Supporting Information

Iron (III)-catalyzed intramolecular stereospecific substitution of the OH group in stereogenic secondary and tertiary alcohols

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   a. Checklist of characterization data of synthesized intermediates

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c. Checklist of characterization data of products

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2. **General information:**

Unless otherwise noted, all reactions were carried out in oven-dried 5 ml vial. All the reagents and solvents were bought from commercial sources and were used without further purification. All reactions were executed with oven-dried glassware under inert condition using argon. 1,2-Dichloroethane (DCE) was distilled using CaH₂. Dry THF, diethyl ether and toluene were obtained from a VAC solvent purifier. NMR spectra were recorded with a 400 MHz (¹H) and 100 MHz (¹³C) spectrometer as solutions in CDCl₃. Chemical shifts (δ) are reported in parts per million (ppm) and are referenced to CDCl₃ (δ = 7.26 ppm) as an internal standard. All coupling constants (J) are expressed in Hz. The description of the signals include: s = singlet, d = doublet, t = triplet, m = multiplet and dd = doublet of doublets, at = apparent triplet. IR spectra were recorded by a Perkin Elmer FT-IR Spectrometer. High-Resolution Mass Spectra (HRMS) were performed with a micrOTOF (Bruker) spectrometer by Na-formate. The molecular fragments are quoted as the relation between mass and charge (m/z). The enantiospecificity (e.s.) of products were determined by chiral HPLC using the corresponding racemic compounds as references. The routine monitoring of reactions was performed by crude ¹H NMR.

3. **List of abbreviations**

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4. General Scheme A for the synthesis of 1a, 1b, 1c, 1d, 1e, 1f, and 1g:

5. General Method A to synthesize 1a, 1b, 1c, 1d, 1e, 1f, and 1g:

To a solution of 2-pyrrolidinone (30 mmol) in 30 mL dry DMF was added CuI (10 mol%), anhydrous K$_2$CO$_3$ (1.1 equiv.) and aryl bromide (2 equiv.). The reaction mixture was refluxed for 48 h. After completion of the reaction, the reaction mixture was allowed to attain room temperature. Aqueous saturated NH$_4$Cl (50 ml) was then added and the aqueous layer was separated and extracted with EtOAc (4 × 50 ml). The combined organic phase were washed with brine (1×50 mL), dried over anhydrous MgSO$_4$ and concentrated under reduced pressure to give the crude product. Purification was carried out by silica gel column chromatography to afford N-aryl-2-pyrrolidinones A.

An oven-dried round-bottomed flask equipped with a magnetic stir bar was charged with dry THF (20 mL) and N-aryl-2-pyrrolidinones A (10 mmol) under argon atmosphere. The solution was cooled to 0 °C and aryl magnesium bromide (1.1 equiv, in 4 mL THF) was added dropwise. The reaction was allowed to attain room temperature and was run at the same temperature for 3 h. The reaction was quenched with saturated NH$_4$Cl solution (30 mL)
extracted into diethyl ether (3×50 mL). The combined organics were dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude residues (ketones, B1-B7) were directly used for the next step (i.e. CBS-reduction) without further purification.

Ketones (B1-B7) were reduced to the alcohols enantioselectively by Corey-Bakshi-Shibata (CBS) reduction method. An oven-dried round-bottomed flask equipped with a magnetic stir bar was charged with BH$_3$/THF complex (1.2 equiv.) and chiral oxazaborolidine catalyst (R-CBS-Ox, 10 mol%) under argon. The solution was cooled to 0 °C and stirred for 15 min. Ketones B1–B7 (5 mmol) dissolved in dry THF (10 mL) were added dropwise and the reaction was continued for 2h at same temperature. After completion of reaction (TLC), the reaction was quenched with saturated NH$_4$Cl solution (30 mL), extracted into ethyl acetate (3×50 mL). The combined organics were dried over sodium sulfate, filtered, and concentrated in vacuo. The residue was purified by silica gel column chromatography to afford the alcohols 1a-1g.

6. General Scheme B to synthesize 1h and 1l:
7. General Method B to synthesize 1h and 1i:

4-Oxo-4-arylbutyric acid C (10 mmol) was dissolved in methanol (10 mL). Acetyl chloride (1.2 equiv.) was added dropwise and the reaction mixture was stirred at room temperature for overnight. After completion of the reaction (TLC), the reaction mixture was extracted into DCM (3 × 50 mL). The combined organics were washed with water (2×50 mL) and brine (1×50 mL); dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to obtain the corresponding methyl esters D. Esters D were used in the next step without further purification.

Ester D (5 mmol) dissolved in dry THF (10 mL) were added dropwise to a solution of RuCl(p-cymene)((S,S)-Ts-DPEN (5 mol%) in 5 : 2 formic acid / triethylamine (10 mL) under argon and stirred for 48 h at 30 °C oil bath. After completion of the reaction, the reaction was quenched with saturated NaHCO₃ solution (30 mL) and extracted into DCM (3×50 mL). The combined organic layers were washed with water (2×50 mL) and brine (1×50 mL); dried on anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography to obtain a non-separable mixture of alcohol (E) and lactone (F).

A mixture of E and F (approx. 5 mmol) were reduced by using LiAlH₄ (0.5 equiv.) in dry THF (20 mL) at room temperature to obtain the products 1h and 1i in quantitative yields.
8. General Scheme C for the synthesis of 1j, 1k, and 1l:

\[
\begin{align*}
\text{N} & \quad \text{i) R-MgBr, THF, rt-60 \degree C, 2h} \\
\text{Me} & \quad \text{ii) NaBH}_4, \text{MeOH, H}_2\text{O, 0 \degree C, 1h} \\
\text{A2} & \quad \text{R = CH=CH}_2, \text{G1, 56\%} \\
& \quad \text{R = Me, G2, 45\%} \\
& \quad \text{R = Et, G3, 55\%} \\
\text{CAL-B, vinyl acetate,} & \quad \text{rt, 12 h} \\
\text{OH} & \quad \text{R = CH=CH}_2, \text{H1, 45\%} \\
& \quad \text{R = Me, H2, 48\%} \\
& \quad \text{R = Et, H3, 45\%} \\
\text{OH} & \quad \text{R = CH=CH}_2, \text{I1, 40\%} \\
& \quad \text{R = Me, I2, 43\%} \\
& \quad \text{R = Et, I3, 45\%} \\
\text{K}_2\text{CO}_3, & \quad \text{MeOH, rt} \\
\text{OH} & \quad \text{R = CH=CH}_2, \text{1j, 90\%, er (S/R) = 98:2} \\
& \quad \text{R = Me, 1k, 95\%, er (R/S) = 98:2} \\
& \quad \text{R = Et, 1l, 93\%, er (R/S) = 99:1}
\end{align*}
\]

9. General Method C to synthesize 1j, 1k, and 1l:

Alcohols 1j, 1k and 1l were prepared by ring opening of lactam A2 with Grignard reagent. After completion by TLC, the following ketones were in situ reduced by NaBH$_4$ for 1 hour to obtain racemate alcohols. Crude reaction mixtures were purified by silica gel column chromatography to obtain G1, G2, and G3 in 56\%, 45\% and 55\% yields.

Alcohols G1, G2, and G3 were used to perform kinetic resolution with *Candida Antarctica* lipase-B (CAL-B) in the excess amount of vinyl acetate for 12 hours. After completion of the reaction, crude mixtures were purified by silica gel column chromatography to alcohols H1, H2, H3 in 45\%, 48\% and 45\% yields and acetylated products I1, I2, I3 in 40\%, 43\%, 45\% yields, respectively.
Acetylated compounds I1, I2, I3 were used to perform deprotection in the present of K2CO3 in MeOH for 2 hours. After completion by TLC, crude mixtures were purified by silica gel column chromatography to alcohols 1j, 1k, and 1l in 90%, 95% and 93% yields, respectively.

