Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

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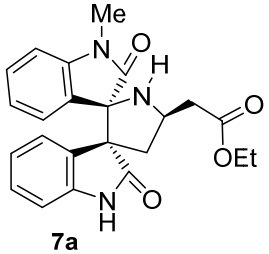
1. General information:

- Chemicals were purchased from Acros or Aldrich and used without further purification unless otherwise noted. Solvents were predistilled according to standard laboratory methods.
- Chromatographic purification of the products was performed on Merck silica gel 60, particle size 0.040-0.063 mm (230-240 mesh, flash).
- Analytical TLC: SIL G-25 UV254 from MACHEREY&NAGEL. Visualization of the developed TLC plates was performed with ultraviolet irradiation (254 nm) or by staining with basic potassium permanganate solution.
- Optical rotation values were measured on a Perkin-Elmer 241 polarimeter.
- Melting points were determined using a Büchi 510 apparatus and are uncorrected.
- Mass spectra were acquired on a Finnigan SSQ7000 (EI/CI) spectrometer and high resolution mass spectra on a Finnigan MAT 95 (EI/CI) or on a ThermoFisher Scientific LTQOrbitrap XL (ESI). All signals over 10% relative intensity are listed.
- IR spectra were taken on a Perkin-Elmer FT-IR Spectrum 100 using a Diamant/KRS5 ATR. Evaluation was done using the supplementary software. The absorption bands are given in wave numbers (cm^{-1}).
- ^{1}H- and ^{13}C- NMR spectra were recorded at ambient temperature on Varian Mercury 300, VNMRS 600 and Inova 400 instruments. The chemical shifts are reported in ppm downfield of tetramethylsilane (TMS) and referenced to residual solvent peaks resonance as internal standard. The order of citation in parentheses is a) multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd= doublet of doublet, ddd= doublet of doublet of doublet, td = triplet of doublet, m = multiplet), b) coupling constants, c) number of protons. Coupling constants (*J*) are reported in Hertz (Hz).
- Analytical HPLC was performed on a Hewlett-Packard 1100 Series instrument using chiral stationary phases (CHIRALPAK IB, CHIRALPAK AD, (s, s)-Whelk Ol).
- Isatin-derived ketimines **5** and 3-substituted oxindoles **6** were synthesized according to the literature. [1-2]

2. General Procedure for the synthesis of products 7:

A 10 mL flask equipped with a stirring bar is charged with 3-substituted oxindole **6** (0.40 mmol, 1.0 equiv), the ketimine **5** (0.42 mmol, 1.05 equiv), catalyst **F** (0.04 mmol, 10 mol %) and MTBE (4.0 mL). The resulting solution is stirred at room temperature for 12 h. The solvent is evaporated to give the residue, which is dissolved in DCM (4.0 mL) and TFA (0.8 mL) is added at 0 ^{o}C. The mixture is stirred at room temperature for 12 h, and then saturated $NaHCO_3$ solution is carefully added to quench the reaction. The resulting solution is extracted with EtOAc (3×10.0 mL). The organic layers are combined, washed with brine and dried over anhydrous Na_2SO_4. The solvent is evaporated in *vacuo* to give the crude product, which is purified by flash chromatography (pentane/EtOAc = 2/1 to 1/2) to provide the desired product **7**.



7a

Ethyl 2-((3*R*,3'*R*,5'*R*)-1-methyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl) acetate (7a)

According to the general procedure, **7a** was obtained as a colorless solid (131.2 mg, 81% yield).

Melting Point: 114-116 ^{o}C.

$[\alpha]_D^{25}$ = –89.2 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK AD; *n*-heptane/EtOH = 7/3; flow rate 1.0 mL/min; T= 30 ^{o}C; retention time: 13.34 min (major), 11.30 min (minor), er: 99:1.

^{1}H NMR (600 MHz, $CDCl_3$): δ = 8.05 (s, 1H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.18 – 7.13 (m, 2H), 6.99 (t, *J* = 7.8 Hz, 1H), 6.75 – 6.67 (m, 3H), 6.60 (d, *J* = 7.8 Hz, 1H), 5.00 – 4.96 (m, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 3.22 (dd, *J* = 13.2, 9.6 Hz, 1H), 3.02 (s, 3H), 2.88 (dd, *J* = 15.6, 9.6 Hz, 1H), 2.77 (dd, *J* = 15.6, 5.4 Hz, 1H), 2.03 (dd, *J* = 13.2, 4.8 Hz, 1H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 176.9, 176.8, 171.8, 144.1, 140.4, 131.3, 129.4, 128.5, 126.3, 126.1, 125.4, 121.8, 121.6, 109.6, 107.6, 73.8, 60.6, 60.1, 51.5, 41.7, 39.4, 26.0, 14.3.

IR (ATR): 3276, 2942, 1948, 1716, 1609, 1469, 1343, 1255, 1184, 1110, 1012, 867, 748 cm^{-1}.

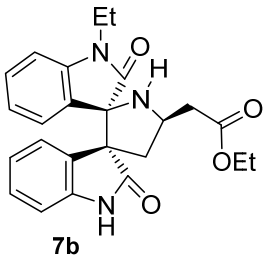
MS (ESI): *m/z* = 406.2 $[M+H]^+$, 428.2 $[M+Na]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{23}H_{24}N_3O_4^+$: 406.1761; found 406.1770.

The gram scale reaction for the synthesis of **7a** was carried out in a similar manner.

A 100 mL flask equipped with a stirring bar was charged with **6a** (980 mg, 4.0 mmol, 1.0 equiv), the ketimine **5a** (1.12 g, 4.2 mmol, 1.05 equiv), catalyst **F** (311 mg, 0.4 mmol, 10 mol %) and MTBE (40 mL). The resulting solution was stirred at room temperature for 24 h. The solvent was evaporated to give the residue, which was dissolved in DCM (40 mL) and TFA (8 mL) was added at 0 ^{o}C. The mixture was stirred at room temperature for 20 h, and then saturated $NaHCO_3$ solution was carefully added to quench the reaction. The resulting solution was extracted with EtOAc (3×50.0 mL). The organic layers were combined, washed with brine and dried over anhydrous Na_2SO_4. The solvent was evaporated in *vacuo* to give the crude product, which was purified by flash chromatography (pentane/EtOAc = 2/1 to 1/1) to provide the desired product **7a**

as a colorless solid (1.22 g, 75% yield, er: 96:4). The analytical data of the gram scale reaction of **7a** are consistent with those of the 0.40 mmol scale experiment.



7b

Ethyl 2-((3*R*,3'*R*,5'*R*)-1-ethyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl) acetate (7b)

According to the general procedure, **7b** was obtained as a colorless solid (122.3 mg, 73% yield).

Melting Point: 106-107 °C.

$[\alpha]_D^{25}$ = –86.1 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK IB; *n*-heptane/EtOH = 7/3; flow rate 1.0 mL/min; T= 30 °C; retention time: 7.27 min (major), 6.31 min (minor), er: 96:4.

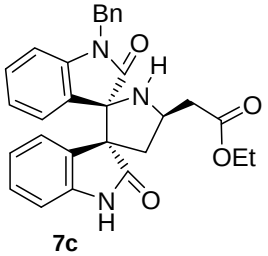
^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.99 (s, 1H), 7.31 (d, *J* = 7.2 Hz, 1H), 7.15 (t, *J* = 7.8 Hz, 2H), 6.94 (t, *J* = 7.8 Hz, 1H), 6.78 (d, *J* = 7.2 Hz, 1H), 6.75 – 6.68 (m, 2H), 6.61 (d, *J* = 7.8 Hz, 1H), 5.01 – 4.97 (m, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.81 – 3.72 (m, 1H), 3.44 – 3.38 (m, 1H), 3.17 (dd, *J* = 13.2, 9.6 Hz, 1H), 3.01 (s, 1H), 2.89 (dd, *J* = 16.2, 9.0 Hz, 1H), 2.78 (dd, *J* = 16.2, 5.4 Hz, 1H), 2.07 (dd, *J* = 13.2, 5.4 Hz, 1H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.03 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 177.0, 176.2, 171.8, 143.1, 140.5, 130.8, 129.3, 128.4, 126.9, 125.9, 125.5, 121.7, 121.5, 109.4, 107.7, 73.8, 60.6, 60.5, 51.8, 41.8, 39.3, 34.4, 14.3, 12.2.

IR (ATR): 3309, 2976, 2299, 2049, 1715, 1609, 1468, 1363, 1293, 1187, 1024, 854, 747, 679 cm^{-1}.

MS (ESI): *m/z* = 420.2 $[M+H]^+$, 442.2 $[M+Na]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{24}H_{26}N_3O_4^+$: 420.1918; found 420.1909.



7c

Ethyl 2-((3*R*,3'*R*,5'*R*)-1-benzyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl) acetate (7c)

According to the general procedure, **7c** was obtained as a colorless solid (167.4 mg, 87% yield).

Melting Point: 120-121 °C.

$[\alpha]_D^{25}$ = –55.7 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK AD; *n*-heptane/EtOH = 7/3; flow rate 1.0 mL/min; T= 30 °C; retention time: 23.34 min (major), 11.96 min (minor), er: 98:2.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (s, 1H), 7.19 – 7.11 (m, 2H), 7.06 – 6.96 (m, 4H), 6.96 – 6.85 (m, 4H), 6.79 (td, *J* = 7.6, 0.8 Hz, 1H), 6.67 (d, *J* = 7.6 Hz, 1H), 6.38 (d, *J* = 7.6 Hz, 1H), 5.15 (d, *J* =

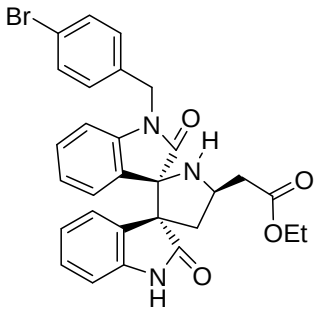
16.0 Hz, 1H), 5.03 – 4.96 (m, 1H), 4.32 (d, *J* = 16.0 Hz, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.13 (dd, *J* = 13.2, 8.8 Hz, 1H), 2.91 (dd, *J* = 16.0, 9.2 Hz, 1H), 2.78 (dd, *J* = 16.0, 5.6 Hz, 1H), 2.17 (dd, *J* = 13.2, 6.4 Hz, 1H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 177.4, 176.4, 171.8, 143.2, 140.7, 135.3, 129.9, 129.4, 128.5 (2C), 128.4, 127.5, 127.2, 126.6 (2C), 125.7, 125.5, 121.9, 121.8, 109.8, 109.0, 73.9, 60.7, 60.6, 52.1, 43.7, 41.9, 39.8, 14.2.

IR (ATR): 3297, 2973, 2289, 2163, 1898, 1719, 1475, 1355, 1219, 1247, 1021, 826, 746 cm^{-1}.

MS (ESI): *m/z* = 482.2 $[M+H]^+$, 985.4 $[2M+Na]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{29}H_{28}N_3O_4^+$: 482.2074; found 482.2067.



7d

Ethyl 2-((3*R*,3'*R*,5'*R*)-1-(4-bromobenzyl)-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7d)

According to the general procedure, **7d** was obtained as a colorless solid (187.8 mg, 84% yield).

Melting Point: 140-142 °C.

$[\alpha]_D^{25}$ = –34.0 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK IB; *n*-heptane/EtOH = 7/3; flow rate 1.0 mL/min; T= 30 °C; retention time: 14.72 min (major), 11.36 min (minor), er: 98:2.

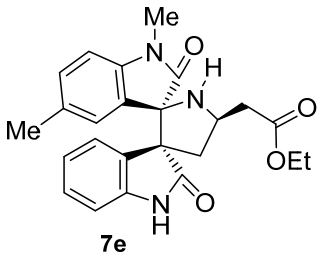
^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.64 (s, 1H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.19 (t, *J* = 7.8 Hz, 1H), 7.08 – 7.02 (m, 2H), 7.00 (d, *J* = 7.8 Hz, 1H), 6.87 (t, *J* = 7.8 Hz, 1H), 6.83 (t, *J* = 7.8 Hz, 1H), 6.79 (d, *J* = 8.4 Hz, 2H), 6.74 (d, *J* = 7.8 Hz, 1H), 6.36 (d, *J* = 7.8 Hz, 1H), 5.08 (d, *J* = 16.2 Hz, 1H), 5.03 – 4.98 (m, 1H), 4.28 (d, *J* = 16.2 Hz, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 3.08 (dd, *J* = 13.2, 9.0 Hz, 1H), 2.92 (dd, *J* = 16.2, 9.0 Hz, 1H), 2.80 (dd, *J* = 16.2, 5.4 Hz, 1H), 2.23 (dd, *J* = 13.2, 6.6 Hz, 1H), 1.28 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 176.8, 176.0, 171.7, 142.8, 140.7, 134.4, 131.7 (2C), 129.4, 129.3, 128.6 (2C), 128.4, 128.0, 125.7, 125.5, 122.2, 121.9, 121.2, 109.5, 108.9, 74.0, 60.9, 60.6, 52.4, 43.3, 41.9, 39.8, 14.3.

IR (ATR): 3327, 2997, 2168, 2040, 1733, 1613, 1456, 1366, 1213, 1017, 903, 745 cm^{-1}.

MS (ESI): *m/z* = 562.1 $[M+H]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{29}H_{27}N_3O_4Br^+$: 560.1180; found 560.1166.



7e

Ethyl 2-((3*R*,3'*R*,5'*R*)-1,5-dimethyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7e)

According to the general procedure, **7e** was obtained as a colorless solid (125.7 mg, 75% yield).

Melting Point: 131-133 °C.

$[\alpha]_D^{25}$ = –75.1 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK IB; *n*-heptane/EtOH = 7/3; flow rate 0.7 mL/min; T= 30 °C; retention time: 11.18 min (major), 9.93 min (minor), er: 97:3.

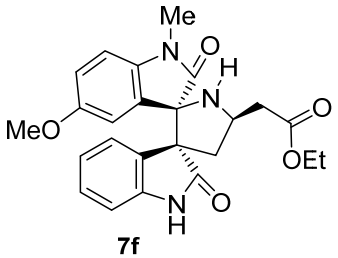
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.69 (s, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.16 (td, *J* = 7.6, 1.2 Hz, 1H), 6.99 (t, *J* = 7.6 Hz, 1H), 6.92 (d, *J* = 7.6 Hz, 1H), 6.68 (d, *J* = 7.6 Hz, 1H), 6.48 – 6.45 (m, 2H), 5.02 – 4.94 (m, 1H), 4.14 (q, *J* = 7.2 Hz, 2H), 3.22 (dd, *J* = 13.2, 9.6 Hz, 1H), 3.02 (s, 3H), 2.94 (bs, 1H), 2.87 (dd, *J* = 16.0, 9.2 Hz, 1H), 2.76 (dd, *J* = 16.0, 5.2 Hz, 1H), 2.08 – 1.98 (m, 4H), 1.25 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.8, 176.7, 171.8, 141.7, 140.4, 131.4, 131.0, 129.5, 128.4, 126.4, 126.2, 126.1, 121.7, 109.5, 107.2, 74.0, 60.5, 60.0, 51.5, 41.7, 39.2, 26.0, 20.8, 14.2.

IR (ATR): 3258, 2925, 2320, 2163, 1989, 1719, 1612, 1468, 1345, 1293, 1245, 1178, 1030, 880, 818, 750, 686 cm^{-1}.

MS (ESI): *m/z* = 420.2 $[M+H]^+$, 442.2 $[M+Na]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{24}H_{26}N_3O_4^+$: 420.1918; found 420.1912.



7f

Ethyl 2-((3*R*,3'*R*,5'*R*)-5-methoxy-1-methyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7f)

According to the general procedure, **7f** was obtained as a colorless solid (137.5 mg, 79% yield).

Melting Point: 152-153 °C.

$[\alpha]_D^{25}$ = –84.2 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK IB; *n*-heptane/EtOH = 8/2; flow rate 0.7 mL/min; T= 30 °C; retention time: 21.23 min (major), 18.94 min (minor), er: 99:1.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.66 – 7.52 (m, 2H), 7.18 (t, *J* = 7.6 Hz, 1H), 7.04 (t, *J* = 7.6 Hz, 1H), 6.70 (d, *J* = 7.6 Hz, 1H), 6.66 (dd, *J* = 8.4, 2.8 Hz, 1H), 6.49 (d, *J* = 8.4 Hz, 1H), 6.23 (d, *J* = 2.4 Hz, 1H), 5.03 – 4.96 (m, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 3.42 (s, 3H), 3.28 (dd, *J* = 13.2, 10.0 Hz, 1H),

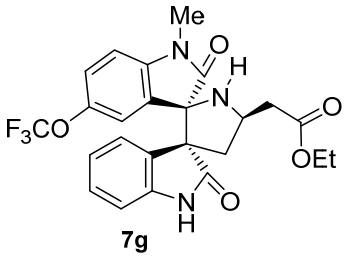
3.03 (s, 3H), 2.87 (dd, *J* = 16.0, 9.2 Hz, 1H), 2.76 (dd, *J* = 16.0, 5.2 Hz, 1H), 1.99 (dd, *J* = 13.2, 4.8 Hz, 1H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.7, 176.5, 171.8, 155.0, 140.3, 137.6, 131.8, 128.5, 126.7, 126.3, 121.7, 115.1, 111.9, 109.6, 107.9, 74.0, 60.6, 59.8, 55.7, 51.3, 41.6, 39.1, 26.0, 14.3.

IR (ATR): 3278, 2954, 2231, 1715, 1464, 1361, 1208, 1091, 859, 750 cm^{-1}.

MS (ESI): *m/z* = 436.2 $[M+H]^+$, 458.2 $[M+Na]^+$.

HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{24}H_{25}N_3O_5Na^+$: 458.1686; found 458.1672.



7g

Ethyl 2-((3*R*,3'*R*,5'*R*)-1-methyl-2,2''-dioxo-5-(trifluoromethoxy)dispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7g)

According to the general procedure, **7g** was obtained as a colorless solid (123.2 mg, 63% yield).

Melting Point: 107-109 °C.

$[\alpha]_D^{25}$ = –105.1 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK IB; *n*-heptane/EtOH = 8/2; flow rate 0.7 mL/min; T= 30 °C; retention time: 20.61 min (major), 15.26 min (minor), er: 98:2.

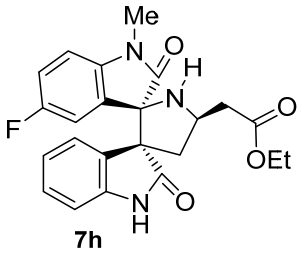
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.73 (s, 1H), 7.61 (d, *J* = 7.2 Hz, 1H), 7.19 (td, *J* = 7.6, 0.8 Hz, 1H), 7.06 (td, *J* = 7.6, 1.2 Hz, 1H), 6.98 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.70 (d, *J* = 8.0 Hz, 1H), 6.56 (d, *J* = 8.8 Hz, 1H), 6.48 (d, *J* = 1.2 Hz, 1H), 5.01 – 4.94 (m, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 3.29 (dd, *J* = 13.2, 9.6 Hz, 1H), 3.05 (s, 3H), 2.91 (s, 1H), 2.86 (dd, *J* = 16.0, 9.2 Hz, 1H), 2.75 (dd, *J* = 16.0, 5.2 Hz, 1H), 1.96 (dd, *J* = 13.2, 4.8 Hz, 1H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 177.1, 176.4, 171.8, 143.7, 142.8, 140.0, 131.4, 128.9, 127.0, 126.1, 122.6, 122.2, 119.7, 109.7, 107.7, 73.6, 60.6, 59.7, 51.2, 41.4, 39.0, 26.1, 14.2.

IR (ATR): 3305, 2945, 2175, 1720, 1619, 1471, 1351, 1226, 818, 749, 677 cm^{-1}.

MS (ESI): *m/z* = 490.2 $[M+H]^+$, 979.3 $[2M+H]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{24}H_{23}N_3O_5F_3^+$: 490.1584; found 490.1572.



7h

Ethyl 2-((3*R*,3'*R*,5'*R*)-5-fluoro-1-methyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7h)

According to the general procedure, **7h** was obtained as a colorless solid (113.4 mg, 67% yield).

Melting Point: 120-122 °C.

$[\alpha]_D^{25}$ = –107.1 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK IB; *n*-heptane/EtOH = 8/2; flow rate 1.0 mL/min; T= 30 °C; retention time: 15.00 min (major), 13.41 min (minor), er: 99:1.

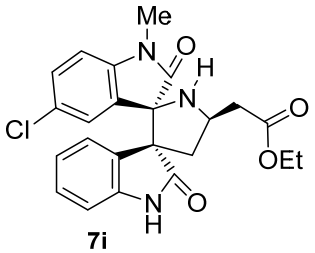
^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.78 (s, 1H), 7.55 (d, *J* = 7.8 Hz, 1H), 7.21 (t, *J* = 7.2 Hz, 1H), 7.05 (t, *J* = 7.8 Hz, 1H), 6.84 (td, *J* = 8.4, 2.4 Hz, 1H), 6.74 (d, *J* = 7.8 Hz, 1H), 6.52 (dd, *J* = 8.4, 4.2 Hz, 1H), 6.40 (dd, *J* = 9.0, 2.4 Hz, 1H), 5.01 – 4.96 (m, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.27 (dd, *J* = 13.2, 10.2 Hz, 1H), 3.06 (s, 3H), 2.87 (dd, *J* = 16.2, 9.6 Hz, 1H), 2.76 (dd, *J* = 16.2, 5.4 Hz, 1H), 1.99 (dd, *J* = 13.2, 4.8 Hz, 1H), 1.27 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 176.8, 176.5, 171.8, 158.9 (d, *J* = 238.4 Hz), 140.2, 140.1, 131.2, 128.8, 127.6 (d, *J* = 8.0 Hz), 126.1, 122.1, 115.6 (d, *J* = 23.4 Hz), 113.8 (d, *J* = 25.7 Hz), 109.7, 107.9 (d, *J* = 7.8 Hz), 73.8, 60.6, 59.8, 51.4, 41.5, 39.2, 26.2, 14.3.

