Supporting Information

Alternating Current Polarity for Electrochemical De Novo Synthesis of 3,3'-Bisoxindoles

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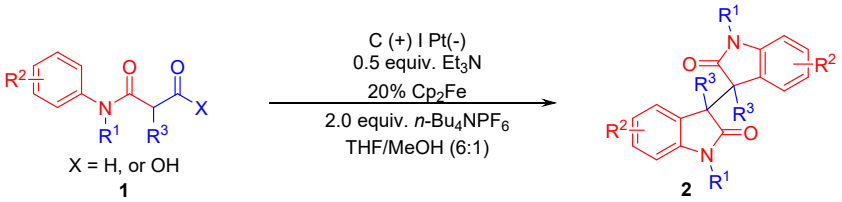
1.General information:

All reactions were carried out under Ar. Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were measured on Bruker AVIII 500M spectrometers with $CDCl_3$ as solvent and the residual protonated solvent as internal standard or 85% H_3PO_4 as external standard for ^{31}P NMR (202 MHz). Chemical shifts were reported in units (ppm) by assigning the residual protonated solvent of $CDCl_3$ resonance in the 1H spectrum as 7.26 ppm and $CDCl_3$ resonance in the ^{13}C spectrum as 77.16 ppm. All coupling constants (*J* values) were reported in Hertz (Hz). Chemical shifts of common trace ^{1}H NMR impurities (ppm): H_2O: 1.56, $CHCl_3$: 7.26. Column chromatography was performed on silica gel 300-400 mesh. The unknown products were further characterized by HRMS-ESI. High-resolution mass spectra (HRMS) were recorded with an Thermo Scientific Q Exactive Plus Orbitrap LC-MS/MS System by ESI on a quadrupole mass analyzer. All crystals were grown via a slow evaporation method.

Electrolysis experiments were performed using IKA Electrasyn 2.0. Graphite was cut into 1.5 x 10 x 0.3 cm^3 pieces before use, and was connected to electrical feed-through on the Teflon cap of the electrochemical cell via stainless steel electrode holder (purchased from Gaoss Union model SUS-1). Saturated calomel electrode (CHI150), platinum wire counter electrode (CHI115) and platinum working electrode (CHI102) were obtained from CH Instruments and Saturated calomel electrode was stored in 3.0 M KCl aqueous solution before use.

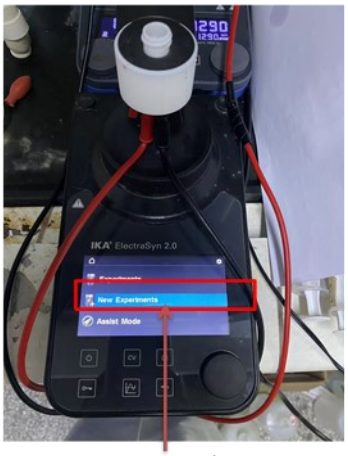
General Procedure A



An oven-dried 10 mL three-necked round-bottomed flask with a magnetic stir bar was charged with **1** (0.4 mmol, 1.0 equiv.), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol, 0.5 equiv.), indium triflate (22.4 mg, 0.04 mmol, 0.1 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with a graphite anode (15 mm x 10 mm x 4 mm) and a platinum plate cathode (10 mm x 10 mmx 0.2 mm). The cell was sealed and flushed with Argon for 10 minutes, followed by the addition via syringe of THF (6 mL) and MeOH (1 mL). The mixture was charged by constant current (I = ± 5 mA) under 80 ºC. The complete consumption of the starting material **1** was checked by TLC (20% - 50% AcOEt/petroleum ether). The reaction solution was concentrated in *vacuo* and extracted with EtOAc and H_2O (3×10 mL). The combined organic layer was dried over $MgSO_4$, filtered and concentrated in *vacuo*. The residue was purified by silica gel column chromatography using 20% to 40% AcOEt/petroleum ether as the eluent to give the corresponding products **2**.

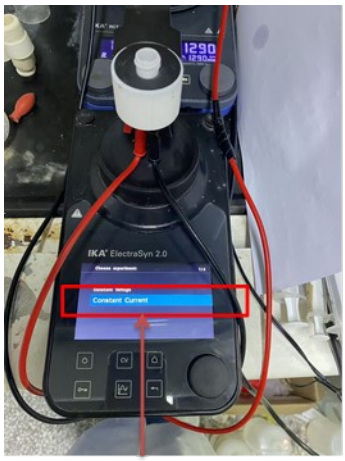
The detailed step-by-step procedure:

Step 1: Turn on the device



Step 2: Select
“New Experiments”

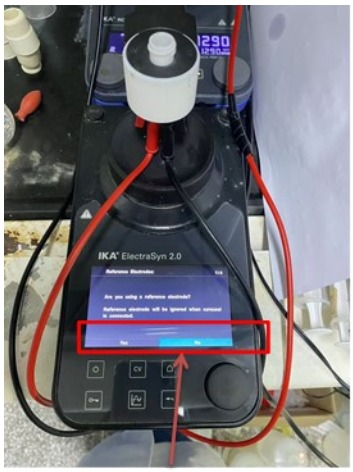
Step 3: Select
“Pulsed Alternating Polarity”



Step 4: Select
“Constant Current”

Step 5: Set the forward
current

Step 6: Set the negative
current

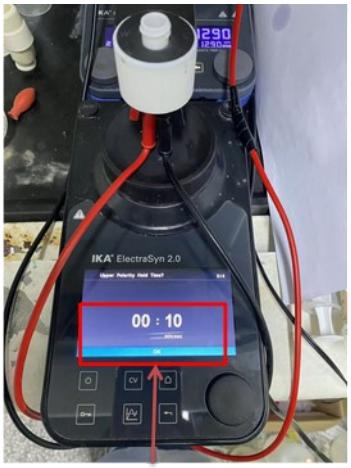


Step 7: Select “No”

Step 8: Select “Time”

Step 9: Set required time

Step 10: Set the amount of substrate in moles



Step 11: Set the time corresponding to the forward current

Step 12： Set the time corresponding to the negative current

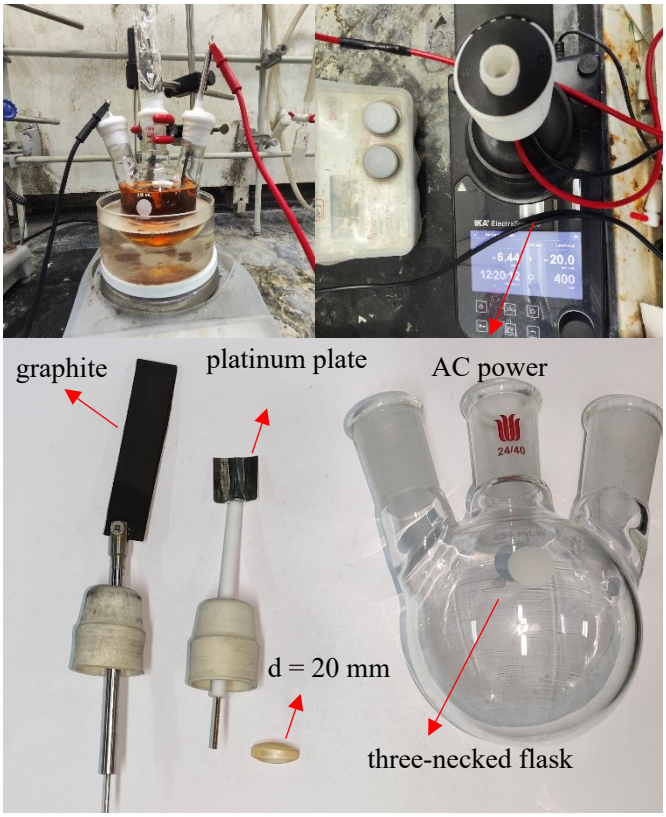
Step 13: Select “No”

Step 14: Select “Start”

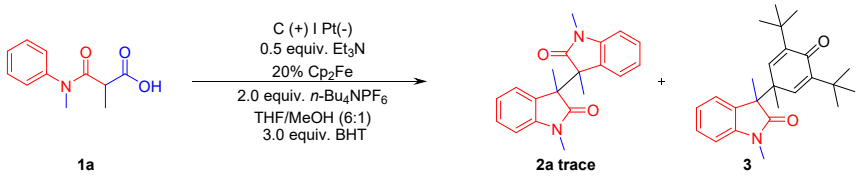
Step 15: It’s done!

Scale-up experiment: An oven-dried 250 mL three-necked round-bottomed flask with a magnetic stir bar was charged with **1d** (5.9 mmol, 1.68 g, 1.0 equiv.), Cp_2Fe (223.2 mg, 1.2 mmol, 0.2 equiv.), triethylamine (409 μL, 2.95 mmol, 0.5 equiv.), indium triflate (331.6 mg, 0.59 mmol, 0.1 equiv.) and *n*-Bu_4NPF_6 (4.57 g, 11.8 mmol, 2.0 equiv.). The flask was equipped with a graphite anode (40 mm x 10 mm x 3 mm) and a platinum plate cathode (20 mm x 20 mm x 1 mm). The cell was sealed and flushed with Argon for 15 minutes, followed by the addition via syringe of THF (174 mL) and MeOH (29 mL). The mixture was charged by constant current (I = ± 20 mA). The complete consumption of the starting material **1d** was checked by TLC (70 % AcOEt/petroleum ether). The reaction solution was concentrated in vacuo and extracted with EtOAc and H_2O (3x10 mL). The combined organic layer was dried over $MgSO_4$, filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography to give the corresponding products **2d** (879 mg, 1.86 mmol, 63%).

Gram-scale experiment:

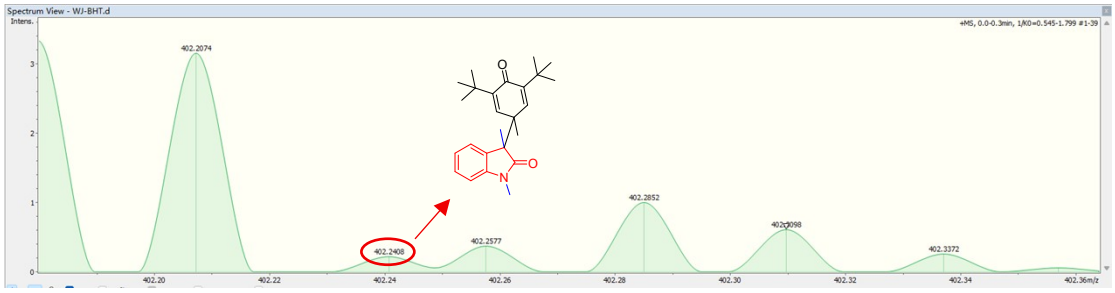

graphite
platinum plate
AC power
d = 20 mm
three-necked flask

2. Control experiments

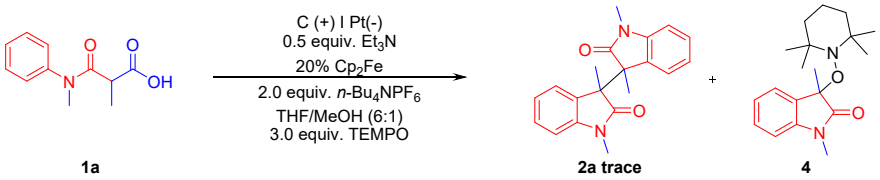


An oven-dried 10 mL three-necked round-bottomed flask with a magnetic stir bar was charged with **1a** (41.4 mg, 0.2 mmol, 1.0 equiv.), Cp_2Fe (7.5 mg, 0.04 mmol, 0.2 equiv.), triethylamine (14 μL, 0.1 mmol, 0.5equiv.), indium triflate (11.2 mg, 0.04 mmol, 0.1 equiv.), *n*-Bu_4NPF_6 (155 mg, 0.4 mmol, 2.0 equiv.) and butylated hydroxytoluene (BHT) (132mg, 0.6 mmol, 3.0 equiv.). The flask was equipped with a graphite anode (15 mm x 10 mm x 4 mm) and a platinum plate cathode (10 mm x 10 mmx 0.2 mm). The cell was sealed and flushed with Argon for 10 minutes, followed by the addition via syringe of THF (6 mL) and MeOH (1 mL). The mixture was charged by constant current (I = ± 5 mA). Only trace amount of **2a** was detected. The crude mixture was analyzed by TIMS.

The mixture was checked by TIMS:

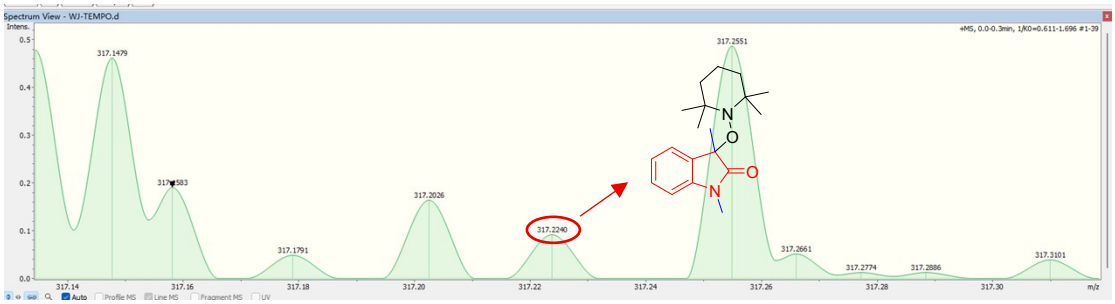


HRMS-ESI: Calcd for $C_{25}H_{33}NNaO_2^+$ [M+Na]$^+$402.2404, found 402.2408.

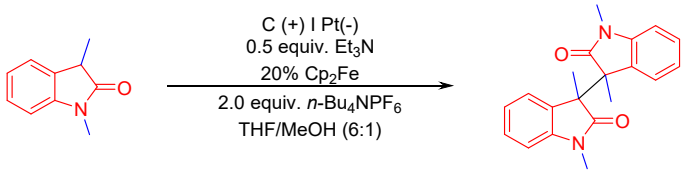


An oven-dried 10 mL three-necked round-bottomed flask with a magnetic stir bar was charged with **1a** (41.4 mg, 0.2 mmol, 1.0 equiv.), Cp_2Fe (7.5 mg, 0.04 mmol, 0.2 equiv.), triethylamine (14 μL, 0.1 mmol, 0.5equiv.), indium triflate (11.2 mg, 0.04 mmol, 0.1 equiv.), *n*-Bu_4NPF_6 (155 mg, 0.4 mmol, 2.0 equiv.) and 2,2,6,6-tetramethylpiperidinyloxy (TEMPO) (93.6 mg, 0.6 mmol, 3.0 equiv.).The flask was equipped with a graphite anode (15 mm x 10 mm x 4 mm) and a platinum plate cathode (10 mm x 10 mmx 0.2 mm). The cell was sealed and flushed with Argon for 10 minutes, followed by the addition via syringe of THF (6 mL) and MeOH (1 mL). The mixture was charged by constant current (I = ± 5 mA). Only trace amount of **2a** was detected. The crude mixture was analyzed by TIMS.

The mixture was checked by TIMS:



HRMS-ESI: Calcd for $C_{19}H_{29}N_2O_2^+$ $[M+H]^+$317.2224, found 317.2240.

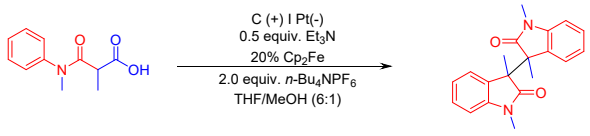


5 **2a 46%**

An oven-dried 10 mL three-necked round-bottomed flask with a magnetic stir bar was charged with **5** (32.2 mg, 0.2 mmol, 1.0 equiv.), Cp_2Fe (7.5 mg, 0.04 mmol, 0.2 equiv.), triethylamine (14 μL, 0.1 mmol, 0.5 equiv.), indium triflate (11.2 mg, 0.02 mmol, 0.1 equiv.) and *n*-Bu_4NPF_6 (155 mg, 0.4 mmol, 2.0 equiv.). The flask was equipped with a graphite anode (15 mm x 10 mm x 4 mm) and a platinum plate cathode (10 mm x 10 mmx 0.2 mm). The cell was sealed and flushed with Argon for 10 minutes, followed by the addition via syringe of THF (6 mL) and MeOH (1 mL). The mixture was charged by constant current (I = ± 5 mA). The complete consumption of the starting material **5** was checked by TLC (30 % AcOEt/petroleum ether). The reaction solution was concentrated in *vacuo* and extracted with EtOAc and H_2O (3×10 mL). The combined organic layer was dried over $MgSO_4$, filtered and concentrated in *vacuo*. The residue was purified by silica gel column chromatography using 20% to 40% AcOEt/petroleum ether as the eluent to give **2a** (15 mg, 0.046 mmol, *dr* = 50 : 50, 46%) as colorlesssolid.

3. Reaction Optimization

3.1 Reaction optimization under alternating polarity current



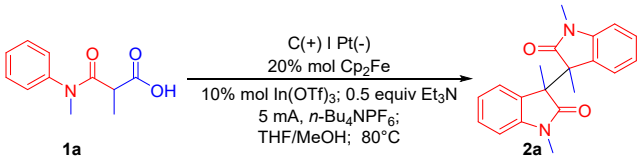
1a **2a**

Entry	Variation from the standard conditions	Yield (%)[b]
1	none	70%
2	30% Cp_2Fe instead of 20% Cp_2Fe	65%
3	10% Cp_2Fe instead of 20% Cp_2Fe	25%
4	Without Cp_2Fe	n. d.
5	$Cu(CF_3SO_3)_2$ instead of $In(CF_3SO_3)_3$	41%
6	$Y(CF_3SO_3)_3$ instead of $In(CF_3SO_3)_3$	69%
7	$Mn(CF_3SO_3)_2$ instead of $In(CF_3SO_3)_3$	69%
8	$Ni(CF_3SO_3)_2$ instead of $In(CF_3SO_3)_3$	44%
9	$Sc(CF_3SO_3)_3$ instead of $In(CF_3SO_3)_3$	53%
10	$Fe(CF_3SO_3)_2$ instead of $In(CF_3SO_3)_3$	49%
11	Without $In(CF_3SO_3)_3$	13%
12	NaOH instead of Et_3N	n.d.
13	$NaHCO_3$ instead of Et_3N	n.d.
14	Pyridine instead of Et_3N	n.d.
15	none Et_3N	n.d.
16	0.2 equiv instead of 0.5 equiv Et_3N	31%
17	0.4 equiv instead of 0.5 equiv Et_3N	56%
18	0.7 equiv instead of 0.5 equiv Et_3N	47%
19	1.0 equiv instead of 0.5 equiv Et_3N	31%
20	3.0 mA (10s) / -3.0 mA (5s) instead of 5.0 mA (10s) / -5.0 mA (5s)	43%
21	1.0 mA (10s) / -1.0 mA (5s) instead of 5.0 mA (10s) / -5.0 mA (5s)	50 %
22	7.0 mA (10s) / -7.0 mA (5s) instead of 5.0 mA (10s) / -5.0 mA (5s)	25%
23	n-Bu_4NOAc instead of *n*-Bu_4NPF_6	n. d.
24	n-Bu_4NBF_4 instead of n-Bu_4NPF_6	53%
25	CH_3CN : MeOH instead of THF : MeOH	63%
26	DMF : MeOH instead of THF : MeOH	47%
27	THF : H_2O instead of THF : MeOH	53%
28	THF : HOAc instead of THF : MeOH	34%
29	60 ℃ instead of 80 ℃	50%
30	100 ℃ instead of 80 ℃	38%
31	n-Bu_4NOAc instead of n-Bu_4NPF_6	n. d.
32	n-Bu_4NBF_4 instead of n-Bu_4NPF_6	53%
32	1/3 Hz; Duty ratio: 67%	50%
34	1/9 Hz; Duty ratio: 67%	38%
35	1/15 Hz; Duty ratio: 33%	16%
36	1/15 Hz; Duty ratio: 53%	36%

[a]Reaction conditions: undivided cell, a graphite anode (15 mm x 10 mm x 4 mm) and a platinum plate cathode (10 mm x 10 mmx 0.2 mm), constant current (I = ± 5 mA), 1a (41.4 mg, 0.2 mmol, 1.0 equiv.), Cp_2Fe (7.5 mg, 0.04 mmol, 0.2 equiv.), triethylamine (14 μL, 0.1 mmol, 0.5equiv.), indium triflate (11.2 mg, 0.04 mmol, 0.1 equiv.), *n*-Bu_4NPF_6 (155 mg, 0.4 mmol, 2.0 equiv.), THF (6 mL) and MeOH (1 mL), rt, in Ar. [b]Isolated yield.

3.2 Reaction optimization under constant current

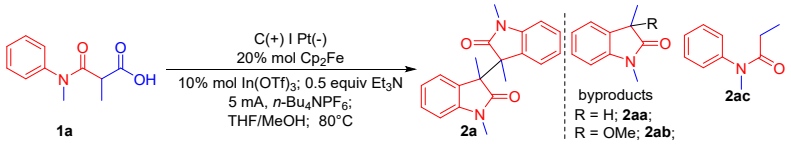
General Procedure B



An oven-dried 10 mL three-necked round-bottomed flask with a magnetic stir bar was charged with **1a** (0.4 mmol, 1.0 equiv.), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 µL, 0.2 mmol, 0.5 equiv.), indium triflate (22.4 mg, 0.04 mmol, 0.1 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with a graphite anode (15 mm x 10 mm x 4 mm) and a platinum plate cathode (10 mm x 10 mmx 0.2 mm). The cell was sealed and flushed with Argon for 10 minutes, followed by the addition via syringe of THF (6 mL) and MeOH (1 mL). The mixture was charged by constant current (I = 5 mA) under 80 ºC. The complete consumption of the starting material **1a** was checked by TLC (20% - 50% AcOEt/petroleum ether). The reaction solution was concentrated in *vacuo* and extracted with EtOAc and H_2O (3×10 mL). The combined organic layer was dried over $MgSO_4$, filtered and concentrated in *vacuo*. The residue was purified by silica gel column chromatography using 20% to 40% AcOEt/petroleum ether as the eluent to give the corresponding products **2a** (24.6 mg, 0.078 mmol, *dr* = 50 : 50, 39%) as colorless solid.

We initiated our study using 2-methyl-3-(methyl(phenyl)amino)-3-oxopropanoic acid **1a** as a model substrate. By utilizing Cp_2Fe as the mediator and $In(OTf)_3$ as the Lewis acid, bisoxindole **2a** was obtained in 39% yield under electrolysis at constant current of 5 mA **(Table S1, entry 1)**. Remarkably, the formation of three byproducts (**2aa**-**2ac**) were observed, likely arising from overoxidation of oxindole-derived radicals (**2aa**, **2ab**). The reaction halted after three hours of electrolysis due to the formation of unknown black deposit on the cathode surface, resulting in incomplete conversion of **1a**. To mitigate overoxidation of the radical intermediates, a lower current density was applied; however, a comparable yield of **2a** was obtained **(entry 2)**, indicating that reducing the current density under constant current conditions did not effectively suppress overoxidation. To improve the yield of **2a**, extensive optimization of current density, base, Cp_2Fe loading, Lewis acids, electrolyte, solvent, and electrode materials under constant current conditions were undertaken. Unfortunately, **2a** was obtained in only acceptable yields in most cases, accompanied by the formation of byproducts (**2aa**–**2ac**).

Table S1. Optimization of the Reaction Conditions under Constant Current[a]

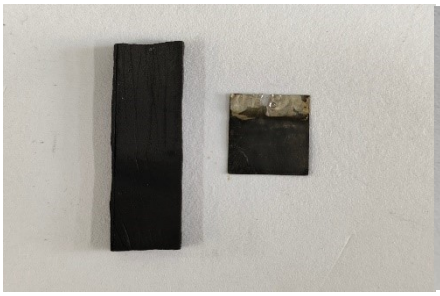


Entry	Variation from the standard conditions	Yield(%)[b]
1	None	39
2	3.0 mA instead of 5.0 mA	37
3	7.0 mA instead of 5.0 mA	31
4	without Et_3N	n.d.
5	10% Cp_2Fe instead of 20% Cp_2Fe	25
6	30% Cp_2Fe instead of 20% Cp_2Fe	35
7	without $In(OTf)_3$	10
8	$Mn(OTf)_2$ instead of $In(OTf)_3$	38
9	$Cu(OTf)_2$ instead of $In(OTf)_3$	31
10	*n*-Bu_4NBF_4 instead of *n*-Bu_4NPF_6	34
11	CH_3CN/MeOH instead of THF/MeOH	37
12	THF/H_2O instead of THF/MeOH	22
13	C(+)|C(-) instead of C(+)|Pt(-)	28
14	Pt(+)|Pt(-) instead of C(+)|Pt(-)	25
15	without electricity	n.r.

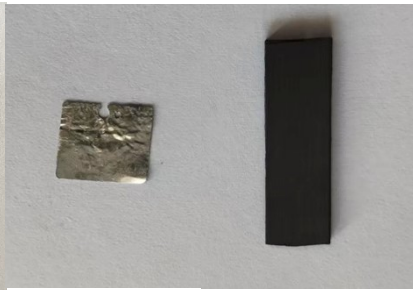
[a]Reaction conditions: undivided cell, graphite felt (25 mm x 10 mm x 4 mm), platinum cathode (10mm x 10 mm x 0.2 mm), **1a** (0.2 mmol, 1.0 equiv.), Cp_2Fe (0.04 mmol, 0.2 equiv.), Et_3N (0.1 mmol, 0.5 equiv.), $In(OTf)_3$ (0.02 mmol, 0.1 equiv.), THF : MeOH (6 mL : 1 mL), *n*-Bu_4NPF_6 (0.4 mmol, 2.0 equiv.), I = 5 mA, 80°C, in Ar. [b]Isolated yield, *dr*=50:50, determined by isolated yields.

3.3 The surfaces of graphite anode and platinum plate cathode under constant current and alternating polarity current

after constant current

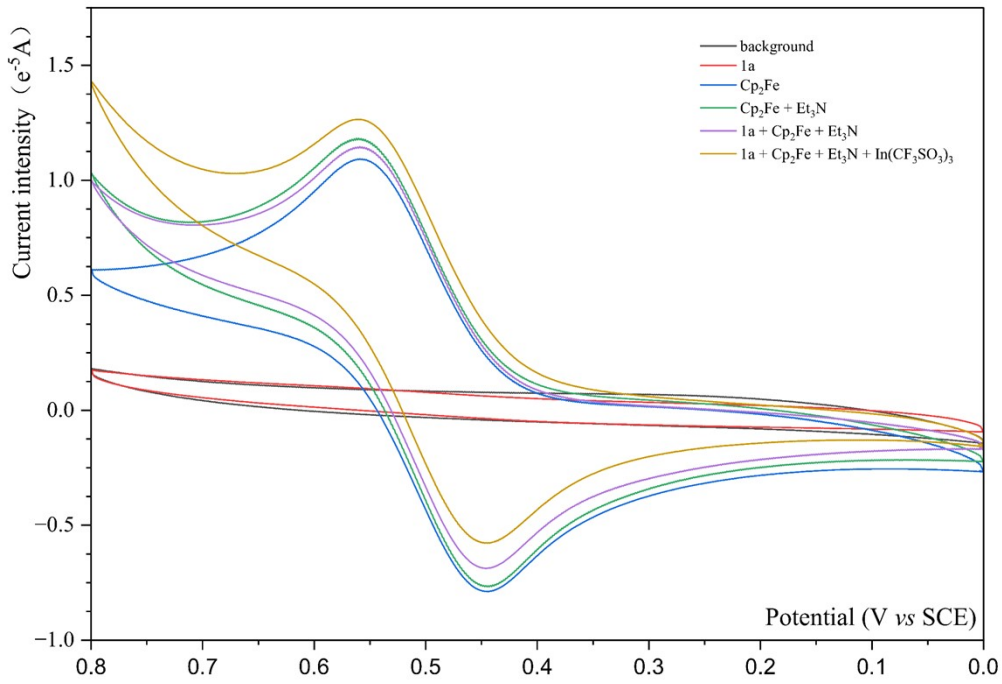


after alternating polarity current



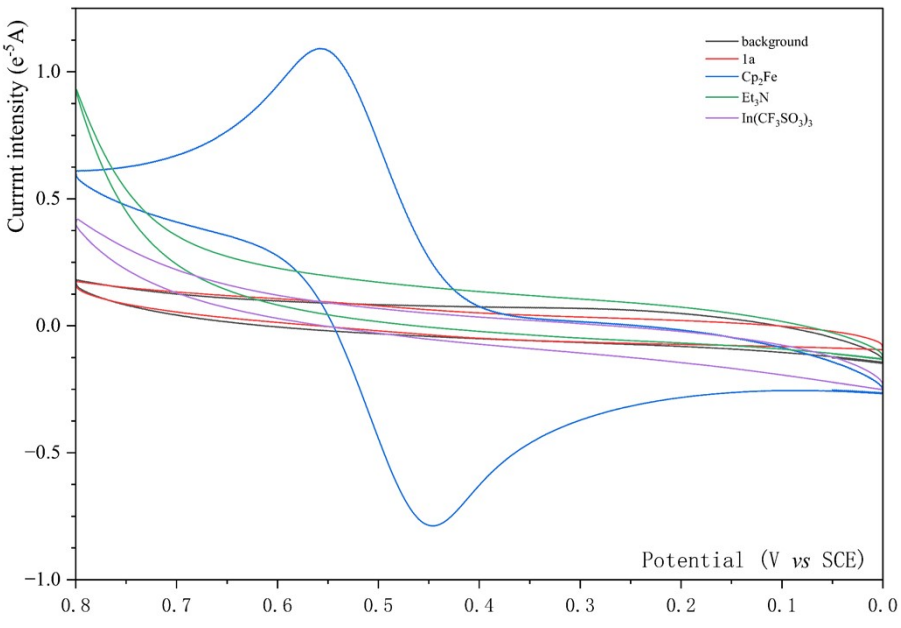
4. Cyclic voltammetry

Cyclic voltammetry was performed with CHI760E Electrochemical Workstation using the cyclic voltammetry mode. A platinum disc (diameter 3 mm) working electrode, a platinum wire counter electrode and a reference electrode (saturated calomel electrode (in a 3.0 M KCl aqueous solution)) were used at a scan rate of 100 mV/s. All electrodes are purchased from CH Instruments. The experiments were conducted in a 25 mL four neck vial without stirring in THF+MeOH (17 mL+3 mL) with *n*-Bu_4NPF_6 (0.1 M) as electrolyte under Ar.



Background: 0.1 M *n*-Bu_4NPF_6 in THF+MeOH (6:1) under Ar; 1.0 mM Cp_2Fe; 4.0 mM Et_3N; 0.25 mM $In(OTf)_3$; and 5.0 mM **1a**.

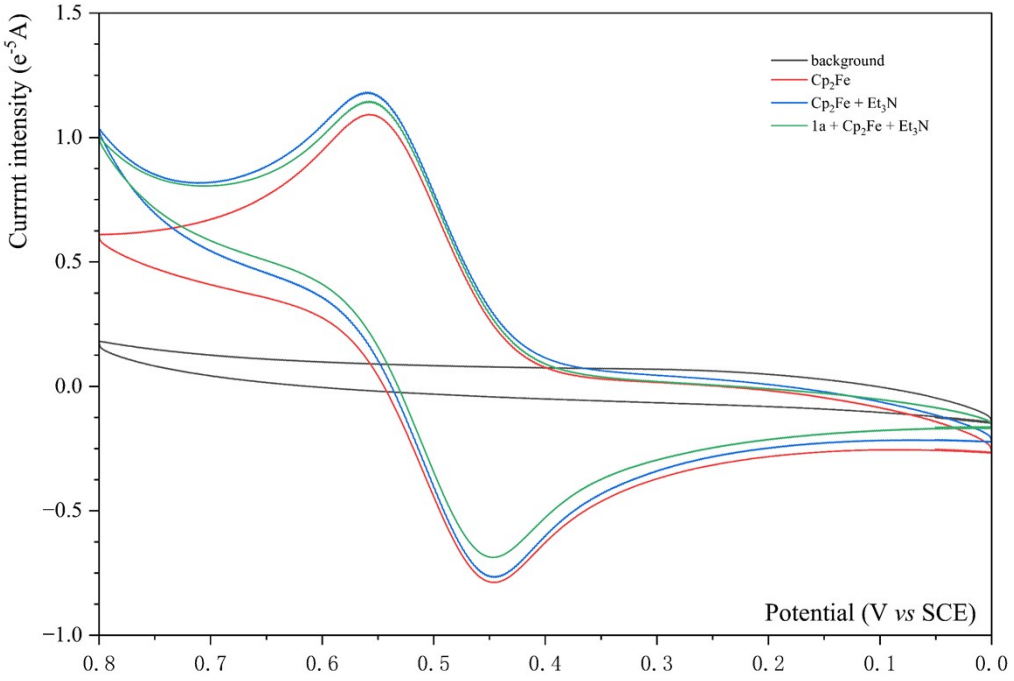
Figure S1: Cyclic voltammetry of ***1a****, Cp_2Fe, Et_3N +Cp_2Fe,* ***1a****+Et_3N +Cp_2Fe,* ***1a****+$In(OTf)_3$+Cp_2Fe, +Et_3N, in the [0V, 0.8V] range*



Backgr ound:

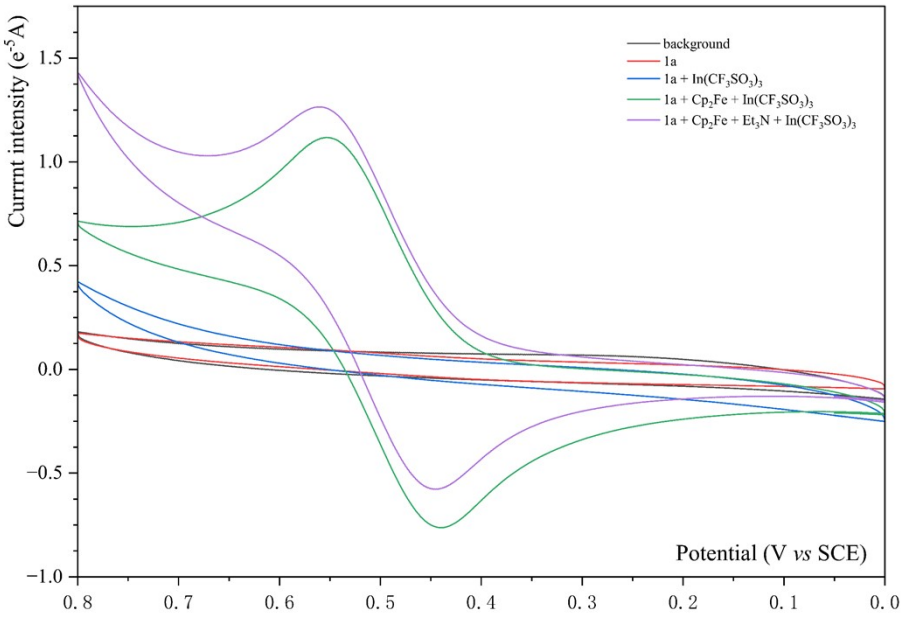
0.1 M *n*-Bu_4NPF_6 in THF+MeOH under Ar; Cp_2Fe (1.0 mM); Et_3N (4.0 mM); $In(OTf)_3$ (0.25 mM); **1a** (5.0 mM).

