Supporting Information

Asymmetric Synthesis of Multifunctional Aryl Allyl Ethers by Nucleophilic Catalysis
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Contents
1. General information ..............................................................................................S2
2. Optimization of the solvent of the asymmetric allylic substitution reaction ....S2
3. General procedure of the asymmetric allylic substitution reactions and analytical data of the products........................................................…S3
4. Synthesis and characterization of MBH carbonate 5a .....................................S11
5. General procedure of asymmetric allylic substitution reaction of 5a ..........S12
6. Synthesis and characterization of chiral MBH alcohol 6 .................................S12
7. General procedure of 1, 3-dipolar cycloadDITION reaction of aryl allyl ether 4a ............................................................................................................S13
8. 1H NMR and 13C NMR spectra of 4, 5, 6, 7 .....................................................S14
9. Chiral HPLC chromatograms of 4, 5, 6, 7 .......................................................S40
1. General information

All glassware was thoroughly oven-dried. Chemicals and solvents were either purchased from commercial suppliers or purified by standard techniques. Thin-layer chromatography plates were visualized by exposure to ultraviolet light and/or staining with phosphomolybdic acid followed by heating on a hot plate. Flash chromatography was carried out using silica gel (160-200 mesh). $^1$H NMR and $^{13}$C NMR spectra were recorded using Bruker AV-300 / AV-400 spectrometers. Chemical shifts are given in δ relative to tetramethylsilane (TMS). Data for $^1$H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet), integration, coupling constant (Hz) and assignment. The spectra were recorded in CDCl$_3$ as the solvent at room temperature, TMS served as internal standard (δ = 0 ppm) for $^1$H NMR and CDCl$_3$ used as an internal standard (δ = 77.00 ppm) for $^{13}$C NMR. Optical rotations were measured on an Autopol IV (d = 589 nm, Hg lamp, 50mm cell) instrument (Rudolph, NJ, USA). High resolution mass spectra were acquired on Thermo Orbitrap Elite, instrument (Agilent, Palo Alto, CA, USA). Enantiomeric excess values were determined by HPLC with Chiralcel OD-H, IC, ID, IB columns on Agilent LC-1260 eluting with i-PrOH and n-hexane.

2. Optimization of the solvent of the asymmetric allylic substitution reaction

\[
\begin{align*}
2a + \text{OBoc} &\quad \rightleftharpoons \quad 3a \quad 1h (20 \text{ mol\%}) \\
\text{solvent} &\quad \text{rt} \\
2a &\quad 3a &\quad 4a
\end{align*}
\]

<table>
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<tr>
<th>Entry</th>
<th>Solvent</th>
<th>t (h)</th>
<th>Yield $^b$ (%)</th>
<th>Ee $^c$ (%)</th>
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<td>3</td>
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4  1,4-dioxane  96  95  95
5  THF  72  92  91
6  Et\textsubscript{2}O  86  57  91
7\textsuperscript{d}  THF  76  90  75
8\textsuperscript{e}  THF  72  -  -
9\textsuperscript{f}  Et\textsubscript{2}O  72  -  -

\textsuperscript{a} Unless otherwise noted, the reaction was carried out with 2a (0.1 mmol), 3a (0.3 mmol) and 1 (20 mol\%) in 2mL specified solvent at room temperature. \textsuperscript{b} The isolated yield. \textsuperscript{c} Determined by HPLC. \textsuperscript{d} The reaction was carried out at 0 °C. \textsuperscript{e} The reaction was carried out at -40 °C.

### 3. General procedure of the asymmetric allylic substitution reactions and analytical data of the products

A solution of phenol 2 (0.1 mmol), MBH carbonate 3 (0.3 mmol) and catalysts 1h (0.02 mmol) in 1, 4-dioxane (2 mL) was stirred at room temperature. The reaction was
monitored by TLC spectroscopy. After the reaction time given, the reaction mixture was directly purified by flash column chromatograph (eluted with EtOAc/petroleum ether: 10:1) to afford the product 4.

Methyl (S)-2-(phenoxy(phenyl)methyl)acrylate (4a) Colorless oil; 95% yield; 95% ee; [α]$_{28}^{D}$ = 128.0 (c 0.550, CH$_2$Cl$_2$); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane/iPrOH = 95:5), 0.5 mL/min, λ = 270 nm, $t_R$ (minor) = 11.1 min, $t_R$ (major) = 13.5 min. $^1$H NMR (300 MHz, CDCl$_3$): 7.46 (d, $J$ = 1.8 Hz, 2H), 7.44-7.20 (m, 5H), 7.04 (dd, $J$ = 0.9, 7.5 Hz, 1H), 6.76 (d, $J$ = 8.1 Hz, 1H), 6.37 (d, $J$ = 7.2 Hz, 1H), 6.05 (d, $J$ = 1.5 Hz, 1H), 6.03 (s, 2H), 5.90 (t, $J$ = 1.2 Hz, 1H), 3.74 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): 166.0, 157.5, 140.1, 138.8, 129.4, 128.5, 128.1, 127.4, 126.3, 121.2, 115.9, 77.2, 52.0. HRMS(ESI) for C$_{17}$H$_{16}$NaO$_3$ [M+Na]$^+$ calcd 291.0992, found 291.0992.

