

## Supporting Information

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## 1. General Information

**General Methods.** <sup>1</sup>H NMR spectra were recorded on 400 spectrophotometers. Chemical shifts (δ) are reported in ppm from the resonance of tetramethyl silane as the internal standard (TMS: 0.00 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz) and integration. All NMR spectra were recorded on a Bruker spectrometer at 400 MHz (<sup>1</sup>H NMR), 100 MHz (<sup>13</sup>C NMR), 377 MHz (<sup>19</sup>F NMR). HRMS was recorded on Waters GCT Premier ESI-TOF. Enantiomeric ratio (ee) values were determined by chiral HPLC with chiral AD-H, IE-H, AS-H, AZ-H, IBN-5 columns with hexane and *i*-PrOH as solvents. Optical rotations were measured with a polarimeter. All air- and moisture-sensitive reactions were performed under an atmosphere of Ar in fire dried glassware. The light source used for photo-Wolff rearrangements in this study is purple Kessil lamp, 0-40 W for every light bulb (λ<sub>max</sub> = 370 nm). The manipulations for palladium-catalyzed reactions were carried out with standard Schlenk techniques under argon. Flash column chromatography was performed using 200-300 mesh silica gel.

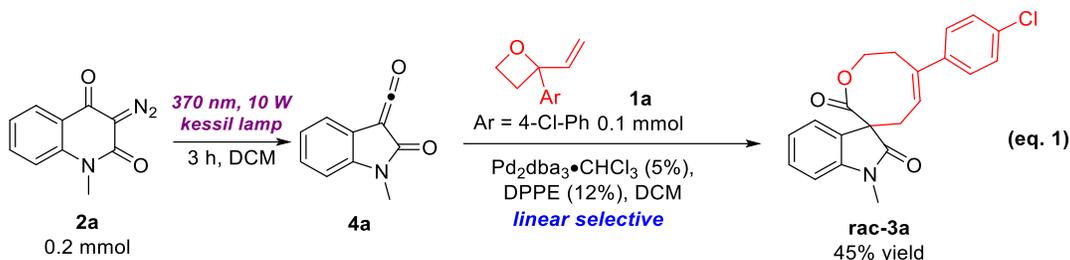
**Materials.** All the solvents were treated according to standard methods and all chemicals were used without purification. Anhydrous solvent (THF, DCM, MeCN, DMF, DMSO, MeOH and Toluene) were taken from JC-Meyer solvent purification system. Vinyl oxetanes **1a-1j**<sup>[1]</sup>, vinyl oxirane **5a**<sup>[2]</sup> and diazo compounds **2**<sup>[3]</sup> were prepared according to the known procedure. Chiral ligands **L1**, **L7** was commercial available; **L2**,<sup>[4]</sup> **L3-L6**<sup>[5]</sup> were synthesized according to previous methods.

## References

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## 2. Preliminary Exploration

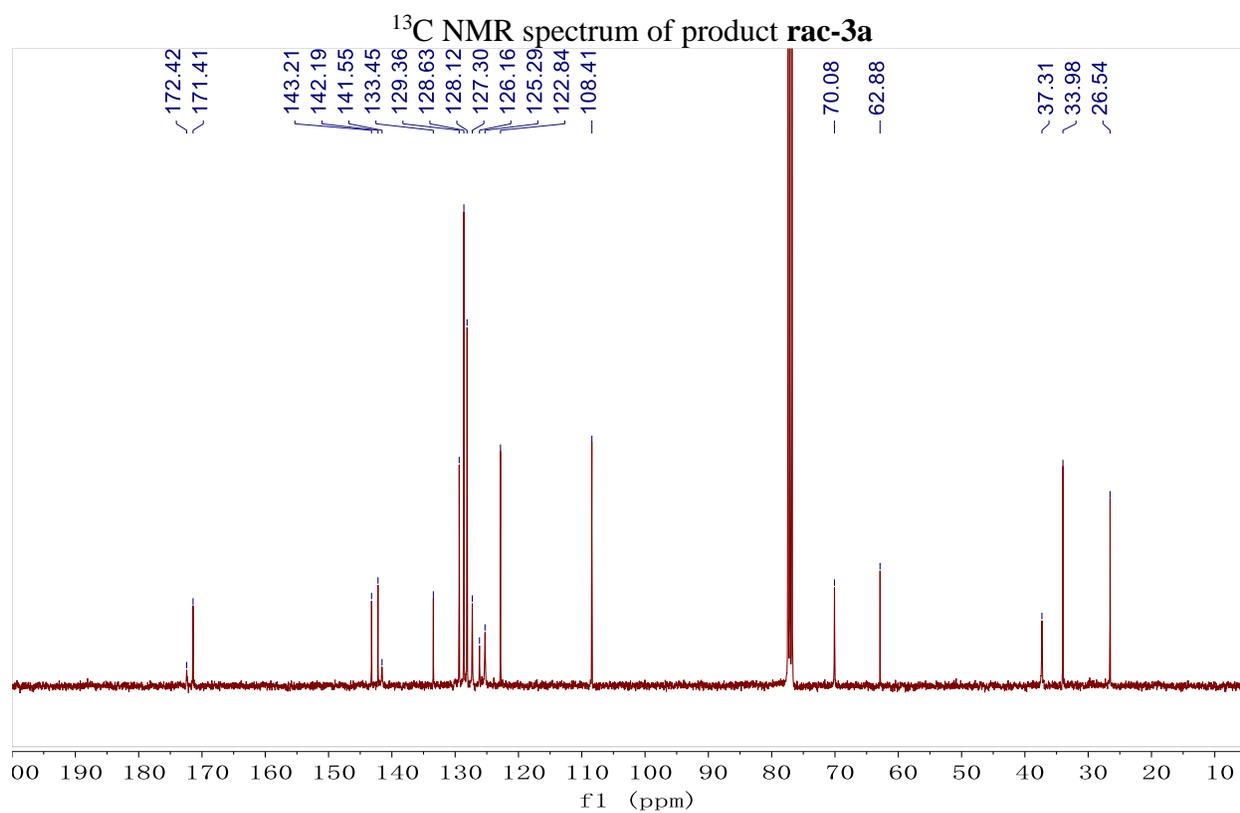
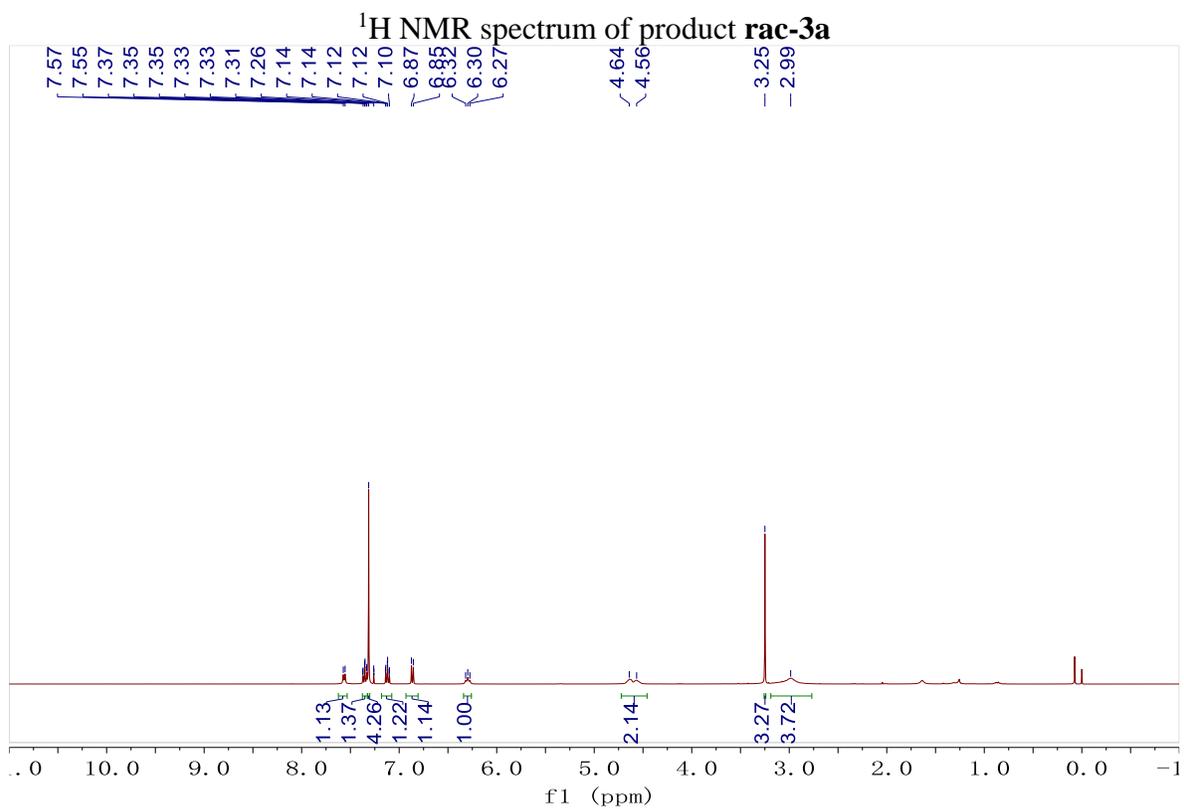
### 2.1 First trial to form spirooxindoles-fused 8 membered ring products



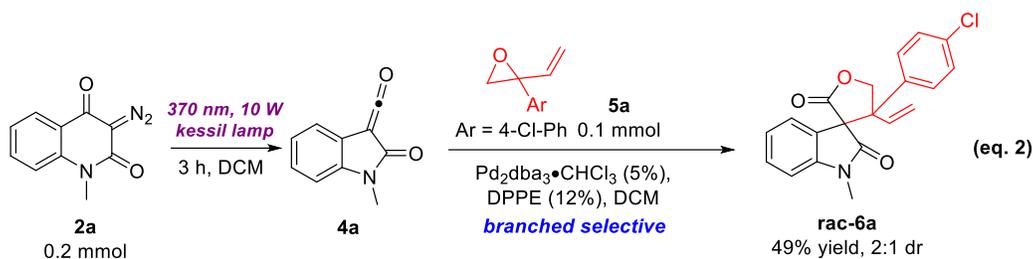
Under argon atmosphere, a flame-dried 10 mL Schlenk tube was charged with 3-diazoquinoline-2,4-diones **2a** (0.2 mmol, 2.0 equiv) and anhydrous DCM (1 mL). The resulting solution was stirred for 3 h at room temperature under the irradiation of 10 W 370 nm kessil lamp. To another a flame-dried 10 mL Schlenk tube was charged with Pd<sub>2</sub>(dba)<sub>3</sub>·CHCl<sub>3</sub> (5 mol %), DPPE (12 mol%) and DCM (1 mL) and the resulting solution was stirred for 30 mins at room temperature. After that, the reaction solution in the first Schlenk together with vinyl oxetanes **1a** (0.1 mmol, 1.0 equiv.) and another 1 mL DCM were added to the second one. Then the resulting solution was stirred for 12 h at 27 °C. Finally, the crude products were purified by flash silica gel chromatography (petrolether/ethyl acetate = 20:3) to give the products **rac-3a** in 45% yield.

#### 6'-(4-Chlorophenyl)-1-methyl-7',8'-dihydro-2'H,4'H-spiro[indoline-3,3'-oxocine]-2,2'-dione (**rac-3a**)

White solid, 45% yield, <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) 7.56 (d, *J* = 7.5 Hz, 1H), 7.38 - 7.33 (m, 1H), 7.31 (s, 4H), 7.14 - 7.12 (m, 1H), 6.86 (d, *J* = 7.7 Hz, 1H), 6.30 (t, *J* = 8.8 Hz, 1H), 4.60 (d, *J* = 30.2 Hz, 2H), 3.25 (s, 3H), 2.99 (s, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) 172.4, 171.4, 143.2, 142.2, 141.6, 133.5, 129.4, 128.6, 128.1, 127.3, 126.2, 125.3, 122.8, 108.4, 70.1, 62.9, 37.3, 34.0, 26.5. HRMS (ESI) for: C<sub>21</sub>H<sub>18</sub>ClNO<sub>3</sub> [M+H]<sup>+</sup>: calcd 368.1048, found 368.1049.



## 2.2 First trial to form spirooxindoles-fused 7 membered ring products

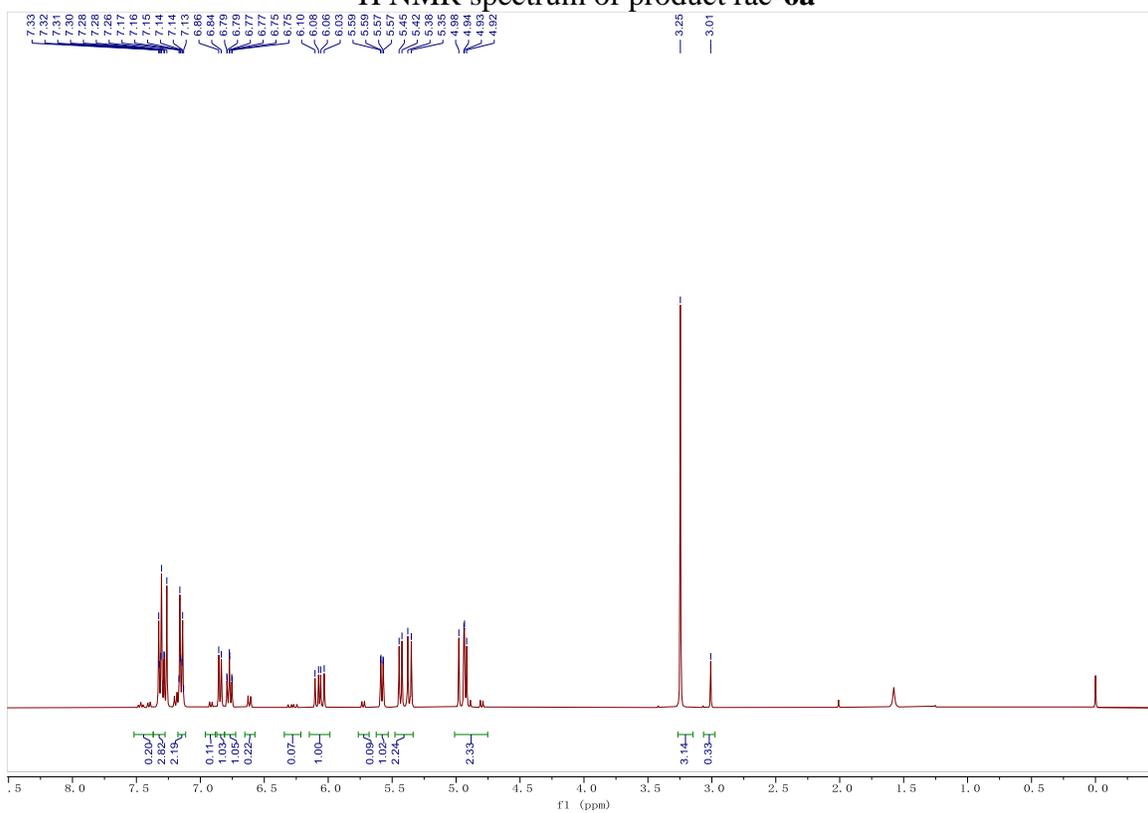


Under argon atmosphere, a flame-dried 10 mL Schlenk tube was charged with 3-diazoquinoline-2,4-diones **2a** (0.2 mmol, 2.0 equiv) and anhydrous DCM (1 mL). The resulting solution was stirred for 3 h at room temperature under the irradiation of 10 W 370 nm kessil lamp. To another flame-dried 10 mL Schlenk tube was charged with Pd<sub>2</sub>(dba)<sub>3</sub>·CHCl<sub>3</sub> (5 mol %), DPPE (12 mol%) and DCM (1 mL) and the resulting solution was stirred for 30 mins at room temperature. After that, the reaction solution in the first Schlenk together with vinyl oxirane **5a** (0.1 mmol, 1.0 equiv.) and another 1 mL DCM were added to the second one. Then the resulting solution was stirred for 12 h at 27 °C. Finally, the crude products were purified by flash silica gel chromatography (petrolether/ethyl acetate = 20:3) to give the products **rac-6a**. The 2:1 dr value was determined by the <sup>1</sup>H NMR analysis from the reaction mixture.

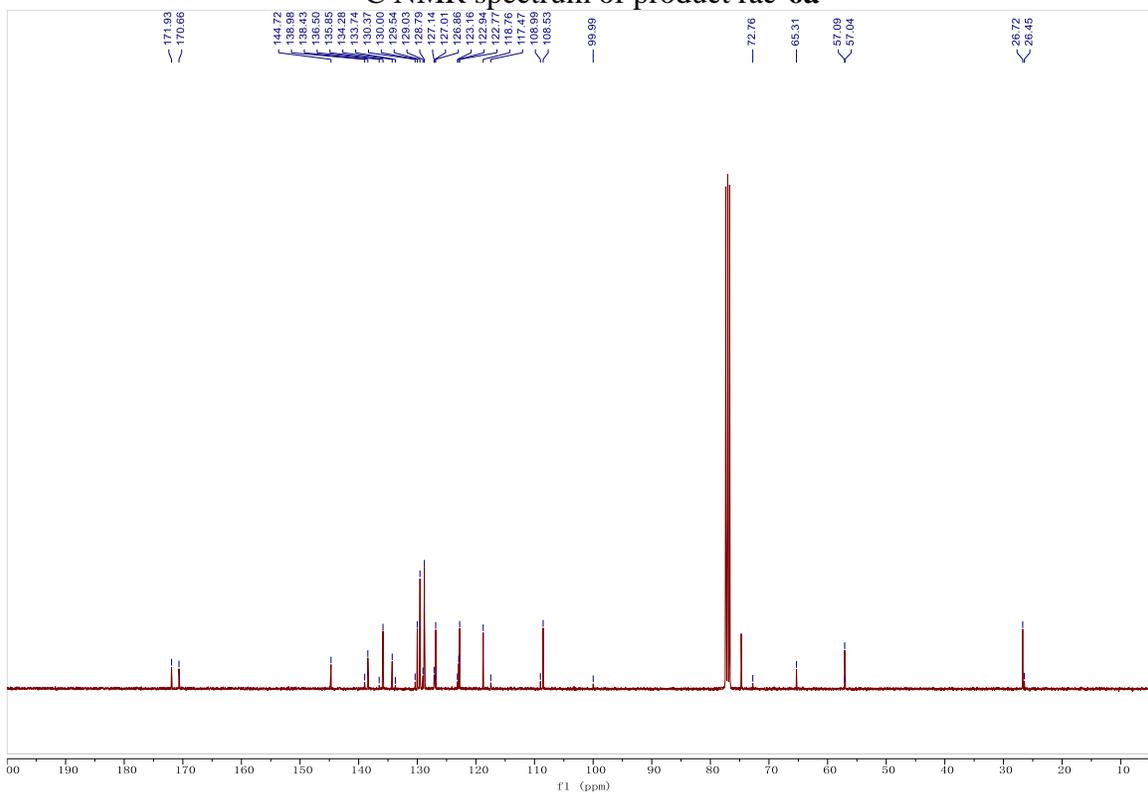
### 4-(4-Chlorophenyl)-1'-methyl-4-vinyl-4,5-dihydro-2H-spiro[furan-3,3'-indoline]-2,2'-dione (**rac-6a**)

White solid, 49% yield (10:1 dr after purification). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) 7.56-7.43 (m, 2H, minor), 7.39-7.23 (m, 3H, major), 7.26-7.08 (m, 2H, major + minor), 6.92 (d, *J* = 7.9 Hz, 1H, minor), 6.85 (d, *J* = 7.8 Hz, 1H, major), 6.75 (d, *J* = 1.1 Hz, 1H, major), 6.62 (d, *J* = 8.6 Hz, 2H, minor), 6.28 (dd, *J* = 17.2, 10.6 Hz, 1H, minor), 6.07 (dd, *J* = 17.6, 11.0 Hz, 1H, major), 5.73 (d, *J* = 8.2 Hz, 1H, minor), 5.58 (dd, *J* = 7.7, 1.2 Hz, 1H, major), 5.47-5.31 (m, 2H, major + minor), 5.07-4.69 (m, 2H major + minor), 3.25 (s, 3H, major), 3.01 (s, 3H, minor). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm, major + minor) 171.9, 170.7, 144.7, 139.0, 138.4, 136.5, 135.9, 134.3, 133.7, 130.4, 130.0, 129.5, 129.0, 128.8, 127.1, 127.0, 126.9, 123.2, 122.9, 122.8, 118.8, 117.5, 109.0, 108.5, 100.0, 72.8, 65.3, 57.1, 57.0, 26.7, 26.5. HRMS (ESI) for: C<sub>20</sub>H<sub>16</sub>ClNO<sub>3</sub> [M+Na]<sup>+</sup>: calcd 376.0711, found 376.0710.

### <sup>1</sup>H NMR spectrum of product rac-6a



### <sup>13</sup>C NMR spectrum of product rac-6a



## 2.3 Computational Details

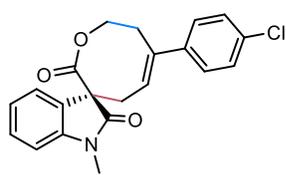
All of the calculations were performed using the Gaussian 16 program.<sup>1</sup> Structures were optimized at the (U)B3LYP level of density functional theory<sup>2</sup> with Grimme's D3(BJ) dispersion correction<sup>3</sup> in gas phase. For optimizations, Ahlrichs's def2SVP basis set was used for all atoms.<sup>4</sup> Frequency calculations have been performed to verify the optimized structures as local minima and to obtain Gibbs free energy at 298 K. To reduce error caused by the breakdown of the harmonic oscillator approximation, Truhlar's quasi-harmonic correction was used to compute molecular entropies by setting all positive frequencies that are less than 100 cm<sup>-1</sup> to 100 cm<sup>-1</sup>.<sup>5</sup> The electronic energies were further refined by carrying out single-point energy calculations using (U)B3LYP functional with Grimme's D3(BJ) dispersion correction. The def2TZVP basis set was applied for all atoms.<sup>4</sup> The SMD solvation model with DCM as the solvent was employed to account for solvation effect.<sup>6</sup> The three-dimensional (3D) structures were depicted using CYLview software.

## Reference

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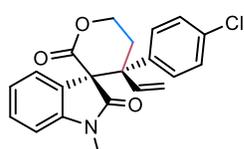
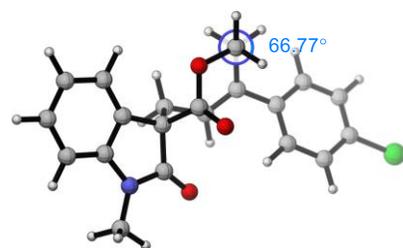
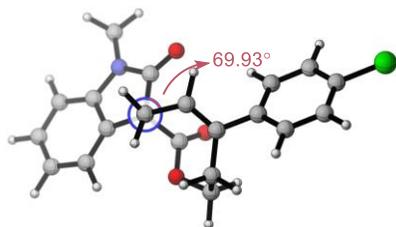
## Additional Computational Results

n = 1



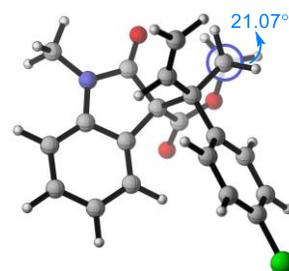
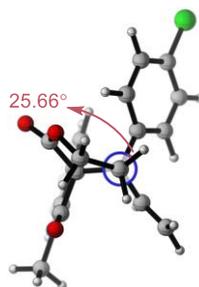
**3d**

$\Delta\Delta G = 0.0$  kcal/mol

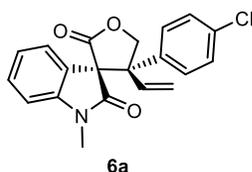


**3d'**

$\Delta\Delta G = 9.5$  kcal/mol

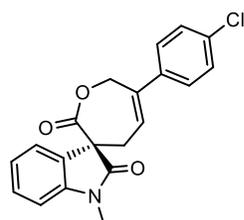
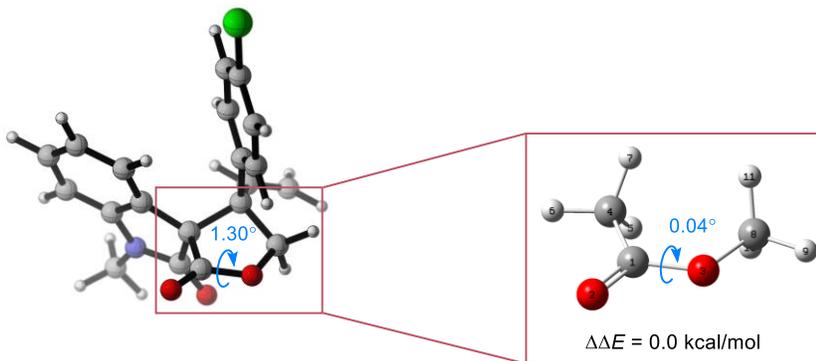


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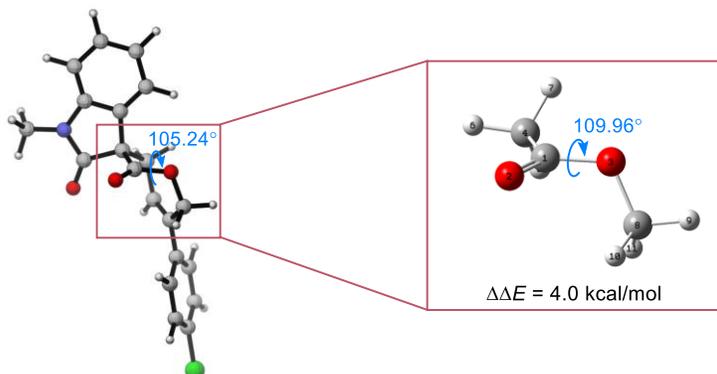
**6a**

$\Delta\Delta G = 0.0$  kcal/mol



**5a'**

$\Delta\Delta G = 2.9$  kcal/mol



**Figure S1** Comparison of thermal stabilities of different products. All energies are given in kcal/mol.

**XYZ coordinate**

3d'

E = -1552.26492444 G = -1551.965875

6	2.22330300	1.64435700	-0.07309600
6	0.98633700	1.08538400	-0.43097700
6	-0.08046000	1.91761400	-0.74269600
1	-1.03295900	1.50189800	-1.06798400
6	0.09200800	3.30777400	-0.65189700
1	-0.74153100	3.97037000	-0.89298000
6	1.32089800	3.84779000	-0.26459900
1	1.43993500	4.93176100	-0.19772700
6	2.41508500	3.01938600	0.02552500
1	3.38099800	3.44044900	0.30789100
6	2.62347700	-0.62085900	-0.08768800
6	1.10706700	-0.42307700	-0.41052100
6	0.76577100	-1.01842700	-1.79088500
6	0.83259400	-3.18974700	-0.72776300
1	1.91780900	-3.33677200	-0.72427700
1	0.34003900	-4.14245900	-0.96198300
6	0.77772800	-0.58219300	2.06449700
1	0.91547000	0.50328000	2.08462600
6	-1.22989200	-0.68606900	0.57099500
6	-2.01857400	-1.19944300	-0.47330000
1	-1.61297100	-1.93762800	-1.16297000
6	-3.33342200	-0.77775400	-0.67146800
1	-3.92609000	-1.18199700	-1.49307400
6	-3.89151100	0.16726700	0.19132300
6	-3.14187700	0.67794300	1.25062100
1	-3.58487500	1.40867600	1.92851700
6	-1.82585900	0.24977500	1.42973300
1	-1.26150600	0.66526100	2.26262500
6	4.54387900	0.83427500	0.49942700
1	5.02354900	-0.15020200	0.56802800
1	4.61657100	1.34546900	1.47328200
1	5.06398800	1.44198400	-0.25889200
17	-5.53605200	0.70269600	-0.05012500
7	3.16360000	0.62579100	0.13251600
8	3.23269900	-1.66800000	-0.01083300
8	0.67094200	-0.35021700	-2.78326100

8	0.51494000	-2.33663500	-1.83868400
6	0.37497000	-2.65816600	0.63708000
1	-0.58619700	-3.10541400	0.92355900
1	1.12266300	-3.00761300	1.35823500

6	1.07216700	-1.26994700	3.16825100
1	1.44557900	-0.75537200	4.05734900
1	0.95363000	-2.35296800	3.24688500
6	0.24379800	-1.11359400	0.74337600

3d

E = -1552.28110903 G = -1551.980965

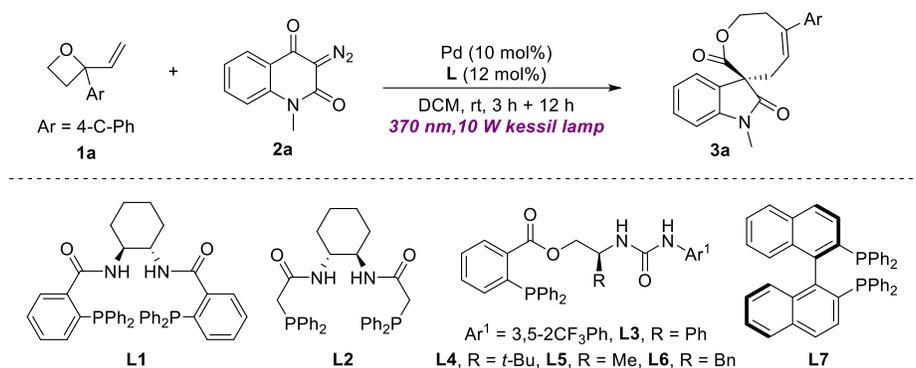
6	3.97341600	0.86857500	-0.04021100
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1	3.40278500	-2.49694200	-0.13834100
6	5.35590000	-1.53819800	-0.03946600
1	5.90962500	-2.47925800	-0.03432400
6	6.04580900	-0.32482500	0.02450000
1	7.13712800	-0.32608200	0.07716400
6	5.36372200	0.90199900	0.02667100
1	5.90751800	1.84622800	0.08109100
6	1.76397000	1.50954600	-0.14262000
6	1.79011100	-0.03615200	-0.17273700
6	1.02717100	-0.60516700	1.02817500
6	-0.34833400	-2.54821900	1.08238800
1	-0.91280600	-1.98746000	1.84257500
1	-0.17846700	-3.57303000	1.44089300
6	-1.05028700	-2.54125600	-0.27985600
1	-0.40928000	-3.08451200	-0.99245500
1	-1.98575100	-3.11290000	-0.18766100
6	-1.34068100	-1.13456000	-0.77751700
6	-0.41655800	-0.32993100	-1.34586100
1	-0.73844100	0.67011600	-1.64262300
6	1.06387300	-0.57554500	-1.45193900
1	1.48952700	-0.06637000	-2.32983500
1	1.30168500	-1.64435400	-1.53569500

6	-2.69521200	-0.58412600	-0.50239600	6	-2.78504400	-0.43506000	-0.16974300
6	-2.82819400	0.72081900	0.01008200	6	-3.54846000	-0.36859200	-1.34940800
1	-1.92908700	1.30197500	0.22699800	1	-3.06797800	-0.57961200	-2.30682000
6	-4.08364300	1.26763600	0.27312700	6	-4.90735200	-0.05963300	-1.32009800
1	-4.17787600	2.27561600	0.67947000	1	-5.49088200	-0.01526800	-2.24070200
6	-5.22918200	0.50680000	0.02778300	6	-5.52796300	0.18225200	-0.09205500
6	-5.12735200	-0.79405200	-0.47200800	6	-4.79821800	0.11791300	1.09721600
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6	-3.86556700	-1.33046800	-0.72611300	6	-3.43943500	-0.19226000	1.05193400
1	-3.79795000	-2.34299200	-1.13073700	1	-2.87717900	-0.21077600	1.98735700
6	3.44962900	3.33575900	-0.01211500	6	3.88445000	3.08553800	0.16310100
1	2.52600500	3.92750200	-0.05113700	1	3.03544400	3.77665500	0.24338700
1	4.08921100	3.60048500	-0.87044700	1	4.49233700	3.35539400	-0.71653800
1	3.99302400	3.56899200	0.91819000	1	4.51128100	3.17191500	1.06574000
17	-6.80695100	1.18268300	0.35897100	17	-7.23173800	0.56302600	-0.04317200
7	3.07655700	1.94262500	-0.05478000	7	3.34924200	1.75207700	0.03409500
8	0.79261300	2.22899500	-0.22130100	8	1.10330200	2.29711000	0.05075700
8	0.53369300	0.02436000	1.92012800	8	1.20273500	-0.13971700	2.20703100
8	0.94466500	-1.95398400	0.90523800	8	0.58145500	-1.83089000	0.84181500
				6	-1.33387800	-0.74769400	-0.22855000
				6	-0.53314700	-0.15017800	-1.13493500
5a'				1	-0.98778900	0.61906000	-1.76361000
E = -1512.93791154	G = -1512.666828			6	-0.84231800	-1.76455000	0.78104200
6	4.11167800	0.58954900	-0.11631200	1	-1.18734700	-2.77644500	0.51605400
6	3.26602300	-0.52804600	-0.24372600	1	-1.23948400	-1.53484000	1.78386700
6	3.79745200	-1.79902700	-0.40090100				
1	3.13931300	-2.66739600	-0.47871500				
6	5.19418500	-1.94768100	-0.44707700	6a			
1	5.63068800	-2.94050200	-0.57267800	E = -1512.94243328	G = -1512.671455		
6	6.02562000	-0.83128600	-0.32720700	6	-2.14155700	1.63469600	-0.11997300
1	7.11001900	-0.96003800	-0.36230900	6	-0.96663200	1.04209900	0.37841000
6	5.49718000	0.45847000	-0.15821300	6	0.12032300	1.83910000	0.71342200
1	6.15297800	1.32496600	-0.06175900	1	1.02443200	1.39864600	1.13313900
6	1.99084100	1.47760300	0.00738400	6	0.03390300	3.22622400	0.51459100
6	1.83093800	-0.05424700	-0.14103800	1	0.88318400	3.86157500	0.77313700
6	1.18625300	-0.63365100	1.11854500	6	-1.12998300	3.79692200	-0.00598000
6	0.94127200	-0.40645200	-1.36004300	1	-1.18237400	4.87776600	-0.15655700
1	1.29093800	0.17791400	-2.22311400	6	-2.24429800	3.00691400	-0.32707700
1	1.11501200	-1.46562200	-1.59964400	1	-3.15973200	3.45673200	-0.71397800

6	-2.67498800	-0.59498300	0.04881300	1	-1.31384100	-2.40641800	-3.66035600
6	-1.17981900	-0.44249100	0.45636800	6	1.17845500	-0.90413300	-0.38583700
6	-0.99620200	-1.08000600	1.84339300	6	1.70031300	-0.05090300	-1.36999300
6	-0.28123500	-1.37674500	-0.45246500	6	2.01510300	-1.25137800	0.68801200
6	-4.47181100	0.90954000	-0.77180900	6	3.00030100	0.44704300	-1.29191200
1	-5.00096500	-0.05156000	-0.79966000	1	1.08602100	0.24601500	-2.21885100
1	-4.47259100	1.35731400	-1.77890100	6	3.31840900	-0.76018500	0.78223500
1	-4.99346300	1.59209700	-0.08093200	1	1.65471000	-1.90283900	1.48404800
7	-3.12346600	0.65461800	-0.32339200	6	3.80633400	0.09295600	-0.20884300
8	-3.32477800	-1.61736300	0.04374900	1	3.38870300	1.11155800	-2.06465400
8	-1.14802600	-0.56219400	2.90883700	1	3.95485500	-1.03656100	1.62382900
8	-0.62186100	-2.36836700	1.68427500	17	5.43284700	0.71683000	-0.09556500
6	-0.77735400	-1.40011200	-1.87981500	6	-0.48929900	-2.71113700	0.29626300
1	-1.00191000	-0.41749700	-2.30794900	1	0.36138300	-3.39721800	0.19913500
6	-0.95332400	-2.48654900	-2.63190600	1	-1.41537100	-3.20019100	-0.02970000
1	-0.74968000	-3.49566800	-2.26368800				

### 3. Details for Condition Optimizations

**Table S1:** The effect of ligands and Pd source.<sup>[a]</sup>



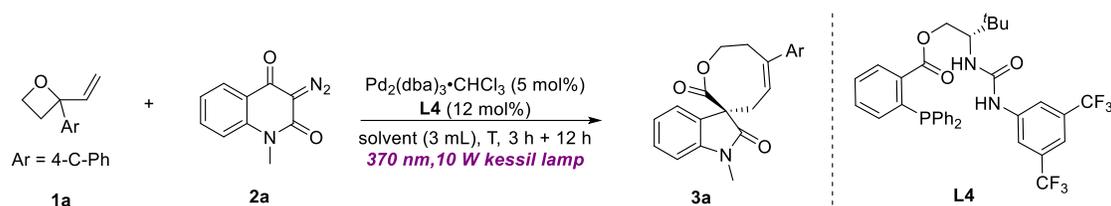
Entry	Ligand	Pd cat.	Yield (%) <sup>[b]</sup>	ee (%) <sup>[c]</sup>
1	<b>L1</b>	Pd <sub>2</sub> (dba) <sub>3</sub> •CHCl <sub>3</sub> (5%)	16%	-60%
2	<b>L2</b>	Pd <sub>2</sub> (dba) <sub>3</sub> •CHCl <sub>3</sub> (5%)	15%	63%
3	<b>L3</b>	Pd <sub>2</sub> (dba) <sub>3</sub> •CHCl <sub>3</sub> (5%)	63%	80%
<b>4</b>	<b>L4</b>	<b>Pd<sub>2</sub>(dba)<sub>3</sub>•CHCl<sub>3</sub> (5%)</b>	<b>75%</b>	<b>87%</b>
5	<b>L5</b>	Pd <sub>2</sub> (dba) <sub>3</sub> •CHCl <sub>3</sub> (5%)	60%	74%
6	<b>L6</b>	Pd <sub>2</sub> (dba) <sub>3</sub> •CHCl <sub>3</sub> (5%)	46%	80%
7	<b>L7</b>	Pd <sub>2</sub> (dba) <sub>3</sub> •CHCl <sub>3</sub> (5%)	29%	31%
13	<b>L4</b>	Pd <sub>2</sub> (dba) <sub>3</sub> (5%)	66%	87%
14	<b>L4</b>	Pd(PPh <sub>3</sub> ) <sub>4</sub> (10%)	N.R.	--
14	<b>L4</b>	Pd(PPh <sub>3</sub> ) <sub>2</sub> Cl <sub>2</sub> (10%)	9%	0%

<sup>[a]</sup>Unless noted otherwise, reactions were performed with **2a** (0.2 mmol, 2.0 equiv.) in dry DCM (1 mL) was irradiated at rt under 10 W 370 nm kessil lamp for 3 h, the resulting solution **2a-1** together with **1a** (0.1 mmol) in another 1 mL DCM were added to the pre-prepared solution of Pd cat. (10 mol%) and chiral ligand (12 mol%) in dry DCM and stirred at 27 °C for 12 hour. <sup>[b]</sup>Determined by <sup>1</sup>H NMR. <sup>[c]</sup>Determined by HPLC analysis on a chiral stationary phase. dba: dibenzylideneacetone.

