

Supporting Information for

Dinuclear zinc-catalyzed enantioselective formal [3+2] cycloaddition of *N*-2,2,2-trifluoroethylisatin ketimines with low reactive aurone derivatives

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General Information

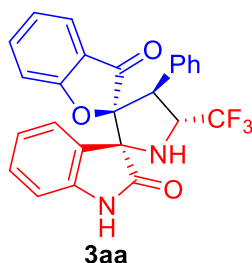
All the dry solvents were treated prior to use according to the standard methods. Unless otherwise noted, all sensitive to air or moisture reactions were carried out under nitrogen using standard Schlenk and vacuum line techniques. Diethylzinc (1.0 mol/L in hexane) was purchased from Aldrich and used as received. The melting point (m. p.) was determined on an electrothermal digital melting point apparatus and uncorrected. Optical rotation values were measured with instruments operating at $\lambda = 589$ nm, corresponding to the sodium D line at 20 °C. Enantiomeric excesses values were determined with HPLC (chiral column; mobile phase hexane/*i*-PrOH). NMR spectra were recorded on NMR spectrometer with CDCl₃ or DMSO-*d*₆ as the solvents, and TMS as an internal standard (400 MHz for ¹H, 100 MHz for ¹³C). Infrared (IR) spectra were recorded by using Ft-IR spectrometer (ν in cm⁻¹). HRMS was determined on a Q-TOF Micro LC/MS System ESI spectrometer. Chiral ligands **L1–L5**,¹ **L6–L8**,² **L9**,³ compounds **1**,⁴ and **2**⁵ were synthesized according to the literature.

General procedures for the catalytic reaction

In a flame-dried Schlenk tube, a solution of diethylzinc (0.04 mL, 1.0 mol/L in hexane, 0.04 mmol) was added to a solution of the chiral ligand **L1** (0.02 mmol) in dry toluene (1.0 mL) under nitrogen at 10 °C. The mixture was stirred for 30 min. Then compound **2** (0.2 mmol) was added and stirred for another 5 min. Substrate **1** (0.2 mmol) was dissolved in 1.0 mL toluene and added dropwise to the mixture by a peristaltic injection pump over 30 min at 10 °C. The mixture was stirred at 10 °C for the necessary time to complete the reaction (detected by TLC). Finally, the solution was diluted with saturated solution of Na₂CO₃ and extracted with toluene (3 x 10 mL). The combined organic layer was dried over MgSO₄ and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel to give products **3**.

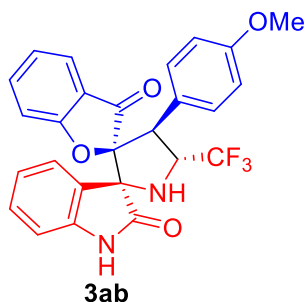
Characterization of 3

(2*S*,2'*S*,4'*S*,5'*R*)-4'-phenyl-5'-(trifluoromethyl)-3*H*-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (**3aa**):



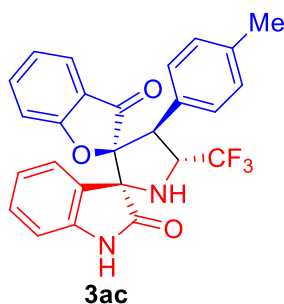
Compound **3aa** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 90% yield (81mg, >20:1 dr): 95% *ee*; $[\alpha]_{\text{D}}^{20} = +139$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO-*d*₆) δ 10.42 (s, 1H), 7.59–7.46 (m, 2H), 7.36–7.27 (m, 2H), 7.26–6.99 (m, 6H), 6.89–6.72 (m, 2H), 6.71–6.60 (m, 1H), 4.96–4.77 (m, 2H), 4.77–4.68 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, DMSO-*d*₆) δ 194.8, 175.2, 170.4, 142.8, 139.5, 132.4, 130.7, 129.4, 128.8, 128.5, 127.6, 125.4 (q, $J = 280.2$ Hz), 123.9, 122.7, 121.6, 121.5, 113.3, 109.7, 98.1, 72.8, 61.9 (q, $J = 29.5$ Hz), 50.7; $^{19}\text{F NMR}$ (376 MHz, DMSO-*d*₆) δ -71.51; **IR** (neat): 2989, 2971, 1728, 1610, 1462, 753, 679 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 451.1264, found 451.1265; **HPLC**: Daicel Chiralpak IA, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 14.84$ min and $t_{\text{minor}} = 10.46$ min.

(2*S*,2'*S*,4'*S*,5'*R*)-4'-(4-methoxyphenyl)-5'-(trifluoromethyl)-3*H*-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (**3ab**):



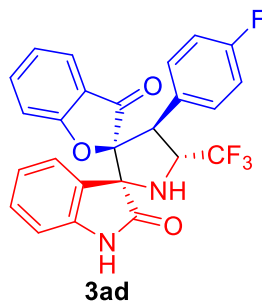
Compound **3ab** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 95% yield (91 mg, >20:1 dr): 97% *ee*; $[\alpha]_{\text{D}}^{20} = +134$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.43–8.33 (m, 1H), 7.43 (d, $J = 7.5$ Hz, 1H), 7.37–7.29 (m, 1H), 7.27–7.20 (m, 2H), 7.16 (s, 1H), 7.05–6.99 (m, 1H), 6.95 (d, $J = 8.4$ Hz, 1H), 6.77 (m, 1H), 6.73–6.65 (m, 2H), 6.60 (d, $J = 8.8$ Hz, 2H), 4.97 (d, $J = 10.9$ Hz, 1H), 4.76–4.51 (m, 1H), 3.58 (s, 3H), 2.84 (d, $J = 7.1$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 194.6, 175.8, 170.2, 159.2, 141.7, 138.4, 130.5, 130.3, 126.1, 125.65 (q, $J = 280.0$ Hz), 124.3, 124.1, 123.3, 122.3, 122.1, 121.7, 113.7, 112.3, 110.3, 97.7, 72.4, 62.6 (q, $J = 30.6$ Hz), 55.0, 50.2; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -73.17; **IR** (neat): 3299, 2988, 1724, 1694, 1514, 1474, 1055, 761, 595 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 481.1370, found 481.1370; **HPLC**: Daicel Chiralpak IA, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 27.82$ min and $t_{\text{minor}} = 17.01$ min.

(2S,2'S,4'S,5'R)-4'-(p-tolyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (**3ac**):



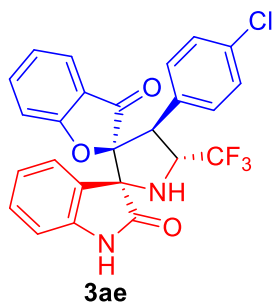
Compound **3ac** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 90% yield (84 mg, >20:1 dr): 94% *ee*; $[\alpha]_D^{20} = +184$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 10.60–10.15 (m, 1H), 7.60–7.47 (m, 2H), 7.25–7.15 (m, 4H), 7.10–6.92 (m, 3H), 6.64 (d, $J = 7.7$ Hz, 1H), 4.92–4.73 (m, 2H), 4.69 (d, $J = 8.9$ Hz, 1H), 2.13 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, DMSO- d_6) δ 194.3, 174.7, 169.9, 142.3, 138.9, 137.1, 130.1, 128.9, 128.8, 127.1, 126.1 (q, $J = 280.2$ Hz), 123.5, 123.4, 122.2, 121.0, 112.8, 109.2, 97.6, 72.4, 61.6 (q, $J = 29.9$ Hz), 49.8, 20.5; $^{19}\text{F NMR}$ (376 MHz, DMSO- d_6) δ -71.45; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 465.1421, found 465.1423; **IR** (neat): 3301, 2972, 2900, 1279, 1635, 1454, 1055, 751, 622 cm^{-1} ; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 27.20$ min and $t_{\text{minor}} = 14.80$ min.

(2S,2'S,4'S,5'R)-4'-(4-fluorophenyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (**3ad**):



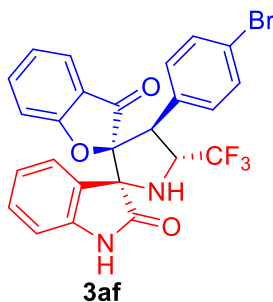
Compound **3ad** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 92% yield (86 mg, >20:1 dr): 96% *ee*; $[\alpha]_D^{20} = +159$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.78 (s, 1H), 7.54 (d, $J = 7.5$ Hz, 1H), 7.47–7.33 (m, 3H), 7.26 (s, 1H), 7.15–7.09 (m, 1H), 7.05 (d, $J = 8.4$ Hz, 1H), 6.91–6.73 (m, 5H), 5.12 (d, $J = 10.9$ Hz, 1H), 4.85–4.66 (m, 1H), 3.00 (d, $J = 8.2$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 194.3, 175.9, 170.1, 162.42 (d, $J = 247.1$ Hz), 141.8, 138.6, 130.9, 130.8, 130.7, 127.3 (d, $J = 3.3$ Hz), 126.1, δ 125.6 (q, $J = 280.0$ Hz), 124.4, 123.9, 122.4, 122.3, 121.6, 115.5, 115.2, 112.3, 110.5, 97.5, 72.5, 62.6 (q, $J = 30.8$ Hz), 50.2; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -73.19, -113.82; **IR** (neat): 2988, 2972, 1732, 1609, 1152, 1463, 1056, 751, 679 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 469.1170, found 469.1171; **HPLC**: Daicel Chiralpak IA, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 13.45$ min and $t_{\text{minor}} = 11.83$ min.

(2S,2'S,4'S,5'R)-4'-(4-chlorophenyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (**3ae**):



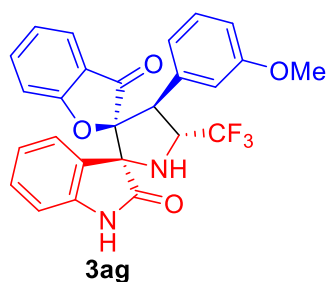
Compound **3ae** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 92% yield (89 mg, >20:1 dr): 94% *ee*; $[\alpha]_{\text{D}}^{20} = +169$ (c 1.0, in THF); **¹H NMR** (400 MHz, CDCl₃) δ 8.49 (s, 1H), 7.54–7.41 (m, 2H), 7.38–7.29 (m, 3H), 7.18–7.09 (m, 3H), 7.05 (d, *J* = 8.4 Hz, 1H), 6.91–6.65 (m, 3H), 5.10 (d, *J* = 10.9 Hz, 1H), 4.88–4.63 (m, 1H), 2.96 (d, *J* = 8.2 Hz, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 194.1, 175.6, 170.1, 141.6, 138.7, 134.1, 130.7, 130.6, 130.1, 128.6, 126.1, 125.5 (q, *J* = 280.2 Hz), 124.4, 123.8, 122.4, 121.6, 112.3, 110.5, 97.4, 72.5, 62.5 (q, *J* = 30.7 Hz), 50.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ -73.24; **IR** (neat): 3344, 2988, 1733, 1615, 1474, 1132, 752, 678 cm⁻¹; **HRMS** (ESI): *m/z* for [M+H]⁺: calcd 485.0874, found 485.0877; **HPLC**: Daicel Chiralpak IA, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, *t*_{major} = 18.00 min and *t*_{minor} = 11.73 min.

(2S,2'S,4'S,5'R)-4'-(4-bromophenyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3af):



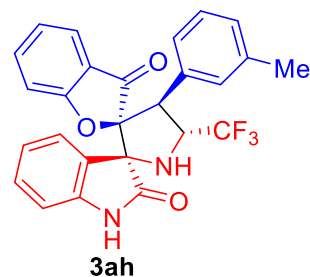
Compound **3af** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 93% yield (98 mg, >20:1 dr): 94% *ee*; $[\alpha]_{\text{D}}^{20} = +118$ (c 1.0, in THF); **¹H NMR** (400 MHz, CDCl₃) δ 8.66 (s, 1H), 7.56–7.34 (m, 2H), 7.34–7.29 (m, 3H), 7.29–7.22 (m, 2H), 7.17–7.09 (m, 1H), 7.05 (d, *J* = 8.4 Hz, 1H), 6.91–6.73 (m, 3H), 5.09 (d, *J* = 10.9 Hz, 1H), 4.86–4.68 (m, 1H), 2.98 (d, *J* = 8.3 Hz, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 194.1, 175.8, 170.1, 141.7, 138.7, 131.5, 130.9, 130.7, 130.6, 126.1, 125.5 (q, *J* = 280.2 Hz), 124.5, 123.8, 122.5, 122.4, 122.3, 121.6, 112.3, 110.5, 97.3, 72.57, 62.5 (q, *J* = 30.9 Hz), 50.2; **¹⁹F NMR** (376 MHz, CDCl₃) δ -73.21; **IR** (neat): 2988, 2972, 1131, 1514, 1473, 1074, 751, 678 cm⁻¹; **HRMS** (ESI): *m/z* for [M+H]⁺: calcd 529.0369, found 529.0373; **HPLC**: Daicel Chiralpak IA, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, *t*_{major} = 21.09 min and *t*_{minor} = 12.48 min.

(2S,2'S,4'S,5'R)-4'-(3-methoxyphenyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3ag):



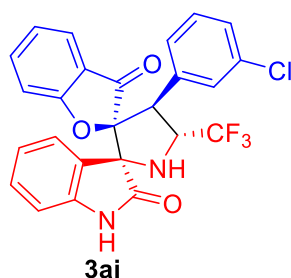
Compound **3ag** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 80% yield (77 mg, >20:1 dr): 91% *ee*; $[\alpha]_{\text{D}}^{20} = +132$ (c 1.0, in THF); **¹H NMR** (400 MHz, DMSO-*d*₆) δ 10.41 (s, 1H), 7.57 – 7.50 (m, 2H), 7.28 – 7.20 (m, 2H), 7.14 – 7.04 (m, 2H), 6.90 – 6.82 (m, 3H), 6.79 (t, *J* = 7.4 Hz, 1H), 6.73 – 6.69 (m, 1H), 6.65 (d, *J* = 7.7 Hz, 1H), 4.90 – 4.73 (m, 2H), 4.69 (d, *J* = 8.6 Hz, 1H), 3.65 (s, 3H); **¹³C NMR** (101 MHz, DMSO-*d*₆) δ 194.7, 175.2, 170.4, 159.3, 142.8, 139.5, 134.0, 130.7, 129.8, 127.6, 126.6 (q, *J* = 280.2 Hz), 124.0, 123.9, 122.8, 121.8, 121.6, 121.5, 114.9, 113.8, 113.3, 109.7, 98.0, 72.8, 62.1 (q, *J* = 29.8 Hz), 55.4, 50.6; **¹⁹F NMR** (376 MHz, DMSO-*d*₆) δ -71.53; **IR** (neat): 3293, 2989, 1724, 1694, 1514, 1474, 1055, 761, 595 cm^{-1} ; **HRMS** (ESI): *m/z* for $[\text{M}+\text{H}]^+$: calcd 481.1370, found 481.1373; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 43.8 min and t_{minor} = 21.5 min.

(2*S*,2'*S*,4'*S*,5'*R*)-4'-(*m*-tolyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3ah):



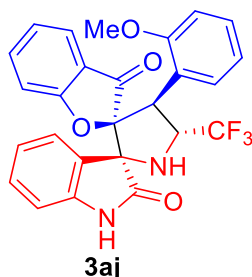
Compound **3ah** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 82% yield (77 mg, >20:1 dr): 95% *ee*; $[\alpha]_{\text{D}}^{20} = +137$ (c 1.0, in THF); **¹H NMR** (400 MHz, CDCl_3) δ 8.58 (s, 1H), 7.44 (d, *J* = 7.5 Hz, 1H), 7.35–7.26 (m, 1H), 7.17–7.08 (m, 3H), 7.05–6.99 (m, 1H), 6.97–6.92 (m, 2H), 6.86–6.60 (m, 4H), 4.98 (d, *J* = 10.9 Hz, 1H), 4.79–4.67 (m, 1H), 2.87 (d, *J* = 7.5 Hz, 1H), 2.11 (s, 3H); **¹³C NMR** (101 MHz, CDCl_3) δ 194.5, 175.9, 170.2, 141.8, 138.3, 137.8, 131.3, 130.6, 130.2, 128.8, 128.1, 126.2, 126.1, 125.7 (q, *J* = 279.9 Hz), 124.3, 124.1, 122.3, 122.1, 121.7, 112.3, 110.4, 97.7, 72.5, 62.5 (q, *J* = 30.7 Hz), 50.9, 21.3; **IR** (neat): 2988, 2901, 1732, 1609, 1463, 1066, 751, 641 cm^{-1} ; **¹⁹F NMR** (376 MHz, CDCl_3) δ -73.13. **HRMS** (ESI): *m/z* for $[\text{M}+\text{H}]^+$: calcd 465.1421, found 465.1425; **HPLC**: Daicel Chiralpak IA, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 16.60 min and t_{minor} = 11.31 min.

(2*S*,2'*S*,4'*S*,5'*R*)-4'-(3-chlorophenyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3ai):



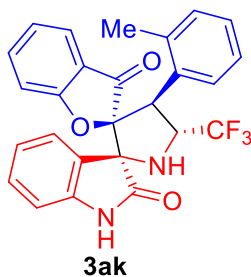
Compound **3ai** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 78% yield (76 mg, >20:1 dr): 91% *ee*; $[\alpha]_D^{20} = +97$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 10.44 (s, 1H), 7.61–7.50 (m, 2H), 7.39 (s, 1H), 7.30–7.16 (m, 5H), 7.13–7.03 (m, 1H), 6.84 (m, 2H), 6.66 (d, $J = 7.7$ Hz, 1H), 4.94–4.80 (m, 2H), 4.74 (d, $J = 8.1$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, DMSO- d_6) δ 194.0, 174.6, 169.8, 142.4, 139.2, 134.4, 132.8, 130.2, 130.2, 128.53, 128.1, 127.8, 127.1, 126.8 (q, $J = 280.2$ Hz), 123.6, 123.3, 122.5, 121.1, 120.9, 112.7, 109.3, 97.3, 72.3, 61.5 (q, $J = 29.7$ Hz), 49.8; $^{19}\text{F NMR}$ (376 MHz, DMSO- d_6) δ -71.61; **IR** (neat): 3301, 2988, 1724, 1694, 1514, 1474, 1055, 761, 595 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 485.0874, found 485.0874; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 22.62$ min and $t_{\text{minor}} = 16.53$ min.

(2S,2'S,4'S,5'R)-4'-(2-methoxyphenyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2'',3''-indoline]-2'',3-dione (3aj):



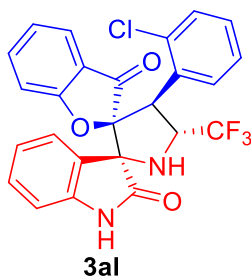
Compound **3aj** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 96% yield (93 mg, >20:1 dr): 98% *ee*; $[\alpha]_D^{20} = +162$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 10.31 (s, 1H), 7.63–7.41 (m, 3H), 7.27–7.12 (m, 2H), 7.09–7.00 (m, 2H), 6.88–6.72 (m, 4H), 6.69–6.54 (m, 1H), 5.60 (d, $J = 10.6$ Hz, 1H), 4.89–4.53 (m, 2H), 3.41 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, DMSO- d_6) δ 194.1, 175.2, 169.9, 157.9, 143.0, 139.0, 130.5, 130.0, 129.3, 127.6, 126.7 (q, $J = 280.2$ Hz), 124.3, 123.8, 122.5, 121.7, 121.4, 120.9, 120.4, 113.1, 111.8, 109.6, 98.1, 72.9, 62.6 (q, $J = 29.3$ Hz), 56.2, 42.4; $^{19}\text{F NMR}$ (376 MHz, DMSO- d_6) δ -71.60; **IR** (neat): 2988, 2972, 1733, 1541, 1464, 1117, 753, 618 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 481.1370, found 481.1372; **HPLC**: Daicel Chiralpak IA, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 11.67$ min and $t_{\text{minor}} = 14.83$ min.

(2S,2'S,4'S,5'R)-4'-(o-tolyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2'',3''-indoline]-2'',3-dione (3ak):



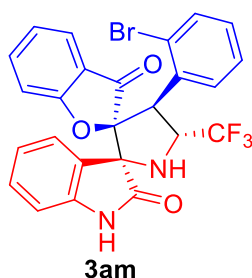
Compound **3ak** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 85% yield (79 mg, >20:1 dr): 93% *ee*; $[\alpha]_{\text{D}}^{20} = +108$ (c 1.0, in THF); **¹H NMR** (400 MHz, DMSO-*d*₆) δ 10.40 (s, 1H), 7.68–7.45 (m, 3H), 7.35–7.16 (m, 2H), 7.14–6.93 (m, 4H), 6.89–6.73 (m, 2H), 6.69–6.59 (m, 1H), 5.53 (d, *J* = 10.3 Hz, 1H), 4.89–4.42 (m, 2H), 2.31 (s, 3H); **¹³C NMR** (101 MHz, DMSO-*d*₆) δ 195.0, 175.2, 170.4, 142.9, 139.6, 137.4, 131.1, 130.9, 130.7, 128.9, 128.0, 127.6, 126.7 (q, *J* = 279.9 Hz), 126.1, 124.1, 124.0, 122.7, 121.6, 121.4, 113.3, 109.8, 98.3, 73.0, 63.3 (q, *J* = 29.3 Hz), 44.5, 20.0; **¹⁹F NMR** (376 MHz, DMSO-*d*₆) δ -71.78; **IR** (neat): 2989, 2972, 1728, 1511, 1463, 1130, 751, 637 cm^{-1} ; **HRMS** (ESI): *m/z* for $[\text{M}+\text{H}]^+$: calcd 465.1421, found 465.1423; **HPLC**: Daicel Chiralpak IA, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 8.83 min and t_{minor} = 11.87 min.

(2*S*,2'*S*,4'*S*,5'*R*)-4'-(2-chlorophenyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3al):

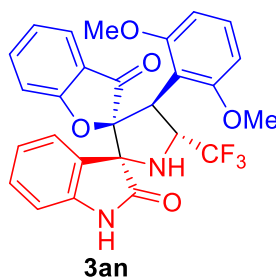


Compound **3al** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 90% yield (87 mg, >20:1 dr): 99% *ee*; $[\alpha]_{\text{D}}^{20} = +169$ (c 1.0, in THF); **¹H NMR** (400 MHz, DMSO-*d*₆) δ 10.39 (s, 1H), 7.84 – 7.78 (m, 1H), 7.62 – 7.54 (m, 2H), 7.34 – 7.27 (m, 2H), 7.26 – 7.21 (m, 2H), 7.21 – 7.04 (m, 3H), 6.89 – 6.77 (m, 2H), 6.67 (d, *J* = 7.7 Hz, 1H), 5.85 (d, *J* = 10.4 Hz, 1H), 4.82 – 4.63 (m, 2H); **¹³C NMR** (101 MHz, DMSO-*d*₆) δ 193.2, 174.5, 169.5, 142.5, 139.0, 133.9, 130.6, 130.2, 130.1, 129.5, 127.1, 126.8, 126.0 (q, *J* = 278.2 Hz), 123.6, 123.3, 122.4, 121.0, 120.9, 112.8, 109.3, 97.2, 72.6, 63.1 (q, *J* = 29.3 Hz), 44.7; **¹⁹F NMR** (376 MHz, DMSO-*d*₆) δ -71.83; **IR** (neat): 2988, 2969, 1731, 1613, 1473, 1055, 748, 560 cm^{-1} ; **HRMS** (ESI): *m/z* for $[\text{M}+\text{H}]^+$: calcd 485.0874, found 485.0874; **HPLC**: Daicel Chiralpak IA, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 12.34 min and t_{minor} = 15.48 min.

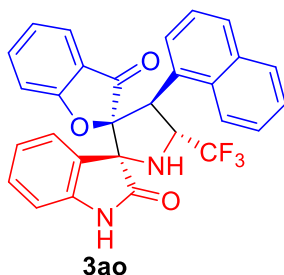
(2*S*,2'*S*,4'*S*,5'*R*)-4'-(2-bromophenyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3am):



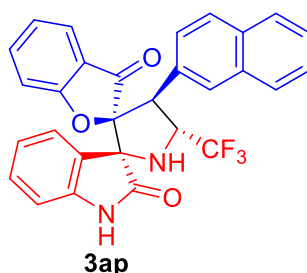
Compound **3am** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 90% yield (95 mg, >20:1 dr): 99% *ee*; $[\alpha]_D^{20} = +65$ (c 1.0, in THF); **¹H NMR** (400 MHz, DMSO-*d*₆) δ 10.36 (s, 1H), 7.90–7.76 (m, 1H), 7.71–7.42 (m, 3H), 7.38–7.00 (m, 5H), 6.95–6.56 (m, 3H), 5.86 (d, *J* = 9.5 Hz, 1H), 4.94–4.45 (m, 2H); **¹³C NMR** (101 MHz, DMSO-*d*₆) δ 193.6, 174.9, 170.0, 143.0, 139.5, 133.4, 132.4, 131.2, 130.7, 130.3, 127.8, 127.6, 126.50 (q, *J* = 278.8 Hz), 125.5, 124.10, 123.8, 122.9, 121.5, 121.4, 113.3, 109.8, 97.8, 73.0, 63.9 (q, *J* = 29.8 Hz), 47.9; **¹⁹F NMR** (376 MHz, DMSO-*d*₆) δ -71.67; **IR** (neat): 2988, 2972, 1733, 1612, 1474, 1055, 749, 560 cm^{-1} ; **HRMS** (ESI): *m/z* for $[M+H]^+$: calcd 529.0369, found 529.0372. **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 15.97 min and t_{minor} = 17.57 min.
(2*S*,2'*S*,4'*S*,5'*R*)-4'-(2,6-dimethoxyphenyl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3am):



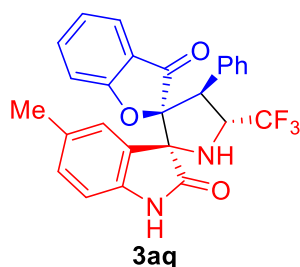
Compound **3an** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 75% yield (77 mg, >20:1 dr): 94% *ee*; $[\alpha]_D^{20} = +130$ (c 1.0, in THF); **¹H NMR** (400 MHz, DMSO-*d*₆) δ 10.19 (s, 1H), 7.53–7.41 (m, 2H), 7.22–7.14 (m, 1H), 7.11–6.94 (m, 3H), 6.83–6.71 (m, 2H), 6.66–6.36 (m, 3H), 5.86–5.58 (m, 2H), 4.55 (d, *J* = 8.7 Hz, 1H), 3.87 (s, 3H), 3.59 (s, 3H); **¹³C NMR** (101 MHz, DMSO-*d*₆) δ 194.0, 175.1, 169.9, 160.6, 158.5, 142.6, 138.2, 129.8, 129.2, 127.3, 126.8 (q, *J* = 280.0 Hz), 124.1, 123.2, 121.5, 121.0, 120.7, 112.3, 108.9, 108.4, 105.6, 104.2, 98.8, 71.7, 57.9 (q, *J* = 29.5 Hz), 56.3, 56.1, 41.0; **¹⁹F NMR** (376 MHz, DMSO-*d*₆) δ -71.85; **IR** (neat): 3331, 2925, 1736, 1613, 1595, 1474, 1108, 760, 620 cm^{-1} ; **HRMS** (ESI) : *m/z* for $[M+H]^+$: calcd 511.1475, found 511.1478; **HPLC**: Daicel Chiralpak IB, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 5.8 min and t_{minor} = 8.47 min.
(2*S*,2'*S*,4'*S*,5'*R*)-4'-(naphthalen-1-yl)-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3ao):



Compound **3ao** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 80% yield (80 mg, >20:1 dr): 97% *ee*; $[\alpha]_D^{20} = +139$ (c 1.0, in THF); **¹H NMR** (400 MHz, DMSO-*d*₆) δ 10.48 (s, 1H), 8.34–8.18 (m, 1H), 7.98–7.66 (m, 3H), 7.65–7.43 (m, 4H), 7.41–7.21 (m, 2H), 7.18–6.97 (m, 2H), 6.85–6.62 (m, 3H), 6.21 (d, *J* = 10.5 Hz, 1H), 5.02–4.70 (m, 2H); **¹³C NMR** (101 MHz, DMSO-*d*₆) δ 194.0, 174.9, 169.9, 142.4, 139.0, 133.3, 131.6, 130.2, 128.6, 128.3, 127.8, 127.1, 126.9, 126.5, 126.2 (q, *J* = 280.0 Hz) 125.7, 124.9, 123.5, 123.5, 122.6, 122.2, 121.2, 120.9, 112.8, 109.4, 97.2, 72.6, 63.0 (q, *J* = 29.5 Hz), 42.6; **¹⁹F NMR** (376 MHz, DMSO-*d*₆) δ -71.76; **IR** (neat): 3243, 2972, 1741, 1692, 1511, 1472, 1055, 754, 580 cm^{-1} ; HRMS (ESI): *m/z* for $[M+H]^+$: calcd 501.1421, found 501.1422; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 27.20 min and t_{minor} = 14.80 min; HPLC: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 8.59 min and t_{minor} = 16.53 min.
(2*S*,2'*S*,4'*S*,5'*R*)-4'-(naphthalen-2-yl)-5'-(trifluoromethyl)-3*H*-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3ap):

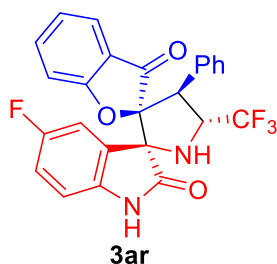


Compound **3ap** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 82% yield (82 mg, >20:1 dr): 90% *ee*; $[\alpha]_D^{20} = -83$ (c 1.0, in THF); **¹H NMR** (400 MHz, DMSO-*d*₆) δ 10.44 (s, 1H), 7.93–7.71 (m, 4H), 7.63–7.40 (m, 5H), 7.28–7.04 (m, 3H), 6.85–6.60 (m, 3H), 5.15–4.89 (m, 2H), 4.80 (d, *J* = 9.0 Hz, 1H); **¹³C NMR** (101 MHz, DMSO-*d*₆) δ 194.7, 175.3, 170.3, 142.9, 139.5, 133.0, 132.8, 130.7, 130.1, 128.8, 128.2, 127.8, 127.7, 126.9, 126.8, 126.7 (q, *J* = 279.8 Hz), 124.0, 123.9, 122.8, 121.6, 121.4, 113.2, 109.8, 98.3, 72.9, 62.2 (q, *J* = 29.8 Hz), 50.8; **¹⁹F NMR** (376 MHz, DMSO-*d*₆) δ -71.48; **IR** (neat): 2988, 2972, 1732, 1617, 1474, 1055, 752, 680 cm^{-1} ; HRMS (ESI): *m/z* for $[M+H]^+$: calcd 501.1421, found 501.1426; **HPLC**: Daicel Chiralpak IC, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 8.71 min and t_{minor} = 25.37 min.
(2*S*,2'*S*,4'*S*,5'*R*)-5'-methyl-4'-phenyl-5'-(trifluoromethyl)-3*H*-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3aq):



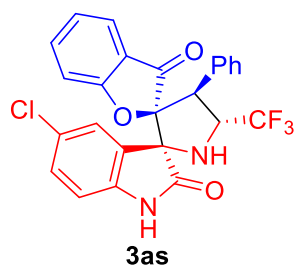
Compound **3aq** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 75% yield (70 mg, >20:1 dr): 94% *ee*; $[\alpha]_{\text{D}}^{20} = +90$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 10.29 (s, 1H), 7.60–7.45 (m, 1H), 7.38 (s, 1H), 7.33–7.27 (m, 2H), 7.26–7.09 (m, 5H), 6.93–6.73 (m, 2H), 6.52 (d, $J = 7.8$ Hz, 1H), 4.91–4.73 (m, 2H), 4.67 (d, $J = 8.8$ Hz, 1H), 2.12 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, DMSO- d_6) δ 194.7, 175.2, 170.4, 140.3, 139.4, 132.5, 130.7, 130.2, 129.4, 128.8, 128.5, 126.6 (q, $J = 279.8$ Hz), 124.0, 123.9, 121.5, 113.1, 109.4, 98.1, 72.9, 62.0 (q, $J = 29.3$ Hz), 50.6, 20.2; $^{19}\text{F NMR}$ (376 MHz, DMSO- d_6) δ -71.56; **IR** (neat): 2988, 2960, 1731, 1509, 1463, 1128, 1066, 752, 698 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 465.1421, found 465.1422; HPLC: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 12.94$ min and $t_{\text{minor}} = 10.16$ min.

(2S,2'S,4'S,5'R)-5''-fluoro-4'-phenyl-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3ar):



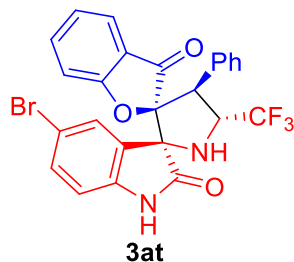
Compound **3ar** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 70% yield (65 mg, >20:1 dr): 91% *ee*; $[\alpha]_{\text{D}}^{20} = +134$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 10.46 (s, 1H), 7.60–7.54 (m, 1H), 7.39–7.29 (m, 3H), 7.28–7.10 (m, 5H), 6.97–6.84 (m, 2H), 6.64 (dd, $J = 8.5, 4.4$ Hz, 1H), 4.95–4.56 (m, 3H); $^{13}\text{C NMR}$ (101 MHz, DMSO- d_6) δ 194.4, 175.2, 170.2, 157.6 (d, $J = 237.3$ Hz), 139.7, 139.0, 132.2, 129.4, 128.8, 128.5, 126.5 (q, $J = 280.2$ Hz), 125.6, 125.5, 124.1, 123.0, 121.4, 117.1 (d, $J = 23.2$ Hz), 115.4 (d, $J = 25.4$ Hz), 113.2, 110.5 (d, $J = 8.0$ Hz), 97.8, 73.0, 61.9 (q, $J = 29.7$ Hz), 50.5; $^{19}\text{F NMR}$ (376 MHz, DMSO- d_6) δ -71.65, -121.99; **IR** (neat): 2988, 2976, 1735, 1511, 1488, 1066, 750, 639 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 469.1170, found 469.1171, **HPLC**: Daicel Chiralpak IC, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 6.67$ min and $t_{\text{minor}} = 13.81$ min.

(2S,2'S,4'S,5'R)-5''-chloro-4'-phenyl-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3as):



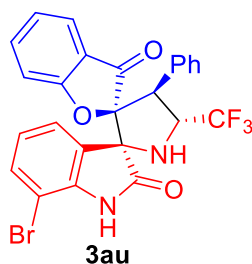
Compound **3as** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 72% yield (70 mg, >20:1 dr): 93% *ee*; $[\alpha]_{\text{D}}^{20} = +68$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 10.59 (s, 1H), 7.61–7.54 (m, 2H), 7.35–7.30 (m, 2H), 7.27–7.10 (m, 6H), 6.90–6.82 (m, 1H), 6.67 (d, $J = 8.3$ Hz, 1H), 4.95–4.54 (m, 3H); $^{13}\text{C NMR}$ (101 MHz, DMSO- d_6) δ 194.4, 174.9, 170.3, 141.7, 139.7, 132.1, 130.4, 129.4, 128.8, 128.5, 127.9, 126.5 (q, $J = 281.1$ Hz), 125.8, 125.5, 124.1, 123.0, 121.4, 113., 111.2, 97.8, 72.9, 61.9 (q, $J = 29.7$ Hz), 50.6; $^{19}\text{F NMR}$ (376 MHz, DMSO- d_6) δ -71.72; **IR** (neat): 2991, 2970, 1734, 1511, 1488, 1066, 750, 639 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 485.0874, found 485.0875; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.2 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 13.85$ min and $t_{\text{minor}} = 12.11$ min.

(2S,2'S,4'S,5'R)-5''-bromo-4'-phenyl-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3at):



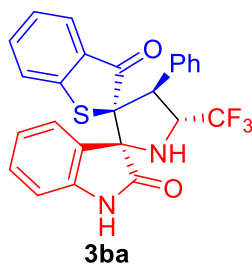
Compound **3at** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 90% yield (95 mg, >20:1 dr): 97% *ee*; $[\alpha]_{\text{D}}^{20} = +19$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 10.59 (s, 1H), 7.73–7.65 (m, 1H), 7.60–7.49 (m, 1H), 7.36–7.02 (m, 8H), 6.92–6.82 (m, 1H), 6.63 (d, $J = 8.3$ Hz, 1H), 4.92–4.66 (m, 3H); $^{13}\text{C NMR}$ (101 MHz, DMSO- d_6) δ 194.6, 174.9, 170.3, 141.9, 139.8, 133.3, 132.1, 130.6, 129.4, 128.8, 128.6, 126.5 (q, $J = 280.8$ Hz), 126.1, 124.1, 123.1, 121.3, 113.3, 113.1, 111.8, 97.8, 72.9, 61.9 (q, $J = 29.5$ Hz), 50.5; $^{19}\text{F NMR}$ (376 MHz, DMSO- d_6) δ -71.73; **IR** (neat): 3380, 2988, 2972, 1735, 1488, 1066, 750, 639 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 529.0369, found 529.0369; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 16.78$ min and $t_{\text{minor}} = 13.46$ min.

(2S,2'S,4'S,5'R)-7''-bromo-4'-phenyl-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3au):



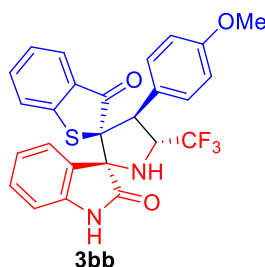
Compound **3au** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 95% yield (101 mg, >20:1 dr): 97% *ee*; $[\alpha]_{\text{D}}^{20} = +262$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO-*d*₆) δ 10.77 (s, 1H), 7.61–7.49 (m, 2H), 7.34–7.08 (m, 8H), 6.90–6.72 (m, 2H), 4.99–4.59 (m, 3H); $^{13}\text{C NMR}$ (101 MHz, DMSO-*d*₆) δ 194.1, 174.6, 169.8, 141.7, 139.1, 133.1, 131.7, 128.9, 128.3, 128.0, 126.3, 126.1 (q, $J = 280.5$ Hz), 125.3, 123.6, 122.7, 122.5, 120.8, 112.8, 101.5, 97.3, 73.0, 61.5 (q, $J = 30.0$ Hz), 50.2; $^{19}\text{F NMR}$ (376 MHz, DMSO-*d*₆) δ -71.60; **IR** (neat): 2958, 2952, 2951, 1733, 1512, 1452, 1130, 1077, 798, 638 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 529.0369, found 529.0369; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 5.58$ min and $t_{\text{minor}} = 6.38$ min.

(2*S*,2'*R*,4'*S*,5'*R*)-4'-phenyl-5'-(trifluoromethyl)-3*H*-dispiro[benzo[*b*]thiophene-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3ba):



Compound **3ba** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 70% yield (76 mg, >20:1 dr): 97% *ee*; $[\alpha]_{\text{D}}^{20} = +246$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO-*d*₆) δ 10.29 (s, 1H), 7.88–7.77 (m, 1H), 7.38–7.11 (m, 8H), 7.02 (m, 2H), 6.88–6.75 (m, 1H), 6.58 (d, $J = 7.7$ Hz, 1H), 5.56 (d, $J = 11.0$ Hz, 1H), 4.80–4.56 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, DMSO-*d*₆) δ 197.0, 176.7, 149.5, 142.9, 136.8, 133.8, 130.8, 130.6, 130.0, 128.5, 128.21, 128.19, 126.5 (q, $J = 281.1$ Hz), 126.2, 125.5, 124.4, 120.5, 109.2, 81.7, 74.2, 60.5 (q, $J = 29.7$ Hz), 49.9; $^{19}\text{F NMR}$ (376 MHz, DMSO-*d*₆) δ -70.28; **IR** (neat): 2988, 2972, 1732, 1394, 1283, 1066, 752, 598 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 467.1036, found 467.1038; **HPLC**: Daicel Chiralpak IB, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 7.40$ min and $t_{\text{minor}} = 7.00$ min.

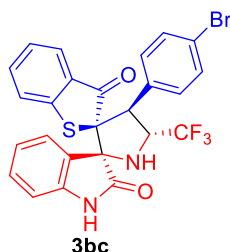
(2*S*,2'*R*,4'*S*,5'*R*)-4'-(4-methoxyphenyl)-5'-(trifluoromethyl)-3*H*-dispiro[benzo[*b*]thiophene-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3bb):



Compound **3bb** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 60% yield (60 mg, >20:1 dr): 88% *ee*; $[\alpha]_{\text{D}}^{20} = +241$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, DMSO-*d*₆) δ 10.28 (s, 1H), 7.93–7.68 (m, 1H), 7.42–7.15 (m, 5H), 7.11–6.92 (m, 2H), 6.87–6.46 (m, 4H), 5.50 (d, $J = 10.4$ Hz, 1H), 4.82–4.38 (m, 2H), 3.63 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, DMSO-*d*₆) δ 197.1, 176.8, 158.9, 149.6, 142.9, 136.7, 131.1, 130.9, 130.5, 128.4, 126.6 (q, $J = 280.7$ Hz), 126.2, 125.6, 125.4, 125.2, 124.4, 120.5, 113.6, 109.2, 81.9, 74.2, 60.7 (q, $J = 29.8$ Hz), 55.3, 49.3;

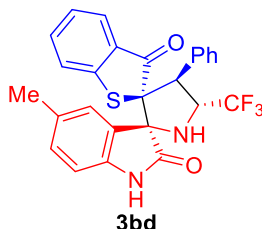
¹⁹F NMR (376 MHz, DMSO-d₆) δ -70.25; **IR** (neat): 2988, 2972, 1732, 1617, 1515, 1412, 1132, 1056, 753, 614 cm⁻¹; **HRMS** (ESI): m/z for [M+H]⁺: calcd 497.1141, found 497.1145; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 7.89 min and t_{minor} = 11.99 min.

(2S,2'R,4'S,5'R)-4'-(4-bromophenyl)-5'-(trifluoromethyl)-3H-dispiro[benzo[b]thiophene-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3bc):



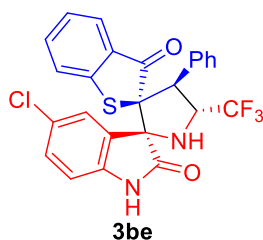
Compound **3bc** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 70% yield (76 mg, >20:1 dr): 89% *ee*; [α]_D²⁰ = +238 (c 1.0, in THF); **¹H NMR** (400 MHz, DMSO-d₆) δ 10.32 (s, 1H), 8.01–7.72 (m, 1H), 7.58–6.96 (m, 9H), 6.89–6.42 (m, 2H), 5.53 (d, *J* = 8.0 Hz, 1H), 4.92–4.23 (m, 2H); **¹³C NMR** (101 MHz, DMSO-d₆) δ 196.8, 176.7, 149.3, 142.9, 136.9, 133.3, 132.2, 131.3, 130.7, 130.6, 128.4, 126.5 (q, *J* = 281.8 Hz), 126.3, 125.7, 124.9, 124.5, 121.6, 120.5, 109.2, 81.4, 74.2, 60.4 (q, *J* = 31.3 Hz), 49.3, **¹⁹F NMR** (376 MHz, DMSO-d₆) δ -70.39; **IR** (neat): 2988, 2972, 1732, 1393, 1283, 1066, 753, 562 cm⁻¹; **HRMS** (ESI): m/z for [M+H]⁺: calcd 545.0141, found 545.0145; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 28.05 min and t_{minor} = 16.90 min.

(2S,2'R,4'S,5'R)-5''-methyl-4'-phenyl-5'-(trifluoromethyl)-3H-dispiro[benzo[b]thiophene-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3bd):



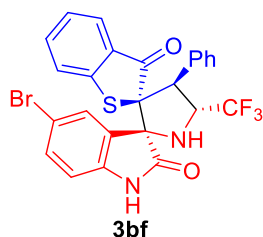
Compound **3bd** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 80% yield (77 mg, >20:1 dr): 91% *ee*; [α]_D²⁰ = +179 (c 1.0, in THF); **¹H NMR** (400 MHz, DMSO-d₆) δ 10.18 (s, 1H), 7.67 (s, 1H), 7.40–7.32 (m, 2H), 7.32–7.25 (m, 3H), 7.24–7.10 (m, 3H), 7.03–6.81 (m, 2H), 6.53–6.36 (m, 1H), 5.64–5.37 (m, 1H), 4.66 (m, 2H), 2.17 (s, 3H); **¹³C NMR** (101 MHz, DMSO-d₆) δ 197.0, 176.8, 149.5, 140.4, 136.8, 133.9, 130.9, 130.7, 129.9, 129.2, 129.1, 128.2, 128.2, 126.5 (q, *J* = 280.1 Hz), 126.1, 125.5, 125.2, 124.3, 108.9, 81.7, 74.2, 60.5 (q, *J* = 30.1 Hz), 50.0, 21.3; **¹⁹F NMR** (376 MHz, DMSO-d₆) δ -70.29; **IR** (neat): 2988, 2975, 1731, 1393, 1283, 1131, 1066, 697, 620 cm⁻¹; **HRMS** (ESI): m/z for [M+H]⁺: calcd 481.1192, found 481.1195; **HPLC**: Daicel Chiralpak IC, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 6.52 min and t_{minor} = 19.06 min.

(2S,2'R,4'S,5'R)-5''-chloro-4'-phenyl-5'-(trifluoromethyl)-3H-dispiro[benzo[b]thiophene-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3be):



Compound **3be** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 83% yield (83 mg, >20:1 dr): 98% *ee*; $[\alpha]_D^{20} = +113$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.75 (s, 1H), 7.75–7.67 (m, 1H), 7.39–7.15 (m, 5H), 7.13–6.96 (m, 5H), 6.90–6.80 (m, 1H), 6.64–6.57 (m, 1H), 5.57 (d, $J = 11.0$ Hz, 1H), 4.66–4.50 (m, 1H), 2.74 (d, $J = 6.6$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 196.5, 177.9, 148.8, 140.4, 136.1, 132.4, 130.9, 130.4, 129.7, 128.0, 127.8, 127.7, 126.9, 126.6, 126.5, 125.4 (q, $J = 281.1$ Hz), 125.1, 123.7, 110.9, 80.7, 73.8, 61.0 (q, $J = 31.1$ Hz), 50.2; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -71.91; **IR** (neat): 3357, 2988, 2972, 1750, 1634, 1586, 1450, 1131, 754, 625 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 501.0646, found 501.0649; **HPLC**: Daicel Chiralpak IC, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 4.25$ min and $t_{\text{minor}} = 8.49$ min.