10. General Scheme D for the synthesis of 1m and 1n:

![Chemical Structure Diagram]

11. General Method D for the synthesis of 1m and 1n:

N-aryl lactam K1 and K2 were prepared following a similar procedure as described in general method 5. To a solution of γ-lactam J (30 mmol) in 30 mL dry DMF was added CuI (10 mol%), anhydrous K2CO3 (1.1 equiv.), and aryl bromide (2 equiv.). The reaction mixture was refluxed for 48 h. After completion of the reaction, the reaction mixture was allowed to attain room temperature. Aqueous saturated NH4Cl (50 ml) was then added and the aqueous layer was separated and extracted with ethyl acetate (4 × 50 ml). The combined organic phase were washed with brine (1×50 mL), dried over anhydrous Na2SO4 and concentrated under reduced pressure to give the crude product. Purification is carried out by usual silica gel column chromatography to afford pure K1 and K2.

A warm solution of N-aryl lactam K (10 mmol) in dry benzene (30 ml) was added slowly to a well stir solution of phenyl lithium (10 mmol) under argon atmosphere. The reaction mixture
was stirred at reflux for 2 h under argon atmosphere. Benzene and ice-water were added at ice
temperature. The combined organic phase was separated, washed with water, dried over
sodium sulfate and concentrated under reduced pressure. The crude residues (ketones, L1–L2)
were directly used without further purification for the CBS-reduction, after which the crude
reaction mixtures were purified by silica gel column chromatography to obtain pure alcohols
1m and 1n.

12. General Scheme E for the synthesis of 1o and 1p:

\[
\begin{align*}
\text{ArMgBr} & \quad \text{THF, 0 to rt }^\circ\text{C} \\
\text{HO} & \quad \text{X} = \text{H, M1, 38 }, \%
\text{M} & \quad \text{X} = \text{p-F, M2, 34 }, \%
\end{align*}
\]

13. General Method E for the synthesis of 1o and 1p:

An oven-dried round-bottomed flask equipped with a magnetic stir bar was charged with dry
THF (20 mL) and chroman-2-one (10 mmol) under argon atmosphere. The solution was
cooled to 0 °C and aryl magnesium bromide (1.1 equiv, in 4 mL THF) was added dropwise.
The reaction was allowed to attain room temperature and was run at the same temperature for
3 h. The reaction was quenched with saturated NH\textsubscript{4}Cl solution (30 mL) extracted into diethyl
ether (3×50 mL). The combined organics were dried over sodium sulfate, filtered, and
concentrated under reduced pressure. The crude (ketones, M1 and M2) was directly used
without further purification for the CBS-reduction after which the crude reaction mixtures
were purified by silica gel column chromatography to obtain pure 1o and 1p in 34% and 39%
overall yields respectively.
14. General Scheme F for the synthesis of enantiomerically enriched tertiary alcohols 3a, 3b, and 3c:

\[
\begin{align*}
\text{Acetophenone (1.0 g, 8.33 mmol) was added to a solution of RuCl(p-cymene)|(S,S)-Ts-DPEN (52.9 mg, 0.083 mmol, 1.0 mol %) in 5:2 formic acid / triethylamine (15 mL) under argon and stirred at 28 °C for 24 h. After completion of the reaction, saturated NaHCO}_3\text{ solution (50 mL) was added and stirred for another 15 min. The reaction mixture was extracted into DCM (3×50 mL). The combined organic layers were washed with water (2×50 mL) and brine (1×50 mL); dried over anhydrous MgSO}_4\text{ and concentrated under reduced pressure. Purification of the crude residue by column chromatography afforded pure alcohol N in 90 % yield.}
\end{align*}
\]

15. General Method F for the synthesis of enantiomerically enriched tertiary alcohols 3a, 3b and 3c:

**Step 1:** Preparation of enantiomerically enriched secondary benzylic alcohols N via Noyori's asymmetric reduction: Acetophenone (1.0 g, 8.33 mmol) was added to a solution of RuCl(p-cymene)|(S,S)-Ts-DPEN (52.9 mg, 0.083 mmol, 1.0 mol %) in 5:2 formic acid / triethylamine (15 mL) under argon and stirred at 28 °C for 24 h. After completion of the reaction, saturated NaHCO}_3\text{ solution (50 mL) was added and stirred for another 15 min. The reaction mixture was extracted into DCM (3×50 mL). The combined organic layers were washed with water (2×50 mL) and brine (1×50 mL); dried over anhydrous MgSO}_4\text{ and concentrated under reduced pressure. Purification of the crude residue by column chromatography afforded pure alcohol N in 90 % yield.
Step 2: The following procedure is representative of the preparation of secondary carbamates O from chiral secondary benzylic alcohol: A solution of alcohol N (5 mmol), diisopropylcarbamoyl chloride (1.1 equiv.), and triethyl amine (1.1 equiv.) in anhydrous DCM (30 mL) was refluxed for 24 h. After completion of the reaction (TLC), the reaction mixture was poured in water (50 mL). The mixture was extracted with diethyl ether (3 × 50 mL). The combined organic parts were washed with water (2×50 mL) and brine (1×50 mL); dried over anhydrous Na$_2$SO$_4$ and concentrated under reduced pressure. The crude product was purified by usual silica gel column chromatography to afford pure carbamate O.

Step 3: Lithiation/borylation of chiral secondary carbamates to tertiary allylic alcohol P:

To a stirred solution of (S)-1-Phenylethyl diisopropylcarbamate O (1 g, 4.01 mmol) in 20 mL anhydrous diethyl ether at −78 °C was added s-BuLi (3.4 mL of 1.4 M solution, 4.8 mmol, 1.2 equiv.) drop wise under an atmosphere of argon. The resulting light yellow homogeneous solution was stirred at −78 °C for 30 min and neat vinylboronic acid pinacol ester (1 mL, 6 mmol, 1.5 equiv.) was added drop wise with vigorous stirring. The reaction mixture was then stirred for 45 minutes at −78 °C. A methanol solution of magnesium bromide (6.0 mL, 6.0 mmol; 1M) was added dropwise under argon. The reaction mixture was stirred at −78 °C for an additional 15 min. and then allowed to attain room temperature and was run at the same temperature for 16 h. The reaction was quenched with the addition of an ice cold solution of 3 M aqueous sodium hydroxide (14.8 mL) and 30% aqueous H$_2$O$_2$ (8.5 mL) and stirred at room temperature for an additional 2 hours. The reaction mixture was extracted by Et$_2$O (3×50 mL). The combined organic layers were washed with water (1×50 mL) and brine (1×50 mL) and concentrated under reduced pressure. The crude product was purified by column chromatography to obtain tertiary allylic alcohol P (474.7 mg, 80%) as a colorless oil.

Step 4: Palladium-catalyzed vinylations of iodoanilines Q:
A mixture of Q (1.0 mmol), 2-phenylbut-3-en-2-ol P (5.0 mmol), Pd(OAc)$_2$ (0.10 mmol), and DPPF as ligand (0.20 mmol) in the presence of K$_2$CO$_3$ (1.5 equiv.) in toluene : H$_2$O (1:1, 2.0 mL) were heated with stirring in a sealed tube at the temperatures 100°C for 3 h. After completion of the reaction (TLC), saturated K$_2$CO$_3$ solution (30 mL) was added and the reaction mixture was extracted into ethyl acetate three times. The combined organic layers were washed with saturated NaCl (1×50 mL); dried over anhydrous Na$_2$SO$_4$ and concentrated under reduced pressure. The residue was purified by column chromatography to obtain a pure alcohol R.