**Table S2:** The effect of the ratio of **1d** and **2a** and solvent volume<sup>[a]</sup>.

Entry	Ratio of <b>1d/2a</b>	Volume (mL)	Yield (%) <sup>[b]</sup>	ee (%) <sup>[c]</sup>
1	1 : 1	3	36%	89%
2	1 : 2	3	75%	87%
3	1 : 3	3	18%	51%
4	2 : 1	3	45%	89%
5	1 : 2	6	31%	86%

<sup>[a]</sup>Unless noted otherwise, reactions were performed with **2a** (0.2 mmol, 2.0 equiv.) in dry DCM (1 mL) was irradiated at rt under 10 W 370 nm kessil lamp for 3 h, the resulting solution **2a-1** together with **1a** (0.1 mmol) in another 1 mL DCM were added to the pre-prepared solution of Pd cat. (10 mol%) and chiral ligand (12 mol%) in dry DCM and stirred at 27 °C for 12 hour. <sup>[b]</sup>Determined by <sup>1</sup>H NMR. <sup>[c]</sup>Determined by HPLC analysis on a chiral stationary phase. dba: dibenzylideneacetone.

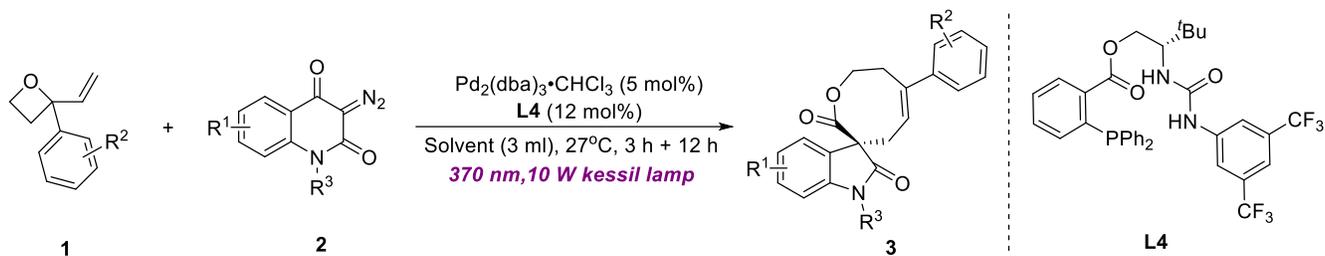
**Table S3:** The effect of solvent and temperature.<sup>[a]</sup>

Entry	Solvent (3 mL)	T (°C)	Yield (%) <sup>[b]</sup>	ee (%) <sup>[c]</sup>
1	DCM	27	75%	87%
2	DCE	27	31%	84%
3	$\text{CHCl}_3$	27	N.R.	--
4	MeCN	27	N.R.	--
5 <sup>[d]</sup>	DCM:DCE (1:2)	27	62%	88%
6 <sup>[d]</sup>	DCM:THF (1:2)	27	16%	86%
7 <sup>[d]</sup>	DCM:Toluene (1:2)	27	34%	77%
8 <sup>[d]</sup>	DCM:MeCN (1:2)	27	61%	94%
9 <sup>[d]</sup>	DCM:MeCN (1.5:1.5)	27	72%	93%
10 <sup>[d]</sup>	DCM:MeCN (2:1)	27	40%	85%
11 <sup>[d]</sup>	DCM:MeCN (1.5:1.5)	35	35%	93%
12 <sup>[d]</sup>	DCM:MeCN (1.5:1.5)	45	14%	93%
12 <sup>[d]</sup>	DCM:PhCN (1.5:1.5)	27	80%	86%
<b>13<sup>[e]</sup></b>	<b>DCM:MeCN:PhCN (1:1:1)</b>	<b>27</b>	<b>78% (75%)</b>	<b>92%</b>

<sup>[a]</sup>Unless noted otherwise, reactions were performed with **2a** (0.2 mmol, 2.0 equiv.) in dry DCM (1 mL) was irradiated at rt under 10 W 370 nm kessil lamp for 3 h, the resulting solution **2a-1** together with **1a** (0.1 mmol) in another 1 mL DCM were added to the pre-prepared solution of Pd cat. (10 mol%) and chiral ligand (12 mol%) in dry DCM and stirred at 27 °C for 12 hour. <sup>[b]</sup>Determined by <sup>1</sup>H NMR. <sup>[c]</sup>Determined by HPLC analysis on a chiral stationary phase. dba: dibenzylideneacetone. <sup>[d]</sup>Reactions were performed with **2a** (0.2 mmol, 2.0 equiv.) in dry DCM (1 mL) was irradiated at rt under 10 W 370 nm kessil lamp for 3 h, the resulting solution **2a-1** together with **1a** (0.1 mmol) in 1 mL another solvent were added to the pre-prepared solution of Pd cat. (10 mol%) and chiral ligand (12 mol%) in dry solvent and stirred at 27 °C for 12 hours. <sup>[d]</sup>Reactions were performed with **2a** (0.2 mmol, 2.0 equiv.) in dry DCM (1 mL) was irradiated at rt under 10 W 370 nm kessil lamp for 3 h, the resulting solution **2a-1** together with **1a** (0.1 mmol) in 1 mL PhCN were added to the pre-prepared solution of Pd cat. (10 mol%) and chiral ligand (12 mol%) in dry MeCN and stirred at 27 °C for 12 hours.

## 4. General Procedures and Characterization of Products

### 4.1 General procedures for the preparation of lactones products

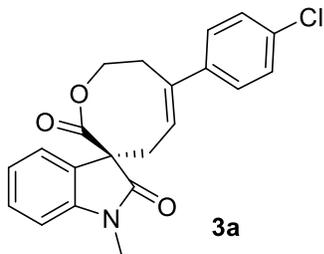


**General procedure A** ( $\text{DCM}:\text{MeCN}:\text{PhCN} = 1:1:1$ , 3 mL) : Under argon atmosphere, a flame-dried 10 mL Schlenk tube was charged with 3-diazoquinoline-2,4-diones **2** (0.2 mmol, 2.0 equiv) and anhydrous DCM (1 mL). The resulting solution was stirred for 3 h at room temperature under the irradiation of 10 W 370 nm kessil lamp. To another a flame-dried 10 mL Schlenk tube was charged with  $\text{Pd}_2(\text{dba})_3 \cdot \text{CHCl}_3$  (5 mol %), chiral ligand **L4** (12 mol%) and MeCN (1 mL) and the resulting solution was stirred for 30 mins at room temperature. After that, the reaction solution in the first Schlenk together with vinyl oxetanes **1** (0.1 mmol, 1.0 equiv.) and 1 mL PhCN were added to the second one. Then the resulting solution was stirred for 12 h at 27 °C. Finally, the crude products were purified by flash silica gel chromatography (petrolether/ethyl acetate = 20:3) to give the products **3**. All the ee values were determined by chiral HPLC analysis of purified products. The racemic products were synthesized using racemic **L3** as ligand.

**General procedure B** ( $\text{DCM} = 3 \text{ mL}$ ) : Under argon atmosphere, a flame-dried 10 mL Schlenk tube was charged with 3-diazoquinoline-2,4-diones **2** (0.2 mmol, 2.0 equiv) and anhydrous DCM (1 mL). The resulting solution was stirred for 3 h at room temperature under the irradiation of 10 W 370 nm kessil lamp. To another a flame-dried 10 mL Schlenk tube was charged with  $\text{Pd}_2(\text{dba})_3 \cdot \text{CHCl}_3$  (5 mol %), chiral ligand **L4** (12 mol%) and DCM (1 mL) and the resulting solution was stirred for 30 mins at room temperature. After that, the reaction solution in the first Schlenk together with vinyl oxetanes **1** (0.1 mmol, 1.0 equiv.) and another 1 mL DCM were added to the second one. Then the resulting solution was stirred for 12 h at 35 °C. Substrate **1b** was performed with **2a** at 27 °C to improve the ee value of **3b**. Finally, the crude products were purified by flash silica gel chromatography (petrolether/ethyl acetate = 20:3) to give the products **3**. All the ee values were determined by chiral HPLC analysis of purified products. The racemic products were synthesized using racemic **L3** as ligand.

## 4.2 Characteration of tetrahydrofurans products

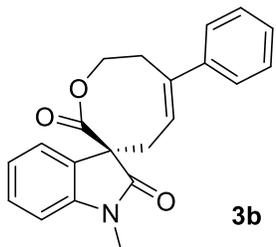
### (*R,E*)-6'-(4-chlorophenyl)-1-methyl-7',8'-dihydro-2'*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (**3a**)



**General procedure A:** White solid, 72% yield, 92% ee,  $[\alpha]_D^{25} = 73.93$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 85:15 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 31.69 min,  $t_R$  (minor) = 17.14 min.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.56 (d,  $J = 7.5$  Hz, 1H), 7.38 - 7.33 (m, 1H), 7.31 (s, 4H), 7.14 - 7.12 (m, 1H), 6.86 (d,  $J = 7.7$  Hz, 1H), 6.30 (t,  $J = 8.8$  Hz, 1H), 4.60 (d,  $J = 30.2$  Hz,

2H), 3.25 (s, 3H), 2.99 (s, 4H).;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.4, 171.4, 143.2, 142.2, 141.6, 133.5, 129.4, 128.6, 128.1, 127.3, 126.2, 125.3, 122.8, 108.4, 70.1, 62.9, 37.3, 34.0, 26.5. HRMS (ESI) for:  $\text{C}_{21}\text{H}_{18}\text{ClNO}_3$   $[\text{M}+\text{H}]^+$ : calcd 368.1048, found 368.1049.

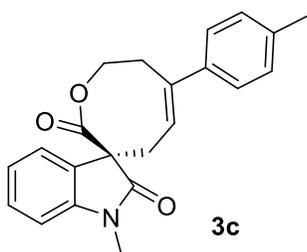
### (*R,E*)-1-methyl-6'-phenyl-7',8'-dihydro-2'*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (**3b**)



**General procedure B:** White solid, 68% yield, 90% ee,  $[\alpha]_D^{25} = 49.93$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 38.05 min,  $t_R$  (minor) = 16.45 min;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.58 (d,  $J = 7.4$  Hz, 1H), 7.44 - 7.28 (m, 6H), 7.12 (t,  $J = 7.6$  Hz, 1H), 6.86 (d,  $J = 7.8$  Hz, 1H), 6.29 (t,  $J = 8.8$  Hz, 1H), 4.63 (s, 2H), 3.25 (d,  $J = 1.1$  Hz, 3H), 3.09 (s, 2H), 2.95 (s, 2H).;  $^{13}\text{C NMR}$

(100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.4, 171.4, 143.8, 143.2, 142.8, 129.3, 128.5, 127.5, 126.7, 126.6, 125.5, 122.8, 108.3, 70.2, 62.9, 37.5, 34.1, 26.5. HRMS (ESI) for:  $\text{C}_{21}\text{H}_{19}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : calcd 334.1438, found 334.1433.

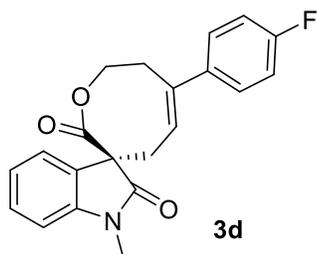
### (*R,E*)-1-methyl-6'-(*p*-tolyl)-7',8'-dihydro-2'*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (**3c**)



**General procedure A:** White solid, 63% yield, 95% ee,  $[\alpha]_D^{25} = 55.93$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 85:15 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 40.83 min,  $t_R$  (minor) = 15.09 min.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.57 (d,  $J = 7.4$  Hz, 1H), 7.34 (t,  $J = 7.8$  Hz, 1H), 7.28 (d,  $J = 7.7$  Hz, 2H), 7.21 - 7.05 (m, 3H), 6.86 (d,  $J = 7.8$  Hz, 1H), 6.26 (t,  $J = 8.8$  Hz, 1H), 4.61 (s, 2H), 3.24 (d,  $J = 1.6$  Hz,

3H), 3.07 (s, 2H), 2.92 (s, 2H), 2.36 (s, 3H).;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.2, 171.3, 143.1, 142.5, 140.8, 137.2, 129.1, 129.1, 126.0, 126.3, 125.6, 125.4, 122.7, 108.2, 62.8, 37.4, 33.9, 26.5, 21.1.; HRMS (ESI) for:  $\text{C}_{22}\text{H}_{21}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : calcd 348.1594, found 348.1598.

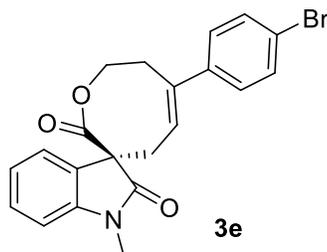
**(*R,E*)-6'-(4-fluorophenyl)-1-methyl-7',8'-dihydro-2'*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3d)**



**General procedure A:** White solid, 74% yield, 93% ee,  $[\alpha]_D^{25} = 57.27$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 42.43 min,  $t_R$  (minor) = 22.31 min.  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.58 (d,  $J = 7.5$  Hz, 1H), 7.44 - 7.32 (m, 3H), 7.26 (d,  $J = 1.6$  Hz, 1H), 7.13 (t,  $J = 7.7$  Hz, 2H), 7.08 - 7.00 (m, 1H), 6.87 (d,  $J = 7.8$  Hz, 1H), 6.26 (s, 1H), 4.60 (d,  $J = 25.7$  Hz, 2H),

3.25 (t,  $J = 1.6$  Hz, 3H), 2.98 (s, 4H).  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 171.4, 170.2, 163.6, 161.1, 143.2, 139.8(0), 139.7(7), 129.3, 128.41 (d,  $J = 8.0$  Hz), 126.7, 126.2, 125.3, 122.8, 115.29 (d,  $J = 21.4$  Hz), 108.4, 70.18, 62.9, 37.3, 34.2, 26.5.  **$^{19}\text{F NMR}$**  (377 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) -115.1. HRMS (ESI) for:  $\text{C}_{21}\text{H}_{18}\text{FNO}_3$   $[\text{M}+\text{H}]^+$ : calcd 352.1343, found 352.1336.

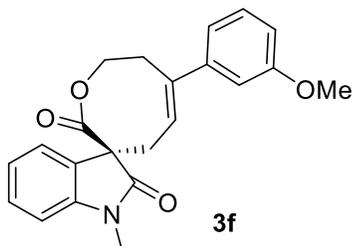
**(*R,E*)-6'-(4-bromophenyl)-1-methyl-7',8'-dihydro-2'*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3e)**



**General procedure A:** White solid, 53% yield, 90% ee,  $[\alpha]_D^{25} = 41.83$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 85:15 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 35.04 min,  $t_R$  (minor) = 18.77 min.  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.56 (d,  $J = 7.5$  Hz, 1H), 7.50 - 7.44 (m, 2H), 7.37 - 7.33 (m, 1H), 7.29 - 7.23 (m, 2H), 7.12 (t,  $J = 7.6$  Hz, 1H), 6.86 (d,  $J = 7.8$  Hz, 1H), 6.30 (t,  $J = 8.8$  Hz, 1H), 4.60 (d,  $J =$

28.4 Hz, 2H), 3.25 (s, 3H), 2.98 (s, 4H).;  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.3, 171.4, 143.2, 142.7, 141.6, 131.6, 129.4, 128.4, 127.4, 126.2, 125.3, 122.8, 121.6, 108.4, 70.2, 62.9, 37.3, 33.9, 26.5. HRMS (ESI) for:  $\text{C}_{21}\text{H}_{18}\text{BrNO}_3$   $[\text{M}+\text{H}]^+$ : calcd 412.0534, found 412.0538.

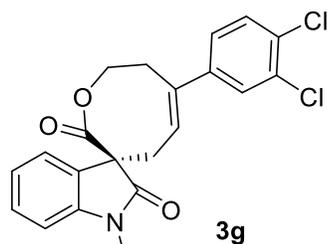
**(*R,E*)-6'-(3-methoxyphenyl)-1-methyl-7',8'-dihydro-2'*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3f)**



**General procedure A:** White solid, 86% yield, 93% ee,  $[\alpha]_D^{25} = 53.90$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); TThe ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 81.58 min,  $t_R$  (minor) = 27.62 min.  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.57 (d,  $J = 7.4$  Hz, 1H), 7.37 - 7.32 (m, 1H), 7.27 (t,  $J = 7.9$  Hz, 1H), 7.13 - 7.09 (m, 1H), 7.00 - 6.95 (m, 1H), 6.91 (t,  $J = 2.1$  Hz, 1H), 6.88 - 6.80 (m, 2H),

6.30 (t,  $J = 8.8$  Hz, 1H), 4.63 (s, 2H), 3.84 (s, 3H), 3.24 (s, 3H), 3.10 (s, 2H), 2.92 (s, 1H).  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.3, 171.4, 159.6, 145.3, 143.2, 142.7, 129.5, 129.2, 126.6, 126.4, 125.5, 122.8, 119.3, 112.8, 112.6, 108.3, 70.2, 62.9, 55.3, 37.5, 34.1, 26.5. HRMS (ESI) for:  $\text{C}_{22}\text{H}_{21}\text{NO}_4$   $[\text{M}+\text{Ha}]^+$ : calcd 364.1543, found 364.1539.

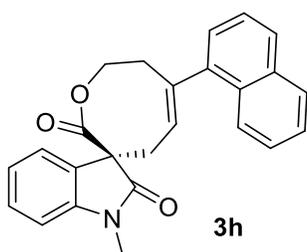
**(*R,E*)-6'-(3,4-dichlorophenyl)-1-methyl-7',8'-dihydro-2'*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3g)**



**General procedure A:** White solid, 78% yield, 80% ee,  $[\alpha]_D^{25} = 52.03$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 37.12 min,  $t_R$  (minor) = 20.68 min;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.55 (d,  $J = 7.5$  Hz, 1H), 7.44 (d,  $J = 2.1$  Hz, 1H), 7.41 (d,  $J = 8.4$  Hz, 1H), 7.35 (t,  $J = 7.8$  Hz, 1H), 7.22 (dd,  $J = 8.3, 2.2$  Hz, 1H), 7.12 (t,  $J = 7.6$  Hz, 1H), 6.87 (d,  $J = 7.8$

Hz, 1H), 6.34 (t,  $J = 8.7$  Hz, 1H), 4.61 (d,  $J = 40.9$  Hz, 2H), 3.25 (s, 3H), 2.97 (s, 4H).;  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.4, 171.3, 143.7, 143.2, 140.5, 132.5, 131.6, 130.4, 129.4, 128.5, 126.3, 126.0, 125.2, 122.9, 108.4, 69.9, 62.9, 37.2, 33.8, 26.5. HRMS (ESI) for:  $\text{C}_{21}\text{H}_{17}\text{Cl}_2\text{NO}_3$   $[\text{M}+\text{H}]^+$ : calcd 402.0658, found 402.0651.

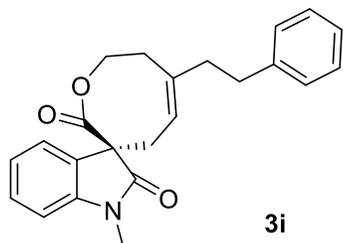
**(*R,E*)-1-methyl-6'-(naphthalen-1-yl)-7',8'-dihydro-2'*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3h)**



**General procedure A:** White solid, 60% yield, 93% ee,  $[\alpha]_D^{25} = 46.03$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak OD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 0.7 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 48.73 min,  $t_R$  (minor) = 58.51 min.  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.96 - 7.84 (m, 4H), 7.65 (d,  $J = 7.4$  Hz, 1H), 7.59 - 7.46 (m, 3H), 7.43 - 7.34 (m, 1H), 7.16 (t,  $J = 7.3$  Hz, 1H), 6.90 (d,  $J = 7.8$  Hz, 1H), 6.47 (t,  $J = 8.8$  Hz, 1H), 4.73 (s, 2H), 3.30 (s,

3H), 3.22 (s, 2H), 3.11 (s, 2H).;  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.4, 171.5, 143.2, 142.7, 141.1, 133.4, 132.7, 129.3, 128.2, 128.2, 127.6, 127.1, 126.4, 126.0, 125.5, 125.3, 125.1, 122.8, 108.4, 70.3, 63.0, 37.6, 34.1, 26.6. HRMS (ESI) for:  $\text{C}_{25}\text{H}_{21}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : calcd 384.1594, found 384.1596.

**(*R,Z*)-1-methyl-6'-phenethyl-7',8'-dihydro-2'*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3i)**

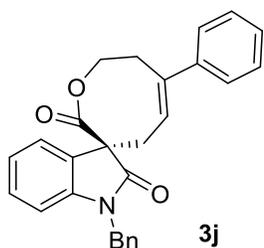


**General procedure A:** White solid, 77% yield, 90% ee,  $[\alpha]_D^{25} = 50.03$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 23.13 min,  $t_R$  (minor) = 17.07 min.  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.40 (d,  $J = 7.3$  Hz, 1H), 7.37 - 7.27 (m, 3H), 7.24 - 7.18 (m, 3H), 7.10 - 7.06 (m, 1H), 6.87

- 6.81 (m, 1H), 5.84 (t,  $J = 8.6$  Hz, 1H), 4.55 (s, 2H), 3.22 (s, 3H), 3.12 - 2.26 (m, 8H).  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.3, 171.5, 143.0, 142.3, 141.4, 129.0, 128.4, 128.3, 126.6, 125.9, 125.5, 122.9, 122.6, 108.2, 70.0, 62.9, 41.9, 37.0, 34.5, 33.6, 26.4. HRMS (ESI) for:  $\text{C}_{23}\text{H}_{23}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : calcd 362.1751, found 362.1755.

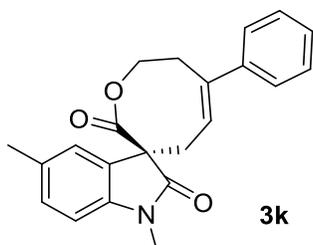
**(*R,E*)-1-benzyl-6'-phenyl-7',8'-dihydro-2'*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3j)**

**General procedure A:** White solid, 81% yield, 94% ee,  $[\alpha]_D^{25} = 56.37$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AS column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 28.16 min,  $t_R$  (minor) = 22.62 min;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.59 (d,  $J = 7.4$  Hz, 1H), 7.42 - 7.33 (m, 4H), 7.33 - 7.19 (m, 7H), 7.12 - 7.03 (m, 1H), 6.74 (d,  $J = 7.8$  Hz, 1H), 6.34 (t,  $J = 8.6$



Hz, 1H), 4.95 (d,  $J = 1.6$  Hz, 2H), 4.64 (d,  $J = 21.6$  Hz, 2H), 3.02 (s, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.5, 171.4, 143.7, 142.2, 135.5, 129.1, 128.8, 128.4, 127.7, 127.5, 127.3, 126.7, 126.5, 126.2, 125.4, 122.7, 109.3, 70.3, 62.9, 43.9, 37.5, 34.0. HRMS (ESI) for:  $\text{C}_{27}\text{H}_{23}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : calcd 410.1751, found 410.1743.

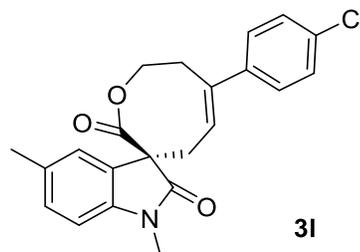
**(R,E)-1,5-dimethyl-6'-phenyl-7',8'-dihydro-2'H,4'H-spiro[indoline-3,3'-oxocine]-2,2'-dione (3k)**



**General procedure B:** White solid, 72% yield, 81% ee,  $[\alpha]_{\text{D}}^{25} = 45.17$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_{\text{R}}$  (major) = 47.58 min,  $t_{\text{R}}$  (minor) = 20.08 min;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.43 - 7.33 (m, 5H), 7.29 (t,  $J = 7.0$  Hz, 1H), 7.14 (d,  $J = 7.9$  Hz, 1H), 6.75 (d,  $J = 7.8$  Hz, 1H), 6.31 (t,  $J = 8.8$  Hz, 1H), 4.64 (s, 2H), 3.23 (s, 3H), 3.02 (s, 4H), 2.38 (s, 3H);  $^{13}\text{C}$

NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.4, 171.6, 143.9, 142.6, 140.8, 132.4, 129.5, 128.5, 127.5, 126.8, 126.7, 126.4, 126.1, 108.1, 70.3, 63.0, 37.4, 34.1, 26.6, 21.3. HRMS (ESI) for:  $\text{C}_{22}\text{H}_{21}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : calcd 348.1594, found 348.1589.

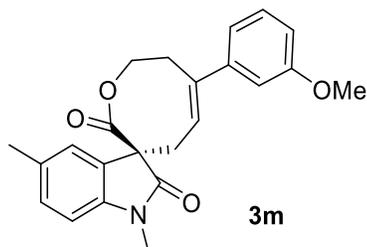
**(R,E)-6'-(4-chlorophenyl)-1,5-dimethyl-7',8'-dihydro-2'H,4'H-spiro[indoline-3,3'-oxocine]-2,2'-dione (3l)**



**General procedure B:** White solid, 48% yield, 89% ee,  $[\alpha]_{\text{D}}^{25} = 43.07$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_{\text{R}}$  (major) = 36.42 min,  $t_{\text{R}}$  (minor) = 19.72 min;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.30 (s, 1H), 7.24 (s, 4H), 7.14 - 7.02 (m, 1H), 6.68 (d,  $J = 7.9$  Hz, 1H), 6.24 (t,  $J = 8.7$  Hz, 1H), 4.53 (d,  $J = 39.9$  Hz, 2H), 3.15 (s, 3H), 2.92 (s, 4H),

2.30 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.5, 171.5, 142.2, 141.4, 140.8, 133.4, 132.4, 129.6, 128.6, 128.2, 127.5, 126.1, 125.9, 108.1, 70.1, 63.0, 37.38, 34.0, 26.5, 21.3. HRMS (ESI) for:  $\text{C}_{22}\text{H}_{20}\text{ClNO}_3$   $[\text{M}+\text{H}]^+$ : calcd 382.1204, found 382.1203.

**(R,E)-6'-(3-methoxyphenyl)-1,5-dimethyl-7',8'-dihydro-2'H,4'H-spiro[indoline-3,3'-oxocine]-2,2'-dione (3m)**

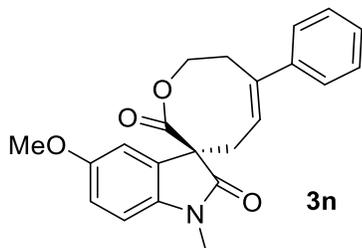


**General procedure B:** White solid, 90% yield, 84% ee,  $[\alpha]_{\text{D}}^{25} = 83.47$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 210$  nm, 25 °C),  $t_{\text{R}}$  (major) = 63.23 min,  $t_{\text{R}}$  (minor) = 33.73 min;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.40 - 7.32 (m, 4H), 7.32 - 7.27 (m, 1H), 7.21 (d,  $J = 2.6$  Hz, 1H), 6.87 (dd,  $J = 8.5, 2.6$  Hz, 1H), 6.76 (d,  $J = 8.5$  Hz, 1H), 6.29 (t,  $J = 8.5$  Hz, 1H), 4.63

(s, 2H), 3.82 (s, 3H), 3.23 (s, 3H), 3.05 (s, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.1, 171.4, 156.0, 143.8,

142.8, 136.8, 128.5, 127.5, 126.7, 126.5, 113.3, 113.2, 108.5, 70.3, 63.2, 56.0, 37.5, 34.1, 26.6.. HRMS (ESI) for:  $C_{22}H_{21}NO_4$   $[M+H]^+$ : calcd 364.1543, found 364.1535.

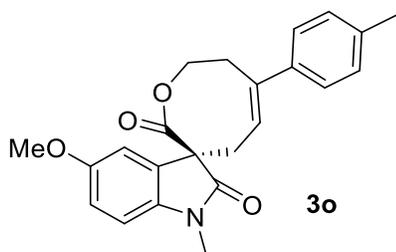
**(*R,E*)-5-Methoxy-1-methyl-6'-phenyl-7',8'-dihydro-2*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3n)**



**General procedure B:** White solid, 88% yield, 86% ee,  $[\alpha]_D^{25} = 38.87$  ( $c = 1.00$  in  $CHCl_3$ ); The ee value was determined by HPLC analysis (Chiralpak IBN-5 column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 210$  nm, 25 °C),  $t_R$  (major) = 28.66 min,  $t_R$  (minor) = 46.63 min;  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  (ppm) 7.44 - 7.32 (m, 4H), 7.32 - 7.27 (m, 1H), 7.21 (d,  $J = 2.6$  Hz, 1H), 6.87 (dd,  $J = 8.5, 2.6$  Hz, 1H), 6.76 (d,  $J = 8.5$  Hz, 1H), 6.29 (t,  $J = 8.5$  Hz, 1H), 4.63

(s, 2H), 3.82 (s, 3H), 3.23 (s, 3H), 3.05 (s, 4H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  (ppm) 172.1, 171.4, 156.0, 143.8, 142.8, 136.8, 128.5, 127.5, 126.7, 126.5, 113.3, 113.2, 108.5, 70.3, 63.2, 56.0, 37.5, 34.1, 26.6. HRMS (ESI) for:  $C_{23}H_{23}NO_4$   $[M+H]^+$ : calcd 364.1543, found 364.1535.

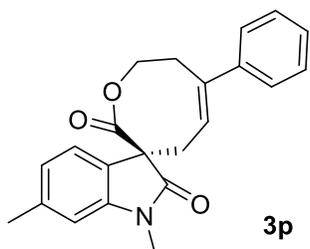
**(*R,E*)-5-methoxy-1-methyl-6'-(*p*-tolyl)-7',8'-dihydro-2*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3o)**



**General procedure B:** White solid, 85% yield, 69% ee,  $[\alpha]_D^{25} = 51.63$  ( $c = 1.00$  in  $CHCl_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 76.43 min,  $t_R$  (minor) = 29.74 min;  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  (ppm) 7.34 - 7.23 (m, 2H), 7.21 (d,  $J = 2.5$  Hz, 1H), 7.16 (d,  $J = 7.9$  Hz, 2H), 6.87 (dd,  $J = 8.5, 2.6$  Hz, 1H), 6.76 (d,  $J = 8.5$  Hz, 1H), 6.26 (t,  $J =$

8.8 Hz, 1H), 4.61 (s, 2H), 3.82 (s, 3H), 3.22 (s, 3H), 3.00 (d,  $J = 37.8$  Hz, 4H), 2.36 (s, 3H).;  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  (ppm) 172.0, 171.3, 156.0, 142.7, 140.8, 137.3, 136.7, 129.2, 127.7, 126.6, 125.7, 113.3, 113.2, 108.5, 70.3, 63.2, 56.0, 37.5, 34.0, 26.6, 21.2. HRMS (ESI) for:  $C_{23}H_{23}NO_4$   $[M+H]^+$ : calcd 378.1700, found 378.1696.

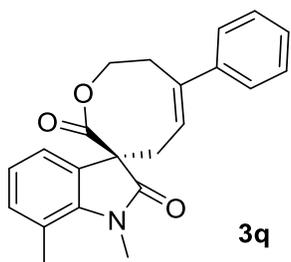
**(*R,E*)-1,6-dimethyl-6'-phenyl-7',8'-dihydro-2*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3p)**



**General procedure B:** White solid, 54% yield, 86% ee,  $[\alpha]_D^{25} = 24.50$  ( $c = 1.00$  in  $CHCl_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_R$  (major) = 52.48 min,  $t_R$  (minor) = 19.27 min;  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  (ppm) 7.45 (d,  $J = 7.6$  Hz, 1H), 7.41 - 7.33 (m, 4H), 7.32 - 7.27 (m, 1H), 7.07 - 6.91 (m, 1H), 6.69 (d,  $J = 1.4$  Hz, 1H), 6.28 (s, 1H), 4.62 (s, 2H), 3.23 (s, 3H), 3.01 (d,  $J = 59.9$  Hz, 4H), 2.40

(s, 3H).;  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  (ppm) 172.7, 171.6, 143.8, 143.3, 142.6, 139.6, 128.5, 127.5, 126.7(3), 126.6(6), 125.2, 123.5, 123.3, 109.3, 70.2, 62.8, 37.5, 34.1, 26.5, 21.9. HRMS (ESI) for:  $C_{22}H_{21}NO_3$   $[M+H]^+$ : calcd 348.1594, found 348.1598.

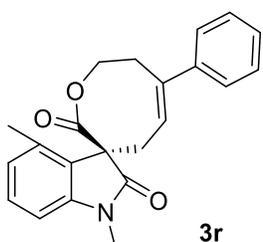
**(*R,E*)-1,7-dimethyl-6'-phenyl-7',8'-dihydro-2*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3q)**



**3q**

**General procedure B:** White solid, 49% yield, 66% ee,  $[\alpha]_{\text{D}}^{25} = 22.73$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_{\text{R}}$  (major) = 46.44 min,  $t_{\text{R}}$  (minor) = 17.37 min.  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.51 - 7.36 (m, 4H), 7.35 - 7.27 (m, 1H), 7.11 (d,  $J = 7.7$  Hz, 1H), 7.03 (t,  $J = 7.6$  Hz, 1H), 6.33 (t,  $J = 8.8$  Hz, 1H), 4.66 (s, 2H), 3.57 (s, 3H), 3.23 - 2.86 (m, 4H), 2.62 (s, 3H).;  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 173.2, 171.6, 143.9, 142.7, 141.0, 133.0, 128.5, 127.5, 126.8, 126.8, 123.4, 122.6, 119.9, 70.4, 62.4, 37.7, 34.1, 29.9, 19.2. HRMS (ESI) for:  $\text{C}_{22}\text{H}_{21}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : calcd 348.1594, found 348.1587.

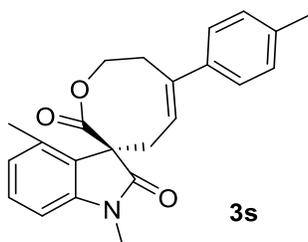
**(*R,E*)-1,4-dimethyl-6'-phenyl-7',8'-dihydro-2*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3r)**



**3r**

**General procedure B:** White solid, 50% yield, 97% ee,  $[\alpha]_{\text{D}}^{25} = 9.13$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_{\text{R}}$  (major) = 50.47 min,  $t_{\text{R}}$  (minor) = 18.42 min.  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.42 - 7.38 (m, 2H), 7.36 - 7.31 (m, 2H), 7.30 - 7.27 (m, 1H), 7.25 - 7.20 (m, 1H), 6.91 - 6.89 (m, 1H), 6.70 (d,  $J = 7.7$  Hz, 1H), 6.41 (t,  $J = 8.7$  Hz, 1H), 4.72 - 4.64 (m, 1H), 4.55 - 4.52 (m, 1H), 3.40 (d,  $J = 11.3$  Hz, 1H), 3.24 (s, 3H), 3.15 - 3.05 (m, 1H), 3.03 - 2.85 (m, 2H), 2.57 (s, 3H).;  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ ) 173.0, 172.1, 144.1, 144.0, 141.3, 136.7, 129.0, 128.4, 128.1, 127.3, 126.7, 126.4, 123.7, 106.1, 70.6, 64.7, 34.4, 34.3, 26.6, 20.6. HRMS (ESI) for:  $\text{C}_{22}\text{H}_{21}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : calcd 348.1594, found 348.1589.

**(*R,E*)-1,4-dimethyl-6'-(*p*-tolyl)-7',8'-dihydro-2*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3s)**

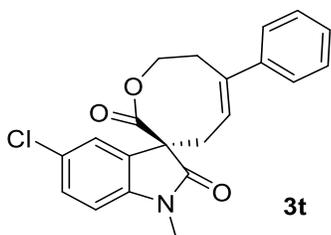


**3s**

**General procedure B:** White solid, 42% yield, 98% ee,  $[\alpha]_{\text{D}}^{25} = 28.20$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_{\text{R}}$  (major) = 48.28 min,  $t_{\text{R}}$  (minor) = 19.60 min.  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.34 (d,  $J = 1.6$  Hz, 2H), 7.31 - 7.24 (m, 1H), 7.18 (d,  $J = 7.9$  Hz, 2H), 6.94 (d,  $J = 7.8$  Hz, 1H), 6.73 (d,  $J = 7.7$  Hz, 1H), 6.41 (t,  $J = 8.7$  Hz, 1H), 4.71 - 4.65 (m, 1H), 4.60 - 4.55 (m,  $J = 10.8, 5.5, 3.1$  Hz, 1H), 3.43 - 3.34 (m, 1H), 3.28 (s, 3H), 3.15 - 3.06 (m, 1H), 3.03 - 2.90 (m, 2H), 2.60 (s, 3H), 2.39 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 173.0, 172.0, 144.1, 141.2, 141.1, 137.1, 136.8, 129.1, 129.0, 127.3, 126.6, 126.4, 123.8, 106.1, 70.6, 64.8, 34.56, 34.3, 26.6, 21.2, 20.7. HRMS (ESI) for:  $\text{C}_{23}\text{H}_{23}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : calcd 362.1751, found 362.1750.

