

## Supporting Information

### **Palladium(II)-Catalyzed Enantioselective Intermolecular Oxidative Diarylation of Internal Enamides**

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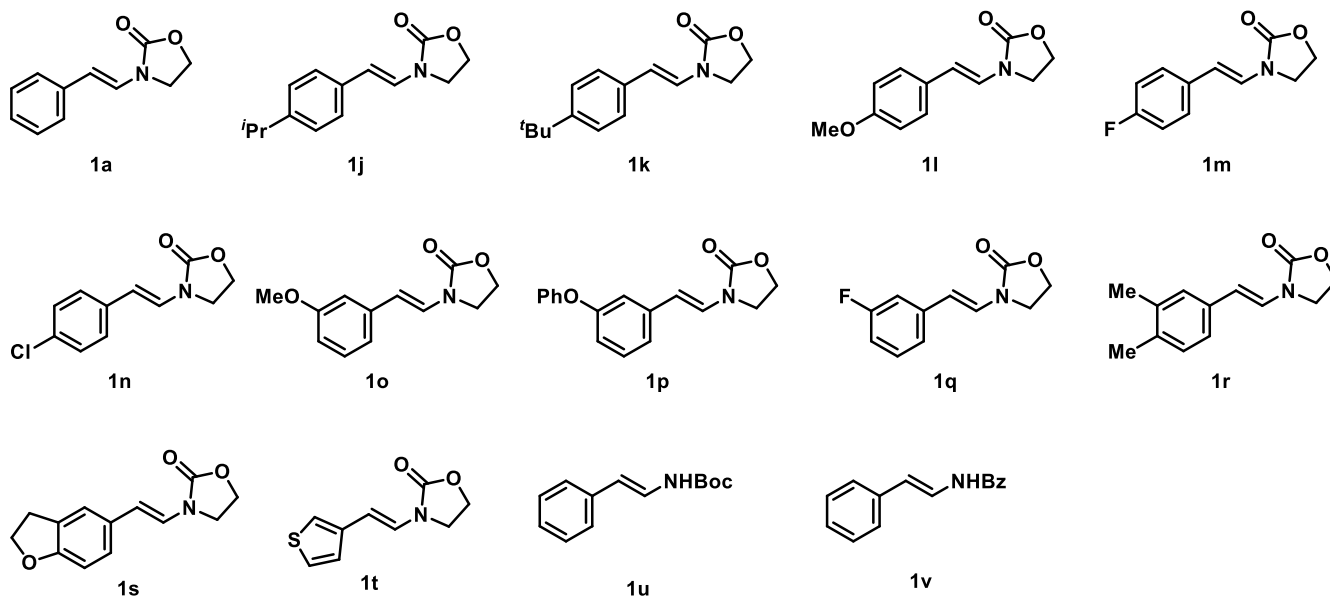
## General Information

All catalytic reactions were carried out using oven dried glassware and under oxygen atmosphere unless otherwise stated.  $\text{Ag}_2\text{CO}_3$  (CAS 534-16-7) was purchased from Bidepharm. 2,5-'Bu<sub>2</sub>BQ (CAS 7440-66-6) was purchased from D&B; DME (CAS 110-71-4) was purchased from from Adamas (99%, SafeDry, with molecular sieves, Water  $\leq$  50 ppm (by K.F.), SafeSeal);  $\text{Pd}(\text{OAc})_2$  (CAS 3375-31-3) was purchased from TCI. Reactions were monitored by thin-layer chromatography (TLC) carried out on 0.20 mm Huanghai silica gel plates (HSGF 254) using UV light as the visualizing agent, and an acidic solution of Phosphomolybdic Acid (PMA) with heat as the stains. All new compounds were characterized by means of <sup>1</sup>H NMR, <sup>13</sup>C NMR, <sup>19</sup>F NMR and HRMS. NMR spectra were recorded using a Bruker AVANCE III 400 MHz NMR spectrometer and can be found at the end of the paper. High-resolution mass spectra (HRMS) were recorded on a Q Exactive plus 4G mass spectrometer using ESI-Quadrupole-Orbitrap LC-MS. HPLC was performed on SHIMADZU LC-2030 Plus and SIL-20A. Optical rotations were recorded on digital automatic polarimeter (WZZ-2S). All <sup>1</sup>H NMR data are reported in  $\delta$  units, parts per million (ppm), and were calibrated relative to the signals for residual chloroform (7.26 ppm) in deuteriochloroform ( $\text{CDCl}_3$ ). All <sup>13</sup>C NMR data are reported in ppm relative to  $\text{CDCl}_3$  (77.16 ppm). <sup>19</sup>F NMR was recorded on a Bruker AVANCE III 400 NMR spectrometer ( $\text{CFCl}_3$  as an external standard and low field is positive) and were obtained with <sup>1</sup>H decoupling. The following abbreviations or combinations thereof were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, bs = broad singlet, m = multiplet.

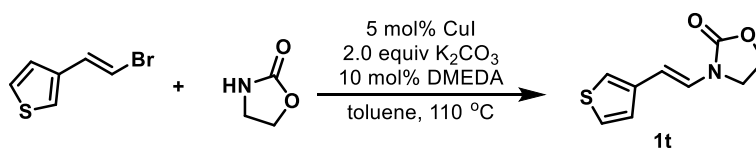
## Experimental procedures and characterization data

### Experimental procedures and characterization data for enamide 1t

Compounds **1a**,<sup>1</sup> **1j**,<sup>2</sup> **1k**,<sup>1</sup> **1l**,<sup>2</sup> **1m**,<sup>1</sup> **1n**,<sup>1</sup> **1o**,<sup>1</sup> **1p**,<sup>2</sup> **1q**,<sup>1</sup> **1r**,<sup>2</sup> **1s**,<sup>2</sup> **1u**<sup>3</sup>, **1v**<sup>4</sup> were synthesized according to the published procedures.



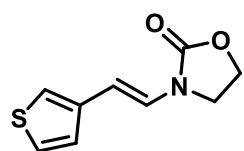
### Experimental procedure for the enamides **1t** synthesis



This procedure was performed according to the known literature<sup>5</sup>. Copper iodide (5 mol%), K<sub>2</sub>CO<sub>3</sub> (2 equiv) and the oxazolidin-2-one (1.5 equiv) are successively added in a tube and then dried by three successive vacuum-nitrogen cycles. Toluene was then added, followed by DMEDA (N,N'-dimethylethylenediamine, 10 mol%) and vinyl bromide<sup>6</sup> (1.0 equiv). The mixture is then heated at 110 °C overnight. When the starting materials were consumed, the reaction mixture was cooled to room temperature, diluted with EtOAc and filtered over silica. After concentration in vacuo the crude enamide is purified by flash chromatography on silica gel.

### Characterization data for enamide **1t**

#### (E)-3-(2-(thiophen-3-yl)vinyl)oxazolidin-2-one (**1t**)



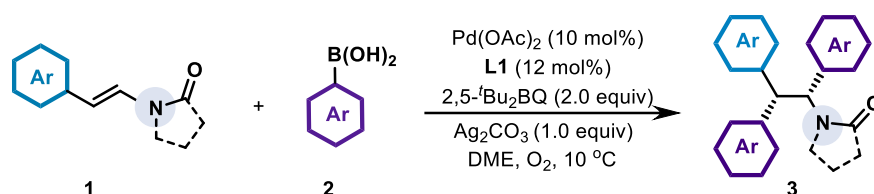
Experimental procedure was followed on 5.3 mmol scale and purification by flash column chromatography on silica gel to afford **1t** as a yellow solid (544.2 mg, 53%). R<sub>f</sub> = 0.53 (PE/Ea = 1/1);

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.23 – 7.19 (m, 2H), 7.11 (d, *J* = 4.8 Hz, 1H), 6.99 (d, *J* = 2.8 Hz, 1H), 5.76 (d, *J* = 14.8 Hz, 1H), 4.44 (t, *J* = 8.4 Hz, 2H), 3.76 (t, *J* = 8.0 Hz, 2H);

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 155.5, 137.7, 126.5, 124.6, 124.2, 120.2, 106.1, 62.4, 42.6.

HRMS (ESI): [M+H]<sup>+</sup> Calcd for C<sub>9</sub>H<sub>10</sub>NO<sub>2</sub>S<sup>+</sup>: 196.0427 found: 196.0428.

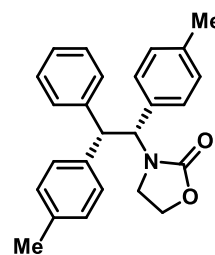
### General Procedures for Pd(II)-Catalyzed Enantioselective Intermolecular Oxidative Diarylation of Internal Enamides:



To an oven-dried 8 mL vial equipped with a magnetic stir bar was added Pd(OAc)<sub>2</sub> (10 mol%, 0.02 mmol, 4.5 mg), (*S*)-*i*-Pr-BiOx **L1** (12 mol %, 0.024 mmol, 5.4 mg) and DME (0.5 mL). The mixture was prestirred at room temperature for 5~10 min. Then enamide (1.0 equiv, 0.20 mmol), arylboronic acid (5.0 equiv, 1.0 mmol), 2,5-*t*Bu<sub>2</sub>BQ (2.0 equiv, 0.40 mmol, 88.1 mg), Ag<sub>2</sub>CO<sub>3</sub> (1.0 equiv, 0.20 mmol, 55.2 mg) and DME (0.5 mL) were subsequently added. The reaction mixture was purged with O<sub>2</sub> for 5-10 min and stirred at 10 or 25 °C for the required time. After the reaction completed, the reaction mixture was quenched with H<sub>2</sub>O and extracted with EA for three times. The combined organic phase was washed with brine and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure by rotary evaporation. The desired product was purified by column chromatography on silica gel.

## Characterization Data for Products:

### 3-((1*R*,2*S*)-2-phenyl-1,2-di-*p*-tolylethyl)oxazolidin-2-one (**3a**)



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/CHCl<sub>3</sub> = 5/1/0.5) afforded **3a** as a white solid (62.6 mg, 84%). **R<sub>f</sub>** = 0.43 (PE/Et<sub>2</sub>O/CHCl<sub>3</sub> = 2/1/0.5);

**[α]<sub>D</sub><sup>23.3</sup>** = - 107.06 (c = 0.17, CH<sub>2</sub>Cl<sub>2</sub>);

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): 7.47 (d, *J* = 7.2 Hz, 2H), 7.33 – 7.29 (m, 4H), 7.20 (d, *J* = 7.2 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 6.93 (d, *J* = 8.0 Hz, 2H), 5.89 (d, *J* = 12.4 Hz, 1H), 4.65 (d, *J* = 12.4 Hz, 1H), 4.00 (td, *J* = 8.4, 5.2 Hz, 1H), 3.81 (dt, *J* = 8.8, 8.4 Hz, 1H), 3.38 (td, *J* = 8.0, 4.8 Hz, 1H), 3.26 (dt, *J* = 8.8,

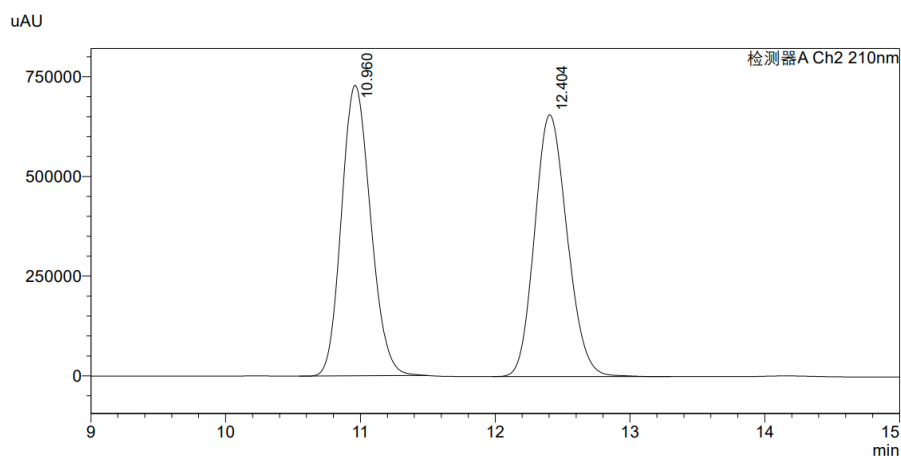
8.4 Hz, 1H), 2.26 (s, 3H), 2.16 (s, 3H);

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 157.9, 142.0, 138.3, 137.7, 136.1, 134.1, 129.4, 129.4, 128.9, 128.3, 127.9, 127.6, 127.0, 61.9, 59.0, 52.2, 40.3, 21.2, 21.0.

**HRMS (ESI)**: [M+H]<sup>+</sup> Calcd for C<sub>25</sub>H<sub>26</sub>NO<sub>2</sub><sup>+</sup>: 372.1958; found: 372.1955.

**HPLC** (Chiralcel AD-H): *n*-Hexane/EtOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 210 nm, t<sub>R</sub> = 13.707 min (major), t<sub>R</sub> = 11.999 min (minor); 97% ee.

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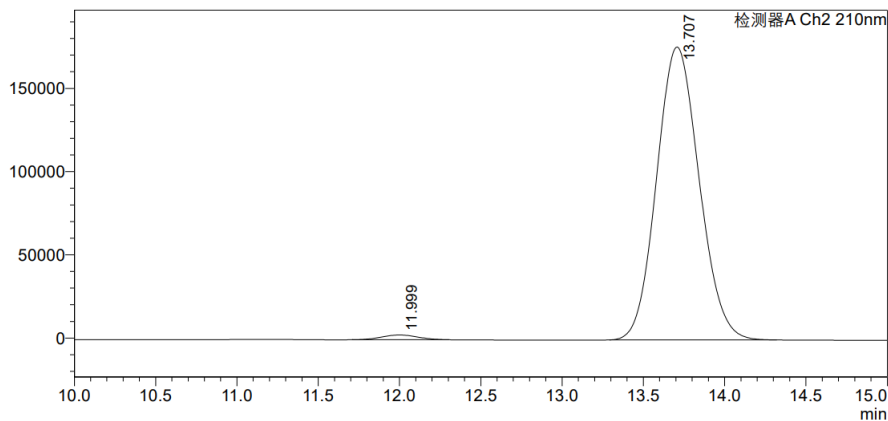


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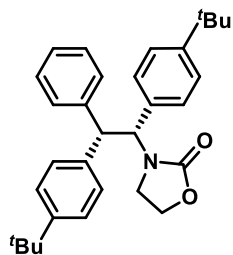
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Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	11.999	44270	2868	1.363		M
2	13.707	3203703	176023	98.637		M
总计		3247972	178891			

**3-((1R,2S)-1,2-bis(4-(tert-butyl)phenyl)-2-phenylethyl)oxazolidin-2-one (3b)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/DCM = 10/1/2) afforded **3b** as a white solid (68.1 mg, 75%). **R<sub>f</sub>** = 0.27 (PE/Et<sub>2</sub>O/DCM = 2/1/0.5);

**[α]<sub>D</sub><sup>23.3</sup>** = - 103.08 (c = 0.26, CH<sub>2</sub>Cl<sub>2</sub>);

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): 7.47 (d, *J* = 7.6 Hz, 2H), 7.31 – 7.29 (m, 4H), 7.25 (d, *J* = 6.4 Hz, 2H), 7.20 (d, *J* = 7.2 Hz, 1H), 7.18 – 7.11 (m, 4H), 5.88 (d, *J* = 12.4 Hz, 1H), 4.63 (d, *J* = 12.4 Hz, 1H), 4.02 (td, *J* = 8.8, 4.8 Hz, 1H), 3.78 (dt, *J* = 8.8, 8.4 Hz, 1H), 3.35 (td, *J* = 8.0, 5.2 Hz, 1H), 3.25 (dt, *J* = 8.8, 8.4 Hz, 1H), 1.25 (s,

9H), 1.17 (s, 9H);

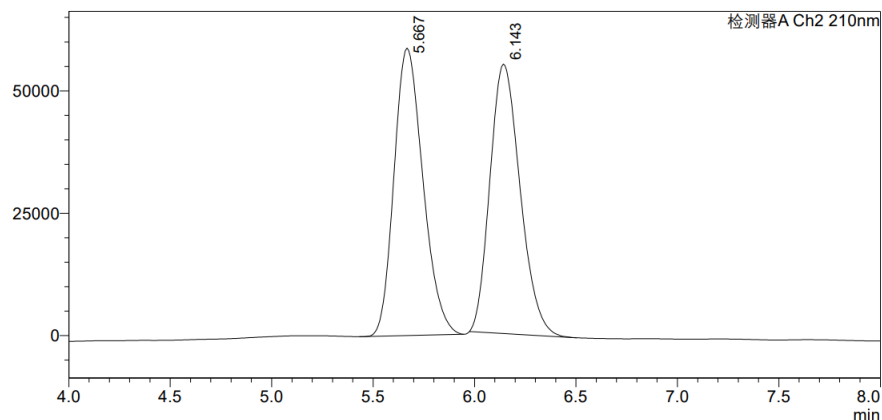
**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 157.9, 150.7, 149.1, 142.2, 138.2, 134.1, 128.9, 128.0, 127.8, 127.6, 127.0, 125.6, 125.5, 61.8, 58.8, 52.2, 40.4, 34.6, 34.4, 31.4, 31.3.

**HRMS (ESI)**: [M+H]<sup>+</sup> Calcd for C<sub>31</sub>H<sub>38</sub>NO<sub>2</sub><sup>+</sup>: 456.2897; found: 456.2898.

**HPLC** (Chiralcel AD-H): *n*-Hexane/EtOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 210 nm, *t<sub>R</sub>* = 5.641 min (major), *t<sub>R</sub>* = 6.088 min (minor); >99% ee.

<Chromatogram>

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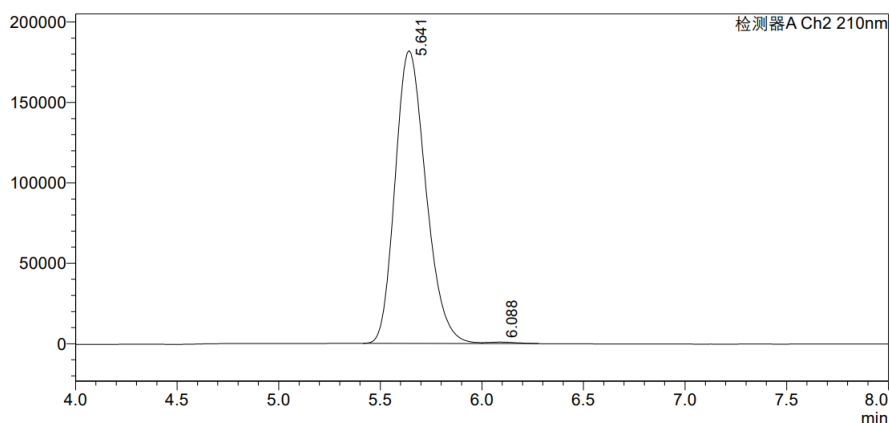


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2	6.143	570057	55047	49.432		M
总计		1153208	113782			

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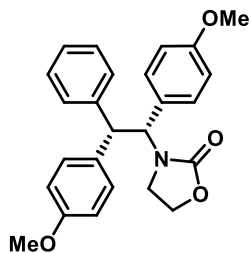
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检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	5.641	1917957	181989	99.525		M
2	6.088	9163	926	0.475		V M
总计		1927120	182914			

**3-((1R,2S)-1,2-bis(4-methoxyphenyl)-2-phenylethyl)oxazolidin-2-one (3c)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/acetone/CHCl<sub>3</sub> = 8/1/0.5~6/1/0.5) afforded **3c** as a white solid (58.6 mg, 73%). **R<sub>f</sub>** = 0.42 (PE/acetone/CHCl<sub>3</sub> = 4/1/0.5);

**[α]<sub>D</sub><sup>23.3</sup>** = - 101.52 (c = 0.22, CH<sub>2</sub>Cl<sub>2</sub>);

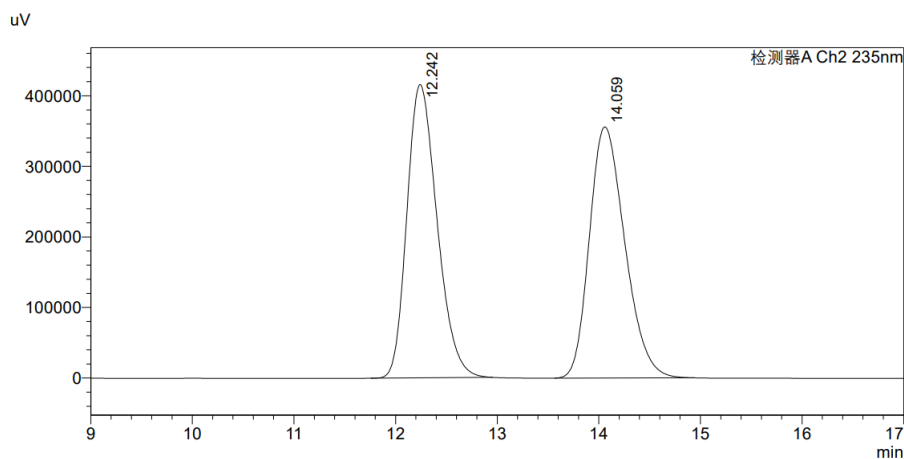
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): 7.45 (d, *J* = 7.2 Hz, 2H), 7.33 – 7.29 (m, 4H), 7.19 (t, *J* = 7.2 Hz, 1H), 7.15 (d, *J* = 8.8 Hz, 2H), 6.78 (d, *J* = 8.8 Hz, 2H), 6.66 (d, *J* = 8.8 Hz, 2H), 5.81 (d, *J* = 12.4 Hz, 1H), 4.59 (d, *J* = 12.4 Hz, 1H), 4.02 (td, *J* = 8.8, 5.2 Hz, 1H), 3.82 (dt, *J* = 8.8, 8.4 Hz, 1H), 3.74 (s, 3H), 3.65 (s, 3H), 3.38 (td, *J* = 8.0, 4.8 Hz, 1H), 3.27 (dt, *J* = 8.8, 8.4 Hz, 1H);

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  159.1, 158.0, 157.9, 142.1, 133.5, 129.6, 129.3, 129.0, 128.9, 127.6, 127.0, 114.1, 114.0, 61.9, 58.9, 55.3, 55.1, 52.0, 40.4.

HRMS (ESI):  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{25}\text{H}_{26}\text{NO}_4^+$ : 404.1856; found: 404.1859.

HPLC (Chiralcel OD-H): *n*-Hexane/EtOH = 90/10, flow rate 1.0 mL/min,  $T = 40\text{ }^\circ\text{C}$ ,  $\lambda = 235\text{ nm}$ ,  $t_{\text{R}} = 14.136\text{ min}$  (major),  $t_{\text{R}} = 12.334\text{ min}$  (minor); 94% ee.

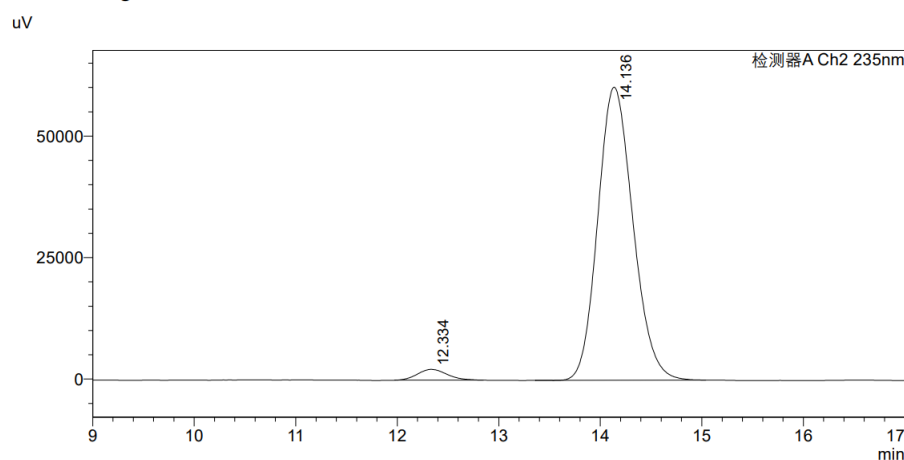
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1	12.242	8584128	415634	49.831		M
2	14.059	8642358	355986	50.169		M
总计		17226486	771620			

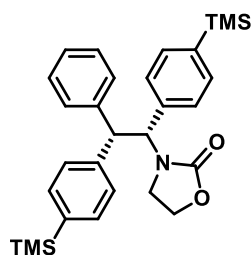
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检测器A Ch2 235nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	12.334	45201	2219	3.018		M
2	14.136	1452379	60303	96.982		M
总计		1497580	62522			

### 3-((1*R*,2*S*)-2-phenyl-1,2-bis(4-(trimethylsilyl)phenyl)ethyl)oxazolidin-2-one (3d)



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/DCM = 10/1/1~5/1/1) afforded **3d** as a white solid (58.8 mg, 60%).  $R_f = 0.40$  (PE/Et<sub>2</sub>O/DCM = 4/1/1);

$[\alpha]_{\text{D}}^{23.3} = -98.75$  ( $c = 0.16$ ,  $\text{CH}_2\text{Cl}_2$ );



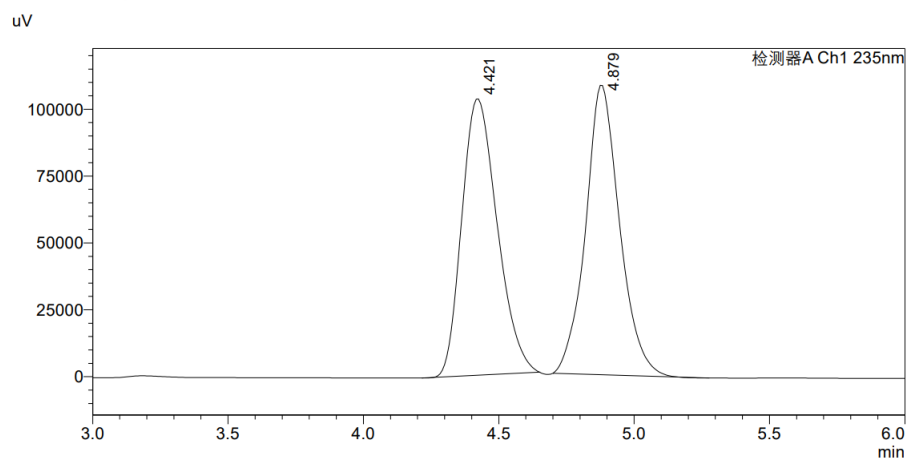
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): 7.49 (d, *J* = 7.6 Hz, 2H), 7.41 (m, 4H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.28 (m, 4H), 7.22 (t, *J* = 7.4 Hz, 1H), 5.96 (d, *J* = 12.4 Hz, 1H), 4.70 (d, *J* = 12.4 Hz, 1H), 4.03 (td, *J* = 8.8, 4.8 Hz, 1H), 3.81 (dt, *J* = 8.8, 8.4 Hz, 1H), 3.39 (td, *J* = 8.4, 5.2 Hz, 1H), 3.26 (dt, *J* = 8.8, 8.4 Hz, 1H), 0.23 (s, 9H), 0.16 (s, 9H);

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 157.9, 141.8, 141.6, 140.3, 138.4, 137.4, 133.8, 133.7, 128.9, 127.8, 127.7, 127.4, 127.1, 61.9, 58.9, 52.4, 40.3, -1.1.

**HRMS (ESI):** [M+H]<sup>+</sup> Calcd for C<sub>29</sub>H<sub>38</sub>NO<sub>2</sub>Si<sub>2</sub><sup>+</sup>: 488.2436; found: 488.2435.

**HPLC** (Chiralcel AD-H): *n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 235 nm, t<sub>R</sub> = 4.347 min (major), t<sub>R</sub> = 4.845 min (minor); 95% ee.

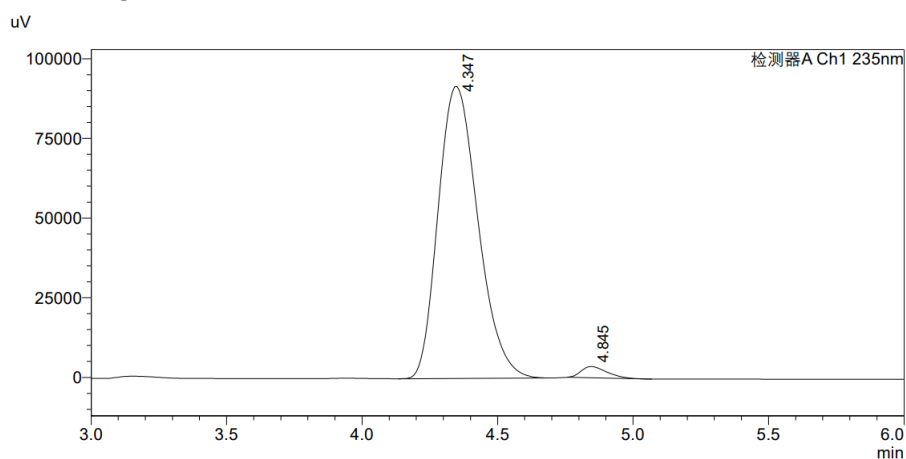
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2	4.879	945484	108186	50.119		M
总计		1886475	211466			

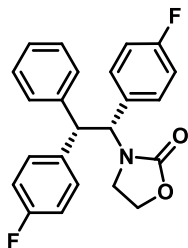
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检测器A Ch1 235nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	4.347	938946	91669	97.601		M
2	4.845	23082	3518	2.399		M
总计		962029	95187			

**3-((1R,2S)-1,2-bis(4-fluorophenyl)-2-phenylethyl)oxazolidin-2-one (3e)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/acetone/ $\text{CHCl}_3 = 10/1/1 \sim 8/1/1$ ) afforded **3e** as a white solid (29.4 mg, 39%).  $R_f = 0.20$  (PE/acetone/ $\text{CHCl}_3 = 8/1/1$ );

$[\alpha]_D^{23.3} = -88.89$  ( $c = 0.15$ ,  $\text{CH}_2\text{Cl}_2$ );

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ): 7.44 (d,  $J = 7.2$  Hz, 2H), 7.36 – 7.32 (m, 4H), 7.23 (t,  $J = 7.6$  Hz, 1H), 7.19 – 7.16 (m, 2H), 6.95 (t,  $J = 8.4$  Hz, 2H), 6.82 (t,  $J = 8.8$  Hz, 2H), 5.80 (d,  $J = 12.8$  Hz, 1H), 4.64 (d,  $J = 12.4$  Hz, 1H), 4.06 (td,  $J = 8.8, 5.2$  Hz, 1H), 3.86 (dt,  $J = 8.8, 8.4$  Hz, 1H), 3.40 (td,  $J = 8.0, 5.2$  Hz, 1H), 3.30 (dt,  $J = 8.8, 8.4$  Hz, 1H)

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  162.3 (d,  $J_{C-F} = 245.9$  Hz), 161.4 (d,  $J_{C-F} = 244.4$  Hz), 157.9, 141.1, 136.9 (d,  $J_{C-F} = 3.5$  Hz), 132.8 (d,  $J_{C-F} = 3.4$  Hz), 130.0 (d,  $J_{C-F} = 8.0$  Hz), 129.0 (d,  $J_{C-F} = 8.0$  Hz), 129.1, 127.6, 127.4, 115.8 (d,  $J_{C-F} = 21.3$  Hz), 115.7 (d,  $J_{C-F} = 21.3$  Hz), 62.0, 59.0, 52.2, 40.5.

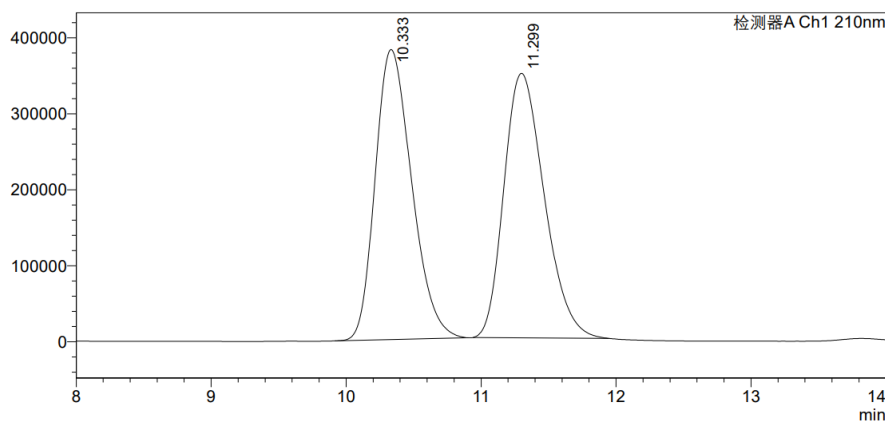
$^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ ):  $\delta$  -113.5, -115.8;

**HRMS (ESI)**:  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{23}\text{H}_{20}\text{F}_2\text{NO}_2^+$ : 380.1457; found: 380.1465.

**HPLC** (Chiralcel OD-H): *n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min,  $T = 40$  °C,  $\lambda = 210$  nm,  $t_R = 11.385$  min (major),  $t_R = 10.440$  min (minor); 98% ee.

<Chromatogram>

uV

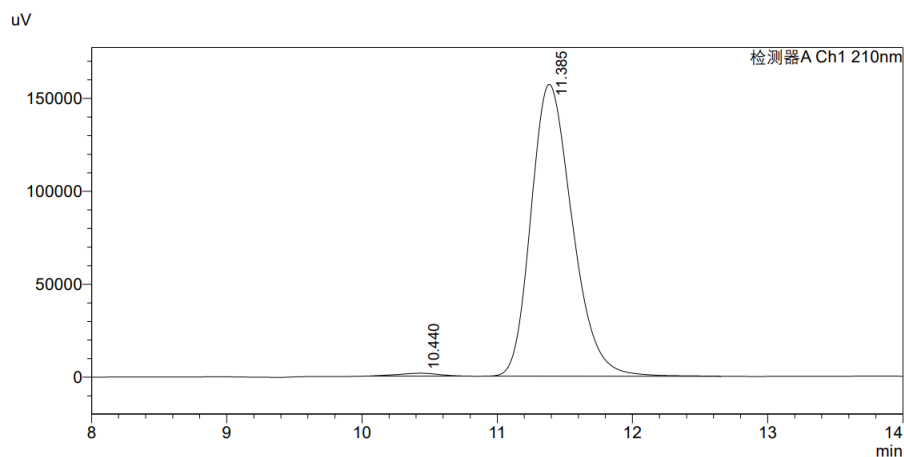


<Peak Table>

检测器A Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	10.333	7180475	381864	49.809		M
2	11.299	7235614	348092	50.191		M
总计		14416089	729956			

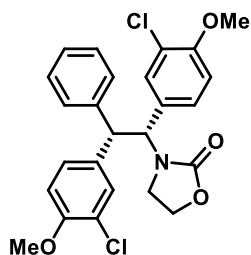
<Chromatogram>



<Peak Table>

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	10.440	33921	1554	1.008		M
2	11.385	3331230	157031	98.992		M
总计		3365151	158585			

**3-((1R,2S)-1,2-bis(3-chloro-4-methoxyphenyl)-2-phenylethyl)oxazolidin-2-one (3f)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/DCM = 4/1/1~3/1/1) afforded **3f** as a white solid (63.1 mg, 67%). **R<sub>f</sub>** = 0.16 (PE/Et<sub>2</sub>O/CHCl<sub>3</sub> = 2/1/0.5);

**[α]<sub>D</sub><sup>23.3</sup>** = - 125.42 (c = 0.16, CH<sub>2</sub>Cl<sub>2</sub>);

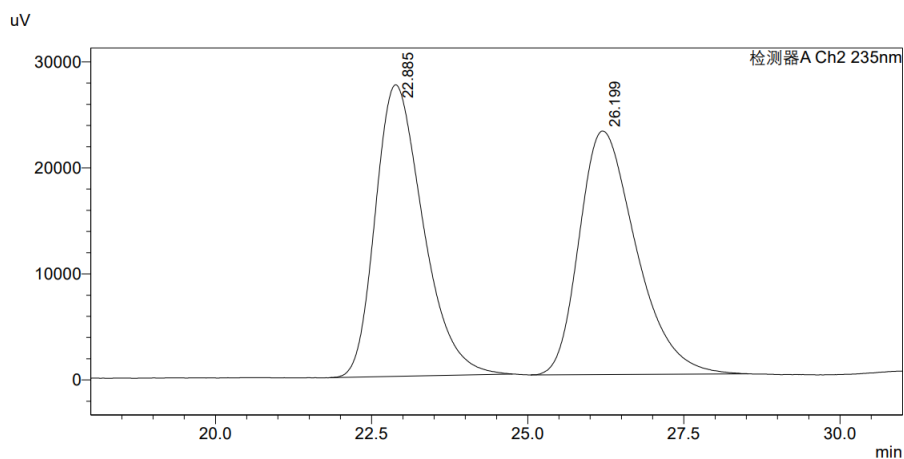
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): 7.43 – 7.40 (m, 3H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.24 – 7.20 (m, 3H), 7.09 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.80 (d, *J* = 8.4 Hz, 1H), 6.69 (d, *J* = 8.8 Hz, 1H), 5.73 (d, *J* = 12.8 Hz, 1H), 4.53 (d, *J* = 12.8 Hz, 1H), 4.05 (td, *J* = 8.8, 5.2 Hz, 1H), 3.87 – 3.81 (m, 4H), 3.76 (s, 3H), 3.36 (td, *J* = 8.4, 5.2 Hz, 1H), 3.28 (dt, *J* = 8.4, 8.4 Hz, 1H);

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 157.8, 154.7, 153.6, 141.0, 134.2, 129.9, 129.9, 129.7, 129.1, 128.1, 127.5, 127.4, 127.0, 122.6, 122.5, 112.3, 112.2, 61.9, 58.5, 56.2, 56.1, 51.6, 40.4.

