Convenient KI-catalyzed regioselective synthesis of 2-sulfonylindoles using water as solvent

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A typical procedure for the preparation of 2-sulfonylindoles:

Indoles **1** (0.2 mmol), sodium sulfinates **2** (0.3 mmol), KI (0.04 mmol) and Oxone® (0.5 mmol) were added successively to water (3 mL). The suspension was vigorously stirred at r. t. for 1 h. Upon completion, the reaction was quenched by addition of sat. aq. $Na_2S_2O_3$ (2 mL). The mixture was extracted with CH_2Cl_2 (3×5 mL) and the combined organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was then purified on a silica gel plate (4:1 petroleum ether-ethyl acetate) to furnish products **3**.

2-(Phenylsulfonyl)-1*H*-indole (3a)

White solid; mp: 136–137°C (Lit.[1] 137–138°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.45 (br s, 1H), 8.05-8.01 (m, 2H), 7.68 (d, J = 8.1 Hz, 1H), 7.60-7.54 (m, 1H), 7.52-7.47 (m, 2H), 7.45 (d, J = 8.4 Hz, 1H), 7.36-7.31 (m, 1H), 7.28-7.22 (m, 1H), 7.21-7.16 (m, 1H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 141.5, 137.2, 134.0, 133.5, 129.4, 127.3, 127.1, 126.1, 122.7, 121.6, 112.4, 109.3;

MS (ESI): m/z (%) 280 [(M+Na)$^+$, 45], 117 (100).

1-Methyl-2-(phenylsulfonyl)-1*H*-indole (3b)

Pale yellow solid; mp: 124–125°C (Lit.[2] 118–119°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 8.01-7.96 (m, 2H), 7.73 (d, J = 8.1 Hz, 1H), 7.61-7.58 (m, 1H), 7.56-7.51 (m, 2H), 7.41-7.36 (m, 2H), 7.33 (d, J = 8.5 Hz, 1H), 7.23-7.18 (m, 1H), 3.87 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 141.3, 139.5, 134.8, 133.4, 129.3, 127.6, 125.7, 125.2, 122.8, 121.2, 110.8, 110.2, 31.0;

MS (ESI): m/z (%) 294 [(M+Na)$^+$, 42], 131 (100).

3-Methyl-2-(phenylsulfonyl)-1*H*-indole (3c)

White solid; mp: 186–187°C (Lit.[1] 167–168°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.44 (br s, 1H), 8.04-8.01 (m, 2H), 7.63 (d, J = 8.2 Hz, 1H), 7.59-7.53 (m, 1H), 7.52-7.46 (m, 2H), 7.44 (d, J = 8.4 Hz, 1H), 7.37-7.31 (m, 1H), 7.20-7.15 (m, 1H), 2.58 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 144.5, 138.6, 137.1, 134.5, 130.0, 127.3, 127.1, 125.9, 122.6, 121.5, 112.4, 108.9, 21.6;

MS (ESI): m/z (%) 294 [(M+Na)$^+$, 81], 117 (100).

4-Methyl-2-(phenylsulfonyl)-1*H*-indole (3d)

White solid; mp: 153–154°C (Lit.[1] 167–170°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.58 (br s, 1H), 8.07-8.03 (m, 2H), 7.60-7.54 (m, 1H), 7.52-

7.48 (m, 2H), 7.30-7.26 (m, 2H), 7.24-7.21 (m, 1H), 6.98-6.95 (m, 1H), 2.55 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 141.6, 137.3, 133.4, 133.2, 132.4, 129.3, 127.3, 127.2, 126.2, 121.4, 109.9, 108.0, 18.5;

MS (ESI): *m/z* (%) 294 [(M+Na)$^+$, 67], 131 (100).

5-Methyl-2-(phenylsulfonyl)-1*H*-indole (3e)

Pale yellow solid; mp: 116–117°C (Lit.[2] 116–117°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.51 (br s, 1H), 8.05-8.01 (m, 2H), 7.58-7.52 (m, 1H), 7.51-7.42 (m, 3H), 7.33 (d, J = 8.5 Hz, 1H), 7.18-7.13 (m, 2H), 2.43 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 141.6, 135.7, 133.8, 133.3, 131.0, 129.3, 128.0, 127.3, 127.2, 121.8, 112.1, 108.8, 21.4;

MS (ESI): *m/z* (%) 294 [(M+Na)$^+$, 58], 131 (100).

7-Methyl-2-(phenylsulfonyl)-1*H*-indole (3f)

Pale yellow solid; mp: 139–140°C (Lit.[2] 139–140°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.34 (br s, 1H), 8.09-8.05 (m, 2H), 7.60-7.50 (m, 4H), 7.25 (d, J = 2.2 Hz, 1H), 7.16-7.07 (m, 2H), 2.50 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 141.7, 137.1, 133.7, 133.4, 129.4, 127.3, 126.8, 126.3, 121.9, 120.2, 109.9, 16.7;

MS (ESI): *m/z* (%) 294 [(M+Na)$^+$, 43], 131 (100).

5-Methoxy-2-(phenylsulfonyl)-1*H*-indole (3g)

White solid; mp: 117–118°C (Lit.[2] 121–122°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.50 (br s, 1H), 8.04-8.00 (m, 2H), 7.58-7.53 (m, 1H), 7.50-7.45 (m, 2H), 7.33 (d, J = 9.0 Hz, 1H), 7.16 (dd, J = 2.0, 0.7 Hz, 1H), 7.05 (d, J = 2.3 Hz, 1H), 7.00 (dd, J = 9.0, 2.5 Hz, 1H), 3.83 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 155.2, 141.6, 134.0, 133.4, 132.7, 129.3, 127.5, 127.2, 117.8, 113.4, 108.8, 102.5, 55.7;

MS (ESI): *m/z* (%) 310 [(M+Na)$^+$, 60], 147 (100).

5-Fluoro-2-(phenylsulfonyl)-1*H*-indole (3h)

Pale yellow solid; mp: 142–143°C (Lit.[2] 144–145°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.52 (br s, 1H), 8.05-8.01 (m, 2H), 7.63-7.57 (m, 1H), 7.55-7.49 (m, 2H), 7.38 (dd, J = 9.0, 4.3 Hz, 1H), 7.32-7.27 (m, 1H), 7.17 (dd, J = 2.1, 0.8 Hz, 1H), 7.10 (td, J = 9.1, 2.5 Hz, 1H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 159.4, 157.5, 141.2, 135.6, 133.7 (d, J = 12.6 Hz), 129.5, 128.0, 127.3, 115.4 (d, J = 26.9 Hz), 113.5 (d, J = 9.5 Hz), 108.9 (d, J = 5.3 Hz), 107.0 (d, J = 23.5Hz);

MS (ESI): *m/z* (%) 298 [(M+Na)$^+$, 100].

5-Bromo-2-(phenylsulfonyl)-1*H*-indole (3i)

Pale yellow solid; mp: 154–155°C (Lit.[2] 158–159°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.57 (br s, 1H), 8.07-8.00 (m, 2H), 7.80 (d, J = 1.4 Hz, 1H), 7.60 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.8 Hz, 2H), 7.40 (dd, J = 8.8, 1.8 Hz, 1H), 7.32 (d, J = 8.8 Hz, 1H), 7.14 (d, J = 1.8 Hz, 1H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 141.1, 135.7, 135.3, 133.8, 129.5, 129.1, 128.6, 127.3, 125.0, 114.8, 113.9, 108.3;

MS (ESI): *m/z* (%) 358 [(M+Na)$^+$, 100].

2-Tosyl-1*H*-indole (3j)

White solid; mp: 192–193°C (Lit.[2] 194–195°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.59 (br s, 1H), 7.93 (dd, J = 6.8, 1.6 Hz, 2H), 7.66 (d, J = 0.6 Hz, 1H), 7.44 (dd, J = 8.4, 0.8 Hz, 1H), 7.35-7.25 (m, 3H), 7.22 (dd, J = 2.1, 0.8 Hz, 1H), 7.20-7.14 (m, 1H), 2.38 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 144.5, 138.5, 137.2, 134.4, 130.0, 127.3, 127.0, 125.9, 122.6, 121.5, 112.4, 108.8, 21.6;

MS (ESI): *m/z* (%) 294 [(M+Na)$^+$, 56], 117 (100).

5-Methyl-2-tosyl-1*H*-indole (3k)

White solid; mp: 154–155°C (Lit.[3] 158–160°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.52 (br s, 1H), 7.92 (d, J = 8.4 Hz, 2H), 7.44 (s, 1H), 7.33 (d, J = 8.5 Hz, 1H), 7.28-7.24 (m, 2H), 7.14 (dd, J = 8.7, 1.3 Hz, 2H), 2.43 (s, 3H), 2.38 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 144.4, 138.7, 135.6, 134.2, 130.9, 129.9, 127.9, 127.32, 127.25, 121.8, 112.0, 108.4, 21.5, 21.4;

MS (ESI): *m/z* (%) 308 [(M+Na)$^+$, 89], 131 (100).

5-Methoxy-2-tosyl-1*H*-indole (3l)

White solid; mp: 163–164°C (Lit.[2] 159–160°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.62 (br s, 1H), 7.91 (d, J = 8.4 Hz, 2H), 7.33-7.23 (m, 3H), 7.14-7.11 (m, 1H), 7.04 (d, J = 2.4 Hz, 1H), 6.98 (dd, J = 9.0, 2.5 Hz, 1H), 3.83 (s, 3H), 2.37 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 155.2, 144.4, 138.7, 134.6, 132.4, 130.0, 127.6, 127.3, 117.6, 113.3, 108.4, 102.6, 55.7, 21.6;

MS (ESI): *m/z* (%) 324 [(M+Na)$^+$, 95], 147 (100).

5-Fluoro-2-tosyl-1*H*-indole (3m)

White solid; mp: 137–138°C (Lit.[2] 142–143°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.75 (br s, 1H), 7.92 (d, J = 8.4 Hz, 2H), 7.37 (dd, J = 9.0, 4.3 Hz, 1H), 7.31-7.26 (m, 3H), 7.14 (d, J = 1.6 Hz, 1H), 7.07 (td, J = 9.1, 2.5 Hz, 1H), 2.39 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 159.4, 157.5, 144.8, 138.3, 136.1, 133.6, 130.1, 127.4, 115.2 (d, J = 26.8 Hz), 113.4 (d, J = 9.6 Hz), 108.5 (d, J = 5.3 Hz), 106.9 (d, J = 23.5 Hz), 21.6;

MS (ESI): *m/z* (%) 312 [(M+Na)$^+$, 75], 338 (100).

5-Bromo-2-tosyl-1*H*-indole (3n)

Pale yellow solid; mp: 186–187°C (Lit.[2] 193–194°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.18 (br s, 1H), 7.89 (d, J = 8.3 Hz, 2H), 7.80 (d, J = 1.6 Hz, 1H), 7.41 (d, J = 1.9 Hz, 1H), 7.34-7.26 (m, 3H), 7.09 (d, J = 1.7 Hz, 1H), 2.42 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 144.9, 138.1, 135.8, 135.5, 130.1, 129.0, 128.7, 127.4, 125.0, 114.7, 113.8, 107.9, 21.6;

MS (ESI): *m/z* (%) 372 [(M+Na)$^+$, 51], 338 (100).

2-((4-Chlorophenyl)sulfonyl)-1*H*-indole (3o)

White solid; mp: 165–166°C (Lit.[3] 167–168°C);

^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.59 (br s, 1H), 7.98-7.93 (m, 2H), 7.68 (d, J = 8.1 Hz, 1H), 7.46-7.42 (m, 3H), 7.37-7.31 (m, 1H), 7.25 (d, J = 1.5 Hz, 1H), 7.22-7.17 (m, 1H);

^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 140.1, 139.9, 137.5, 133.3, 129.7, 128.7, 126.9, 126.3, 122.7, 121.7, 112.5, 109.6;

MS (ESI): *m/z* (%) 314 [(M+Na)$^+$, 100].

2-((4-Chlorophenyl)sulfonyl)-5-methyl-1*H*-indole (3p)

Pale yellow solid; mp: 142–143°C (Lit.[2] 152–153°C);

^{1}H NMR (CDCl$_3$, 500 MHz, ppm): δ 9.37 (s, 1H), 7.94 (d, J = 8.7 Hz, 2H), 7.45-7.42 (m, 3H), 7.32 (d, J = 8.6 Hz, 1H), 7.17 (dd, J = 8.6, 1.5 Hz, 1H), 7.15 (dd, J = 2.1, 0.8 Hz, 1H), 2.43 (s, 3H);

^{13}C NMR (CDCl$_3$, 125 MHz, ppm): δ 140.1, 140.0, 135.9, 133.2, 131.2, 129.6, 128.6, 128.3, 127.3, 121.8, 112.1, 109.1, 21.4;

MS (ESI): *m/z* (%) 328 [(M+Na)$^+$, 57], 131 (100).

2-((4-Chlorophenyl)sulfonyl)-5-methoxy-1*H*-indole (3q)

White solid; mp: 124–125°C;

^{1}H NMR (CDCl$_3$, 500 MHz, ppm): δ 9.39 (br s, 1H), 7.93 (d, J = 8.7 Hz, 2H), 7.44 (d, J = 8.7 Hz, 2H), 7.32 (d, J = 9.0 Hz, 1H), 7.15-7.12 (m, 1H), 7.04 (d, J = 2.3 Hz, 1H), 7.01 (dd, J = 9.0, 2.4 Hz, 1H), 3.84 (s, 3H);

^{13}C NMR (CDCl$_3$, 125 MHz, ppm): δ 155.2, 140.1, 140.0, 133.5, 132.7, 129.6, 128.6, 127.5, 118.1, 113.4, 109.1, 102.5, 55.7;

MS (ESI): *m/z* (%) 344 [(M+Na)$^+$, 56], 147 (100);

HRMS (ESI) *m/z*: (M+Na)$^+$ calcd for $C_{15}H_{12}ClNO_3S$ 344.0125, found 344.0112.

2-((4-Chlorophenyl)sulfonyl)-5-fluoro-1*H*-indole (3r)

White solid; mp: 148–149°C;

^{1}H NMR (CDCl$_3$, 500 MHz, ppm): δ 9.36 (br s, 1H), 7.95 (d, J = 8.8 Hz, 2H), 7.49 (d, J = 8.8 Hz, 2H), 7.38 (dd, J = 9.0, 4.3 Hz, 1H), 7.31 (dd, J = 9.0, 2.5 Hz, 1H), 7.16 (dd, J = 2.1, 0.8 Hz, 1H), 7.13 (dd, J = 9.1, 2.5 Hz, 1H);

^{13}C NMR (CDCl$_3$, 125 MHz, ppm): δ 159.4, 157.5, 140.4, 139.7, 135.1, 133.9, 129.8, 128.8, 127.3 (d, J = 10.5 Hz), 115.6 (d, J = 26.9 Hz), 113.5 (d, J = 9.4 Hz), 109.2 (d, J = 5.3 Hz), 107.0 (d, J = 23.6 Hz);

MS (ESI): *m/z* (%) 332 [(M+Na)$^+$, 56], 131 (100);

HRMS (ESI) *m/z*: (M+Na)$^+$ calcd for $C_{14}H_9ClFNO_2SNa$ 331.9924, found 331.9903.

5-Bromo-2-((4-chlorophenyl)sulfonyl)-1*H*-indole (3s)

Brown solid; mp: 182–183°C (Lit.[2] 184–185°C);

^{1}H NMR (CDCl$_3$, 500 MHz, ppm): δ 9.13 (br s, 1H), 7.94 (d, J = 8.7 Hz, 2H), 7.82 (d, J = 1.8 Hz, 1H), 7.51 (d, J = 8.7 Hz, 1H), 7.45 (dd, J = 8.8, 1.9 Hz, 1H), 7.32 (d, J = 8.7 Hz, 1H), 7.13 (dd, J = 2.1, 0.8 Hz, 1H);

^{13}C NMR (CDCl$_3$, 125 MHz, ppm): δ 140.5, 139.6, 135.7, 134.9, 129.8, 129.6, 129.4, 128.8, 128.6, 126.2, 125.1, 123.8, 115.0, 113.8, 112.7, 108.6;

MS (ESI): *m/z* (%) 394 [(M+Na)$^+$, 43], 338 (100).

2-(Methylsulfonyl)-1*H*-indole (3t)

White solid; mp: 169–170°C (Lit.[2] 167–168°C);

^{1}H NMR (CDCl$_3$, 500 MHz, ppm): δ 9.46 (br s, 1H), 7.73 (dd, J = 8.1, 0.6 Hz, 1H), 7.51 (dd, J = 8.5, 0.8 Hz, 1H), 7.43-7.37 (m, 1H), 7.26-7.22 (m, 2H), 3.24 (s, 3H);

^{13}C NMR (CDCl$_3$, 125 MHz, ppm): δ 137.0, 133.3, 126.9, 126.3, 122.7, 121.7, 112.5, 108.6, 45.5;

MS (ESI): *m/z* (%) 218 [(M+Na)$^+$, 40], 117 (100).

5-Methyl-2-(methylsulfonyl)-1*H*-indole (3u)

White solid; mp: 154–155°C (Lit.[2] 159–160°C);

^{1}H NMR (CDCl$_3$, 500 MHz, ppm): δ 9.28 (br s, 1H), 7.50 (s, 1H), 7.39 (d, J = 8.5 Hz, 1H), 7.23

(dd, J = 8.5, 1.4 Hz, 1H), 7.15-7.13 (m, 1H), 3.22 (s, 3H), 2.47 (s, 3H);
^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 135.5, 133.1, 131.2, 128.2, 127.2, 121.9, 112.1, 108.2, 45.5, 21.4;
MS (ESI): *m/z* (%) 232 [$(M+Na)^+$, 39], 131 (100).

5-Methoxy-2-(methylsulfonyl)-1*H*-indole (3v)

White solid; mp: 176–177°C (Lit.[2] 177–178°C);
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.15 (br s, 1H), 7.38 (d, J = 8.9 Hz, 1H), 7.14 (dd, J = 2.1, 0.7 Hz, 1H), 7.10 (d, J = 2.4 Hz, 1H), 7.07 (dd, J = 8.9, 2.5 Hz, 1H), 3.87 (s, 3H), 3.22 (s, 3H);
^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 155.3, 133.5, 132.2, 127.5, 118.0, 113.3, 108.2, 102.6, 55.7, 45.6;
MS (ESI): *m/z* (%) 248 [$(M+Na)^+$, 33], 147 (100).

