

# Supporting Information

## Copper-Catalyzed Enantioselective Radical Sulfonylation from Sodium Hydrogen Sulfite: Construction of Chiral Sulfonated Dihydrofuran-2(3*H*)-ones

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## 1. Supplementary Notes

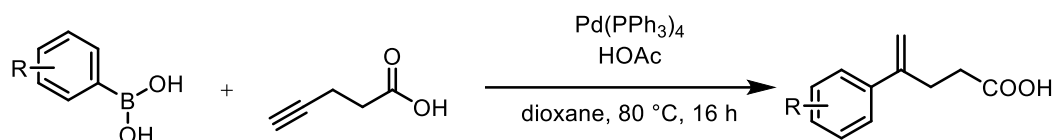
All glassware was thoroughly oven-dried. Chemicals and solvents were either purchased from commercial suppliers or purified by standard techniques. Thin-layer chromatography plates were visualized by exposure to ultraviolet light and/or I<sub>2</sub>. Flash chromatography was carried out using silica gel (200–300 mesh). Visualization on TLC was achieved by use of UV light (254 nm). <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on a Bruker AM-400 (400 MHz). The spectra were recorded in deuteriochloroform (CDCl<sub>3</sub>) as solvent at room temperature, <sup>1</sup>H and <sup>13</sup>C NMR chemical shifts are reported in ppm relative to the residual solvent peak. The residual solvent signals were used as references and the chemical shifts were converted to the TMS scale (CDCl<sub>3</sub>: δ<sub>H</sub> = 7.26 ppm, δ<sub>C</sub> = 77.0 ppm). Data for <sup>1</sup>H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, dd = doublet, br = broad), integration, coupling constant (Hz) and assignment. Data for <sup>13</sup>C NMR are reported as chemical shift. Electrospray-ionisation HRMS data were acquired on a Q-TOF mass spectrometer (Waters SYNAPT G2-Si) LC-MS TOF. The enantiomeric excess values were determined by chiral HPLC with a Shimadzu instrument and a Daicel CHIRALCEL and CHIRALPAK column.

## 2. Substrates involved in the reactions and their synthesis

### 2.1 General procedure for the preparation of enoic acid.

Enoic acid **1** are known compounds, which were prepared according to literatures. <sup>[1-2]</sup>

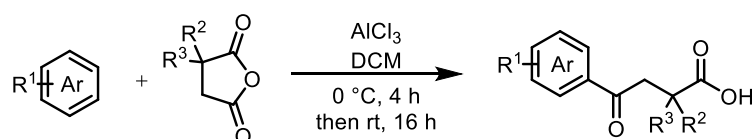
Method A<sup>[1]</sup>:



An oven dried round-bottom-flask was charged with arylboronic acid (2.49 g, 20.4 mmol, 1.0 equiv), Pd(PPh<sub>3</sub>)<sub>4</sub> (1.178 g, 1.02 mmol, 0.05 equiv) and dry dioxane (100 mL). Next 4-pentynoic acid (2.31 mL,  $\rho = 0.99$  g/mL, 20.4 mmol, 1.00 equiv) and acetic acid (150  $\mu$ L,  $\rho = 1.05$  g mL<sup>-1</sup>, 2.04 mmol, 0.10 equiv) were added and the mixture was heated to 90°C overnight (ca. 16 h). The solvent was removed in vacuum and the crude product was filtered by silica gel column (eluent: CH<sub>2</sub>Cl<sub>2</sub>) to remove the catalyst and boronic acid residues. The resulting product mixture was then separated by preparative HPLC (Lux Amylose, 20 mL/min, H<sub>2</sub>O/CH<sub>3</sub>CN, 75:25, 45 min,  $R_t = 33$  min product) and afforded the pure product. Compounds **1a** and **1ae** are characterized in the reference.<sup>[1]</sup>

Method B: <sup>[2]</sup>

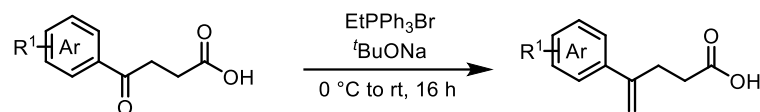
Step I. General procedure for synthesis of ketone precursors using Friedel-Craft reaction



A 100 mL, three-necked, round-bottomed flask is charged with powdered dihydrofuran-2,5-dione (1.0 equiv) and arene (1.0 equiv) under dry nitrogen. The resulting white mixture was cooled to 0 °C before anhydrous aluminum trichloride (1.2 equiv) was added in one portion. The reaction mixture was stirred over a period of 4 h before allowing it to warm to room temperature for 16 h. The reaction was poured in ice and 10 mL of concentrated hydrochloric acid was added under stirring at 0 °C. The organic layer was separated and the aqueous layer was extracted with DCM twice. The combined organic layers were washed with water, dried over MgSO<sub>4</sub> and concentrated.

The product was engaged in the next step without further purification.

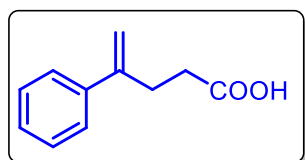
Step II. General procedure for synthesis of enoic acid using Wittig reaction.



Under nitrogen, to a solution of <sup>t</sup>BuONa (2.6 equiv) in dry THF (0.5 M) was added bromo(ethyl)triphenylphosphorane (1.3 equiv) in portions at 0 °C. The mixture was stirred at 0 °C for 30 min and a solution of ketone (1.0 equiv) in dry THF (1 M) was added dropwise and the reaction was stirred at 0 °C for 1 h and at rt overnight. The solvent was removed in vacuo and the residue diluted with DCM and aqueous NaOH (1 M). The aqueous layer was separated, washed with dichloromethane, and acidified to pH 1 with concentrated HCl. DCM was added and the organic compound was extracted twice with DCM. The organic layer was washed with water, dried over MgSO<sub>4</sub> and concentrated. The crude product was purified by SiO<sub>2</sub> column chromatography to give pure enoic acid. Compounds **1a**<sup>[2a]</sup>, **1b**<sup>[2a]</sup>, **1c**<sup>[2a]</sup>, **1d**<sup>[2a]</sup>, **1e**<sup>[2a]</sup>, **1f**<sup>[2b]</sup>, **1g**<sup>[2a]</sup>, **1h**<sup>[2a]</sup>, **1i**<sup>[2c]</sup>, **1j**<sup>[2a]</sup>, **1k**<sup>[2d]</sup>, **1l**<sup>[2d]</sup>, **1m**<sup>[2c]</sup>, **1n**<sup>[2a]</sup>, **1o**<sup>[2d]</sup> and **1p**<sup>[2f]</sup> are characterized in references.

### Characterization of representative enoic acid

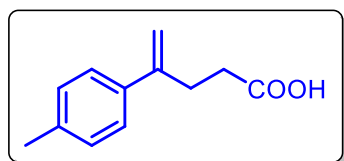
#### 4-phenylpent-4-enoic acid (**1a**)



Purification by flash chromatography (PE/EA = 4/1). White solid, 88% yield.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 11.43 (s, 1H), 7.39 (d, *J* = 7.1 Hz, 2H), 7.35 – 7.23 (m, 3H), 5.32 (s, 1H), 5.10 (s, 1H), 2.89 – 2.80 (m, 2H), 2.56 – 2.49 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 179.7, 146.6, 140.5, 128.5, 127.7, 126.1, 113.0, 33.0, 30.2.

#### 4-(p-tolyl)pent-4-enoic acid (**1c**)

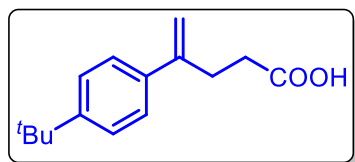


Purification by flash chromatography (PE/EA = 4/1). White solid, 76% yield.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 11.35 (s, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 5.29 (s, 1H), 5.06 (d, *J* = 0.6 Hz, 1H), 2.86 – 2.79 (m, 2H), 2.55 – 2.49 (m, 2H), 2.34 (s, 3H).

**<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 179.8, 146.3, 137.5, 137.5, 129.2, 126.0, 112.2, 33.1, 30.2, 21.1.

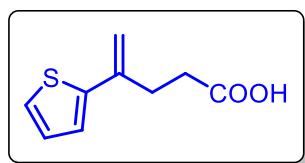
#### 4-(4-(*tert*-butyl)phenyl)pent-4-enoic acid (1d)



Purification by flash chromatography (PE/EA = 4/1). White solid, 74% yield.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 7.35 (s, 4H), 5.32 (s, 1H), 5.07 (d, *J* = 1.2 Hz, 1H), 2.89 – 2.79 (m, 2H), 2.56 – 2.52 (m, 2H), 1.32 (s, 9H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 179.2, 150.7, 146.2, 137.3, 125.7, 125.4, 112.3, 34.5, 33.0, 31.3, 30.1. HRMS (ESI) for C<sub>15</sub>H<sub>21</sub>O<sub>2</sub><sup>+</sup> [M+H]<sup>+</sup> calcd. 233.1536, found: 233.1538.

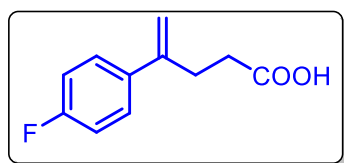
#### 4-(thiophen-2-yl)pent-4-enoic acid (1o)



Purification by flash chromatography (PE/EA = 4/1). Yellow solid, 75% yield.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 7.28 (dd, *J* = 5.0, 2.9 Hz, 1H), 7.25 – 7.21 (m, 2H), 5.40 (s, 1H), 5.06 (d, *J* = 0.6 Hz, 1H), 2.83 – 2.78 (m, 2H), 2.64 – 2.60 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 179.5, 141.8, 140.9, 125.8, 125.7, 120.5, 111.6, 33.0, 30.0.

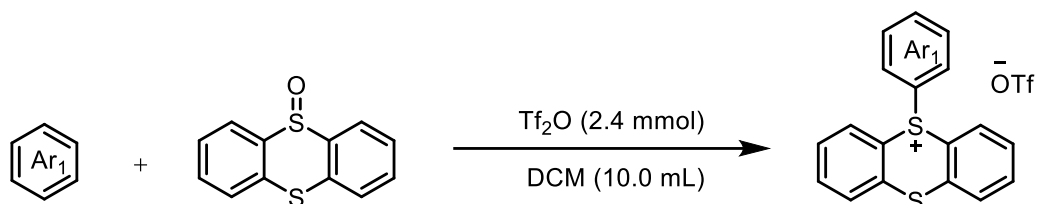
#### 4-(4-fluorophenyl)pent-4-enoic acid (1n)



Purification by flash chromatography (PE/EA = 4/1). White solid, 71% yield.

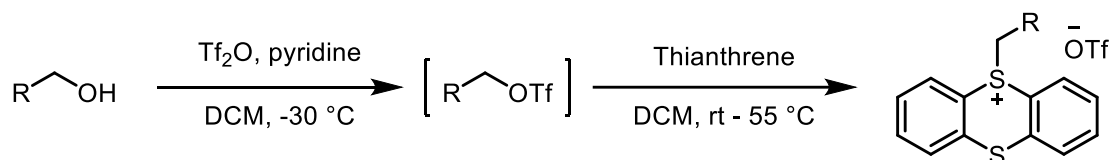
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 11.76 (s, 1H), 7.39 – 7.32 (m, 2H), 7.01 (t, *J* = 8.7 Hz, 2H), 5.26 (s, 1H), 5.08 (s, 1H), 2.83 – 2.79 (m, 2H), 2.55 – 2.48 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 179.8, 162.5 (d, *J* = 245.2 Hz), 145.6, 136.52 (d, *J* = 3.2 Hz), 127.74 (d, *J* = 7.9 Hz), 115.3 (d, *J* = 21.4 Hz), 113.0, 33.0, 30.2. **<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)** δ (ppm) = –114.68 – –114.76 (m).

## 2.2 Preparation of aryl thianthrenium salts<sup>[3]</sup>



A 25 mL schlenk tube was charged with thianthrene S-oxide (2.0 mmol, 1.0 equiv), CH<sub>2</sub>Cl<sub>2</sub> (5.0 mL) and arene (2.0 mmol, 1.0 equiv) under a nitrogen atmosphere. The reaction mixture was then cooled to -40 °C and stirred at this temperature. Tf<sub>2</sub>O (2.4 mmol, 1.2 equiv) was added dropwisely. The reaction mixture was stirred at -40 °C for 30 min, and then allowed to stir at room temperature for 12 h, neutralized by a saturated aqueous NaHCO<sub>3</sub> solution, and extracted with CH<sub>2</sub>Cl<sub>2</sub>. The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated to dryness under reduced pressure. The crude product was purified by crystallization from CH<sub>2</sub>Cl<sub>2</sub> / Et<sub>2</sub>O system as a white solid. Compounds **2a**<sup>[3a]</sup>, **2q**<sup>[3b]</sup>, **2r**<sup>[3b]</sup>, **2s**<sup>[3a]</sup>, **2t**<sup>[3a]</sup>, **2u**<sup>[3a]</sup>, **2v**<sup>[3a]</sup>, **2w**<sup>[3a]</sup>, **2x**<sup>[3a]</sup>, **2y**<sup>[3a]</sup>, **2z**<sup>[3a]</sup> and **2aa**<sup>[3c]</sup> are characted in references

## 2.3 Preparation of alkyl thianthrenium salts<sup>[4]</sup>

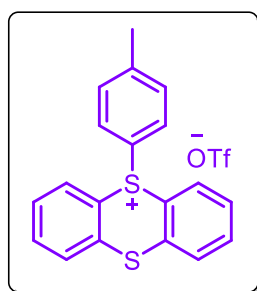


A flame-dried 100 mL flask was placed under an atmosphere of nitrogen and charged with a stir bar and alcohol (5.0 mmol, 1.0 equiv). The alcohol was dissolved in CH<sub>2</sub>Cl<sub>2</sub> (20.0 mL) and cooled to -30 °C before adding pyridine (483 μL, 6.0 mmol, 1.2 equiv). While stirring, triflic anhydride (1.0 mL, 29.3 mmol, 1.20 equiv) was added dropwisely, and then the reaction mixture was stirred for 3 h while remaining at -5 °C. While the flask was still in a -5 °C bath, 0.5 M H<sub>2</sub>SO<sub>4</sub> (30 mL) was added. The flask was removed from the cold bath, and the mixture was transferred to a separatory funnel and extracted with 3×20 mL of CH<sub>2</sub>Cl<sub>2</sub>. The organic layers were combined and washed 1× 50 mL of distilled water. The collected organic layers were then dried over MgSO<sub>4</sub>, then filtered and concentrated to a 10 mL liquid under vacuum (without heating), which was used directly in the next step.

Flame-dried 25 mL Schlenk tube was added thianthrene (1.08g, 5.0 mmol), then the above liquid was added. The mixture was stirred at 55 °C for 24 h. The mixture was carefully condensed under reduced pressure at 25 °C and purified by precipitation with Et<sub>2</sub>O/CH<sub>2</sub>Cl<sub>2</sub>. Most of the unreacted dibenzothiophene was removed by repeating the precipitation procedure 2 or 3 times. If the salt still did not precipitate, it was subjected to silica gel chromatography with acetone/CH<sub>2</sub>Cl<sub>2</sub>. Compounds **2ab**<sup>[4]</sup>, **2ac**<sup>[4]</sup> and **2ad**<sup>[4]</sup> are characterized in references.

## Characterization of representative thianthrenium salts

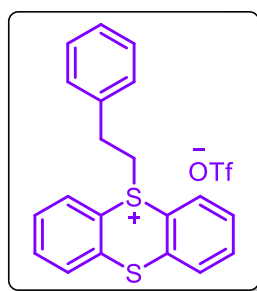
### 5-(p-tolyl)-5H-thianthren-5-ium triflate (**2a**)



Purification by flash chromatography (DCM/MeOH = 10/1). White solid, 95% yield.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 8.51 (d, *J* = 7.9 Hz, 2H), 7.92 – 7.83 (m, 4H), 7.81 – 7.73 (m, 2H), 7.25 (d, *J* = 8.3 Hz, 2H), 7.07 (d, *J* = 8.3 Hz, 2H), 2.33 (s, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 144.4, 136.5, 135.1, 134.8, 131.5, 130.5, 130.2, 127.9, 120.9 (q, *J* = 319.0 Hz), 120.1, 118.5, 21.3. **<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)** δ (ppm) = –77.98.

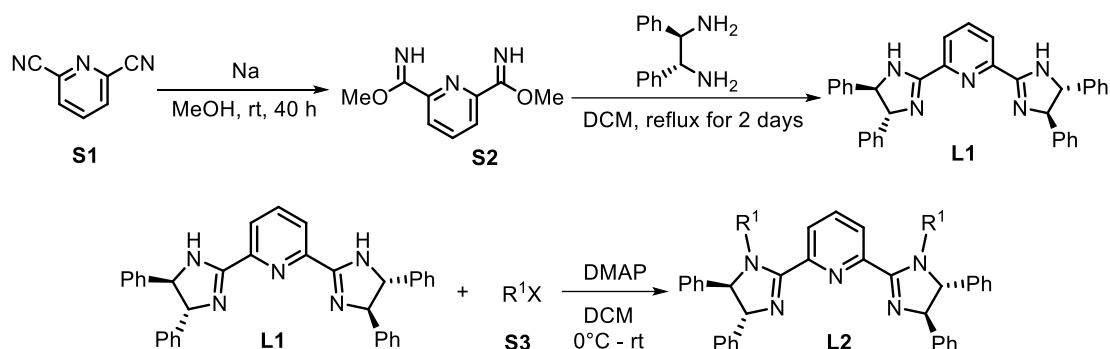
### 5-phenethyl-5H-thianthren-5-ium triflate (**2ab**)



Purification by flash chromatography (DCM/MeOH = 10/1). White solid, 90% yield.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 8.13 (d, *J* = 7.9 Hz, 2H), 7.88 – 7.80 (m, 2H), 7.78 – 7.71 (m, 2H), 7.64 – 7.56 (m, 2H), 7.25 – 7.15 (m, 3H), 7.12 (dd, *J* = 8.2, 6.3 Hz, 2H), 4.09 – 3.97 (m, 2H), 2.99 (t, *J* = 7.7 Hz, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 135.8, 135.1, 134.6, 134.4, 130.3, 129.9, 129.0, 128.7, 127.7, 120.9 (q, *J* = 318.7 Hz), 117.3, 41.7, 30.6. **<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)** δ (ppm) = –77.93.

## 2.4 General procedure for the synthesis of Pybim ligands<sup>[5]</sup>



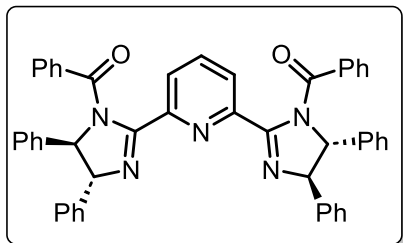
To pyridine-2,6-carbodinitrile **S1** (41.5 mmol) in anhydrous MeOH (100 mL), Na (5.2 mmol) was added. After stirring for 40 h at room temperature, acetic acid (5.25 mmol) was added and the solvent was removed under reduced pressure. Compound **S2** was obtained as a yellow powder (100 %) and was used directly for further reaction.

A 100 mL pressure tube was charged with **S2** (804 mg, 4.17 mmol), (*R,R*)-1,2-diphenylethylenediamine (8.75 mmol) and DCM (20 mL). After the resulting mixture was stirred at refluxing temperature for two days, water (20 mL) was added and the phases were separated. The aqueous phase was extracted with DCM (20 mL x 2). The combined organic layer was dried over MgSO<sub>4</sub> and the solvent was removed in vacuo to give a yellow solid, which was purified by crystallization to give **L1** as a white solid.

A 100 mL round-bottom flask was charged with **L1** (1.0 mmol), DMAP (3.0 mmol) and DCM (15 mL). The resulting mixture was cooled to 0 °C, and acyl chloride (2.5 mmol) was added neat at once. The ice bath was removed, and the reaction mixture was stirred at room temperature for 5 hours. The solvent was removed under vacuo, the residue was partitioned between saturated NH<sub>4</sub>Cl (25 mL) and ethyl acetate (25 mL), and the aqueous phase was re-extracted with ethyl acetate (25 mL x 2). The combined organic layer was dried over MgSO<sub>4</sub>, and the solvent was removed in vacuo to give **L2** as a white solid, which was purified by column chromatography on silica gel.

## Characterization of representative Pybim ligands

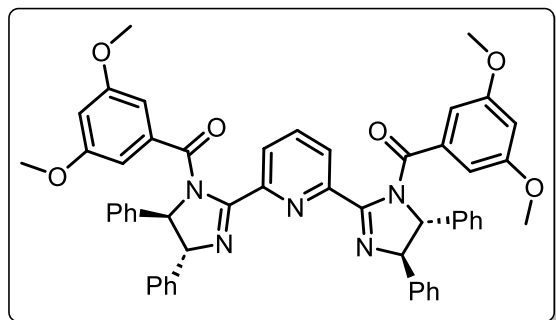
### **((4*R*,4'*R*,5*R*,5'*R*)-pyridine-2,6-diylbis(4,5-diphenyl-4,5-dihydro-1*H*-imidazole-2,1-diyl))bis(phenylmethanone) (L17)**



Purification by flash chromatography (PE/EA = 2/1). White solid, 65% yield.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 7.93 (d, *J* = 7.7 Hz, 2H), 7.78 (t, *J* = 7.8 Hz, 1H), 7.36 (m, 19H), 7.25 – 7.21 (m, 2H), 7.10 – 7.02 (m, 9H), 5.16 (m, 4H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 169.6, 160.7, 149.5, 141.6, 134.0, 137.4, 134.8, 131.6, 129.2, 129.1, 128.4, 128.2, 128.0, 127.7, 126.4, 125.2, 124.6, 79.2, 71.5.

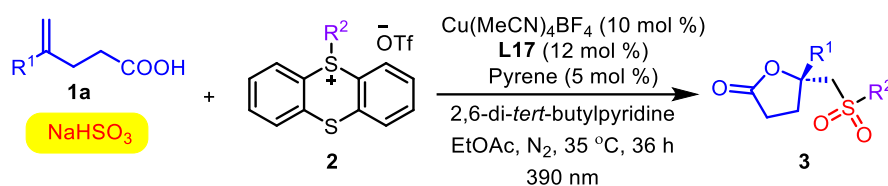
### **((4*R*,4'*R*,5*R*,5'*R*)-pyridine-2,6-diylbis(4,5-diphenyl-4,5-dihydro-1*H*-imidazole-2,1-diyl))bis((3,5-dimethoxyphenyl)methanone) (L16)**



Purification by flash chromatography (PE/EA = 2/1). White solid, 60% yield.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 8.06 (s, 2H), 7.88 (s, 1H), 7.49 (s, 4H), 7.39 (m, 11H), 7.14 (s, 5H), 6.50 (s, 4H), 6.32 (s, 2H), 5.14 (s, 4H), 3.20 (s, 12H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 169.3, 160.2, 149.5, 142.1, 139.5, 137.3, 136.0, 129.4, 129.2, 128.3, 127.7, 126.3, 125.1, 124.5, 106.1, 105.2, 79.5, 71.1, 54.8.

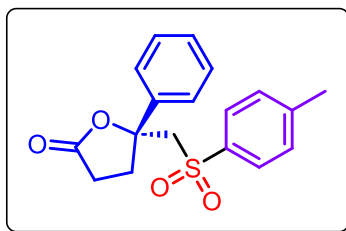
### 3. General procedure for the synthesis of compound 3



In a screw-capped vial,  $\text{Cu}(\text{MeCN})_4\text{BF}_4$  (3.2 mg, 10 mol %) and **L17** (8.7 mg, 12 mol %) were combined with EtOAc (2 mL) under  $\text{N}_2$  atmosphere. The resultant solution was stirred for 30 min at room temperature. Enoic acid **1** (0.1 mmol, 1.0 equiv), thianthrenium salt **2** (0.15 mmol, 1.5 equiv),  $\text{NaHSO}_3$  (15.6 mg, 0.15 mmol, 1.5 equiv), pyrene (5 mol %) and 2,6-di-*tert*-butylpyridine (0.2 mmol, 2.0 equiv) were added under  $\text{N}_2$  atmosphere. The reaction was placed 4 cm away from a 390 nm Kessil lamp and irradiated for 36 h while stirring at 900 rpm. Then the mixture was purified directly by flash column chromatography (*n*-hexane/EtOAc (v/v): 5/1 - 2/1) to provide the desired product **3**.

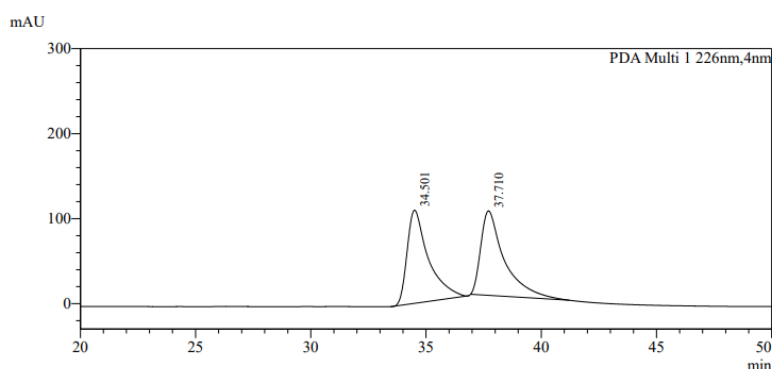
## 4. Characterization of all products and HPLC chromatograph

### (*R*)-5-phenyl-5-(tosylmethyl)dihydrofuran-2(3*H*)-one (3a)<sup>[6]</sup>



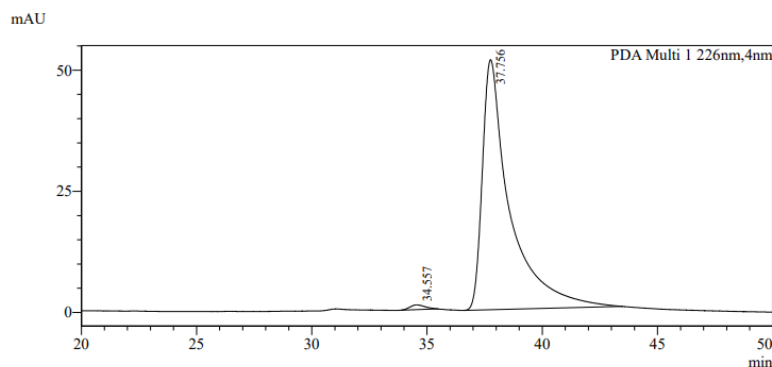
Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 80% yield, 97% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.70 (d, *J* = 8.4 Hz, 2H), 7.35 – 7.29 (m, 7H), 3.78 – 3.69 (m, 2H), 3.41 – 3.33 (m, 1H), 2.67 – 2.60 (m, 1H), 2.90 – 2.81 (m, 1H), 2.54 – 2.45 (m, 1H), 2.42 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 175.3, 145.0, 142.0, 137.6, 129.9, 128.9, 128.5, 127.9, 124.5, 84.7, 65.1, 32.5, 28.3, 21.7.

**HPLC analysis:** CHIRALPAK AD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 0.5 mL/min, wave length = 226 nm, *t*<sub>R</sub> = 34.56 min (minor), *t*<sub>R</sub> = 37.76 min (major).



<Peak Table>

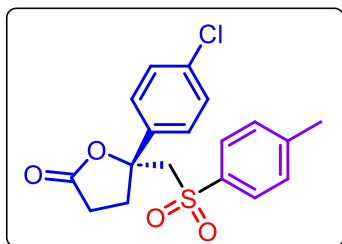
| PDA Ch1 226nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 34.501    | 7064326 | 109565 | 49.956 |
| 2             | 37.710    | 7076709 | 99389  | 50.044 |



<Peak Table>

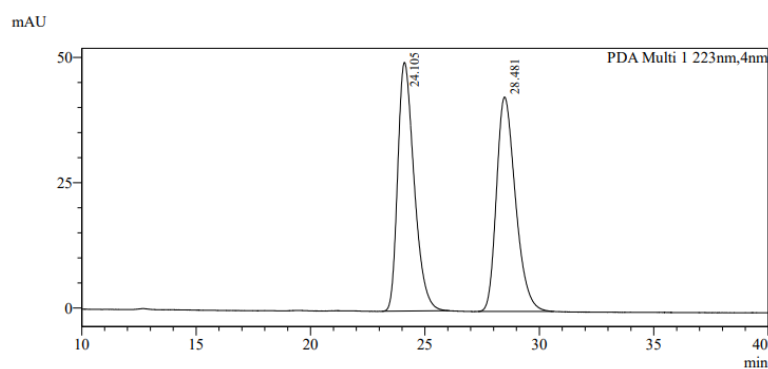
| PDA Ch1 226nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 34.557    | 44260   | 966    | 1.020  |
| 2             | 37.756    | 4293897 | 51582  | 98.980 |

**(*R*)-5-(4-chlorophenyl)-5-(tosylmethyl)dihydrofuran-2(3*H*)-one (3b)**<sup>[6]</sup>



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). white solid, 76% yield, 90% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  (ppm) = 7.65 (d, *J* = 8.4 Hz, 2H), 7.31 – 7.23 (m, 6H), 3.72 (t, *J* = 15.2 Hz, 2H), 3.33 – 3.26 (m, 1H), 2.89 – 2.80 (m, 1H), 2.65 – 2.58 (m, 1H), 2.54 – 2.45 (m, 1H), 2.43(s, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**  $\delta$  (ppm) = 175.0, 145.2, 139.9, 137.3, 134.7, 129.9, 129.0, 127.9, 126.3, 84.3, 65.0, 32.6, 28.2, 21.7.

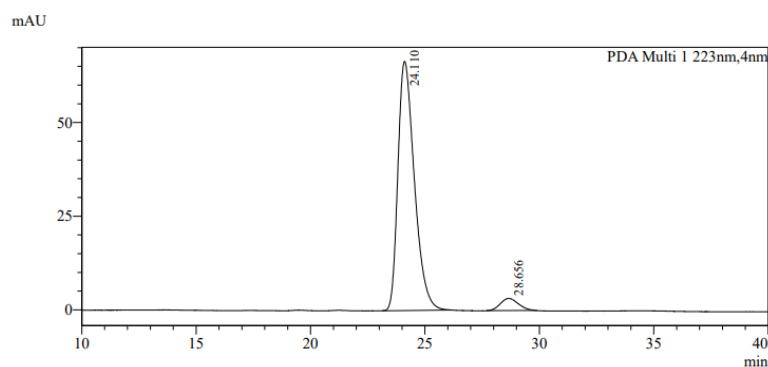
HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 223 nm, *t<sub>R</sub>* = 24.11 min (major), *t<sub>R</sub>* = 28.66 min (minor).



<Peak Table>

PDA Ch1 223nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 24.105    | 2474118 | 49663  | 50.005 |
| 2     | 28.481    | 2473659 | 42801  | 49.995 |

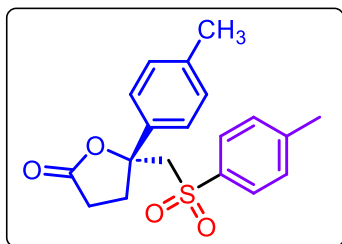


<Peak Table>

PDA Ch1 223nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 24.110    | 3325496 | 66534  | 94.890 |
| 2     | 28.656    | 179088  | 3250   | 5.110  |

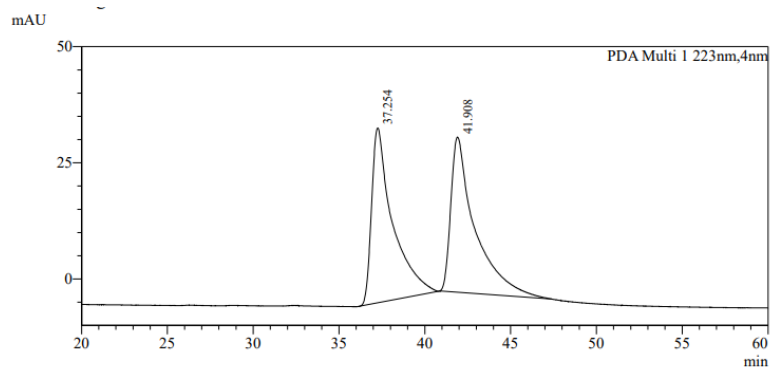
**(*R*)-5-(*p*-tolyl)-5-(tosylmethyl)dihydrofuran-2(3*H*)-one (3c)**<sup>[6]</sup>



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 80% yield, 94% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.68 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.15 (dd, *J* = 23.6, 8.0 Hz, 4H), 3.72 (dd, *J* = 17.2, 15.2 Hz, 2H), 3.37 – 3.29 (m, 1H), 2.86 – 2.78 (m, 1H), 2.53 – 2.46 (m, 1H), 2.43 (s, 3H), 2.32 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 175.4, 144.9, 138.8, 138.5, 137.5, 129.8, 129.5, 127.9,

124.5, 84.8, 65.2, 32.4, 28.3, 21.7, 21.0.

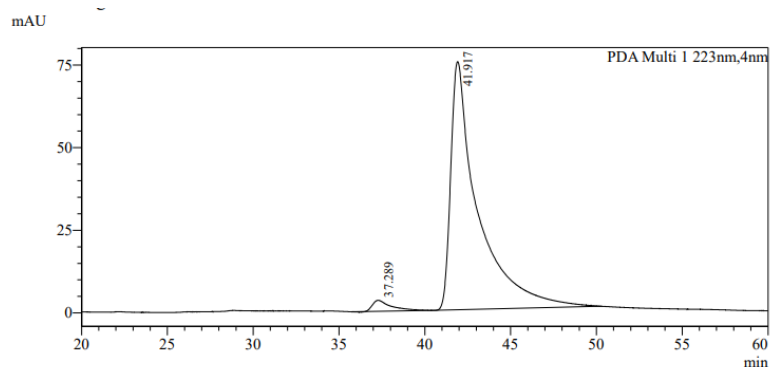
HPLC analysis: CHIRALPAK AD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 0.5 mL/min, wave length = 223 nm, *t*<sub>R</sub> = 37.29 min (minor), *t*<sub>R</sub> = 41.92 min (major).



<Peak Table>

PDA Ch1 223nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 37.254    | 3204438 | 37613  | 49.616 |
| 2     | 41.908    | 3254002 | 33358  | 50.384 |

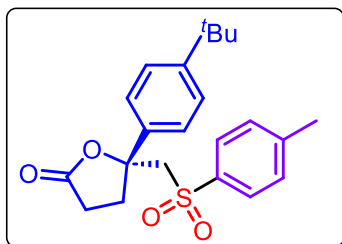


<Peak Table>

PDA Ch1 223nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 37.289    | 252497  | 3340   | 2.961  |
| 2     | 41.917    | 8273619 | 75074  | 97.039 |

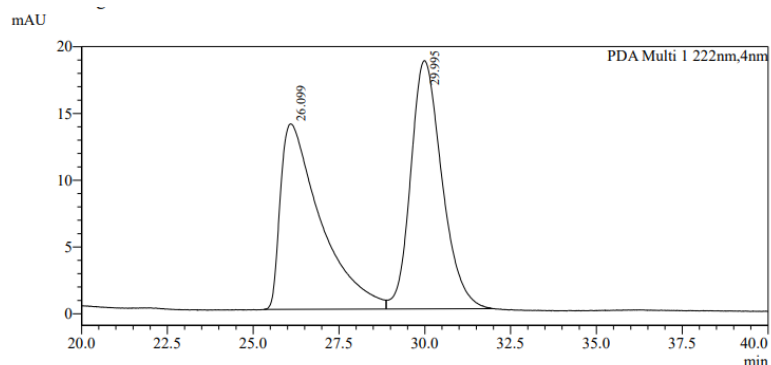
**(*R*)-5-(4-(*tert*-butyl)phenyl)-5-(tosylmethyl)dihydrofuran-2(3*H*)-one (3d)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 99% yield, 98% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 7.67 (d, *J* = 8.4 Hz, 2H), 7.33 – 7.20 (m, 6H), 3.75 (dd, *J* = 16.8, 15.2 Hz, 2H), 3.39 – 3.31 (m, 1H), 2.89 – 2.80 (m, 1H), 2.68 – 2.61 (m, 1H), 2.54 – 2.45 (m, 1H), 2.41 (s, 3H), 1.28 (s, 9H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 175.5, 151.6, 144.8, 138.7, 137.6, 129.8, 127.9, 125.8, 124.3, 84.8, 65.1, 34.5,

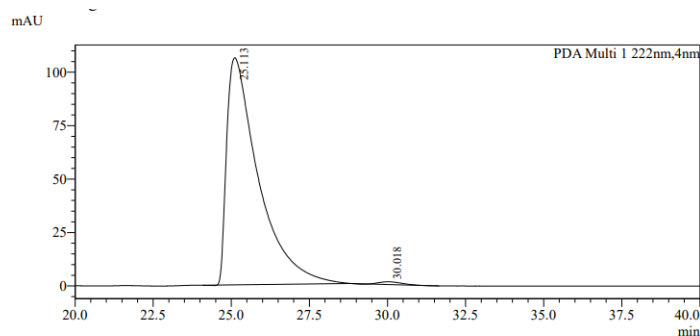
32.3, 31.2, 28.3, 21.7. HRMS (ESI) for C<sub>22</sub>H<sub>27</sub>O<sub>4</sub>S<sup>+</sup> [M+H]<sup>+</sup> calcd. 387.1625, found: 387.1625.

HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 90:10, flow rate = 1.0 mL/min, wave length = 222 nm, *t*<sub>R</sub> = 25.11 min (major), *t*<sub>R</sub> = 30.02 min (minor).



<Peak Table>

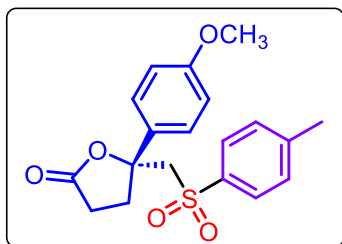
| PDA Ch1 222nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 26.099    | 1133772 | 13887  | 49.019 |
| 2             | 29.995    | 1179136 | 18593  | 50.981 |



<Peak Table>

| PDA Ch1 222nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 25.113    | 7518330 | 106256 | 99.209 |
| 2             | 30.018    | 59911   | 1278   | 0.791  |

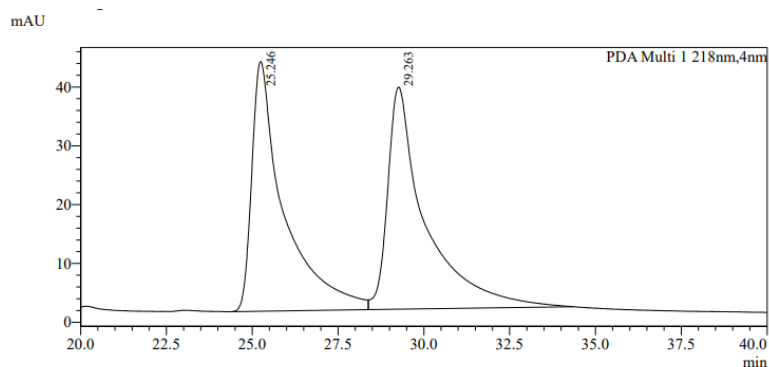
**(*R*)-5-(4-methoxyphenyl)-5-(tosylmethyl)dihydrofuran-2(3*H*)-one (3e)<sup>[6]</sup>**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 99% yield, 87% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.66 (d, *J* = 8.0 Hz, 2H), 7.30 – 7.21 (m, 4H), 6.83 – 6.81 (m, 2H), 3.78 (s, 3H), 3.72 (dd, *J* = 16.4, 15.2 Hz, 2H), 3.33 – 3.25 (m, 1H), 2.85 – 2.77 (m, 1H), 2.68 – 2.61 (m, 1H), 2.53 – 2.44 (m, 1H), 2.42 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 175.4, 159.6, 144.9, 137.5, 133.4, 129.8, 127.9, 126.1,

114.1, 84.7, 65.3, 55.4, 32.4, 28.4, 21.7.

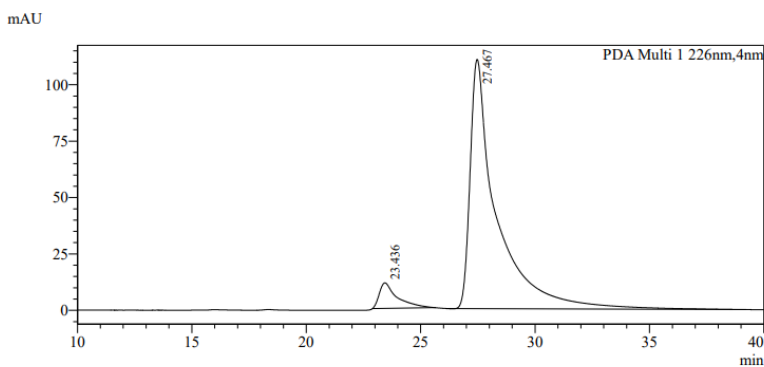
HPLC analysis: CHIRALPAK AD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 218 nm, *t*<sub>R</sub> = 23.44 min (minor), *t*<sub>R</sub> = 27.47 min (major) 错误!未找到引用源。.



<Peak Table>

PDA Ch1 218nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 25.246    | 2765848 | 42453  | 48.916 |
| 2     | 29.263    | 2888398 | 37776  | 51.084 |

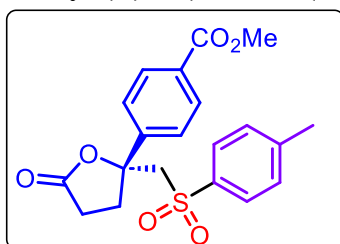


<Peak Table>

PDA Ch1 226nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 23.436    | 617843  | 11337  | 6.448  |
| 2     | 27.467    | 8964389 | 110498 | 93.552 |

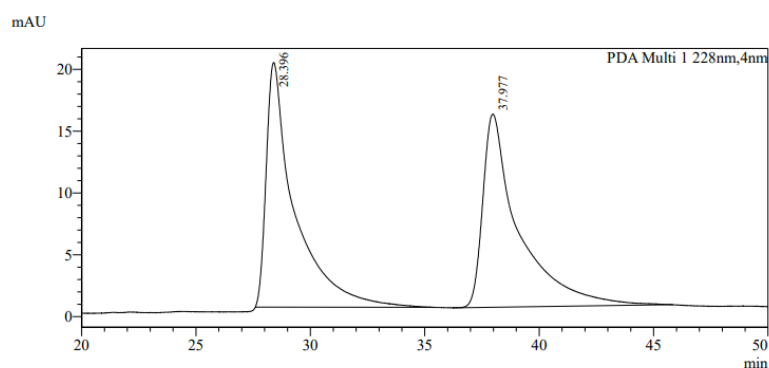
**methyl (*R*)-4-(5-oxo-2-(tosylmethyl)tetrahydrofuran-2-yl)benzoate (3f)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 70% yield, 92% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  (ppm) = 7.99 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 3.92 (s, 3H), 3.74 (s, 2H), 3.40 – 3.32 (m, 1H), 2.92 – 2.84 (m, 1H), 2.66 – 2.59 (m, 1H), 2.55 – 2.46 (m, 1H), 2.42 (s, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**  $\delta$  (ppm) = 174.9, 166.2, 146.5, 145.2, 137.3, 130.3,

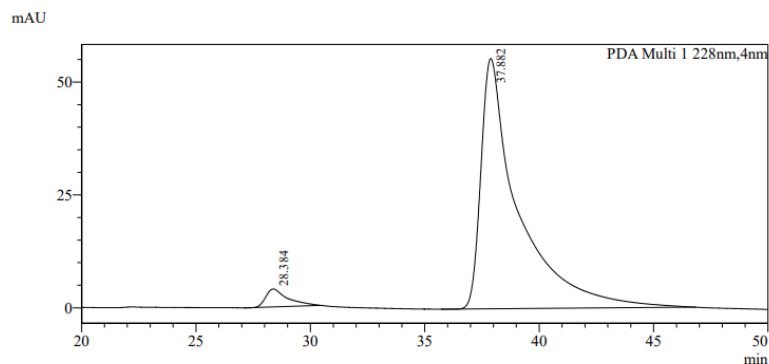
130.1, 129.9, 127.9, 124.7, 84.4, 64.7, 52.3, 32.7, 28.1, 21.7. HRMS (ESI) for C<sub>20</sub>H<sub>20</sub>NaO<sub>6</sub>S<sup>+</sup> [M+Na]<sup>+</sup> calcd. 411.0878, found: 411.0865.

HPLC analysis: CHIRALPAK AD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 228 nm, *t<sub>R</sub>* = 28.38 min (minor), *t<sub>R</sub>* = 37.88 min (major).



<Peak Table>

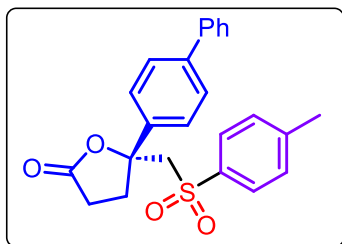
| PDA Ch1 228nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 28.396    | 1712243 | 19793  | 50.083 |
| 2             | 37.977    | 1706590 | 15620  | 49.917 |



<Peak Table>

| PDA Ch1 228nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 28.384    | 260704  | 3960   | 3.955  |
| 2             | 37.882    | 6331152 | 55431  | 96.045 |

**(*R*)-5-([1,1'-biphenyl]-4-yl)-5-(tosylmethyl)dihydrofuran-2(3*H*)-one (3g)<sup>[6]</sup>**



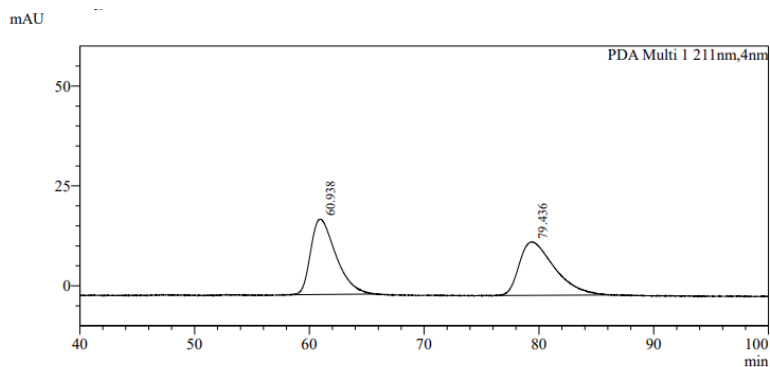
Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). white solid, 76% yield, 91% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**

$\delta$  (ppm) = 7.60 (d, *J* = 8.4 Hz, 2H), 7.46 – 7.44 (m, 4H), 7.38 – 7.34 (m, 2H), 7.30 – 7.27 (m, 3H), 7.21 – 7.18 (m, 2H), 3.72 (t, *J* = 15.2 Hz, 2H), 3.32 – 3.25 (m, 1H), 2.83 – 2.75 (m, 1H), 2.65 – 2.58 (m, 1H), 2.50 – 2.41 (m, 1H), 2.31 (s, 3H). **<sup>13</sup>C NMR (100**

**MHz, CDCl<sub>3</sub>)**  $\delta$  (ppm) = 175.3, 145.0, 141.5, 140.5, 134.0, 137.5,

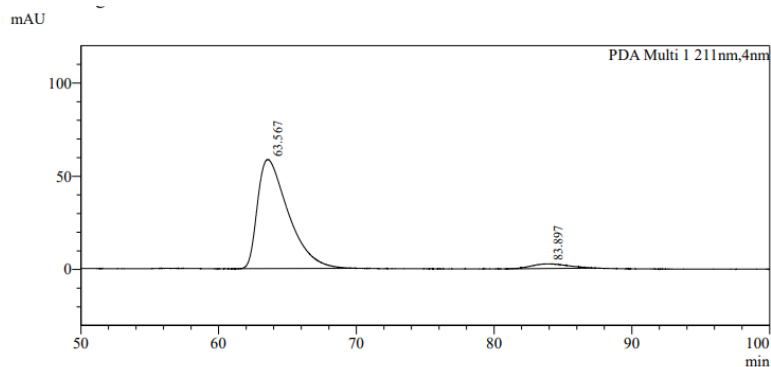
129.9, 128.9, 127.9, 127.8, 127.5, 127.1, 125.2, 84.7, 65.1, 32.5, 28.3, 21.7.

HPLC analysis: CHIRALPAK AD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 211 nm, *t<sub>R</sub>* = 63.57 min (major), *t<sub>R</sub>* = 83.90 min (minor).



<Peak Table>

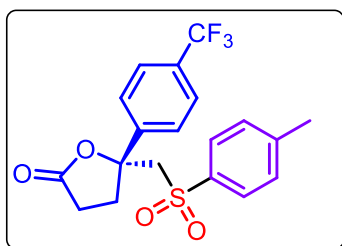
| PDA Ch1 211nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 60.938    | 2890782 | 18870  | 50.304 |
| 2             | 79.436    | 2855841 | 13398  | 49.696 |



<Peak Table>

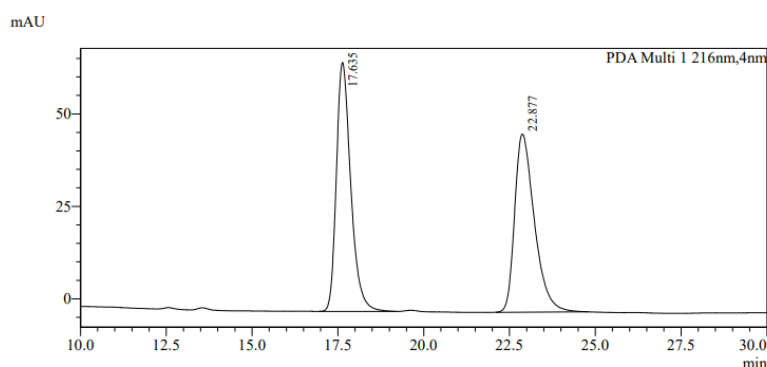
| PDA Ch1 211nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 63.567    | 9112161 | 58538  | 95.321 |
| 2             | 83.897    | 447303  | 2456   | 4.679  |

**(*R*)-5-(tosylmethyl)-5-(4-(trifluoromethyl)phenyl)dihydrofuran-2(3H)-one (3h)**



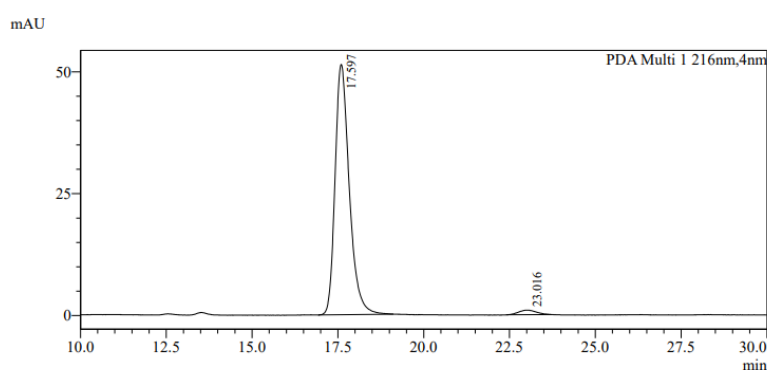
Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). white solid, 67% yield, 96% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 7.64 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 3.76 (s, 2H), 3.37 – 3.29 (m, 1H), 2.92 – 2.84 (m, 1H), 2.68 – 2.61 (m, 1H), 2.55 – 2.46 (m, 1H), 2.42 (s, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 174.7, 145.2, 137.1, 130.8 (d, *J* = 33 Hz), 129.9, 127.9, 125.9 (q, *J* = 3.0 Hz), 125.3, 125.0, 122.3, 84.2, 64.8, 32.8, 28.0, 21.6. **<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)** δ –62.80. HRMS (ESI) for C<sub>19</sub>H<sub>17</sub>F<sub>3</sub>NaO<sub>4</sub>S<sup>+</sup> [M+Na]<sup>+</sup> calcd. 421.0698, found: 421.0692.

HPLC analysis: CHIRALPAK IA (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 223 nm, *t*<sub>R</sub> = 17.60 min (major), *t*<sub>R</sub> = 23.02 min (minor).



<Peak Table>

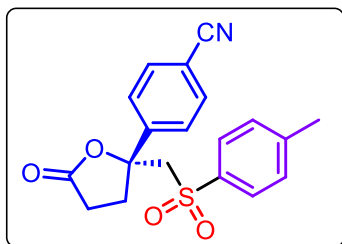
| PDA Ch1 216nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 17.635    | 1966437 | 67335  | 50.493 |
| 2             | 22.877    | 1928024 | 48137  | 49.507 |



<Peak Table>

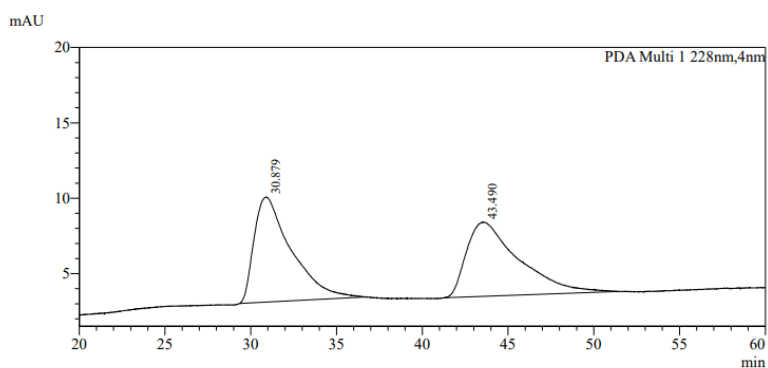
| PDA Ch1 216nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 17.597    | 1481144 | 51364  | 97.776 |
| 2             | 23.016    | 33687   | 931    | 2.224  |

**(R)-4-(5-oxo-2-(tosylmethyl)tetrahydrofuran-2-yl)benzonitrile (3i)**



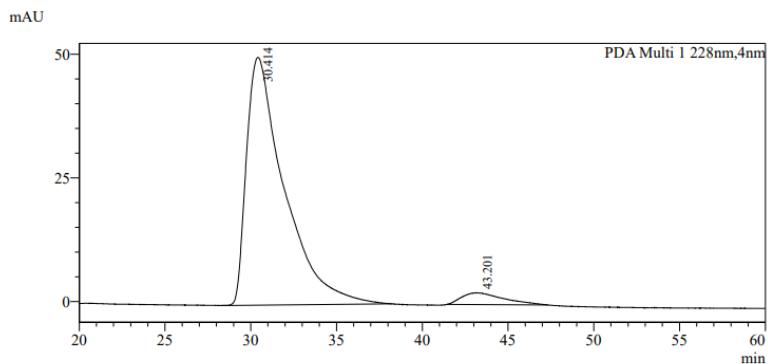
Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). white solid, 99% yield, 90% ee.  $[\alpha]_D^{20} = -30.0$  ( $c = 0.12$ ,  $\text{CHCl}_3$ ).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) = 7.67 (dd,  $J = 13.6, 8.4$  Hz, 4H), 7.48 (d,  $J = 8.4$  Hz, 2H), 7.32 (d,  $J = 8.4$  Hz, 2H), 3.74 – 3.67 (m, 2H), 3.39 – 3.31 (m, 1H), 2.94 – 2.85 (m, 1H), 2.64 – 2.48 (m, 2H), 2.45 (s, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) = 174.5, 146.7, 145.5, 137.2, 132.7, 130.0, 127.9, 125.7, 118.0, 112.7, 84.0, 64.7, 32.7, 28.0, 21.7. HRMS (ESI) for  $\text{C}_{19}\text{H}_{17}\text{NaO}_4\text{S}^+$   $[\text{M}+\text{Na}]^+$  calcd. 378.0776, found: 378.0777.

HPLC analysis: CHIRALPAK IA (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 228 nm,  $t_R = 30.41$  min (major),  $t_R = 43.20$  min (minor).



<Peak Table>

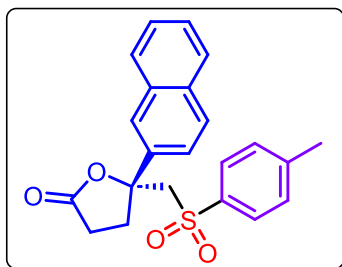
| PDA Ch1 228nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 30.879    | 1047182 | 6963   | 50.151 |
| 2             | 43.490    | 1040877 | 4922   | 49.849 |



<Peak Table>

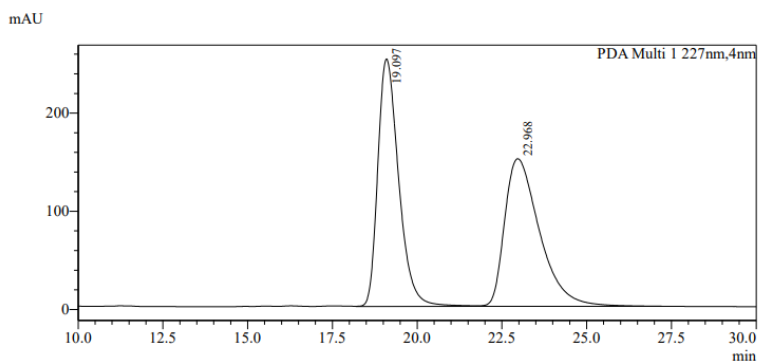
| PDA Ch1 228nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 30.414    | 7674329 | 50066  | 95.033 |
| 2             | 43.201    | 401116  | 2375   | 4.967  |

**(R)-5-(naphthalen-2-yl)-5-(tosylmethyl)dihydrofuran-2(3H)-one (3j)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 75% yield, 90% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.81 – 7.76 (m, 4H), 7.64 (d, *J* = 8.0 Hz, 2H), 7.52 – 7.50 (m, 2H), 7.33 – 7.31 (m, 1H), 7.18 (d, *J* = 8.0 Hz, 2H), 3.88 – 3.80 (m, 2H), 3.44 – 3.37 (m, 1H), 2.92 – 2.84 (m, 1H), 2.79 – 2.72 (m, 1H), 2.58 – 2.49 (m, 1H), 2.34 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 175.4, 144.9, 138.4, 137.3, 132.9, 132.7, 129.7, 129.1, 128.3, 127.9, 127.6, 126.9, 123.8, 122.2, 84.8, 64.9, 32.4, 28.2, 21.6. HRMS (ESI) for C<sub>22</sub>H<sub>20</sub>NaO<sub>4</sub>S<sup>+</sup> [M+Na]<sup>+</sup> calcd. 403.0975, found: 403.0973.