**HRMS (ESI)**: [M-Cl+Na]<sup>+</sup> Calcd for C<sub>25</sub>H<sub>23</sub>ClNO<sub>4</sub>Na<sup>+</sup>: 459.1208; found: 459.1214.

**HPLC** (Chiralcel OD-H): *n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 235 nm, *t<sub>R</sub>* = 25.922 min (major), *t<sub>R</sub>* = 23.155 min (minor); 98% ee.

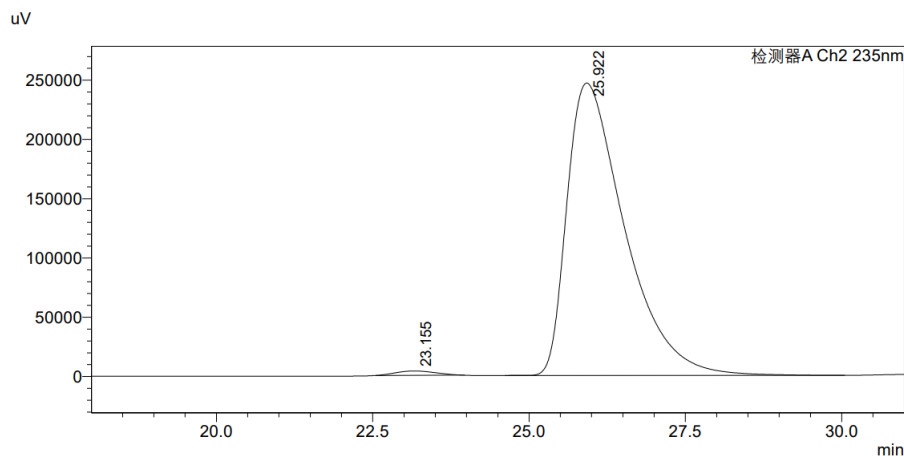
<Chromatogram>



<Peak Table>

检测器A Ch2 235nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	22.885	1462228	27496	50.117		M
2	26.199	1455411	22965	49.883		M
总计		2917640	50461			

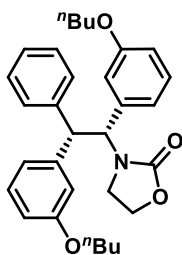
<Chromatogram>



<Peak Table>

检测器A Ch2 235nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	23.155	163519	3672	1.020		M
2	25.922	15872120	246740	98.980		M
总计		16035640	250412			

**3-((1R,2S)-1,2-bis(3-butoxyphenyl)-2-phenylethyl)oxazolidin-2-one (3g)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/DCM = 8/1/1~5/1/1) afforded **3g** as a white solid (49.2 mg, 50%). *R<sub>f</sub>* = 0.23 (PE/Et<sub>2</sub>O/DCM = 4/1/1);

$[\alpha]_D^{23.3} = -87.45$  (c = 0.17, CH<sub>2</sub>Cl<sub>2</sub>);

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 7.47 (d, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.20 (t, *J* = 7.2 Hz, 1H), 7.15 (t, *J* = 7.6 Hz, 1H), 7.02 (t, *J* = 8.0 Hz, 1H), 6.96 (d, *J* = 7.6 Hz, 1H), 6.93 (bs, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.80 (bs, 1H), 6.73 (dd, *J* = 8.0, 2.4 Hz, 1H), 6.56 (dd, *J* = 8.0, 2.4 Hz, 1H), 5.85 (d, *J* = 12.4 Hz, 1H), 4.61 (d, *J* = 12.4 Hz, 1H),

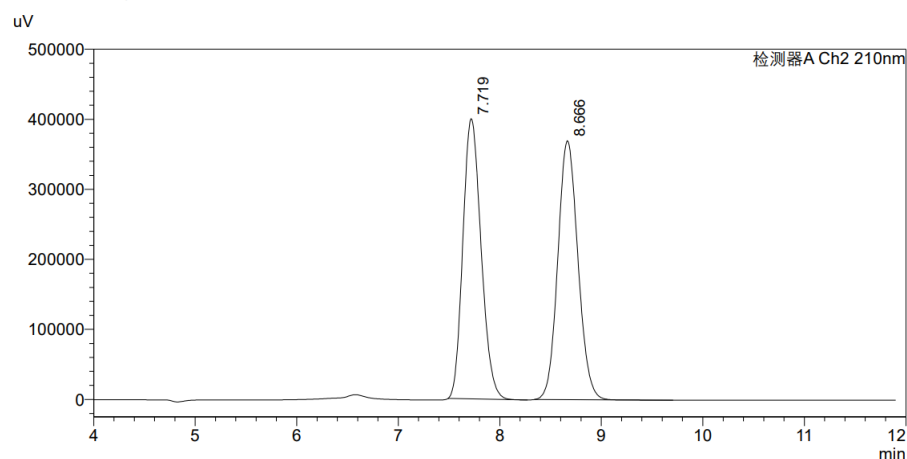
4.03 (td, *J* = 9.2, 5.2 Hz, 1H), 3.88 (t, *J* = 6.4 Hz, 2H), 3.85 – 3.79 (m, 3H), 3.38 (td, *J* = 8.4, 5.2 Hz, 1H), 3.31 (dt, *J* = 8.8, 8.4 Hz, 1H), 1.75 – 1.64 (m, 4H), 1.51 – 1.38 (m, 4H), 0.95 (dt, *J* = 10.0, 7.6 Hz, 6H);

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 159.4, 159.2, 157.9, 142.7, 141.5, 138.5, 129.6, 129.6, 129.0, 127.7, 127.2, 120.4, 120.3, 114.9, 114.6, 114.0, 112.5, 67.8, 67.6, 61.9, 59.2, 52.6, 40.5, 31.4, 19.4, 19.3, 14.0.

**HRMS (ESI):** [M+Na]<sup>+</sup> Calcd for C<sub>31</sub>H<sub>37</sub>NO<sub>4</sub>Na<sup>+</sup>: 510.2615; found: 510.2626

**HPLC** (Chiralcel AD-H): *n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 210 nm, *t<sub>R</sub>* = 7.707 min (major), *t<sub>R</sub>* = 8.658 min (minor); >99% ee.

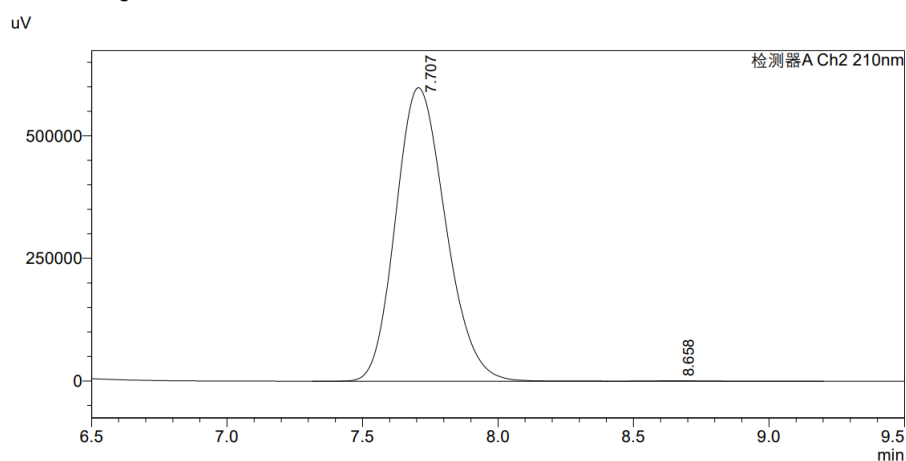
<Chromatogram>



<Peak Table>

检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	7.719	4992977	399916	49.508		M
2	8.666	5092234	369786	50.492		M
总计		10085211	769702			

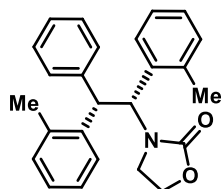
<Chromatogram>



<Peak Table>

检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	7.707	7683431	599057	99.873		M
2	8.658	9757	790	0.127		M
总计		7693188	599846			

**3-((1*R*,2*S*)-2-phenyl-1,2-di-*o*-tolylethyl)oxazolidin-2-one (3h)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/CHCl<sub>3</sub> = 10/1/0.5~5/1/0.5) afforded **3h** as a white solid (47.5 mg, 64%). **R<sub>f</sub>** = 0.49 (PE/acetone/CHCl<sub>3</sub> = 5/1/0.5);

**[α]<sub>D</sub><sup>23.3</sup>** = -297.50 (c = 0.16, CH<sub>2</sub>Cl<sub>2</sub>);

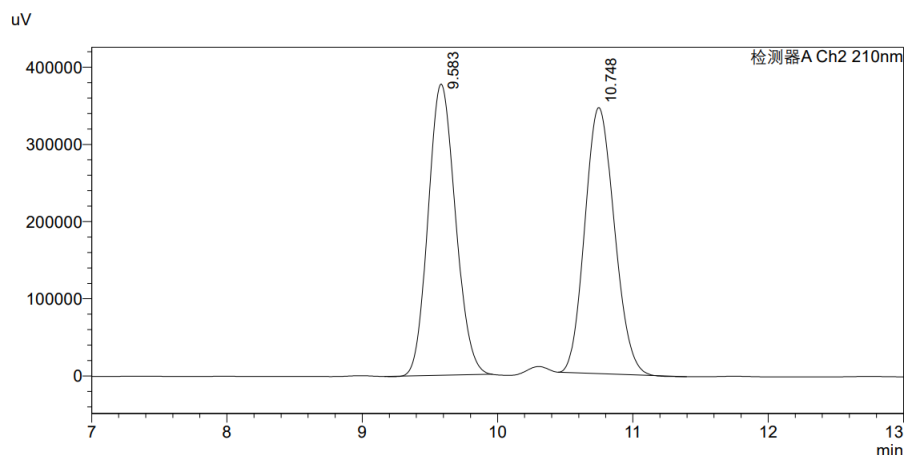
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): 7.45 (d, *J* = 7.2 Hz, 2H), 7.37 (d, *J* = 7.6 Hz, 1H), 7.31 (t, *J* = 7.2 Hz, 2H), 7.23 – 7.20 (m, 2H), 7.14 (d, *J* = 6.8 Hz, 1H), 7.11 – 7.03 (m, 3H), 7.00 – 6.94 (m, 2H), 6.12 (d, *J* = 12.0 Hz, 1H), 4.95 (d, *J* = 12.4 Hz, 1H), 4.06 (td, *J* = 8.4, 5.6 Hz, 1H), 3.95 (dt, *J* = 8.8, 8.4 Hz, 1H), 3.61 (td, *J* = 8.8, 5.6 Hz, 1H), 3.16 (dt, *J* = 8.8, 8.4 Hz, 1H), 2.51 (s, 3H), 2.36 (s, 3H);

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 157.4, 140.5, 139.2, 137.8, 135.8, 135.3, 131.4, 130.8, 128.7, 127.8, 127.3, 126.5, 126.5, 126.5, 126.3, 125.6, 61.8, 56.1, 47.3, 40.8, 20.3, 20.2.

**HRMS (ESI):** [M+H]<sup>+</sup> Calcd for C<sub>25</sub>H<sub>26</sub>NO<sub>2</sub><sup>+</sup>: 372.1958; found: 372.1958.

**HPLC** (Chiralcel AD-H): *n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, T = 40 °C,  $\lambda$  = 210 nm,  $t_R$  = 10.339 min (major),  $t_R$  = 8.998 min (minor); 71% ee.

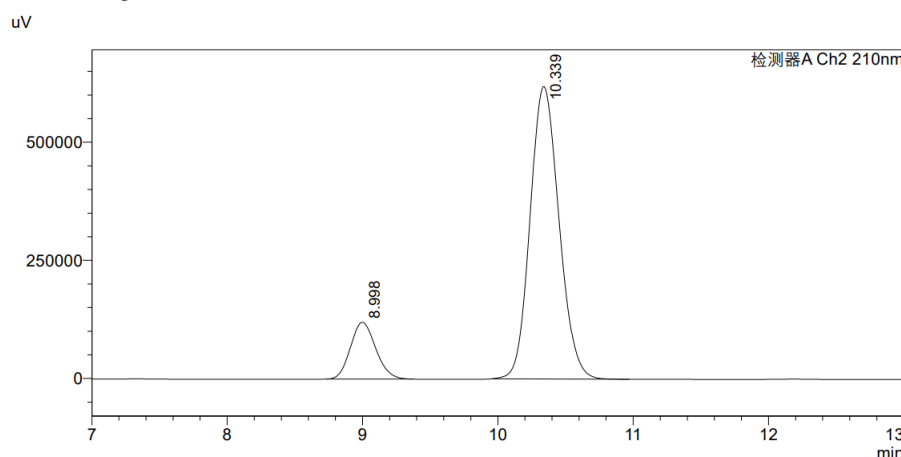
<Chromatogram>



<Peak Table>

检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	9.583	5307150	377433	50.333		M
2	10.748	5237009	344787	49.667		M
总计		10544159	722220			

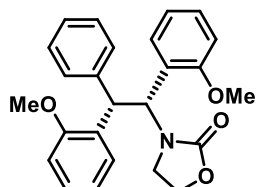
<Chromatogram>



<Peak Table>

检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	8.998	1563597	120311	14.526		M
2	10.339	9200170	618857	85.474		M
总计		10763767	739168			

### 3-((1R,2S)-1,2-bis(2-methoxyphenyl)-2-phenylethyl)oxazolidin-2-one (**3i**)



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/acetone/CHCl<sub>3</sub> = 10/1/0.5~4/1/0.5) afforded **3i** as a white solid (47.1 mg, 58%);

$[\alpha]_D^{29.1} = -10.91$  (c = 0.22, CHCl<sub>2</sub>);

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 7.53 (d, *J* = 7.2 Hz, 2H), 7.36 – 7.33 (m, 2H), 7.29 (d, *J* = 7.2 Hz, 2H), 7.19 – 7.12 (m, 2H), 6.99 (td, *J* = 7.8, 1.2 Hz, 1H), 6.81 – 6.03 (m, 3H), 6.66 (d, *J* = 8.0 Hz, 1H), 6.24 (d, *J* = 12.8 Hz, 1H), 5.40 (d, *J* = 12.8 Hz, 1H), 3.97 (td, *J* = 8.8, 6.0 Hz, 1H), 3.88 (dt, *J* = 8.4, 8.4 Hz, 1H), 3.84 (s, 3H), 3.76 (s, 3H), 3.56 (td, *J* = 8.4, 6.0 Hz, 1H), 3.21 (dt, *J* = 8.8, 8.0 Hz, 1H);

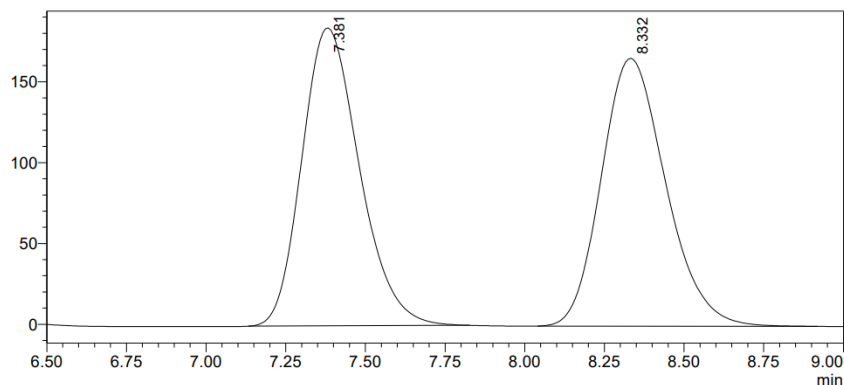
<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 157.7, 157.4, 156.5, 141.7, 130.2, 129.1, 128.9, 128.5, 128.4, 127.7, 127.4, 126.7, 125.7, 120.6, 120.1, 110.8, 110.7, 61.7, 55.7, 55.6, 53.8, 43.4, 41.9.

HRMS (ESI): [M+H]<sup>+</sup> Calcd for C<sub>25</sub>H<sub>26</sub>NO<sub>4</sub><sup>+</sup>: 404.1856; found: 404.1854.

HPLC (Chiralcel AS-H): n-Hexane/EtOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 210 nm, t<sub>R</sub> = 8.343 min (major), t<sub>R</sub> = 7.388 min (minor); 48% ee.

<Chromatogram>

mV



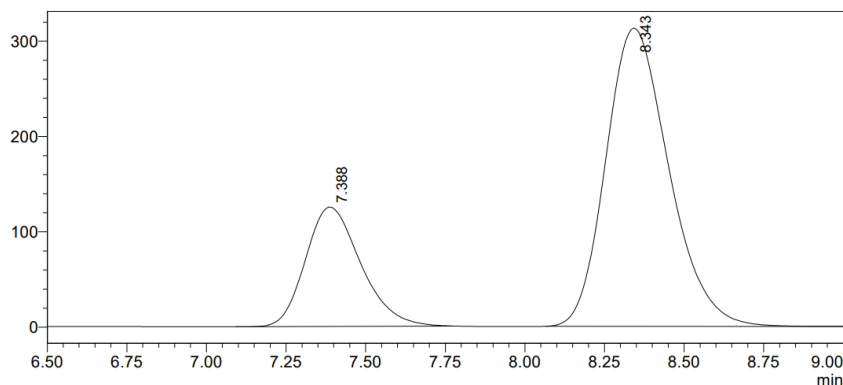
<Peak Table>

检测器A Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	7.381	2318568	184382	49.898		M
2	8.332	2328026	165698	50.102		M

<Chromatogram>

mV

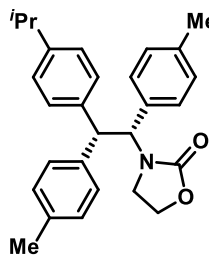


<Peak Table>

检测器A Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	7.388	1513628	125065	26.009		M
2	8.343	4306031	312783	73.991		M

### 3-((1*R*,2*R*)-2-(4-isopropylphenyl)-1,2-di-*p*-tolylethyl)oxazolidin-2-one (**3j**)



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/DCM = 10/1/2-5/1/1) afforded **3j** as a white solid (59.6 mg, 72%). R<sub>f</sub> = 0.29 (PE/Et<sub>2</sub>O/DCM = 5/1/1);

[α]<sub>D</sub><sup>23.3</sup> = - 140.42 (c = 0.16, CH<sub>2</sub>Cl<sub>2</sub>);

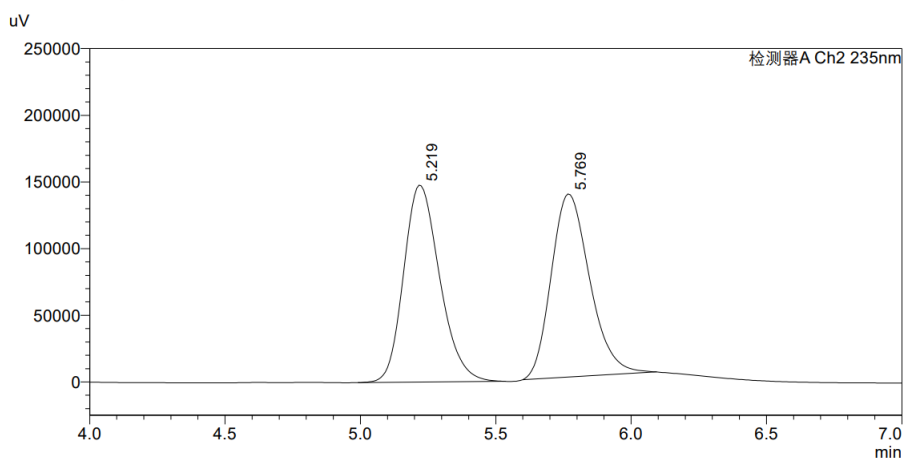
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 7.37 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 4H), 7.05 (d, *J* = 8.0 Hz, 2H), 6.93 (d, *J* = 8.0 Hz, 2H), 5.87 (d, *J* = 12.4 Hz, 1H), 4.60 (d, *J* = 12.4 Hz, 1H), 4.00 (td, *J* = 8.8, 5.2 Hz, 1H), 3.80 (td, *J* = 8.8, 8.4 Hz, 1H), 3.38 (td, *J* = 8.4, 4.8 Hz, 1H), 3.26 (dt, *J* = 8.8, 8.4 Hz, 1H), 2.84 (sept, *J* = 7.2 Hz, 1H), 2.26 (s, 3H), 2.16 (s, 3H), 1.20 (d, *J* = 7.2 Hz, 6H);

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  158.0, 147.3, 139.2, 138.7, 137.6, 135.9, 134.2, 129.4, 129.4, 128.3, 127.9, 127.4, 127.0, 61.9, 59.0, 51.9, 40.3, 33.7, 24.0, 21.2, 21.0.

HRMS (ESI):  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{28}\text{H}_{32}\text{NO}_2$ : 414.2428; found: 414.2421.

HPLC (Chiralcel AS-H): *n*-Hexane/EtOH = 90/10, flow rate 1.0 mL/min,  $T = 40\text{ }^\circ\text{C}$ ,  $\lambda = 235\text{ nm}$ ,  $t_{\text{R}} = 5.769\text{ min}$  (major),  $t_{\text{R}} = 5.217\text{ min}$  (minor); 98% ee.

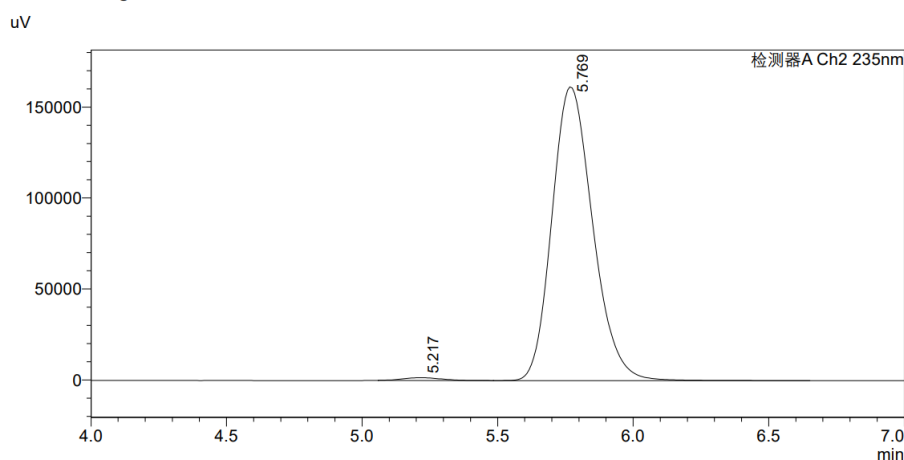
<Chromatogram>



<Peak Table>

检测器A Ch2 235nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	5.219	1368876	147724	50.205		M
2	5.769	1357700	137288	49.795		M
总计		2726576	285012			

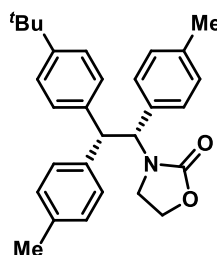
<Chromatogram>



<Peak Table>

检测器A Ch2 235nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	5.217	14611	1522	0.857		M
2	5.769	1690967	161454	99.143		M
总计		1705578	162975			

### 3-((1*R*,2*R*)-2-(4-(*tert*-butyl)phenyl)-1,2-di-*p*-tolylethyl)oxazolidin-2-one (3k)



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/DCM = 10/1/1~8/1/1) afforded **3k** as a white solid (61.6 mg, 72%).  $R_f = 0.34$  (PE/Et<sub>2</sub>O/DCM = 8/1/1);

$[\alpha]_{\text{D}}^{23.3} = -147.30$  ( $c = 0.15$ ,  $\text{CH}_2\text{Cl}_2$ );

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ): 7.39 (d,  $J = 8.4\text{ Hz}$ , 2H), 7.32 – 7.28 (m, 4H), 7.16 (d,  $J = 7.6\text{ Hz}$ , 2H), 7.05 (d,  $J = 7.6\text{ Hz}$ , 2H), 6.93 (d,  $J = 7.6\text{ Hz}$ , 2H), 5.88 (d,  $J = 12.4\text{ Hz}$ , 1H), 4.60 (d,  $J = 12.8\text{ Hz}$ , 1H), 4.00 (td,  $J = 8.8$ ,



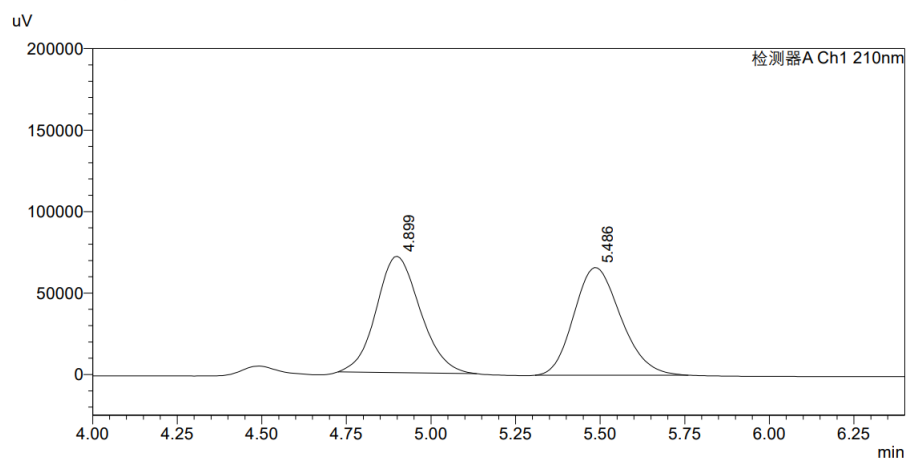
5.2 Hz, 1H), 3.79 (dt,  $J = 8.8, 8.8$  Hz, 1H), 3.39 (td,  $J = 8.4, 4.8$  Hz, 1H), 3.27 (dt,  $J = 8.8, 8.4$  Hz, 1H), 2.26 (s, 3H), 2.16 (s, 3H), 1.27 (s, 9H);

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  158.0, 149.6, 138.8, 138.7, 137.6, 135.9, 134.2, 129.4, 129.4, 128.3, 127.9, 127.1, 125.8, 61.9, 59.0, 51.8, 40.3, 34.5, 31.4, 21.2, 21.0.

HRMS (ESI):  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{29}\text{H}_{33}\text{NO}_2^+$ : 428.2584; found: 428.2584.

HPLC (Chiralcel AS-H): *n*-Hexane/EtOH = 90/10, flow rate 1.0 mL/min,  $T = 40$  °C,  $\lambda = 210$  nm,  $t_R = 5.473$  min (major),  $t_R = 4.853$  min (minor); 95% ee.

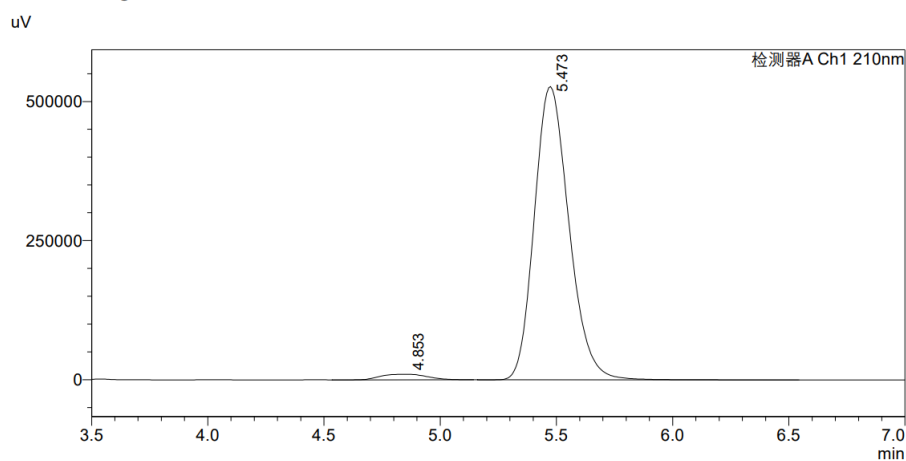
<Chromatogram>



<Peak Table>

检测器A Ch1 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	4.899	645883	71403	50.297		M
2	5.486	638264	66097	49.703		M
总计		1284147	137500			

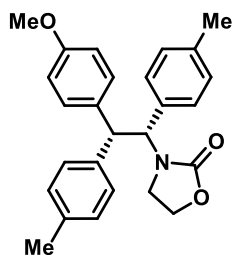
<Chromatogram>



<Peak Table>

检测器A Ch1 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	4.853	138328	10060	2.466		M
2	5.473	5471669	527193	97.534		M
总计		5609996	537253			

**3-((1*R*,2*R*)-2-(4-methoxyphenyl)-1,2-di-*p*-tolylethyl)oxazolidin-2-one (3I)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/CHCl<sub>3</sub> = 4/1/0.5~PE/acetone/CHCl<sub>3</sub> = 8/1/0.5) afforded **3I** as a white solid (54.6 mg, 68%). **R<sub>f</sub>** = 0.21 (PE/Et<sub>2</sub>O/CHCl<sub>3</sub> = 2/1/0.5);

**[α]<sub>D</sub><sup>23.3</sup>** = - 104.44 (c = 0.15, CH<sub>2</sub>Cl<sub>2</sub>);

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): 7.37 (d, *J* = 8.8 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 7.6 Hz, 2H), 6.92 (t, *J* = 7.6 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 5.82 (d, *J* = 12.8 Hz, 1H), 4.57 (d, *J* = 12.4 Hz, 1H), 4.01 (td, *J* = 8.8, 5.2 Hz, 1H), 3.85 (dt, *J* = 8.4, 8.8 Hz, 1H), 3.75 (s, 3H), 3.38 (td, *J* = 8.4, 5.2 Hz, 1H), 3.26 (dt, *J* = 8.4, 8.4 Hz, 1H), 2.25 (s, 3H), 2.15 (s, 3H);

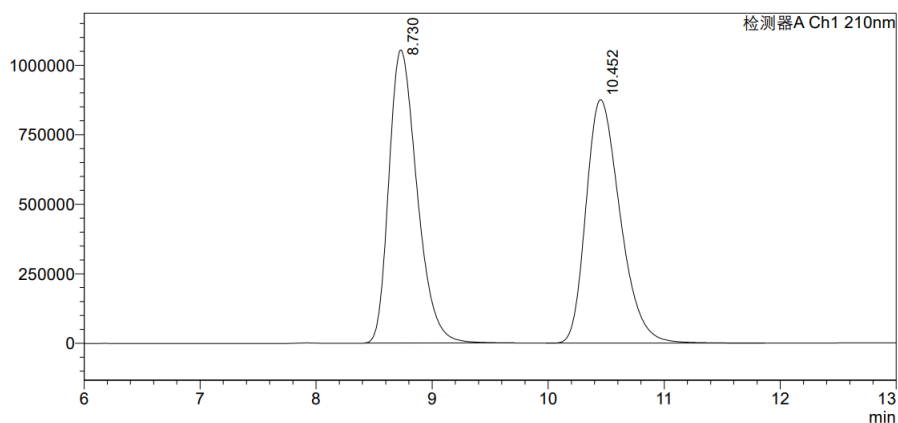
**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 158.4, 158.0, 138.7, 137.6, 135.9, 134.2, 129.4, 129.4, 128.6, 128.3, 127.8, 114.3, 61.9, 59.1, 55.3, 51.3, 40.3, 21.2, 21.0.

**HRMS (ESI)**: [M+H]<sup>+</sup> Calcd for C<sub>26</sub>H<sub>28</sub>NO<sub>3</sub><sup>+</sup>: 402.2064; found: 402.2068.

**HPLC** (Chiralcel AS-H): *n*-Hexane/EtOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 210 nm, t<sub>R</sub> = 10.397 min (major), t<sub>R</sub> = 8.689 min (minor); 98% ee.