5-Fluoro-2-(methylsulfonyl)-1*H*-indole (3w)

Pale yellow solid; mp: 160–161°C (Lit.[2] 157–158°C);
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.51 (br s, 1H), 7.45 (dd, J = 9.1, 4.3 Hz, 1H), 7.36 (dd, J = 9.0, 2.5 Hz, 1H), 7.18 (dd, J = 2.0, 0.8 Hz, 1H), 7.16 (dd, J = 9.1, 2.5 Hz, 1H), 3.24 (s, 3H);
^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 159.5, 157.6, 134.8, 133.5, 127.2, 115.6 (d, J = 26.9 Hz), 113.6 (d, J = 9.6 Hz), 108.4 (d, J = 5.4 Hz), 107.1 (d, J = 23.5 Hz), 45.5;
MS (ESI): *m/z* (%) 236 [$(M+Na)^+$, 8], 338 (100).

5-Bromo-2-(methylsulfonyl)-1*H*-indole (3x)

Pale yellow solid; mp: 188–189°C (Lit.[2] 188–189°C);
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 9.29 (br s, 1H), 7.88 (d, J = 1.7 Hz, 1H), 7.51-7.47 (m, 1H), 7.38 (d, J = 8.8 Hz, 1H), 7.15 (d, J = 1.4 Hz, 1H), 3.24 (s, 3H);
^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 135.3, 134.6, 129.4, 128.6, 125.2, 115.0, 113.9, 107.8, 45.5;
MS (ESI): *m/z* (%) 296 [$(M+Na)^+$, 6], 338 (100).

References

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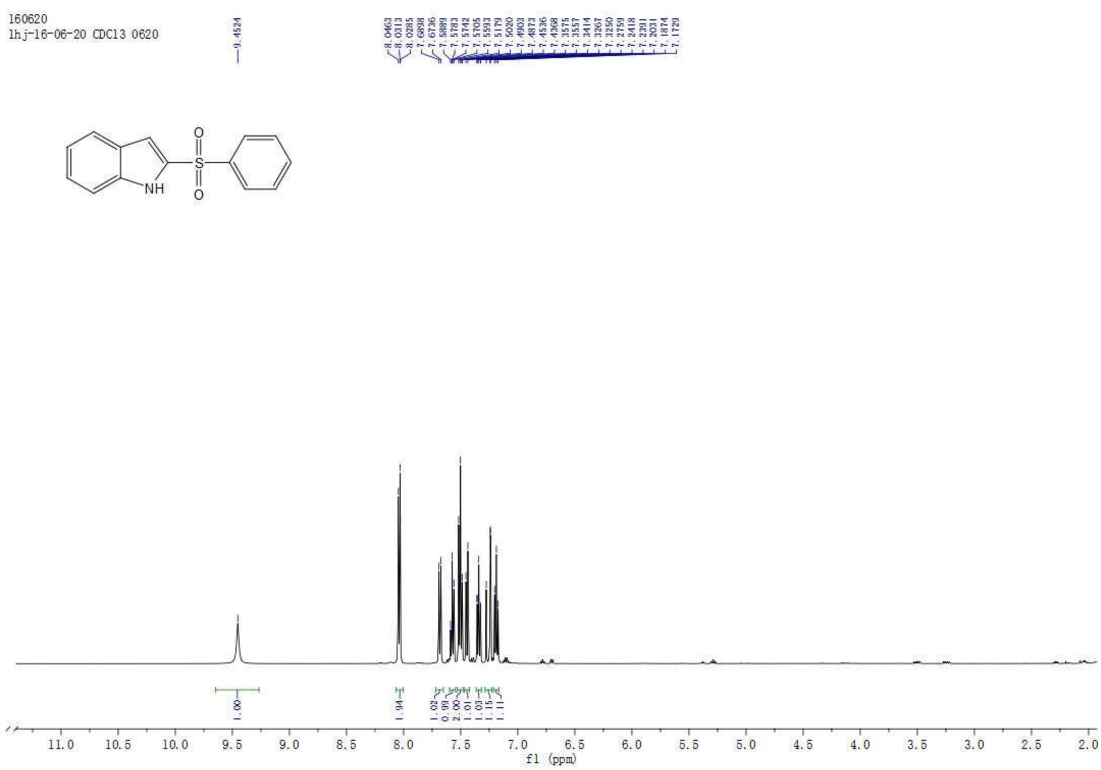
3a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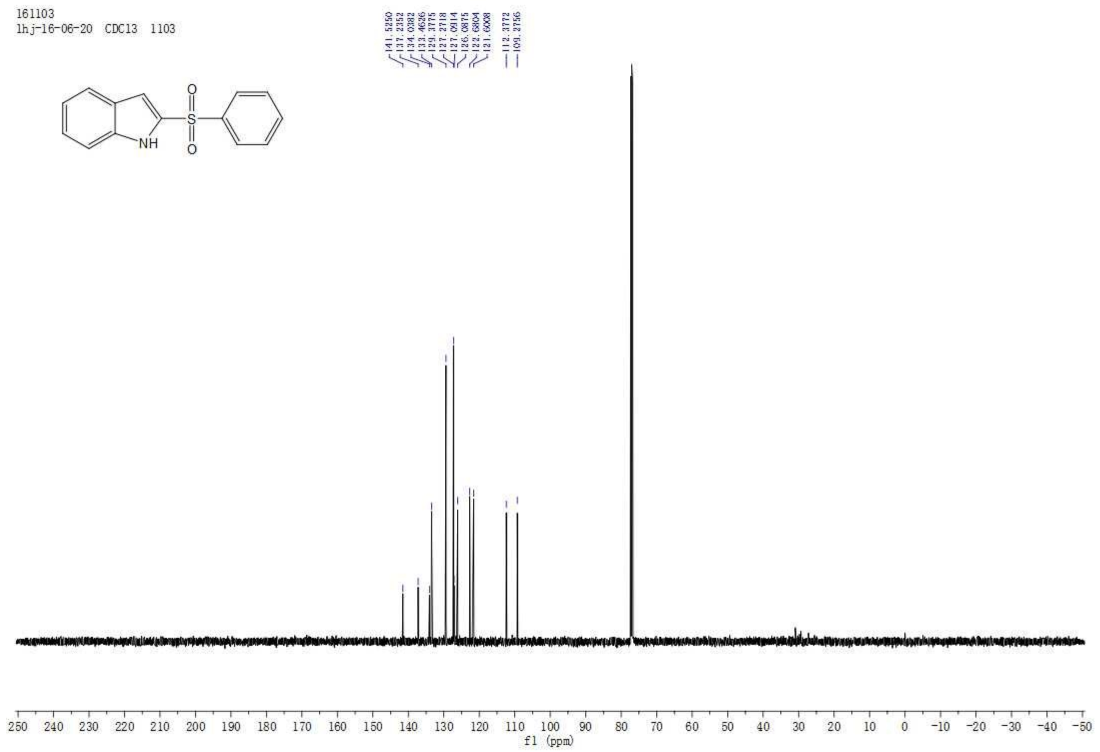
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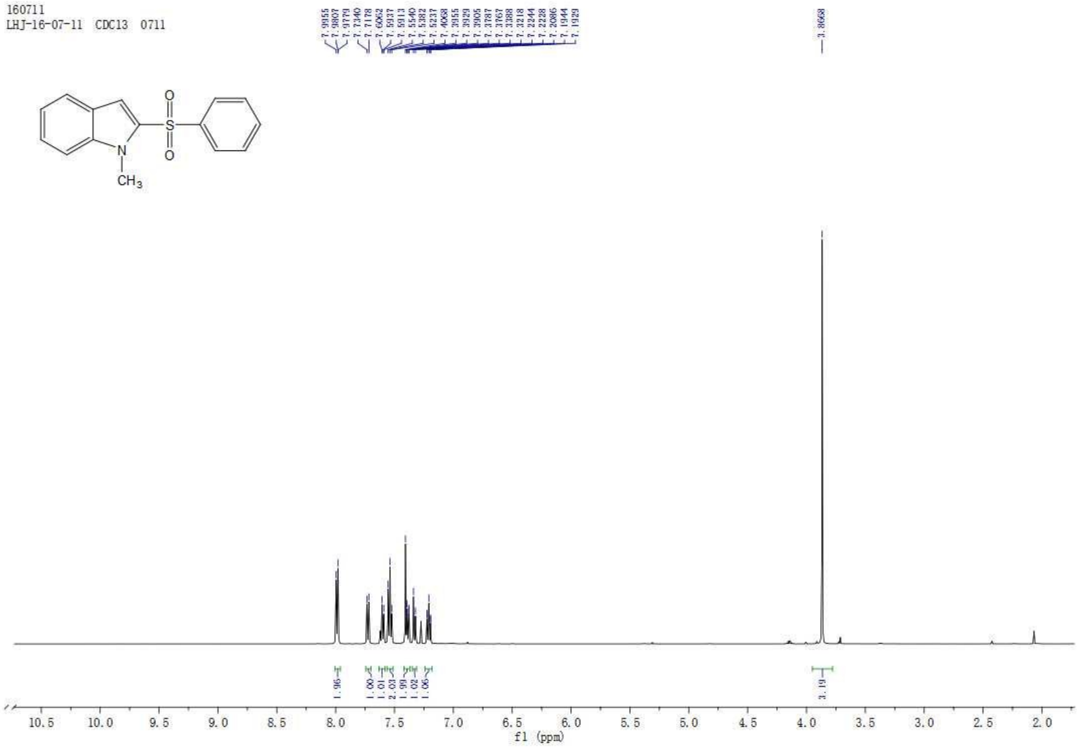
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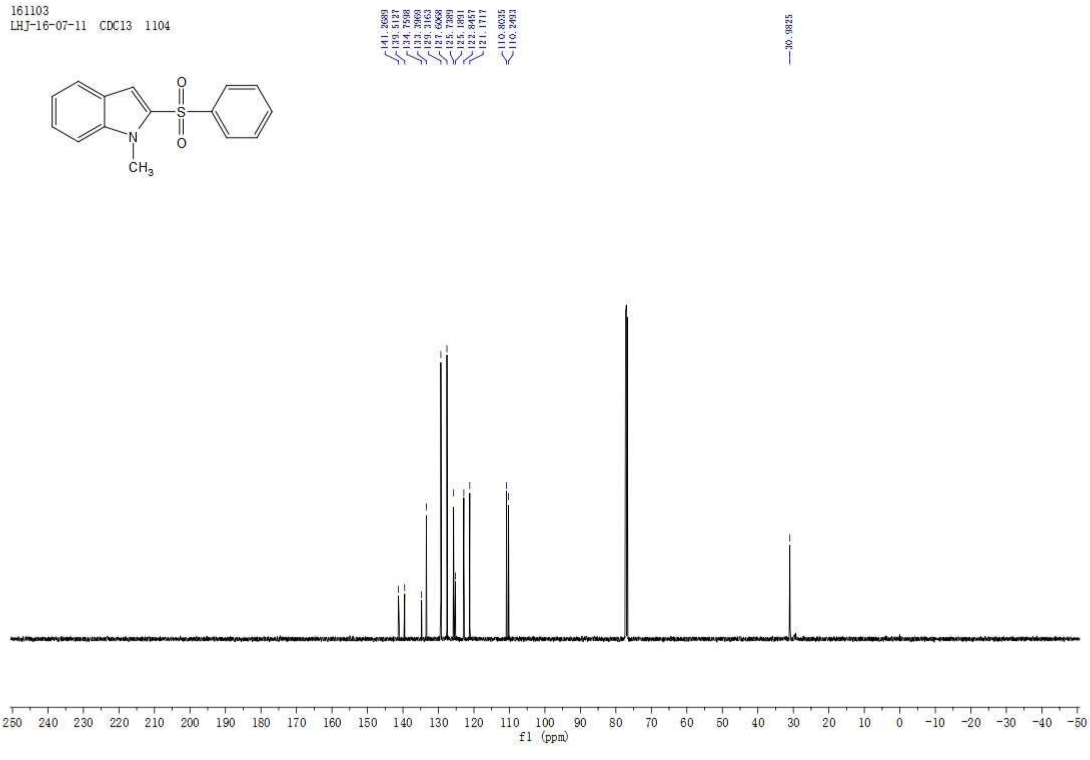
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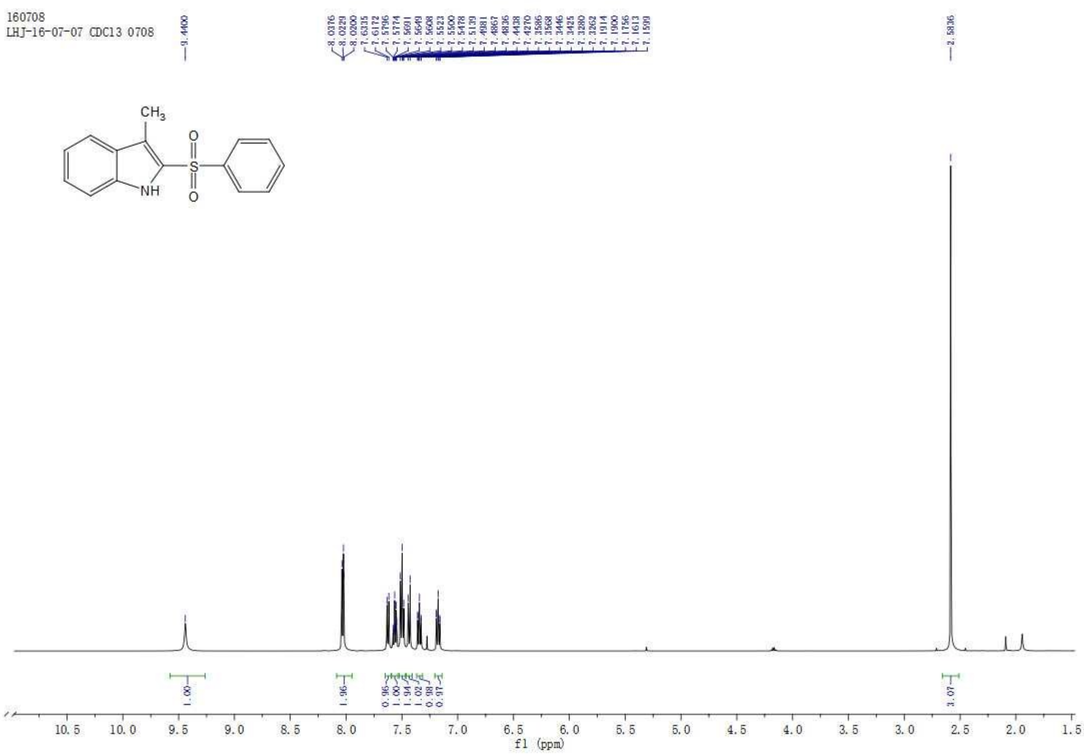

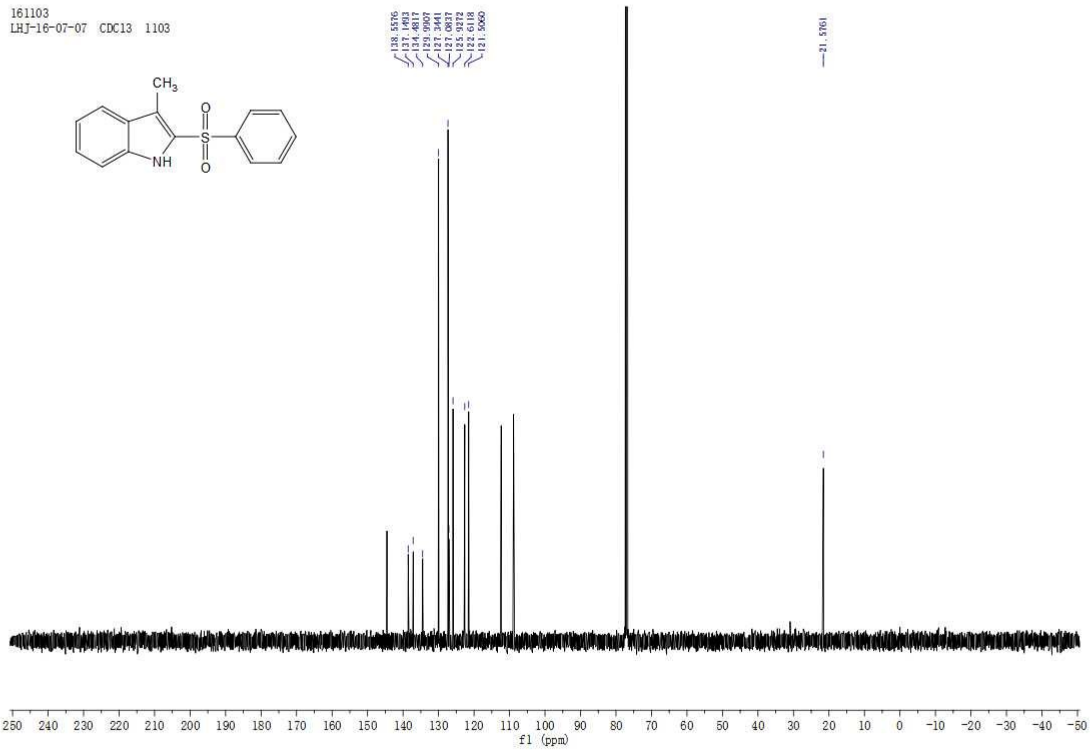

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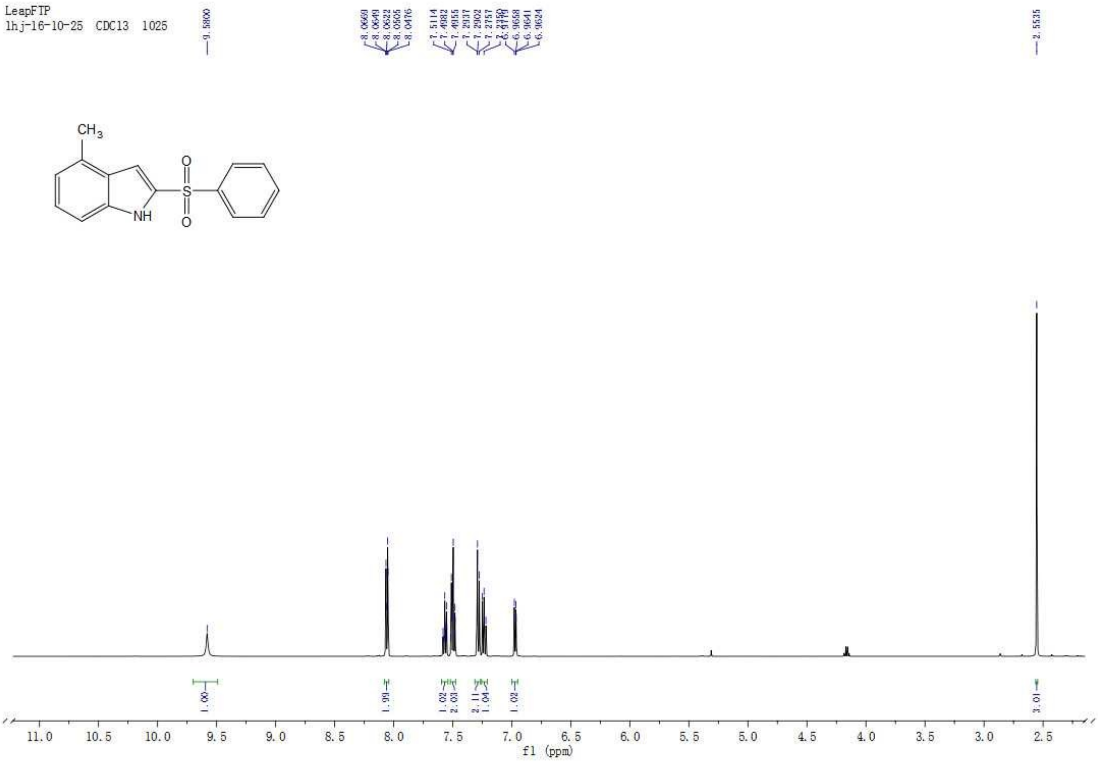