(2S,2'R,4'S,5'R)-5''-bromo-4'-phenyl-5'-(trifluoromethyl)-3H-dispiro[benzo[b]thiophene-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (3bf):



Compound **3bf** was purified by flash chromatography (petroleum ether/EtOAc = 3/1) to afford a yellow solid in 85% yield (93 mg, >20:1 dr): 97% *ee*; $[\alpha]_D^{20} = +48$ (c 1.0, in THF); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.79 (s, 1H), 7.87–7.78 (m, 1H), 7.38–7.27 (m, 2H), 7.21–6.96 (m, 7H), 6.89–6.79 (m, 1H), 6.58 (d, $J = 8.3$ Hz, 1H), 5.57 (d, $J = 11.0$ Hz, 1H), 4.62–4.49 (m, 1H), 2.74 (d, $J = 6.6$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 196.5, 177.8, 148.8, 140.9, 136.1, 133.3, 132.5, 130.9, 130.4, 129.8, 128.0, 127.8, 127.3, 126.5, 125.4 (q, $J = 280.9$ Hz), 125.1, 123.7, 113.8, 111.5, 80.8, 73.8, 61.1 (q, $J = 31.0$ Hz), 50.2; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -71.91; **IR** (neat): 3354, 2988, 2959, 1740, 1585, 1474, 1128, 1066, 753, 695 cm^{-1} ; **HRMS** (ESI): m/z for $[\text{M}+\text{H}]^+$: calcd 545.0141, found 545.0140; **HPLC**: Daicel Chiralpak IC, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 4.93$ min and $t_{\text{minor}} = 9.00$ min.

Derivatization of 3aa

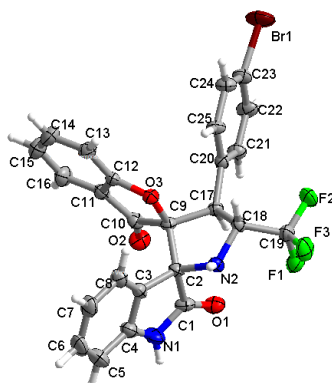
To a stirred solution of **3aa** (0.3 mmol, 1.0 equiv.) in DMF (2.0 mL) at 0 °C, was added NaH (60% dispersion in mineral oil, 15 mg, 0.36 mmol, 1.2 equiv.) in one portion and stirred for 5 minutes. Benzyl bromide (1.2 equiv.) was added at 0 °C, and continued to be stirred at room temperature. The progress of the reaction was monitored by TLC. The reaction mixture was then poured into saturated aqueous NH₄Cl (2.0 mL) and extracted with ethyl acetate (2.0 mL). The combined organic portions were washed with water and brine, dried over Na₂SO₄, and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel to give products **4**.

(2*S*,2'*S*,4'*S*,5'*R*)-1''-benzyl-4'-phenyl-5'-(trifluoromethyl)-3H-dispiro[benzofuran-2,3'-pyrrolidine-2',3''-indoline]-2'',3-dione (4): Compound **4** was purified by flash chromatography (petroleum ether/EtOAc = 6/1) to afford a white solid in 95% yield (86 mg, >20:1 dr): 94% *ee*; [α]_D²⁰ = +123 (c 1.0, in THF); ¹H NMR (400 MHz, CDCl₃) δ 7.57–7.50 (m, 1H), 7.42–7.29 (m, 7H), 7.27–7.20 (m, 3H), 7.18–7.08 (m, 3H), 7.05–6.97 (m, 2H), 6.86–6.79 (m, 1H), 6.76 (t, *J* = 7.4 Hz, 1H), 6.48 (d, *J* = 7.8 Hz, 1H), 5.25–5.14 (m, 2H), 4.93–4.77 (m, 1H), 4.57 (d, *J* = 15.6 Hz, 1H), 2.95 (d, *J* = 8.4 Hz, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 194.3, 174.1, 170.1, 143.6, 138.4, 135.4, 131.3, 130.5, 129.3, 129.1, 128.8, 128.3, 128.1, 127.6, 127.0, 125.8, 124.3, 123.7, 122.5, 122.2, 121.7, 112.3, 109.5, 97.7, 72.1, 62.4 (q, *J* = 31.0 Hz), 51.1, 44.4; ¹⁹F NMR (376 MHz, CDCl₃) δ -73.23; IR (neat): 2920, 1724, 1610, 1127, 856, 752, 698 cm⁻¹; HRMS (ESI): *m/z* for [M+H]⁺: calcd 541.1734, found 541.1730; HPLC: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 60/40, flow rate = 1 mL/min, λ = 254 nm, *t*_{major} = 6.12 min and *t*_{minor} = 8.86 min.

References

1. Trost, B. M.; Ito, H. *J. Am. Chem. Soc.* **2000**, *122*, 12003-12004.
2. (a) Hua, Y.-Z.; Chen, J.-W.; Yang, H.; Wang, M.-C. *J. Org. Chem.* **2018**, *83*, 1160–1166; (b) Liu, M.-M.; Yang, X.-C.; Hua, Y.-Z.; Chang, J.-B.; Wang, M.-C. *Org. Lett.* **2019**, *21*, 2111–2115.
3. Yang, X.-C.; Liu, M.-M.; Mathey, F.; Yang, H.; Hua, Y.-Z.; Wang, M.-C. *J. Org. Chem.* **2019**, *84*, 7762–7093.
4. (a) Wu, D.; Mei, H.; Tan, P.; Lu, W.; Zhu, J.; Wang, W.; Huang, J.; Li, J. *Tetrahedron Lett* **2015**, *56*, 4383–4387; (b) Formánek, B.; Tauchman, J.; Císařová, I.; Veselý, J. *J. Org. Chem.* **2020**, *85*, 8510–8521.
5. Li, X.; Su, J.; Liu, Z.; Zhu, Y.; Dong, Z.; Qiu, S.; Wang, J.; Lin, L.; Shen, Z.; Yan, W.; Wang, K.; Wang, R. *Org. Lett.* **2016**, *18*, 956-959.

X-ray crystal structure of 3af (PE/EA = 4/1 at rt)



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 202008157

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 202008157

Bond precision:	C-C = 0.0129 Å	Wavelength=1.54184	
Cell:	a=7.0824 (5)	b=9.9984 (8)	c=11.3089 (9)
	alpha=113.621 (8)	beta=101.541 (7)	gamma=100.342 (7)
Temperature:	293 K		
	Calculated	Reported	
Volume	688.15 (11)	688.15 (10)	
Space group	P 1	P 1	
Hall group	P 1	P 1	
Moiety formula	C25 H16 Br F3 N2 O3, C4 H8 O2	C25 H16 Br F3 N2 O3, C4 H8 O2	
Sum formula	C29 H24 Br F3 N2 O5	C29 H24 Br F3 N2 O5	
Mr	617.40	617.41	
Dx, g cm ⁻³	1.490	1.490	
Z	1	1	
Mu (mm ⁻¹)	2.584	2.584	
F000	314.0	314.0	
F000'	314.33		
h, k, lmax	8, 11, 13	8, 11, 13	
Nref	4928 [2464]	3900	
Tmin, Tmax	0.694, 0.772	0.436, 1.000	
Tmin'	0.630		
Correction method=	# Reported T Limits: Tmin=0.436 Tmax=1.000		
AbsCorr =	MULTI-SCAN		
Data completeness=	1.58/0.79	Theta(max)= 67.108	
R(reflections)=	0.0487 (3281)	wR2(reflections)= 0.1303 (3900)	
S =	1.028	Npar= 372	

The following ALERTS were generated. Each ALERT has the format
test-name ALERT alert-type alert-level.
Click on the hyperlinks for more details of the test.

● **Alert level C**

PLAT090_ALERT_3_C	Poor Data / Parameter Ratio (Zmax > 18)	6.62	Note
PLAT245_ALERT_2_C	U(iso) H1 Smaller than U(eq) N1 by	0.021	Ang**2
PLAT260_ALERT_2_C	Large Average Ueq of Residue Including O4	0.157	Check
PLAT341_ALERT_3_C	Low Bond Precision on C-C Bonds	0.0129	Ang.
PLAT915_ALERT_3_C	No Flack x Check Done: Low Friedel Pair Coverage	58	%

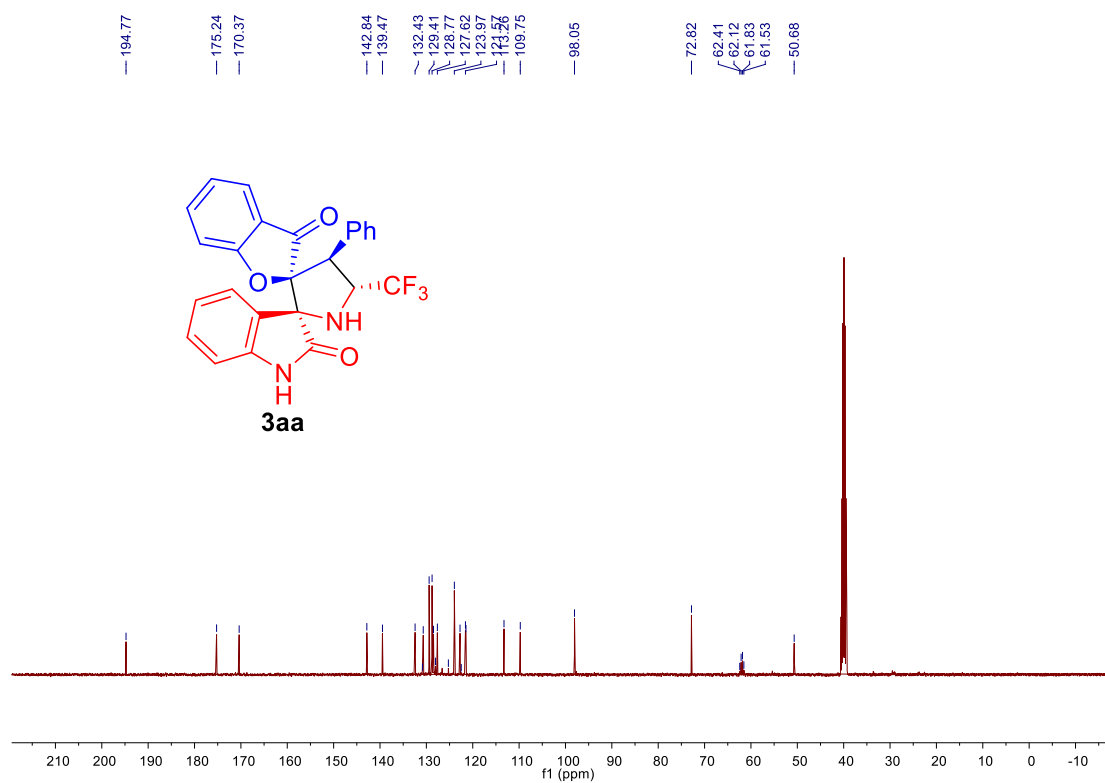
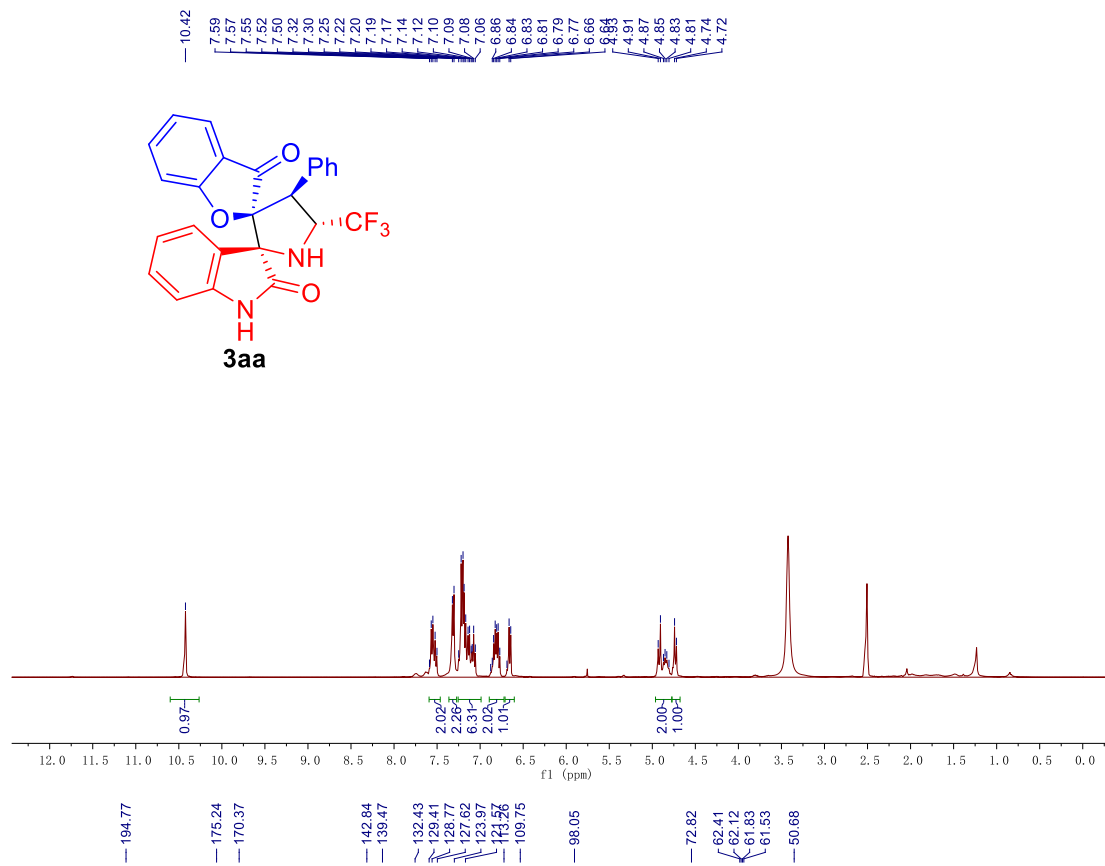
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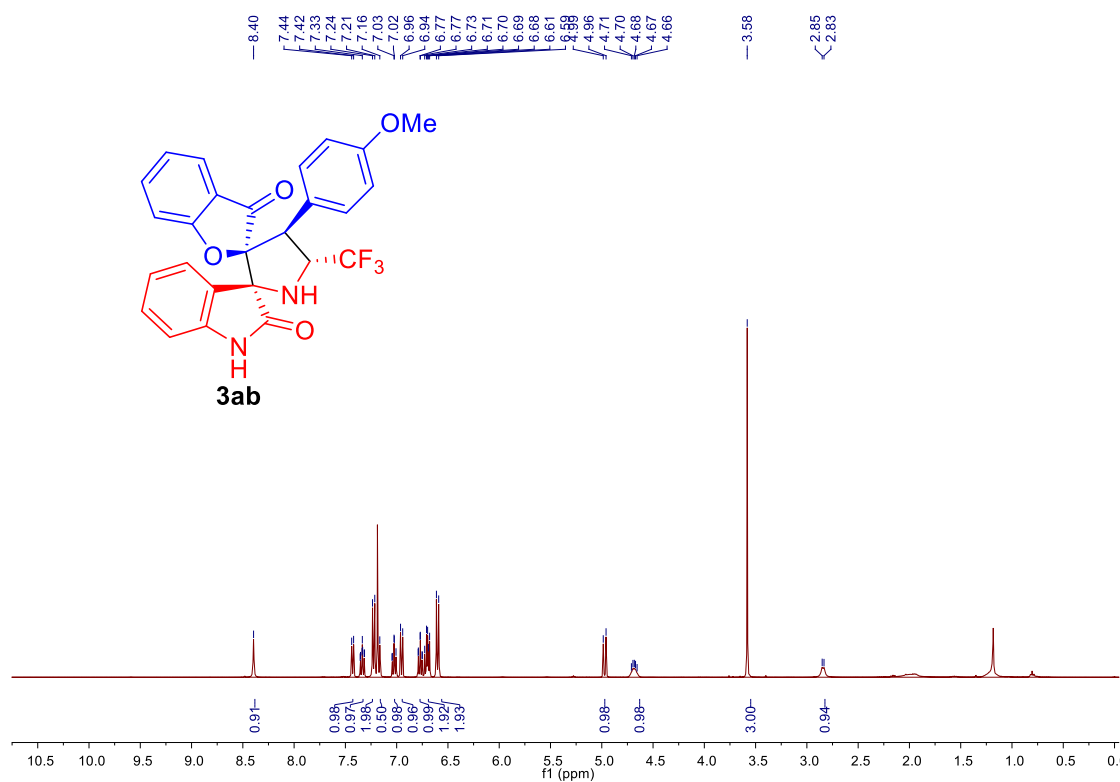
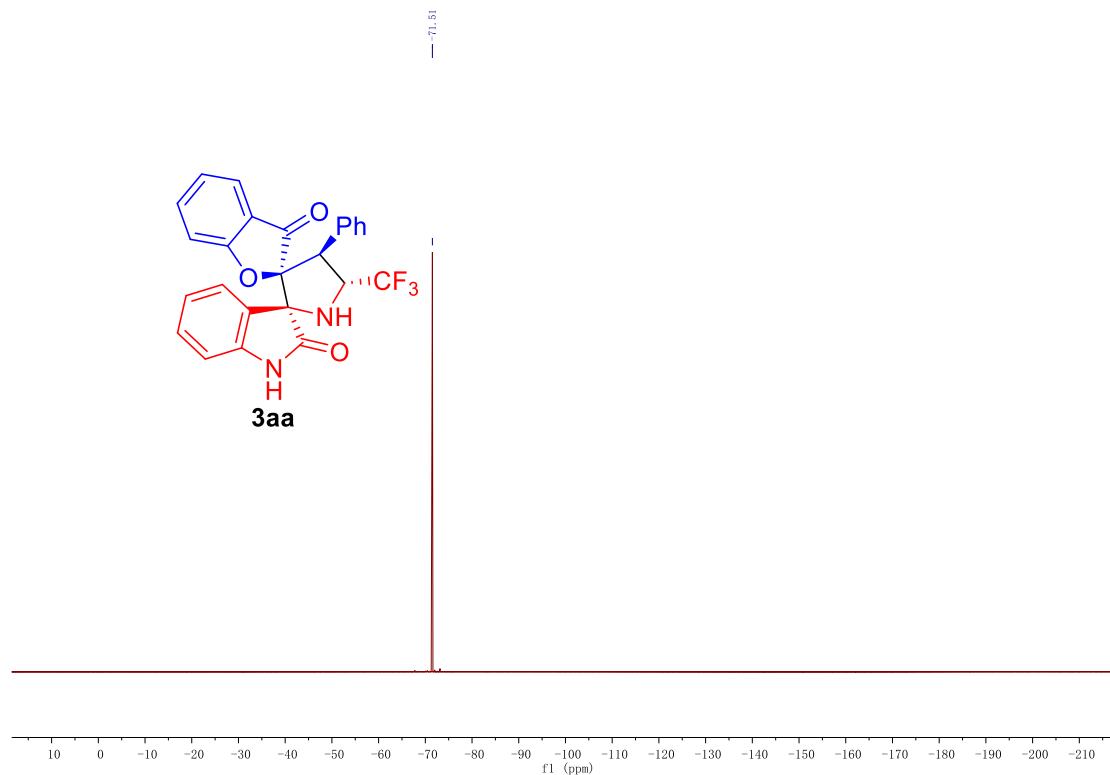
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PLAT003_ALERT_2_G	Number of Uiso or Uij Restrained non-H Atoms ...	2	Report
PLAT012_ALERT_1_G	No shelx_res_checksum Found in CIF		Please Check
PLAT172_ALERT_4_G	The CIF-Embedded .res File Contains DFIX Records	6	Report
PLAT186_ALERT_4_G	The CIF-Embedded .res File Contains ISOR Records	1	Report
PLAT199_ALERT_1_G	Reported _cell_measurement_temperature	293	Check
PLAT200_ALERT_1_G	Reported _diffn_ambient_temperature	293	Check
PLAT242_ALERT_2_G	Low 'MainMol' Ueq as Compared to Neighbors of	C19	Check
PLAT398_ALERT_2_G	Deviating C-O-C Angle From 120 for O3	107.6	Degree
PLAT791_ALERT_4_G	Model has Chirality at C2 (Sohnke SpGr)	S	Verify
PLAT791_ALERT_4_G	Model has Chirality at C9 (Sohnke SpGr)	S	Verify
PLAT791_ALERT_4_G	Model has Chirality at C17 (Sohnke SpGr)	S	Verify
PLAT791_ALERT_4_G	Model has Chirality at C18 (Sohnke SpGr)	R	Verify
PLAT860_ALERT_3_G	Number of Least-Squares Restraints	21	Note
PLAT909_ALERT_3_G	Percentage of I>2sig(I) Data at Theta(Max) Still	63%	Note
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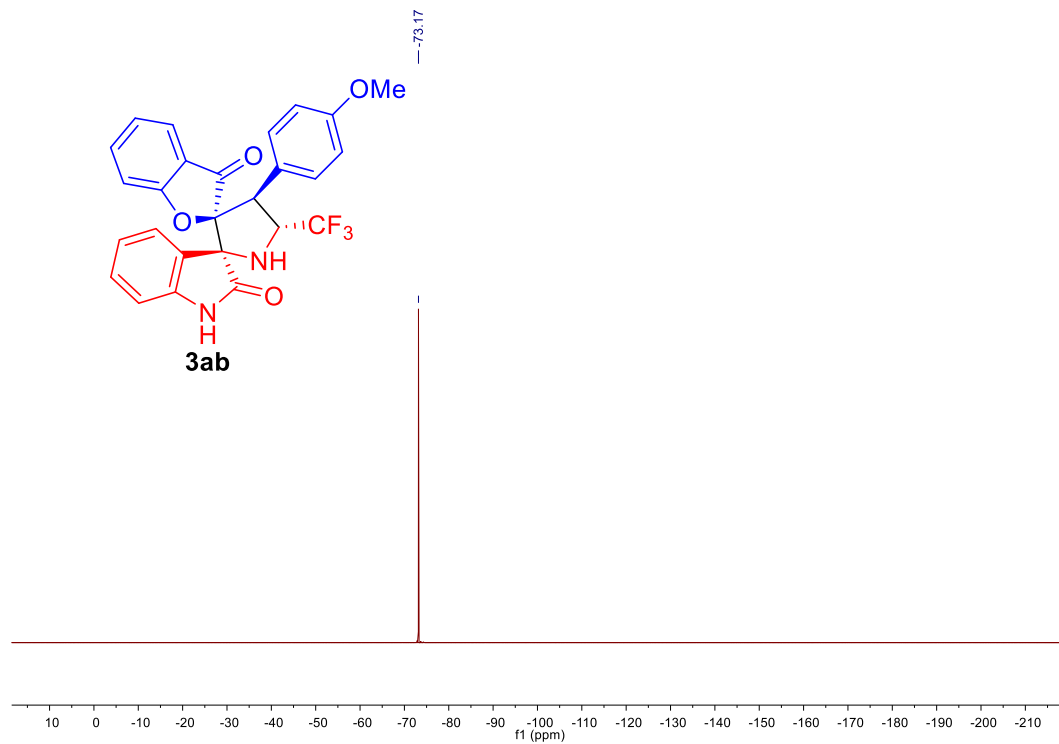
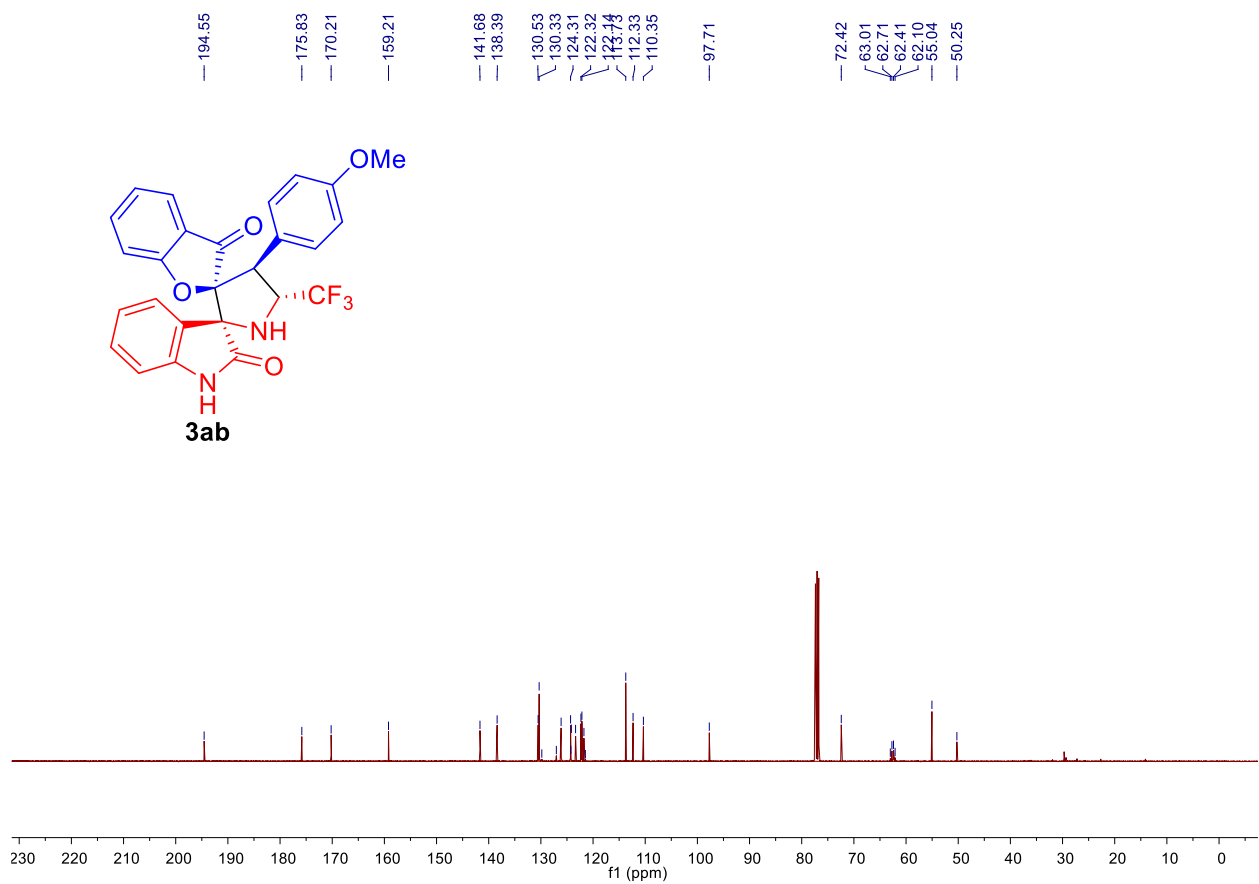
0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
5 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
16 **ALERT level G** = General information/check it is not something unexpected

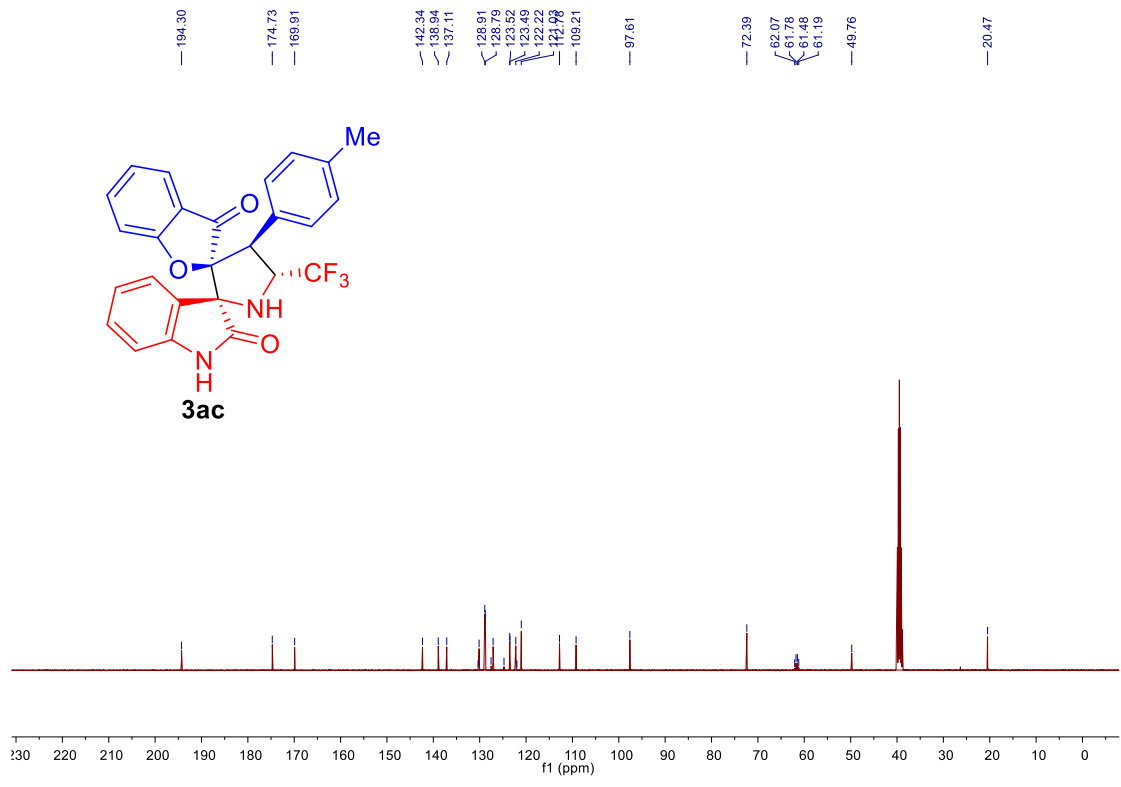
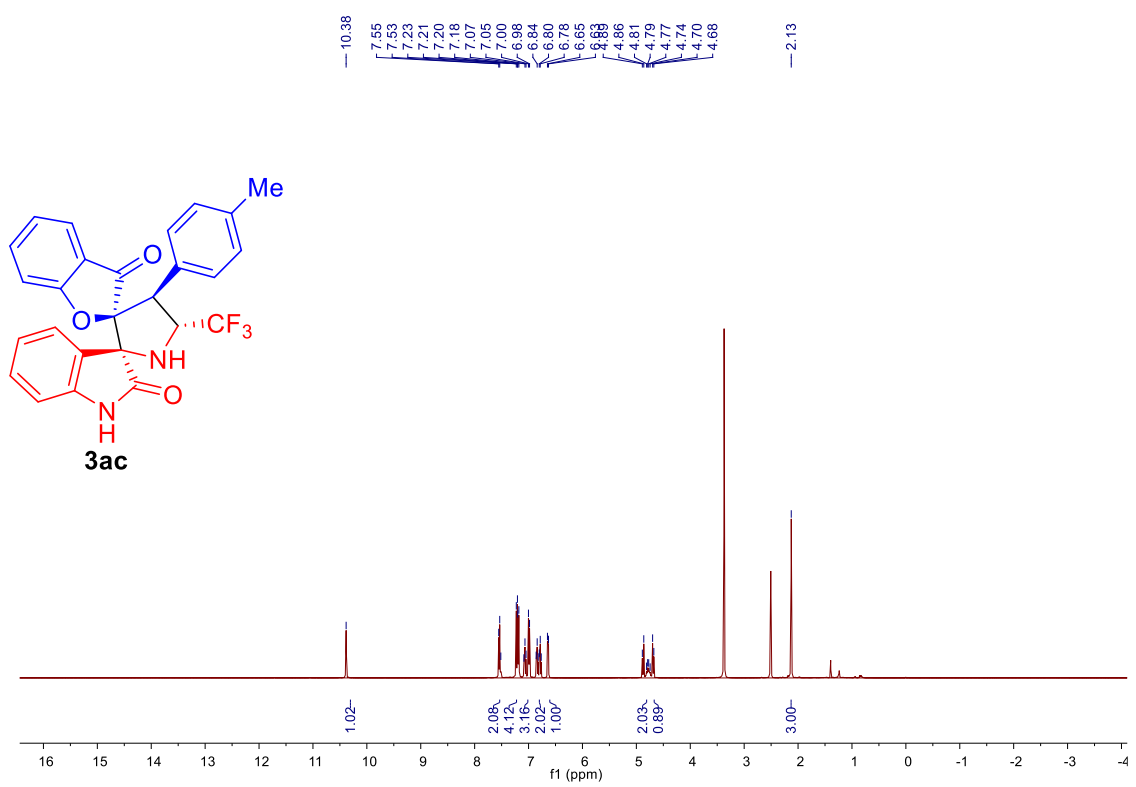
3 **ALERT type 1** CIF construction/syntax error, inconsistent or missing data
7 **ALERT type 2** Indicator that the structure model may be wrong or deficient
5 **ALERT type 3** Indicator that the structure quality may be low
6 **ALERT type 4** Improvement, methodology, query or suggestion
0 **ALERT type 5** Informative message, check

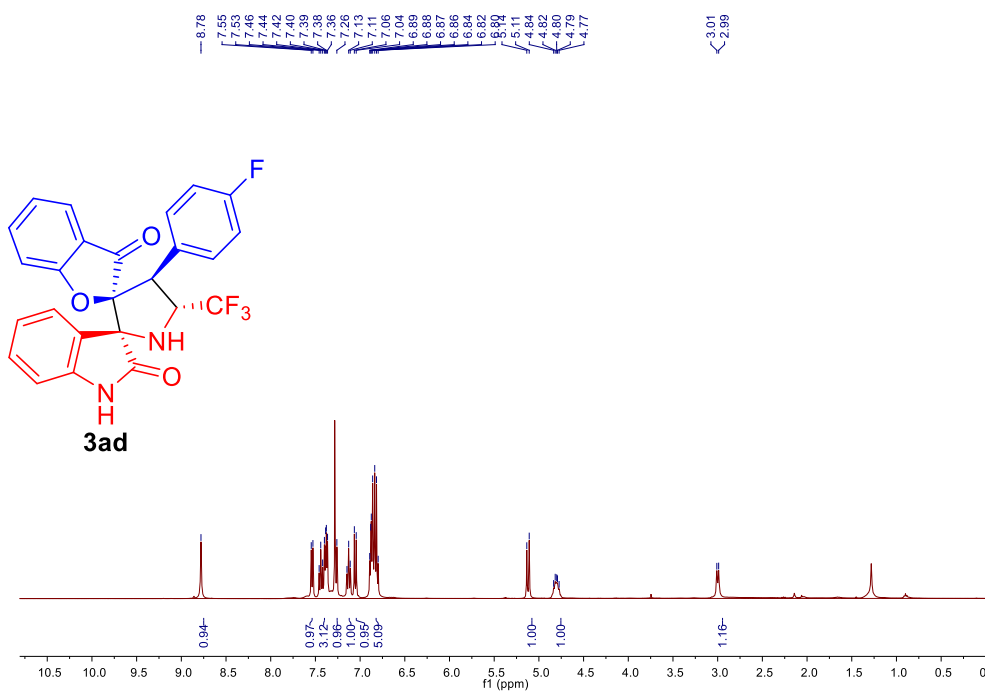
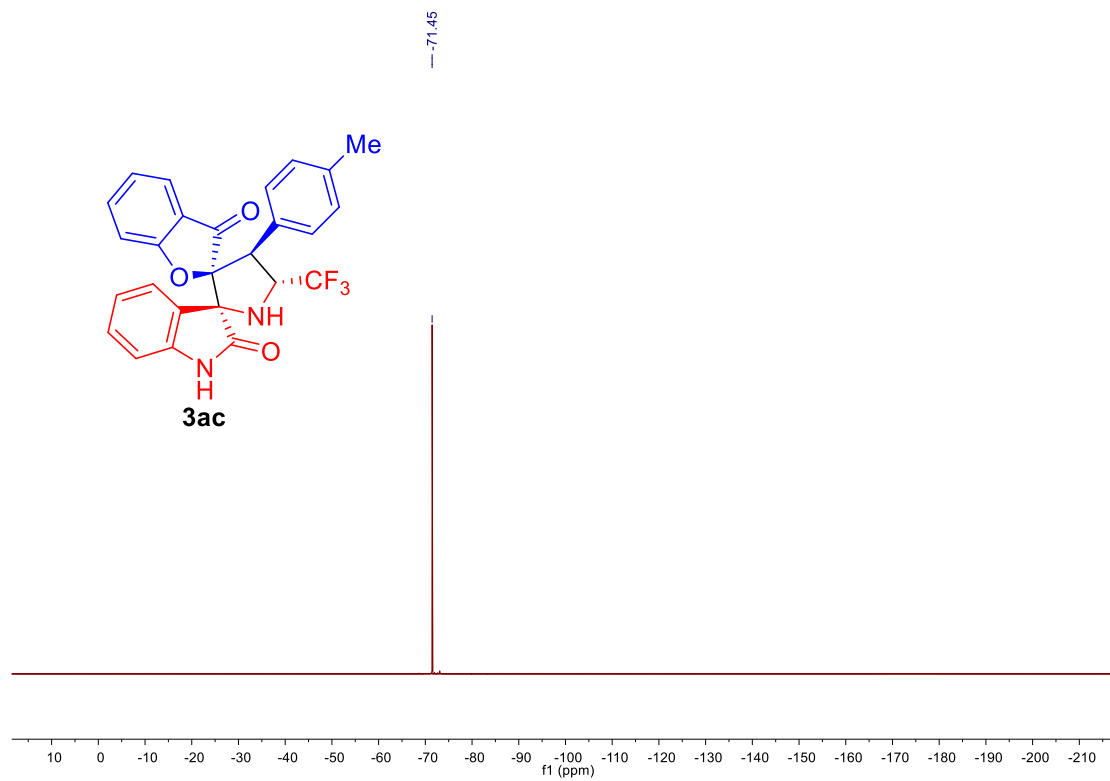
NMR Spectra of compounds

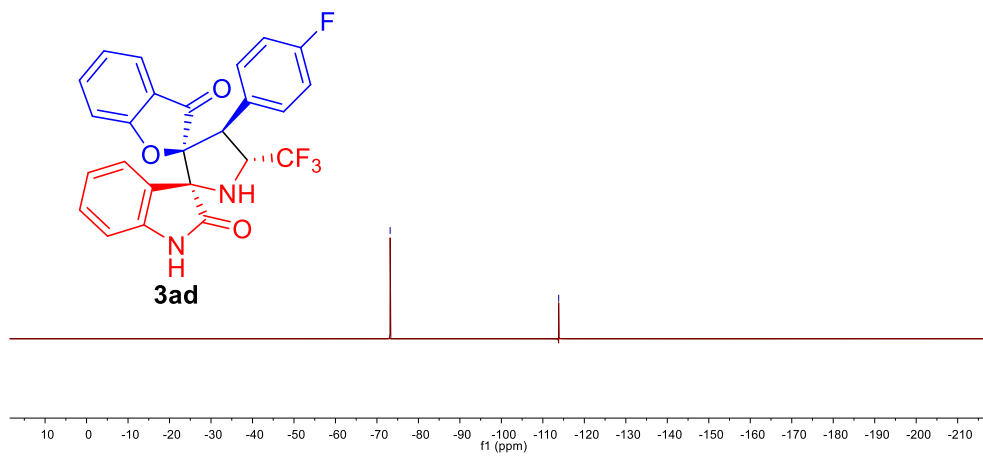
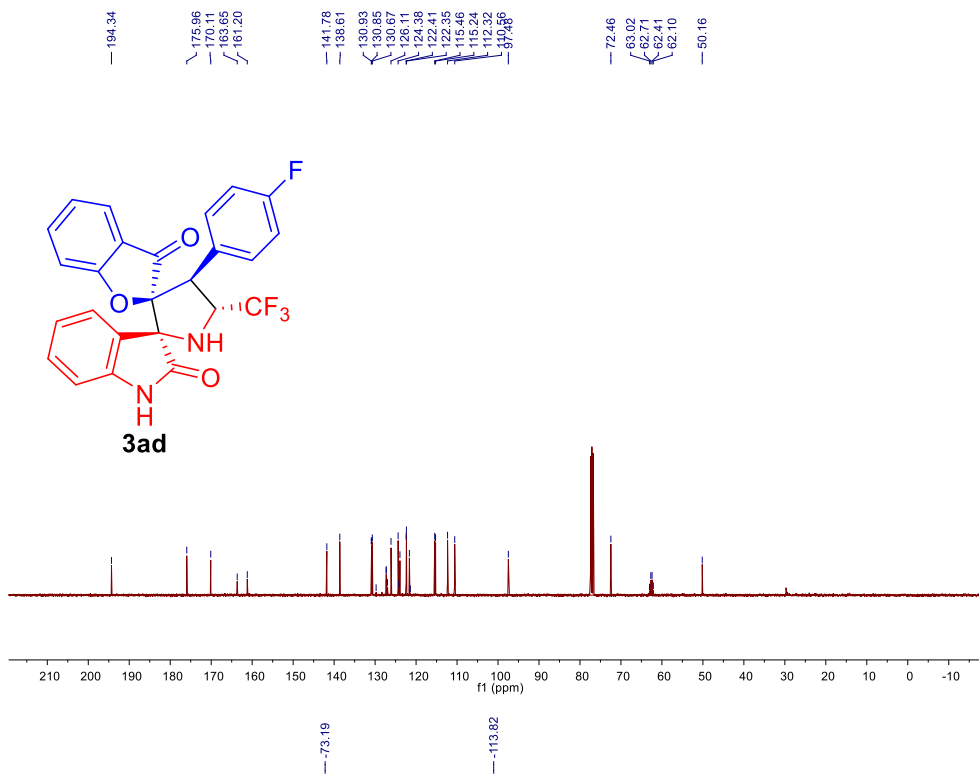


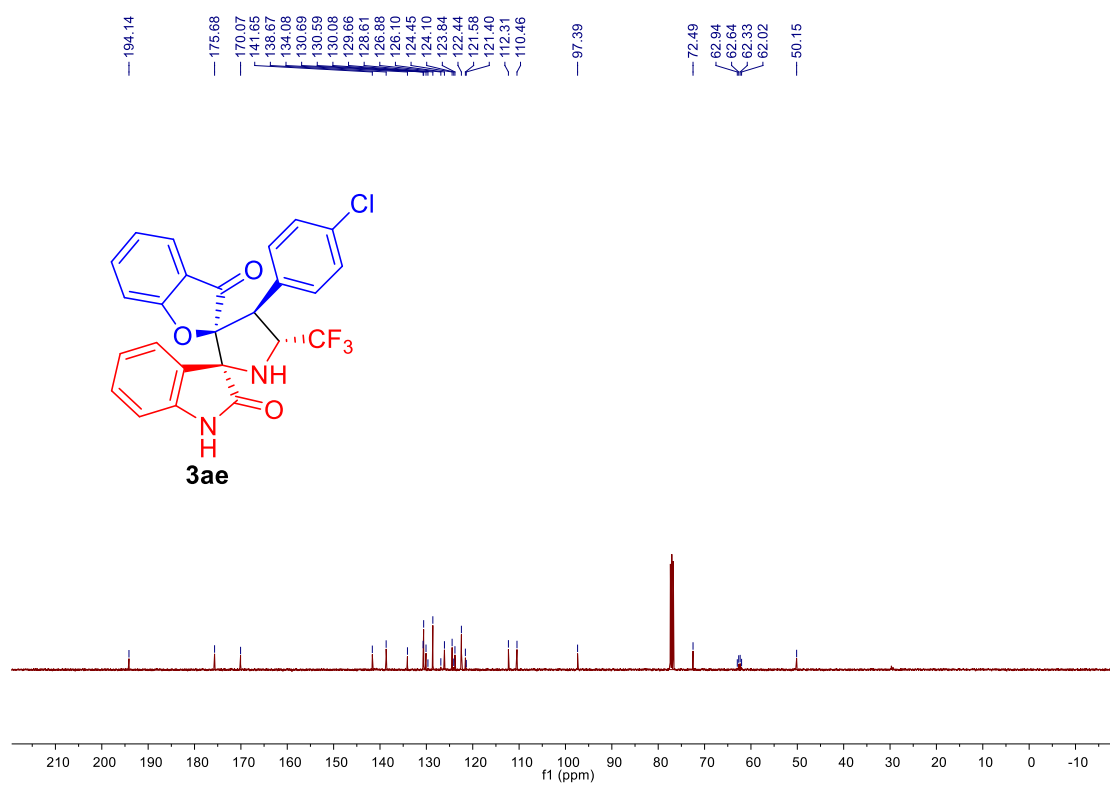
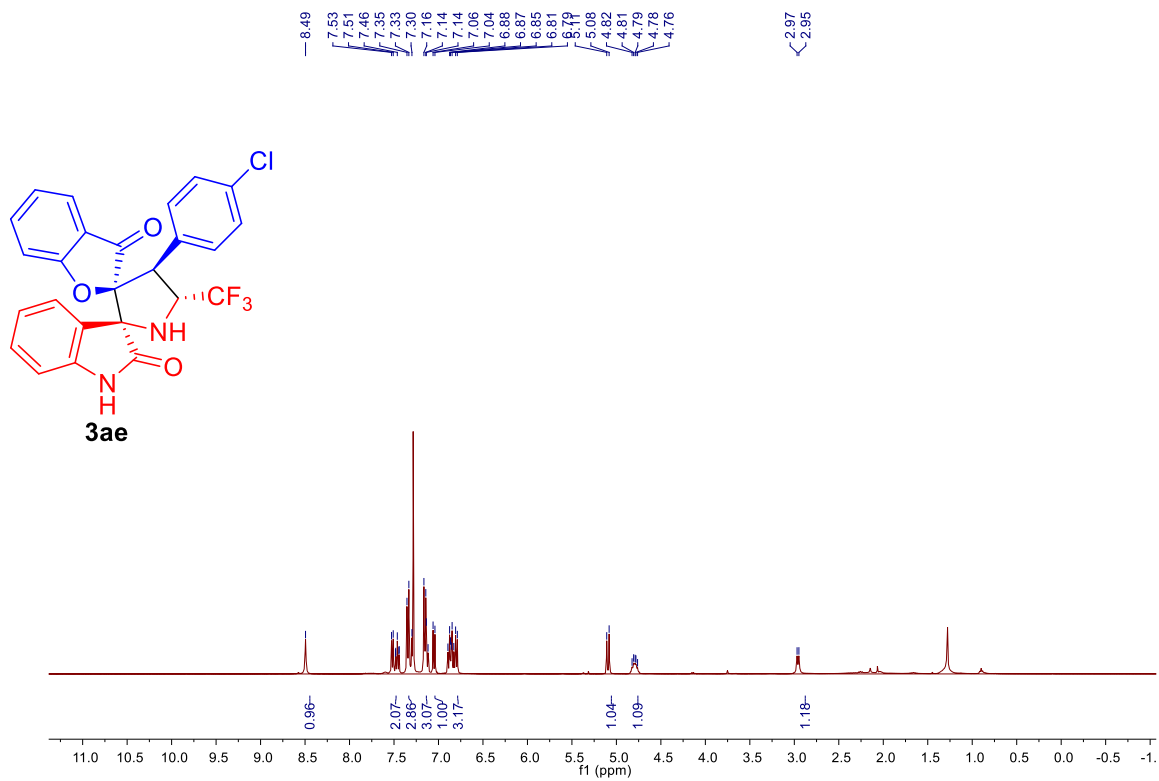