<Chromatogram>

uV



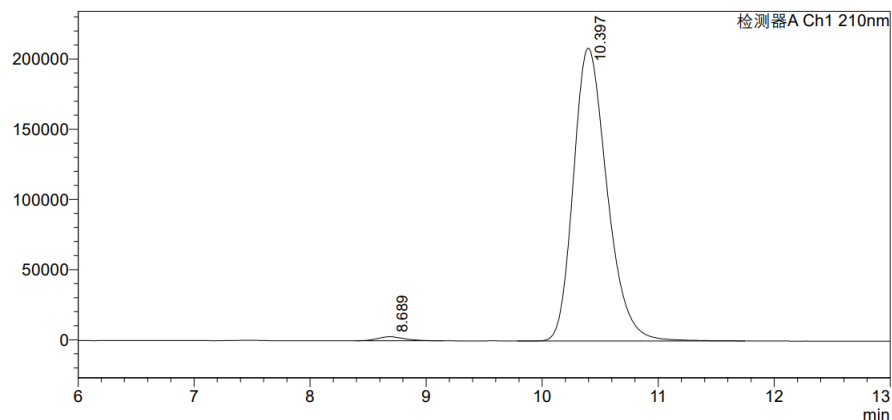
<Peak Table>

检测器A Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	8.730	17768386	1053524	49.792		M
2	10.452	17916830	875255	50.208		M
总计		35685216	1928778			

<Chromatogram>

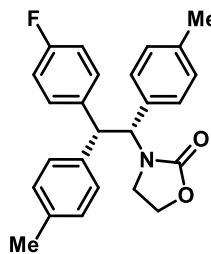
uV



<Peak Table>

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	8.689	46536	2864	1.095		M
2	10.397	4202519	208618	98.905		M
总计		4249055	211481			

3-((1*R*,2*R*)-2-(4-fluorophenyl)-1,2-di-*p*-tolylethyl)oxazolidin-2-one (**3m**)



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/CHCl<sub>3</sub> = 4/1/0.5~PE/acetone/CHCl<sub>3</sub> = 8/1/0.5) afforded **3m** as a white solid (41.0 mg, 53%). **Rf** = 0.31 (PE/Et<sub>2</sub>O/CHCl<sub>3</sub> = 2/1/0.5);

**[α]<sub>D</sub><sup>23.3</sup>** = - 146.67 (c = 0.08, CH<sub>2</sub>Cl<sub>2</sub>);

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): 7.43 (td, *J* = 6.8, 2.0 Hz, 2H), 7.27 (d, *J* = 7.6 Hz, 2H), 7.12 (d, *J* = 7.6 Hz, 2H), 7.06 (d, *J* = 7.6 Hz, 2H), 7.00 (t, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 7.6 Hz, 2H), 5.84 (d, *J* = 12.4 Hz, 1H), 4.64 (d, *J* = 12.4 Hz, 1H), 4.04 (td, *J* = 8.8, 5.2 Hz, 1H), 3.87 (dt, *J* = 8.8, 8.4 Hz, 1H), 3.36 (td, *J* = 8.4, 5.2 Hz, 1H), 3.23 (dt, *J* = 8.8, 8.4 Hz, 1H), 2.26 (s, 3H), 2.17 (s, 3H);

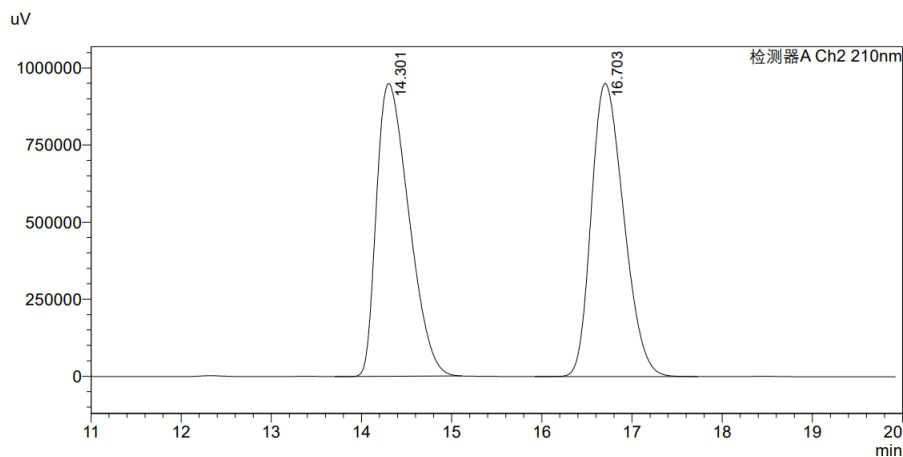
**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 161.8 (d, *J*<sub>C-F</sub> = 243.9 Hz), 157.9, 138.2, 137.9 (d, *J*<sub>C-F</sub> = 3.2 Hz), 137.8, 136.3, 133.8, 129.5, 129.5, 129.1 (d, *J*<sub>C-F</sub> = 7.9 Hz), 128.3, 127.8, 115.8 (d, *J*<sub>C-F</sub> = 21.2 Hz), 61.9, 59.1, 51.3, 40.3, 21.2, 21.0.

**<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>): δ -115.9;

**HRMS (ESI)**: [M+H]<sup>+</sup> Calcd for C<sub>25</sub>H<sub>25</sub>FNO<sub>2</sub><sup>+</sup>: 390.1864; found: 390.1866.

**HPLC** (Chiralcel AD-H): *n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 210 nm, *t<sub>R</sub>* = 14.419 min (major), *t<sub>R</sub>* = 16.807 min (minor); 98% ee.

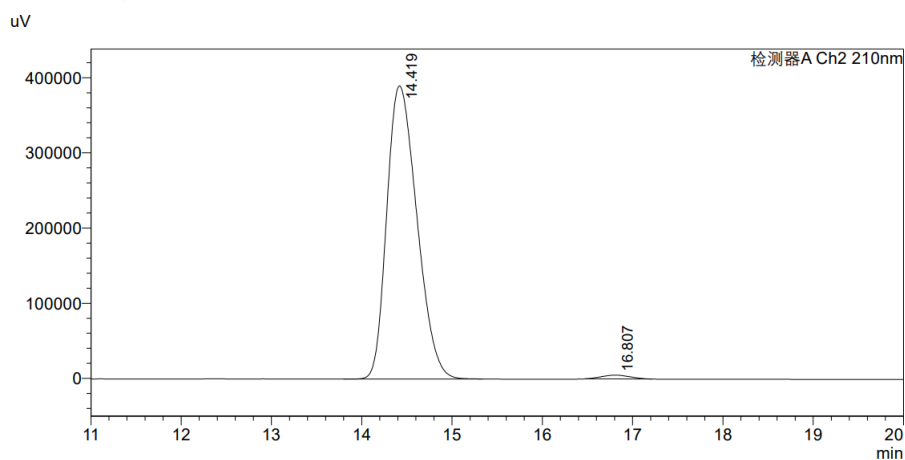
<Chromatogram>



<Peak Table>

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	14.301	23738063	949745	49.928		M
2	16.703	23806062	950682	50.072		M
总计		47544125	1900427			

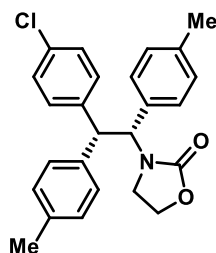
<Chromatogram>



<Peak Table>

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	14.419	9043554	390000	98.808		M
2	16.807	109093	4994	1.192		M
总计		9152647	394994			

**3-((1*R*,2*R*)-2-(4-chlorophenyl)-1,2-di-*p*-tolylethyl)oxazolidin-2-one (3n)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/CHCl<sub>3</sub>= 4/1/0.5~2/1/0.5) afforded **3n** as a white solid (36.7 mg, 45%). *R<sub>f</sub>* = 0.33 (PE/acetone/DCM = 8/1/1);

$[\alpha]_D^{23.3} = -129.74$  (*c* = 0.13, CH<sub>2</sub>Cl<sub>2</sub>);

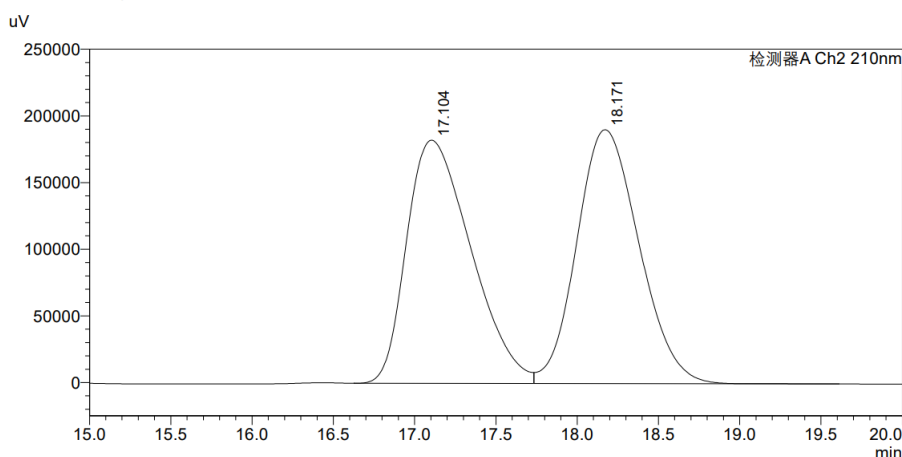
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 7.40 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 6.8 Hz, 2H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.06 (d, *J* = 7.6 Hz, 2H), 6.93 (d, *J* = 7.6 Hz, 2H), 5.83 (d, *J* = 12.4 Hz, 1H), 4.62 (d, *J* = 12.4 Hz, 1H), 4.05 (td, *J* = 8.8, 5.2 Hz, 1H), 3.89 (dt, *J* = 8.8, 8.8 Hz, 1H), 3.35 (td, *J* = 8.4, 5.2 Hz, 1H), 3.26 (dt, *J* = 9.2, 8.4 Hz, 1H), 2.26 (s, 3H), 2.17 (s, 3H);

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 157.9, 140.7, 137.9, 137.9, 136.4, 133.7, 132.8, 129.6, 129.5, 129.1, 129.0, 128.3, 127.8, 61.9, 58.9, 51.5, 40.3, 21.2, 21.0.

**HRMS (ESI):** [M-Cl+Na]<sup>+</sup> Calcd for C<sub>25</sub>H<sub>24</sub>NO<sub>2</sub>Na<sup>+</sup>: 393.1699; found: 393.1693.

**HPLC** (Chiralcel AD-H): *n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 210 nm, *t<sub>R</sub>* = 16.988 min (major), *t<sub>R</sub>* = 18.236 min (minor); 97% ee.

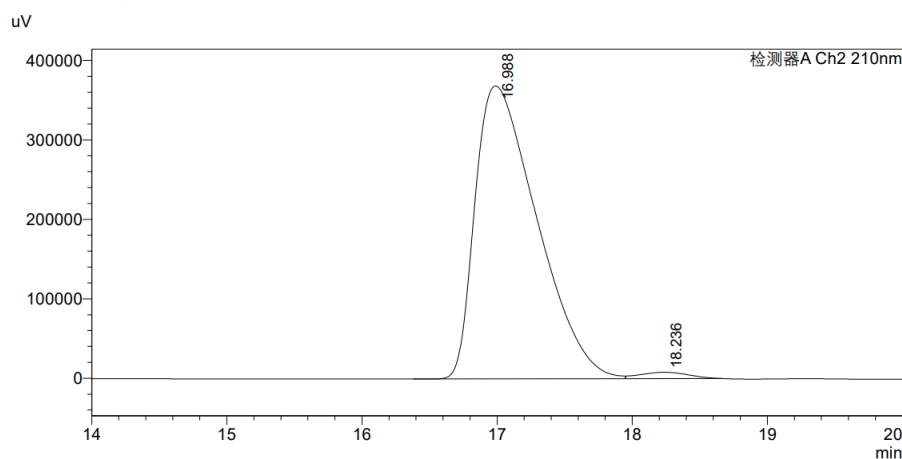
<Chromatogram>



<Peak Table>

检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	17.104	5028192	182388	49.719		M
2	18.171	5084933	190426	50.281		V M
总计		10113125	372813			

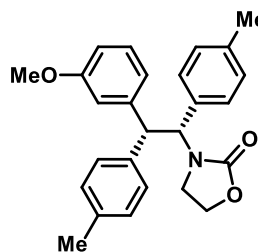
<Chromatogram>



<Peak Table>

检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	16.988	11755167	368819	98.304		M
2	18.236	202752	8098	1.696		V M
总计		11957920	376918			

**3-((1*R*,2*R*)-2-(3-methoxyphenyl)-1,2-di-*p*-tolylethyl)oxazolidin-2-one (3o)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/DCM= 8/1/1~5/1/1) afforded **3o** as a white solid (45.0 mg, 56%). *R<sub>f</sub>* = 0.20 (PE/Et<sub>2</sub>O/DCM= 4/1/1);

$[\alpha]_D^{23.3} = -129.02$  (*c* = 0.17, CH<sub>2</sub>Cl<sub>2</sub>);

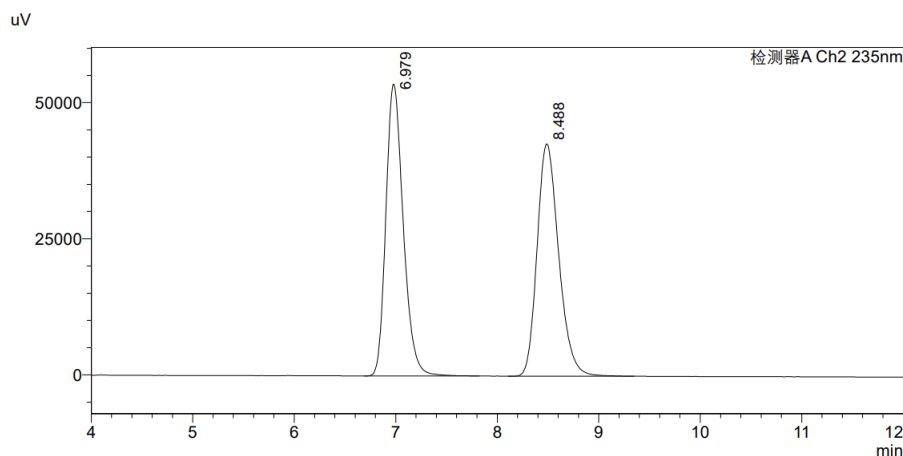
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 7.28 (d, *J* = 8.0 Hz, 2H), 7.22 (t, *J* = 8.0 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.06 (d, *J* = 8.0 Hz, 3H), 7.02 (bs, 1H), 6.93 (d, *J* = 7.6 Hz, 2H), 6.73 (dd, *J* = 8.4, 2.8 Hz, 1H), 5.87 (d, *J* = 12.4 Hz, 1H), 4.61 (d, *J* = 12.8 Hz, 1H), 4.01 (td, *J* = 8.8, 5.2 Hz, 1H), 3.85 (dt, *J* = 8.8, 8.4 Hz, 1H), 3.80 (s, 3H), 3.40 (td, *J* = 8.4, 5.2 Hz, 1H), 3.26 (dt, *J* = 8.8, 8.4 Hz, 1H), 2.26 (s, 3H), 2.16 (s, 3H);

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 159.9, 157.9, 143.5, 138.2, 137.7, 136.1, 134.0, 129.9, 129.4, 129.4, 128.3, 127.9, 120.0, 113.2, 112.5, 61.9, 58.8, 55.3, 52.1, 40.3, 21.2, 21.0.

**HRMS (ESI):** [M+H]<sup>+</sup> Calcd for C<sub>26</sub>H<sub>28</sub>NO<sub>3</sub><sup>+</sup>: 402.2064; found: 402.2061.

**HPLC** (Chiralcel AS-H): *n*-Hexane/EtOH = 90/10, flow rate 1.0 mL/min, T = 40 °C,  $\lambda$  = 235 nm,  $t_R$  = 8.479 min (major),  $t_R$  = 6.971 min (minor); 95% ee.

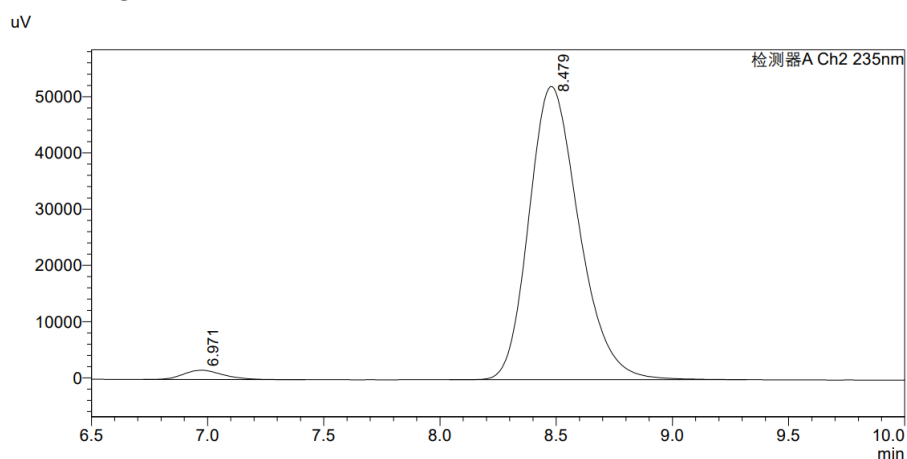
<Chromatogram>



<Peak Table>

检测器A Ch2 235nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	6.979	638119	53598	50.233		
2	8.488	632210	42663	49.767		
总计		1270329	96261			

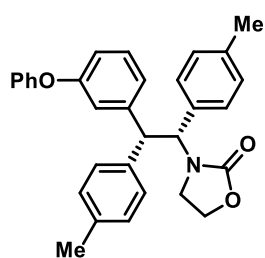
<Chromatogram>



<Peak Table>

检测器A Ch2 235nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	6.971	19560	1654	2.440		M
2	8.479	782095	52102	97.560		M
总计		801655	53756			

### 3-((1*R*,2*R*)-2-(3-phenoxyphenyl)-1,2-di-*p*-tolylethyl)oxazolidin-2-one (**3p**)



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/acetone/CHCl<sub>3</sub> = 8/1/1) afforded **3p** as a white solid (74.8 mg, 81%). **R<sub>f</sub>** = 0.30 (PE/acetone/CHCl<sub>3</sub> = 8/1/1);

$[\alpha]_D^{23.3}$  = - 125.42 (c = 0.16, CH<sub>2</sub>Cl<sub>2</sub>);

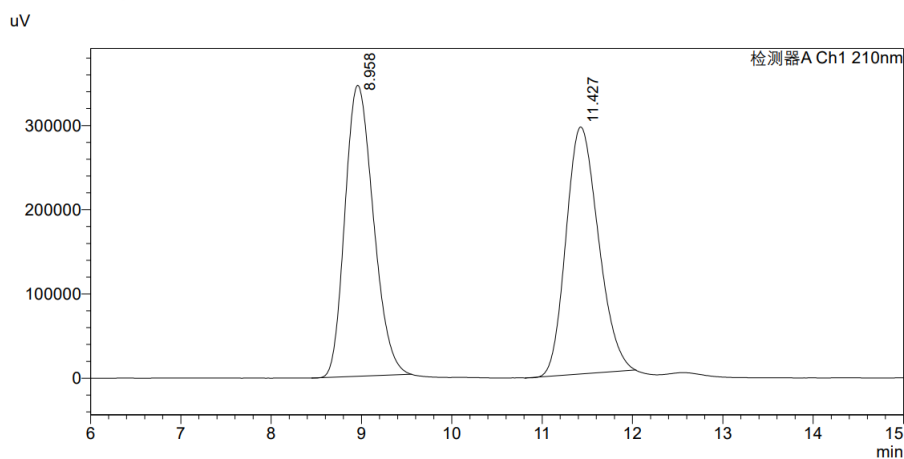
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 7.35 (t, *J* = 7.6 Hz, 2H), 7.32 – 7.26 (m, 4H), 7.16 – 7.14 (m, 3H), 7.11 (d, *J* = 7.2 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 2H), 6.98 (dd, *J* = 10.0, 8.8, 1.2 Hz, 4H), 6.84 (dt, *J* = 7.6, 2.0 Hz, 1H), 5.85 (d, *J* = 12.4 Hz, 1H), 4.63 (d, *J* = 12.8 Hz, 1H), 4.07 (td, *J* = 8.8, 5.2 Hz, 1H), 3.93 (td, *J* = 8.8, 8.4 Hz, 1H), 3.44 (td, *J* = 8.4, 5.2 Hz, 1H), 3.26 (td, *J* = 8.8, 8.4 Hz, 1H), 2.27 (s, 3H), 2.20 (s, 3H);

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 157.8, 157.4, 157.2, 144.2, 137.9, 137.7, 136.2, 133.9, 130.3, 129.8, 129.4, 129.4, 128.3, 127.9, 123.2, 122.5, 118.8, 118.7, 117.4, 61.8, 59.1, 51.9, 40.4, 21.1, 21.0.

HRMS (ESI): [M+H]<sup>+</sup> Calcd for C<sub>31</sub>H<sub>30</sub>NO<sub>3</sub><sup>+</sup>: 464.2220; found: 464.2218.

HPLC (Chiralcel OD-H): *n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 210 nm, t<sub>R</sub> = 11.416 min (major), t<sub>R</sub> = 8.999 min (minor); >99% ee.

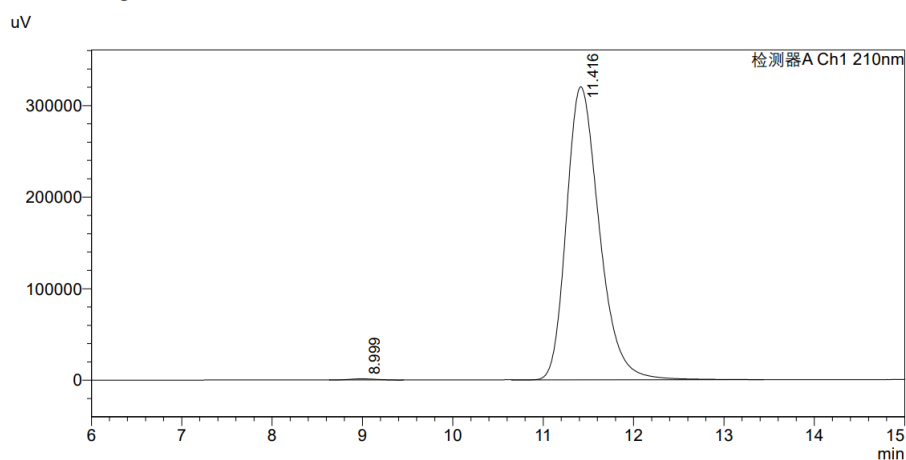
<Chromatogram>



<Peak Table>

检测器A Ch1 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	8.958	7562447	345610	50.806		M
2	11.427	7322615	293312	49.194		M
总计		14885062	638922			

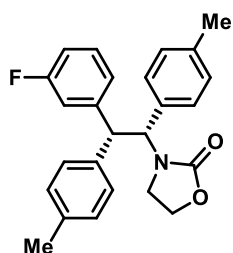
<Chromatogram>



<Peak Table>

检测器A Ch1 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	8.999	24384	1297	0.300		M
2	11.416	8108731	320022	99.700		M
总计		8133115	321318			

### 3-((1*R*,2*R*)-2-(3-fluorophenyl)-1,2-di-*p*-tolylethyl)oxazolidin-2-one (**3q**)



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/acetone/DCM = 10/1/1) afforded **3q** as a white solid (44.1 mg, 57%). R<sub>f</sub> = 0.29 (PE/acetone/DCM = 8/1/1);

[α]<sub>D</sub><sup>23.3</sup> = -166.98 (c = 0.21, CH<sub>2</sub>Cl<sub>2</sub>);

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): 7.27 – 7.24 (m, 4H), 7.15 – 7.11 (m, 3H), 7.04 (d, *J* = 8.0 Hz, 2H), 6.93 (d, *J* = 7.6 Hz, 2H), 6.89 – 6.84 (m, 1H), 5.82 (d, *J* = 12.8 Hz, 1H), 4.64 (d, *J* = 12.8 Hz, 1H), 4.01 (td, *J* = 8.8, 5.2 Hz, 1H), 3.87 (dt, *J* = 8.8, 8.4 Hz, 1H), 3.38 (td, *J* = 8.4, 5.2 Hz, 1H), 3.24 (dt, *J* = 8.4, 8.4 Hz, 1H), 2.24 (s, 3H), 2.15 (s, 3H);

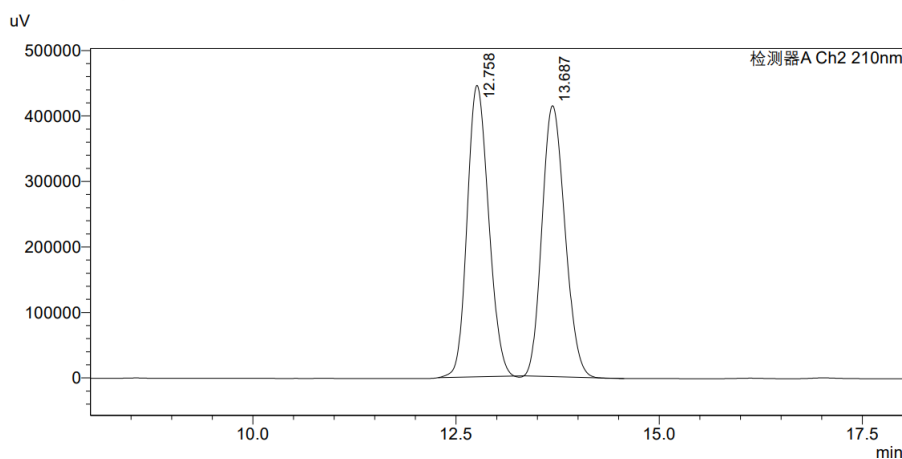
**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 163.0 (d, *J*<sub>C-F</sub> = 244.8 Hz), 157.9, 144.7 (d, *J*<sub>C-F</sub> = 6.7 Hz), 137.8, 137.7, 136.4, 133.7, 130.5 (d, *J*<sub>C-F</sub> = 8.3 Hz), 129.5, 129.4, 128.3, 127.9, 123.2 (d, *J*<sub>C-F</sub> = 2.8 Hz), 114.8 (d, *J*<sub>C-F</sub> = 21.4 Hz), 114.0 (d, *J*<sub>C-F</sub> = 20.9 Hz), 61.9, 59.0, 51.9, 40.4, 21.2, 21.0.

**<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>): δ -112.4;

**HRMS (ESI):** [M+H]<sup>+</sup> Calcd for C<sub>25</sub>H<sub>25</sub>FNO<sub>2</sub><sup>+</sup>: 390.1864; found: 390.1858.

**HPLC** (Chiralcel AD-H): *n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 210 nm, t<sub>R</sub> = 13.695 min (major), t<sub>R</sub> = 12.796 min (minor); 99% ee.

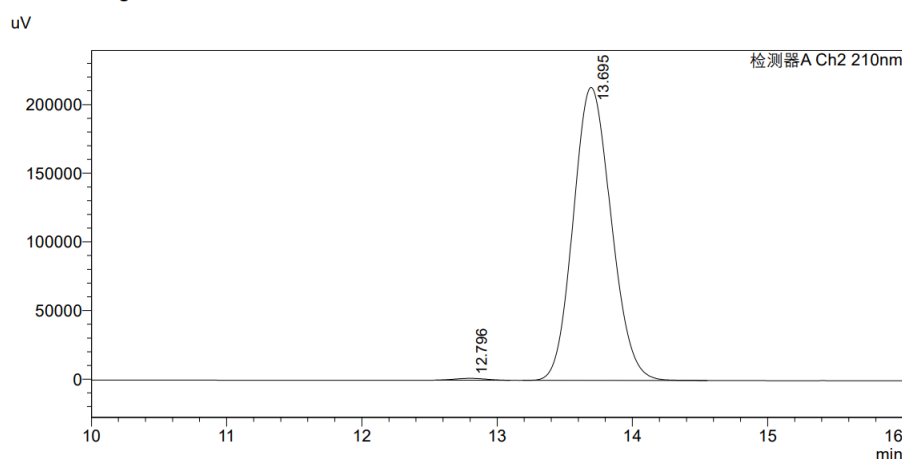
<Chromatogram>



<Peak Table>

检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	12.758	8173066	445083	50.130		M
2	13.687	8130622	413770	49.870		M
总计		16303688	858853			

<Chromatogram>

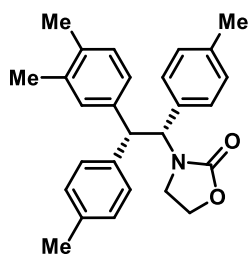


<Peak Table>

检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	12.796	22113	1373	0.528		M
2	13.695	4165842	213457	99.472		M
总计		4187955	214831			

**3-((1*R*,2*R*)-2-(3,4-dimethylphenyl)-1,2-di-*p*-tolylethyl)oxazolidin-2-one (3r)**





General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/acetone/ $\text{CHCl}_3 = 10/1/1$ ) afforded **3r** as a white solid (42.2 mg, 53%).  $R_f = 0.21$  (PE/acetone/ $\text{CHCl}_3 = 8/1/1$ );

$[\alpha]_D^{23.3} = -111.67$  ( $c = 0.16$ ,  $\text{CH}_2\text{Cl}_2$ );

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ): 7.27 (d,  $J = 9.2$  Hz, 2H), 7.19 (bs, 2H), 7.13 (d,  $J = 8.0$  Hz, 2H), 7.05 (d,  $J = 8.0$  Hz, 3H), 6.92 (d,  $J = 8.0$  Hz, 2H), 5.85 (d,  $J = 12.8$  Hz, 1H), 4.55 (d,  $J = 12.8$  Hz, 1H), 4.01 (td,  $J = 8.8, 5.2$  Hz, 1H), 3.86 (dt,  $J = 8.4, 8.8$  Hz, 1H), 3.41 (td,  $J = 8.4, 5.2$  Hz, 1H), 3.27 (dt,  $J = 8.8, 8.4$  Hz, 1H), 2.26 (s, 3H), 2.22 (s, 3H), 2.18 (s, 3H), 2.15 (s, 3H);

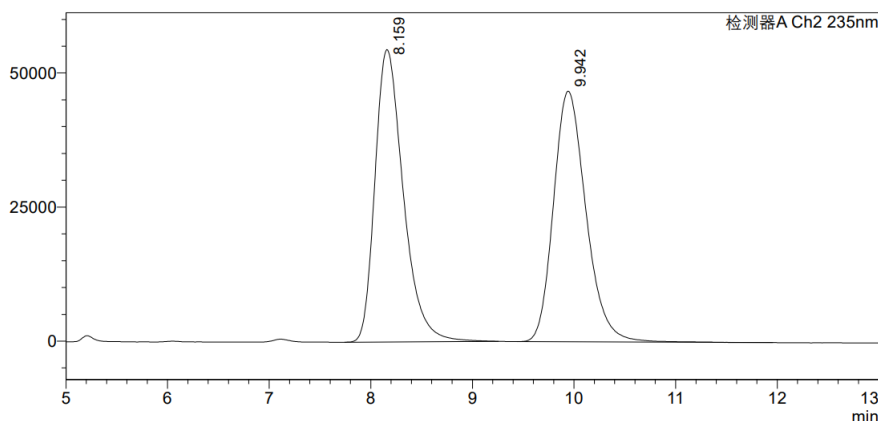
$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  157.9, 139.4, 138.8, 137.5, 137.0, 135.9, 135.1, 134.3, 130.1, 129.4, 129.3, 128.9, 128.3, 127.8, 124.8, 61.8, 58.9, 51.7, 40.2, 21.2, 21.0, 20.1, 19.5.

**HRMS (ESI)**:  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{27}\text{H}_{30}\text{NO}_2^+$ : 400.2271; found: 400.2270.

**HPLC** (Chiralcel AS-H): *n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min,  $T = 40^\circ\text{C}$ ,  $\lambda = 235$  nm,  $t_R = 9.942$  min (major),  $t_R = 8.160$  min (minor); 96% ee.

<Chromatogram>

uv



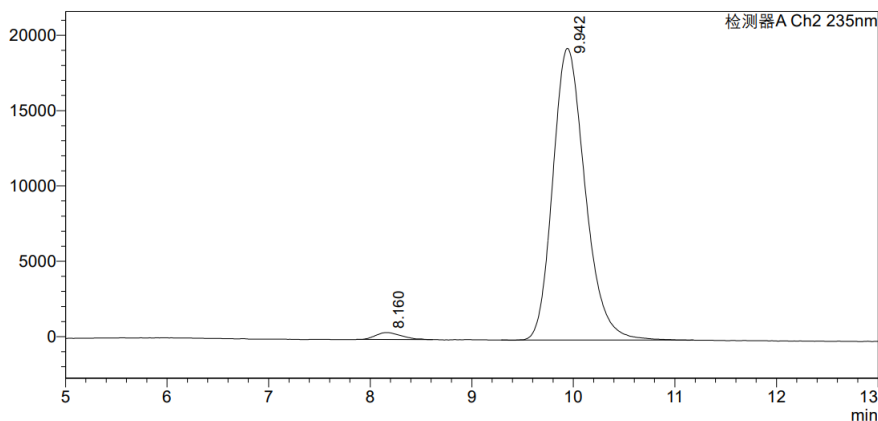
<Peak Table>

检测器A Ch2 235nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	8.159	1057051	54530	50.414		M
2	9.942	1039683	46748	49.586		M
总计		2096733	101278			

<Chromatogram>

uv

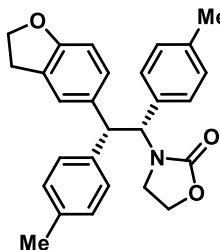


<Peak Table>

检测器A Ch2 235nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	8.160	8067	460	1.846		M
2	9.942	428963	19360	98.154		M
总计		437029	19820			

3-((1*R*,2*R*)-2-(2,3-dihydrobenzofuran-5-yl)-1,2-di-*p*-tolylethyl)oxazolidin-2-one (**3s**)



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/acetone/CHCl<sub>3</sub> = 10/1/1–8/1/1) afforded **3s** as a white solid (50.5 mg, 61%). **R<sub>f</sub>** = 0.19 (PE/acetone/CHCl<sub>3</sub> = 8/1/1);

**[α]<sub>D</sub><sup>23.3</sup>** = - 111.76 (c = 0.17, CH<sub>2</sub>Cl<sub>2</sub>);

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): 7.30 (bs, 1H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 6.8 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 7.6 Hz, 2H), 6.93 (d, *J* = 8.0 Hz, 2H), 6.69 (d, *J* = 8.0 Hz, 1H), 5.82 (d, *J* = 12.4 Hz, 1H),

4.58 – 4.47 (m, 3H), 4.04 (td, *J* = 8.8, 5.2 Hz, 1H), 3.89 (dt, *J* = 8.8, 8.4 Hz, 1H), 3.41 (td, *J* = 8.4, 5.6 Hz, 1H), 3.26 (dt, *J* = 9.2, 8.4 Hz, 1H), 3.20 – 3.09 (m, 2H), 2.26 (s, 3H), 2.16 (s, 3H);

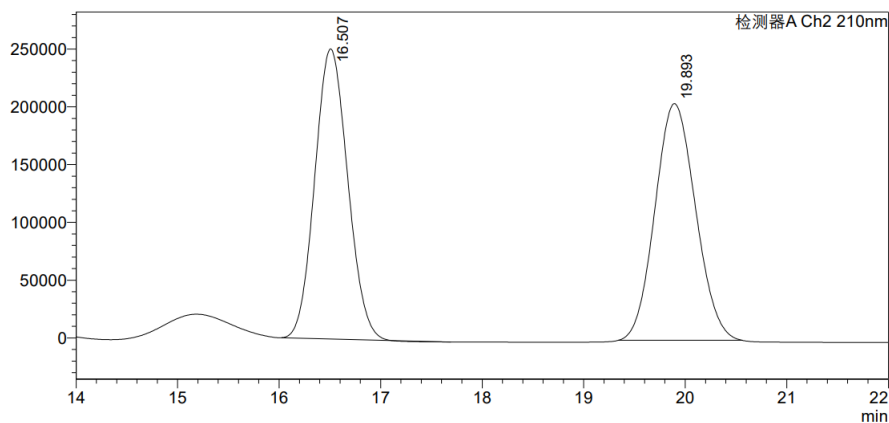
**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 159.1, 158.0, 138.9, 137.6, 135.9, 134.2, 134.1, 129.4, 129.4, 128.3, 127.9, 127.7, 127.3, 123.9, 109.1, 71.4, 61.9, 59.0, 51.4, 40.2, 29.9, 21.2, 21.0.

**HRMS (ESI)**: [M+H]<sup>+</sup> Calcd for C<sub>27</sub>H<sub>28</sub>NO<sub>3</sub><sup>+</sup>: 414.2064; found: 414.2055.

**HPLC** (Chiralcel AD-H): *n*-Hexane/EtOH = 90/10, flow rate 1.0 mL/min, T = 40 °C, λ = 210 nm, t<sub>R</sub> = 19.891 min (major), t<sub>R</sub> = 16.525 min (minor); 97% ee.