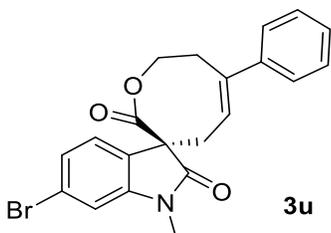
**(*R,E*)-5-chloro-1-methyl-6'-phenyl-7',8'-dihydro-2*H*,4'*H*-spiro[indoline-3,3'-oxocine]-2,2'-dione (3t)**

**General procedure B:** White solid, 66% yield, 6% ee,  $[\alpha]_{\text{D}}^{25} = 2.30$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak IBN-5 column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_{\text{R}}$  (major) = 32.75 min,  $t_{\text{R}}$  (minor) = 28.27 min.  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.67-7.52



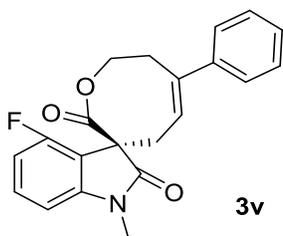
(m, 1H), 7.46-7.28 (m, 6H), 6.79 (d,  $J = 8.3$  Hz, 1H), 6.26 (t,  $J = 8.4$  Hz, 1H), 4.64 (s, 2H), 3.24 (s, 3H), 3.02 (s, 4H).;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 171.9, 170.7, 143.6, 143.1, 141.7, 129.2, 128.5, 128.1, 127.8, 127.6, 126.7, 125.9, 109.2, 70.4, 62.9, 37.4, 34.0, 26.6. HRMS (ESI) for:  $\text{C}_{21}\text{H}_{18}\text{ClNO}_3$   $[\text{M}+\text{Na}]^+$ : calcd 390.0867, found 390.0869.

**(R,E)-5-bromo-1-methyl-6'-phenyl-7',8'-dihydro-2'H,4'H-spiro[indoline-3,3'-oxocine]-2,2'-dione (3u)**



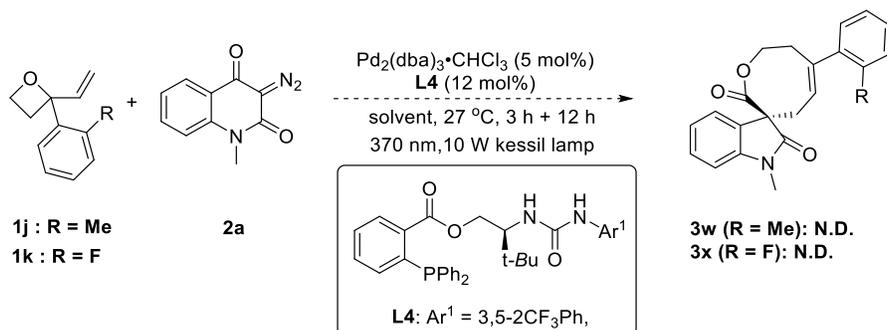
**General procedure B:** White solid, 63% yield, 2% ee,  $[\alpha]_{\text{D}}^{25} = 1.20$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak IBN-5 column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_{\text{R}}$  (major) = 26.99 min,  $t_{\text{R}}$  (minor) = 30.89 min.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.44 (d,  $J = 8.0$  Hz, 1H), 7.38 (d,  $J = 4.3$  Hz, 4H), 7.33 (dt,  $J = 8.8, 4.4$  Hz, 1H), 7.26 (d,  $J = 1.7$  Hz, 1H), 7.04 (d,  $J = 1.6$  Hz, 1H), 6.25 (s, 1H), 4.65 (s, 2H), 3.25 (s, 3H), 3.04 (d,  $J = 64.9$  Hz, 4H).;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 172.1, 170.8, 144.5, 143.6, 143.1, 128.5, 127.6, 126.8, 126.6, 127.0, 125.6, 125.2, 123.0, 111.8, 70.3, 62.6, 37.4, 34.0, 26.6. HRMS (ESI) for:  $\text{C}_{21}\text{H}_{18}\text{BrNO}_3$   $[\text{M}+\text{Na}]^+$ : calcd 434.0361, found 434.0362.

**(R,E)-4-fluoro-1-methyl-6'-phenyl-7',8'-dihydro-2'H,4'H-spiro[indoline-3,3'-oxocine]-2,2'-dione (3v)**

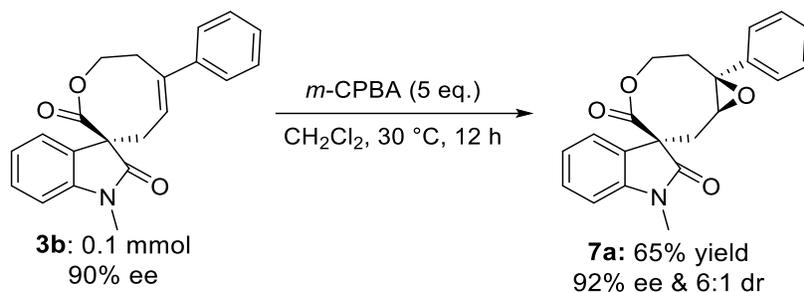


**General procedure B:** White solid, 58% yield, 70% ee,  $[\alpha]_{\text{D}}^{25} = 25.20$  ( $c = 1.00$  in  $\text{CHCl}_3$ ); The ee value was determined by HPLC analysis (Chiralpak AD column, hexane/*i*-PrOH, 90:10 v/v, flow rate 1.0 mL/min,  $\lambda = 254$  nm, 25 °C),  $t_{\text{R}}$  (major) = 28.68 min,  $t_{\text{R}}$  (minor) = 15.78 min.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 7.45 - 7.17 (m, 6H), 6.88 - 6.83 (m, 1H), 6.68 (dd,  $J = 7.8, 0.8$  Hz, 1H), 6.28 - 6.22 (m, 1H), 4.67 (t,  $J = 10.4$  Hz, 1H), 4.49 - 4.44 (m, 1H), 3.65 - 3.25 (m, 2H), 3.25 (s, 3H), 2.81 (d,  $J = 15.4$  Hz, 2H).;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 171.2, 169.9, 160.7, 158.2, 145.4 (d,  $J = 8.9$  Hz), 144.1, 142.4, 142.4, 131.1 (d,  $J = 9.0$  Hz), 128.4, 127.3, 127.1, 127.1, 126.9, 126.9, 111.2 (d,  $J = 22.4$  Hz), 104.5, 104.4, 69.9, 63.3(3), 63.2(9), 34.1, 27.1.  $^{19}\text{F NMR}$  (377 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) -62.9. HRMS (ESI) for:  $\text{C}_{21}\text{H}_{18}\text{FNO}_3$   $[\text{M}+\text{H}]^+$ : calcd 352.1343, found 352.1337.

**Unsuccessful results:**



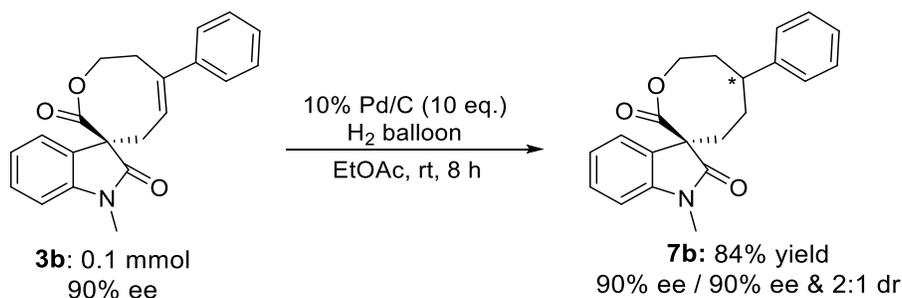
## 5. Synthetic Transformations



Under argon atmosphere, a flame-dried 10 mL Schlenk tube was charged with **3b** (0.1 mmol) anhydrous DCM (1.5 mL), then *m*-CPBA (5.0 eq) was added, the mixture was stirred at 30 °C for 12 hours. The solution was quenched with sodium thiosulfate, extracted with ethyl acetate, dried over anhydrous sodium sulfate, purified by chromatography on silica gel (petroleum ether/ EtOAc = 10/1) to afford the desired product **7a** as white solid with 65% yield.

### (3R)-1'-methyl-8-phenyl-5,9-dioxaspiro[bicyclo[6.1.0]nonane-3,3'-indoline]-2',4-dione (**7a**)

White solid, 65% yield, 92% ee, 6:1 dr. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm, major + minor) 7.57 - 7.53 (m, 3H), 7.44 - 7.28 (m, 4H), 7.12 (td, *J* = 7.6, 1.0 Hz, 1H), 6.88 (d, *J* = 7.8 Hz, 1H), 4.88 (t, *J* = 12.2 Hz, 1H), 4.28-4.24 (m, 1H), 4.07 (dd, *J* = 10.7, 4.5 Hz, 1H), 3.28 (s, 3H), 2.58 (dd, *J* = 13.4, 4.4 Hz, 2H), 2.51 - 2.25 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm, major + minor) 173.3, 172.2, 143.5, 140.4, 129.6, 129.6, 128.6, 128.5, 128.2(2), 128.1(7), 127.6, 127.5, 125.2, 125.1, 124.5, 123.1, 109.4, 108.0, 66.1, 62.8, 57.7, 57.3, 57.1, 38.3, 37.5, 26.6, 26.5. HRMS (ESI) for: C<sub>21</sub>H<sub>19</sub>NO<sub>4</sub> [M+H]<sup>+</sup>: calcd 372.1207, found 372.1206. [α]<sub>D</sub><sup>25</sup> = -22.3 (*c* = 1.00, CHCl<sub>3</sub>); The dr value was determined by the <sup>1</sup>H NMR analysis of the chiral reaction mixture; The ee value was determined by HPLC analysis (IE-H, hexane/*i*-PrOH = 80/20, detector: 254 nm, flow rate: 1.0 mL/min), *t*<sub>R</sub> (major) = 28.35 min, *t*<sub>R</sub> (minor) = 41.49 min.



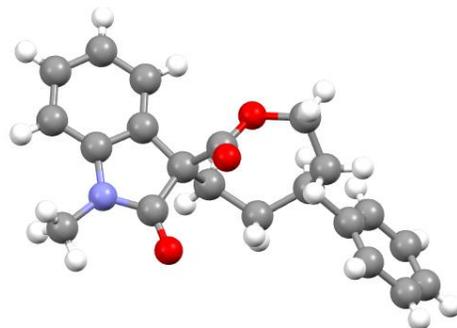
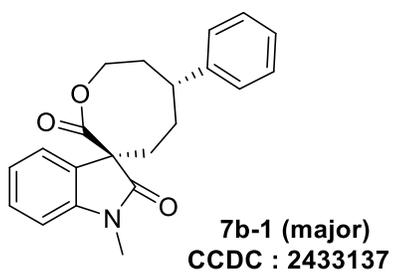
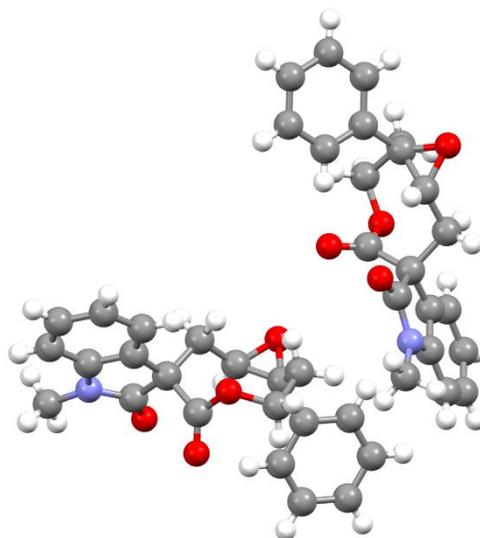
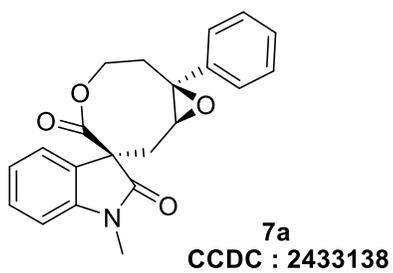
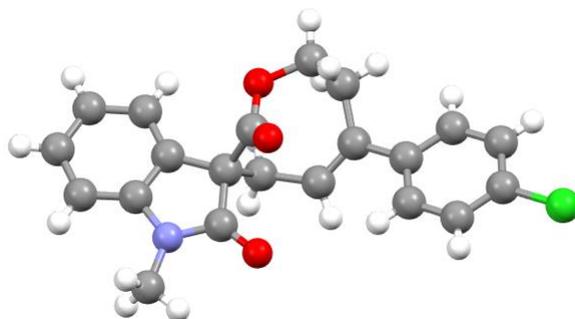
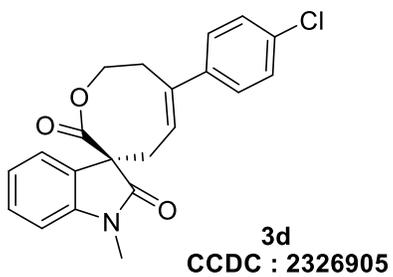
Under argon atmosphere, a flame-dried 10 mL Schlenk tube was charged with compound **3b** (0.1 mmol, 1.0 equiv.), 10% Pd/C (1.0 mmol, 10 equiv.), EtOAc (1.0 mL) and a stir bar was added. The resulting solution was stirred at room temperature under H<sub>2</sub> (1 atm). Then the reaction mixture was filtered through a short pad of silica with EtOAc and concentrated under reduced pressure. The crude

product was purified by flash column chromatography on silica (petrol ether/EtOAc = 25/1 to 10/1) to obtain hydrogenated product **7b**.

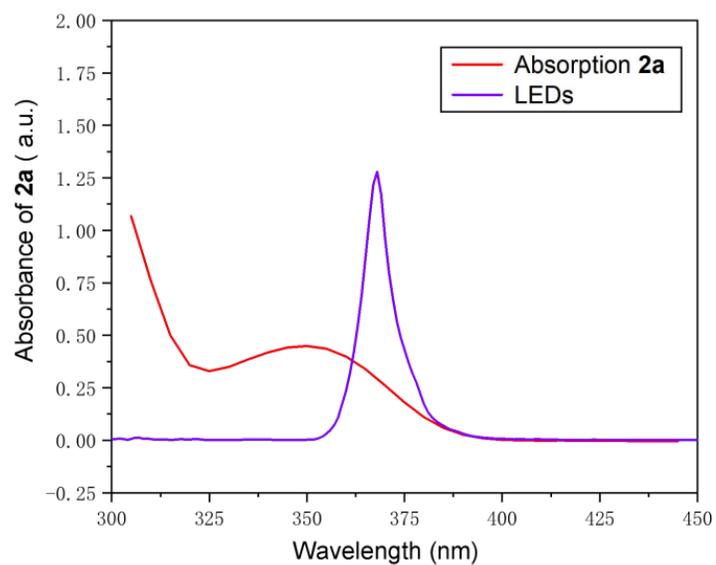
**(3R)-1-methyl-6'-phenylspiro[indoline-3,3'-oxocane]-2,2'-dione (7b)**

White solide, 84% yield, 90% ee/ 90% ee, 2:1 dr. **7b-1 (major)**: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) 7.59 (dd, *J* = 7.5, 1.2 Hz, 1H), 7.40 - 7.24 (m, 3H), 7.22 - 7.17 (m, 3H), 7.12 (td, *J* = 7.6, 1.0 Hz, 1H), 6.84 (d, *J* = 7.8 Hz, 1H), 4.72 (dd, *J* = 10.9, 5.8 Hz, 1H), 4.38 (ddd, *J* = 12.3, 10.8, 4.4 Hz, 1H), 3.23 (s, 3H), 2.77 (d, *J* = 9.7 Hz, 1H), 2.67 (dd, *J* = 15.7, 9.1 Hz, 2H), 2.53 - 2.39 (m, 1H), 2.34 - 2.19 (m, 1H), 2.12 - 1.97 (m, 1H), 1.86 (dd, *J* = 14.6, 4.4 Hz, 1H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) 174.0, 172.2, 149.9, 143.2, 129.1, 128.8, 127.4, 126.3, 126.0, 124.6, 122.8, 108.2, 67.3, 57.2, 45.6, 40.6, 38.2, 33.0, 26.4. HRMS (ESI) for: C<sub>21</sub>H<sub>21</sub>NO<sub>3</sub> [M+H]<sup>+</sup>: calcd 358.1414, found 358.1414 [ $\alpha$ ]<sub>D</sub><sup>25</sup> = -10.27 (*c* = 1.00, CHCl<sub>3</sub>); The dr value was determined by the <sup>1</sup>H NMR analysis of the chiral reaction mixture; The ee value was determined by HPLC analysis (IE-H, hexane/*i*-PrOH = 80/20, detector: 254 nm, flow rate: 1.0 mL/min), *t*<sub>R</sub> (major) = 29.98 min, *t*<sub>R</sub> (minor) = 24.25 min. **7b-2 (minor)**: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) 7.68 (dd, *J* = 7.5, 1.2 Hz, 1H), 7.44 - 7.27 (m, 3H), 7.20 (d, *J* = 7.4 Hz, 3H), 7.14 (td, *J* = 7.6, 1.0 Hz, 1H), 6.87 (d, *J* = 7.8 Hz, 1H), 4.71 (td, *J* = 11.7, 4.0 Hz, 1H), 4.63 - 4.48 (m, 1H), 3.25 (s, 3H), 3.00 - 2.84 (m, 1H), 2.72 - 2.43 (m, 2H), 2.34 - 2.10 (m, 3H), 1.95 (dd, *J* = 14.6, 4.0 Hz, 1H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) 173.0, 171.9, 149.4, 143.1, 129.0, 128.8, 128.1, 126.38, 126.3, 126.1, 125.4, 122.7, 108.4, 67.9, 58.2, 44.1, 40.4, 38.4, 34.3, 26.6. HRMS (ESI) for: C<sub>21</sub>H<sub>21</sub>NO<sub>4</sub> [M+H]<sup>+</sup>: calcd 358.1414, found 358.1407. [ $\alpha$ ]<sub>D</sub><sup>25</sup> = -2.13 (*c* = 1.00, CHCl<sub>3</sub>); The dr value was determined by the <sup>1</sup>H NMR analysis of the chiral reaction mixture; The ee value was determined by HPLC analysis (IE-H, hexane/*i*-PrOH = 80/20, detector: 254 nm, flow rate: 1.0 mL/min), *t*<sub>R</sub> (major) = 33.03 min, *t*<sub>R</sub> (minor) = 28.51 min.

## 6. X-Ray Structure of Product 3d

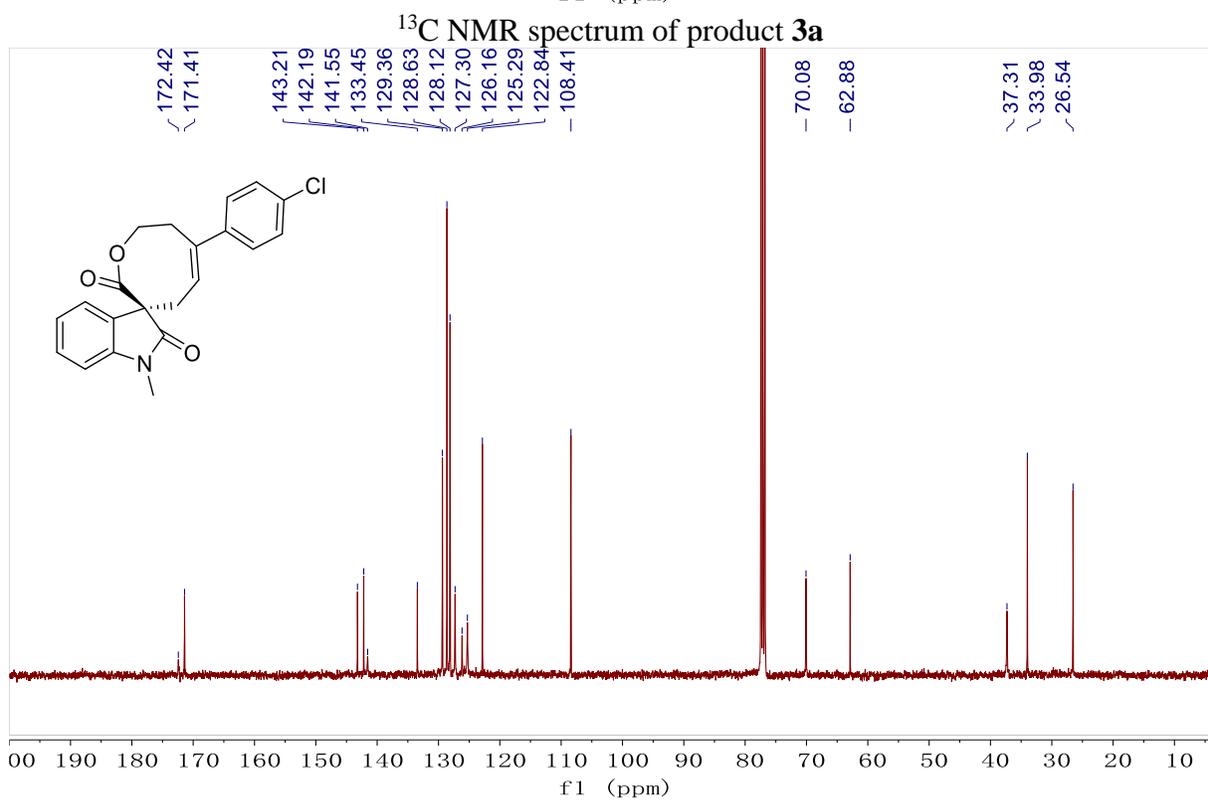
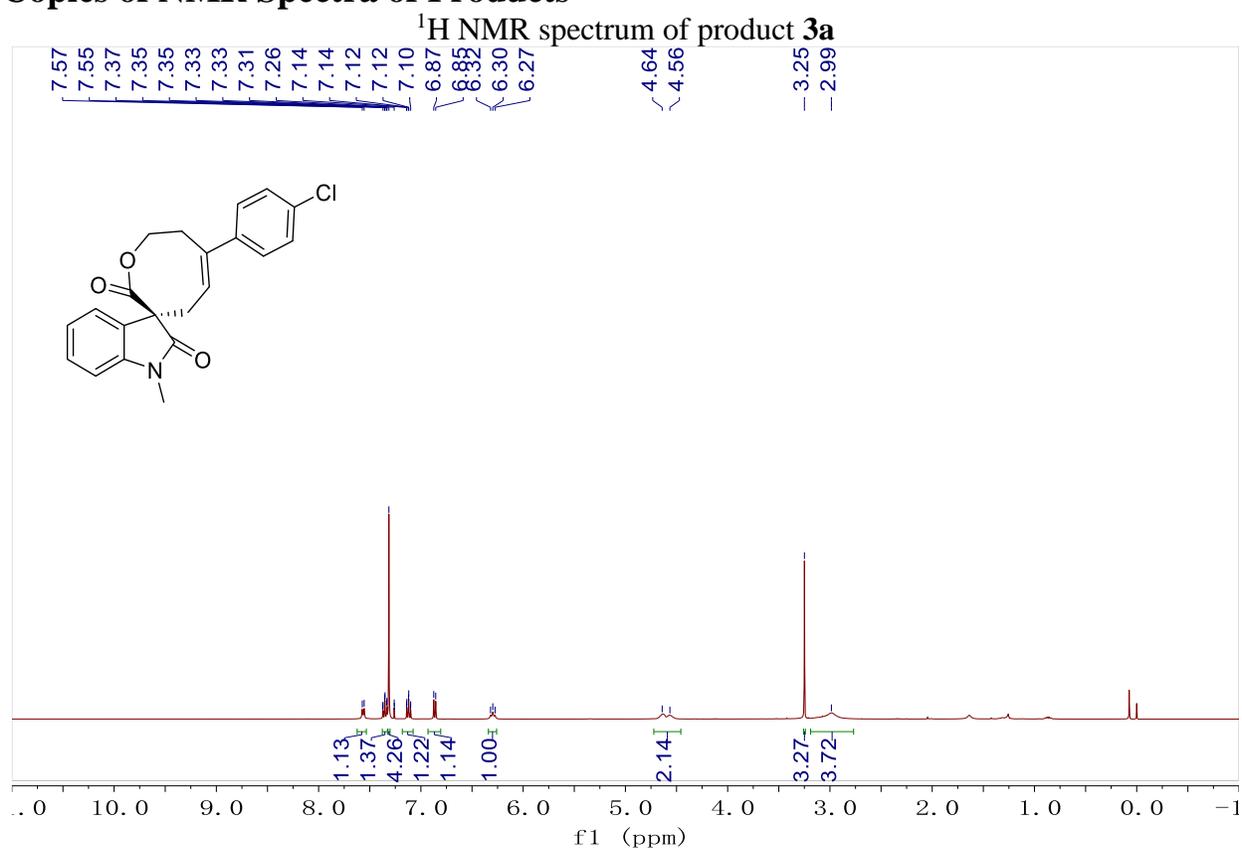


## 7. Absorption Spectrum of Diazo 2a and Emission Spectroscopy of LEDs

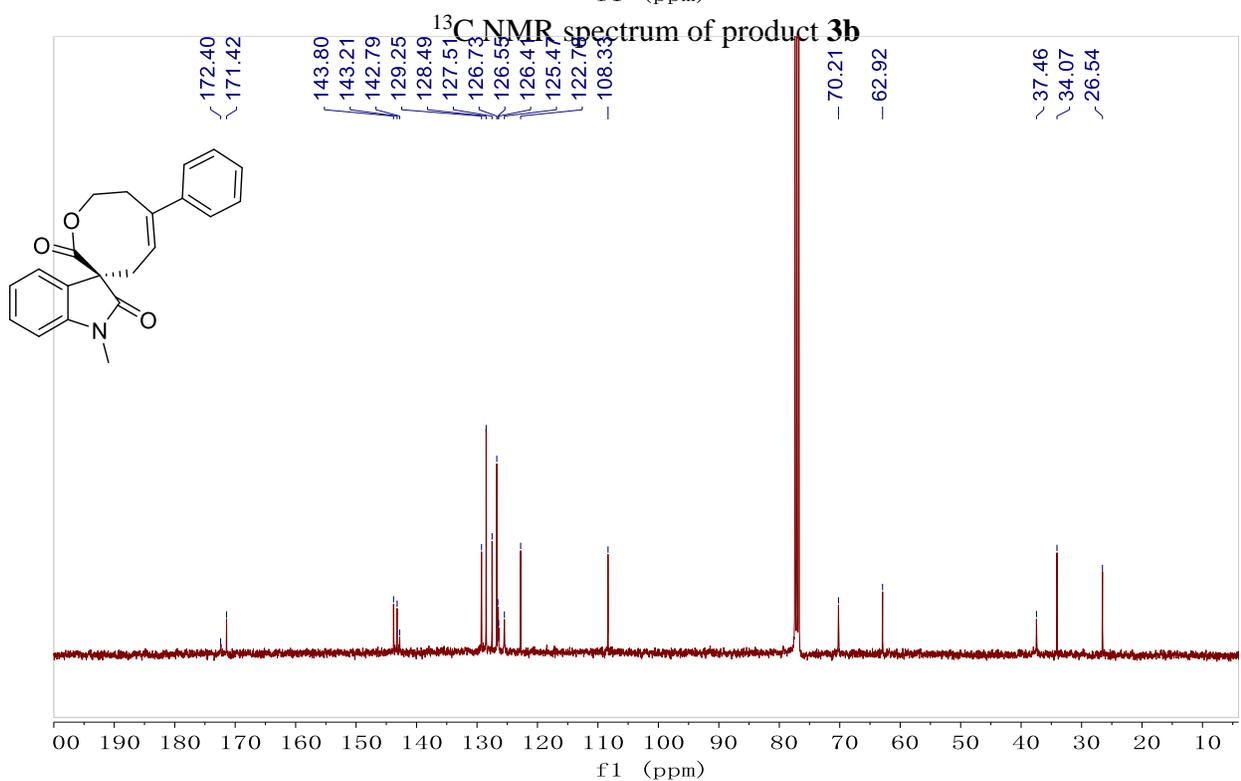
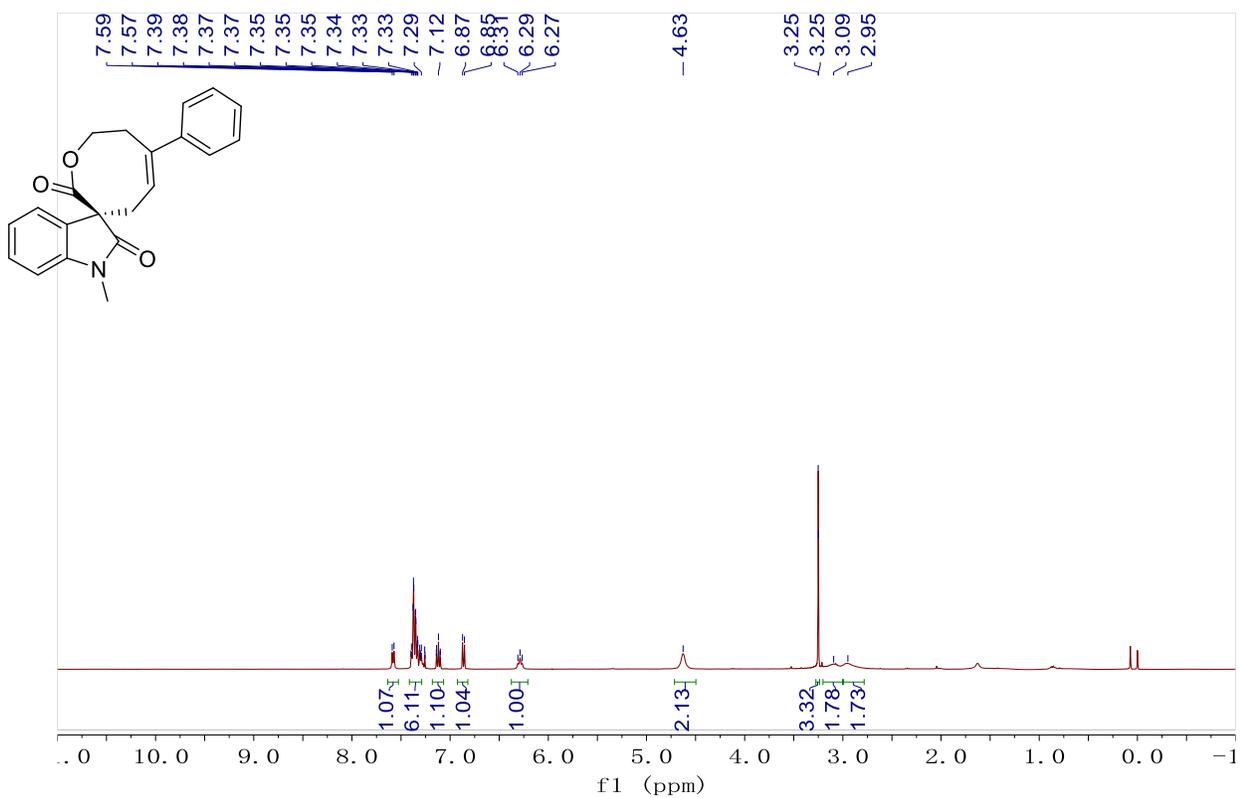


UV/Vis: Measurements were made on an Agilent G9800A Spectro Fluorophotometer.

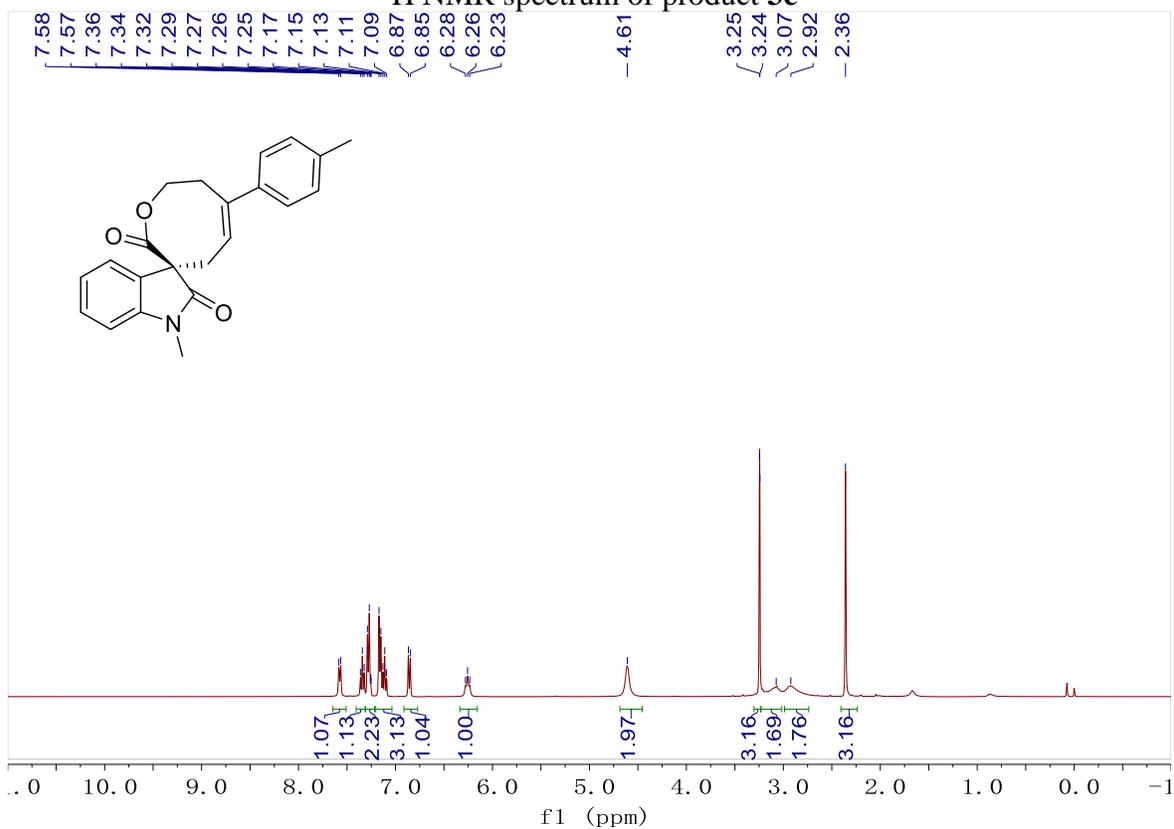
## 8. Copies of NMR Spectra of Products



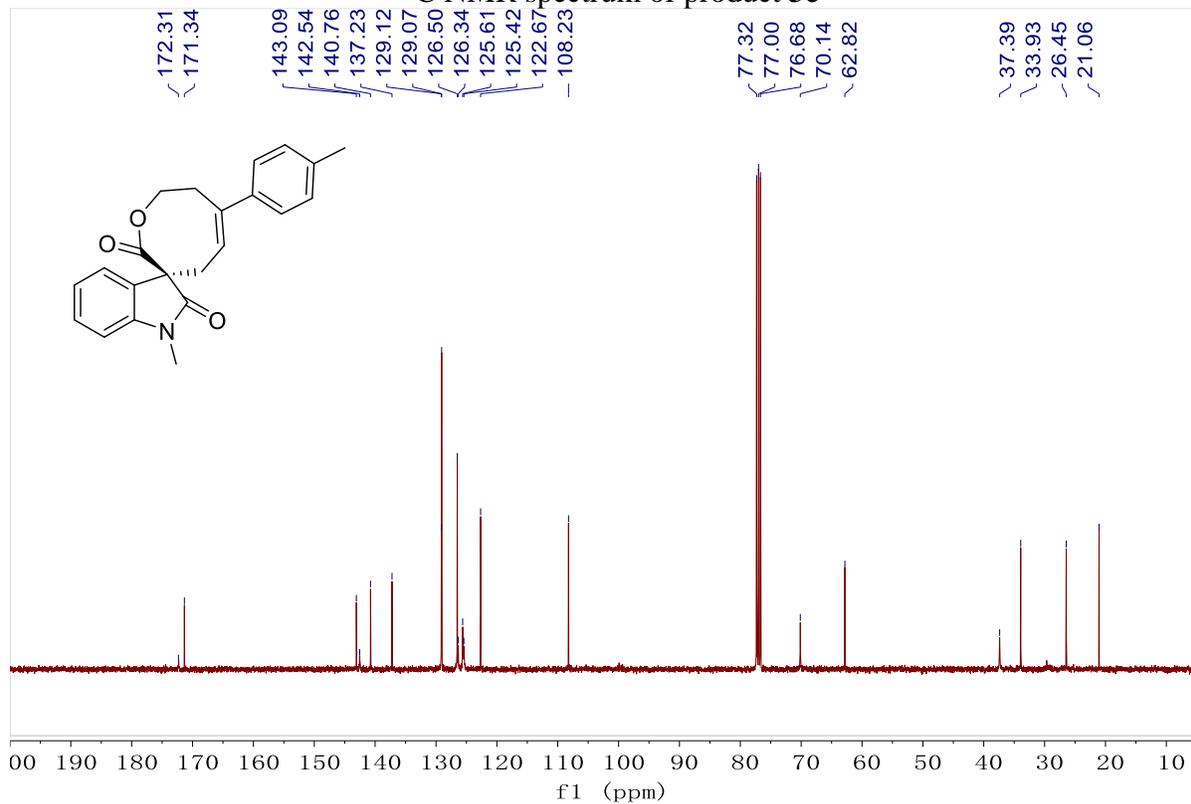
<sup>1</sup>H NMR spectrum of product **3b**



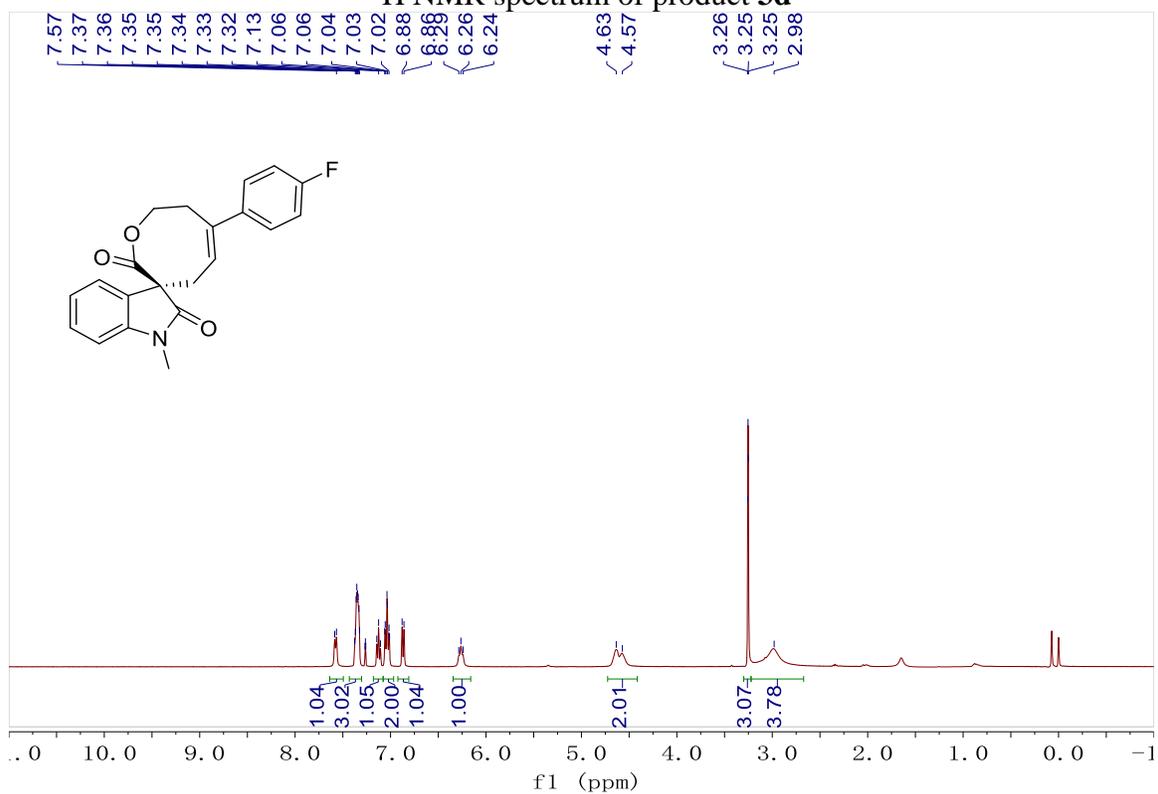
<sup>1</sup>H NMR spectrum of product **3c**