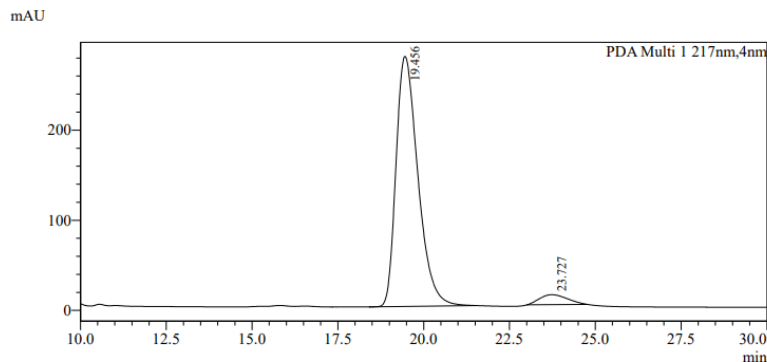
HPLC analysis: CHIRALPAK AS-H (*n*-hexane/*i*-PrOH) = 50:50, flow rate = 1.0 mL/min, wave length = 217 nm, t<sub>R</sub> = 19.46 min (major), t<sub>R</sub> = 23.73 min (minor).



<Peak Table>

PDA Ch1 227nm

| Peak# | Ret. Time | Area     | Height | Aera%  |
|-------|-----------|----------|--------|--------|
| 1     | 19.097    | 10824923 | 252063 | 50.059 |
| 2     | 22.968    | 10799555 | 150389 | 49.941 |

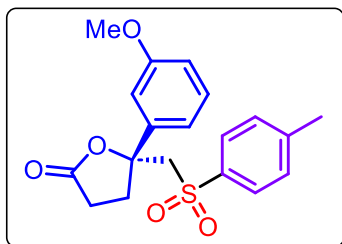


<Peak Table>

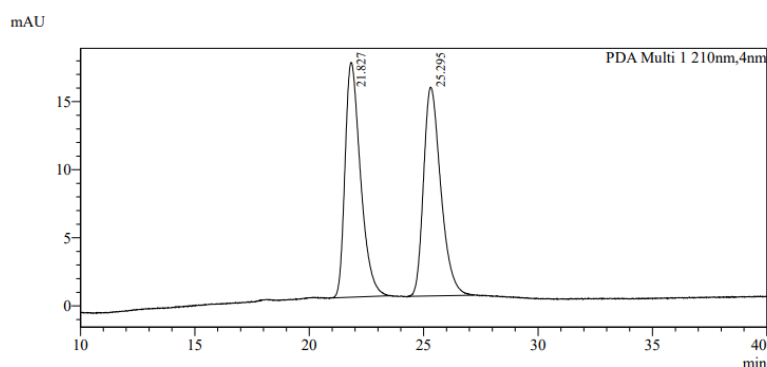
PDA Ch1 217nm

| Peak# | Ret. Time | Area     | Height | Aera%  |
|-------|-----------|----------|--------|--------|
| 1     | 19.456    | 12312895 | 277454 | 95.164 |
| 2     | 23.727    | 625767   | 11077  | 4.836  |

**(R)-5-(3-methoxyphenyl)-5-(tosylmethyl)dihydrofuran-2(3H)-one (3k)**

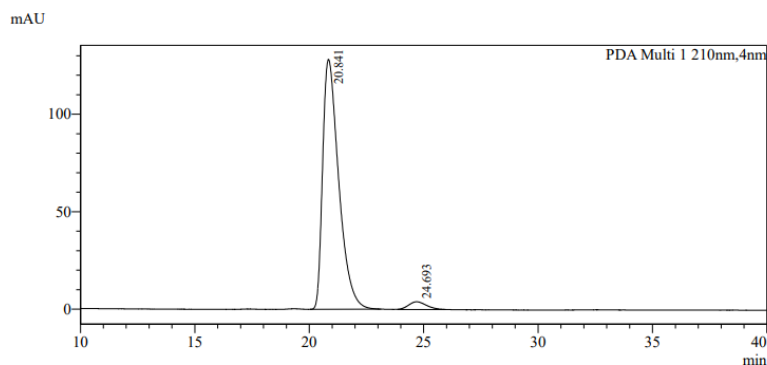


Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 78% yield, 93% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.63 (d, *J* = 8.0 Hz, 2H), 7.24 – 7.15 (m, 3H), 6.80 – 6.73 (m, 3H), 3.69 (s, 3H), 3.66 (d, *J* = 3.6 Hz, 2H), 3.32 – 3.25 (m, 1H), 2.82 – 2.74 (m, 1H), 2.57 – 2.38 (m, 2H), 2.35 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 174.3, 158.9, 144.0, 142.6, 136.5, 129.0, 128.8, 126.9, 115.6, 112.7, 109.4, 83.6, 63.9, 54.3, 31.4, 27.2, 20.6. HRMS (ESI) for C<sub>19</sub>H<sub>20</sub>NaO<sub>5</sub>S<sup>+</sup> [M+Na]<sup>+</sup> calcd. 383.0924, found: 383.0920. HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 210 nm, t<sub>R</sub> = 20.84 min (minor), t<sub>R</sub> = 24.69 min (major).



<Peak Table>

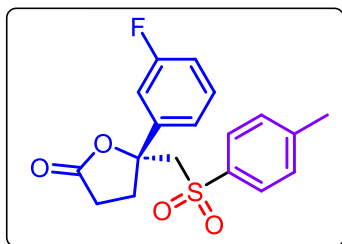
| PDA Ch1 210nm |           |        |        |        |
|---------------|-----------|--------|--------|--------|
| Peak#         | Ret. Time | Area   | Height | Aera%  |
| 1             | 21.827    | 810065 | 17246  | 50.176 |
| 2             | 25.295    | 804378 | 15346  | 49.824 |



<Peak Table>

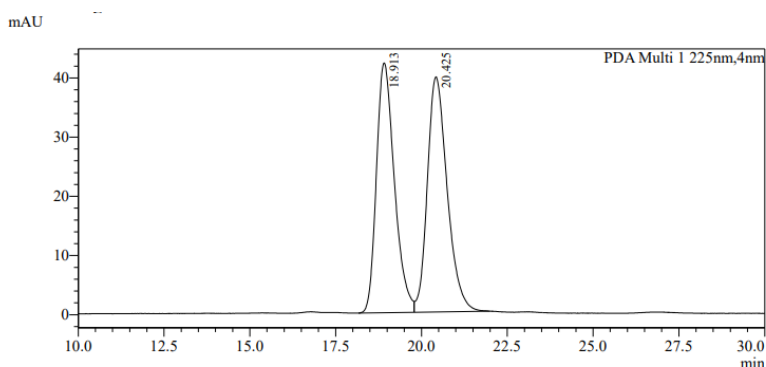
| PDA Ch1 210nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 20.841    | 6273265 | 128239 | 96.713 |
| 2             | 24.693    | 213179  | 3964   | 3.287  |

**(R)-5-(3-fluorophenyl)-5-(tosylmethyl)dihydrofuran-2(3H)-one (3l)**



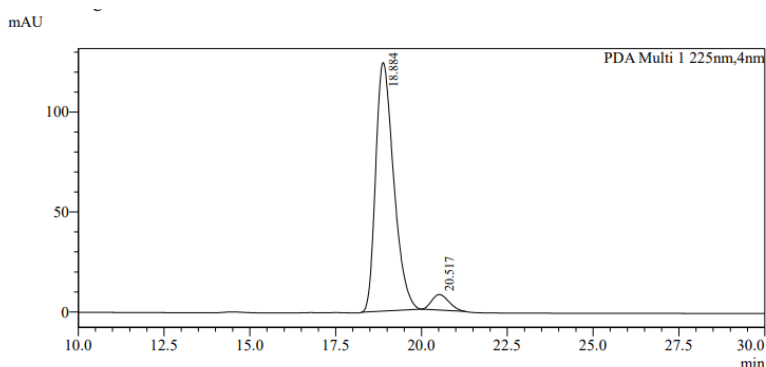
Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 86% yield, 89% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 7.62 (d, *J* = 8.4 Hz, 2H), 7.25 – 7.19 (m, 3H), 7.03 (d, *J* = 8.0 Hz, 1H), 6.97 – 6.89 (m, 2H), 3.69 – 3.61 (m, 2H), 3.30 – 3.21 (m, 1H), 2.83 – 2.75 (m, 1H), 2.56 – 2.39 (m, 2H), 2.36 (s, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 175.0, 162.8 (d, *J* = 247 Hz), 145.2, 144.4 (d, *J* = 7.0 Hz), 137.3, 130.7 (d, *J* = 8.0 Hz), 129.9, 127.9, 120.3 (d, *J* = 3.0 Hz), 115.5 (d, *J* = 21 Hz), 112.4 (d, *J* = 24 Hz), 84.1 (d, *J* = 2.0 Hz), 64.8, 32.6, 28.1, 21.7. **<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)** δ –110.92 – –110.98 (m). HRMS (ESI) for C<sub>18</sub>H<sub>17</sub>FN<sub>4</sub>O<sub>4</sub>S<sup>+</sup> [M+Na]<sup>+</sup> calcd. 371.0725, found: 371.0721.

HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 225 nm, t<sub>R</sub> = 18.88 min (major), t<sub>R</sub> = 20.52 min (minor).



<Peak Table>

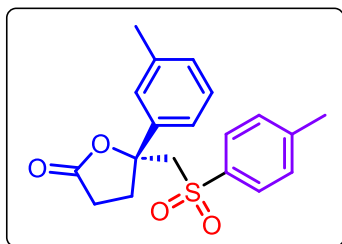
| PDA Ch1 225nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 18.913    | 1536170 | 42193  | 49.017 |
| 2             | 20.425    | 1597797 | 39698  | 50.983 |



<Peak Table>

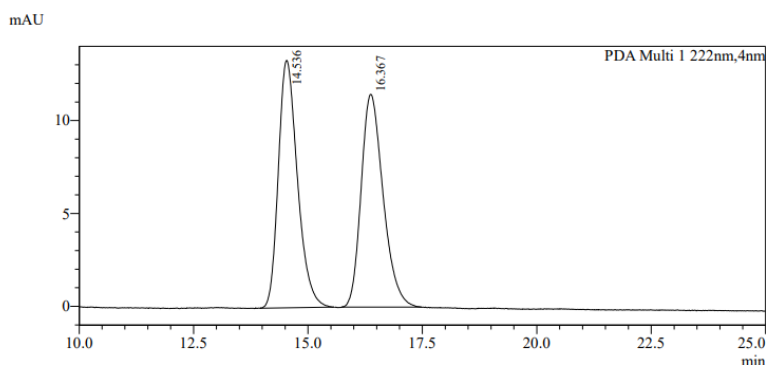
| PDA Ch1 225nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 18.884    | 4549081 | 124344 | 94.471 |
| 2             | 20.517    | 266258  | 7712   | 5.529  |

**(*R*)-5-(*m*-tolyl)-5-(tosylmethyl)dihydrofuran-2(3*H*)-one (3m)**



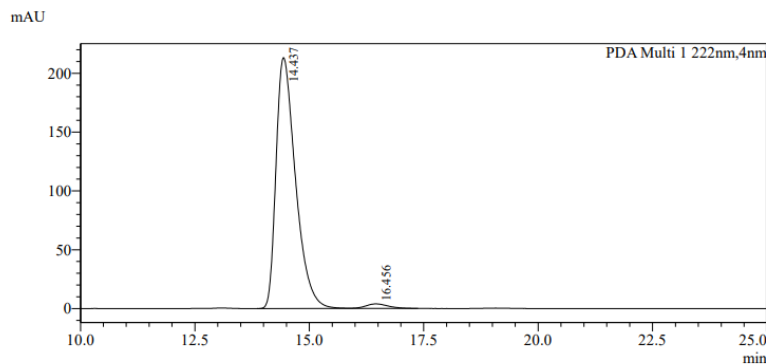
Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 72% yield, 96% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.69 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.24 – 7.20 (m, 1H), 7.10 – 7.08 (m, 3H), 3.77 – 3.69 (m, 2H), 3.40 – 3.32 (m, 1H), 2.88 – 2.80 (m, 1H), 2.66 – 2.59 (m, 1H), 2.54 – 2.47 (m, 1H), 2.43 (s, 3H), 2.30 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 175.5, 144.9, 141.9, 138.7, 137.5, 129.8, 129.2, 128.8, 127.9, 125.1, 121.6, 84.8, 65.1, 32.4, 28.3, 21.7, 21.5. HRMS (ESI) for C<sub>19</sub>H<sub>20</sub>NaO<sub>4</sub>S<sup>+</sup> [M+Na]<sup>+</sup> calcd. 367.0975, found: 367.0972.

HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 222 nm, *t*<sub>R</sub> = 14.44 min (major), *t*<sub>R</sub> = 16.46 min (minor).



<Peak Table>

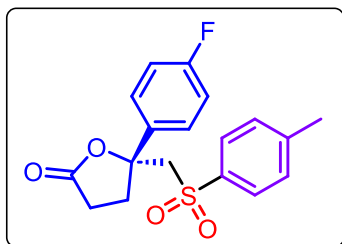
| PDA Ch1 222nm |           |        |        |        |
|---------------|-----------|--------|--------|--------|
| Peak#         | Ret. Time | Area   | Height | Aera%  |
| 1             | 14.536    | 386944 | 13318  | 50.254 |
| 2             | 16.367    | 383029 | 11442  | 49.746 |



<Peak Table>

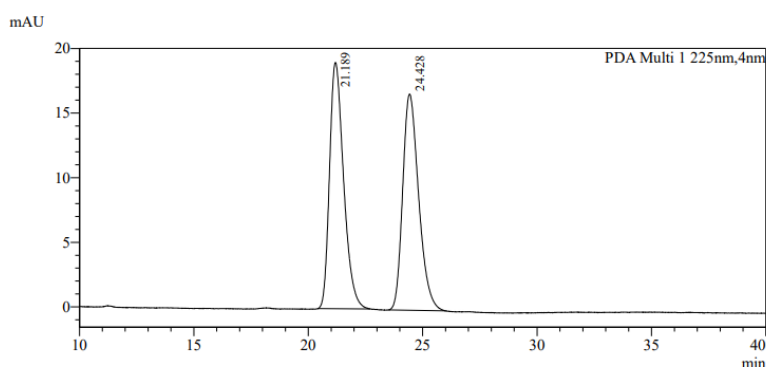
| PDA Ch1 222nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 14.437    | 6320132 | 213233 | 97.888 |
| 2             | 16.456    | 136390  | 3899   | 2.112  |

**(*R*)-5-(4-fluorophenyl)-5-(tosylmethyl)dihydrofuran-2(3*H*)-one (3n)<sup>[6]</sup>**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 86% yield, 91% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.67 (d, *J* = 8.0 Hz, 2H), 7.32 – 7.29 (m, 4H), 7.03 – 6.99 (m, 2H), 3.76 – 3.68 (m, 2H), 3.36 – 3.28 (m, 1H), 2.89 – 2.81 (m, 1H), 2.54 – 2.46 (m, 1H), 2.54 – 2.46 (m, 1H), 2.43 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 175.1, 163.8, 161.3, 145.1, 137.4 (t, *J* = 3.0 Hz), 129.9, 127.9, 126.7 (d, *J* = 21 Hz), 115.8 (d, *J* = 8.0 Hz), 84.4, 65.2, 32.6, 28.2, 21.7. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –112.86 – –110.93 (m).

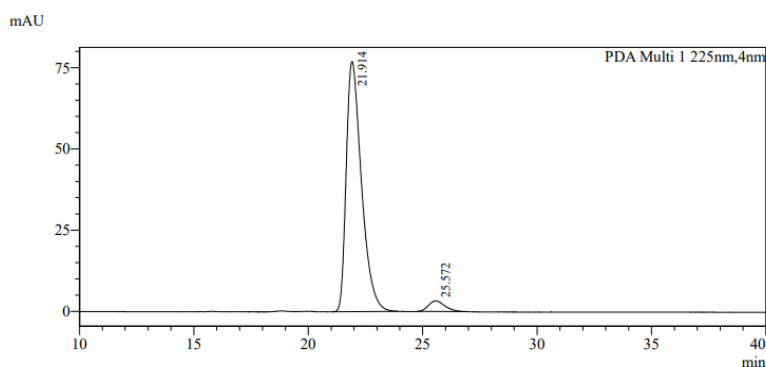
HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 225 nm, *t*<sub>R</sub> = 21.91 min (major), *t*<sub>R</sub> = 25.57 min (minor).



<Peak Table>

PDA Ch1 225nm

| Peak# | Ret. Time | Area   | Height | Aera%  |
|-------|-----------|--------|--------|--------|
| 1     | 21.189    | 815680 | 19058  | 50.115 |
| 2     | 24.428    | 811929 | 16731  | 49.885 |

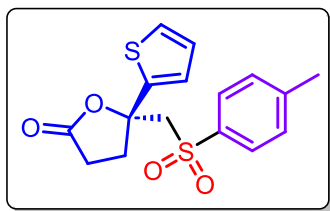


<Peak Table>

PDA Ch1 225nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 21.914    | 3565883 | 77010  | 95.604 |
| 2     | 25.572    | 163952  | 3262   | 4.396  |

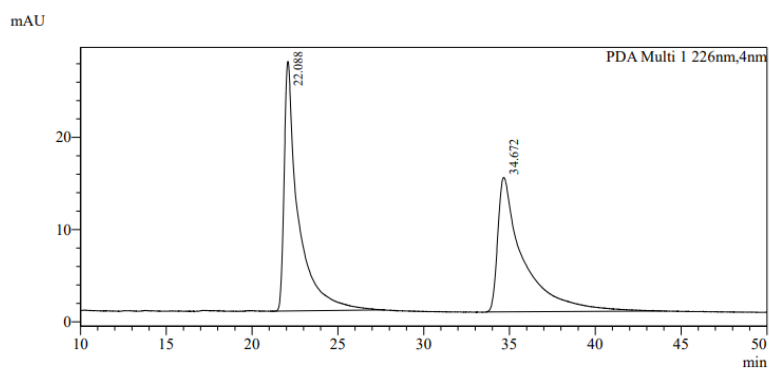
**(S)-5-(thiophen-2-yl)-5-(tosylmethyl)dihydrofuran-2(3H)-one (3o)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 70% yield, 94% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 7.67 (d, *J* = 8.0 Hz, 2H), 7.31 – 7.27 (m, 3H), 7.22 – 7.21 (m, 1H), 6.93 (dd, *J* = 5.2, 1.2 Hz, 1H), 3.81 – 3.73 (m, 2H), 3.28 – 3.20 (m, 1H), 2.84 – 2.76 (m, 1H), 2.69 – 2.50 (m, 2H), 2.43 (s, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 175.2, 145.0,

142.4, 137.4, 129.9, 127.9, 127.6, 124.7, 121.8, 83.3, 64.6, 32.4, 28.4, 21.7. HRMS (ESI) for C<sub>16</sub>H<sub>16</sub>NaO<sub>4</sub>S<sub>2</sub><sup>+</sup> [M+Na]<sup>+</sup> calcd. 359.0383, found: 359.0379.

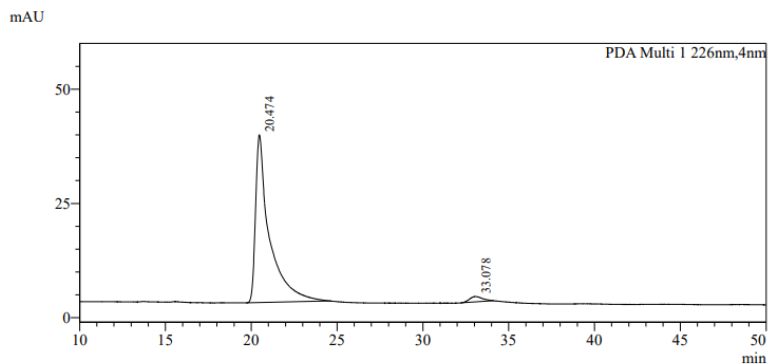
HPLC analysis: CHIRALPAK AD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 226 nm, t<sub>R</sub> = 20.47 min (major), t<sub>R</sub> = 33.09 min (minor).



<Peak Table>

PDA Ch1 226nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 22.088    | 1552279 | 27076  | 50.592 |
| 2     | 34.672    | 1515935 | 14572  | 49.408 |

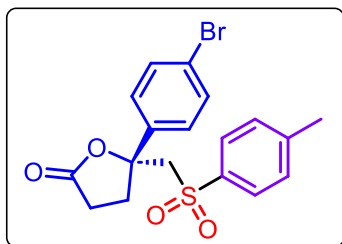


<Peak Table>

PDA Ch1 226nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 20.474    | 2072736 | 36703  | 97.140 |
| 2     | 33.078    | 61021   | 1164   | 2.860  |

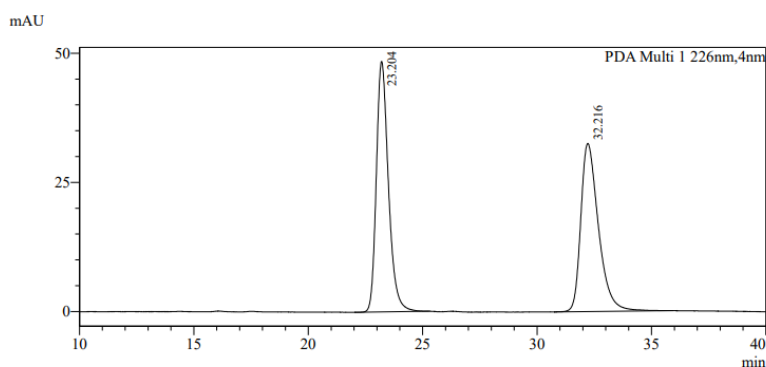
**(*R*)-5-(4-bromophenyl)-5-(tosylmethyl)dihydrofuran-2(3*H*)-one (3p)<sup>[6]</sup>**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 79% yield, 94% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.64 (d, *J* = 8.4 Hz, 2H), 7.44 (d, *J* = 8.8 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 3.75 – 3.68 (m, 2H), 3.32 – 3.25 (m, 1H), 2.88 – 2.80 (m, 1H), 2.64 – 2.57 (m, 1H), 2.53 – 2.47 (m, 1H), 2.44 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 174.9, 145.2, 140.5, 137.3, 132.0, 129.9, 127.9,

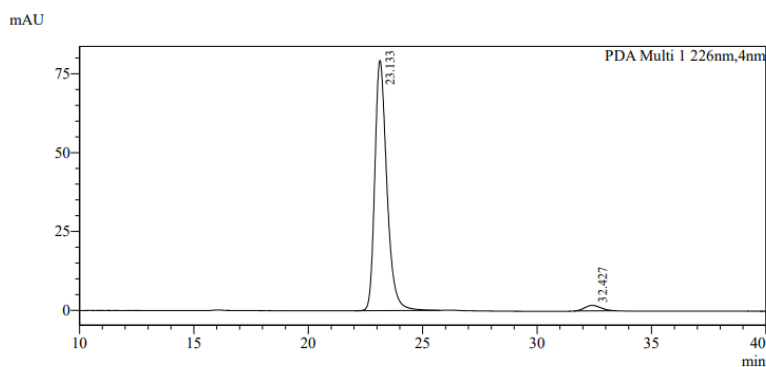
126.6, 122.8, 84.3, 64.9, 32.6, 28.1, 21.7.

HPLC analysis: CHIRALPAK IA (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 226 nm, *t*<sub>R</sub> = 23.13 min (major), *t*<sub>R</sub> = 32.43 min (minor).



<Peak Table>

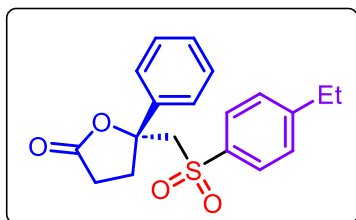
| PDA Ch1 226nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 23.204    | 1782963 | 48495  | 50.473 |
| 2             | 32.216    | 1749578 | 32548  | 49.527 |



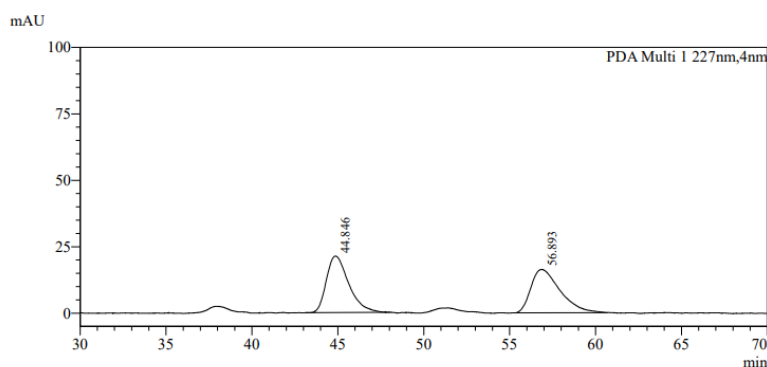
<Peak Table>

| PDA Ch1 226nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 23.133    | 2907813 | 79329  | 97.156 |
| 2             | 32.427    | 85113   | 1744   | 2.844  |

**(*R*)-5-(((4-ethylphenyl)sulfonyl)methyl)-5-phenyldihydrofuran-2(3*H*)-one (3q)**

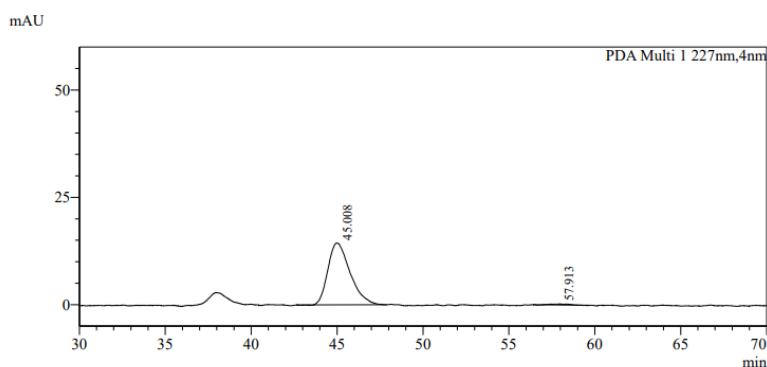


Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 90% yield, 97% ee.  $[\alpha]_D^{20} = +5.1$  (*c* = 0.1, CHCl<sub>3</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm) = 7.72 (d, *J* = 8.4 Hz, 2H), 7.33 – 7.28 (m, 7H), 3.74 (d, *J* = 17.2, 14.8 Hz, 2H), 3.41 – 3.33 (m, 1H), 2.90 – 2.82 (m, 1H), 2.74 – 2.61 (m, 3H), 2.54 – 2.45 (m, 1H), 1.24 (t, *J* = 7.6 Hz, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm) = 175.4, 151.1, 141.9, 137.7, 128.9, 128.8, 128.5, 128.0, 124.6, 84.8, 65.1, 32.5, 28.9, 28.3, 15.1. HRMS (ESI) for C<sub>19</sub>H<sub>20</sub>NaO<sub>4</sub>S<sup>+</sup> [M+Na]<sup>+</sup> calcd. 367.0980, found: 367.0977. HPLC analysis: CHIRALPAK IA (*n*-hexane/*i*-PrOH) = 95:5, flow rate = 1.0 mL/min, wave length = 227 nm, *t*<sub>R</sub> = 45.00 min (major), *t*<sub>R</sub> = 57.91 min (minor).



<Peak Table>

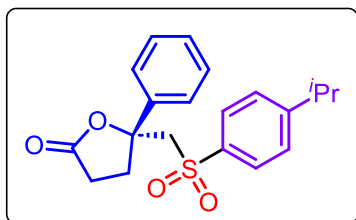
| PDA Ch1 227nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 44.846    | 1920757 | 21256  | 49.960 |
| 2             | 56.893    | 1923834 | 16298  | 50.040 |



<Peak Table>

| PDA Ch1 227nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 45.008    | 1294333 | 14421  | 98.627 |
| 2             | 57.913    | 18023   | 234    | 1.373  |

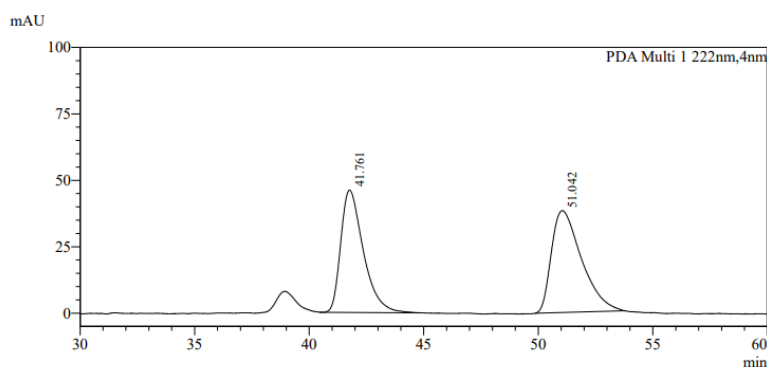
**(*R*)-5-(((4-isopropylphenyl)sulfonyl)methyl)-5-phenyldihydrofuran-2(3H)-one (3r)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1); yellow solid, 85% yield, 97% ee.  $[\alpha]_{\text{D}}^{20} = +5.4$  ( $c = 0.4$ ,  $\text{CHCl}_3$ ).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) = 7.72 (d,  $J = 9.2$  Hz, 2H), 7.35 – 7.27 (m, 7H), 3.78 – 3.70 (m, 2H), 3.40 – 3.32 (m, 1H), 3.00 – 2.83 (m, 2H), 2.69 – 2.62 (m, 1H), 2.55 – 2.46 (m, 1H), 1.26 (d,  $J = 7.2$  Hz, 6H).  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )

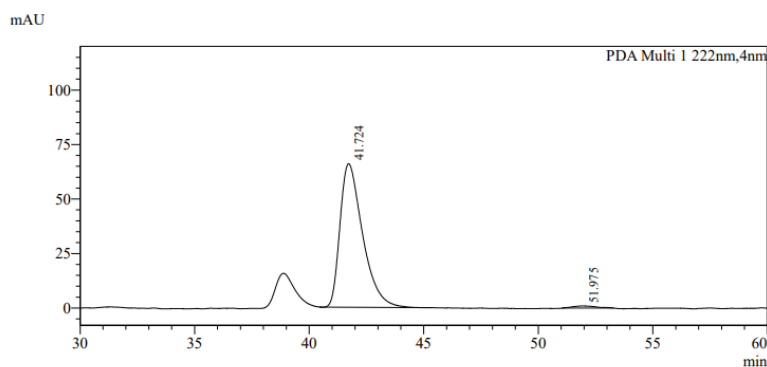
$\delta$  175.3, 155.6, 141.8, 137.7, 128.9, 128.5, 128.0, 127.4, 124.6, 84.8, 65.1, 34.3, 32.5, 28.3, 23.6. HRMS (ESI) for  $\text{C}_{20}\text{H}_{22}\text{NaO}_4\text{S}^+$   $[\text{M}+\text{Na}]^+$  calcd. 381.1137, found: 381.1128.

HPLC analysis: CHIRALPAK IA (*n*-hexane/*i*-PrOH) = 95:5, flow rate = 1.0 mL/min, wave length = 222 nm,  $t_{\text{R}} = 41.72$  min (major),  $t_{\text{R}} = 51.98$  min (minor).



<Peak Table>

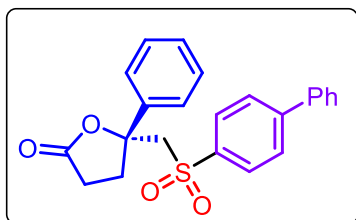
| PDA Ch1 222nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 41.761    | 3151823 | 45995  | 47.329 |
| 2             | 51.042    | 3507545 | 38333  | 52.671 |



<Peak Table>

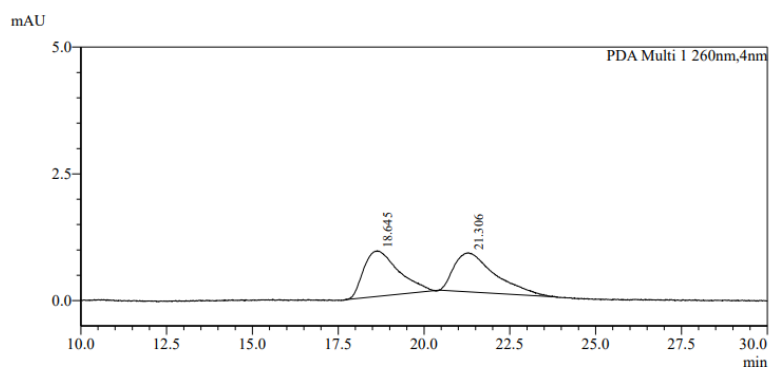
| PDA Ch1 222nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 41.724    | 4527678 | 65805  | 98.550 |
| 2             | 51.975    | 66607   | 949    | 1.450  |

**(*R*)-5-((([1,1'-biphenyl]-4-ylsulfonyl)methyl)-5-phenyldihydrofuran-2(3*H*)-one (3s)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). white solid, 38% yield, 92% ee.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) = 7.86 (d,  $J$  = 8.4 Hz, 2H), 7.69 (d,  $J$  = 8.4 Hz, 2H), 7.58 (d,  $J$  = 7.2 Hz, 2H), 7.50 – 7.40 (m, 3H), 7.33 – 7.27 (m, 5H), 3.85 – 3.77 (m, 2H), 3.40 – 3.33 (m, 1H), 2.91 – 2.83 (m, 1H), 2.70 – 2.63 (m, 1H), 2.55 – 2.47 (m, 1H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) = 175.4, 146.8, 141.7, 139.1, 138.9, 129.1, 128.9, 128.7, 128.6, 128.5, 127.9, 127.4, 124.6, 84.7, 65.2, 32.7, 28.3. HRMS (ESI) for  $\text{C}_{23}\text{H}_{20}\text{NaO}_4\text{S}^+$   $[\text{M}+\text{Na}]^+$  calcd. 415.0975, found: 415.0970.

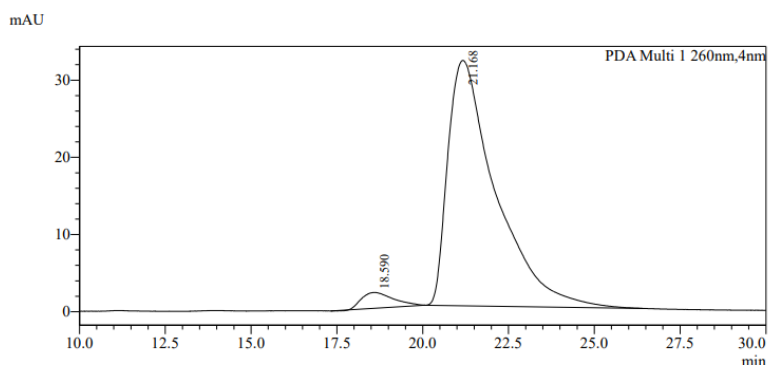
HPLC analysis: CHIRALPAK AD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 260 nm,  $t_R$  = 18.59 min (minor),  $t_R$  = 21.17 min (major).



<Peak Table>

PDA Ch1 260nm

| Peak# | Ret. Time | Area  | Height | Aera%  |
|-------|-----------|-------|--------|--------|
| 1     | 18.645    | 63158 | 898    | 49.715 |
| 2     | 21.306    | 63881 | 768    | 50.285 |

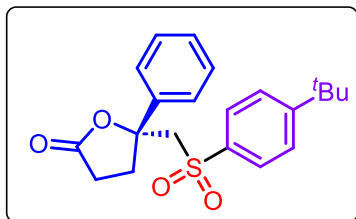


<Peak Table>

PDA Ch1 260nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 18.590    | 130679  | 2048   | 4.055  |
| 2     | 21.168    | 3091650 | 31791  | 95.945 |

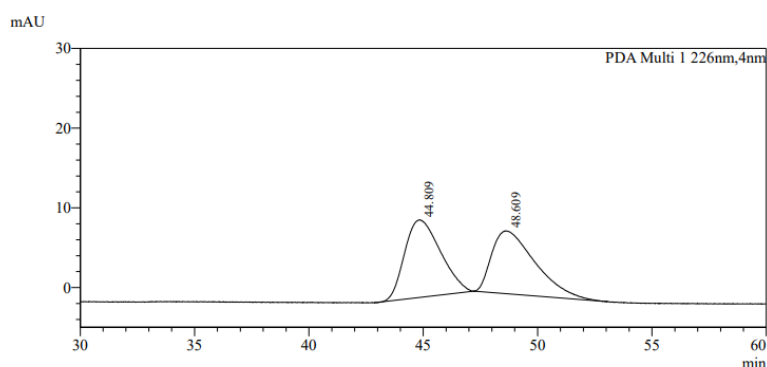
**(R)-5-(((4-(tert-butyl)phenyl)sulfonyl)methyl)-5-phenyldihydrofuran-2(3H)-one**  
**(3t)**<sup>[6]</sup>



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 85% yield, 96% ee.  $[\alpha]_{\text{D}}^{20} = +24.0$  (*c* = 0.1, CHCl<sub>3</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm) = 7.72 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.33 – 7.27 (m, 5H), 3.75 (t, *J* = 15.6 Hz, 2H), 3.40 – 3.32 (m, 1H), 2.92 – 3.84 (m, 1H), 2.70 – 2.63 (m, 1H), 2.55 – 2.47 (m, 1H), 1.33 (s, 9H). <sup>13</sup>C NMR (100

MHz, CDCl<sub>3</sub>)  $\delta$  (ppm) = 175.4, 157.8, 141.8, 137.3, 128.9, 128.5, 127.7, 126.3, 124.6, 84.8, 65.1, 35.3, 32.5, 31.0, 28.3.

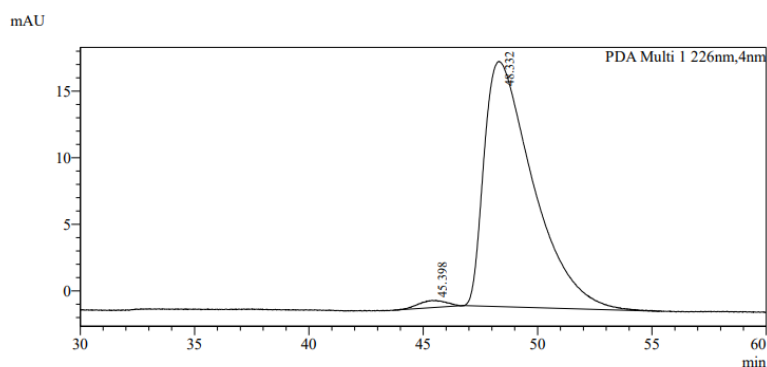
HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 95:5, flow rate = 1.0 mL/min, wave length = 226 nm, *t*<sub>R</sub> = 45.40 min (major), *t*<sub>R</sub> = 48.33 min (minor).



<Peak Table>

PDA Ch1 226nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 44.809    | 1059765 | 9729   | 50.349 |
| 2     | 48.609    | 1045064 | 7875   | 49.651 |

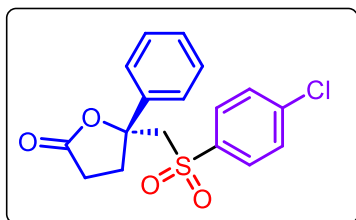


<Peak Table>

PDA Ch1 226nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 45.398    | 44593   | 543    | 1.599  |
| 2     | 48.332    | 2743749 | 18408  | 98.401 |

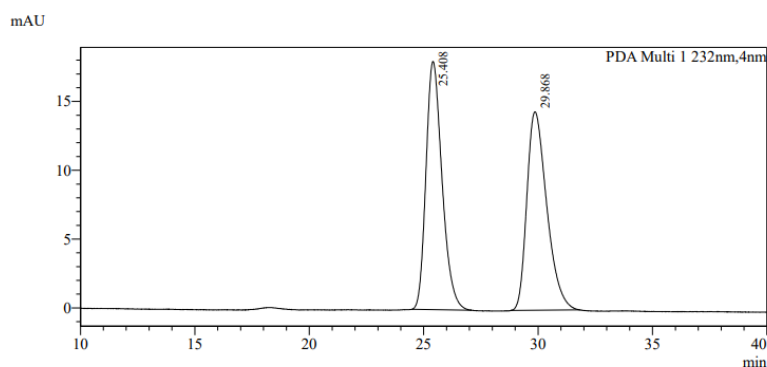
**(*R*)-5-(((4-chlorophenyl)sulfonyl)methyl)-5-phenyldihydrofuran-2(3*H*)-one (3u)**<sup>[6]</sup>



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 50% yield, 89% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  (ppm) = 7.74 (d, *J* = 8.8 Hz, 2H), 7.47 (d, *J* = 8.8 Hz, 2H), 7.36 – 7.27 (m, 5H), 3.77 (t, *J* = 15.6 Hz, 2H), 3.35 – 3.27 (m, 1H), 2.86 – 2.78 (m, 1H), 2.68 – 2.61 (m, 1H), 2.54 – 2.45 (m, 1H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**  $\delta$  (ppm) = 175.1, 141.5,

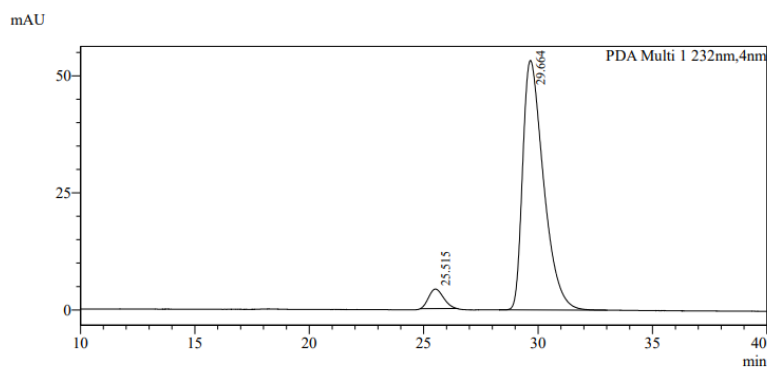
140.7, 138.9, 129.6, 129.5, 129.0, 128.7, 124.5, 84.4, 65.2, 32.8, 28.1.

HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 232 nm, *t<sub>R</sub>* = 25.52 min (minor), *t<sub>R</sub>* = 29.66 min (major).



<Peak Table>

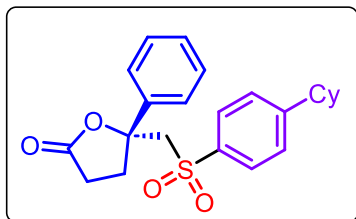
| PDA Ch1 232nm |           |        |        |        |
|---------------|-----------|--------|--------|--------|
| Peak#         | Ret. Time | Area   | Height | Aera%  |
| 1             | 25.408    | 881171 | 18037  | 50.188 |
| 2             | 29.868    | 874557 | 14421  | 49.812 |



<Peak Table>

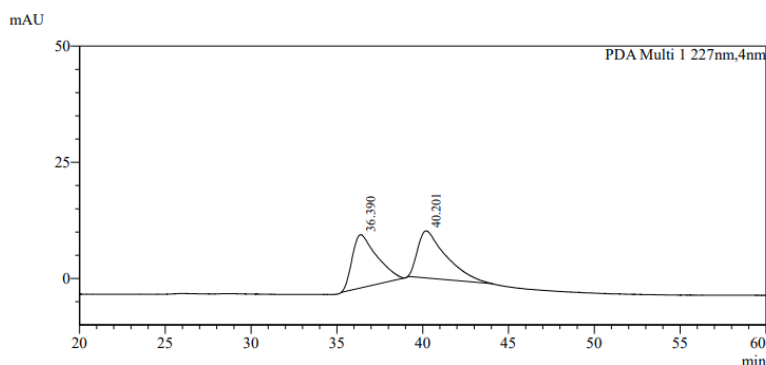
| PDA Ch1 232nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 25.515    | 187389  | 4177   | 5.241  |
| 2             | 29.664    | 3388277 | 53301  | 94.759 |

**(*R*)-5-(((4-cyclohexylphenyl)sulfonyl)methyl)-5-phenyldihydrofuran-2(3*H*)-one (3v)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 80% yield, 94% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.70 (d, *J* = 8.4 Hz, 2H), 7.32 – 7.27 (m, 7H), 3.74 (dd, *J* = 16.8, 14.8 Hz, 2H), 3.40 – 3.32 (m, 1H), 2.91 – 2.82 (m, 1H), 2.69 – 2.62 (m, 1H), 2.56 – 2.46 (m, 2H), 1.87 – 1.75 (m, 5H), 1.42 – 1.34 (m, 5H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

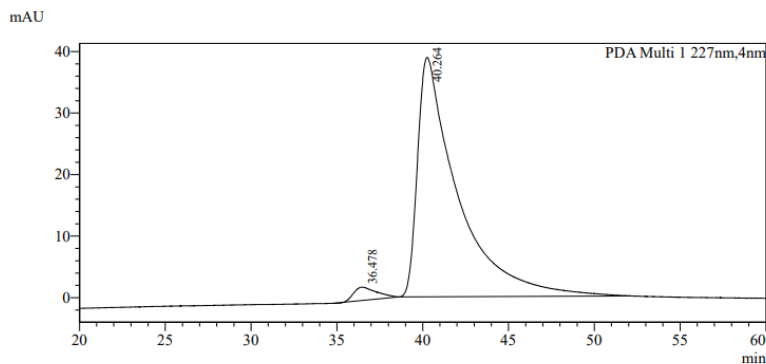
δ(ppm) = 175.3, 154.7, 141.8, 137.6, 128.9, 128.5, 127.9, 127.8, 124.6, 84.8, 65.1, 44.6, 34.0, 34.0, 32.4, 28.3, 26.6, 25.9. HRMS (ESI) for C<sub>23</sub>H<sub>26</sub>NaO<sub>4</sub>S<sup>+</sup> [M+Na]<sup>+</sup> calcd. 421.1450, found: 421.1443. HPLC analysis: CHIRALPAK AD-H (*n*-hexane/*i*-PrOH) = 92:8, flow rate = 0.8 mL/min, wave length = 227 nm, t<sub>R</sub> = 36.48 min (minor), t<sub>R</sub> = 40.26 min (major).



<Peak Table>

PDA Ch1 227nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 36.390    | 1139667 | 11450  | 49.271 |
| 2     | 40.201    | 1173390 | 10140  | 50.729 |

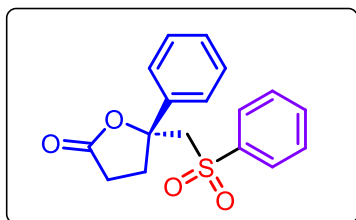


<Peak Table>

PDA Ch1 227nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 36.478    | 196825  | 2158   | 3.013  |
| 2     | 40.264    | 6336269 | 38896  | 96.987 |

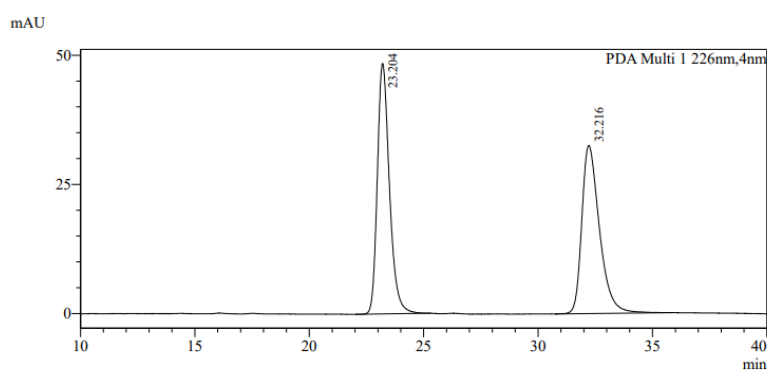
**(*R*)-5-phenyl-5-((phenylsulfonyl)methyl)dihydrofuran-2(3*H*)-one (3w)**<sup>[6]</sup>



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). white solid, 55% yield, 94% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.83 – 7.81 (m, 2H), 7.64 – 7.61 (m, 1H), 7.54 – 7.50 (m, 2H), 7.36 – 7.27 (m, 5H), 3.81 – 3.72 (m, 2H), 3.40 – 3.32 (m, 1H), 2.89 – 2.81 (m, 1H), 2.69 – 2.62 (m, 1H), 2.54 – 2.45 m, 1H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 175.3,

141.8 140.4, 133.9, 129.3 128.9, 128.6, 127.9, 124.6, 84.7, 65.0, 32.6, 28.2.

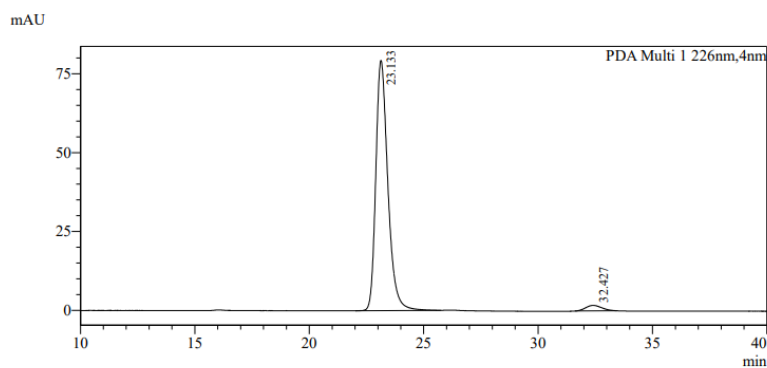
HPLC analysis: CHIRALPAK IA (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 226 nm, t<sub>R</sub> = 23.13 min (major), t<sub>R</sub> = 32.43 min (minor).



<Peak Table>

PDA Ch1 226nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 23.204    | 1782963 | 48495  | 50.473 |
| 2     | 32.216    | 1749578 | 32548  | 49.527 |

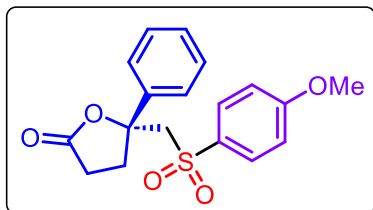


<Peak Table>

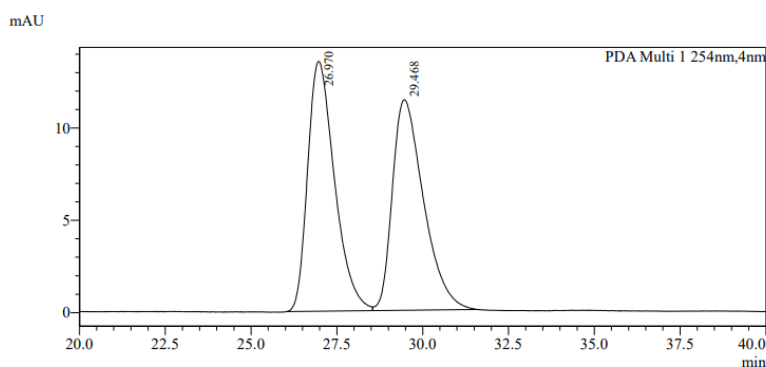
PDA Ch1 226nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 23.133    | 2907813 | 79329  | 97.156 |
| 2     | 32.427    | 85113   | 1744   | 2.844  |

**(*R*)-5-(((4-methoxyphenyl)sulfonyl)methyl)-5-phenyldihydrofuran-2(3*H*)-one**  
**(3x)<sup>[6]</sup>**

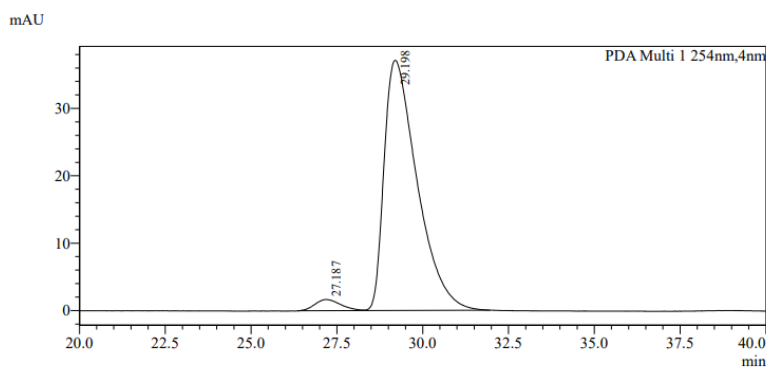


Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 73% yield, 93% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.74 (d, *J* = 8.8 Hz, 2H), 7.36 – 7.27 (m, 5H), 6.96 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 3H), 3.73 (dd, *J* = 16.8, 15.2 Hz, 2H), 3.41 – 3.33 (m, 1H), 2.89 – 2.81 (m, 1H), 2.66 – 2.56 (m, 1H), 2.54 – 2.45 (m, 1H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 175.4, 163.9, 142.0, 132.0, 130.2, 128.9, 128.5, 124.5, 114.4, 84.8, 65.3, 55.7, 32.5, 28.3. HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 254 nm, *t*<sub>R</sub> = 27.19 min (minor), *t*<sub>R</sub> = 29.20 min (major).



<Peak Table>

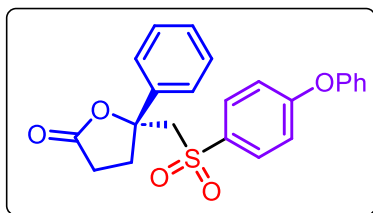
| PDA Ch1 254nm |           |        |        |        |
|---------------|-----------|--------|--------|--------|
| Peak#         | Ret. Time | Area   | Height | Aera%  |
| 1             | 26.970    | 739108 | 13535  | 50.579 |
| 2             | 29.468    | 722194 | 11408  | 49.421 |



<Peak Table>

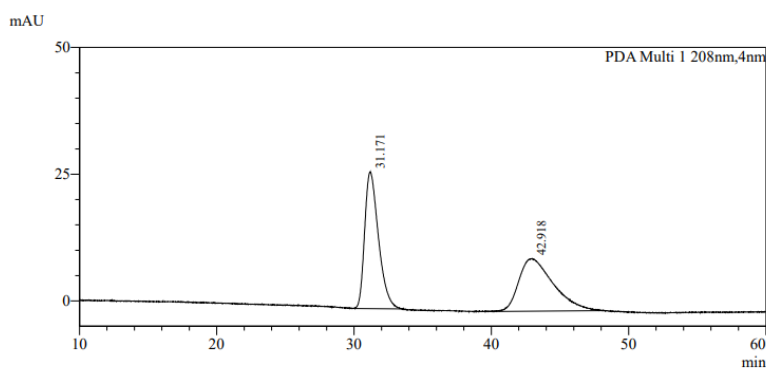
| PDA Ch1 254nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 27.187    | 87311   | 1663   | 3.395  |
| 2             | 29.198    | 2484195 | 37121  | 96.605 |

**(R)-5-(((4-phenoxyphenyl)sulfonyl)methyl)-5-phenyldihydrofuran-2(3H)-one (3y)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 71% yield, 93% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.74 (dd, *J* = 6.8, 2.0 Hz, 2H), 7.44 – 7.40 (m, 2H), 7.37 – 7.29 (m, 5H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.08 – 7.06 (m, 2H), 7.00 (dd, *J* = 2.8, 2.0 Hz, 2H), 3.75 (dd, *J* = 17.6, 15.2 Hz, 2H), 3.39 – 3.31 (m, 1H), 2.88 – 2.80 (m, 1H), 2.68 – 2.61 (m, 1H), 2.54 – 2.45 (m, 1H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 175.3, 162.7, 154.8, 141.8, 133.7, 130.3, 130.3, 128.9, 128.6, 125.2, 124.6, 120.5, 117.6, 84.7, 65.3, 32.6, 28.3. HRMS (ESI) for C<sub>23</sub>H<sub>20</sub>NaO<sub>5</sub>S<sup>+</sup> [M+Na]<sup>+</sup> calcd. 431.0924, found: 431.0922.

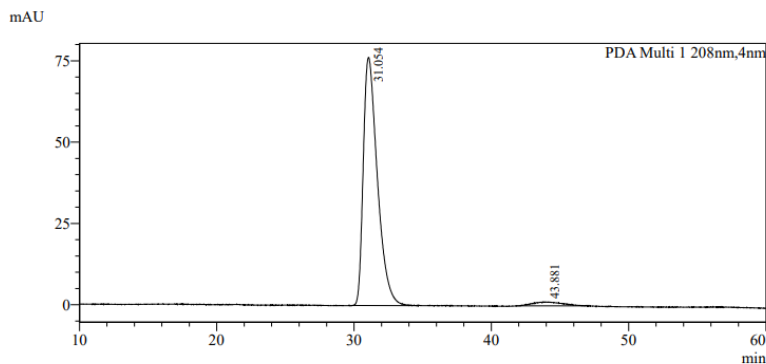
HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 208 nm, t<sub>R</sub> = 25.604 min (major), t<sub>R</sub> = 35.861 min (minor).



