

Base-regulated tunable synthesis of pyridobenzoxazepinones and pyridobenzoxazines

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General considerations

Nuclear Magnetic Resonance spectra were recorded on Bruker Avance 300 and Bruker ARX 400 spectrometers. All ^1H NMR experiments were reported in δ units, parts per million (ppm), and were measured relative to residual chloroform (7.26 ppm) or DMSO (2.5 ppm) in the deuterated solvent. All ^{13}C NMR spectra were reported in ppm relative to deuteriochloroform (77.0 ppm) or $[\text{D}_6]\text{DMSO}$ (39.5 ppm) and all were obtained with ^1H decoupling. All coupling constants J were reported in Hz. The following abbreviations were used to describe peak splitting patterns when appropriate: s = singlet, d = doublet, t = triplet, dd = doublet of doublet, m = multiplet and br s = broad singlet. All measurements were carried out at room temperature unless otherwise stated. Electron impact (EI) mass spectra were recorded on AMD 402 mass spectrometer (70 eV). High resolution mass spectra (HRMS) were recorded on Agilent 6210. The data were given as mass units per charge (m/z). Gas chromatography analysis was performed on an Agilent HP-5890 instrument with a FID detector and HP-5 capillary column (polydimethylsiloxane with 5 % phenyl groups, 30 m, 0.32 mm i.d., 0.25 μm film thickness) using argon as carrier gas. The products were isolated from the reaction mixture by column chromatography on silica gel 60, 0.063-0.2 mm, 70-230 mesh (Merck).

Materials

DMSO (99.8 %), NMP (99.5%), DMF (anhydrous, 99.8%), DMA (anhydrous, 99.8 %) was purchased from Sigma-Aldrich or Acros and used without further purification. All Chemicals were commercial available and were used without further purification unless otherwise noted.

Representative procedure for the synthesis of pyridobenzoxazepinones and pyridobenzoxazines

A Wheaton vial (4 mL) equipped with a septum, a small cannula was charged with $\text{Pd}(\text{OAc})_2$ (2 mol%), BuPAD_2 (6 mol%), 3-bromo-2-chloropyridine (0.50 mmol), 2-aminophenol (0.50 mmol), sodium carbonate (1.5 mmol) and a magnetic stirring bar. The vial was purged with argon before DMSO (2.0 mL) was injected by using a syringe. The vial (or several vials) was placed in an alloy plate, which was transferred into a 300 mL autoclave of the 4560 series from Parr Instruments under argon atmosphere. After flushing the autoclave three times with CO , a pressure of 10 bars CO was adjusted at ambient temperature. Afterwards the reaction was performed for 12 h at 120°C . After the reaction was completed, the autoclave was cooled down with ice-water mixture to room temperature and the pressure was released carefully. The solution was diluted with acetone and then silica gel was added into the solution. After evaporation of the organic solvent, the crude product was purified by column chromatography using ethyl acetate/*n*-pentane.

Representative procedure for the synthesis of pyridobenzoxazines

To an oven-dried 10 mL Schlenk tube was added 3-bromo-2-chloropyridine (0.50 mmol), 2-aminophenol (0.50 mmol), cesium carbonate (1.0 mmol) and a magnetic stirring bar. The Schlenk tube was purged with argon before DMSO (2.0 mL) was injected by using a syringe. Then the reaction was performed for 24 h at 120°C . After the reaction was completed, the reaction mixture was diluted with acetone and then silica gel was added into the solution. After evaporation of the organic solvent, the crude product was purified by column chromatography using ethyl acetate/*n*-pentane.

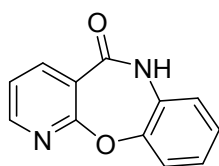
The procedure of the two-step reaction for the mechanism investigation

A Wheaton vial (4 mL) equipped with a septum, a small cannula was charged with 3-bromo-2-chloropyridine (0.50 mmol), 2-aminophenol (0.50 mmol), sodium carbonate (1.0 mmol), hexadecane (100 μL , internal standard) and a magnetic stirring bar. The vial was purged with argon before DMSO (2.0 mL) was injected by using a syringe. The vial was placed in an alloy plate, which was transferred into a 300 mL autoclave of the 4560 series from Parr Instruments under argon atmosphere. After flushing the autoclave three times with N_2 , a pressure of 3 bars N_2 was adjusted at ambient temperature. Afterwards the first-step reaction was performed for 12 h at 120°C . After completion of the first-step reaction, the autoclave was

cooled down with ice-water mixture to room temperature and the pressure was released carefully. 150 μ L reaction mixture was transferred out of the vial for GC and GC-MS analysis. And then Pd(OAc)₂ (2 mol%), BuPAD₂ (6 mol%), sodium carbonate (0.5 mmol) was added into the reaction mixture under the argon. The vial (or several vials) was placed in an alloy plate, which was transferred into a 300 mL autoclave of the 4560 series from Parr Instruments under argon atmosphere. After flushing the autoclave three times with CO, a pressure of 10 bars CO was adjusted at ambient temperature. Afterwards the second-step reaction was performed for 12 h at 120°C. After the reaction was completed, the autoclave was cooled down with ice-water mixture to room temperature and the pressure was released carefully. The solution was diluted with acetone and then silica gel was added into the solution. After evaporation of the organic solvent, the crude product was purified by column chromatography using ethyl acetate/*n*-pentane.

Analytic data

Benzo[*b*]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one^[1]



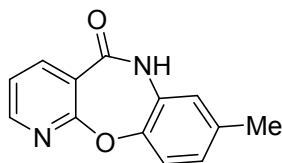
White solid; ¹H NMR (300 MHz, [D₆]DMSO): δ = 10.76 (br s, 1H), 8.51 (dd, *J* = 1.8, 4.7 Hz, 1H), 8.27 (dd, *J* = 1.8, 7.5 Hz, 1H), 7.46 (dd, *J* = 4.8, 7.7 Hz, 1H), 7.34 (dd, *J* = 1.8, 7.4 Hz, 1H), 7.26-7.14 (m, 3H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 164.5, 162.3, 152.4, 148.1, 142.4, 130.5, 126.4, 125.6, 122.5, 121.9, 121.7, 120.0.

GC-MS (EI, 70 eV): *m/z* (%) = 212 (100), 184 (36), 155 (41), 130 (9), 77 (11), 52 (11).

HRMS (EI): calcd. for [C₁₂H₈O₂N₂]⁺ 212.05803, found 212.05831.

8-Methylbenzo[*b*]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



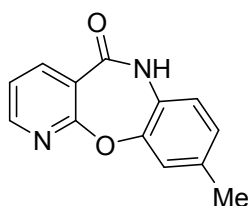
White solid; ¹H NMR (300 MHz, [D₆]DMSO): δ = 10.69 (br s, 1H), 8.49 (dd, *J* = 1.8, 4.8 Hz, 1H), 8.26 (dd, *J* = 2.1, 7.7 Hz, 1H), 7.45 (dd, *J* = 4.8, 7.8 Hz, 1H), 7.21 (d, *J* = 9.0 Hz, 1H), 6.98 (s, 1H), 6.98-6.65 (m, 1H), 2.25 (s, 3H)

¹³C NMR (100MHz, [D₆]DMSO): δ = 164.5, 162.5, 152.3, 146.0, 142.3, 135.7, 130.1, 126.0, 122.4, 121.8, 121.5, 120.1, 20.3.

GC-MS (EI, 70 eV): *m/z* (%) = 226 (100), 211 (11), 197 (64), 183 (7), 169 (24), 155 (12), 77 (14), 51 (8).

HRMS (EI): calcd. for [C₁₃H₁₀O₂N₂]⁺ 226.07368, found 226.07358.

9-Methylbenzo[*b*]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one^[1a]



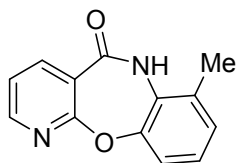
Pale yellow solid; ¹H NMR (300 MHz, [D₆]DMSO): δ = 10.64 (br s, 1H), 8.49 (dd, *J* = 2.1, 4.5 Hz, 1H), 8.25 (dd, *J* = 2.1, 7.5 Hz, 1H), 7.45 (dd, *J* = 4.8, 7.8 Hz, 1H), 7.16 (s, 1H), 7.08-7.00 (m, 2H), 2.26 (s, 3H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 164.9, 162.9, 152.8, 148.5, 142.8, 135.9, 128.3, 127.3, 123.0, 122.6, 122.0, 120.6, 20.6.

GC-MS (EI, 70 eV): *m/z* (%) = 226 (100), 211 (5), 197 (79), 169 (41), 77 (44), 65 (24), 50 (41), 39 (30).

HRMS (EI): calcd. for [C₁₃H₁₀O₂N₂]⁺ 226.07368, found 226.07395.

7-Methylbenzo[*b*]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



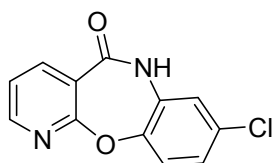
Pale yellow solid; ¹H NMR (300 MHz, [D₆]DMSO): δ = 10.17 (br s, 1H), 8.48 (dd, *J* = 2.1, 4.8 Hz, 1H), 8.22 (dd, *J* = 2.1, 7.7 Hz, 1H), 7.46 (dd, *J* = 4.8, 7.8 Hz, 1H), 7.21-7.16 (m, 1H), 7.11-7.08 (m, 2H), 2.34 (s, 3H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 165.3, 163.4, 152.6, 150.8, 142.6, 132.2, 129.4, 128.4, 126.2, 123.1, 121.1, 119.9, 18.3 (d, *J* = 3.0 Hz).

GC-MS (EI, 70 eV): *m/z* (%) = 226 (100), 211 (14), 197 (70), 183 (19), 169 (49), 77 (67), 65 (41), 50 (48), 39 (46).

HRMS (EI): calcd. for [C₁₃H₁₀O₂N₂]⁺ 226.07368, found 226.07401.

8-Chlorobenzo[*b*]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one^[1b]



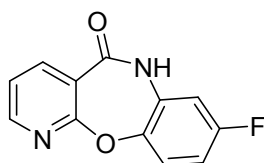
White solid; ¹H NMR (300 MHz, [D₆]DMSO): δ = 10.83 (br s, 1H), 8.52 (dd, *J* = 2.1, 4.8 Hz, 1H), 8.28 (dd, *J* = 1.8, 7.7 Hz, 1H), 7.49 (dd, *J* = 4.8, 7.8 Hz, 1H), 7.40-7.36 (m, 1H), 7.23-7.20 (m, 2H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 164.3, 161.8, 152.6, 146.7, 142.5, 132.9, 129.9, 125.1, 123.5, 122.8, 121.0, 119.7.

GC-MS (EI, 70 eV): m/z (%) = 246 (100), 218 (28), 211 (65), 183 (43), 155 (85), 77 (76), 63 (46), 50 (78), 38 (27).

HRMS (EI): calcd. for $[\text{C}_{12}\text{H}_7\text{O}_2\text{N}_2\text{Cl}]^+$ 246.01906, found 246.01951; calcd. for $[\text{C}_{12}\text{H}_7\text{O}_2\text{N}_2^{31}\text{Cl}]^+$ 248.01676.

8-Fluorobenzo[b]pyrido[3,2-f][1,4]oxazepin-5(6H)-one



Pale pink solid; **¹H NMR (300 MHz, [D₆]DMSO):** δ = 10.83 (br s, 1H), 8.52 (dd, *J* = 2.1, 4.8 Hz, 1H), 8.27 (dd, *J* = 2.1, 7.5 Hz, 1H), 7.48 (dd, *J* = 4.8, 7.5 Hz, 1H), 7.42-7.37 (m, 1H), 7.04-6.99 (m, 2H);

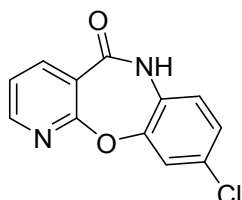
¹⁹F NMR (282 MHz, [D₆]DMSO): δ = -115.46 (m);

¹³C NMR (100 MHz, [D₆]DMSO): δ = 164.3, 162.1, 159.2 (d, *J* = 241 Hz), 152.6, 144.3 (d, *J* = 3.0 Hz), 142.5, 132.0 (d, *J* = 12.0 Hz), 123.3 (d, *J* = 9.0 Hz), 122.7, 119.8, 111.8 (d, *J* = 23.0 Hz), 108.1 (d, *J* = 27.0 Hz).

GC-MS (EI, 70 eV): m/z (%) = 230 (100), 202 (35), 173 (66), 147 (17), 77 (44), 50 (50).

HRMS (EI): calcd. for $[\text{C}_{12}\text{H}_7\text{O}_2\text{N}_2\text{F}]^+$ 230.04861, found 230.04875.

9-Chlorobenzo[b]pyrido[3,2-f][1,4]oxazepin-5(6H)-one



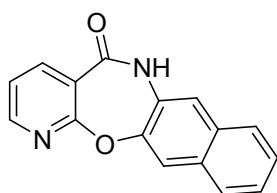
White solid; **¹H NMR (300 MHz, [D₆]DMSO):** δ = 10.83 (br s, 1H), 8.52 (dd, *J* = 2.1, 4.8 Hz, 1H), 8.28 (dd, *J* = 2.1, 7.5 Hz, 1H), 7.49 (dd, *J* = 4.8, 7.5 Hz, 1H), 7.46 (d, *J* = 2.4 Hz, 1H), 7.33 (dd, *J* = 2.4, 8.4 Hz, 1H), 7.20 (d, *J* = 8.7 Hz, 1H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 164.6, 162.2, 153.1, 148.8, 143.0, 130.3, 129.2, 127.0, 123.4, 122.4, 122.4, 120.3.

GC-MS (EI, 70 eV): m/z (%) = 246 (95), 211 (100), 183 (46), 155 (34), 77 (18).

HRMS (EI): calcd. for $[\text{C}_{12}\text{H}_7\text{O}_2\text{N}_2\text{Cl}]^+$ 246.01906 found 246.01935, calcd. for $[\text{C}_{12}\text{H}_7\text{O}_2\text{N}_2^{37}\text{Cl}]^+$ 248.01611, found 248.01628.

Naphtho[2,3-*b*]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



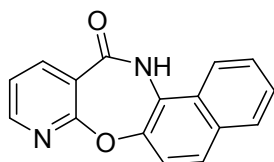
White solid; **¹H NMR (300 MHz, [D₆]DMSO)**: δ = 11.06 (br s, 1H), 8.54 (dd, *J* = 2.1, 4.8 Hz, 1H), (d, *J* = 2.1, 7.7 Hz, 1H), 7.95 (s, 1H), 7.93-7.84 (m, 2H), 7.67 (s, 1H), 7.51-7.45 (m, 3H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 165.1, 162.2, 153.0, 148.3, 142.8, 131.5, 131.1, 130.3, 127.7, 127.4, 126.9, 126.3, 123.2, 120.5, 119.4, 119.1.

GC-MS (EI, 70 eV): *m/z* (%) = 262 (100), 234 (37), 205 (52), 114 (13).

HRMS (EI): calcd. for [C₁₆H₁₀O₂N₂]⁺ 262.07368, found 262.07392.

Naphtho[2,1-*b*]pyrido[3,2-*f*][1,4]oxazepin-12(13*H*)-one



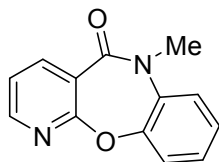
White solid; **¹H NMR (300 MHz, [D₆]DMSO)**: δ = 11.03 (br s, 1H), 8.51 (dd, *J* = 1.8, 4.8 Hz, 1H), 8.27 (dd, *J* = 2.1, 7.7 Hz, 1H), 8.22 (d, *J* = 8.4 Hz, 1H), 7.97 (d, *J* = 7.5 Hz, 1H), 7.84 (d, *J* = 8.7 Hz, 1H), 7.66-7.53 (m, 2H), 7.55 (d, *J* = 8.7 Hz, 1H), 7.47 (dd, *J* = 4.8, 7.5 Hz, 1H).

¹³C NMR (100 MHz, [D₆]DMSO): δ = 165.2, 163.5, 152.1, 146.8, 142.2, 131.5, 128.1, 126.8, 126.7, 126.1, 124.5, 122.7, 122.6, 121.0, 120.6.

GC-MS (EI, 70 eV): *m/z* (%) = 262 (100), 234 (41), 205 (59).

HRMS (EI): calcd. for [C₁₆H₁₀O₂N₂]⁺ 262.07368, found 262.07381.

6-Methylbenzo[*b*]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



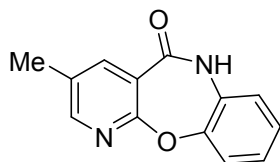
Solid white; **¹H NMR (300 MHz, [D₆]DMSO)**: δ = 8.47 (dd, *J* = 0.9, 4.1 Hz 1H), 8.25 (dd, *J* = 1.5, 7.8 Hz, 1H), 7.53-7.26 (m, 5H), 3.51 (s, 3H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 163.9, 163.2, 151.9, 150.5, 142.7, 134.8, 126.7, 126.6, 123.2, 122.6, 121.8, 120.2, 36.1.

GC-MS (EI, 70 eV): *m/z* (%) = 226 (100), 209 (9), 197 (24), 181 (23), 169 (31).

HRMS (EI): calcd. for $[C_{13}H_{10}O_2N_2]^+$ 226.07368, found 226.07387.

3-Methylbenzo[b]pyrido[3,2-f][1,4]oxazepin-5(6H)-one



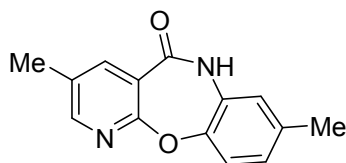
White solid; **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 10.69 (br s, 1H), 8.31 (s, 1H), 8.06 (s, 1H), 7.32 (d, J = 8.4 Hz, 1H), 7.22-7.13 (m, 3H), 2.32 (s, 3H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 165.1, 161.0, 152.6, 148.8, 142.7, 132.4, 131.0, 126.7, 126.0, 122.3, 122.2, 119.7, 17.3.

GC-MS (EI, 70 eV): m/z (%) = 226 (100), 198 (36), 169 (24).

HRMS (EI): calcd. for $[C_{13}H_{10}O_2N_2]$ 226.07368, found 226.07372.

3,8-Dimethylbenzo[b]pyrido[3,2-f][1,4]oxazepin-5(6H)-one



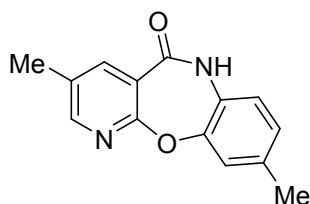
Pale yellow solid, **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 10.63 (br s, 1H), (dd, J = 0.9, 2.3 Hz, 1H), (dd, J = 0.9, 2.3 Hz, 1H), 7.19 (d, J = 7.8 Hz, 1H), 6.98-6.93 (m, 2H), 2.31 (s, 3H), 2.24 (s, 3H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 164.6, 160.7, 152.0, 146.2, 142.1, 135.6, 131.8, 130.1, 125.9, 121.8, 121.4, 119.2, 20.3, 16.8.

GC-MS (EI, 70 eV): m/z (%) = 240 (100), 225 (13), 211 (67), 183 (17).

HRMS (EI): calcd. for $[C_{14}H_{12}O_2N_2]^+$ 240.08933, found 240.08975.

3,9-Dimethylbenzo[b]pyrido[3,2-f][1,4]oxazepin-5(6H)-one



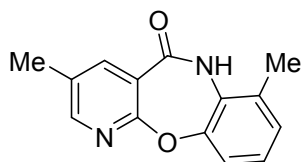
Pale yellow solid, **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 10.59 (br s, 1H), 8.29 (dd, J = 0.6, 2.6 Hz, 1H), 8.05 (dd, J = 0.6, Hz, 1H), 7.14 (s, 1H), 7.07-6.99 (m, 2H), 2.31 (s, 3H), 2.26 (s, 3H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 165.0, 161.1, 152.5, 148.7, 142.6, 135.8, 132.3, 128.3, 127.2, 122.5, 121.9, 119.8, 20.5, 17.3.

GC-MS (EI, 70 eV): m/z (%) = 240 (100), 211 (55), 197 (9), 183 (18).

HRMS (EI): calcd. for $[C_{14}H_{12}O_2N_2]^+$ 240.08933, found 240.08955.

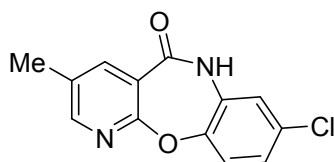
3,7-Dimethylbenzo[b]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



GC-MS (EI, 70 eV): m/z (%) = 240 (100), 223 (13), 211 (65), 196 (19), 183 (39), 77 (21), 65(22), 51 (29), 39 (42).

HRMS (EI): calcd. for $[C_{14}H_{12}O_2N_2]^+$ 240.08933, found 240.08958

8-Chloro-3-methylbenzo[b]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



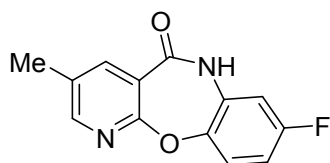
Pale yellow solid; **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 10.77 (br s, 1H), 8.32 (dd, J = 0.9, 2.7 Hz, 1H), 8.07 (d, J = 0.9, 2.6 Hz, 1H), 7.35 (dd, J = 1.2, 7.8 Hz, 1H), 7.22-7.18 (m, 2H), 2.31 (s, 3H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 164.9, 160.5, 152.8, 147.4, 142.8, 132.7, 132.5, 130.3, 125.6, 123.9, 121.5, 119.4, 17.3.

GC-MS (EI, 70 eV): m/z (%) = 260 (100), 231 (25), 225 (68), 197 (33), 169 (41).

HRMS (EI): calcd. for $[C_{13}H_9O_2Cl]^+$ 260.03471, found 260.03481; calcd. for $[C_{13}H_9O_2^{37}Cl]^+$ 262.03176, found 262.03243.

8-Fluoro-3-methylbenzo[b]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



White solid; **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 10.78 (br s, 1H), 8.32 (dd, J = 0.9, 2.4 Hz, 1H), 8.07 (dd, J = 0.9, 2.7 Hz, 1H), 7.38-7.33 (m, 1H), 7.03-6.97 (m, 2H), 2.32 (s, 3H).

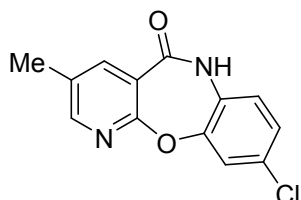
^{19}F NMR (282 MHz, $[D_6]DMSO$): δ = -115.6 (m).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 164.9, 159.6 (d, J = 240.8 Hz), 160.8, 152.8, 144.9 (d, J = 4.5 Hz), 142.7, 132.6, 132.4 (d, J = 11.3 Hz), 123.7 (d, J = 9.8 Hz), 119.5, 112.3 (d, J = 23.3 Hz), 108.6 (d, J = 27.0 Hz), 17.3.

GC-MS (EI, 70 eV): m/z (%) = 244 (100), 215 (34), 187 (25).

HRMS (EI): calcd. for $[C_{13}H_9O_2N_2F]^+$ 244.06426, found 244.06486.

9-Chloro-3-methylbenzo[b]pyrido[3,2-f][1,4]oxazepin-5(6H)-one



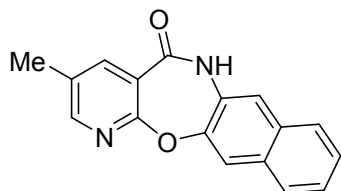
White solid; **¹H NMR (300 MHz, [D₆]DMSO):** δ = 10.78 (br s, 1H), 8.33 (dd, *J* = 0.9, 1.2 Hz, 1H), 8.08 (dd, *J* = 0.9, 2.6 Hz, 1H), 7.44 (d, *J* = 2.4 Hz, 1H), 7.31 (dd, *J* = 2.4, 8.4 Hz, 1H), 7.19 (d, *J* = 8.7 Hz, 1H), 2.32 (s, 3H).

¹³C NMR (100 MHz, [D₆]DMSO): δ = 164.2, 159.9, 152.2, 148.5, 142.3, 132.3, 129.8, 128.7, 126.3, 122.8, 121.8, 119.0, 16.8.

GC-MS (EI, 70 eV): m/z (%) = 260 (100), 231 (22), 225 (77), 197 (41), 169 (23), 78 (30), 63 (55), 51 (49), 39 (34).

HRMS (EI): calcd. for $[C_{13}H_9N_2O_2Cl]^+$ 260.03471, found 260.03499; calcd. for $[C_{13}H_9N_2O_2^{37}Cl]^+$ 262.03176, found 262.03239.

3-Methylnaphtho[2,3-b]pyrido[3,2-f][1,4]oxazepin-5(6H)-one



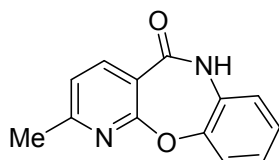
Pale pink solid; **¹H NMR (300 MHz, [D₆]DMSO):** δ = 10.99 (br s, 1H), 8.34 (s, 1H), 8.09 (s, 1H), 7.92 (s, 1H), 7.92-7.83 (m, 2H), 7.65 (s, 1H), 7.50-7.42 (m, 2H), 2.32 (s, 3H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 165.2, 160.4, 152.7, 148.5, 142.6, 132.6, 131.4, 131.1, 130.4, 127.7, 127.4, 126.9, 126.3, 119.7, 119.2, 118.9, 17.3.

GC-MS (EI, 70 eV): m/z (%) = 276 (100), 248 (39), 220 (42), 205 (12).

HRMS (EI): calcd. for $[C_{17}H_{12}O_2N_2]^+$ 276.08933, found 276.08920.

2-Methylbenzo[b]pyrido[3,2-f][1,4]oxazepin-5(6H)-one



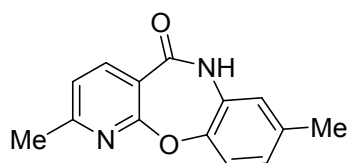
White solid; **¹H NMR (300 MHz, [D₆]DMSO)**: δ = 10.64 (br s, 1H), 8.14 (d, *J* = 7.8 Hz, 1H), 7.33 (dd, *J* = 1.5, 7.7 Hz, 1H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.24-7.12 (m, 3H), 2.49 (s, 3H, overlapped by the solvent residual peak of [D₆]DMSO),.

¹³C NMR (75 MHz, [D₆]DMSO): δ = 165.1, 162.9, 162.1, 148.5, 142.9, 131.1, 126.8, 125.9, 122.4, 122.2, 122.1, 117.3, 24.2.

GC-MS (EI, 70 eV): *m/z* (%) = 226 (100), 211 (4), 198 (67), 182 (13), 169 (39), 64 (51), 52 (88), 39 (42).

HRMS (EI): calcd. for [C₁₃H₁₀O₂N₂]⁺ 226.07368, found 226.07392.

2,8-Dimethylbenzo[b]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



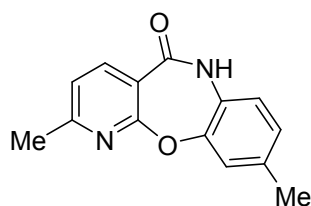
White solid; **¹H NMR (300 MHz, [D₆]DMSO)**: δ = 10.57 (br s, 1H), 8.12 (d, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 7.8 Hz, 1H), 7.20 (d, *J* = 8.7 Hz, 1H), 6.96-6.93 (m, 2H), 2.50 (s, 3H, overlapped by the solvent residual peak of [D₆]DMSO), 2.24 (s, 3H).

¹³C NMR (100 MHz, [D₆]DMSO): δ = 164.6, 162.3, 161.8, 145.9, 142.4, 135.6, 130.2, 125.8, 121.7, 121.6, 121.6, 116.9, 23.7, 20.3.

GC-MS (EI, 70 eV): *m/z* (%) = 240 (100), 225 (15), 211 (87), 197 (12), 183 (23), 169 (18), 65 (33), 52 (26), 39 (39).

HRMS (EI): calcd. for [C₁₄H₁₂O₂N₂]⁺ 240.08933, found 240.08944.

2,9-Dimethylbenzo[b]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



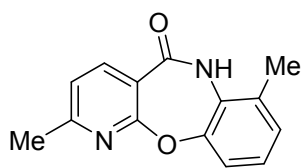
White solid; **¹H NMR (300 MHz, [D₆]DMSO)**: δ = 10.55 (br s, 1H), 8.12 (d, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 7.8 Hz, 1H), 7.16 (s, 1H), 7.07-7.00 (m, 2H), 2.48 (s, 3H, overlapped by the solvent residual peak of [D₆]DMSO), 2.26 (s, 3H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 164.5, 162.3, 161.7, 147.9, 142.3, 135.2, 127.9, 126.7, 122.1, 121.7, 121.3, 116.9, 23.7, 20.0.

GC-MS (EI, 70 eV): *m/z* (%) = 240 (100), 211 (89), 197 (15), 183 (31), 169 (19), 65 (51), 51 (35), 39 (39).

HRMS (EI): calcd. for [C₁₄H₁₂O₂N₂]⁺ 240.08933, found 240.08958.

2,7-Dimethylbenzo[*b*]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



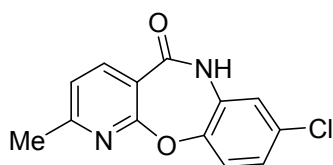
White solid; ¹H NMR (300 MHz, [D₆]DMSO): δ = 10.05 (br s, 1H), (d, *J* = 7.5 Hz, 1H), (dd, *J* = 0.6, 7.8 Hz, 1H), 7.20-7.16 (m, 1H), 7.117.05 (m, 2H), 2.48 (s, 1H), 2.33 (s, 3H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 165.3, 162.6, 150.7, 144.2, 142.6, 134.7, 132.0, 129.5, 128.3, 126.1, 122.4, 121.4, 119.9, 117.9, 112.5, 24.2, 18.3.

GC-MS (EI, 70 eV): *m/z* (%) = 240 (100), 225 (17), 211 (52), 183 (17).

HRMS (EI): calcd. for [C₁₄H₁₂O₂N₂]⁺ 240.08933, found 240.08972.

8-Chloro-2-methylbenzo[*b*]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



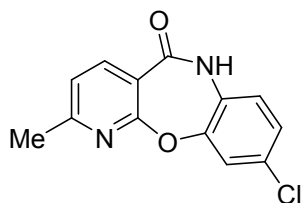
White solid; ¹H NMR (300 MHz, [D₆]DMSO): δ = 10.73 (br s, 1H), 8.15 (d, *J* = 7.8 Hz, 1H), 7.37 (d, *J* = 9.0 Hz, 1H), 7.33 (d, *J* = 7.5 Hz, 1H), 7.22-7.18 (m, 2H), 2.50 (s, 3H, overlapped by the solvent residual peak of [D₆]DMSO).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 164.3, 162.7, 161.1, 146.7, 142.6, 132.1, 129.8, 124.9, 123.6, 122.0, 120.9, 116.5, 23.7.

GC-MS (EI, 70 eV): *m/z* (%) = 260 (100), 231 (33), 225 (78), 197 (56), 169 (60), 91 (37), 78 (58), 63 (83), 51 (60), 39 (48).

HRMS (EI): calcd. for [C₁₃H₉O₂N₂Cl]⁺ 260.03471, found 260.03503.

9-Chloro-2-methylbenzo[*b*]pyrido[3,2-*f*][1,4]oxazepin-5(6*H*)-one



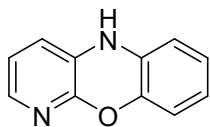
White solid; ¹H NMR (300 MHz, [D₆]DMSO): δ = 10.73 (br s, 1H), 8.15 (d, *J* = 7.8 Hz, 1H), 7.46 (d, *J* = 2.1 Hz, 1H), 7.35-7.30 (m, 2H), 7.19 (d, *J* = 8.7 Hz, 1H), 2.50 (s, 3H, overlapped by the solvent residual peak of [D₆]DMSO).

¹³C NMR (100 MHz, [D₆]DMSO): δ = 164.2, 162.7, 161.0, 148.3, 142.5, 129.9, 128.6, 126.3, 122.8, 122.1, 121.9, 116.6, 23.7.

GC-MS (EI, 70 eV): m/z (%) = 260 (99.5), 231 (15), 225 (100), 197 (45), 169 (20).

HRMS (EI): calcd. for $[\text{C}_{13}\text{H}_9\text{O}_2\text{N}_2\text{Cl}]^+$ 260.03471, found 260.03459; calcd. for $[\text{C}_{13}\text{H}_9\text{O}_2\text{N}_2^{37}\text{Cl}]^+$ 262.03176, found 262.03256.

5*H*-Benzo[*b*]pyrido[3,2-*e*][1,4]oxazine^[3]



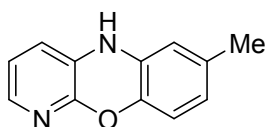
White solid; **¹H NMR (300 MHz, [D₆]DMSO):** δ = 9.00 (br s, 1H), 7.53 (dd, *J* = 1.5, 5.1 Hz, 1H), 6.88 (d, *J* = 0.9, 7.8 Hz, 1H), 6.79-6.74 (m, 1H), 6.66-6.53 (m, 4H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 145.9, 142.2, 141.6, 139.0, 131.3, 124.1, 121.2, 120.5, 116.4, 114.9, 114.0.

GC-MS (EI, 70 eV): m/z (%) = 184 (100), 155 (29), 129 (8), 92 (12).

HRMS (EI): calcd. for $[\text{C}_{11}\text{H}_8\text{ON}_2]^+$ 184.06311, found 184.06293.

7-Methyl-5*H*-benzo[*b*]pyrido[3,2-*e*][1,4]oxazine



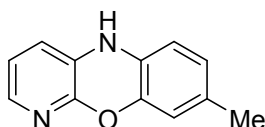
White solid; **¹H NMR (300 MHz, [D₆]DMSO):** δ = 8.97 (br s, 1H), 7.52 (dd, *J* = 1.5, 5.1 Hz, 1H), 6.88-6.85 (m, 1H), 6.56-6.52 (m, 2H), 6.44-6.39 (m, 2H), 2.10 (s, 3H)

¹³C NMR (75 MHz, [D₆]DMSO): δ = 145.9, 141.4, 140.1, 139.1, 133.0, 130.9, 121.3, 120.4, 116.4, 114.6, 114.5, 20.3.

