

Supporting Information for

**Diastereoselective Assembly of Dispiro-cyclopentene-linked Bisoxindoles *via*
Annulation Involving Isatylidene Malononitriles and Benzylamines**

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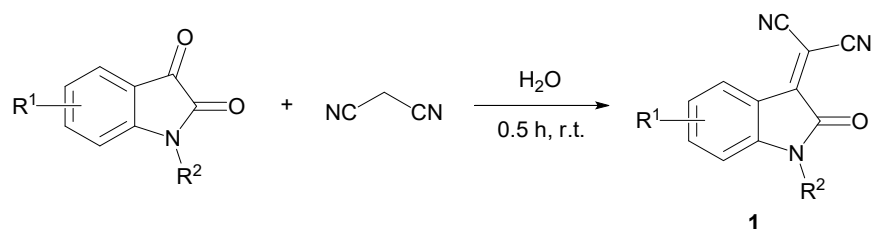
Table of Contents

1. General information	S2
2. General Procedure for Preparations of Isatylidene Malononitriles	S2
3. General Procedure for the Synthesis of Products	S3
4. Gram-Scale Reaction for the Synthesis of 3a	S3
5. X-Ray Crystal Structure and Data of Compound 3a	S4
6. Characterization of Products	S6
7. NMR Spectra	S15

1. General Information

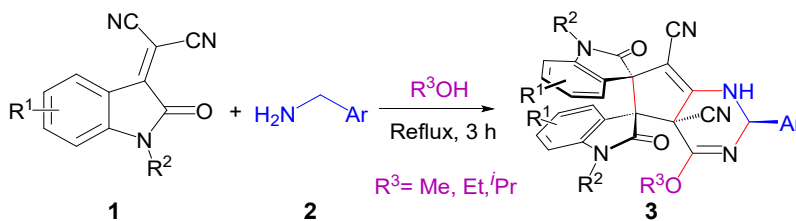
General Experimental Information. All solvents and chemicals used in this study were of analytical grade and employed without any additional purification steps. The melting points of substances were determined using an Electrothermal 9100 apparatus. Infrared (IR) spectra recorded using a Shimadzu-IR 460 spectrophotometer (ν in cm^{-1}). Proton (^1H), carbon-13 (^{13}C) and fluorine-19 (^{19}F) nuclear magnetic resonance (NMR) spectra were acquired using a Bruker DRX-500 Avance instrument. DMSO was utilized as the solvent, and tetramethylsilane (TMS) served as the internal standard. The spectrometer operated at frequencies of 500.1 MHz for ^1H NMR, 125.7 MHz for ^{13}C NMR and 471 MHz for ^{19}F NMR. Chemical shifts (δ) were expressed in parts per million (ppm), and coupling constants (J) were recorded in hertz (Hz). Mass spectrometry (MS) data were obtained using a Finnigan-MAT-8430 MS spectrometer, with an ionization potential of 70 electronvolts (eV). Elemental analyses for carbon (C), hydrogen (H), and nitrogen (N) content were carried out with a Heraeus CHN-O-Rapid analyzer. X-ray crystallography data were collected using a Marresearch 345 dtb diffractometer, which utilized Mo K α radiation (wavelength of 0.71073 Å). The measurements were performed at a temperature of 295 Kelvin (K). Analytical thin-layer chromatography (TLC) was conducted on silica gel plates with a UV-254 indicator, using appropriate eluents. Compounds were visualized under short-wavelength UV light.

2. General Procedure for Preparations of Isatylidene malononitriles



In a reaction vessel, 10.0 mmol of isatin (1.471 g) and 12.0 mmol of malononitrile (0.793 g, 1.2 equiv.) were added to 5 mL of water. After 0.5 hours at room temperature, the precipitated solid was collected by filtration and washed with water, affording the 2-(2-oxoindolin-3-ylidene)malononitrile as a red solid (up to a 97% yield). All the other isatylidene malononitrile derivatives were prepared according to the above-mentioned procedure, but with a reaction time of 1-2 hours.

3. General Procedure for the Synthesis of products 3 from 1 and 2



A mixture of **1** (2 mmol) and **2** (1 mmol) in R^3OH (3 mL) was refluxed in an oil bath for 3 hours. Centrifuging the precipitates, followed by recrystallization in a 50/50 solution of MeOH and DCM, and then washing the resulting crystals with cold ethanol yielded pure products.

4. Gram-Scale Reaction for the Synthesis of 3a

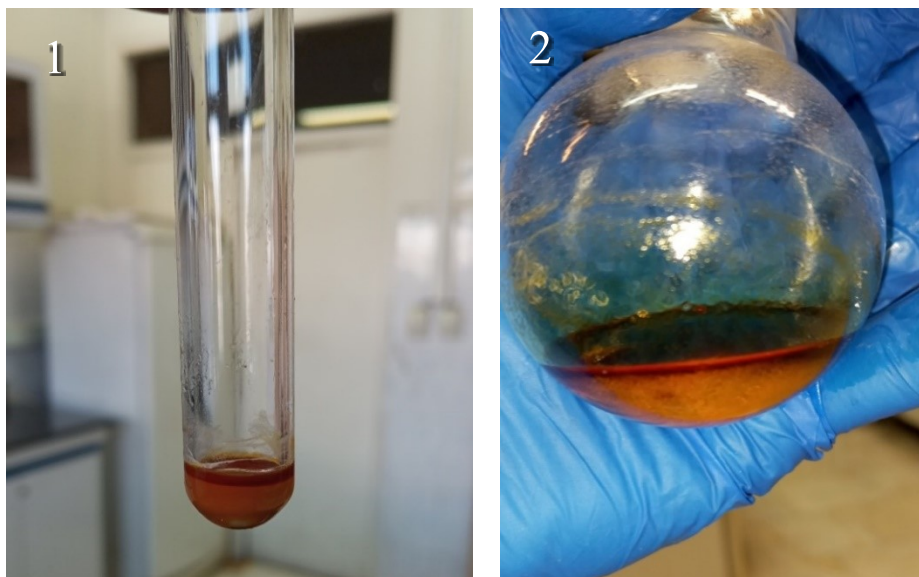


Figure S1. Reaction Setup.

Figure S2. Reaction Setup for Gram Scale.

To a dry round bottom flask equipped with a reflux condenser, was added isatylidene malononitrile **1a** (1.950 g, 10 mmol), benzylamine **2a** (0.536 g, 5 mmol) and 20 mL of methanol as solvent. The reaction mixture was then stirred at reflux temperature in an oil bath for 12 hours. Centrifuging the precipitates, followed by recrystallization in a 50/50 solution of MeOH and DCM, and then washing the resulting crystals with cold ethanol, will yield pure product **3a** (4.06 mmol, 2.08 g).

5. X-Ray Crystal Structure and Data of compound **3a**

Single crystals of compound **3a** (CCDC number 2358733) were grown by slow evaporation technique at ambient temperature. For preparation of a nearly saturated solution, 200 mg of **3a** was dissolved in 5 mL of MeCN:DCM (1:1) by warming to 60 °C. The solution was kept in a 25 mL beaker tightly covered with aluminum foil with few small holes for solvent evaporation. Colorless and transparent crystals obtained after 4 days. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/getstructures.

Table S1. Crystal data and structure refinement for **3a**. (CCDC: 2358733).

Identification code	3a
Empirical formula	C ₃₀ H ₂₀ N ₆ O ₃
Formula weight	512/16
Temperature/K	290
Crystal system	Monoclinic
Space group	P 21/c
a/Å	11.230(2)
b/Å	14.496(3)
c/Å	17.861(4)
α/°	90
β/°	101.09(3)
γ/°	90
Volume/Å ³	2853.3(10)
Z	4
ρ _{calc} /cm ³	1.289
μ/mm ⁻¹	0.09
F(000)	1152
Crystal size/mm ³	0.3 × 0.25 × 0.1
Radiation	Mo Kα (λ = 0.71073 Å)
Theta range for data collection/°	1.8 to 26.4
Index ranges	-14 ≤ h ≤ 14, -17 ≤ k ≤ 17, -22 ≤ l ≤ 22
Reflections collected	20409
Independent reflection	5642 [R _{int} = 0.065]
Data/restraints/parameters	5642 /0/381
Goodness-of-fit on F ²	1.144
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0810, wR ₂ = 0.2040
Final R indexes [all data]	R ₁ = 0.0807, wR ₂ = 0.2038
Largest diff. peak/hole / e Å ⁻³	0.43/-0.24

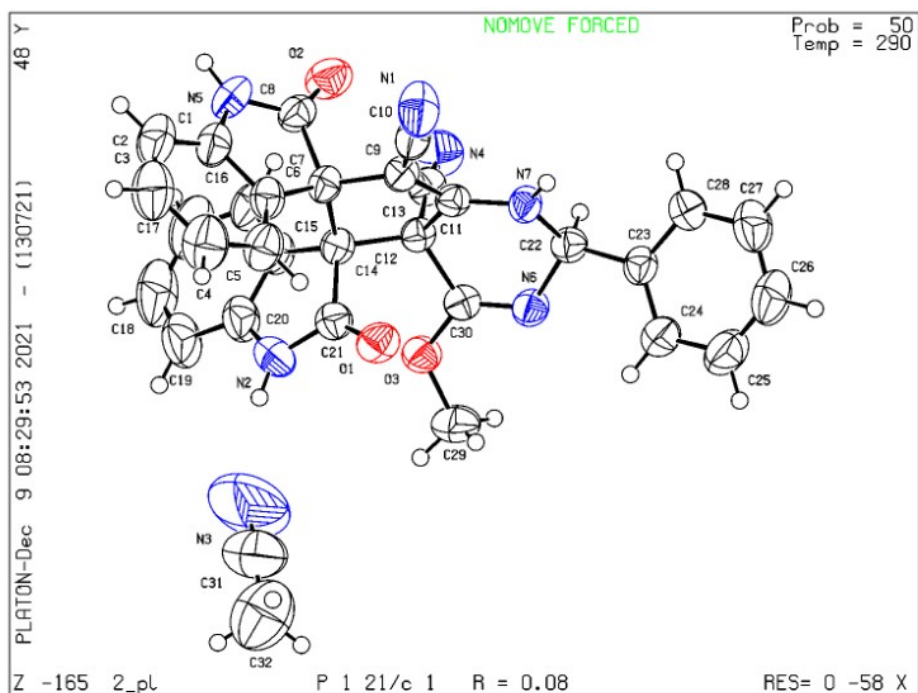


Figure S3. X-ray crystallographic analysis of **3a**.

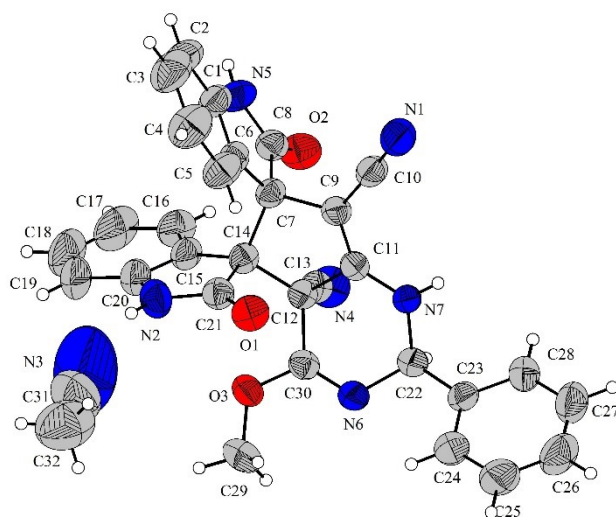
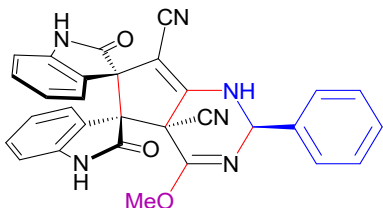


Figure S4. X-ray Crystal Structure (CCDC: 2358733) of Dispiro-bisoxindole Compound **3a** together with one Molecule of MeCN.

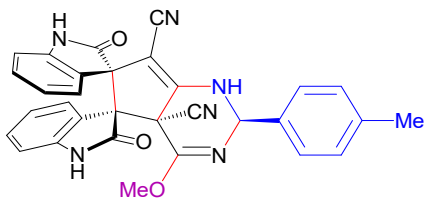
6. Characterization of Products

4'-Methoxy-2,2''-dioxo-2'-phenyl-1',2'-dihydro-4a'*H*-dispiro[indoline-3,5'-cyclopenta[*d*]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3a)



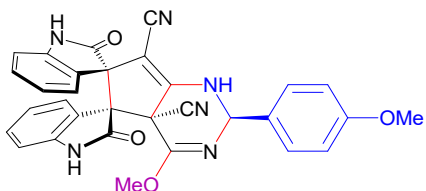
Colorless powder (235 mg from 1 mmol scale, 92% yield); mp: 270-275 °C (dec); IR (KBr, cm^{-1}) 3428, 3244, 2194, 1708, 1670, 1642, 1619; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.77 (s, 1H), 10.56 (s, 1H), 9.62 (s, 1H), 7.75 (d, $J = 6.4$ Hz, 1H), 7.55 (td, $J = 5.0, 2.3$ Hz, 2H), 7.50 (dd, $J = 7.5, 2.3$ Hz, 2H), 7.46 – 7.43 (m, 1H), 7.29 (d, $J = 7.2$ Hz, 1H), 7.24 (t, $J = 7.5$ Hz, 1H), 6.95 (t, $J = 6.5$ Hz, 1H), 7.02 (t, $J = 6.9$ Hz, 1H), 6.95 (t, $J = 6.5$ Hz, 1H), 6.70 (d, $J = 8.0$ Hz, 1H), 6.62 (d, $J = 7.1$ Hz, 1H), 6.18 (s, 1H), 3.50 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.8, 174.3, 155.3, 153.2, 143.1, 142.9, 140.5, 130.3, 130.2, 128.7, 128.6, 127.6, 126.2, 125.7, 123.9, 122.6, 122.1, 121.9, 115.9, 113.7, 109.9, 109.8, 71.9, 71.6, 62.8, 58.6, 54.5, 46.7. MS: m/z (%): 512 (23), 485 (10), 428 (24), 380 (17), 351 (15), 262 (88), 234 (89), 205 (38), 132 (70), 104 (100), 77 (55). Anal. Calcd for $\text{C}_{30}\text{H}_{20}\text{N}_6\text{O}_3$ (512.16) C, 70.30; H, 3.93; N, 16.40. Found: C, 70.67; H, 3.95; N, 16.48.

4'-Methoxy-2,2''-dioxo-2'-(*p*-tolyl)-1',2'-dihydro-4a'*H*-dispiro[indoline-3,5'-cyclopenta[*d*]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3b)



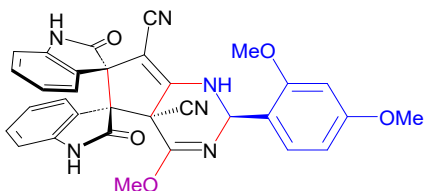
Colorless powder (244 mg from 1 mmol scale, 93% yield); mp: 277-278 °C (dec); IR (KBr, cm^{-1}) 3449, 3289, 2211, 1729, 1703, 1686, 1648, 1617; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.80 (s, 1H), 10.59 (s, 1H), 9.61 (s, 1H), 7.78 (d, $J = 7.7$ Hz, 1H), 7.45 (d, $J = 7.7$ Hz, 2H), 7.30 – 7.33 (m, 3H), 7.24 (t, $J = 7.4$ Hz, 1H), 7.17 (t, $J = 7.5$ Hz, 1H), 7.03 (t, $J = 8.0$ Hz, 1H), 6.96 (t, $J = 7.8$ Hz, 1H), 6.72 (d, $J = 7.9$ Hz, 1H), 6.64 (d, $J = 7.7$ Hz, 1H), 6.16 (s, 1H), 3.50 (s, 3H), 2.36 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.9, 174.3, 155.3, 153.3, 143.1, 143.0, 138.1, 137.6, 130.3, 130.3, 129.1, 127.6, 126.3, 125.8, 124.0, 122.7, 122.1, 121.9, 116.0, 113.8, 109.9, 109.9, 71.8, 71.5, 62.9, 58.7, 54.5, 46.8, 20.9. MS: m/z (%): 525 (12), 499 (55), 442 (100), 394 (11), 365 (12), 262 (73), 234 (76), 205 (30), 132 (95), 118 (67), 89 (53). Anal. Calcd for $\text{C}_{31}\text{H}_{22}\text{N}_6\text{O}_3$ (526.18) C, 70.71; H, 4.21; N, 15.96. Found: C, 71.16; H, 4.20; N, 16.07.

4'-Methoxy-2'-(4-methoxyphenyl)-2,2''-dioxo-1',2'-dihydro-4a'*H*-dispiro[indoline-3,5'-cyclopenta[*d*]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3c)



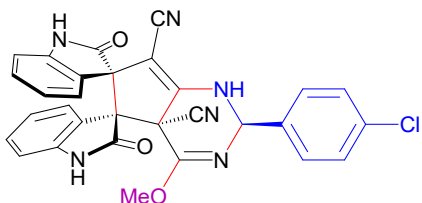
Yellow powder (209 mg from 1 mmol scale, 77% yield); mp: 266-267 °C (dec); IR (KBr, cm^{-1}) 3425, 3355, 2210, 1727, 1688, 1643, 1618; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.78 (s, 1H), 10.57 (s, 1H), 9.56 (s, 1H), 7.73 (d, $J = 7.8$ Hz, 1H), 7.46 (d, $J = 8.8$ Hz, 2H), 7.28 (d, $J = 7.5$ Hz, 1H), 7.24 (t, $J = 7.5$ Hz, 1H), 7.17 (t, $J = 7.8$ Hz, 1H), 7.05 (d, $J = 8.8$ Hz, 2H), 7.03 (t, $J = 7.6$ Hz, 1H), 6.96 (t, $J = 7.7$ Hz, 1H), 6.70 (d, $J = 7.8$ Hz, 1H), 6.61 (d, $J = 7.8$ Hz, 1H), 6.13 (s, 1H), 3.81 (s, 3H), 3.49 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.7, 174.2, 159.5, 155.1, 153.2, 143.0, 142.9, 132.6, 132.6, 130.2, 130.1, 128.8, 126.2, 125.7, 123.9, 122.6, 122.0, 121.8, 115.9, 113.8, 109.8, 109.7, 71.5, 71.1, 62.7, 58.5, 55.3, 54.4, 46.6. MS: m/z (%): 542 (50), 515 (20), 458 (14), 410 (40), 393 (22), 346 (21), 288 (100), 273 (30), 196 (20), 149 (75), 134 (23), 119 (24), 92 (36), 77 (75). Anal. Calcd for $\text{C}_{31}\text{H}_{22}\text{N}_6\text{O}_4$ (542.17) C, 68.63; H, 4.09; N, 15.49. Found: C, 69.07; H, 4.14; N, 15.54.

(2,4-Dimethoxyphenyl)-4'-methoxy-2,2''-dioxo-1',2'-dihydro-4a'*H*-dispiro[indoline-3,5'-cyclopenta[*d*]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3d)



Yellow powder (261 mg from 1 mmol scale, 91% yield); mp: 234-236 °C (dec); IR (KBr, cm^{-1}) 3369, 3228, 2198, 1713, 1681, 1641, 1617; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.73 (s, 1H), 10.52 (s, 1H), 9.44 (s, 1H), 7.72 (dd, $J = 7.8, 1.2$ Hz, 1H), 7.32 – 7.30 (m, 1H), 7.26 (dd, $J = 7.8, 1.2$ Hz, 1H), 7.22 (td, $J = 7.8, 1.4$ Hz, 1H), 7.15 (td, $J = 7.6, 1.3$ Hz, 1H), 7.01 (td, $J = 7.8, 1.2$ Hz, 1H), 6.93 (td, $J = 7.6, 1.1$ Hz, 1H), 6.70 – 6.64 (m, 3H), 6.60 (d, $J = 7.8$ Hz, 1H), 6.31 (s, 1H), 3.88 (s, 3H), 3.81 (s, 3H), 3.52 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.8, 174.1, 160.9, 157.6, 156.2, 153.4, 142.9, 142.8, 130.1, 130.0, 129.0, 126.1, 125.6, 123.9, 122.5, 121.9, 121.7, 119.8, 115.9, 113.3, 109.7, 109.6, 105.4, 98.0, 70.6, 64.9, 62.9, 58.6, 55.7, 55.4, 54.4, 47.0. MS: m/z (%): 571 (22), 545 (49), 488 (10), 262 (65), 234 (71), 205 (36), 164 (100), 134 (30), 92 (33), 77 (55). Anal. Calcd for $\text{C}_{32}\text{H}_{24}\text{N}_6\text{O}_5$ (572.18) C, 67.13; H, 4.23; N, 14.68. Found: C, 67.48; H, 4.27; N, 14.74.