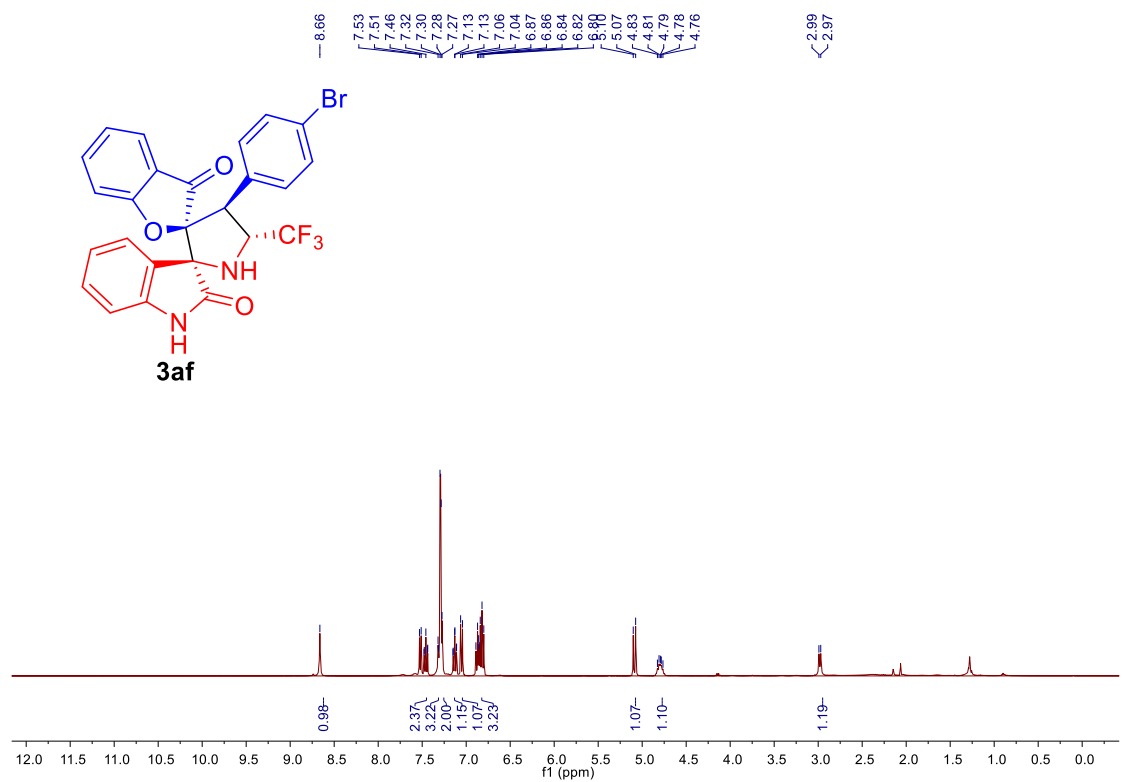
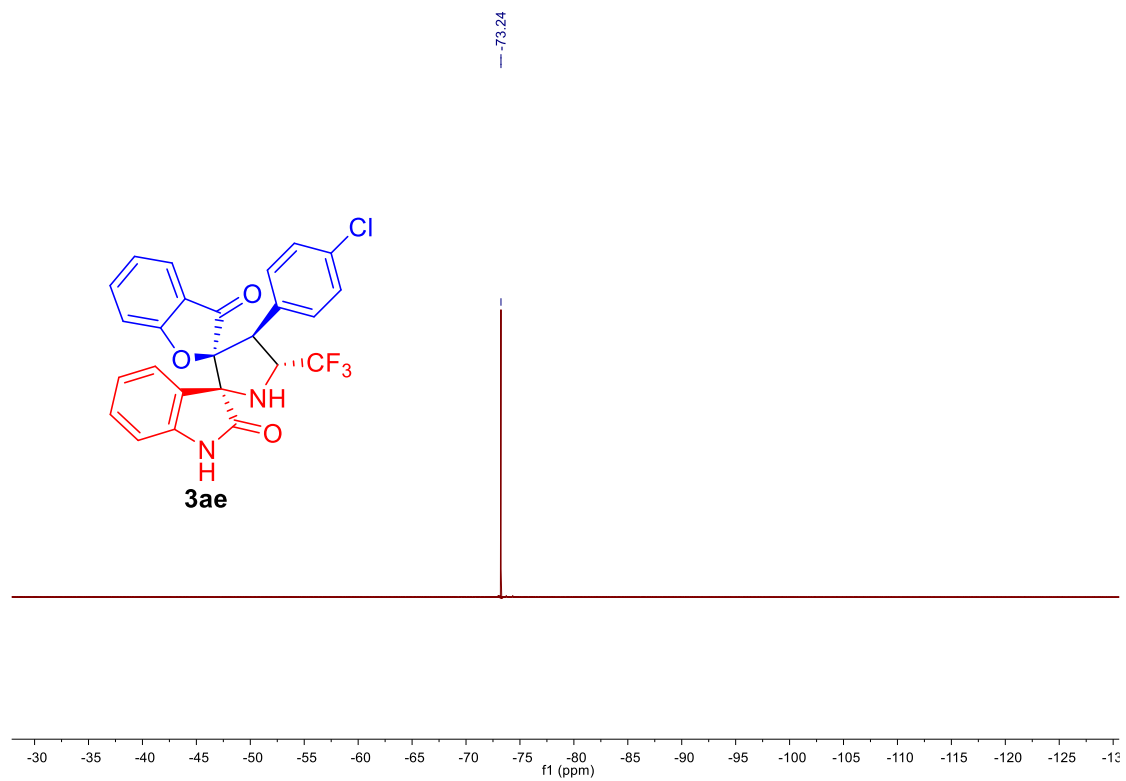


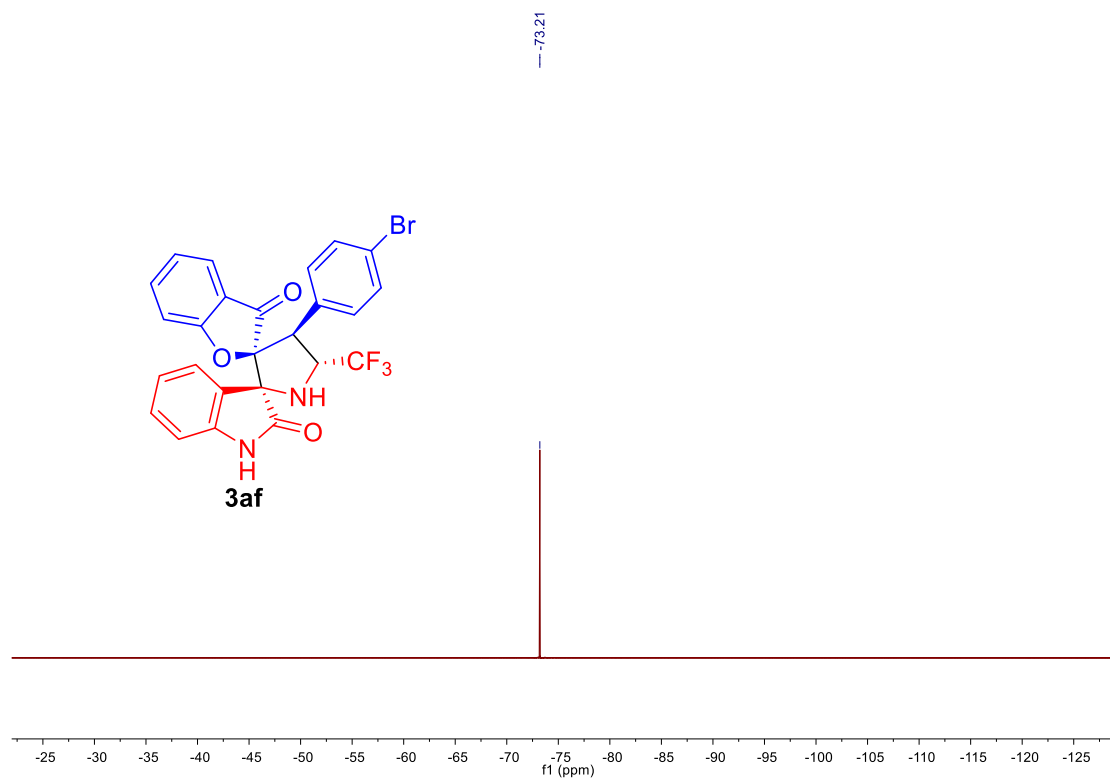
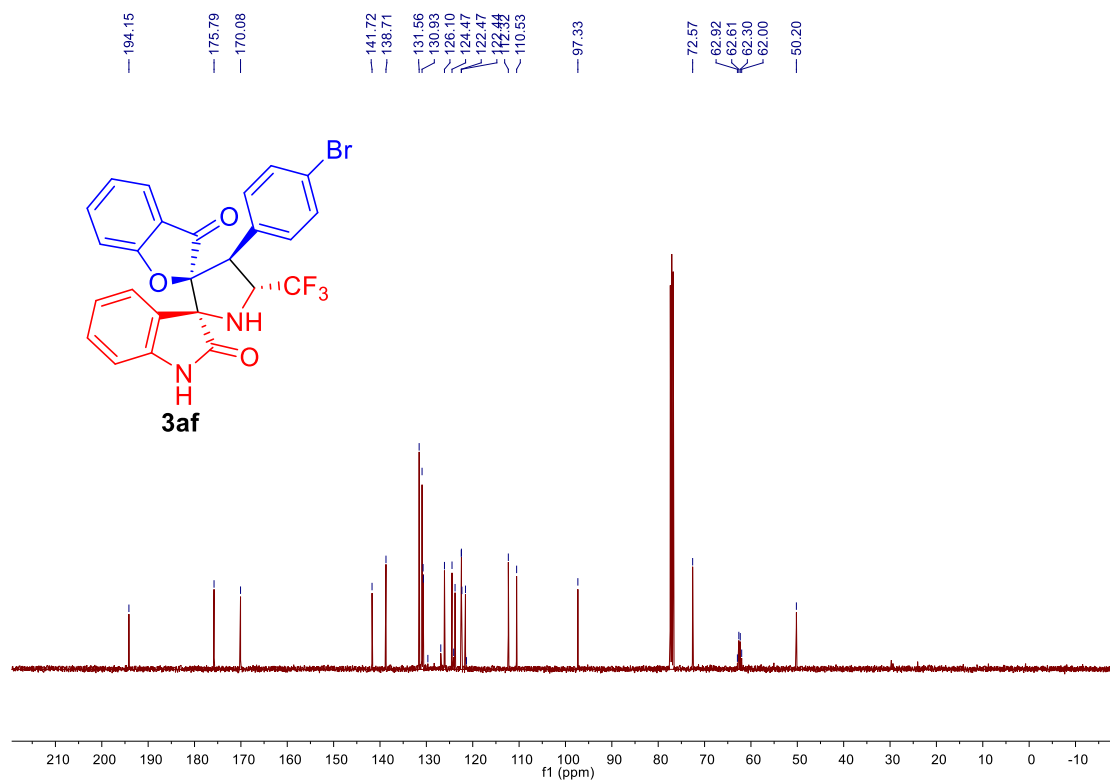


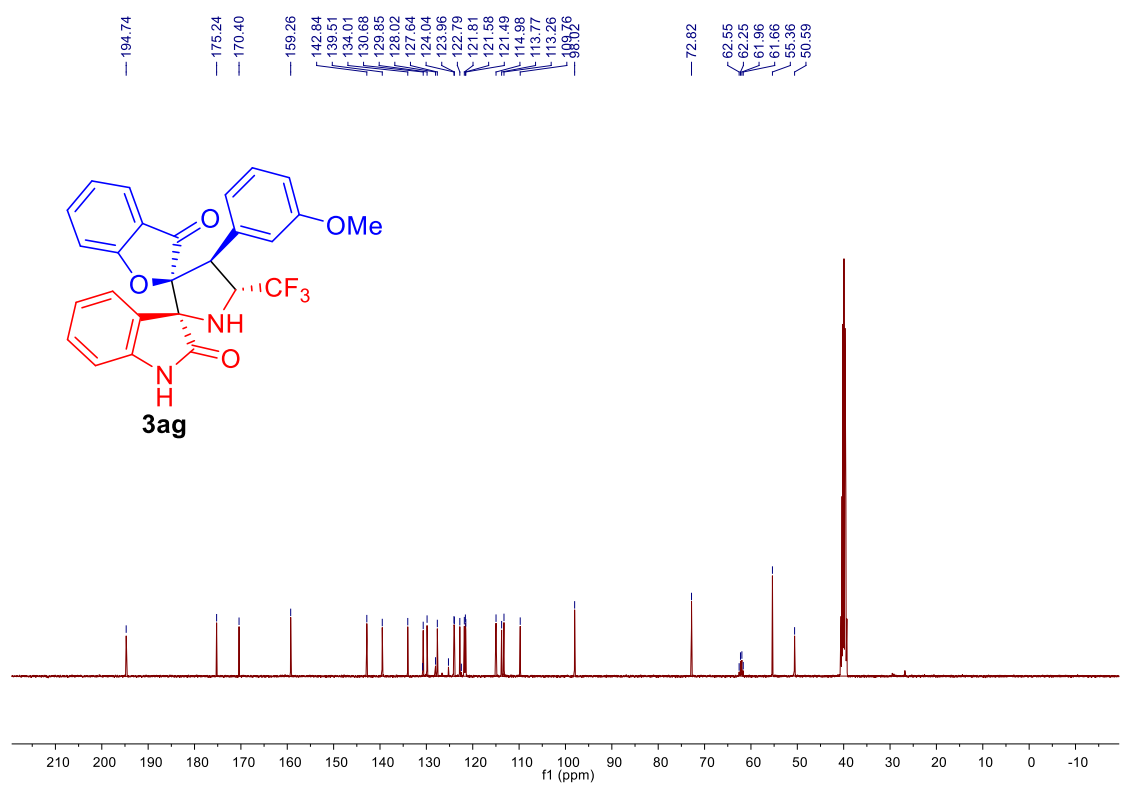
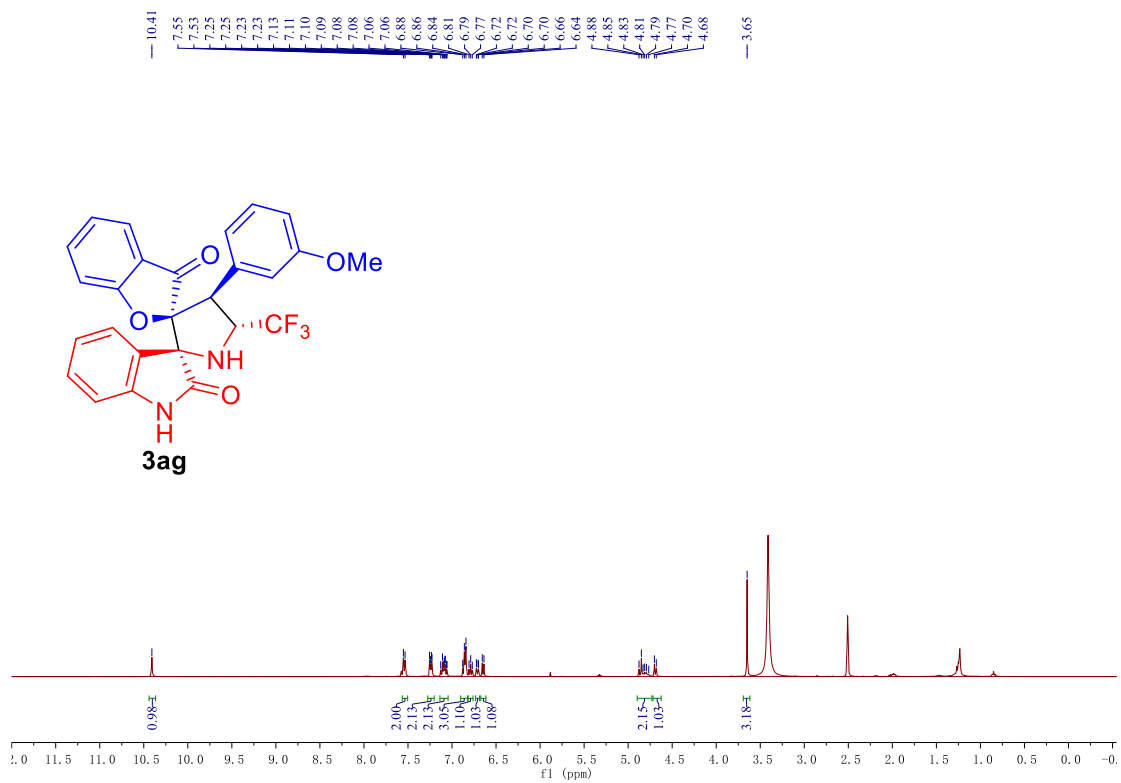


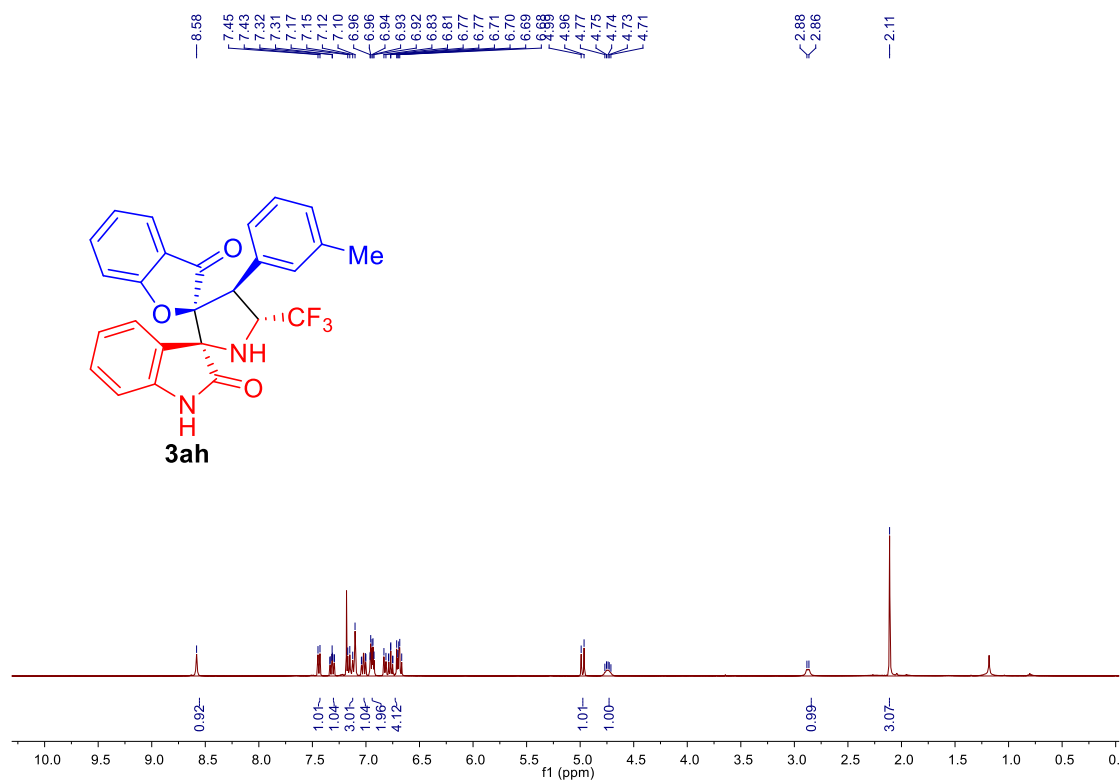
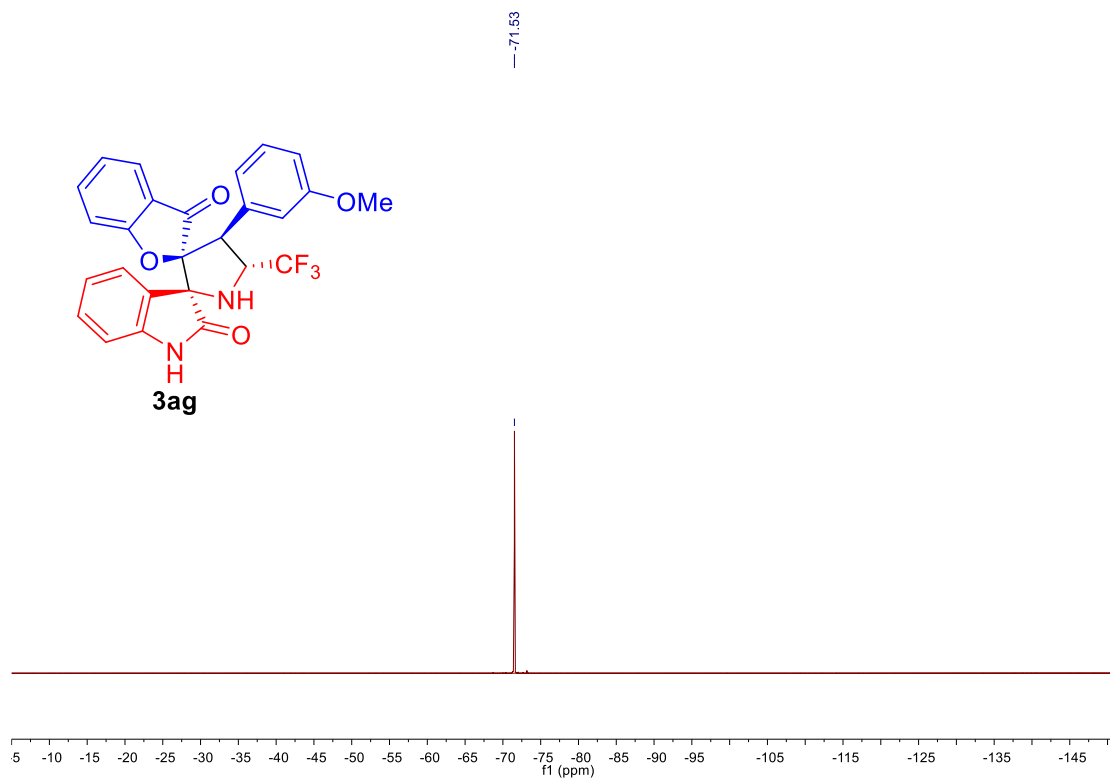


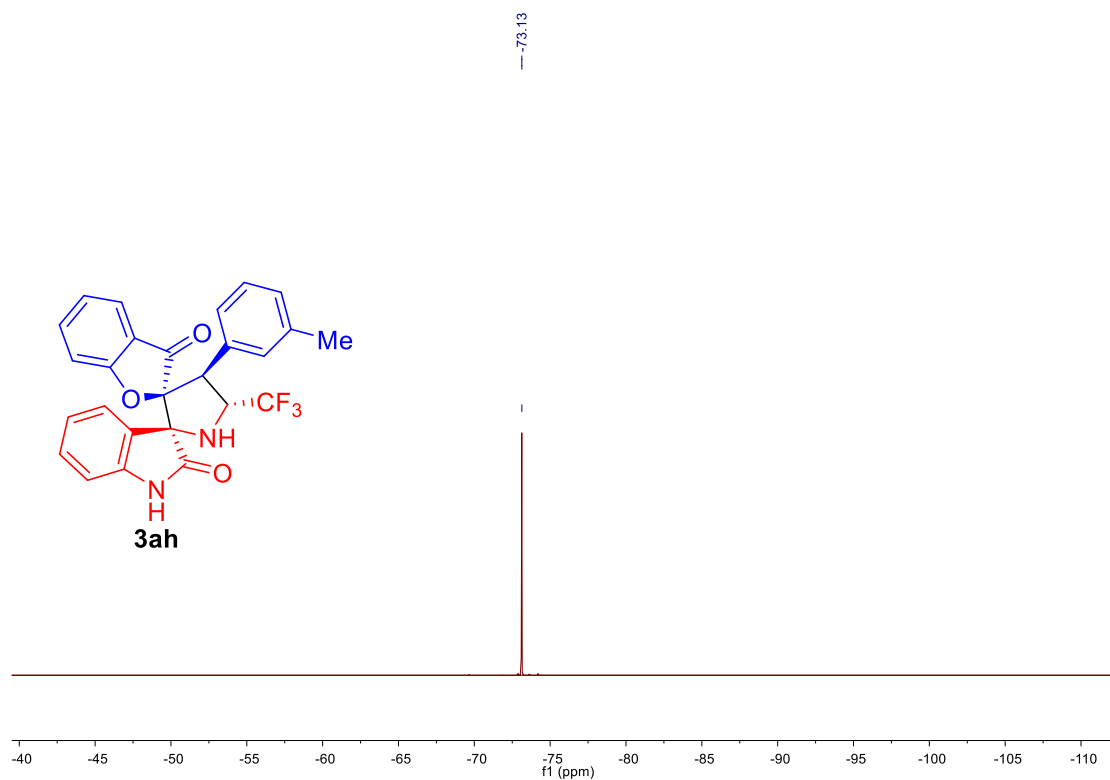
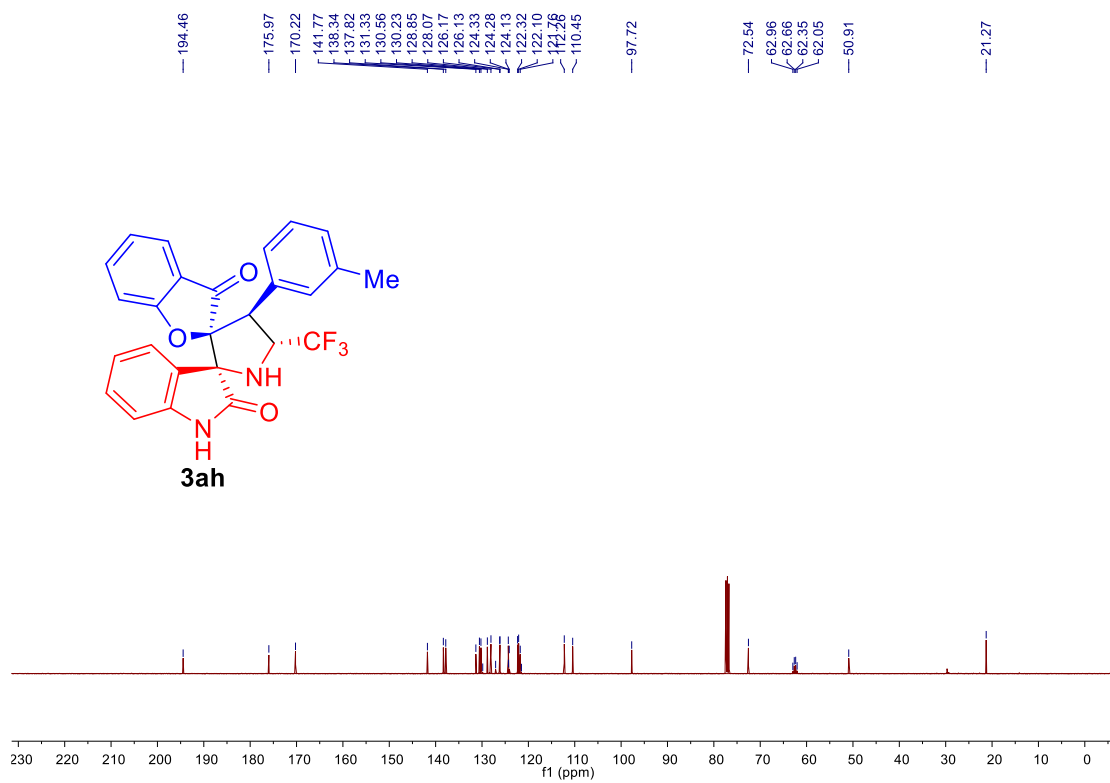


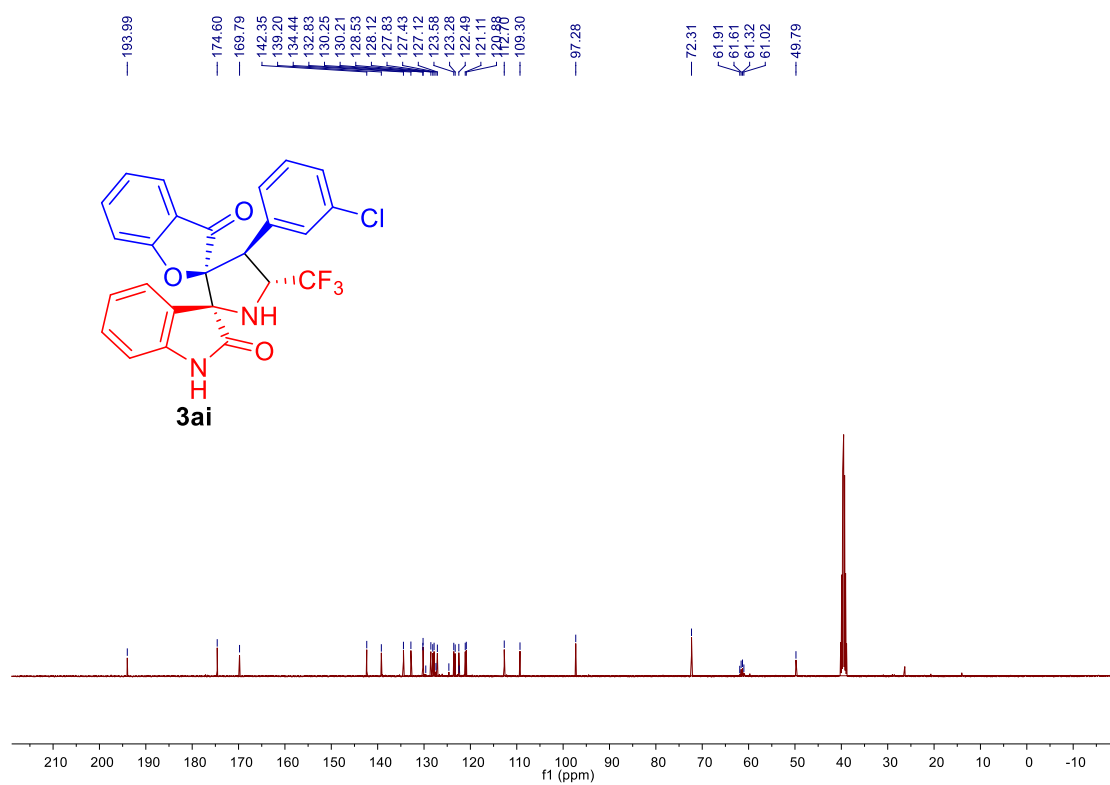
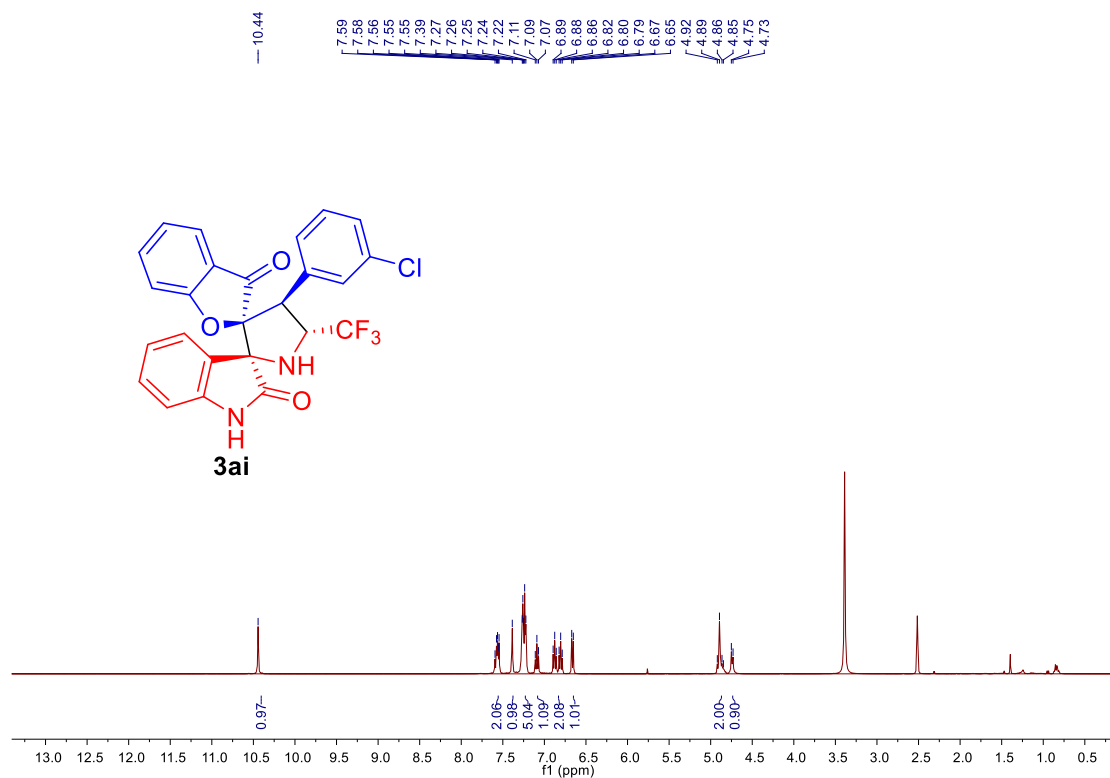


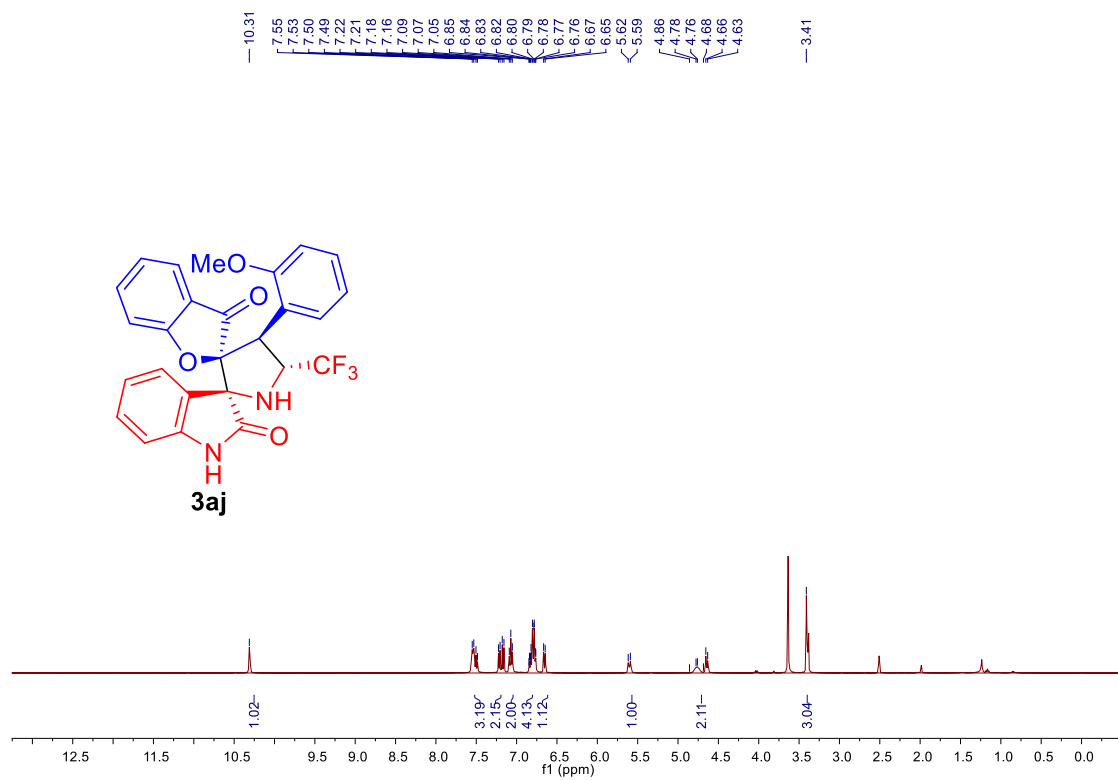
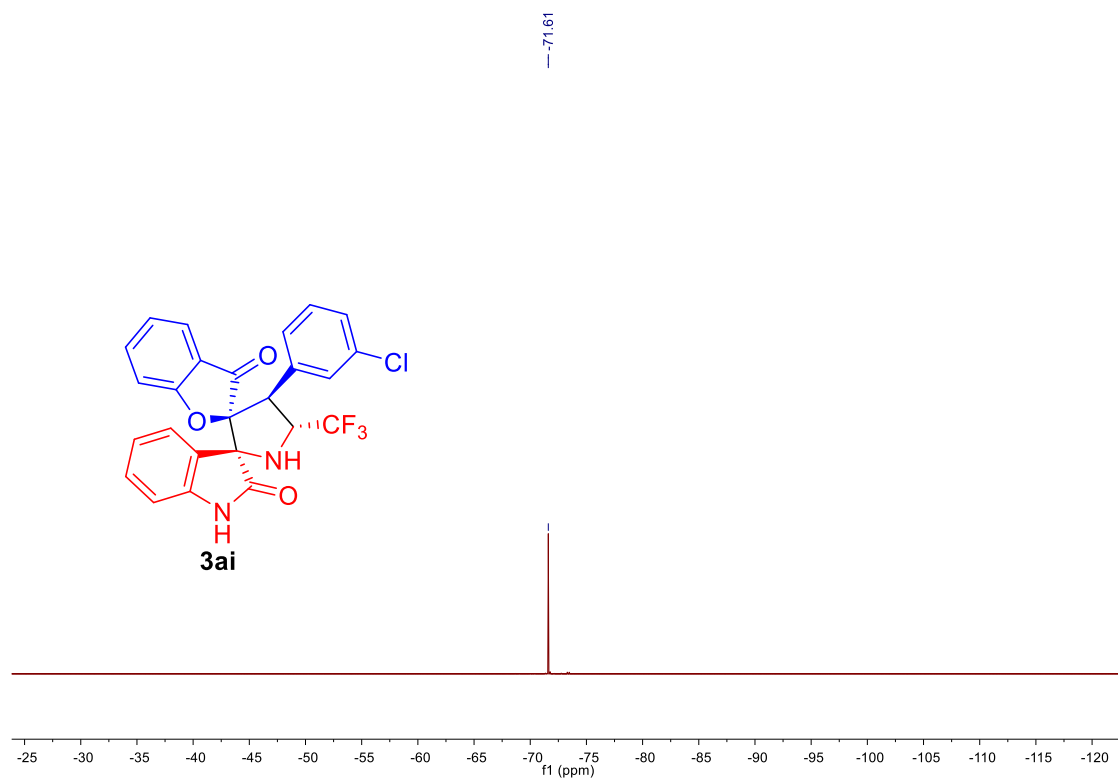


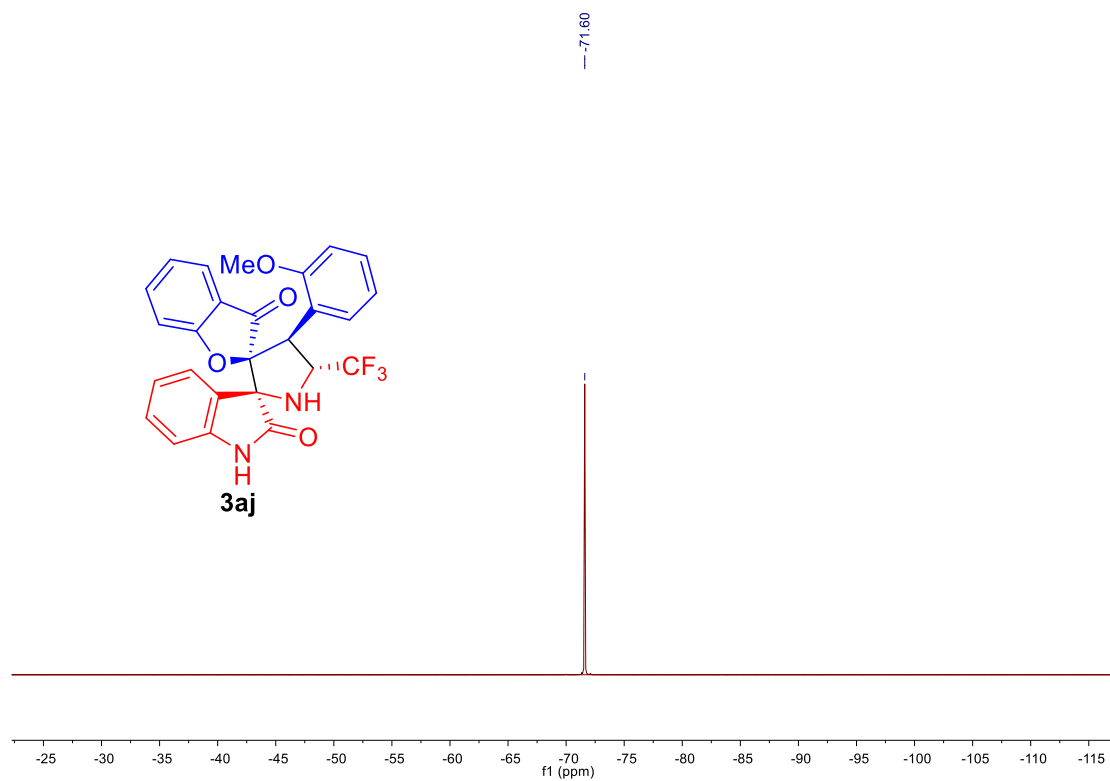
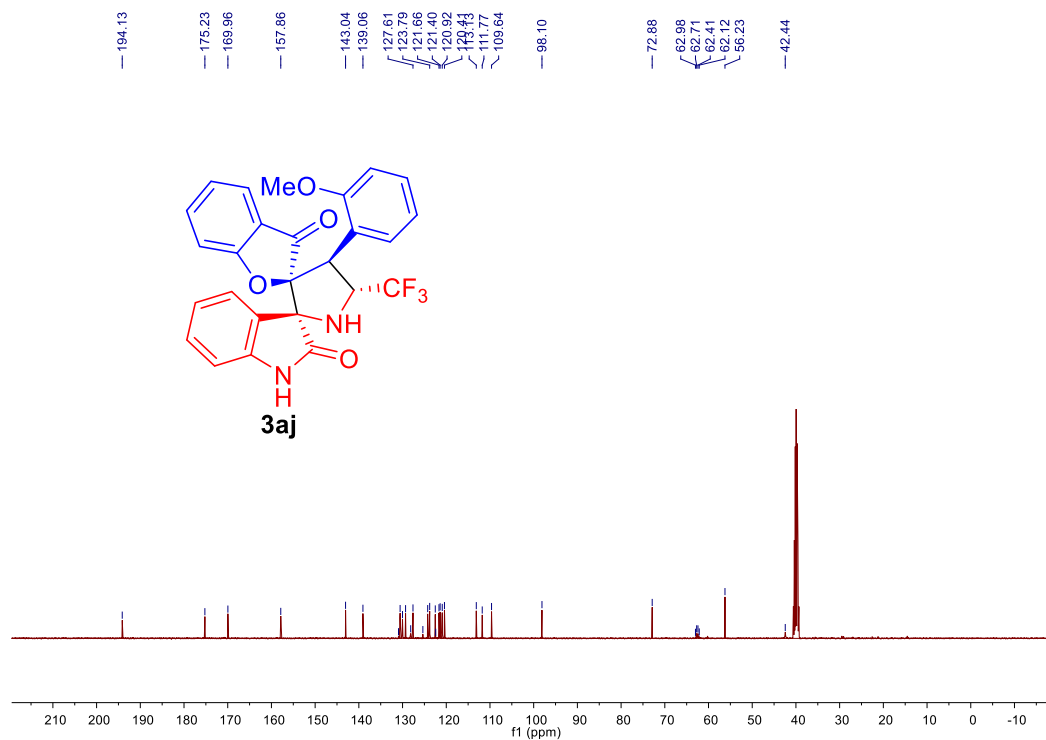


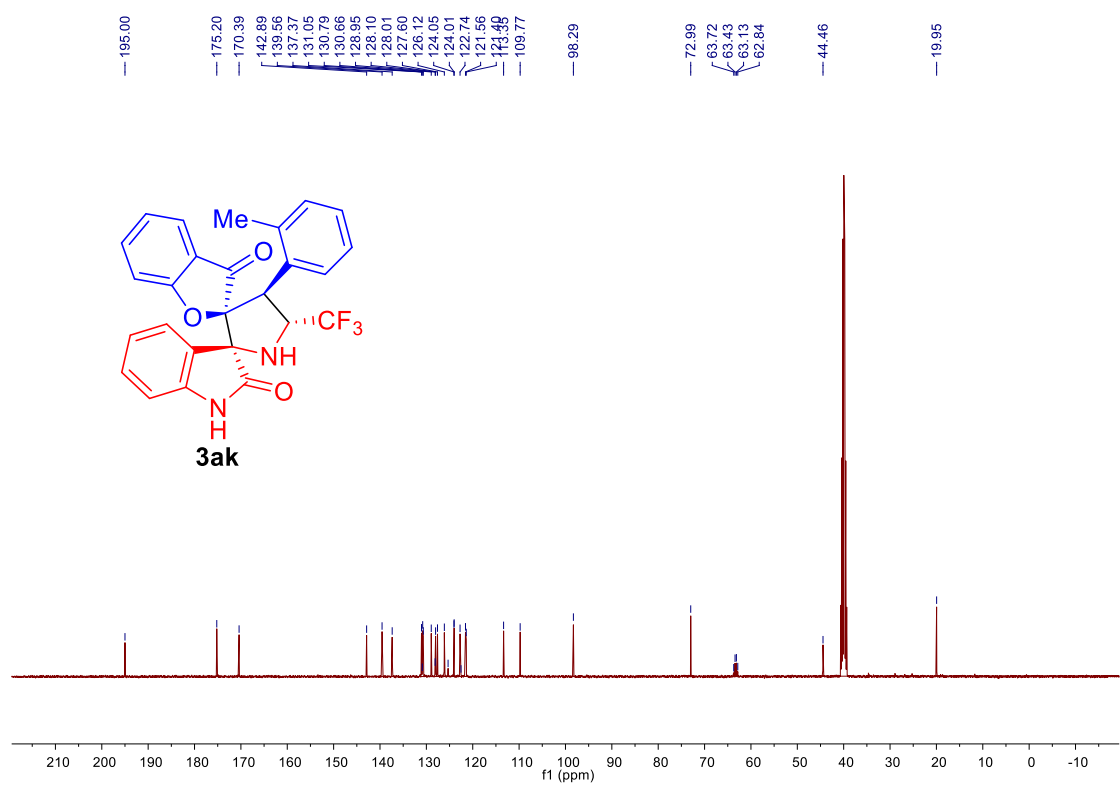
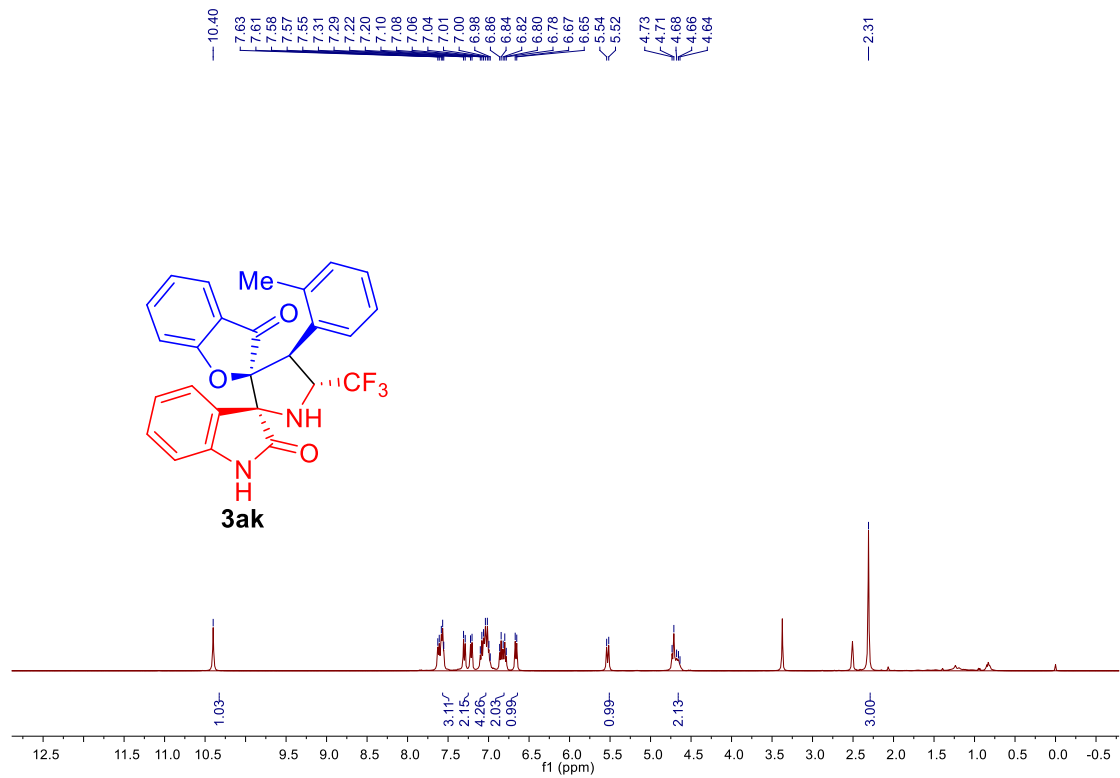