<Chromatogram>

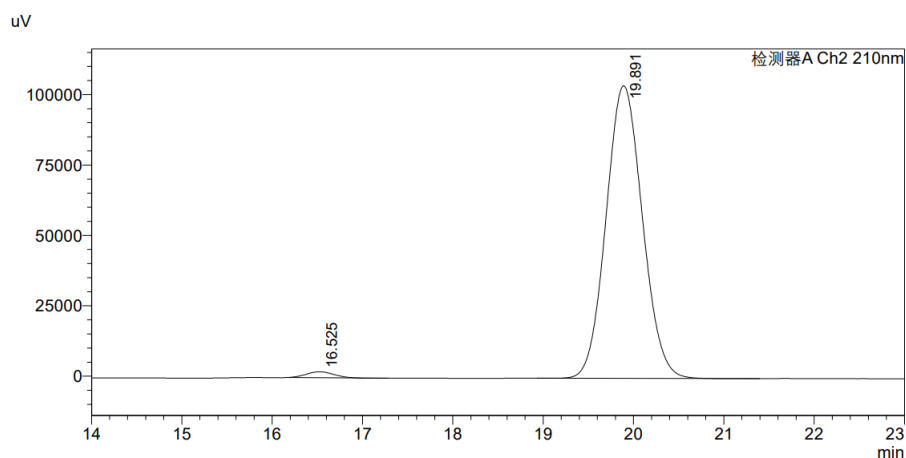
uv



<Peak Table>

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	16.507	5650982	251060	49.813		M
2	19.893	5693333	204838	50.187		M
总计		11344315	455898			

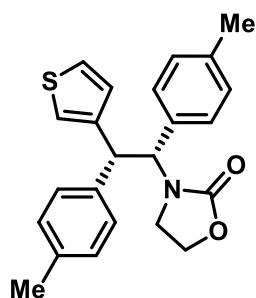
<Chromatogram>



<Peak Table>

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	16.525	44758	2104	1.517		M
2	19.891	2906531	103952	98.483		M
总计		2951289	106056			

3-((1R,2R)-2-(thiophen-3-yl)-1,2-di-p-tolyethyl)oxazolidin-2-one (3t)



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/CHCl<sub>2</sub> = 6/1/1) afforded **3t** as a white solid (55.1 mg, 73%). R<sub>f</sub> = 0.44 (PE/Et<sub>2</sub>O/CHCl<sub>2</sub> = 3/1/1);

[ $\alpha$ ]<sub>D</sub><sup>29.1</sup> = - 81.82 (c = 0.25, CHCl<sub>2</sub>);

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 7.29 – 7.24 (m, 4H), 7.17 – 7.16 (m, 1H), 7.3 (d, J = 8.0 Hz, 2H), 7.05 (d, J = 7.6 Hz, 2H), 6.94 (d, J = 8.0 Hz, 2H), 5.77 (d, J = 12.4 Hz, 1H), 4.80 (d, J = 12.4 Hz, 1H), 4.05 (td, J = 8.4, 5.2 Hz, 1H), 3.92 (dt, J = 8.8, 8.4 Hz, 1H), 3.44 (td, J = 8.4, 5.2 Hz, 1H), 3.30 (dt, J = 9.2, 8.0 Hz, 1H), 2.26

(s, 3H), 2.17 (s, 3H);

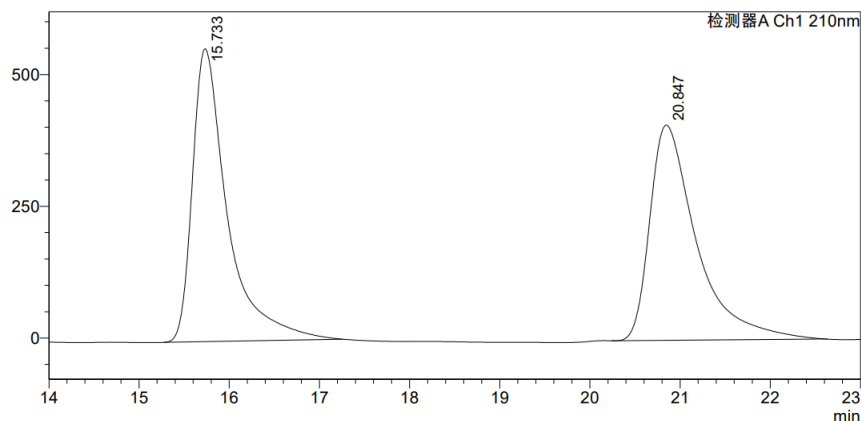
<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  157.9, 142.6, 137.9, 137.6, 136.1, 133.9, 129.4, 129.3, 128.2, 128.0, 127.1, 126.3, 120.6, 61.9, 59.7, 47.4, 40.3, 21.2, 21.0.

**HRMS (ESI):** [M+H]<sup>+</sup> Calcd for C<sub>23</sub>H<sub>23</sub>NO<sub>2</sub>SNa<sup>+</sup>: 400.1342; found: 400.1344.

**HPLC** (Chiralcel AD-H): n-Hexane/EtOH = 90/10, flow rate 1.0 mL/min, T = 40 °C,  $\lambda$  = 210 nm, t<sub>R</sub> = 15.673 min (major), t<sub>R</sub> = 20.746 min (minor); 82% ee.

<Chromatogram>

mV

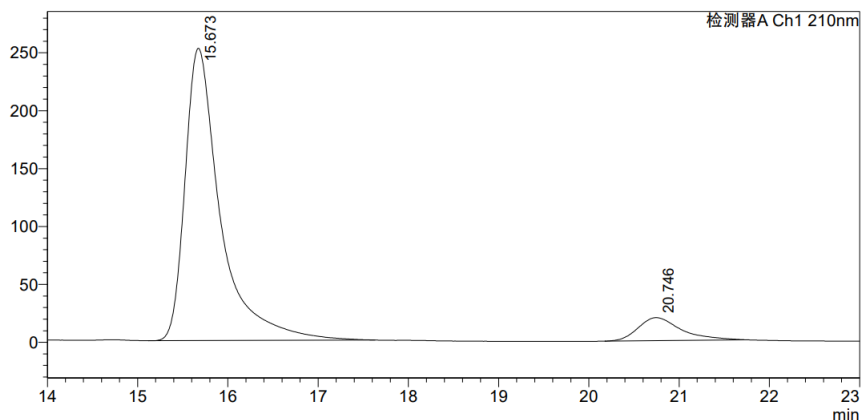


<Peak Table>

检测器A Ch1 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	15.733	14981472	555724	50.459		M
2	20.847	14708732	408239	49.541		M
总计		29690204	963963			

<Chromatogram>

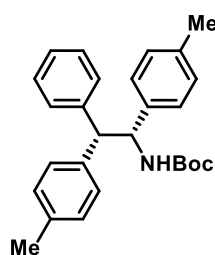
mV



<Peak Table>

检测器A Ch1 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	15.673	6903259	252510	91.240		M
2	20.746	662823	19940	8.760		M
总计		7566082	272450			

**tert-butyl ((1*R*,2*S*)-2-phenyl-1,2-di-*p*-tolylethyl)carbamate (**3u**)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel (PE/Et<sub>2</sub>O/CHCl<sub>3</sub>= 20/1/0.5~15/1/0.5) afforded **3u** as a white solid (29.4 mg, 37%). *R<sub>f</sub>* = 0.39 (PE/Et<sub>2</sub>O/CHCl<sub>3</sub>= 10/1/0.5);

$[\alpha]_D^{23.3} = +37.58$  (*c* = 0.11, CHCl<sub>2</sub>);

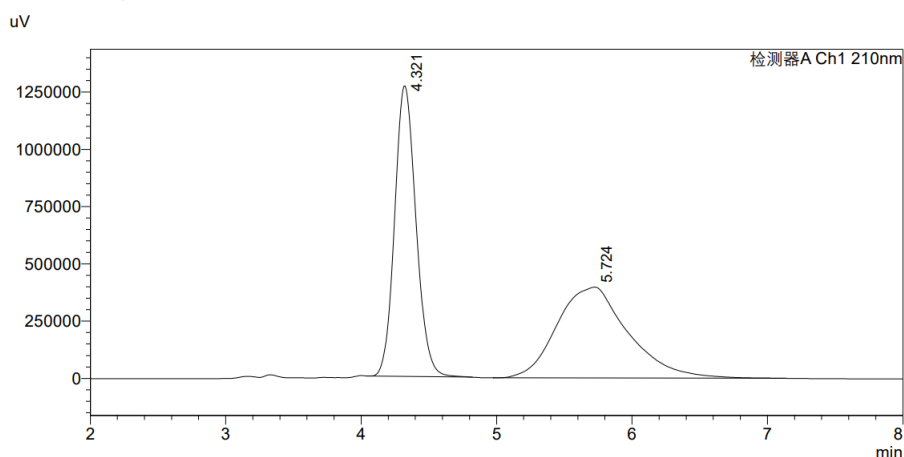
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 7.28 – 7.24 (m, 4H), 7.19 – 7.15 (m, 1H), 7.00 – 6.98 (m, 6H), 6.93 (d, *J* = 8.0 Hz, 2H), 5.41 (bs, 1H), 4.78 (bs, 1H), 4.17 (d, *J* = 9.6 Hz, 1H), 2.25 (s, 3H), 2.20 (s, 3H), 1.28 (s, 9H);

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 155.1, 138.9, 136.5, 135.9, 129.1, 128.9, 128.9, 128.6, 128.4, 127.1, 126.8, 77.2, 57.8, 28.4, 21.2, 21.1.

HRMS (ESI): [M+H]<sup>+</sup> Calcd for C<sub>27</sub>H<sub>32</sub>NO<sub>2</sub>: 402.2428; found: 402.2425.

**HPLC** (Chiralcel AS-H): *n*-Hexane/EtOH = 90/10, flow rate 1.0 mL/min,  $\lambda$  = 210 nm,  $t_R$  = 4.325 min (major),  $t_R$  = 5.706 min (minor); 75% ee.

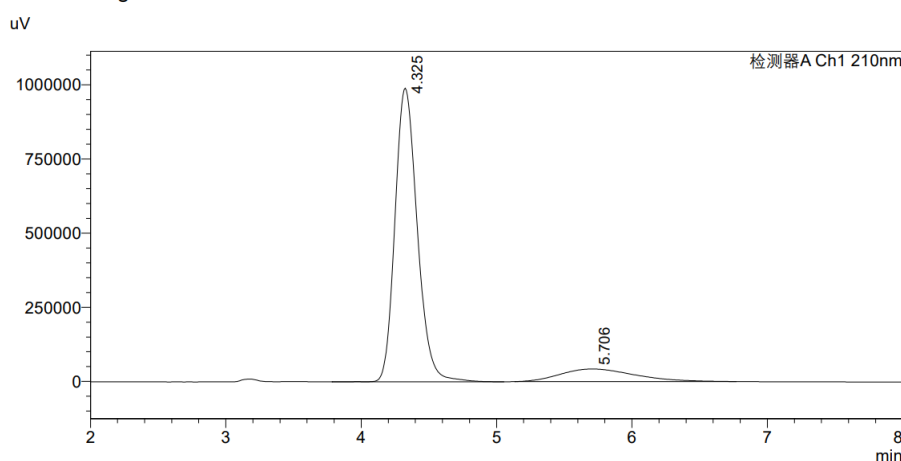
<Chromatogram>



<Peak Table>

检测器A Ch1 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	4.321	13957639	1267410	49.034		M
2	5.724	14507306	396755	50.966		M
总计		28464945	1664165			

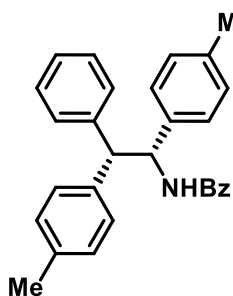
<Chromatogram>



<Peak Table>

检测器A Ch1 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	4.325	11373323	990187	87.504		M
2	5.706	1624182	42539	12.496		M
总计		12997505	1032727			

**N-((1*R*,2*S*)-2-phenyl-1,2-di-*p*-tolylethyl)benzamide (3v)**



General procedure was followed on 0.2 mmol scale and purification by flash column chromatography on silica gel afforded **3v** as a white solid (13.8 mg, 17%).

$[\alpha]_D^{29.1} = +46.39$  ( $c = 0.24$ ,  $\text{CHCl}_2$ );

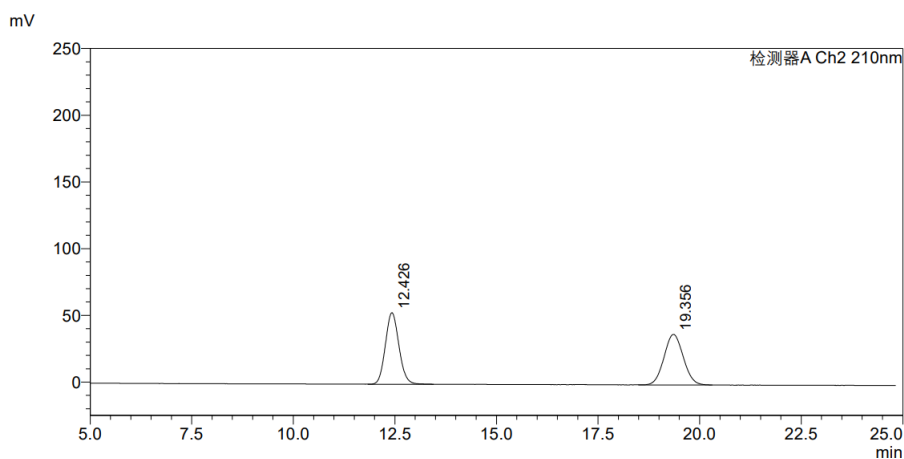
$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ): 7.41 (d,  $J = 7.6$  Hz, 3H), 7.36 (d,  $J = 7.2$  Hz, 1H), 7.28 (d,  $J = 7.2$  Hz, 2H), 7.26 – 7.14 (m, 4H), 7.06 (d,  $J = 8.0$  Hz, 4H), 6.99 – 6.94 (m, 4H), 6.33 (d,  $J = 8.4$  Hz, 1H), 5.94 (t,  $J = 8.8$  Hz, 1H), 4.38 (d,  $J = 9.6$  Hz, 1H), 2.22 (s, 3H), 2.20 (s, 3H);

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 166.7, 141.4, 138.6, 138.4, 136.8, 136.2, 134.9, 131.4, 129.3, 129.1, 128.8, 128.7, 128.6, 128.3, 127.2, 127.1, 126.9, 77.2, 57.4, 55.9, 21.2, 21.1.

HRMS (ESI): [M+H]<sup>+</sup> Calcd for C<sub>29</sub>H<sub>28</sub>NO<sup>+</sup>: 406.2165; found: 406.2155.

HPLC (Chiralcel AD-H): *n*-Hexane/EtOH = 90/10, flow rate 1.0 mL/min, λ = 210 nm, t<sub>R</sub> = 19.333 min (major), t<sub>R</sub> = 12.380 min (minor); 97% ee.

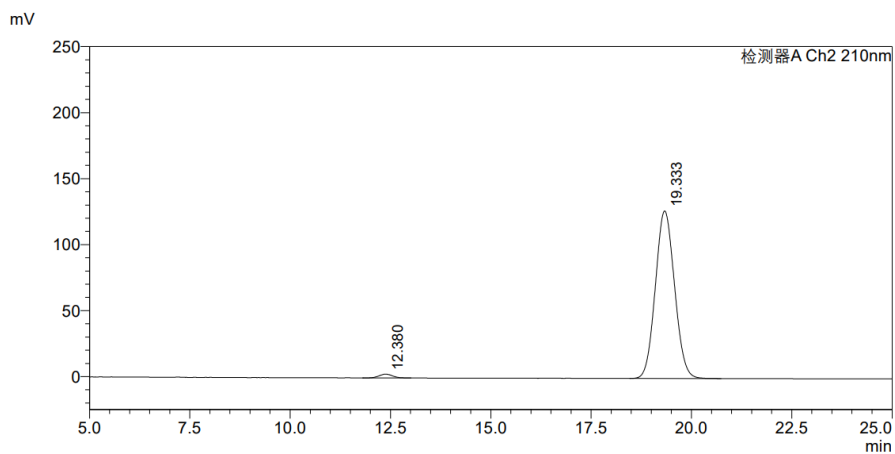
<Chromatogram>



<Peak Table>

检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	12.426	1248030	53616	49.978		V
2	19.356	1249124	37915	50.022		V
总计		2497154	91531			

<Chromatogram>



<Peak Table>

检测器A Ch2 210nm						
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark
1	12.380	69428	2939	1.615		V
2	19.333	4229864	126987	98.385		V
总计		4299292	129926			

## X-Ray Diffraction Analysis of Compound 3b

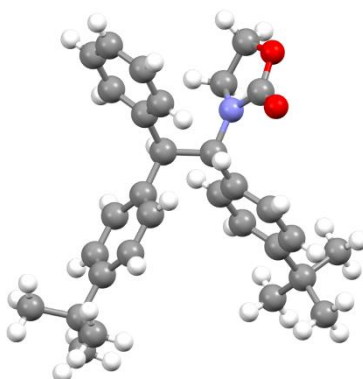


Fig. S1 Crystal data and structure refinement for **3b** (CCDC: 2109912)

Empirical formula	C <sub>31</sub> H <sub>37</sub> N O <sub>2</sub>	
Formula weight	455.61	
Temperature	293(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 6.4789(5) Å	a = 90°.
	b = 18.2766(13) Å	b = 90°.
	c = 22.8772(16) Å	c = 90°.
Volume	2708.9(3) Å <sup>3</sup>	
Z	4	
Density (calculated)	1.117 Mg/m <sup>3</sup>	
Absorption coefficient	0.530 mm <sup>-1</sup>	
F(000)	984	
Crystal size	0.150 x 0.110 x 0.060 mm <sup>3</sup>	
Theta range for data collection	3.095 to 70.239°.	
Index ranges	-7<=h<=7, -22<=k<=19, -27<=l<=22	
Reflections collected	19163	
Independent reflections	5098 [R(int) = 0.0636]	
Completeness to theta = 67.679°	98.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7533 and 0.4811	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	5098 / 42 / 343	
Goodness-of-fit on F <sup>2</sup>	1.040	
Final R indices [I>2sigma(I)]	R1 = 0.0539, wR2 = 0.1366	
R indices (all data)	R1 = 0.0689, wR2 = 0.1504	
Absolute structure parameter	-0.09(19)	

Extinction coefficient

n/a

Largest diff. peak and hole

0.256 and -0.190 e.Å<sup>-3</sup>

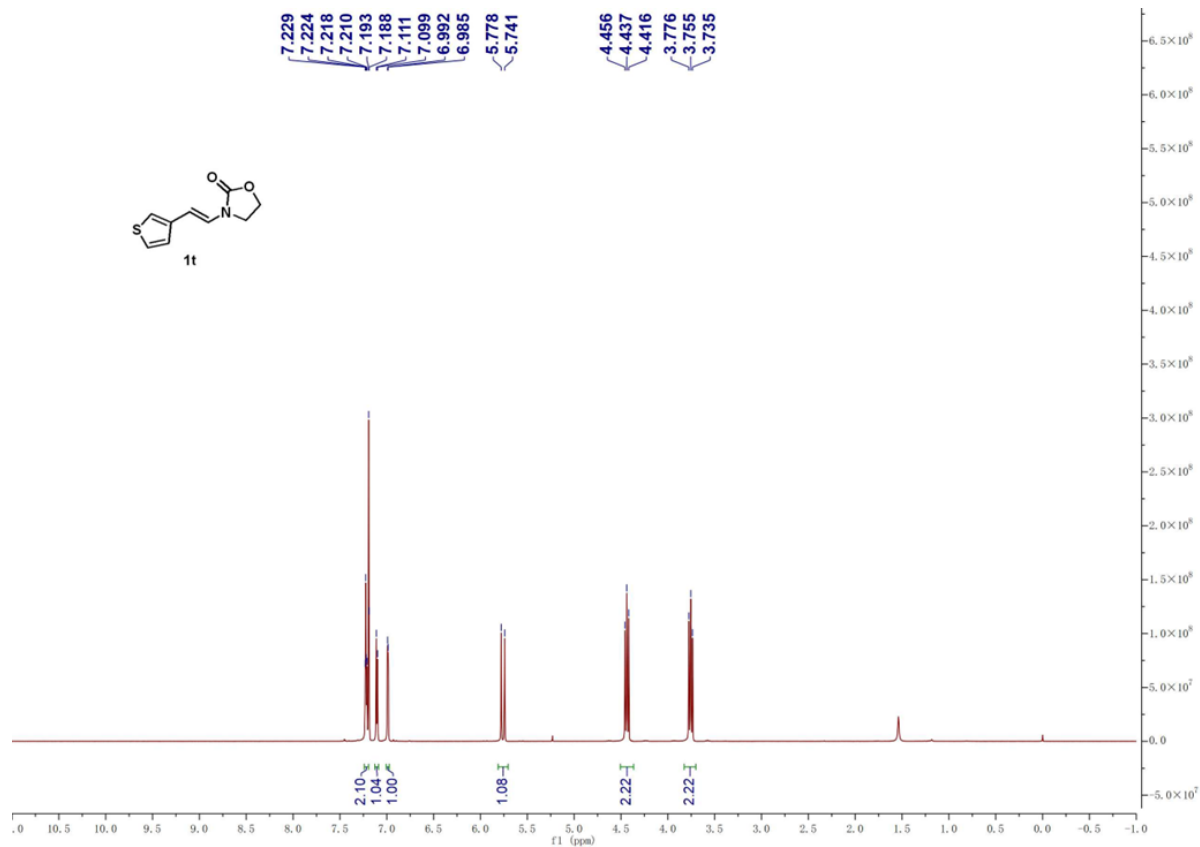


## References

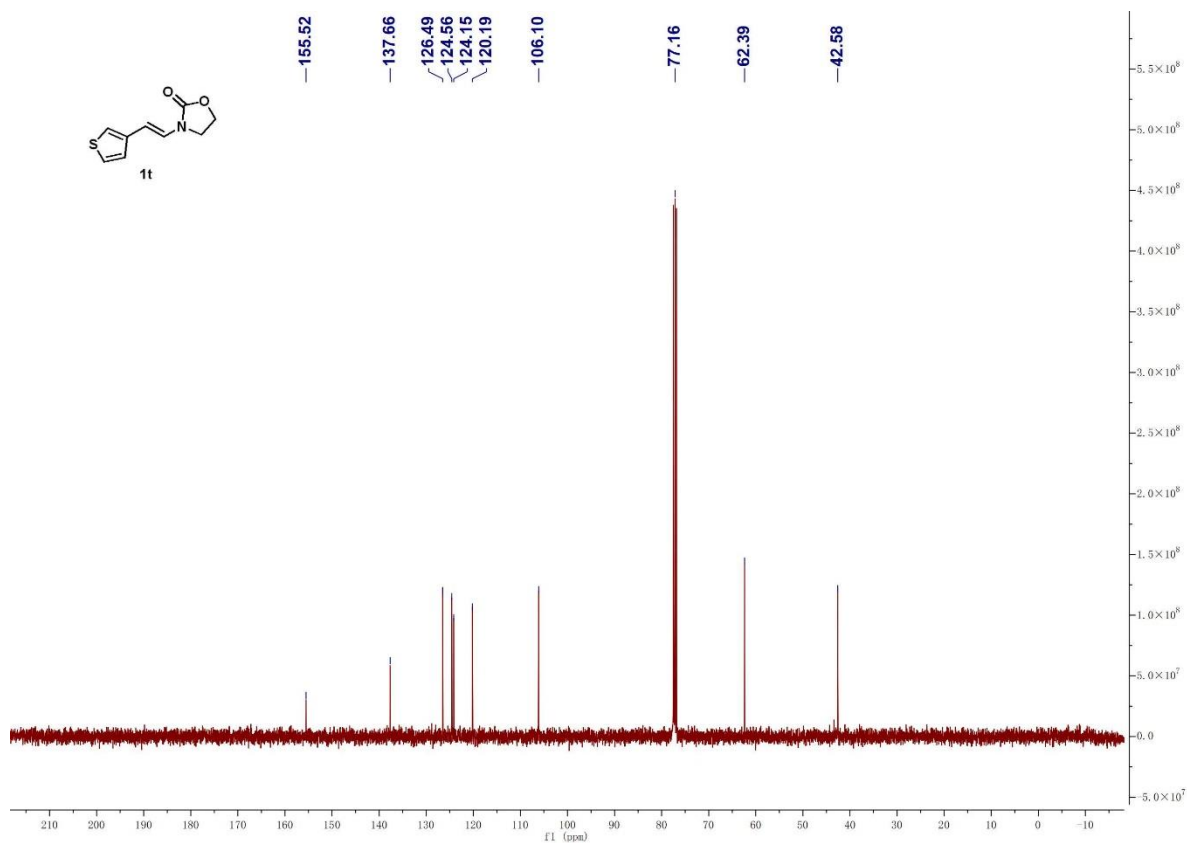
- (1) Y. Xi, C. Wang, Q. Zhang, J. Qu and Y. Chen, *Angew. Chem. Int. Ed.*, 2021, **60**, 2699.
- (2) Y. Xi, W. Huang, C. Wang, H. Ding, T. Xia, K. Fang, J. Qu and Y. Chen, *J. Am. Chem. Soc.*, 2022, **144**, 8389.
- (3) P. Li, N. Ma, Z. Wang, Q. Dai and C. Hu, *J. Org. Chem.*, 2018, **83**, 8233.
- (4) C. W. Cheung and S. L. Buchwald, *J. Org. Chem.*, 2012, **77**, 7526.
- (5) T. B. Nguyen, A. Martel, R. Dhal and G. Dujardin, *J. Org. Chem.*, 2008, **73**, 2621.
- (6) A. H. Cherney and S. E. Reisman, *J. Am. Chem. Soc.*, 2014, **136**, 14365.

## NMR Spectrum

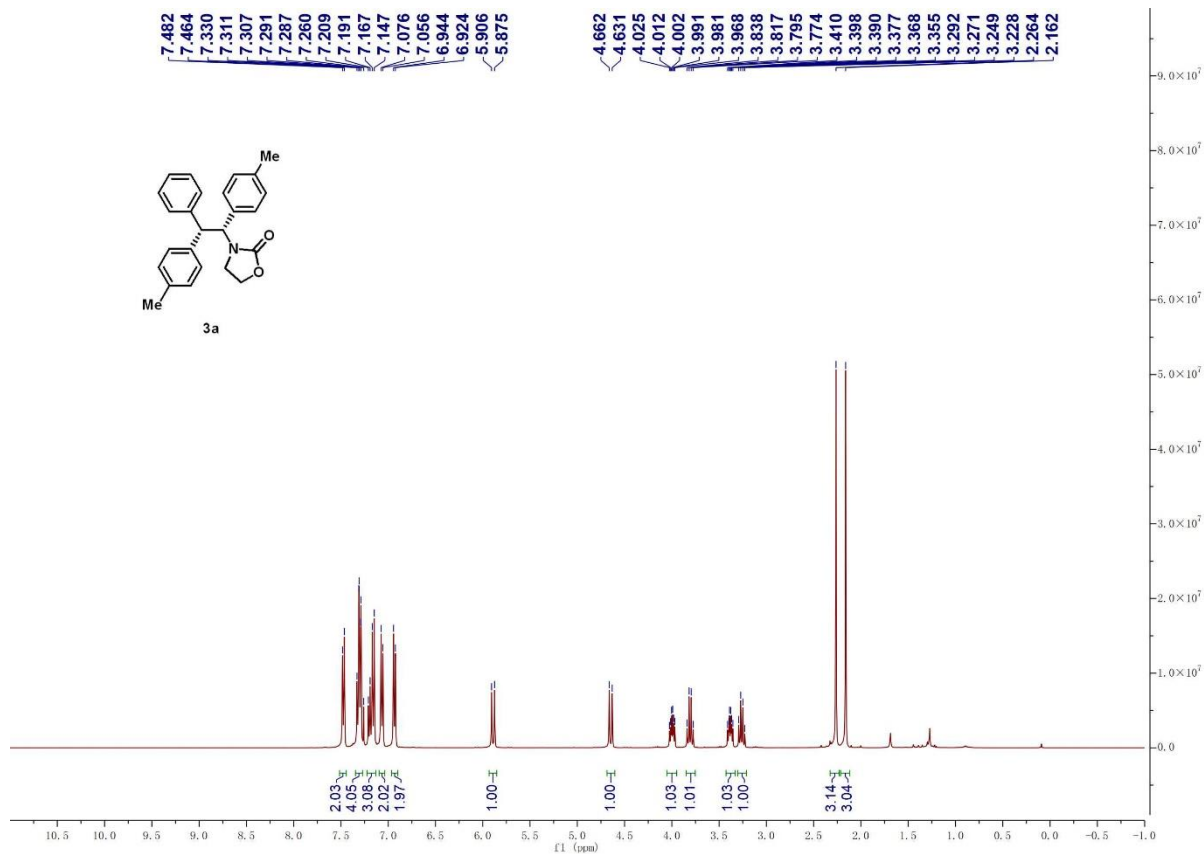
$^1\text{H}$  NMR-spectrum (400 MHz,  $\text{CDCl}_3$ ) of **1t**



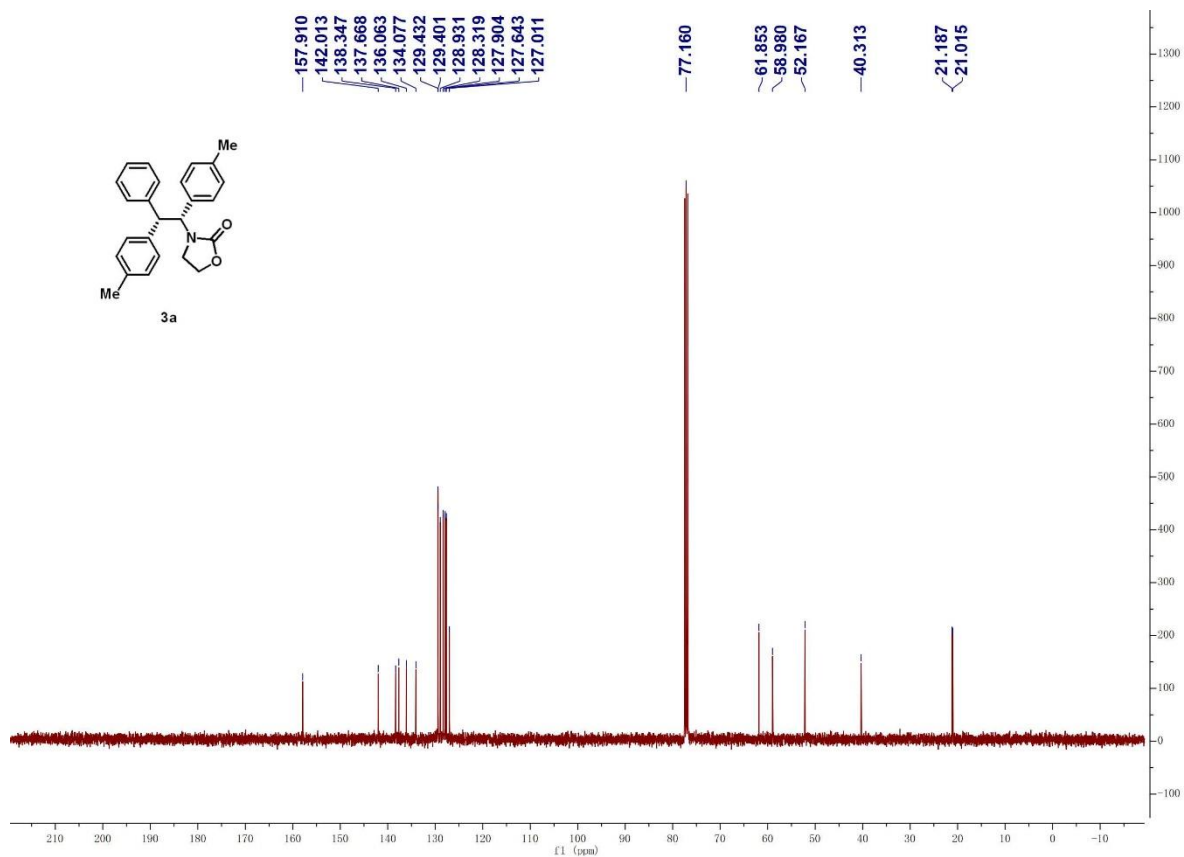
$^{13}\text{C}$  NMR-spectrum (100 MHz,  $\text{CDCl}_3$ ) of **1t**



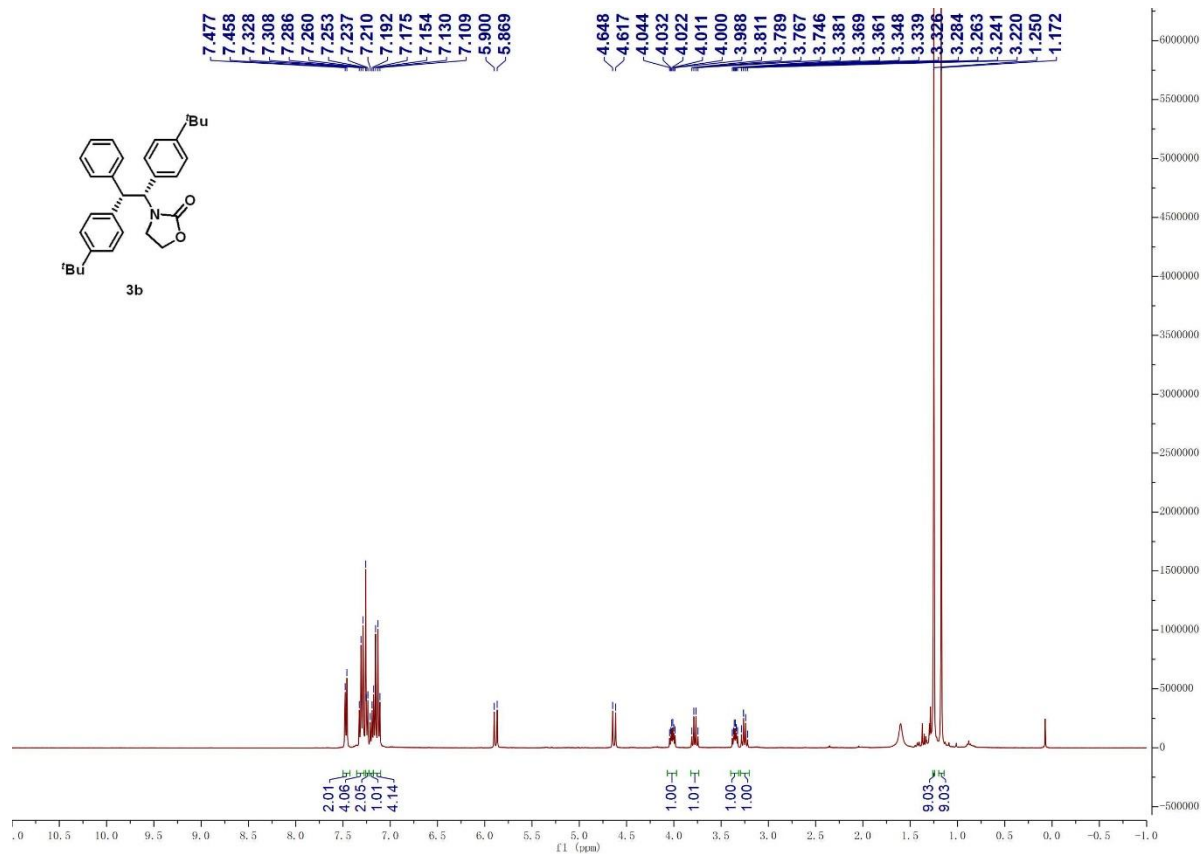
**<sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of 3a**



