Supporting Information

Metal-free radical cascade chloromethylation of unactivated alkenes: synthesis of polychloro-substituted indolines

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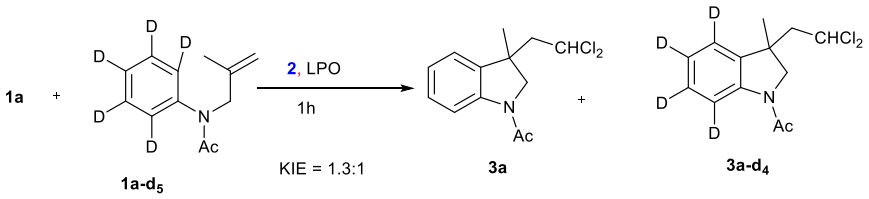
1. General experimental details

General Information: All chemicals were used as received without further purification unless stated otherwise. NMR spectra were recorded at ambient temperature on a 300, 400 or 500 MHz NMR spectrometer. Chemical shifts (δ) are given in ppm relative to TMS, the coupling constants *J* are given in Hz. HRMS were recorded on a TOF LC/MS equipped with electrospray ionization (ESI) probe operating in positive or negative ion mode.

Experimental procedure: Under N_2, the mixture of **1** (0.2 mmol), CH_2Cl_2 or CCl_4 (1 mL) and LPO (0.4 mmol) were added into the tube and sealed. The mixture was stirred at 100 ^oC for 6 h (16 h for CCl_4). Then, the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography on silica gel to give the product.

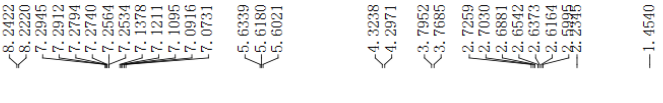
2. Mechanism Studies

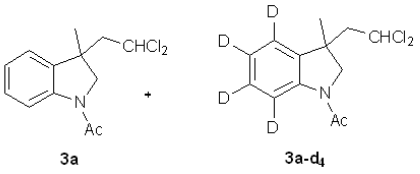
Intermolecular competition experiment with isotopically labeled 1a-d_5



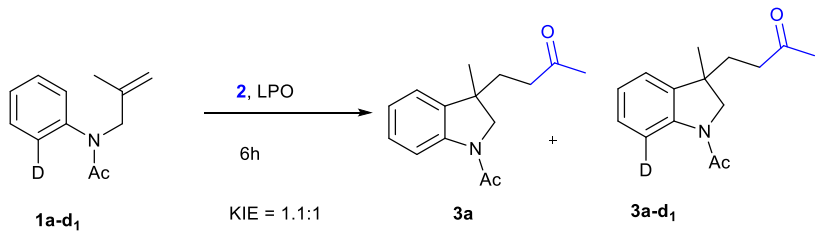
Under N_2, the mixture of **1a** (18.9 mg, 0.1 mmol), **1a-d_5** (19.4 mg, 0.1 mmol), **2** (1 mL) and LPO (0.4 mmol) were added into the tube and sealed. The mixture was stirred at 100 ^oC for 1 h. Then, the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography on silica gel to give the **3a** and **3a-d_4** in 40% yield.

^1H NMR ($CDCl_3$, 400 MHz): δ 8.23 (d, *J* = 8.1 Hz, 0.48H), 7.29-7.25 (m, 1H), 7.14-7.07 (m, 2H), 5.62 (t, *J* = 6.4 Hz, 1H), 4.31 (d, *J* = 10.7 Hz, 1H), 3.78 (d, *J* = 10.7 Hz, 1H), 2.74-2.60 (m, 2H), 2.23 (s, 3H), 1.45 (s, 3H).

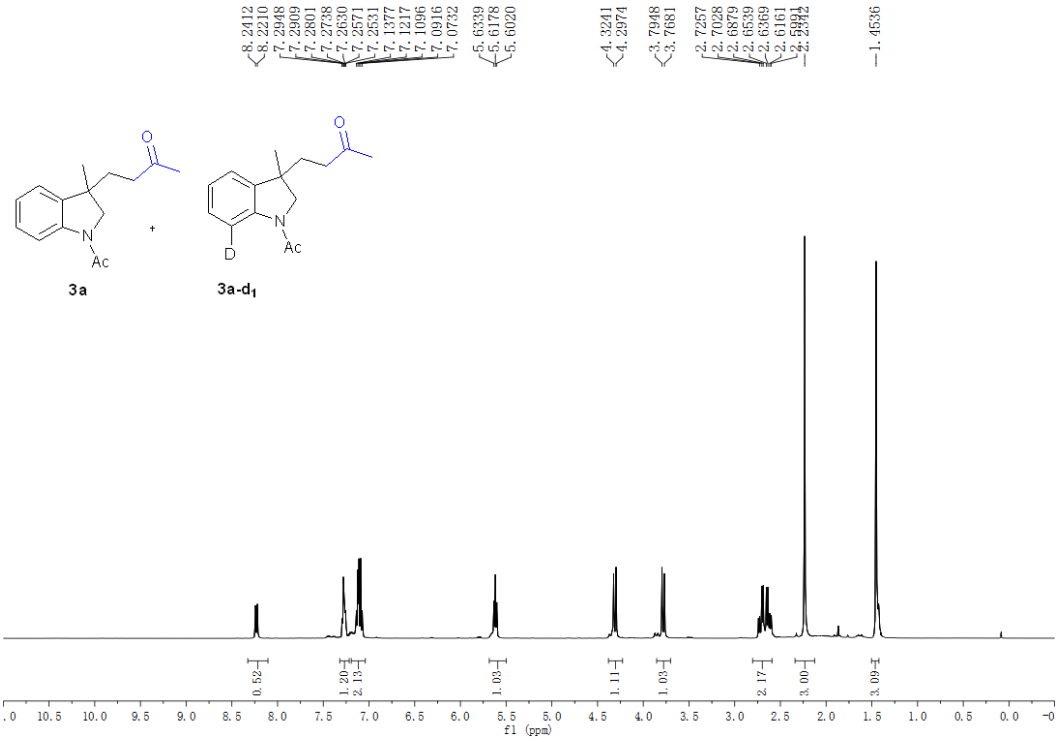




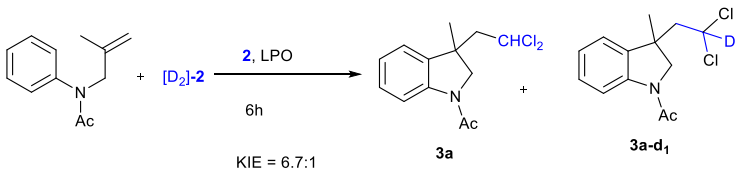
Intramolecular competition experiment isotopically labeled [D_1]-1a



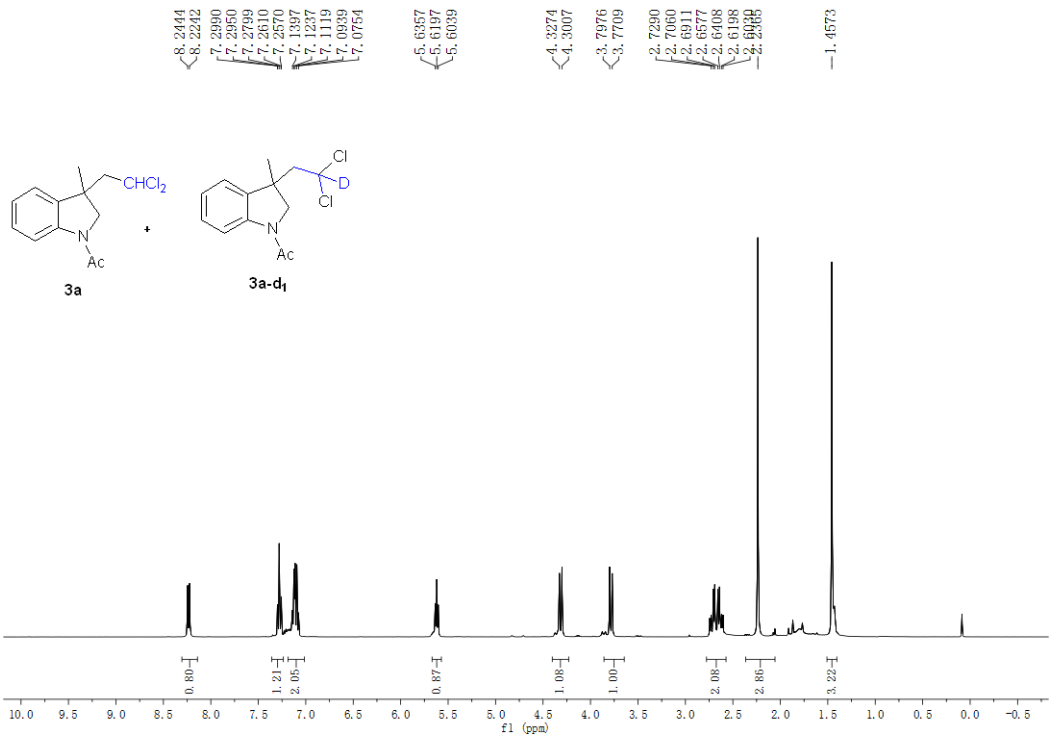
Under N_2, the mixture of **1a-d$_1$** (38.0 mg, 0.2 mmol), **2** (1 mL) and LPO (0.4 mmol) were added into the tube and sealed. The mixture was stirred at 100 ^oC for 6 h. Then, the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography on silica gel to give the **3a** and **3a-d$_1$** in 76% yield. ^1H NMR ($CDCl_3$, 400 MHz): δ 8.23 (d, *J* = 8.1 Hz, 0.52H), 7.29-7.25 (m, 1H), 7.14-7.07 (m, 2H), 5.62 (t, *J* = 6.4 Hz, 1H), 4.31 (d, *J* = 10.7 Hz, 1H), 3.78 (d, *J* = 10.7 Hz, 1H), 2.74-2.59 (m, 2H), 2.23 (s, 3H), 1.45 (s, 3H).



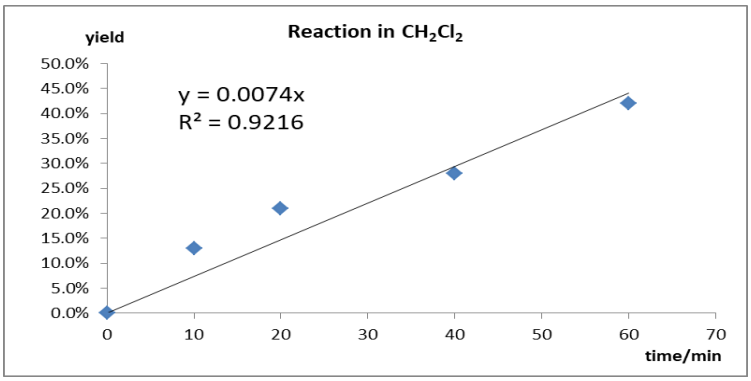
The KIE studies on solvent (competition reaction):

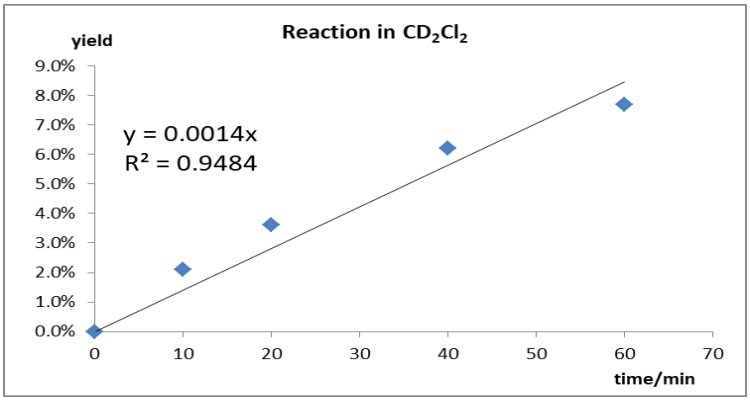


Under N_2, the mixture of **1a** (37.8 mg, 0.2 mmol), LPO (0.4 mmol), **2** (0.5 mL) and **[d$_2$]-2** (0.5 mL) were added into the tube and sealed. The mixture was stirred at 100 ^oC for 6 h. Then, the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography on silica gel to give the **3a** and **3a-d$_1$** in 72% yield. ^1H NMR ($CDCl_3$, 400 MHz): δ 8.23 (d, *J* = 8.1 Hz, 1H), 7.29-7.25 (m, 1H), 7.14-7.07 (m, 2H), 5.62 (t, *J* = 6.4 Hz, 0.87H), 4.31 (d, *J* = 10.7 Hz, 1H), 3.78 (d, *J* = 10.7 Hz, 1H), 2.74-2.60 (m, 2H), 2.23 (s, 3H), 1.46 (s, 3H).



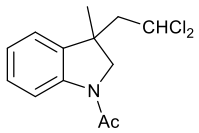
The Kinetic Isotopic Effect Studies on Solvent (parallel reaction): In eight parallel sealed tubes, the mixture of **1a** (0.1 mmol) was treated by LPO (0.2 mmol) in CH_2Cl_2 (0.5 mL) or CD_2Cl_2 (0.5 mL) (four experiments for each). Then the reaction was quenched by EtOAc in specified time. The mixture was analyzed by GC-MS to give the yield of product. A significant intermolecular kinetic isotope effect (k_H/k_D = 5.2:1) was observed. The results were listed below:





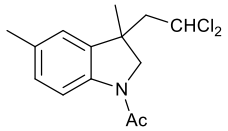
3. Characterization data of the products

1-(3-(2,2-dichloroethyl)-3-methylindolin-1-yl)ethan-1-one (3a)



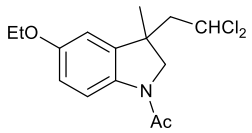
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (42.3mg, 78%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.22 (d, *J* = 8.1 Hz, 1H), 7.29-7.24 (m, 1H), 7.13-7.06 (m, 2H), 5.61 (t, *J* = 6.4 Hz, 1H), 4.30 (d, *J* = 10.7 Hz, 1H), 3.78 (d, *J* = 10.7 Hz, 1H), 2.73-2.59 (m, 2H), 2.23 (s, 3H), 1.45 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.8, 142.2, 136.8, 128.9, 124.2, 122.3, 117.5, 70.4, 61.3, 53.9, 43.6, 26.9, 24.4. HRMS (ESI) *m/z* calcd for $C_{13}H_{16}Cl_2NO$ $(M+H)^+$ 272.0603, found 272.0605.

1-(3-(2,2-dichloroethyl)-3,5-dimethylindolin-1-yl)ethan-1-one (3b)



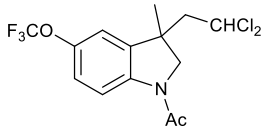
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (45.6mg, 80%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.09 (d, *J* = 8.2 Hz, 1H), 7.06 (d, *J* = 8.2 Hz, 1H), 6.91 (s, 1H), 5.62 (t, *J* = 6.4 Hz, 1H), 4.29 (d, *J* = 10.7 Hz, 1H), 3.76 (d, *J* = 10.7 Hz, 1H), 2.73-2.58 (m, 2H), 2.24 (s, 3H), 2.21 (s, 3H), 1.43 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.6, 139.8, 136.9, 133.9, 129.4, 122.8, 117.2, 70.5, 61.4, 53.9, 43.5, 26.9, 24.3, 21.3. HRMS (ESI) *m/z* calcd for $C_{14}H_{18}Cl_2NO$ $(M+H)^+$ 286.0760, found 286.0761.

1-(3-(2,2-dichloroethyl)-5-ethoxy-3-methylindolin-1-yl)ethan-1-one (3c)



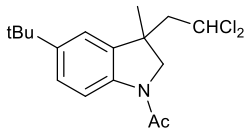
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 4/1) gave a brown liquid (47.9mg, 76%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.12 (d, *J* = 8.8 Hz, 1H), 6.77 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.67 (d, *J* = 2.5 Hz, 1H), 5.61 (t, *J* = 6.4 Hz, 1H), 4.28 (d, *J* = 10.7 Hz, 1H), 4.02 (q, *J* = 6.9 Hz, 2H), 3.76 (d, *J* = 10.7 Hz, 1H), 2.71-2.57 (m, 2H), 2.20 (s, 3H), 1.43-1.39 (m, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.1, 156.1, 138.3, 135.8, 118.2, 113.6, 109.5, 70.4, 64.1, 61.4, 53.9, 43.7, 26.8, 24.1, 15.0. HRMS (ESI) *m/z* calcd for: $C_{15}H_{20}Cl_2NO_2$ $(M+H)^+$ 316.0866, found 316.0870.

1-(3-(2,2-dichloroethyl)-3-methyl-5-(trifluoromethoxy)indolin-1-yl)ethan-1-one (3d)



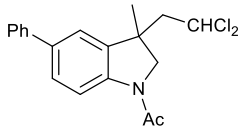
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 4/1) gave a coloress liquid (50.4mg, 71%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.24 (d, *J* = 8.8 Hz, 1H), 7.12 (d, *J* = 8.5 Hz, 1H), 6.97 (s, 1H), 5.66 (t, *J* = 6.5 Hz, 1H), 4.35 (d, *J* = 10.7 Hz, 1H), 3.84 (d, *J* = 10.7 Hz, 1H), 2.72-2.61 (m, 2H), 2.24 (s, 3H), 1.46 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.8, 145.4, 140.7, 138.8, 121.7, 120.6 (q, J_{C-F} = 255.3 Hz), 118.2, 115.7, 69.9, 61.5, 53.6, 43.6, 26.6, 24.2. HRMS (ESI) *m/z* calcd for $C_{14}H_{15}Cl_2F_3NO_2$ $(M+H)^+$ 356.0426, found 356.0429.

1-(5-(*tert*-butyl)-3-(2,2-dichloroethyl)-3-methylindolin-1-yl)ethan-1-one (3e)



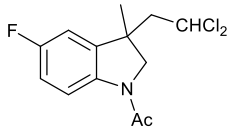
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (51.0mg, 78%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.12 (d, *J* = 8.5 Hz, 1H), 7.30 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.11 (d, *J* = 1.4 Hz, 1H), 5.63 (t, *J* = 6.4 Hz, 1H), 4.28 (d, *J* = 10.7 Hz, 1H), 3.78 (d, *J* = 10.7 Hz, 1H), 2.74-2.61 (m, 2H), 2.22 (s, 3H), 1.46 (s, 3H), 1.32 (s, 9H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.5, 147.5, 139.8, 136.6, 125.8, 118.9, 116.9, 70.5, 61.6, 54.0, 43.8, 34.8, 31.7, 26.7, 24.2. HRMS (ESI) *m/z* calcd for $C_{17}H_{24}Cl_2NO$ $(M+H)^+$ 328.1229, found 328.1226.

1-(3-(2,2-dichloroethyl)-3-methyl-5-phenylindolin-1-yl)ethan-1-one (3f)



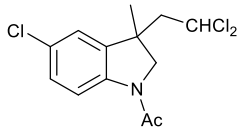
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (50.7mg, 73%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.30 (d, *J* = 8.4 Hz, 1H), 7.58 (d, *J* = 7.5 Hz, 2H), 7.54-7.51 (m, 1H), 7.46 (d, *J* = 7.4 Hz, 2H), 7.37-7.34 (m, 2H), 5.69 (t, *J* = 6.4 Hz, 1H), 4.37 (d, *J* = 10.7 Hz, 1H), 3.84 (d, *J* = 10.7 Hz, 1H), 2.81-2.66 (m, 2H), 2.26 (s, 3H), 1.51 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.8, 141.5, 140.8, 137.6, 137.5, 128.9, 127.9, 127.3, 127.0, 120.9, 117.6, 70.4, 61.6, 53.9, 43.7, 26.9, 24.4. HRMS (ESI) *m/z* calcd for $C_{19}H_{20}Cl_2NO$ $(M+H)^+$ 348.0916, found 348.0921.

1-(3-(2,2-dichloroethyl)-5-fluoro-3-methylindolin-1-yl)ethan-1-one (3g)



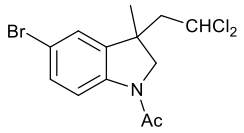
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (41.0 mg, 71%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.22-8.18 (m, 1H), 6.98-6.93 (m, 1H), 6.84-6.81 (m, 1H), 5.63 (t, *J* = 6.5 Hz, 1H), 4.34 (d, *J* = 10.7 Hz, 1H), 3.81 (d, *J* = 10.7 Hz, 1H), 2.72-2.59 (m, 2H), 2.23 (s, 3H), 1.45 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.5, 159.7 (d, J_{C-F} = 236.7 Hz), 138.8 (d, J_{C-F} = 7.3 Hz), 138.2 (d, J_{C-F} = 1.7 Hz), 118.5 (d, J_{C-F} = 7.9 Hz), 115.3 (d, J_{C-F} = 22.5 Hz), 109.7 (d, J_{C-F} = 24.1 Hz), 70.0, 61.4, 53.7, 43.6, 26.7, 24.1. HRMS (ESI) *m/z* calcd for $C_{13}H_{15}Cl_2FNO$ $(M+H)^+$ 290.0509, found 290.0511.

1-(5-chloro-3-(2,2-dichloroethyl)-3-methylindolin-1-yl)ethan-1-one (3h)



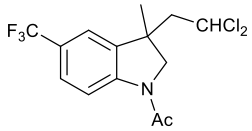
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (46.9 mg, 77%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.15 (d, *J* = 8.6 Hz, 1H), 7.23-7.20 (m, 1H), 7.08-7.07 (m, 1H), 5.62 (t, *J* = 6.5 Hz, 1H), 4.33 (d, *J* = 10.7 Hz, 1H), 3.79 (d, *J* = 10.7 Hz, 1H), 2.71-2.58 (m, 2H), 2.22 (s, 3H), 1.43 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.9, 140.7, 138.9, 129.1, 128.9, 122.6, 118.5, 70.1, 61.4, 53.6, 43.6, 26.8, 24.2. HRMS (ESI) *m/z* calcd for $C_{13}H_{15}Cl_3NO$ $(M+H)^+$: 306.0214, found 306.0211.

1-(5-bromo-3-(2,2-dichloroethyl)-3-methylindolin-1-yl)ethan-1-one (3i)



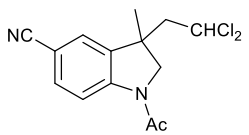
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (50.2 mg, 72%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.11 (d, *J* = 8.6 Hz, 1H), 7.37 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.22 (d, *J* = 1.5 Hz, 1H), 5.65 (t, *J* = 6.5 Hz, 1H), 4.33 (d, *J* = 10.7 Hz, 1H), 3.79 (d, *J* = 10.7 Hz, 1H), 2.72-2.58 (m, 2H), 2.22 (s, 3H), 1.44 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.9, 141.3, 139.2, 131.8, 125.5, 118.9, 116.5, 70.1, 61.3, 53.7, 43.6, 26.8, 24.3. HRMS (ESI) *m/z* calcd for $C_{13}H_{15}BrCl_2NO$ $(M+H)^+$ 349.9709, found 349.9713.

1-(3-(2,2-dichloroethyl)-3-methyl-5-(trifluoromethyl)indolin-1-yl)ethan-1-one (3j)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (54.2 mg, 80%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.32 (d, *J* = 8.5 Hz, 1H), 7.54 (d, *J* = 8.4, 1H), 7.35 (s, 1H), 5.68 (t, *J* = 6.7 Hz, 1H), 4.39 (d, *J* = 10.8 Hz, 1H), 3.86 (d, *J* = 10.7 Hz, 1H), 2.77-2.63 (m, 2H), 2.26 (s, 3H), 1.48 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.2, 144.7, 137.6, 126.5 (q, $J_{C\text{-}F}$ = 3.4 Hz), 126.0 (q, $J_{C\text{-}F}$ = 32.2 Hz), 124.1 (q, $J_{C\text{-}F}$ = 269.9 Hz), 119.3 (q, $J_{C\text{-}F}$ = 3.2 Hz), 117.1, 69.8, 61.4, 53.5, 43.4, 26.7, 24.3. HRMS (ESI) *m/z* calcd for $C_{14}H_{15}Cl_2F_3NO$ $(M+H)^+$ 340.0477, found 340.0481.

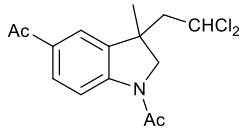
1-acetyl-3-(2,2-dichloroethyl)-3-methylindoline-5-carbonitrile (3k)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 3/1) gave a coloress liquid (39.7 mg, 67%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.29 (d, *J* = 8.3 Hz, 1H), 7.56 (d, *J* = 8.4,

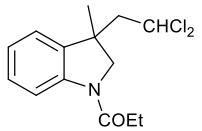
1H), 7.39 (s, 1H), 5.68-5.65 (m, 1H), 4.38 (d, *J* = 10.9 Hz, 1H), 3.85 (d, *J* = 10.8 Hz, 1H), 2.74-2.61 (m, 2H), 2.25 (s, 3H), 1.46 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 169.5, 145.7, 138.2, 133.9, 126.3, 119.1, 117.6, 106.9, 69.8, 61.4, 53.5, 43.5, 26.9, 24.5. HRMS (ESI) *m/z* calcd for $C_{14}H_{15}Cl_2N_2O$ $(M+H)^+$ 297.0556, found 297.0555.

1,1'-(3-(2,2-dichloroethyl)-3-methylindoline-1,5-diyl)bis(ethan-1-one) (3l)



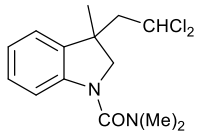
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 3/1) gave a coloress liquid (38.2 mg, 61%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.25 (d, *J* = 8.4 Hz, 1H), 7.87 (d, *J* = 8.5 Hz, 1H), 7.77 (s, 1H), 5.65 (t, *J* = 6.5 Hz, 1H), 4.39 (d, *J* = 10.8 Hz, 1H), 3.85 (d, *J* = 10.7 Hz, 1H), 2.78-2.73 (m, 1H), 2.67-2.61 (m, 1H), 2.58 (s, 3H), 2.25 (s, 3H), 1.46 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.9, 169.5, 146.1, 137.8, 133.3, 130.9, 122.2, 116.6, 70.2, 61.7, 53.7, 43.3, 27.1, 26.7, 24.5. HRMS (ESI) *m/z* calcd for $C_{15}H_{18}Cl_2NO_2$ $(M+H)^+$ 314.0709, found 314.0712.

1-(3-(2,2-dichloroethyl)-3-methylindolin-1-yl)propan-1-one (3m)



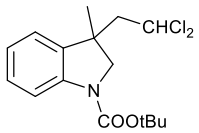
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (38.8 mg, 68%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.26 (d, *J* = 8.1 Hz, 1H), 7.29-7.25 (m, 1H), 7.14-7.06 (m, 2H), 5.63 (t, *J* = 6.4 Hz, 1H), 4.30 (d, *J* = 10.7 Hz, 1H), 3.76 (d, *J* = 10.7 Hz, 1H), 2.73-2.59 (m, 2H), 2.49-2.43 (m, 2H), 1.45 (s, 3H), 1.24 (t, *J* = 7.3 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 172.2, 142.4, 136.7, 128.9, 124.1, 122.3, 117.4, 70.4, 60.4, 53.9, 43.6, 29.4, 26.9, 8.8. HRMS (ESI) *m/z* calcd for $C_{14}H_{18}Cl_2NO$ $(M+H)^+$ 286.0760, found 286.0761.

3-(2,2-dichloroethyl)-N,N,3-trimethylindoline-1-carboxamide (3n)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 10/1) gave a coloress liquid (46.8 mg, 78%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.19 (t, *J* = 7.2 Hz, 1H), 7.11 (d, *J* = 7.0 Hz, 1H), 6.96-6.93 (m, 2H), 5.73-5.70 (m, 1H), 3.98 (d, *J* = 10.6 Hz, 1H), 3.71 (d, *J* = 10.6 Hz, 1H), 2.95 (s, 6H), 2.72-2.56 (m, 2H), 1.42 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.8, 143.6, 136.9, 128.3, 122.8, 121.9, 113.7, 70.5, 62.4, 53.3, 42.9, 38.3, 25.3. HRMS (ESI) *m/z* calcd for $C_{14}H_{19}Cl_2N_2O$ $(M+H)^+$ 301.0869, found 301.0873.

