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Supporting Information

Rapid abnormal [3+2]-cycloaddition of isatin *N*, *N*'-cyclic azomethine imine 1,3-dipoles with chalcones

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I. General Methods

All reactions were carried out without strict water-free and oxygen-free conditions. All reagents were obtained from commercial suppliers unless otherwise stated. All solvents and reagents were directly used for reactions without further purification unless otherwise stated. Flash chromatography was performed using silica gel (200-300 mesh). Reactions were monitored by TLC or/and colour changes of reaction solution. Visualization was achieved under a UV lamp (254 nm and 365 nm), I_2 and by developing the plates with phosphomolybdic acid. ^{1}H and ^{13}C NMR were recorded on 400 and 600 MHz NMR spectrometers with tetramethylsilane (TMS) as the internal standard and were calibrated using residual undeuterated solvent as an internal reference (DMSO-d_6: ^{1}HNMR= 2.50, ^{13}C NMR= 39.52). IR spectra were acquired on an FT-IR spectrometer and are reported in wavenumbers (cm^{-1}). High-resolution mass spectra were obtained using electrospray ionization (ESI). The following abbreviations are used for the multiplicities: s: singlet, d: doublet, t: triplet, m: multiplet, br s: broad singlet for proton spectra. Coupling constants (*J*) are reported in Hertz (Hz).

II. Preparation of intermediates

Pyrazolidine-3-ones were obtained by the reaction of hydroazine monohydrate with methyl acrylate in ethanol under refluxing condition.[1] All isatin *N*, *N*'-cyclic azomethine imines **1** were prepared by the condensation of isatins and the above pyrazolidone in menthol under rt or refluxing condition.[1] All chalcones **2** were prepared by the condensation of arylaldehydes and arylmethyl ketones under basic conditions at 0°C-rt, with appropriate modification that the reaction solution should be neutralized using diluted hydrochloric acid after reaction completed.[2] In the preparation of 4-nitrochalcones, 5% aqueous NaOH solution was added dropwise to a solution of 4-nitroacetophenone (1.0 equiv.) and aromatic aldehyde (1.1 equiv.) in ethanol at 0°C. The resulting mixture stirred for 5 min, before it was neutralized by diluted hydrochloric acid, filtered, washed with iced ethanol and to obtain products.

III. General procedure for condition optimization

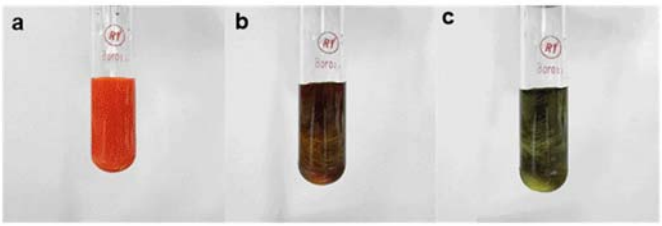
A 10 mL tube was charged with isatin *N*, *N'*-cyclic azomethine imine **1a** (0.5 mmol, 1.0 equiv.), chalcone **2a** (0.5-1.0 mmol, 1.0-2.0 equiv.) and solvent (1.5-3.0 mL). The suspended solution was vigorously stirred at rt, and then base was added. When the reaction mixture became clear and the colour of solution appeared dark green, the reaction finished. The solution was added in turn by 1 mL 1 M hydrochloric acid and 30 mL water. The resulting mixture was extracted with EtOAc (4 x 10 mL). The combined organic layers were dry with Na_2SO_4, filtered, and concentrated. The residue was purified by flash silica gel chromatography eluated with petroleum ether:EtOAc (2:1) to EtOAc to afford the corresponding product **3a**.

IV. General procedure for typical procedure for 1,3-dipolar cycloaddition

A 50 mL round-bottom flask was charged with isatin *N*, *N'*-cyclic azomethine imines **1** (1.0 mmol, 1.0 equiv.), chalcones **2** (2.0 mmol, 2.0 equiv.) and DMSO (6.0 mL). The suspended solution was vigorously stirred at rt, and then K_2CO_3 (2.0 mmol, 2.0 equiv.) was added. When the reaction mixture became clear and the colour of solution appeared dark green, the reaction finished. The solution was added in turn by 2 mL 1 M hydrochloric acid and 60 mL water. The resulting mixture was extracted with EtOAc (4 x 20 mL). The combined organic layers were dry with Na_2SO_4, filtered, and concentrated. The residue was purified by flash silica gel chromatography eluated with petroleum ether:EtOAc (2:1) to EtOAc to afford the corresponding products **3**.

V. The phenomenon of the reaction.

The phenomenon of the reaction for **1a** and **2a** under K_2CO_3 in DMSO at rt. (**a**) no K_2CO_3 (0 min, a red cloudy solution); (**b**) added K_2CO_3 (1 min, a red and green solution); (**c**) reaction finished (3 min, a dark green solution).



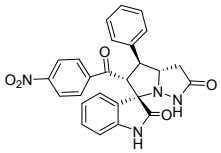
VI. Deriverziation of 3g

To a solution of **3g** (244 mg, 0.5 mmol, 1.0 equiv.) in DMSO (3.0 mL) was added DDQ (170 mg, 0.75 mol, 1.5 equiv.). The mixture was stirred at rt for 10 min, before it was quenched with saturated $NaHCO_3$ solution (20 mL). The aqueous solution was extracted with EtOAc (3 × 10 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel

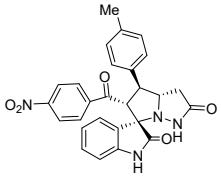
with petroleum ether:EtOAc (5:1 to 2:1) to furnish **6a**.

$NaBH_4$ (21 mg, 0.55 mmol, 1.1 equiv.) was added to a solution of **3g** (244 mg, 0.5 mmol, 1.0 equiv) in DMSO (3.0 mL) at rt. The mixture was stirred at rt for 45 min. The resulting mixture was saturated NH_4Cl solution (10 mL). The aqueous solution was extracted with EtOAc (3 × 15 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel with petroleum ether:EtOAc (2:1) to EtOAc to furnish **6b**.

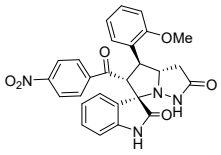
VII. Data for all new compounds:



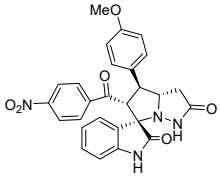
3a: 426 mg, 88% yield, a yellow solid, m.p. 108-110 °C, *dr> 20:1*; IR (thin film): ν_{max} 3421, 3430, 3197, 3066, 1711, 1687, 1530, 1342, 751, 738 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 10.26 (br s, 1 H), 9.31 (br s, 1 H), 8.13 (d, *J*= 8.4 Hz, 2 H), 7.54 (d, *J*= 8.0 Hz, 4 H), 7.47 (d, *J*= 7.6 Hz, 1 H), 7.36 (ψt, *J*= 7.2 Hz, 2 H), 7.25 (ψt, *J*= 7.4 Hz, 1 H), 7.19 (ψt, *J*= 7.6 Hz, 1 H), 7.01 (ψt, *J*= 7.4 Hz, 1 H), 6.56 (d, *J*= 8.0 Hz, 1 H), 4.85 (d, *J*= 11.2 Hz, 1 H), 4.45-4.33 (m, 2 H), 3.00 (dd, *J*= 17.2, 8.0 Hz, 1 H), 2.61 (dd, *J*= 17.0, 9.8 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.3, 176.2, 173.8, 150.3, 142.8, 141.8, 140.0, 130.4, 129.5, 129.2, 128.3, 127.9, 127.6, 123.8 (2 C), 122.1, 110.4, 76.8, 68.1, 60.7, 51.5, 37.3; HRMS (ESI): *m/z* calcd for $C_{26}H_{20}N_4O_5Na$ $[M+Na]^+$ 491.1331, found 491.1366.



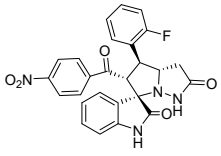
3b: 314 mg, 65% yield, a yellow solid, m.p. 236-238 °C, *dr> 20:1*; IR (thin film): ν_{max} 3430, 3167, 3064, 1706, 1686, 1527, 1342, 748, 688 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 10.29 (br s, 1 H), 9.34 (br s, 1 H), 8.21 (d, *J*= 8.4 Hz, 2 H), 7.82 (d, *J*= 8.8 Hz, 2 H), 7.51 (d, *J*= 7.6 Hz, 1 H), 7.39 (d, *J*= 8.4 Hz, 2 H), 7.18-7.13 (m, 3 H), 6.99 (ψt, *J*= 7.4 Hz, 1 H), 6.57 (d, *J*= 8.0 Hz, 1 H), 4.83 (d, *J*= 11.6 Hz, 1 H), 4.66 (dd, *J*= 11.6, 8.8 Hz, 1 H), 4.41 (dd, *J*= 17.8, 8.6 Hz, 1 H), 3.06 (dd, *J*= 17.2, 8.4 Hz, 1 H), 2.61 (dd, *J*= 17.2, 10.0 Hz, 1 H), 2.29 (s, 3 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.2, 176.3, 173.7, 150.3, 142.8, 141.8, 136.9, 136.8, 130.4, 129.8, 129.5, 128.2, 128.0, 123.8 (2 C), 122.1, 110.3, 76.8, 68.8, 60.8, 51.2, 37.3, 21.1; HRMS (ESI): *m/z* calcd for $C_{27}H_{22}N_4O_5Na$ $[M+Na]^+$ 505.1488, found 505.1497.



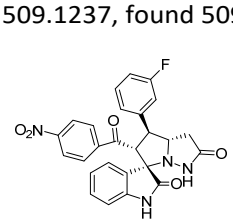
3c: 304 mg, 61% yield, a yellow solid, m.p. > 320 °C, *dr> 20:1*; IR (thin film): ν_{max} 3391, 3109, 3069, 1708, 1686, 1527, 1342, 831, 750 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 10.16 (br s, 1 H), 9.29 (br s, 1 H), 8.16 (d, *J*= 8.8 Hz, 2 H), 7.57 (d, *J*= 8.8 Hz, 2 H), 7.46 (dd, *J*= 7.6, 1.2 Hz, 1 H), 7.39 (d, *J*= 7.2 Hz, 1 H), 7.26 (td, *J*= 7.6, 1.2 Hz, 1 H), 7.20 (td, *J*= 7.6, 0.4 Hz, 1 H), 7.05-7.00 (m, 2 H), 6.92 (ψt, *J*= 7.4 Hz, 1 H), 6.55 (d, *J*= 7.6 Hz, 1 H), 5.21 (d, *J*= 12.0 Hz, 1 H), 4.52 (dd, *J*= 11.6, 8.0 Hz, 1 H), 4.30 (dd, *J*= 18.0, 9.2 Hz, 1 H), 3.91 (s, 3 H), 2.92 (dd, *J*= 17.6, 8.8 Hz, 1 H), 2.64 (dd, *J*= 17.4, 9.8 Hz, 1 H);^{13}C NMR (150 MHz, DMSO-d_6): δ 196.6, 176.3, 174.1, 158.0, 150.3, 142.9, 141.9, 130.4, 129.5, 128.9, 128.7, 127.6, 127.1, 124.0, 123.9, 122.1, 121.1, 111.8, 110.4, 76.6, 67.3, 57.6, 56.0, 47.4, 38.5; HRMS (ESI): *m/z* calcd for $C_{27}H_{22}N_4O_6Na$ $[M+Na]^+$ 521.1437, found 521.1459.



3d: 393 mg, 78% yield, a yellow solid, m.p. 245-246 °C, *dr> 20:1*; IR (thin film): ν_{max} 3309, 3064, 3011, 1713, 1693, 1524, 1341, 793, 709 cm^{-1};^1H NMR (400 MHz, DMSO-d_6) δ 10.15 (br s, 1 H), 9.29 (br s, 1 H), 8.15 (d, *J*= 7.6 Hz, 2 H), 7.55 (d, *J*= 7.6 Hz, 2 H), 7.46-7.45 (m, 3 H), 7.20 (ψt, *J*= 7.6 Hz, 1 H), 7.01 (ψt, *J*= 7.4 Hz, 1 H), 6.92 (d, *J*= 7.6 Hz, 2 H), 6.54 (d, *J*= 7.2 Hz, 1 H), 4.79 (d, *J*= 10.4 Hz, 1 H), 4.39-4.29 (m, 2 H), 3.73 (s, 3 H), 2.96 (dd, *J*= 17.2, 7.6 Hz, 1 H), 2.60 (dd, *J*= 17.2, 8.8 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.3, 176.3, 173.7, 158.9, 150.3, 142.8, 141.8, 131.7, 130.4, 129.4, 128.0, 123.9, 123.8, 122.1, 114.7, 110.3, 76.8, 68.8, 60.9, 55.5, 50.9, 37.2; HRMS (ESI): *m/z* calcd for $C_{27}H_{22}N_4O_6Na$ $[M+Na]^+$ 521.1437, found 521.1436.

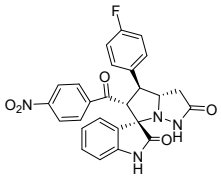


3e: 443 mg, 91% yield, a yellow solid, m.p. > 320 °C, *dr> 20:1*; IR (thin film): ν_{max} 3424, 3195, 3065, 1714, 1686, 1528, 1342, 749, 690 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6) δ 10.22 (br s, 1 H), 9.33 (br s, 1 H), 8.14 (d, *J*= 9.0 Hz, 2 H), 7.68 (td, *J*= 7.8, 1.2 Hz, 1 H), 7.51 (d, *J*= 9.0 Hz, 2 H), 7.41 (d, *J*= 7.8 Hz, 1 H), 7.35-7.32 (m, 1 H), 7.25-7.18 (m, 3 H), 7.02 (td, *J*= 7.8, 0.6 Hz, 1 H), 6.54 (d, *J*= 7.8 Hz, 1 H), 5.01 (d, *J*= 11.4 Hz, 1 H), 4.55 (dd, *J*= 11.4, 8.4 Hz, 1 H), 4.39 (dd, *J*= 18.0, 9.0 Hz, 1 H), 2.90 (dd, *J*= 17.4, 8.4 Hz, 1 H), 2.66 (dd, *J*= 17.4, 9.6 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.3, 176.1, 173.7, 150.2, 142.9, 141.8, 130.5 (d), 129.7 (d), 129.6, 127.7, 126.4, 126.3, 125.3 (d), 123.8, 123.7, 122.2, 116.3, 116.2, 110.4, 76.5, 67.4, 59.2, 55.4, 37.6; ^{19}F NMR (376 MHz, DMSO-d_6): -116.1. HRMS (ESI): *m/z* calcd for $C_{26}H_{19}FN_4O_5Na$ $[M+Na]^+$ 509.1237, found 509.1320.

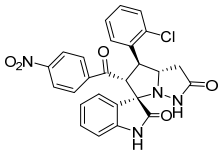


3f: 418 mg, 86% yield, a yellow solid, m.p. 236-237 °C, *dr> 20:1*; IR (thin film): ν_{max} 3436, 3176, 3053, 1703, 1691, 1610, 1529, 1340, 754, 688 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 10.24 (br s, 1 H), 9.31 (br s, 1 H), 8.13 (d, *J*= 8.8 Hz, 2 H), 7.56 (d, *J*= 8.8 Hz, 2 H), 7.47-7.39 (m, 4 H), 7.19 (td, *J*= 7.6, 0.8 Hz, 1 H), 7.11-7.06 (m, 1 H), 7.24 (ψt, *J*= 7.4 Hz, 1 H), 7.00 (td, *J*= 7.6, 0.8 Hz, 1 H), 6.55 (d, *J*= 7.6 Hz, 1 H), 4.89 (d, *J*= 11.2 Hz, 1 H), 4.47 (dd, *J*= 11.2, 8.4 Hz, 1 H), 4.35 (dd, *J*= 18.0, 8.4 Hz, 1 H), 3.01 (dd, *J*= 17.2, 8.4 Hz, 1 H), 2.61 (dd, *J*= 17.2, 10.0 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.5, 176.2, 173.8, 150.3, 142.9, 141.8, 131.1 (d), 130.5, 129.7, 127.9, 124.6 (d), 123.8, 123.7, 122.1, 115.6, 115.3, 114.6,

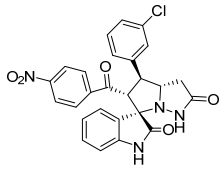
114.4, 110.5, 76.8, 68.5, 60.5, 51.1, 37.0; ^{19}F NMR (376 MHz, DMSO-d_6): -112.8. HRMS (ESI): *m/z* calcd for $C_{26}H_{19}FN_4O_5Na$ [M+Na]$^+$509.1237, found 509.1230.



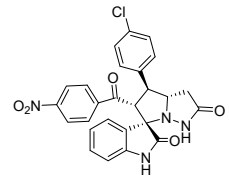
3g: 462 mg, 95% yield, a yellow solid, m.p. 230-232 °C, *dr> 20:1*; IR (thin film): ν_{max} 3399, 3162, 3060, 2835, 1705, 1686, 1526, 1344, 751, 689 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.21 (br s, 1 H), 9.31 (br s, 1 H), 8.13 (d, *J*= 8.8 Hz, 2 H), 7.62-7.61 (m, 2 H), 7.54 (d, *J*= 8.8 Hz, 2 H), 7.47 (d, *J*= 7.6 Hz, 1 H), 7.21-7.17 (m, 3 H), 7.01 (ψt, *J*= 7.4 Hz, 1 H), 6.55 (d, *J*= 7.6 Hz, 1 H), 4.83 (d, *J*= 11.6 Hz, 1 H), 4.44 (dd, *J*= 11.2, 8.4 Hz, 1 H), 4.34 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.99 (dd, *J*= 17.2, 8.4 Hz, 1 H), 2.61 (dd, *J*= 17.2, 10.0 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.4, 176.2, 173.8, 150.3, 142.9, 141.8, 136.1, 130.5, 129.7, 127.9, 123.9 (d), 122.1, 116.1, 115.9, 110.5, 76.8, 68.7, 60.8, 50.7, 37.1; ^{19}F NMR (376 MHz, DMSO-d_6): -115.6. HRMS (ESI): *m/z* calcd for $C_{26}H_{19}FN_4O_5Na$ [M+Na]$^+$ 509.1237, found 509.1228.



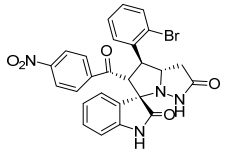
3h: 320 mg, 64% yield, a yellow solid, m.p. 266-267 °C, *dr> 20:1*; IR (thin film): ν_{max} 3431, 3176, 3053, 1706, 1693, 1612, 1527, 1338,750, 686 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.22 (br s, 1 H), 9.38 (br s, 1 H), 8.14 (d, *J*= 8.8 Hz, 2 H), 7.85 (dd, *J*= 7.6, 0.8 Hz, 1 H), 7.55 (d, *J*= 8.8 Hz, 2 H), 7.50 (dd, *J*= 8.0, 1.2 Hz, 1 H), 7.39 (td, *J*= 7.6, 0.8 Hz, 1 H), 7.36 (d, *J*= 7.6 Hz, 1 H), 7.30 (td, *J*= 7.6, 1.2 Hz, 1 H), 7.21 (ψt, *J*= 7.4 Hz, 1 H), 7.04 (ψt, *J*= 7.2 Hz, 1 H), 6.55 (d, *J*= 7.6 Hz, 1 H), 5.15 (d, *J*= 11.2 Hz, 1 H), 4.84 (dd, *J*= 11.2, 8.4 Hz, 1 H), 4.35 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.86 (dd, *J*= 17.2, 8.4 Hz, 1 H), 2.69 (dd, *J*= 17.2, 9.6 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.3, 176.2, 173.6, 150.3, 143.0, 141.8, 137.0, 134.1, 130.7, 130.3, 129.9, 129.6, 129.5, 128.5, 127.5, 123.9, 123.7, 122.4, 110.6, 76.6, 68.2, 60.1, 48.1, 37.9; HRMS (ESI): *m/z* calcd for $C_{26}H_{19}ClN_4O_5Na$ [M+Na]$^+$ 525.0492, found 525.0513.



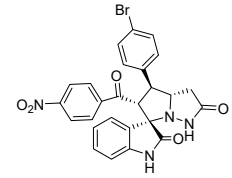
3i: 368 mg, 73% yield, a yellow solid, m.p. 255-256 °C, *dr> 20:1*; IR (thin film): ν_{max} 3363, 3174, 3065, 2829, 1705, 1683, 1531, 1473, 1342, 748, 686 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.17 (br s, 1 H), 9.31 (br s, 1 H), 8.13 (d, *J*= 8.8 Hz, 2 H), 7.67 (s, 1 H), 7.57-7.54 (m, 3 H), 7.47 (d, *J*= 7.6 Hz, 1 H), 7.40 (ψt, *J*= 8.0 Hz, 1 H), 7.32 (dd, *J*= 8.0, 0.8 Hz, 1 H), 7.19 (ψt, *J*= 7.4 Hz, 1 H), 7.01 (ψt, *J*= 7.4 Hz, 1 H), 6.53 (d, *J*= 8.0 Hz, 1 H), 4.88 (d, *J*= 11.2 Hz, 1 H), 4.46 (dd, *J*= 11.2, 8.4 Hz, 1 H), 4.36 (dd, *J*= 18.0, 8.4 Hz, 1 H), 3.00 (dd, *J*= 17.6, 8.4 Hz, 1 H), 2.60 (dd, *J*= 17.2, 10.0 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.5, 176.2, 173.8, 150.3, 142.9, 142.6, 141.9, 133.8, 131.1, 130.5, 129.7, 128.8, 127.9, 127.7, 127.1, 123.8, 123.7, 122.1, 110.5, 76.8, 68.6, 60.9, 51.0, 37.0; HRMS (ESI): *m/z* calcd for $C_{26}H_{19}ClN_4O_5Na$ [M+Na]$^+$ 525.0492, found 525.0510.



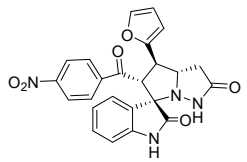
3j: 446 mg, 89% yield, a yellow solid, m.p. 244-246 °C, *dr*> 20:1; ν_{max} 3404, 3168, 3066, 2830, 1705, 1686, 1527, 1343, 749, 687 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6) δ 10.16 (br s, 1 H), 9.30 (br s, 1 H), 8.13 (d, *J*= 9.0 Hz, 2 H), 7.59 (d, *J*= 9.0 Hz, 2 H), 7.53 (d, *J*= 8.4 Hz, 2 H), 7.47 (d, *J*= 7.8 Hz, 1 H), 7.42 (d, *J*= 8.4 Hz, 2 H), 7.20 (td, *J*= 7.8, 1.2 Hz, 1 H), 7.01 (ψt, *J*= 7.5 Hz, 1 H), 6.53 (d, *J*= 7.8 Hz, 1 H), 4.83 (dd, *J*= 11.4 Hz, 1 H), 4.45 (dd, *J*= 11.4, 8.4 Hz, 1 H), 4.33 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.99 (dd, *J*= 17.4, 8.4 Hz, 1 H), 2.60 (dd, *J*= 17.4, 9.9 Hz, 1 H);^{13}C NMR (100 MHz, DMSO-d_6): δ 196.4,176.2, 173.8, 150.3, 142.9, 141.8, 139.0, 132.4, 130.5 (2 C), 129.7, 129.2, 127.9, 123.8 (2 C), 122.2, 110.5, 76.8, 68.7, 60.8, 50.8, 37.1; HRMS (ESI): *m/z* calcd for $C_{26}H_{20}ClN_4O_5$ [M+H]$^+$ 503.1122, found 503.1141.



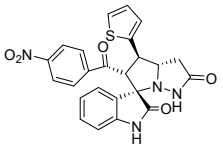
3k: 466 mg, 85% yield, a yellow solid, m.p. 231-232 °C, *dr> 20:1*; IR (thin film): ν_{max} 3174, 3072, 1714, 1703, 1683, 1525, 1340, 755, 739/m.p : cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.33 (br s, 1 H), 9.38 (br s, 1 H), 8.13 (d, *J*= 8.8 Hz, 2 H), 7.85 (dd, *J*= 7.6, 1.2 Hz, 1 H), 7.66 (dd, *J*= 8.0, 0.8 Hz, 1 H), 7.55 (d, *J*= 8.8 Hz, 2 H), 7.44 (ψt, *J*= 7.6, 0.8 Hz, 1 H), 7.30 (d, *J*= 7.6 Hz, 1 H), 7.24-7.18 (m, 2 H), 7.03 (td, *J*= 7.6, 0.8 Hz, 1 H), 6.57 (d, *J*= 7.6 Hz, 1 H), 5.11 (d, *J*= 11.2 Hz, 1 H), 4.86 (dd, *J*= 11.2, 8.4 Hz, 1 H), 4.31 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.84 (dd, *J*= 17.2, 8.0 Hz, 1 H), 2.70 (dd, *J*= 17.2, 9.6 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.3, 176.1, 173.4, 150.3, 143.1, 141.7, 138.6, 133.6, 130.7, 129.8, 129.6, 129.2, 127.4, 125.1, 123.9, 123.6, 122.3, 110.7, 100.0, 76.5, 68.6, 60.6, 50.2, 37.4; HRMS (ESI): *m/z* calcd for $C_{26}H_{19}BrN_4O_5Na$ [M+Na]$^+$ 569.0437, found 569.0517.



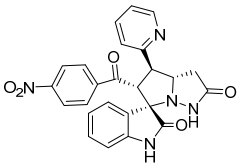
3l: 508 mg, 93% yield, a yellow solid, m.p. 230-231 °C, *dr*> 20:1; IR (thin film): ν_{max} 3406, 3177, 3077, 1706, 1686, 1530, 1343, 750 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6) δ 10.19 (br s, 1 H), 9.30 (br s, 1 H), 8.13 (d, *J*= 9.0 Hz, 2 H), 7.57-7.52 (m, 6 H), 7.46 (d, *J*= 7.8 Hz, 1 H), 7.19 (td, *J*= 7.8, 0.6 Hz, 1 H), 7.00 (td, *J*= 7.8, 0.6 Hz, 1 H), 6.54 (d, *J*= 7.8 Hz, 1 H), 4.83 (dd, *J*= 12.0 Hz, 1 H), 4.43 (dd, *J*= 11.4, 8.4 Hz, 1 H), 4.33 (dd, *J*= 18.6, 8.4 Hz, 1 H), 2.99 (dd, *J*= 17.4, 8.4 Hz, 1 H), 2.60 (dd, *J*= 17.1, 9.9 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.4, 176.2, 173.8, 150.3, 142.9, 141.8, 139.5, 132.8, 130.8, 129.7, 127.9, 123.8, 123.7, 122.1, 120.9, 110.5, 76.8, 68.6, 60.7, 50.8, 37.1; HRMS (ESI): *m/z* calcd for $C_{26}H_{19}BrN_4O_5Na$ [M+Na]$^+$ 569.0437, found 569.0528.



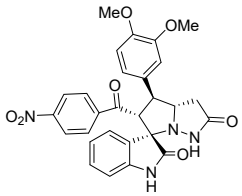
3m: 376 mg, 82% yield, a yellow solid, m.p. 211-212 °C, *dr*> 20:1; IR (thin film): ν_{max} 3379, 3164, 3067, 2835, 1709, 1691, 1527, 1344, 729, 690 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.17 (br s, 1 H), 9.30 (br s, 1 H), 8.16 (d, *J*= 9.2 Hz, 2 H), 7.61 (dd, *J*= 2.5, 0.8 Hz, 1 H), 7.55 (dt, *J*= 9.2, 2.0 Hz, 1 H), 7.40 (d, *J*= 7.2 Hz, 1 H), 7.18 (td, *J*= 7.6, 1.2 Hz, 1 H), 6.98 (td, *J*= 7.6, 0.8 Hz, 1 H), 6.53 (d, *J*= 7.2 Hz, 1 H), 6.42 (d, *J*= 3.6 Hz, 1 H), 6.40 (dd, *J*= 3.2, 2.0 Hz, 1 H), 4.79 (d, *J*= 11.2 Hz, 1 H), 4.55 (dd, *J*= 11.2, 8.0 Hz, 1 H), 4.45 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.98 (dd, *J*= 17.4, 8.6 Hz, 1 H), 2.68 (dd, *J*= 17.2, 10.0 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 195.8, 176.0, 173.8, 152.9, 150.3, 143.1, 142.8, 141.6, 130.5, 129.6, 127.9, 123.9, 123.5, 122.1, 111.0, 110.4, 107.1, 76.5, 65.8, 58.5, 44.9, 37.3; HRMS (ESI): *m/z* calcd for $C_{24}H_{18}N_4O_6Na$ [M+Na]$^+$ 481.1124, found 481.1125.



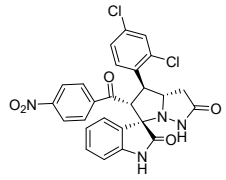
3n: 386 mg, 81% yield, a yellow solid, m.p. 214-215 °C, *dr> 20:1*; IR (thin film): ν_{max} 3189, 3112, 3072, 3061, 3000, 2952, 2814, 1714, 1685, 1529, 1344, 749 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.29 (br s, 1 H), 9.32 (br s, 1 H), 8.17 (d, *J*= 8.4 Hz, 2 H), 7.57 (d, *J*= 8.4 Hz, 2 H), 7.45-7.42 (m, 2 H), 7.21-7.18 (m, 2 H), 7.02-6.98 (m, 2 H), 6.57 (d, *J*= 7.6 Hz, 1 H), 4.74-4.73 (m, 2 H), 4.48-4.43 (m, 1 H), 3.01 (dd, *J*= 17.2, 8.4 Hz, 1 H), 2.69 (dd, *J*= 17.2, 10.0 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 195.9, 176.0, 173.6, 150.4, 142.8, 142.7, 141.6, 130.6, 129.7, 128.1, 127.8, 125.9, 125.5, 124.1, 123.5, 122.2, 110.5, 76.9, 68.6, 61.6, 48.8, 37.0; HRMS (ESI): *m/z* calcd for $C_{24}H_{18}N_4O_5SNa$ [M+Na]$^+$ 497.0896, found 497.0920.



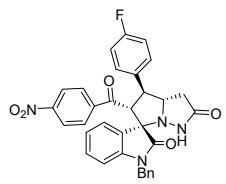
3o: 248 mg, 53% yield, a brown solid, m.p. 205-206 °C, *dr*> 20:1; IR (thin film): ν_{max} 3411, 3173, 3068, 2825, 1712, 1687, 1525, 1342, 748, 690 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.15 (br s, 1 H), 9.29 (br s, 1 H), 8.54 (d, *J*= 8.4 Hz, 2 H), 8.18 (d, *J*= 8.8 Hz, 2 H), 7.79 (td, *J*= 7.6, 1.6 Hz, 1 H), 7.53-7.47 (m, 4 H), 7.29-7.27 (m, 1 H), 7.20 (ψt, *J*= 7.8 Hz, 1 H), 7.01 (ψt, *J*= 7.6 Hz, 1 H), 6.54 (d, *J*= 7.6 Hz, 1 H), 5.15 (d, *J*= 11.2 Hz, 1 H), 4.65 (dd, *J*= 11.0, 8.2 Hz, 1 H), 4.41 (dd, *J*= 17.8, 9.0 Hz, 1 H), 3.09 (dd, *J*= 17.2, 8.8 Hz, 1 H), 2.76 (dd, *J*= 17.2, 10.0 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.6, 176.3, 173.8, 158.6, 150.2, 149.9, 142.7, 142.0, 137.4, 130.4, 129.4, 127.8, 124.4, 124.0 (2 C), 123.0, 122.0, 110.3, 76.5, 67.1, 59.1, 52.8, 37.3; HRMS (ESI): *m/z* calcd for $C_{25}H_{19}N_5O_5Na$ [M+Na]$^+$ 492.1284, found 492.1297.



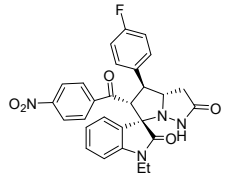
3p: 428 mg, 81% yield, a yellow solid, m.p. 278-279 °C, *dr*> 20:1; IR (thin film): ν_{max} 3392, 3165, 3063, 3011, 2837, 1705, 1692, 1522, 1341, 753 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.14 (br s, 1 H), 9.28 (br s, 1 H), 8.14 (dt, *J*= 8.8, 2.0 Hz, 2 H), 7.58 (dt, *J*= 8.8, 2.0 Hz, 2 H), 7.46 (d, *J*= 7.2 Hz, 1 H), 7.19 (td, *J*= 7.6, 1.2 Hz, 1 H), 7.10 (d, *J*= 2.0 Hz, 1 H), 7.05 (dd, *J*= 8.4, 2.0 Hz, 1 H), 7.01 (td, *J*= 7.6, 1.2 Hz, 1 H), 6.91 (d, *J*= 8.4 Hz, 1 H), 6.54 (d, *J*= 7.6 Hz, 1 H), 4.84 (d, *J*= 12.0 Hz, 1 H), 4.37 (m, 2 H), 3.81 (s, 3 H), 3.71 (s, 3 H), 2.97 (dd, *J*= 17.2, 7.2 Hz, 1 H), 2.60 (dd, *J*= 17.2, 9.6 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.3, 176.3, 173.7, 150.3, 149.3, 148.4, 142.7, 141.8, 132.1, 130.4, 129.6, 128.0, 123.9, 123.8, 122.0, 120.3, 112.5, 112.0, 110.3, 76.9, 68.6, 60.6, 56.0, 55.9, 51.5, 37.2; HRMS (ESI): *m/z* calcd for $C_{28}H_{24}N_4O_7Na$ [M+Na]$^+$ 551.1543, found 551.1573.



3q: 386 mg, 72% yield, a yellow solid, m.p. 278-279 °C, *dr*> 20:1; IR (thin film): ν_{max} 3321, 3084, 1702, 1693, 1539, 1348, 769, 693 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.19 (br s, 1 H), 9.36 (br s, 1 H), 8.12 (dt, *J*= 8.8, 2.2 Hz, 2 H), 7.89 (d, *J*= 8.8 Hz, 1 H), 7.66 (d, *J*= 2.0 Hz, 1 H), 7.51 (dt, *J*= 8.8, 2.2 Hz, 2 H), 7.47 (dd, *J*= 8.4, 2.4 Hz, 1 H), 7.33 (d, *J*= 7.6 Hz, 1 H), 7.19 (td, *J*= 7.8, 1.0 Hz, 1 H), 7.02(td, *J*= 7.6, 0.8 Hz, 1 H), 6.52 (d, *J*= 7.2 Hz, 1 H), 5.10 (d, *J*= 11.2 Hz, 1 H), 4.79 (dd, *J*= 11.2, 8.4 Hz, 1 H), 4.33 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.84 (dd, *J*= 17.2, 8.4 Hz, 1 H), 2.67 (dd, *J*= 17.2, 10.0 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.3, 176.0, 173.5, 150.2, 143.0, 141.8, 136.2, 135.0, 133.1, 131.4, 130.6, 129.6 (2 C), 128.5, 127.4, 123.7, 123.5, 122.3, 110.5, 76.4, 68.0, 60.2, 47.6, 37.5; HRMS (ESI): *m/z* calcd for $C_{26}H_{18}Cl_2N_4O_5Na$ [M+Na]$^+$ 559.0552, found 559.0588.

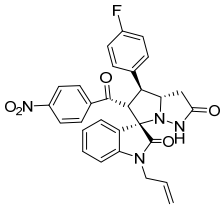


3r: 380 mg, 66% yield, a yellow solid, m.p. 196-200 °C, *dr*> 20:1; IR (thin film): ν_{max} 3176, 3063, 3046, 2928, 1718, 1689, 1525, 1510, 1343, 742, 699 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.43 (br s, 1 H), 8.09 (d, *J*= 8.8 Hz, 2 H), 7.64-7.58 (m, 5 H), 7.23-7.06 (m, 7 H), 6.93 (d, *J*= 8.0 Hz, 2 H), 6.62 (d, *J*= 7.6 Hz, 1 H), 5.01 (d, *J*= 11.6 Hz, 1 H), 4.56-4.51 (m, 2 H), 4.44-4.32 (m, 2 H), 3.03 (dd, *J*= 17.2, 8.0 Hz, 1 H), 2.65 (dd, *J*= 17.4, 9.8 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 195.5, 174.9, 174.1, 150.5, 143.3, 141.1, 135.8, 135.7 (d), 130.5, 130.4 (d), 129.7, 128.7, 128.1, 127.6, 127.5, 124.0, 123.3, 122.9, 116.1, 115.9, 109.9, 76.5, 68.7, 60.1, 51.1, 43.6, 37.0; ^{19}F NMR (376 MHz, DMSO-d_6): -115.4. HRMS (ESI): *m/z* calcd for $C_{33}H_{25}N_4O_5Na$ [M+Na]$^+$ 599.1707, found 599.1707.

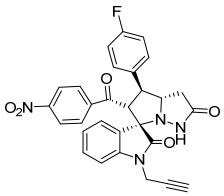


3s: 375 mg, 73% yield, a yellow solid, m.p. >300 °C, *dr*> 20:1; IR (thin film): ν_{max} 3433, 3212, 3113, 2987, 2836, 1703, 1525, 1512, 1348, 752, 709 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6) δ 9.31 (br s, 1 H), 8.12 (d, *J*= 9.0 Hz, 2 H), 7.63-7.61 (m, 2 H), 7.54 (d, *J*= 7.8 Hz, 1 H), 7.50 (d, *J*= 8.4 Hz, 2 H), 7.30 (td, *J*= 7.8, 0.6 Hz, 1 H), 7.20 (ψt, *J*= 9.0 Hz, 2 H), 7.09 (td, *J*= 7.8, 0.6 Hz, 1 H), 6.79 (d, *J*= 7.8 Hz, 1 H), 4.90 (d, *J*= 11.4 Hz, 1 H), 4.49 (dd, *J*= 11.4, 8.4 Hz, 1 H), 4.36 (dd, *J*= 18.0, 8.4 Hz, 1 H), 3.27-3.17 (m, 2 H), 3.02 (dd, *J*= 17.4, 8.4 Hz, 1 H), 2.62 (dd, *J*= 17.4, 10.2 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.2,

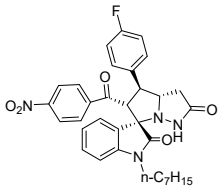
174.0, 173.6, 150.3, 143.1, 141.8, 136.0, 130.6, 130.4 (d), 129.6, 127.8, 123.8, 123.3, 122.6, 116.0, 115.9, 109.2, 76.3, 68.6, 60.6, 50.6, 37.0, 24.6, 12.0; ^{19}F NMR (376 MHz, DMSO-d_6): -115.6. HRMS (ESI): *m/z* calcd for $C_{28}H_{23}FN_4O_5Na$ $[M+Na]^+$ 537.1550, found 537.1600.



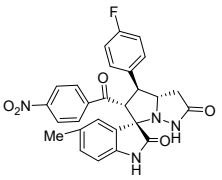
3t: 400 mg, 76% yield, a yellow solid, m.p. 194-195 °C, *dr*> 20:1; IR (thin film): ν_{max} 3405, 3232, 3078, 1706, 1528, 1510, 863, 755 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.31 (br s, 1 H), 8.08 (dd, *J*= 8.8, 1.2 Hz, 2 H), 7.60-7.57 (m, 2 H), 7.53-7.47 (m, 3 H), 7.24 (ψt, *J*= 7.8 Hz, 1 H), 7.17-7.13 (m, 2 H), 7.06 (ψt, *J*= 7. 6 Hz, 1 H), 6.64 (d, *J*= 8.0 Hz, 1 H), 5.23-5.16 (m, 1 H), 4.90-4.77 (m, 3 H), 4.49-4.30 (m, 2 H), 3.87-3.82 (m, 1 H), 3.68 (dd, *J*= 16.2, 5.4 Hz, 1 H), 3.00 (dd, *J*= 17.4, 8.2 Hz, 1 H), 2.58 (dd, *J*= 17.4, 9.8 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.0, 174.2, 173.8, 150.4, 143.2, 141.6, 135.9 (d), 131.6, 130.5, 130.4 (d), 129.6, 127.7, 123.9, 123.2, 122.7, 117.6, 116.0, 115.9, 109.8, 76.4, 68.6, 60.5, 50.6, 42.2, 37.0; ^{19}F NMR (376 MHz, DMSO-d_6): -115.5. HRMS (ESI): *m/z* calcd for $C_{29}H_{23}N_4O_5FNa$ $[M+Na]^+$ 549.1550, found 549.1601.



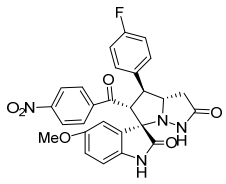
3u: 425 mg, 84%, a white solid, m.p. 213-214 °C, *dr> 20:1*; IR (thin film): ν_{max} 3395, 3271, 3232, 3112, 2124, 1710, 1696, 1520, 1512, 1342, 861, 748 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6) δ 9.31 (br s, 1 H), 8.10 (d, *J*= 8.4 Hz, 2 H), 7.64-7.61 (m, 2 H), 7.56 (d, *J*= 7.2 Hz, 1 H), 7.53-7.51 (m, 2 H), 7.34 (td, *J*= 7.8, 1.2 Hz, 1 H), 7.21-7.17 (m, 2 H), 7.13 (td, *J*= 7.8, 0.6 Hz, 1 H), 6.84 (d, *J*= 7.8 Hz, 1 H), 4.92 (d, *J*= 11.4 Hz, 1 H), 4.49 (dd, *J*= 11.4, 9.0 Hz, 1 H), 4.12 (dd, *J*= 17.7, 2.7 Hz, 1 H), 4.06 (dd, *J*= 17.7, 2.7 Hz, 1 H), 3.02 (dd, *J*= 17.4, 7.8 Hz, 1 H), 2.89 (t, *J*= 2.4 Hz, 1 H), 2.62 (dd, *J*= 17.1, 9.9 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 195.8, 173.8, 173.6, 150.5, 142.2, 141.3, 135.8 (d), 130.5, 130.4, 130.3, 129.4, 127.8, 124.0, 123.1 (d), 116.0, 115.9, 109.9, 77.4, 76.5, 74.5, 68.6, 60.5, 50.7, 37.0, 29.4; ^{19}F NMR (376 MHz, DMSO-d_6): -115.5. HRMS (ESI): *m/z* calcd for $C_{29}H_{21}N_4O_5FNa$ $[M+Na]^+$ 547.1394, found 547.1421.



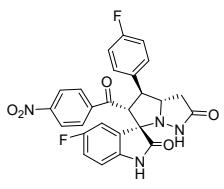
3v: 356 mg, 61% yield, a yellow solid, m.p. 189-190 °C, *dr*> 20:1; IR (thin film): ν_{max} 3401, 3227, 3110, 2931, 2858, 1702, 1522, 1512, 1345, 862, 747 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.30 (br s, 1 H), 8.11 (dt, *J*= 8.8, 2.0 Hz, 2 H), 7.60-7.56 (m, 2 H), 7.54-7.52 (m, 3 H), 7.26 (td, *J*= 7.8, 1.0 Hz, 1 H), 7.17-7.11 (m, 2 H), 7.06 (td, *J*= 7.6, 0.6 Hz, 1 H), 6.76 (d, *J*= 7.6 Hz, 1 H), 4.91 (d, *J*= 11.6 Hz, 1 H), 4.47 (dd, *J*= 11.6, 8.4 Hz, 1 H), 4.32 (dd, *J*= 18.4, 8.8 Hz, 1 H), 3.19-3.16 (m, 1 H), 3.13-3.06 (m, 1 H), 3.00 (dd, *J*= 17.6, 8.4 Hz, 1 H), 2.58 (dd, *J*= 17.2, 10.0 Hz, 1 H), 1.20-1.11 (m, 2 H), 1.03-0.84 (m, 7 H), 0.79 (t, *J*= 7.2 Hz, 3 H), 0.74-0.63 (m, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.0, 174.2, 173.7, 150.4, 143.5, 141.8, 135.9, 130.7, 130.5 (d), 129.8, 127.9, 124.0, 123.3, 122.6, 116.1, 115.9, 109.4, 76.4, 68.6, 60.1, 50.8, 37.0, 31.6, 28.9, 26.9, 26.6, 22.6, 14.4 (2 C); ^{19}F NMR (376 MHz, DMSO-d_6): -115.5. HRMS (ESI): *m/z* calcd for $C_{33}H_{33}FN_4O_5Na$ $[M+Na]^+$ 607.2333, found 607.2357.



3w: 390 mg, 78% yield, a yellow solid, m.p. 230-231 °C, *dr*> 20:1; IR (thin film): ν_{max} 3377, 3190, 3078, 2824, 1703, 1689, 1528, 1510, 1344, 821, 686 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.03 (br s, 1 H), 9.25 (br s, 1 H), 8.09 (dt, *J*= 8.8, 2.0 Hz, 2 H), 7.59-7.55 (m, 2 H), 7.51 (dt, *J*= 8.8, 2.0 Hz, 1 H), 7.21 (s, 1 H), 7.17-7.13 (m, 2 H), 6.95 (d, *J*= 7.6 Hz, 1 H), 6.38 (d, *J*= 8.0 Hz, 1 H), 4.78 (d, *J*= 11.6 Hz, 1 H), 4.40 (dd, *J*= 11.2, 8.8 Hz, 1 H), 4.30 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.96 (dd, *J*= 17.4, 8.2 Hz, 1 H), 2.57 (dd, *J*= 17.4, 9.8 Hz, 1 H), 2.27 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.4, 176.3, 173.8, 150.3, 141.9, 140.3, 136.2 (d), 131.0, 130.8, 130.4, 130.3, 129.7, 128.3, 123.8 (d), 116.1, 115.9, 110.1, 76.9, 68.8, 61.0, 50.6, 37.1, 21.3; ^{19}F NMR (376 MHz, DMSO-d_6): -115.6; HRMS (ESI): *m/z* calcd for $C_{27}H_{21}FN_4O_5Na$ $[M+Na]^+$ 523.1394, found 523.1409.