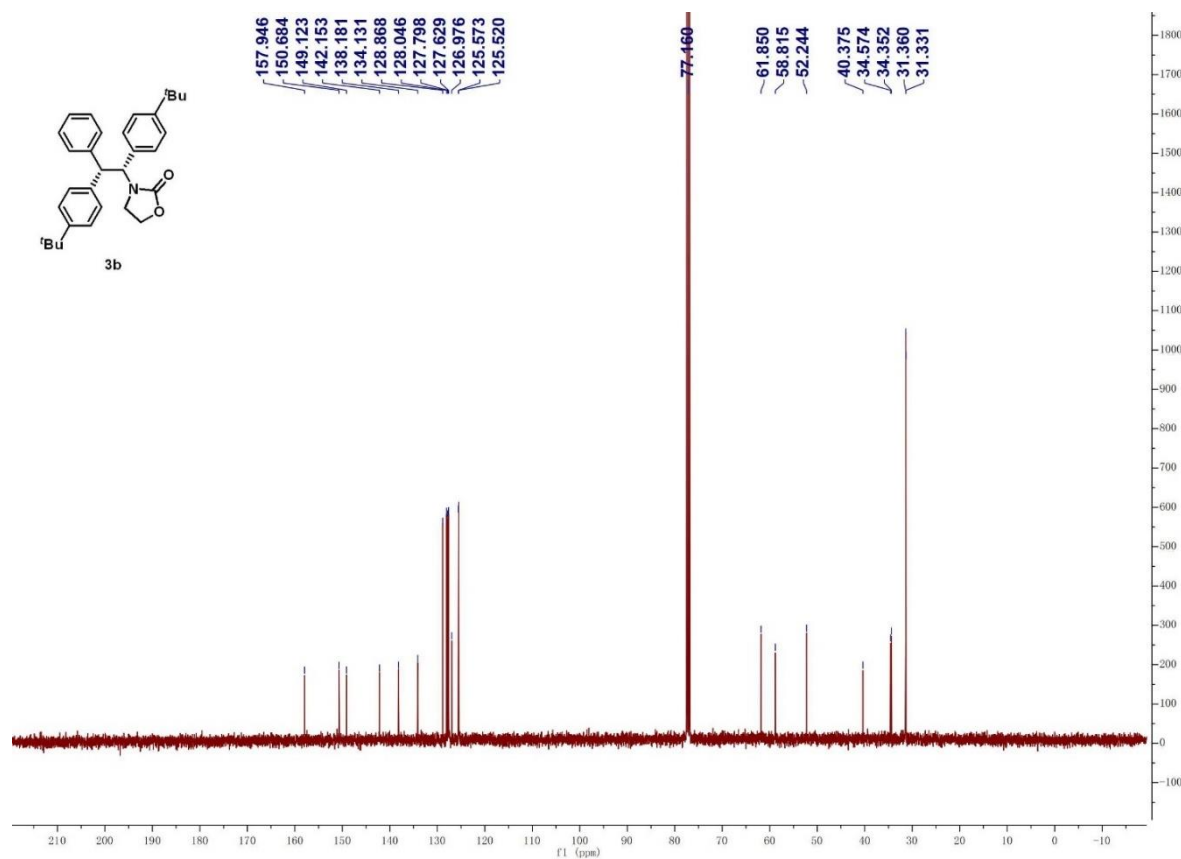
**<sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of 3a**



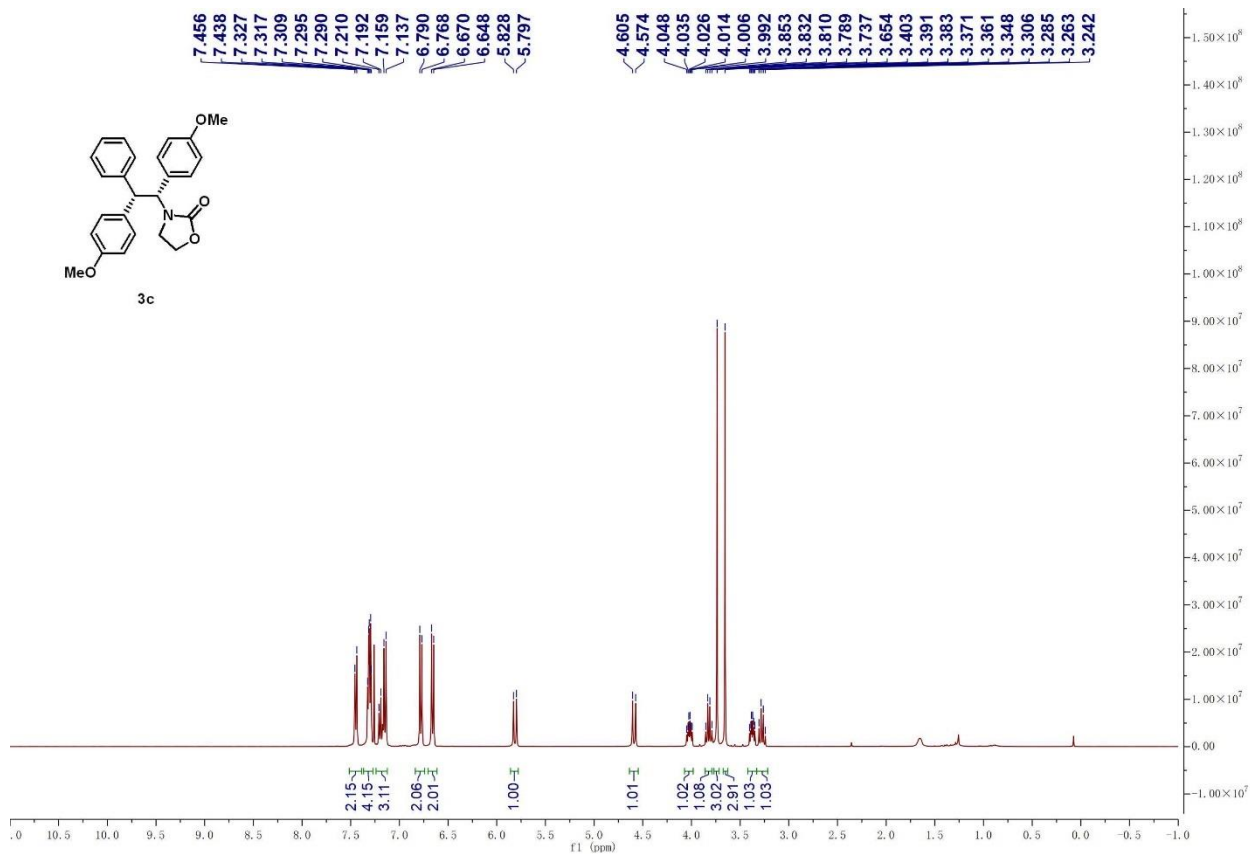
### $^1\text{H}$ NMR-spectrum (400 MHz, $\text{CDCl}_3$ ) of **3b**



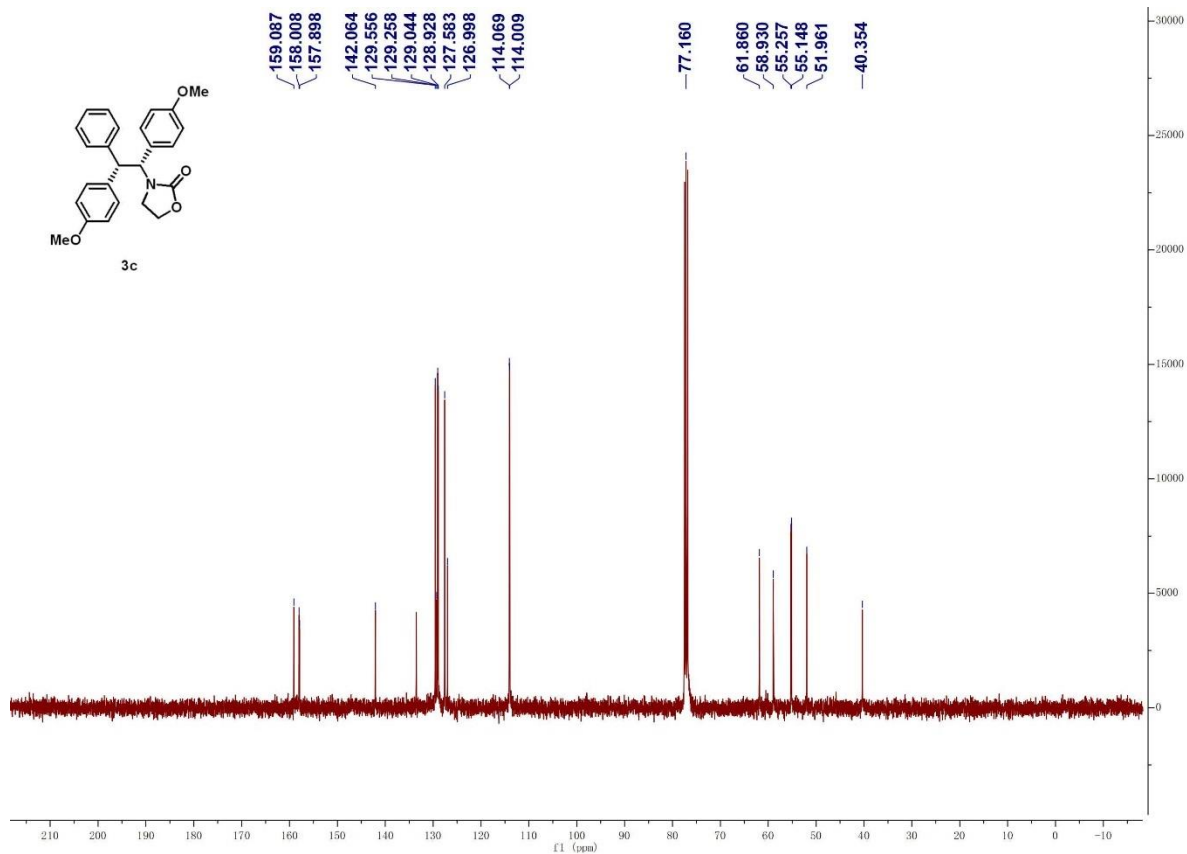
### $^{13}\text{C}$ NMR-spectrum (100 MHz, $\text{CDCl}_3$ ) of **3b**



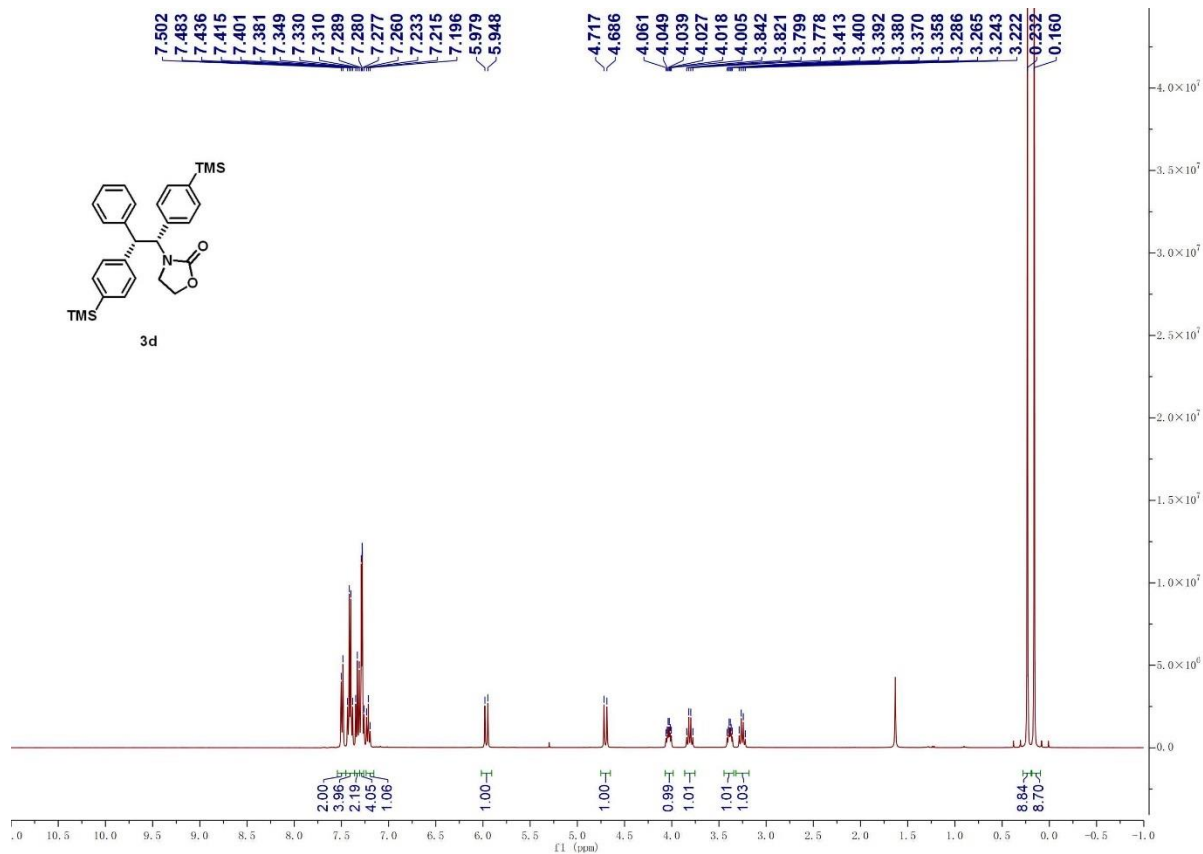
### $^1\text{H}$ NMR-spectrum (400 MHz, $\text{CDCl}_3$ ) of **3c**



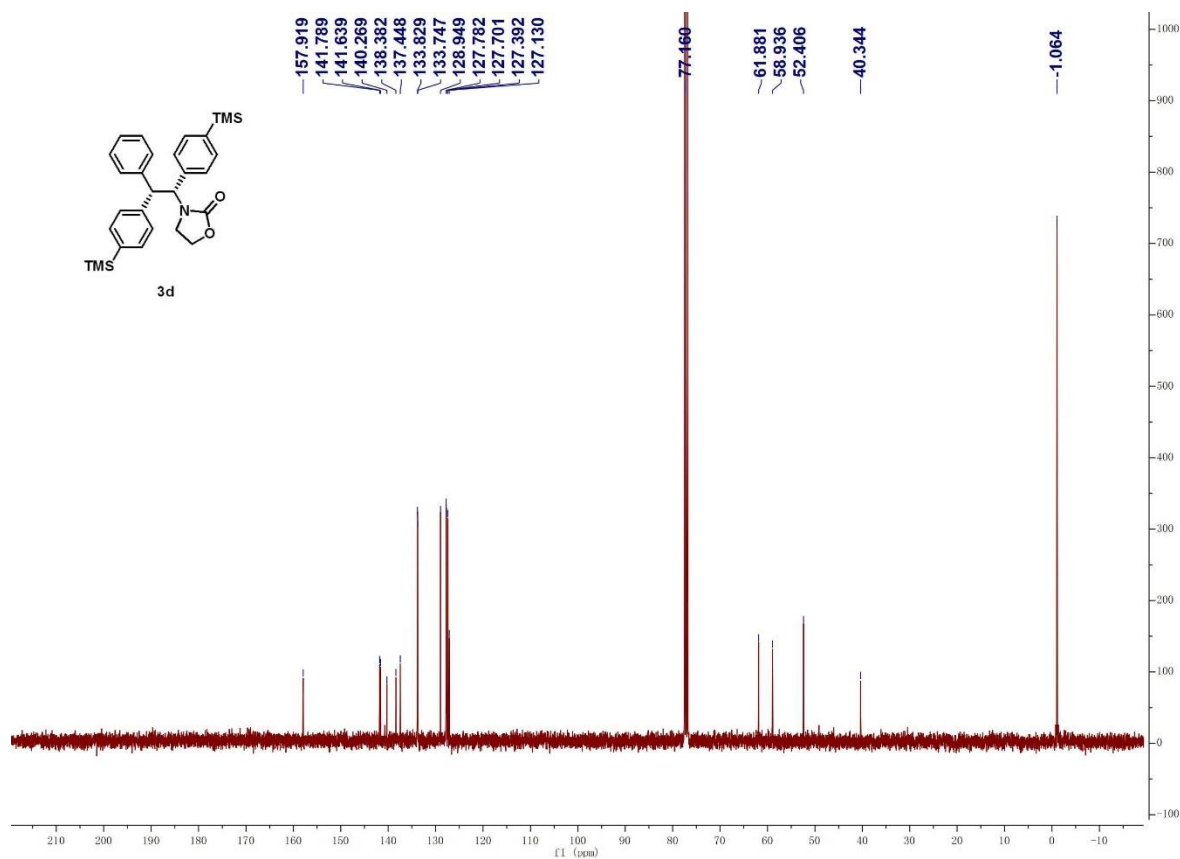
### $^{13}\text{C}$ NMR-spectrum (100 MHz, $\text{CDCl}_3$ ) of **3c**



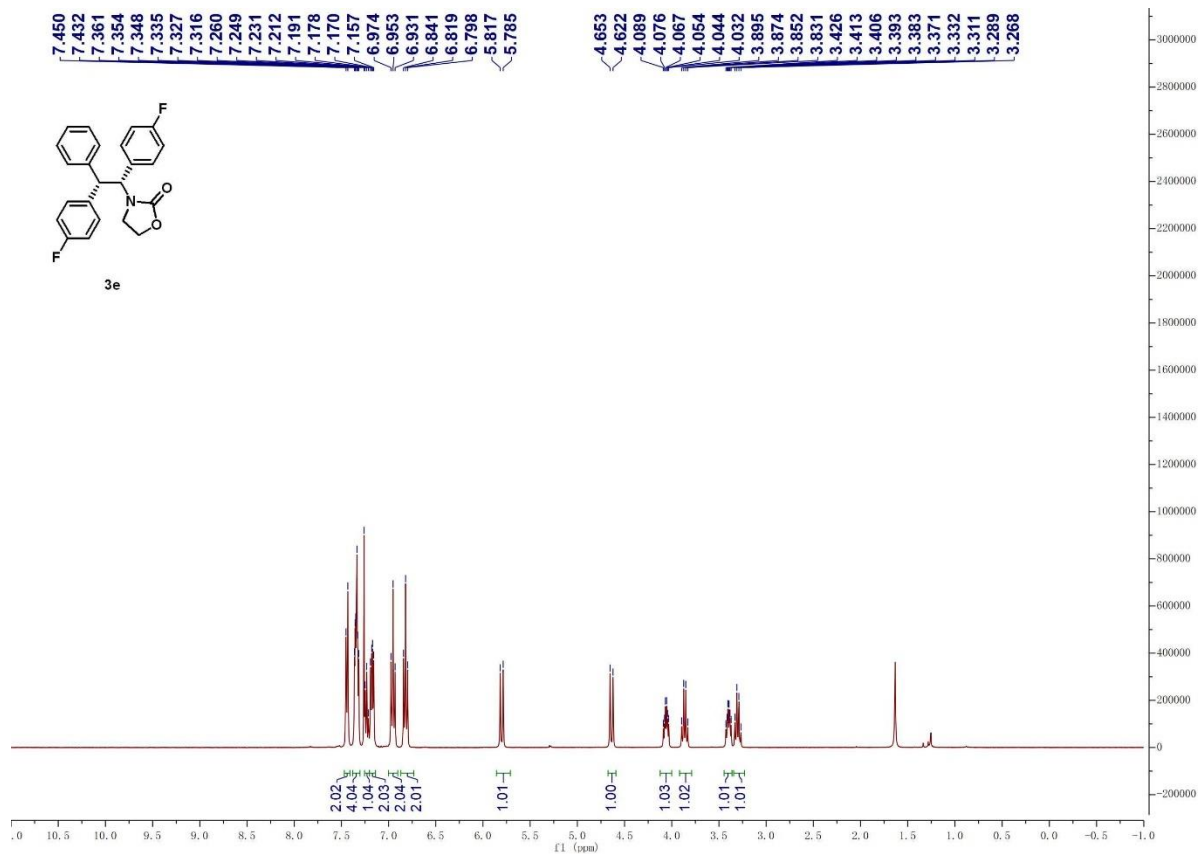
<sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of **3d**



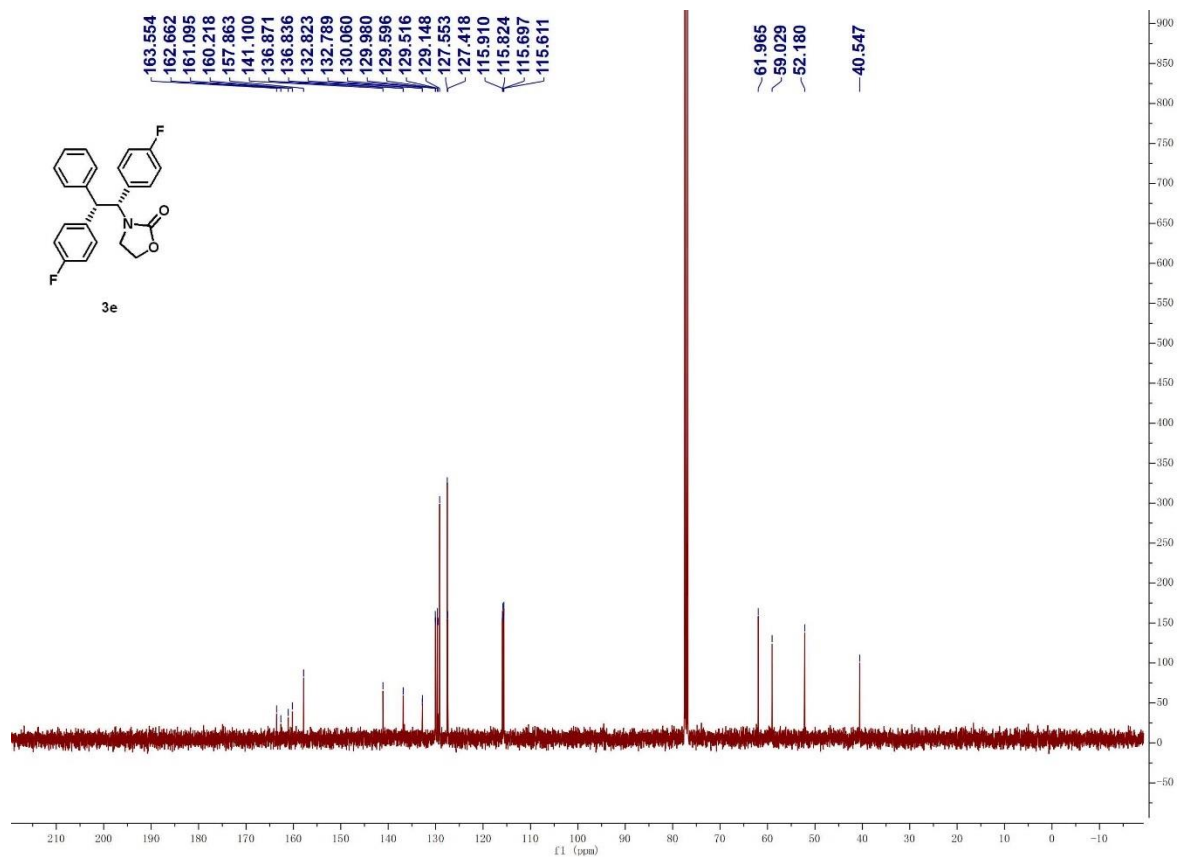
<sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of **3d**



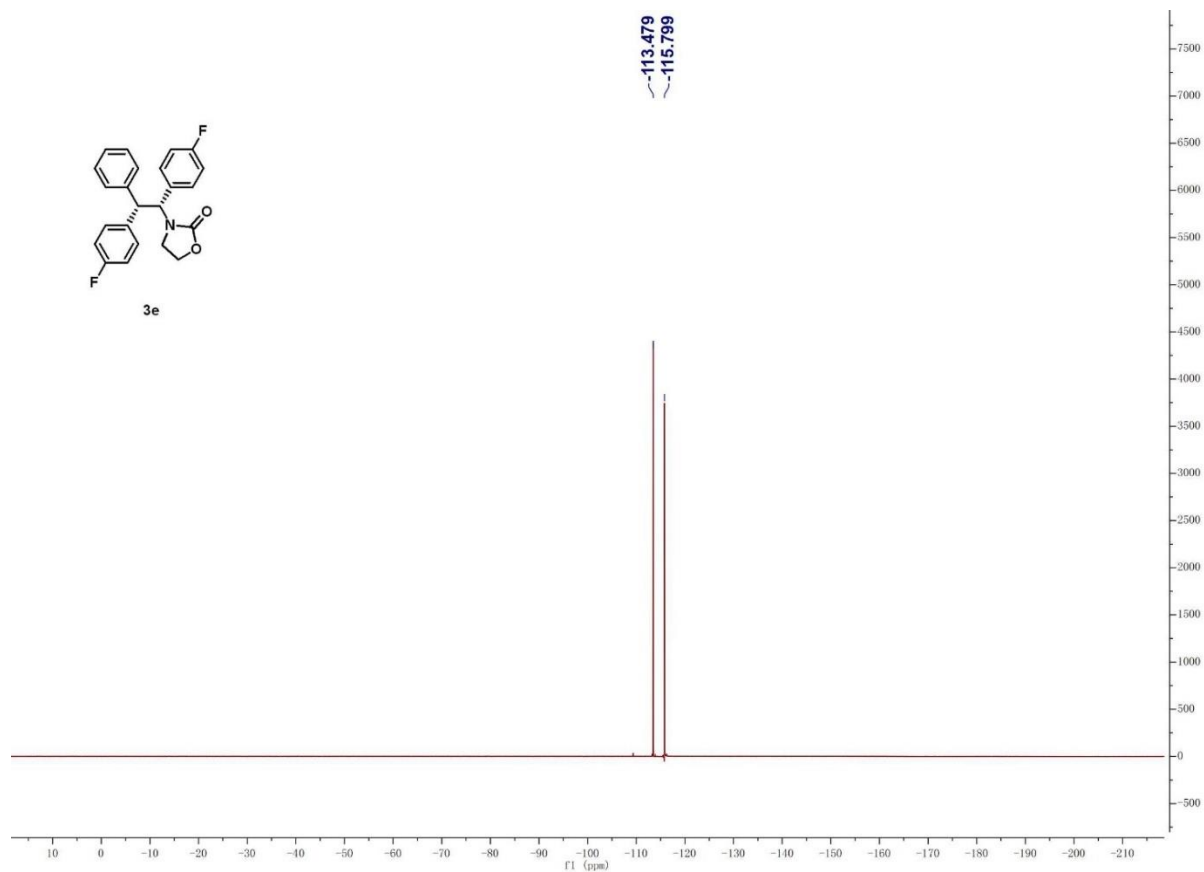
### <sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of **3e**



### <sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of **3e**

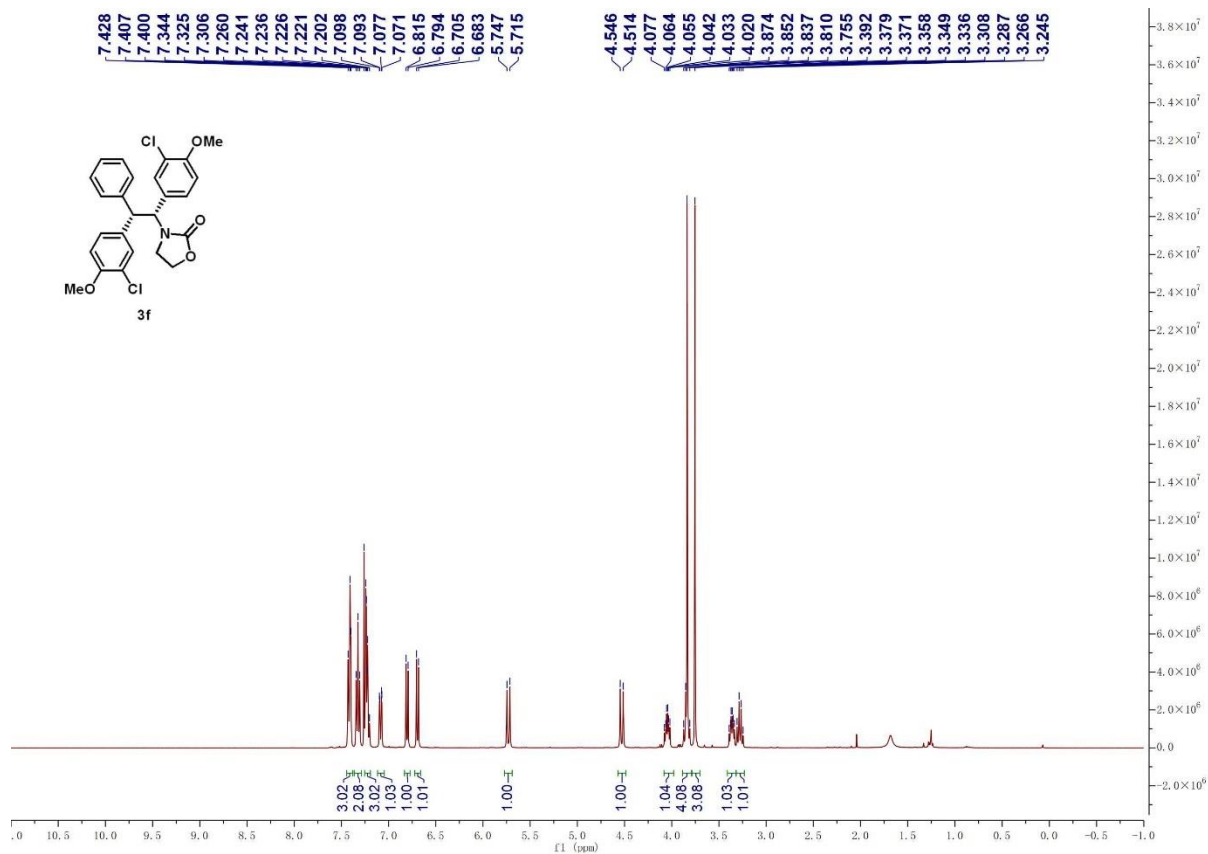


<sup>19</sup>F NMR-spectrum (376 MHz, CDCl<sub>3</sub>) of **3e**

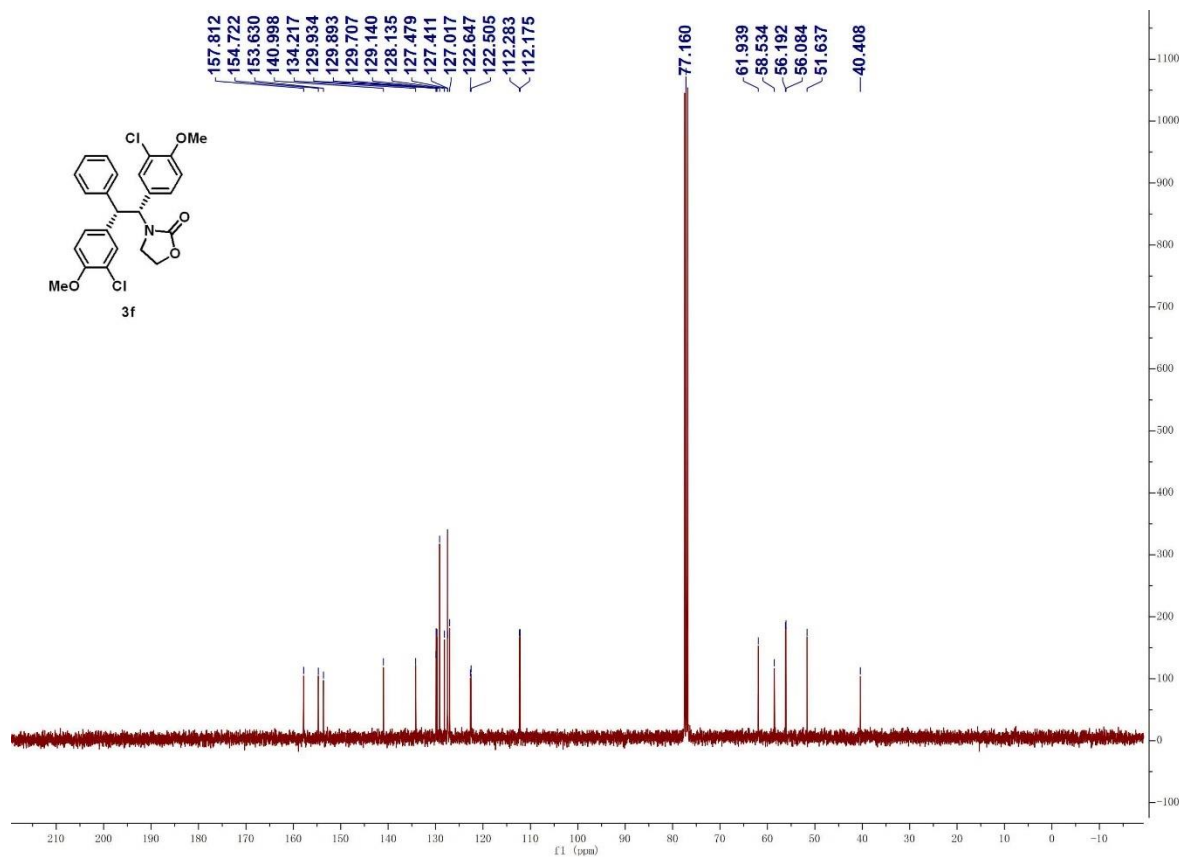




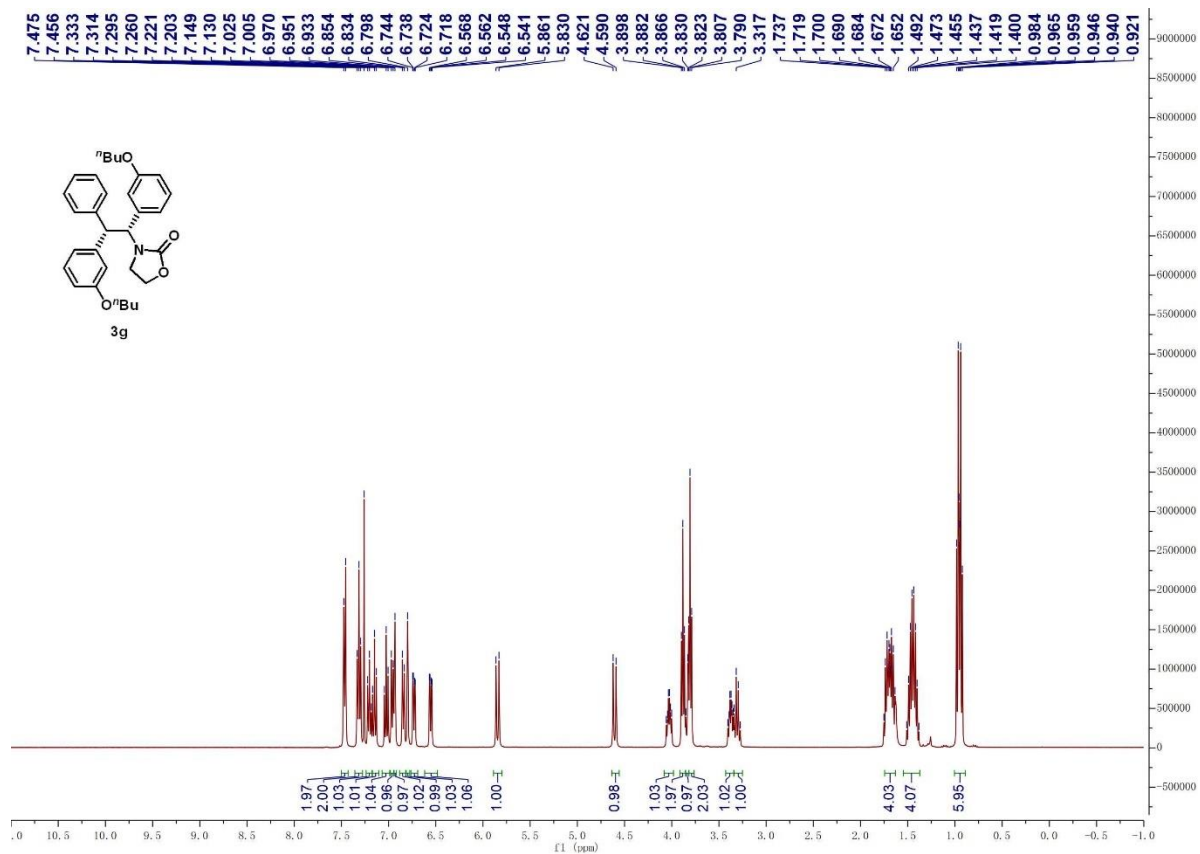
### <sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of **3f**