**Step 3: Hydrogenation of a tertiary alcohol R:**

Tertiary alcohol R (1 mmol) was dissolved in ACN (10 mL) at room temperature. Pd/C (10 % wt) was added under argon atmosphere, and the reaction vessel was cooled to 0°C. Then, the atmosphere was substituted with H$_2$ (1 atm) and the reaction mixture was stirred at the same temperature for 1 h. After the completion of reaction (TLC), the mixture was filtered through a tight packed pad of Celite®. The filtrate was concentrated and purified via silica gel (100-200 mess) column chromatography to obtain pure 3a, 3b, and 3c.

16. **General Scheme G for the synthesis of 2-iodo-N-phenylaniline (Q):**

![General Scheme G]

17. **General Method G for the synthesis of 2-iodo-N-phenylaniline (Q):**

To a solution of 2-idoaniline (2.0 mmol, 438.04 mg) and 2-(Trimethylsilyl)phenyl trifluoromethanesulfonate (2.2 mmol, 656.37 mg) in acetonitrile (20 mL) was added CsF (4.0 mmol, 607.6 mg). The reaction was allowed to stir at room temperature for 12 h. After completion of the reaction (TLC), H$_2$O (10 mL) was added carefully and stirred for 15 min. The mixture was extracted with DCM (3×50 mL) and the combined organic layers were
washed with water (1×50 mL) and brine (1×50 mL); dried over anhydrous MgSO$_4$ and concentrated under reduced pressure. The residue was purified by column chromatography to obtain 2-iodo-N-phenylaniline (Q, 92.0%).

18. General Scheme H for the synthesis of dioxygen-centered nucleophiles 1h’

![Chemical structure](image)

19. General Method H for the synthesis of dioxygen-centered nucleophiles 1h’

A mixture of 2-bromoacetophenone (10 mmol, 199 mg), anhydrous K$_2$CO$_3$ (1 equiv. 138mg, 10 mmol), and methyl ethyl ketone (2 equiv. 144mg) in acetone (20 mL) was stirred at room temperature for 12 h. After completion of the reaction it was diluted with water, extracted in DCM, washed with water, brine and dried over the anhydrous sodium sulphate. The solvent was removed under vacuum. The crude product was recrystallized from 2-propanol gave pure compound dimethyl 2-(2-oxo-2-phenylethyl)malonate S (90 %, 225 mg). The compound S (approx. 8 mmol) was reduced by using LiAlH$_4$ (0.5 equiv.) in dry THF (20 mL) at room temperature to obtain the products (3-(hydroxymethyl)-1-phenylbutane-1,4-diol) 1h’ in quantitative yields.

20. General Scheme I for the synthesis of dinucleofuges 1h”

![Chemical structure](image)
21. General Method I for the synthesis of dinucleofuges 1h’’

Similar to the synthesis of di-O-centered nucleophiles 1h’’, a substitution reaction of ethyl bromoacetate with dibenzoylmethane generated ethyl 3-benzoyl-4-oxo-4-phenylbutanoate T, followed by then LiAlH₄ reduction to give 2-(hydroxy(phenyl)methyl)-1-phenylbutane-1,4-diol 1h’’. 
**Table S1: Optimization of reaction conditions for secondary benzylic alcohols**

![Diagram of reaction](image)

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalysts</th>
<th>Solvent</th>
<th>Temp (°C)</th>
<th>Yield (%) †</th>
<th>e.s. (%) ‡</th>
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<td>FeF₃(III)</td>
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<td>FeCl₂(II)</td>
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<td>NR</td>
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<td>Fe₄[Fe(CN)₆]₃</td>
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<td>NR</td>
<td>0</td>
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<td>10</td>
<td>93</td>
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<tr>
<td>9</td>
<td>FeCl₃</td>
<td>DCE</td>
<td>90</td>
<td>35</td>
<td>92</td>
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<td>NR</td>
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<td>Iron(III) tartrate</td>
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<tr>
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</tr>
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<td>NR</td>
<td>0</td>
</tr>
<tr>
<td>23</td>
<td>Fe(OTf)₃ + AgPF₆(10 mol %)</td>
<td>DCE</td>
<td>90</td>
<td>NR</td>
<td>0</td>
</tr>
<tr>
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<td>Fe(OTf)₃ + MS (3Å)</td>
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<td>98</td>
<td>99</td>
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<tr>
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<td>MS (3Å)</td>
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<td>NR</td>
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<tr>
<td>27</td>
<td>CF₃SO₃H (10 mol%)</td>
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<td>NR</td>
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<td>Without catalyst</td>
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<tr>
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<td>Cu(OTf)₂</td>
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<td>Co(OTf)₂</td>
<td>DCE</td>
<td>90</td>
<td>&lt;10</td>
<td>0</td>
</tr>
</tbody>
</table>

**Reaction condition:**  
*All reactions were performed using 0.5 mmol of 1a, 0.050 mmol of catalyst (10 mol %) in DCE as solvent (2.0 mL), MS (3Å) = 300 mg, at 90 °C temperature for time 24 h under argon atmosphere.  
†NMR yield. ‡Enantiospecificity was determined by chiral stationary phase HPLC analysis.  
§The purity of catalyst has been determined by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) analysis. NR, no reaction.

---

22. Table S1: Optimization of reaction conditions for secondary benzylic alcohols
23. Table S2: Optimization of reaction conditions for tertiary alcohols

\[
\begin{array}{cccccc}
\text{Entry} & \text{Solvent (mL)} & \text{Temp (°C)} & \text{Time (h)} & \text{Yield (%)†} & \text{e.s. (%)‡} \\
1 & \text{DCE} & 90 & 24 & 100 & 09 \\
2 & \text{n-Hexane} & 90 & 24 & 100 & 26.8 \\
3 & \text{DCE + n-Hexane (0.5+0.5)} & 90 & 24 & 100 & 40 \\
4 & \text{DCE + n-Hexane (0.4+0.6)} & 90 & 24 & 100 & 32.2 \\
5 & \text{DCE + n-Hexane (0.3+0.7)} & 90 & 24 & 100 & 32.2 \\
6 & \text{DCE + n-Hexane (0.2+0.8)} & 90 & 24 & 100 & 29 \\
7 & \text{DCE + n-Hexane (0.25+0.25)} & 90 & 24 & 100 & 36 \\
8 & \text{DCE + n-Hexane (01+01)} & 90 & 24 & 100 & 35 \\
9 & \text{DCE + n-Hexane (0.5+0.5)} & 80 & 24 & 100 & 50.53 \\
10 & \text{DCE + n-Hexane (0.5+0.5)} & 60 & 24 & 100 & 64 \\
11 & \text{DCE + n-Hexane (0.5+0.5)} & \text{rt} & 48 & 98 & 96 \\
\end{array}
\]

**Reaction condition** †All reactions were performed using 0.2 mmol of 3a, MS (3Å) = 100 mg, and 0.020 mmol of catalyst (10 mol %) in the indicated solvent (01 mL) under argon atmosphere. ‡NMR yield. ‡Enantiomeric excess was determined by chiral stationary phase HPLC analysis. NR, no reaction.