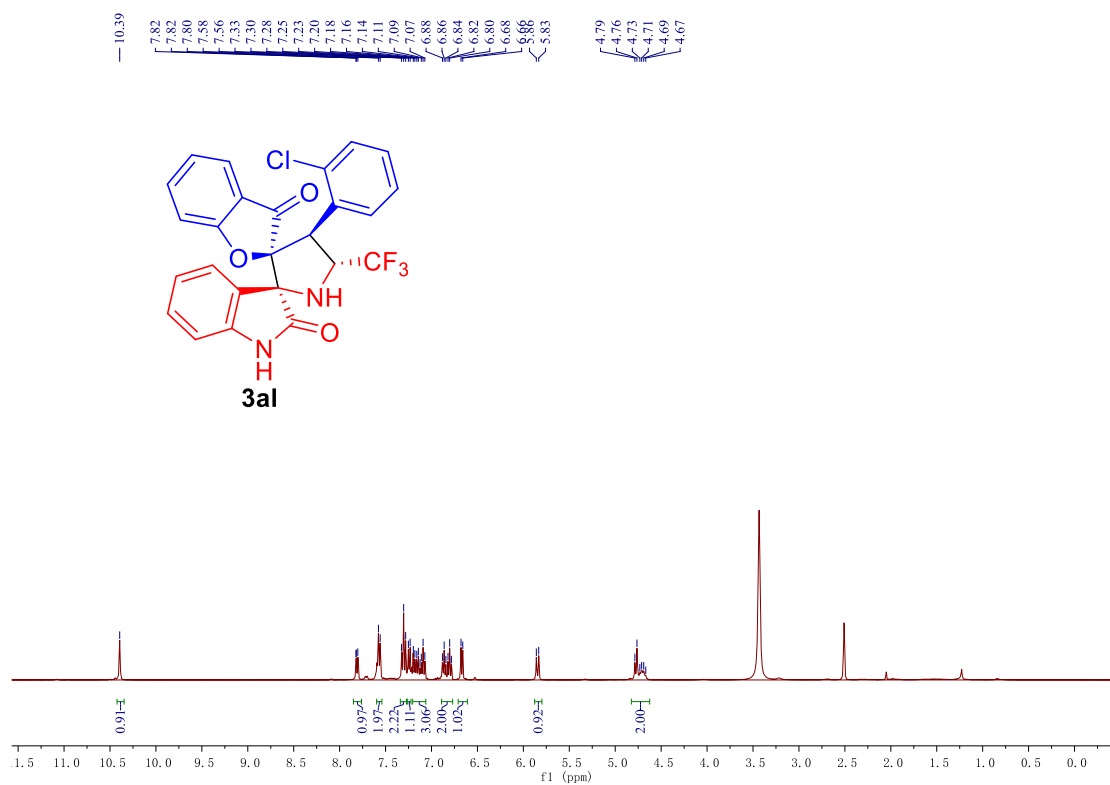
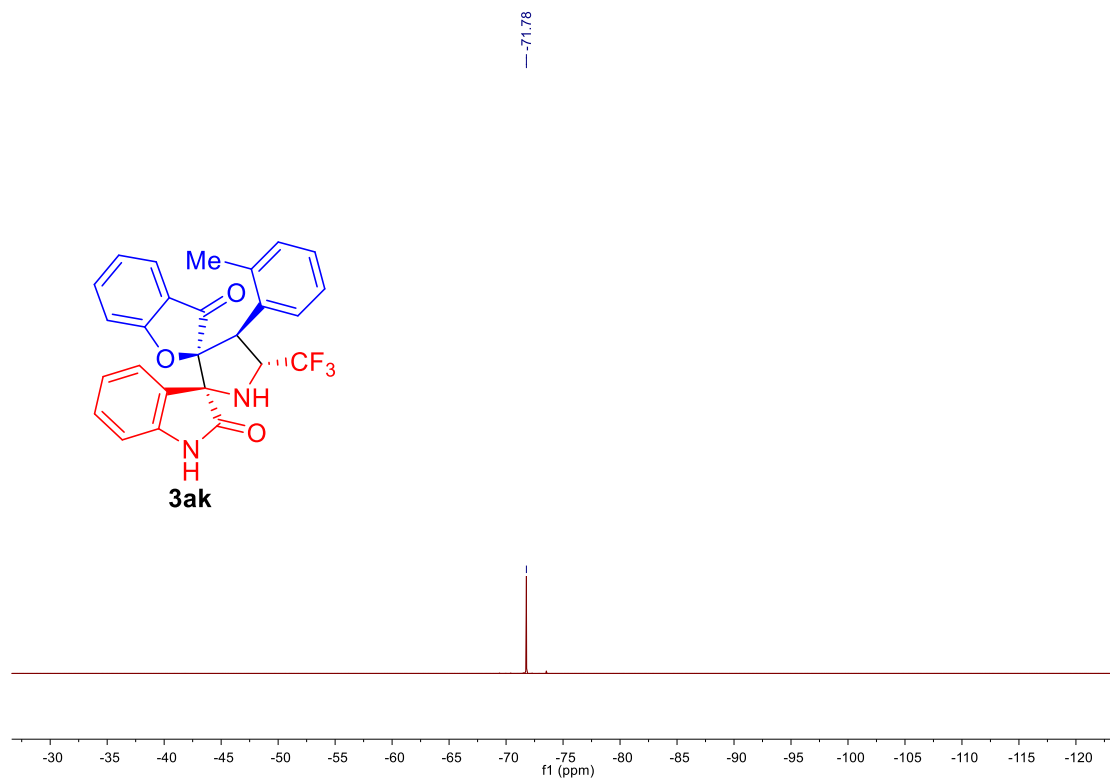


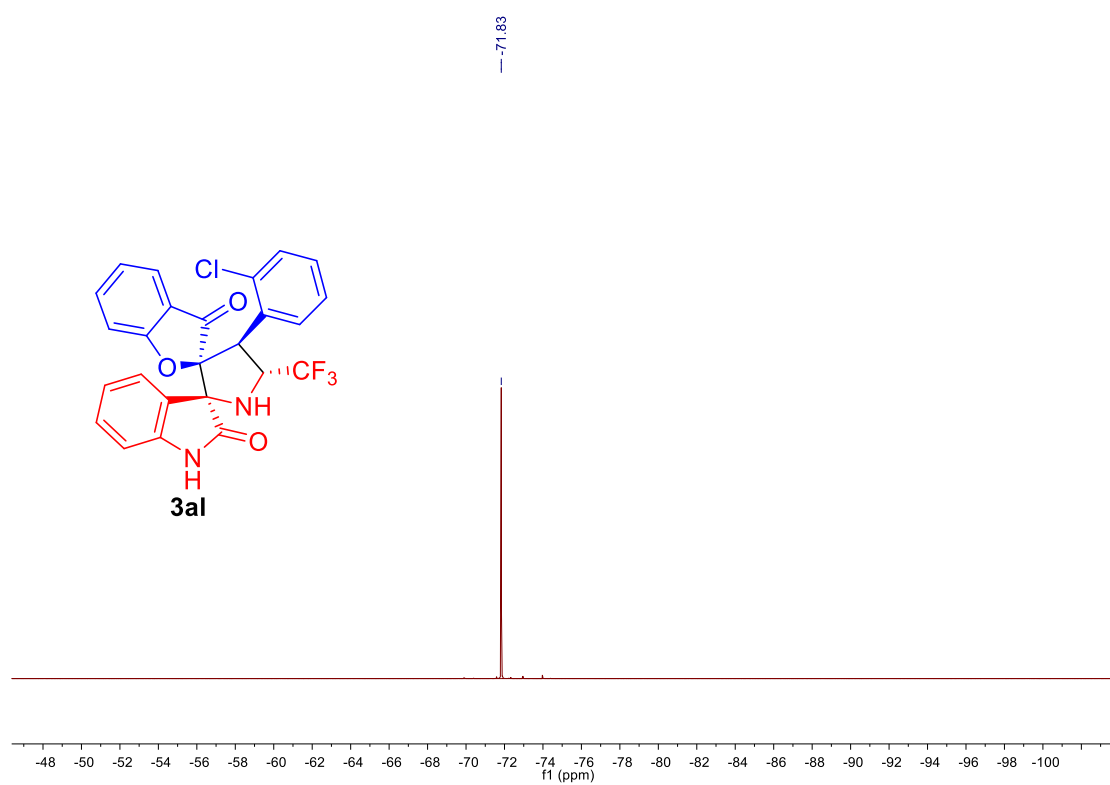
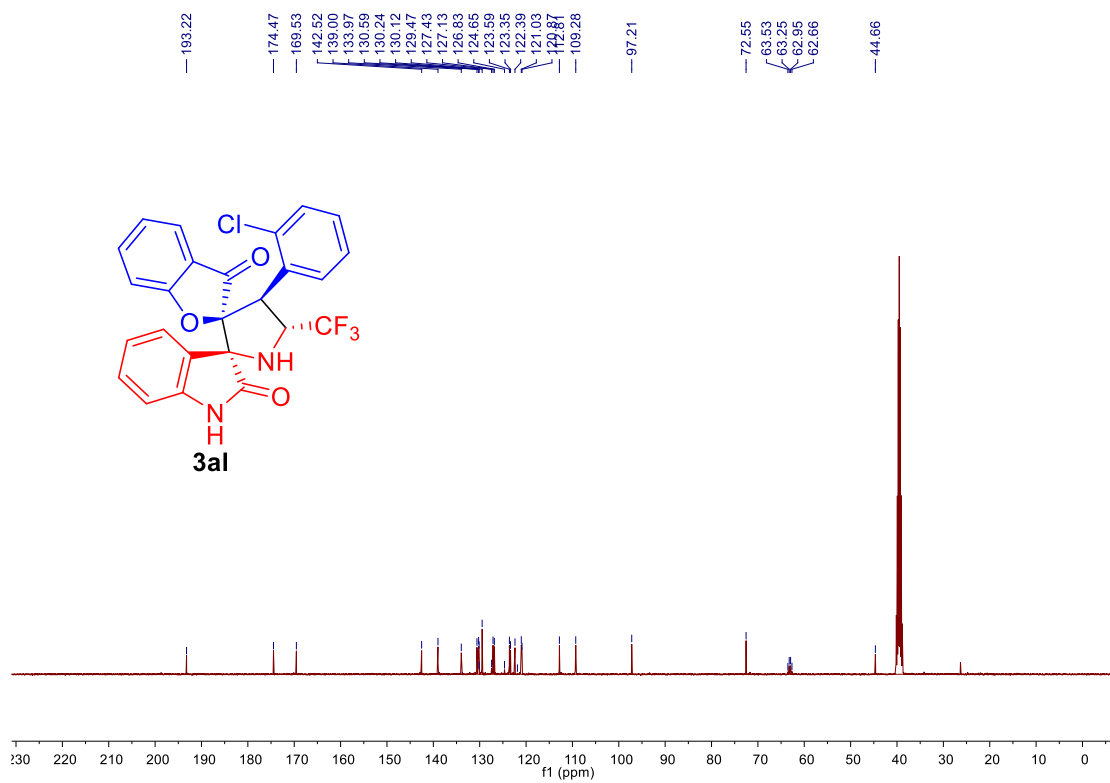


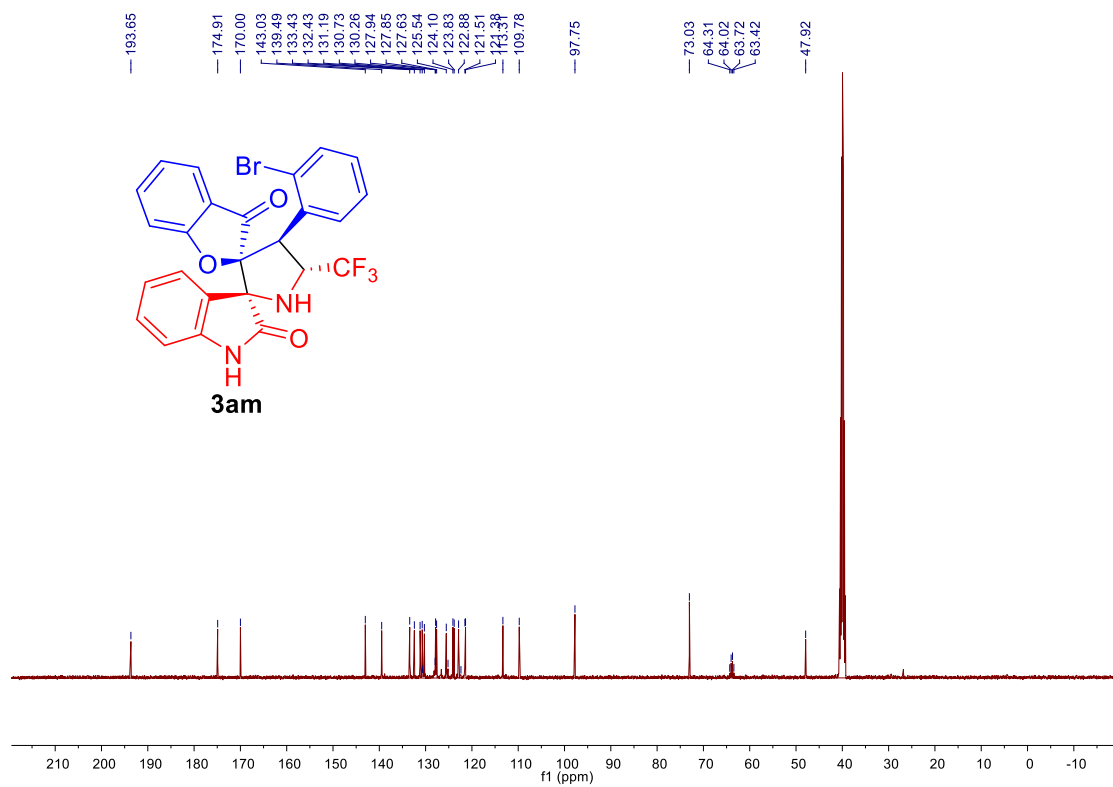
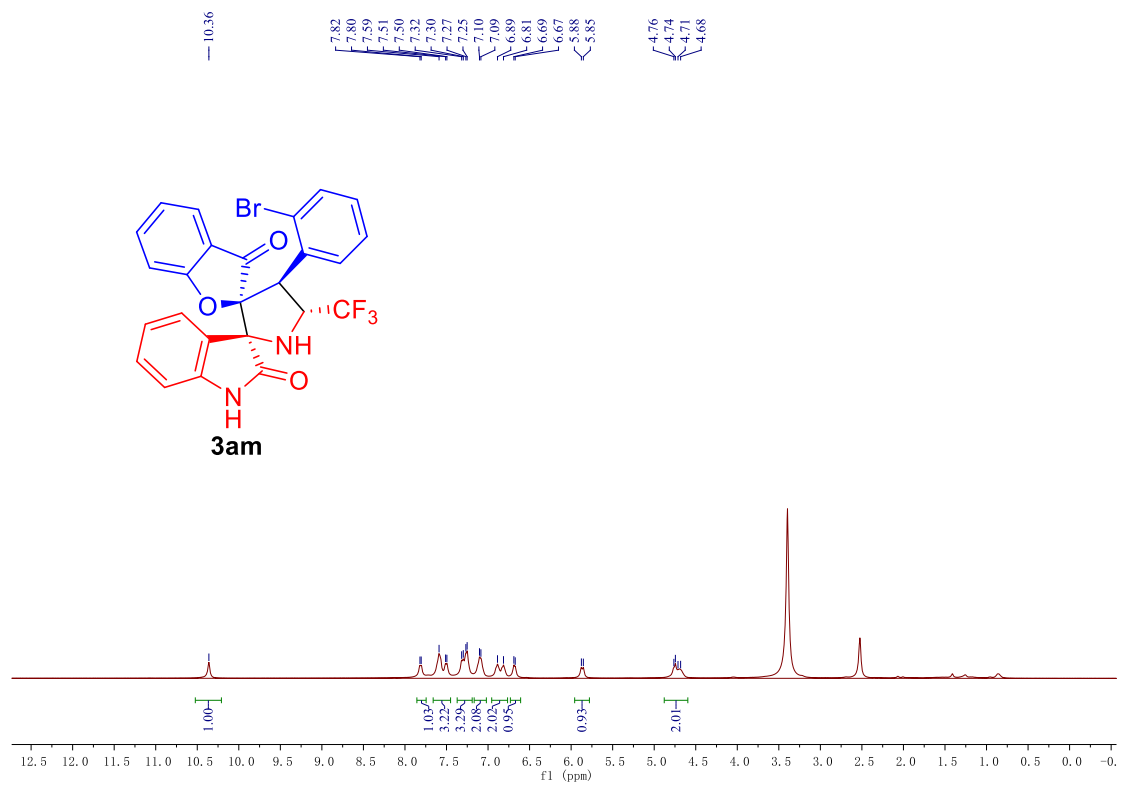


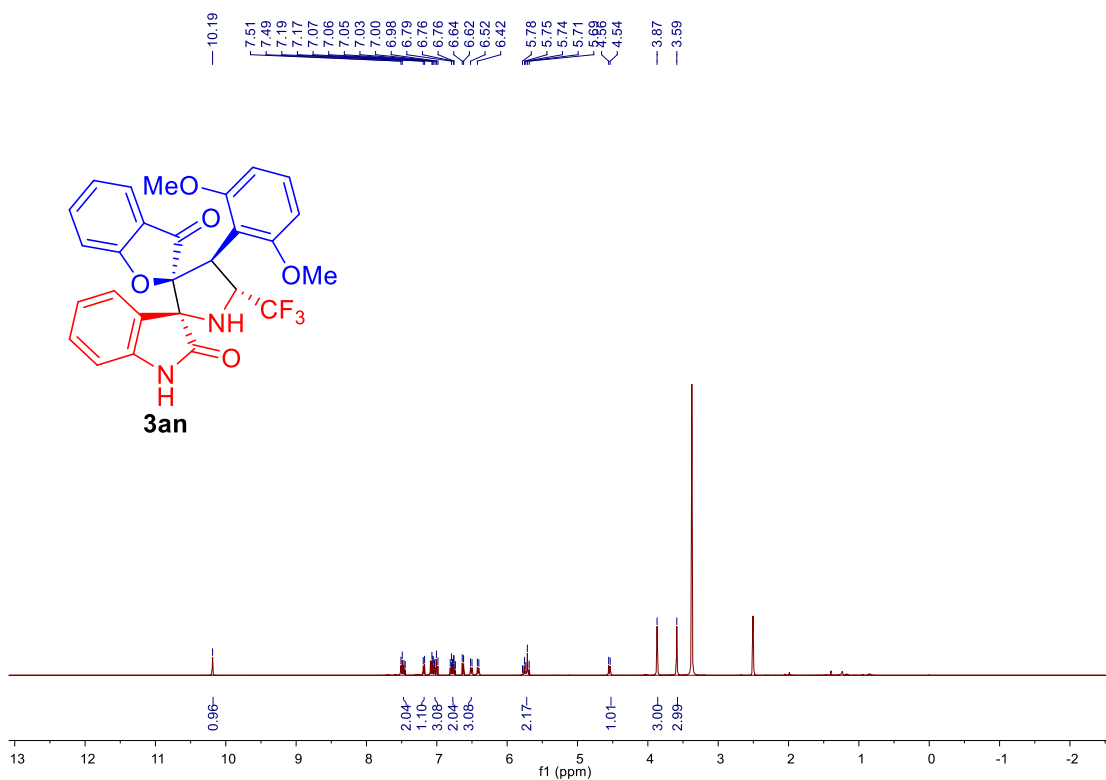
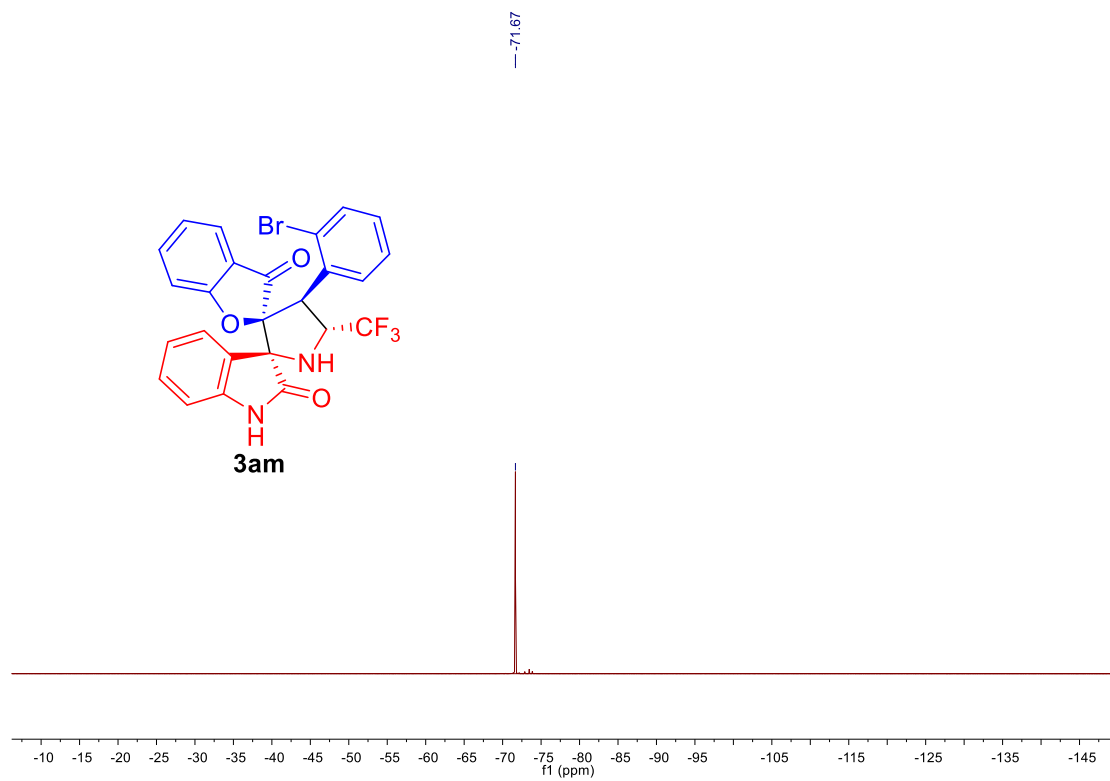


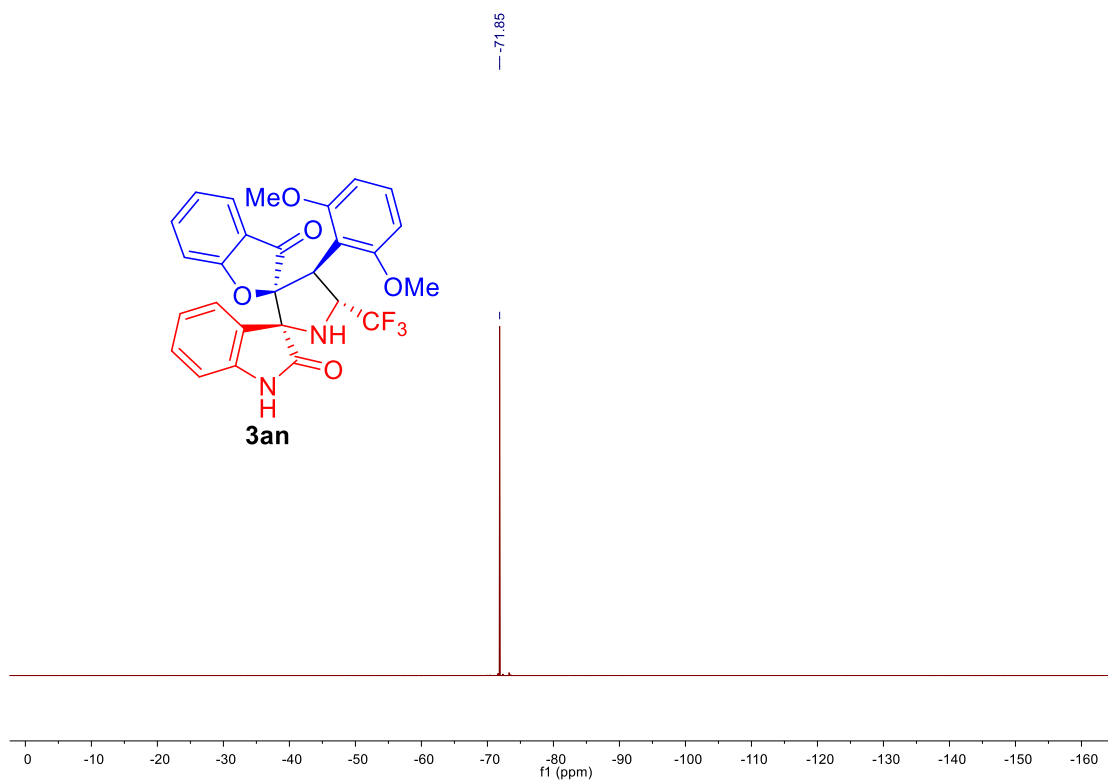
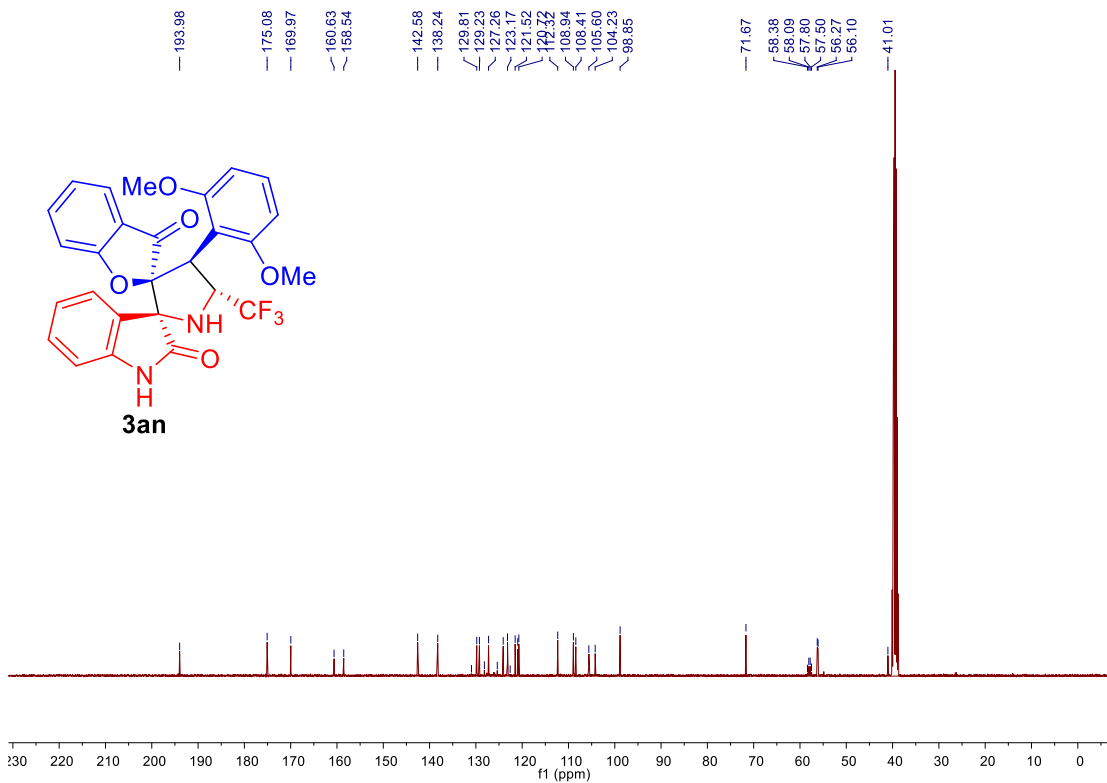


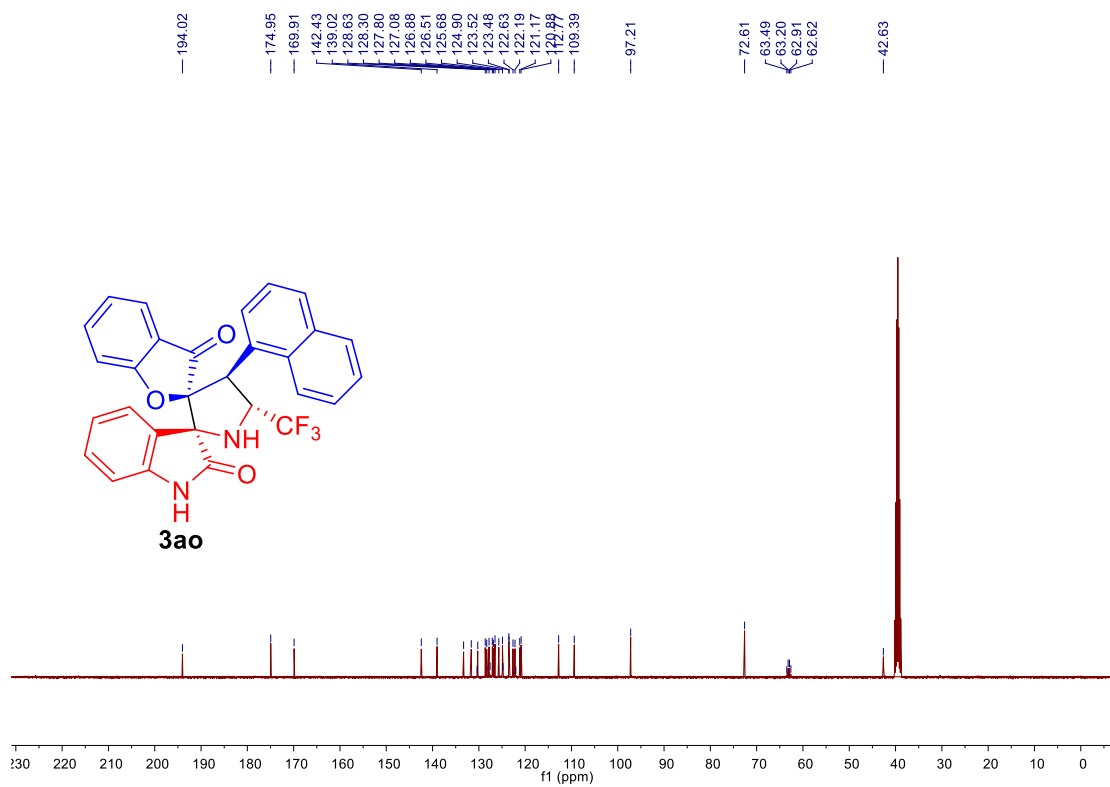
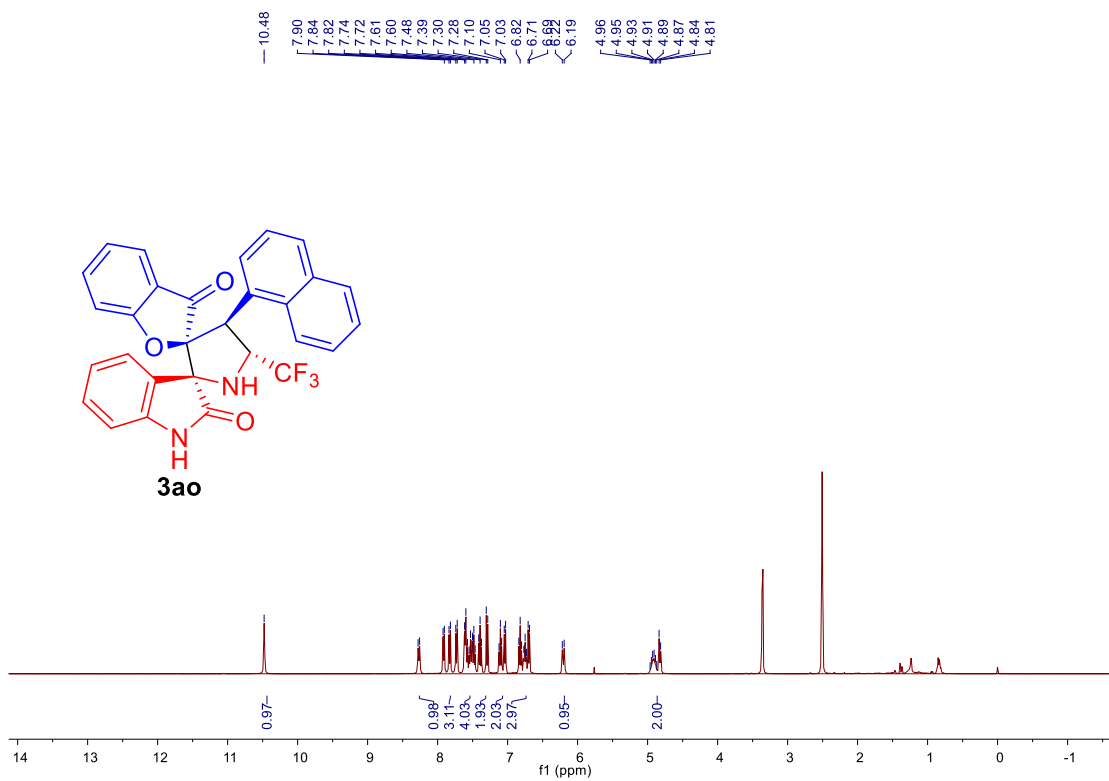


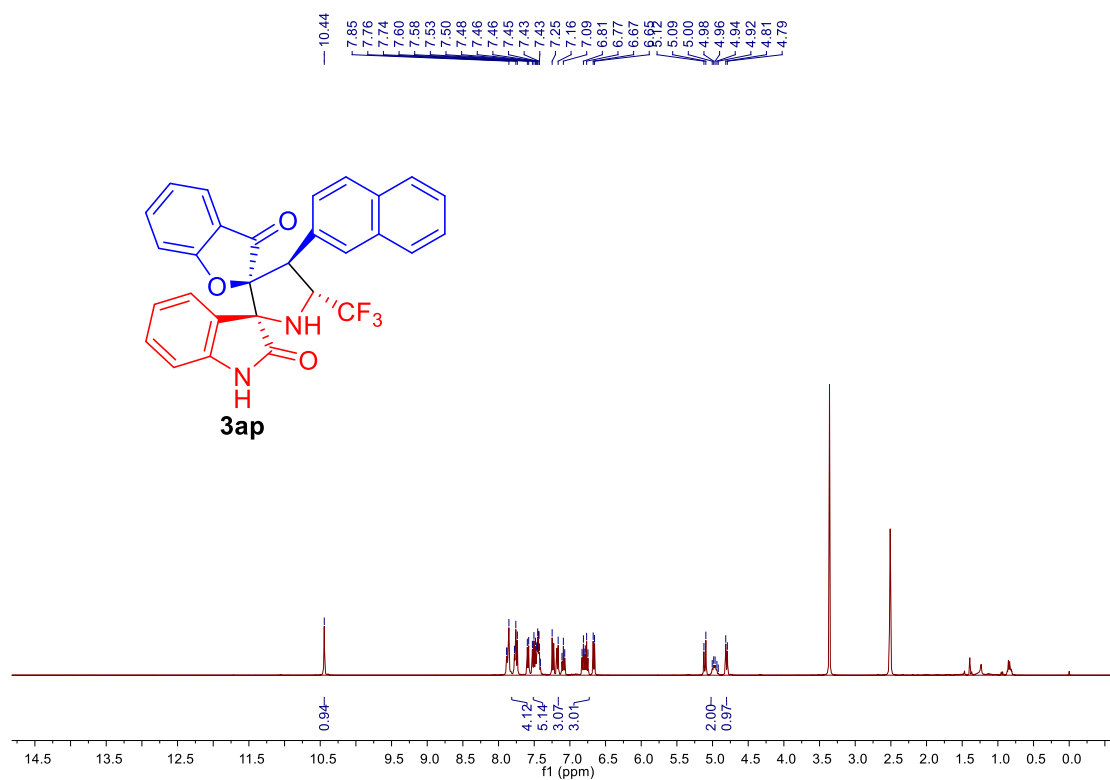
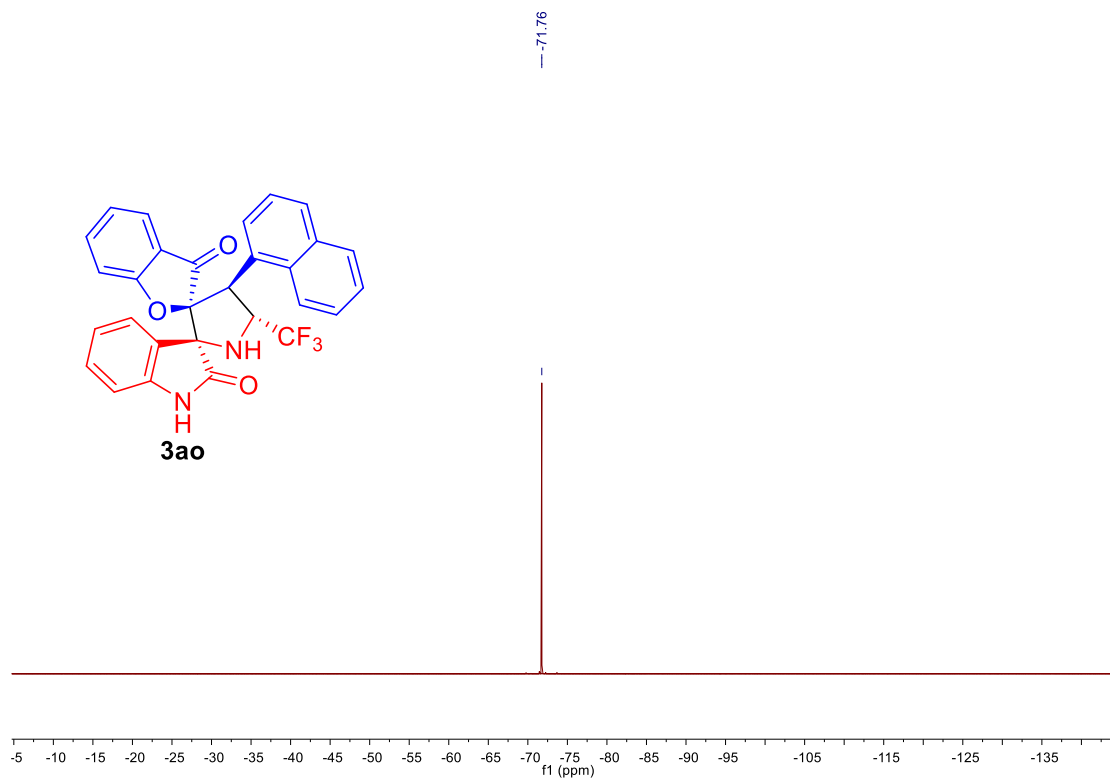


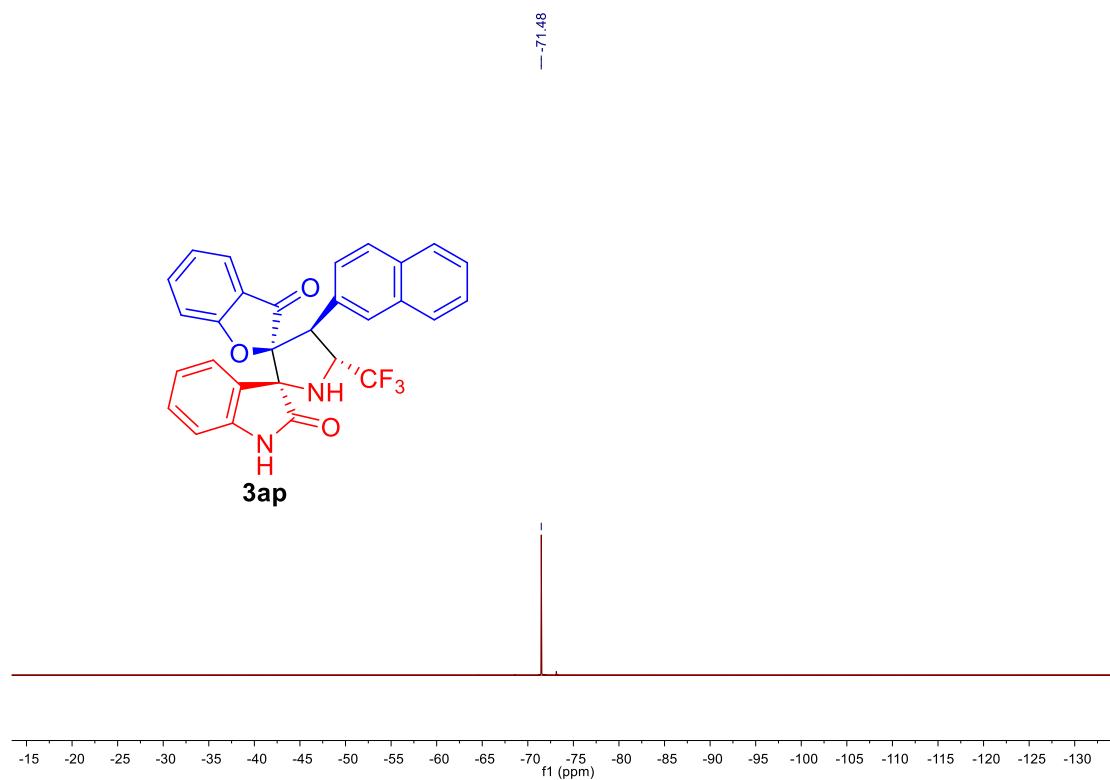
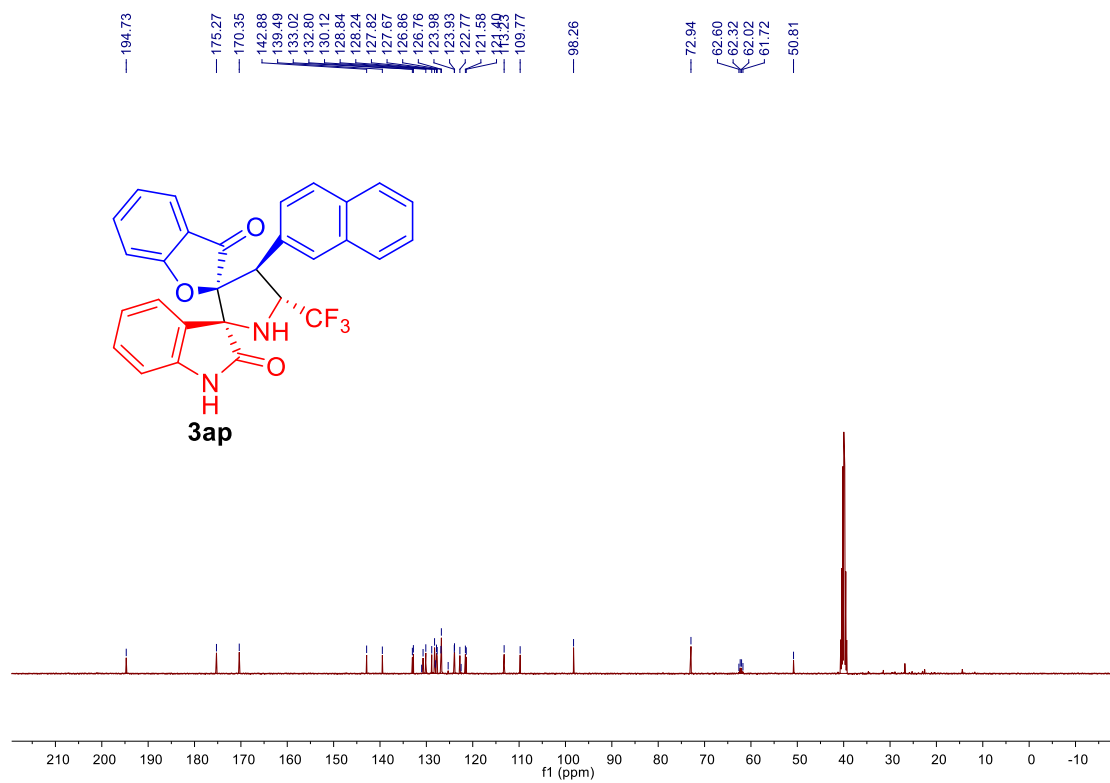