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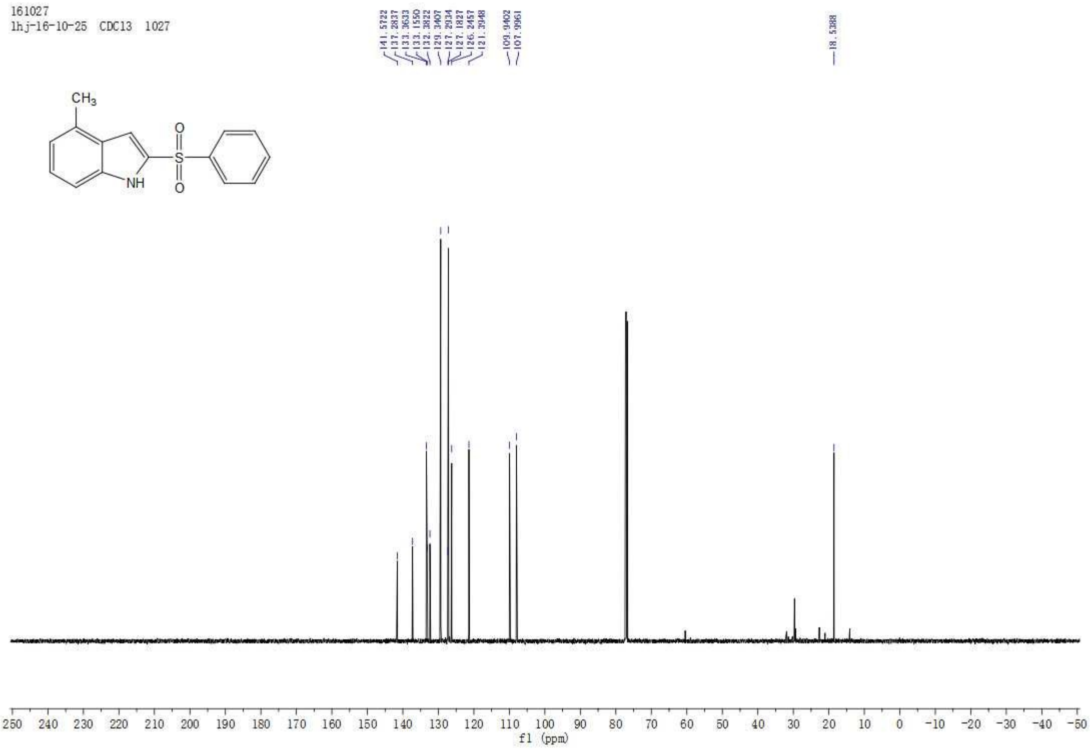
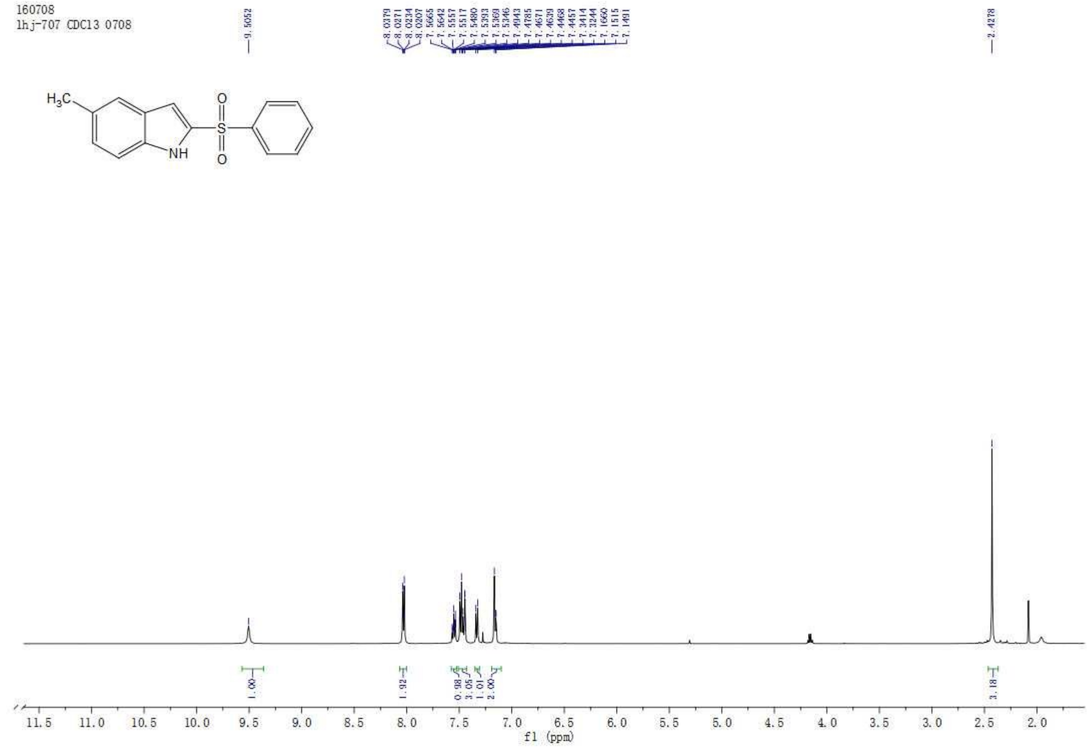
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
f1 (ppm)

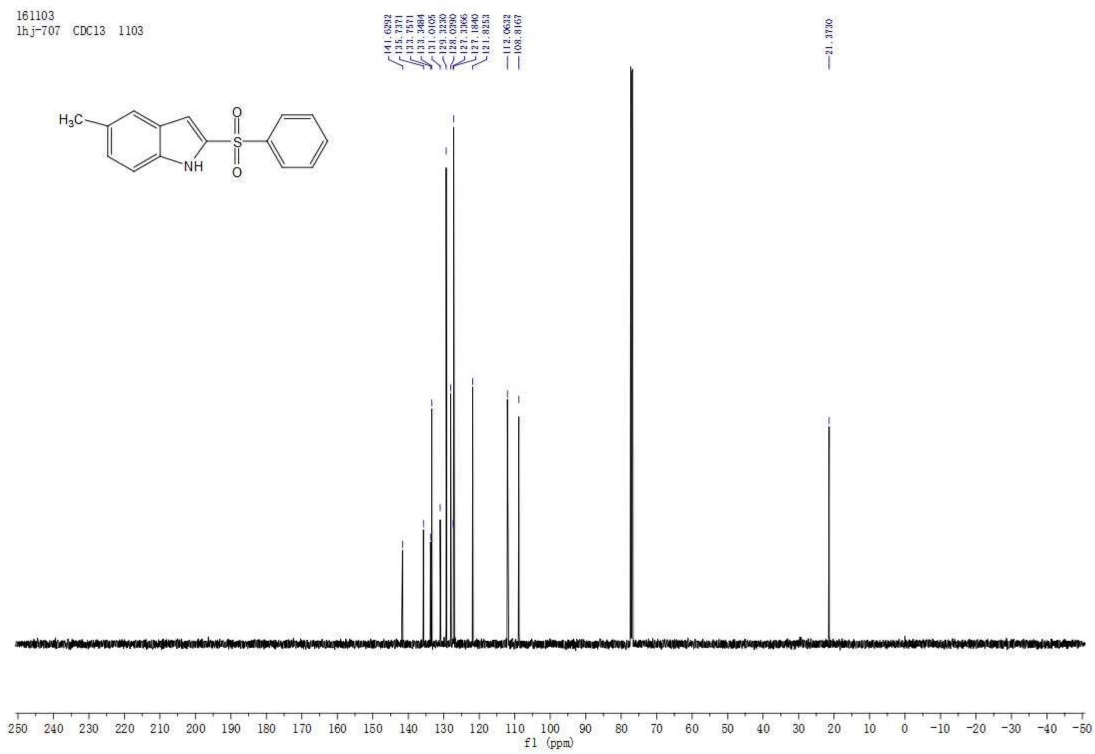


161027
1hj-16-10-25 CDCl3 1027
141.5722
137.2837
133.3623
133.1550
132.3822
129.3407
127.2934
127.1827
126.2457
121.3948
109.9402
107.9961
18.5388
CH3
O
S
O
NH
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50
f1 (ppm)

3e

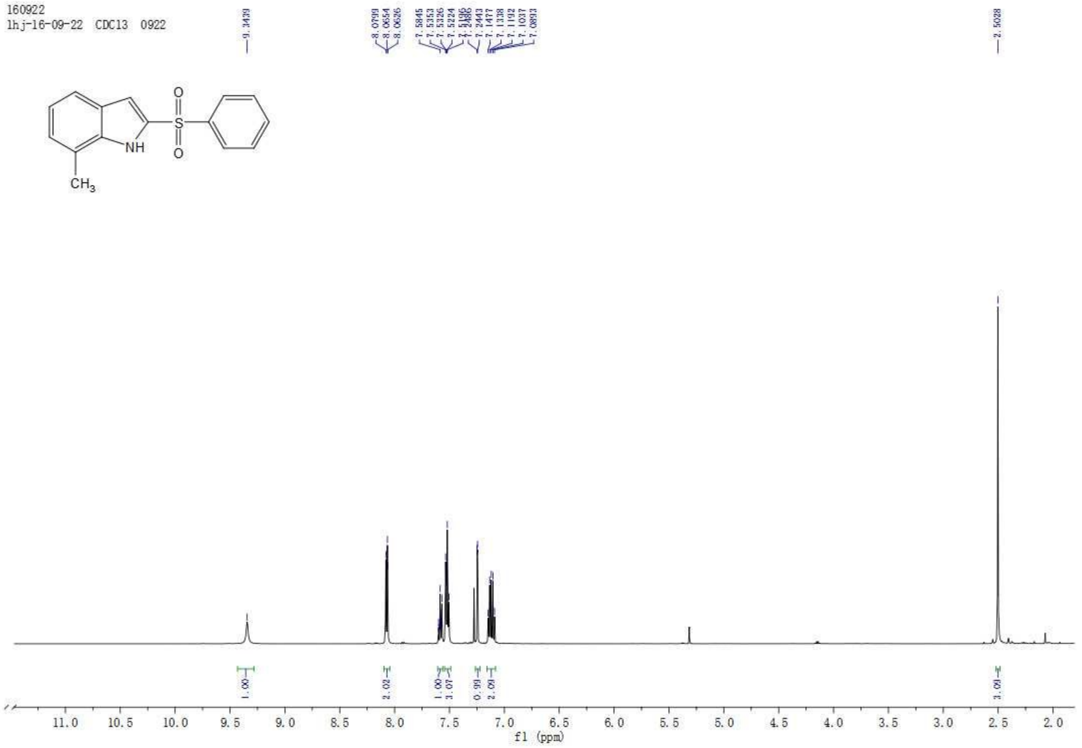


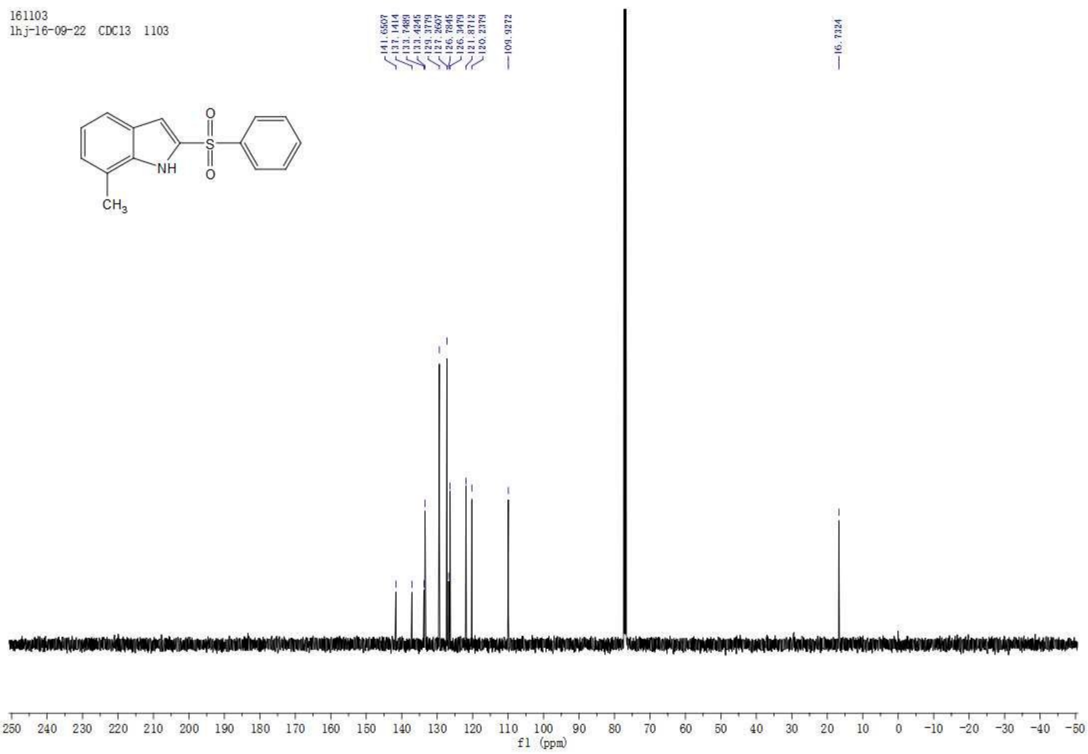
160708
1hj-707 CDCl3 0708
9.5052
8.0379
8.0271
8.0234
8.0207
7.5665
7.5642
7.5557
7.5517
7.5480
7.5393
7.5369
7.5346
7.4943
7.4785
7.4671
7.4639
7.4468
7.4457
7.3414
7.3244
7.1660
7.1515
7.1491
2.4278
H3C
NH
O
S
O
1.00
1.92
0.98
3.05
1.01
2.00
3.18
f1 (ppm)



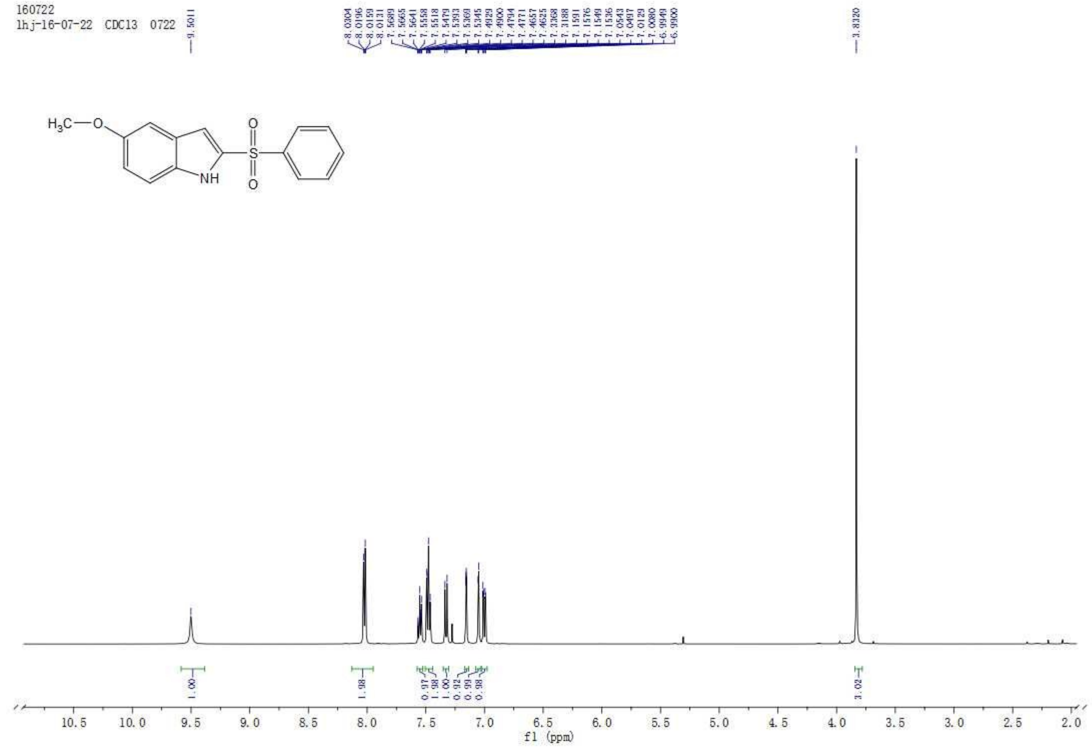
161103
1hj-707 CDCl3 1103
141.6292
135.7371
133.7571
133.3484
131.0105
129.3230
128.0390
127.1366
127.1840
121.8253
112.0632
108.8167
21.3730
H3C
NH
O
S
O
f1 (ppm)