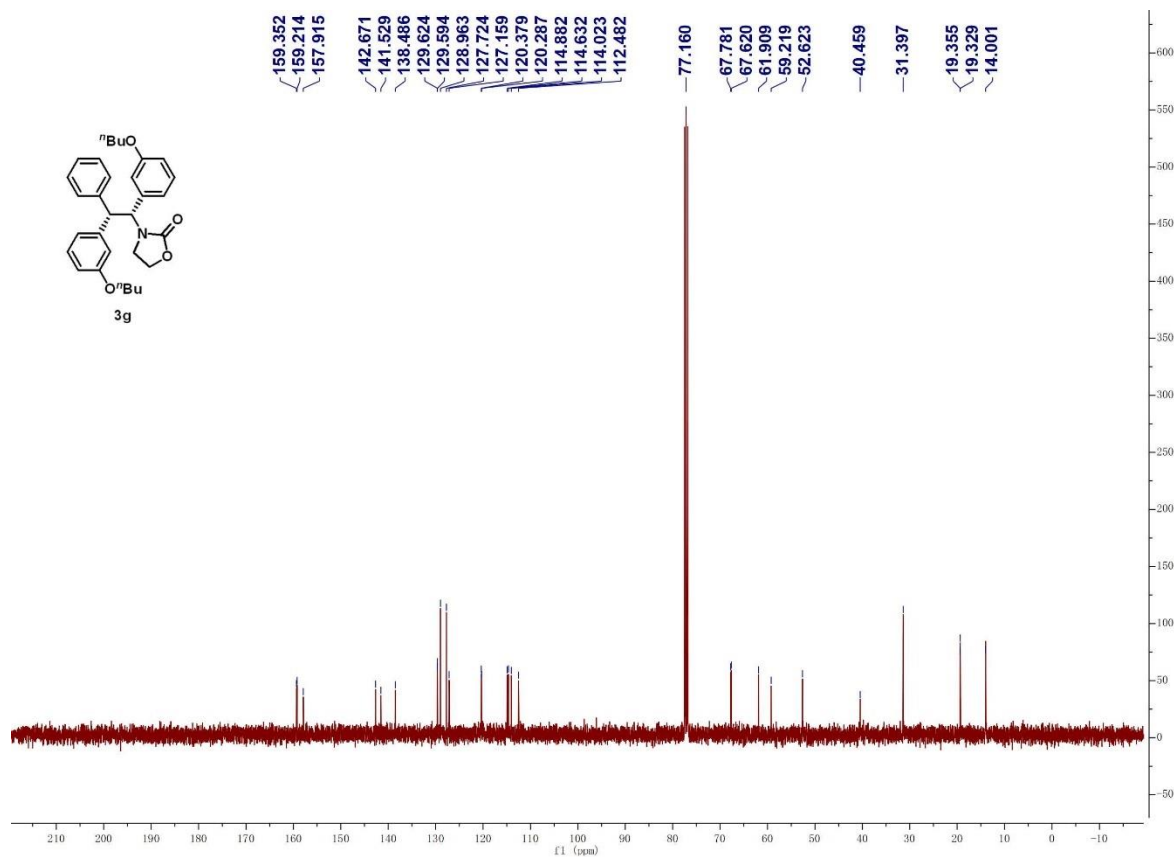
### <sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of **3f**



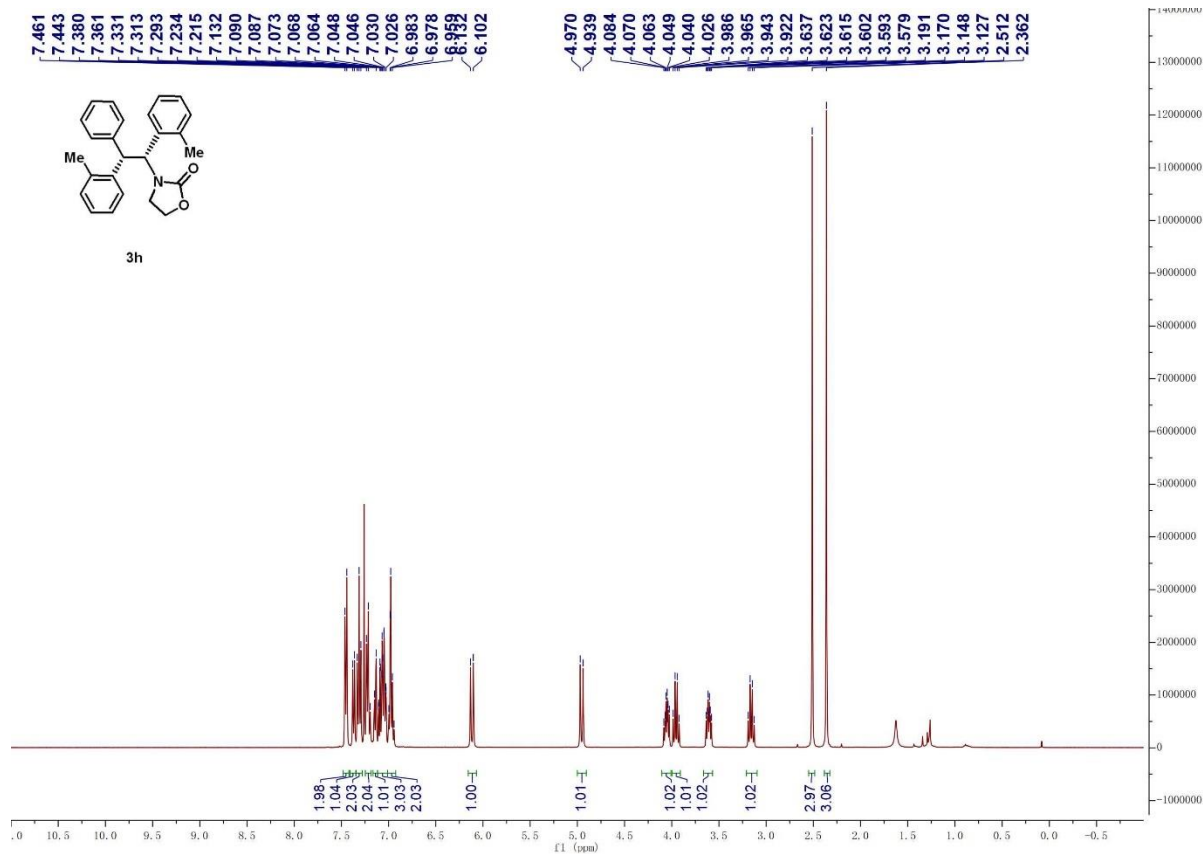
<sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of **3g**



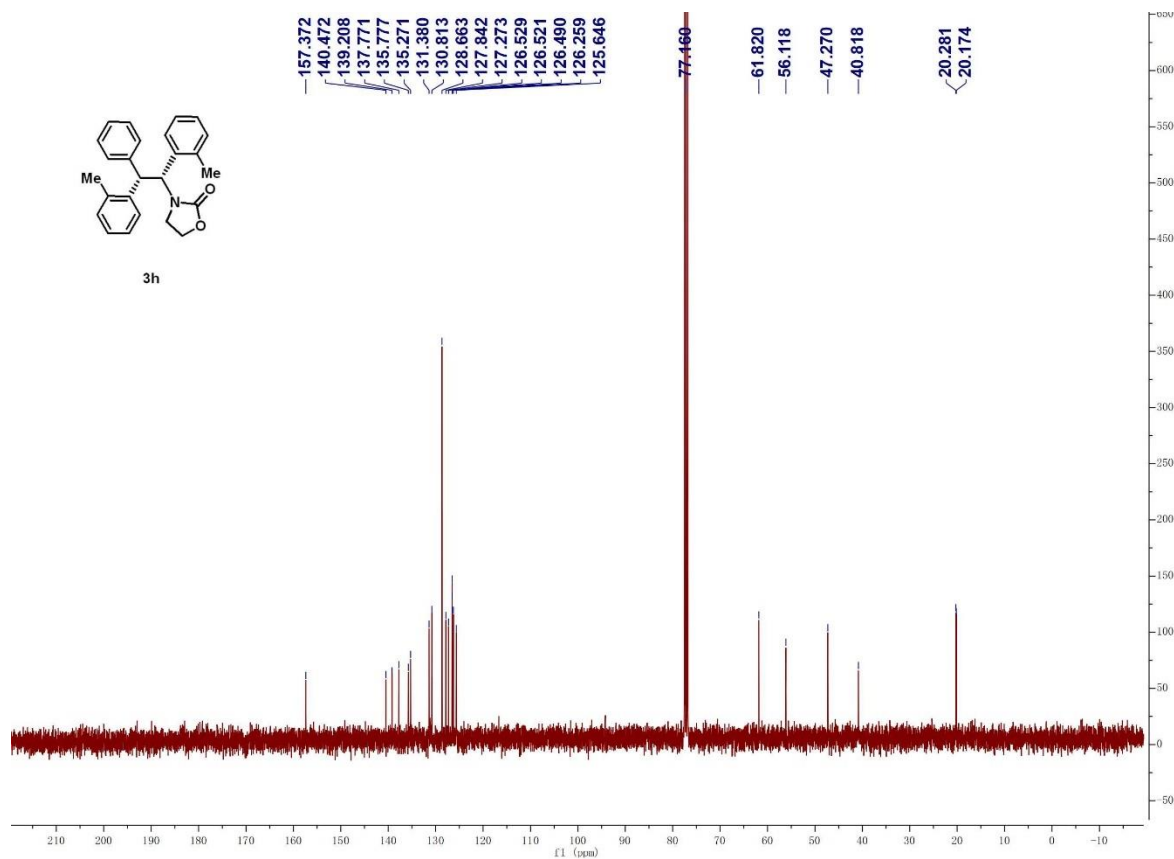
<sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of **3g**



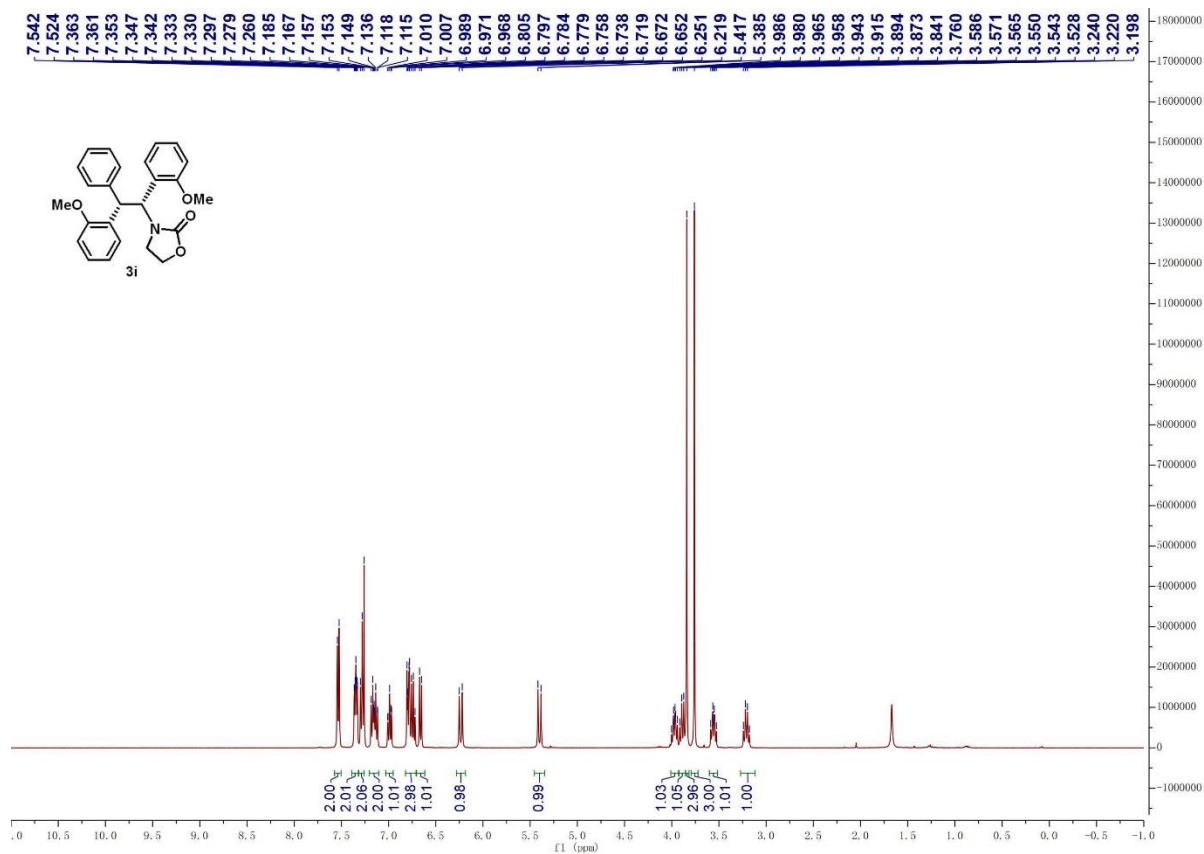
### $^1\text{H}$ NMR-spectrum (400 MHz, $\text{CDCl}_3$ ) of **3h**



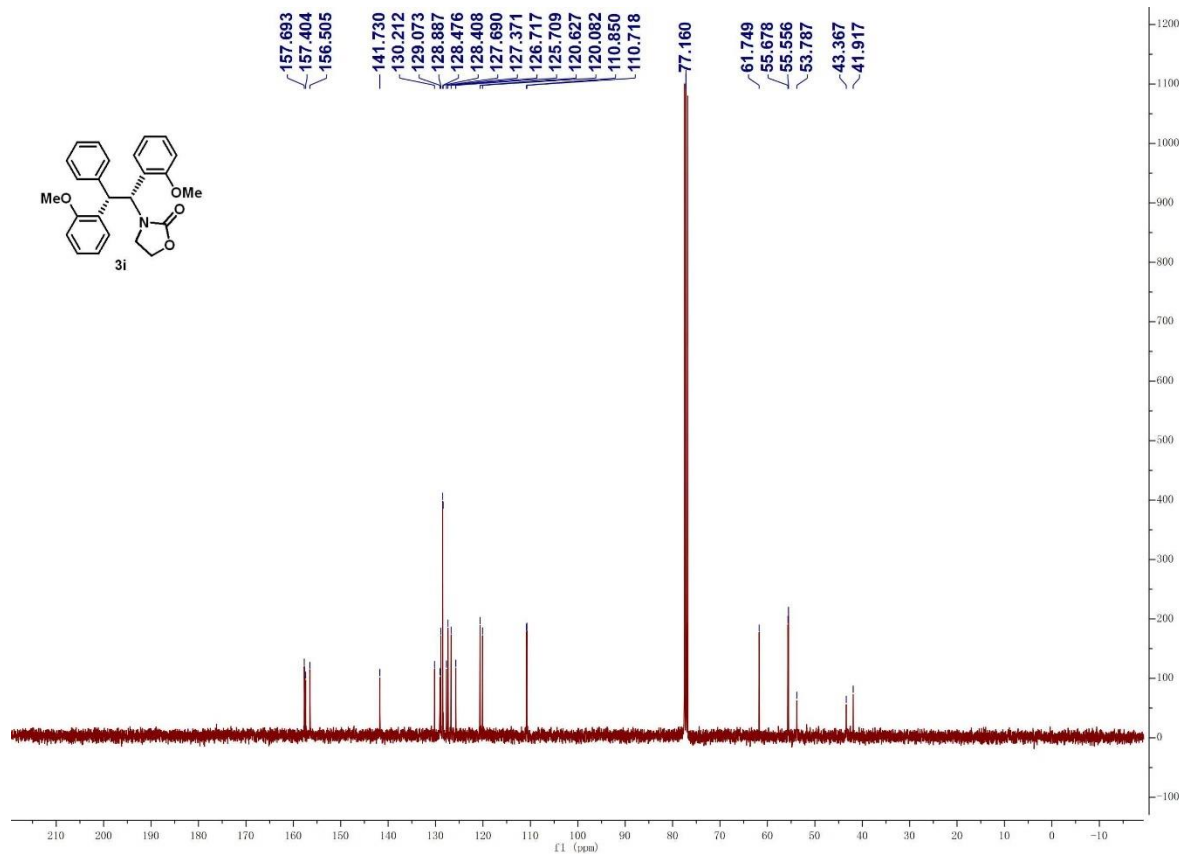
### $^{13}\text{C}$ NMR-spectrum (100 MHz, $\text{CDCl}_3$ ) of **3h**



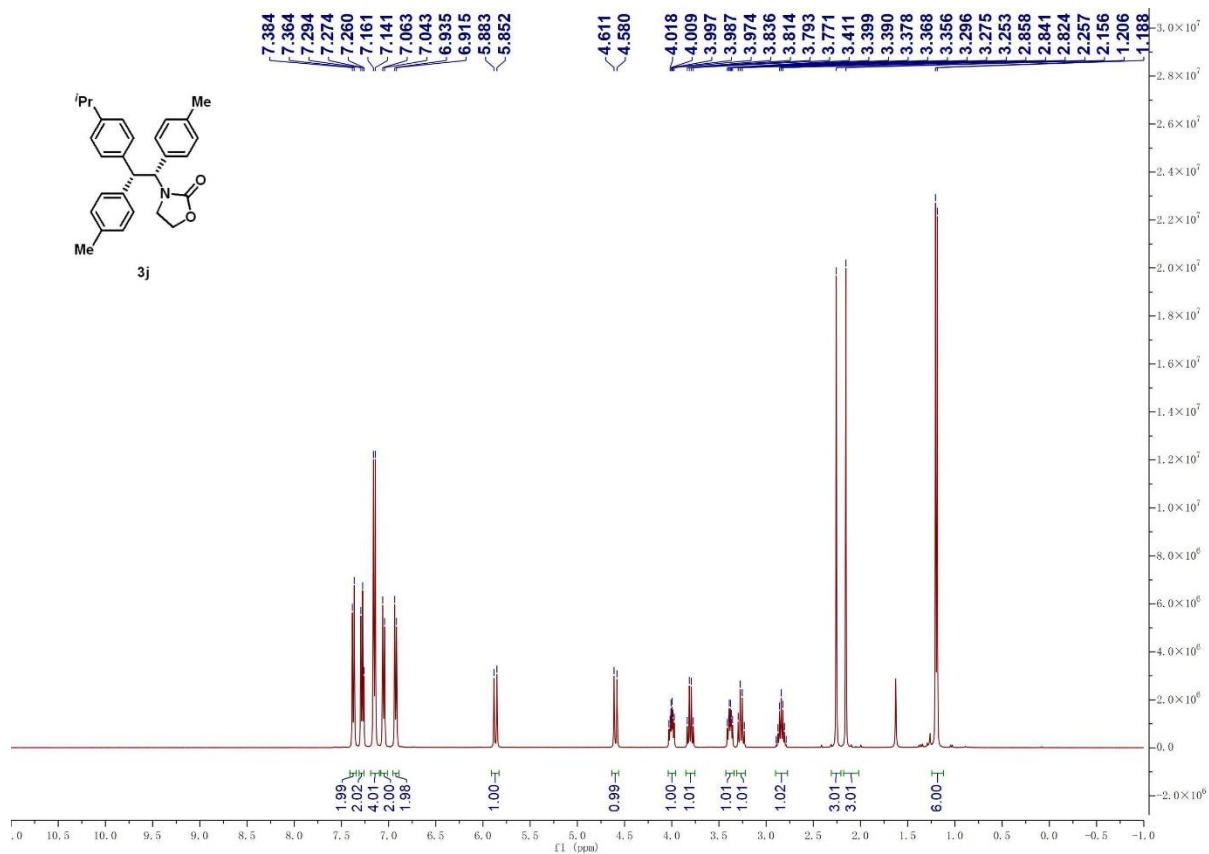
**<sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of 3i**



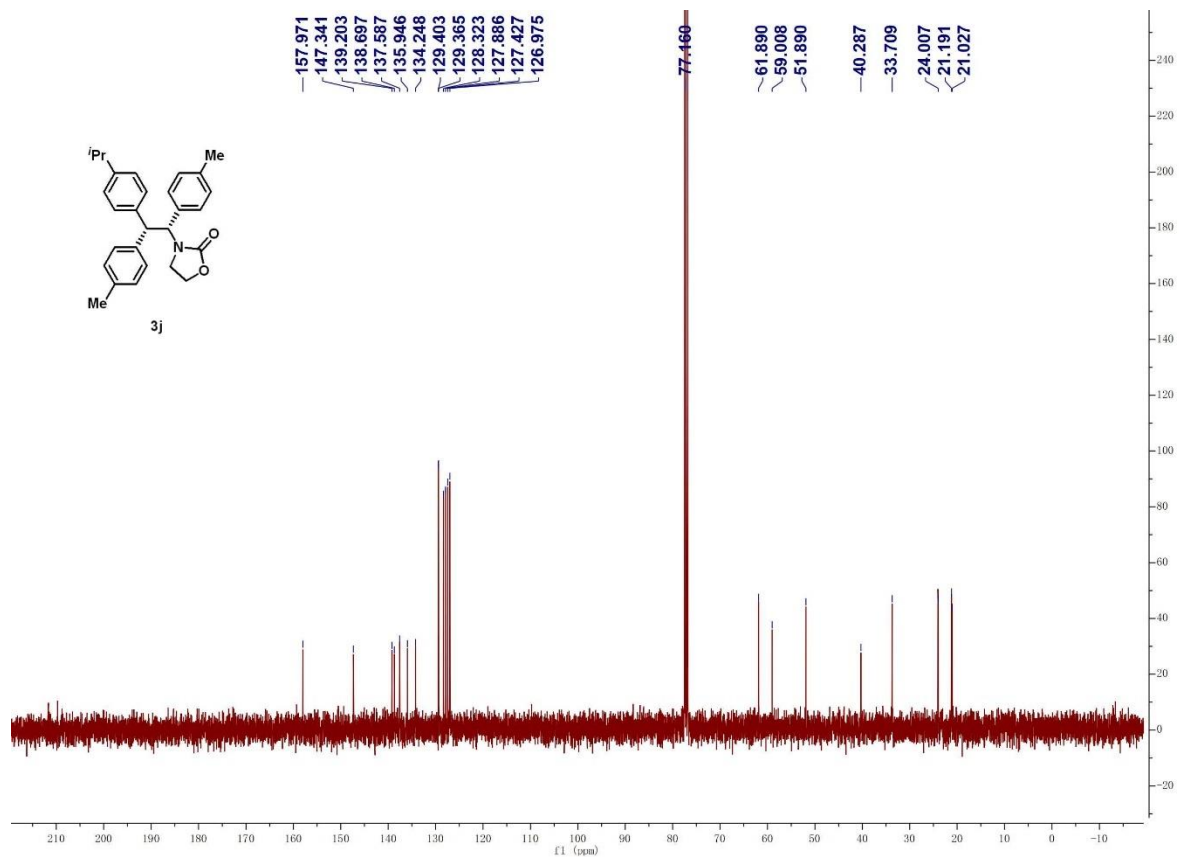
**<sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of 3i**



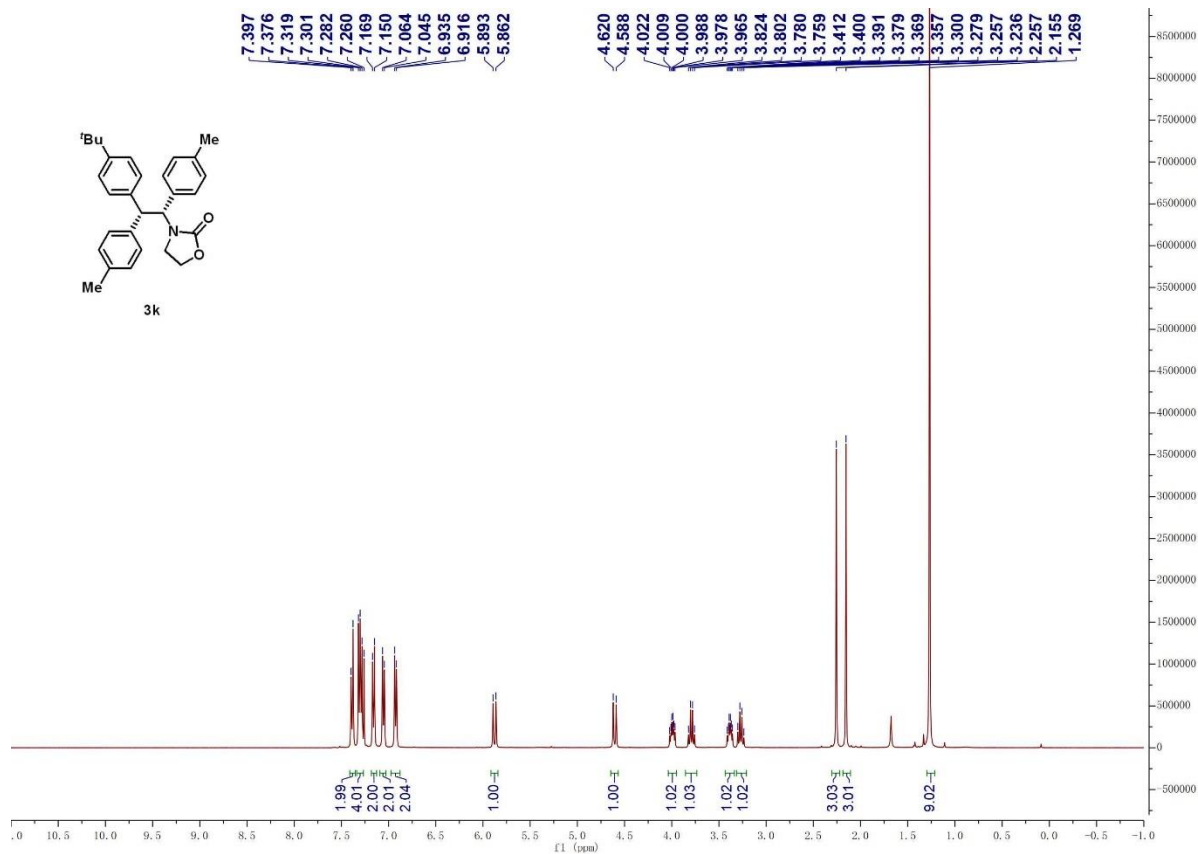
### $^1\text{H}$ NMR-spectrum (400 MHz, $\text{CDCl}_3$ ) of **3j**



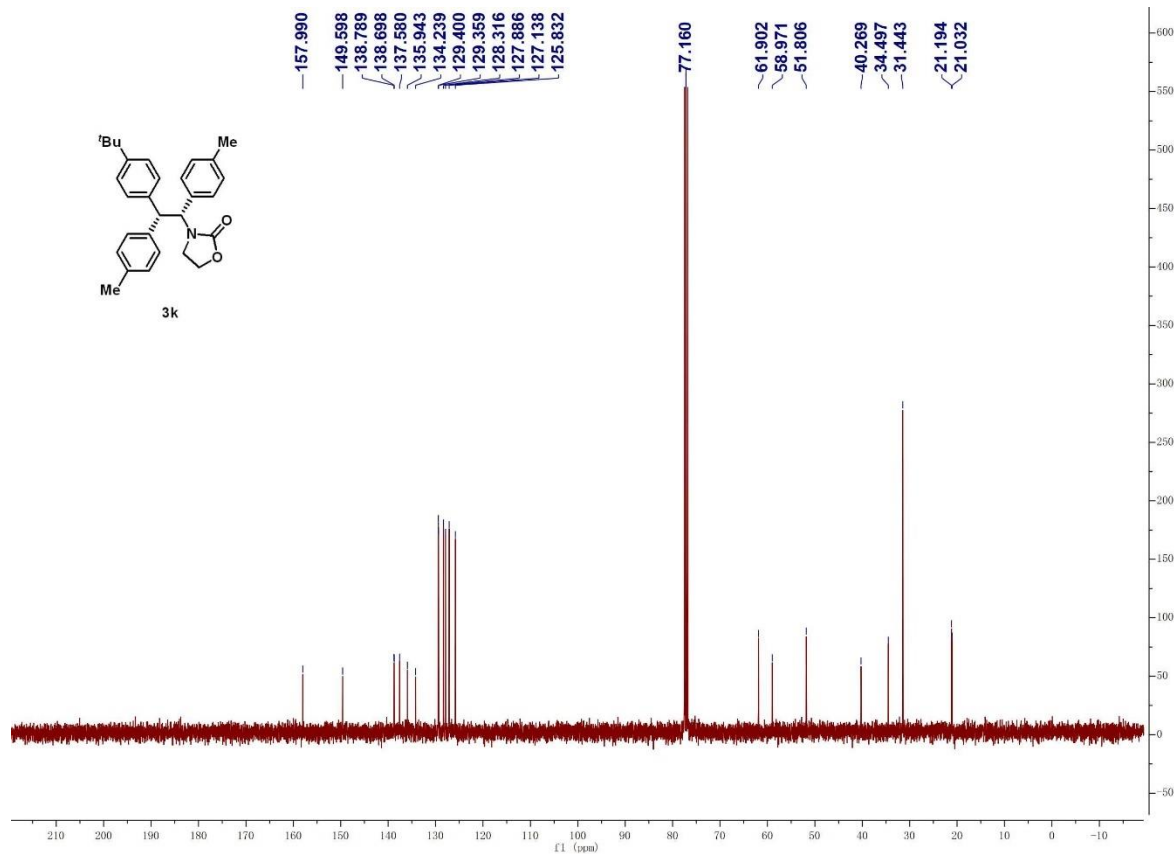
### $^{13}\text{C}$ NMR-spectrum (100 MHz, $\text{CDCl}_3$ ) of **3j**



**<sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of 3k**

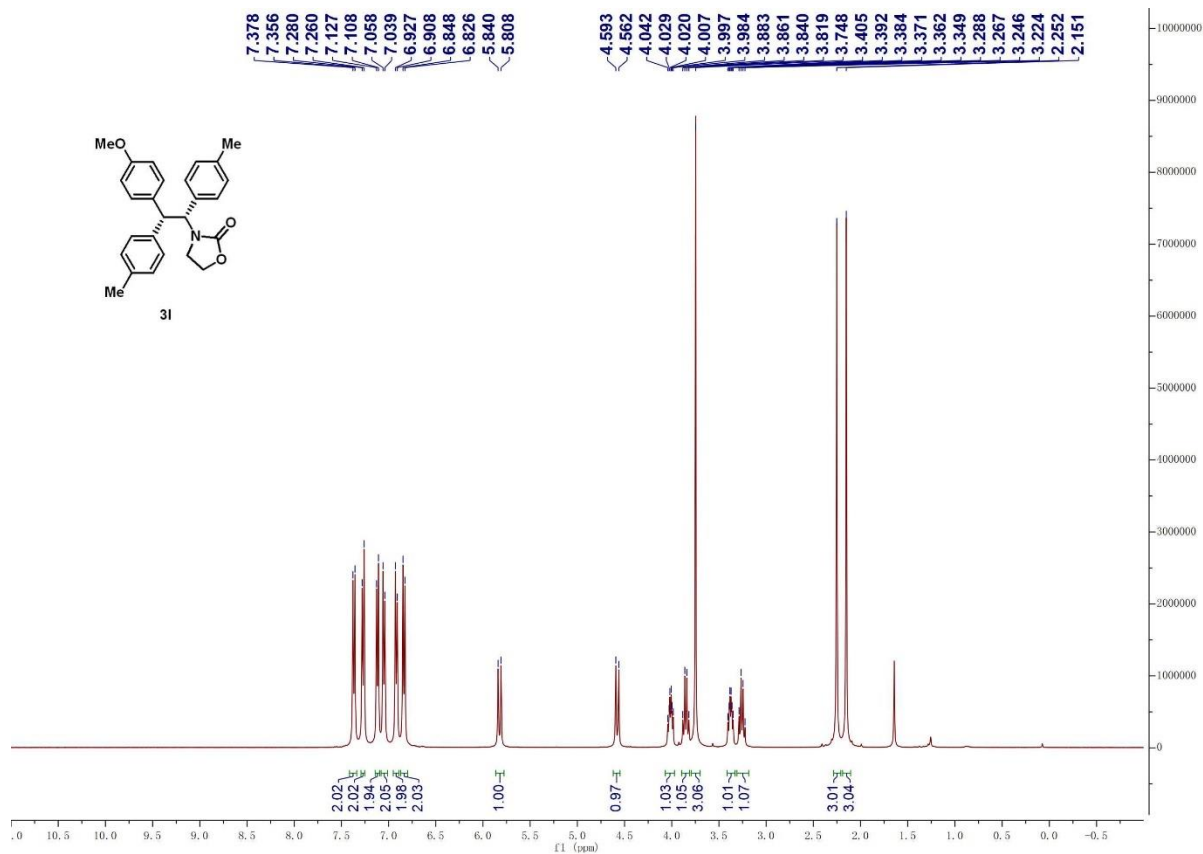


**<sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of 3k**

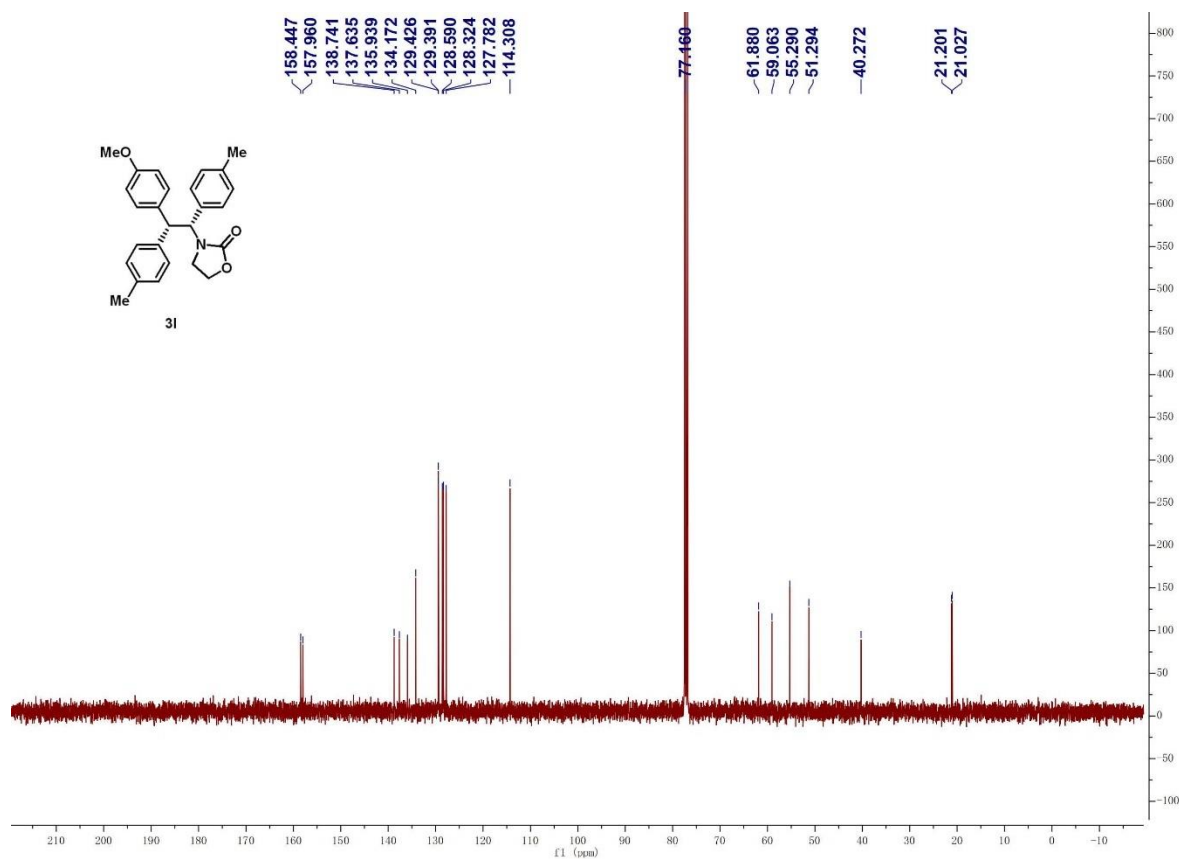




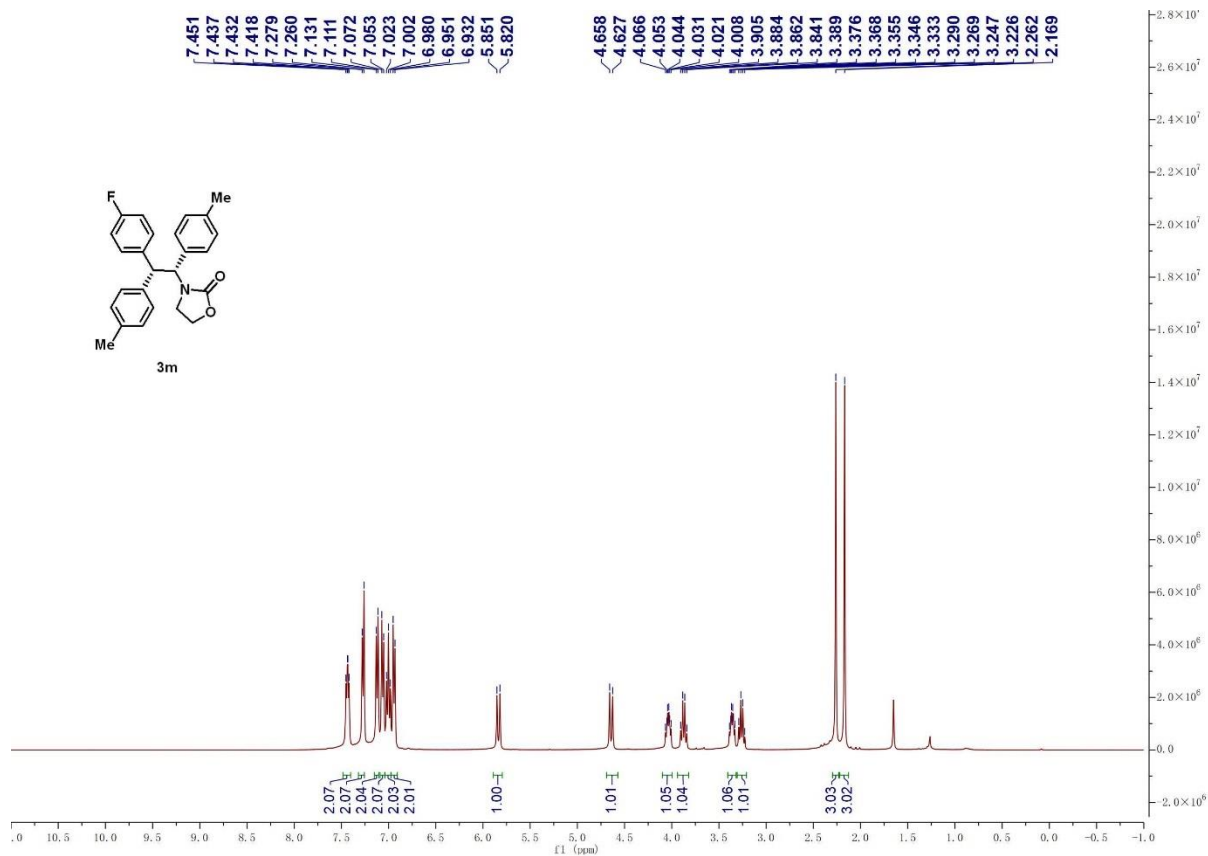
### $^1\text{H}$ NMR-spectrum (400 MHz, $\text{CDCl}_3$ ) of **31**