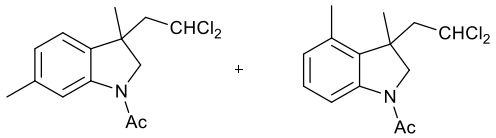
***tert*-butyl 3-(2,2-dichloroethyl)-3-methylindoline-1-carboxylate (3o)**



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a coloress liquid (48.0 mg, 73%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.89 (s, 0.53H), 7.50 (s, 0.46H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.10 (d, *J* = 7.2 Hz, 1H), 7.01 (t, *J* = 7.4 Hz, 1H), 5.63 (s, 1H), 4.20 (d, *J* = 11.5 Hz, 1H), 3.71 (s, 1H), 2.72-2.57 (m, 2H), 1.58 (s, 9H), 1.43 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 153.9, 152.3, 142.3, 136.3, 128.6, 122.6, 122.4, 115.1, 80.8, 70.4, 60.1, 53.9, 42.8, 28.5, 26.7. HRMS (ESI) *m/z* calcd for $C_{16}H_{22}Cl_2NO_2$ $(M+H)^+$ 330.1022, found 330.1028.

<u>1-(3-(2,2-dichloroethyl)-3,6-dimethylindolin-1-yl)ethan-1-one</u> (3p) <u>and</u>

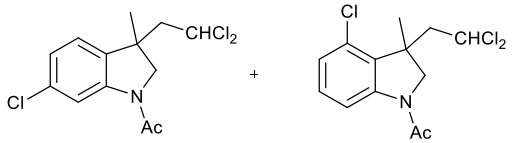
<u>1-(3-(2,2-dichloroethyl)-3,4-dimethylindolin-1-yl)ethan-1-one</u> (3p')



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (37.3 mg, 72%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.16 (d, *J* = 8.1 Hz, 0.43H), 8.08 (s, 0.38H), 7.17 (t, *J* = 7.8 Hz, 0.55H), 7.00 (d, *J* = 7.6 Hz, 0.58H), 6.90 (d, *J* = 7.6 Hz, 0.46H), 6.84 (d, *J* = 7.6 Hz, 0.51H), 5.59 (t, *J* = 6.4 Hz, 0.5H), 5.48-5.45 (m, 0.47H), 4.38 (d, *J* = 10.7 Hz, 0.5H), 4.29 (d, *J* = 10.7 Hz, 0.47H), 3.76 (d, *J* = 10.7 Hz, 0.46H), 3.70 (d, *J* = 10.7 Hz, 0.5H), 2.87-2.81 (m, 0.53H), 2.73-2.56 (m, 1.59H), 2.36 (s, 3H), 2.22 (s, 3H), 1.55 (s, 3H), 1.43 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.8, 168.6, 143.2, 142.3, 139.0, 133.9, 133.7, 132.6, 129.0, 127.2, 124.9, 121.9, 118.1, 115.4, 70.5, 70.4, 61.6, 61.1, 54.1, 52.5, 44.5, 43.3, 27.0, 26.6, 24.6, 24.4, 21.8, 19.1. MS (EI): 285, 272, 243, 188, 146, 131.

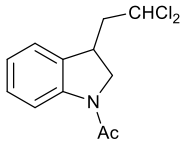
<u>1-(6-chloro-3-(2,2-dichloroethyl)-3-methylindolin-1-yl)ethan-1-one</u> (3q) <u>and</u>

<u>1-(4-chloro-3-(2,2-dichloroethyl)-3-methylindolin-1-yl)ethan-1-one</u> (3q')



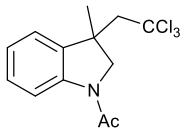
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (41.3 mg, 74%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.26-8.21 (m, 1H), 7.21 (t, *J* = 8.1 Hz, 1H), 7.05-6.99 (m, 1H), 5.63-5.54 (m, 1H), 4.47 (d, *J* = 10.7 Hz, 0.73H), 4.32 (d, *J* = 10.7 Hz, 0.27H), 3.81-3.75 (m, 1H), 3.06-3.01 (m, 0.73H), 2.78-2.61 (m, 1.28H), 2.21 (s, 3H), 1.60 (s, 2.23H), 1.43 (s, 0.81H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 168.8, 168.7, 144.6, 143.0, 135.2, 134.4, 131.3, 130.3, 129.9, 125.3, 124.1, 122.9, 117.6, 115.9, 70.5, 70.0, 61.4, 61.3, 53.7, 51.1, 44.7, 43.2, 26.8, 26.5, 24.4, 24.2. MS (EI): 307, 263, 208, 166, 131.

<u>1-(3-(2,2-dichloroethyl)indolin-1-yl)ethan-1-one</u> (3r)



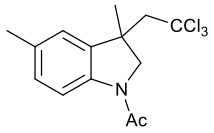
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (28.3 mg, 55%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.23 (d, *J* = 8.1 Hz, 1H), 7.28-7.24 (m, 1H), 7.22-7.17 (m, 1H), 7.07 (d, *J* = 7.4 Hz, 1H), 5.88 (d, *J* = 6.2 Hz, 1H), 4.30-4.25 (m, 1H), 3.84-3.70 (m, 2H), 2.70-2.64 (m, 1H), 2.54-2.48 (m, 1H), 2.25 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.6, 142.6, 132.6, 128.8, 124.0, 123.9, 117.4, 71.4, 55.2, 48.9, 37.8, 24.4. HRMS (ESI) *m/z* calcd for $C_{12}H_{14}Cl_2NO$ $(M+H)^+$ 258.0447, found 258.0448.

1-(3-methyl-3-(2,2,2-trichloroethyl)indolin-1-yl)ethan-1-one (5a)



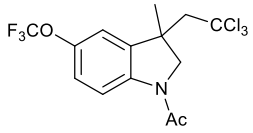
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (41.5 mg, 68%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.22 (d, *J* = 8.1 Hz, 1H), 7.29-7.24 (m, 1H), 7.18 (d, *J* = 7.4 Hz, 1H), 7.09 (t, *J* = 7.4 Hz, 1H), 4.52 (d, *J* = 10.8 Hz, 1H), 3.95 (d, *J* = 10.8 Hz, 1H), 3.36-3.21 (m, 2H), 2.26 (s, 3H), 1.62 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.8, 141.5, 138.1, 128.9, 124.2, 122.5, 117.4, 97.4, 62.9, 62.0, 45.3, 26.8, 24.4. HRMS (ESI) *m/z* calcd for $C_{13}H_{15}Cl_3NO$ $(M+H)^+$ 306.0214, found 306.0217.

1-(3,5-dimethyl-3-(2,2,2-trichloroethyl)indolin-1-yl)ethan-1-one (5b)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (43.4 mg, 68%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.09 (d, *J* = 8.2 Hz, 1H), 7.06 (d, *J* = 8.5 Hz, 1H), 6.98 (s, 1H), 4.50 (d, *J* = 11.0 Hz, 1H), 3.94 (d, *J* = 10.8 Hz, 1H), 3.35-3.19 (m, 2H), 2.35 (s, 3H), 2.25 (s, 3H), 1.61 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.5, 139.1, 138.3, 133.8, 129.4, 123.0, 117.1, 97.4, 62.9, 62.2, 45.3, 26.7, 24.3, 21.3. HRMS (ESI) *m/z* calcd for $C_{14}H_{17}Cl_3NO$ $(M+H)^+$ 320.0370, found 320.0368.

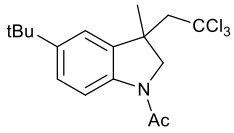
1-(3-methyl-3-(2,2,2-trichloroethyl)-5-(trifluoromethoxy)indolin-1-yl)ethan-1-one (5c)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 4/1) gave a coloress liquid (58.4 mg, 75%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.23 (d, *J* = 8.8 Hz, 1H), 7.11 (d, *J* = 8.8 Hz, 1H), 7.03 (s, 1H), 4.57 (d, *J* = 10.8 Hz, 1H), 3.97 (d, *J* = 10.8 Hz, 1H), 3.31-3.21 (m, 2H), 2.25 (s, 3H), 1.62 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.8, 145.4, 140.2, 139.5, 121.7,

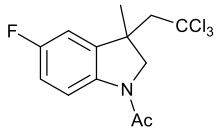
120.6 (q, $J_{C\text{-}F}$ = 255.1 Hz), 118.1, 116.1, 96.9, 62.5, 62.1, 45.2, 26.9, 24.2. HRMS (ESI) *m/z* calcd for $C_{14}H_{14}Cl_3F_3NO_2$ $(M+H)^+$ 390.0037, found 390.0042.

1-(5-(*tert*-butyl)-3-methyl-3-(2,2,2-trichloroethyl)indolin-1-yl)ethan-1-one (5d)



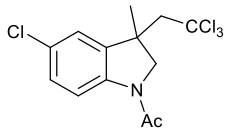
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (48.4 mg, 67%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.12 (d, *J* = 8.5 Hz, 1H), 7.30 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.16 (d, *J* = 1.5 Hz, 1H), 4.50 (d, *J* = 10.8 Hz, 1H), 3.95 (d, *J* = 10.8 Hz, 1H), 3.36-3.21 (m, 2H), 2.25 (s, 3H), 1.61 (s, 3H), 1.34 (s, 9H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.4, 147.5, 139.1, 137.7, 125.8, 119.2, 116.8, 97.5, 62.9, 62.3, 45.5, 34.8, 31.7, 26.7, 24.3. HRMS (ESI) *m/z* calcd for $C_{17}H_{23}Cl_3NO$ $(M+H)^+$ 362.0840, found 362.0843.

1-(5-fluoro-3-methyl-3-(2,2,2-trichloroethyl)indolin-1-yl)ethan-1-one (5e)



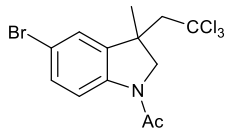
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (49.1 mg, 76%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.20-8.17 (m, 1H), 6.97-6.87 (m, 2H), 4.55 (d, *J* = 10.8 Hz, 1H), 3.96 (d, *J* = 10.9 Hz, 1H), 3.30-3.20 (m, 2H), 2.24 (s, 3H), 1.61 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.5, 159.6 (d, $J_{C\text{-}F}$ = 241.6 Hz), 139.9 (d, $J_{C\text{-}F}$ = 7.2 Hz), 137.6 (d, $J_{C\text{-}F}$ = 1.5 Hz), 118.4 (d, $J_{C\text{-}F}$ = 7.9 Hz), 115.3 (d, $J_{C\text{-}F}$ = 22.5 Hz), 110.0 (d, $J_{C\text{-}F}$ = 24.2 Hz), 97.0, 62.5, 62.0, 45.2, 26.8, 24.2. HRMS (ESI) *m/z* calcd for $C_{13}H_{14}Cl_3FNO$ $(M+H)^+$ 324.0120, found 324.0121.

1-(5-chloro-3-methyl-3-(2,2,2-trichloroethyl)indolin-1-yl)ethan-1-one (5f)



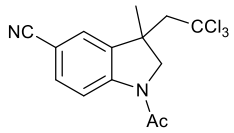
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (49.8 mg, 73%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.15 (d, *J* = 8.6 Hz, 1H), 7.21 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.14 (d, *J* = 1.9 Hz, 1H), 4.54 (d, *J* = 11.0 Hz, 1H), 3.95 (d, *J* = 10.9 Hz, 1H), 3.31-3.19 (m, 2H), 2.25 (s, 3H), 1.61 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.8, 140.2, 139.9, 128.9, 128.8, 122.9, 118.4, 96.9, 62.6, 62.0, 45.3, 26.9, 24.3. HRMS (ESI) *m/z* calcd for $C_{13}H_{14}Cl_4NO$ $(M+H)^+$ 339.9824, found 339.9827.

1-(5-bromo-3-methyl-3-(2,2,2-trichloroethyl)indolin-1-yl)ethan-1-one (5g)



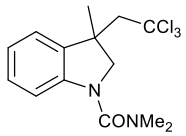
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (49.8 mg, 65%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.10 (d, *J* = 8.6 Hz, 1H), 7.36 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.28 (s, 1H), 4.53 (d, *J* = 11.0 Hz, 1H), 3.95 (d, *J* = 10.9 Hz, 1H), 3.31-3.19 (m, 2H), 2.24 (s, 3H), 1.61 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.8, 140.6, 140.2, 131.8, 125.8, 118.8, 116.4, 96.9, 62.6, 61.9, 45.3, 26.9, 24.3. HRMS (ESI) *m/z* calcd for $C_{13}H_{14}BrCl_3NO$ $(M+H)^+$ 383.9319, found 383.9324.

1-acetyl-3-methyl-3-(2,2,2-trichloroethyl)indoline-5-carbonitrile (5h)



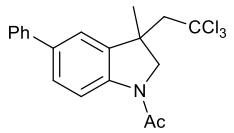
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 4/1) gave a coloress liquid (38.3 mg, 58%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.30 (d, *J* = 8.2 Hz, 1H), 7.56 (d, *J* = 8.4 Hz, 1H), 7.46 (s, 1H), 4.61 (d, *J* = 11.0 Hz, 1H), 4.00 (d, *J* = 10.9 Hz, 1H), 3.33-3.22 (m, 2H), 2.28 (s, 3H), 1.63 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 169.3, 145.1, 138.8, 133.8, 126.5, 118.9, 117.5, 106.8, 96.5, 62.4, 61.7, 44.9, 27.2, 24.4. HRMS (ESI) *m/z* calcd for $C_{14}H_{14}Cl_3N_2O$ $(M+H)^+$ 331.0166, found 331.0165.

N,N,3-trimethyl-3-(2,2,2-trichloroethyl)indoline-1-carboxamide (5i)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 10/1) gave a coloress liquid (46.1 mg, 69%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.22-7.16 (m, 2H), 6.97-6.92 (m, 2H), 4.26 (d, *J* = 10.7 Hz, 1H), 3.83 (d, *J* = 10.7 Hz, 1H), 3.34-3.16 (m, 2H), 2.96 (s, 6H), 1.59 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.8, 143.1, 137.9, 128.3, 123.0, 121.8, 113.5, 97.6, 63.0, 62.2, 44.6, 38.3, 25.4. HRMS (ESI) *m/z* calcd for $C_{14}H_{18}Cl_3N_2O$ $(M+H)^+$ 335.0479, found 335.0482.

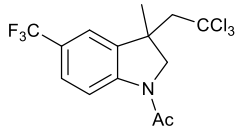
1-(3-methyl-5-phenyl-3-(2,2,2-trichloroethyl)indolin-1-yl)ethan-1-one (5j)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (66.7 mg, 88%). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.18 (d, *J* = 8.4 Hz, 1H), 7.49-7.16 (m, 8H), 4.45 (d, *J* = 10.9 Hz, 1H), 3.88 (d, *J* = 10.9 Hz, 1H), 3.32-3.14 (m, 2H), 2.17(s, 3H), 1.56 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 168.6, 140.8, 140.7, 138.6, 137.3, 128.8, 127.7, 127.2, 126.9, 121.1,

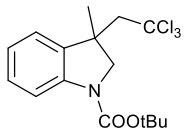
117.5, 97.2, 62.8, 62.2, 45.3, 26.8, 24.3. HRMS (ESI) *m/z* calcd for $C_{19}H_{19}Cl_3NO$ $(M+H)^+$ 382.0527, found 382.0533.

1-(3-methyl-3-(2,2,2-trichloroethyl)-5-(trifluoromethyl)indolin-1-yl)ethan-1-one (5k)



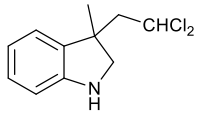
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a coloress liquid (56.7 mg, 88%). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.31 (d, *J* = 8.4 Hz, 1H), 7.54-7.51 (m, 1H), 7.39 (s, 1H), 4.59 (d, *J* = 11.0 Hz, 1H), 4.00 (d, *J* = 10.9 Hz, 1H), 3.36-3.21 (m, 2H), 2.28 (s, 3H), 1.64 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.1, 144.2, 138.4, 126.5 (q, J_{C-F} = 3.0 Hz), 125.9 (q, J_{C-F} = 15.8 Hz), 122.3, 119.7 (q, J_{C-F} = 3.5 Hz), 117.1, 96.7, 62.5, 61.9, 45.1, 26.9, 24.3. HRMS (ESI) *m/z* calcd for $C_{14}H_{14}Cl_3F_3NO$ $(M+H)^+$ 374.0088, found 374.0093.

***tert*-butyl 3-methyl-3-(2,2,2-trichloroethyl)indoline-1-carboxylate (5l)**



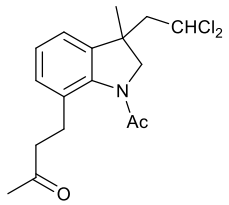
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a coloress liquid (65.3 mg, 90%). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.88-7.54 (m, 1H), 7.26-7.14 (m, 2H), 7.03-6.98 (m, 1H), 4.40 (d, *J* = 11.7 Hz, 1H), 3.93(s, 1H), 3.35-3.18 (m, 2H), 1.59 (s, 12H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 152.4, 141.3, 137.8, 128.6, 122.7, 122.5, 115.0, 97.4, 81.3, 63.0, 60.9, 44.2, 28.5, 26.7. HRMS (ESI) *m/z* calcd for $C_{16}H_{21}Cl_3NO_2$ $(M+H)^+$ 364.0632, found 364.0635.

3-(2,2-dichloroethyl)-3-methylindoline (7)



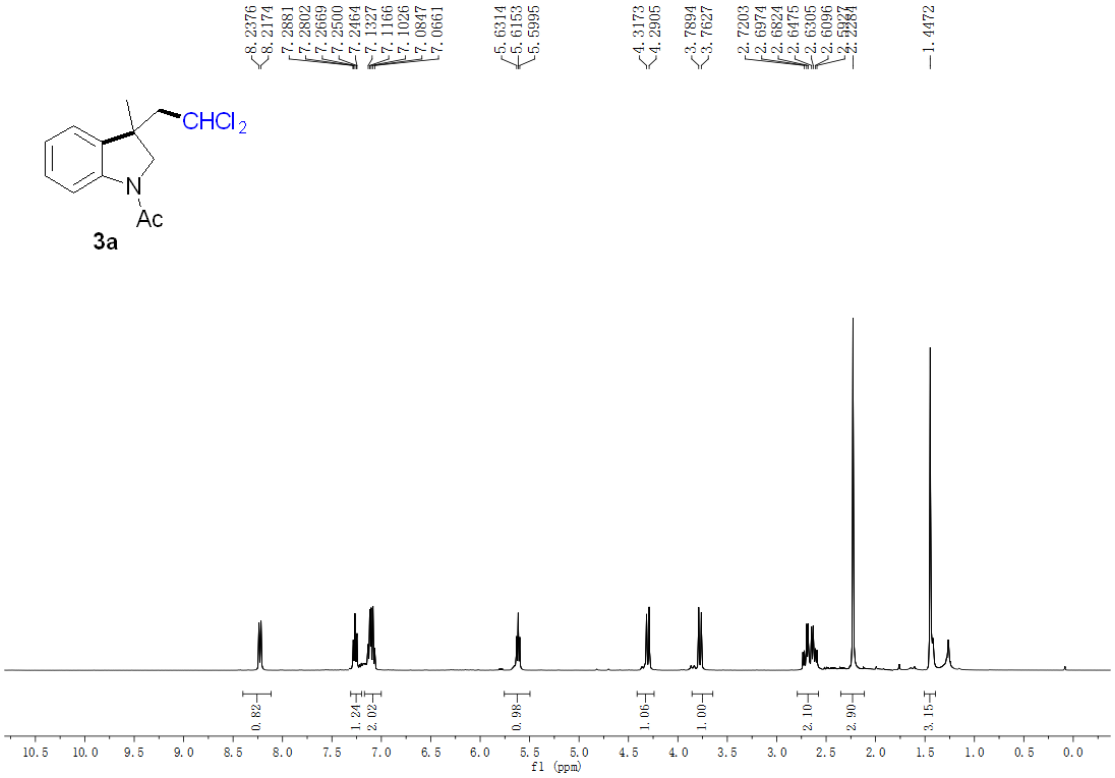
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 10/1) gave a coloress liquid (39.1 mg, 85%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.12-7.06 (m, 2H), 6.79 (t, *J* = 7.4 Hz, 1H), 6.69 (t, *J* = 7.8 Hz, 1H), 5.82-5.79 (m, 1H), 3.78 (d, *J* = 9.2 Hz, 1H), 3.37 (d, *J* = 9.2 Hz, 1H), 3.23 (s, 1H), 2.77-2.72 (m, 1H), 2.65-2.59 (m, 1H), 1.43 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 150.5, 134.9, 128.4, 122.7, 119.1, 110.2, 71.1, 60.0, 53.4, 45.2, 25.3. HRMS (ESI) *m/z* calcd for $C_{11}H_{14}Cl_2N$ $(M+H)^+$ 230.0498, found 230.0496.

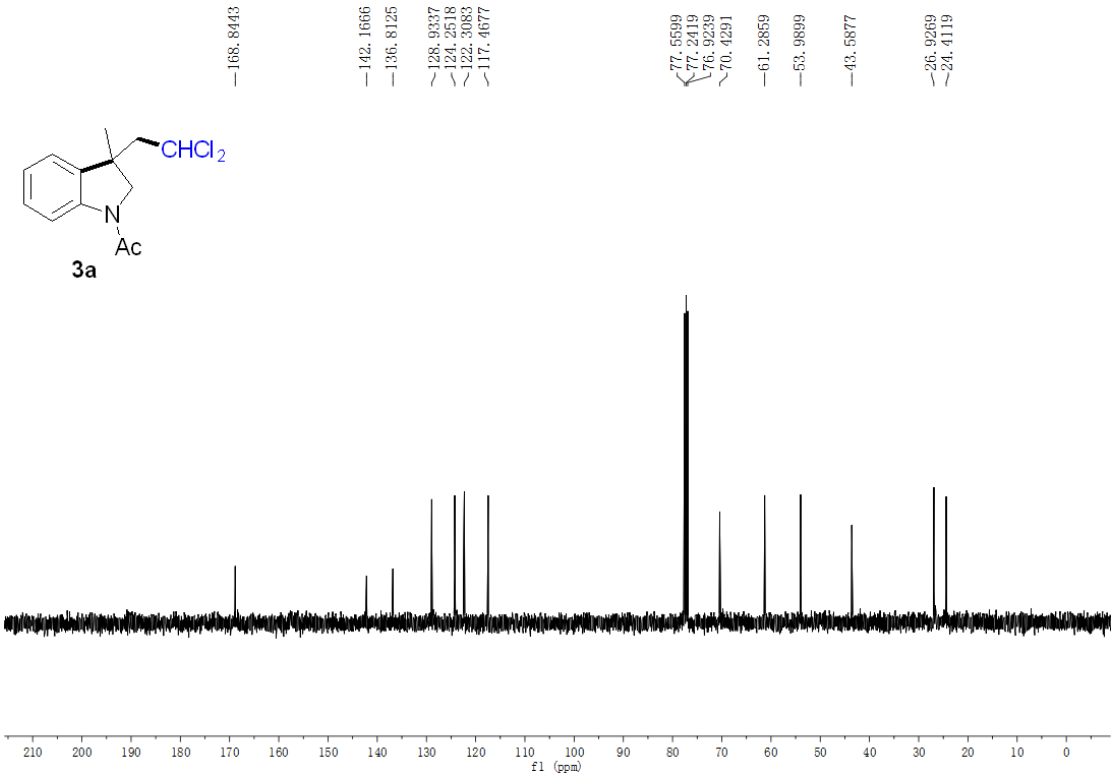
4-(1-acetyl-3-(2,2-dichloroethyl)-3-methylindolin-7-yl)butan-2-one (8)

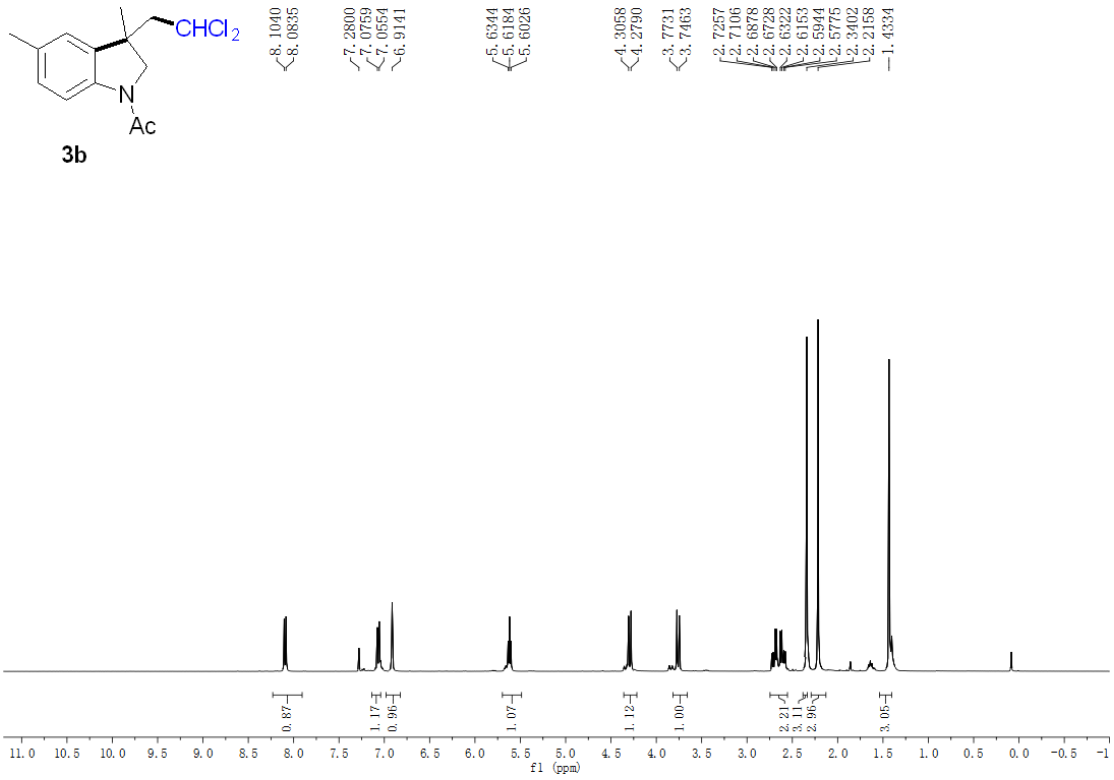


Flash column chromatography on silica gel (petroleum ether/ethyl acetate 3/1) gave a brown liquid (47.7 mg, 70%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.15-7.09 (m, 2H), 7.00-6.98 (m, 1H), 5.80-5.77 (m, 1H), 4.24 (d, *J* = 10.9 Hz, 1H), 3.80 (d, *J* = 11.0 Hz, 1H), 2.93-2.81 (m, 4H), 2.67-2.51 (m, 2H), 2.30 (s, 3H), 2.16 (s, 3H), 1.38 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 208.7, 169.4, 140.9, 140.0, 132.3, 129.5, 126.1, 119.8, 70.3, 62.7, 51.8, 44.5, 43.5, 29.8, 29.7, 27.6, 23.6. HRMS (ESI) *m/z* calcd for $C_{17}H_{22}Cl_2NO_2$ $(M+H)^+$ 342.1022, found 342.1025.