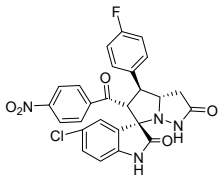


3x: 346 mg, 67% yield, a yellow solid, m.p. 256-257 °C, *dr*> 20:1; IR (thin film): ν_{max} 3326, 3170, 3108, 3080, 1735, 1689, 1524, 1514, 1345, 817, 677 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.03 (br s, 1 H), 9.28 (br s, 1 H), 8.10 (d, *J*= 8.8 Hz, 2 H), 7.60-7.54 (m, 4 H), 7.14 (ψt, *J*= 8.8 Hz, 2 H), 6.97 (d, *J*= 2.4 Hz, 1 H), 6.75 (dd, *J*= 8.4, 2.4 Hz, 1 H), 6.43 (d, *J*= 8.4 Hz, 1 H), 4.79 (d, *J*= 11.2 Hz, 1 H), 4.40 (dd, *J*= 11.4, 8.6 Hz, 1 H), 4.29 (dd, *J*= 18.0, 8.4 Hz, 1 H), 3.71 (s, 3 H), 2.94 (dd, *J*= 17.2, 8.0 Hz, 1 H), 2.57 (dd, *J*= 17.4, 9.8 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.3, 176.1, 173.8, 155.1, 150.3, 141.8, 136.2, 161.1, 130.5, 130.4, 129.7, 125.1, 123.9, 116.1, 115.9 (d), 114.3, 110.8, 77.2, 68.7, 60.8, 56.2, 50.9, 37.1; ^{19}F NMR (376 MHz, DMSO-d_6): -115.6; HRMS (ESI): *m/z* calcd for $C_{27}H_{21}FN_4O_6Na$ $[M+H]^+$ 539.1343, found 539.1351.

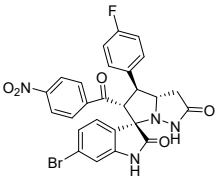


3y: 469 mg, 93% yield, a yellow solid, m.p. 215-216 °C, *dr*> 20:1; IR (thin film): ν_{max} 3416, 3174, 3073, 2928, 2849, 1705, 1693, 1526, 1344, 738 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6) δ 10.19 (br s, 1 H), 9.34 (br s, 1 H), 8.16 (d, *J*= 8.4 Hz, 2 H), 7.63-7.61 (m, 4 H), 7.44 (dd, *J*= 8.4, 2.4 Hz, 1 H), 7.19 (ψt, *J*= 8.7 Hz, 2 H), 7.06 (td, *J*= 8.7, 2.4 Hz, 1 H), 6.54 (dd, *J*= 8.4, 4.2 Hz, 1 H), 4.87 (d, *J*= 11.4 Hz, 1 H), 4.49 (dd, *J*= 11.7, 2.7 Hz, 1 H), 4.33 (dd, *J*= 18.0, 8.4 Hz, 1 H), 3.08 (dd, *J*= 17.4, 8.4 Hz, 1 H), 2.57 (dd, *J*= 17.4, 4.2 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.2, 176.2, 173.8, 150.4, 141.6, 139.1, 135.8, 130.4, 130.3,

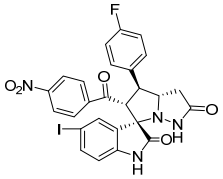
129.7, 125.5, 123.9, 117.1 (d), 116.0, 115.9, 115.7, 111.1, 77.1, 68.6, 60.5, 50.5, 36.6; ^{19}F NMR (376 MHz, DMSO-d_6): -115.5, -121.4; HRMS (ESI): *m/z* calcd for $C_{26}H_{18}F_2N_4O_5Na$ [M+Na]$^+$ 527.1143, found 527.1145.



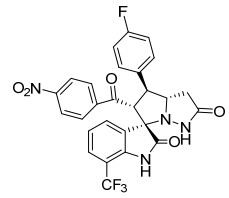
3z: 432 mg, 83% yield, a yellow solid, m.p. 171-172 °C, *dr*> 20:1; I IR (thin film): ν_{max} 3434, 3188, 2812, 1705, 1688, 1527, 1510, 1343, 706, 680cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.31 (br s, 1 H), 9.33 (br s, 1 H), 8.15 (d, *J*= 8.4 Hz, 2 H), 7.65-7.60 (m, 4 H), 7.51 (d, *J*= 2.0 Hz, 1 H), 7.26 (ddt, *J*= 8.4, 2.0 Hz, 1 H), 7.18 (ψt, *J*= 9.0 Hz, 2 H), 6.55 (d, *J*= 8.4 Hz, 1 H), 4.88 (d, *J*= 11.6 Hz, 1 H), 4.47 (dd, *J*= 11.6, 8.8 Hz, 1 H), 4.32 (dd, *J*= 18.0, 8.4 Hz, 1 H), 3.03 (dd, *J*= 17.0, 8.0 Hz, 1 H), 2.59 (dd, *J*= 17.6, 10.0 Hz, 1 H) ; ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.3, 176.0, 173.6, 150.4, 141.8, 141.6, 135.6 (d), 130.5, 130.4 (d), 129.7, 127.8, 126.3, 125.8, 123.9, 116.0, 115.8, 111.7, 76.8, 68.7, 60.7, 50.5, 36.5; ^{19}F NMR (376 MHz, DMSO-d_6): -115.5; HRMS (ESI): *m/z* calcd for $C_{27}H_{21}N_4O_6FNa$ [M+Na]$^+$ 539.1343, found 539.1372.



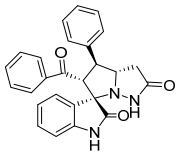
3aa: 508 mg, 80% yield, a yellow solid, m.p. 201-202 °C, *dr*> 20:1; IR (thin film): ν_{max} 3270, 3072, 1715, 1690, 1611, 1527, 1512, 1344, 1288, 826, 696 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6) δ 10.33 (br s, 1 H), 9.32 (br s, 1 H), 8.17 (d, *J*= 8.4 Hz, 2 H), 7.63 (dd, *J*= 9.0 Hz, 1 H), 7.61-7.59 (m, 2 H), 7.46 (d, *J*= 8.4 Hz, 1 H), 7.22-7.17 (m, 3 H), 6.70 (d, *J*= 1.8 Hz, 1 H), 4.88 (d, *J*= 11.4 Hz, 1 H), 4.43 (dd, *J*= 11.4, 8.4 Hz, 1 H), 4.33 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.98 (dd, *J*= 17.4, 8.4 Hz, 1 H), 2.61 (dd, *J*= 17.4, 11.2 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.2, 176.1, 173.9, 150.5, 144.4, 141.6, 135.8, 130.5, 130.4, 129.9, 129.8, 124.7, 124.0, 123.4, 123.2, 116.0 (d), 113.2, 76.8, 68.1, 60.7, 51.5, 37.3; ^{19}F NMR (376 MHz, DMSO-d_6): -115.4; HRMS (ESI): *m/z* calcd for $C_{26}H_{18}N_4O_5FBrNa$ [M+Na]$^+$ 547.1394, found 547.1421.



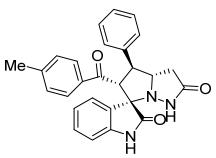
3ab: 447 mg, 73% yield, a yellow solid, m.p. 169-170 °C, *dr*> 20:1; IR (thin film): ν_{max} 3373,3111, 3042, 1731, 1693, 1670, 1527, 1511, 1345, 824, 668cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6) δ 10.32 (br s, 1 H), 9.32 (br s, 1 H), 8.15 (dt, *J*= 9.0, 1.8 Hz, 2 H), 7.66-7.63 (m, 3 H), 7.59 (dt, *J*= 9.0, 1.8 Hz, 2 H), 7.53 (dd, *J*= 8.1, 1.5 Hz, 1 H), 7.19 (ψt, *J*= 9.0 Hz, 2 H), 6.38 (d, *J*= 7.8 Hz, 1 H), 4.86 (d, *J*= 11.4 Hz, 1 H), 4.43 (dd, *J*= 11.4, 9.0 Hz, 1 H), 4.31 (dd, *J*= 17.4, 9.0 Hz, 1 H), 2.95 (dd, *J*= 17.4, 7.2 Hz, 1 H), 2.61 (dd, *J*= 17.4, 7.2 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 195.7, 175.2, 172.9, 149.7, 142.0, 141.1, 138.6, 135.2, 135.1 (d), 129.9, 129.8, 129.0, 125.8, 123.3, 115.4, 115.3, 112.1, 76.0, 68.2, 60.3, 50.0, 36.1; ^{19}F NMR (376 MHz, DMSO-d_6): -115.5. HRMS (ESI): *m/z* calcd for $C_{26}H_{18}N_4O_5FINa$ [M+Na]$^+$ 635.0204, found 635.0235.



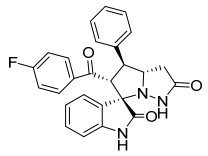
3ac: 277 mg, 50% yield, a yellow solid, m.p. 210-211 °C, *dr*> 20:1; IR (thin film): ν_{max} 3186, 3078, 2831, 1732, 1721, 1693, 1529, 1512, 1344, 742, 697 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.58 (br s, 1 H), 9.31 (br s, 1 H), 8.06 (dt, *J*= 8.8, 2.2 Hz, 2 H), 7.69 (d, *J*= 7.6 Hz, 1 H), 7.61-7.57 (m, 2 H), 7.45 (d, *J*= 8.0 Hz, 1 H), 7.41 (dt, *J*= 8.8, 2.2 Hz, 2 H), 7.19-7.14 (m, 3 H), 4.85 (d, *J*= 11.6 Hz, 1 H), 4.40 (dd, *J*= 11.2, 8.8 Hz, 1 H), 4.32 (dd, *J*= 17.6, 8.4 Hz, 1 H), 2.97 (dd, *J*= 17.4, 7.8 Hz, 1 H), 2.58 (dd, *J*= 17.2, 10.0 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 196.9, 177.1, 173.6, 150.1, 142.0, 140.3, 135.8, 131.9, 130.6 (d), 129.3, 126.9, 125.8, 123.9, 122.2, 116.1, 115.9, 116.3, 111.3, 75.3, 68.9, 61.8, 50.7, 36.7; ^{19}F NMR (376 MHz, DMSO-d_6): -60.1, -60.8, -115.5. HRMS (ESI): *m/z* calcd for $C_{27}H_{18}N_4O_5F_4Na$ [M+Na]$^+$ 577.1111, found 577.1155.



5a: 148 mg, 35%, a white solid, m.p. 138-139 °C, *dr> 20:1*; IR (thin film): ν_{max} 3397, 3175, 3063, 2830, 1710, 1682, 1619, 1474, 695, 752 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.27 (br s, 1 H), 9.30 (br s, 1 H), 7.55-7.45 (m, 6 H), 7.37-7.32 (m, 4 H), 7.24 (ψt, *J*= 7.4 Hz, 1 H), 7.16 (td, *J*= 7.6, 0.8 Hz, 1 H), 6.99 (td, *J*= 7.4, 0.8 Hz, 1 H), 6.56 (d, *J*= 7.6 Hz, 1 H), 4.80 (d, *J*= 11.6 Hz, 1 H), 4.45 (dd, *J*= 11.2, 8.4 Hz, 1 H), 4.38 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.97 (dd, *J*= 17.2, 8.4 Hz, 1 H), 2.62 (dd, *J*= 17.2, 9.6 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.0, 176.5, 173.8, 142.8, 140.3, 136.9, 133.9, 130.1, 129.3, 128.9, 128.2 (2 C), 128.1, 127.6, 124.1, 121.9, 110.1, 77.1, 68.6, 59.6, 52.1, 37.4; HRMS (ESI): *m/z* calcd for $C_{26}H_{21}N_3O_3Na$ [M+Na]$^+$ 446.1481, found 446.1565.

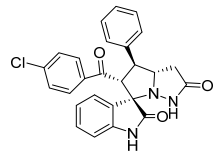


5b: 187 mg, 43% yield, a white solid, m.p. 254-255 °C, *dr> 20:1*; IR (thin film): ν_{max} 3367, 3125, 3033, 2830, 1715, 1692, 1608, 1454, 843, 792, 683 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.27 (br s, 1 H), 9.27 (br s, 1 H), 7.47-7.44 (m, 3 H), 7.40 (d, *J*= 8.0 Hz, 2 H), 7.33 (ψt, *J*= 7.6 Hz, 2 H), 7.22 (ψt, *J*= 7.2 Hz, 1 H), 7.15 (td, *J*= 7.6, 0.8 Hz, 1 H), 7.14 (d, *J*= 8.0 Hz, 2 H), 6.97 (td, *J*= 7.6, 0.8 Hz, 1 H), 6.57 (d, *J*= 7.6 Hz, 1 H), 4.76 (d, *J*= 11.6 Hz, 1 H), 4.44 (dd, *J*= 11.6, 8.4 Hz, 1 H), 4.36 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.96 (dd, *J*= 17.2, 8.4 Hz, 1 H), 2.61 (dd, *J*= 17.2, 10.0 Hz, 1 H), 2.28 (s, 3 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 195.1, 176.5, 173.8, 144.4, 142.7, 140.3, 134.4, 130.0, 129.5, 129.2, 128.4, 128.2, 128.1, 127.6, 124.1, 121.8, 110.1, 77.2, 68.6, 59.1, 52.1, 37.5, 21.6; HRMS (ESI): *m/z* calcd for $C_{27}H_{23}N_3O_3Na$ [M+Na]$^+$ 460.1637, found 460.1633.

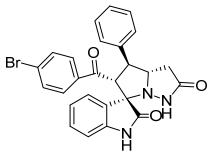


5c: 371 mg, 84% yield, a white solid, m.p. 225-226 °C, *dr> 20:1*; IR (thin film): ν_{max} 3219, 3087, 2695, 1713, 1688, 1674, 1597, 1233, 749, 702 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.36 (br s, 1 H), 9.30 (br s, 1 H), 7.54-7.43 (m, 5 H), 7.34 (ψt, *J*= 7.8 Hz, 2 H), 7.23 (t, *J*= 7.4 Hz, 1 H), 7.18 (ψt, *J*= 8.8 Hz, 2 H), 7.15 (d, *J*= 7.2 Hz, 1 H), 6.98 (ψt, *J*= 7.4 Hz, 1 H), 6.60 (d, *J*= 7.6 Hz, 1 H), 4.77 (dd, *J*= 11.2 Hz, 1 H), 4.45-4.34 (m, 2 H), 2.99 (dd, *J*= 17.2, 8.4 Hz, 1 H), 2.61 (dd, *J*= 17.0, 9.8 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 194.7, 176.4, 173.8, 142.8, 140.1, 133.6 (2 C), 131.3, 131.2, 130.1, 129.2, 128.2, 128.1, 127.6, 124.0, 121.9, 116.1, 115.9,

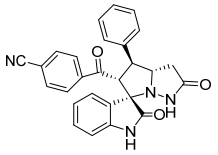
110.2, 77.1, 68.6, 59.6, 51.9, 37.4; HRMS (ESI): *m/z* calcd for $C_{26}H_{20}N_3O_3FNa$ $[M+Na]^+$ 464.1386, found 464.1419.



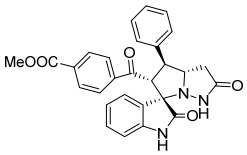
5d: 325 mg, 71% yield, a yellow solid, m.p. 217-218 °C, *dr> 20:1*; IR (thin film): ν_{max} 3244, 3036, 1718, 1683, 1473, 754, 688 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6) δ 10.25 (br s, 1 H), 9.29 (br s, 1 H), 7.54-7.52 (m, 3 H), 7.47-7.44 (m, 3 H), 7.40 (d, *J*= 8.4 Hz, 1 H), 7.33 (ψt, *J*= 7.8 Hz, 2 H), 7.16 (ψt, *J*= 7.8 Hz, 1 H), 6.99 (ψt, *J*= 7.5 Hz, 1 H), 6.55 (d, *J*= 7.8 Hz, 1 H), 4.77 (dd, *J*= 11.4 Hz, 1 H), 4.47 (dd, *J*= 11.4, 8.4 Hz, 1 H), 4.35 (dd, *J*= 18.0, 8.4 Hz, 1 H), 2.97 (dd, *J*= 17.4, 8.4 Hz, 1 H), 2.60 (dd, *J*= 17.4, 10.4 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.0, 176.4, 173.7, 142.8, 139.2, 136.8, 133.9, 132.2, 130.2, 130.1, 129.2, 128.8, 128.2, 128.1, 124.0, 121.8, 110.1, 77.0, 68.4, 59.6, 51.3, 37.2; HRMS (ESI): *m/z* calcd for $C_{26}H_{21}N_3O_3Cl$ $[M+H]^+$ 458.1271, found 458.1364.



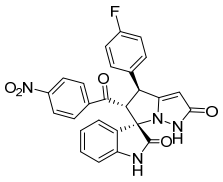
5e: 402 mg, 80% yield, a white solid, m.p. 181-182 °C, *dr> 20:1*; IR (thin film): ν_{max} 3408, 3183, 3063, 1710, 1684, 1339, 752, 697 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 10.29 (br s, 1 H), 9.30 (br s, 1 H), 7.56 (d, *J*= 8.0 Hz, 2 H), 7.50-7.46 (m, 3 H), 7.36-7.33 (m, 4 H), 7.24 (ψt, *J*= 6.8 Hz, 1 H), 7.18 (ψt, *J*= 7.6 Hz, 1 H), 6.99 (ψt, *J*= 7.4 Hz, 1 H), 6.58 (d, *J*= 7.2 Hz, 1 H), 4.77 (d, *J*= 11.6 Hz, 1 H), 4.45-4.33 (m, 2 H), 2.98 (dd, *J*= 17.2, 8.4 Hz, 1 H), 2.61 (dd, *J*= 17.0, 9.4 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.4, 176.4, 173.8, 142.7, 140.1, 135.9, 132.0, 130.2, 130.1, 129.2, 128.2, 128.1 (2 C), 127.6, 123.9, 121.9, 110.2, 77.0, 68.7, 60.2, 59.7, 51.8, 37.4; HRMS (ESI): *m/z* calcd for $C_{26}H_{20}N_3O_3BrNa$ $[M+Na]^+$ 524.0586, found 524.0600.



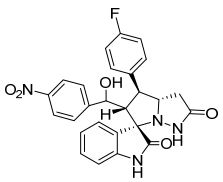
5f: 365 mg, 81% yield, a white solid, m.p. 243-344 °C, *dr> 20:1*; IR (thin film): ν_{max} 3202, 3076, 3028, 2821, 2237,1706, 1687, 1616, 1475, 1336, 757 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 10.18 (br s, 1 H), 9.29 (br s, 1 H), 7.81 (d, *J*= 8.4 Hz, 2 H), 7.52 (d, *J*= 7.2 Hz, 2 H), 7.47-7.45 (m, 3 H), 7.36 (ψt, *J*= 7.6 Hz, 2 H), 7.25 (ψt, *J*= 7.6 Hz, 1 H), 7.18 (td, *J*= 7.6, 0.8 Hz, 1 H), 7.00 (td, *J*= 7.6, 0.8 Hz, 1 H), 6.54 (d, *J*= 7.6 Hz, 1 H), 4.81 (dd, *J*= 11.2 Hz, 1 H), 4.42 (dd, *J*= 11.2, 8.4 Hz, 1 H), 4.34 (dd, *J*= 17.6, 8.4 Hz, 1 H), 2.97 (dd, *J*= 17.6, 8.4 Hz, 1 H), 2.61 (dd, *J*= 17.2, 9.6 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.3, 176.3, 173.7, 142.8, 140.3, 140.0, 132.8, 130.4, 129.2, 128.7, 128.3, 128.0, 127.6, 123.8, 122.0, 118.5, 115.7, 110.3, 76.8, 68.8, 60.4, 51.6, 37.3; HRMS (ESI): *m/z* calcd for $C_{27}H_{20}N_4O_3Na$ $[M+Na]^+$ 471.1433, found 471.1419.



5g: 194 mg, 40% yield, a white solid, m.p. 204-205 °C, *dr> 20:1*; IR (thin film): ν_{max} 3398, 3121, 3019, 1710, 1699, 1684, 1609, 1445, 782, 727, 693 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 10.28 (br s, 1 H), 9.29 (br s, 1 H), 7.85 (d, *J*= 8.4 Hz, 2 H), 7.51 (d, *J*= 7.2 Hz, 2 H), 7.46-7.43 (m, 3 H), 7.35 (ψt, *J*= 7.6 Hz, 2 H), 7.24 (ψt, *J*= 7.4 Hz, 1 H), 7.16 (ψt, *J*= 7.4 Hz, 1 H), 6.98 (ψt, *J*= 7.4 Hz, 1 H), 6.54 (d, *J*= 7.6 Hz, 1 H), 4.81 (dd, *J*= 11.2 Hz, 1 H), 4.43 (dd, *J*= 11.2, 8.0 Hz, 1 H), 4.35 (dd, *J*= 17.6, 8.4 Hz, 1 H), 3.85 (s, 3 H), 2.96 (dd, *J*= 17.6, 8.0 Hz, 1 H), 2.61 (dd, *J*= 17.2, 9.6 Hz, 1 H); ^{13}C NMR (150 MHz, DMSO-d_6): δ 196.5, 176.3, 173.8, 165.9, 142.8, 140.4, 140.1, 133.7, 130.2, 129.5, 129.3, 128.4, 128.3, 128.0, 127.6, 123.8, 121.9, 110.3, 76.9, 68.8, 60.4, 53.0, 51.6, 37.3; HRMS (ESI): *m/z* calcd for $C_{28}H_{23}N_3O_5Na$ $[M+Na]^+$ 504.1535, found 504.1513.



6a: 173 mg, 72% yield, a yellow solid, m.p. 202-203 °C, *dr> 20:1*; IR (thin film): ν_{max} 3214, 3105, 3080, 1732, 1696, 1523, 1517, 1345, 749 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 10.80 (br s, 1 H), 10.05 (br s, 1 H), 8.10 (d, *J*= 8.8 Hz, 2 H), 7.51-7.47 (m, 4 H), 7.26 (ψt, *J*= 8.8 Hz, 2 H), 7.12 (ψt, *J*= 7.6, 0.8 Hz, 1 H), 6.91 (ψt, *J*= 7.6 Hz, 1 H), 6.85 (d, *J*= 6.8 Hz, 1 H), 6.49 (d, *J*= 8.0 Hz, 1 H), 5.53 (d, *J*= 8.0 Hz, 1 H), 5.35 (s, 1 H), 4.95 (d, *J*= 8.0 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 195.9, 174.6, 166.1, 150.1, 148.5, 142.1, 141.4, 136.7, 131.2, 130.4, 129.5, 125.5, 125.2, 123.9, 122.9, 116.4, 116.2, 110.7, 86.2, 70.1, 66.0, 42.6; ^{19}F NMR (376 MHz, DMSO-d_6): -115.0; HRMS (ESI): *m/z* calcd for $C_{26}H_{17}N_4O_5Na$ $[M+Na]^+$ 507.1081, found 507.1098.



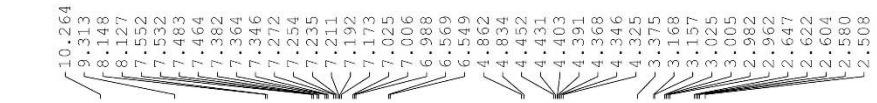
6b: 169 mg, 69% yield, a white solid, m.p. 233-234 °C, *dr> 20:1*; IR (thin film): ν_{max} 3396, 3207, 3058, 2923, 1716, 1696, 1507, 1349, 855 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 10.19 (br s, 1 H), 9.27 (br s, 1 H), 7.75 (ψd, *J*= 8.8 Hz, 3 H), 7.28 (ψt, *J*= 7.6 Hz, 1 H), 7.14 (d, *J*= 8.8 Hz, 2 H), 7.06 (ψt, *J*= 7.6 Hz, 1 H), 6.99-6.96 (m, 2 H), 6.84 (d, *J*= 7.6 Hz, 1 H), 6.79 (ψt, *J*= 8.8 Hz, 2 H), 5.26 (br s, 1 H), 4.36(d, *J*= 9.6 Hz, 1 H), 4.08-4.01 (m, 1 H), 3.72 (dd, *J*= 12.0, 7.2 Hz, 1 H), 3.25 (dd, *J*= 12.0, 9.6 Hz, 1 H), 2.79 (dd, *J*= 17.2, 10.0 Hz, 1 H), 2.39 (dd, *J*= 17.2, 10.0 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 194.7, 176.4, 173.8, 142.8, 140.1, 133.6 (2 C), 131.3, 131.2, 130.1, 129.2, 128.2, 128.1, 127.6, 124.0, 121.9, 116.1, 115.9, 110.22, 77.1, 68.6, 59.6, 51.9, 37.4; ^{19}F NMR (376 MHz, DMSO-d_6): -117.1; HRMS (ESI): *m/z* calcd for $C_{26}H_{21}N_4O_5Na$ $[M+Na]^+$ 511.1394, found 511.1405.

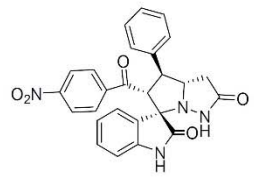
VIII. References

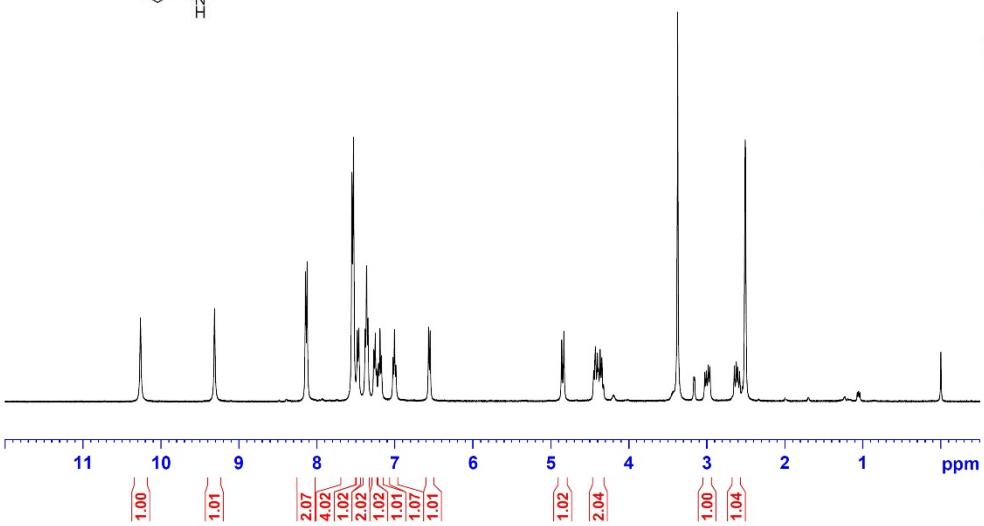
1. X. Wang, P. Yang, Y. Zhang, C.-Z. Tang, F. Tian, L. Peng and L.-X. Wang, *Org. Lett.*, 2017, **19**, 646–649.
2. E. P. Kohler and H. M. Chadwell, *Org. Synth.*, 1922, **1**, 1–3.

IX. Data of all new compounds 3, 5 and 6.

^{1}H NMR and ^{13}C NMR Spectra for Compound **3a**





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DS                     2
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FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                 141.71
DW                 62.400 usec
DE                  6.50 usec
TE                   0.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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NUC1                   1H
P1                  12.66 usec
SI                  65536
SF          400.1300000 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
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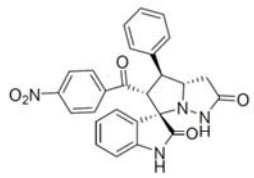

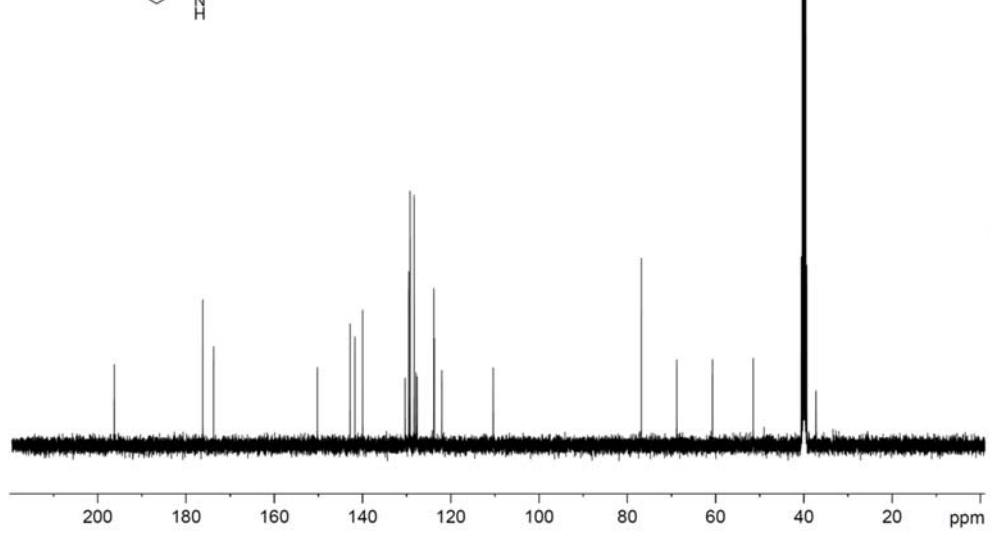


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PROCNO                  1
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PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                   669
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                 195.5
DW                 20.800 usec
DE                  6.50 usec
TE                   0.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
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NUC1                   13C
P1                  9.44 usec
SI                  32768
SF          100.6127690 MHz
WDW                    no
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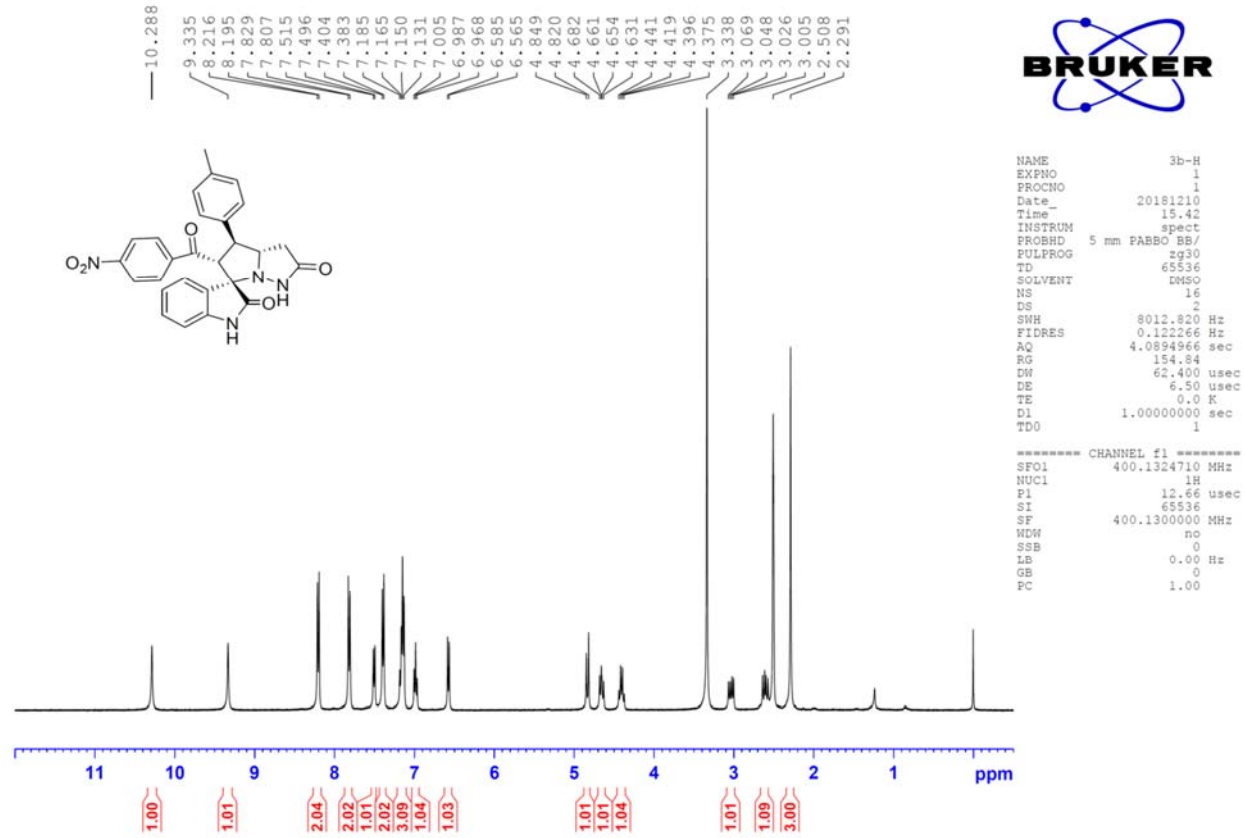
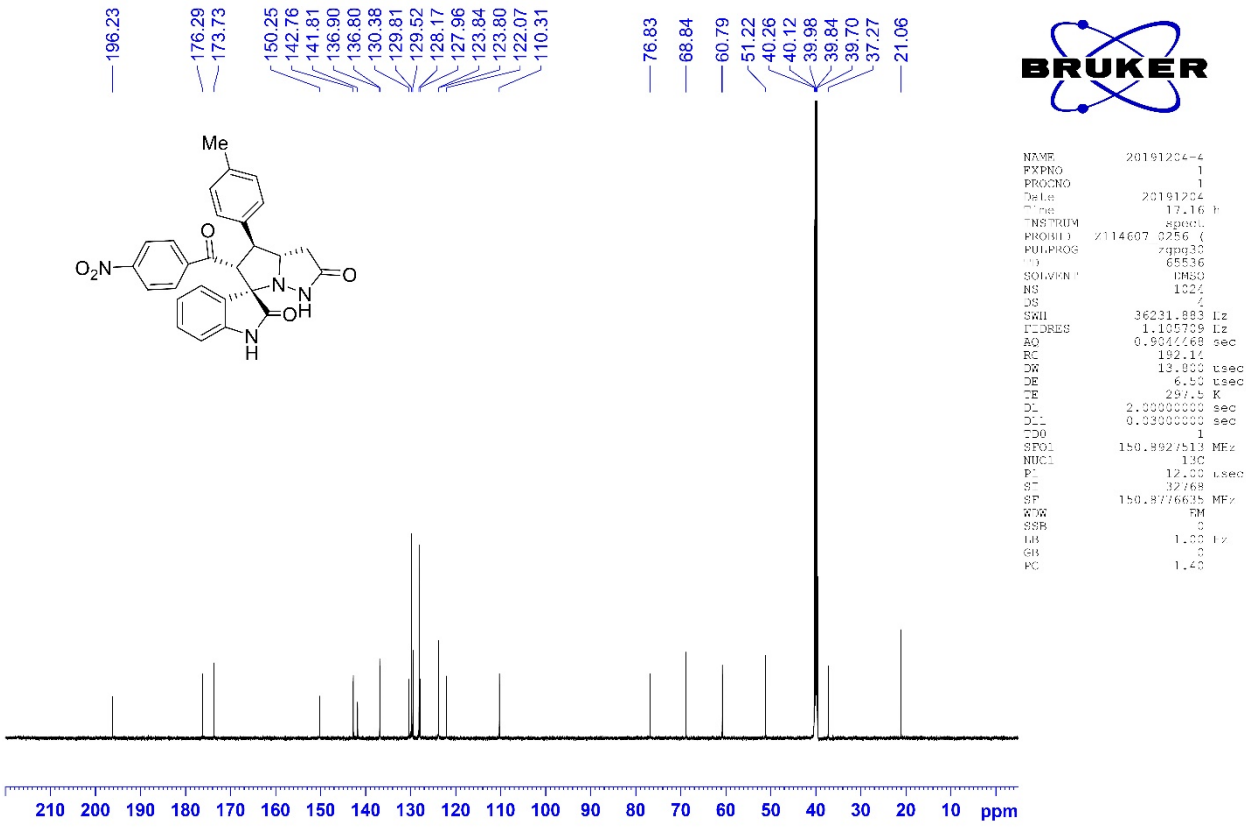
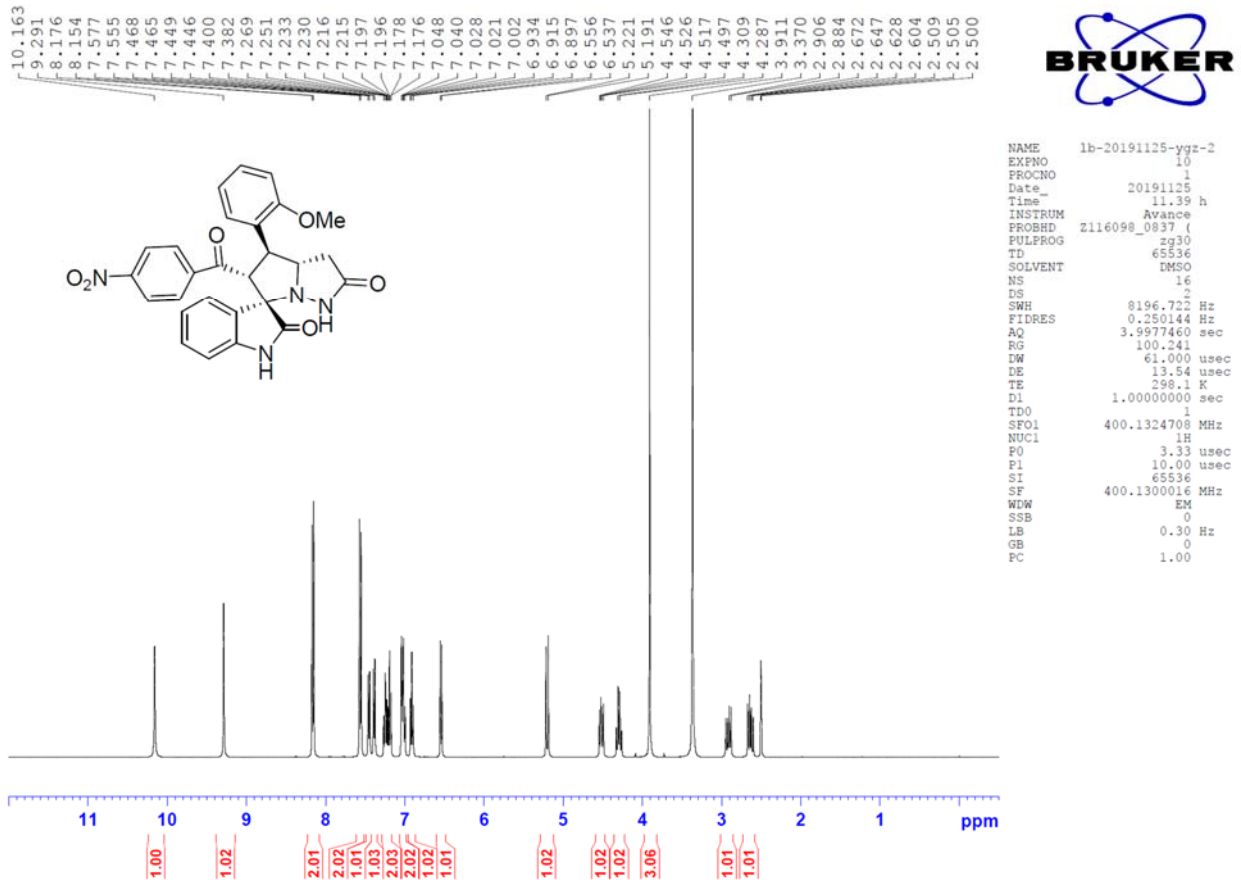
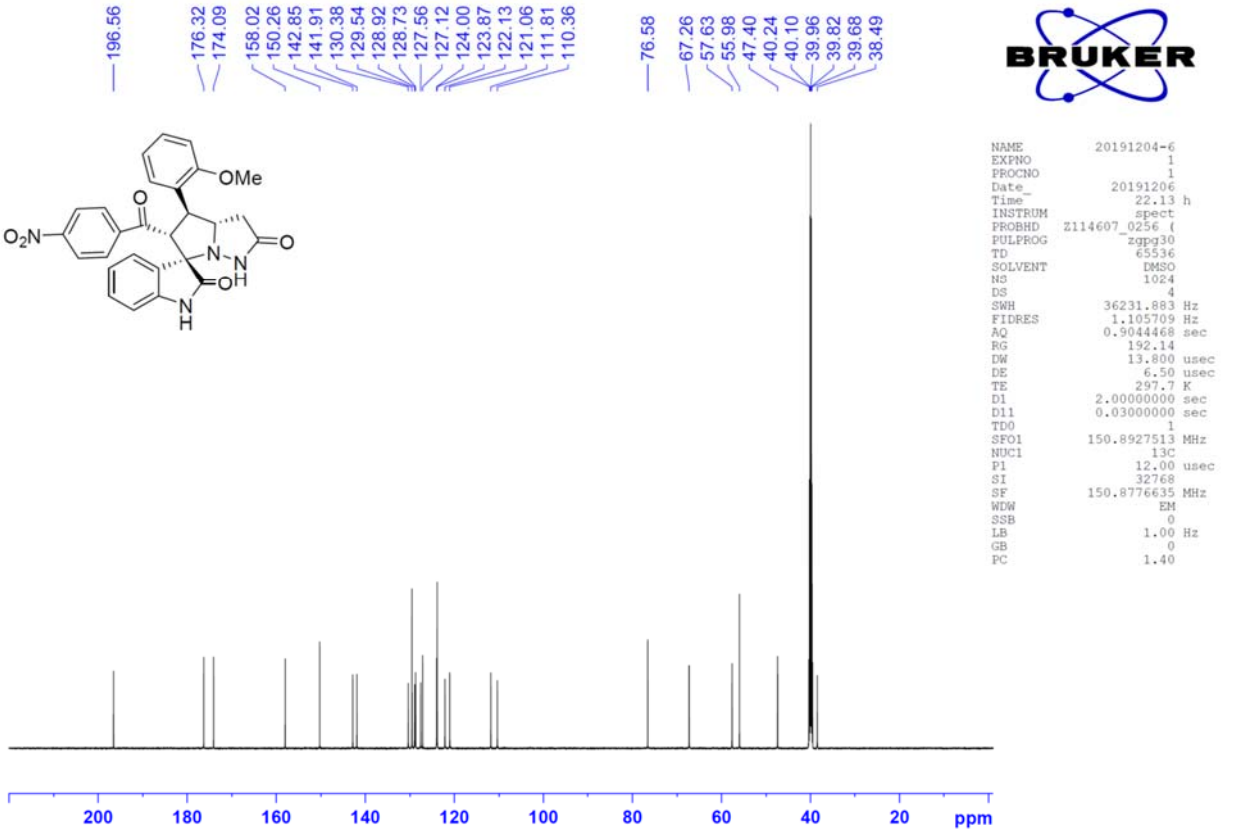
^{1}H NMR and ^{13}C NMR Spectra for Compound **3b**



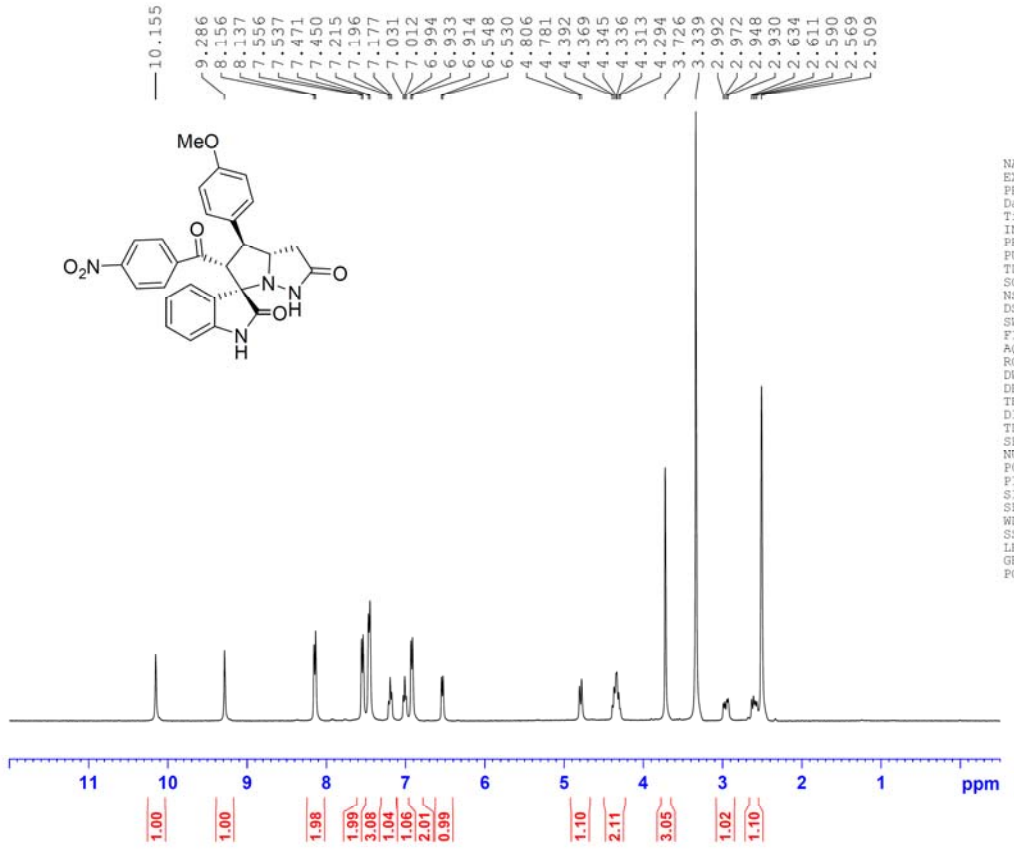


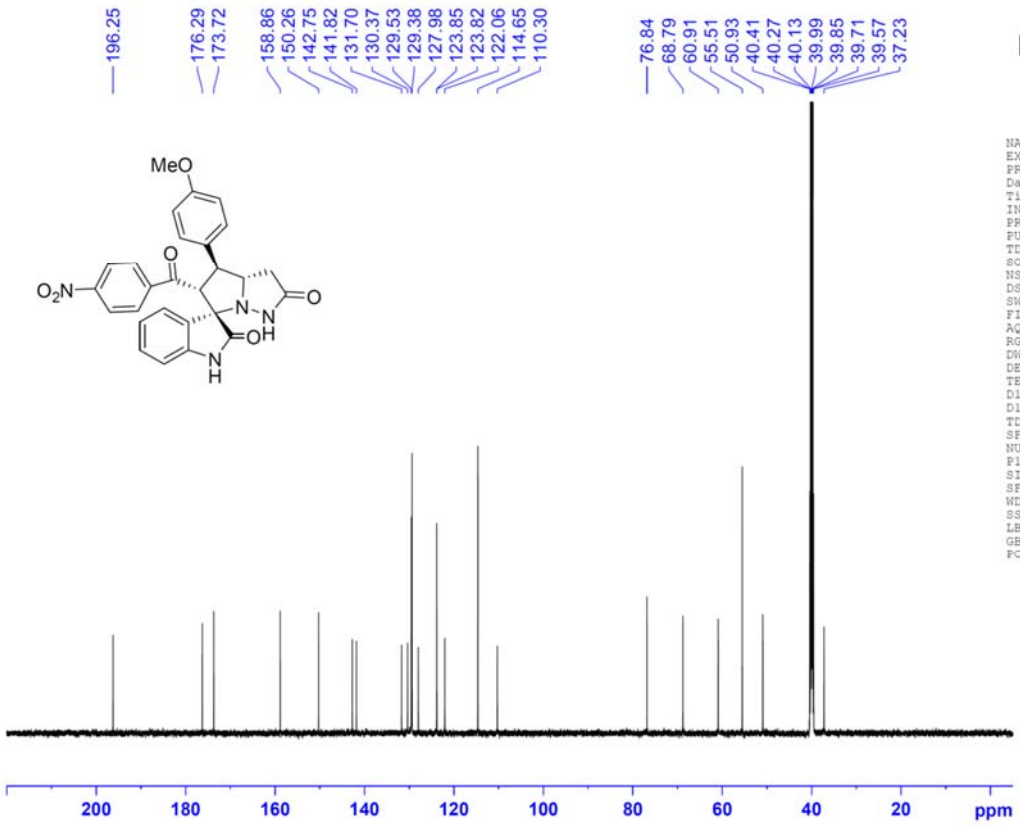
^{1}H NMR and ^{13}C NMR Spectra for Compound **3c**