GC-MS (EI, 70 eV): m/z (%) = 198 (100), 169 (15), 155 (7), 99 (7).

HRMS (EI): calcd. for $[\text{C}_{12}\text{H}_{10}\text{ON}_2]^+$ 198.07876, found 198.07861.

8-Methyl-5*H*-benzo[*b*]pyrido[3,2-*e*][1,4]oxazine



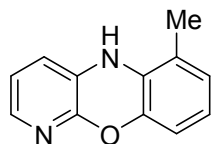
White solid; **¹H NMR (300 MHz, [D₆]DMSO):** δ = 8.90 (br s, 1H), 7.51 (dd, *J* = 1.5, 5.1 Hz, 1H), 6.88-6.85 (m, 1H), 6.59-6.46 (m, 4H), 2.10 (s, 3H)

¹³C NMR (75 MHz, [D₆]DMSO): δ = 146.1, 142.0, 141.5, 138.9, 130.4, 128.6, 124.2, 122.6, 120.4, 115.6, 113.8, 20.1.

GC-MS (EI, 70 eV): m/z (%) = 198 (100), 169 (11), 116 (9).

HRMS (EI): calcd. for $[C_{12}H_{10}ON_2]^+$ 198.07876, found 198.07876.

6-Methyl-5H-benzo[b]pyrido[3,2-e][1,4]oxazine



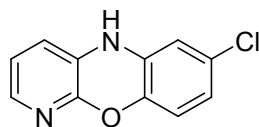
Light yellow solid; **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 8.26 (br s, 1H), 7.56 (d, J = 4.8 Hz, 1H), 6.89 (d, J = 7.8 Hz, 1H), 6.64-6.48 (m, 4H), 2.09 (s, 3H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 146.5, 142.7, 141.9, 139.6, 129.6, 126.2, 123.4, 121.3, 120.9, 117.3, 113.3, 17.3.

GC-MS (EI, 70 eV): m/z (%) = 198 (100), 169 (15), 99 (7).

HRMS (EI): calcd. for $[C_{12}H_{10}ON_2]^+$ 198.07876, found 198.07874.

7-Chloro-5H-benzo[b]pyrido[3,2-e][1,4]oxazine



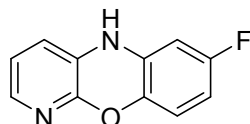
White solid; **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 9.20 (br s, 1H), 7.56 (dd, J = 1.5, 5.1 Hz, 1H), (d, J = 7.8 Hz, 1H), 6.66-6.57 (m, 4H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 145.0, 141.8, 141.2, 138.8, 132.9, 127.5, 120.9, 120.3, 117.1, 116.3, 113.3.

GC-MS (EI, 70 eV): m/z (%) = 218 (100), 183 (14), 155 (29), 109 (11).

HRMS (EI): calcd. for $[C_{11}H_7ON_2Cl]^+$ 218.02414, found 218.02456.

7-Fluoro-5H-benzo[b]pyrido[3,2-e][1,4]oxazine



White solid; **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 9.19 (br s, 1H), 7.56 (dd, J = 1.5, 5.1 Hz, 1H), 6.92 (dd, J = 0.6, 7.2 Hz, 1H), 6.69-6.59 (m, 2H), 6.46-6.36 (m, 2H).

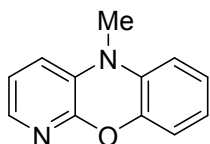
^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 158.5 (d, J = 236.3 Hz), 144.9, 141.7, 138.9, 138.5 (d, J = 3.0 Hz), 132.8 (d, J = 11.3 Hz), 120.8, 117.1, 115.7 (d, J = 9.8 Hz), 106.3 (d, J = 22.5 Hz), 101.0 (d, J = 27.8 Hz).

^{19}F NMR (282 MHz, $[D_6]DMSO$): δ = -118.8 (m);

GC-MS (EI, 70 eV): m/z (%) = 202 (100), 173 (29), 101 (13).

HRMS (EI): calcd. for $[C_{11}H_7ON_2F]^+$ 202.05369, found 202.05378.

5-Methyl-5H-benzo[b]pyrido[3,2-e][1,4]oxazine



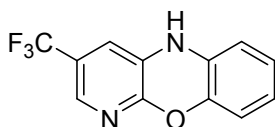
White solid; **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 7.66-7.64 (m, 1H), 6.95-6.87 (m, 2H), 6.75-6.70 (m, 3H), 6.65-6.60 (m, 1H), 3.16 (s, 3H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 146.2, 143.8, 141.7, 140.6, 133.4, 124.8, 122.2, 120.9, 117.1, 115.3, 113.3, 113.3, 28.5.

GC-MS (EI, 70 eV): m/z (%) = 198 (100), 183 (86), 169 (6).

HRMS (EI): calcd. for $[C_{12}H_{10}ON_2]^+$ 198.07876, found 198.07824.

3-(Trifluoromethyl)-5H-benzo[b]pyrido[3,2-e][1,4]oxazine



Pale yellow solid; **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 9.69 (br s, 1H), 7.86 (q, J = 1.2 Hz, 1H), 7.15 (d, J = Hz, 1H), 6.84-6.78 (m, 1H), 6.73-6.66 (m, 2H), 6.63-6.60 (m, 1H).

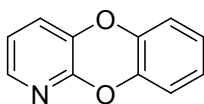
^{19}F NMR (292 MHz, $[D_6]DMSO$): δ = -60.04 (s).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 149.6, 142.5, 139.8, 139.6, 130.3, 124.2 (q, J = 269 Hz), 117.9 (q, J = 32.2 Hz), 116.9, 115.6, 115.1.

GC-MS (EI, 70 eV): m/z (%) = 252 (100), 223 (15), 126 (10).

HRMS (EI): calcd. for $[C_{12}H_7ON_2F_3]^+$ 252.05050, found 252.05056.

Benzo[5,6][1,4]dioxino[2,3-b]pyridine^[2]



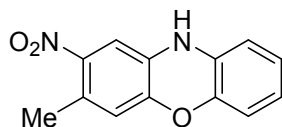
White solid; **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 7.83-7.82 (m, 1H), 7.44-7.40 (m, 1H), 7.11-7.00 (m, 5H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 148.7, 141.6, 141.2, 140.6, 137.4, 124.8, 124.6, 124.4, 121.5, 116.9, 116.2.

GC-MS (EI, 70 eV): m/z (%) = 185 (100), 129 (11), 102 (13), 93 (9).

HRMS (ESI): calcd. for $[C_{11}H_7NO_2+H]^+$ 186.05495, found 186.05504.

3-Methyl-2-nitro-10H-phenoxazine



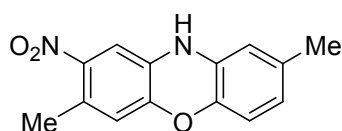
Dark red powder, **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 9.26 (br s, 1H), 7.30 (s, 1H), 6.83-6.77 (m, 1H), 6.72-6.65 (m, 2H), 6.55 (d, J = 7.2 Hz, 1H), 6.34 (s, 1H), 2.40 (s, 3H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 142.2, 140.4, 139.1, 138.1, 132.8, 129.4, 124.3, 122.2, 115.2, 114.8, 114.2, 111.4, 20.9.

GC-MS (EI, 70 eV): m/z (%) = 242 (100), 225 (13), 212 (12), 196 (97), 167 (33).

HRMS (ESI): calcd. for $[C_{13}H_{10}N_2O_3 - H]^-$ 241.06187, found 241.06206.

2,7-Dimethyl-3-nitro-5H-benzo[b]pyrido[3,2-*e*][1,4]oxazine



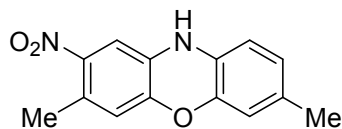
Brown solid, **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 9.16 (br s, 1H), 7.26 (s, 1H), 6.55-6.46 (m, 2H), 6.34-6.33 (m, 1H), 6.32-6.31 (m, 1H), 2.49 (s, 3H), 2.10 (s, 3H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 141.0, 140.5, 139.6, 138.6, 133.9, 133.1, 129.5, 122.9, 115.5, 115.4, 115.2, 111.8, 21.4, 20.7.

GC-MS (EI, 70 eV): m/z (%) = 256 (100), 239 (12), 226 (10), 210 (97), 180 (14), 167 (17).

HRMS (EI): calcd. for $[C_{14}H_{12}O_3N_2]^+$ 256.08424, found 256.08438.

2,8-Dimethyl-3-nitro-5H-benzo[b]pyrido[3,2-*e*][1,4]oxazine



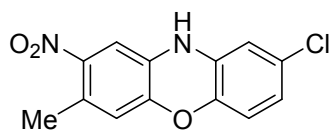
Dark red solid, **1H NMR (300 MHz, $[D_6]DMSO$):** δ = 9.15 (br s, 1H), 7.27-7.25 (m, 1H), 6.60-6.57 (m, 1H), 6.50-6.43 (m, 2H), 6.30-6.28 (m, 1H), 2.39 (s, 3H), 2.11 (s, 3H).

^{13}C NMR (75 MHz, $[D_6]DMSO$): δ = 141.9, 140.3, 138.7, 138.3, 132.9, 131.7, 126.7, 124.4, 115.8, 114.7, 114.0, 111.4, 21.0, 20.1.

GC-MS (EI, 70 eV): m/z (%) = 256 (100), 239 (10), 226 (11), 210 (97), 180 (14), 167 (16).

HRMS (EI): calcd. for $[C_{14}H_{12}O_3N_2]^+$ 256.08424, found 256.08439.

7-Chloro-2-methyl-3-nitro-5*H*-benzo[*b*]pyrido[3,2-*e*][1,4]oxazine

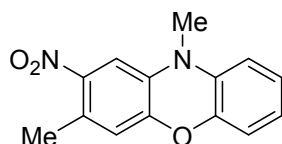


Dark red solid, **¹H NMR (300 MHz, [D₆]DMSO)**: δ = 9.33 (br s, 1H), 7.29 (s, 1H), 6.72-6.64 (m, 2H), 6.52 (d, *J* = 2.4 Hz, 1H), 6.36 (s, 1H), 2.39 (s, 3H).

¹³C NMR (75 MHz, [D₆]DMSO): δ = 141.1, 140.3, 139.7, 136.9, 132.7, 131.1, 127.6, 121.3, 116.5, 115.3, 113.5, 111.6, 20.7.

HRMS (ESI): calcd. for [C₁₃H₉ClN₂O₃ - H]⁻ 275.02289, found 275.02344; calcd. for [C₁₃H₉³⁷ClN₂O₃ - H]⁻ 277.02036, found 277.02079.

2,5-Dimethyl-3-nitro-5*H*-benzo[*b*]pyrido[3,2-*e*][1,4]oxazine



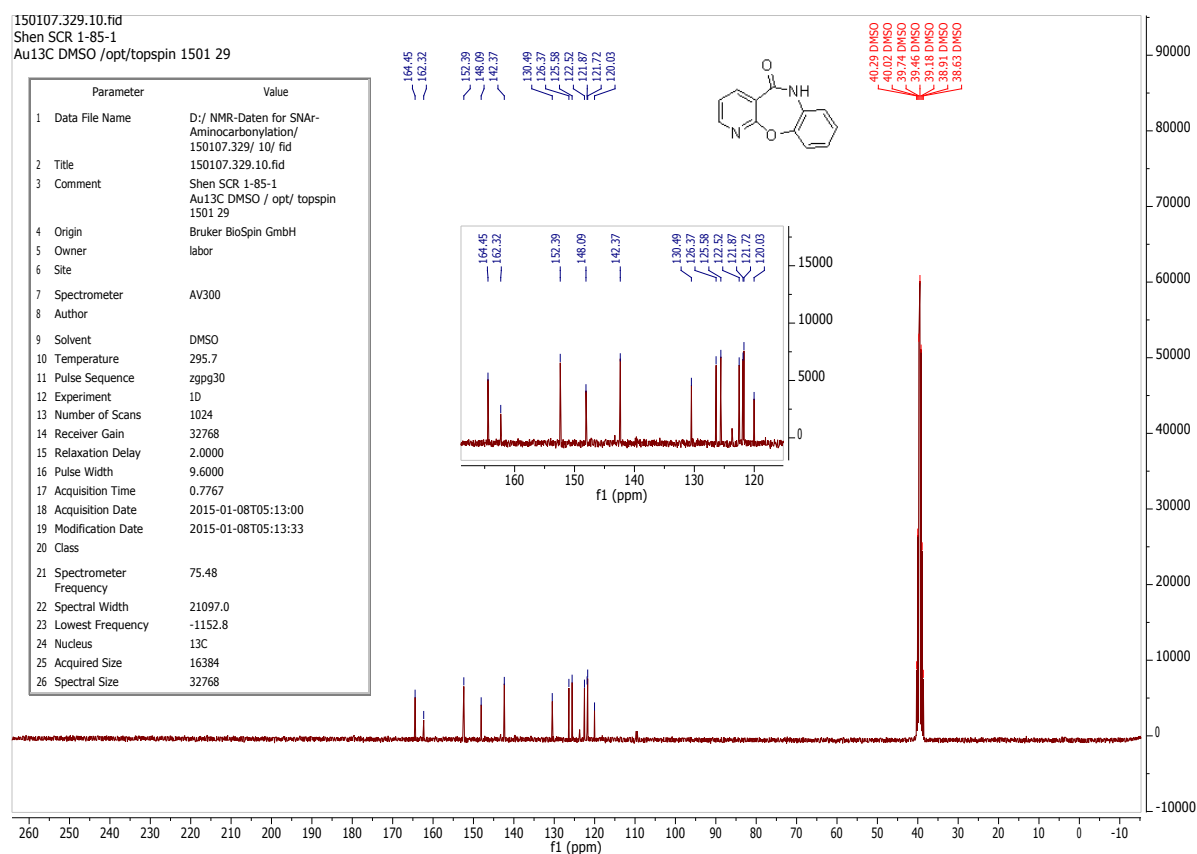
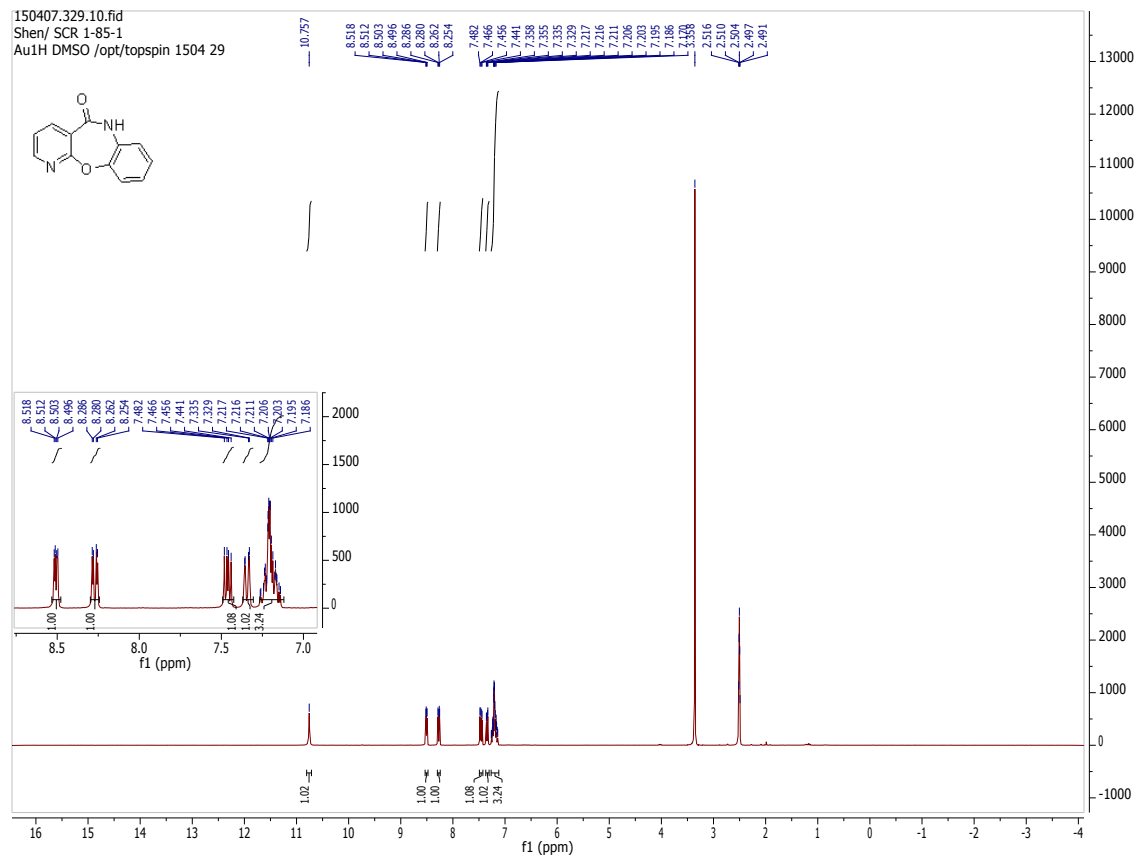
Dark red solid, **¹H NMR (300 MHz, [D₆]DMSO)**: δ = 7.33 (s, 1H), 6.96-6.90 (m, 1H), 6.86-6.79 (m, 2H), 6.76-6.73 (m, 2H), 3.13 (s, 3H), 2.48 (s, 3H partially overlapped by the solvent residual peak of [D₆]DMSO)

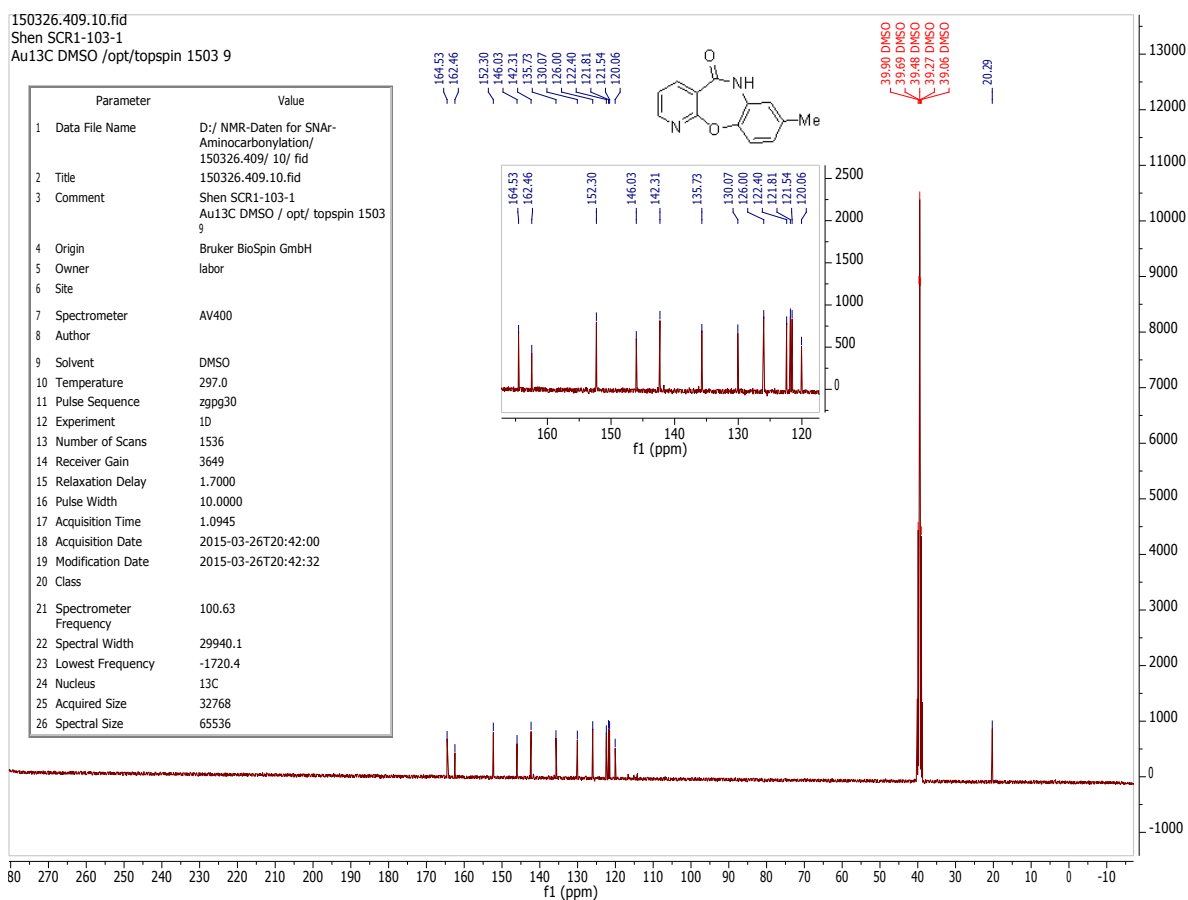
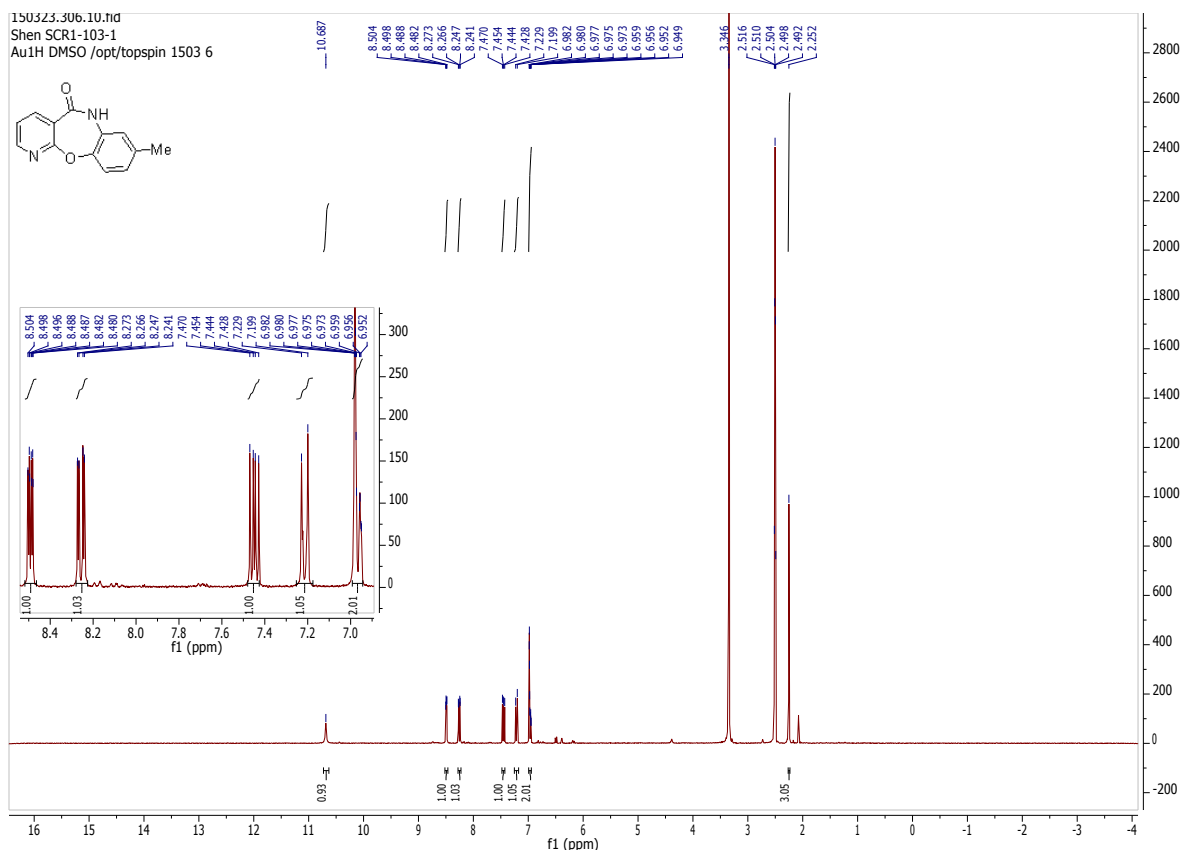
¹³C NMR (75 MHz, [D₆]DMSO): δ = 143.7, 141.9, 139.6, 139.4, 132.6, 131.9, 124.5, 122.7, 115.1, 114.6, 113.3, 110.8, 31.2, 20.9.

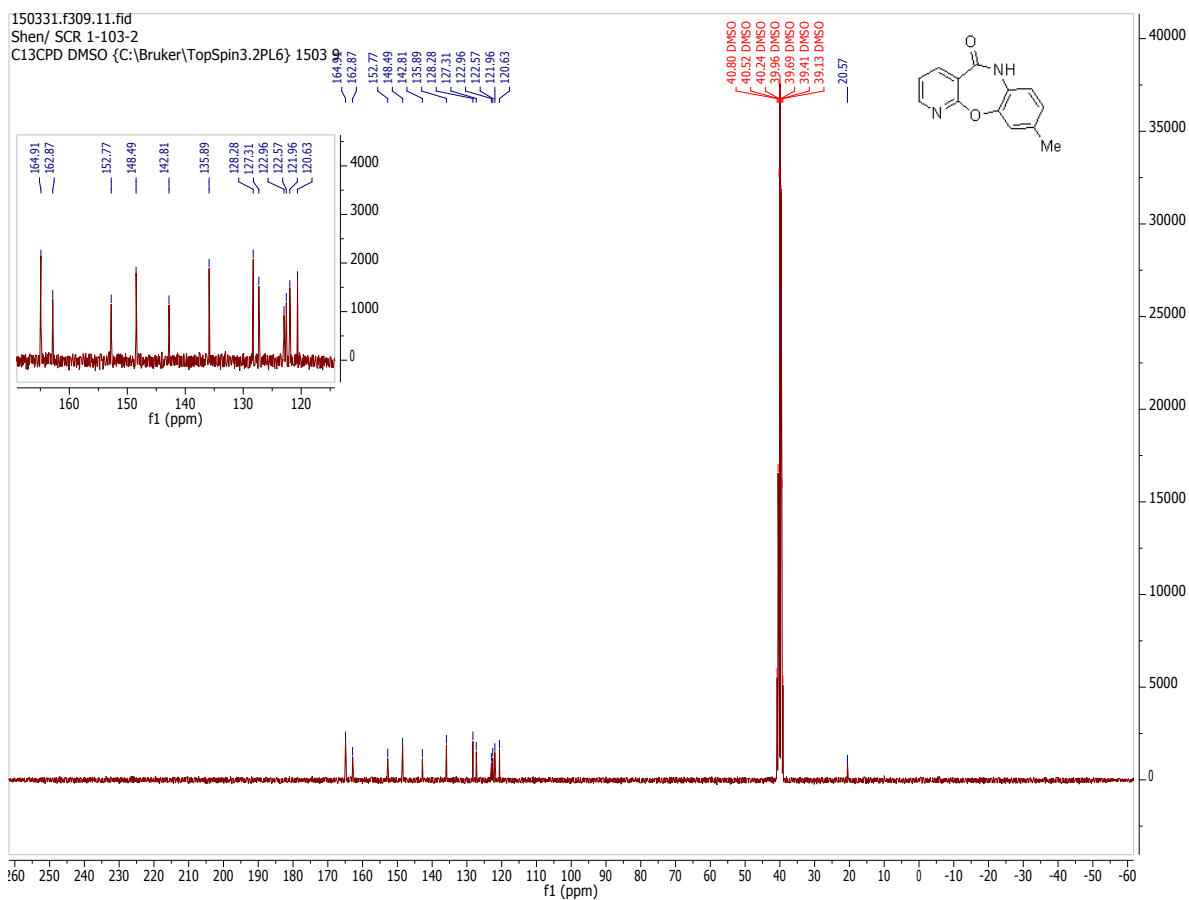
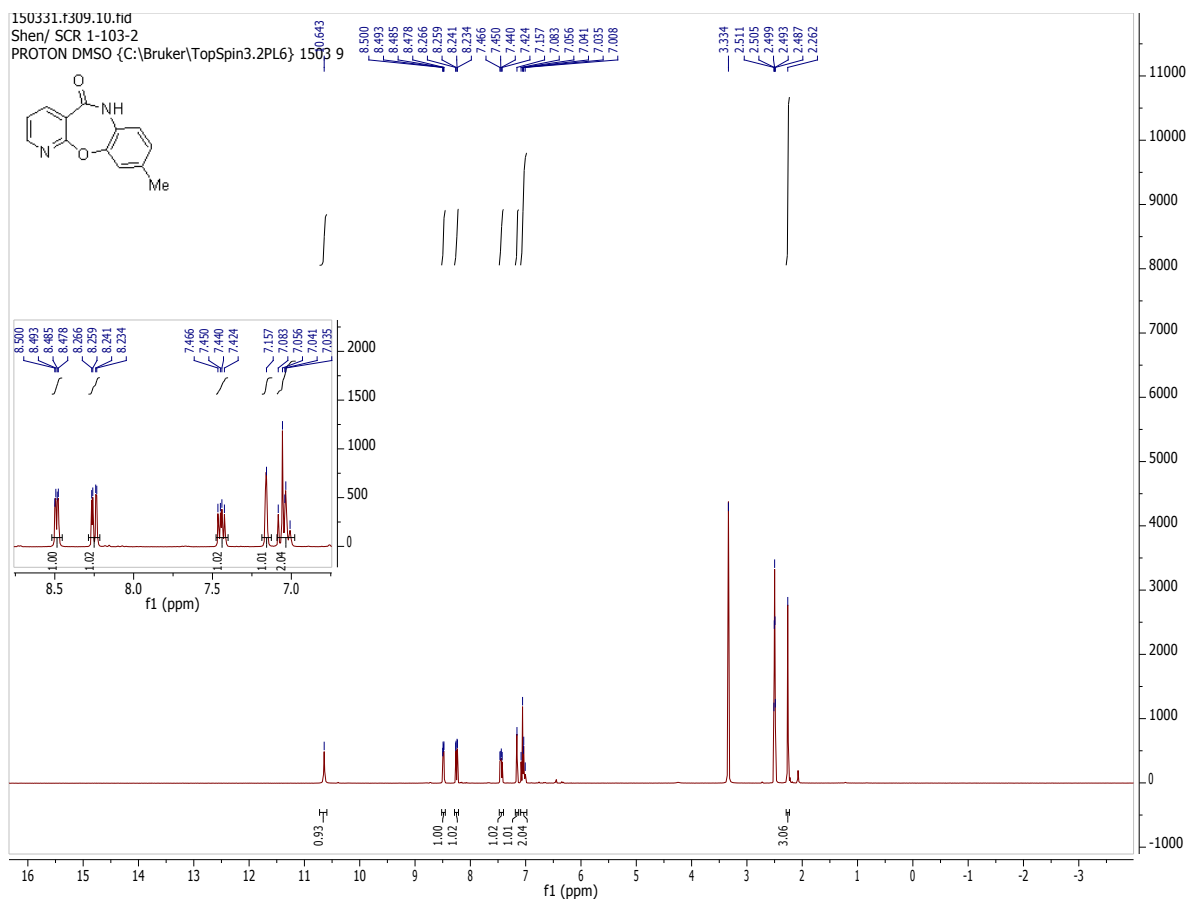
GC-MS (EI, 70 eV): *m/z* (%) = 256 (100), 239 (10), 226 (7), 210 (74), 195 (52).