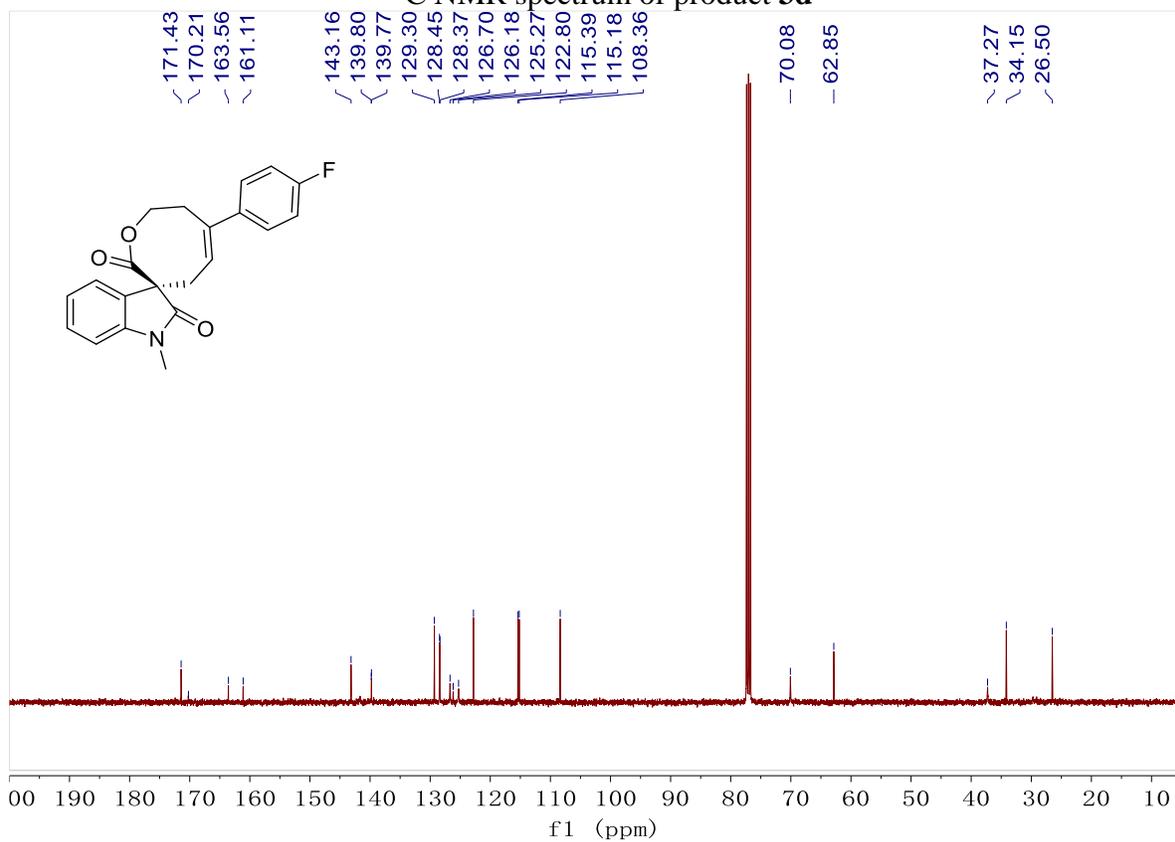
<sup>13</sup>C NMR spectrum of product **3c**



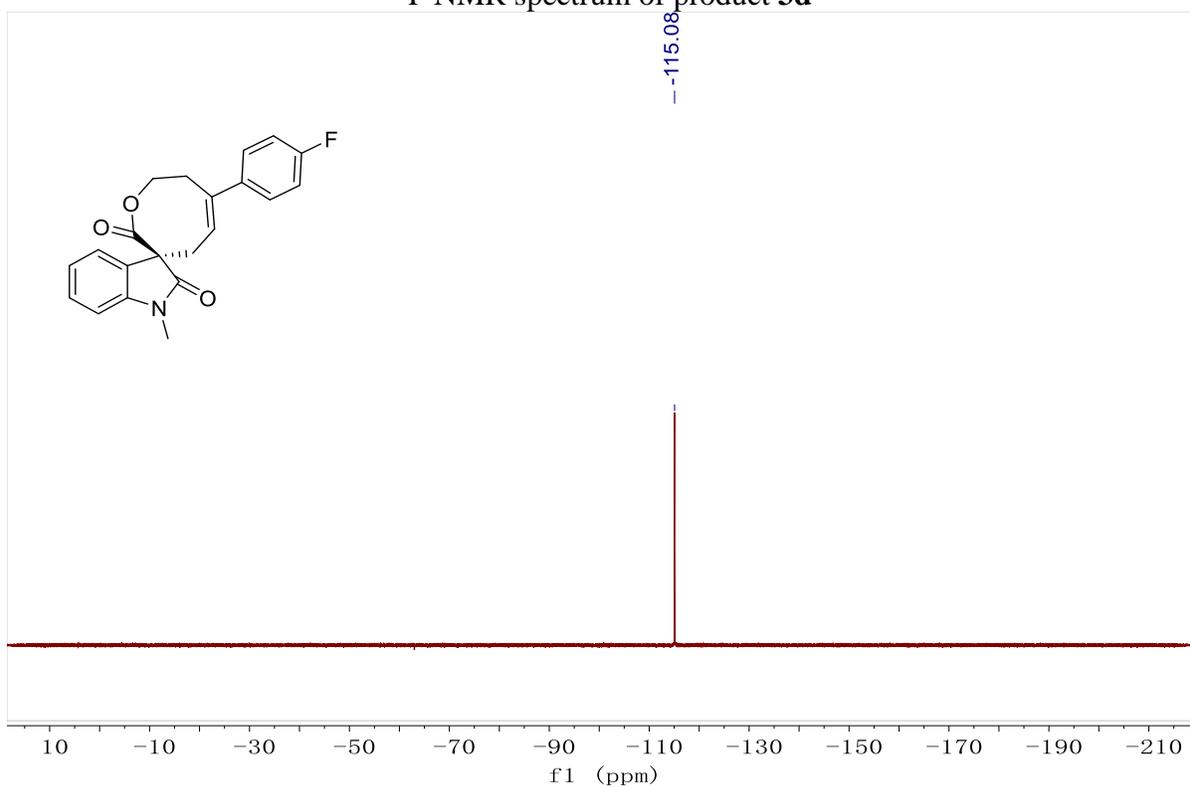
<sup>1</sup>H NMR spectrum of product **3d**



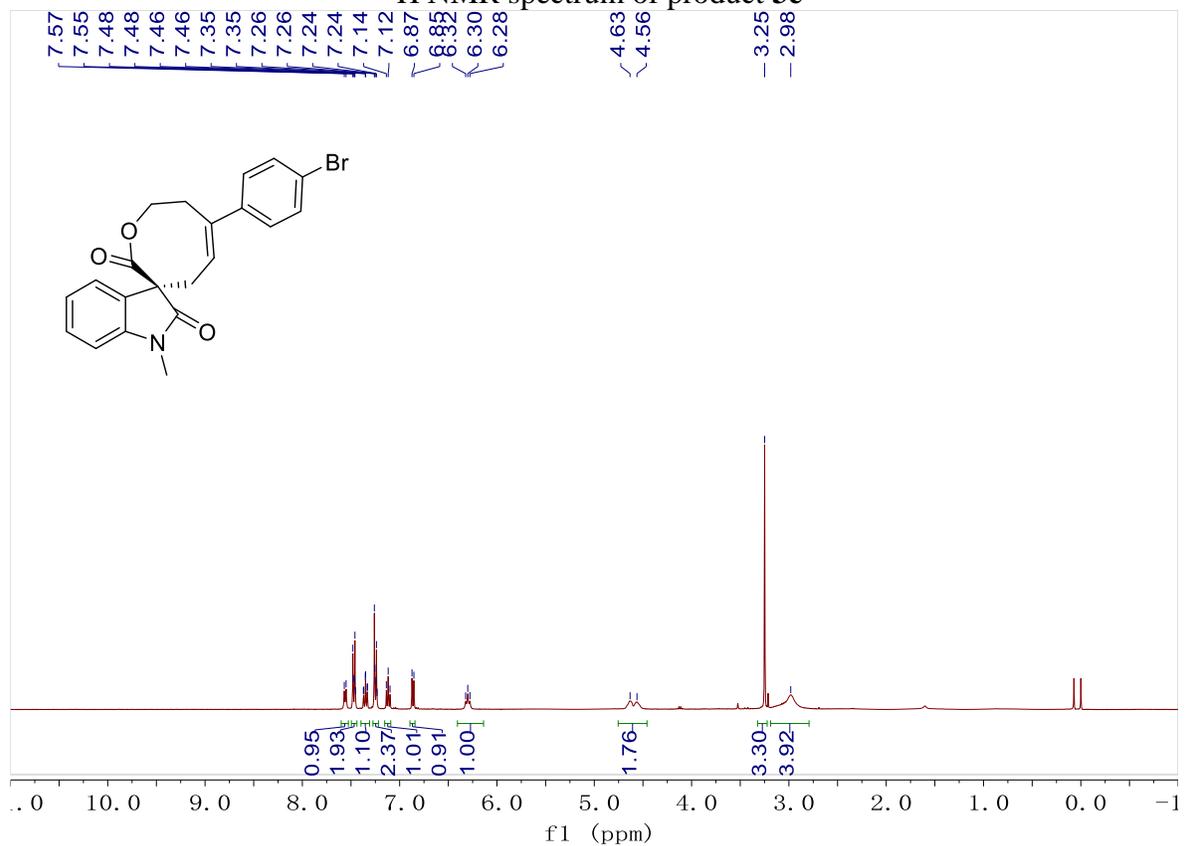
<sup>13</sup>C NMR spectrum of product **3d**

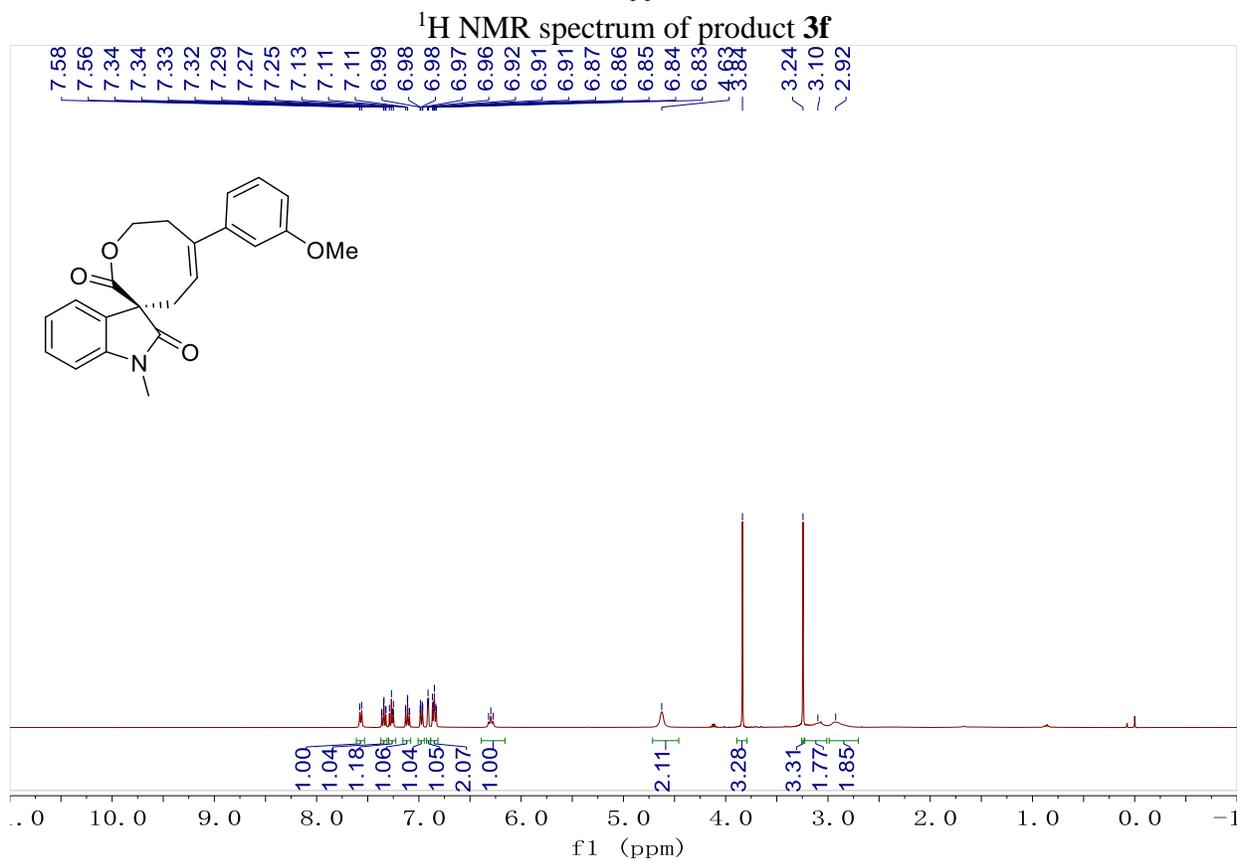
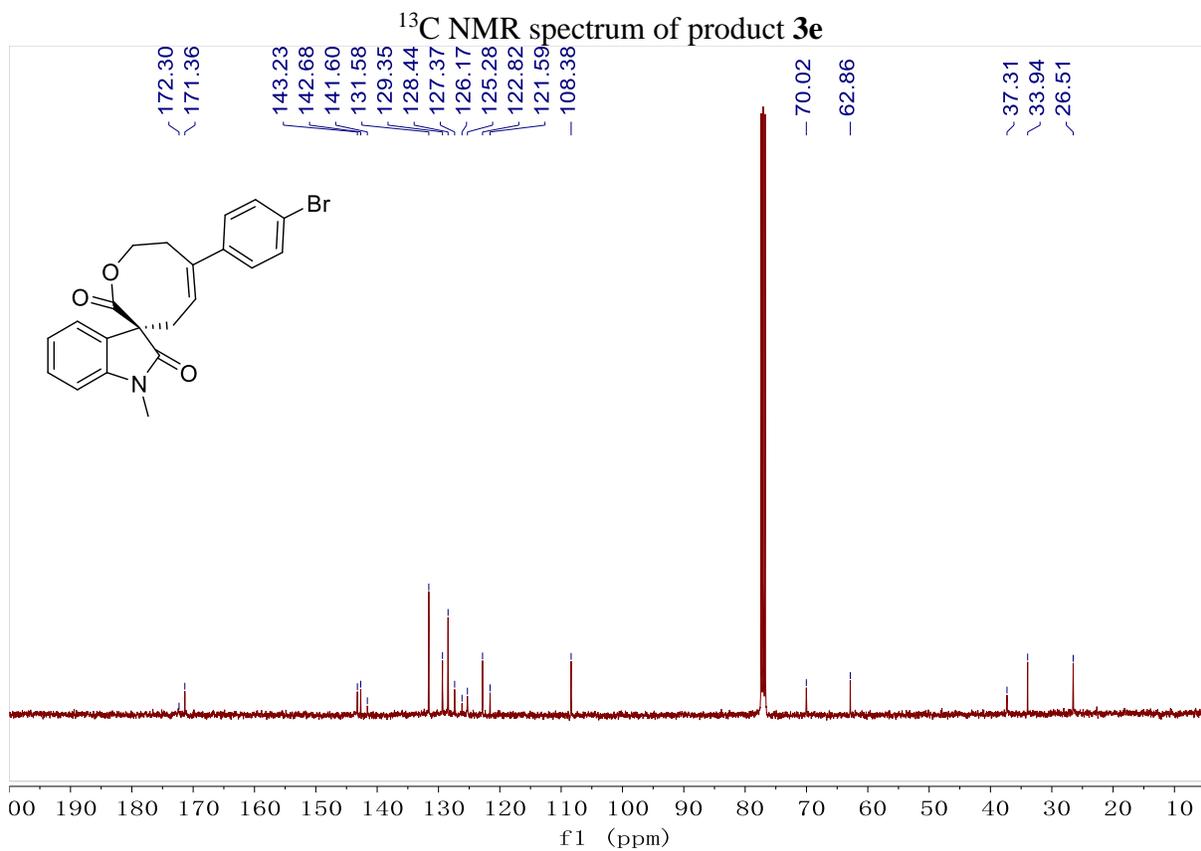


<sup>19</sup>F NMR spectrum of product **3d**

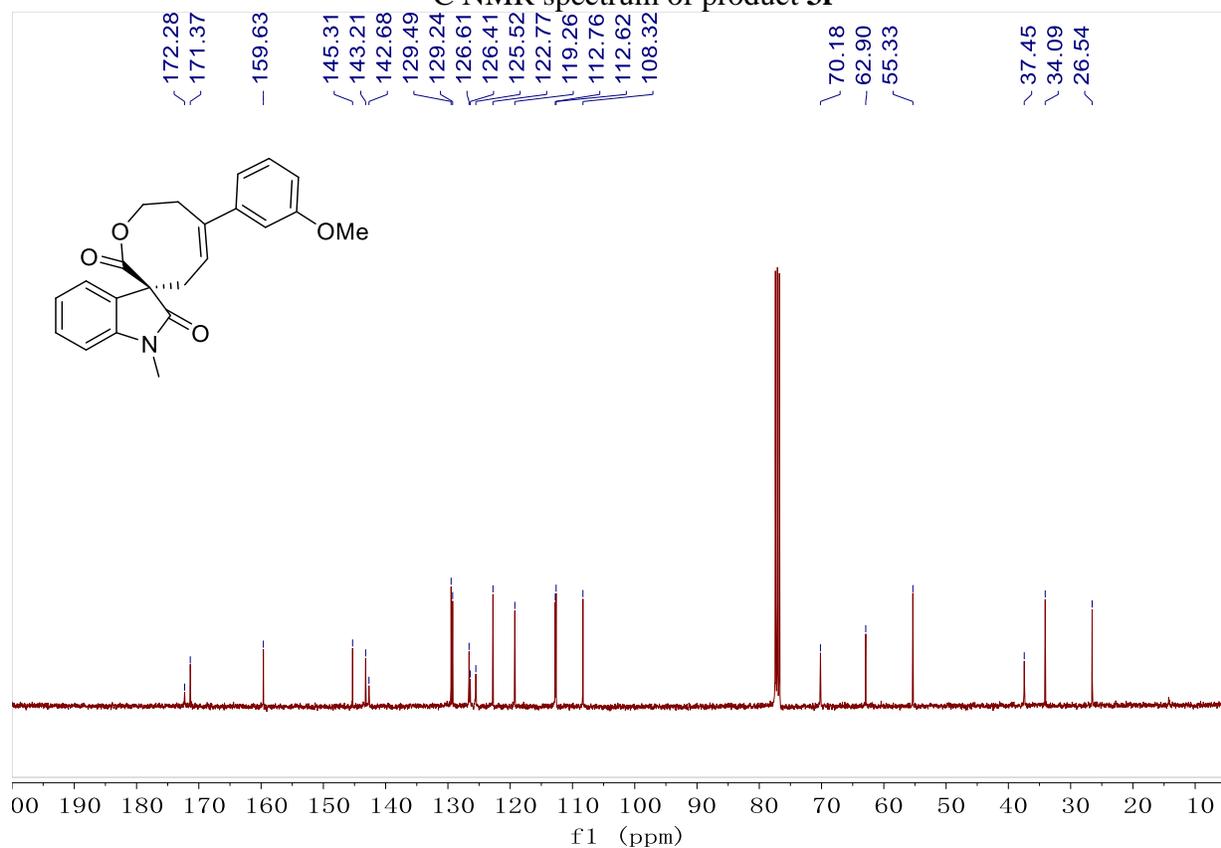


<sup>1</sup>H NMR spectrum of product **3e**

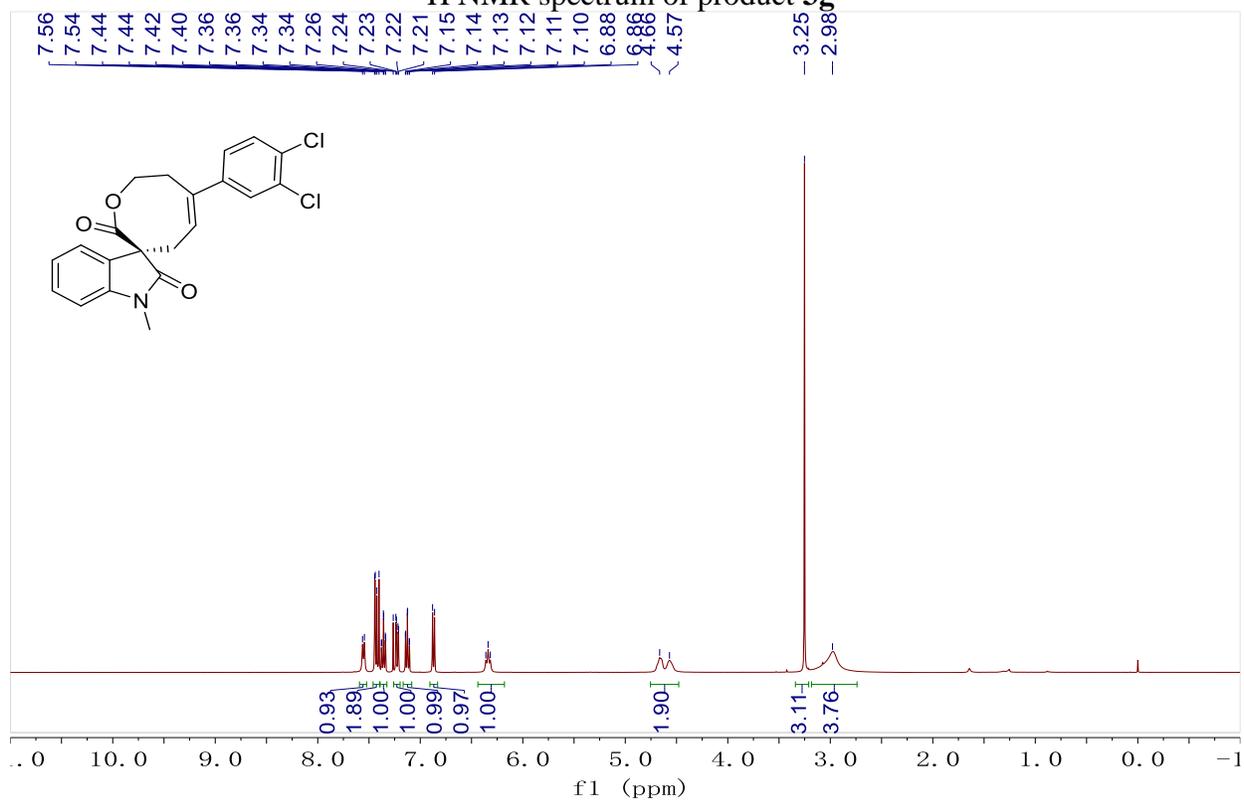


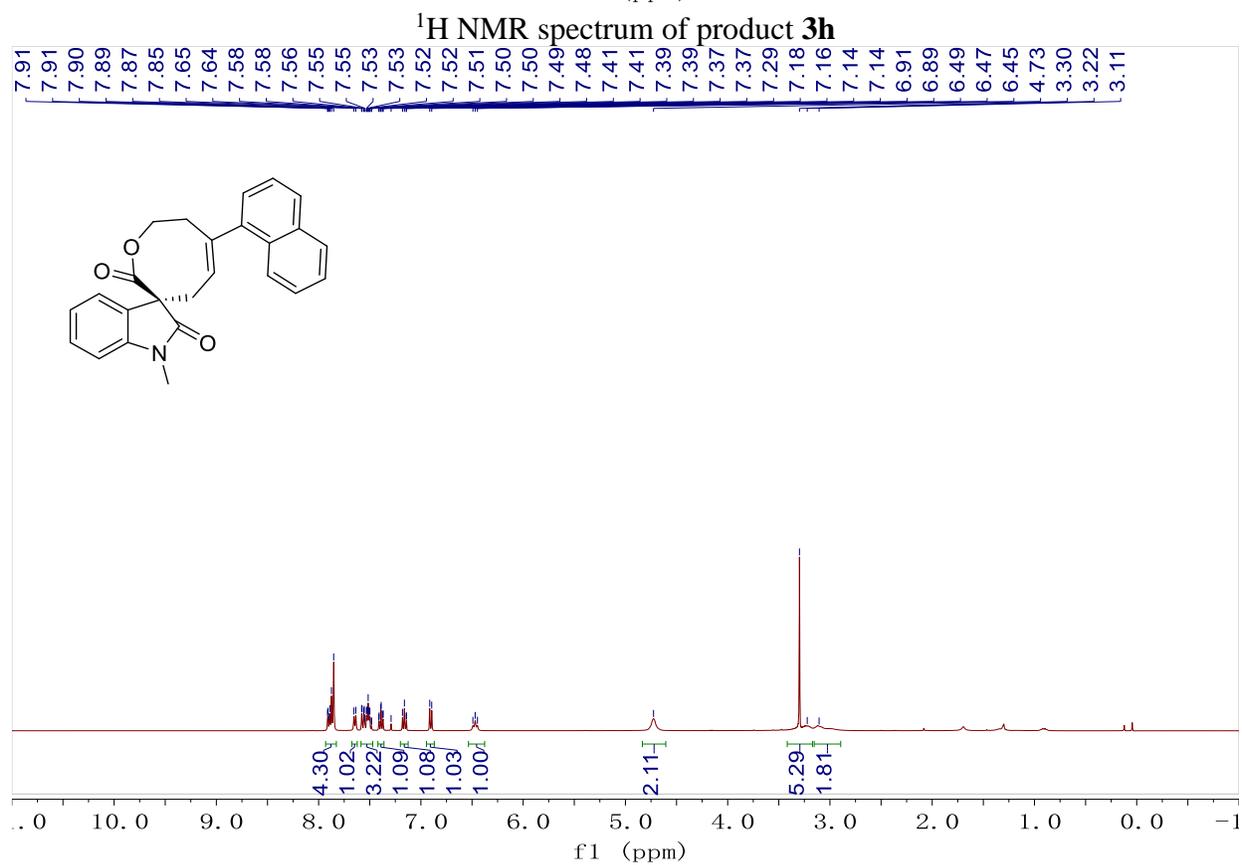
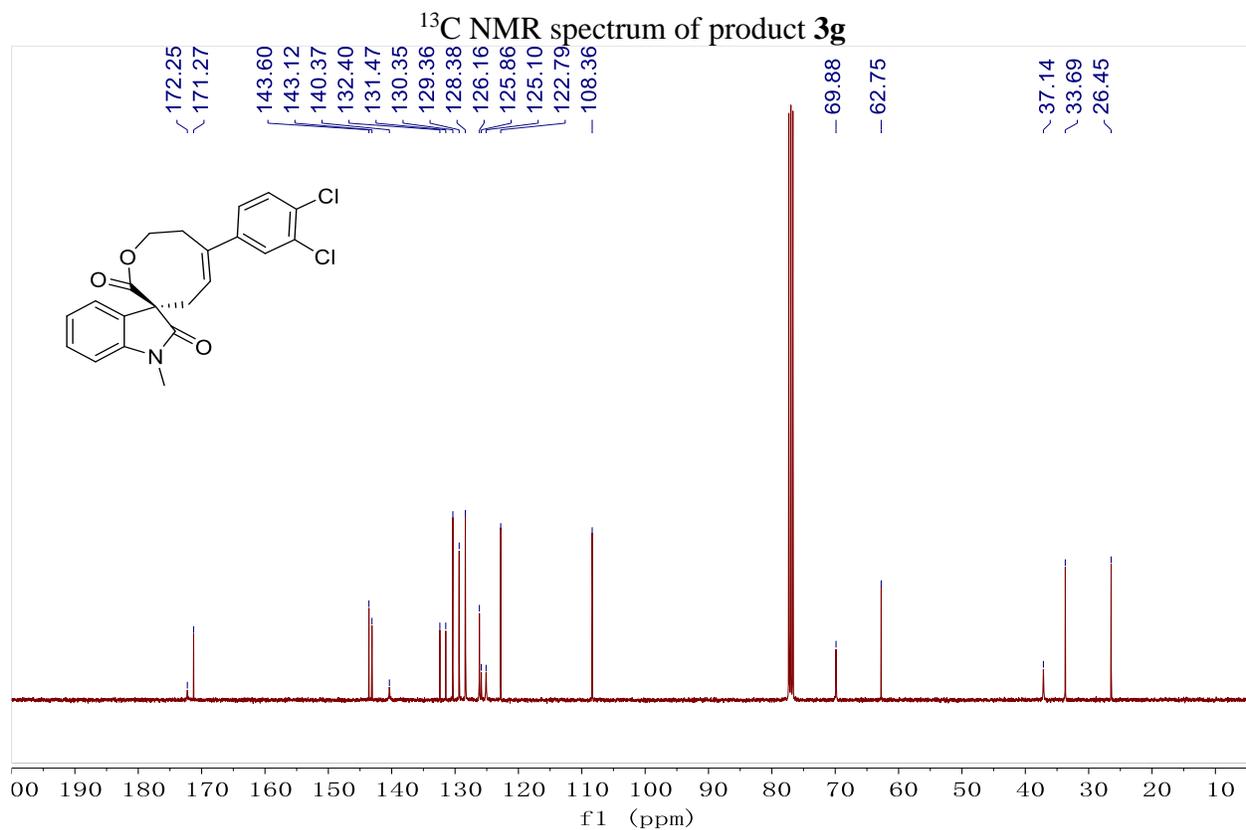


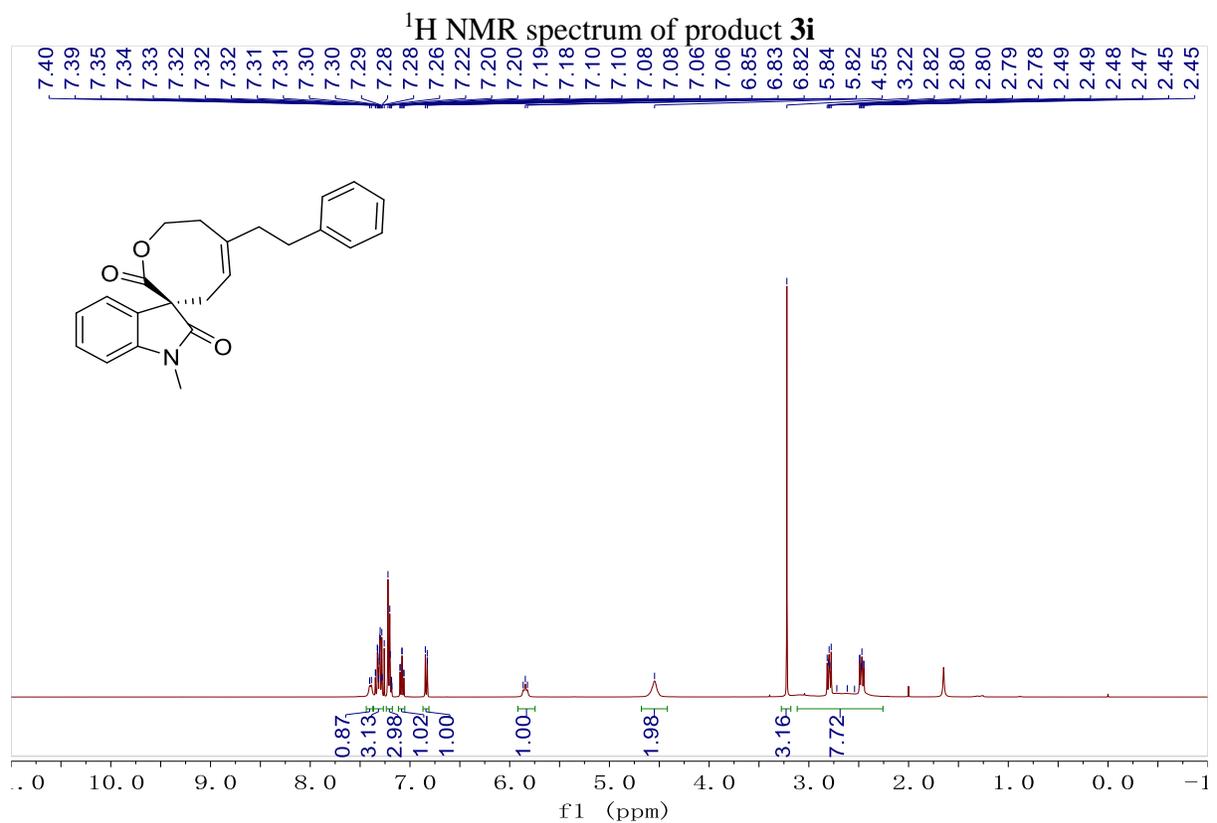
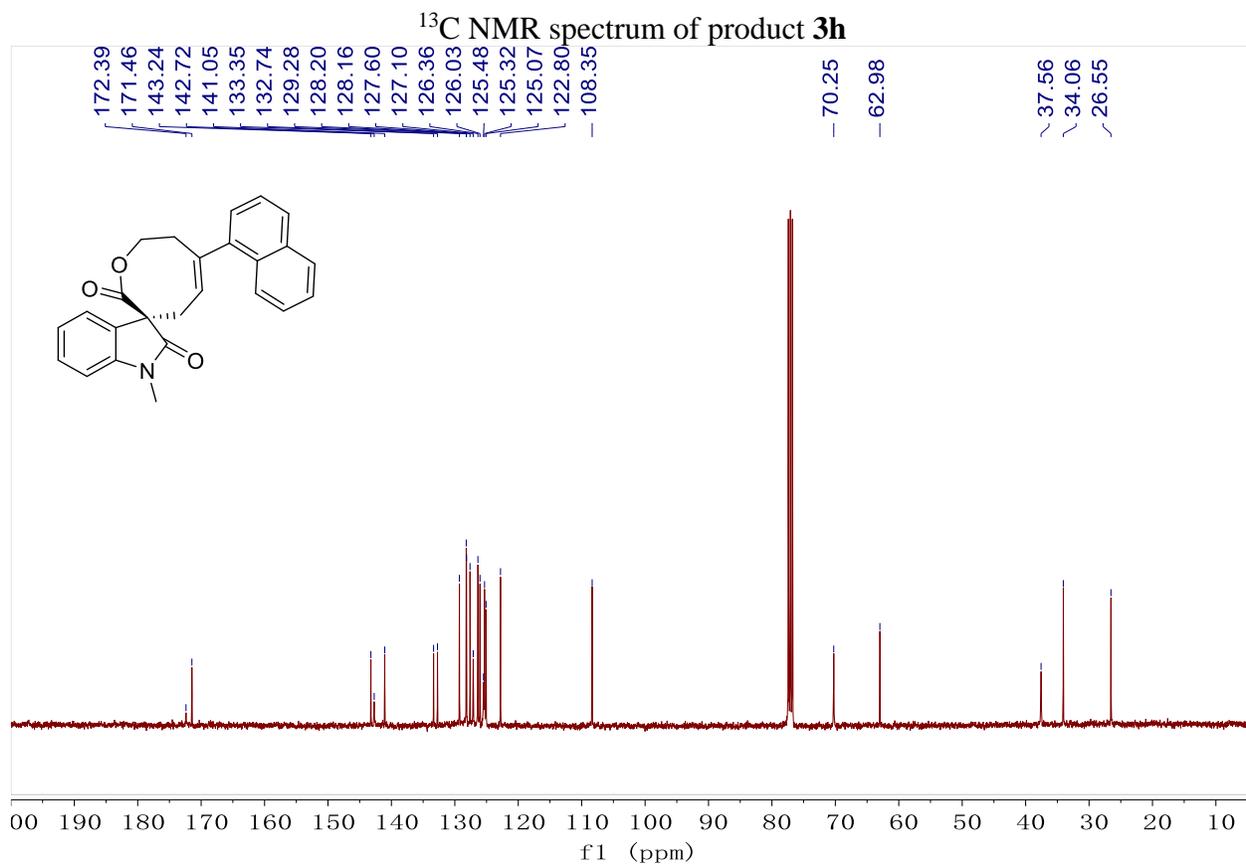
<sup>13</sup>C NMR spectrum of product **3f**

