

# **An Highly Enantioselective Synthesis of Multifunctionalized Dihydrofurans by Copper-Catalyzed Asymmetric [4+1] Cycloadditions of $\alpha$ -Benzylidene- $\beta$ -Ketoester with Diazo Compound**

## **Supporting Information**

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## **Contents**

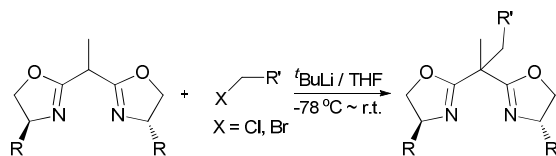
1. General information
2. General Procedure for the Synthesis of Chiral Sidearm Oxazolines
3. General procedure for the Synthesis of Chiral 2, 3-dihydrofuran
4. X-ray Data of **3g**
5.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra
6. HPLC spectra

## 1. General information

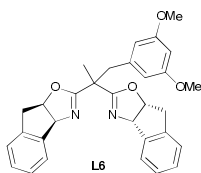
All reactions were carried out under dry nitrogen atmosphere. Dichloromethane (DCM) was distilled over calcium hydride prior to use.  $\text{CuPF}_6(\text{CH}_3\text{CN})_4$ ,  $\text{CuCl}$ , and  $\text{AgSbF}_6$  were purchased from Aldrich, and the diazo esters,<sup>[1]</sup> substrate,<sup>[2]</sup> and ligand<sup>[3]</sup> were prepared according to literature procedures. Powdered M.S.  $4\text{\AA}$  was preactivated for 8 hours at  $250\text{ }^\circ\text{C}$  under vacuum prior to use.

NMR spectra were recorded on a Varian Mercury-300 or a Varian Mercury-400 nuclear magnetic resonance spectrometer. Chemical shifts are reported in parts per million (ppm) down field from TMS, using residual  $\text{CDCl}_3$  as an internal standard.

## 2. General Procedure for the Synthesis of Chiral Sidearm Oxazolines.



To a solution of bisoxazoline (2 mmol) in dried THF (30 mL) was added dropwise *t*-BuLi (1.3 mL, 1.7 M in hexanes, 2.2 mmol) within 15-20 min at  $-78\text{ }^\circ\text{C}$ . The resulting yellow solution was stirred for an additional 1 h at this temperature. Then a solution of halide (2.8 mmol) in THF (10 mL) was added dropwise at  $-78\text{ }^\circ\text{C}$  over 10 min. The solution was slowly warmed to room temperature and was stirred for a further 10 h. The mixture was filtered through a thin layer (40 mm) of silica gel (100-200 mesh). Then concentrated and the residue was purified by flash chromatography that filled with silica gel (400 mesh), and PE (petroleum ether) /Actone 3/1 as eluent.



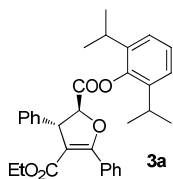
**(3aS,3a'S,8aR,8a'R)-2,2'-(1-(3,5-dimethoxyphenyl)propane-2,2-diyl)bis(8,8a-dihydro-3aH-indeno[1,2-d]oxazole)**

Yield: 45%. bp:  $119\text{--}121\text{ }^\circ\text{C}$ .  $[\alpha]_{\text{D}}^{20} = -302.6\text{ }^\circ$  ( $c = 1.00$ ,  $\text{CHCl}_3$ ).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.50-7.47 (m, 2H), 7.30-7.22 (m, 6H), 6.22 (t,  $J = 2.4\text{ Hz}$ , 1H), 6.18 (d,  $J = 2.4\text{ Hz}$ , 2H), 5.53 (d,  $J$

= 8.0 Hz, 1H), 5.49 (d,  $J$  = 8.0 Hz, 1H), 5.30-5.21 (m, 2H), 3.60 (s, 6H), 3.34-3.24 (m, 3H), 3.07 (d,  $J$  = 13.6 Hz, 1H), 2.88 (ABd,  $J$  = 6.8 Hz,  $J$  = 12.4 Hz, 2H), 1.34 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ): 168.1, 167.5, 160.0, 141.5, 141.5, 139.5, 139.4, 138.5, 128.2, 128.1, 127.2, 127.1, 125.5, 125.4, 124.9, 124.8, 108.1, 98.4, 82.9, 82.8, 76.3, 54.8, 42.7, 41.6, 39.4, 39.3, 20.5; IR (neat) 3024, 2938, 2836, 1648, 1596, 1460, 1429, 1346, 1310, 1294, 1275, 1205, 1152, 1089, 1059, 998, 856, 751, 699; LRMS-ESI ( $m/z$ ): 495.2 ( $\text{M} + \text{H}^+$ ); Anal. Calcd. For  $\text{C}_{31}\text{H}_{30}\text{N}_2\text{O}_4$ : C, 75.28; H, 6.11; N, 5.66; Found: C, 75.13; H, 6.12; N, 5.60.

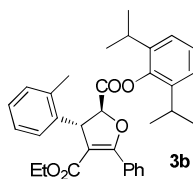
### 3. General procedure for the Synthesis of Chiral 2, 3-dihydrofuran.

$\text{CuCl}$  (2.5 mg, 0.025 mmol),  $\text{AgSbF}_6$  (8.6 mg, 0.025 mmol), **L4** (13 mg, 0.03 mmol), 200 mg M.S. 4Å, and  $\text{CH}_2\text{Cl}_2$  (2 mL) were stirred under nitrogen for 1 hour at room temperature. Then the substrate (0.5 mmol) was added, and the mixture was stirred for another 5 min. Then, a solution of the 2,6-diisopropylphenyl diazoacetate (**2**) (492 mg, 2.0 mmol) in  $\text{CH}_2\text{Cl}_2$  (2.0 mL) was dropwise added through a syringe pump for 9-10 hours. After another 1 h of stirring at room temperature, the mixture was filtered through a thin layer (40 mm) of silica gel (100-200 mesh), and washed with DCM to remove the catalyst. The filtrate was concentrated under reduced pressure, and the residue was purified by flash chromatography.



#### (2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl 3,5-diphenyl-2,3-dihydrofuran-2,4-dicarboxylate

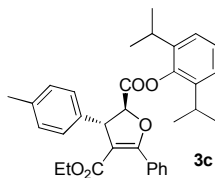
Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 15/1 as eluate. Yield : 82%.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.02 (dd,  $J$  = 1.5 Hz, 8.1 Hz, 2H), 7.47-7.16 (m, 11H), 5.29 (d,  $J$  = 3.3 Hz, 1H), 4.89 (d,  $J$  = 3.6 Hz, 1H), 4.13-3.96 (m, 2H), 2.95 (br, 2H), 1.24-1.06 (m, 15H); The ee of **3a** listed in **table 3** was determined by HPLC analysis using a Chiralpak AD column with Hexane/*i*-PrOH 95/5 as eluent, 314 nm, Flow = 0.6 mL/min, ee = 96%,  $t_r$  (minor) = 7.06 min,  $t_r$  (major) = 18.01 min;  $[\alpha]_{\text{D}}^{20}$  = + 185.8° ( $c$  = 0.97,  $\text{CHCl}_3$ ).



**(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl**

**5-phenyl-3-(o-tolyl)-2,3-dihydrofuran-2,4-dicarboxylate**

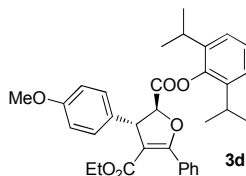
Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 15/1 as eluate. Yield: 71%.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.06 (d,  $J$  = 6.0 Hz, 2H), 7.45-7.16 (m, 10H), 5.19 (dd,  $J$  = 3.0 Hz, 10.2 Hz, 2H), 4.10-3.96 (m, 2H), 2.98 (br, 2H), 2.56 (s, 3H), 1.27-1.04 (m, 15H); The ee of **3b** listed in **table 3** was determined by HPLC analysis using a Chiralpak AD column with Hexane/*i*-PrOH 95/5 as eluent, 314 nm, Flow = 0.6 mL/min, ee = 95%,  $t_r$  (minor) = 5.98 min,  $t_r$  (major) = 11.32 min;  $[\alpha]_D^{20}$  = + 199.1° ( $c$  = 1.02,  $\text{CHCl}_3$ ).



**(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl**

**5-phenyl-3-(p-tolyl)-2,3-dihydrofuran-2,4-dicarboxylate**

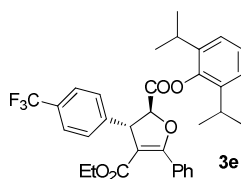
Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 15/1 as eluate. Yield: 89%.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.02 (d,  $J$  = 8.4 Hz, 2H), 7.42-7.15 (m, 10H), 5.28 (d,  $J$  = 3.6 Hz, 1H), 4.87 (d,  $J$  = 3.6 Hz, 1H), 4.13-3.95 (m, 2H), 2.96 (br, 2H), 2.34 (s, 3H), 1.24-1.06 (m, 15H); The ee of **3c** listed in **table 3** was determined by HPLC analysis using a Chiralpak AD column with Hexane/*i*-PrOH 95/5 as eluent, 314 nm, Flow = 0.6 mL/min, ee = 95%,  $t_r$  (minor) = 7.29 min,  $t_r$  (major) = 20.29 min;  $[\alpha]_D^{20}$  = + 332.7° ( $c$  = 1.00,  $\text{CHCl}_3$ ).