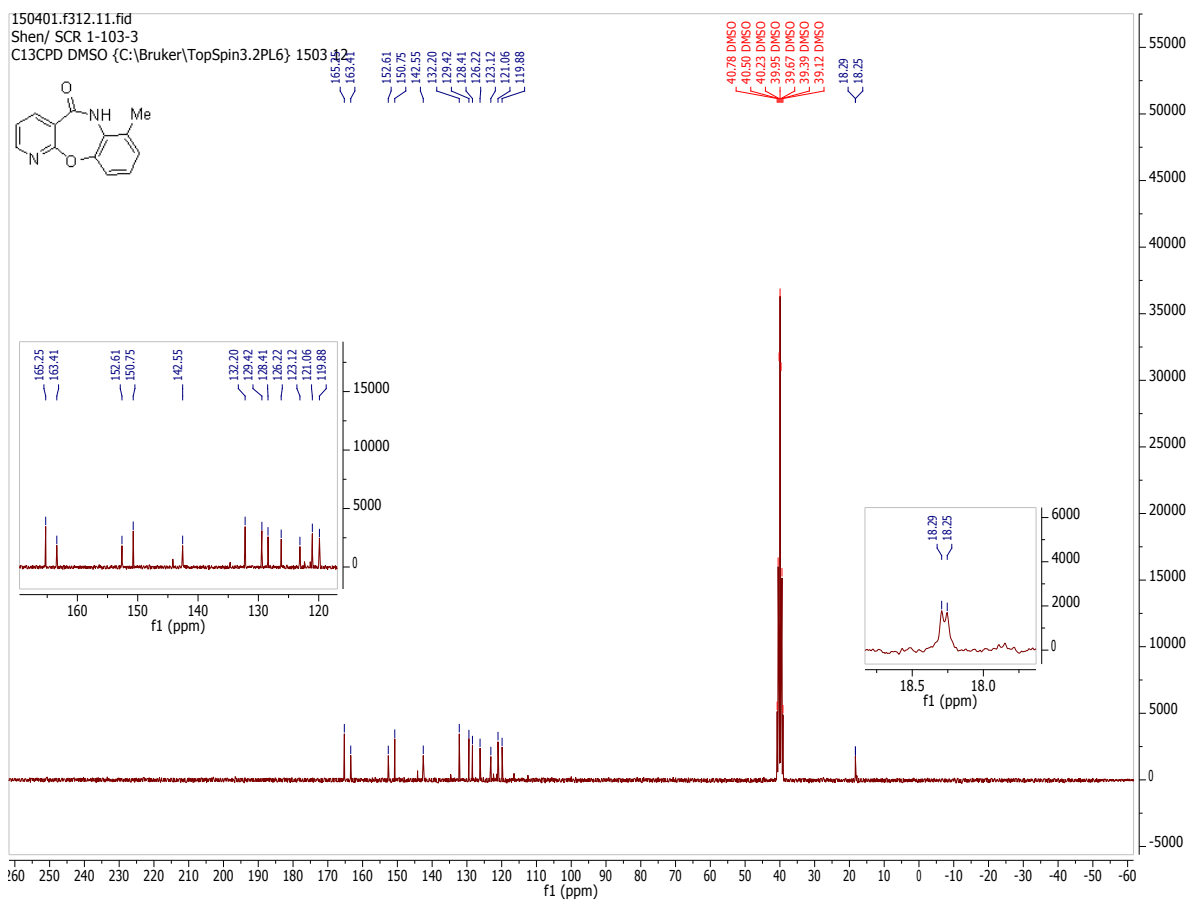
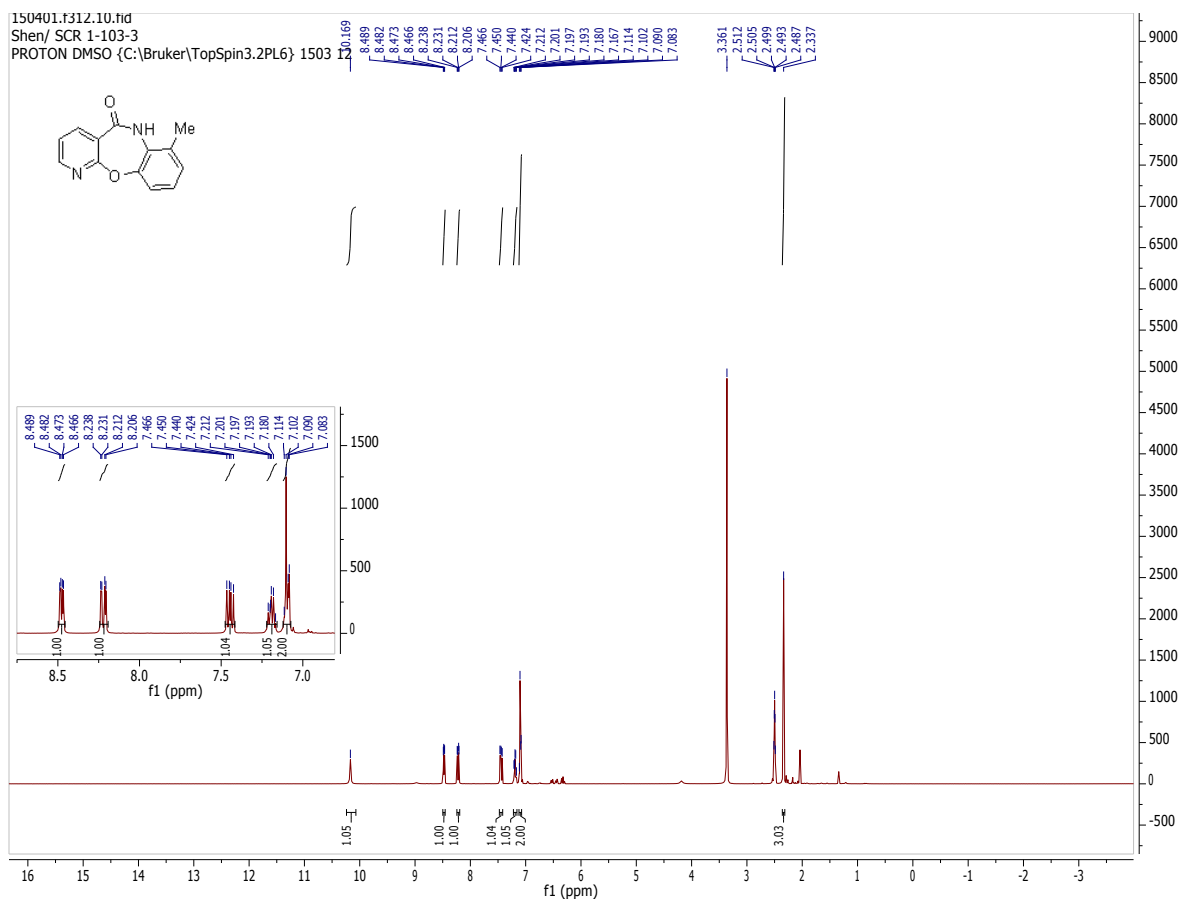
HRMS (EI): calcd. for [C₁₄H₁₂O₃N₂]⁺ 256.08424, found 256.08482

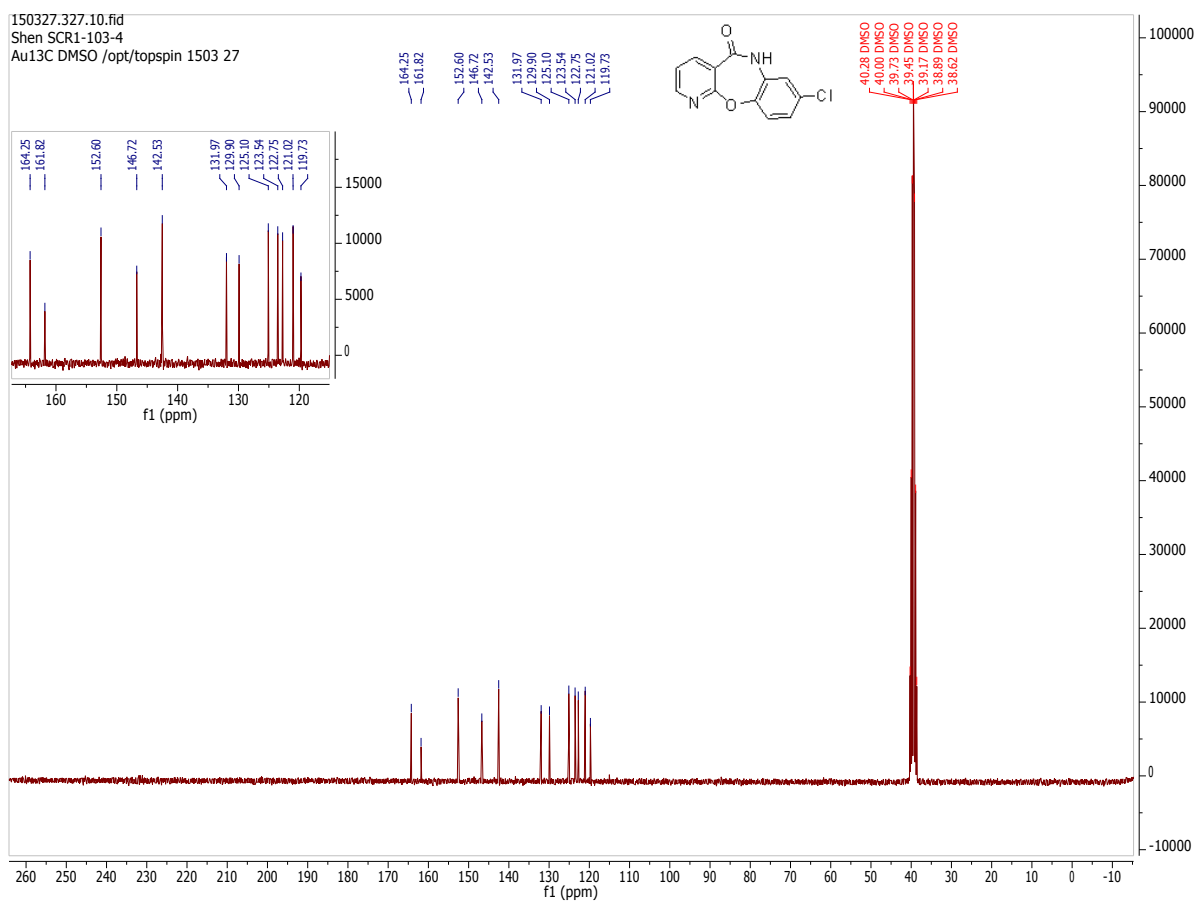
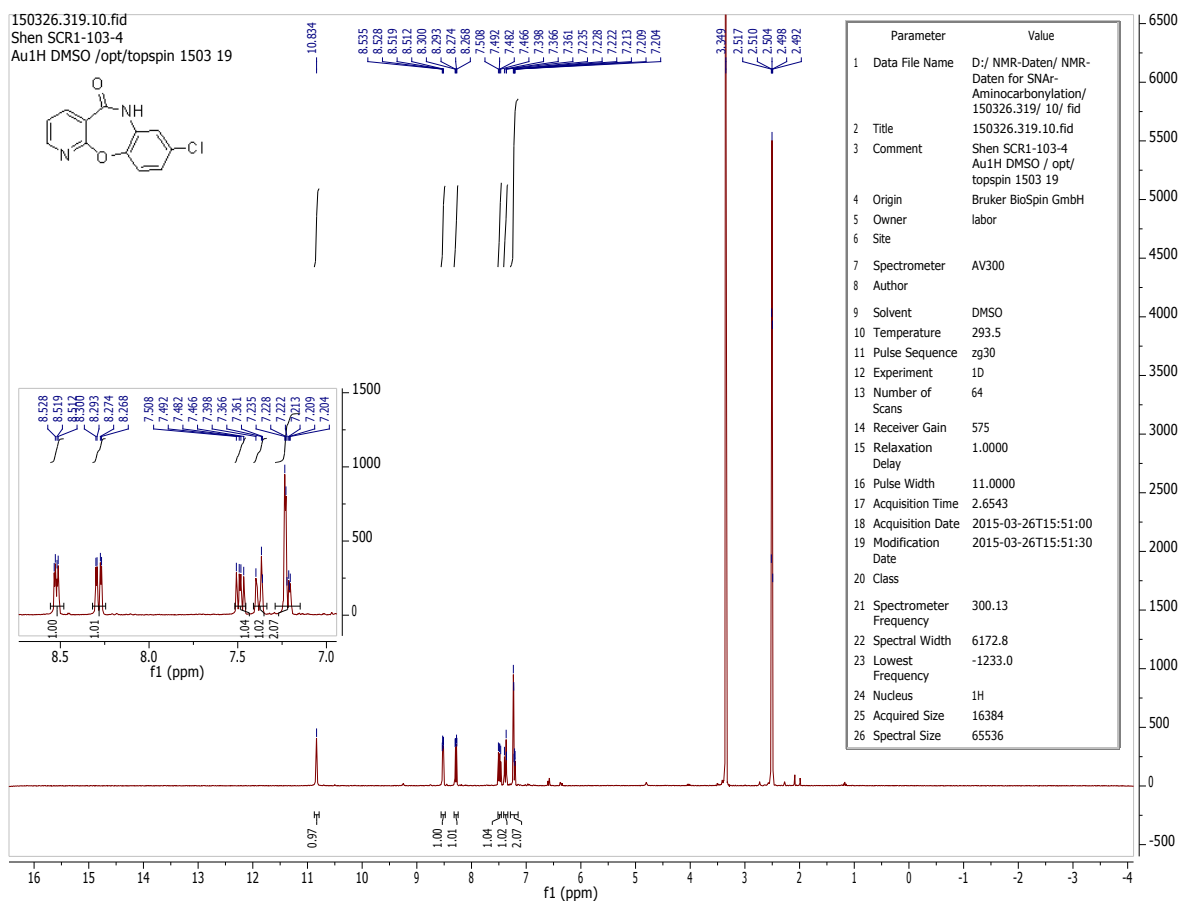
Spectrum Copies

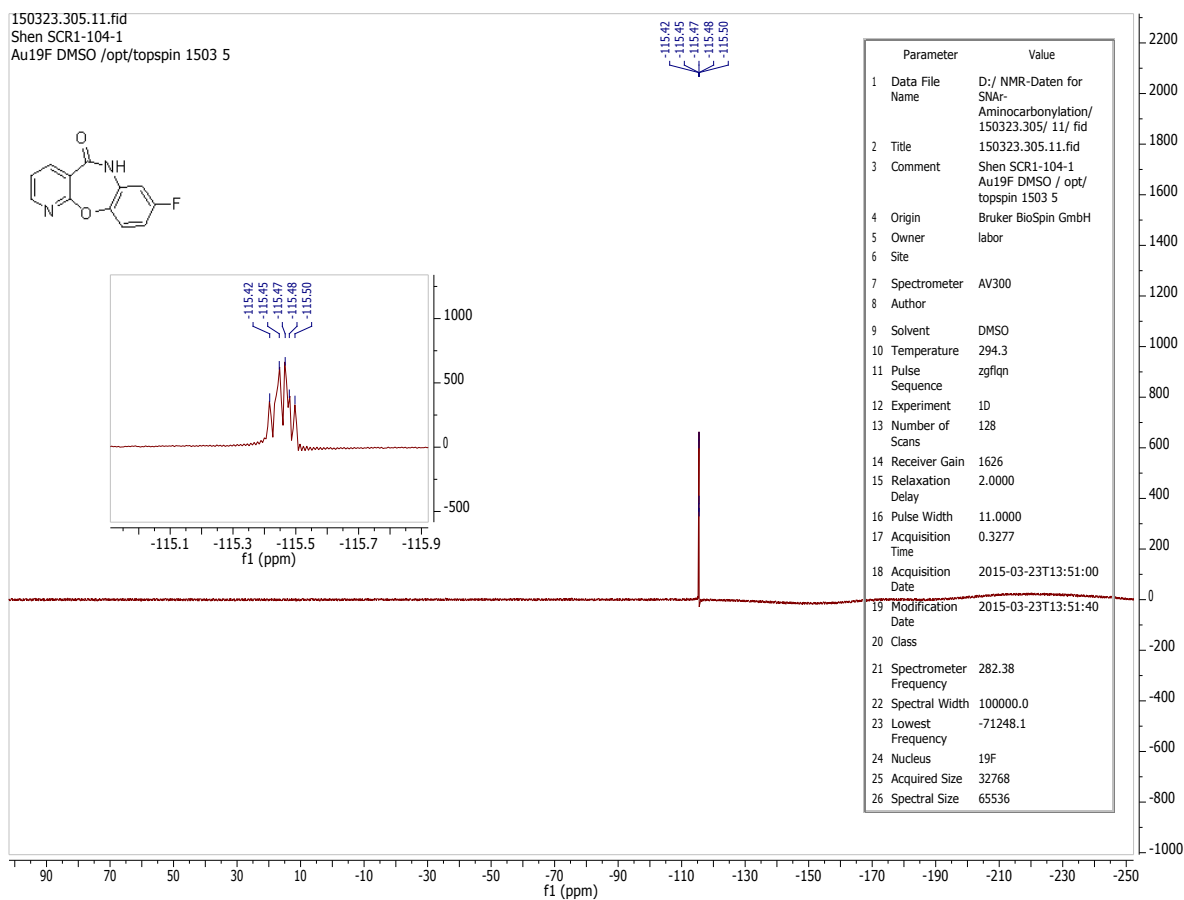
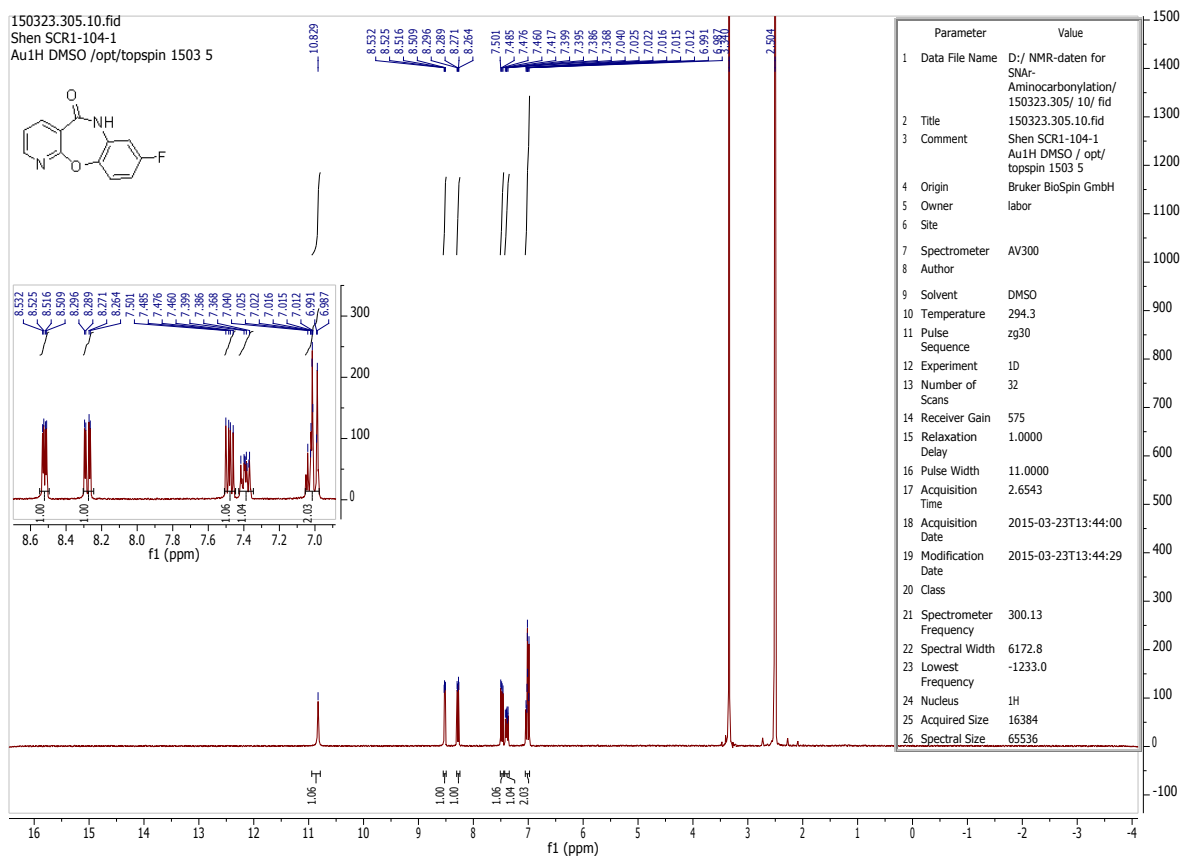




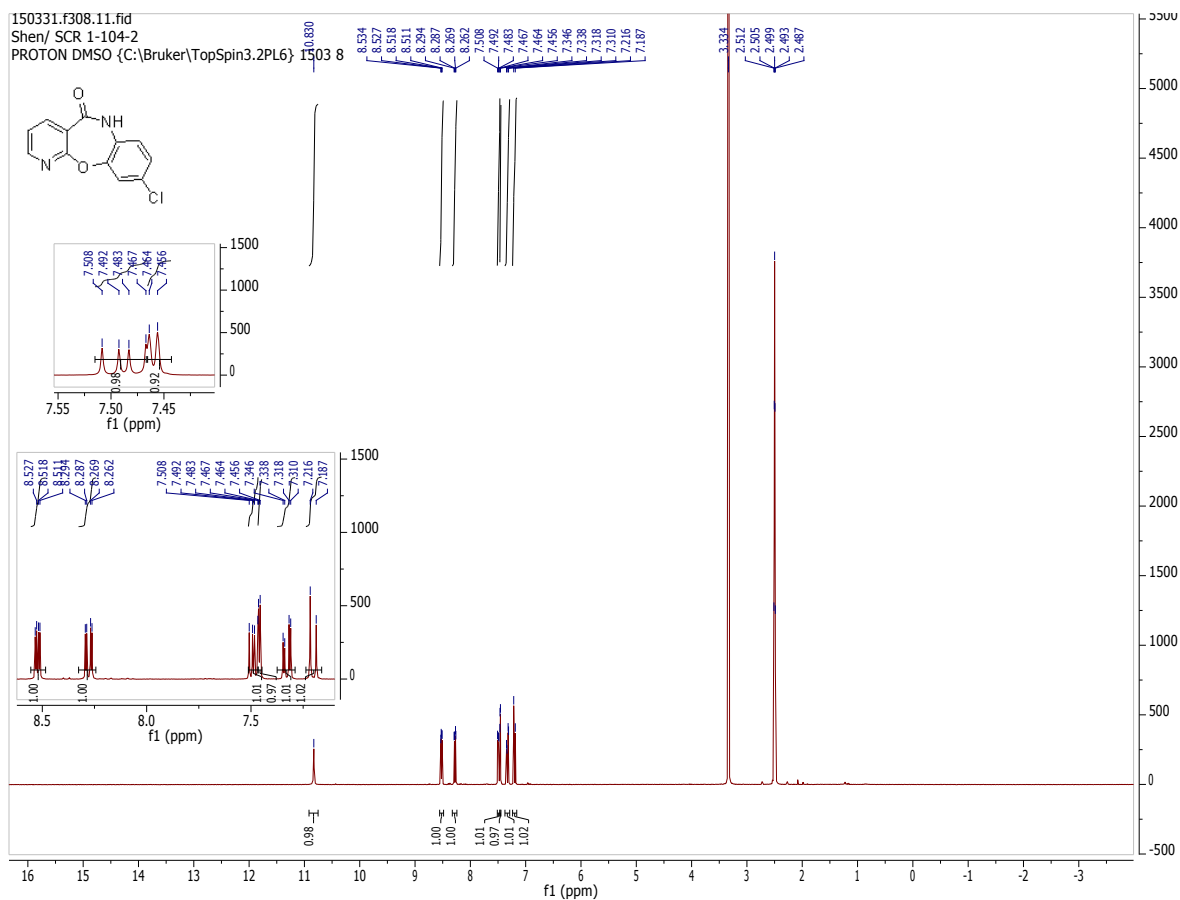
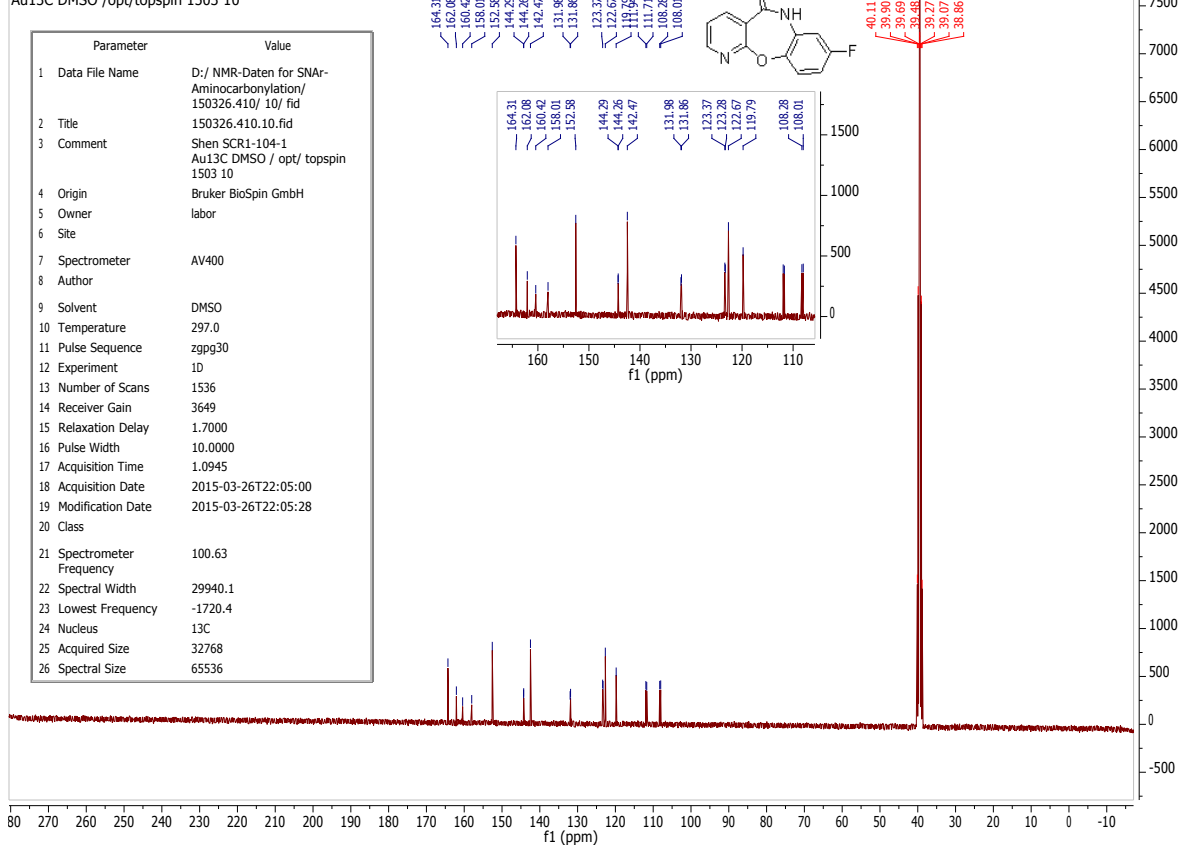


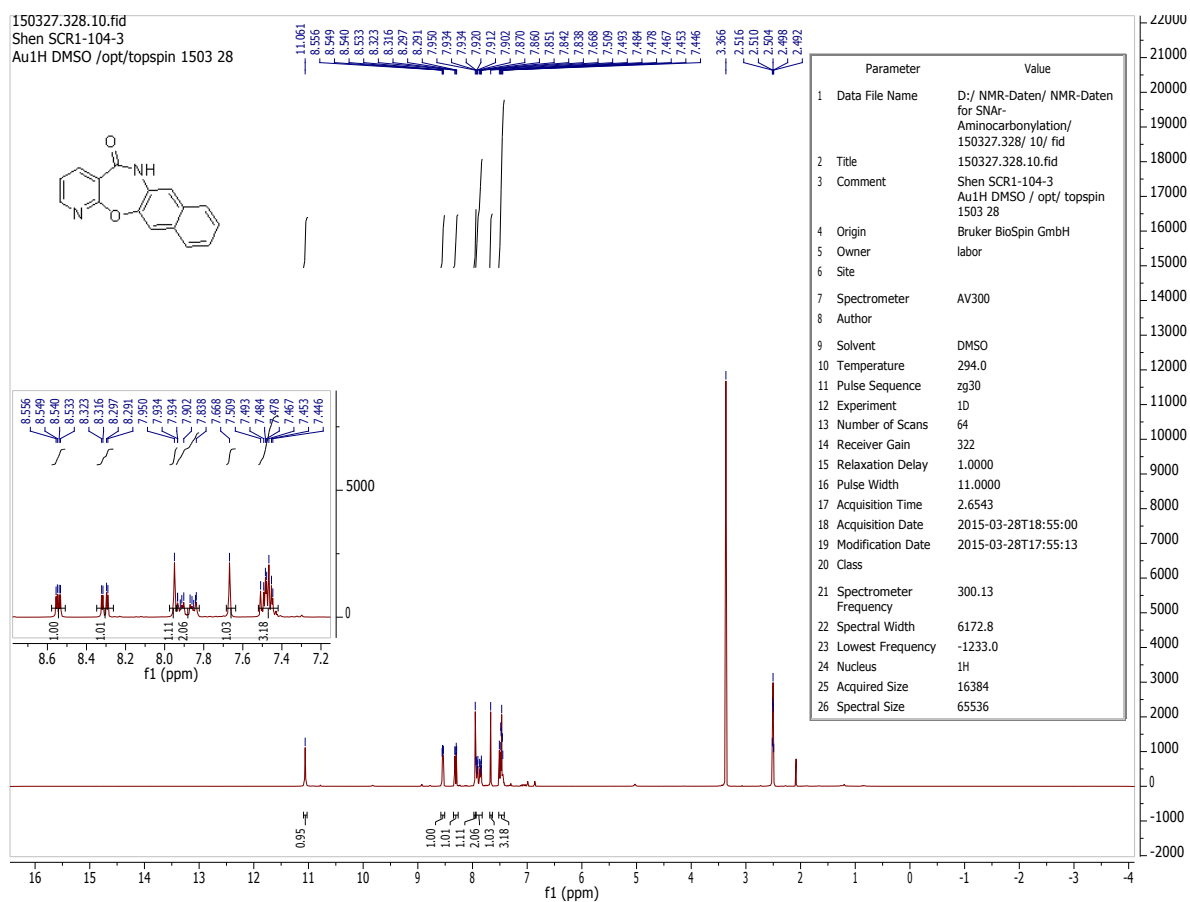
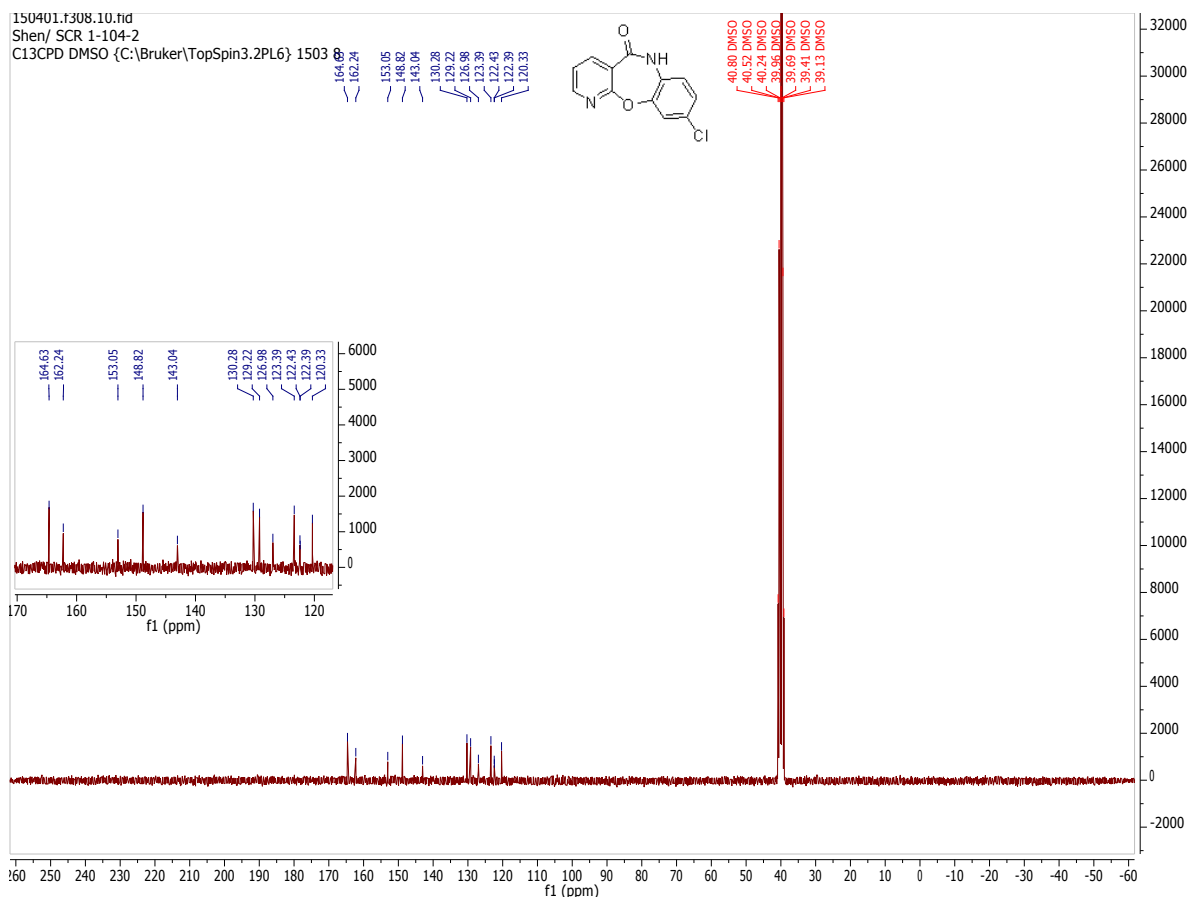