3f

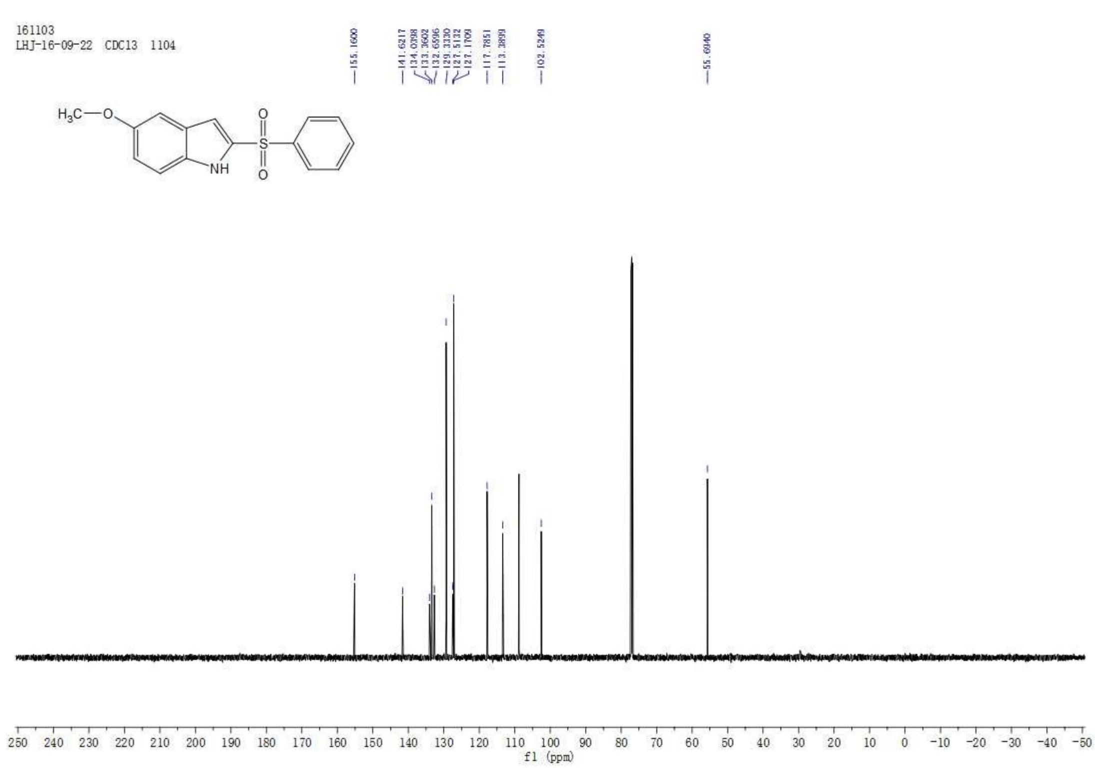




3g

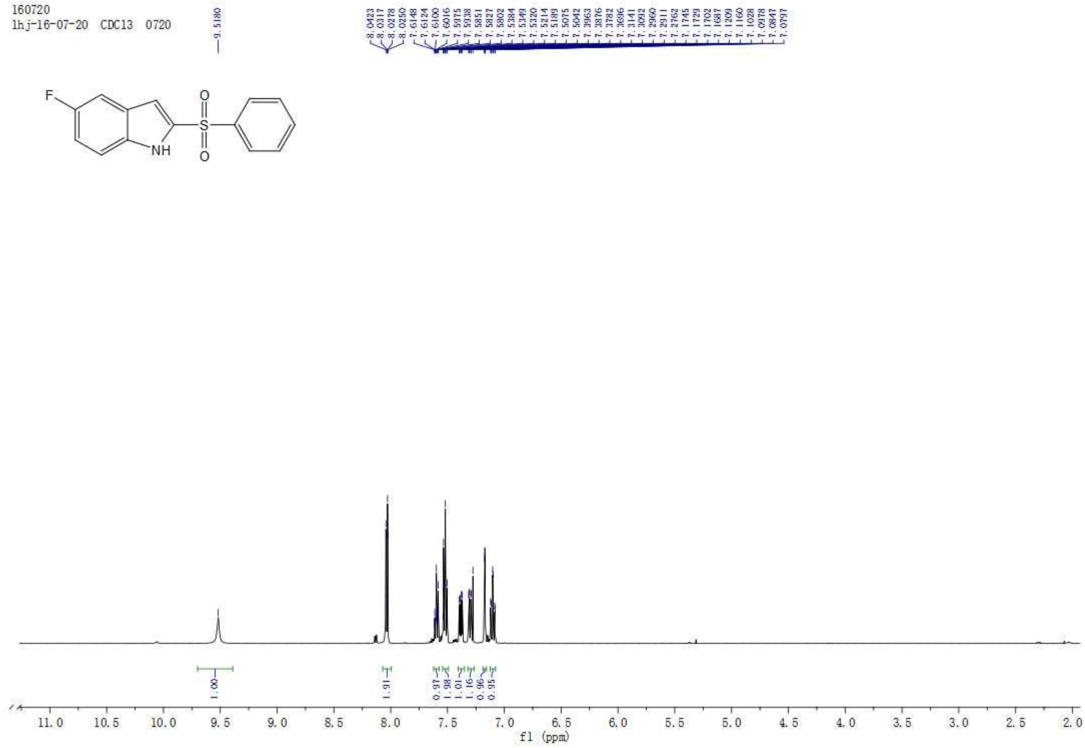


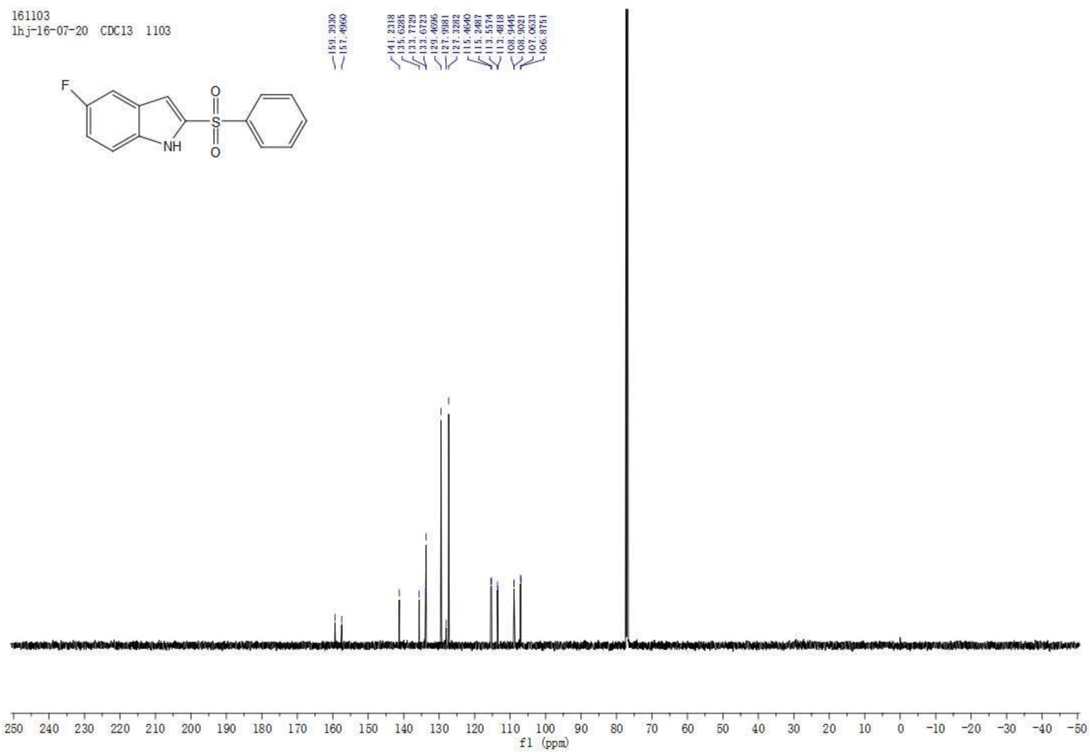
160722
lhj-16-07-22 CDCl3 0722
f1 (ppm)



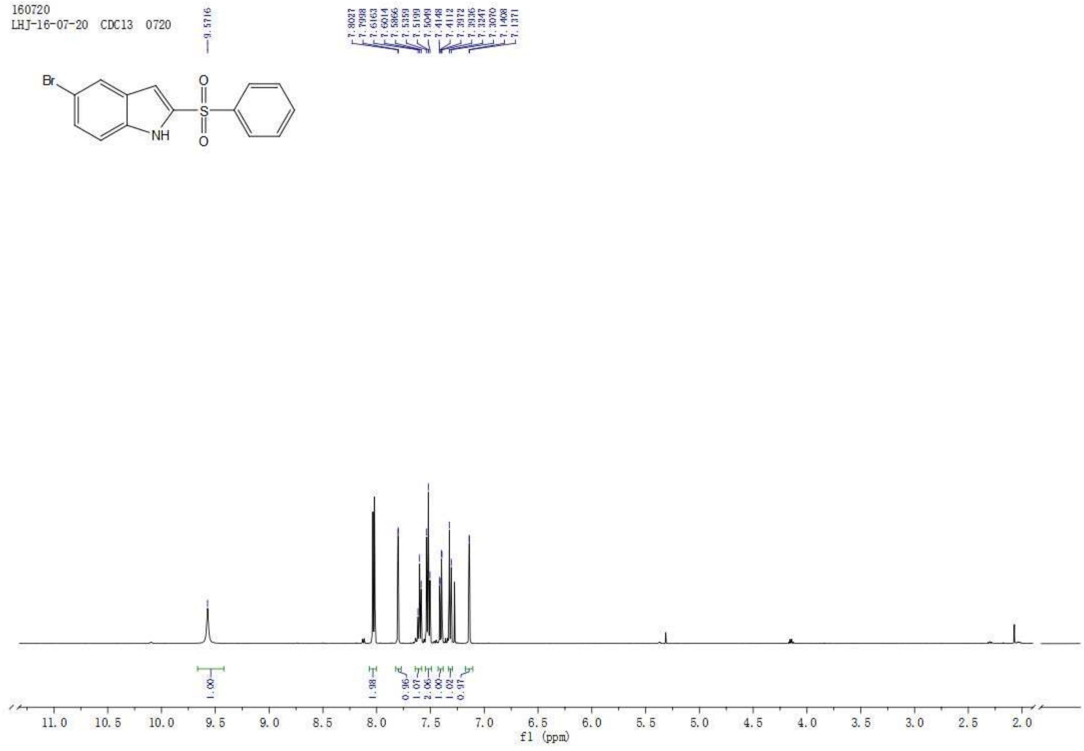
161103
LHJ-16-09-22 CDCl3 1104
f1 (ppm)

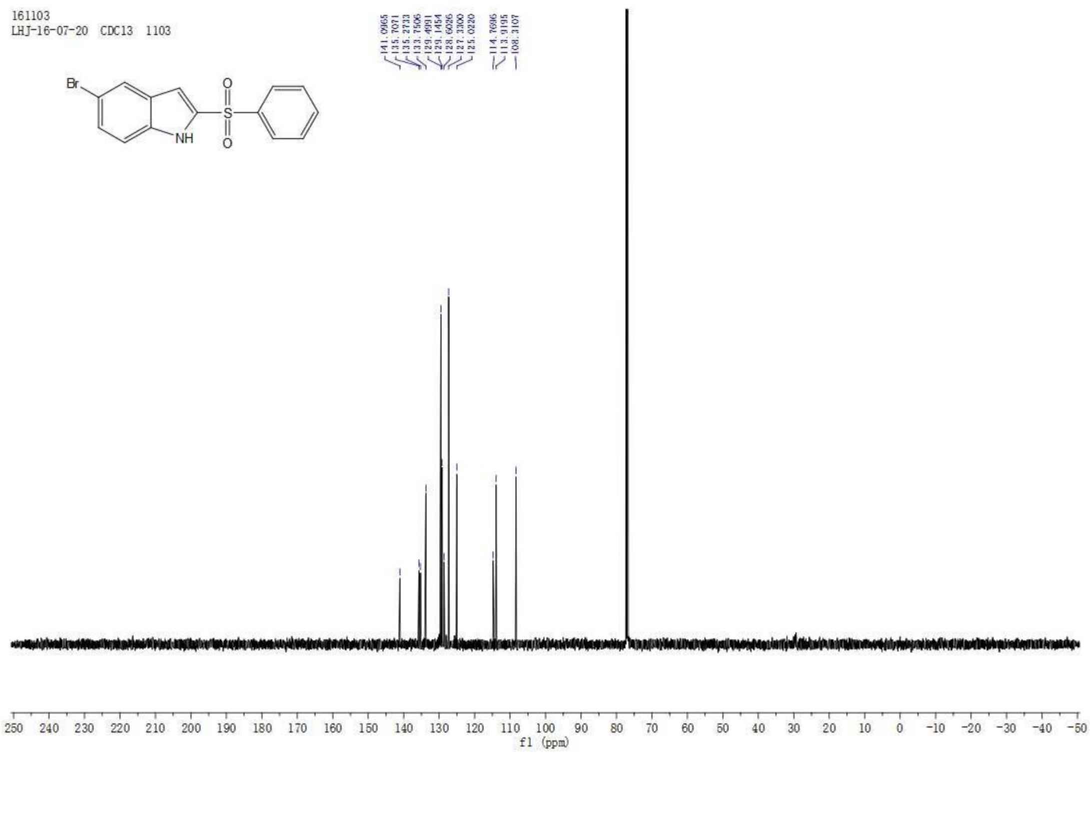
3h


160720
lhj-16-07-20 CDCl3 0720
F
O
S
O
NH
f1 (ppm)

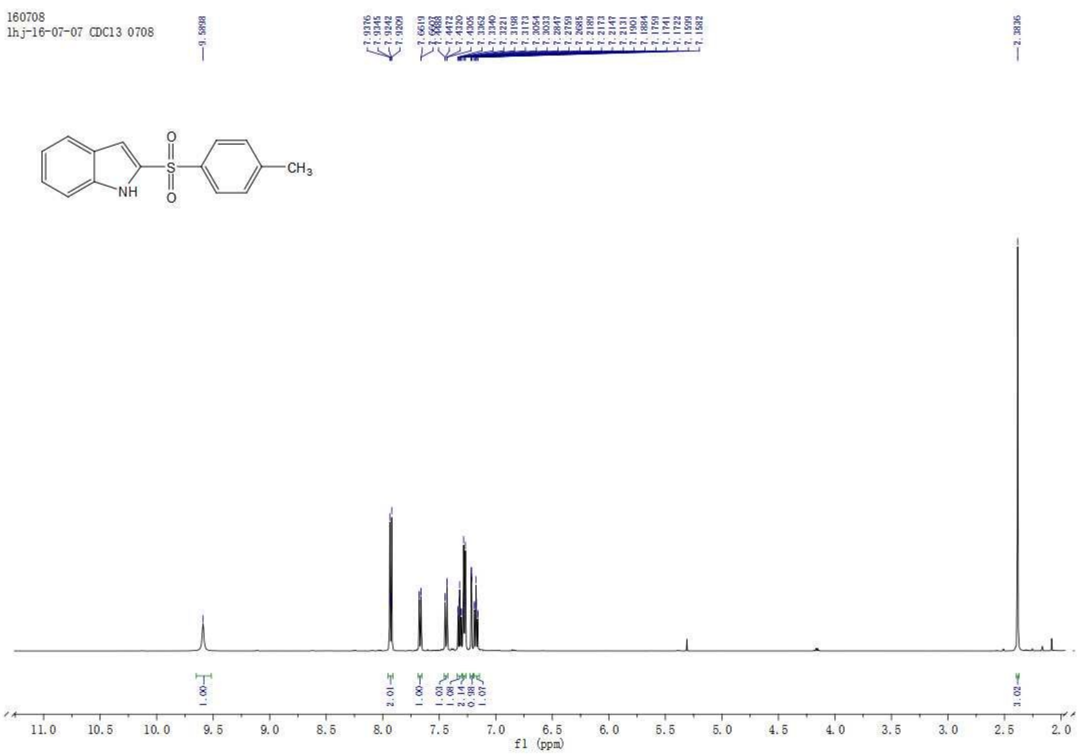

161103
lhj-16-07-20 CDCl3 1103
F
O
S
O
NH
f1 (ppm)

