Electronic Supplementary Material (ESI) for Chemical Communications.

Supplementary Information

Chemical resolution of spiroindanones and synthesis of chiroptical polymers with circularly polarized luminescence

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Table of content

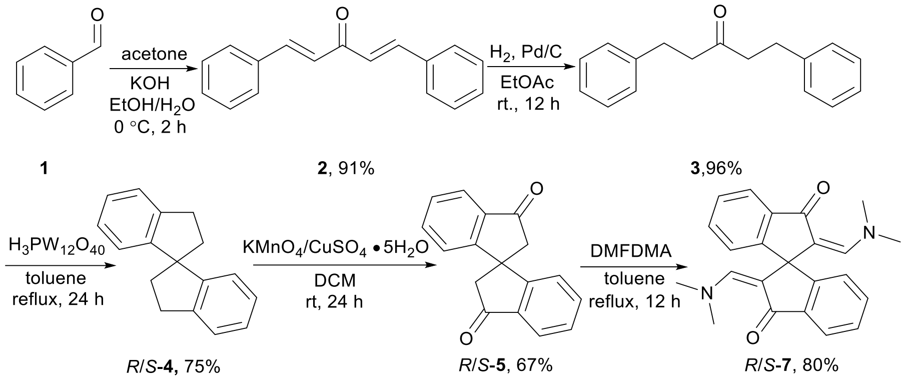
I. General Remarks

All reagents were obtained from commercial suppliers and used without further purification unless otherwise stated.

NMR spectra were recorded on an Agilent 400-MR DD2 spectrometer. The ^{1}H NMR (400 MHz) chemical shifts were measured relative to $CDCl_3$ or DMSO-d_6 as the internal reference (DMSO-d_6: δ = 2.50 ppm; $CDCl_3$: δ = 7.26 ppm). The ^{13}C NMR (100 MHz) chemical shifts were given using $CDCl_3$ or DMSO- d_6 as the internal standard (DMSO-d_6: δ = 39.52 ppm; $CDCl_3$: δ = 77.16 ppm). High-resolution mass spectra (HRMS) were obtained with a Shimadzu LCMS-ITTOF (ESI). The molecular weights of the polymers were tested by gel permeation chromatography (GPC) on Shimadzu LC-20AD with Shodex GPC KF-805L using polystyrene as standard and THF eluent at 40 °C. Nitrogen adsorption/desorption isotherms of the materials were measured using a ASAP 2460 surface area and porosimetry analyzer, at 77K. Enantiomeric excess was analyzed by Shimadzu LC-20AT with CHIRALCEL OD-H 5 μm 4.6 × 250 mm using hexane and Isopropyl alcohol ($V_{Hexane}/V_{i\text{-}PrOH}$ = 96/4, 1 mL/min) as the eluent at 30 °C. X-Ray single-crystal diffraction data were collected on a Bruker APEX-II CCD diffractometer. Absorption spectra were obtained on a HITACHI U-2910 spectrometer. Fluorescence spectra were collected on a Horiba Jobin Yvon-Edison Fluoromax-4 fluorescence spectrometer with a calibrated integrating sphere system. Phosphorescent spectra in solution at 77K were collected on a HITACHI F-7100 fluorescence spectrophotometer. Transient PL decay spectra were procured with Horiba Single Photon Counting Controller: FluoroHub and Horiba TBX Picosecond Photon Detection. Cyclic voltammogram were performed on LK2005A with a solution of tetrabutylammonium hexafluorophosphate (Bu_4NPF_6, 0.1 M) in DCM as electrolyte and ferrocene/ferrocenium (Fc/Fc^+) as standard. Three-electrode system (Ag/Ag^+, platinum wire, and glassy carbon electrode as reference, counter, and work electrode, respectively) was used in the CV measurement. Circular dichroism (CD) spectra were collected on JASCO J-1500 spectropolarimeter. Circularly polarized luminescence (CPL) spectra were recorded on OLIS CPL Solo.

II. Synthesis and Characterization

1. Synthesis of *R/S*-7 from benzaldehyde and acetone



Scheme S1. Synthetic procedures of *R/S*-**7**

Synthesis of **2**

A 100 mL flask equipped with a magnetic stir bar was charged with benzaldehyde (50 mmol) and KOH (11.2 g, 75 mmol) dissolved in EtOH (30 mL) at 0 ºC. The solution of acetone (1.9 mL, 25 mmol) in EtOH (10 mL) was added dropwise to the resulting mixture. Then the mixture was stirred at room temperature for 2 h. The precipitation was filtered and washed with water and cold EtOH to give **2** as pale-yellow powder in 91% yield (5.3 g) after dried under vacuum. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.75 (d, J =16.0 Hz, 2H), 7.64-7.61 (m, 4H), 7.43-7.41 (m, 6H), 7.09 (d, J =16.0 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 188.9, 143.4, 134.8, 130.5, 128.9, 128.4, 125.4 ppm. HRMS (ESI$^+$): calcd for $C_{17}H_{14}O$ $[M+H]^+$, 235.1117; found 235.1114.

Synthesis of **3**

A 100 mL flask equipped with a magnetic stir bar was charged with Pd/C (200 mg, 5 *w*t %), **2** (936 mg, 4 mmol) and EtOAc (30 mL). The mixture was stirred at room temperature for 12 h with an atmosphere of H_2 maintained by an inflated balloon. The mixture was filtered by diatomite and the filtrate was evaporated to give **3** as a colorless liquid in 96% yield (920 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.28 (t, J =8.0 Hz, 4H), 7.21-7.15 (m, 6H), 2.89 (t, J = 7.6 Hz, 4H), 2.72 (t, J =7.6 Hz, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 209.2, 141.0, 128.5, 128.3, 126.1, 44.5, 29.7. ppm. HRMS (ESI$^+$): calcd for $C_{17}H_{18}O$ $[M+H]^+$,239.1430; found 239.1432.

Synthesis of *R/S*-**4**

A 100 mL flask equipped with water segregator and reflux condenser was charged with **3** (2.4 g, 10 mmol), $H_3PW_{12}O_{40}$ (1.5 mmol) and toluene (40 mL). Then the mixture was refluxed until no water was separated. The mixture was filtered after cooled to room temperature. Then the filtrate was concentrated and purified on column chromatography on silica gel (PE as eluent) to give the product *R*/*S*-**4** as a colorless liquid in 75% yield (1.65 g). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.28-7.11 (m, 6H), 6.92 (d, *J* = 7.6 Hz, 2H), 3.01 (t, *J* = 8.0 Hz, 3H), 2.33-2.14 (m, 5H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.4, 143.7, 126.7, 126.6, 124.3, 123.4, 60.7, 40.5, 30.9 ppm. HRMS (ESI^+): calcd for $C_{17}H_{16}$ $[M+H]^+$, 221.1325; found 221.1327.

Synthesis of *R*/*S*-**5**

A 50 mL flask equipped with a magnetic stir bar was charged with *R/S*-**4** (0.5 mmol.), oxidant (2 g) (The oxidant was prepared by grinding equal amounts of potassium permanganate and copper sulfate pentahydrate in a mortar) and CH_2Cl_2 (20 mL), which was stirred at room temperature for 24 h. Then the mixture was filtered by diatomite and washed with CH_2Cl_2. Then the filtrate was concentrated and purified on column chromatography on silica gel (PE/EA = 3/1, v/v) to give *R*/*S*-**5** as a white solid in 67% yield (83mg). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.83 (d, *J* = 7.6 Hz, 2H), 7.59 (t, *J* = 7.6 Hz, 2H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.08 (d, *J* = 8.0 Hz, 2H), 3.13 (dd, J_1 = 30.4, J_2 = 18.8, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = δ 204.2, 159.8, 136.1, 135.6, 128.5, 124.4, 123.5, 53.0, 47.9 ppm. HRMS (ESI^+): calcd for $C_{17}H_{12}O_2$ $[M+H]^+$, 249.0910; found 249.0911.

Synthesis of *R*/*S*-**7**

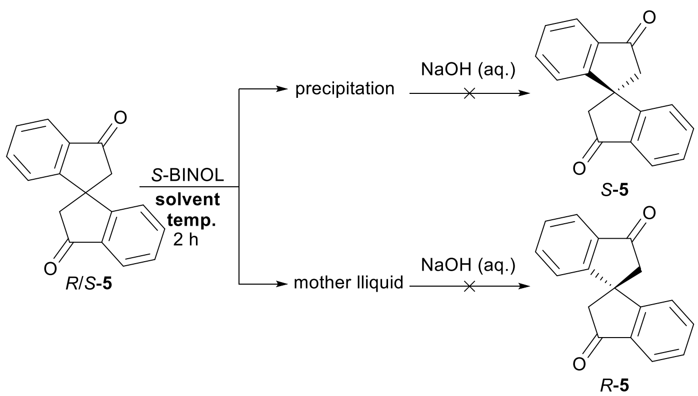
A 25 mL Schlenk tube equipped with a magnetic stir bar was charged with *R*/*S*-**5** (0.24 mmol, 60mg), DMFDMA (*N,N*-Dimethylformamide dimethyl acetal) (180 μL, 1.2 mmol, 5 equiv.) and toluene (2 mL). The mixture was stirred at 120 °C for 12 h. After cooling to room temperature, solvent was removed by rotary evaporator and *R*/*S*-**7** was obtained by column chromatography on silica gel (EA/MeOH = 10/1, v/v) in 80% yield (69 mg). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.79 (t, *J* = 4.0 Hz, 2H), 7.57 (s, 2H), 7.31 (t, *J* = 4.0 Hz, 4H), 6.90 (s, 2H), 2.77 (s, 12H) ppm. ^{13}C NMR

(100 MHz, $CDCl_3$): δ = 193.1, 156.3, 148.3, 136.9, 133.1, 127.4, 123.7, 123.0, 112.1, 55.0, 43.4 ppm. HRMS (ESI$^+$): calcd for $C_{23}H_{23}N_2O_2$ [M+H]$^+$, 359.1754; found 359.1754.

2. Chemical resolution of *R/S*-5 and *R/S*-7

(1) Chemical resolution of *R/S*-5 with *S*-BINOL

Table S1. Chemical resolution of *R/S*-**5** with *S*-BINOL[a]

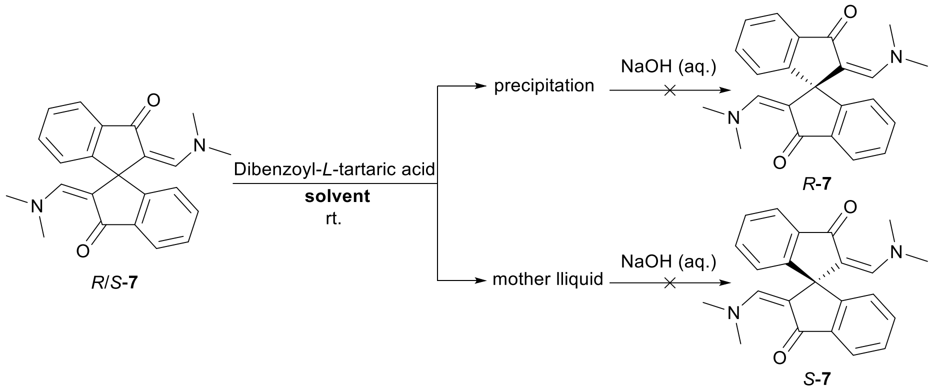


Entry	Solvent	Conc. (mol L^{-1})	Temperature (°C)	Ratio (*R/S*-**5** : *S*-BINOL)	Precipitation *S*-**5** ee (%)	Mother liquid *R*-**5** ee (%)
1	toluene	0.2	25	1.0 : 0.60	--	--
2	toluene	0.2	0	1.0 : 0.60	--	--
3	toluene	0.2	-15	1.0 : 0.60	--	--
4	toluene	0.2	-25	1.0 : 0.60	--	--
5	DCM	0.2	25	1.0 : 0.60	no precipitation	--
6	toluene : DCM (1 : 1)	0.2	25	1.0 : 0.60	--	--
7	toluene	0.1	25	1.0 : 0.60	--	--

[a] Reaction condition: *R/S*-**5** (1 mmol), *S*-BINOL (0.6 mmol), solvent (5 mL), stirred for 2 h.

(2) Chemical resolution of *R/S*-7 with dibenzoyl-*L*-tartaric acid

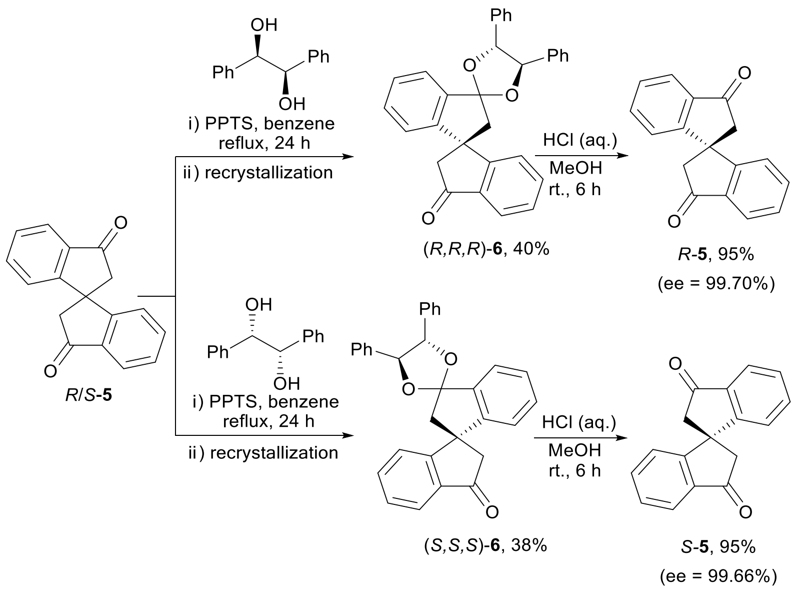
Table S2. Chemical resolution of *R/S*-**7** with dibenzoyl-*L*-tartaric acid[a]



Entry	solvent	Conc. (mol L^{-1})	Ratio (*R/S*-**7** : Dibenzoyl-*L*-tartaric acid)	Precipitation *R*-**7** ee (%)	Mother liquid *S*-**7** ee (%)
1	MeOH	0.2	1.0 : 0.50	no precipitation	--
2	EtOAc	0.2	1.0 : 0.50	no precipitation	--
3	MeCN	0.2	1.0 : 0.50	--	--
4	Toluene	0.2	1.0 : 0.50	--	--
5	Toluene : DCM (1 : 1)	0.2	1.0 : 0.50	--	--
6	MeOH : MeCN (1 : 1)	0.2	1.0 : 0.50	no precipitation	--
7	MeOH : DCM (1 : 1)	0.2	1.0 : 0.50	no precipitation	--
8	MeOH : Acetone (1 : 1)	0.2	1.0 : 0.50	no precipitation	--

[a] Reaction condition: *R/S*-**7** (1 mmol), dibenzoyl-*L*-tartaric acid (0.5 mmol), solvent (5 mL), stirred at room temperature for 2 h.

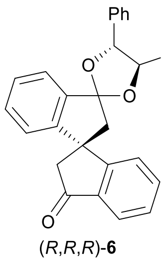
(3) Chemical resolution of *R/S*-5 with 1,2-diphenylethane-1,2-diol



Scheme S2. Chemical resolution of *R*/*S*-**5** with 1,2-diphenylethane-1,2-diol

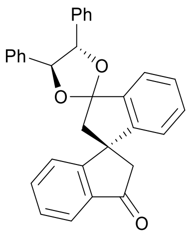
General procedure for the synthesis of (*R*,*R*,*R*-6) and (*S*,*S*,*S*-6)

A 100 mL flask equipped with water segregator and reflux condenser was charged with *R/S*-**5** (1.24 g, 5 mmol), pyridinium *para*-toluenesulfonate (251 mg, 1 mmol), (1*R*,2*R*)-1,2-diphenylethane-1,2-diol (2.14 g, 10 mmol) and Benzene (25 mL). Then the mixture was refluxed and dehydrated for 24 h. After cooling to temperature, solvent was removed by rotary evaporator and a white solid was obtained by column chromatography on silica gel (PE/EA=10/1, v/v), which was recrystallized with hexane twice to give the product.



(*R*,*R*,*R*)-**6**

(*R*,*R*,*R*-**6**) was synthesized from *R*/*S*-**5** in 40% yield (888 mg) using (1*R*,2*R*)-1,2-diphenylethane-1,2-diol as the chiral resolution agent. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.82 (d, J = 7.2 Hz, 1H), 7.74-7.68 (m, 2H), 7.52 (t, J = 7.2, 1H), 7.47-7.30 (m, 13H), 6.85 (d, J = 7.2 Hz, 1H), 5.25 (d, J = 8.8 Hz, 1H), 4.99 (d, J = 8.8 Hz, 1H), 3.16-2.83 (m, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 204.7, 160.6, 149.6, 142.0, 137.1, 136.3,136.1, 131.5, 129.0, 128.9, 127.7, 127.3, 125.8, 124.2, 123.5, 122.9, 116.2, 85.9, 85.4. 54.3, 53.3, 50.9 ppm. HRMS (ESI$^+$): calcd for $C_{31}H_{24}O_3$ $[M+Na]^+$, 467.1618; found 467.1626.

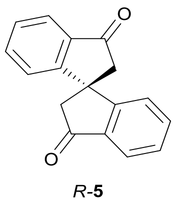


(*S*,*S*,*S*)-**6**

(*S,S,S*-**6**) was synthesized from *R*/*S*-**5** in 38% yield (844 mg) using (1*S*,2*S*)-1,2-diphenylethane-1,2-diol as the chiral resolution agent. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.82 (d, J = 6.8 Hz, 1H), 7.74-7.68 (m, 2H), 7.52 (t, J = 6.8, 1H), 7.46-7.32 (m, 13H), 6.85 (d, J = 6.8 Hz, 1H), 5.25 (d, J = 8.4 Hz, 1H), 4.99 (d, J = 8.0 Hz, 1H), 3.16-2.83 (m, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 204.7, 160.5, 149.6, 142.0, 137.1, 136.3, 136.1, 131.5, 128.9, 128.8, 127.7, 127.3, 125.8, 124.1, 123.5, 122.9, 116.2, 85.9, 85.4, 54.3, 53.3, 50.9 ppm. HRMS (ESI$^+$): calcd for $C_{31}H_{24}O_3$ $[M+Na]^+$, 467.1618; found 467.1618.

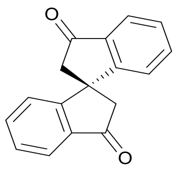
General procedure for the synthesis of *R*-5 and *S*-5

(*R*,*R*,*R*)-**6** was added to the solution of HCl (2.5 M) in methanol and stirred for 6 h. The solution was poured into water after hydrolysis was complete. The mixture was extracted with water/dichloromethane and the organic phase was dried with anhydrous Na_2SO_4. The organic phase was concentrated and purified by column chromatography (PE/EA = 3/1, v/v) to give the product as a white solid.



R-**5**

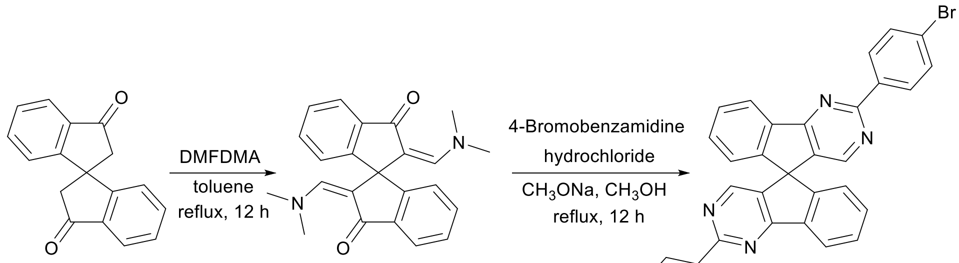
R-**5** was synthesized from (*R*,*R*,*R*)-**6** (1 mmol) in 95% yield (235 mg). The enantiomeric excess (e.e.) of *R*-**5** was 99.70% which was detected by HPLC. Absolute configuration was determined by X-ray single crystal data (Flack parameter = 0.01). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82 (d, J = 8.0 Hz, 2H), 7.58 (t, J = 7.6 Hz, 2H), 7.45 (t, J = 7.6 Hz, 2H), 7.08 (d, J = 7.6 Hz, 2H), 3.13 (dd, J_1 = 30.4, J_2 = 18.8, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 204.1, 159.8, 136.1, 135.6, 128.5, 124.4, 123.5, 53.0, 47.9 ppm. HRMS (ESI$^+$): calcd for $C_{17}H_{12}O_2$ [M+H]$^+$, 249.0910; found 249.0910.



S-**5**

S-**5** was synthesized from (*S*,*S*,*S*)**-6** (1 mmol) in 95% yield (234 mg). The enantiomeric excess (e.e.) of *S*-**5** was 99.66% which was detected by HPLC. Absolute configuration was determined by X-ray single crystal data (Flack parameter = 0.04). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82 (d, J = 7.6 Hz, 2H), 7.58 (t, J = 7.6 Hz, 2H), 7.45 (t, J = 7.6 Hz, 2H), 7.08 (d, J = 8.0 Hz, 2H), 3.13 (dd, J_1 = 30.0, J_2 = 19.2, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 204.1, 159.8, 136.1, 135.6, 128.5, 124.4, 123.5, 53.0, 47.9 ppm. HRMS (ESI$^+$): calcd for $C_{17}H_{12}O_2$ [M+H]$^+$, 249.0910; found 249.0906.

3. Synthesis of *R/S*-, *R*- and *S*-8 from *R/S*-, *R*- and *S*-5



R/S-**5**
R-**5**
S-**5**

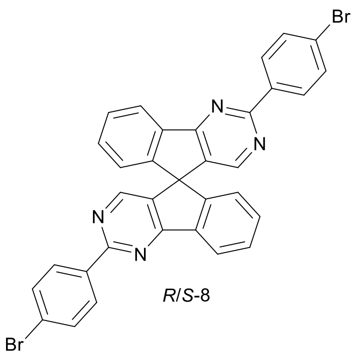
R/S-**7**
R-**7**
S-**7**
without purification

R/S-**8,** 76%
R-**8,** 78%
S-**8,** 72%
(yield of two steps)

Scheme S3. Synthetic procedures of *R/S*-, *R*- and *S*-**8**

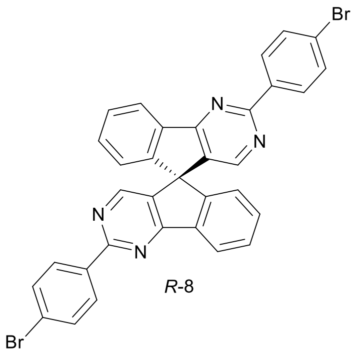
General procedure for the synthesis of *R*/*S*-, *R*- and *S*-8

A 25 mL Schlenk tube equipped with a magnetic stir bar was charged with *R*/*S*-**5** (0.24 mmol, 60mg), DMFDMA (*N,N*-Dimethylformamide dimethyl acetal) (180 μL, 1.2 mmol, 5 equiv.) and toluene (2 mL). The mixture was stirred at 120 °C for 12 h. After cooling to room temperature, the crude product of *R*/*S*-**7** was obtained by removing the solvent under reduced pressure without purification. Subsequently, 4-Bromobenzamidine hydrochloride (188 mg, 0.8 mmol) and sodium methoxide (44 mg, 0.8 mmol) were added into the 25 mL Schlenk tube filled with the crude product of *R*/*S*-**7.** And methanol (2 mL) was added. Then the mixture was stirred at 100 °C for 12 h. After cooling to room temperature, the mixture was extracted with water/dichloromethane and the organic phase was dried with anhydrous Na_2SO_4. *R*/*S*-**8** was obtained by column chromatography on silica gel (PE/EA = 10/1, v/v).