Methyl (S)-2-(phenyl(o-tolyloxy)methyl)acrylate (4b) Colorless oil; 95% yield; 91% ee; [α]$_{28}^{D}$ = 82.8 (c 1.000, CH$_2$Cl$_2$); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane/iPrOH = 95:5), 0.5 mL/min, λ = 270 nm, $t_R$ (minor) = 11.2 min, $t_R$ (major) = 14.7 min. $^1$H NMR (300 MHz, CDCl$_3$): 7.48-7.45 (m, 2H), 7.36-7.28 (m, 2H), 7.12 (dd, $J$ = 0.6, 7.2 Hz, 1H), 7.05 (d, $J$ = 1.5 Hz, 1H), 6.84 (dd, $J$ = 0.9, 7.5 Hz, 1H), 6.76 (d, $J$ = 8.1 Hz, 1H), 6.37 (d, $J$ = 7.2 Hz, 1H), 6.05 (d, $J$ = 1.5 Hz, 1H), 6.03 (s, 2H), 5.90 (t, $J$ = 1.2 Hz, 1H), 3.74 (s, 3H), 2.27 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): 166.1, 155.5, 140.7, 139.4, 130.8, 128.5, 128.0, 127.2, 126.7, 125.7, 120.8, 112.8, 76.9, 52.0, 16.6. HRMS(ESI) for C$_{18}$H$_{18}$NaO$_3$ [M+Na]$^+$ calcd 305.1148, found 305.1147.

Methyl (S)-2-(phenyl(m-tolyloxy)methyl)acrylate (4c) Colorless oil; 94% yield; 90% ee; [α]$_{28}^{D}$ = 120.3 (c 1.185, CH$_2$Cl$_2$); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane/iPrOH = 95:5), 0.5 mL/min, λ = 270 nm, $t_R$ (minor) = 11.1 min, $t_R$ (major) = 12.9 min. $^1$H NMR (300 MHz, CDCl$_3$): 7.45-7.43 (m, 2H), 7.37-7.28 (m, 2H), 7.10 (t, $J$ = 7.8 Hz, 1H), 6.75-6.69 (m, 3H), 6.38 (s, 1H), 6.14 (s, 1H), 5.98 (t, $J$ = 1.1 Hz, 1H), 3.74 (s, 3H), 2.28 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): 166.1, 157.5, 140.2, 139.4, 128.0, 127.2, 126.7, 125.7, 120.8, 112.8, 76.9, 52.0, 16.6. HRMS(ESI) for C$_{18}$H$_{18}$NaO$_3$ [M+Na]$^+$ calcd 305.1148, found 305.1147.
Methyl (S)-2-(phenyl(p-tolyloxy)methyl)acrylate (4d). Colorless oil; 93% yield; 91% ee; \([\alpha]_{28}^D = 110.0 (c 1.200, \text{CH}_2\text{Cl}_2)\); The enantiomeric excess was determined by HPLC with an OD-H column. (\text{n-hexane}:iPrOH = 95:5), 0.5 mL/min, \(\lambda = 270 \text{ nm}\), \(t_R(\text{minor}) = 11.2 \text{ min}\), \(t_R(\text{major}) = 10.1 \text{ min}\). \(^1\text{H NMR}\) (300 MHz, CDCl\(_3\)): 7.46-7.42 (m, 2H), 7.37-7.25 (m, 3H), 7.02 (d, \(J = 8.1 \text{ Hz}\), 2H), 6.82 (dd, \(J = 2.1, 6.6 \text{ Hz}\), 2H), 6.38 (s, 1H), 6.11 (s, 1H), 5.97 (t, \(J = 1.1 \text{ Hz}\), 1H), 3.74 (s, 3H); \(^{13}\text{C NMR}\) (100 MHz, CDCl\(_3\)): 166.2, 155.5, 140.3, 139.0, 130.5, 129.9, 128.5, 128.1, 127.5, 126.3, 115.8, 77.4, 52.0, 20.5. HRMS(ESI) for C\(_{18}\)H\(_{18}\)NaO\(_3\) [M+Na]\(^+\) calcld 305.1148, found 305.1149.

Methyl (S)-2-((2-methoxyphenoxy)(phenyl)methyl)acrylate (4e). Colorless oil; 54% yield; 87% ee; \([\alpha]_{25}^D = 70.4 (c 0.6, \text{CHCl}_3)\); The enantiomeric excess was determined by HPLC analysis with an OD-H column. (\text{n-hexane}:iPrOH = 97:3), 0.5 mL/min, \(\lambda = 270 \text{ nm}\), \(t_R(\text{major}) = 16.5 \text{ min}\), \(t_R(\text{minor}) = 22.4 \text{ min}\). \(^1\text{H NMR}\) (400 MHz, CDCl\(_3\)): 7.52-7.50 (m, 2H), 7.38-7.29 (m, 3H), 6.95-6.83 (m, 4H), 6.45 (s, 1H), 6.19 (s, 1H), 6.18 (s, 1H), 3.87 (s, 3H), 3.76 (s, 3H); \(^{13}\text{C NMR}\) (100 MHz, CDCl\(_3\)): 166.1, 150.3, 147.1, 140.3, 139.1, 128.4, 128.1, 127.5, 126.3, 122.1, 120.8, 116.5, 112.4, 78.5, 56.1, 52.0. HRMS (ESI) For C\(_{18}\)H\(_{18}\)NaO\(_4\) [M+Na]\(^+\) calcld 321.1097, found 321.1098.