IR (ATR): 3303, 2938, 2287, 1715, 1616, 1469, 1336, 1270, 1183, 1109, 1025, 872, 807, 751, 682 cm^{-1}.

MS (ESI): *m/z* = 424.2 $[M+H]^+$, 446.1 $[M+Na]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{23}H_{23}N_3O_4F^+$: 424.1667; found 424.1661.



7i

Ethyl 2-((3*R*,3'*R*,5'*R*)-5-chloro-1-methyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7i)

According to the general procedure, **7i** was obtained as a colorless solid (108.9 mg, 62% yield).

Melting Point: 123-125 °C.

$[\alpha]_D^{25}$ = –77.4 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK IB; *n*-heptane/EtOH = 7/3; flow rate 0.7 mL/min; T= 30 °C; retention time: 23.45 min (major), 19.13 min (minor), er: 99:1.

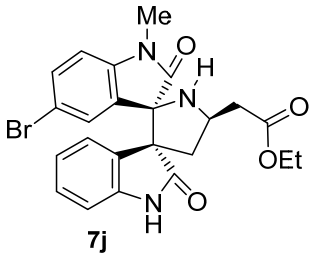
^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.94 (s, 1H), 7.50 (s, 1H), 7.22 (t, *J* = 7.8 Hz, 1H), 7.12 (d, *J* = 8.4 Hz, 1H), 7.06 (t, *J* = 7.8 Hz, 1H), 6.76 (d, *J* = 7.2 Hz, 1H), 6.63 (s, 1H), 6.53 (d, *J* = 8.4 Hz, 1H), 5.00 – 4.95 (m, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.29 – 3.19 (m, 1H), 3.05 (s, 3H), 2.89 (dd, *J* = 16.2, 9.6 Hz, 1H), 2.77 (dd, *J* = 16.2, 4.8 Hz, 1H), 2.04 – 1.99 (m, 1H), 1.27 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 176.6, 176.5, 171.7, 142.6, 140.2, 130.9, 130.8, 129.3, 128.9, 127.5, 127.1, 126.0, 122.2, 109.9, 108.5, 73.7, 60.7, 60.0, 51.5, 41.3, 39.0, 26.2, 14.2.

IR (ATR): 3278, 2954, 2231, 1715, 1464, 1361, 1208, 1091, 859, 750 cm^{-1}.

MS (ESI): *m/z* = 440.1 $[M+H]^+$, 462.1 $[M+Na]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{23}H_{23}N_3O_4Cl^+$: 440.1372; found 440.1361.



7j

Ethyl 2-((3*R*,3'*R*,5'*R*)-5-bromo-1-methyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7j)

According to the general procedure, **7j** was obtained as a colorless solid (102.4 mg, 53% yield).

Melting Point: 115-116 °C.

$[\alpha]_D^{25}$ = –57.5 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK AD; *n*-heptane/EtOH = 7/3; flow rate 1.0 mL/min; T= 30 °C; retention time: 12.63 min (major), 9.97 min (minor), er: 98:2.

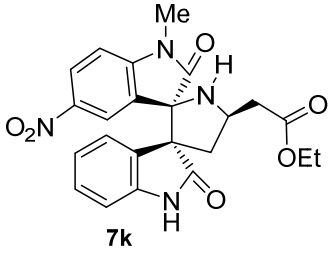
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.58 (s, 1H), 7.56 (s, 1H), 7.25 – 7.18 (m, 2H), 7.10 – 7.05 (m, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.68 (d, *J* = 2.0 Hz, 1H), 6.47 (d, *J* = 8.4 Hz, 1H), 5.00 – 4.93 (m, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 3.26 (dd, *J* = 13.2, 9.6 Hz, 1H), 3.04 (s, 3H), 2.90 (s, 1H), 2.875 (dd, *J* = 16.0, 9.2 Hz, 1H), 2.75 (dd, *J* = 16.0, 5.2 Hz, 1H), 1.98 (dd, *J* = 13.2, 4.8 Hz, 1H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.6, 176.2, 171.8, 143.2, 140.1, 132.1, 131.3, 128.9, 128.8, 127.7, 126.2, 122.1, 114.2, 109.7, 108.8, 73.7, 60.6, 59.8, 51.3, 41.4, 38.9, 26.1, 14.2.

IR (ATR): 3290, 2940, 2639, 2318, 1960, 1706, 1609, 1465, 1342, 1230, 1103, 1035, 824, 765, 676 cm^{-1}.

MS (ESI): *m/z* = 484.1 $[M+H]^+$, 969.2 $[2M+H]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{23}H_{23}N_3O_4Br^+$: 484.0867; found 484.0863.



7k

Ethyl 2-((3*R*,3'*R*,5'*R*)-1-methyl-5-nitro-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7k)

According to the general procedure, **7k** was obtained as a brown solid (73.8 mg, 41% yield).

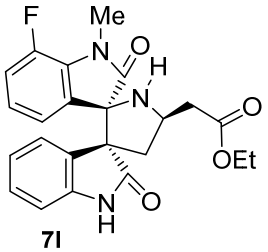
Melting Point: 144-145 °C.

$[\alpha]_D^{25}$ = –48.8 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK IB; *n*-heptane/EtOH = 7/3; flow rate 1.0 mL/min; T= 30 °C; retention time: 15.24 min (major), 12.50 min (minor), er: 95:5.

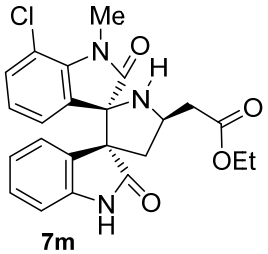
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.08 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.77 – 7.75 (m, 1H), 7.44 (d, *J* = 2.4 Hz, 1H), 7.31 (s, 1H), 7.23 – 7.16 (m, 2H), 6.71 – 6.66 (m, 2H), 5.01 – 4.94 (m, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 3.32 (dd, *J* = 13.2, 10.0 Hz, 1H), 3.16 (s, 3H), 2.96 (s, 1H), 2.89 (dd, *J* = 16.4, 9.6 Hz, 1H), 2.78 (dd, *J* = 16.4, 4.8 Hz, 1H), 1.99 (dd, *J* = 13.2, 4.4 Hz, 1H), 1.28 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 177.5, 176.1, 171.9, 149.8, 142.5, 139.8, 131.2, 129.2, 126.5, 126.3, 126.1, 122.6, 121.5, 109.9, 107.0, 72.9, 60.7, 59.7, 51.1, 41.1, 38.8, 26.4, 14.2.
IR (ATR): 3313, 2927, 2668, 2307, 1718, 1608, 1471, 1329, 1291, 1186, 1027, 827, 750, 681 cm^{-1}.
MS (ESI): *m/z* = 473.1 $[M+Na]^+$.
HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{23}H_{22}N_4O_6Na^+$: 473.1432; found 473.1416.



7l

Ethyl 2-((3*R*,3'*R*,5'*R*)-7-fluoro-1-methyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7l)
According to the general procedure, **7l** was obtained as a colorless solid (120.1 mg, 71% yield).
Melting Point: 107-109 °C.
$[\alpha]_D^{25}$ = –90.0 (c = 1.0, $CHCl_3$).
HPLC: (s,s)-Whelk Ol; *n*-heptane/EtOH = 8/2; flow rate 1.0 mL/min; T= 30 °C; retention time: 9.38 min (major), 12.12 min (minor), er: 95:5.
^{1}H NMR (600 MHz, $CDCl_3$): δ = 8.12 (s, 1H), 7.43 (d, *J* = 7.8 Hz, 1H), 7.18 (t, *J* = 7.8 Hz, 1H), 6.99 (t, *J* = 7.68 Hz, 1H), 6.87 (dd, *J* = 11.4, 8.4 Hz, 1H), 6.75 (d, *J* = 7.8 Hz, 1H), 6.63 – 6.60 (m, 1H), 6.53 (d, *J* = 7.8 Hz, 1H), 5.00 – 4.95 (m, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.25 (d, *J* = 2.4 Hz, 3H), 3.24 – 3.18 (m, 1H), 2.99 (s, 1H), 2.87 (dd, *J* = 16.2, 9.6 Hz, 1H), 2.76 (dd, *J* = 16.2, 4.8 Hz, 1H), 2.02 (dd, *J* = 13.2, 5.4 Hz, 1H), 1.27 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR (150 MHz, $CDCl_3$): δ = 176.8, 176.6, 171.8, 147.2 (d, *J* = 242.1 Hz), 140.4, 130.9, 130.7 (d, *J* = 8.1 Hz), 129.1 (d, *J* = 2.9 Hz), 128.6, 126.0, 122.0 (d, *J* = 6.2 Hz), 121.9, 121.3 (d, *J* = 3.0 Hz), 117.4 (d, *J* = 19.1 Hz), 109.7, 73.8, 60.6, 60.2, 51.5, 41.6, 39.3, 28.5 (d, *J* = 6.2 Hz), 14.3.
IR (ATR): 3321, 2971, 2189, 1730, 1614, 1466, 1366, 1209, 1027, 908, 749 cm^{-1}.
MS (ESI): *m/z* = 424.2 $[M+H]^+$, 446.2 $[M+Na]^+$.
HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{23}H_{22}N_3O_4FNa^+$: 446.1487; found 446.1476.



7m

Ethyl 2-((3*R*,3'*R*,5'*R*)-7-chloro-1-methyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7m)
According to the general procedure, **7m** was obtained as a colorless solid (119.4 mg, 68% yield).
Melting Point: 105-106 °C.
$[\alpha]_D^{25}$ = –111.0 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK IB; *n*-heptane/EtOH = 7/3; flow rate 0.7 mL/min; T= 30 °C; retention time: 23.45 min (major), 19.13 min (minor), er: 97:3.

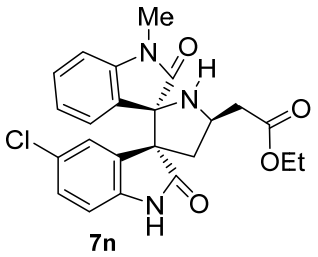
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.19 (s, 1H), 7.44 (d, *J* = 7.6 Hz, 1H), 7.16 (td, *J* = 8.0, 1.2 Hz, 1H), 7.06 – 6.96 (m, 2H), 6.78 – 6.73 (m, 1H), 6.63 – 6.61 (m, 1H), 6.59 – 6.52 (m, 1H), 5.00 – 4.92 (m, 1H), 4.14 (q, *J* = 7.2 Hz, 2H), 3.38 (s, 3H), 3.20 (dd, *J* = 13.2, 9.6 Hz, 1H), 2.92 (s, 1H), 2.85 (dd, *J* = 16.0, 9.6 Hz, 1H), 2.74 (dd, *J* = 16.0, 5.2 Hz, 1H), 1.97 (dd, *J* = 13.2, 5.2 Hz, 1H), 1.25 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 177.4, 176.7, 171.8, 140.4, 139.9, 131.7, 131.0, 129.0, 128.7, 126.1, 124.0, 122.2, 121.8, 114.8, 109.8, 73.2, 60.6, 60.2, 51.4, 41.5, 39.2, 29.4, 14.2.

IR (ATR): 3289, 2926, 2660, 2330, 2089, 1729, 1461, 1366, 1215, 1032, 743 cm^{-1}.

MS (ESI): *m/z* = 440.1 $[M+H]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{23}H_{23}N_3O_4Cl^+$: 440.1372; found 440.1360.



7n

Ethyl 2-((3*R*,3'*R*,5'*R*)-5''-chloro-1-methyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (7n)

According to the general procedure, **7n** was obtained as a colorless solid (135.2 mg, 77% yield).

Melting Point: 112-113 °C.

$[\alpha]_D^{25}$ = –101.9 (c = 1.0, $CHCl_3$).

HPLC: CHIRALPAK IB; *n*-heptane/EtOH = 7/3; flow rate 1.0 mL/min; T= 30 °C; retention time: 9.71 min (major), 11.46 min (minor), er: 86:14.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.91 (s, 1H), 7.38 (d, *J* = 2.0 Hz, 1H), 7.20 – 7.11 (m, 2H), 6.80 – 6.73 (m, 2H), 6.65 (d, *J* = 8.4 Hz, 1H), 6.61 (d, *J* = 8.0 Hz, 1H), 5.00 – 4.93 (m, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 3.18 (dd, *J* = 13.2, 9.6 Hz, 1H), 3.02 (s, 3H), 2.99 (s, 1H), 2.84 (dd, *J* = 16.0, 8.8 Hz, 1H), 2.75 (dd, *J* = 16.0, 5.6 Hz, 1H), 2.07 – 1.96 (m, 1H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 176.5, 176.4, 171.7, 144.0, 138.9, 132.8, 129.7, 128.4, 127.2, 126.3, 126.0, 125.3, 121.9, 110.5, 107.8, 73.7, 60.6, 60.3, 51.5, 41.6, 39.4, 26.0, 14.2.

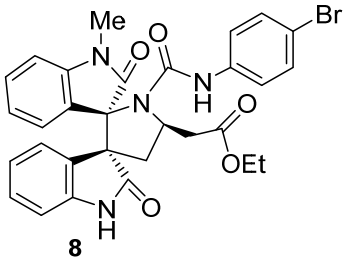
IR (ATR): 3161, 2941, 2657, 2159, 1973, 1719, 1613, 1464, 1368, 1203, 1101, 1025, 839, 739 cm^{-1}.

MS (ESI): *m/z* = 440.1 $[M+H]^+$, 462.1 $[M+Na]^+$.

HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{23}H_{22}N_3O_4ClNa^+$: 462.1191; found 462.1180.

3. Procedure for the synthesis of 8:

To a vial containing a solution of **7a** (156.4 mg, 0.40 mmol, 1.0 equiv) in dry DCM (4.0 mL) was added 4-bromophenyl isocyanate (119.0 mg, 0.60 mmol, 1.5 equiv) at 0°C. The mixture was stirred at room temperature for 12 h. Then the solvent was removed on a rotary evaporator and the residue was purified by flash chromatography (pentane/EtOAc = 1/1 to 1/2) to afford the desired product **8** as a colorless solid (195.1 mg, 81% yield).



8

Ethyl 2-((3*R*,3'*R*,5'*R*)-1'-((4-bromophenyl)carbamoyl)-1-methyl-2,2''-dioxodispiro[indoline-3,2'-pyrrolidine-3',3''-indolin]-5'-yl)acetate (8)

Melting Point: 175-177 ^{o}C.

$[\alpha]_D^{25}$ = +21.7 (c = 1.0, $CHCl_3$).

^{1}H NMR (600 MHz, $CDCl_3$): δ = 8.68 (d, *J* = 13.8 Hz, 1H), 7.58 (s, 1H), 7.39 (d, *J* = 7.2 Hz, 1H), 7.32 (t, *J* = 7.8 Hz, 1H), 7.24 – 7.19 (m, 4H), 7.13 (t, *J* = 7.2 Hz, 1H), 6.98 (t, *J* = 7.8 Hz, 1H), 6.79 (d, *J* = 7.8 Hz, 1H), 6.65 (d, *J* = 8.4 Hz, 1H), 6.53 (t, *J* = 7.8 Hz, 1H), 6.19 (d, *J* = 7.8 Hz, 1H), 5.49 – 5.42 (m, 1H), 4.20 – 4.11 (m, 2H), 3.41 (dd, *J* = 16.2, 4.8 Hz, 1H), 2.96 – 2.91 (m, 4H), 2.83 (dd, *J* = 13.2, 7.2 Hz, 1H), 2.59 (dd, *J* = 13.2, 8.4 Hz, 1H), 2.11 (s, 1H), 1.22 (td, *J* = 7.2, 1.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 176.9, 172.3, 171.8, 171.7, 152.0, 143.5, 142.0, 137.9, 131.5 (2C), 130.0, 129.2, 128.5, 124.4, 123.8, 122.2, 121.2, 120.9 (2C), 115.2, 110.1, 108.7, 73.5, 61.6, 58.6, 53.6, 41.3, 40.6, 26.6, 14.1.

IR (ATR): 3272, 2927, 2651, 2077, 1990, 1723, 1603, 1483, 1353, 1235, 1162, 1013, 936, 823, 752, 683 cm^{-1}.

MS (ESI): *m/z* = 603.1 $[M+H]^+$, 1229.2 $[2M+Na]^+$.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{30}H_{28}N_4O_5Br^+$: 603.1238; found 603.1223.

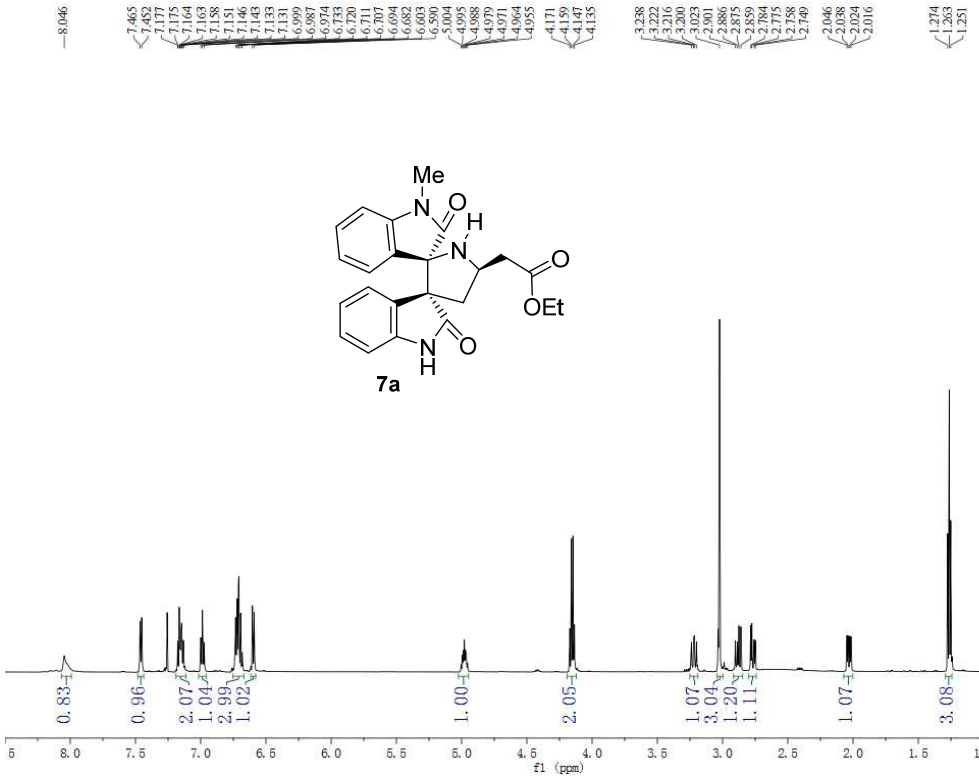
Reference:

[1] W. J. Yan, D. Wang, J. C. Feng, P. Li, D. P. Zhao, R. Wang, *Org. Lett*. **2012**, *14*, 2512-2515.

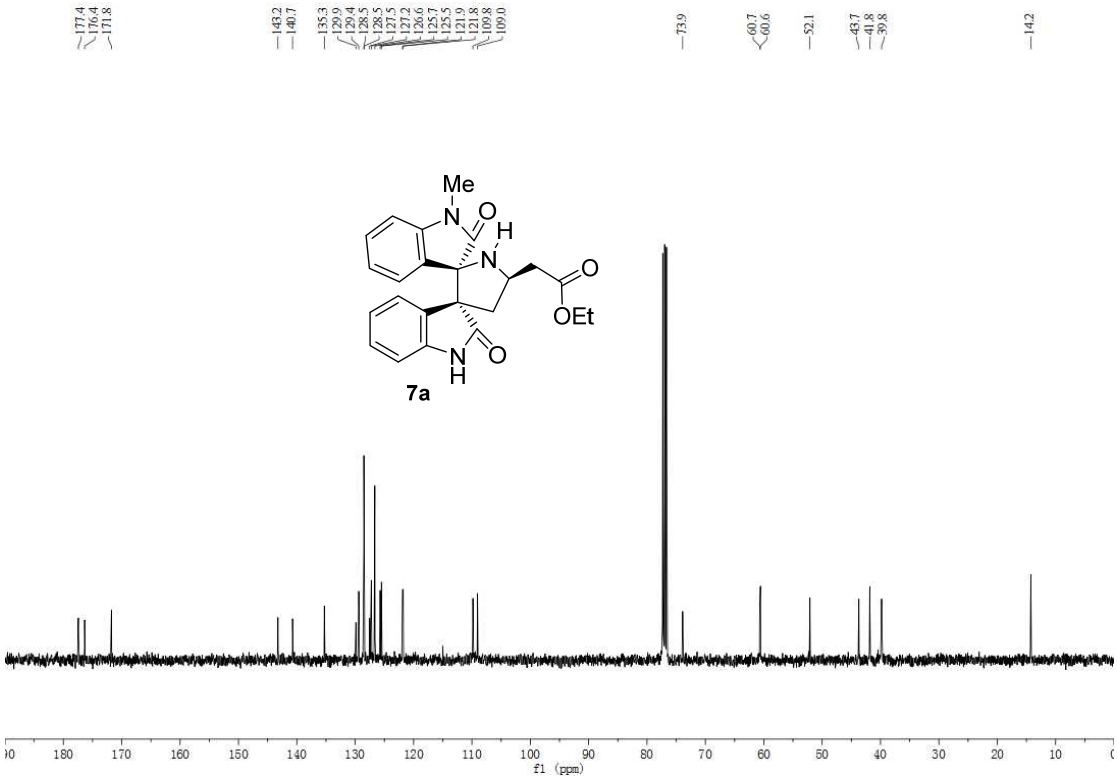
[2] Y.-M. Li, X. Li, F.-Z. Peng, Z.-Q. Li, S.-T. Wu, Z.-W. Sun, H.-B. Zhang and Z.-H. Shao, *Org. Lett.* **2011**, *13*, 6200-6203.