4. Copies of ^{1}H NMR and ^{13}C NMR spectra of the products



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3.7463
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2.7106
2.6878
2.6728
2.6322
2.6153
2.5944
2.5775
2.3402
2.2158
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1.07
1.12
1.00
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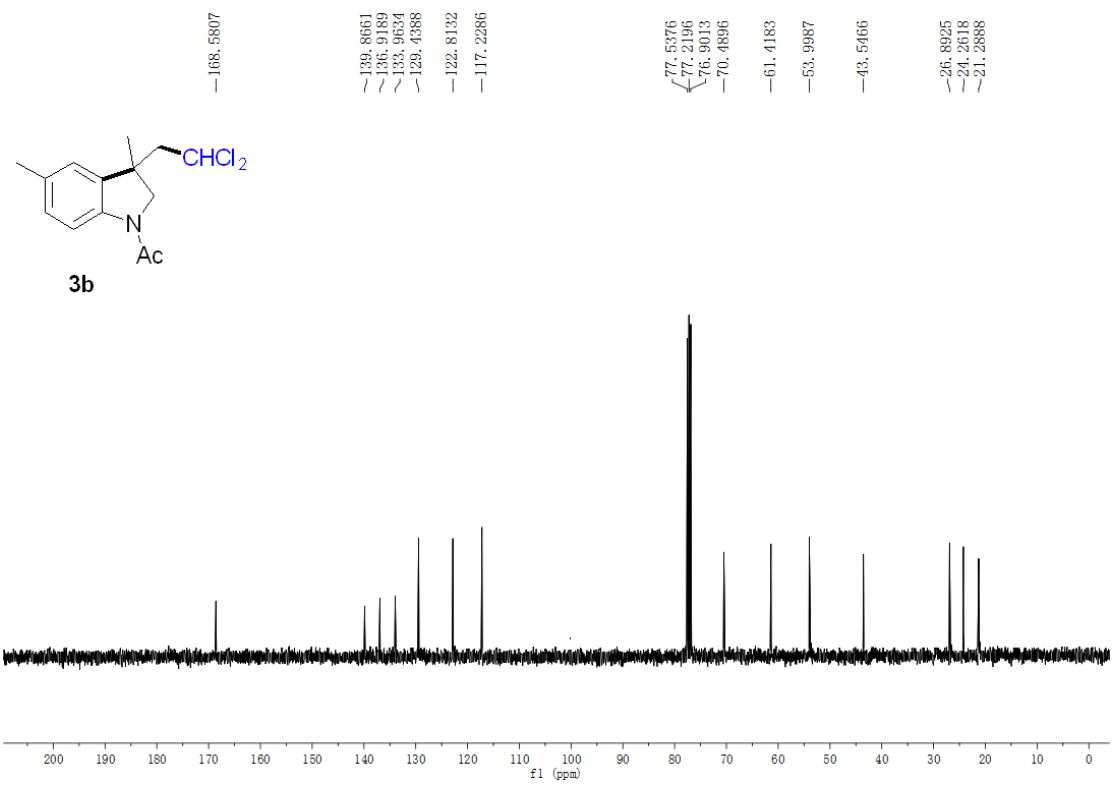
2.11
2.96
3.05
f1 (ppm)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70.4896
61.4183
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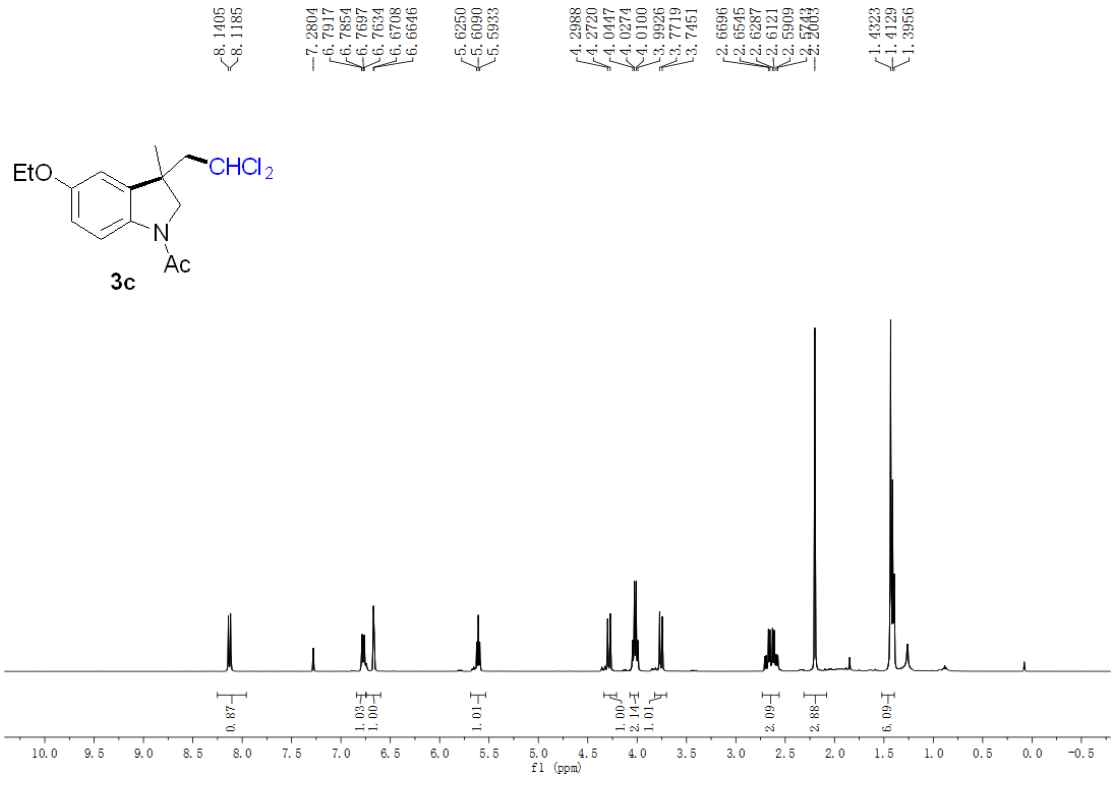
43.5466
26.8925
24.2618
21.2888
CHCl2
N
Ac
3b
f1 (ppm)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5.6090
5.5933
4.2988
4.2720
4.0447
4.0274
4.0100
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3.7719
3.7451
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EtO
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Ac
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1.00
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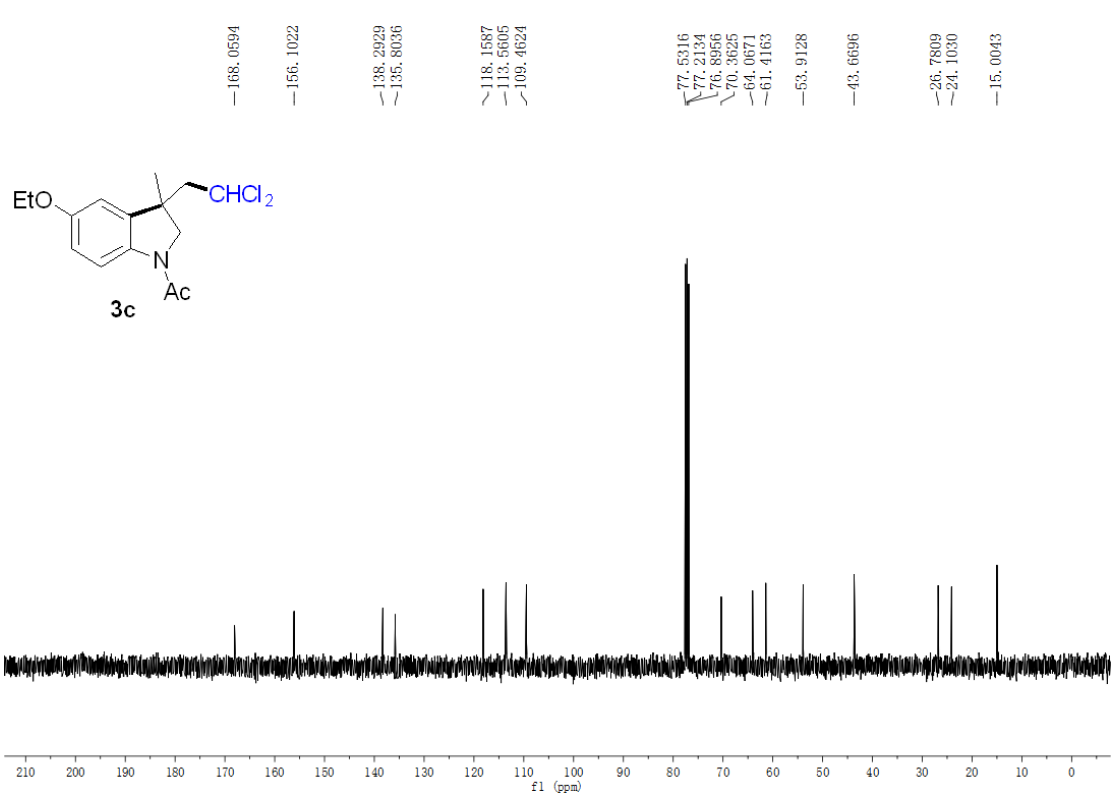
6.09
f1 (ppm)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70.3625
64.0671
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43.6696
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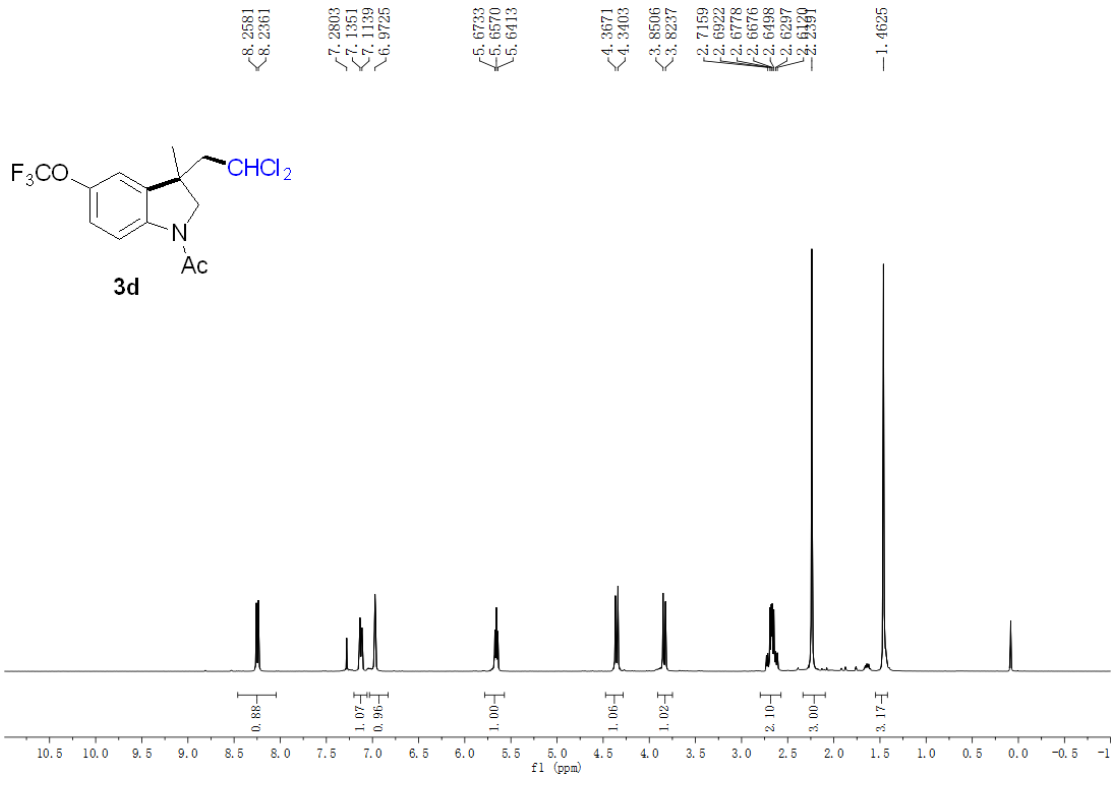
EtO
CHCl2
N
Ac
3c
f1 (ppm)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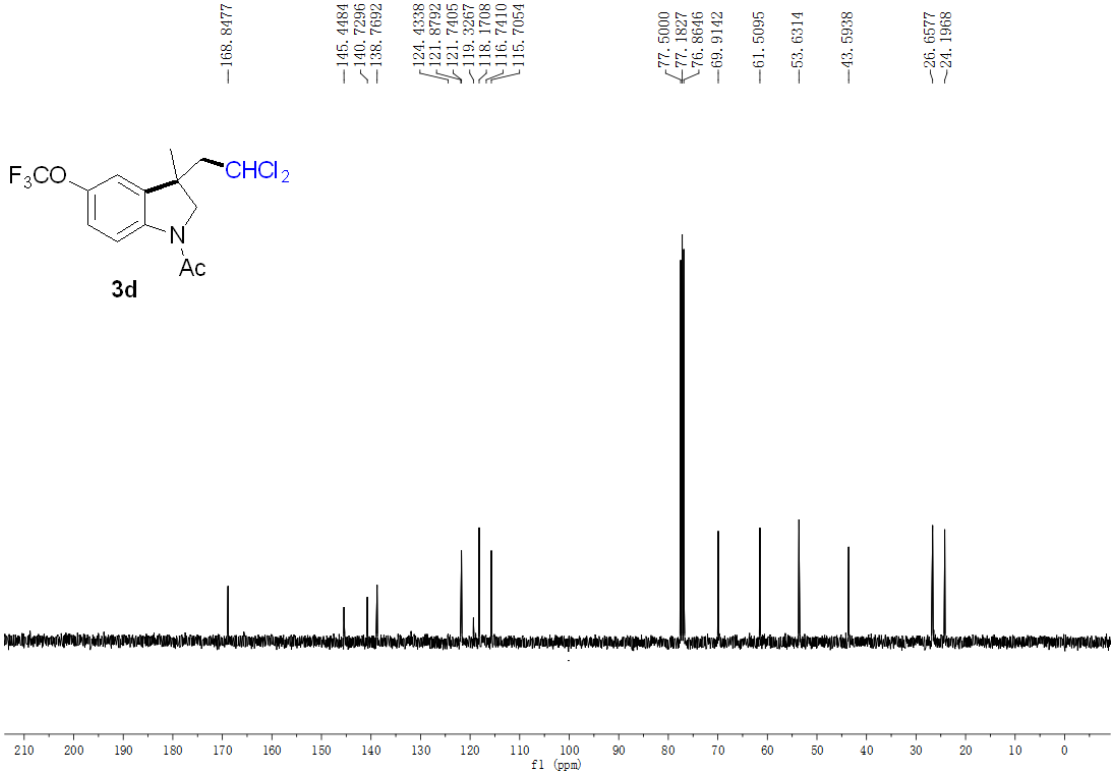
f1 (ppm)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Ac
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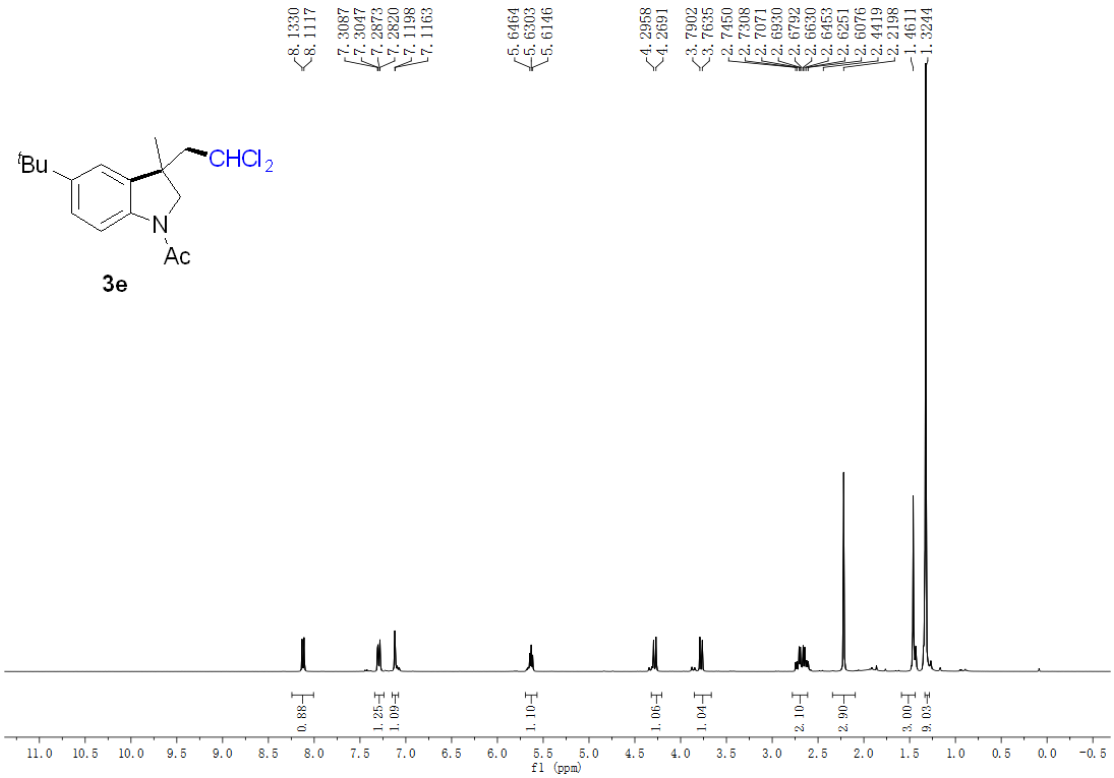
f1 (ppm)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4.2958
4.2691
3.7902
3.7635
2.7450
2.7308
2.7071
2.6930
2.6792
2.6630
2.6453
2.6251
2.6076
2.4419
2.2198
1.4611
1.3244
tBu
CHCl2
N
Ac
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1.06
1.04
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2.90
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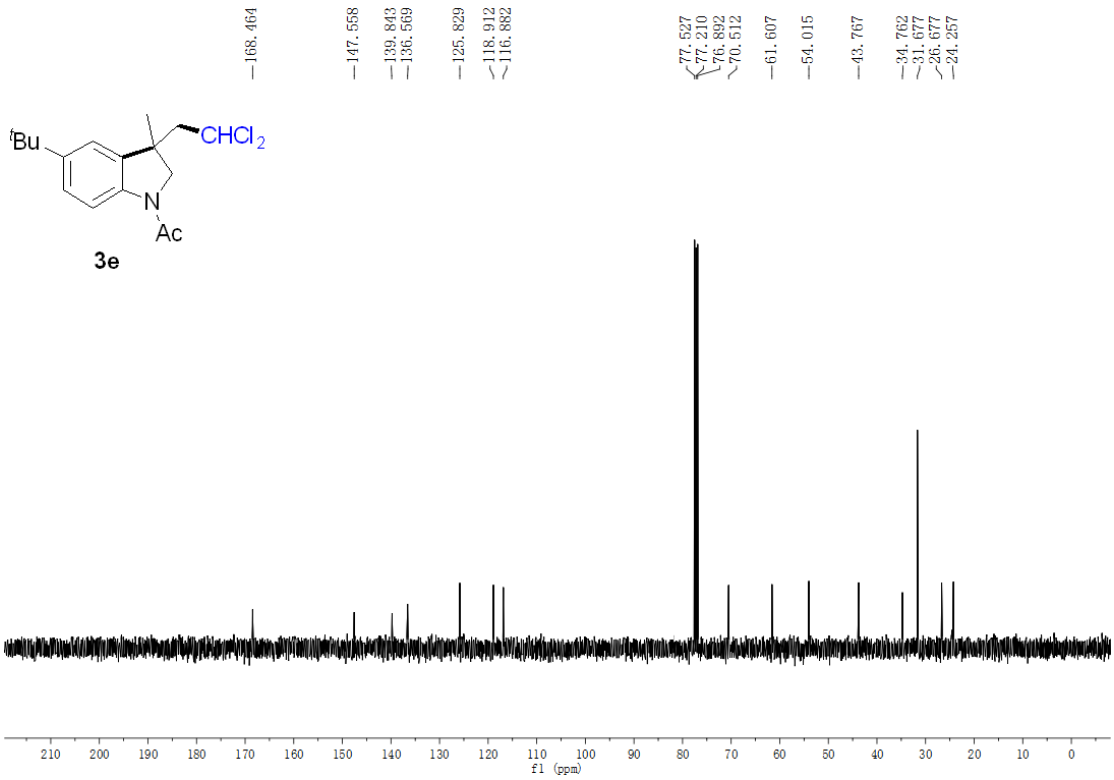
f1 (ppm)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Ac