**(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl**

**3-(4-methoxyphenyl)-5-phenyl-2,3-dihydrofuran-2,4-dicarboxylate**

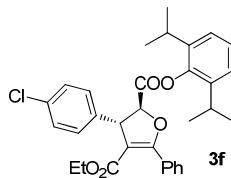
Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 15/1 as eluate. Yield: 85%.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.02 (dd,  $J$  = 0.9 Hz, 6.6 Hz, 2H), 7.43-7.16 (m, 8H), 6.91 (d,  $J$  = 8.4 Hz, 2H), 5.27 (d,  $J$  = 3.9 Hz, 1H), 4.86 (d,  $J$  = 3.6 Hz, 1H), 4.11-3.99 (m, 2H), 3.77 (s, 3H), 2.96 (br, 2H), 1.24-1.07 (m, 15H); The ee of **3d** listed in **table 3** was determined by HPLC analysis using a Chiralpak AD column with Hexane/*i*-PrOH 97/3 as eluent, 314 nm, Flow = 0.6 mL/min, ee = 90%,  $t_r$  (minor) = 10.02 min,  $t_r$  (major) = 25.71 min;  $[\alpha]_D^{20}$  = + 186.2  $^\circ$  ( $c$  = 1.06,  $\text{CHCl}_3$ ).



**(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl**

**5-phenyl-3-(4-(trifluoromethyl)phenyl)-2,3-dihydrofuran-2,4-dicarboxylate**

Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 15/1 as eluate. Yield: 82%.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.04 (d,  $J$  = 6.3 Hz, 2H), 7.67 (d,  $J$  = 8.1 Hz, 2H), 7.56 (d,  $J$  = 8.1 Hz, 2H), 7.47-7.43 (m, 3H), 7.24-7.17 (m, 3H), 5.29 (d,  $J$  = 3.0 Hz, 1H), 4.99 (d,  $J$  = 3.6 Hz, 1H), 4.14-3.97 (m, 2H), 2.95 (br, 2H), 1.25-1.06 (m, 15H); The ee of **3e** listed in **table 3** was determined by HPLC analysis using a Chiralpak AD column with Hexane/*i*-PrOH 95/5 as eluent, 314 nm, Flow = 0.6 mL/min, ee = 90%,  $t_r$  (minor) = 6.51 min,  $t_r$  (major) = 11.69 min;  $[\alpha]_D^{20}$  = + 175.3  $^\circ$  ( $c$  = 1.01,  $\text{CHCl}_3$ ).

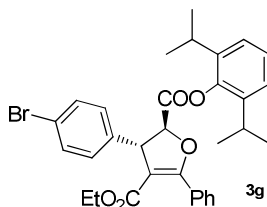


**(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl**

**3-(4-chlorophenyl)-5-phenyl-2,3-dihydrofuran-2,4-dicarboxylate**

Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 15/1 as eluate. Yield: 93%.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.03 (d,  $J$  = 6.3 Hz, 2H), 7.44-7.17 (m, 10H), 5.25 (d,  $J$  = 3.3 Hz, 1H), 4.87 (d,  $J$  = 3.3 Hz, 1H), 4.11-3.99 (m, 2H), 2.94 (br, 2H), 1.24-1.06 (m, 15H);

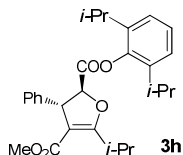
The ee of **3f** listed in **table 3** was determined by HPLC analysis using a Chiralpak AD column with Hexane/*i*-PrOH 95/5 as eluent, 314 nm, Flow = 0.6 mL/min, ee = 96%,  $t_r$  (minor) = 7.20 min,  $t_r$  (major) = 19.38 min;  $[\alpha]_D^{20} = +205.3^\circ$  ( $c = 0.99$ ,  $\text{CHCl}_3$ ).



**(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl**

**3-(4-bromophenyl)-5-phenyl-2,3-dihydrofuran-2,4-dicarboxylate**

Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 15/1 as eluate. Yield: 81%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.01-7.98 (m, 2H), 7.53-7.42 (m, 5H), 7.30-7.17 (m, 5H), 5.23 (d,  $J = 3.2$  Hz, 1H), 4.83 (d,  $J = 3.6$  Hz, 1H), 4.12-3.99 (m, 2H), 2.91 (br, 2H), 1.23-1.08 (m, 15H); The ee of **3g** listed in **table 3** was determined by HPLC analysis using a Chiralpak AD column with Hexane/*i*-PrOH 95/5 as eluent, 314 nm, Flow = 0.6 mL/min, ee = 96%,  $t_r$  (minor) = 7.40 min,  $t_r$  (major) = 19.04 min;  $[\alpha]_D^{20} = +193.5^\circ$  ( $c = 1.01$ ,  $\text{CHCl}_3$ ).

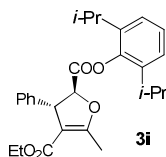


**(2S,3S)-2-(2,6-diisopropylphenyl) 4-methyl**

**5-isopropyl-3-phenyl-2,3-dihydrofuran-2,4-dicarboxylate**

Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 15/1 as eluate. Yield: 79%.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.39-7.17 (m, 8H), 5.12 (d,  $J = 3.9$  Hz, 1H), 4.63 (d,  $J = 3$  Hz, 1H), 3.80 (hep,  $J = 6.3$  Hz, 1H), 3.60 (s, 3H), 2.91 (hep,  $J = 6.3$  Hz, 2H), 1.34-1.17 (m, 18H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  176.5, 168.9, 165.0, 145.0, 142.2, 140.1, 128.8, 127.4, 126.9, 124.0, 104.4, 85.6, 52.6, 50.9, 27.5, 27.0, 23.4, 19.7, 19.3; IR (neat) 3183, 3065, 3029, 2966, 2872, 1820, 1757, 1709, 1643, 1495, 1468, 1385, 1347, 1306, 1257, 1163, 1114, 1067, 1035, 936, 867, 840, 792, 756, 729, 699, 608; LRMS-ESI ( $m/z$ ): 451.3 ( $\text{M}+\text{H}^+$ ); HRMS-ESI calcd. For  $\text{C}_{28}\text{H}_{35}\text{O}_5^+$  is 451.2484; Found: 451.2475; The ee of **3h** listed in **table 3** was determined by HPLC

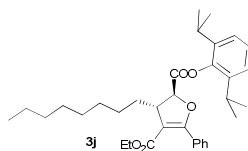
analysis using a Chiralpak OD-H column with Hexane/*i*-PrOH 97/3 as eluent, 235 nm, Flow = 0.5 mL/min, ee = 89%,  $t_r$  (major) = 7.59 min,  $t_r$  (minor) = 9.71 min;  $[\alpha]_D^{20} = +207.6^\circ$  (c = 1.07, CHCl<sub>3</sub>).



**(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl**

**5-methyl-3-phenyl-2,3-dihydrofuran-2,4-dicarboxylate**

Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 15/1 as eluate. Yield: 65%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 7.38-7.16 (m, 8H), 5.17 (d, *J* = 4.2 Hz, 1H), 4.67 (d, *J* = 4.2 Hz, 1H), 4.09 (q, *J* = 7.5 Hz, 2H), 2.89 (br, 2H), 2.45 (s, 3H), 1.23-1.09 (m, 15H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 168.7, 168.4, 164.7, 145.0, 142.1, 140.1, 128.7, 127.4, 127.1, 126.9, 124.0, 106.5, 85.7, 59.6, 52.7, 27.6, 22.9, 14.1; IR (neat) 3030, 2965, 2929, 2871, 1774, 1705, 1654, 1456, 1383, 1313, 1250, 1164, 1094, 1029, 938, 841, 783, 757, 700; LRMS-ESI (*m/z*): 437 (M + H<sup>+</sup>); HRMS-ESI calcd. For C<sub>27</sub>H<sub>32</sub>NaO<sub>5</sub><sup>+</sup> is 459.2147; Found: 459.2143; The ee of **3i** listed in **table 3** was determined by HPLC analysis using a Chiralpak AD column with Hexane/*i*-PrOH 98/2 as eluent, 254 nm, Flow = 0.4 mL/min, ee = 90%,  $t_r$  (minor) = 10.61 min,  $t_r$  (major) = 11.73 min;  $[\alpha]_D^{20} = +247.1^\circ$  (c = 1.07, CHCl<sub>3</sub>).

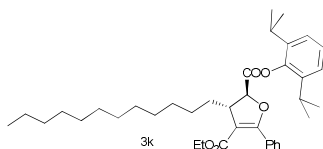


**(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl**

**3-octyl-5-phenyl-2,3-dihydrofuran-2,4-dicarboxylate**

Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 20/1 as eluate. Yield: 68%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 7.89-7.87 (m, 2H), 7.43-7.35 (m, 3H), 7.23-7.15 (m, 3H), 5.09 (d, *J* = 3.3 Hz, 1H), 4.22-4.12 (m, 2H), 3.73 (tt, *J* = 3.6 Hz, *J* = 8.4 Hz, 1H), 2.91 (br, 2H), 2.00-1.91 (m, 1H), 1.80-1.14 (m, 29H), 0.88-0.86 (m, 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 169.7, 164.9, 164.2, 145.1, 140.1, 130.7, 129.7, 129.3, 127.5, 126.8, 123.9, 106.4, 82.0, 59.8, 48.9,

33.9, 31.7, 29.6, 29.2, 29.1, 27.5, 26.2, 23.6, 22.6, 14.1, 14.0; IR (neat): 2962, 2927, 2856, 1754, 1692, 1629, 1447, 1374, 1258, 1219, 1163, 1142, 1084, 1061, 1029, 793, 762, 728, 693; LRMS-EI ( $m/z$ ):  $m/z$  (% relative intensity): 315 ( $M^+$ , 100.00), 343 ( $M^+$ , 38.38); Anal. Calcd. For:  $C_{34}H_{46}O_5$ : C: 76.37, H: 8.67, Found: C: 76.20, H: 8.48; The ee of **3j** listed in **table 3** was determined by HPLC analysis using a Chiralpak AD-H column with Hexane/*i*-PrOH 93/7 as eluent, 320 nm, Flow = 0.7 mL/min, ee = 81%,  $t_r$  (minor) = 5.27 min,  $t_r$  (major) = 8.03 min;  $[\alpha]_D^{20} = +70.0^\circ$  (c = 0.915,  $CHCl_3$ ).