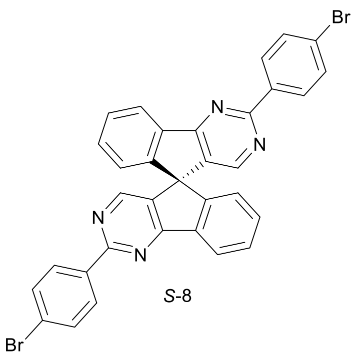
R/*S*-8

R/*S*-**8** was synthesized from *R*/*S*-**5** (0.24 mmol) in 76% yield (115 mg) of two steps. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.48 (d, *J* = 8.0 Hz, 2H), 8.34 (d, *J* = 7.6 Hz, 1H), 8.20 (s, 1H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 8.0 Hz, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.8, 164.3, 151.8, 148.5, 138.8, 136.6, 135.5, 132.6, 131.9, 130.0, 129.3, 125.7, 124.1, 122.9, 59.4 ppm. HRMS (ESI$^+$): calcd for $C_{33}H_{18}{}^{79}Br^{79}BrN_4$ [M+H]$^+$ 628.9971, found 628.9974; calcd for $C_{33}H_{18}{}^{81}Br^{79}BrN_4$ [M+H]$^+$, 630.9951; found 630.9950, calcd for $C_{33}H_{18}{}^{81}Br^{81}BrN_4$ [M+H]$^+$, 632.9930; found 632.9934.



R-8

***R*-8** was synthesized from ***R*-5** (0.24 mmol) in 78% yield (118 mg) of two steps. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.48 (d, J = 8.4 Hz, 2H), 8.34 (d, J = 7.6 Hz, 1H), 8.20 (s, 1H), 7.66 (d, J = 8.4 Hz, 2H), 7.58 (t, J = 7.2 Hz, 1H), 7.41 (t, J = 7.6 Hz, 1H), 6.86 (d, J = 7.6 Hz, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.7, 164.2, 151.7, 148.5, 138.7, 136.5, 135.4, 132.6, 131.8, 129.9, 129.3, 125.7, 124.0, 122.9, 59.4 ppm. HRMS (ESI$^+$): calcd for $C_{33}H_{18}{}^{79}Br^{79}BrN_4$ $[M+H]^+$ 628.9971, found 628.9969; calcd for $C_{33}H_{18}{}^{81}Br^{79}BrN_4$ $[M+H]^+$, 630.9951; found 630.9952, calcd for $C_{33}H_{18}{}^{81}Br^{81}BrN_4$ $[M+H]^+$, 632.9930; found 632.9929.



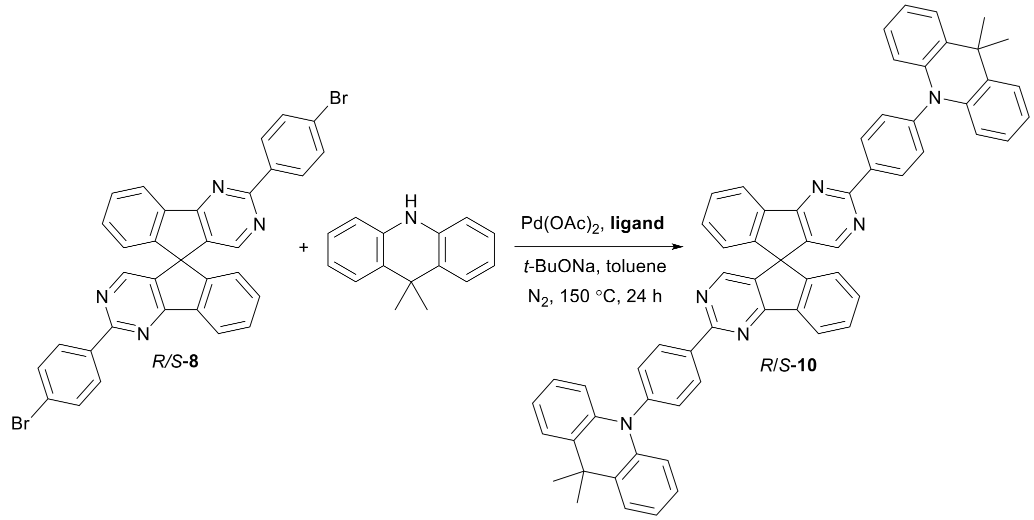
S-8

***S*-8** was synthesized from ***S*-5** (0.24 mmol) in 72% yield (108 mg) of two steps. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.48 (d, J = 8.8 Hz, 2H), 8.34 (d, J = 7.6 Hz, 1H), 8.20 (s, 1H), 7.66 (d, J = 8.8 Hz, 2H), 7.59 (t, J = 7.2 Hz, 1H), 7.41 (t, J = 8.0 Hz, 1H), 6.86 (d, J = 7.6 Hz, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.7, 164.3, 151.7, 148.5, 138.8, 136.6, 135.5, 132.6, 131.8, 130.0, 129.3, 125.7, 124.1, 122.9, 59.4 ppm. HRMS (ESI$^+$): calcd for $C_{33}H_{18}{}^{79}Br^{79}BrN_4$ $[M+H]^+$ 628.9971, found 628.9971; calcd for $C_{33}H_{18}{}^{81}Br^{79}BrN_4$ $[M+H]^+$, 630.9951; found 630.9954, calcd for $C_{33}H_{18}{}^{81}Br^{81}BrN_4$ $[M+H]^+$, 632.9930; found 632.9935.

4. Optimization of Buchwald–Hartwig amination and polymerization reaction conditions

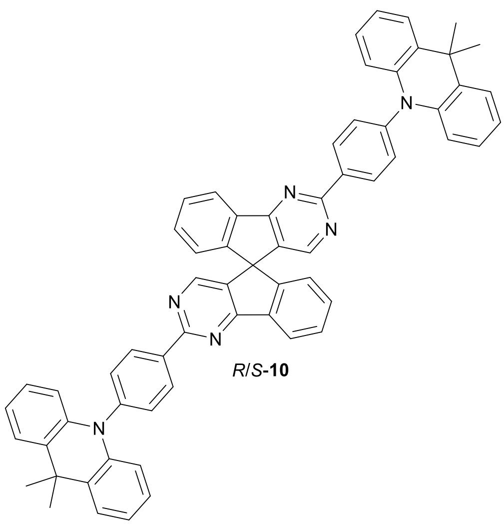
(1) Optimization of Buchwald–Hartwig amination reaction conditions of *R*/*S*-8 with 9,9-dimethyl-9,10-dihydroacridine

Table S3. Optimization of Buchwald–Hartwig amination reaction conditions[a]



Entry	Phosphine ligand	yield (%)[b]
1	PPh_3	86
2	PCy_3	87
3	P(*p*-PhMe)$_3$	89
4	P(*p*-PhCl)$_3$	90
5	P(*p*-PhCF_3)$_3$	63
6	P(*p*-PhOMe)$_2$Ph	54
7	JohnPhos	41
8	P(*p*-PhOMe)$_3$	89
9	RuPhos	99 (88[c])
10	Rac-BINAP	74
11	DPPF	92
12	XantPhos	92
13	DPE-Phos	96
14	DPPB	97

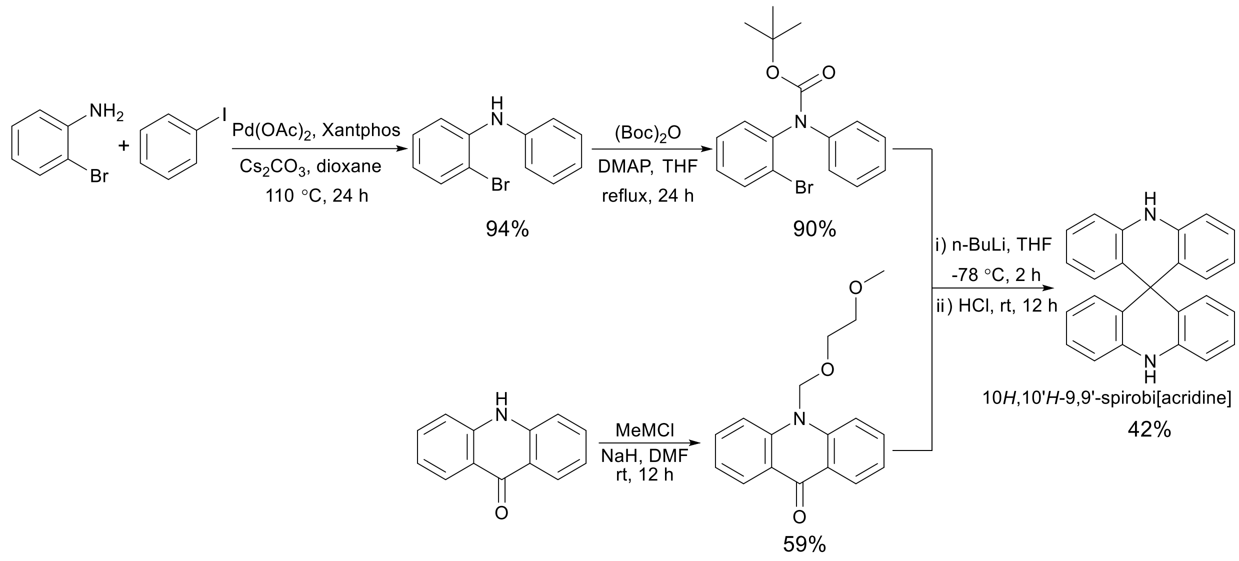
[a]Reaction conditions: *R*/*S*-**8** (0.05 mmol), 9,9-Dimethyl-10(9*H*)-acridinyl (0.11 mmol), Pd$(OAc)_2$ (10 mol%), Phosphine ligand (20 mol%), *t*-BuONa (3 equiv.), toluene (2 mL) with N_2 atmosphere at 150 ºC in oil bath for 24 h. [b]NMR yield (The NMR yields determined by the characteristic proton signal of *R*/*S*-**10** at 1.73 ppm relative to the internal standard signal of CH_2Br_2 at 4.93 ppm.). [c]Isolated yield (The residue was purified by column chromatography on silica gel (PE/EA = 20/1, v/v) to give the isolated yield of *R*/*S*-**10**).



R/*S*-**10**

A 25 mL Schlenk tube equipped with a magnetic stir bar was charged with *R*/*S*-**8** (0.05 mmol), 9,9-Dimethyl-10(9*H*)-acridinyl (0.11 mmol), Pd(OAc)$_2$ (10 mol%), phosphine ligand (20 mol%), *t*-BuONa (3 equiv.) and toluene (2 mL), which was stirred at 150 ºC under N_2 atmosphere for 24 h. Then the mixture was filtered by diatomite and washed with CH_2Cl_2. The filtrate was concentrated and purified on column chromatography on silica gel (PE/EA = 20/1, v/v) to give *R*/*S*-**10** as a white solid in 88% yield (39 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.87 (d, *J* = 8.0 Hz, 4H), 8.42 (d, *J* = 7.6 Hz, 2H), 8.32 (s, 2H), 7.63 (t, *J* = 7.6 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 4H), 7.50-7.45 (m, 6H), 7.02-6.92 (m, 10H), 6.40 (d, *J* = 8.0 Hz, 4H), 1.73 (s, 12H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.9, 164.6, 151.9, 148.6, 143.7, 140.7, 138.8, 137.6, 135.5, 132.7, 131.6, 131.1, 130.1, 129.4, 126.4, 125.3, 124.1, 123.0, 120.7, 114.1, 59.4, 36.0, 31.3 ppm. HRMS (ESI$^+$): calcd for $C_{63}H_{46}N_6$ $[M+H]^+$, 887.3857; found 887.3855.

(2) Synthesis of 10*H*,10'*H*-9,9'-spirobi[acridine]



Scheme S4. Synthetic procedures of 10*H*,10'*H*-9,9'-spirobi[acridine]

Synthesis of 2-bromo-*N*-phenylaniline

A 250 mL flask equipped with a magnetic stir bar was charged with 2-bromoaniline (3.39 mL, 30 mmol), iodobenzene (3.69 mL, 33 mmol), $Pd(OAc)_2$ (336 mg, 2 mmol, 5 mol %), XantPhos (1.74 g, 3 mmol, 10 mol %), Cs_2CO_3 (20 g, 50 mmol, 1.6 equiv.) and dry and air-free dioxane (120 mL) under an N_2 atmosphere. Then the mixture was allowed to stir for 24 h at 110 °C in an oil bath. After cooling to room temperature, the mixture was filtrated by diatomite. The filtrate was concentrated and the residue was purified by column chromatography on silica gel (PE/DCM = 4/1, v/v) to give the desired product (6.9 g, yield = 94%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53 (d, J = 8.0 Hz, 1H), 7.33 (t, J = 8.0 Hz, 2H), 7.26 (d, J = 8.0 Hz, 1H), 7.19-7.15 (m, 3H), 7.05 (t, J = 7.6 Hz, 1H), 7.53 (t, J = 7.6 Hz, 1H), 6.10 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 141.6, 141.4, 132.9, 129.5, 128.1, 122.7, 120.9, 120.3, 115.8, 112.2 ppm. HRMS (ESI$^+$): calcd for $C_{12}H_{10}{}^{79}BrN$ $[M+Na]^+$, 269.9889; found 269.9886; calcd for $C_{12}H_{10}{}^{81}BrN$ $[M+Na]^+$, 271.9868; found 271.9866.

Synthesis of *t*-butyl (2-bromophenyl)(phenyl)carbamate

A 100 mL flask equipped with a magnetic stir bar was charged with 2-bromo-*N*-phenylaniline (2.48 g, 10 mmol), Boc_2O (20 mmol, 2 equiv.), 4-dimethylaminopyridine (DMAP) (10 mmol, 1 equiv.) and THF (50 mL), which was refluxed for 24 h. Then the mixture was extracted with water/dichloromethane and the organic phase was dried with anhydrous Na_2SO_4. The organic phase was concentrated and purified by column chromatography on silica gel (PE) to give the product as a white solid (3.1 g, yield = 90%). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.65 (d, J = 8.0 Hz, 1H), 7.33-7.28 (m, 6H), 7.18-7.11 (m, 2H), 1.44 (s, 9H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 152.9, 141.8, 141.6, 133.4, 130.7, 128.7, 128.5, 128.4, 125.1, 124.3, 81.4, 28.1 ppm. HRMS (ESI$^+$): calcd for $C_{17}H_{18}{}^{79}BrNO_2$ $[M+Na]^+$, 370.0413; found 370.0407; calcd for $C_{17}H_{18}{}^{81}BrNO_2$ $[M+Na]^+$, 372.0393; found 372.0388.

Synthesis of 10-((2-methoxyethoxy)methyl)acridin-9(10*H*)-one

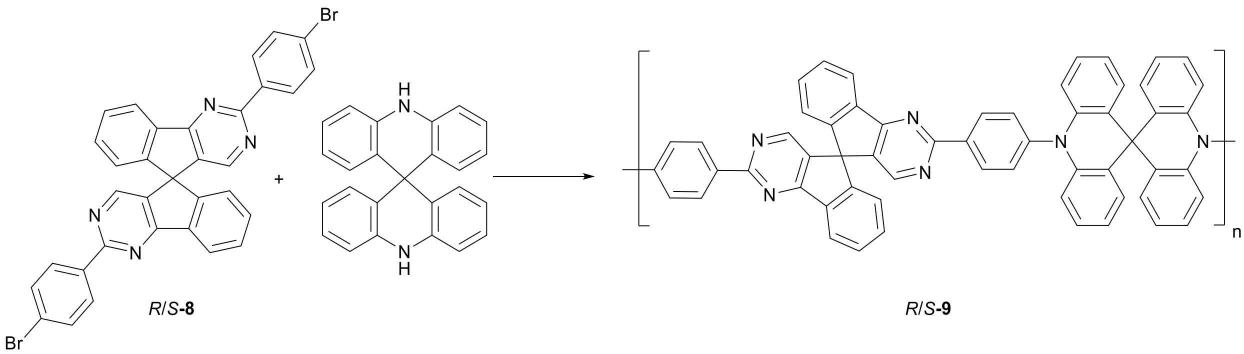
A 150 mL flask equipped with a magnetic stir bar was charged with 9(10*H*)-Acridone (5.85 g, 30 mmol) dissolved in DMF (100 mL). To the solution was added NaH (1.6 g, 40 mmol) at 0 °C. Then 2-methoxyethoxymethyl chloride (6.3 mL) was added dropwise after the mixture was stirred for 1 h at 0 °C. The mixture was stirred at room temperature for 12 h. Then the mixture was extracted with water/dichloromethane and the organic phase was dried with anhydrous Na_2SO_4. The organic phase was concentrated and purified by column chromatography on silica gel (PE/EA = 10/1, v/v) to give the product as a white solid (5 g, yield = 59%). ^1H NMR (400 MHz, $CDCl_3$): δ = 8.52 (d, J = 8.0 Hz, 2H), 7.74-7.69 (m, 4H), 7.32 (t, J = 6.0 Hz, 2H), 5.80 (s, 2H), 3.86 (t, J = 4.8 Hz, 2H), 3.64 (t, J = 4.8 Hz, 2H), 3.44 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 178.5, 142.4, 133.9, 127.6, 122.4, 122.1, 115.3, 72.2, 67.3, 59.2 ppm. HRMS (ESI$^+$): calcd for $C_{17}H_{17}NO_3$ $[M+Na]^+$, 284.1281; found 284.1281.

Synthesis of 10*H*,10'*H*-9,9'-spirobi[acridine]

A 100 mL flask equipped with a magnetic stir bar was charged with *t*-butyl (2-bromophenyl)(phenyl)carbamate (2.8 g, 8 mmol) dissolved in THF (60 mL). Then *n*-BuLi (3.6 mL, 9 mmol) was added dropwise into the solution at -78 °C and stirred for 2 h. Then a solution of 10-((2-methoxyethoxy)methyl)acridin-9(10*H*)-one (2.3 g, 8.1 mmol) in THF (25 mL) was added dropwise into the mixture at -78 °C. The mixture was stirred for 2 h and slowly rose to room temperature. To the solution was added HCl (15 mL, 1 mol/L). The resulting mixture was stirred for 12 h. Then the mixture was extracted with water/dichloromethane and the organic phase was dried with anhydrous Na_2SO_4. The organic phase was concentrated and purified by column chromatography on silica gel (PE/DCM = 1/1, v/v) to give 10*H*,10'*H*-9,9'-spirobi[acridine] as a yellow solid (1.2 g, yield = 42%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.02 (t, J = 8.0 Hz, 4H), 6.96 (d, J = 8.0 Hz, 4H), 6.70 (dd, J_1 = 14.0, J_2 = 7.6, 8H), 6.21 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 135.7, 132.3, 130.6, 126.9, 120.8, 113.1, 47.1 ppm. HRMS (ESI$^+$): calcd for $C_{25}H_{18}N_2$ [M+Na]$^+$, 369.1362; found 369.1356.

(3) Optimization of polymerization conditions of *R*/*S*-8 with 10*H*,10'*H*-9,9'-spirobi[acridine]

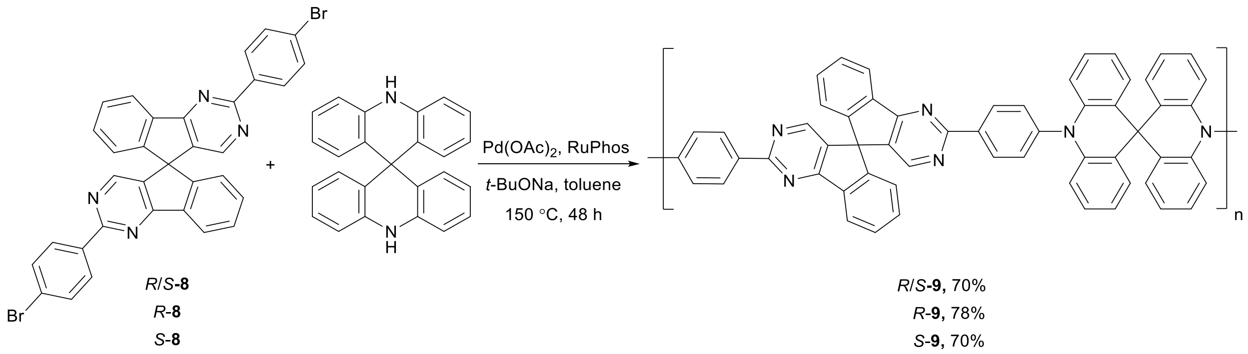
Table S4. Optimization of polymerization reaction conditions ^{a}



Entry	Catalyst	ligand	Temperature (°C)	Time (h)	M_n (kDa)
1	$Pd(OAc)_2$	RuPhos	150	24	3698
2	$Pd(OAc)_2$	RuPhos	150	36	6621
3	$Pd(OAc)_2$	RuPhos	150	48	12543
4	$Pd_2(dba)_3$	P^tBu_3	120	24	3685

^{a}Reaction conditions: *R*/*S*-**8** (0.1 mmol), 10*H*,10'*H*-9,9'-spirobi[acridine] (0.1 mmol), [Pd] (10 mol %), ligand (20 mol %), *t*-BuONa (0.3 mmol), toluene (2 mL), N_2 atmosphere.