<Peak Table>

PDA Ch1 208nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 31.171    | 1884801 | 26943  | 50.982 |
| 2     | 42.918    | 1812177 | 10348  | 49.018 |

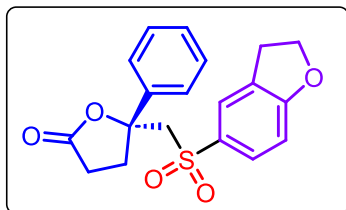


<Peak Table>

PDA Ch1 208nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 31.054    | 5447499 | 76290  | 96.827 |
| 2     | 43.881    | 178497  | 1179   | 3.173  |

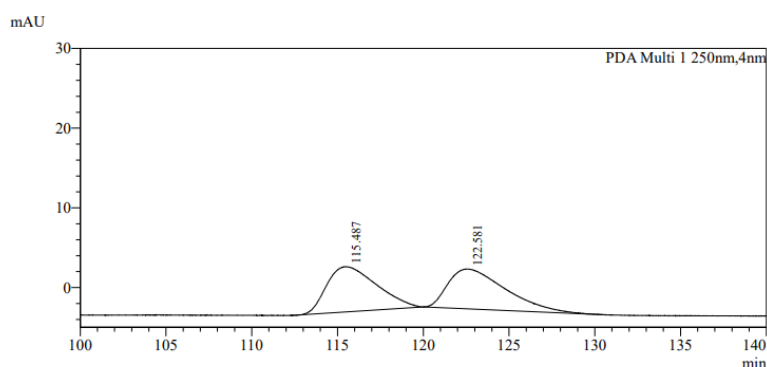
**(*R*)-5-(((2,3-dihydrobenzofuran-5-yl)sulfonyl)methyl)-5-phenyldihydrofuran-2(3H)-one (3z)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 85% yield, 98% ee.  $[\alpha]_{\text{D}}^{20} = +10.3$  ( $c = 0.12$ ,  $\text{CHCl}_3$ ).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) = 7.60 – 7.58 (m, 2H), 7.36 – 7.27 (m, 5H), 6.81 (d,  $J = 8.0$  Hz, 1H), 4.67 (t,  $J = 9.2$  Hz, 2H), 3.72 (dd,  $J = 16.8, 15.2$  Hz, 2H), 3.40 – 3.19 (m, 3H), 2.89 – 2.81 (m, 1H), 2.67 – 2.60 (m, 1H), 2.54 – 2.45 (m, 1H).

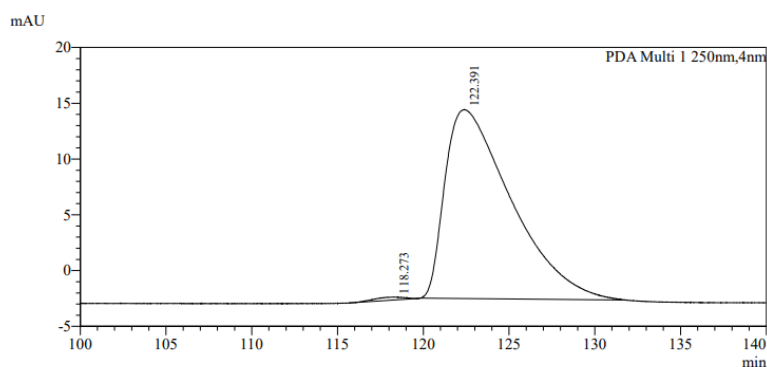
$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) = 175.4, 164.8, 142.0, 133.0, 129.7, 128.8, 128.6, 128.5, 125.3, 124.6, 109.7, 84.8, 72.5, 65.4, 32.4, 28.9, 28.3. HRMS (ESI) for  $\text{C}_{19}\text{H}_{18}\text{NaO}_5\text{S}^+$   $[\text{M}+\text{Na}]^+$  calcd. 381.0773, found: 381.0763.

HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 90:10, flow rate = 0.6 mL/min, wave length = 254 nm,  $t_{\text{R}} = 71.87$  min (minor),  $t_{\text{R}} = 74.29$  min (major).



<Peak Table>

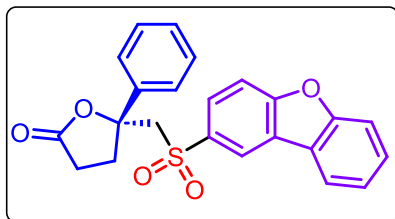
| PDA Ch1 250nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 115.487   | 1142531 | 5671   | 49.345 |
| 2             | 122.581   | 1172844 | 5002   | 50.655 |



<Peak Table>

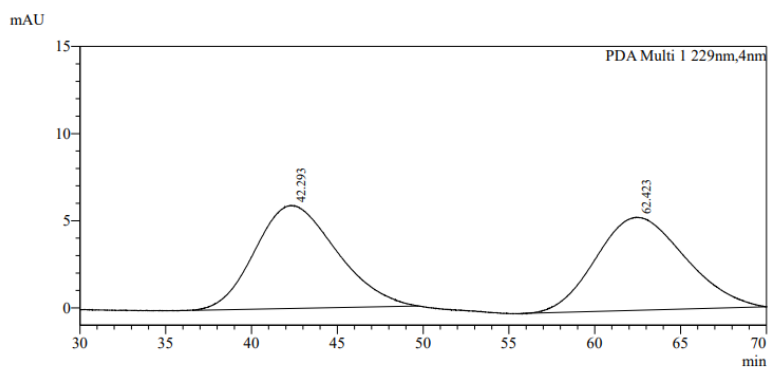
| PDA Ch1 250nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 118.273   | 34352   | 267    | 0.747  |
| 2             | 122.391   | 4566226 | 16934  | 99.253 |

**(*R*)-5-((dibenzo[*b,d*]furan-2-ylsulfonyl)methyl)-5-phenyldihydrofuran-2(3*H*)-one (3aa)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 55% yield, 89% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 8.38 (d, *J* = 2.0 Hz, 1H), 8.07 – 7.97 (m, 1H), 7.92 – 7.80 (m, 1H), 7.65 – 7.61 (m, 2H), 7.58 – 7.53 (m, 1H), 7.45 – 7.41 (m, 1H), 7.32 – 7.25 (m, 4H), 7.24 – 7.19 (m, 1H), 3.90 – 3.81 (m, 2H), 3.41 – 3.34 (m, 1H), 2.93 – 2.84 (m, 1H), 2.72 – 2.65 (m, 1H), 2.57 – 2.48 (m, 1H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 175.3, 158.8, 157.1, 141.5, 134.9, 128.8, 128.7, 128.6, 126.9, 125.1, 124.7, 123.8, 122.9, 121.9, 121.3, 112.5, 112.1, 84.7, 65.6, 32.8, 28.3. HRMS (ESI) for C<sub>23</sub>H<sub>19</sub>O<sub>5</sub>S<sup>+</sup> [M+H]<sup>+</sup> calcd. 429.0773, found: 429.0768.

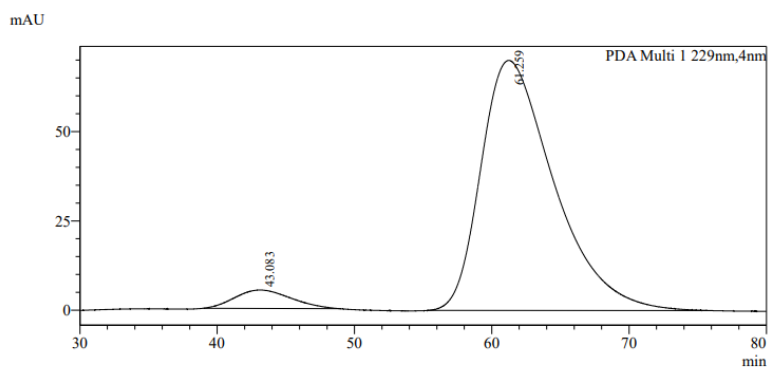
HPLC analysis: CHIRALPAK OZ-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 229 nm, *t*<sub>R</sub> = 43.08 min (minor), *t*<sub>R</sub> = 61.26 min (major).



<Peak Table>

PDA Ch1 229nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 42.293    | 1959127 | 5905   | 50.406 |
| 2     | 62.423    | 1927576 | 5340   | 49.594 |

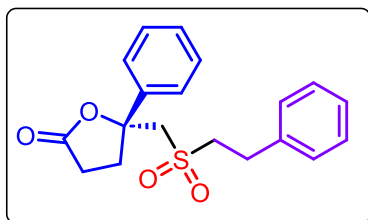


<Peak Table>

PDA Ch1 229nm

| Peak# | Ret. Time | Area     | Height | Aera%  |
|-------|-----------|----------|--------|--------|
| 1     | 43.083    | 1463099  | 5164   | 5.349  |
| 2     | 61.259    | 25891283 | 69998  | 94.651 |

**(*R*)-5-((phenethylsulfonyl)methyl)-5-phenyldihydrofuran-2(3*H*)-one (3ab)**

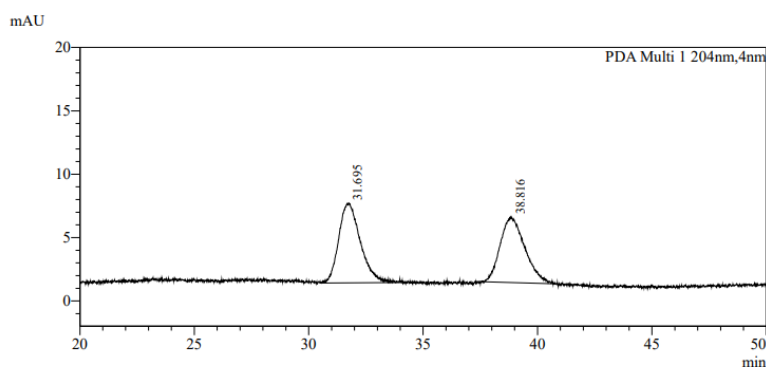


Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow oil, 68% yield, 95% ee.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) = 7.42 – 7.28 (m, 7H), 7.24 – 7.18 (m, 3H), 3.53 – 3.44 (m, 2H), 3.26 – 3.18 (m, 3H), 3.10 – 3.05 (m, 2H), 2.78 – 2.70 (m, 1H), 2.64 – 2.58 (m, 1H), 2.52 – 2.43 (m, 1H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) = 175.2, 141.5, 137.2,

129.2, 128.9, 128.8, 128.6, 127.1, 124.5, 84.2, 61.9, 57.0, 32.8, 28.2, 27.9. HRMS (ESI) for  $\text{C}_{19}\text{H}_{20}\text{NaO}_4\text{S}^+$   $[\text{M}+\text{Na}]^+$  calcd. 367.0980, found: 367.0972.

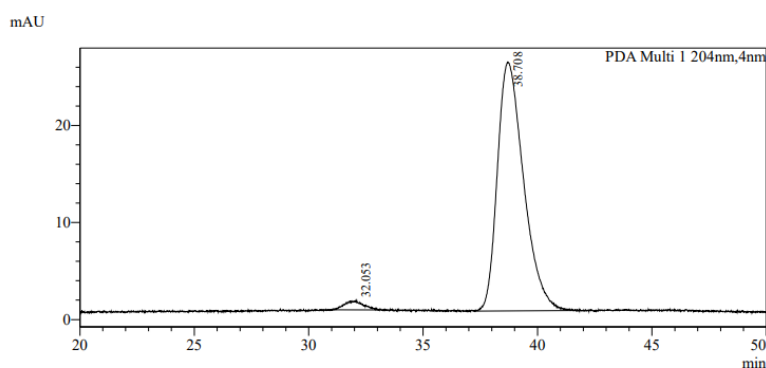
HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 204 nm,  $t_R$  = 32.50 min (minor),  $t_R$  = 38.71 min (major).



<Peak Table>

PDA Ch1 204nm

| Peak# | Ret. Time | Area   | Height | Aera%  |
|-------|-----------|--------|--------|--------|
| 1     | 31.695    | 414354 | 6294   | 51.796 |
| 2     | 38.816    | 385621 | 5151   | 48.204 |

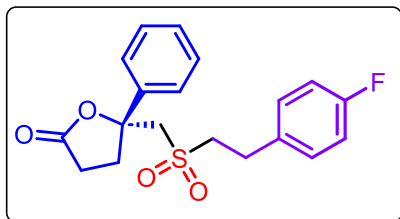


<Peak Table>

PDA Ch1 204nm

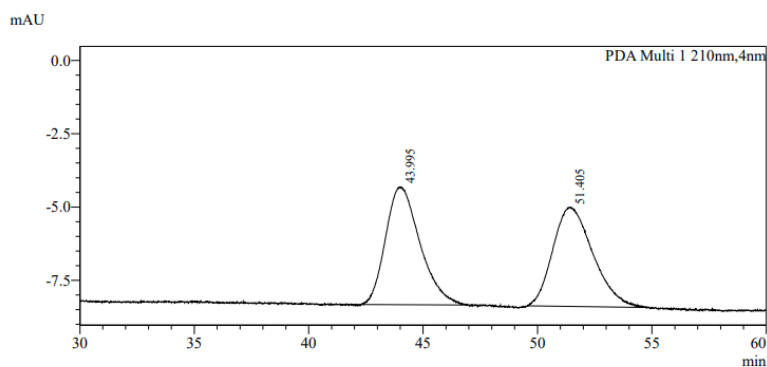
| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 32.053    | 54123   | 947    | 2.578  |
| 2     | 38.708    | 2045278 | 25627  | 97.422 |

**(*R*)-5-(((4-fluorophenethyl)sulfonyl)methyl)-5-phenyldihydrofuran-2(3*H*)-one (3ac)**



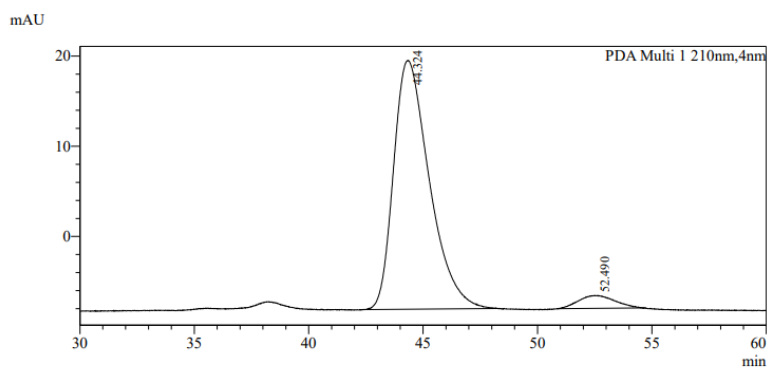
Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow oil, 45% yield, 90% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  (ppm) = 7.45 – 7.34 (m, 5H), 7.18 – 7.14 (m, 2H), 7.01 – 6.96 (m, 2H), 3.50 (dd, *J* = 18.0, 15.6 Hz, 2H), 3.28 – 3.19 (m, 3H), 3.08 – 3.06 (m, 2H), 2.78 – 2.70 (m, 1H), 2.64 – 2.58 (m, 1H), 2.53 – 2.44 (m, 1H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**  $\delta$  (ppm) = 175.2, 163.1, 141.5, 132.9 (d, *J* = 2.0 Hz), 130.1 (d, *J* = 8.0 Hz), 129.2, 128.9, 124.4, 115.8 (d, *J* = 21 Hz), 84.2, 61.9, 57.0, 32.9, 27.9, 27.4. **<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)**  $\delta$  –116.78 – –116.85 (m). HRMS (ESI) for C<sub>19</sub>H<sub>20</sub>FO<sub>4</sub>S<sup>+</sup> [M+H]<sup>+</sup> calcd. 385.0886, found: 385.0877.

HPLC analysis: CHIRALPAK OD-H (*n*-hexane/*i*-PrOH) = 90:10, flow rate = 1.0 mL/min, wave length = 210 nm, *t<sub>R</sub>* = 44.32 min (major), *t<sub>R</sub>* = 52.49 min (minor).



<Peak Table>

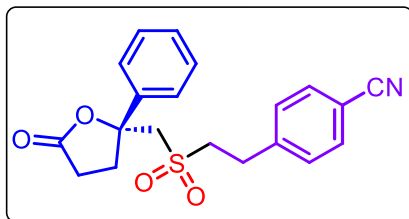
| PDA Ch1 210nm |           |        |        |        |
|---------------|-----------|--------|--------|--------|
| Peak#         | Ret. Time | Area   | Height | Aera%  |
| 1             | 43.995    | 419852 | 4021   | 50.437 |
| 2             | 51.405    | 412573 | 3380   | 49.563 |



<Peak Table>

| PDA Ch1 210nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 44.324    | 2999390 | 27600  | 94.952 |
| 2             | 52.490    | 159464  | 1442   | 5.048  |

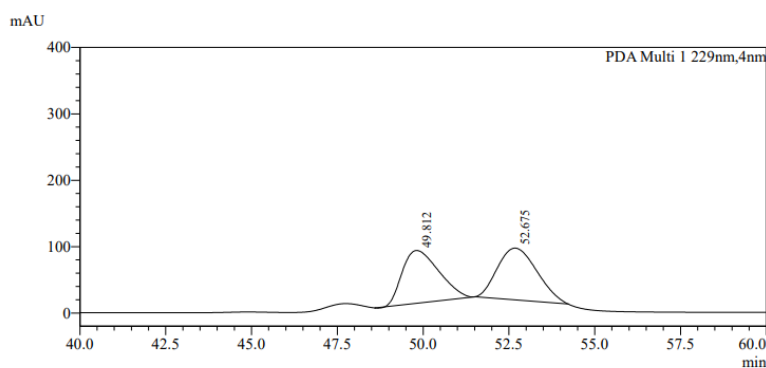
**(*R*)-4-(2-(((5-oxo-2-phenyltetrahydrofuran-2-yl)methyl)sulfonyl)ethyl)benzonitrile (3ad)**



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow oil, 71% yield, 89% ee. **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ (ppm) = 7.60 (d, *J* = 8.4 Hz, 2H), 7.46 – 7.32 (m, 7H), 3.55 (s, 2H), 3.34 – 3.13 (m, 5H), 2.78 – 2.70 (m, 1H), 2.64 – 2.45 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)** δ (ppm) = 175.2, 142.8, 141.4, 132.7, 129.5, 129.3, 129.0,

124.3, 118.6, 111.2, 84.1, 62.1, 56.2, 33.0, 28.1, 27.8. HRMS (ESI) for C<sub>20</sub>H<sub>19</sub>NNaO<sub>4</sub>S<sup>+</sup> [M+Na]<sup>+</sup> calcd. 392.0933, found: 392.0934.

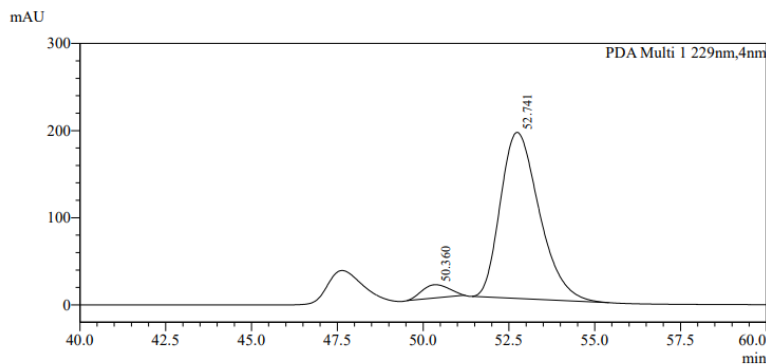
HPLC analysis: CHIRALPAK IA (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 229 nm, t<sub>R</sub> = 50.36 min (minor), t<sub>R</sub> = 52.74 min (major).



<Peak Table>

PDA Ch1 229nm

| Peak# | Ret. Time | Area    | Height | Aera%  |
|-------|-----------|---------|--------|--------|
| 1     | 49.812    | 5875793 | 79508  | 48.423 |
| 2     | 52.675    | 6258495 | 77957  | 51.577 |

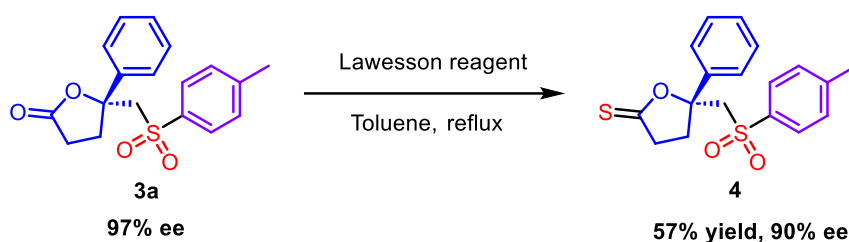


<Peak Table>

PDA Ch1 229nm

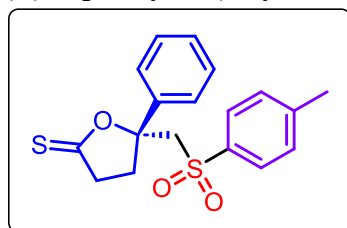
| Peak# | Ret. Time | Area     | Height | Aera%  |
|-------|-----------|----------|--------|--------|
| 1     | 50.360    | 839460   | 15175  | 5.281  |
| 2     | 52.741    | 15055701 | 190541 | 94.719 |

## 5.Transformations of 3a



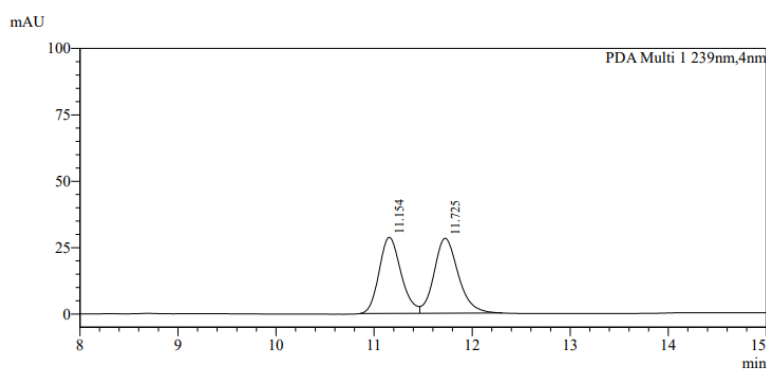
A solution of **3a** (66.1 mg, 0.2 mmol) and Lawesson reagent (181.8 mg, 0.45 mmol) in toluene (2.0 mL) was stirred and refluxed. After 12 h, the mixture was concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel to afford the corresponding product **4** as a white solid in 57% yield (39.5 mg).

### (*R*)-5-phenyl-5-(tosylmethyl)dihydrofuran-2(3*H*)-thione (**4**)



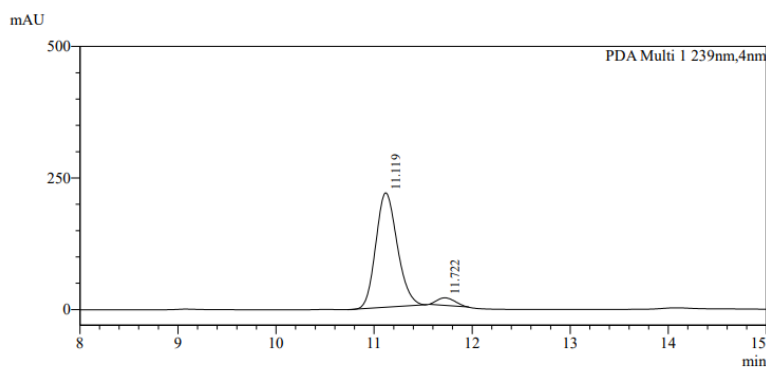
Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). yellow solid, 57% yield, 90% ee. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm) = 7.69 (d, *J* = 8.0 Hz, 2H), 7.32 – 7.28 (m, 7H), 3.89 – 3.80 (m, 2H), 3.41 – 3.28 (m, 2H), 2.97 – 2.88 (m, 1H), 2.73 – 2.68 (m, 1H), 2.42 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm) = 145.1, 140.4, 137.2, 129.9, 129.0, 128.8, 128.0, 124.8, 94.5, 64.3, 44.0, 33.8, 21.7. HRMS (ESI) for C<sub>18</sub>H<sub>18</sub>NaO<sub>3</sub>S<sub>2</sub><sup>+</sup> [M+Na]<sup>+</sup> calcd. 369.0590, found: 369.0588.

HPLC analysis: CHIRALPAK IA (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 239 nm, *t*<sub>R</sub> = 30.745 min (major), *t*<sub>R</sub> = 33.818 min (minor).



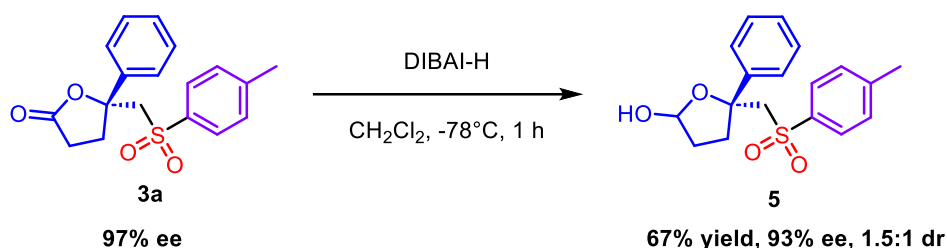
<Peak Table>

| PDA Ch1 239nm |           |        |        |        |
|---------------|-----------|--------|--------|--------|
| Peak#         | Ret. Time | Area   | Height | Acr%   |
| 1             | 11.154    | 445502 | 28665  | 48.055 |
| 2             | 11.725    | 481565 | 28240  | 51.945 |



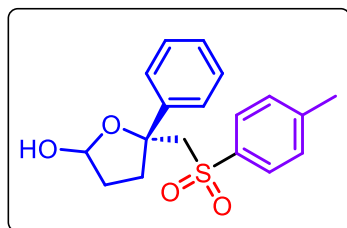
<Peak Table>

| PDA Ch1 239nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 11.119    | 3310263 | 217478 | 94.854 |
| 2             | 11.722    | 179603  | 14248  | 5.146  |



To a solution of **3a** (66.1 mg, 0.2 mmol) in  $\text{CH}_2\text{Cl}_2$  (1 mL) was added a solution of DIBAL-H (1M in toluene, 0.2 mL, 0.22 mmol) at  $-78^\circ\text{C}$  and the mixture was stirred for 1 hour. The reaction was quenched by addition of  $\text{H}_2\text{O}$  (0.4 mL) and NaOH (0.4 mL, 2M), and then the aqueous layer was extracted with  $\text{CH}_2\text{Cl}_2$  ( $3 \times 20$  mL). The combined organic phase was dried over  $\text{Na}_2\text{SO}_4$  and concentrated in vacuo. The residue was purified by flash chromatography on silica gel to afford the corresponding product **5** as a white solid in 67% yield (44.5 mg).

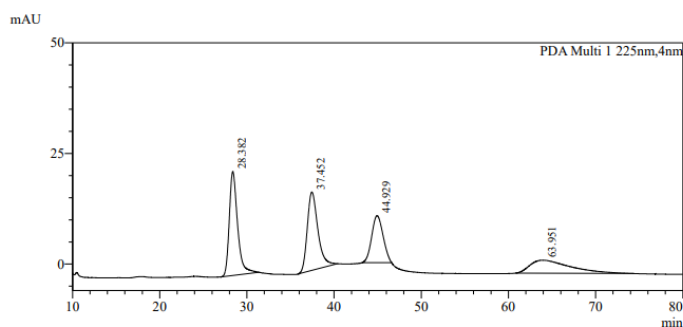
#### (5R)-5-phenyl-5-(tosylmethyl)tetrahydrofuran-2-ol (**5a**)



Purification by flash chromatography (*n*-hexane/ethyl acetate = 4/1). white solid, 67% yield, 93% ee, 1.5:1 dr.  **$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )**  $\delta$  (ppm) = 7.69 (d,  $J$  = 8.0 Hz, 2H), 7.39 – 7.19 (m, 7H), 5.65 (s, 1H), 4.01 (d,  $J$  = 6.8 Hz, 1H), 3.79 (s, 2H), 3.03 – 2.90 (m, 1H), 2.40 (s, 3H), 2.29 – 2.24 (m, 1H), 2.04 – 1.99 (m, 1H), 1.90 – 1.84 (m, 1H).  **$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )**  $\delta$  (ppm) = 144.5, 144.0, 138.1, 129.6, 128.3, 128.0, 127.4, 124.9, 100.1, 84.8, 66.2,

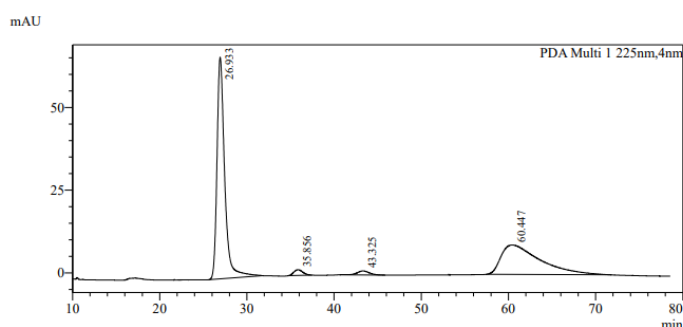
35.4, 33.1, 21.6. HRMS (ESI) for  $\text{C}_{18}\text{H}_{20}\text{NaO}_4\text{S}^+$   $[\text{M}+\text{Na}]^+$  calcd. 355.0975, found: 355.0971.

HPLC analysis: CHIRALPAK OJ-H (*n*-hexane/*i*-PrOH) = 80:20, flow rate = 1.0 mL/min, wave length = 225 nm,  $t_{\text{R}1}$  = 26.93 min (major),  $t_{\text{R}1}$  = 35.86 min (minor);  $t_{\text{R}2}$  = 43.33 min (minor),  $t_{\text{R}2}$  = 60.45 min (major).



<Peak Table>

| PDA Ch1 225nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 28.382    | 1552352 | 23482  | 31.257 |
| 2             | 37.452    | 1501594 | 17705  | 30.235 |
| 3             | 44.929    | 965925  | 10636  | 19.449 |
| 4             | 63.951    | 946599  | 2940   | 19.060 |

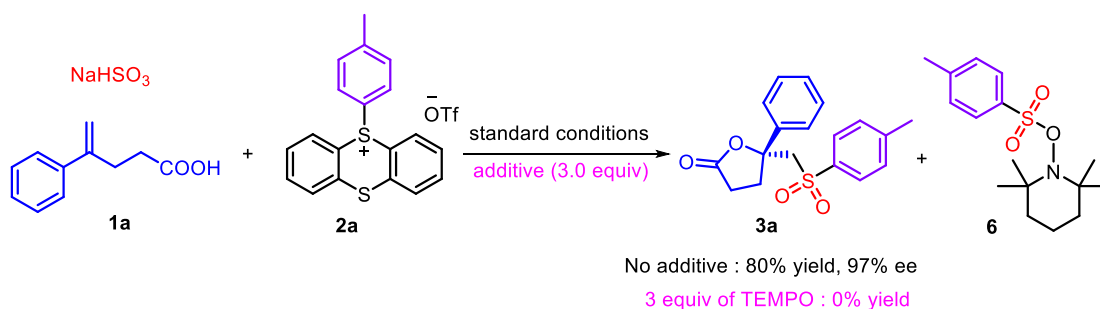


<Peak Table>

| PDA Ch1 225nm |           |         |        |        |
|---------------|-----------|---------|--------|--------|
| Peak#         | Ret. Time | Area    | Height | Aera%  |
| 1             | 26.933    | 4092302 | 66928  | 57.695 |
| 2             | 35.856    | 122282  | 1729   | 1.724  |
| 3             | 43.325    | 114978  | 1196   | 1.621  |
| 4             | 60.447    | 2763445 | 8880   | 38.960 |

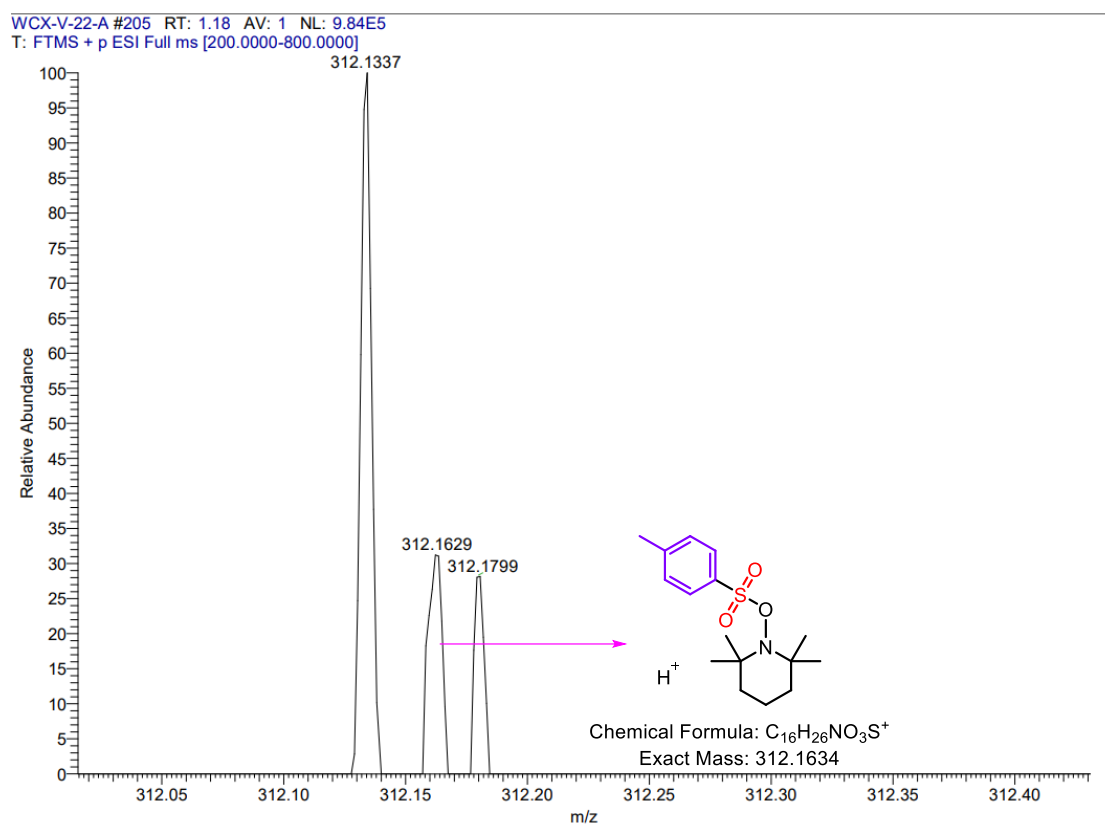
## 6. Mechanistic studies

### a) Radical trapping experiment with TEMPO.



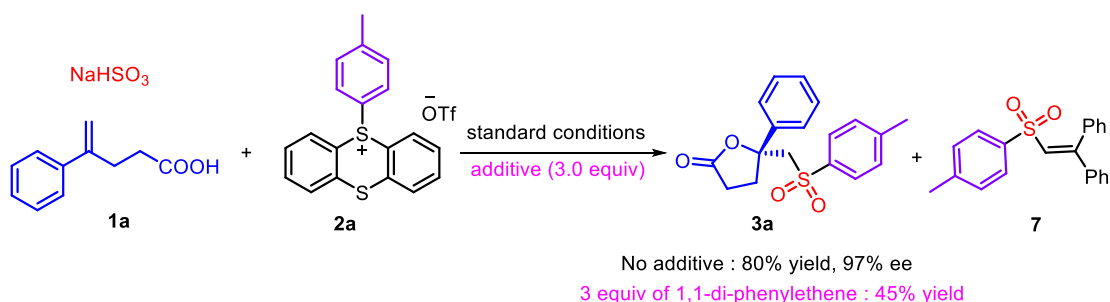
In a screw-capped vial,  $\text{Cu}(\text{MeCN})_4\text{BF}_4$  (3.2 mg, 10 mol %) and **L17** (8.7 mg, 12 mol %) were combined with EtOAc (2 mL) under  $\text{N}_2$  atmosphere. The resultant solution was stirred for 30 min at room temperature. Enoic acid **1** (0.1 mmol, 1.0 equiv), thianthrenium salt **2** (0.15 mmol, 1.5

equiv), NaHSO<sub>3</sub> (15.6 mg, 0.15 mmol, 1.5 equiv), TEMPO (3.0 equiv), pyrene (5 mol %) and 2,6-di-*tert*-butylpyridine (0.2 mmol, 2.0 equiv) were added under N<sub>2</sub> atmosphere. The reaction was placed 4 cm away from a 390 nm Kessil lamp and irradiated for 36 h while stirring at 900 rpm. TLC and HRMS analysis demonstrated that product **3a** was not founded. Compound **6** was detected by HRMS.



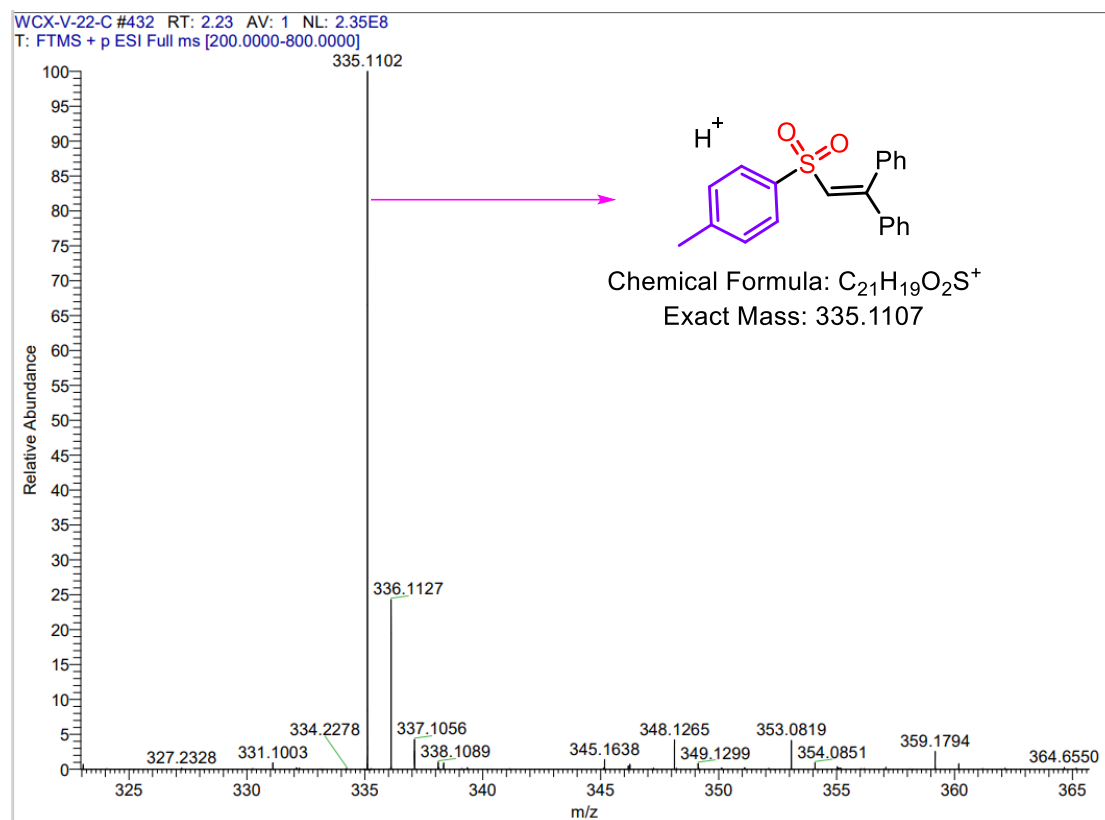
**Figure S1** Radical trapping experiment with TEMPO

**b) Radical trapping experiment with 1,1-diphenylethylene.**



In a screw-capped vial, Cu(MeCN)<sub>4</sub>BF<sub>4</sub> (3.2 mg, 10 mol %) and **L17** (8.7 mg, 12 mol %) were combined with EtOAc (2 mL) under N<sub>2</sub> atmosphere. The resultant solution was stirred for 30 min at room temperature. Enoic acid **1** (0.1 mmol, 1.0 equiv), thianthrenium salt **2** (0.15 mmol, 1.5 equiv), NaHSO<sub>3</sub> (15.6 mg, 0.15 mmol, 1.5 equiv), 1,1-di-phenylethene (0.3 mmol, 3 equiv), pyrene

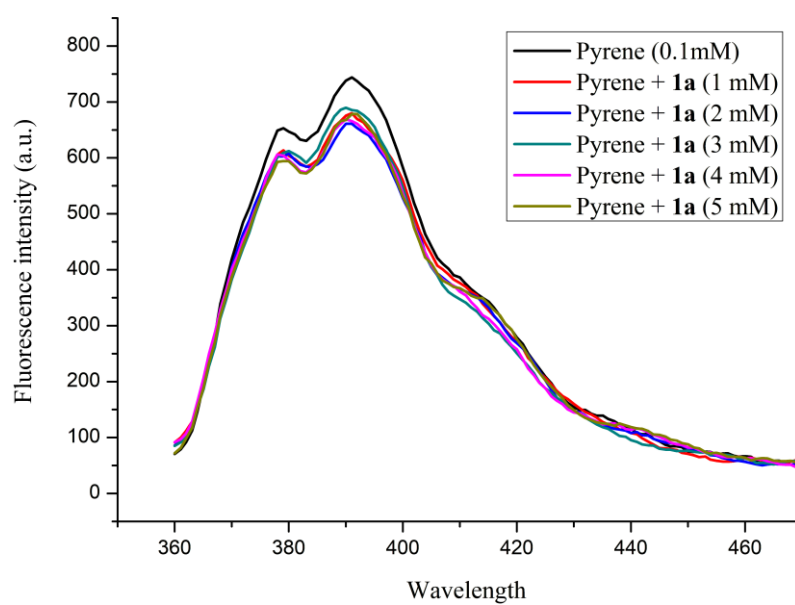
(5 mol %) and 2,6-di-*tert*-butylpyridine (0.2 mmol, 2.0 equiv) were added under N<sub>2</sub> atmosphere. The reaction was placed 4 cm away from a 390 nm Kessil lamp and irradiated for 36 h while stirring at 900 rpm. Product **3a** was obtained in 45% yield. Compound **7** was detected by HRMS.



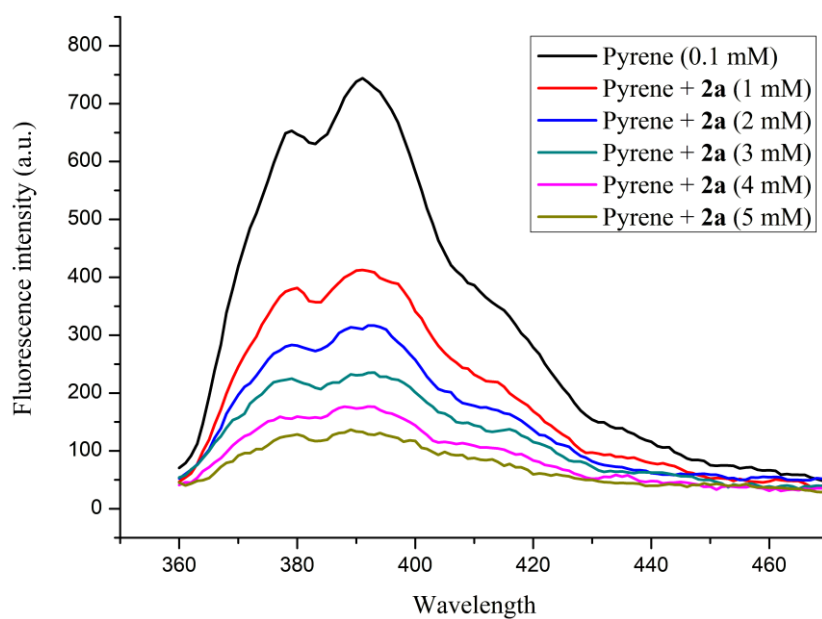
**Figure S2** Radical trapping experiment with 1,1-diphenylethylene.

**c) Stern-Volmer fluorescence quenching experiments.**

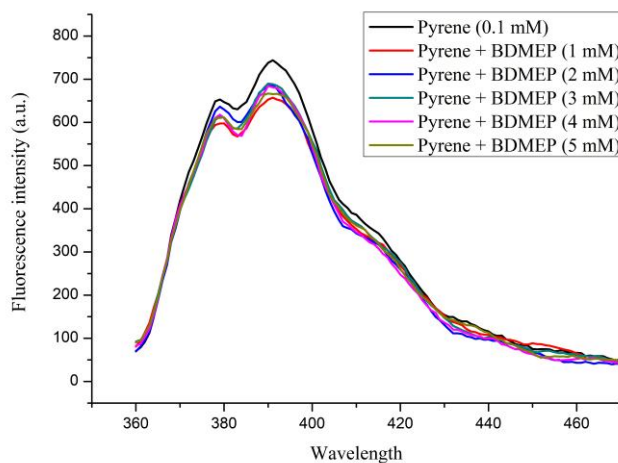
Stern-Volmer fluorescence quenching experiments were run with freshly prepared solutions of 0.1 mM pyrene in degassed dry CH<sub>3</sub>CN added with the appropriate amount of a quencher in a screw-top quartz cuvette at room temperature. The solutions were irradiated at 345 nm and fluorescence was measured from 360 nm to 420 nm.



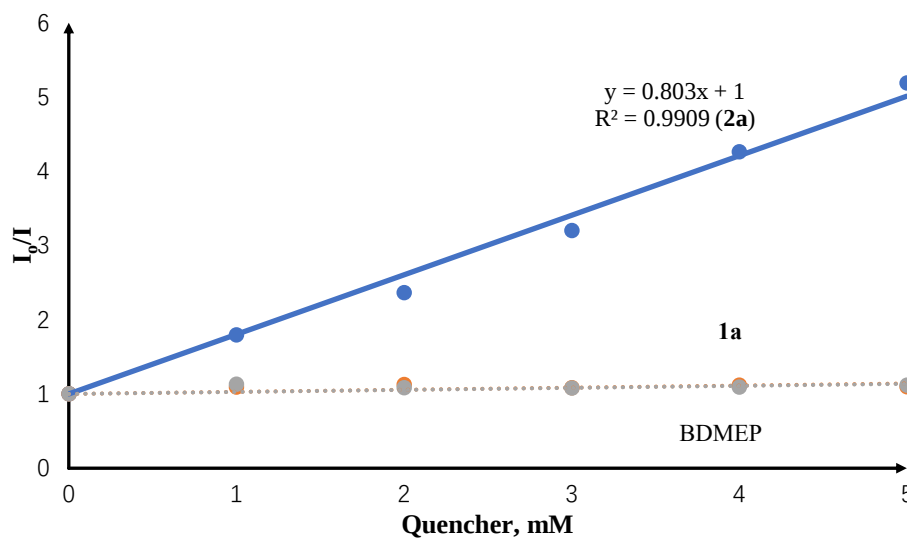
**Figure S3** Fluorescence quenching experiments of pyrene and **1a**.



**Figure S4** Fluorescence quenching experiments of pyrene and **2a**.



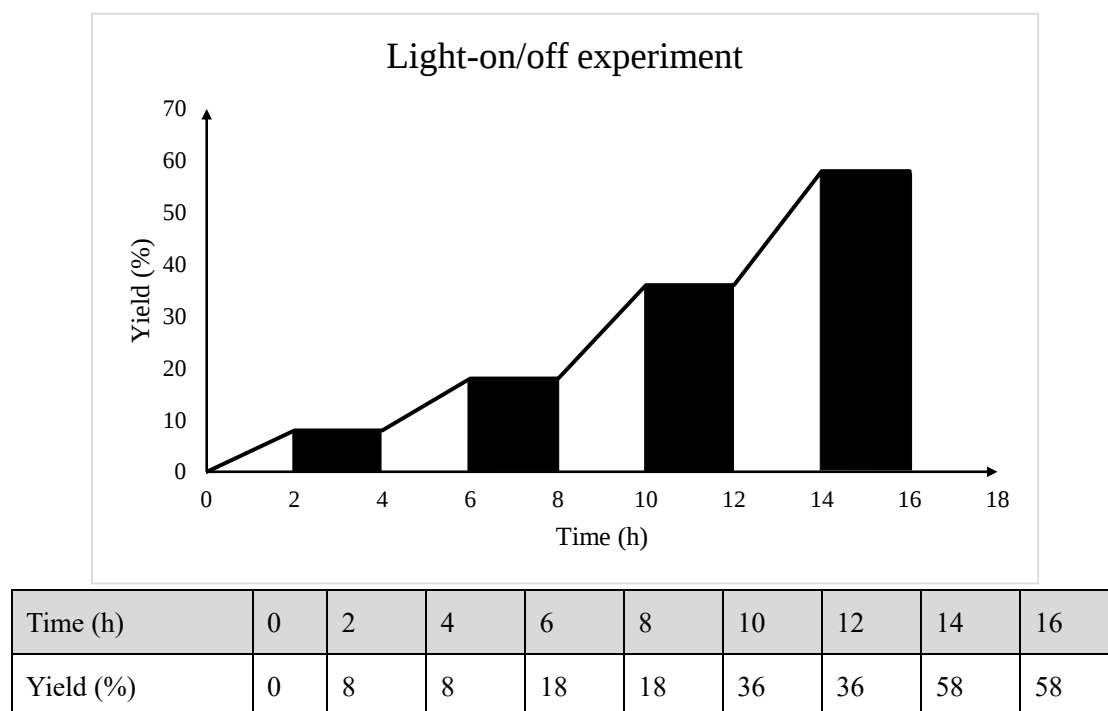
**Figure S5** Fluorescence quenching experiments of pyrene and BDMEP.



**Figure S6** Stern-Volmer plots of pyrene with different quenchers.

#### ***d) light on-off experiments***

To further study the necessity of continuous irradiation with visible light for the progress of the reaction, the reaction proceeding was monitored by  $^1\text{H}$  NMR spectroscopy using dibromomethane as an internal standard before and after light irradiation and dark periods. The control experiments shown below with successive intervals of irradiation and dark periods resulted in total interruption of the reaction progress in the absence of light, and recuperation of reactivity upon further illumination, demonstrating that light is a necessary component for the reaction proceeding.

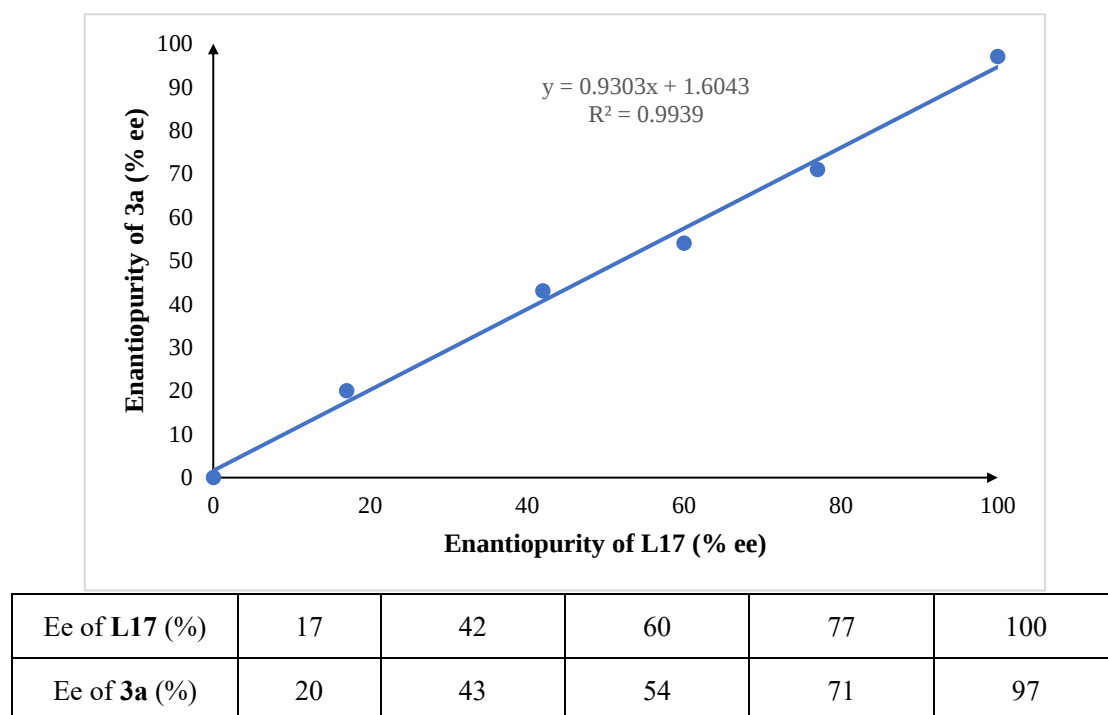


**Figure S7** Light-on/off experiment.

***e) Non-linear effect studies***

Investigation into non-linear effects was conducted by comparing the ee value of the chiral ligand **L17** and that of the product **3a**. 6 reactions containing **L17** of racemic, 17%, 42%, 60%, 77%, and > 99% optical purity were run in parallel. The nonlinear effect study revealed a linear relationship between the ee of the product **3a** and the enantiopurity of the ligand **L17**, indicating a single chiral ligand is likely involved in the enantiodetermining transition state.

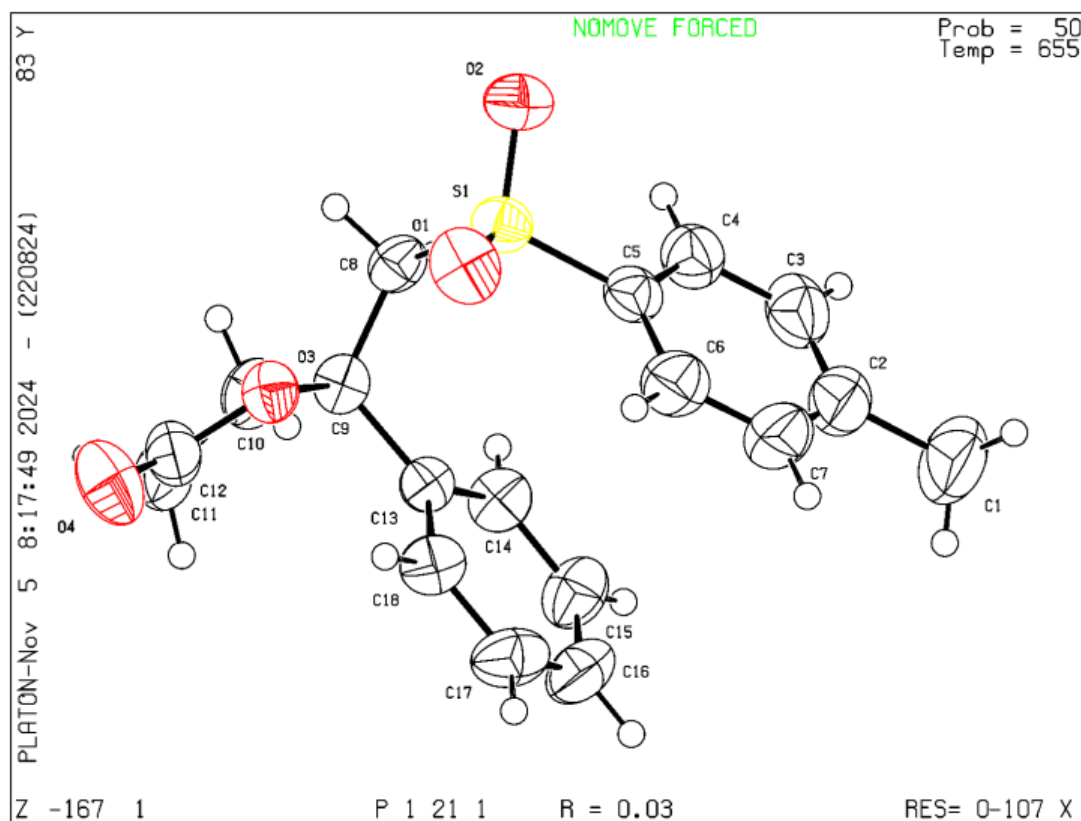
In a screw-capped vial, Cu(MeCN)<sub>4</sub>BF<sub>4</sub> (3.2 mg, 10 mol %) and **L17** with different ee values (8.7 mg, 12 mol %) were combined with EtOAc (2 mL) under N<sub>2</sub> atmosphere. The resultant solution was stirred for 30 min at room temperature. Enoic acid **1** (0.1 mmol, 1.0 equiv), thianthrenium salt **2** (0.15 mmol, 1.5 equiv), NaHSO<sub>3</sub> (15.6 mg, 0.15 mmol, 1.5 equiv), pyrene (5 mol %) and 2,6-di-*tert*-butylpyridine (0.2 mmol, 2.0 equiv) were added under N<sub>2</sub> atmosphere. The reaction was placed 4 cm away from a 390 nm Kessil lamp and irradiated for 36 h while stirring at 900 rpm. Then the mixture was purified directly by flash column chromatography (*n*-hexane/EtOAc (v/v): 4/1) to provide the desired product **3a**. The ee of **3a** was determined by HPLC analysis: (Chiralcel AD-H, 0.5 mL/min, *n*-hexane/*i*-PrOH = 80:20, 226 nm, t<sub>R</sub> = 34.56 min (minor), t<sub>R</sub> = 37.76 min (major)). A graph of ee of product vs. ee of ligand was then plotted.



**Figure S8** Non-linear effects.

## 7. Crystal data and structural refinement for **3a**

The crystal structure of compound **3a** has been deposited at the Cambridge Crystallographic Data Centre (CCDC 2401076).