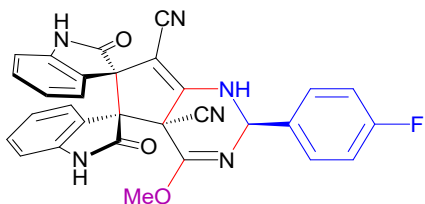
2'-(4-Chlorophenyl)-4'-methoxy-2,2''-dioxo-1',2'-dihydro-4a'H-dispiro[indoline-3,5'-cyclopenta[d]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3e)



Colorless powder (239 mg from 1 mmol scale, 88% yield); mp: 265-267 °C (dec); IR (KBr, cm^{-1}) 3287, 3203, 2210, 1730, 1704, 1687, 1648, 1618; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.78 (s, 1H), 10.57 (s, 1H), 9.61 (s, 1H), 7.76 (d, $J = 7.7$ Hz, 1H), 7.58 (s, 4H), 7.30 (d, $J = 7.7$ Hz,

1H), 7.24 (t, $J = 7.8$ Hz, 1H), 7.17 (t, $J = 7.7$ Hz, 1H), 7.03 (t, $J = 7.7$ Hz, 1H), 6.96 (t, $J = 7.6$ Hz, 1H), 6.71 (d, $J = 7.8$ Hz, 1H), 6.63 (d, $J = 7.8$ Hz, 1H), 6.24 (s, 1H), 3.51 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.7, 174.2, 155.6, 153.2, 143.0, 142.9, 139.3, 133.3, 130.3, 130.2, 129.5, 128.6, 126.2, 125.7, 123.9, 122.5, 122.1, 121.8, 115.8, 113.6, 109.9, 109.8, 72.1, 70.8, 62.8, 58.7, 54.6, 46.7. MS: m/z (%): 549 (2), 534 (12), 519 (24), 462 (24), 262 (98), 234 (100), 205 (41), 154 (92), 137 (98). Anal. Calcd for $\text{C}_{30}\text{H}_{19}\text{ClN}_6\text{O}_3$ (546.12) C, 65.88; H, 3.50; N, 15.36. Found: C, 66.27; H, 3.54; N, 15.42.

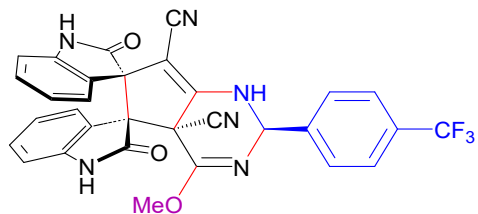
2'-(4-Fluorophenyl)-4'-methoxy-2,2''-dioxo-1',2'-dihydro-4a'H-dispiro[indoline-3,5'-cyclopenta[d]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3f)



Colorless powder (238 mg from 1 mmol scale, 90% yield); mp: 267-270 °C (dec); IR (KBr, cm^{-1}) 3291, 3199, 2213, 1730, 1706, 1686, 1649, 1618; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.79 (s, 1H), 10.57 (s, 1H), 9.60 (s, 1H), 7.75 (d, $J = 6.9$ Hz, 1H), 7.59 (dd, $J = 8.7, 5.7$, 2H), 7.33

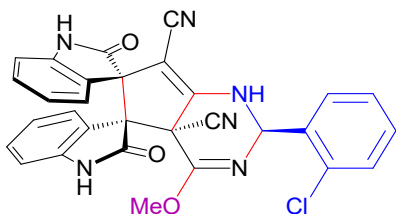
(t, $J = 8.8$, 2H), 7.29 (d, $J = 6.8$ Hz, 1H), 7.24 (t, $J = 7.7$ Hz, 1H), 7.16 (t, $J = 7.6$ Hz, 1H), 7.03 (t, $J = 7.5$ Hz, 1H), 6.95 (t, $J = 7.7$ Hz, 1H), 6.70 (d, $J = 7.9$ Hz, 1H), 6.62 (d, $J = 7.7$ Hz, 1H), 6.23 (s, 1H), 3.50 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.7, 174.3, 162.3 (d, $J = 243.2$ Hz), 155.5, 153.2, 143.1, 142.9, 136.8 (d, $J = 3.0$ Hz), 130.3, 130.2, 129.8 (d, $J = 8.6$ Hz), 126.2, 125.7, 123.9, 122.6, 122.1, 121.9, 115.9, 115.4 (d, $J = 21.4$ Hz), 113.6, 109.9, 109.8, 72.0, 70.8, 62.8, 58.7, 54.6, 46.7; ^{19}F NMR (471 MHz, $\text{DMSO-}d_6$) δ -113.5. MS: m/z (%): 530 (25), 503 (50), 446 (83), 407 (23), 262 (100), 234 (78), 141 (51), 122 (41). Anal. Calcd for $\text{C}_{30}\text{H}_{19}\text{FN}_6\text{O}_3$ (530.15) C, 67.92; H, 3.61; N, 15.84. Found: C, 68.41; H, 3.66; N, 15.91.

4'-Methoxy-2,2''-dioxo-2'-(4-(trifluoromethyl)phenyl)-1',2'-dihydro-4a'H-dispiro[indoline-3,5'-cyclopenta[d]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3g)



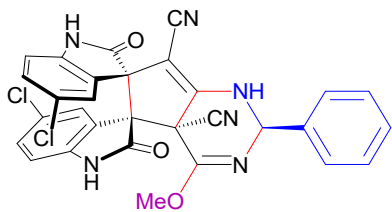
Colorless powder (217 mg from 1 mmol scale, 75% yield); mp: 242-244 °C (dec); IR (KBr, cm^{-1}) 3408, 3198, 2215, 1717, 1685, 1649, 1620; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.79 (s, 1H), 10.58 (s, 1H), 9.61 (s, 1H), 7.80 – 7.77 (m, 2H), 7.75 (d, $J = 7.6$ Hz, 1H), 7.53 – 7.51 (m, 2H), 7.29 (d, $J = 7.6$ Hz, 1H), 7.24 (t, $J = 7.6$ Hz, 1H), 7.17 (t, $J = 7.7$ Hz, 1H), 7.03 (t, $J = 7.7$ Hz, 1H), 6.96 (t, $J = 7.6$ Hz, 1H), 6.70 (d, $J = 7.9$ Hz, 1H), 6.62 (d, $J = 7.7$ Hz, 1H), 6.23 (s, 1H), 3.50 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.8, 174.5, 156.6, 153.4, 143.0, 142.9, 142.8, 130.0, 129.9, 129.6, 126.2, 125.7, 124.2, 123.9, 123.9, 123.0, 122.0, 121.9, 121.7, 115.7, 113.8, 109.7, 109.6, 72.3, 71.9, 62.5, 57.4, 54.1, 46.8; ^{19}F NMR (471 MHz, $\text{DMSO-}d_6$) δ -60.9. MS: m/z (%): 579 (9), 552 (13), 495 (20), 262 (100), 234 (65), 205 (28), 172 (81), 156 (12), 149 (21). Anal. Calcd for $\text{C}_{31}\text{H}_{19}\text{F}_3\text{N}_6\text{O}_3$ (580.15) C, 64.14; H, 9.82; N, 14.48. Found: C, 64.59; H, 3.32; N, 14.52.

2'-(2-Chlorophenyl)-4'-methoxy-2,2''-dioxo-1',2'-dihydro-4a'H-dispiro[indoline-3,5'-cyclopenta[d]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3h)



Yellow powder (193 mg from 1 mmol scale, 71% yield); mp: 250-252 °C (dec); IR (KBr, cm^{-1}) 3332, 3205, 3065, 2212, 1721, 1685, 1652, 1620; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.80 (s, 1H), 10.60 (s, 1H), 9.80 (s, 1H), 7.75 (d, $J = 7.8$ Hz, 1H), 7.59 (dd, $J = 7.5, 1.9$ Hz, 1H), 7.56 (d, $J = 6.7$ Hz, 1H), 7.52 (t, $J = 7.3$ Hz, 1H), 7.48 (td, $J = 7.5, 1.9$ Hz, 1H), 7.29 (d, $J = 7.8$ Hz, 1H), 7.25 (t, $J = 7.9$ Hz, 1H), 7.17 (t, $J = 7.7$ Hz, 1H), 7.03 (t, $J = 7.4$ Hz, 1H), 6.96 (t, $J = 7.7$ Hz, 1H), 6.71 (d, $J = 7.7$ Hz, 1H), 6.63 (d, $J = 8.1$ Hz, 1H), 6.44 (s, 1H), 3.55 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.7, 174.2, 156.7, 153.3, 143.0, 142.9, 141.9, 134.2, 130.6, 130.5, 130.3, 129.8, 129.4, 127.8, 126.2, 125.7, 123.8, 122.4, 122.1, 122.0, 116.3, 113.3, 109.9, 109.8, 73.7, 71.9, 62.9, 58.7, 54.7, 47.1. MS: m/z (%): 546 (3), 519 (16), 425 (12), 360 (41), 297 (20), 235 (25), 170 (43), 133 (42), 89 (76), 66 (100). Anal. Calcd for $\text{C}_{30}\text{H}_{19}\text{ClN}_6\text{O}_3$ (546.12) C, 65.88; H, 3.50; N, 15.36. Found: C, 65.57; H, 3.56; N, 15.44.

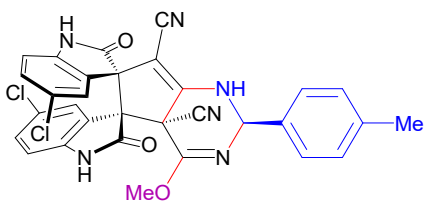
5,5''-Dichloro-4'-methoxy-2,2''-dioxo-2'-phenyl-1',2'-dihydro-4a'H-dispiro[indoline-3,5'-cyclopenta[d]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3i)



Colorless powder (262 mg from 1 mmol scale, 90% yield); mp: 275-277 °C (dec); IR (KBr, cm^{-1}) 3427, 3244, 2209, 1719, 1674, 1643, 1620; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 11.20 (s, 1H), 10.96 (s, 1H), 9.85 (s, 1H), 7.72 (d, $J = 2.4$ Hz, 1H), 7.52 (t, $J = 6.5$ Hz, 2H), 7.51 – 7.47 (m, 2H),

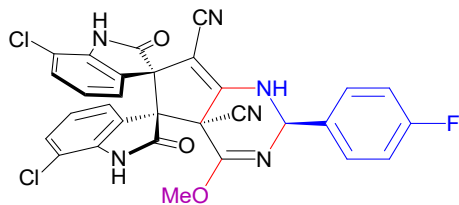
7.45 (td, $J = 6.4, 1.7$ Hz, 1H), 7.36 (dd, $J = 8.4, 2.2$ Hz, 1H), 7.28 (dd, $J = 8.4, 2.2$ Hz, 1H), 7.24 (d, $J = 2.2$ Hz, 1H), 6.80 (dd, $J = 8.4, 4.3$ Hz, 1H), 6.72 (dd, $J = 8.3, 4.2$ Hz, 1H), 6.22 (s, 1H), 3.52 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.3, 173.8, 154.6, 153.1, 141.9, 141.8, 140.1, 130.5, 130.5, 128.8, 128.6, 127.6, 126.2, 126.0, 126.0, 125.7, 125.6, 124.2, 115.5, 113.3, 111.7, 111.6, 71.7, 70.6, 62.8, 58.2, 54.7, 46.5. MS: m/z (%): 542 (100), 518 (12), 496 (51), 410 (37), 393 (22), 338 (20), 304 (30), 288 (60), 273 (49), 250 (11), 219 (75), 167 (13), 149 (35), 132 (13), 104 (33), 84 (65), 66 (90). Anal. Calcd for $\text{C}_{30}\text{H}_{18}\text{Cl}_2\text{N}_6\text{O}_3$ (580.08) C, 61.97; H, 3.12; N, 14.45. Found: C, 62.47; H, 3.17; N, 14.52.

5,5''-Dichloro-4'-methoxy-2,2''-dioxo-2'-(*p*-tolyl)-1',2'-dihydro-4a'*H*-dispiro[indoline-3,5'-cyclopenta[*d*]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3j)



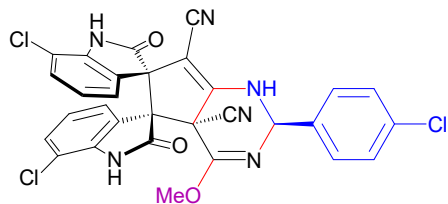
Colorless powder (270 mg from 1 mmol scale, 91% yield); mp: 269-272 °C (dec); IR (KBr, cm^{-1}) 3441, 3290, 2215, 1720, 1672, 1640, 1625; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 11.18 (s, 1H), 10.95 (s, 1H), 9.79 (s, 1H), 7.71 (d, $J = 2.4$ Hz, 1H), 7.40 (d, $J = 7.9$ Hz, 2H), 7.36 (dd, $J = 8.4, 2.4$ Hz, 1H), 7.30 (d, $J = 7.7$ Hz, 2H), 7.28 (dd, $J = 8.5, 2.5$ Hz, 1H), 7.23 (d, $J = 2.4$ Hz, 1H), 6.80 (dd, $J = 8.1, 4.2$ Hz, 1H), 6.71 (dd, $J = 8.0, 4.0$ Hz, 1H), 6.16 (s, 1H), 3.51 (s, 3H), 2.36 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.3, 173.7, 154.6, 153.1, 141.9, 141.8, 138.1, 137.2, 130.5, 130.4, 129.1, 127.4, 126.2, 126.0, 125.9, 125.7, 125.7, 124.2, 115.5, 113.3, 111.6, 111.6, 71.5, 70.5, 62.8, 58.2, 54.6, 46.5, 20.9. MS: m/z (%): 542 (26), 526 (3), 510 (6), 485(2), 427 (6), 410 (11), 392 (3), 365 (6), 346 (8), 330 (4), 304 (3), 288 (13), 264 (26), 235 (2), 167 (12), 138 (11), 116 (16), 84 (67), 66 (100). Anal. Calcd for $\text{C}_{31}\text{H}_{20}\text{Cl}_2\text{N}_6\text{O}_3$ (594.10) C, 62.53; H, 3.39; N, 14.11. Found: C, 62.97; H, 3.44; N, 14.16.

7,7''-Dichloro-2'-(4-fluorophenyl)-4'-methoxy-2,2''-dioxo-1',2'-dihydro-4a'*H*-dispiro[indoline-3,5'-cyclopenta[*d*]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3k)



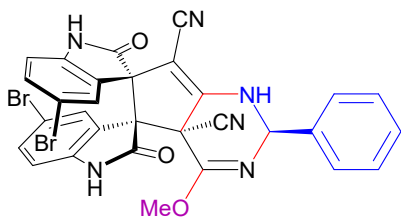
Colorless powder (265 mg from 1 mmol scale, 89% yield); mp: 255-258 °C (dec); IR (KBr, cm^{-1}) 3425, 3289, 3222, 1733, 1720, 1686, 1648, 1618; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 11.38 (s, 1H), 11.15 (s, 1H), 9.79 (s, 1H), 7.68 (d, $J = 7.7$ Hz, 1H), 7.58 (dd, $J = 8.4, 5.3$ Hz, 2H), 7.39 (d, $J = 8.0$ Hz, 1H), 7.34 (t, $J = 8.7$ Hz, 2H), 7.31 (d, $J = 8.4$ Hz, 1H), 7.23 (d, $J = 7.7$ Hz, 1H), 7.11 (t, $J = 8.0$ Hz, 1H), 7.03 (t, $J = 7.9$ Hz, 1H), 6.26 (s, 1H), 3.52 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.5, 173.9, 162.30 (d, $J = 245.9$ Hz), 154.9, 153.0, 140.8, 140.7, 136.4, 130.7, 130.5, 129.7 (d, $J = 7.5$ Hz), 125.3, 124.8, 124.2, 123.9, 123.5, 123.3, 115.4 (d, $J = 22.0$ Hz), 115.2, 114.1, 114.0, 113.2, 71.1, 70.8, 63.4, 59.1, 54.7, 46.6; ^{19}F NMR (471 MHz, $\text{DMSO-}d_6$) δ -113.3. MS: m/z (%): 541 (43), 517 (45), 477 (71), 462 (14), 435 (57), 407 (22), 391 (16), 368 (27), 339 (21), 302 (17), 268 (31), 230 (27), 203 (15), 176 (28), 150 (44), 123 (41), 84 (64), 66 (100). Anal. Calcd for $\text{C}_{30}\text{H}_{17}\text{Cl}_2\text{FN}_6\text{O}_3$ (598.07) C, 60.11; H, 2.86; N, 14.02. Found: C, 60.57; H, 2.84; N, 14.06.

7,7''-Dichloro-2'-(4-chlorophenyl)-4'-methoxy-2,2''-dioxo-1',2'-dihydro-4a'H-dispiro [indoline-3,5'-cyclopenta[d]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3l)



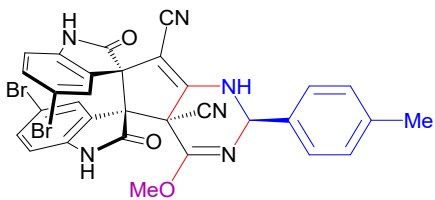
Colorless powder (265 mg from 1 mmol scale, 86% yield); mp: 266-267 °C (dec); IR (KBr, cm^{-1}) 3432, 3291, 3240, 2208, 1741, 1719, 1691, 1648, 1618; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 11.36 (s, 1H), 11.13 (s, 1H), 9.78 (s, 1H), 7.69 (d, $J = 7.6$ Hz, 1H), 7.58 (d, $J = 7.5$ Hz, 2H), 7.56 (d, $J = 7.4$ Hz, 2H), 7.38 (d, $J = 7.6$ Hz, 1H), 7.30 (d, $J = 8.0$ Hz, 1H), 7.23 (d, $J = 8.0$ Hz, 1H), 7.11 (t, $J = 7.8$ Hz, 1H), 7.02 (t, $J = 7.4$ Hz, 1H), 6.26 (s, 1H), 3.53 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.5, 173.9, 155.1, 153.1, 140.7, 140.7, 139.0, 133.4, 130.7, 130.6, 129.5, 128.6, 125.4, 124.8, 124.3, 123.9, 123.6, 123.4, 115.4, 114.1, 114.0, 113.2, 71.3, 70.8, 63.4, 59.1, 54.8, 46.6. MS: m/z (%): 548 (2), 532 (11), 476 (3), 449 (7), 419 (8), 347 (7), 330 (28), 302 (30), 284 (32), 257 (10), 239 (11), 203 (12), 166 (22), 138 (27), 119 (11), 102 (22), 84 (61), 66 (100). Anal. Calcd for $\text{C}_{30}\text{H}_{17}\text{Cl}_3\text{N}_6\text{O}_3$ (614.04) C, 58.51; H, 2.78; N, 13.65. Found: C, 58.96; H, 2.79; N, 13.70.