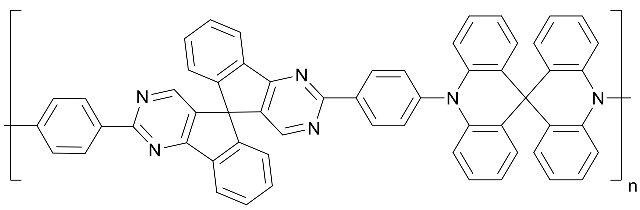
5. Synthesis of *R*/*S*-, *R*- and *S*-9 from monomers *R*/*S*-, *R*- and *S*-8



Scheme S5. Synthetic procedures of *R*/*S*-, *R*- and *S*-**9**

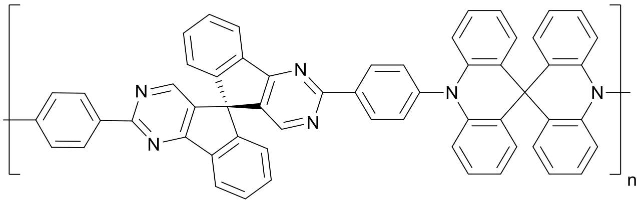
General procedure for the synthesis of *R*/*S*-, *R*- and *S*-9

a 25 mL Schlenk tube equipped with a magnetic stir bar was charged with *R*/S-**8** (62 mg, 0.1 mmol), 10*H*,10'*H*-9,9'-spirobi[acridine] (35 mg, 0.1 mmol, 1 equiv.), $Pd(OAc)_2$ (2 mg, 10 mol %, 0.01 mmol), RuPhos (9 mg, 20 mol %, 0.02 mmol), *t*-BuONa (30 mg, 0.3 mmol, 3 equiv.) and toluene (2 mL), which was stirred at 150 ºC under N_2 atmosphere for 48 h. After cooling to room temperature, the mixture was poured into methanol, and the precipitate was filtrated and washed with excess methanol. The solid was purified by Soxhlet extraction with acetone/methanol for 12 h and dichloromethane overnight successively. Then the extracting solution of dichloromethane was concentrated to an appropriate volume, precipitated by methanol. The precipitate was filtrated and dried under vacuum to give the product.



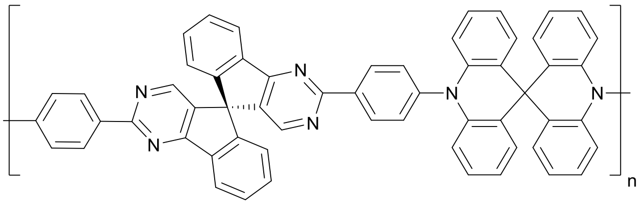
R/*S*-**9**

R/*S*-**9** was synthesized from *R*/*S*-**8** and 10*H*,10'*H*-9,9'-spirobi[acridine] in 70% yield. The average molecular weight detected by GPC showed M_n = 12543, M_w = 24256, M_w/M_n = 1.93.



R-**9**

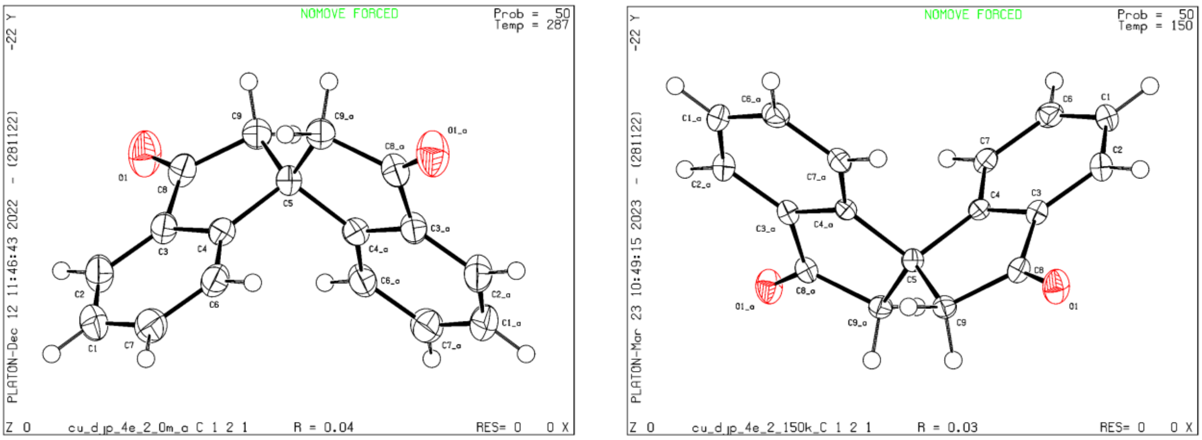
R-**9** was synthesized from *R*-**8** and 10*H*,10'*H*-9,9'-spirobi[acridine] in 78% yield. The average molecular weight detected by GPC showed M_n = 12665, M_w = 25063, M_w/M_n = 1.97.



S-**9**

S-**9** was synthesized from *S*-**8** and 10*H*,10'*H*-9,9'-spirobi[acridine] in 70% yield. The average molecular weight detected by GPC showed M_n = 12347, M_w = 25646, M_w/M_n = 2.07.

III. X-ray crystallographic analyses



R-**5** *S*-**5**

Fig. S1. Ellipsoid plot diagram of crystal structures of *R*-**5** and *S*-**5**

Table S5. Crystal data and structure refinement for *R*-**5**

Parameter	*R*-**5**
Empirical formula	$C_{17}H_{12}O_2$

Formula weight	248.27
Temperature/K	287.0
Crystal system	monoclinic
Space group	C2
a/Å	17.755(7)
b/Å	5.479(2)
c/Å	6.556(3)
α /°	90
β /°	103.98(2)
γ /°	90
Volume/Å^3	618.9(5)
Z	2
ρ calcg/cm^3	1.332
μ /mm^{-1}	0.693
F(000)	260.0
Crystal size/mm^3	0.32 × 0.13 × 0.07
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	10.268 to 134.064
Index ranges	-20≤ h ≤ 18, -6 ≤ k ≤ 6, -7 ≤ l ≤ 7
Reflections collected	2945
Independent reflections	1062 [R_{int} = 0.0444, R_{sigma} = 0.0434]
Data/restraints/parameters	1062/1/87
Goodness-of-fit on F^2	1.123
Final R indexes [I>=2σ (I)]	R_1 = 0.0378, wR_2 = 0.0943
Final R indexes [all data]	R_1 = 0.0389, wR_2 = 0.0951
Largest diff. peak/hole / e Å^{-3}	0.21/-0.14
Flack parameter	0.01(17)

Table S6. Crystal data and structure refinement for *S*-**5**

Parameter	*S*-**5**
Empirical formula	$C_{17}H_{12}O_2$
Formula weight	248.27

Temperature/K	150.0
Crystal system	monoclinic
Space group	C2
a/Å	17.6508(8)
b/Å	5.4506(2)
c/Å	6.5314(3)
α /°	90
β /°	103.9500(10)
γ /°	90
Volume/Å^3	609.84(5)
Z	2
ρ calcg/cm^3	1.352
μ /mm^{-1}	0.703
F(000)	260.0
Crystal size/mm^3	0.39 × 0.22 × 0.13
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	10.328 to 143.826
Index ranges	-20≤ h ≤ 21, -6 ≤ k ≤ 6, -8 ≤ l ≤ 8
Reflections collected	5405
Independent reflections	1159 [R_{int} = 0.0257, R_{sigma} = 0.0220]
Data/restraints/parameters	1159/1/87
Goodness-of-fit on F^2	1.106
Final R indexes [I>=2σ (I)]	R_1 = 0.0257, wR_2 = 0.0657
Final R indexes [all data]	R_1 = 0.0257, wR_2 = 0.0658
Largest diff. peak/hole / e Å^{-3}	0.15/-0.14
Flack parameter	0.04(6)

IV. GPC and BET results

Table S7. Molecular weights of *R*/*S*-, *R*- and *S*-**9**

	M_n (g mol^{-1})	M_w (g mol^{-1})	M_w/M_n
S-**9**	12347	25646	2.07
R-**9**	12665	25063	1.97
R/*S*-**9**	12543	24256	1.93

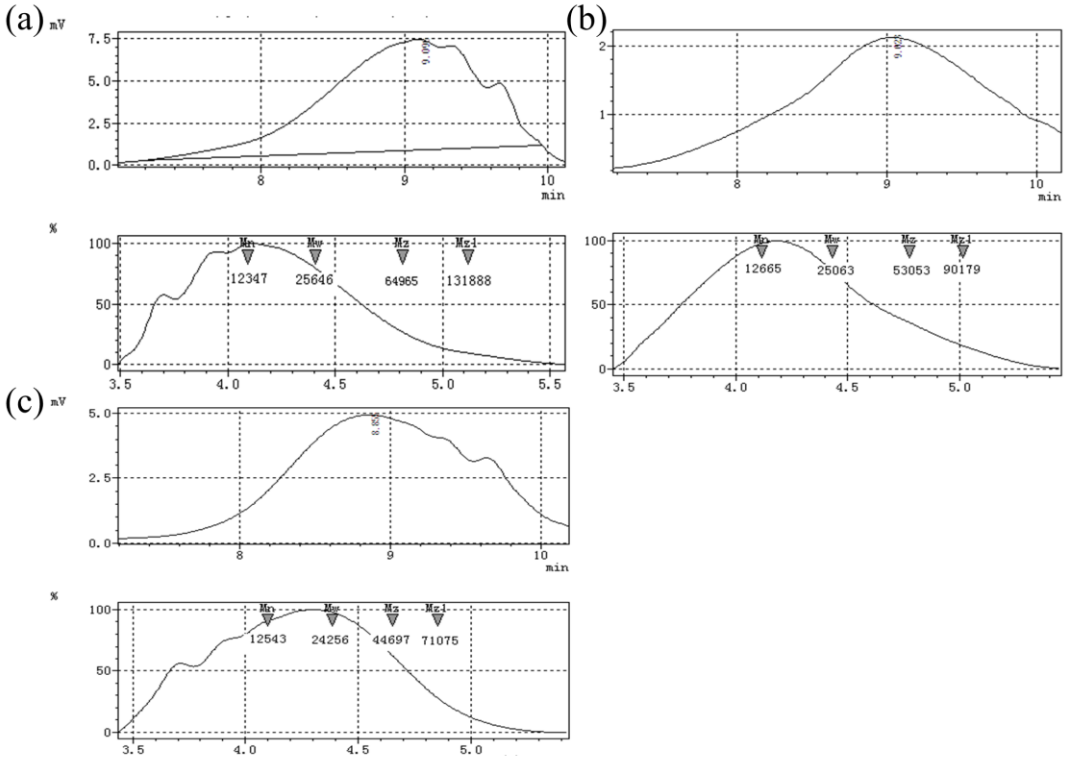


Fig. S2. GPC results of (a) *S*-**9,** (b) *R*-**9**, and (c) *R*/*S*-**9** (Shodex GPC KF-805L, eluent: THF, temperature: 40 °C, 1 mL/min)

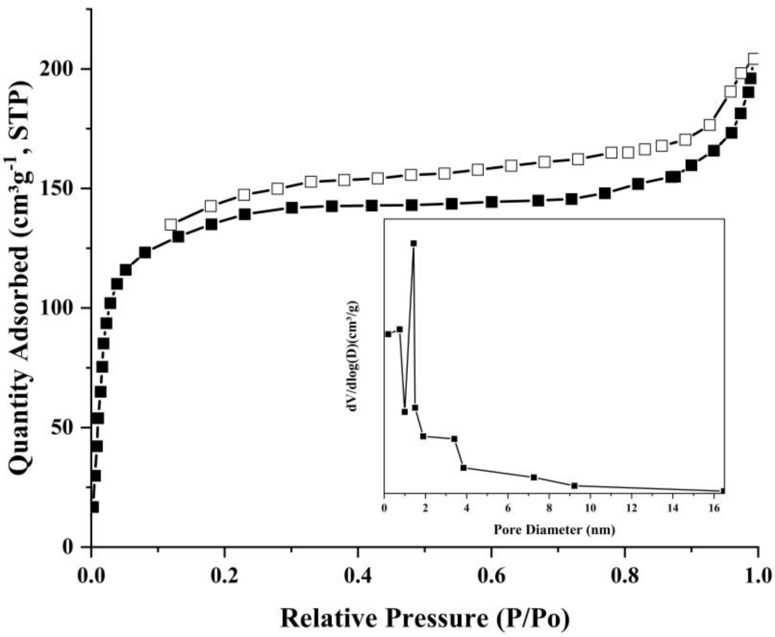


Fig. S3. N_2 adsorption/desorption isotherms of *R*-**9** at 77 K, where the inset shows the pore size distribution.

V. Photophysical Properties

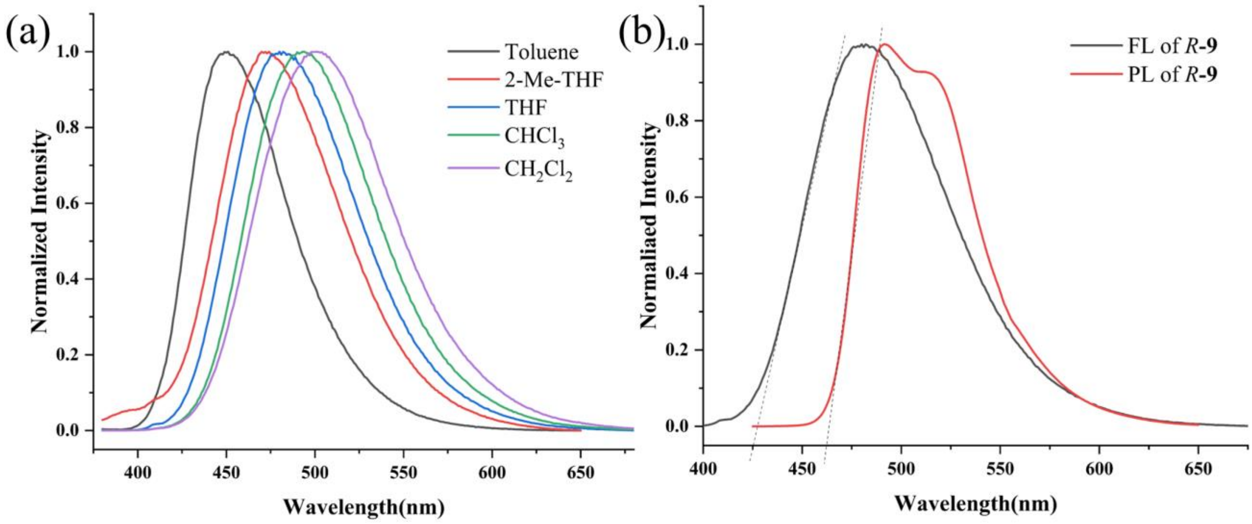


Fig. S4. (a) Normalized fluorescence spectra of *R*-**9** in different solvents (20 mg L^{-1}). (b) Normalized fluorescence and phosphorescence spectra of *R*-**9** in THF solution (77K).

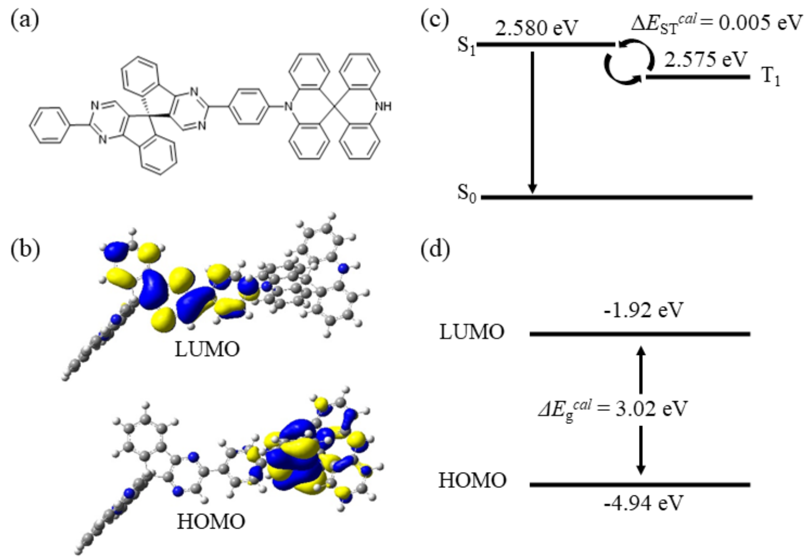


Fig. S5. (a) Repeat units of *R*-**9**. (b) HOMO and LUMO distributions of repeat units of *R*-**9**. (c) Calculated S1 and T1 energy levels. (d) Calculated LUMO and HOMO energy levels.

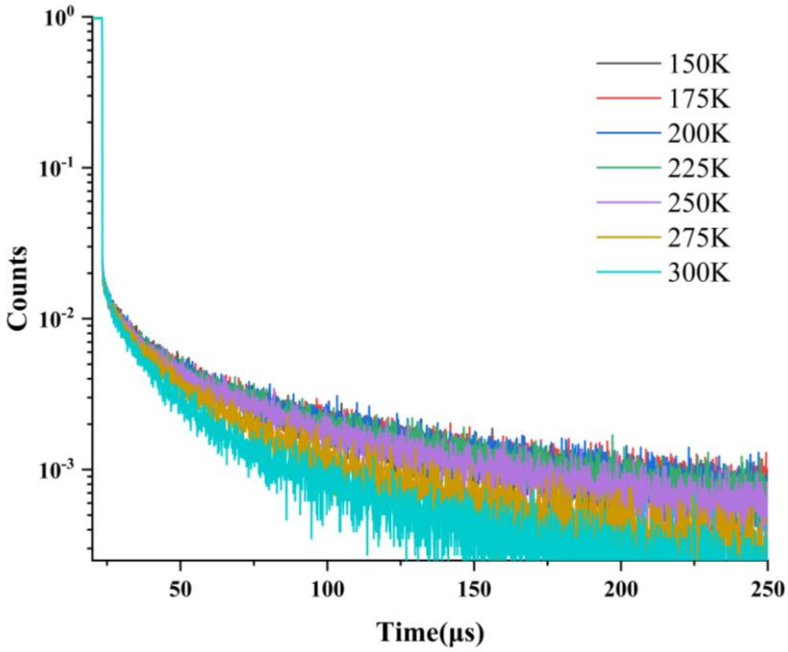


Fig. S6. Temperature-dependent transient photoluminescence spectra of *R*-**9** in neat film under high vacuum (1 mm Hg).

Table S8. Summary of the physical properties of *R*-**9**

	λ_{abs}[a] (nm)	λ_{em}[b] (nm)	τ_P(c) (ns)	τ_D[c] (μs)	Φ (%)	E_{S1}/E_{T1}[e] (eV)
R-**9**	262, 291, 369	482	40	126	20[a]/19.4[d]	2.93/2.69

[a] Measured in THF (20 mg L^{-1}) at 298 K. [b] Measured in THF (20 mg L^{-1}) at 298 K and attributed to the S_1-S_0 transition. [c] τ_P (prompt lifetime) and τ_D (delayed lifetime) determined from the transient decay spectrum at 298 K. [d] Measured in a neat film at 298 K. [f] Energy of S_1 and T_1 determined from the fluorescence and phosphorescence spectra at 77 K.

VI. Electrochemical Measurements

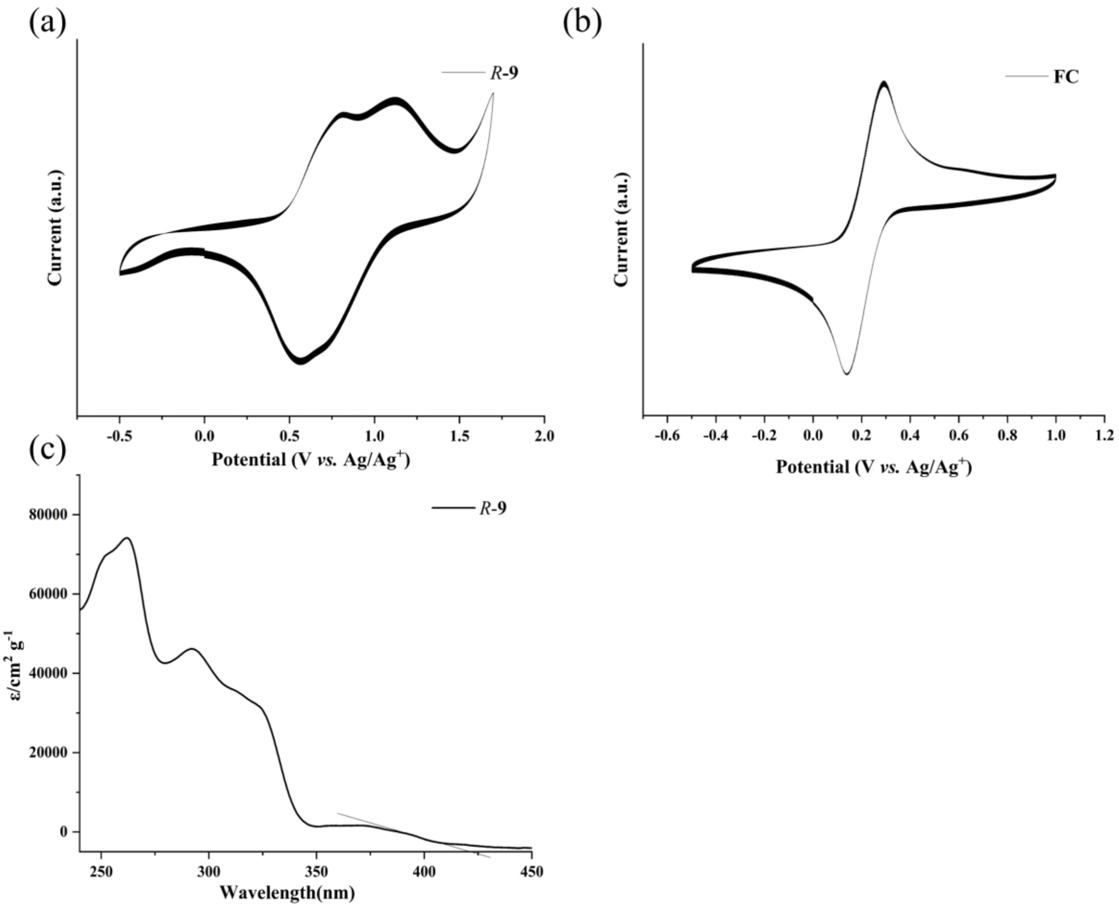


Fig. S7. (a) Cyclic voltammetry characteristic curve of *R*-**9** in dry and degassed DCM (0.8 mg mL^{-1}). (b) Cyclic voltammetry characteristic curve of ferrocene in dry and degassed DCM (0.19 mg mL^{-1}). (c) UV-vis absorption spectrum of *R*-**9** in THF (20 mg L^{-1}).

Table S9. Electrochemical properties of *R*-**9**

	E_{HOMO} [a]	E_g [b]	E_{LOMO} [c]
R-**9**	-5.43 eV	2.95 eV	-2.48 eV

[a] Measured in dry dichloromethane solution, where $E_{HOMO} = -4.8 - (E_{ox} - E_{Fc})$. [b] Calculated from the UV-vis absorption spectrum. [c] $E_{LUMO} = E_{HOMO} + E_g$.

VII. Chiroptical Properties

Table S10. Summary of the chiroptical properties of *R*- and *S*-**9**

	CD[a] λ (nm)	ε [a] (cm^2 g^{-1})	$\Delta\varepsilon$ [a] (cm^2 g^{-1})	g_{abs} (10^{-3})	CPL[a] λ (nm)	g_{lum} (10^{-3})
R-**9**	270	55996	360	6.4	451	-2.4
S-**9**	270	77417	-472	-6.1	451	2.4

[a] Measured in THF (20 mg L^{-1}) at 298 K.