Methyl (S)-2-((3-methoxyphenoxy)(phenyl)methyl)acrylate (4f). Colorless oil; 69% yield; 92% ee; \([\alpha]_{25}^D = 104.0 (c 1.0, \text{CHCl}_3)\); The enantiomeric excess was determined by HPLC analysis with an OD-H column. (\text{n-hexane}:iPrOH = 97:3), 0.5 mL/min, \(\lambda = 270 \text{ nm}\), \(t_R(\text{major}) = 16.4 \text{ min}\), \(t_R(\text{minor}) = 18.3 \text{ min}\). \(^1\text{H NMR}\) (400 MHz, CDCl\(_3\)): 7.45-7.43 (m, 2H), 7.36-7.25 (m, 3H), 7.14-7.10 (m, 1H), 6.53-6.47 (m,
Methyl (S)-2-((4-methoxyphenoxy)(phenyl)methyl)acrylate (4g) Colorless oil; 89\% yield; [\alpha]_D^{27} = 86.6 (c 1.0, CH_2Cl_2); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 95:5), 0.5 mL/min, \lambda = 270 nm, t_R(minor) = 11.2 min, t_R(major) = 12.4 min. \_H NMR (300 MHz, CDCl_3): 7.45-7.42 (m, 2H), 7.37-7.25 (m, 3H), 6.89-6.83 (m, 2H), 6.79-6.74 (m, 2H), 6.39 (s, 1H), 6.04 (s, 1H), 5.97 (t, \_J = 1.2 Hz, 1H), 3.74 (s, 3H), 3.73 (s, 3H); \_C NMR (75 MHz, CDCl_3): 166.1, 154.1, 151.7, 140.3, 139.0, 128.5, 128.1, 127.4, 126.2, 117.1, 114.5, 78.2, 55.6, 52.0. HRMS (ESI) for C_{18}H_{19}O_4Na^+ calcd 321.1097, found 321.1098.

Methyl (S)-2-((2-fluorophenoxy)(phenyl)methyl)acrylate (4h) Colorless oil; 58\% yield; 95\% ee; [\alpha]_D^{28} = 107.7 (c 0.665, CH_2Cl_2); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 97:3), 0.5 mL/min, \lambda = 270 nm, t_R(minor) = 12.8 min, t_R(major) = 13.8 min. \_H NMR (300 MHz, CDCl_3): 7.46 (dd, \_J = 1.2, 8.1 Hz, 2H), 7.37-7.26 (m, 3H), 7.08-7.01 (m, 1H), 6.96-6.86 (m, 3H), 6.42 (s, 1H), 6.17 (s, 1H), 6.09 (t, \_J = 1.1 Hz, 1H), 3.74 (s, 3H); \_C NMR (100 MHz, CDCl_3): 165.9, 154.8, 151.5, 145.5, 145.4, 140.0, 138.4, 128.5, 128.3, 127.4, 126.4, 124.2, 124.1, 121.9, 121.8, 117.2, 116.5, 116.2, 78.6, 52.0. HRMS(ESI) for C_{17}H_{15}FNaO_3 [M+Na]^+ calcd 309.0897, found 309.0897.

Methyl (S)-2-((3-fluorophenoxy)(phenyl)methyl)acrylate (4i) Colorless oil; 76\% yield; 95\% ee; [\alpha]_D^{28} = 106.6 (c 0.910, CH_2Cl_2); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 97:3), 0.5 mL/min, \lambda
= 270 nm, t<sub>R(minor)</sub> = 13.1 min, t<sub>R(major)</sub> = 12.0 min. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): 7.43 (dd, <i>J</i> = 1.7, 8.0 Hz, 2H), 7.38-7.30 (m, 3H), 7.20-7.12 (m, 1H), 6.72-6.60 (m, 3H), 6.40 (s, 1H), 6.13 (s, 1H), 5.93 (t, <i>J</i> = 1.1 Hz, 1H), 3.76 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): 165.9, 165.1, 161.8, 158.9, 158.8, 139.9, 138.3, 130.2, 130.1, 128.6, 128.3, 127.3, 126.5, 111.6, 111.5, 108.2, 107.9, 103.9, 103.5, 77.6, 52.1. HRMS(ESI) for C<sub>17</sub>H<sub>15</sub>FNaO<sub>3</sub> [M+Na]<sup>+</sup> calcd 309.0897, found 309.0894.