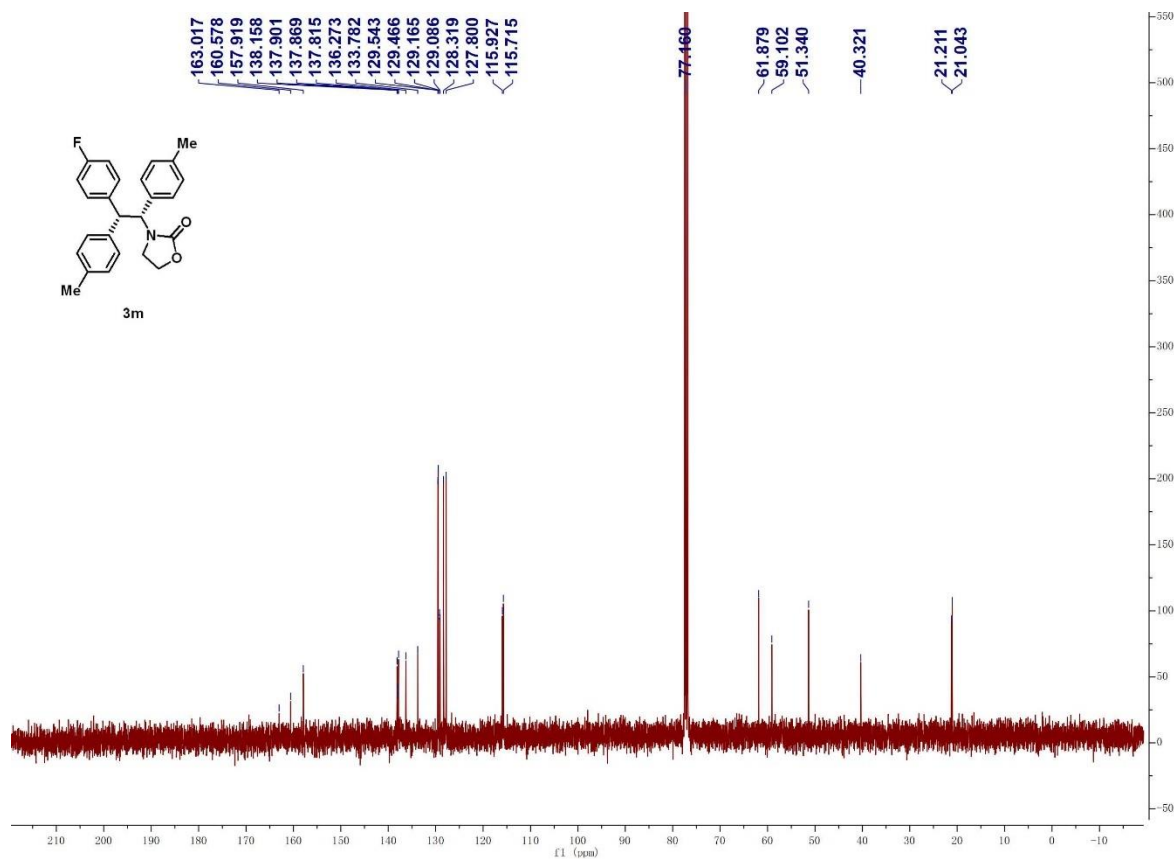
### $^{13}\text{C}$ NMR-spectrum (100 MHz, $\text{CDCl}_3$ ) of **31**



<sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of **3m**

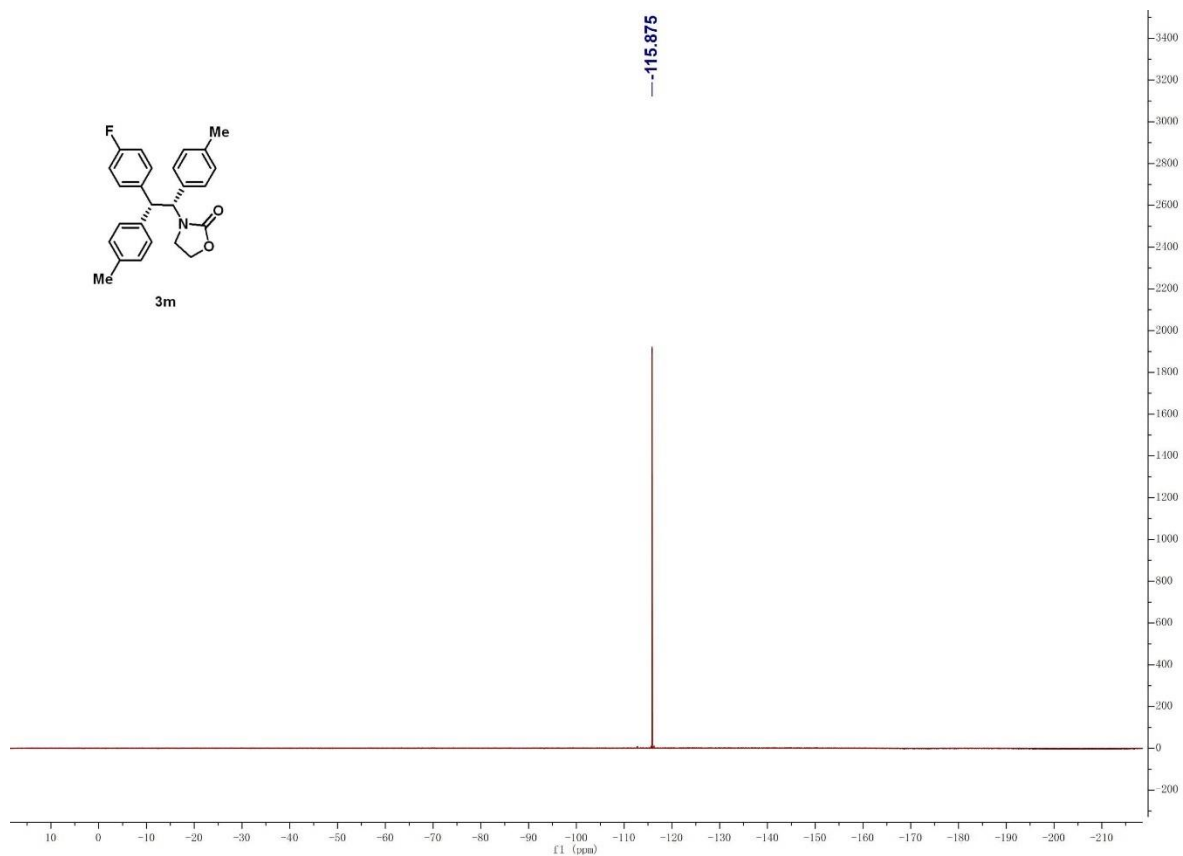


<sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of **3m**

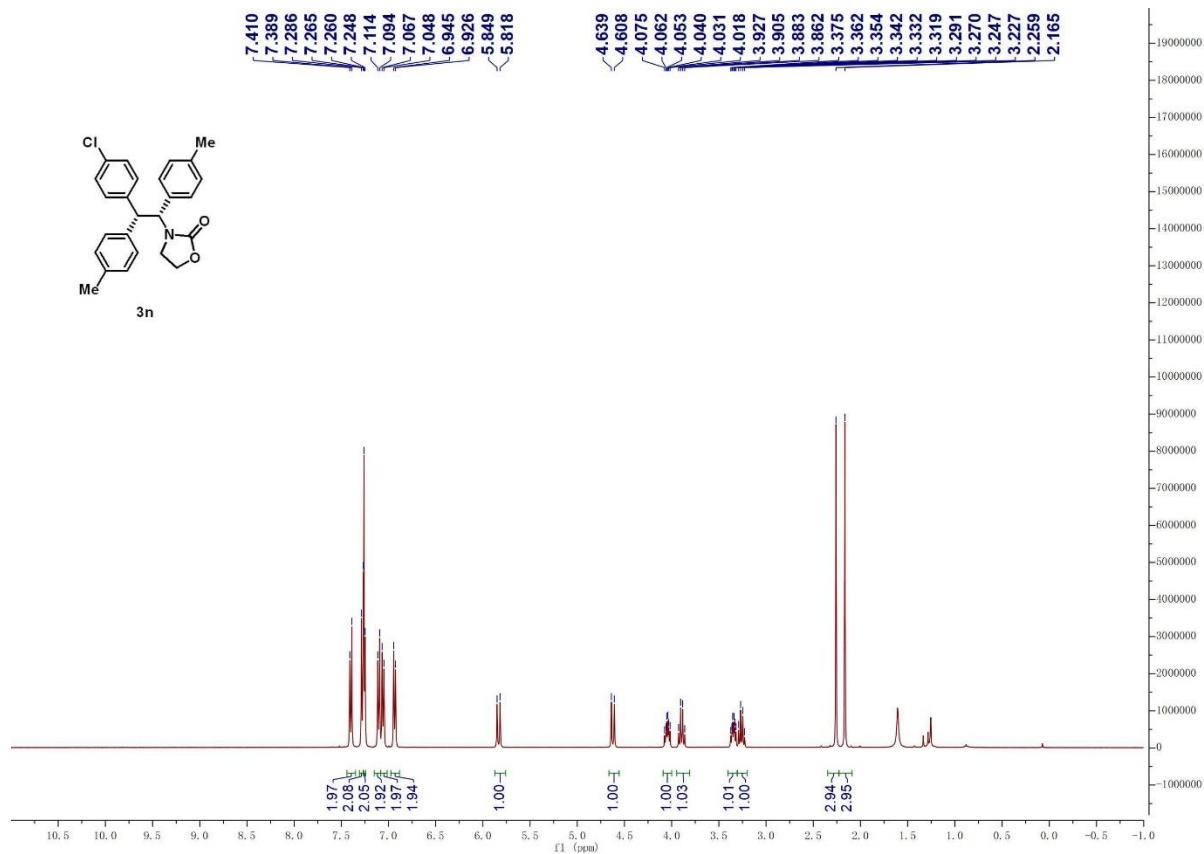




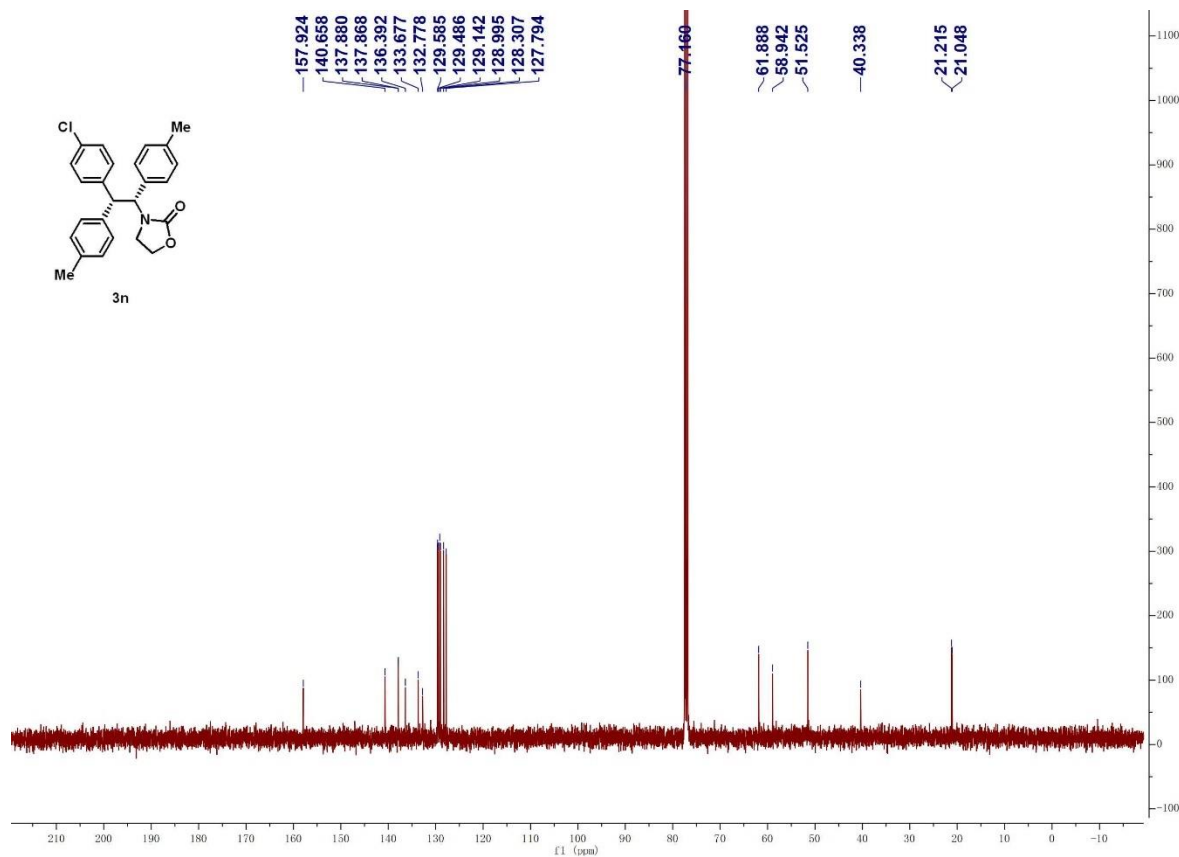
**$^{19}\text{F}$  NMR-spectrum (376 MHz,  $\text{CDCl}_3$ ) of **3m****



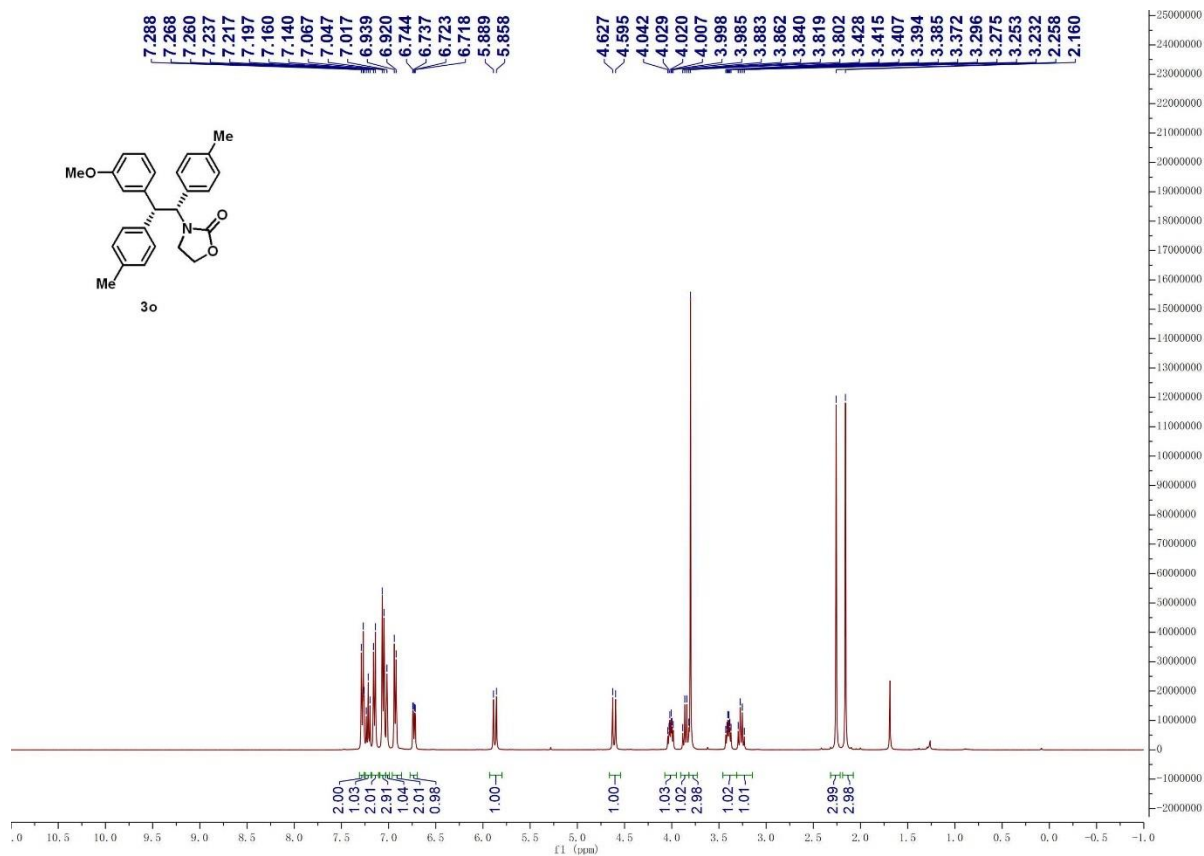
### $^1\text{H}$ NMR-spectrum (400 MHz, $\text{CDCl}_3$ ) of **3n**



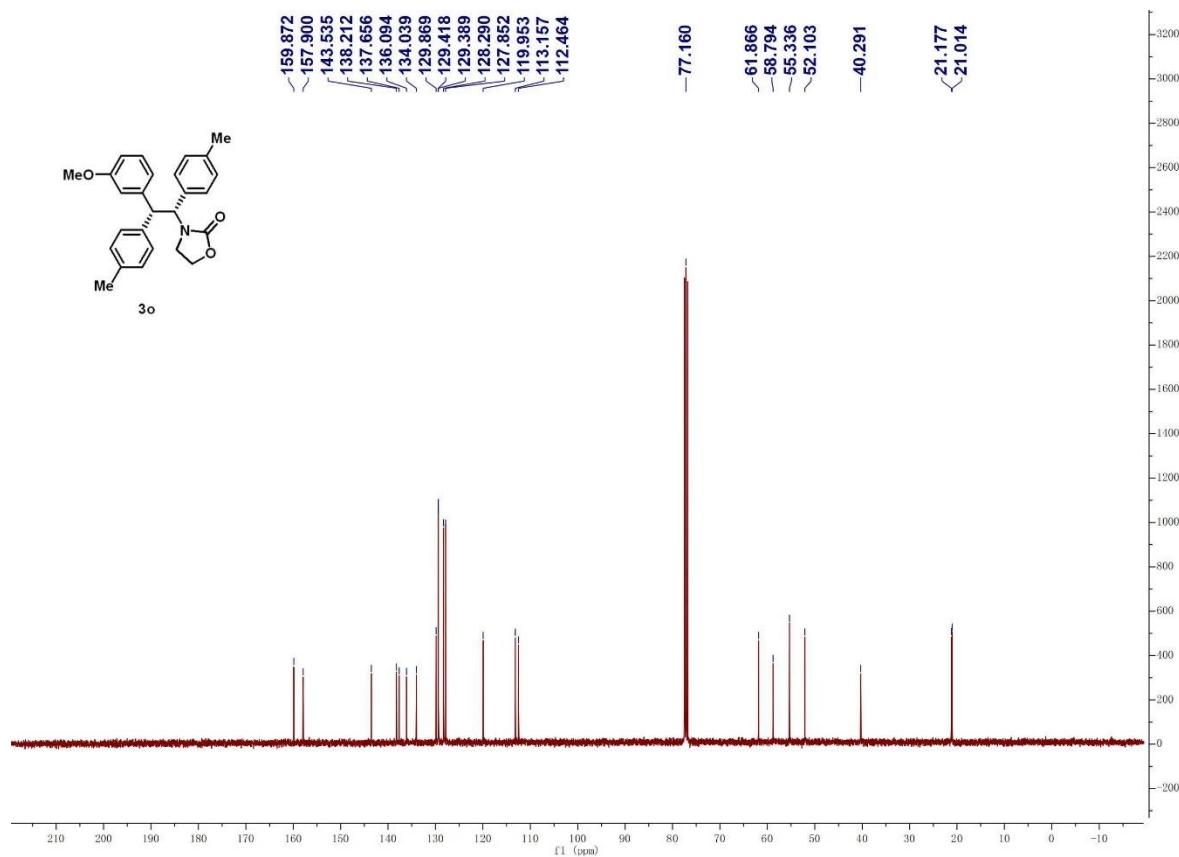
### $^{13}\text{C}$ NMR-spectrum (100 MHz, $\text{CDCl}_3$ ) of **3n**



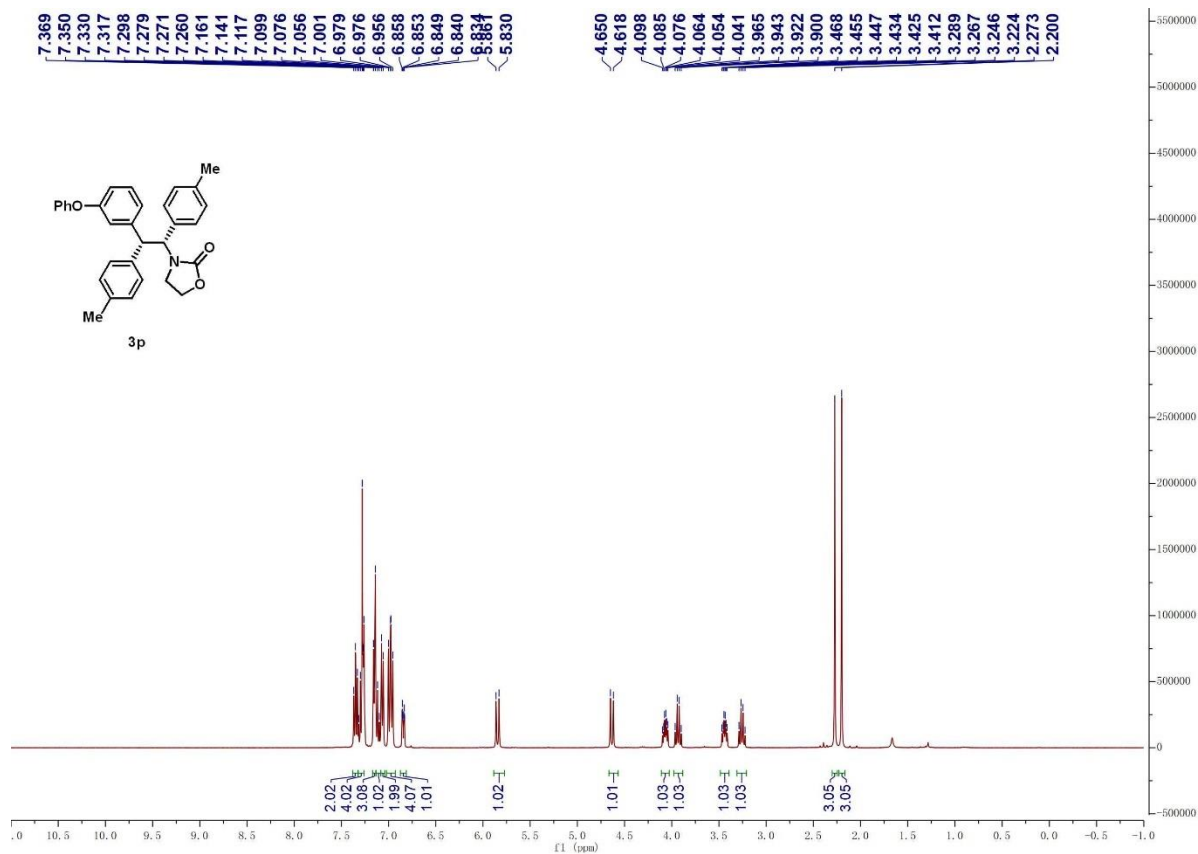
### $^1\text{H}$ NMR-spectrum (400 MHz, $\text{CDCl}_3$ ) of **3o**



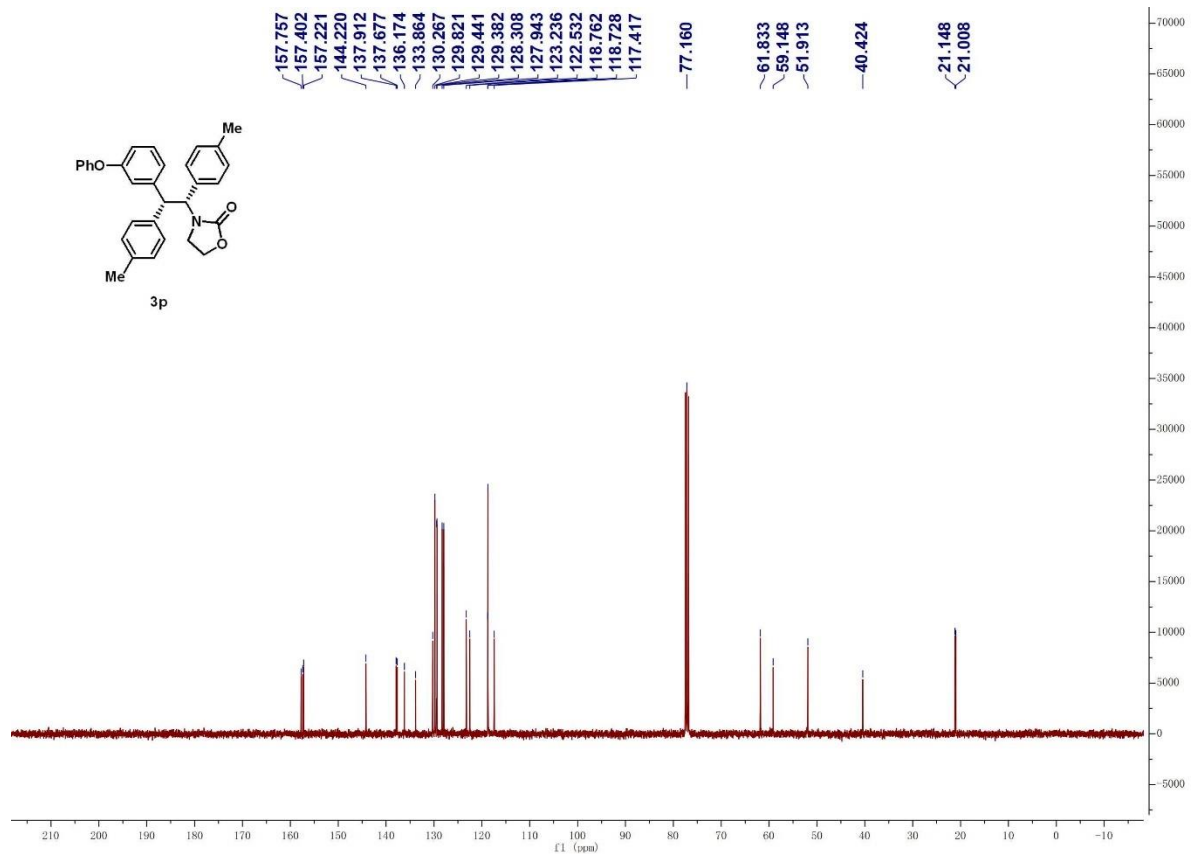
### $^{13}\text{C}$ NMR-spectrum (100 MHz, $\text{CDCl}_3$ ) of **3o**



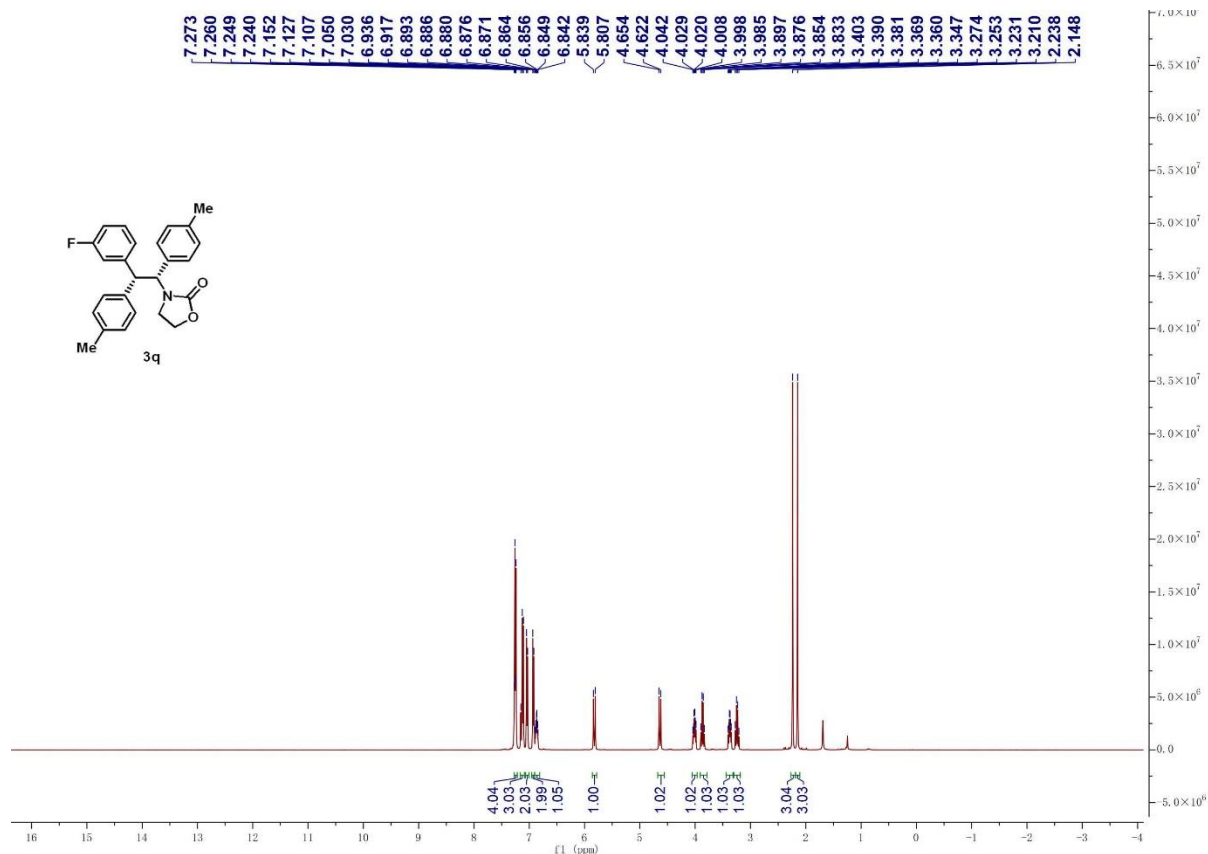
<sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of **3p**