5,5''-Dibromo-4'-methoxy-2,2''-dioxo-2'-phenyl-1',2'-dihydro-4a'H-dispiro[indoline-3,5'-cyclopenta[d]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3m)



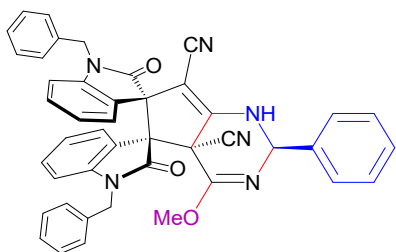
Colorless powder (283 mg from 1 mmol scale, 85% yield); mp: 265-266 °C (dec); IR (KBr, cm^{-1}) 3425, 3374, 2211, 1719, 1684, 1643, 1617; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 11.19 (s, 1H), 10.95 (s, 1H), 9.83 (s, 1H), 7.84 (d, $J = 2.2$ Hz, 1H), 7.53 – 7.50 (m, 4H), 7.48 (dd, $J = 8.2, 2.2$ Hz, 1H), 7.45 (t, $J = 6.6$ Hz, 1H), 7.41 (dd, $J = 8.3, 2.1$ Hz, 1H), 7.36 (d, $J = 2.2$ Hz, 1H), 6.76 (d, $J = 8.4$ Hz, 1H), 6.67 (d, $J = 8.2$ Hz, 1H), 6.22 (s, 1H), 3.52 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.2, 173.6, 154.6, 153.1, 142.3, 142.2, 140.1, 133.3, 133.2, 128.7, 128.7, 128.6, 128.4, 127.5, 126.0, 124.5, 115.5, 113.8, 113.7, 113.2, 112.1, 112.1, 71.6, 70.6, 62.7, 58.1, 54.6, 46.5. MS: m/z (%): 542 (5), 518(3), 419 (4), 301 (5), 273 (6), 250 (2), 172 (3), 147 (47), 119 (100), 92 (88), 66 (29). Anal. Calcd for $\text{C}_{30}\text{H}_{18}\text{Br}_2\text{N}_6\text{O}_3$ (667.98) C, 53.75; H, 2.71; N, 12.54. Found: C, 59.27; H, 2.79; N, 12.50.

5,5''-Dibromo-4'-methoxy-2,2''-dioxo-2'-(*p*-tolyl)-1',2'-dihydro-4a'*H*-dispiro[indoline-3,5'-cyclopenta[*d*]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3n)



Colorless powder (293 mg from 1 mmol scale, 86% yield); mp: 260-262 °C (dec); IR (KBr, cm^{-1}) 3316, 3231, 2216, 1726, 1686, 1644, 1617; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 11.18 (s, 1H), 10.94 (s, 1H), 9.77 (s, 1H), 7.83 (d, $J = 2.2$ Hz, 1H), 7.48 (dd, $J = 8.3, 2.1$ Hz, 1H), 7.42 – 7.39 (m, 3H), 7.35 (d, $J = 2.1$ Hz, 1H), 7.31 (d, $J = 8.1$ Hz, 2H), 6.75 (d, $J = 8.4$ Hz, 1H), 6.66 (d, $J = 8.4$ Hz, 1H), 6.16 (s, 1H), 3.51 (s, 3H), 2.37 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.2, 173.6, 154.6, 153.1, 142.3, 142.2, 138.1, 137.2, 133.3, 133.3, 129.0, 128.7, 128.4, 127.4, 126.0, 124.5, 115.5, 113.8, 113.6, 113.2, 112.1, 112.0, 71.5, 70.5, 62.7, 58.1, 54.6, 46.5, 20.9. MS: m/z (%): 544 (2), 489 (6), 473 (7), 443 (8), 420 (19), 392 (9), 264 (44), 211 (31), 181 (23), 132 (25), 116 (58), 91 (100), 66 (35). Anal. Calcd for $\text{C}_{31}\text{H}_{20}\text{Br}_2\text{N}_6\text{O}_3$ (682.00) C, 54.41; H, 2.95; N, 12.28. Found: C, 54.86; H, 3.02; N, 12.35.

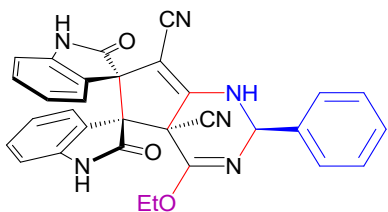
1,1''-Dibenzyl-4'-methoxy-2,2''-dioxo-2'-phenyl-1',2'-dihydro-4a'*H*-dispiro[indoline-3,5'-cyclopenta[*d*]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3o)



Colorless powder (312 mg from 1 mmol scale, 90% yield); mp: 251-253 °C (dec); IR (KBr, cm^{-1}) 3422, 3283, 2213, 1718, 1678, 1643, 1610; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 9.88 (s, 1H), 7.81 (d, $J = 7.7$ Hz, 1H), 7.58 (d, $J = 7.3$ Hz, 2H), 7.52 (t, $J = 7.6$ Hz, 2H), 7.45 (t, $J = 7.4$ Hz, 1H),

7.33 – 7.28 (m, 2H), 7.26 – 7.19 (m, 7H), 7.07 – 7.04 (m, 2H), 7.02 – 6.98 (m, 3H), 6.93 (t, $J = 7.6$ Hz, 1H), 6.81 (d, $J = 8.0$ Hz, 1H), 6.68 (d, $J = 8.0$ Hz, 1H), 6.26 (s, 1H), 4.84 – 4.72 (m, 2H), 3.40 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 175.2, 172.5, 154.7, 153.3, 143.5, 143.4, 140.3, 135.3, 130.4, 130.3, 128.7, 128.6, 128.6, 128.5, 127.6, 127.5, 127.4, 127.4, 127.0, 126.0, 125.6, 123.1, 123.0, 122.9, 121.7, 115.6, 113.6, 109.9, 109.7, 71.7, 71.6, 62.6, 58.3, 54.7, 46.7, 43.3, 43.0. MS: m/z (%): 533 (2), 469 (10), 442 (25), 414 (10), 385 (3), 323 (10), 306 (8), 250 (11), 223 (10), 204 (3), 118 (5), 91 (15), 91 (100), 65 (15). Anal. Calcd for $\text{C}_{44}\text{H}_{32}\text{N}_6\text{O}_3$ (692.25) C, 76.28; H, 4.66; N, 12.13. Found: C, 76.87; H, 4.71; N, 12.20.

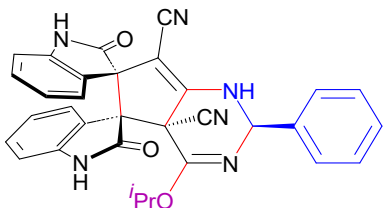
4'-Ethoxy-2,2''-dioxo-2'-phenyl-1',2'-dihydro-4a'*H*-dispiro[indoline-3,5'-cyclopenta[*d*]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3p).



Colorless powder (206 mg from 1 mmol scale, 78% yield); mp: 251-253 °C (dec); IR (KBr, cm^{-1}) 3380, 3196, 3085, 2255, 2207, 1727 1645, 1618; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.77 (s, 1H), 10.56 (s, 1H), 9.60 (s, 1H), 7.74 (d, $J = 7.8$ Hz, 1H), 7.54 (dd, $J = 8.4, 1.4$ Hz, 2H),

7.49 (td, $J = 6.7, 1.9$ Hz, 2H), 7.45 – 7.40 (m, 1H), 7.30 (d, $J = 7.7$ Hz, 1H), 7.24 (t, $J = 7.7$ Hz, 1H), 7.17 (t, $J = 7.7$ Hz, 1H), 7.02 (t, $J = 7.7$ Hz, 1H), 6.95 (t, $J = 7.6$ Hz, 1H), 6.70 (d, $J = 7.7$ Hz, 1H), 6.62 (d, $J = 7.5$ Hz, 1H), 6.15 (s, 1H), 3.94 [(AB) X_3 system, $\Delta\nu_{\text{AB}} = 70.8$ Hz, $^2J_{\text{AX}} = ^2J_{\text{BX}} = 7.0$ Hz, CH_2], 0.97 (t, $J = 7.0$ Hz, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) δ 176.7, 174.0, 154.6, 153.2, 143.0, 142.9, 140.4, 130.0, 130.0, 128.5, 128.4, 127.5, 126.2, 125.6, 124.0, 122.6, 121.9, 121.7, 115.8, 113.7, 109.6, 109.5, 71.7, 71.5, 63.2, 62.6, 58.6, 46.4, 18.5. MS: m/z (%): 527 (8), 500 (7), 428 (8), 339 (9), 296 (7), 262 (16), 236 (8), 219 (64), 190 (6), 165 (16), 141 (15), 116 (32), 84 (71), 66 (100). Anal. Calcd for $\text{C}_{31}\text{H}_{22}\text{N}_6\text{O}_3$ (526.18) C, 70.71; H, 4.21; N, 15.96. Found: C, 71.16; H, 4.25; N, 16.02.

**4'-Isopropoxy-2,2''-dioxo-2'-phenyl-1',2'-dihydro-4a'H-dispiro[indoline-3,5'
cyclopenta[d]pyrimidine-6',3''-indoline]-4a',7'-dicarbonitrile (3q).**



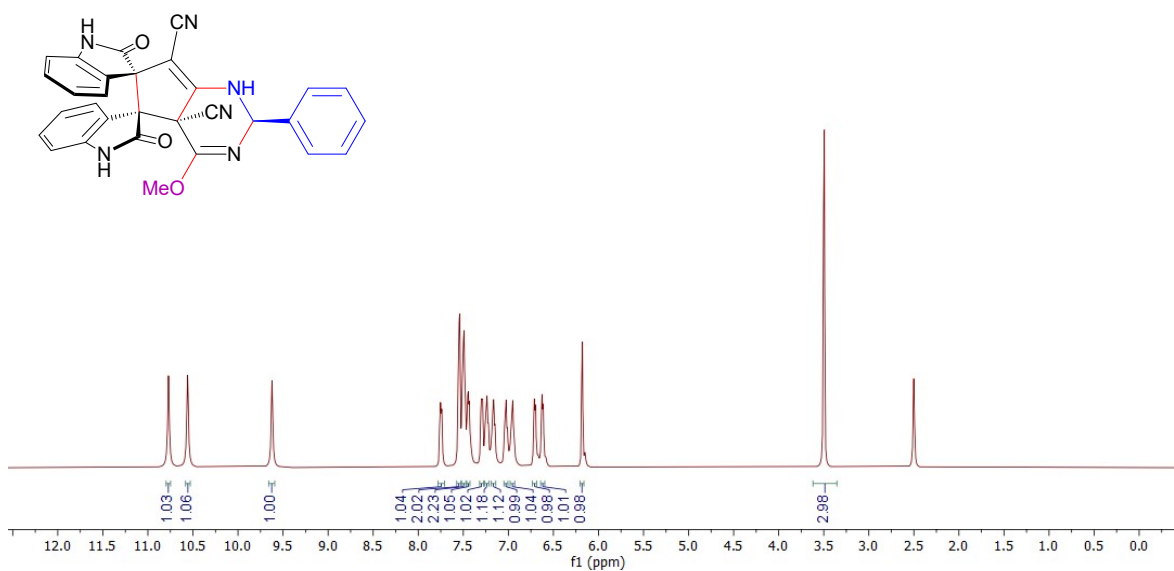
Colorless powder (98 mg from 1 mmol scale, 36% yield);
mp: 262-263 °C (dec); IR (KBr, cm^{-1}) 3377, 3251, 2205,
1712, 1643, 1617; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ
10.76 (s, 1H), 10.55 (s, 1H), 9.59 (s, 1H), 7.72 (d, $J = 7.6$
Hz, 1H), 7.53 – 7.51 (m, 2H), 7.49 – 7.46 (m, 2H), 7.42 (t,

$J = 7.1$ Hz, 1H), 7.29 (d, $J = 7.7$ Hz, 1H), 7.23 (t, $J = 7.7$ Hz, 1H), 7.16 (t, $J = 7.7$ Hz, 1H), 7.01 (t,
 $J = 7.7$ Hz, 1H), 6.94 (t, $J = 7.7$ Hz, 1H), 6.69 (d, $J = 7.8$ Hz, 1H), 6.61 (d, $J = 7.8$ Hz, 1H), 6.14
(s, 1H), 4.50 – 4.42 (m, 1H), 1.72 (d, $J = 6.8$ Hz, 3H), 1.58 (d, $J = 6.9$ Hz, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR
(125 MHz, $\text{DMSO-}d_6$) δ 176.6, 173.9, 154.2, 153.0, 142.9, 142.8, 140.4, 130.0, 129.8, 128.4,
128.3, 127.6, 126.2, 125.8, 123.9, 122.7, 122.1, 121.9, 115.7, 113.7, 109.4, 109.2, 71.7, 71.5, 70.1,
62.6, 58.6, 46.6, 23.7, 22.2. MS: m/z (%): 539 (14), 513 (16), 428 (19), 379 (33), 290 (55), 233
(100), 132 (75), 77 (46). Anal. Calcd for $\text{C}_{32}\text{H}_{24}\text{N}_6\text{O}_3$ (540.19) C, 71.10; H, 4.48; N, 15.55. Found:
C, 71.46; H, 4.53; N, 15.60.

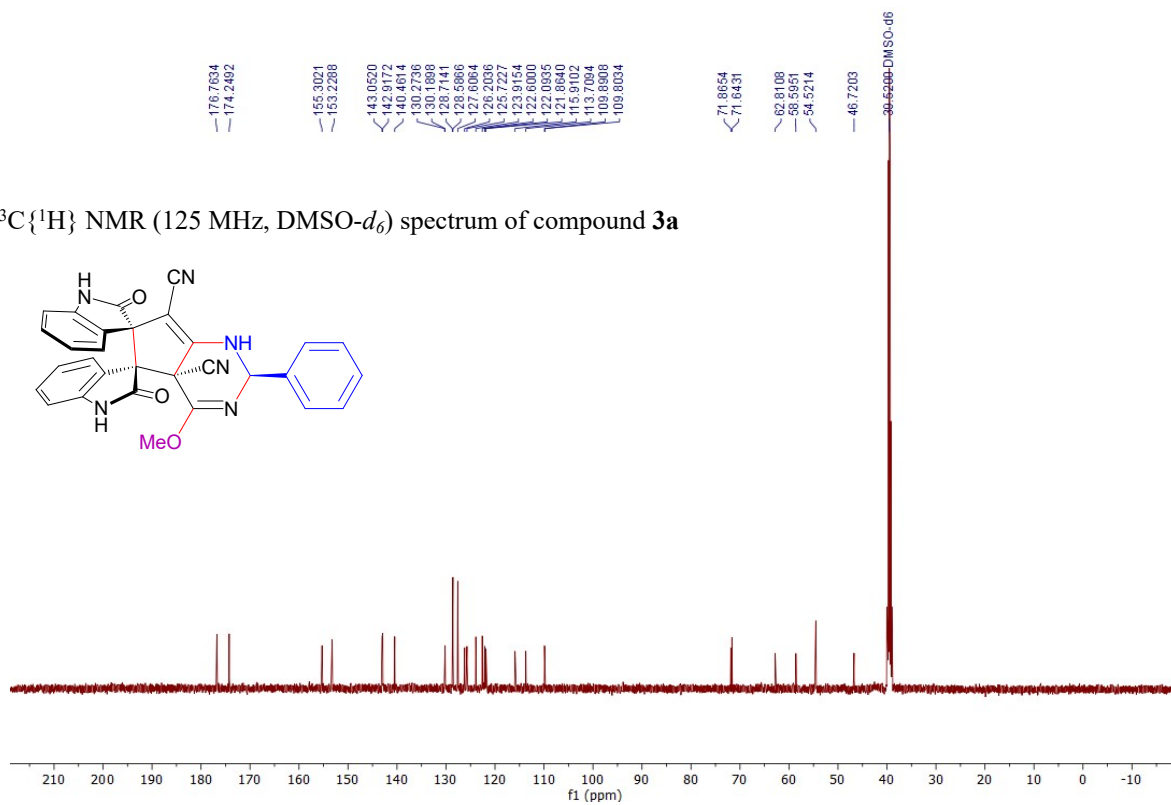
7. NMR Spectra



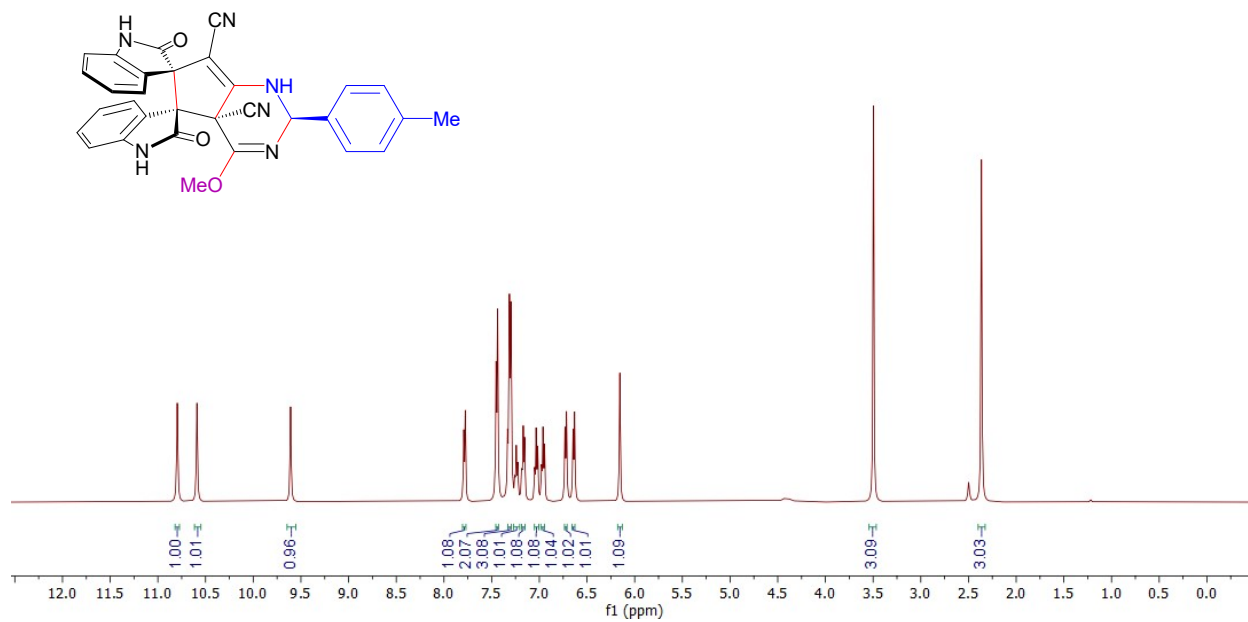
^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound **3a**