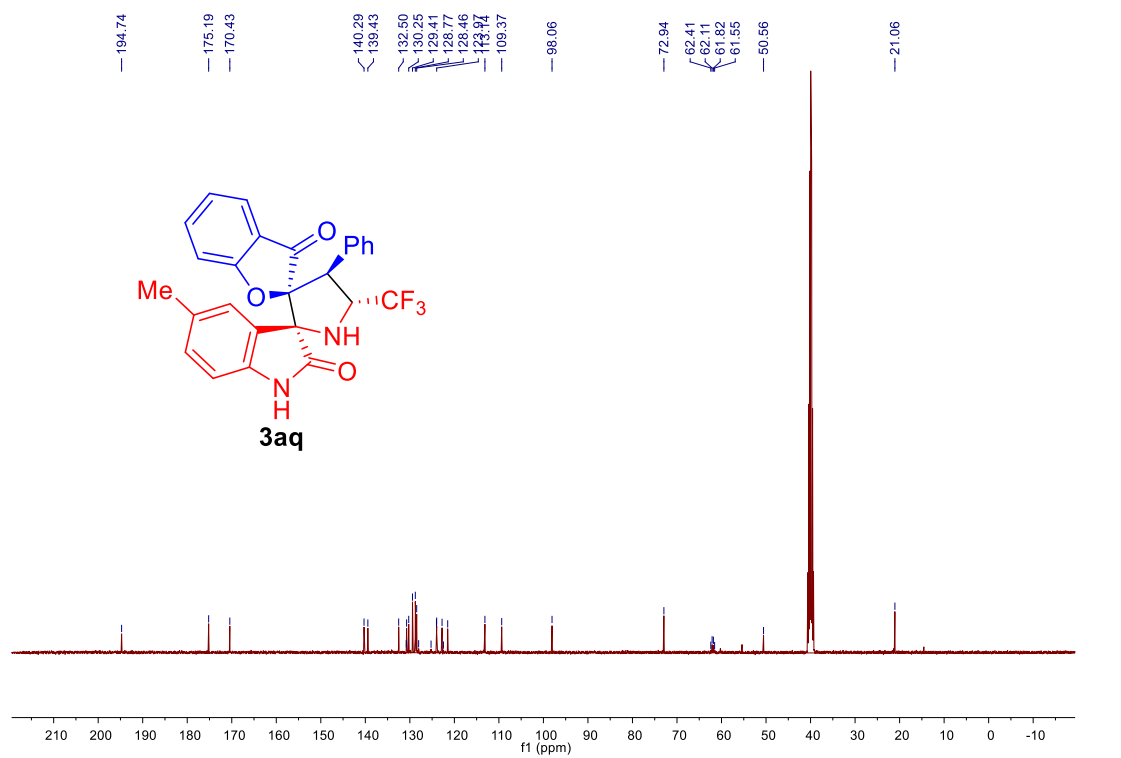
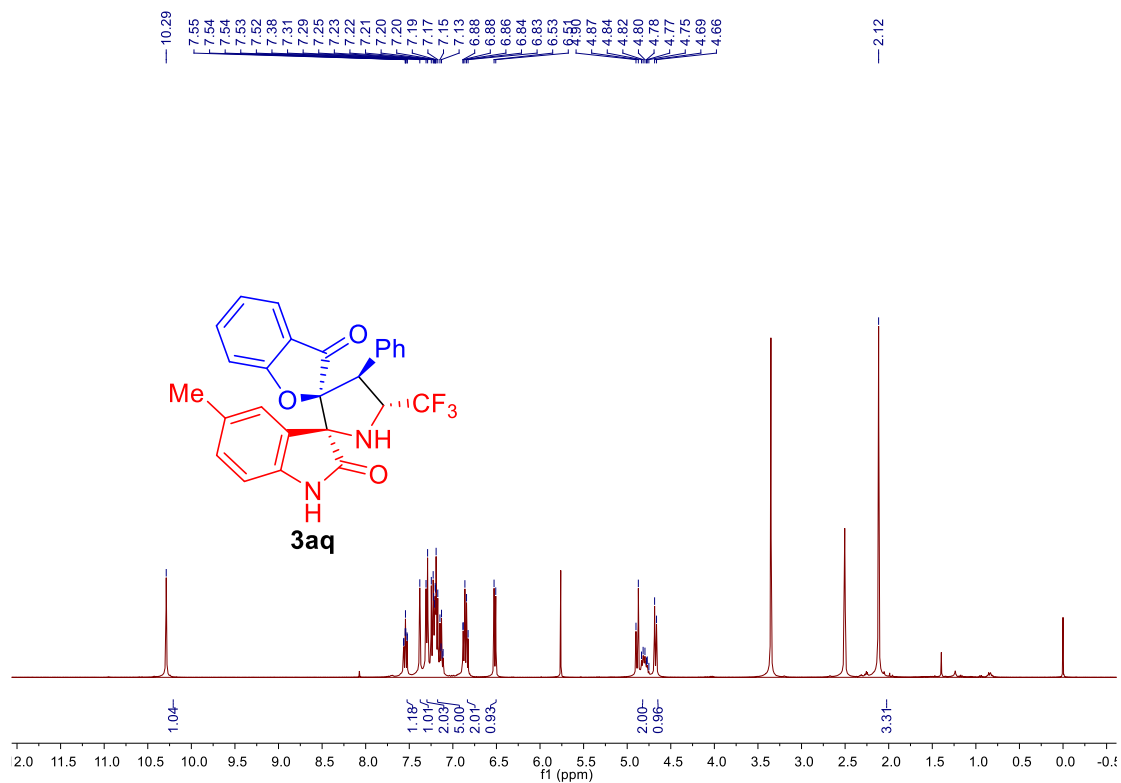


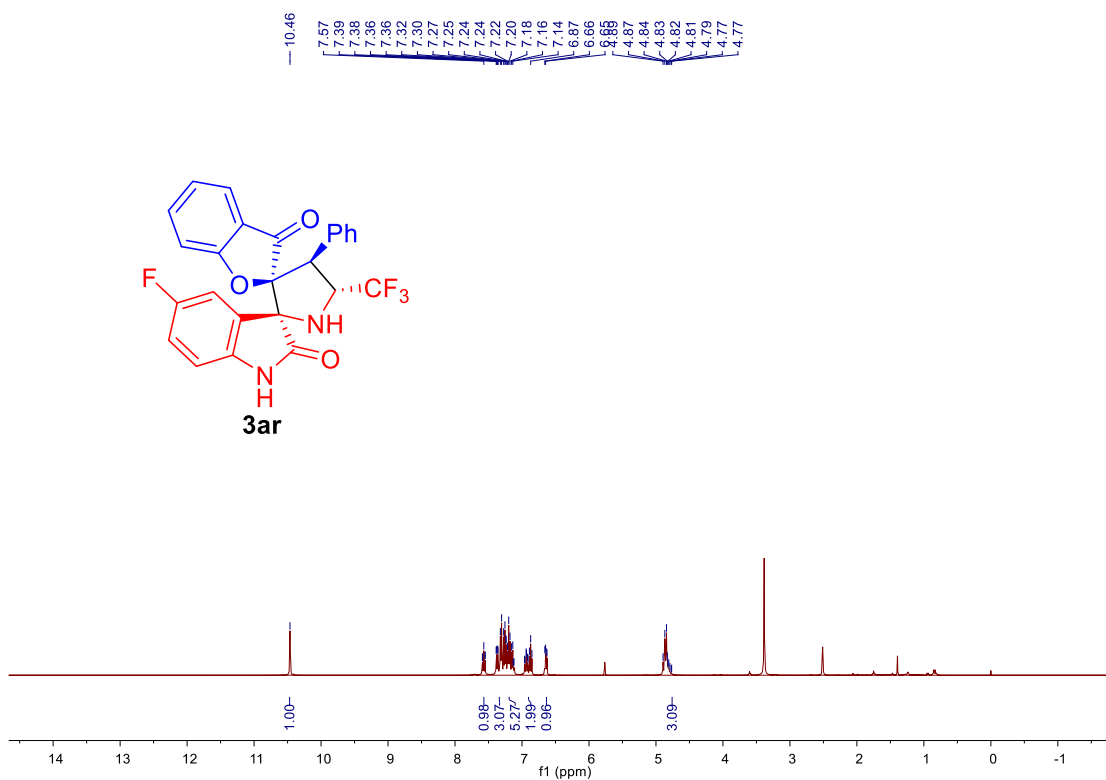
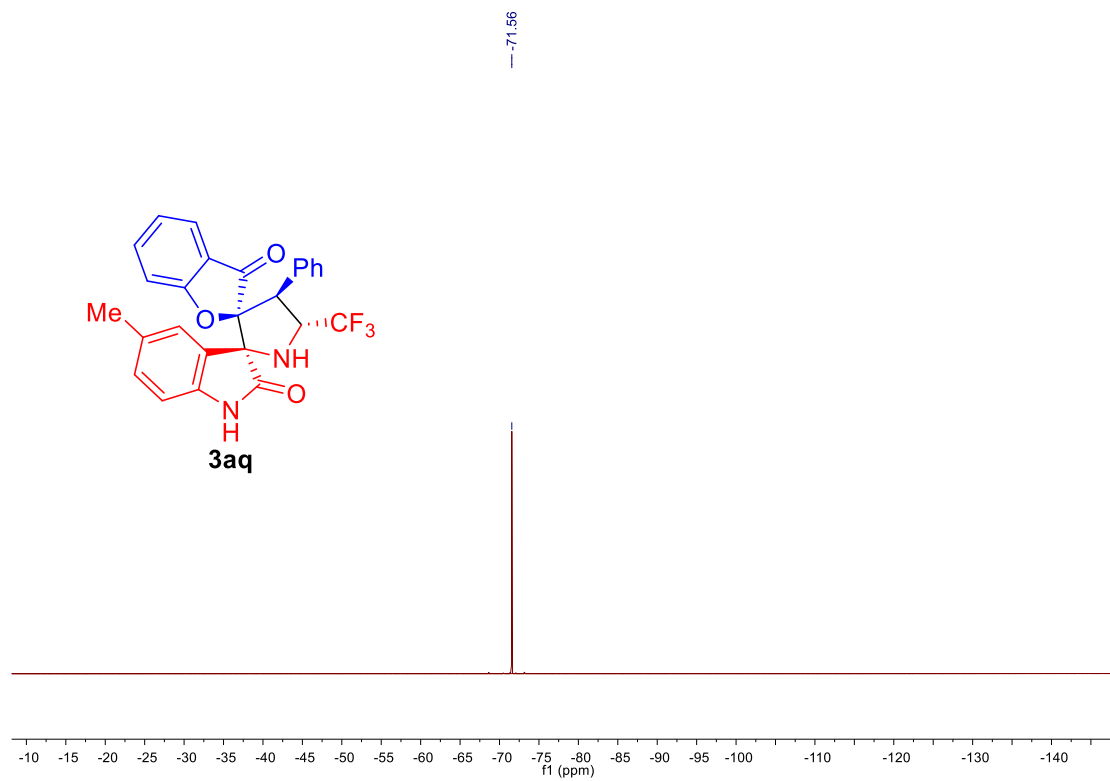


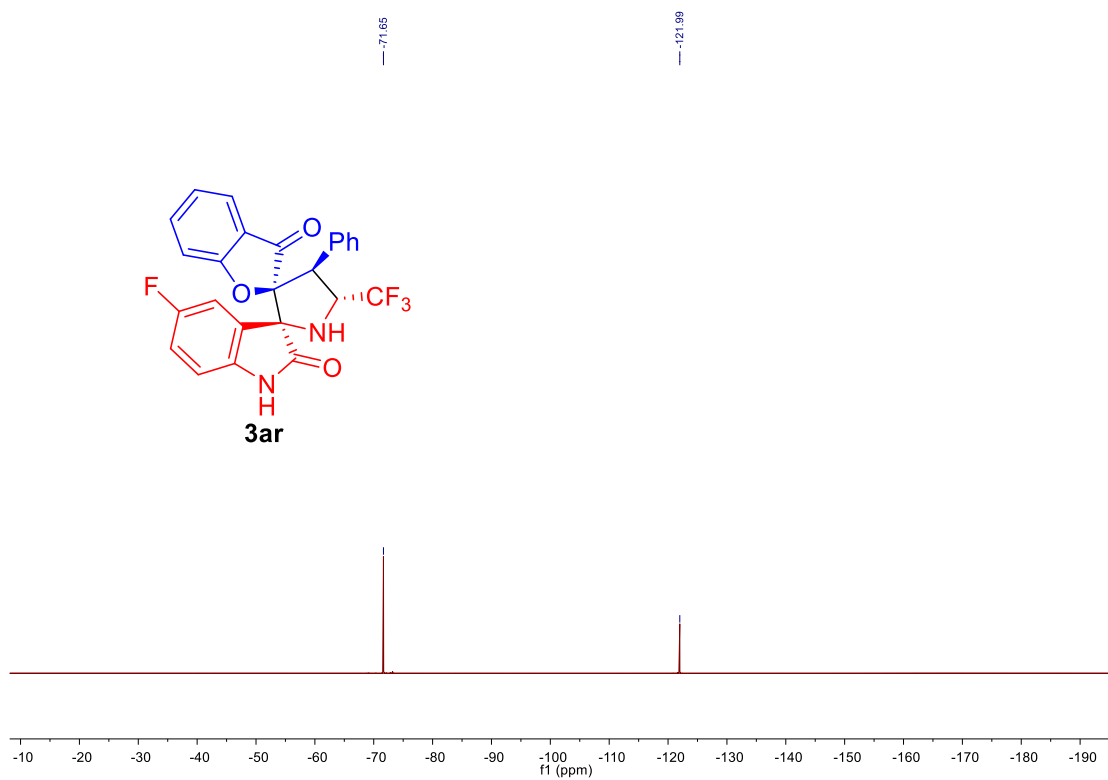
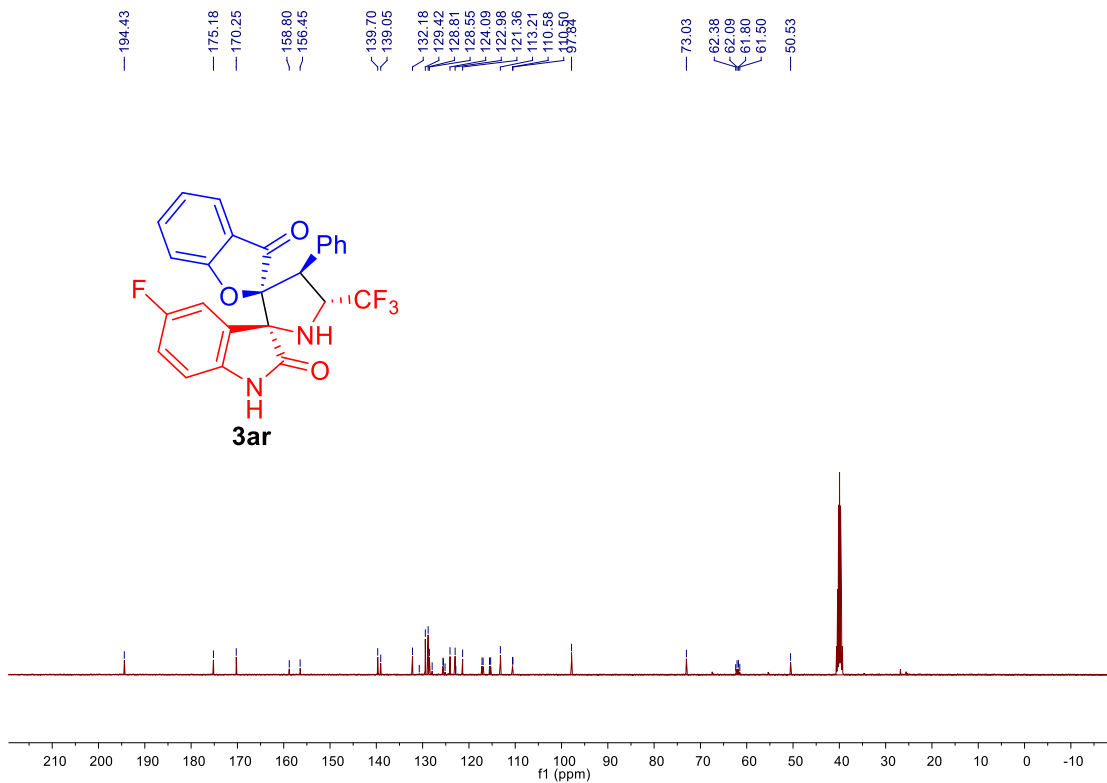


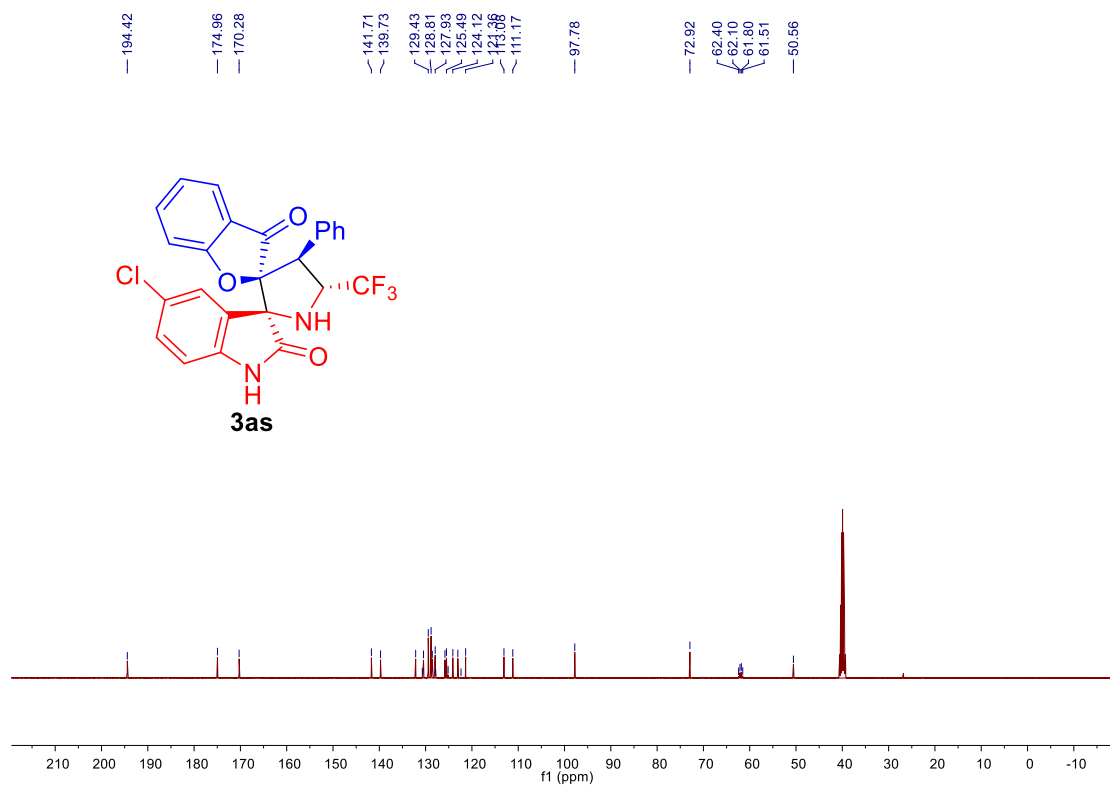
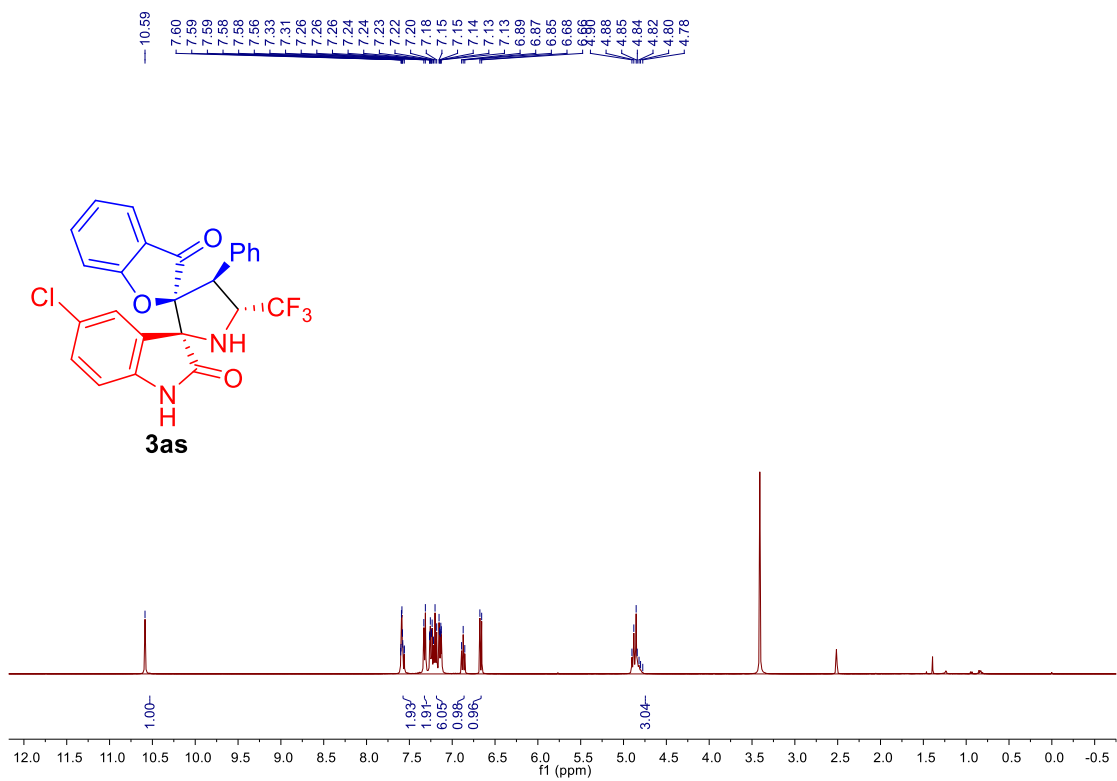


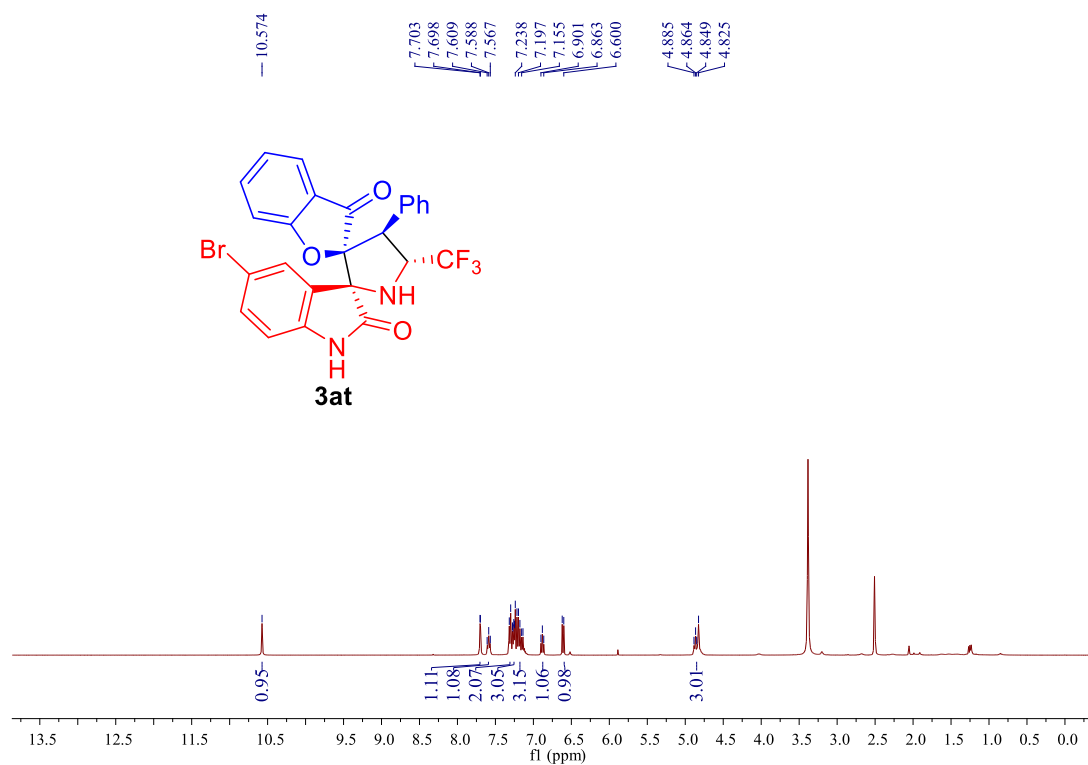
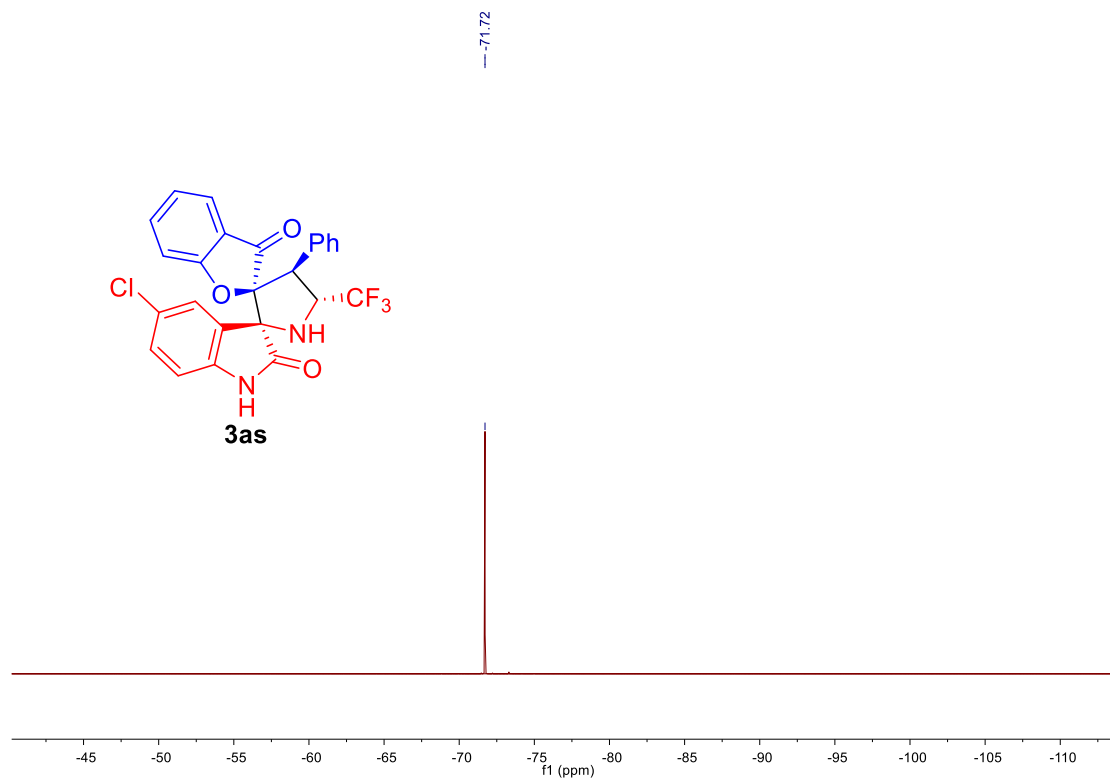


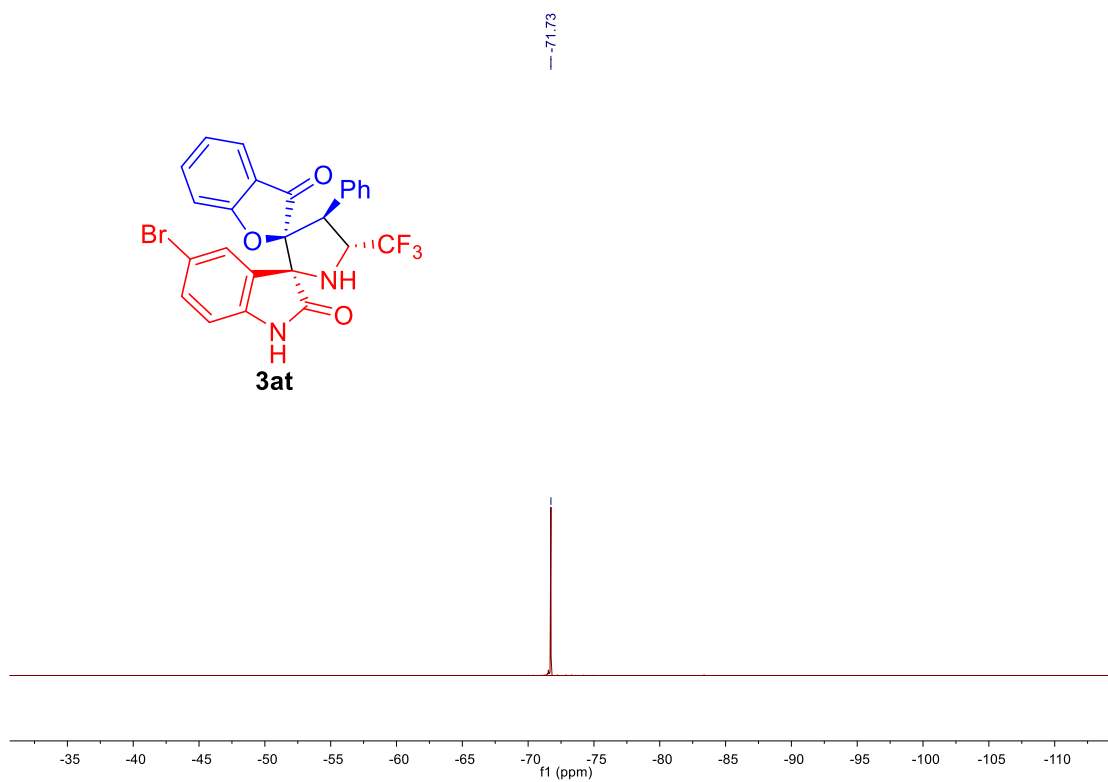
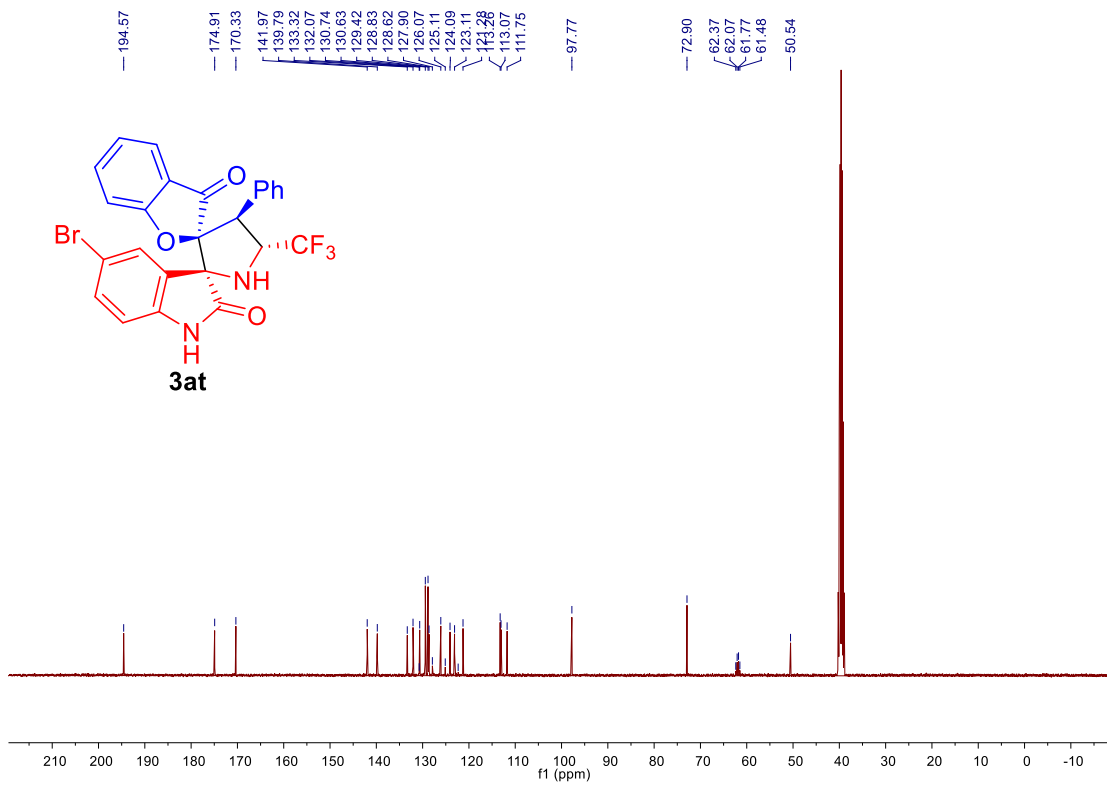


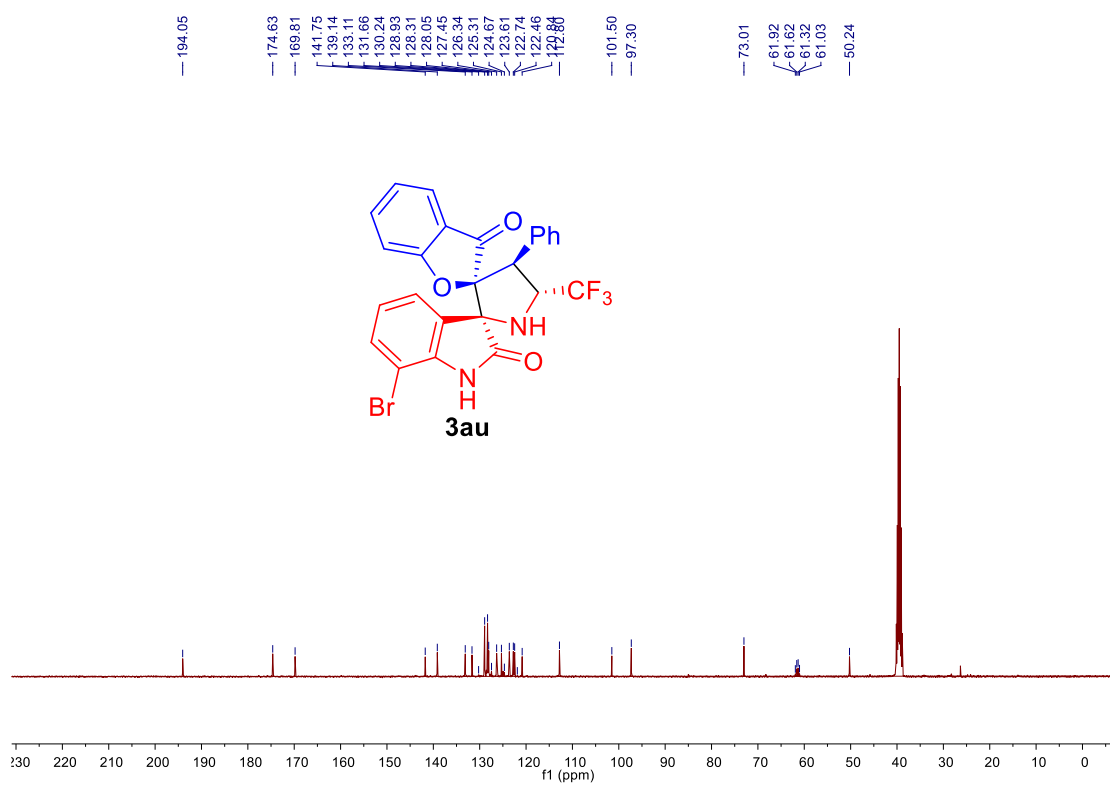
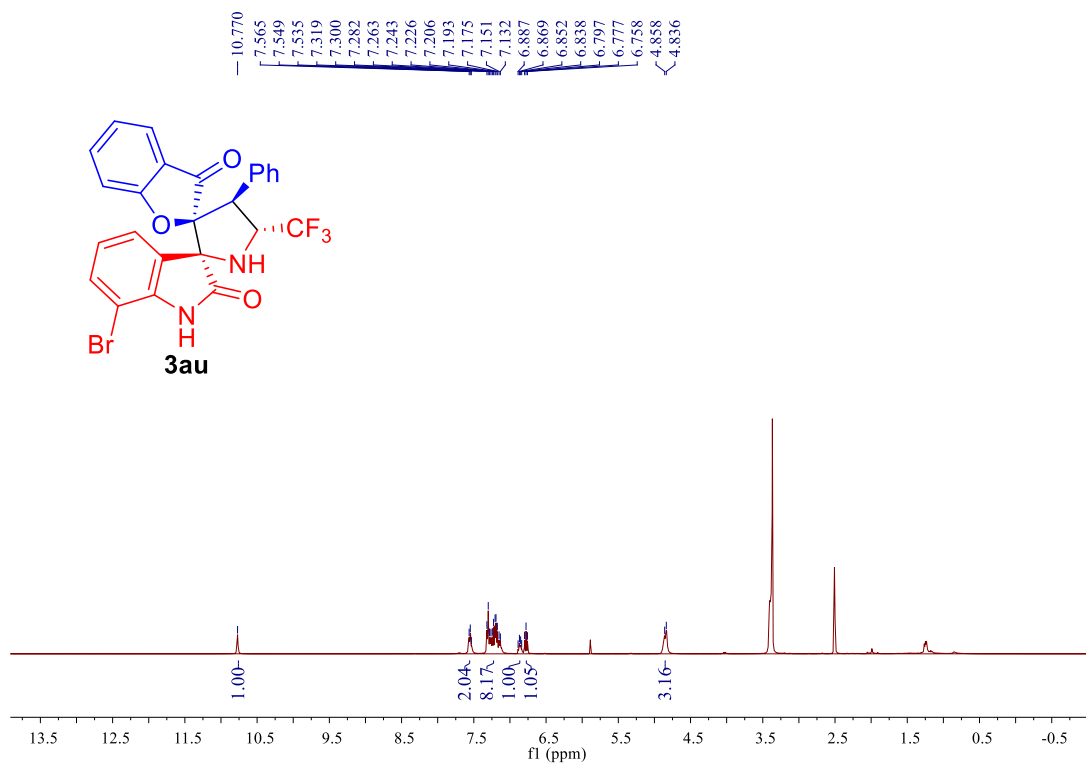


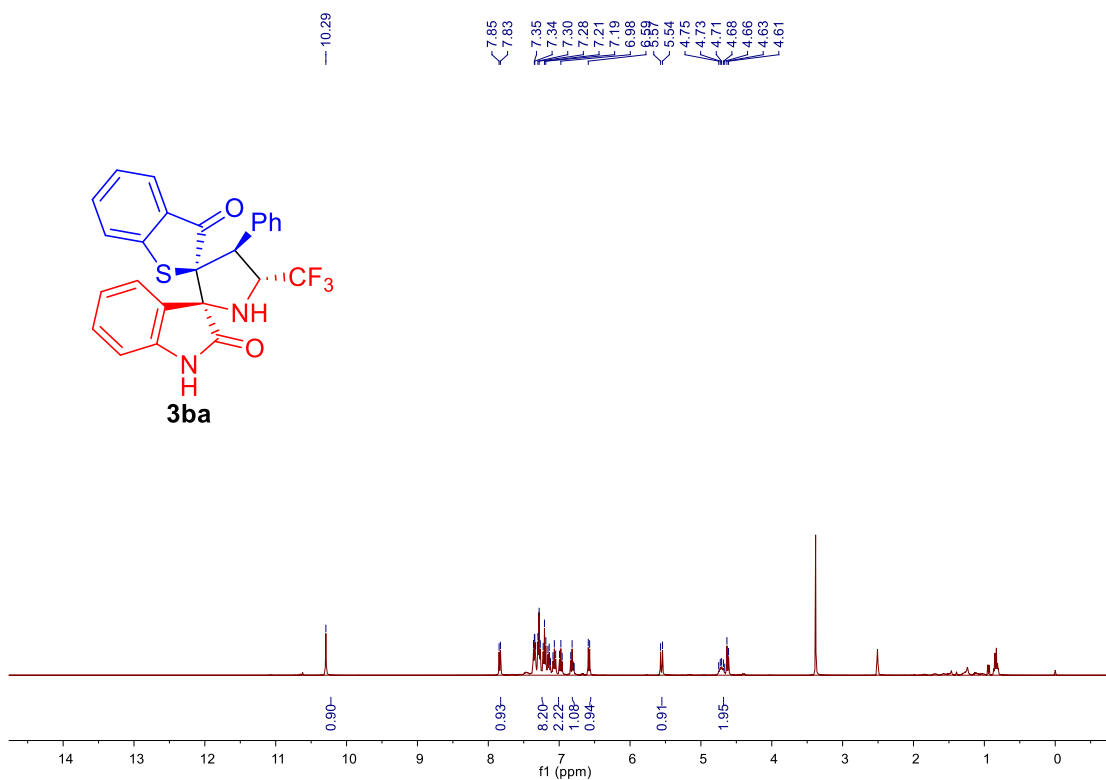
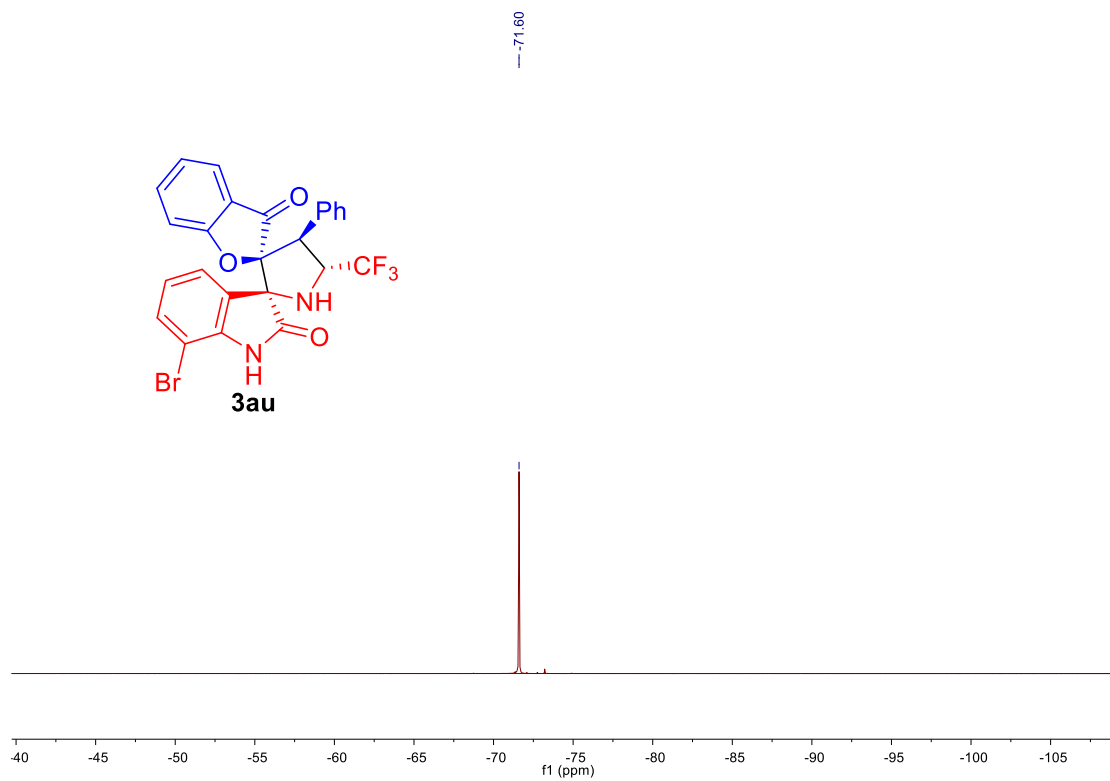


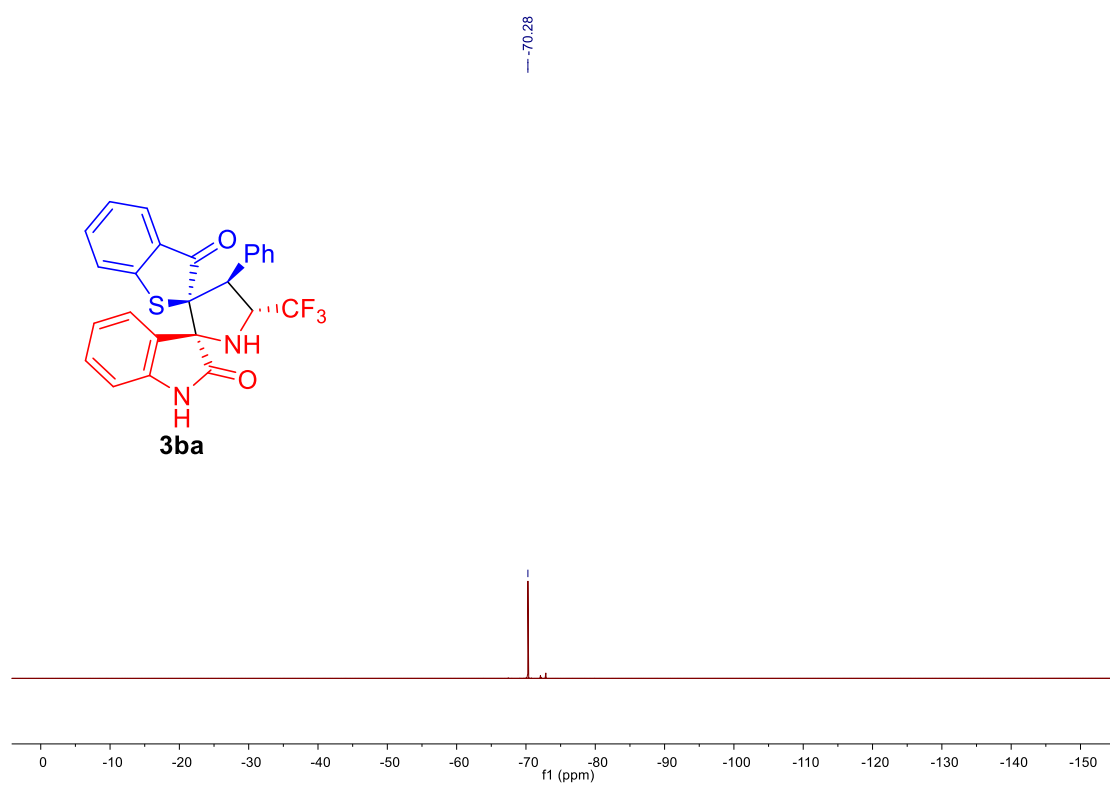
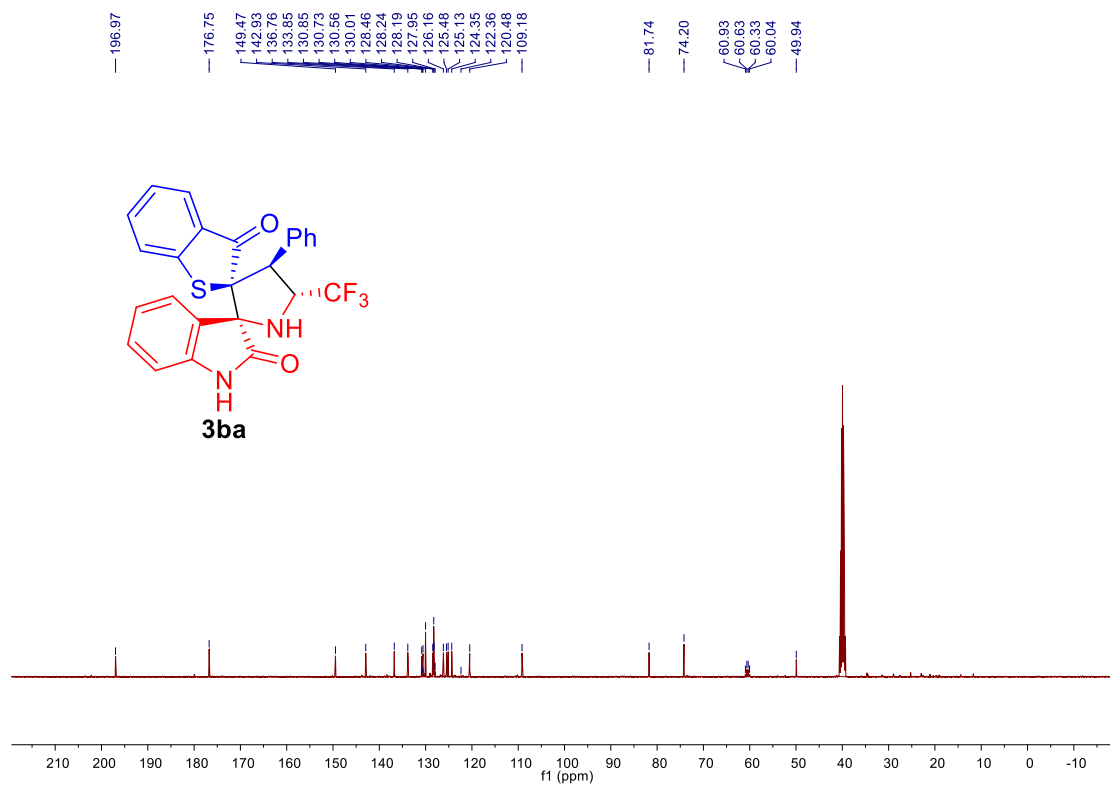