**Table 1 Crystal data and structure refinement for 3a.**

|                                    |                                                  |
|------------------------------------|--------------------------------------------------|
| Identification code                | <b>3a</b>                                        |
| Empirical formula                  | C <sub>18</sub> H <sub>18</sub> O <sub>4</sub> S |
| Formula weight                     | 330.38                                           |
| Temperature/K                      | 655.0                                            |
| Crystal system                     | monoclinic                                       |
| Space group                        | P2 <sub>1</sub>                                  |
| a/Å                                | 5.8777(2)                                        |
| b/Å                                | 15.5812(5)                                       |
| c/Å                                | 9.2526(3)                                        |
| α/°                                | 90                                               |
| β/°                                | 102.9160(10)                                     |
| γ/°                                | 90                                               |
| Volume/Å <sup>3</sup>              | 825.93(5)                                        |
| Z                                  | 2                                                |
| ρ <sub>calc</sub> /cm <sup>3</sup> | 1.328                                            |
| μ/mm <sup>-1</sup>                 | 1.893                                            |
| F(000)                             | 348.0                                            |
| Crystal size/mm <sup>3</sup>       | 0.22 × 0.21 × 0.2                                |
| Radiation                          | CuKα (λ = 1.54178)                               |
| 2θ range for data collection/°     | 15.026 to 136.96                                 |

|                                             |                                                               |
|---------------------------------------------|---------------------------------------------------------------|
| Index ranges                                | -7 ≤ h ≤ 6, -13 ≤ k ≤ 18, -11 ≤ l ≤ 11                        |
| Reflections collected                       | 6665                                                          |
| Independent reflections                     | 2358 [R <sub>int</sub> = 0.0300, R <sub>sigma</sub> = 0.0386] |
| Data/restraints/parameters                  | 2358/1/209                                                    |
| Goodness-of-fit on F <sup>2</sup>           | 1.063                                                         |
| Final R indexes [I ≥ 2σ (I)]                | R <sub>1</sub> = 0.0309, wR <sub>2</sub> = 0.0816             |
| Final R indexes [all data]                  | R <sub>1</sub> = 0.0312, wR <sub>2</sub> = 0.0819             |
| Largest diff. peak/hole / e Å <sup>-3</sup> | 0.17/-0.26                                                    |
| Flack parameter                             | 0.084(9)                                                      |

## 8. Computational details

Gaussian 16 program was used for all the calculations.<sup>[7]</sup> The PBE0<sup>[8]</sup> density functional with D3BJ<sup>[9-10]</sup> dispersion correction and def2-SVP<sup>[11]</sup> basis sets were utilized for structures optimization and frequency calculations at 298.15 K and 1 atm. Frequency calculations were employed to confirm the minima (zero imaginary frequency) and transition state (only one imaginary frequency) at the same level of theory as above calculations. Intrinsic reaction coordinate (IRC)<sup>[12]</sup> calculations were performed for all the transition states. Further refining of single point energies with solvent effect was calculated at the PBE0-D3BJ and def2-TZVP<sup>[11, 13]</sup> level of theory. The solvent effects were performed with SMD<sup>[14]</sup> model in dichloromethane.

The results of DFT calculations of the reaction process without copper catalyst are outlined in Figure S9.

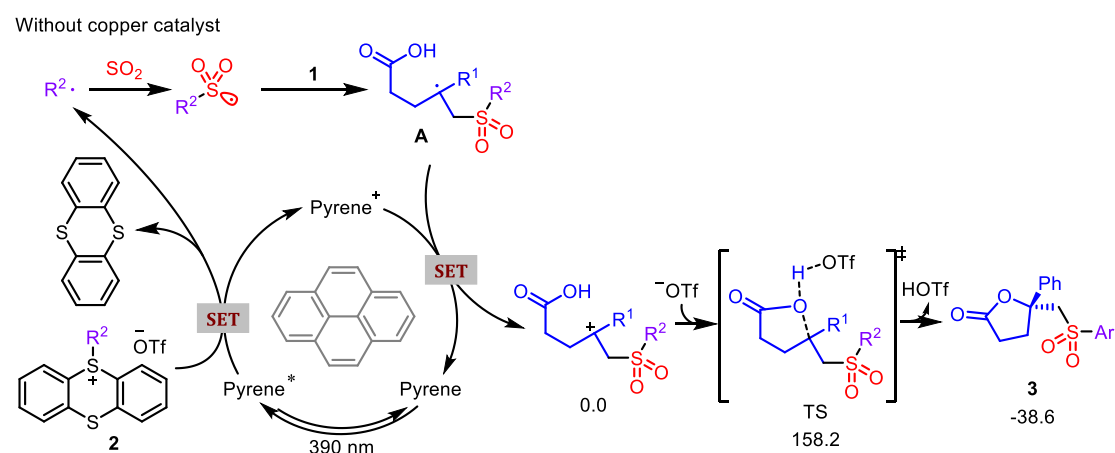


Figure S9. Computational studies of enantioselective radical sulfonylation (in kcal/mol).

### Cartesian coordinates of related structures

L\*Cu<sup>I</sup>X

|    |          |          |          |
|----|----------|----------|----------|
| 6  | 1.787462 | 2.291132 | -1.56976 |
| 6  | 1.54574  | 1.21829  | -0.71281 |
| 7  | 0.308702 | 0.831447 | -0.39937 |
| 6  | -0.74828 | 1.403672 | -0.98303 |
| 6  | -0.60262 | 2.483437 | -1.8538  |
| 6  | 0.686941 | 2.938989 | -2.1267  |
| 6  | 2.543749 | 0.240933 | -0.23596 |
| 7  | 3.869448 | 0.448022 | 0.048506 |
| 6  | 4.536511 | -0.87061 | 0.015281 |
| 6  | 3.309337 | -1.79993 | 0.248596 |
| 7  | 2.169255 | -0.9984  | -0.19121 |
| 6  | -1.97301 | 0.611571 | -0.73569 |
| 7  | -1.80875 | -0.66042 | -0.56456 |
| 6  | -3.11413 | -1.24758 | -0.29929 |
| 6  | -4.11802 | -0.19946 | -0.849   |
| 7  | -3.28557 | 1.016858 | -0.75424 |
| 29 | 0.116746 | -1.24195 | 0.082448 |
| 6  | 3.159824 | -2.25007 | 1.68431  |
| 6  | 2.403849 | -1.52475 | 2.608656 |
| 6  | 2.327243 | -1.93485 | 3.938409 |
| 6  | 3.007911 | -3.07551 | 4.358774 |
| 6  | 3.761765 | -3.80741 | 3.44133  |
| 6  | 3.834263 | -3.3979  | 2.112401 |
| 6  | 5.264431 | -1.08935 | -1.28836 |
| 6  | 6.652371 | -0.93567 | -1.33803 |
| 6  | 7.33929  | -1.11096 | -2.53724 |
| 6  | 6.644007 | -1.4343  | -3.70092 |
| 6  | 5.257949 | -1.58295 | -3.6616  |
| 6  | 4.571389 | -1.41432 | -2.46174 |
| 6  | -3.29852 | -2.63779 | -0.83847 |
| 6  | -4.3341  | -3.43501 | -0.3364  |
| 6  | -4.53739 | -4.72129 | -0.83307 |
| 6  | -3.69925 | -5.22843 | -1.82731 |
| 6  | -2.66301 | -4.43946 | -2.32645 |
| 6  | -2.4675  | -3.14778 | -1.83899 |
| 6  | -5.40621 | -0.12537 | -0.08153 |
| 6  | -5.42115 | 0.296614 | 1.252484 |
| 6  | -6.61219 | 0.314031 | 1.972163 |
| 6  | -7.7996  | -0.10009 | 1.367513 |
| 6  | -7.79041 | -0.52198 | 0.039925 |
| 6  | -6.59879 | -0.52868 | -0.68302 |

|   |          |          |          |
|---|----------|----------|----------|
| 1 | 2.805162 | 2.592386 | -1.81819 |
| 1 | -1.46579 | 2.946095 | -2.33126 |
| 1 | 0.834816 | 3.774039 | -2.81394 |
| 1 | 5.248021 | -0.91495 | 0.849367 |
| 1 | 3.394919 | -2.68911 | -0.39246 |
| 1 | -3.23503 | -1.28999 | 0.79989  |
| 1 | -4.33169 | -0.40138 | -1.91247 |
| 1 | 1.847395 | -0.64271 | 2.282575 |
| 1 | 1.731042 | -1.35946 | 4.6504   |
| 1 | 2.950935 | -3.39604 | 5.401407 |
| 1 | 4.295622 | -4.70464 | 3.762743 |
| 1 | 4.424922 | -3.97645 | 1.396267 |
| 1 | 7.193109 | -0.65702 | -0.43106 |
| 1 | 8.424704 | -0.99151 | -2.56254 |
| 1 | 7.182949 | -1.57276 | -4.64094 |
| 1 | 4.708735 | -1.83851 | -4.57072 |
| 1 | 3.485085 | -1.53848 | -2.44347 |
| 1 | -4.98899 | -3.04189 | 0.446027 |
| 1 | -5.35461 | -5.33138 | -0.44124 |
| 1 | -3.85767 | -6.2368  | -2.21651 |
| 1 | -2.00564 | -4.83019 | -3.10672 |
| 1 | -1.65588 | -2.52748 | -2.22545 |
| 1 | -4.49545 | 0.626999 | 1.732549 |
| 1 | -6.6162  | 0.654552 | 3.010033 |
| 1 | -8.73458 | -0.08676 | 1.931985 |
| 1 | -8.71848 | -0.83856 | -0.44102 |
| 1 | -6.59436 | -0.84515 | -1.72885 |
| 6 | -3.84658 | 2.286196 | -0.98098 |
| 6 | 4.56231  | 1.59783  | 0.433008 |
| 8 | -4.86289 | 2.380958 | -1.62397 |
| 8 | 5.769407 | 1.598523 | 0.446581 |
| 6 | -3.17302 | 3.44652  | -0.34092 |
| 6 | -2.43044 | 3.32677  | 0.840123 |
| 6 | -3.35737 | 4.706769 | -0.92193 |
| 6 | -1.85771 | 4.455716 | 1.418183 |
| 1 | -2.31782 | 2.353744 | 1.323301 |
| 6 | -2.76568 | 5.829203 | -0.35322 |
| 1 | -3.97192 | 4.784294 | -1.82154 |
| 6 | -2.01306 | 5.703761 | 0.815887 |
| 1 | -1.29666 | 4.36412  | 2.350234 |
| 1 | -2.90122 | 6.80982  | -0.81443 |
| 1 | -1.55898 | 6.588282 | 1.268735 |
| 6 | 3.742317 | 2.768263 | 0.854    |
| 6 | 4.128363 | 4.041912 | 0.425551 |

|   |          |          |          |
|---|----------|----------|----------|
| 6 | 2.644497 | 2.623068 | 1.709688 |
| 6 | 3.380126 | 5.155115 | 0.79686  |
| 1 | 5.014044 | 4.144314 | -0.20559 |
| 6 | 1.915164 | 3.741584 | 2.100676 |
| 1 | 2.374033 | 1.635809 | 2.091006 |
| 6 | 2.269792 | 5.004901 | 1.629175 |
| 1 | 3.670623 | 6.147743 | 0.445975 |
| 1 | 1.07071  | 3.626194 | 2.783059 |
| 1 | 1.689137 | 5.881386 | 1.925979 |
| 7 | -0.38243 | -2.85873 | 1.072565 |
| 6 | -0.85516 | -3.89484 | 1.267256 |
| 6 | -1.47246 | -5.18663 | 1.465251 |
| 1 | -1.9489  | -5.23106 | 2.454898 |
| 1 | -2.23803 | -5.33707 | 0.687733 |
| 1 | -0.71612 | -5.98118 | 1.394756 |

L\*Cu<sup>II</sup>X

|    |          |          |          |
|----|----------|----------|----------|
| 6  | -2.73464 | -3.21328 | -1.81673 |
| 6  | -2.33013 | -2.06743 | -1.13318 |
| 7  | -1.04144 | -1.73428 | -1.09547 |
| 6  | -0.07094 | -2.45813 | -1.6609  |
| 6  | -0.39864 | -3.62423 | -2.34952 |
| 6  | -1.74698 | -3.98552 | -2.42446 |
| 6  | -3.1556  | -0.9989  | -0.50755 |
| 7  | -4.44601 | -1.01937 | -0.11692 |
| 6  | -4.89198 | 0.386601 | 0.080679 |
| 6  | -3.51905 | 1.127561 | 0.170095 |
| 7  | -2.57754 | 0.164376 | -0.38911 |
| 6  | 1.236407 | -1.75337 | -1.5166  |
| 7  | 1.175371 | -0.52927 | -1.06019 |
| 6  | 2.522109 | 0.033516 | -0.99428 |
| 6  | 3.364817 | -0.95242 | -1.84539 |
| 7  | 2.474046 | -2.14623 | -1.86427 |
| 29 | -0.60533 | 0.132569 | -0.55335 |
| 6  | -3.1085  | 1.573145 | 1.551157 |
| 6  | -2.48324 | 0.697124 | 2.444135 |
| 6  | -2.12193 | 1.126063 | 3.719854 |
| 6  | -2.38803 | 2.436078 | 4.116585 |
| 6  | -3.01792 | 3.313923 | 3.233555 |
| 6  | -3.37259 | 2.885452 | 1.955299 |
| 6  | -5.77724 | 0.856337 | -1.04266 |
| 6  | -7.12798 | 1.114276 | -0.80062 |
| 6  | -7.95496 | 1.553313 | -1.83232 |

|   |          |          |          |
|---|----------|----------|----------|
| 6 | -7.4396  | 1.727402 | -3.11504 |
| 6 | -6.09224 | 1.464828 | -3.36631 |
| 6 | -5.264   | 1.034502 | -2.33412 |
| 6 | 2.588758 | 1.464897 | -1.44619 |
| 6 | 3.359492 | 2.385196 | -0.73196 |
| 6 | 3.424045 | 3.716643 | -1.14236 |
| 6 | 2.712157 | 4.136795 | -2.26611 |
| 6 | 1.947267 | 3.219001 | -2.98785 |
| 6 | 1.891603 | 1.887513 | -2.58291 |
| 6 | 4.730816 | -1.24499 | -1.29855 |
| 6 | 4.883741 | -1.81466 | -0.02856 |
| 6 | 6.154476 | -2.07772 | 0.473593 |
| 6 | 7.282302 | -1.76561 | -0.2872  |
| 6 | 7.135114 | -1.198   | -1.55076 |
| 6 | 5.862019 | -0.94399 | -2.0583  |
| 1 | -3.78516 | -3.49381 | -1.88031 |
| 1 | 0.362969 | -4.23751 | -2.82855 |
| 1 | -2.03004 | -4.88338 | -2.9778  |
| 1 | -5.43783 | 0.435366 | 1.031733 |
| 1 | -3.55532 | 2.00762  | -0.48903 |
| 1 | 2.861042 | -0.02063 | 0.055087 |
| 1 | 3.451146 | -0.58942 | -2.88243 |
| 1 | -2.26993 | -0.33304 | 2.144048 |
| 1 | -1.63809 | 0.433507 | 4.412136 |
| 1 | -2.11396 | 2.770659 | 5.119497 |
| 1 | -3.24268 | 4.336572 | 3.54588  |
| 1 | -3.86918 | 3.575445 | 1.266931 |
| 1 | -7.53886 | 0.957136 | 0.199289 |
| 1 | -9.00944 | 1.755399 | -1.63318 |
| 1 | -8.08919 | 2.069263 | -3.92371 |
| 1 | -5.68697 | 1.601841 | -4.37122 |
| 1 | -4.20749 | 0.839687 | -2.54452 |
| 1 | 3.921386 | 2.055323 | 0.146298 |
| 1 | 4.043619 | 4.426764 | -0.58945 |
| 1 | 2.769016 | 5.177721 | -2.59291 |
| 1 | 1.402986 | 3.541547 | -3.8783  |
| 1 | 1.297328 | 1.171651 | -3.1584  |
| 1 | 4.00609  | -2.06545 | 0.575368 |
| 1 | 6.268901 | -2.52838 | 1.461756 |
| 1 | 8.279935 | -1.97066 | 0.10708  |
| 1 | 8.015885 | -0.95918 | -2.15037 |
| 1 | 5.746541 | -0.51591 | -3.057   |
| 6 | 2.946801 | -3.33916 | -2.51183 |
| 6 | -5.32183 | -2.087   | 0.224446 |

|   |          |          |          |
|---|----------|----------|----------|
| 8 | 3.671467 | -3.21337 | -3.45965 |
| 8 | -6.50017 | -1.86833 | 0.280061 |
| 6 | 2.555826 | -4.62684 | -1.90612 |
| 6 | 2.127917 | -4.73482 | -0.57443 |
| 6 | 2.676051 | -5.7773  | -2.69969 |
| 6 | 1.797075 | -5.98108 | -0.05286 |
| 1 | 2.089346 | -3.85114 | 0.066219 |
| 6 | 2.331579 | -7.01815 | -2.17641 |
| 1 | 3.04473  | -5.6766  | -3.72295 |
| 6 | 1.889165 | -7.11955 | -0.85538 |
| 1 | 1.485175 | -6.07186 | 0.989749 |
| 1 | 2.420756 | -7.91416 | -2.7942  |
| 1 | 1.632259 | -8.09788 | -0.44261 |
| 6 | -4.70281 | -3.39416 | 0.553575 |
| 6 | -5.38828 | -4.55551 | 0.17597  |
| 6 | -3.51426 | -3.49062 | 1.290624 |
| 6 | -4.8515  | -5.80348 | 0.476019 |
| 1 | -6.33944 | -4.46423 | -0.35361 |
| 6 | -2.99445 | -4.74152 | 1.609673 |
| 1 | -3.01707 | -2.5885  | 1.656656 |
| 6 | -3.65322 | -5.89665 | 1.187343 |
| 1 | -5.37839 | -6.70992 | 0.170399 |
| 1 | -2.08358 | -4.81862 | 2.207268 |
| 1 | -3.24494 | -6.87869 | 1.436783 |
| 7 | -0.19073 | 1.937249 | 0.091749 |
| 6 | 0.048234 | 2.996873 | 0.475631 |
| 6 | 0.336726 | 4.321691 | 0.962786 |
| 1 | -0.42488 | 4.610223 | 1.702511 |
| 1 | 1.332548 | 4.331401 | 1.429108 |
| 1 | 0.330623 | 5.029726 | 0.121174 |

B-os

|   |          |          |          |
|---|----------|----------|----------|
| 6 | 4.453429 | 1.141958 | 1.851012 |
| 6 | 3.716394 | 0.47344  | 0.873802 |
| 7 | 2.639429 | 1.036201 | 0.344624 |
| 6 | 2.140354 | 2.203212 | 0.752378 |
| 6 | 2.779271 | 2.900267 | 1.776127 |
| 6 | 3.958694 | 2.363717 | 2.300149 |
| 6 | 3.966195 | -0.85661 | 0.26875  |
| 7 | 4.829523 | -1.80971 | 0.736864 |
| 6 | 5.083059 | -2.73814 | -0.37358 |
| 6 | 3.834104 | -2.4763  | -1.2804  |
| 7 | 3.366073 | -1.1537  | -0.83549 |

|    |          |          |          |
|----|----------|----------|----------|
| 6  | 0.851887 | 2.474167 | 0.055024 |
| 7  | 0.438767 | 1.556903 | -0.76067 |
| 6  | -0.89654 | 1.898657 | -1.23344 |
| 6  | -1.3867  | 2.894265 | -0.15144 |
| 7  | -0.07741 | 3.449489 | 0.288789 |
| 29 | 1.607092 | -0.06959 | -0.94795 |
| 6  | 2.748182 | -3.52246 | -1.20988 |
| 6  | 2.39557  | -4.12757 | 0.001562 |
| 6  | 1.398327 | -5.09766 | 0.043748 |
| 6  | 0.741731 | -5.47452 | -1.12722 |
| 6  | 1.080926 | -4.87108 | -2.33713 |
| 6  | 2.077935 | -3.89936 | -2.37621 |
| 6  | 6.384663 | -2.4723  | -1.08606 |
| 6  | 6.890665 | -3.46214 | -1.93529 |
| 6  | 8.055966 | -3.23858 | -2.66243 |
| 6  | 8.731562 | -2.02387 | -2.54484 |
| 6  | 8.236568 | -1.03784 | -1.69488 |
| 6  | 7.067657 | -1.26104 | -0.96853 |
| 6  | -0.86463 | 2.476606 | -2.6328  |
| 6  | -2.07012 | 2.614674 | -3.33152 |
| 6  | -2.07932 | 3.177043 | -4.60442 |
| 6  | -0.88996 | 3.602226 | -5.19755 |
| 6  | 0.312873 | 3.455061 | -4.51212 |
| 6  | 0.324868 | 2.893708 | -3.23511 |
| 6  | -2.11502 | 2.283755 | 1.022915 |
| 6  | -1.73123 | 1.049961 | 1.564305 |
| 6  | -2.39799 | 0.532191 | 2.672901 |
| 6  | -3.45193 | 1.237456 | 3.253699 |
| 6  | -3.8418  | 2.462003 | 2.713613 |
| 6  | -3.17539 | 2.981112 | 1.605939 |
| 1  | 5.36177  | 0.709799 | 2.269448 |
| 1  | 2.387594 | 3.844383 | 2.149942 |
| 1  | 4.490434 | 2.906874 | 3.083704 |
| 1  | 5.087128 | -3.76259 | 0.021621 |
| 1  | 4.161149 | -2.37397 | -2.32483 |
| 1  | -1.53537 | 1.006516 | -1.23022 |
| 1  | -1.98088 | 3.706971 | -0.58403 |
| 1  | 2.905873 | -3.85701 | 0.928679 |
| 1  | 1.138445 | -5.56604 | 0.995384 |
| 1  | -0.03372 | -6.24348 | -1.09692 |
| 1  | 0.57013  | -5.16121 | -3.25782 |
| 1  | 2.337658 | -3.42497 | -3.32562 |
| 1  | 6.370212 | -4.4202  | -2.02357 |
| 1  | 8.445059 | -4.02097 | -3.31771 |

|   |          |          |          |
|---|----------|----------|----------|
| 1 | 9.649624 | -1.85038 | -3.11034 |
| 1 | 8.767492 | -0.08894 | -1.59035 |
| 1 | 6.697664 | -0.48856 | -0.29051 |
| 1 | -2.99936 | 2.265946 | -2.87611 |
| 1 | -3.02638 | 3.27548  | -5.13974 |
| 1 | -0.90124 | 4.041245 | -6.19764 |
| 1 | 1.250161 | 3.773844 | -4.9741  |
| 1 | 1.276849 | 2.760897 | -2.71544 |
| 1 | -0.92466 | 0.461242 | 1.120395 |
| 1 | -2.09672 | -0.43759 | 3.07746  |
| 1 | -3.97366 | 0.829095 | 4.122657 |
| 1 | -4.67186 | 3.01763  | 3.155396 |
| 1 | -3.47252 | 3.947105 | 1.192957 |
| 6 | -3.95863 | -2.19675 | -0.748   |
| 6 | -2.58087 | -1.82551 | -0.30306 |
| 6 | -4.40158 | -1.54849 | -2.00741 |
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| 8 | 0.274496 | -1.51513 | 0.273309 |
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| 6 | -4.40973 | -3.60827 | 1.24735  |
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| 1 | -2.43529 | -1.98082 | 0.772222 |
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| 1 | -1.29876 | -3.54973 | -0.68304 |
| 1 | -6.57787 | -2.81851 | -1.27514 |
| 1 | -8.08477 | -4.12297 | 0.125941 |
| 1 | -7.28691 | -5.11969 | 2.273725 |
| 1 | -4.911   | -4.79271 | 2.965165 |
| 1 | -3.37747 | -3.48216 | 1.576214 |
| 6 | -6.23577 | 0.074656 | -0.58286 |
| 6 | -5.88994 | 0.043111 | 0.767009 |
| 6 | -6.88758 | -0.18872 | 1.704209 |
| 6 | -8.22033 | -0.38314 | 1.313411 |
| 6 | -8.53867 | -0.30308 | -0.04905 |
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| 1 | -4.85406 | 0.199538 | 1.072232 |

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| 1  | -9.57773 | -0.42264 | -0.3659  |
| 1  | -7.79261 | -0.02056 | -2.06946 |
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| 1  | -9.13806 | -0.09882 | 3.247464 |
| 1  | -9.21258 | -1.75705 | 2.62651  |
| 1  | -10.2837 | -0.51335 | 1.945637 |
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| 6  | 1.696666 | 6.361589 | 1.62397  |
| 6  | 2.200448 | 5.202129 | -0.44608 |
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| 6  | 1.843796 | -0.93897 | 3.747397 |
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| 1  | 1.686231 | -0.11248 | 5.735569 |

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| 6 | 2.212156 | 2.383021 | 0.021975 |
| 7 | 2.535105 | 1.113283 | -0.22797 |
| 6 | 3.727969 | 0.595192 | 0.029905 |
| 6 | 4.776084 | 1.417286 | 0.441826 |

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| 6  | -1.3329  | 1.844061 | -0.63435 |
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| 6  | -0.81771 | -0.34406 | 3.040264 |
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| 6  | 7.491967 | -4.30949 | -2.12202 |
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| 1  | 5.274232 | 3.434829 | 1.001929 |
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| 1  | -1.86142 | 0.885288 | -0.55409 |
| 1  | 2.858949 | -3.11976 | -2.01788 |
| 1  | 4.374262 | -3.88362 | 0.315865 |
| 1  | -0.85612 | 1.170959 | -3.20251 |

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| 1 | -2.87653 | 4.173982 | -0.83717 |
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| 1  | -5.41416 | -3.19594 | 2.602381 |
| 6  | -9.27391 | -0.89379 | 1.251098 |
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| 6  | 3.502864 | -0.42221 | 2.762524 |
| 6  | 5.494731 | 0.812118 | 4.272133 |
| 1  | 6.881018 | -0.46615 | 3.203471 |
| 6  | 3.157049 | 0.537231 | 3.709741 |
| 1  | 2.706822 | -0.91664 | 2.20065  |
| 6  | 4.15627  | 1.167713 | 4.451797 |
| 1  | 6.27246  | 1.290329 | 4.871642 |
| 1  | 2.104    | 0.78257  | 3.870615 |
| 1  | 3.889274 | 1.927172 | 5.190324 |
| 6  | 0.982328 | 5.541577 | 0.035199 |
| 6  | 1.640041 | 6.603939 | 0.667741 |
| 6  | 1.220568 | 5.272326 | -1.31981 |
| 6  | 2.580065 | 7.350643 | -0.03479 |
| 1  | 1.405804 | 6.825078 | 1.711388 |
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| 1  | 2.324923 | 5.838478 | -3.08166 |
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| 7  | 1.430044 | 1.422061 | 1.721115 |
| 6  | 2.84689  | 1.782358 | 1.883922 |
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| 6  | 4.006745 | 1.683833 | 5.499804 |
| 6  | 3.508241 | 2.888037 | 5.992311 |
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| 6  | 2.591131 | 3.394201 | 3.817252 |
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| 1  | -0.67898 | 4.477539 | 1.388386 |

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| 1 | -7.49759 | -3.26903 | 4.161245 |
| 1 | -5.2123  | -2.32163 | 4.438714 |
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| 1 | 4.192972 | 0.390934 | 3.772509 |
| 1 | 4.567497 | 1.013853 | 6.155644 |
| 1 | 3.675918 | 3.165131 | 7.03562  |
| 1 | 2.413265 | 4.692369 | 5.529449 |
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| 1 | 3.775649 | 0.547098 | -3.17406 |
| 1 | 6.242443 | 1.005297 | -3.16127 |
| 1 | 7.231759 | 2.347491 | -1.31302 |
| 1 | 5.785602 | 3.253391 | 0.485947 |
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| 6 | 2.300192 | -1.28209 | 0.5992   |
| 6 | 1.565629 | -2.18255 | -2.15088 |
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| 1 | 2.138984 | -0.54614 | 1.391819 |
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| 1 | 2.657264 | -2.03575 | -2.08206 |

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| 6  | 5.8944   | -2.4854  | -2.03292 |
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| 1  | 6.485691 | -1.79743 | -2.64228 |
| 1  | 4.936189 | -5.74798 | -1.89522 |
| 1  | 3.812427 | -4.9094  | 0.176316 |
| 6  | 6.474196 | -4.32866 | -3.64895 |
| 1  | 6.067665 | -5.292   | -3.98495 |
| 1  | 7.548642 | -4.47275 | -3.44858 |
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| 6  | -5.49845 | 0.476651 | -0.77367 |
| 6  | 1.523621 | 4.31007  | -0.46791 |
| 8  | -6.58908 | -0.02797 | -0.89348 |
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| 6  | -5.21639 | 1.866218 | -1.21567 |
| 6  | -6.20778 | 2.823904 | -0.96771 |
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| 1  | -3.28777 | 1.484086 | -2.13963 |
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| 1  | -2.94695 | 3.8328   | -2.8126  |
| 1  | -4.66318 | 5.552643 | -2.28719 |
| 6  | 0.558277 | 4.174007 | -1.59216 |
| 6  | 0.170705 | 5.352833 | -2.24593 |
| 6  | 0.227253 | 2.934167 | -2.15302 |
| 6  | -0.52373 | 5.288786 | -3.44792 |
| 1  | 0.464581 | 6.312262 | -1.81549 |
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| 1  | 0.513862 | 1.988953 | -1.69141 |

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TS1<sub>R</sub>

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| 6  | 0.090851 | 1.772312 | 4.376288 |
| 6  | 1.192356 | 2.232401 | 5.093419 |
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| 6  | 2.195552 | 3.001031 | 3.034394 |
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| 6  | 2.137226 | -4.83761 | -1.93173 |
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| 1 | 3.024246 | 3.483127 | 2.508972 |
| 1 | 3.026743 | 5.812774 | 0.347559 |
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| 6 | 4.188303 | 0.948107 | 1.309208 |

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| 1  | 2.603148 | -2.5617  | -0.23559 |
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| 1  | 7.385827 | -0.41418 | -4.22572 |
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| 6  | 6.407702 | -2.79979 | -1.64423 |
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| 8  | 1.486747 | 5.380728 | -0.99811 |

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| 6  | 2.196965 | 1.713568 | 0.956245 |
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| 6  | 1.964249 | 3.983108 | 1.656624 |
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| D <sub>R</sub> |          |          |          |
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| 1  | -5.02852 | 2.875132 | 0.027418 |

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| 6 | -1.07166 | 3.634997 | 1.510504 |

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| 6  | 1.637144 | 8.127984 | 0.567222 |
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| 6  | -0.13623 | 6.745528 | -0.36414 |
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| 6  | 2.766506 | 9.094834 | 0.379695 |
| 1  | 3.55868  | 8.941167 | 1.126397 |
| 1  | 3.209834 | 9.012518 | -0.62215 |
| 1  | 2.406974 | 10.13058 | 0.496639 |
| 16 | -1.83731 | 5.234928 | 1.158089 |
| 8  | -2.5243  | 5.58454  | 2.4003   |
| 8  | -2.57148 | 5.122454 | -0.10028 |

A

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| HOTf |          |          |          |
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| 1 | 1.171684 | 0.652975 | 1.131595 |

OTf

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| 8  | 1.625952 | -6.10915 | 3.908853 |
| 8  | 4.051522 | -6.1666  | 4.464418 |
| 6  | 2.516269 | -8.04953 | 5.39674  |
| 9  | 3.545887 | -8.87121 | 5.637283 |
| 9  | 1.448767 | -8.82157 | 5.156835 |
| 9  | 2.271226 | -7.3876  | 6.534696 |

3

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| 6 | -1.0904  | 7.723408 | 2.502848 |
| 6 | -1.01258 | 8.302369 | 3.925513 |
| 6 | 0.278152 | 7.658123 | 1.805649 |
| 6 | -0.56067 | 7.109831 | 4.747259 |
| 6 | -1.10734 | 5.927427 | 3.973296 |
| 8 | -1.4312  | 6.338397 | 2.723953 |
| 8 | -1.25234 | 4.800591 | 4.334463 |
| 6 | -2.15202 | 8.330637 | 1.61442  |
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| 6 | -3.53671 | 8.165151 | -0.36399 |
| 6 | -4.0514  | 9.438045 | -0.11968 |
| 6 | -3.62478 | 10.15082 | 0.997177 |
| 6 | -2.68336 | 9.596727 | 1.863221 |
| 1 | -0.35683 | 9.178936 | 3.994603 |
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| 1 | 0.223031 | 6.946294 | 0.967301 |
| 1 | -0.93105 | 7.088983 | 5.780045 |
| 1 | 0.536284 | 7.008702 | 4.796725 |
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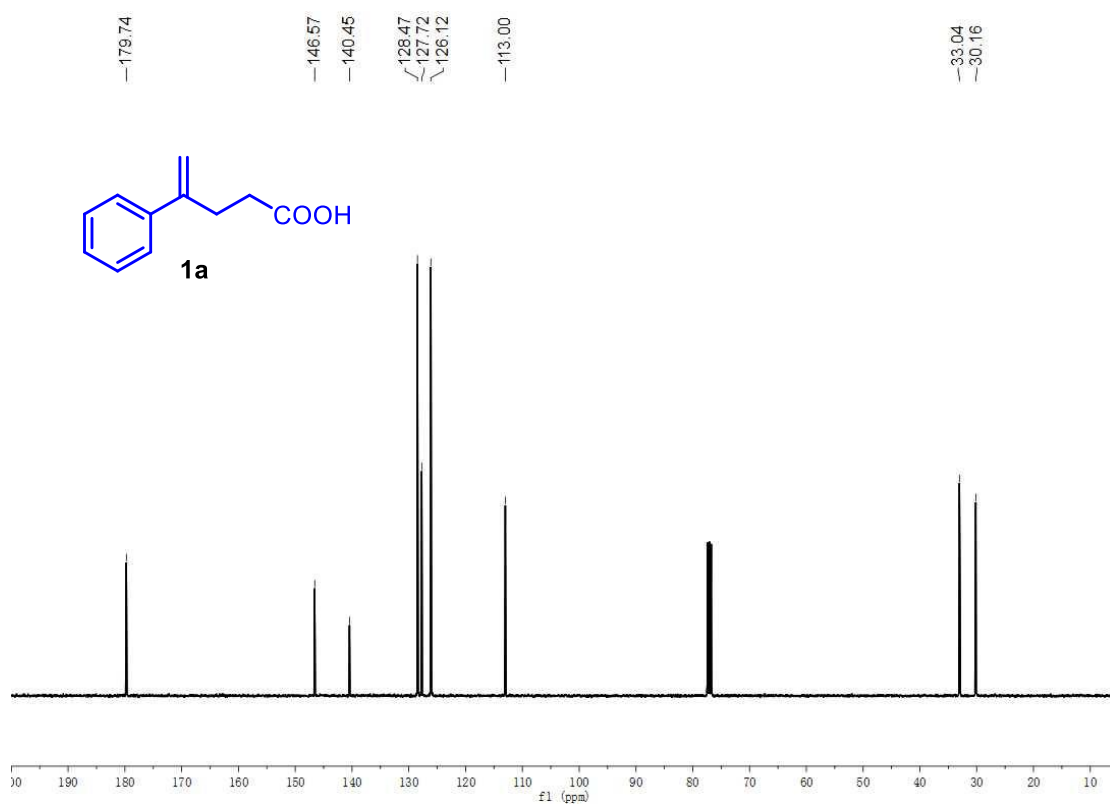
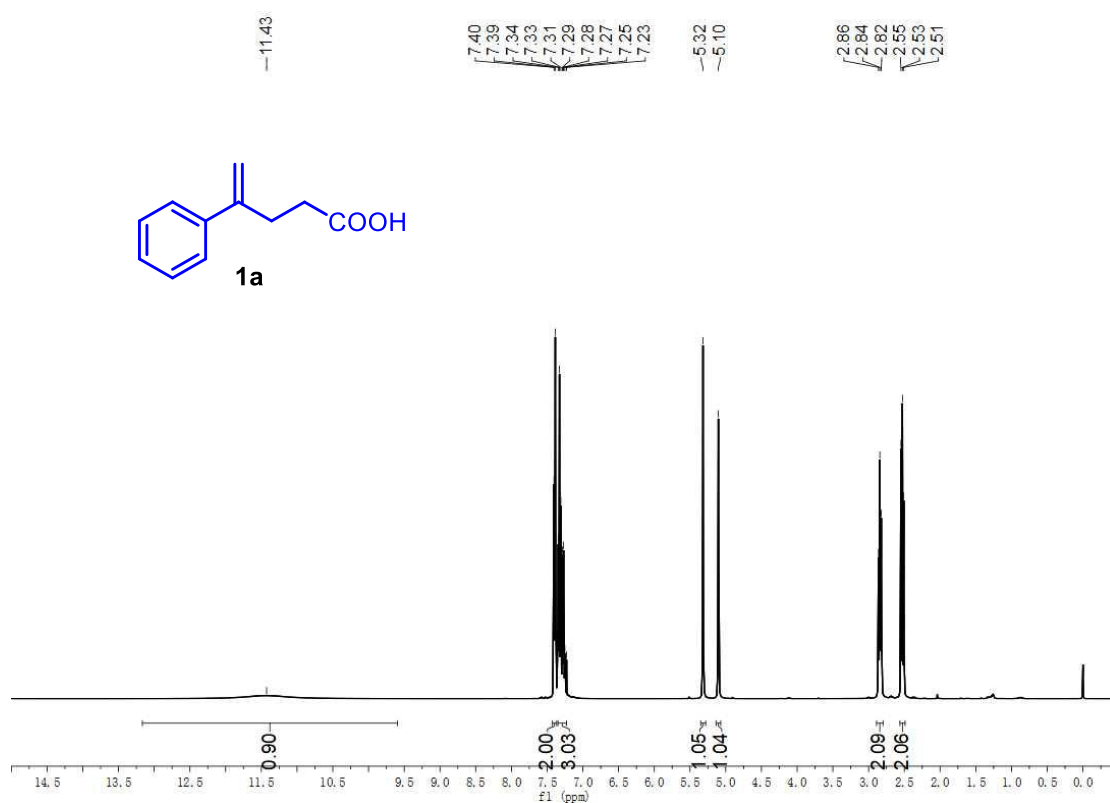
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| 6  | 1.153854 | 12.35745 | 4.185581 |
| 6  | 0.2141   | 12.41497 | 3.148778 |
| 6  | 0.167208 | 11.43252 | 2.163033 |
| 1  | 2.770811 | 9.494866 | 3.218125 |
| 1  | 2.820367 | 11.24379 | 4.994587 |
| 1  | -0.4924  | 13.24836 | 3.110829 |
| 1  | -0.55481 | 11.46762 | 1.344943 |
| 6  | 1.214894 | 13.43006 | 5.23159  |
| 1  | 0.264469 | 13.97617 | 5.307562 |
| 1  | 1.99988  | 14.16461 | 4.986527 |
| 1  | 1.456716 | 13.01487 | 6.220782 |
| 16 | 0.996277 | 9.100123 | 0.989539 |
| 8  | 2.366976 | 8.68788  | 0.691539 |
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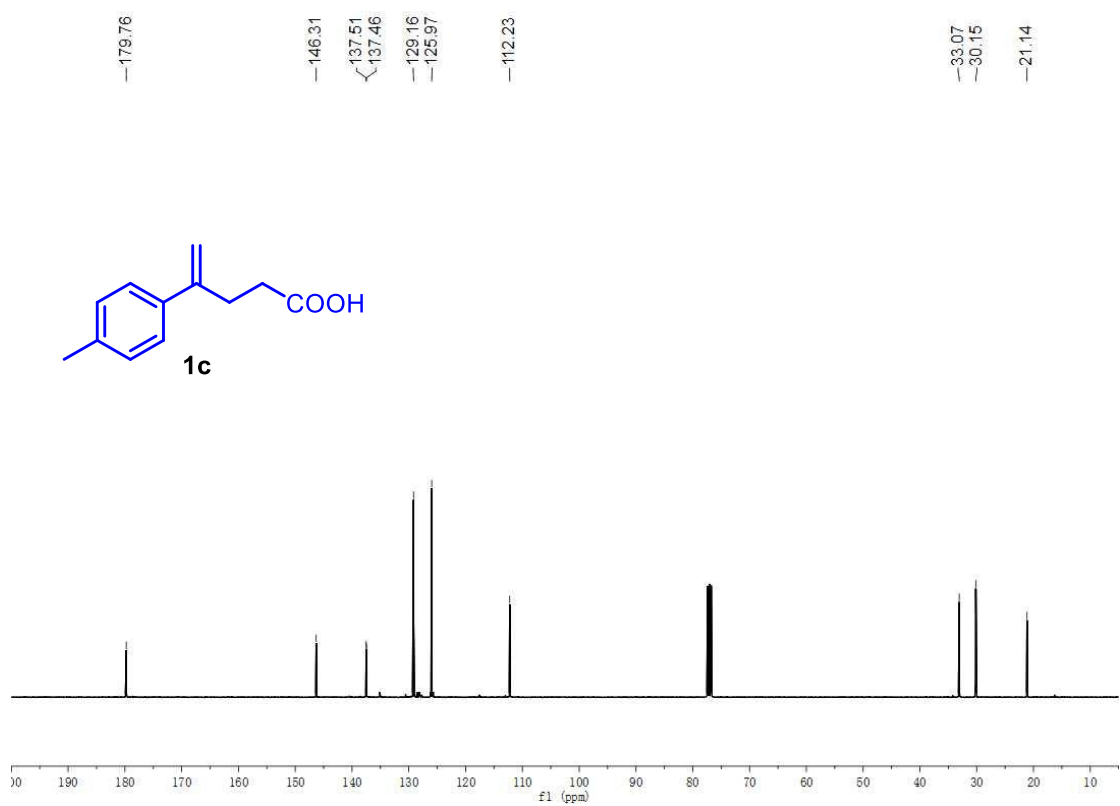
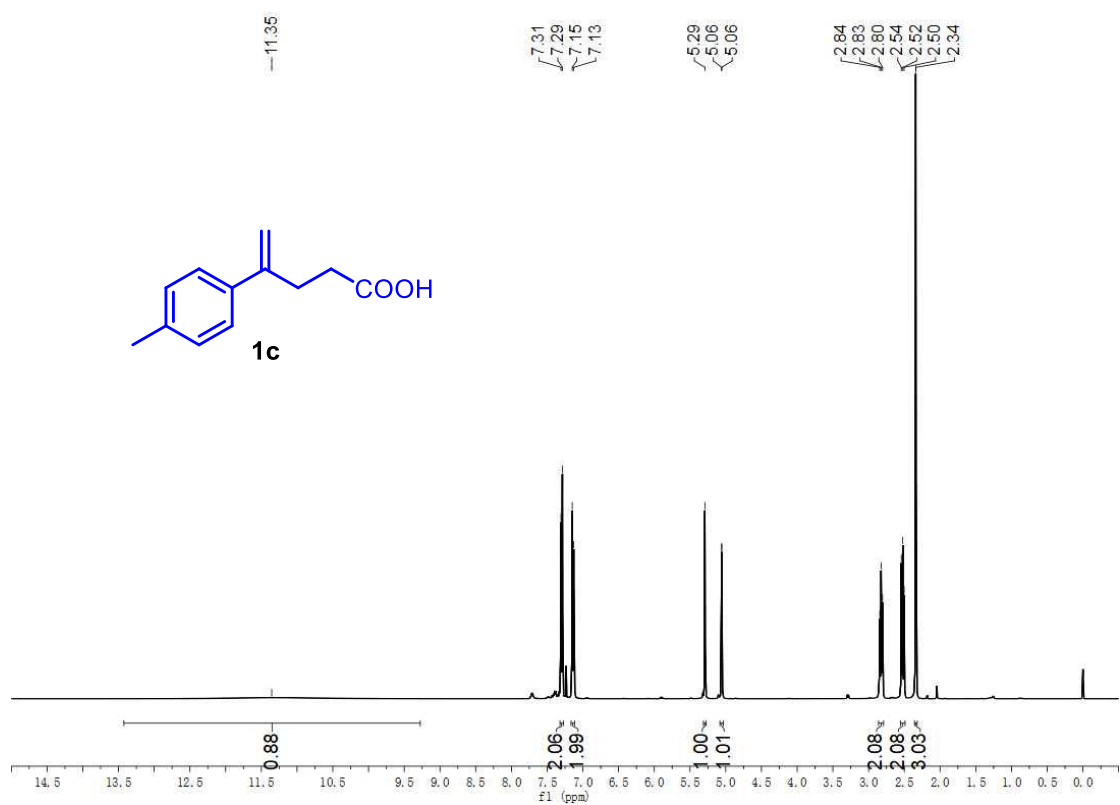
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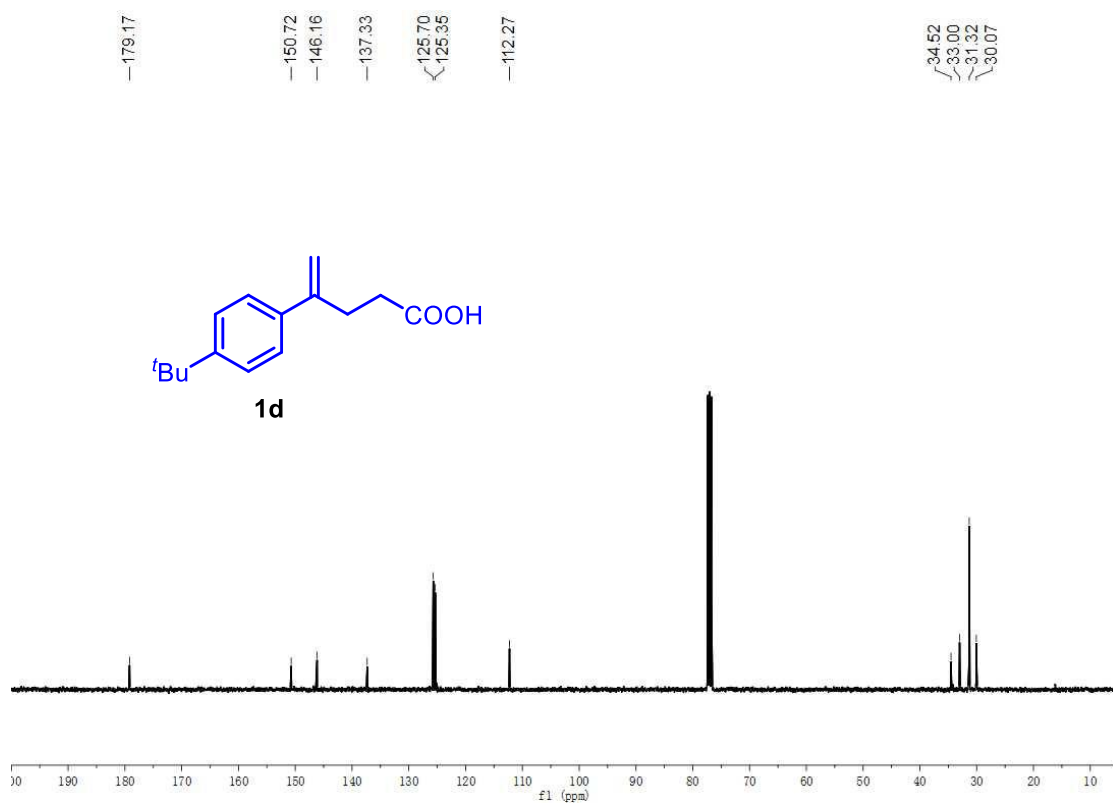
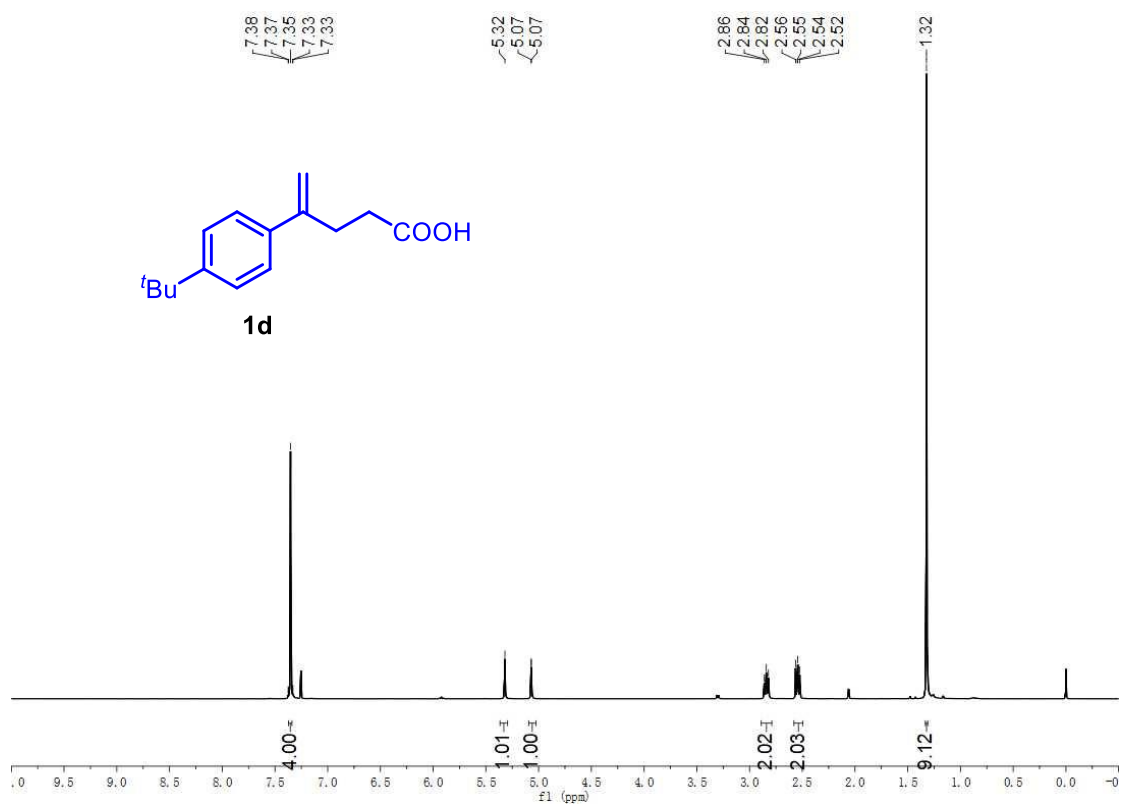
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| 6 | -2.23529 | 8.475138 | 1.737958 |
| 6 | -0.65398 | 7.272765 | 4.828046 |
| 6 | -1.47857 | 6.150154 | 4.234838 |
| 8 | -1.80168 | 6.478091 | 2.957757 |
| 8 | -1.82115 | 5.122822 | 4.732712 |
| 6 | 0.101888 | 7.438257 | 1.824493 |
| 6 | 0.845965 | 8.499829 | 1.299084 |
| 6 | 2.054622 | 8.265357 | 0.653919 |
| 6 | 2.540072 | 6.963432 | 0.527292 |
| 6 | 1.804789 | 5.902478 | 1.047137 |
| 6 | 0.589404 | 6.137153 | 1.690934 |
| 1 | -0.12439 | 9.166253 | 3.815621 |
| 1 | -1.85143 | 8.96645  | 4.21462  |
| 1 | -3.20439 | 8.449011 | 2.254643 |
| 1 | -1.93908 | 9.526833 | 1.604395 |
| 1 | -0.92128 | 7.428905 | 5.880639 |
| 1 | 0.403073 | 6.962221 | 4.790461 |
| 1 | 0.472822 | 9.52396  | 1.374628 |
| 1 | 2.617752 | 9.104082 | 0.238865 |
| 1 | 3.489424 | 6.779062 | 0.019204 |
| 1 | 2.172797 | 4.878414 | 0.949036 |
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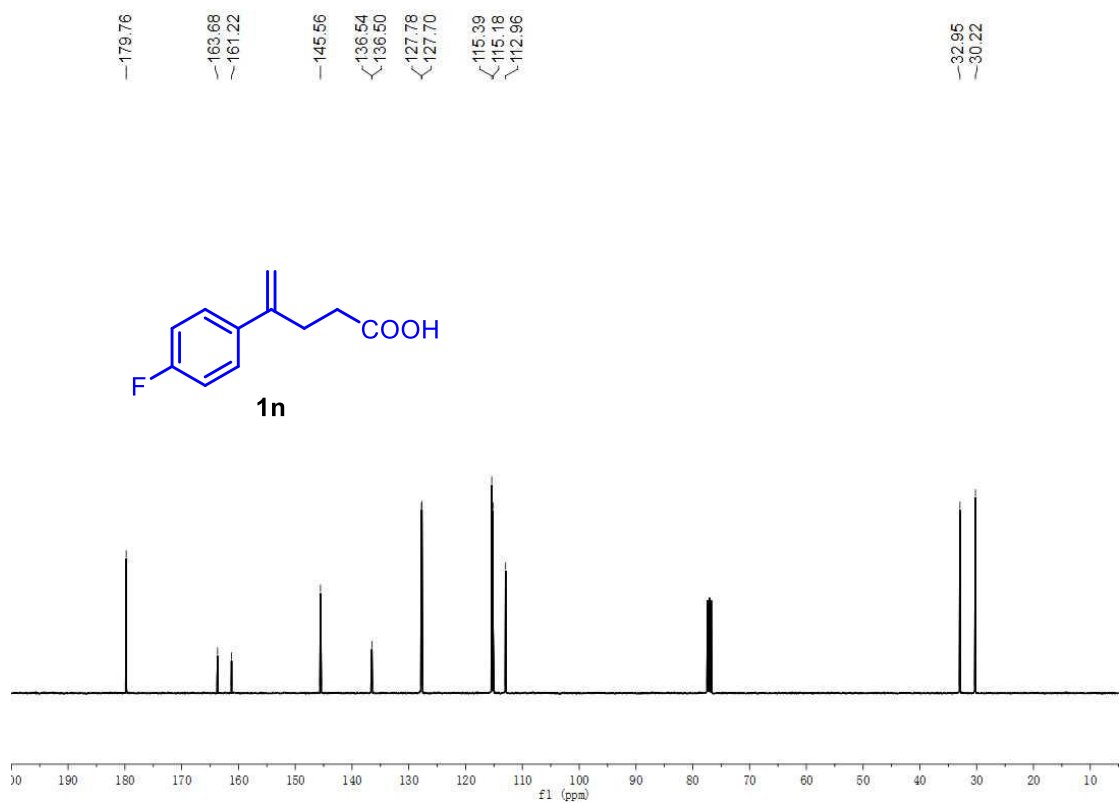
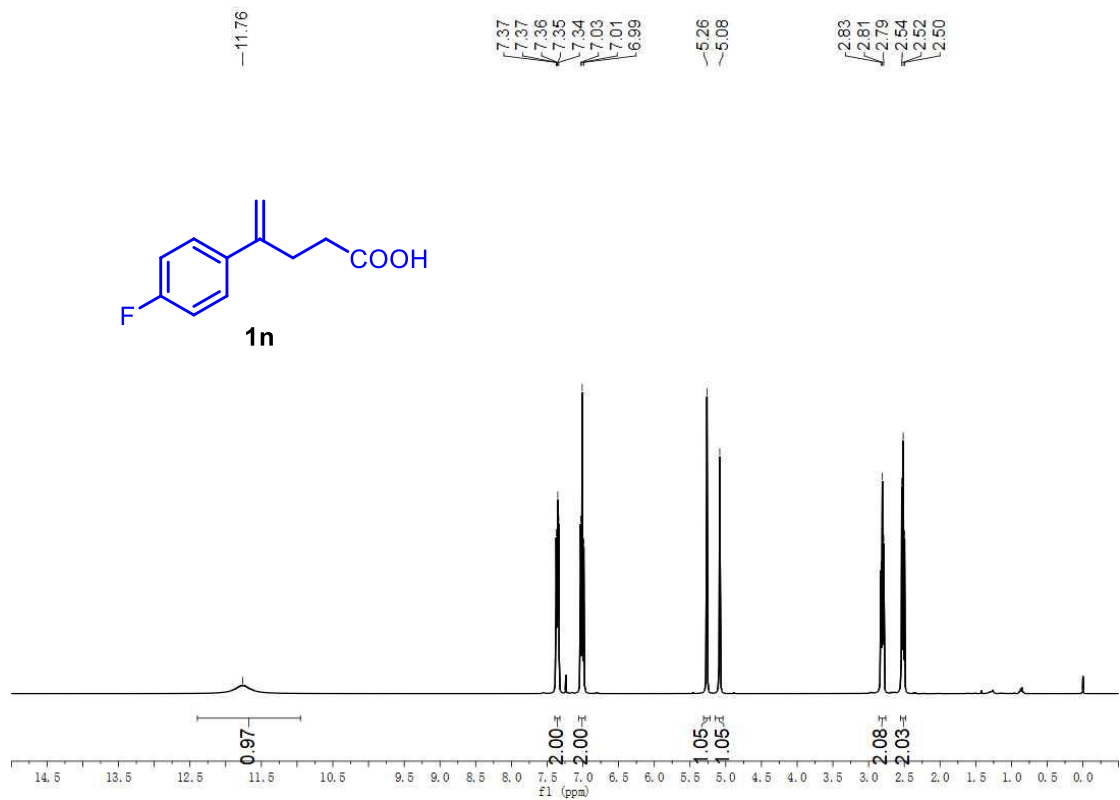
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| 6  | -3.89582 | 4.208522 | 0.582836 |
| 6  | -2.85213 | 3.427028 | 0.063006 |
| 6  | -1.74301 | 4.074786 | -0.49167 |
| 6  | -1.65495 | 5.464306 | -0.50833 |
| 1  | -4.65014 | 6.201856 | 0.952999 |
| 1  | -4.77612 | 3.716884 | 1.004905 |
| 1  | -0.93055 | 3.480447 | -0.91778 |
| 1  | -0.79334 | 5.982044 | -0.93296 |
| 6  | -2.93037 | 1.930572 | 0.115343 |
| 1  | -2.13424 | 1.461809 | -0.47912 |
| 1  | -3.89929 | 1.567834 | -0.26004 |
| 1  | -2.8311  | 1.573216 | 1.153253 |
| 16 | -2.58218 | 7.975243 | 0.030497 |
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| 8  | -1.43393 | 8.356065 | -0.79097 |