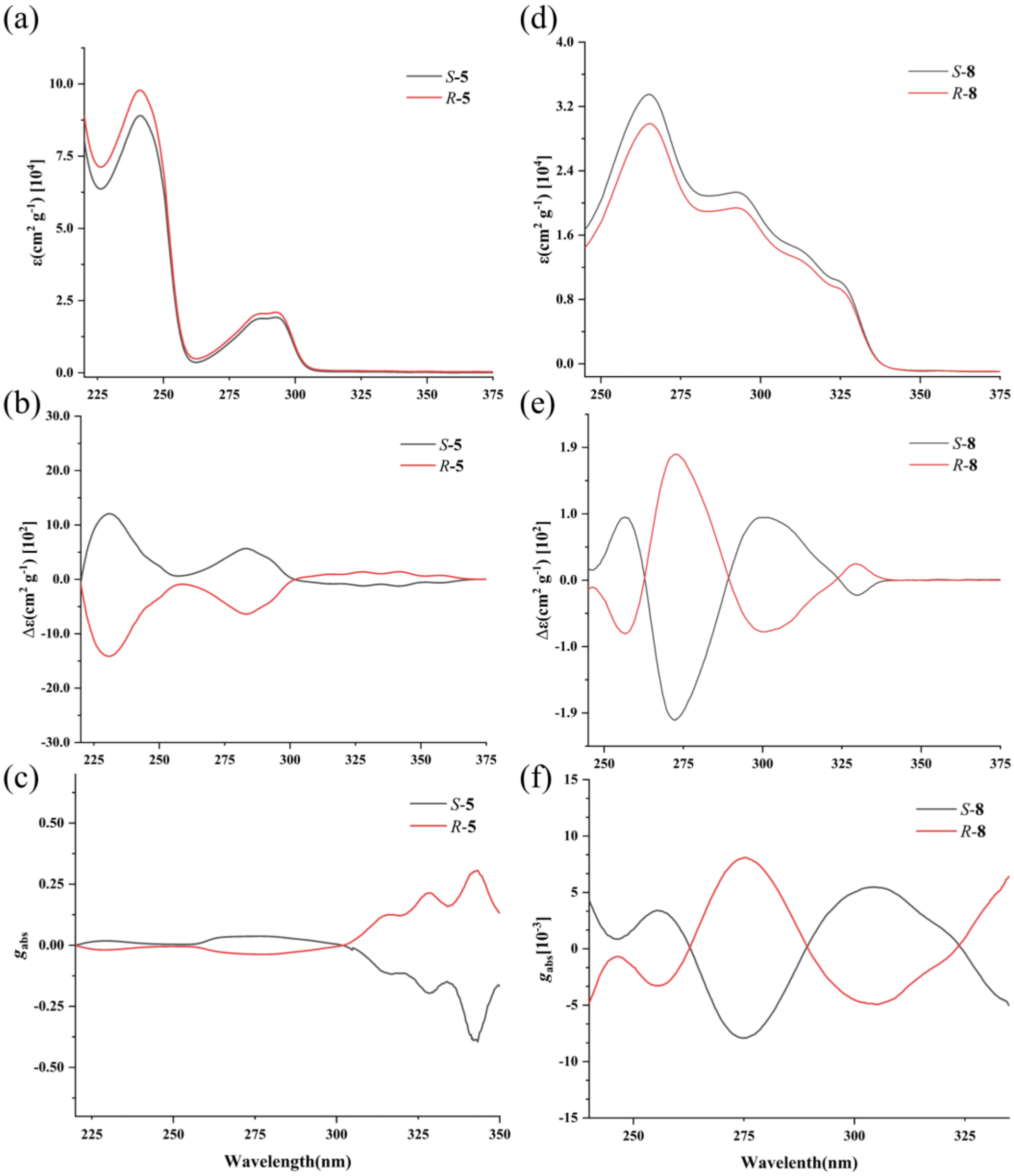


Fig. S8. (a) UV-vis absorption, (b) Circular dichroism (CD), (c) g_{abs} spectra of *R*- and *S*-**5** in THF (10^{-4} mol L^{-1}) at 298 K; (d) UV-vis absorption, (e) Circular dichroism (CD), (f) g_{abs} spectra of *R*- and *S*-**8** in THF (10^{-4} mol L^{-1}) at 298 K.

VIII. Computational Geometry Data

Geometrically optimized Cartesian coordinates of *R*-**9**

C	-6.52879400	-0.13719300	-0.28082500
C	-7.54922400	-0.03555300	0.67989500
C	-7.41297000	-1.13795800	1.63734200
C	-6.30373800	-1.91386400	1.25830500
C	-5.63152300	-1.33510400	0.00356800
C	-4.16503600	-0.95869900	0.22065300
C	-3.33134300	-1.63695400	-0.69082800
C	-4.15825600	-2.48942600	-1.54715300
C	-5.50272400	-2.32787700	-1.16055300
C	-3.80453400	-3.34564400	-2.59026800
C	-4.81773900	-4.04509400	-3.24648100
C	-6.15620800	-3.88695800	-2.86356500
C	-6.50867600	-3.02709000	-1.81796200
N	-3.69063700	-0.12429600	1.13219800
C	-2.35796300	0.03858300	1.11381200
C	-1.51002300	-0.60881100	0.19296500
N	-2.01248700	-1.47291300	-0.71726600
C	-6.52125400	0.82972300	-1.27152900
N	-7.44071400	1.80379700	-1.29824600
C	-8.37682100	1.81659500	-0.33014900
N	-8.47409400	0.91975500	0.67548800
C	-8.17945100	-1.46890100	2.75407900
C	-7.81978500	-2.59626700	3.49322900
C	-6.71609500	-3.37260000	3.11620300
C	-5.94917300	-3.03795800	1.99497500
C	-9.38646500	2.90424500	-0.37324900
C	-0.04121300	-0.39940200	0.18783500
C	0.79435700	-1.35879800	-0.40844500
C	2.17575500	-1.19220300	-0.42068200
C	2.75572800	-0.05883600	0.15878500
C	1.93331300	0.90852100	0.74453200
C	0.55056200	0.74084300	0.75712500
N	4.18003000	0.11133900	0.14449500
C	4.78202300	0.79139600	-0.93338200
C	6.17944400	0.95669300	-0.99232500
C	7.13186500	0.39452400	0.07903900
C	6.34434300	-0.24537600	1.23386700
C	4.94195600	-0.37347700	1.22481000
C	7.99037600	1.55228500	0.63433500
C	9.36557200	1.66055700	0.36011600
N	10.00286700	0.69897100	-0.41427300
C	9.39348500	-0.48679200	-0.80633400
C	8.02022400	-0.68737000	-0.57739900
C	7.05595000	-0.74382700	2.33246100
C	6.42706100	-1.35923900	3.40835800
C	5.03673800	-1.48445200	3.39082600
C	4.30278300	-0.99896300	2.31636800

C	3.98328100	1.31750900	-1.96967800
C	4.55611200	1.99283500	-3.03987800
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C	8.20527800	-2.88447100	-1.62917500
C	7.45424000	-1.89621000	-0.99854200
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C	8.11727900	3.63713800	1.90120000
C	9.48415400	3.72946300	1.62232100
C	10.10372900	2.74890100	0.85909300
H	-2.76312900	-3.45592300	-2.87741200
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H	10.06836600	4.56400100	2.00080000
H	11.16775700	2.81323300	0.63930400

IX. References

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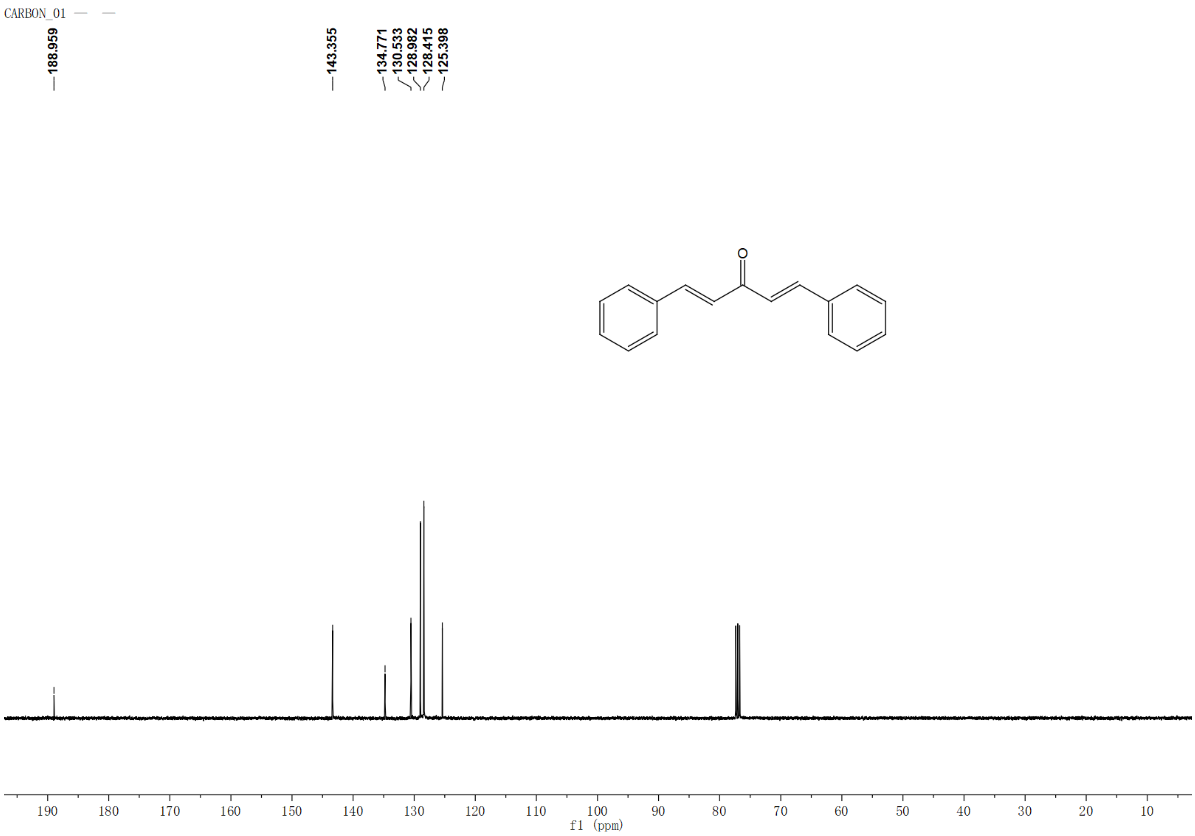
X. Copies of ^1H and ^{13}C NMR spectra

^1H NMR spectrum of **2**($CDCl_3$)

^{13}C NMR spectrum of **2**($CDCl_3$)



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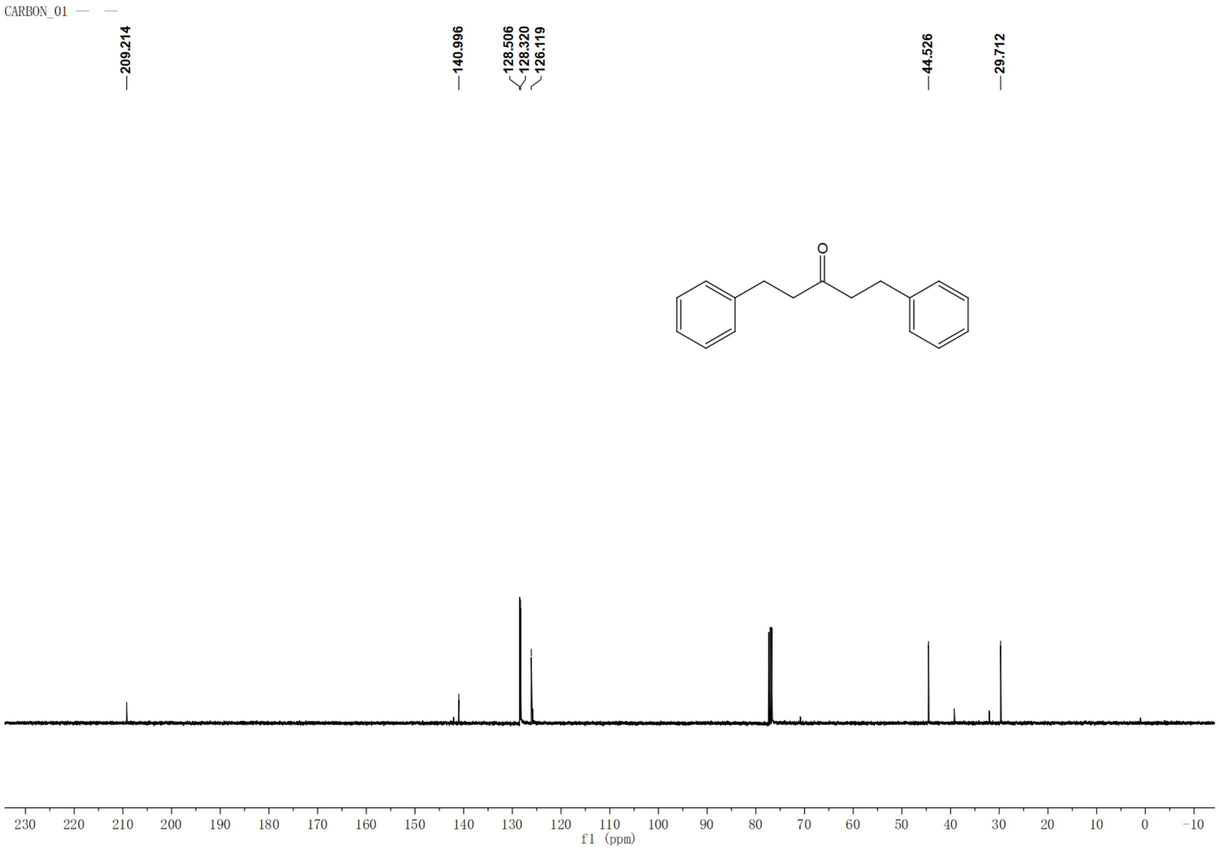
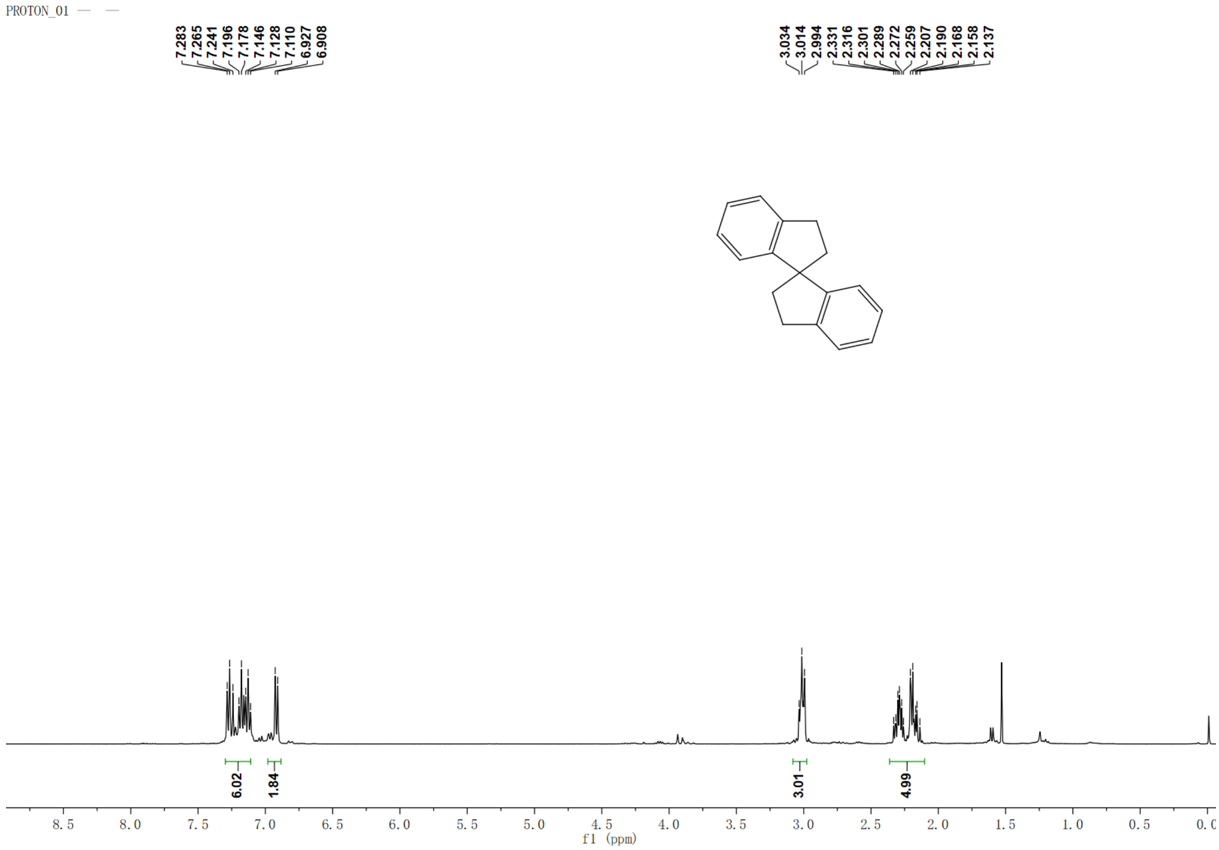


[1]H NMR spectrum of *R*/*S*-**4** ($CDCl_3$)



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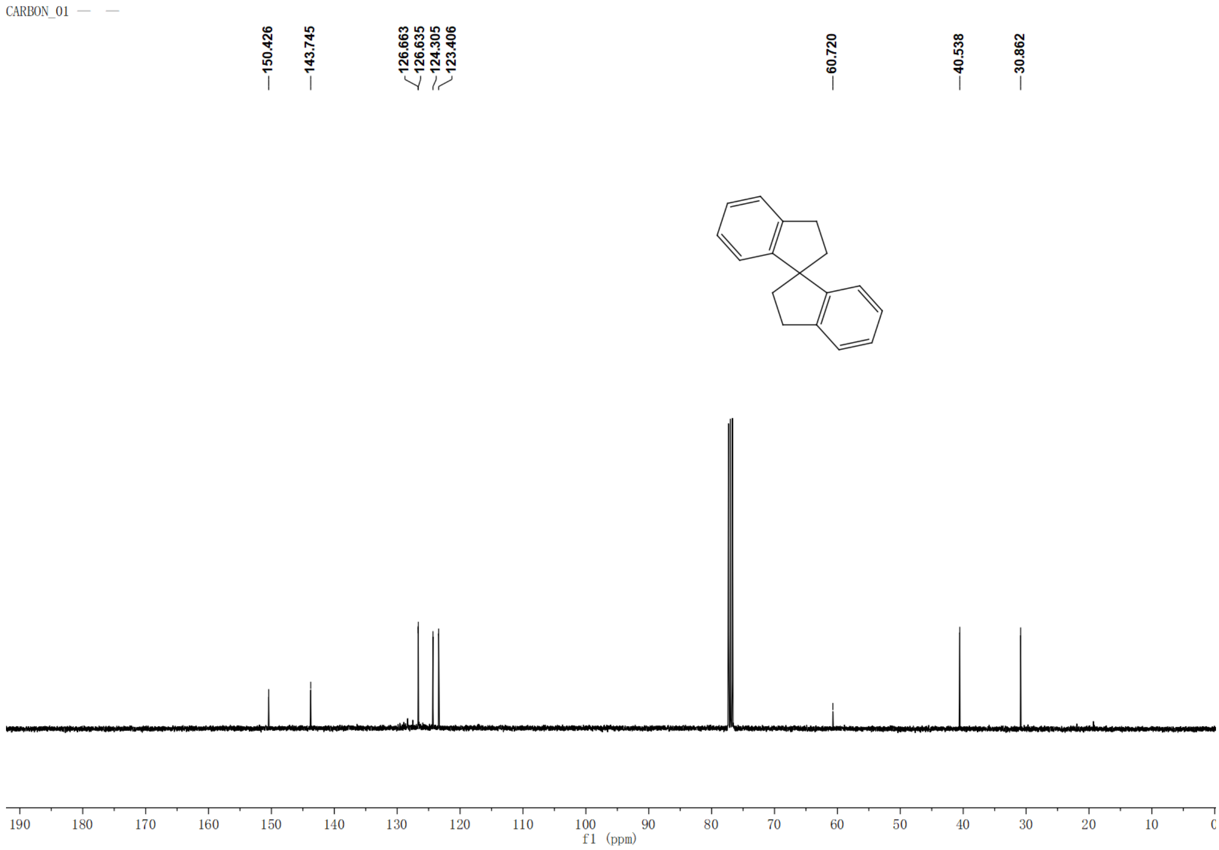

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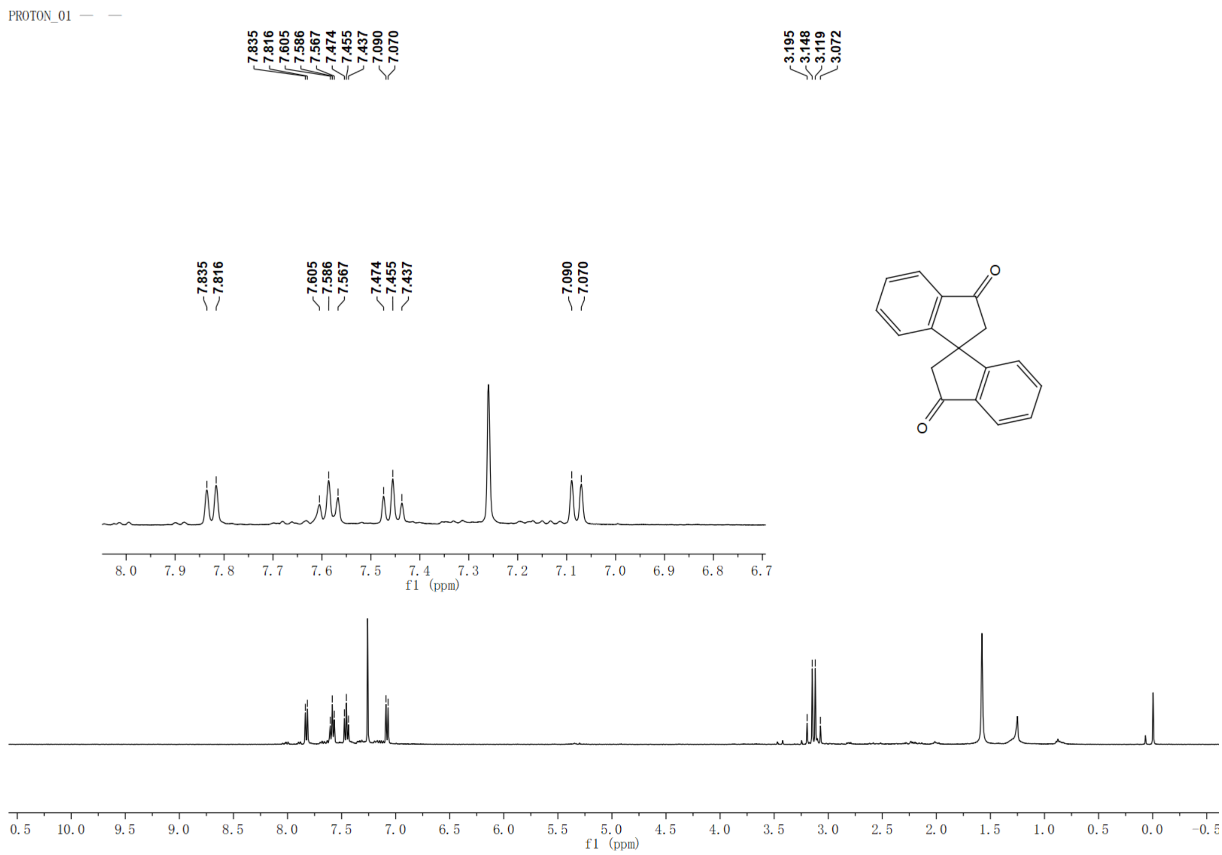