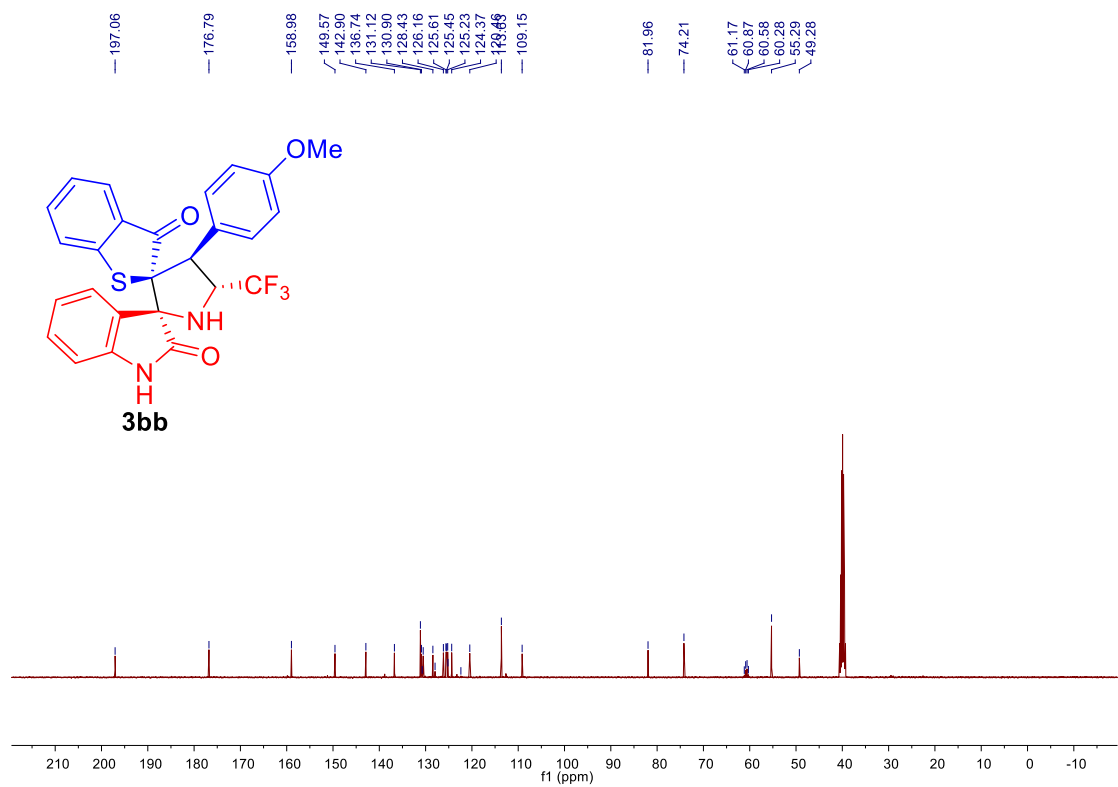
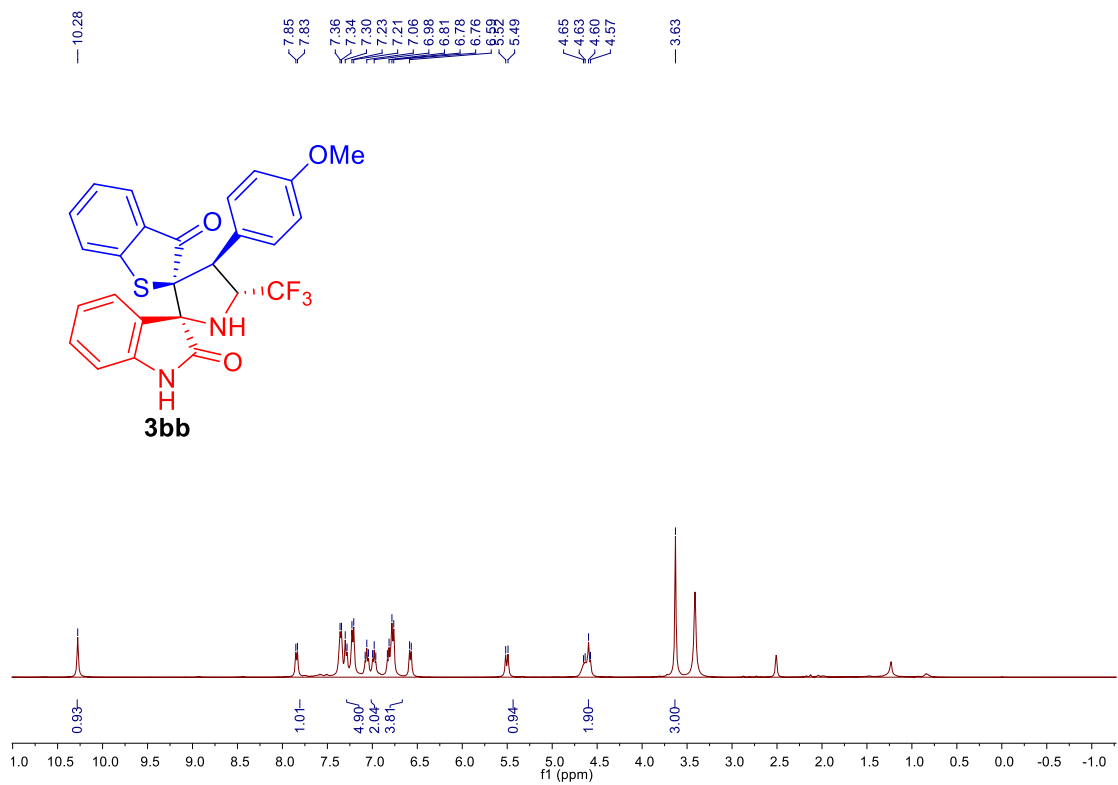


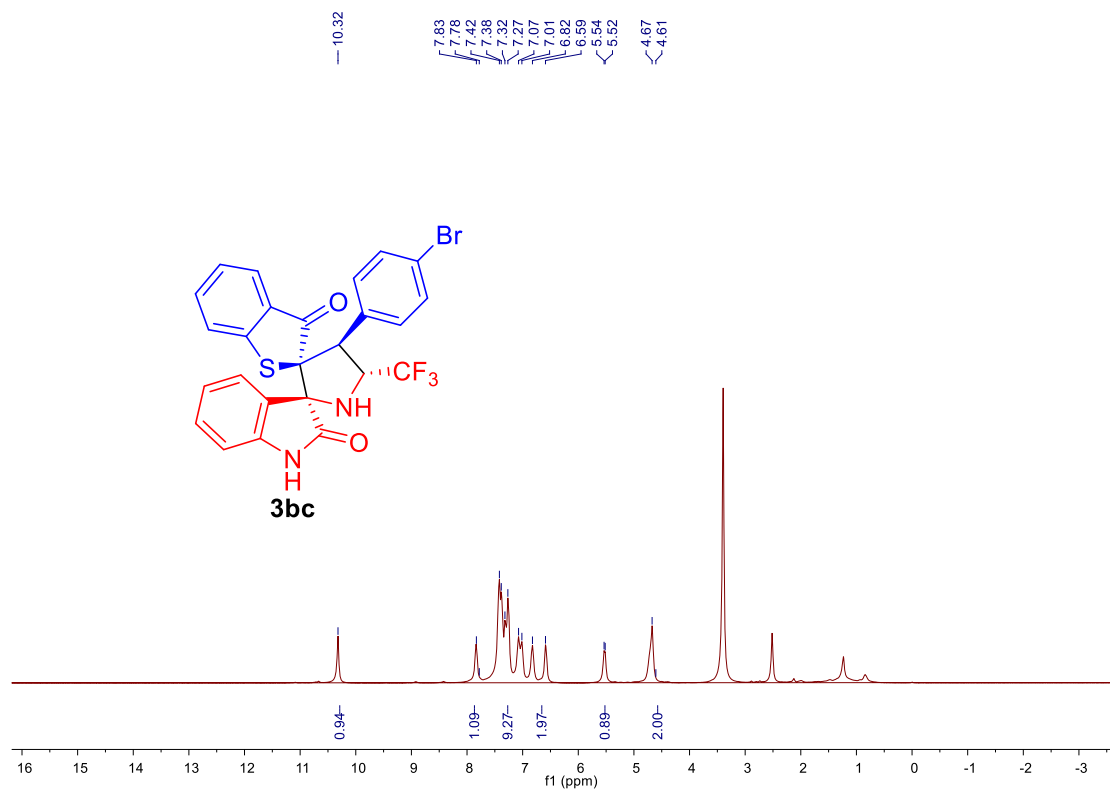
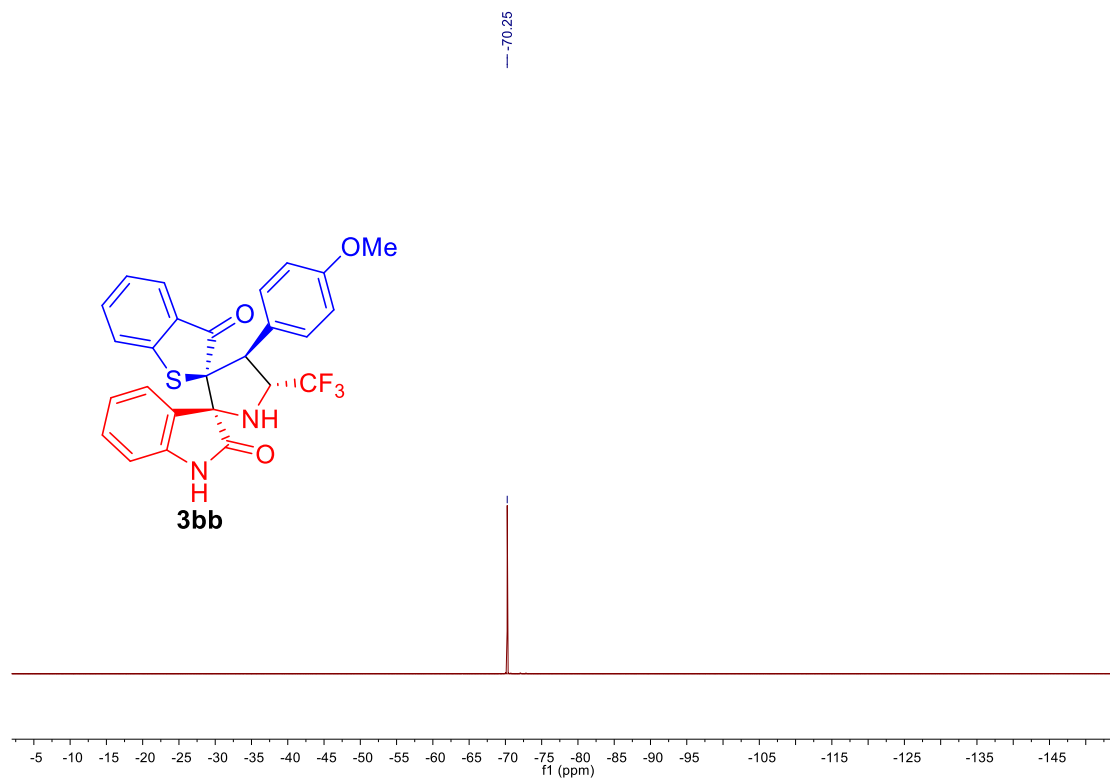


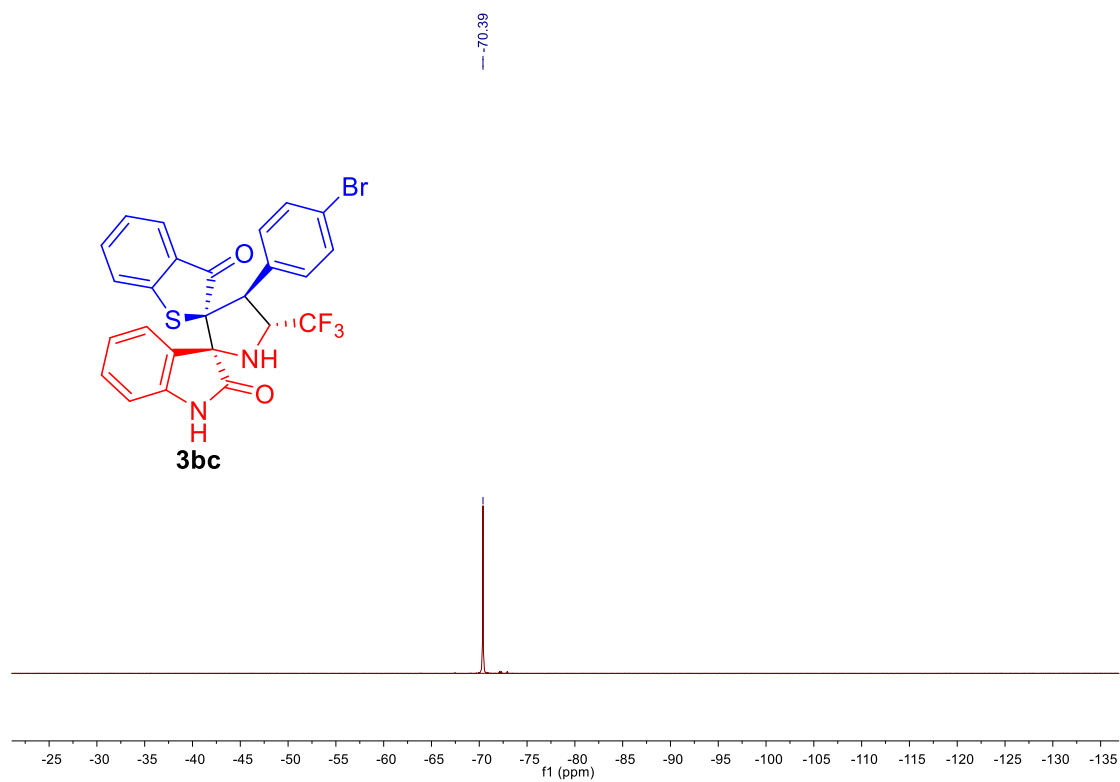
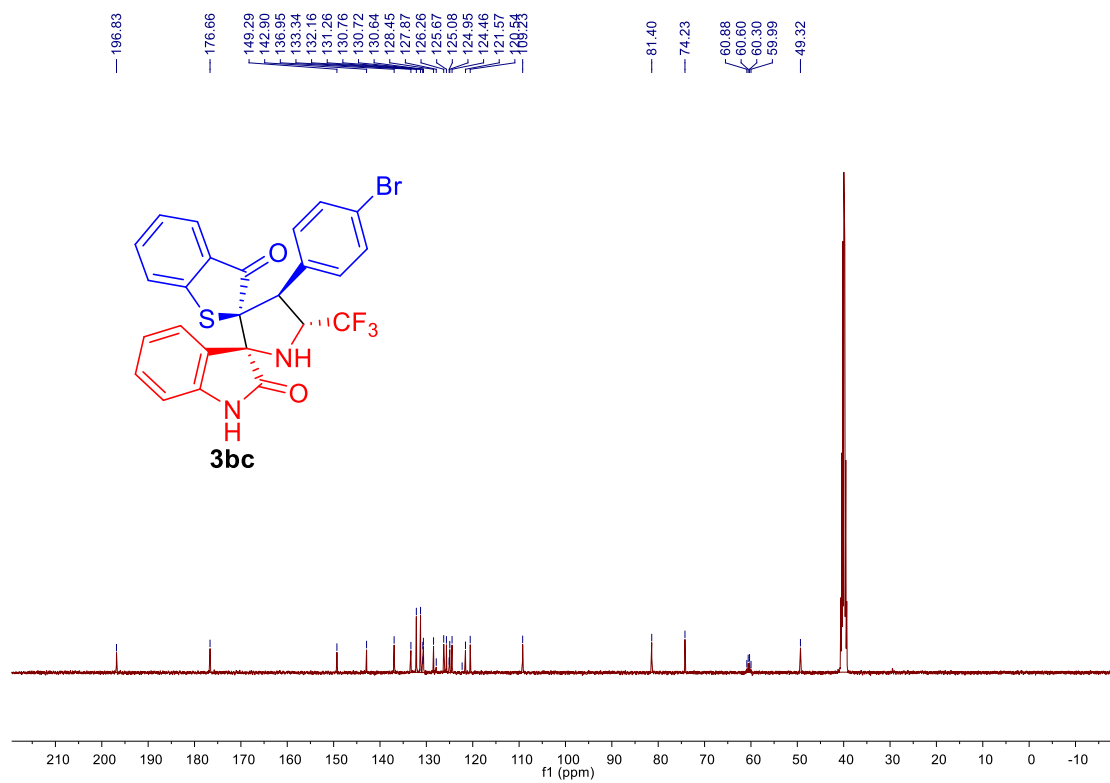


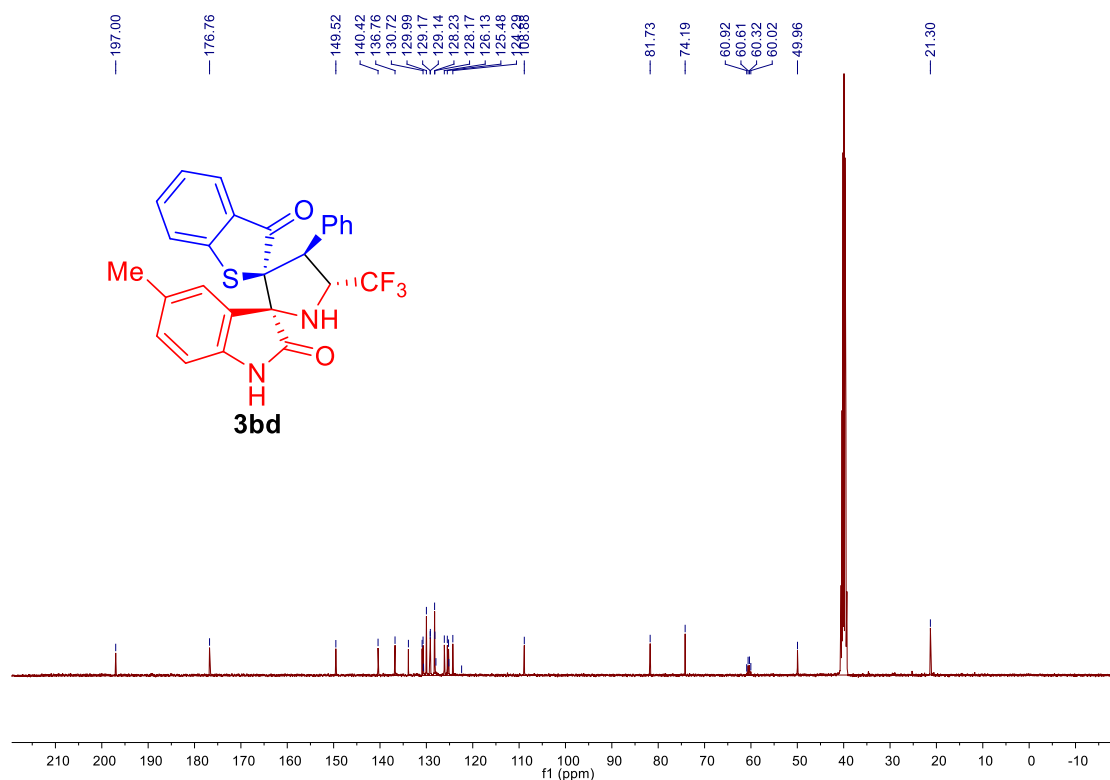
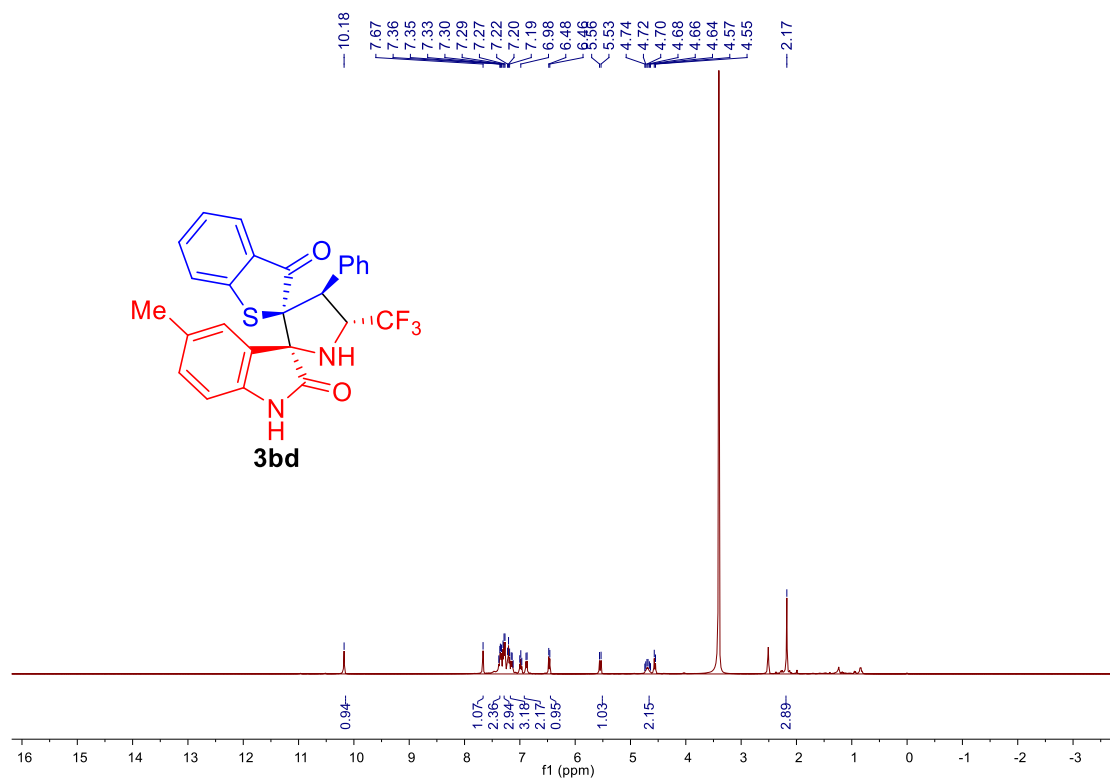


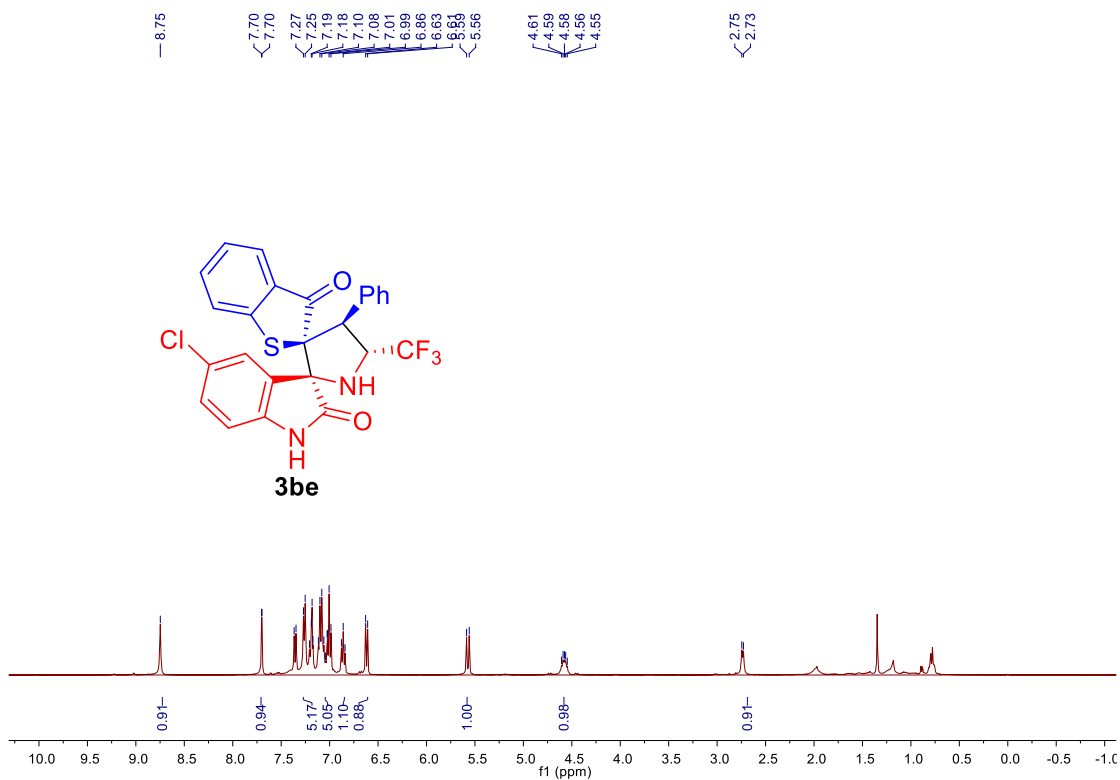
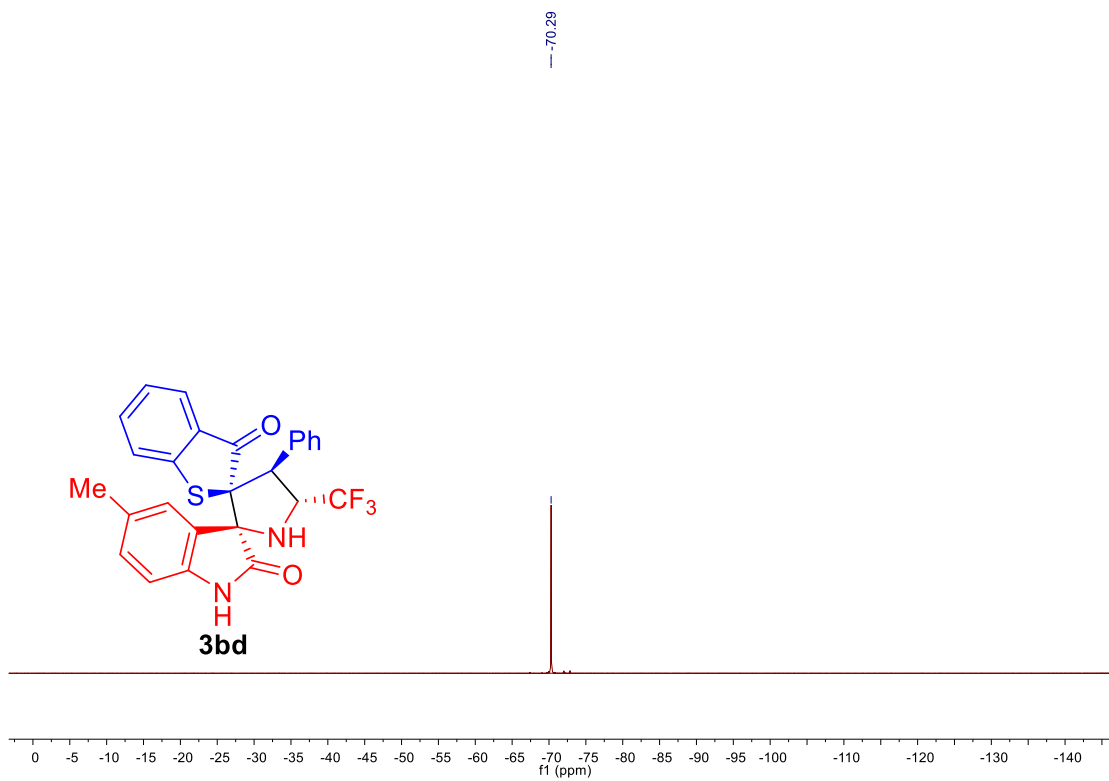


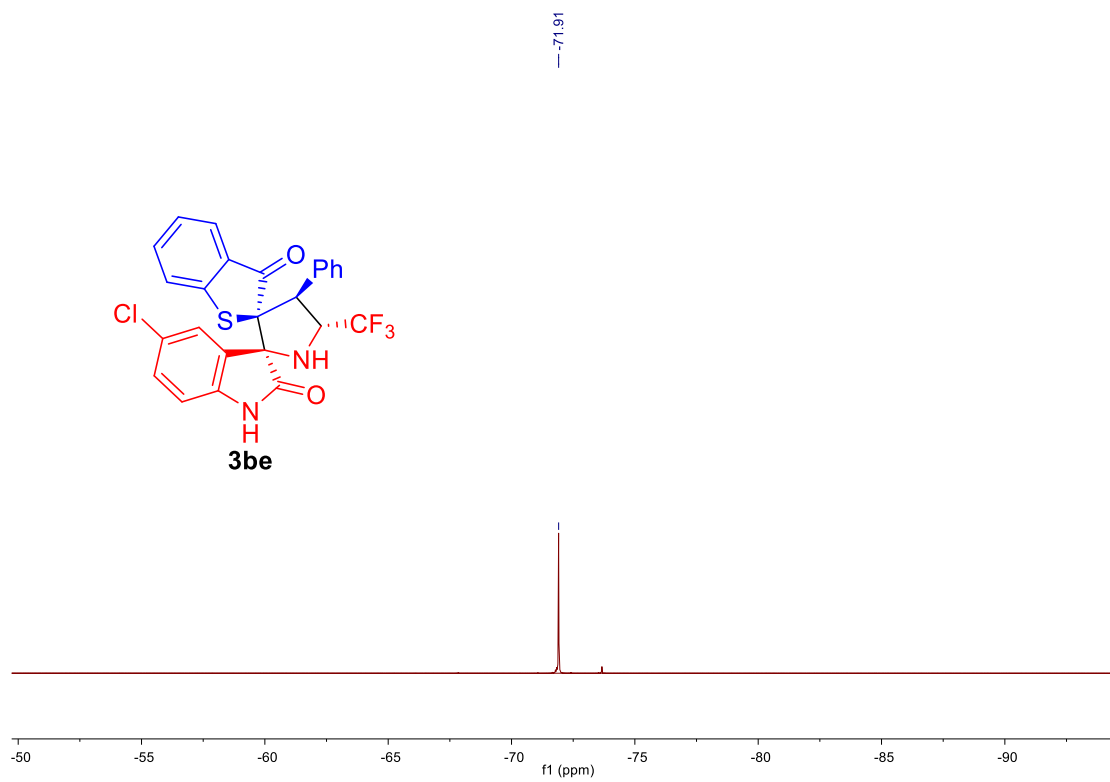
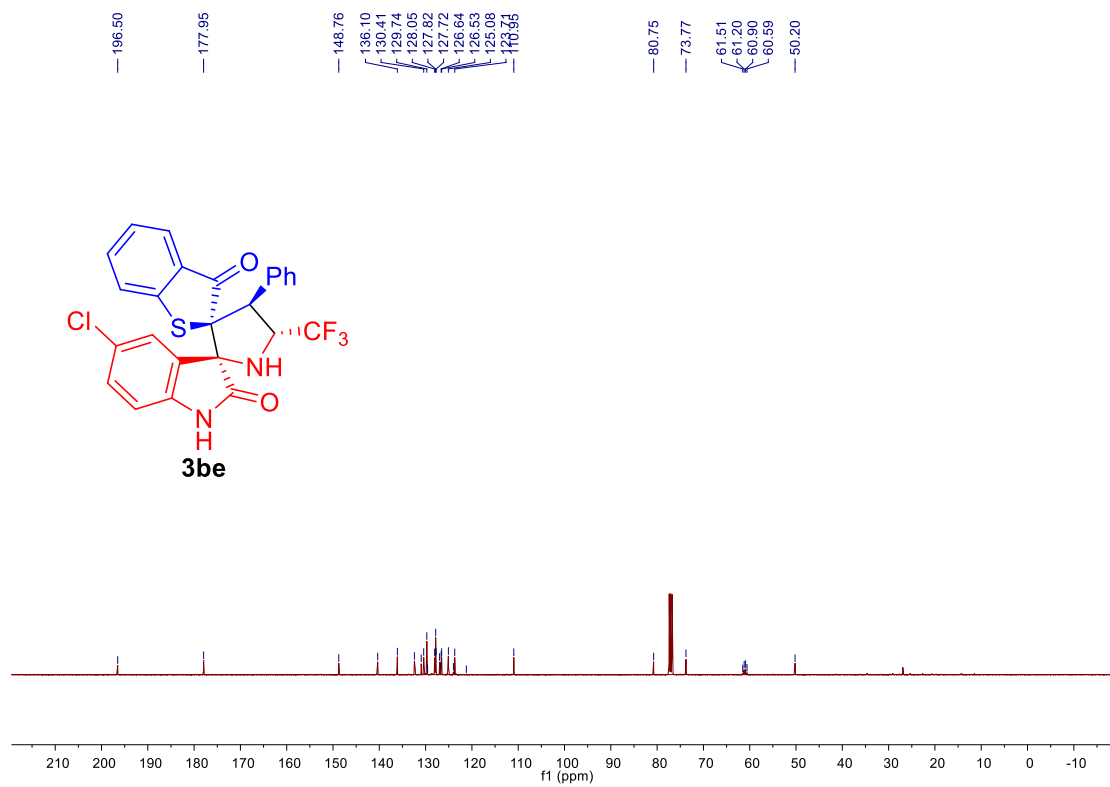


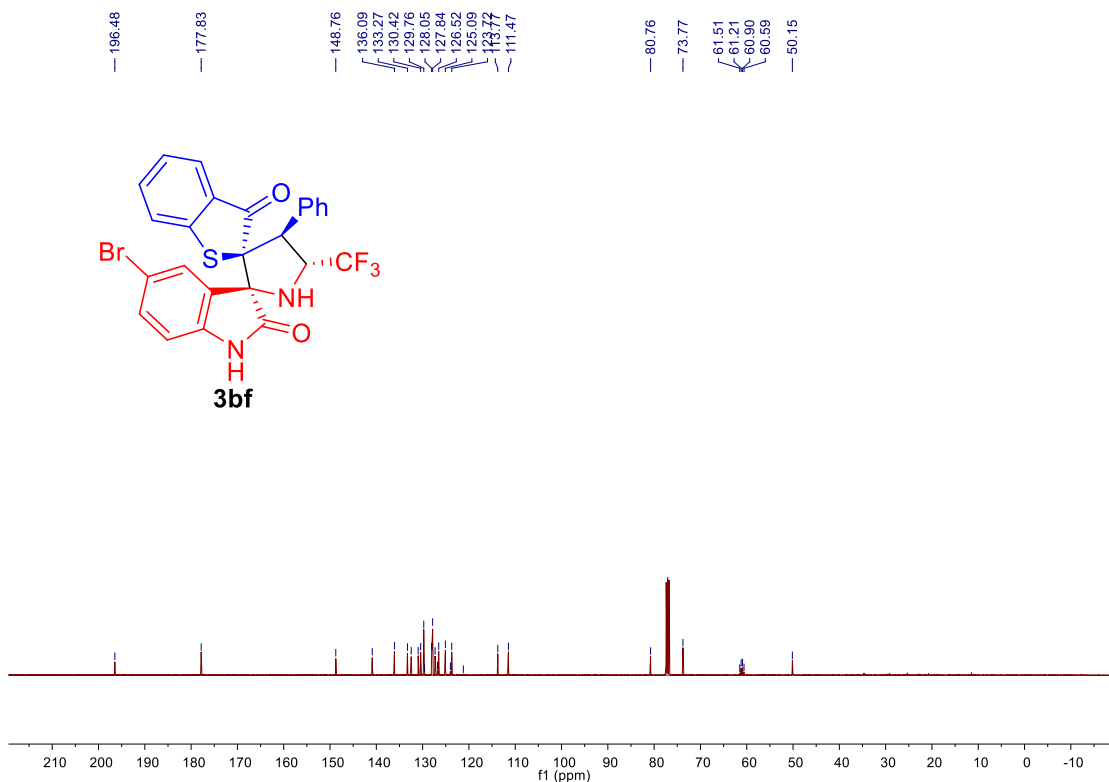
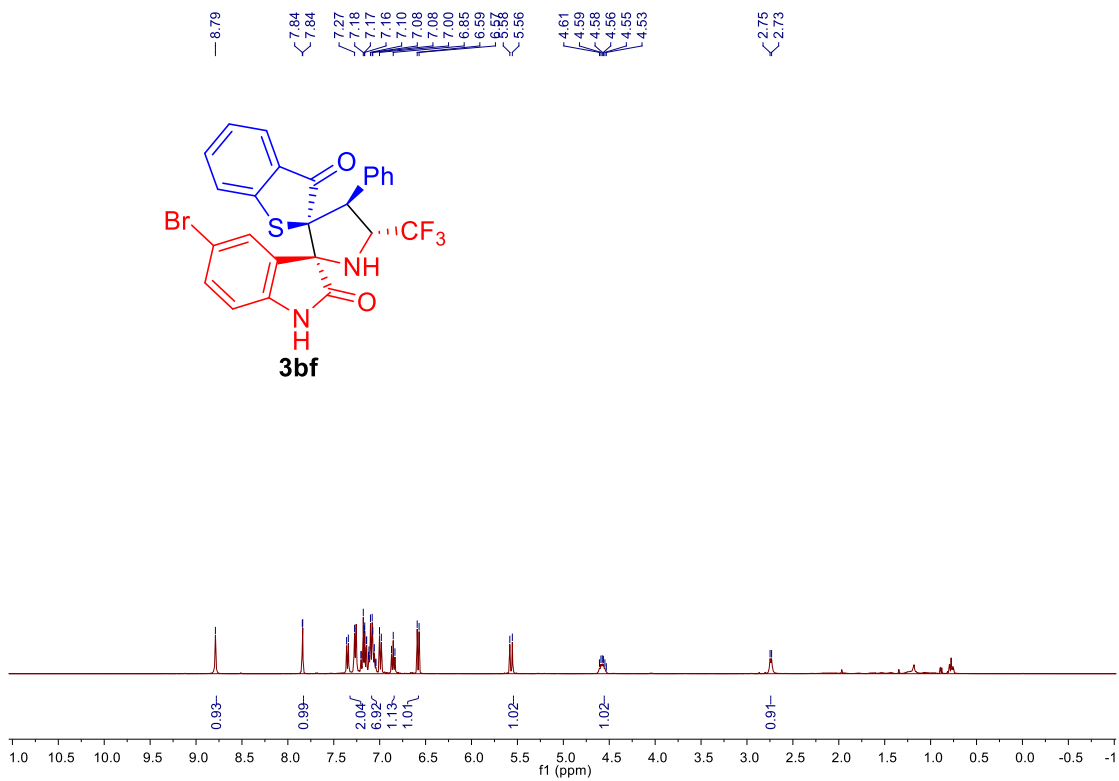


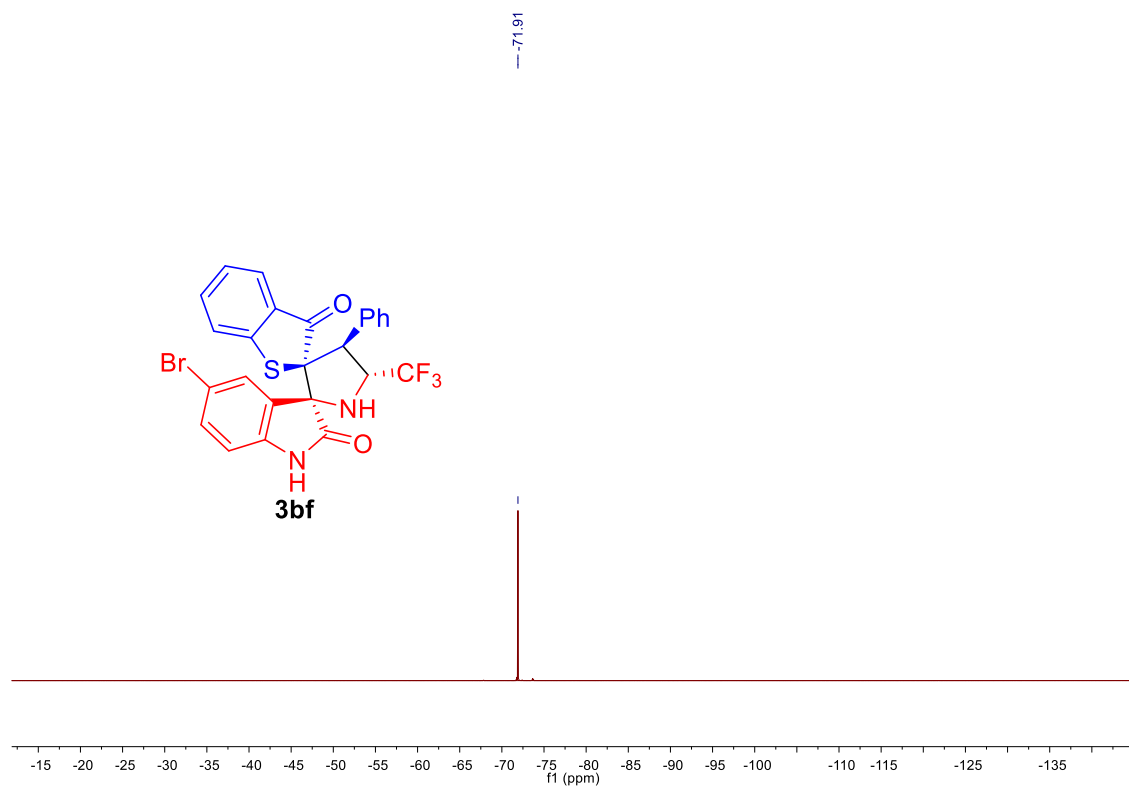


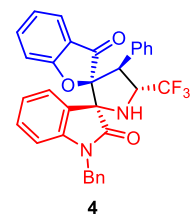
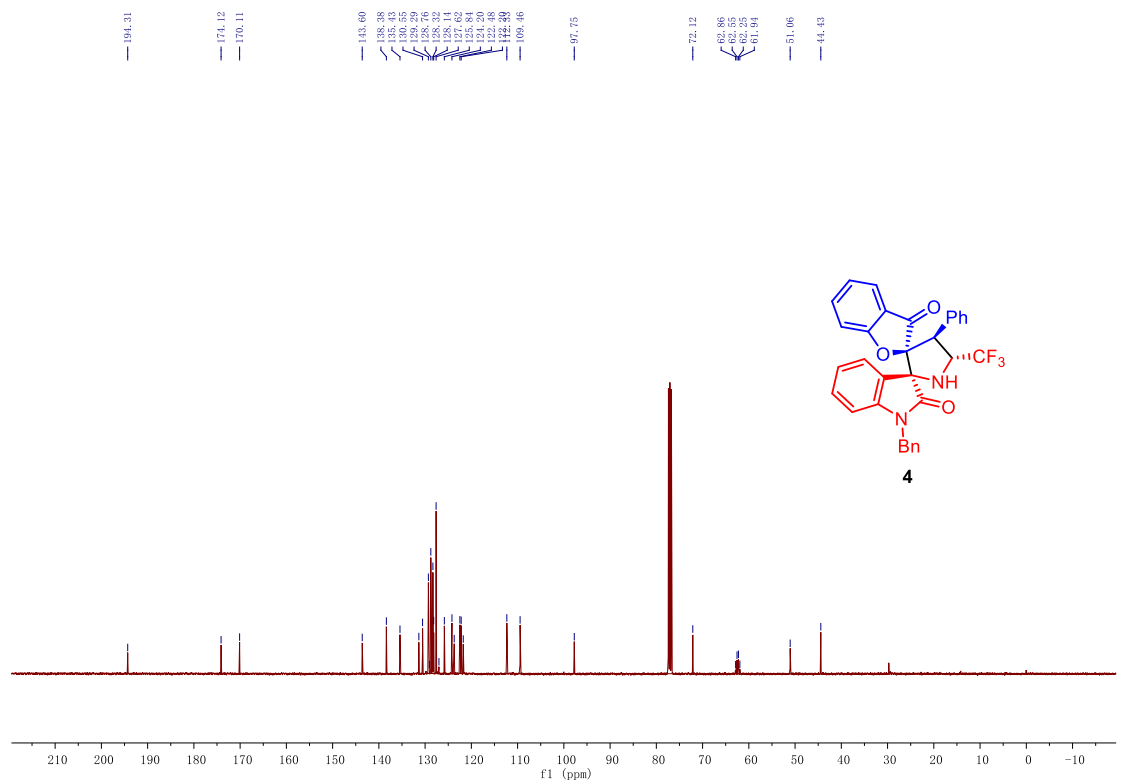
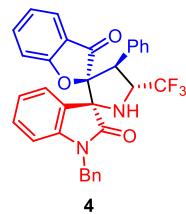
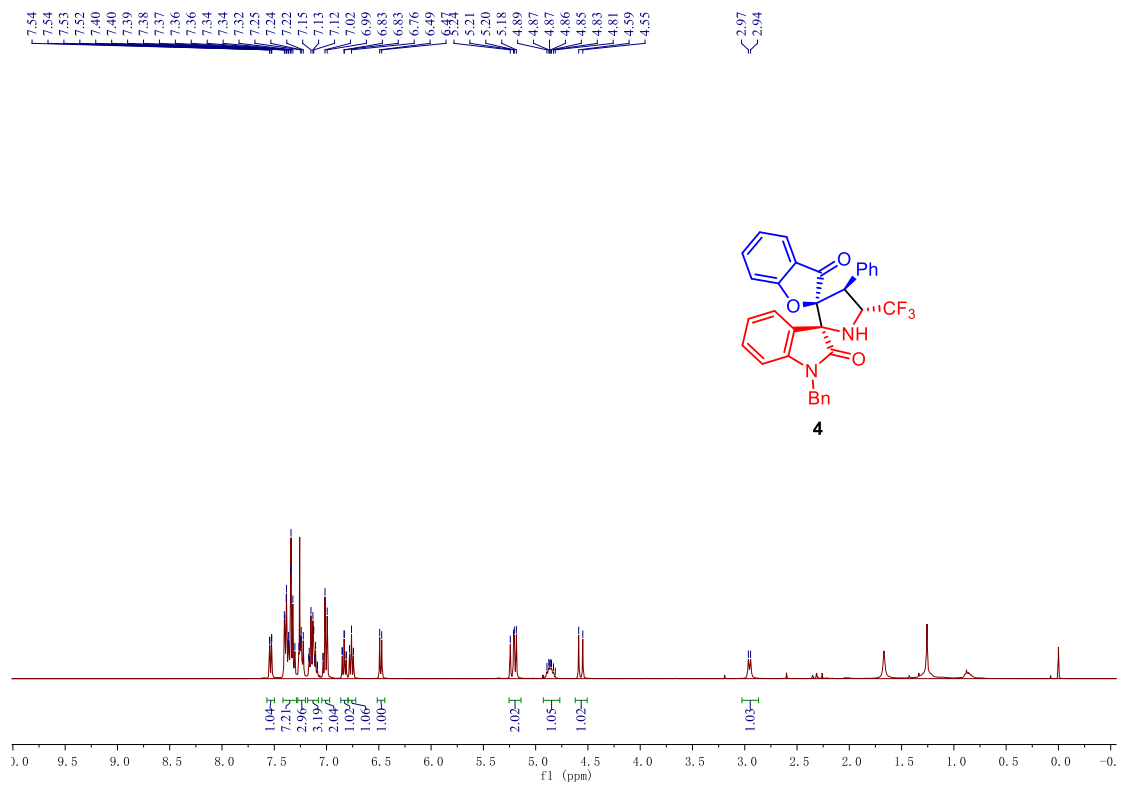