### (2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl

### 3-dodecyl-5-phenyl-2,3-dihydrofuran-2,4-dicarboxylate

Purified by flash chromatography that filled with silica gel (400 mesh), PE/EtOAc = 20/1 as eluate. Yield: 74%.  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  7.88-8.86 (m, 2H), 7.44-7.38 (m, 3H), 7.24-7.16 (m, 3H), 5.08 (d,  $J = 3.0$  Hz, 1H), 4.22-4.12 (m, 2H), 3.71 (tt,  $J = 3.0$  Hz,  $J = 9.0$  Hz), 2.89 (br, 2H), 2.00-1.90 (m, 1H), 1.77-1.57 (m, 1H), 1.33-1.17 (m, 37 H), 0.90-0.85 (m, 3H);  $^{13}C$  NMR (75 MHz,  $CDCl_3$ ):  $\delta$  169.7, 164.9, 164.2, 145.1, 140.1, 130.7, 129.8, 129.3, 127.5, 126.8, 124.0, 106.5, 82.0, 59.8, 49.0, 33.9, 31.9, 31.4, 30.1, 29.6, 29.6, 29.5, 29.3, 27.5, 26.2, 23.7, 22.6, 14.15, 14.08; IR (neat): 3064, 2962, 2927, 2856, 1755, 1692, 1630, 1493, 1462, 1373, 1313, 1217, 1163, 1143, 1083, 1062, 1030, 966, 848, 827, 792, 762, 727, 693; LRMS-ESI ( $m/z$ ):  $m/z$  (% relative intensity): 315 ( $M^+$ , 100.00), 343 ( $M^+$ , 41.08), 475 ( $M^+$ , 14.57); Anal. Calcd. For:  $C_{38}H_{54}O_5$ : C: 77.25, H: 9.21, Found: C: 77.24, H: 9.07; The ee of **3k** listed in **table 3** was determined by HPLC analysis using a Chiralpak AD-H column with Hexane/*i*-PrOH 95/5 as eluent, 320 nm, Flow = 0.7 mL/min, ee = 78 %,  $t_r$  (minor) = 5.23 min,  $t_r$  (major) = 8.43 min;  $[\alpha]_D^{20} = +63.0^\circ$  (c = 0.978,  $CHCl_3$ ).

### References:

- [1] S. Son and G. C. Fu, *J. Am. Chem. Soc.* **2007**, *129*, 1046.
- [2] J.-L. Zhou, Y. Liang, C. Deng, H.-L. Zhou, Z. Wang, X.-L. Sun, J.-C. Zheng, Z.-X. Yu and Y. Tang, *Angew. Chem. Int. Ed.*, **2011**, *50*, 7874.
- [3] a) J. Li, S.-H. Liao, H. Xiong, Y.-Y. Zhou, X.-L. Sun, Y. Zhang, X.-G. Zhou, and Y. Tang,

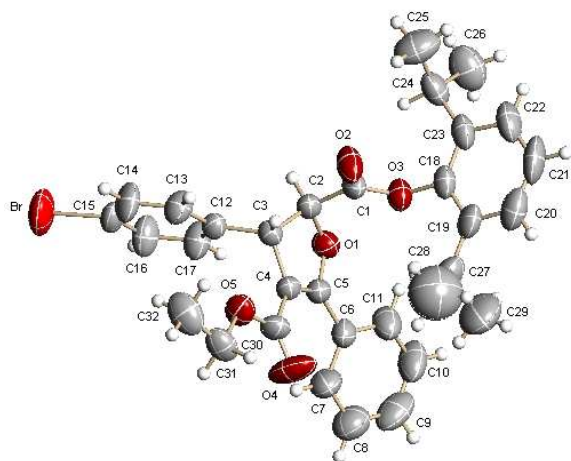


*Angew chem. Int. Ed.* **2012**, *51*, 8838; b) J.-H. Chen, S.-H. Liao, X.-L. Sun, Q. Shen, and Y. Tang,

*Tetrahedron*, **2012**, *68*, 5042.

#### 4. X-ray Data of **3g**

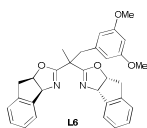
Crystal of **3g** suitable for X-ray crystallographic analysis was obtained by recrystallization from petrol ether/DCM. The ORTEP drawing of **3g** is shown in Figure 1 (deposition number: CCDC 915948).



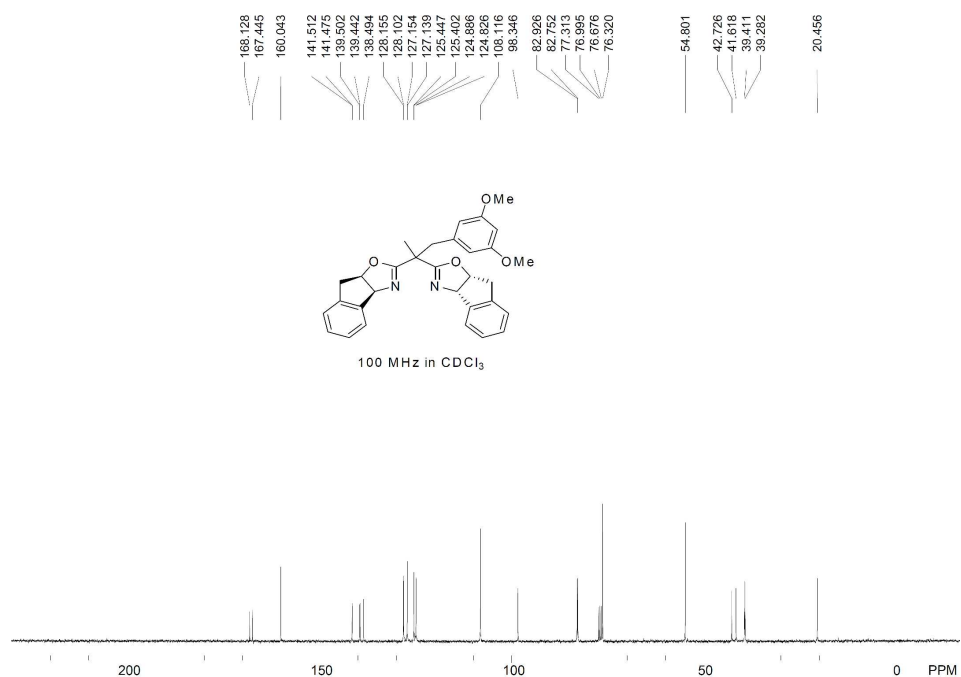
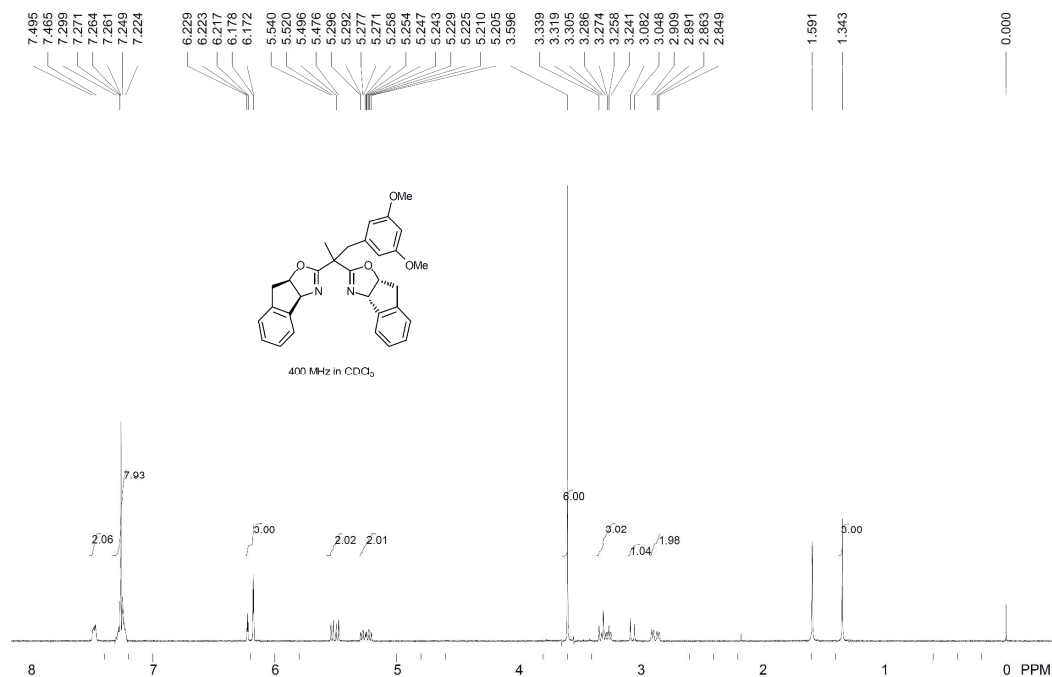
Temperature	293 (2) K
Radiation	MoK $\alpha$
$\lambda$ (Å)	0.71073
Reflections collected/unique	18158/6845 [(int) = 0.1015]
Refinement method	Full-matrix least-squares on F <sup>2</sup>
$\theta$ range (°)	1.81–27.50
Formula	C <sub>32</sub> H <sub>33</sub> BrO <sub>5</sub>
Formula weight	577.49
Crystal size (mm)	0.369 x 0.357 x 0.231 mm
Crystal system	Orthorhombic
Space group	P2 (1) 2 (1) 2 (1)
a (Å)	11.8375 (15)
b (Å)	15.0508 (19)
c (Å)	16.968 (2)
v (Å <sup>3</sup> )	3023.0 (7)
$\alpha$ (°)	90
$\beta$ (°)	90
$\gamma$ (°)	90
Z	4
dcalc (g/cm <sup>3</sup> )	1.269
Representative bonds: (Å)	
C(2)–O(1)	1.436(4)