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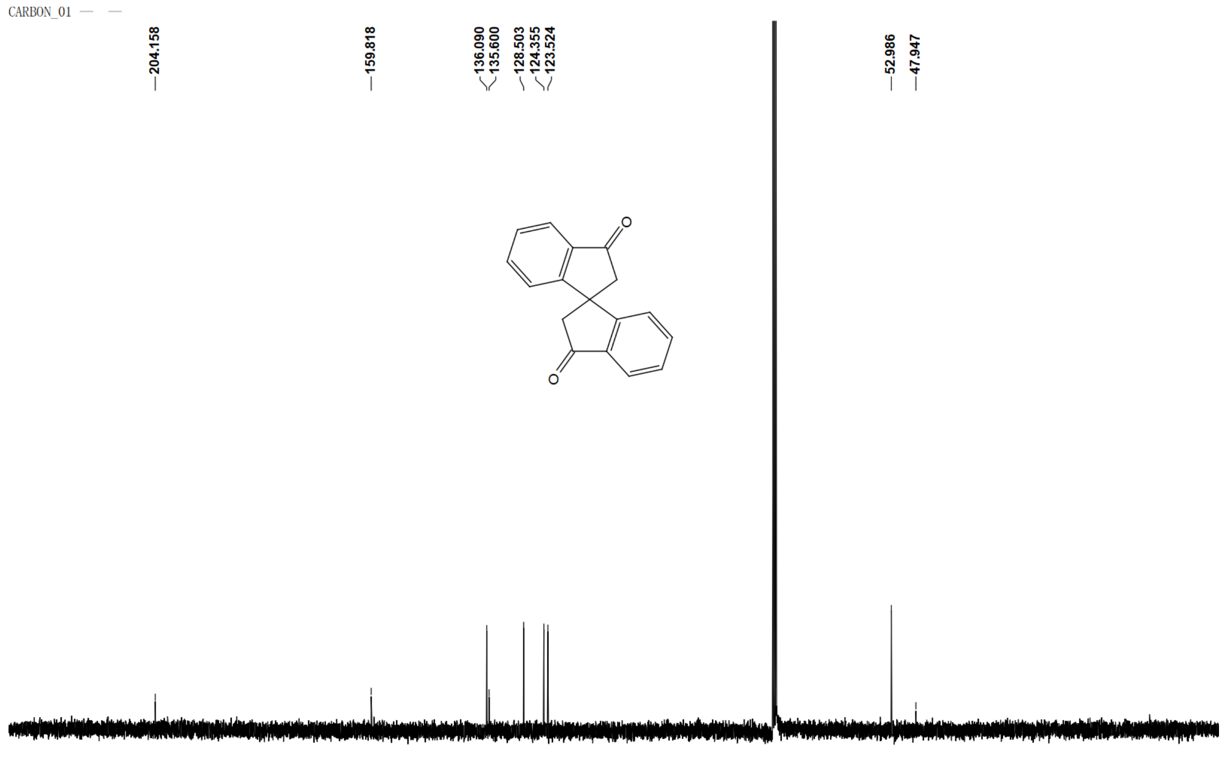

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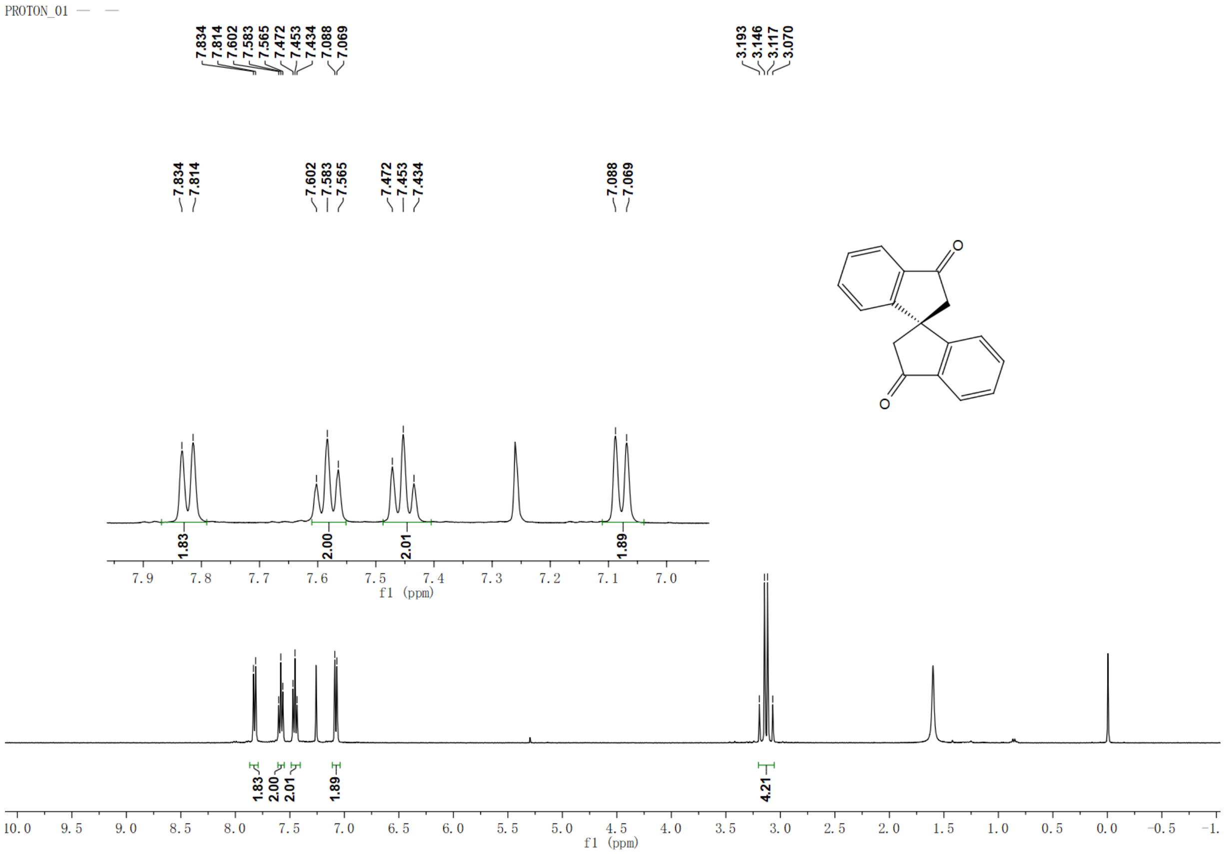

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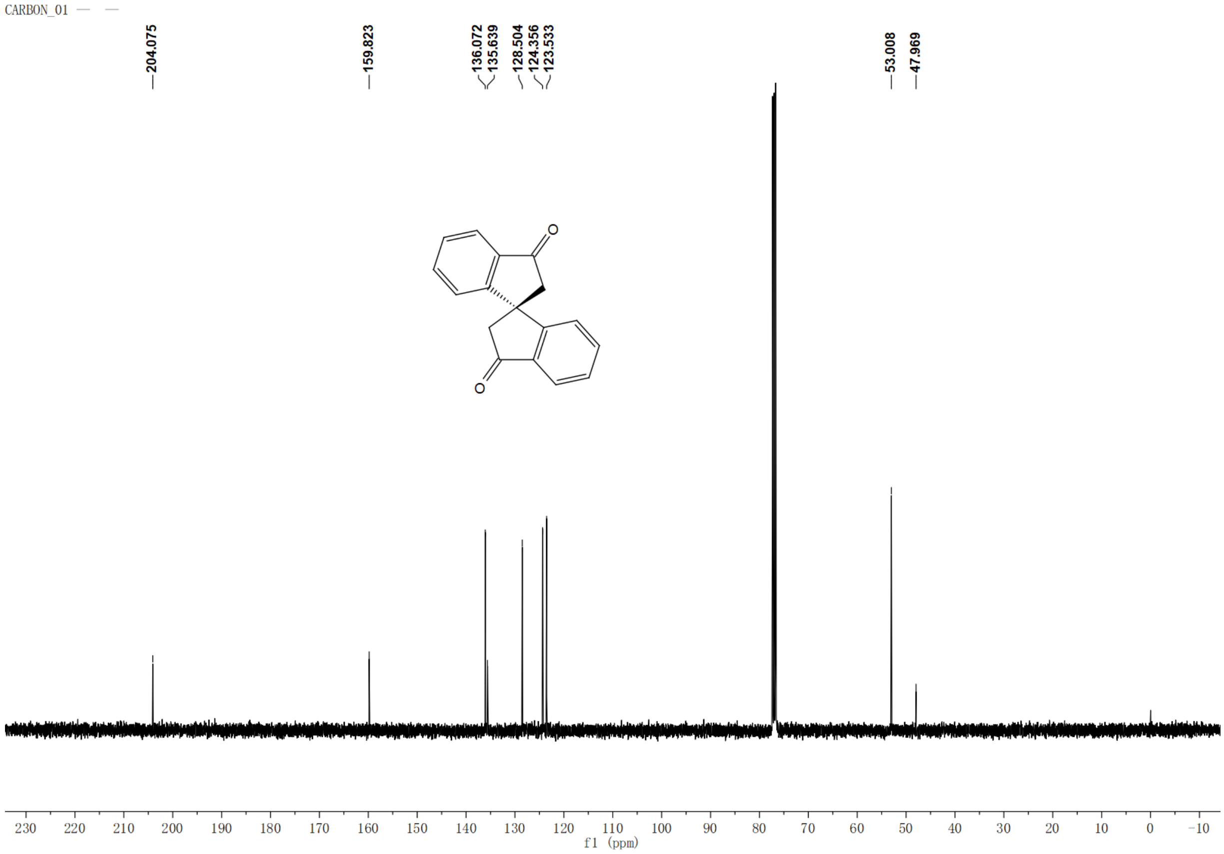

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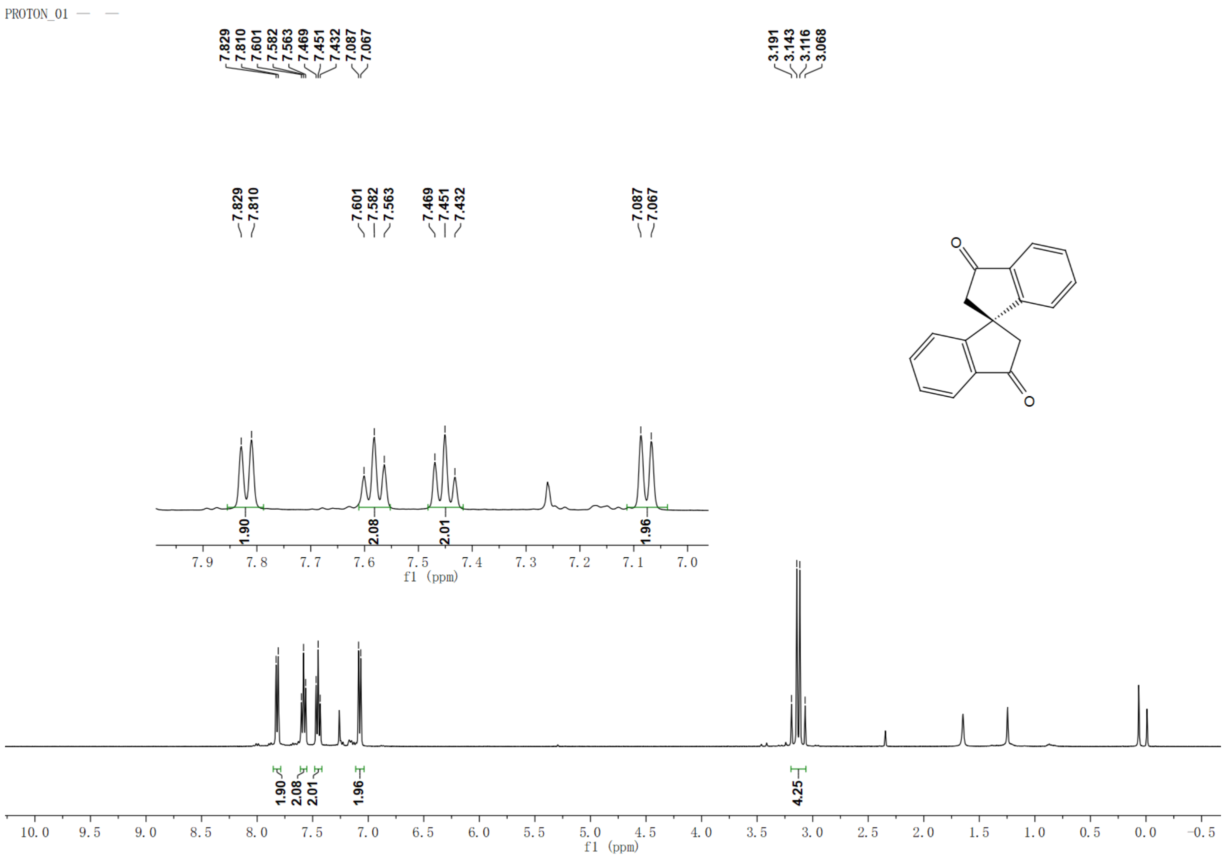

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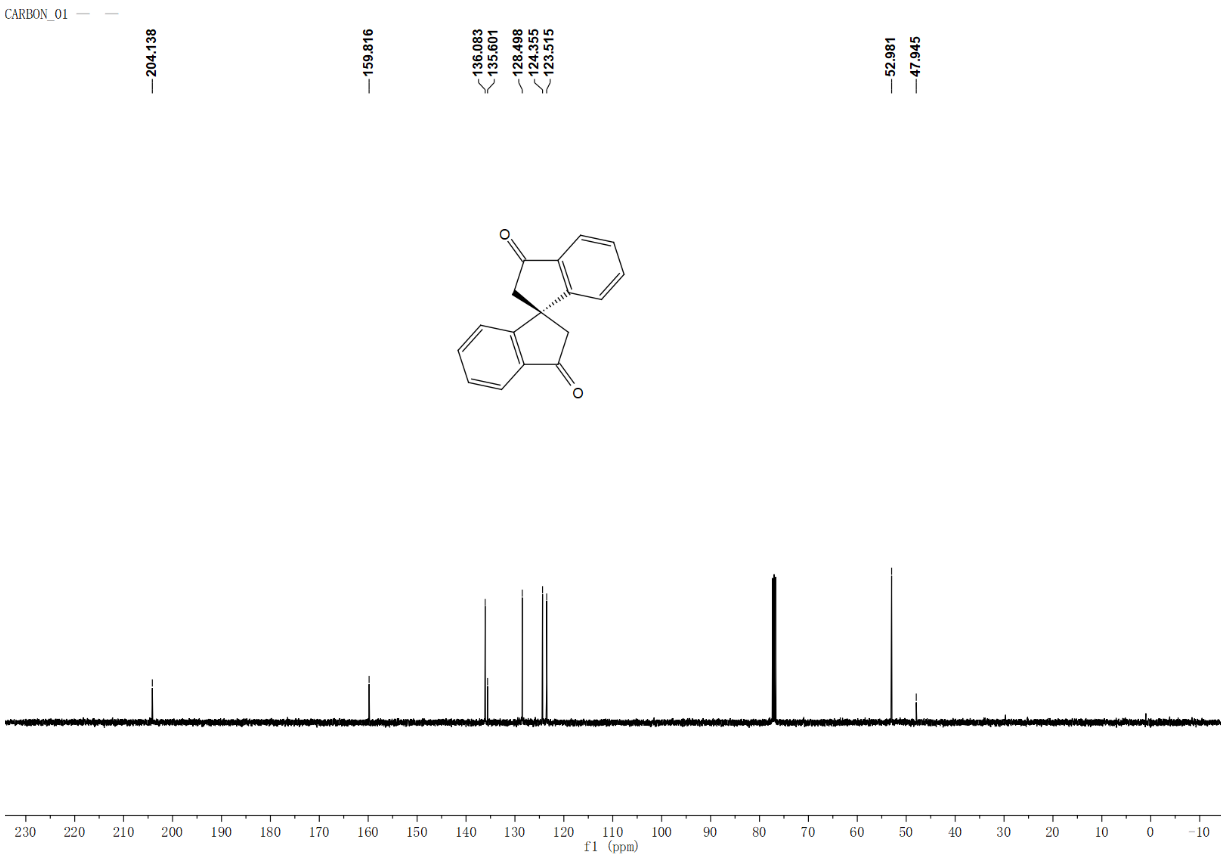

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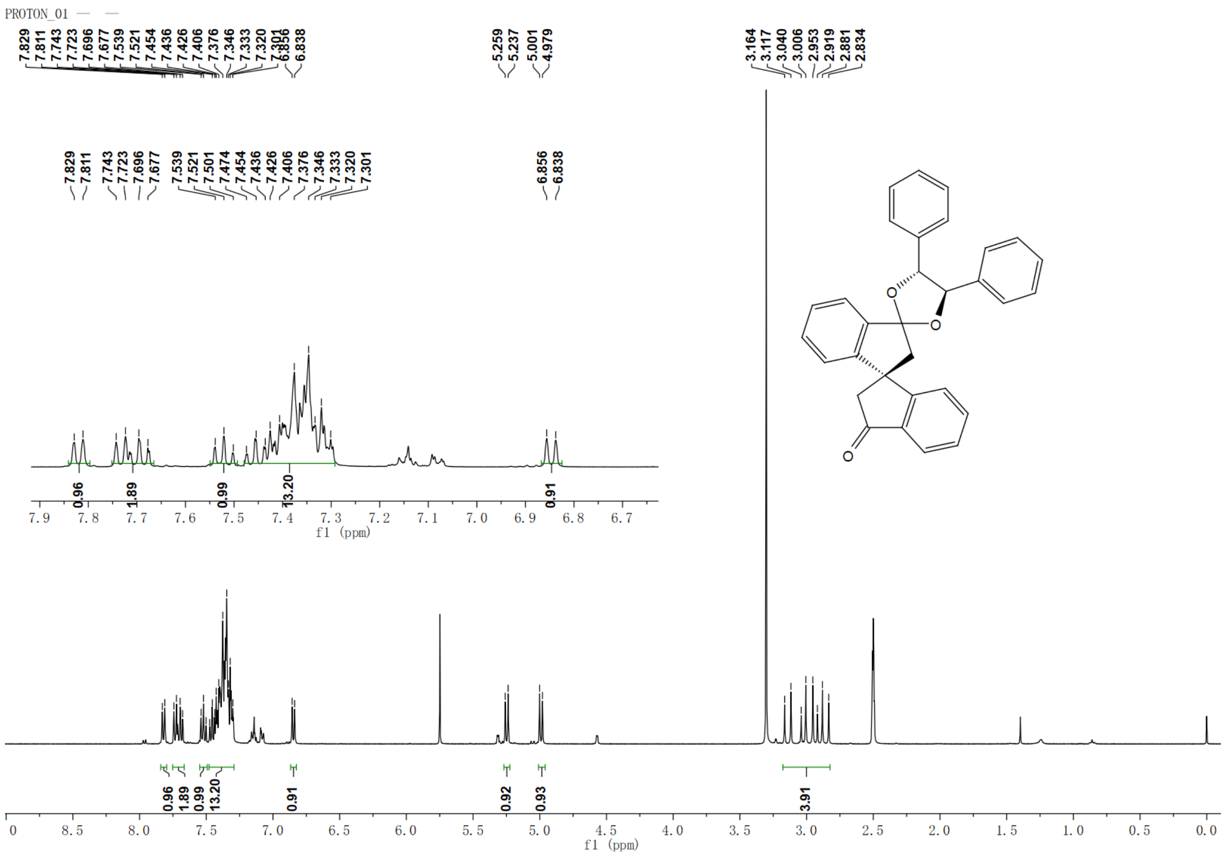

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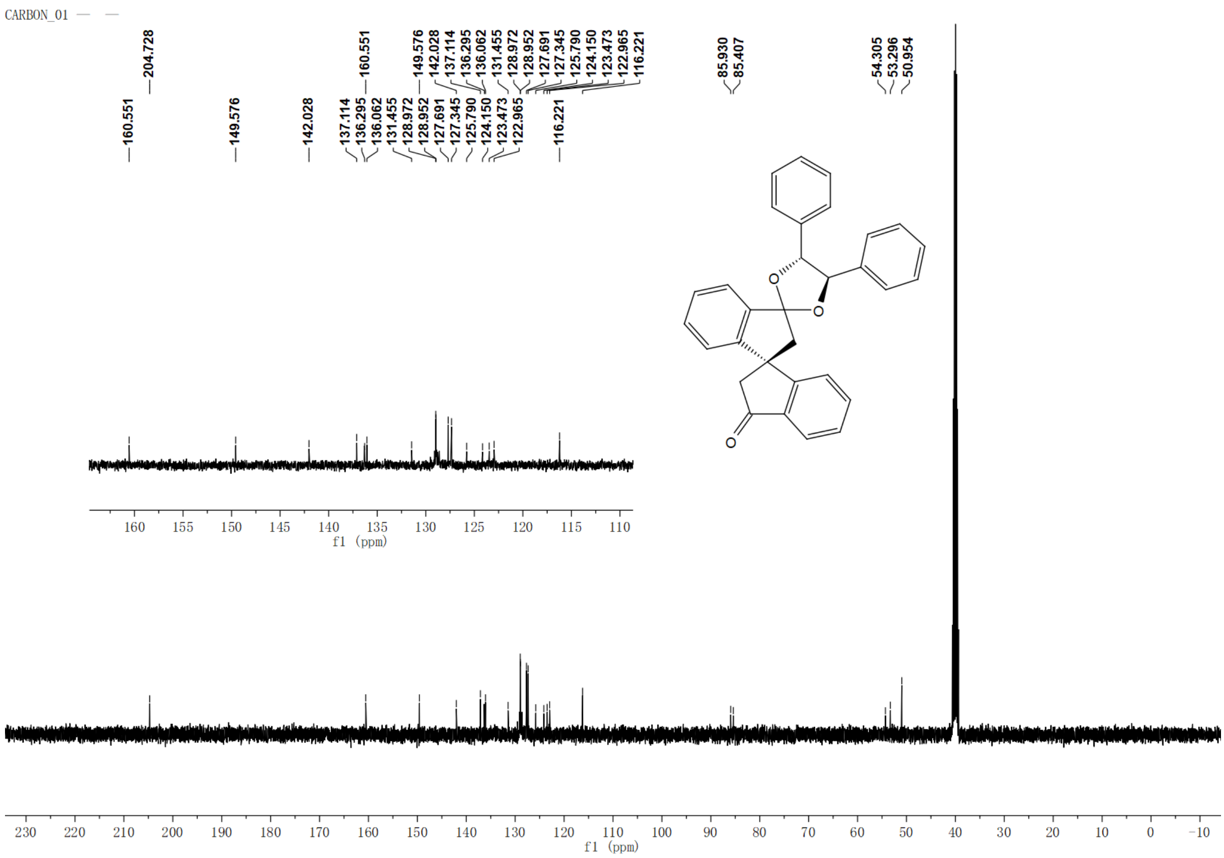