4. NMR Spectra and Chiral HPLC Data:

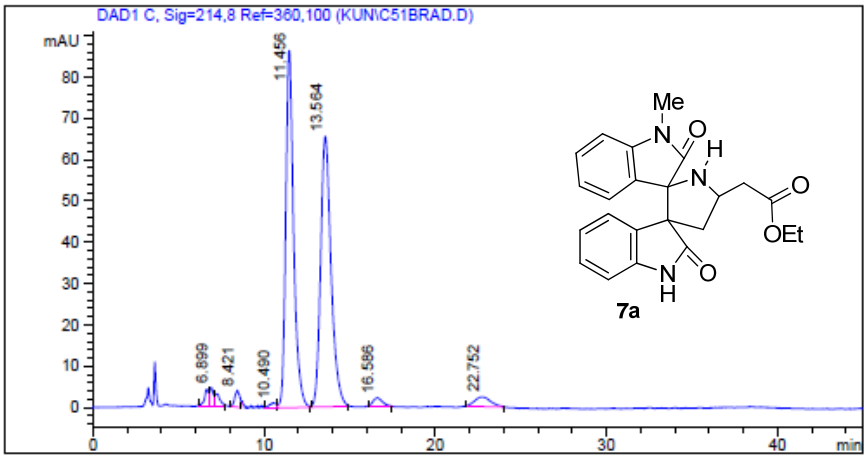
^{1}H NMR of **7a:**



^{13}C NMR of **7a:**



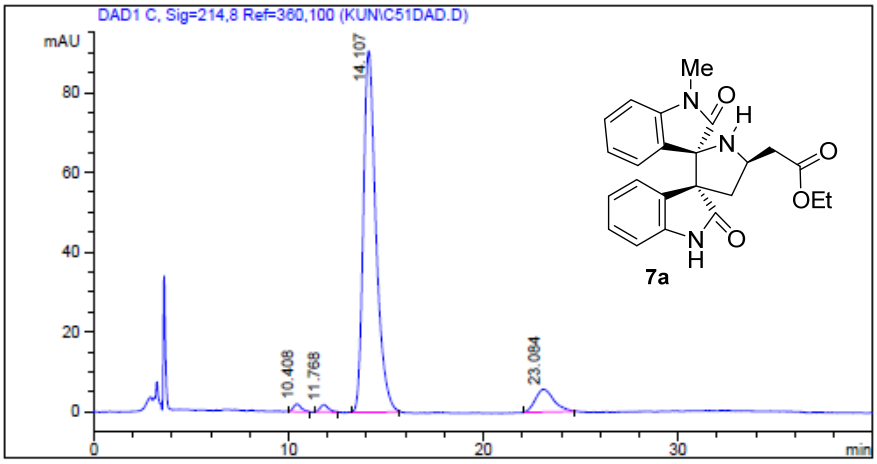
Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	40.8	41.0
Flow in ml/min:	1.00	1.00



7a

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.64	0.23	4.08	63.69	1.04
2	6.90	0.22	4.46	70.93	1.16
3	7.23	0.28	2.99	63.30	1.04
4	8.42	0.25	4.10	81.71	1.34
5	10.49	0.35	1.25	37.05	0.61
6	11.46	0.48	86.50	2765.44	45.32
7	13.56	0.64	65.45	2780.65	45.57
8	16.59	0.48	2.08	83.00	1.36
9	22.75	0.79	2.35	156.41	2.56
Total				6102.18	100.00

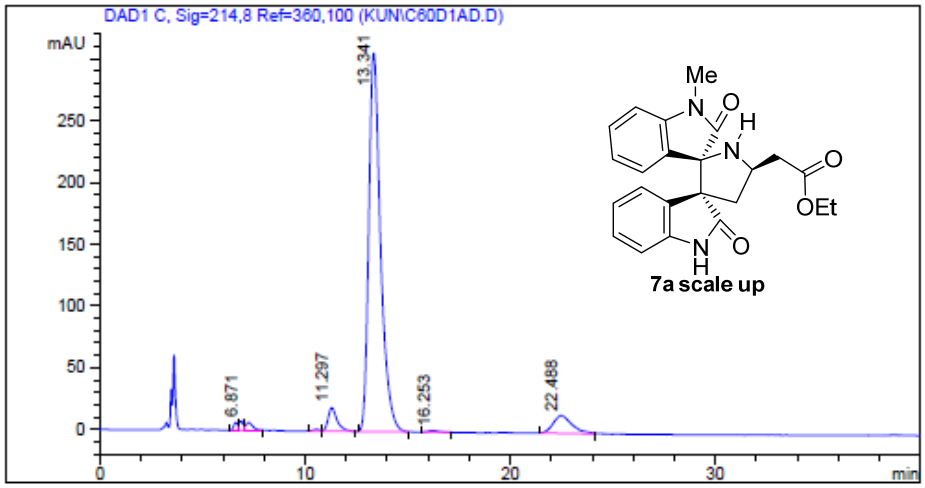
Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	40.0	41.0
Flow in ml/min:	1.00	1.00



7a

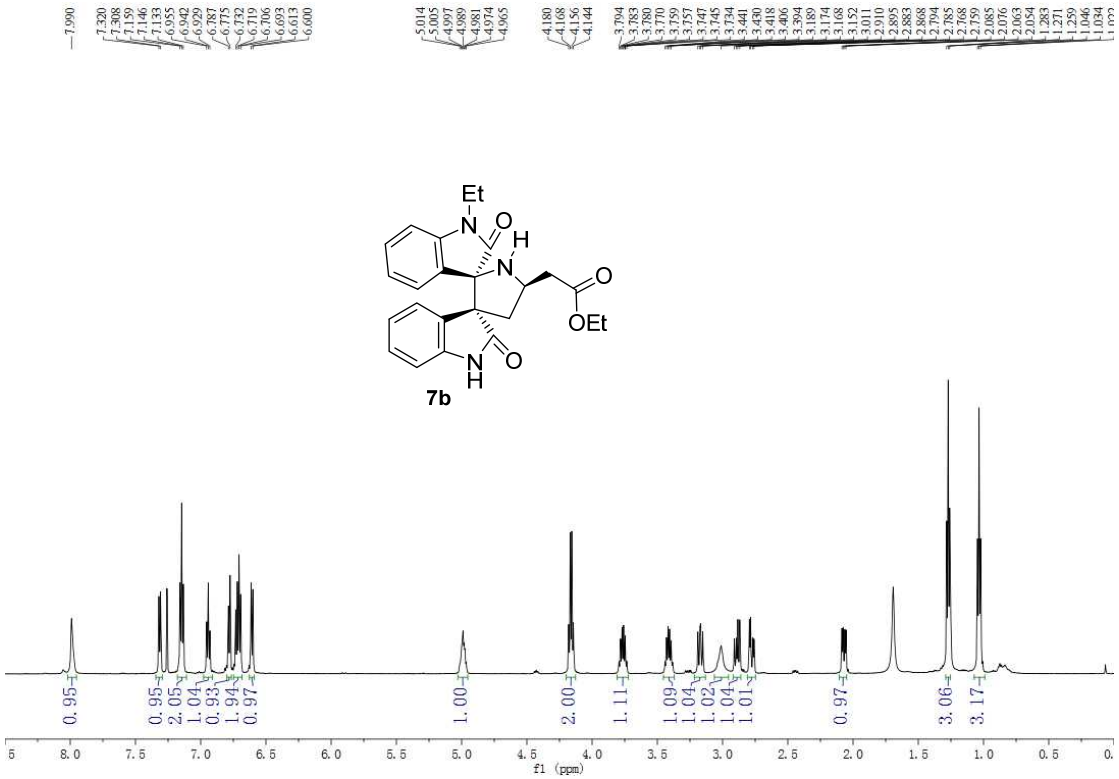
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	10.41	0.34	1.94	53.72	1.17
2	11.77	0.37	1.83	55.68	1.22
3	14.11	0.67	90.65	4087.43	89.39
4	23.08	0.78	5.68	375.93	8.22
Total				4572.76	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	38.9	39.7
Flow in ml/min:	1.00	1.00

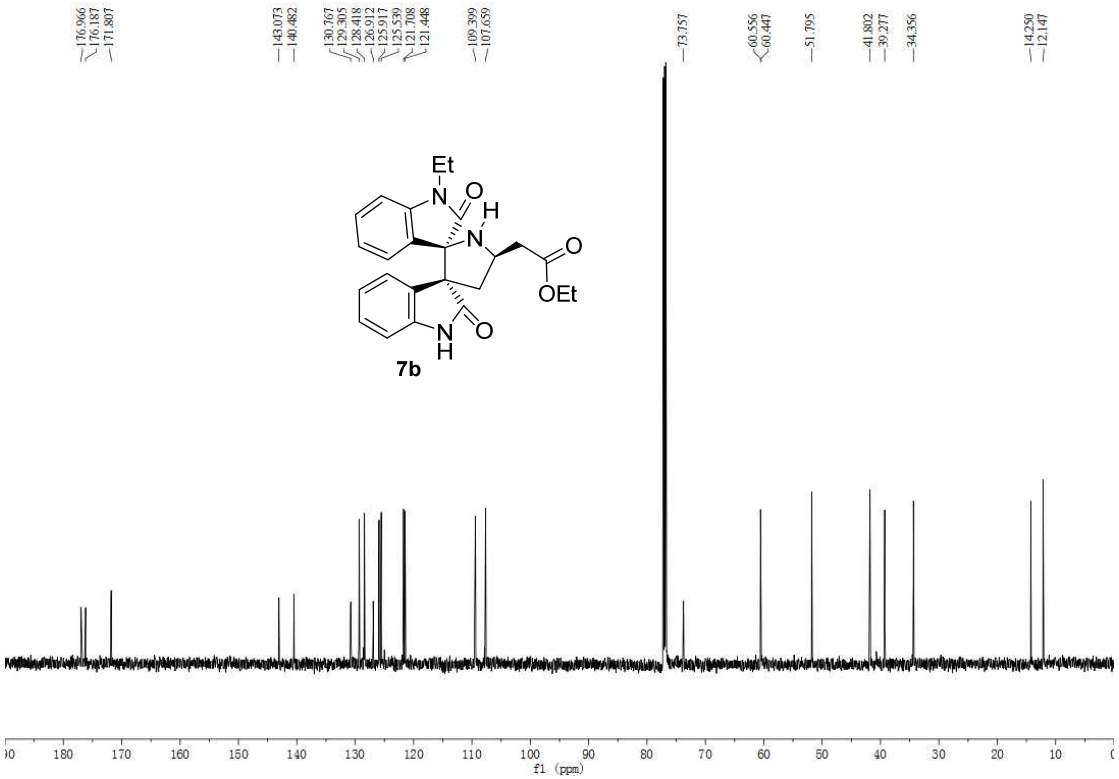


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.60	0.18	6.51	80.49	0.57
2	6.87	0.22	7.52	116.27	0.82
3	7.24	0.33	6.38	150.14	1.06
4	10.57	0.31	1.47	37.71	0.27
5	11.30	0.45	19.09	591.94	4.17
6	13.34	0.60	306.51	12239.26	86.16
7	16.25	0.46	1.34	50.17	0.35
8	22.49	0.80	14.53	939.13	6.61
Total				14205.12	100.00

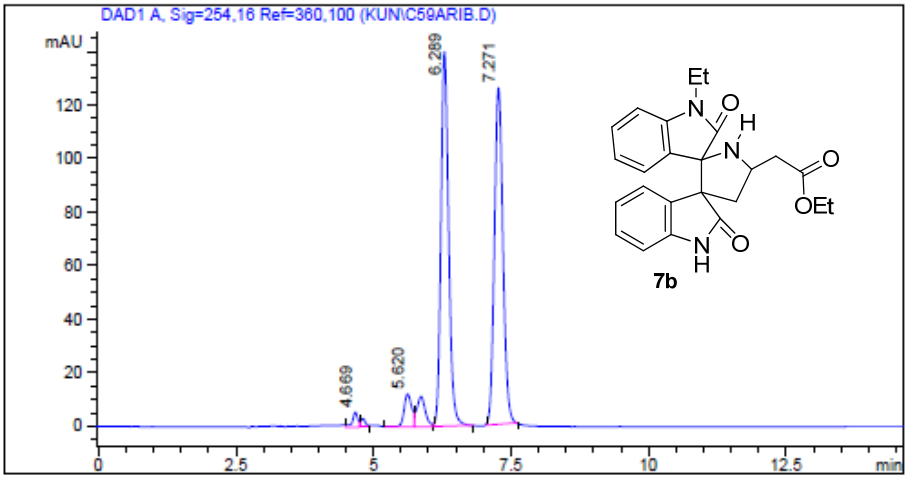
^{1}H NMR of **7b:**



^{13}C NMR of **7b:**

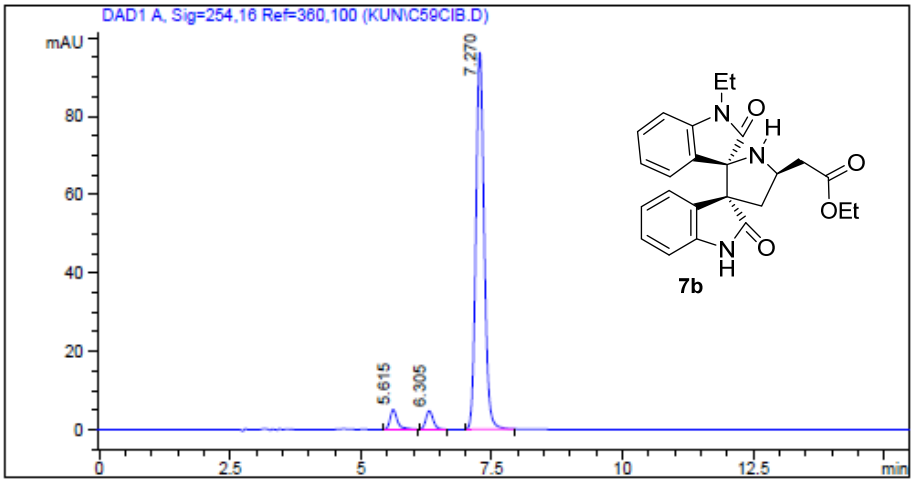


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	58.6	59.1
Flow in ml/min:	1.00	1.00



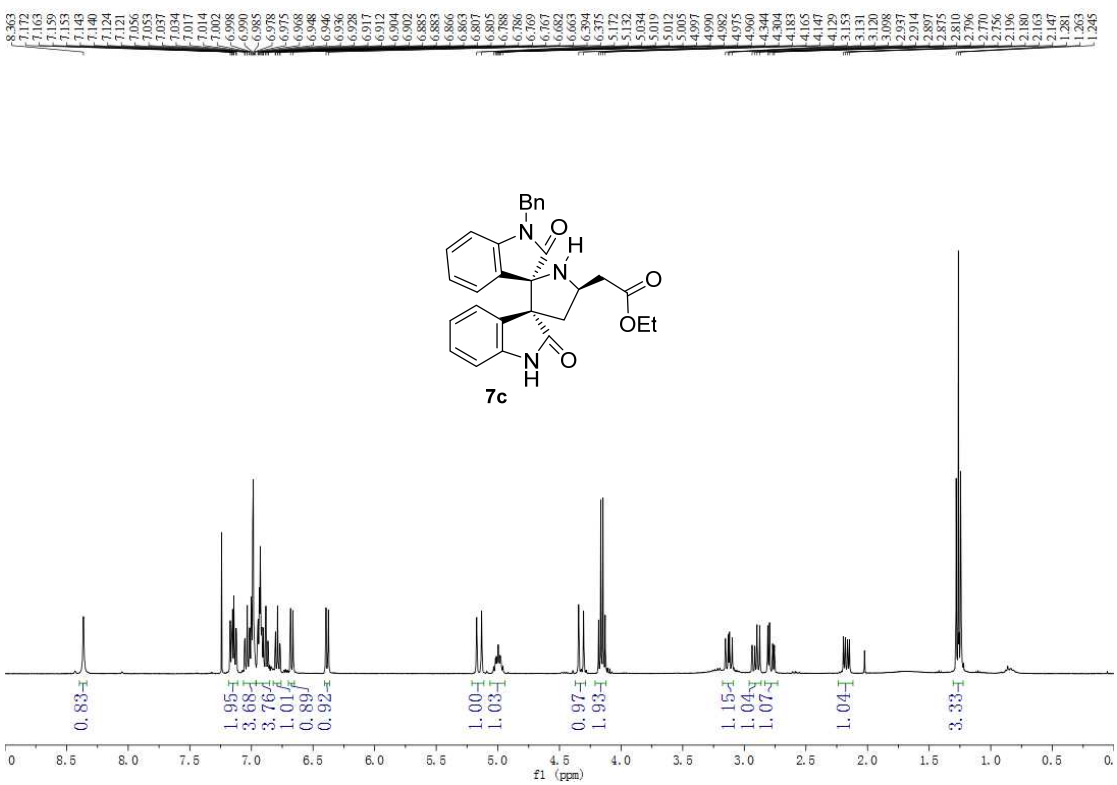
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.67	0.10	5.58	38.36	1.27
2	4.80	0.10	3.20	20.73	0.69
3	5.62	0.15	12.38	122.67	4.06
4	5.86	0.16	11.38	121.85	4.04
5	6.29	0.16	140.14	1357.43	44.96
6	7.27	0.18	125.76	1358.26	44.99
Total				3019.30	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	59.0	58.9
Flow in ml/min:	1.00	1.00

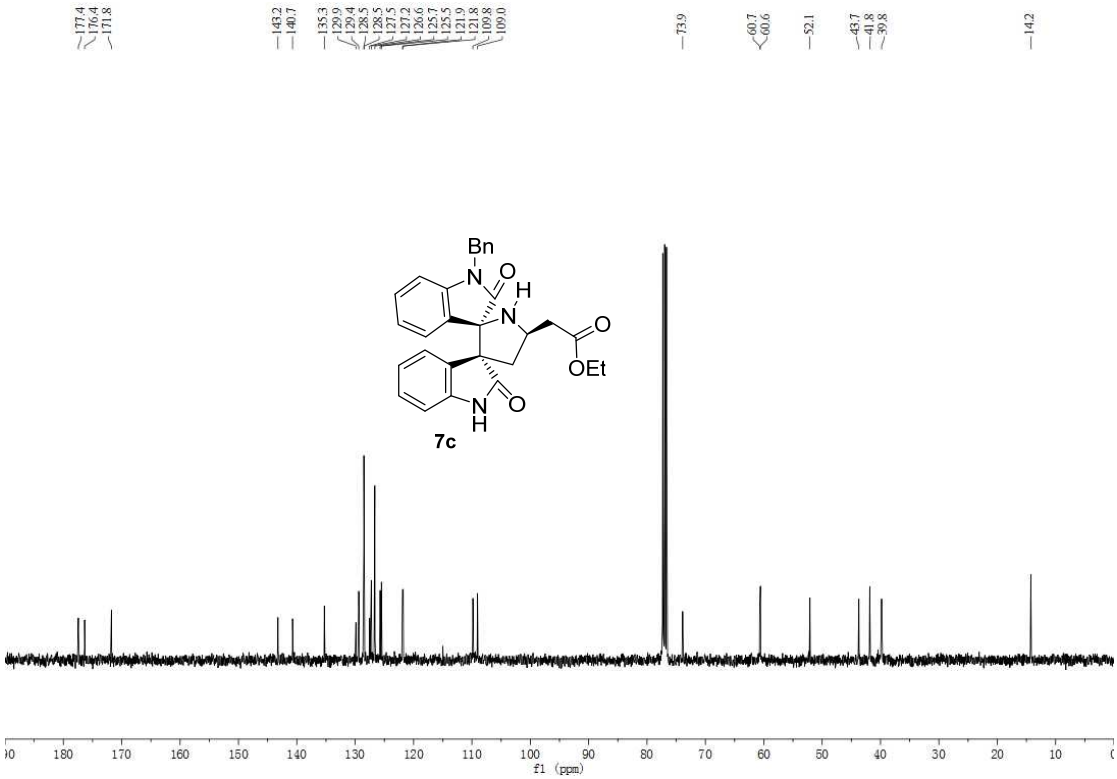


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	5.61	0.15	5.07	50.16	4.32
2	6.30	0.15	4.75	47.48	4.09
3	7.27	0.17	96.30	1064.44	91.60
Total				1162.09	100.00