**Methyl (S)-2-((4-fluorophenoxy)(phenyl)methyl)acrylate (4j)** Colorless oil; 70% yield; [\(\alpha\)]<sub>28</sub> D = 106.5 (c 0.550, CH<sub>2</sub>Cl<sub>2</sub>); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 97:3), 0.5 mL/min, \(\lambda = 270\) nm, t<sub>R(minor)</sub> = 11.5 min, t<sub>R(major)</sub> = 10.1 min. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): 7.44-7.41 (m, 2H), 7.38-7.29 (m, 3H), 6.94-6.84 (m, 2H), 6.39 (t, <i>J</i> = 0.8 Hz, 1H), 6.06 (s, 1H), 5.94 (t, <i>J</i> = 1.1 Hz, 1H), 3.75 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): 166.0, 159.1, 155.9, 153.7, 140.1, 138.6, 128.6, 128.2, 127.4, 126.4, 117.2, 117.1, 115.9, 115.6, 78.2, 52.1. HRMS(ESI) for C<sub>17</sub>H<sub>15</sub>FNaO<sub>3</sub> [M+Na]<sup>+</sup> calcd 309.0897, found 309.0896.

**Methyl (S)-2-((2-chlorophenoxy)(phenyl)methyl)acrylate (4k)** Colorless oil; 79% yield; 93% ee; [\(\alpha\)]<sub>28</sub> D = 82.3 (c 0.700, CH<sub>2</sub>Cl<sub>2</sub>); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 95:5), 0.5 mL/min, \(\lambda = 270\) nm, t<sub>R(minor)</sub> = 12.3 min, t<sub>R(major)</sub> = 14.9 min. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): 7.51-7.48 (m, 2H), 7.37-7.27 (m, 4H), 7.12-7.06 (m, 1H), 6.89-6.82 (m, 2H), 6.39 (s, 1H), 6.22 (s, 1H), 6.16 (d, <i>J</i> = 0.9 Hz, 1H), 3.75 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): 165.9, 152.8, 140.1, 138.5, 130.3, 128.5, 128.2, 127.5, 127.2, 126.2, 123.6, 121.8, 115.2, 77.7, 52.0. HRMS(ESI) for C<sub>17</sub>H<sub>15</sub>ClNaO<sub>3</sub> [M+Na]<sup>+</sup> calcd 325.0602, found 325.0600.

**Methyl (S)-2-((3-chlorophenoxy)(phenyl)methyl)acrylate (4l)** Colorless oil; 93% yield; 92% ee; [\(\alpha\)]<sub>28</sub> D = 96.0 (c 0.950, CH<sub>2</sub>Cl<sub>2</sub>); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 95:5), 0.5 mL/min, \(\lambda = 270\) nm, t<sub>R(minor)</sub> = 13.1 min, t<sub>R(major)</sub> = 12.0 min. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): 7.43 (dd, <i>J</i> = 1.7, 8.0 Hz, 2H), 7.38-7.30 (m, 3H), 7.20-7.12 (m, 1H), 6.72-6.60 (m, 3H), 6.40 (s, 1H), 6.13 (s, 1H), 5.93 (t, <i>J</i> = 1.1 Hz, 1H), 3.76 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): 165.9, 165.1, 161.8, 158.9, 158.8, 139.9, 138.3, 130.2, 130.1, 128.6, 128.3, 127.3, 126.5, 111.6, 111.5, 108.2, 107.9, 103.9, 103.5, 77.6, 52.1. HRMS(ESI) for C<sub>17</sub>H<sub>15</sub>ClNaO<sub>3</sub> [M+Na]<sup>+</sup> calcd 325.0602, found 325.0600.
Methyl (S)-2-((4-chlorophenoxy)(phenyl)methyl)acrylate (4m) Colorless oil; 73% yield; 94% ee; [α]28 D = 107.8 (c 0.850, CH2Cl2); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 95:5), 0.5 mL/min, λ = 270 nm, tR(minor) = 12.2 min, tR(major) = 10.4 min. 1H NMR (300 MHz, CDCl3): 7.44-7.41 (m, 2H), 7.38-7.30 (m, 3H), 7.17 (d, J = 9.0 Hz, 2H), 6.84 (d, J = 9.3 Hz, 2H), 6.39 (s, 1H), 6.10 (s, 1H), 5.92 (t, J = 1.1 Hz, 1H), 3.75 (s, 3H); 13C NMR (100 MHz, CDCl3): 166.0, 158.3, 139.9, 138.3, 129.3, 128.6, 128.3, 127.3, 126.4, 126.1, 117.2, 77.6, 52.1. HRMS(ESI) for C17H15ClNaO3 [M+Na]+ calcd 325.0602, found 325.0591.

Methyl (S)-2-((3-bromophenoxy)(phenyl)methyl)acrylate (4n) Colorless oil; 93% yield; 89% ee; [α]28 D = 93.1 (c 1.057, CH2Cl2); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 95:5), 0.5 mL/min, λ = 270 nm, tR(minor) = 13.3 min, tR(major) = 11.7 min. 1H NMR (300 MHz, CDCl3): 7.42 (dd, J = 1.5, 8.1 Hz, 2H), 7.38-7.29 (m, 3H), 7.11-7.06 (m, 3H), 6.86-6.82 (m, 1H), 6.39 (s, 1H), 6.12 (s, 1H), 5.92 (t, J = 1.1 Hz, 1H), 3.75 (s, 3H); 13C NMR (100 MHz, CDCl3): 165.9, 158.3, 139.8, 138.2, 130.5, 128.6, 128.3, 127.3, 126.5, 124.4, 122.7, 119.5, 114.5, 77.5, 52.1. HRMS(ESI) for C17H14BrO3 [M-H]− calcd 345.0132, found 345.0128.