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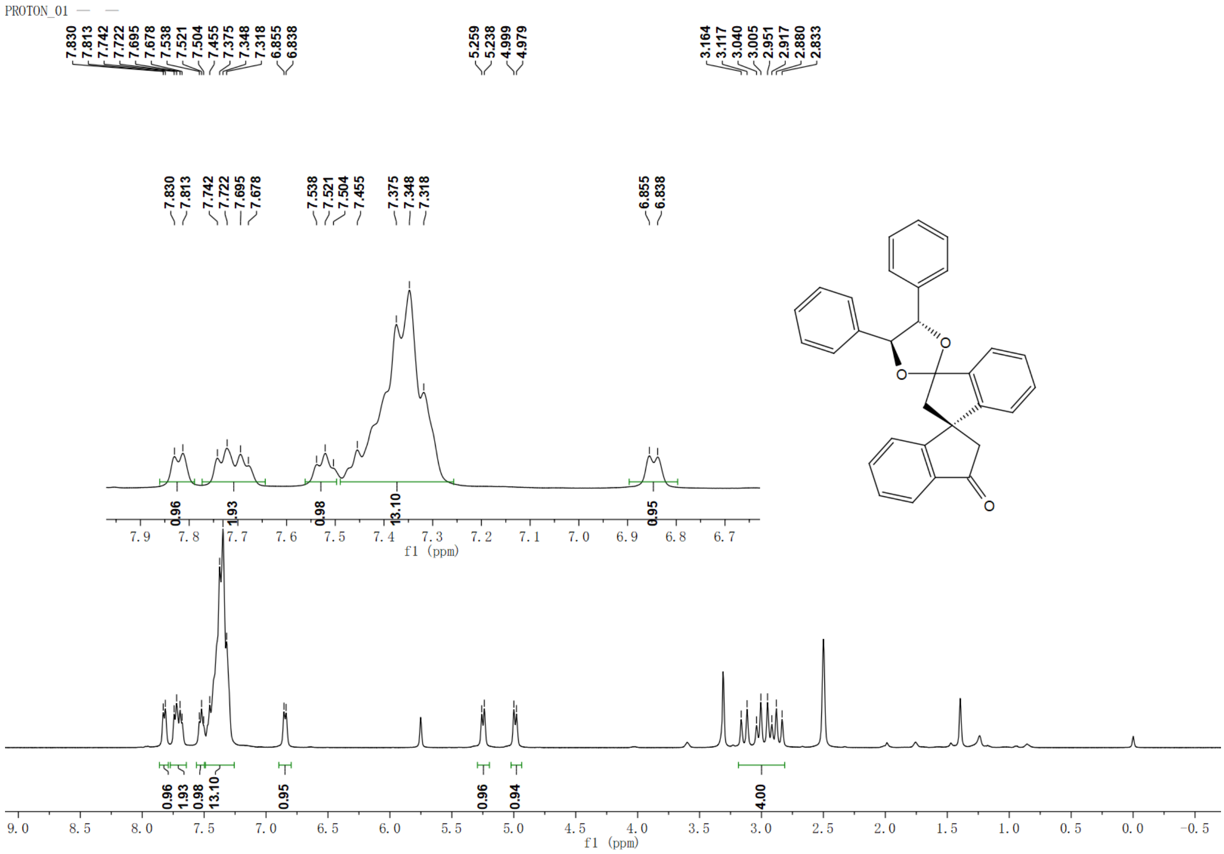

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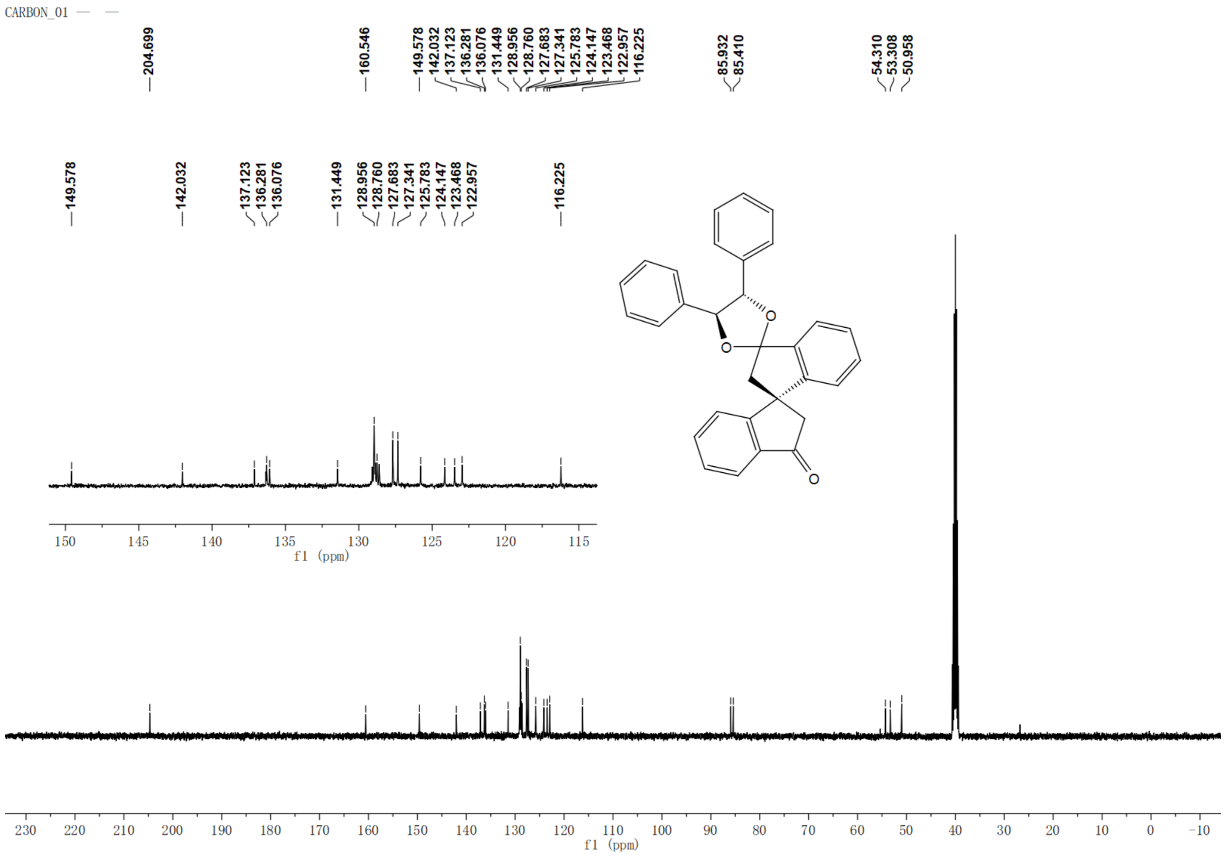

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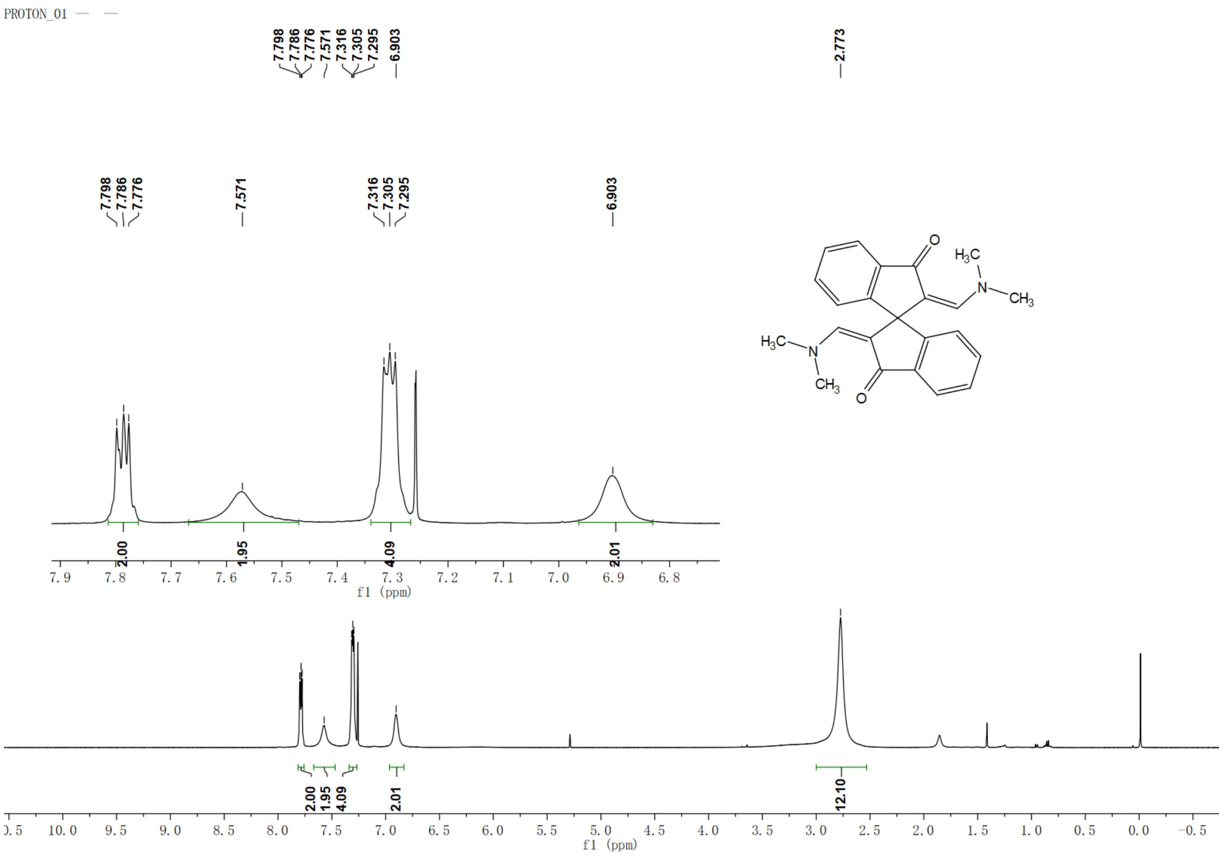

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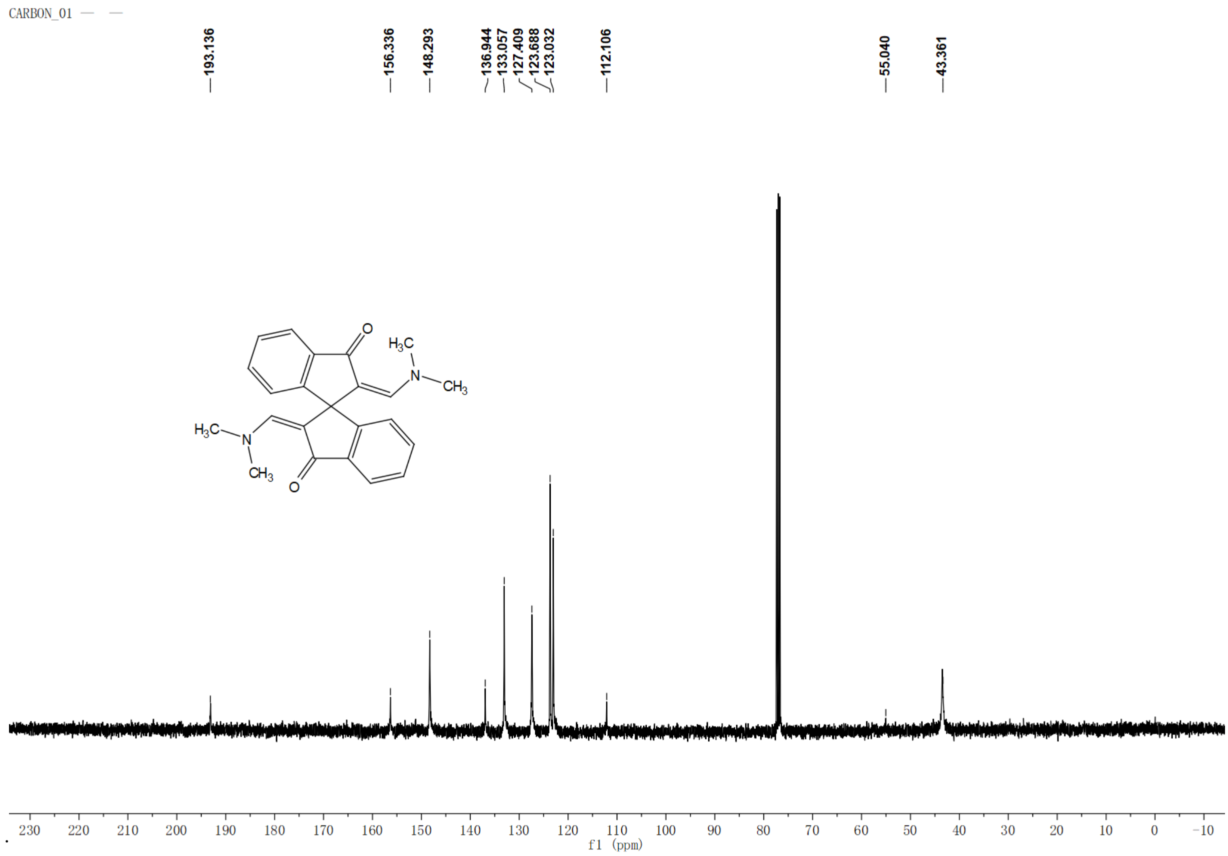

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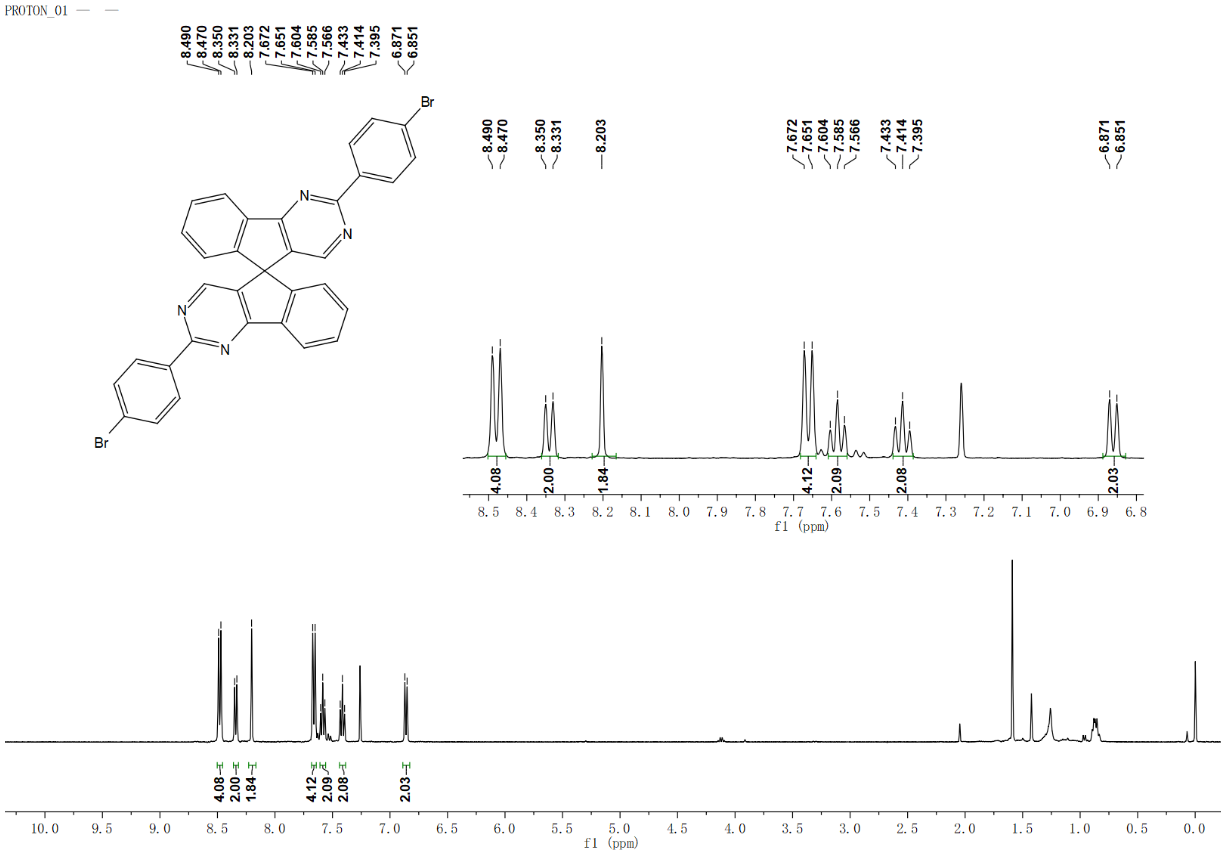

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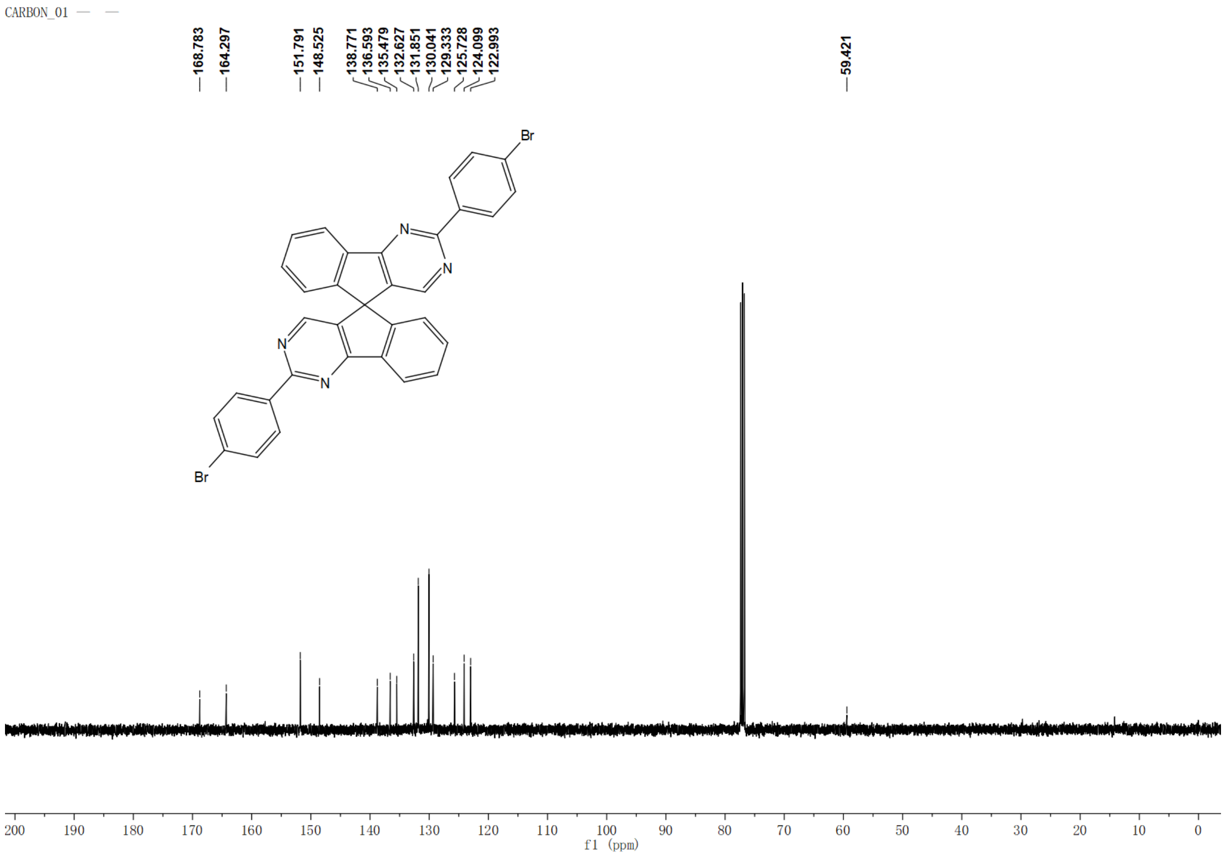

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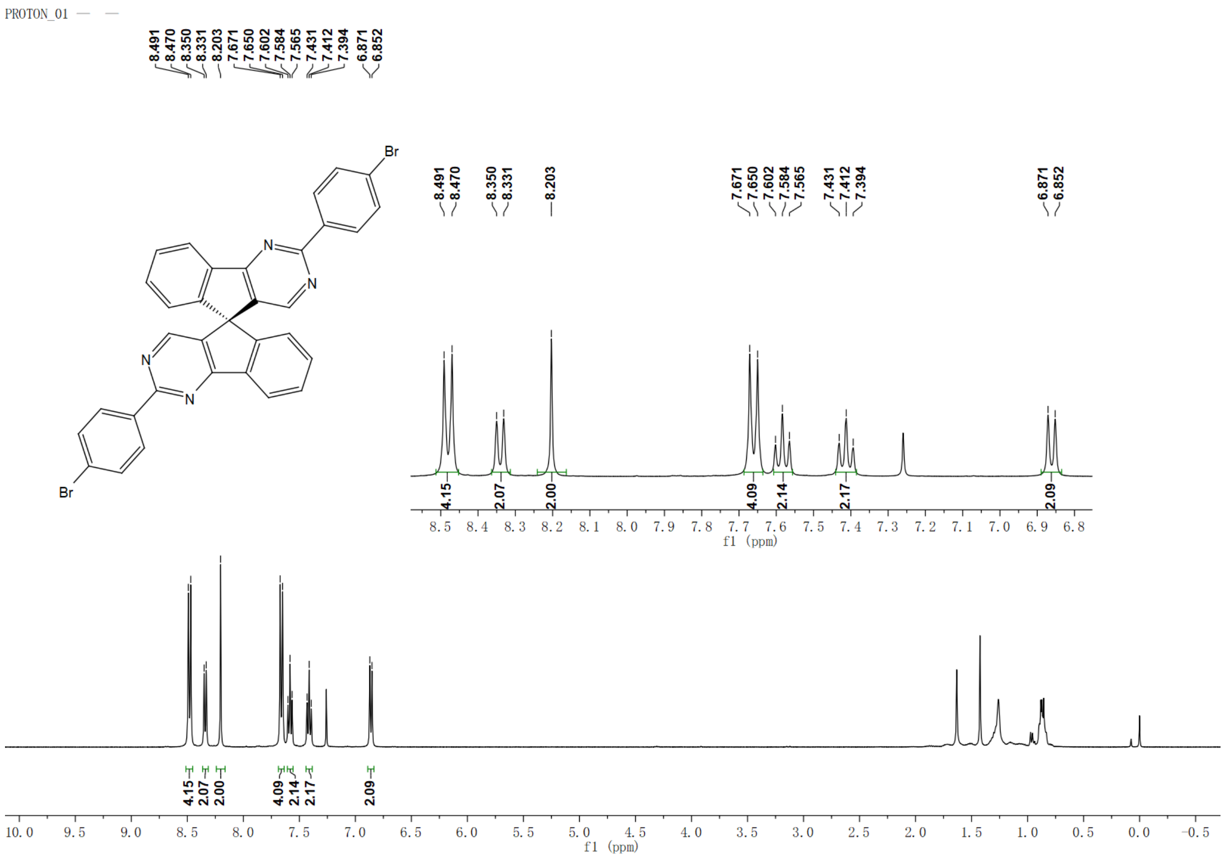