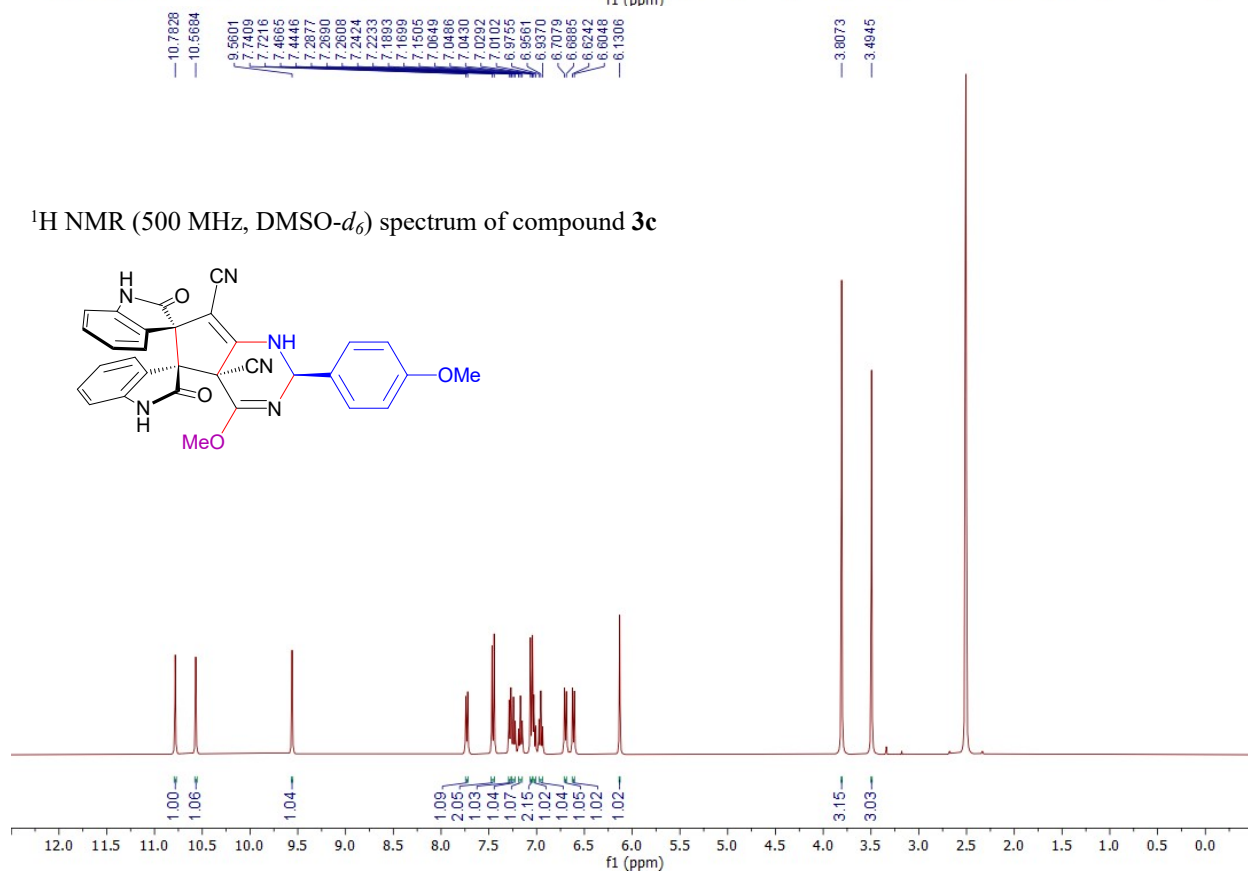
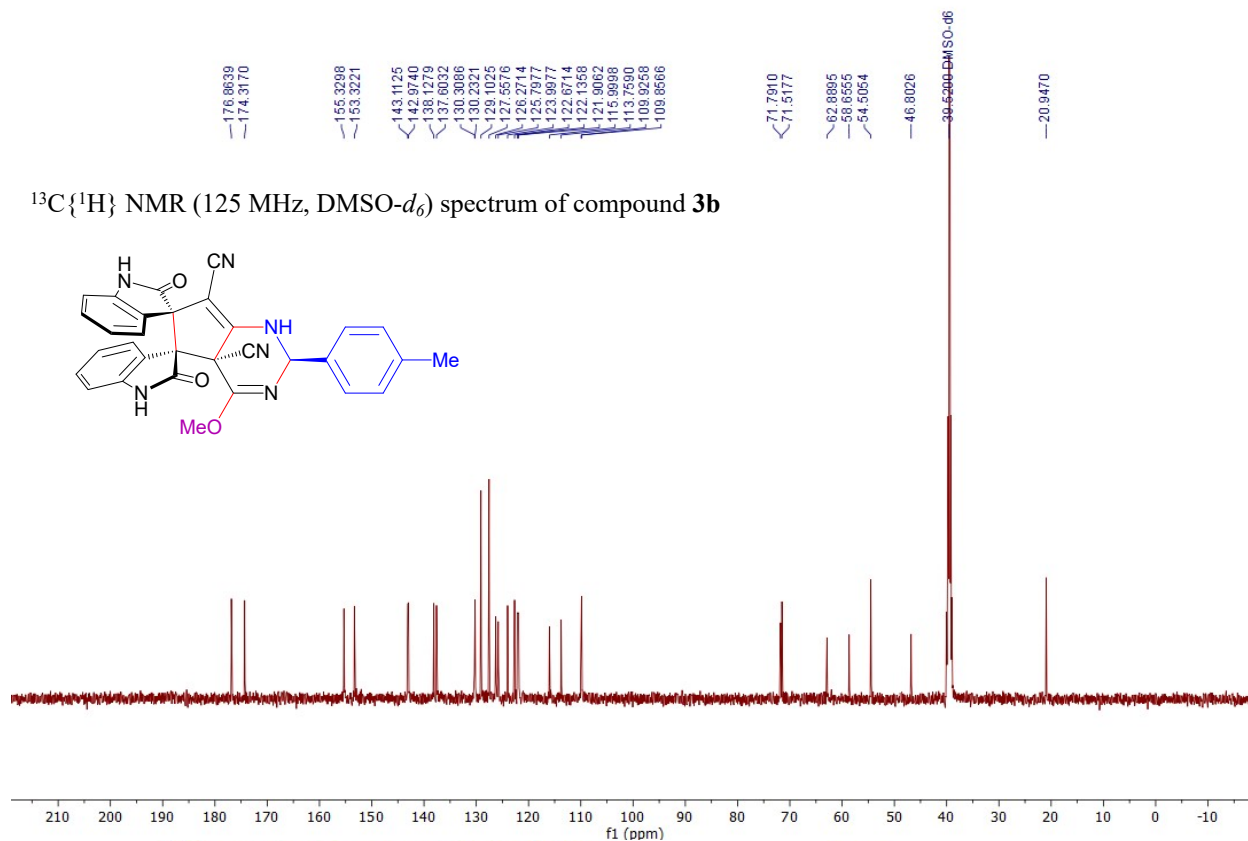


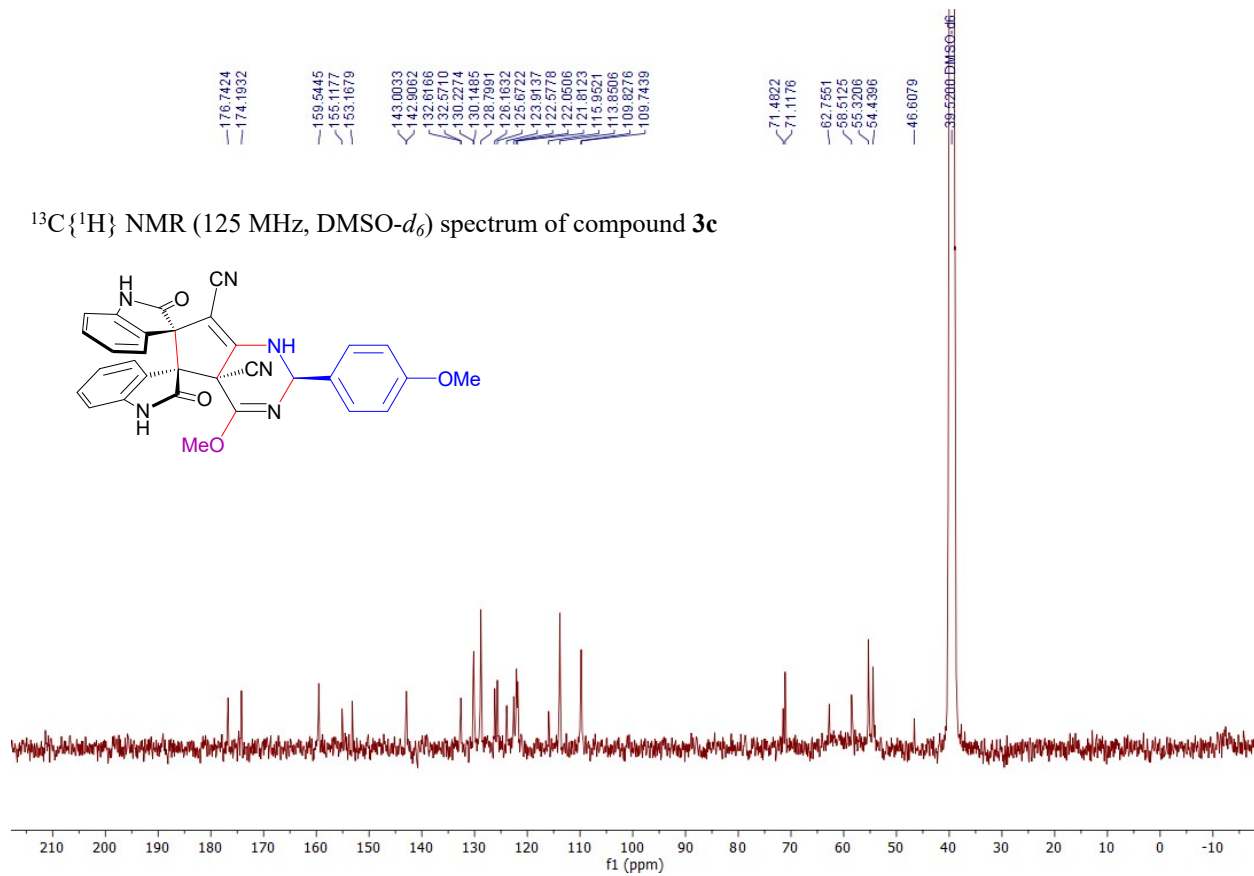
$^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) spectrum of compound **3a**



^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound **3b**

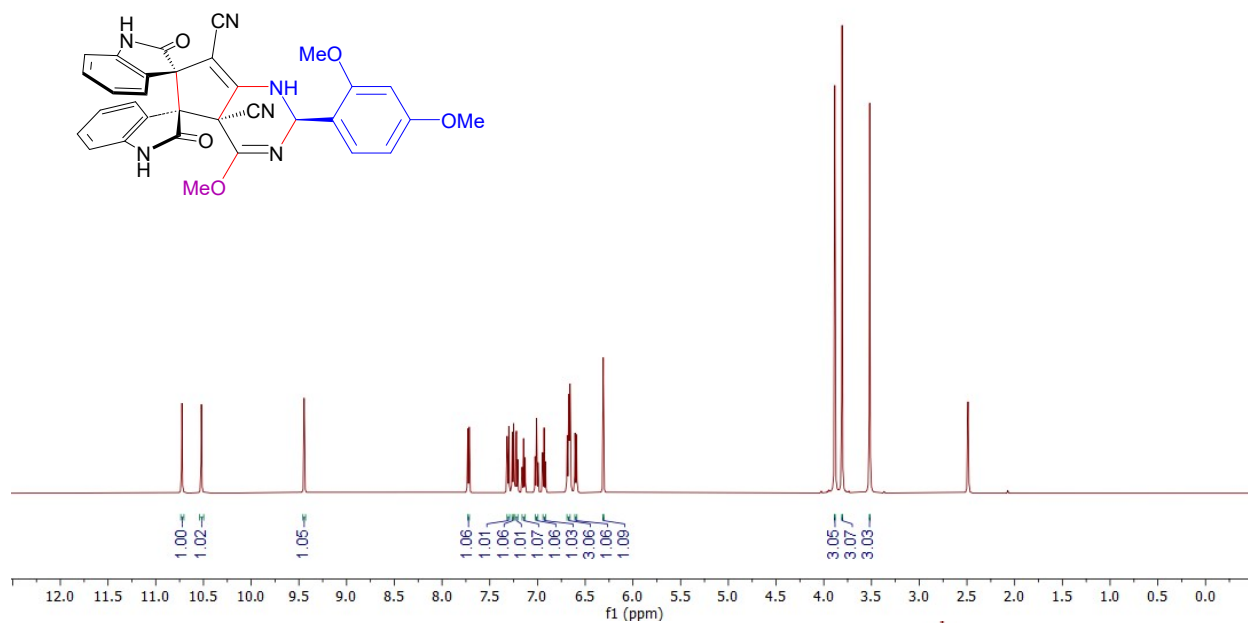






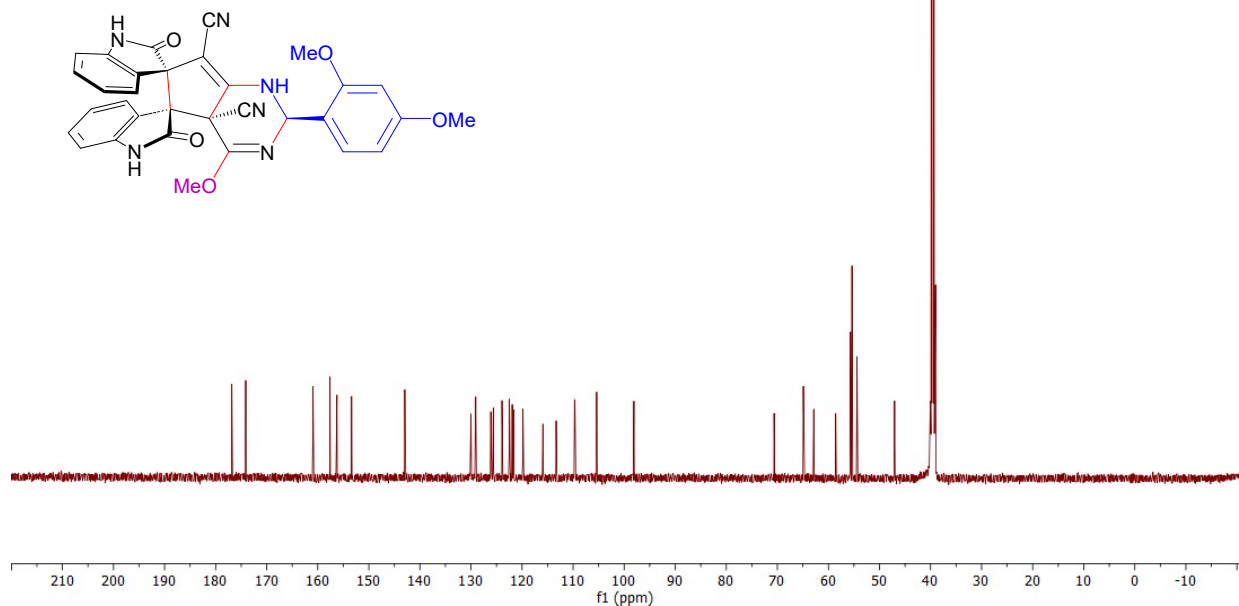
-10.7254
 -10.5220
 -9.4449
 7.7324
 7.7300
 7.7168
 7.7144
 7.3188
 7.3105
 7.3005
 7.2897
 7.2693
 7.2577
 7.2382
 7.2355
 7.2227
 7.2074
 7.2047
 7.1817
 7.1589
 7.1464
 7.1437
 7.1309
 7.1284
 7.0235
 7.0235
 7.0104
 7.0082
 6.9951
 6.9927
 6.9486
 6.9445
 6.9314
 6.9292
 6.9161
 6.9140
 6.8986
 6.8970
 6.8677
 6.8623
 6.8581
 6.8544
 6.8495
 6.6062
 6.5907
 6.3106
 3.8847
 3.8081
 3.5189

^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound **3d**



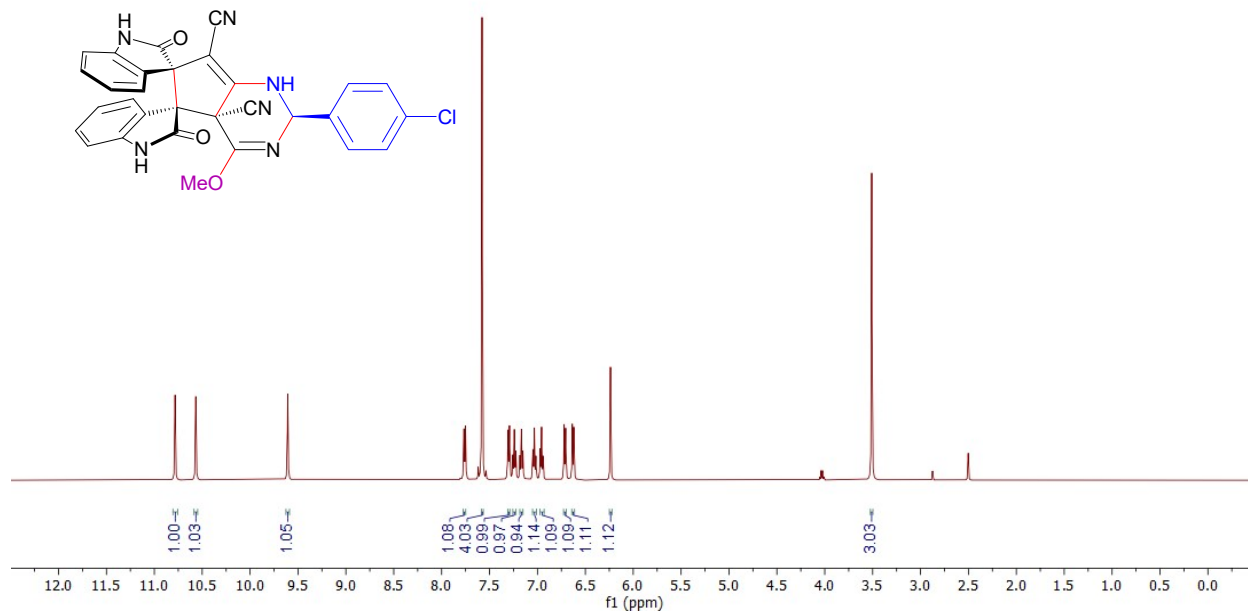
176.8347
 174.1032
 160.9335
 157.0334
 156.9136
 153.3774
 142.9550
 142.8400
 130.1143
 129.9814
 128.0638
 126.0147
 125.6147
 123.9256
 122.4710
 121.9199
 121.6741
 119.8188
 115.9139
 113.3172
 109.7135
 109.6422
 105.3805
 98.0860
 70.6365
 64.9119
 62.8663
 58.5768
 55.6868
 55.3855
 54.4023
 47.0405
 39.5200, $\text{DMSO-}d_6$

$^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) spectrum of compound **3d**

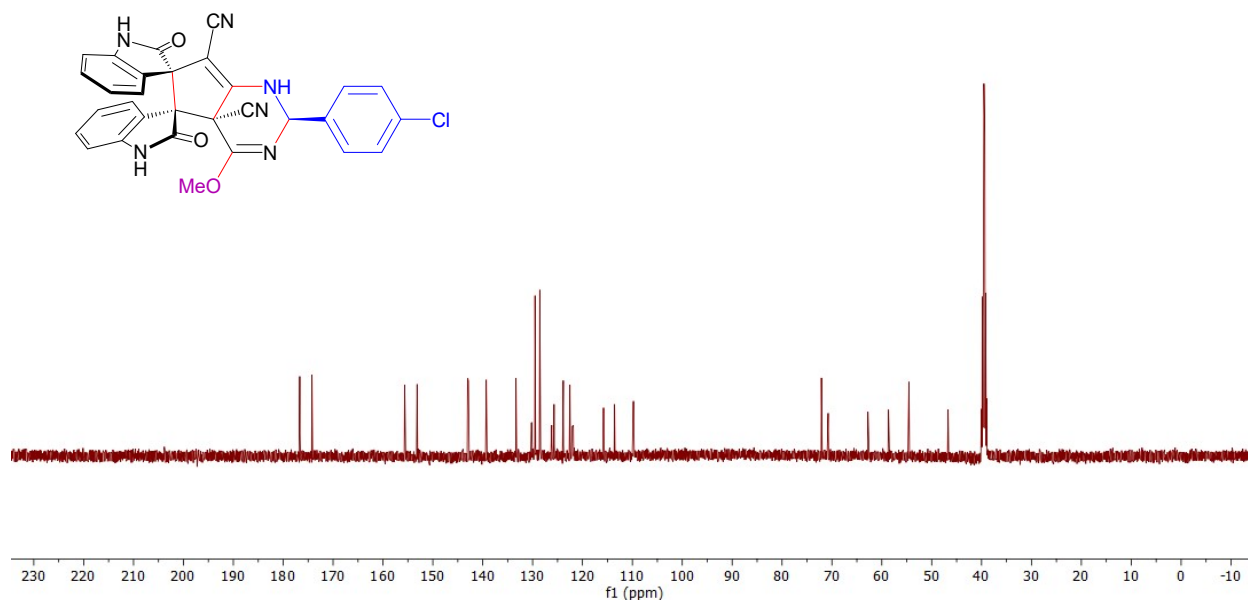




^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound **3e**

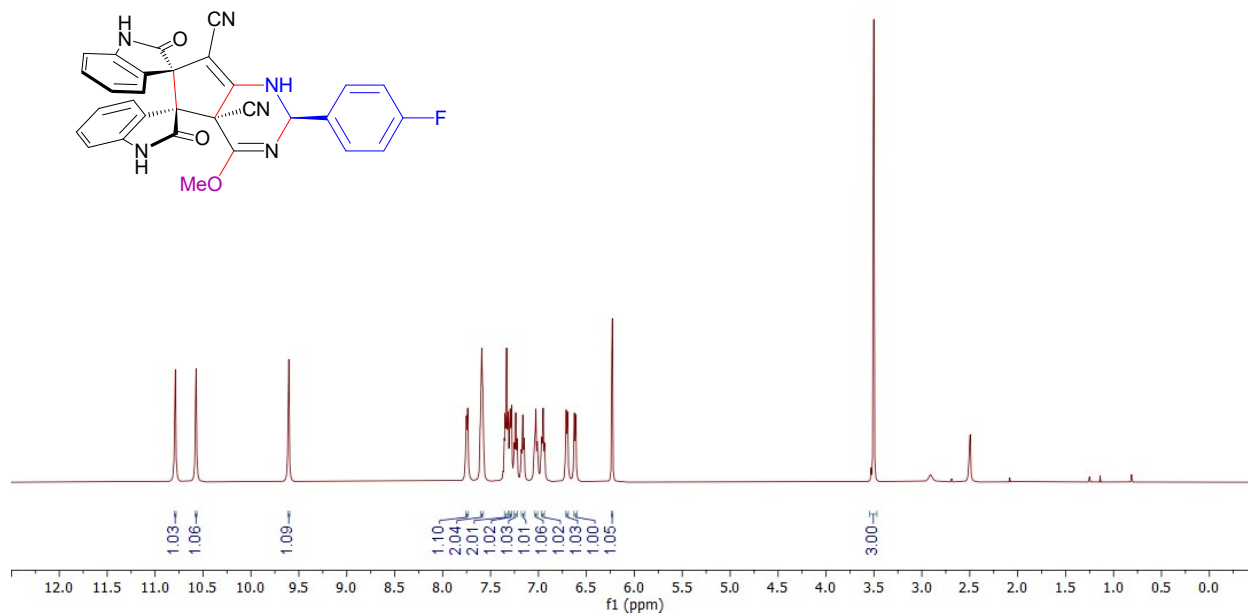


$^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) spectrum of compound **3e**