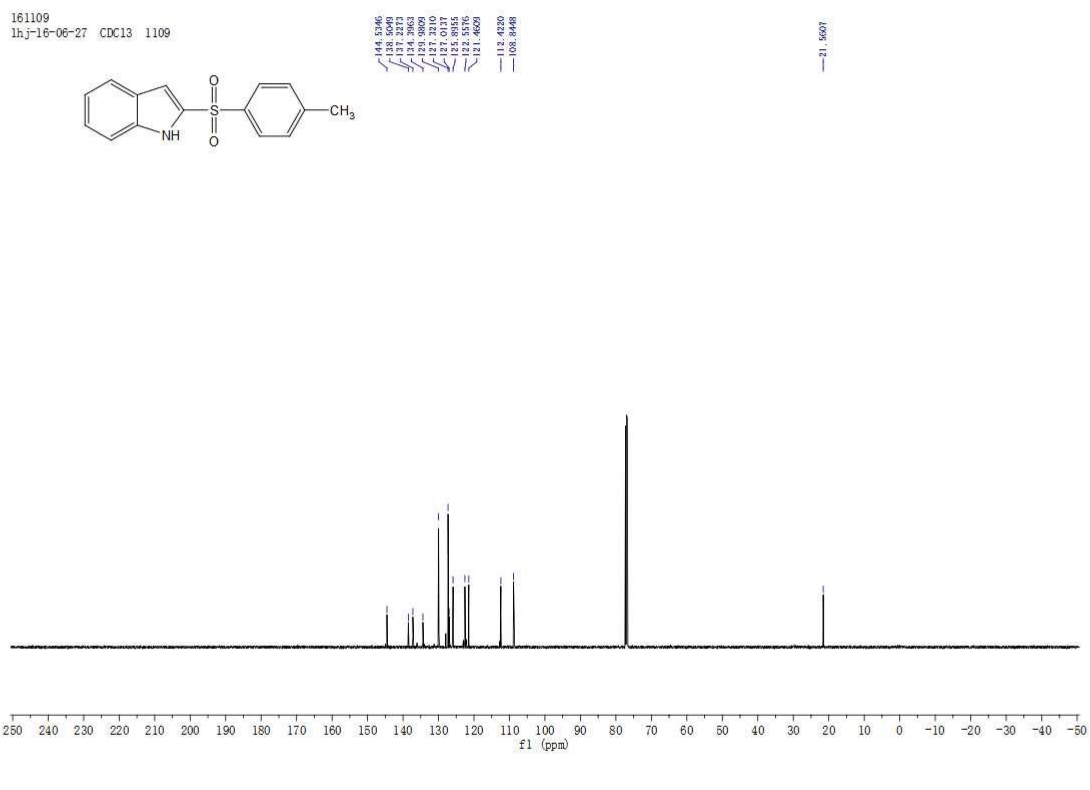
3i


160720
LHJ-16-07-20 CDCl3 0720
9.5716
7.8027
7.7988
7.6163
7.6014
7.5866
7.5359
7.5199
7.5049
7.4148
7.4112
7.3972
7.3936
7.3247
7.3070
7.1408
7.1371
Br
NH
O
S
O
1.00
1.98
0.96
1.07
2.06
1.00
1.02
0.97
f1 (ppm)

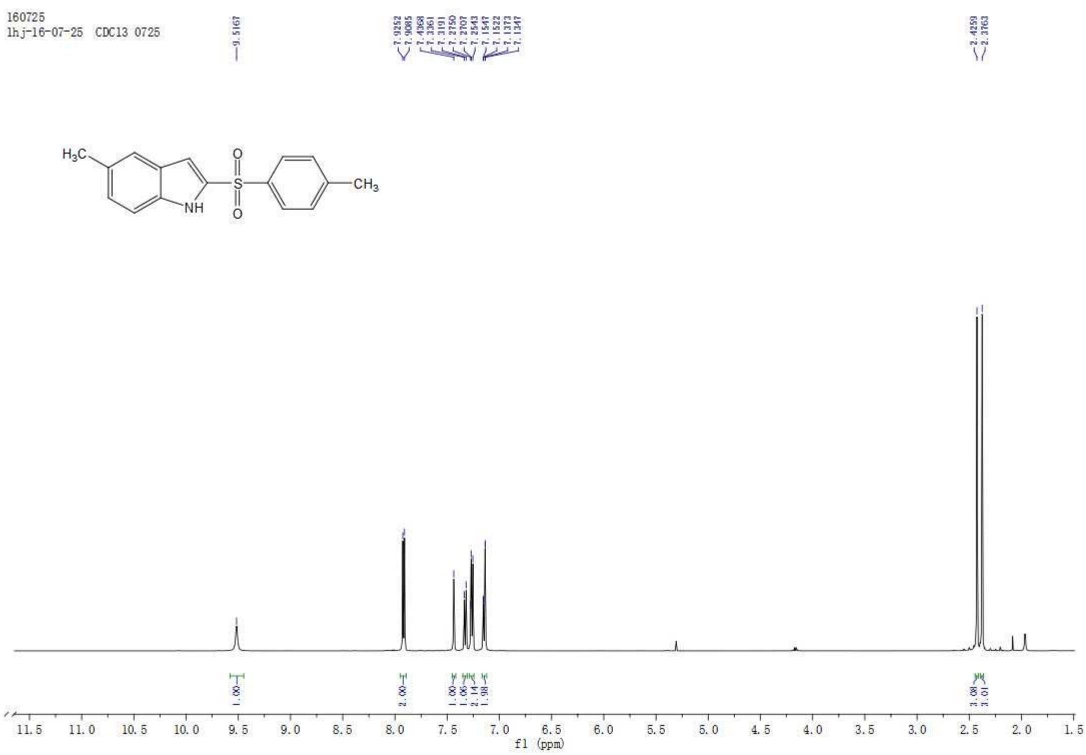

161103
LHJ-16-07-20 CDCl3 1103
141.0965
135.7071
135.2733
133.7506
129.4991
129.1454
128.6026
127.3300
125.0220
114.7696
113.9195
108.3107
Br
NH
O
S
O
f1 (ppm)

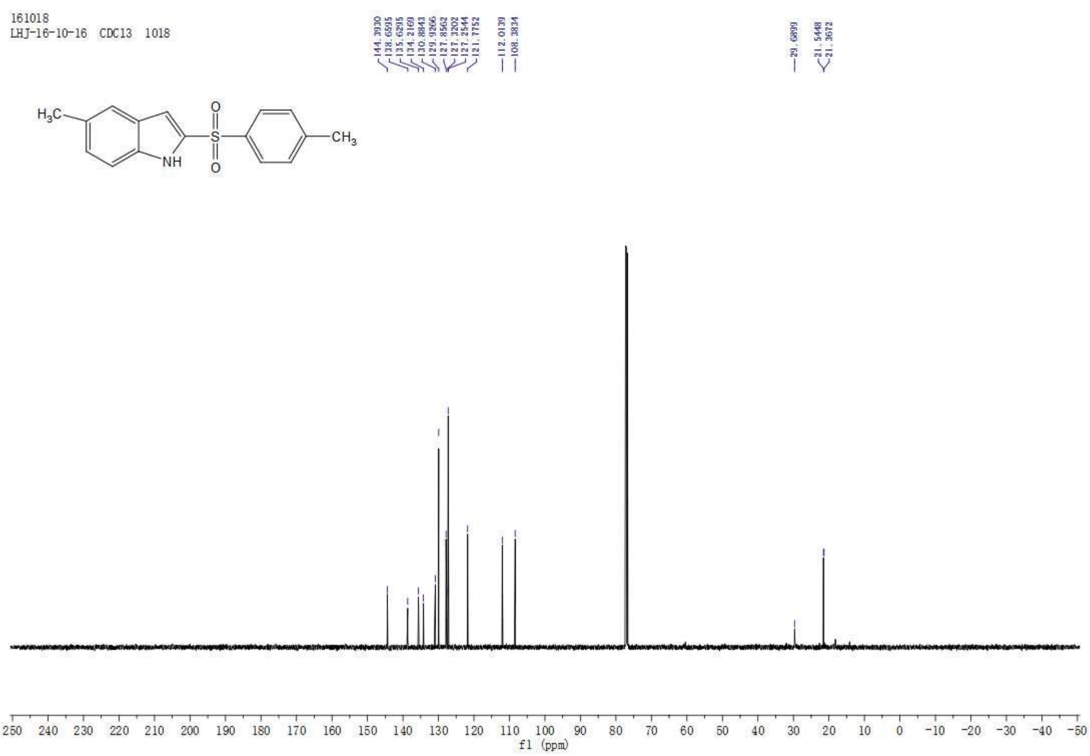
3j


160708
lhj-16-07-07 CDCl3 0708
NH
O
S
O
CH3
f1 (ppm)

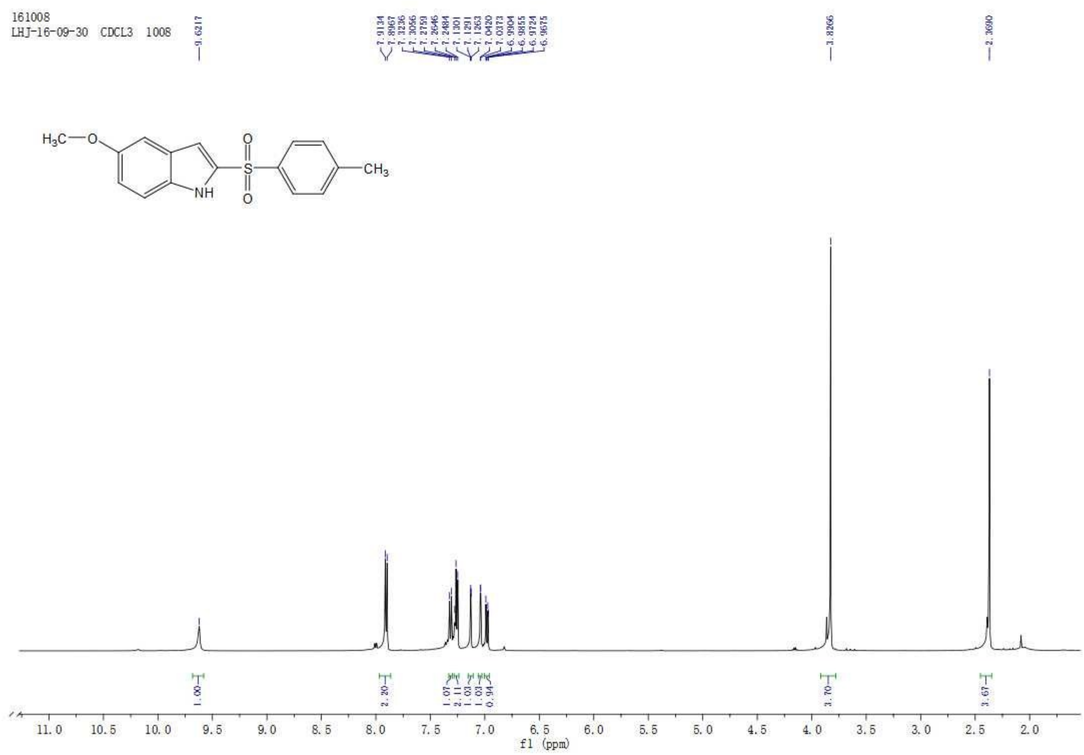

161109
lhj-16-06-27 CDCl3 1109
NH
O
S
O
CH3
f1 (ppm)

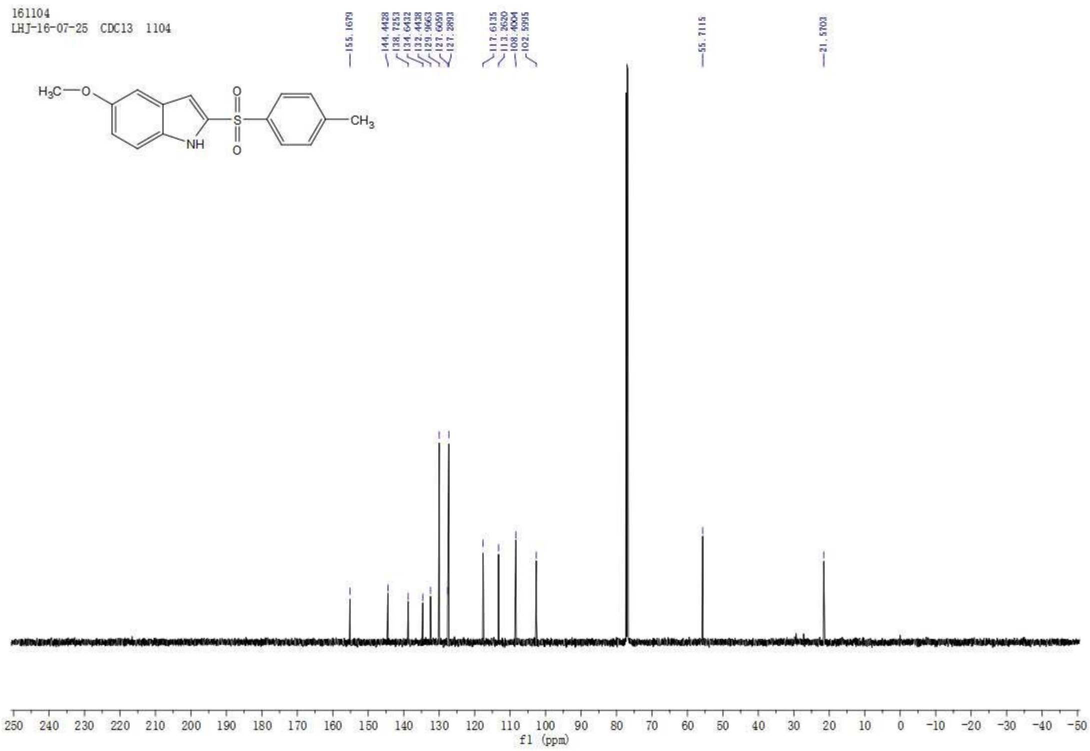
3k


160725
lhj-16-07-25 CDCl3 0725
f1 (ppm)