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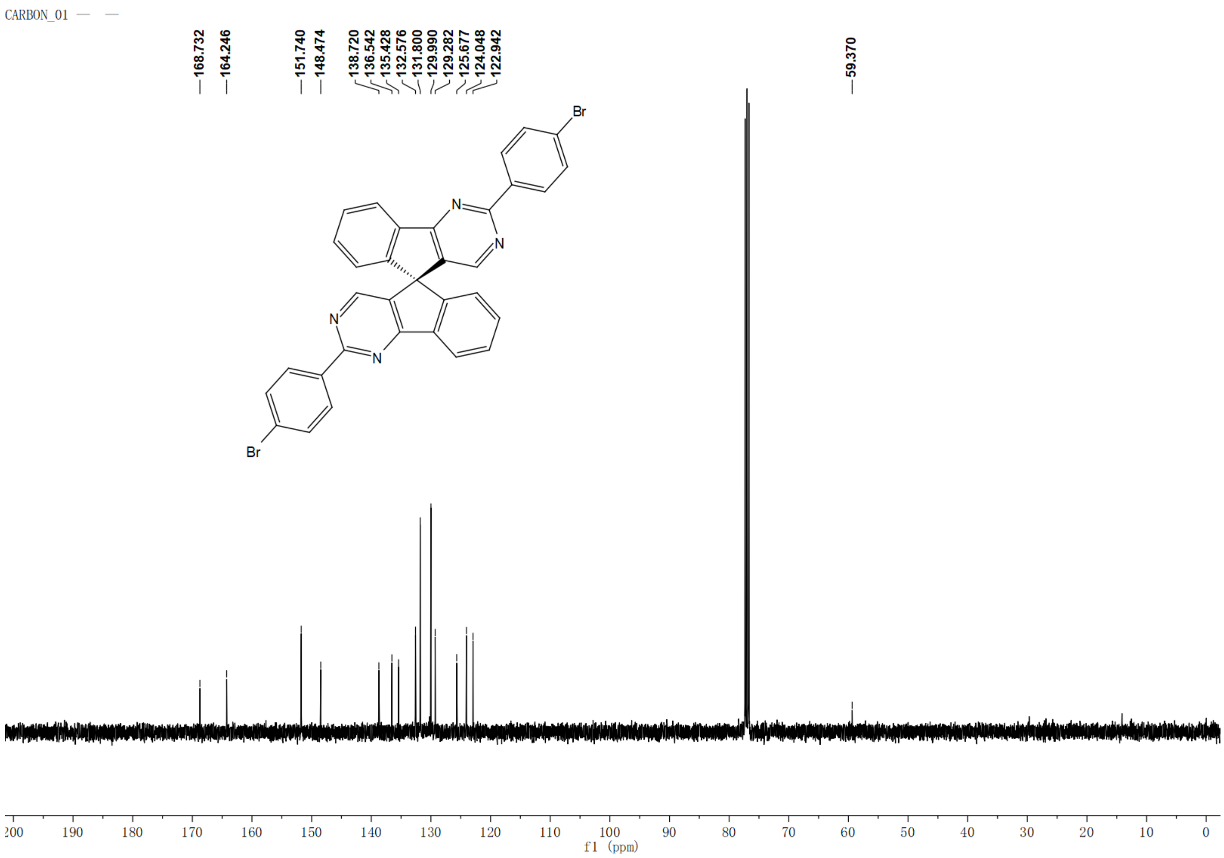

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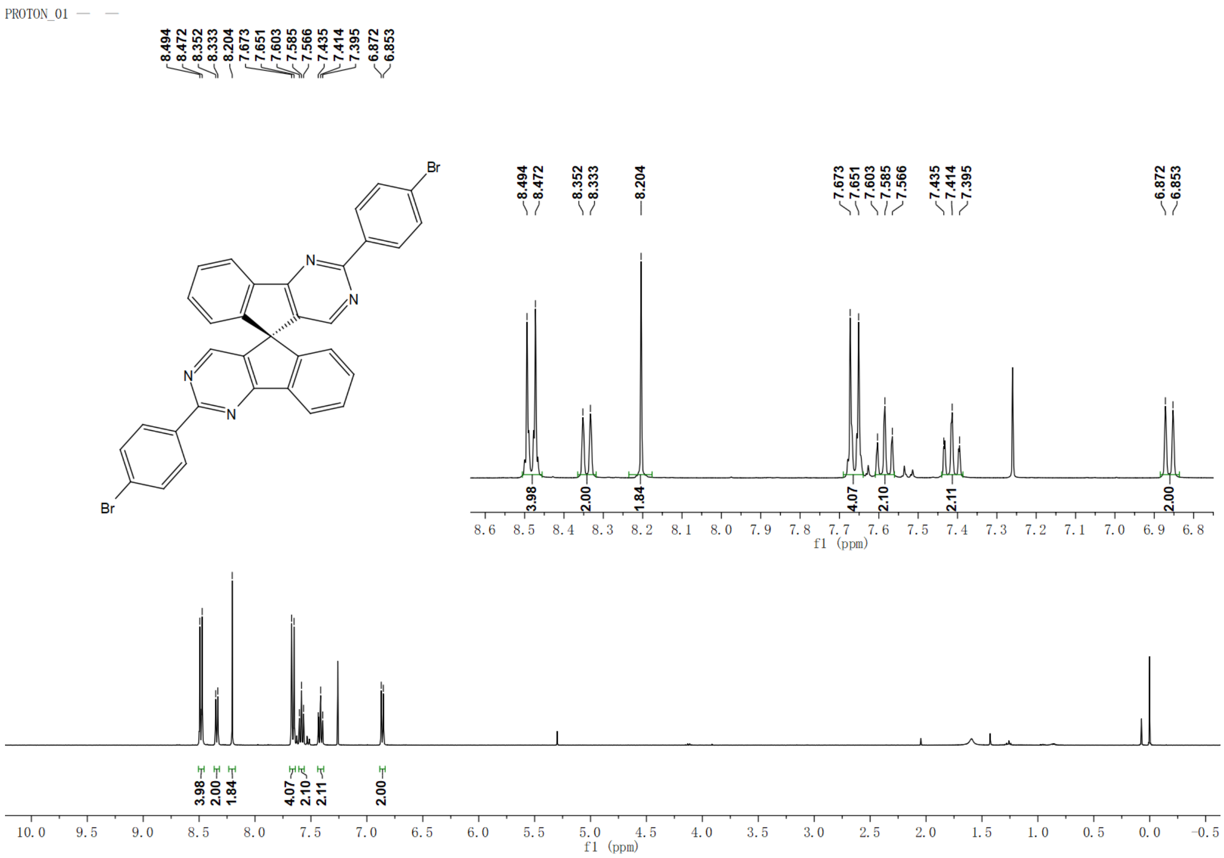

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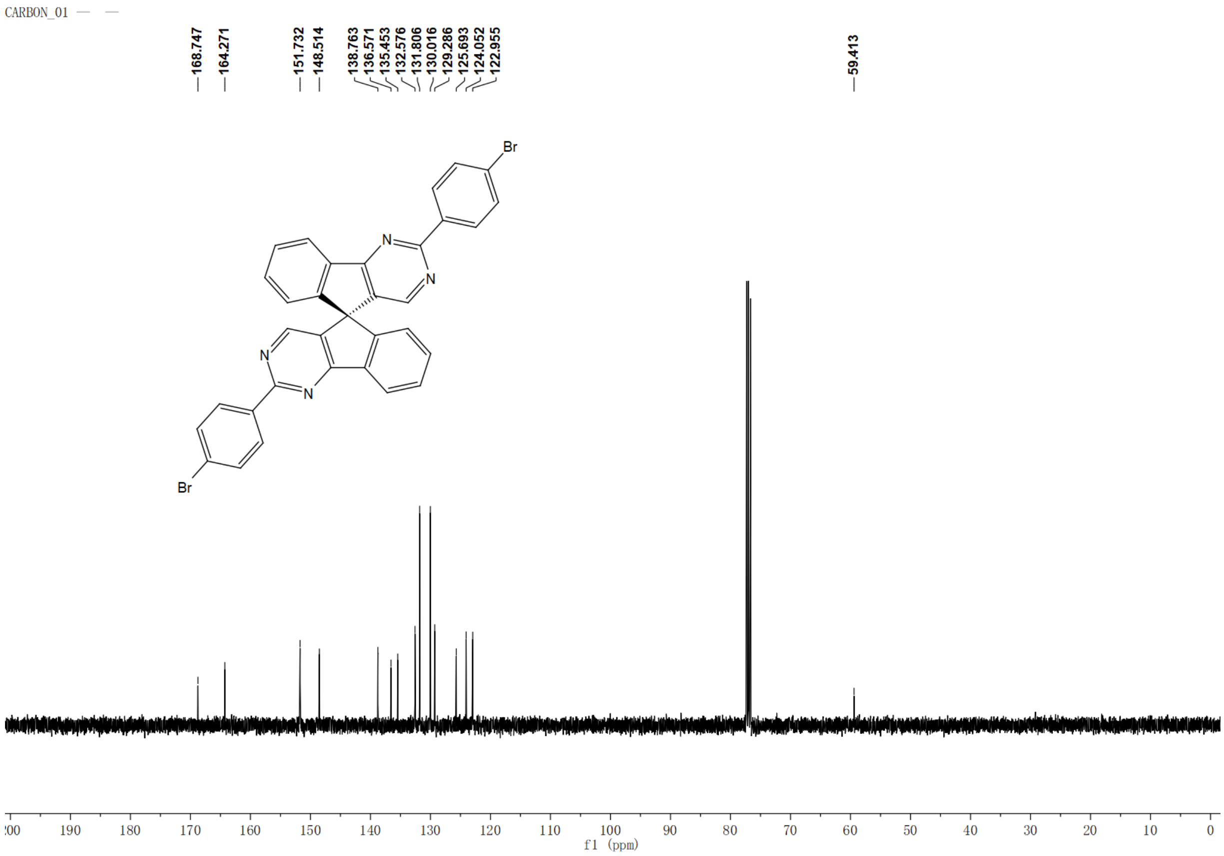

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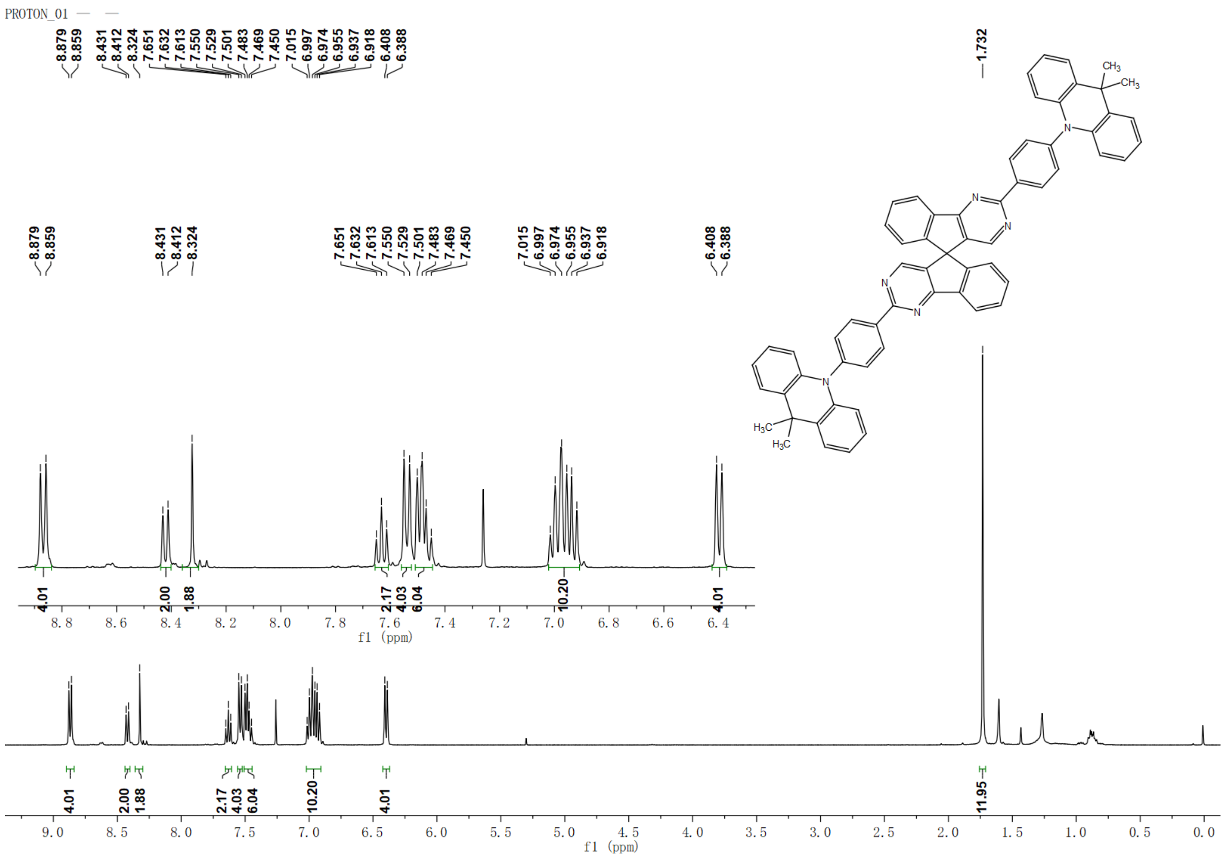

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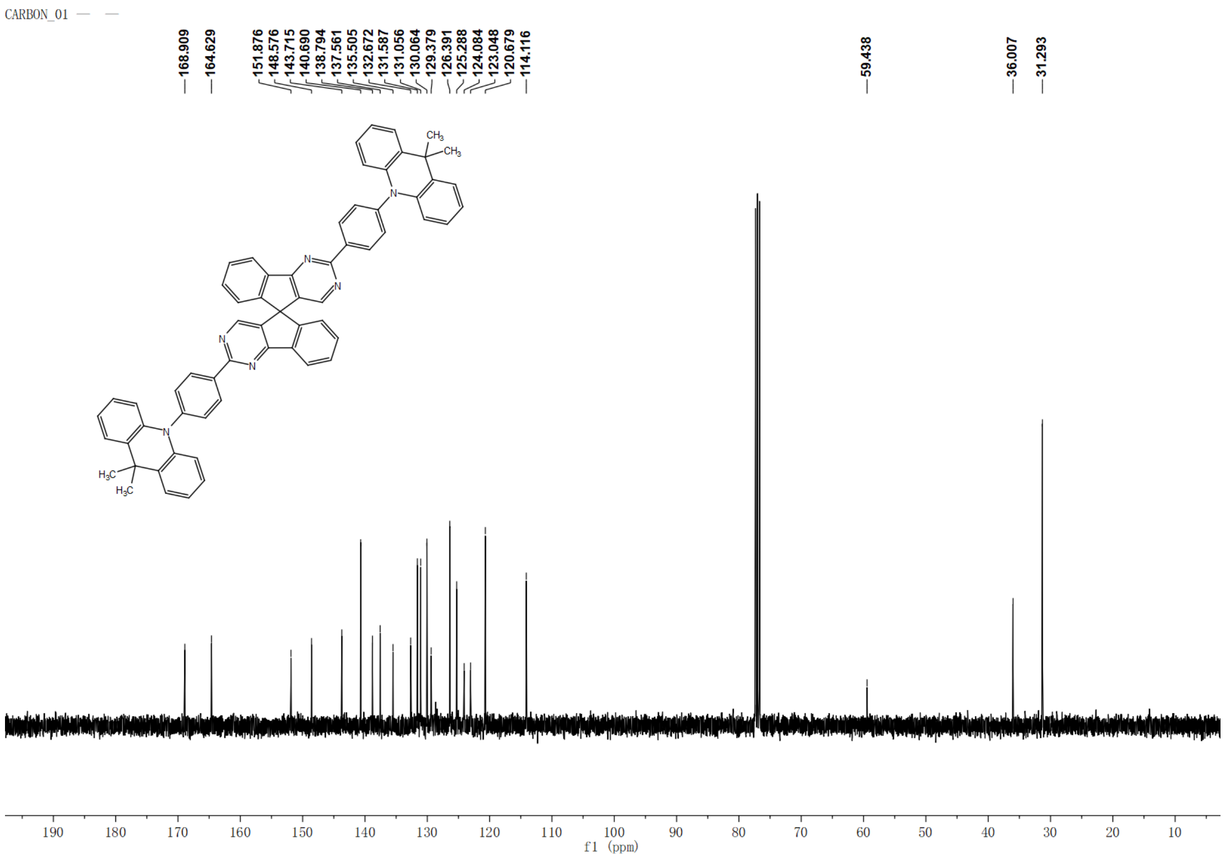
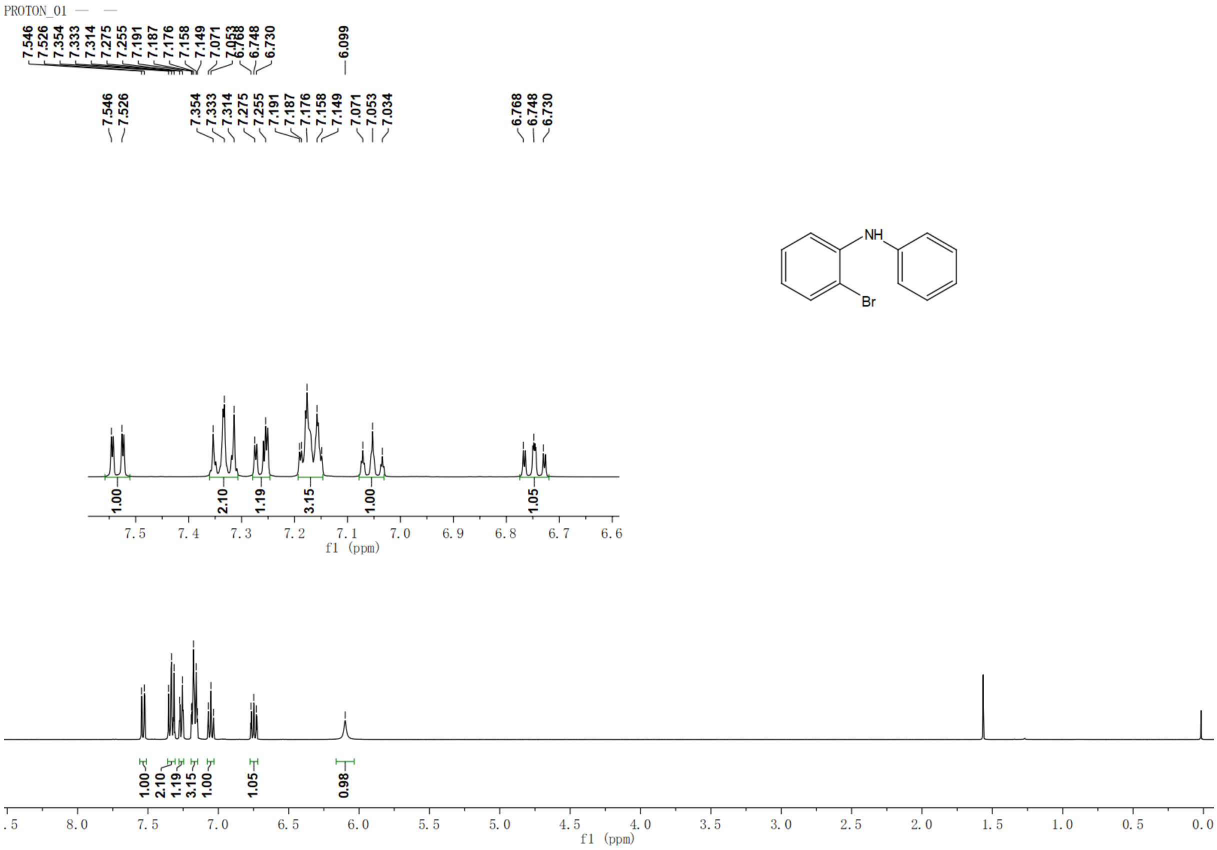
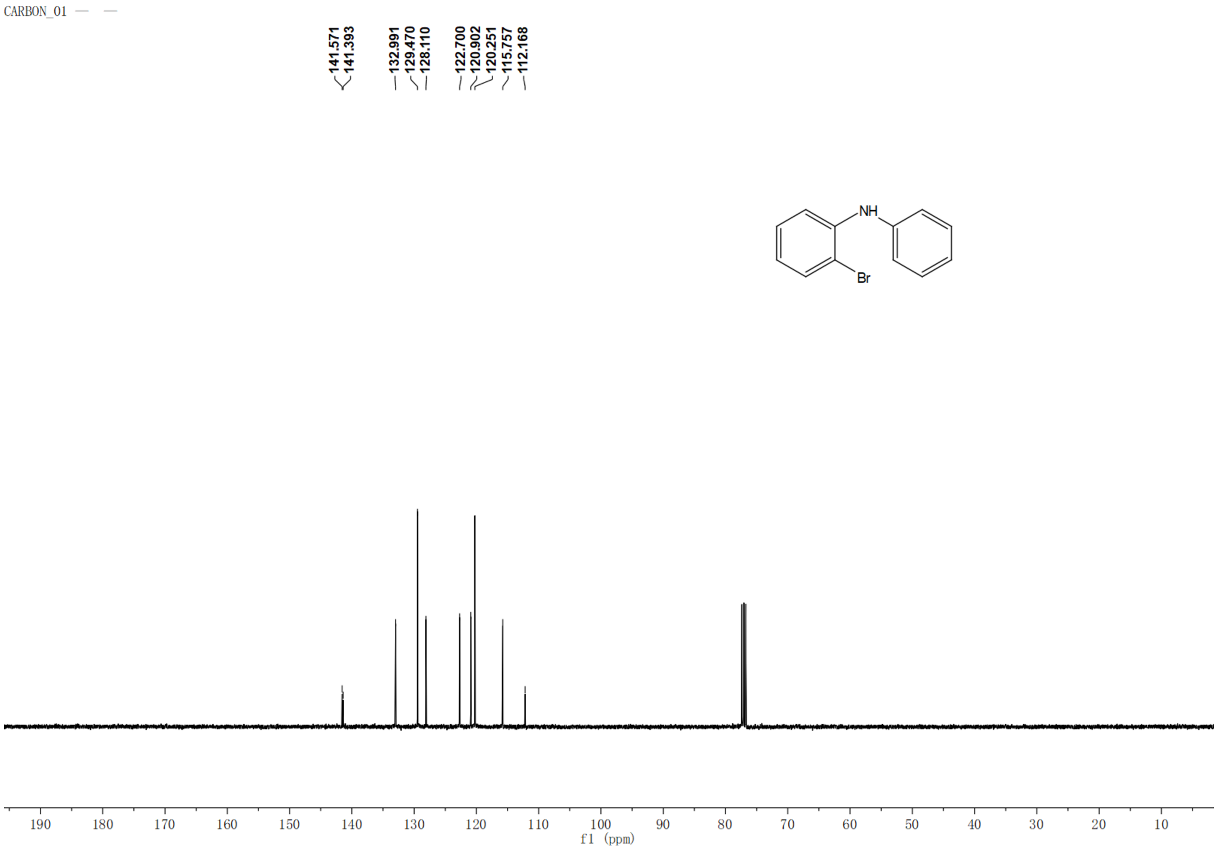