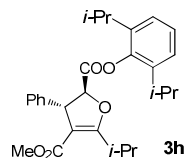
C(2)-C(1)	1.521(5)
C(2)-C(3)	1.522(5)
C(3)-C(12)	1.527(5)
C(3)-C(4)	1.519(5)
C(4)-C(30)	1.452(5)
C(4)-C(5)	1.363(5)
C(5)-C(6)	1.458(5)
C(5)-O(1)	1.361(4)
C(3)-H(3)	0.9800
C(2)-H(2)	0.9800

## 5. $^1\text{H}$ and $^{13}\text{C}$ NMR spectra



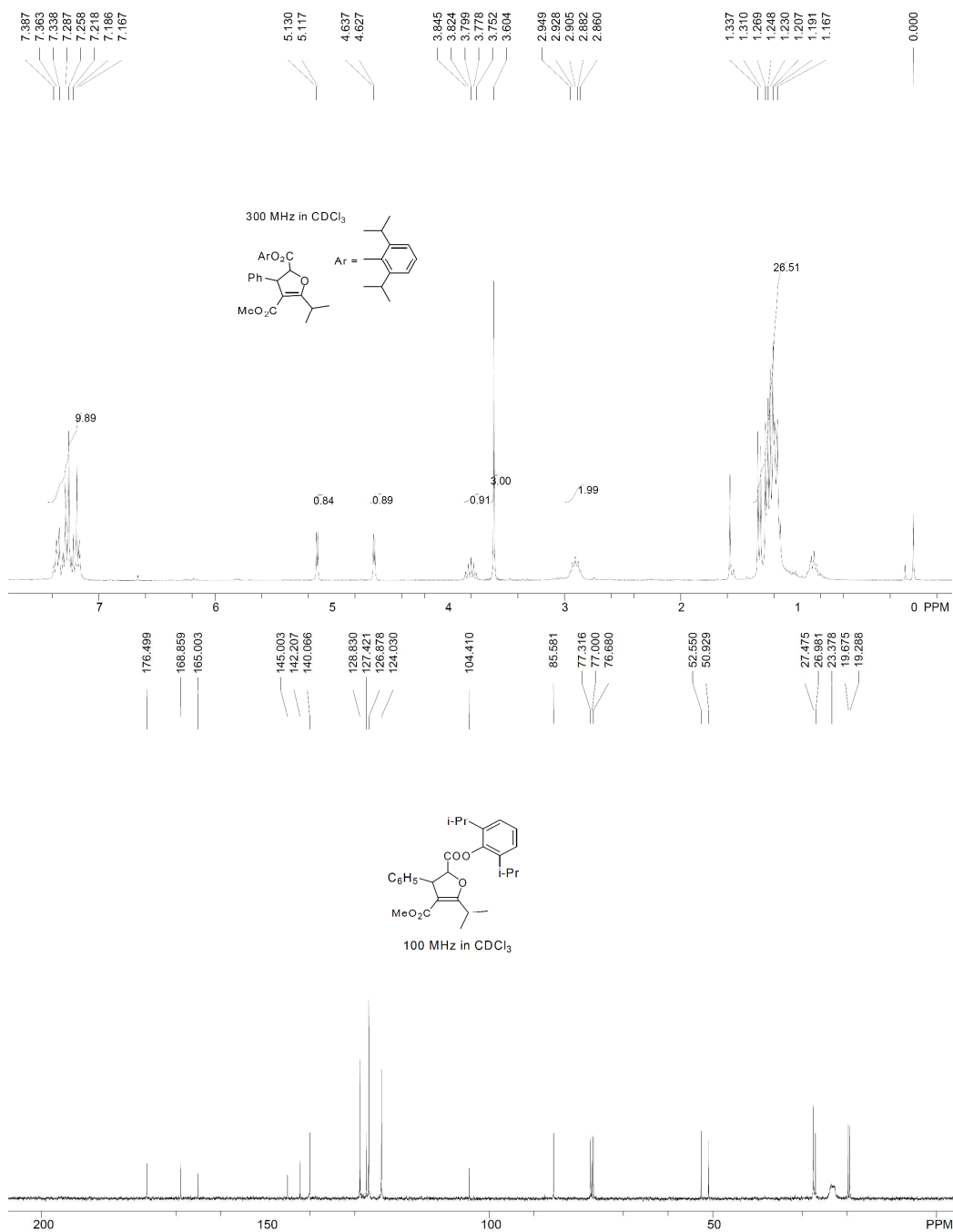
**(3aS,3a'S,8aR,8a'R)-2,2'-(1-(3,5-dimethoxyphenyl)propane-2,2-diyl)bis(8,8a-dihydro-3aH-indeno[1,2-d]oxazole)**

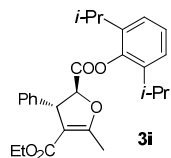




**(2S,3S)-2-(2,6-diisopropylphenyl) 4-methyl**

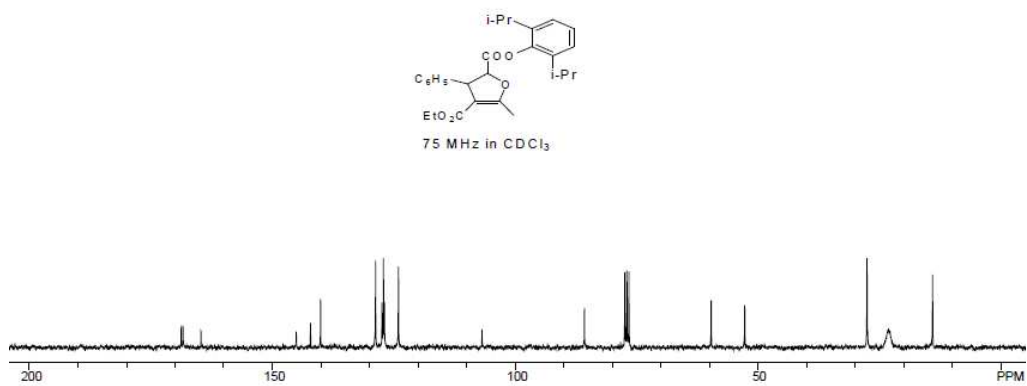
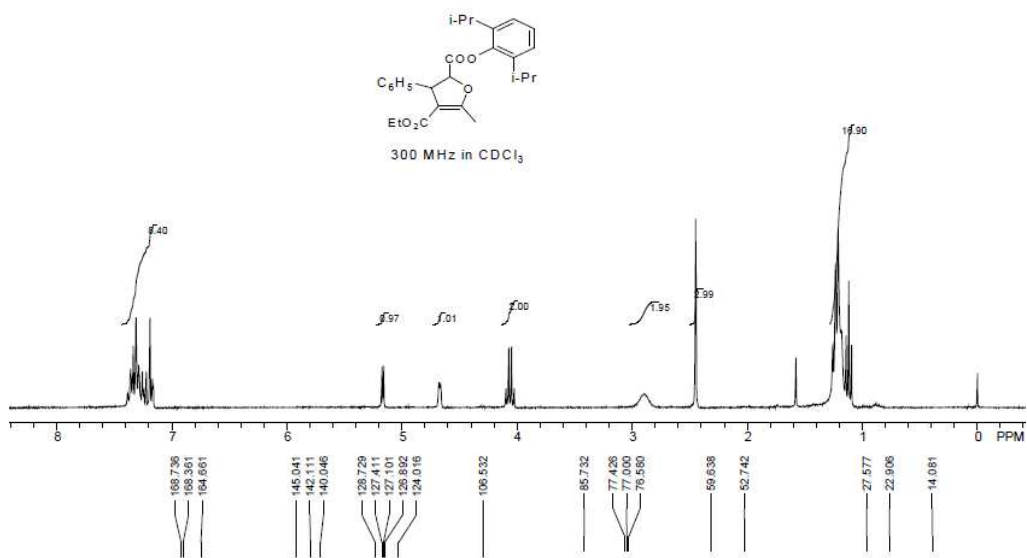
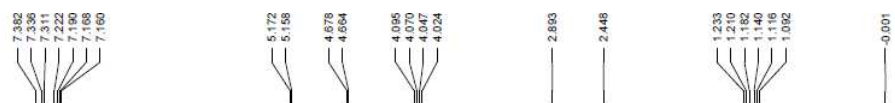
**5-isopropyl-3-phenyl-2,3-dihydrofuran-2,4-dicarboxylate**