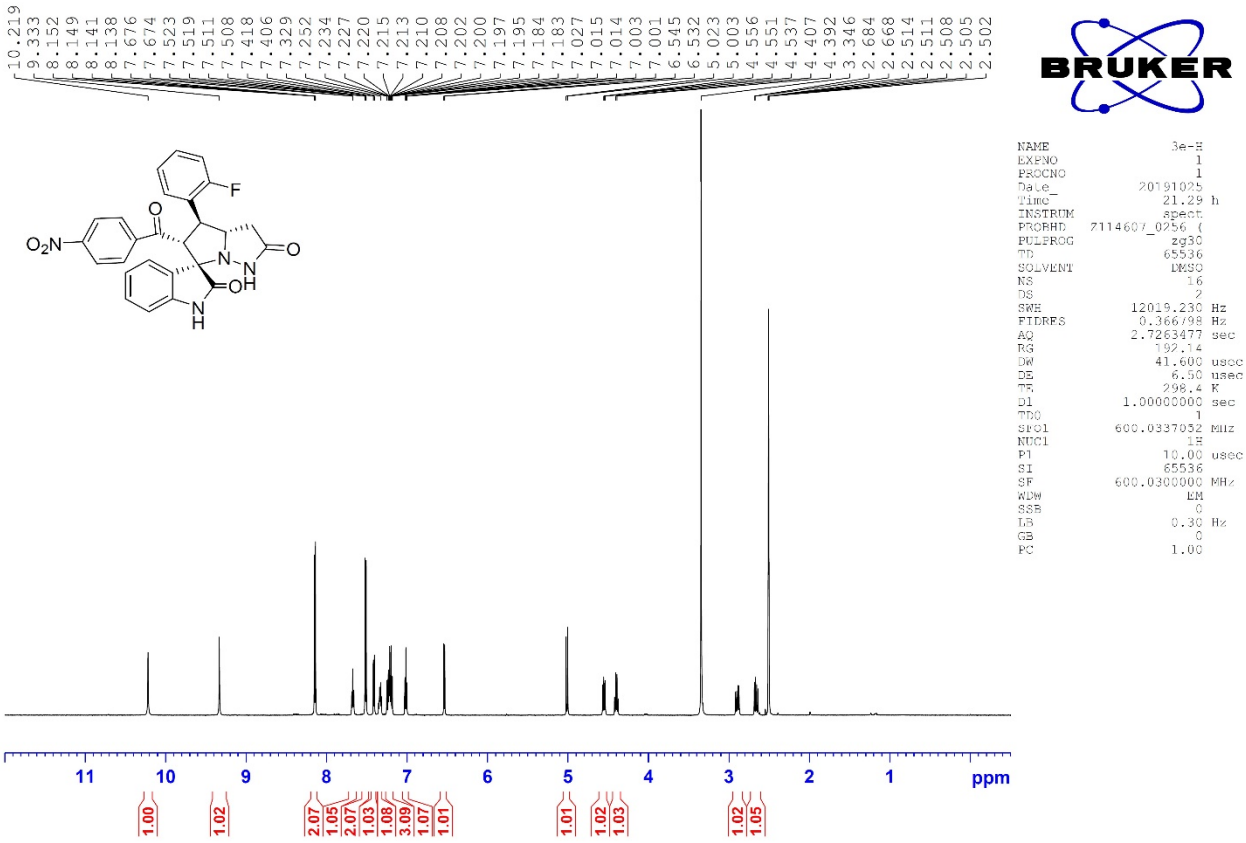


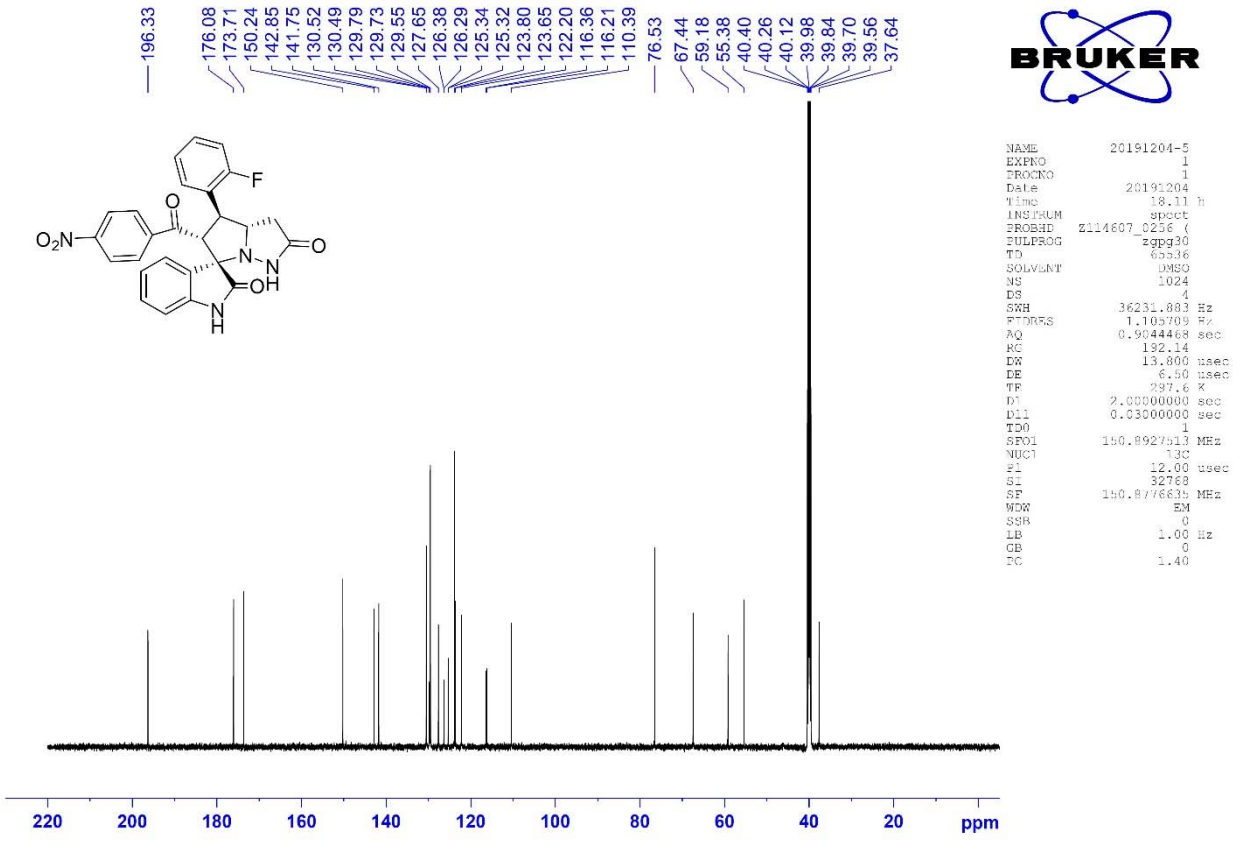
^{1}H NMR and ^{13}C NMR Spectra for Compound **3d**

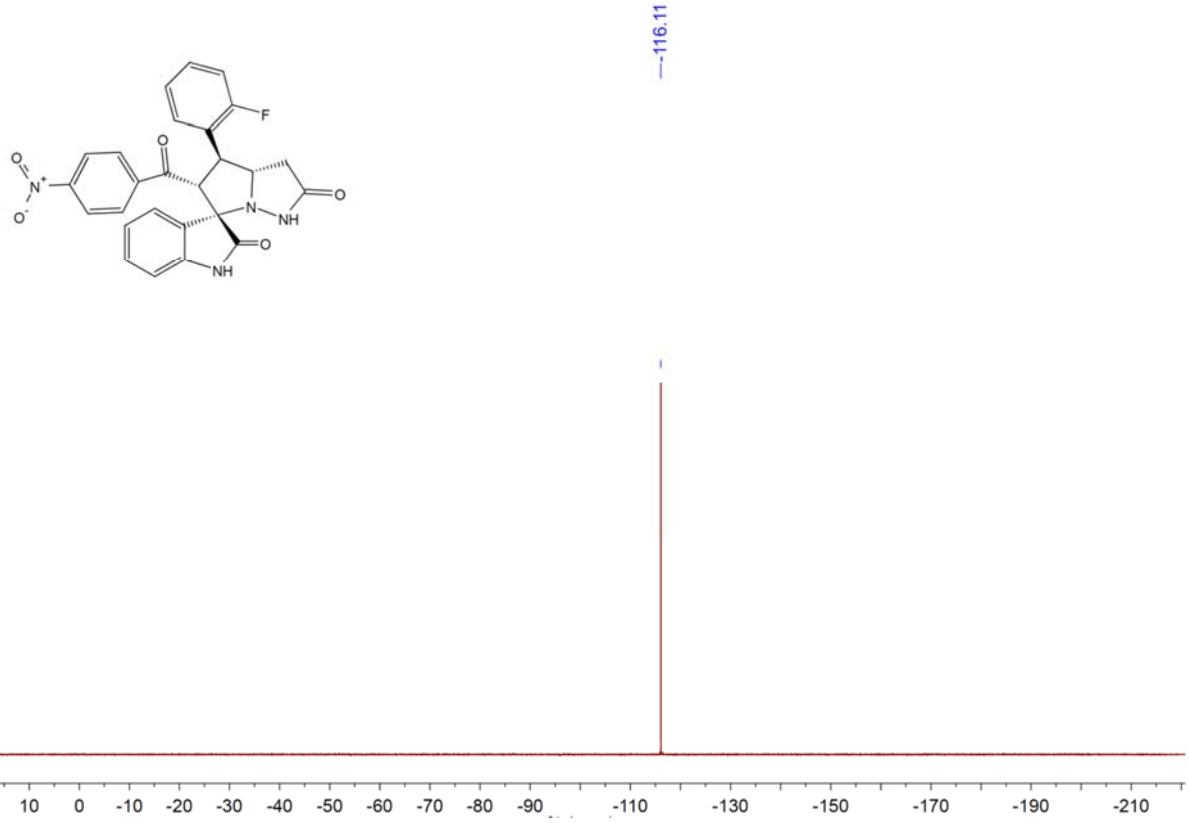




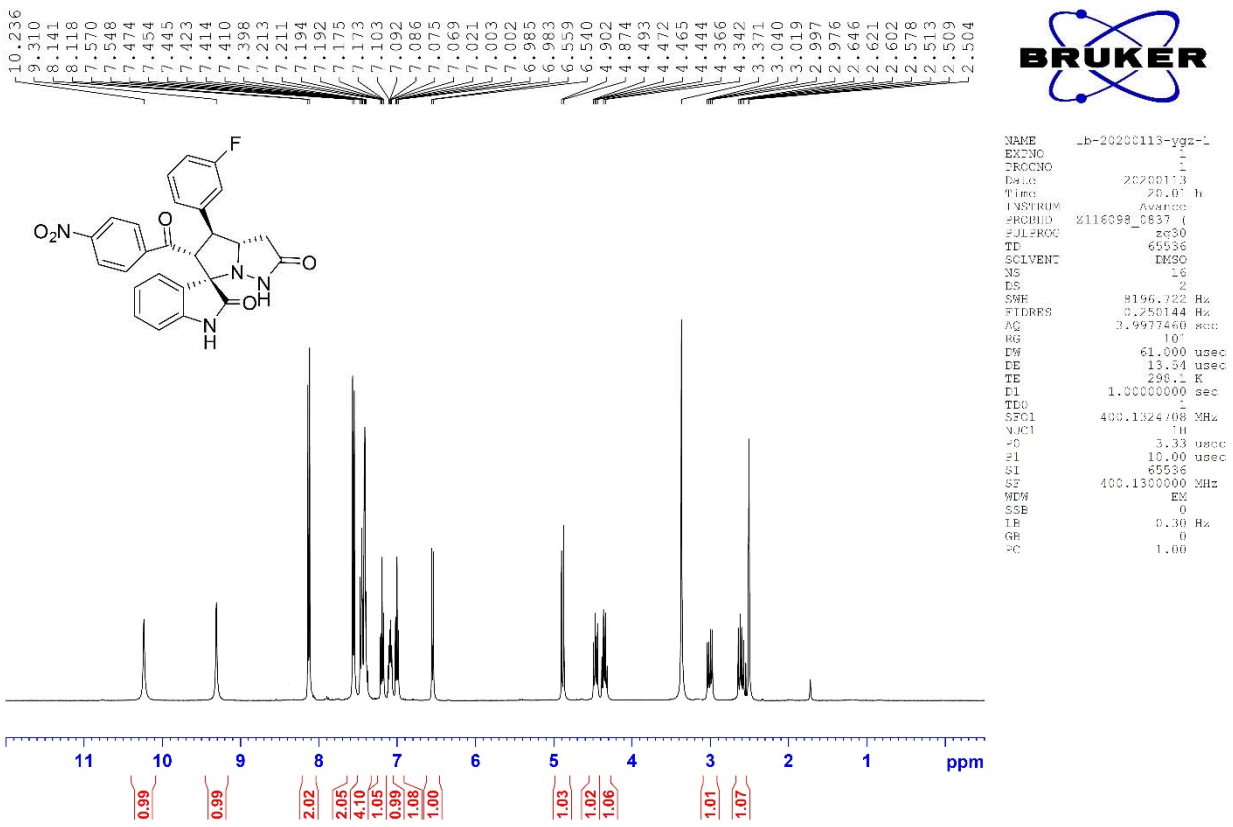
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3e**

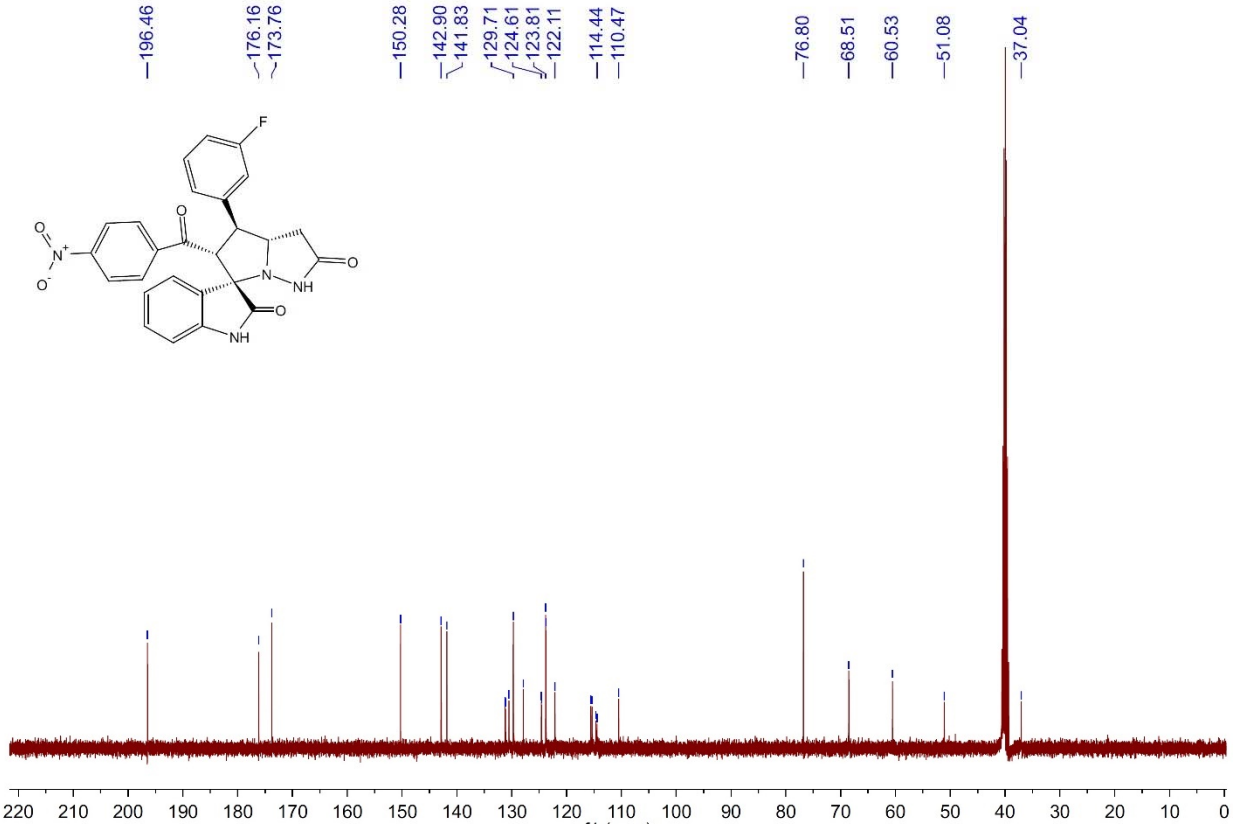






^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3f**




196.46
176.16
173.76
150.28
142.90
141.83
129.71
124.61
123.81
122.11
114.44
110.47
76.80
68.51
60.53
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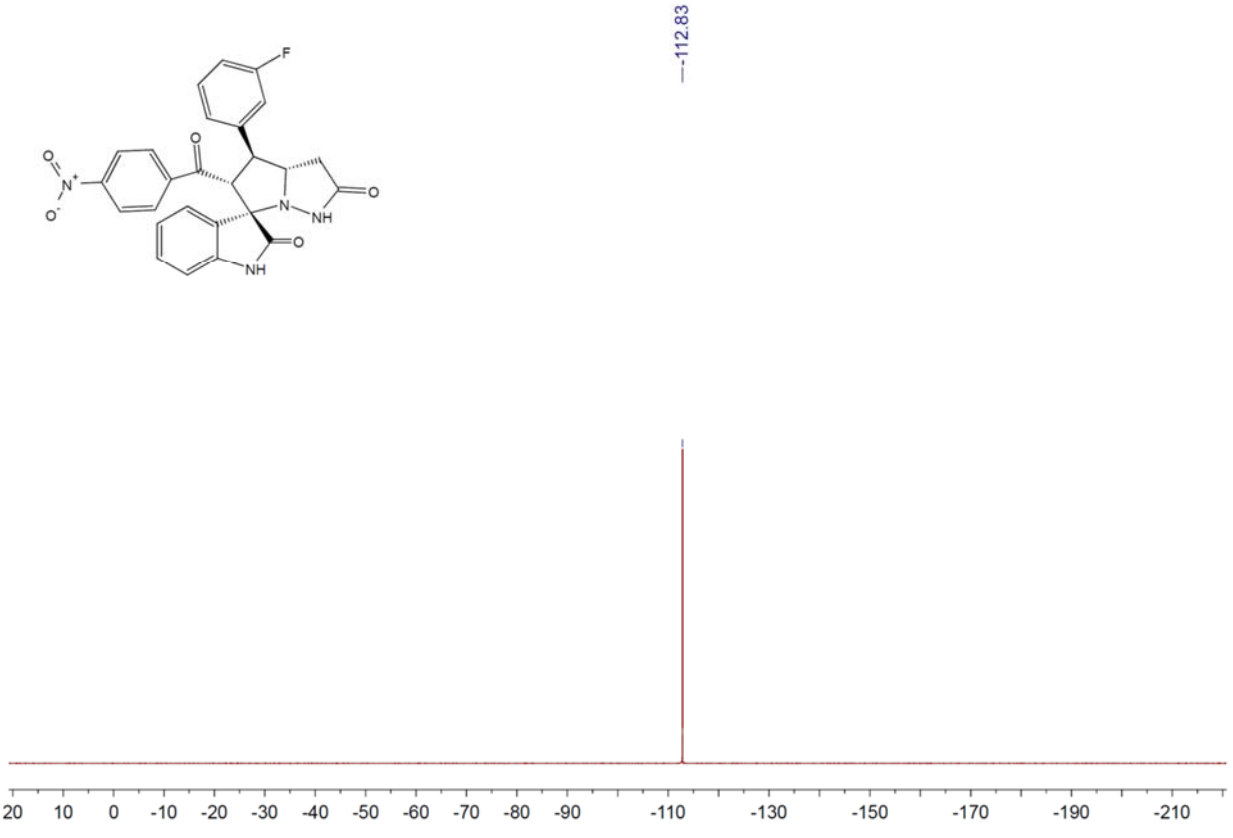
37.04
F
O
N
NH
O
NH
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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NH
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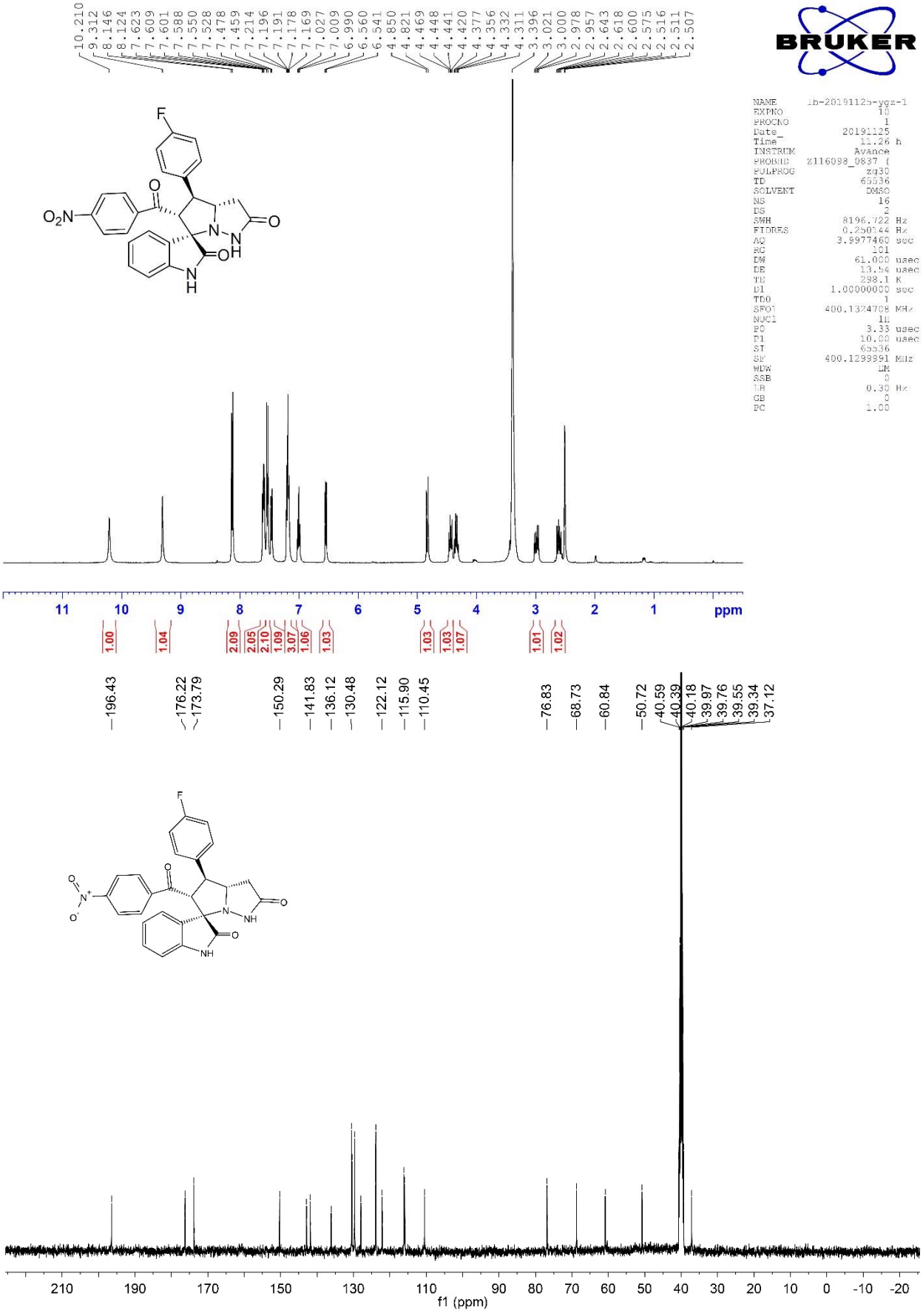
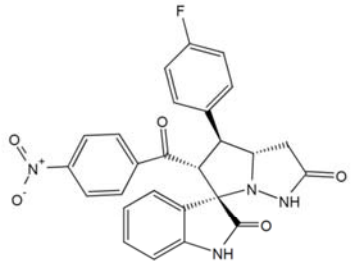
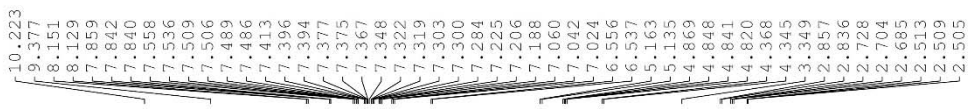
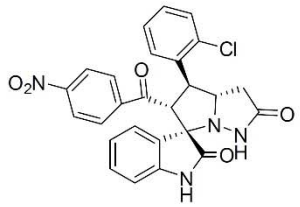
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3g**

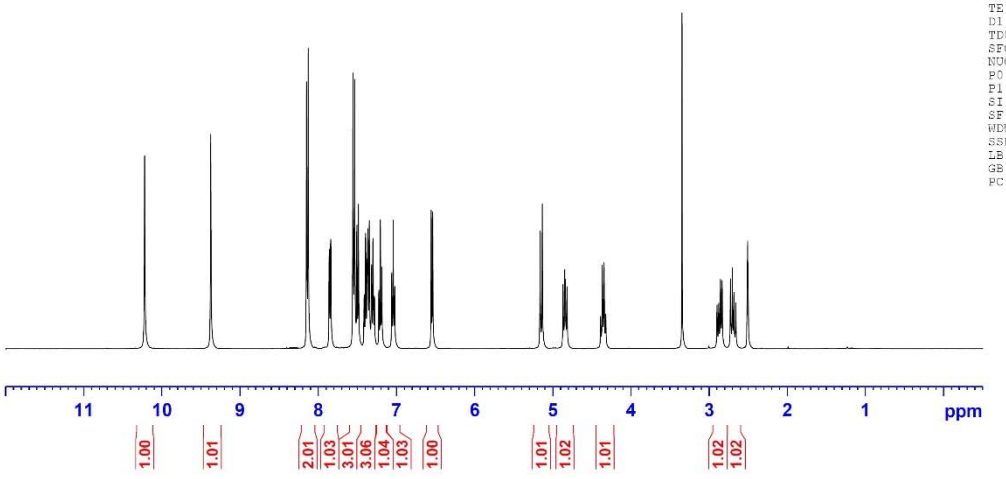




^{1}H NMR and ^{13}C NMR Spectra for Compound **3h**





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FIDRES             0.250144 Hz
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RG                       101
DW                    61.000 usec
DE                    13.54 usec
TE                    298.2 K
D1                 1.00000000 sec
TD0                          1
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NUC1                        1H
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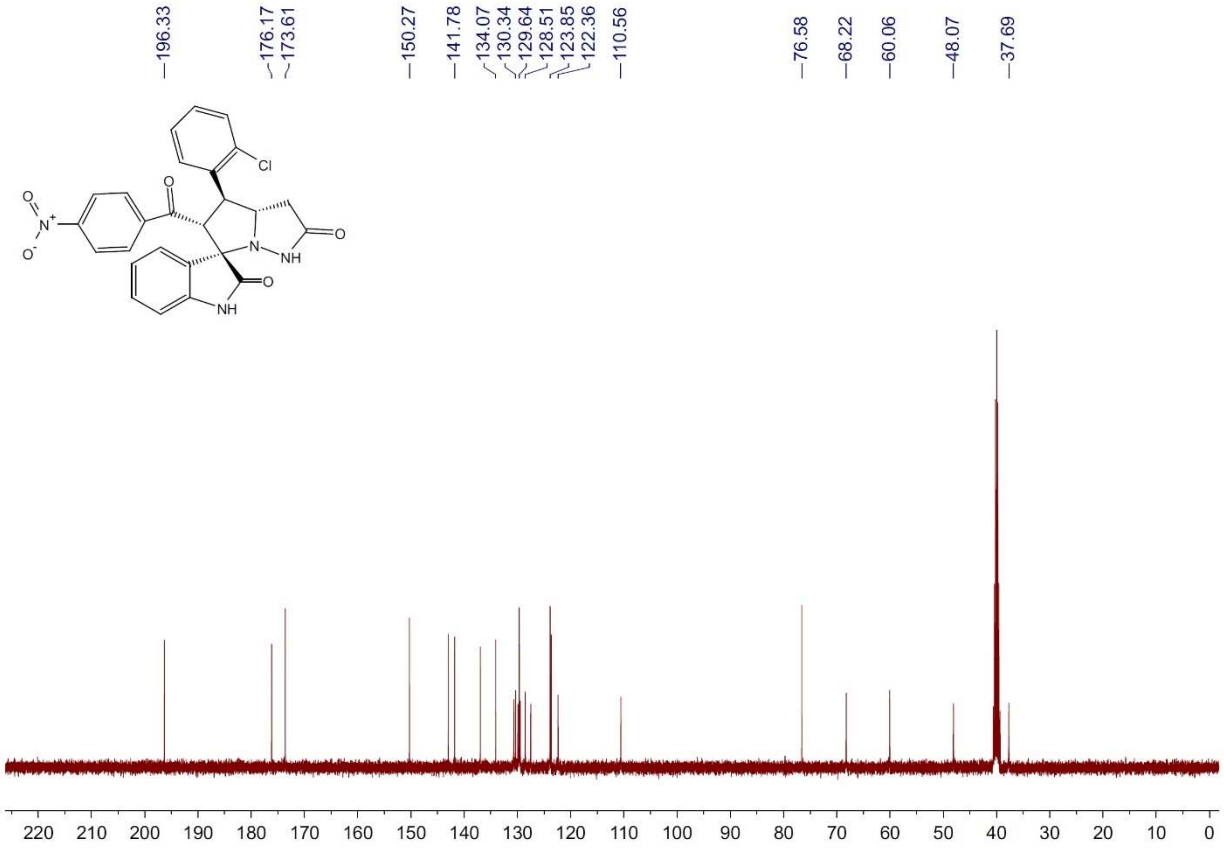
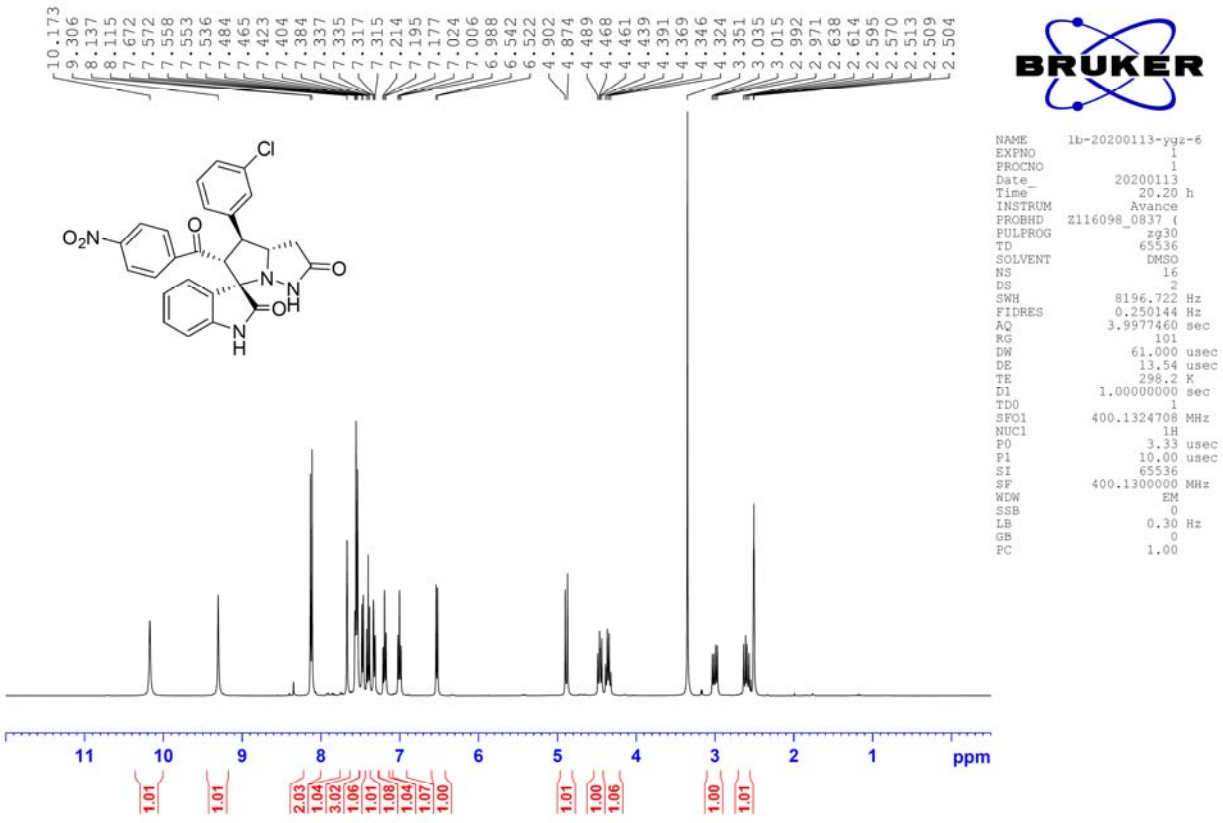
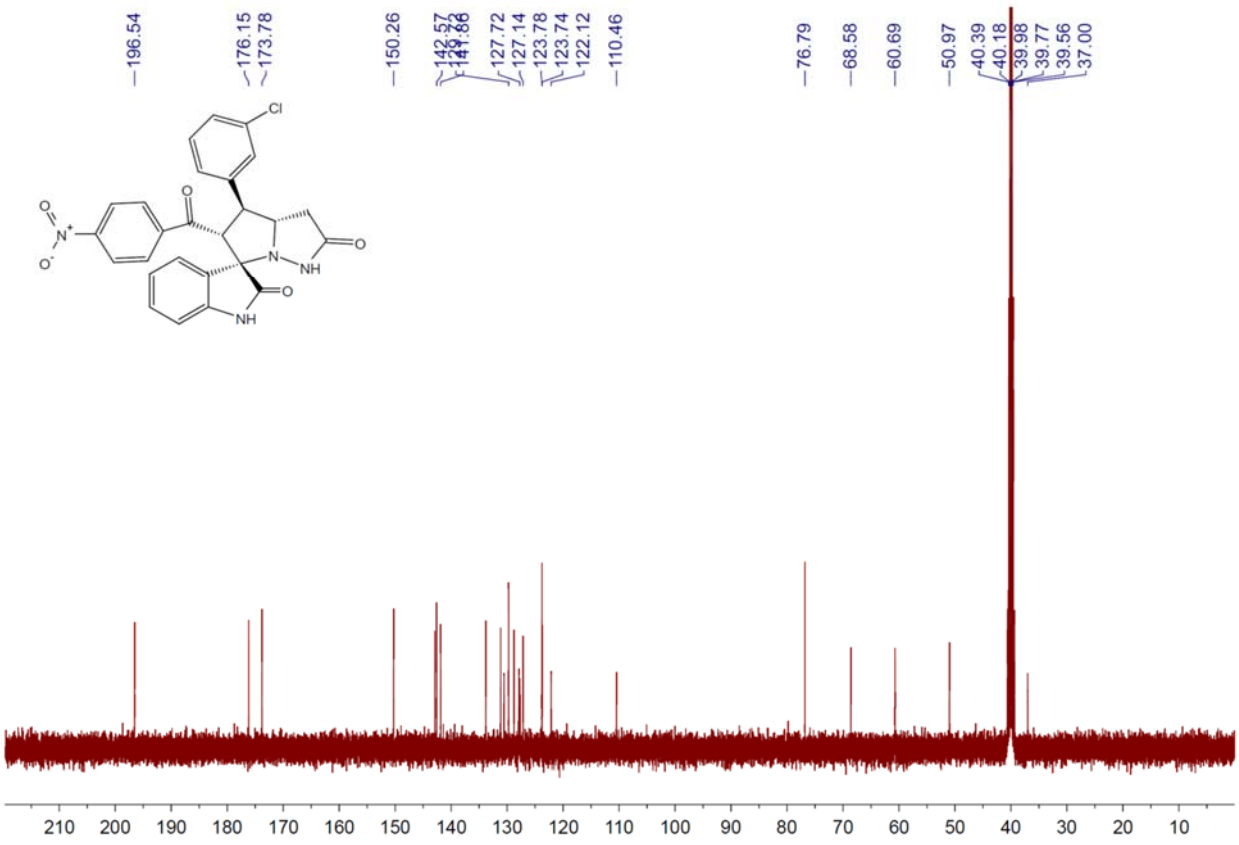
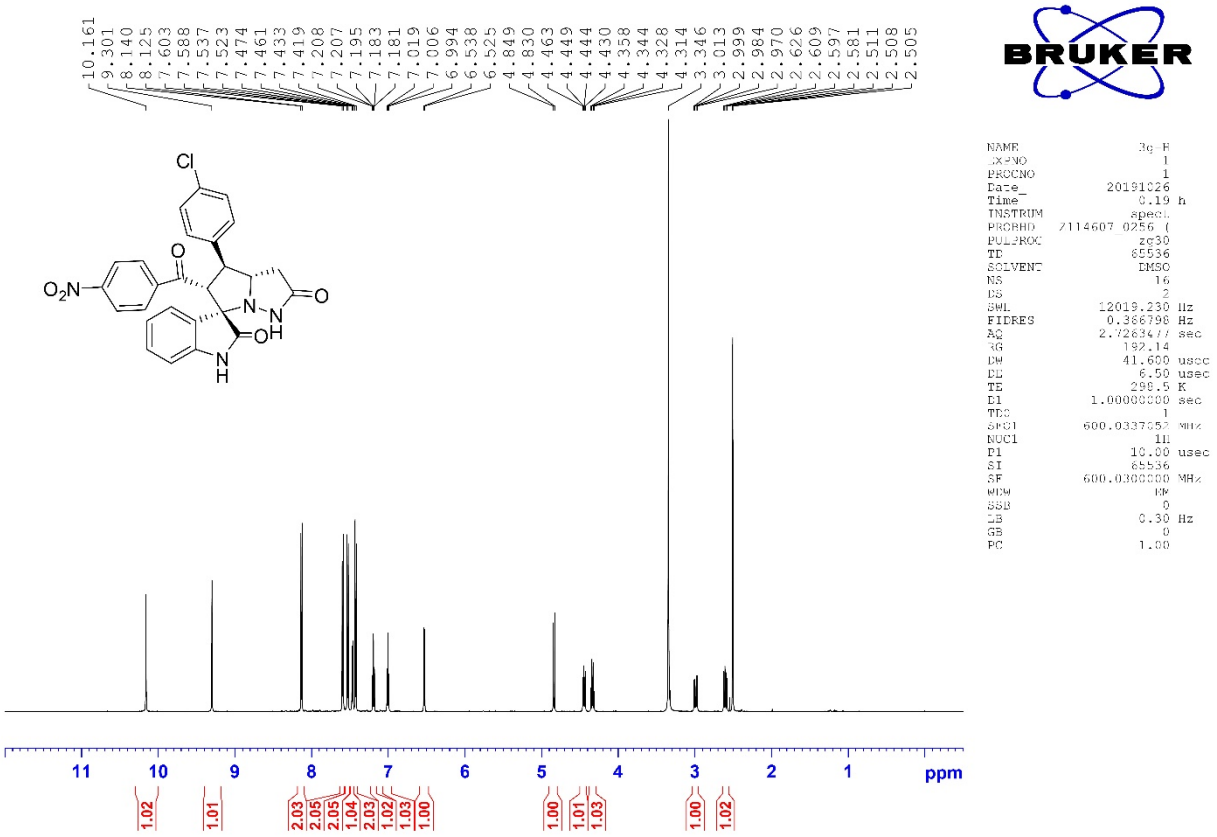


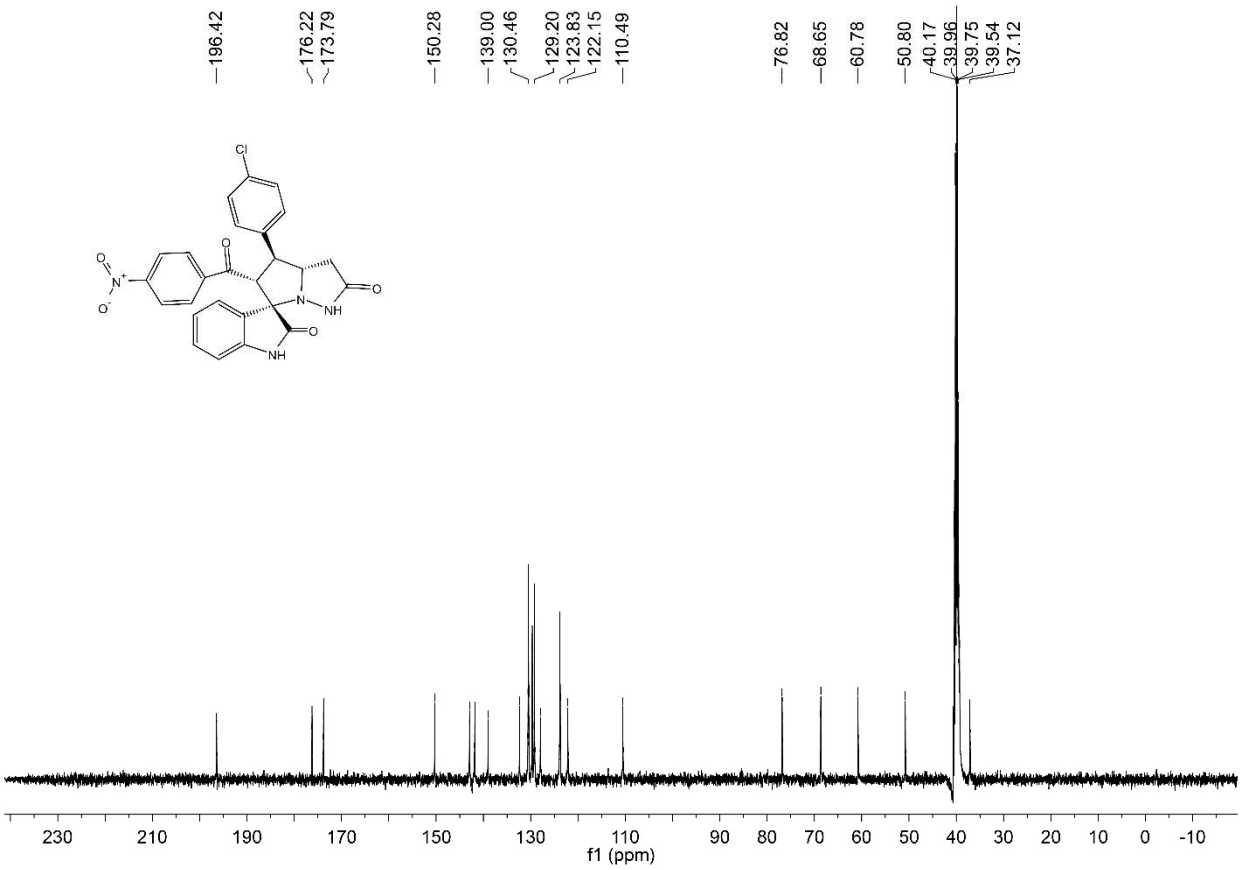
^{1}H NMR and ^{13}C NMR Spectra for Compound **3i**