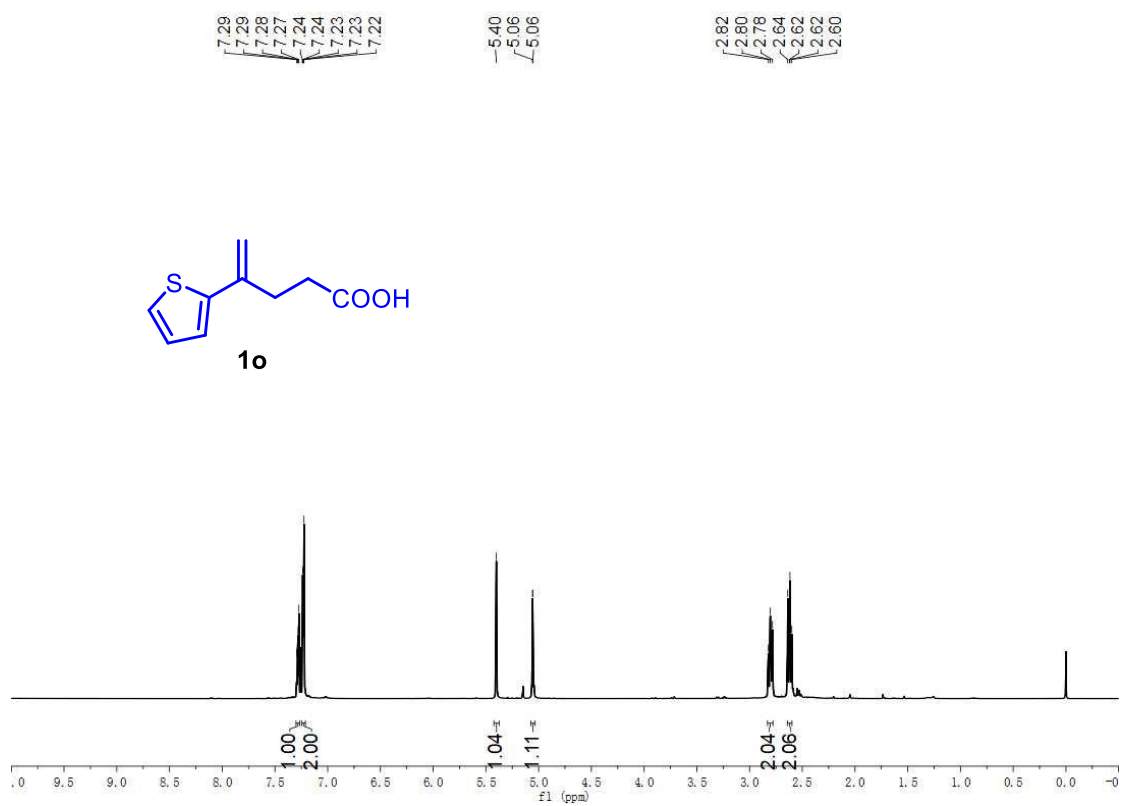
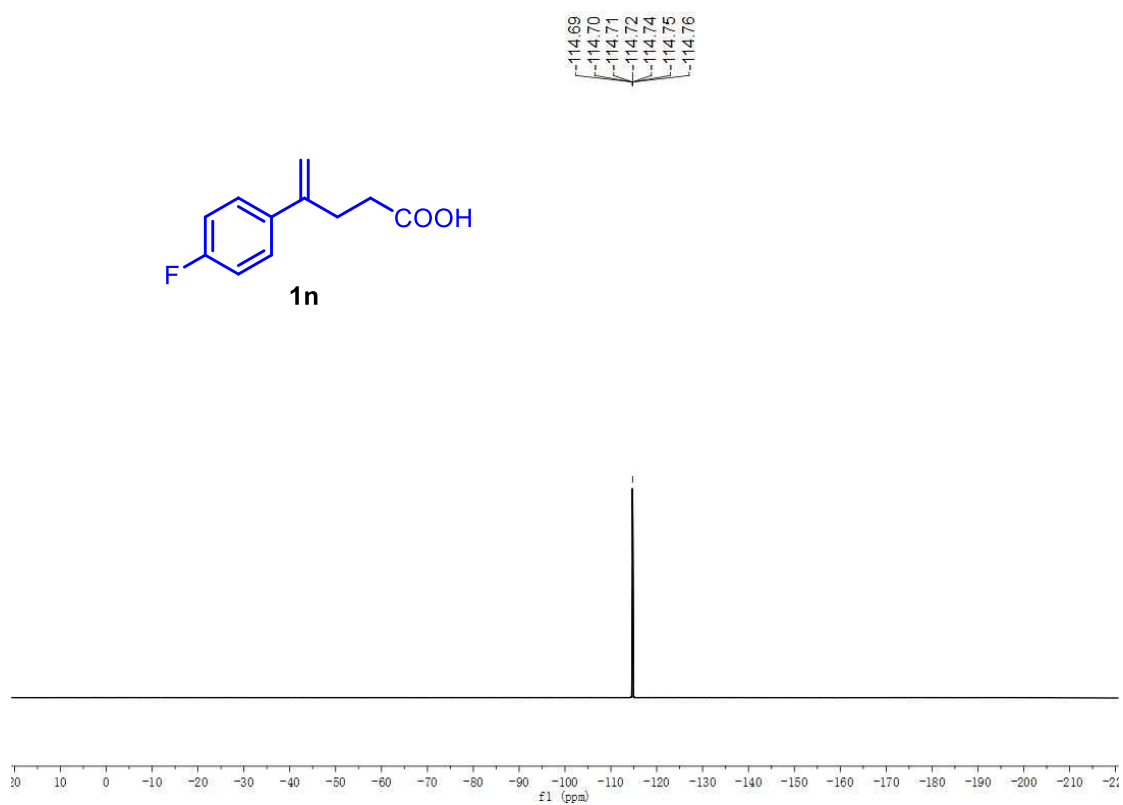
## 9. NMR spectra of compounds 1, 2 and 3

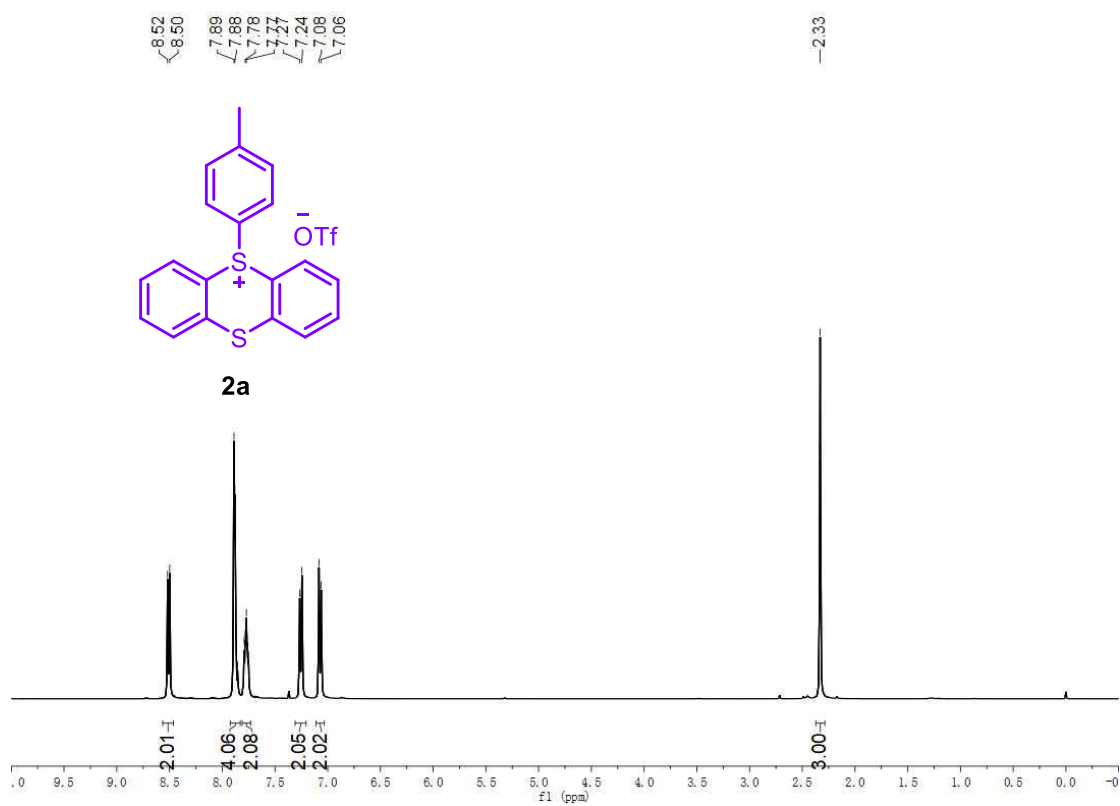
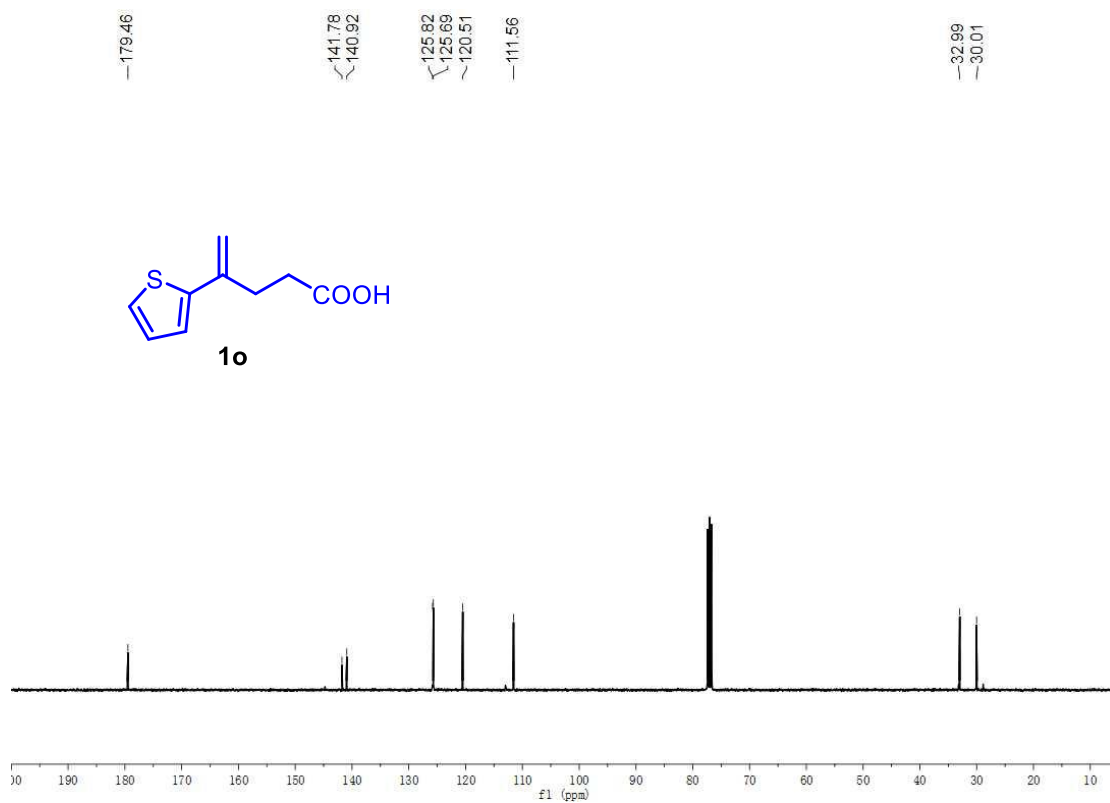


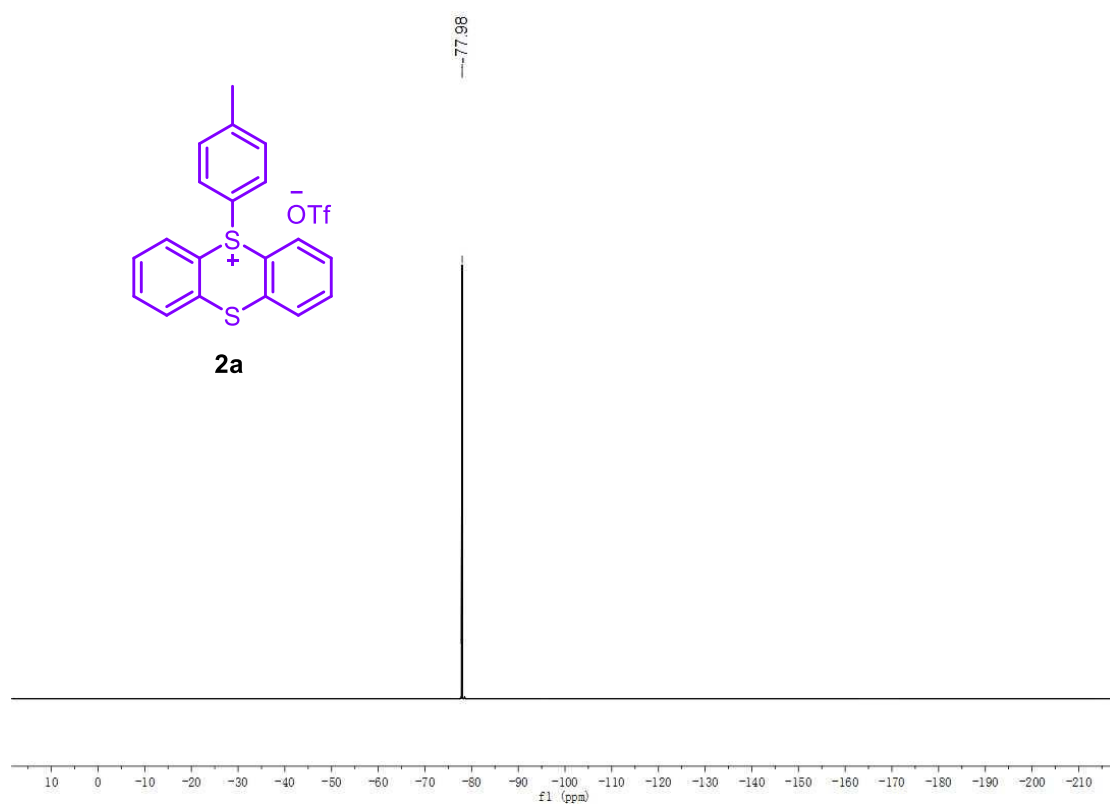
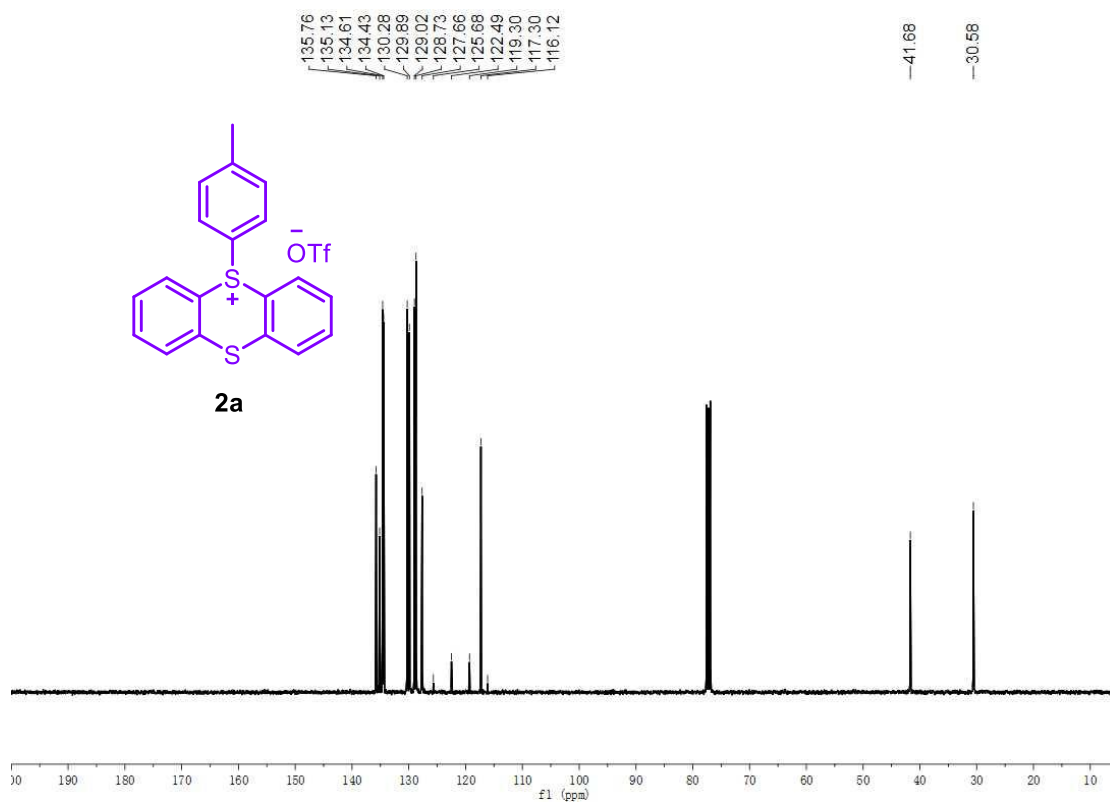


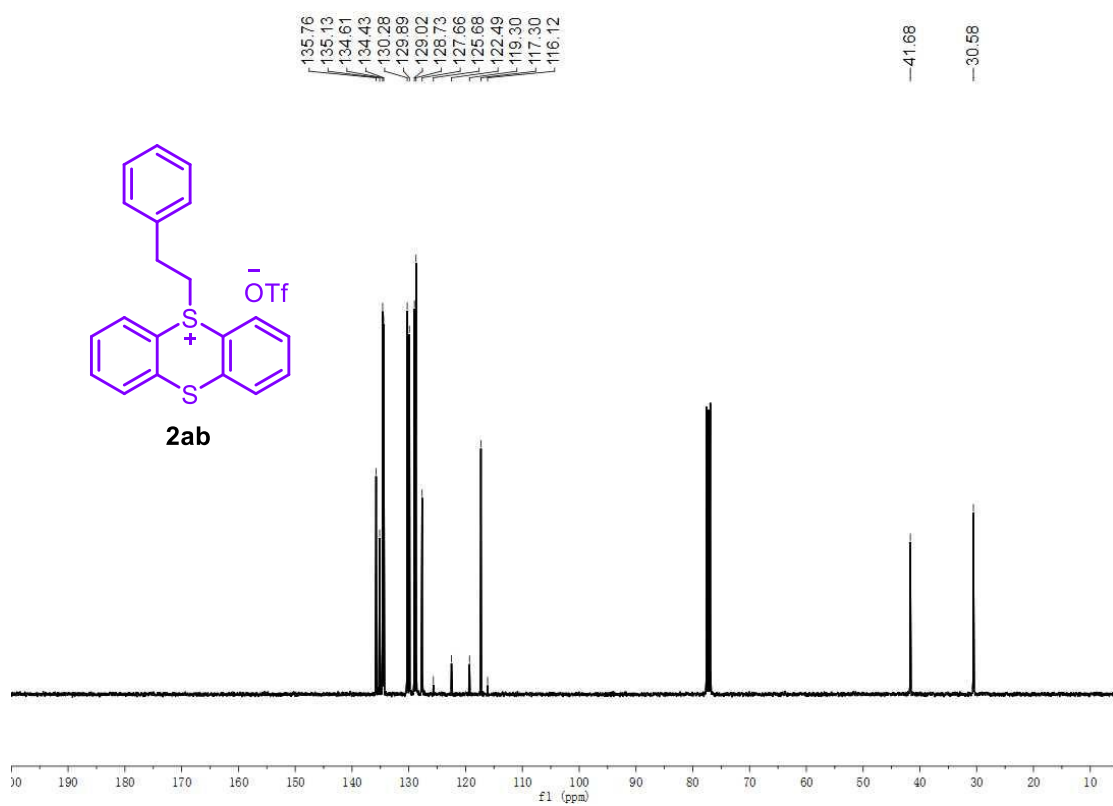
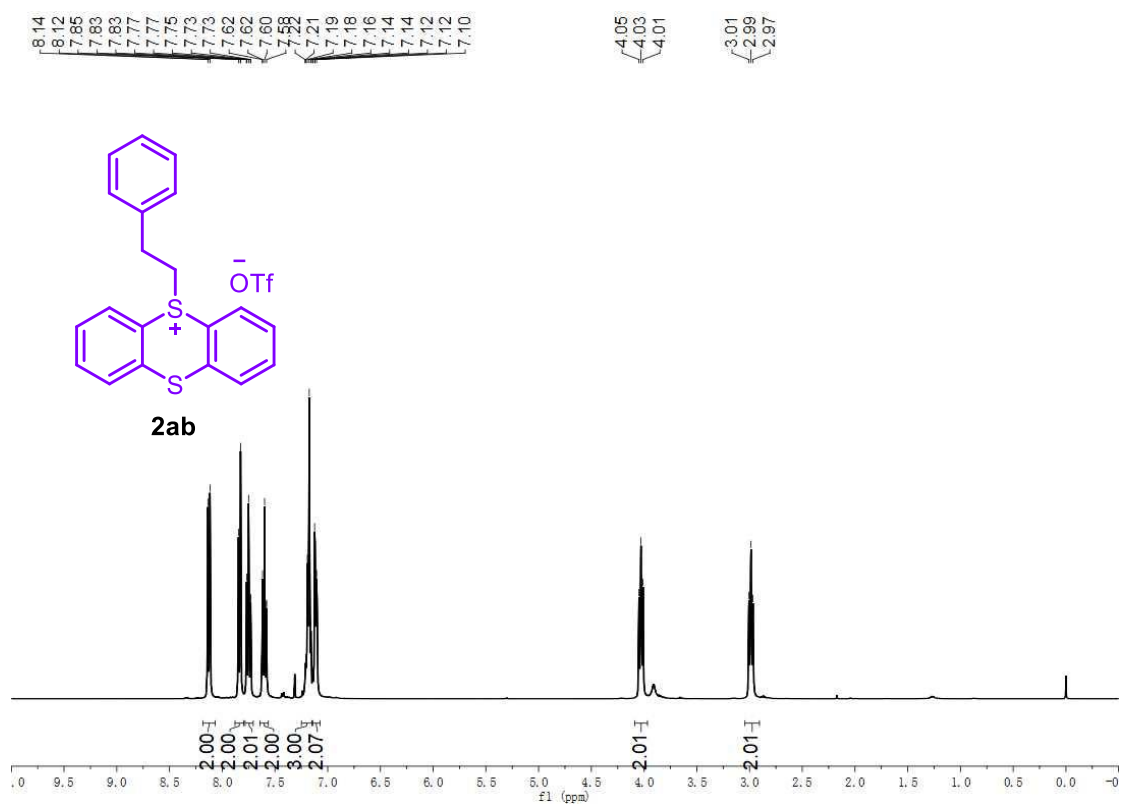


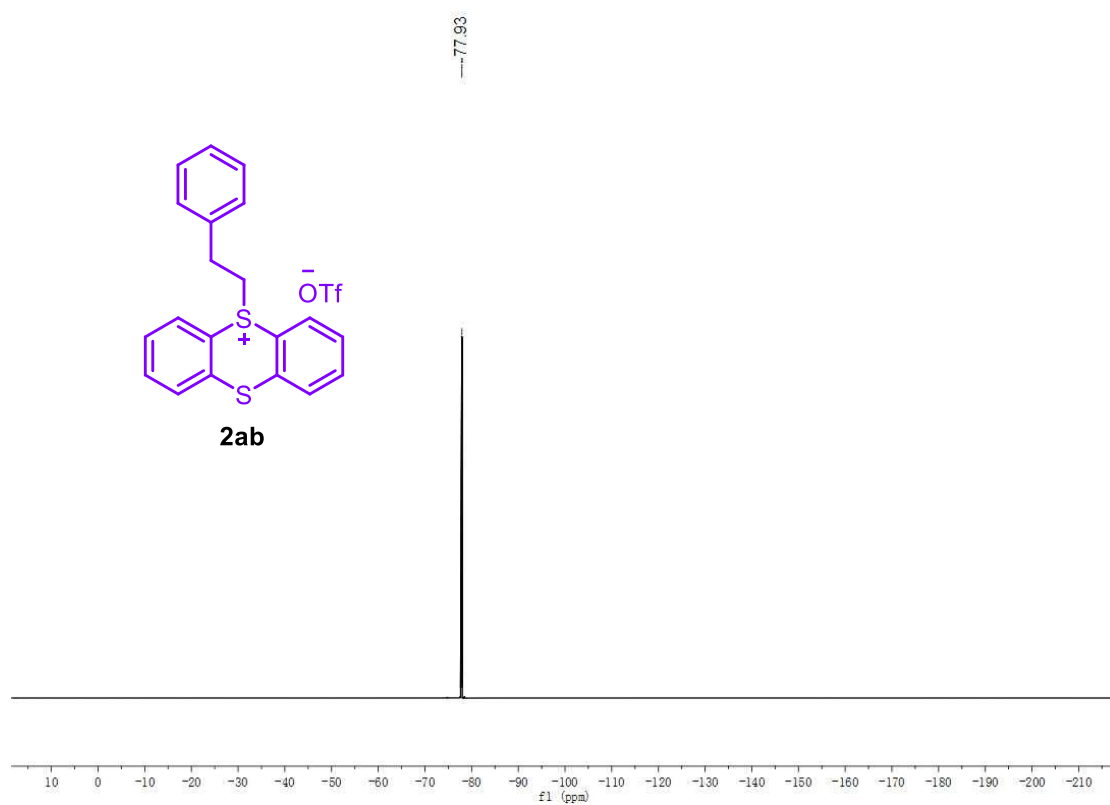


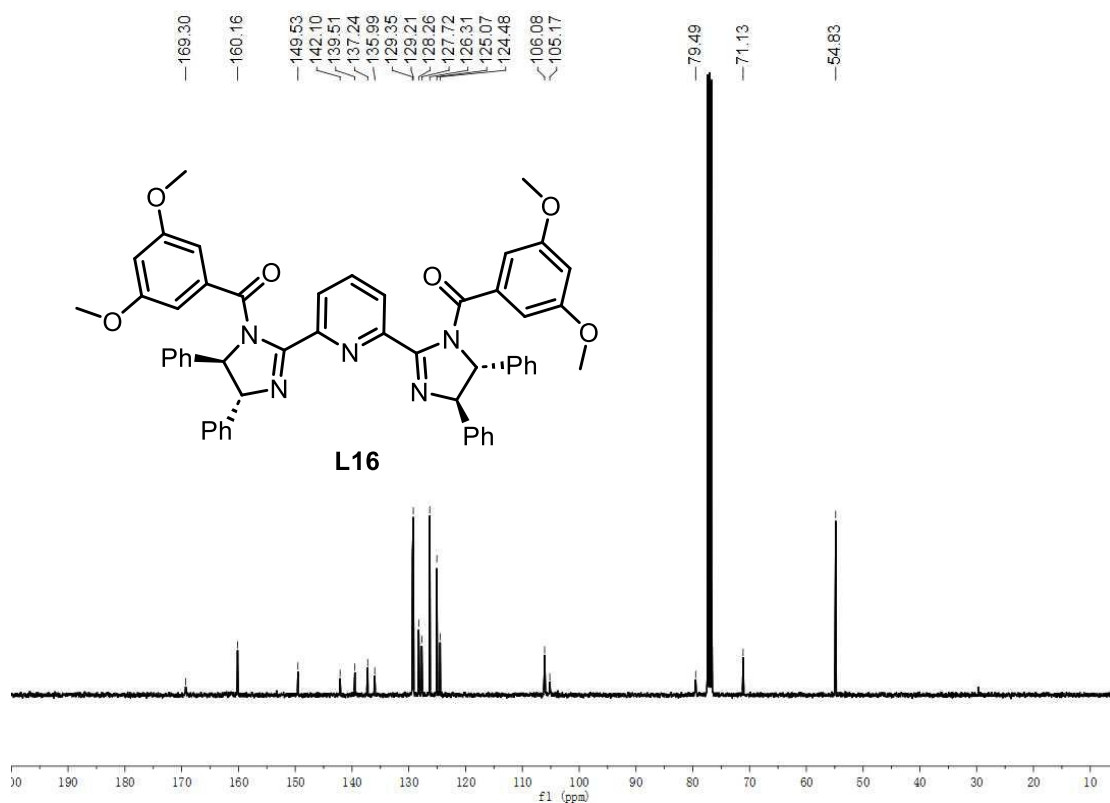
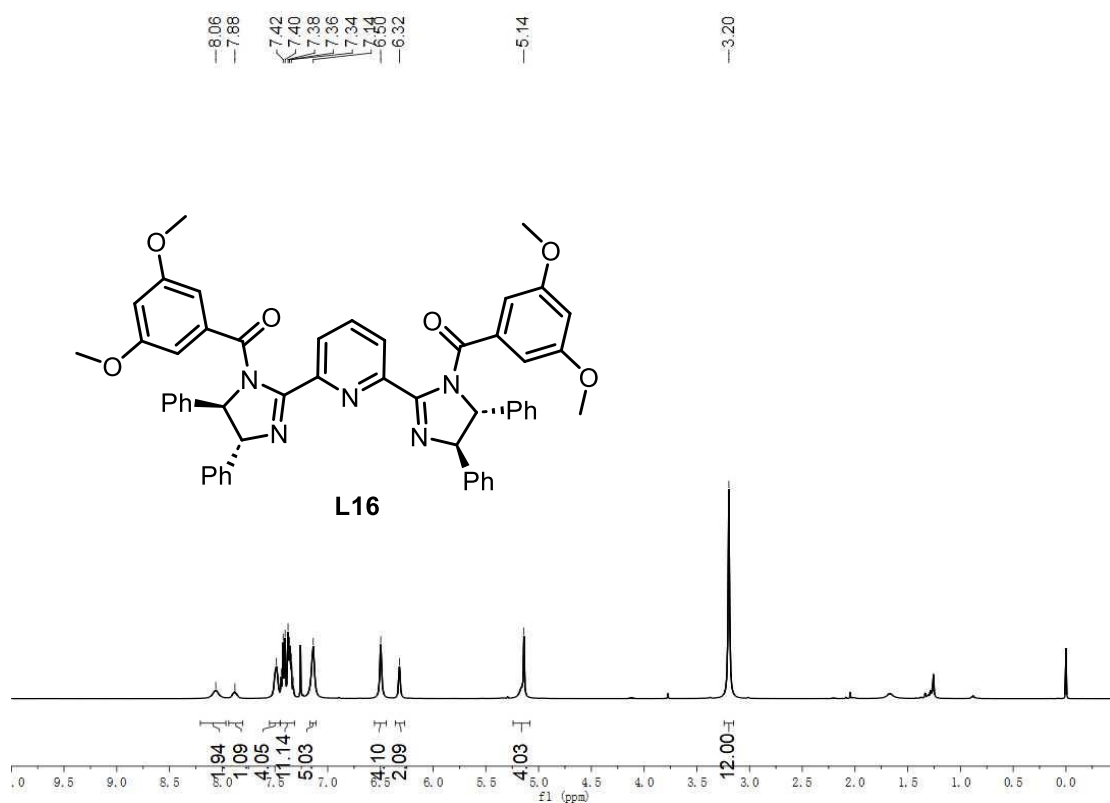


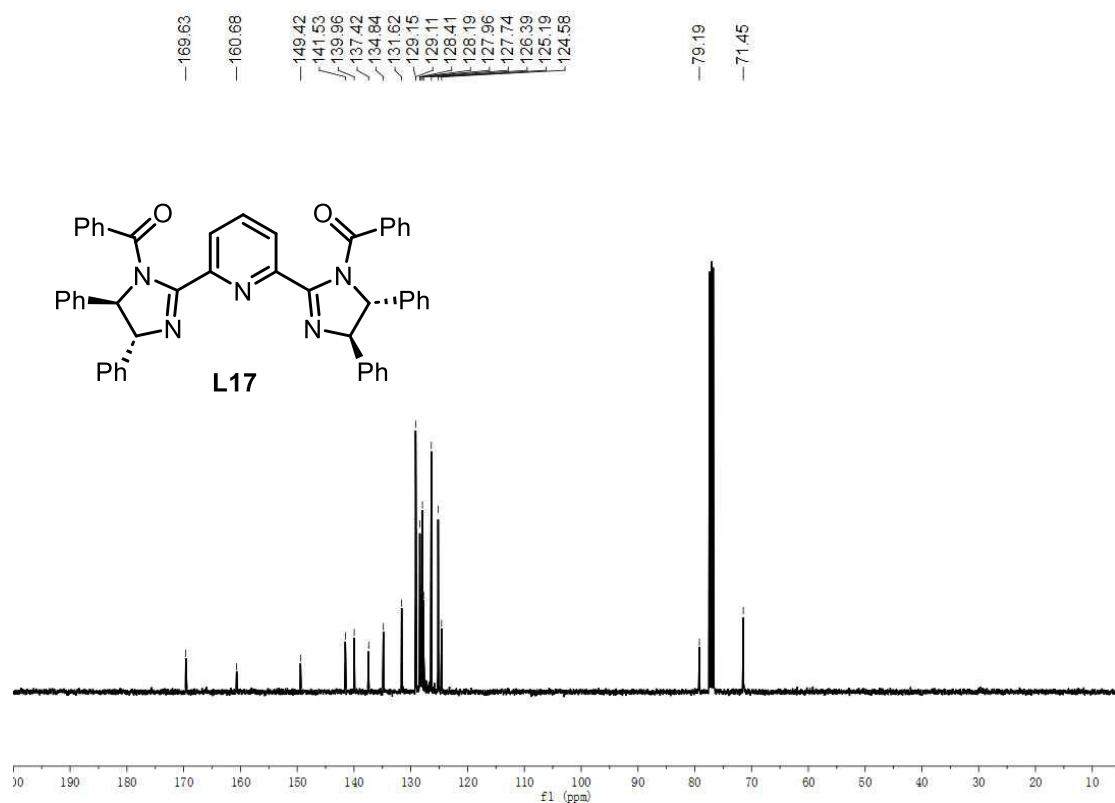
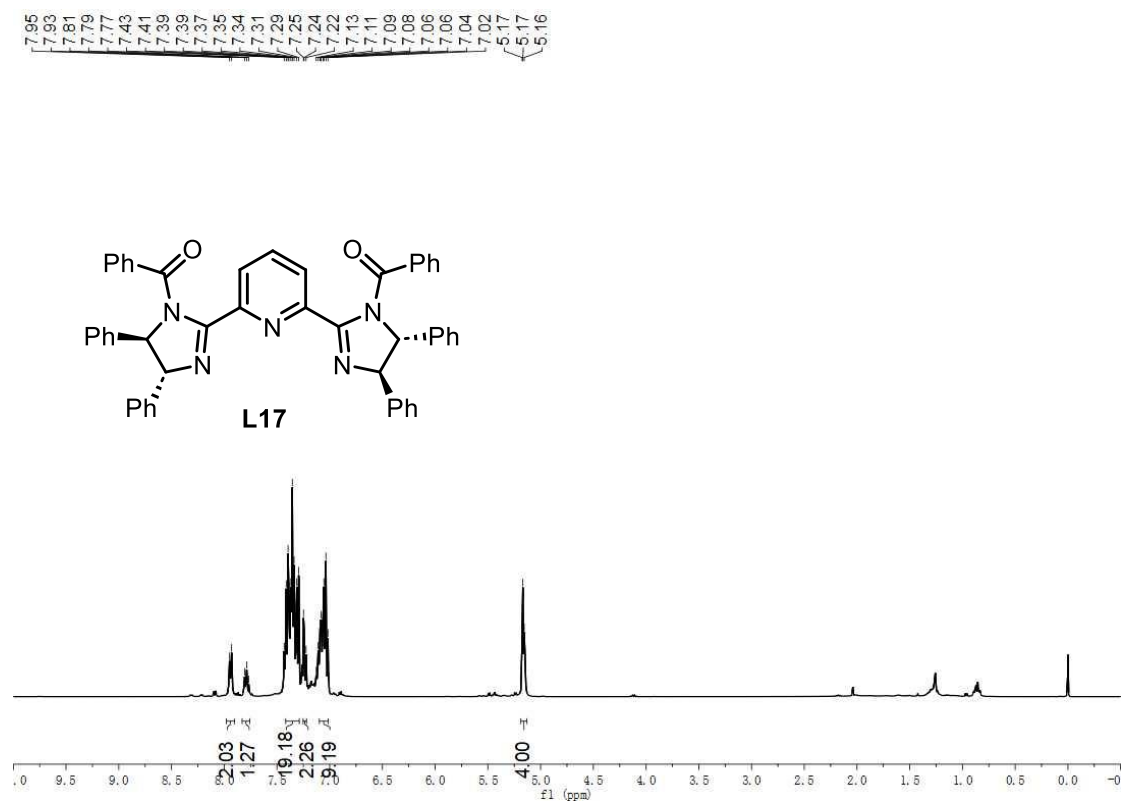


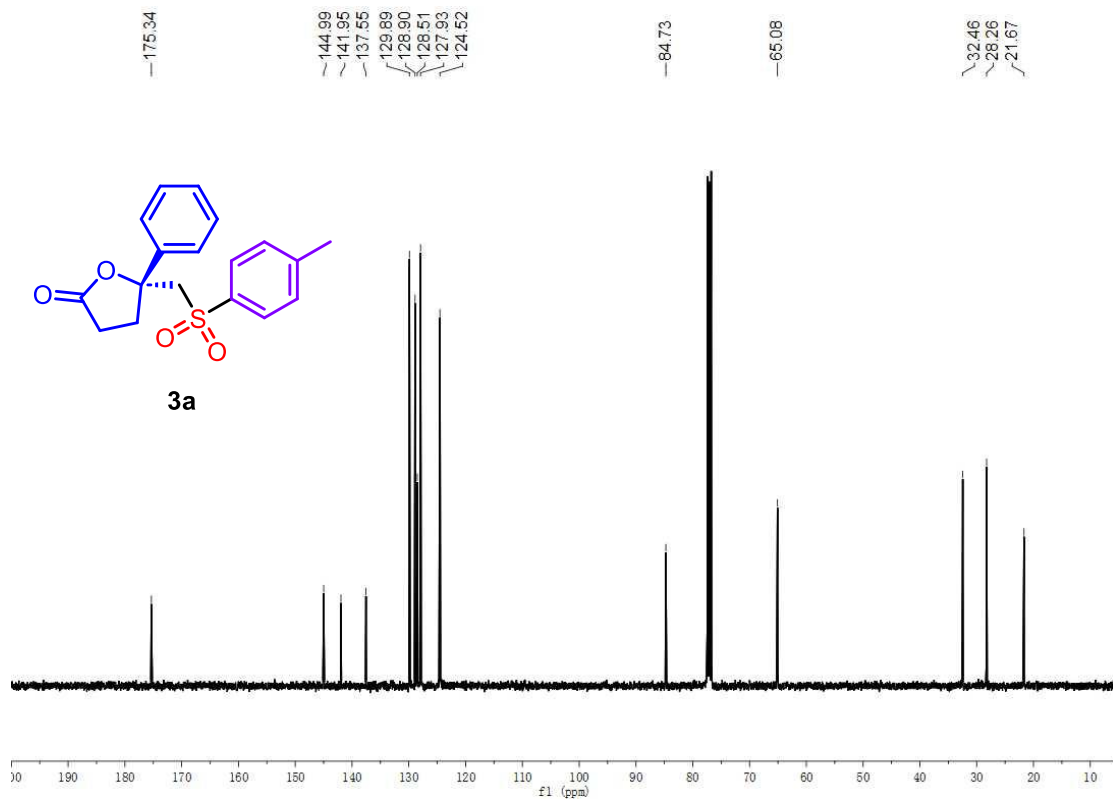
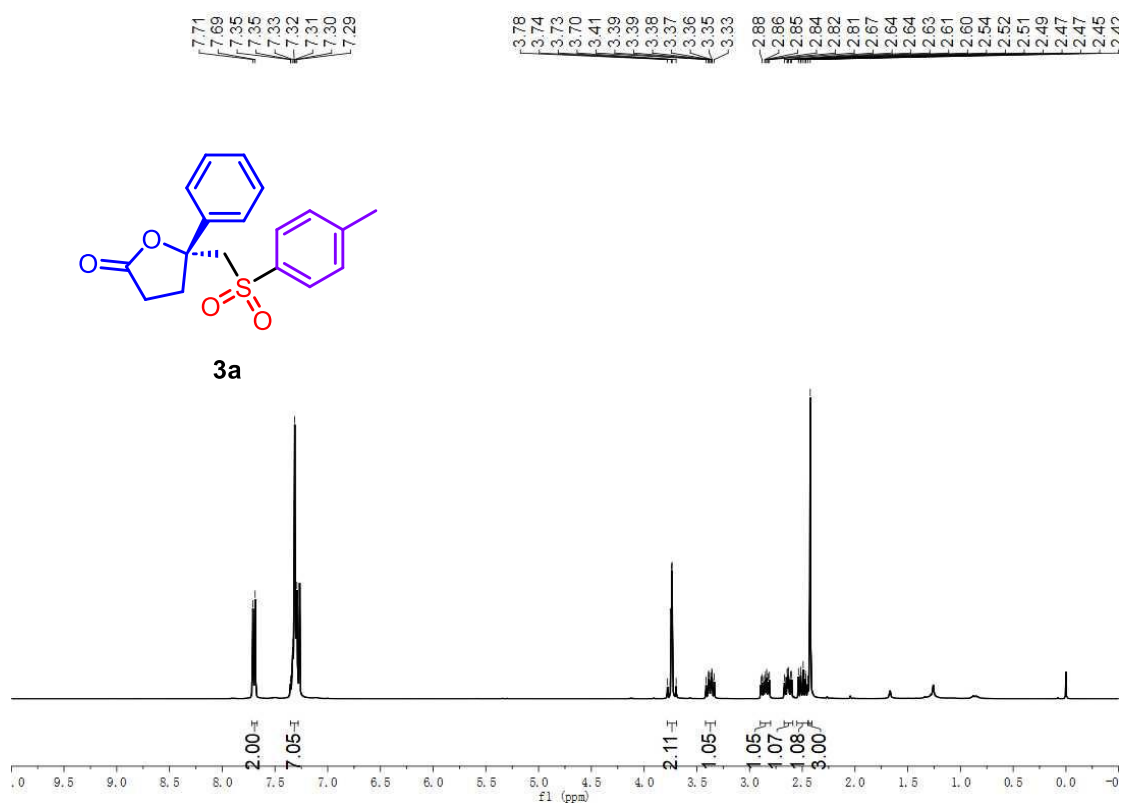


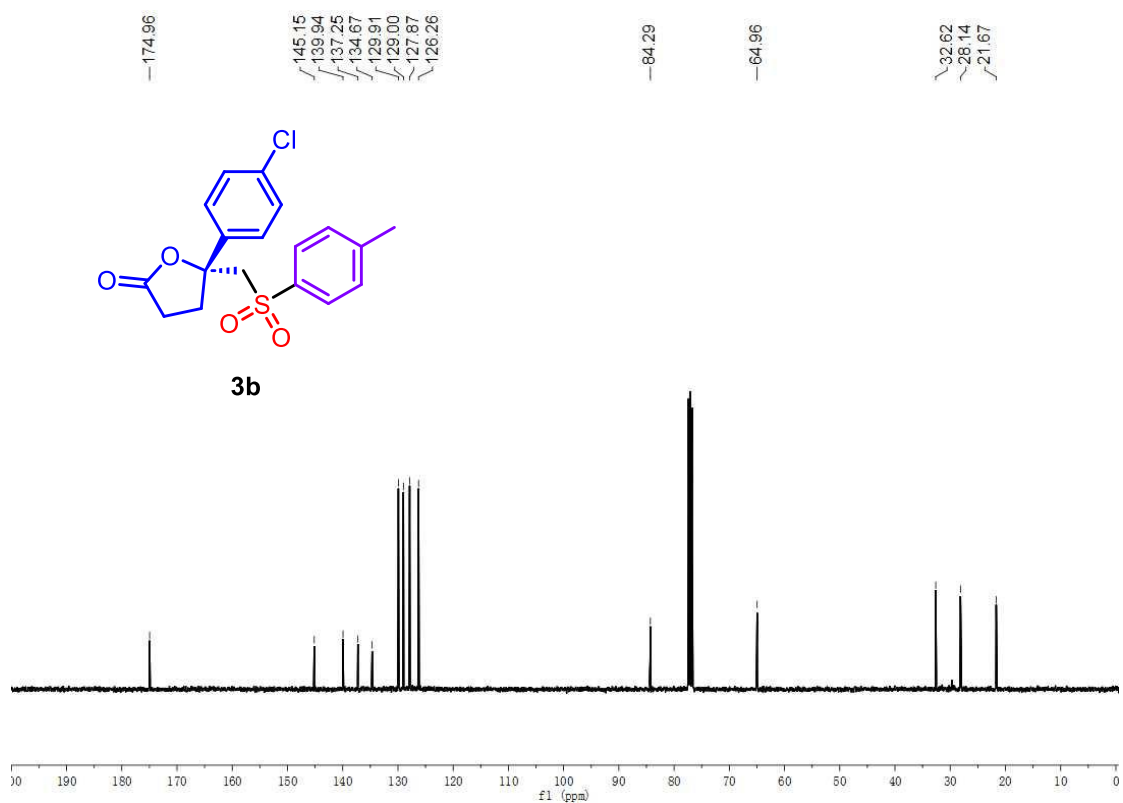
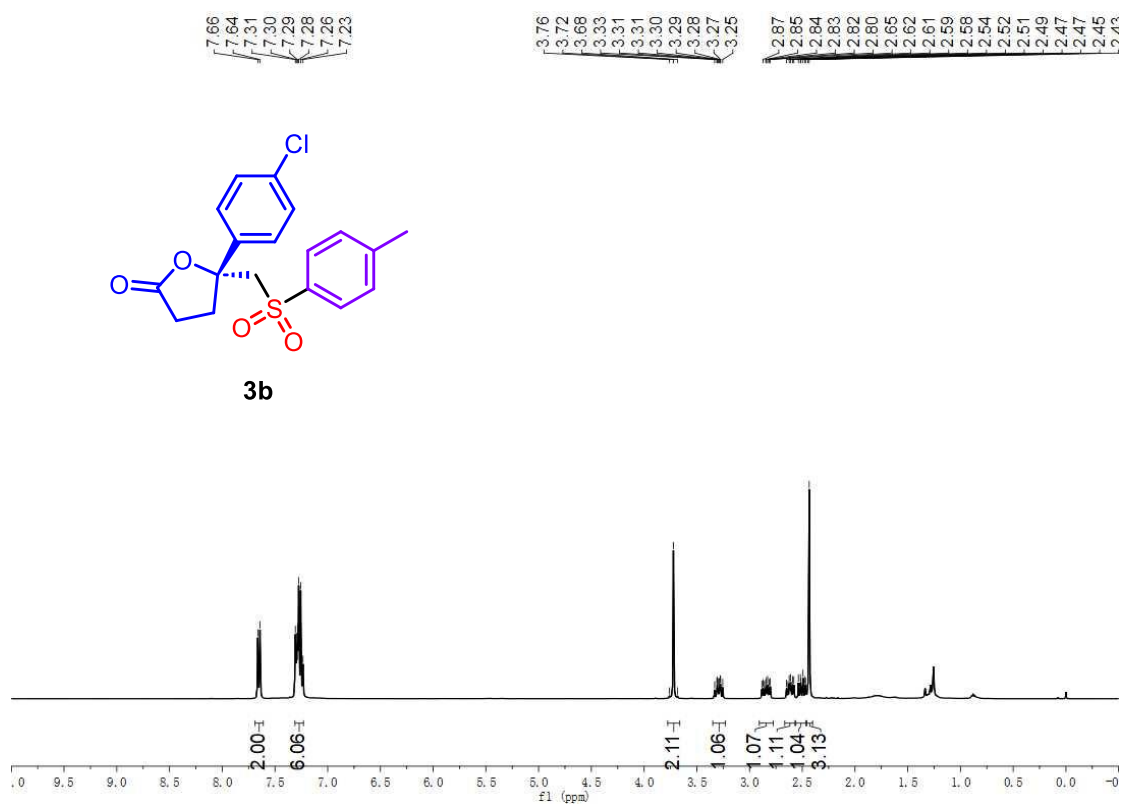


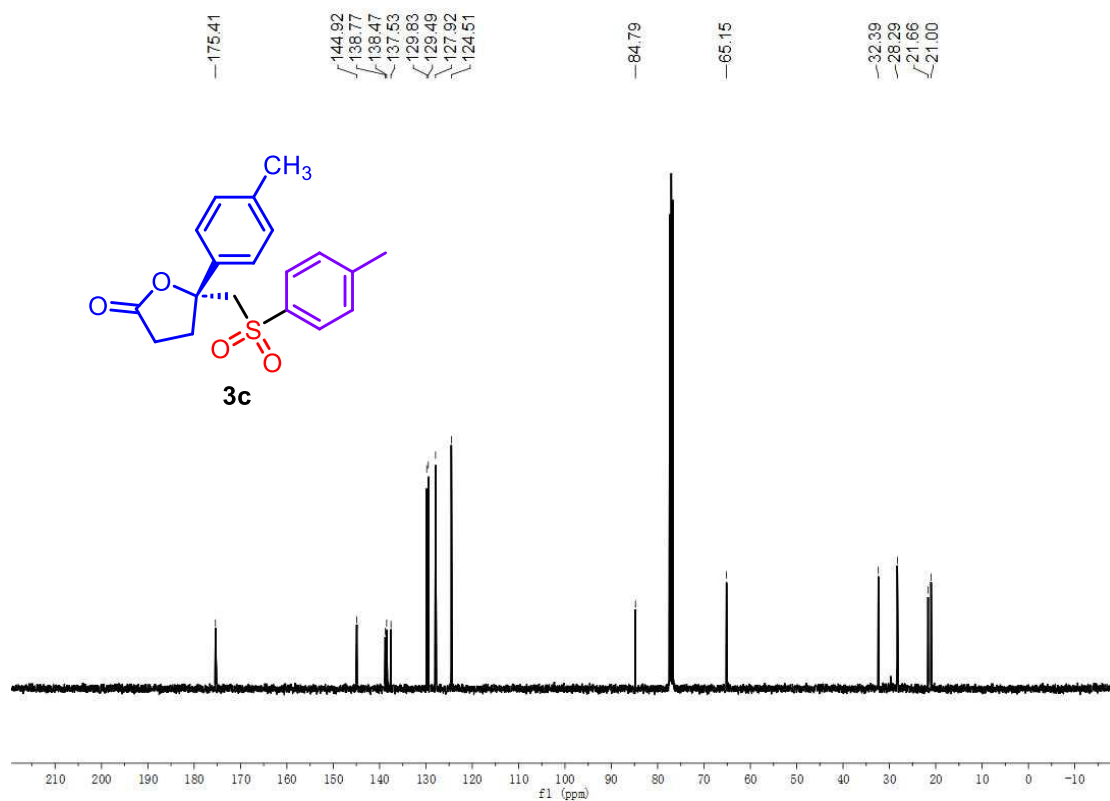
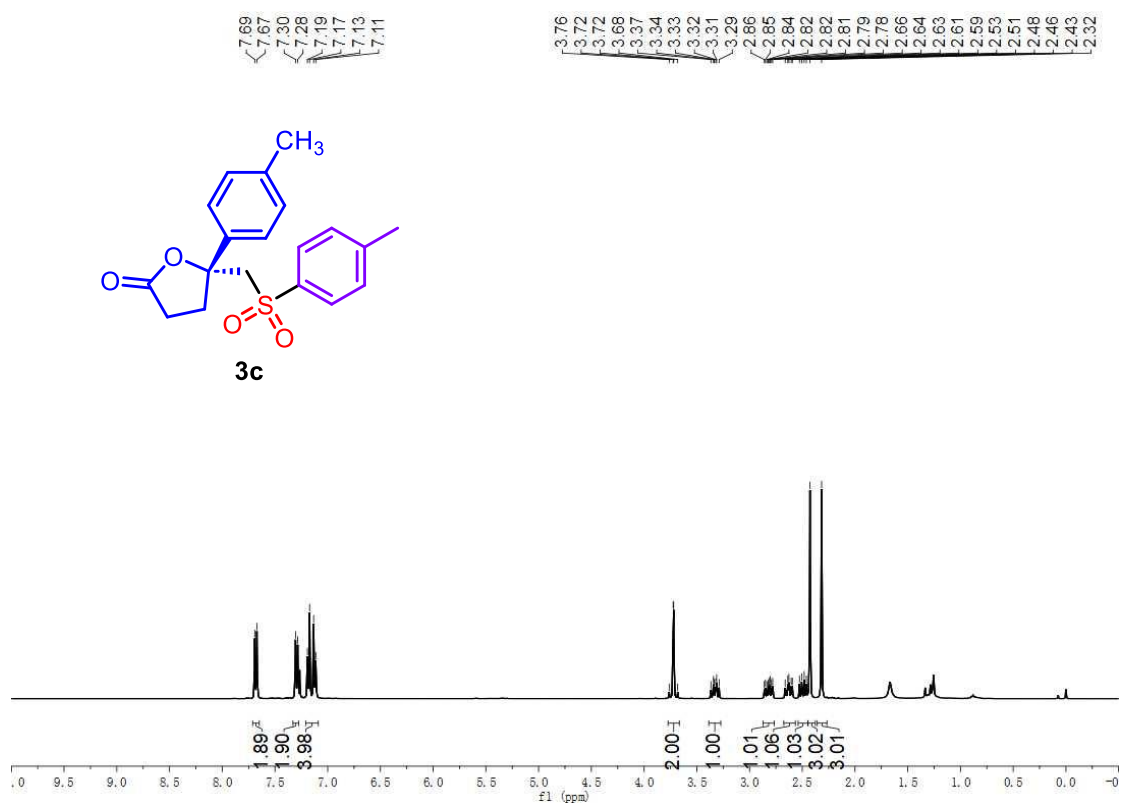


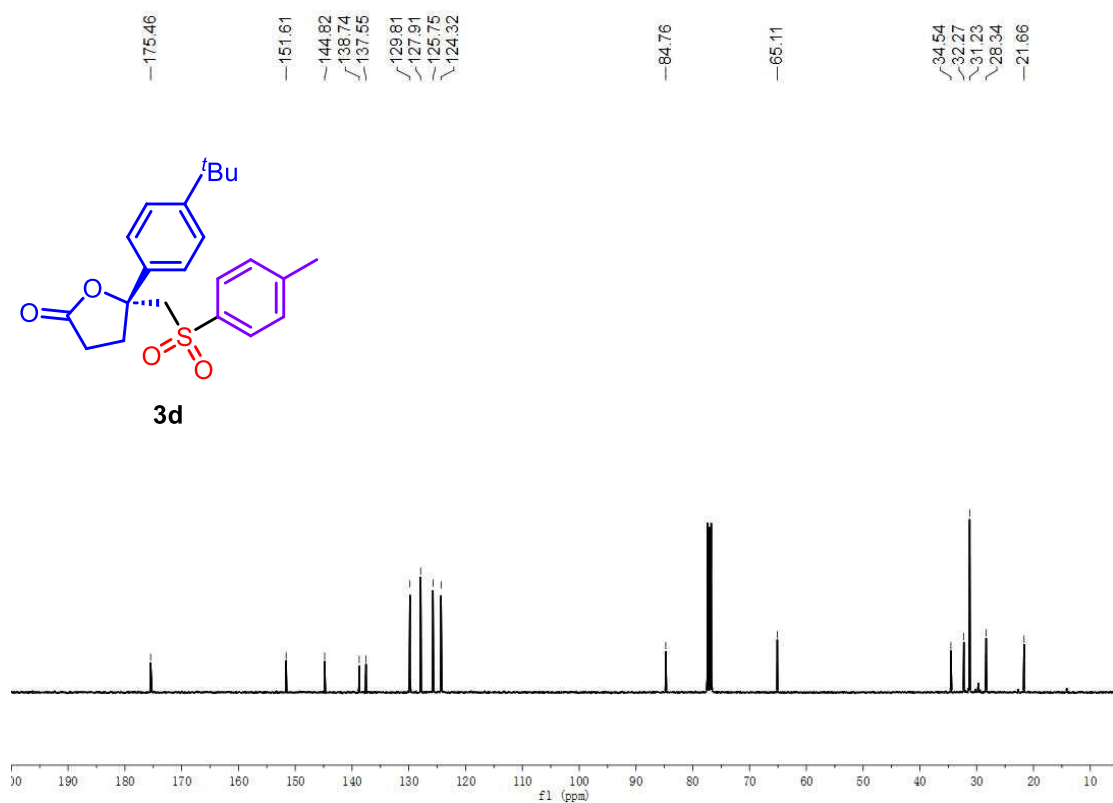
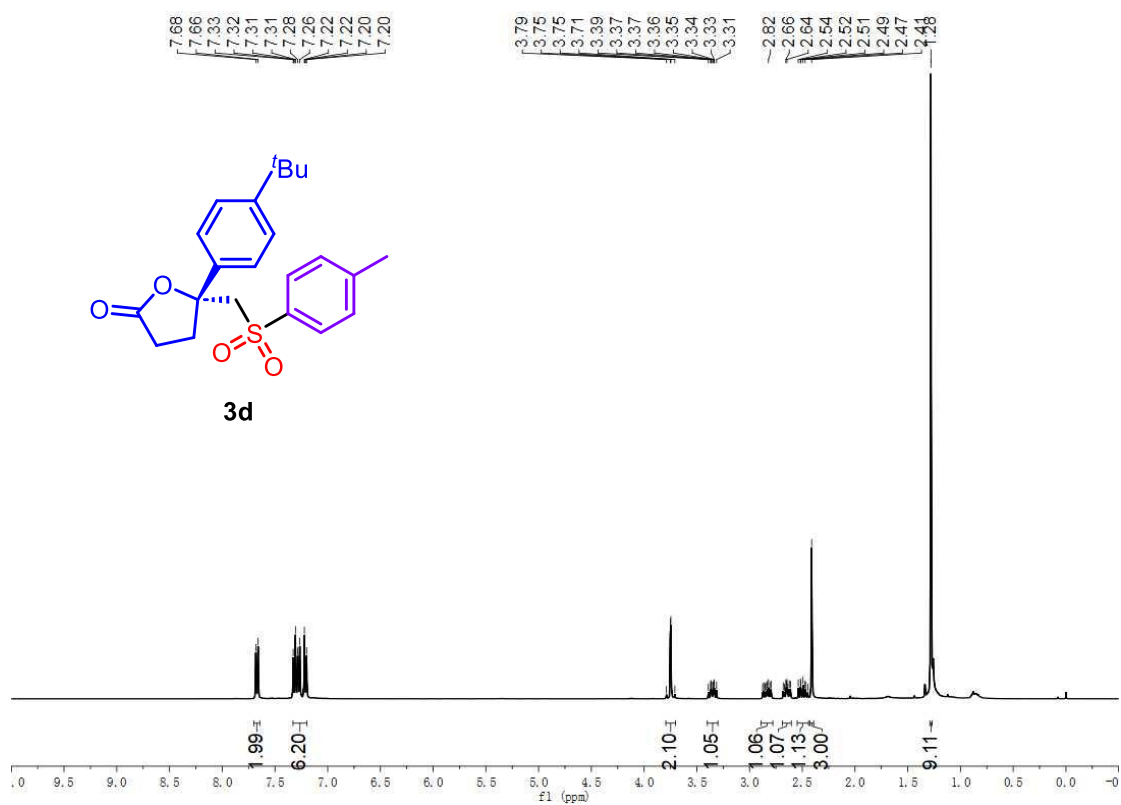