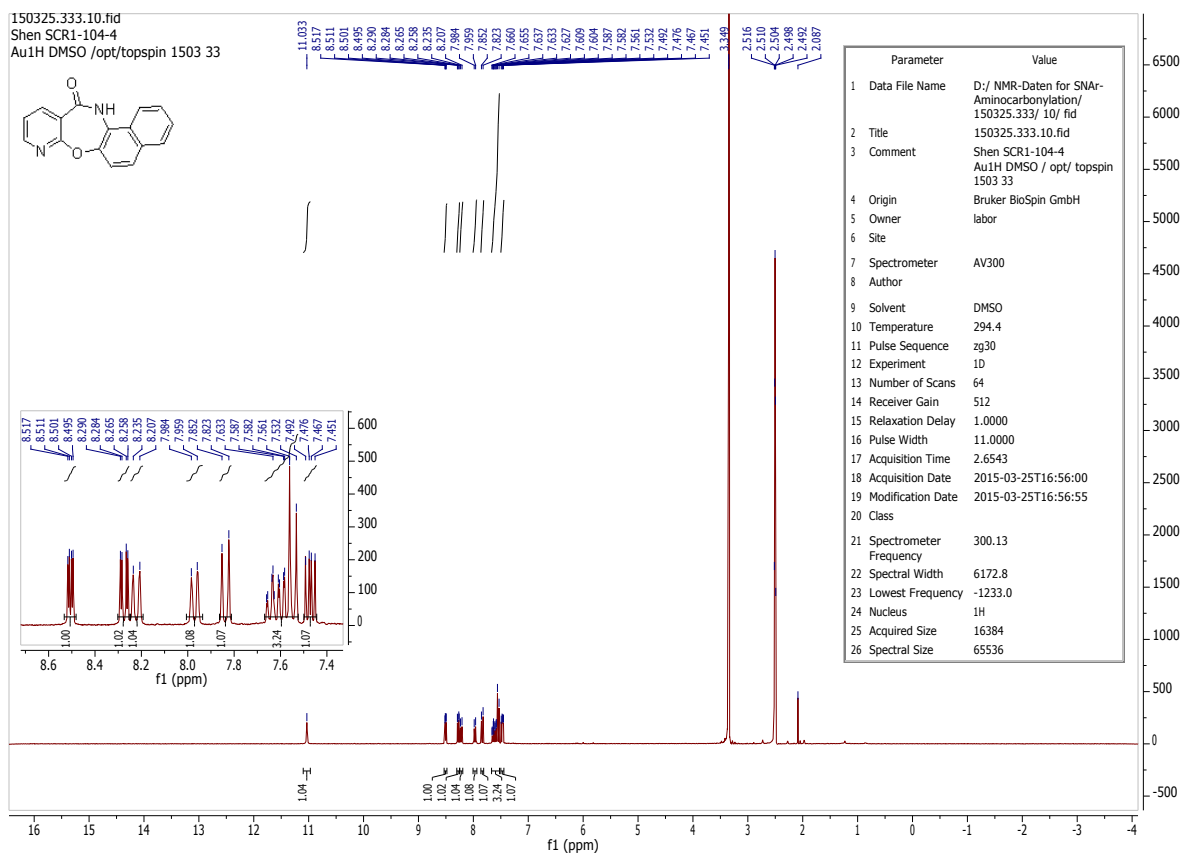
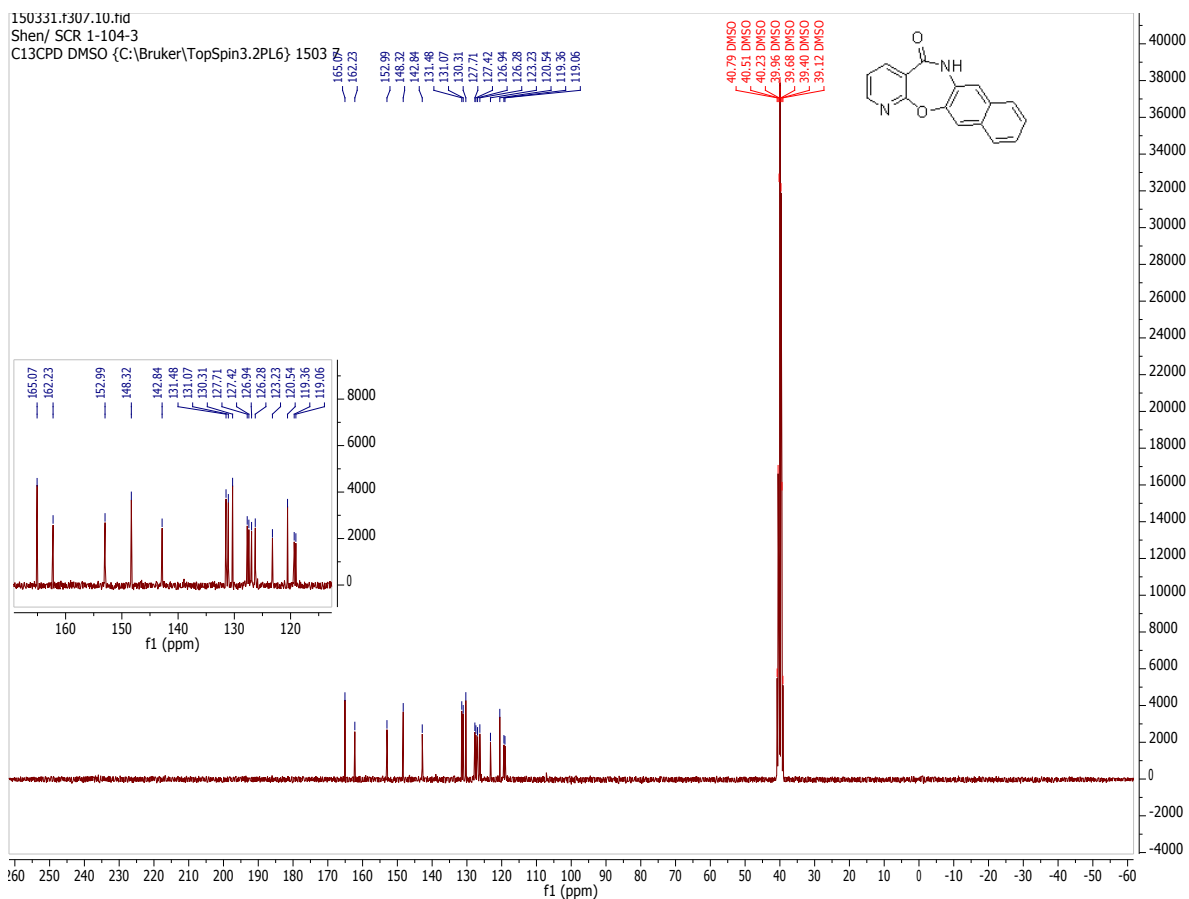


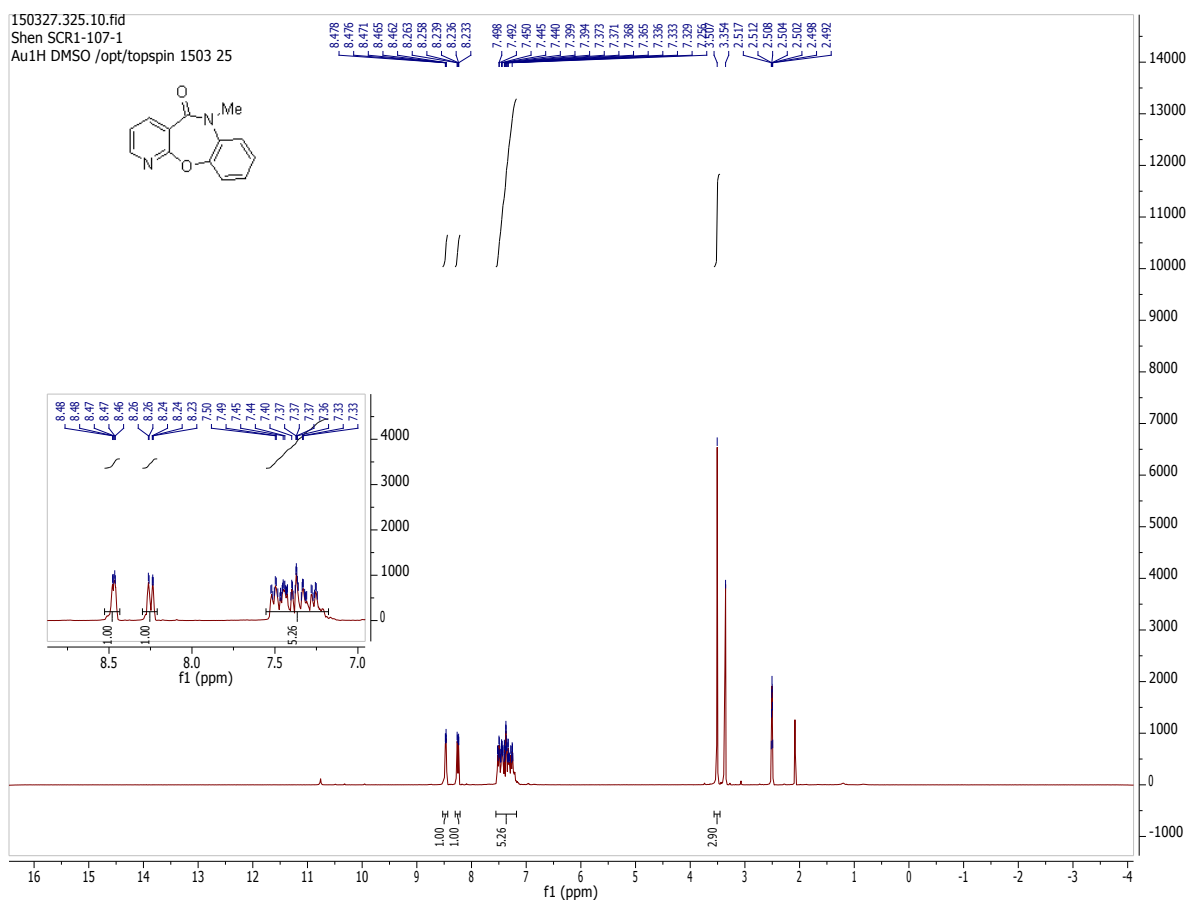
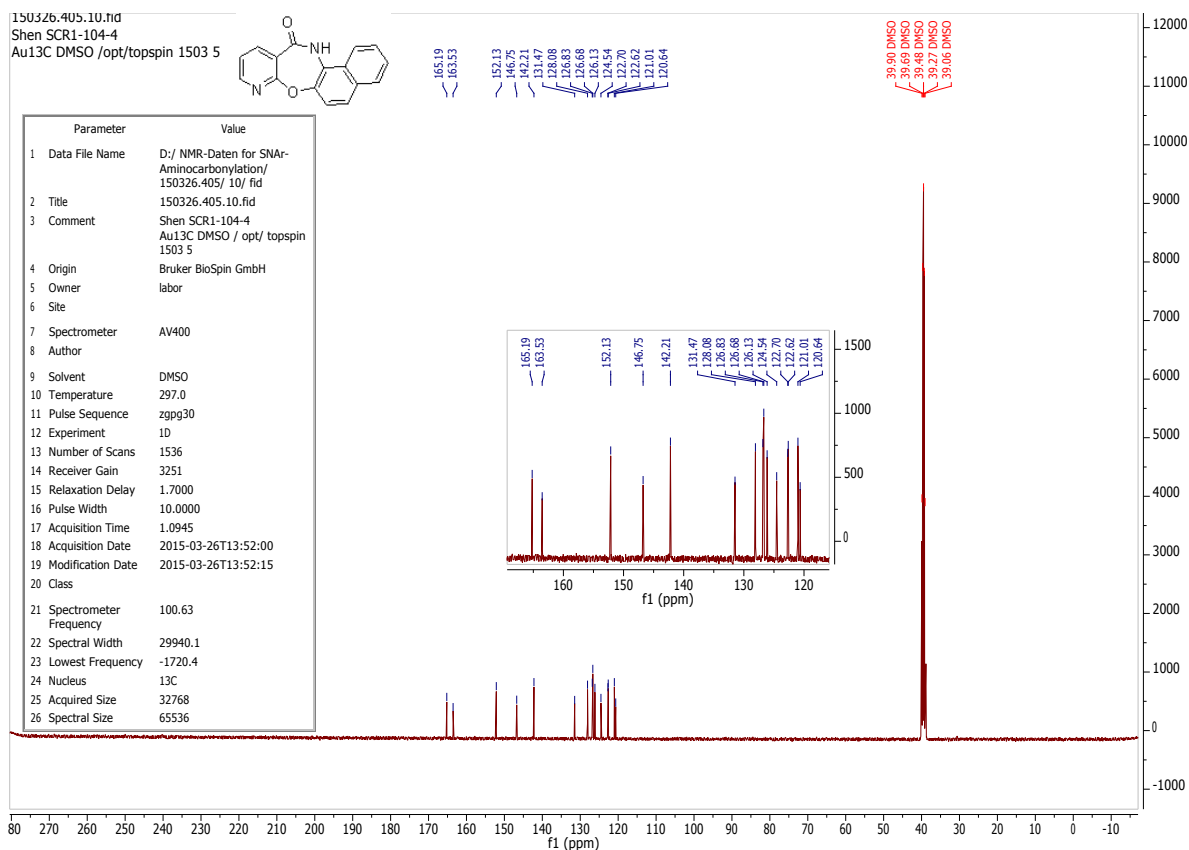


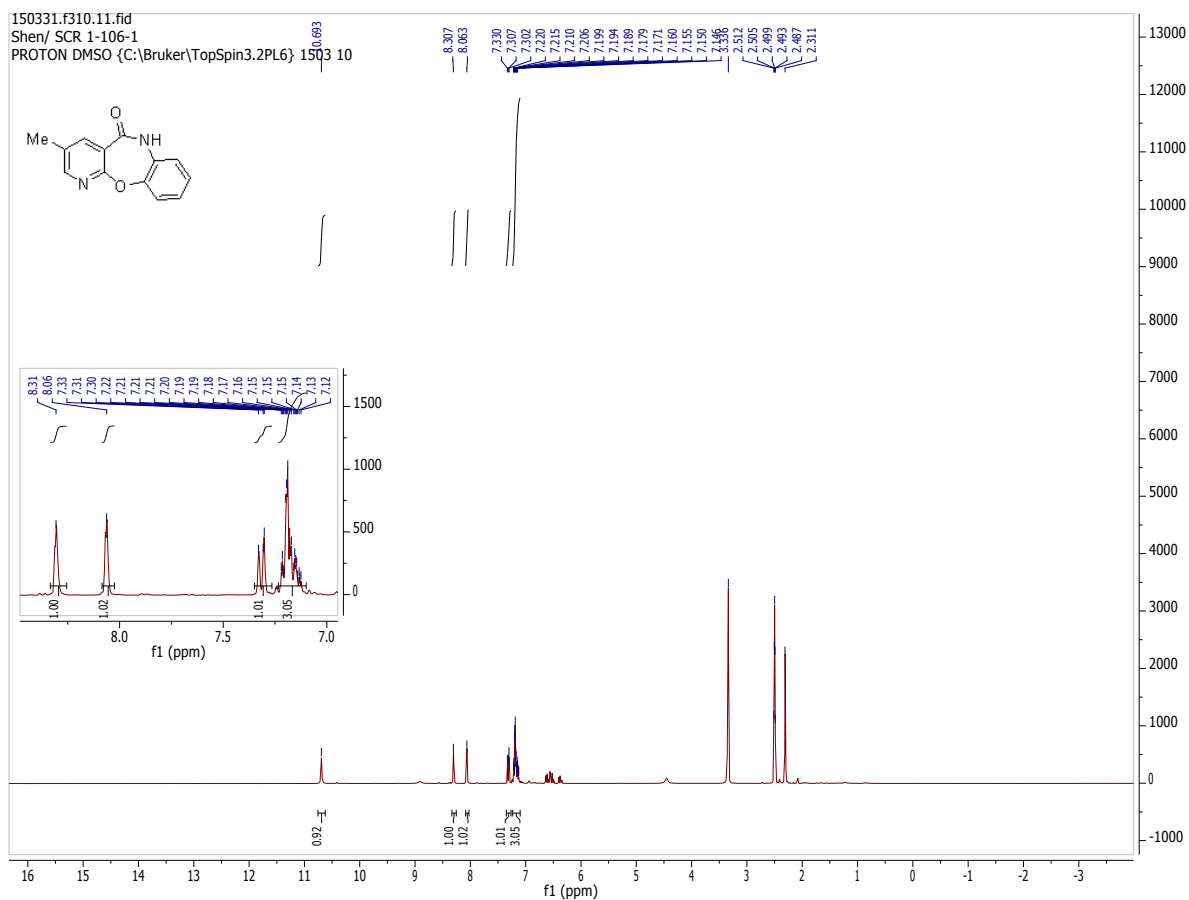
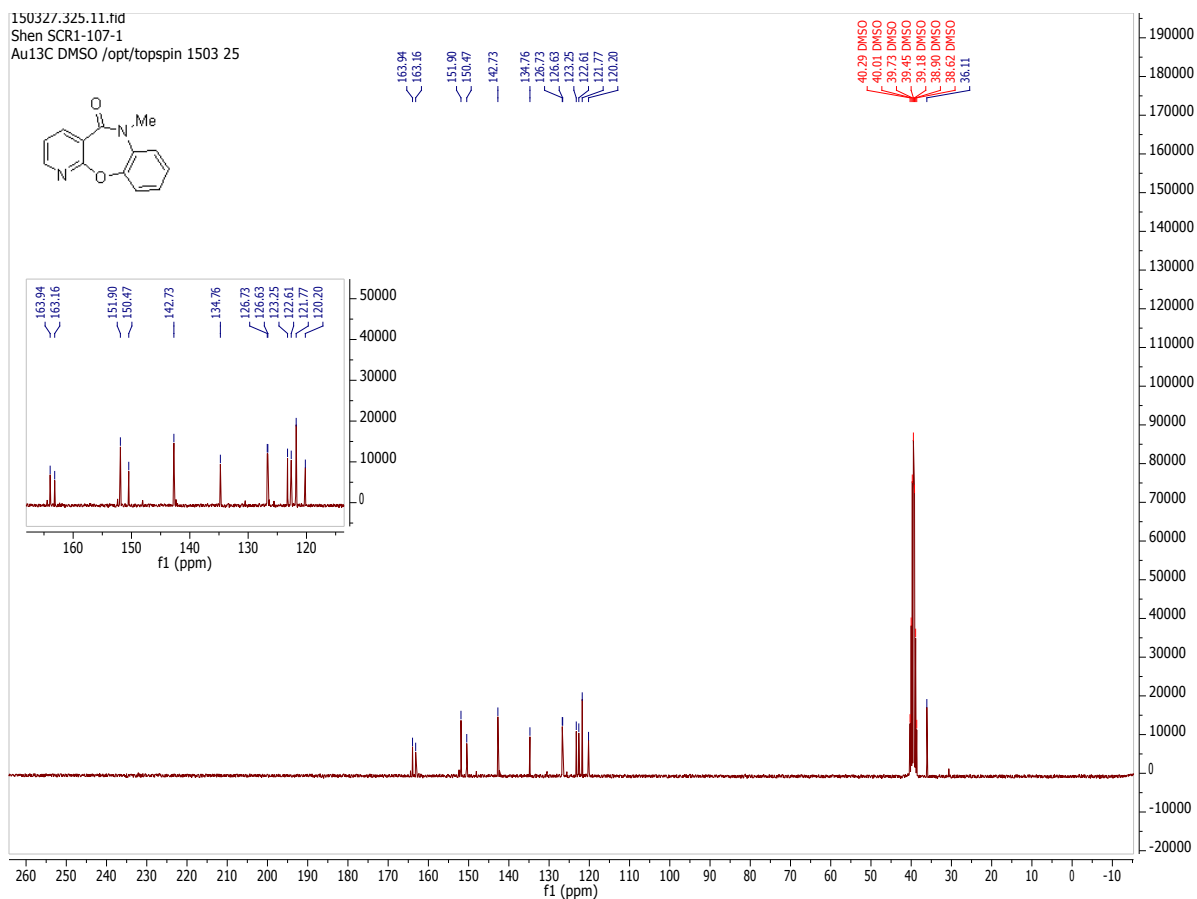
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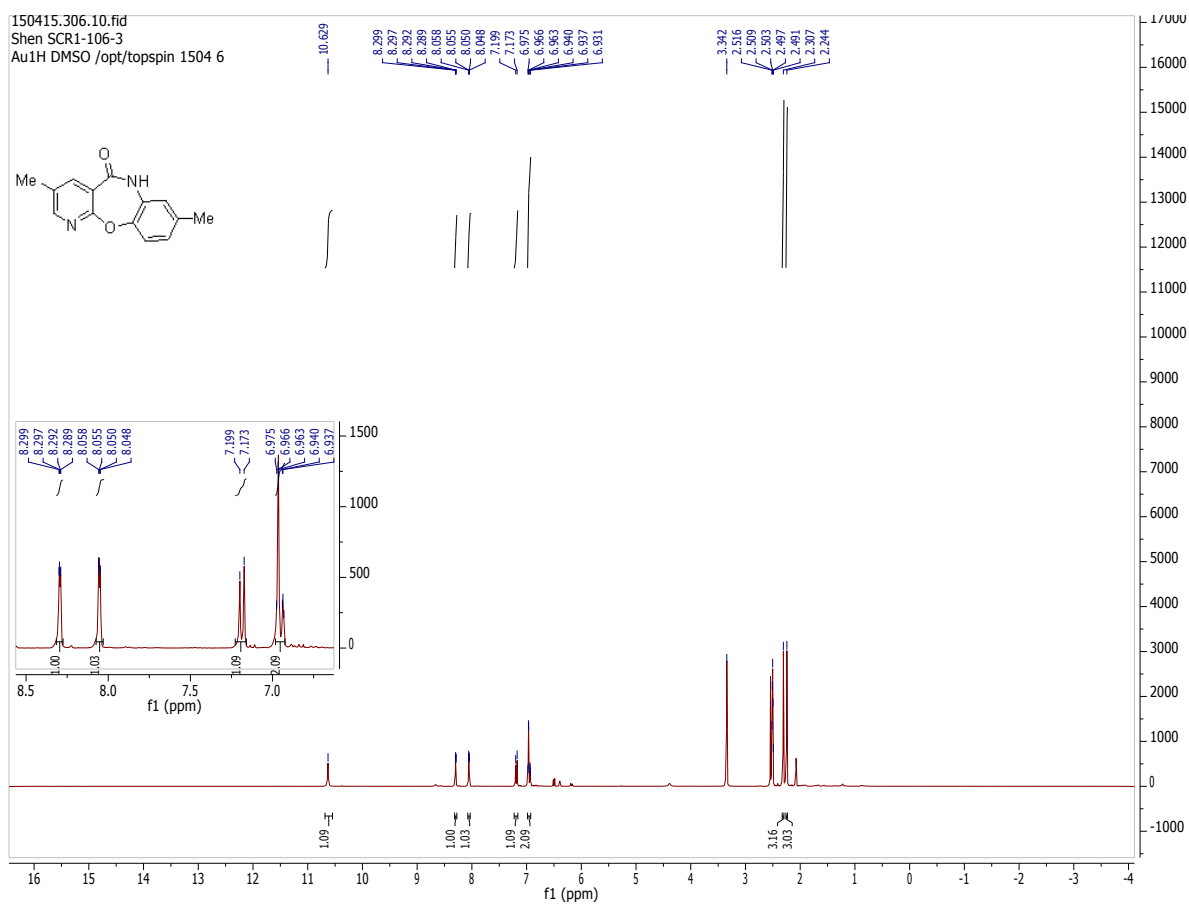
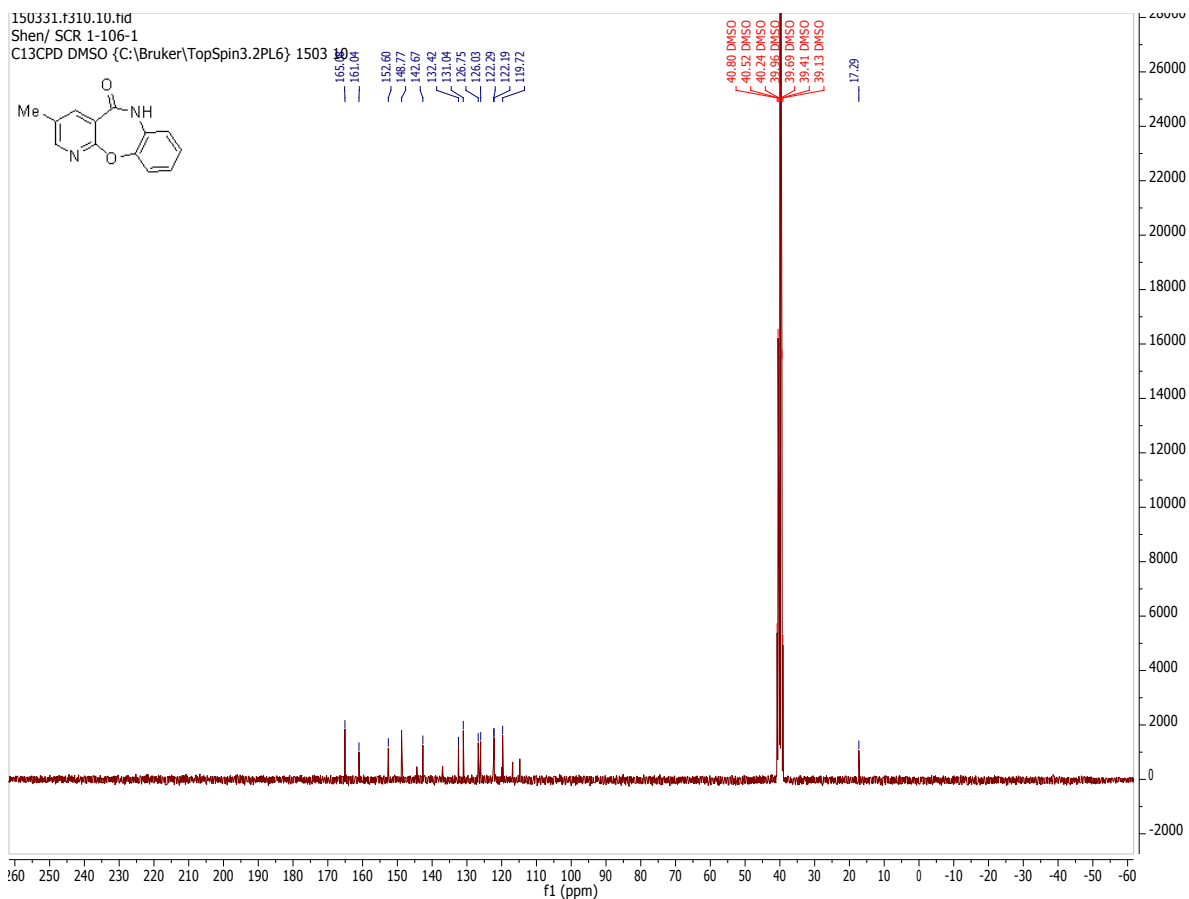


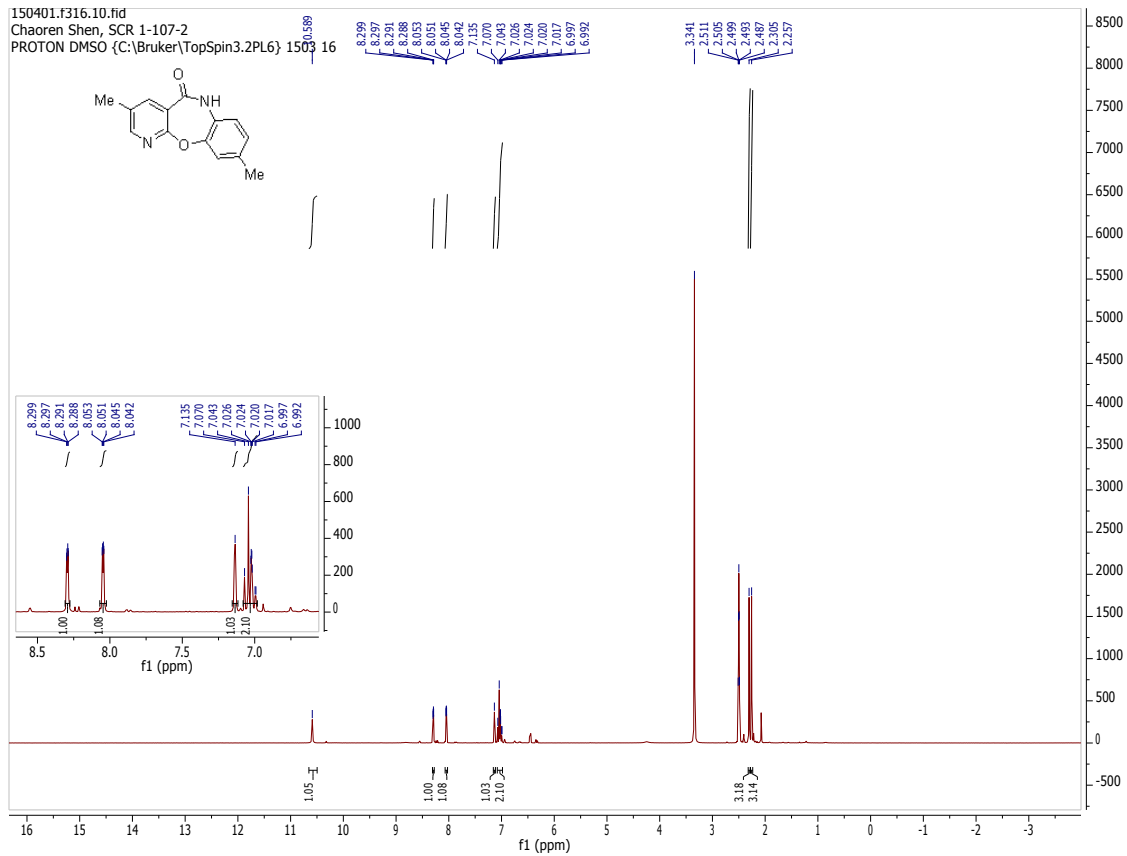
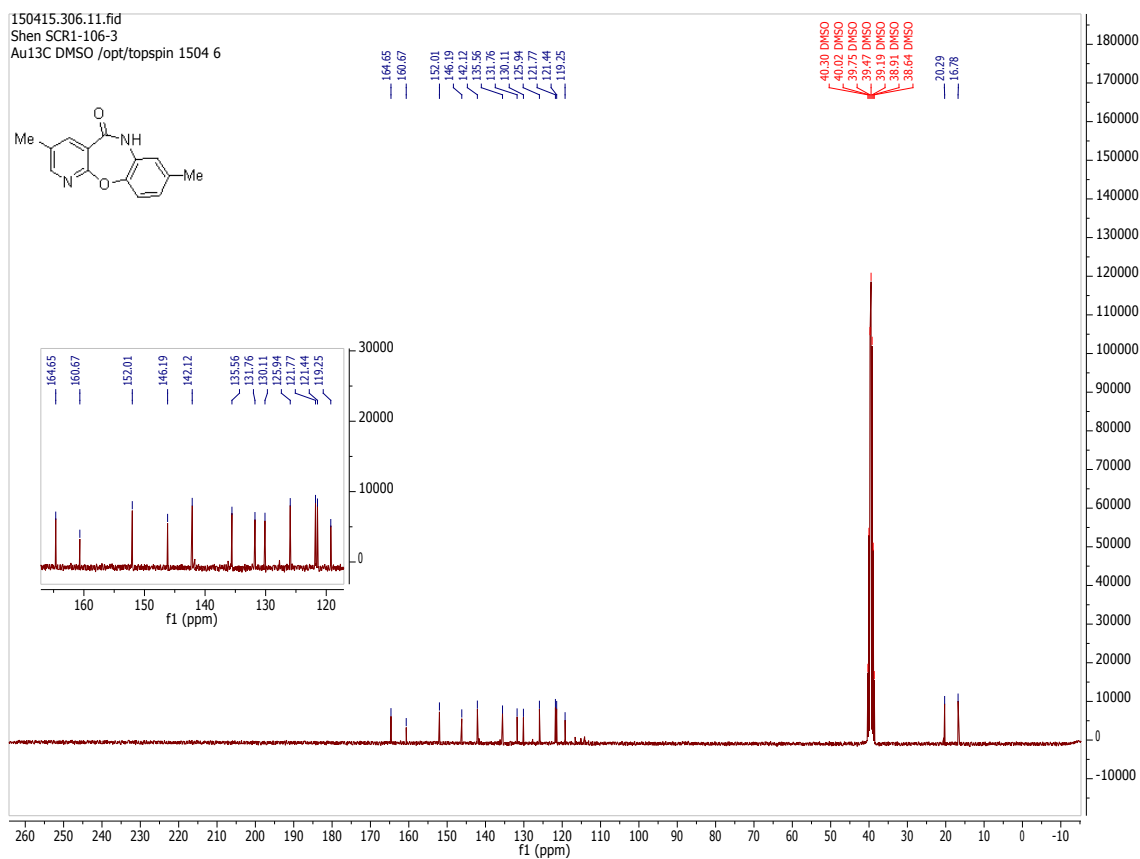


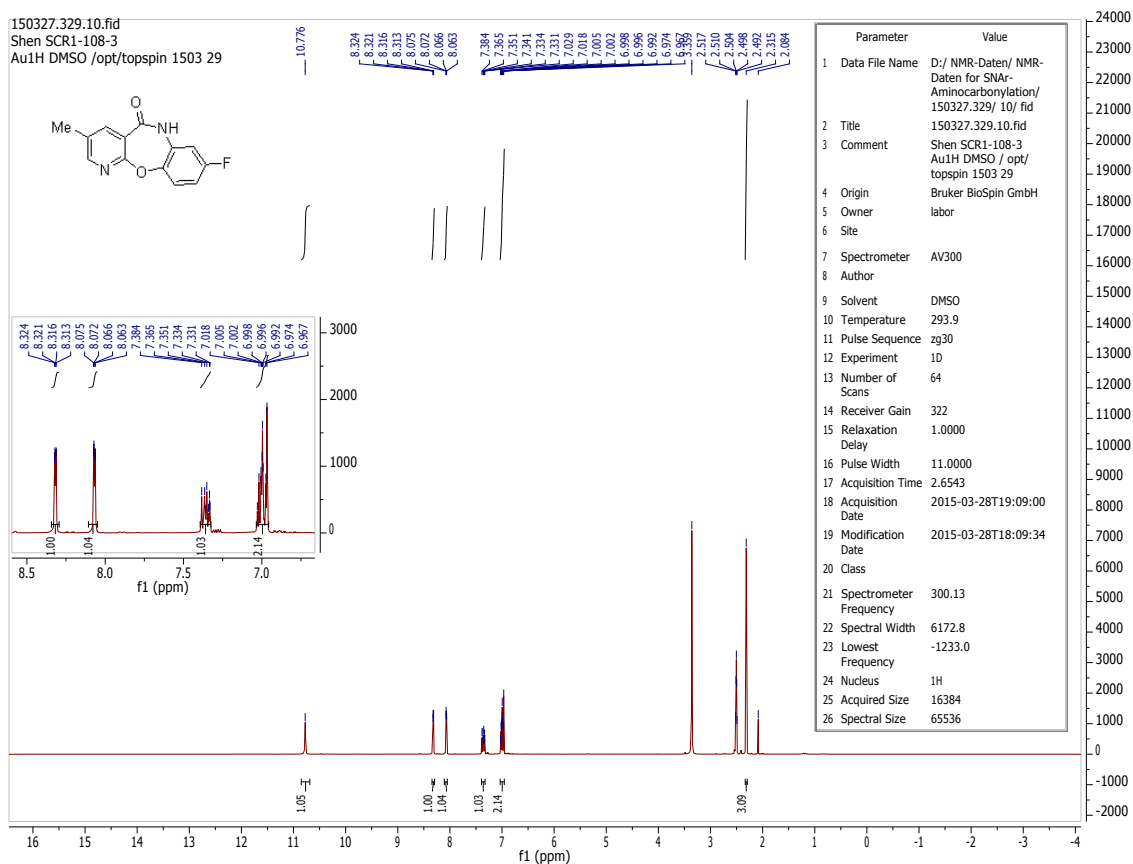
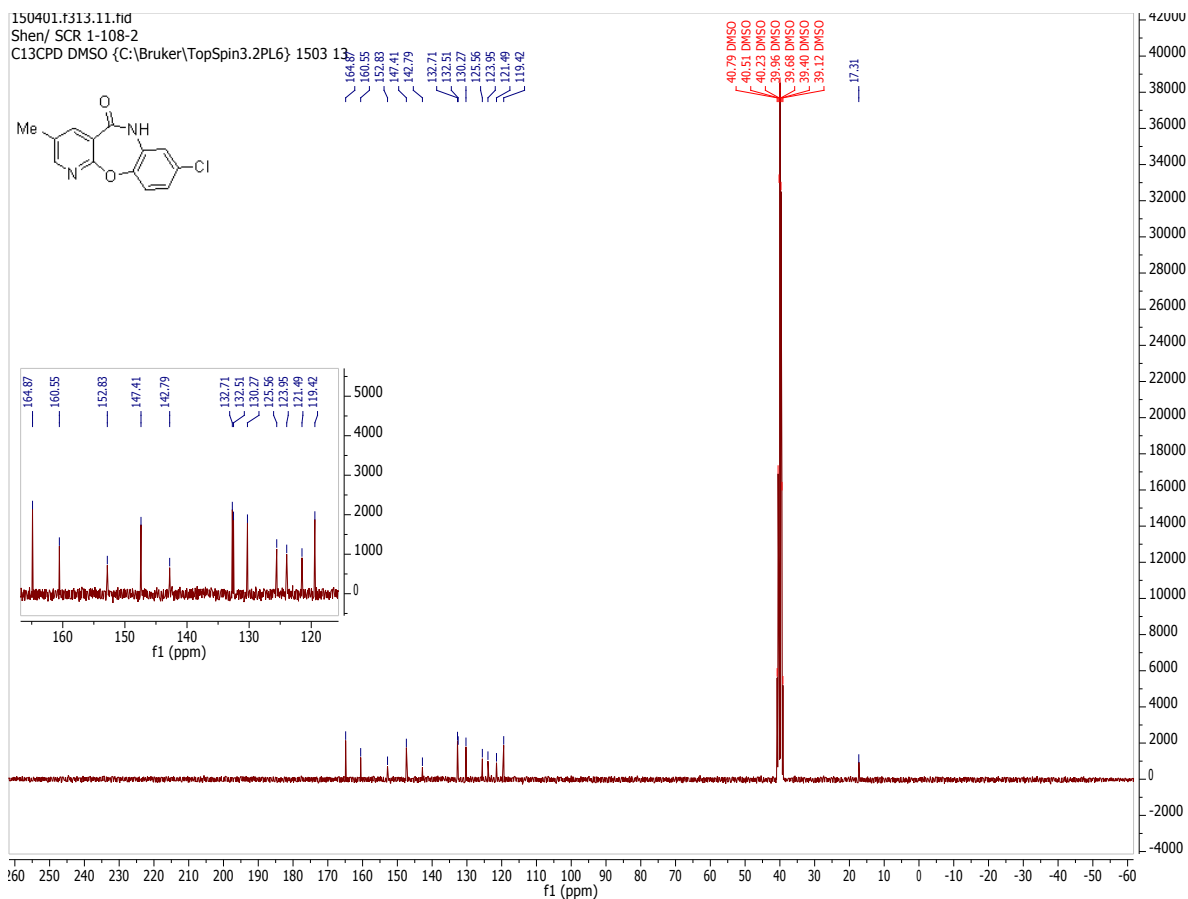