Methyl (S)-2-((naphthalen-1-yloxy)(phenyl)methyl)acrylate (4o) Colorless oil; 94%
yield; 86% ee; $\alpha$ 28 D = 24.9 (c 0.907, CH$_2$Cl$_2$); The enantiomeric excess was determined by HPLC with an OD-H column. ($n$-hexane:iPrOH = 95:5), 0.5 mL/min, $\lambda$ = 270 nm, $t_R$(minor) = 16.1 min, $t_R$(major) = 12.8 min. $^1$H NMR (300 MHz, CDCl$_3$): 8.35-8.31 (m, 1H), 7.78-7.75 (m, 1H), 7.56-7.52 (m, 2H), 7.48-7.20 (m, 7H), 6.78 (d, $J$ = 7.5 Hz, 1H), 6.39 (d, $J$ = 5.1 Hz, 2H), 6.07 (t, $J$ = 1.1 Hz, 2H), 3.73 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): 166.1, 152.9, 140.2, 139.0, 134.5, 128.5, 128.1, 127.2, 126.3, 125.9, 125.7, 125.2, 122.0, 120.7, 106.8, 77.2, 52.0. HRMS(ESI) for C$_{21}$H$_{18}$NaO$_3$ [M+Na]$^+$ calcd 341.1148, found 341.1144.

Methyl (S)-2-(phenoxy(o-tolyl)methyl)acrylate (4p) Colorless oil; 73% yield; 95% ee; $\alpha$ 28 D = 65.4 (c 0.700, CH$_2$Cl$_2$); The enantiomeric excess was determined by HPLC with an OD-H column. ($n$-hexane:iPrOH = 95:5), 0.5 mL/min, $\lambda$ = 270 nm, $t_R$(minor) = 13.3 min, $t_R$(major) = 15.2 min. $^1$H NMR (300 MHz, CDCl$_3$): 7.41-7.37 (m, 1H), 7.25-7.19 (m, 5H), 6.94-6.88 (m, 3H), 6.44 (s, 1H), 6.34 (s, 1H), 5.74 (t, $J$ = 1.2 Hz, 1H), 3.75 (s, 3H), 2.36 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): 166.3, 157.9, 139.1, 136.3, 136.2, 130.6, 129.4, 128.2, 127.6, 127.1, 126.2, 121.1, 115.6, 74.5, 52.1, 19.2. HRMS(ESI) for C$_{18}$H$_{18}$NaO$_3$ [M+Na]$^+$ calcd 305.1148, found 305.1151.

Methyl (S)-2-((3-fluorophenyl)(phenoxy)methyl)acrylate (4r) Colorless oil; 93% yield; 95% ee; $\alpha$ 28 D = 106.1 (c 0.867, CH$_2$Cl$_2$); The enantiomeric excess was determined by HPLC with an OD-H column. ($n$-hexane:iPrOH = 97:3), 0.5 mL/min, $\lambda$ = 270 nm, $t_R$(minor) = 10.3 min, $t_R$(major) = 21.9 min. $^1$H NMR (300 MHz, CDCl$_3$): 7.33-7.15 (m, 5H), 7.00-6.89 (m, 4H), 6.40 (s, 1H), 6.15 (s, 1H), 5.99 (t, $J$ = 1.1 Hz, 1H),
3.75 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): 165.8, 164.4, 161.2, 157.2, 141.6, 141.5, 139.7, 130.1, 130.0, 129.4, 126.6, 123.0, 123.0, 121.4, 115.8, 115.2, 114.9, 114.4, 114.1, 76.5, 76.4, 52.1. HRMS(ESI) for C$_{18}$H$_{17}$FNaO$_3$ [M+Na]$^+$ calcd 309.0897, found 309.0898.

Methyl (S)-2-((4-fluorophenyl)(phenoxy)methyl)acrylate (4s) Colorless oil; 95% yield; 95% ee; $[^{[α}]_{28}^D = 132.5$ (c 1.135, CH$_2$Cl$_2$); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 97:3), 0.5 mL/min, $λ = 270$ nm, $t_R($minor$) = 10.0$ min, $t_R($major$) = 17.6$ min. $^1$H NMR (300 MHz, CDCl$_3$): 7.42 (dd, $J = 5.4$, 8.7 Hz, 2H), 7.26-7.21 (m, 2H), 7.02 (t, $J = 8.7$ Hz, 2H), 6.95-6.89 (m, 3H), 6.39 (s, 1H), 6.13 (s, 1H), 5.99 (t, $J = 1.1$ Hz, 1H), 3.74 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): 165.9, 164.1, 160.8, 157.3, 139.9, 134.7, 134.6, 129.4, 129.2, 129.1, 126.1, 121.3, 115.9, 115.6, 115.3, 76.6, 52.1. HRMS(ESI) for C$_{18}$H$_{17}$FNaO$_3$ [M+Na]$^+$ calcd 309.0897, found 309.0896.