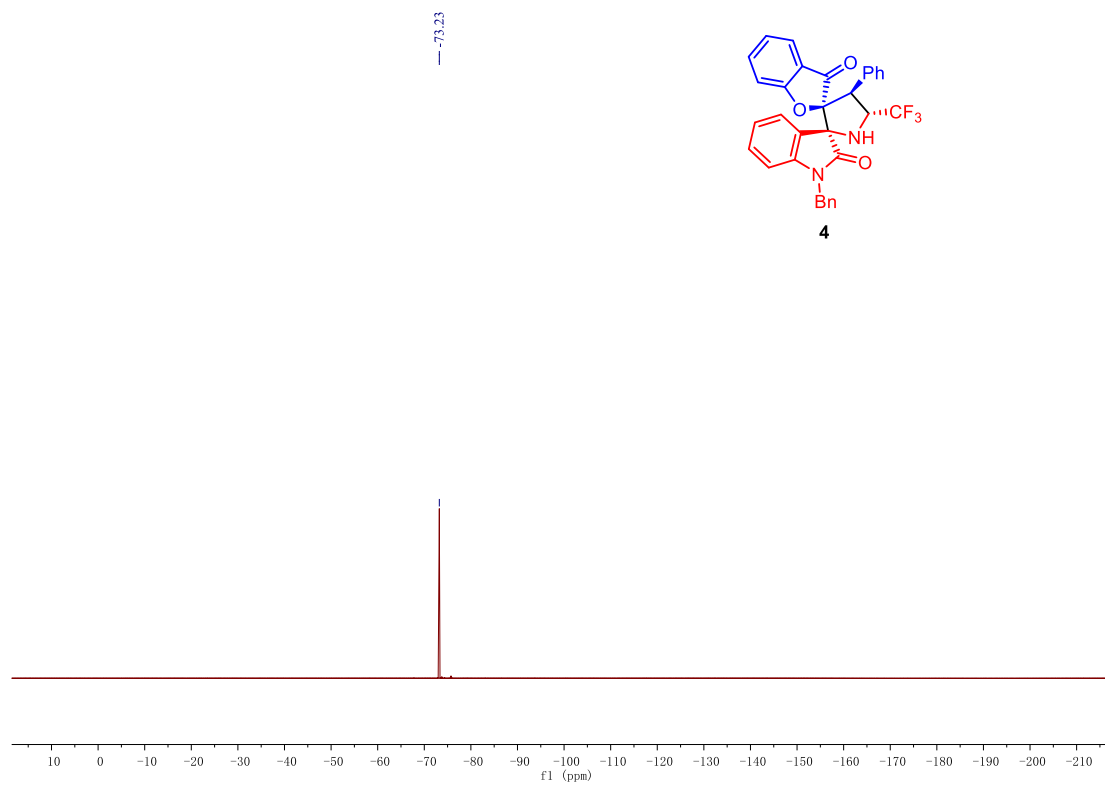




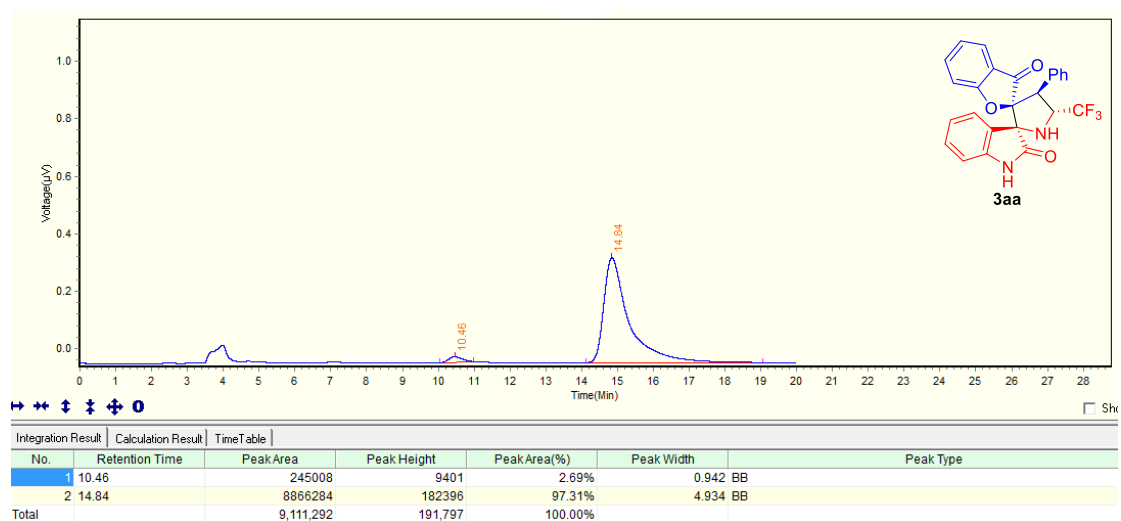
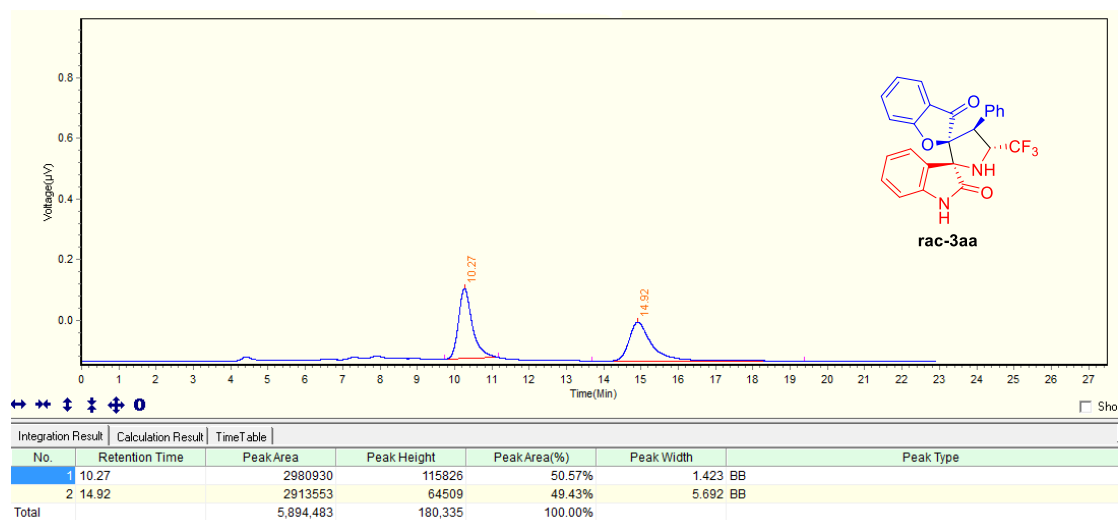


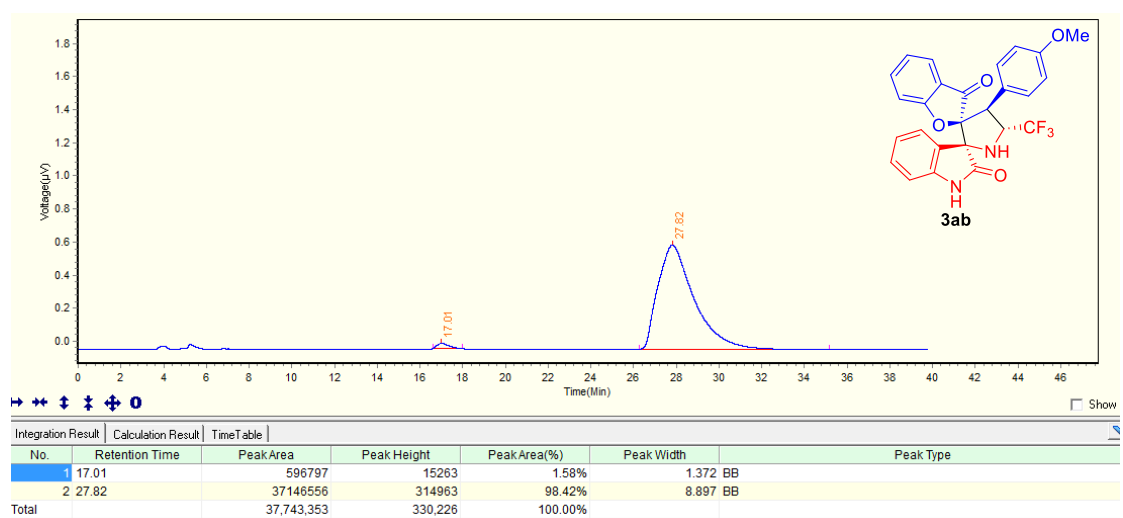
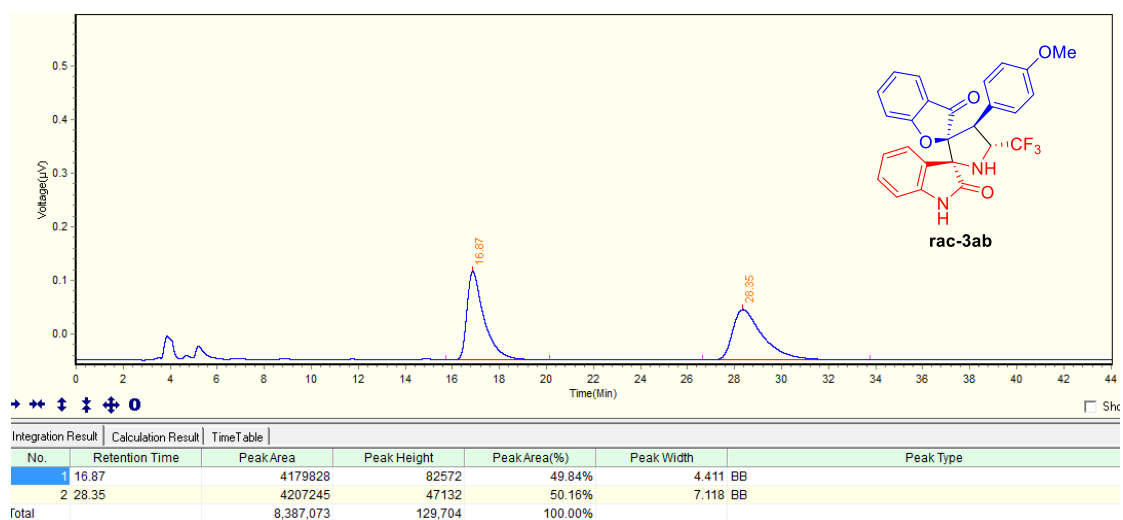


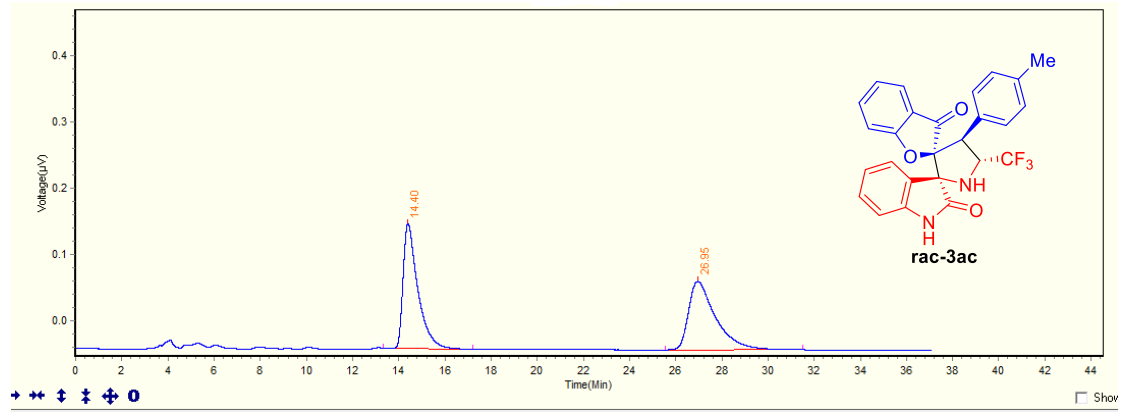




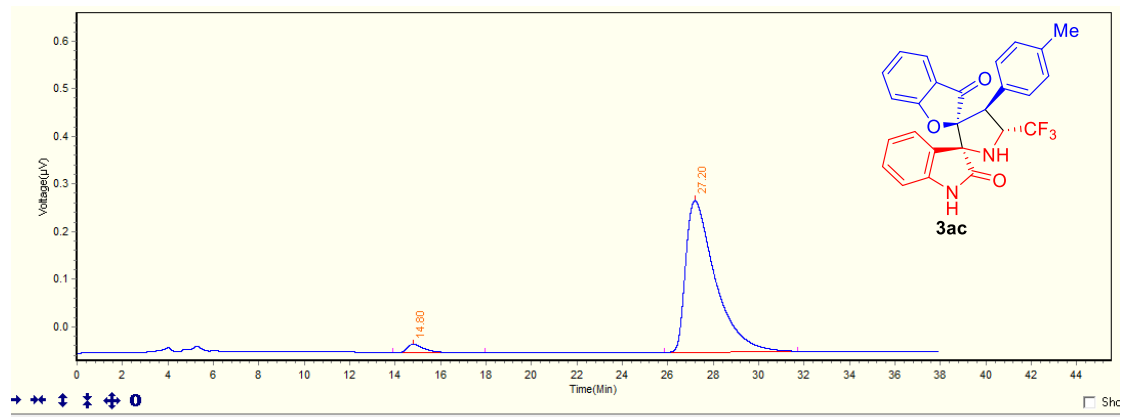
HPLC spectra of compounds



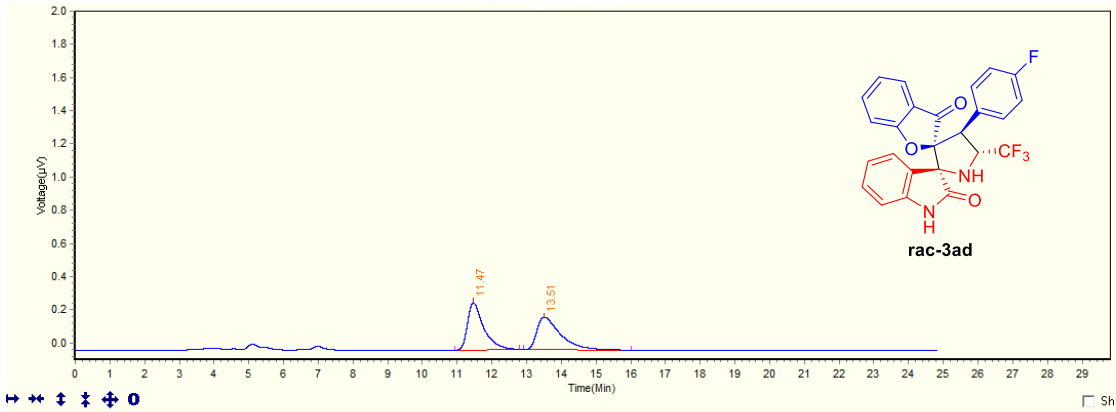




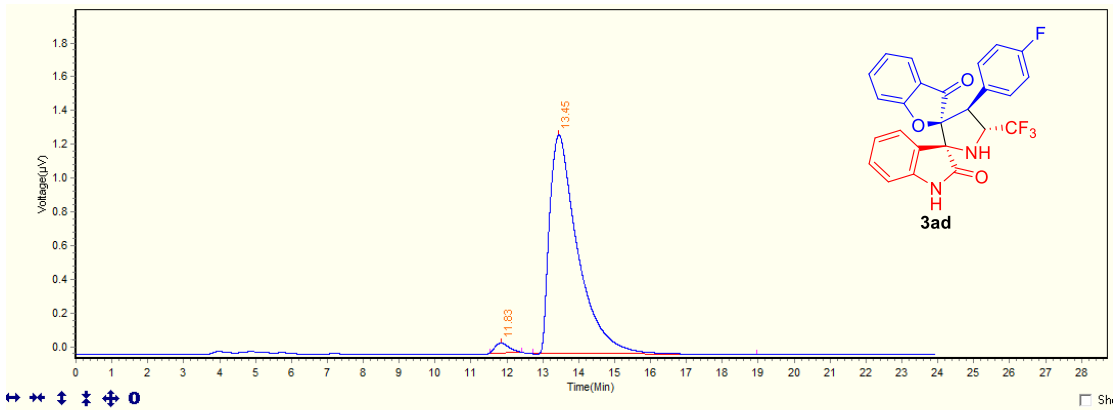
Integration Result		Calculation Result		TimeTable		
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	14.40	4154323	94161	49.81%	3.883	BB
2	26.95	4185221	51495	50.19%	5.958	BB
Total		8,339,544	145,656	100.00%		



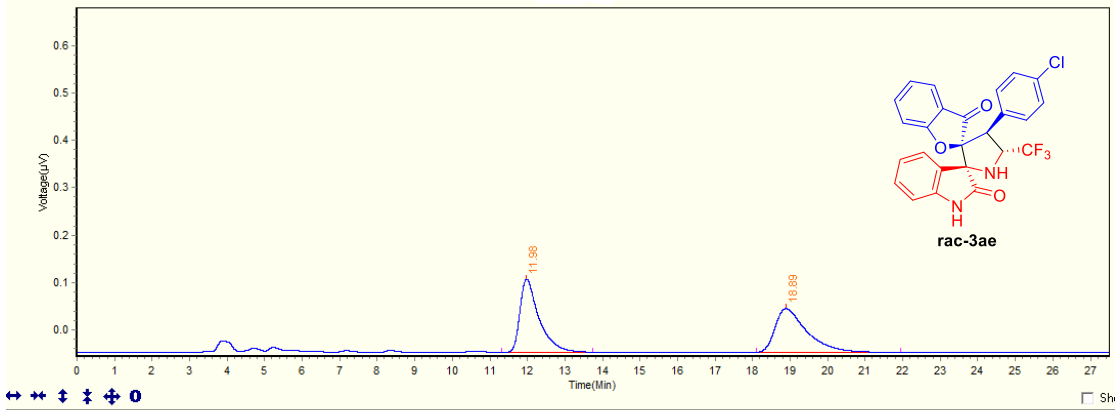
Integration Result		Calculation Result		TimeTable		
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	14.80	422960	8918	2.86%	4.06	BB
2	27.20	14386644	159110	97.14%	5.865	BB
Total		14,809,604	168,028	100.00%		



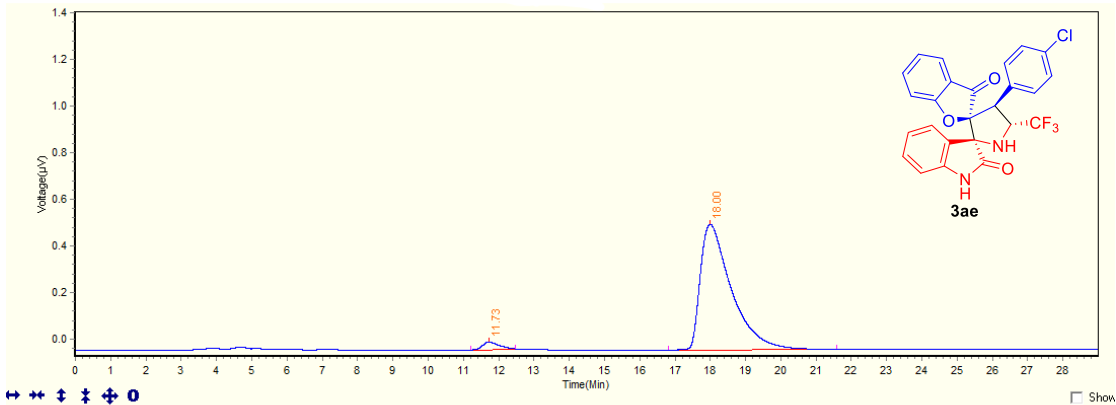
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	11.47	4621110	142698	50.19%	1.872	BB
2	13.51	4586114	97646	49.81%	3.103	BB
Total		9,207,224	240,344	100.00%		



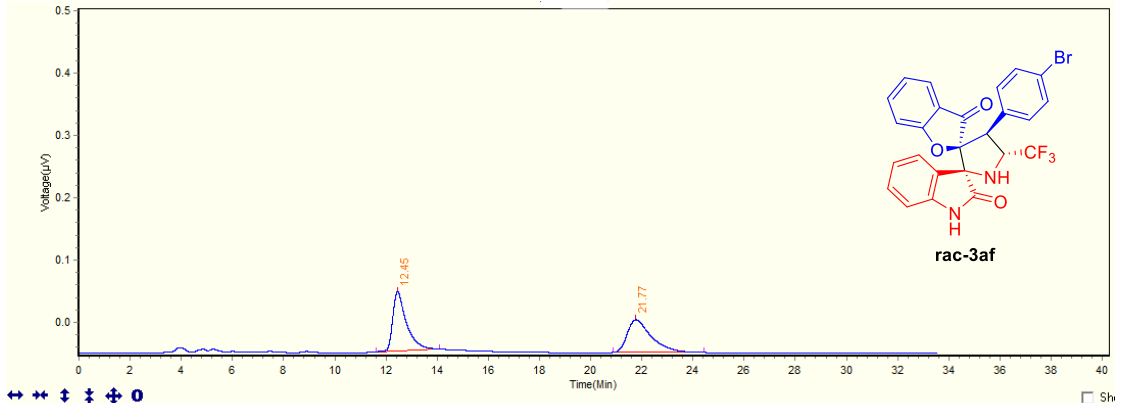
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	11.83	749337	28128	2.07%	0.886	BB
2	13.45	35401129	649634	97.93%	6.23	BB
Total		36,150,466	677,762	100.00%		



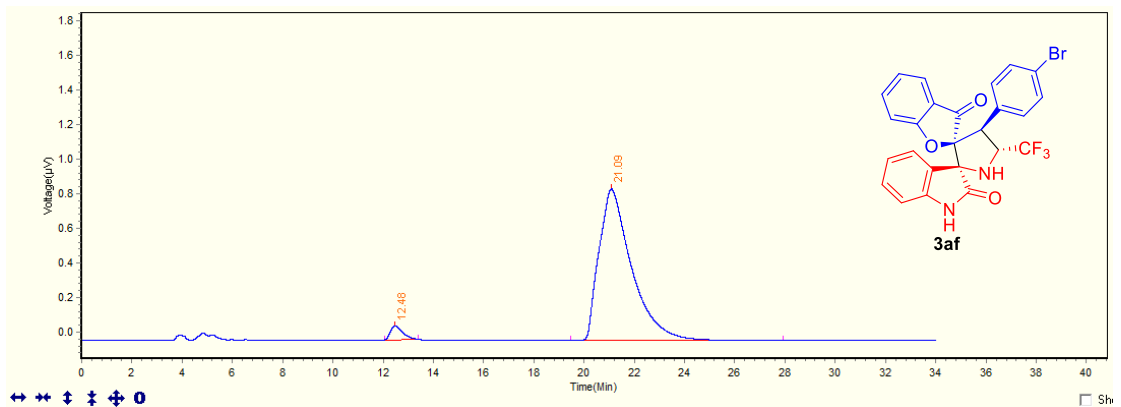
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	11.98	2731344	77483	50.11%	2.426	BB
2	18.89	2718971	46225	49.89%	3.843	BB
Total		5,450,315	123,708	100.00%		



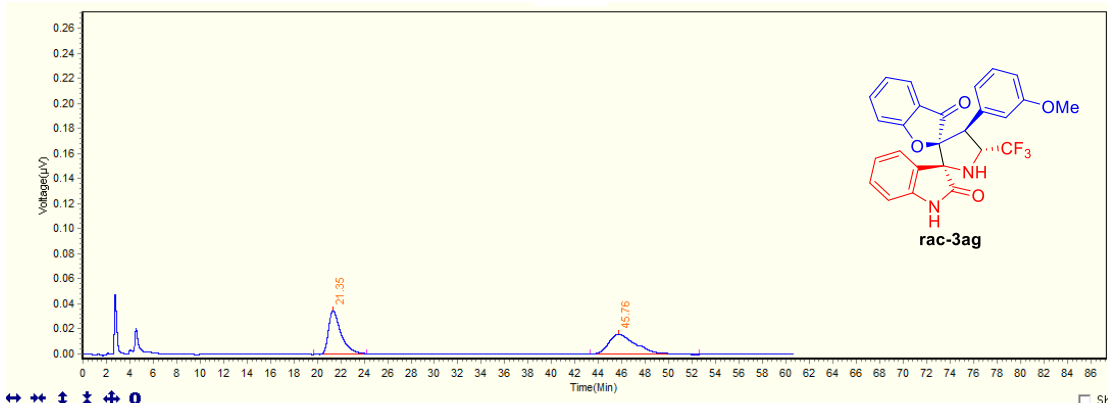
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	11.73	500945	15859	2.96%	1.265	BB
2	18.00	16404616	268692	97.04%	4.771	BB
Total		16,905,561	284,551	100.00%		



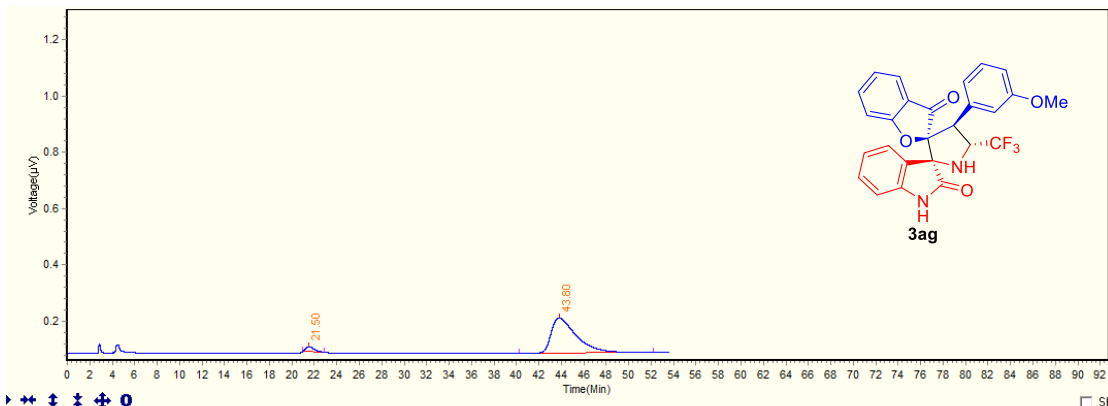
No.	Retention Time	PeakArea	Peak Height	PeakArea(%)	Peak Width	Peak Type
1	12.45	1764057	47327	50.61%	2.459	BB
2	21.77	1721348	26193	49.39%	3.548	BB
Total		3,485,405	73,520	100.00%		



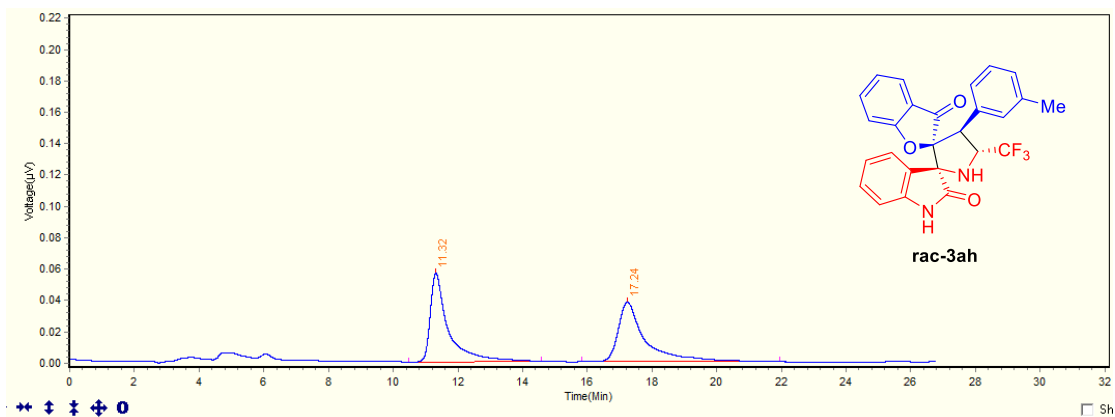
No.	Retention Time	PeakArea	Peak Height	PeakArea(%)	Peak Width	Peak Type
1	12.48	1334165	39618	3.26%	1.349	BB
2	21.09	39601014	438361	96.74%	8.459	BB
Total		40,935,179	477,979	100.00%		



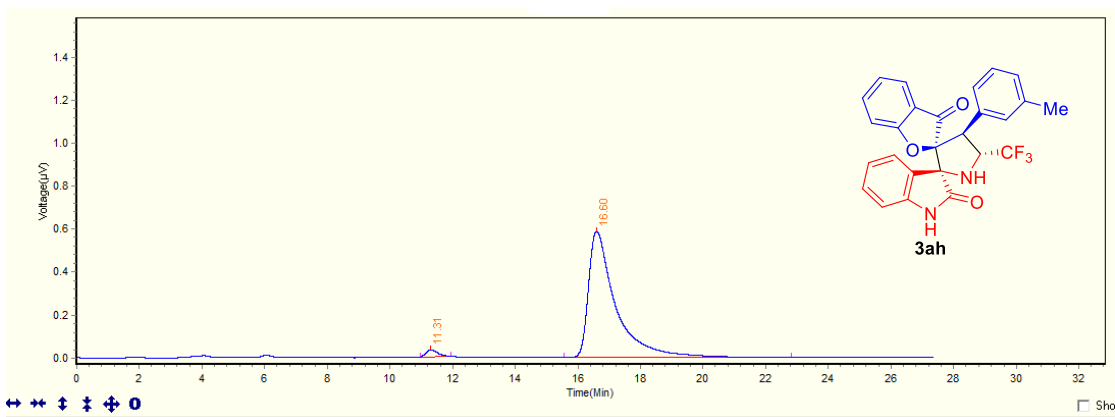
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	21.35	1265524	17303	49.80%	4.528	BB
2	45.76	1275886	7874	50.20%	9.318	BB
Total		2,541,410	25,177	100.00%		



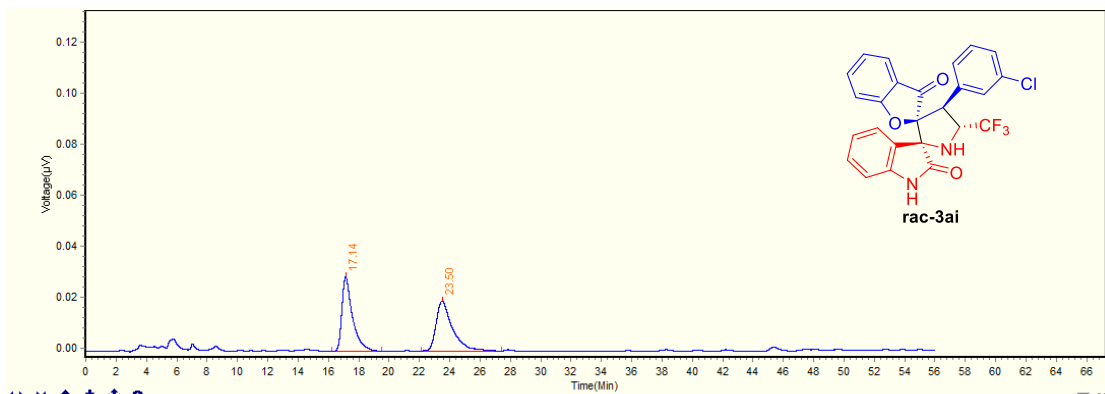
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	21.50	503408	9303	4.93%	1.929	BB
2	43.80	9702445	62277	95.07%	11.944	BB
total		10,205,853	71,580	100.00%		



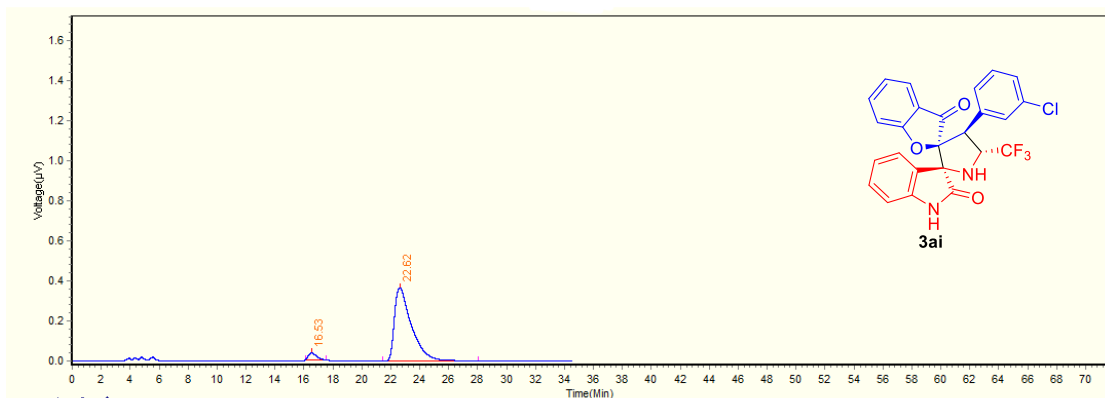
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	11.32	1087587	28148	49.59%	4.101 BB	
2	17.24	1105736	18885	50.41%	6.12 BB	
Total		2,193,323	47,033	100.00%		



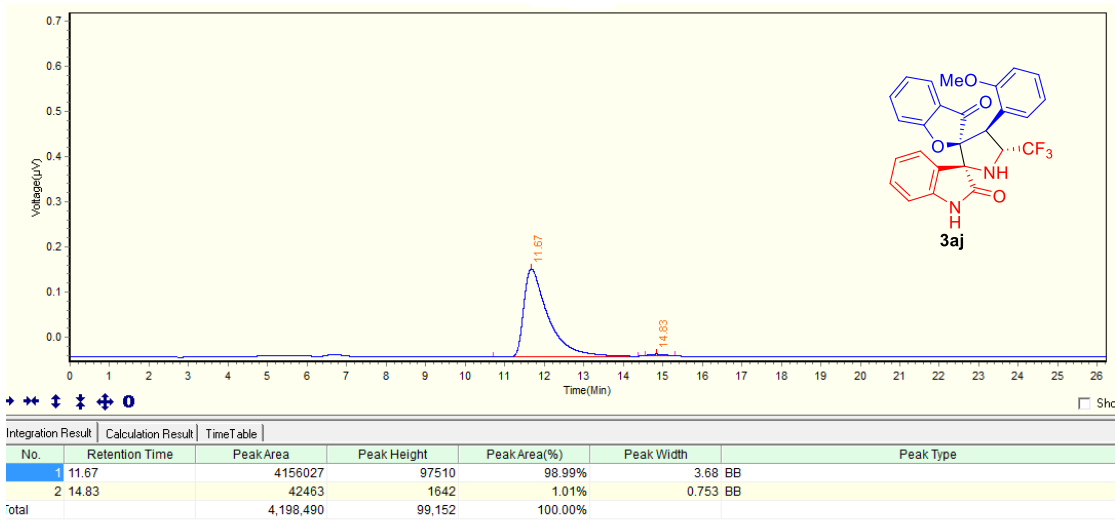
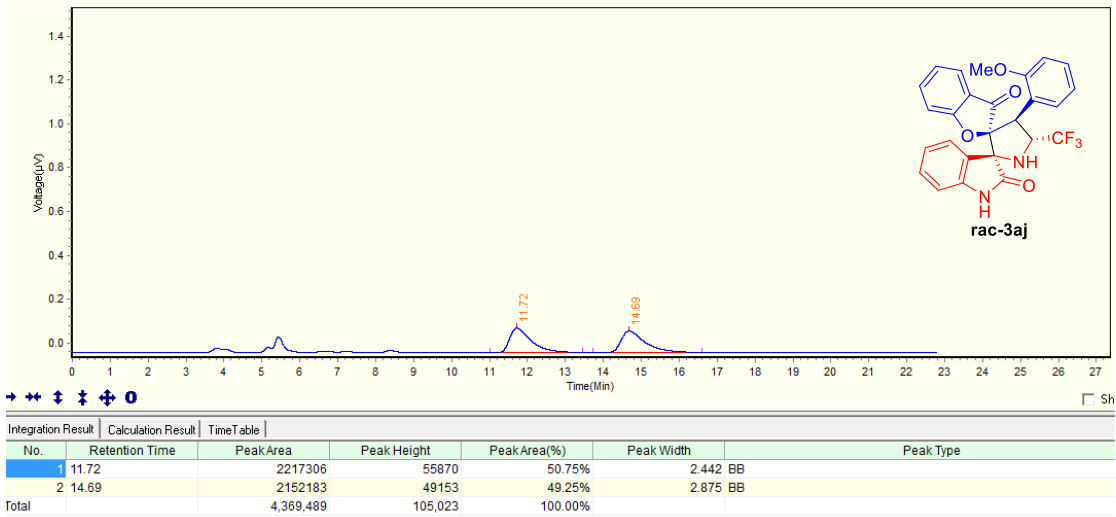
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	11.31	420480	15916	2.31%	0.977 BB	
2	16.60	17777717	293820	97.69%	7.259 BB	
Total		18,198,197	309,736	100.00%		

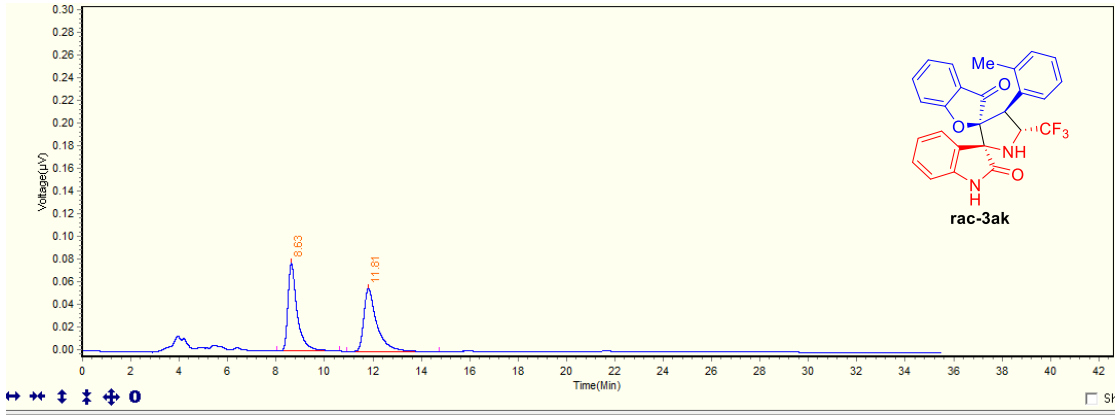


No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	17.14	704126	14546	49.17%	3.296	BB
2	23.50	727951	9820	50.83%	5.296	BB
Total		1,432,077	24,366	100.00%		

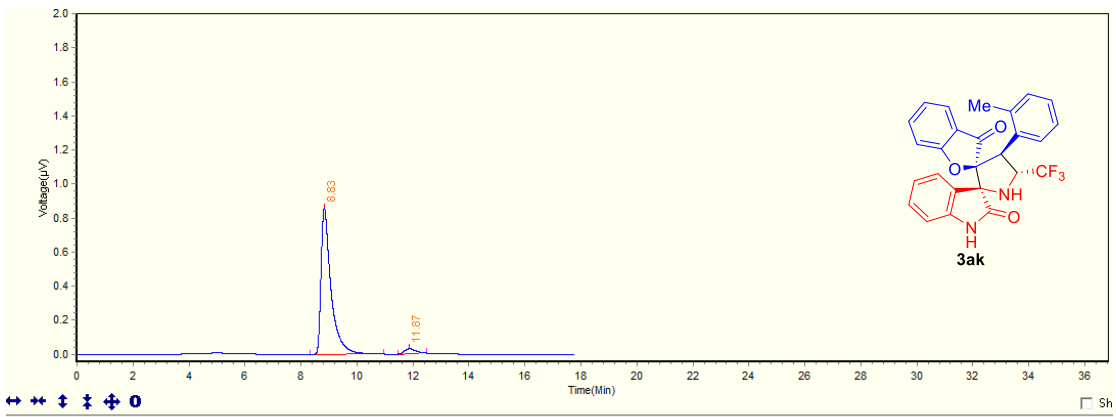


No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	16.53	683844	17537	4.39%	1.42	BB
2	22.62	14903947	183459	95.61%	6.603	BB
Total		15,587,791	200,996	100.00%		

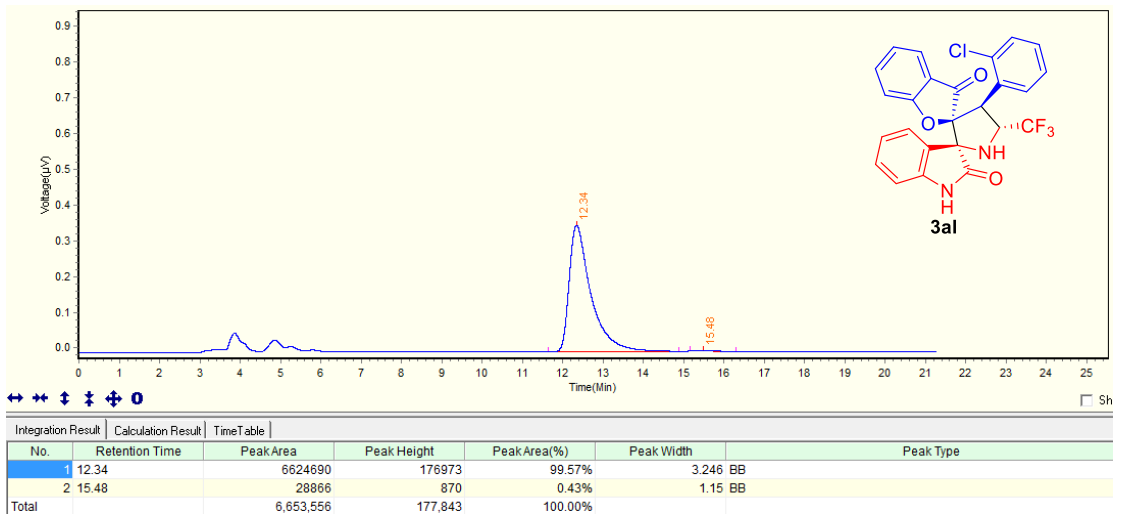
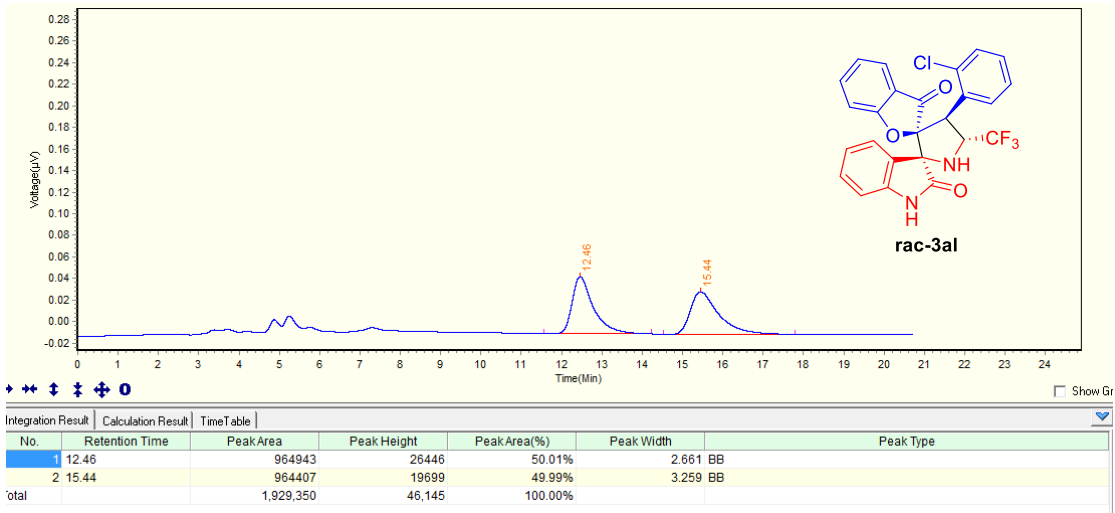


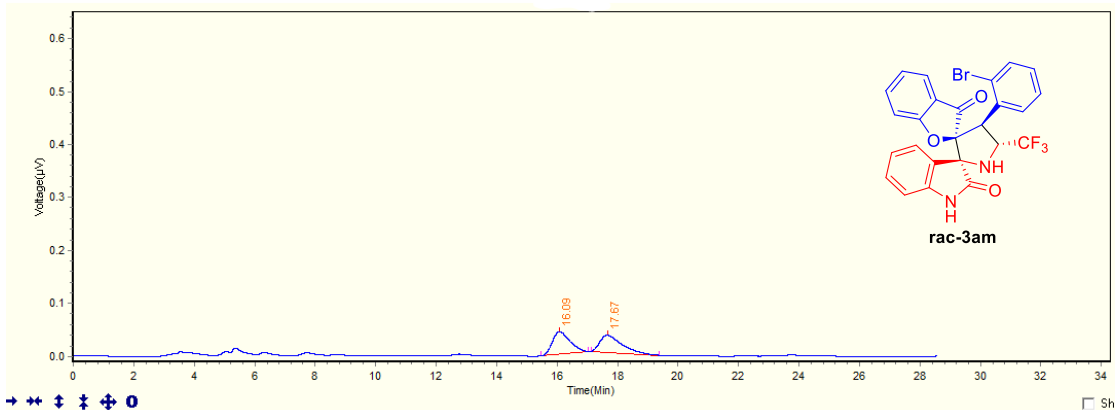


No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	8.63	1028969	38571	49.94%	2.591	BB
2	11.81	1031303	27697	50.06%	3.823	BB
Total		2,060,272	66,268	100.00%		