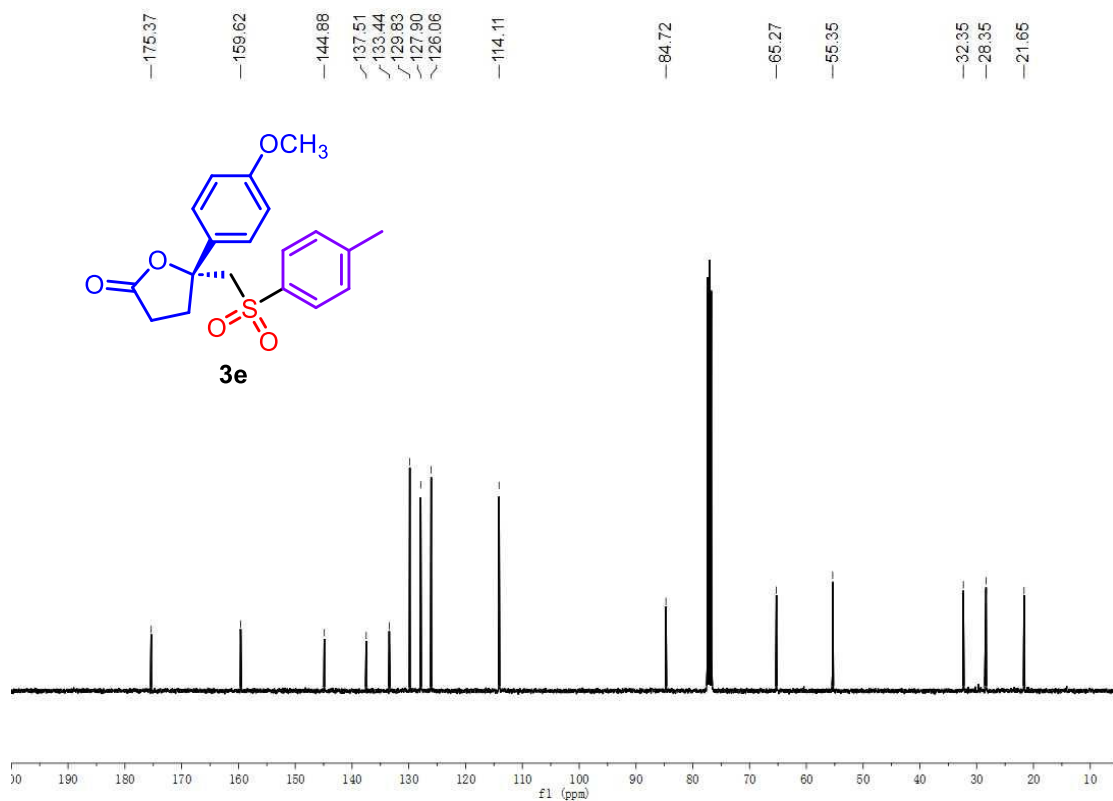
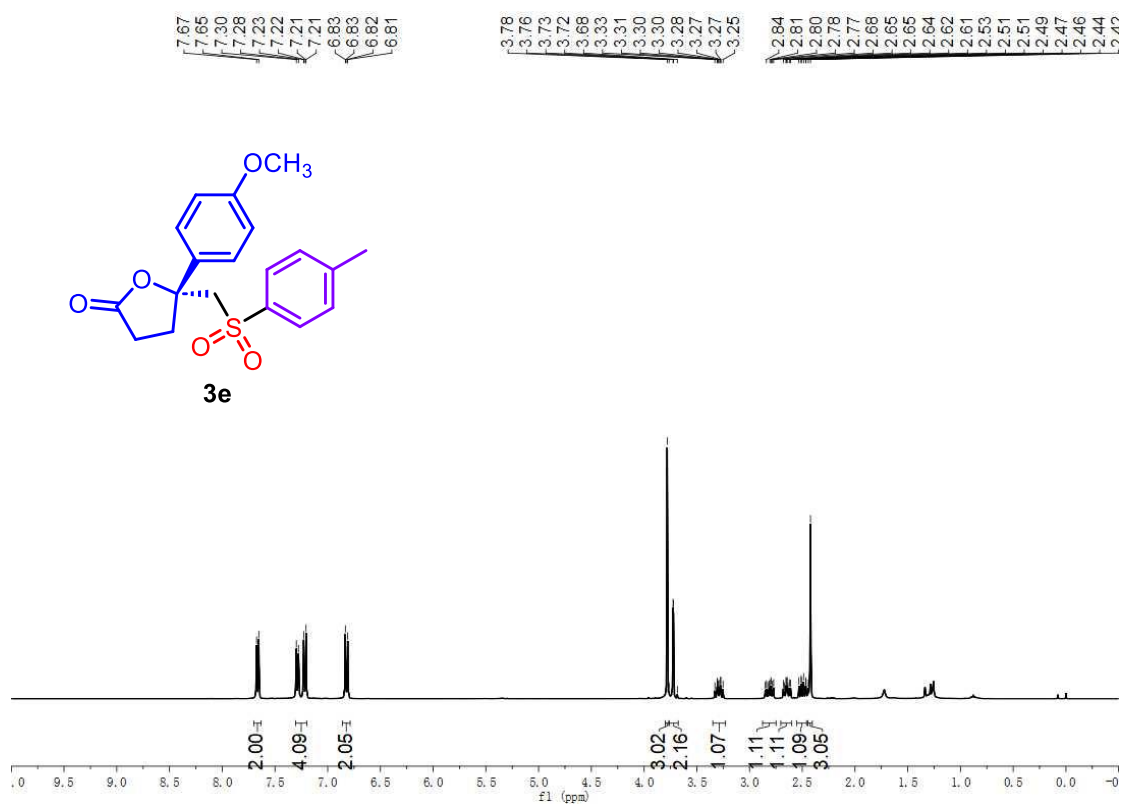


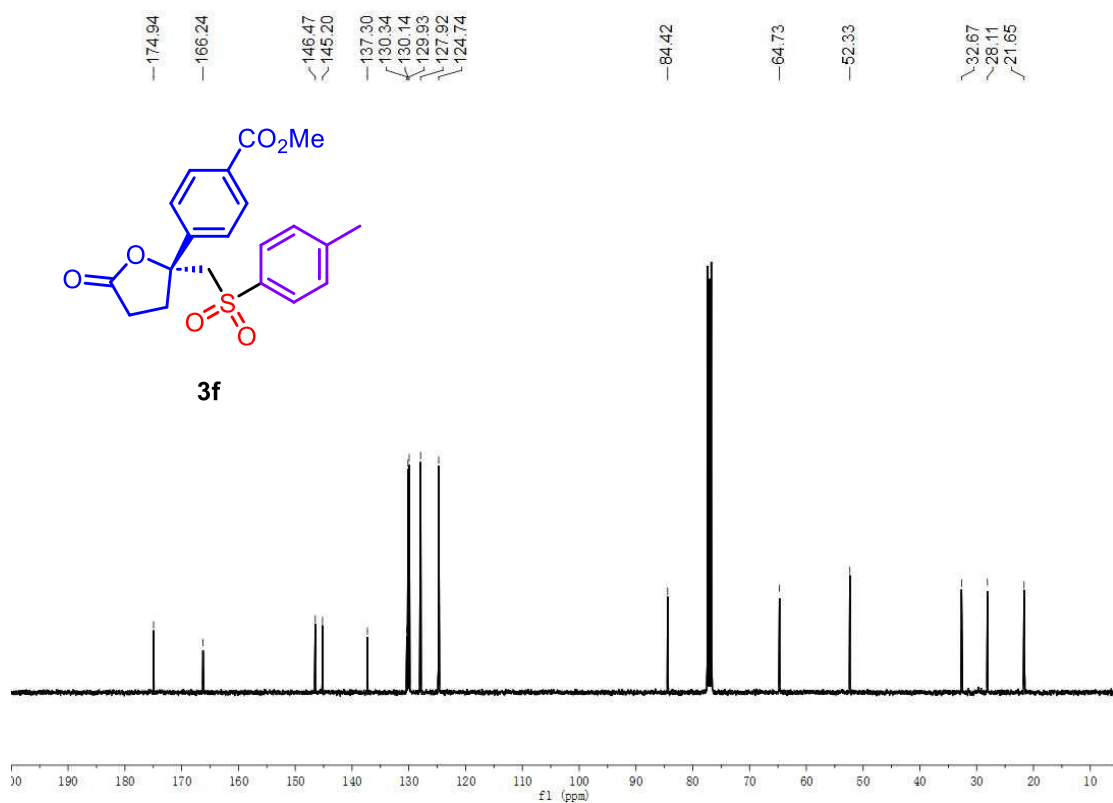
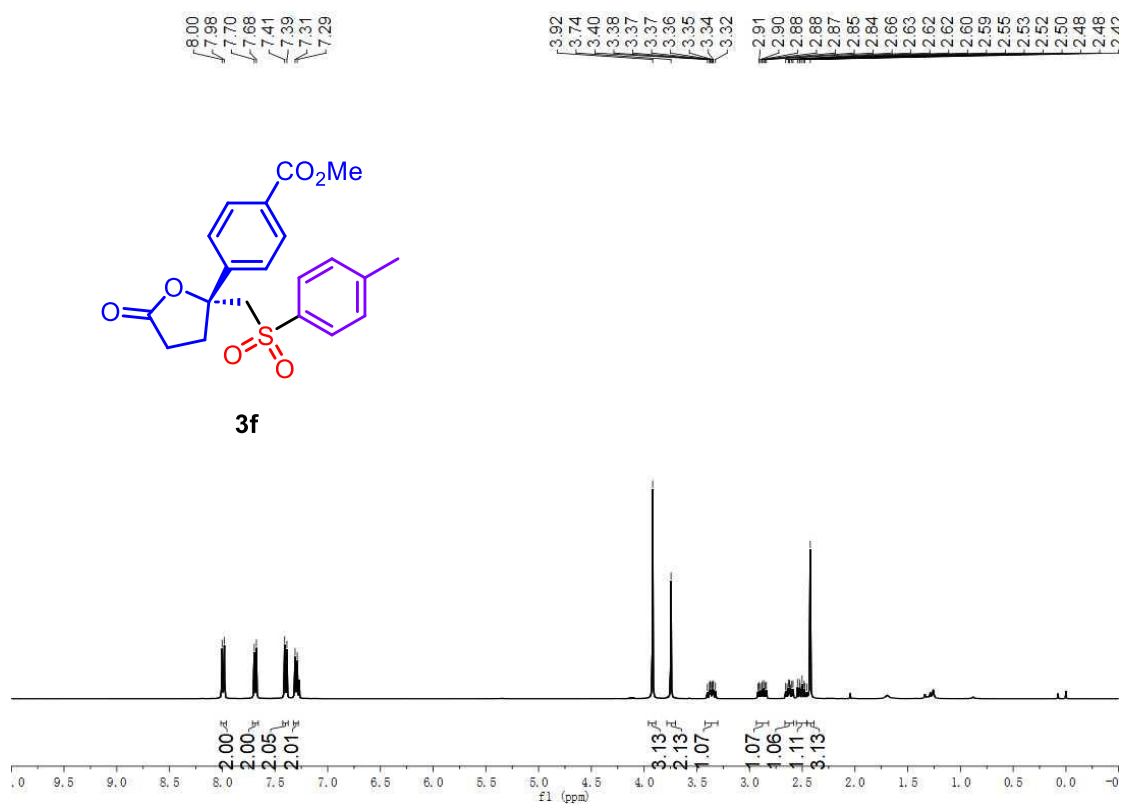


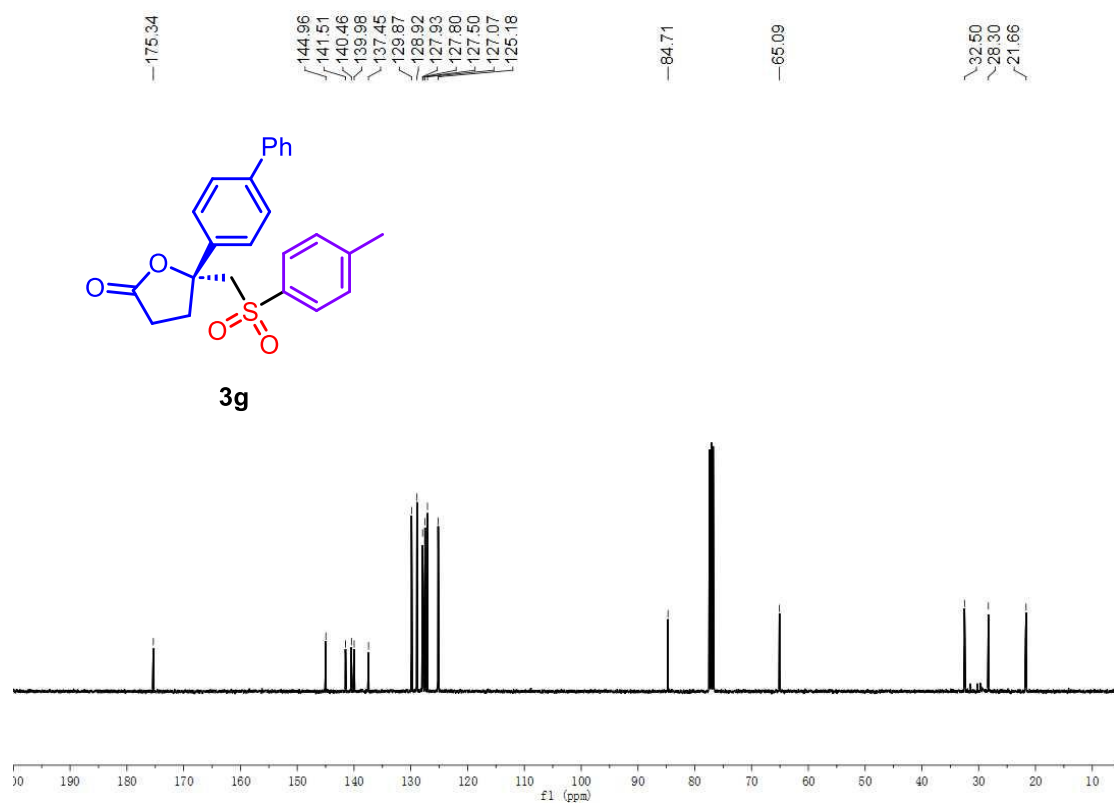
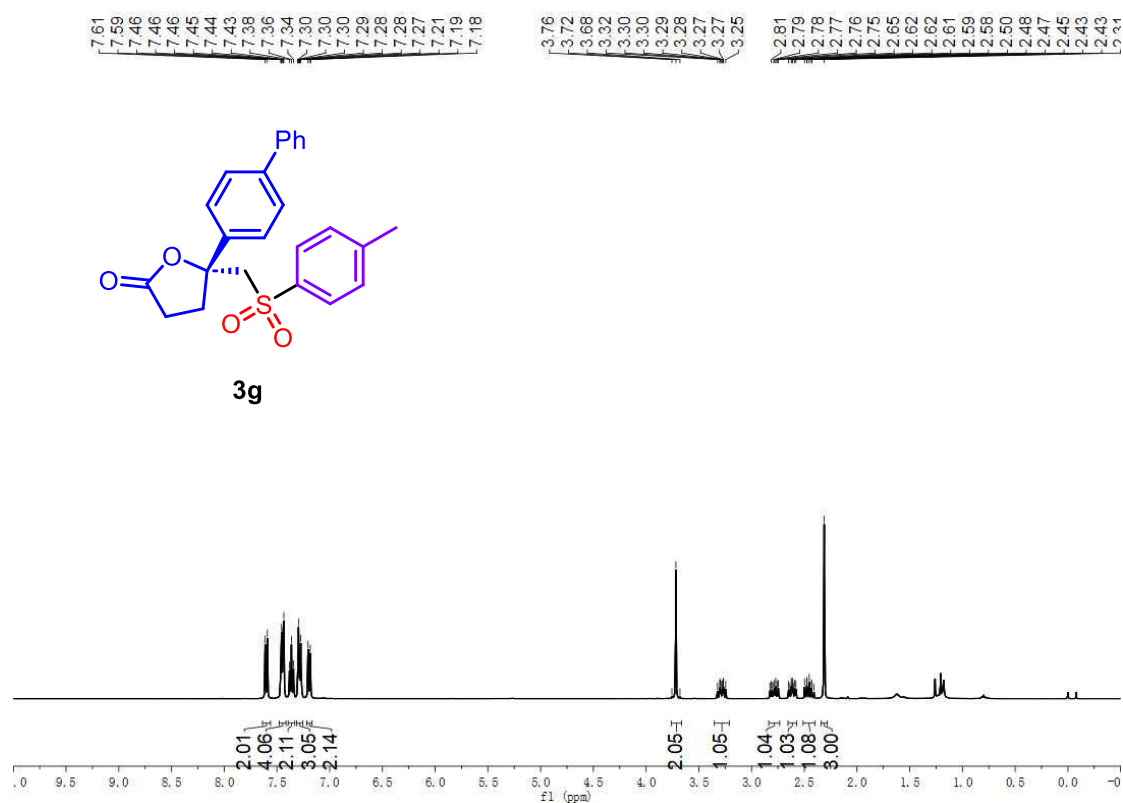


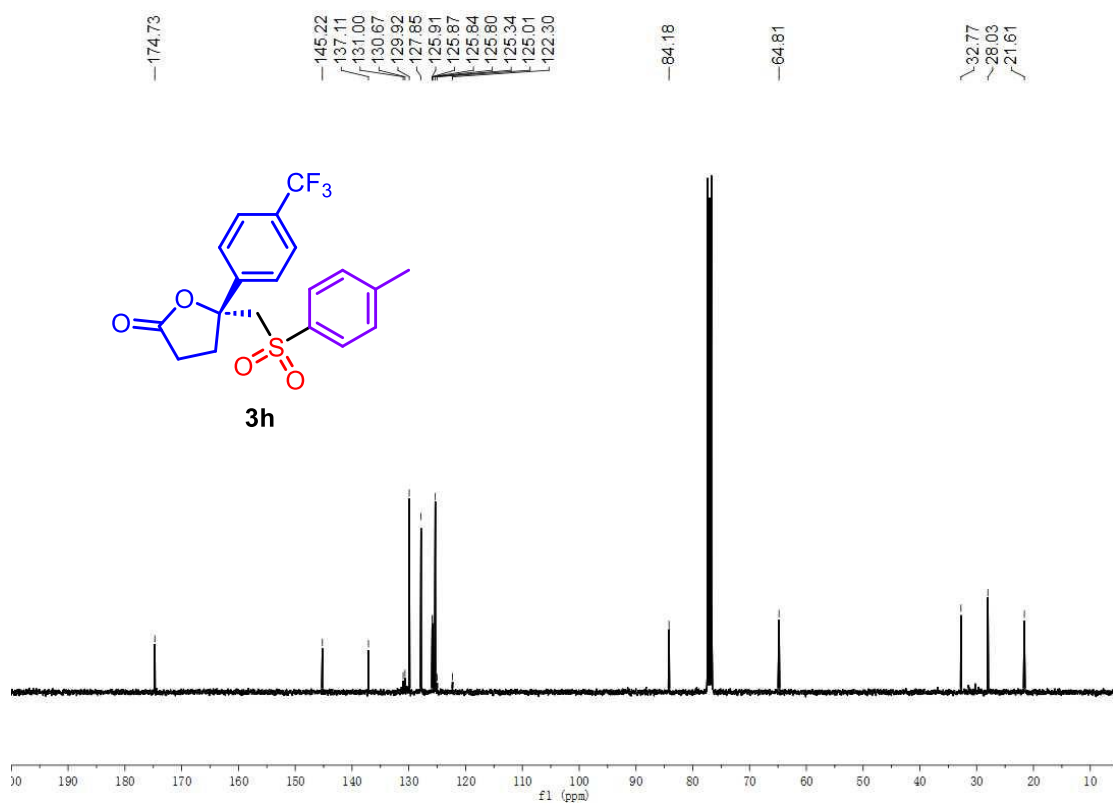
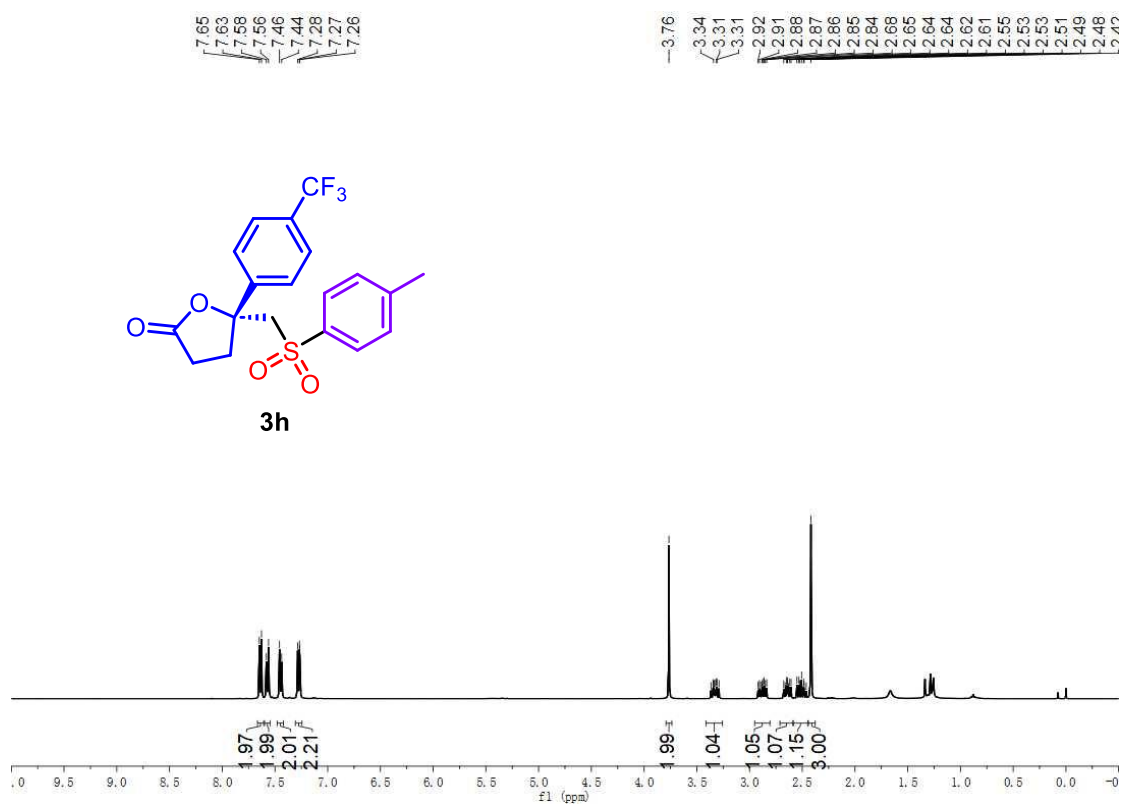


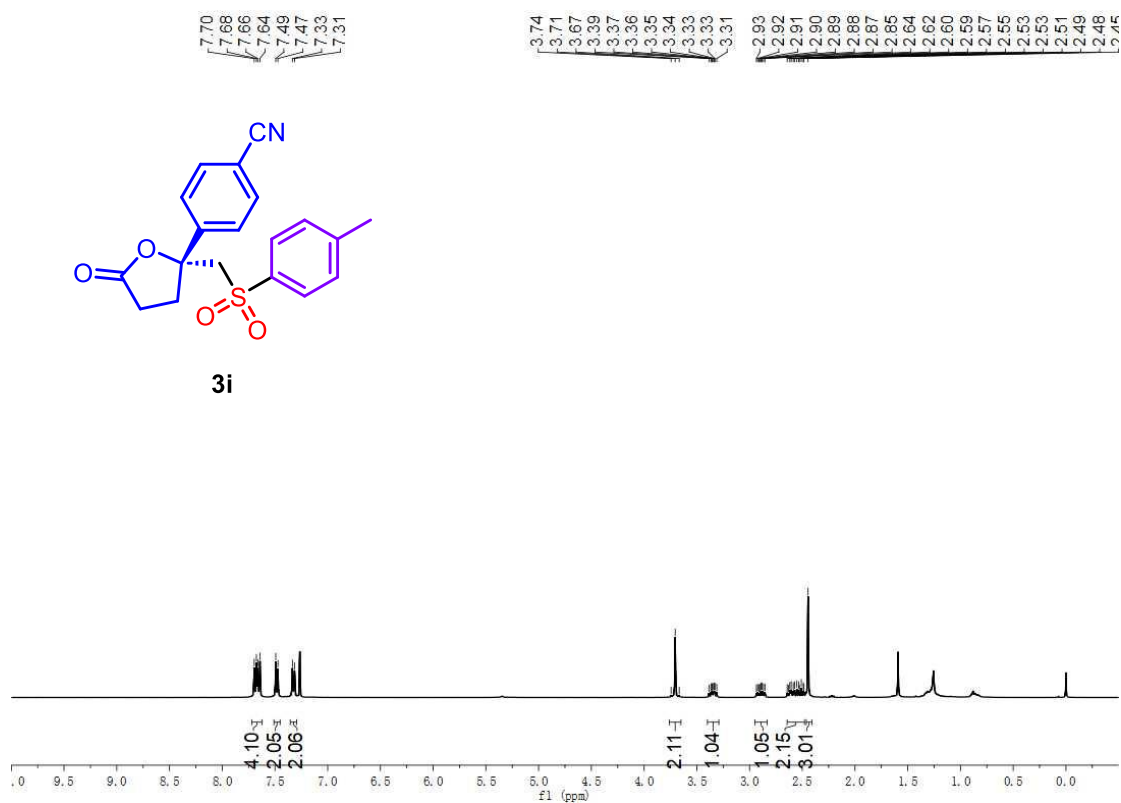
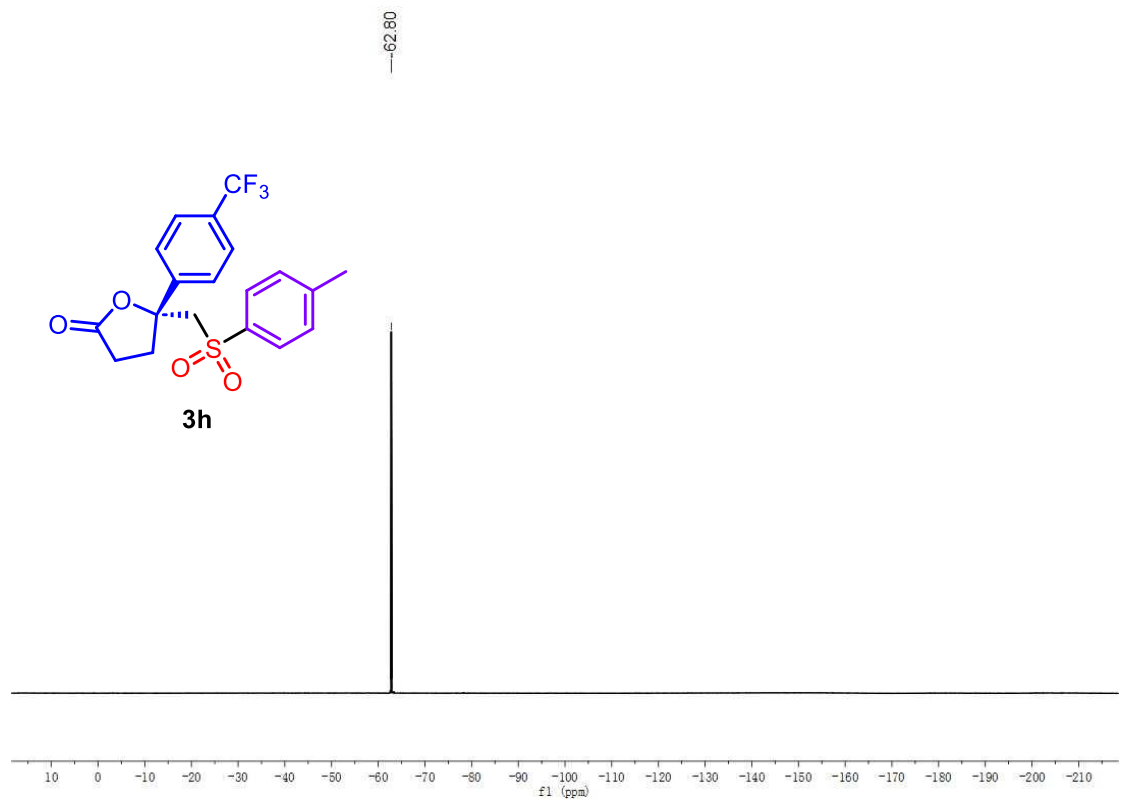


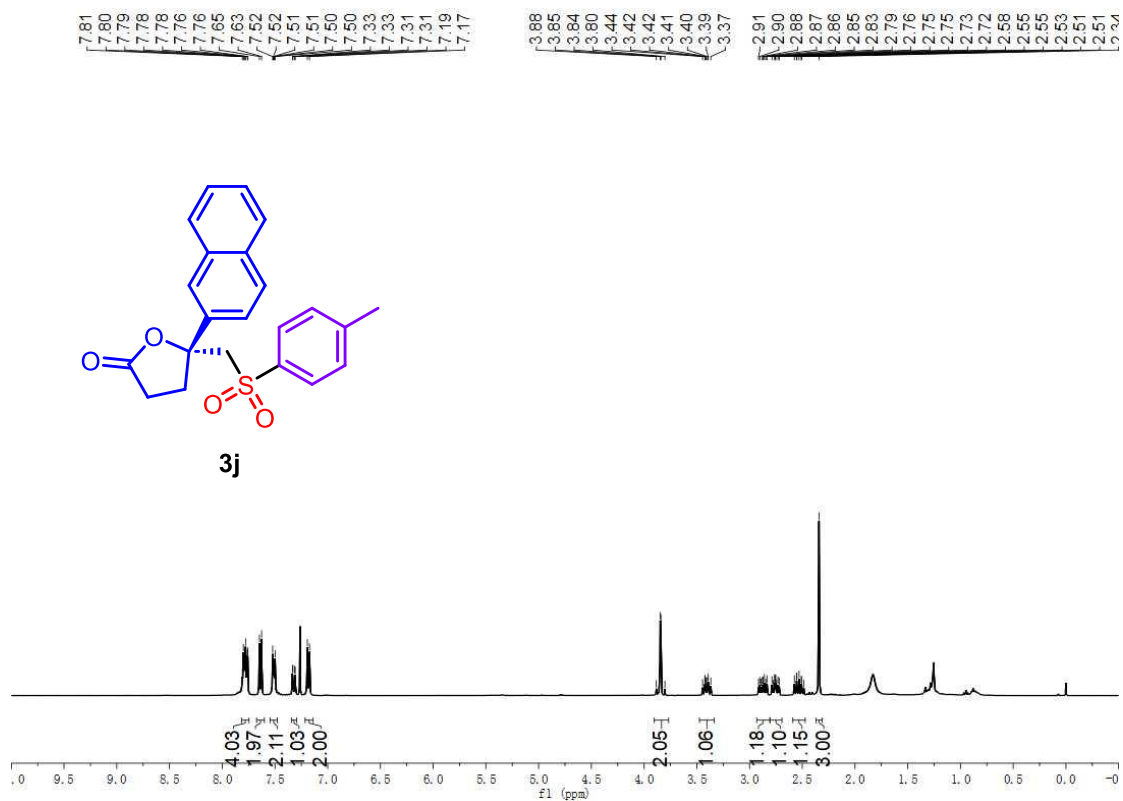
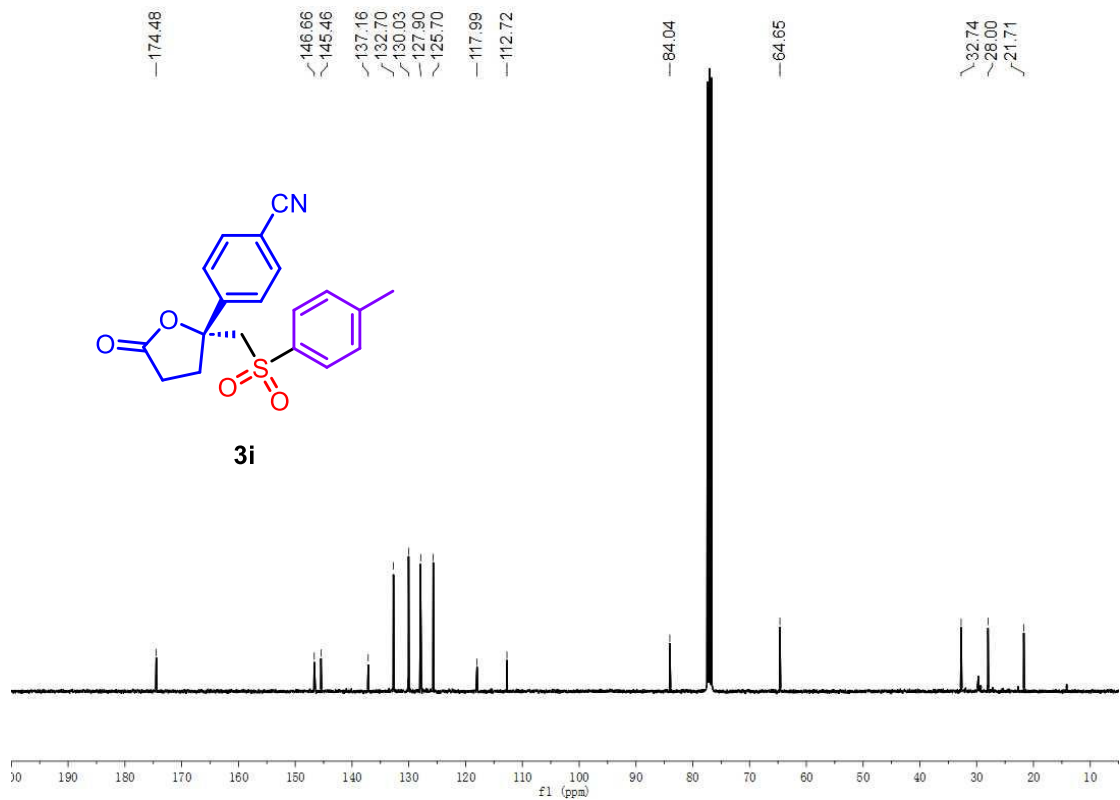


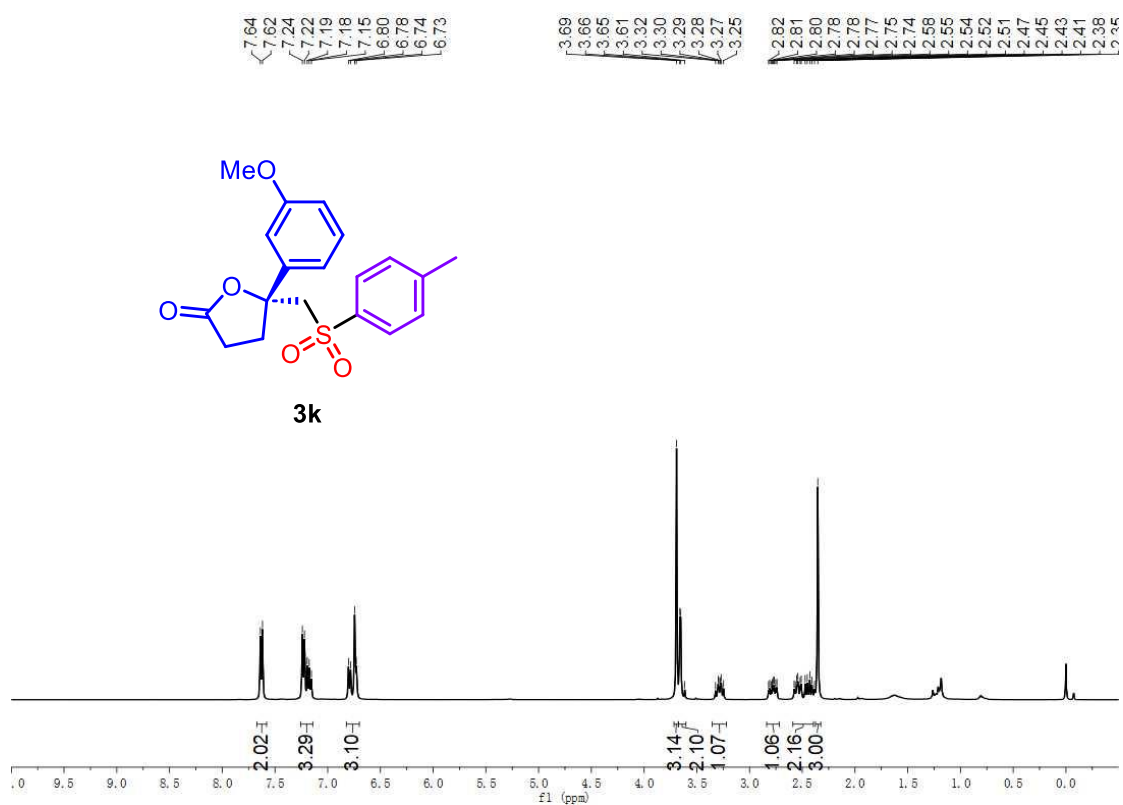
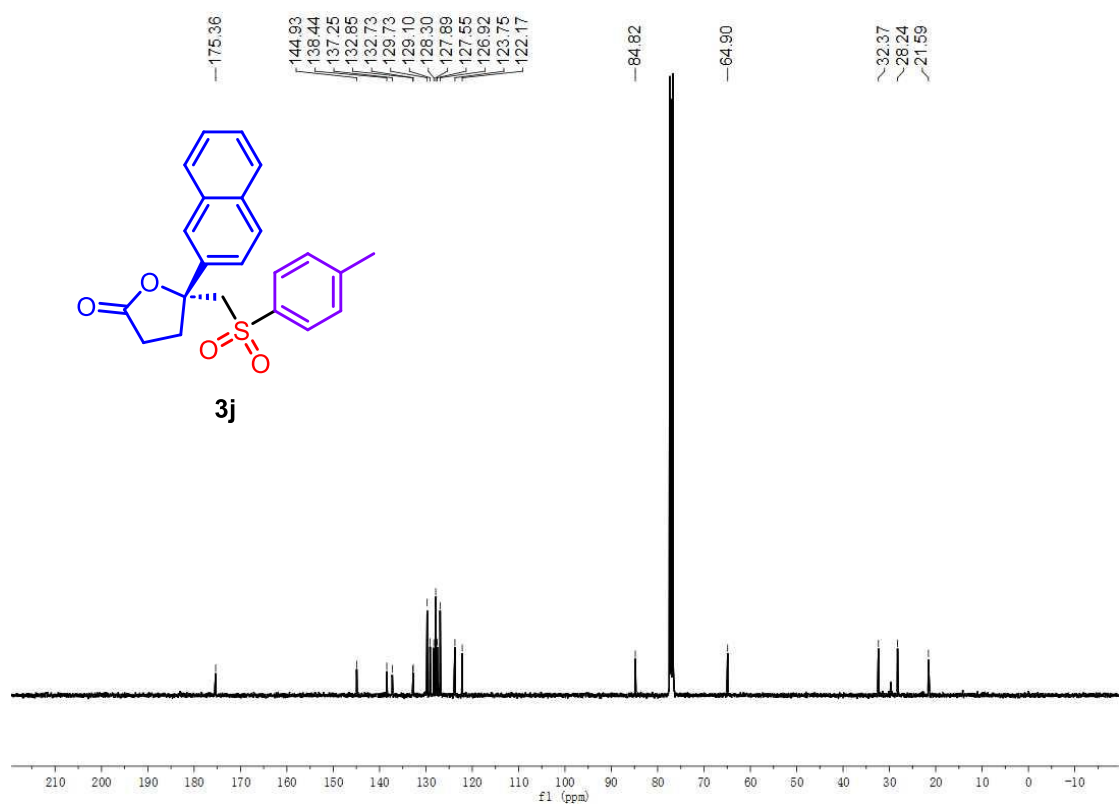


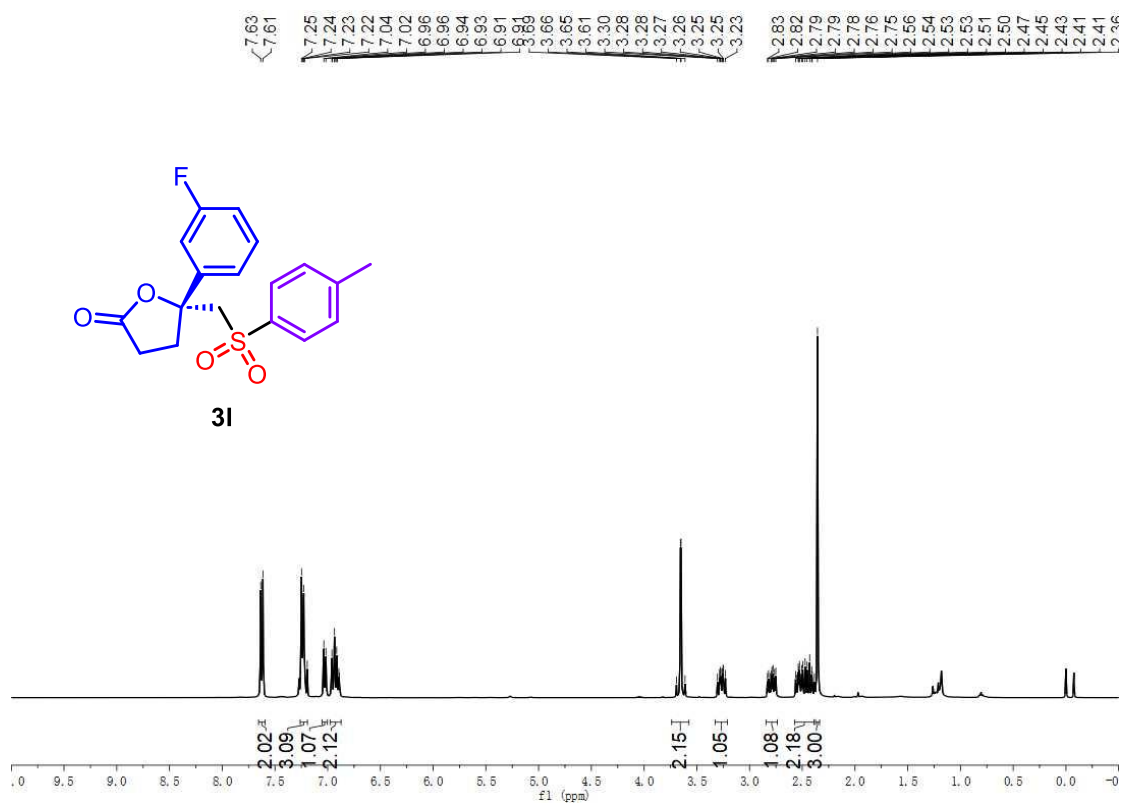
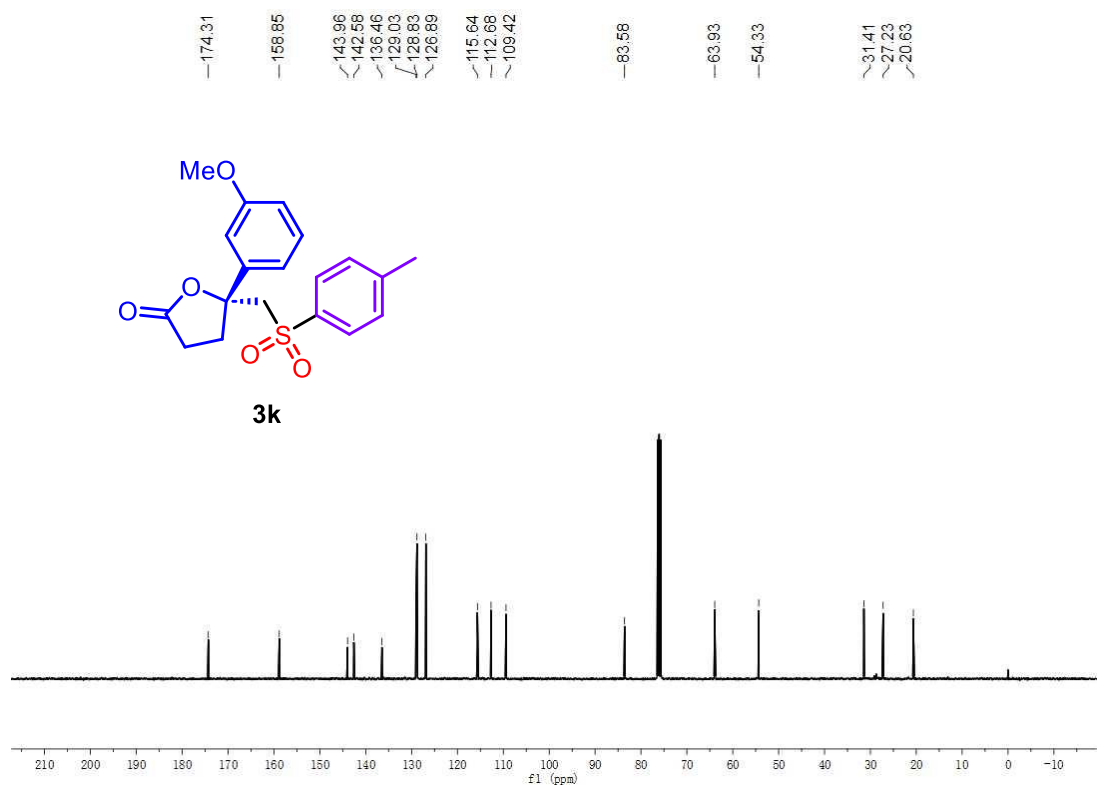


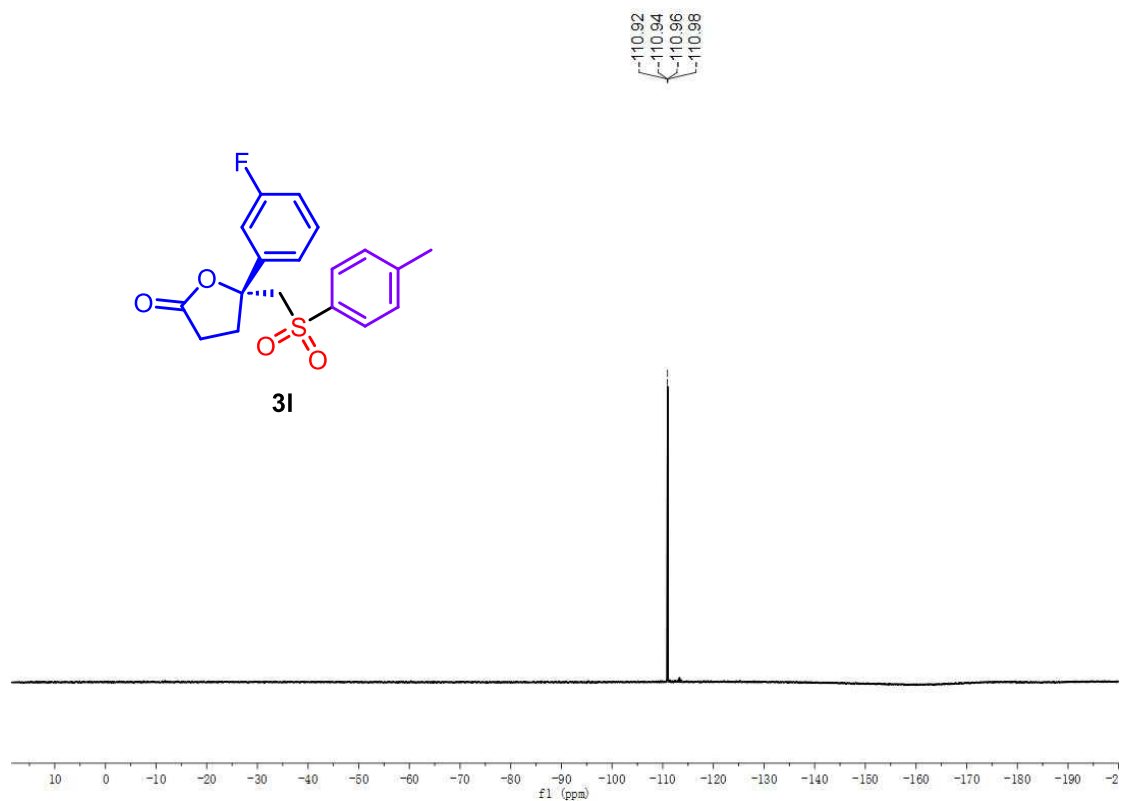
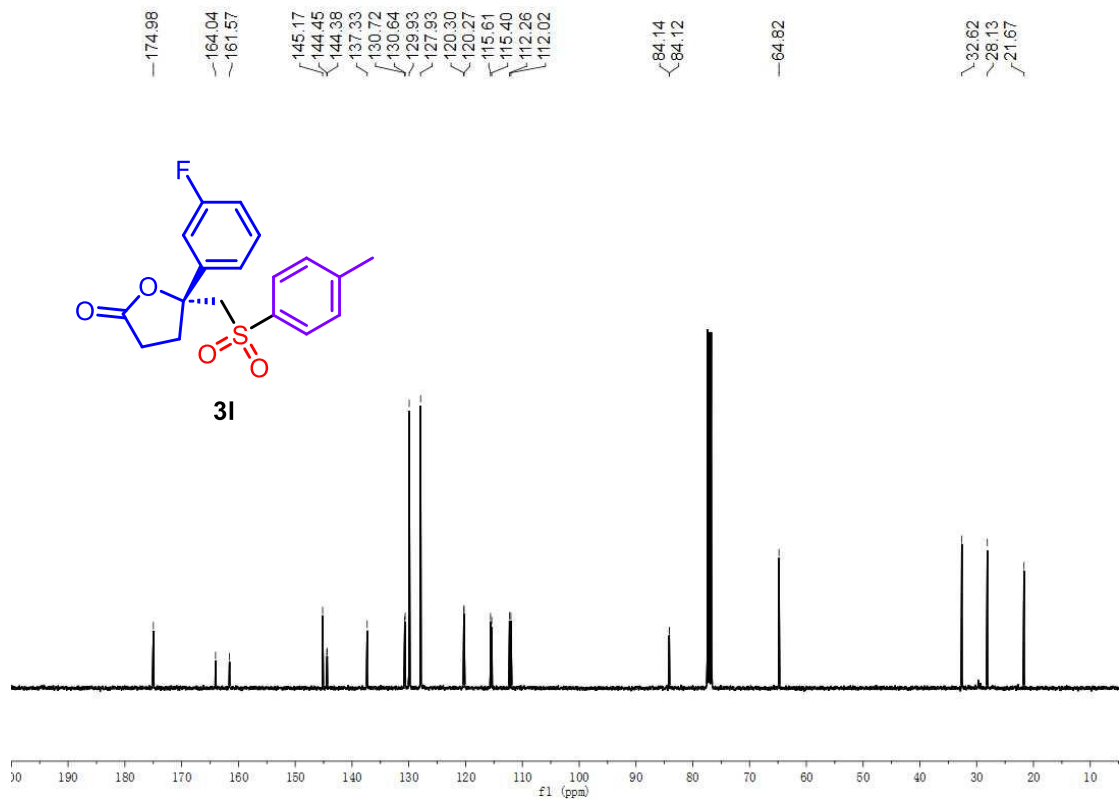


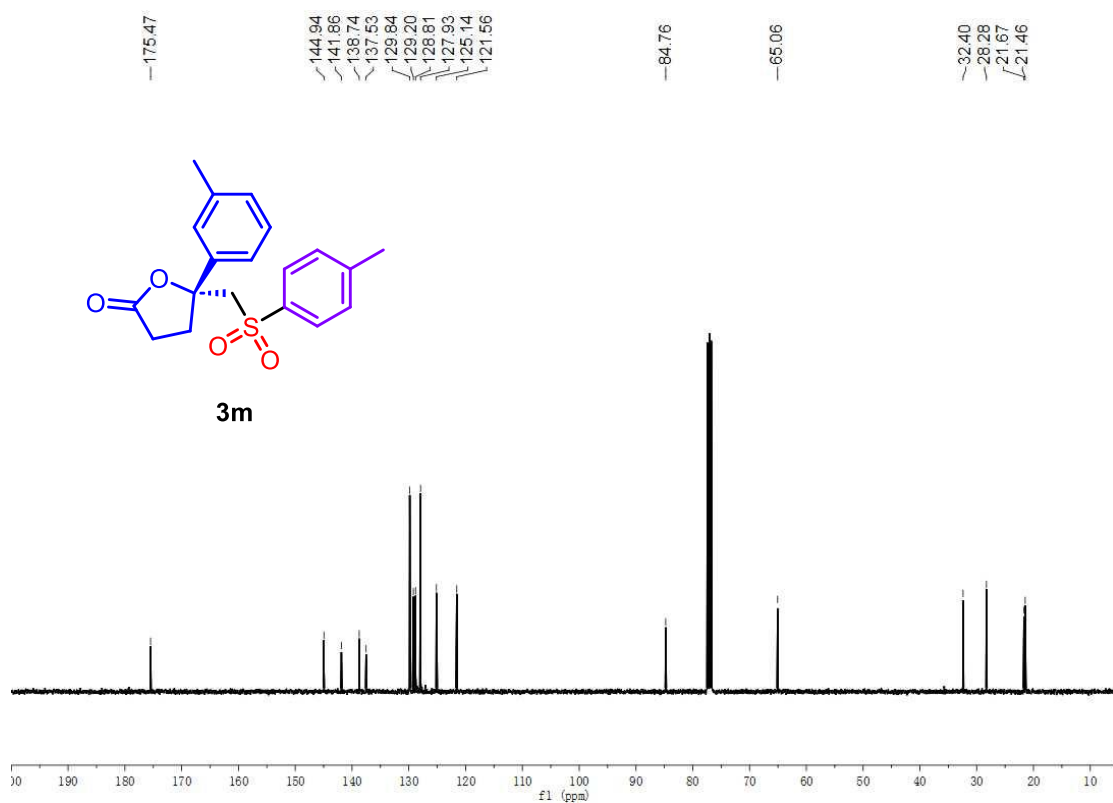
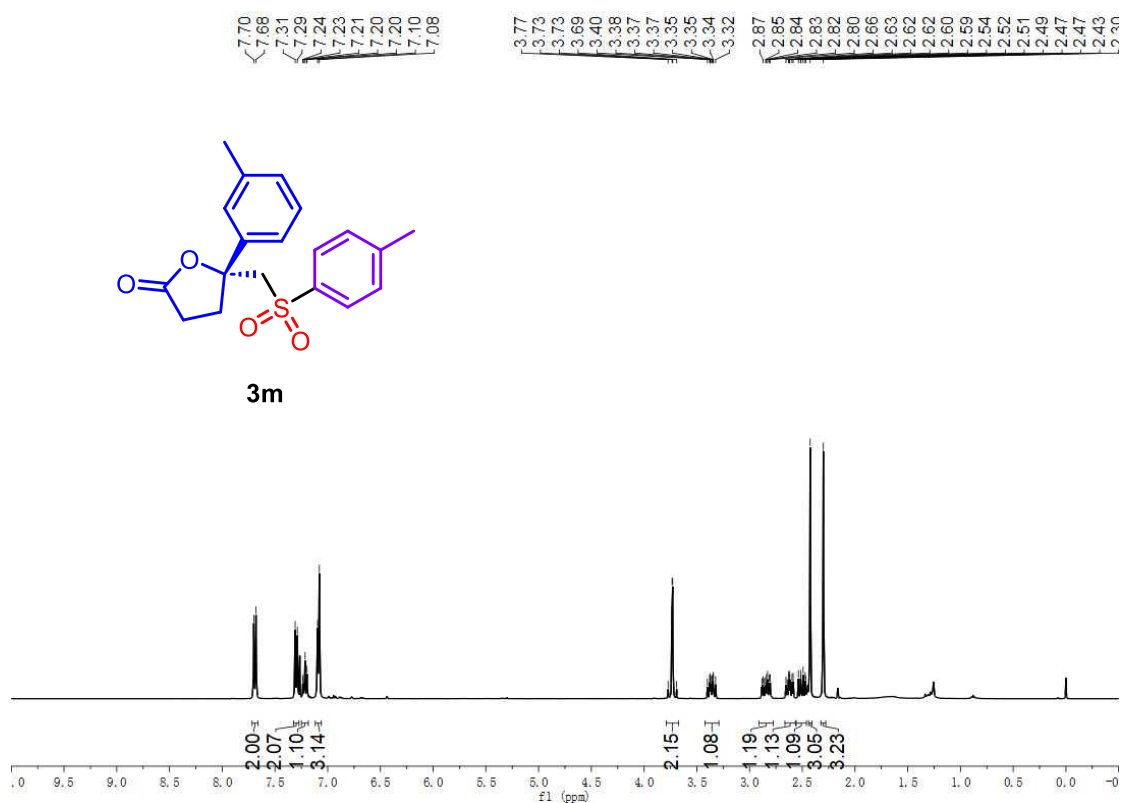