Methyl (S)-2-((4-chlorophenyl)(phenoxy)methyl)acrylate (4t) Colorless oil; 92% yield; 91% ee; $[^{[α}]_{28}^D = 119.1$ (c 0.833, CH$_2$Cl$_2$); The enantiomeric excess was determined by HPLC with an IA column. (n-hexane:iPrOH = 95:5), 0.5 mL/min, $λ = 270$ nm, $t_R($minor$) = 18.8$ min, $t_R($major$) = 17.0$ min. $^1$H NMR (300 MHz, CDCl$_3$): 7.40-7.37 (m, 2H), 7.33-7.29 (m, 2H), 7.24-7.21 (m, 2H), 6.96-6.88 (m, 3H), 6.39 (s, 1H), 6.11 (s, 1H), 6.00 (t, $J = 1.1$ Hz, 1H), 3.74 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): 165.8, 157.2, 139.7, 137.4, 133.9, 129.4, 128.8, 128.7, 126.3, 121.4, 115.8, 76.5, 52.1. HRMS(ESI) for C$_{17}$H$_{15}$ClNaO$_3$ [M+Na]$^+$ calcd 325.0602, found 325.0604.

Methyl (S)-2-((4-bromophenyl)(phenoxy)methyl)acrylate (4u) Colorless oil; 79% yield; 91% ee; $[^{[α}]_{28}^D = 117.7$ (c 1.025, CH$_2$Cl$_2$); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 97:3), 0.5 mL/min, $λ = 270$ nm, $t_R($minor$) = 9.8$ min, $t_R($major$) = 17.5$ min. $^1$H NMR (300 MHz, CDCl$_3$): 7.46 (dd, $J = 2.0$, 6.8 Hz, 2H), 7.33 (dd, $J = 1.8$, 6.6 Hz, 2H), 7.23-7.20 (m, 2H), 6.95-6.88 (m,
3H), 6.39 (s, 1H), 6.10 (s, 1H), 5.99 (t, J = 1.1 Hz, 1H), 3.74 (s, 3H); 13C NMR (100 MHz, CDCl3): 165.8, 157.2, 139.7, 138.0, 131.6, 129.4, 129.1, 126.4, 122.1, 121.4, 115.8, 76.5, 52.0. HRMS(ESI) for C17H13BrNaO3 [M+Na]+ calcd 369.0097, found 369.0100.

Methyl (S)-2-((4-nitrophenyl)(phenoxy)methyl)acrylate (4v) Colorless oil; 77% yield; 93% ee; [α]28 D = 150.9 (c 0.790, CH2Cl2); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 97:3), 0.5 mL/min, λ = 270 nm, tR(minor) = 25.0 min, tR(major) = 46.8 min. 1H NMR (300 MHz, CDCl3): 8.20 (dd, J = 1.8, 6.9 Hz, 2H), 7.66 (d, J = 8.7 Hz, 2H), 7.25 (dd, J = 7.5, 8.7 Hz, 2H), 6.99-6.89 (m, 3H), 6.45 (s, 1H), 6.23 (s, 1H), 6.10 (s, 1H), 3.77 (s, 3H); 13C NMR (100 MHz, CDCl3): 165.6, 156.8, 147.6, 146.3, 139.2, 129.6, 128.1, 127.1, 123.7, 121.8, 115.8, 76.2, 52.2. HRMS(ESI) for C17H15NNaO5 [M+Na]+ calcd 336.0842, found 336.0847.

Methyl (S)-2-((3,5-dibromophenyl)(phenoxy)methyl)acrylate (4w) Colorless oil; 96% yield; 91% ee; [α]28 D = 90.1 (c 1.083, CH2Cl2); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 95:5), 0.5 mL/min, λ = 270 nm, tR(minor) = 8.7 min, tR(major) = 27.9 min. 1H NMR (300 MHz, CDCl3): 7.59-7.54 (m, 3H), 7.28-7.22 (m, 2H), 6.98-6.88 (m, 3H), 6.43 (s, 1H), 6.05 (s, 1H), 6.04 (s, 1H), 3.77 (s, 3H); 13C NMR (100 MHz, CDCl3): 165.5, 156.9, 143.0, 139.0, 133.7, 129.5, 129.1, 127.0, 123.0, 121.7, 115.8, 75.8, 52.2. HRMS(ESI) for C17H14Br2NaO3 [M+Na]+ calcd 448.9181, found 448.9181.

Methyl (R)-2-(phenoxy(thiophen-2-yl)methyl)acrylate (4x) Colorless oil; 93% yield; 92% ee; [α]28 D = 145.3 (c 0.950, CH2Cl2); The enantiomeric excess was determined by HPLC with an OD-H column. (n-hexane:iPrOH = 95:5), 0.5 mL/min, λ = 270 nm, tR(minor) = 12.9 min, tR(major) = 14.6 min. 1H NMR (300 MHz, CDCl3): 7.59 (t, J = 1.7 Hz, 1H), 7.54 (d, J = 1.8 Hz, 1H), 7.28-7.23 (m, 2H), 6.99-6.88 (m, 3H), 6.43 (s, 1H), 6.05-6.04 (m, 2H), 3.77 (s, 3H); 13C NMR (100 MHz, CDCl3): 165.5, 156.9,
143.0, 139.1, 133.8, 129.6, 129.2, 127.1, 123.0, 121.8, 115.8, 75.9, 52.2. HRMS(ESI) for C$_{15}$H$_{14}$SNaO$_3$[M+Na]$^+$ calcld 297.0556, found 297.0563.