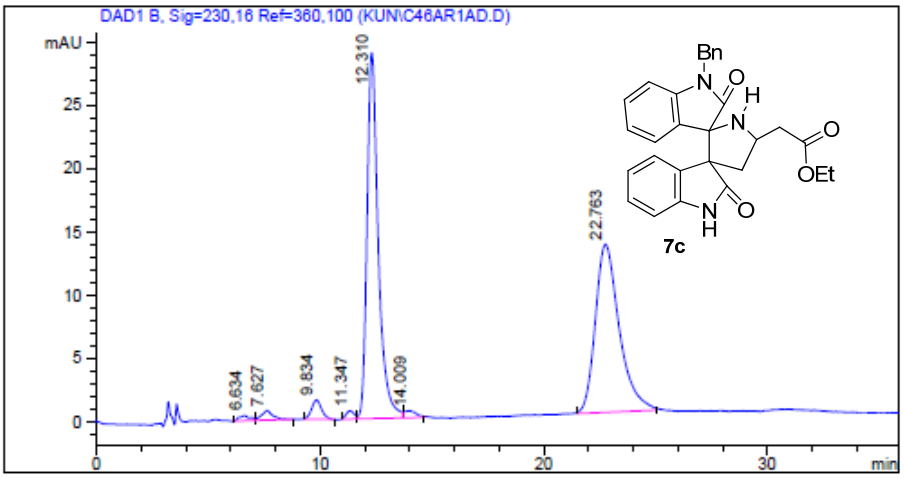
^{1}H NMR of **7c:**



^{13}C NMR of **7c:**

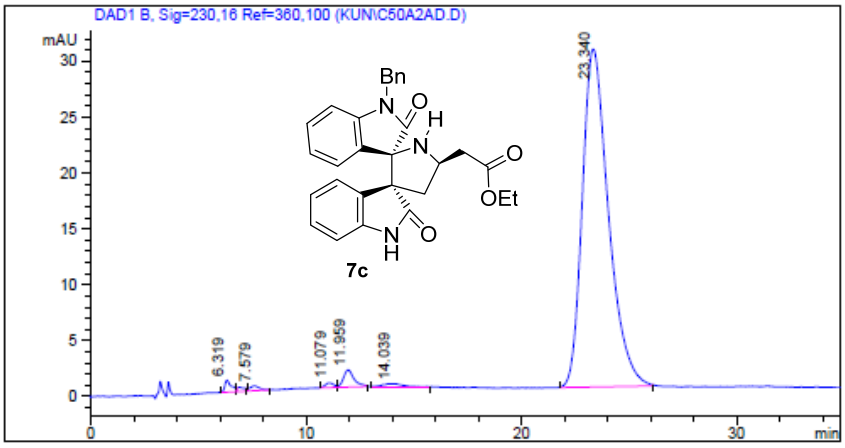


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	41.3	42.3
Flow in ml/min:	1.00	1.00



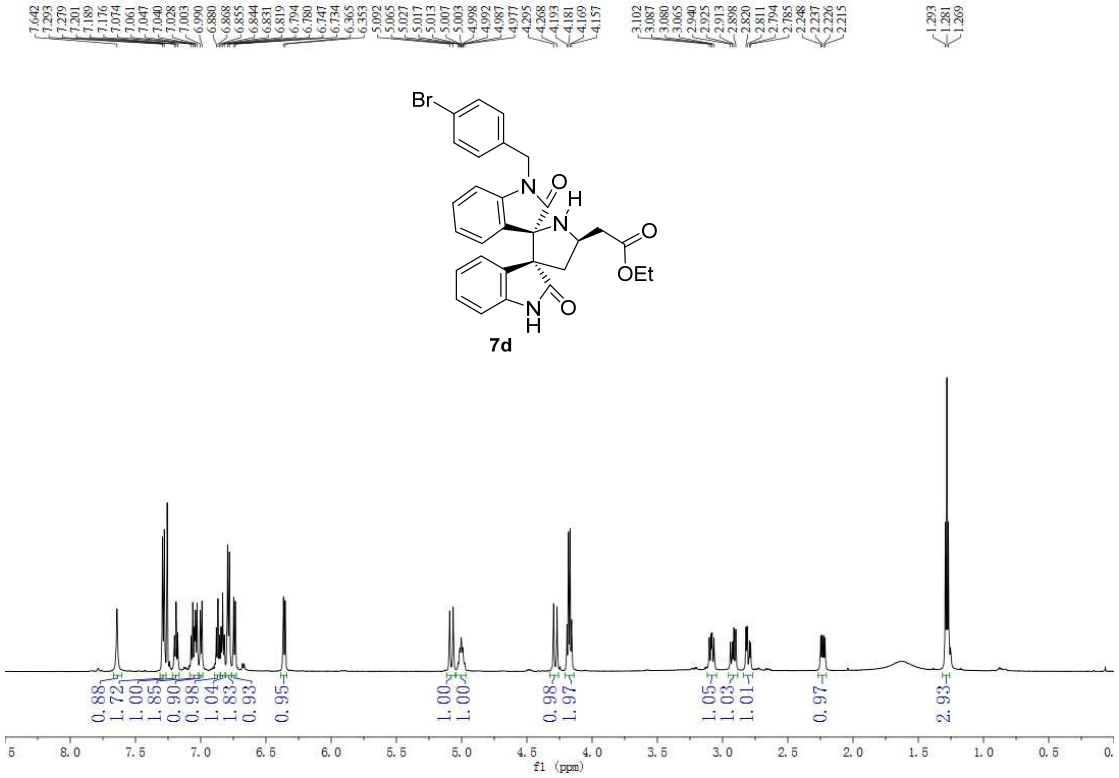
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.63	0.43	0.41	13.90	0.65
2	7.63	0.45	0.77	28.24	1.33
3	9.83	0.46	1.53	47.43	2.23
4	11.35	0.31	0.65	16.22	0.76
5	12.31	0.51	28.93	1007.48	47.45
6	14.01	0.45	0.55	20.53	0.97
7	22.76	1.05	13.30	989.26	46.60
Total				2123.06	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	40.0	40.6
Flow in ml/min:	1.00	1.00

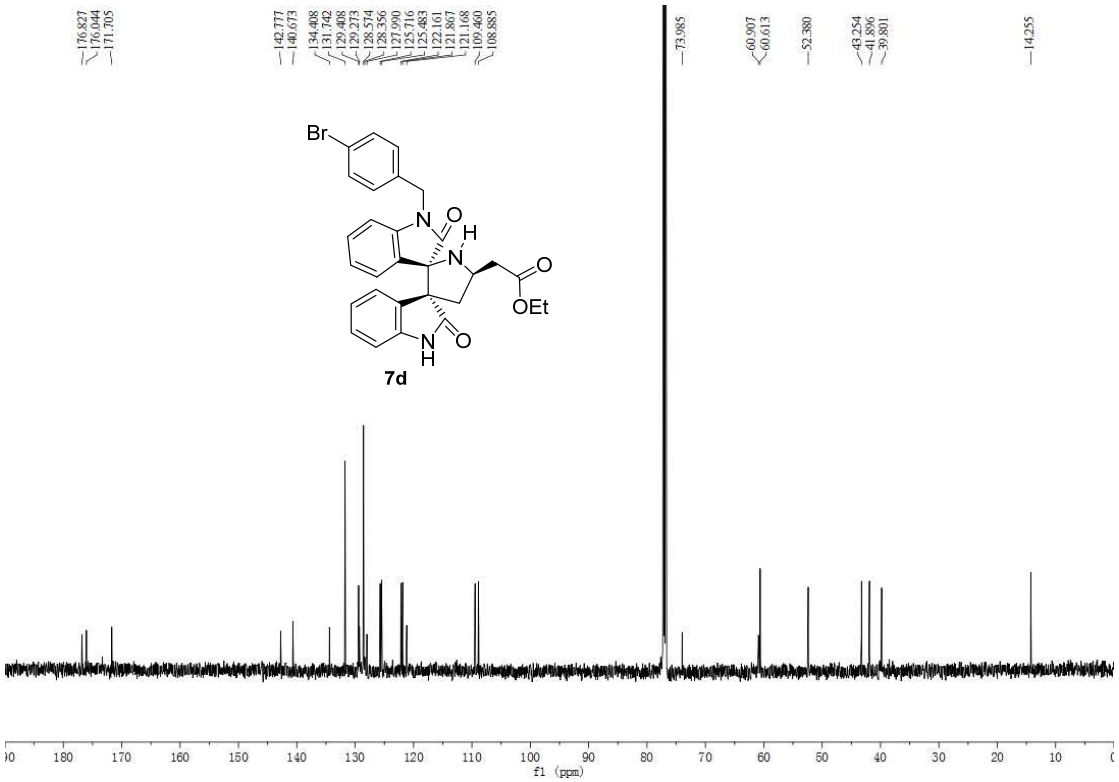


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.32	0.29	1.07	22.79	0.85
2	6.90	0.29	0.38	8.50	0.32
3	7.58	0.37	0.44	13.72	0.51
4	11.08	0.34	0.42	11.51	0.43
5	11.96	0.45	1.53	52.56	1.95
6	14.04	1.24	0.30	22.64	0.84
7	23.34	1.24	30.23	2559.31	95.10
Total				2691.04	100.00

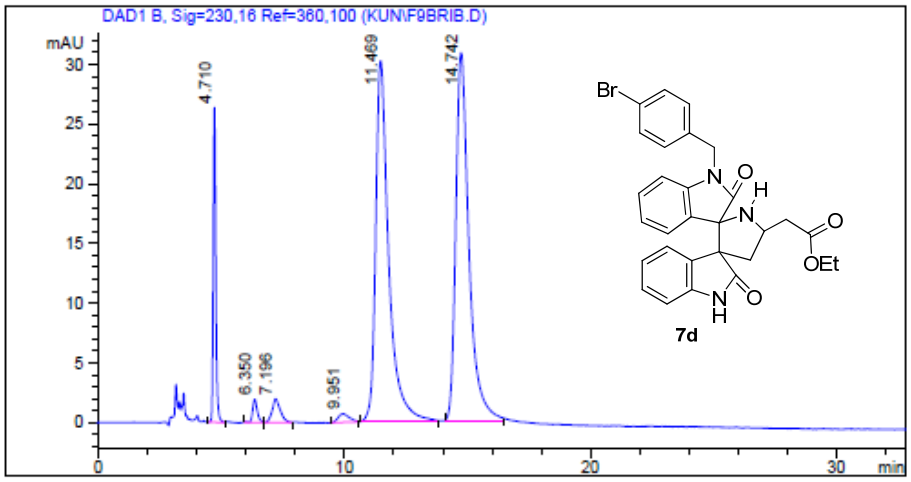
^{1}H NMR of **7d:**



^{13}C NMR of **7d:**

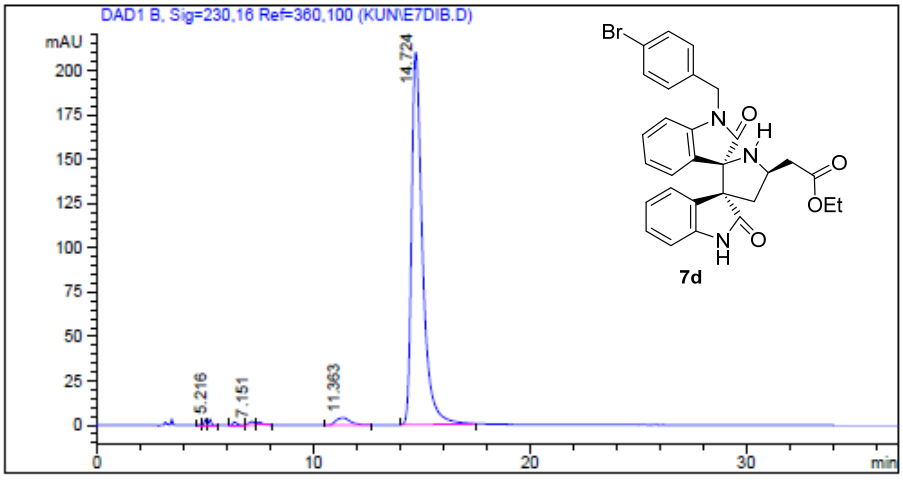


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	60.3	61.2
Flow in ml/min:	1.00	1.00



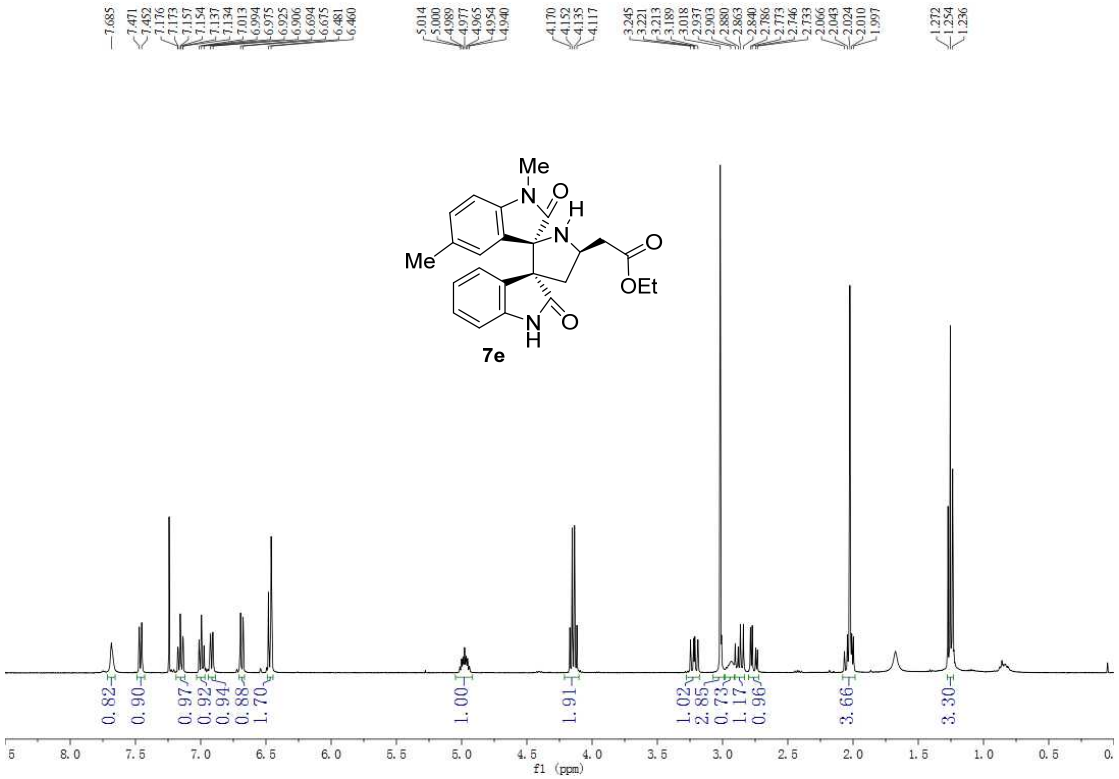
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.71	0.11	26.39	188.94	7.46
2	6.35	0.20	1.95	26.20	1.03
3	7.20	0.32	2.00	45.54	1.80
4	9.95	0.40	0.72	22.87	0.90
5	11.47	0.61	30.19	1105.18	43.65
6	14.74	0.56	30.81	1143.38	45.16
Total				2532.12	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	59.9	60.0
Flow in ml/min:	1.00	1.00

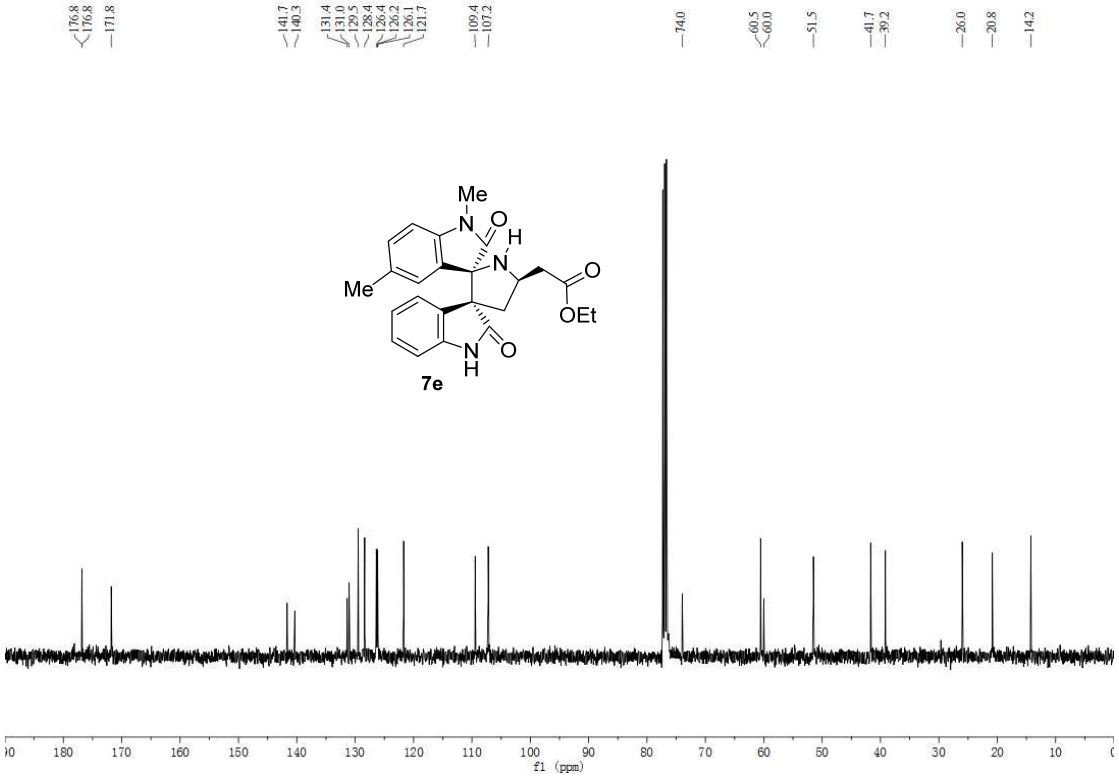


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.76	0.15	0.42	4.08	0.05
2	5.00	0.11	3.32	22.96	0.30
3	5.22	0.12	3.39	25.94	0.34
4	6.35	0.20	1.74	23.31	0.30
5	7.15	0.27	1.96	34.78	0.45
6	7.49	0.27	1.73	32.51	0.42
7	11.36	0.62	4.10	191.95	2.49
8	14.72	0.52	210.11	7371.72	95.65
Total				7707.26	100.00

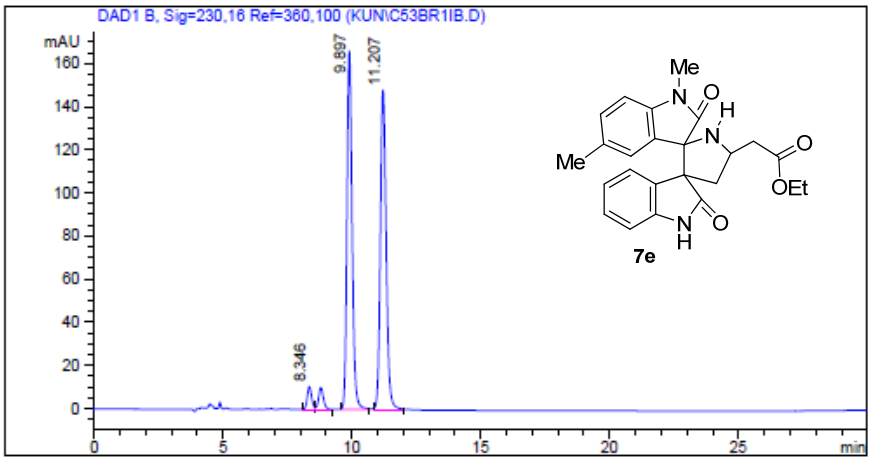
^{1}H NMR of **7e:**



^{13}C NMR of **7e:**

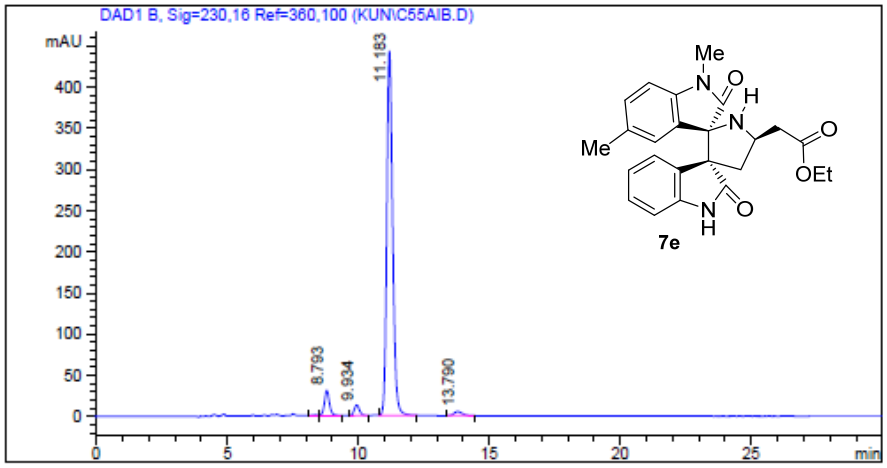


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	41.7	42.1
Flow in ml/min:	0.70	0.70