24. Inductively Coupled Plasma Mass Spectrometry analysis of Fe(OTf)₃ catalyst

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) was used for detecting trace elemental impurities in the Fe(OTf)₃ catalyst (purity 90.00%, Table S1). The major metal impurities were individually screened as catalysts for the transformation (entry 29-32, Table S1). However, none of the trace metals outperformed Fe(OTf)₃ as catalyst in the intramolecular substitution reaction.

25. Rate order determination

The reaction of 1a to 2a was performed using five different concentrations of catalyst (0, 5, 10, 15, and 20 mol%). The reactions were monitored by using ¹H NMR spectroscopy and the initial rates were determined below 20% conversion. Duplicates of the reactions were made and the data is the mean value of these duplicates.
**Fig S1**: Rate order determination

**Reaction condition**: 1a (0.2 mmol), DCE (1 mL), MS (3 Å) (200 mg), and catalyst (0, 5, 10, 15, and 20 mol%) were heated in an oil bath at 90° C. Initial rates of the reaction were determined below 20% conversion (up to 2h) by $^1$H NMR spectroscopy. The values are the mean value of two reactions.

**26. In-situ UV-visible spectroscopy analysis**

*N*-methyl anisole (1r), represent an *N*-centered nucleophile exhibits the absorption bands at 320 cm$^{-1}$ (Fig. 2) in UV-vis spectrum in DCE. When Fe is added a blue shift to 275 cm$^{-1}$ is observed. A similar trend is found in the UV–vis spectrum for the substrate 1a (Fig. 3).

**Fig. S2**: Interaction of Fe(OTf)$_3$ with nucleophile (1r)
Fig.S3: Interaction of Fe(OTf)$_3$ with 1a

27. ESI-MS/MS of intermediate of the standard reaction

Fig.S4: ESI-MS/MS of intermediate of the reaction

28. Characterization data of all starting alcohols:

All characterization data for alcohols 1a, 1c, 1d, 1e, 1f, 1g, 1j, 1k, 1l, 1m, 1n, 1o, 1p, 3a, 3b, and 3c which are not reported previously, are supplemented below. Alcohols 1b, 1h, and 1i were previously reported and the obtained NMR data (see copies of NMR attached below) matched with the reported values.
(S)-4-((4-methoxyphenyl)amino)-1-phenylbutan-1-ol (1a)\(^1\)

![](image)

**IR** (neat) 3360.64, 3028, 2932.25, 2831, 1617, 1512.40, 1455, 1296, 1235.54, 1178, 1119, 1119.18, 1034.64, 913, 819, 749, 701 cm\(^{-1}\). **\(^1\)H NMR** (400 MHz, Chloroform-d) \(\delta = 7.37 – 7.33\) (m, 4H), \(7.30 – 7.27\) (m, 1H), 6.78 (d, \(J = 9.0\) Hz, 2H), 6.61 (d, \(J = 8.9\) Hz, 2H), 4.72 (dd, \(J = 7.5, 5.3\) Hz, 1H), 3.75 (s, 3H), 3.11 (t, \(J = 6.8\) Hz, 2H), 1.89 (dddd, \(J = 10.2, 8.4, 6.7, 5.7\) Hz, 1H), 1.81 – 1.71 (m, 1H), 1.70 – 1.60 (m, 2H) ppm. **\(^13\)C NMR** (100 MHz, CDCl\(_3\)) \(\delta = 152.5, 144.6, 142.0, 128.5, 127.6, 125.8, 114.9, 114.7, 74.3, 55.8, 45.4, 36.8, 26.0\) ppm. **HRMS (ESI)** calcd. for C\(_{17}\)H\(_{22}\)NO\(_2\) [M+H] \(m/z\) 272.1572 found \(m/z\) 272.1645. The enantiomeric ratio of 1a was determined by HPLC analysis using Daicel Chiralcel OD-H column: \(n\)-Hexane: isopropanol = 90:10, flow rate 1.0 mL/min, \(\lambda = 254\) nm (channel 1), 232 nm (channel 2): \(t_1\) (major) = 25.45 min, \(t_2\) (minor) = 30.12 min.

(S)-1-phenyl-4-(phenylamino)butan-1-ol (1b)\(^1\)

![](image)

**IR** (neat) 3354.64, 3029, 2930.25, 2835, 1615, 1510.40, 1465, 1316, 1145.54, 1178, 1119.18, 1034.64, 911, 819, 750, 711 cm\(^{-1}\). **\(^1\)H NMR** (400 MHz, Chloroform-d) \(\delta = 7.36\) (d, \(J = 4.3\) Hz, 4H), \(7.32 – 7.25\) (m, 1H), \(7.21 – 7.12\) (m, 2H), 6.70 (tt, \(J = 7.3, 1.1\) Hz, 1H), \(6.63 – 6.54\) (m, 2H), 4.73 (dd, \(J = 7.5, 5.4\) Hz, 1H), 3.14 (t, \(J = 6.9\) Hz, 2H), 1.97 – 1.82 (m, 2H), 1.81 – 1.72 (m, 1H), 1.69 – 1.60 (m, 1H). **\(^13\)C NMR** (101 MHz, CDCl\(_3\)) \(\delta = 148.3, 144.5, 129.2, 128.5, 127.6, 125.8, 117.4, 112.9, 74.3, 43.9, 36.6, 25.9\) ppm. **HRMS (ESI)** calcd. for C\(_{16}\)H\(_{20}\)NO [M+H] \(m/z\) 242.1548 found \(m/z\) 242.1545. The enantiomeric ratio of 1b was determined by HPLC analysis using Daicel Chiralcel OD-H column: \(n\)-Hexane: isopropanol = 90:10, flow
rate 1.0 mL/min, \( \lambda = 254 \text{ nm (channel 1)}, 232 \text{ nm (channel 2)} \): \( t_1 \) (major) = 22.1 min, \( t_2 \) (minor) = 32.8 min.

(S)-1-(4-fluorophenyl)-4-((4-methoxyphenyl)amino)butan-1-ol (1c)

\[
\begin{array}{c}
\text{IR (neat) } 3367.48, 2994, 2935.19, 2834.38, 1603, 1511.78, 1464, 1386, 1235.4, 1179, 1092.8, 1035.34, 821.49, 755, 718, 574 \text{ cm}^{-1}. \\
^1\text{H NMR} \ (400 \text{ MHz, Chloroform-}d) \ \delta = 7.36 – 7.31 \text{ (m, 4H), 7.26 (d, 1H), 4.89 (t, } J = 7.2 \text{ Hz, 1H), 4.14 – 4.06 \text{ (m, 1H), 3.98 – 3.89 \text{ (m, 1H), 2.38 – 2.27 \text{ (m, 1H), 2.05 – 1.96 \text{ (m, 1H), 1.86 – 1.76 \text{ (m, 1H) ppm.}}) \text{ C NMR} \ (100 \text{ MHz, CDCl}_3) \ \delta = 162.3 \ (J_{C:F} = 320 \text{ Hz), 152.8, 141.6, 127.5 \ (J_{C:F} = 10 \text{ Hz), 115.3 \ (J_{C:F} = 20 \text{ Hz), 115.1, 114.9, 73.6, 55.8, 45.6, 37.0, 25.9 \text{ ppm. HRMS (ESI) calcd. for C}_{17}H_{21}FNO_2 [M+H]^+ m/z 290.1569 found m/z 290.1551. The enantiomeric ratio of 1c was determined by HPLC analysis using Daicel Chiralcel OD-H column: n-Hexane: isopropanol = 90:10, flow rate 1.0 mL/min, \( \lambda = 254 \text{ nm (channel 1), 232 nm (channel 2)} \): \( t_1 \) (major) = 22.81 min, \( t_2 \) (minor) = 28.03 min.}
\end{array}
\]