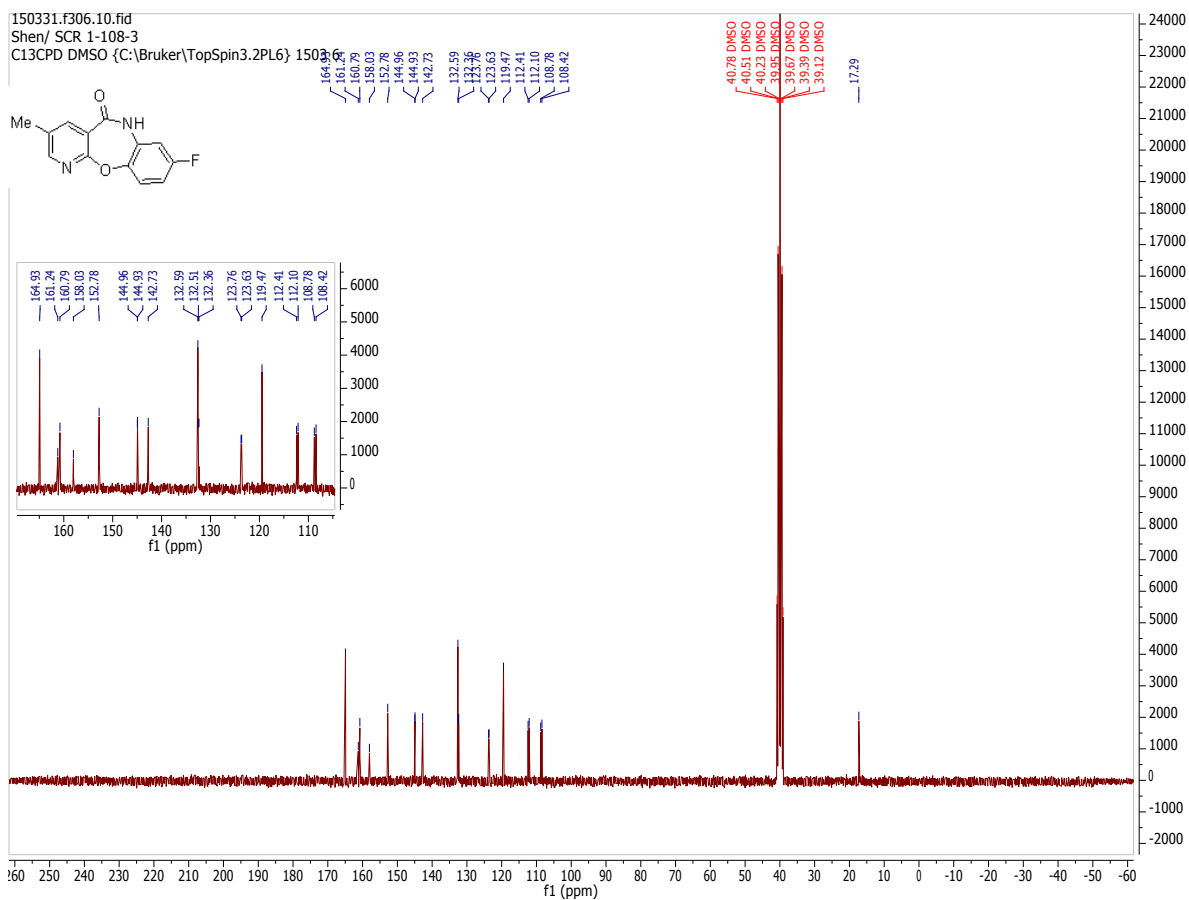
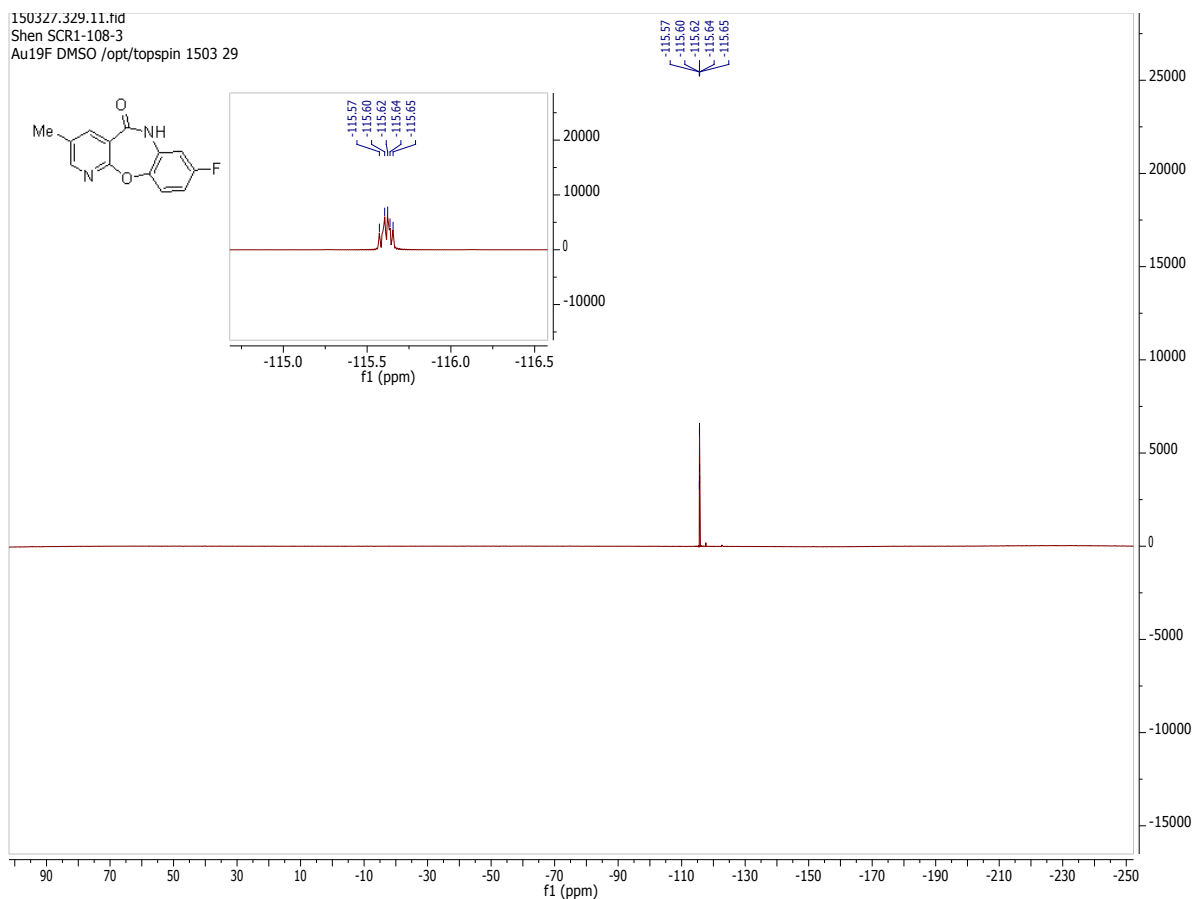


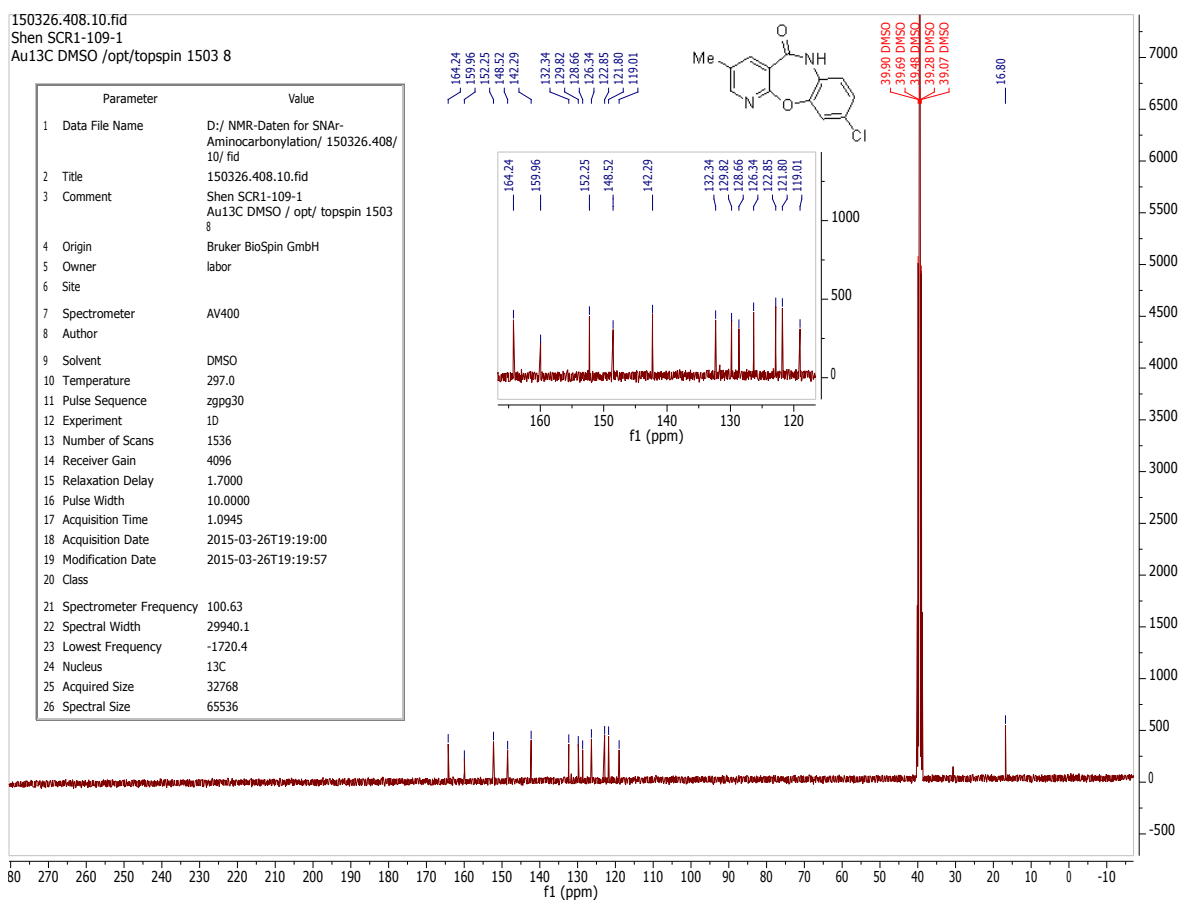
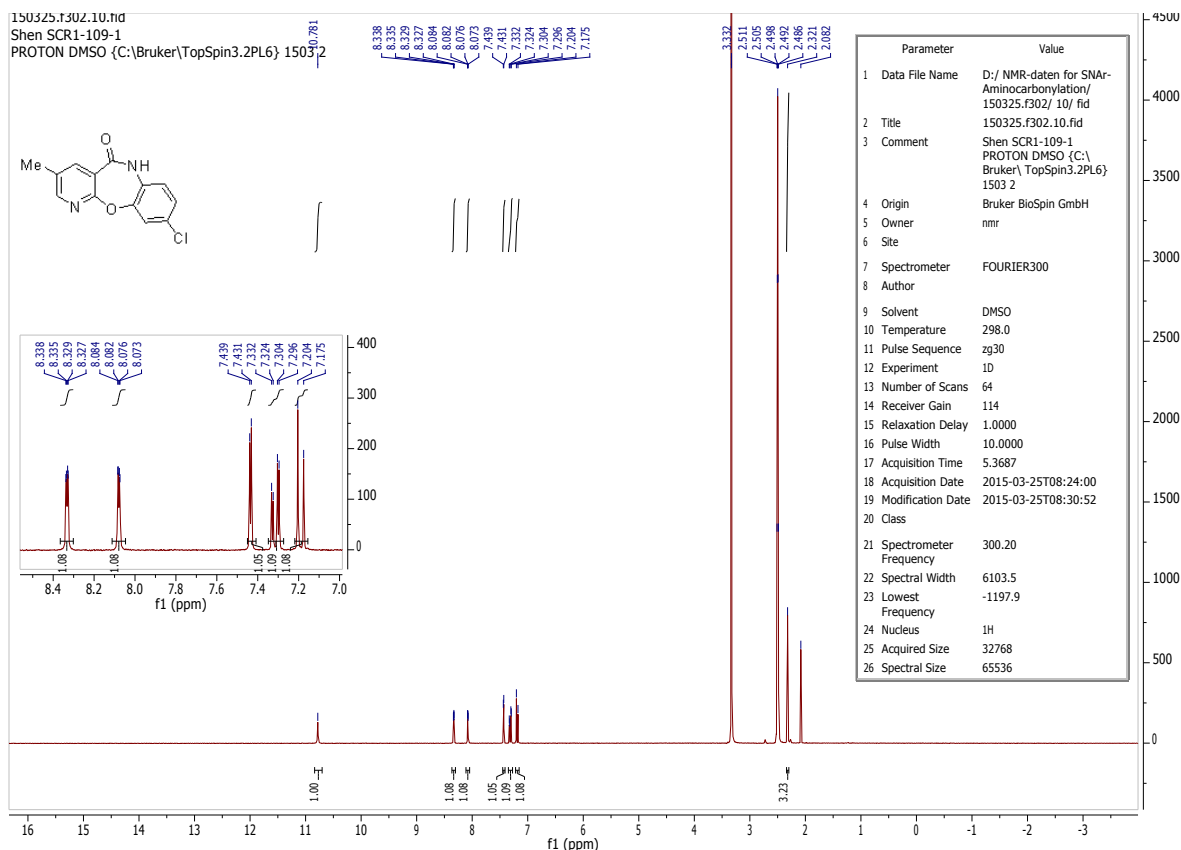


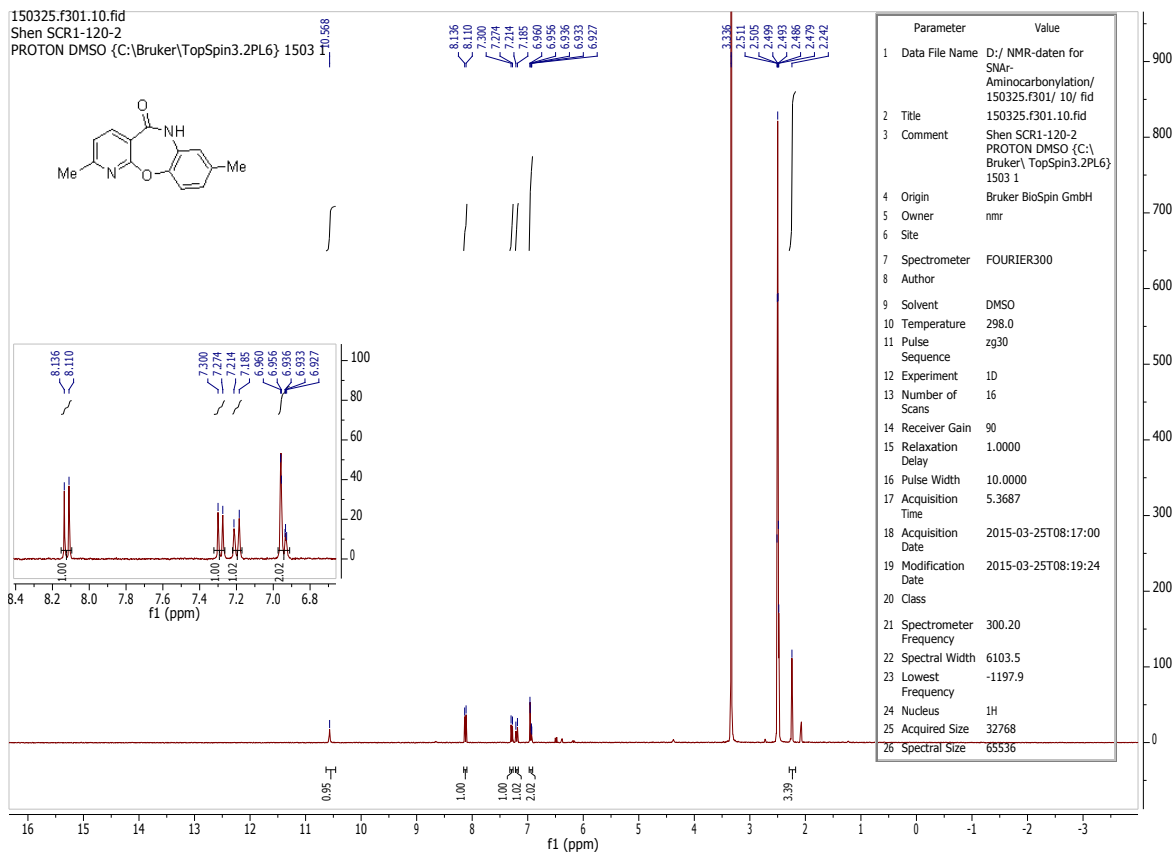
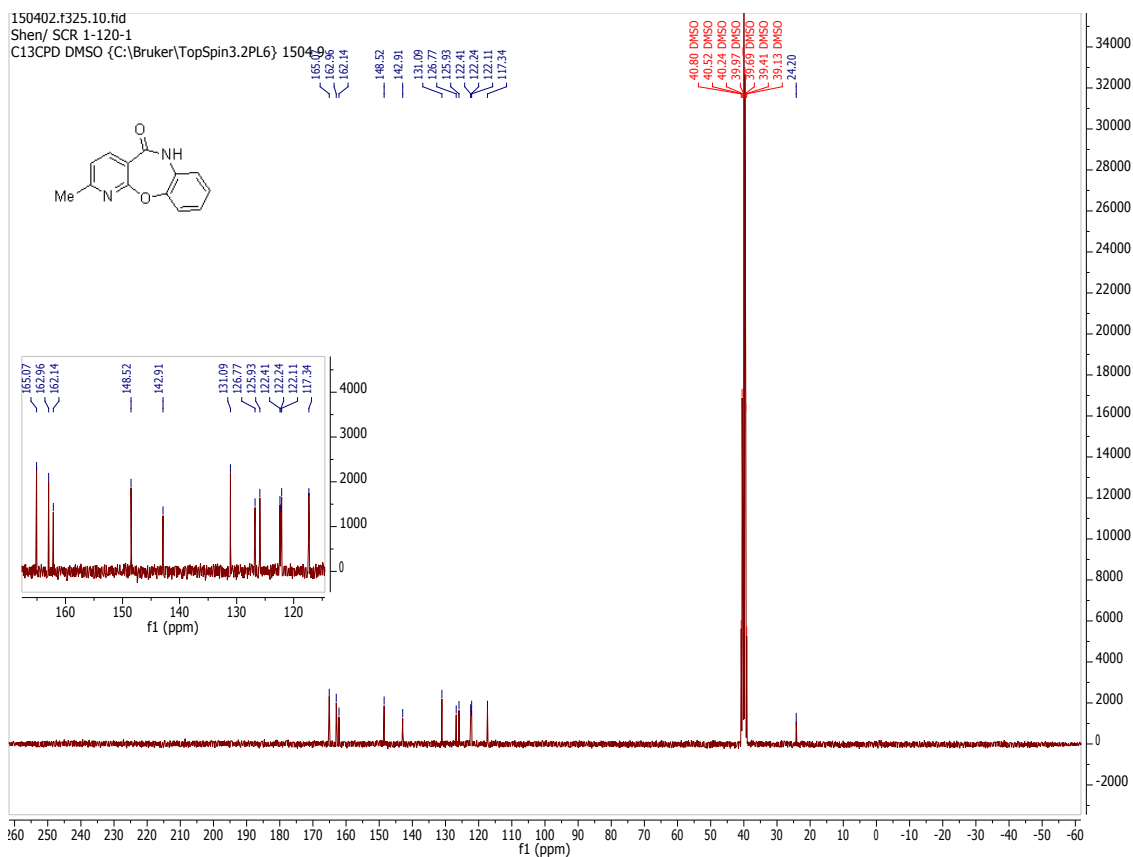


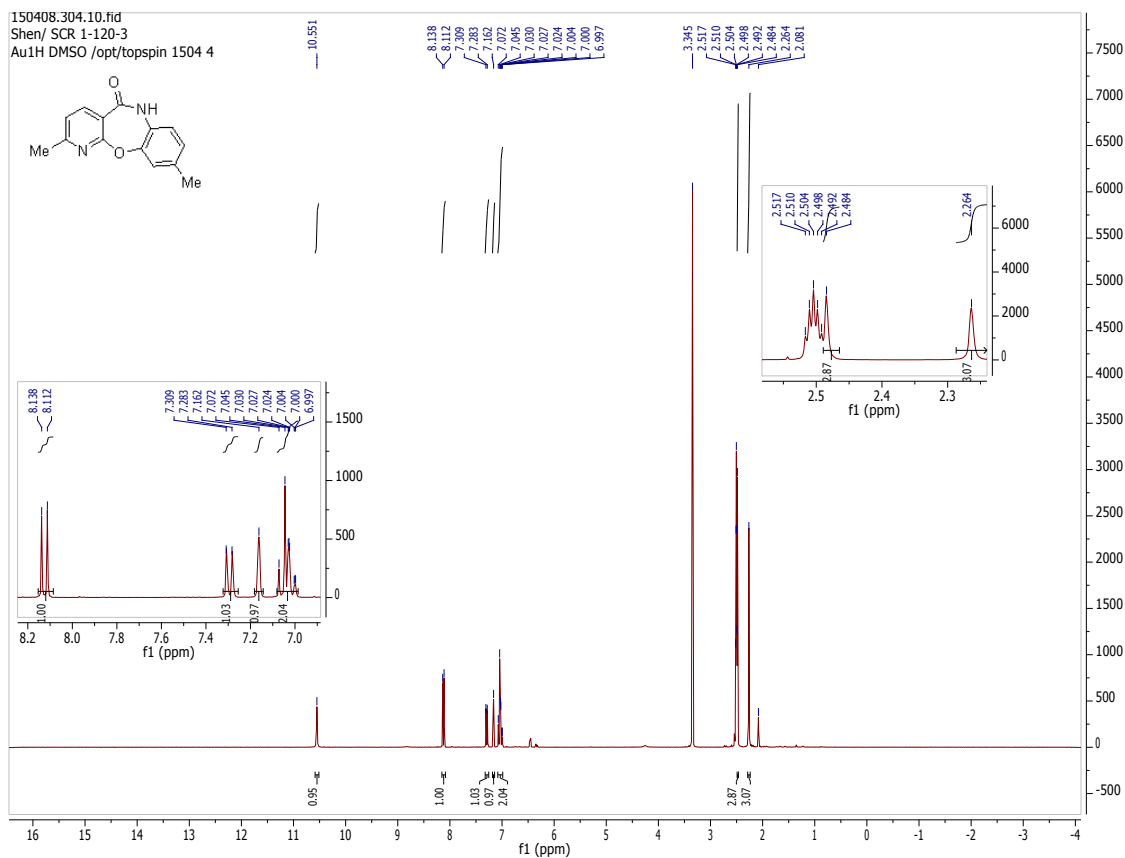
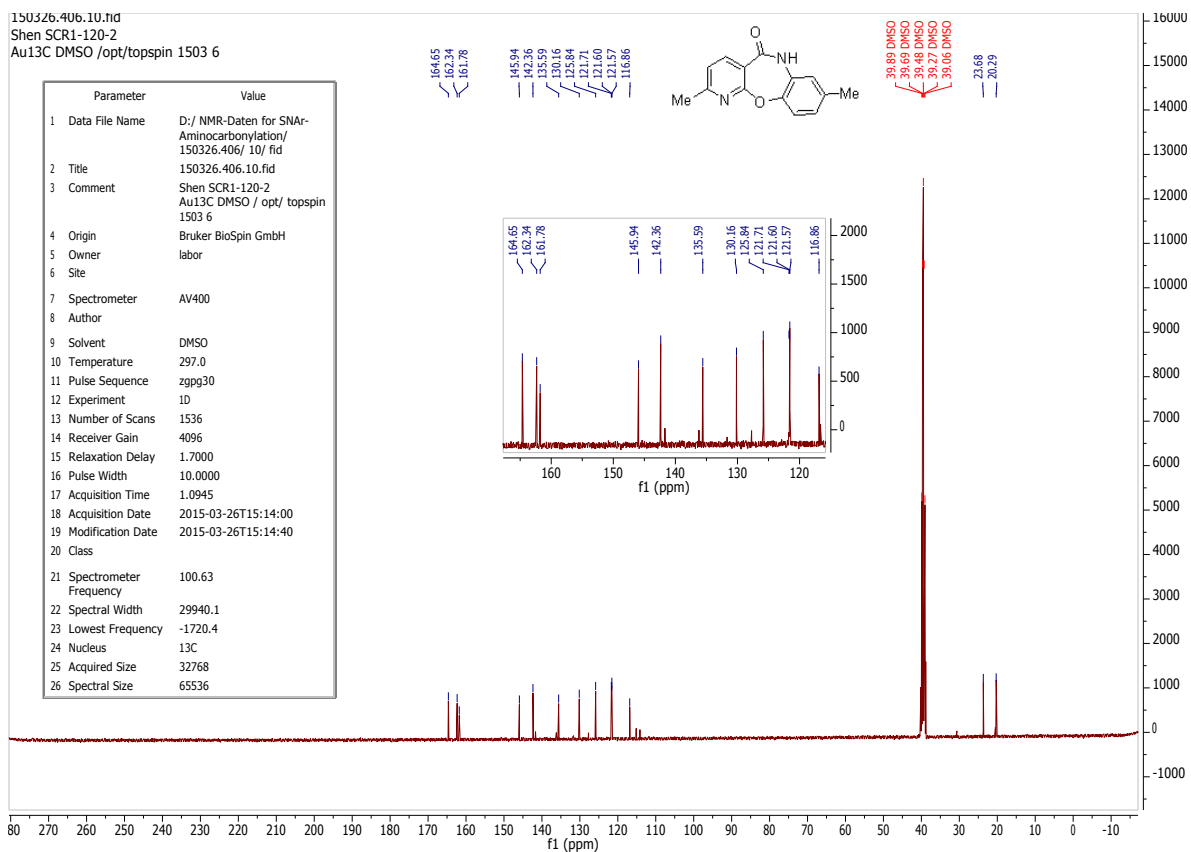


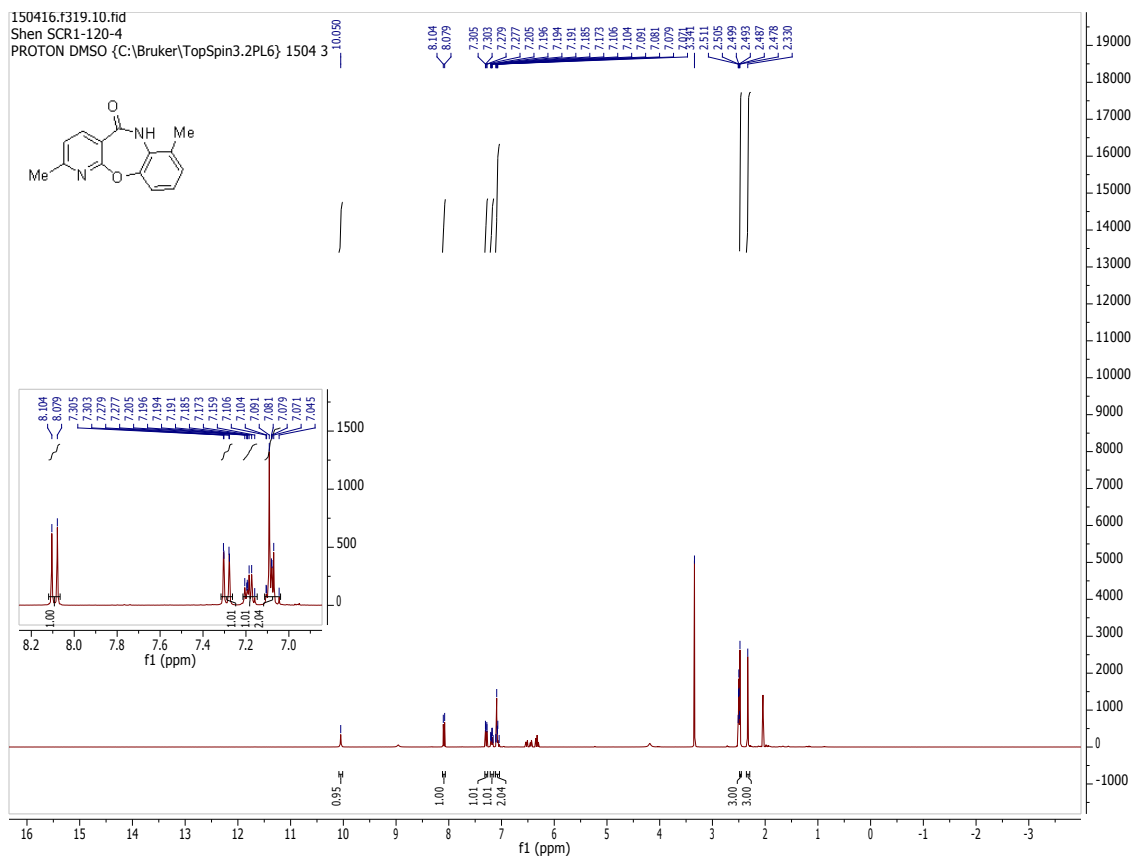
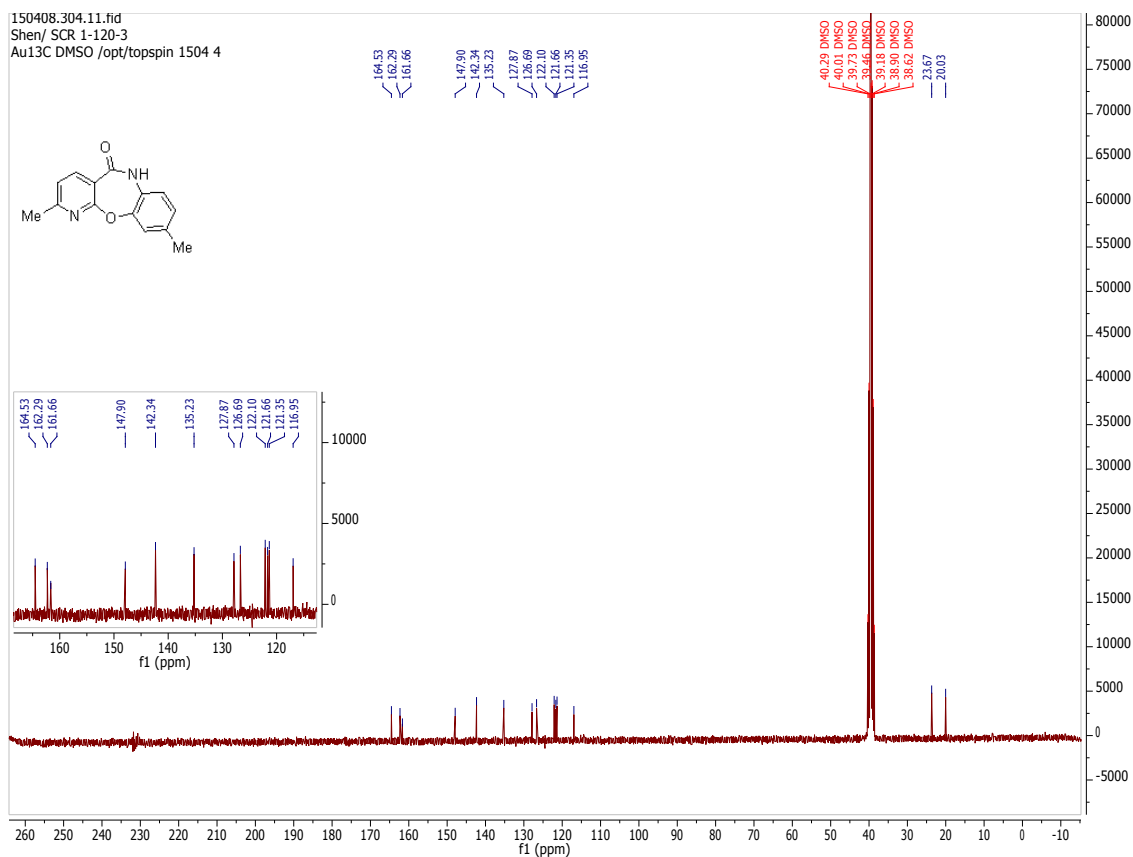