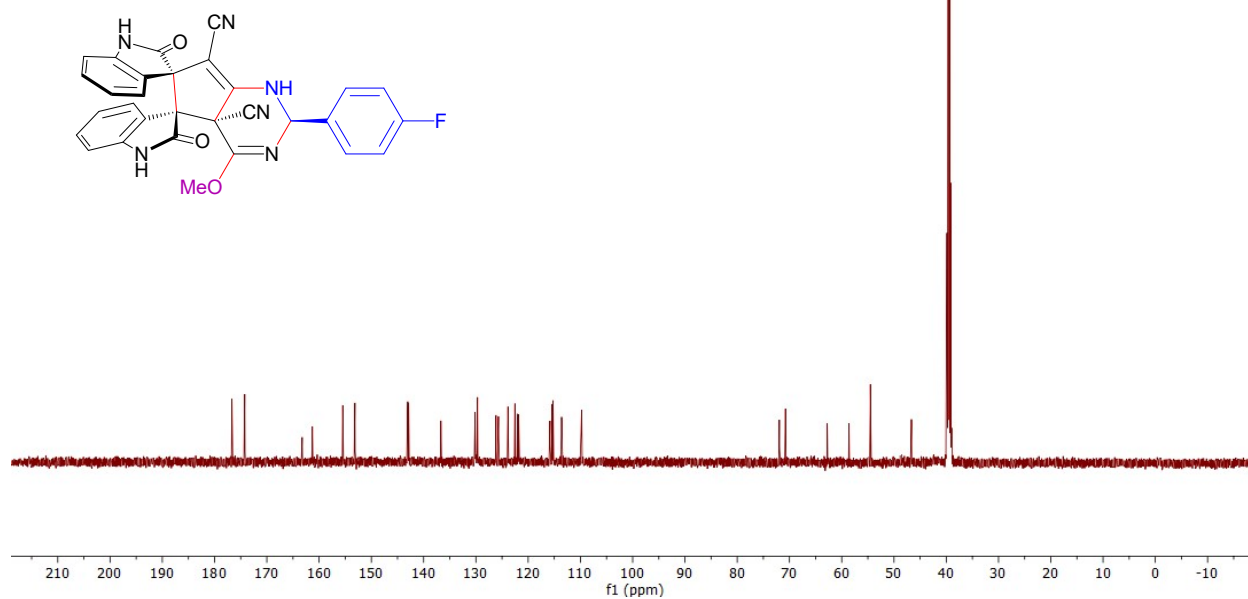




^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound **3f**

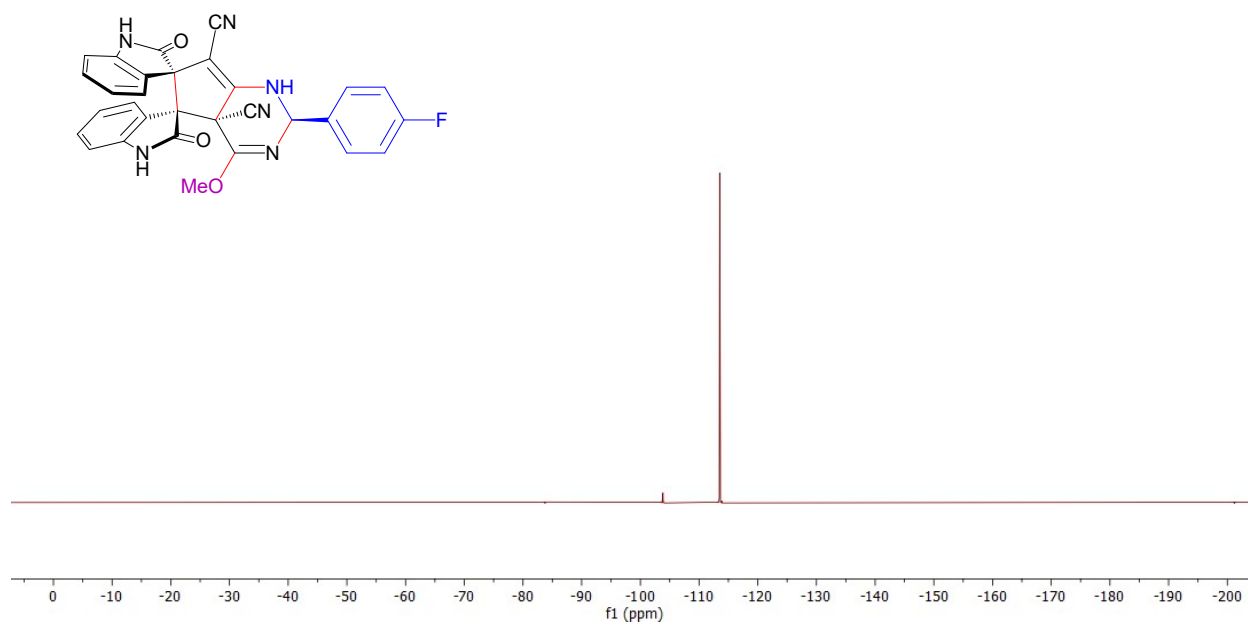


$^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) spectrum of compound **3f**



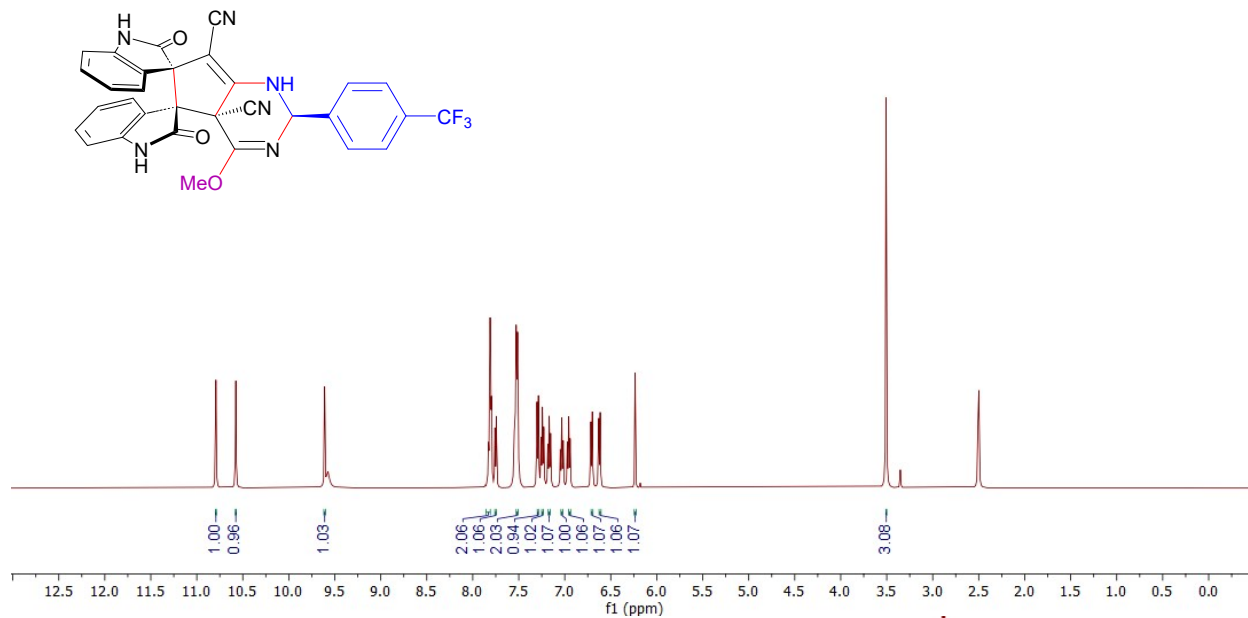
—113.5136

^{19}F NMR (471 MHz, $\text{DMSO-}d_6$) spectrum of compound **3f**

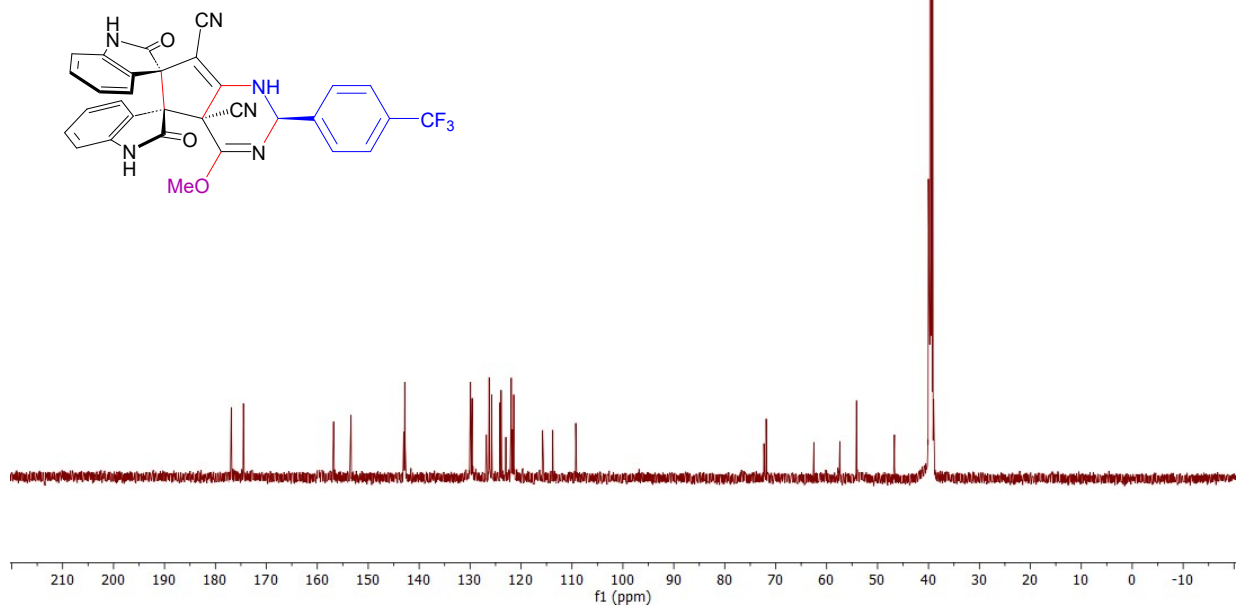




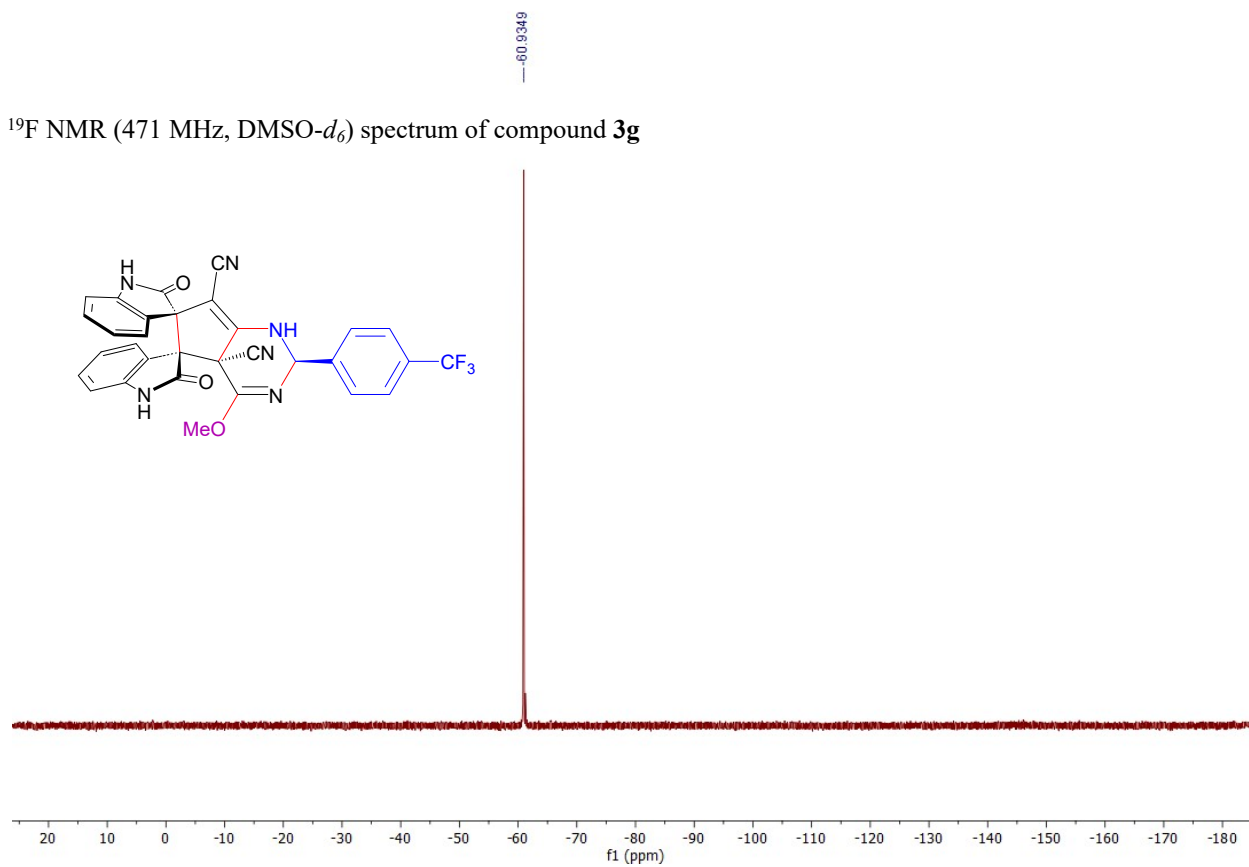
^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound **3g**

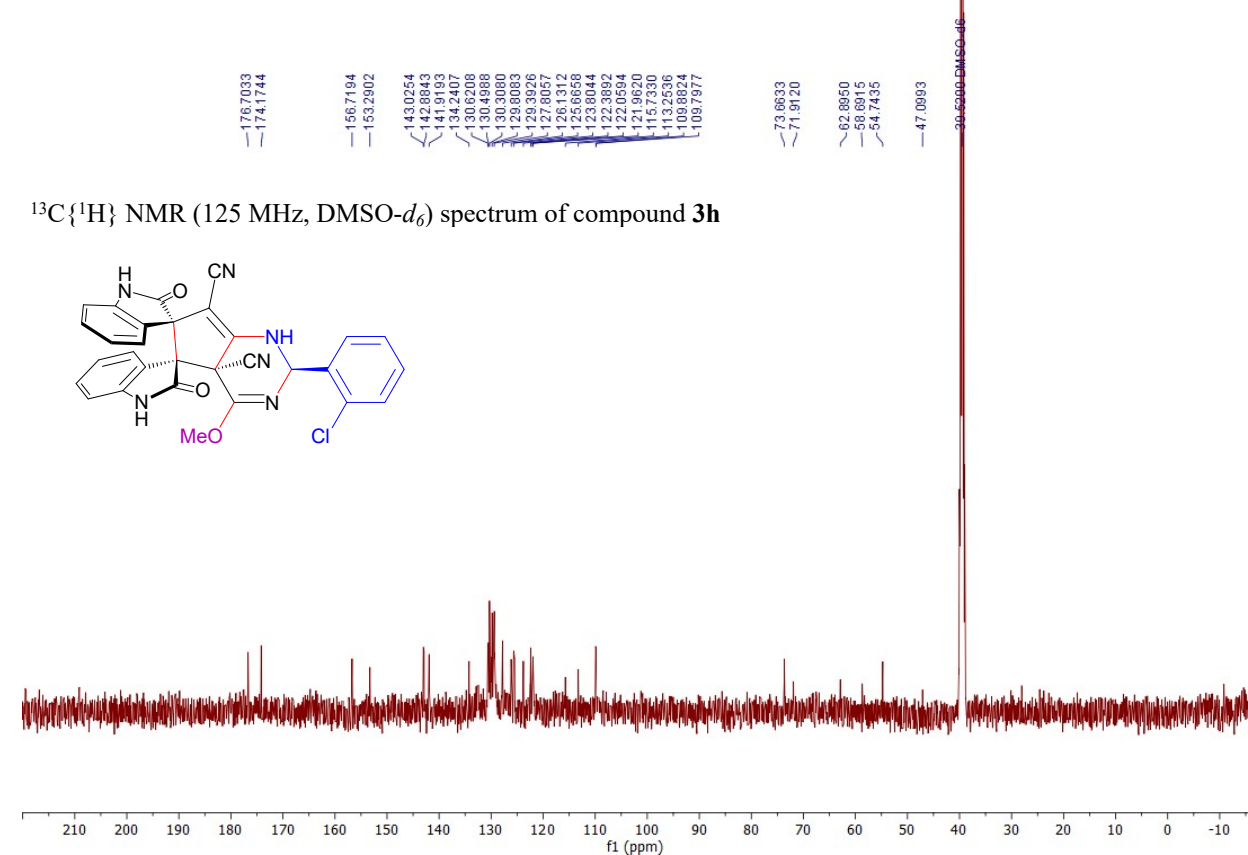
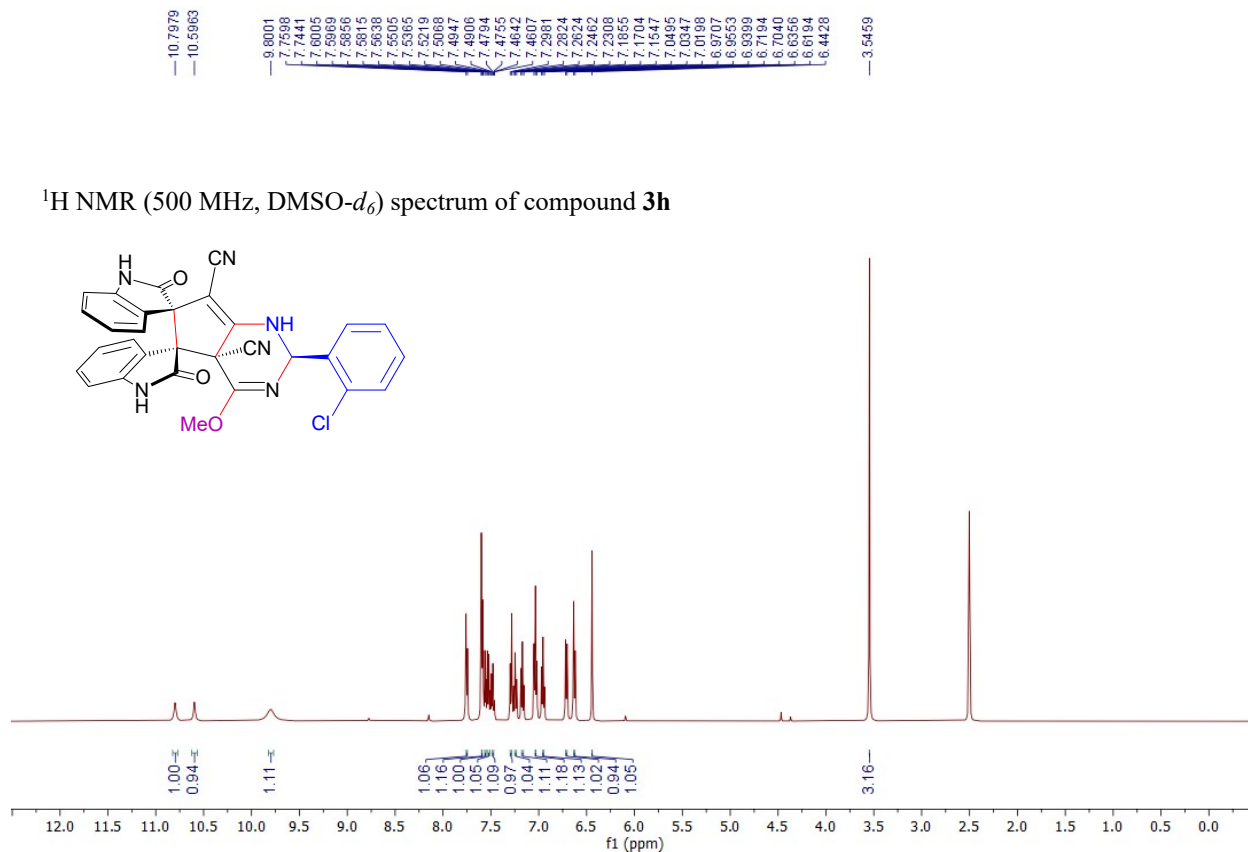