(S)-1-(4-chlorophenyl)-4-((4-methoxyphenyl)amino)butan-1-ol (1d)

\[
\begin{array}{c}
\text{IR (neat) } 3370.58, 2935, 2830.34, 1616, 1512.28, 1463, 1365, 1295, 1237.3, 1179, 1088.9, 1036.44, 818.9, 770, 702, 475 \text{ cm}^{-1}. \ \text{H NMR} \ (400 \text{ MHz, Chloroform-}d) \ \delta = 7.29 \ (d, J = 5.5 \text{ Hz, 4H), 6.81 – 6.75 \text{ (m, 2H), 6.66 – 6.56 \text{ (m, 2H), 4.70 (dd, } J = 7.4, 5.3 \text{ Hz, 1H), 3.75 (s, 3H), 3.10 (s, 2H), 1.89 – 1.79 \text{ (m, 2H), 1.77 – 1.62 \text{ (m, 2H) ppm. \text{ C NMR (100 MHz,}})
\end{array}
\]
CDCl$_3$ δ = 152.8, 141.6, 127.5, 127.4, 115.4, 115.2, 115.1, 114.9, 73.6, 55.8, 45.6, 37.0, 25.9 ppm. **HRMS (ESI)** calcd. for C$_{17}$H$_{21}$ClNO$_2$ [M+H] $m/z$ 306.1267 found $m/z$ 306.1255. The enantiomeric ratio of 1d was determined by HPLC analysis using Daicel Chiralcel OD-H column: *n*-Hexane: isopropanol = 90:10, flow rate 1.0 mL/min, $\lambda$ = 254 nm (channel 1), 232 nm (channel 2): t$_1$ (major) = 45.3 min, t$_2$ (minor) = 53.7 min.

(S)-4-((4-methoxyphenyl)amino)-1-(3-(trifluoromethoxy)phenyl)butan-1-ol (1e)

![Chemical structure of 1e](image)

**IR** (neat) 3371.68, 2934, 2831.44, 1606, 1513.81, 1465, 1360, 1190, 1240.71, 1201, 1080.1, 811.9, 765, 701, 470 cm$^{-1}$. **$^1$H NMR** (400 MHz, Chloroform-$d$) δ = 7.36 (t, $J$ = 7.9 Hz, 1H), 7.26 (s, 1H), 7.23 (s, 1H), 7.15 – 7.10 (m, 1H), 6.81 – 6.74 (m, 2H), 6.65 – 6.56 (m, 2H), 4.76 (t, $J$ = 6.3 Hz, 1H), 3.75 (s, 3H), 3.12 (td, $J$ = 6.7, 1.7 Hz, 2H), 1.87 (dd, $J$ = 7.3, 6.2 Hz, 2H), 1.78 – 1.67 (m, 1H) ppm. **$^{13}$C NMR** (101 MHz, CDCl$_3$) δ = 152.5, 149.4, 147.2, 142.2, 129.7, 124.1, 121.7, 119.7, 119.2, 118.3, 114.9, 114.8, 73.5, 55.8, 45.2, 37.1, 26.0 ppm. **HRMS (ESI)** calcd. for C$_{18}$H$_{21}$F$_3$NO$_3$ [M+H] $m/z$ 356.1395 found $m/z$ 356.1385. The enantiomeric ratio of 1e was determined by HPLC analysis using Daicel Chiralcel OD-H column: *n*-Hexane: isopropanol = 90:10, flow rate 1.0 mL/min, $\lambda$ = 254 nm (channel 1), 232 nm (channel 2): t$_1$ (major) = 16.01 min, t$_2$ (minor) = 21.1 min.

(S)-4-((4-methoxyphenyl)amino)-1-(p-tolyl)butan-1-ol (1f)

![Chemical structure of 1f](image)
IR (neat) 3361.8, 2937, 2830.86, 1614, 1511.98, 1462, 1293, 1235, 1179, 1119.8, 1035.67, 818.6, 518.77 cm\(^{-1}\). \(^1\)H NMR (400 MHz, Chloroform-\(d\)) \(\delta = 7.24\) (d, \(J = 8.1\) Hz, 2H), 7.17 – 7.13 (m, 2H), 6.80 – 6.75 (m, 2H), 6.67 – 6.60 (m, 2H), 4.68 (dd, \(J = 7.5, 5.2\) Hz, 1H), 3.75 (s, 3H), 3.14 – 3.07 (m, 3H), 2.34 (s, 3H), 1.94 – 1.81 (m, 2H), 1.78 – 1.71 (m, 1H), 1.65 (dt, \(J = 9.1, 6.2\) Hz, 1H) ppm. \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta = 152.7, 141.6, 137.2, 129.1, 125.8, 115.1, 114.9, 99.9, 74.1, 55.8, 45.7, 36.7, 25.9, 21.1\) ppm. HRMS (ESI) calcd. for C\(_{18}\)H\(_{23}\)NO\(_2\)Na [M+Na]\(^+\) \(m/z\) 308.1618 found \(m/z\) 308.1621. The enantiomeric ratio of 1f was determined by HPLC analysis using Daicel Chiralcel AD column: \(n\)-Hexane : isopropanol = 90:10, flow rate 1.0 mL/min, \(\lambda = 254\) nm (channel 1), 232 nm (channel 2): \(t_1\) (major) = 28.8 min, \(t_2\) (minor) = 29.6 min.

(S)-1-(3-methoxyphenyl)-4-((4-methoxyphenyl)amino)butan-1-ol (1g)

IR (neat) 3370, 3030, 2831, 1616, 1505, 1468, 1440, 1417, 1311, 1267, 1170, 1118, 1094, 1030, 817, 755 cm\(^{-1}\). \(^1\)H NMR (400 MHz, Chloroform-\(d\)) \(\delta = 7.30 – 7.22\) (m, 1H), 6.92 (dd, \(J = 4.2, 1.8\) Hz, 2H), 6.81 (ddd, \(J = 8.3, 2.6, 1.1\) Hz, 1H), 6.79 – 6.75 (m, 2H), 6.59 (d, \(J = 8.9\) Hz, 2H), 4.68 (dd, \(J = 7.2, 5.5\) Hz, 1H), 3.81 (s, 3H), 3.74 (s, 3H), 3.09 (t, \(J = 6.8\) Hz, 2H), 1.94 – 1.81 (m, 2H), 1.78 – 1.70 (m, 1H), 1.69 – 1.61 (m, 1H) ppm. \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta = 159.7, 152.4, 146.4, 129.4, 118.1, 114.9, 114.8, 114.6, 112.9, 111.3, 74.1, 55.8, 55.2, 45.2, 36.8, 25.9 ppm. HRMS (ESI) calcd. for C\(_{18}\)H\(_{24}\)NO\(_3\)M+ [M+H]\(^+\) \(m/z\) 302.1752 found \(m/z\) 302.1751. The enantiomeric ratio of 1g was determined by HPLC analysis using Daicel Chiralcel AD column: \(n\)-Hexane: isopropanol = 90:10, flow rate 1.0 mL/min, \(\lambda = 254\) nm (channel 1), 232 nm (channel 2): \(t_1\) (major) = 42.5 min, \(t_2\) (minor) = 48.9 min.
(S)-1-phenylbutane-1,4-diol (1h)²

\[ \text{1H NMR (400 MHz, Chloroform-}d\text{) } \delta = 7.39 - 7.32 (m, 4H), 7.28 (d, } J = 5.7 \text{ Hz, 1H), 4.74 (t, } J = 6.3 \text{ Hz, 1H), 3.72 - 3.66 (m, 2H), 1.87 (td, } J = 7.0, 5.9 \text{ Hz, 2H), 1.74 - 1.63 (m, 2H) ppm.} \]
\[ \text{13C NMR (100 MHz, CDCl}_3\text{) } \delta = 144.7, 128.4, 127.5, 125.8, 74.3, 62.8, 36.2, 29.2. \]
\[ \text{HRMS (ESI) calcd. for } C_{10}H_{15}O_2 \text{ [M+H]} m/z 167.1070 \text{ found } m/z 167.1076. \]

The enantiomeric ratio of 1h was determined by HPLC analysis using Daicel Chiralcel OD-H column: \( n \)-Hexane: isopropanol = 95:5, flow rate 0.5 mL/min, \( \lambda = 254 \) nm: \( t_1 \) (minor) = 44.3 min, \( t_2 \) (major) = 48.4 min.