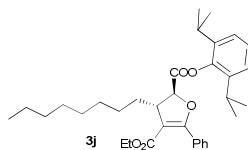




**(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl**

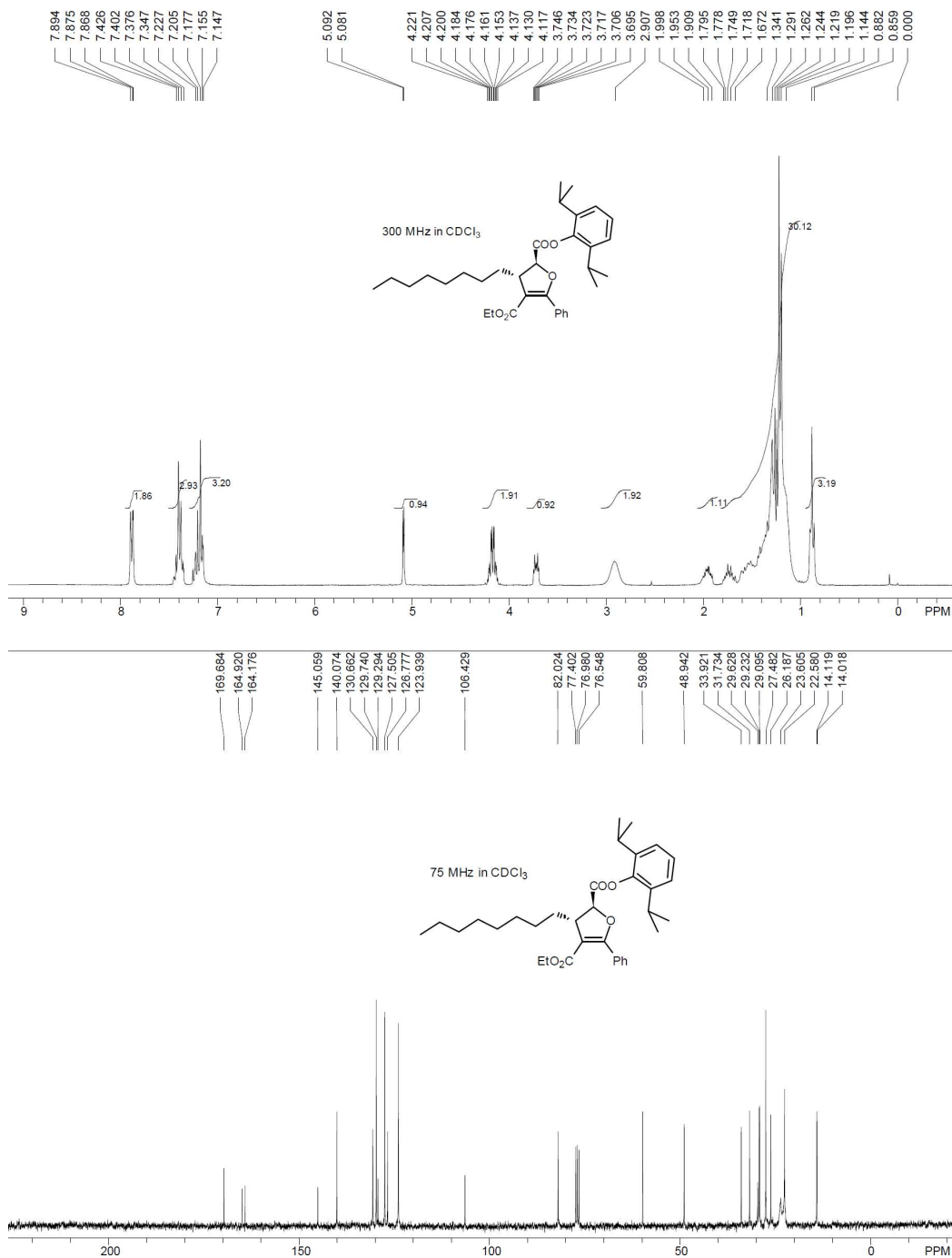
**5-methyl-3-phenyl-2,3-dihydrofuran-2,4-dicarboxylate**

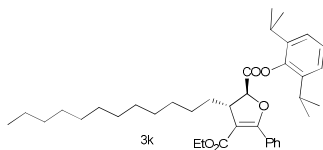




**(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl**

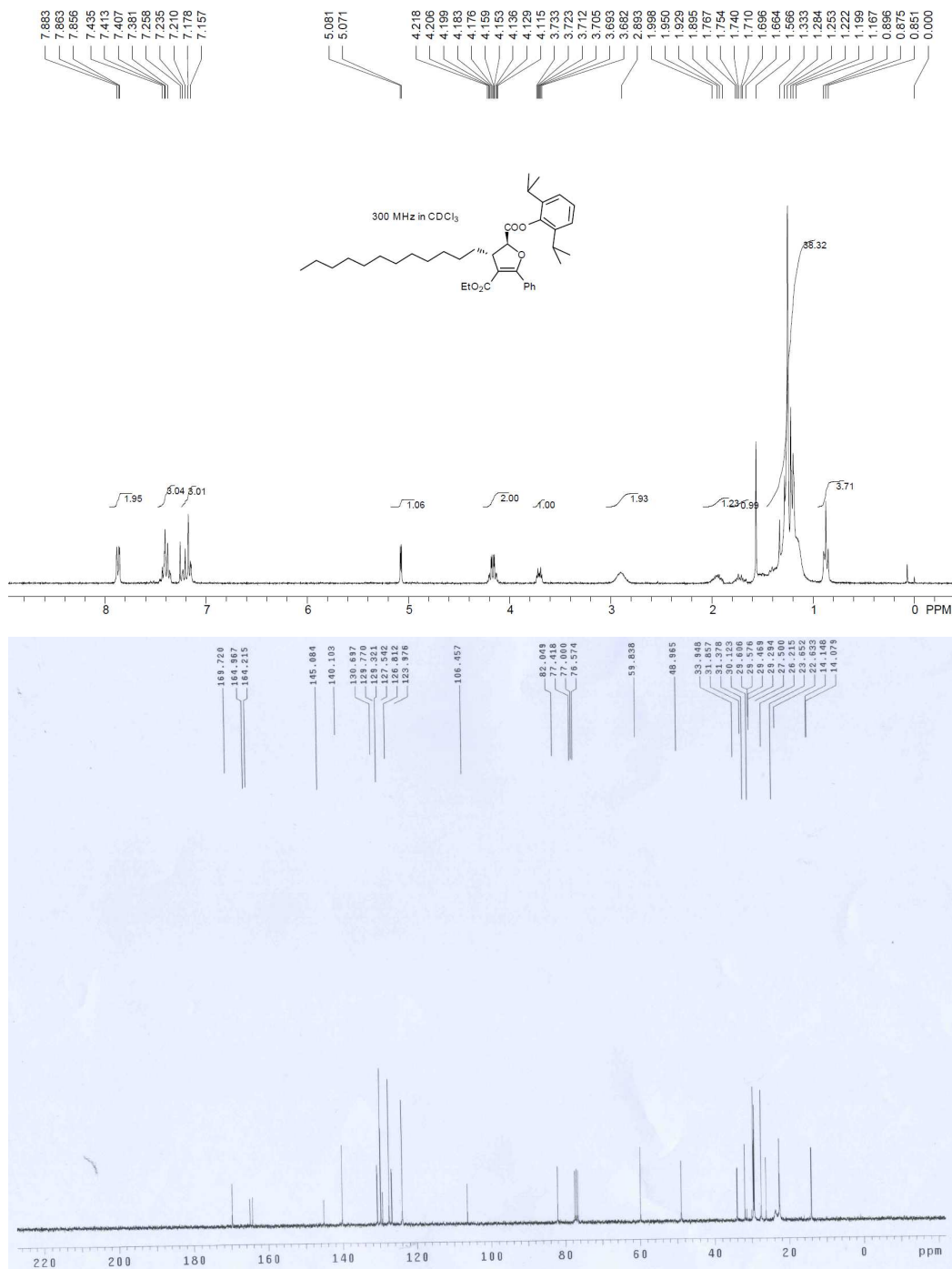
**3-octyl-5-phenyl-2,3-dihydrofuran-2,4-dicarboxylate**



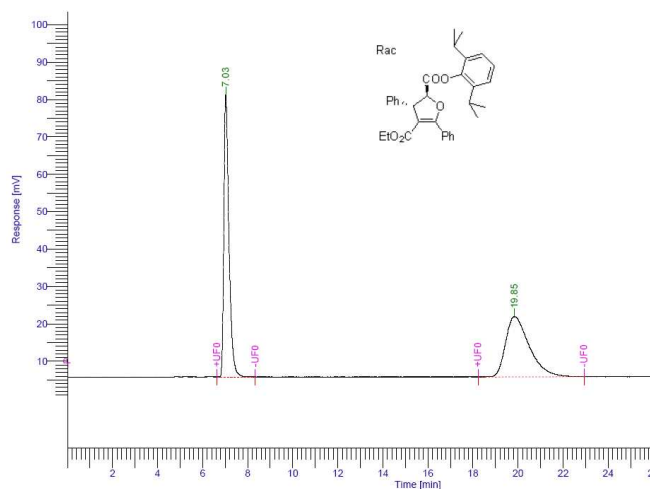
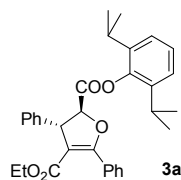