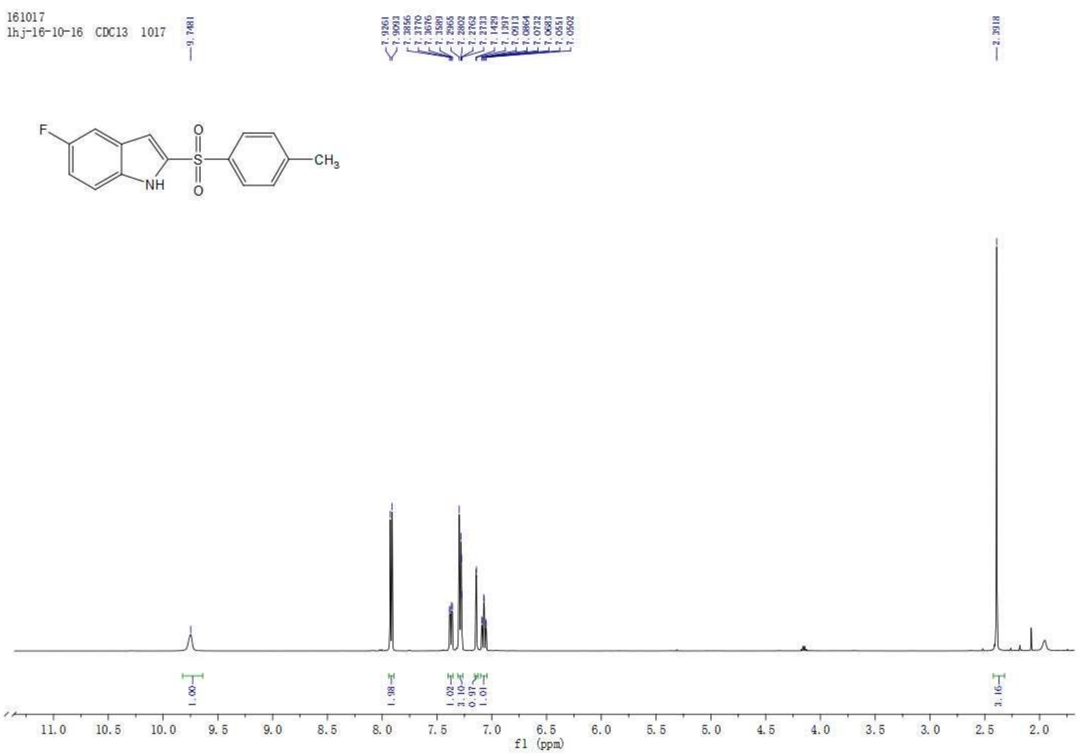

161018
LHJ-16-10-16 CDCl3 1018
f1 (ppm)

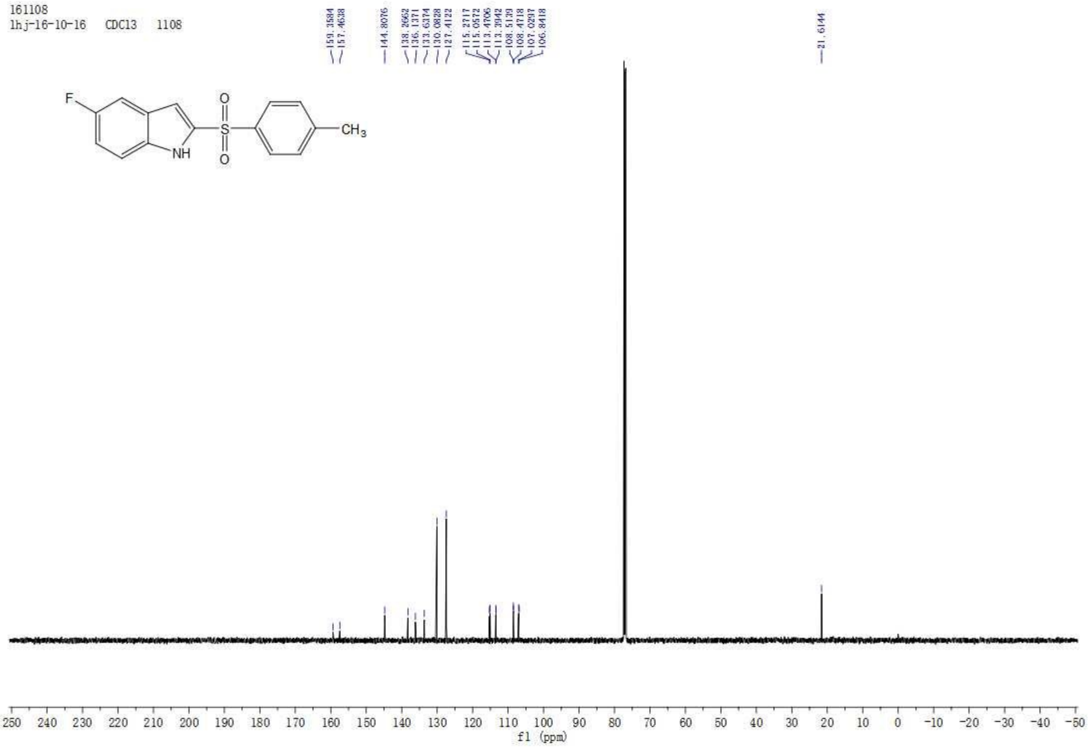
3l



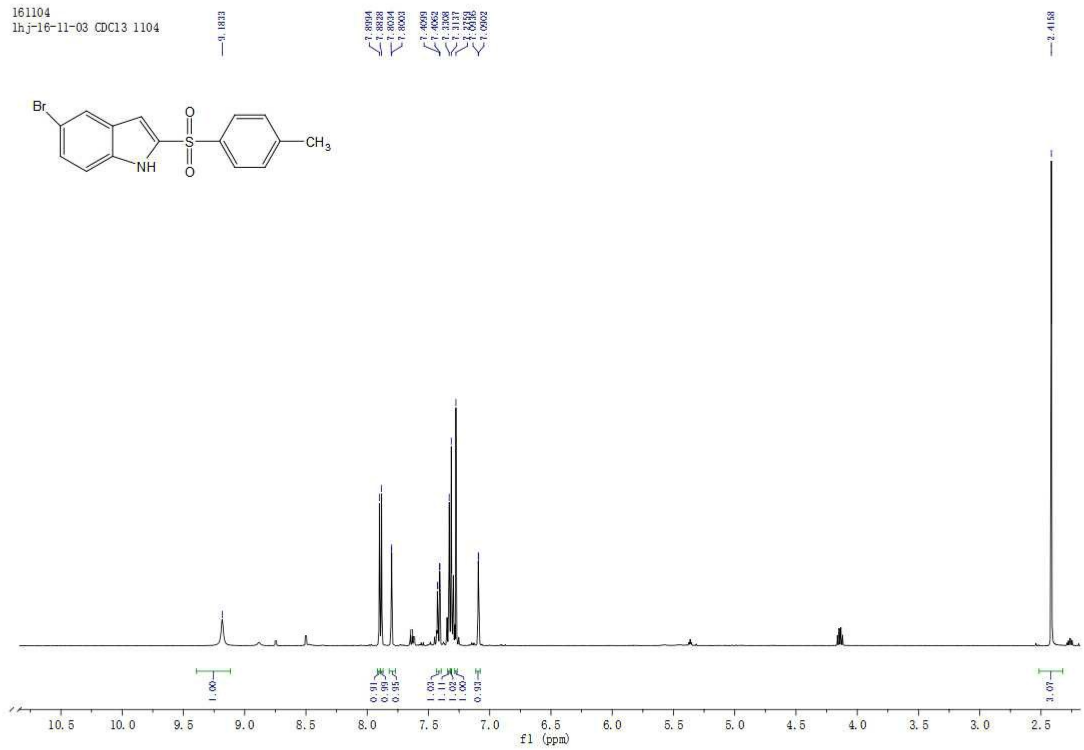


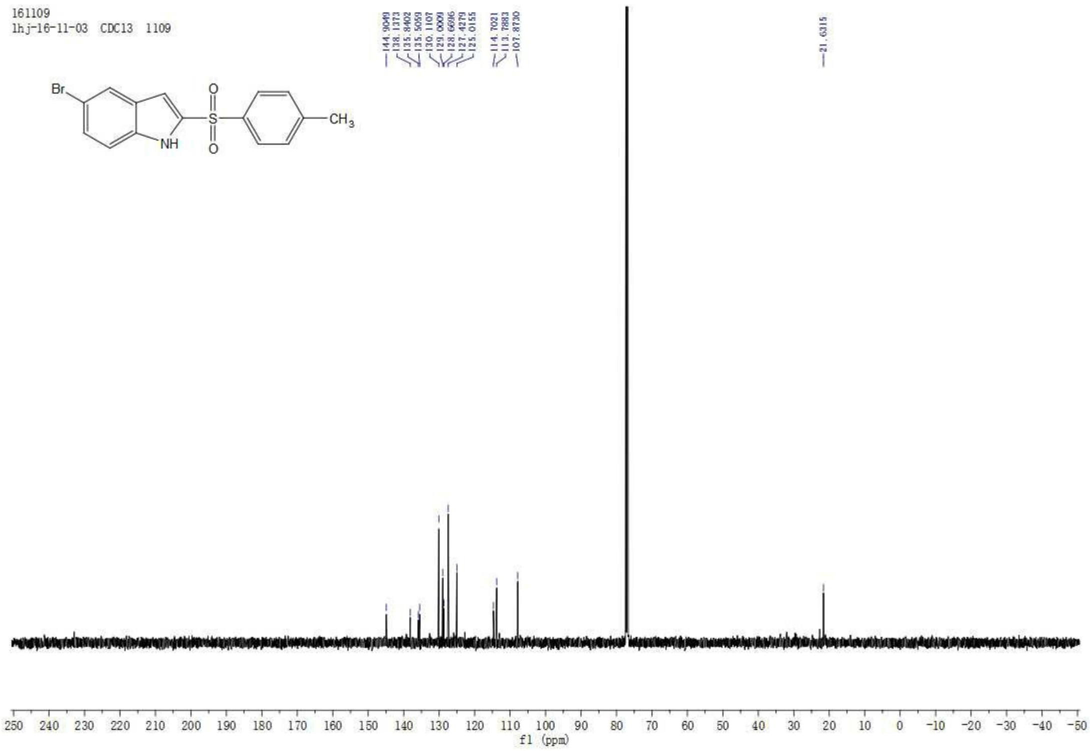
3m.


161017
lhj-16-10-16 CDCl3 1017
F
O
S
O
NH
CH3
11.0 10.5 10.0 9.5 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
f1 (ppm)

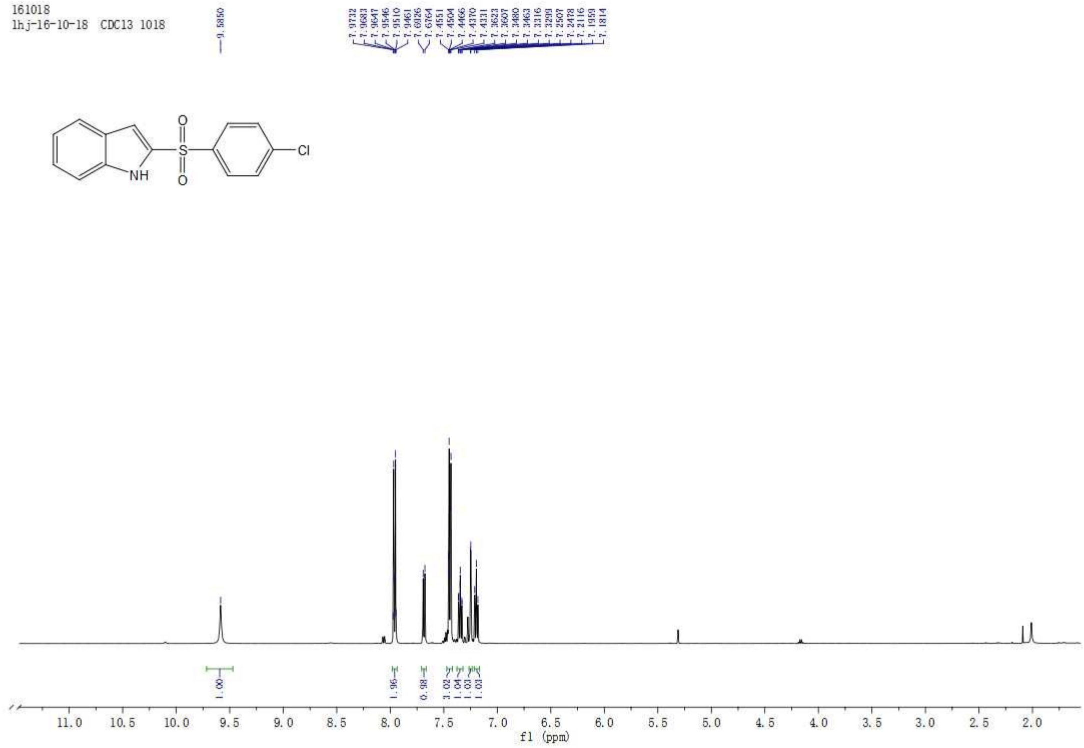

161108
lhj-16-10-16 CDCl3 1108
F
O
S
O
NH
CH3
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50
f1 (ppm)

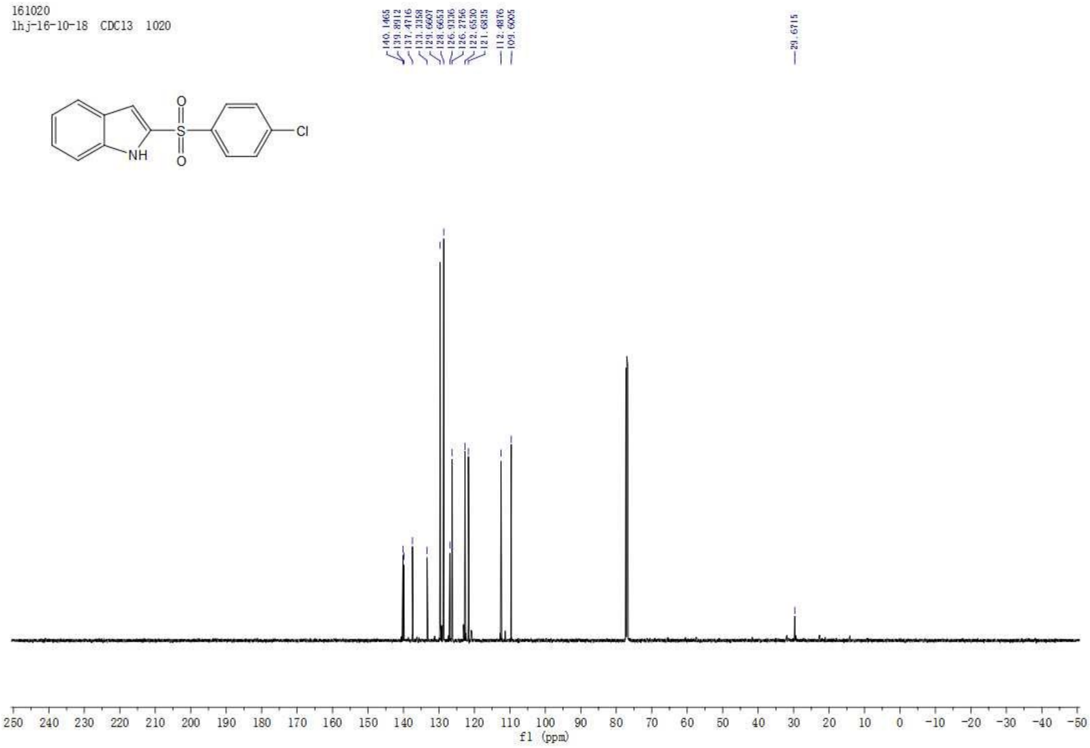
3n



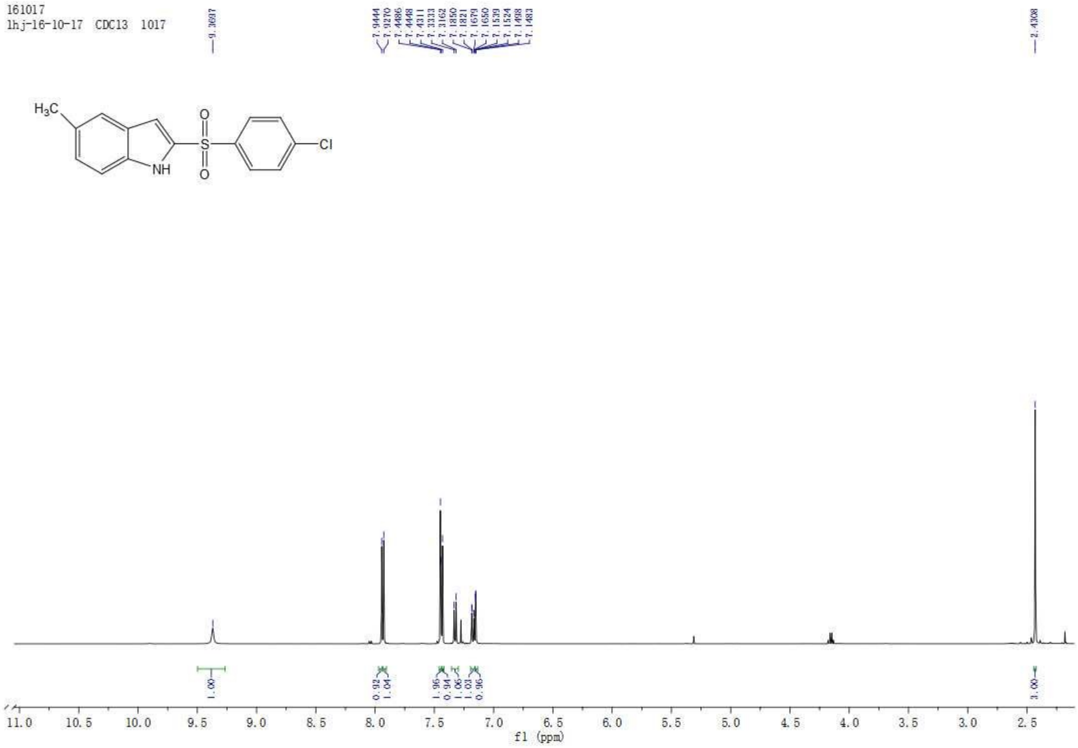


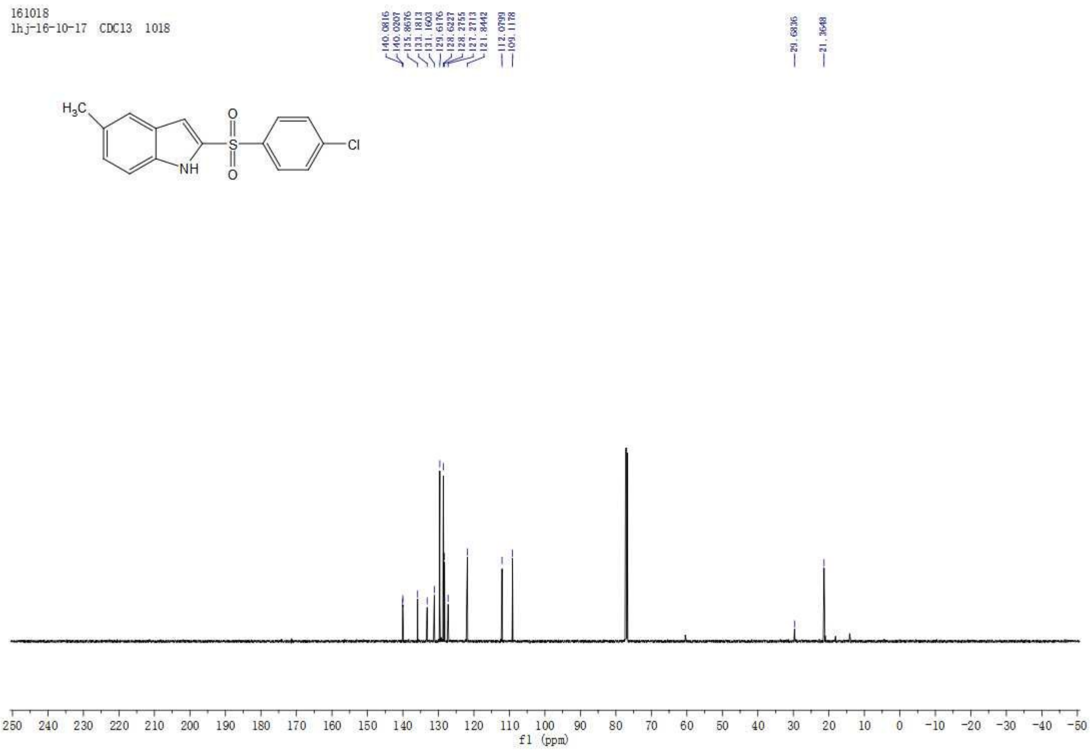
3o


161018
lhj-16-10-18 CDCl3 1018
f1 (ppm)

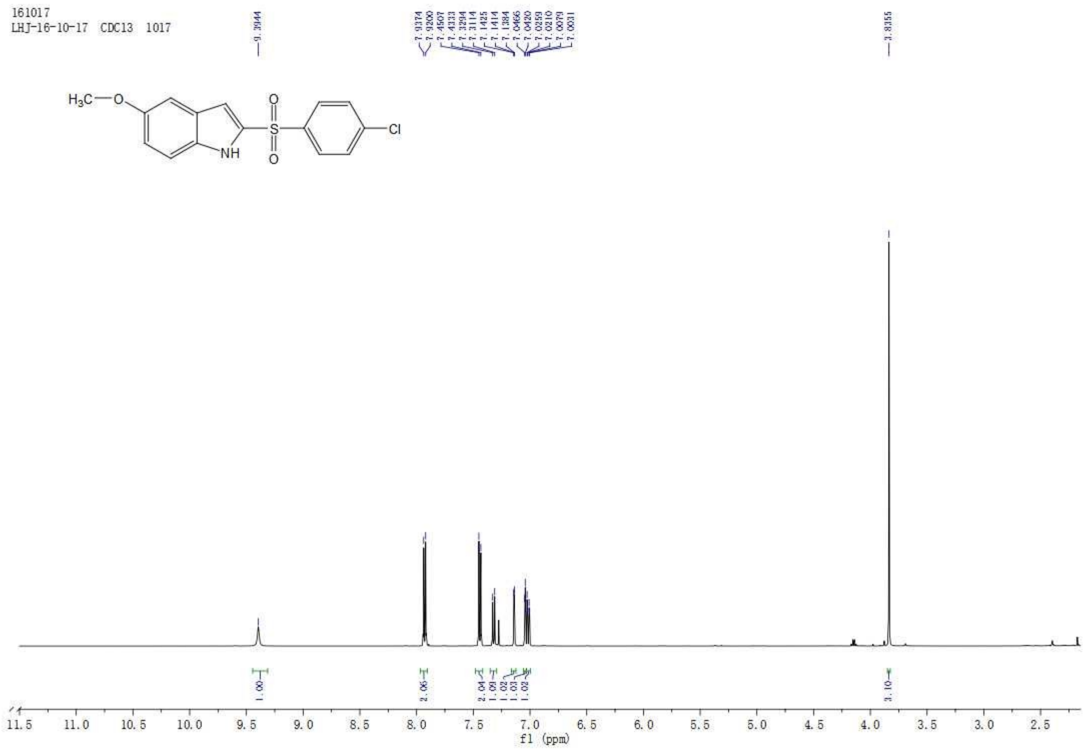

161020
lhj-16-10-18 CDCl3 1020
29.6715
f1 (ppm)