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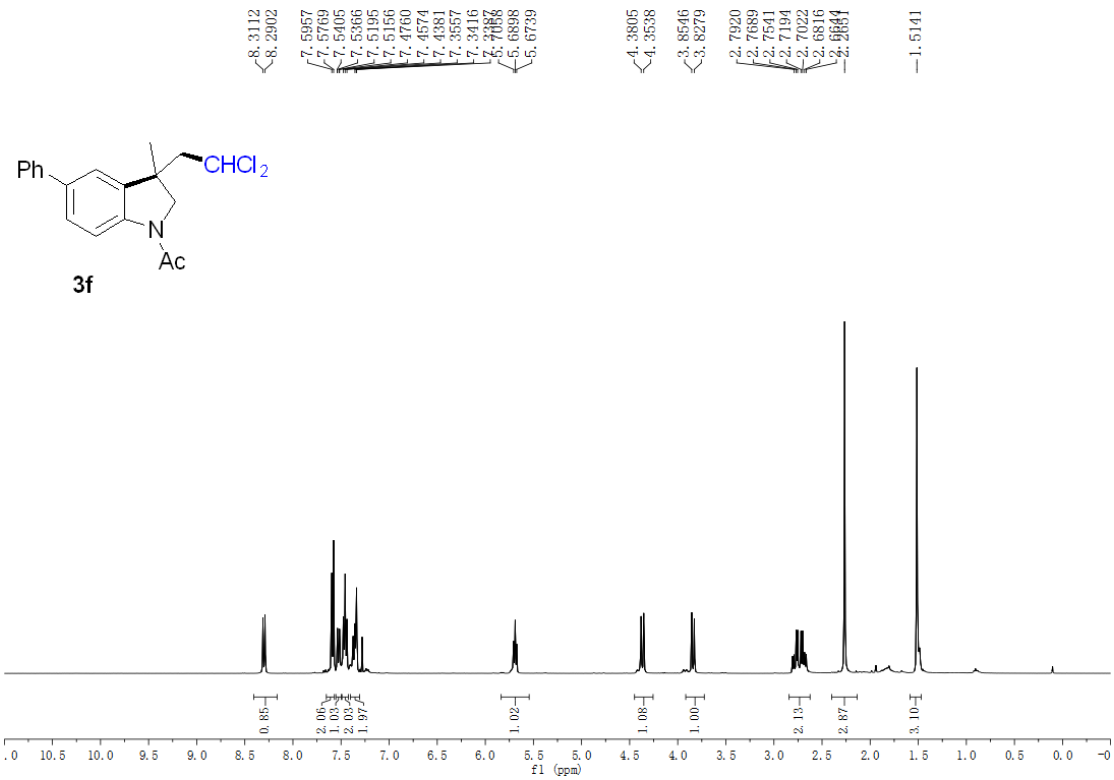
f1 (ppm)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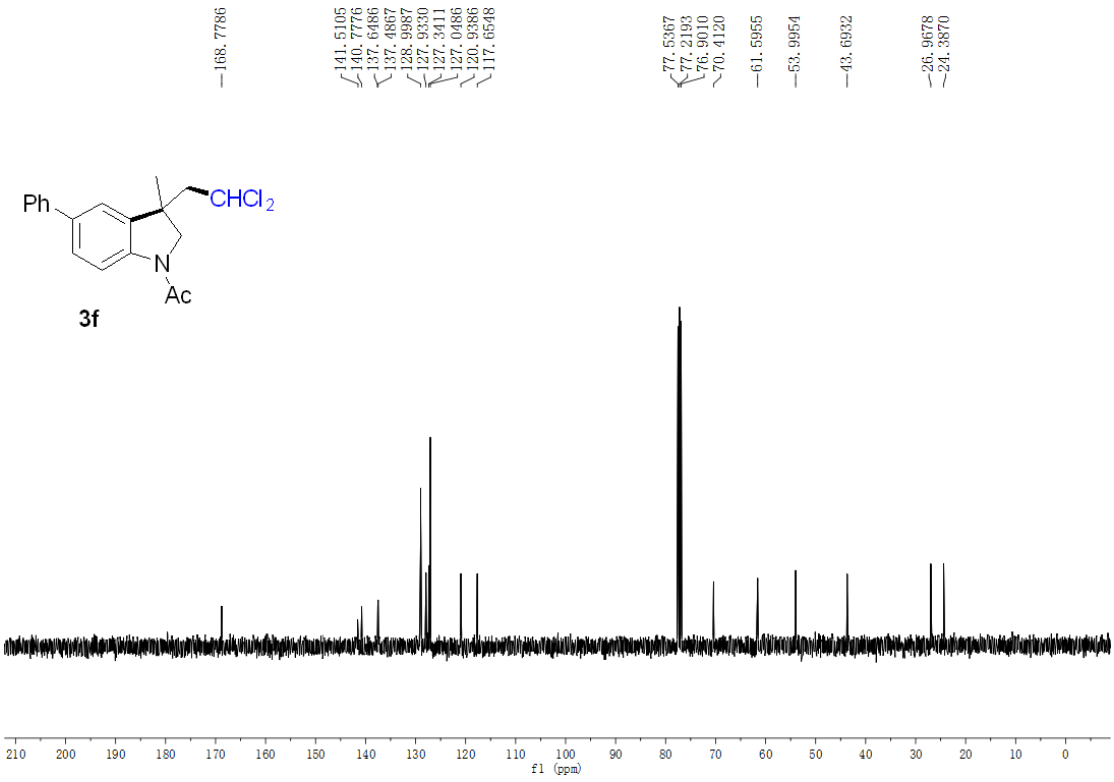
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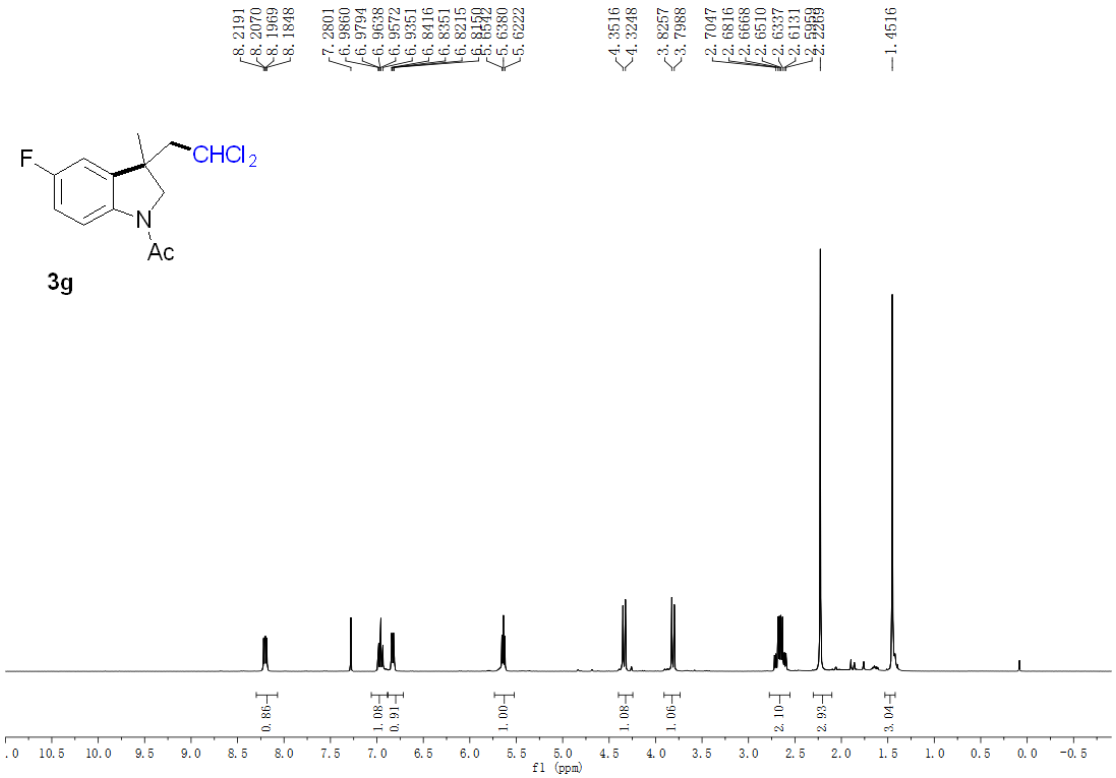
Ac
3f
f1 (ppm)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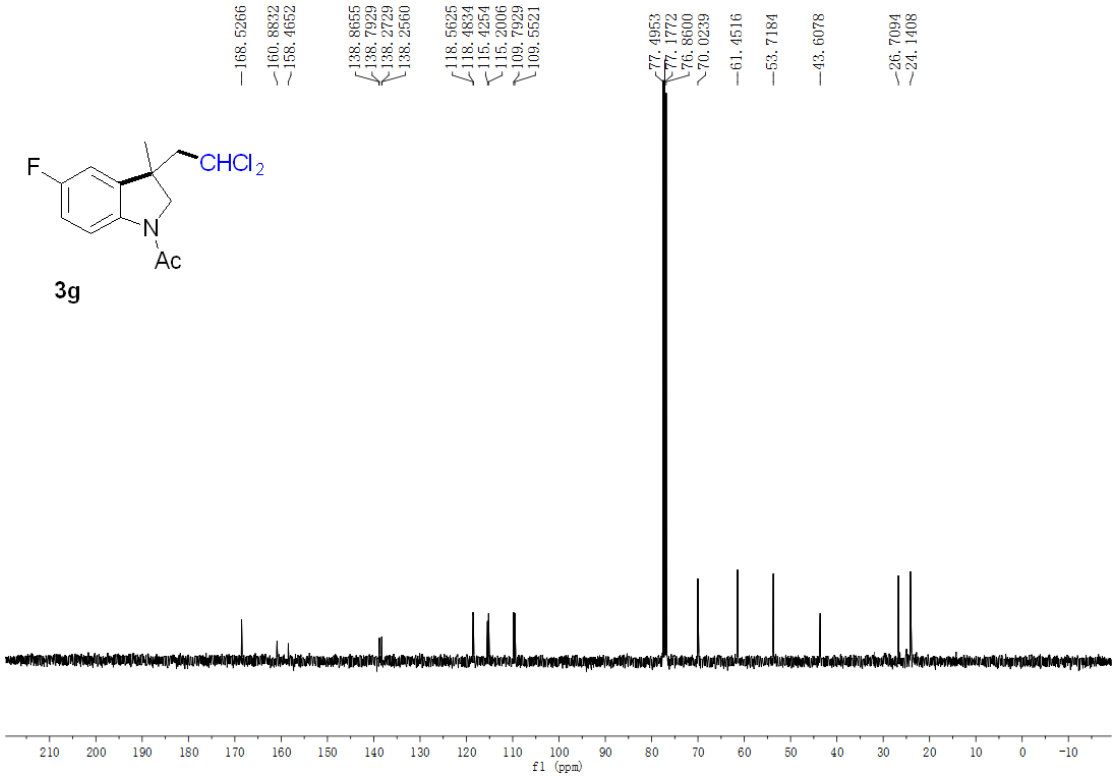
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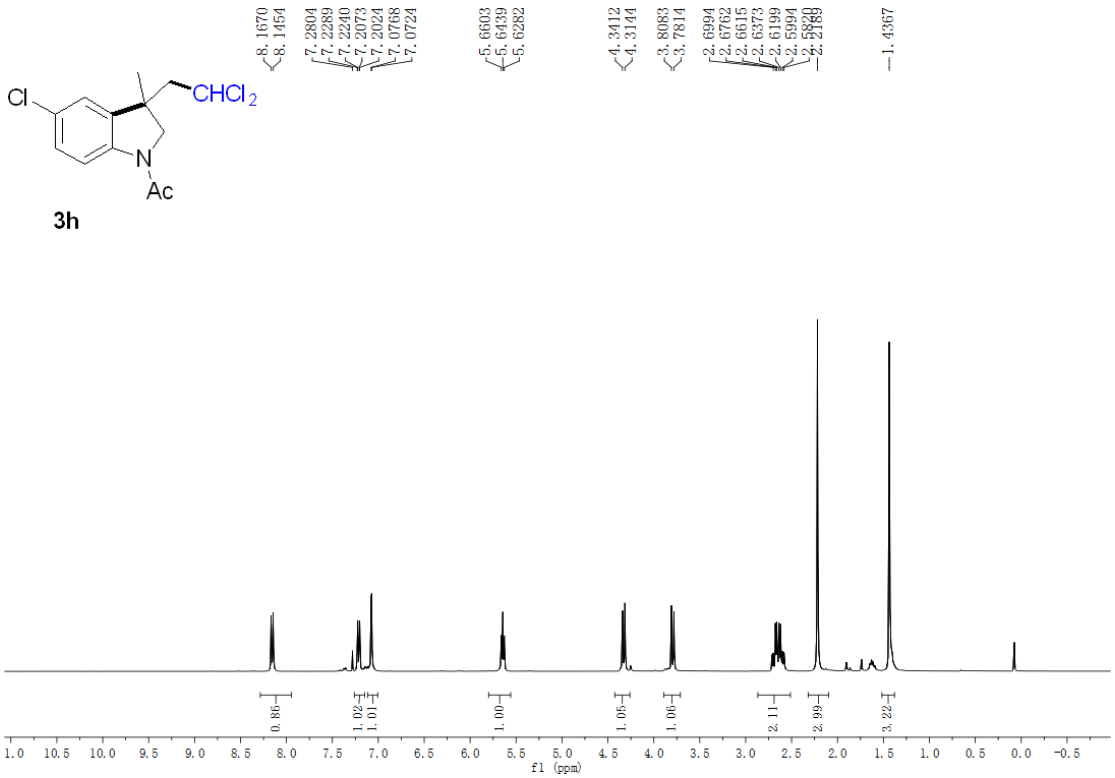
Ac
3g
f1 (ppm)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CHCl2
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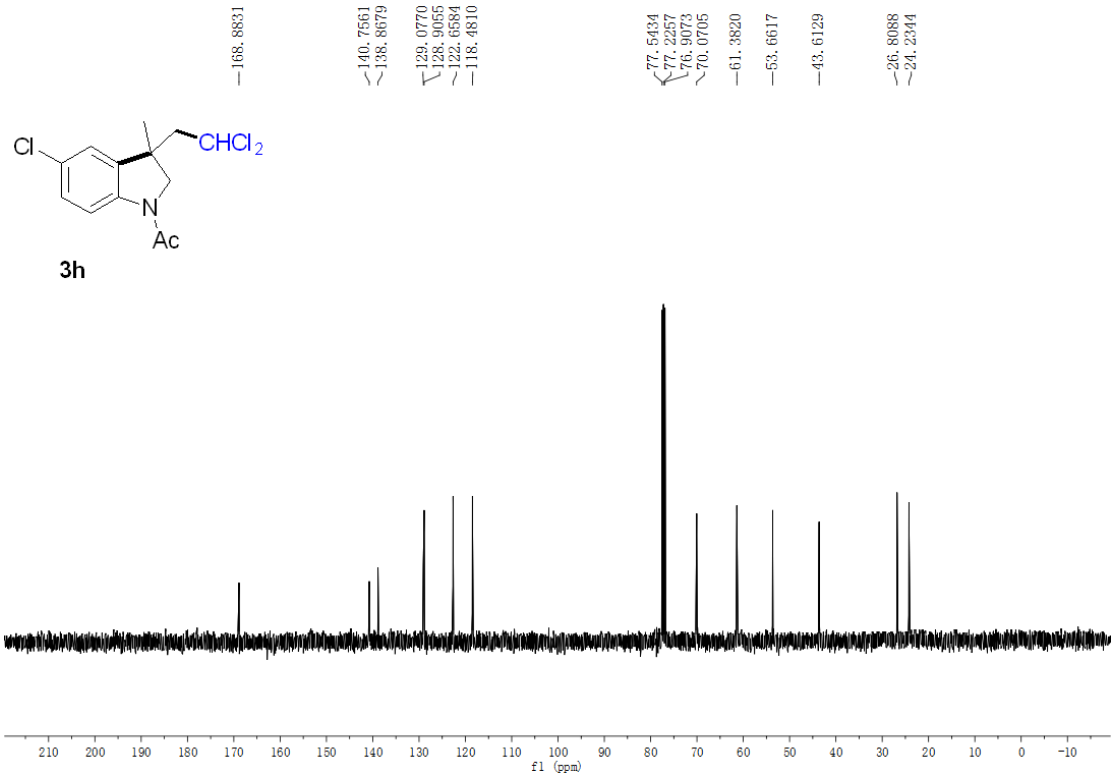
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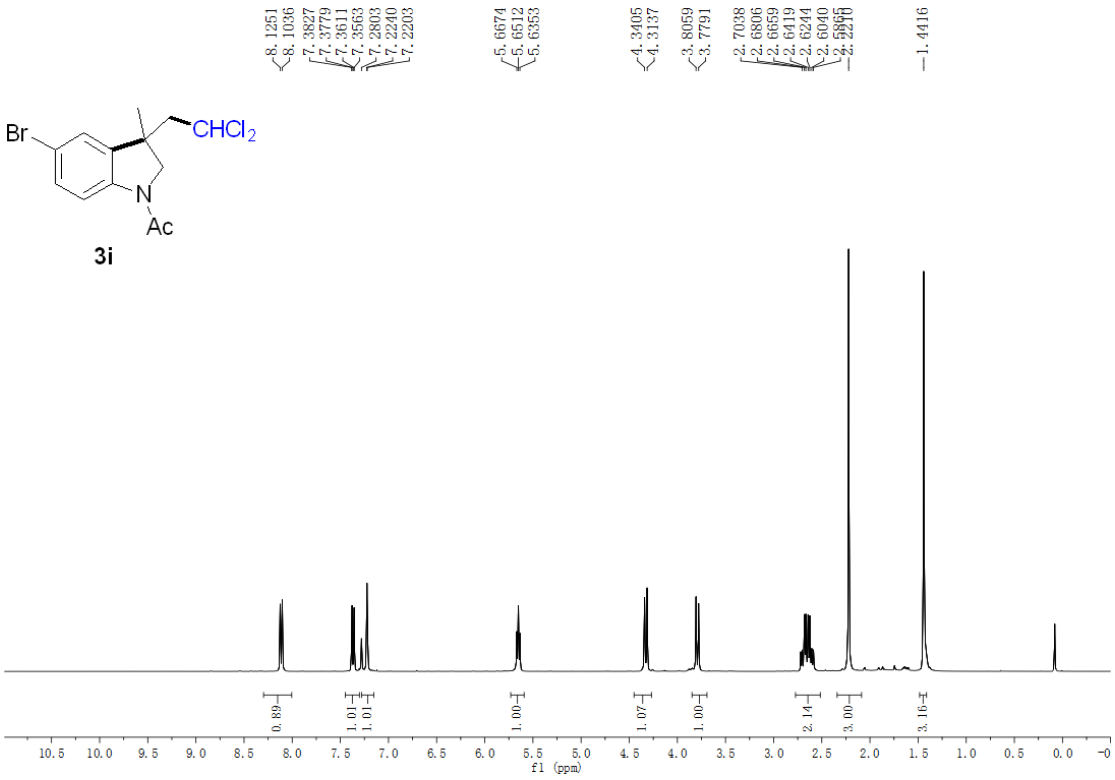
3h
f1 (ppm)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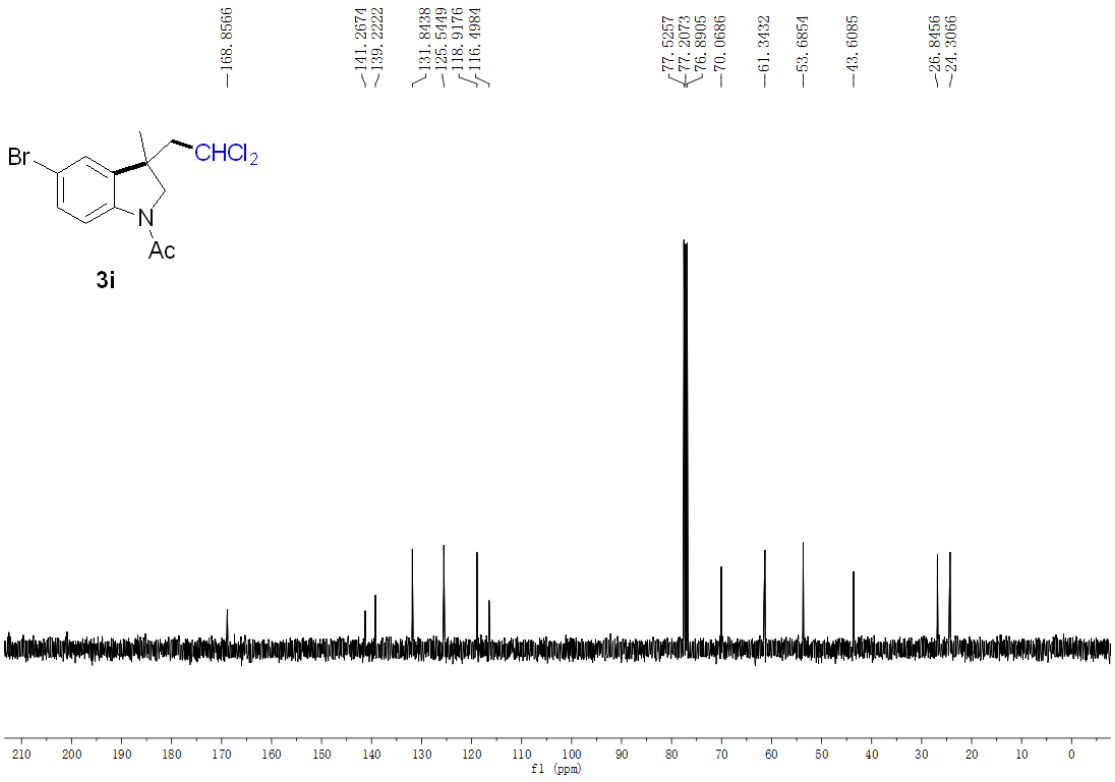
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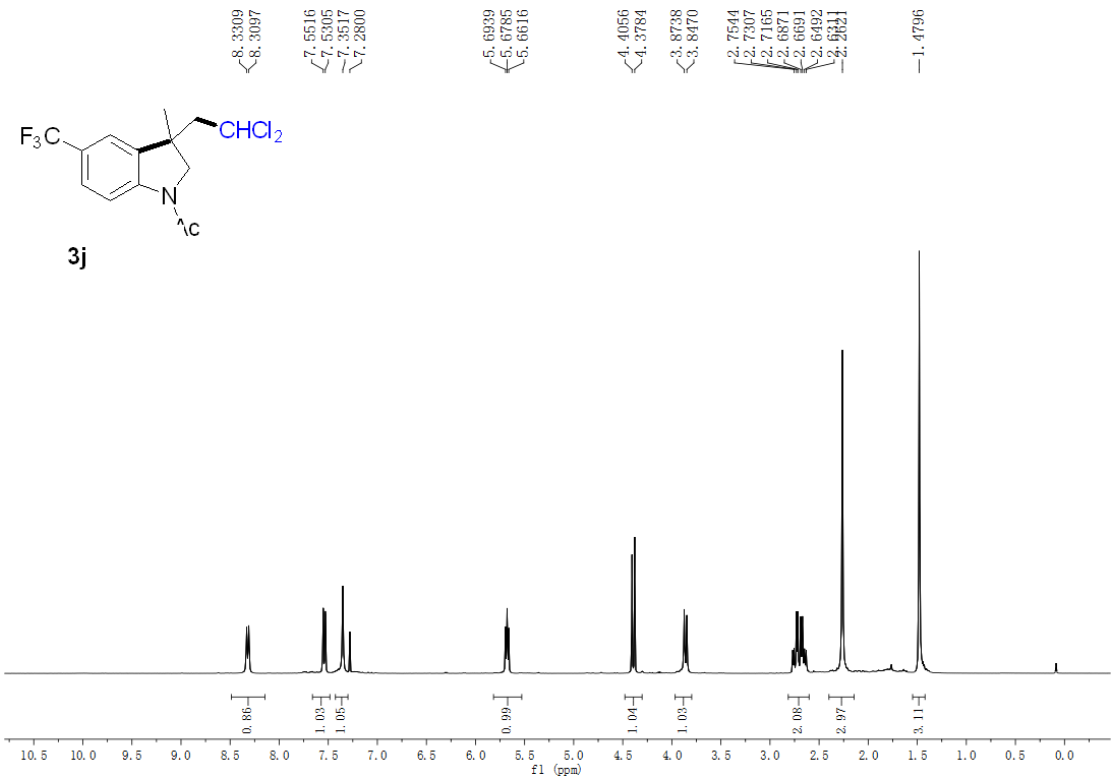
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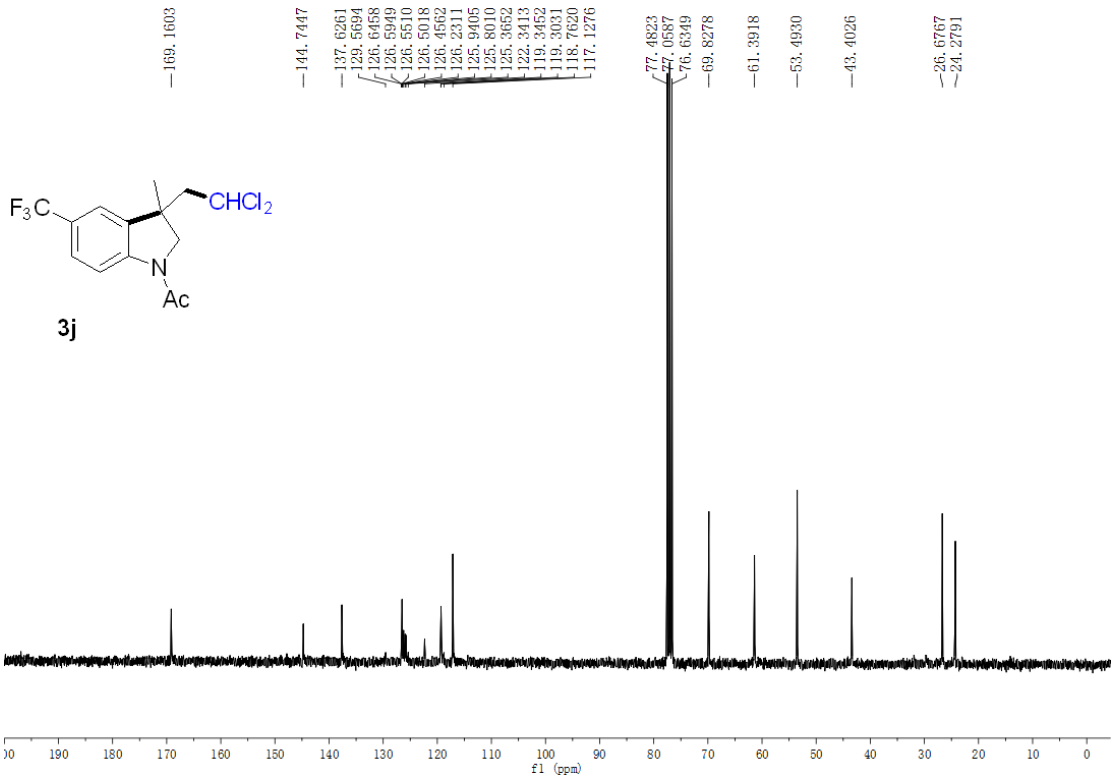
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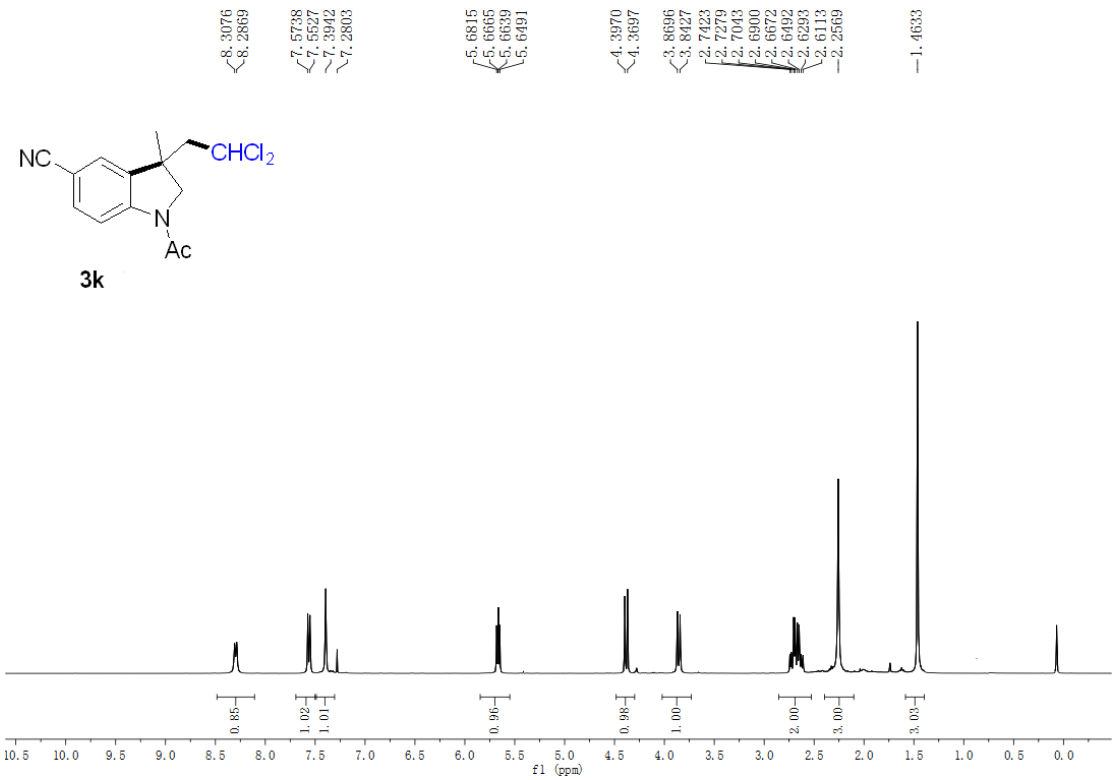
N
Ac
3j
f1 (ppm)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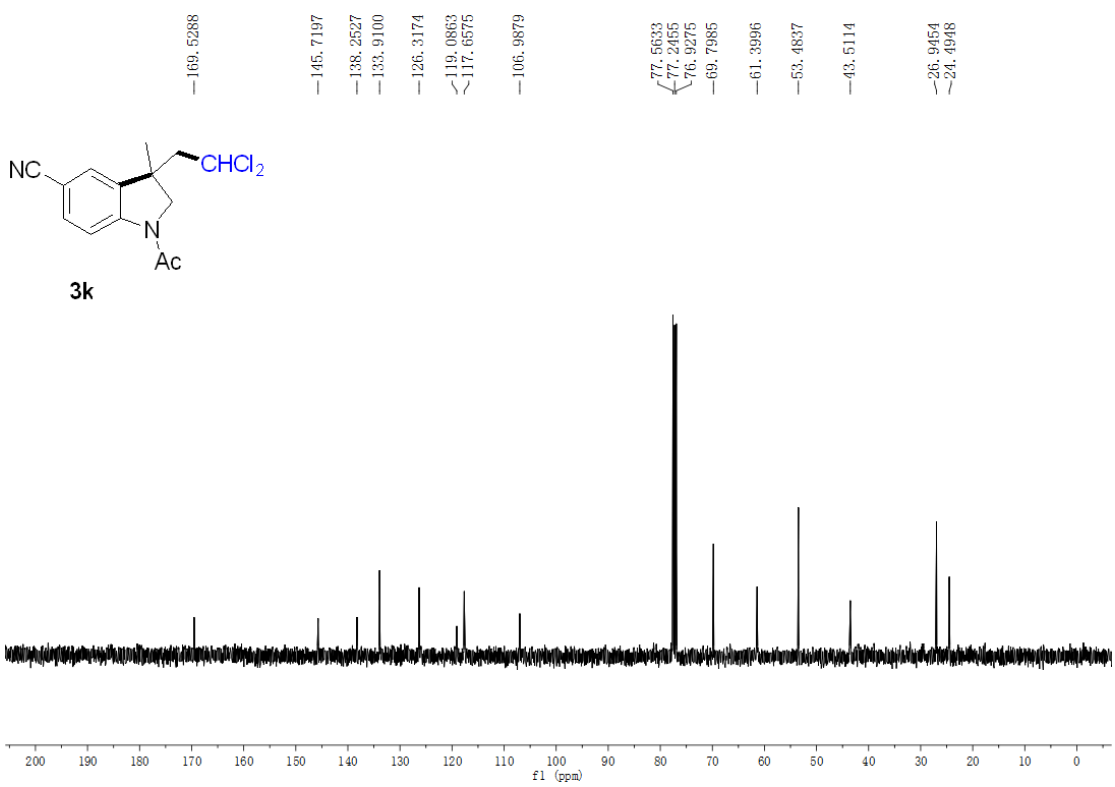
8.2869
7.5738
7.5527
7.3942
7.2803
5.6815
5.6665
5.6639
5.6491
4.3970
4.3697
3.8696
3.8427
2.7423
2.7279
2.7043
2.6900
2.6672
2.6492
2.6293
2.6113
2.2569
1.4633
NC
CHCl2
N
Ac
3k
0.85
1.02
1.01
0.96
0.98
1.00
2.00
3.00
3.03
f1 (ppm)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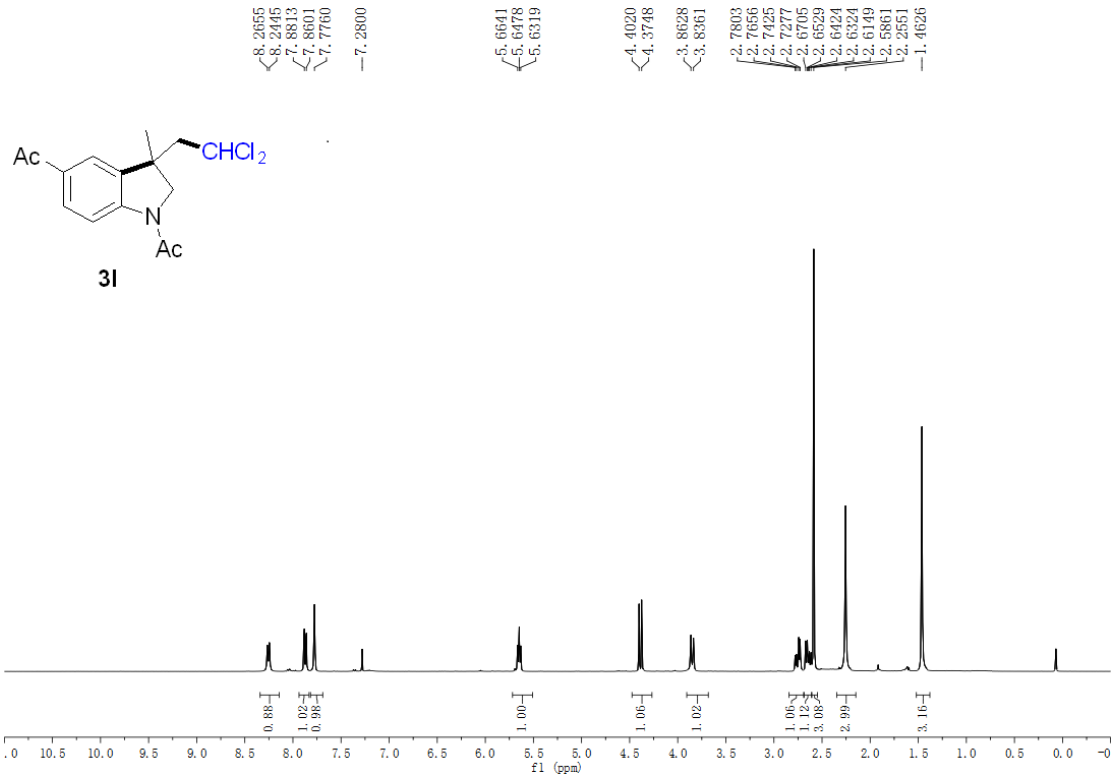
145.7197
138.2527
133.9100
126.3174
119.0863
117.6575
106.9879
77.5633
77.2455
76.9275
69.7985
61.3996
53.4837
43.5114
26.9454
24.4948
NC
CHCl2
N
Ac
3k
f1 (ppm)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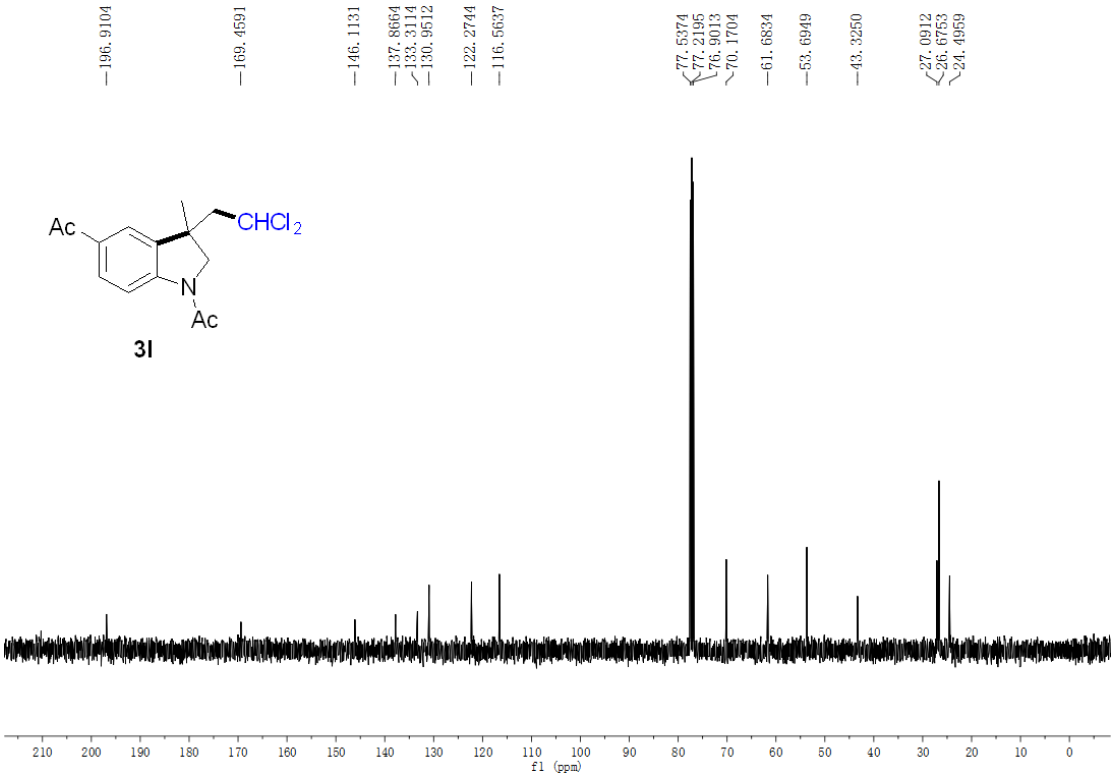
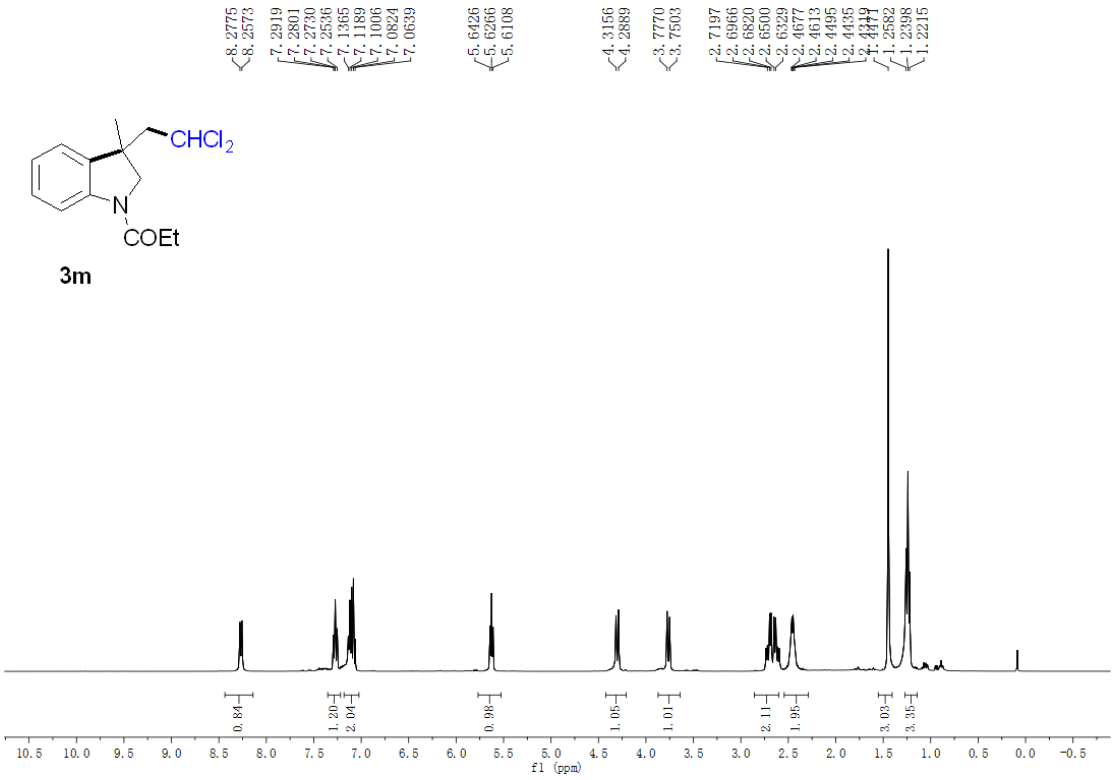
8.2445
7.8813
7.8601
7.7760
7.2800
5.6641
5.6478
5.6319
4.4020
4.3748
3.8628
3.8361
2.7803
2.7656
2.7425
2.7277
2.6705
2.6529
2.6424
2.6324
2.6149
2.5861
2.2551
1.4626
Ac
CHCl2
N
Ac
3l
0.88
1.02
0.98
1.00
1.06
1.02
1.06
1.12
3.08
2.99
3.16
f1 (ppm)