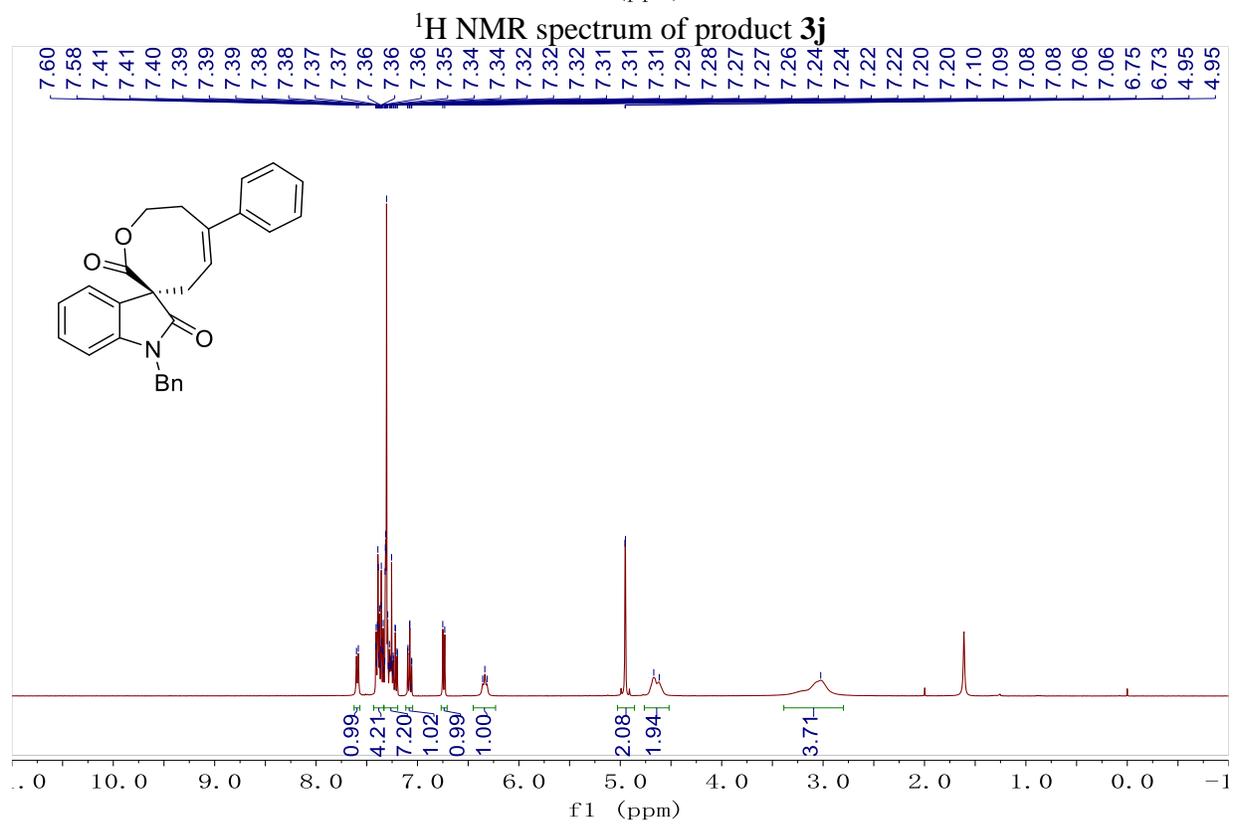
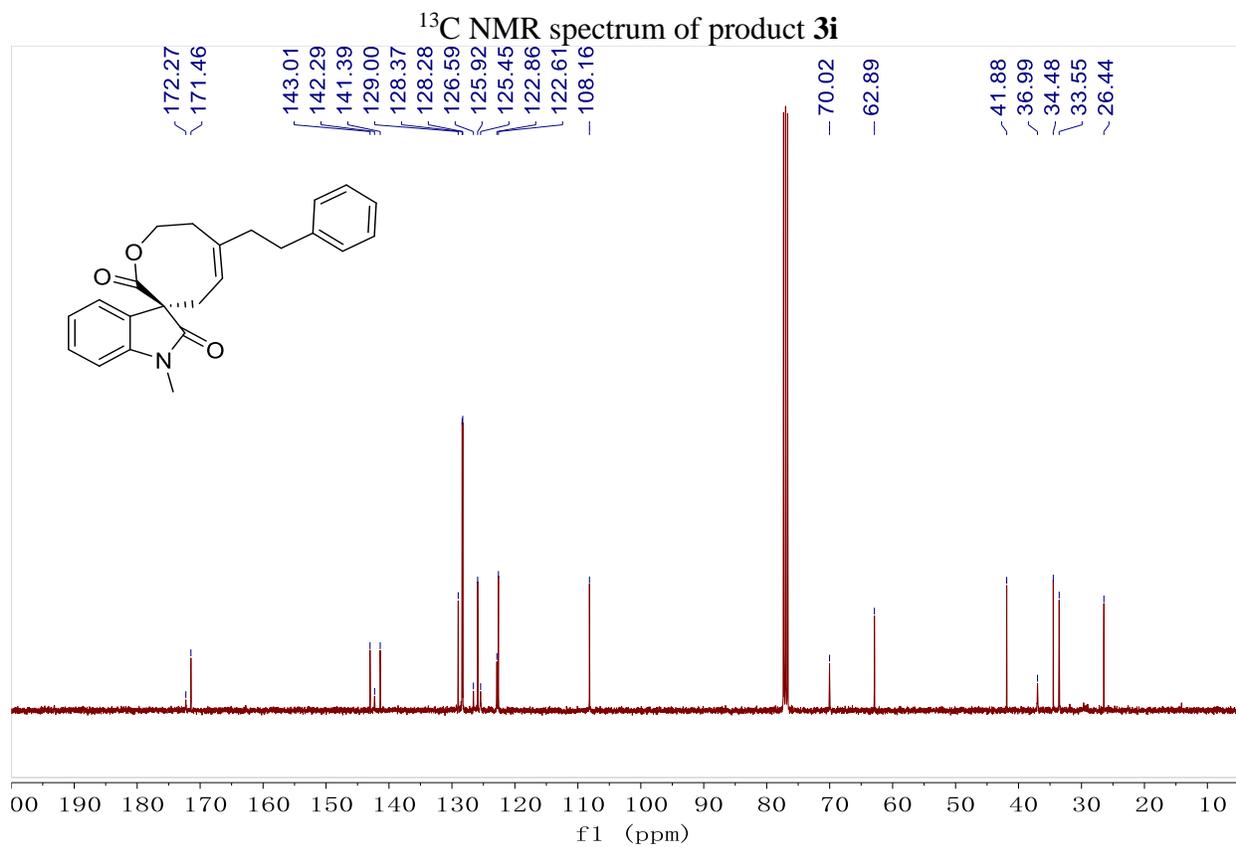


<sup>1</sup>H NMR spectrum of product **3g**

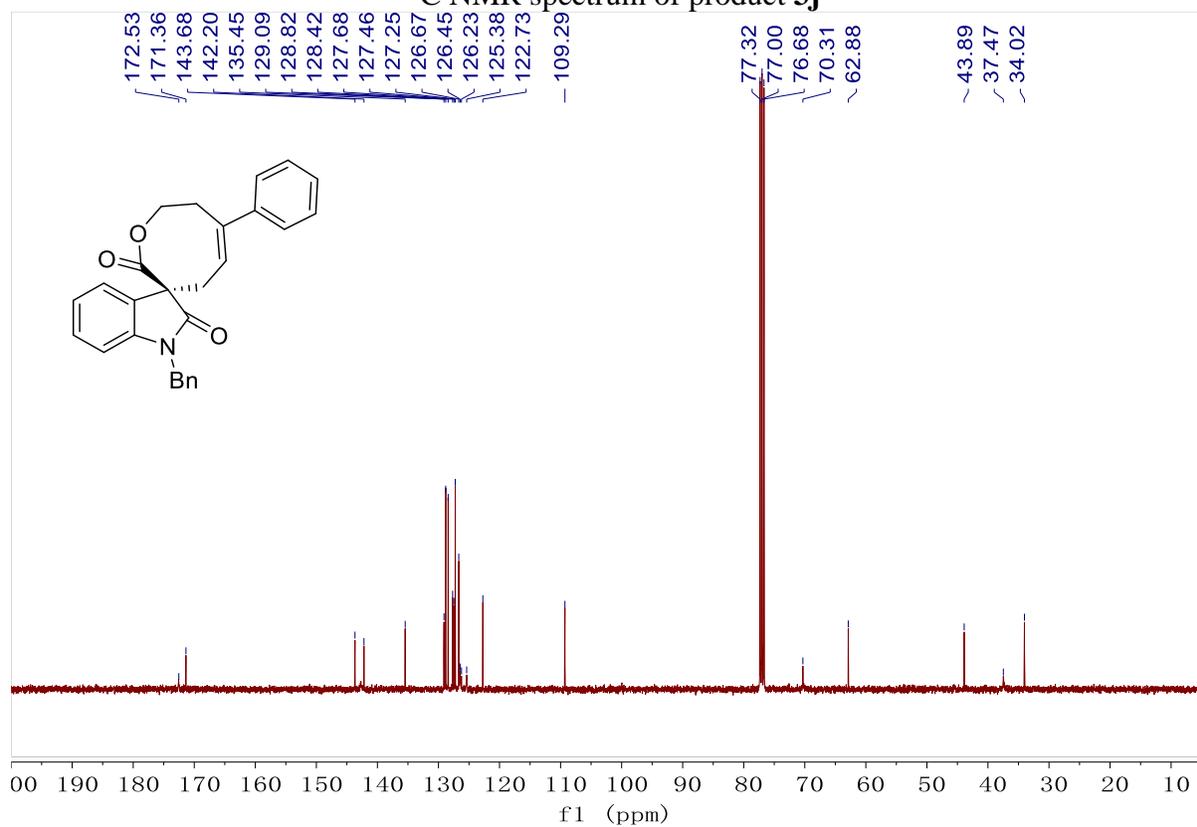




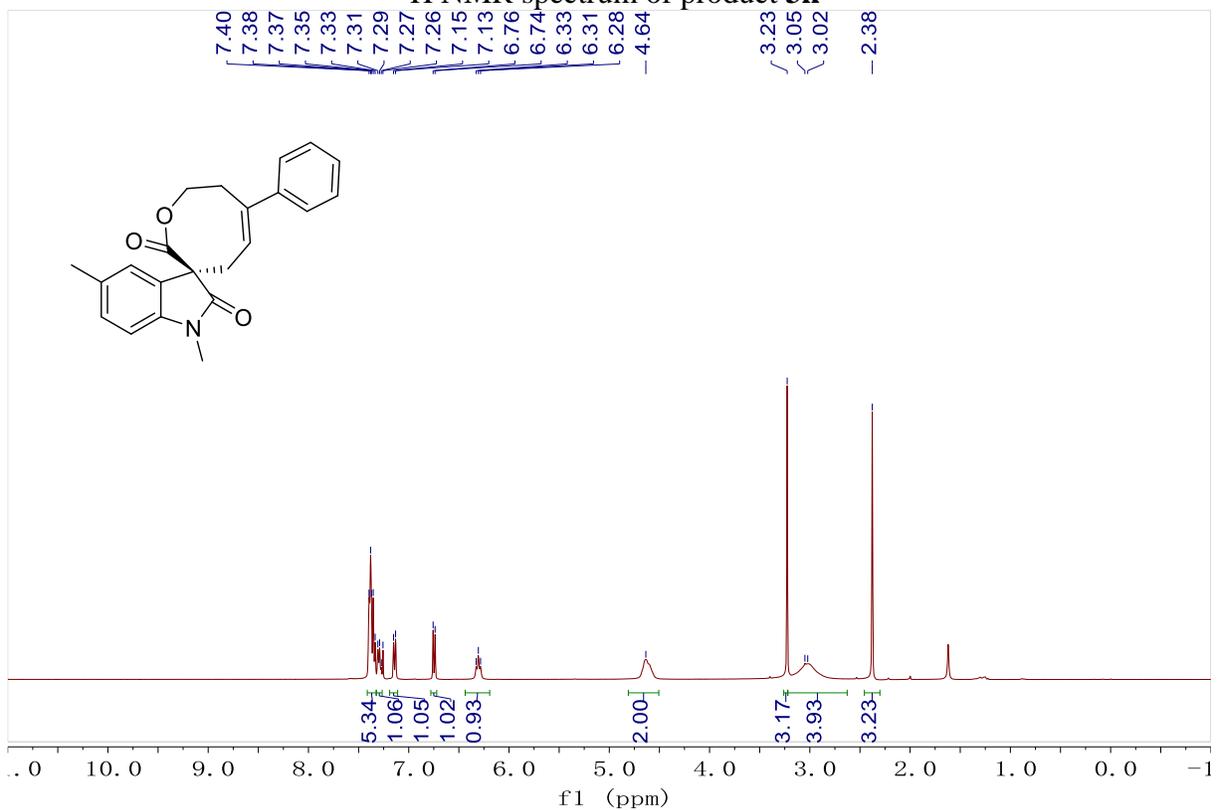




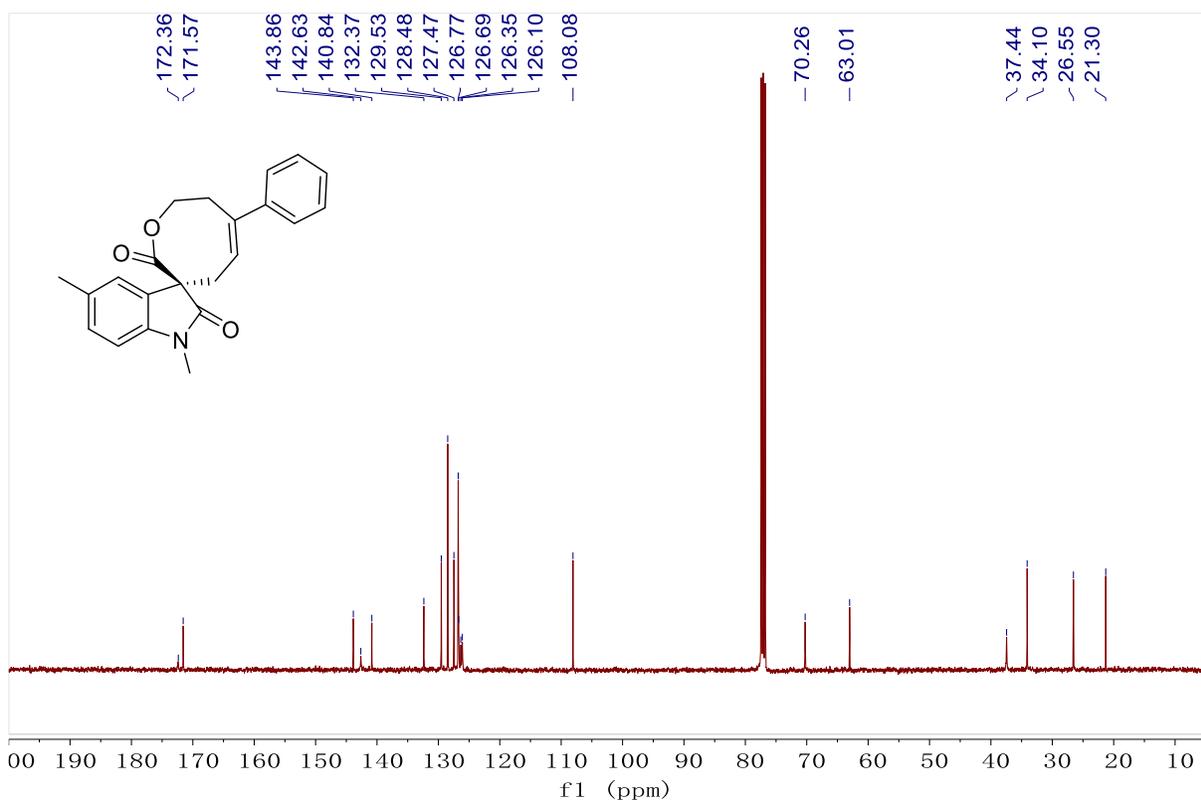
<sup>13</sup>C NMR spectrum of product **3j**



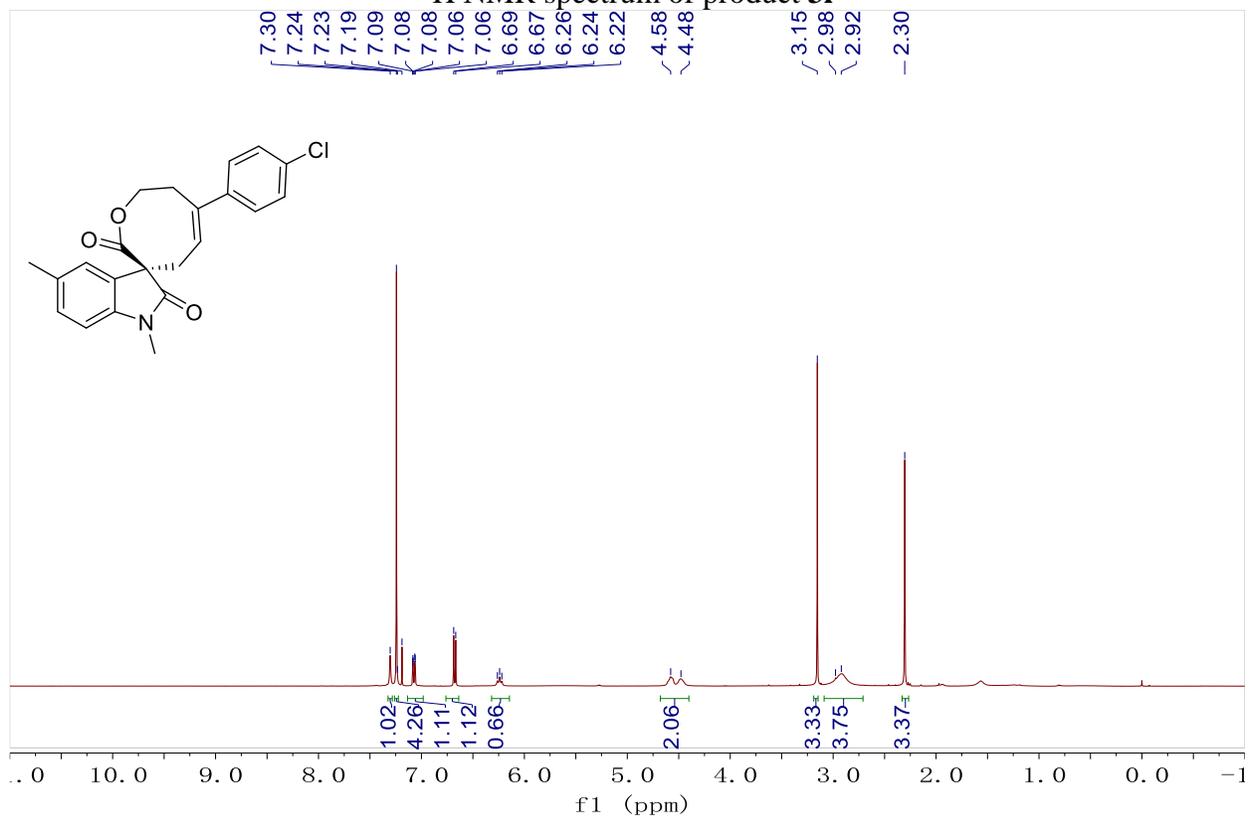
<sup>1</sup>H NMR spectrum of product **3k**



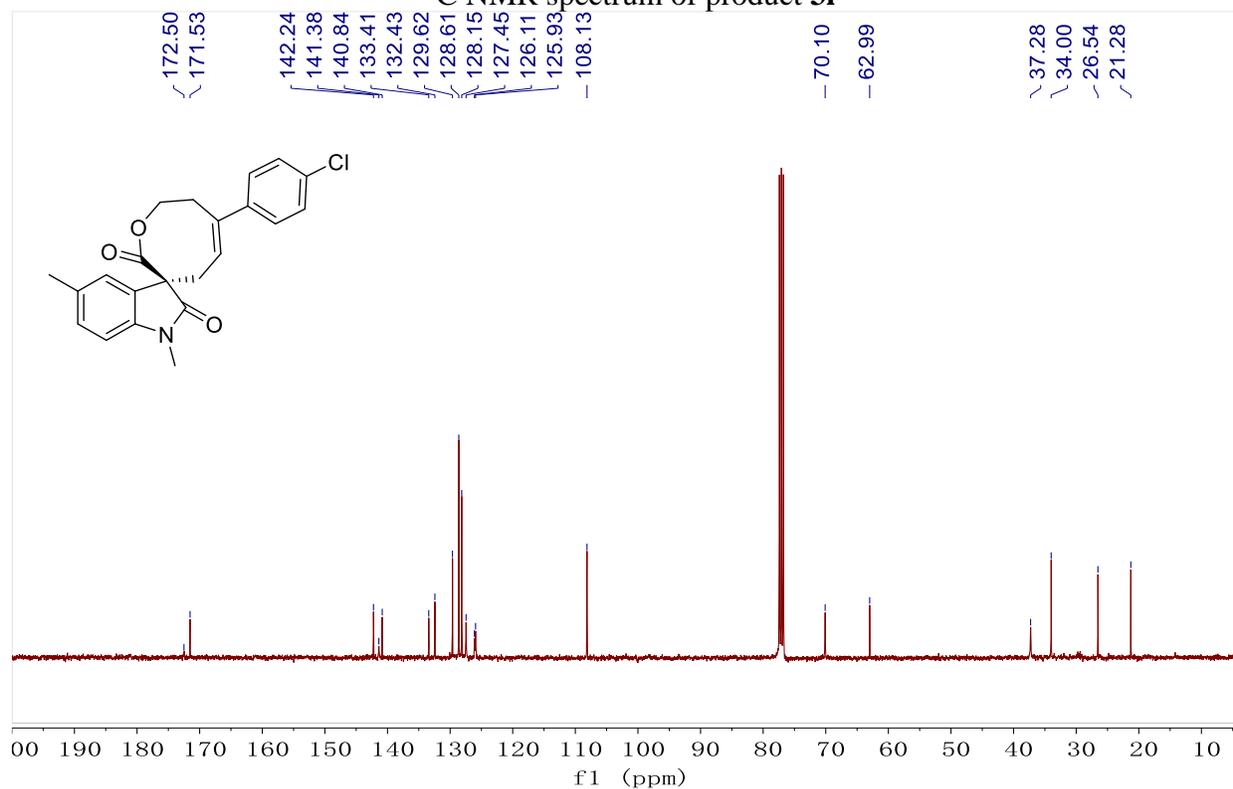
<sup>13</sup>C NMR spectrum of product **3k**



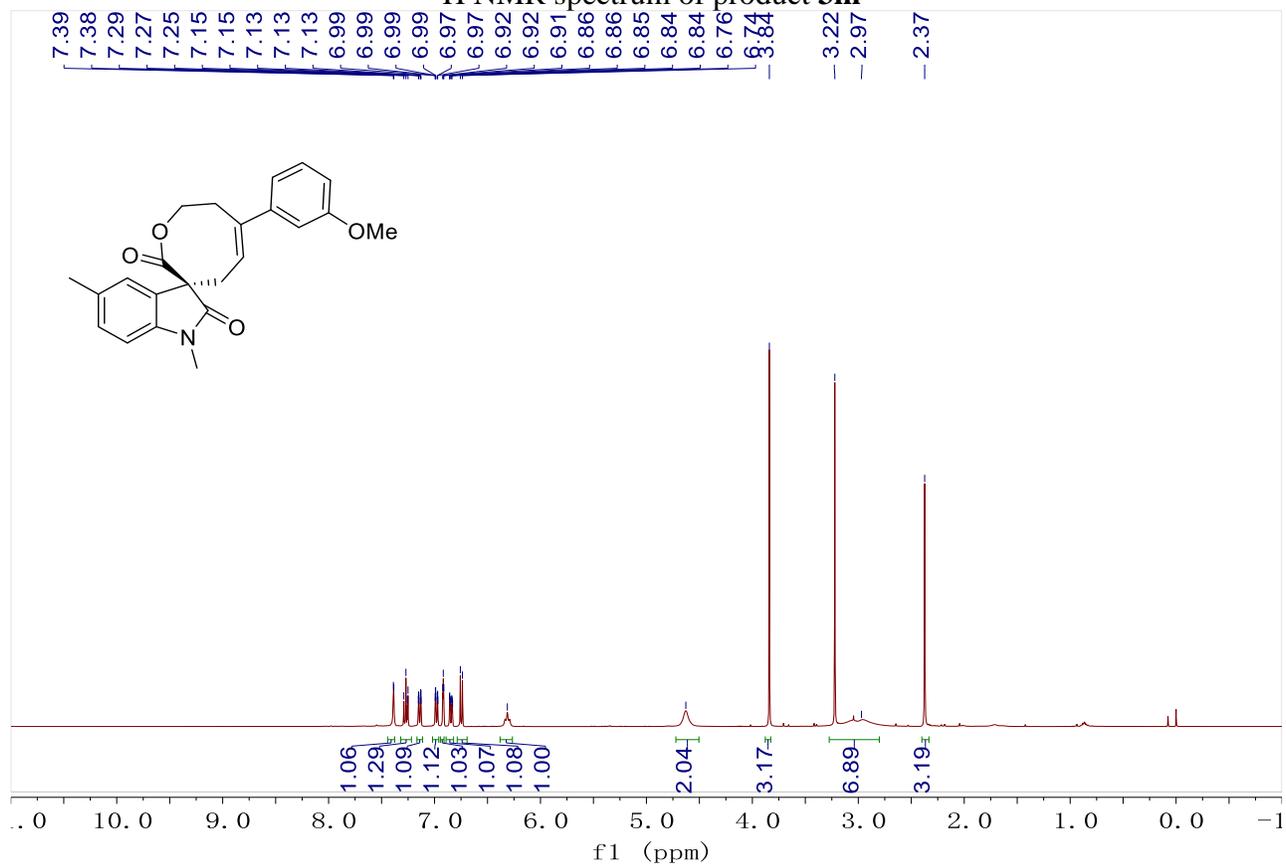
<sup>1</sup>H NMR spectrum of product **3l**

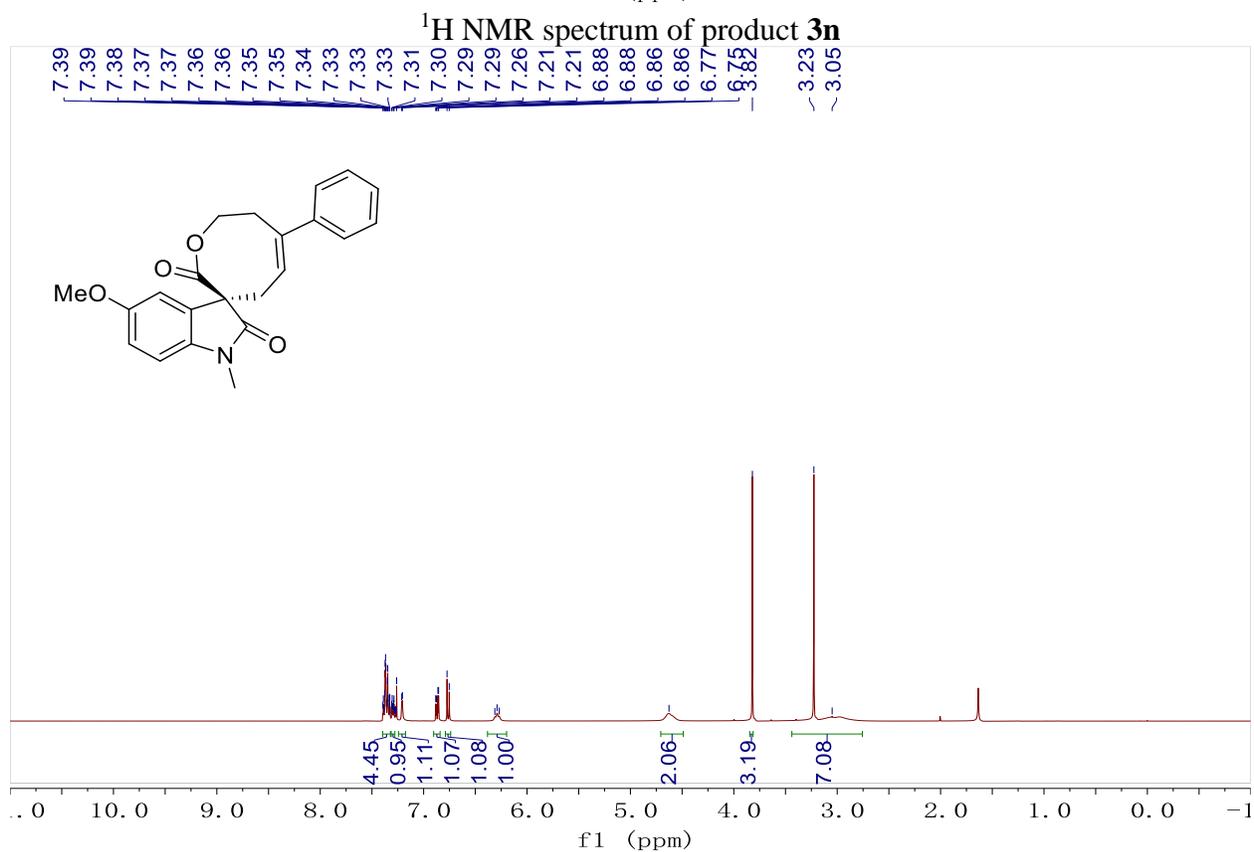
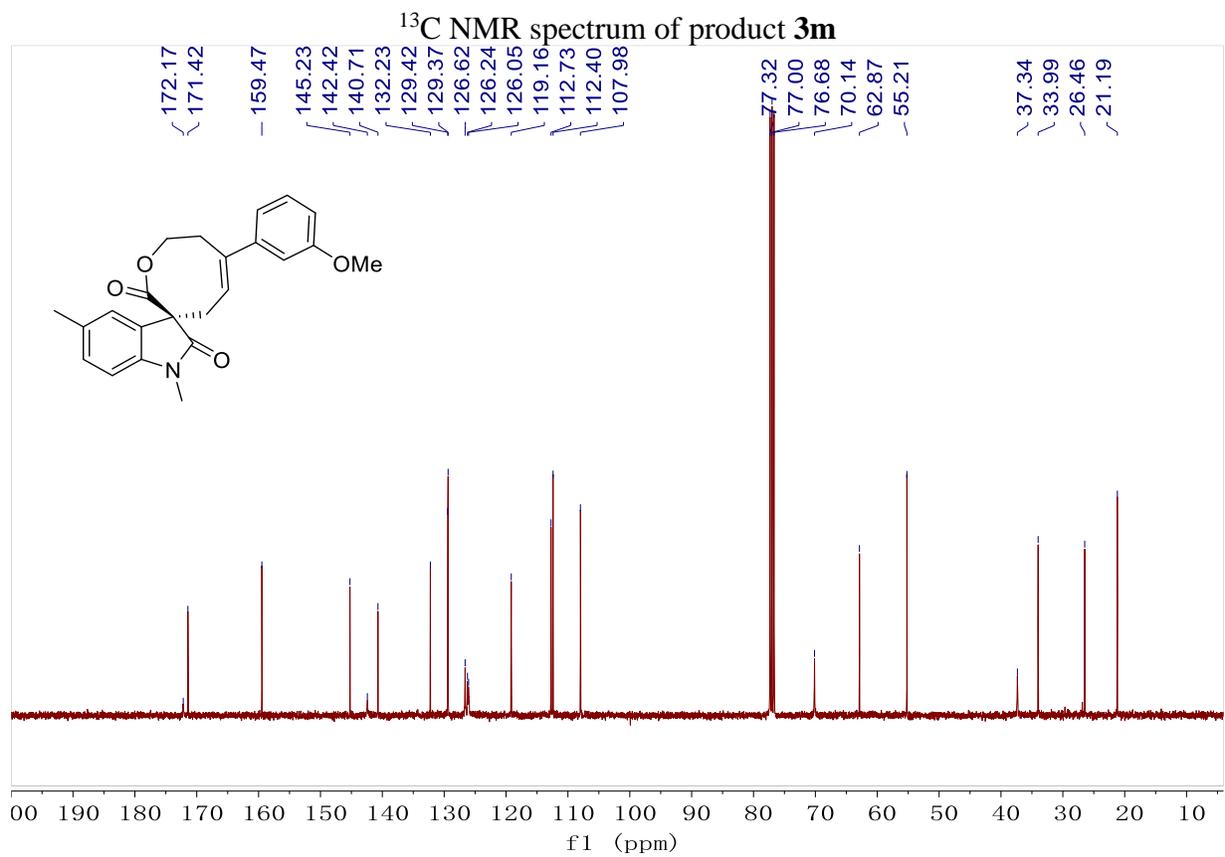


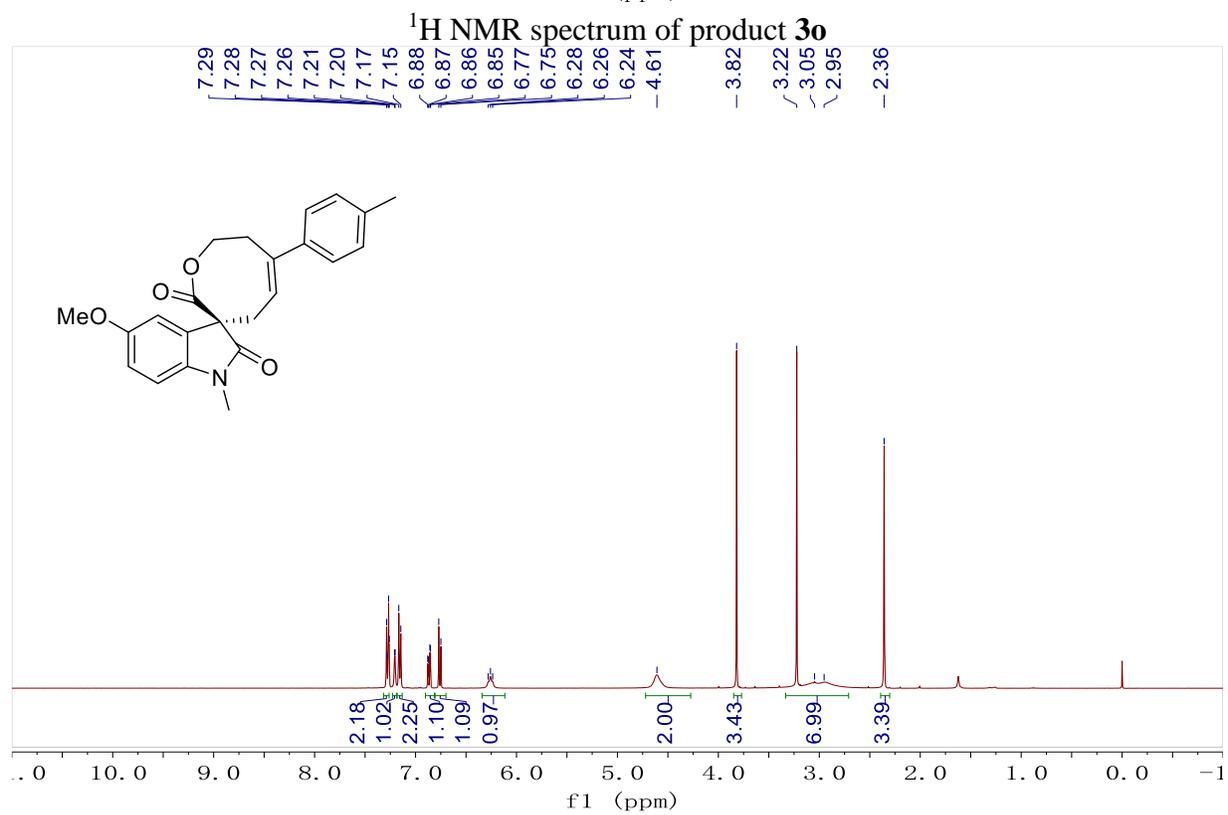
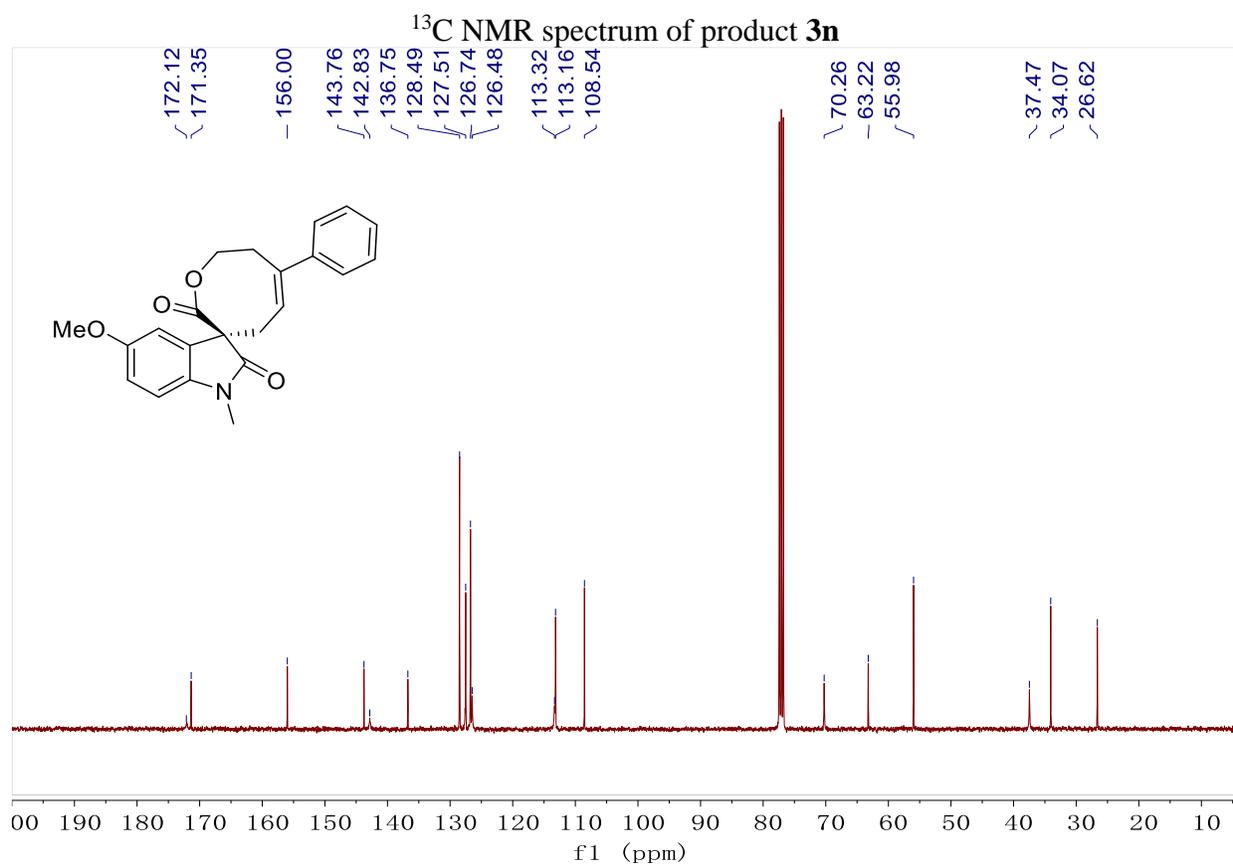
<sup>13</sup>C NMR spectrum of product **3l**