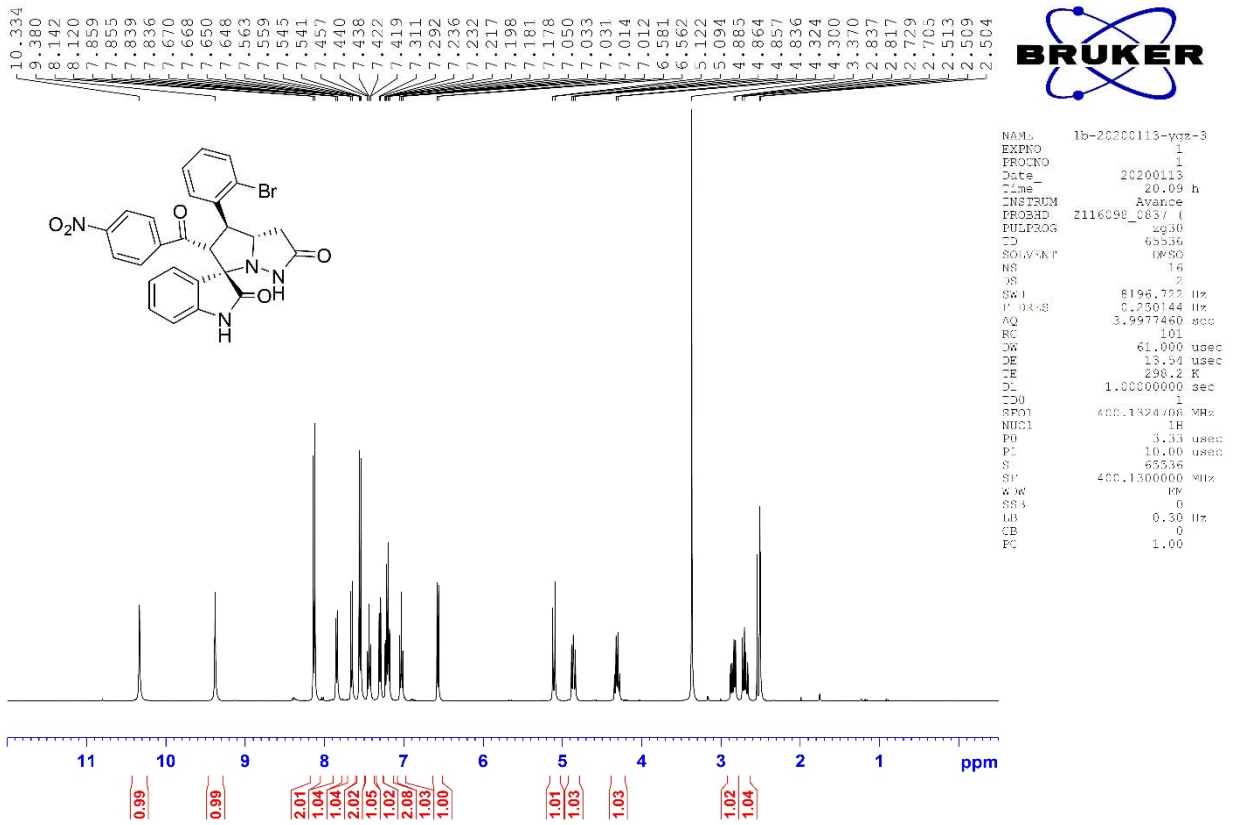


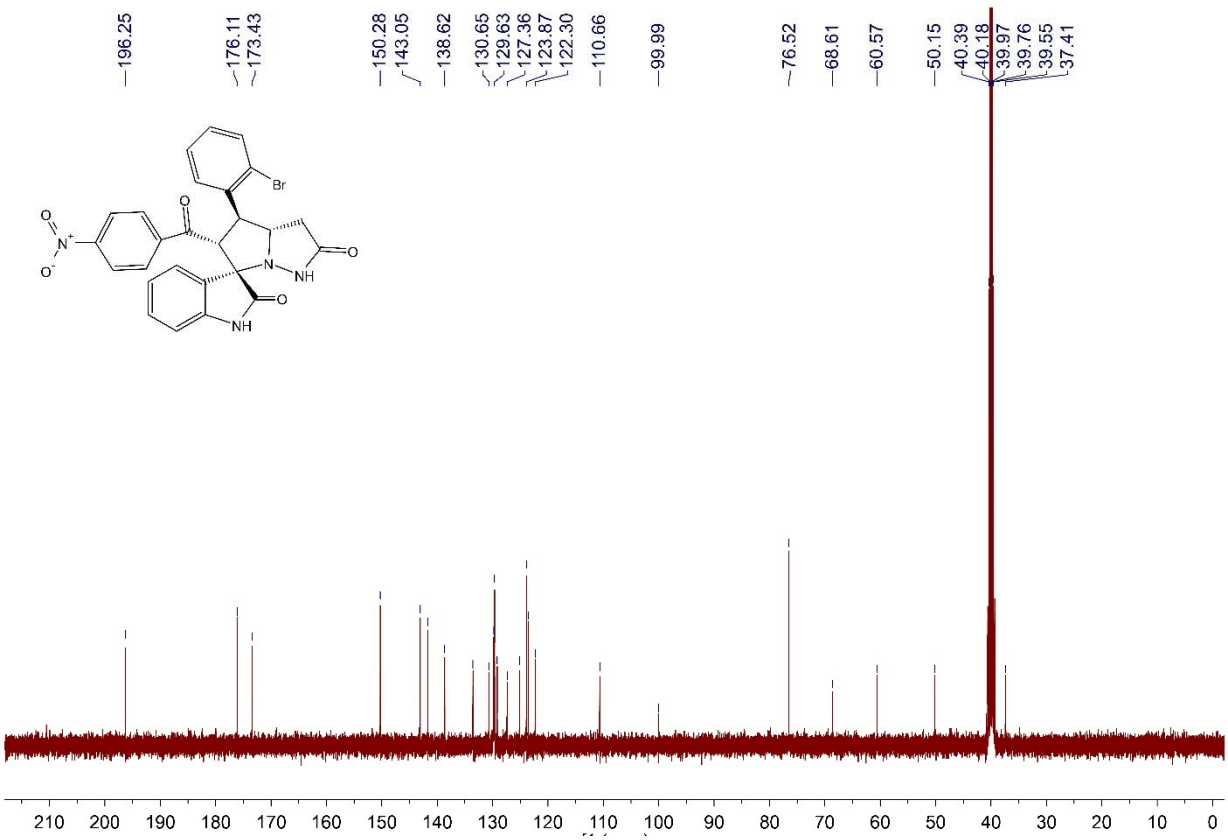
^{1}H NMR and ^{13}C NMR Spectra for Compound **3j**



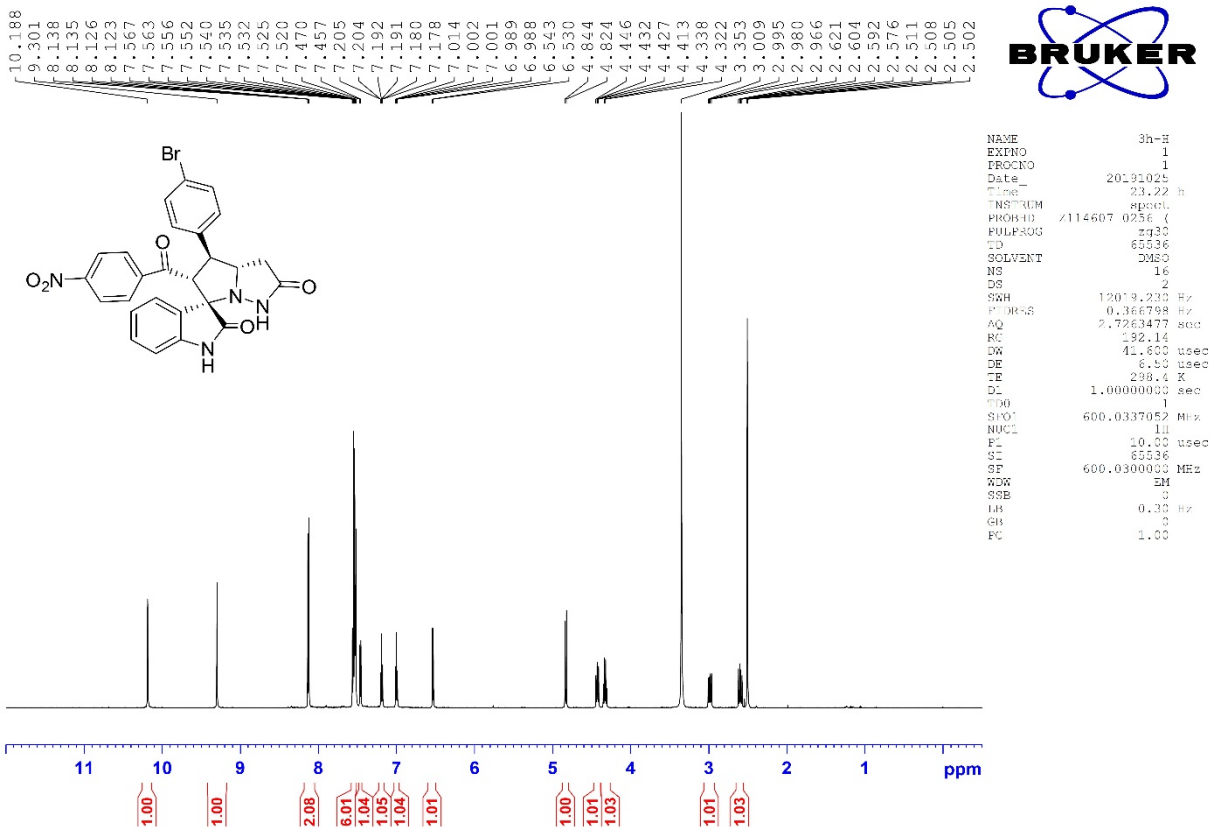


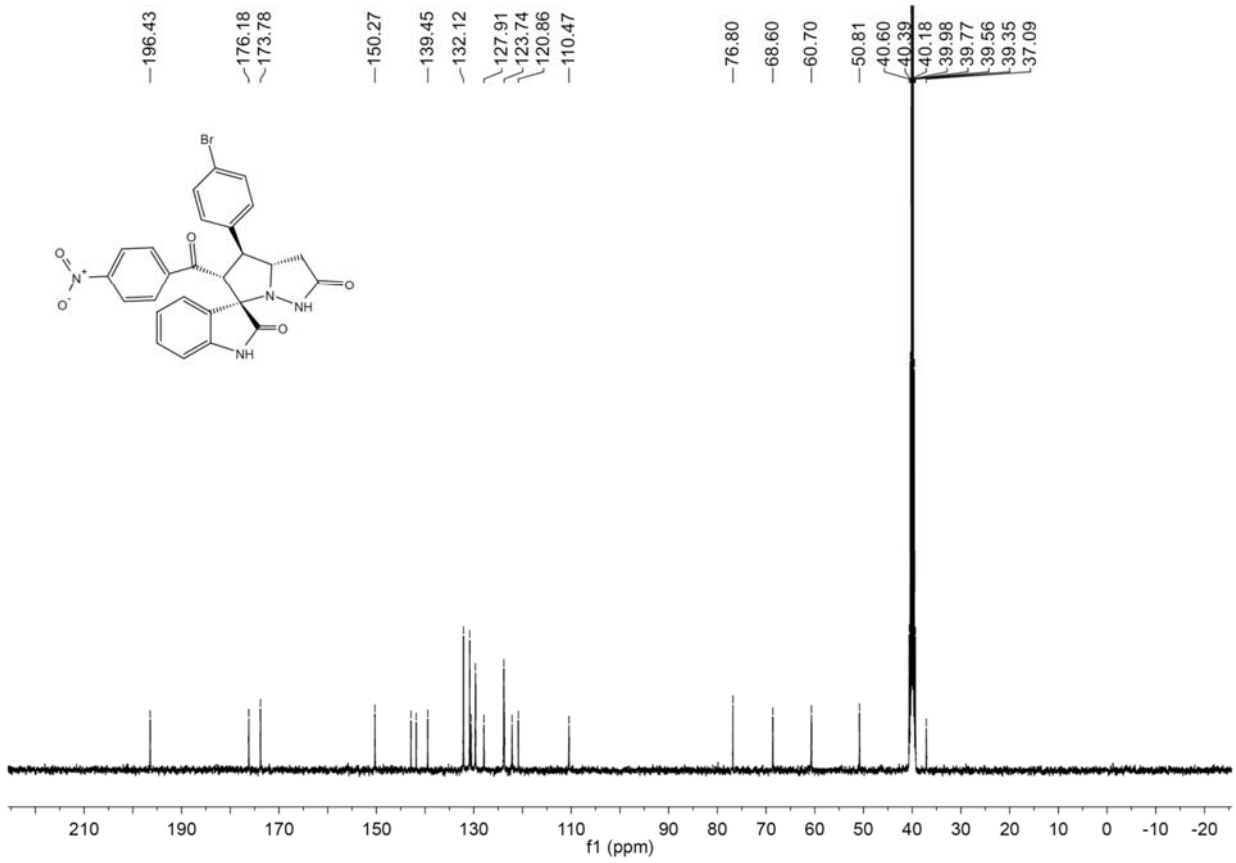
^{1}H NMR and ^{13}C NMR Spectra for Compound **3k**



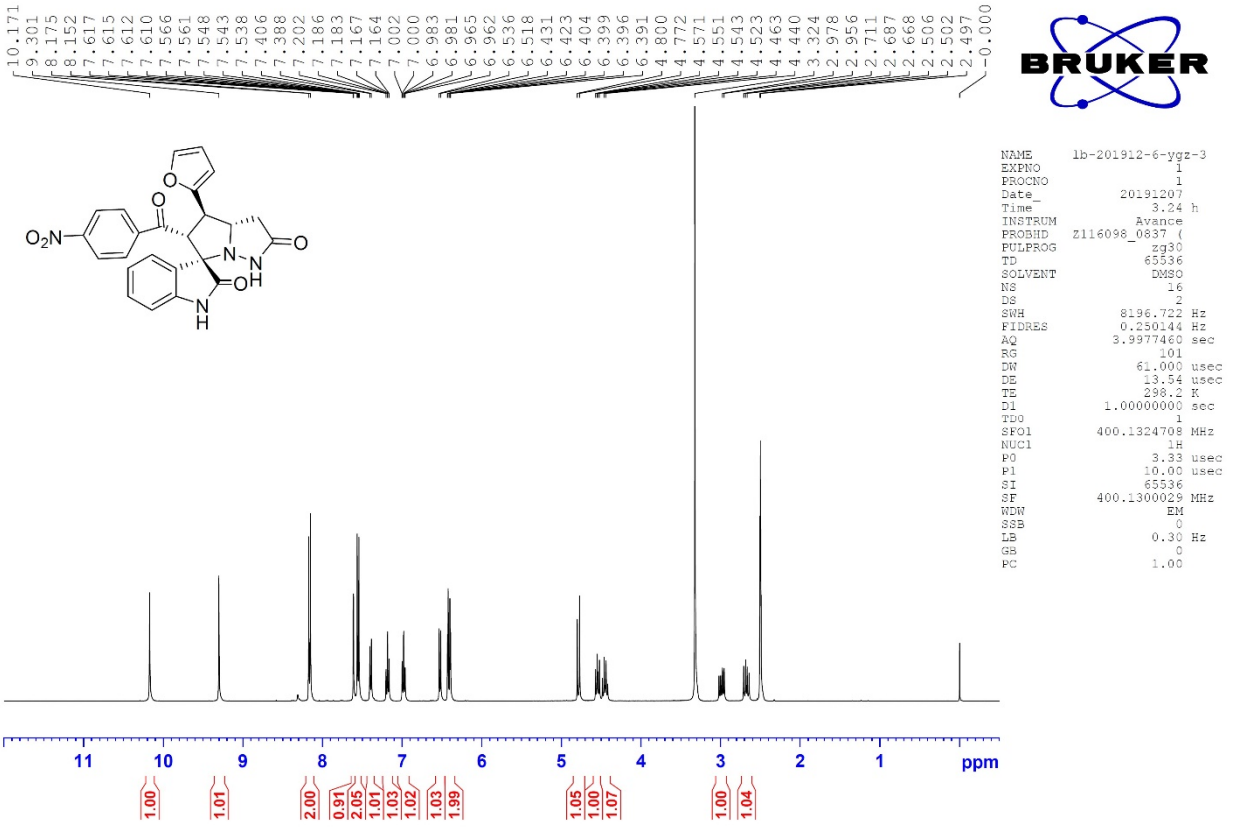


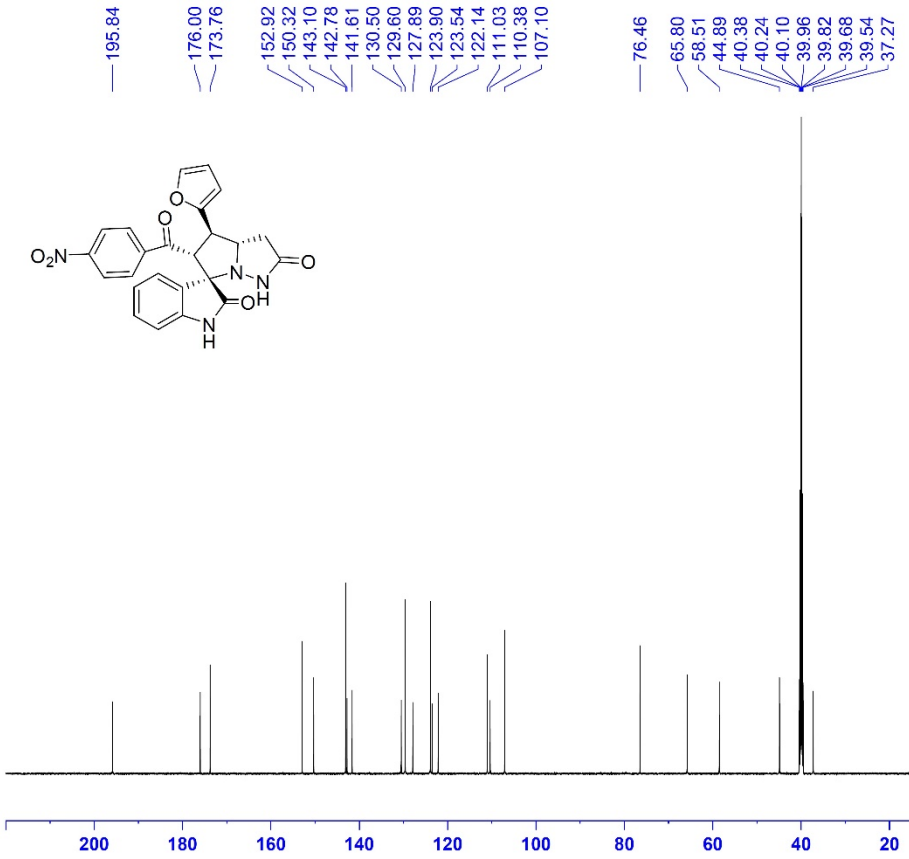
^{1}H NMR and ^{13}C NMR Spectra for Compound **3l**





^{1}H NMR and ^{13}C NMR Spectra for Compound **3m**





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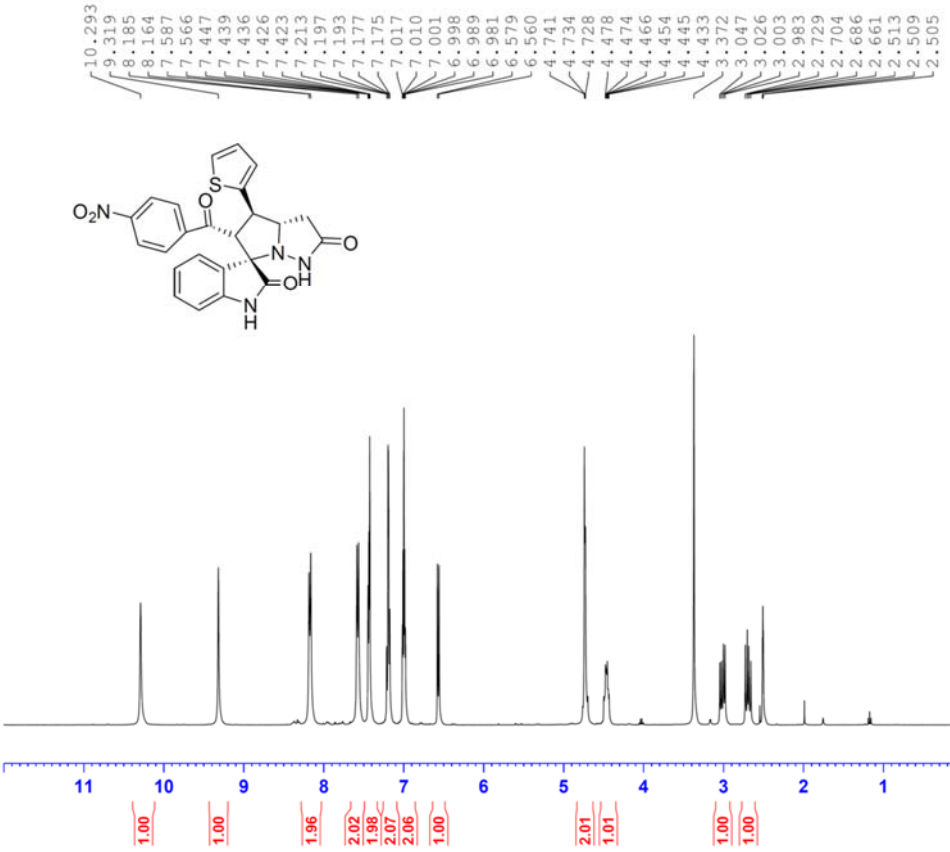
NAME                3i-C
EXPNO                  1
PROCNO                 1
Date_           20191218
Time_              12.41 h
INSTRUM             spect
PROBHD    Z11460/ 0256 (
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                    1024
DS                       4
SWH             36231.883 Hz
FIDRES          1.105709 Hz
AQ              0.9044468 sec
RG                  192.14
DW                  13.800 usec
DE                    6.50 usec
TE                  297.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1
SFO1          150.8927513 MHz
NUC1                  13C
P1                  12.00 usec
SI                  32768
SF            150.8776635 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

^{1}H NMR and ^{13}C NMR Spectra for Compound **3n**



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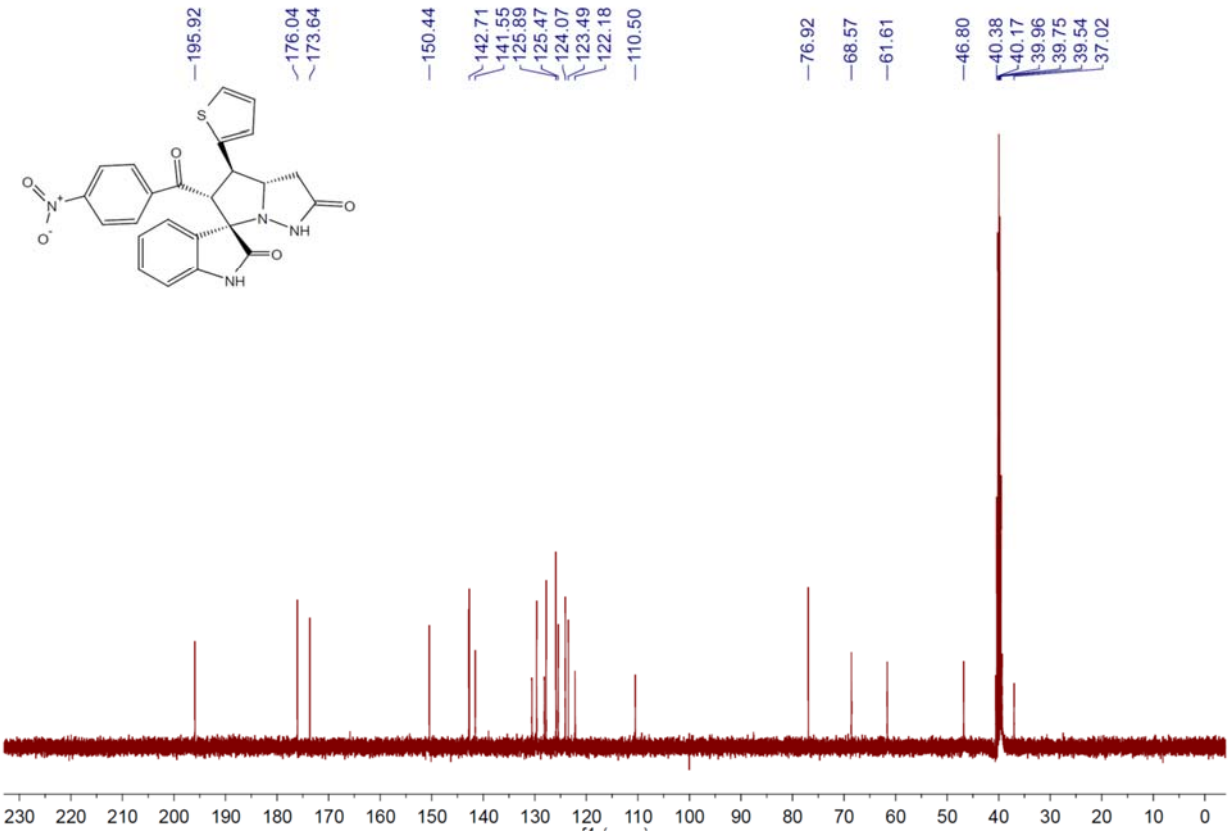
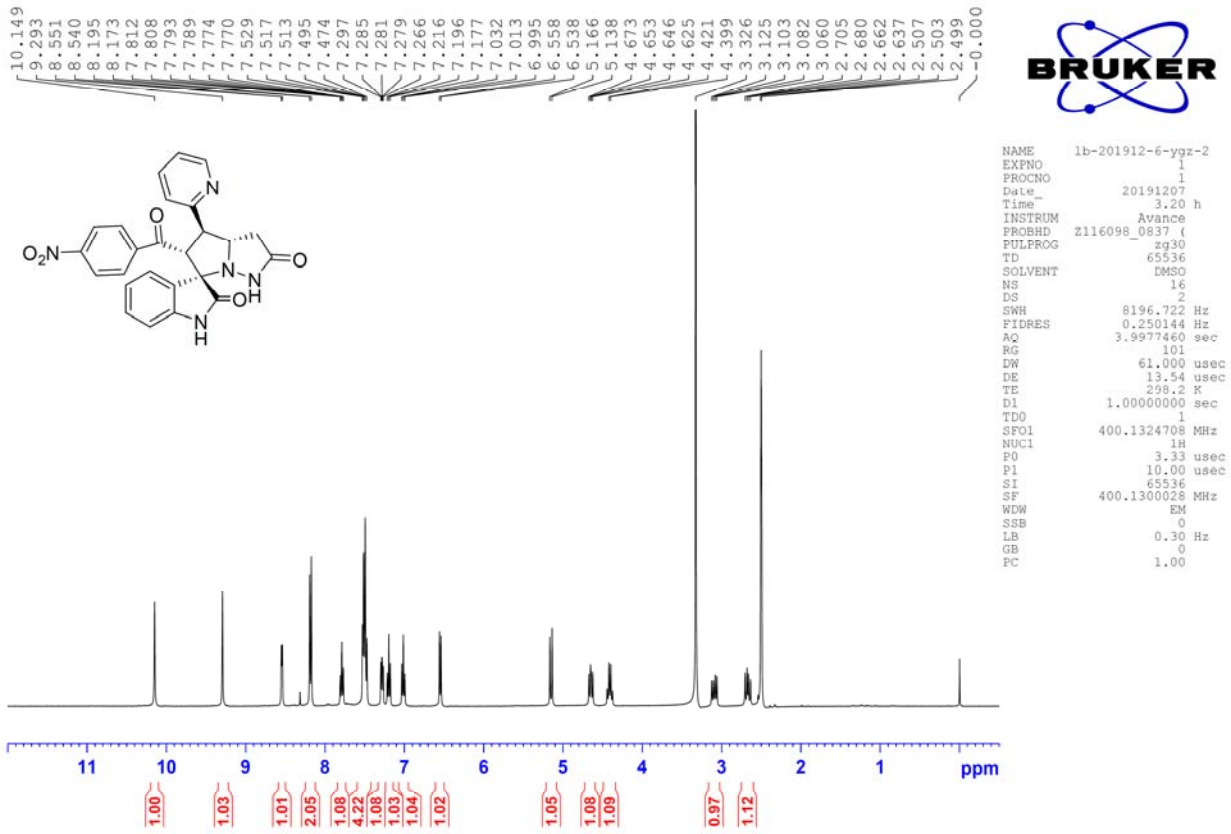
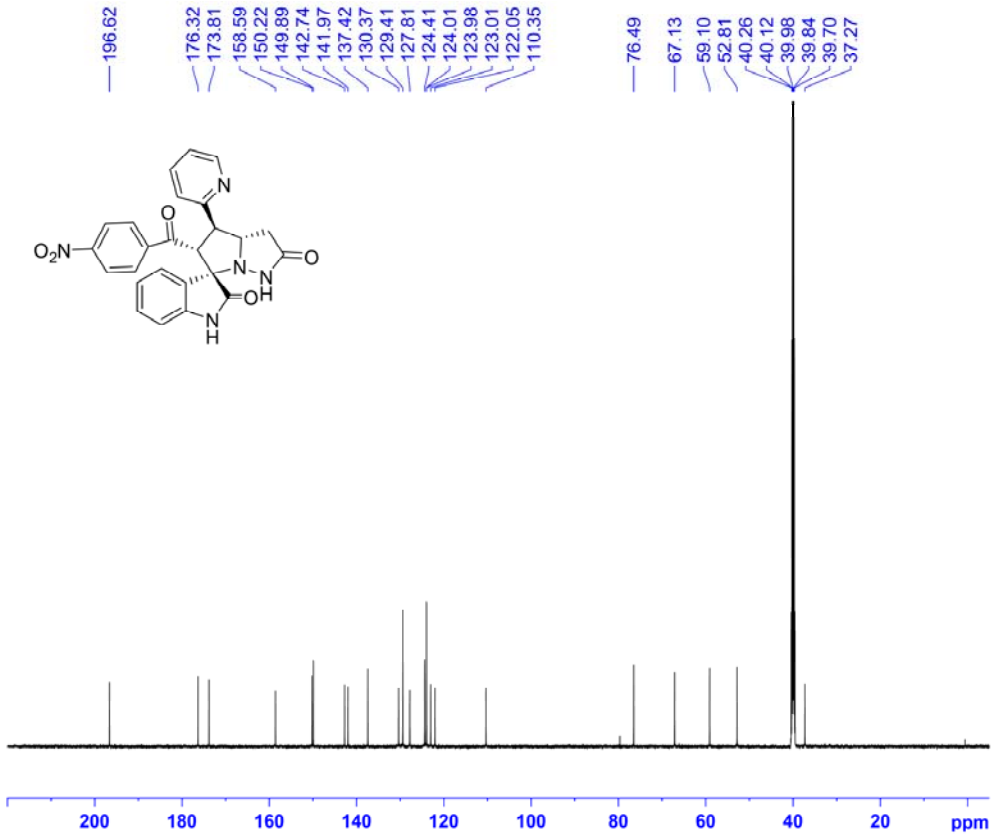
NAME          1b-20200113-ygz-4
EXPNO                  1
PROCNO                 1
Date_           20200113
Time_              20.12 h
INSTRUM            Avance
PROBHD    Z116098_0837 (
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                      16
DS                       2
SWH              8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9977460 sec
RG                     101
DW                  61.000 usec
DE                   13.54 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                   1H
P0                   3.33 usec
P1                  10.00 usec
SI                  65536
SF            400.1300000 MHz
WDW                   EM
SSB                    0
LB                   0.30 Hz
GB                     0
PC                   1.00
```



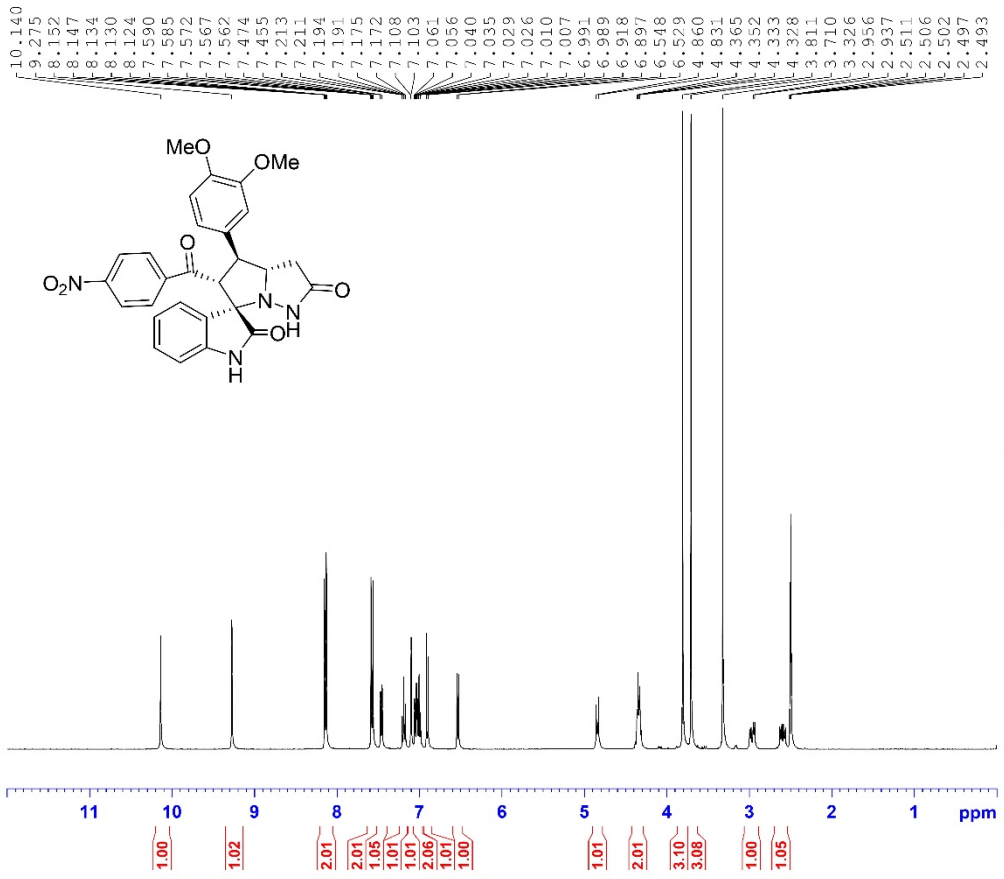
^{1}H NMR and ^{13}C NMR Spectra for Compound **3o**



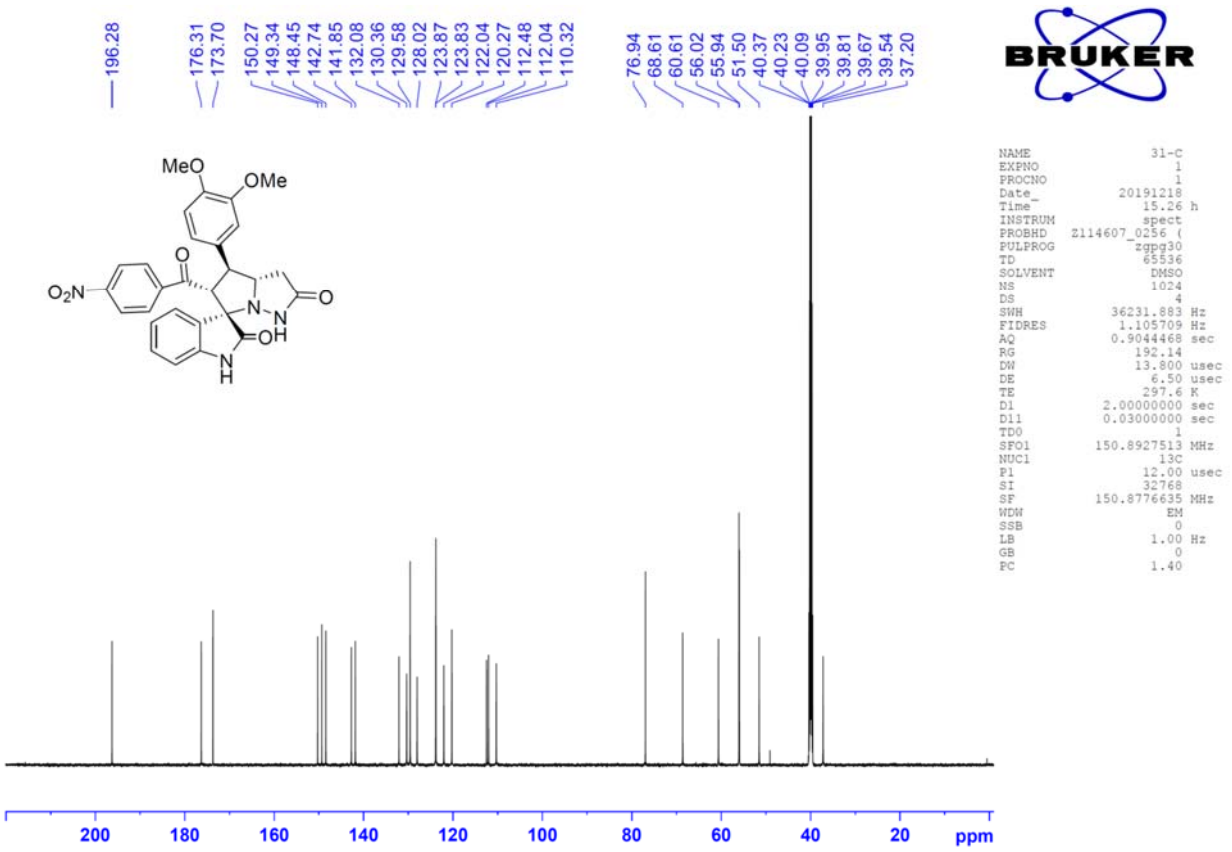


```
NAME                3j-C
EXPNO                  1
PROCNO                 1
Date_           20191218
Time                13.35 h
INSTRUM             spect
PROBHD    Z114607_0256 (
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                  1024
DS                     4
SWH          36231.883 Hz
FIDRES        1.105709 Hz
AQ            0.9044468 sec
RG               192.14
DW               13.800 usec
DE                6.50 usec
TE                297.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1        150.8927513 MHz
NUC1                13C
P1                12.00 usec
SI                32768
SF          150.8776635 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

^{1}H NMR and ^{13}C NMR Spectra for Compound **3p**

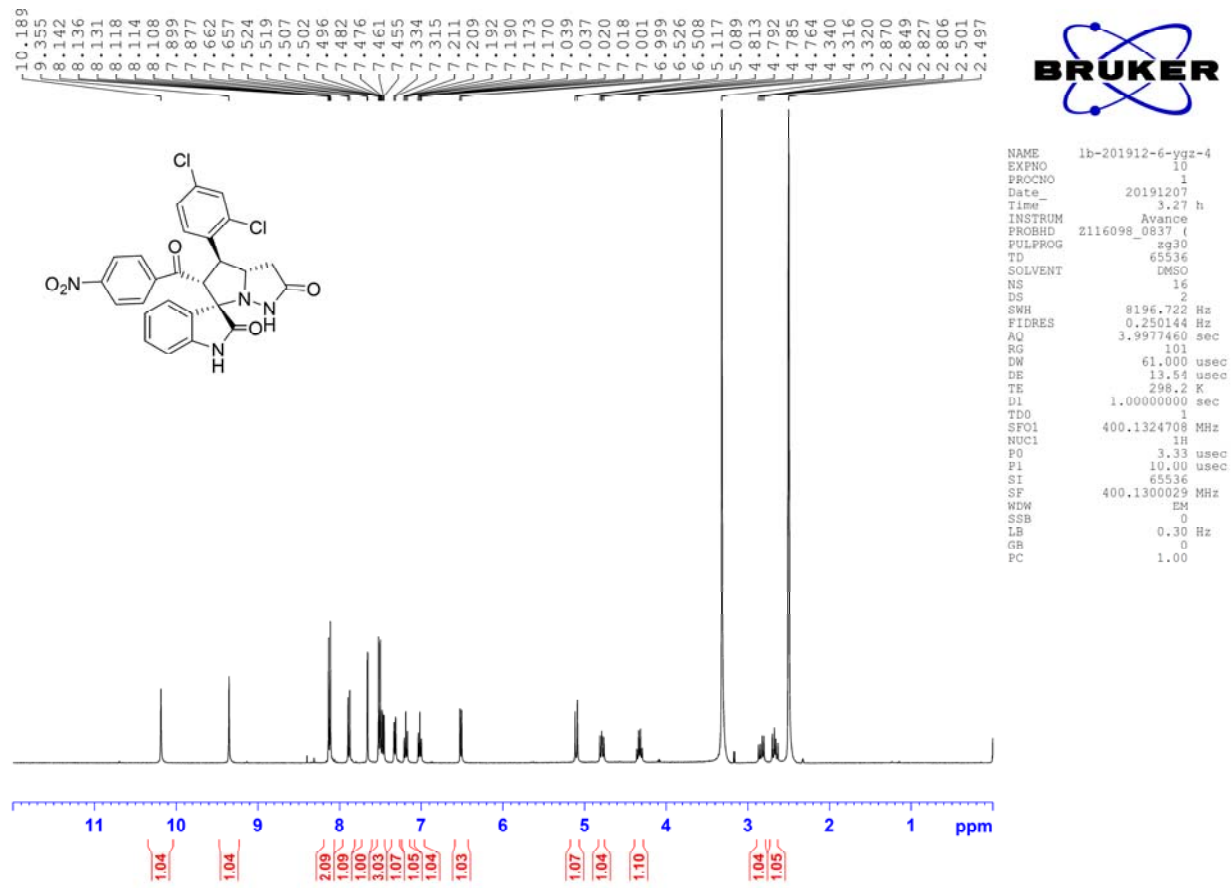


```
NAME          lb-201912-6-yqz-5
EXPNO                        10
PROCNO                        1
Date_                 20191207
Time                      3.31 h
INSTRUM                 Avance
PROBHD    Z116098_0837 (
PULPROG                   zg30
TD                       65536
SOLVENT                   DMSO
NS                          16
DS                           2
SWH                 8196.722 Hz
FIDRES              0.250144 Hz
AQ                  3.9977460 sec
RG                         181
DW                      61.000 usec
DE                      13.54 usec
TE                      298.2 K
D1                 1.00000000 sec
TD0                          1
SFO1               400.1324709 MHz
NUC1                        1H
PS                       3.33 usec
P1                      10.00 usec
SI                       65536
SF                 400.1300029 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                      1.00
```

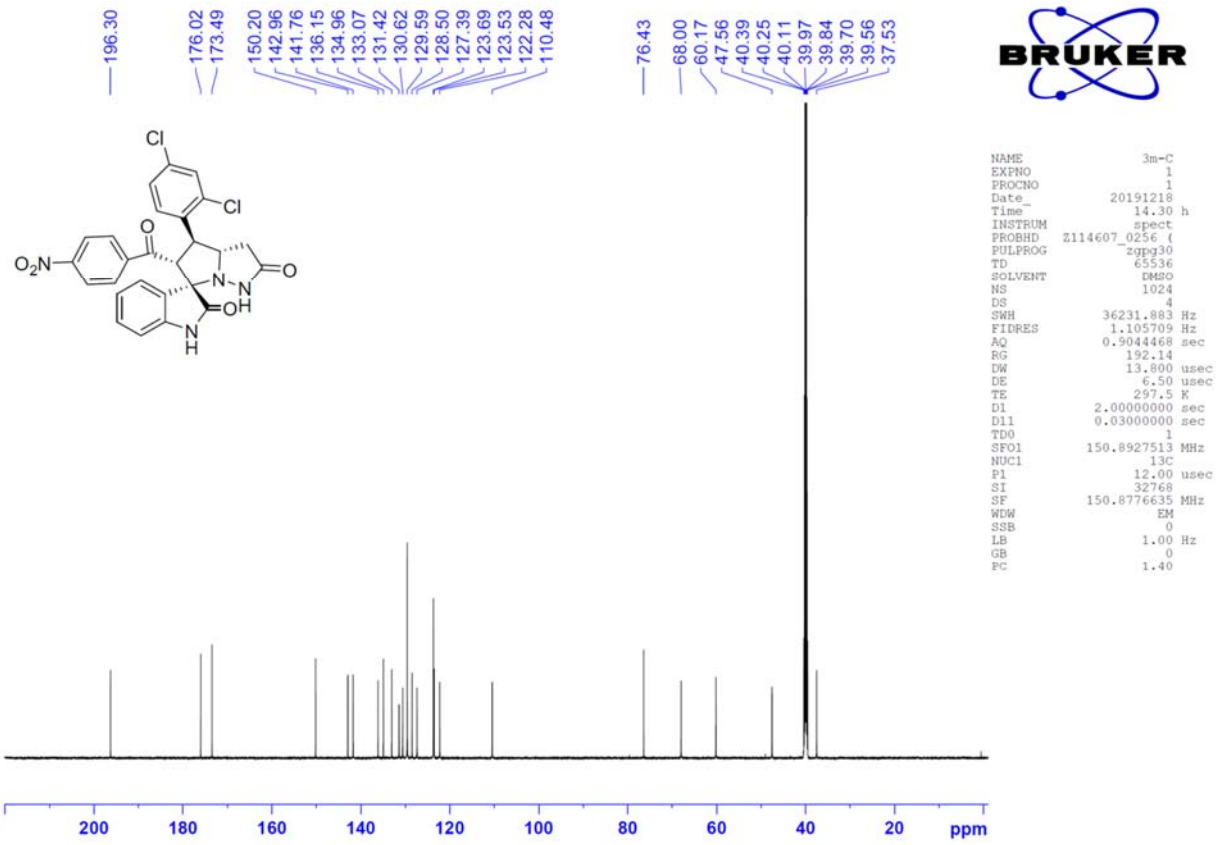


NAME 31-C
EXPNO 1
PROCNO 1
Date_ 20191218
Time_ 15.26 h
INSTRUM spect
PROBHD Z114607_0256 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 192.14
DW 13.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.8927513 MHz
NUC1 13C
P1 12.00 usec
SI 32768
SF 150.8776635 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

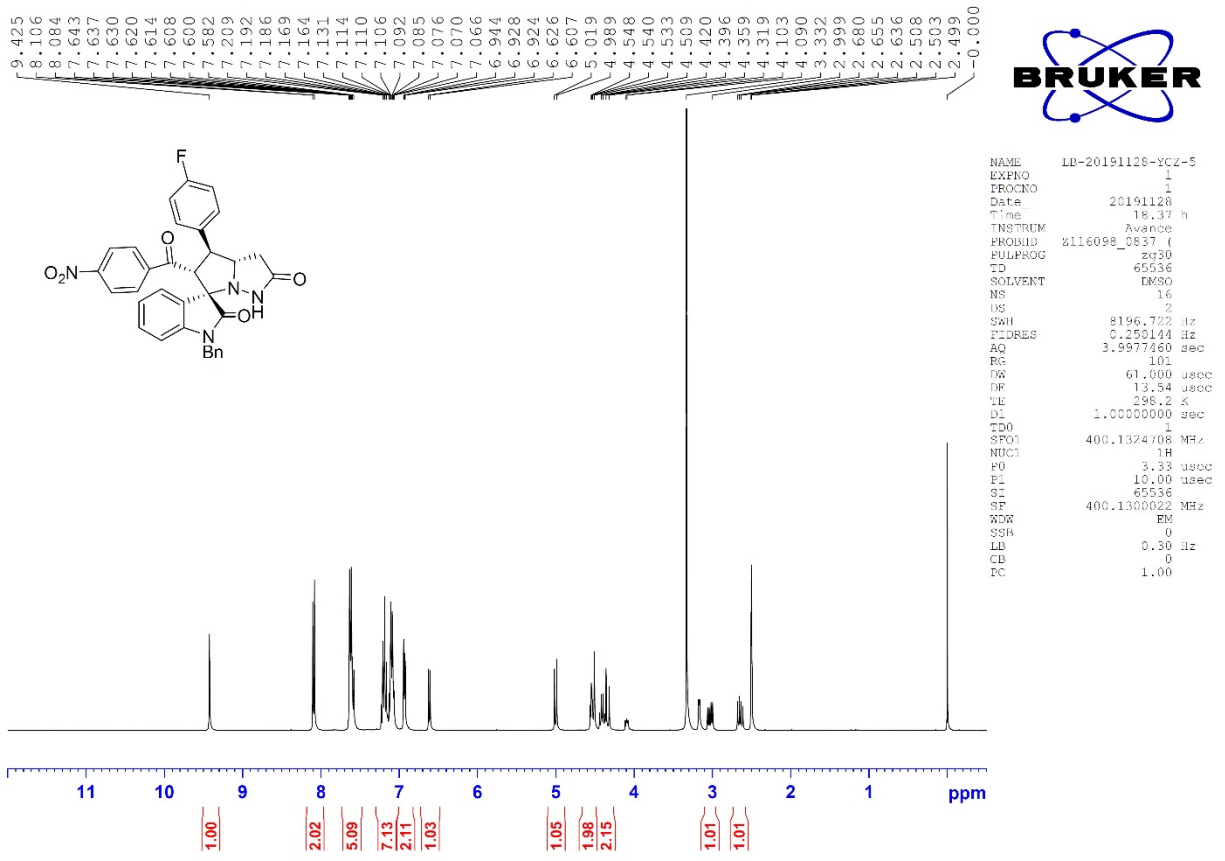
^{1}H NMR and ^{13}C NMR Spectra for Compound **3q**