196.9104
169.4591
146.1131
137.8664
133.3114
130.9512
122.2744
116.5637
77.5374
77.2195
76.9013
70.1704
61.6834
53.6949
43.3250
27.0912
26.6753
24.4959
Ac
CHCl2
N
Ac
3l
f1 (ppm)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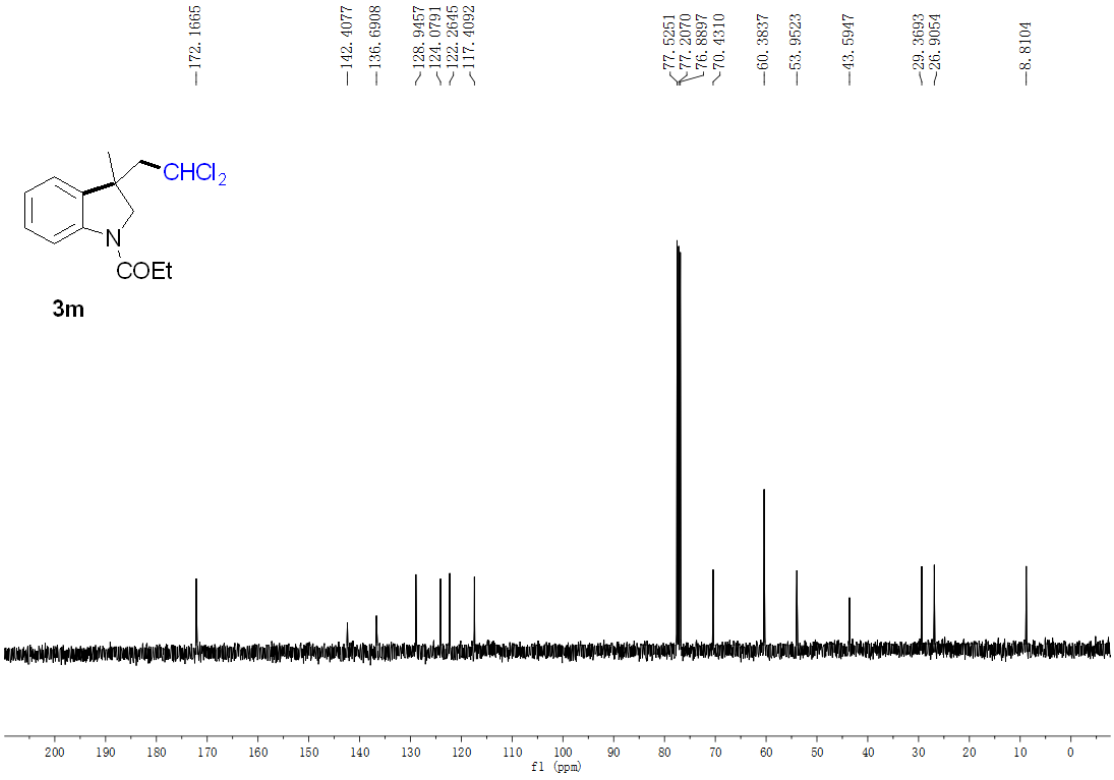
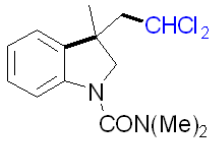
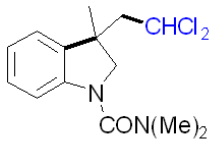
8.2573
7.2919
7.2801
7.2730
7.2536
7.1365
7.1189
7.1006
7.0824
7.0639
5.6426
5.6266
5.6108
4.3156
4.2889
3.7770
3.7503
2.7197
2.6966
2.6820
2.6500
2.6329
2.4677
2.4613
2.4495
2.4435
1.2582
1.2398
1.2215
CHCl2
N
COEt
3m
0.84
1.20
2.04
0.98
1.05
1.01
2.11
1.95
3.03
3.35
f1 (ppm)


172.1665
142.4077
136.6908
128.9457
124.0791
122.2645
117.4092
77.5251
77.2070
76.8897
70.4310
60.3837
53.9523
43.5947
29.3693
26.9054
8.8104
CHCl2
N
COEt
3m
f1 (ppm)


CHCl2
N
CON(Me)2

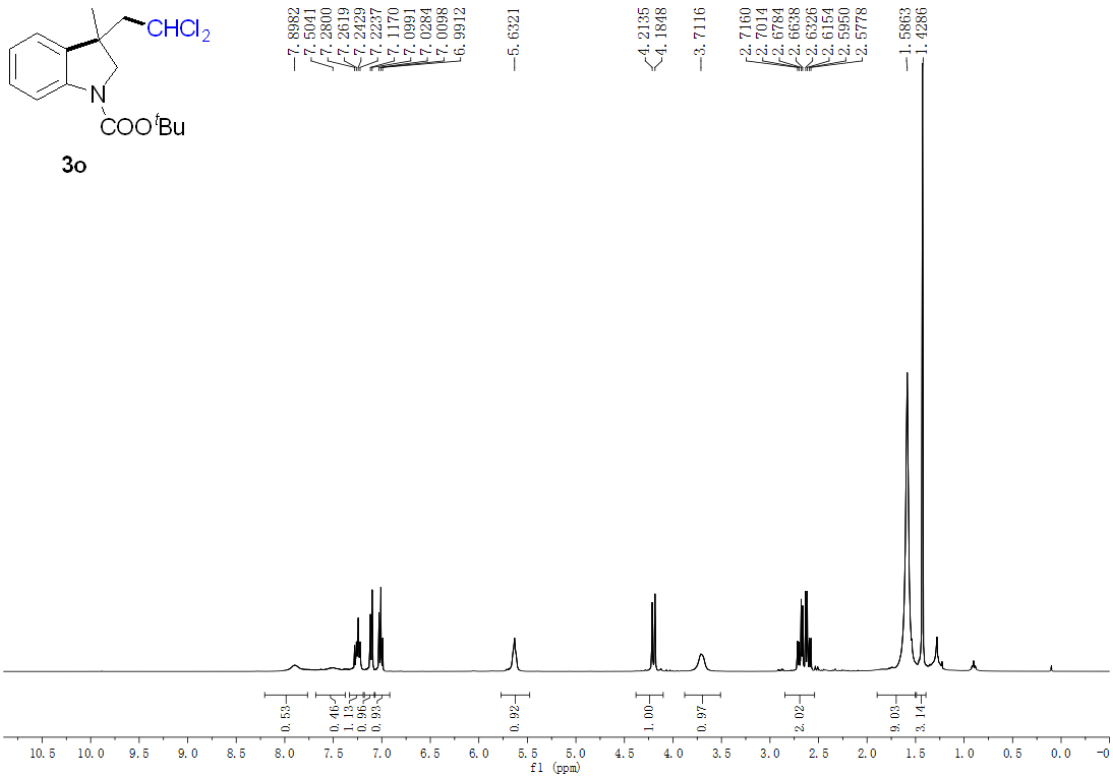
3n

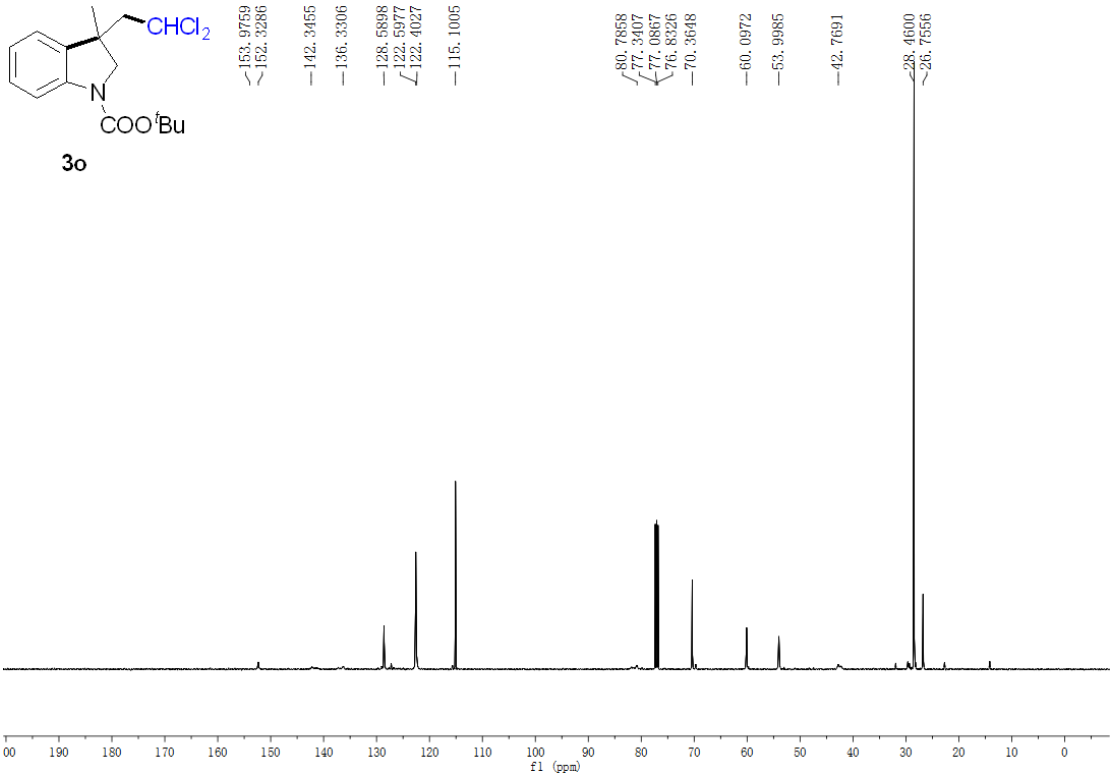
7.2801
7.2138
7.1957
7.1753
7.1194
7.1018
6.9637
6.9481
6.9288
5.7326
5.7197
5.7142
5.7013
3.9954
3.9689
3.7239
3.6974
2.9483
2.7175
2.7047
2.6799
2.6671
2.6216
2.6030
2.5840
2.5655
1.4221
1.01
0.96
1.94
0.97
1.00
1.00
6.01
2.02
3.02
f1 (ppm)

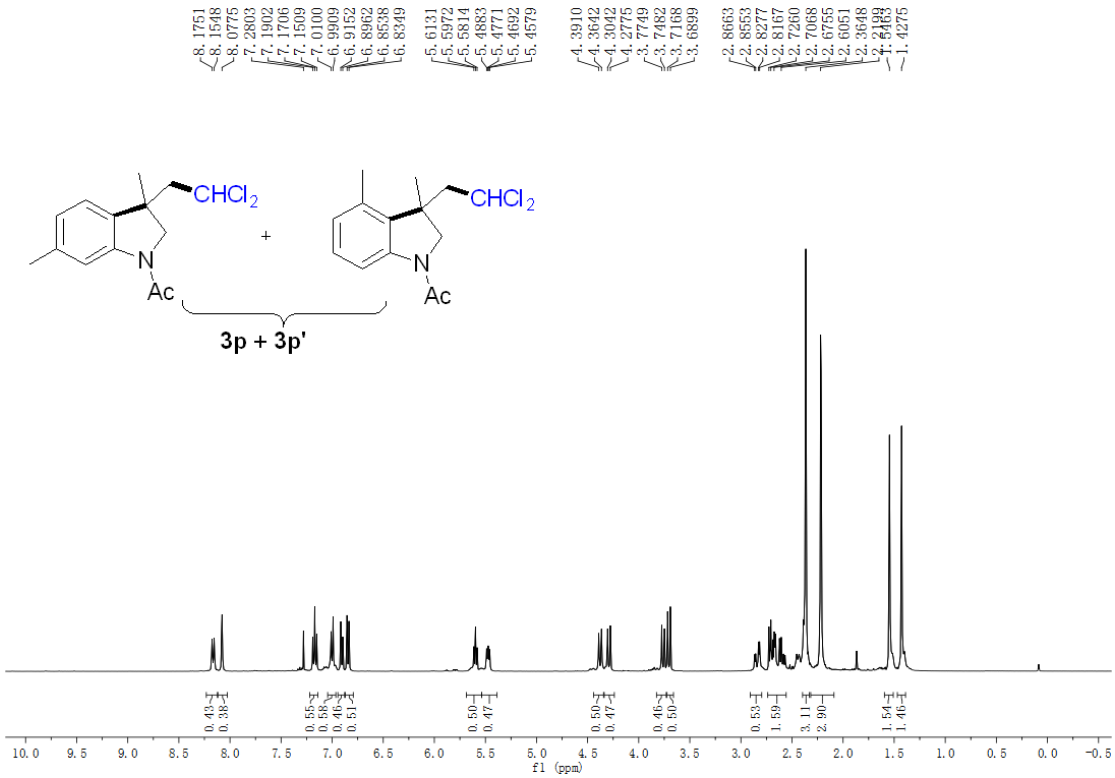

CHCl2
N
CON(Me)2

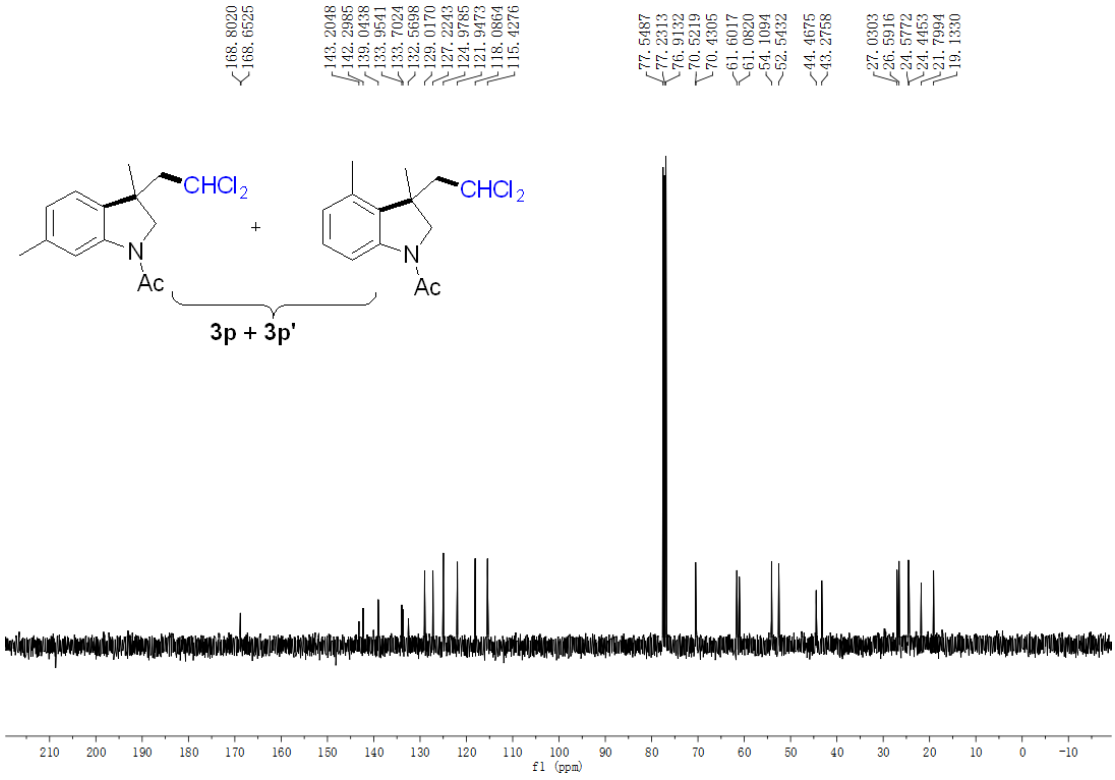
3n

159.8632
143.5891
136.9158
128.2751
122.8164
121.9777
113.7096
77.5719
77.2541
76.9356
70.5179
62.3937
53.2833
42.9779
38.2643
25.3058
f1 (ppm)


CHCl2
N
COO'Bu
3o
7.8982
7.5041
7.2800
7.2619
7.2429
7.2237
7.1170
7.0991
7.0284
7.0098
6.9912
5.6321
4.2135
4.1848
3.7116
2.7160
2.7014
2.6784
2.6638
2.6326
2.6154
2.5950
2.5778
1.5863
1.4286
0.53
0.46
1.13
0.96
0.93
0.92
1.00
0.97
2.02
9.03
3.14
f1 (ppm)


CHCl2
N
COO'Bu
3o
153.9759
152.3286
142.3455
136.3306
128.5898
122.5977
122.4027
115.1005
80.7858
77.3407
77.0867
76.8326
70.3648
60.0972
53.9985
42.7691
28.4600
26.7556
f1 (ppm)


8.1751
8.1548
8.0775
7.2803
7.1902
7.1706
7.1509
7.0100
6.9909
6.9152
6.8962
6.8538
6.8349
5.6131
5.5972
5.5814
5.4883
5.4771
5.4692
5.4579
4.3910
4.3642
4.3042
4.2775
3.7749
3.7482
3.7168
3.6899
2.8663
2.8553
2.8277
2.8167
2.7260
2.7068
2.6755
2.6051
2.3648
1.5483
1.4275
CHCl2
N
Ac
+
3p + 3p'
0.43
0.38
0.55
0.58
0.46
0.51
0.50
0.47
0.50
0.47
0.46
0.50
0.53
1.59
3.11
2.90
1.54
1.46
f1 (ppm)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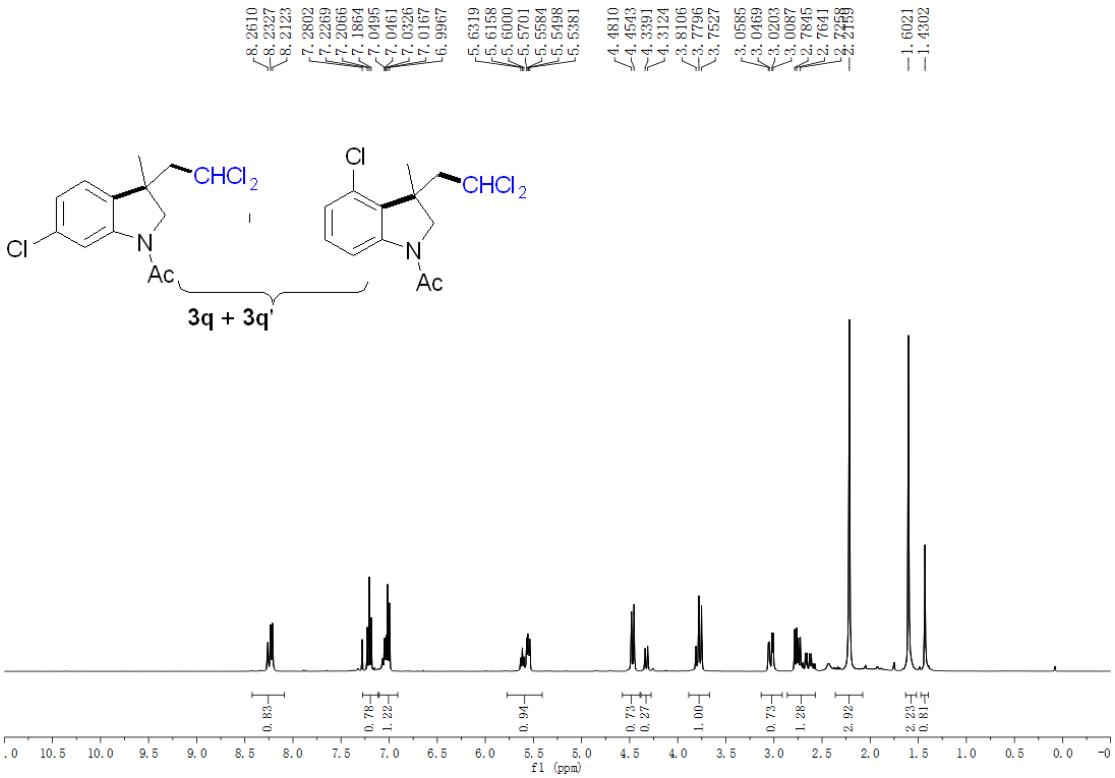
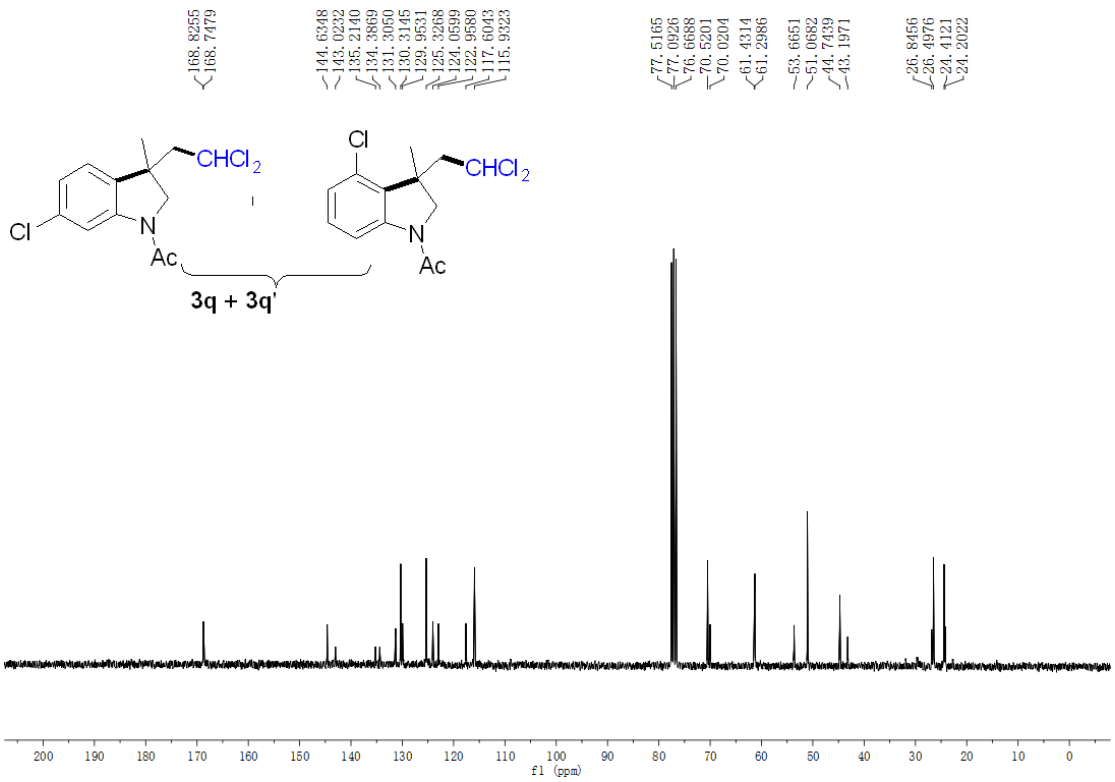
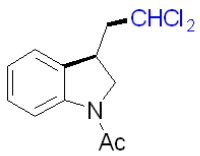
168.6525
143.2048
142.2985
139.0438
133.9541
133.7024
132.5698
129.0170
127.2243
124.9785
121.9473
118.0864
115.4276
77.5487
77.2313
76.9132
70.5219
70.4305
61.6017
61.0820
54.1094
52.5432
44.4675
43.2758
27.0303
26.5916
24.5772
24.4453
21.7994
19.1330
CHCl2
N
Ac
+
3p + 3p'
f1 (ppm)

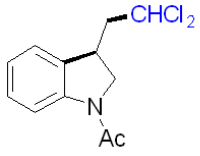

8.2610
8.2327
8.2123
7.2802
7.2269
7.2066
7.1864
7.0495
7.0461
7.0326
7.0167
6.9967
5.6319
5.6158
5.6000
5.5701
5.5584
5.5498
5.5381
4.4810
4.4543
4.3391
4.3124
3.8106
3.7796
3.7527
3.0585
3.0469
3.0203
3.0087
2.7845
2.7641
1.6021
1.4302
CHCl2
Cl
N
Ac
3q + 3q'
0.83
0.78
1.22
0.94
0.73
0.27
1.00
0.73
1.28
2.92
2.23
0.81
f1 (ppm)


168.8255
168.7479
144.6348
143.0232
135.2140
134.3869
131.3050
130.3145
129.9531
125.3268
124.0599
122.9580
117.6043
115.9323
77.5165
77.0926
76.6688
70.5201
70.0204
61.4314
61.2986
53.6651
51.0682
44.7439
43.1971
26.8456
26.4976
24.4121
24.2022
CHCl2
Cl
N
Ac
3q + 3q'
f1 (ppm)