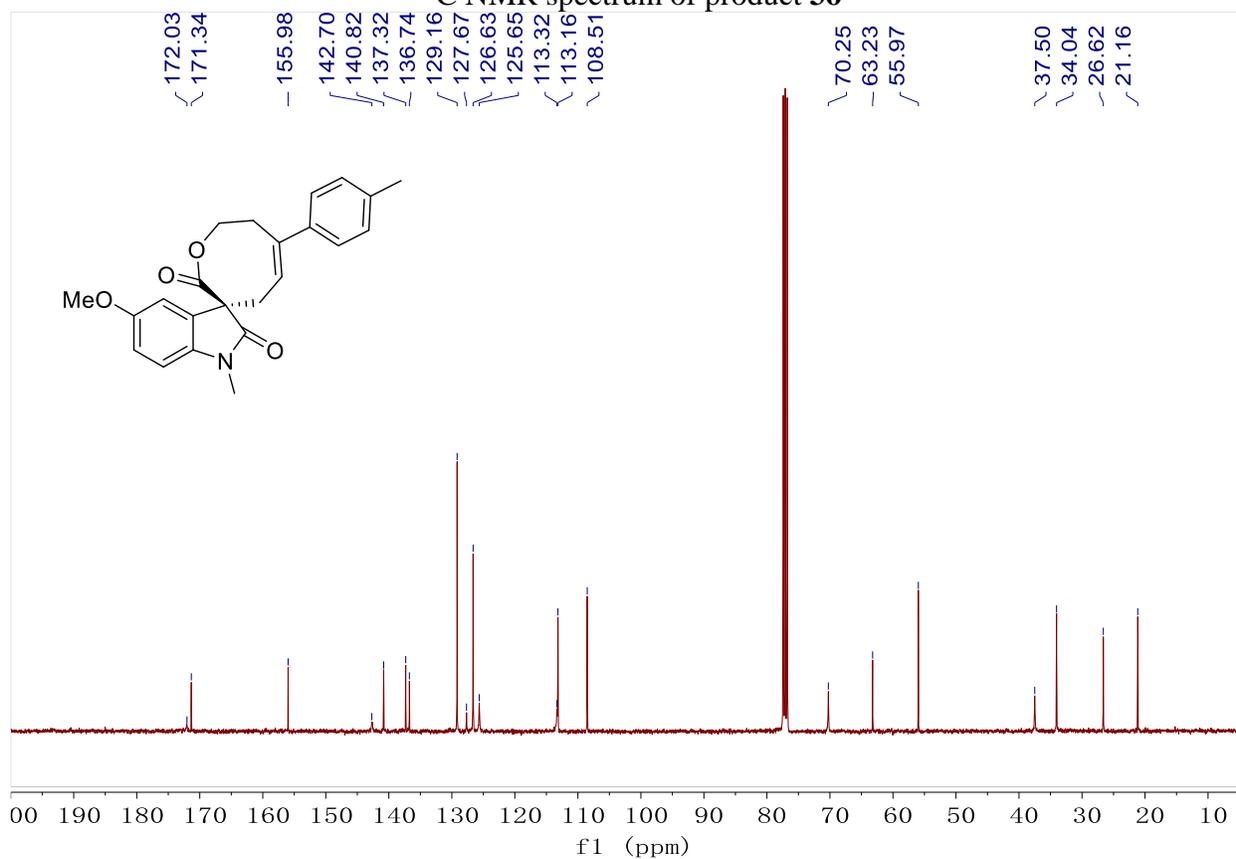
<sup>1</sup>H NMR spectrum of product **3m**



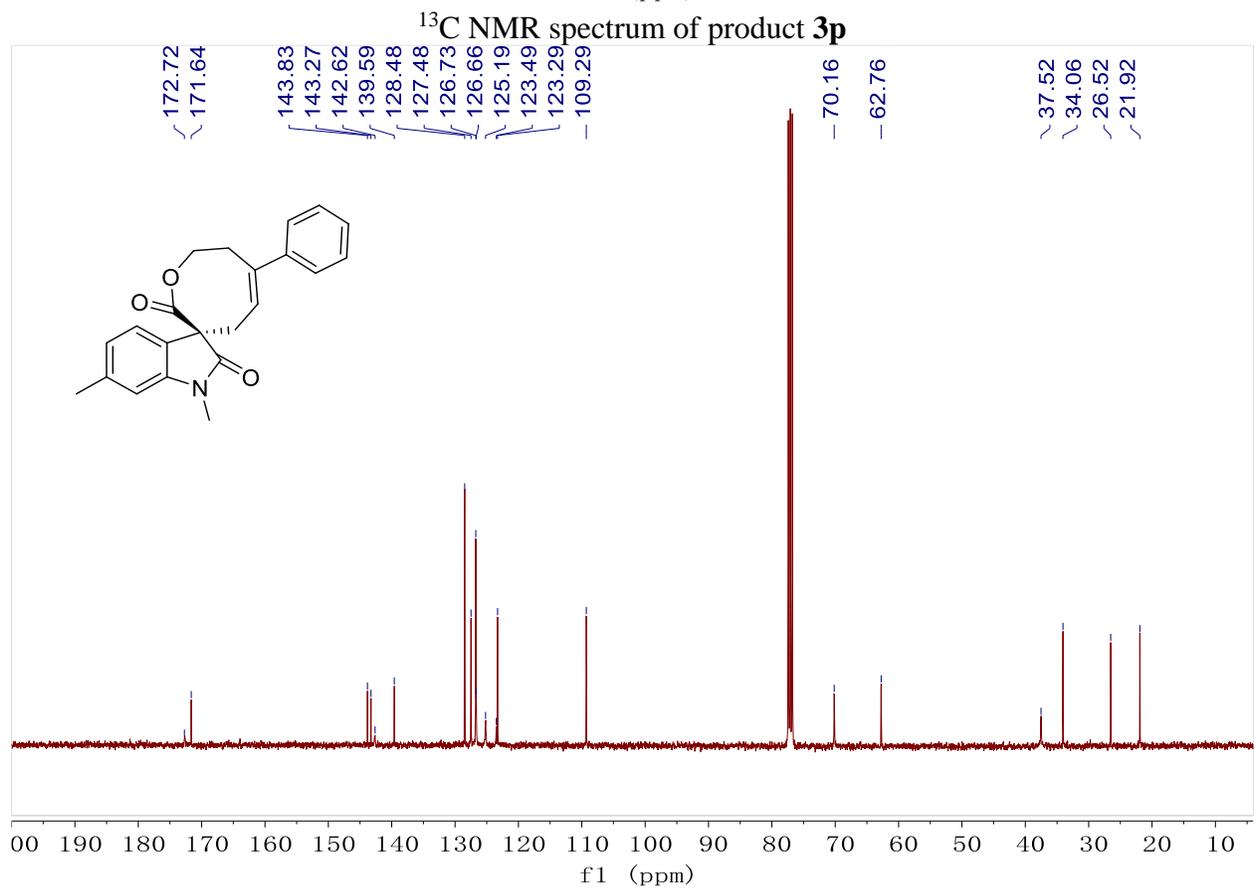
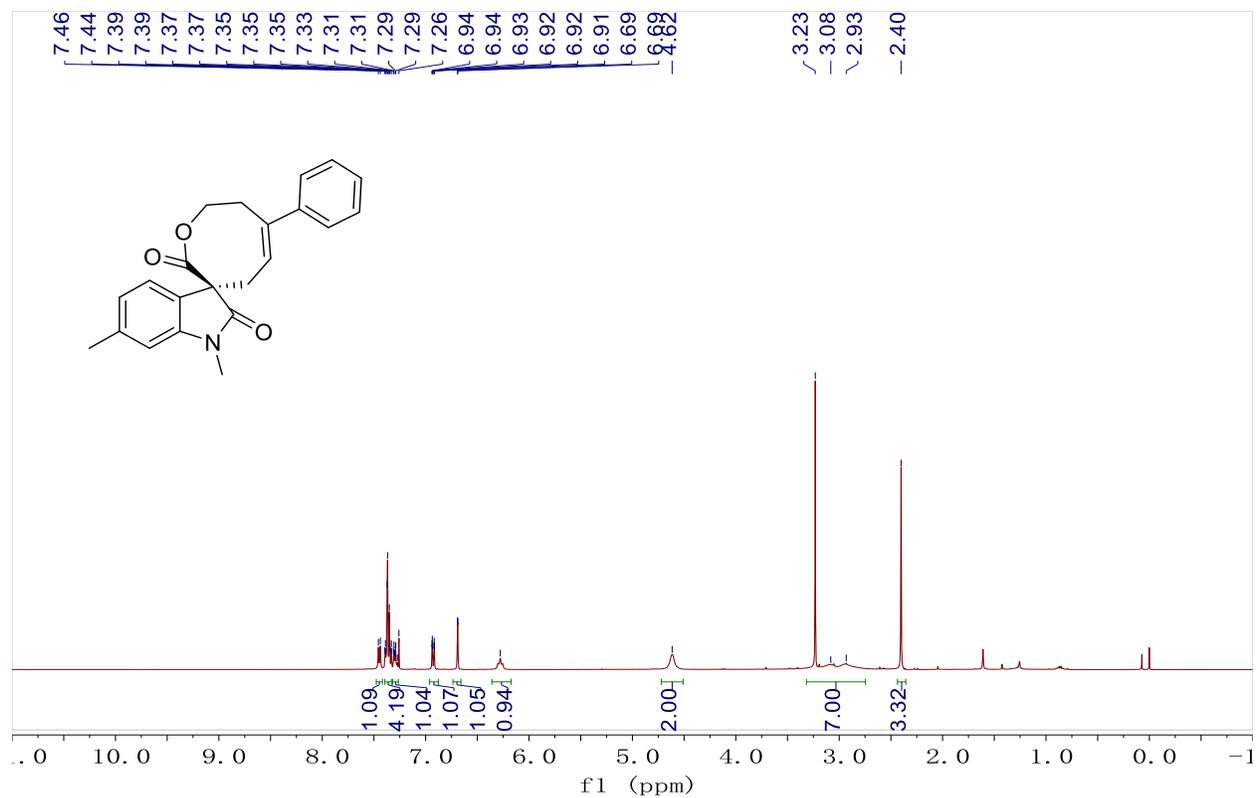




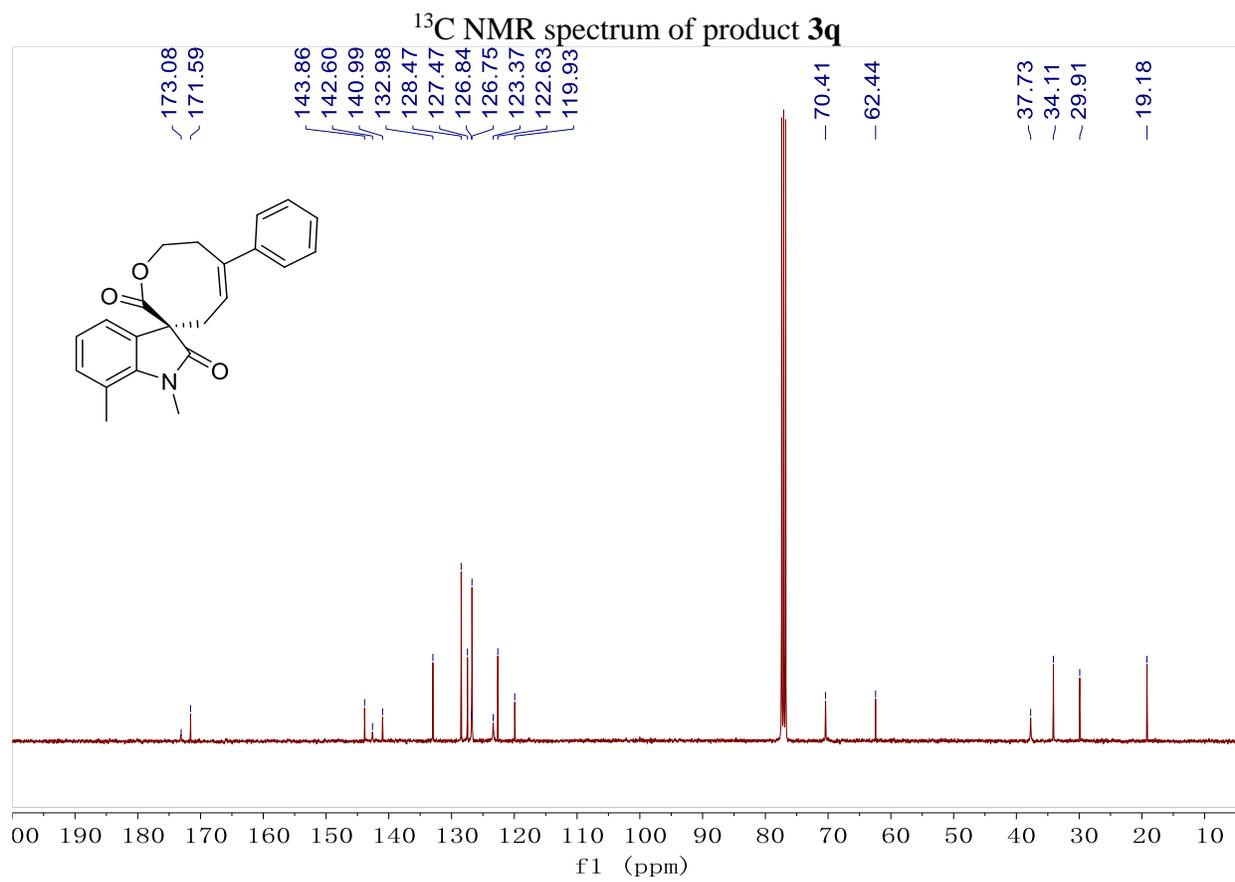
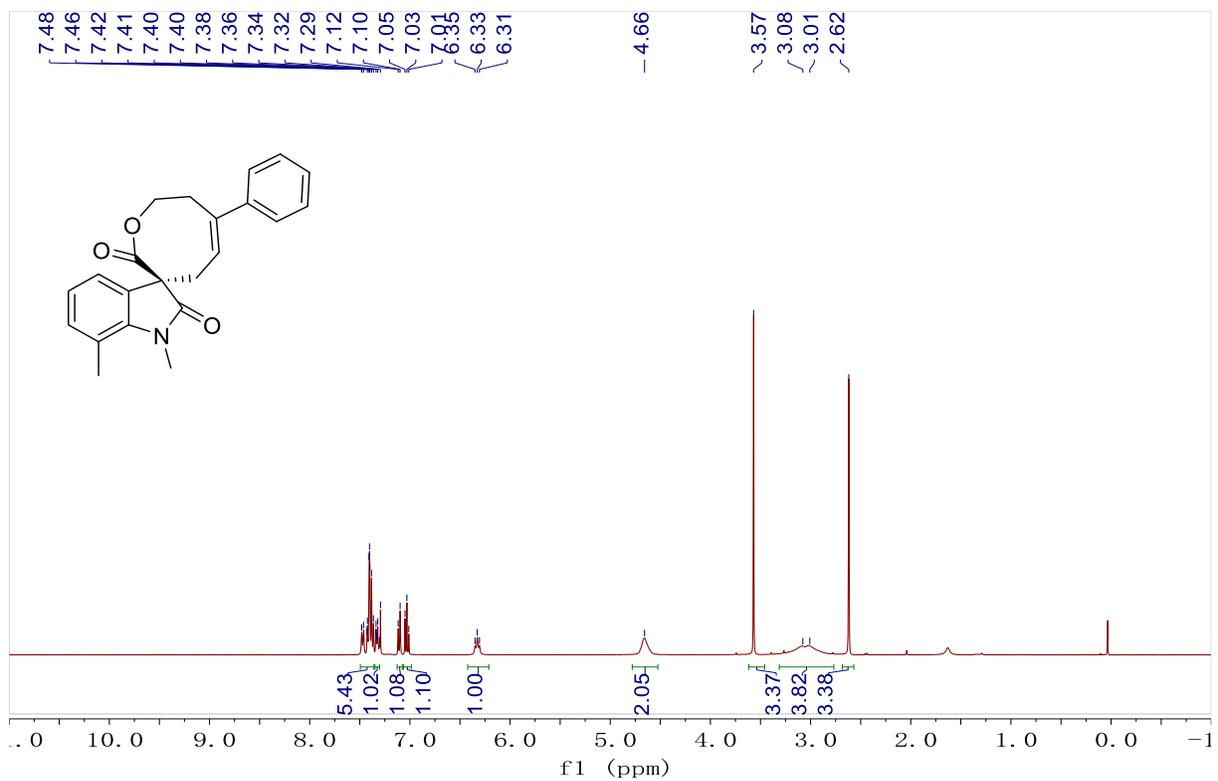
<sup>13</sup>C NMR spectrum of product **3o**



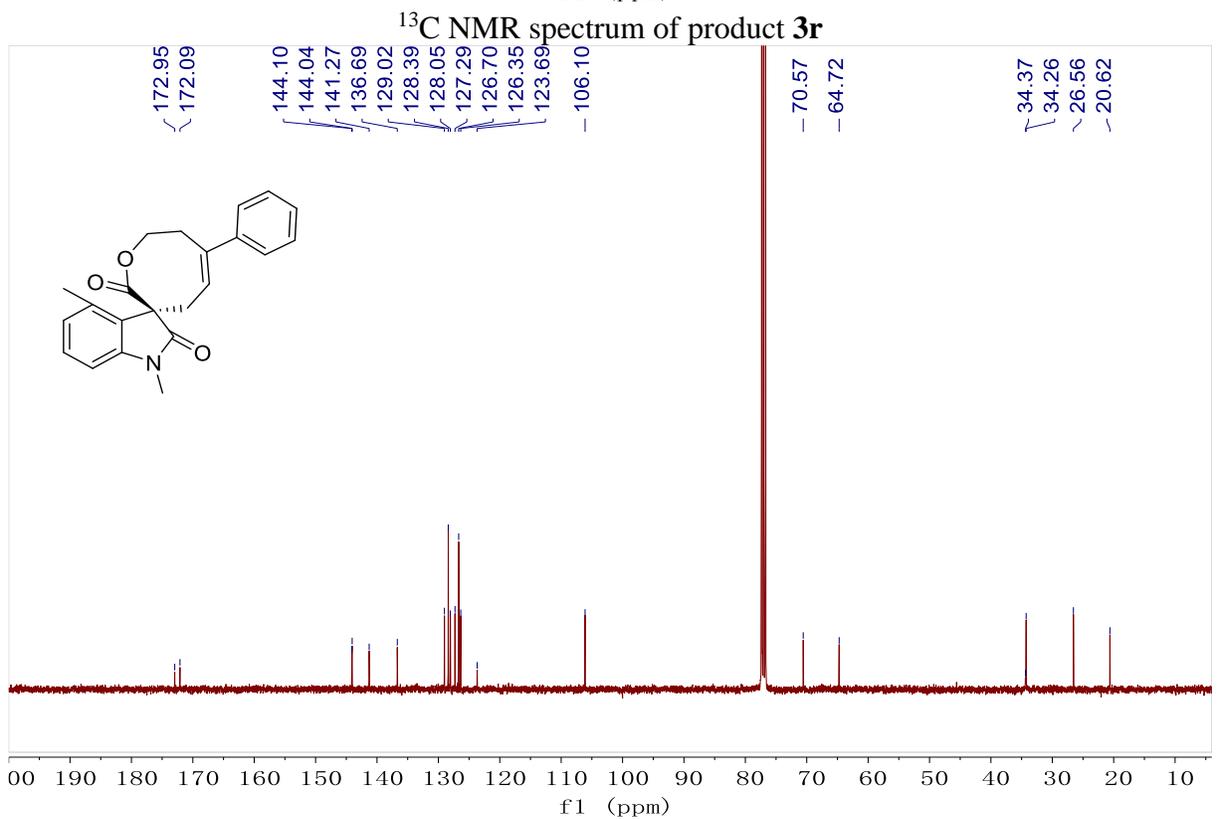
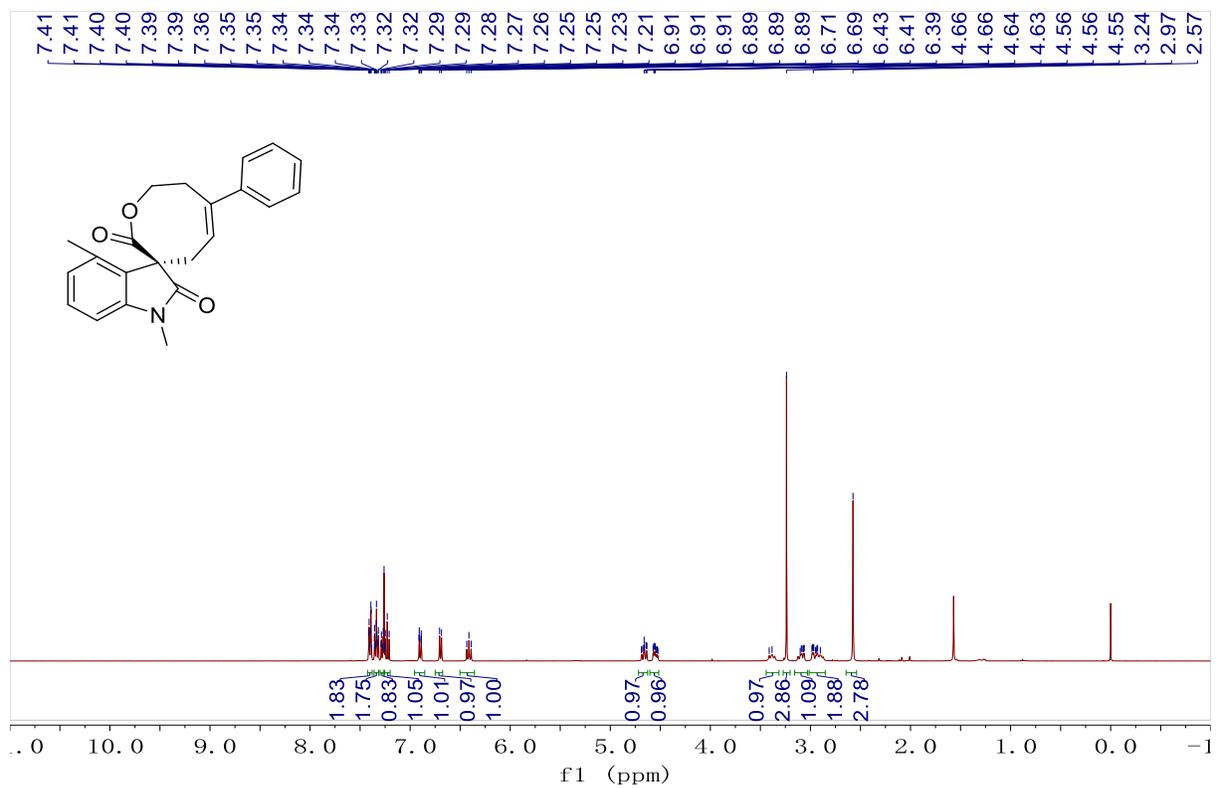
<sup>1</sup>H NMR spectrum of product **3p**



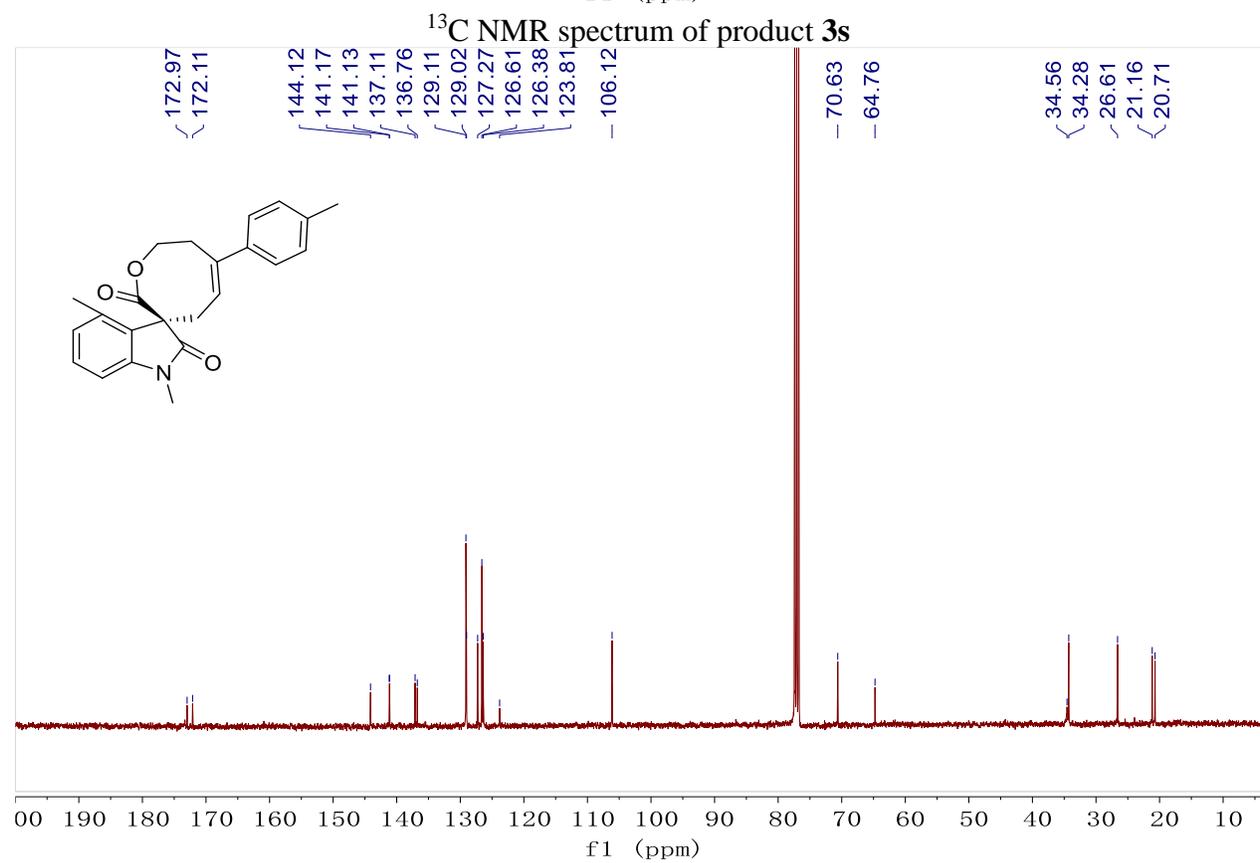
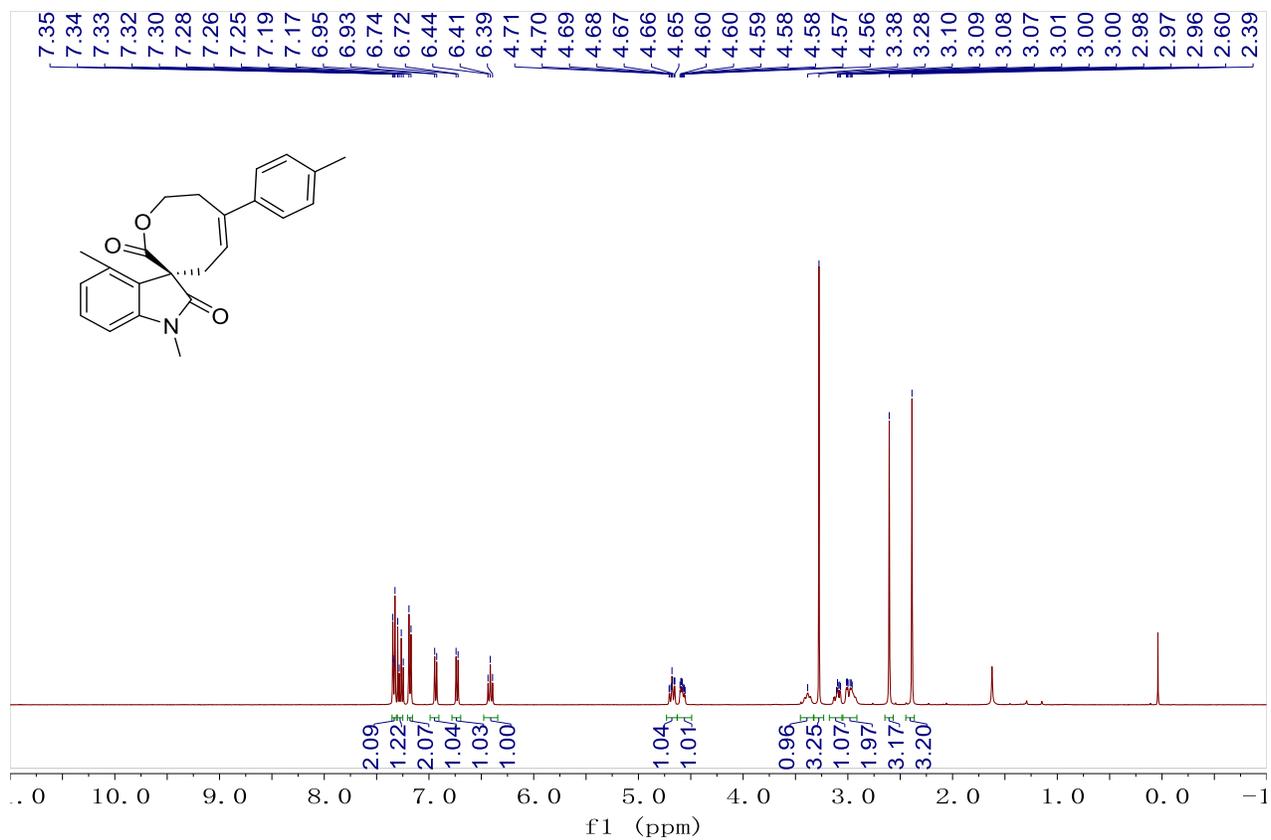
**<sup>1</sup>H NMR spectrum of product 3q**



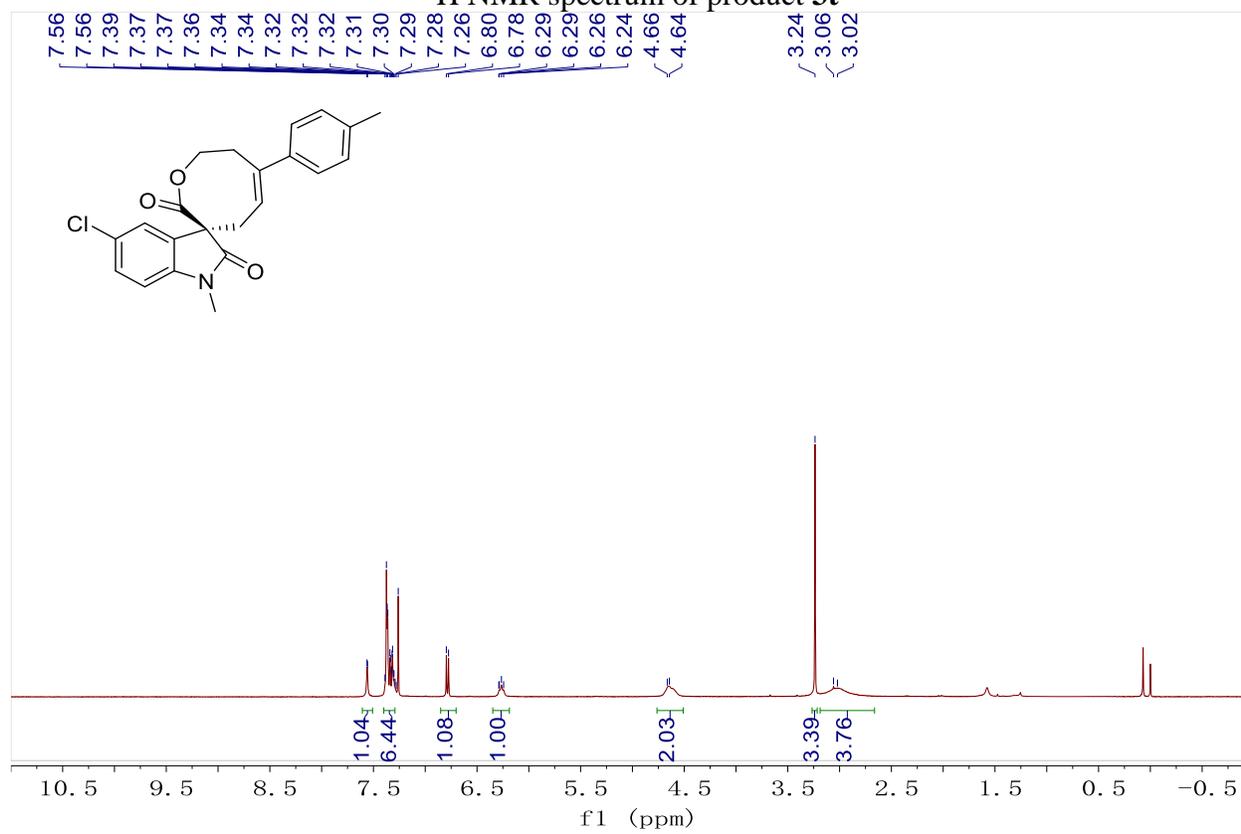
**<sup>1</sup>H NMR spectrum of product 3r**



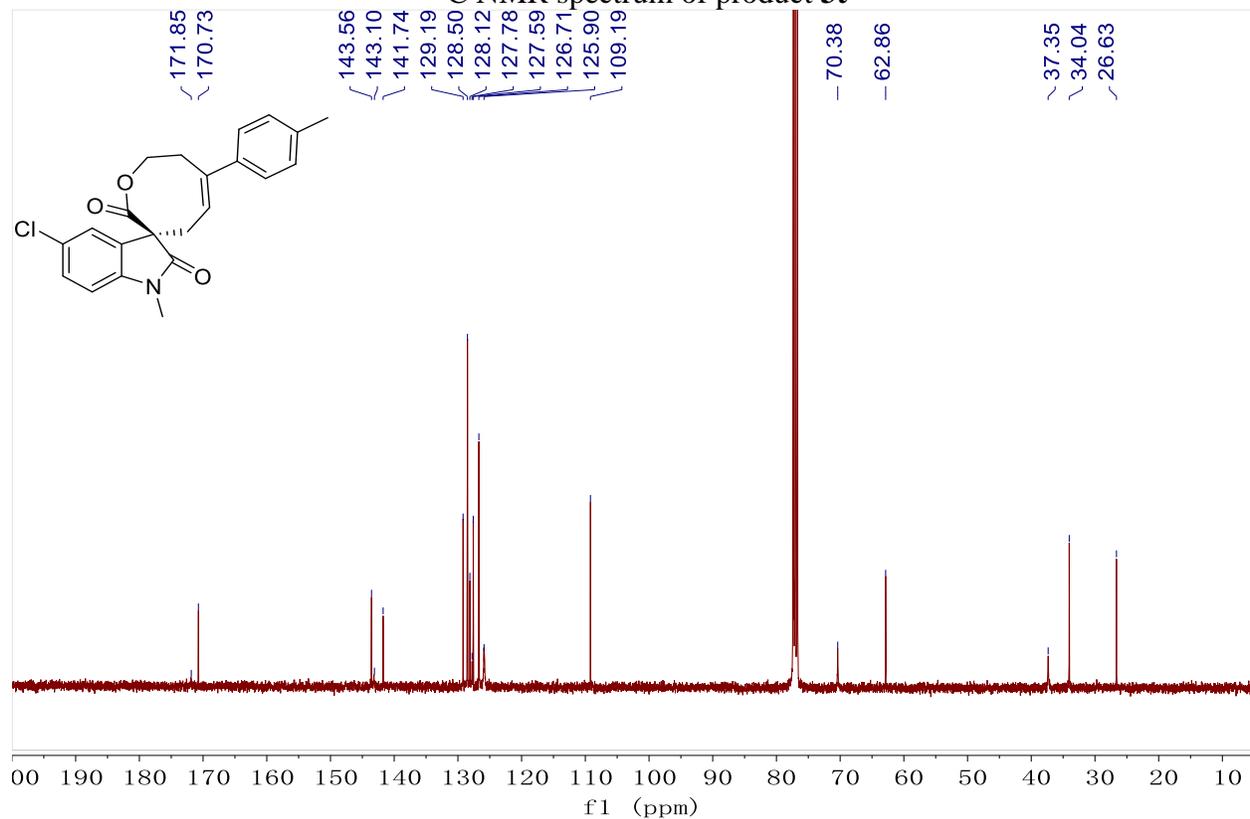
**<sup>1</sup>H NMR spectrum of product 3s**