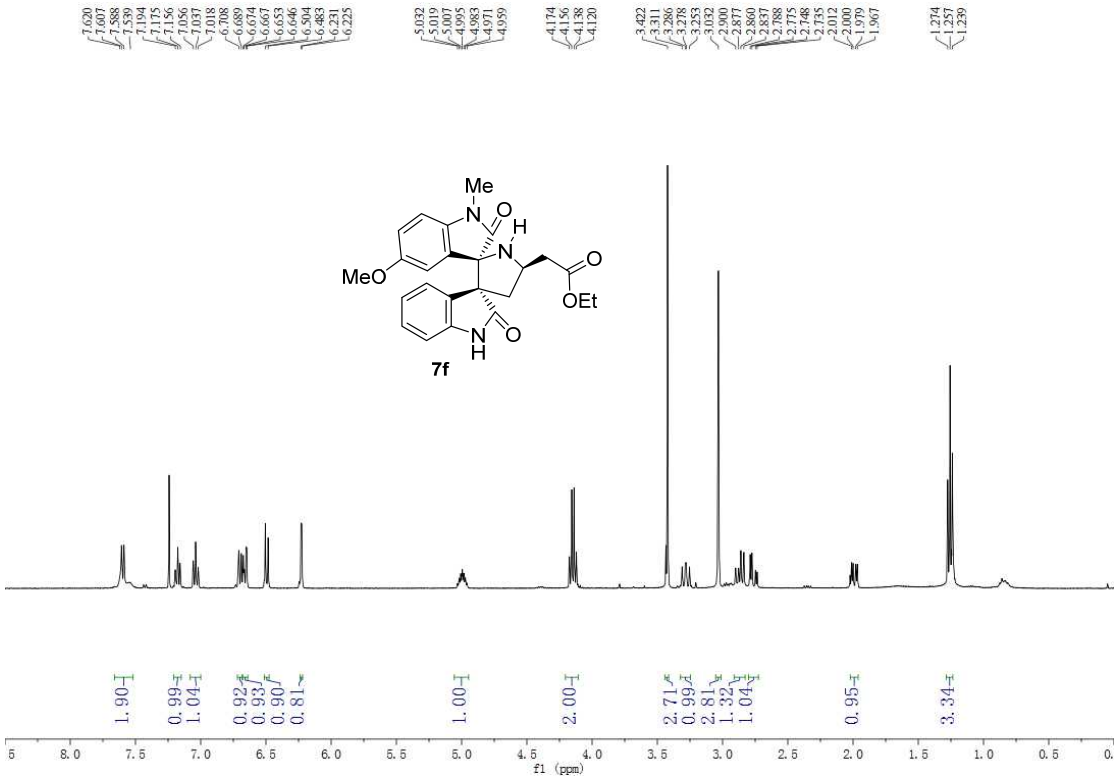
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	8.35	0.19	10.56	128.24	2.61
2	8.80	0.20	10.17	133.33	2.72
3	9.90	0.21	166.19	2319.52	47.27
4	11.21	0.24	148.11	2325.91	47.40
Total				4906.99	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	42.0	42.6
Flow in ml/min:	0.70	0.70

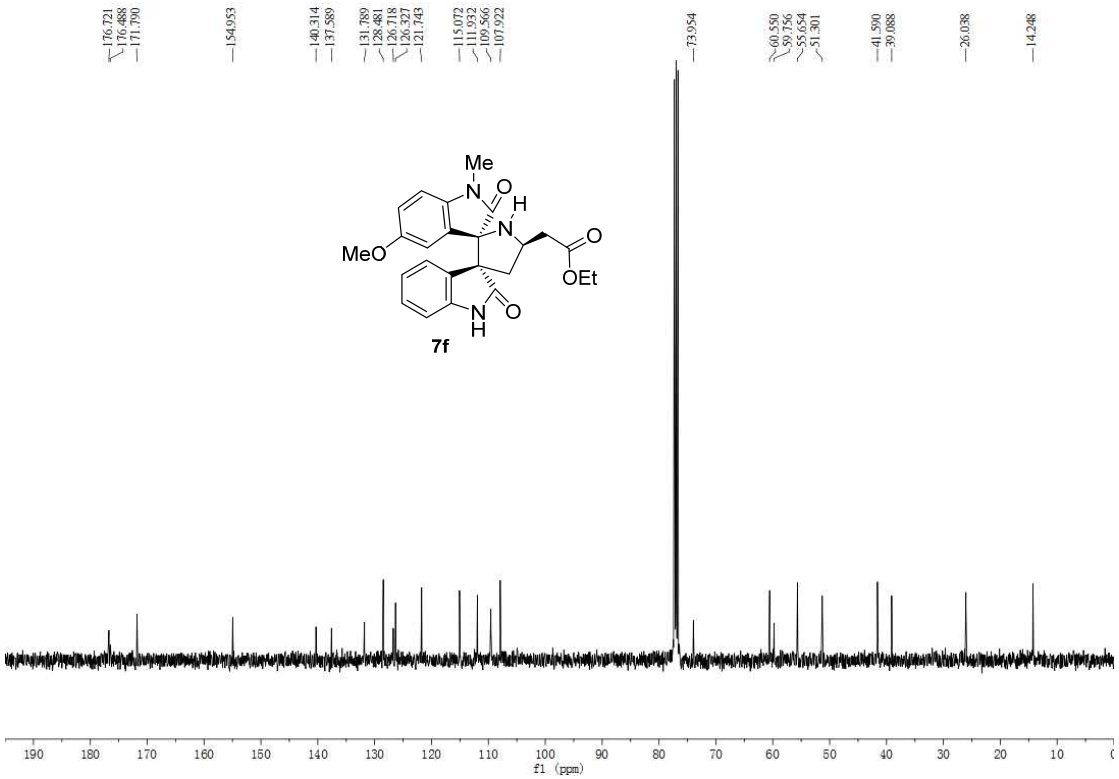


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	8.35	0.24	1.31	21.86	0.29
2	8.79	0.20	30.93	421.35	5.50
3	9.93	0.22	13.00	188.30	2.46
4	11.18	0.24	443.00	6915.34	90.24
5	13.79	0.35	5.06	116.17	1.52
Total				7663.03	100.00

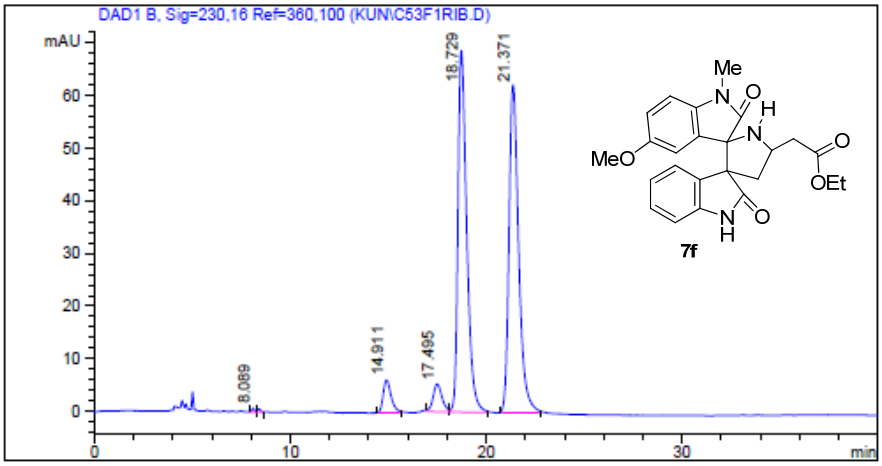
^{1}H NMR of **7f:**



^{13}C NMR of **7f:**

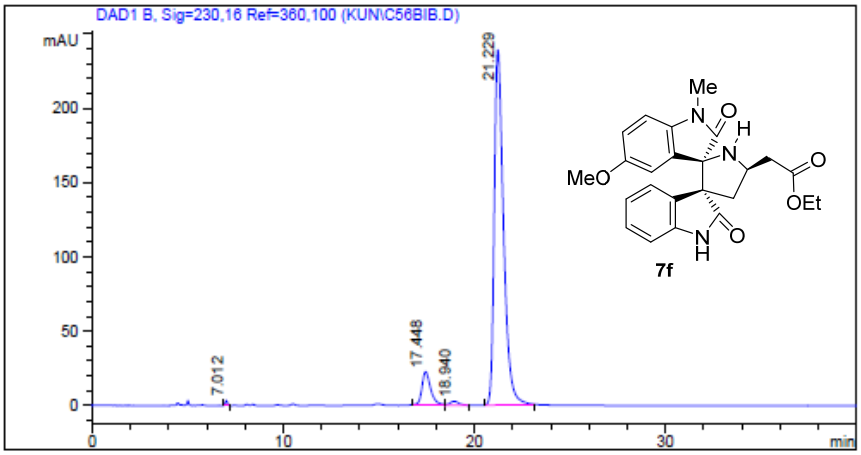


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	37.1	37.6
Flow in ml/min:	0.70	0.70



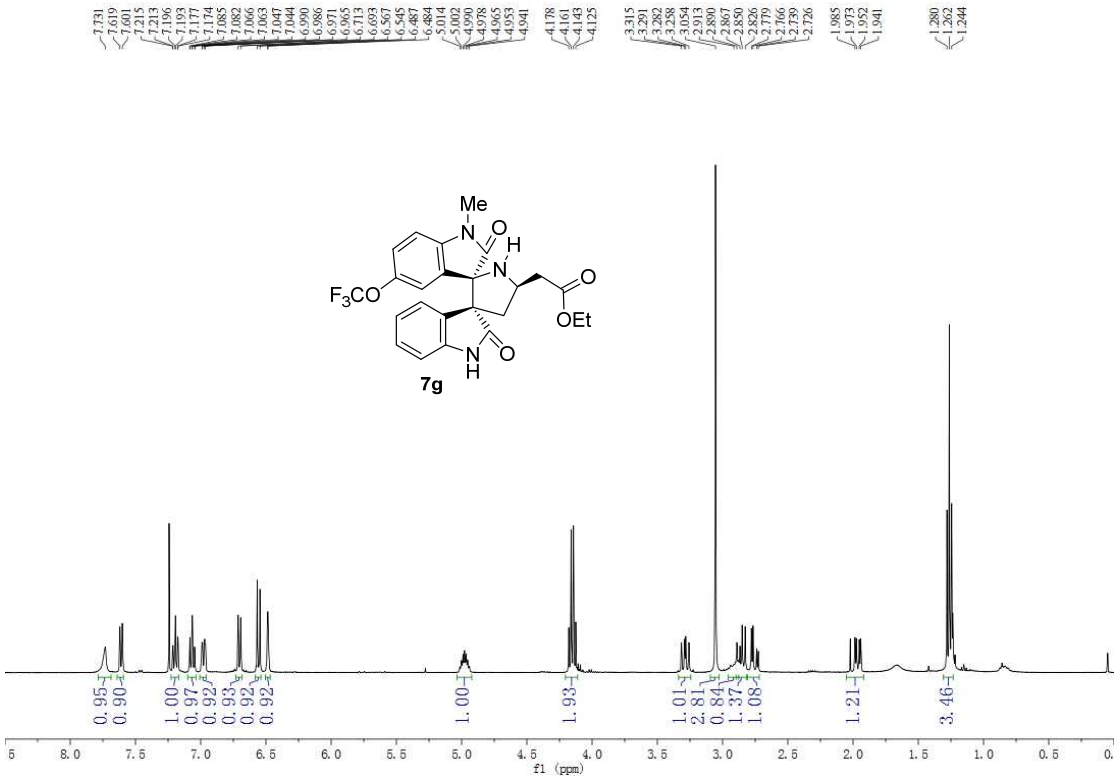
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	8.09	0.14	0.68	6.01	0.13
2	8.39	0.14	0.64	5.98	0.13
3	14.91	0.41	6.23	176.56	3.82
4	17.50	0.48	5.25	167.22	3.62
5	18.73	0.47	68.70	2133.91	46.15
6	21.37	0.52	62.27	2134.41	46.16
Total				4624.09	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	36.9	37.0
Flow in ml/min:	0.70	0.70

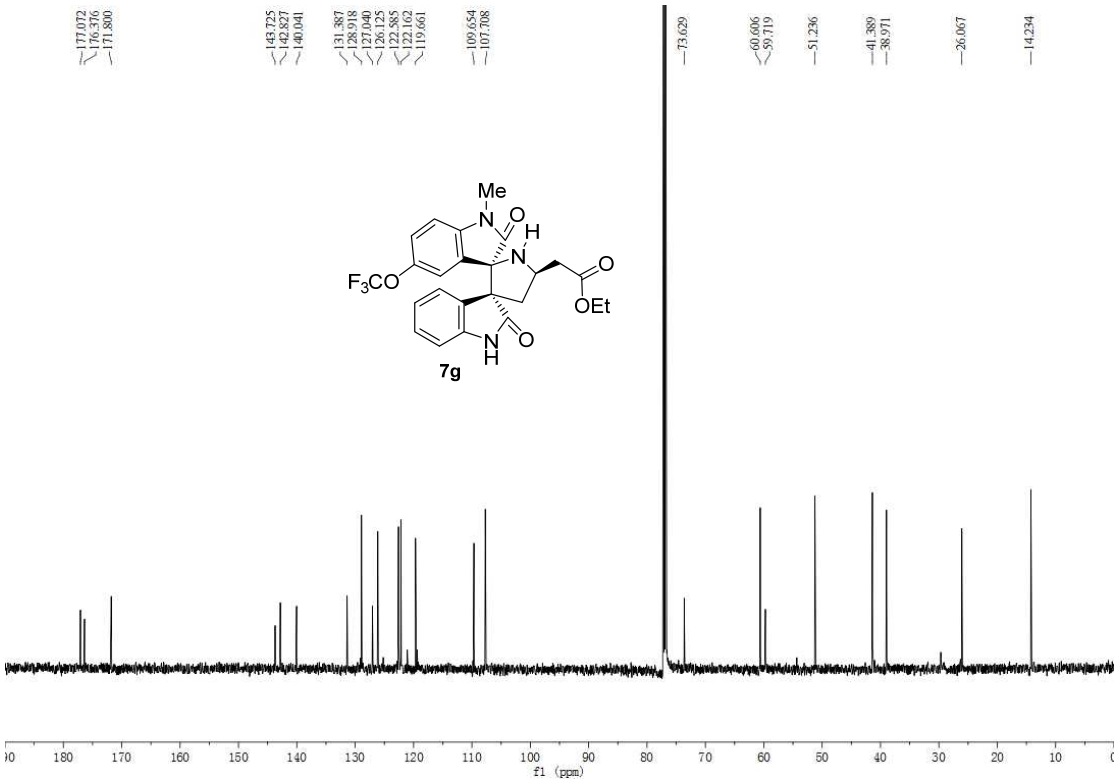


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	7.01	0.12	3.50	26.90	0.30
2	17.45	0.48	22.52	720.97	8.08
3	18.94	0.46	2.75	90.96	1.02
4	21.23	0.51	239.18	8086.46	90.60
Total				8925.29	100.00

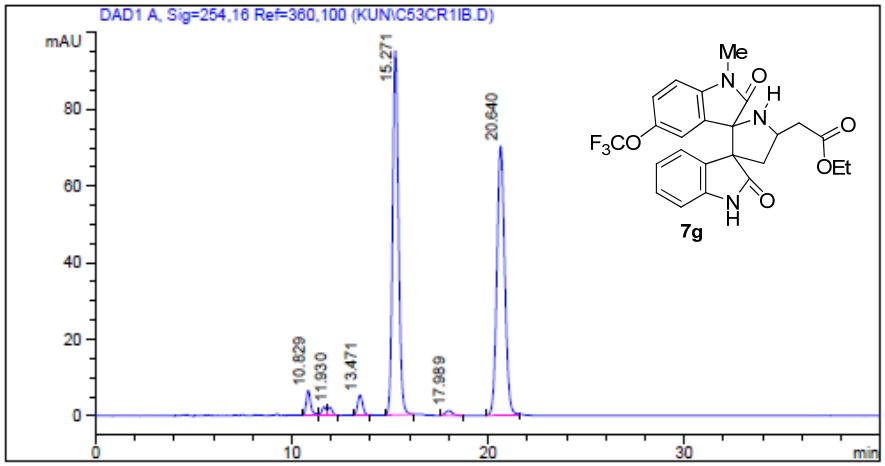
^{1}H NMR of **7g:**



^{13}C NMR of **7g:**

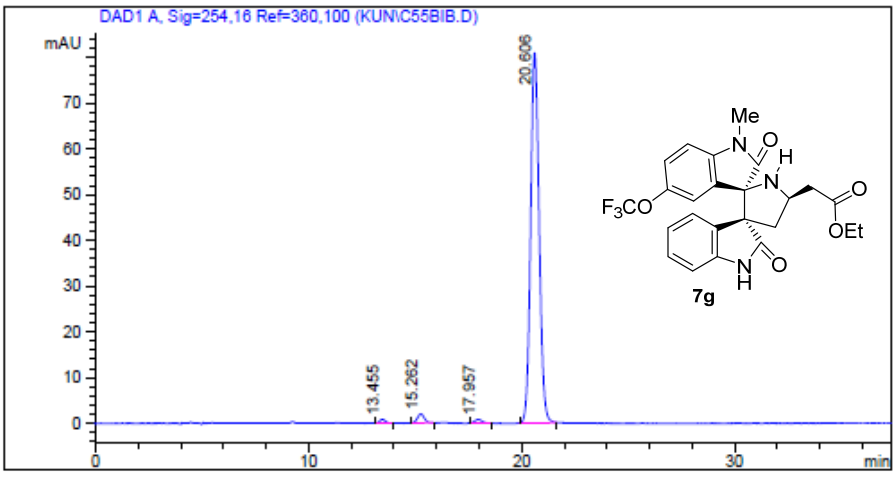


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	37.8	38.2
Flow in ml/min:	0.70	0.70



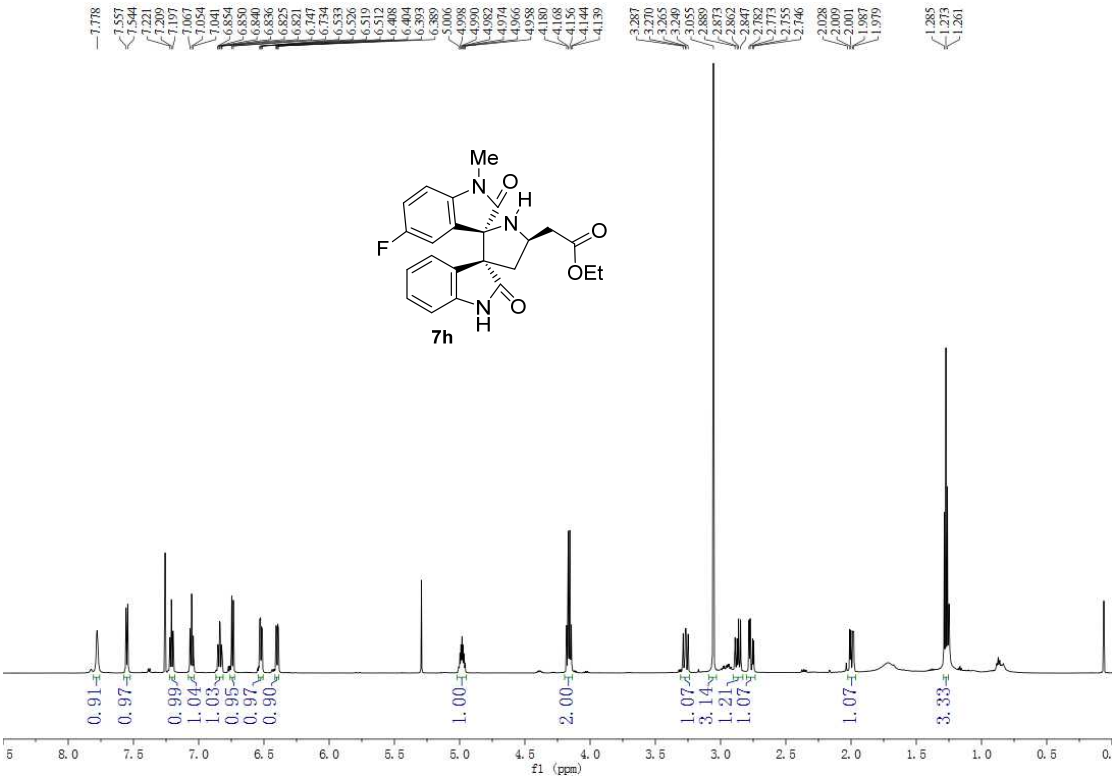
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	10.83	0.25	6.44	104.70	2.47
2	11.64	0.23	2.20	34.14	0.81
3	11.93	0.23	2.12	32.78	0.77
4	13.47	0.28	5.24	92.89	2.19
5	15.27	0.32	95.10	1982.96	46.80
6	17.99	0.38	1.25	31.74	0.75
7	20.64	0.43	70.33	1958.17	46.21
Total				4237.39	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	37.6	37.7
Flow in ml/min:	0.70	0.70