*Figure S2: Cyclic voltammetry of **1a**, Cp_2Fe, Et_3N, $In(OTf)_3$ in the [0V, 0.8V] range*



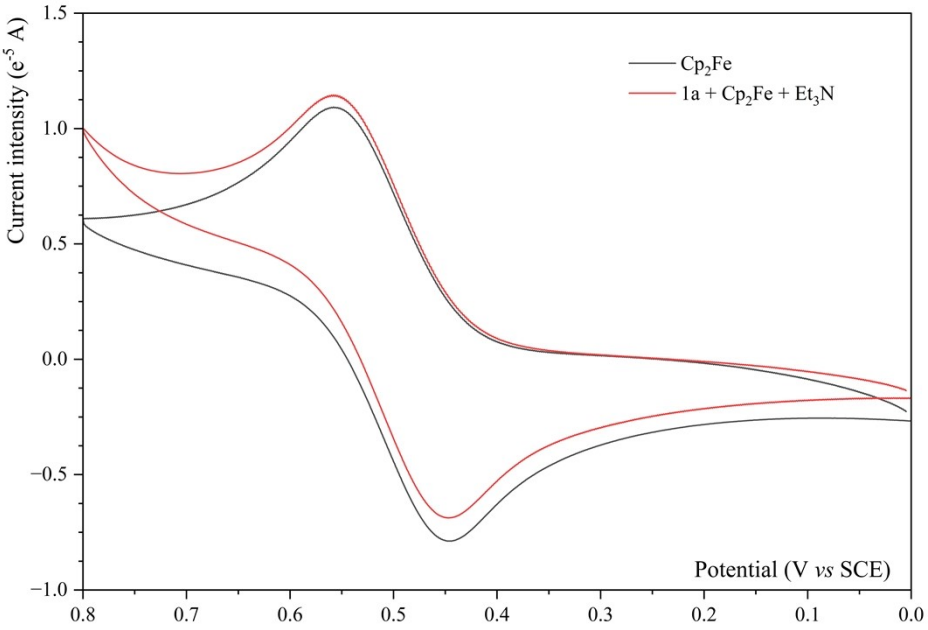
Background: 0.1 M *n*-Bu_4NPF_6 in THF+MeOH under Ar; Cp_2Fe (1.0 mM); Et_3N (4.0 mM); **1a** (5.0 mM).

*Figure S3: Cyclic voltammetry of Cp_2Fe, Cp_2Fe+Et_3N, **1a**+ Cp_2Fe+Et_3N in the [0V, 0.8V] range*



Background: 0.1 M *n*-Bu_4NPF_6 in THF+MeOH under Ar; Cp_2Fe (1.0 mM); Et_3N (4.0 mM); $In(OTf)_3$ (0.25 mM); **1a** (5.0 mM).

Figure S4: Cyclic voltammetry of ***1a****, **1a**+In(OTf)$_3$, **1a**+In(OTf)$_3$+Cp$_2$Fe, **1a**+In(OTf)$_3$+Cp$_2$Fe,+Et$_3$N, in the [0V, 0.8V] range*

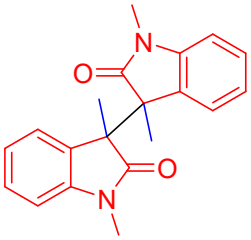


Note: background: 0.1 M *n*-Bu_4NPF_6 in THF+MeOH under Ar; Cp_2Fe (1.0 mM); Et_3N (4.0 mM); **1a** (5.0 mM).

*Figure S5: Cyclic voltammetry of Cp$_2$Fe, **1a**+Cp$_2$Fe+Et$_3$N, in the [0V, 0.8V] range*

5. Spectral Data

1,1',3,3'-tetramethyl-[3,3'-biindoline]-2,2'-dione (2a)

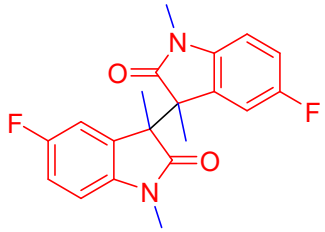


Compound **2a** was prepared from 2-methyl-3-(methyl(phenyl)amino)-3-oxopropanoic acid **1a** (82.8 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2a** (44.2 mg, 0.14 mmol, *dr* = 50 : 50, 70%) as colorless solid.

(±)-D,L-2a: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.05 (d, *J* = 7.5 Hz, 2H), 7.01 (t, *J* = 7.7 Hz, 2H), 6.81 (t, *J* = 7.4 Hz, 2H), 6.44 (d, *J* = 7.7 Hz, 2H), 3.08 (s, 6H), 1.75 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.3, 141.7, 132.7, 131.2, 126.3, 114.8, 109.1, 51.5, 30.0, 15.6.

Meso-2a: **^{1}H NMR (500 MHz, $CDCl_3$, 300K):** δ (ppm) 7.23 (t, *J* = 7.5 Hz. 2H), 6.85 (t, *J* = 7.5 Hz, 2H), 6.71 (d, *J* = **7.7** Hz, 2H), 6.60 (d. *J* = 5.96 Hz, 2H), 2.96 (s, 6H), 1.67 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.8, 143.9, 131.2, 128.6, 123.7, 121.7, 108.0, 51.7, 26.0, 17.5.
HRMS-ESI: Calcd for $C_{20}H_{21}N_2O_2^+$ $[M+H]^+$ 321.1598, found 321.1596.

5,5'-difluoro-1,1',3,3'-tetramethyl-[3,3'-biindoline]-2,2'-dione (2b)



Compound **2b** was prepared from 3-((4-fluorophenyl)(methyl)amino)-2-methyl-3-oxopropanoic acid **1b** (90.0 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column

chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2b** (36.3 mg, 0.102 mmol, *dr*= 50 : 50, 51%) as colorless solid.

(±)-D,L-2b: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.82 (dd, *J* = 8.4 Hz, *J* = 2.6 Hz, 2H), 6.74 (td, *J* = 8.7 Hz, *J* = 2.6 Hz, 2H), 6.42 (q, *J* = 4.2 Hz. 2H), 3.11 (s, 6H), 1.72 (s, 6H).

^{19}F(470 MHz, $CDCl_3$, 300K): δ (ppm) -120.95

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.7, 159.0 (d, *J* = 240.7 Hz), 138.6 (d, *J* = 1.9 Hz), 132.6 (d, *J* = 7.8 Hz), 114.5 (d, *J* = 23.6 Hz), 111.3 (d, *J* = 25.5 Hz), 108.1 (d, *J* = 8.0 Hz), 51.4, 26.1, 16.3.

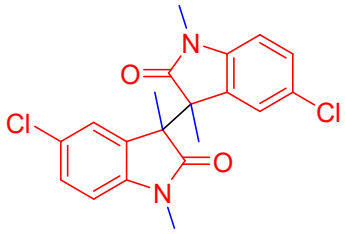
Meso-2b: R_f: 0.15 (30% EtOAc/petroleum ether); **^{1}H NMR (500 MHz, $CDCl_3$, 300K):** δ (ppm) 6.98 (td, *J* = 8.7 Hz, *J* = 2.6 Hz, 2H), 6.67 (q, *J* = 4.2 Hz, 2H), 6.4 (d, *J* = 6.9 Hz, 2H), 2.98 (s, 6H), 1.65, (s, 6H)

^{19}F(470 MHz, $CDCl_3$, 300K): δ (ppm) -120.85

^{13}C NMR (125 MHz,$CDCl_3$, 300K): δ (ppm) 177.2, 158.7 (d, *J* = 239.6 Hz), 139.8 (d, *J* = 1.8 Hz), 132.3 (d, *J* = 8.1 Hz), 115.0 (d, *J* = 23.5 Hz), 111.9 (d, *J* = 25.0 Hz), 108.6 (d, *J* = 8.1 Hz), 52.0, 26.2, 17.5.

HRMS-ESI: Calcd for $C_{20}H_{19}F_2N_2O_2^+$ $[M+H]^+$ 357.1409, found 357.1409.

5,5'-dichloro-1,1',3,3'-tetramethyl-[3,3'-biindoline]-2,2'-dione (2c)



Compound **2c** was prepared from 3-((4-chlorophenyl)(methyl)amino)-2-methyl-3-oxopropanoic acid **1c** (96.4 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2c** (52.0 mg, 0.134 mmol, *dr*= 45 : 55, 67%) as colorless solid.

(±)-D,L-2c: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.03-7.00 (m, 4H), 6.41 (d, *J* = 8.0 Hz, 2H), 3.10 (s, 6H), 1.72 (s, 6H).

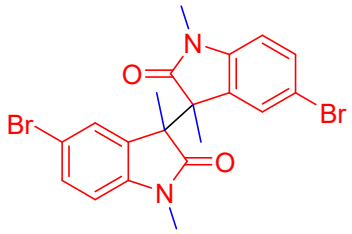
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.4, 141.2, 132.4, 128.3. 127.6, 123.6, 108.6, 51.4, 26.0, 15.7.

Meso-2c: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.26 (dd, *J* = 8.3 Hz, *J* = 2.0 Hz, 2H), 6.67 (d, *J* = 8.3 Hz, 2H), 6.60 (s, 2H), 2.97 (s, 6H), 1.64 (s. 6H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.0, 142.4, 132.3, 128.8, 127.2, 124.2, 109.0, 51.9, 26.2, 17.2.

HRMS-ESI: Calcd for $C_{20}H_{19}Cl_2N_2O_2^+$ $[M+H]^+$ 389.0818 found 389.0820.

5,5'-dibromo-1,1',3,3'-tetramethyl-[3,3'-biindoline]-2,2'-dione (2d)

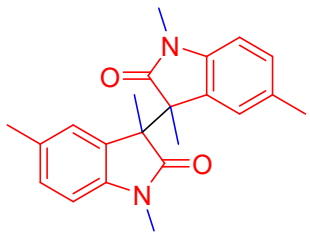


Compound **2d** was prepared from 3-((4-bromophenyl)(methyl)amino)-2-methyl-3-oxopropanoic acid **1d** (114.0 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 µL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2d** (65.0 mg, 0.144 mmol, *dr*= 45 : 55, 72%) as colorless solid.

(±)-D,L-2d: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.20-7.12(m, 4H), 6.37 (d, *J* = 8.7 Hz, 2H), 3.11 (s, 6H), 1.71 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.3, 141.7, 132.7, 131.2, 126.3, 114.8, 109.1, 51.5, 26.0, 15.6.

Meso-2d: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.40 (d, *J* = 8.3 Hz, *J* = 1.9 Hz, 2H), 6.7 (s, 2H), 6.63 (d, *J* = 8.2 Hz, 2H), 2.95 (s, 6H), 1.63 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 176.8, 142.9, 132., 131.6, 126.9, 114.4, 109.5, 51.9, 26.2, 17.1.
HRMS-ESI: Calcd for $C_{20}H_{19}Br_2N_2O_2^+$ $[M+H]^+$ 476.9808 found 476.9808.

1,1',3,3',5,5'-hexamethyl-[3,3'-biindoline]-2,2'-dione (2e)

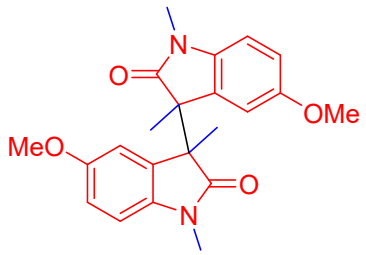


Compound **2e** was prepared from 2-methyl-3-(methyl(p-tolyl)amino)-3-oxopropanoic acid **1e** (88.4 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 µL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2e** (32.0 mg, 0.092 mmol, *dr*= 55 : 45, 46%) as colorless solid.

(±)-D,L-2e: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.98 (s, 2H), 6.81 (d, *J* = 7.8 Hz, 2H), 6.3 (d, *J* = 7.8 Hz, 2H), 3.10 (s, 6H), 2.20 (s, 6H), 1.72 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 178.2, 140.2, 131.4, 131.2, 128.3, 123.9, 107.1, 51.2, 25.8, 21.1, 16.1.

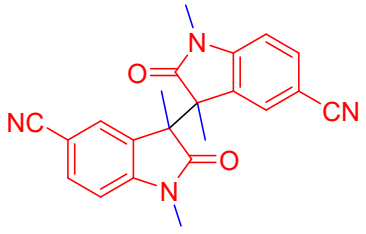
Meso-2e: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.03 (d, *J* = 7.7 Hz, 2H), 6.58 (d, *J* = 7.7 Hz, 2H), 6.38 (s, 2H), 2.92 (s, 6H), 2.20 (s, 6H), 1.64 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.7, 141.5, 131.2, 131.0, 128.7, 124.7, 107.5, 51.9, 26.0, 21.2, 17.3.
HRMS-ESI: Calcd for $C_{22}H_{25}N_2O_2^+$ $[M+H]^+$ 349.1911 found 349.1910.

5,5'-dimethoxy-1,1',3,3'-tetramethyl-[3,3'-biindoline]-2,2'-dione (2f)



Compound **2f** was prepared from 3-((4-methoxyphenyl)(methyl)amino)-2-methyl-3-oxopropanoic acid **1f** (47.4 mg, 0.2 mmol), Cp_2Fe (7.5 mg, 0.04 mmol, 0.2 equiv.), triethylamine (14 µL, 0.1 mmol), indium triflate (11.2 mg, 0.04 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (155 mg, 0.4 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **(±)-D,L-2f** (13.7 mg, 0.036 mmol, 36%) as colorless solid.
(±)-D,L-2f: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.73 (d, *J* =2.6 Hz, 2H), 6.57 (dd, *J* = 8.4 Hz, *J* = 2.6 Hz, 2H), 6.39 (d, *J* =8.5 Hz, 2H), 3.69 (s, 6H), 3.10 (s, 6H), 1.74 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.9, 155.8, 136.2, 132.6, 113.1, 110.2, 107.9, 55.9, 51.5, 26.0, 16.6.
HRMS-ESI: Calcd for $C_{22}H_{25}N_2O_4^+$ $[M+H]^+$ 381.1809, found 381.1811.

1,1',3,3'-tetramethyl-2,2'-dioxo-[3,3'-biindoline]-5,5'-dicarbonitrile (2g)



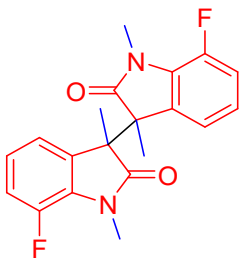
Compound **2g** was prepared from 3-((4-bromophenyl)(methyl)amino)-2-methyl-3-oxopropanoic acid **1g** (92.8 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 µL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General**

Procedure A). The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2g** (42.9 mg, 0.116 mmol, *dr*= 50 : 50, 58%) as colorless solid.

(±)-D,L-2g: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.40 (dd, *J* = 8.0 Hz, *J* = 1.3 Hz, 2H), 7.27 (d, *J* = 1.3 Hz, 2H), 6.60 (d, *J* = 8.0 Hz, 2H), 3.18 (s, 6H), 1.76 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.4, 146.5, 133.9, 131.6, 126.4, 118.8, 108.5, 105.5, 50.9, 26.3, 16.0.

Meso-2g: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.64 (dd, *J* = 7.7 Hz, *J* = 1.5 Hz, 2H), 6.89 (s, 2H), 6.85 (d, *J* = 8.3 Hz, 2H), 3.03 (s, 6H), 1.67 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 176.9, 147.6, 134.5, 131.4, 127.0, 118.9, 108.9, 105.4, 51.5, 26.4, 17.4.
HRMS-ESI: Calcd for $C_{22}H_{19}N_4O_2^+$ $[M+H]^+$ 371.1503 found 371.1503.

7,7'-difluoro-1,1',3,3'-tetramethyl-[3,3'-biindoline]-2,2'-dione (2h)



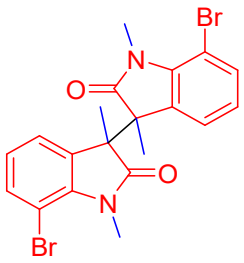
Compound **2h** was prepared from 3-((2-fluorophenyl)(methyl)amino)-2-methyl-3-oxopropanoic acid **1h** (90.0 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2h** (36.3 mg, 0.102 mmol, *dr*= 50 : 50, 51%) as colorless solid.

(±)-D,L-2h: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.87-6.84 (m, 2H), 6.82-6.77 (m, 4H), 3.31 (d, *J* = 2.8 Hz, 6H), 1.73 (s, 6H).
^{19}F(470 MHz, $CDCl_3$, 300K): δ (ppm) -136.8.
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.7, 147.3 (d, *J* = 243.7 Hz), 133.9 (d, *J* = 2.8 Hz), 129.4 (d, *J* = 8.1 Hz), 122.6 (d, *J* = 6.2 Hz), 118.9 (d, *J* = 6.7 Hz), 116.4 (d, *J* = 18.9 Hz), 51.5, 28.3 (d, *J* = 6.0 Hz), 16.5.

Meso-2h: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.99 (dd, *J* = 8.4 Hz, *J* = 0.8 Hz, 1H), 6.98 (m, *J* = 8.4 Hz. *J* = 0.8 Hz, 1H), 6.83 (m, 2H), 6.41 (d, *J* = 6.5 Hz, 2H), 3.20 (d, *J* = 2.7 Hz, 6H), 1.65 (s, 6H).
^{19}F(470 MHz, $CDCl_3$, 300K): δ (ppm) -136.1.

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.2, 147.7 (d, *J* = 244.4 Hz), 133.8 (d, *J* = 2.7 Hz), 130.6 (d, *J* = 7.7 Hz), 122.3 (d, *J* = 6.4 Hz), 119.6 (d, *J* = 3.1 Hz), 116.7 (d, *J* = 19.4 Hz), 52.1, 28.6 (d, *J* = 5.6 Hz), 17.6.
HRMS-ESI: Calcd for $C_{20}H_{19}F_2N_2O_2^+$ $[M+H]^+$ 357.1409, found 357.1409.

7,7'-dibromo-1,1',3,3'-tetramethyl-[3,3'-biindoline]-2,2'-dione (2i)

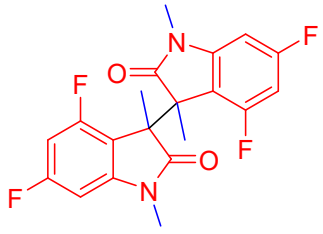


Compound **2i** was prepared from 3-((2-bromophenyl)(methyl)amino)-2-methyl-3-oxopropanoic acid **1i** (114.0 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (20% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2i** (35.2 mg, 0.074 mmol, *dr*= 40 : 60, 37%) as colorless solid.

(±)-D,L-2i: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.19 (dd, *J* = 8.3 Hz, *J* = 1.0 Hz, 2H), 6.96 (dd, *J* = 7.5 Hz, *J* = 1.0 Hz, 2H). 6.72 (t, *J* = 7.8 Hz, 2H), 3.49 (s, 6H), 1.72 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 178.4, 140.0, 134.2, 133.9, 123.2, 122.1, 101.9, 51.1, 29.9, 16.5.

Meso-2i: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.74 (d, *J* = 8.0 Hz, 2H), 6.75 (t, *J* = 7.8 Hz, 2H), 6.53 (t, *J* = 6.2 Hz, 2H), 3.36 (s, 6H), 1.62 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.9, 141.3, 134.6, 133.7, 122.9, 122.7, 102.5, 51.6, 29.8, 17.5.
HRMS-ESI: Calcd for $C_{20}H_{19}Br_2N_2O_2^+$ $[M+H]^+$ 476.9808 found 476.9808.

4,4',6,6'-tetrafluoro-1,1',3,3'-tetramethyl-[3,3'-biindoline]-2,2'-dione (2j)



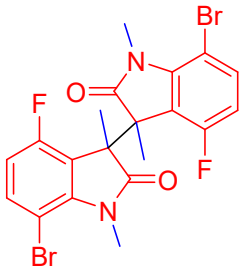
Compound **2j** was prepared from 3-((3,5-difluorophenyl)(methyl)amino)-2-methyl-3-oxopropanoic acid **1j** (97.2 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General**

Procedure A). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2j** (51.7 mg, 0.132 mmol, *dr*= 40 : 60, 66%) as colorless solid.

(±)-D,L-2j: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.27 (td, *J* = 9.9 Hz, *J* = 1.9 Hz, 2H), 6.16 (dd, *J* = 8.4 Hz, *J* = 1.9 Hz, 2H), 3.10 (s, 6H), 1.77 (s, 6H).
^{19}F(470 MHz, $CDCl_3$, 300K): δ (ppm) -107.7 (d, *J* = 7.7 Hz), -113.5 (d, *J* = 8.7 Hz).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 176.9, 163.3 (dd, *J* = 248.9 Hz, *J* = 13.4 Hz), 158.6 (dd, *J* = 250.9, Hz, *J* = 13.8 Hz), 146.3 (t, *J* = 13.6 Hz), 112.0 (dd, *J* = 19.7 Hz, *J* = 3.7 Hz), 97.5 (t, *J* = 26.3Hz), 92.9 (dd, *J* = 27.2 Hz, *J* = 3.2 Hz), 52.1, 26.7, 14.8.

Meso-2j: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.37- 6.28 (m, 4H), 2.98 (s, 6H), 1.74 (d, *J* = 1.0 Hz, 6H).
^{19}F(470 MHz, $CDCl_3$, 300K): δ (ppm) -107.8 (d, *J* = 8.7 Hz), -110.9 (d, *J* = 8.7 Hz).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.1, 163.7 (dd, *J* = 249.6 Hz, *J* = 13.0 Hz), 159.5 (dd, *J* = 250.5 Hz, *J* = 14.2Hz), 146.5 (t, *J* = 13.0 Hz), 112.3 (dd, *J* = 19.7 Hz, *J* = 3.7 Hz), 97.8 (t, *J* = 26.7 Hz), 93.2 (dd, *J* = 27.0 Hz, *J* = 3.3 Hz), 53.7 (d, *J* = 2.3 Hz), 26.6, 16.2 (d, *J* = 5.0 Hz).
HRMS-ESI: Calcd for $C_{20}H_{17}F_4N_2O_2^+$ $[M+H]^+$ 393.1221 found 393.1221.

7,7'-dibromo-4,4'-difluoro-1,1',3,3'-tetramethyl-[3,3'-biindoline]-2,2'-dione (2k)

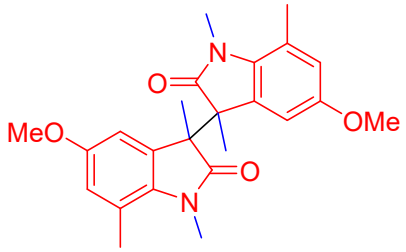


Compound **2k** was prepared from 3-((2-bromo-5-fluorophenyl)(methyl)amino)-2-methyl-3-oxopropanoic acid **1k** (120.8 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 µL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (20% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2k** (46.0 mg, 0.090 mmol, *dr*= 50 : 50, 45%) as the mixture of inseparable diastereoisomers.

^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.32 (dd, *J* = 9.1 Hz, *J* = 5.0 Hz, 1H), 7.16 (dd, *J* = 9.0 Hz, *J* = 5.0 Hz, 1H), 6.52 (t, *J* = 9.0 Hz, 1H), 6.47 (t, *J* = 9.0 Hz, 1H), 3.49 (s, 3H), 3.36 (s, 3H), 1.76 (s, 3H), 1.74 (s, 3H).
^{19}F(470 MHz, $CDCl_3$, 300K): δ (ppm) -115.8, -118.0
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.1, 176.8, 158.8 (d, *J* = 248.2 Hz), 158.2 (d, *J* = 248.2 Hz), 142.6 (d, *J* = 8.2 Hz), 142.4 (d, *J* = 8.2 Hz), 135.8 (d, *J* = 9.1 Hz), 135.5 (d, *J* = 9.3 Hz), 119.6 (d, *J* = 20.0 Hz), 119.3 (d, *J* = 20.4 Hz), 111.7 (d, *J* = 23.9 Hz), 110.9 (d, *J* = 23.9 Hz), 96.9

(d, J = 3.7 Hz), 96.4 (d, J = 3.7 Hz), 53.8 (d, J = 2.7 Hz), 52.3 (d, J = 2.7 Hz), 29.9, 29.8, 15.8 (d, J = 5.0 Hz), 14.6.
HRMS-ESI: Calcd for $C_{20}H_{17}Br_2F_2N_2O_2^+$ $[M+H]^+$ 512.9619 found 512.9620.

5,5'-dimethoxy-1,1',3,3',7,7'-hexamethyl-[3,3'-biindoline]-2,2'-dione (2l)

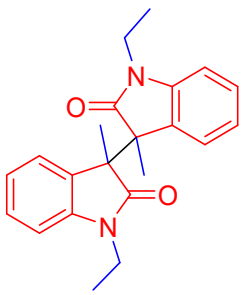


Compound **2l** was prepared from 3-((4-methoxy-2-methylphenyl)(methyl)amino)-2-methyl-3-oxopropanoic acid **1l** (100.4 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 µL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and n-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (60% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2l** (38.4 mg, 0.094 mmol, dr= 50 : 50, 47%) as colorless solid.

(±)-D,L-2l: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.55 (d, J = 2.4 Hz, 2H), 6.33 (d, J = 2.4 Hz, 2H), 3.68 (s, 6H), 3.37 (s, 6H), 2.30 (s, 6H), 1.70 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 178.7, 155.1, 134.0, 133.3, 120.0, 117.2, 107.3, 55.8, 51.0, 29.3, 19.1, 17.0.

Meso-2l: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.52 (s, 2H), 6.10 (s, 2H), 3.61 (s, 6H), 3.21 (s, 6H), 2.45 (s, 6H), 1.61 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 178.3, 154.6, 135.4, 132.9, 120.3, 117.3, 108.3, 55.7, 51.6, 29.5, 19.2, 17.7.
HRMS-ESI: Calcd for $C_{24}H_{29}N_2O_4^+$ $[M+H]^+$ 409.2122 found 409.2122.

1,1'-diethyl-3,3'-dimethyl-[3,3'-biindoline]-2,2'-dione (2m)



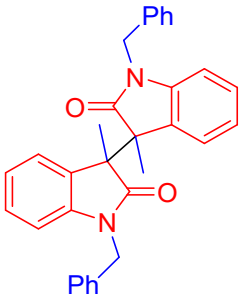
Compound **2m** was prepared from 3-(ethyl(phenyl)amino)-2-methyl-3-oxopropanoic acid **1m** (88.4 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 µL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and n-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The

flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2m** (48.7 mg, 0.140 mmol, *dr*= 45 : 55, 70%) as colorless solid.

(±)-D,L-2m: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.14 (d, *J* = 7.4 Hz, 2H), 7.02 (t, *J* = 7.8 Hz, 2H), 6.49 (t, *J* = 7.5 Hz, 2H), 6.51 (d, *J* = 7.8 Hz, 2H), 3.81-3.71 (m, 2H), 3.66-3.56 (m, 2H), 1.74 (s, 6H), 1.20 (t, *J* = 7.3 Hz, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 178.1, 141.9, 131.6, 128.1, 123.8, 121.8, 107.7, 50.7, 34.6, 17.1, 12.6.

Meso-2m: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.22 (t, *J* = 7.6 Hz, 2H), 6.84 (t, *J* = 7.2 Hz, 2H), 6.71 (d, *J* = 7.8 Hz, 2H), 6.60 (s, 2H), 3.77-3.67 (m, 2H), 3.44-3.34 (m, 2H). 1.66 (s, 6H), 0.89 (t, *J* = 6.7 Hz. 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.3, 143.1, 131.5, 128.5, 124.1, 121.5, 108.0, 51.4, 34.3, 17.5, 12.0.
HRMS-ESI: Calcd for $C_{22}H_{25}N_2O_2^+$ $[M+H]^+$ 349.1911 found 349.1911.

1,1'-dibenzyl-3,3'-dimethyl-[3,3'-biindoline]-2,2'-dione (2n)

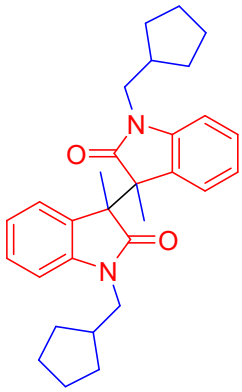


Compound **2n** was prepared from 3-(benzyl(phenyl)amino)-2-methyl-3-oxopropanoic acid **1n** (113.2 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (20% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2n** (45.8 mg, 0.110 mmol, *dr*= 55 : 45, 40%) as colorless solid.

(±)-D,L-2n: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.32-7.25 (m, 6H), 7.24-7.20 (m, 4H), 7.06 (dd, *J* = 7.4 Hz, *J* = 0.8 Hz, 2H), 6.94 (td, *J* = 7.7 Hz, *J* = 1.1 Hz, 2H), 6.66 (td, *J* = 7.7 Hz, *J* = 0.8 Hz, 2H), 6.46 (d, *J* = 7.7 Hz, 2H), 5.01 (d, *J* = 15.5 Hz, 2H), 4.68 (d, *J* = 15.5 Hz, 2H), 1.84 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 178.6, 142.0, 135.8, 131.5, 128.8, 128.0, 127.72, 127.71, 123.7, 122.3, 108.7, 50.9, 43.9, 17.9.

Meso-2n: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.19-7.11 (m, 8H), 6.95 (s, 4H), 6.81 (t, *J* = 7.2 Hz, 2H), 6.69 (s, 2H), 6.58 (d, *J* = 7.7 Hz, 2H), 4.93 (d, *J* = 15.8 Hz, 2H), 4.65 (d, *J* = 15.8 Hz, 2H), 1.80 (s, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 178.1, 143.0, 135.9, 131.5, 128.7, 128.5, 127.2, 127.1, 124.0, 122.1, 109.3, 51.4, 43.9, 18.7.
HRMS-ESI: Calcd for $C_{32}H_{29}N_2O_2^+$ $[M+H]^+$ 473.2224 found 473.2222.

1,1'-bis(cyclopentylmethyl)-3,3'-dimethyl-[3,3'-biindoline]-2,2'-dione (2o)

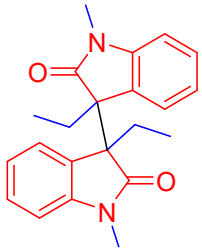


Compound **2o** was prepared from 3-((cyclopentylmethyl)(phenyl)amino)-2-methyl-3-oxopropanoic acid **1o** (110.0 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 µL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (20% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2o** (47.4 mg, 0.104 mmol, *dr*= 50 : 50, 52%) as colorless solid.

(±)-D,L-2o: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.01-6.95 (m, 4H), 6.79 (td, *J* = 7.5 Hz, *J* = 0.7 Hz, 2H), 6.36 (d, *J* = 7.6 Hz, 2H), 3.06 (dd, *J* = 13.8 Hz, *J* = 5.4Hz, 2H), 3.04 (s, 6H), 2.28 (dd, *J* = 13.7 Hz, *J* = 7.9 Hz, 2H), 1.50-1.36 (m, 4H), 1.28-1.08 (m, 10H), 1.06 -0.9 (m, 4H)
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 178.2, 143.4, 129.0, 128.0, 124.0, 121.3, 107.1, 56.9, 37.4, 34.1, 34.0, 32.5, 25.8, 25.2, 24.8.

Meso-2o: **^{1}H NMR (500 MHz, $CDCl_3$, 300K):** δ (ppm) 7.21 (t, *J* = 7.6 Hz, 2H), 6.8 (s, 2H), 6.65 (d, *J* = 7.9 Hz, 2H), 6.50 (s, 2H), 2.91 (s, 6H), 2.79 (dd, *J* = 13.4 Hz, *J* = 6.0 Hz, 2H), 2.20 (dd, *J* = 13.4 Hz, *J* = 6.7 Hz, 2H), 1.45-1.32 (m, 6H), 1.28-1.11 (m, 6H), 1.09-1.00 (m, 4H), 0.78-0.65 (m, 2H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.0, 144.8, 129.5, 128.4, 124.7, 121.1, 107.7, 57.5, 37.2, 35.7, 33.9, 33.1, 26.0, 25.0, 24.9.
HRMS-ESI: Calcd for $C_{30}H_{37}N_2O_2^+$ $[M+H]^+$ 457.2850 found 457.2850.

3,3'-diethyl-1,1'-dimethyl-[3,3'-biindoline]-2,2'-dione (2p)

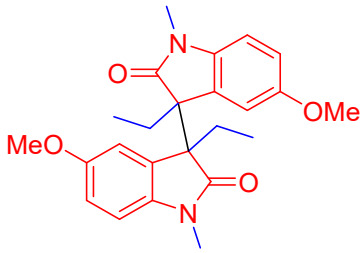


Compound **2p** was prepared from 2-formyl-*N*-methyl-*N*-phenylbutanamide **1p** (82.0 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2p** (48.0 mg, 0.138 mmol, *dr*= 45 : 55, 69%) as colorless solid.

(±)-D,L-2p: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.99 (td, *J* = 5.7 Hz, 4H), 6.81 (t *J* = 7.3 Hz, 2H), 6.40 (d, *J* = 7.9 Hz, 2H), 3.05 (s, 6H), 2.82-2.73 (m, 2H), 2.36-2.28 (m, 2H), 0.38 (t, *J* = 7.3 Hz, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.6, 143.7, 128.8, 128.1, 123.2, 121.7, 107.2, 57.5, 25.7, 21.6, 9.0.

Meso-2p: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.22 (td, *J* = 7.7 Hz, *J* = 1.1 Hz, 2H), 6.85 (t, *J* = 7.4 Hz, 2H), 6.69 (d, *J* = 7.7 Hz, 2H), 6.54 (s, 2H), 2.95 (s, 6H), 2.81-2.70 (m, 2H), 2.14-2.06 (m, 2H), 0.43 (t, *J* = 7.3 Hz, 6H)
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 176.9, 145.0, 129.0, 128.5, 124.0, 121.6, 107.8, 58.1, 25.9, 21.4, 8.8.
HRMS-ESI: Calcd for $C_{22}H_{25}N_2O_4^+$ $[M+H]^+$ 349.1911 found 349.1911.

3,3'-diethyl-5,5'-dimethoxy-1,1'-dimethyl-[3,3'-biindoline]-2,2'-dione (2q)



Compound **2q** was prepared from 2-formyl-*N*-(4-methoxyphenyl)-*N*-methylbutanamide **1q** (94.0 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The

flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2q** (57.9 mg, 0.142 mmol, *dr*= 80 : 20, 71%) as colorlesssolid.

(±) -D,L-2q: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.67 (d, *J* = 2.4 Hz, 2H), 6.56 (dd, *J* = 8.5 Hz, *J* = 2.6 Hz, 2H), 6.35 (d, *J* = 8.5 Hz, 2H), 3.69 (s, 6H), 3.08 (s, 6H), 2.81-2.70 (m, 2H), 2.36-2.20 (m, 2H), 0.39 (t, *J* = 7.4 Hz, 6H).

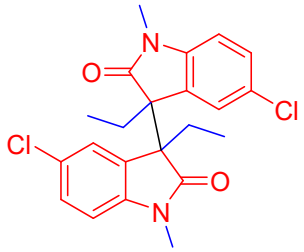
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 177.2, 155.7, 137.2, 130.2, 112.9, 110.3, 107.6, 57.8, 55.9, 25.9, 21.9, 9.0.