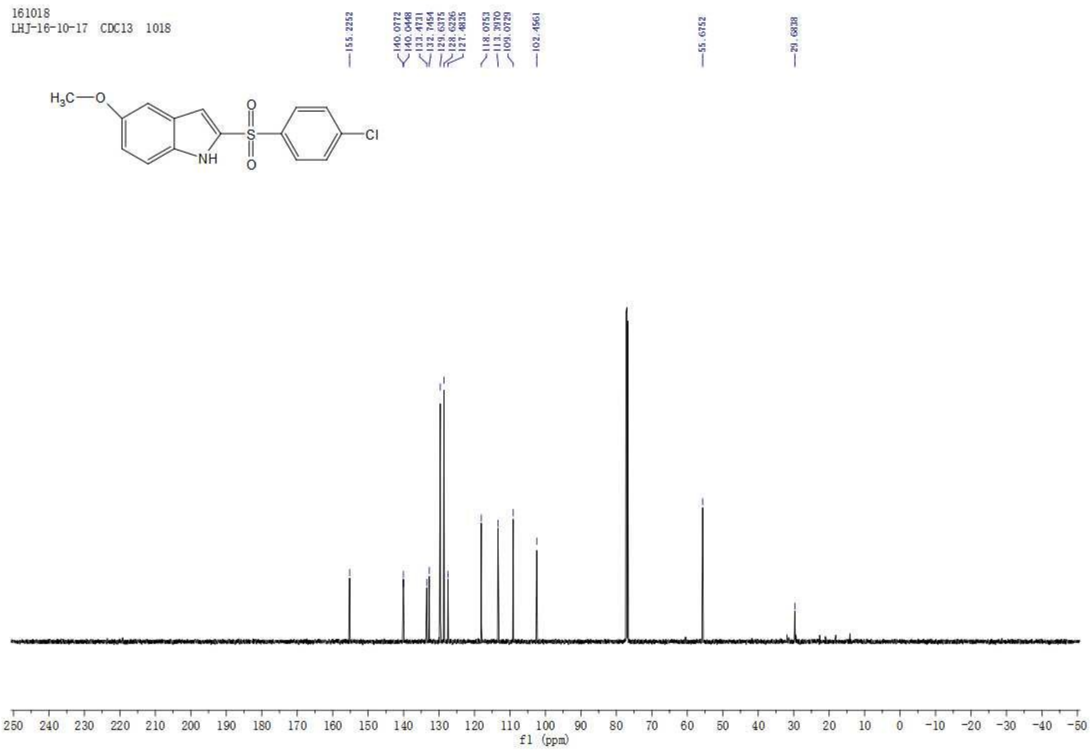
3p


161017
lhj-16-10-17 CDCl3 1017
f1 (ppm)

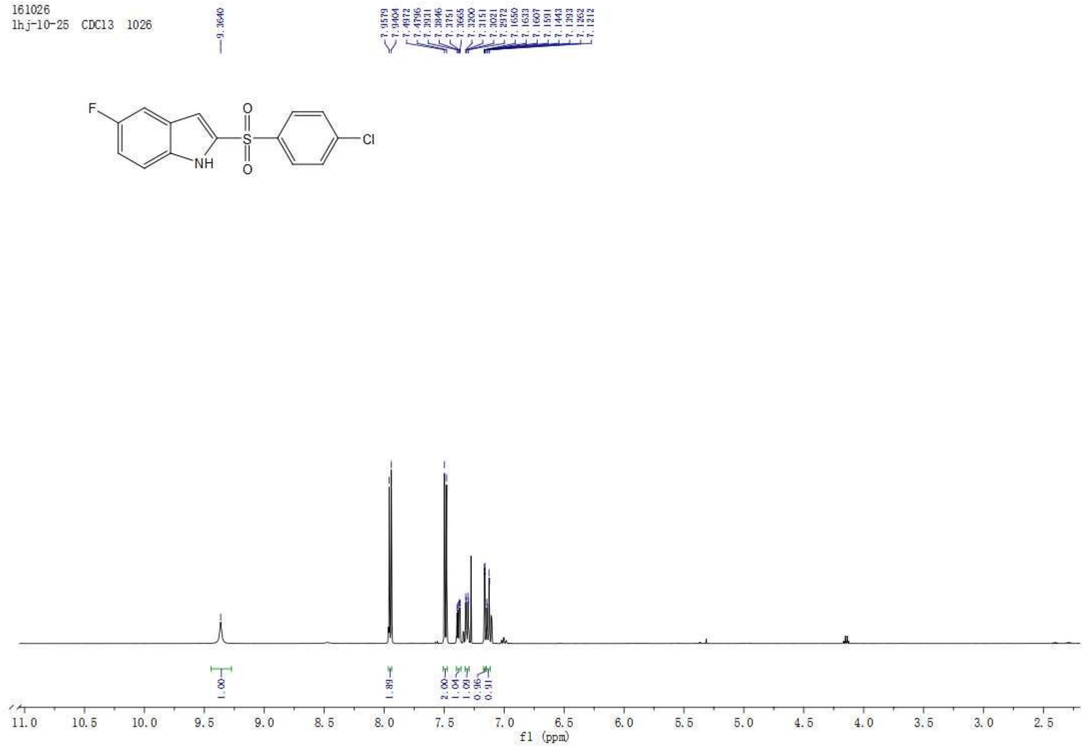

161018
lhj-16-10-17 CDCl3 1018
f1 (ppm)

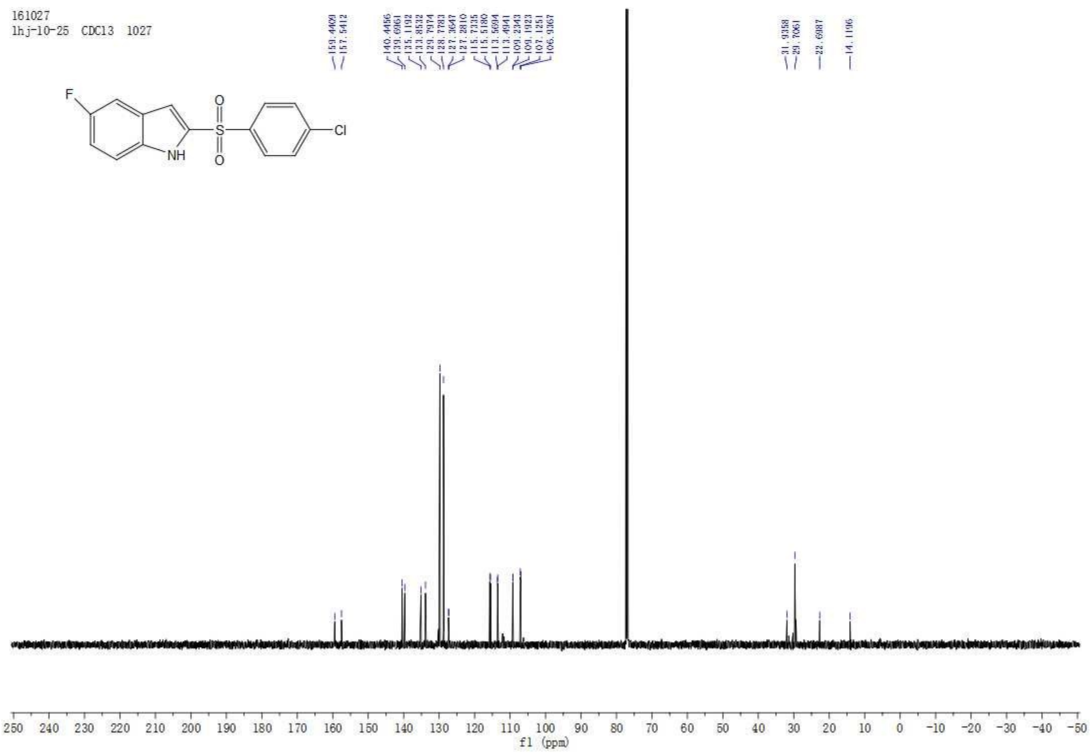
3q



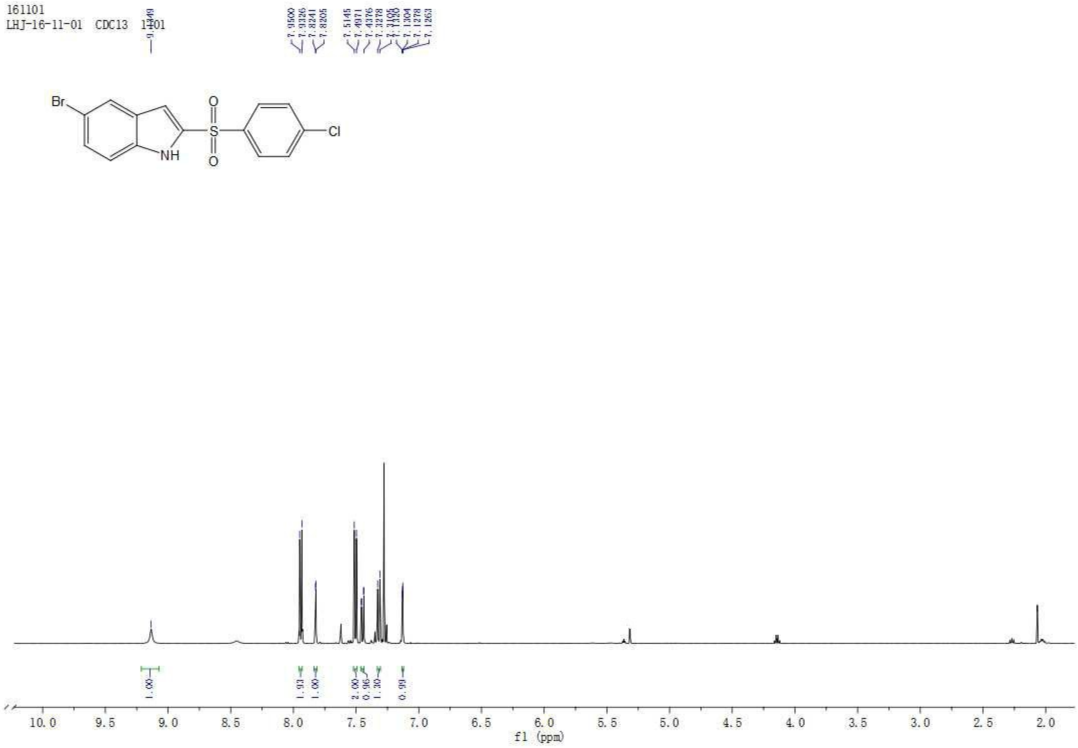


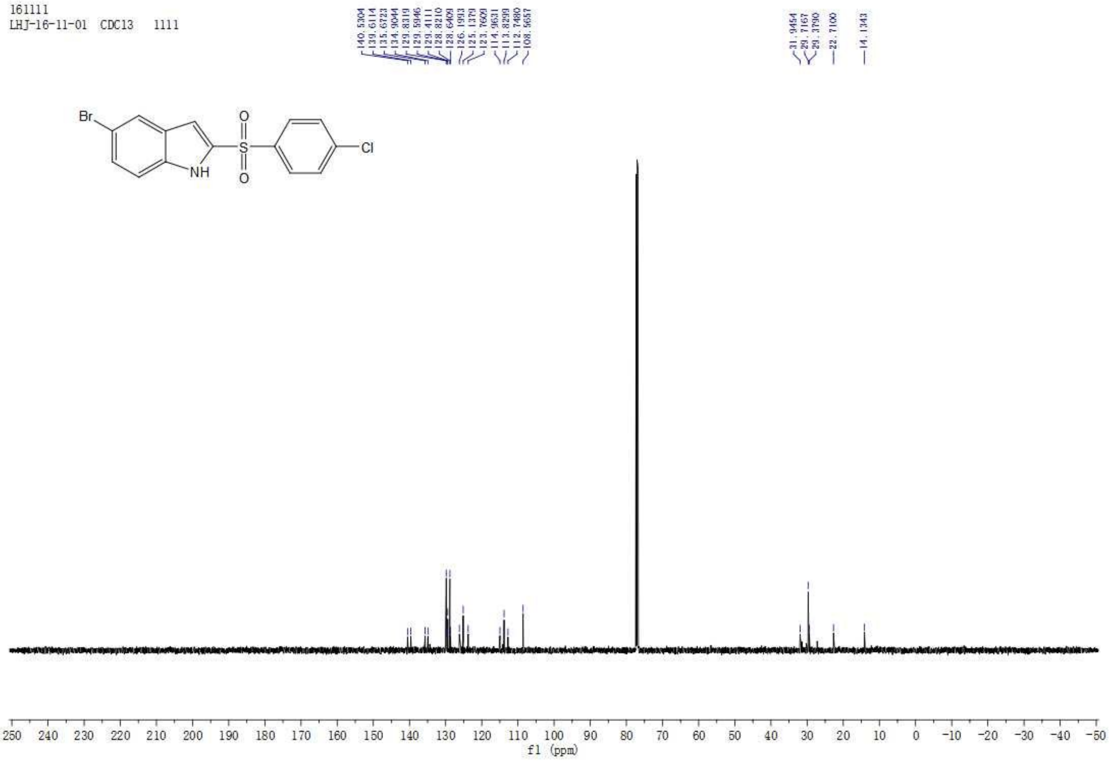
3r


161026
lhj-10-25 CDCl3 1026
f1 (ppm)

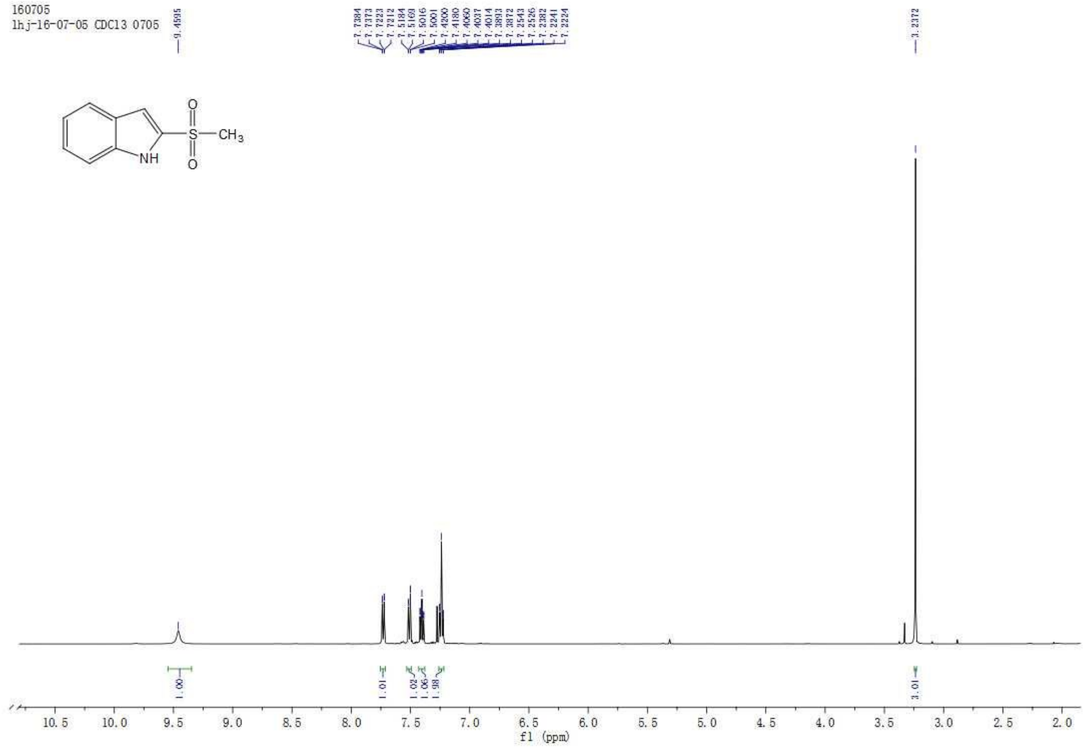

161027
lhj-10-25 CDCl3 1027
f1 (ppm)

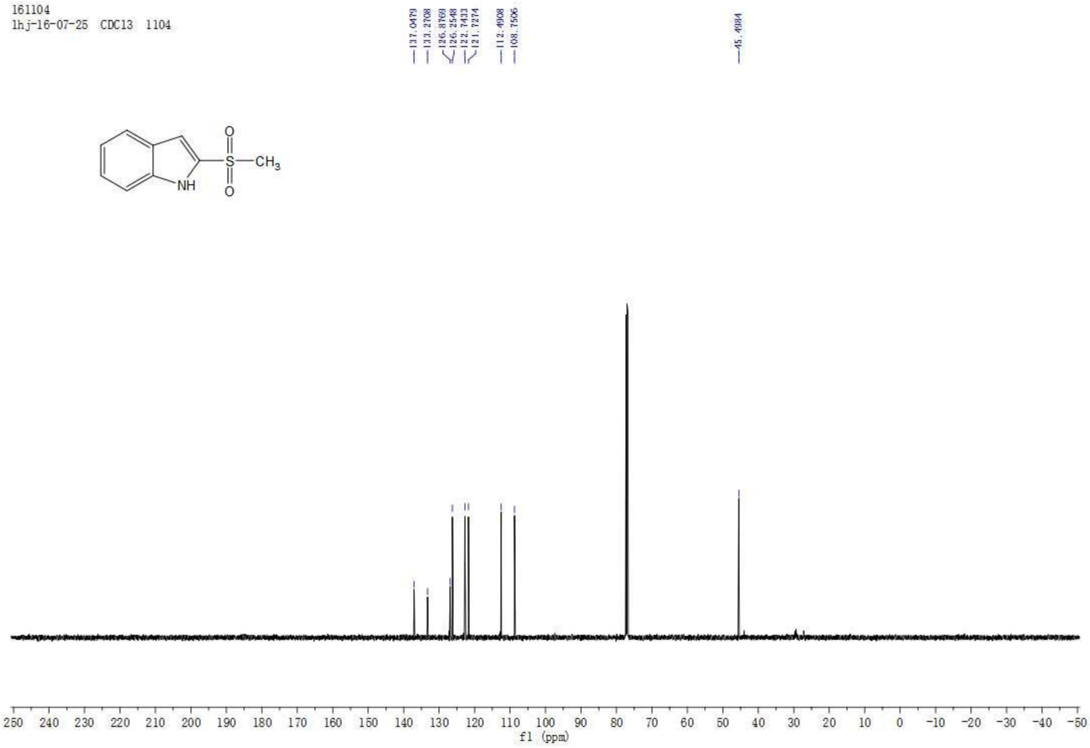
3s


161101
LHJ-16-11-01 CDC13 1101
7.9500
7.9326
7.8241
7.8205
7.5145
7.4971
7.4376
7.3278
7.1304
7.1278
7.1263
Br
NH
O
S
O
Cl
1.00
1.93
1.00
2.00
0.96
1.30
0.99
10.0 9.5 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
f1 (ppm)