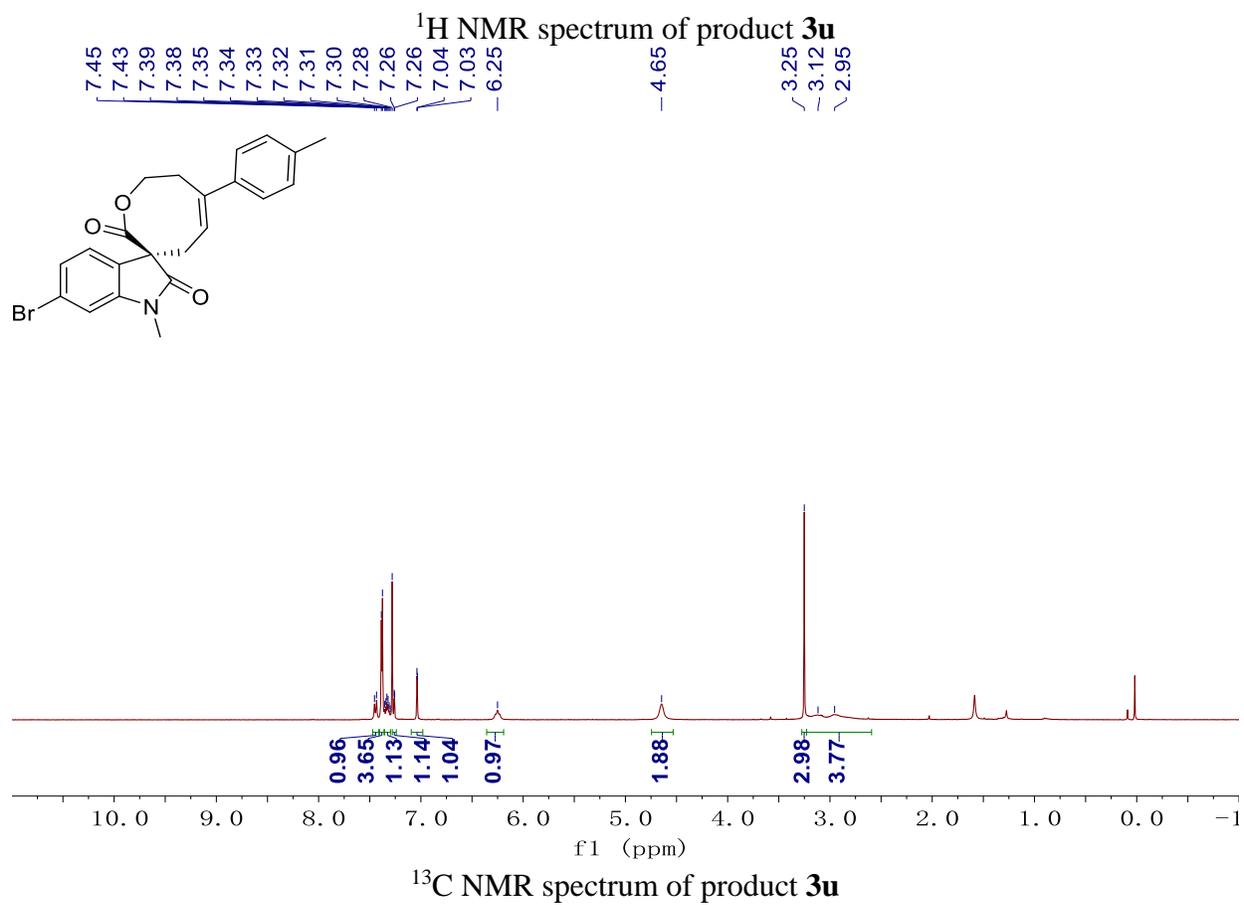


<sup>1</sup>H NMR spectrum of product **3t**

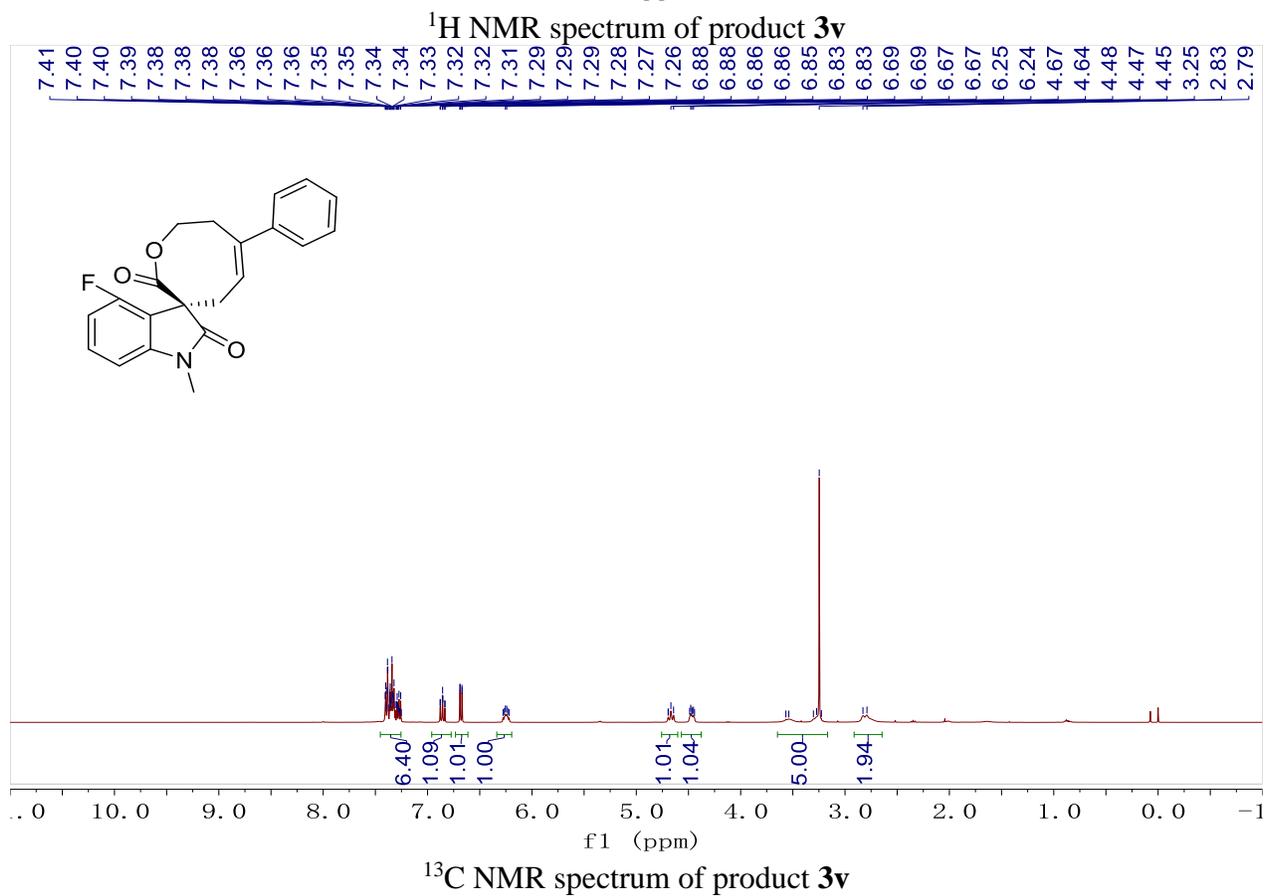
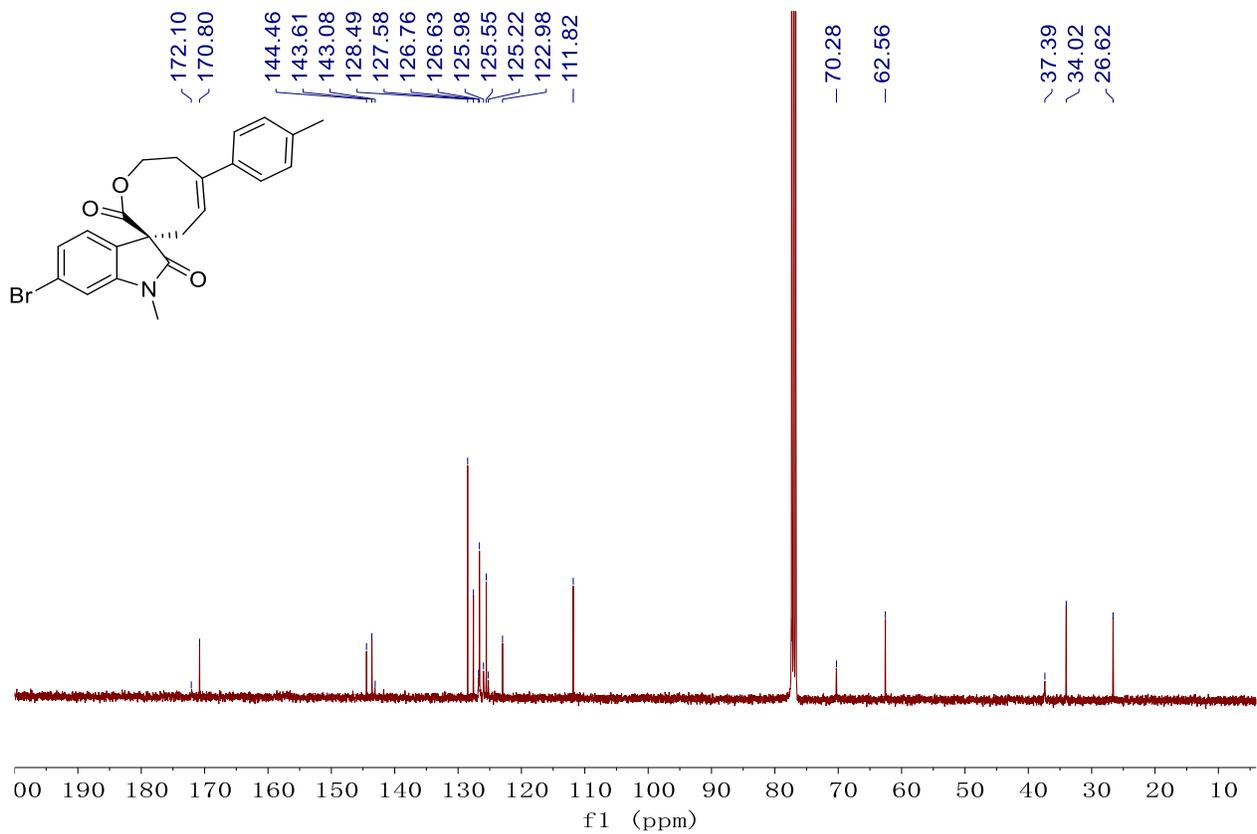


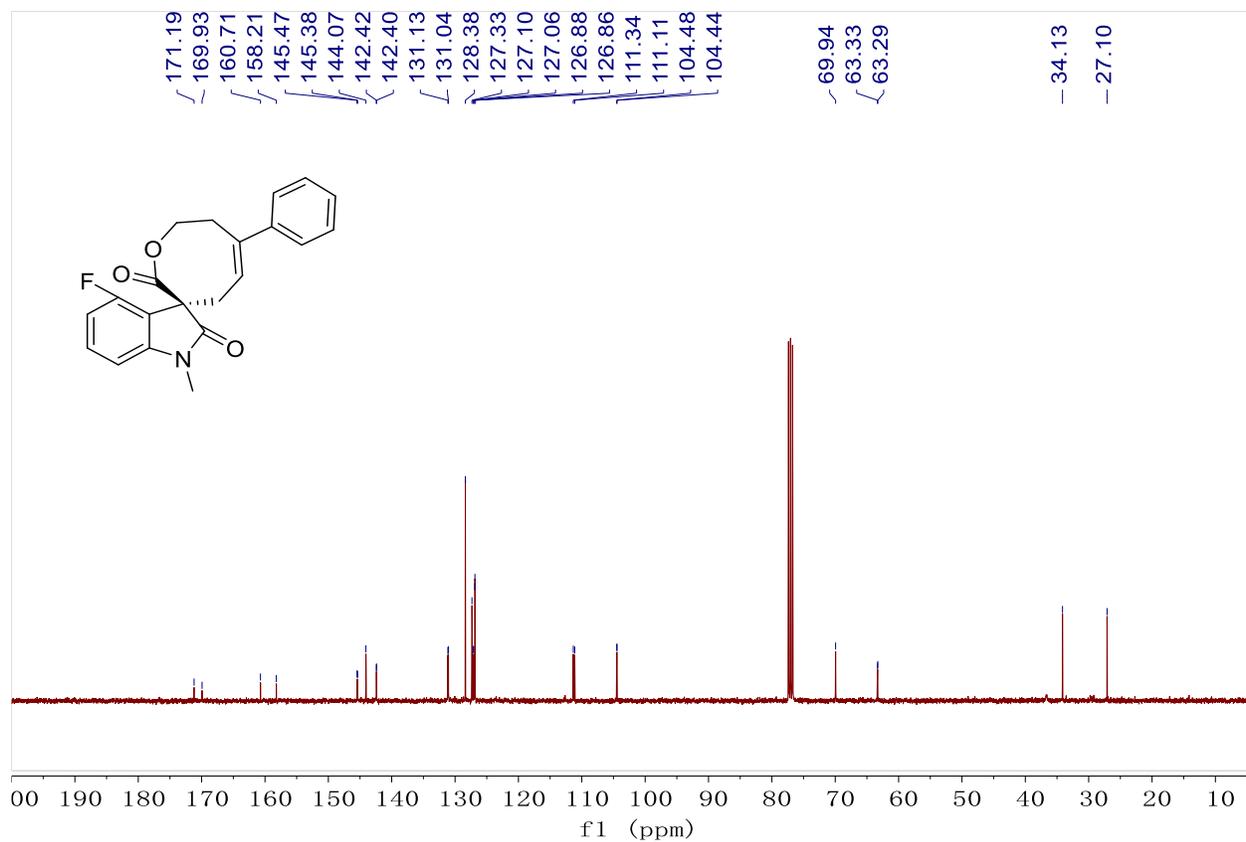
<sup>13</sup>C NMR spectrum of product **3t**



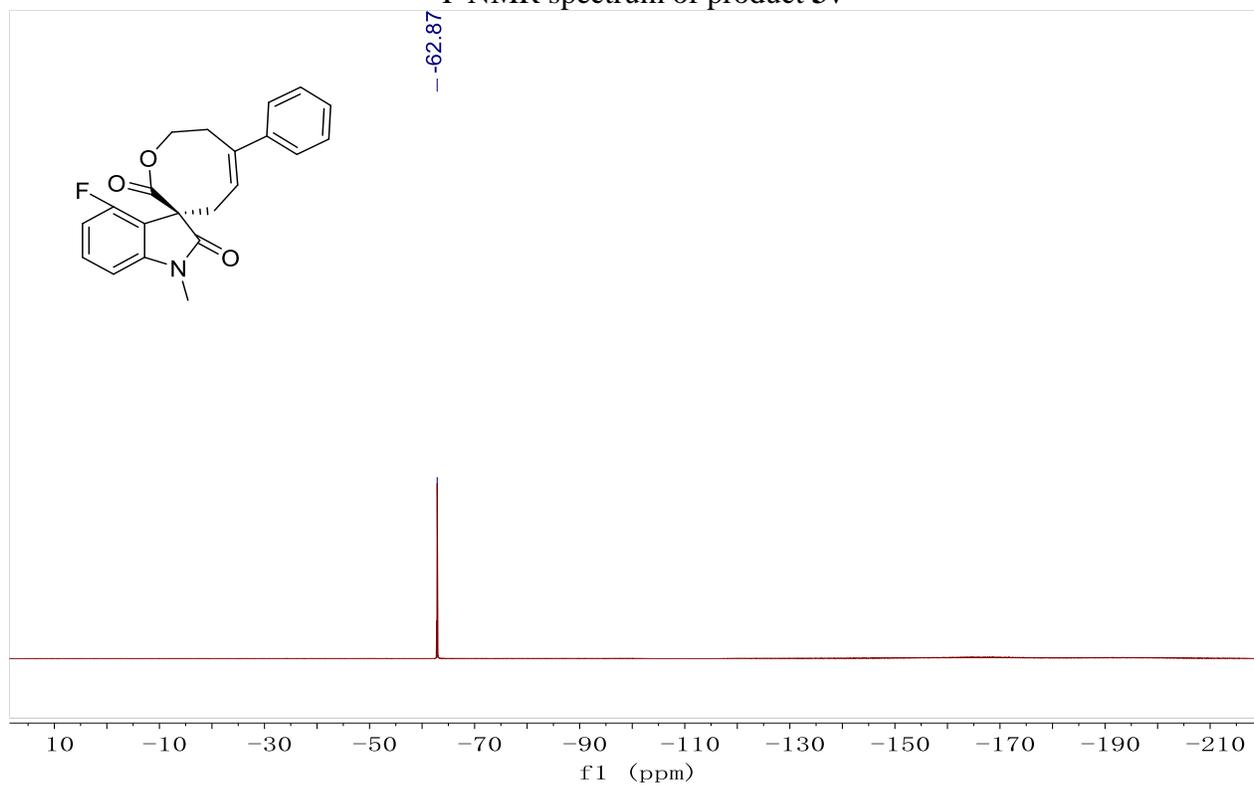


**<sup>13</sup>C NMR spectrum of product 3u**

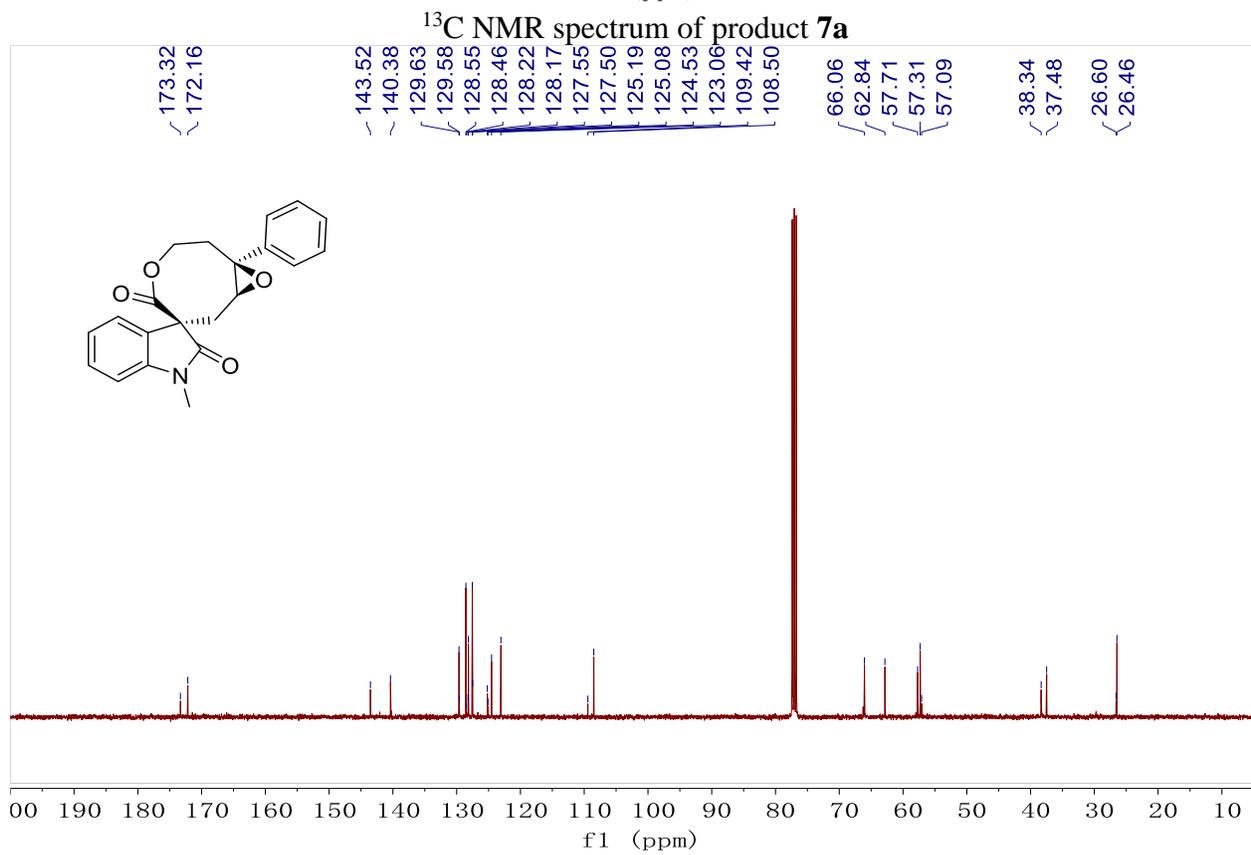
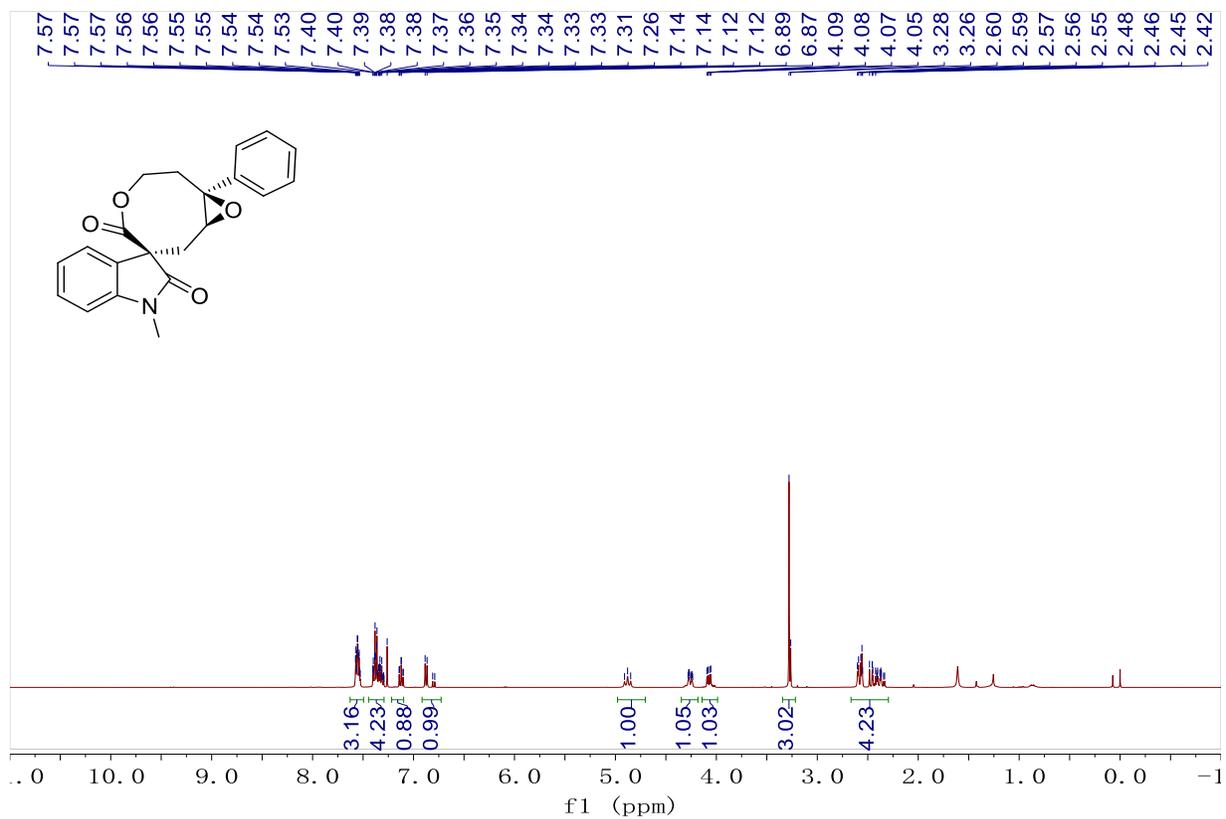




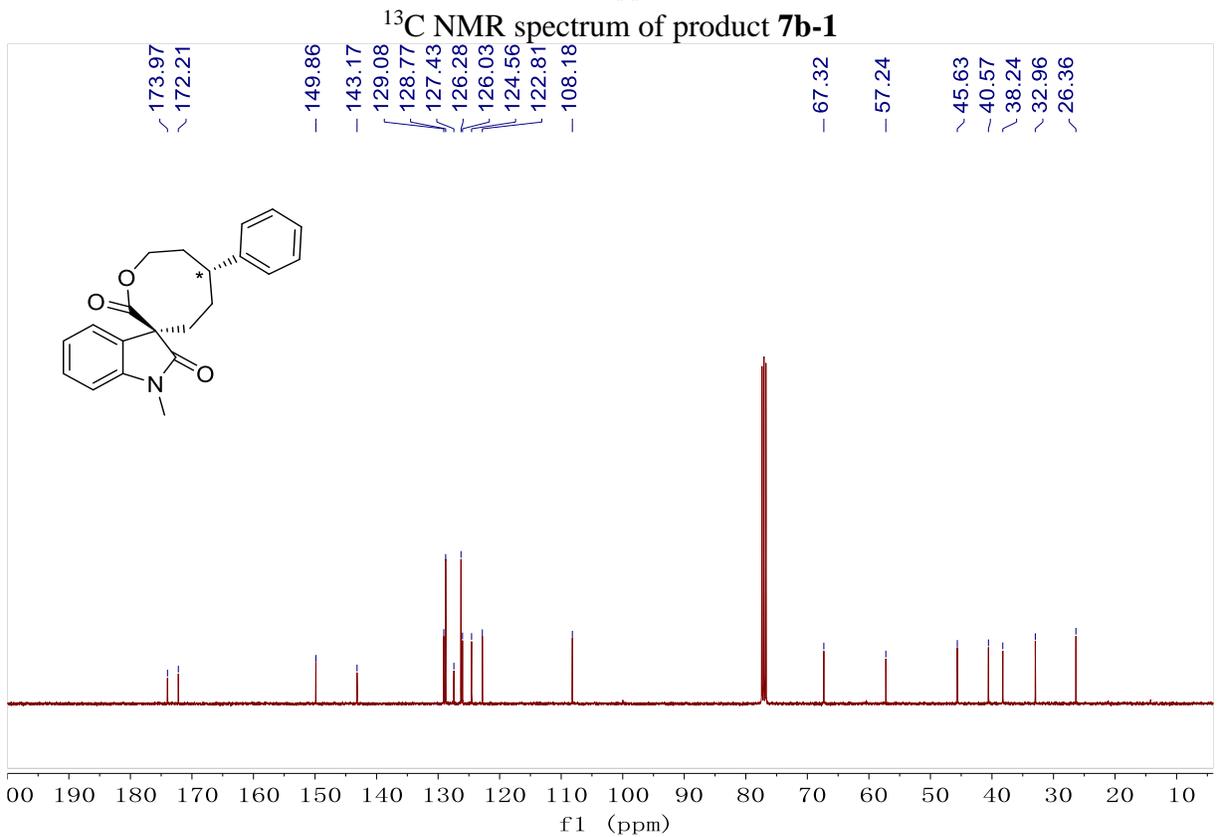
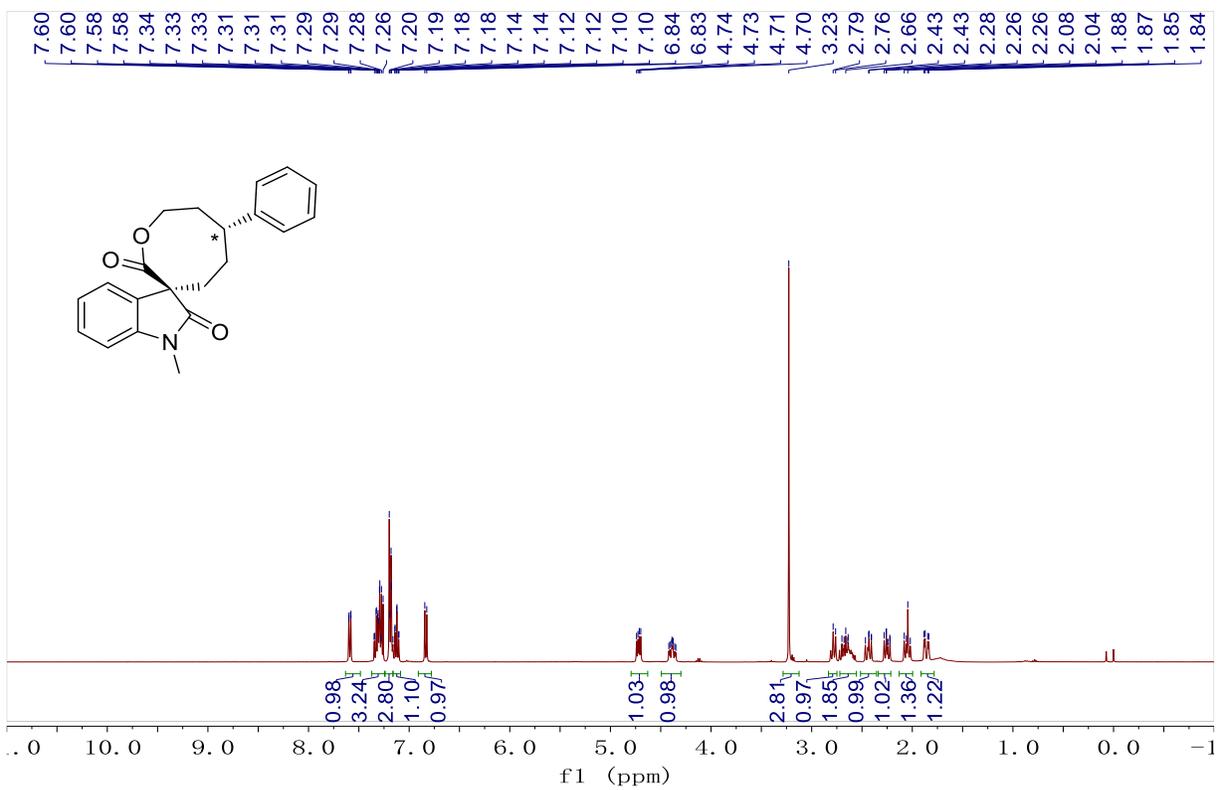
$^{19}\text{F}$  NMR spectrum of product **3v**



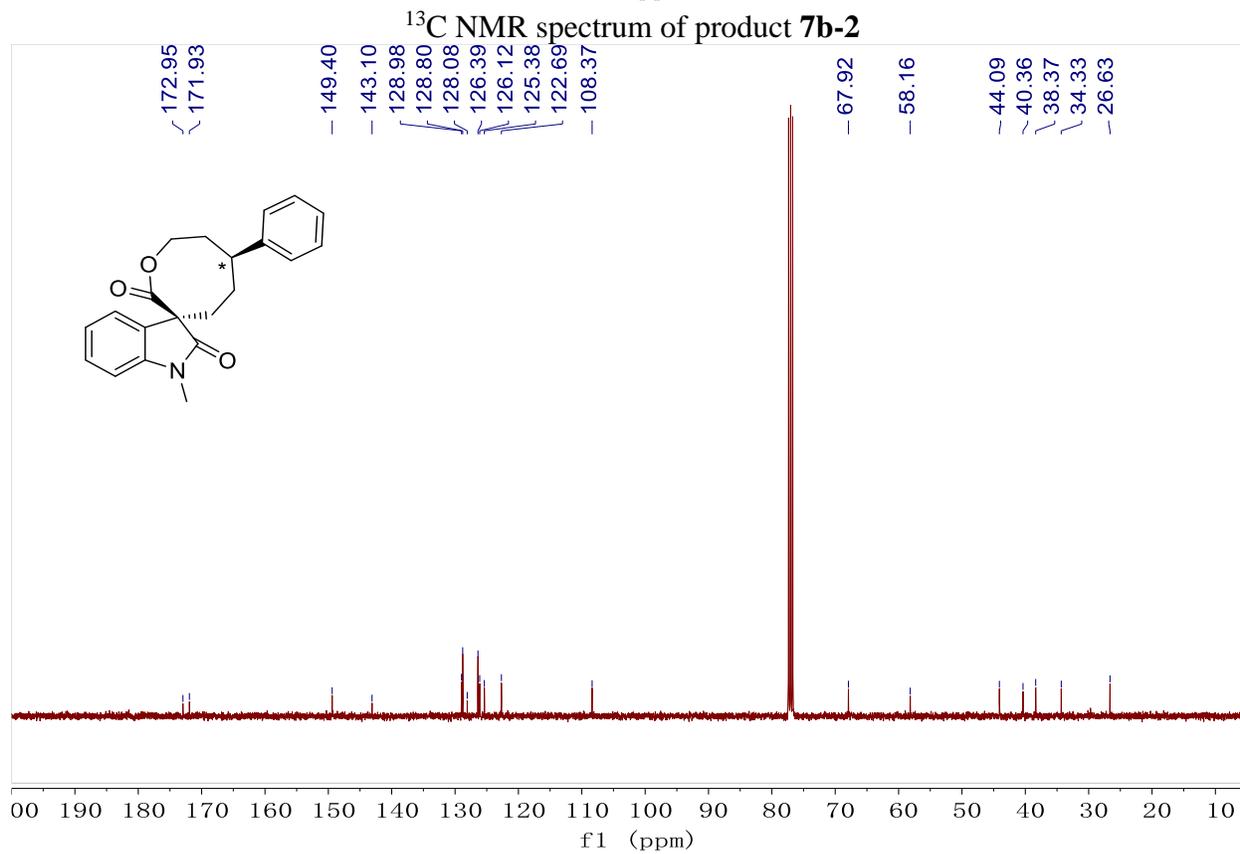
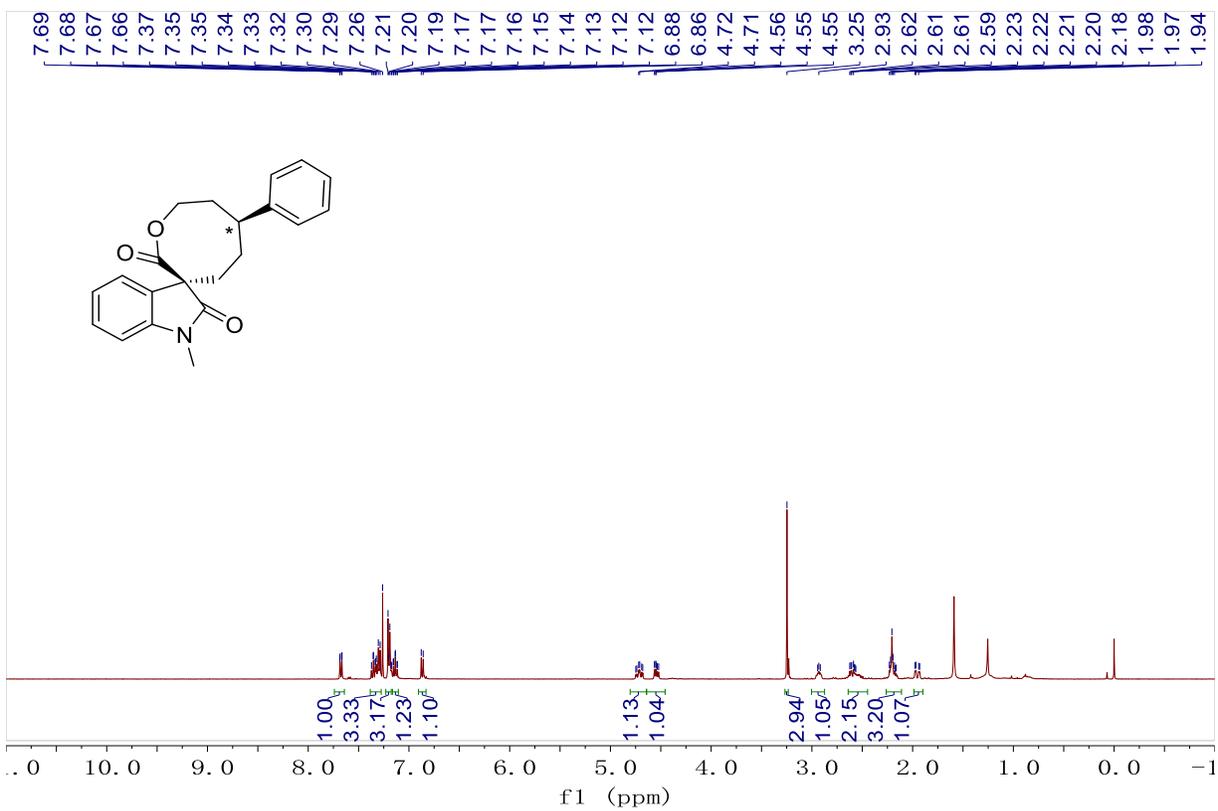
$^1\text{H}$  NMR spectrum of product **7a**



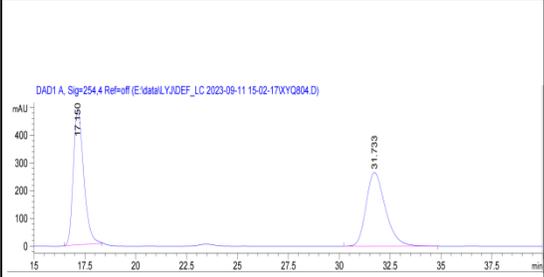
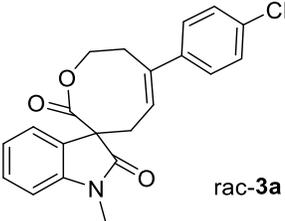
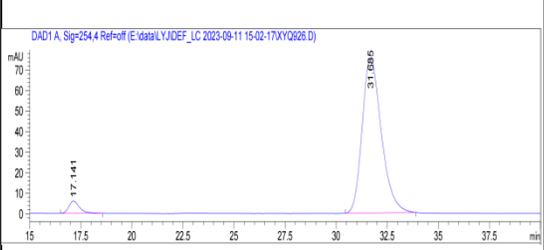
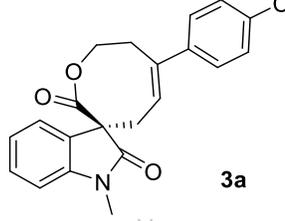
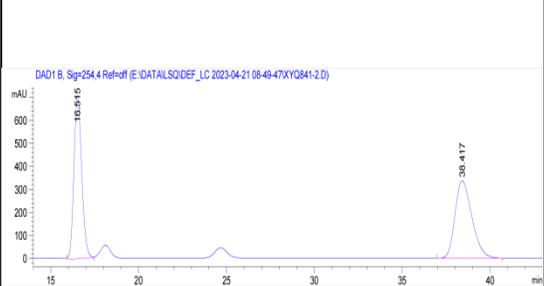
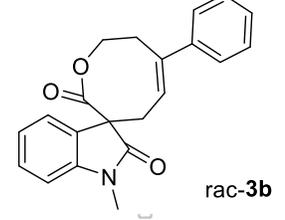
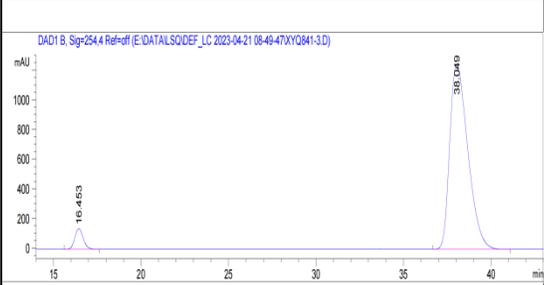
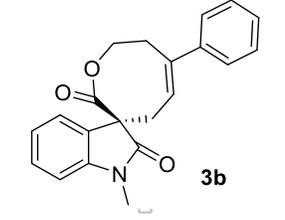
**<sup>1</sup>H NMR spectrum of product 7b-1**