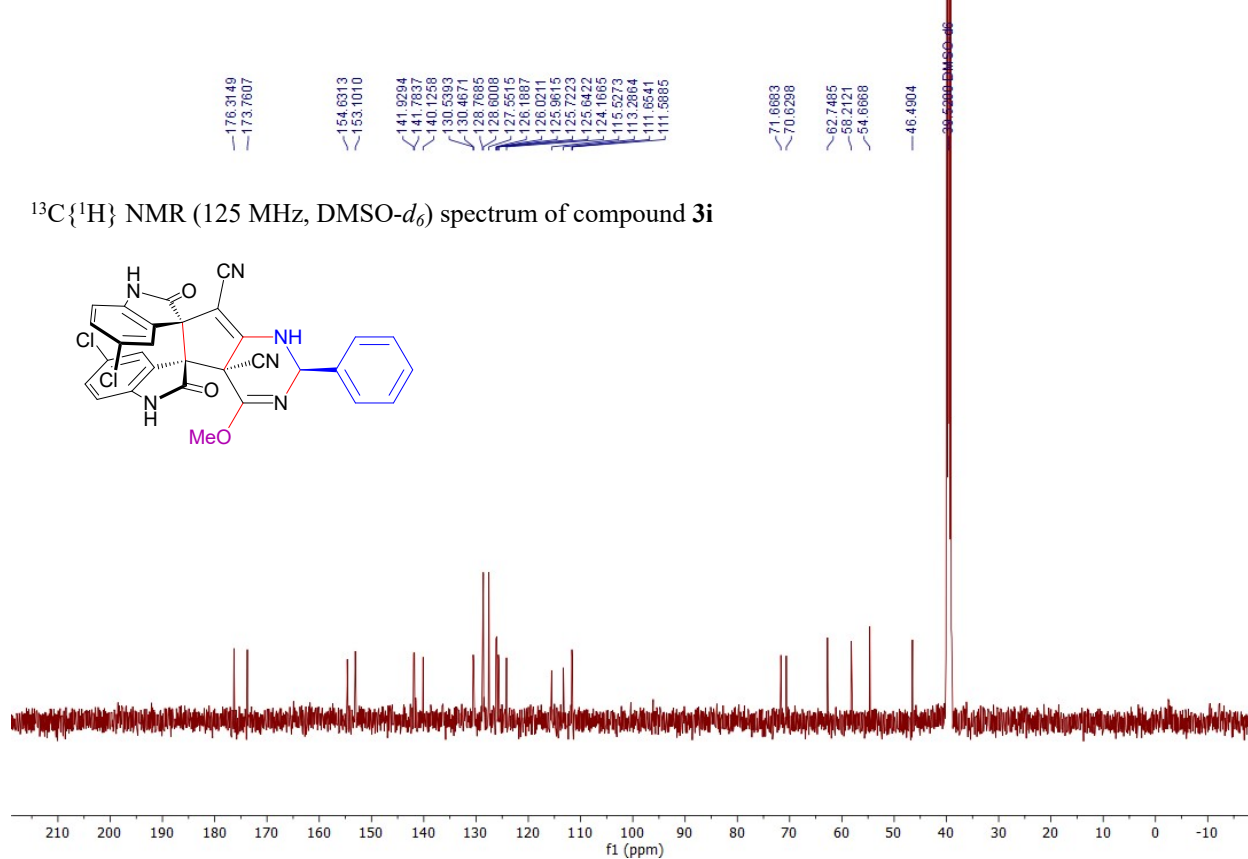
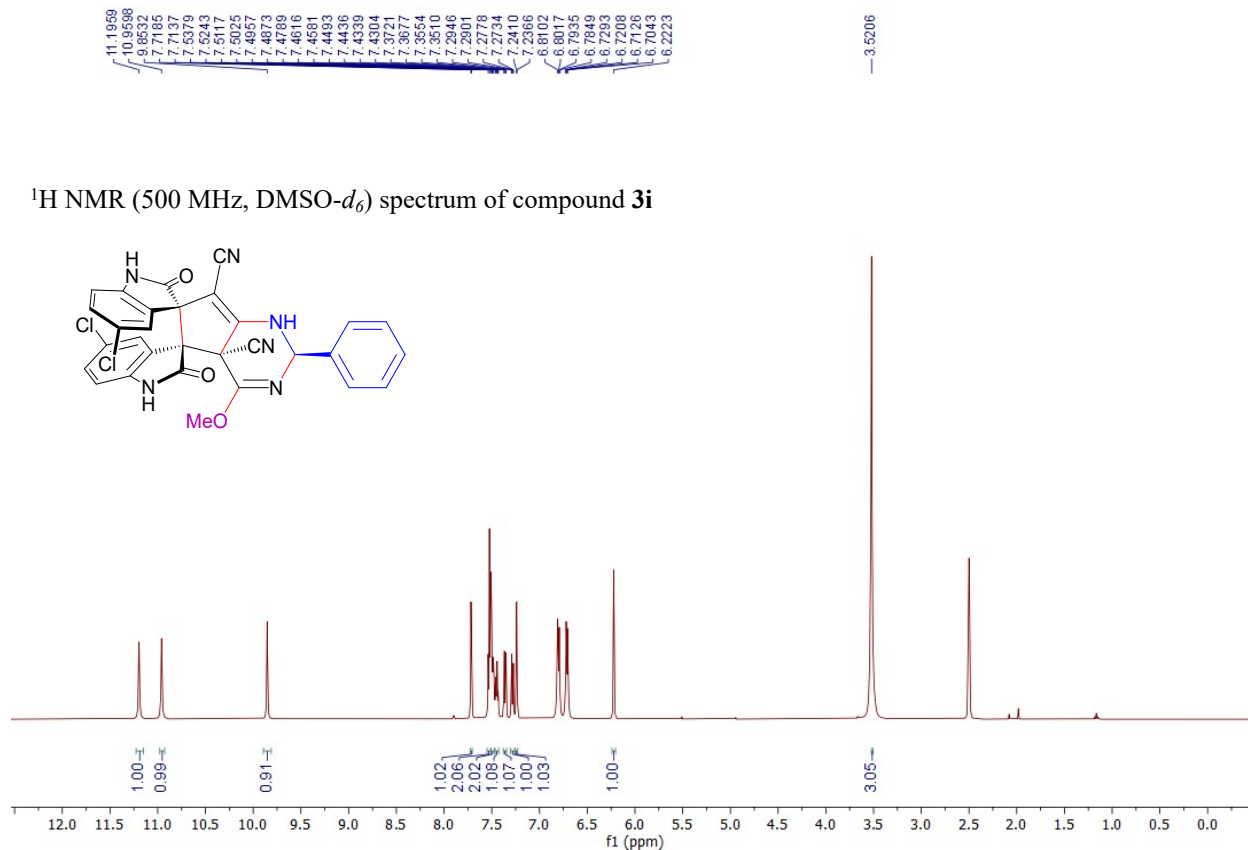
$^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) spectrum of compound **3g**



^{19}F NMR (471 MHz, $\text{DMSO-}d_6$) spectrum of compound **3g**

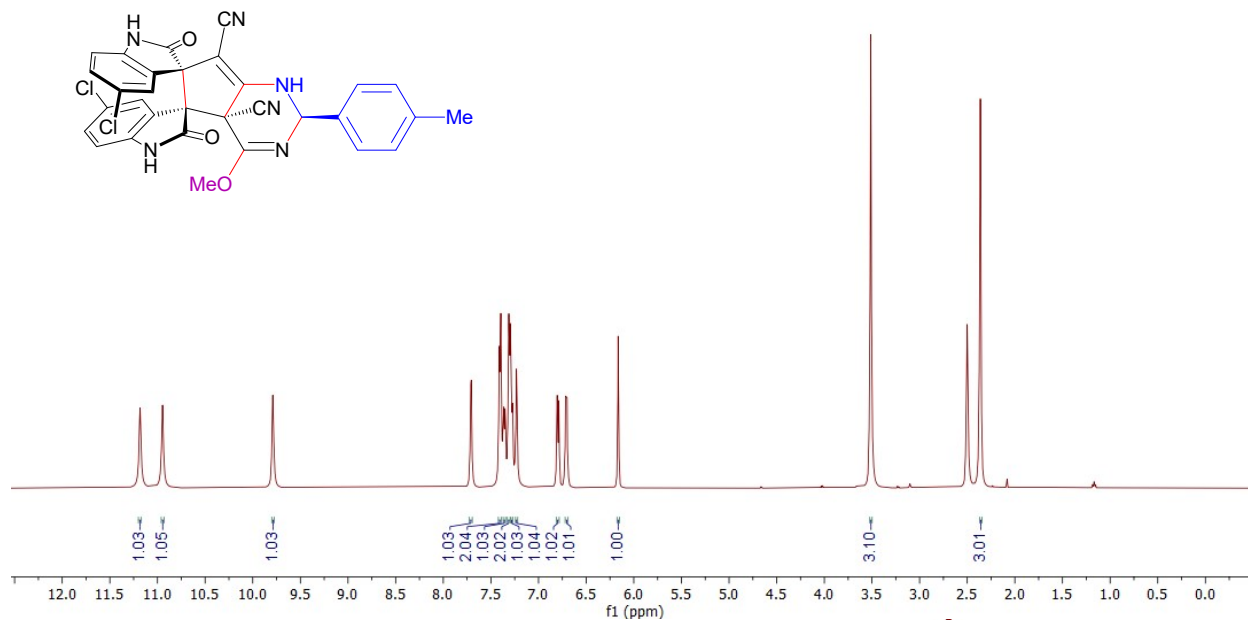




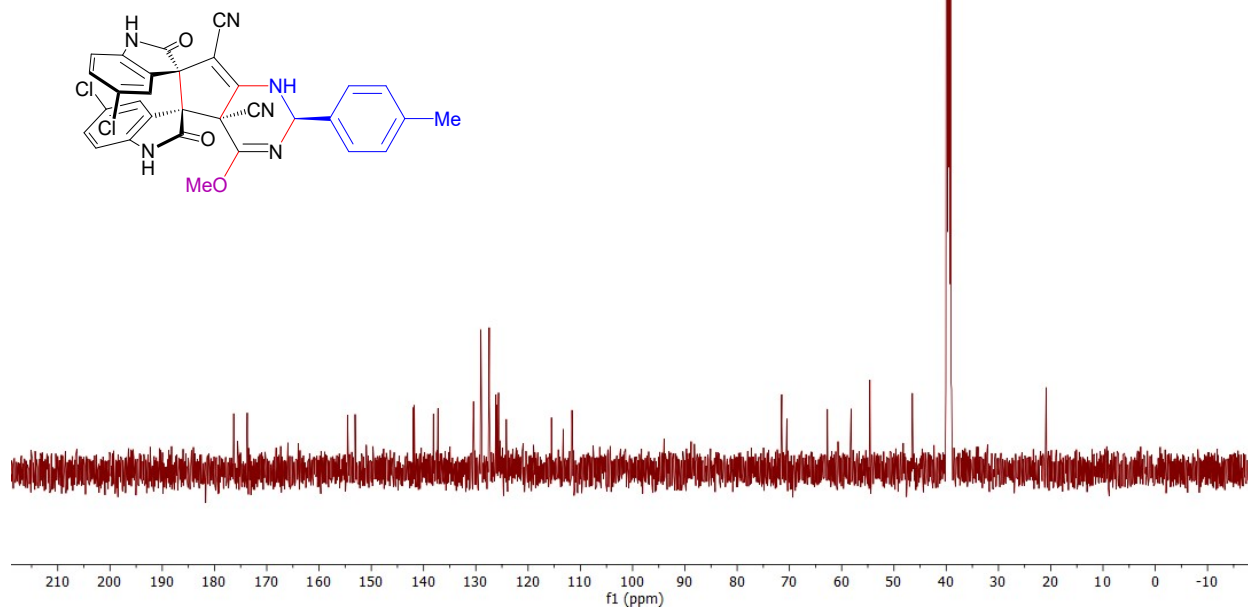


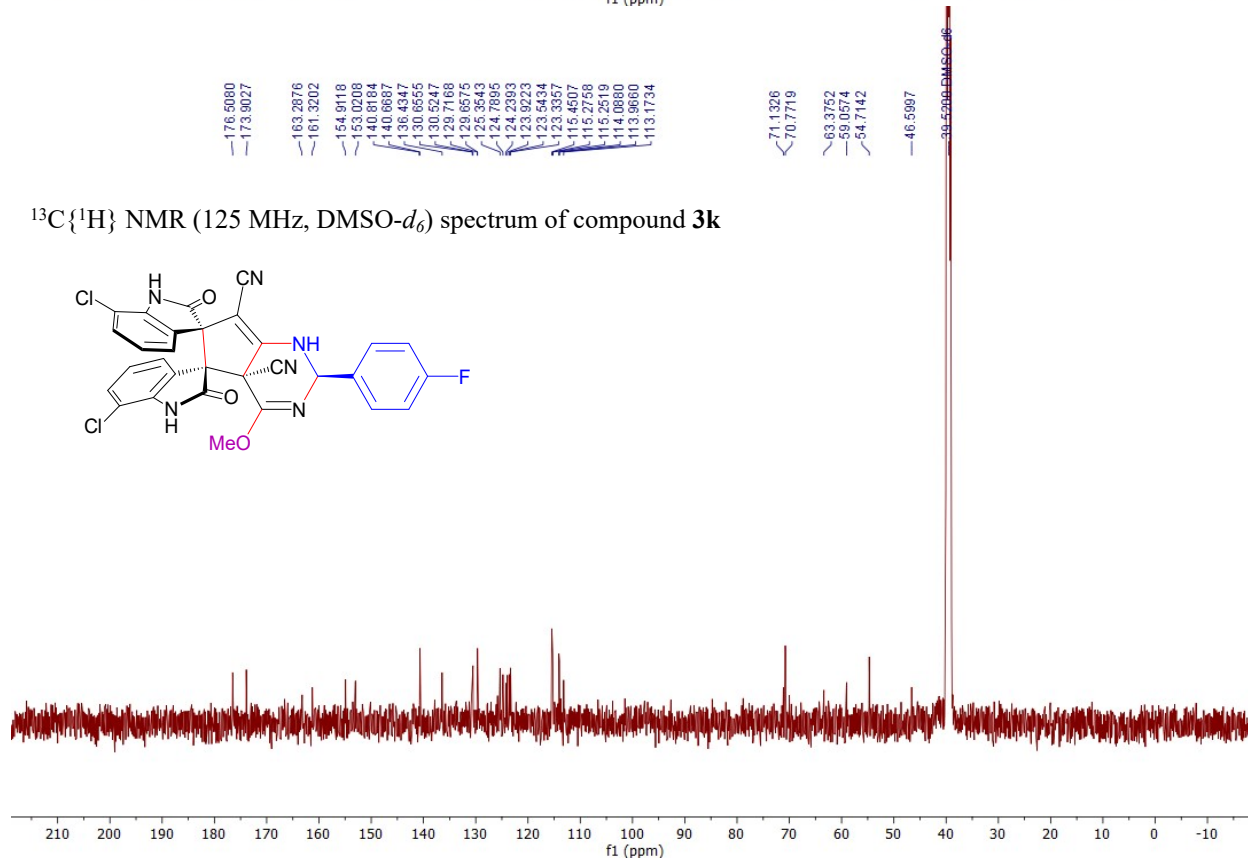
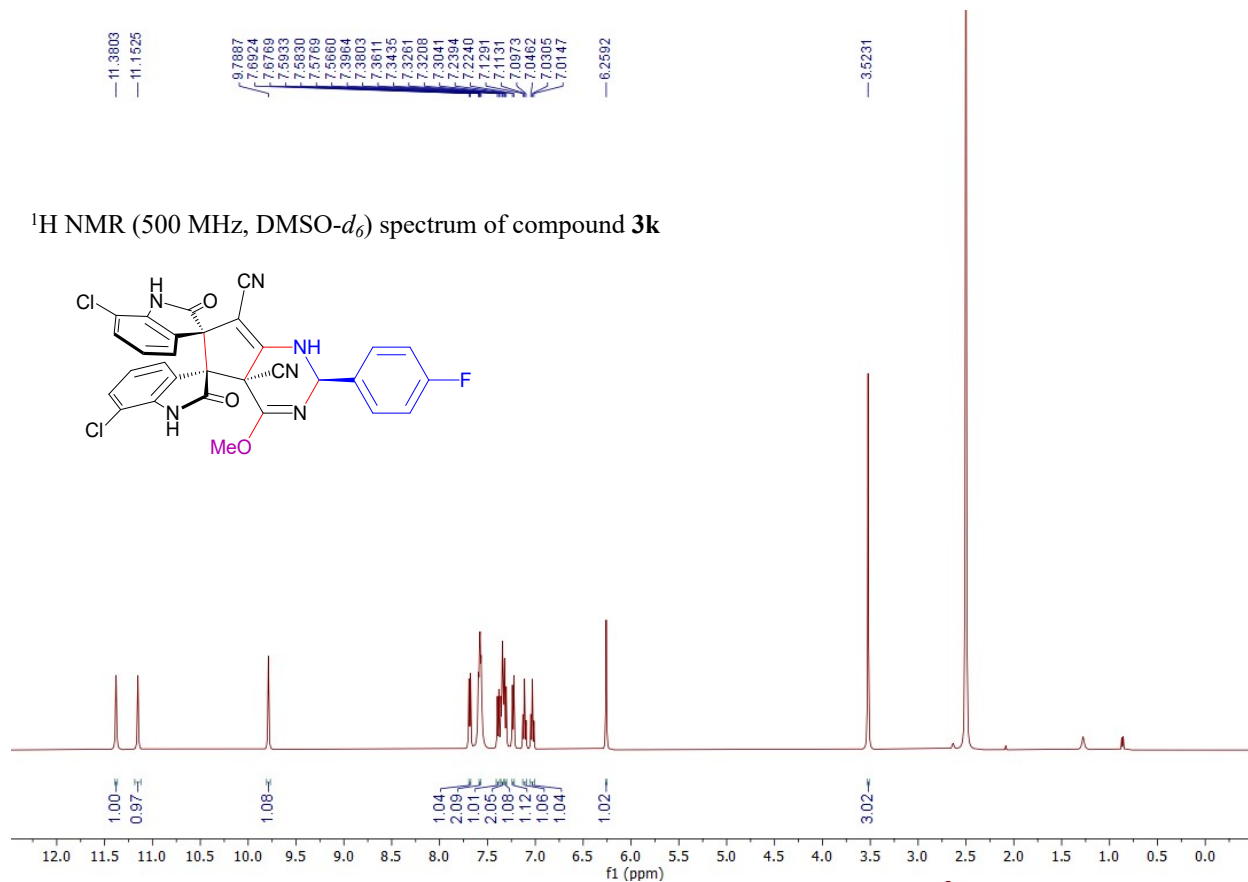


