinese Academic of Sciences
High Resolution MS Data Report

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-06-OS0462
Sample Serial Number: HBSF-LiJ06-S34
Operator: Li
Date: 2012/06/01



4b

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
460 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-4 O: 0-4 S: 0-1 Br: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
409.0139	409.0136	0.3	0.7	14.0	2.6	C21 H16 N O S Br
	409.0150	-1.1	-2.7	30.5	1035.8	C28 H N4 O
	409.0124	1.5	3.7	26.0	1024.5	C25 H3 N3 O4
	409.0123	1.6	3.9	14.5	1.8	C19 H14 N4 S Br
	409.0157	-1.8	-4.4	21.0	942.9	C22 H7 N3 O4 S

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

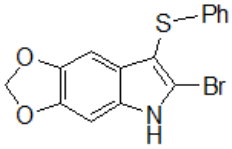
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-01-OS0168

Sample Serial Number: HBSF-Lj8-S5

Operator: Li

Date: 2012/03/18



4c

Elemental Composition Report
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Monoisotopic Mass, Odd and Even Electron Ions
191 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-30 H: 0-80 N: 0-1 O: 0-2 S: 0-1 Cl: 0-1 Br: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
346.9613	346.9616	-0.3	-0.9	11.0	1.0	C15 H10 N O2 S Br
	346.9596	1.7	4.9	22.0	213.3	C22 H2 N S Cl

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

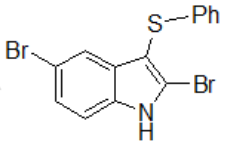
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-04-OS0311

Sample Serial Number: HBSF-LiA22-S11

Operator: Li

Date: 2012/04/25



4d

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
749 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-1 O: 0-6 S: 0-2 Br: 0-3 I: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
380.8824	380.8822	0.2	0.5	10.0	5547044.5	C14 H9 N S Br2
	380.8829	-0.5	-1.3	0.5	5547044.5	C8 H15 O3 S2 Br2
	380.8835	-1.1	-2.9	1.5	5547043.0	C7 H11 O5 Br I
	380.8810	1.4	3.7	5.5	5547044.0	C11 H11 S Br I
	380.8838	-1.4	-3.7	3.0	5547008.5	C6 H8 N O6 S2 I

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

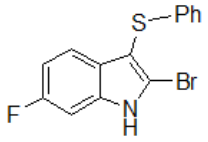
Instrument: Waters Micromass GCT Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-T12-04-OS0318

Sample Serial Number: HBSF-LiA22-S18

Operator: Li

Date: 2012/04/26



4e

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
701 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
320.9619	320.9618	0.1	0.4	21.0	5	C20 N O2 Cl
	320.9623	-0.4	-1.3	10.0	1	C14 H9 N Br S F
	320.9615	0.4	1.3	1.0	2	C7 H13 N O6 Cl Br
	320.9624	-0.5	-1.7	11.5	3	C14 H6 O5 Cl S
	320.9629	-1.0	-3.1	17.0	4	C17 H N O3 Cl F

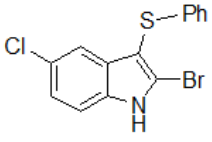
Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-01-OS0166

Sample Serial Number: HBSF-Lj8-S3

Operator: Li



4f

Date: 2012/03/18
Elemental Composition Report
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Monoisotopic Mass, Odd and Even Electron Ions
123 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-20 H: 0-80 N: 0-1 O: 0-2 S: 0-1 Cl: 0-1 Br: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
336.9323	336.9323	0.0	0.0	14.5	33.7	C17 H6 O S Br
	336.9328	-0.5	-1.5	10.0	1.1	C14 H9 N S Cl Br

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

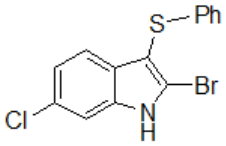
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-04-OS0315