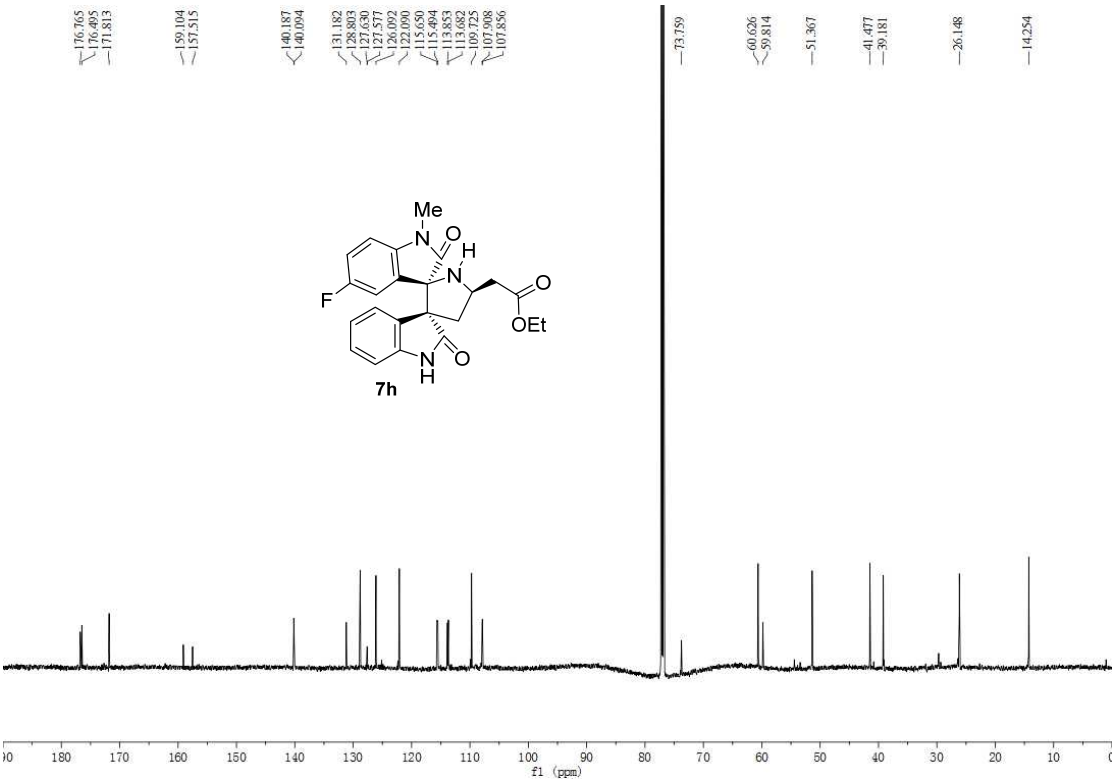


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	13.46	0.28	0.84	14.90	0.64
2	15.26	0.35	1.98	44.15	1.90
3	17.96	0.36	0.83	20.44	0.88
4	20.61	0.43	80.96	2247.69	96.58
Total				2327.19	100.00

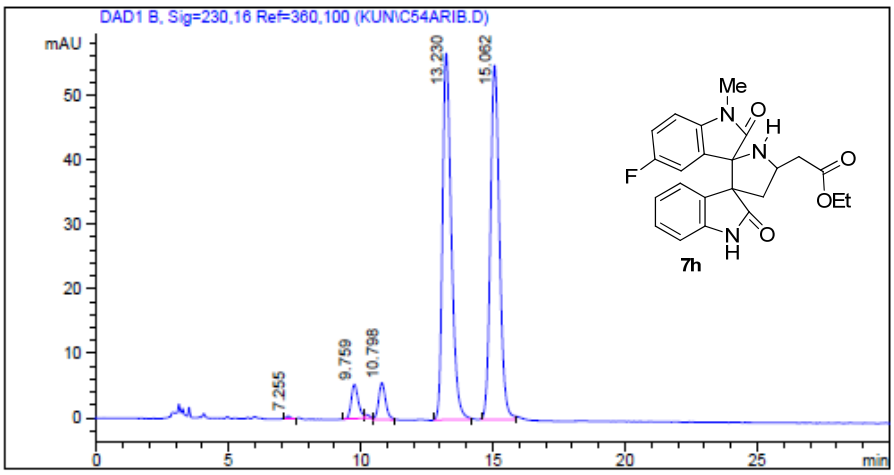
^{1}H NMR of **7h:**



^{13}C NMR of **7h:**

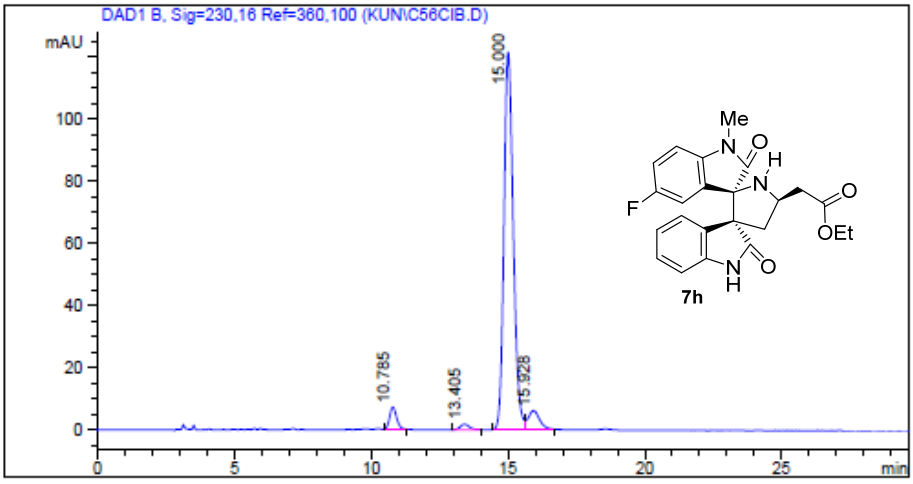


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	53.1	53.2
Flow in ml/min:	1.00	1.00



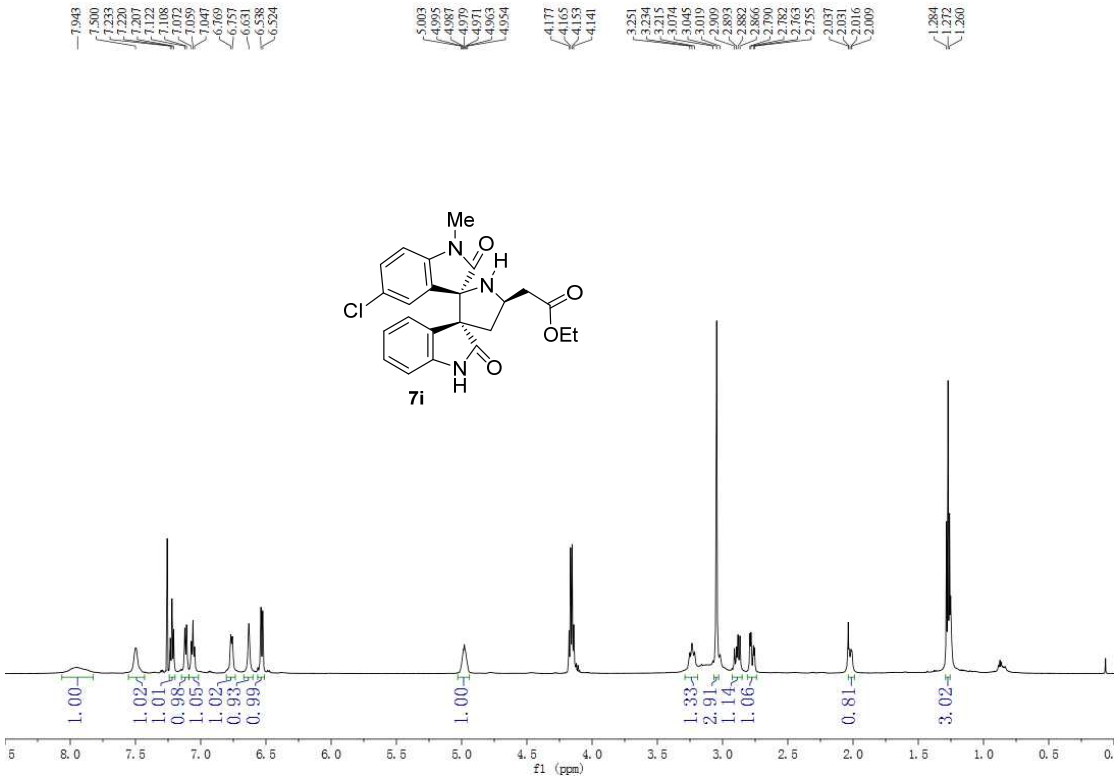
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	7.25	0.19	0.45	5.73	0.20
2	9.76	0.31	5.30	98.33	3.47
3	10.19	0.19	0.59	6.52	0.23
4	10.80	0.28	5.72	105.76	3.73
5	13.23	0.35	56.81	1311.45	46.26
6	15.06	0.37	54.91	1307.09	46.11
Total				2834.89	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	52.9	53.2
Flow in ml/min:	1.00	1.00

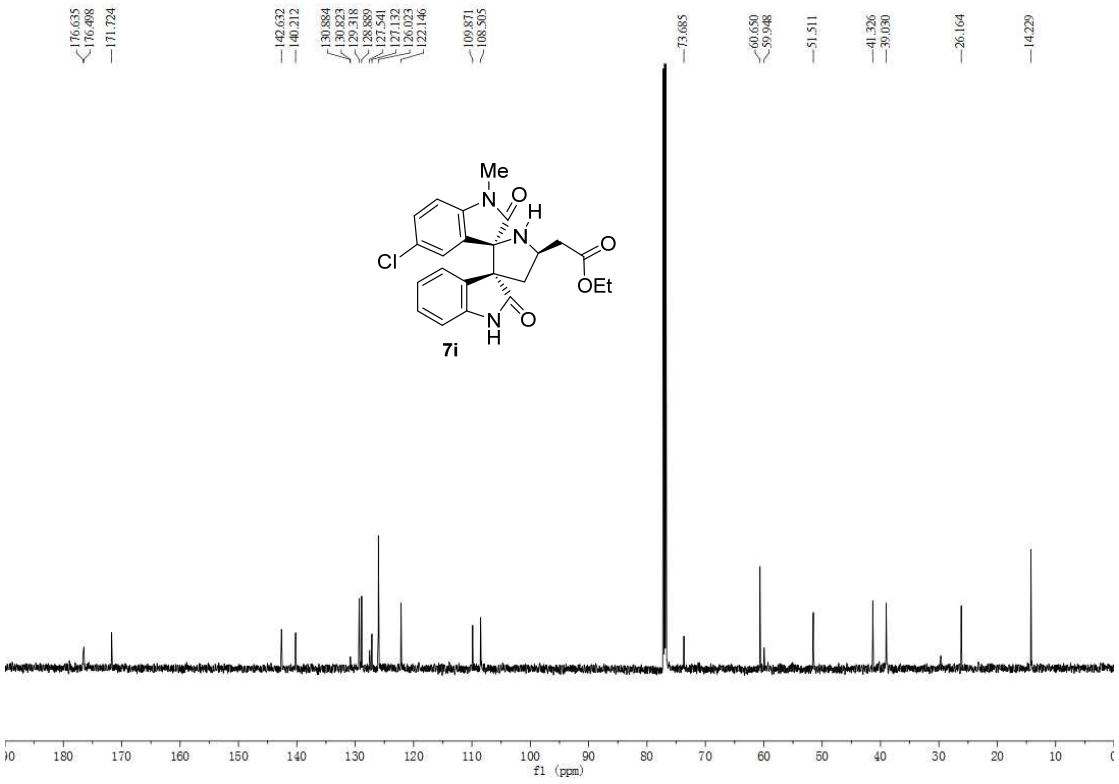


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	10.78	0.28	7.16	128.04	3.95
2	13.41	0.37	1.78	41.97	1.30
3	15.00	0.36	121.74	2889.37	89.17
4	15.93	0.43	6.17	181.05	5.59
Total				3240.42	100.00

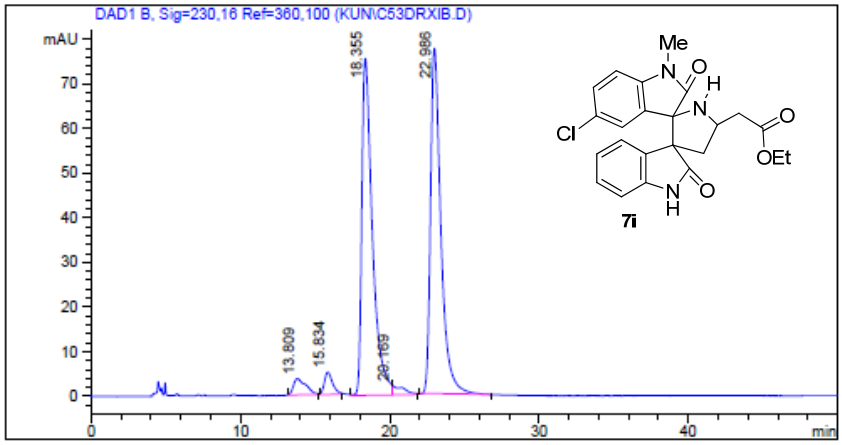
^{1}H NMR of **7i:**



^{13}C NMR of **7i:**

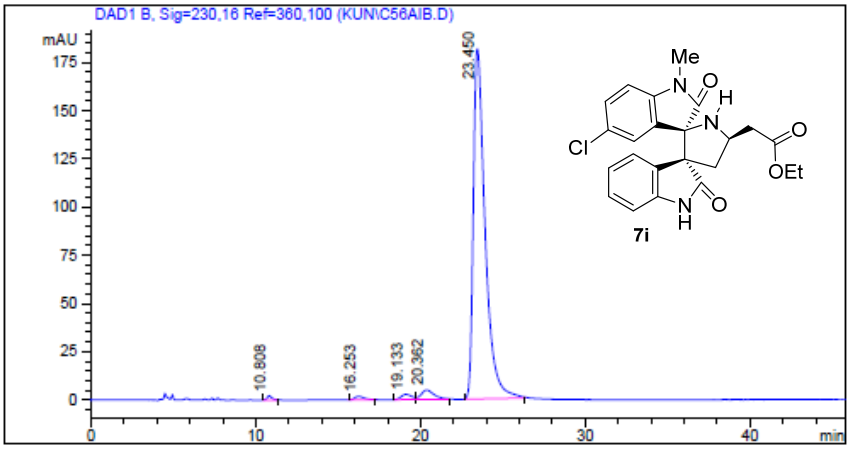


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	41.0	42.0
Flow in ml/min:	0.70	0.70



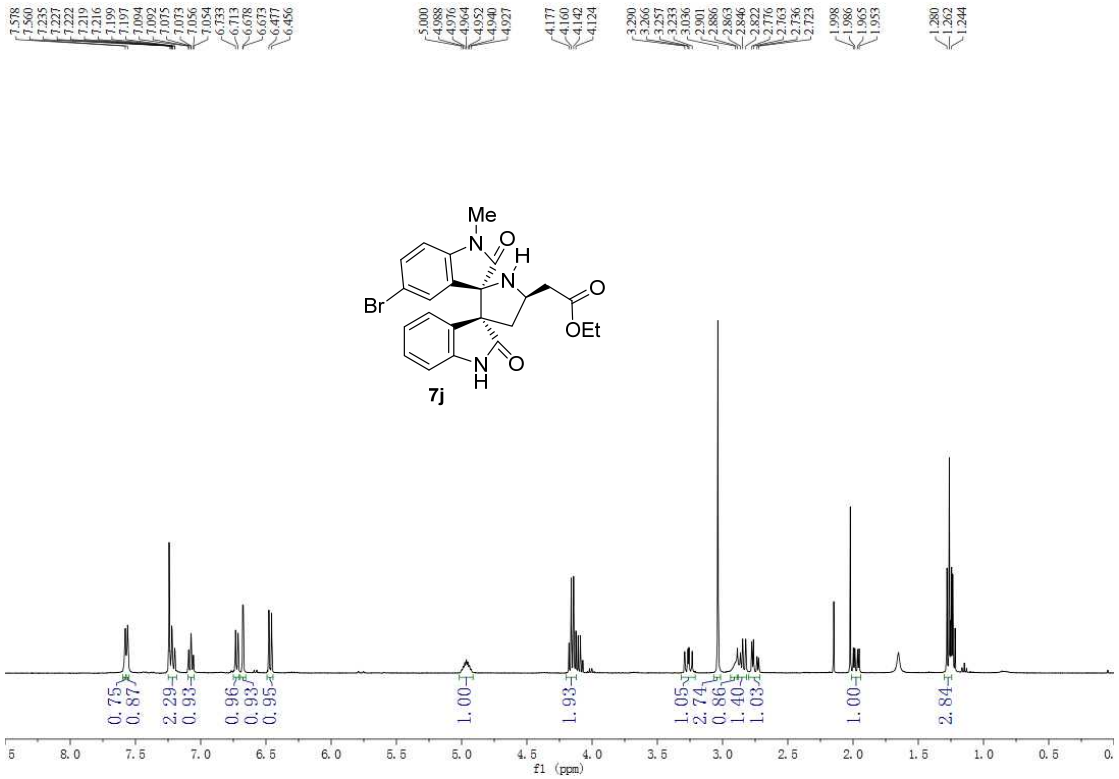
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	13.81	0.73	3.65	213.32	2.68
2	15.83	0.56	5.00	192.81	2.42
3	18.36	0.82	75.47	3722.18	46.78
4	20.17	0.83	2.16	107.49	1.35
5	22.99	0.80	77.38	3721.67	46.77
Total				7957.47	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	41.8	42.0
Flow in ml/min:	0.70	0.70

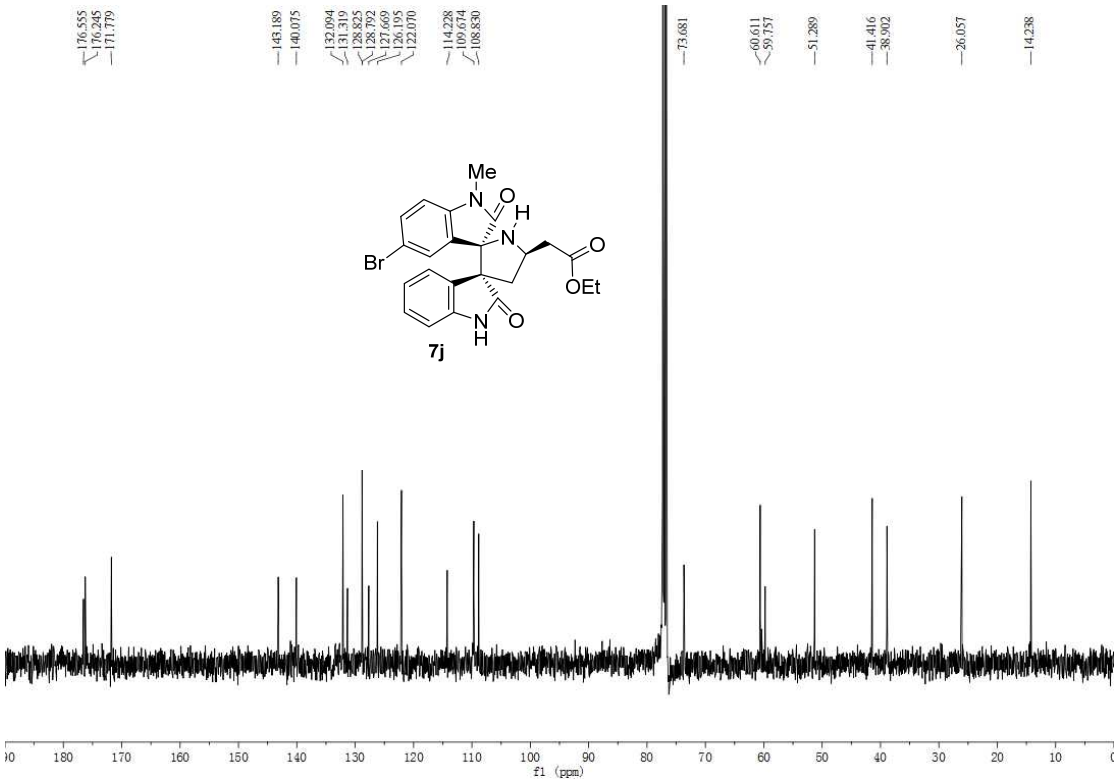


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	10.81	0.32	2.29	49.24	0.52
2	16.25	0.52	1.82	74.01	0.78
3	19.13	0.55	2.76	124.28	1.30
4	20.36	0.76	4.86	268.37	2.81
5	23.45	0.74	181.72	9023.39	94.59
Total				9539.29	100.00