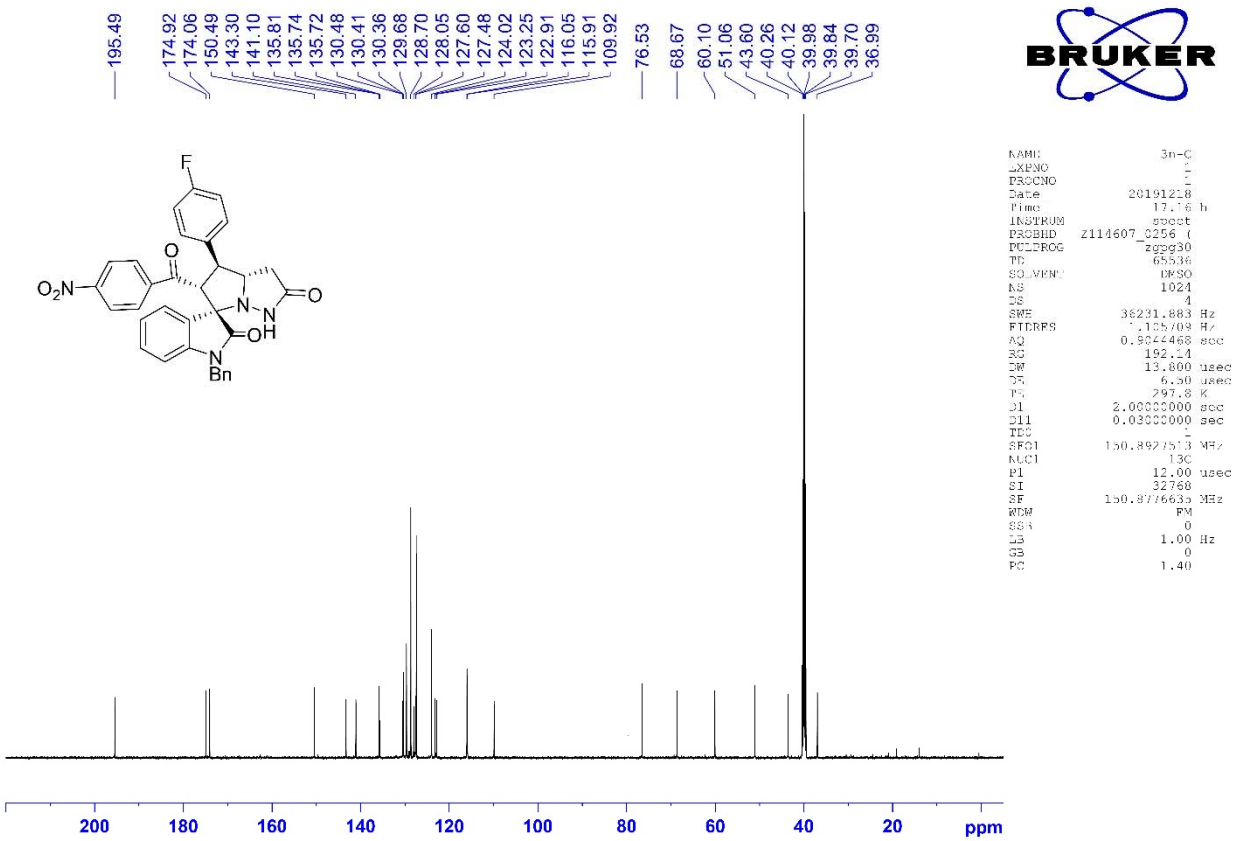


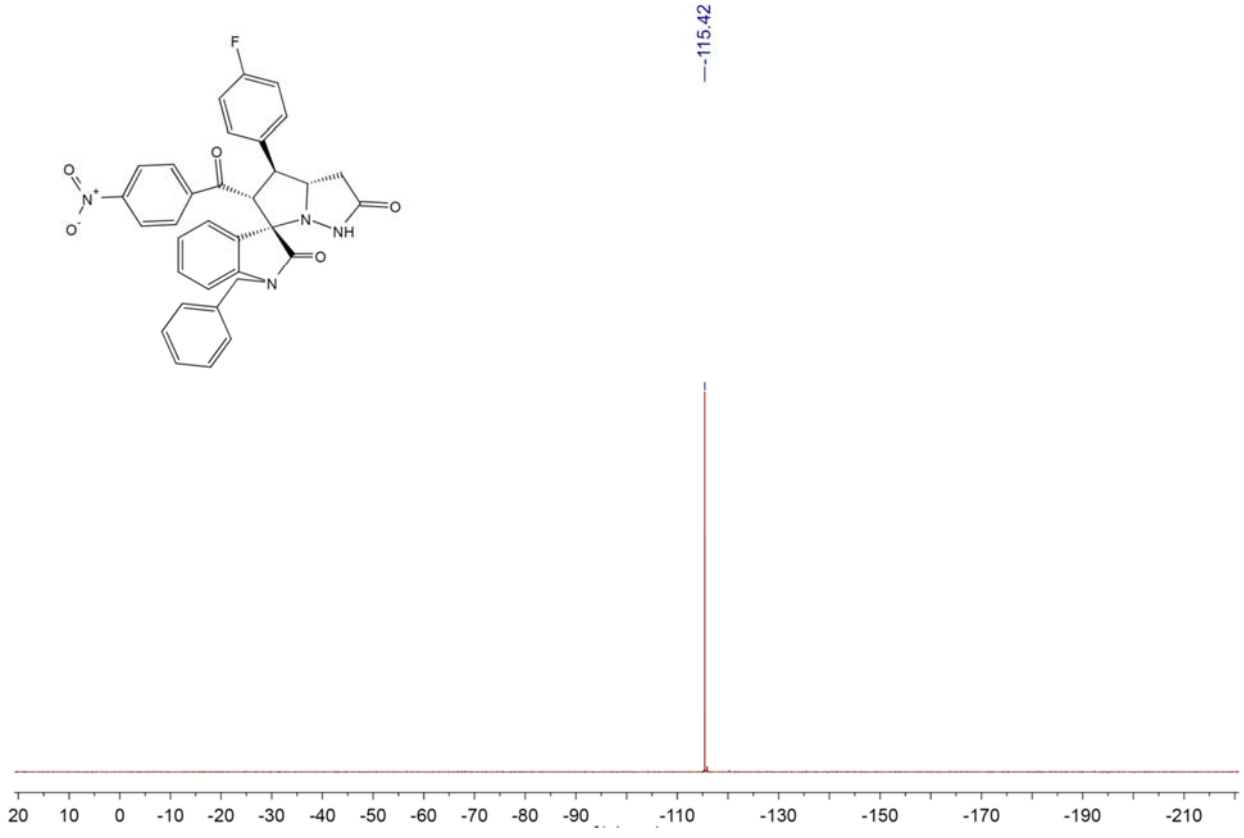
NAME 1b-201912-6-ygz-4
EXPNO 10
PROCNO 1
Date_ 20191207
Time_ 3.27 h
INSTRUM Avance
PROBHD Z116098_0837 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8196.722 Hz
FIDRES 0.250144 Hz
AQ 3.9977460 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
SI 65536
SF 400.1300029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



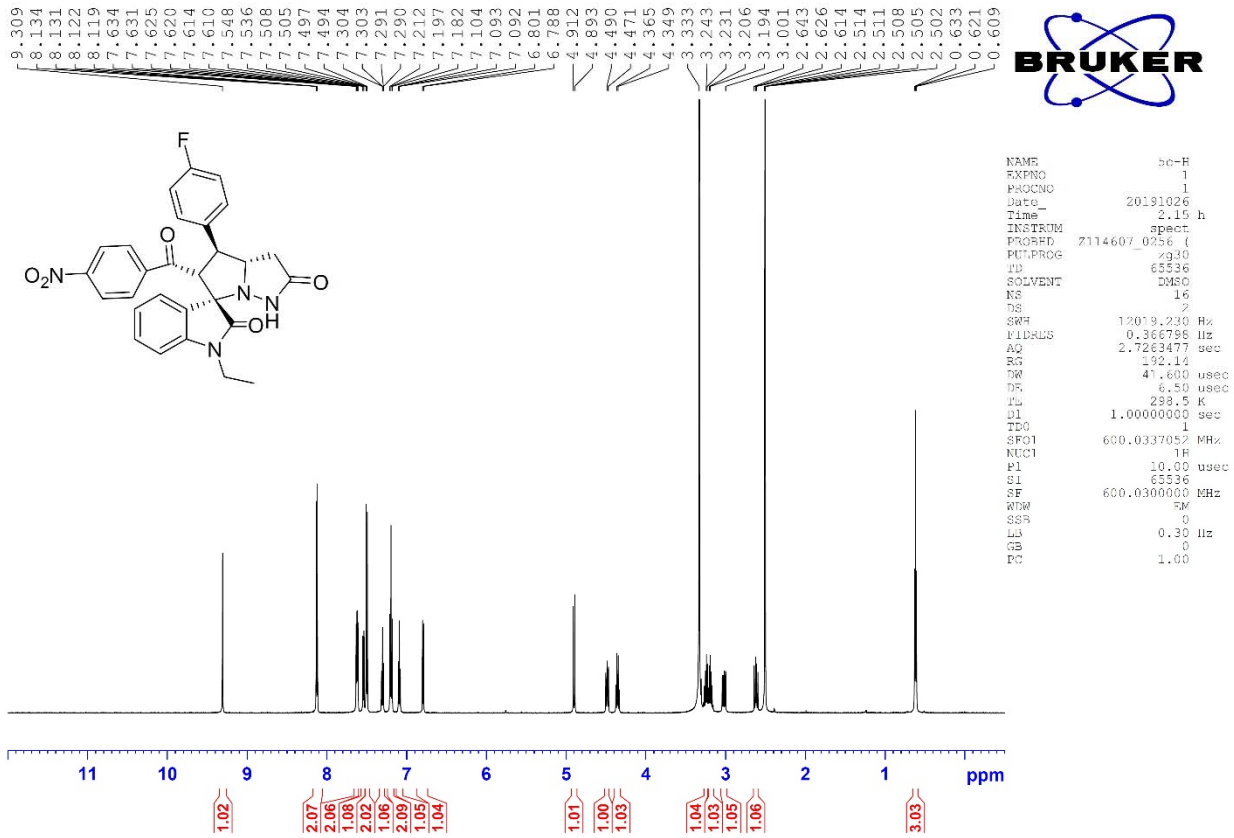
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3r**

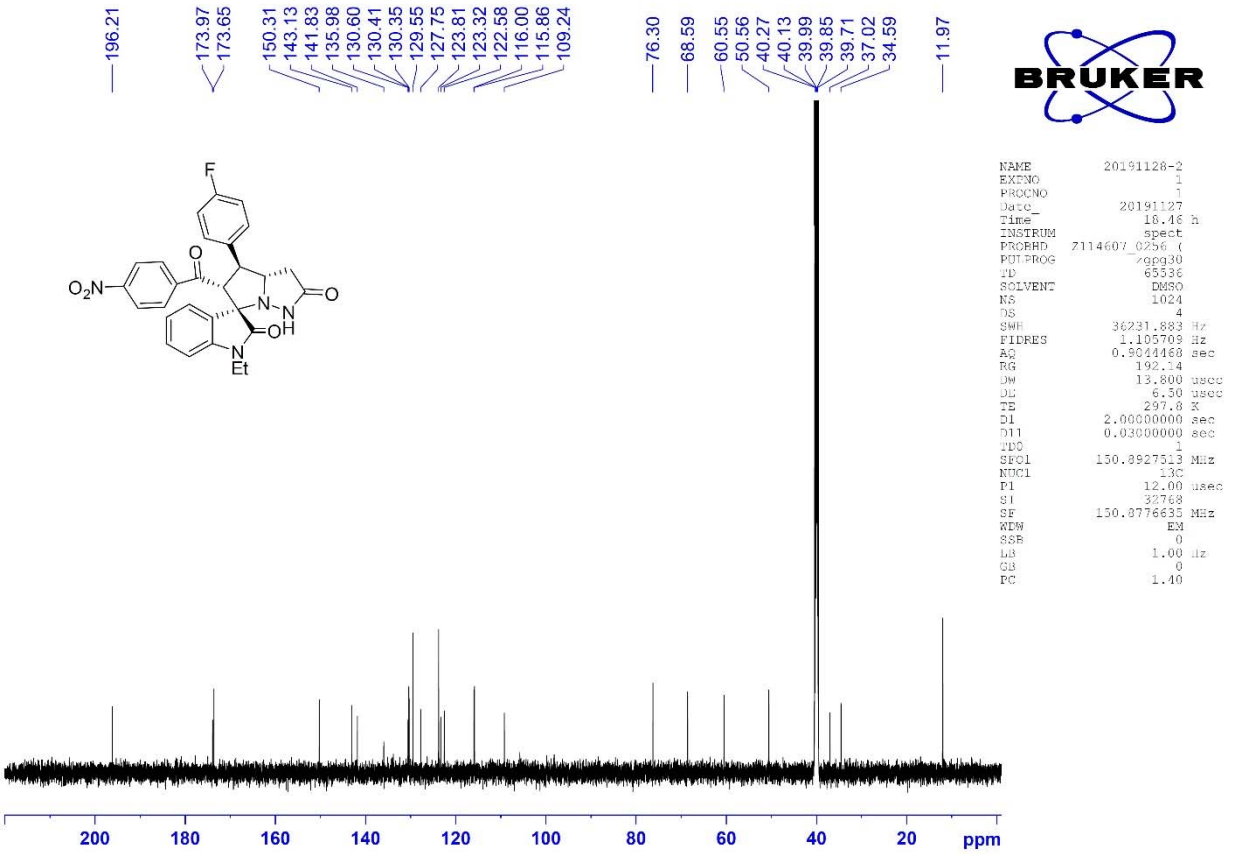


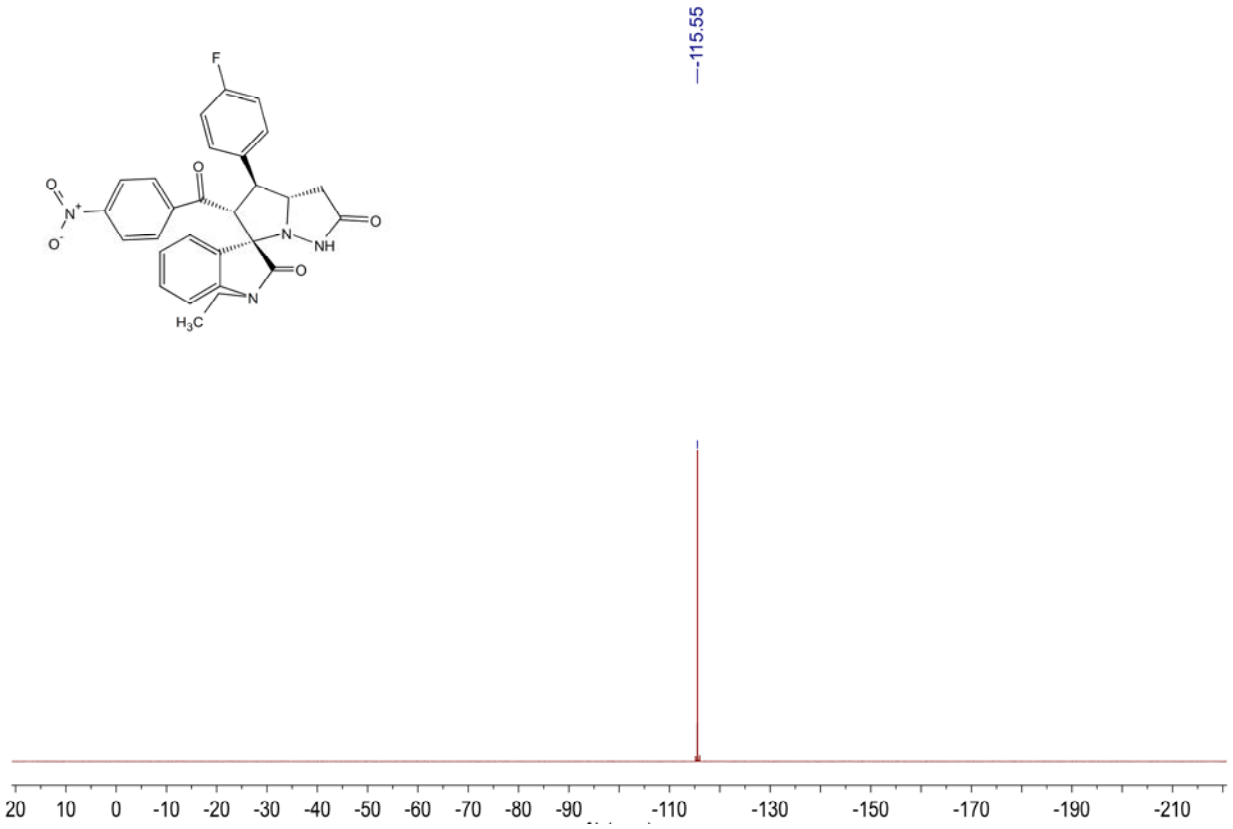

195.49
174.92
174.06
150.49
143.30
141.10
135.81
135.74
135.72
130.48
130.41
130.36
129.68
128.70
128.05
127.60
127.48
124.02
123.25
122.91
116.05
115.91
109.92
76.53
68.67
60.10
51.06
43.60
40.26
40.12
39.98
39.84
39.70
36.99
BRUKER
NAME 3n-C
EXPNO 1
PROCNO 1
Date 20191218
Time 17.16 h
INSTRUM spect
PROBHD Z114607_0256 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 36231.883 Hz
FIDRES 0.552709 Hz
AQ 0.9022468 sec
RG 192.24
DW 13.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.8927513 MHz
NUC1 13C
P1 12.00 usec
SI 32768
SF 150.8778633 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm


115.42
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210

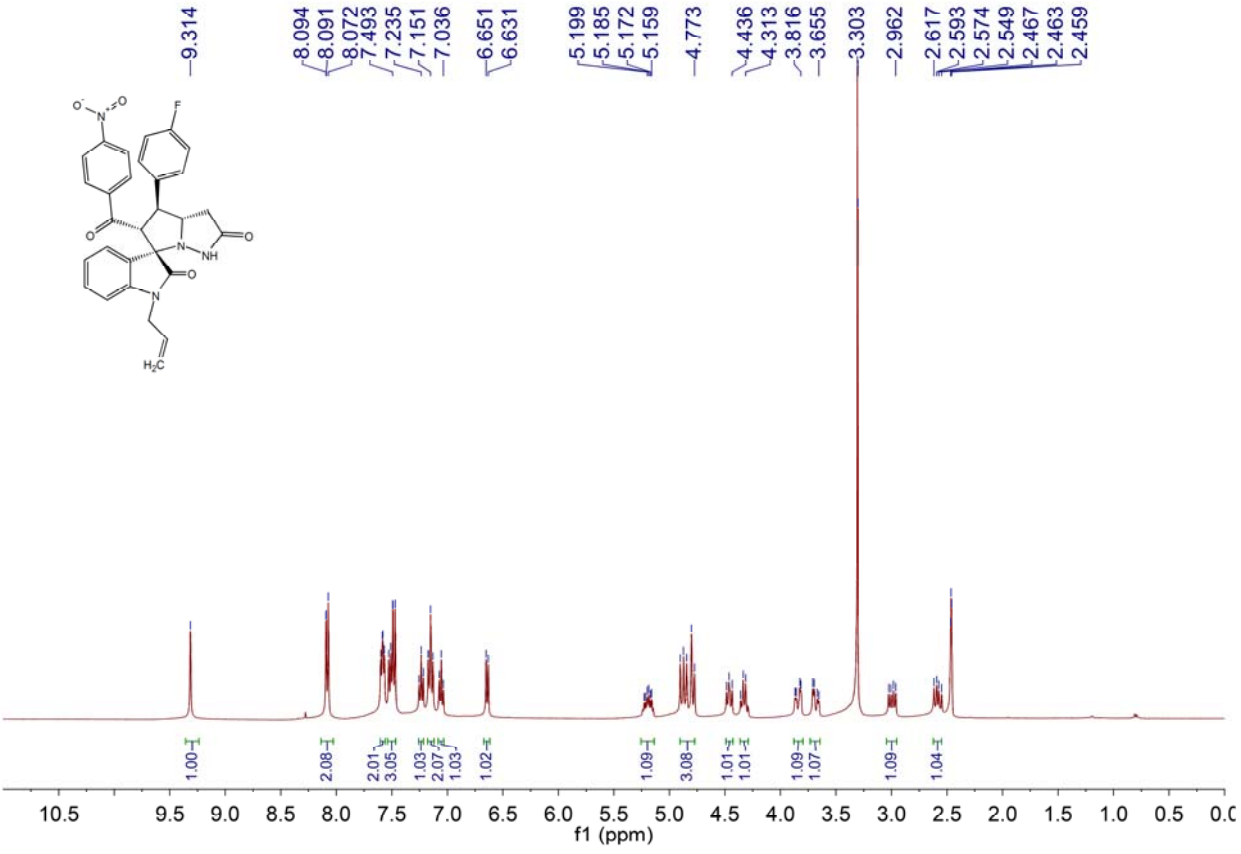
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3s**

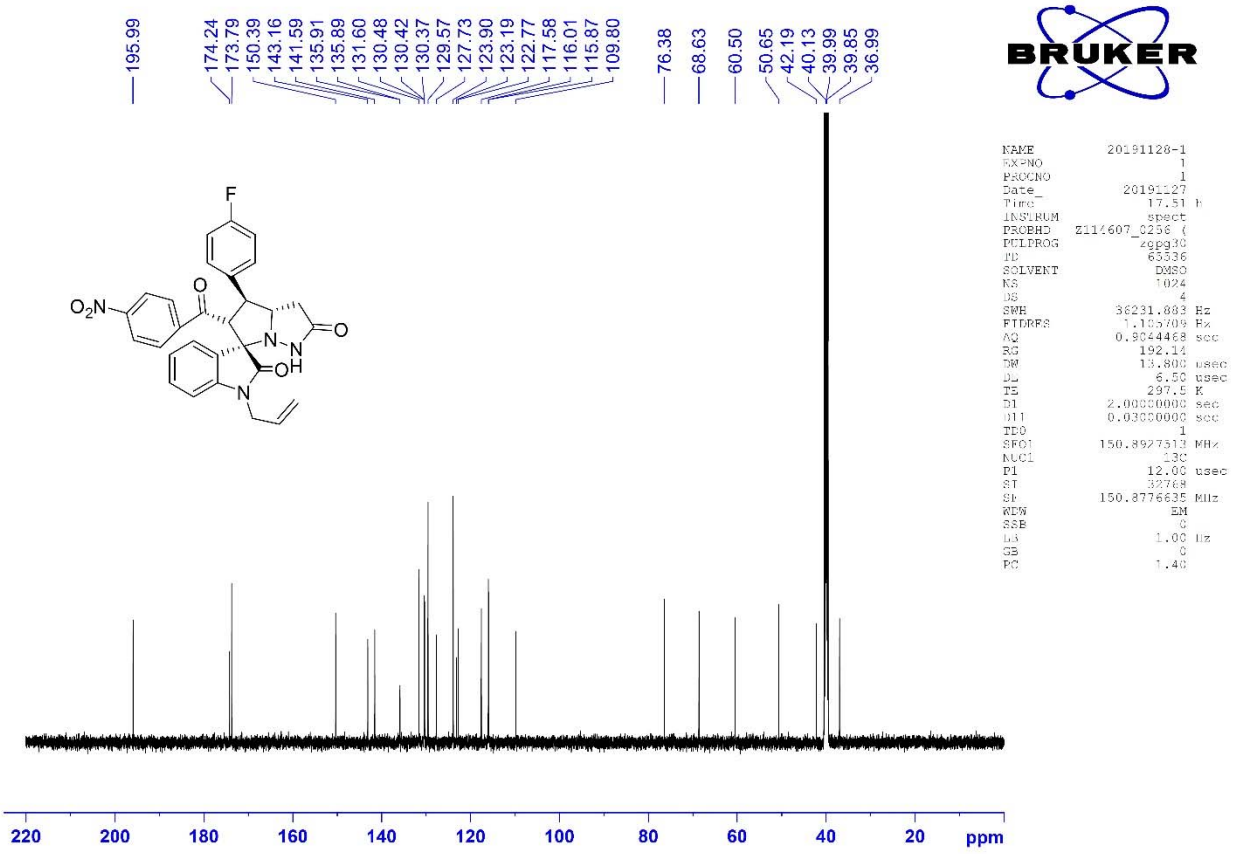


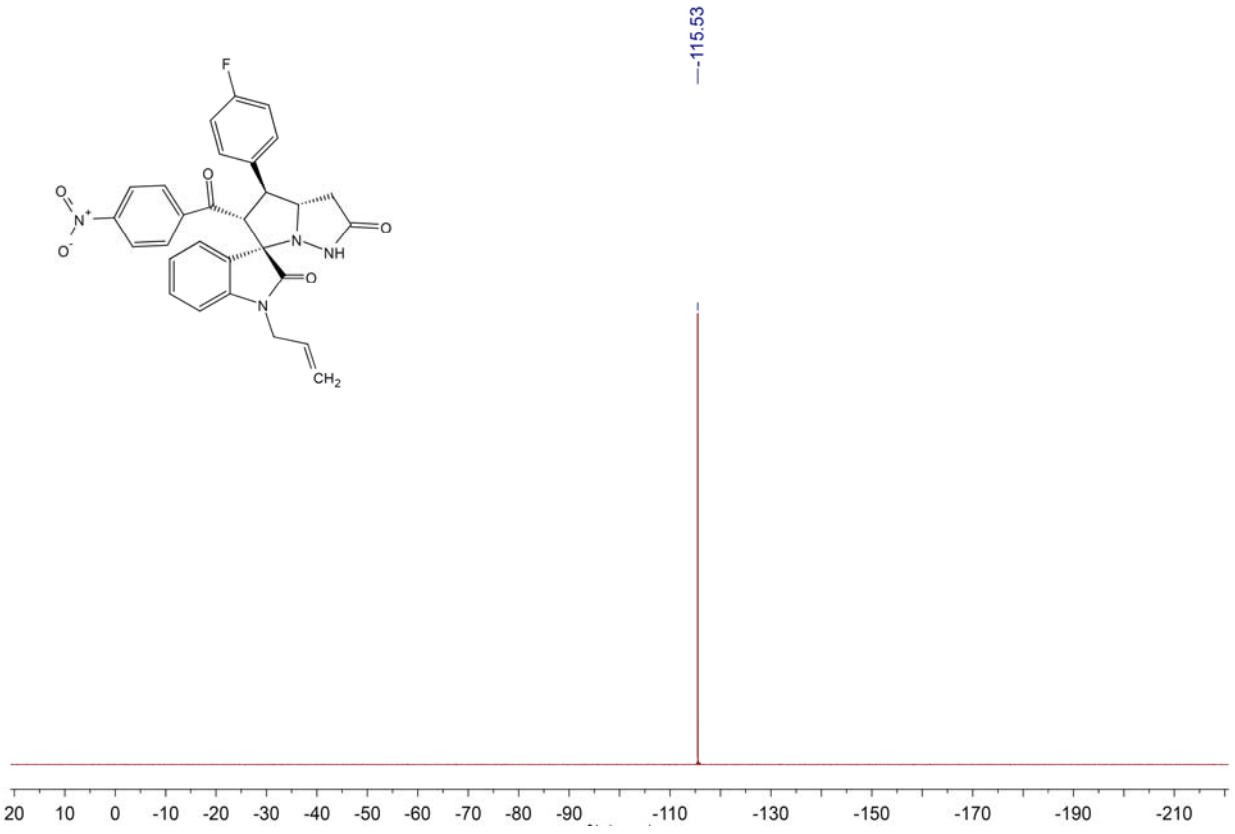




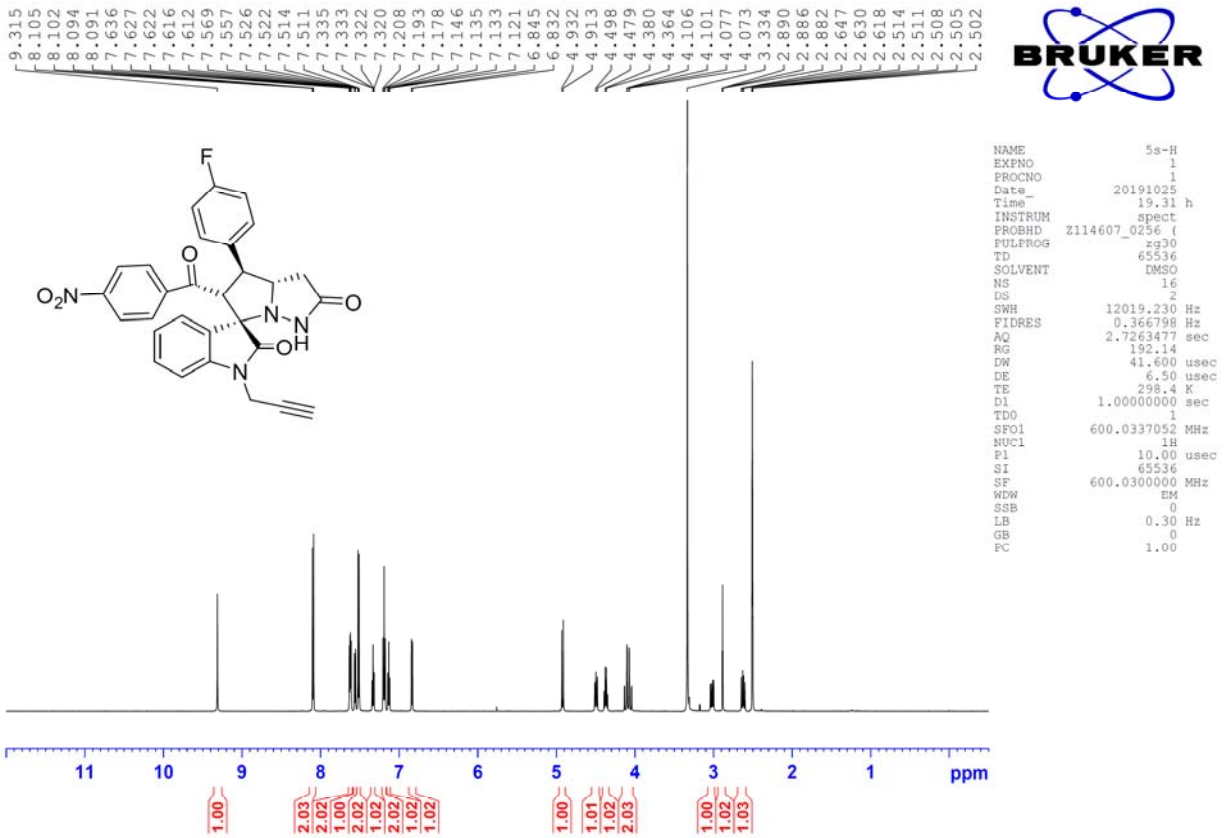
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3t**

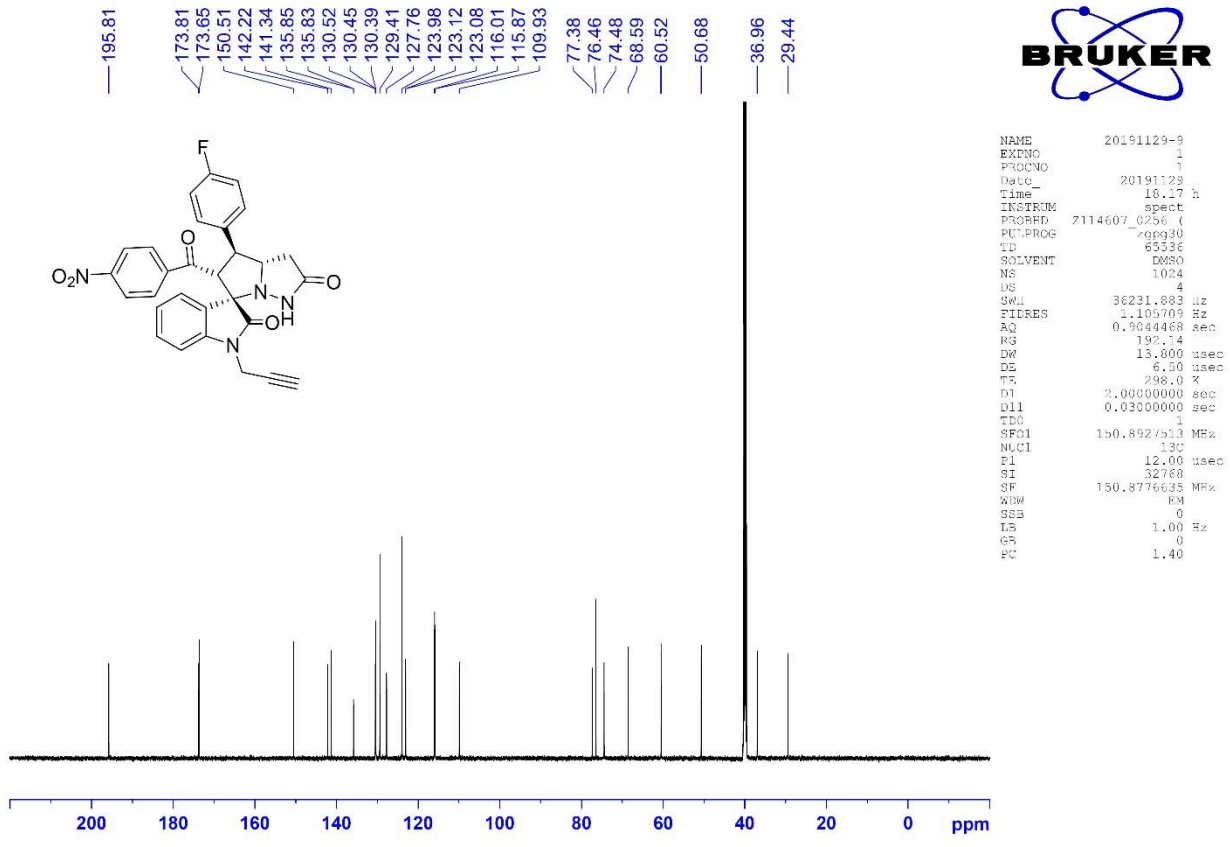


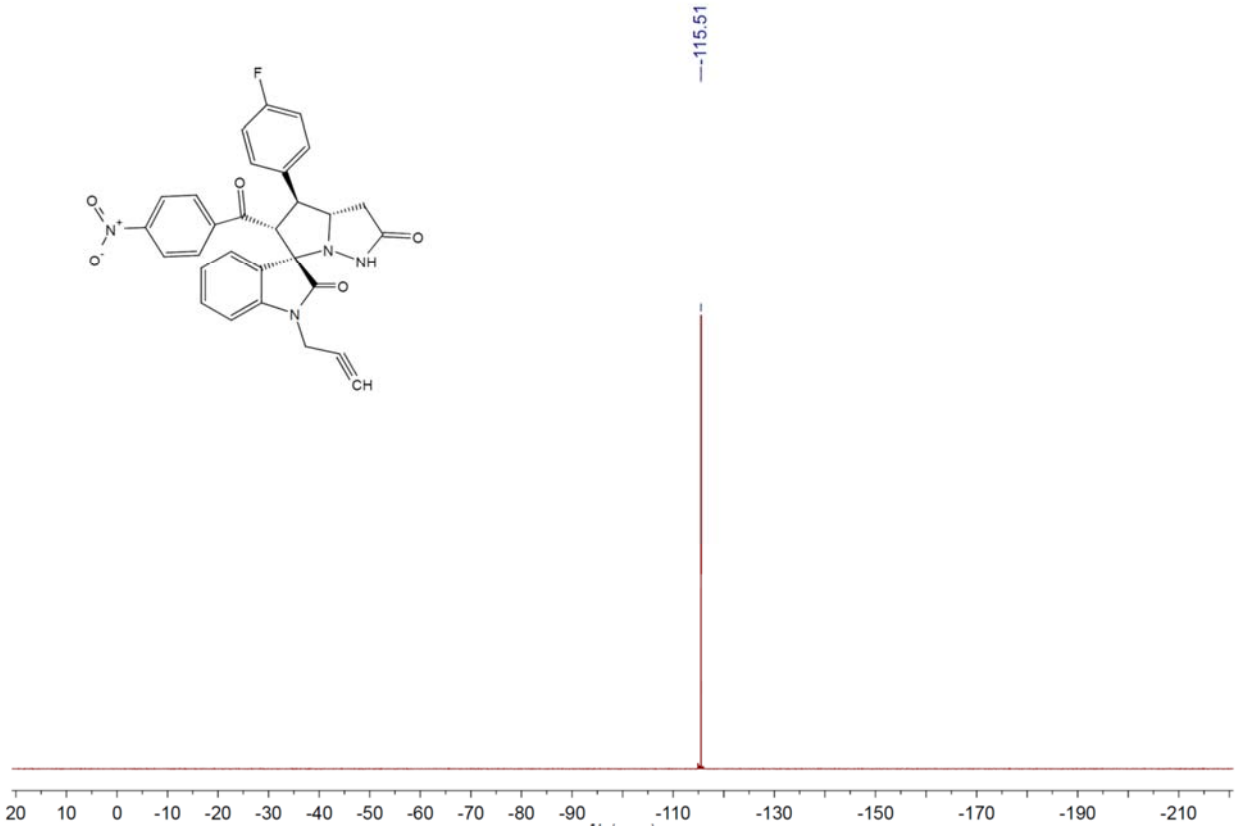

195.99
174.24
173.79
150.39
143.16
141.59
135.91
135.89
131.60
130.48
130.42
130.37
129.57
127.73
123.90
123.19
122.77
117.58
116.01
115.87
109.80
76.38
68.63
60.50
50.65
42.19
40.13
39.99
39.85
36.99
BRUKER
NAME 20191128-1
EXPNO 1
PROCNO 1
Date_ 20191127
Time 17.51 h
INSTRUM spect
PROBHD Z114607_0256 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 38231.883 Hz
FIDRES 1.165709 Hz
AQ 0.8044468 sec
RG 192.14
DW 13.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.8927513 MHz
NUC1 13C
P1 12.00 usec
SI 32768
SF 150.8776635 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
220 200 180 160 140 120 100 80 60 40 20 ppm


-115.53
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210

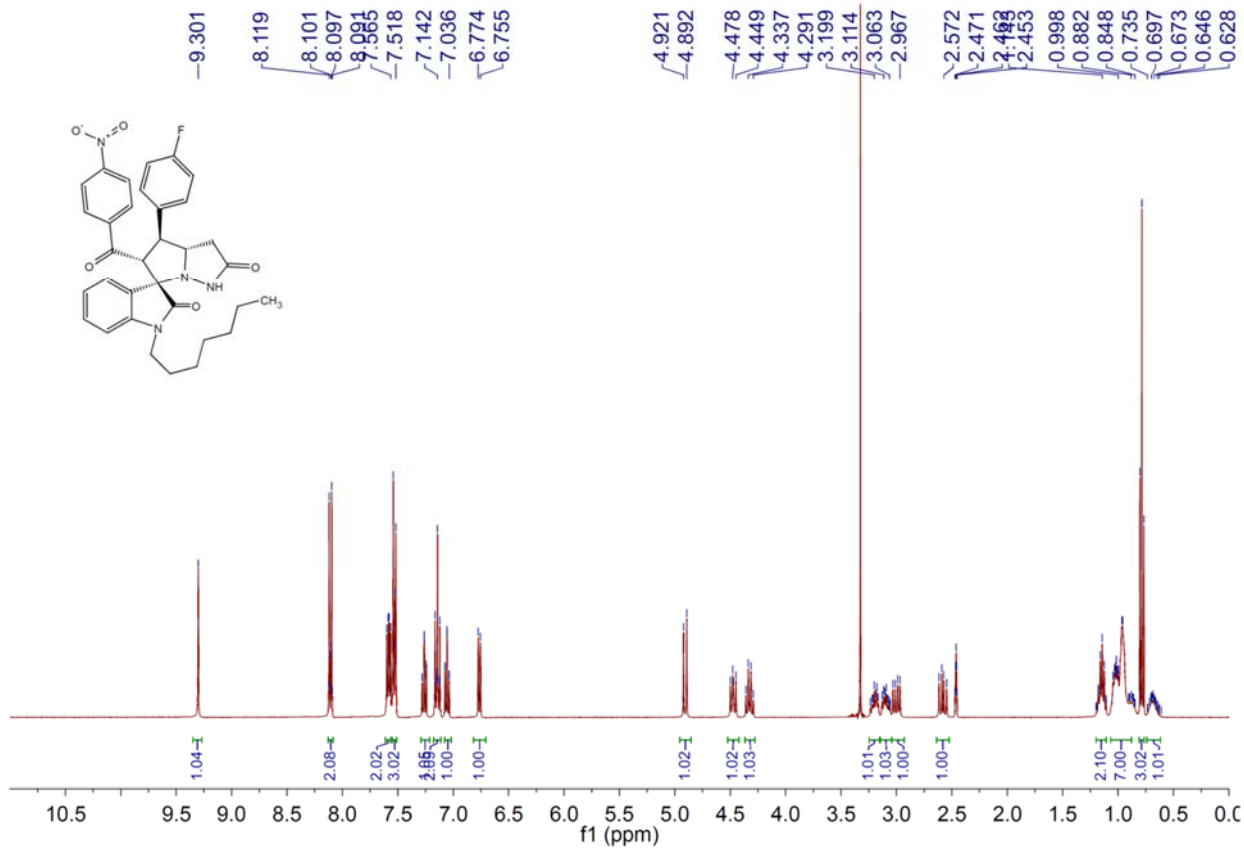
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3u**