Meso-2q: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 6.77 (dd, *J* = 8.5 Hz, *J* = 2.5 Hz, 2H), 6.61 (d, *J* = 8.5 Hz, 2H), 6.22 (s ,2H), 3.65 (s, 6H), 2.94 (s, 6H), 2.78-2.70 (m, 2H), 2.10-2.00 (m, 2H), 0.44 (t, *J* = 7.3 Hz, 6H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 176.5, 155.2, 138.8, 130.3, 113.1, 111.5, 107.9, 58.3, 55.9, 26.0, 23.2, 8.8.

HRMS-ESI: Calcd for $C_{24}H_{29}N_2O_4^+$ $[M+H]^+$ 409.2122 found 409.2120.

5,5'-dichloro-3,3'-diethyl-1,1'-dimethyl-[3,3'-biindoline]-2,2'-dione (2r)



Compound **2r** was prepared from *N*-(4-chlorophenyl)-2-formyl-*N*-methylbutanamide **1r** (95.6 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **2r** (45.8 mg, 0.110 mmol, *dr*= 50 : 50, 55%) as colorlesssolid.

(±)-D,L-2r: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.01 (dd, *J* = 8.3 Hz, *J* = 2.0 Hz, 2H), 6.98 (d, *J* = 2.0 Hz, 2H), 6.38 (d, *J* = 8.2 Hz, 2H) 3.10 (s, 6H), 2.77-2.68 (m, 2H), 2.36-2.27 (m, 2H), 0.43 (t, *J* = 7.4 Hz, 6H).

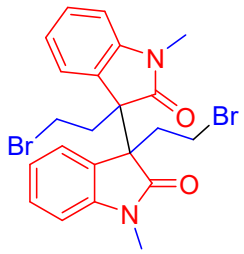
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 176.9, 142.2, 130.3, 128.2, 127.5, 123.7, 108.4, 57.9, 25.9, 21.3, 9.0.

Meso-2r: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.25 (dd, *J* = 8.6 Hz, *J* = 1.6 Hz, 2H), 6.65 (d, *J* = 8.3 Hz, 2H), 6.52 (s, 2H), 2.95 (s, 6H), 2.75-2.66 (m, 2H), 2.06-1.97 (m, 2H), 0.45 (t, *J* = 7.3 Hz, 6H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 176.1, 143.5, 130.2, 128.7, 127.1, 124.4, 108.8, 58.3, 26.1, 23.0, 8.7.

HRMS-ESI: Calcd for $C_{22}H_{23}Cl_2N_2O_4^+$ $[M+H]^+$ 417.1131 found 417.1133.

3,3'-bis(2-bromoethyl)-1,1'-dimethyl-[3,3'-biindoline]-2,2'-dione (2s)



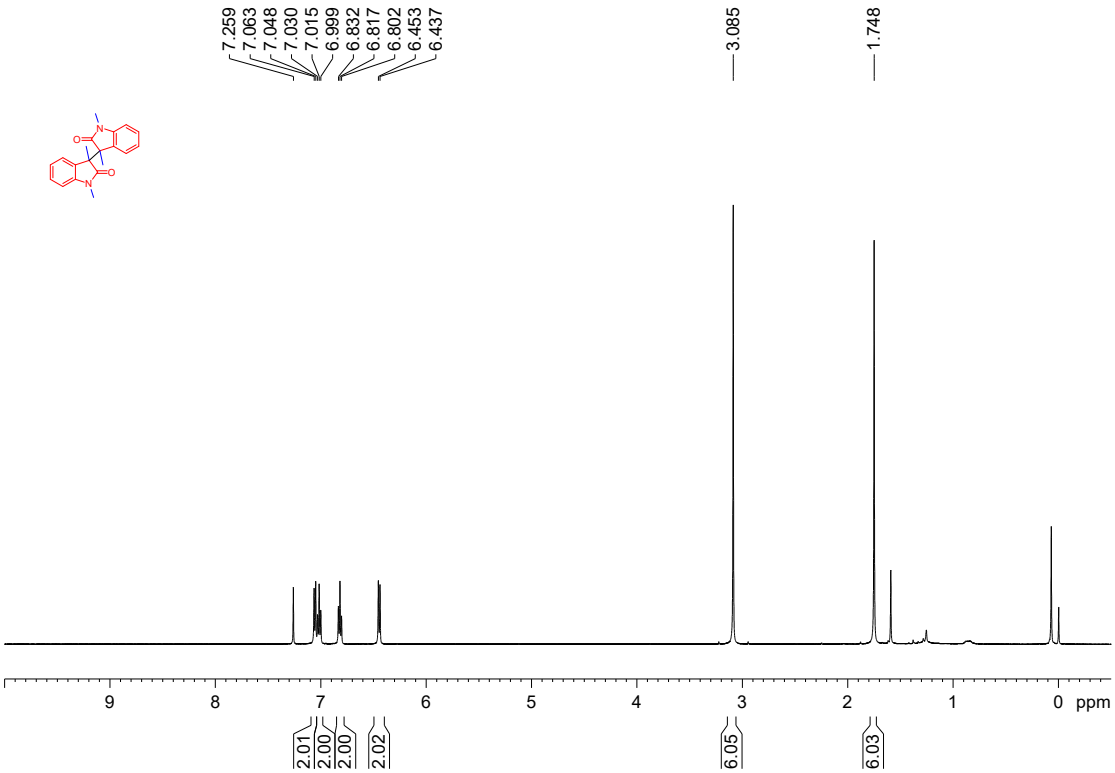
Compound **2s** was prepared from 4-bromo-2-formyl-*N*-methyl-*N*-phenylbutanamide **1s** (113.2 mg, 0.4 mmol), Cp_2Fe (14.9 mg, 0.08 mmol, 0.2 equiv.), triethylamine (28 μL, 0.2 mmol), indium triflate (22.4 mg, 0.08 mmol, 0.2 equiv.) and *n*-Bu_4NPF_6 (309.6 mg, 0.8 mmol, 2.0 equiv.). The flask was equipped with the solution was charged in constant current mode (**General Procedure A**). The consumption of the starting material was checked by TLC (30% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **(±)-D,L-2s** (20.1 mg, 0.080 mmol, 40%) as colorless solid.

(±)-D,L-2s: ^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.05 (td, *J* = 7.6 Hz, *J* = 1.0 Hz, 2H), 7.02 (d, *J* = 7.4 Hz, 2H), 6.86 (t, *J* = 7.7 Hz, 2H), 6.43 (t, *J* = 7.8 Hz, 2H), 3.58-3.42 (m, 2H), 3.06 (s, 6H), 3.05-2.99 (m, 2H), 2.98-2.91 (m, 2H), 2.80-2.69 (m, 2H).

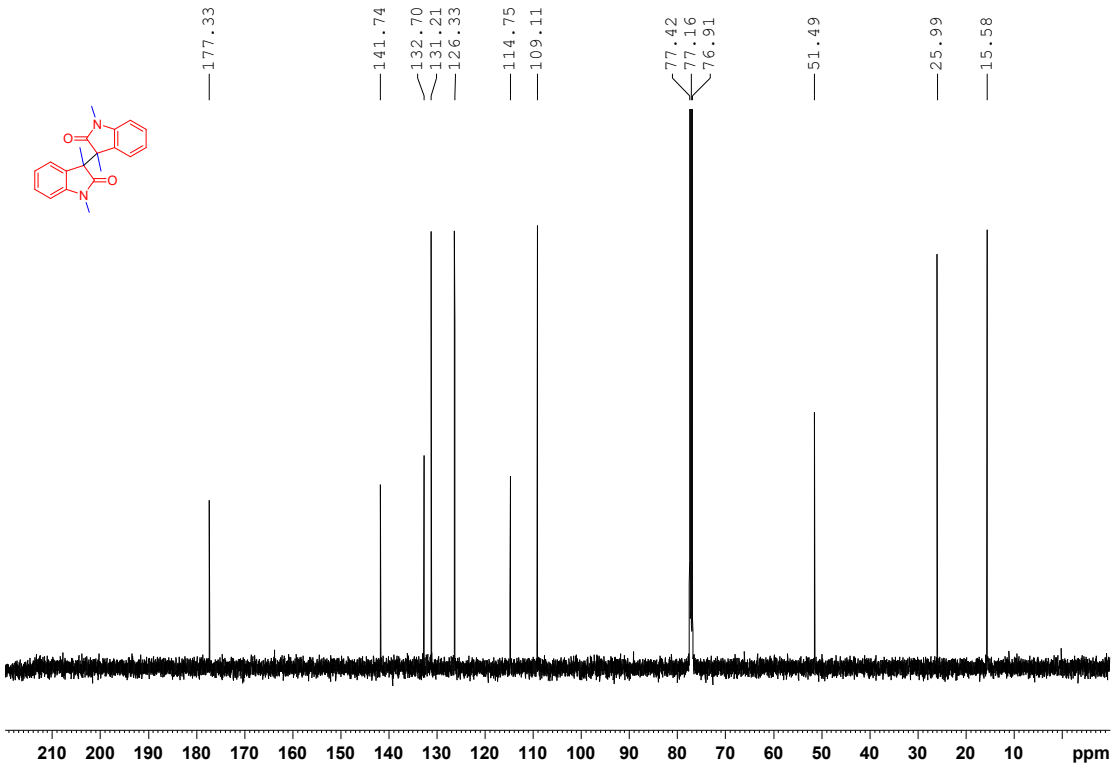
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 176.6, 143.6, 129.1, 126.1, 123.6, 122.0, 107.9, 55.2, 40.8, 31.4, 26.0.

HRMS-ESI: Calcd for $C_{22}H_{23}Br_2N_2O_2^+$ $[M+H]^+$ 505.0121 found 505.0121.

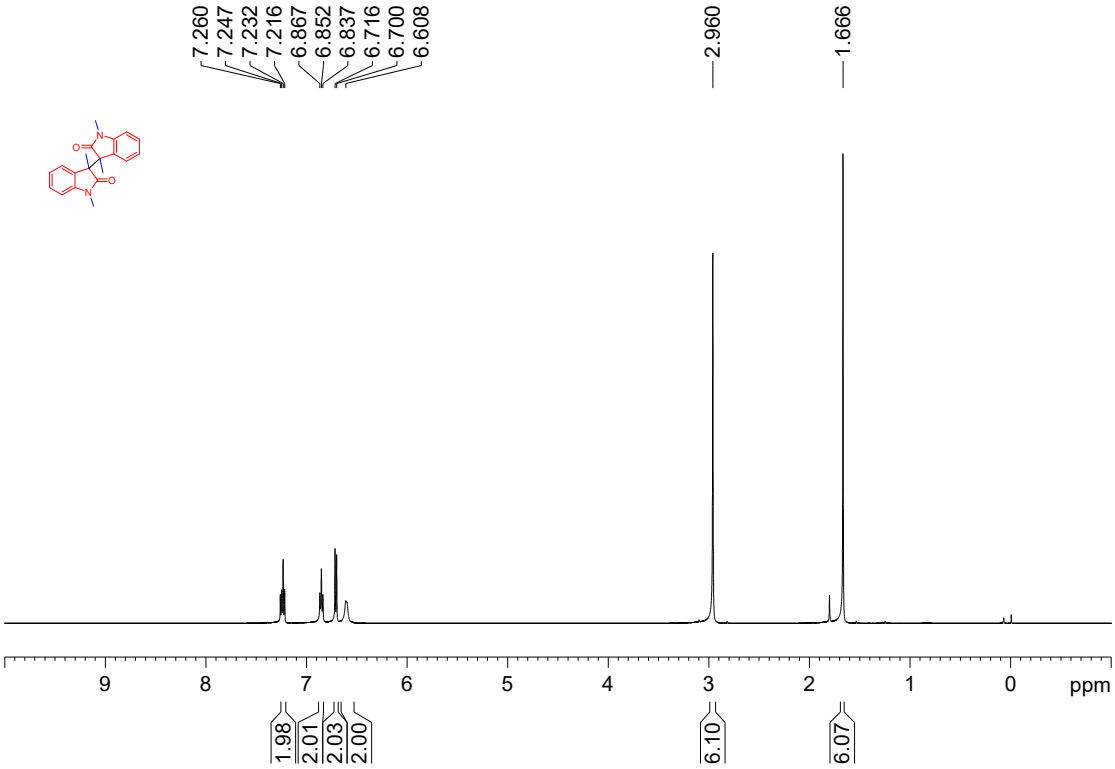
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2a**



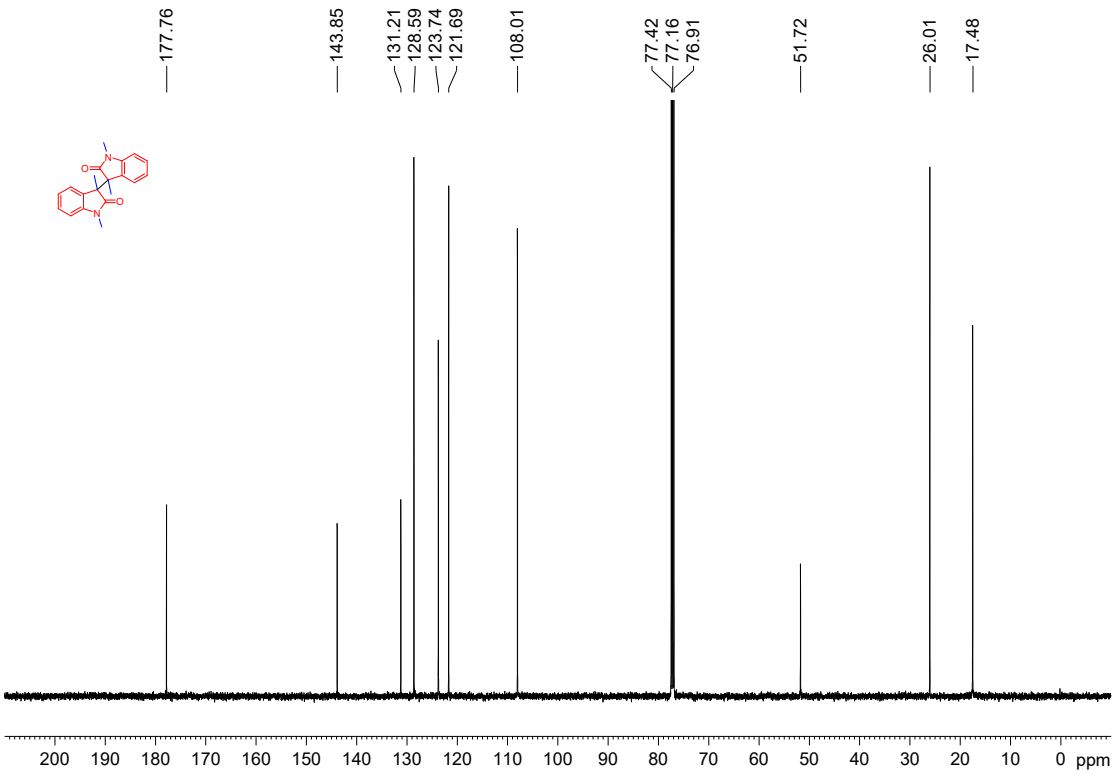
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2a**



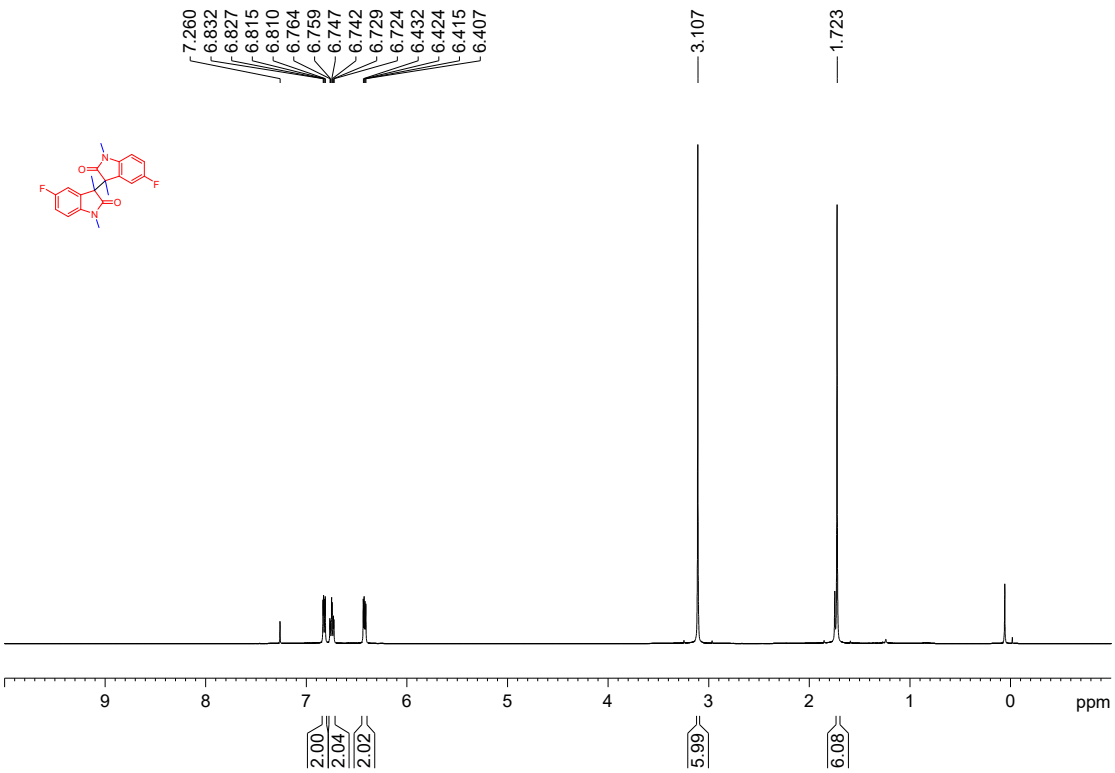
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2a**



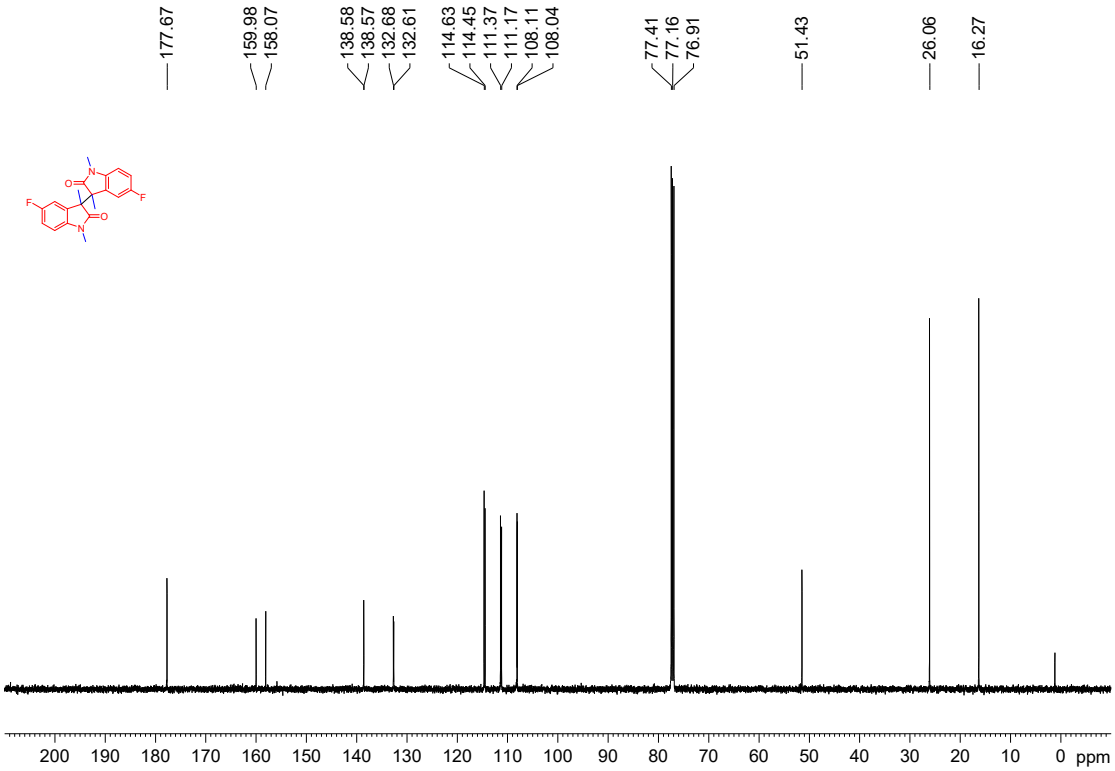
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2a**



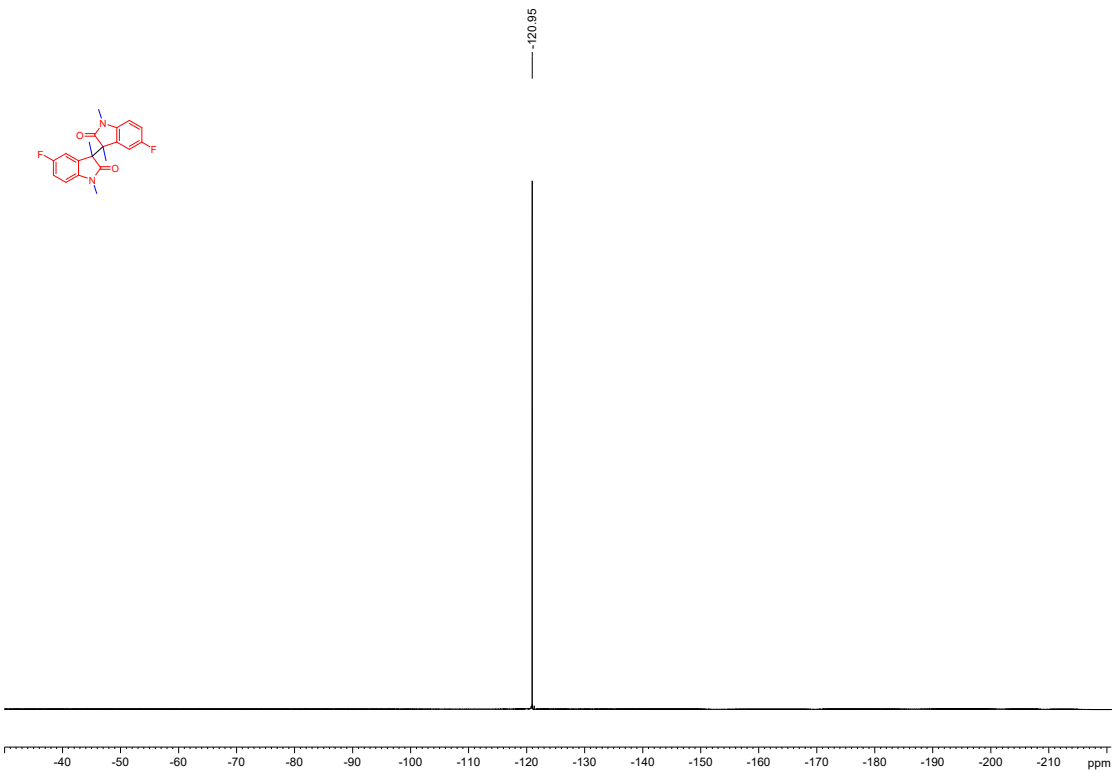
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2b**



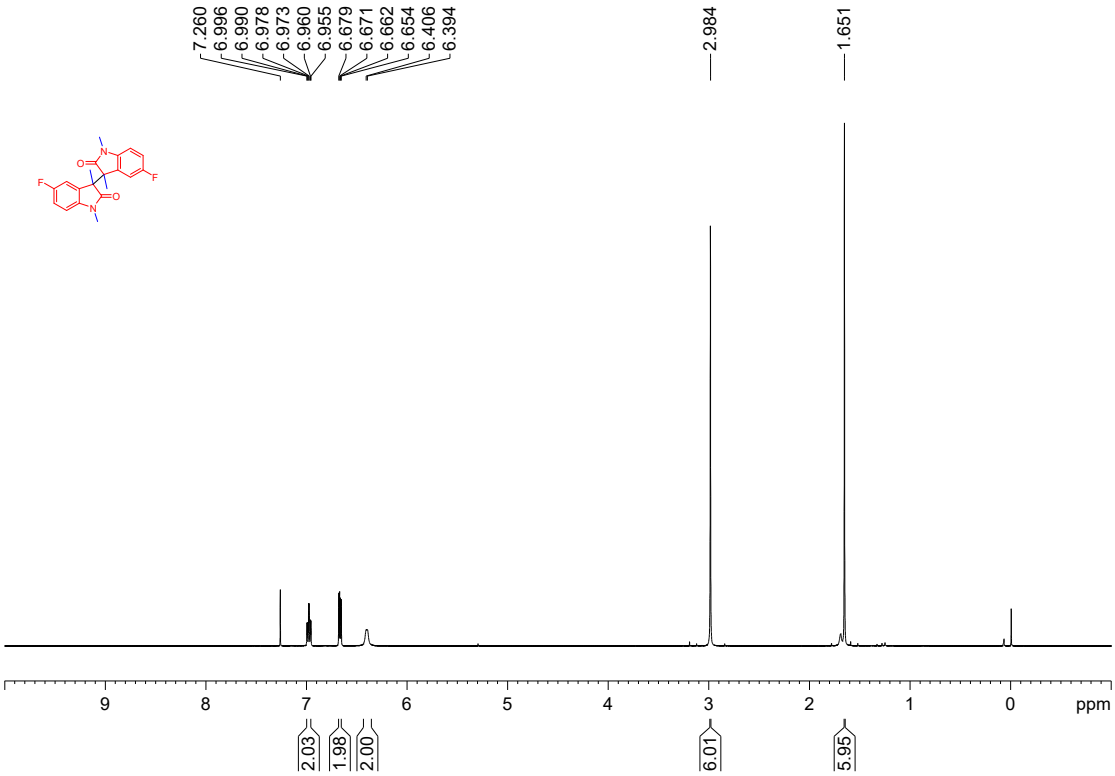
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2b**



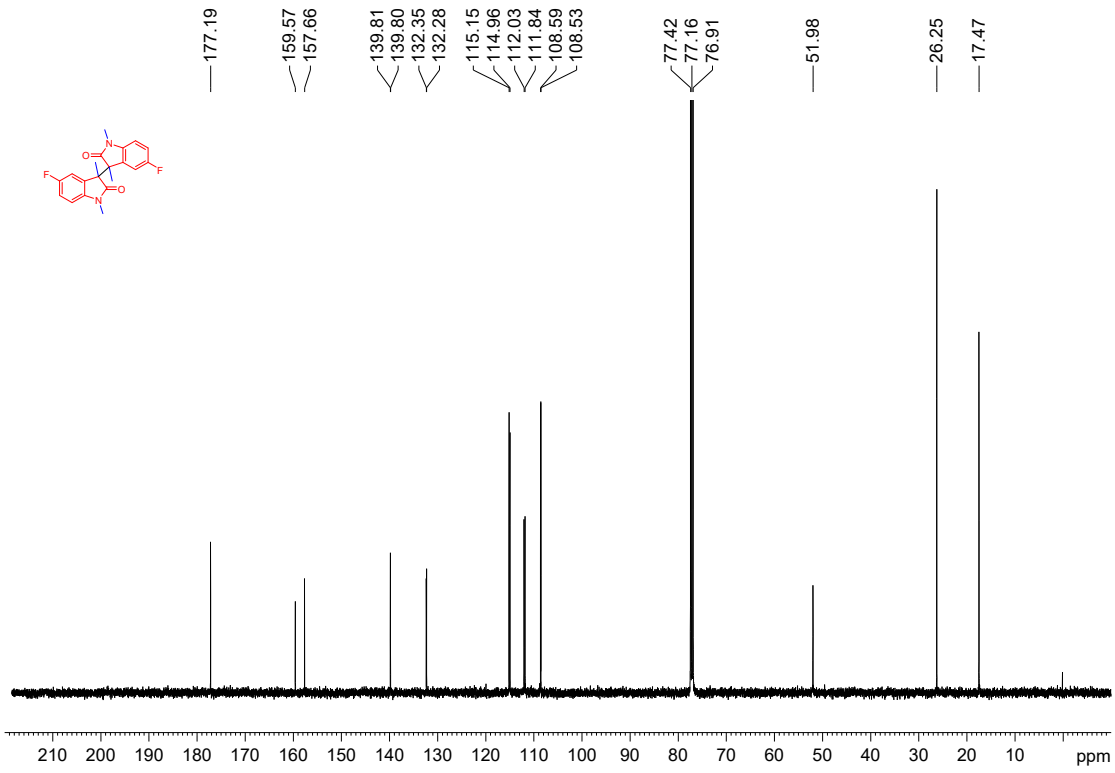
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **(±)-D,L-2b**



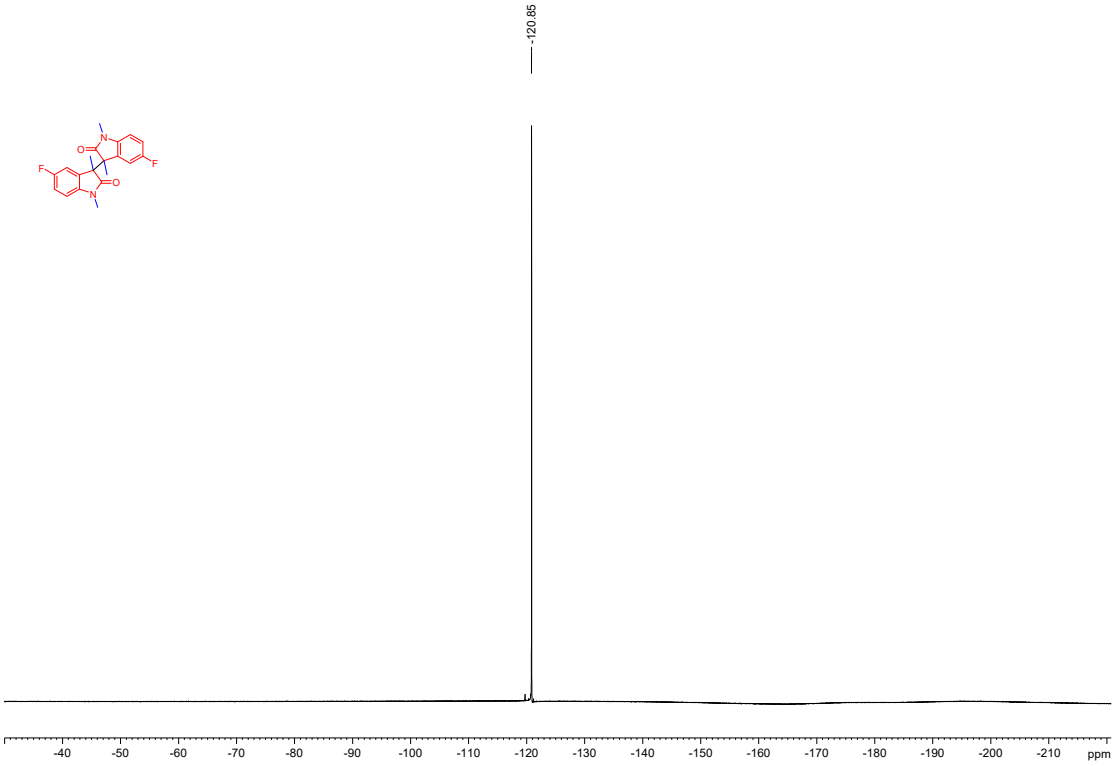
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2b**



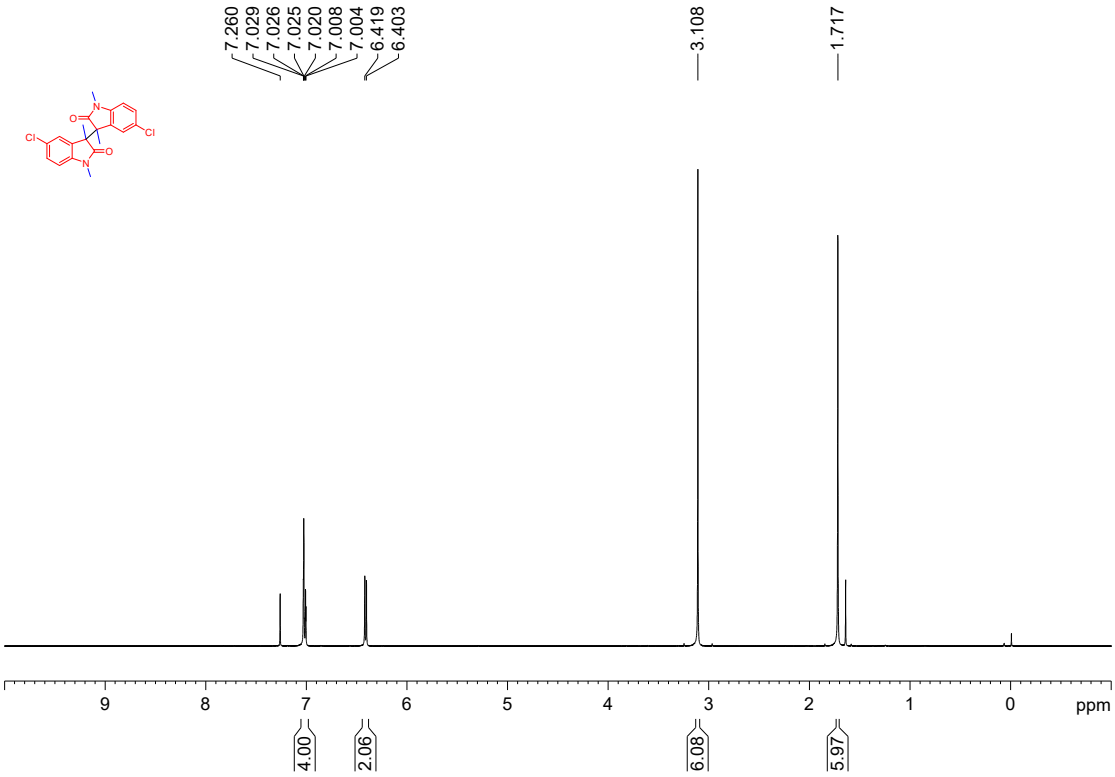
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2b.**



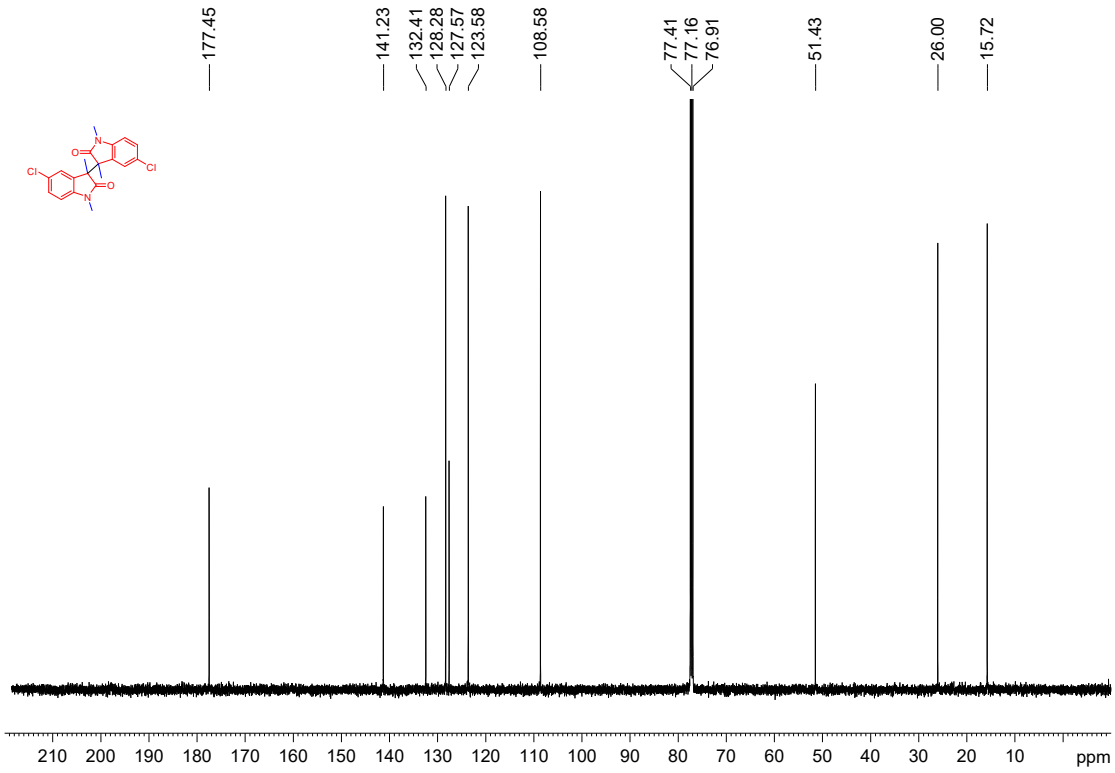
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **meso-2b**