(2S,3S)-2-(2,6-diisopropylphenyl) 4-ethyl

3-dodecyl-5-phenyl-2,3-dihydrofuran-2,4-dicarboxylate



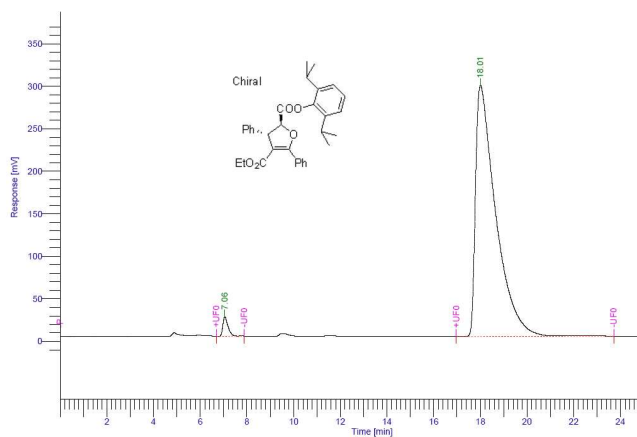
## 6. HPLC spectra



### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	7.03	1183485.7777	75496.7122	50.13	50.13
2	19.85	1177501.3369	16144.4718	49.87	49.87
		2360987.1146	91641.1840	100.00	100.00

AD column, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm, 25 oC

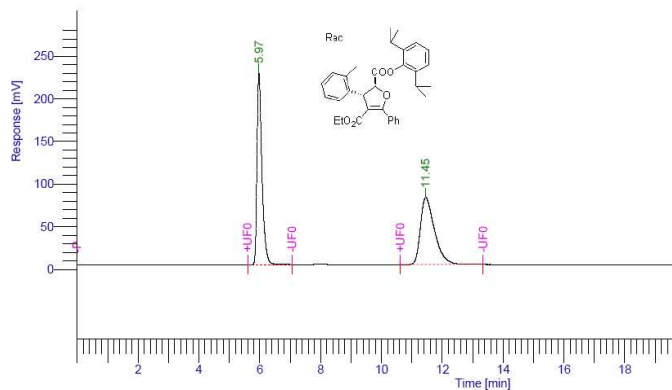
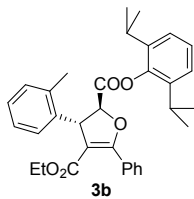


### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	7.06	348847.7419	22836.6238	1.93	1.93
2	18.01	17690570.7018	295245.4500	98.07	98.07
		18039418.4437	318082.0738	100.00	100.00

AD column, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm, 25 oC



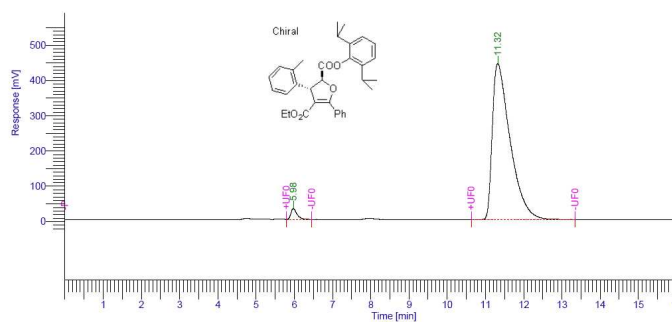


## Jiaolong Zhou HPLC Report

zjl

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	5.97	2.5237e+06	2.243e+05	50.02	50.02
2	11.45	2.5217e+06	7.858e+04	49.98	49.98
		5.0453e+06	3.029e+05	100.00	100.00

zjl: AD, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm

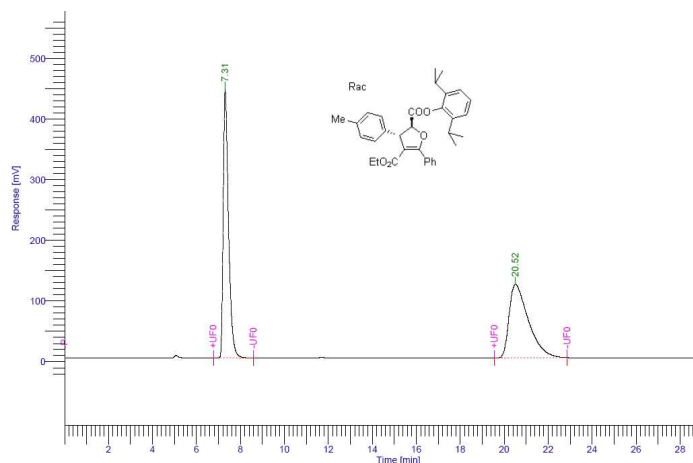
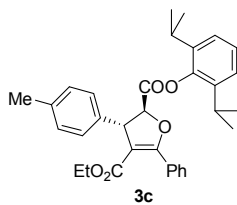


## Jiaolong Zhou HPLC Report

zjl

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	5.98	337700.5983	3.055e+04	2.30	2.30
2	11.32	1.4335e+07	4.420e+05	97.70	97.70
		1.4672e+07	4.725e+05	100.00	100.00

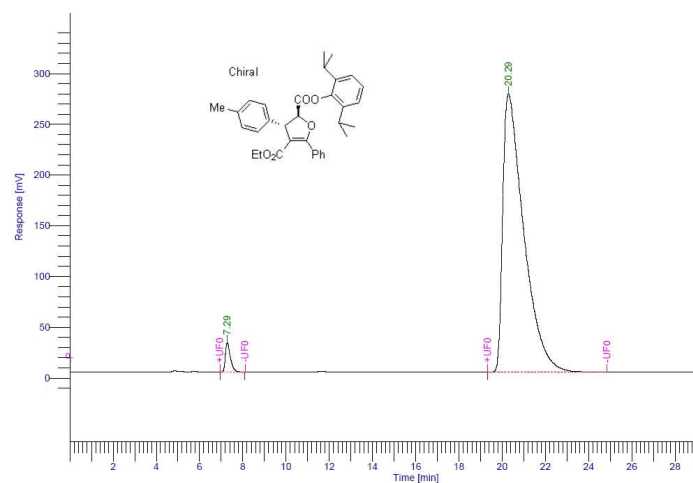
zjl: AD, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm



### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	7.31	7395780.8240	443041.9357	50.03	50.03
2	20.52	7387389.9865	121272.8503	49.97	49.97
		14783170.8105	564314.7860	100.00	100.00

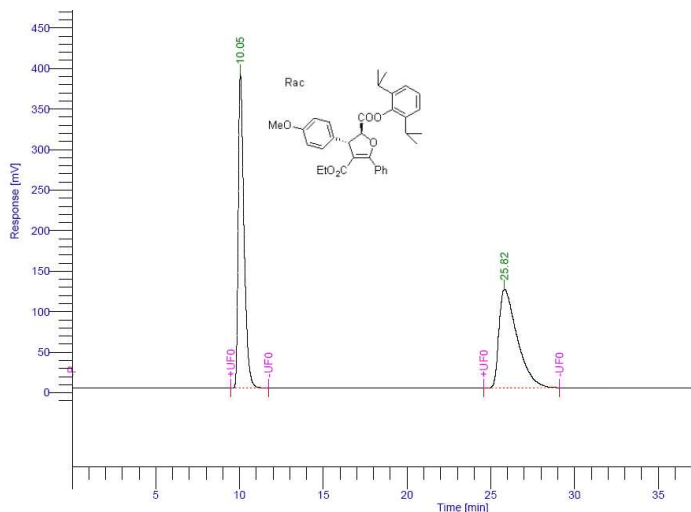
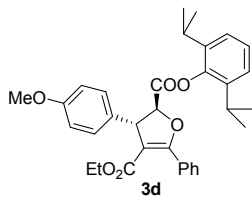
AD column, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm, 25 °C



### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	7.29	470165.0039	29012.2974	2.48	2.48
2	20.29	18468065.1247	273997.5033	97.52	97.52
		18938230.1286	303009.8008	100.00	100.00

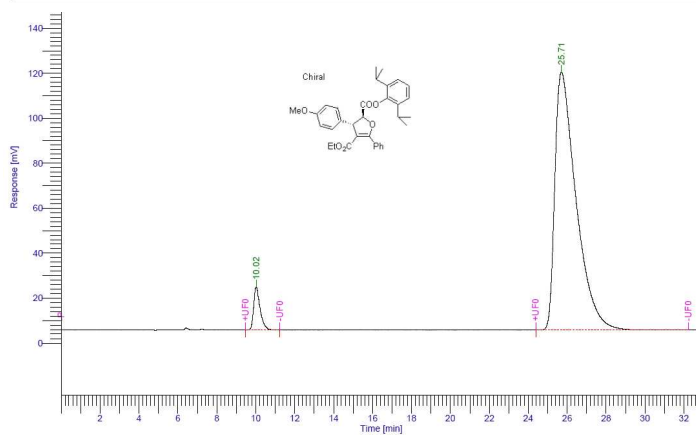
AD column, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm, 25 °C



### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	10.05	9211558.9533	388126.2180	49.91	49.91
2	25.82	9245798.6013	122153.8330	50.09	50.09
		18457357.5546	510280.0510	100.00	100.00

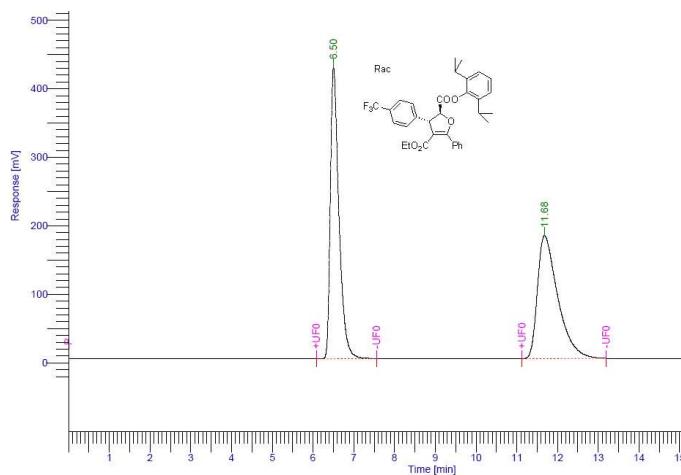
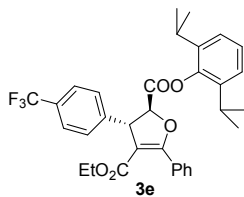
AD column, Hexane/iPrOH 97/3, 0.6 mL/min, 314 nm, 25 °C



### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	10.02	443391.7552	19143.4808	4.87	4.87
2	25.71	8669986.4924	114583.1057	95.13	95.13
		9113378.2475	133726.5865	100.00	100.00

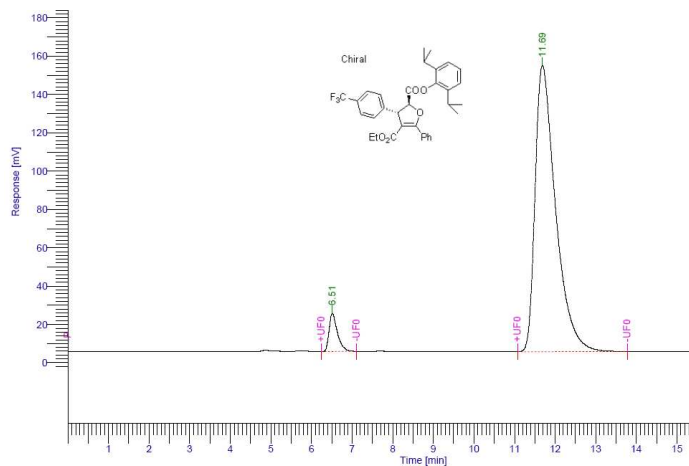
AD column, Hexane/iPrOH 97/3, 0.6 mL/min, 314 nm, 25 °C



### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	6.50	6225211.3002	425346.6407	50.00	50.00
2	11.68	6225752.0747	179680.0487	50.00	50.00
		12450963.3749	605026.6893	100.00	100.00

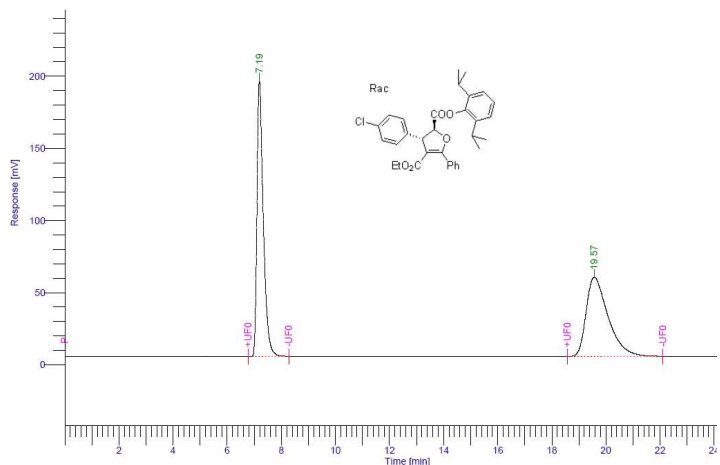
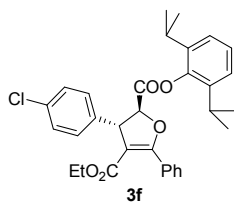
AD column, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm, 25 oC



### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	6.51	283182.4250	19889.2111	5.14	5.14
2	11.69	5227235.2142	149371.8095	94.86	94.86
		5510417.6392	169261.0206	100.00	100.00

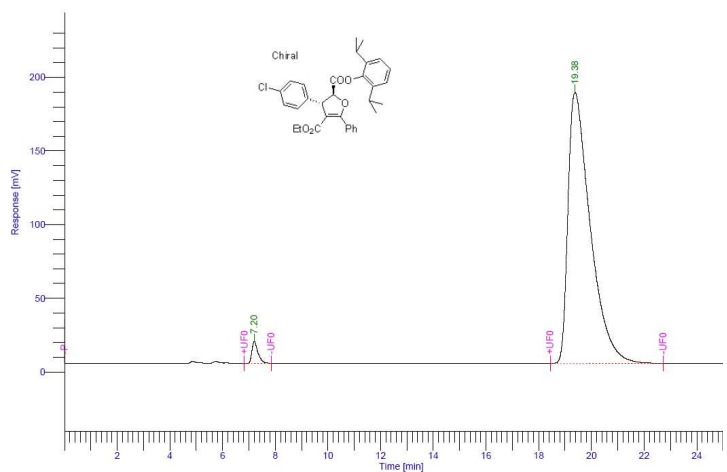
AD column, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm, 25 oC



### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	7.19	3092696.6699	191124.0123	49.94	49.94
2	19.57	3099884.5611	54935.6799	50.06	50.06
		6192581.2310	246059.6922	100.00	100.00

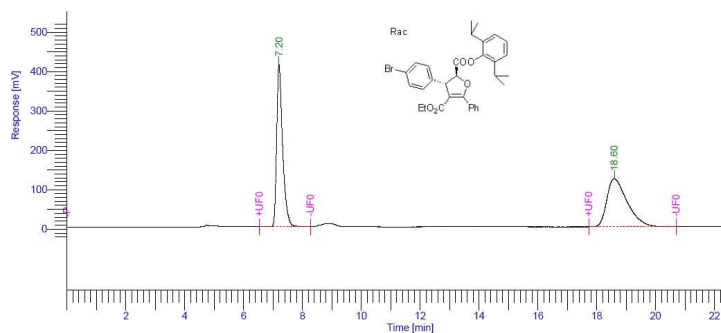
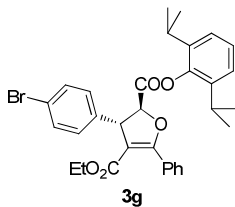
AD column, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm, 25 oC



### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	7.20	238571.8736	14964.6312	2.16	2.16
2	19.38	10789690.2978	184213.0722	97.84	97.84
		11028262.1714	199177.7034	100.00	100.00

AD column, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm, 25 oC



## Jiaolong Zhou HPLC Report

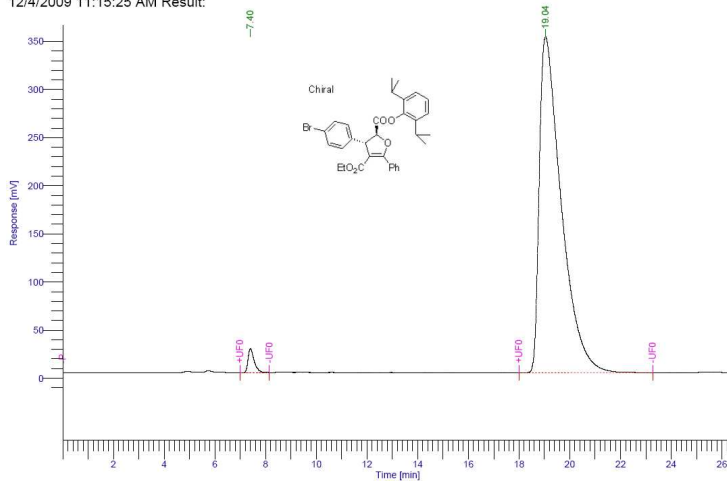
Zjl

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	7.20	5.8282e+06	4.119e+05	49.81	49.81
2	18.60	5.8716e+06	1.219e+05	50.19	50.19
		1.1700e+07	5.338e+05	100.00	100.00

Zjl: AD, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm

Page 2 of 2

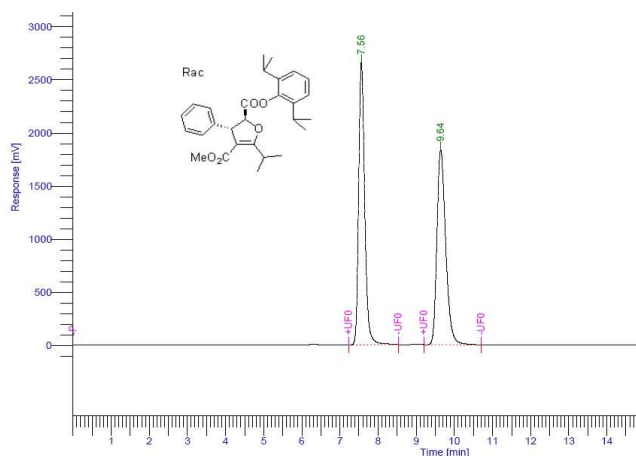
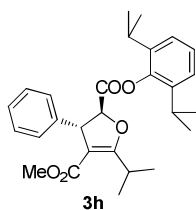
12/4/2009 11:15:25 AM Result:



## Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	7.40	416025.5228	25120.9748	1.95	1.95
2	19.04	20933961.7220	349045.9696	98.05	98.05
		21349987.2448	374166.9644	100.00	100.00

AD column, Hexane/iPrOH 95/5, 0.6 mL/min, 314 nm, 25 oC

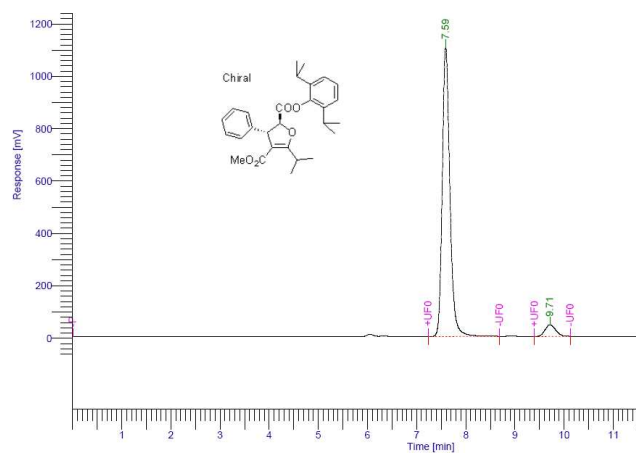


## Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	7.56	29435025.2148	2655142.3186	50.01	50.01
2	9.64	29424222.6066	1834432.8502	49.99	49.99
		58859247.8214	4489575.1688	100.00	100.00