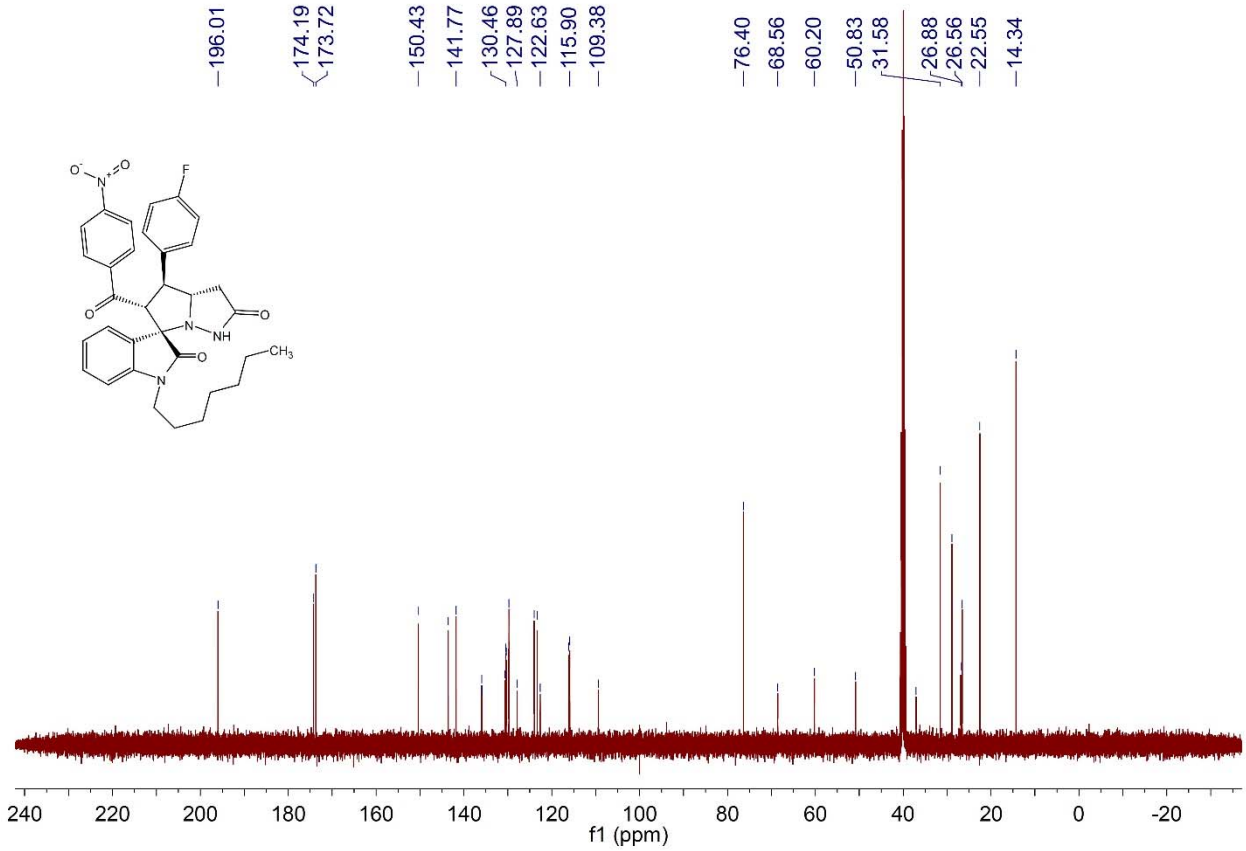






^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3v**

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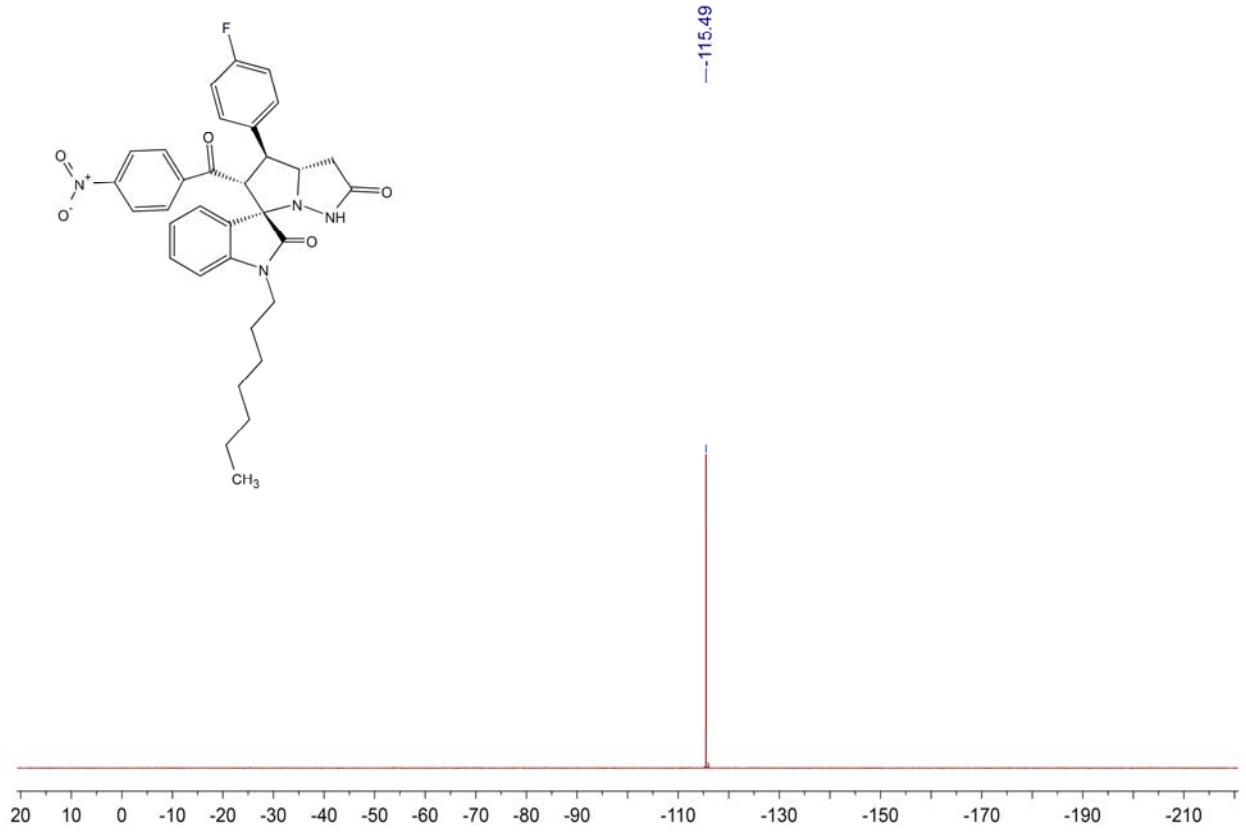
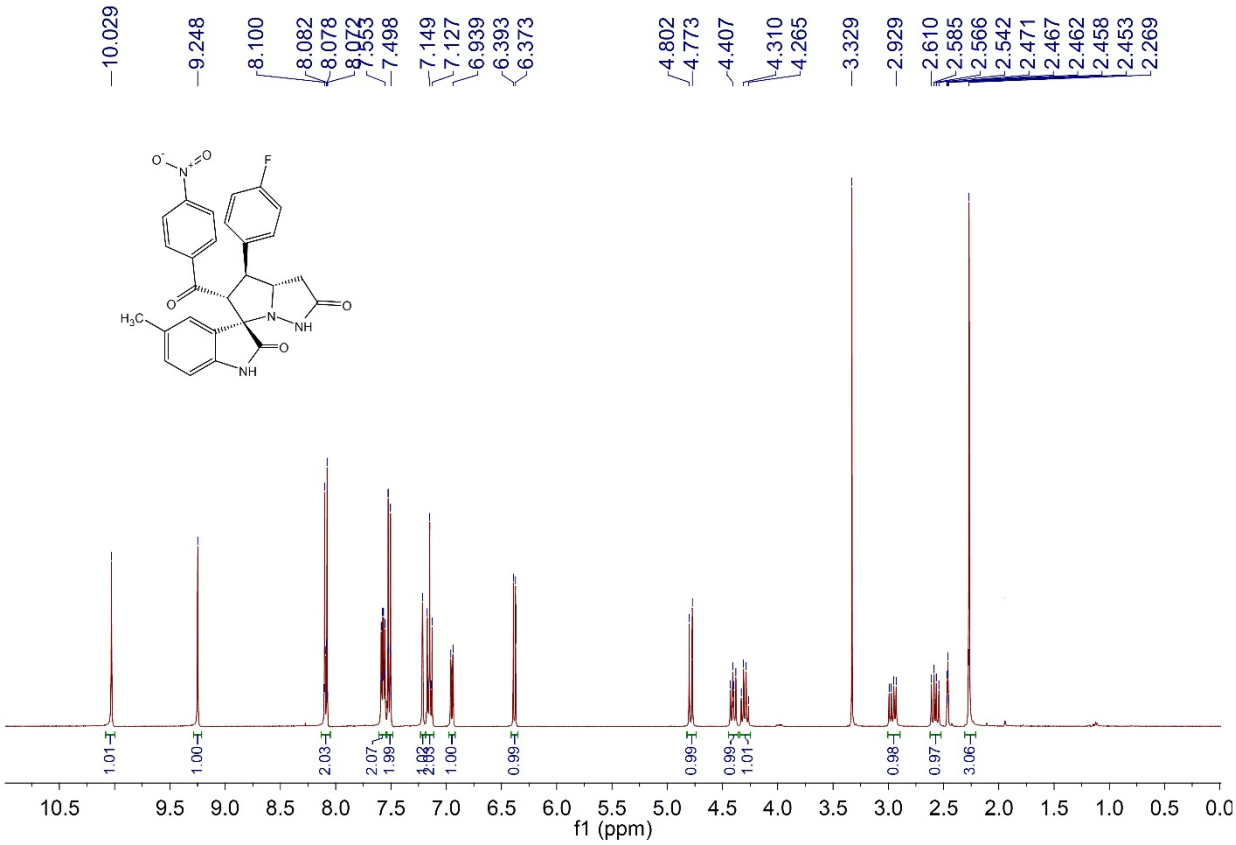
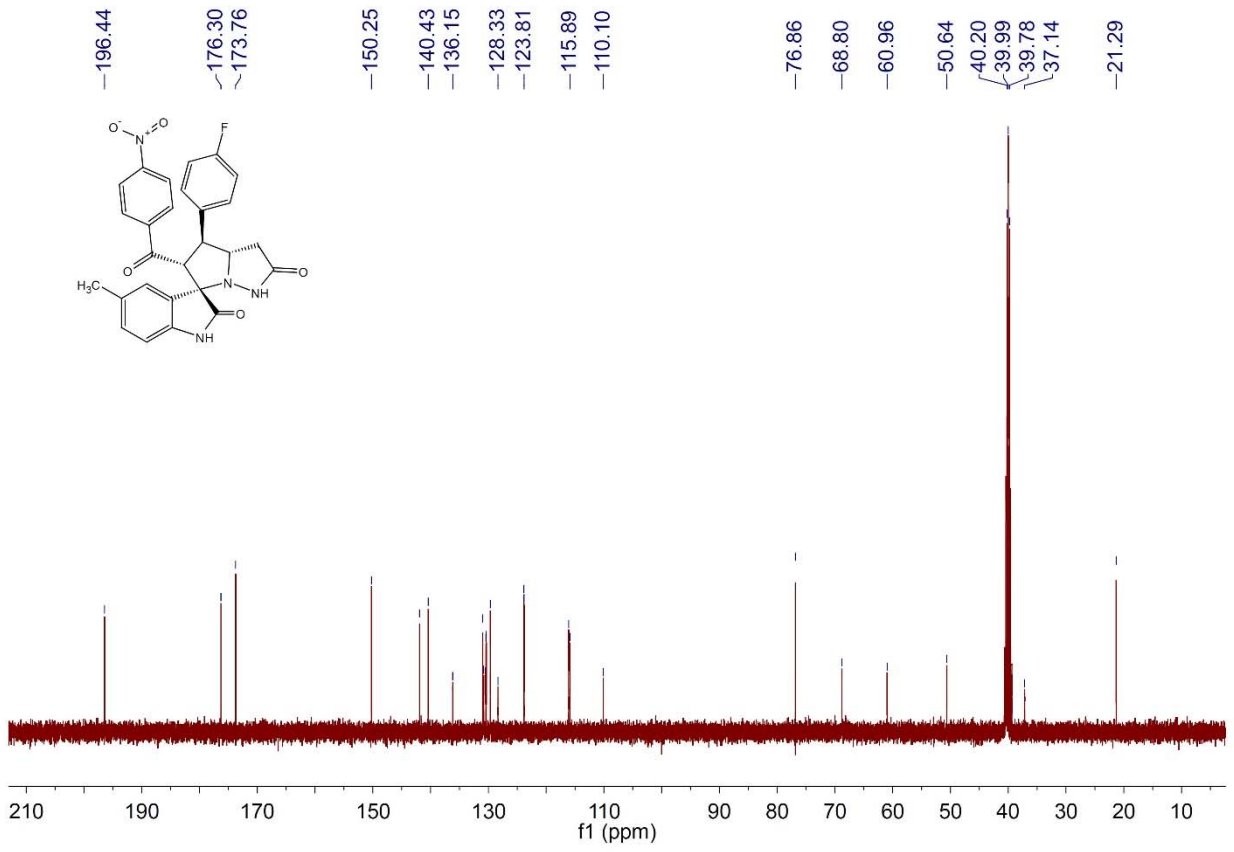
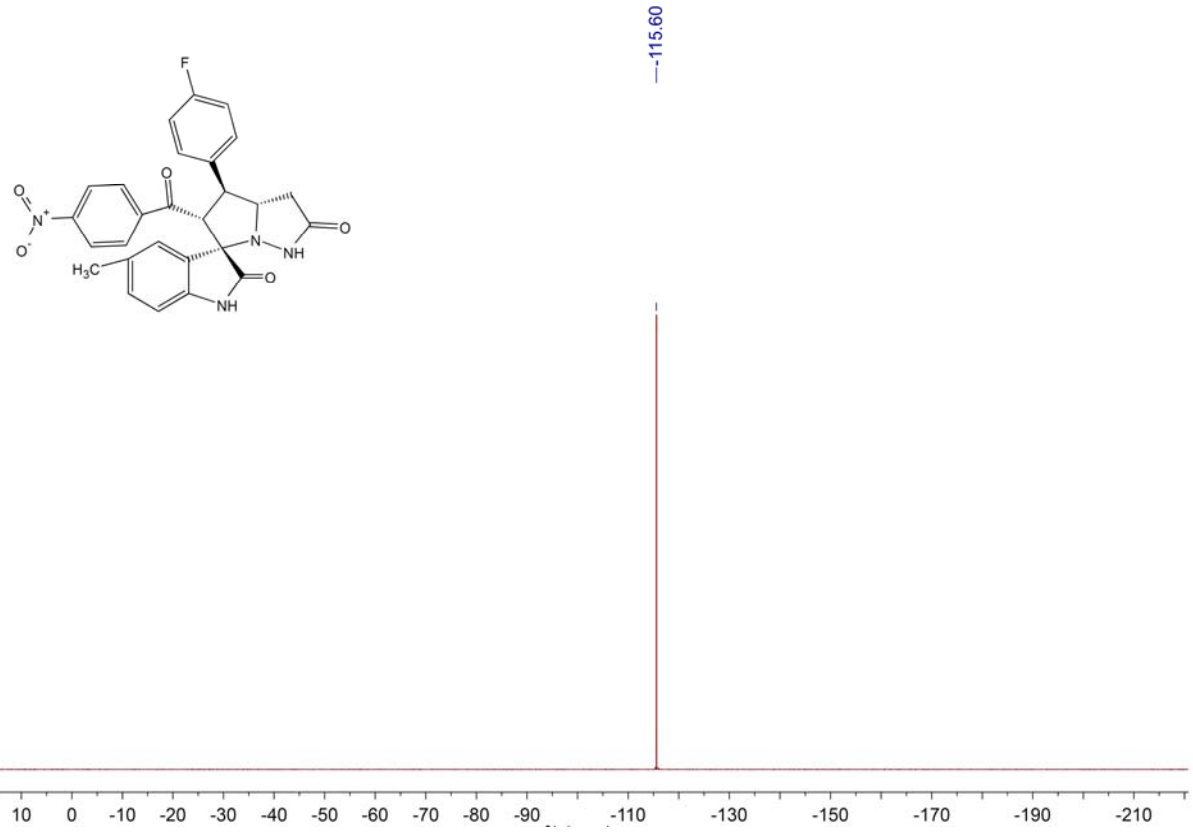
174.19
173.72
150.43
141.77
130.46
127.89
122.63
115.90
109.38
76.40
68.56
60.20
50.83
31.58
26.88
26.56
22.55
14.34
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)


-115.49
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210

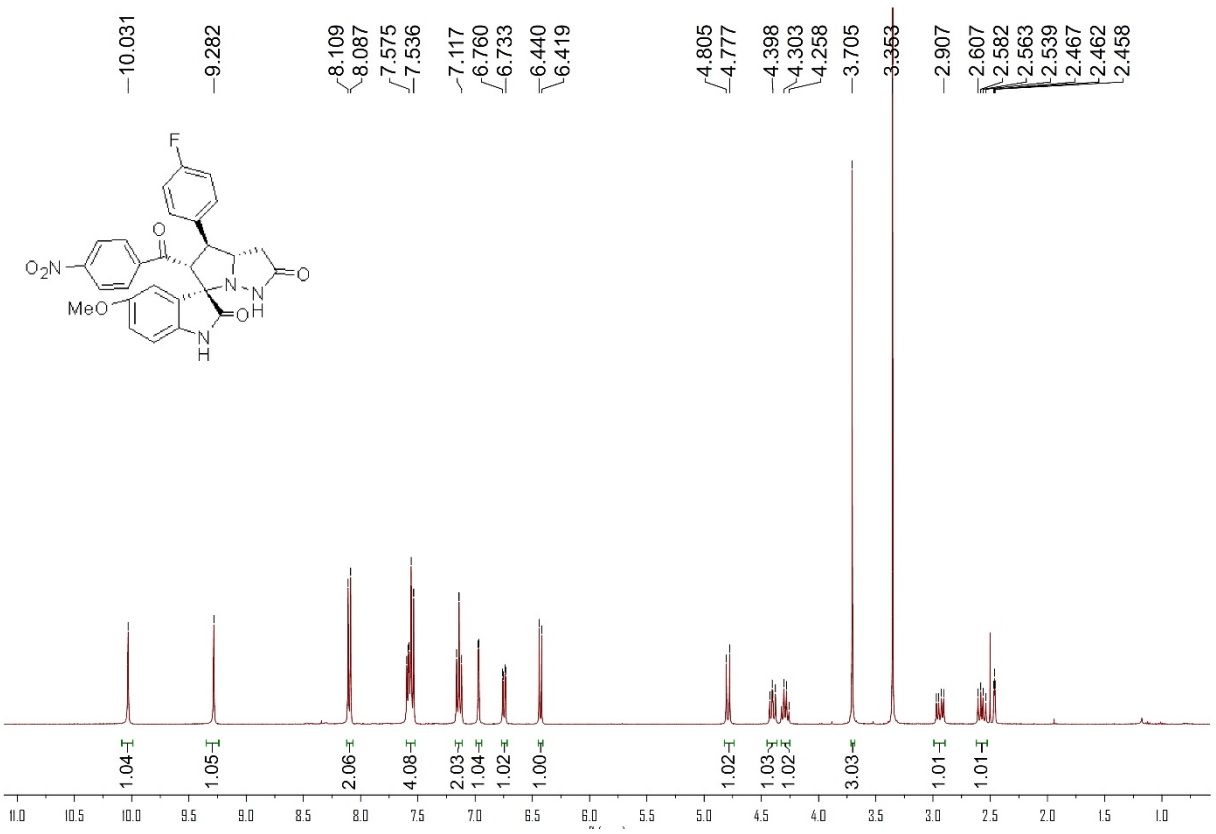
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3w**

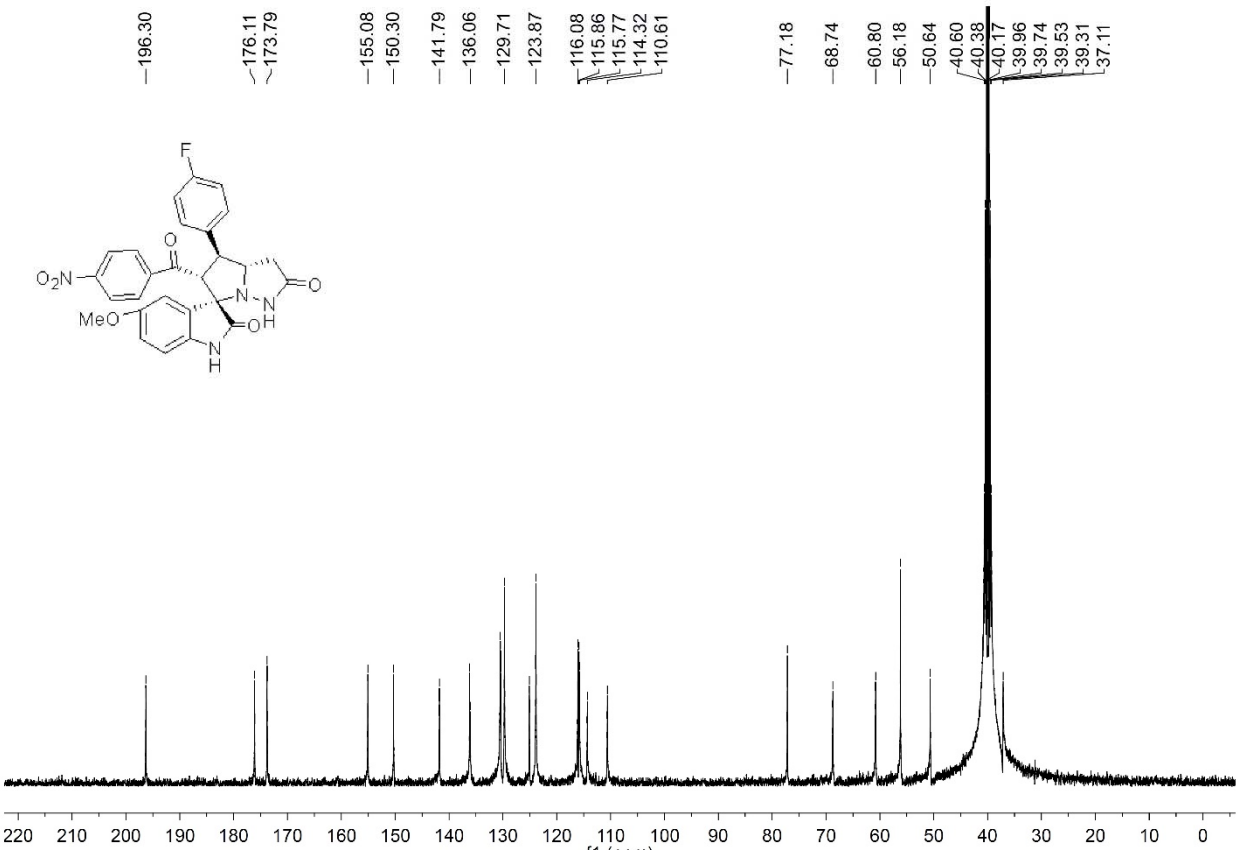


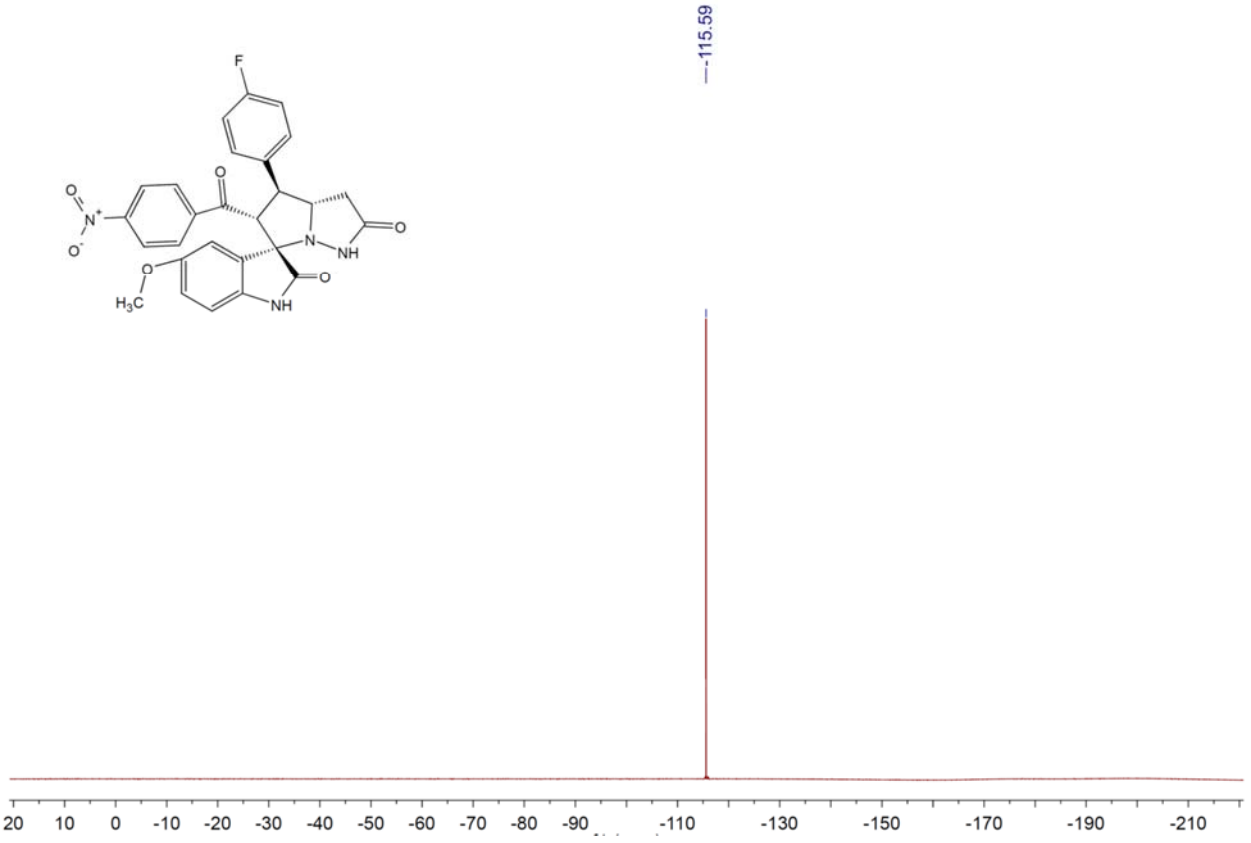




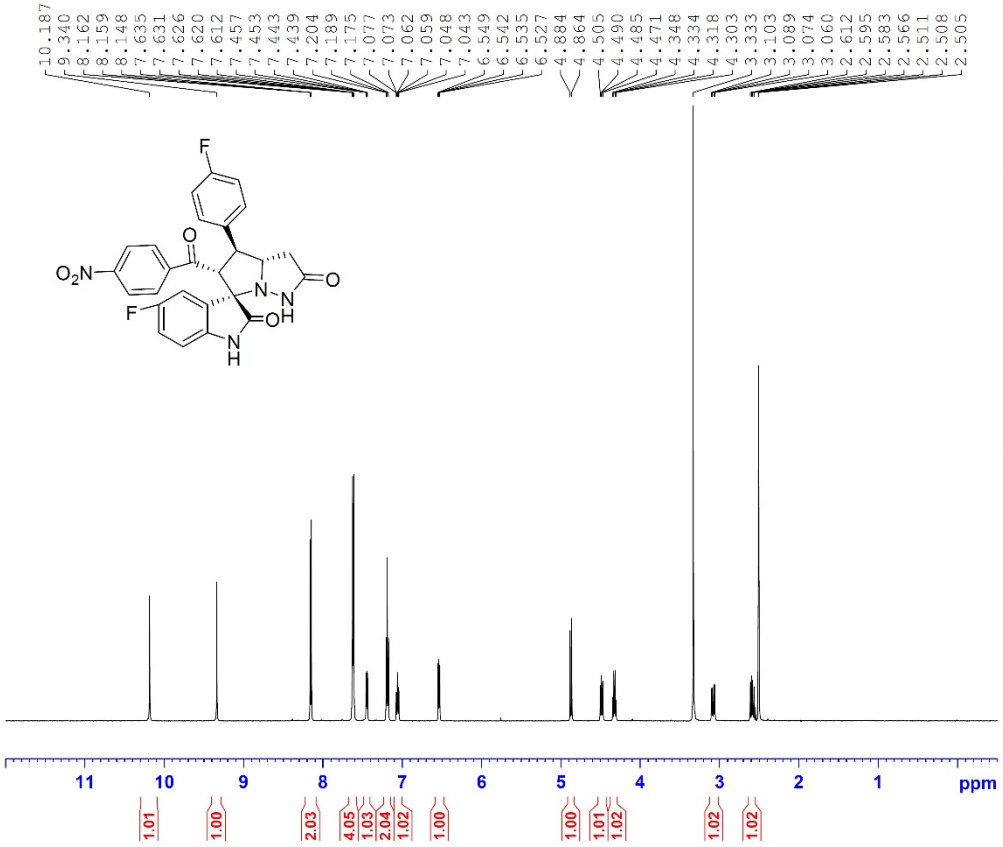
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3x**

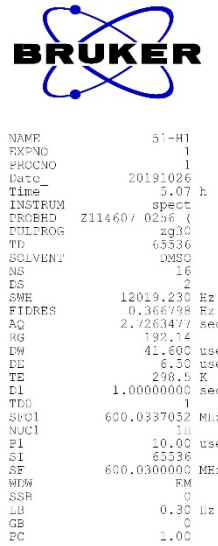


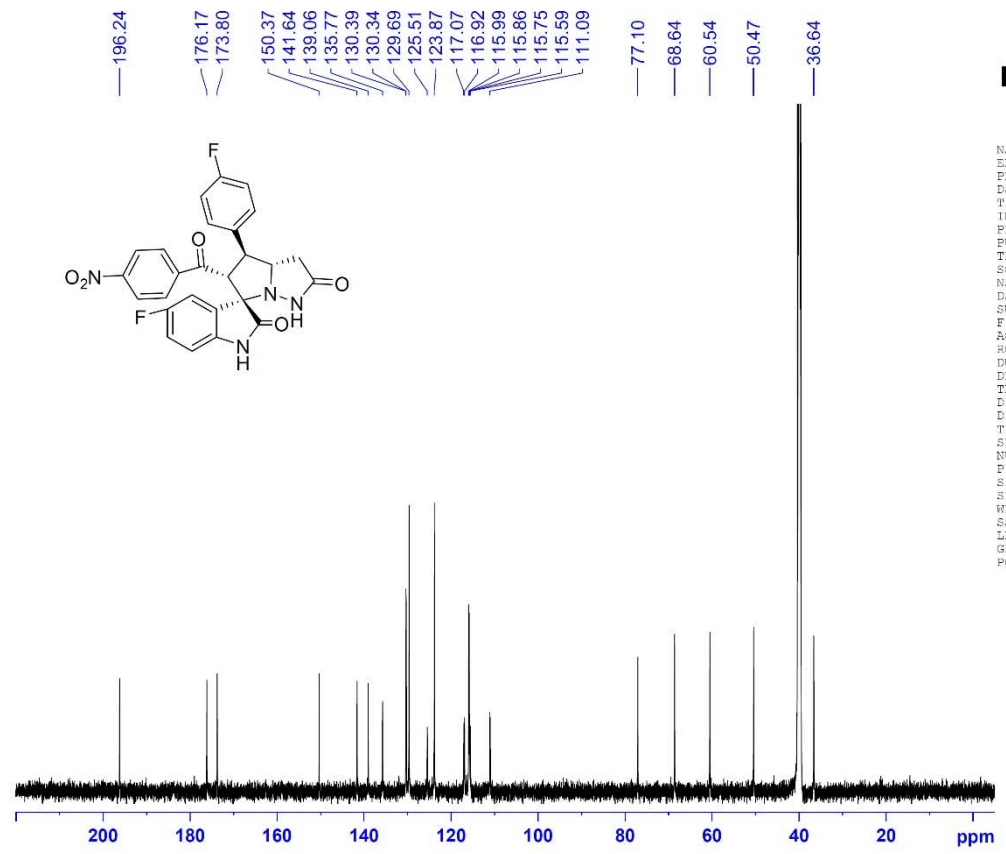

196.30
176.11
173.79
155.08
150.30
141.79
136.06
129.71
123.87
116.08
115.86
115.77
114.32
110.61
77.18
68.74
60.80
56.18
50.64
40.60
40.38
40.17
39.96
39.74
39.53
39.31
37.11
F
O
O2N
MeO
N
N
H
O
OH
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

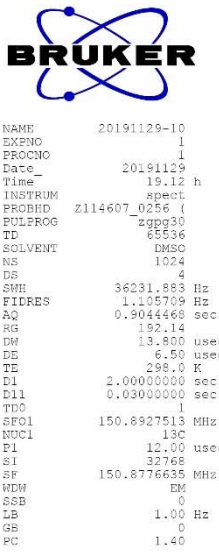

115.59
F
O
O
N
O
N
NH
O
O
H3C
NH
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210

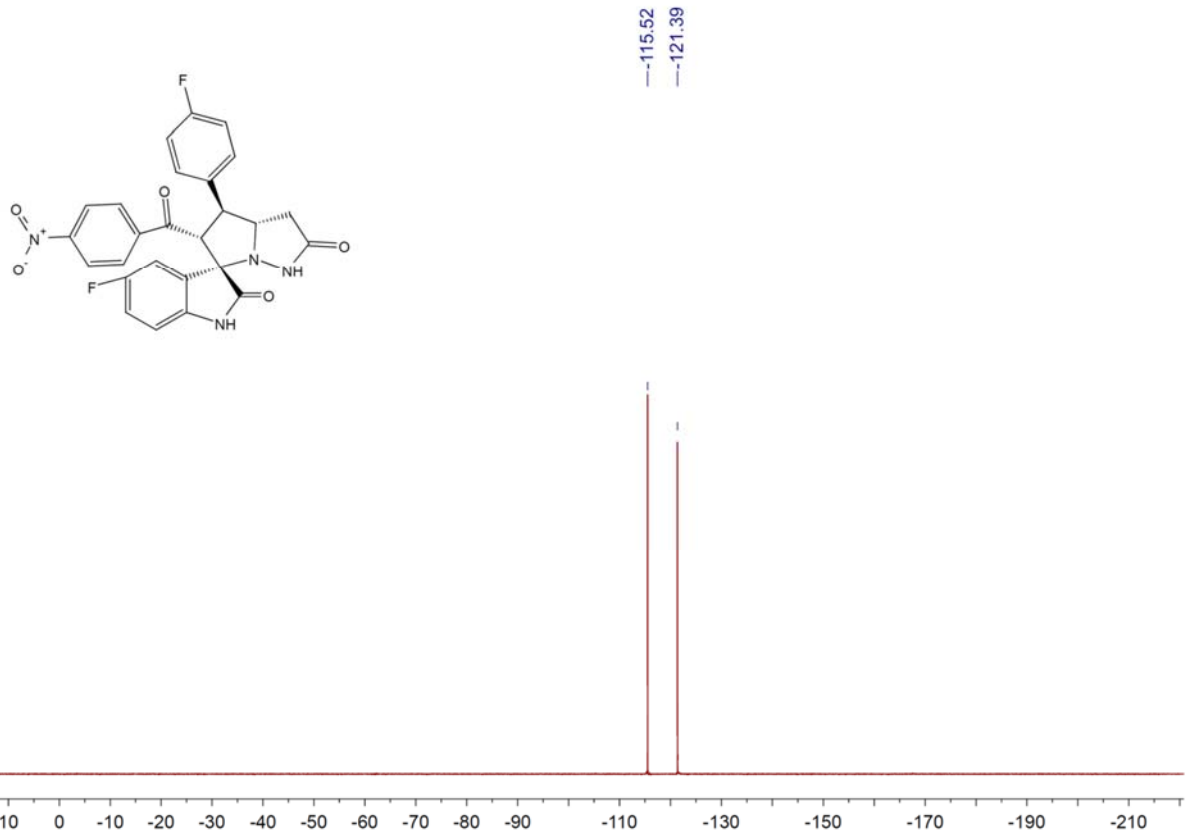
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3y**



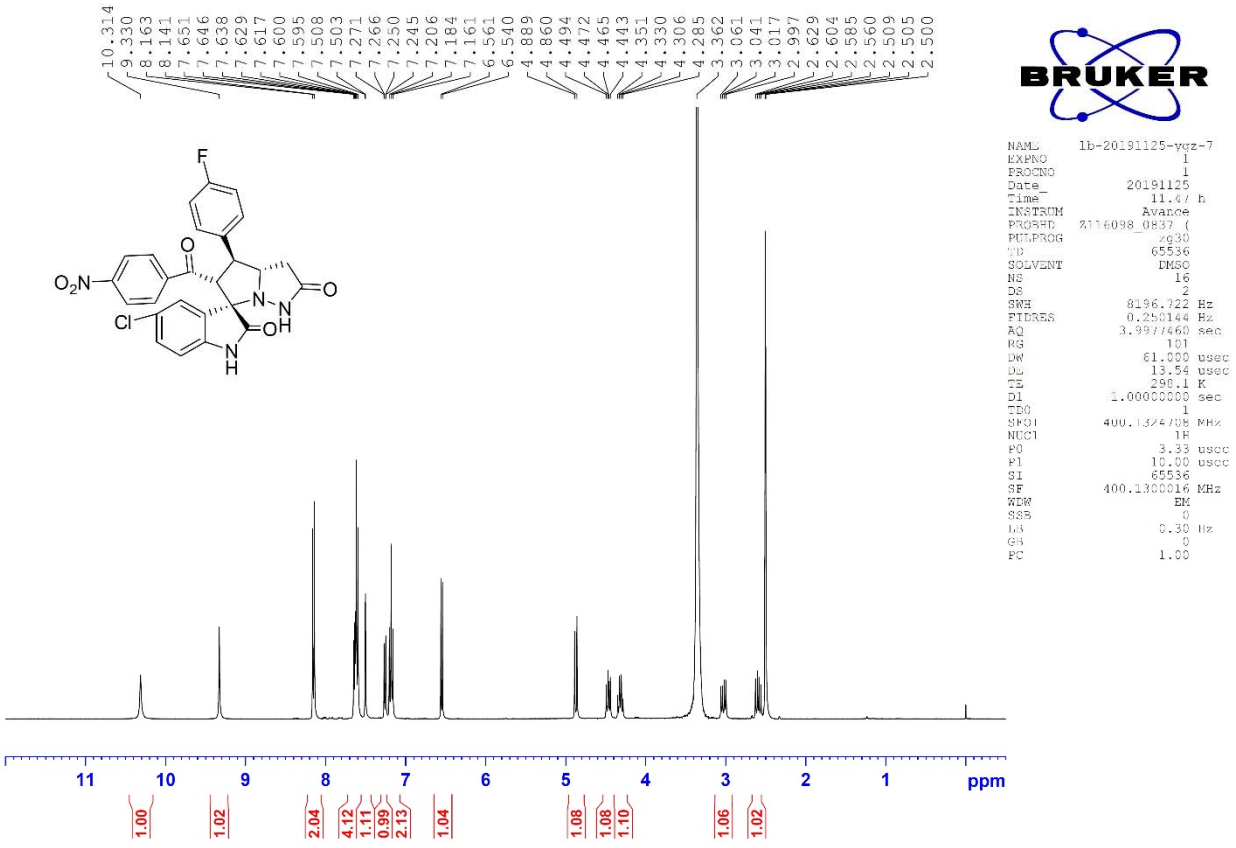


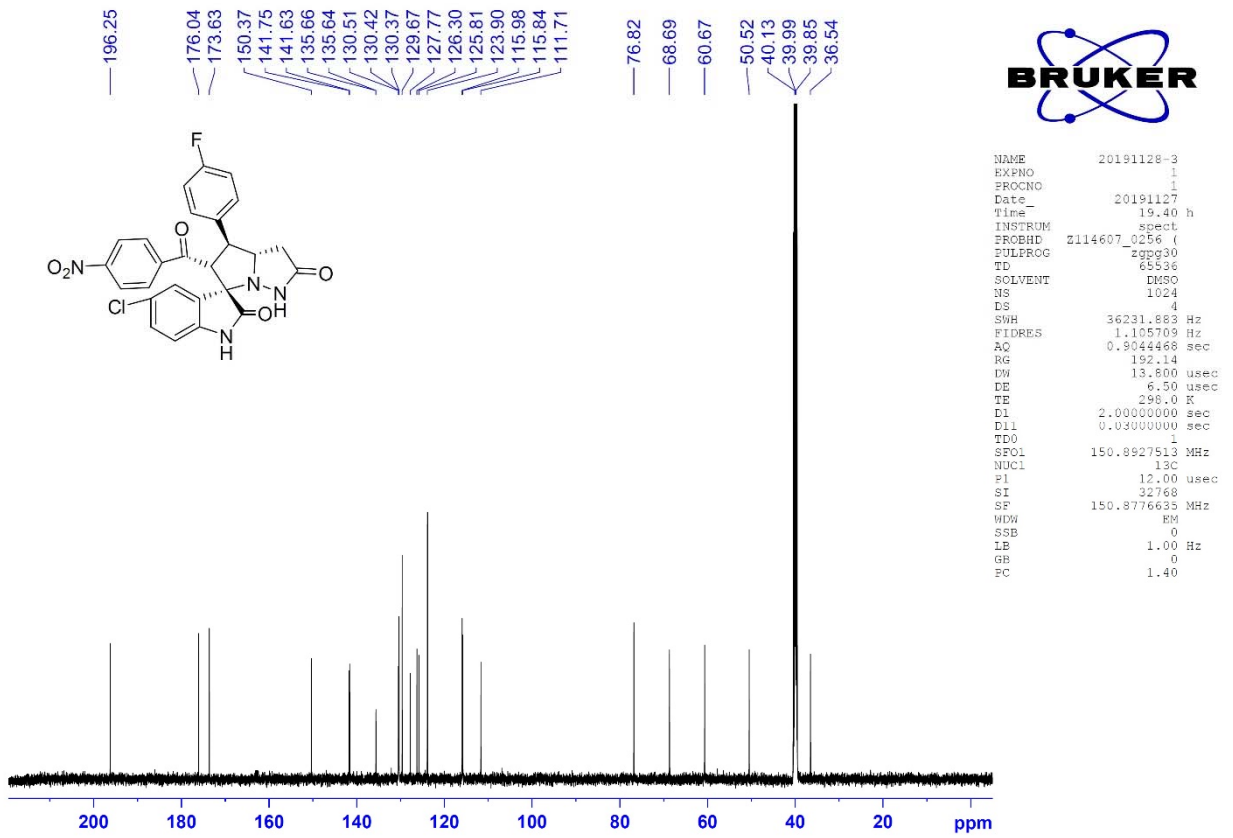






^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3z**





BRUKER

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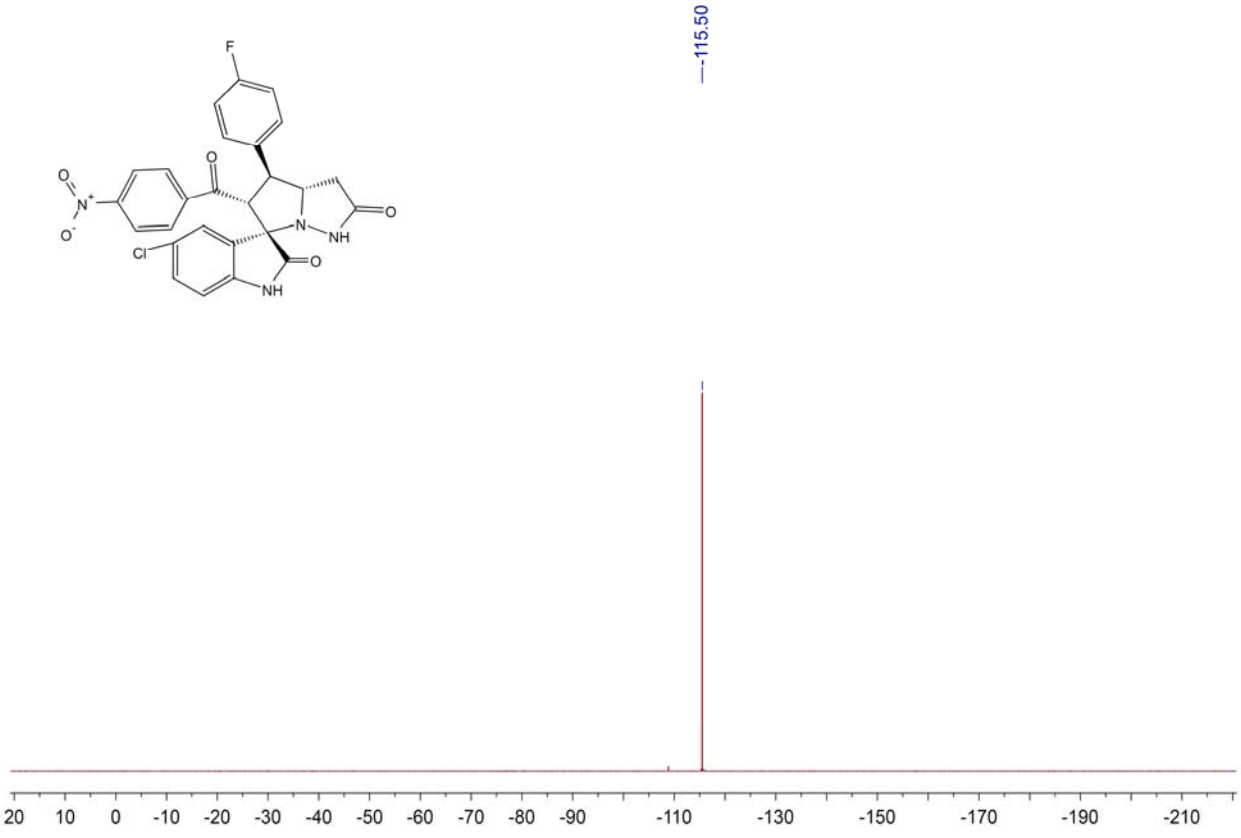
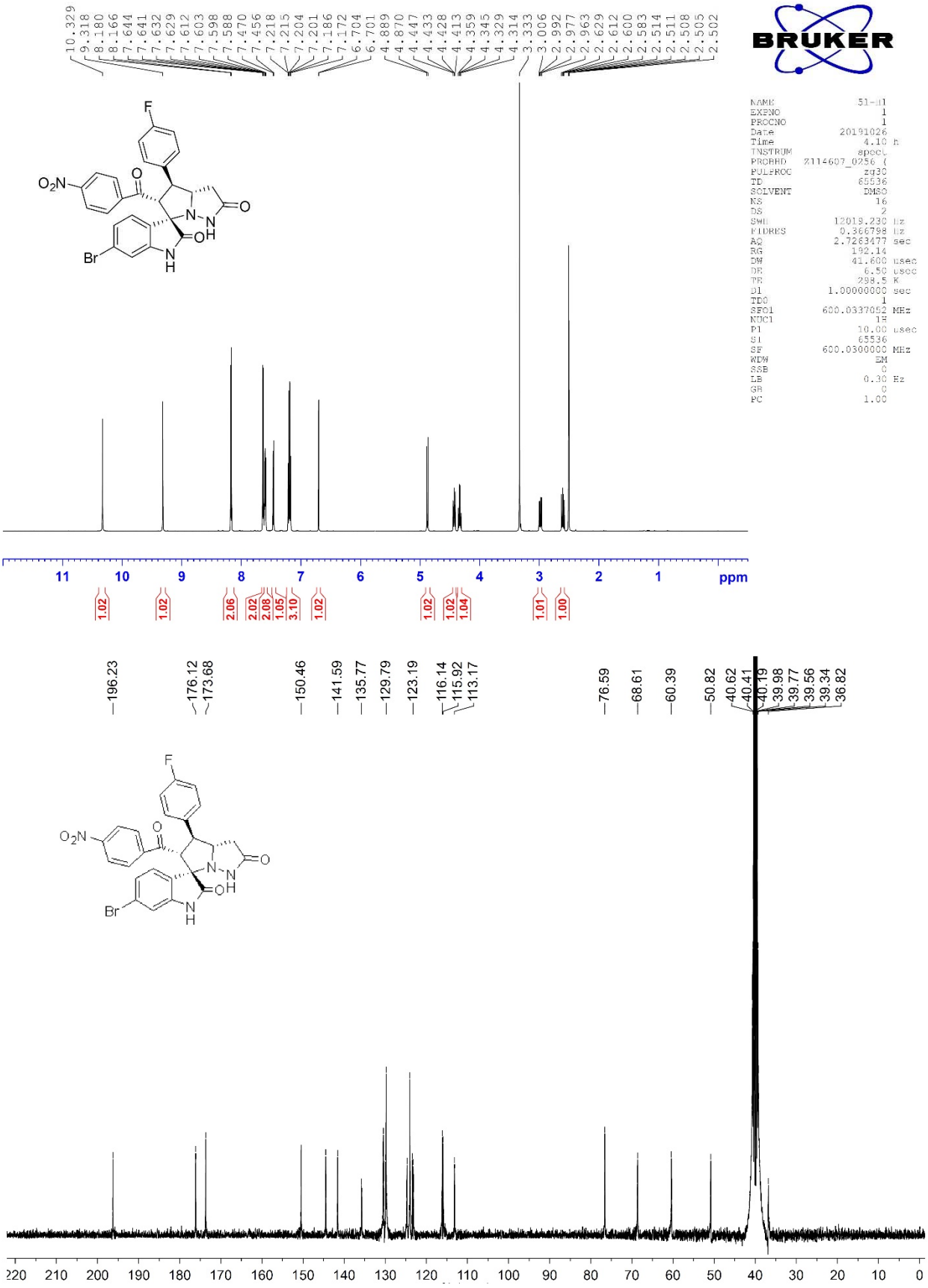
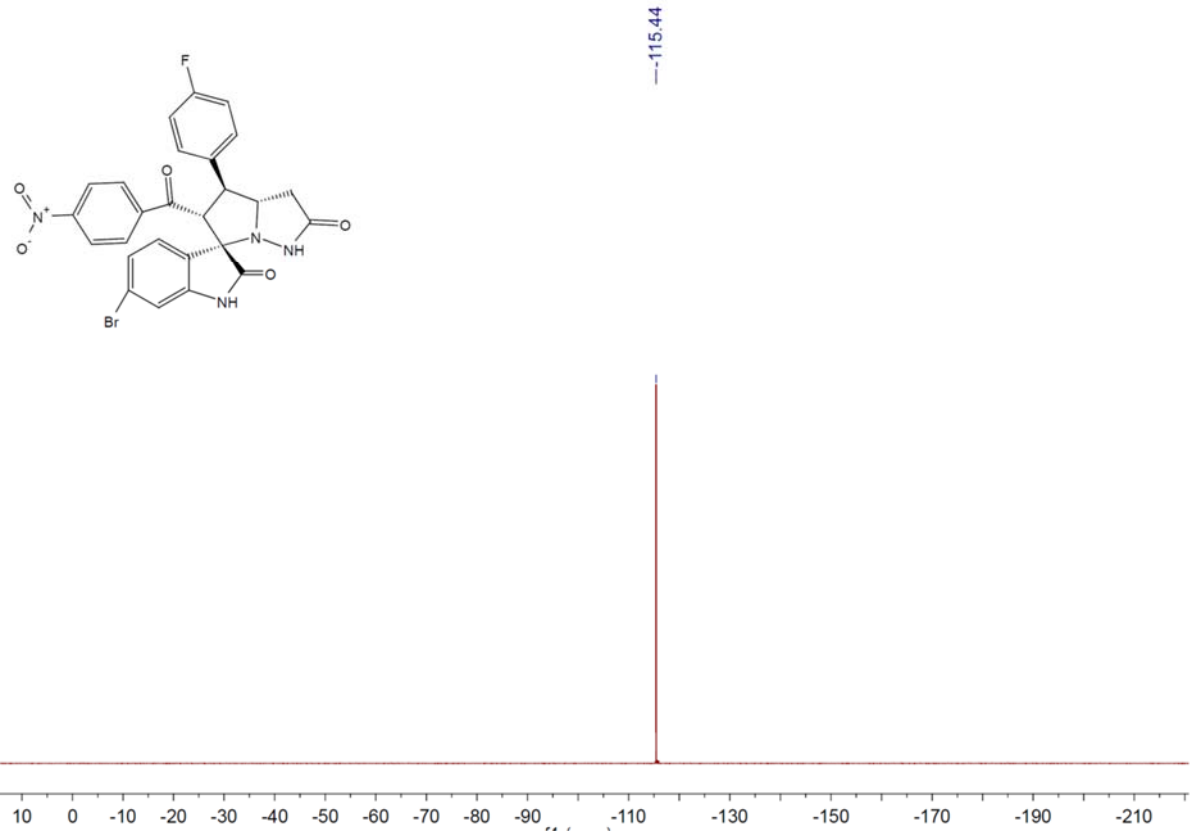
NAME            20191128-3
EXPNO                    1
PROCNO                   1
Date_             20191127
Time                19.40 h
INSTRUM               spect
PROBHD    Z114607_0256 (
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                     1024
DS                        4
SWH              36231.883 Hz
FIDRES            1.105709 Hz
AQ                0.9044468 sec
RG                   192.14
DW                   13.800 usec
DE                    6.50 usec
TE                   298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1
SFO1          150.8927513 MHz
NUC1                   13C
P1                   12.00 usec
SI                   32768
SF            150.8776635 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