^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound **3j**

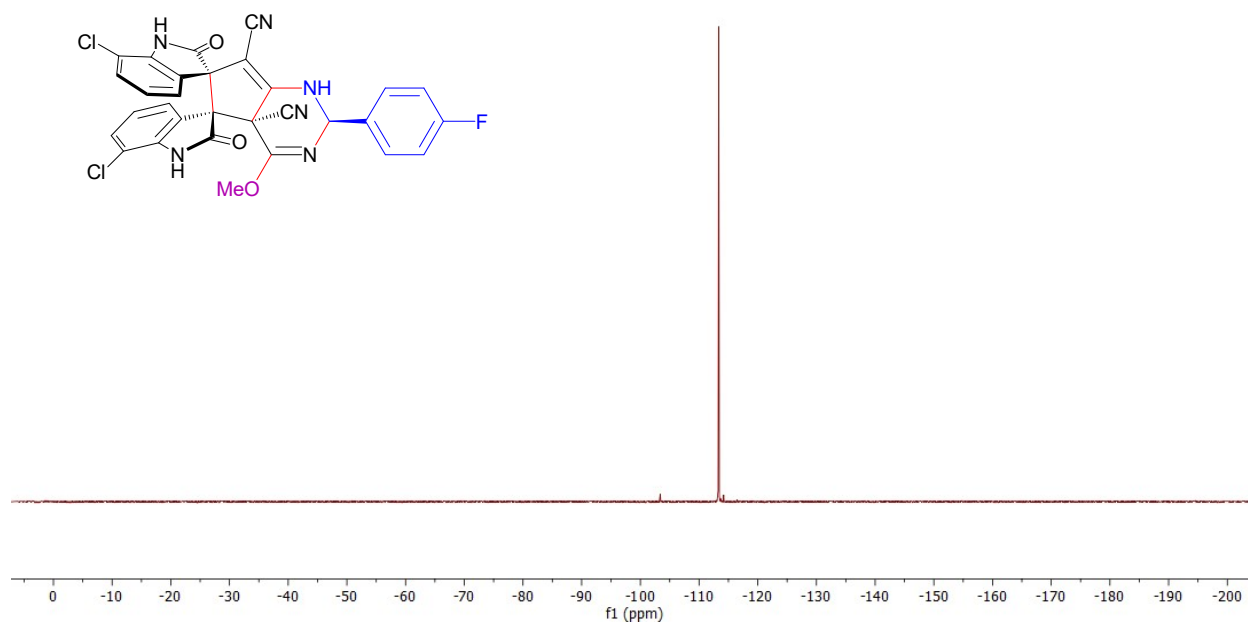


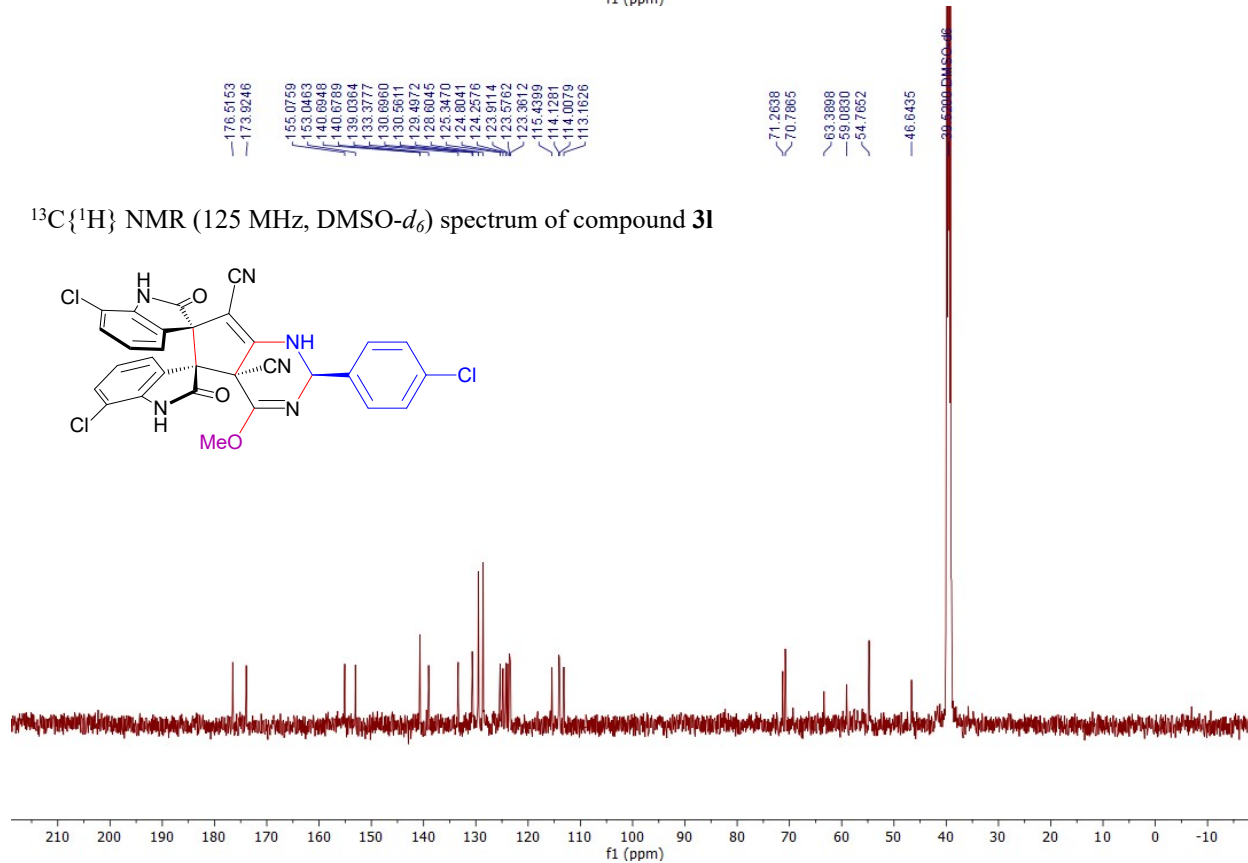
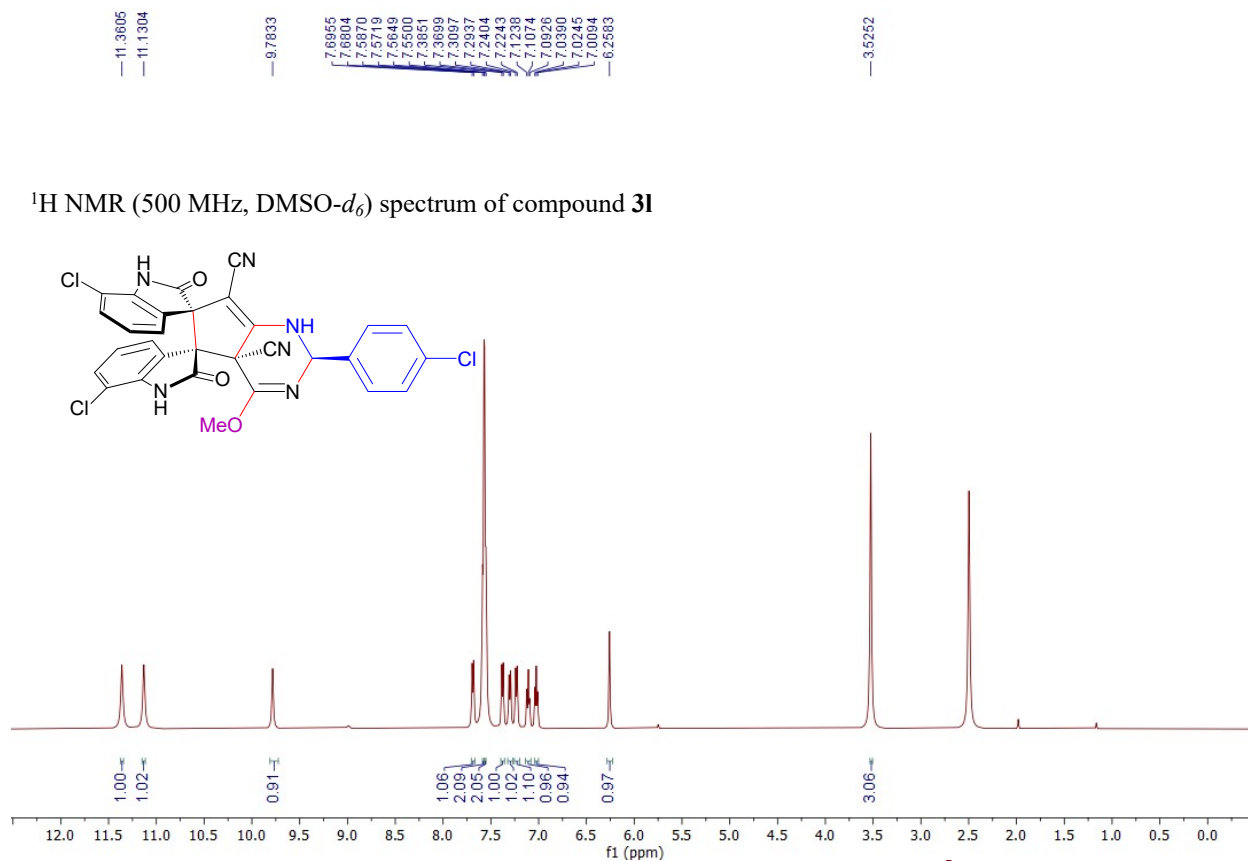
$^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) spectrum of compound **3j**

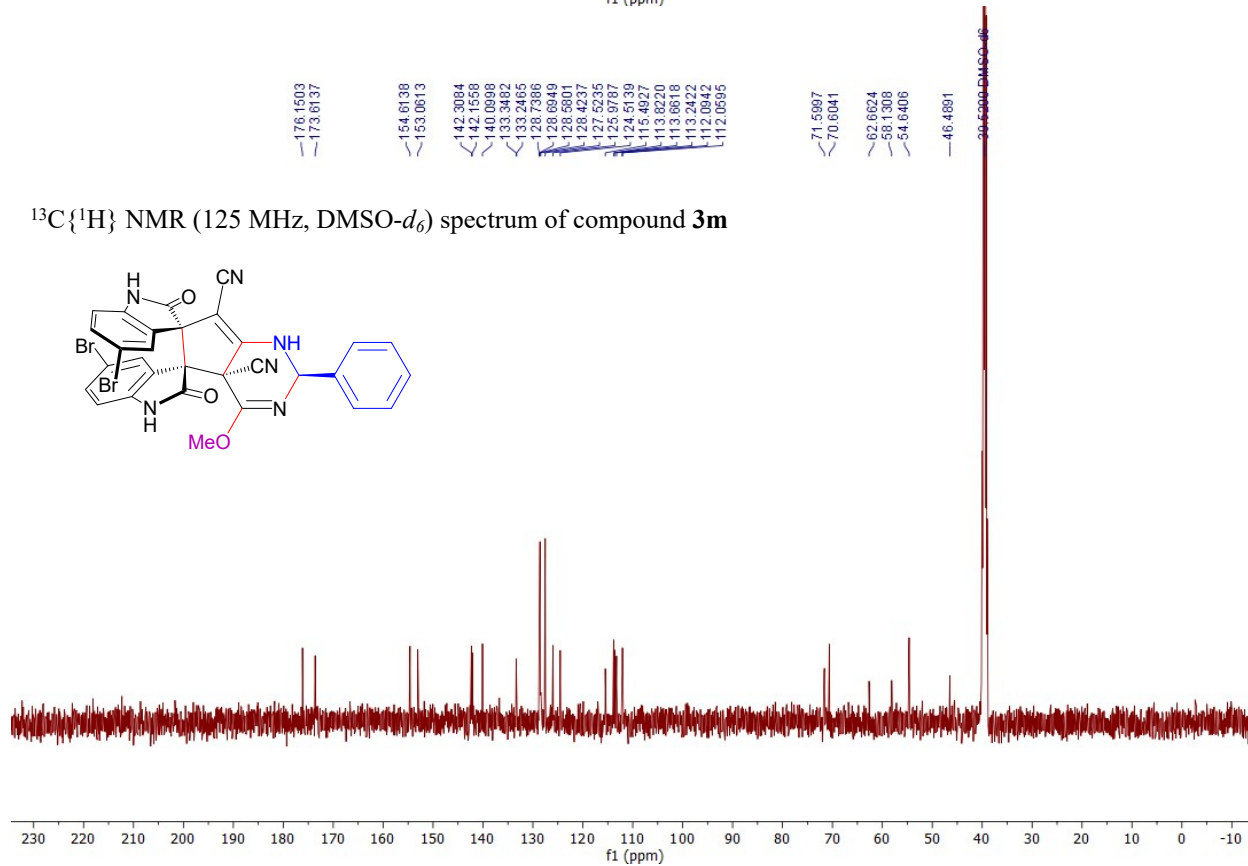
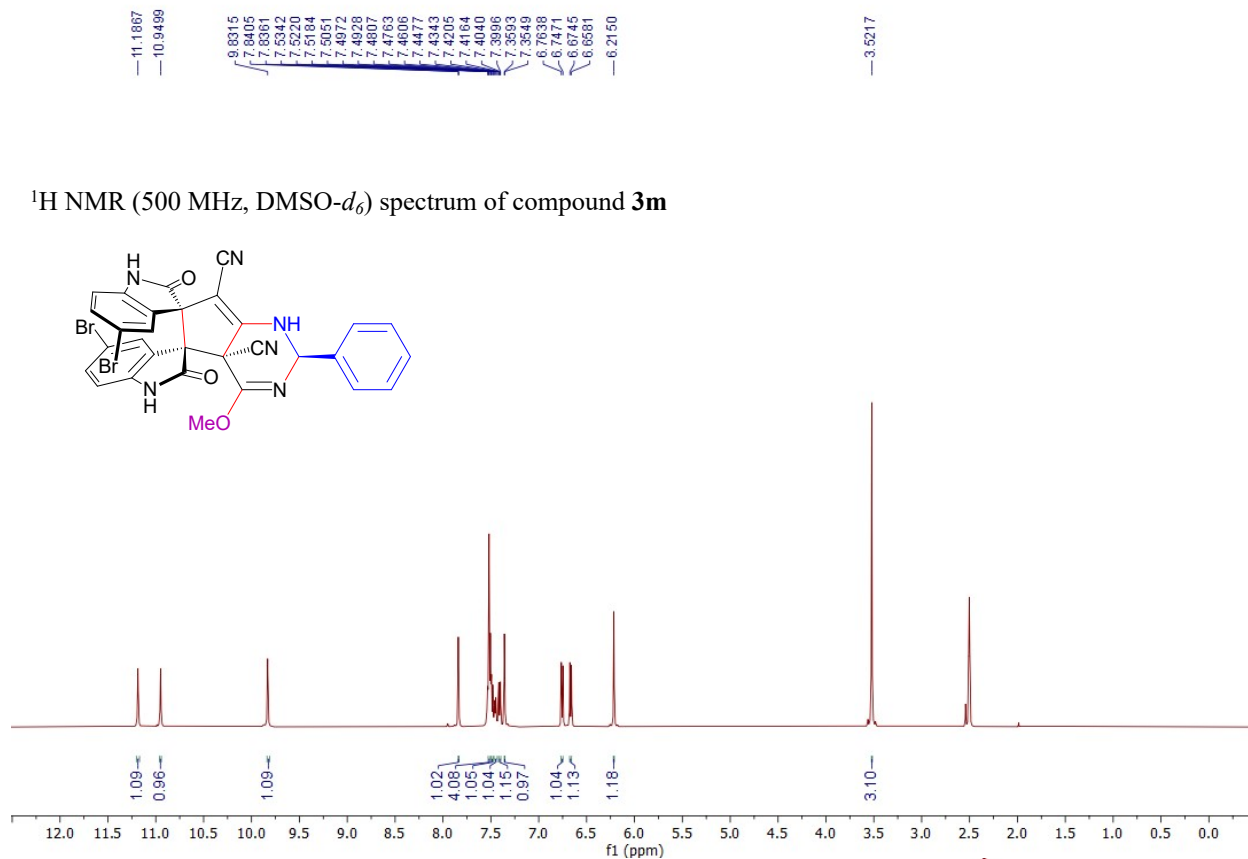