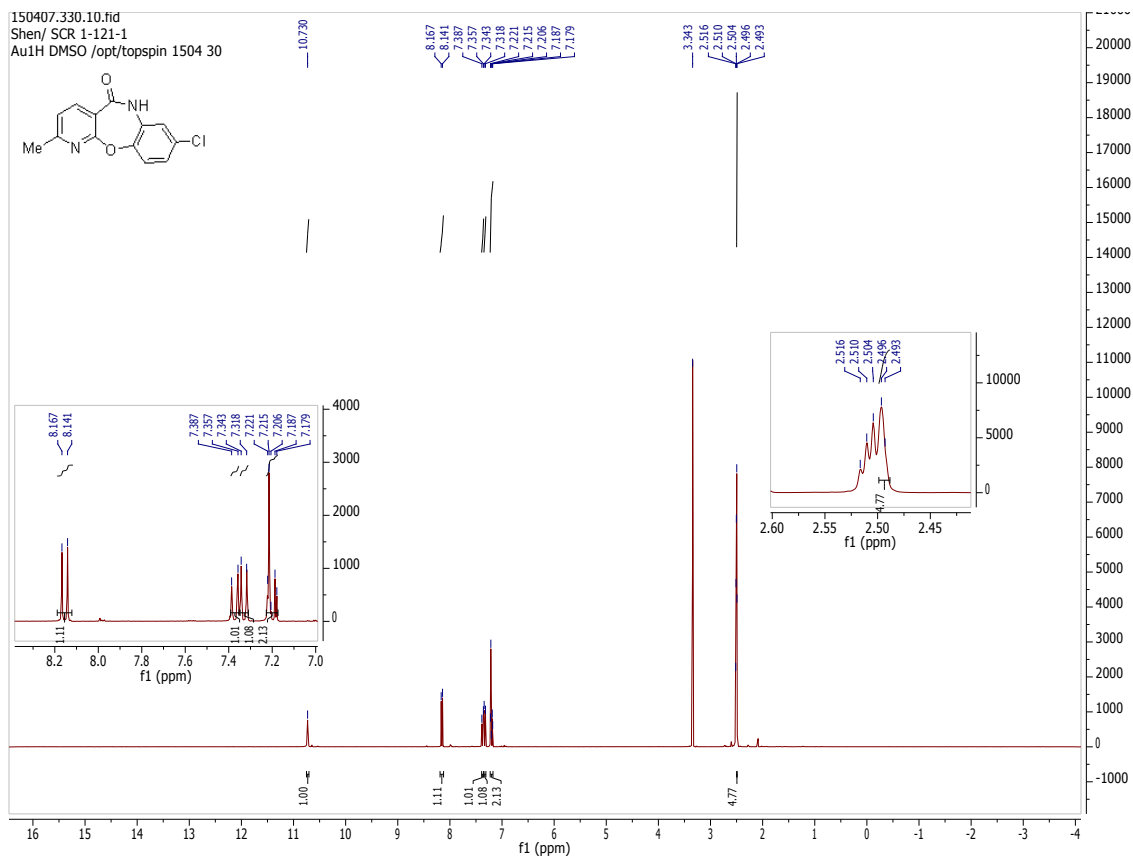
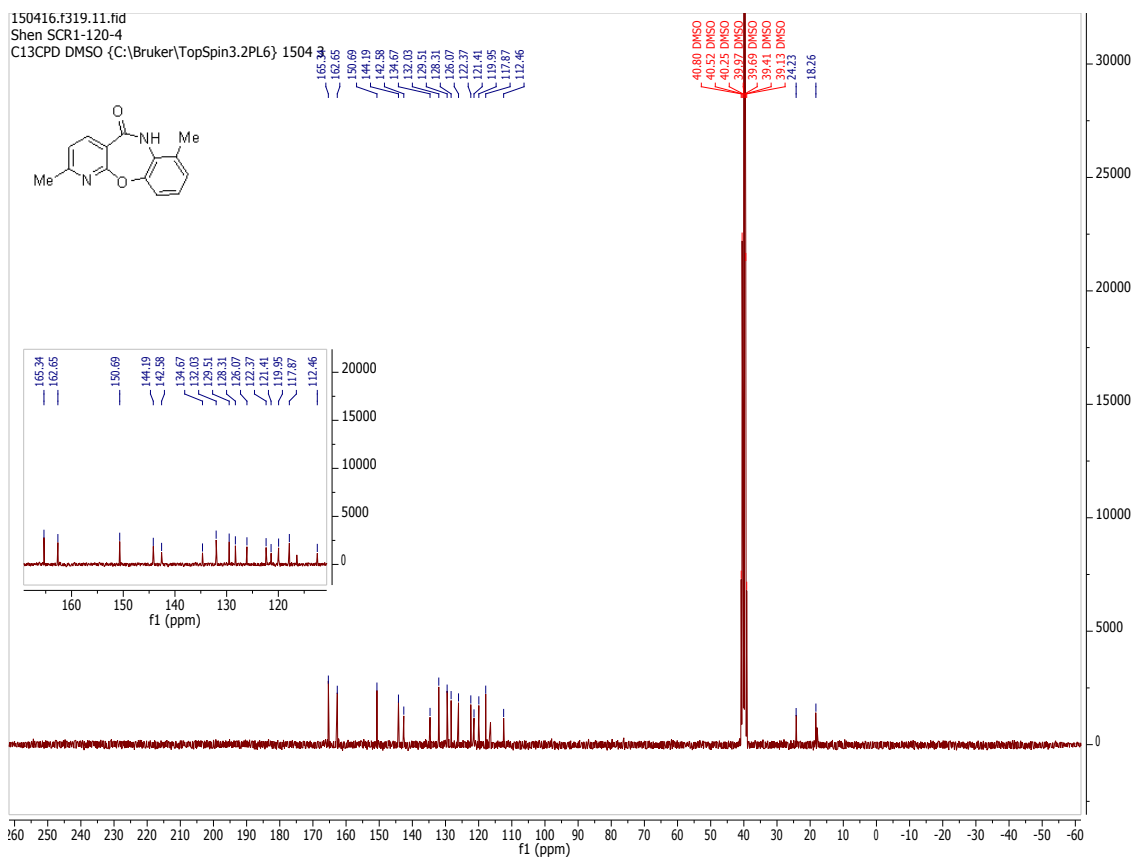


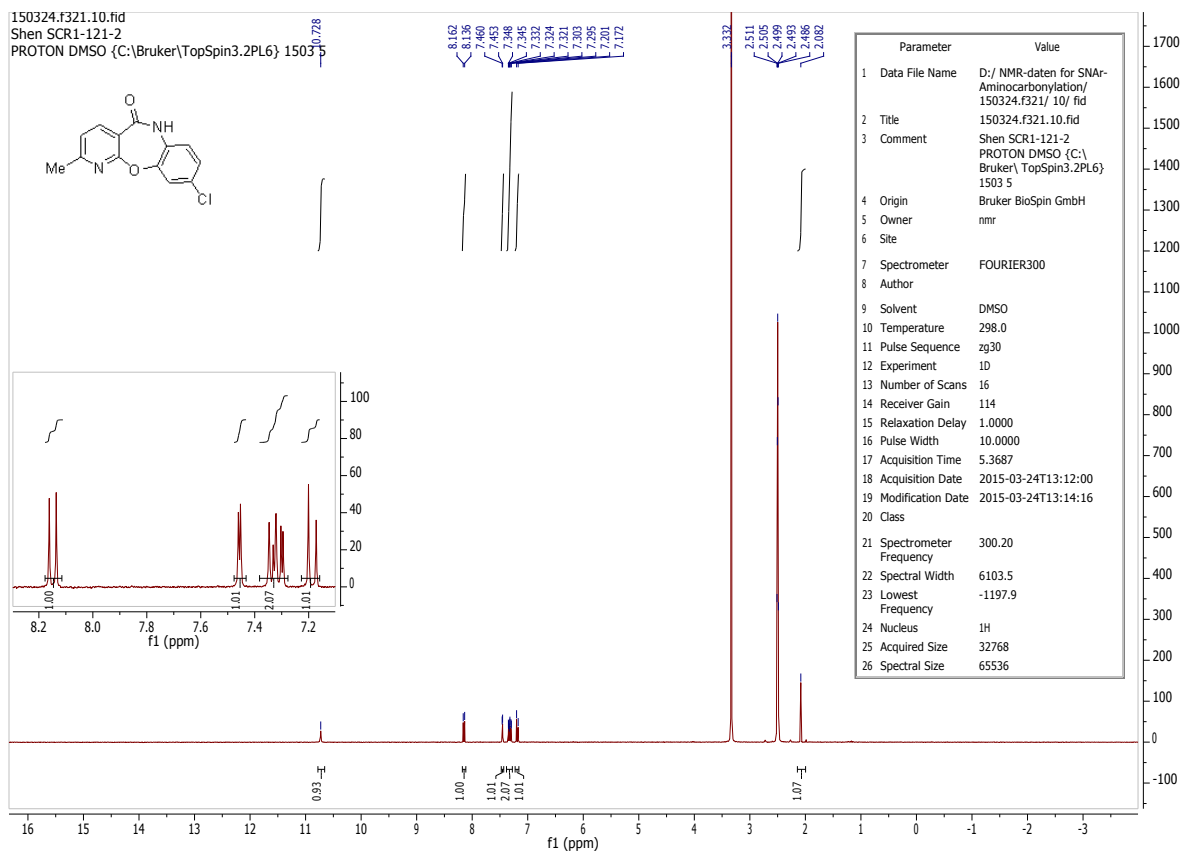
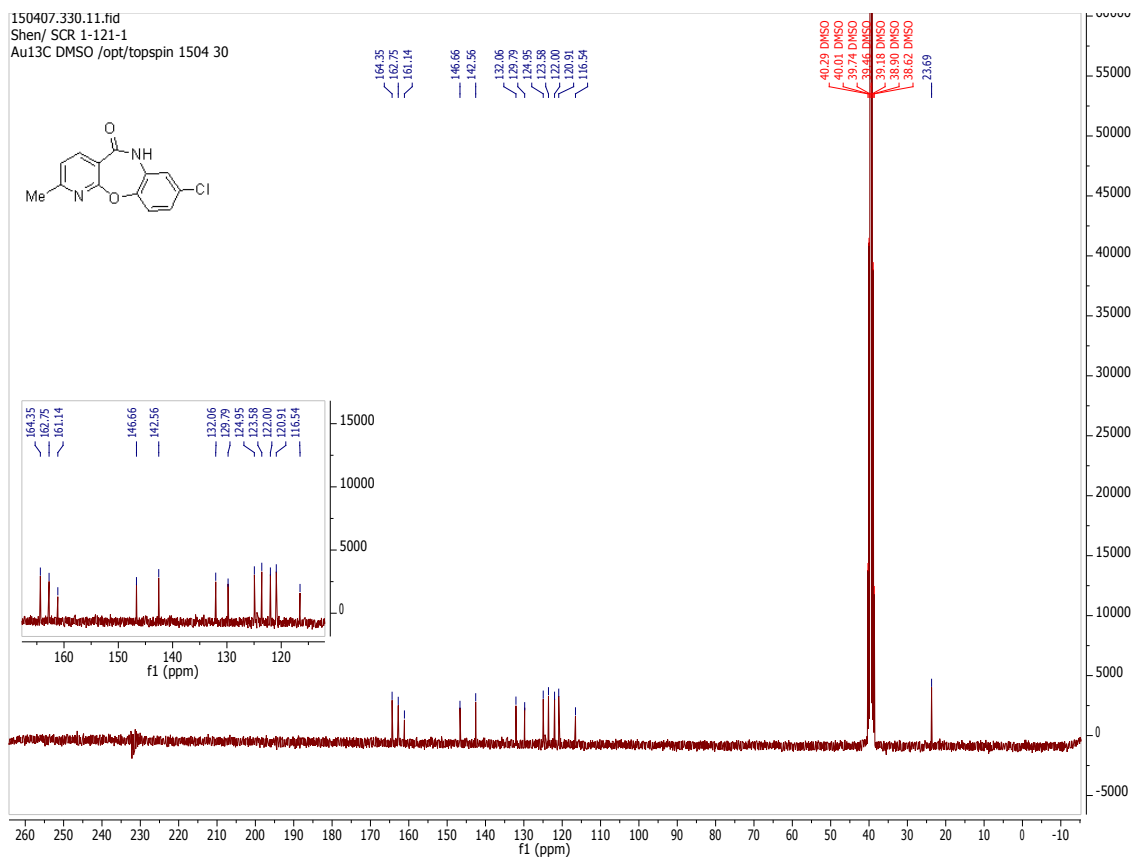








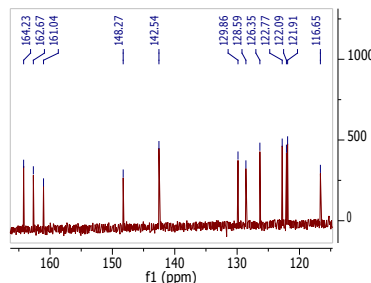
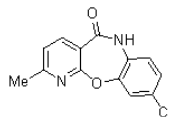




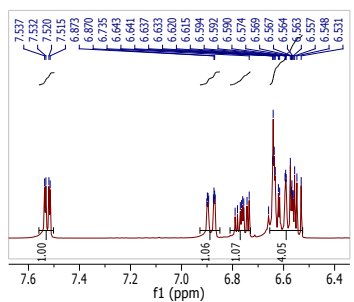
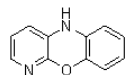
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5 Owner	labor
6 Site	
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8 Author	
9 Solvent	DMSO
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12 Experiment	1D
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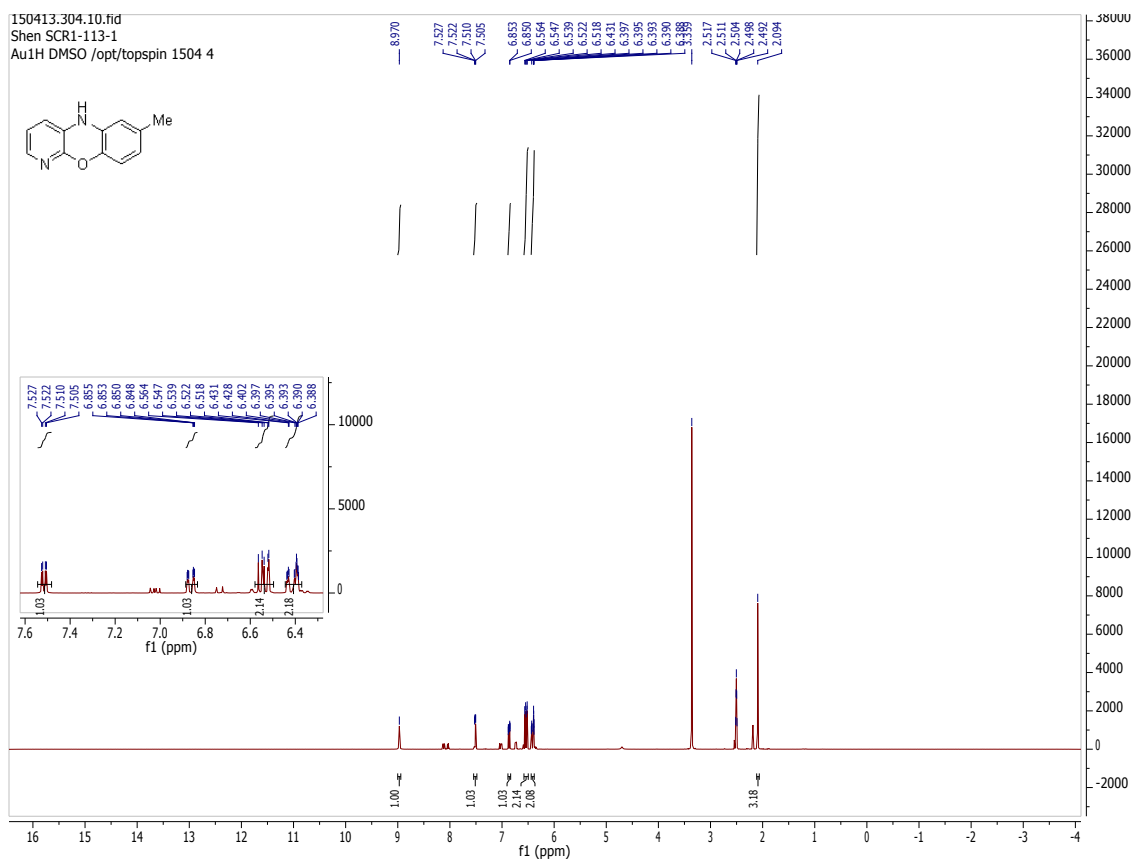