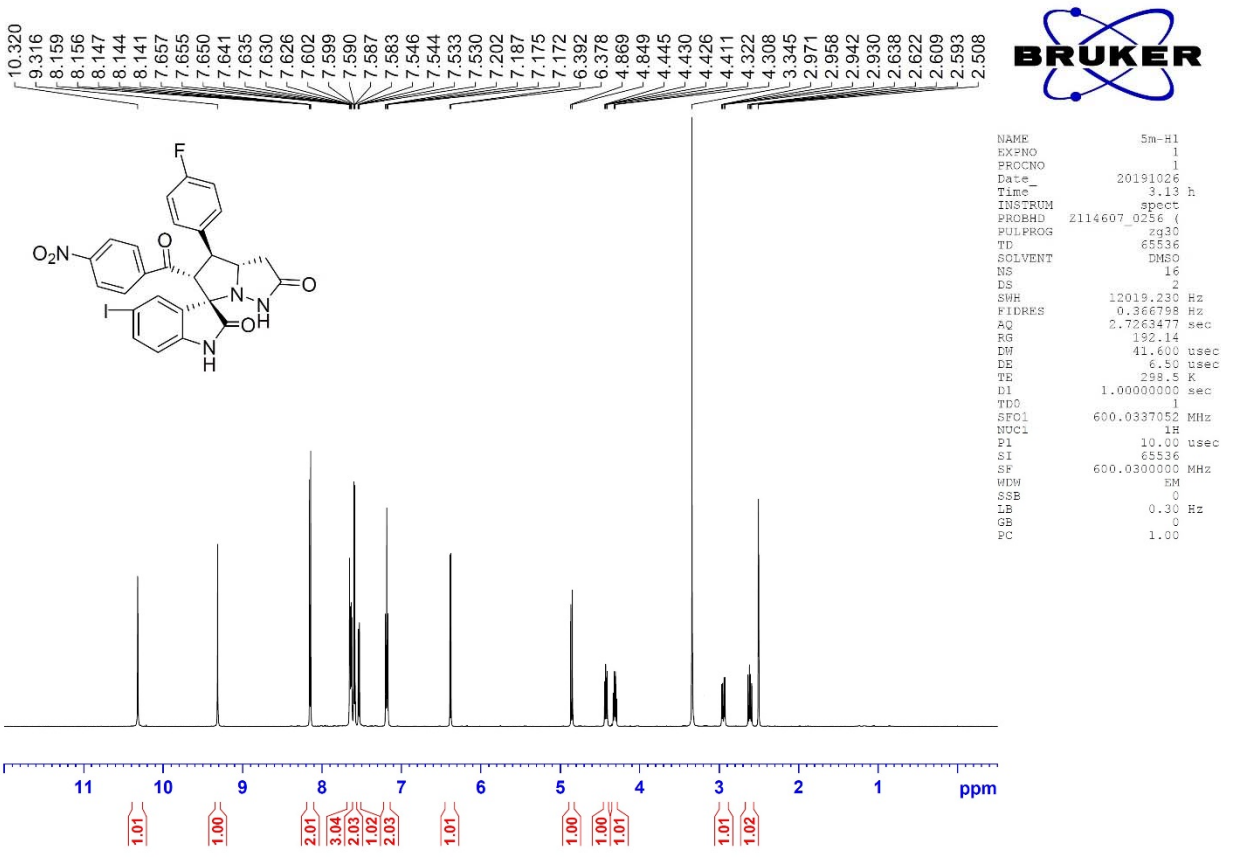


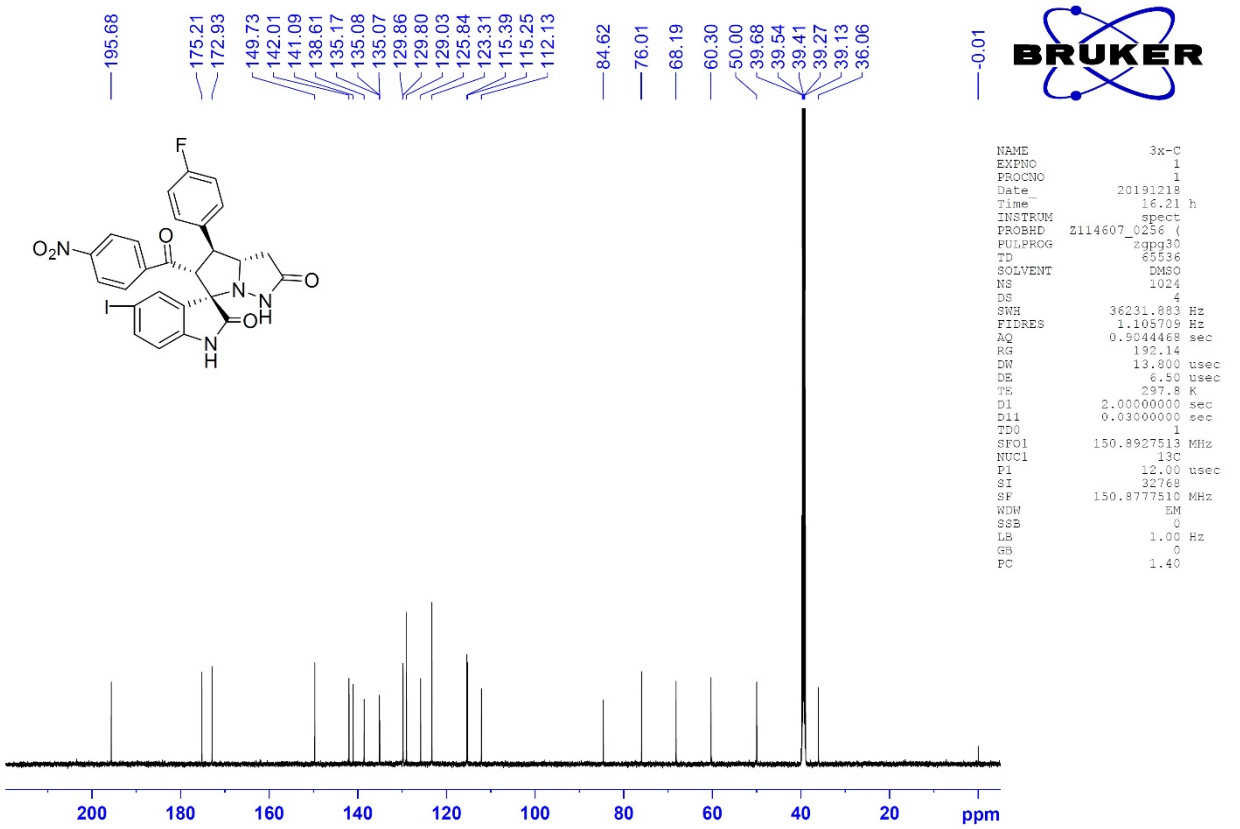
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3aa**





^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3ab**

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175.21
172.93
149.73
142.01
141.09
138.61
135.17
135.08
135.07
129.86
129.80
129.03
125.84
123.31
115.39
115.25
112.13
84.62
76.01
68.19
60.30
50.00
39.68
39.54
39.41
39.27
39.13
36.06
-0.01
BRUKER
NAME 3x-C
EXPNO 1
PROCNO 1
Date_ 20191218
Time_ 16.21 h
INSTRUM spect
PROBHD Z114607_0256 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 192.14
DW 13.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.8927513 MHz
NUC1 13C
P1 12.00 usec
SI 32768
SF 150.8777510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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180
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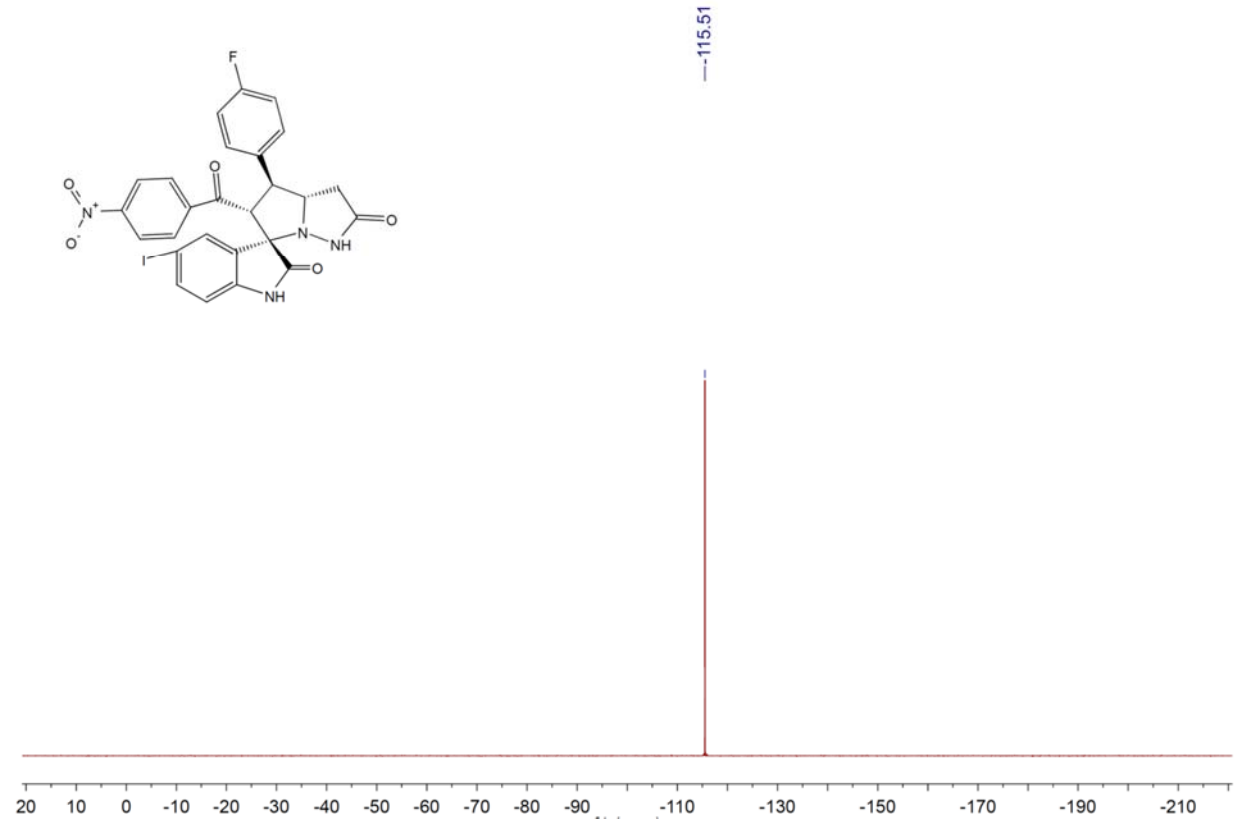
140
120
100
80
60
40
20
ppm


-115.51
20
10
0
-10
-20
-30
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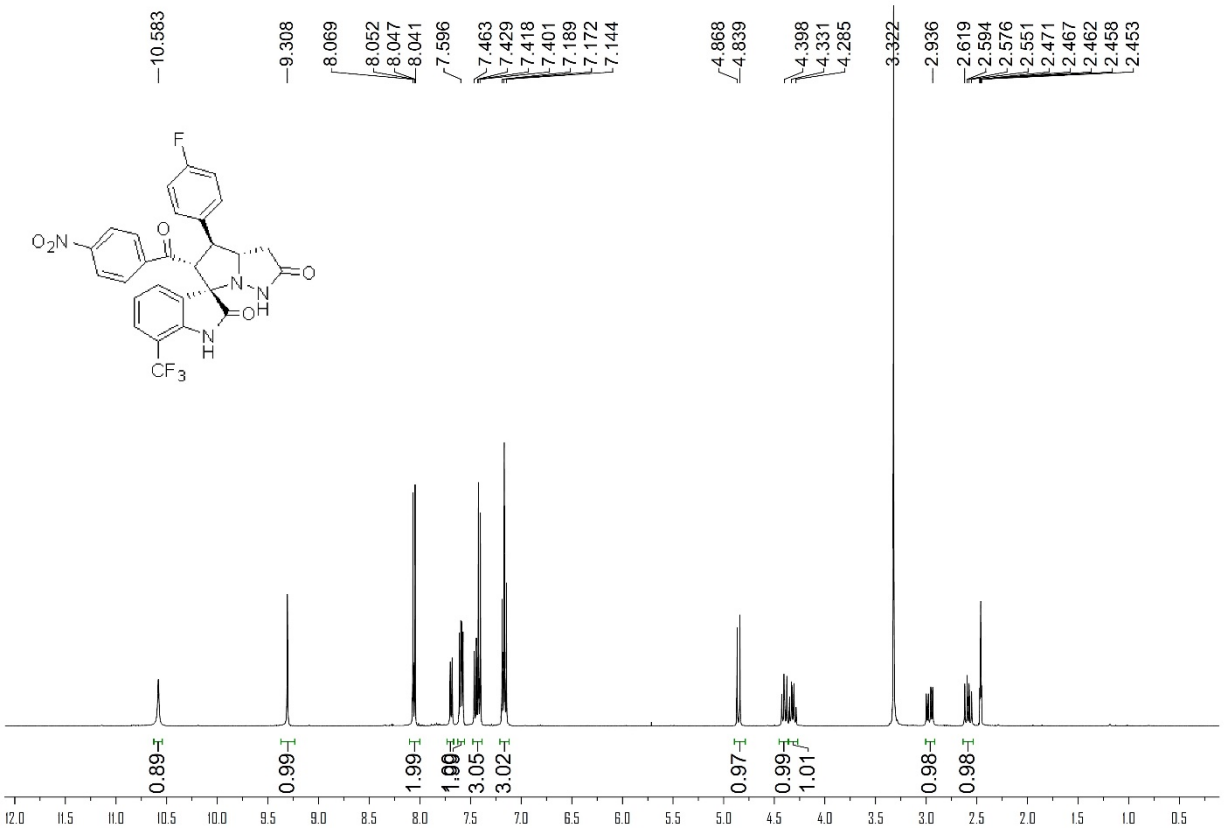
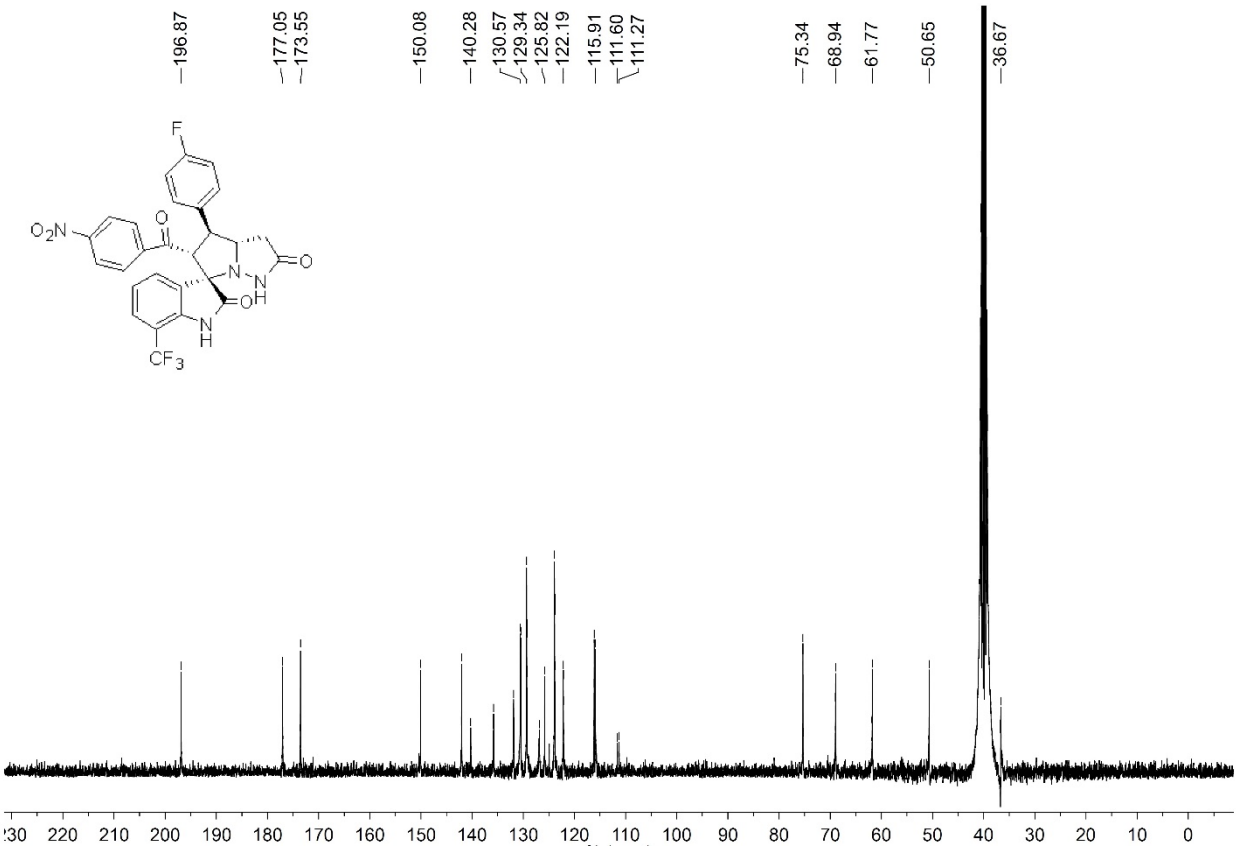
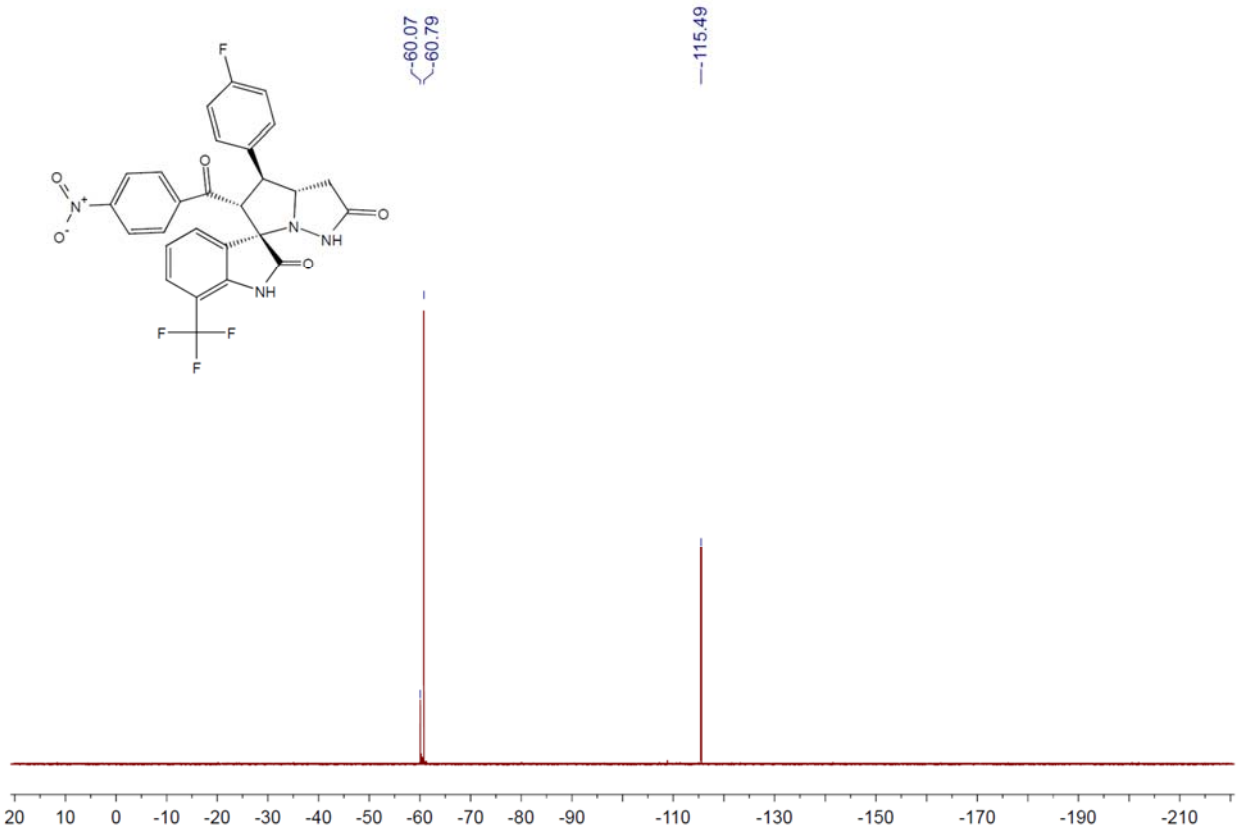
-70
-80
-90
-110
-130
-150
-170
-190
-210

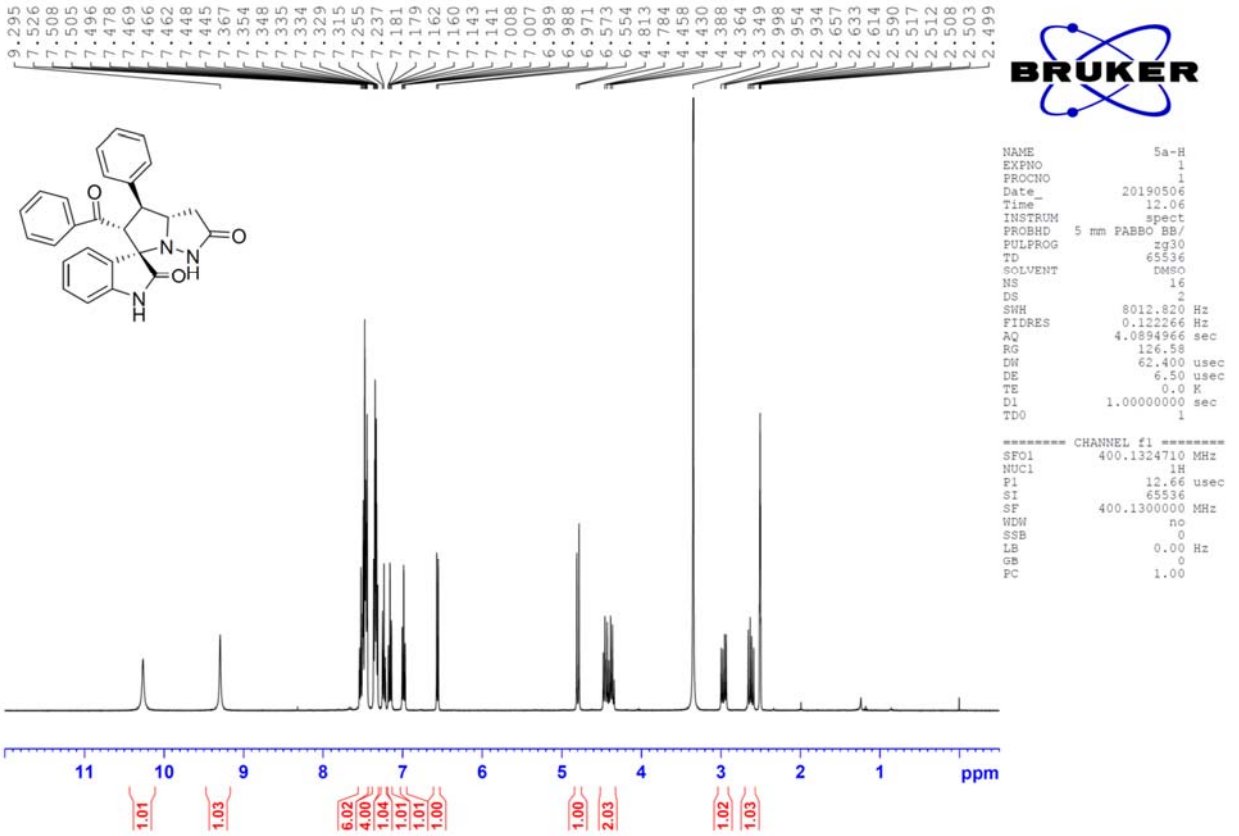
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **3ac**

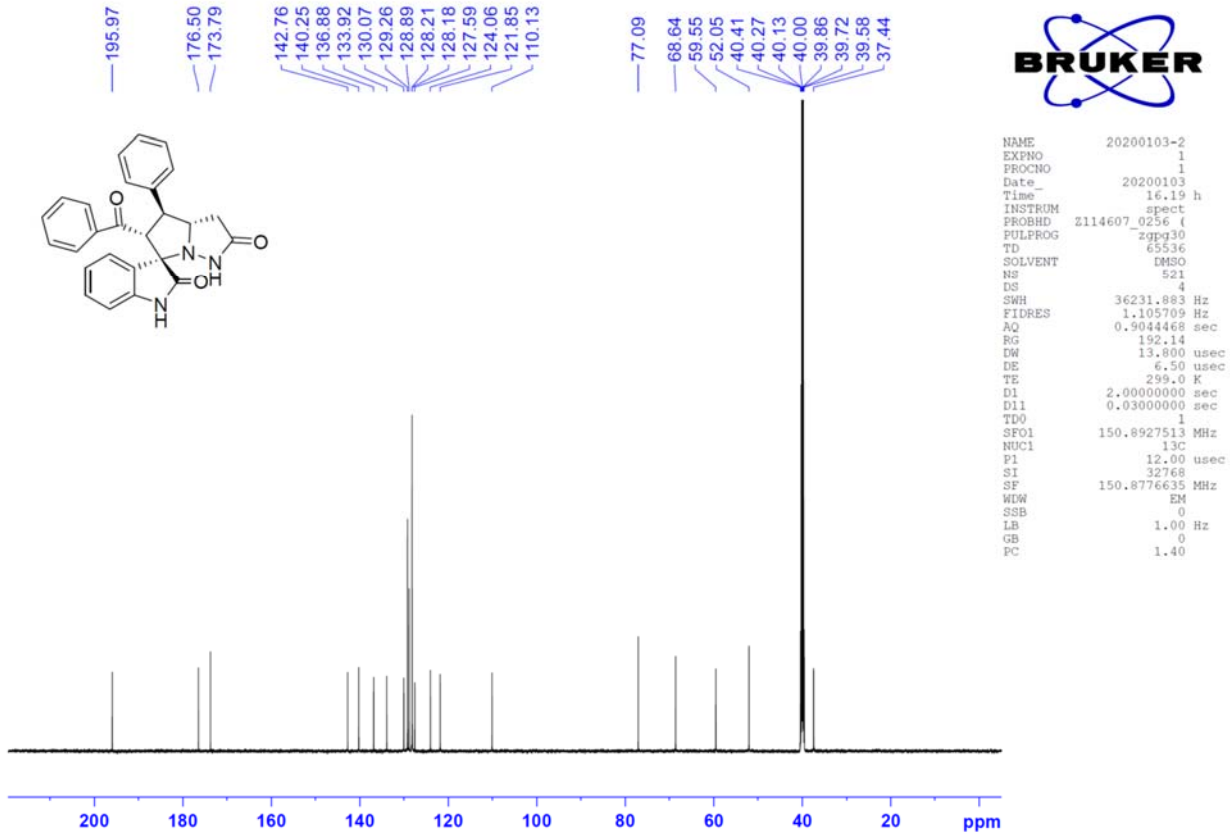




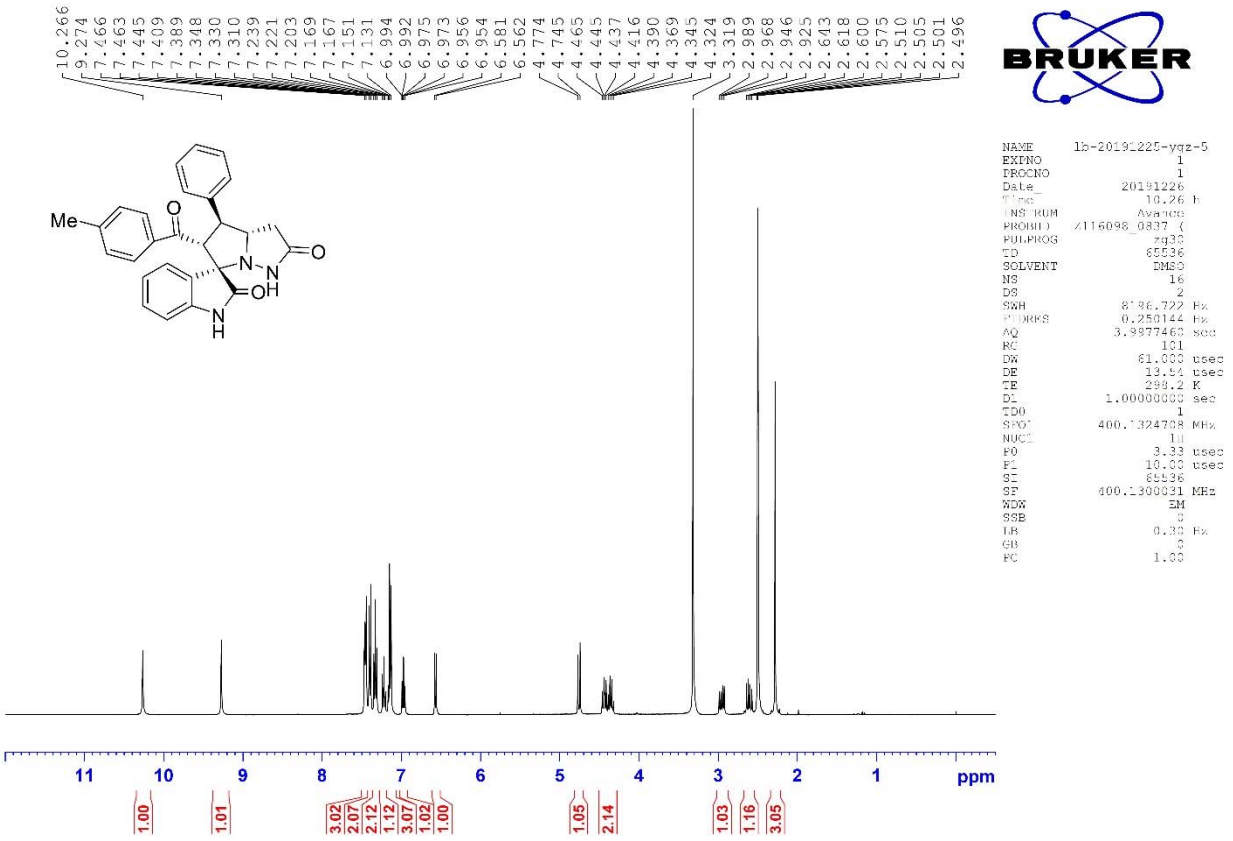


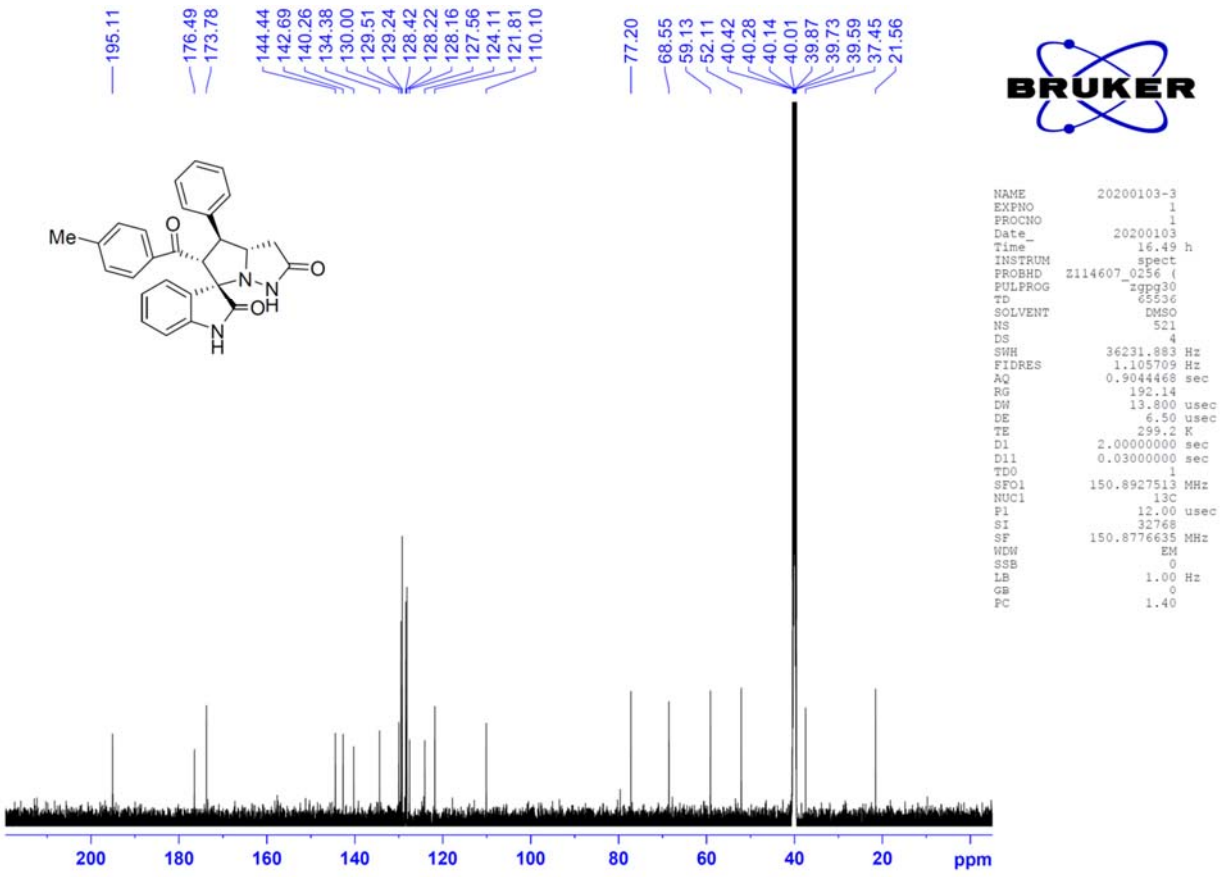
^{1}H NMR and ^{13}C NMR Spectra for Compound **5a**



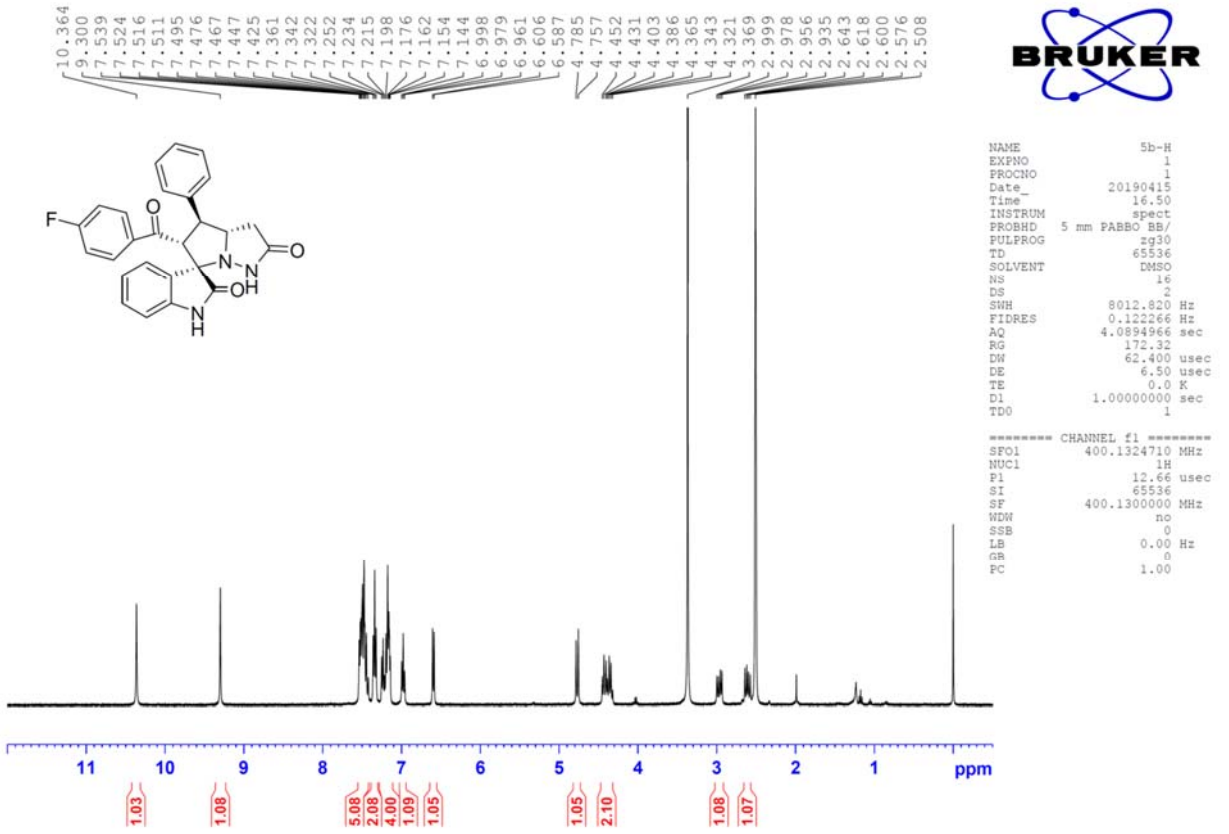


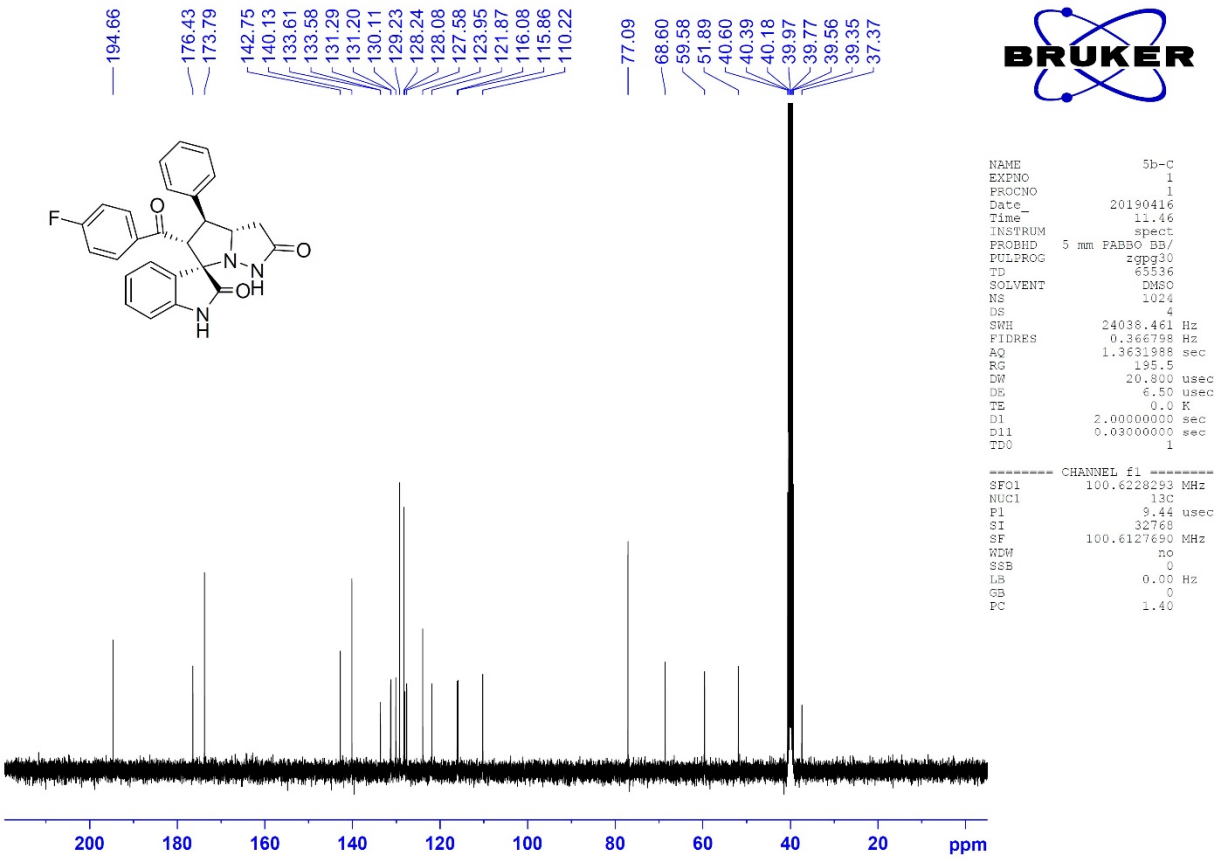
^{1}H NMR and ^{13}C NMR Spectra for Compound **5b**