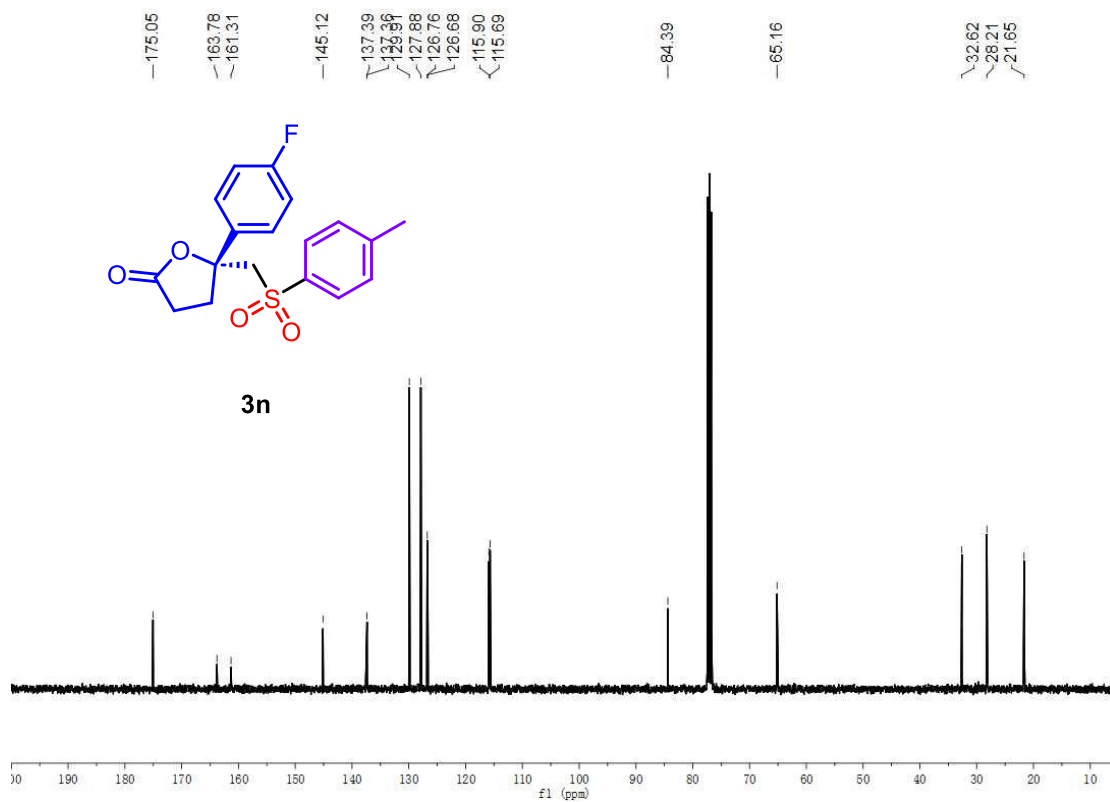
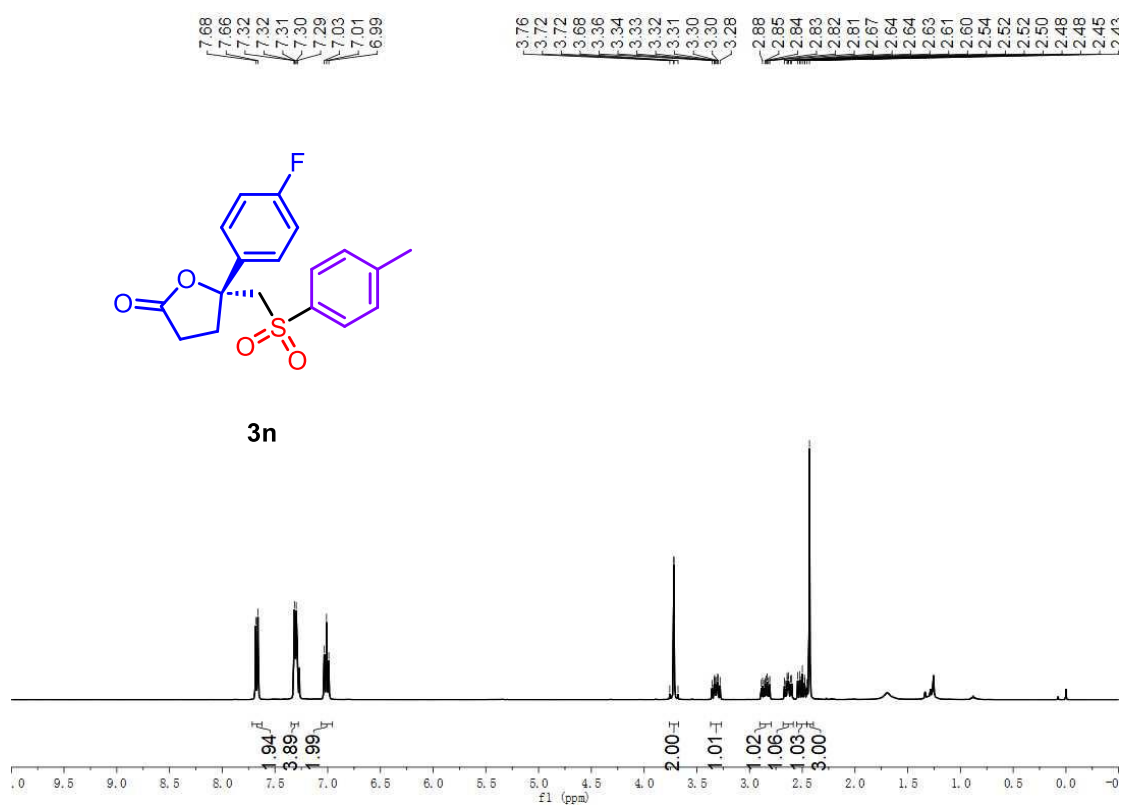


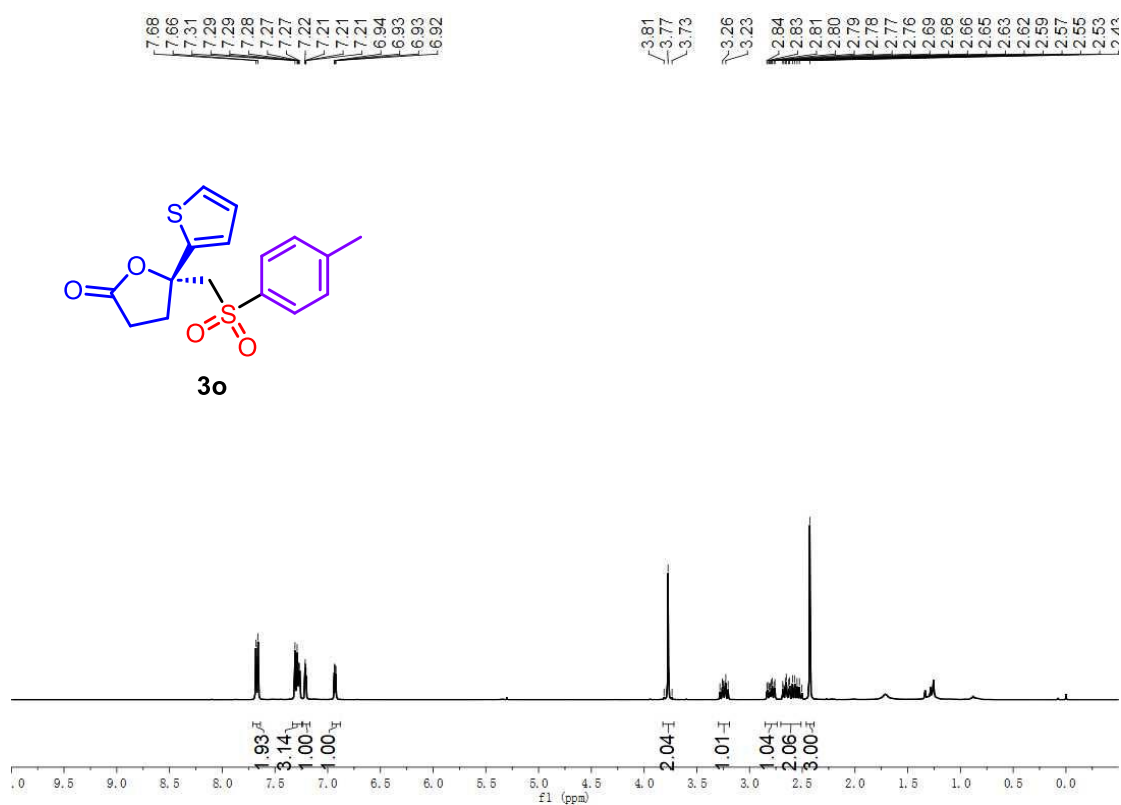
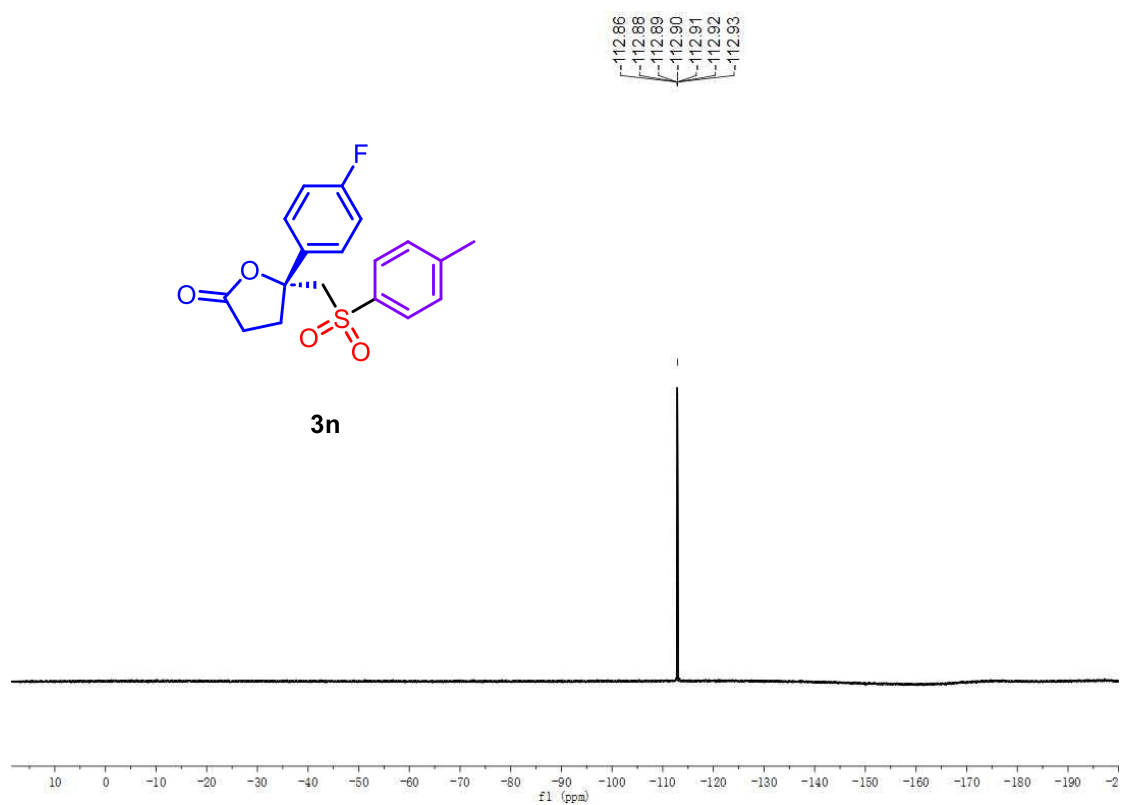


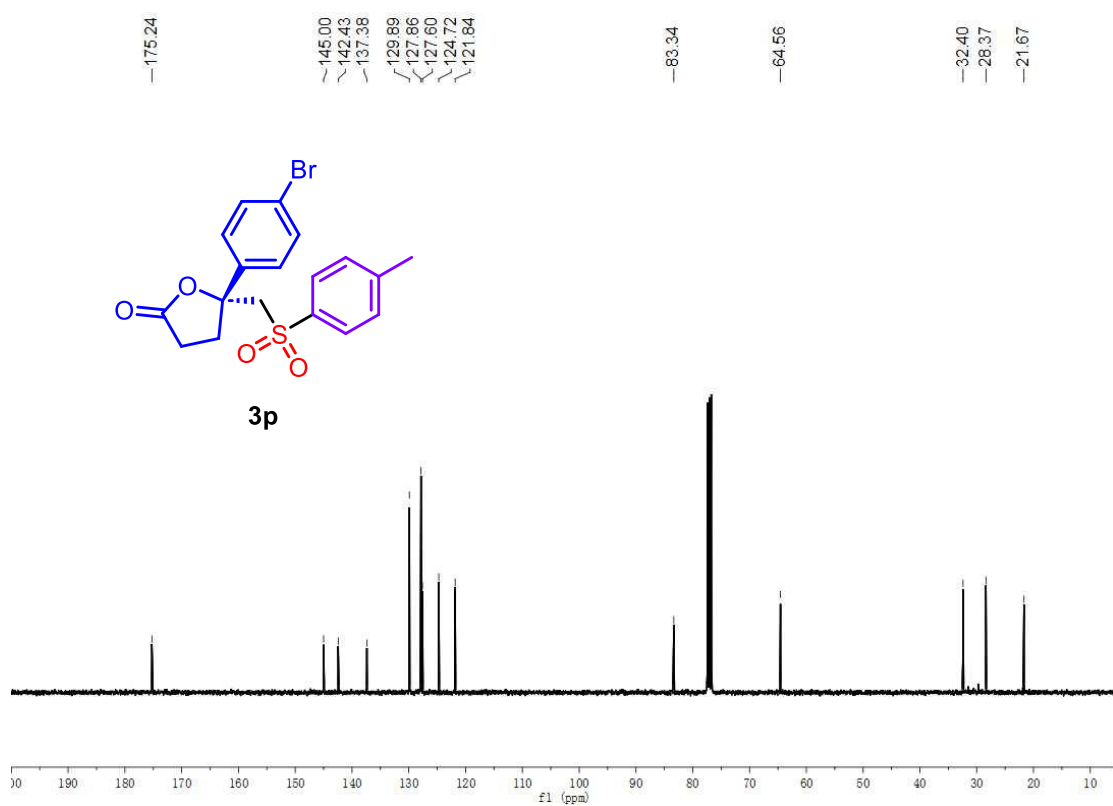
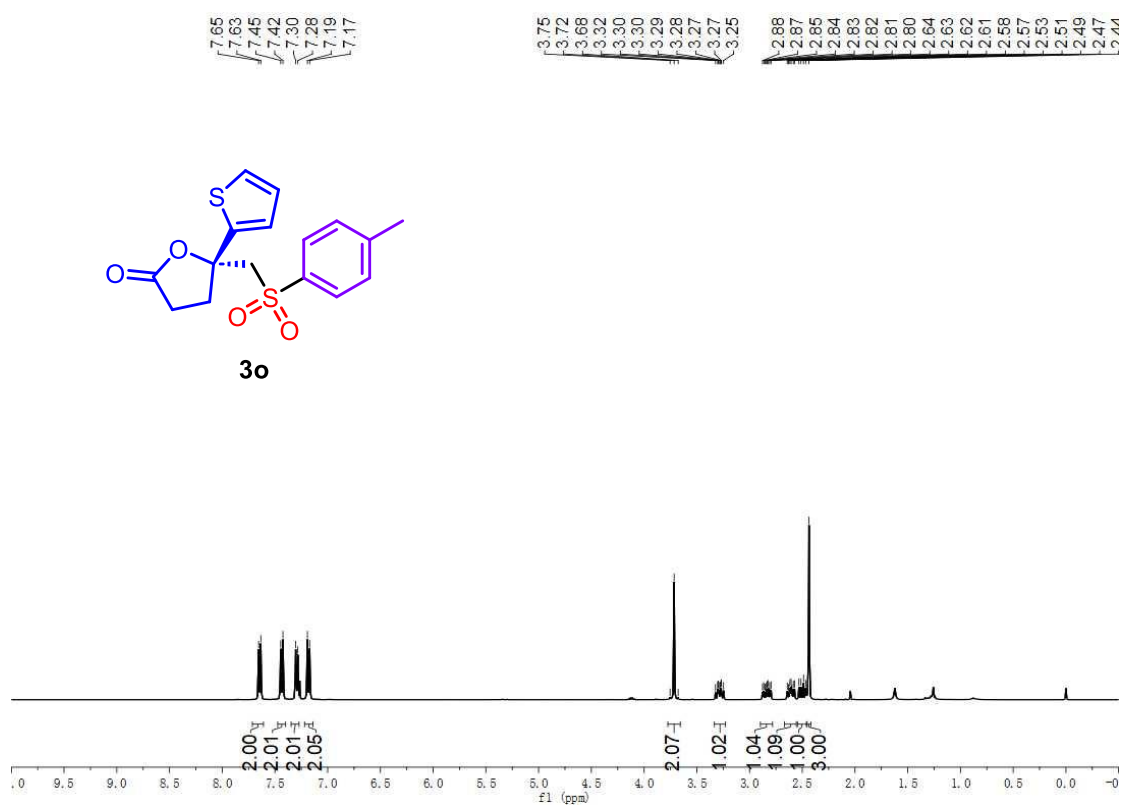


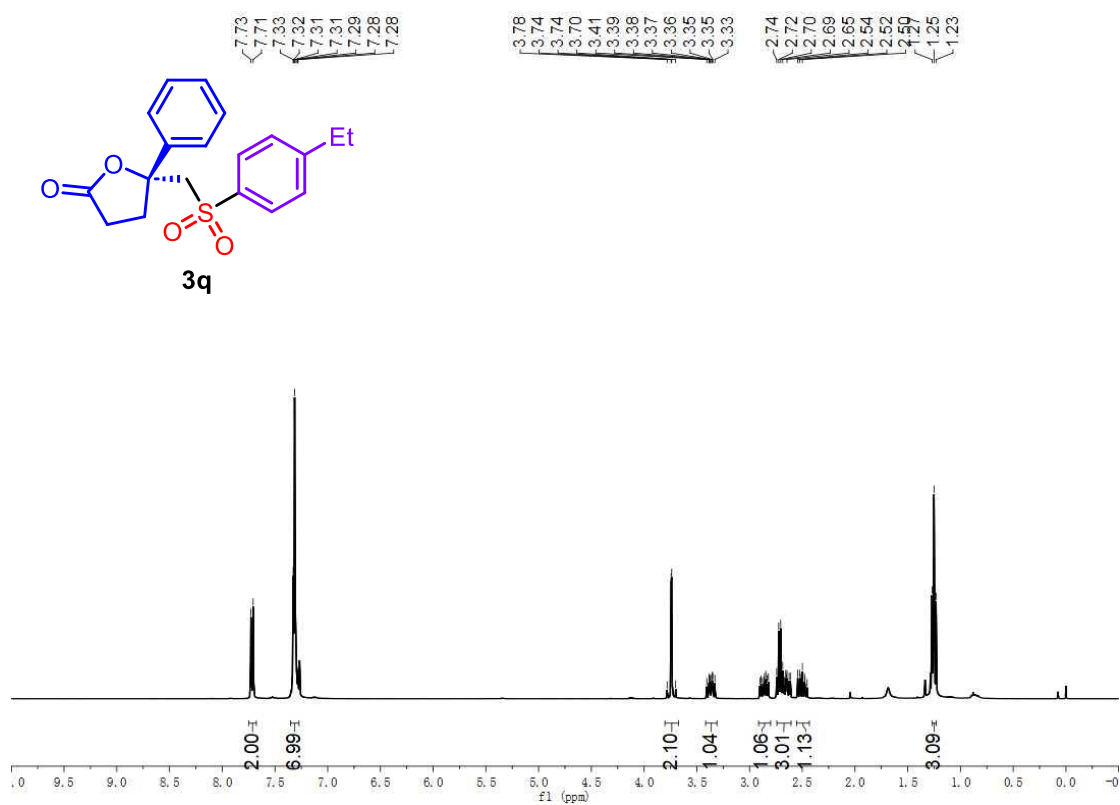
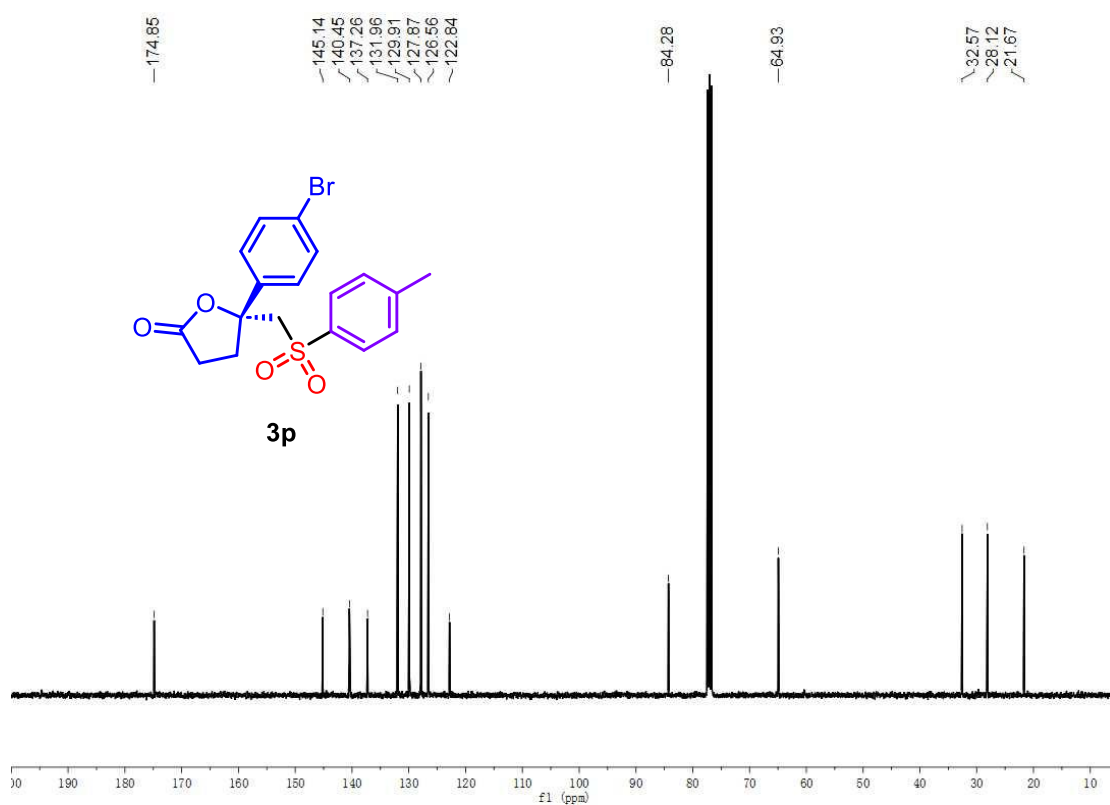


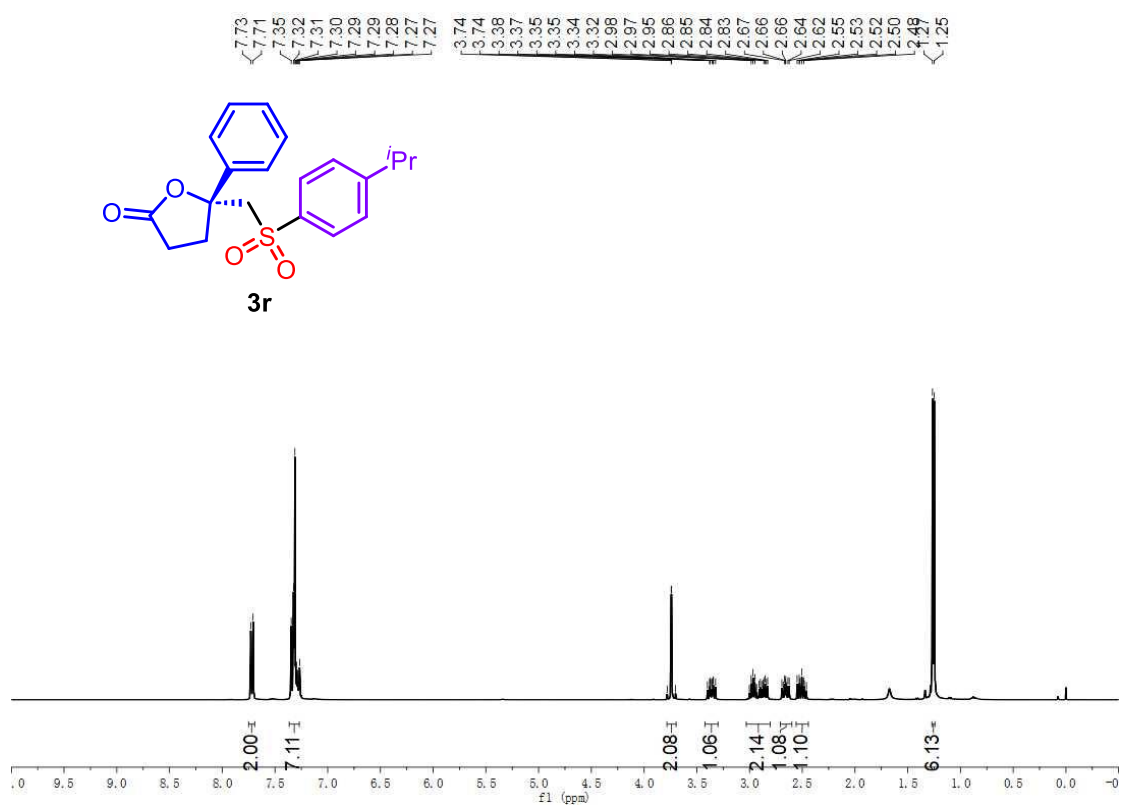
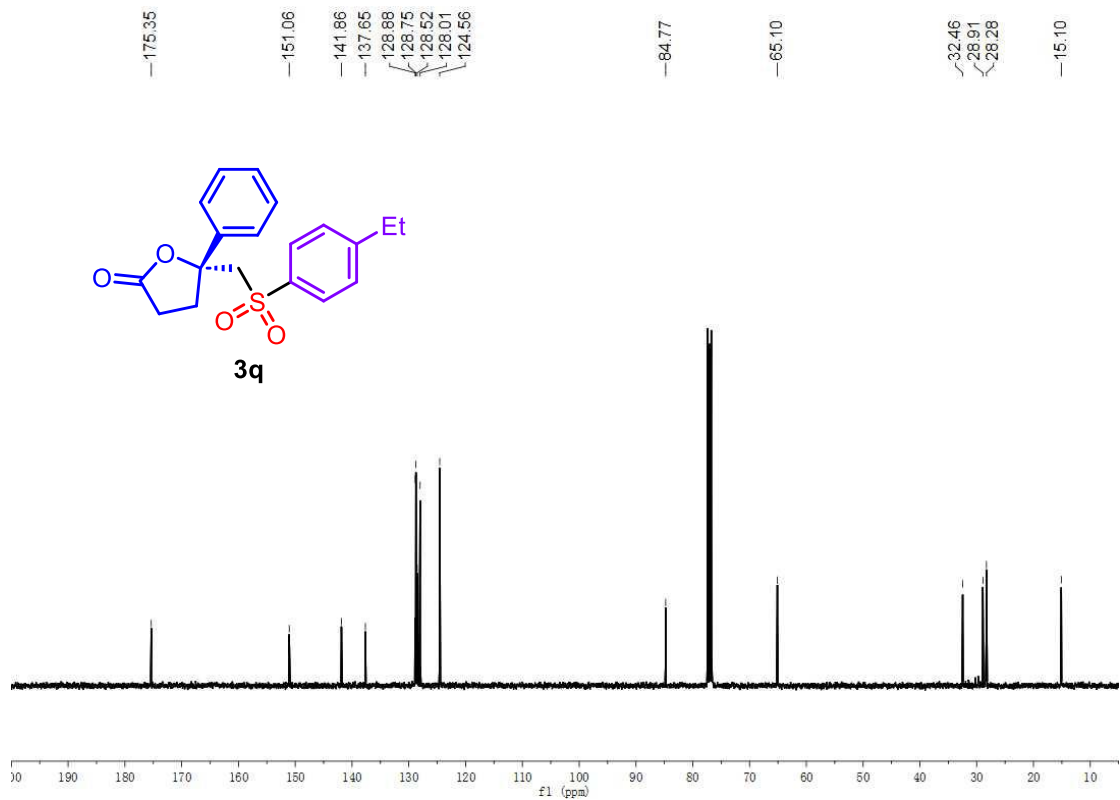


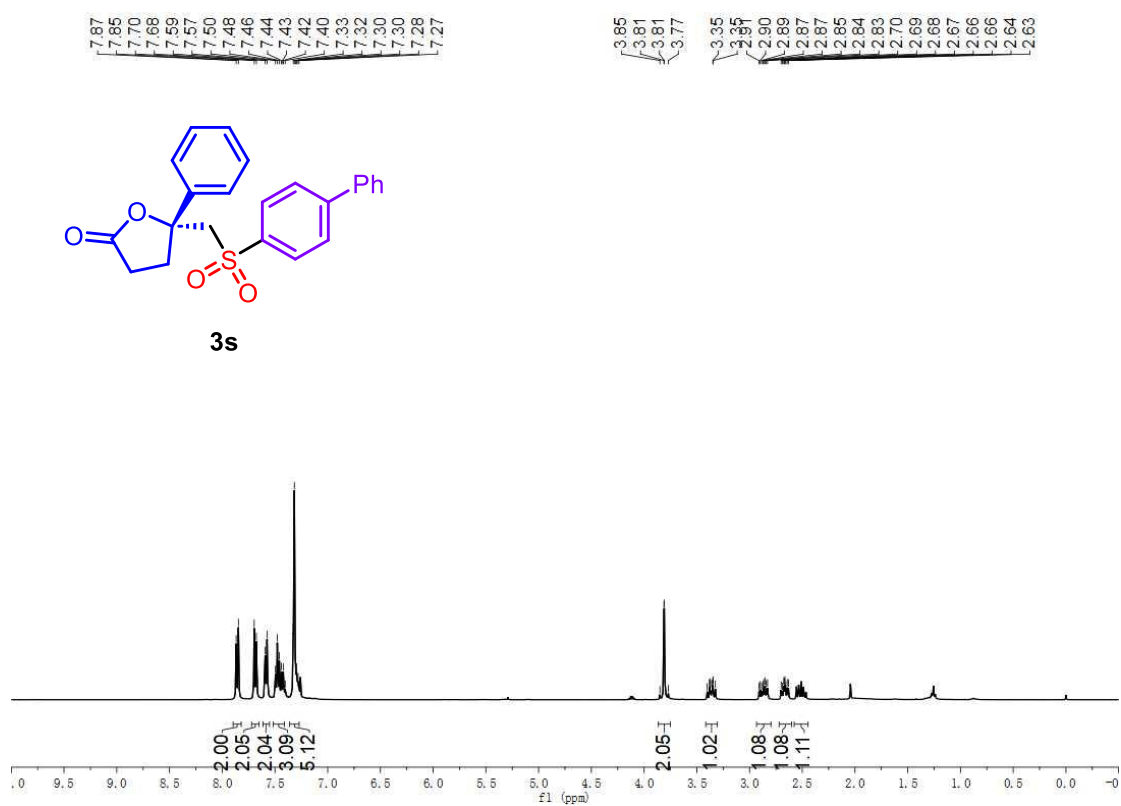
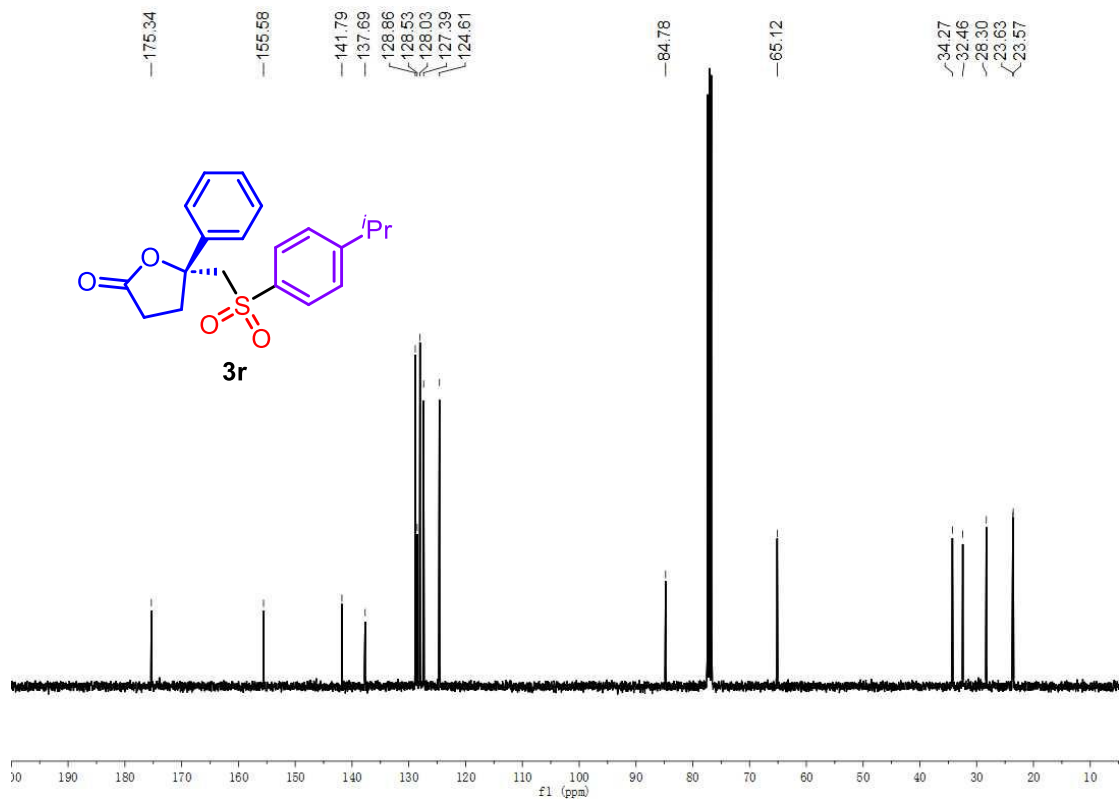


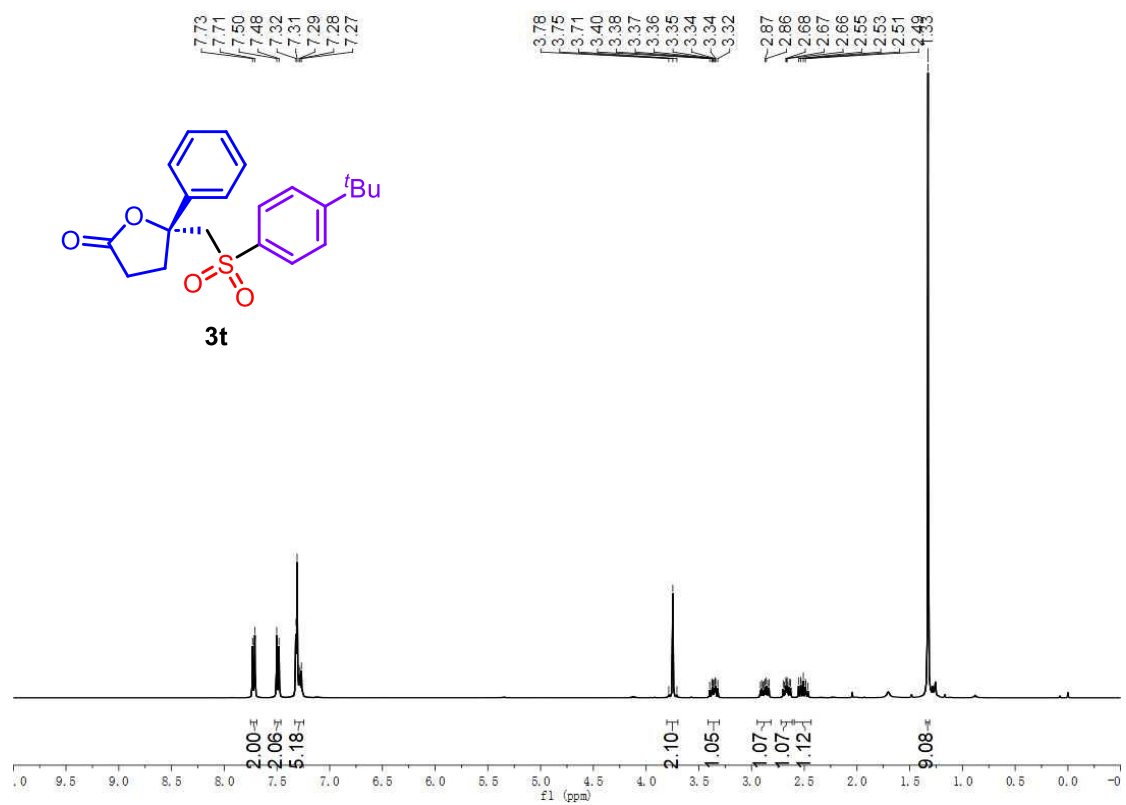
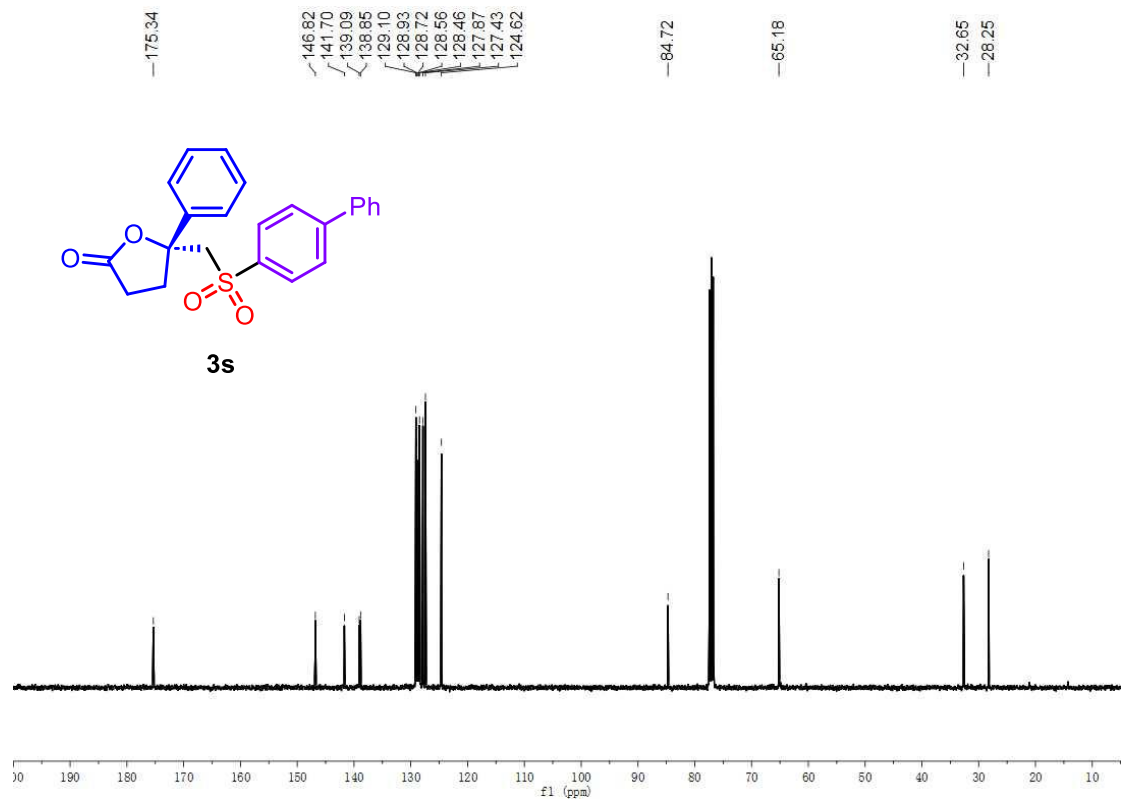


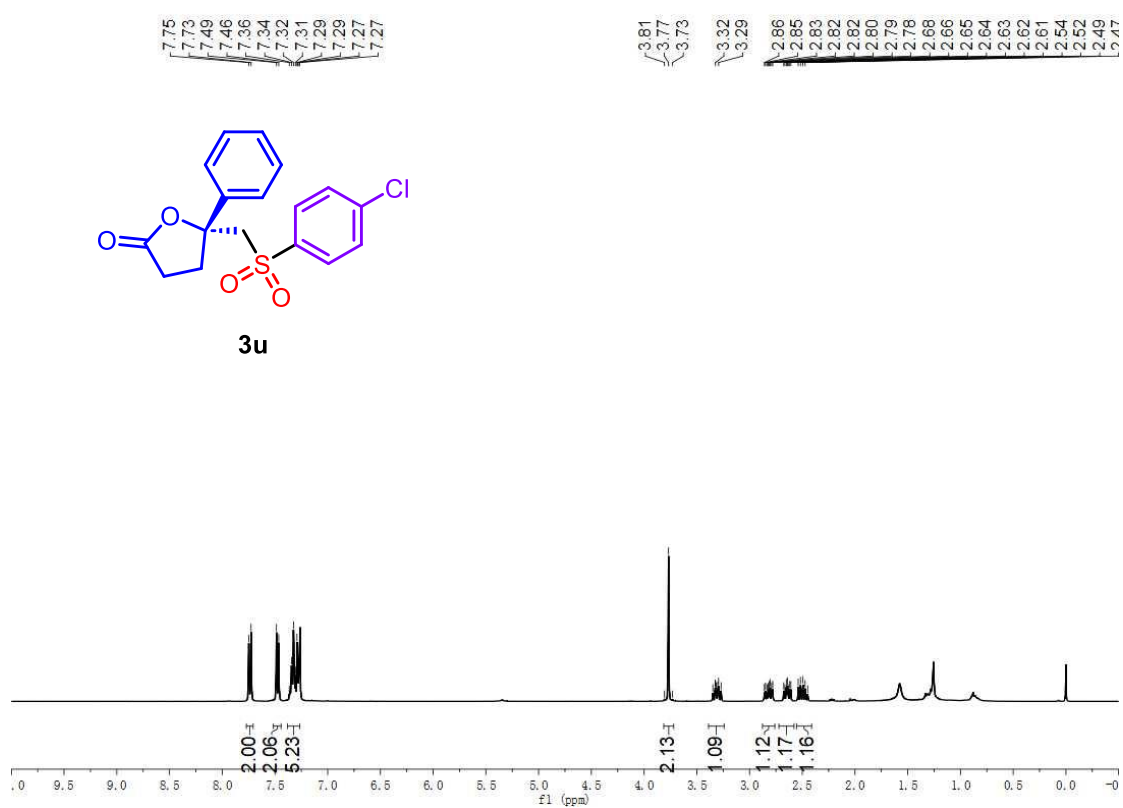
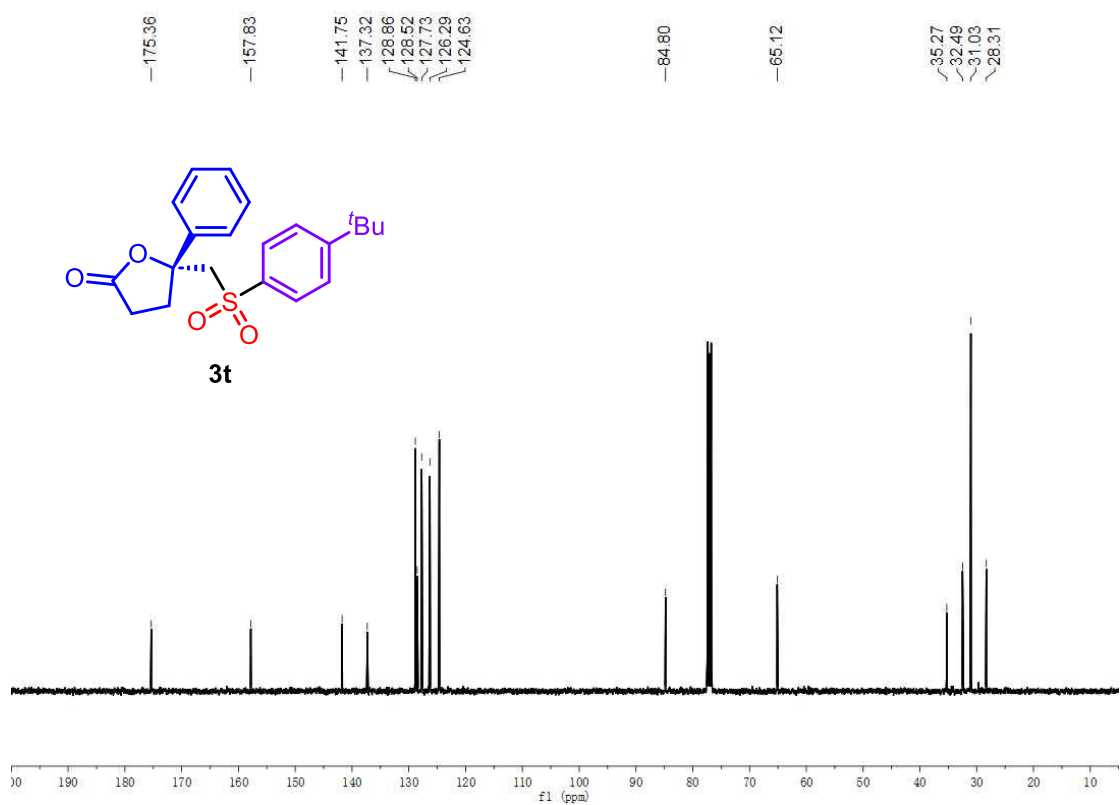


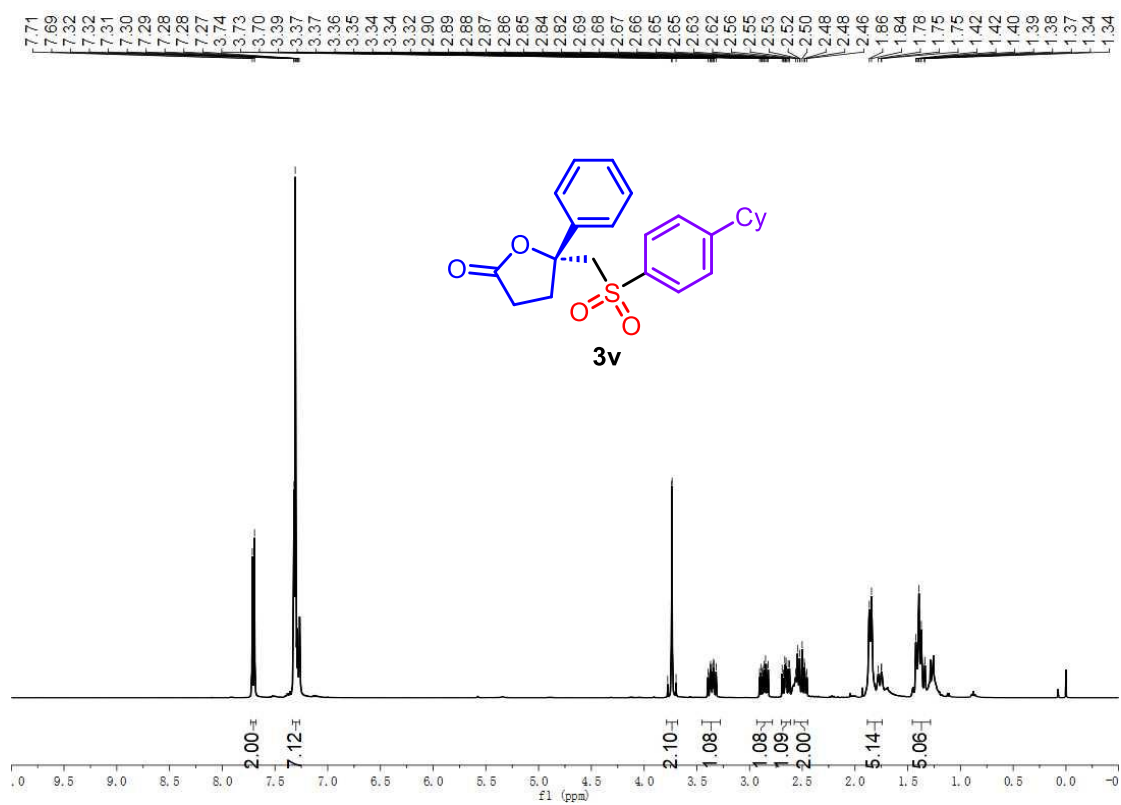
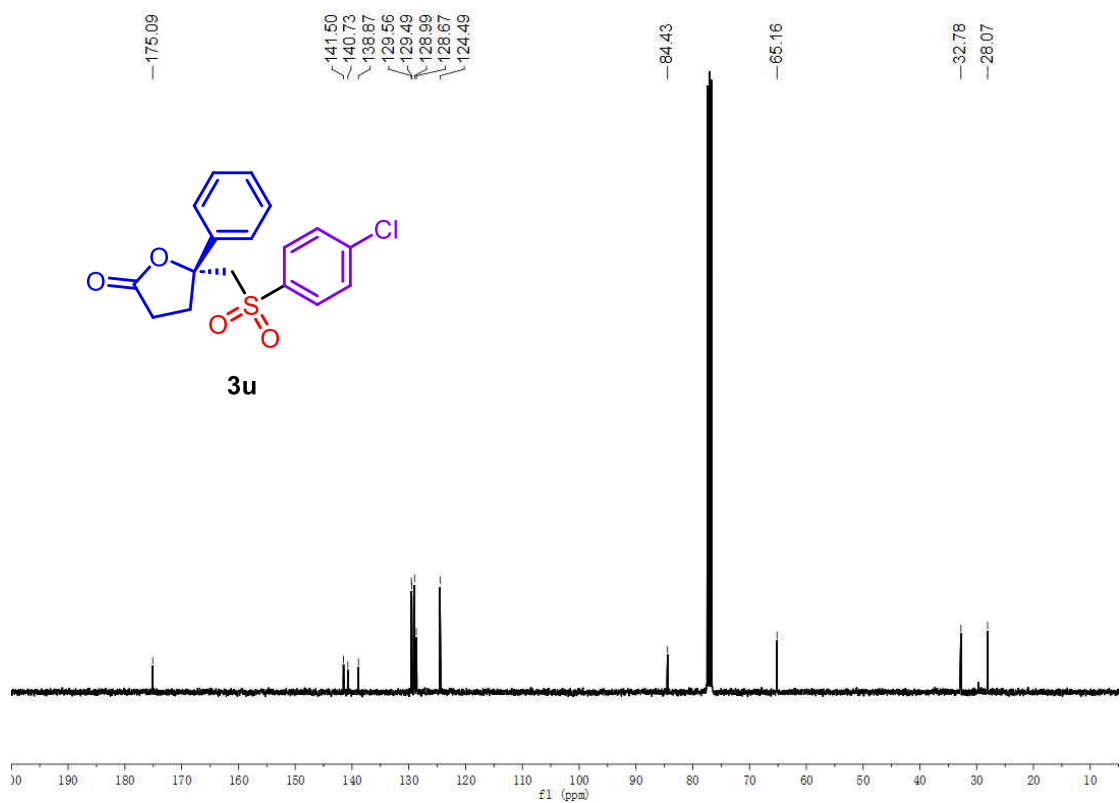


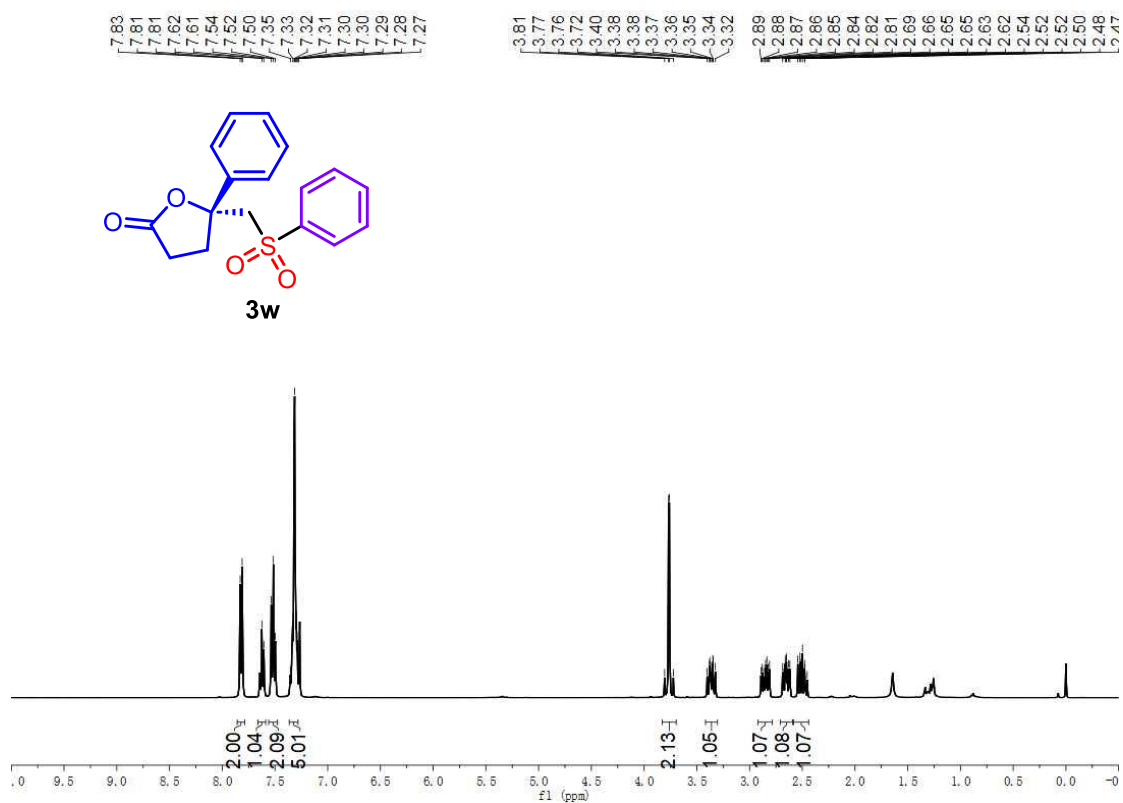
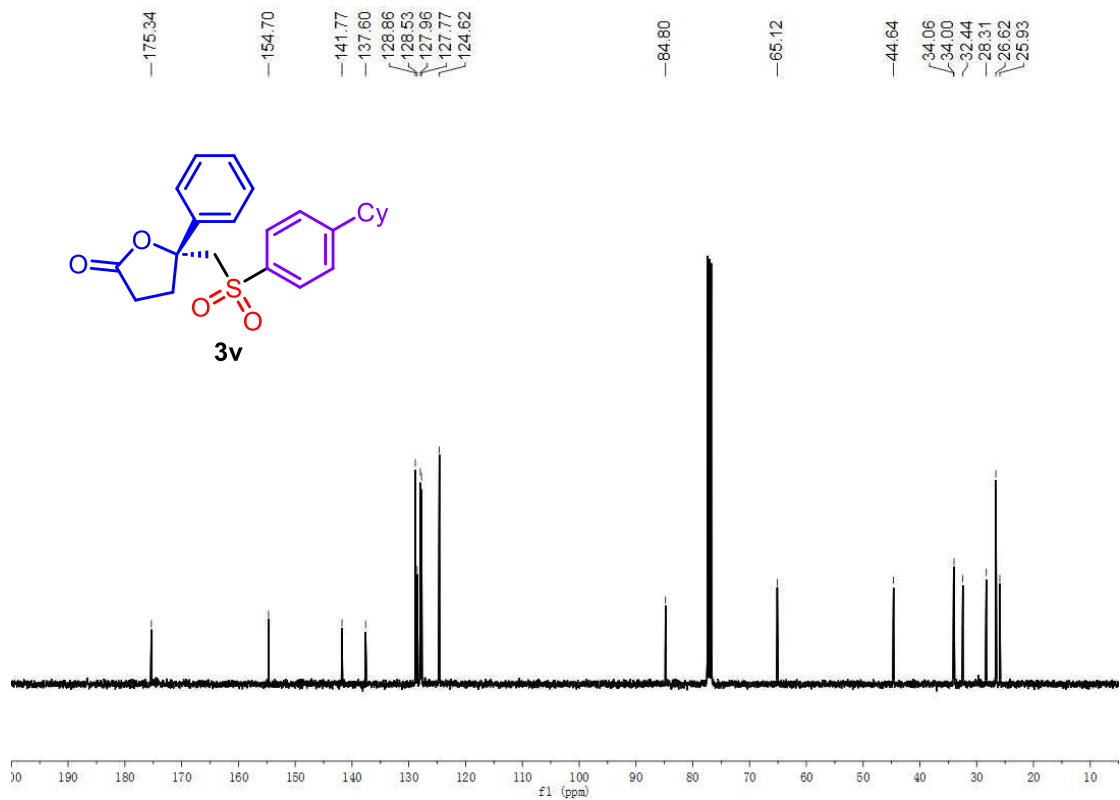