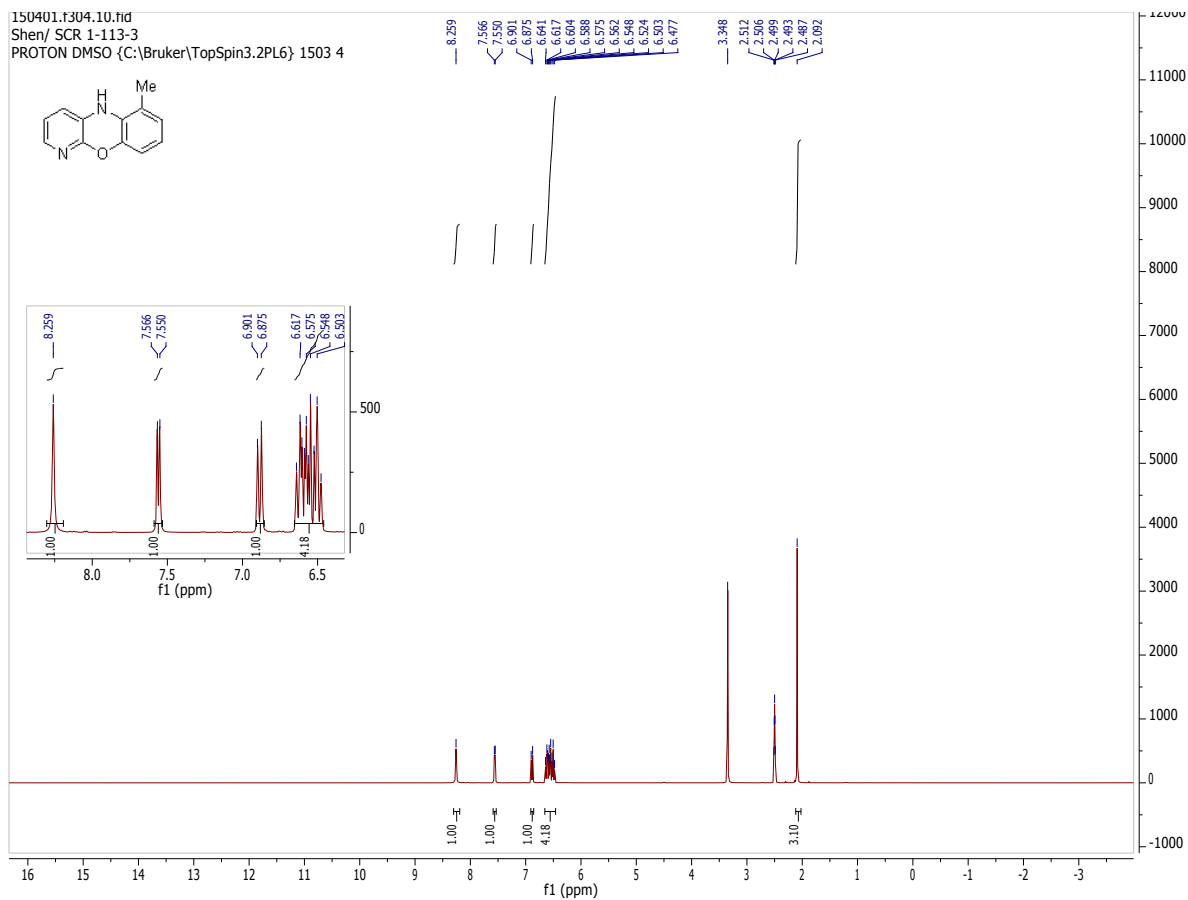
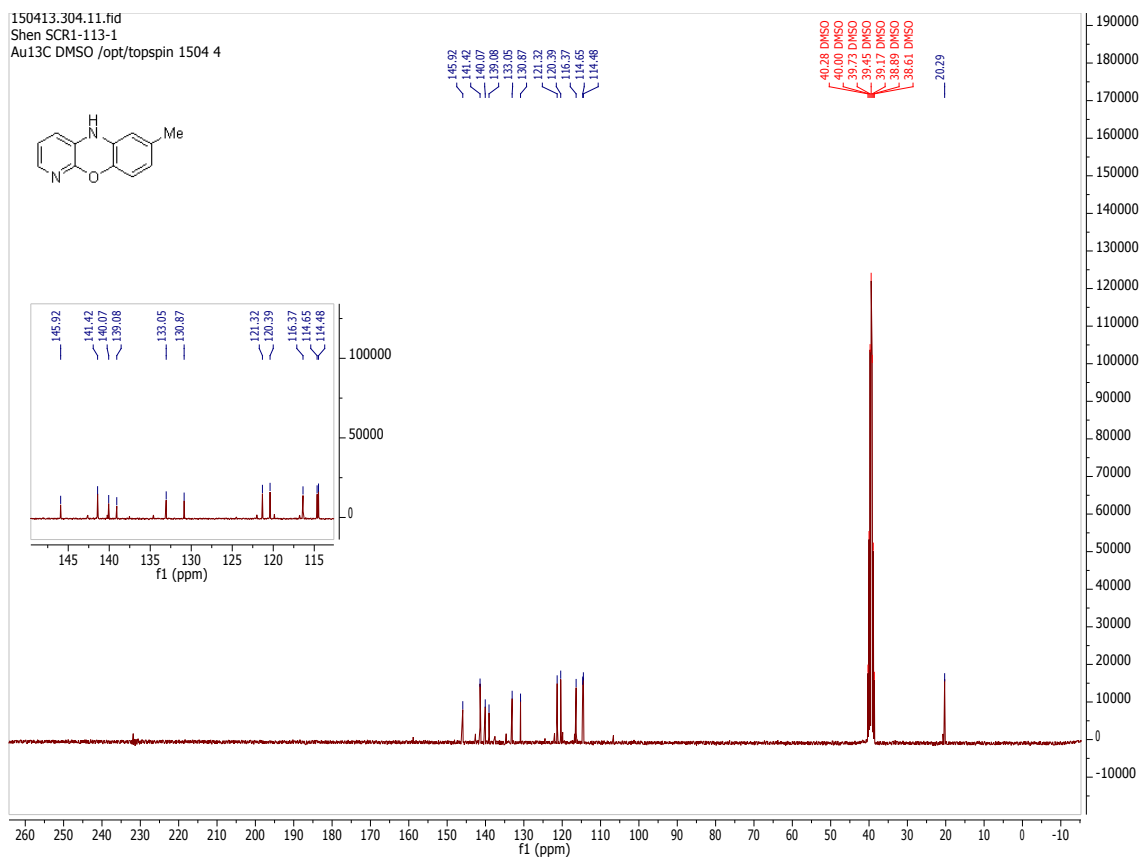
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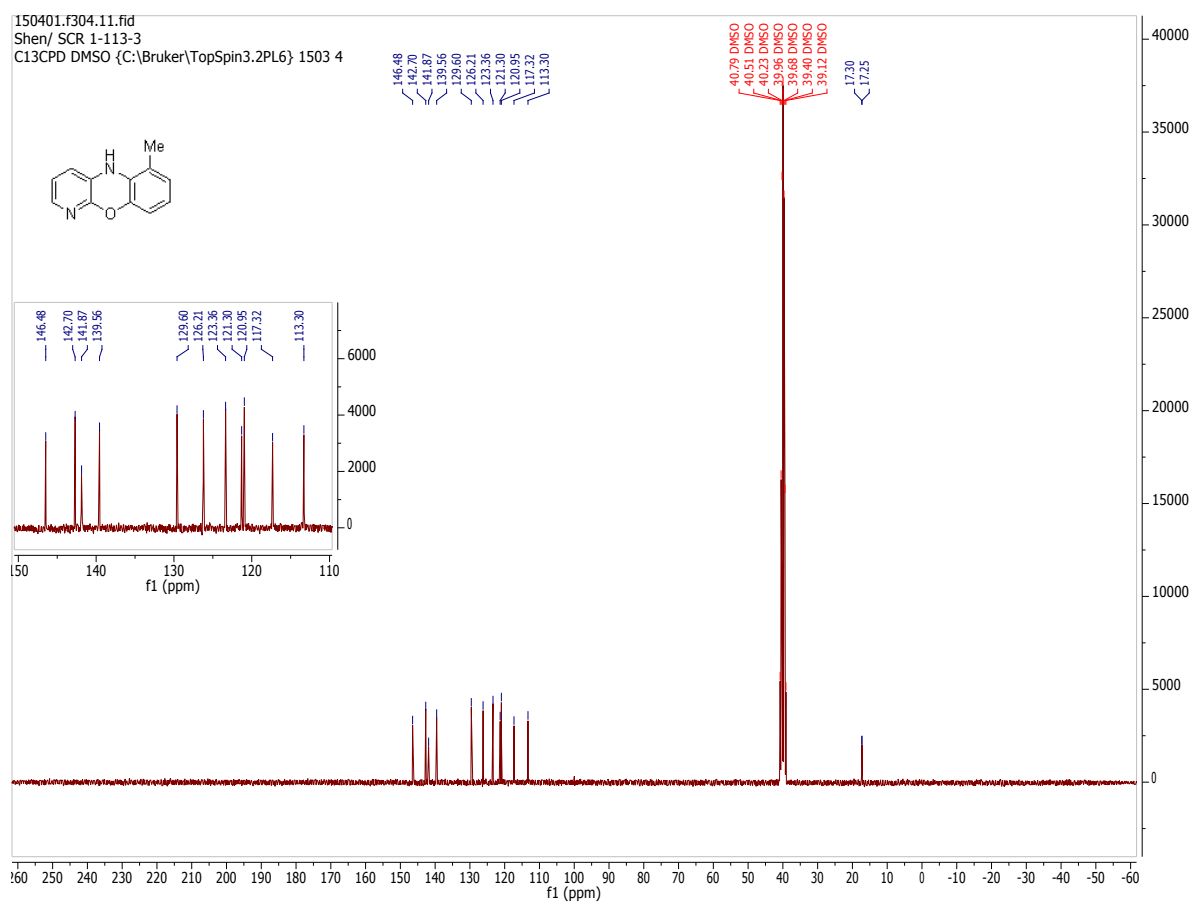
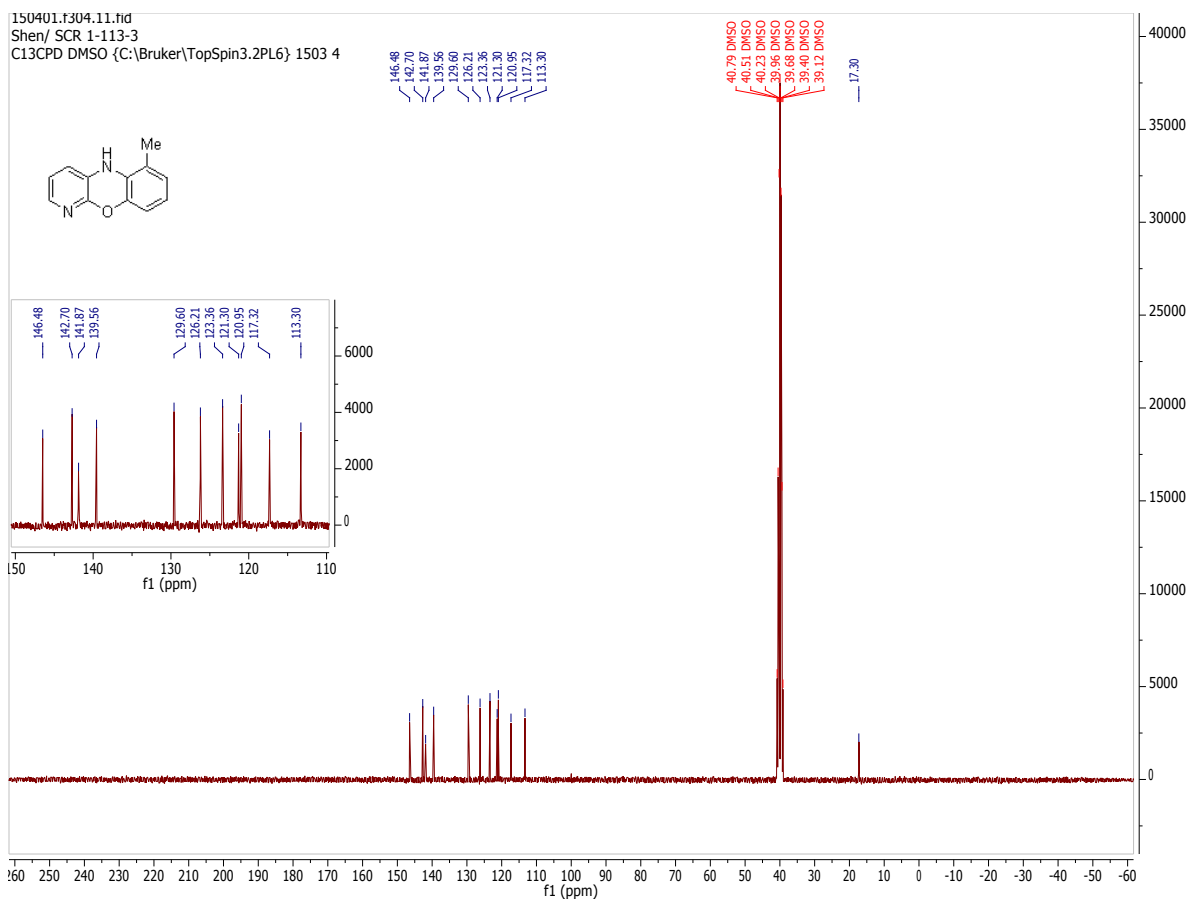
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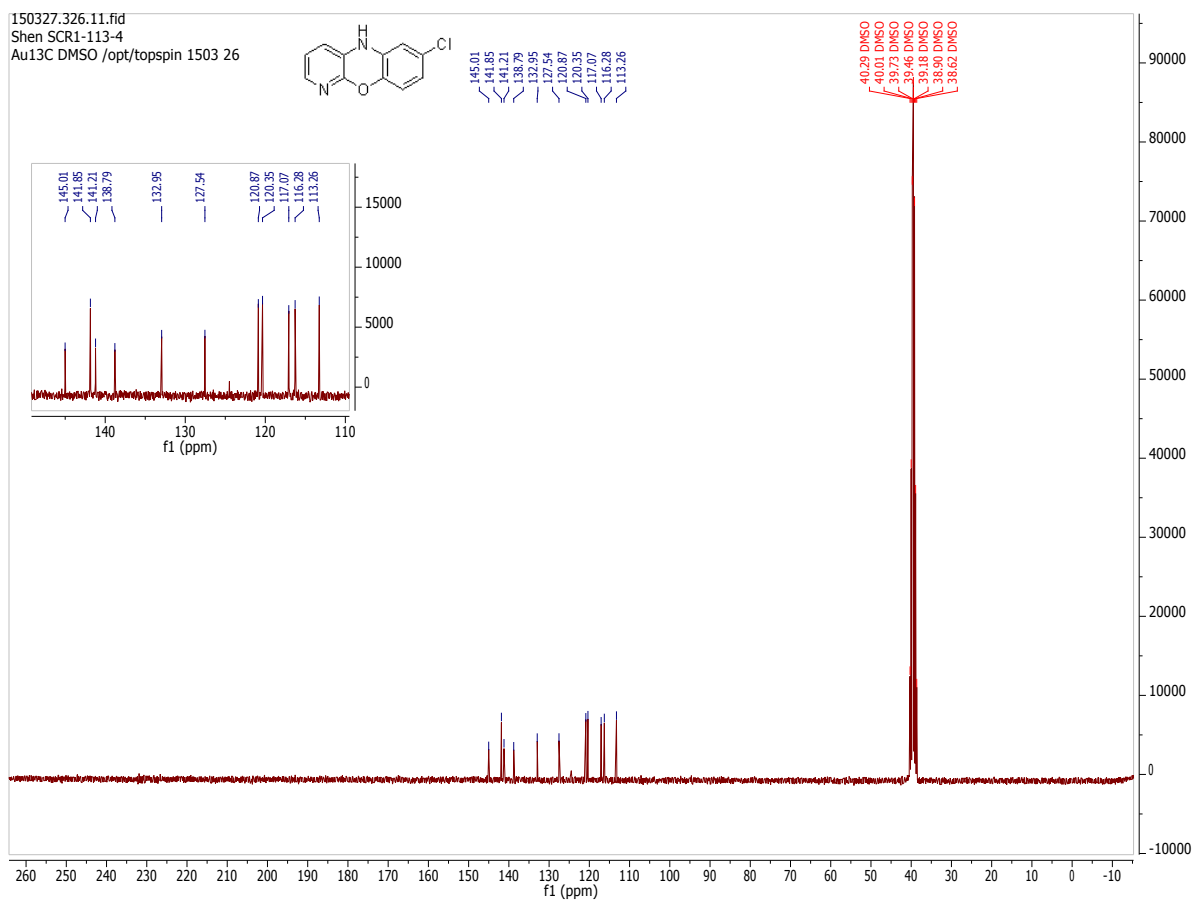
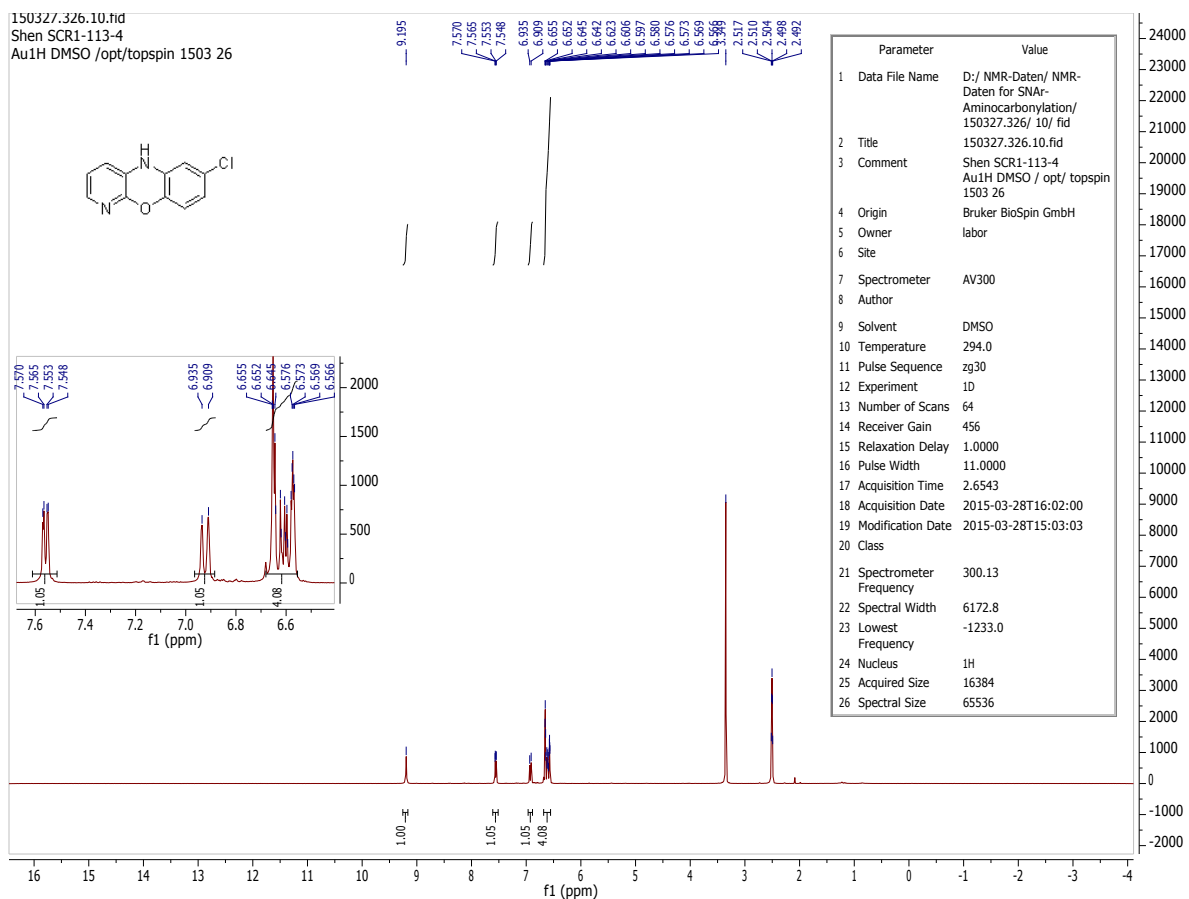
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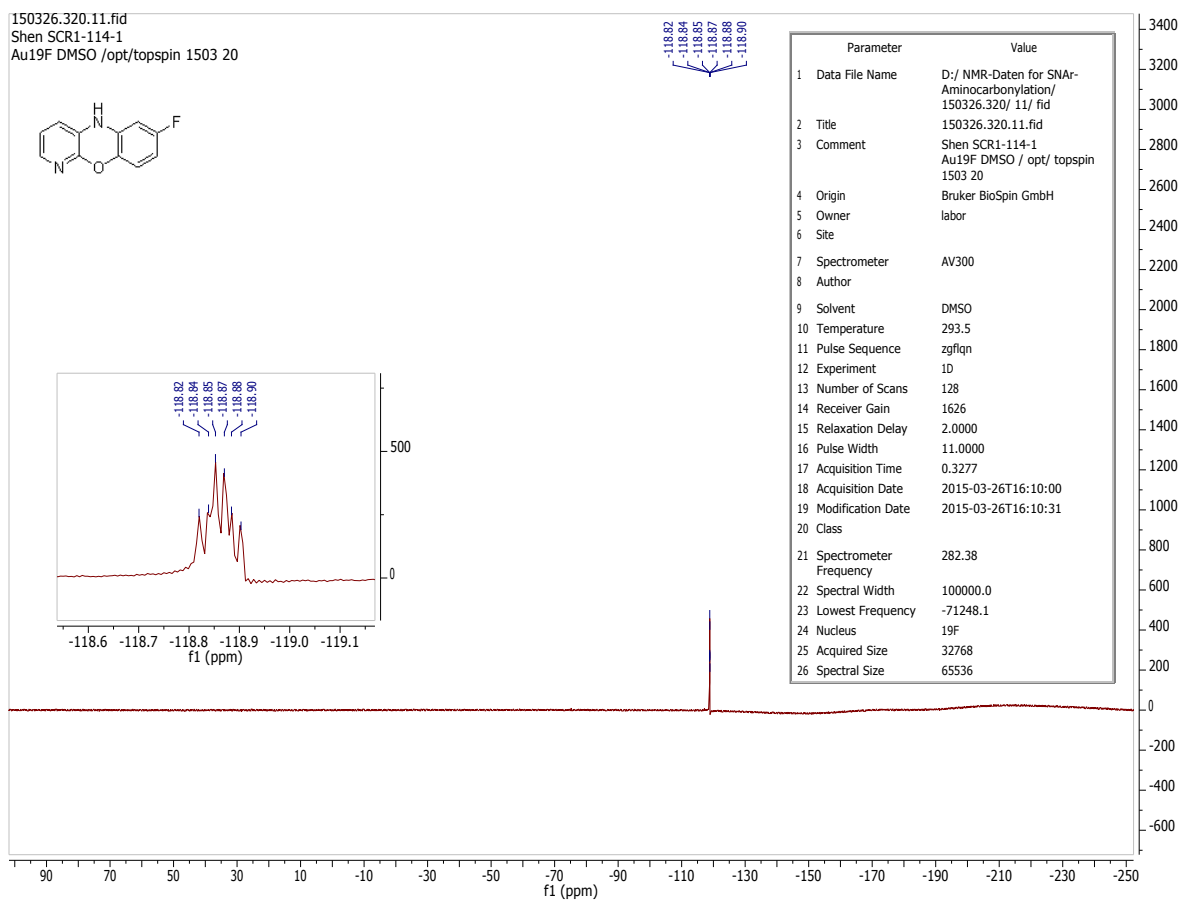
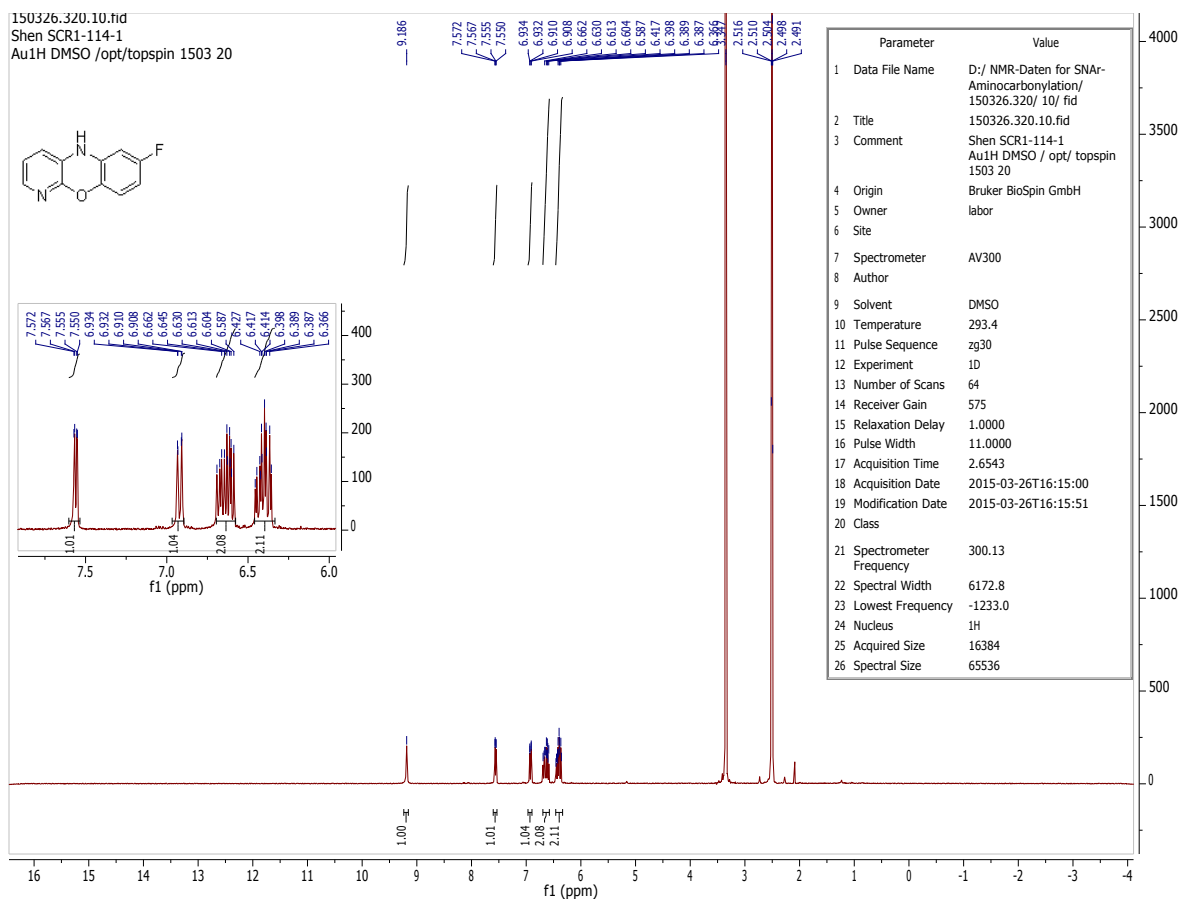
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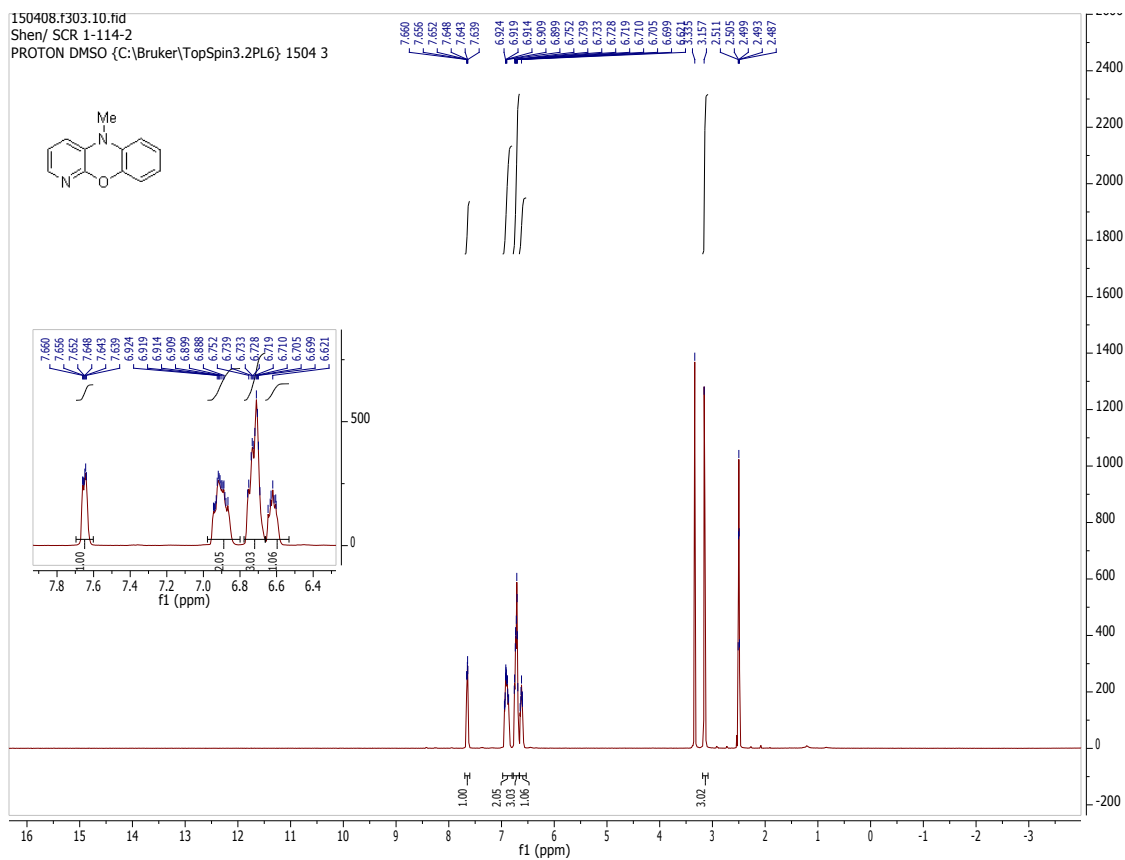
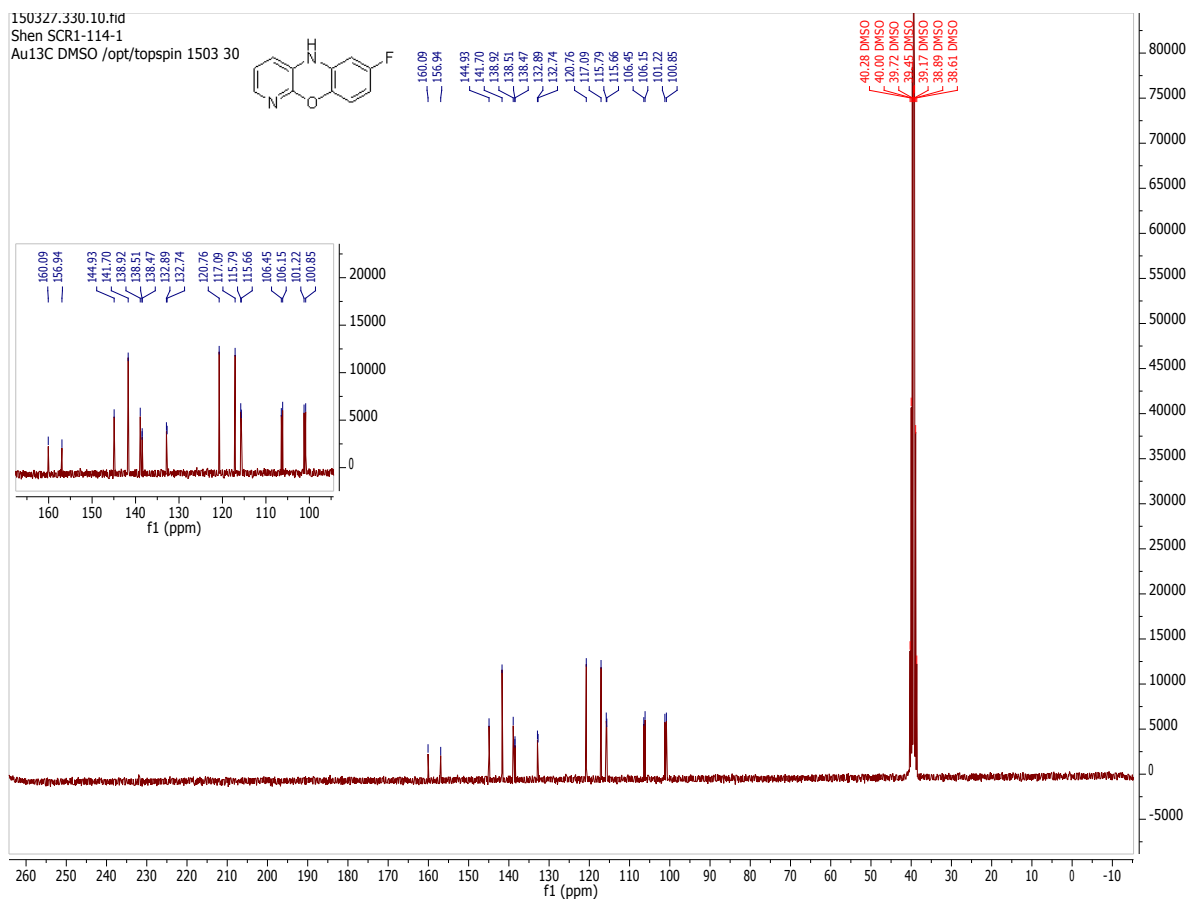


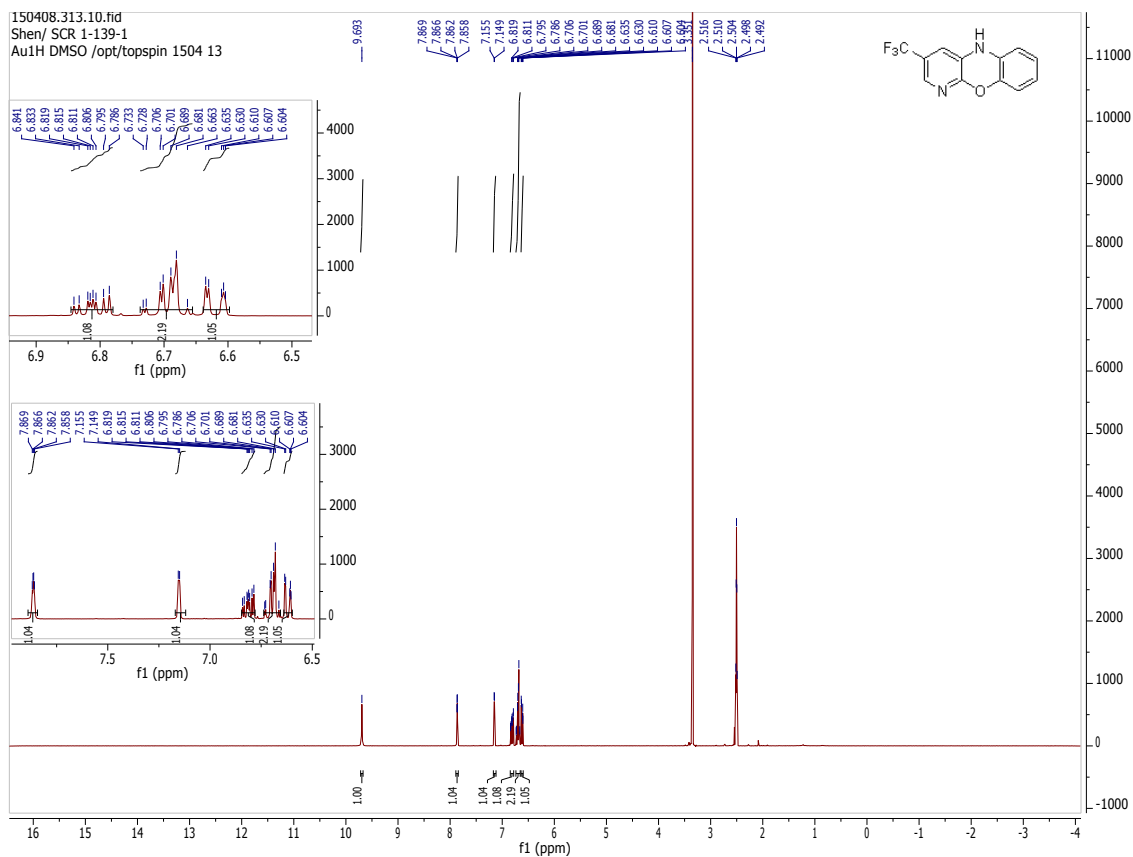
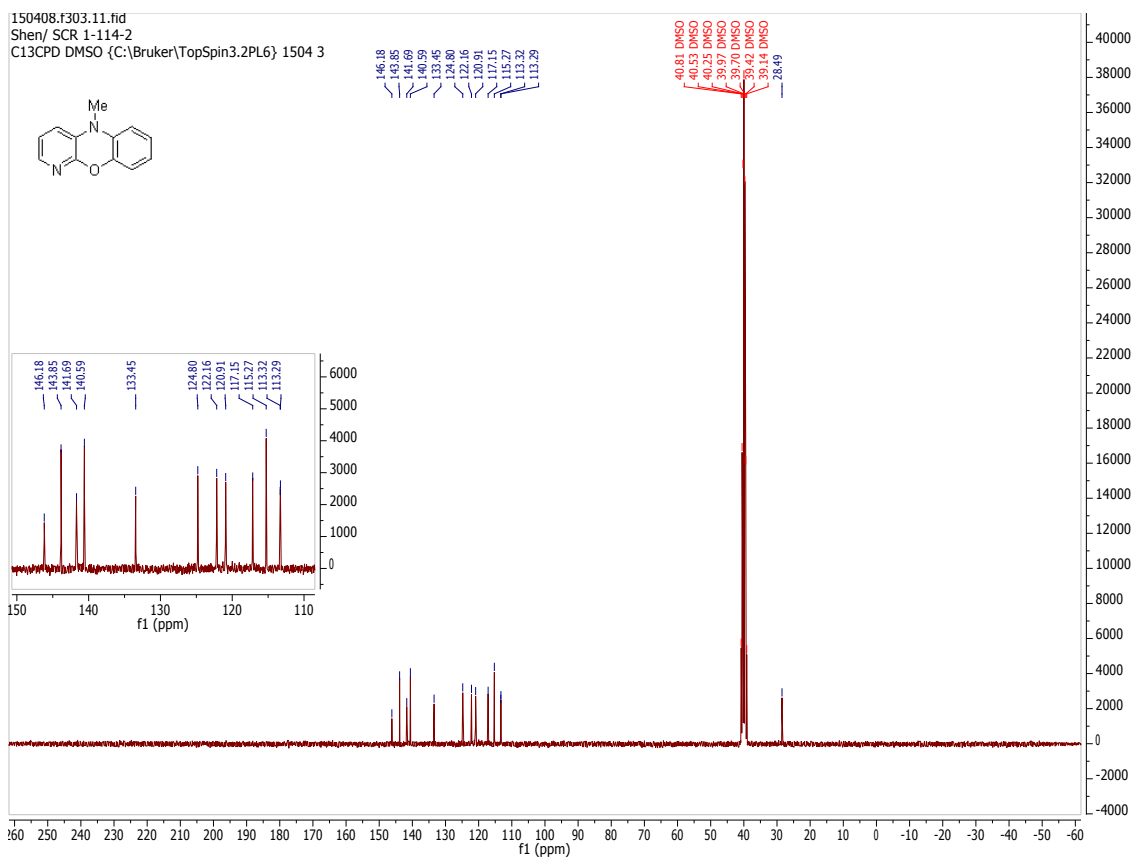


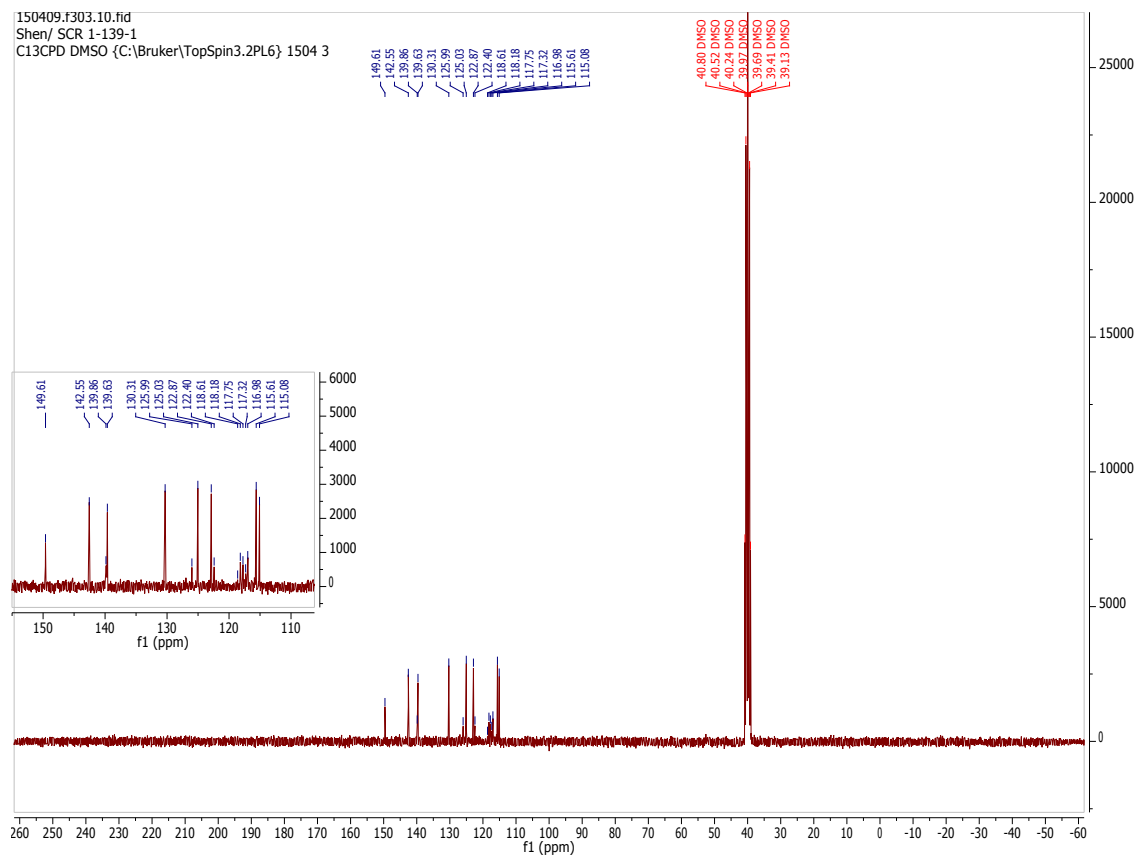
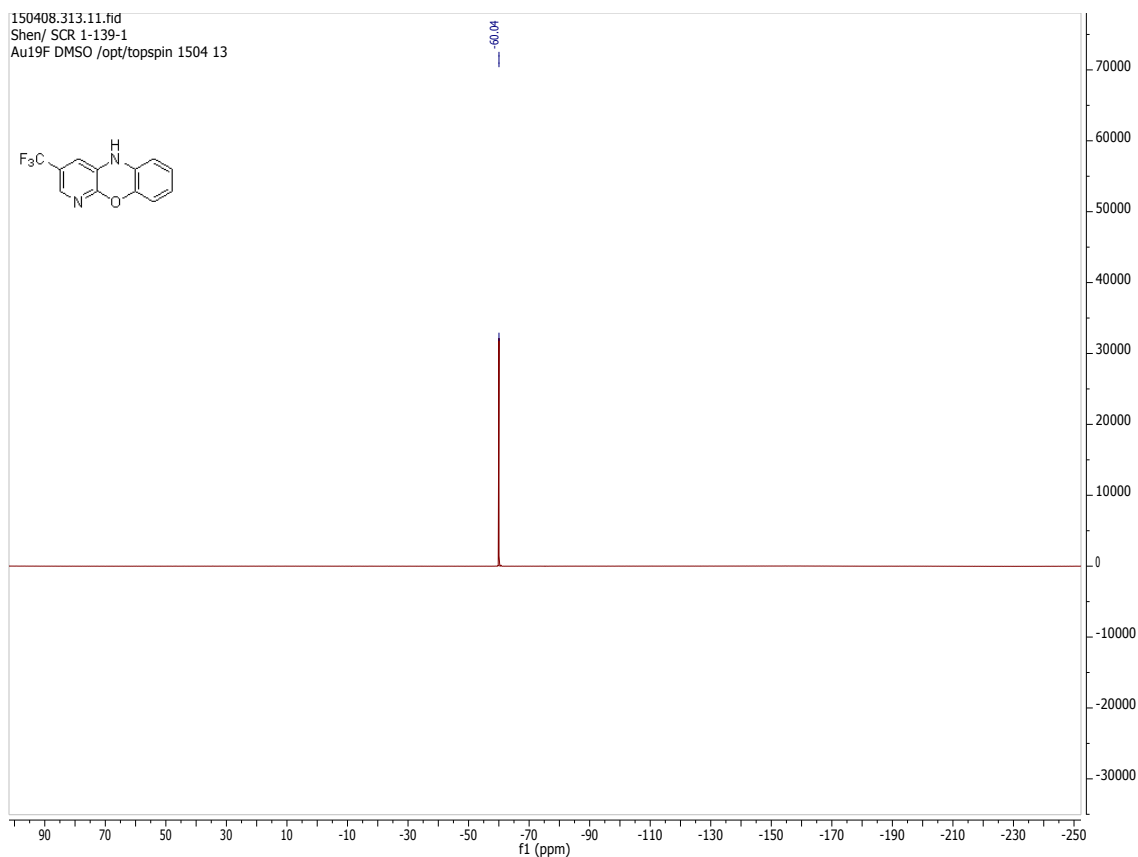


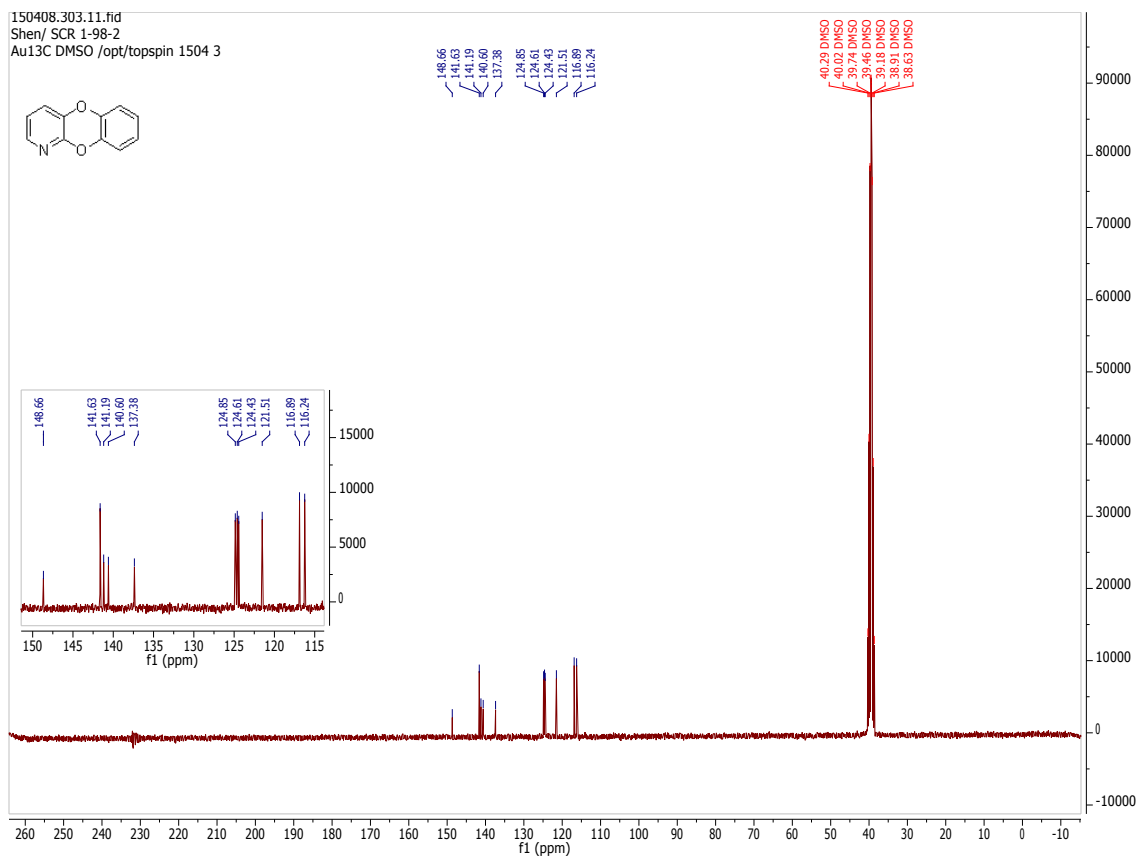
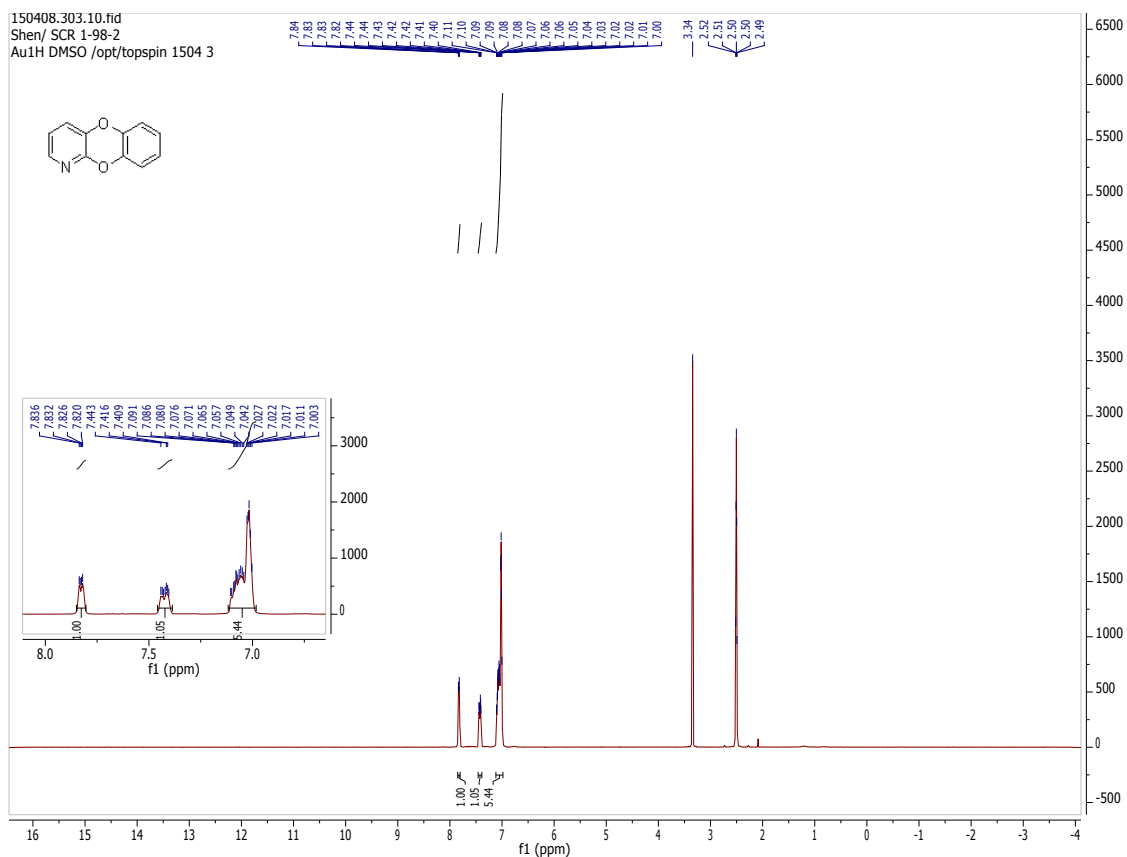