CHCl2
N
Ac

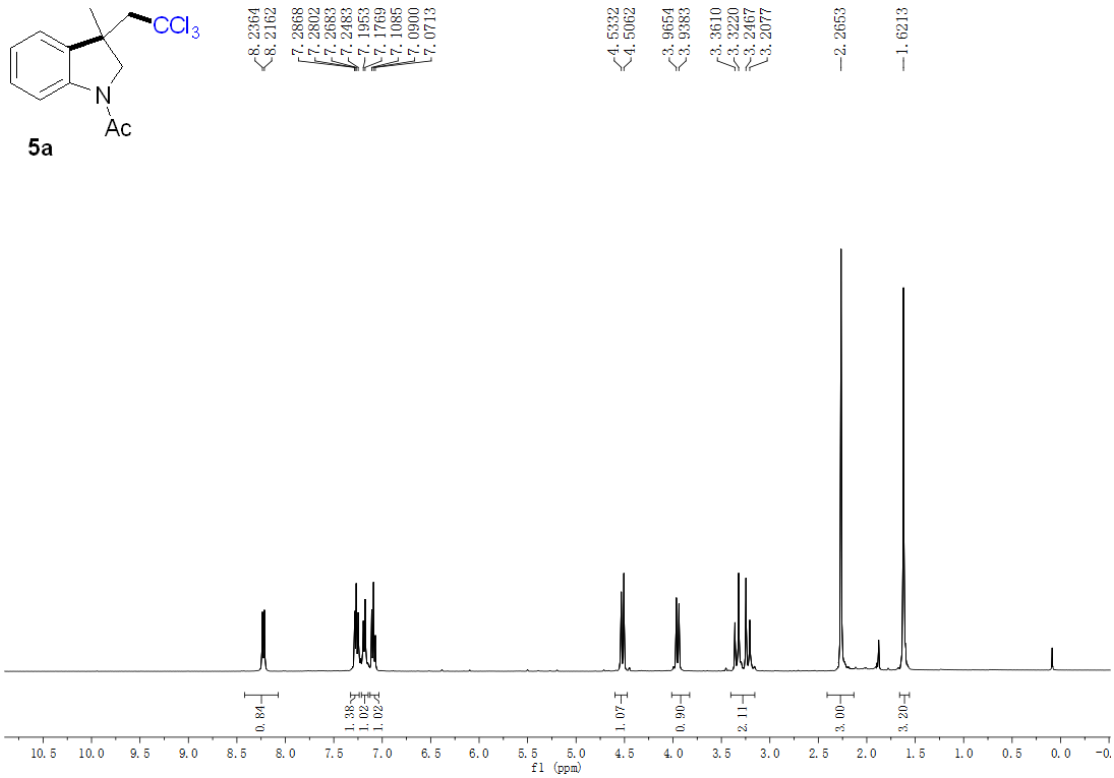
3r

8.2402
8.2200
7.2806
7.2654
7.2458
7.2166
7.1977
7.1757
7.0909
7.0724
7.0539
5.8930
5.8775
5.8620
4.3000
4.2739
4.2505
3.8407
3.8148
3.8014
3.7351
2.6894
2.6531
2.6389
2.5355
2.5159
2.4986
2.4794
2.2528
0.75
1.29
1.14
0.95
1.08
1.04
2.02
1.01
1.32
3.00
f1 (ppm)


CHCl2
N
Ac

3r

168.6406
142.6225
132.5879
128.8180
124.0452
123.8914
117.4272
77.5267
77.2090
76.8915
71.3637
55.2325
48.9902
37.7870
24.4020
f1 (ppm)


CCl3
N
Ac
5a
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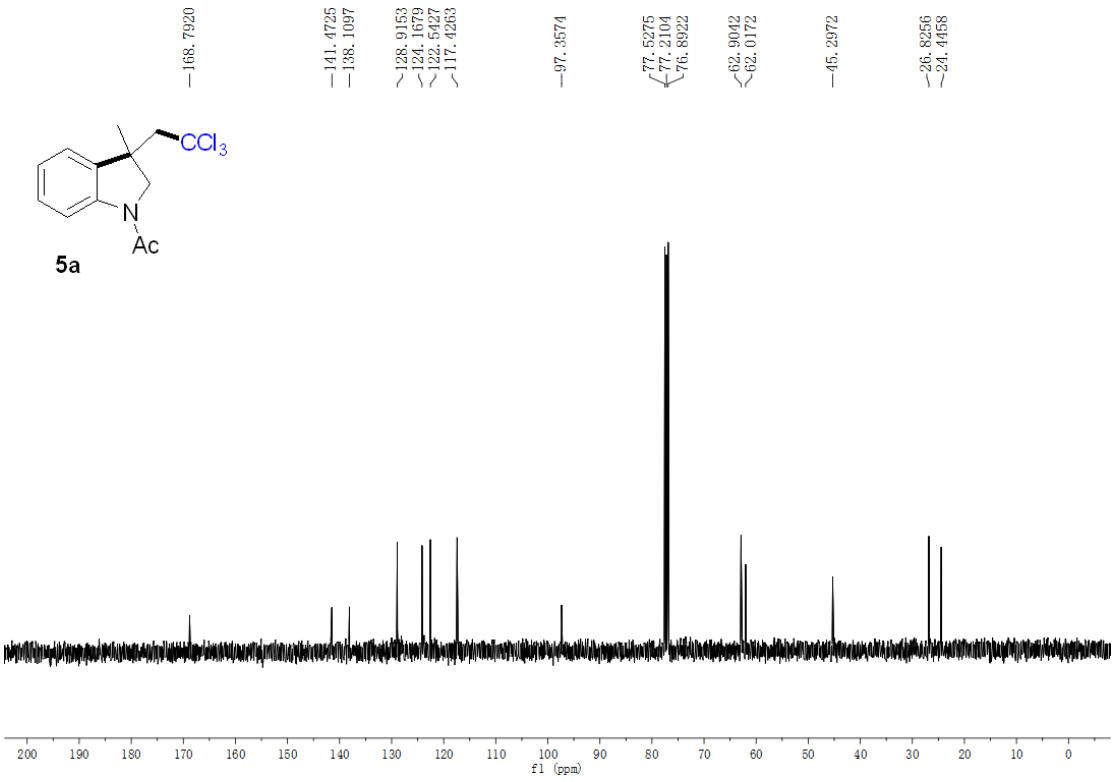
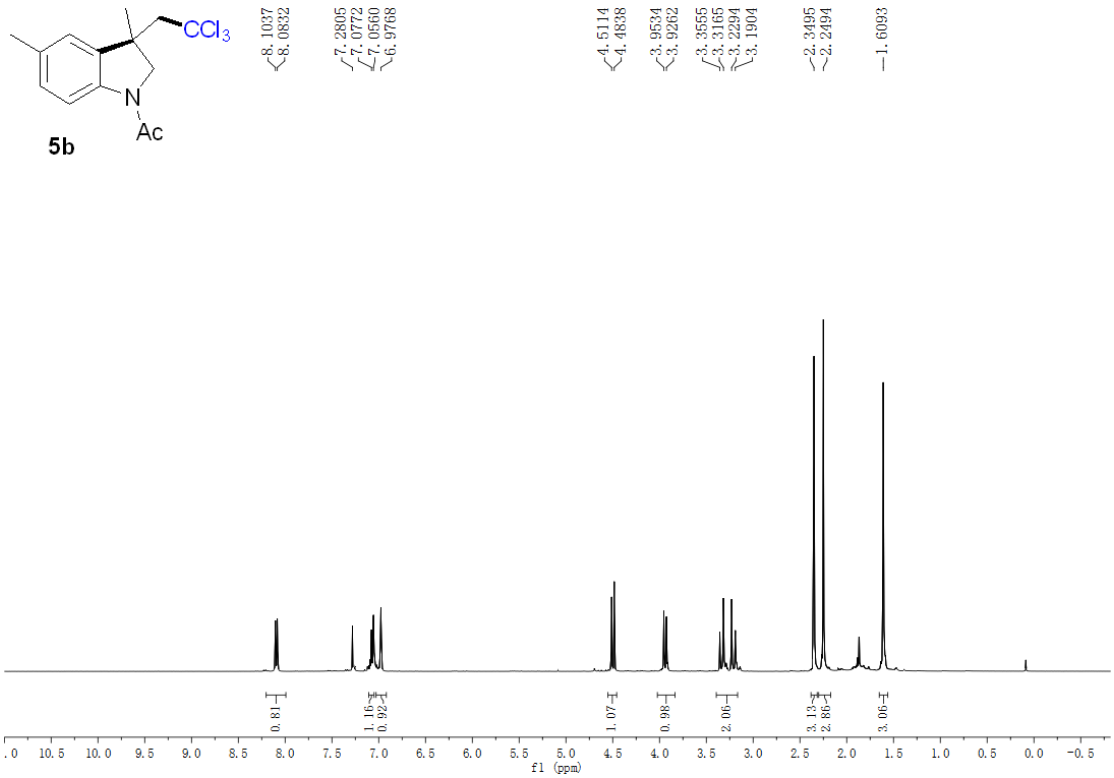
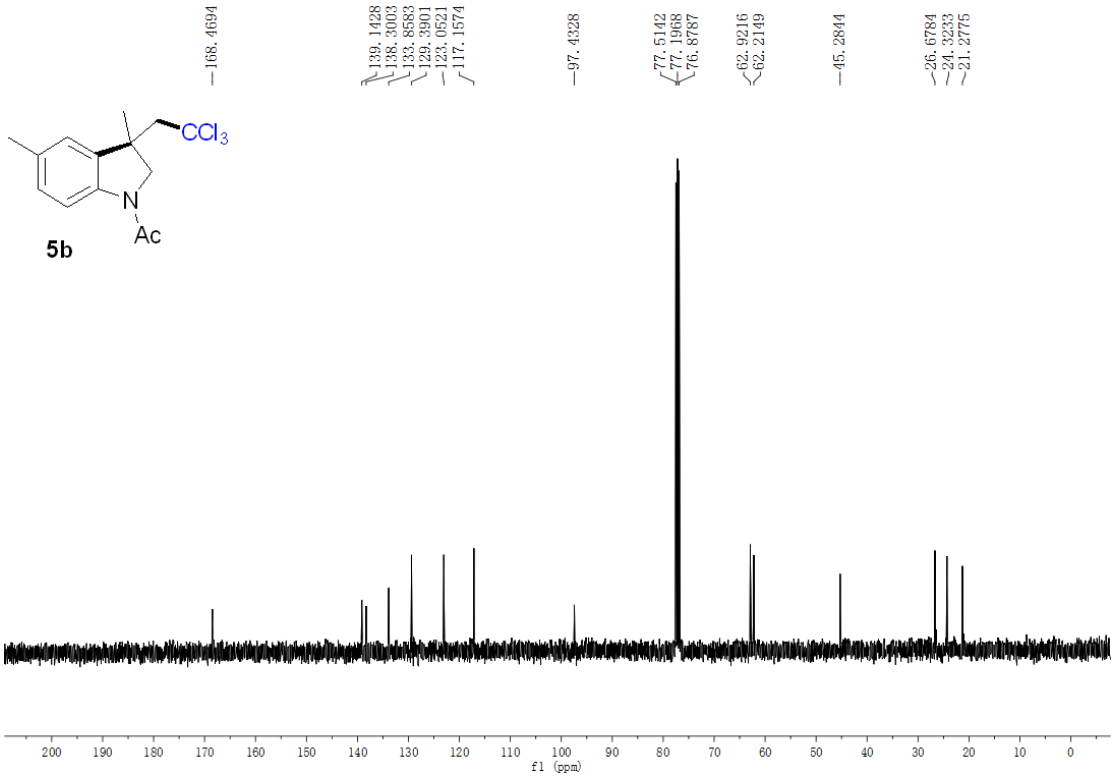
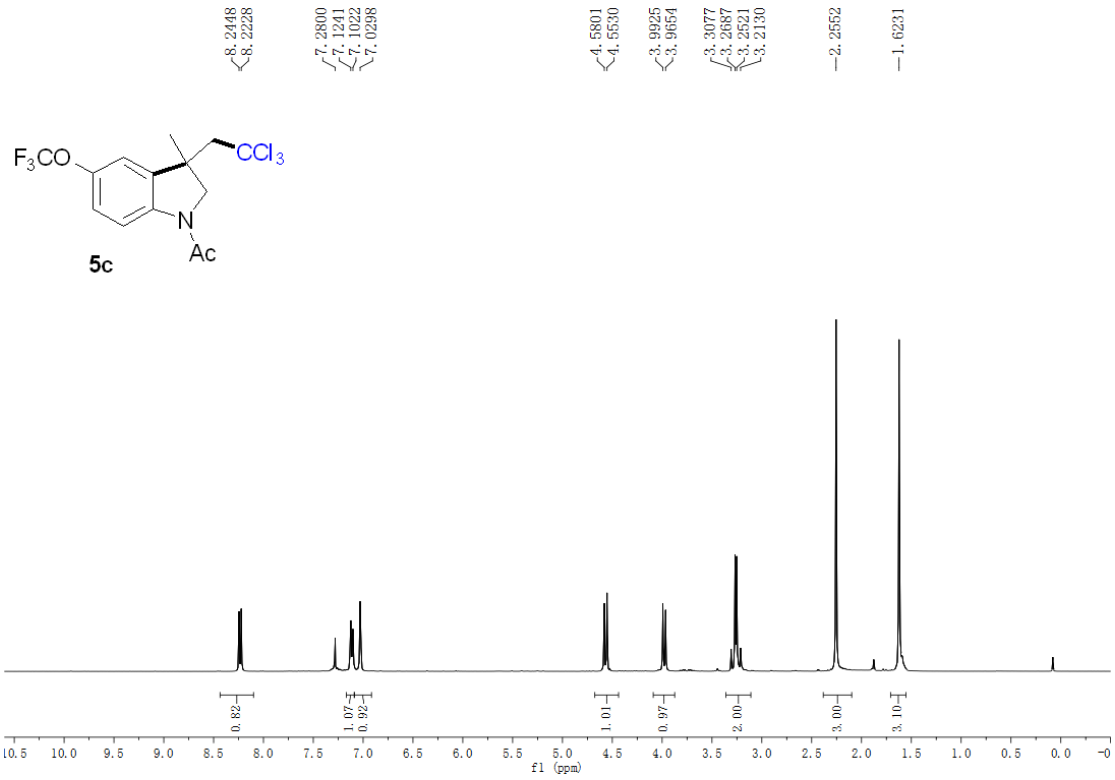
8.2162
7.2868
7.2802
7.2683
7.2483
7.1953
7.1769
7.1085
7.0900
7.0713
4.5332
4.5062
3.9654
3.9383
3.3610
3.3220
3.2467
3.2077
2.2653
1.6213
0.84
1.38
1.02
1.02
1.07
0.90
2.11
3.00
3.20
f1 (ppm)


CCl3
N
Ac
5a
168.7920
141.4725
138.1097
128.9153
124.1679
122.5427
117.4263
97.3574
77.5275
77.2104
76.8922
62.9042
62.0172
45.2972
26.8256
24.4458
f1 (ppm)


5b
8.1037
8.0832
7.2805
7.0772
7.0560
6.9768
4.5114
4.4838
3.9534
3.9262
3.3555
3.3165
3.2294
3.1904
2.3495
2.2494
1.6093
0.81
1.16
0.92
1.07
0.98
2.06
3.13
2.86
3.06
f1 (ppm)


5b
168.4694
139.1428
138.3003
133.8583
129.3901
123.0521
117.1574
97.4328
77.5142
77.1968
76.8787
62.9216
62.2149
45.2844
26.6784
24.3233
21.2775
f1 (ppm)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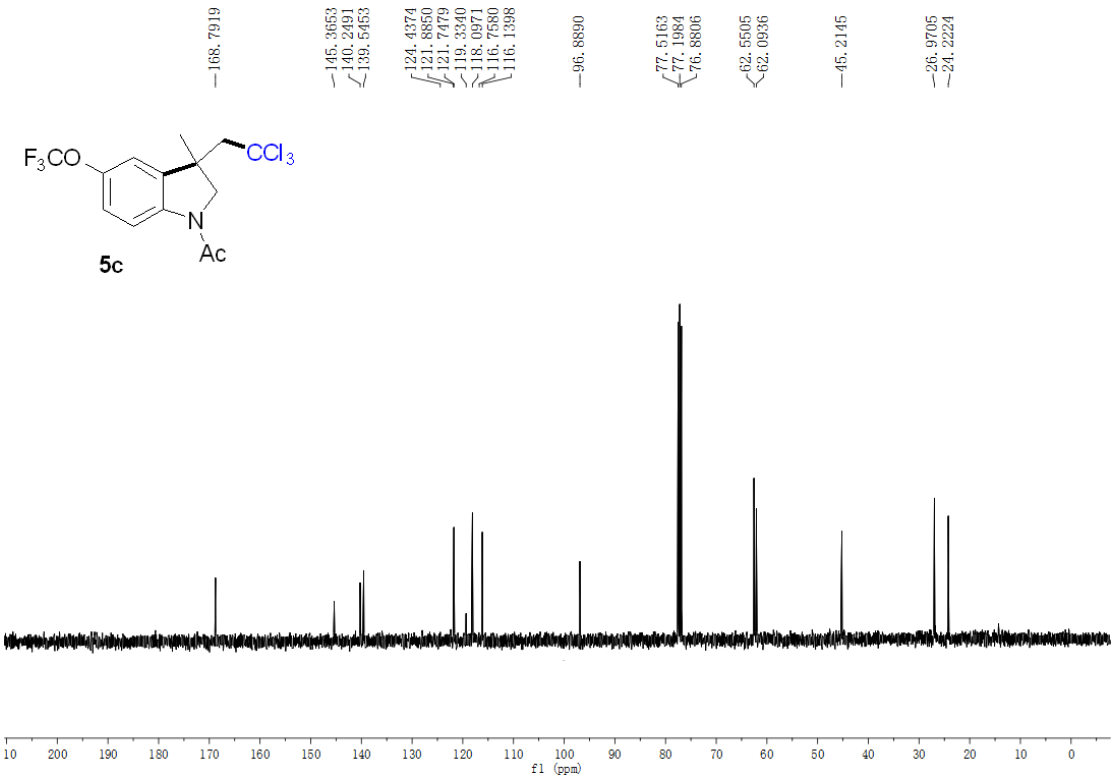
4.5801
4.5530
3.9925
3.9654
3.3077
3.2687
3.2521
3.2130
2.2552
1.6231
F3CO
CCl3
N
Ac
5c
0.82
1.07
0.92
1.01
0.97
2.00
3.00
3.10
f1 (ppm)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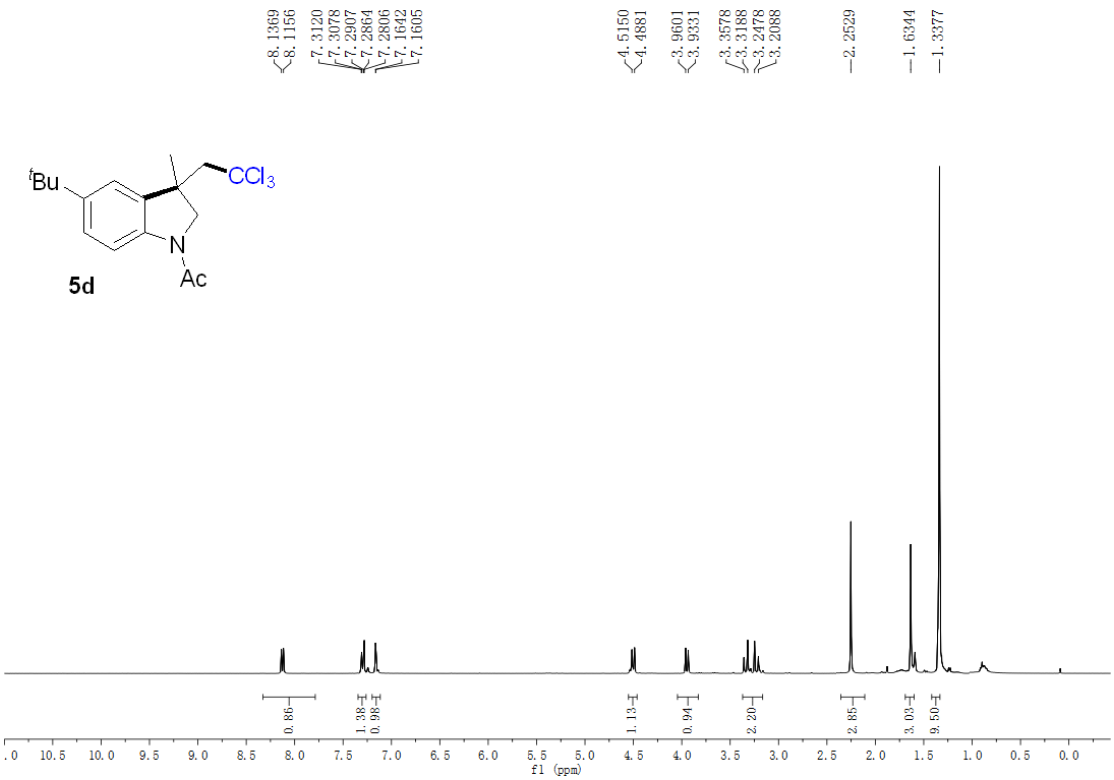
139.5453
124.4374
121.8850
121.7479
119.3340
118.0971
116.7580
116.1398
96.8890
77.5163
77.1984
76.8806
62.5505
62.0936
45.2145
26.9705
24.2224
F3CO
CCl3
N
Ac
5c
f1 (ppm)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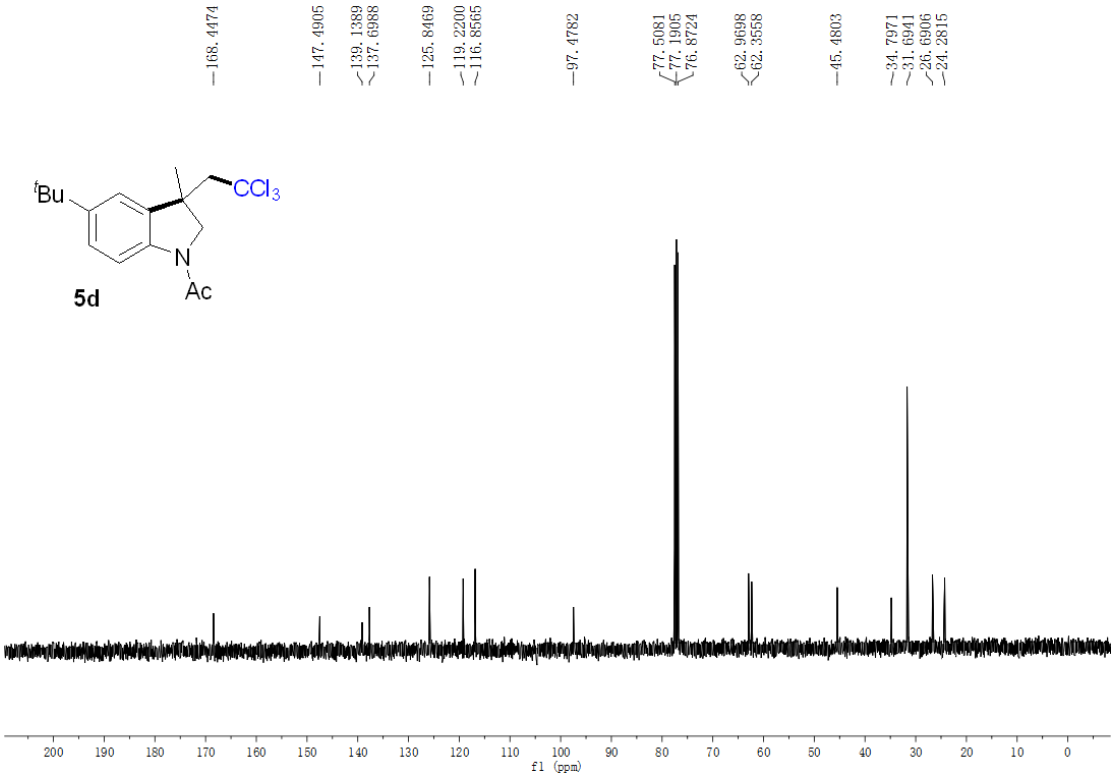
8.1156
7.3120
7.3078
7.2907
7.2864
7.2806
7.1642
7.1605
4.5150
4.4881
3.9601
3.9331
3.3578
3.3188
3.2478
3.2088
2.2529
1.6344
1.3377
tBu
CCl3
N
Ac
5d
0.86
1.38
0.98
1.13
0.94
2.20
2.85
3.03
9.50
f1 (ppm)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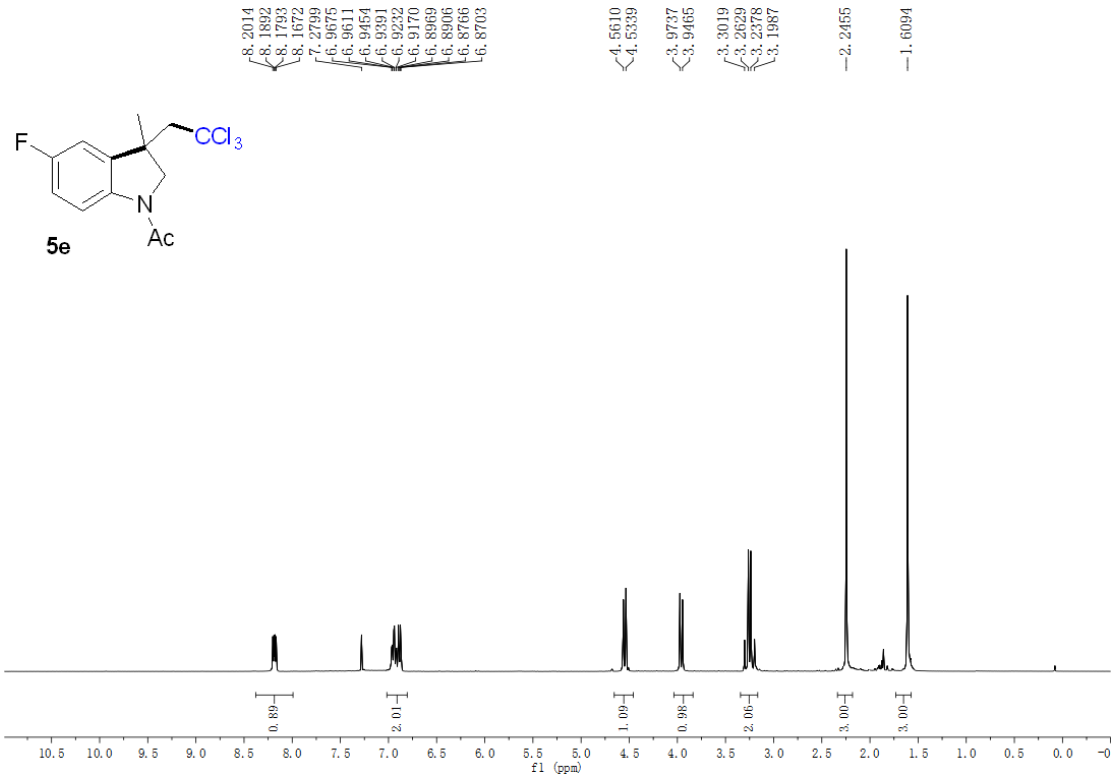
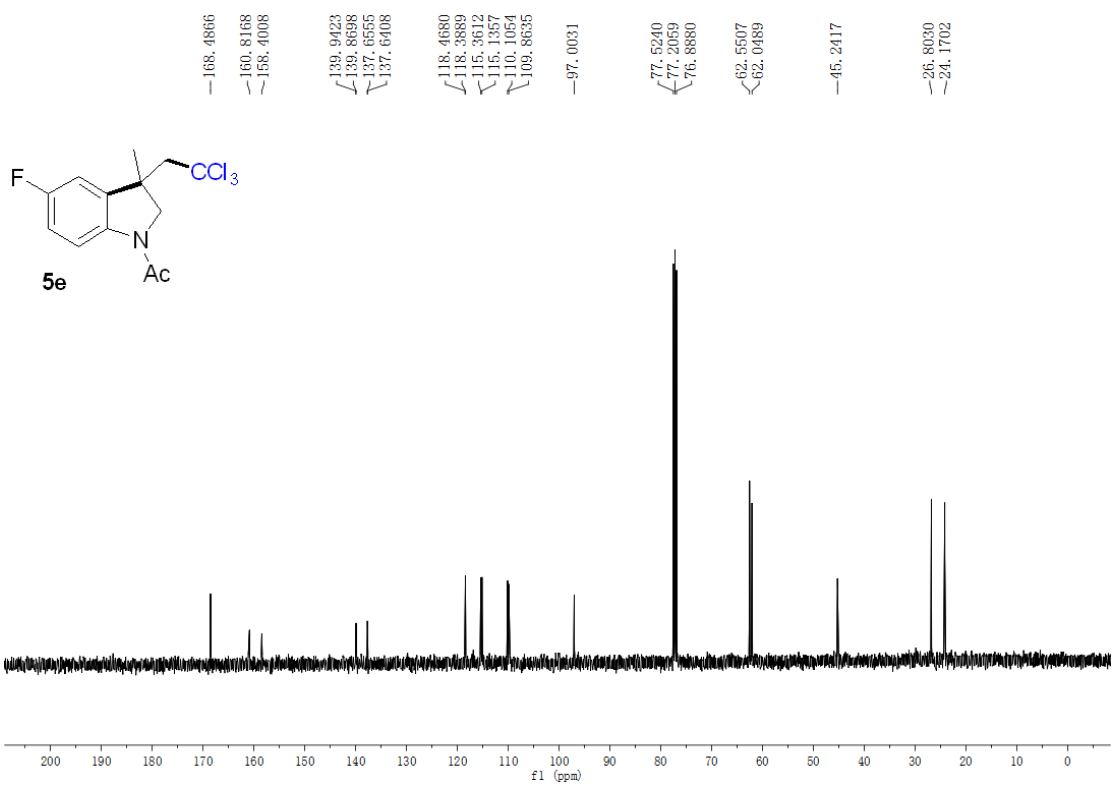
125.8469
119.2200
116.8565
97.4782
77.5081
77.1905
76.8724
62.9698
62.3558
45.4803
34.7971
31.6941
26.6906
24.2815
tBu
CCl3
N
Ac
5d
f1 (ppm)