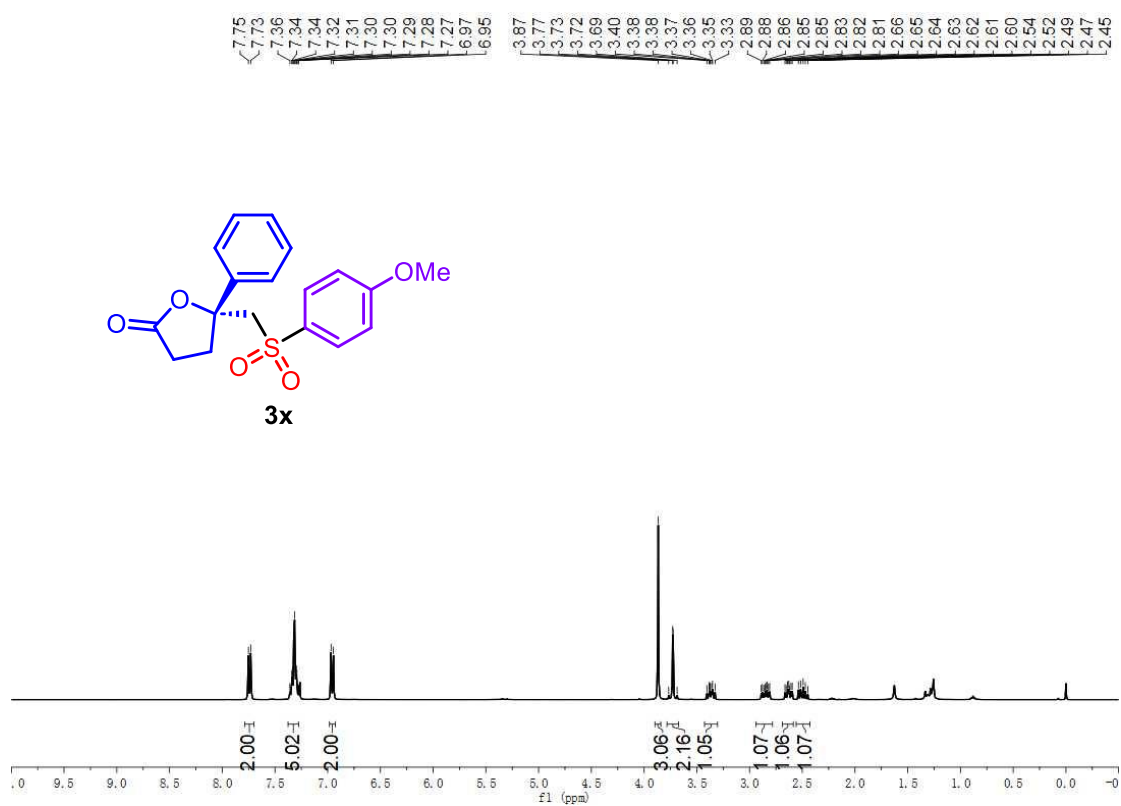
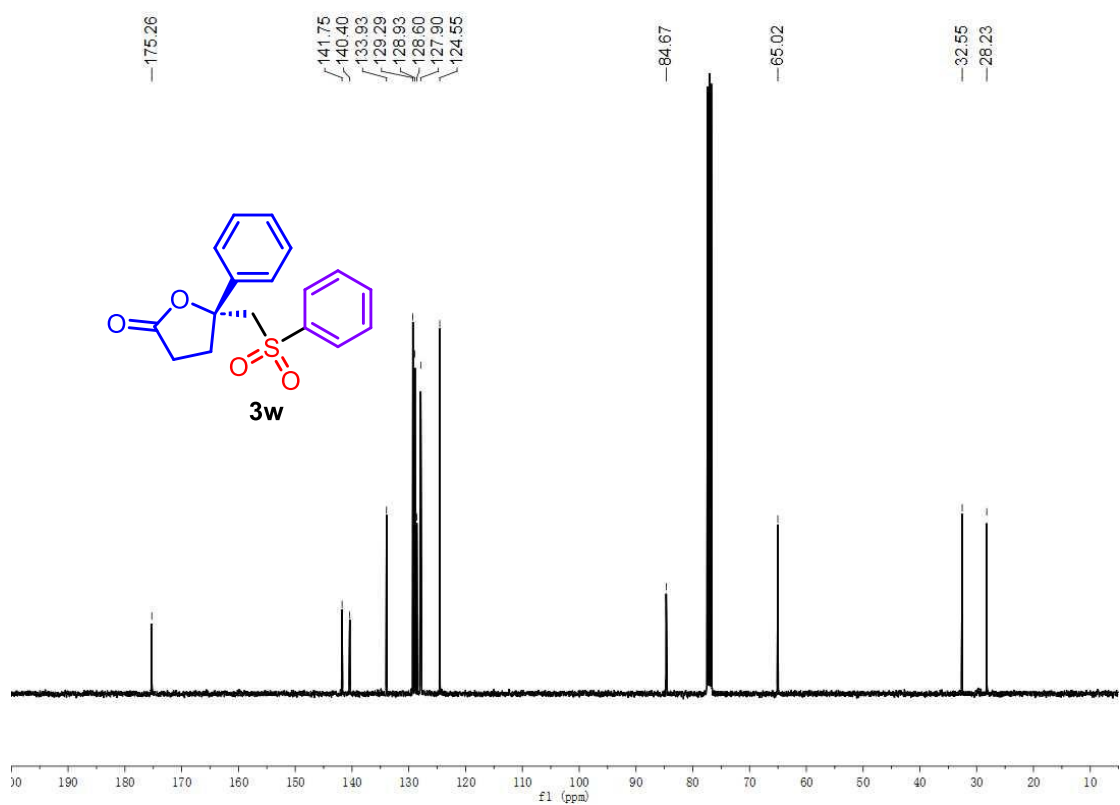


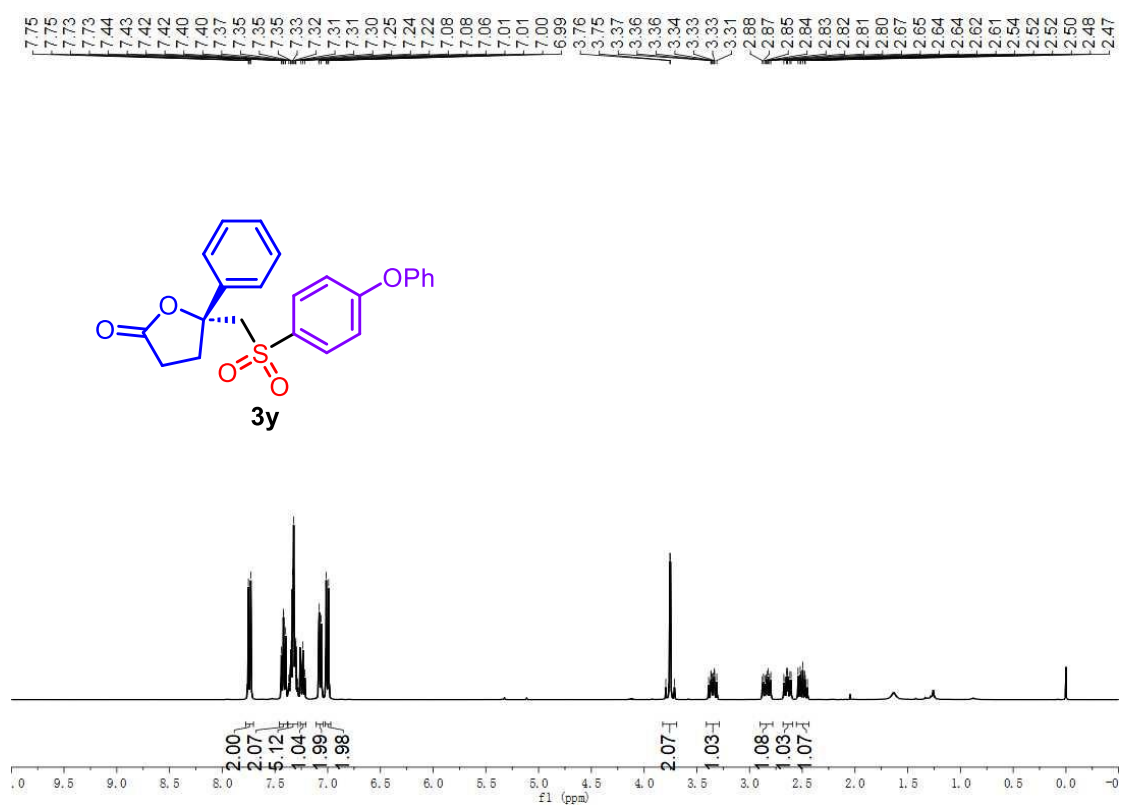
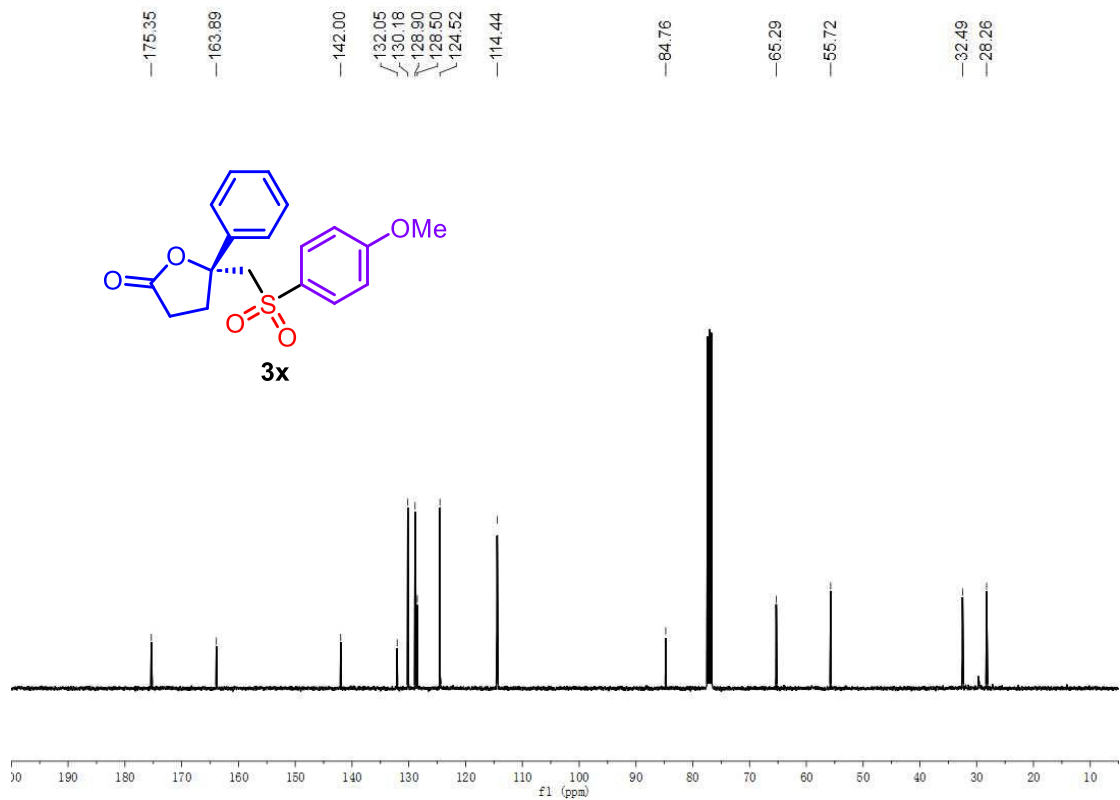


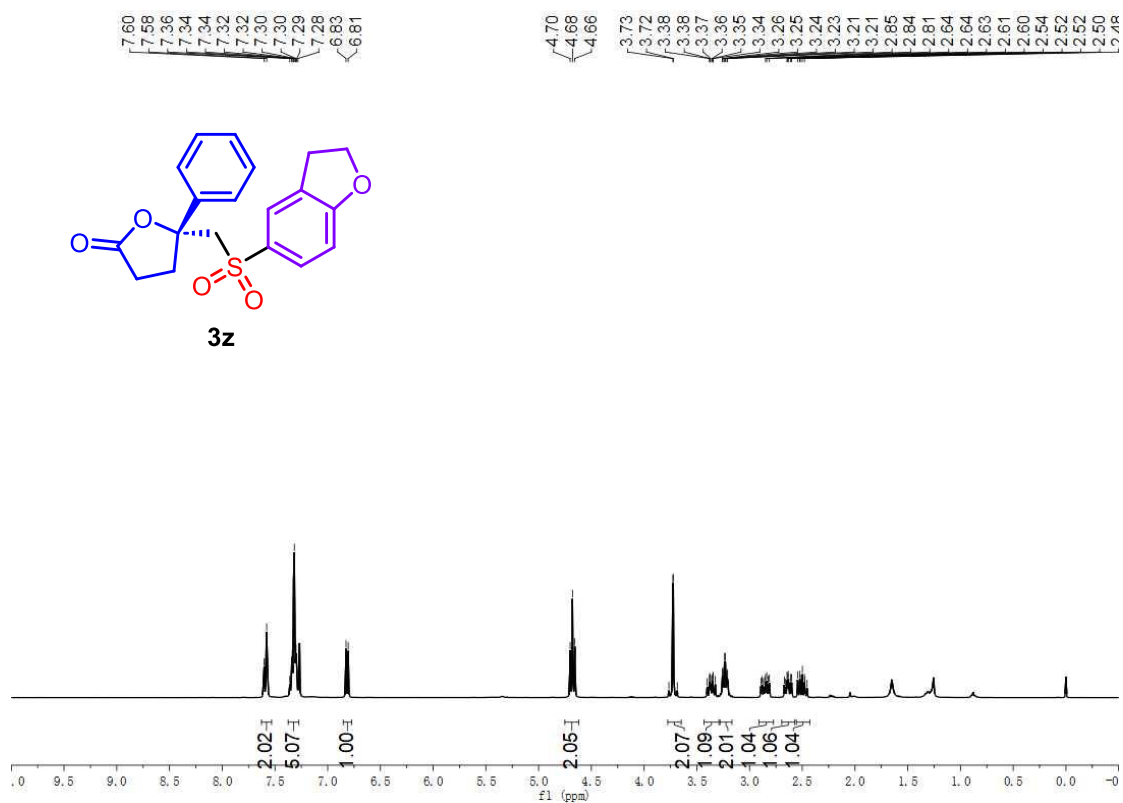
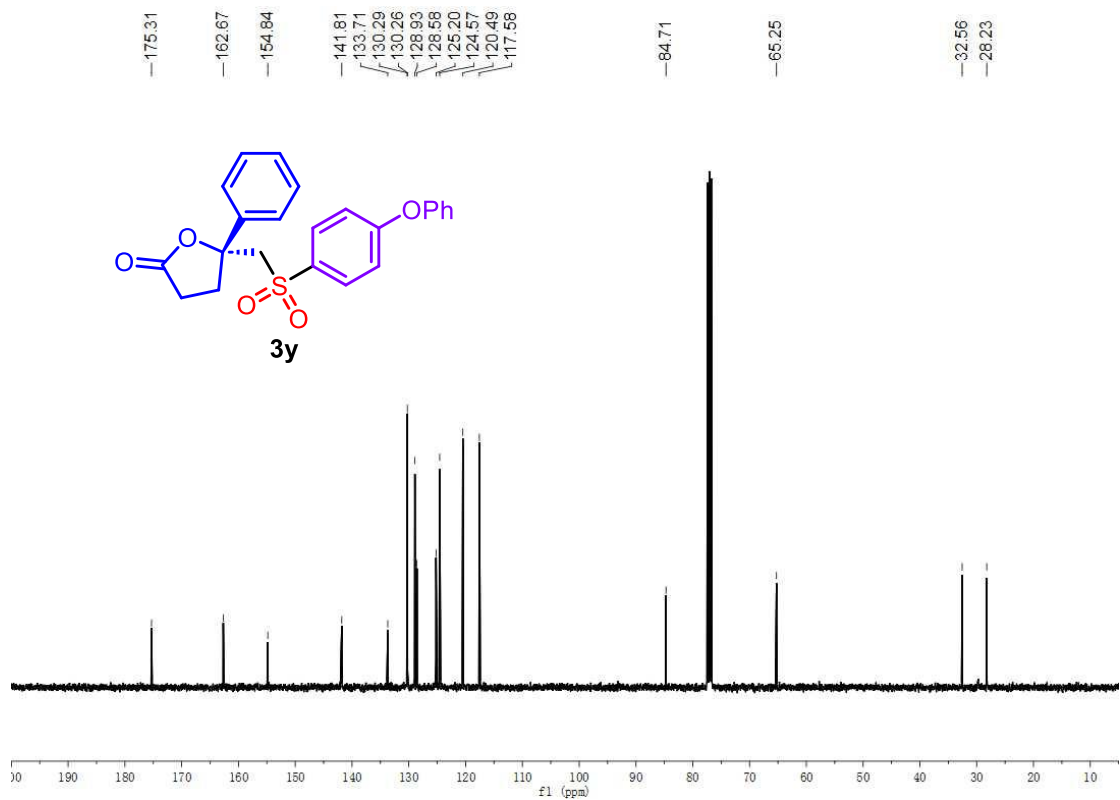


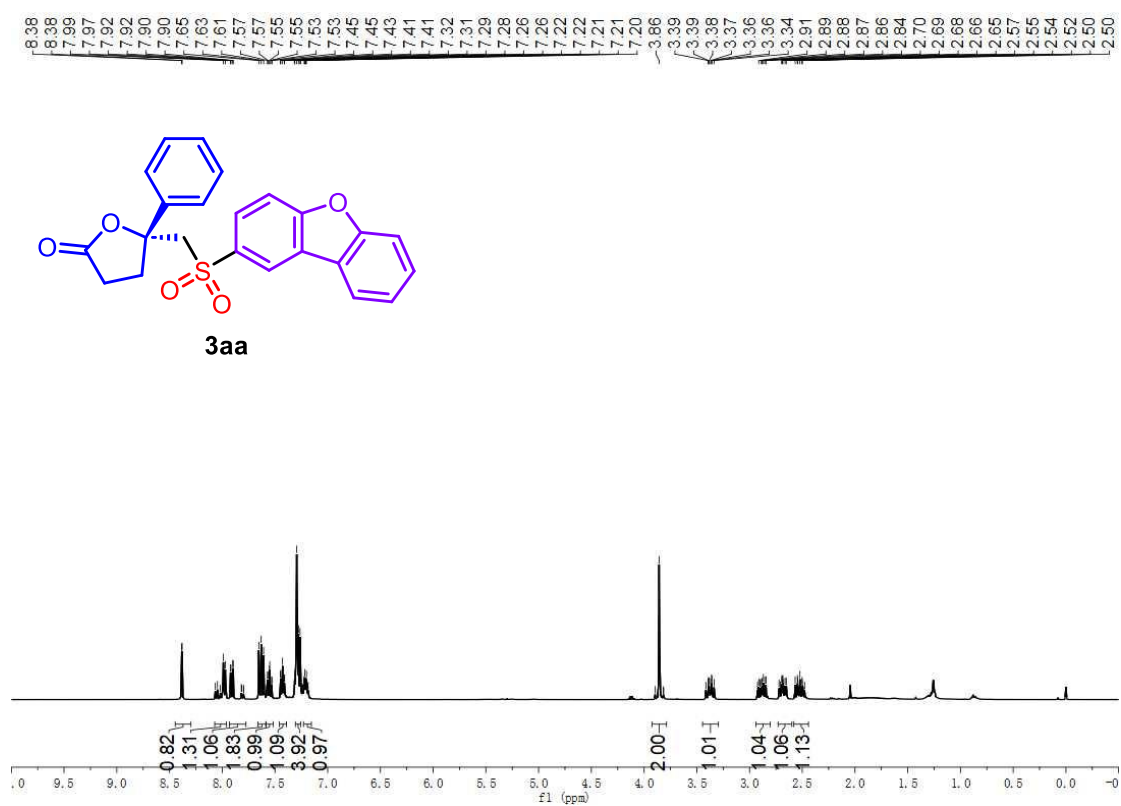
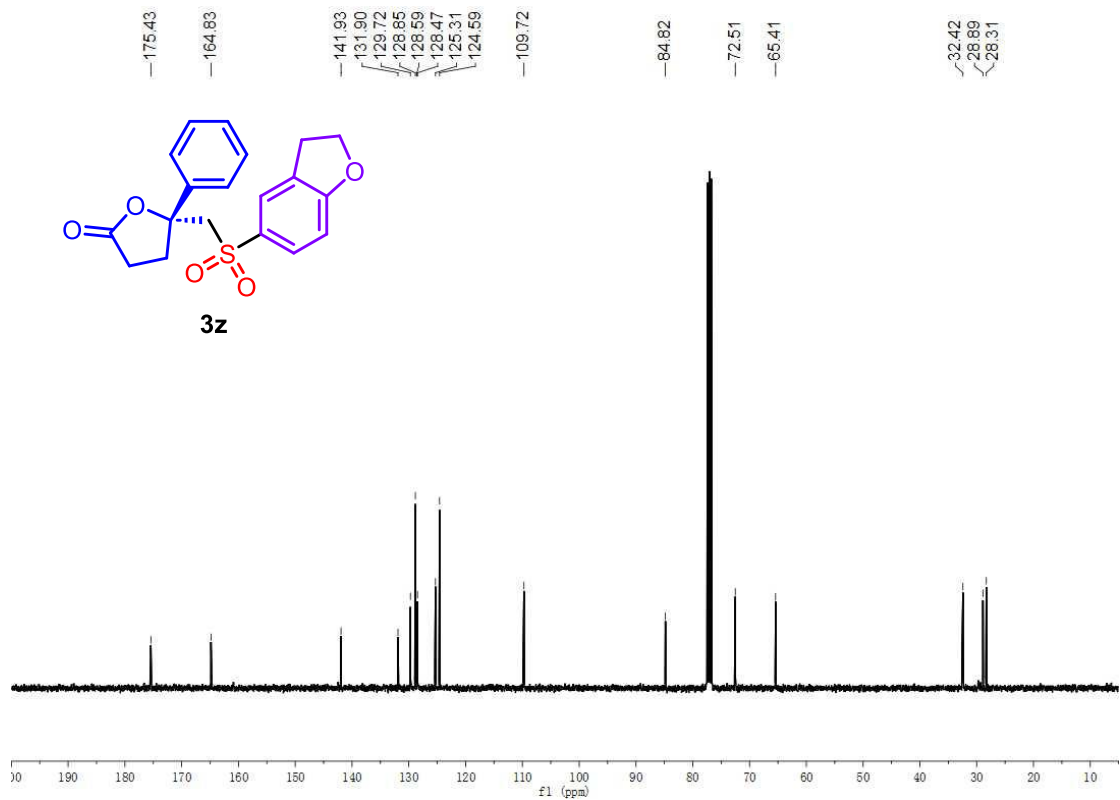


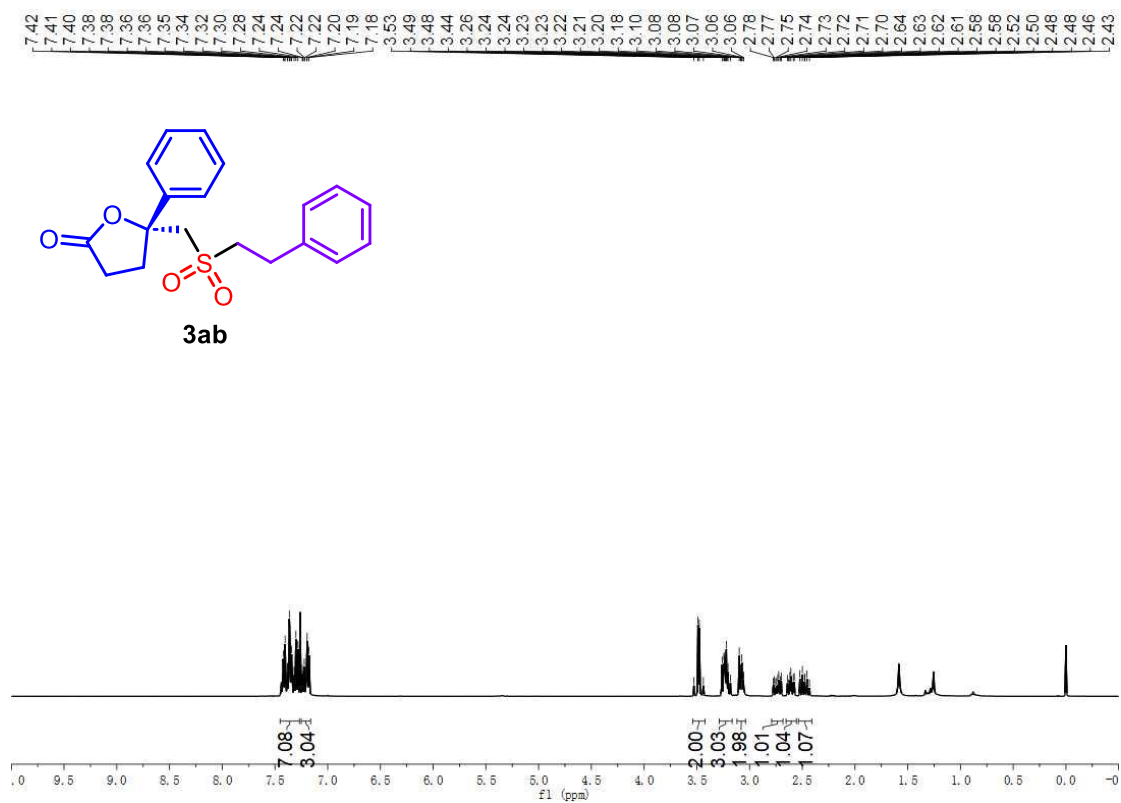
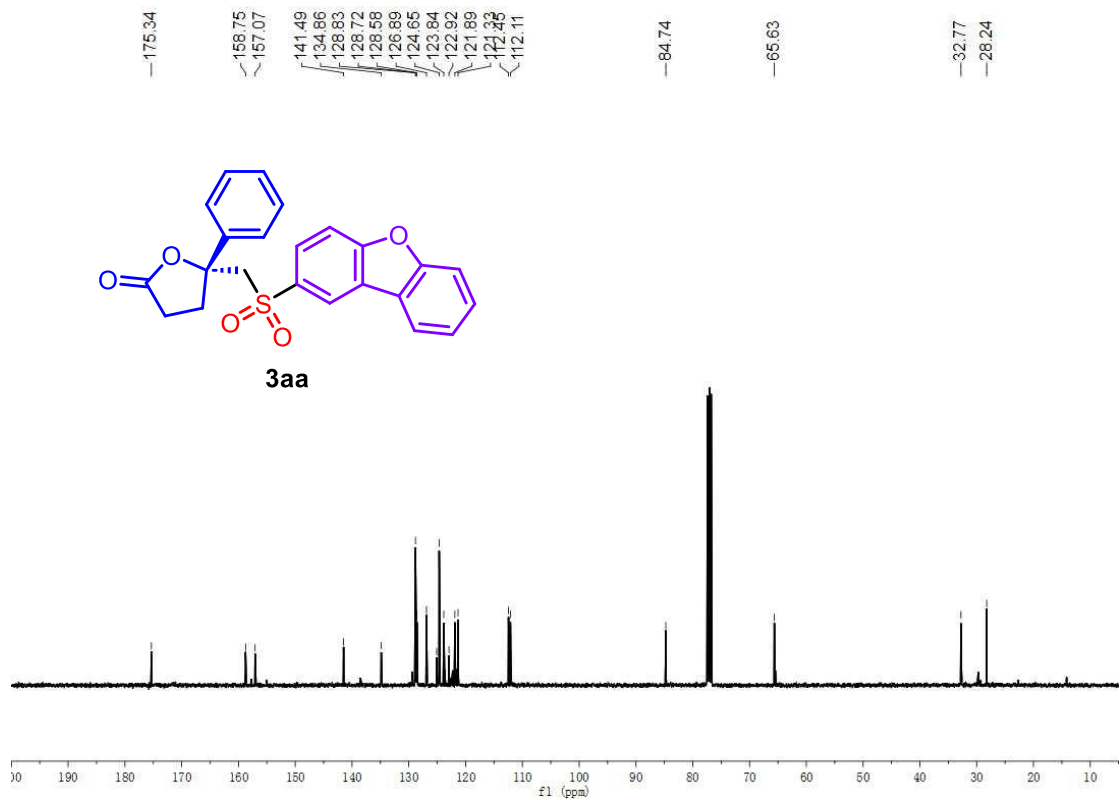


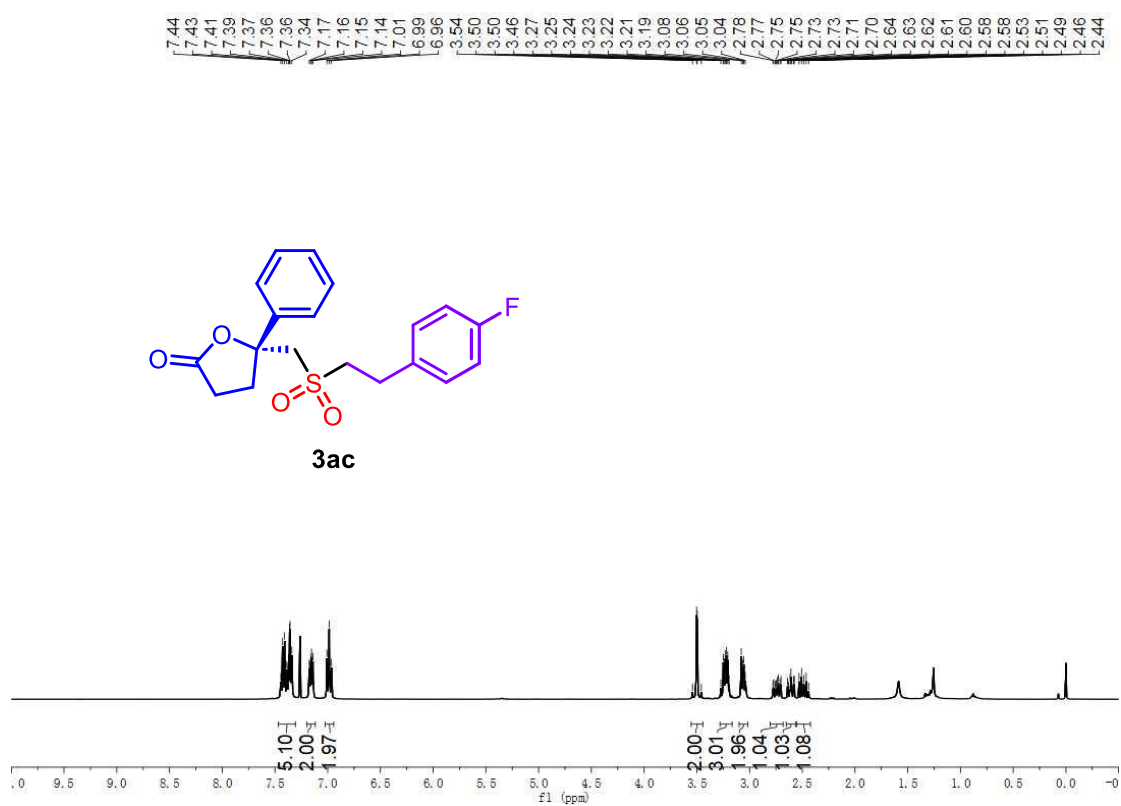
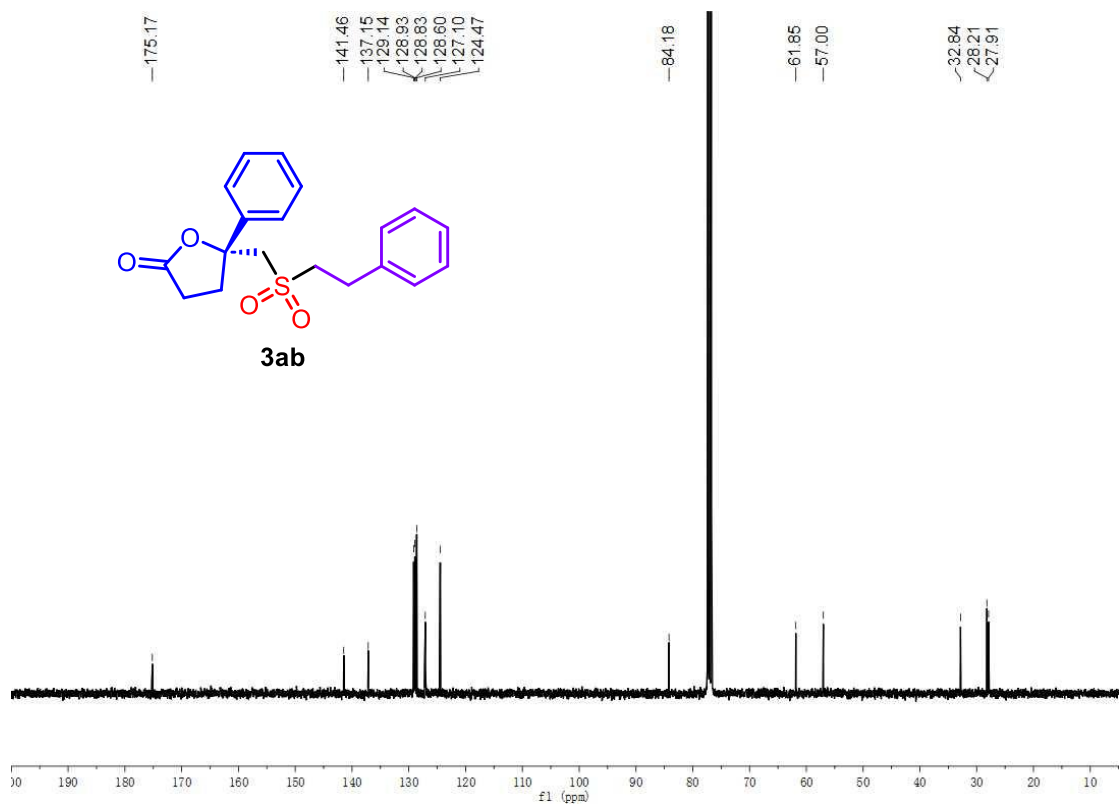


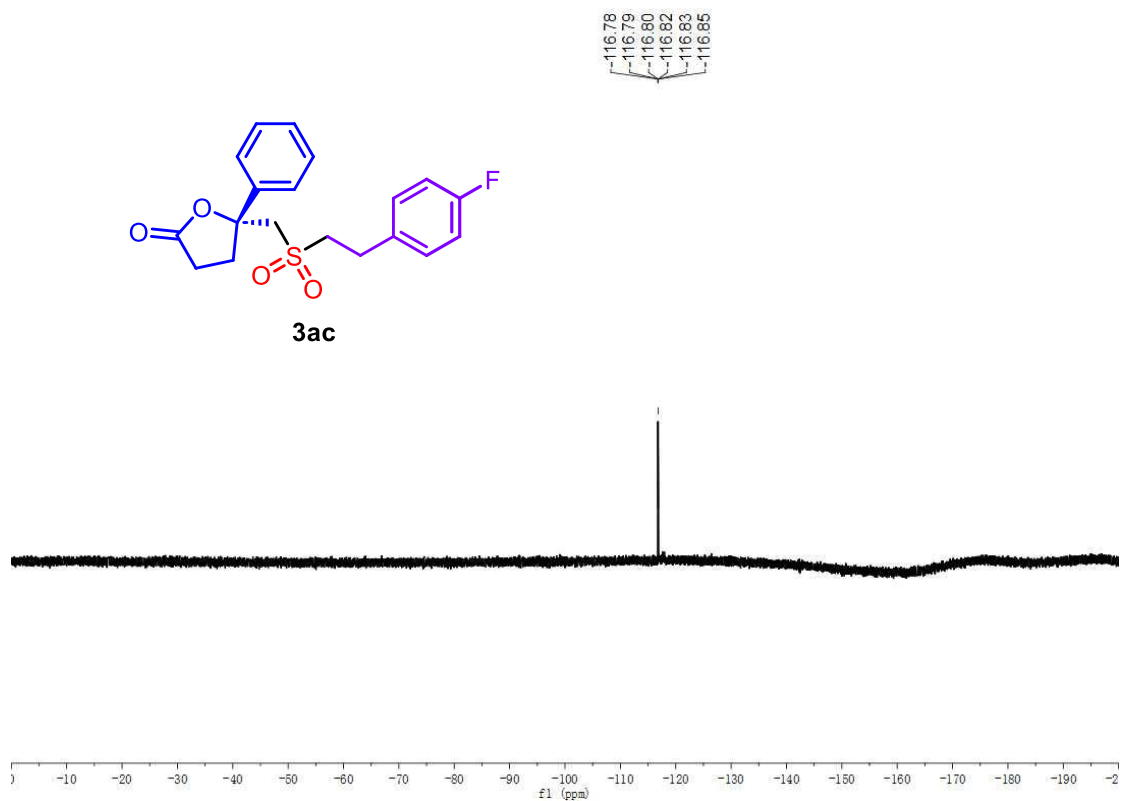
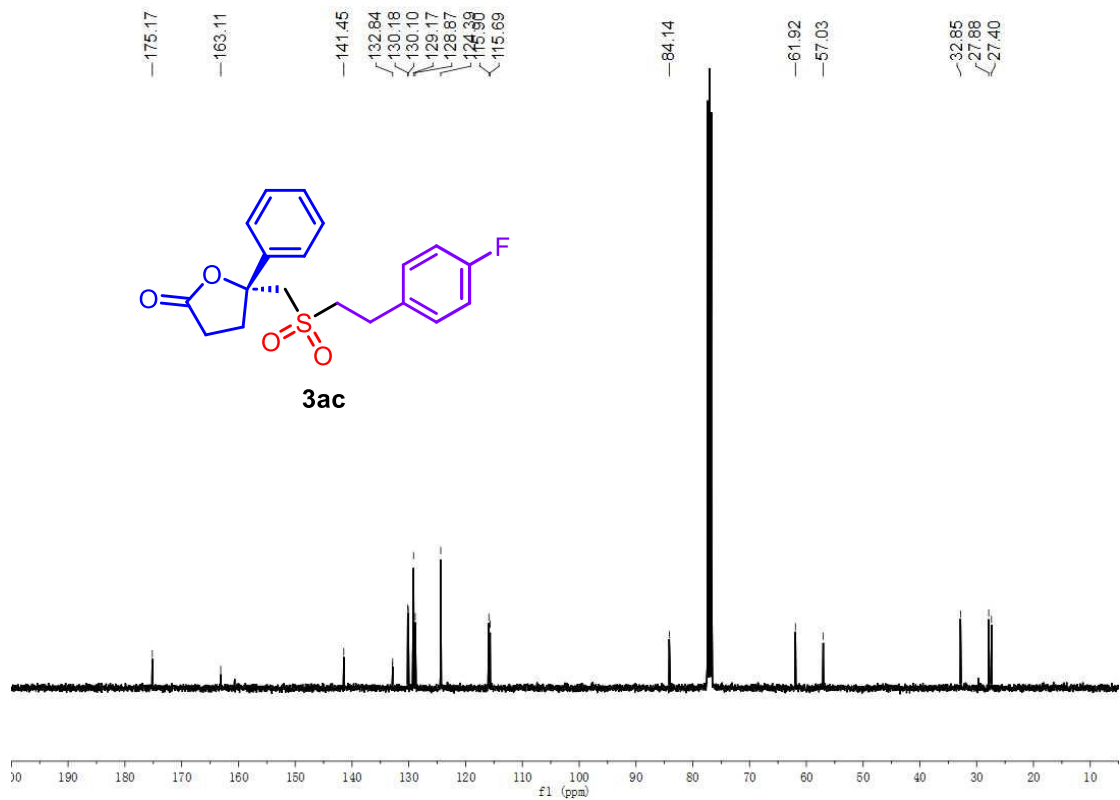


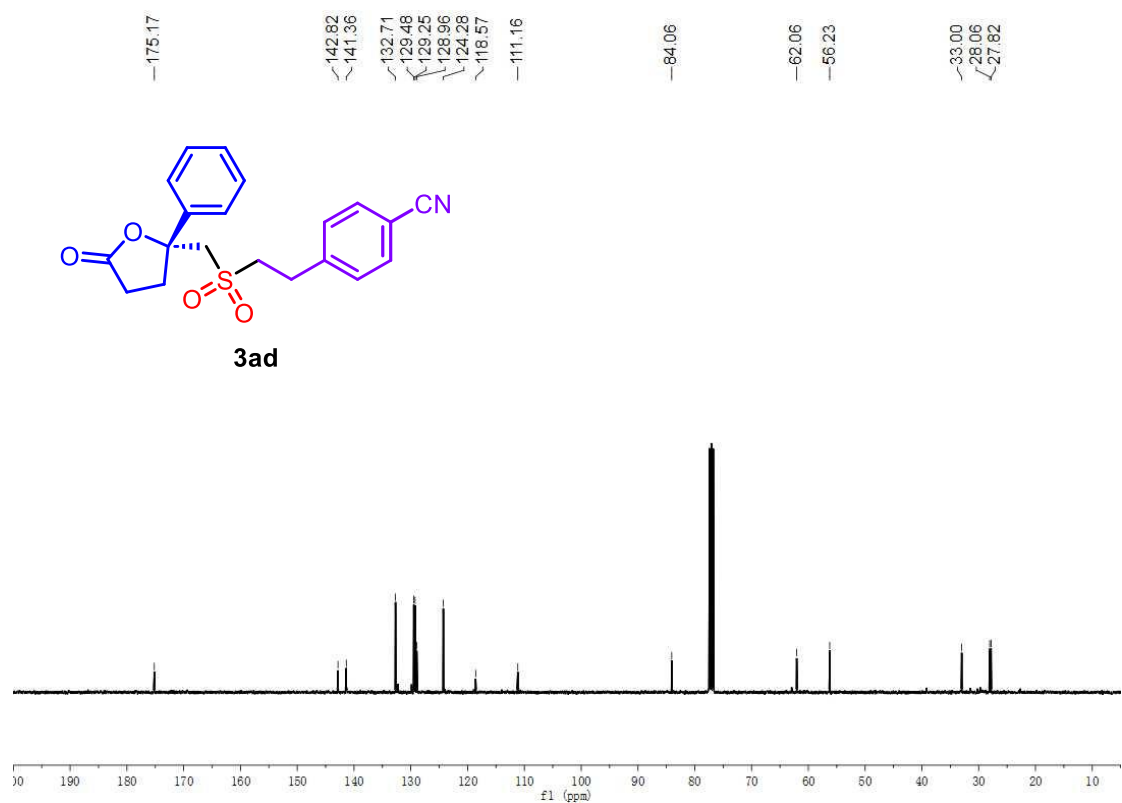
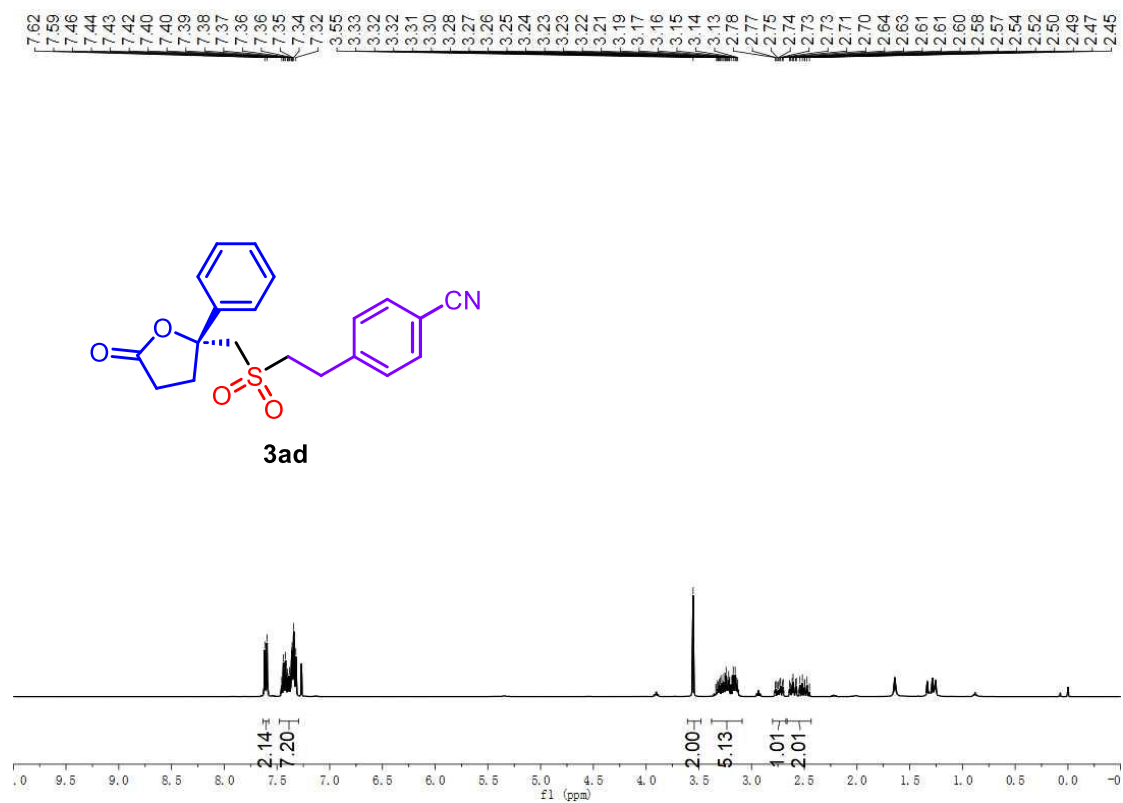


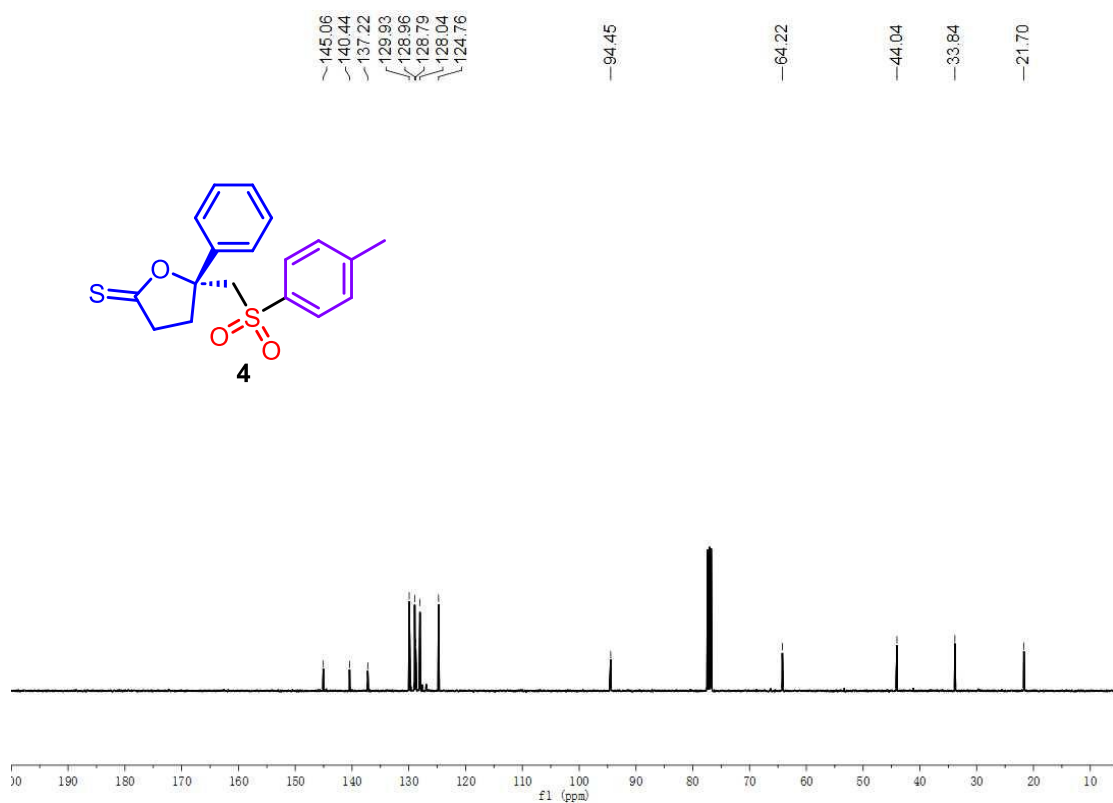
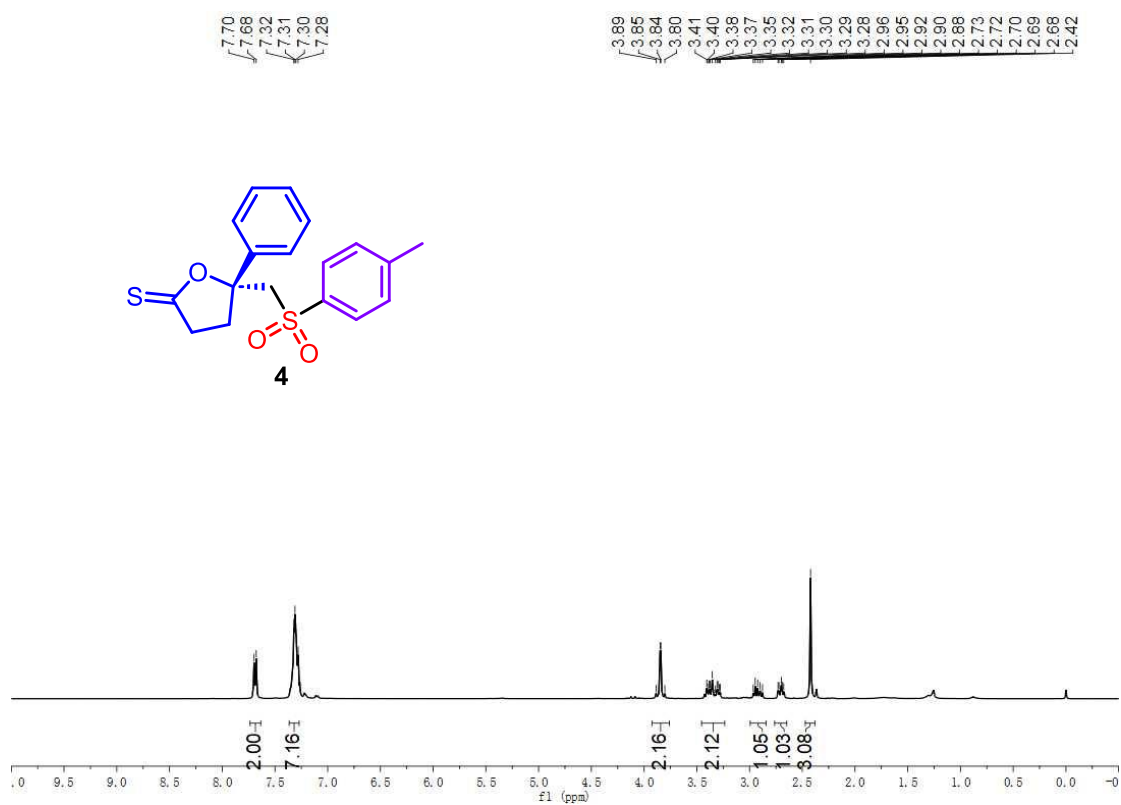


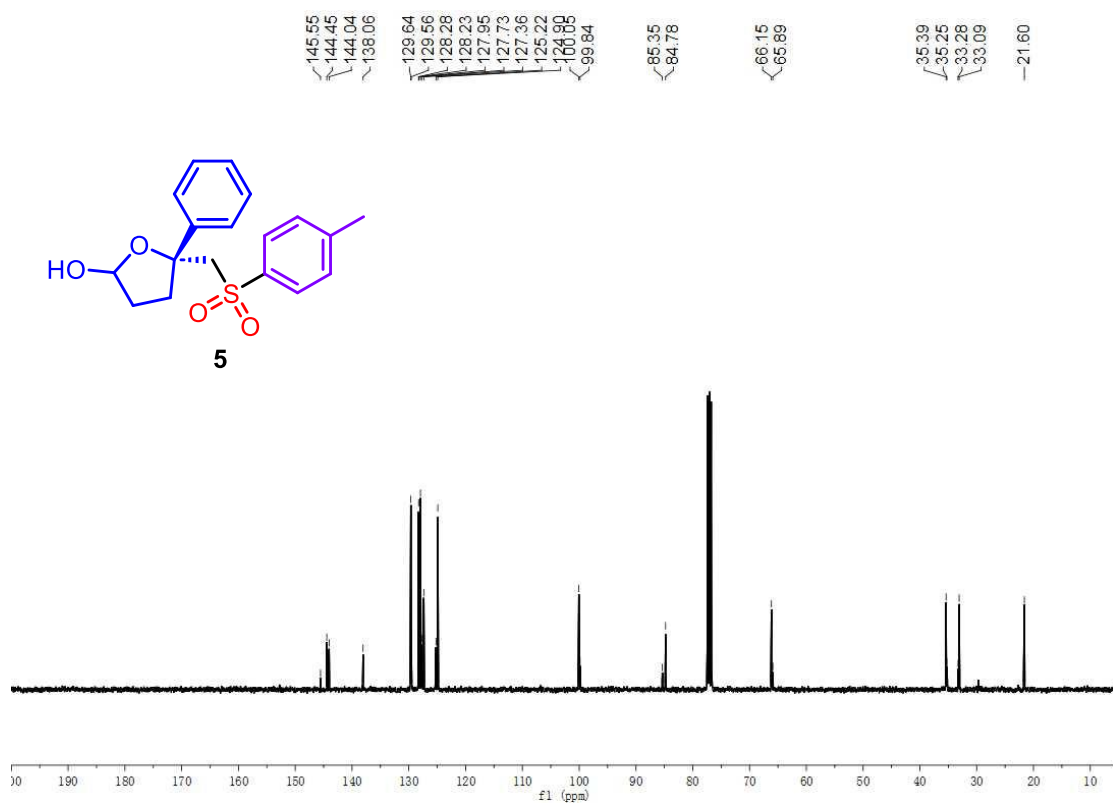
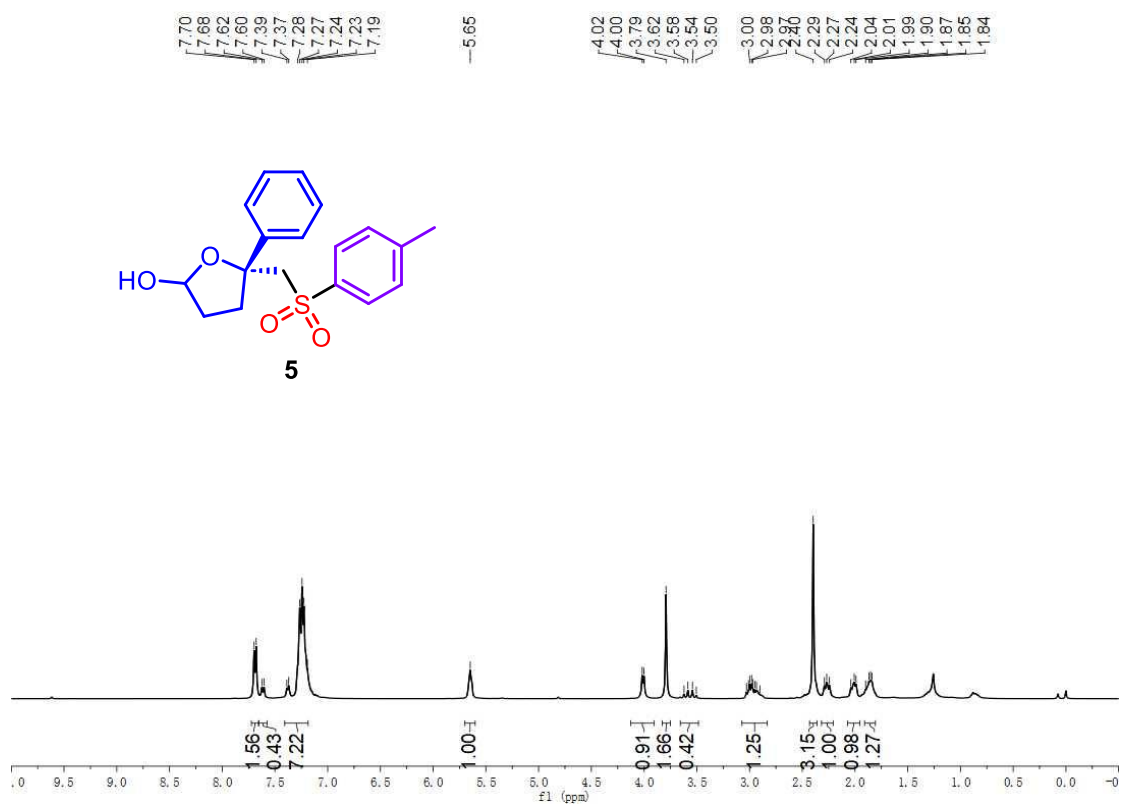












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