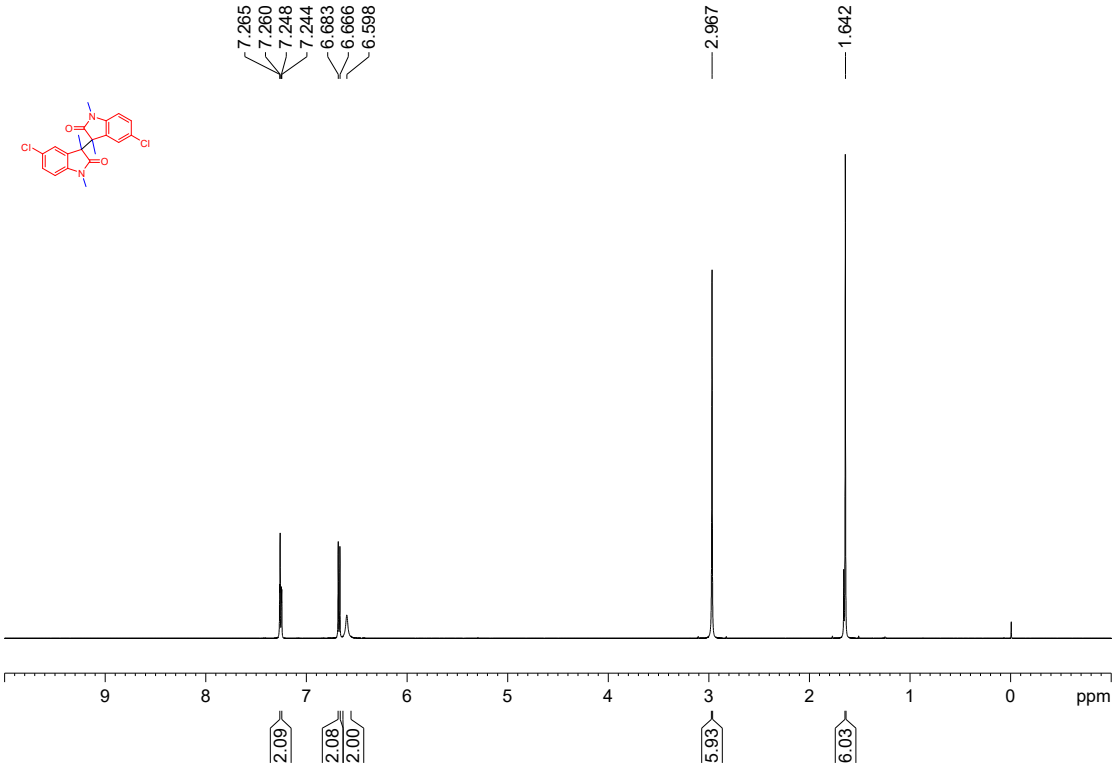
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D, L-2c**



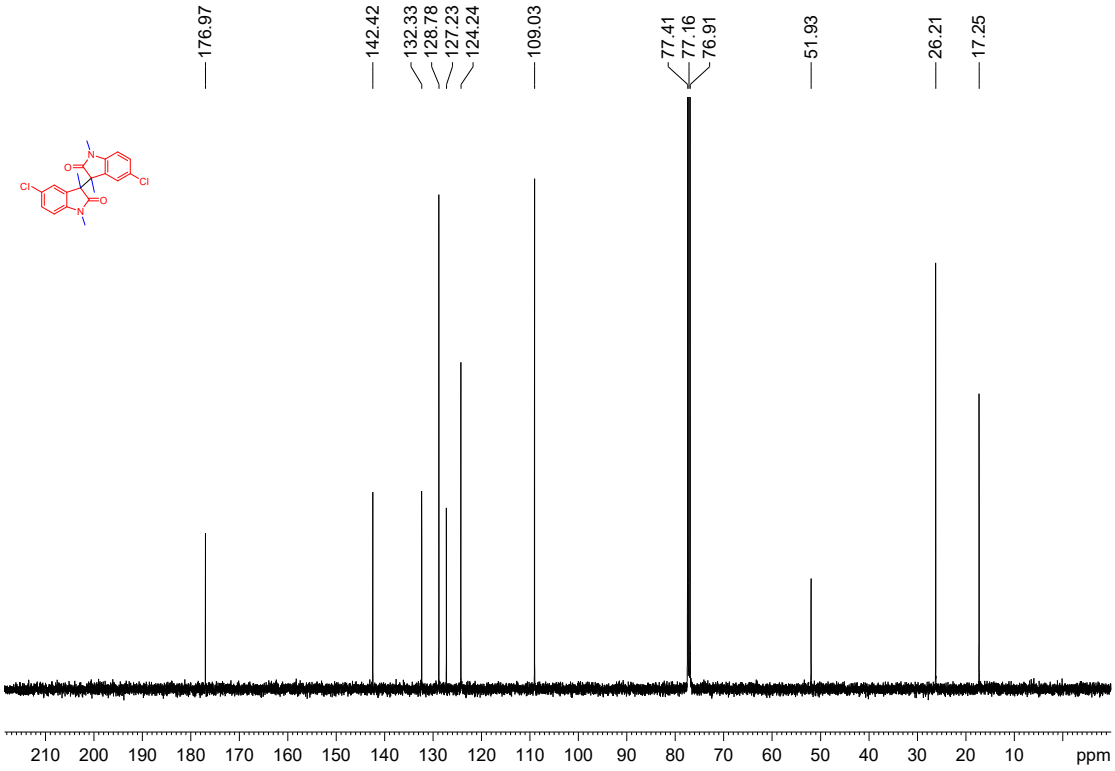
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D, L-2c**



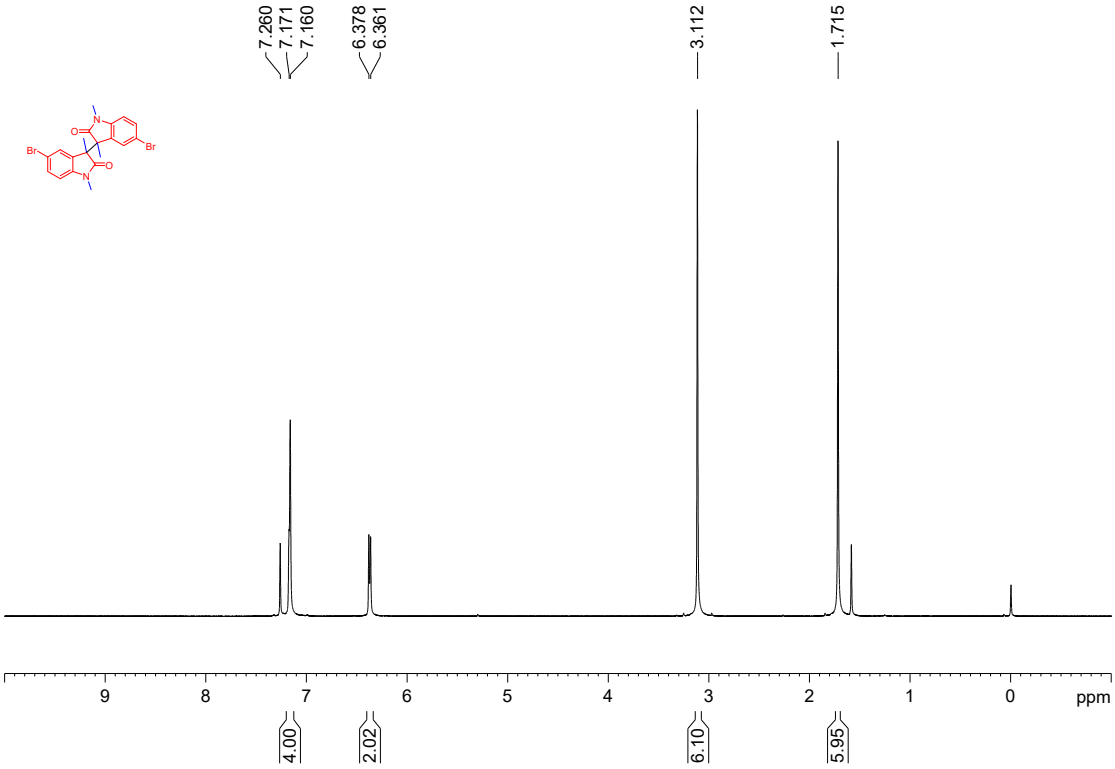
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2c**



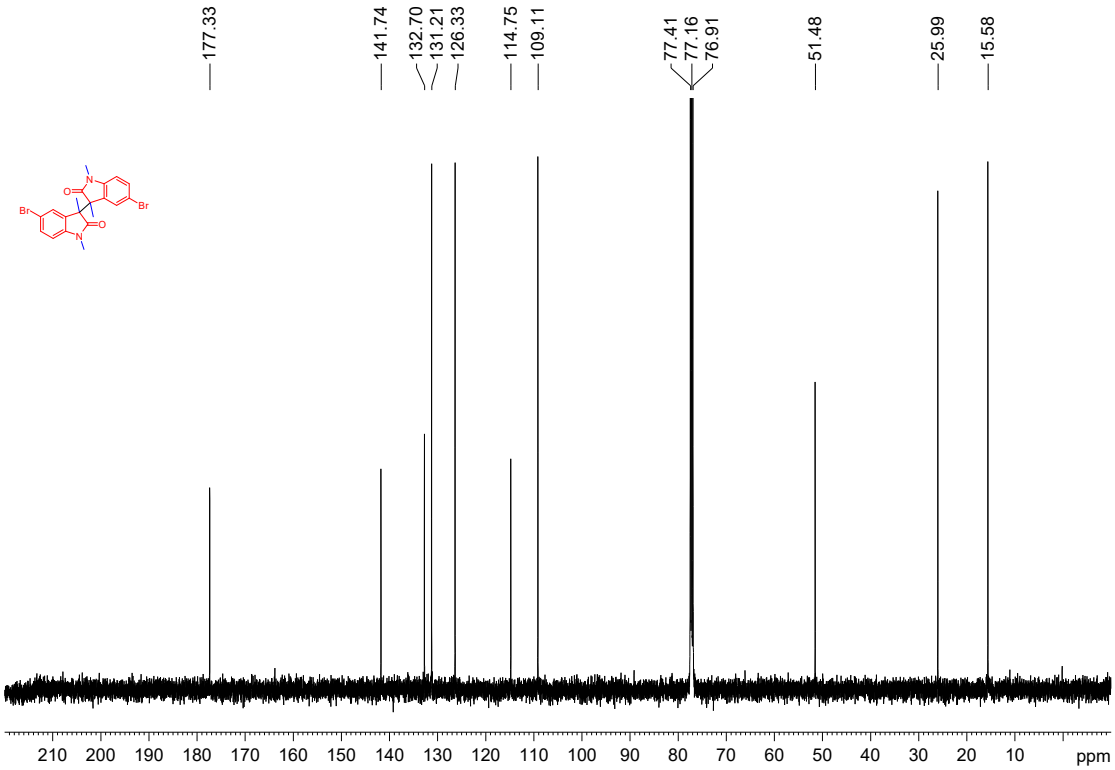
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2c**



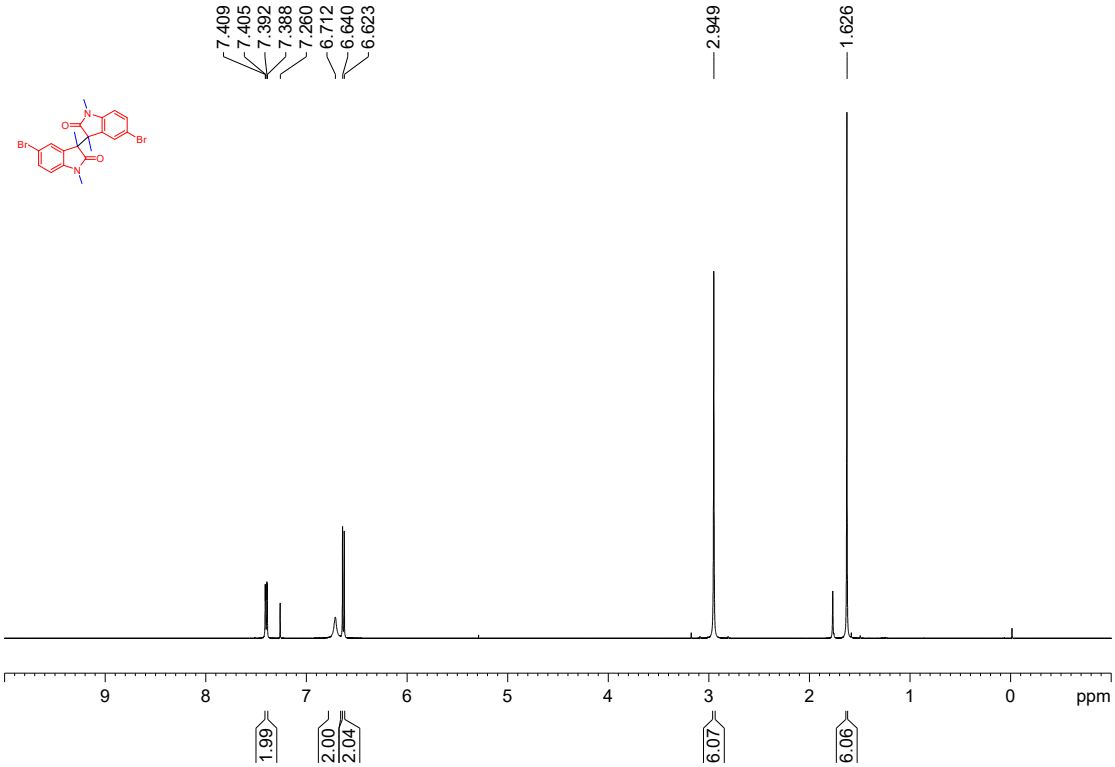
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D, L-2d**



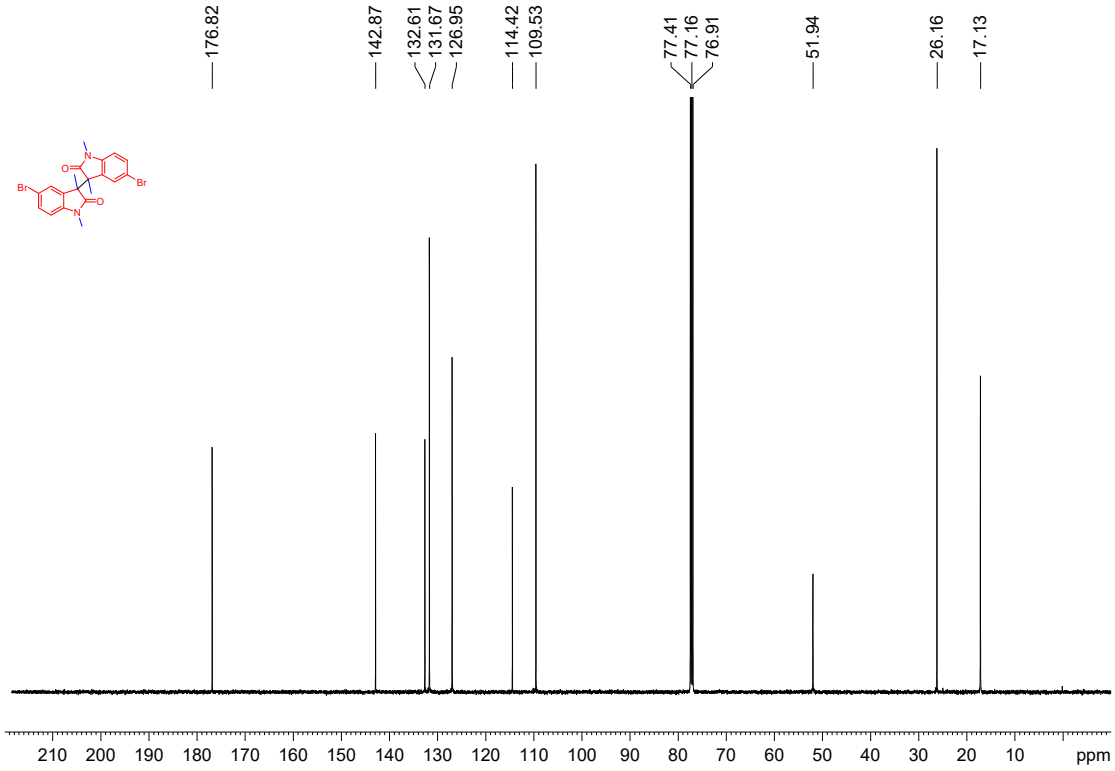
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D, L-2d**



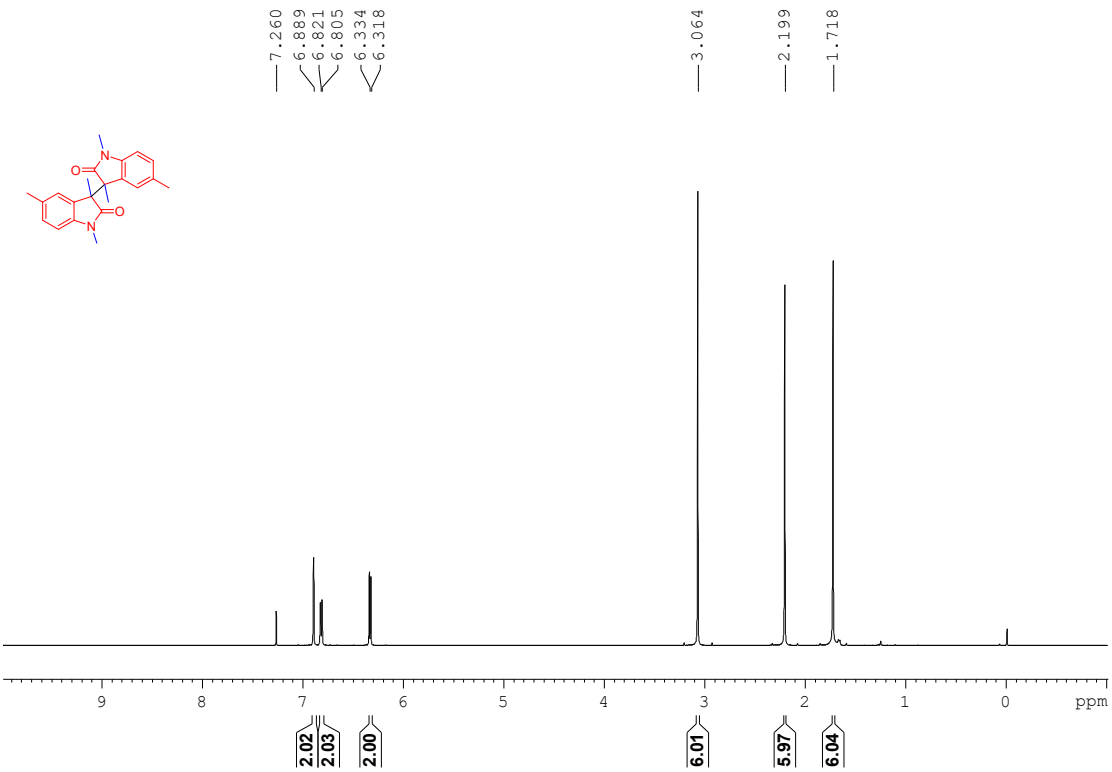
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2d**



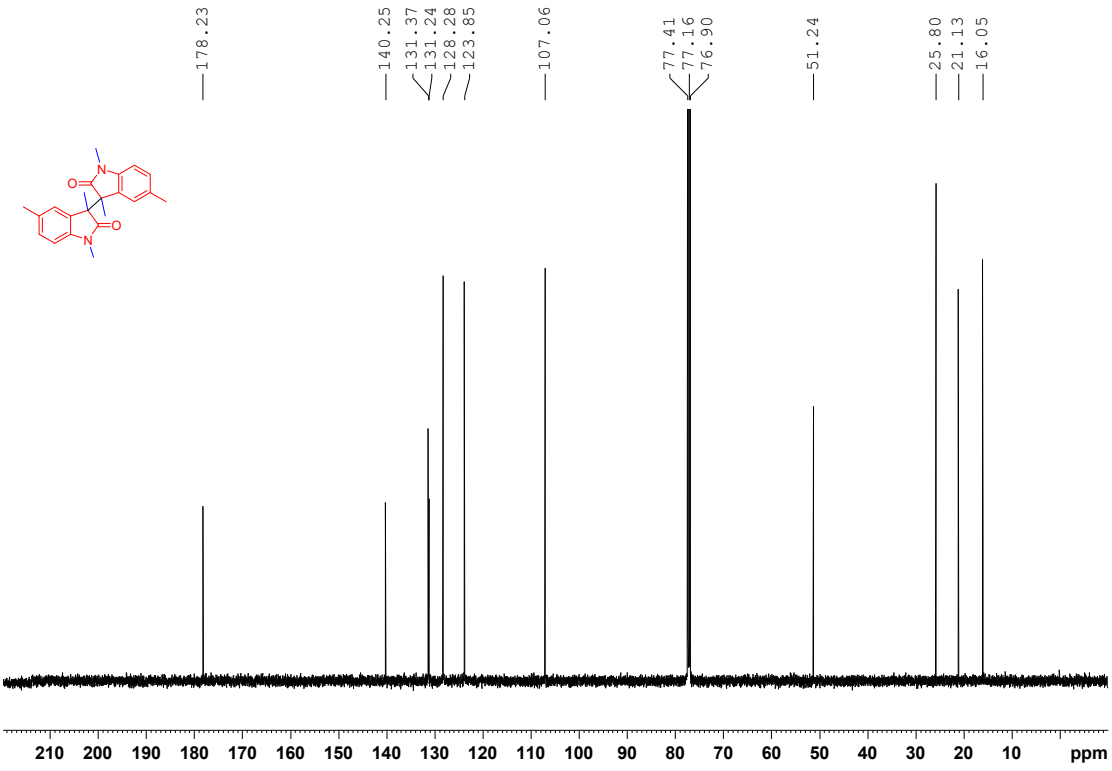
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2d**



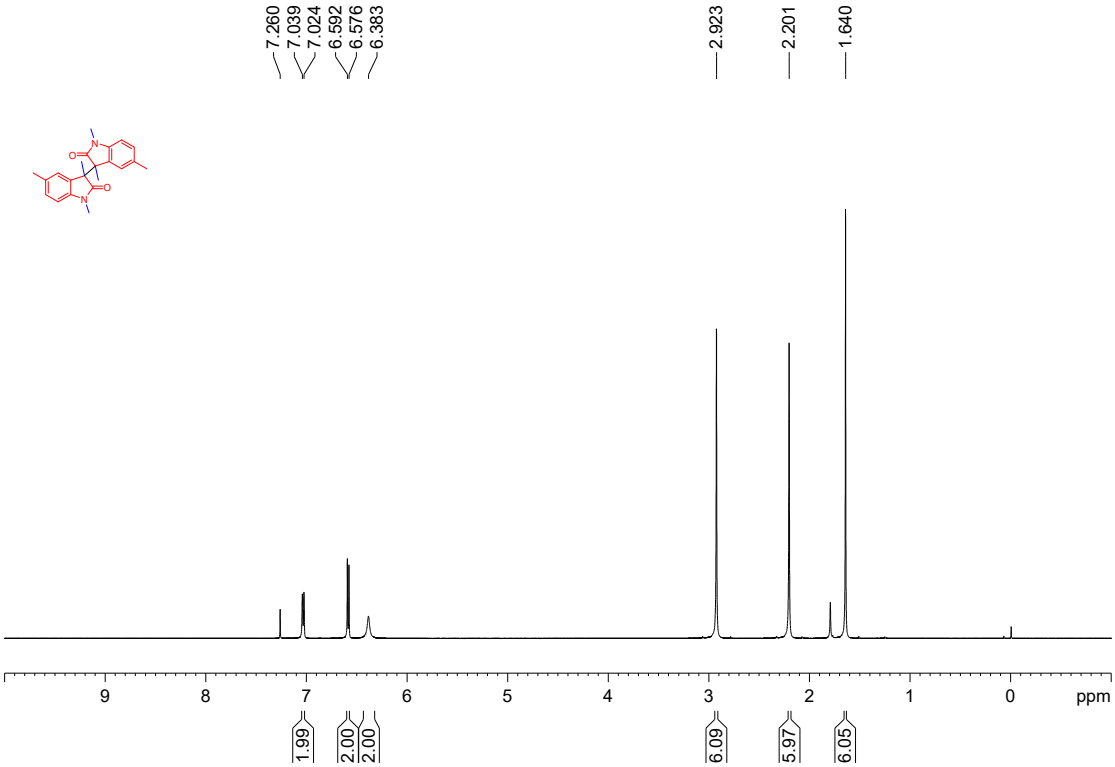
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2e**



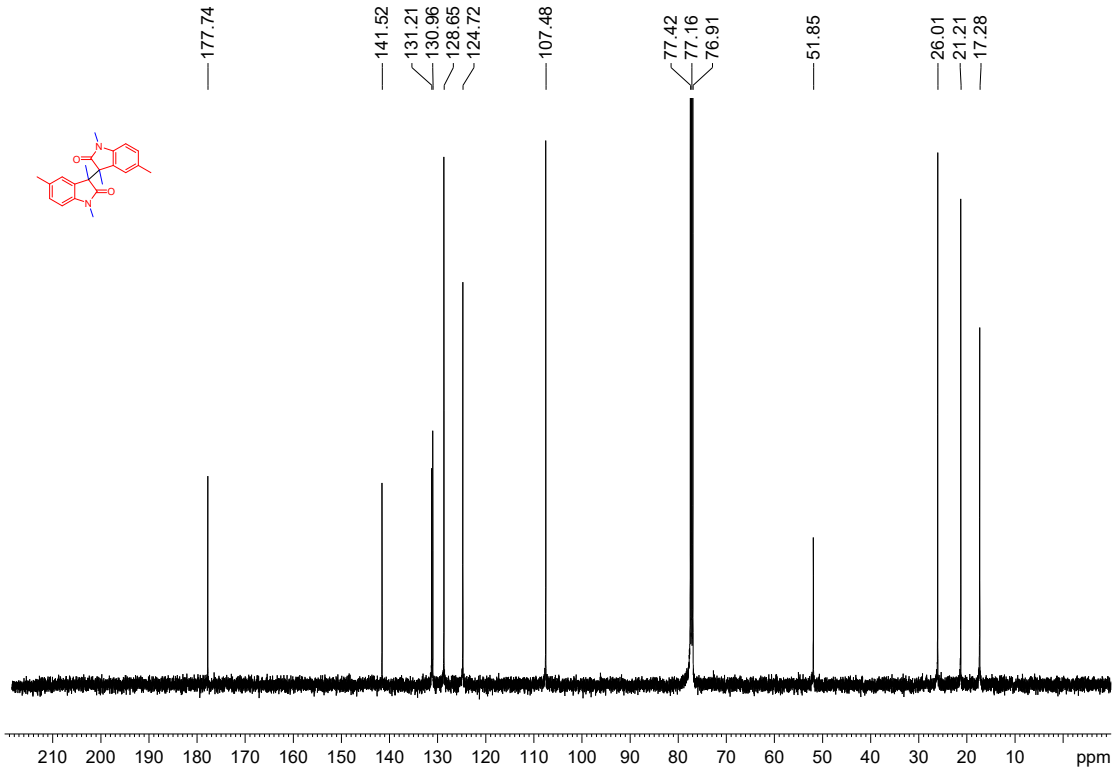
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2e**



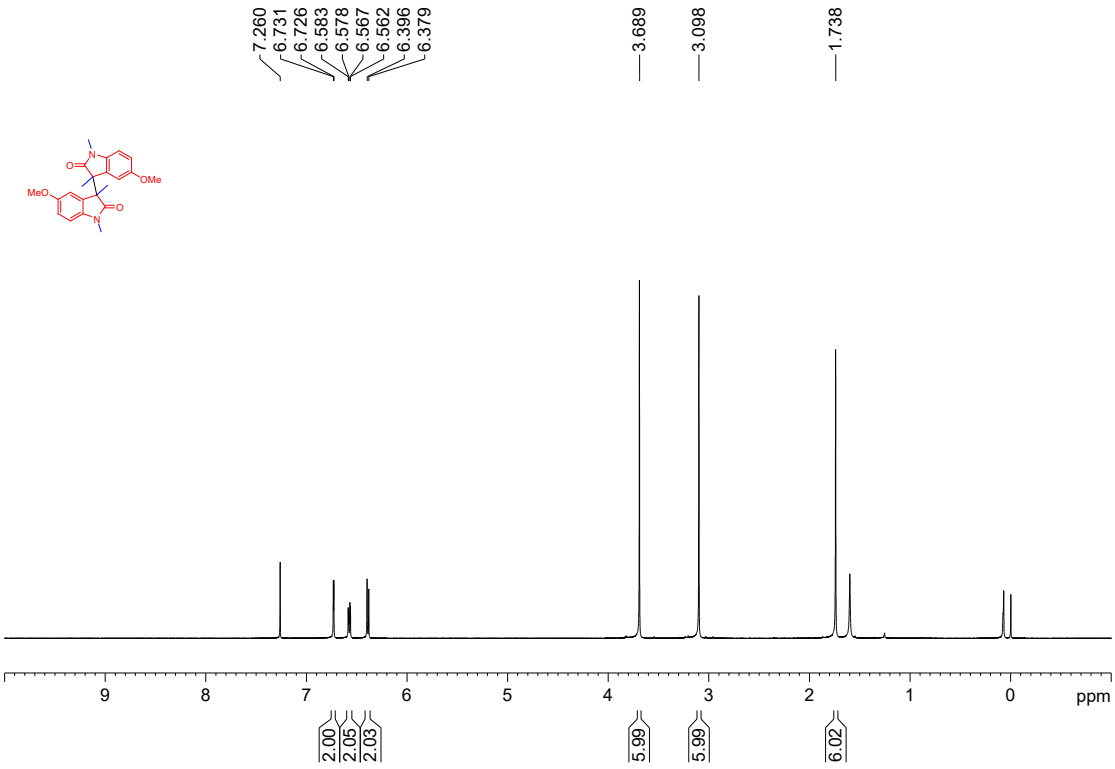
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2e**



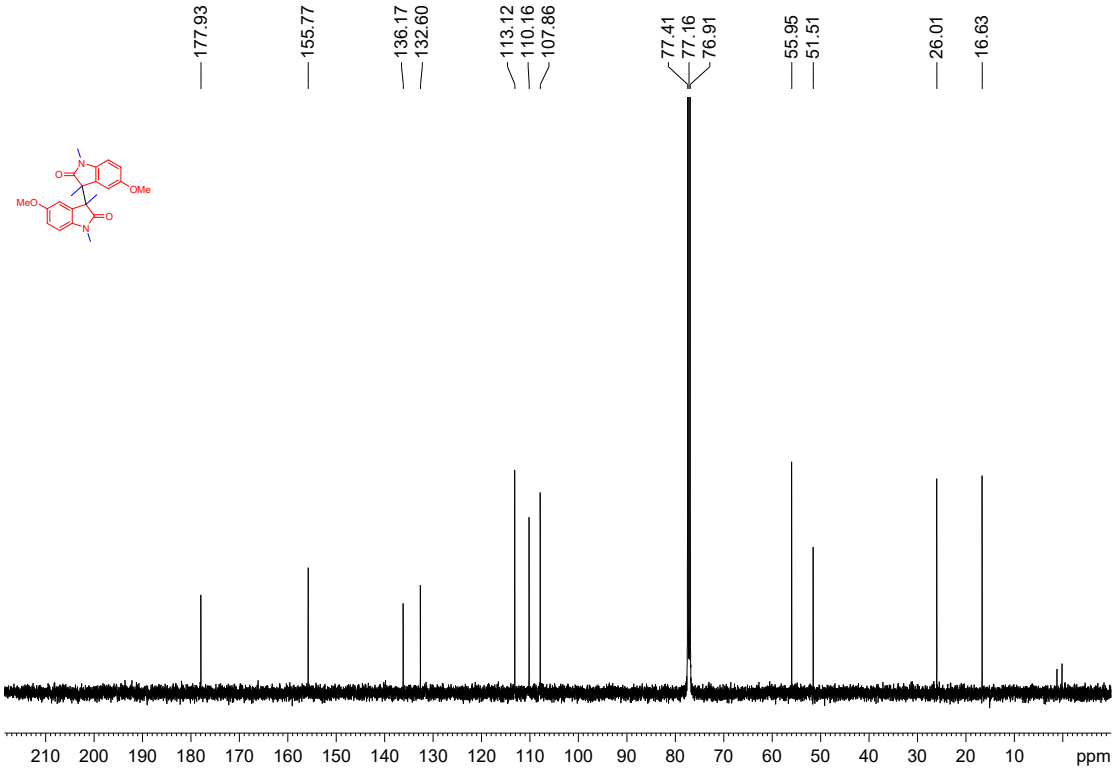
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2e**