8.2014
8.1892
8.1793
8.1672
7.2799
6.9675
6.9611
6.9454
6.9391
6.9232
6.9170
6.8969
6.8906
6.8766
6.8703
4.5610
4.5339
3.9737
3.9465
3.3019
3.2629
3.2378
3.1987
2.2455
1.6094
F
CCl3
N
Ac
5e
0.89
2.01
1.09
0.98
2.06
3.00
3.00
f1 (ppm)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139.8698
137.6555
137.6408
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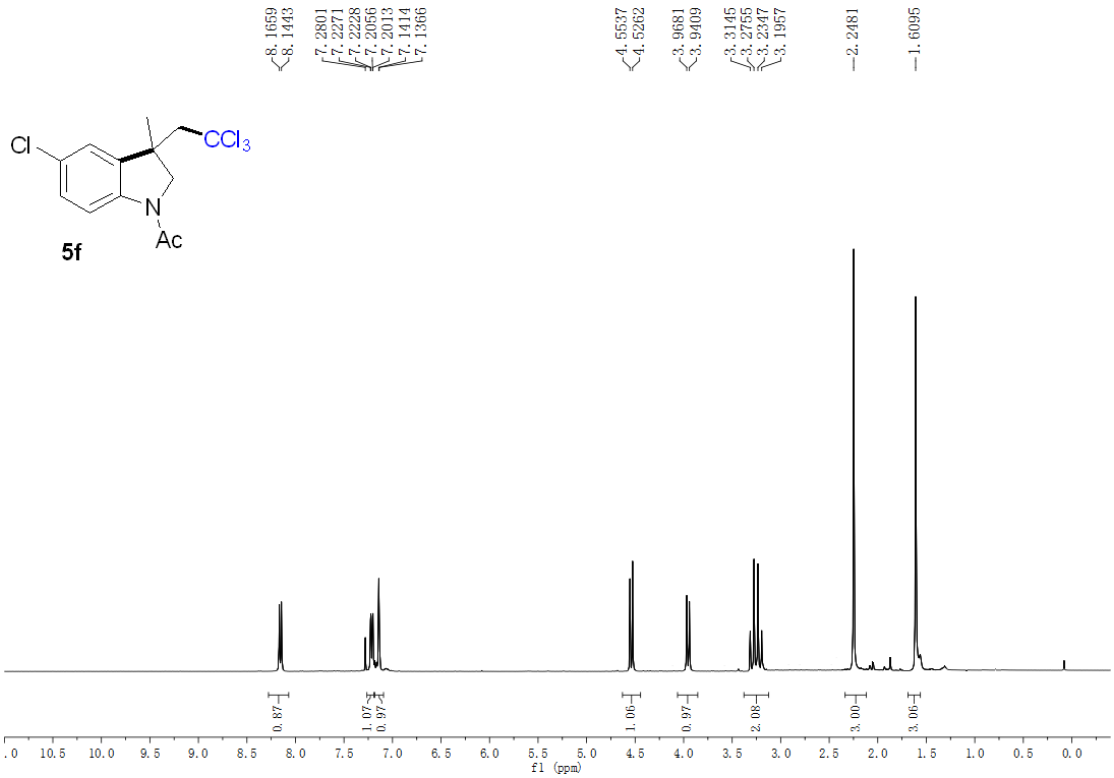
118.3889
115.3612
115.1357
110.1054
109.8635
97.0031
77.5240
77.2059
76.8880
62.5507
62.0489
45.2417
26.8030
24.1702
F
CCl3
N
Ac
5e
f1 (ppm)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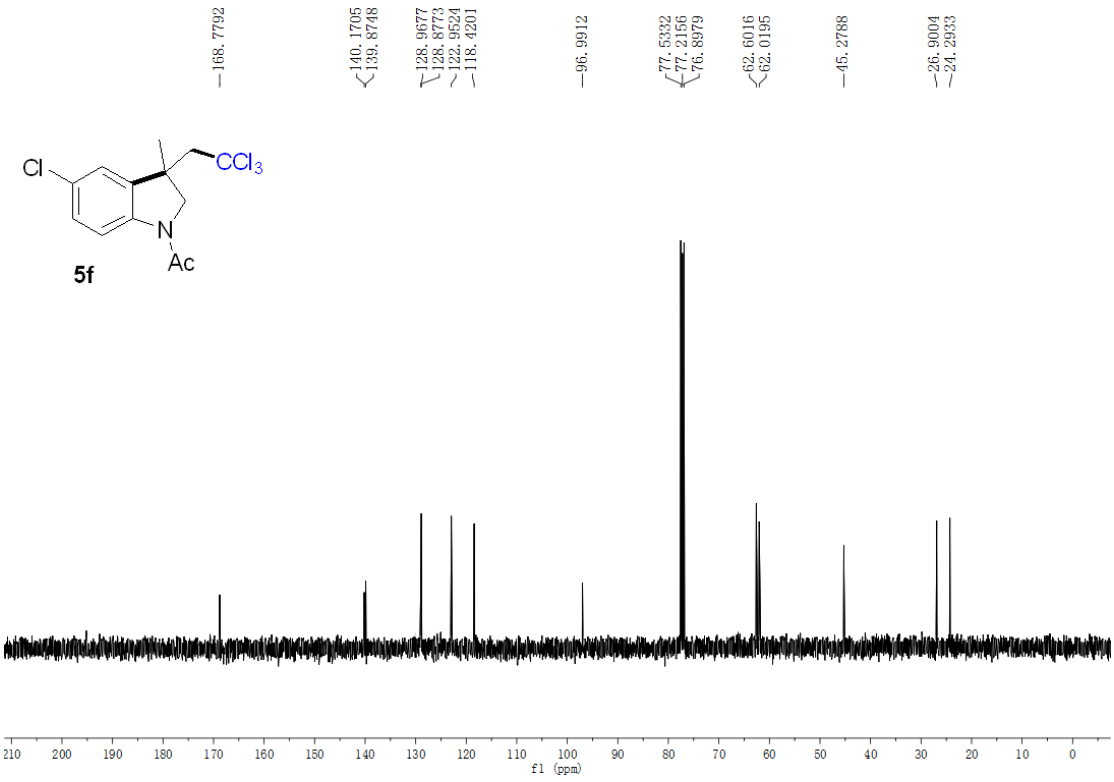
7.2801
7.2271
7.2228
7.2056
7.2013
7.1414
7.1366
4.5537
4.5262
3.9681
3.9409
3.3145
3.2755
3.2347
3.1957
2.2481
1.6095
Cl
CCl3
N
Ac
5f
0.87
1.07
0.97
1.06
0.97
2.08
3.00
3.06
f1 (ppm)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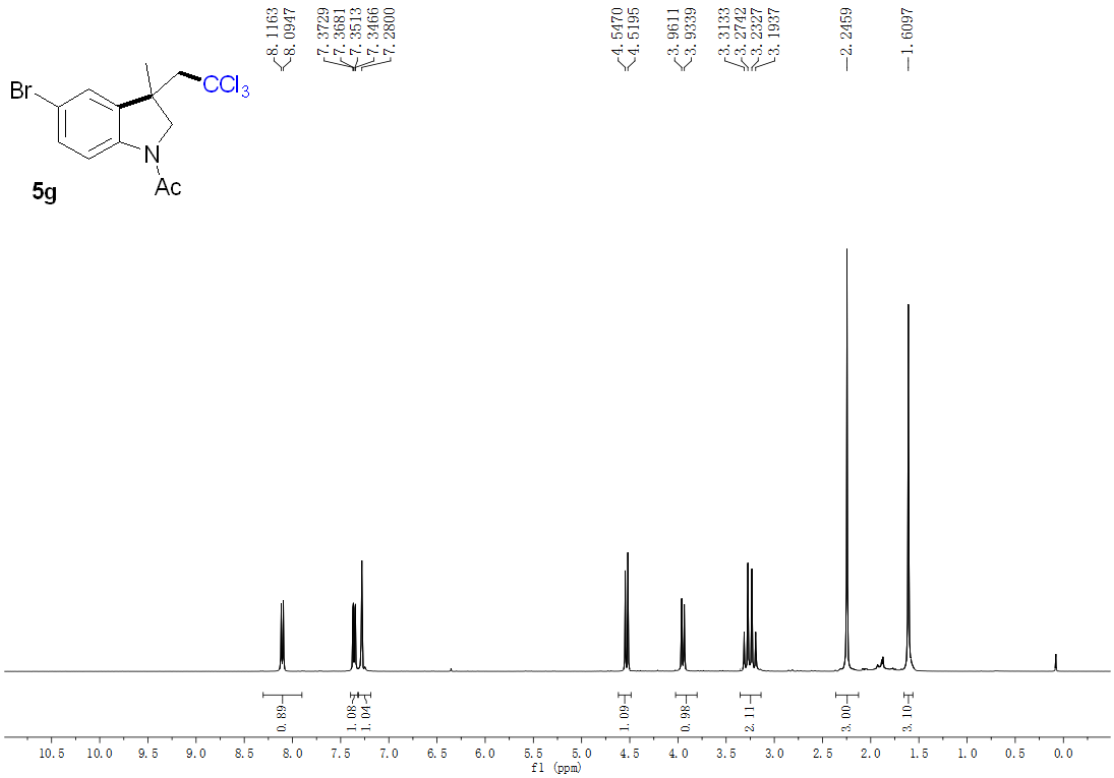
139.8748
128.9677
128.8773
122.9524
118.4201
96.9912
77.5332
77.2156
76.8979
62.6016
62.0195
45.2788
26.9004
24.2933
Cl
CCl3
N
Ac
5f
f1 (ppm)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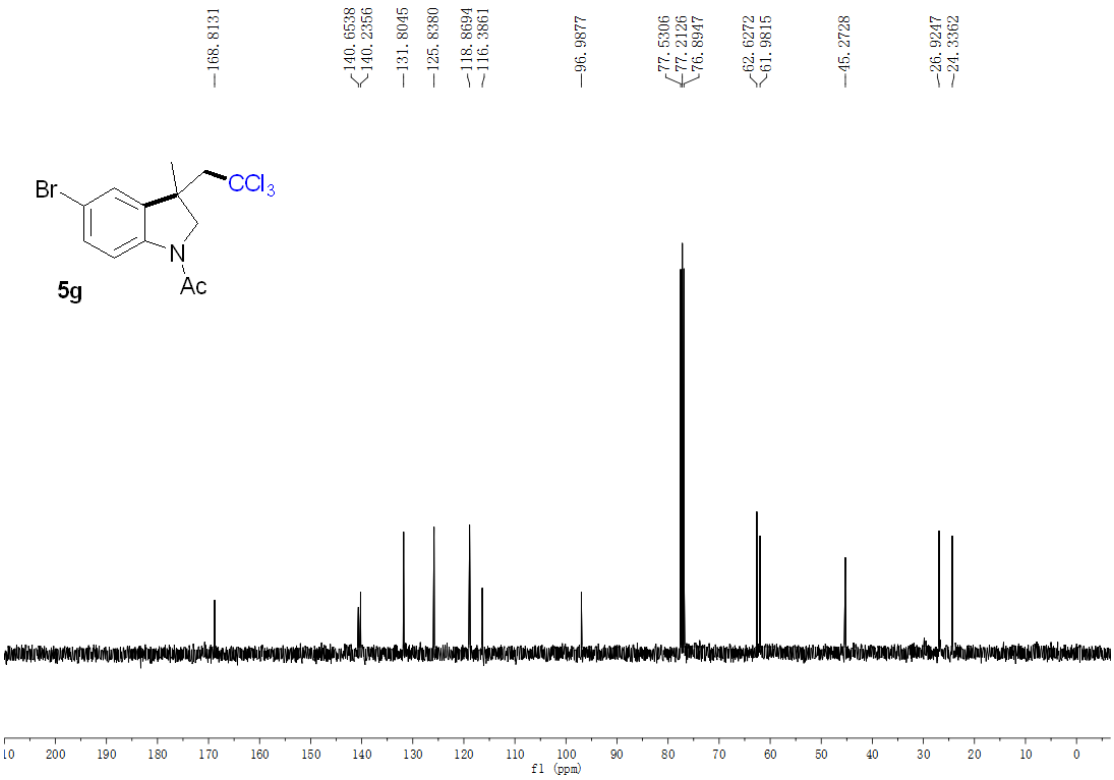
8.0947
7.3729
7.3681
7.3513
7.3466
7.2800
4.5470
4.5195
3.9611
3.9339
3.3133
3.2742
3.2327
3.1937
2.2459
1.6097
Br
CCl3
N
Ac
5g
0.89
1.08
1.04
1.09
0.98
2.11
3.00
3.10
f1 (ppm)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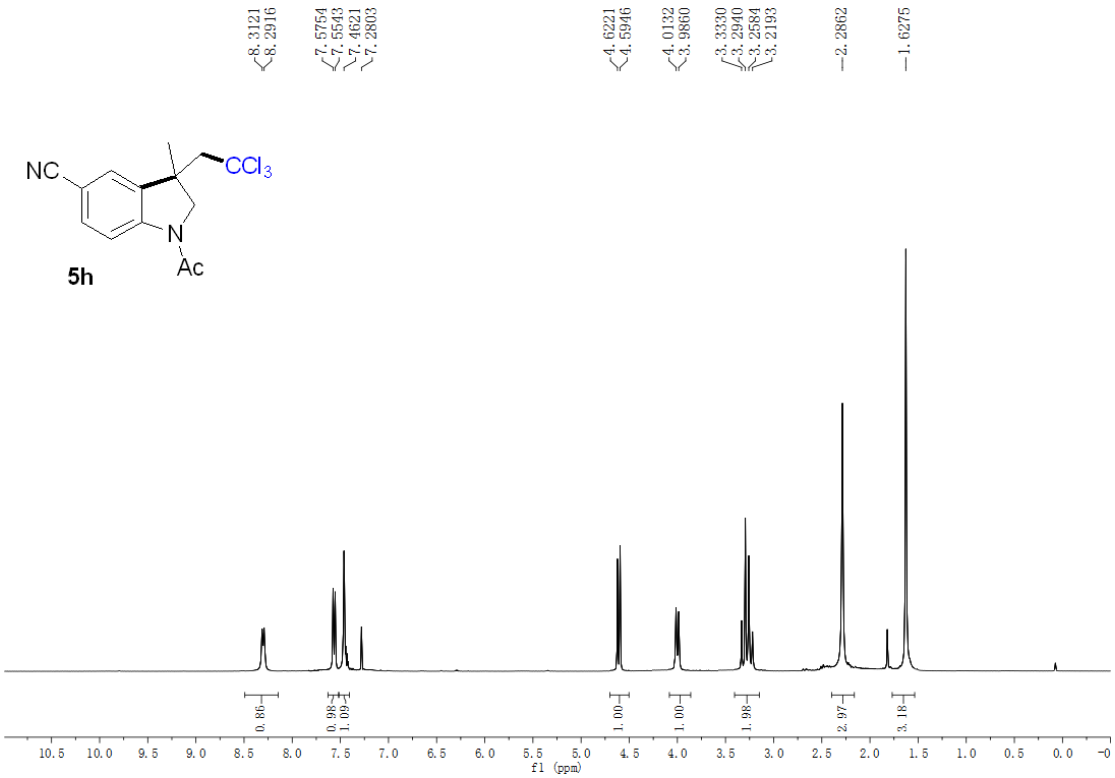
140.2356
131.8045
125.8380
118.8694
116.3861
96.9877
77.5306
77.2126
76.8947
62.6272
61.9815
45.2728
26.9247
24.3362
Br
CCl3
N
Ac
5g
f1 (ppm)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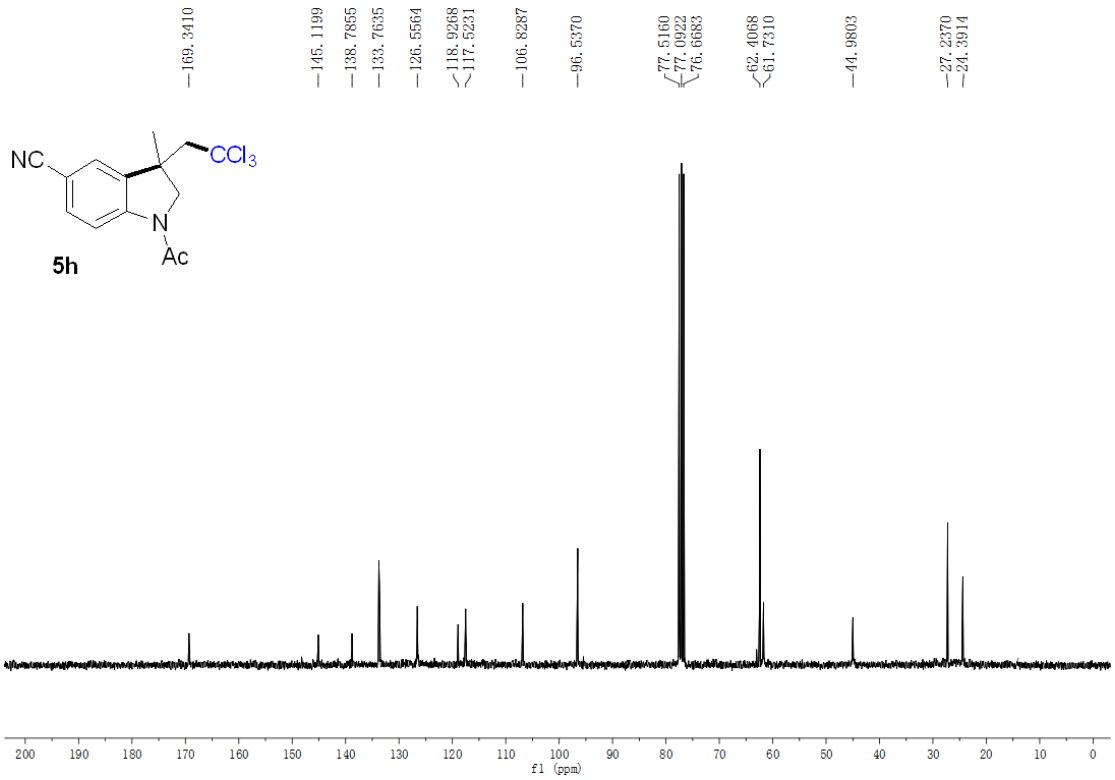
7.5543
7.4621
7.2803
4.6221
4.5946
4.0132
3.9860
3.3330
3.2940
3.2584
3.2193
2.2862
1.6275
NC
CCl3
N
Ac
5h
0.86
0.98
1.09
1.00
1.00
1.98
2.97
3.18
f1 (ppm)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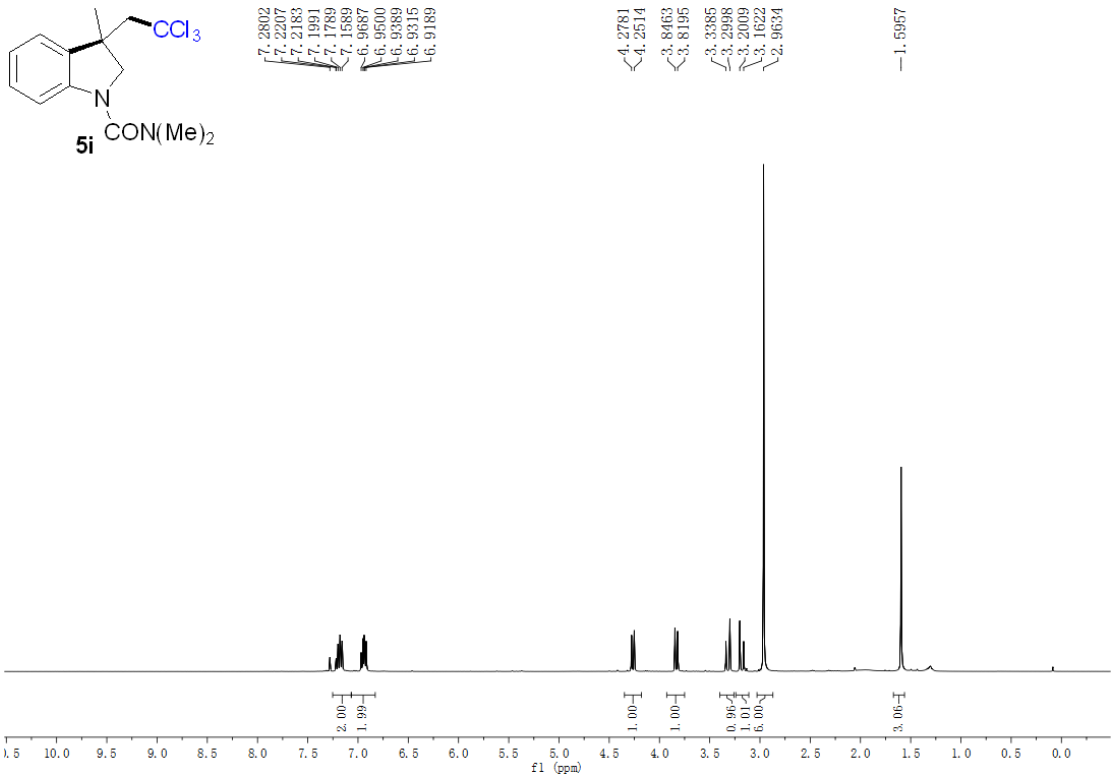
138.7855
133.7635
126.5564
118.9268
117.5231
106.8287
96.5370
77.5160
77.0922
76.6683
62.4068
61.7310
44.9803
27.2370
24.3914
NC
CCl3
N
Ac
5h
f1 (ppm)


CCl3
N
CON(Me)2
5i
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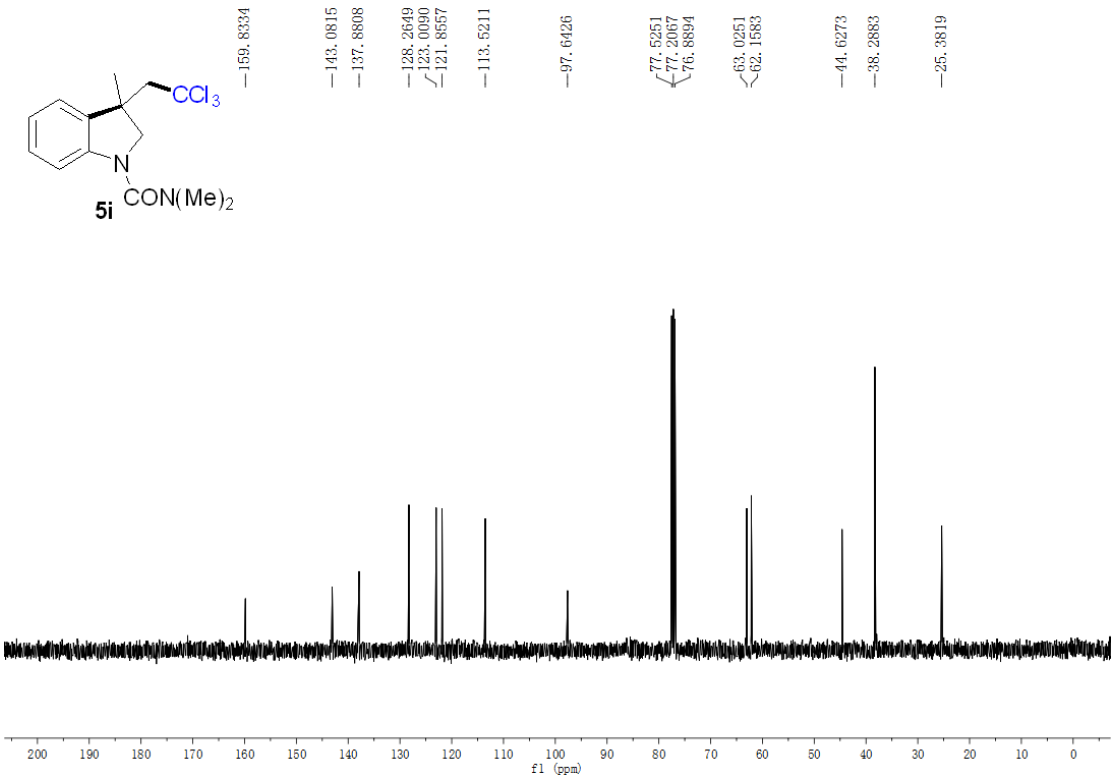
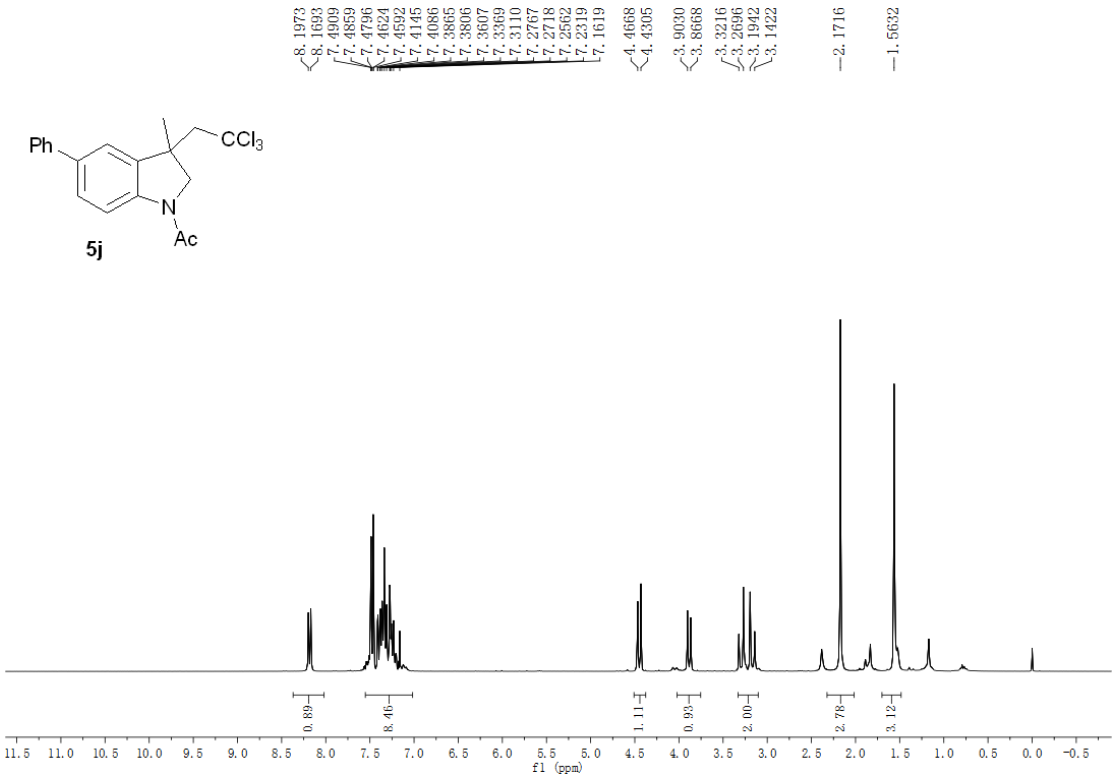
7.2207
7.2183
7.1991
7.1789
7.1589
6.9687
6.9500
6.9389
6.9315
6.9189
4.2781
4.2514
3.8463
3.8195
3.3385
3.2998
3.2009
3.1622
2.9634
1.5957
2.00
1.99
1.00
1.00
0.96
1.01
6.00
3.06
f1 (ppm)