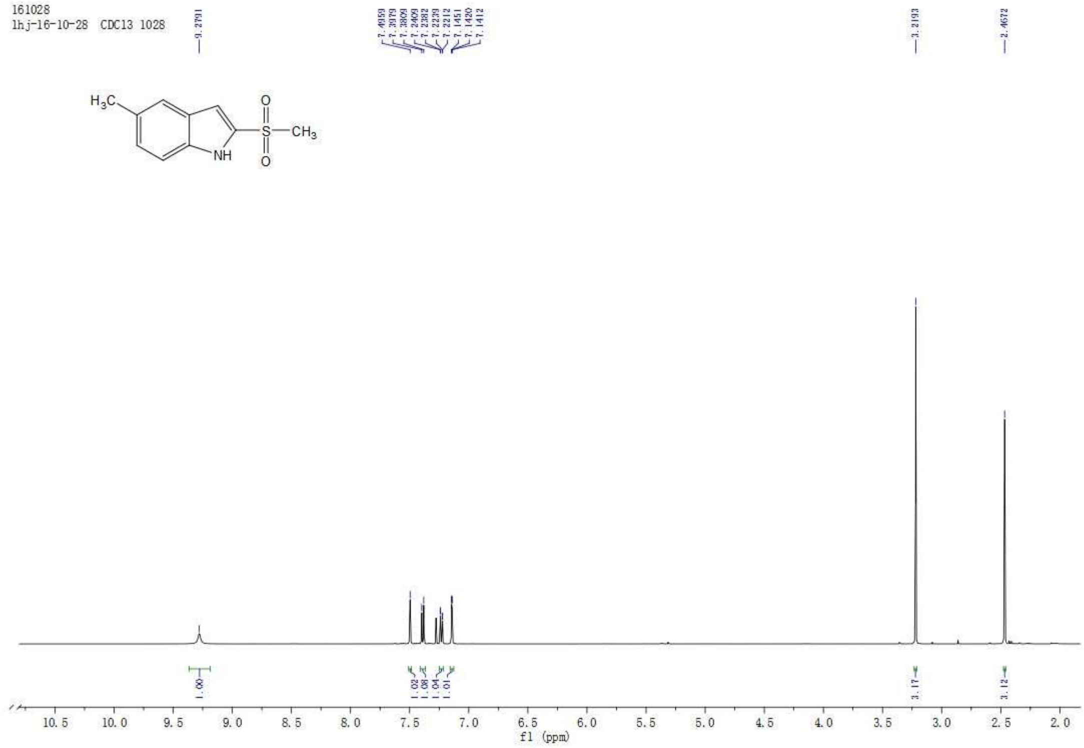

161111
LHJ-16-11-01 CDC13 1111
140.5304
139.6114
135.6723
134.9044
129.8319
129.5946
129.4111
128.8210
128.6409
126.1993
125.1170
123.7609
114.9631
113.8299
112.7480
108.5657
31.9454
29.7167
29.3790
22.7100
14.1343
Br
NH
O
S
O
Cl
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50
f1 (ppm)

3t

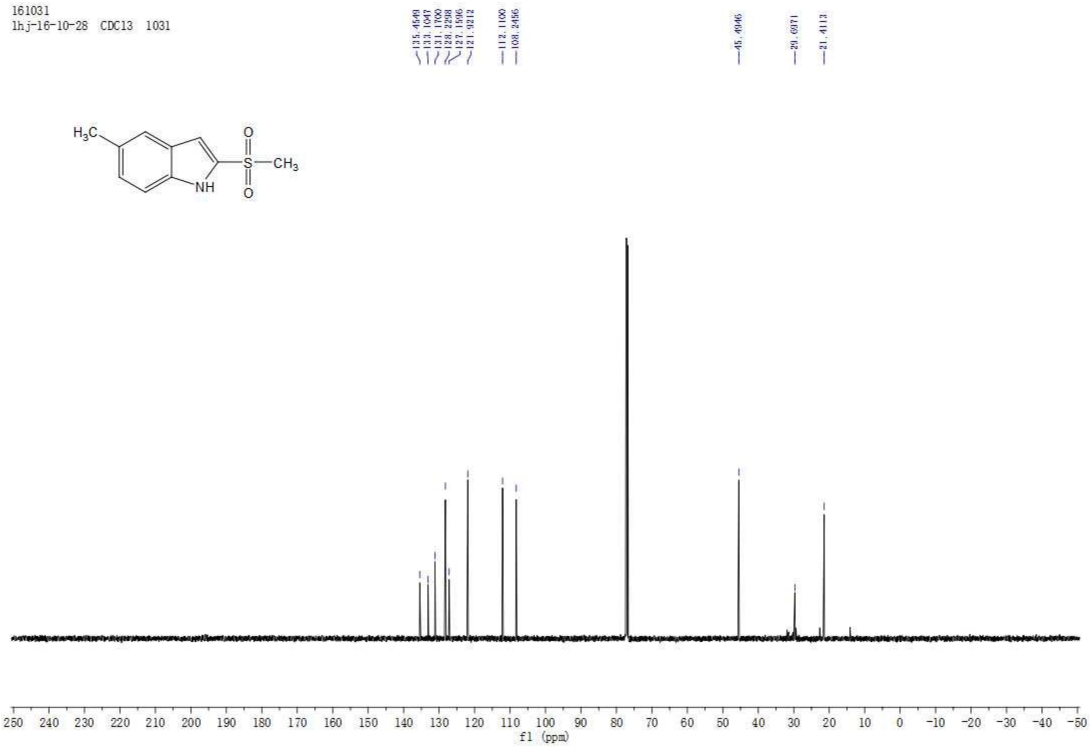




3u

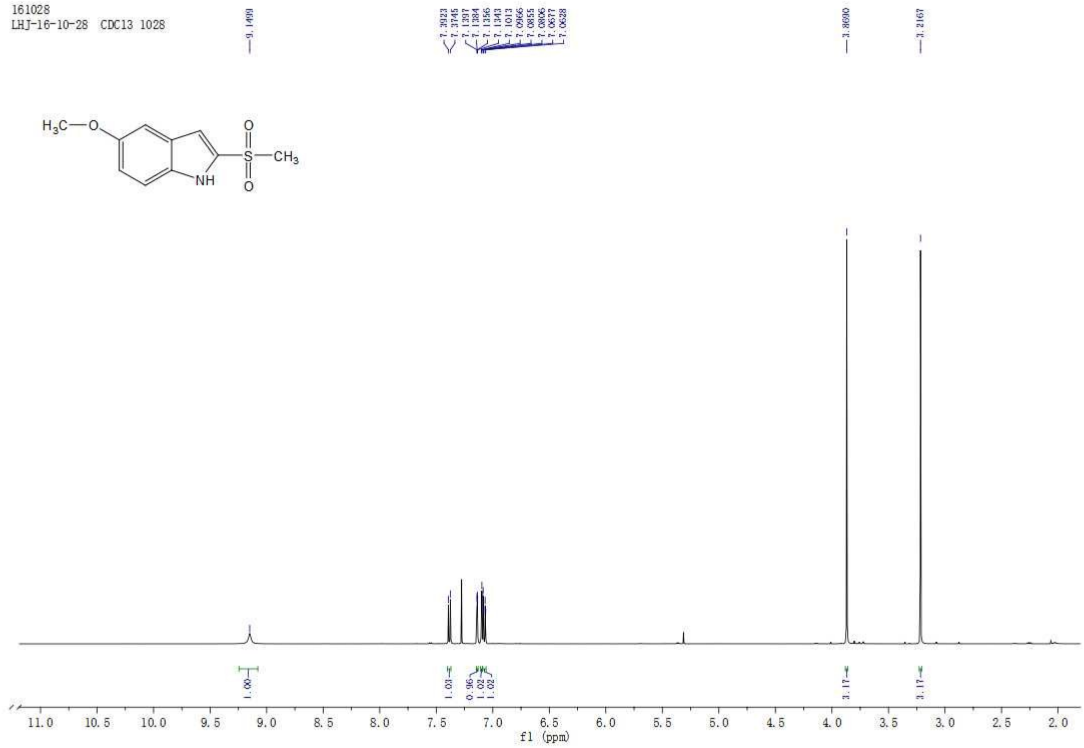


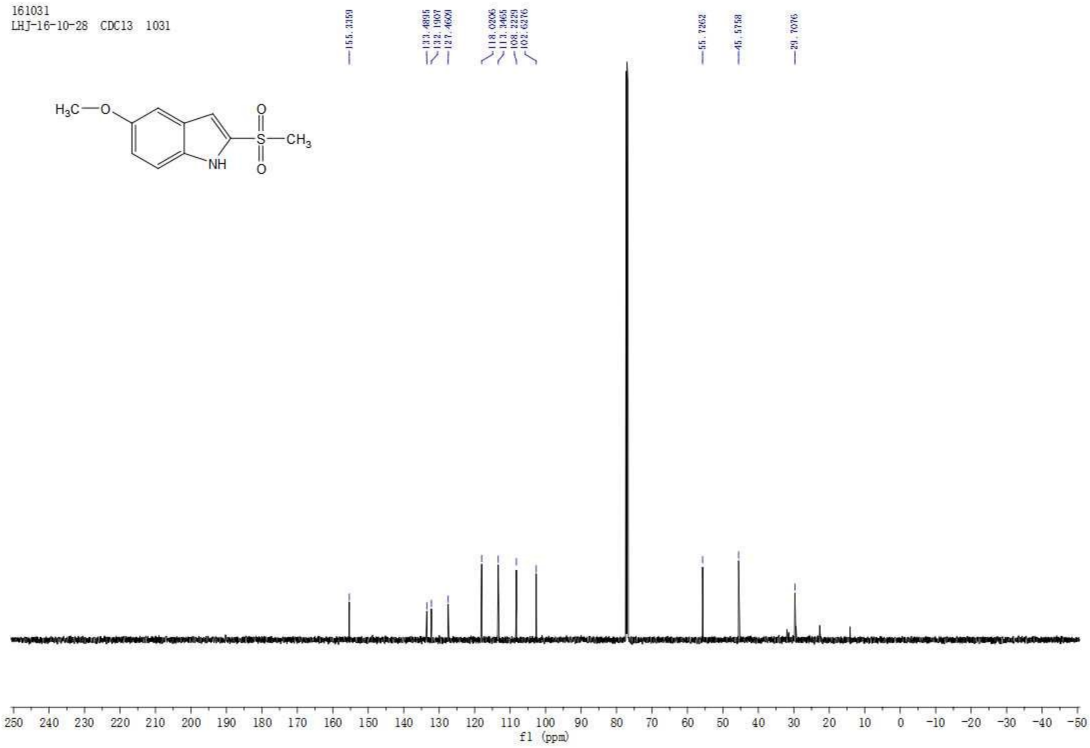
161028
lhj-16-10-28 CDCl3 1028
9.2791
7.4959
7.3979
7.3809
7.2409
7.2382
7.2239
7.2212
7.1451
7.1420
7.1412
3.2193
2.4672
H3C
S
O
O
CH3
NH
1.00
1.02
1.08
1.04
1.01
3.17
3.12
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f1 (ppm)



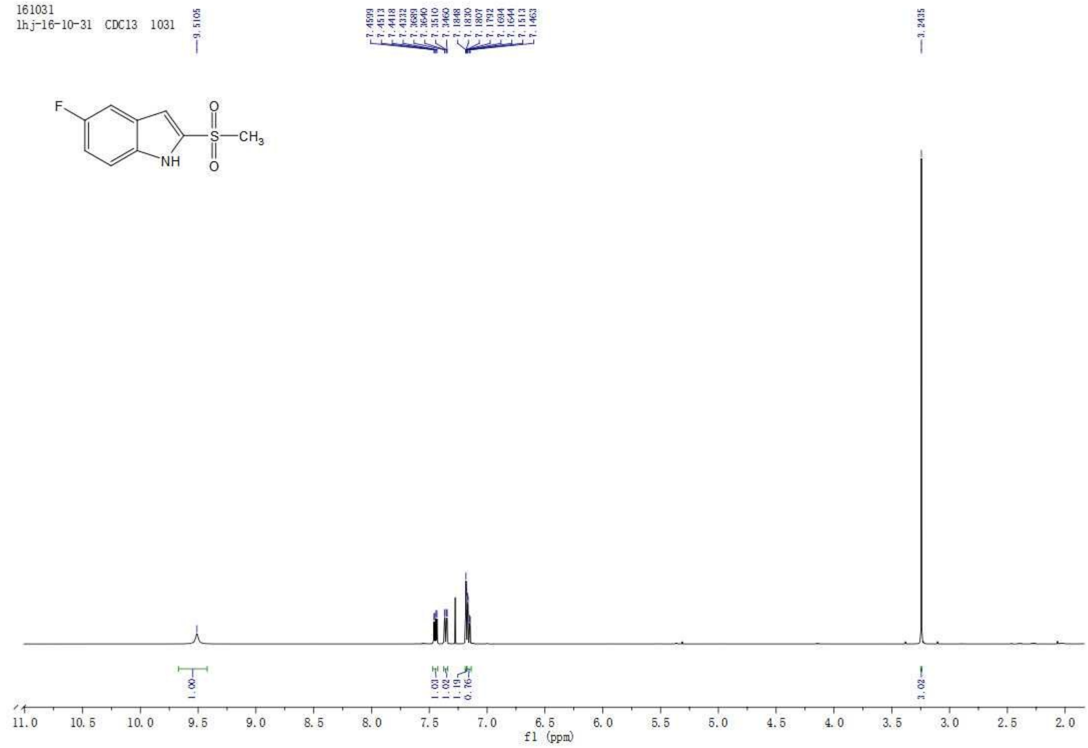
161031
lhj-16-10-28 CDCl3 1031
135.4549
133.1047
131.1700
128.2298
127.1596
121.9212
112.1100
108.2456
45.4946
29.6971
21.4113
H3C
S
O
O
CH3
NH
250
240
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
f1 (ppm)

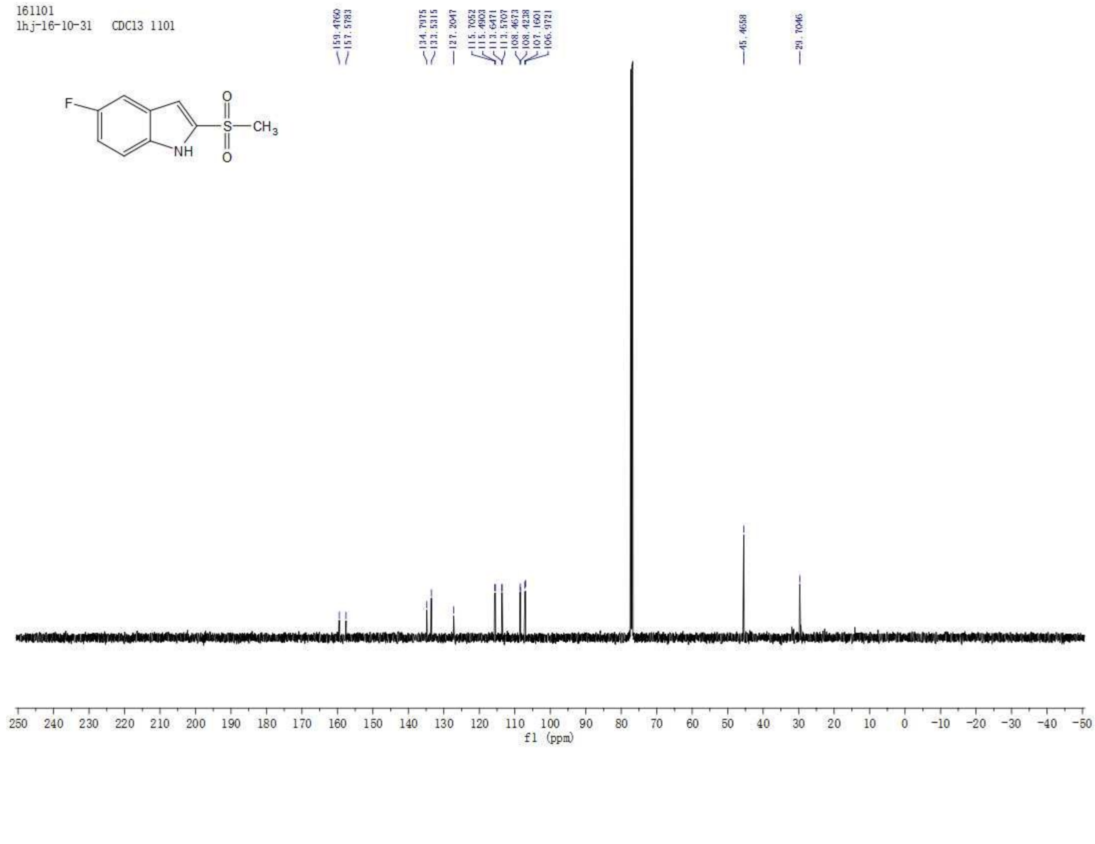
3v


161028
LHJ-16-10-28 CDC13 1028
f1 (ppm)


161031
LHJ-16-10-28 CDC13 1031
f1 (ppm)

3w


161031
lhj-16-10-31 CDCl3 1031
9.5105
7.4599
7.4513
7.4418
7.4332
7.3689
7.3640
7.3510
7.3460
7.1848
7.1830
7.1807
7.1792
7.1694
7.1644
7.1513
7.1463
3.2435
F
O
S
CH3
NH
O
1.00
1.03
1.02
1.19
0.76
3.02
f1 (ppm)


161101
lhj-16-10-31 CDCl3 1101
159.4160
157.5783
134.7975
133.5315
127.2047
115.7052
115.4903
113.6471
113.5707
108.4673
108.4238
107.1601
106.9721
45.4658
29.7046
F
O
S
CH3
NH
O
f1 (ppm)

3x