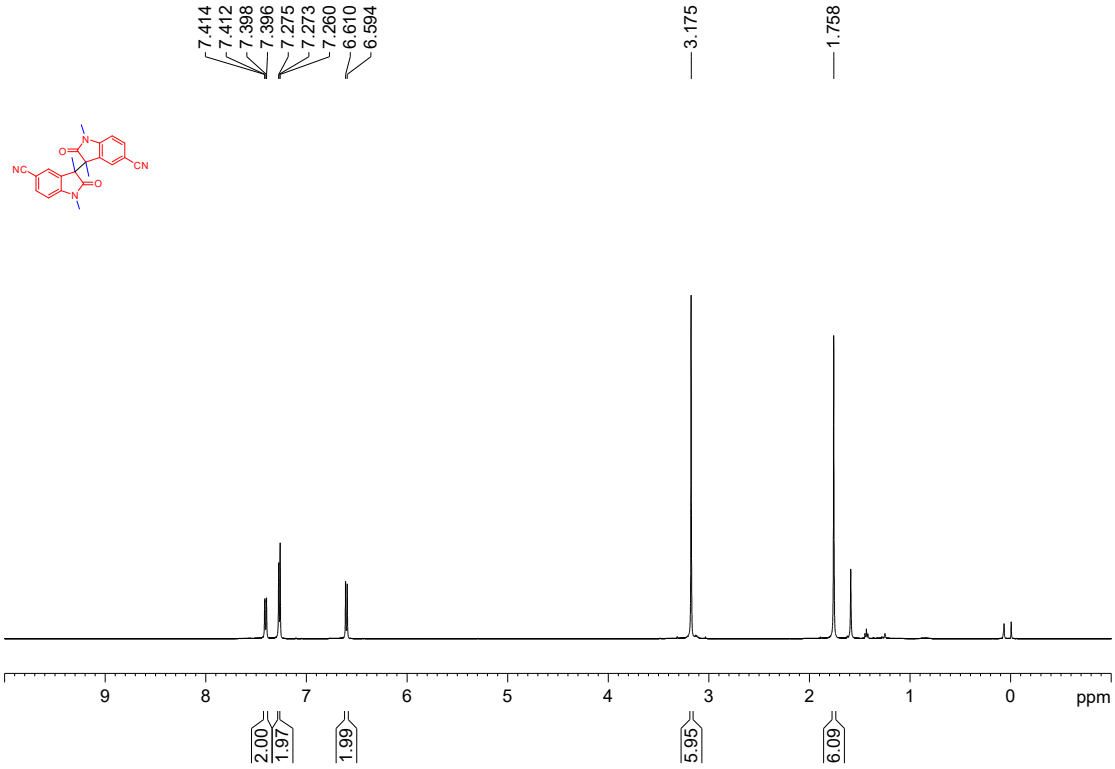
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2f**



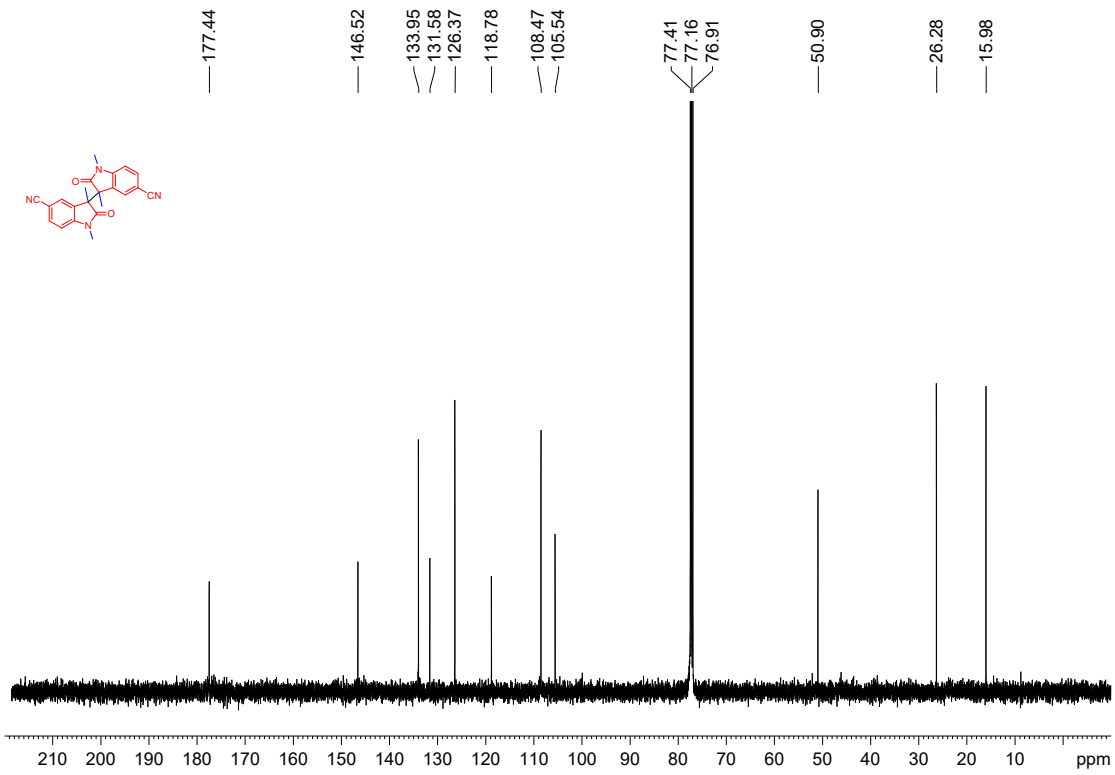
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2f**



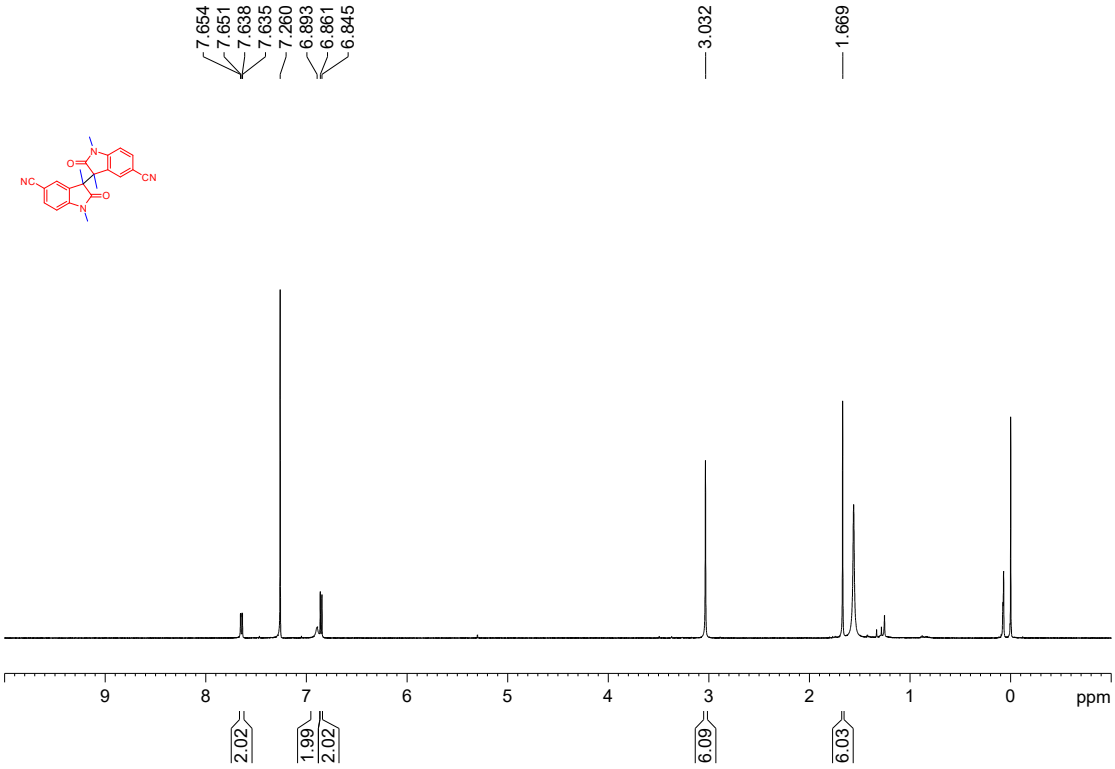
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2g**



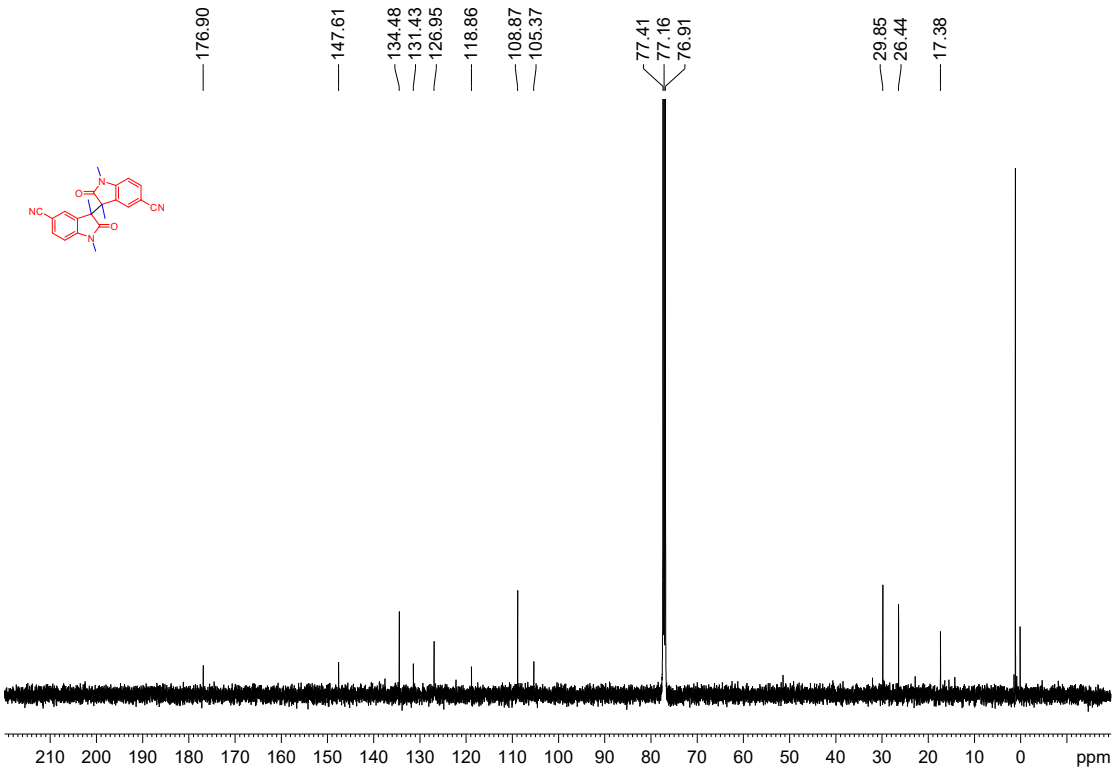
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2g**



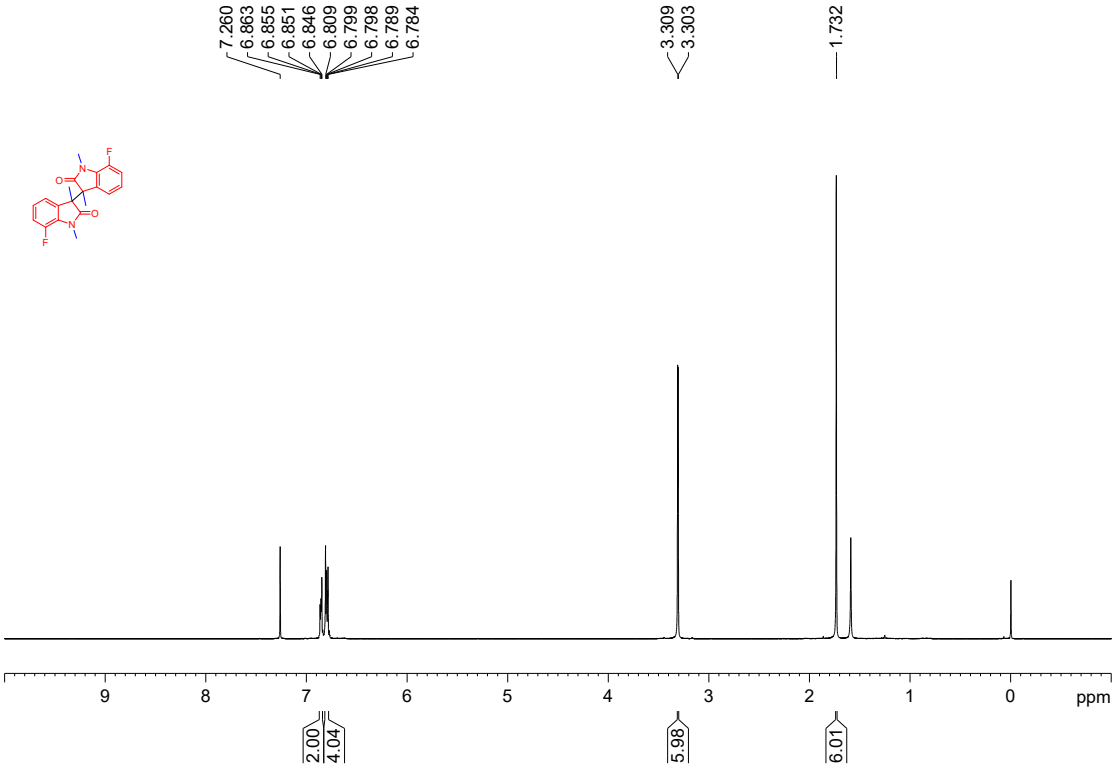
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2g**



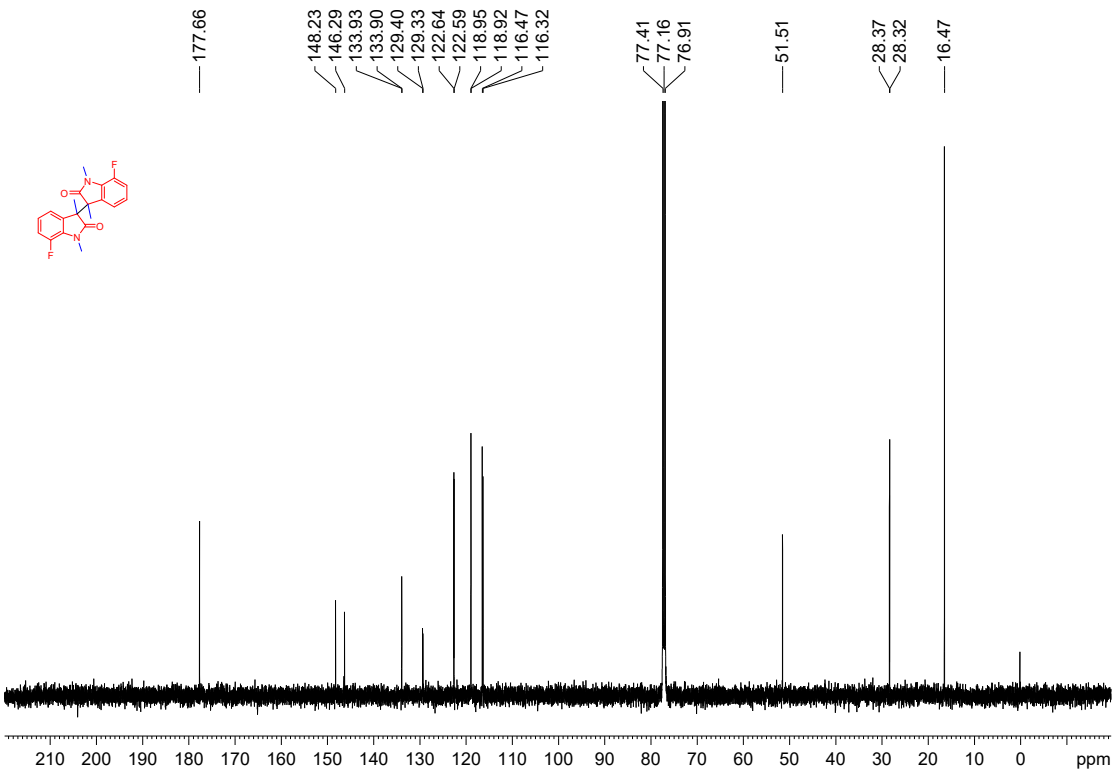
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2g**



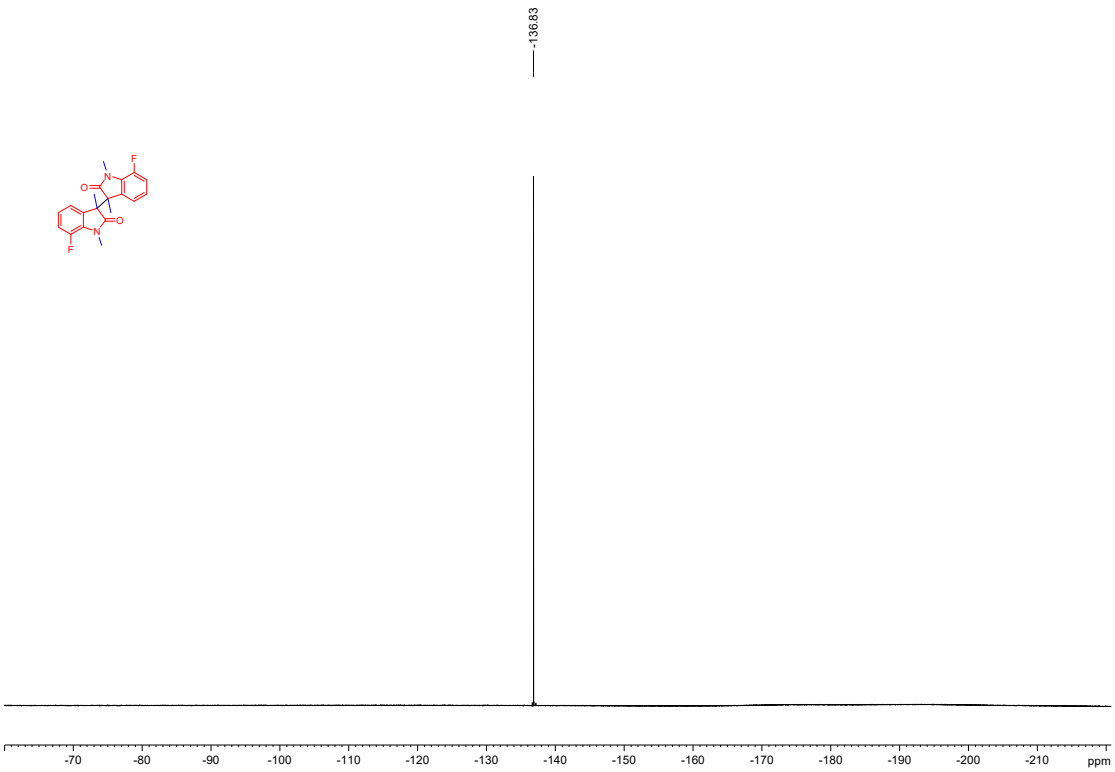
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D, L-2h**



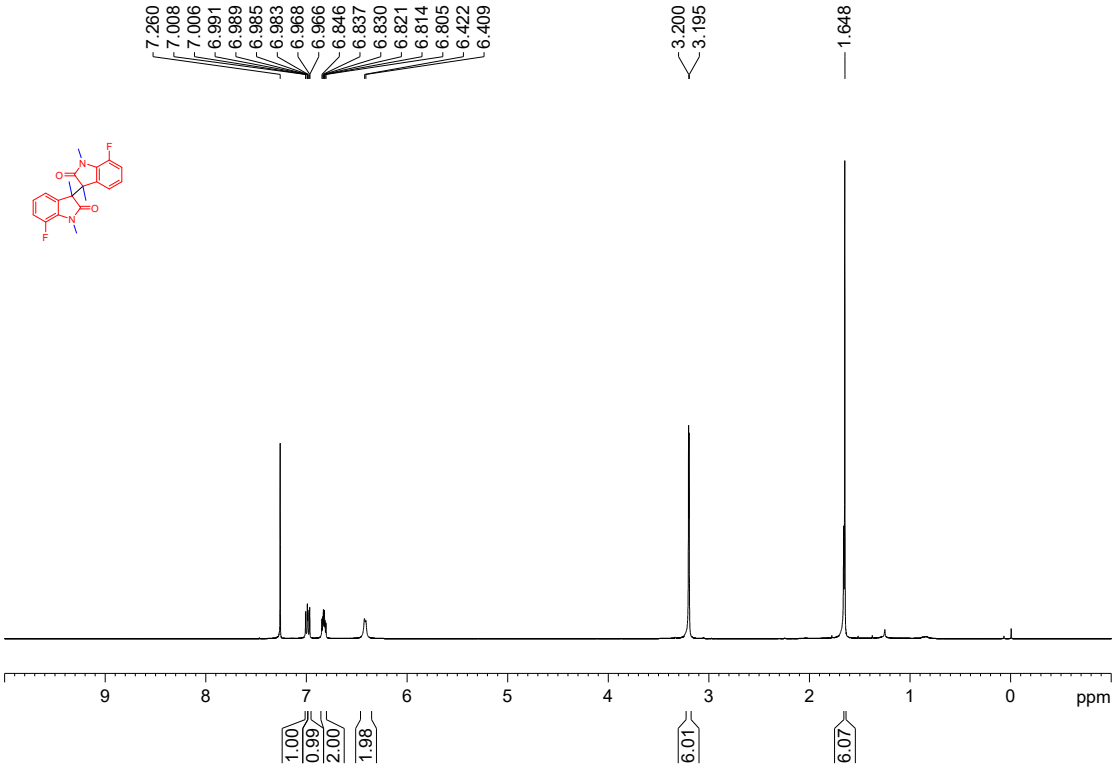
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D, L-2h**



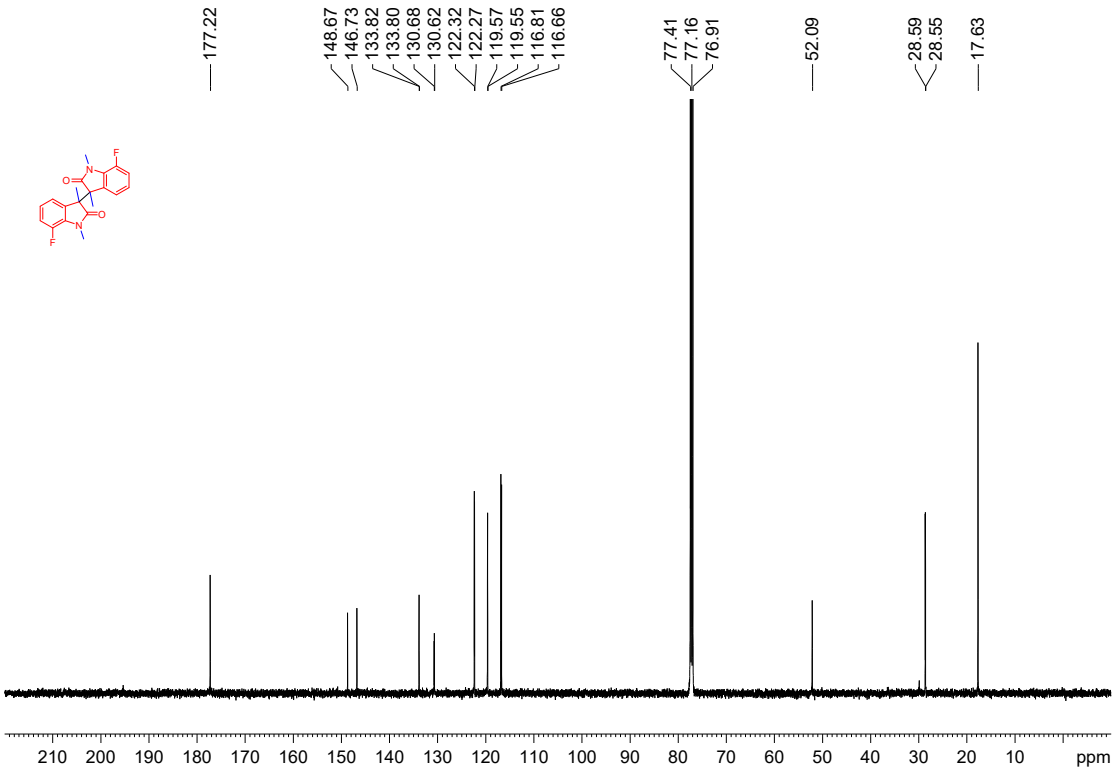
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **(±)-D, L-2h**



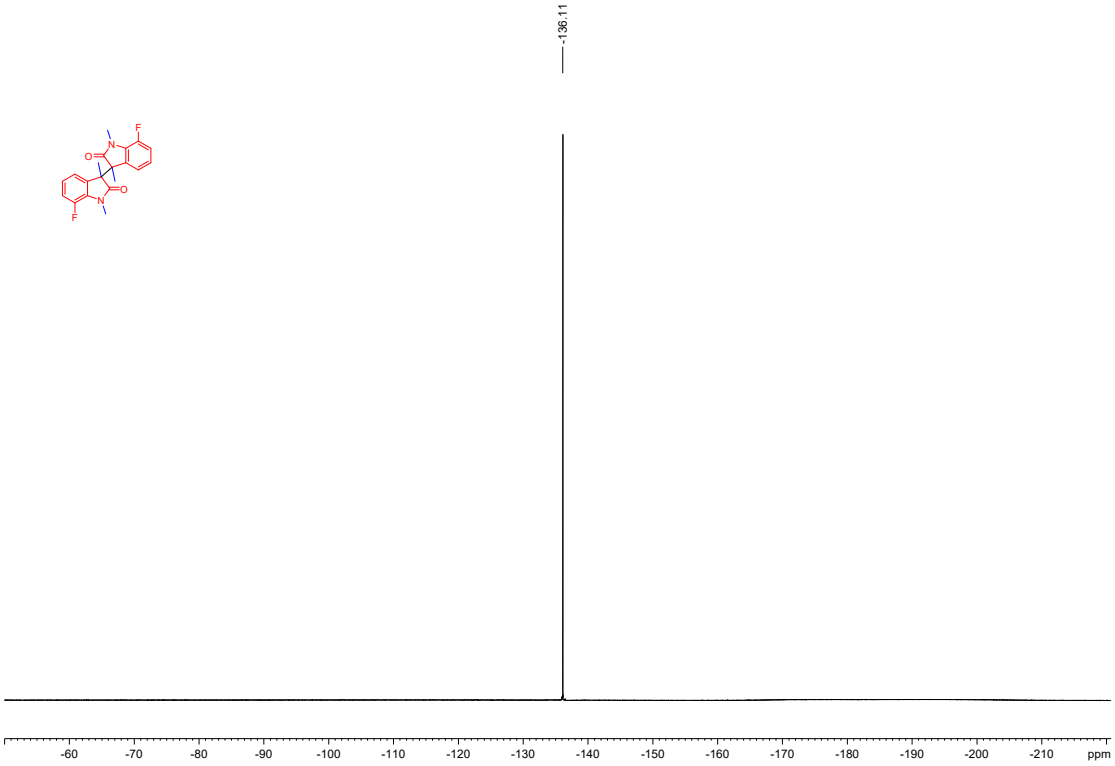
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2h**



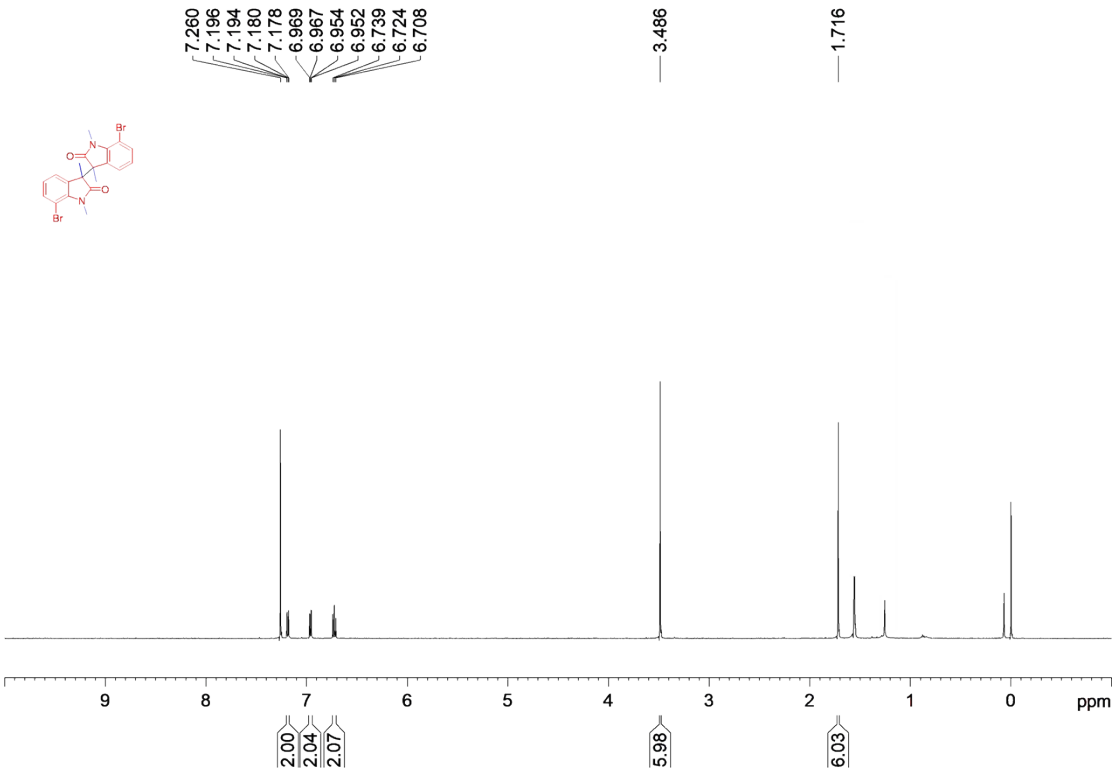
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2h**



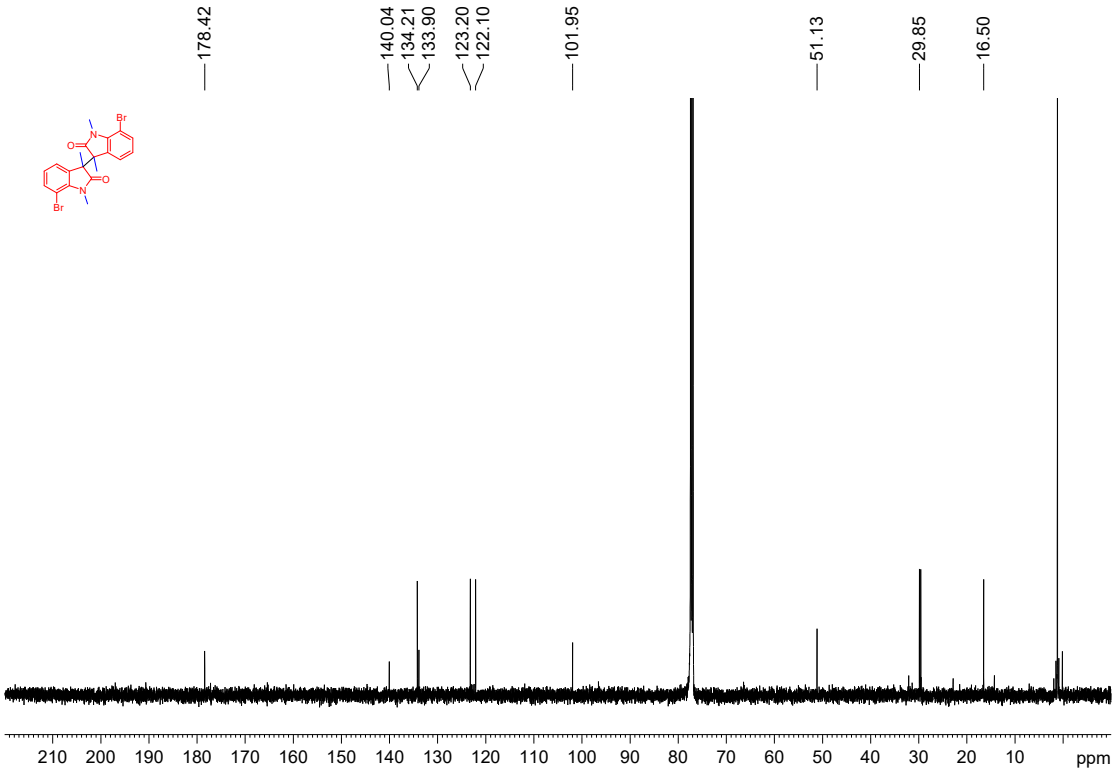
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **meso-2h**



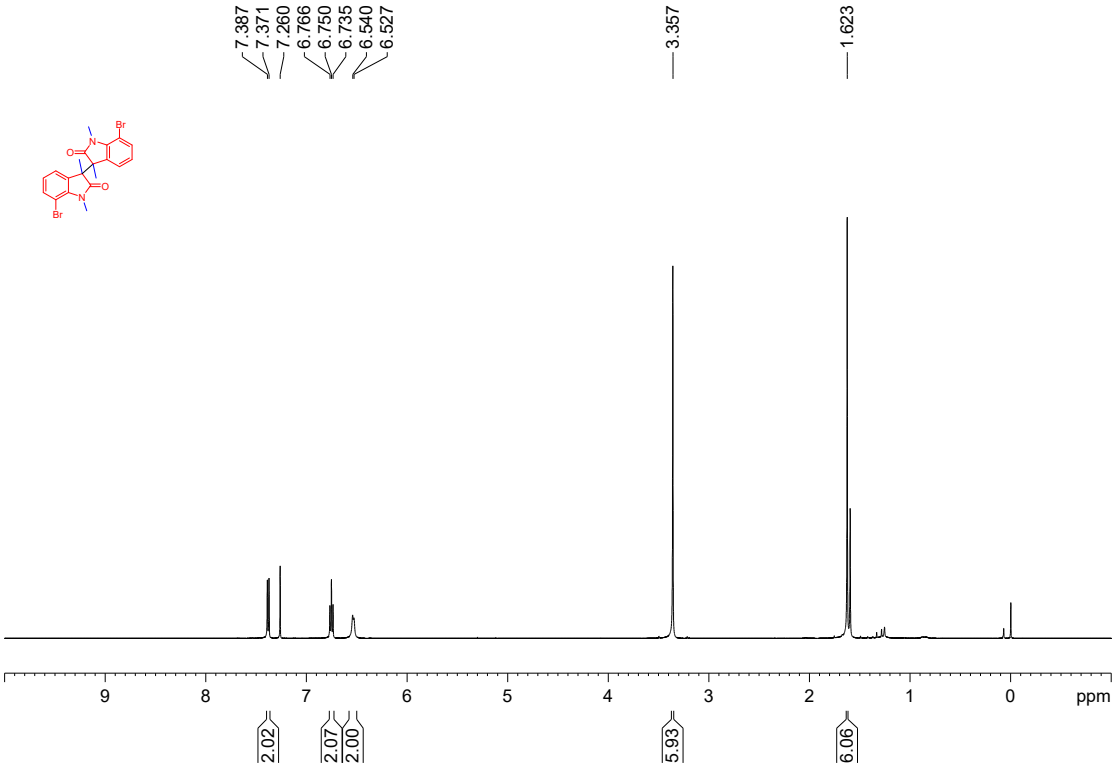
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2i**