CCl3
N
CON(Me)2
5i
159.8334
143.0815
137.8808
128.2649
123.0090
121.8557
113.5211
97.6426
77.5251
77.2067
76.8894
63.0251
62.1583
44.6273
38.2883
25.3819
f1 (ppm)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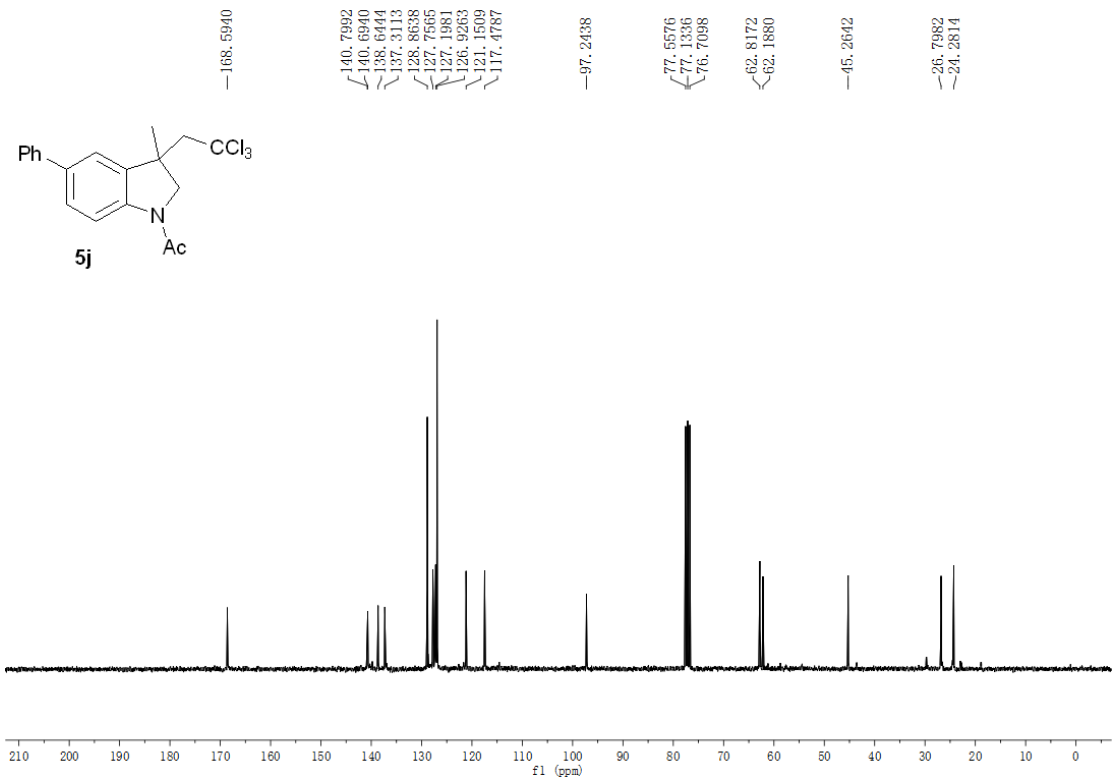
7.4592
7.4145
7.4086
7.3865
7.3806
7.3607
7.3369
7.3110
7.2767
7.2718
7.2562
7.2319
7.1619
4.4668
4.4305
3.9030
3.8668
3.3216
3.2696
3.1942
3.1422
2.1716
1.5632
Ph
CCl3
N
Ac
5j
0.89
8.46
1.11
0.93
2.00
2.78
3.12
f1 (ppm)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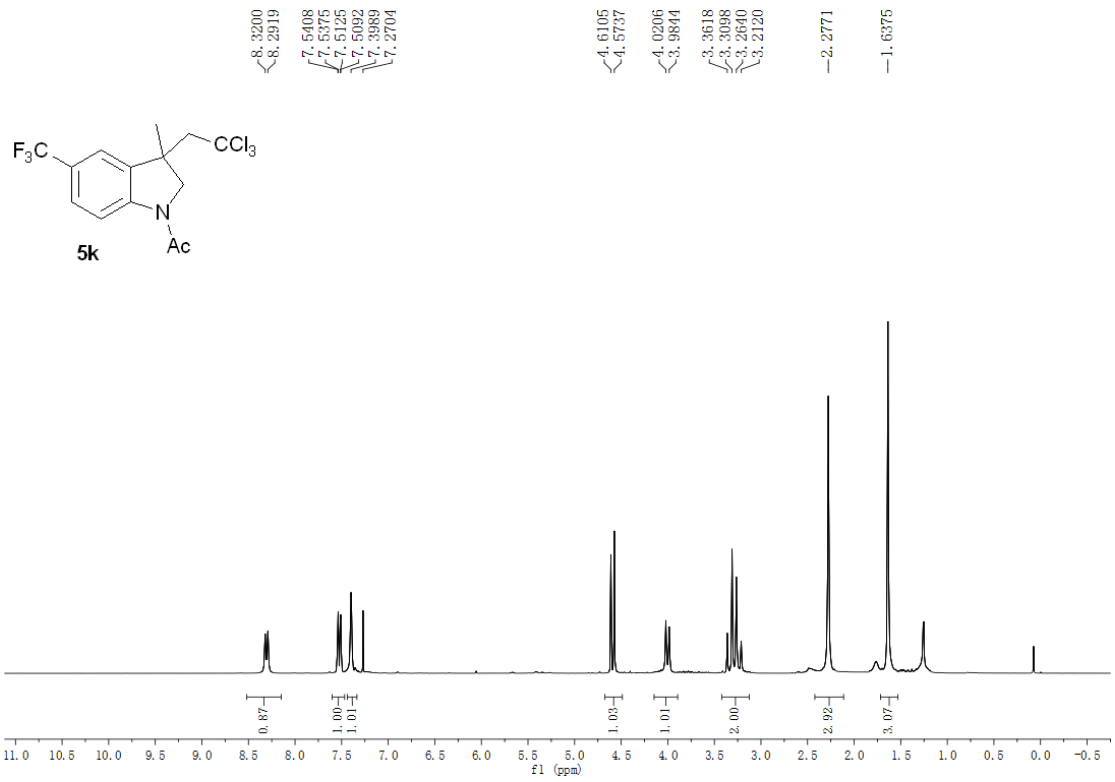
140.6940
138.6444
137.3113
128.8638
127.7565
127.1981
126.9263
121.1509
117.4787
97.2438
77.5576
77.1336
76.7098
62.8172
62.1880
45.2642
26.7982
24.2814
Ph
CCl3
N
Ac
5j
f1 (ppm)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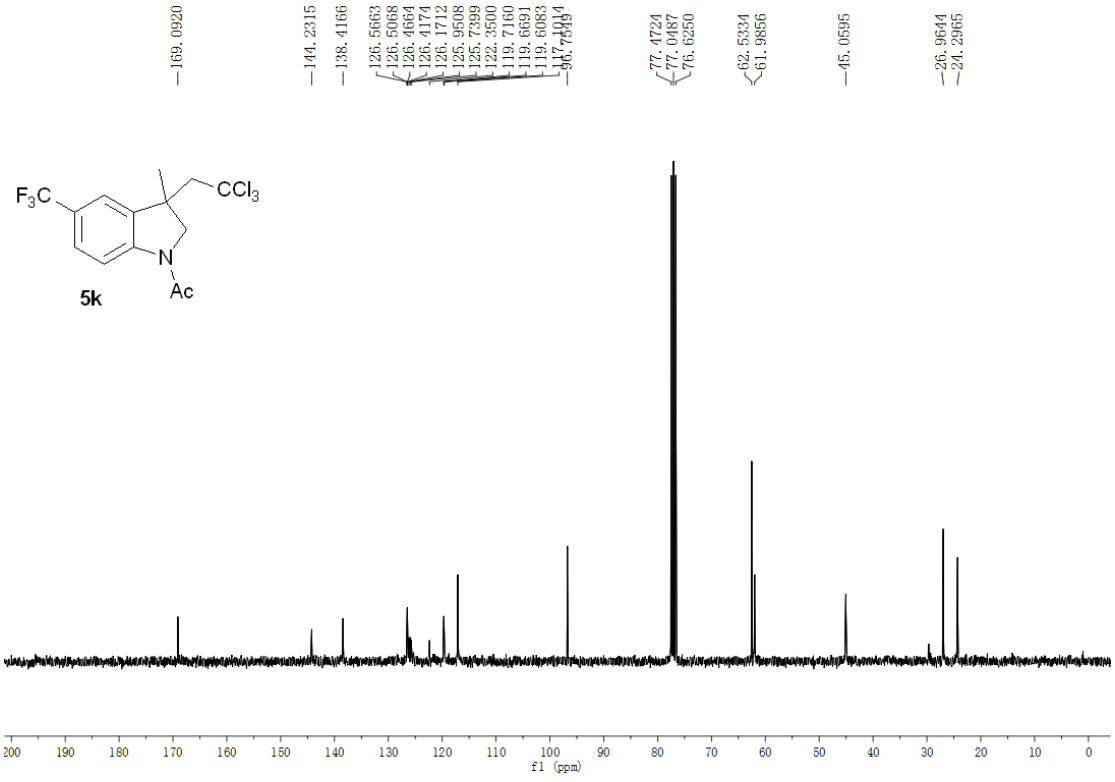
8.2919
7.5408
7.5375
7.5125
7.5092
7.3989
7.2704
4.6105
4.5737
4.0206
3.9844
3.3618
3.3098
3.2640
3.2120
2.2771
1.6375
F3C
CCl3
N
Ac
5k
0.87
1.00
1.01
1.03
1.01
2.00
2.92
3.07
f1 (ppm)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126.5068
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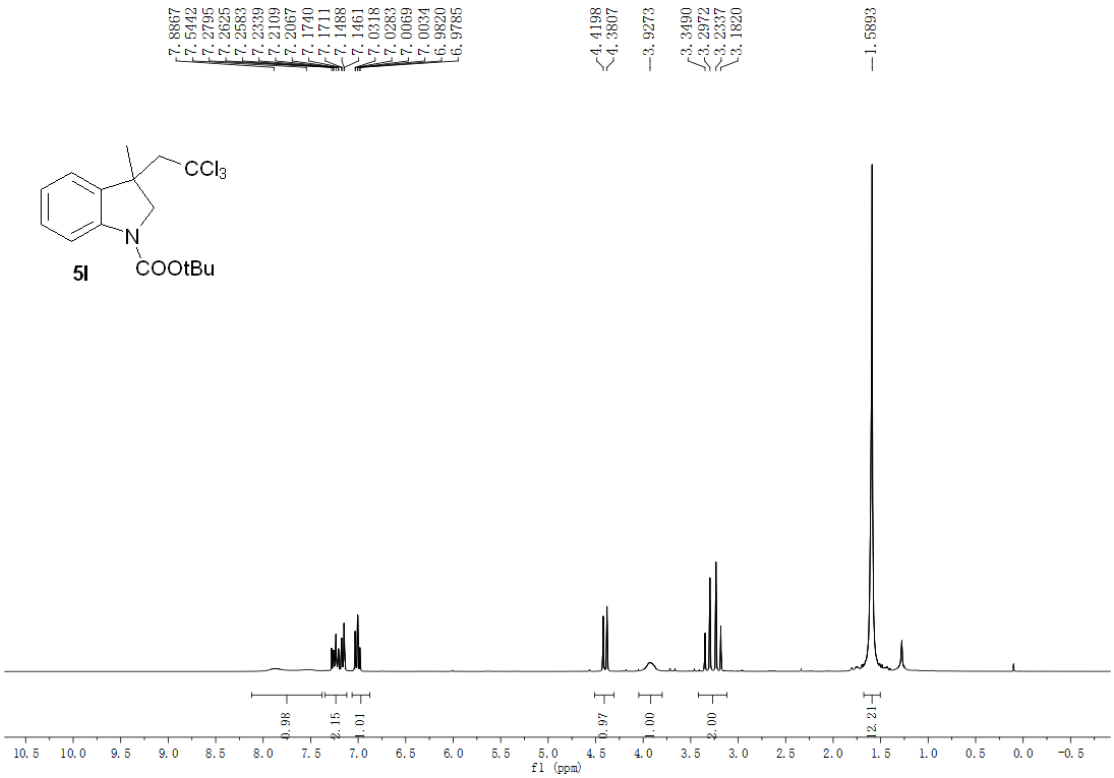
126.4174
126.1712
125.9508
125.7399
122.3500
119.7160
119.6691
119.6083
96.7549
77.4724
77.0487
76.6250
62.5334
61.9856
45.0595
26.9644
24.2965
F3C
CCl3
N
Ac
5k
f1 (ppm)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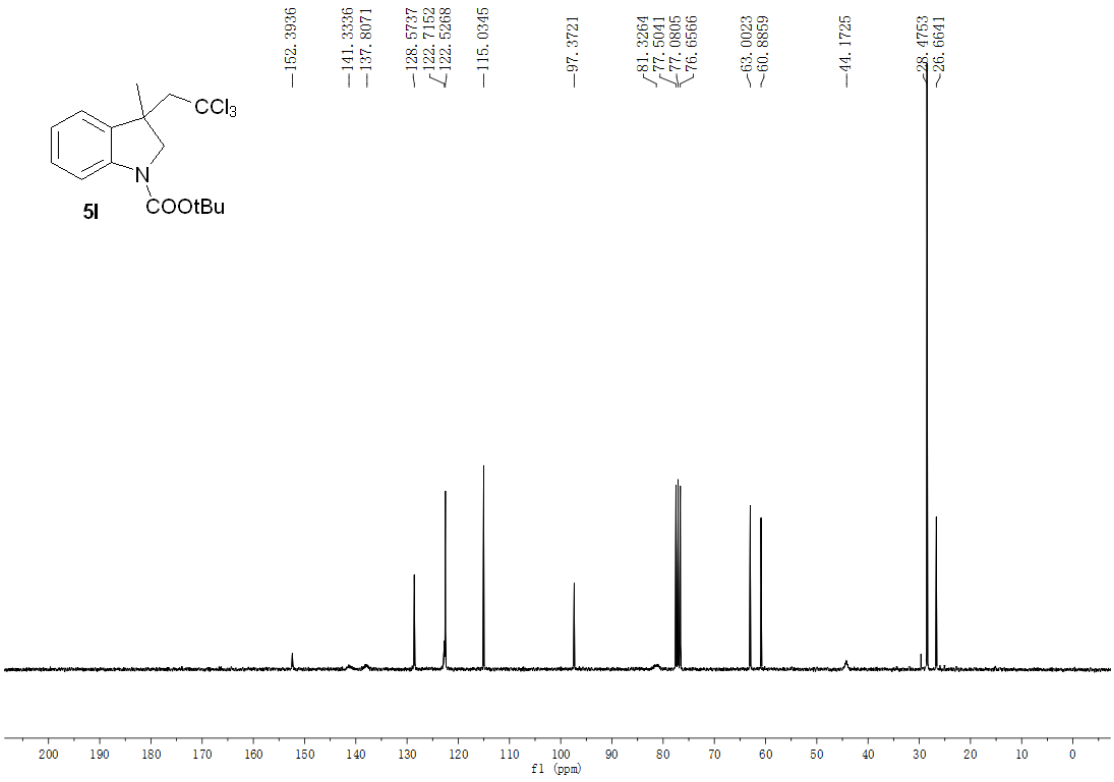
7.2795
7.2625
7.2583
7.2339
7.2109
7.2067
7.1740
7.1711
7.1488
7.1461
7.0318
7.0283
7.0069
7.0034
6.9820
6.9785
4.4198
4.3807
3.9273
3.3490
3.2972
3.2337
3.1820
1.5893
CCl3
N
COOtBu
5l
0.98
2.15
1.01
0.97
1.00
2.00
12.21
f1 (ppm)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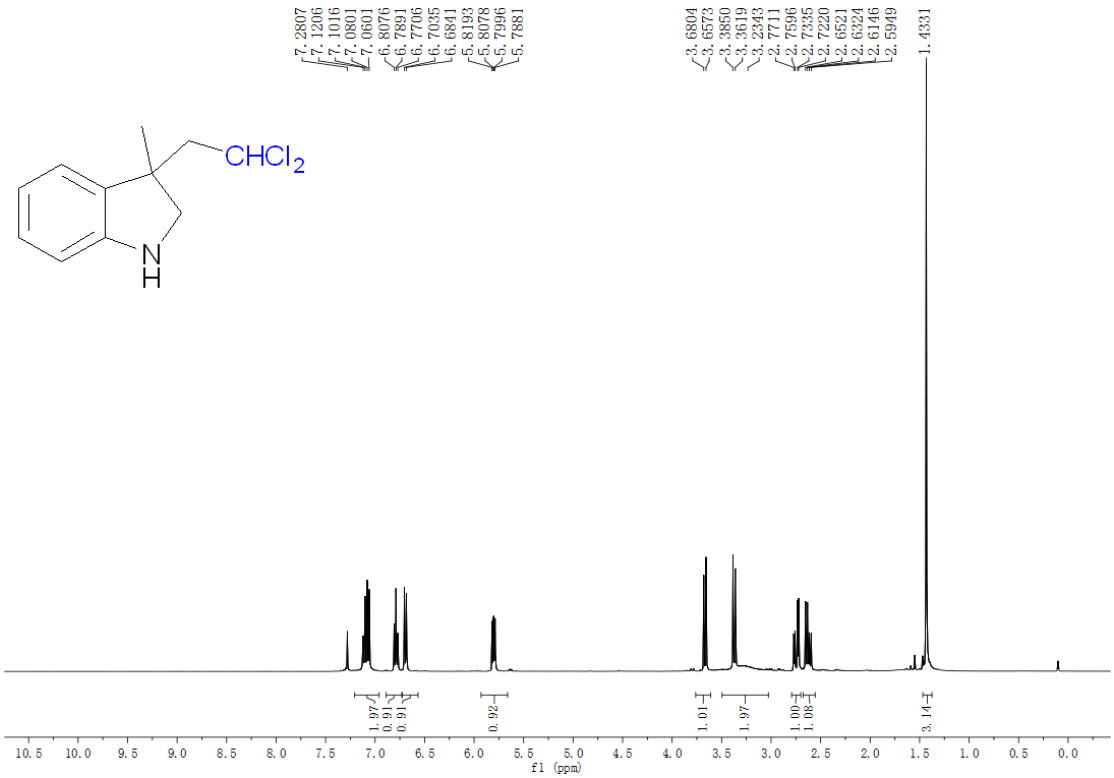
141.3336
137.8071
128.5737
122.7152
122.5268
115.0345
97.3721
81.3264
77.5041
77.0805
76.6566
63.0023
60.8859
44.1725
28.4753
26.6641
CCl3
N
COOtBu
5l
f1 (ppm)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7.1016
7.0801
7.0601
6.8076
6.7891
6.7706
6.7035
6.6841
5.8193
5.8078
5.7996
5.7881
3.6804
3.6573
3.3850
3.3619
3.2343
2.7711
2.7596
2.7335
2.7220
2.6521
2.6324
2.6146
2.5949
1.4331
CHCl2
N
H
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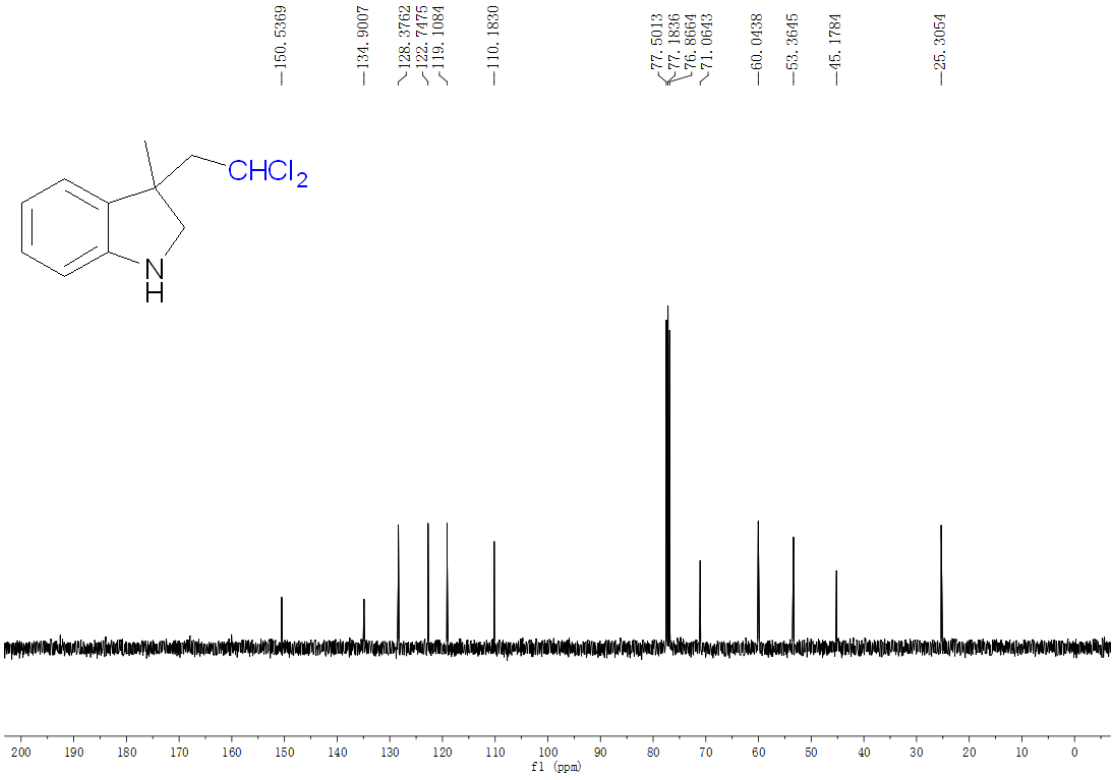
0.91
0.91
0.92
1.01
1.97
1.00
1.08
3.14
f1 (ppm)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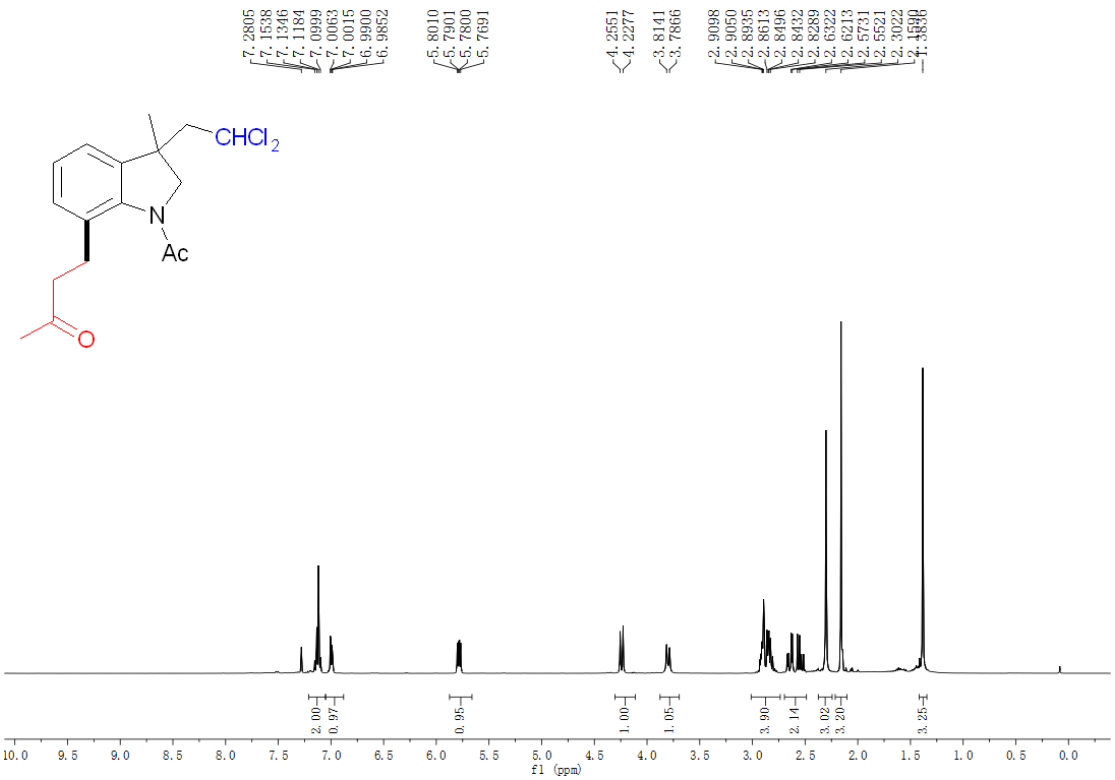
128.3762
122.7475
119.1084
110.1830
77.5013
77.1836
76.8664
71.0643
60.0438
53.3645
45.1784
25.3054
CHCl2
N
H
f1 (ppm)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7.1346
7.1184
7.0999
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7.0015
6.9900
6.9852
5.8010
5.7901
5.7800
5.7691
4.2551
4.2277
3.8141
3.7866
2.9098
2.9050
2.8935
2.8613
2.8496
2.8432
2.8289
2.6322
2.6213
2.5731
2.5521
2.3022
CHCl2
N
Ac
O
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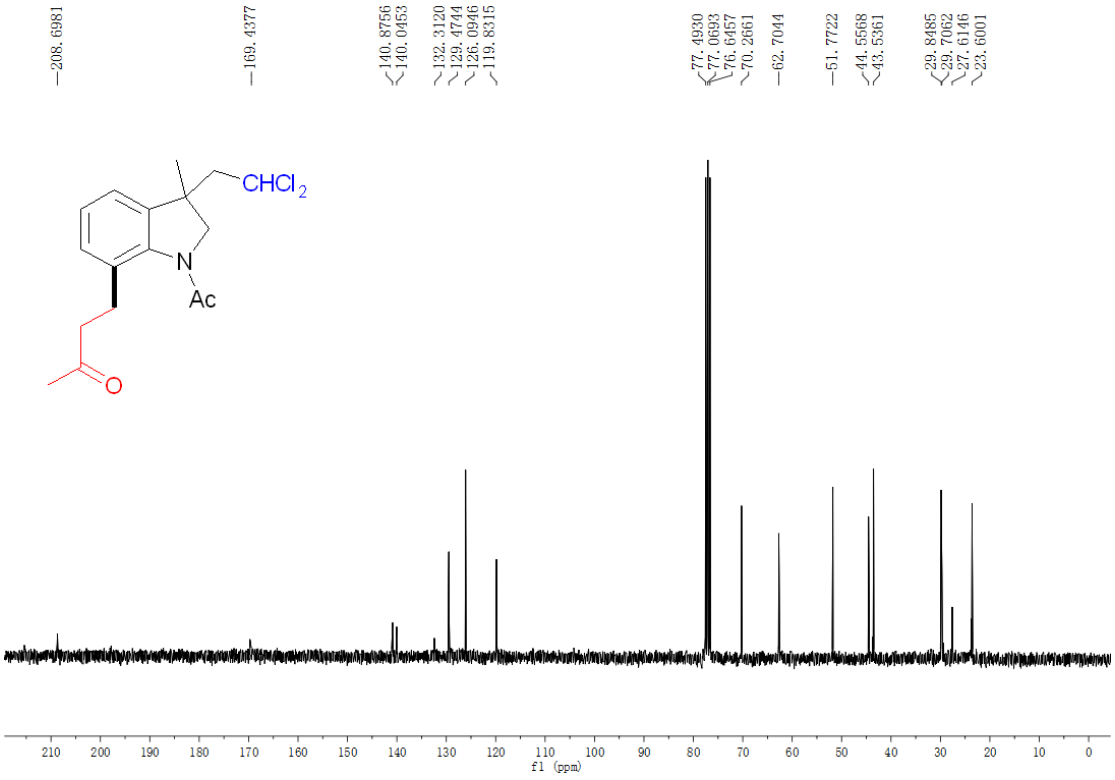
0.95
1.00
1.05
3.90
2.14
3.02
3.20
3.25
f1 (ppm)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126.0946
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77.4930
77.0693
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70.2661
62.7044
51.7722
44.5568
43.5361
29.8485
29.7062
27.6146
23.6001
CHCl2
N
Ac
O
f1 (ppm)