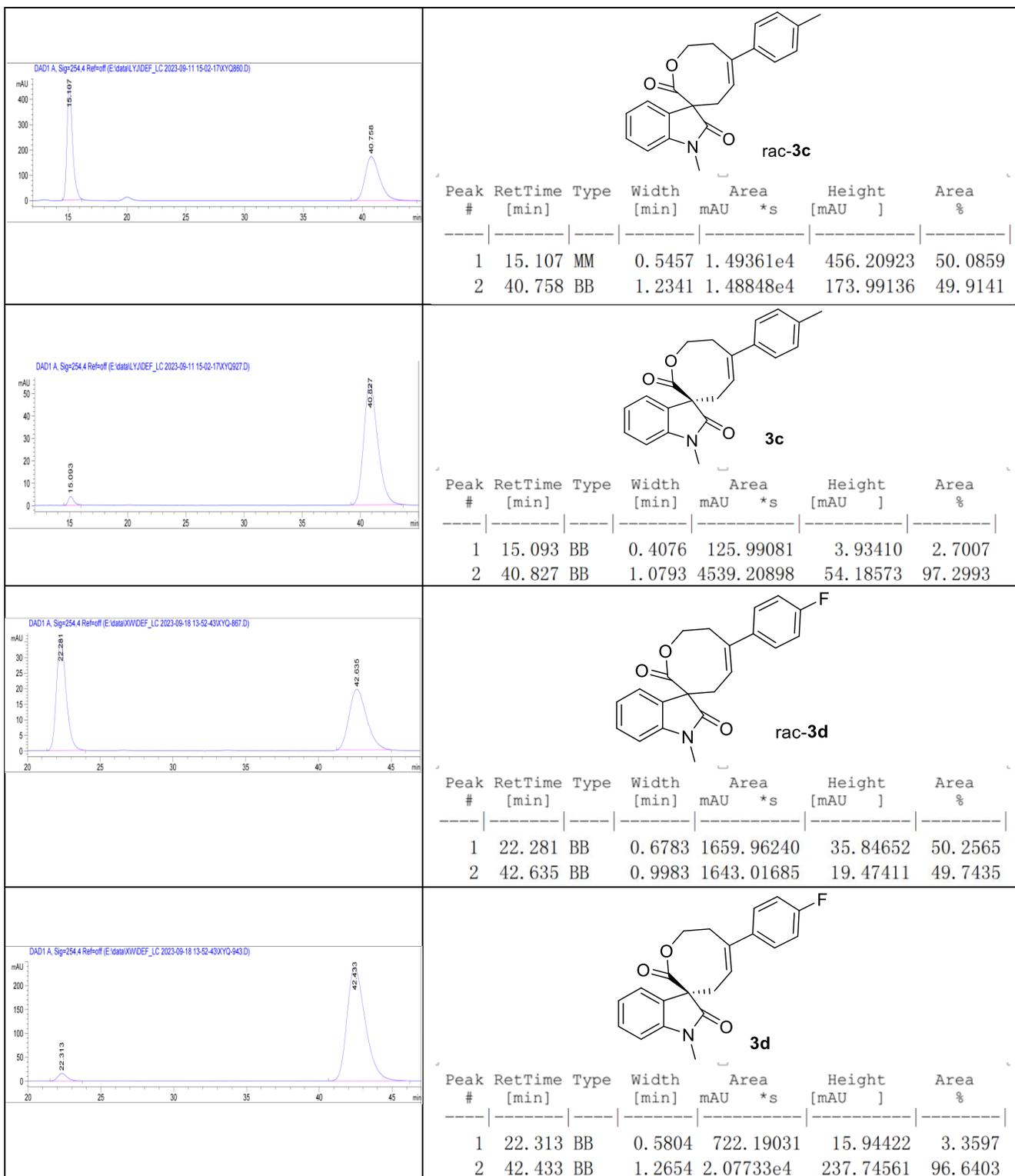


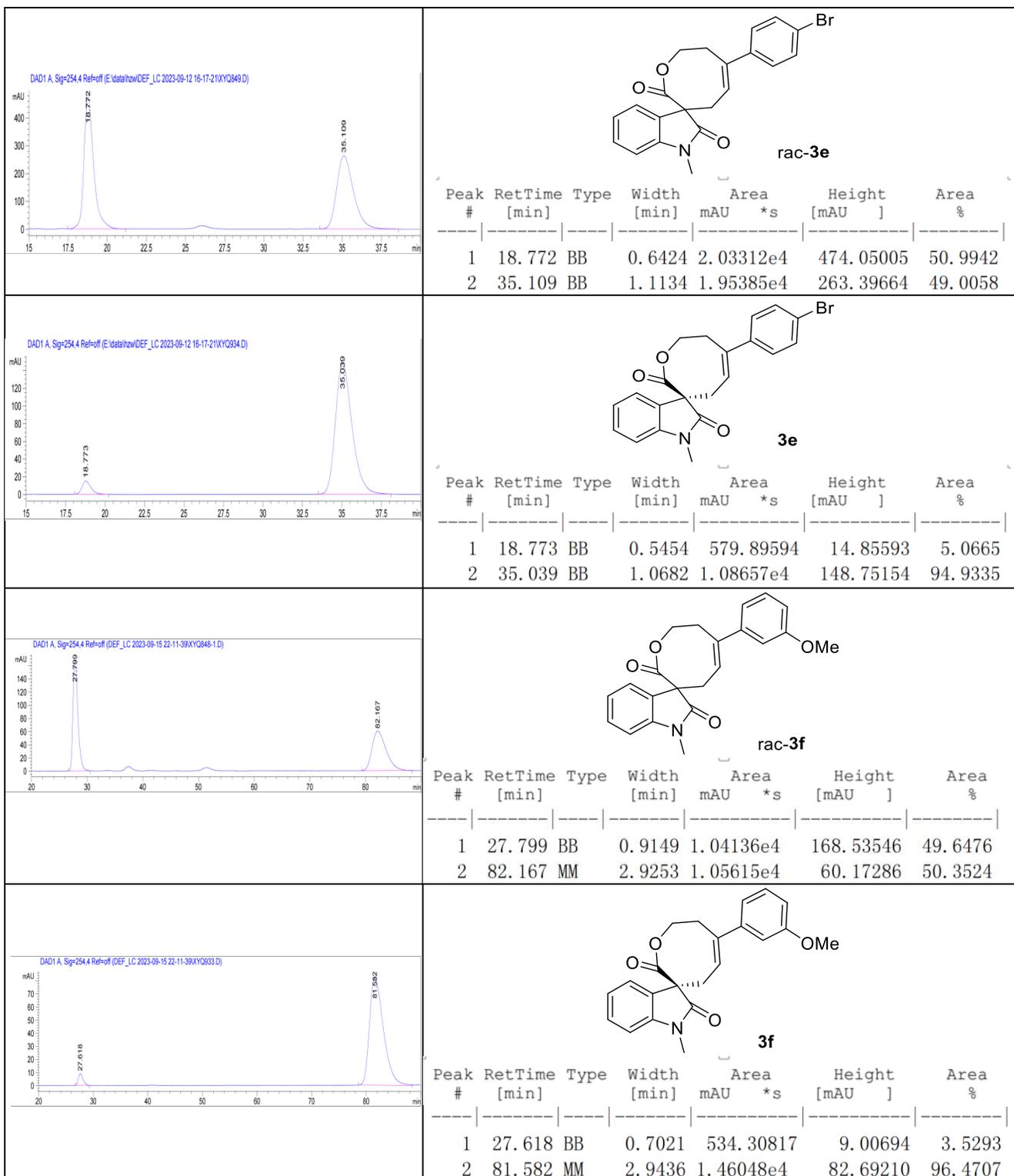
**<sup>1</sup>H NMR spectrum of product 7b-2**

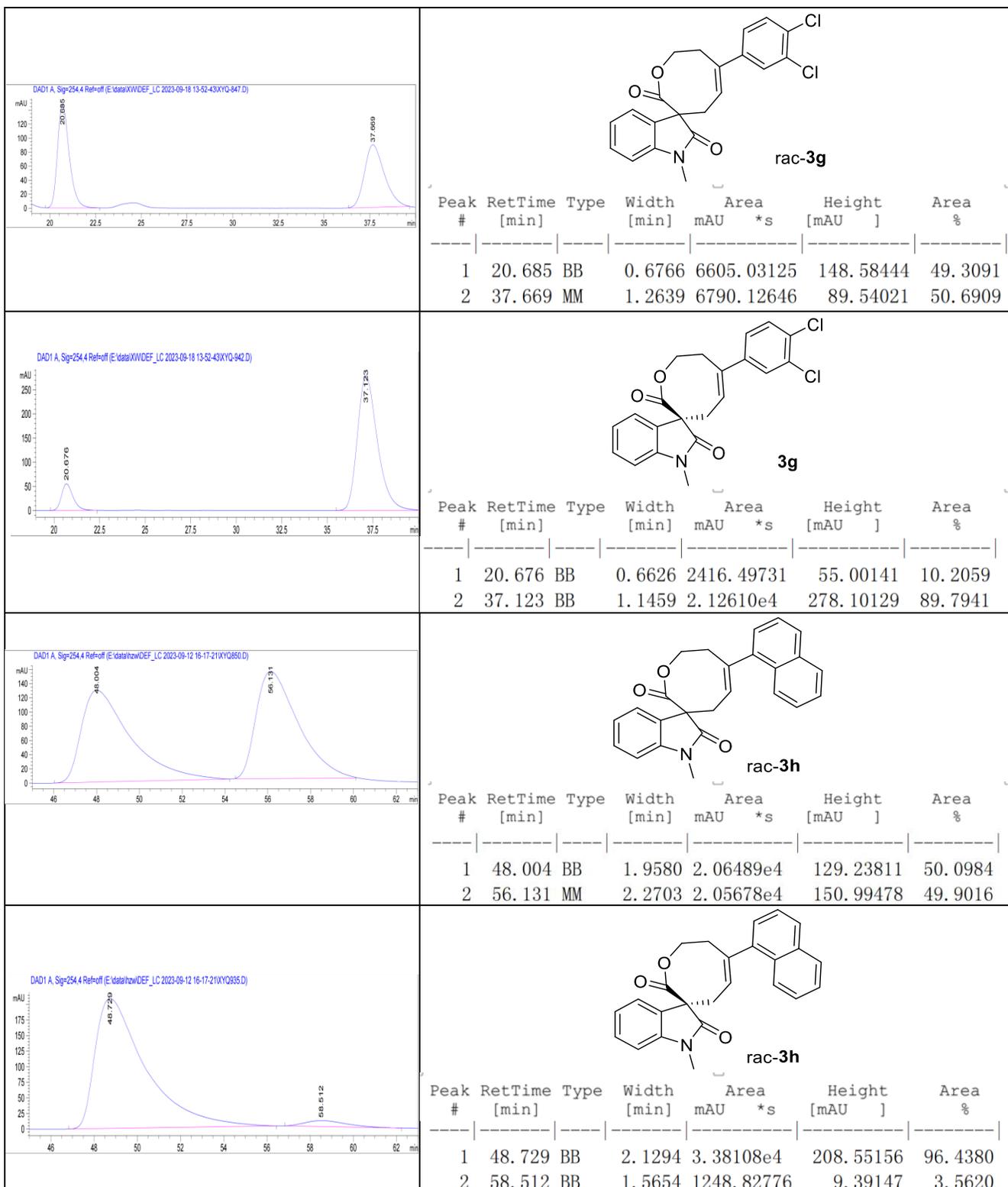


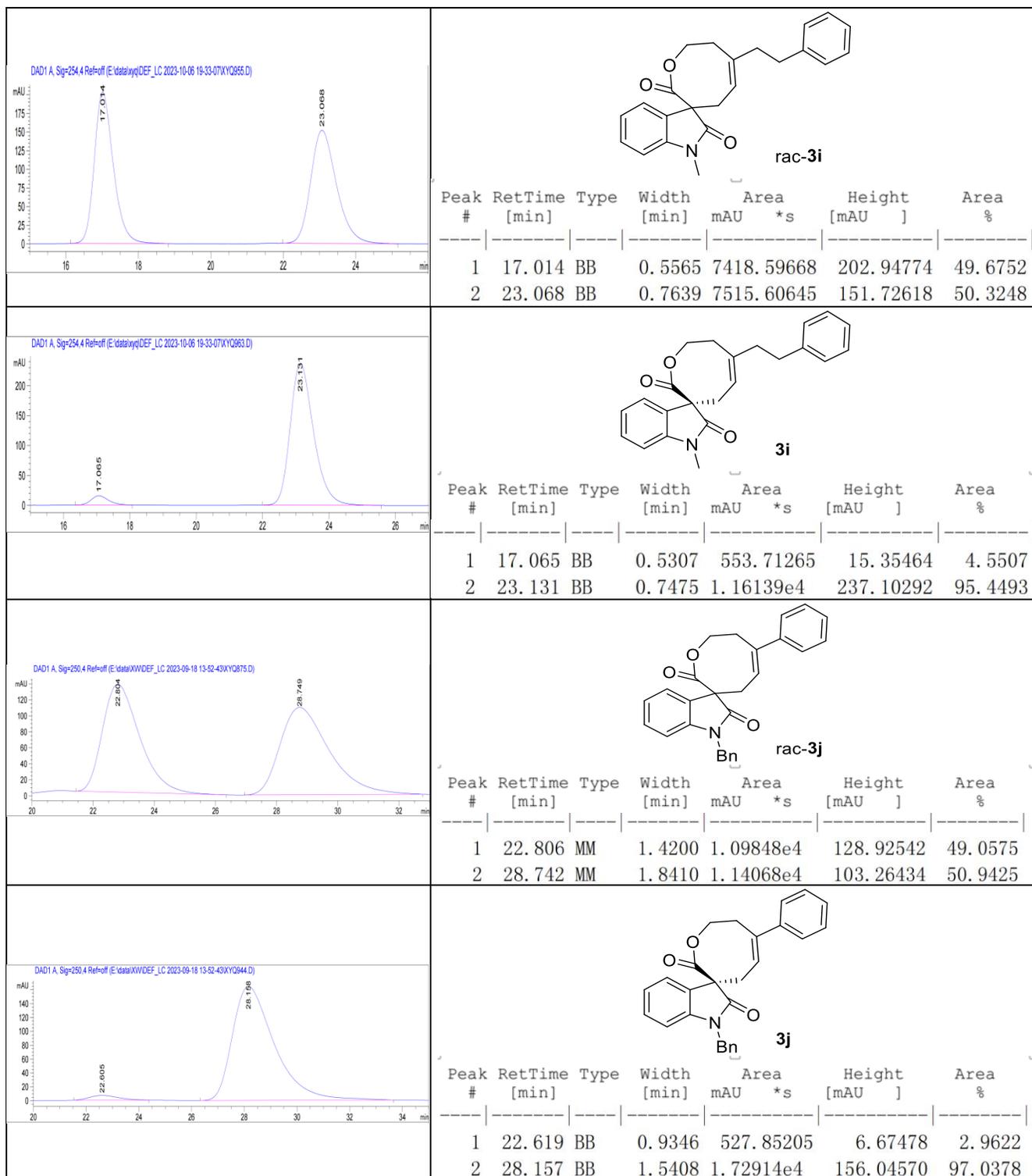
## 8. Copies of HPLC Data of Products

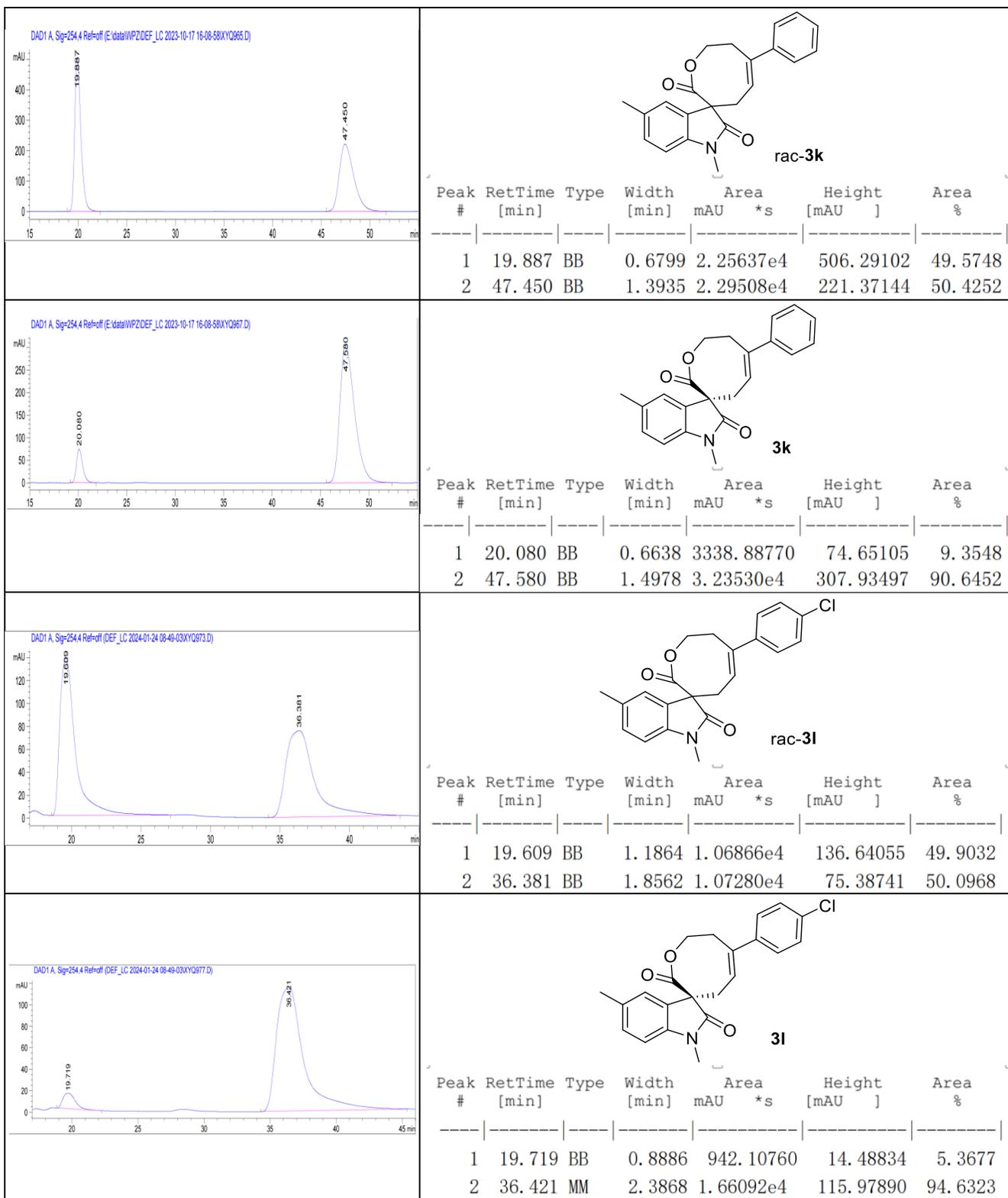
 <p>DAD1 A, Sig=254.4 Ref=off (E:\data\LYJDEF_LC 2023-09-11 15-02-17\XYQ804.D)</p>	<div style="text-align: center;">  <p><b>rac-3a</b></p> </div> <table border="1" data-bbox="722 472 1445 630"> <thead> <tr> <th>Peak #</th> <th>RetTime [min]</th> <th>Type</th> <th>Width [min]</th> <th>Area mAU *s</th> <th>Height [mAU]</th> <th>Area %</th> </tr> </thead> <tbody> <tr> <td>1</td> <td>17.150</td> <td>MM</td> <td>0.5912</td> <td>1.73189e4</td> <td>488.21744</td> <td>49.7438</td> </tr> <tr> <td>2</td> <td>31.733</td> <td>BB</td> <td>0.9975</td> <td>1.74973e4</td> <td>265.28867</td> <td>50.2562</td> </tr> </tbody> </table>	Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %	1	17.150	MM	0.5912	1.73189e4	488.21744	49.7438	2	31.733	BB	0.9975	1.74973e4	265.28867	50.2562
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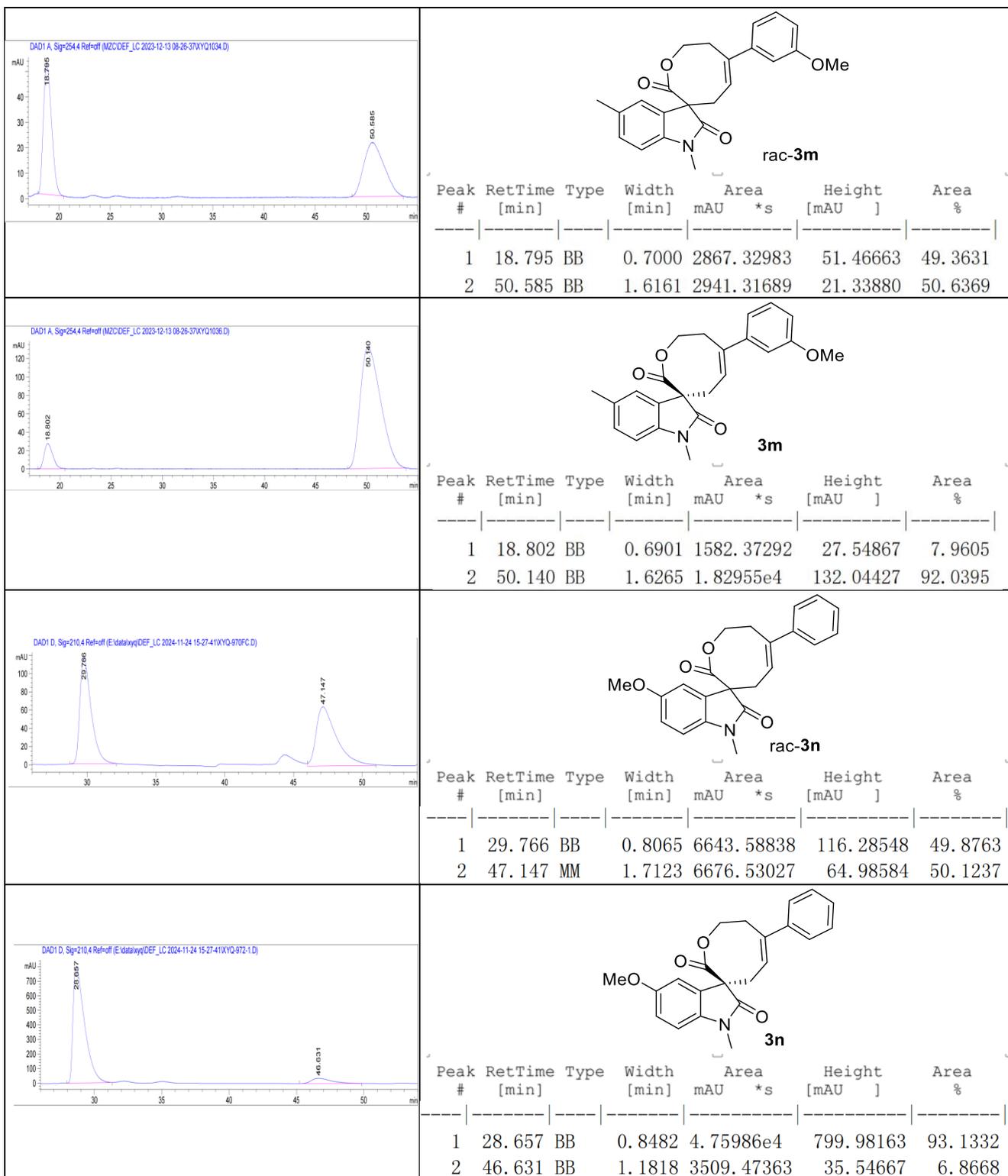


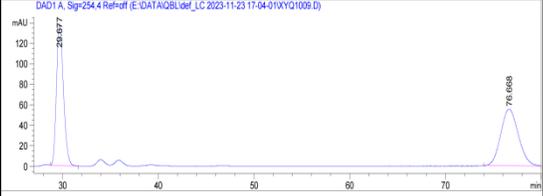
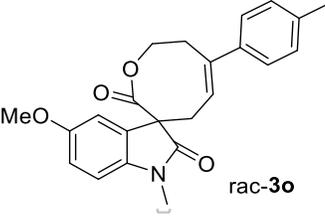
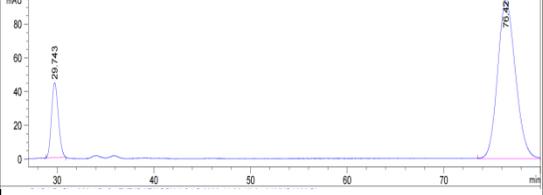
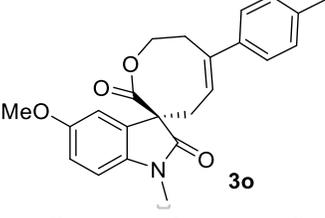
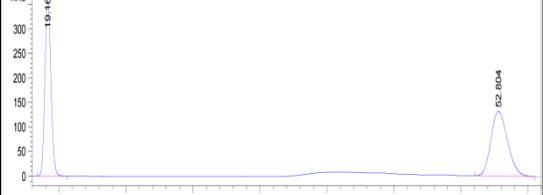
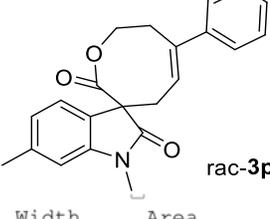
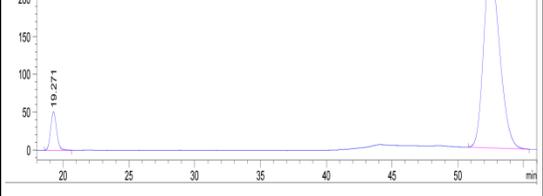
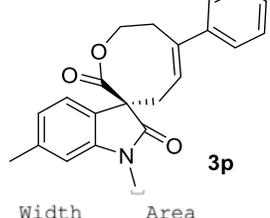


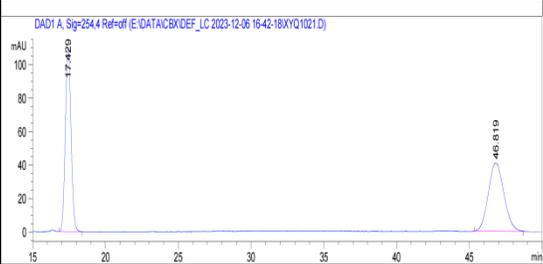
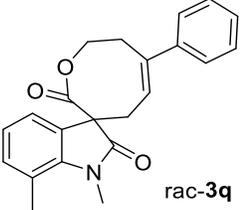
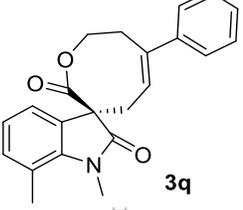
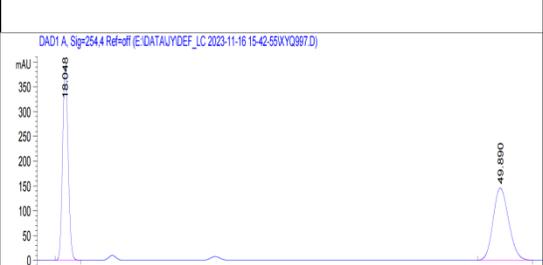
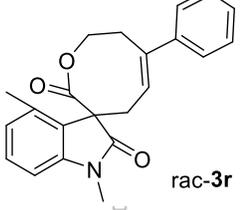
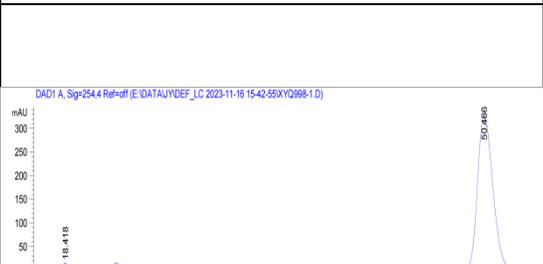
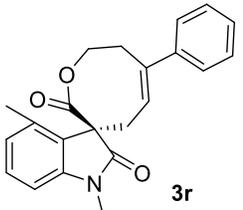


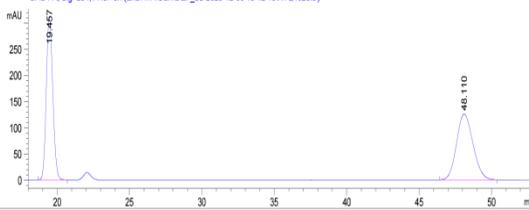
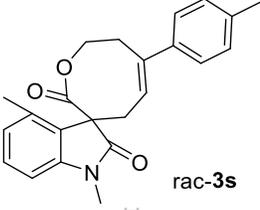
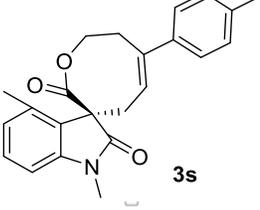
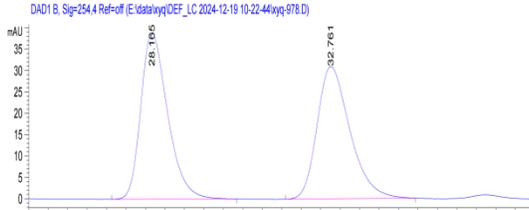
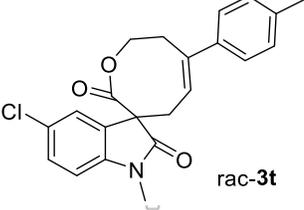
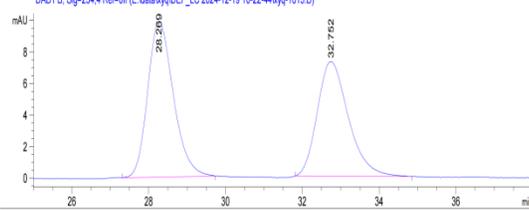
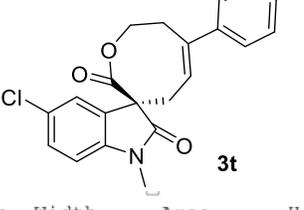


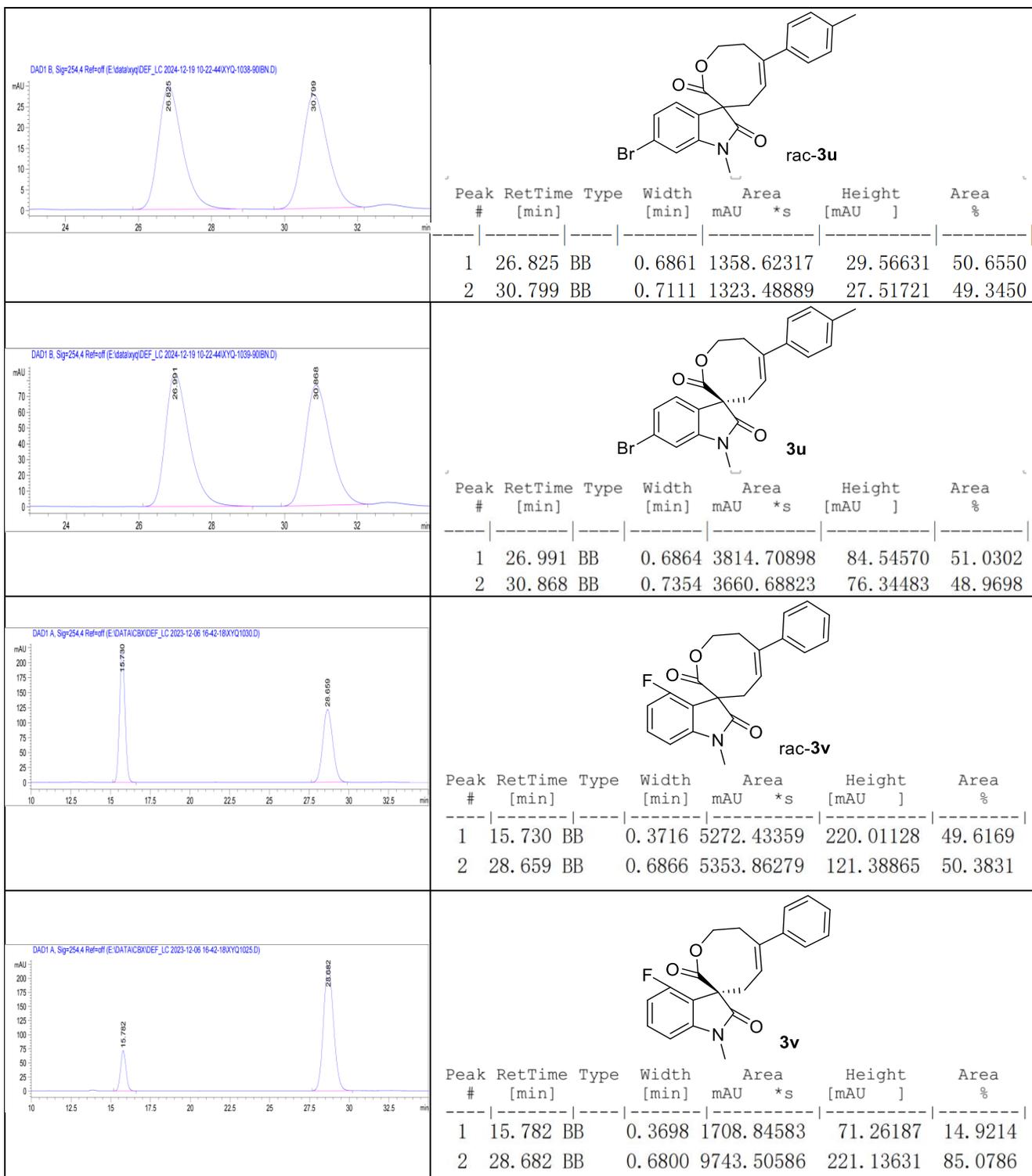


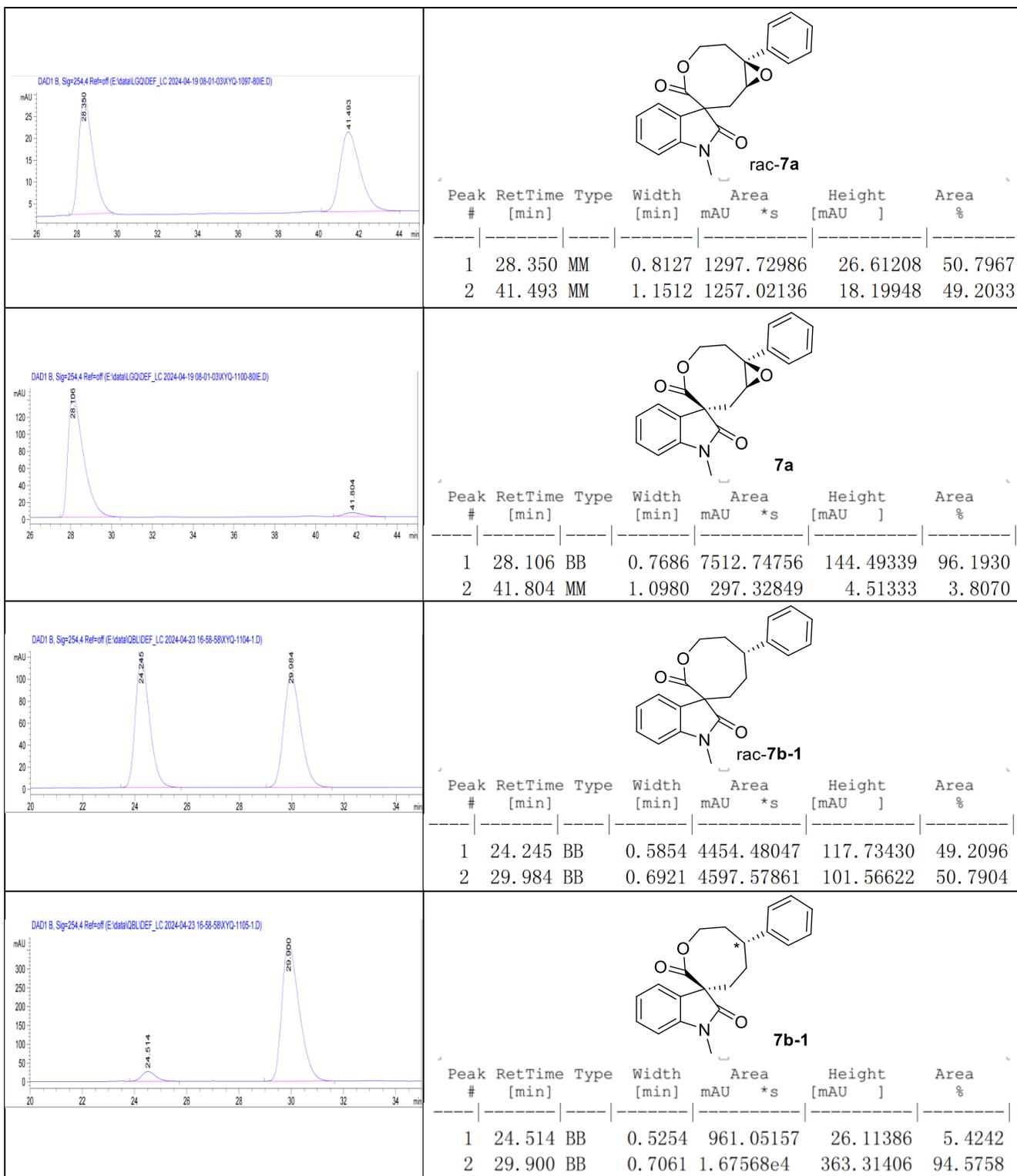


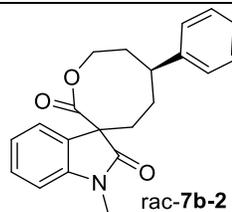
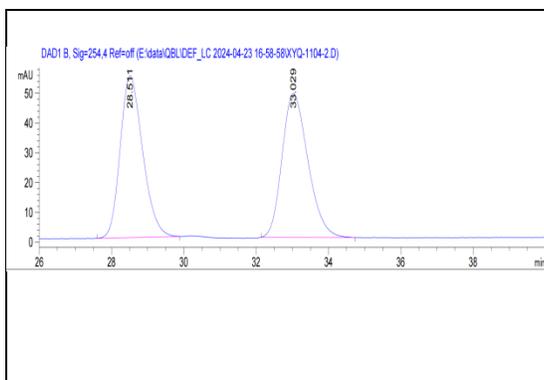
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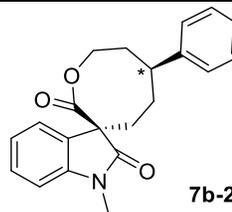
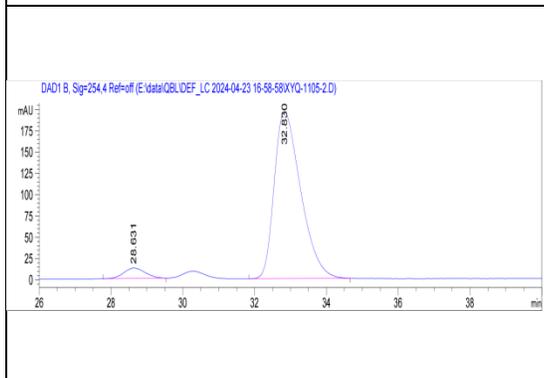
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2	33.029	BB	0.6984	2404.90479	48.34358	50.6046



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