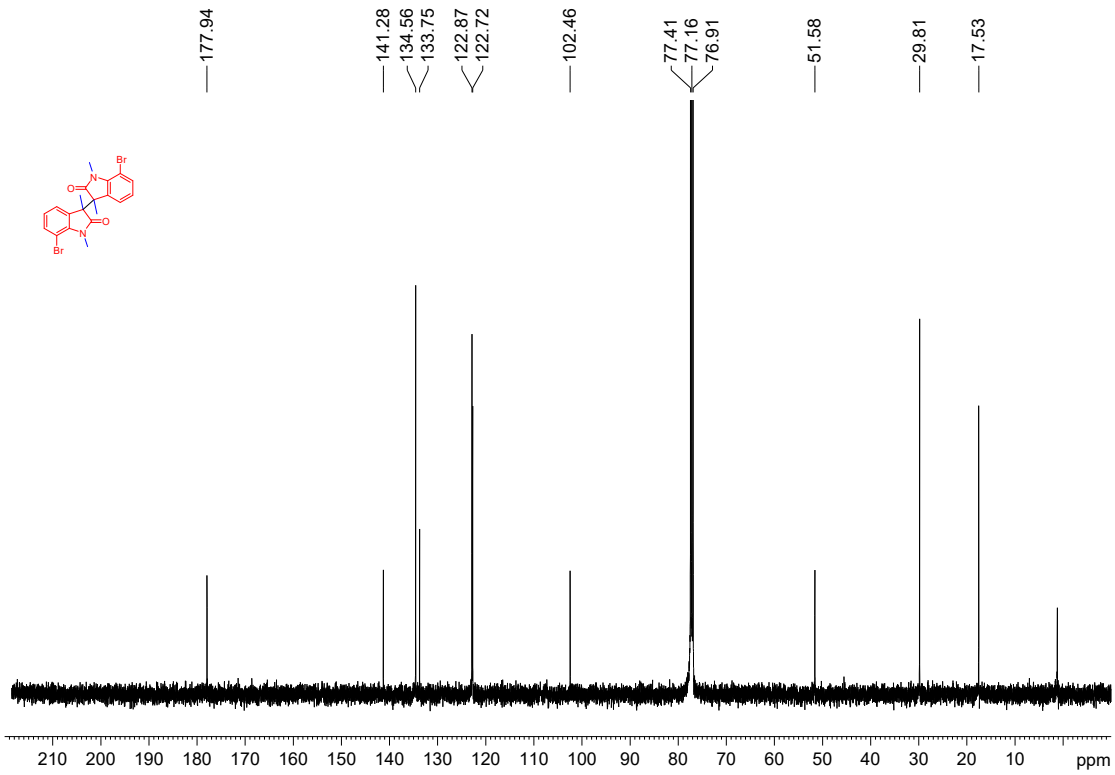
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2i**



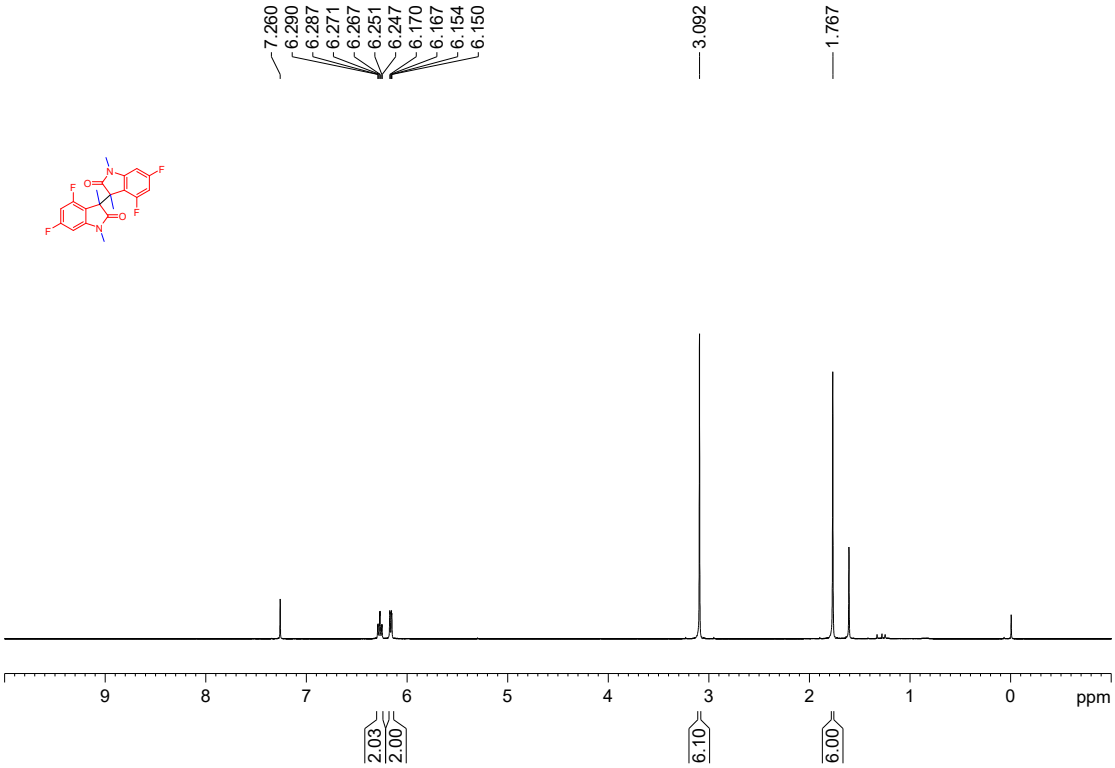
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2i**



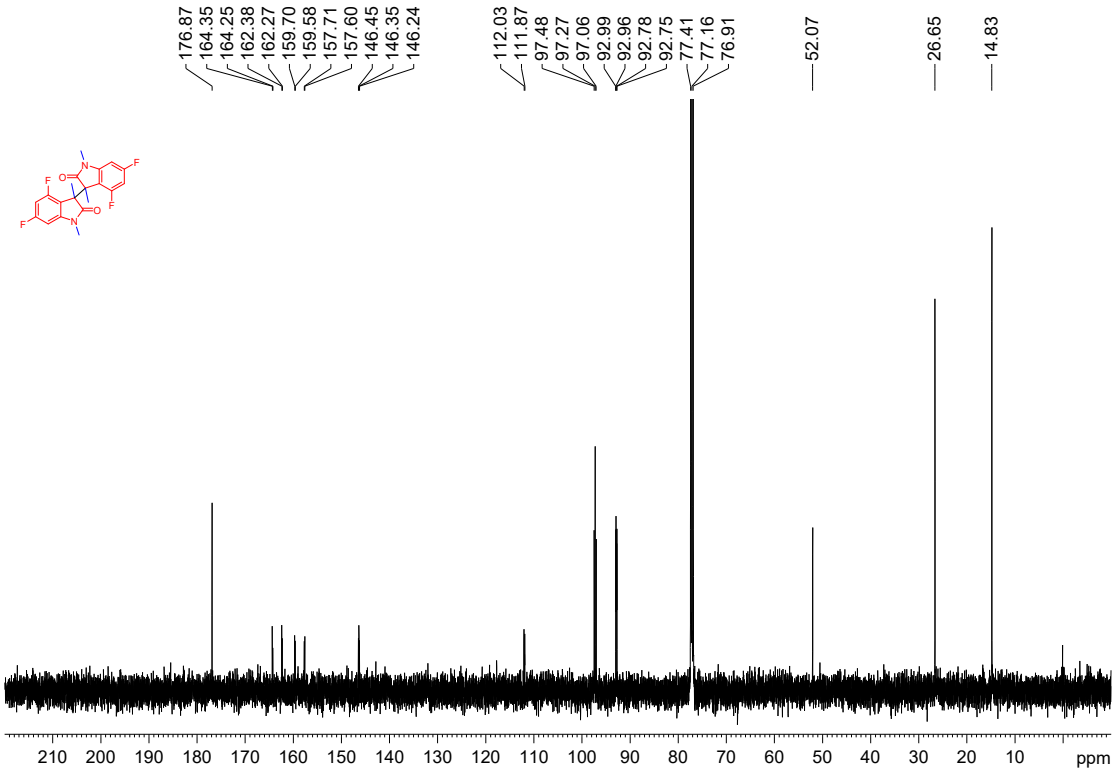
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2i**



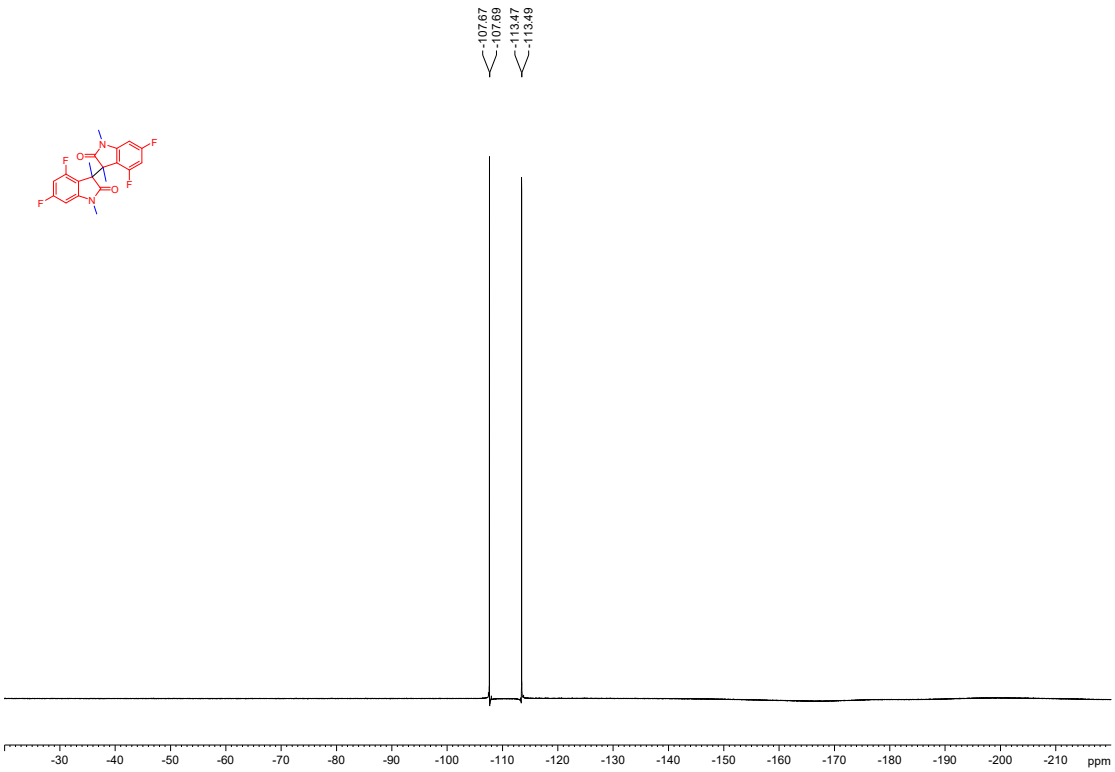
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2j**



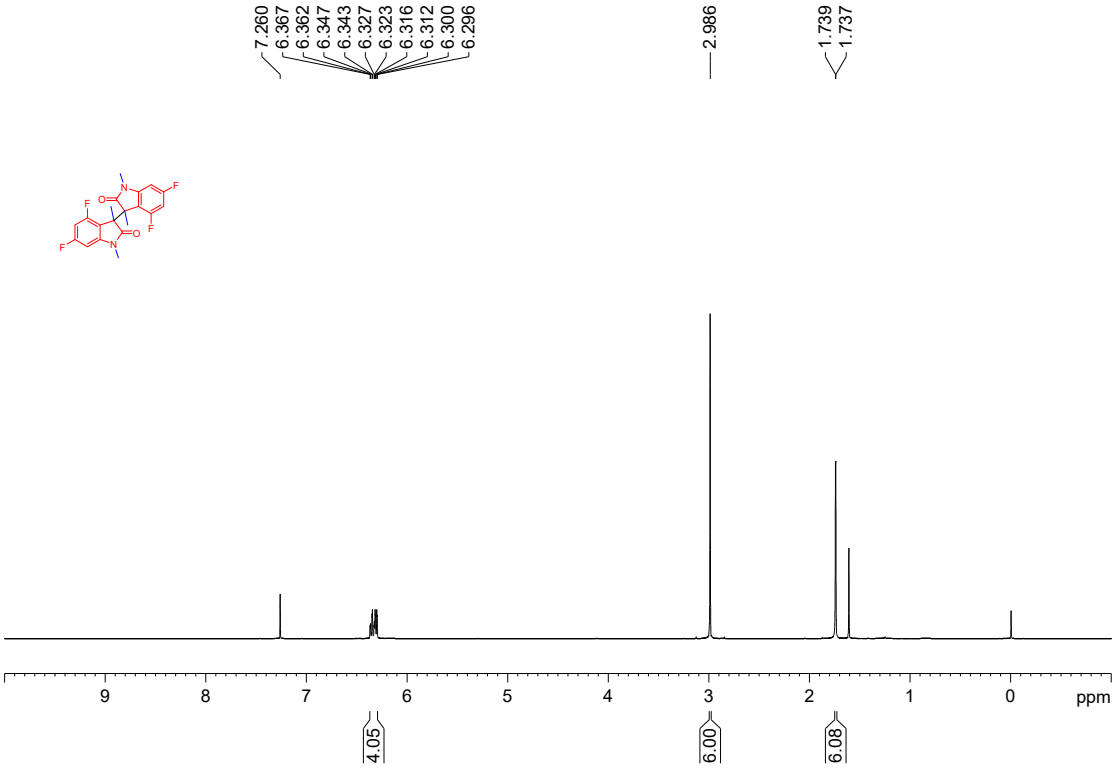
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2j**



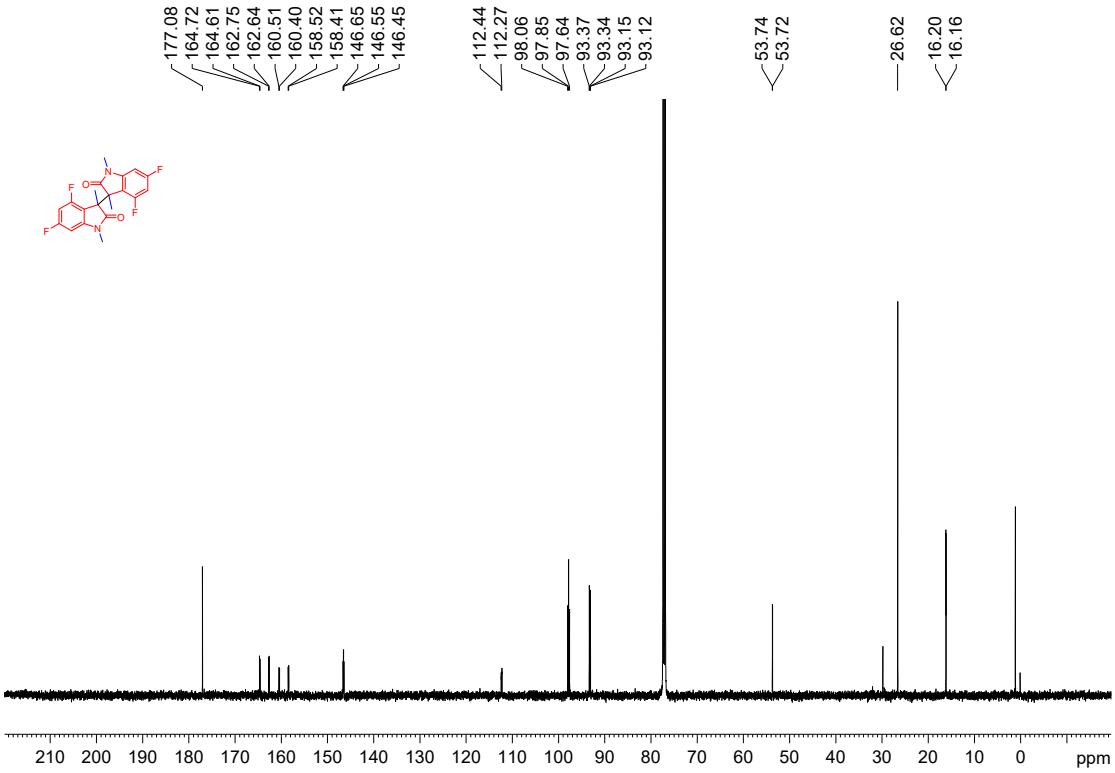
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **(±)-D,L-2j**



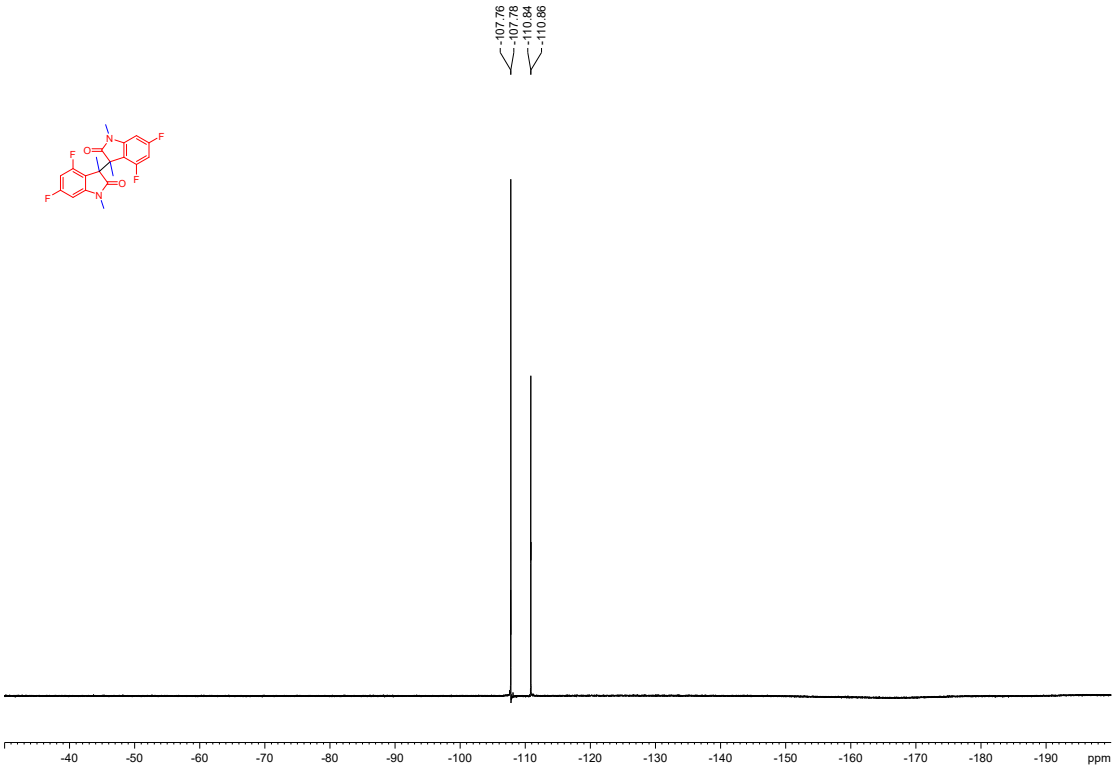
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2j**



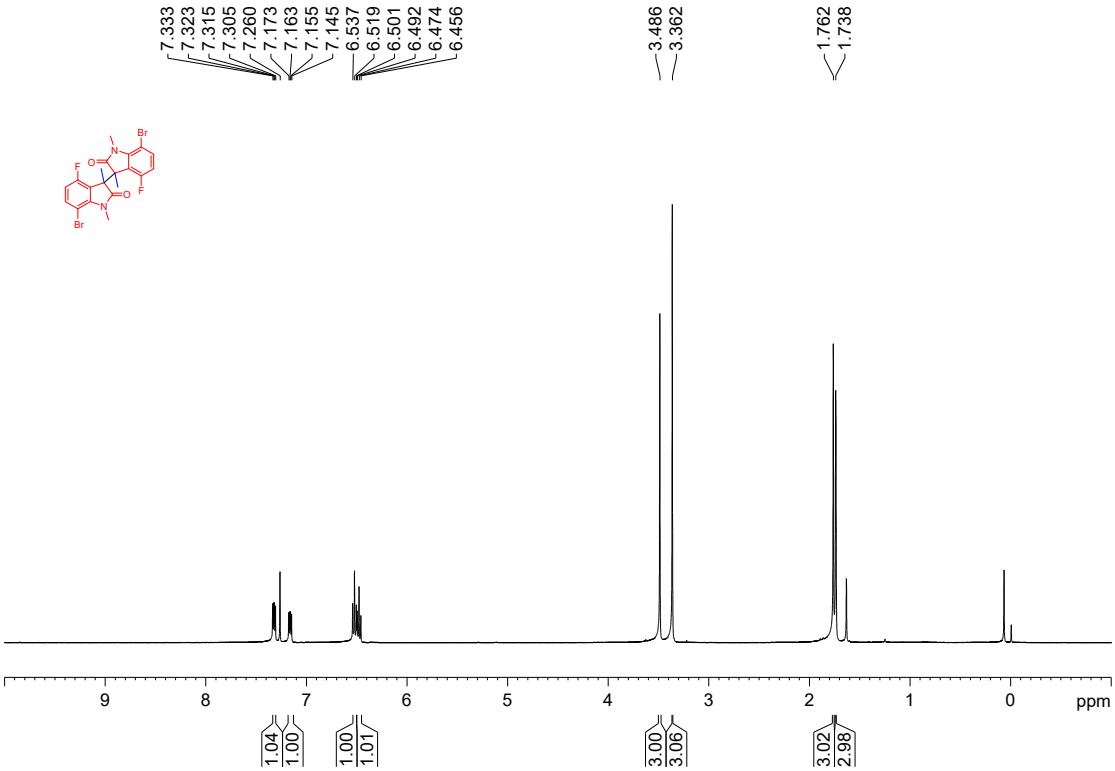
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2j**



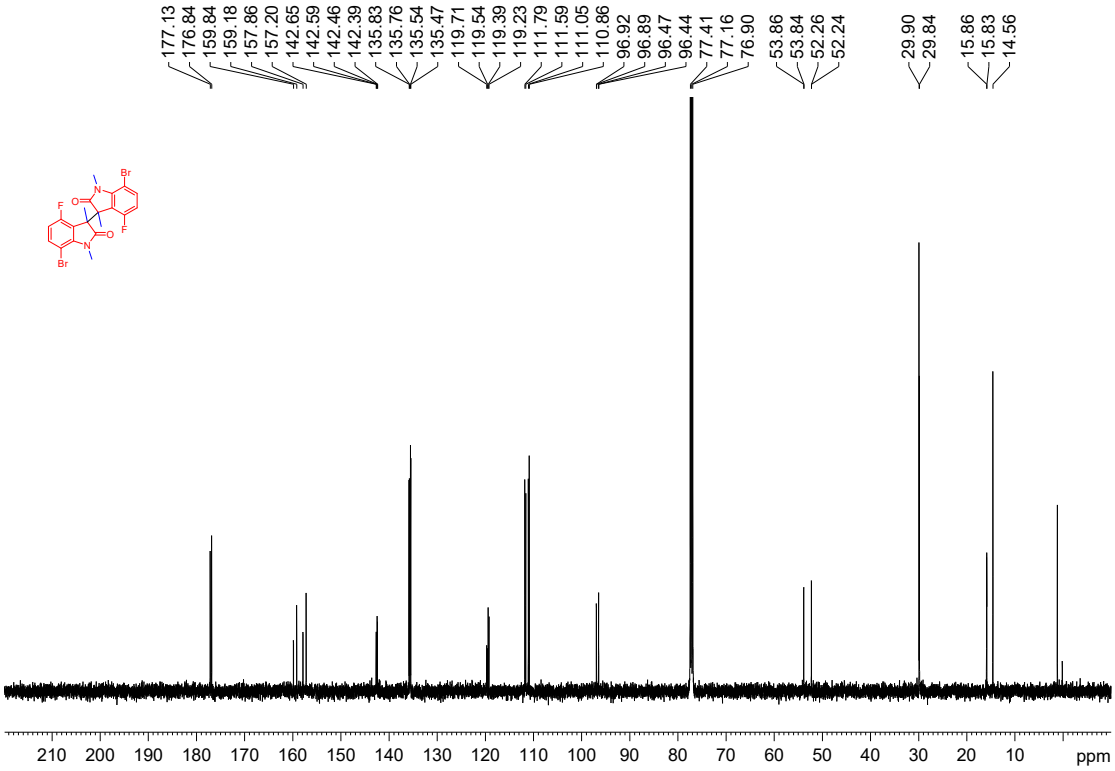
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **meso-2j**



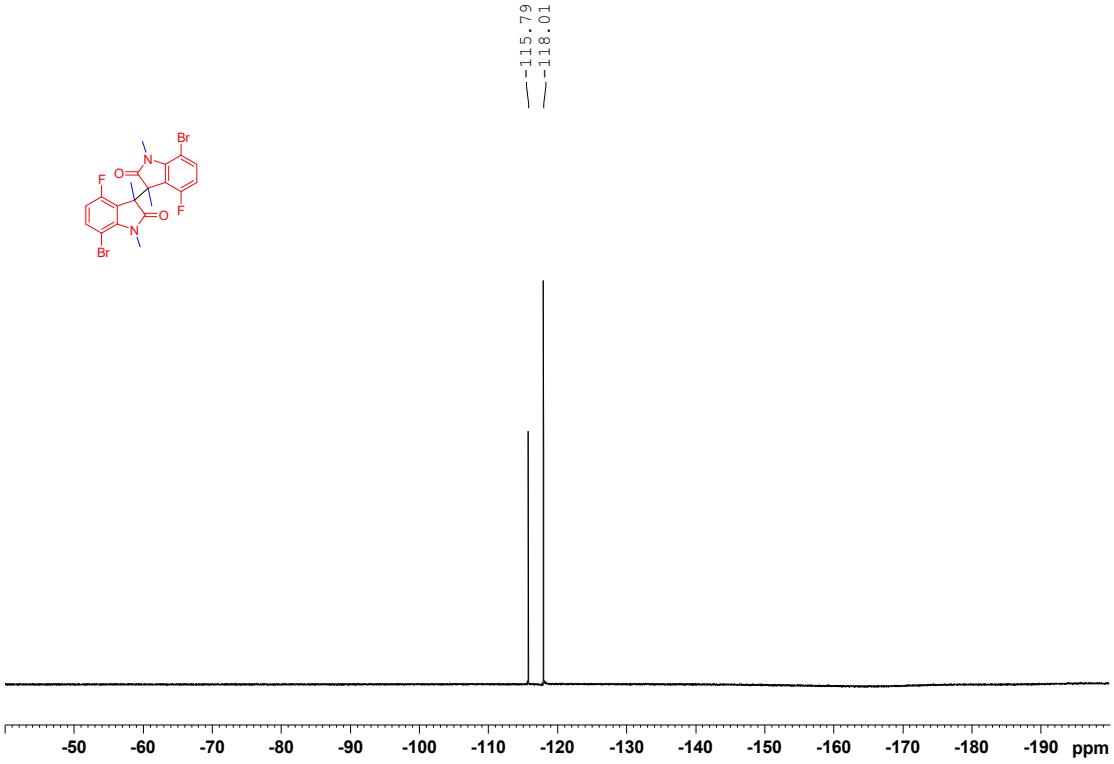
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **2k** (*the mixture of diastereoisomers*)



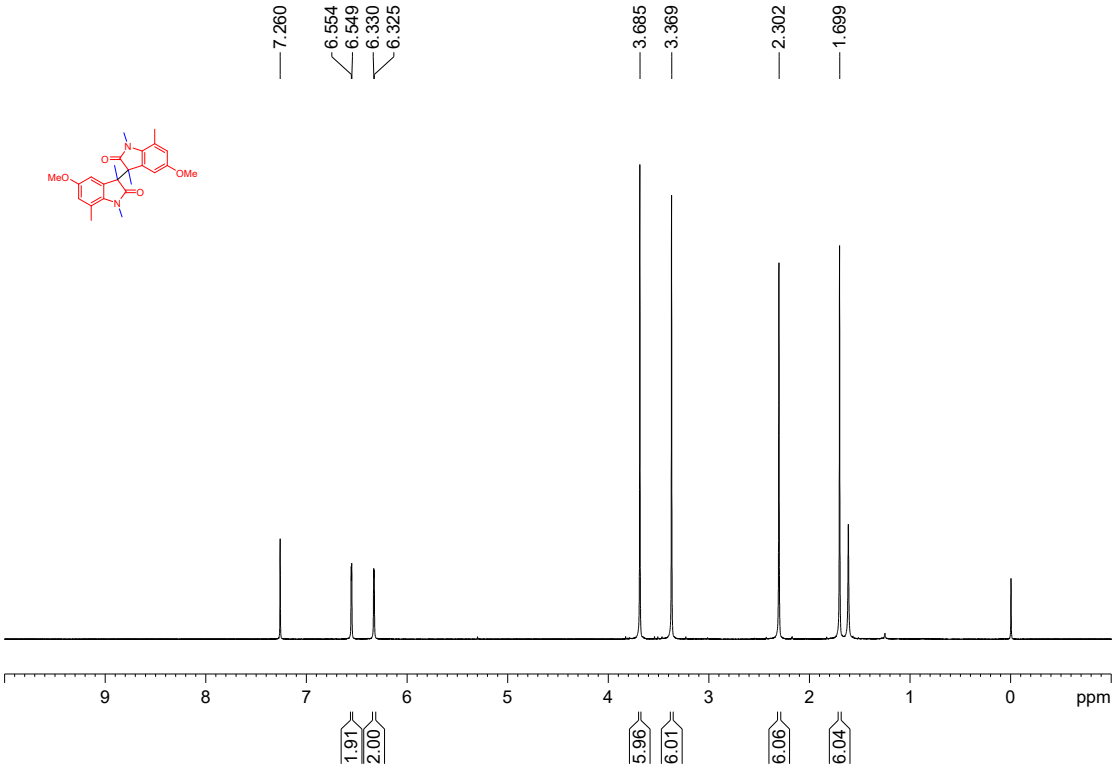
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **2k**, (*the mixture of diastereoisomers*)



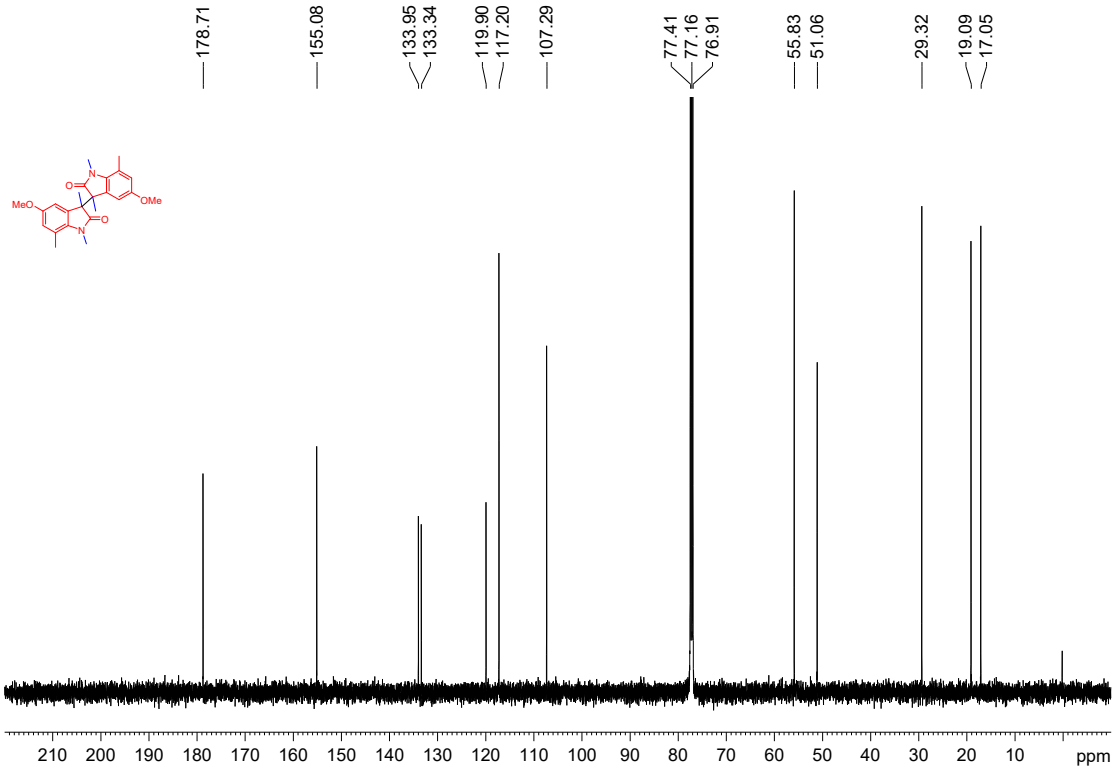
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **2k**, (*the mixture of diastereoisomers*)



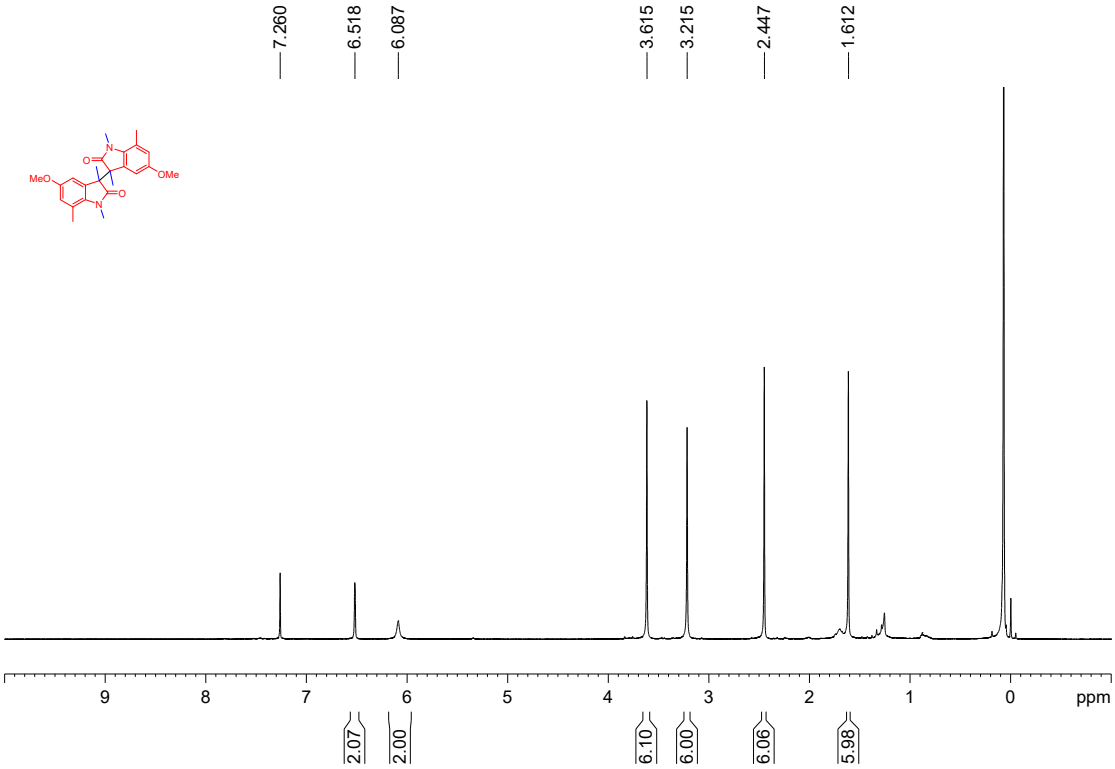
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2l**