(S)-1-(4-fluorophenyl)butane-1,4-diol (1i)³

\[ \text{1H NMR (400 MHz, Chloroform-}d\text{) } \delta = 7.28 \text{ (ddd, } J = 8.0, 5.1, 2.3 \text{ Hz, 2H), 7.07 - 6.94 (m, 2H), 4.66 (t, } J = 6.3 \text{ Hz, 1H), 3.70 - 3.55 (m, 2H), 3.08 (s, 2H), 1.80 (q, } J = 7.1 \text{ Hz, 2H), 1.69 - 1.56 (m, 2H).} \]
\[ \text{13C NMR (101 MHz, CDCl}_3\text{) } \delta = 162.0 \text{ (} J_{C,F} = 244 \text{ Hz), 140.4, 140.4, 127.3 (} J_{C,F} = 8 \text{ Hz), 115.2 (} J_{C,F} = 20 \text{ Hz), 73.6, 62.7, 36.5, 29.0. ppm HRMS (ESI) calcd. for } C_{10}H_{13}FNaO_2 \text{ [M+Na]} m/z 207.0797 \text{ found } m/z 207.0797. \]

The enantiomeric ratio of 1i was determined by HPLC analysis using Daicel Chiralcel AD column: \( n \)-Hexane: isopropanol = 95:5, flow rate 1.0 mL/min, \( \lambda = 254 \) nm (channel 1), 232 nm (channel 2): \( t_1 \) (major) = 111.0 min, \( t_2 \) (minor) = 118.4 min.
(S)-6-((4-methoxyphenyl)amino)hexan-3-ol (1j)

\[
\text{IR (neat) } \text{cm}^{-1} 3382, 3030, 2931, 2870, 1661, 1614, 1510, 1451, 1354, 1311, 1217, 1170, 1118, 1040, 1036, 917, 754 \text{ cm}^{-1}.
\]

\[^{1}\text{H NMR}\] (400 MHz, Chloroform-d) \( \delta = 6.85 – 6.74 \) (m, 2H), 6.69 – 6.56 (m, 2H), 5.88 (ddd, \( J = 17.2, 10.4, 6.1 \) Hz, 1H), 5.24 (dt, \( J = 17.2, 1.5 \) Hz, 1H), 5.12 (dt, \( J = 10.4, 1.4 \) Hz, 1H), 4.13 (dtd, \( J = 5.9, 4.7, 1.3 \) Hz, 1H), 3.76 (s, 3H), 3.16 – 3.02 (m, 4H), 1.81 – 1.57 (m, 4H) ppm.

\[^{13}\text{C NMR}\] (100 MHz, CDCl\textsubscript{3}) \( \delta = 152.1, 142.4, 141.0, 114.8, 114.4, 72.5, 55.7, 45.0, 34.6, 25.4 \) ppm. HRMS (ESI) calcd. for C\textsubscript{13}H\textsubscript{20}NO\textsubscript{2} [M+H] 222.1489 \textit{m/z} found 222.1498 \textit{m/z}. The enantiomeric ratio of 1j was determined by HPLC analysis using Daicel Chiralcel OJ-H column: \textit{n-Hexane}: isopropanol = 90:10, flow rate 1.0 mL/min, \( \lambda = 254 \) nm (channel 1), 232 nm (channel 2): \( t_1 \) (major) = 46.3 min, \( t_2 \) (minor) = 49.7 min.

(R)-5-((4-methoxyphenyl)amino)pentan-2-ol (1k)

\[^{1}\text{H NMR}\] (400 MHz, Chloroform-d) \( \delta = 6.83 – 6.76 \) (m, 2H), 6.65 – 6.59 (m, 2H), 3.90 – 3.81 (m, 1H), 3.77 (s, 3H), 3.11 (td, \( J = 6.8, 2.0 \) Hz, 2H), 1.82 – 1.64 (m, 2H), 1.57 (tdd, \( J = 8.3, 5.9, 3.8 \) Hz, 2H), 1.22 (d, \( J = 6.2 \) Hz, 3H) ppm. \[^{13}\text{C NMR}\] (100 MHz, CDCl\textsubscript{3}) \( \delta = 152.2, 142.5, 114.8, 114.4, 72.7, 55.7, 45.2, 36.9, 26.0, 23.6 \) ppm.

The enantiomeric ratio of 1k was determined by HPLC analysis using Daicel Chiralcel OJ-H column: \textit{n-Hexane}: isopropanol = 85:15, flow rate 0.5 mL/min, \( \lambda = 254 \) nm (channel 1), 232 nm (channel 2): \( t_1 \) (major) = 58.9 min, \( t_2 \) (minor) = 63.2 min.
(R)-6-((4-methoxyphenyl)amino)hex-1-en-3-ol (1l)

\[
\text{IR (neat) cm}^{-1} \quad 3382, 3029, 2935, 2874, 2833, 1660, 1615, 1513.49, 1456.9, 1385, 1238.21, 1179, 1111, 1036.69, 969, 819.74, 753 \text{ cm}^{-1}.
\]
\[
\text{H NMR (400 MHz, Chloroform-}d\text{)} \quad \delta = 6.84 – 6.74 (m, 2H), 6.69 – 6.57 (m, 2H), 3.77 (s, 3H), 3.56 (dddd, \text{ } J = 8.5, 7.5, 4.9, 3.7 \text{ Hz, 1H}), 3.19 – 3.02 (m, 2H), 2.86 (s, 2H), 1.81 – 1.39 (m, 6H), 0.96 (t, \text{ } J = 7.5 \text{ Hz, 3H}) \text{ ppm.}
\]
\[
\text{C NMR (100 MHz, CDCl}_3\text{)} \quad \delta = 152.0, 142.3, 114.6, 114.4, 72.9, 55.6, 45.2, 34.6, 30.2, 25.9, 10.0 \text{ ppm.}
\]
\[
\text{HRMS (ESI) calcd. For C}_{13}\text{H}_{22}\text{NO}_2 [M+H]} m/z 224.1645 \text{ found 224.2652 m/z.}
\]