Sample Serial Number: HBSF-LiA22-S15

Operator: Li

Date: 2012/04/25



4g

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
759 formula(e) evaluated with 6 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-1 O: 0-6 S: 0-1 Cl: 0-1 Br: 0-3 I: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
336.9331	336.9328	0.3	0.9	10.0	5546027.5	C14 H9 N S Cl Br
	336.9323	0.8	2.4	14.5	5546027.0	C17 H6 O S Br
	336.9340	-0.9	-2.7	1.5	5546025.5	C7 H11 O5 Cl I
	336.9315	1.6	4.7	5.5	5546025.5	C11 H11 S Cl I
	336.9347	-1.6	-4.7	-1.0	5546028.0	C7 H17 N O2 S Br2
	336.9348	-1.7	-5.0	10.5	5546027.0	C13 H6 O6 Br

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

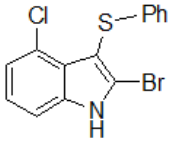
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-04-OS0313

Sample Serial Number: HBSF-LiA22-S13

Operator: Li

Date: 2012/04/25



4h

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
759 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-1 O: 0-6 S: 0-1 Cl: 0-1 Br: 0-3 I: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
336.9327	336.9328	-0.1	-0.3	10.0	33.9	C14 H9 N S Cl Br
	336.9323	0.4	1.2	14.5	95.2	C17 H6 O S Br
	336.9315	1.2	3.6	5.5	276.6	C11 H11 S Cl I
	336.9340	-1.3	-3.9	1.5	276.1	C7 H11 O5 Cl I
	336.9313	1.4	4.2	4.0	29.9	C10 H13 N O2 Br2

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

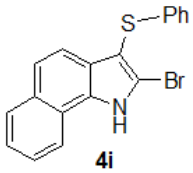
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-04-OS0381

Sample Serial Number: HBSF-LiM6-S32

Operator: Li

Date: 2012/05/18



4i

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
393 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-4 O: 0-4 S: 0-1 Br: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
352.9875	352.9875	0.0	0.0	24.5	1261.0	C24 H O4
	352.9874	0.1	0.3	13.0	0.1	C18 H12 N S Br
	352.9885	-1.0	-2.8	9.5	19.7	C12 H10 N4 O4 Br

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

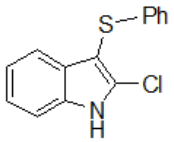
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-04-OS0314

Sample Serial Number: HBSF-LiA22-S14

Operator: Li

Date: 2012/04/25



4j

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
422 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-1 O: 0-6 S: 0-1 Cl: 0-1 Br: 0-3 I: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
259.0223	259.0222	0.1	0.4	10.0	169.7	C14 H10 N S Cl
	259.0218	0.5	1.9	14.5	682.2	C17 H7 O S

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

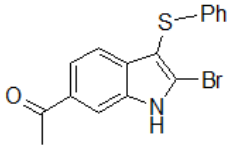
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-06-OS0464

Sample Serial Number: HBSF-LiJ06-S36

Operator: Li

Date: 2012/06/01



4k

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
382 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-4 O: 0-4 S: 0-1 Br: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
344.9828	344.9823	0.5	1.4	11.0	3.2	C16 H12 N O S Br
	344.9844	-1.6	-4.6	18.0	604.1	C17 H3 N3 O4 S

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

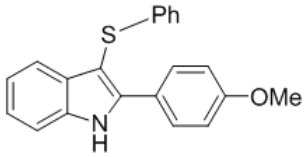
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-06-OS0465

Sample Serial Number: HBSF-LiJ06-S37

Operator: Li

Date: 2012/06/01



5a

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
369 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-4 O: 0-4 S: 0-1 Br: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
331.1029	331.1031	-0.2	-0.6	14.0	2.2	C21 H17 N O S
	331.1021	0.8	2.4	3.5	8236.4	C14 H24 N2 O2 Br
	331.1017	1.2	3.6	14.5	4.8	C19 H15 N4 S