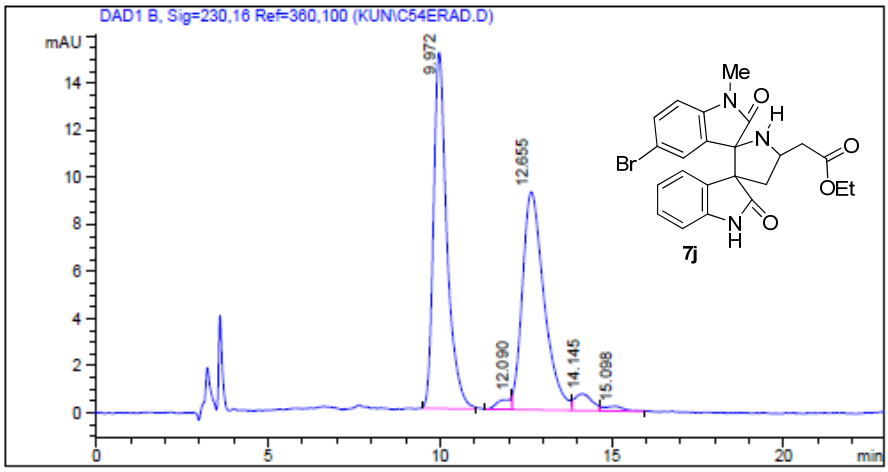
^{1}H NMR of **7j:**



^{13}C NMR of **7j:**

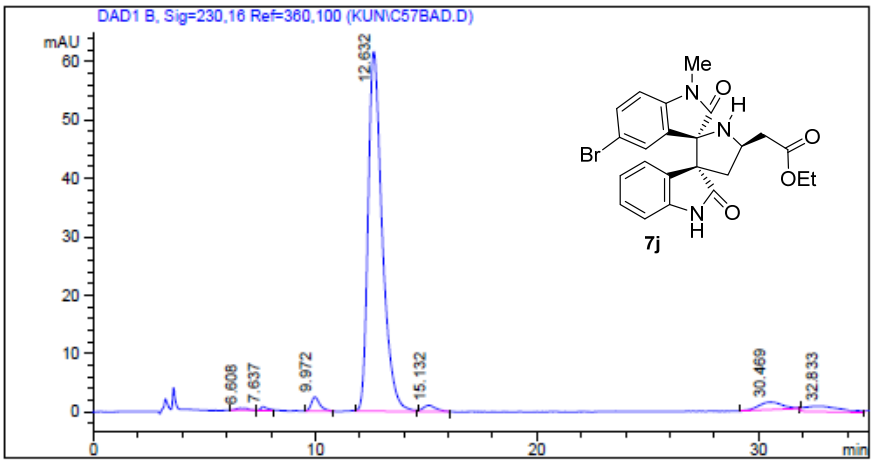


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	40.6	40.8
Flow in ml/min:	1.00	1.00



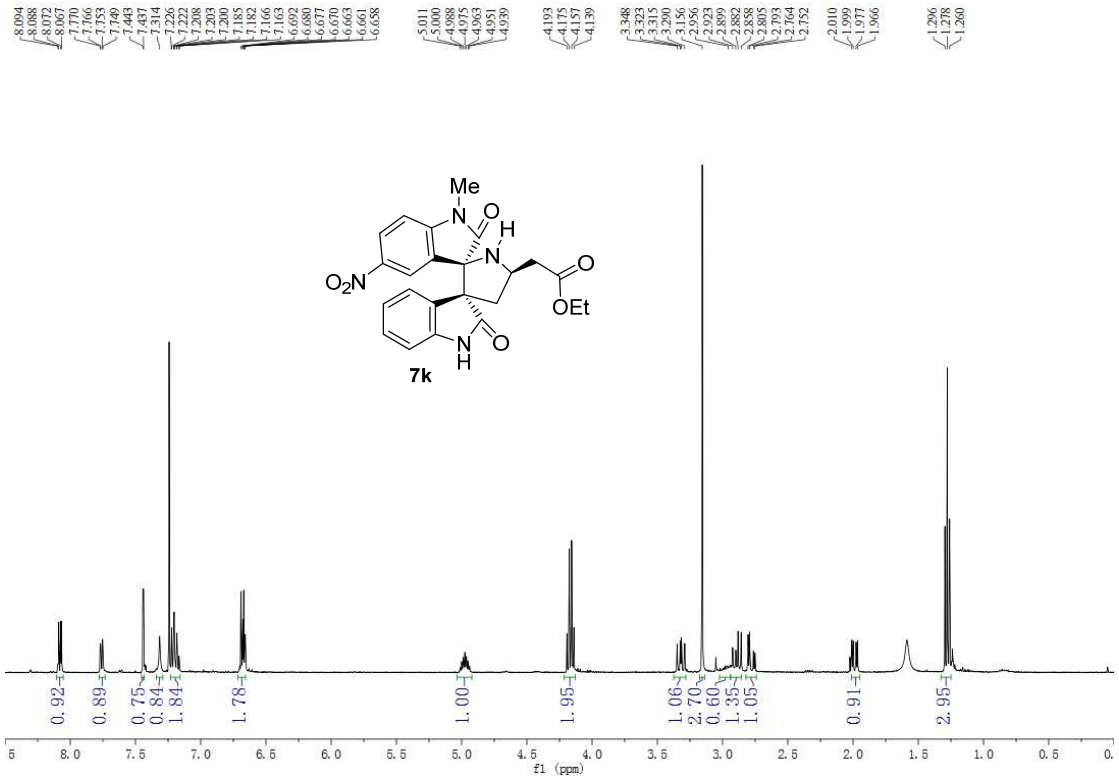
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	9.97	0.41	15.13	411.04	47.55
2	12.09	0.35	0.59	12.36	1.43
3	12.66	0.73	9.26	407.63	47.16
4	14.15	0.57	0.72	24.74	2.86
5	15.10	0.69	0.21	8.61	1.00
Total				864.39	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	40.3	40.6
Flow in ml/min:	1.00	1.00

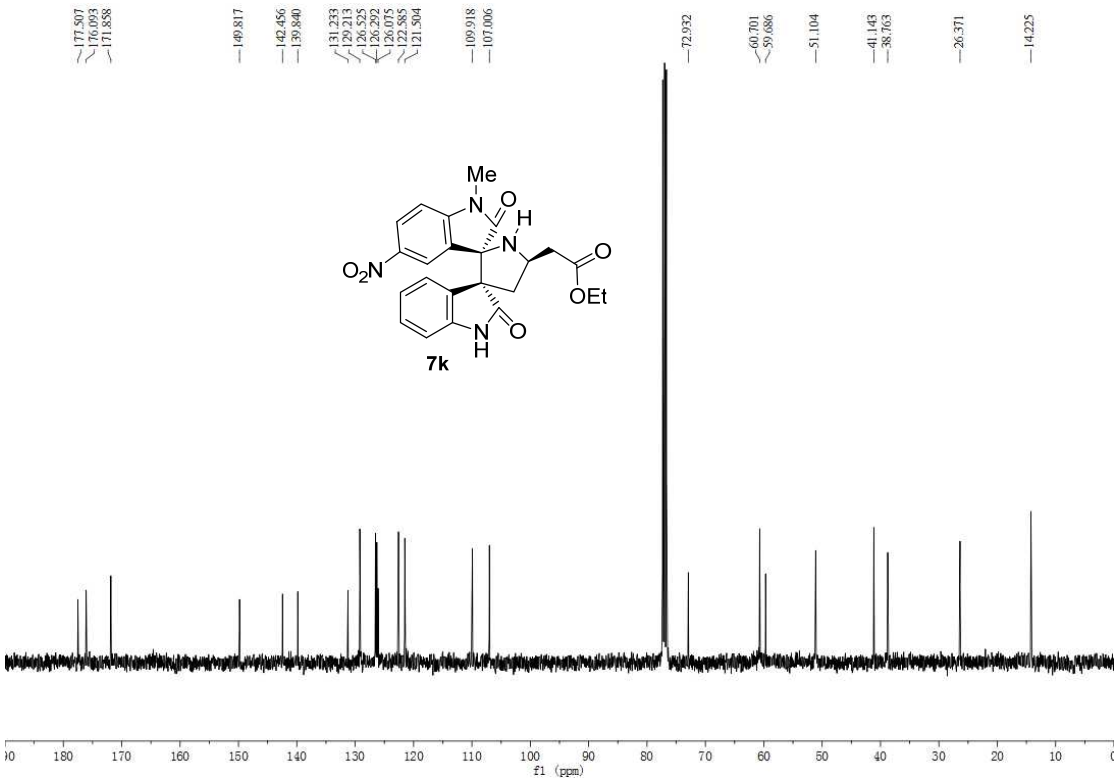


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.61	0.47	0.37	14.19	0.46
2	7.64	0.31	0.65	14.29	0.47
3	9.97	0.40	2.44	65.79	2.14
4	12.63	0.66	61.62	2721.99	88.67
5	15.13	0.50	1.09	44.80	1.46
6	30.47	0.89	1.32	98.53	3.21
7	32.83	1.89	0.97	110.10	3.59
Total				3069.68	100.00

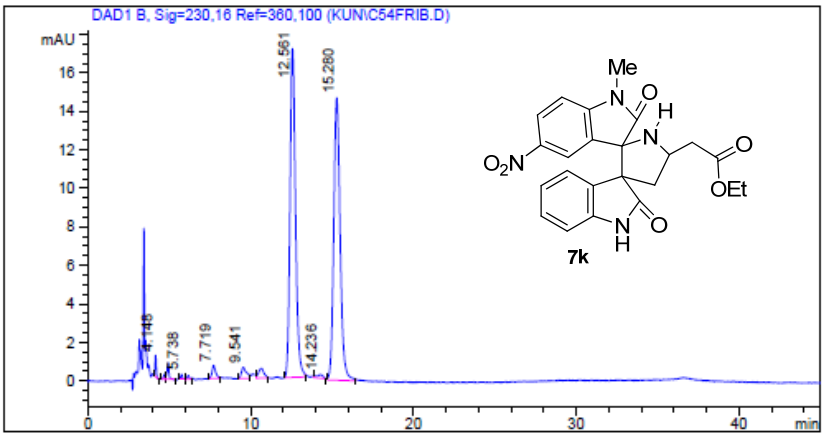
^{1}H NMR of **7k:**



^{13}C NMR of **7k:**

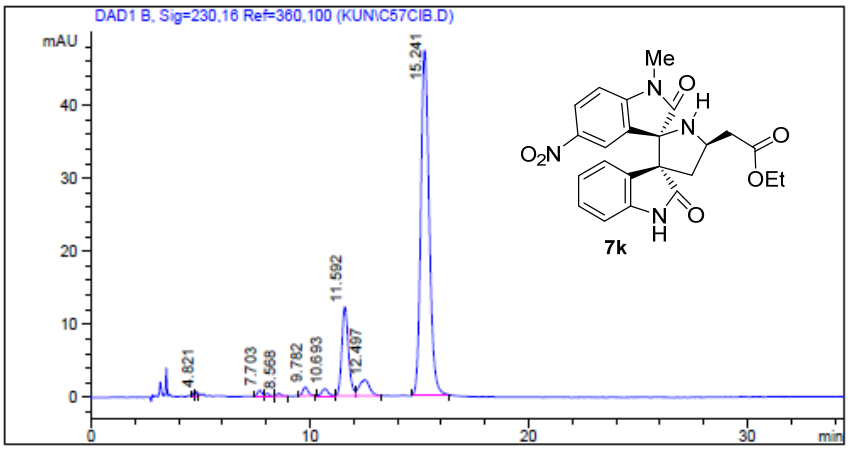


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	59.1	60.0
Flow in ml/min:	1.00	1.00



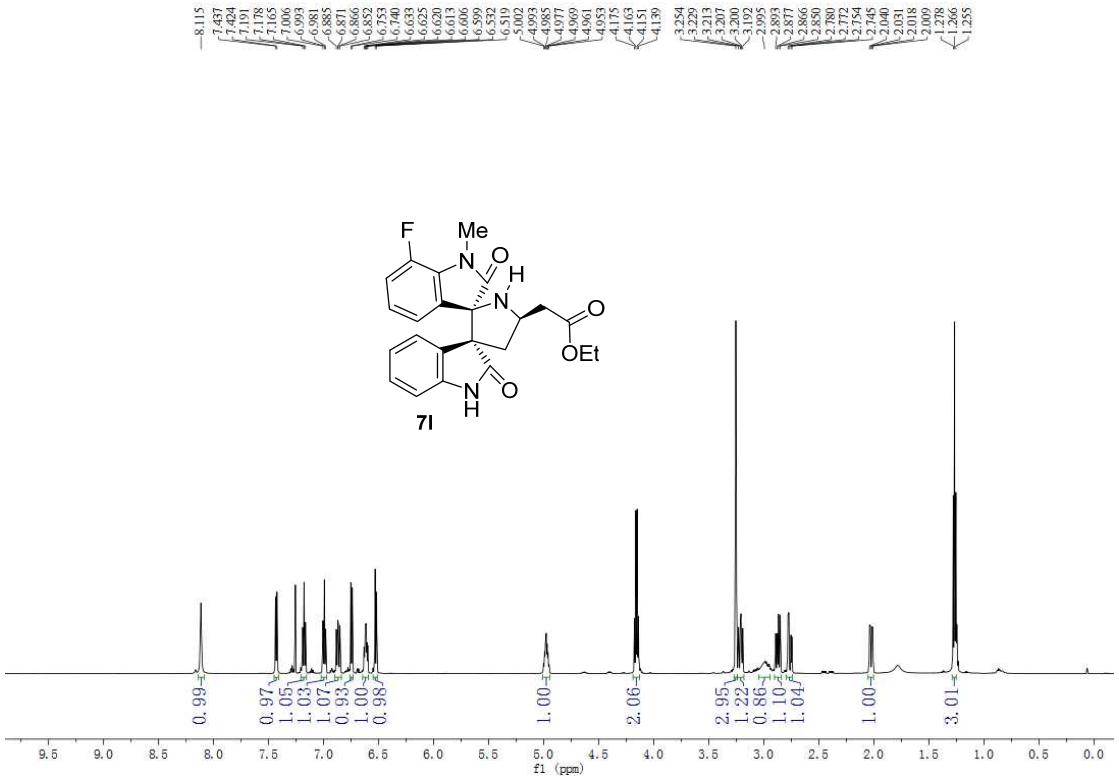
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.15	0.08	1.10	5.50	0.64
2	4.69	0.10	0.25	1.62	0.19
3	4.91	0.13	0.84	7.19	0.84
4	5.74	0.12	0.25	1.82	0.21
5	6.16	0.13	0.20	1.81	0.21
6	7.72	0.22	0.68	10.67	1.24
7	9.54	0.26	0.58	12.51	1.45
8	10.63	0.29	0.51	12.37	1.44
9	12.56	0.36	17.09	399.79	46.49
10	14.24	0.44	0.19	4.93	0.57
11	15.28	0.46	14.67	401.68	46.71
Total				859.88	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	59.2	59.7
Flow in ml/min:	1.00	1.00

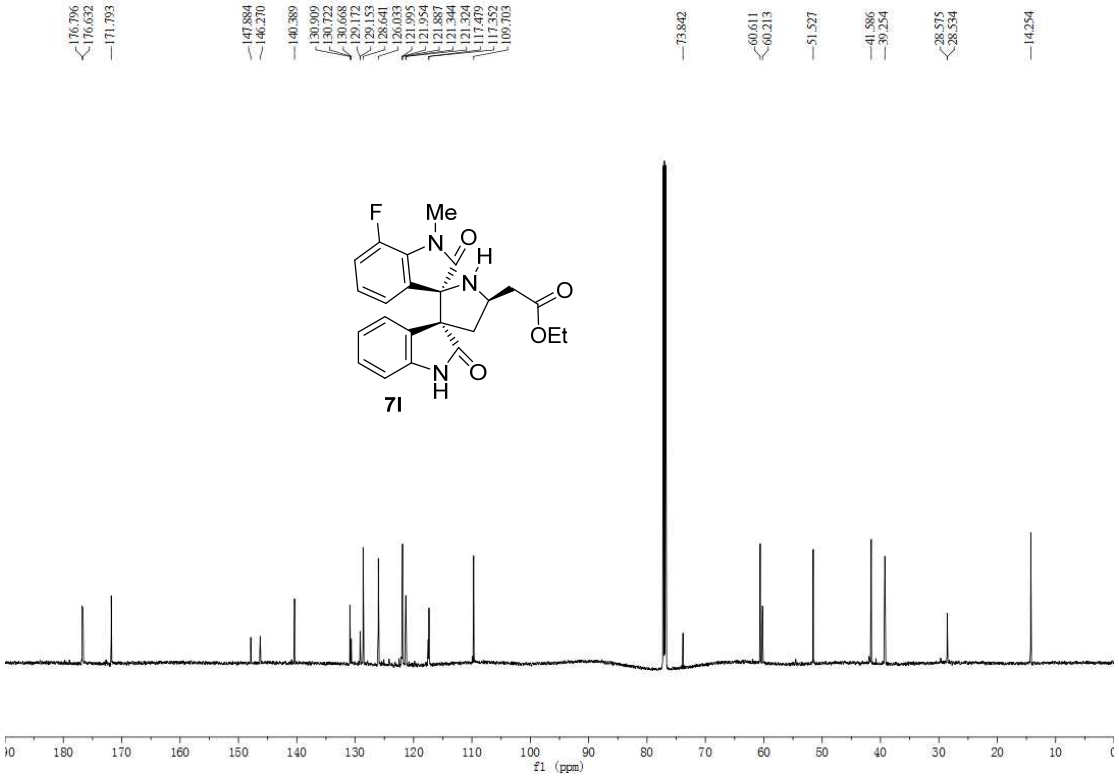


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.69	0.09	0.61	3.52	0.21
2	4.82	0.09	0.68	4.12	0.25
3	7.70	0.22	0.82	12.25	0.73
4	8.05	0.20	0.39	5.24	0.31
5	8.57	0.22	0.39	5.81	0.35
6	9.78	0.27	1.27	22.45	1.35
7	10.69	0.32	0.98	20.27	1.22
8	11.59	0.32	12.18	258.65	15.51
9	12.50	0.45	2.18	72.14	4.32
10	15.24	0.41	47.30	1263.69	75.75
Total				1668.14	100.00

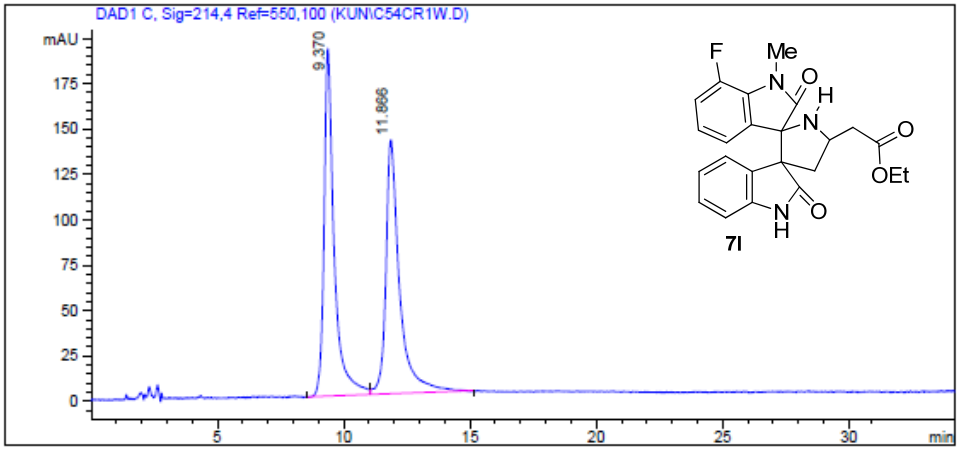
^{1}H NMR of **7l:**



^{13}C NMR of **7l:**

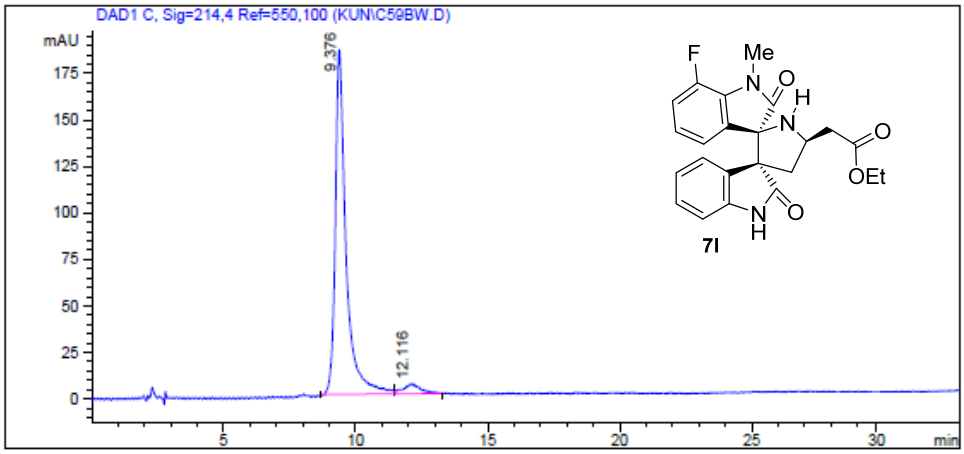


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	58.0	57.8
Flow in ml/min:	1.0	1.0