The enantiomeric ratio of 1l was determined by HPLC analysis using Daicel Chiralcel OJ-H column: 

\[
\text{IR (neat) cm}^{-1} \quad 3362.14, 3029, 2935.27, 2833, 1611, 1522.80, 1450, 1206, 1230.44, 1186, 1121, 1044.51, 911, 820, 789 \text{ cm}^{-1}.
\]
\[
\text{H NMR (400 MHz, Chloroform-}d\text{)} \quad \delta = 7.39 – 7.31 (m, 4H), 7.29 (dd, \text{ } J = 6.3, 2.3 \text{ Hz, 1H}), 6.81 – 6.73 (m, 2H), 6.59 – 6.52 (m, 2H), 4.72 – 4.61 (m, 1H), 3.74 (s, 3H), 3.05 (t, \text{ } J = 7.0 \text{ Hz, 2H}), 1.91 – 1.80 (m, 1H), 1.80 – 1.70 (m, 1H), 1.68 – 1.58 (m, 2H), 1.57 – 1.48 (m, 1H), 1.40 (ddd, \text{ } J = 10.3, 7.9, 5.5 \text{ Hz, 1H}) \text{ ppm.}
\]
\[
\text{C NMR (100 MHz, CDCl}_3\text{)} \quad \delta = 152.0, 144.7, 142.6, 128.5, 127.6, 125.8, 114.9, 114.1, 74.5, 55.8, 44.8, 38.8, 29.5, 23.4 \text{ ppm.}
\]
\[
\text{HRMS (ESI) calcd. For C}_{15}\text{H}_{24}\text{NO}_2 [M+H]} m/z 286.1811 \text{ found m/z 286.1807.}
\]
The enantiomeric ratio of 1m was determined by HPLC analysis using Daicel
Chiralcel OD-H column: *n*-Hexane: isopropanol = 90:10, flow rate 1.0 mL/min, $\lambda = 254$ nm (channel 1), 232 nm (channel 2): $t_1$ (minor) = 29.23 min, $t_2$ (major) = 31.38 min.

**(S)-3-(2-((4-methoxyphenyl)amino)phenyl)-1-phenylpropan-1-ol (1n)*

![Chemical structure of 1n]

**IR** (neat) 3366.44, 3031, 2945.55, 2823, 1615, 1520.79, 1441, 1216, 1202.40, 1184, 1116, 1040.50, 916, 821, 780, 711, 498 cm$^{-1}$. **$^1$H NMR** (400 MHz, Chloroform-*$d$) $\delta = 7.34$ (m, 4H), 7.31 – 7.26 (m, 1H), 7.18 – 7.14 (m, 1H), 7.13 – 7.05 (m, 2H), 7.01 – 6.94 (m, 2H), 6.89 – 6.82 (m, 3H), 4.68 (dd, $J = 8.7$, 4.4 Hz, 0H), 3.80 (s, 1H), 2.82 – 2.68 (m, 1H), 2.16 – 1.99 (m, 0H) pmp. **$^{13}$C NMR** (101 MHz, CDCl$_3$) $\delta = 154.8$, 144.4, 142.9, 137.0, 130.0, 129.7, 128.5, 127.7, 126.9, 125.8, 121.3, 120.5, 116.8, 114.7, 73.4, 55.6, 39.0, 27.1 ppm. **HRMS (ESI)** calcd. for C$_{22}$H$_{23}$NO$_2$Na [M+Na] $m/z$ 356.1621 found $m/z$ 356.1631.

The enantiomeric ratio of 1n was determined by HPLC analysis using Daicel Chiralcel OD-H column: *n*-Hexane: isopropanol = 90:10, flow rate 1.0 mL/min, $\lambda = 254$ nm (channel 1), 232 nm (channel 2): $t_1$ (major) = 26.2 min, $t_2$ (minor) = 46.6 min.

**(S)-2-(3-hydroxy-3-phenylpropyl)phenol (1o)*

![Chemical structure of 1o]

$^1$H NMR (400 MHz, Chloroform-$d$) $\delta = 7.41 – 7.27$ (m, 6H), 7.21 – 7.12 (m, 2H), 6.91 (td, $J = 7.5$, 1.4 Hz, 2H), 4.66 (dd, $J = 10.3$, 3.6 Hz, 1H), 2.99 (ddd, $J = 14.2$, 10.6, 6.1 Hz, 1H),
2.77 (dd, $J = 14.3$, 6.5, 4.1 Hz, 1H), 2.51 (d, $J = 35.1$ Hz, 1H), 2.14 (dddd, $J = 14.3$, 10.3, 6.1, 4.1 Hz, 1H), 2.05 – 1.90 (m, 1H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta = 154.6$, 143.9, 130.5, 128.6, 127.9, 127.7, 127.1, 125.8, 120.8, 116.2, 73.1, 39.3, 25.9 ppm.

The enantiomeric ratio of 1o was determined by HPLC analysis using Daicel Chiralcel OJ-H column: $n$-Hexane: isopropanol = 90:10, flow rate 0.5 mL/min, $\lambda = 254$ nm (channel 1), 232 nm (channel 2): $t_1$ (major) = 43.8 min, $t_2$ (minor) = 46.4 min.

(S)-2-(3-(4-fluorophenyl)-3-hydroxypropyl)phenol (1p)

![Chemical structure](image)

IR (neat) 3357, 2934.80, 2836.60, 1622, 1555, 1513, 1473, 1464, 1237, 1179, 1116, 1035, 992, 922, 821 cm$^{-1}$. $^1$H NMR (400 MHz, Chloroform-d) $\delta = 7.34 - 7.27$ (m, 2H), 7.15 (ddd, $J = 8.5$, 7.0, 1.6 Hz, 2H), 7.08 – 6.98 (m, 2H), 6.95 – 6.83 (m, 2H), 4.63 (dd, $J = 10.2$, 3.6 Hz, 1H), 2.95 (ddd, $J = 14.1$, 10.3, 6.3 Hz, 1H), 2.75 (ddd, $J = 14.3$, 6.7, 4.2 Hz, 2H), 2.10 (ddddd, $J = 14.3$, 10.4, 6.2, 4.2 Hz, 1H), 1.95 (ddddd, $J = 14.0$, 10.3, 6.7, 3.6 Hz, 1H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta = 162.3$ ($J_{C-F} = 240$ Hz), 154.4, 139.7, 139.7, 130.6, 127.7, 127.5 ($J_{C-F} = 10$ Hz), 127.1, 120.8, 116.1, 115.4 ($J_{C-F} = 20$ Hz), 72.4, 39.4, 25.9 ppm. HRMS (ESI) calcd. for C$_{15}$H$_{15}$FO$_2$Na [M+Na] $269.0948$ m/z found 269.0973 m/z. The enantiomeric ratio of 1p was determined by HPLC analysis using Daicel Chiralcel AD column: $n$-Hexane : isopropanol = 90:10, flow rate 1.0 mL/min, $\lambda = 254$ nm (channel 1), 232 nm (channel 2): $t_1$ (minor) = 12.3 min, $t_2$ (major) = 16.2 min.
(S)-2-phenyl-4-(2-(phenylamino)phenyl)butan-2-ol (3a)$^5$

\[
\text{IR (neat) 3355, 3141, 2901.89, 2853.68, 1609.54, 1515.43, 1389.12, 1360, 1256, 1223.34, 1189.64, 1155, 1015, 816, 711, 459 \text{ cm}^{-1}. \text{ } ^1\text{H NMR (400 MHz, Chloroform-}d\text{)} \delta = 7.47 – 7.42 \text{ (m, 2H), 7.39 – 7.33 (m, 2H), 7.29 – 7.26 (m, 1H), 7.25 – 7.21 (m, 2H), 7.15 – 7.08 (m, 2H), 6.96 – 6.86 (m, 5H), 2.66 – 2.57 (m, 1H), 2.40 (ddd, } J = 14.0, 11.0, 5.1 \text{ Hz, 1H), 2.18 – 2.00 (m, 1H), 1.59 \text{ (s, 3H) ppm. } ^{13}\text{C NMR (100 MHz, CDCl}_3\text{)} \delta = 147.2, 144.2, 140.5, 132.3, 129.9, 129.2, 128.3, 126.8, 126.7, 124.7, 121.8, 120.2, 119.2, 117.2, 74.8, 44.0, 30.6, 26.2 \text{ ppm. HRMS (ESI) calcd. for C}_{22}\text{H}_{23}\text{NONa }[\text{M+Na}] m/z 340.1671 \text{ found } m/z 340.1672. \text{ The enantiomeric ratio of } 3a \text{ was determined by HPLC analysis using Daicel Chiralcel OD-H column: } n-\text{Hexane: isopropanol = 90:10, flow rate 1.0 mL/min, } \lambda = 254 \text{ nm (channel 1), 232 nm (channel 2): } t_1 \text{ (minor) = 8.7 min, } t_2 \text{ (major) = 10.1 min.}
\]