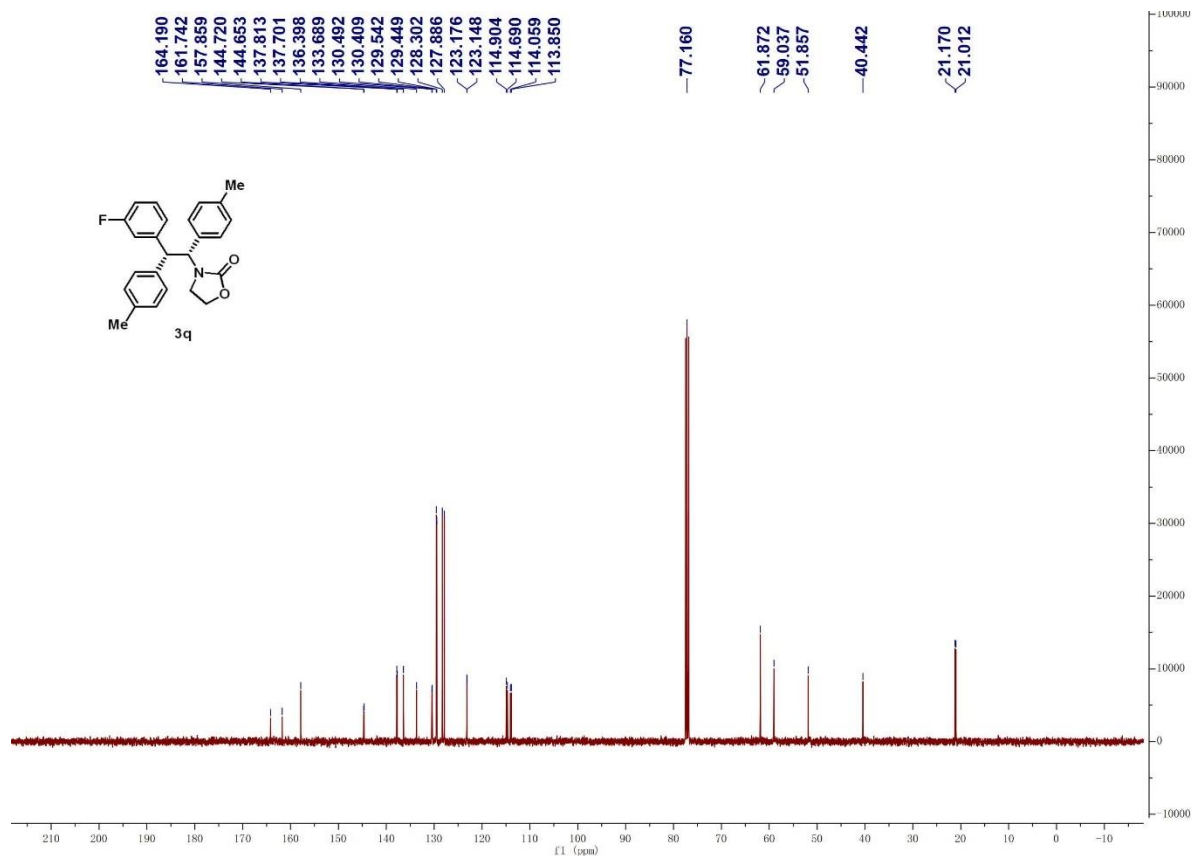
<sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of **3p**



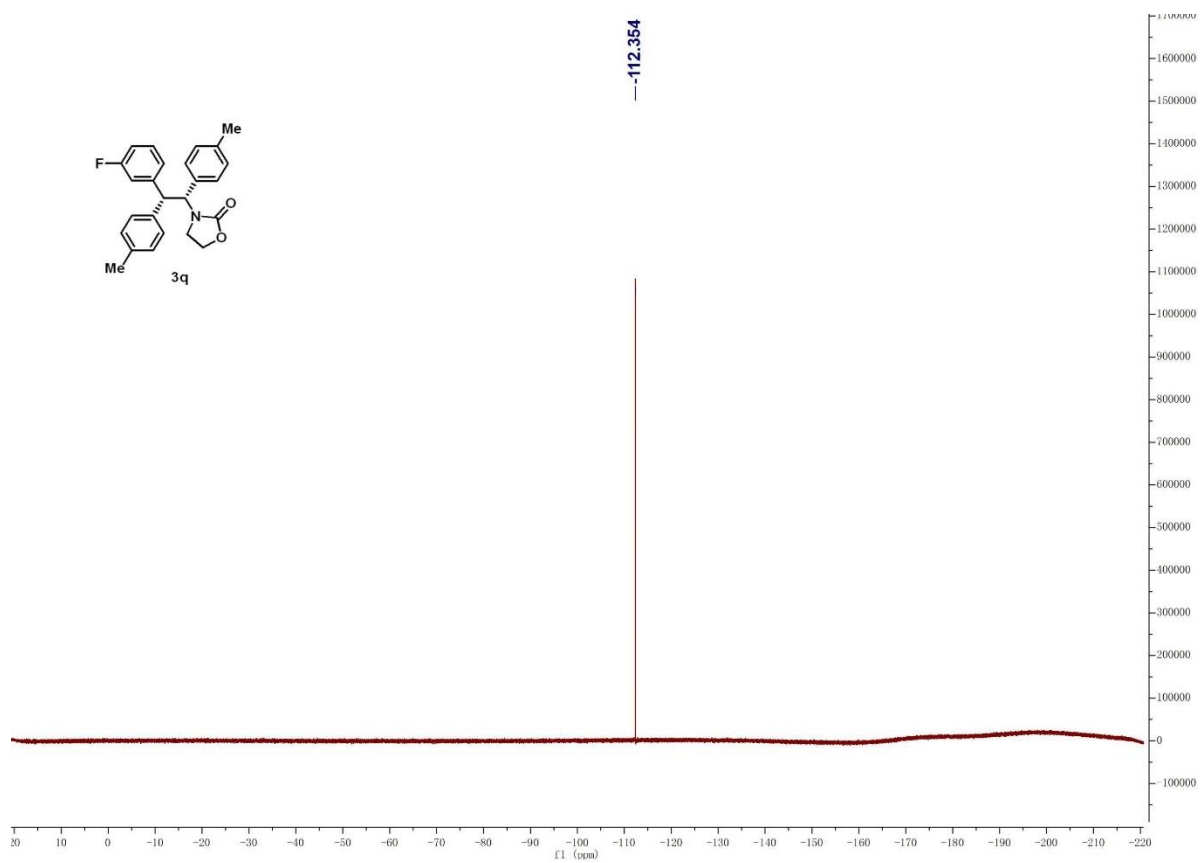
# <sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of **3q**



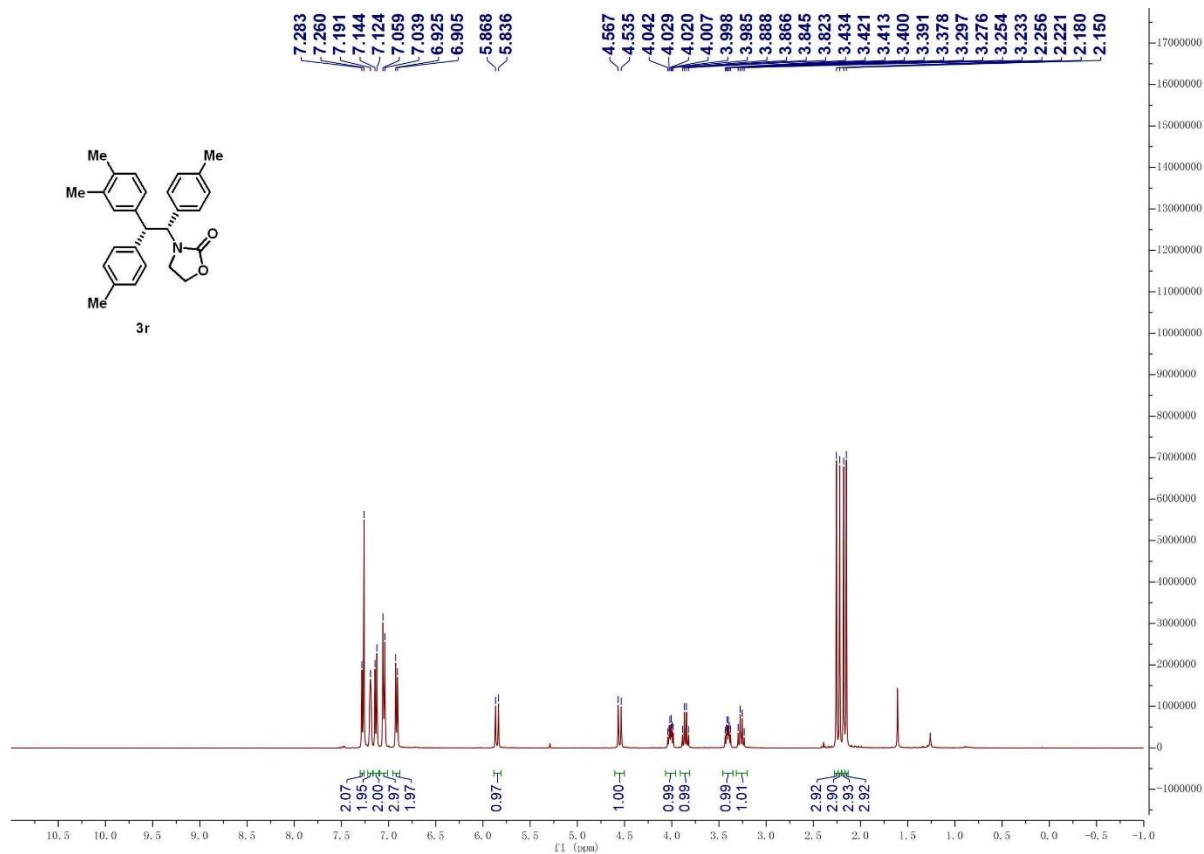
# <sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of **3q**



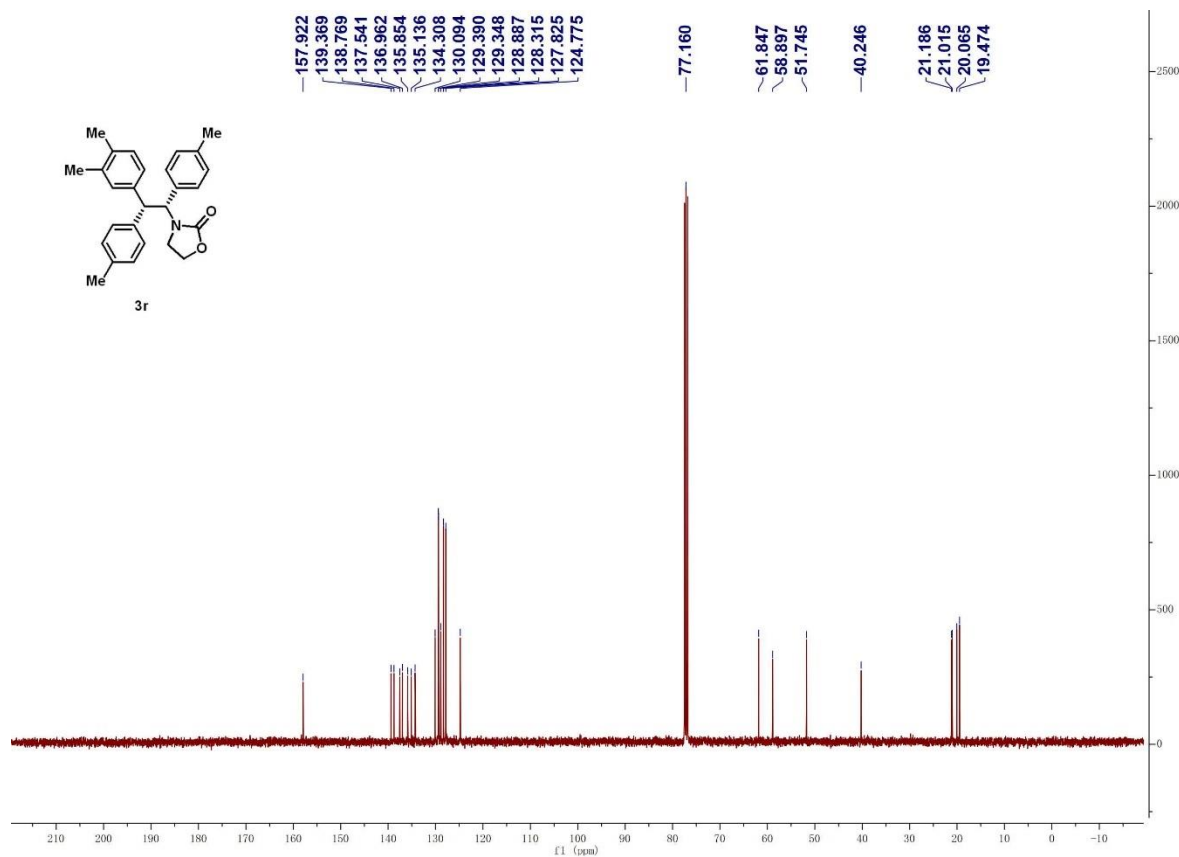
**$^{19}\text{F}$  NMR-spectrum (376 MHz,  $\text{CDCl}_3$ ) of **3q****



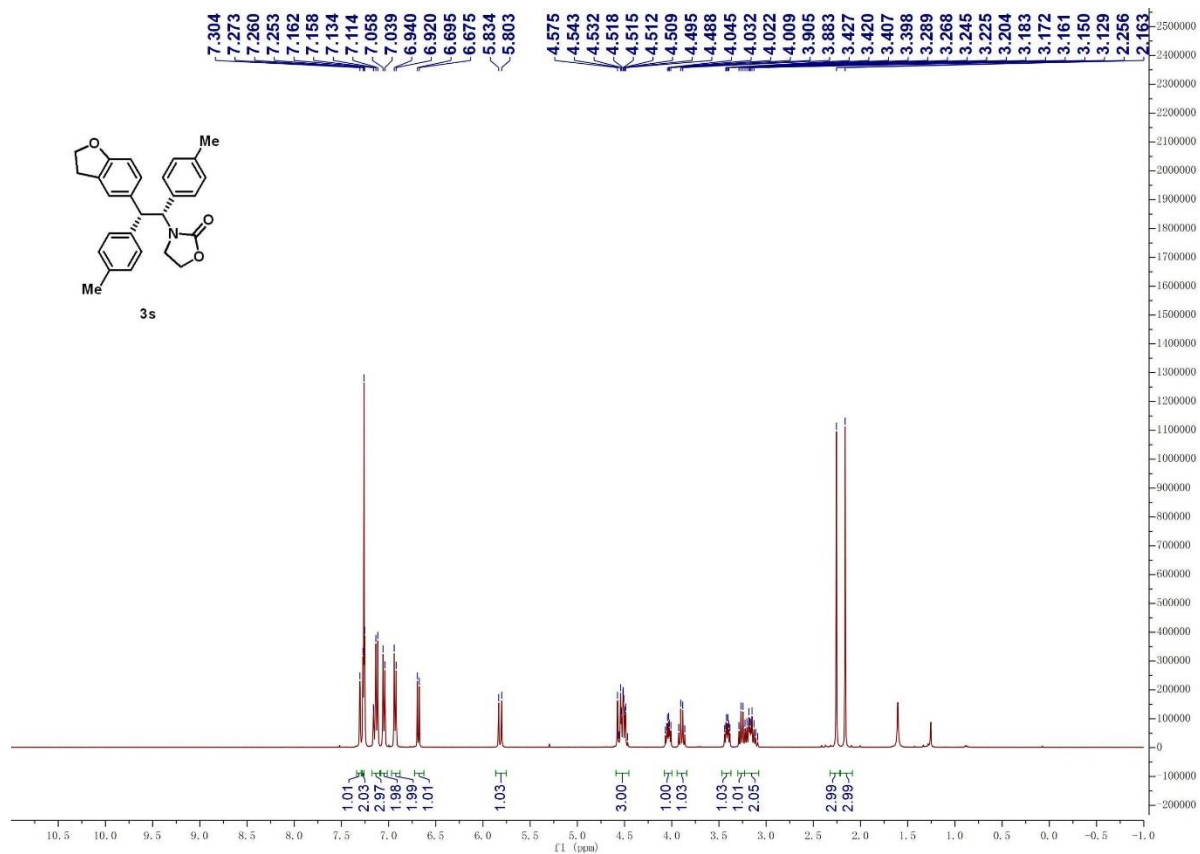
### $^1\text{H}$ NMR-spectrum (400 MHz, $\text{CDCl}_3$ ) of **3r**



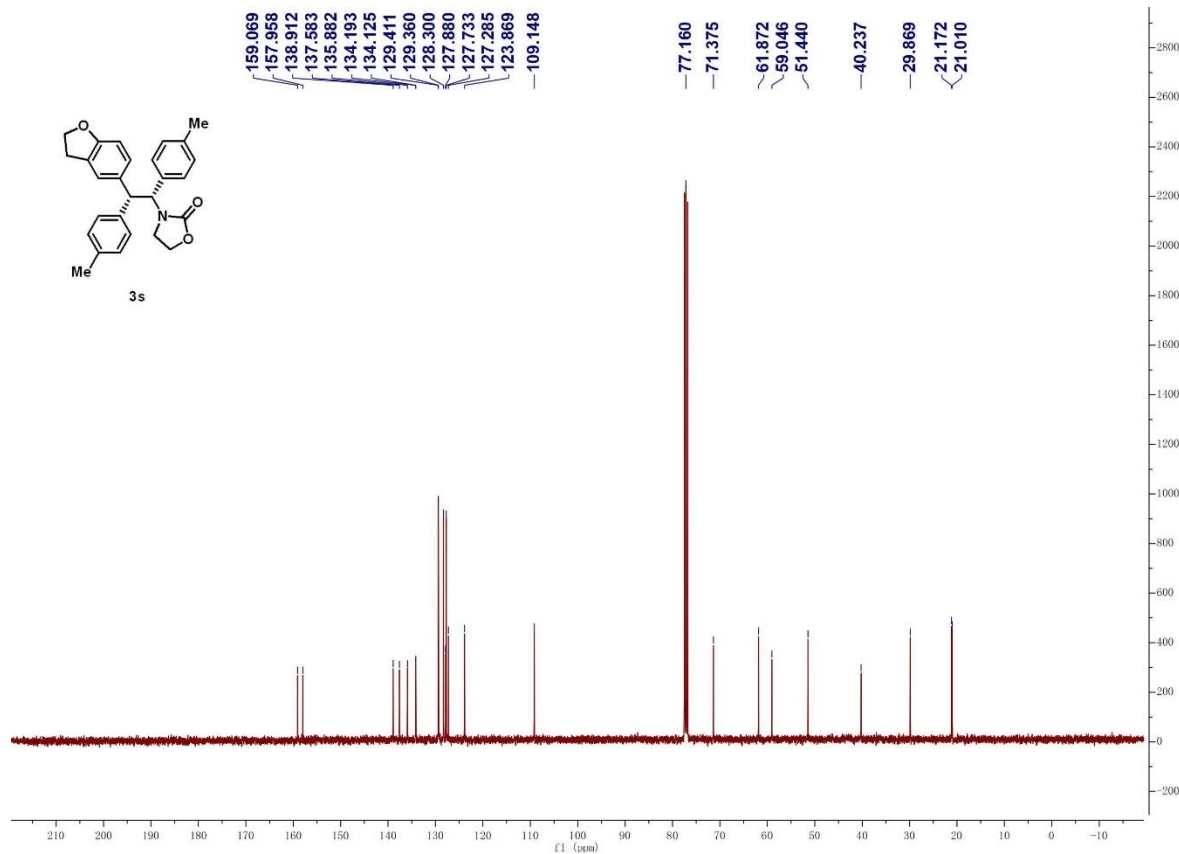
### $^{13}\text{C}$ NMR-spectrum (100 MHz, $\text{CDCl}_3$ ) of **3r**



<sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of **3s**

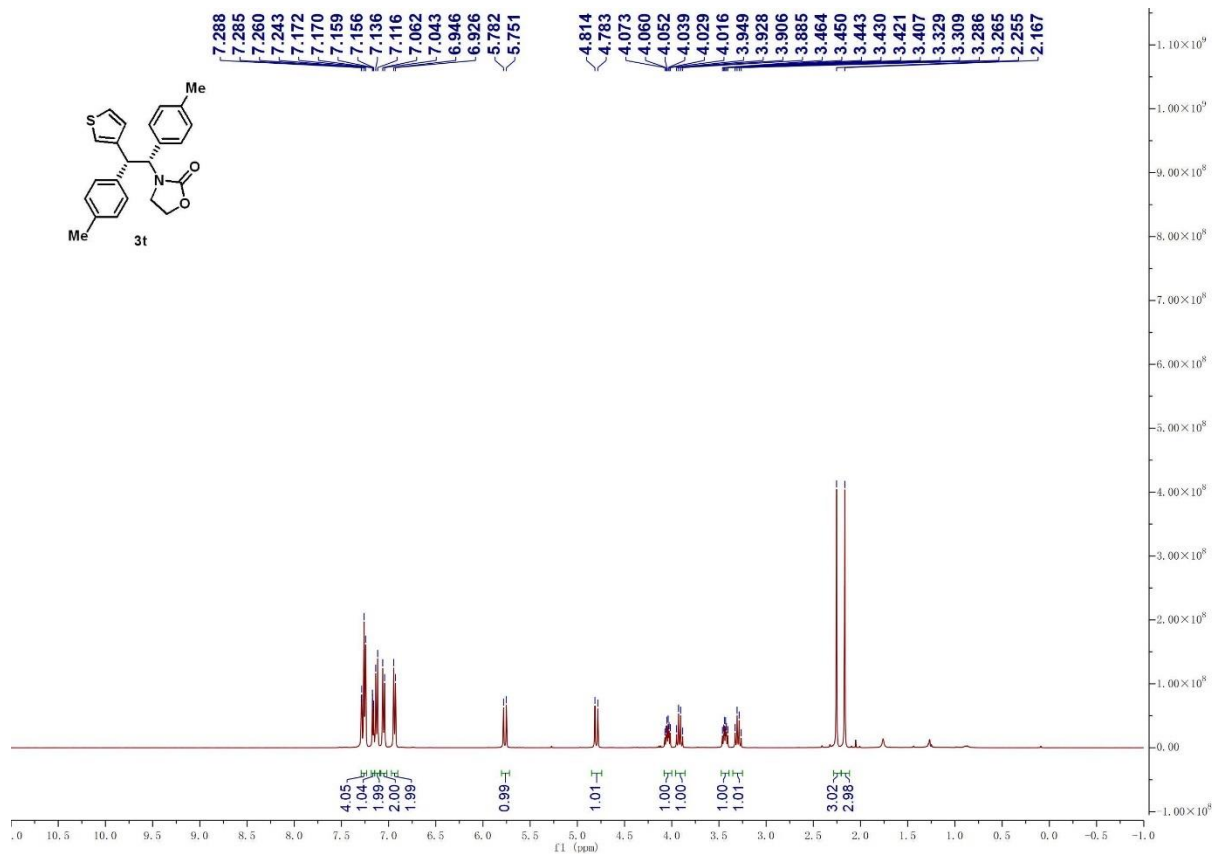


<sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of **3s**

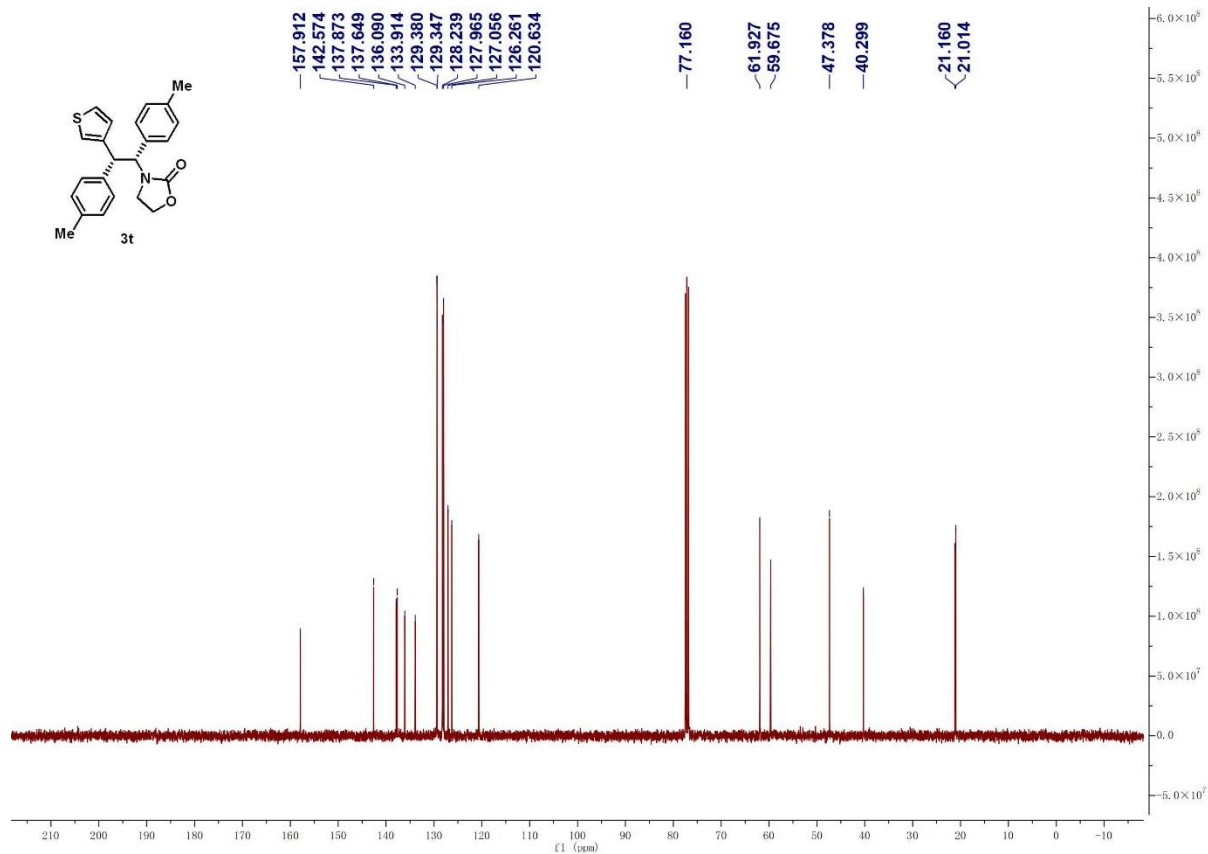




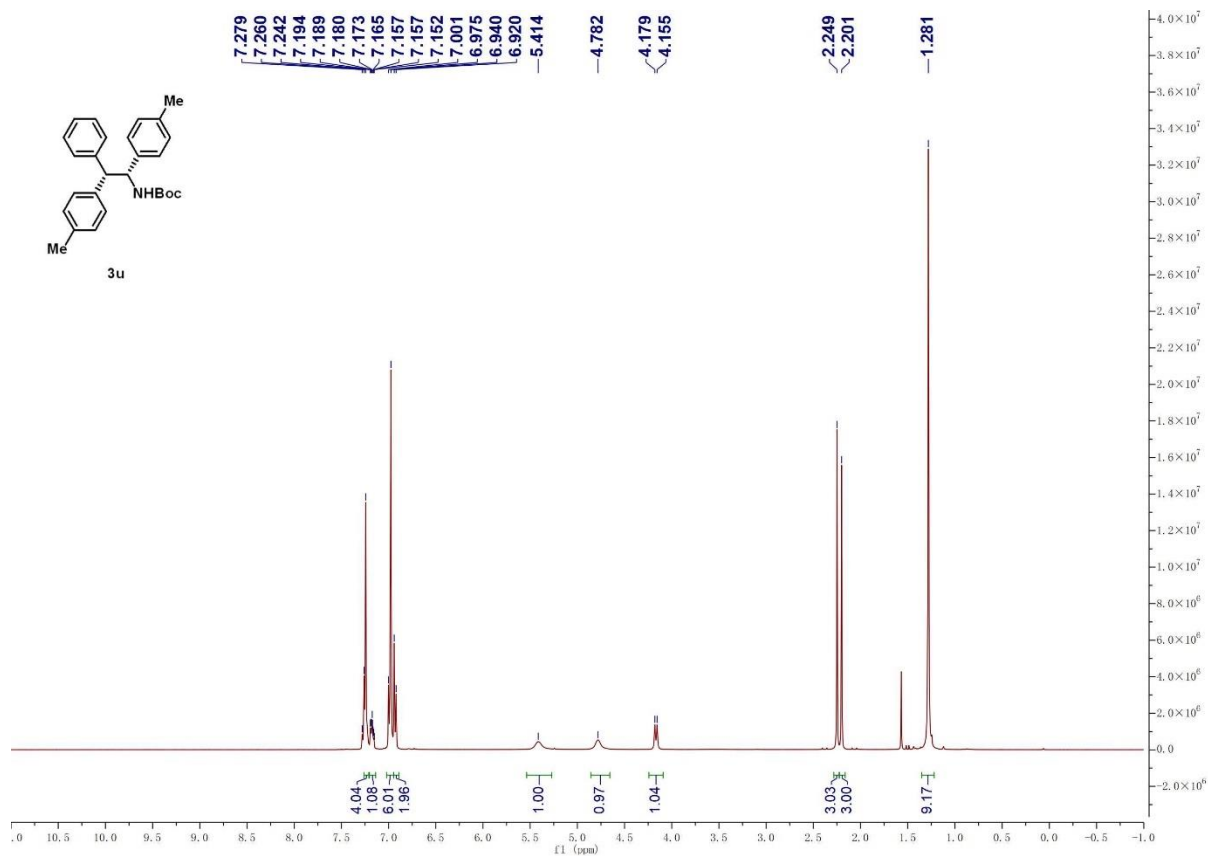
**<sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of 3t**



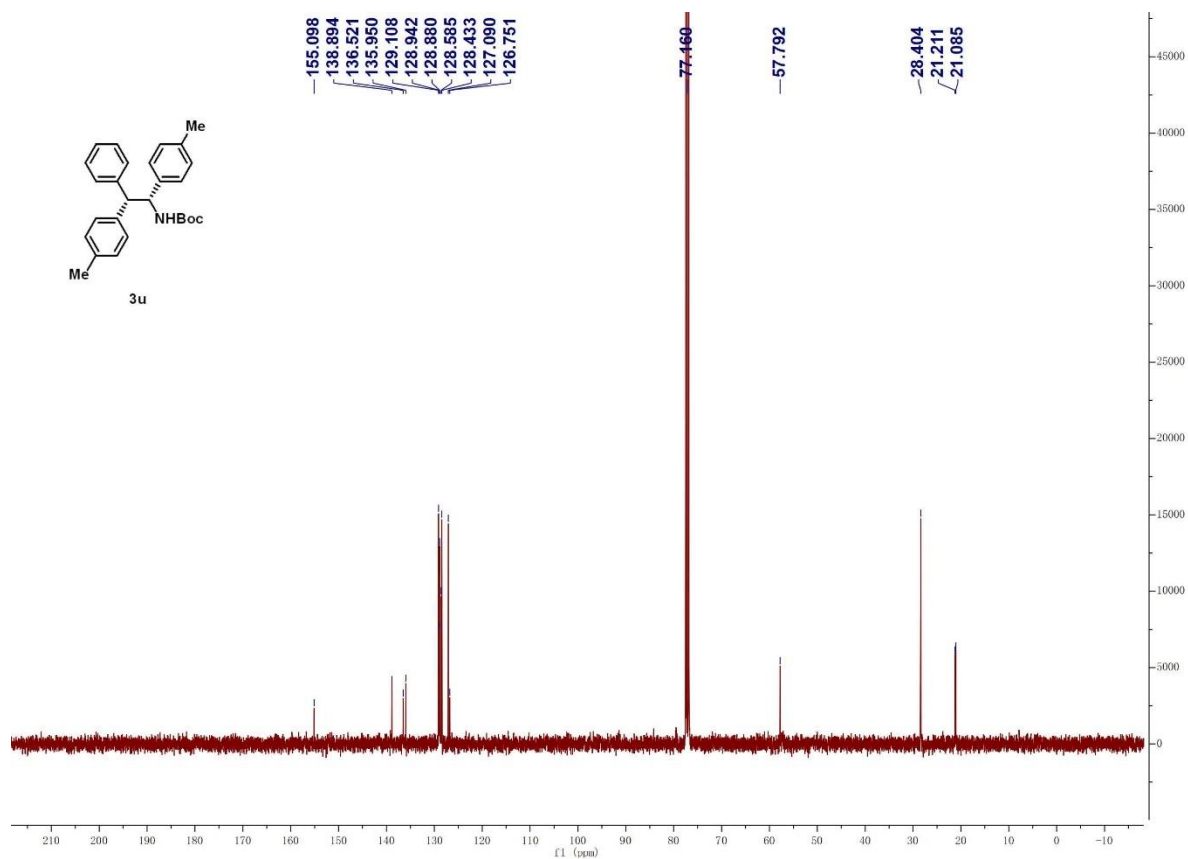
**<sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of 3t**



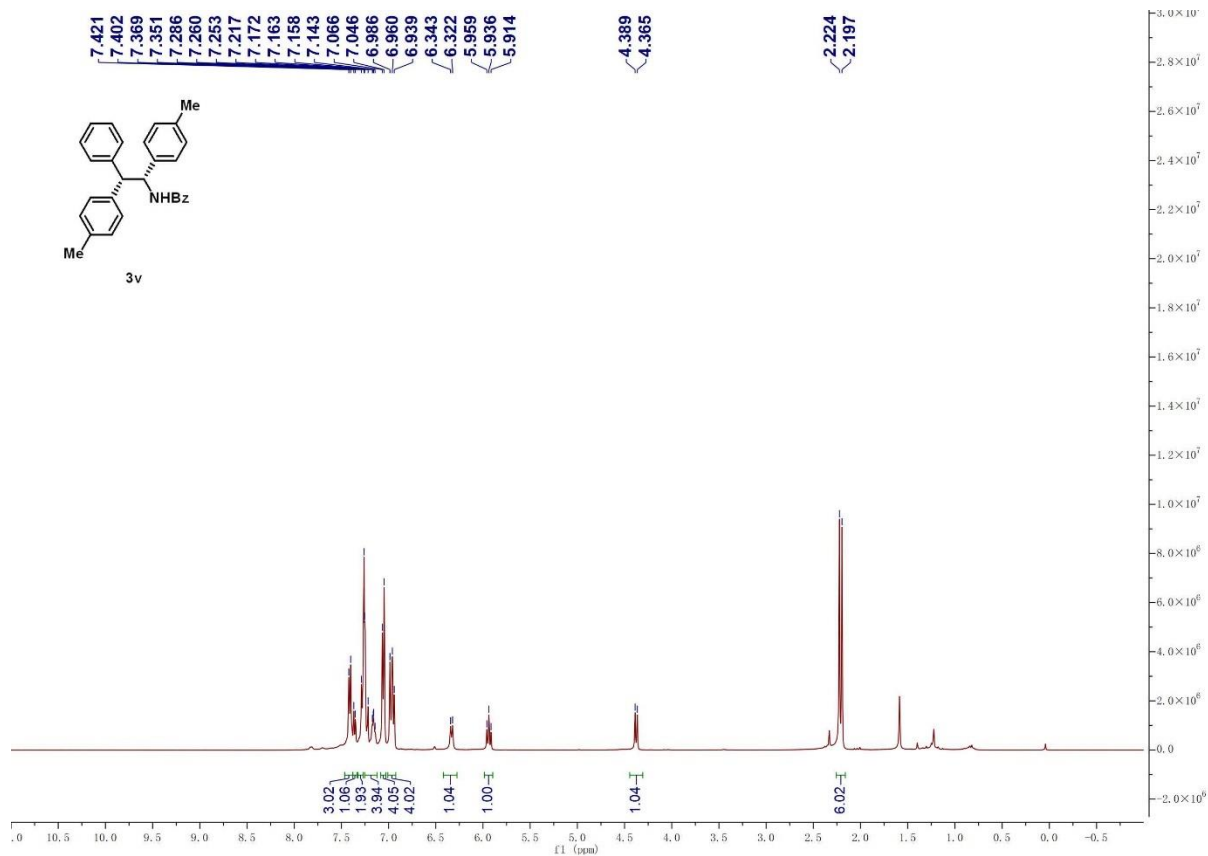
### <sup>1</sup>H NMR-spectrum (400 MHz, CDCl<sub>3</sub>) of **3u**



### <sup>13</sup>C NMR-spectrum (100 MHz, CDCl<sub>3</sub>) of **3u**



### $^1\text{H}$ NMR-spectrum (400 MHz, $\text{CDCl}_3$ ) of **3v**



### $^{13}\text{C}$ NMR-spectrum (100 MHz, $\text{CDCl}_3$ ) of **3v**