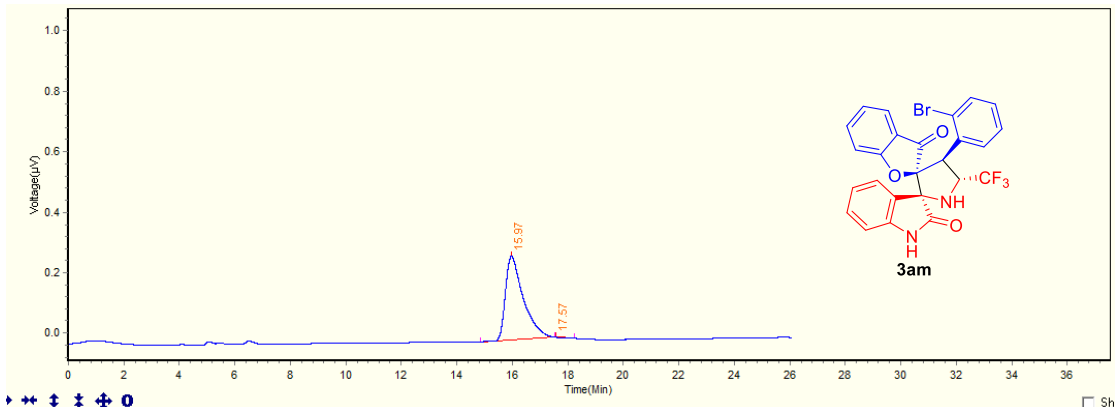


No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	8.83	10610797	429251	96.42%	2.629	BB
2	11.87	393595	14676	3.58%	1.023	BB
Total		11,004,392	443,927	100.00%		

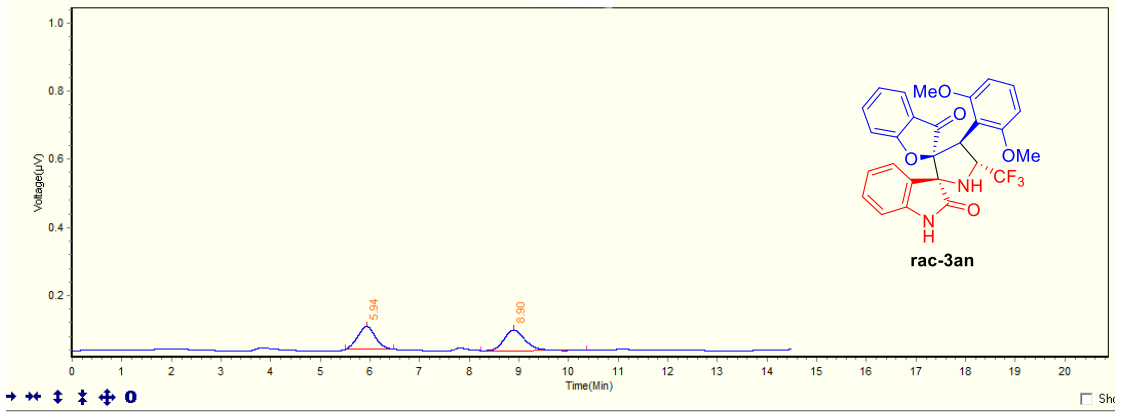




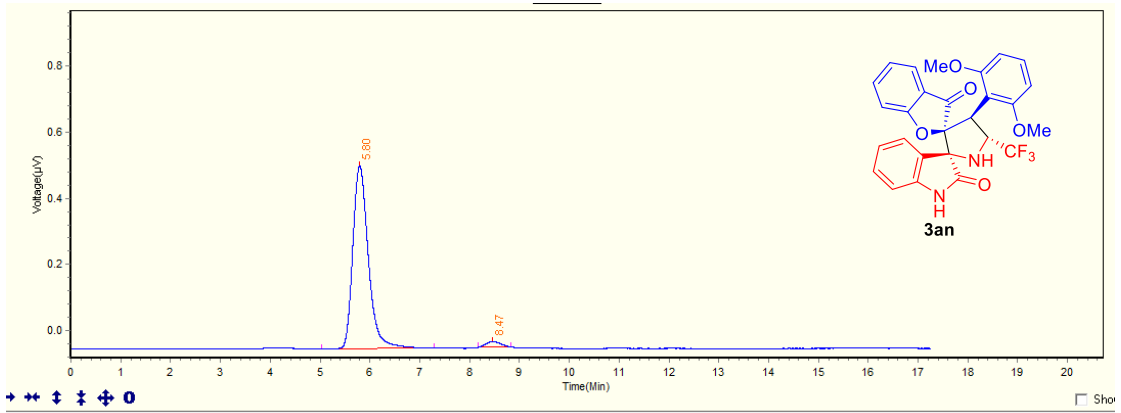
No.	Retention Time	PeakArea	Peak Height	PeakArea(%)	Peak Width	Peak Type
1	16.09	860949	21445	51.30%	1.563	BB
2	17.67	817347	16106	48.70%	2.243	BB
Total		1,678,296	37,551	100.00%		



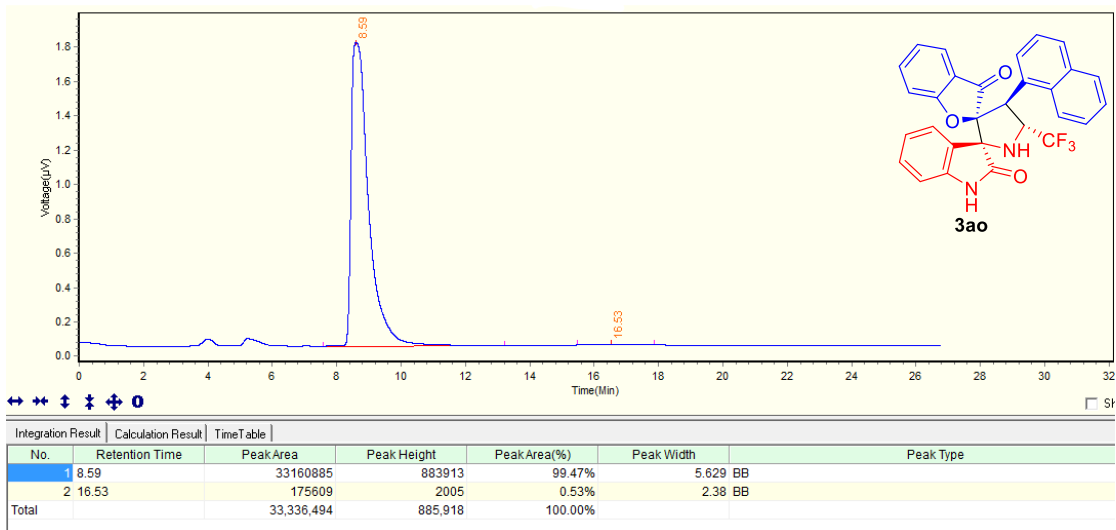
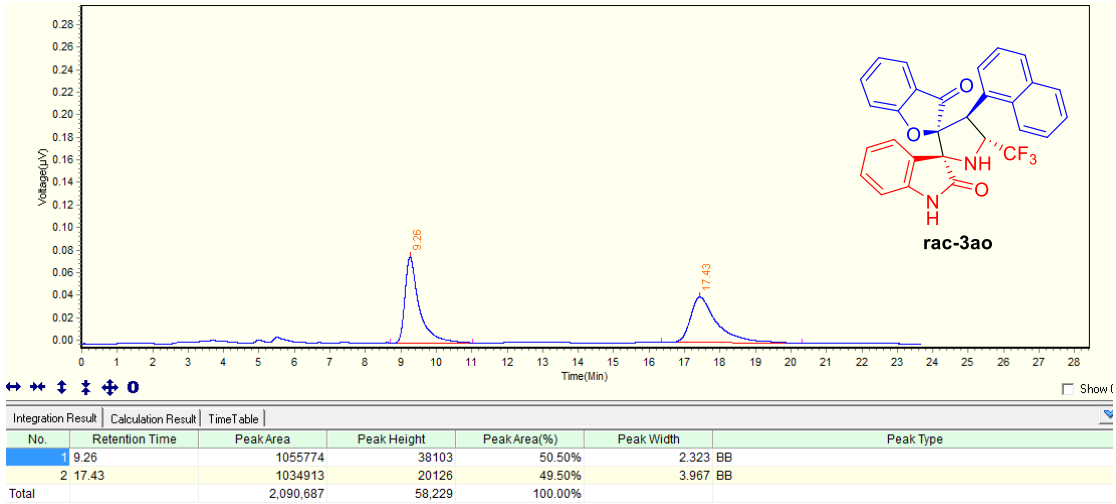
No.	Retention Time	PeakArea	Peak Height	PeakArea(%)	Peak Width	Peak Type
1	15.97	6152587	137980	99.86%	2.688	BB
2	17.57	8709	-2	0.14%	0.668	BB
total		6,161,296	137,978	100.00%		

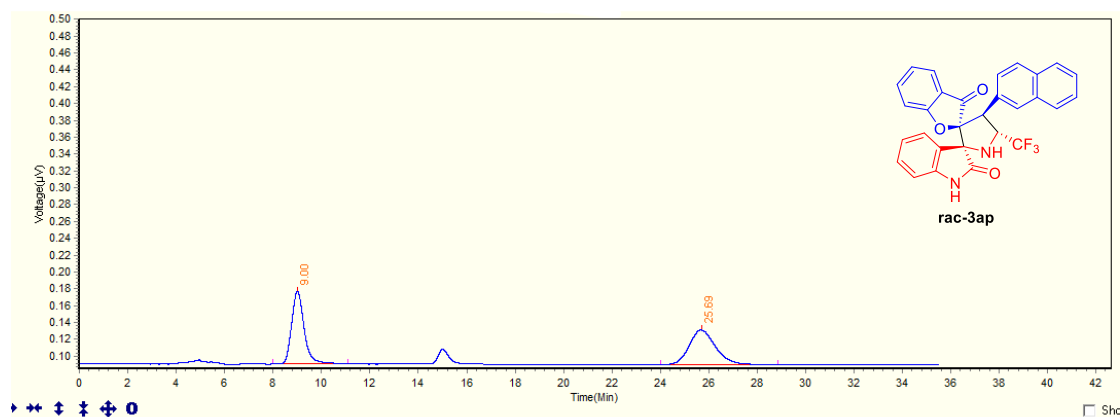


No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	5.94	821094	33632	48.79%	0.972	BB
2	8.90	861825	30016	51.21%	2.131	BB
Total		1,682,919	63,648	100.00%		

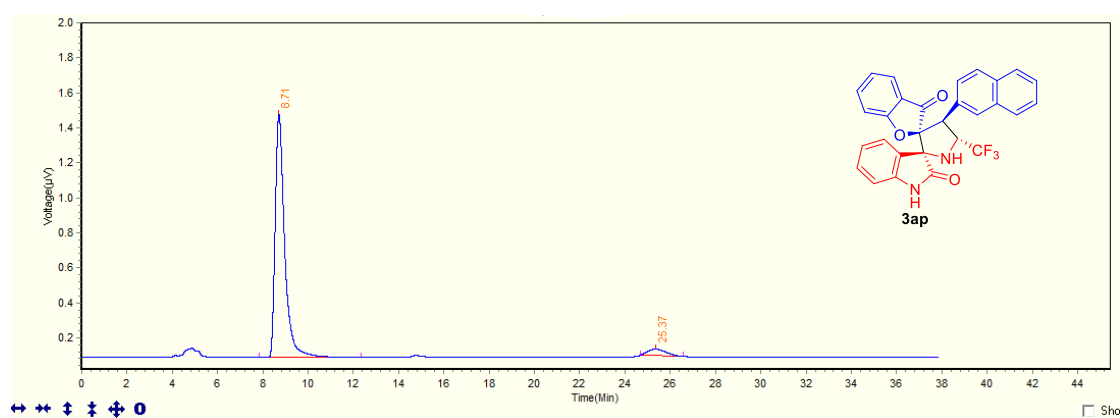


No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	5.80	5969847	275374	97.16%	2.258	BB
2	8.47	174327	8106	2.84%	0.657	BB
Total		6,144,174	283,480	100.00%		

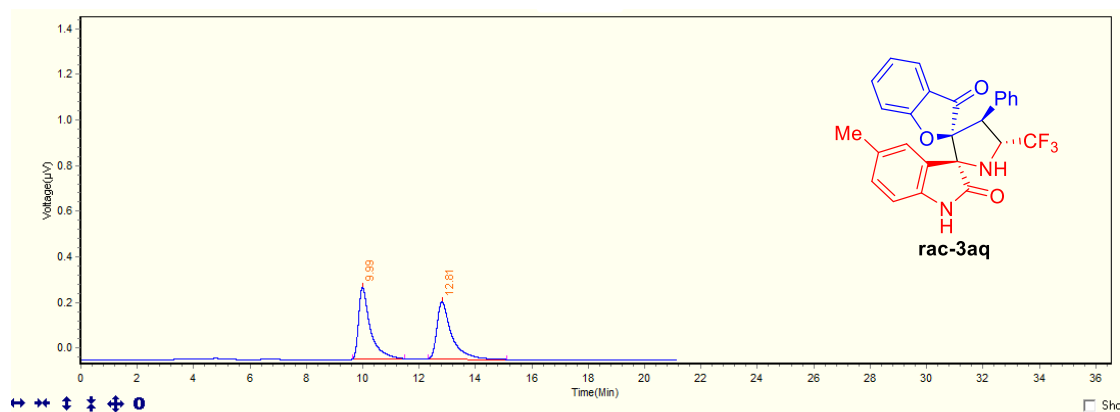




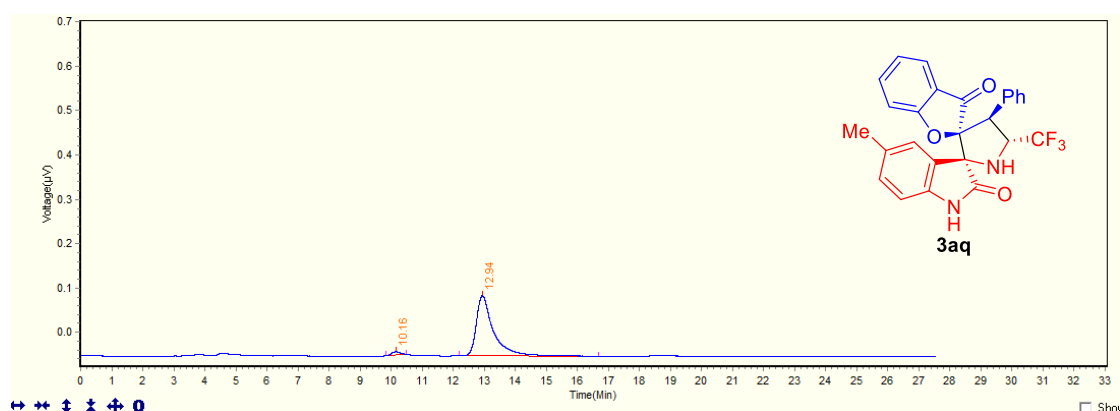
No.	Retention Time	PeakArea	Peak Height	PeakArea(%)	Peak Width	Peak Type
1	9.00	1549434	43262	50.12%	3.102 BB	
2	25.69	1542255	20493	49.88%	4.843 BB	
total		3,091,689	63,755	100.00%		



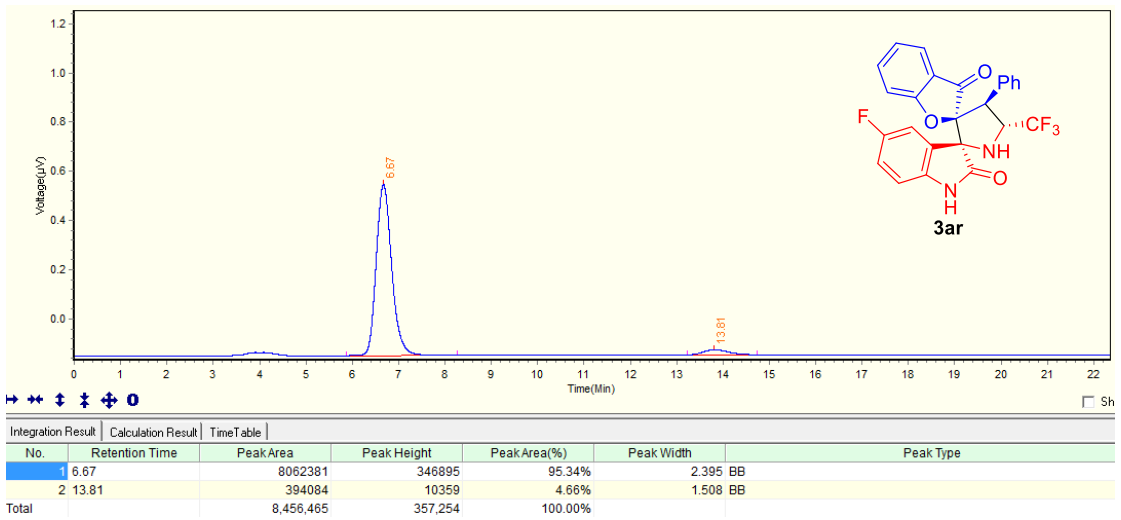
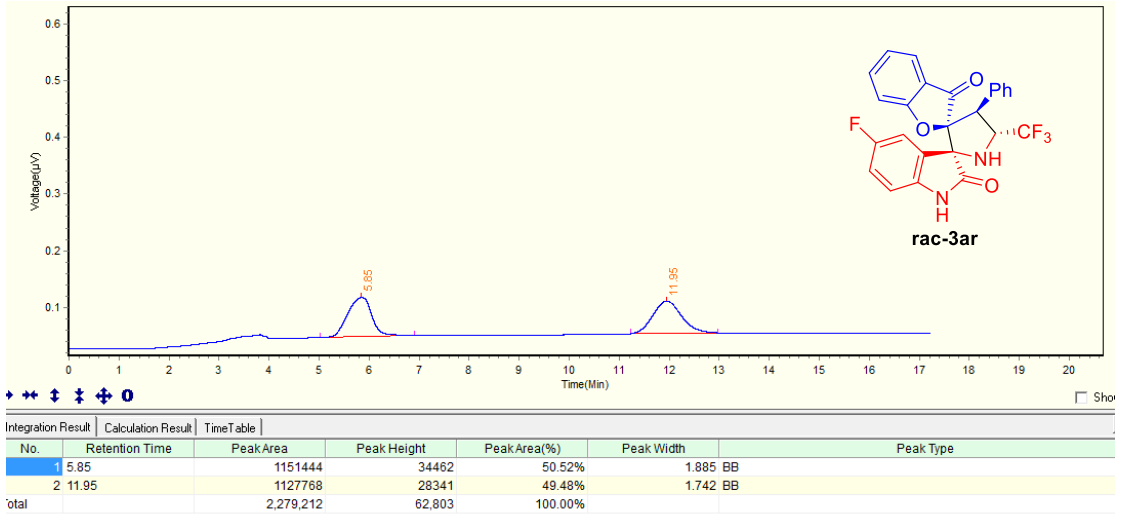
No.	Retention Time	PeakArea	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	8.71	20020734	692900	95.04%	4.503 BB	
2	25.37	1045318	18528	4.96%	1.892 BB	
Total		21,066,052	711,428	100.00%		

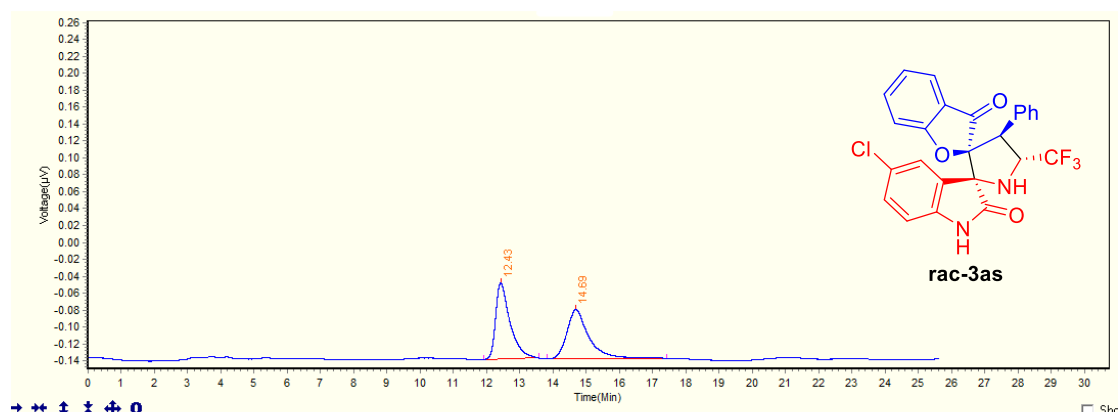


No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	9.99	4486388	157791	49.71%	1.848	BB
2	12.81	4538976	125798	50.29%	2.789	BB
Total		9,025,364	283,589	100.00%		

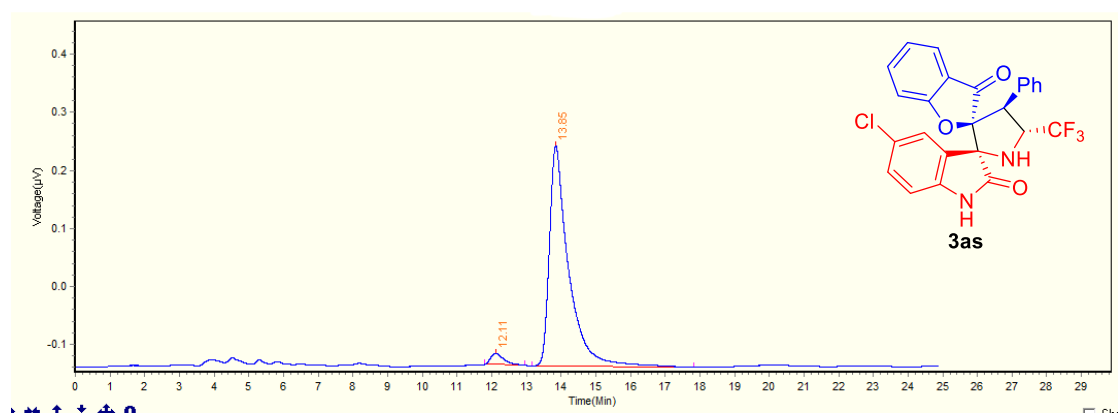


No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	10.16	73282	3616	2.75%	0.648	BB
2	12.94	2589126	67772	97.25%	4.473	BB
Total		2,662,408	71,388	100.00%		

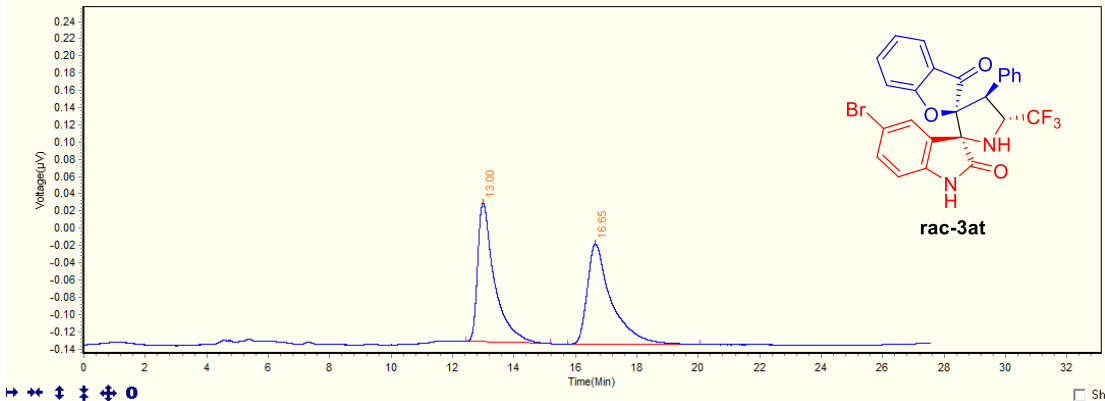




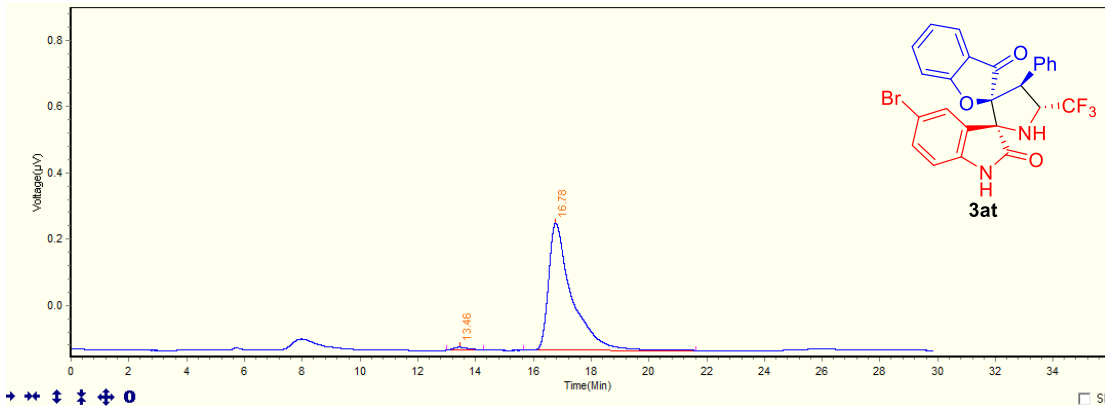
Integration Result		Calculation Result		TimeTable		
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	12.43	1350842	45048	49.77%	1.652	BB
2	14.89	1363138	29175	50.23%	3.613	BB
Total		2,713,980	74,223	100.00%		



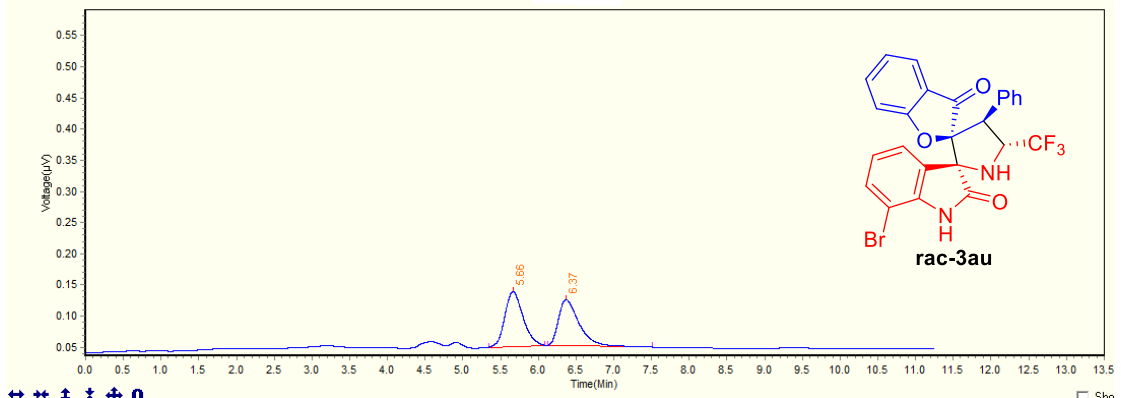
Integration Result		Calculation Result		TimeTable		
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	12.11	257205	9608	3.40%	1.136	BB
2	13.85	7308754	189928	96.60%	4.664	BB
Total		7,565,959	199,536	100.00%		



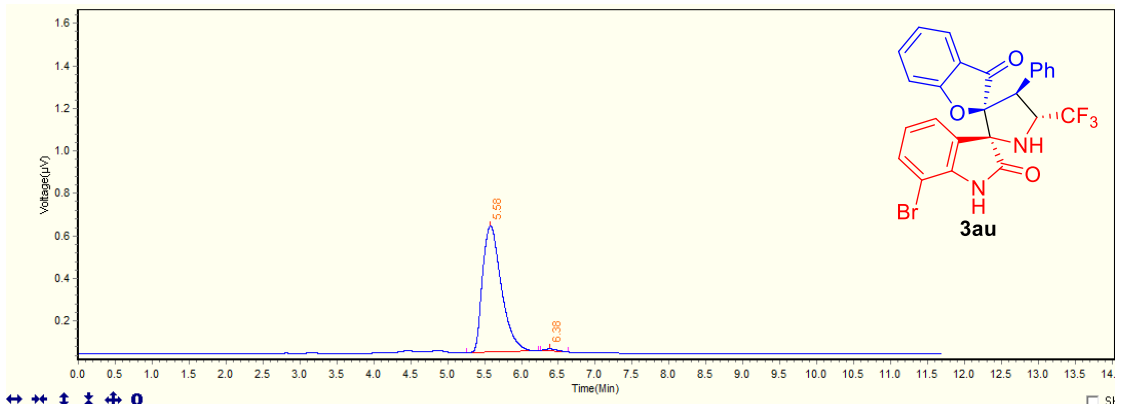
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	13.00	3076660	80491	49.23%	2.751	BB
2	16.65	3172462	58224	50.77%	4.309	BB
Total		6,249,122	138,715	100.00%		



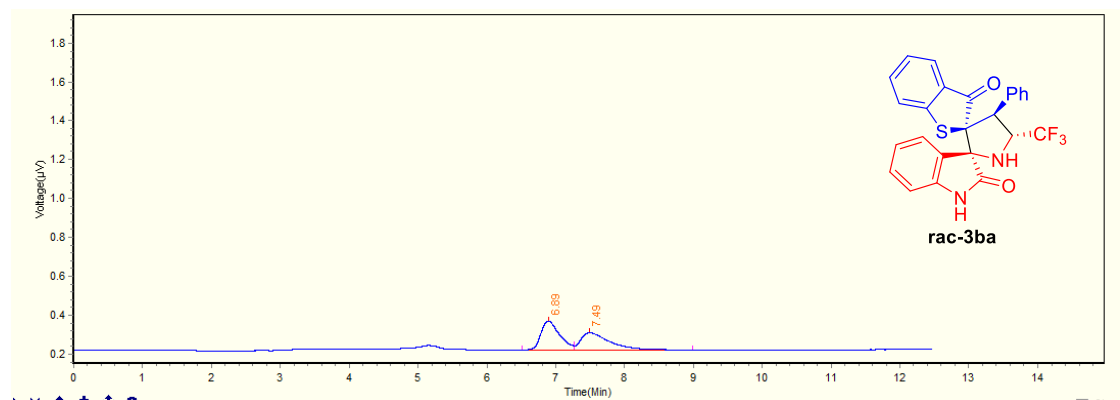
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	13.46	152193	4311	1.41%	1.248	BB
2	16.78	10671598	191794	98.59%	5.99	BB
Total		10,823,791	196,105	100.00%		



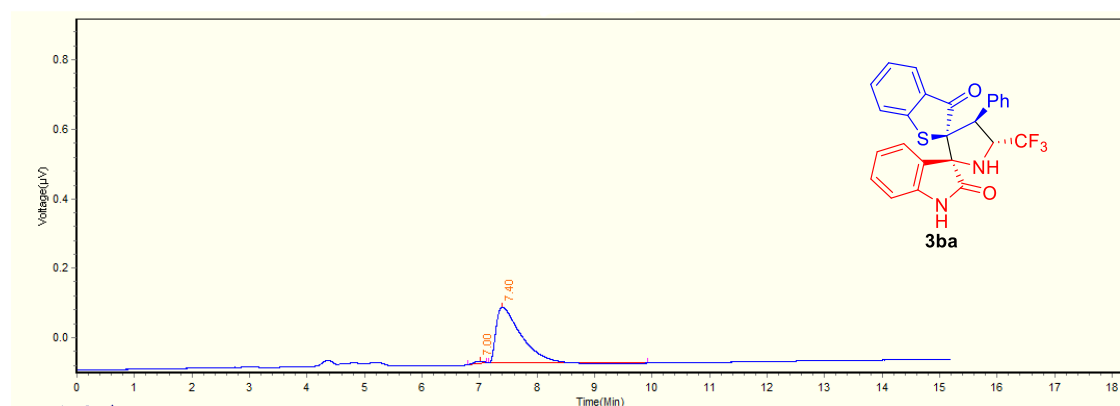
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	5.66	732465	43799	51.09%	0.749	BB
2	6.37	701233	36977	48.91%	1.387	BB
Total		1,433,698	80,776	100.00%		



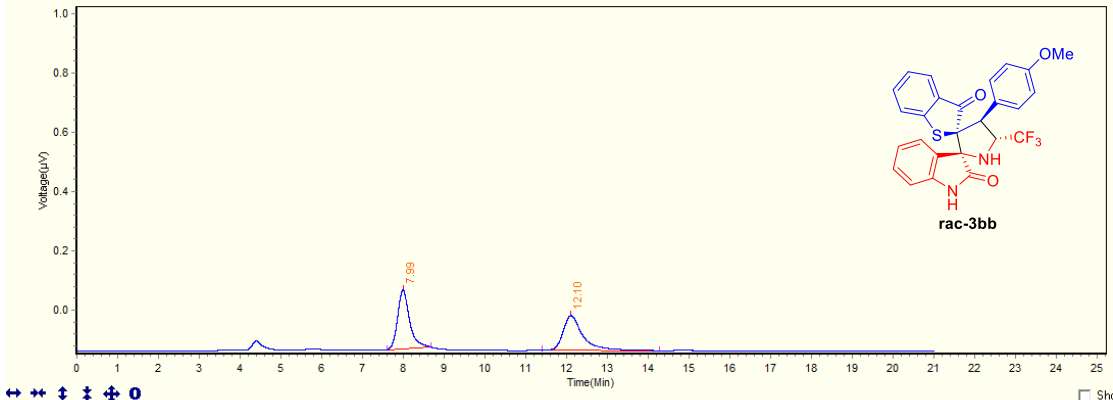
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	5.58	5272558	298098	98.92%	0.973	BB
2	6.38	57603	4744	1.08%	0.376	BB
Total		5,330,061	302,842	100.00%		



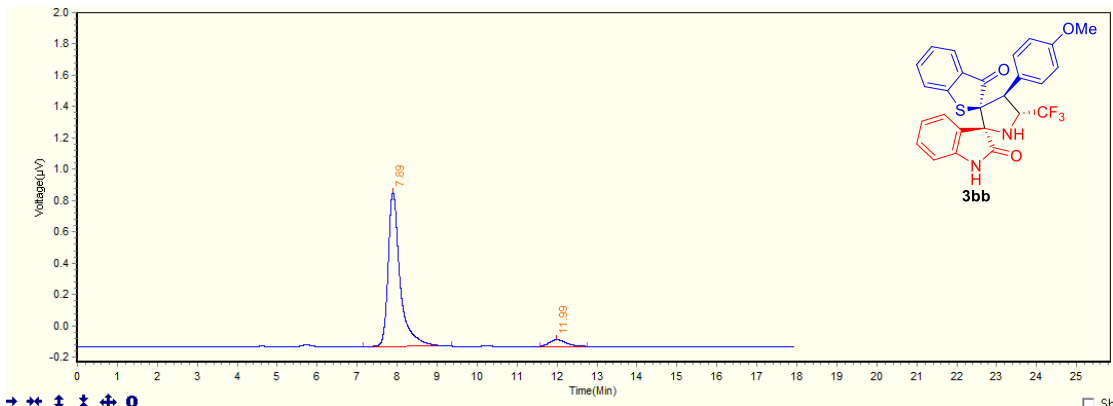
No.	Retention Time	PeakArea	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	6.89	1379269	73948	51.79%	0.757 VB	
2	7.49	1283950	43969	48.21%	1.719 VB	
Total		2,663,219	117,917	100.00%		



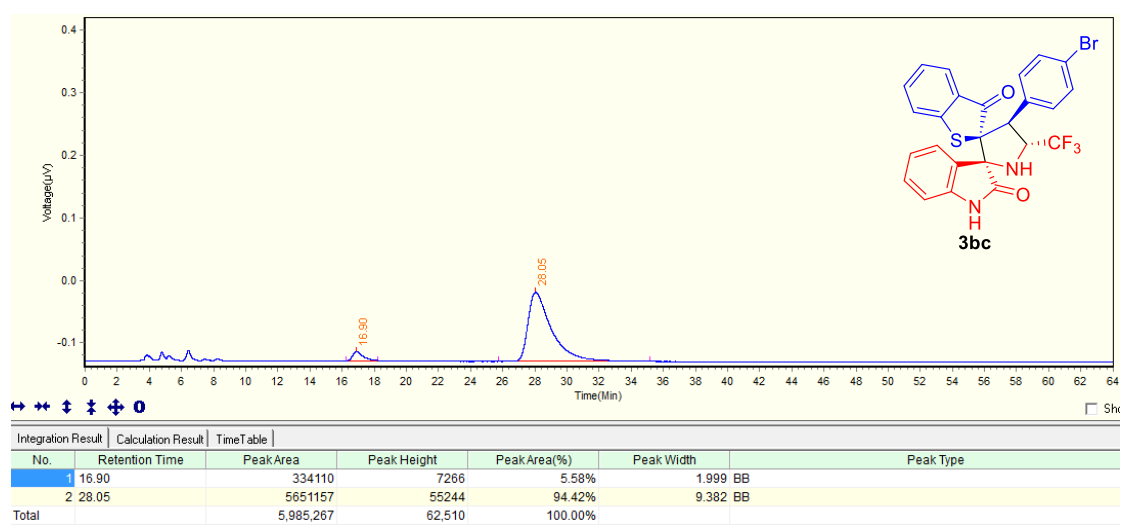
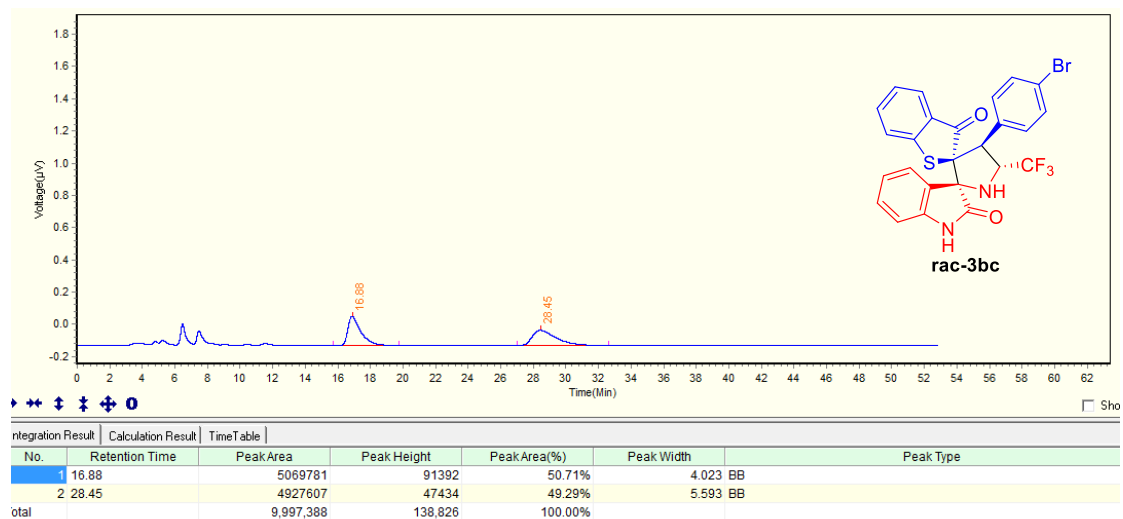
No.	Retention Time	PeakArea	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	7.00	31350	2780	1.31%	0.326 BB	
2	7.40	2366652	79366	98.69%	2.765 BB	
Total		2,398,002	82,146	100.00%		

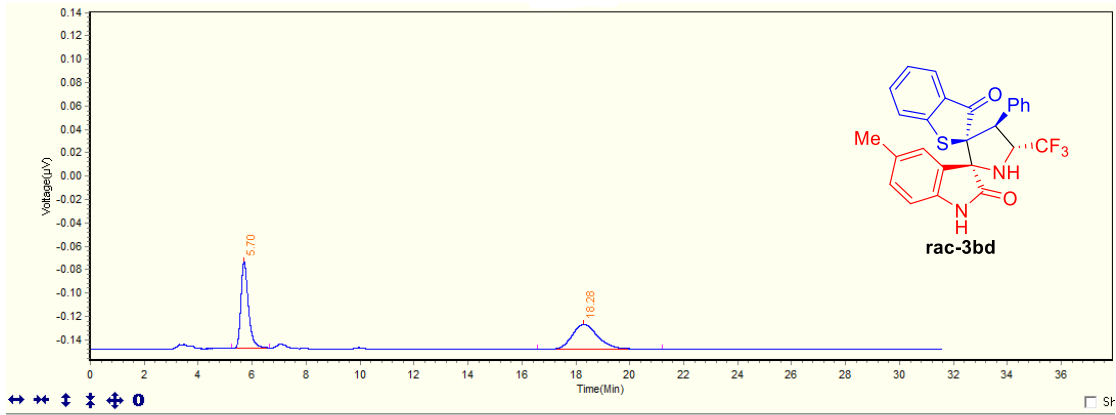


No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	7.99	2015166	99444	50.28%	1.078	BB
2	12.10	1992853	58791	49.72%	2.885	BB
Total		4,008,019	158,235	100.00%		

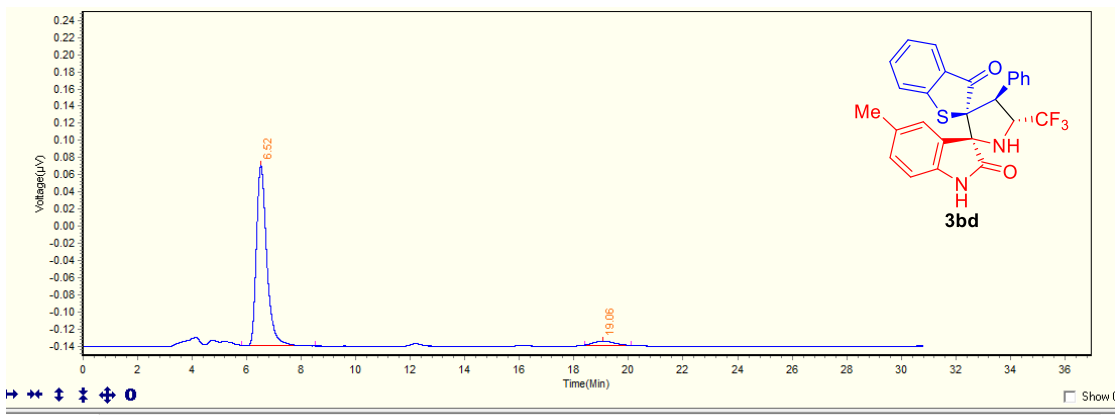


No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	7.89	10322129	490894	94.21%	2.222	BB
2	11.99	634773	21934	5.79%	1.183	BB
Total		10,956,902	512,828	100.00%		

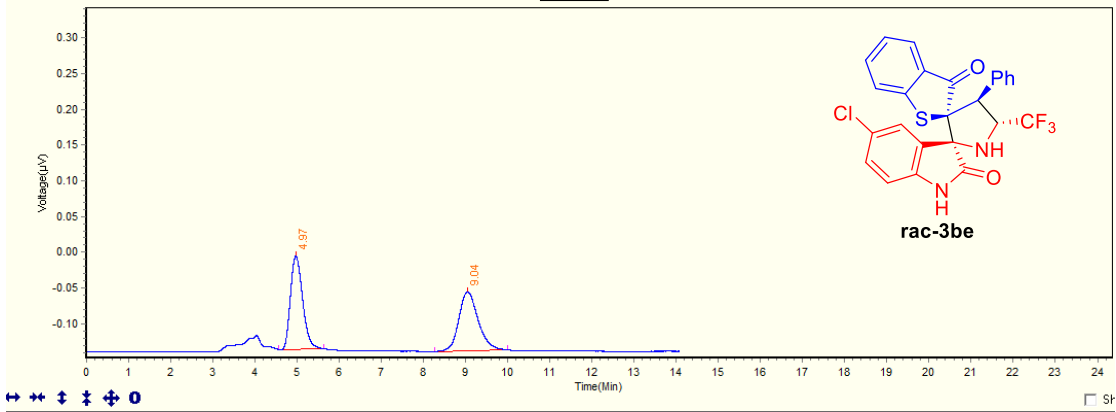




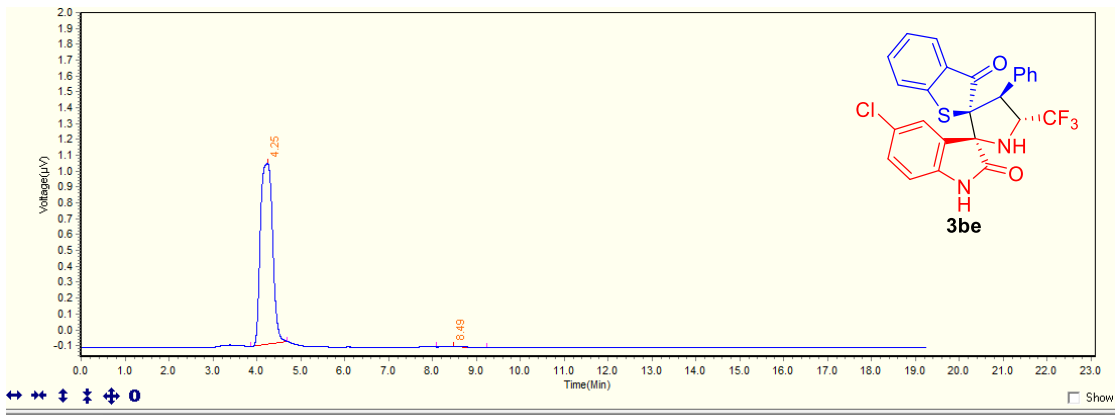
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	5.70	703522	36760	49.65%	1.391	BB
2	18.28	713513	10593	50.35%	4.647	BB
Total		1,417,035	47,353	100.00%		



No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	6.52	2750831	104710	95.67%	2.718	BB
2	19.06	124599	2402	4.33%	1.705	BB
Total		2,875,430	107,112	100.00%		



No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	4.97	1304529	64991	49.88%	1.073	BB
2	9.04	1310767	41215	50.12%	1.731	BB
Total		2,615,296	106,206	100.00%		



No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	Peak Type
1	4.25	10972180	571874	99.38%	0.837	BB
2	8.49	68200	2943	0.62%	1.147	BB
Total		11,040,380	574,817	100.00%		