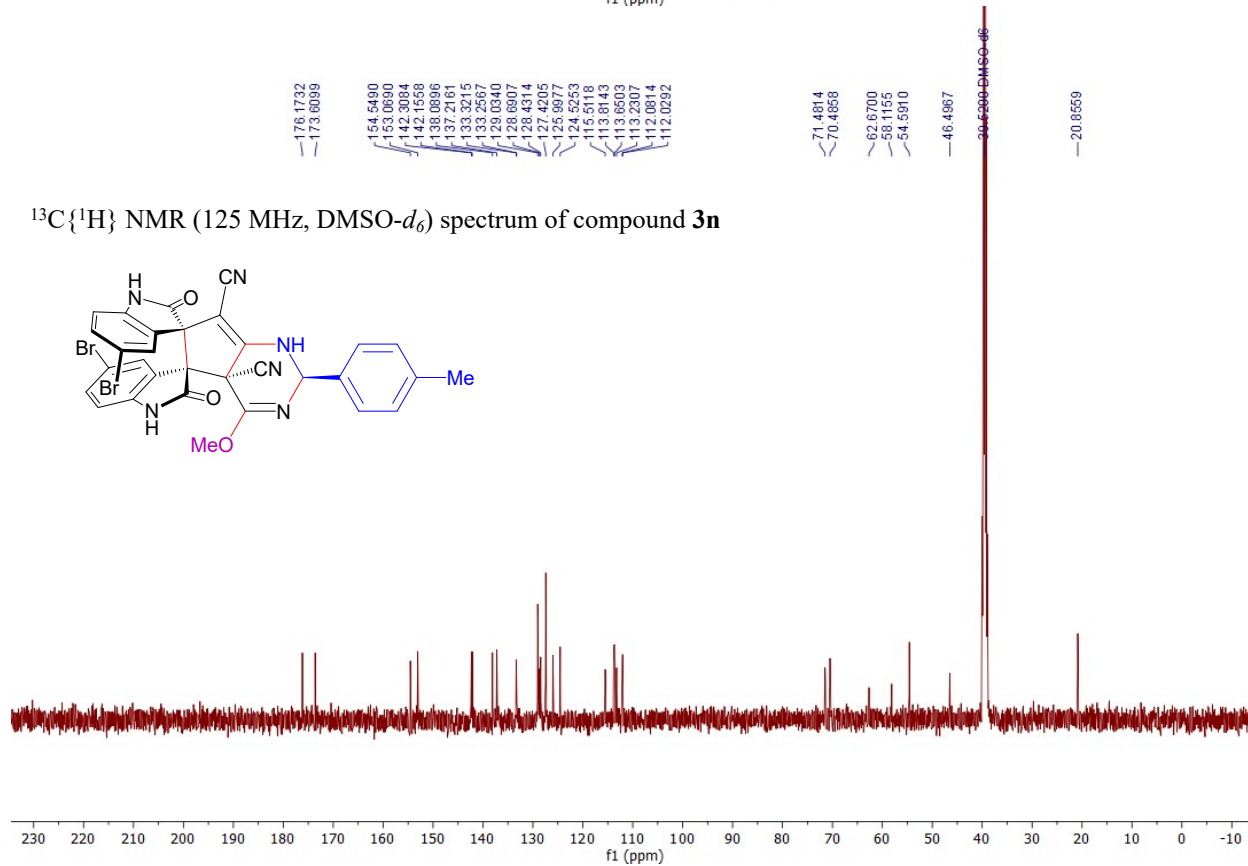
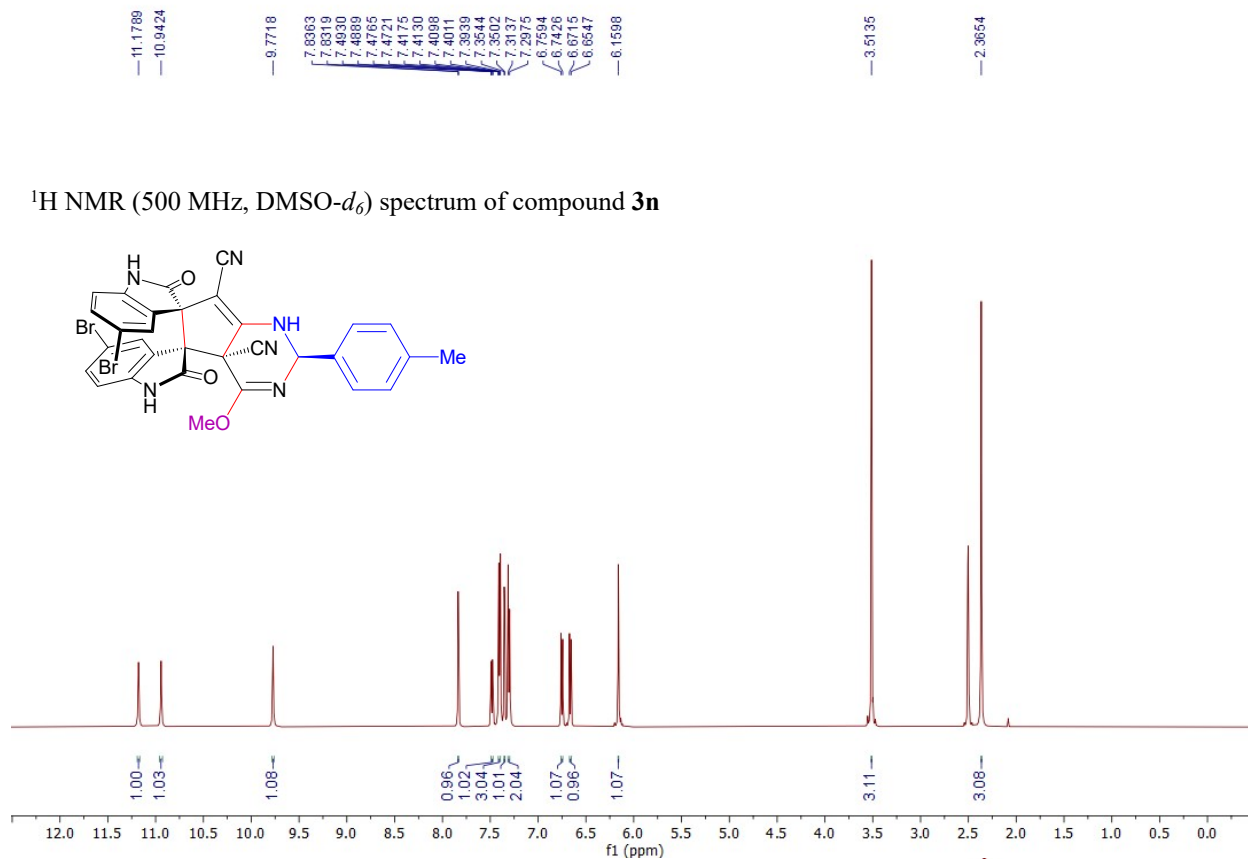


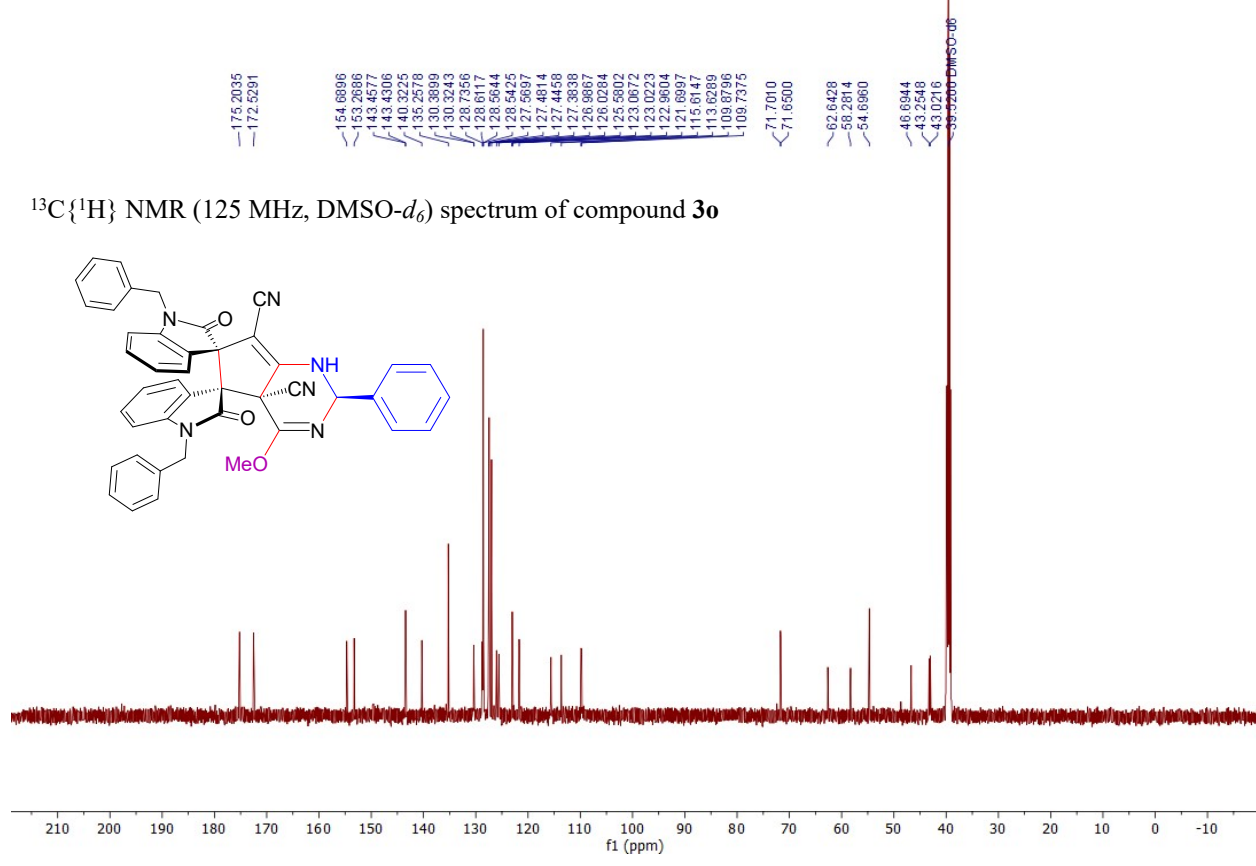
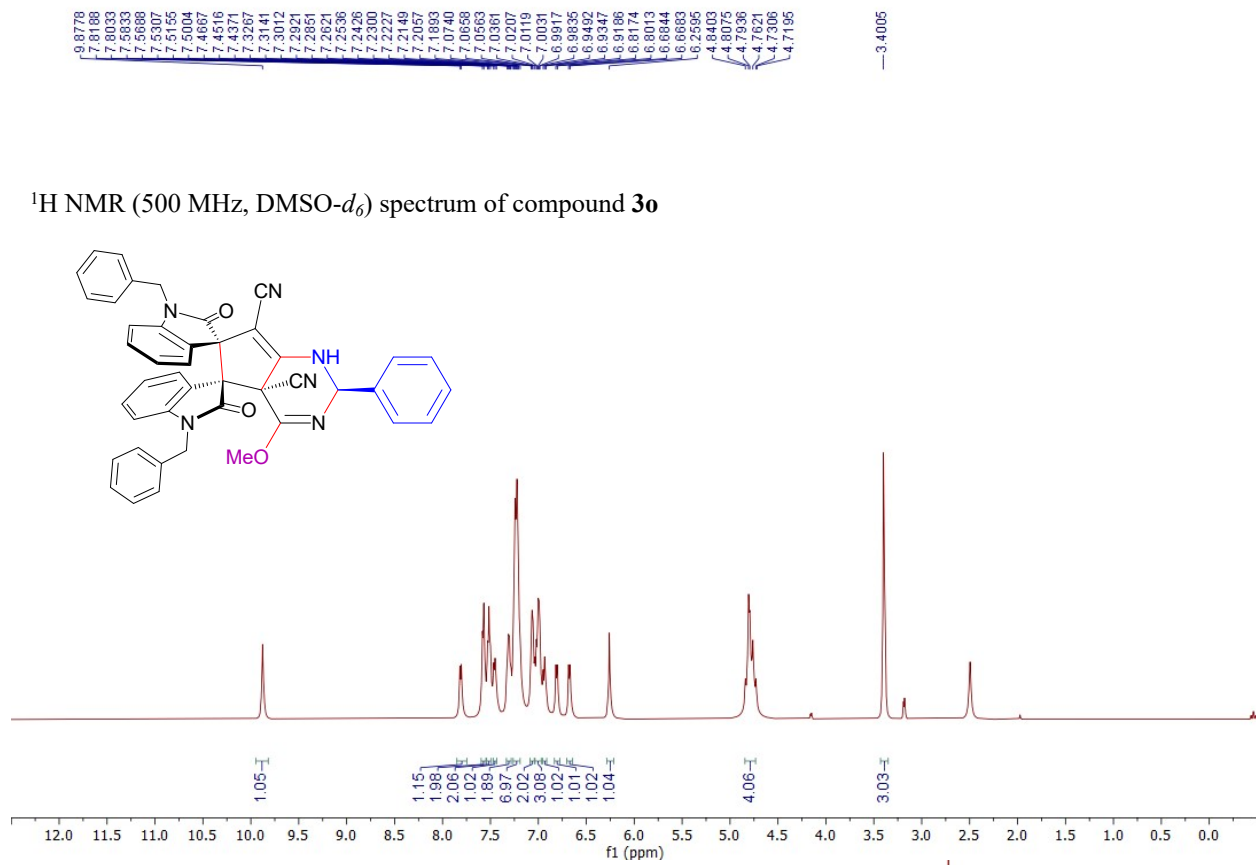
^{19}F NMR (471 MHz, $\text{DMSO-}d_6$) spectrum of compound **3k**





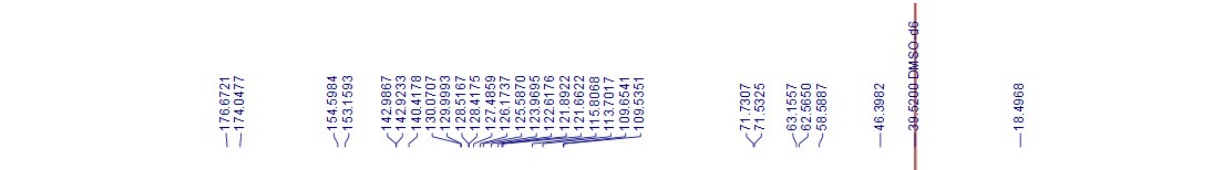
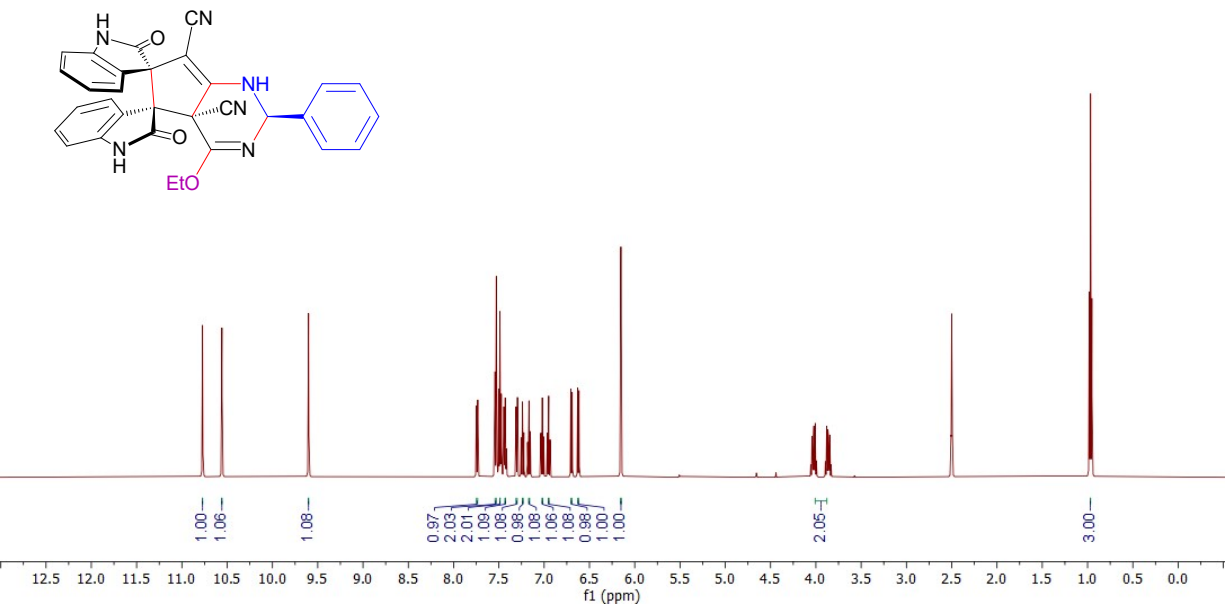




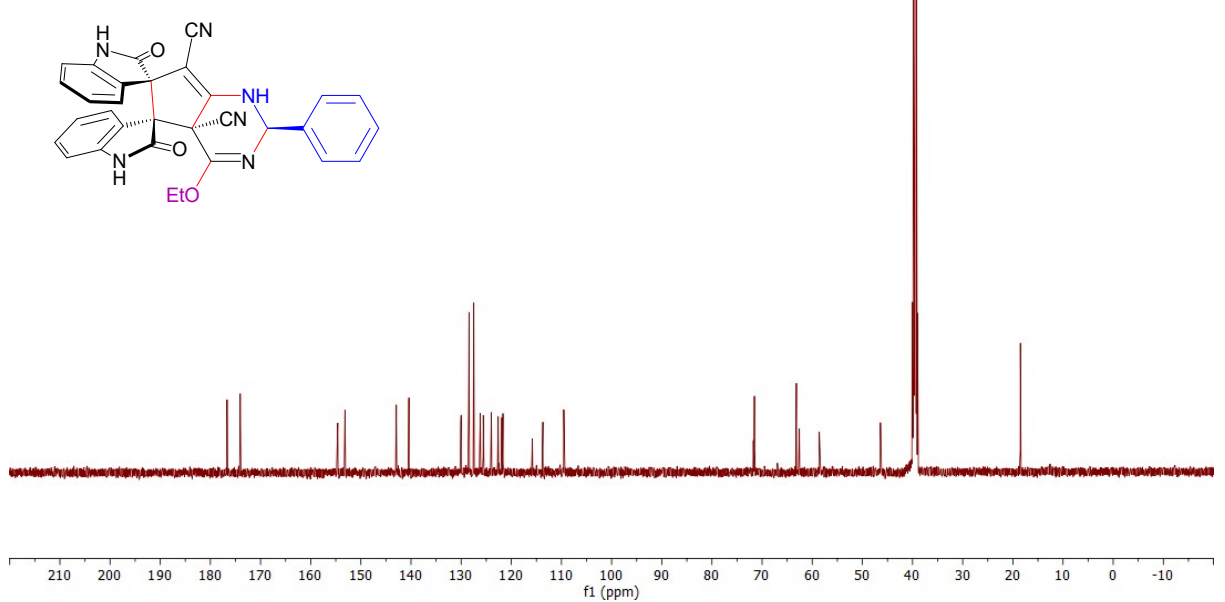


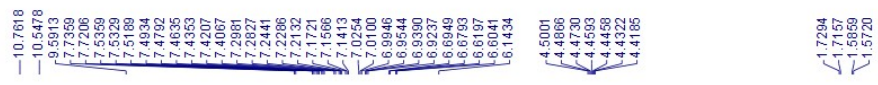


¹H NMR (500 MHz, DMSO-*d*₆) spectrum of compound **3p**

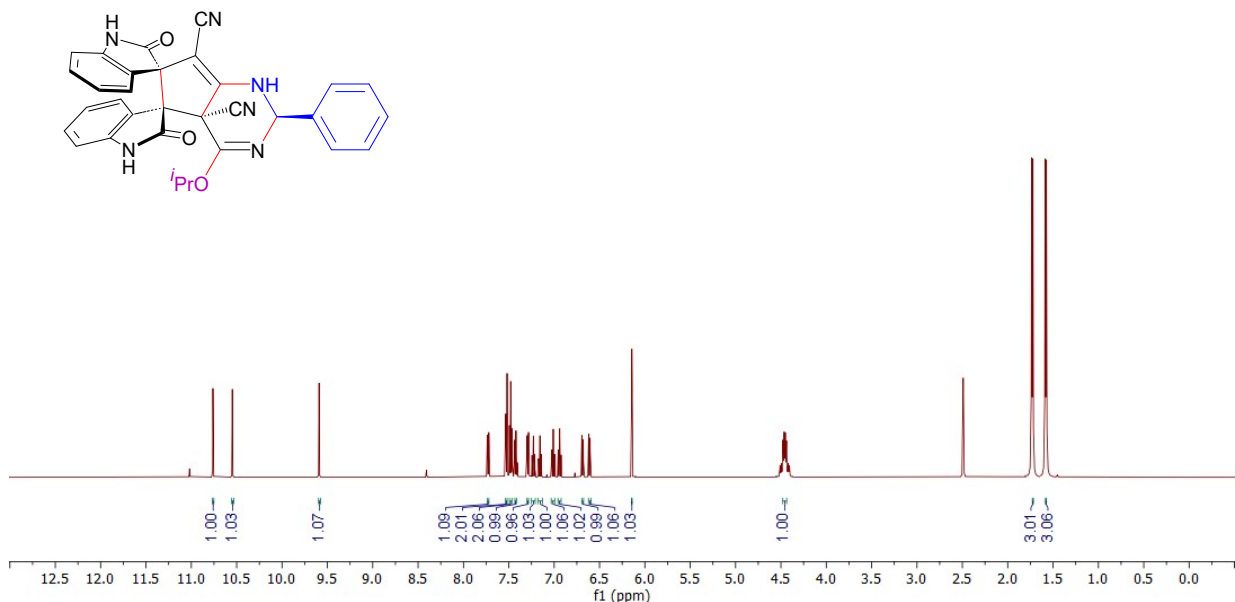


¹³C{¹H} NMR (125 MHz, DMSO-*d*₆) spectrum of compound **3p**





^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound **3q**



$^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, $\text{DMSO-}d_6$) spectrum of compound **3q**