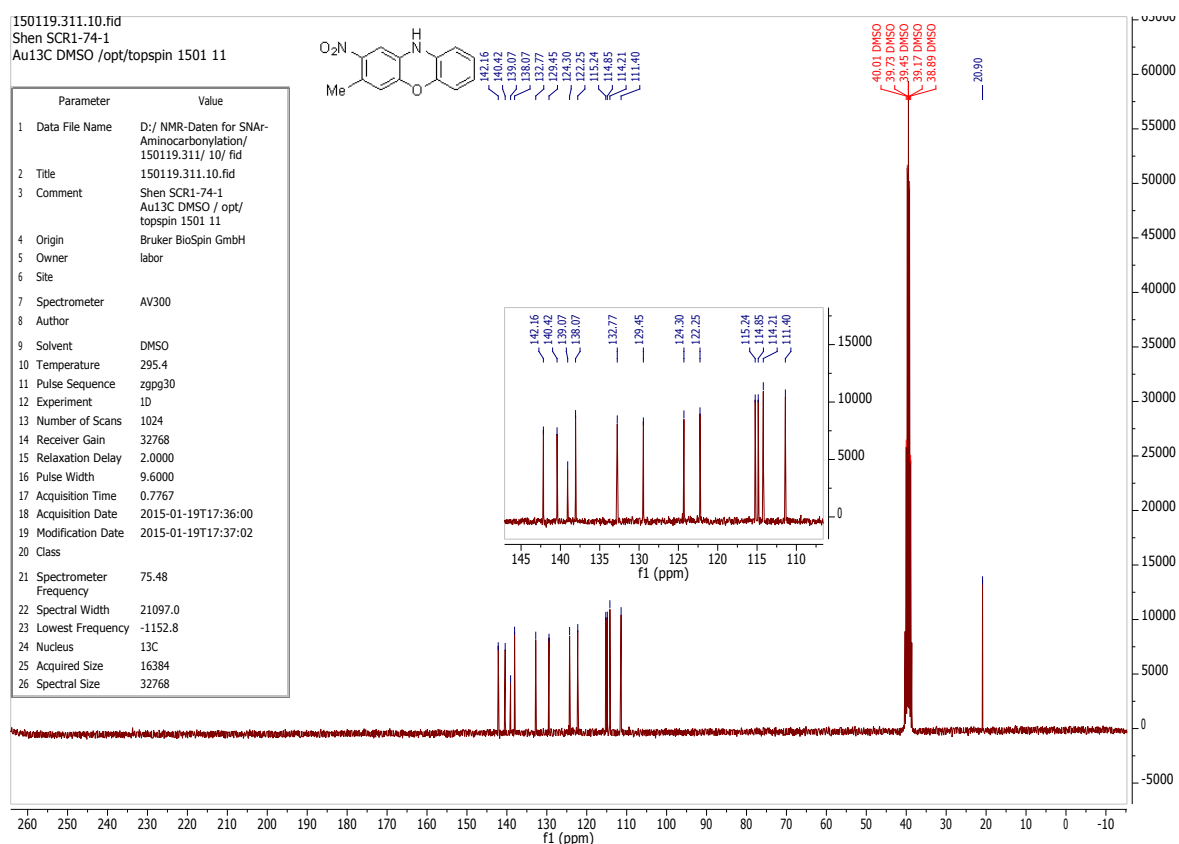
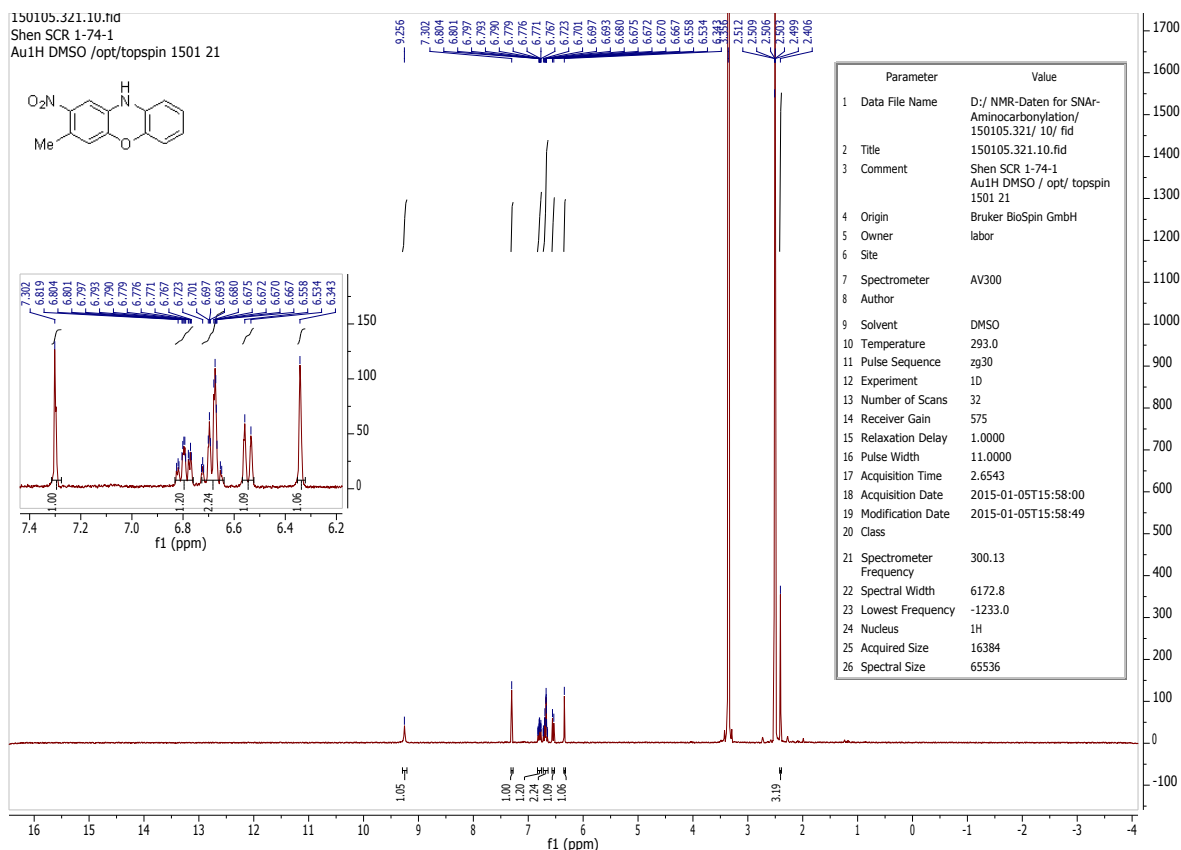


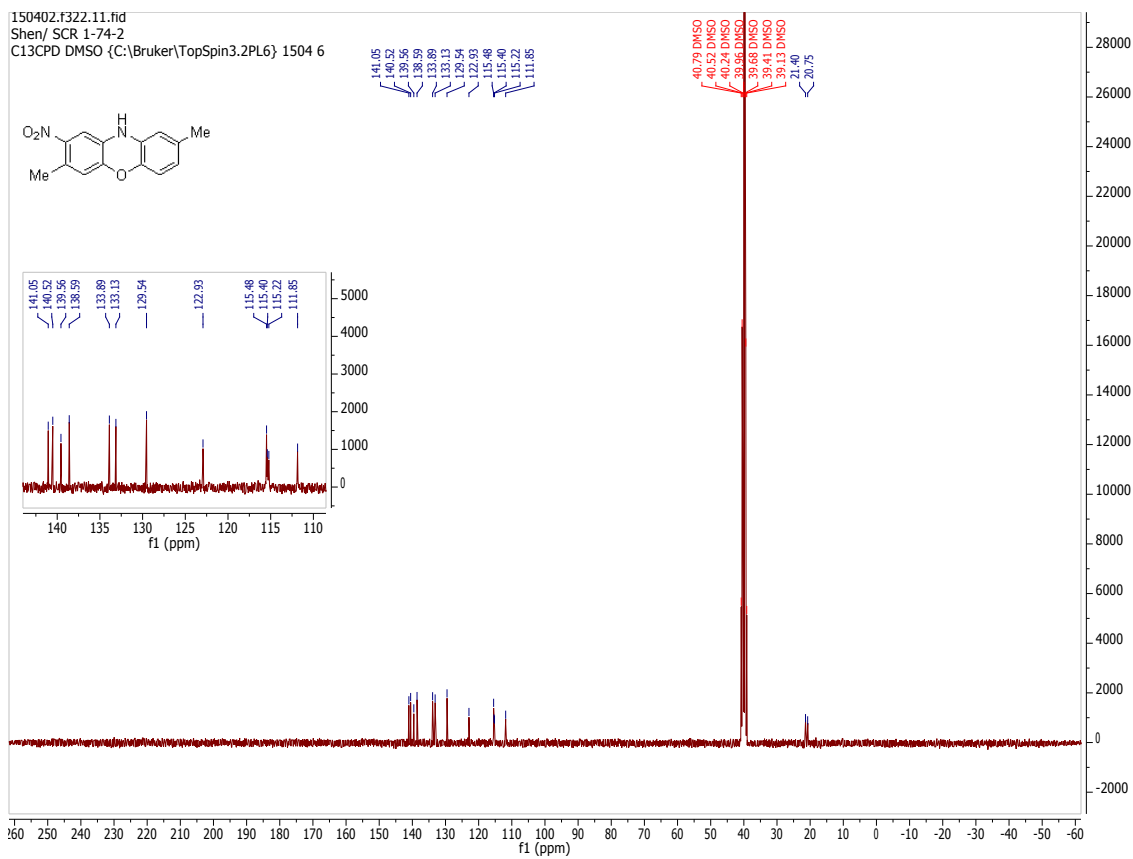
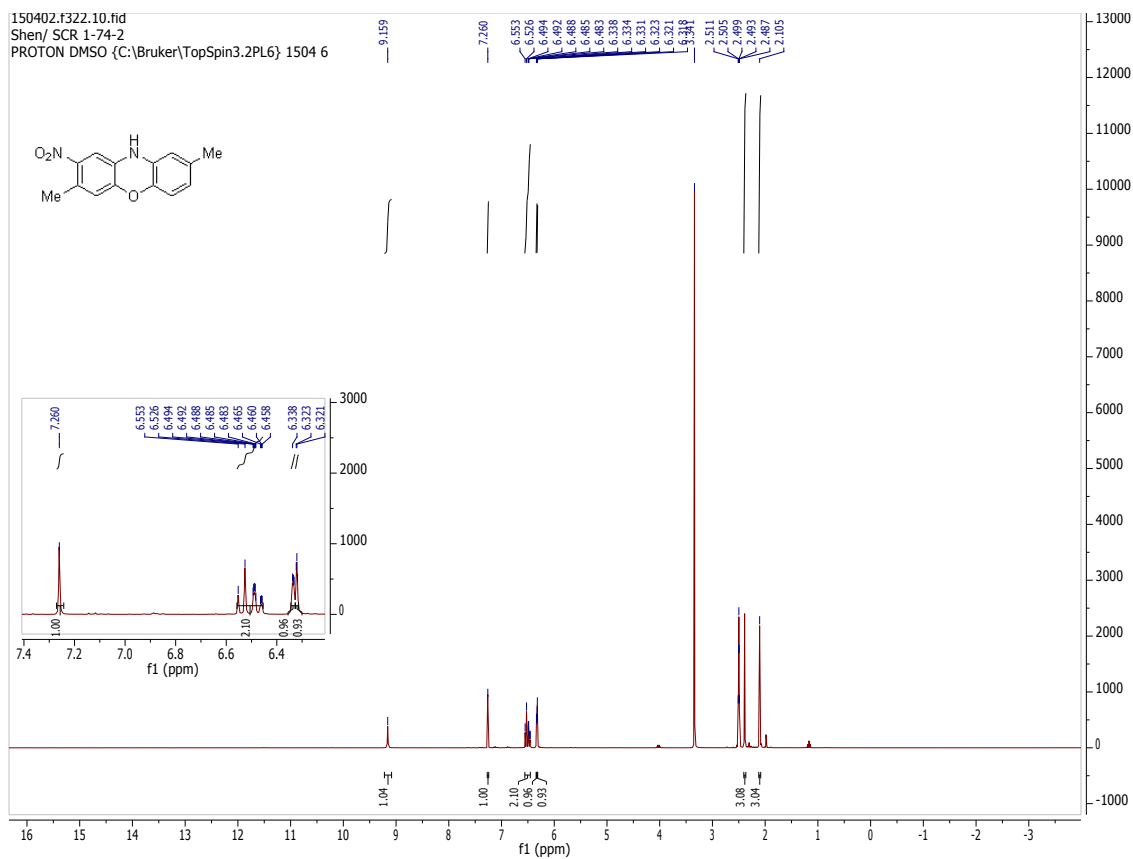


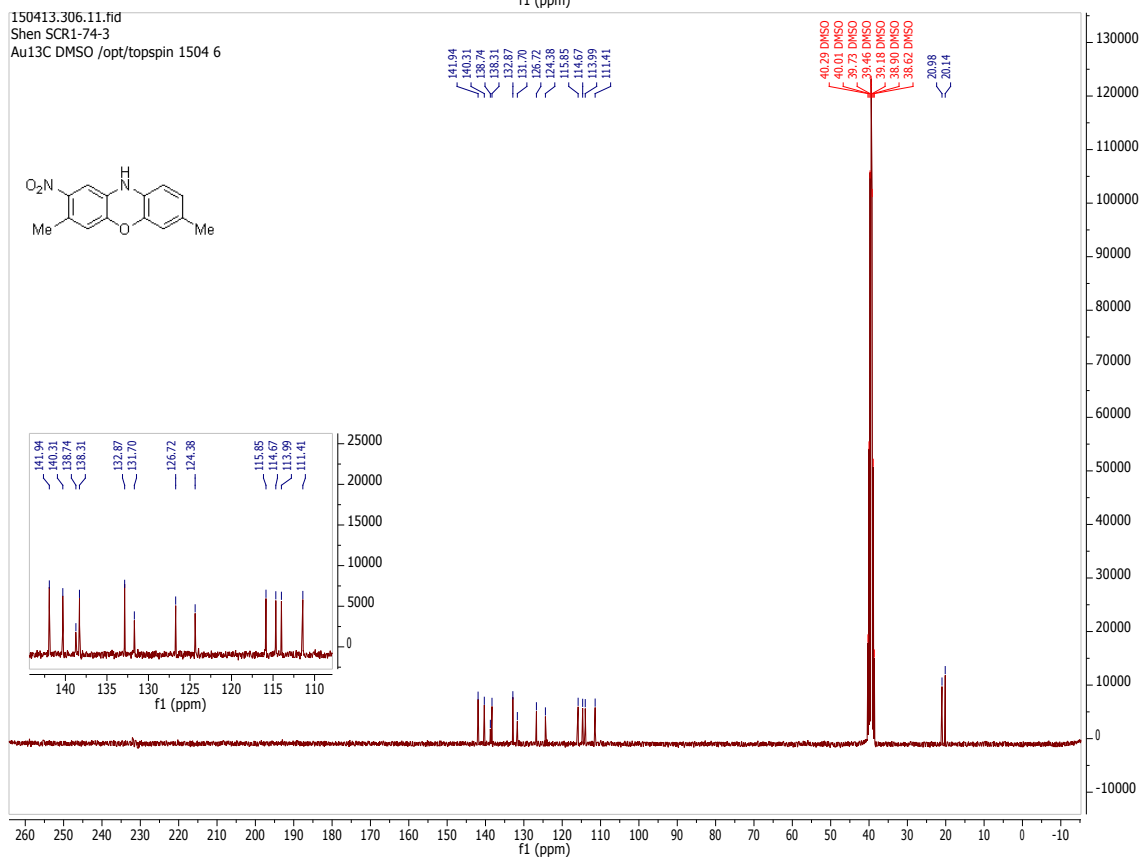
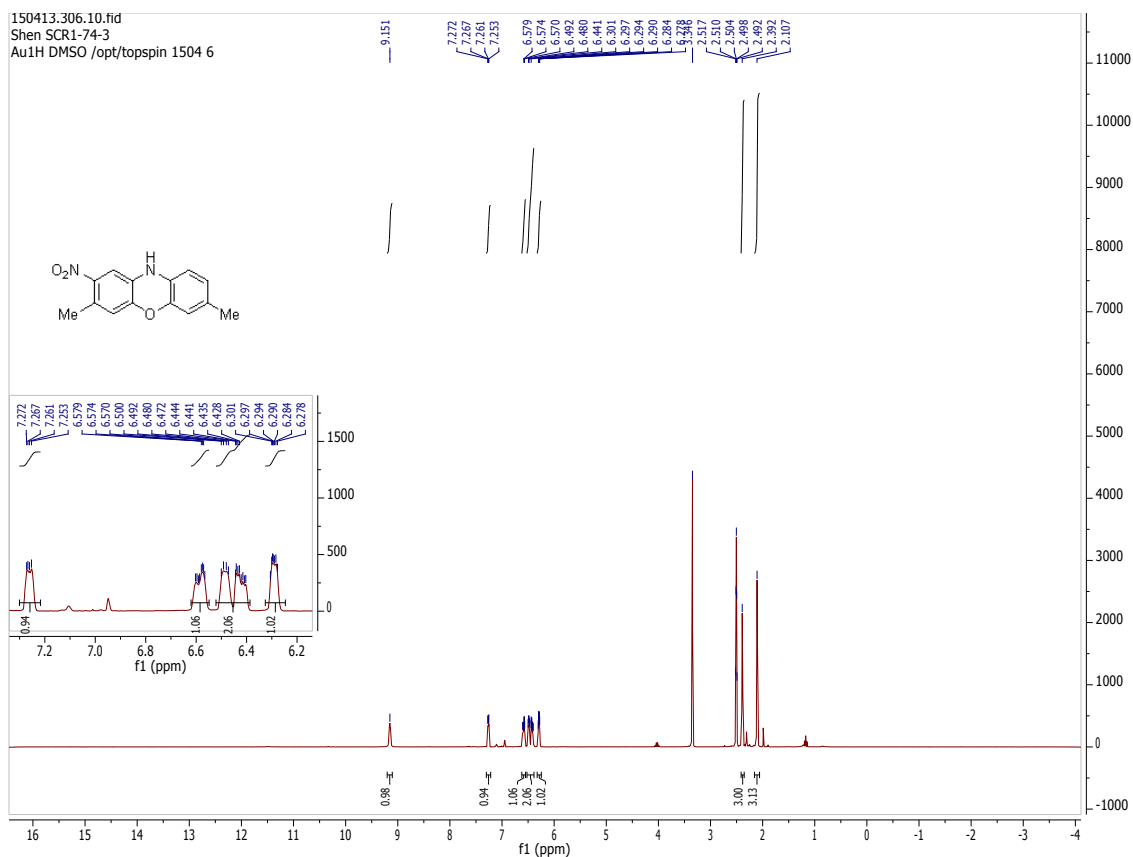


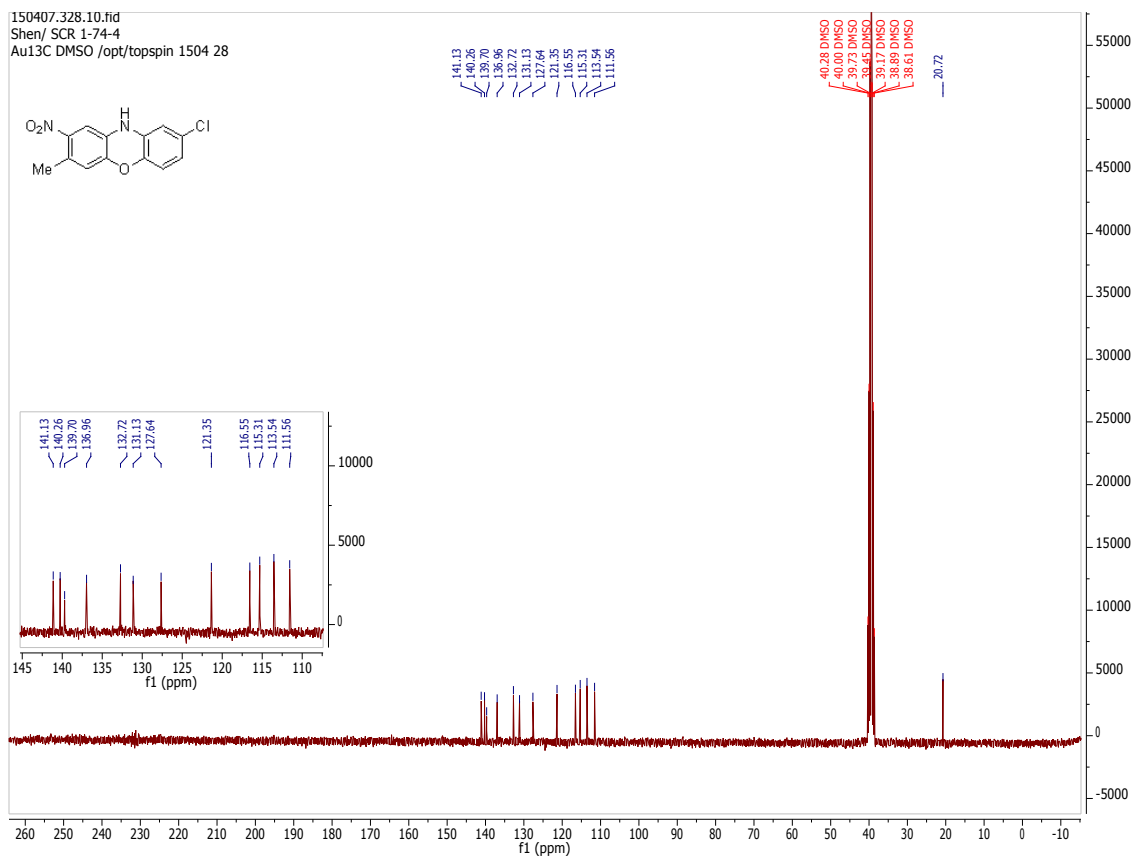
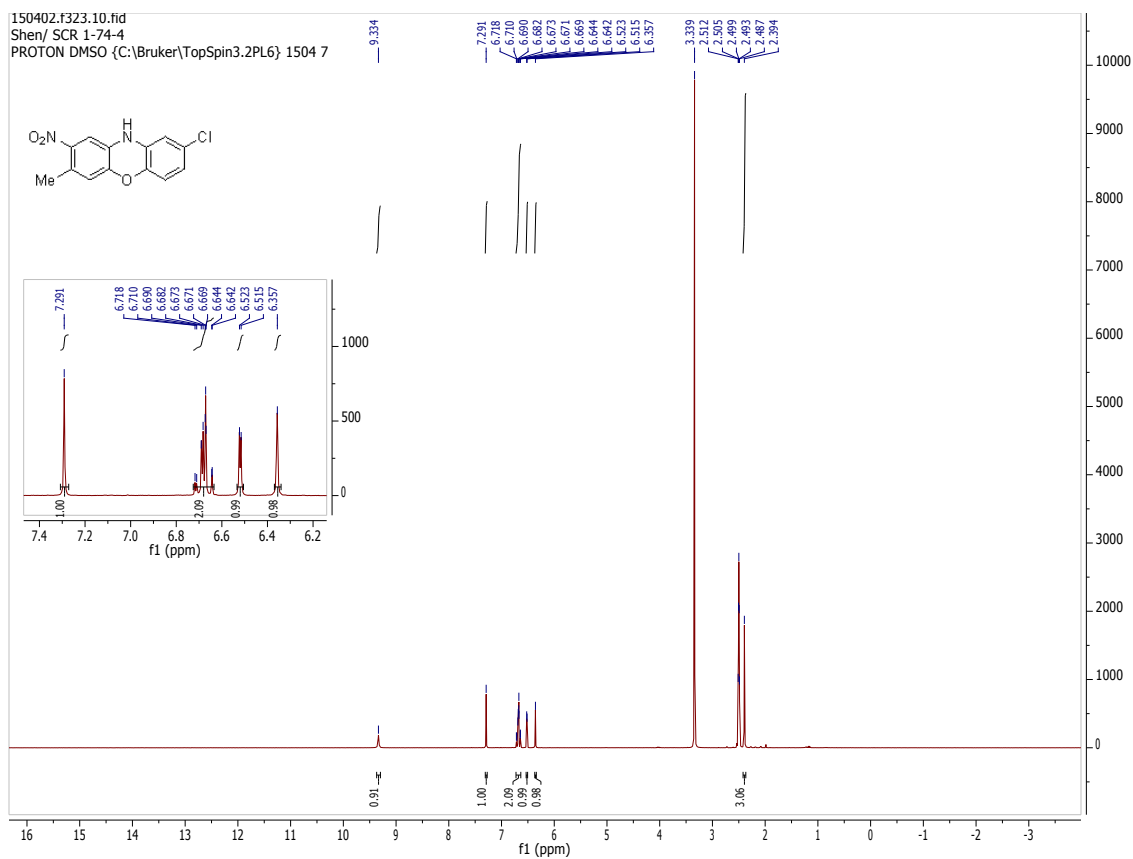


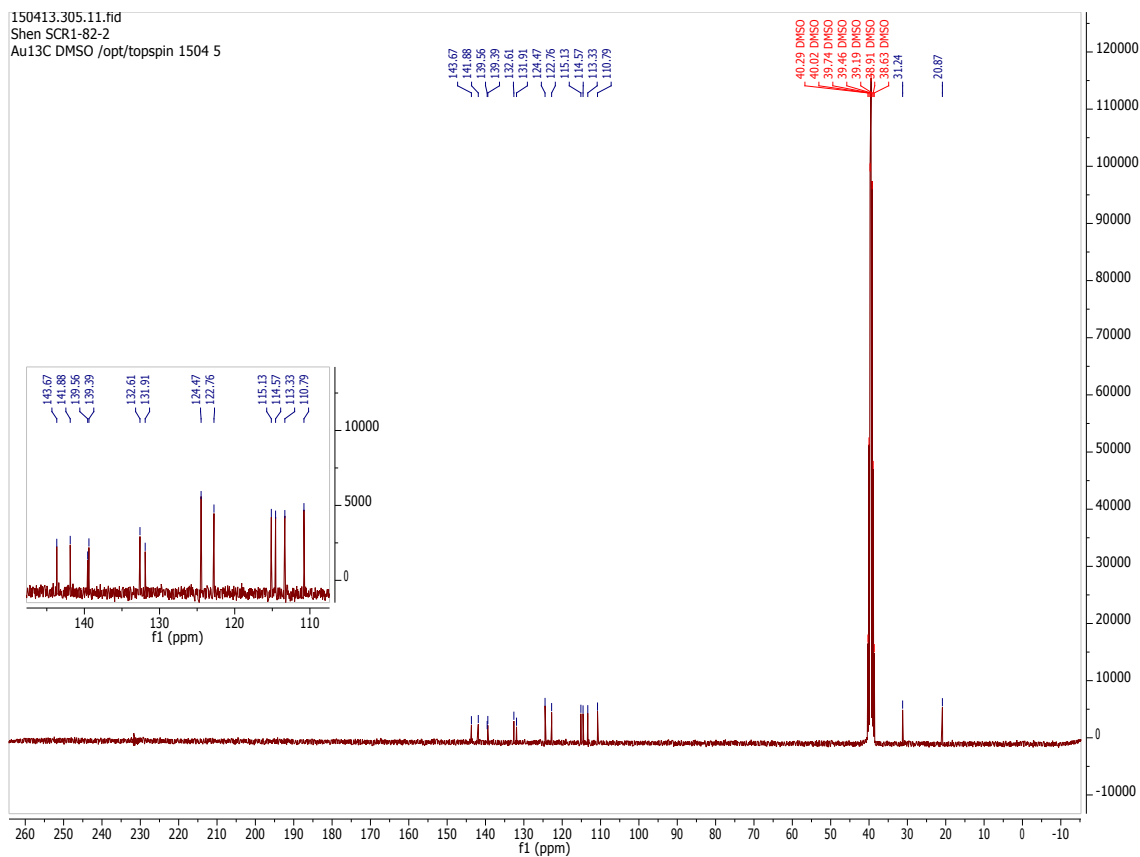
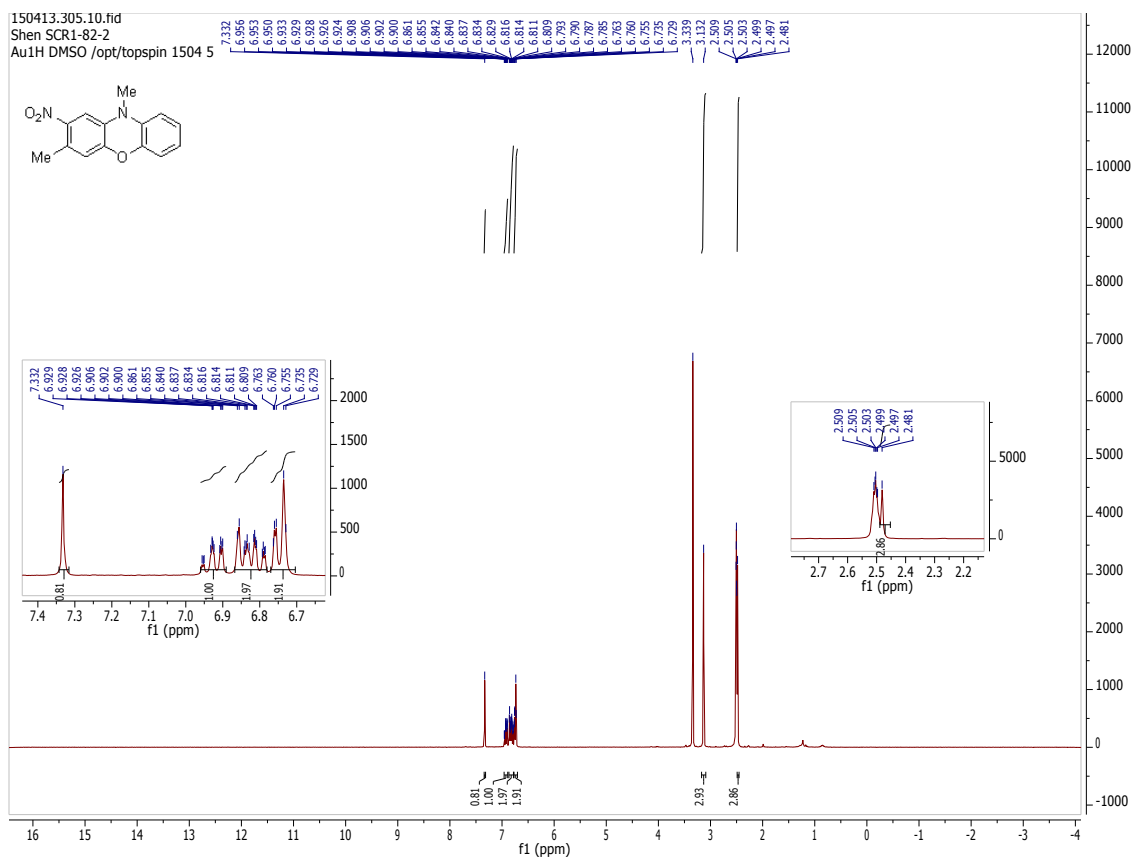












Reference

- [1] (a) J. M. Klunder, K. D. Hargrave, M. West, E. Cullen, K. Pal, M. L. Behnke, S. R. Kapadia, D. W. McNeil, J. C. Wu, G. C. Chow and J. Adams, *J. Med. Chem.*, 1992, **35**, 1887; (b) J.-F. F. Liébgeois, F. A. Rogistert, Jacques Bruhwyler, J. Damas, T. P. Nguyen, M.-O. Inarejos, E. M. G. Chleide, M. G. A. Mercier, and J. E. Delarge, *J. Med. Chem.*, 1994, **37**, 519-525
- [2] (a) H. H. Lee and W. A. Denny, *J. Chem. Soc., Perkin Trans. 1*, 1990, 1071-1074; (b) R. G. Sutherland, A. Piorko, C. C. Lee, S. H. Simonsen, V. M. Lynch, *J. Heterocyclic Chem.*, 1988, 25, 1911-1916.
- [3] Y. Ito,; Y. Hamada, *Chemical and Pharmaceutical Bulletin*, 1978, **26**, 1375-1383.