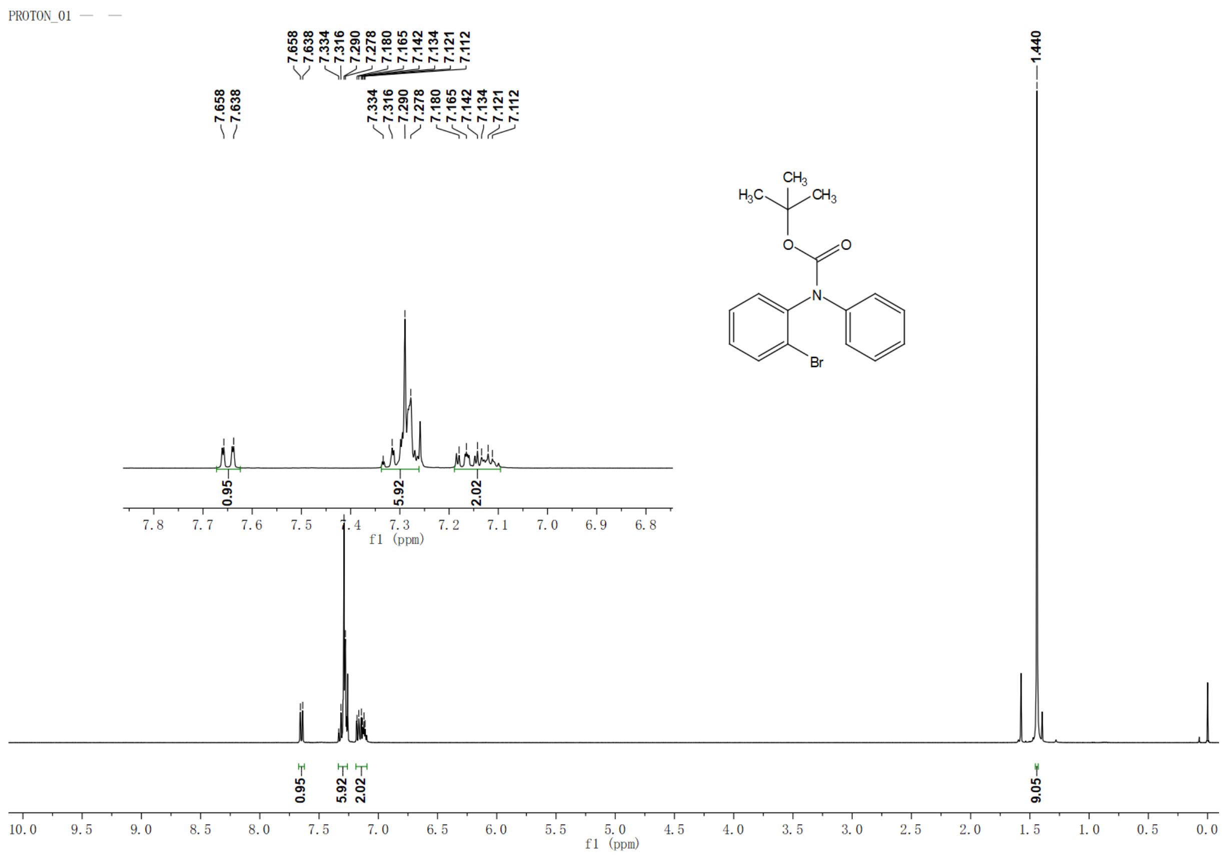
^{1}H NMR spectrum of **2-bromo-*N*-phenylaniline** ($CDCl_3$)



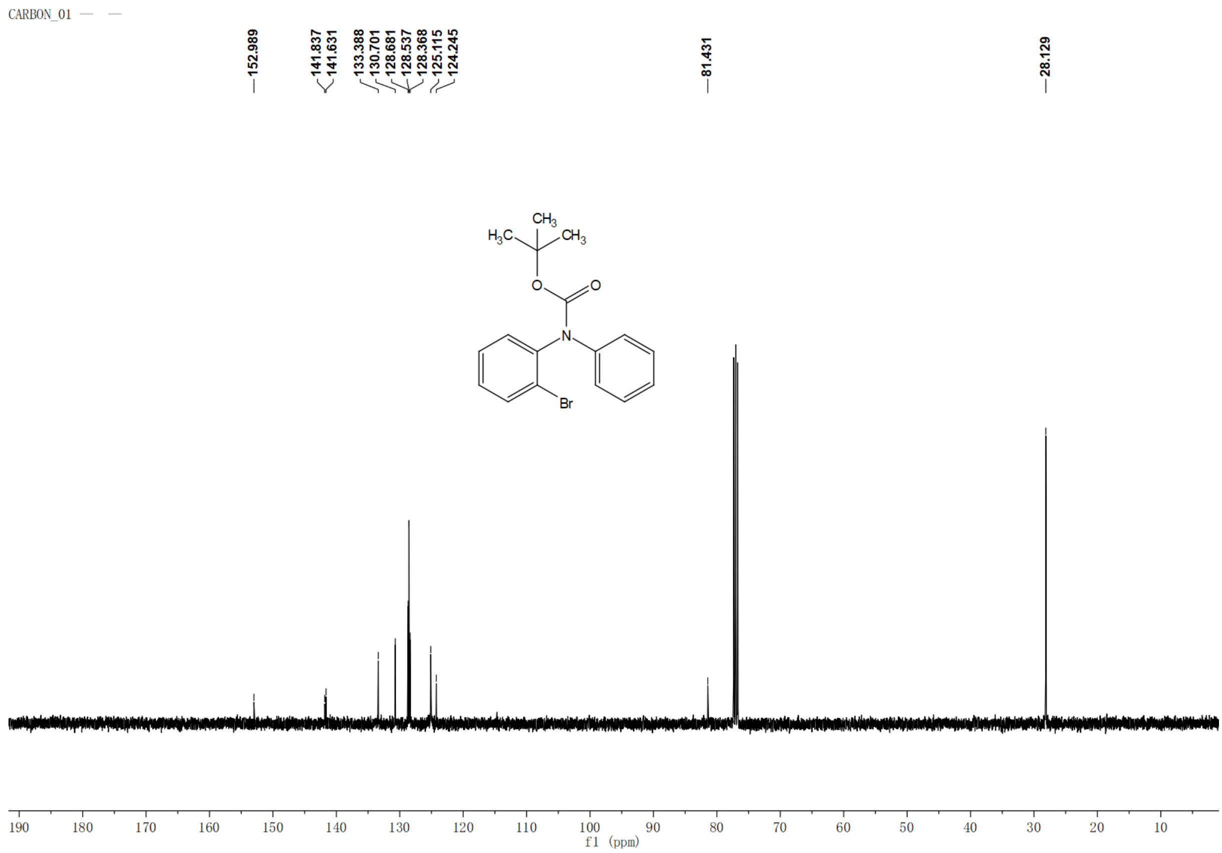
^{13}C NMR spectrum of **10*H*,10'*H*-9,9'-spirobi[acridine]** ($CDCl_3$)



[1]H NMR spectrum of ***t*-butyl (2-bromophenyl)(phenyl)carbamate** ($CDCl_3$)



[13]C NMR spectrum of ***t*-butyl (2-bromophenyl)(phenyl)carbamate** ($CDCl_3$)



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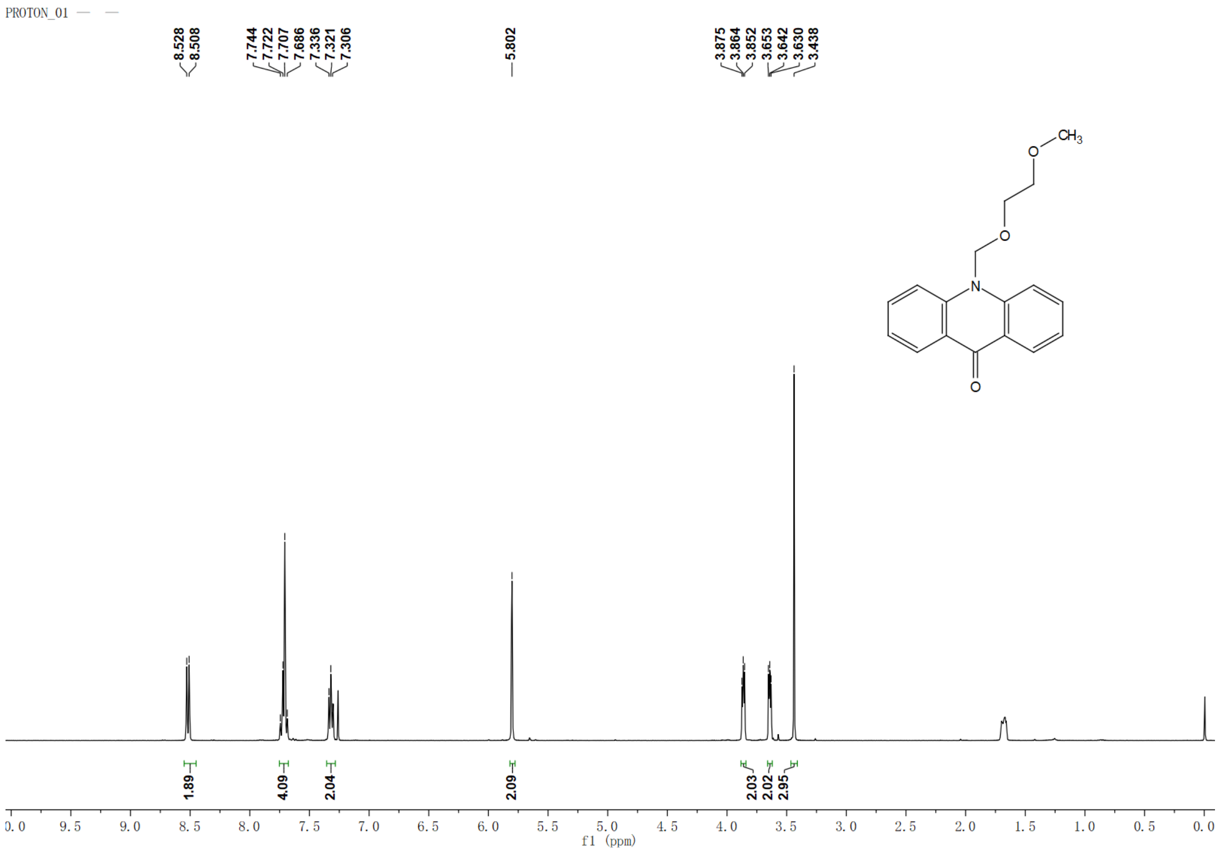
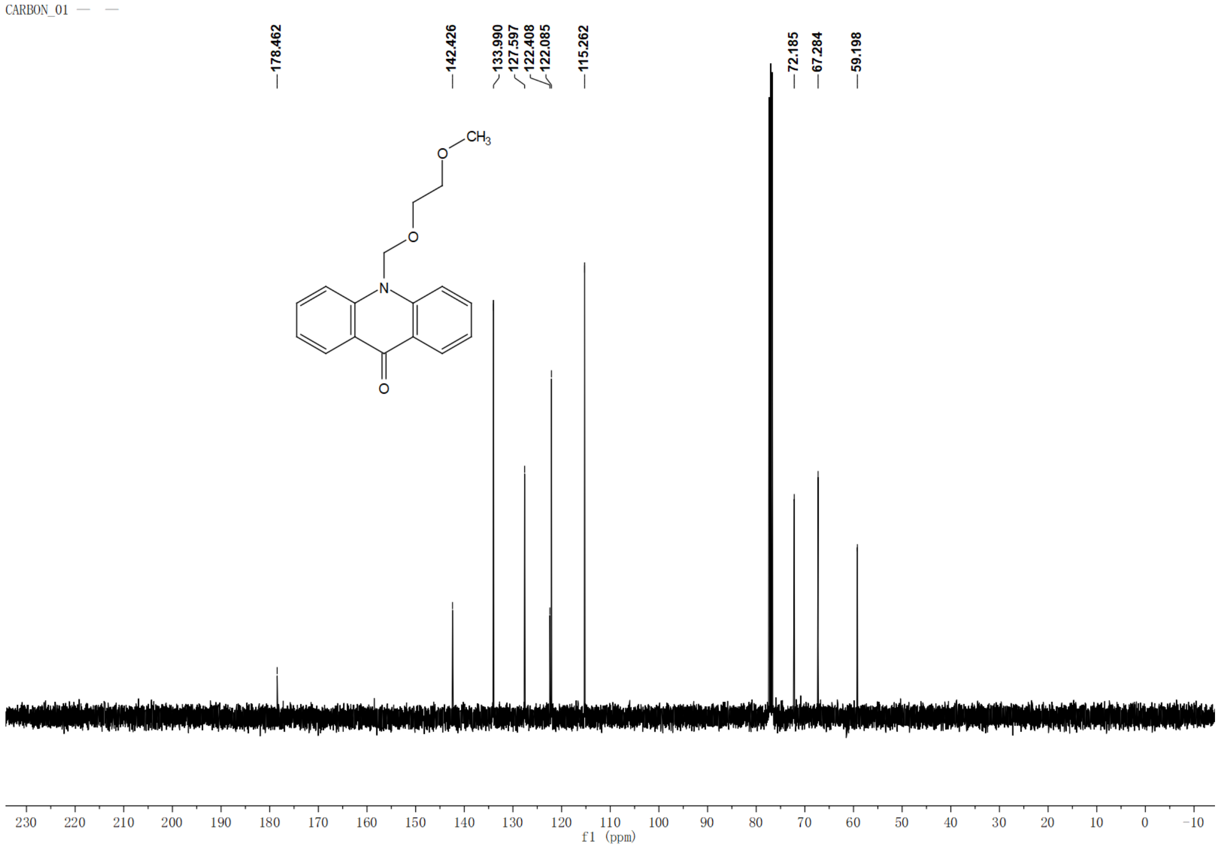
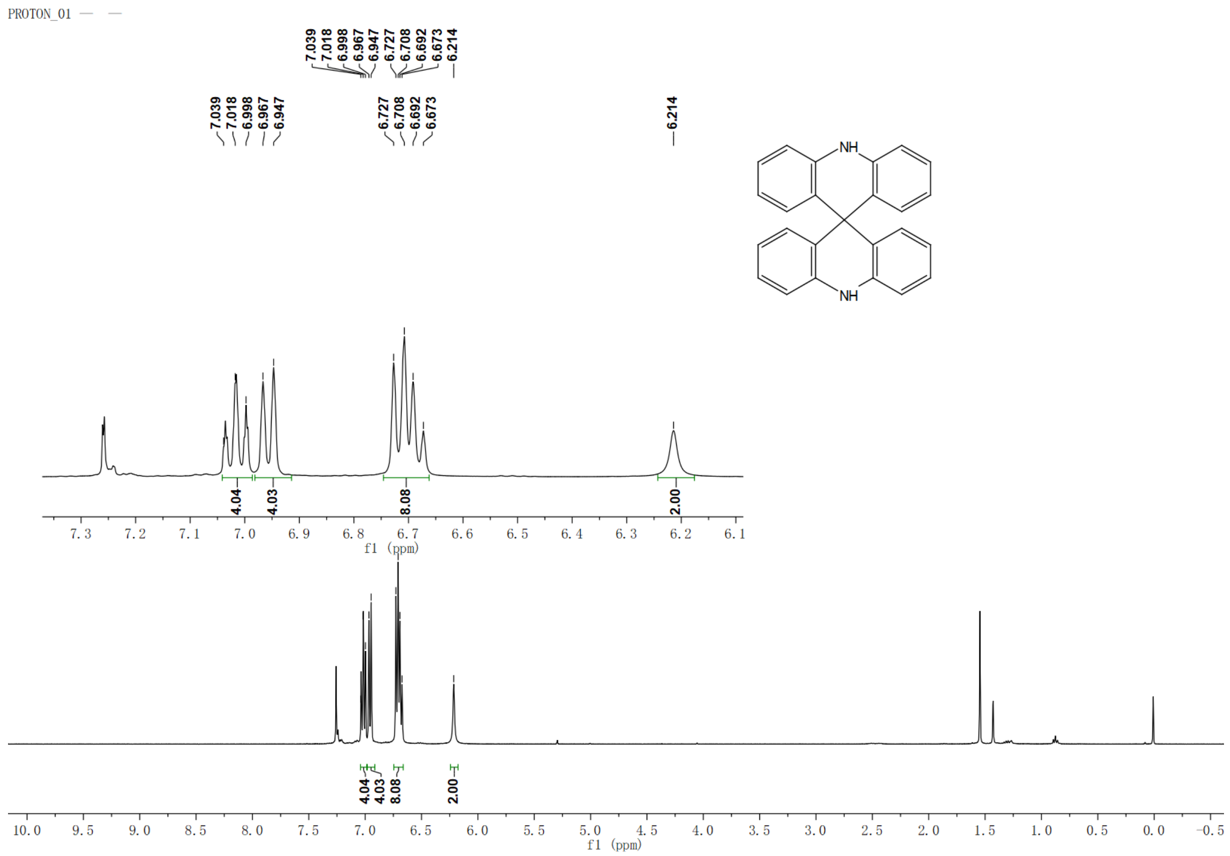


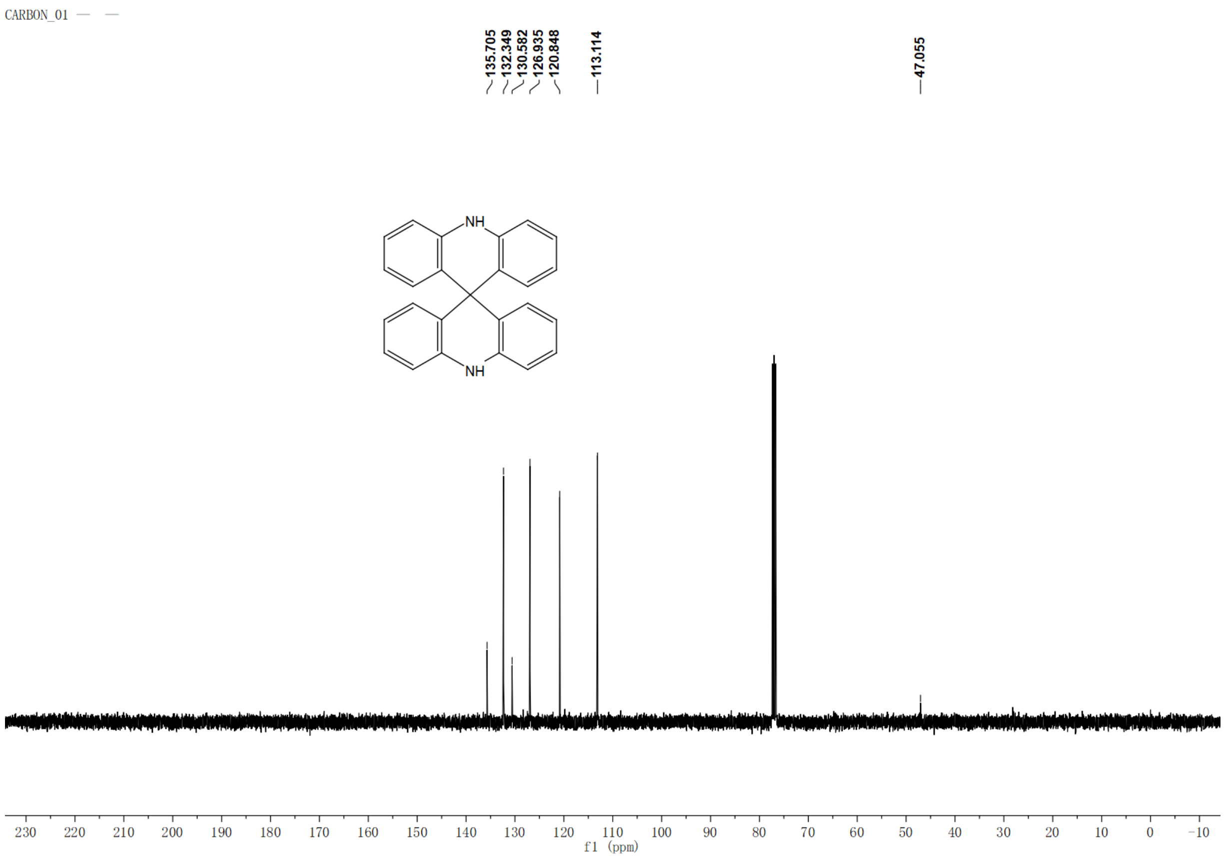
[13]C NMR spectrum of **10-((2-methoxyethoxy)methyl)acridin-9(10*H*)-one** ($CDCl_3$)



^{1}H NMR spectrum of **10*H*,10'*H*-9,9'-spirobi[acridine]** ($CDCl_3$)



^{13}C NMR spectrum of **10*H*,10'*H*-9,9'-spirobi[acridine]** ($CDCl_3$)



[1]H NMR spectrum of *R*/*S*-**9** ($CDCl_3$)