4. Synthesis and characterization of MBH carbonate 5a

To a solution of racemic MBH alcohol (10 mmol) and pyridine (1 mL) in CH$_2$Cl$_2$ (20 mL) was added Phenyl chloroformate (12 mmol) at room temperature. The reaction was monitored by TLC spectroscopy. After completion of the reaction, the reaction mixture was directly purified by flash column chromatograph to afford the product 5a.

**Methyl 2-(((phenoxycarbonyl)oxy)(phenyl)methyl)acrylate (5a)** Colorless solid; 70% yield. $^1$H NMR (300 MHz, CDCl$_3$): 7.47-7.44 (m, 2H), 7.41-7.32 (m, 5H), 7.24-7.13 (m, 3H), 6.63 (s, 1H), 6.48 (s, 1H), 6.02-6.01 (m, 1H), 3.72 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$): 165.1, 152.5, 151.0, 138.9, 136.7, 129.4, 128.8, 128.6, 127.7, 126.4, 126.0, 120.9, 77.5, 52.1.

5. General procedure of asymmetric allylic substitution reaction of 5a

A solution of MBH carbonate 5a (0.2 mmol) and catalyst 1h (0.04 mmol) in THF (4 mL) was stirred at room temperature. The reaction was monitored by TLC spectroscopy. After the reaction time given, the reaction mixture was directly purified by flash column chromatograph (eluted with EtOAc/petroleum ether: 10:1) to afford the product 4a.
6. Synthesis and characterization of chiral MBH alcohol 6

To a solution of 4g (0.1 mmol) in CH$_3$CN:H$_2$O (4:1, 2.5mL) was added ceric ammonium nitrate (CAN, 0.3 mmol) at room temperature. After stirring for 5 minutes at room temperature, all the solvents were removed and the residue was purified by flash column chromatograph to afford the product 6.

**Methyl (S)-2-(hydroxy(phenyl)methyl)acrylate (6)** Colorless oil; 71% yield; 92% ee; [α]$^\text{D}$ = 70.0 (c = 0.100, MeOH); The enantiomeric excess was determined by HPLC with an IC column. (n-hexane : iPrOH = 95:5), 0.5 mL/min, $\lambda$ = 254 nm, $t_R(\text{minor})$ = 15.2 min, $t_R(\text{major})$ = 23.8 min. $^1$H NMR (300 MHz, CDCl$_3$): 7.39-7.26 (m, 5H), 6.34 (t, $J$ = 0.9 Hz, 1H), 5.84 (t, $J$ = 1.2 Hz, 1H), 5.56 (d, $J$ = 5.7 Hz, 1H), 3.72 (s, 3H), 3.08 (d, $J$ = 5.7 Hz, 1H); $^{13}$C NMR (75 MHz, CDCl$_3$): 166.7, 141.9, 141.2, 128.4, 127.8, 126.5, 126.1, 73.2, 51.9. HRMS(ESI) for C$_{11}$H$_{12}$NaO$_3$ [M+Na]$^+$ calcd 215.0679, found 215.0682.

7. General procedure of 1, 3-dipolar cycloaddition reaction of aryl allyl ether 4a

To a solution of 4a (0.1 mmol) and hydroximoyl chloride (0.12 mmol) in DCM (1mL) was added DIPEA (0.1 mmol) at room temperature. The reaction was monitored by TLC spectroscopy. After the reaction was complete, the reaction mixture was directly purified by flash column chromatograph to afford the products 7a and 7a'.

**Methyl 5-((S)-phenoxy(phenyl)methyl)-3-phenyl-4,5-dihydroisoxazole-5-carboxylate (7a) one of the two diastereomers** Colorless oil; 40% yield; 90% ee; [α]$^\text{D}$ = -69.9 (c = 0.77, CH$_2$Cl$_2$); The enantiomeric excess was determined by HPLC.
with an OD column. \((n\text{-hexane : }\text{iPrOH} = 90:10)\), 0.5 mL/min, \(\lambda = 270\) nm, \(t_{R,\text{ minor}} = 21.3\) min, \(t_{R,\text{ major}} = 28.2\) min. \(^1\)H NMR (300 MHz, CDCl\(_3\)): 7.52-7.46 (m, 5H), 7.36-7.28 (m, 3H), 7.25-7.16 (m, 4H), 6.91 (t, \(J = 7.5\) Hz, 1H), 6.84-6.81 (m, 2H), 5.86 (s, 1H), 4.05 (d, \(J = 17.4\) Hz, 1H), 3.76 (s, 3H), 3.56 (d, \(J = 17.4\) Hz, 1H); \(^1\)C NMR (75 MHz, CDCl\(_3\)): 170.0, 157.2, 156.9, 134.6, 130.3, 129.4, 128.6, 128.6, 128.4, 128.4, 127.5, 126.7, 121.7, 115.8, 90.8, 79.2, 53.2, 38.6. HRMS(ESI) for C\(_{24}\)H\(_{21}\)KNO\(_4\) [M+K]\(^+\) calcd 426.1102, found 426.1106.