^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **5c**



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PROCNO                 1
Date_           20190416
Time               11.46
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SOLVENT              DMSO
NS                   1024
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  195.5
DW                 20.800 usec
DE                  6.50 usec
TE                   0.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                   9.44 usec
SI                  32768
SF            100.6127690 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
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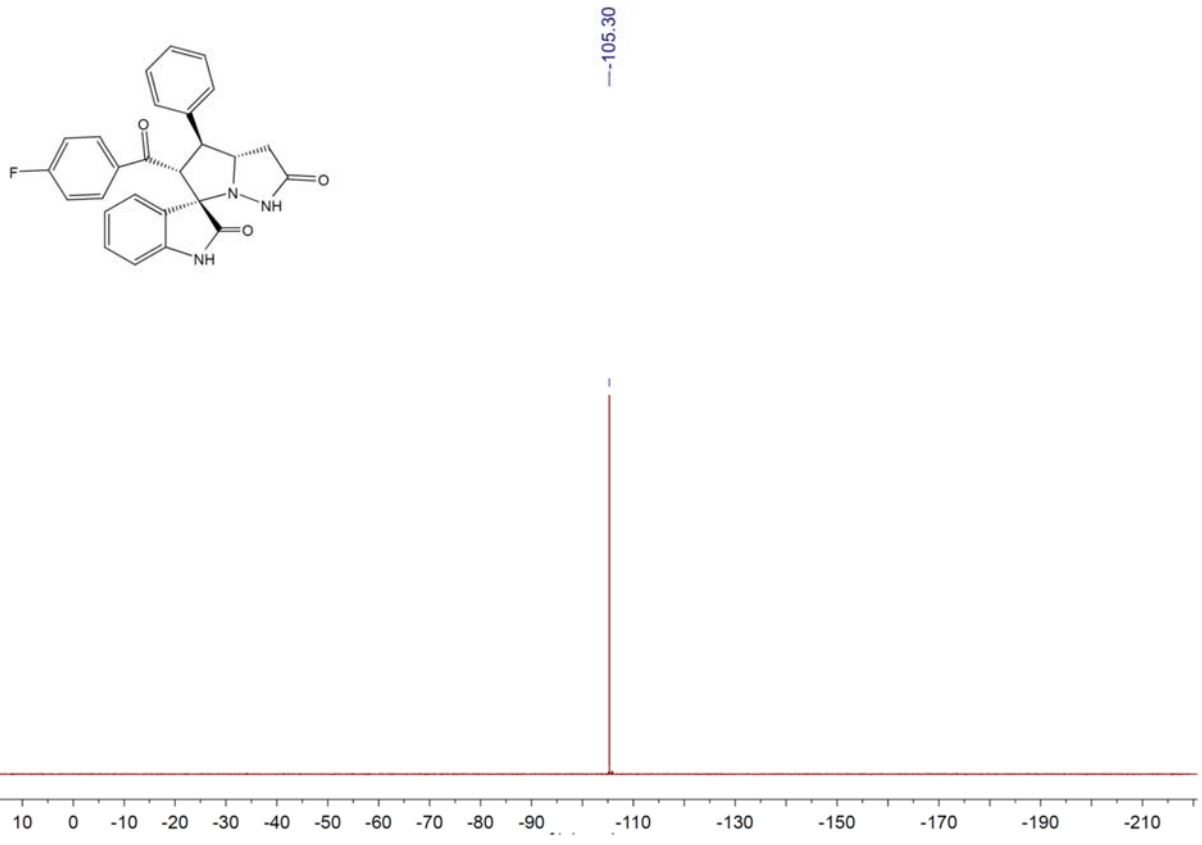
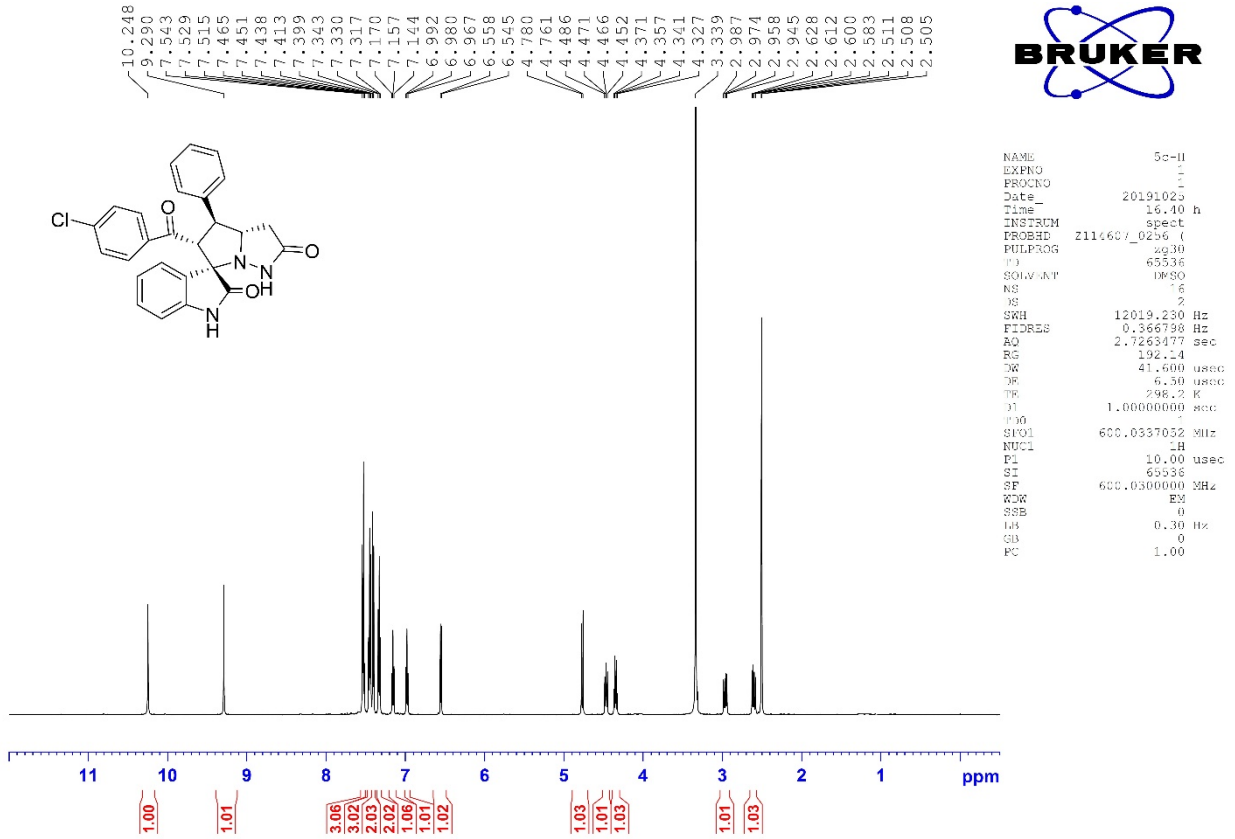
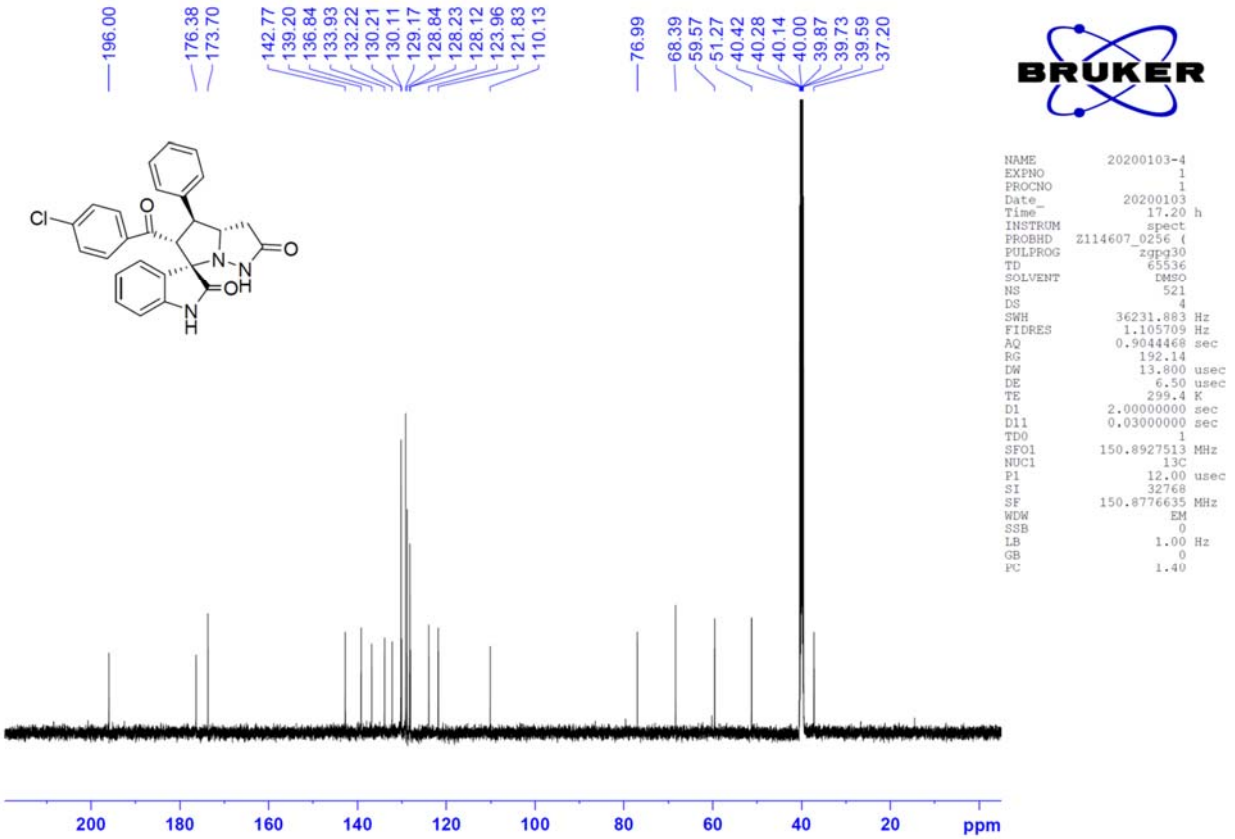
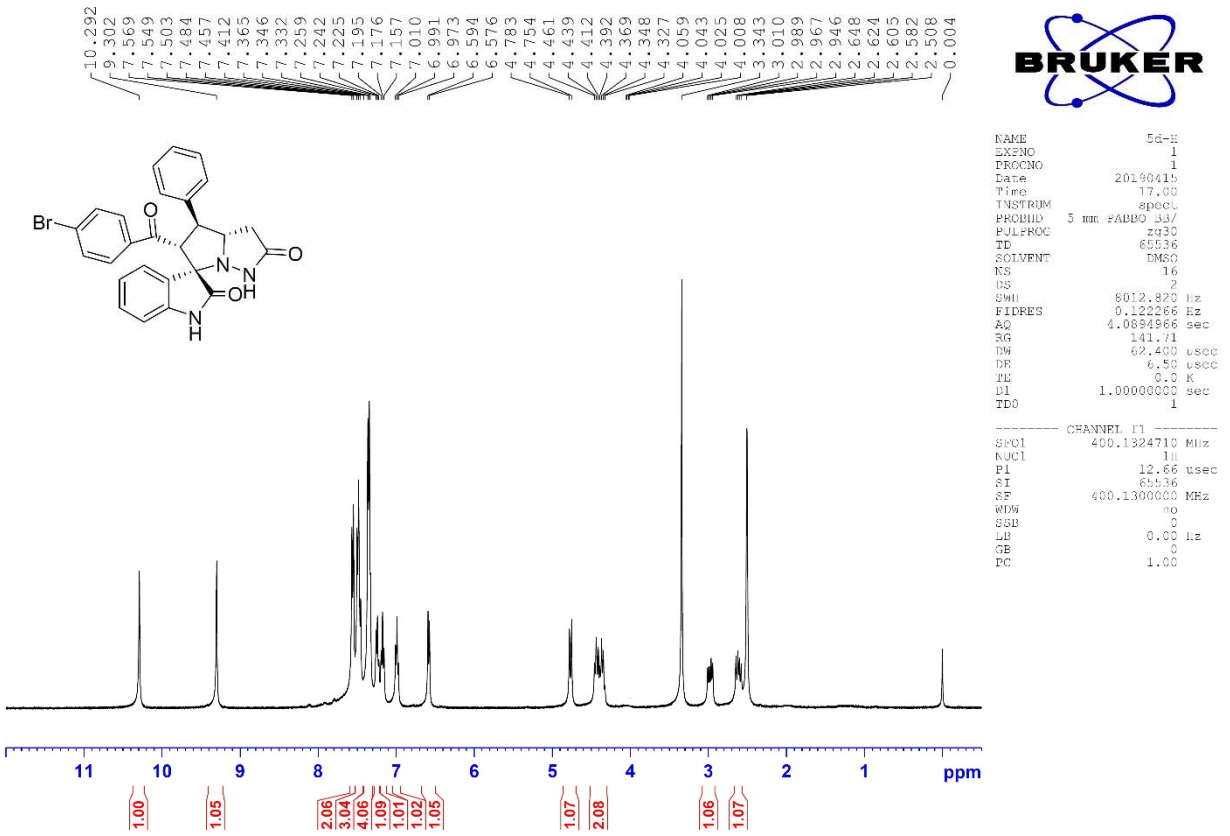


^{1}H NMR and ^{13}C NMR Spectra for Compound **5d**





^{1}H NMR and ^{13}C NMR Spectra for Compound **5e**

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PROCNO                   1
Date              20190415
Time                 17.00
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PULPROG              zg30
TD                  65536
SOLVENT               DMSO
NS                      16
DS                       2
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FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                   141.71
DW                   62.400 usec
DE                    6.50 usec
TE                     0.0 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
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NUC1                    1H
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SI                    65536
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WDW                     no
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LB                    0.00 Hz
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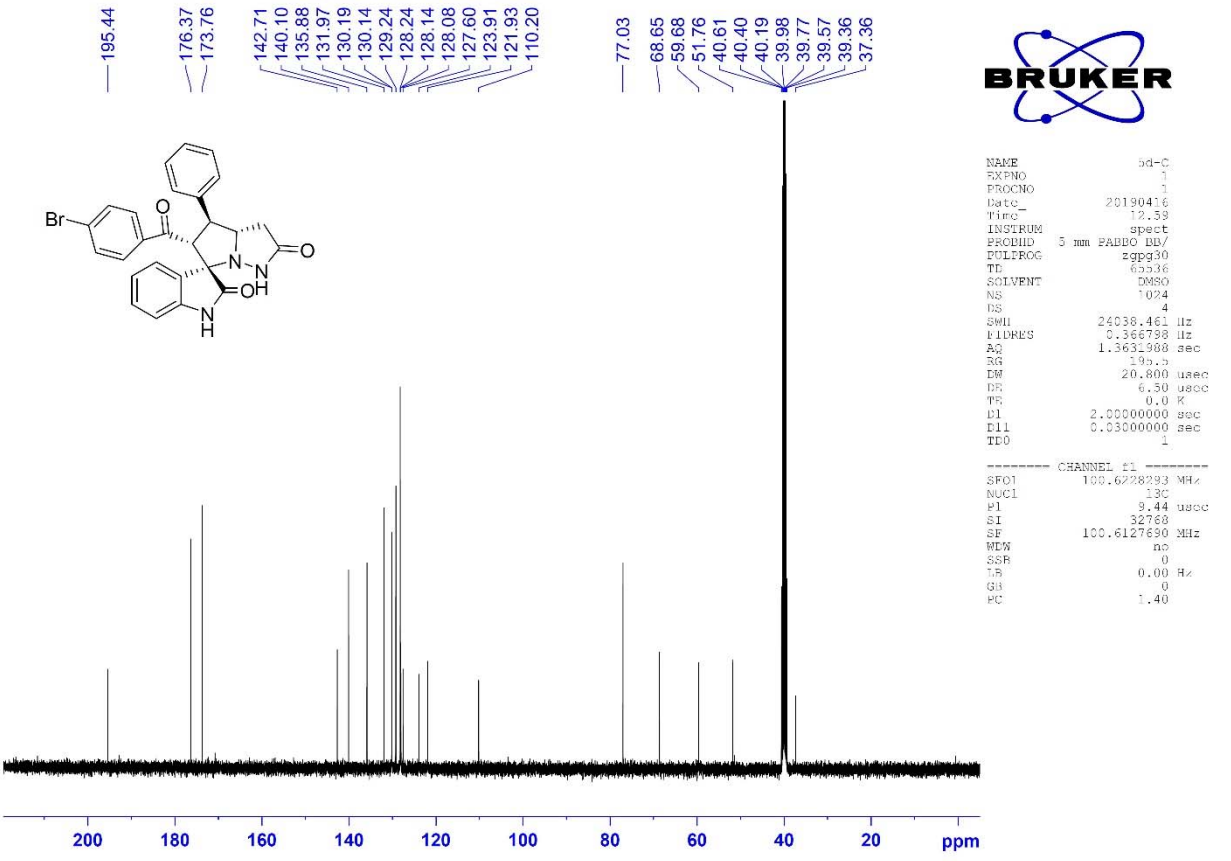
```

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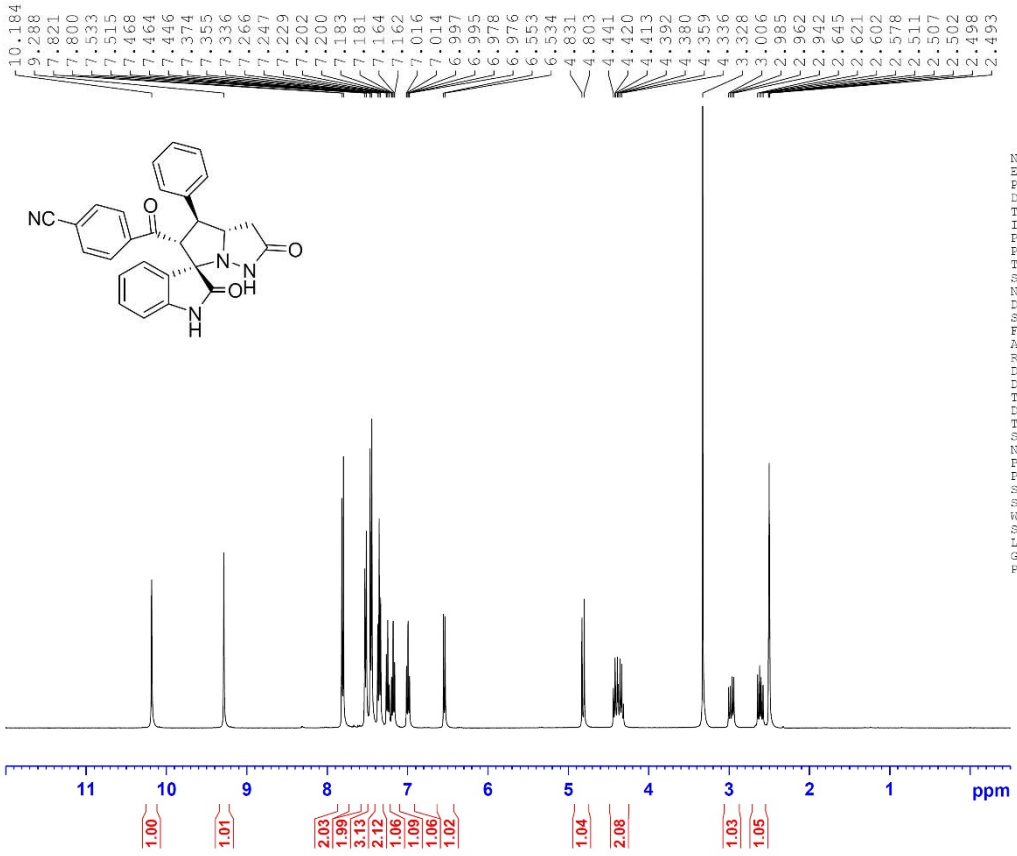
NAME                  5d-C
EXPNO                    1
PROCNO                   1
Date_             20190416
Time_                12.59
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT               DMSO
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                   285.0
DW                   20.800 usec
DE                    6.50 usec
TE                     0.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            100.6228293 MHz
NUC1                    13C
P1                    9.44 usec
SI                   32768
SF              100.6127690 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.40
```

^{1}H NMR and ^{13}C NMR Spectra for Compound **5f**



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PROCNO	1
Date_	20200102
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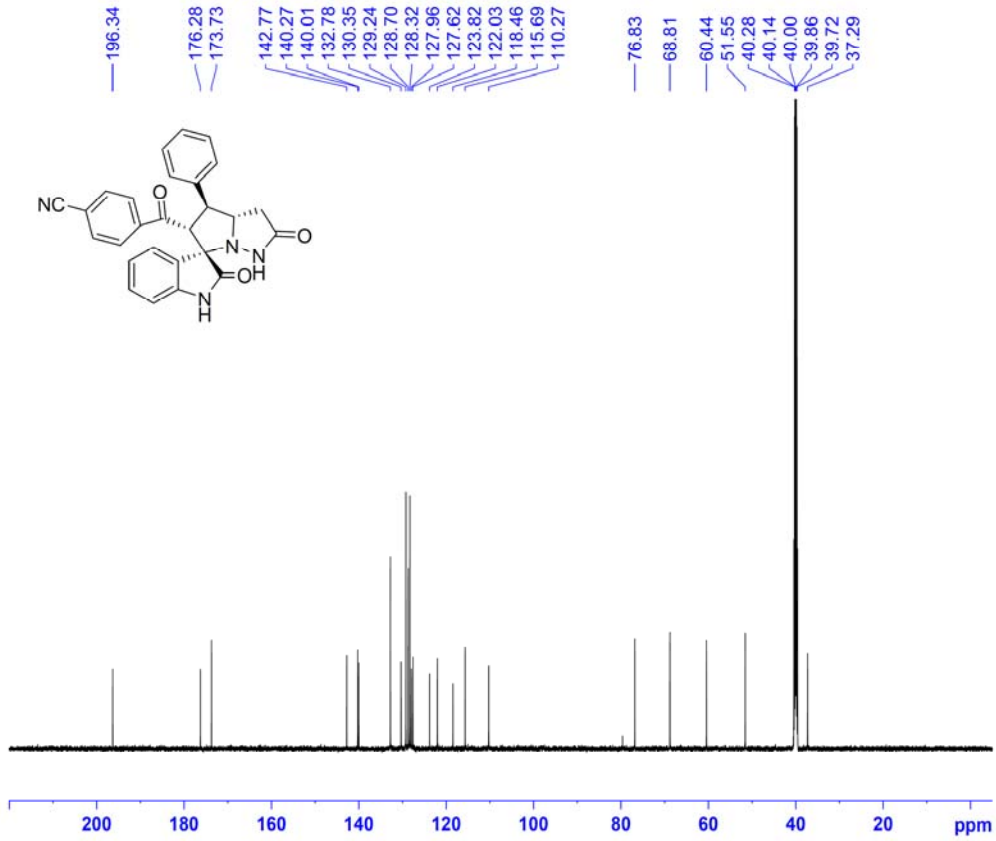
FIDRES	0.250144 Hz
AQ	3.9977460 sec
RG	101
DW	61.000 usec
DE	13.54 usec
TE	298.1 K
D1	1.00000000 sec
TD0	1
SFO1	400.1324708 MHz
NUC1	1H
P0	3.33 usec
P1	10.00 usec
SI	65536
SF	400.1300026 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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PROCNO	1
Date_	20200103
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SOLVENT	DMSO
NS	521
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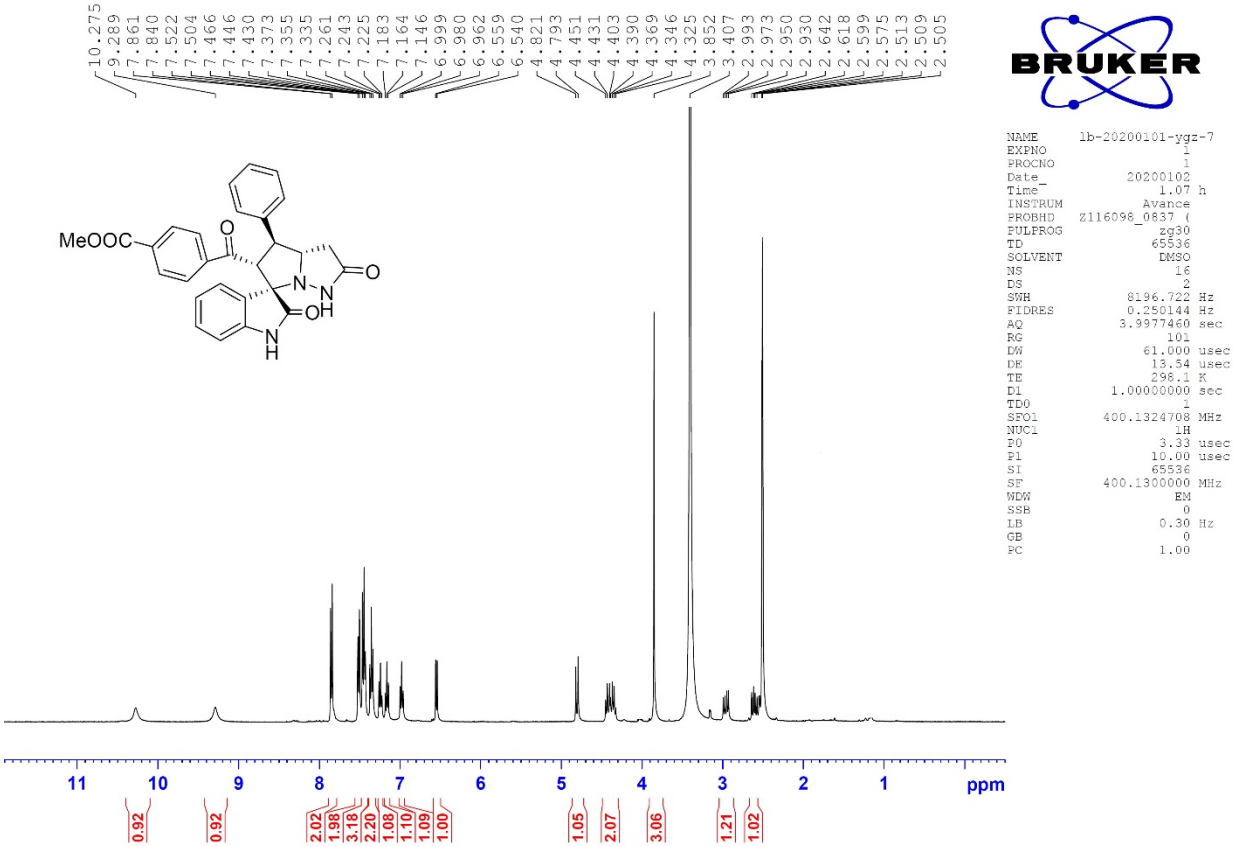
FIDRES	1.105709 Hz
AQ	0.9044468 sec
RG	192.14
DW	13.800 usec
DE	6.50 usec
TE	299.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	150.8927513 MHz
NUC1	13C
P1	12.00 usec
SI	32768
SF	150.8776635 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR and ^{13}C NMR Spectra for Compound **5g**



BRUKER

NAME	lb-20200101-ygz-7
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PROCNO	1
Date_	20200102
Time	1.07 h
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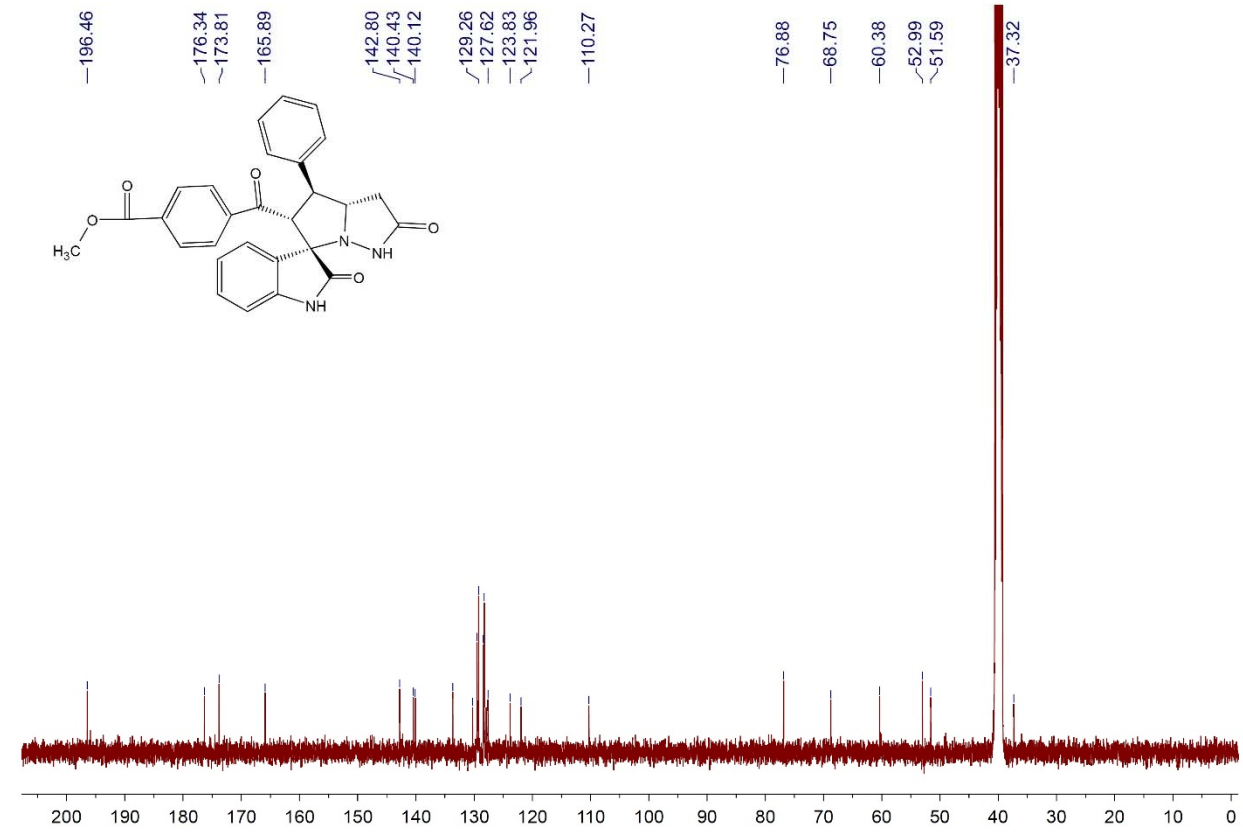
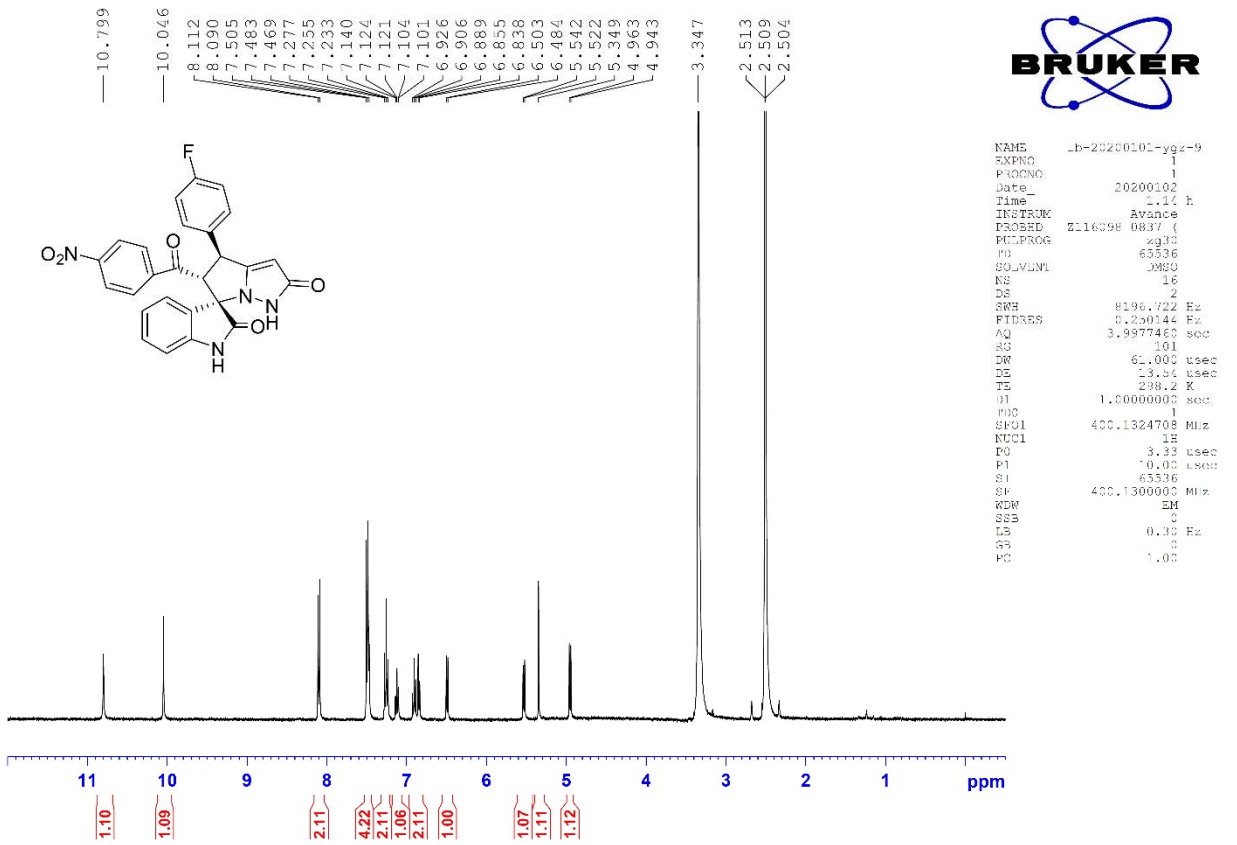
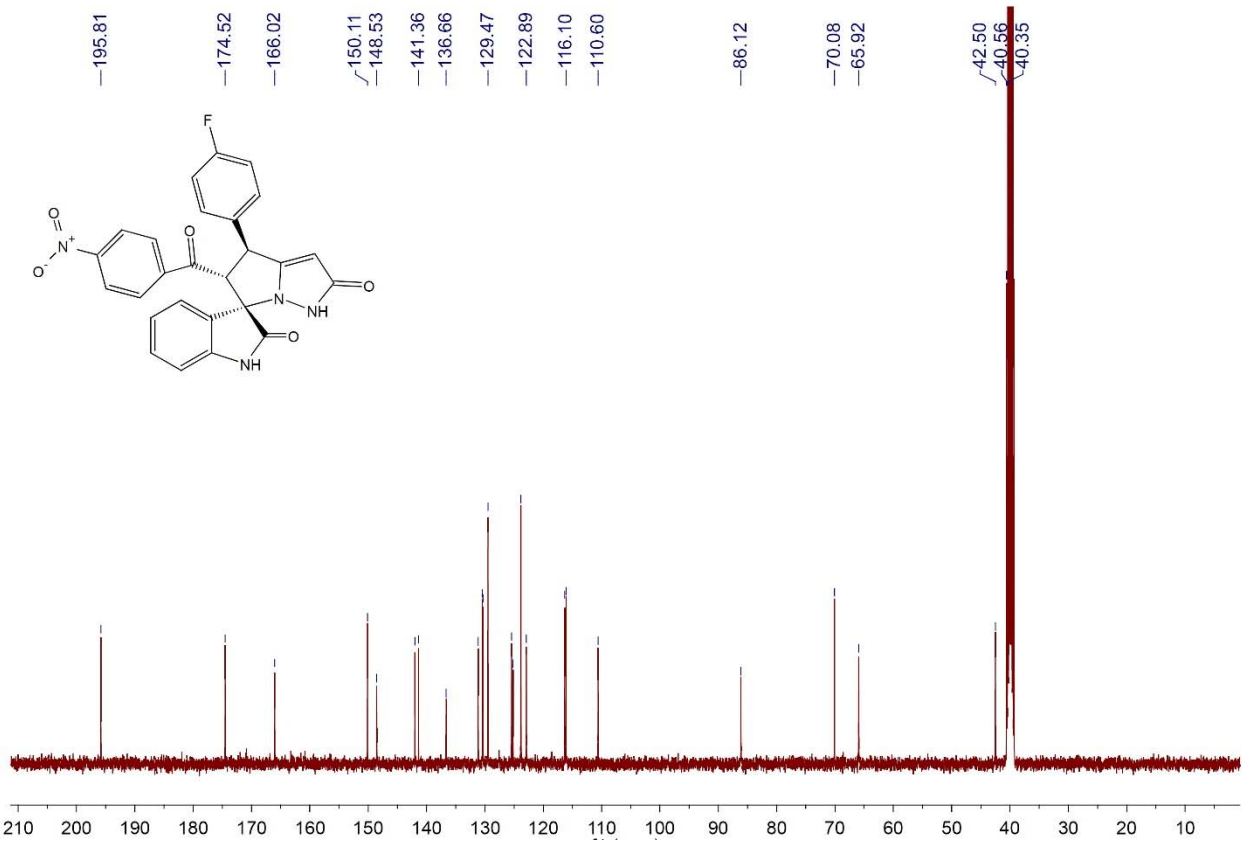
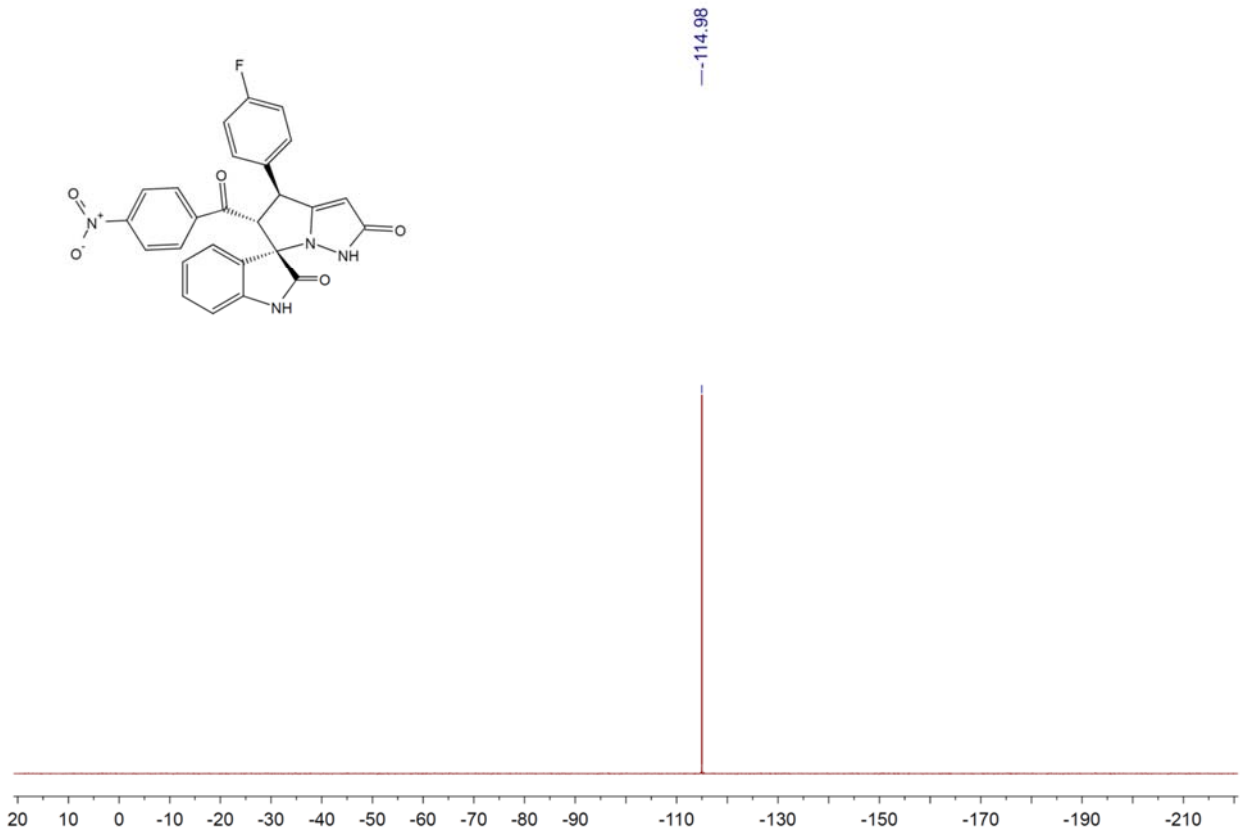
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	8196.722 Hz
FIDRES	0.250144 Hz
AQ	3.9977460 sec
RG	101
DW	61.000 usec
DE	13.54 usec
TE	298.1 K
D1	1.00000000 sec
TD0	1
SFO1	400.1324708 MHz
NUC1	1H
P0	3.33 usec
P1	10.00 usec
SI	65536
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



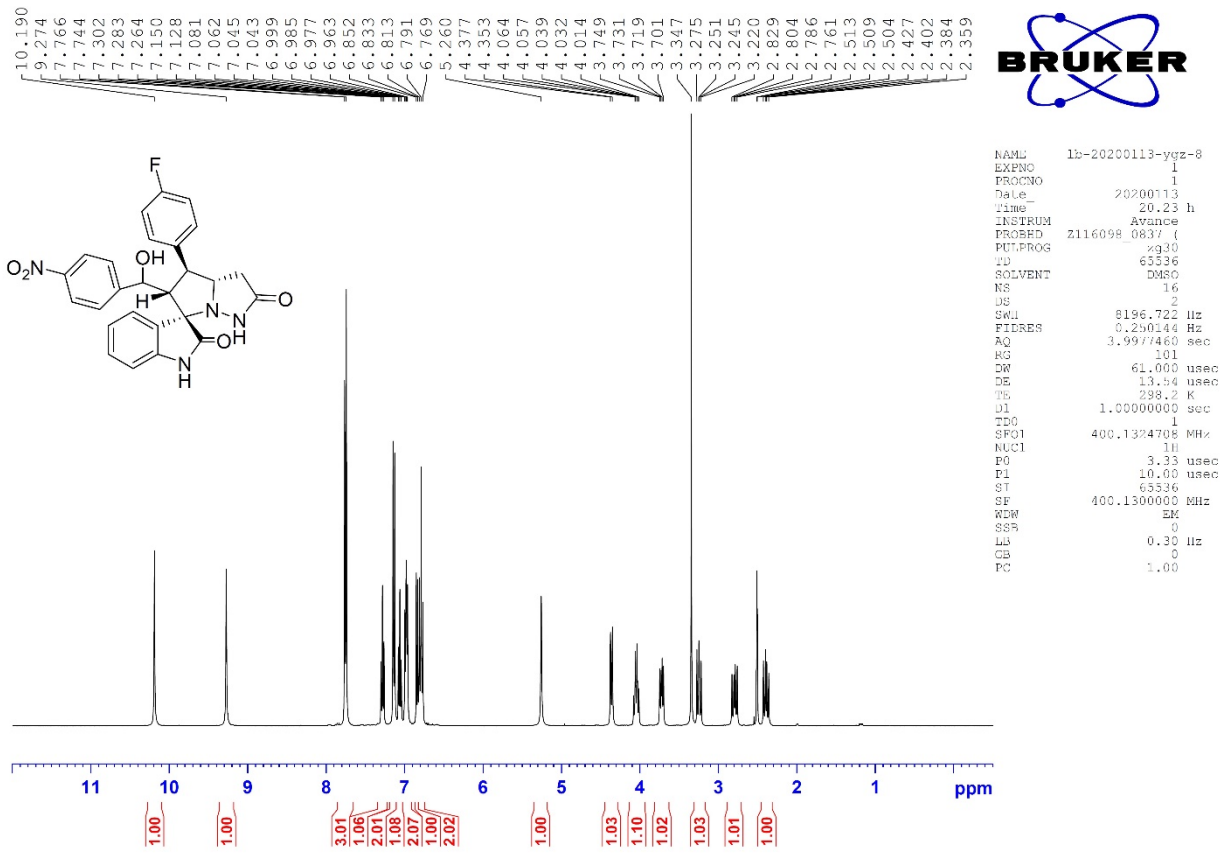
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **6a**

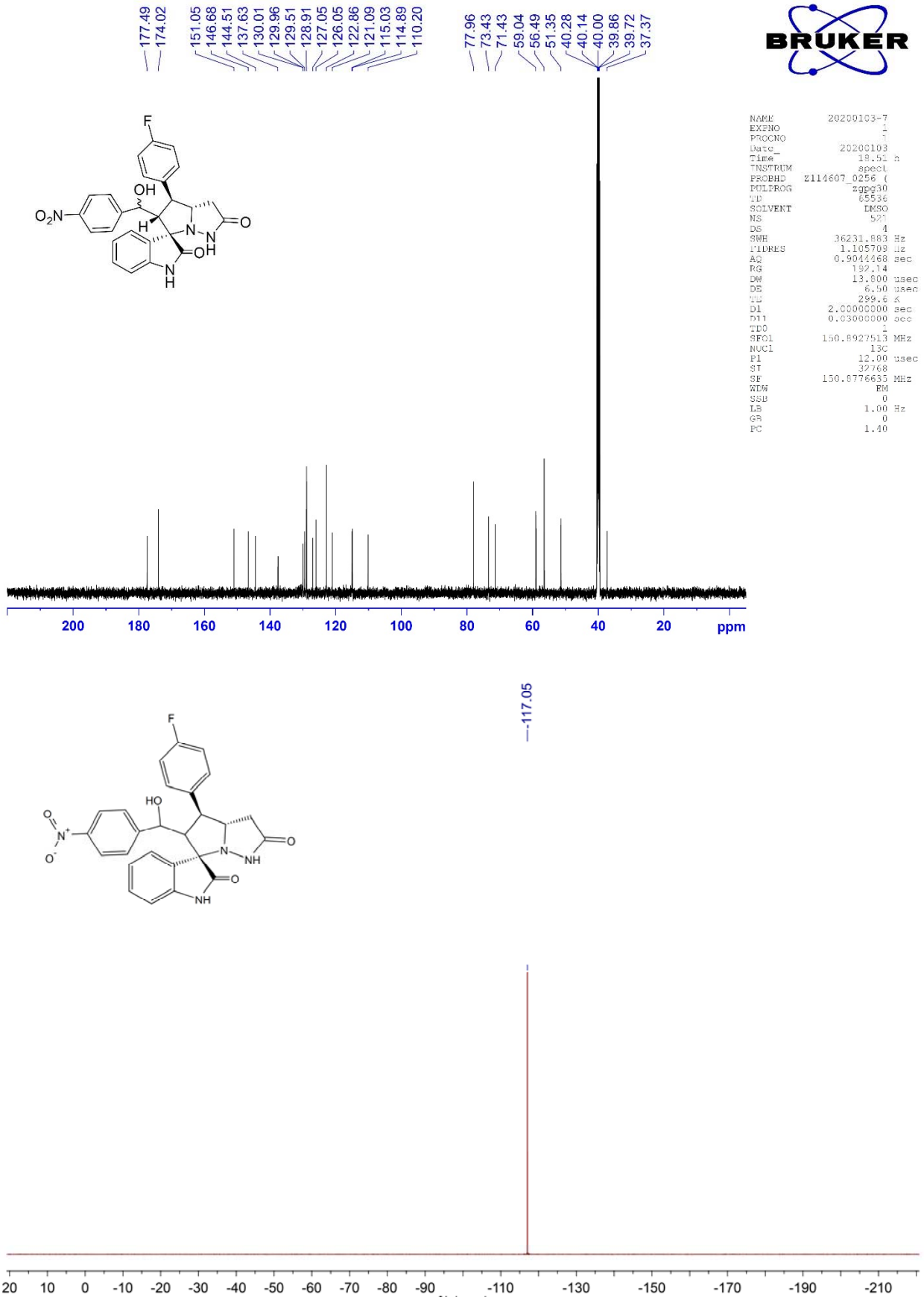






^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Compound **6b**





X. Data of X-ray crystal structure for 3t (CCDC 1973541).

checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

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: 2_a

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Bond precision:  C-C = 0.0027 A          Wavelength=0.71073

Cell:            a=14.0549(7)    b=8.6729(4)        c=20.6537(9)
                 alpha=90        beta=90.332(2)     gamma=90
Temperature:     273 K

                Calculated                Reported
Volume          2517.6(2)                 2517.6(2)
Space group     P 21/n                    P 1 21/n 1
Hall group      -P 2yn                    -P 2yn
Moiety formula  C29 H23 F N4 O5           C29 H23 F N4 O5
Sum formula     C29 H23 F N4 O5           C29 H23 F N4 O5
Mr              526.51                    526.51
Dx,g cm-3       1.389                     1.389
Z               4                         4
Mu (mm-1)       0.102                     0.102
F000            1096.0                    1096.0
F000'           1096.56
h,k,lmax        18,11,26                  18,11,26
Nref            5823                      5803
Tmin,Tmax       0.975,0.985               0.692,0.735
Tmin'           0.972

Correction method= # Reported T Limits: Tmin=0.692 Tmax=0.735
AbsCorr = MULTI-SCAN

Data completeness= 0.997          Theta(max)= 27.554

R(reflections)= 0.0533( 4523)     wR2(reflections)= 0.1390( 5803)

S = 1.015                 Npar= 352
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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

```
PLAT220_ALERT_2_C NonSolvent Resd 1  C   Ueq(max) / Ueq(min) Range        4.0 Ratio
PLAT242_ALERT_2_C Low    MainMol Ueq as Compared to Neighbors of         N1 Check
PLAT368_ALERT_2_C Short  C(sp2)-C(sp2) Bond  C28      - C29      .       1.23 Ang.
```

Alert level G

```
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms .............          1 Report
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K)       273 Check
PLAT200_ALERT_1_G Reported   _diffrn_ambient_temperature ..... (K)       273 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact  O4       ..C9             2.89 Ang.
                                              x,-1+y,z  =      1_545 Check
PLAT793_ALERT_4_G Model has Chirality at C7          (Centro SPGR)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C8          (Centro SPGR)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C16         (Centro SPGR)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C19         (Centro SPGR)          S Verify
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .     Please Do !
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   3 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   9 ALERT level G = General information/check it is not something unexpected

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

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 20/12/2019; check.def file version of 13/12/2019

Datablock 2_a - ellipsoid plot