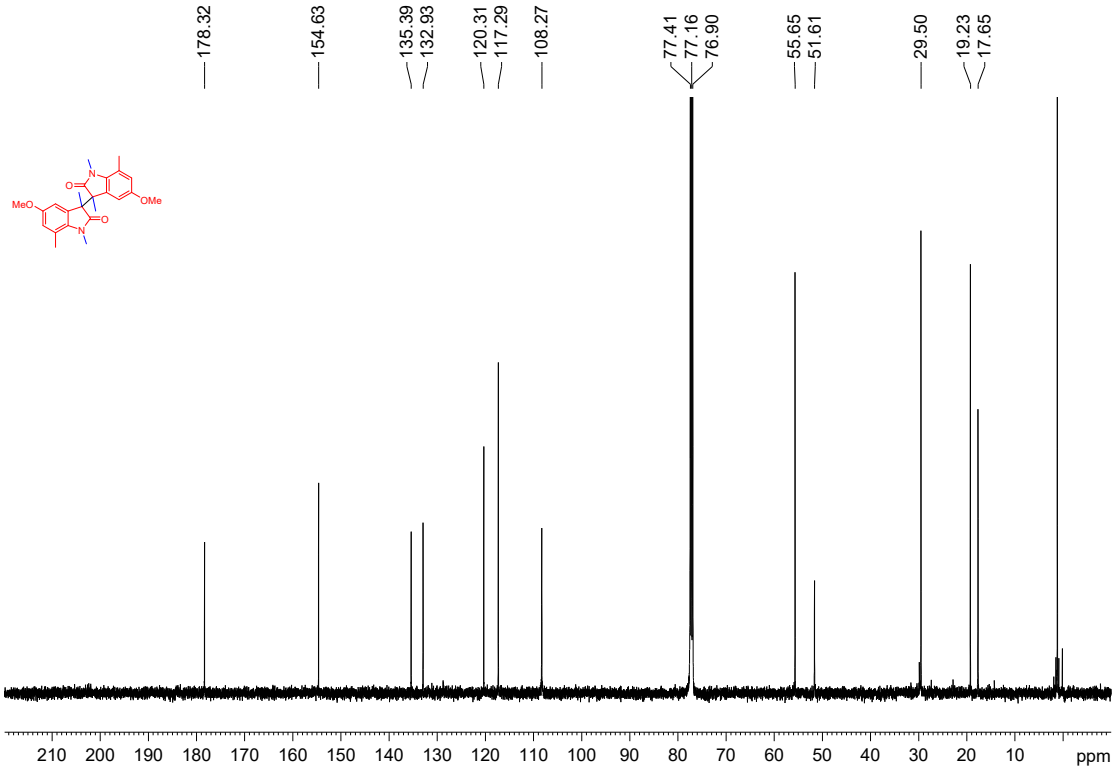
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2l**



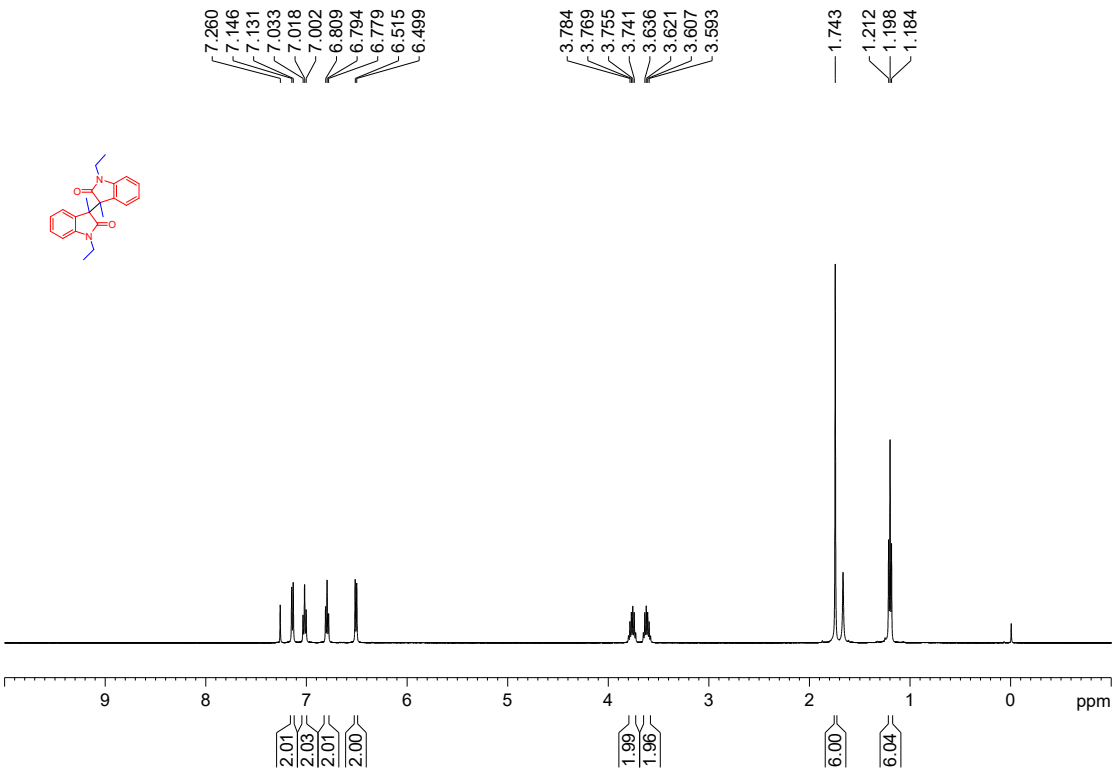
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2l**



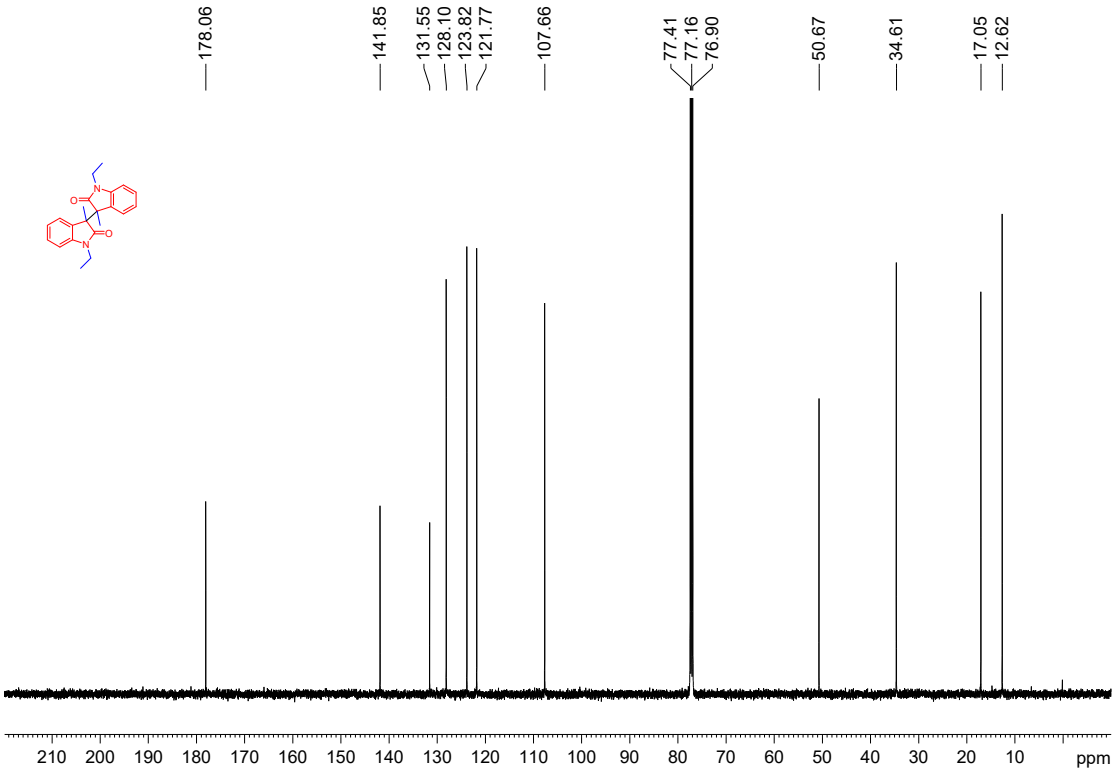
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2l**



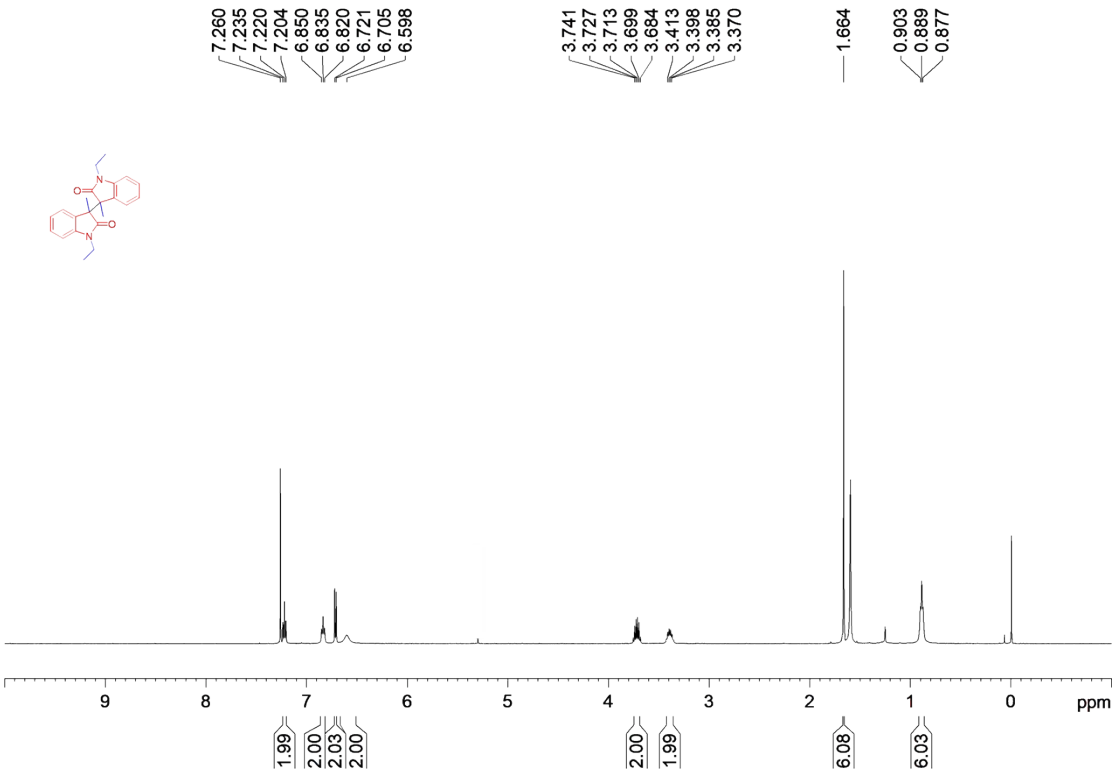
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2m**



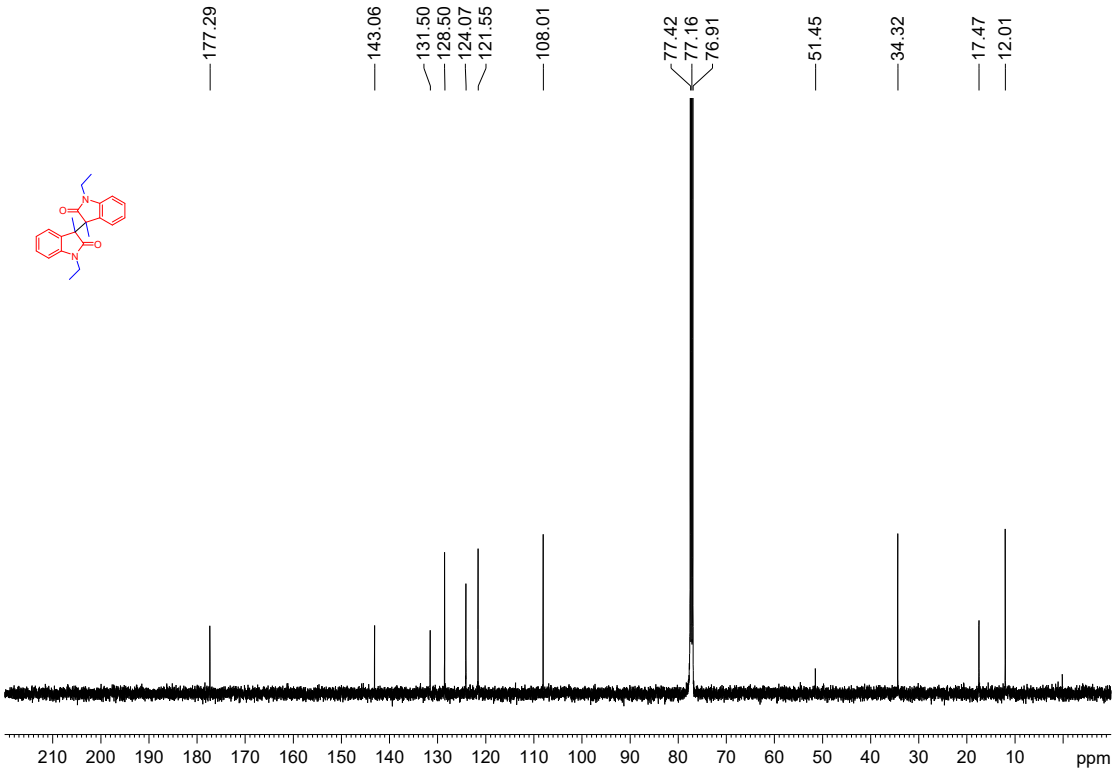
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2m**



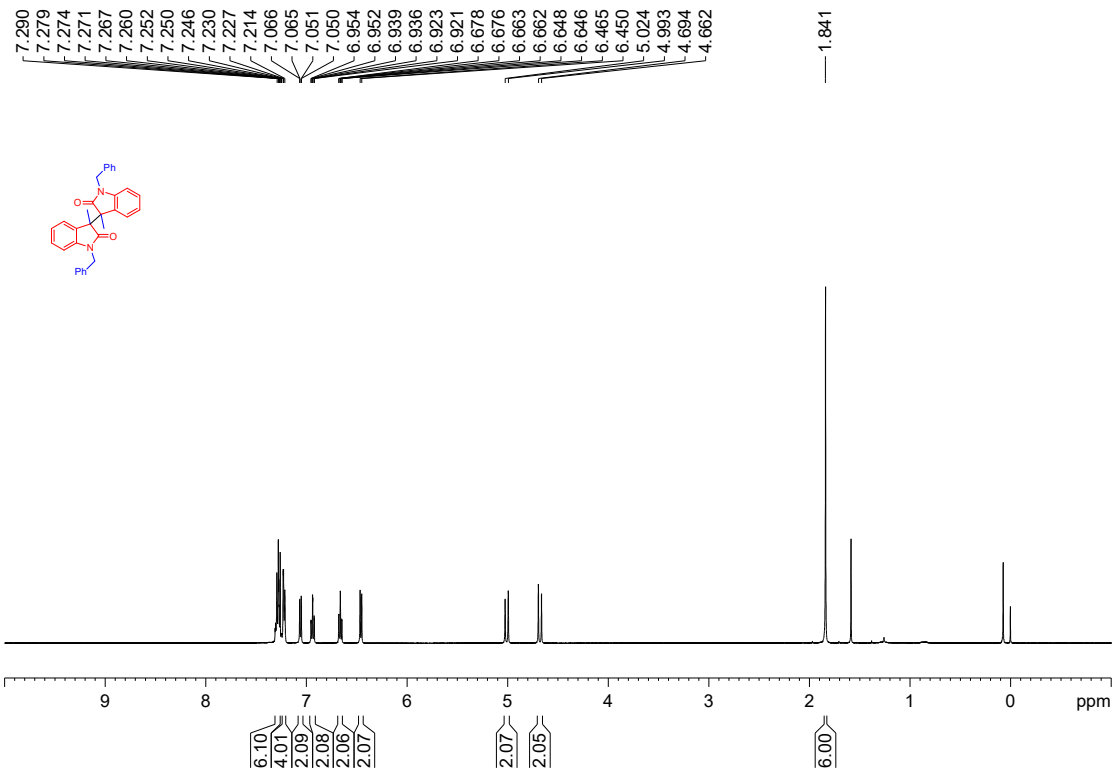
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2m**



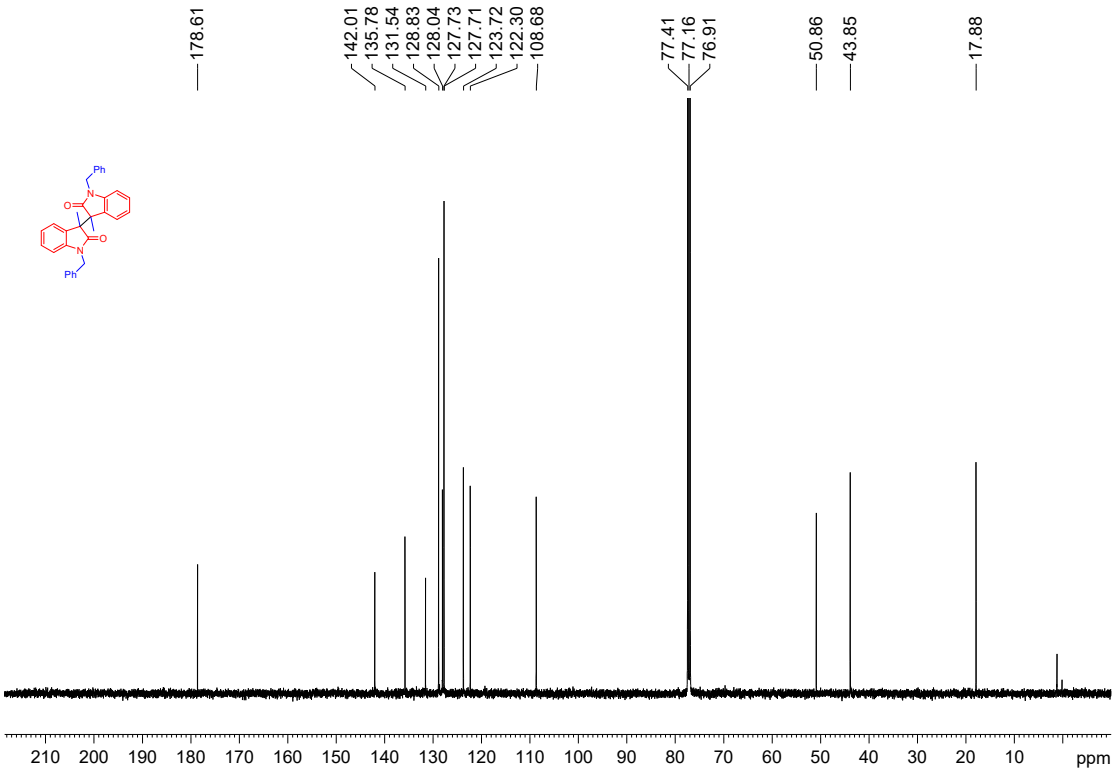
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2m**



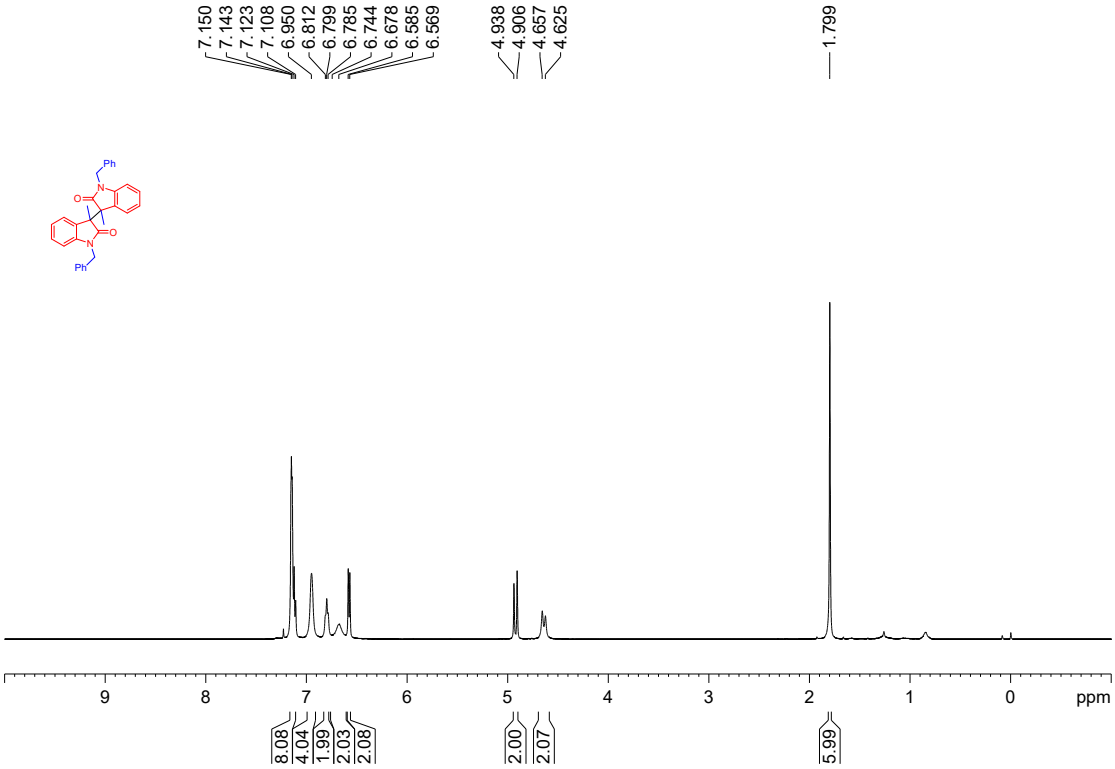
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2n**



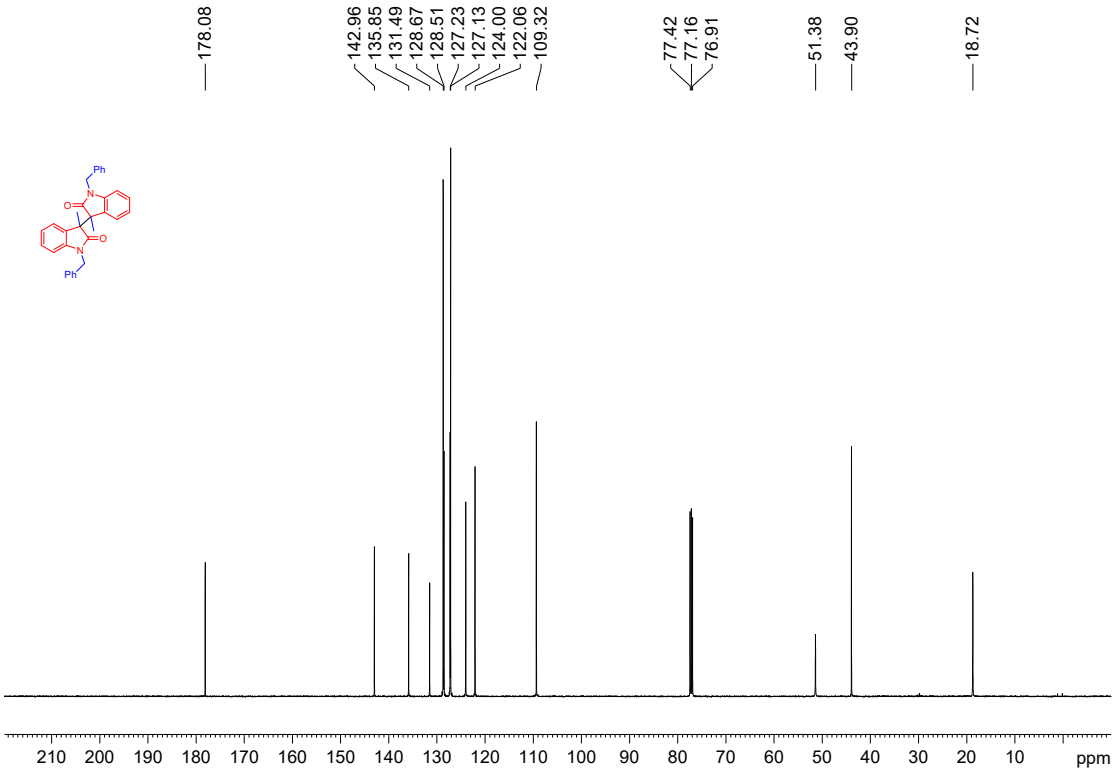
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2n**



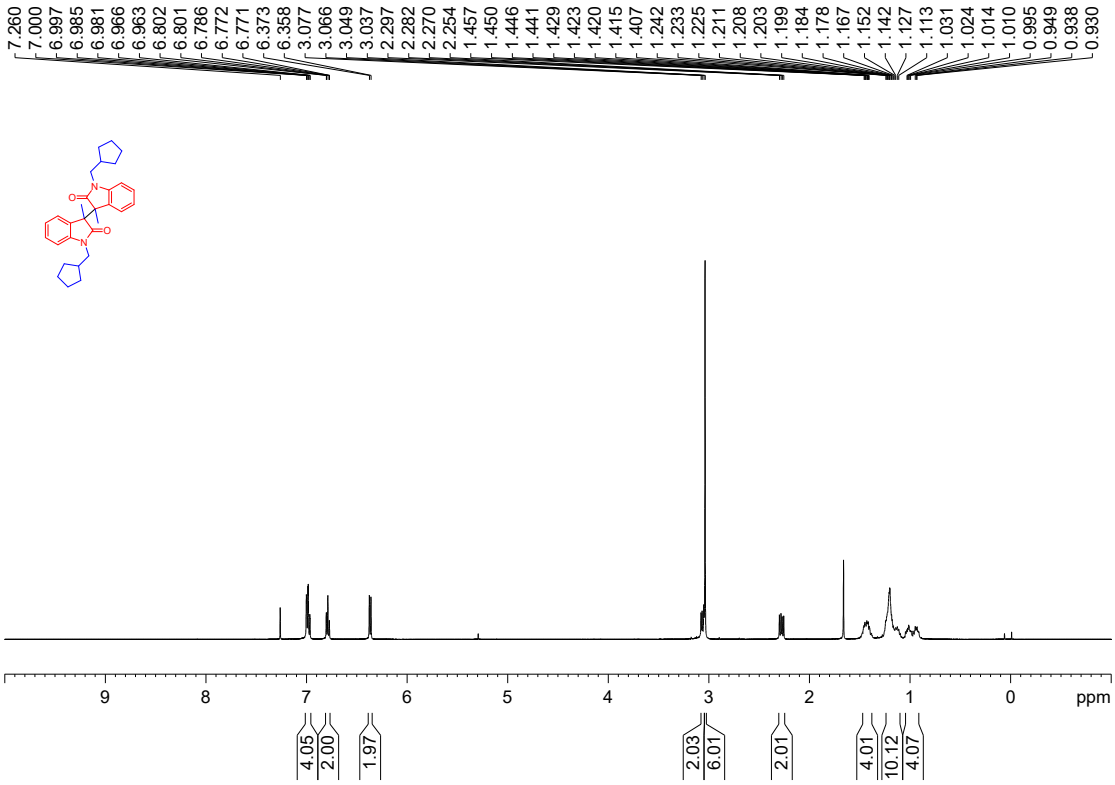
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2n**



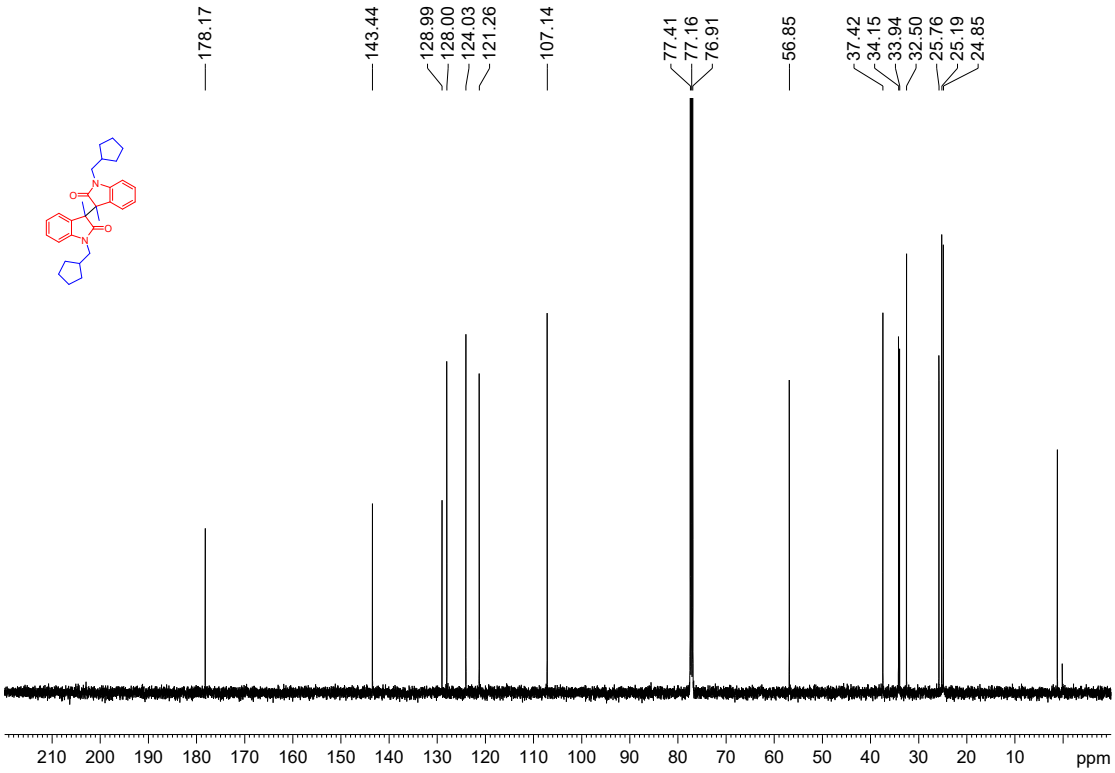
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2n**



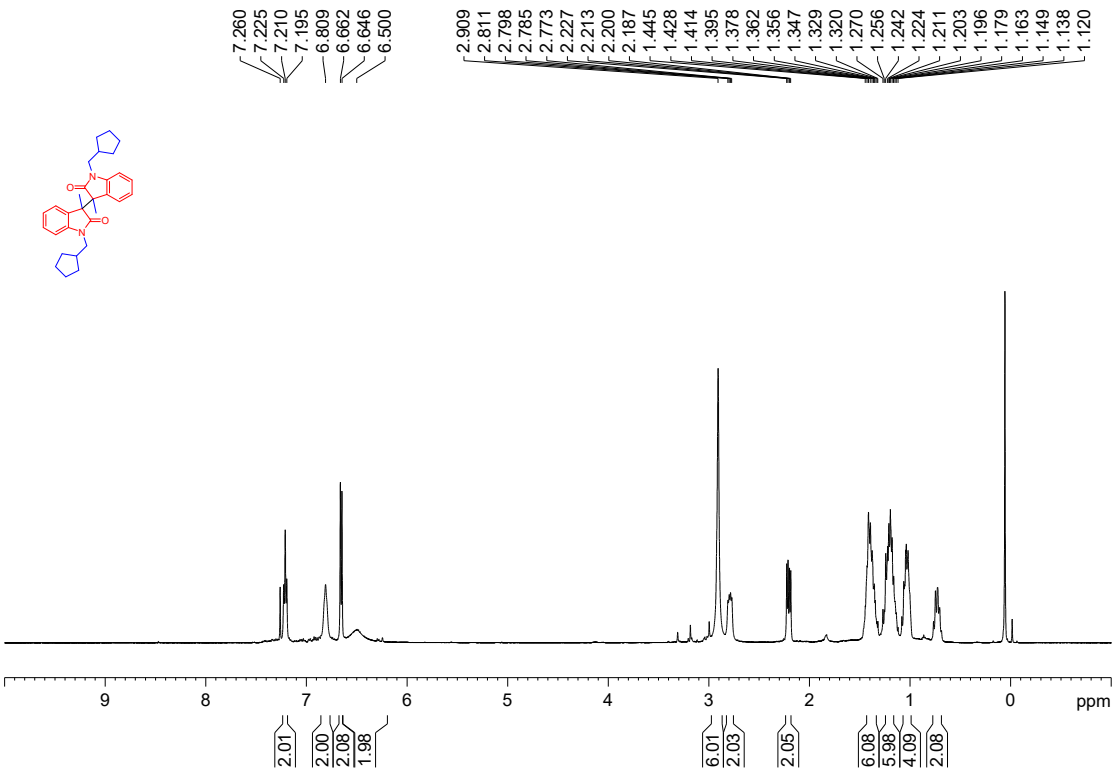
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2o**



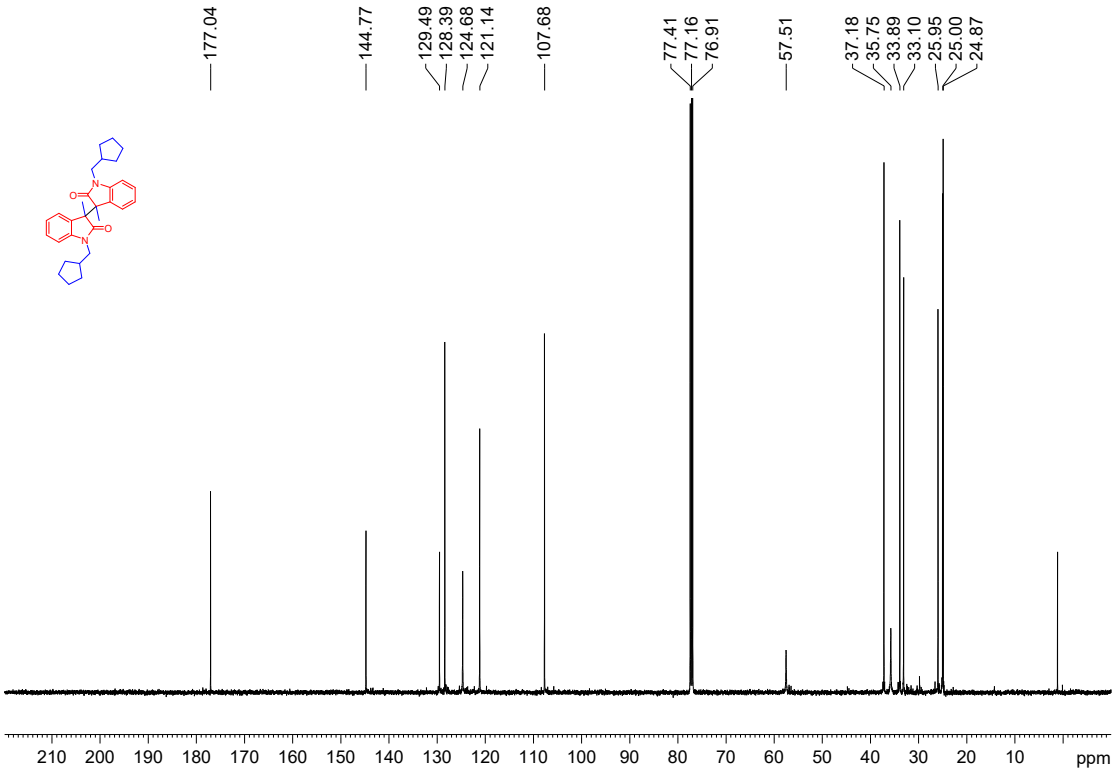
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2o**