Methyl 5-((5)-phenoxy(phenyl)methyl)-3-phenyl-4,5-dihydroisoxazole-5-carboxylate (7a’) another of the two diastereomers Colorless oil; 40% yield; 90% ee; \([\alpha]_{27}^D = -24.6\) (c = 0.78, CH\(_2\)Cl\(_2\)); The enantiomeric excess was determined by HPLC with an IA column. \((n\text{-hexane:iPrOH} = 90:10)\), 0.5 mL/min, \(\lambda = 270\) nm, \(t_{R,\text{ minor}} = 18.5\) min, \(t_{R,\text{ major}} = 23.7\) min. \(^1\)H NMR (300 MHz, CDCl\(_3\)): 7.62 (dd, \(J = 1.5, 5.7\) Hz, 2H), 7.47 (d, \(J = 5.1\) Hz, 2H), 7.39-7.30 (m, 6H), 7.16 (t, \(J = 6\) Hz, 2H), 6.87 (d, \(J = 5.7\) Hz, 3H), 5.69 (s, 1H), 3.78 (s, 5H); \(^1\)C NMR (75 MHz, CDCl\(_3\)): 170.4, 157.6, 156.1, 135.0, 130.3, 129.3, 129.0, 128.7, 128.7, 127.7, 126.8, 121.8, 116.6, 92.2, 81.4, 53.0, 39.0. HRMS(ESI) for C\(_{24}\)H\(_{21}\)KNO\(_4\) [M+K]\(^+\) calcd 426.1102, found 426.1106.
8. $^1$H NMR and $^{13}$C NMR spectra of 4, 5, 6, 7
$4b$

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$\text{Joxin 3136  EN17041001 13c dce13}$

$\text{S16}$
4c

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4d

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4e

S19
4g

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S34
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3oxin 3410 J2-OH 13c cdc13
7a (one of the two diastereomers)
7a’ (another of the two diastereomers)
9. Chiral HPLC chromatograms of 4, 5, 6, 7

4a

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---|---------------------|------------------|-------------------|------------------|----------------
1  | 12.775              | 0.3287           | 2744.57227        | 125.12720        | 49.7545        
2  | 13.838              | 0.3503           | 2771.65674        | 119.02826        | 50.2455        

Peak | Retention time (min) | Peak width (min) | Peak area (mAU*s) | Peak height (mAU) | Peak area (%)  
---|---------------------|------------------|-------------------|------------------|----------------
1  | 12.839              | 0.3469           | 189.21298         | 9.09151          | 2.4847         
2  | 13.835              | 0.4062           | 7425.78271        | 304.65503        | 97.5153        

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--- | --- | --- | --- | --- | ---  
1 | 10.220 | 0.3842 | 6486.33643 | 256.19669 | 50.3183  
2 | 11.823 | 0.4032 | 6404.28076 | 243.30074 | 49.6817

Peak | Retention time (min) | Peak width (min) | Peak area (mAU*s) | Peak height (mAU) | Peak area (%)  
--- | --- | --- | --- | --- | ---  
1 | 10.135 | 0.4351 | 9939.78809 | 380.73264 | 96.5366  
2 | 11.507 | 0.4003 | 356.60742 | 14.84893 | 3.4634
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### Peak 1
- Retention time (min): 14.395
- Peak width (min): 0.4241
- Peak area (mAU*s): 1.06802e4
- Peak height (mAU): 379.88190
- Peak area (%): 49.7665

### Peak 2
- Retention time (min): 16.366
- Peak width (min): 0.4854
- Peak area (mAU*s): 1.07805e4
- Peak height (mAU): 331.77011
- Peak area (%): 50.2335

### Peak 1
- Retention time (min): 13.326
- Peak width (min): 0.3924
- Peak area (mAU*s): 170.46632
- Peak height (mAU): 7.24092
- Peak area (%): 2.7786

### Peak 2
- Retention time (min): 15.230
- Peak width (min): 0.4345
- Peak area (mAU*s): 5964.57568
- Peak height (mAU): 228.81248
- Peak area (%): 97.2214
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Peak | Retention time (min) | Peak width (min) | Peak area (mAU*s) | Peak height (mAU) | Peak area (%) 
--- | --- | --- | --- | --- | --- 
1 | 9.797 | 0.5448 | 4800.68457 | 146.85161 | 49.9841 
2 | 17.329 | 0.5063 | 4803.73633 | 143.75893 | 50.0159 

Peak | Retention time (min) | Peak width (min) | Peak area (mAU*s) | Peak height (mAU) | Peak area (%) 
--- | --- | --- | --- | --- | --- 
1 | 9.842 | 0.4379 | 262.86804 | 9.96642 | 4.6244 
2 | 17.475 | 0.4949 | 5400.86914 | 163.98764 | 95.3756
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### Table 1: Peak Characteristics

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### Table 2: Peak Characteristics

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<th>Peak</th>
<th>Retention time (min)</th>
<th>Peak width (min)</th>
<th>Peak area (mAU*s)</th>
<th>Peak height (mAU)</th>
<th>Peak area (%)</th>
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7a (one of the two diastereomers)

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Peak 1:

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7a’ (another of the two diastereomers)

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