Warning -- Signal level out-of-range in peak

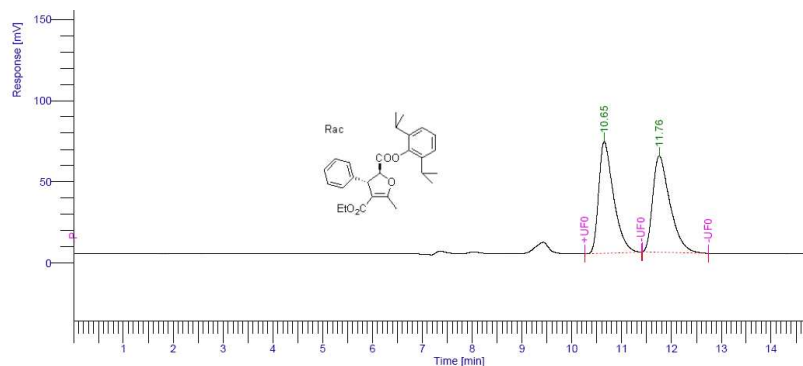
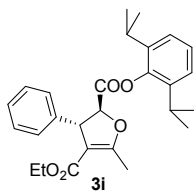
OD-H column, Hexane/iPrOH 97/3, 0.5 mL/min, 235 nm, 25 °C



## Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	7.59	11953859.2233	1104391.9837	94.58	94.58
2	9.71	685537.9996	44769.3137	5.42	5.42
		12639397.2229	1149161.2974	100.00	100.00

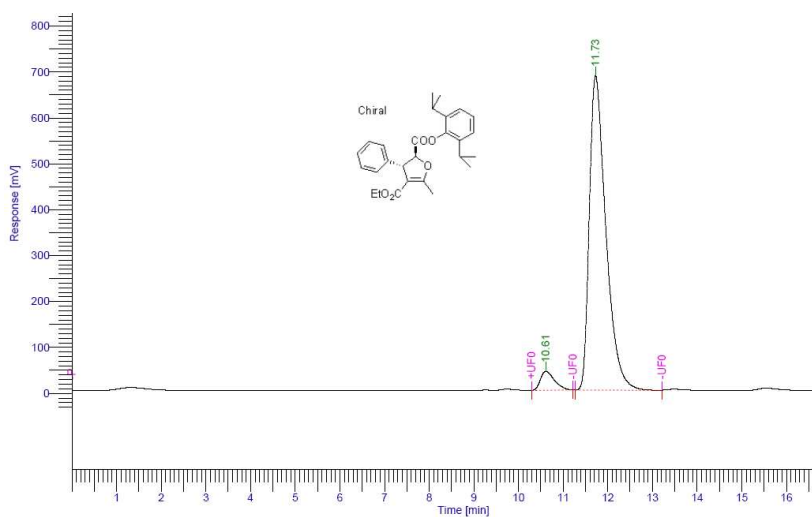
OD-H column, Hexane/iPrOH 97/3, 0.5 mL/min, 235 nm, 25 °C



### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area/Height [s]	Absorbance Ratio
1	10.65	1419783.5621	68677.1863	49.90	49.90	20.6733	-----
2	11.76	1425412.9965	59362.0071	50.10	50.10	24.0122	-----
		2845196.5586	128039.1935	100.00	100.00		0.0000

AD column, Hexane/iPrOH 98/2, 0.4 mL/min, 254 nm, 25 oC

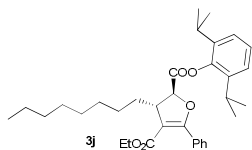


### Jiaolong Zhou HPLC Report

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1	10.61	904455.4561	41689.5039	4.89	4.89
2	11.73	17583418.2491	685118.1619	95.11	95.11
		18487873.7052	726807.6658	100.00	100.00

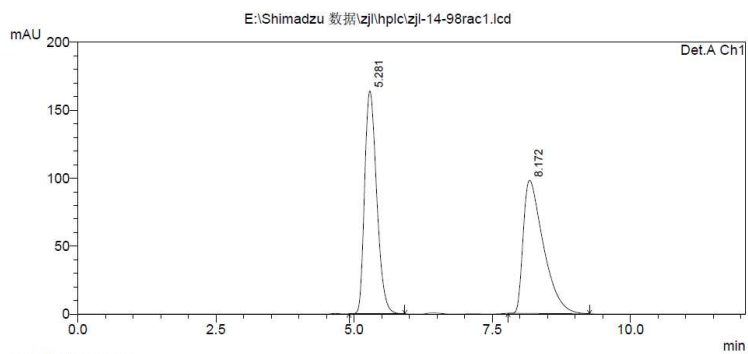
AD column, Hexane/iPrOH 98/2, 0.4 mL/min, 254 nm, 25 oC





Description : AD-H; 320 nm; Flow = 0.7 mL/min; i-PrOH/Hexane = 7/93

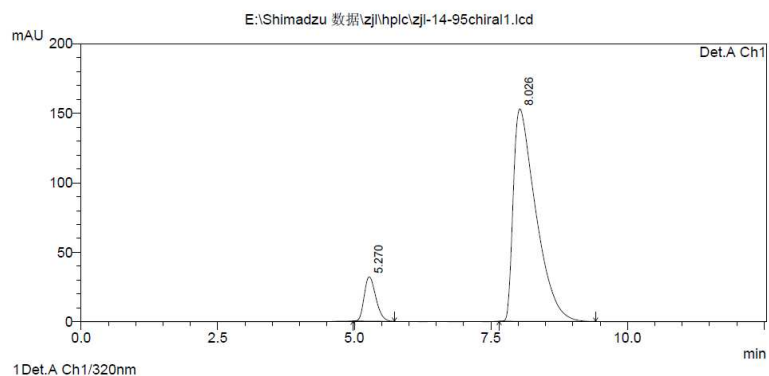
### <Chromatogram>



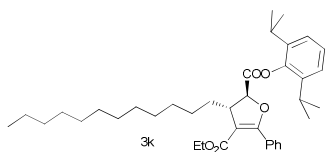
PeakTable					
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.281	2495681	163981	50.073	62.599
2	8.172	2488419	97973	49.927	37.401
Total		4984100	261953	100.000	100.000

Description : AD-H; 320 nm; Flow = 0.7 mL/min; i-PrOH/Hexane = 7/93

### <Chromatogram>

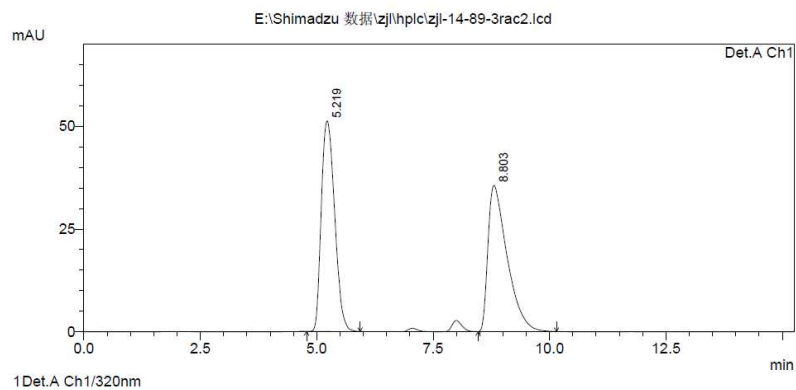


PeakTable					
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.270	471322	31900	9.684	17.268
2	8.026	4395945	152833	90.316	82.732
Total		4867267	184733	100.000	100.000



Description : AD-H; 320 nm; Flow = 0.7 mL/min; i-PrOH/Hexane = 5/95

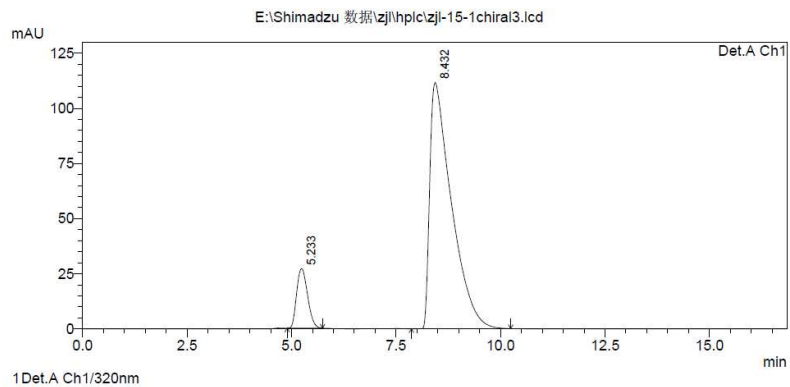
# <Chromatogram>



PeakTable					
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.219	1023495	51209	50.116	59.017
2	8.803	1018744	35560	49.884	40.983
Total		2042239	86769	100.000	100.000

Description : AD-H; 320 nm; Flow = 0.7 mL/min; i-PrOH/Hexane = 5/95

# <Chromatogram>



PeakTable					
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.233	488845	26950	10.818	19.444
2	8.432	4029913	111650	89.182	80.556
Total		4518759	138600	100.000	100.000