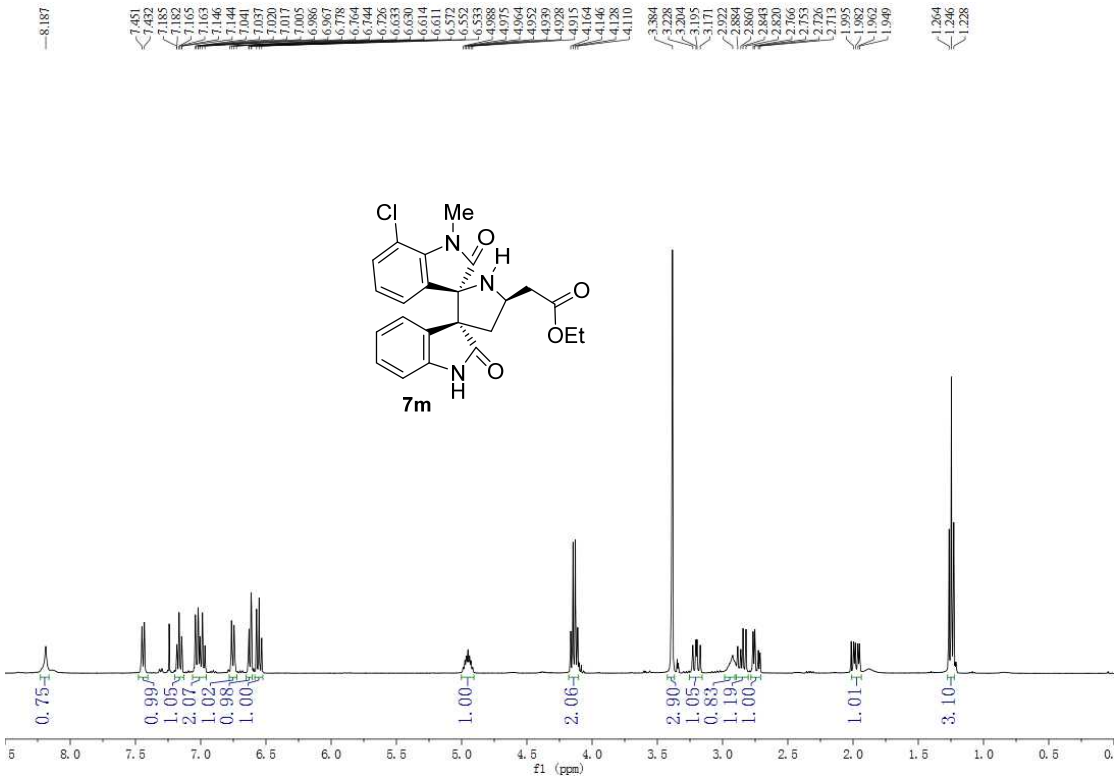
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	9.37	0.46	191.53	5320.62	50.30
2	11.87	0.63	139.71	5256.91	49.70
Total				10577.53	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	57.5	57.6
Flow in ml/min:	1.0	1.0

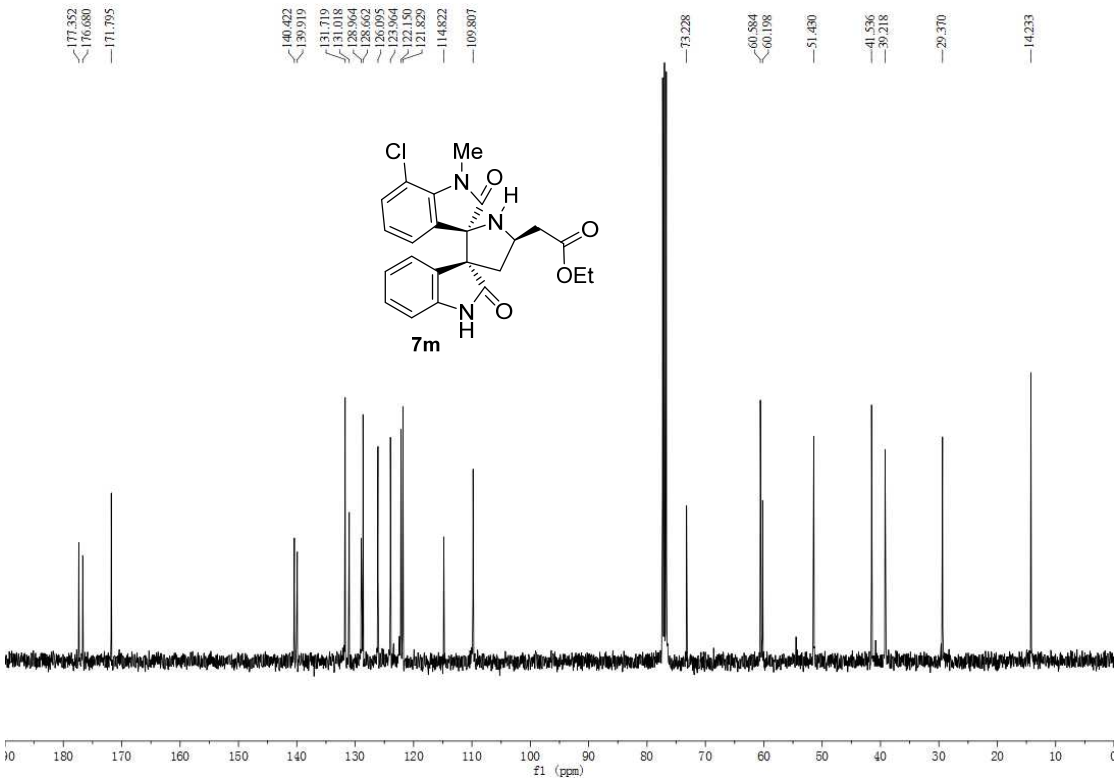


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	9.38	0.47	185.72	5234.88	95.43
2	12.12	0.76	5.51	250.97	4.57
Total				5485.85	100.00

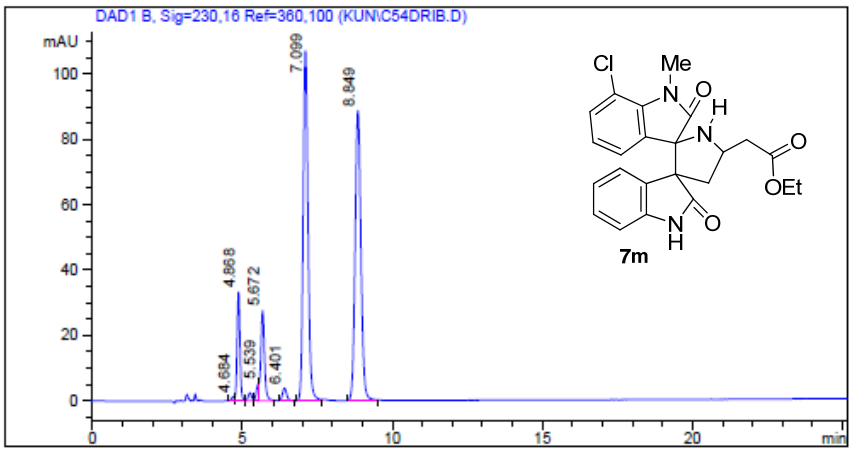
^{1}H NMR of **7m:**



^{13}C NMR of **7m:**

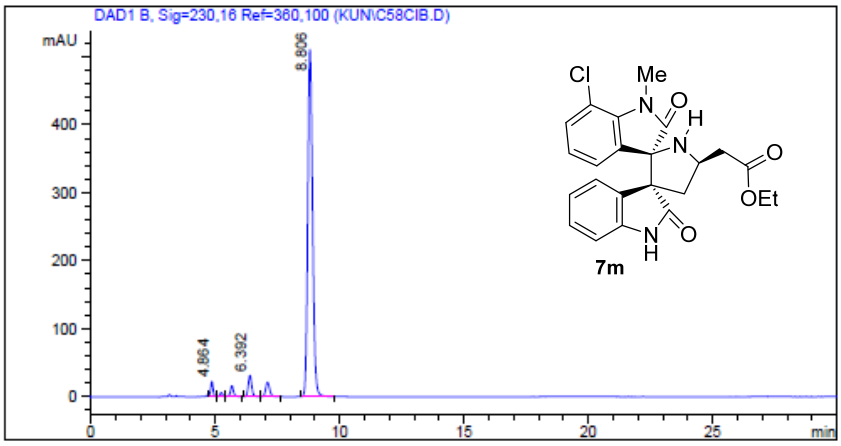


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	59.6	60.4
Flow in ml/min:	1.00	1.00



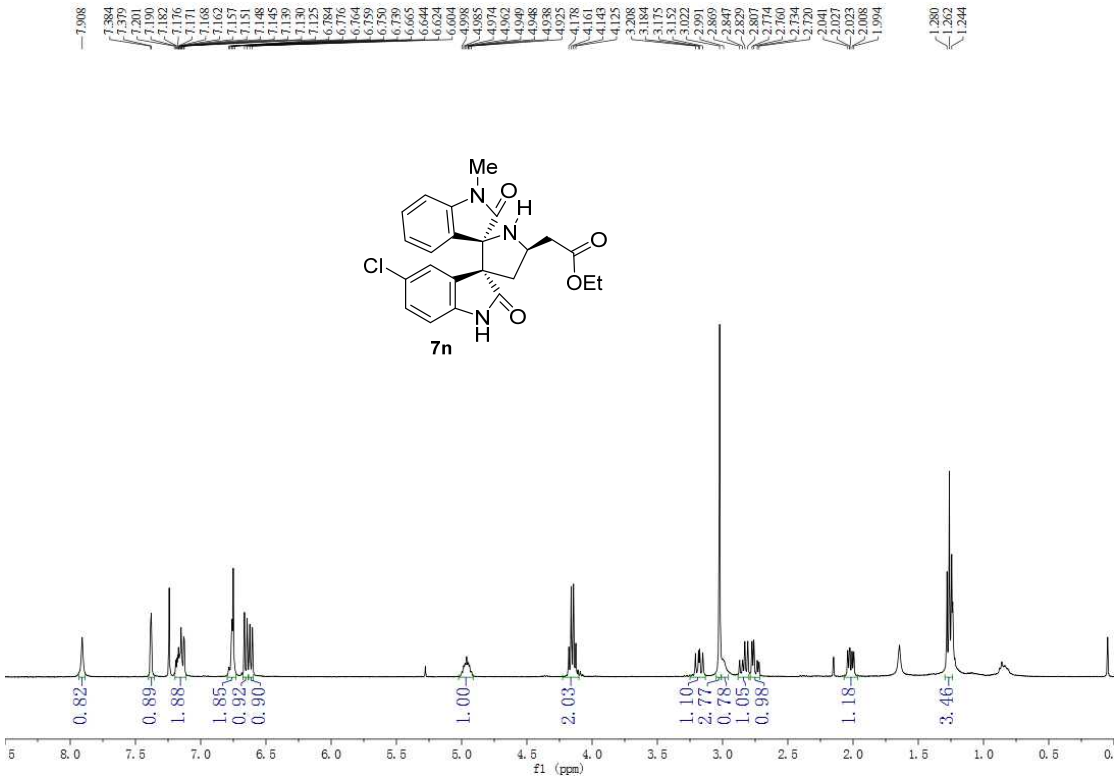
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.68	0.08	1.31	7.04	0.24
2	4.87	0.11	33.37	227.03	7.68
3	5.25	0.12	2.47	20.34	0.69
4	5.54	0.09	5.28	27.85	0.94
5	5.67	0.14	27.61	240.08	8.12
6	6.40	0.15	3.85	37.67	1.27
7	7.10	0.17	107.07	1196.87	40.50
8	8.85	0.21	88.75	1198.09	40.55
Total				2954.97	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	59.3	60.0
Flow in ml/min:	1.00	1.00

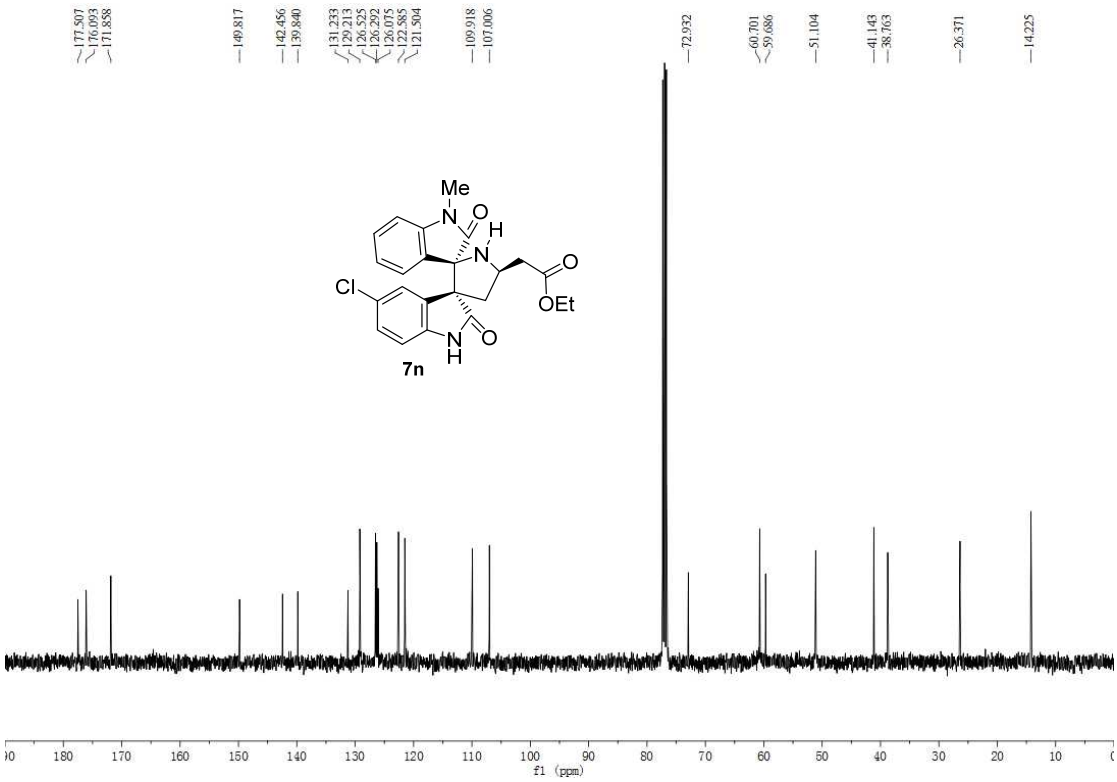


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.86	0.11	22.49	157.25	2.02
2	5.24	0.12	6.14	50.86	0.65
3	5.67	0.14	16.20	151.97	1.95
4	6.39	0.15	31.28	308.80	3.96
5	7.10	0.18	21.37	247.58	3.18
6	8.81	0.21	511.05	6876.91	88.24
Total				7793.38	100.00

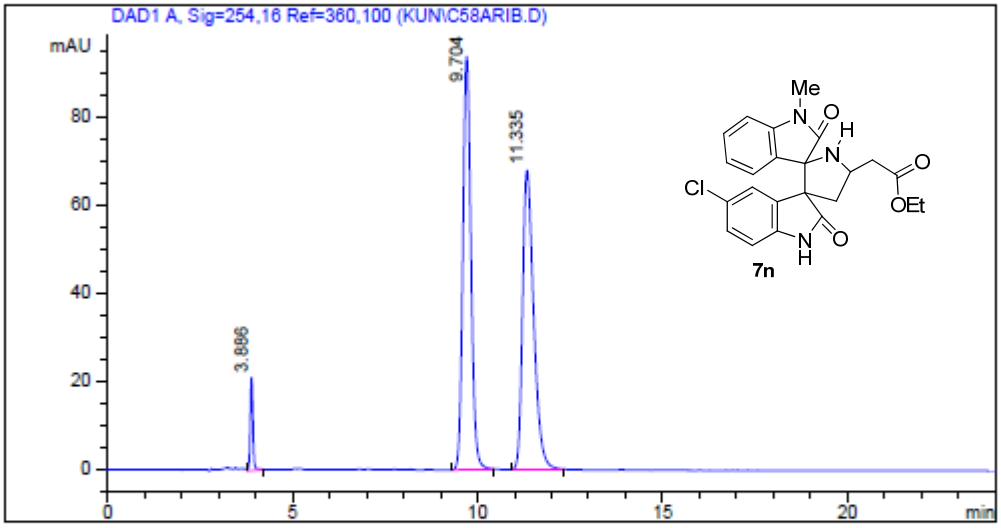
^{1}H NMR of **7n:**



^{13}C NMR of **7n:**

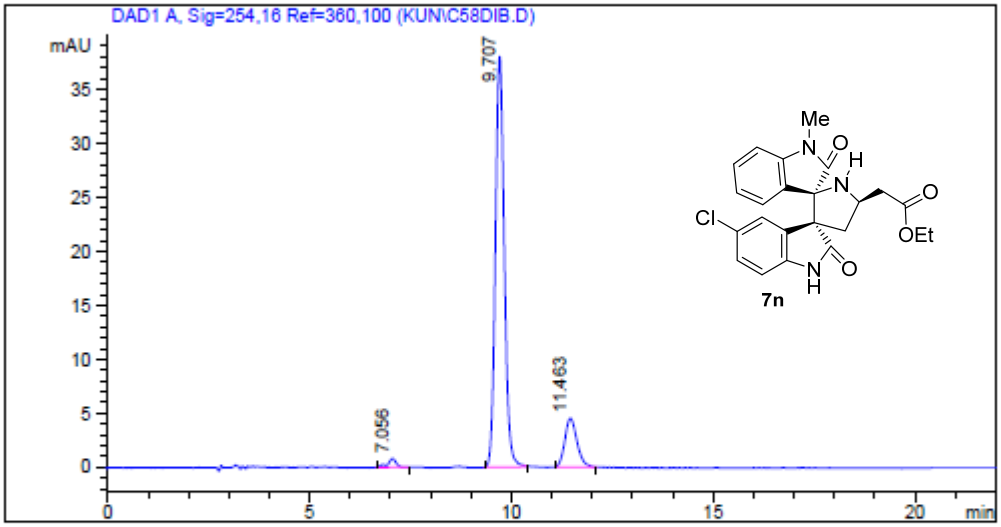


Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	57.5	57.9
Flow in ml/min:	1.00	1.00



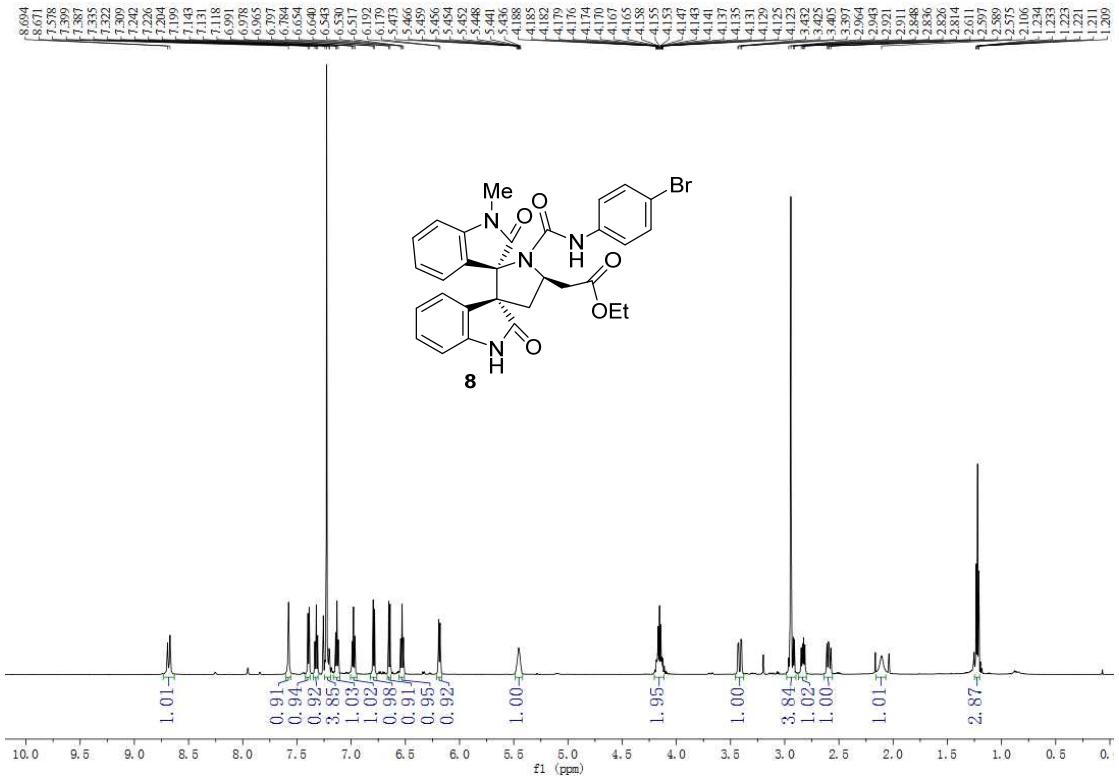
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.89	0.08	21.24	107.90	3.65
2	9.70	0.24	93.81	1426.79	48.30
3	11.33	0.32	67.89	1419.05	48.04
Total				2953.74	100.00

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	57.7	58.3
Flow in ml/min:	1.00	1.00



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	7.06	0.21	0.81	11.95	1.73
2	9.71	0.24	38.04	583.65	84.41
3	11.46	0.32	4.53	95.87	13.86
Total				691.47	100.00

^{1}H NMR of **8:**



^{13}C NMR of **8:**