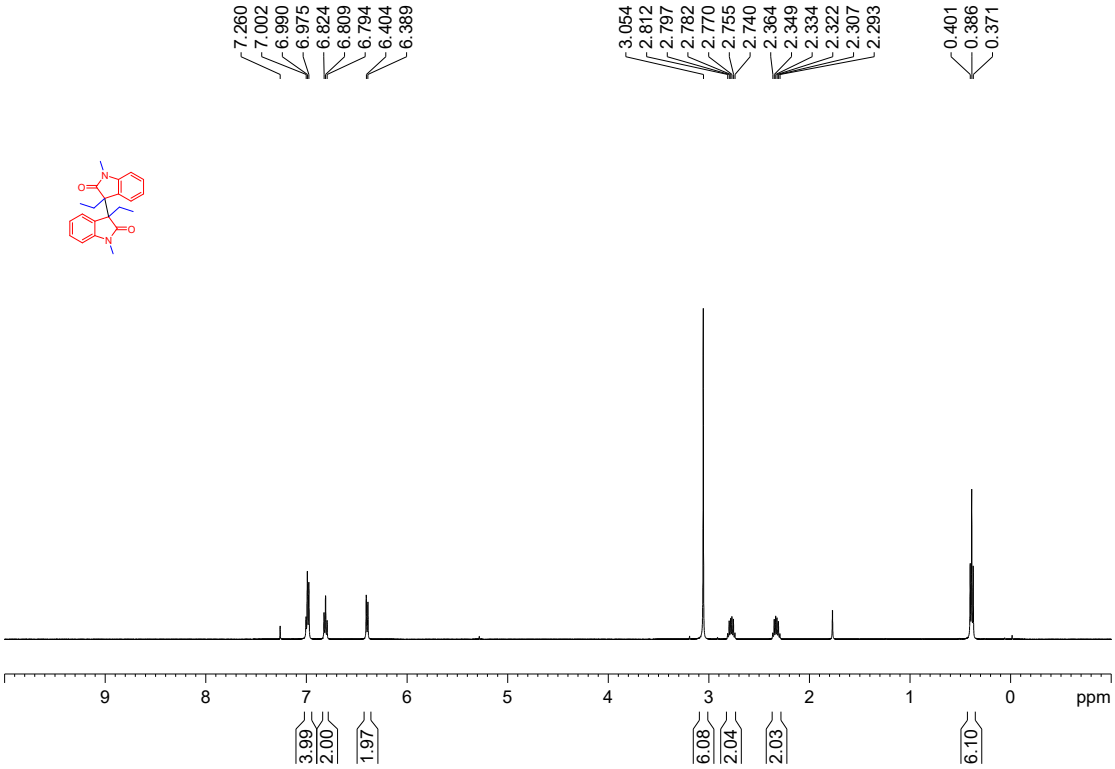
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2o**



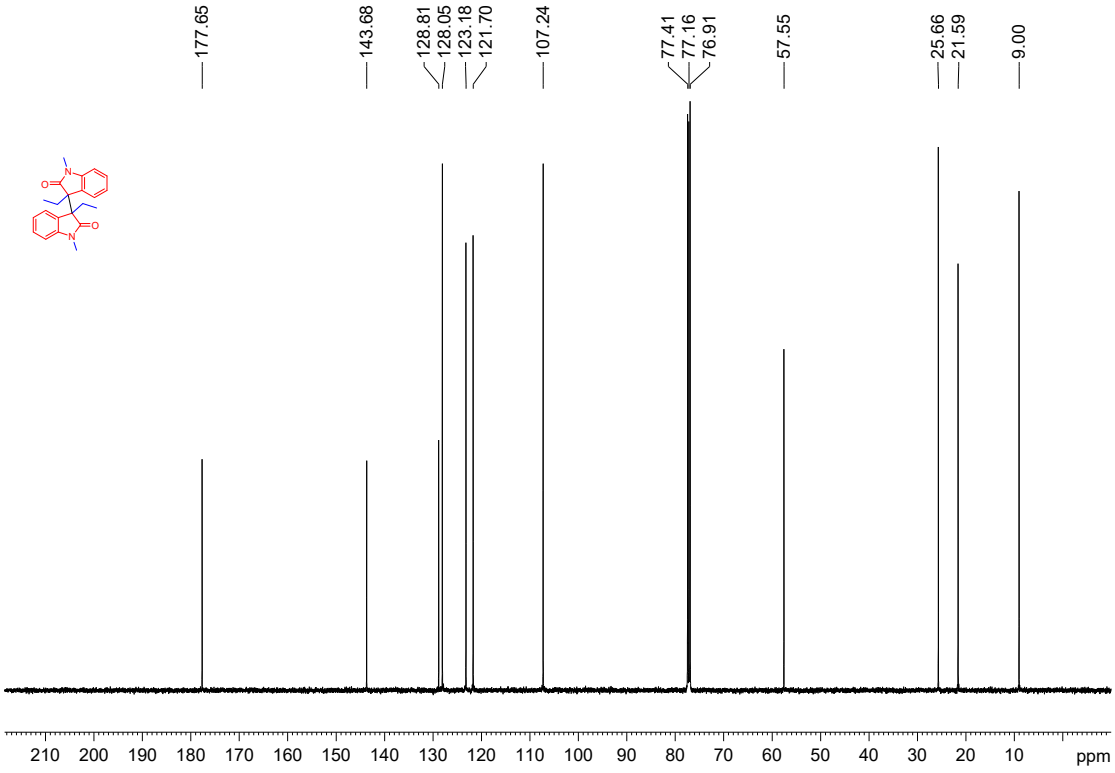
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2o**



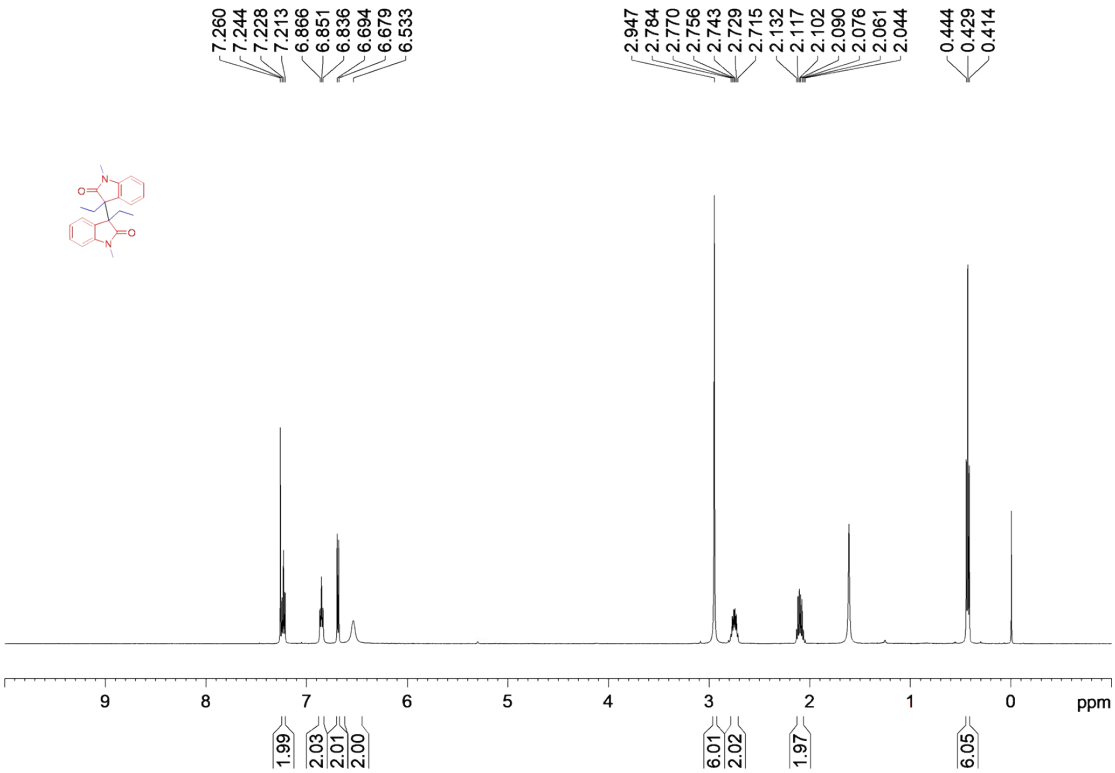
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2p**



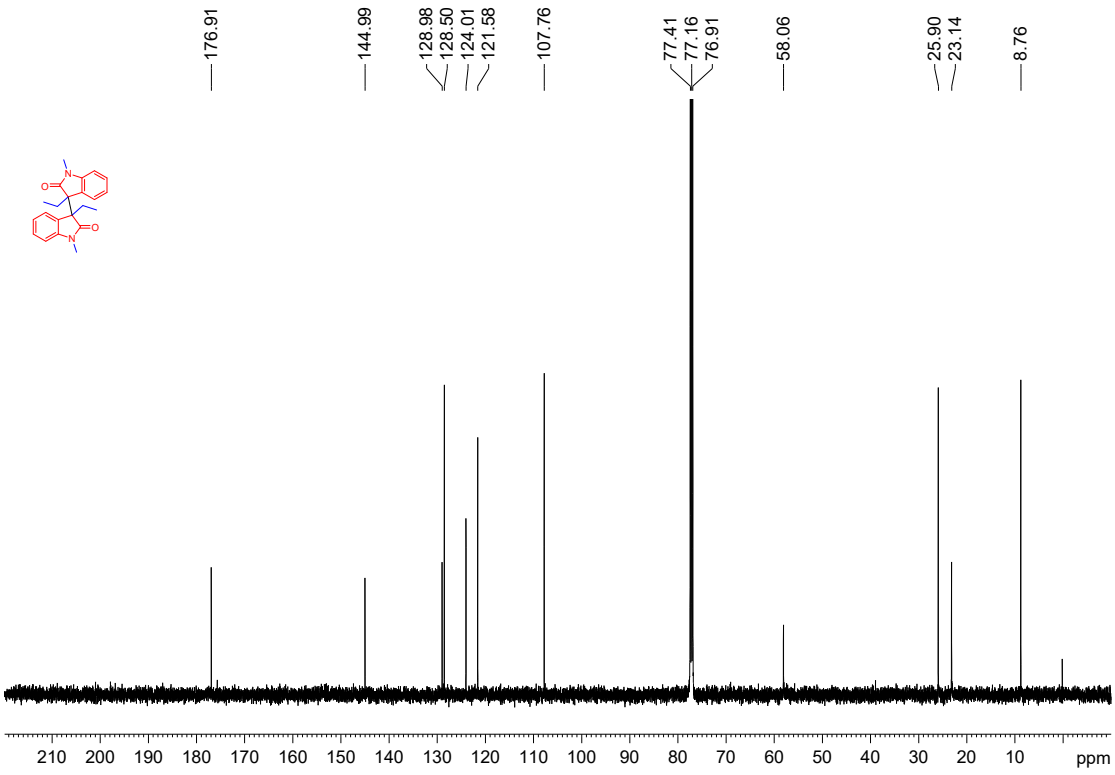
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2p**



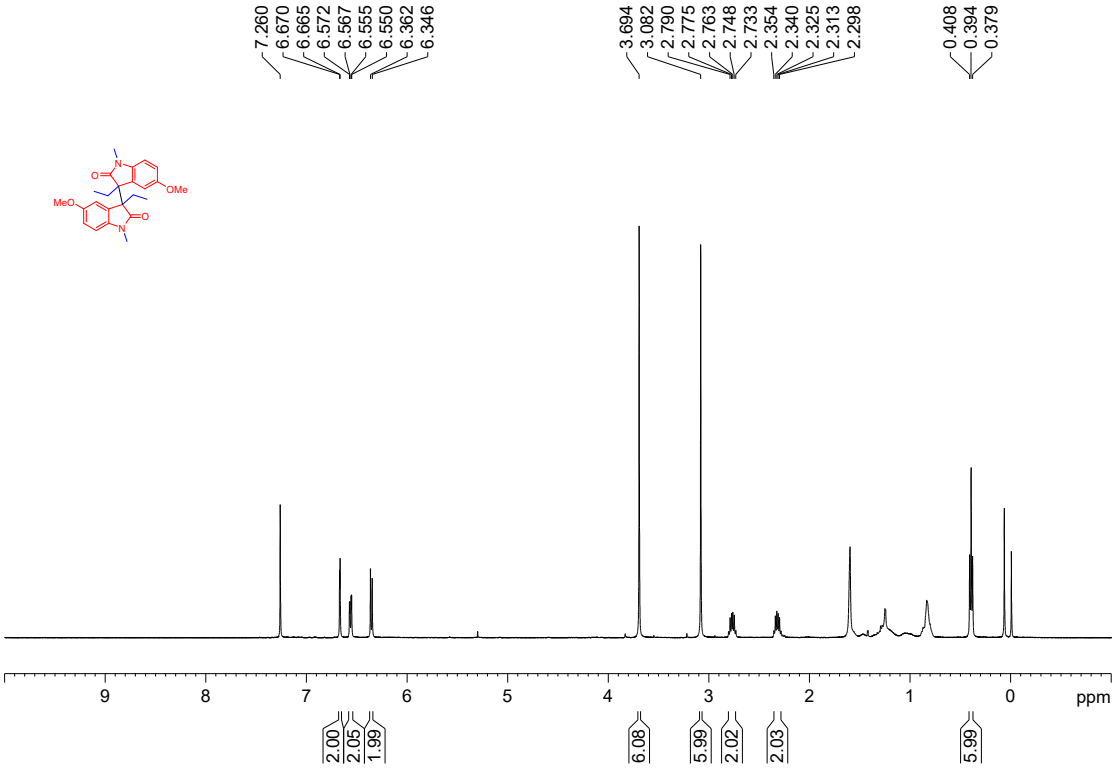
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2p**



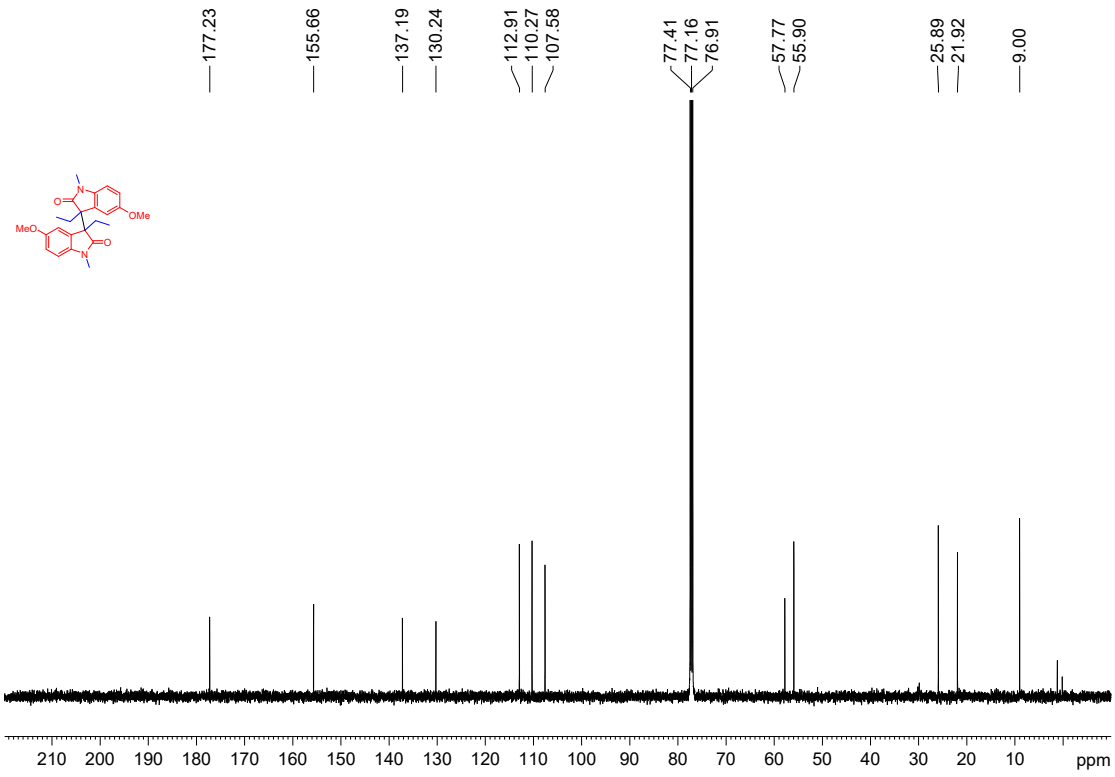
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2p**



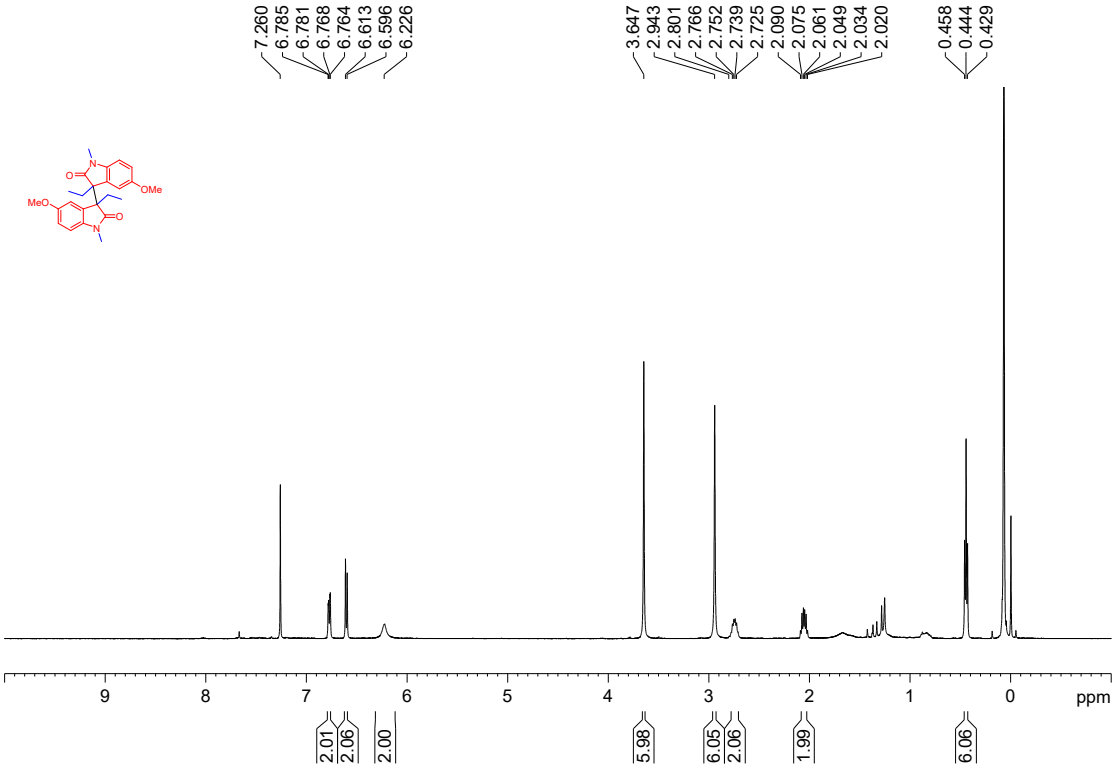
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2q**



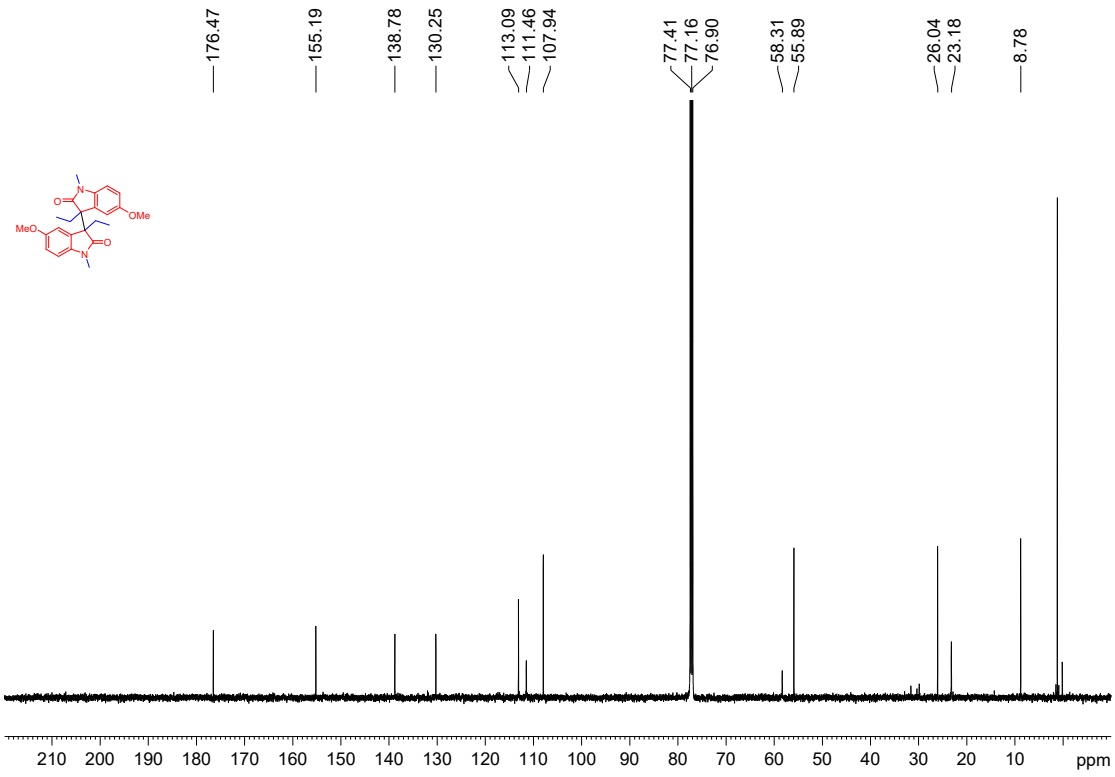
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2q**



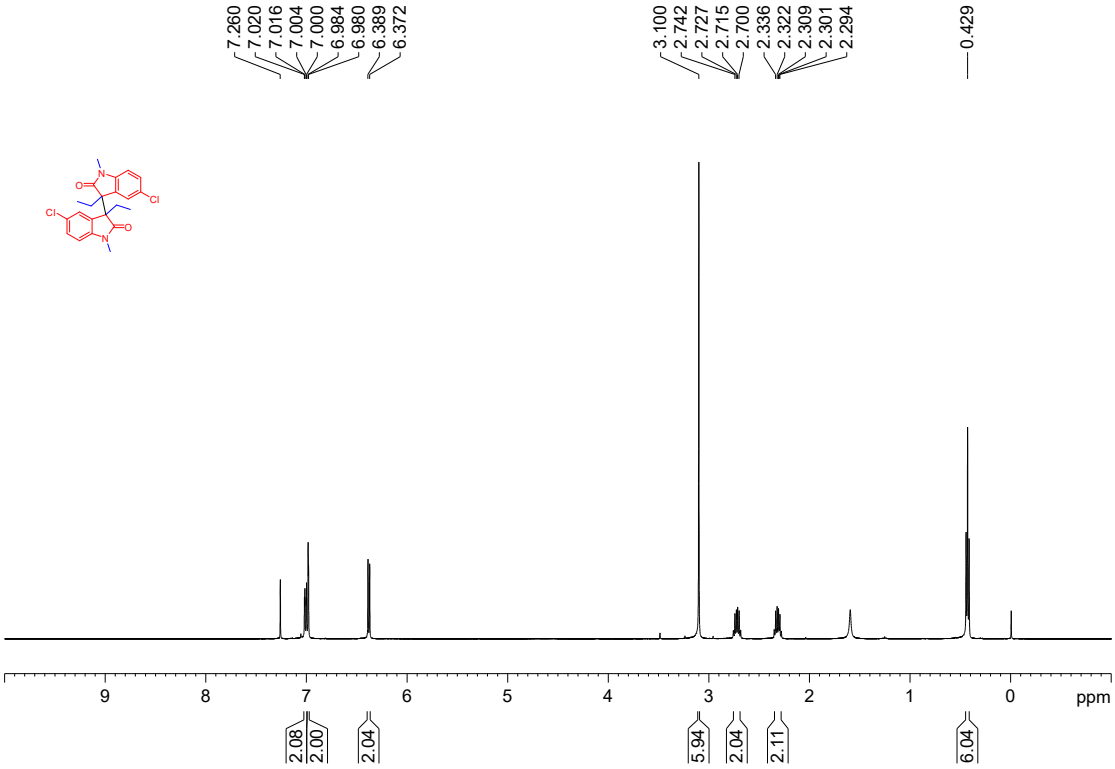
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2q**



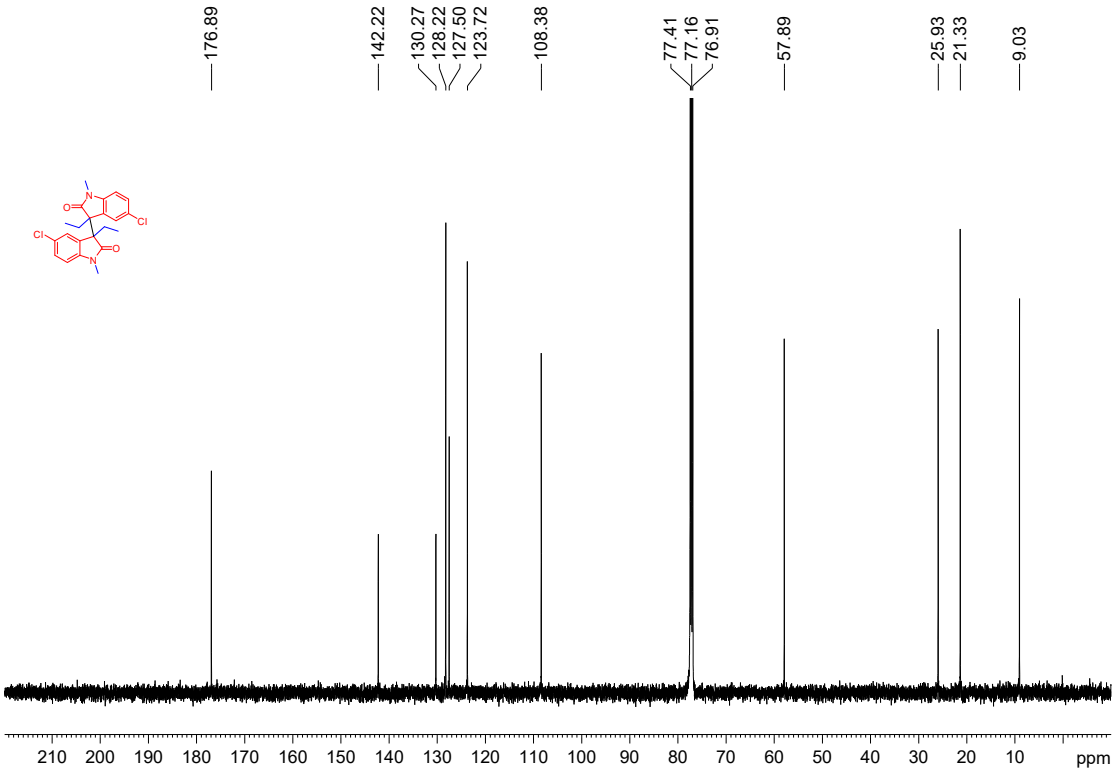
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2q**



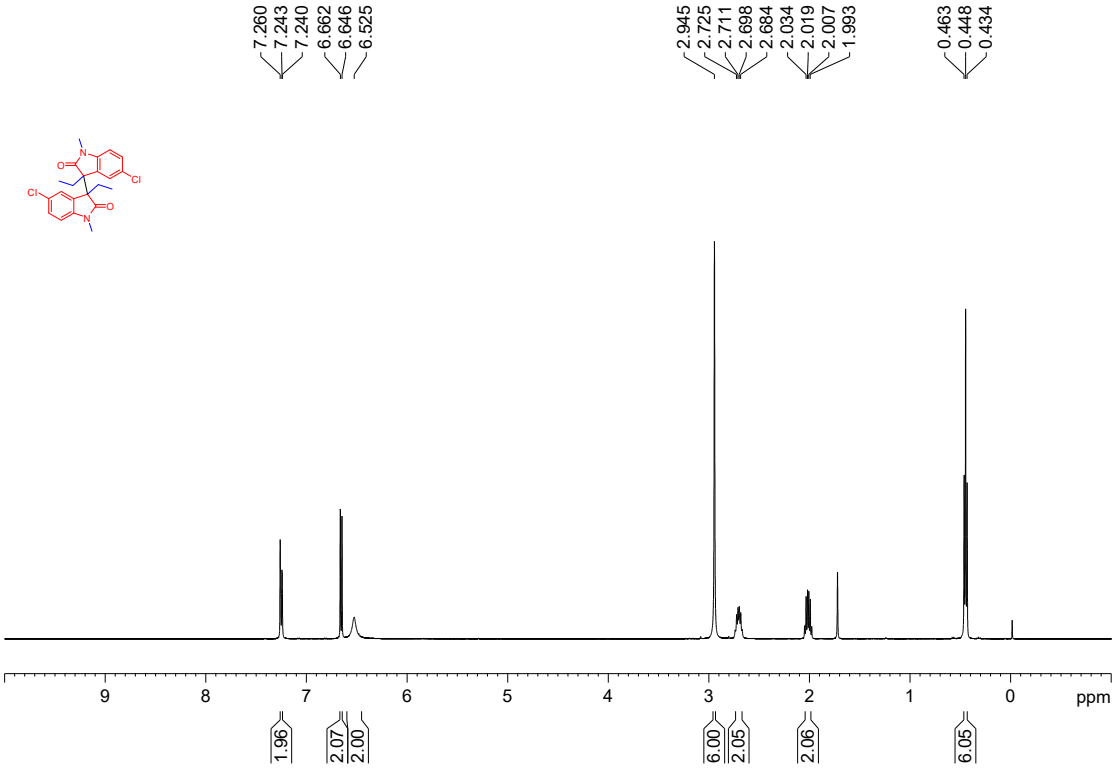
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2r**



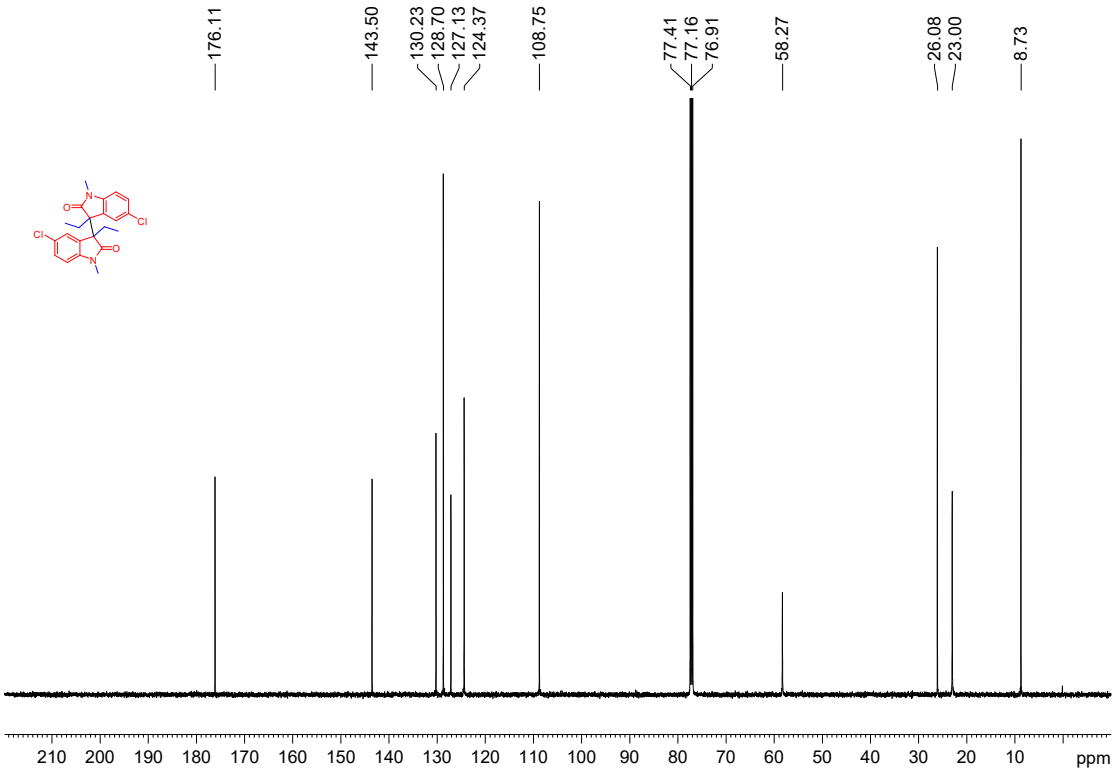
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2r**



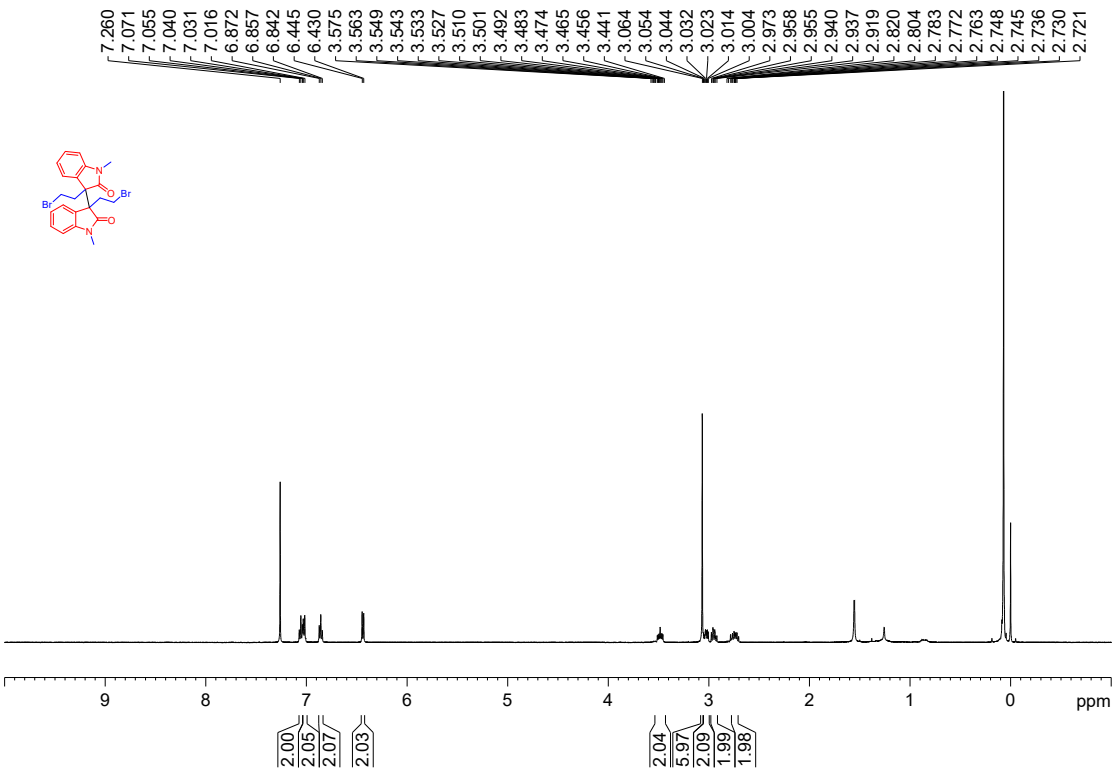
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **meso-2r**



^{13}C NMR (125 MHz, $CDCl_3$, 300K), **meso-2r**



^{1}H NMR (500 MHz, $CDCl_3$, 300K), **(±)-D,L-2s**



^{13}C NMR (125 MHz, $CDCl_3$, 300K), **(±)-D,L-2s**