(S)-2-(3-hydroxy-3-phenylbutyl)phenol (3b)$^5$

\[
\text{IR (neat) 3361.83, 3058., 3028, 2975, 2929, 1582.51, 1489, 1455.94, 1374, 1243, 1218, 1119, 1065, 1029, 944, 890, 753.71, 699, 548 \text{ cm}^{-1}. \text{ } ^1\text{H NMR (400 MHz, Chloroform-}d\text{)} \delta = 7.54 – 7.44 \text{ (m, 2H), 7.37 (dd, } J = 8.5, 6.9 \text{ Hz, 2H), 7.30 – 7.23 (m, 1H), 7.11 – 7.05 (m, 1H), 7.00 (dd, } J = 7.4, 1.8 \text{ Hz, 1H), 6.87 – 6.75 (m, 2H), 6.57 \text{ (s, 1H), 2.65 (td, } J = 9.4, 4.8 \text{ Hz, 1H), 2.46 (ddt, } J = 15.8, 9.4, 4.7 \text{ Hz, 1H), 2.21 (ddd, } J = 14.3, 9.4, 6.2 \text{ Hz, 1H), 2.07 (ddd, } J = 14.5, 9.4, 5.5 \text{ Hz, 1H), 1.63 \text{ (s, 3H). } ^{13}\text{C NMR (101 MHz, CDCl}_3\text{)} \delta = 153.83, 147.10, 130.01,}
\]
128.32, 127.46, 126.79, 125.44, 124.67, 120.38, 115.95, 75.47, 43.86, 30.49, 24.76 ppm.

**HRMS (ESI)** calcd. for C_{16}H_{18}O_{2}Na [M+Na] m/z 265.1211 found m/z 265.1199.

The enantiomeric ratio of 3b was determined by HPLC analysis using Daicel Chiralcel OD-H column: *n*-Hexane: isopropanol = 90:10, flow rate 1.0 mL/min, λ = 254 nm (channel 1), 232 nm (channel 2): t_{1} (minor) = 11.1 min, t_{2} (major) = 12.8 min.

**(R)-2-(3-hydroxy-3,7-dimethylloctyl)phenol (3c)**

![Structure of 3c]

**IR** (neat) 3338.03, 3071, 3036, 2953, 2868, 1593.65, 1490, 1457.54, 1366, 1243, 1175, 1089, 1041, 912, 847, 751.42 cm\(^{-1}\). **\(^1\)H NMR** (400 MHz, Chloroform-\(d\)) δ = 7.09 – 6.97 (m, 2H), 6.82 – 6.72 (m, 2H), 2.71 – 2.56 (m, 2H), 1.74 – 1.66 (m, 2H), 1.53 – 1.38 (m, 3H), 1.31 – 1.22 (m, 2H), 1.18 (s, 3H), 1.10 (dd, J = 7.7, 1.1 Hz, 2H), 0.80 (d, J = 6.6 Hz, 6H) ppm. **\(^{13}\)C NMR** (101 MHz, CDCl\(_3\)) δ = 154.0, 129.9, 128.8, 127.4, 120.1, 116.2, 73.9, 42.4, 40.9, 39.3, 27.8, 26.7, 24.3, 22.6, 21.9 ppm. **HRMS (ESI)** calcd. for C_{16}H_{26}O_{2}Na [M+Na] m/z 273.1837 found m/z 273.1825. The enantiomeric ratio of 3c was determined by HPLC analysis using Daicel Chiralcel OJ-H column: *n*-Hexane: isopropanol = 90:10, flow rate 0.5 mL/min, λ = 254 nm (channel 1), 232 nm (channel 2): t_{1} (major) = 15.8 min, t_{2} (minor) = 21.2 min.
29. Experimental procedures and characterization data of all final products:

(R)-1-(4-methoxyphenyl)-2-phenylpyrrolidine (2a)

To an oven-dried 5 ml vial equipped with a magnetic stir bar was added substrate amino-alcohol 1a (135.5 mg, 0.5 mmol), MS (3Å) (300 mg), and Fe(OTf)₃ (25.05 mg, 0.05 mmol). The tube was sealed with a teflon-lined cap, connected to a vacuum and backfilled with argon three times by piercing with a needle attached to a Schlenk line. Then 2.0 ml of anhydrous DCE was added by syringe and the mixture was stirred at 90 °C for 24 hours. After this, the reaction was cooled to room temperature and the crude was concentrated under vacuum. The crude residue was purified by column chromatography with ethyl acetate and hexanes (1:20) as solvent to obtain the pure product 2a (98%, 133 mg) as colorless oil. IR (neat) 3059, 3044, 2966, 2901, 2829, 1618, 1513, 1490, 1450, 1363, 1262, 1240, 1179, 1174, 1042, 966, 811.93, 770.69, 747.12, 519.20 cm⁻¹. ¹H NMR (400 MHz, Chloroform-d) δ = 7.37 – 7.32 (m, 4H), 7.28 (d, J = 4.2 Hz, 1H), 6.82 – 6.74 (m, 2H), 6.66 (d, J = 8.9 Hz, 2H), 4.73 (dd, J = 7.5, 5.2 Hz, 1H), 3.75 (s, 3H), 3.12 (td, J = 6.8, 1.9 Hz, 2H), 1.94 – 1.84 (m, 2H), 1.81 – 1.73 (m, 1H), 1.72 – 1.64 (m, 1H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 131.7, 128.5, 127.6, 125.8, 115.3, 115.2, 114.9, 99.9, 74.3, 55.8, 45.8, 36.8, 25.9 ppm. HRMS (ESI) calcd. for C₁₇H₂₀ONa [M+Na] m/z 254.1546 found m/z 254.1539.

The enantiomeric ratio of 2a was determined by HPLC analysis using Daicel Chiralcel OJ-H column: n-Hexane : isopropanol = 95:05, flow rate 0.5 mL/min, λ = 254 nm (channel 1): t₁ (minor) = 13.1 min, t₂ (major) = 14.4 min.
(R)-1,2-diphenylpyrrolidine (2b)

Alcohol 1b (120.5 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)$_3$ (25.05 mg, 0.05 mmol) were treated as described for 1a for 48 h. After completion of reaction (TLC), the crude was concentrated under vacuum and purified by a fast column chromatographic using silica gel (mess 100-200) and dichloromethane eluent obtain pure 2b (115.6 mg, 0.96 mmol, 96% yield) as colorless oil. IR (neat) 3060, 3034, 2965, 2911, 2834, 1601, 1512, 1455, 1362, 1264, 1239, 1180, 1101, 1034, 965, 812.73, 771.90, 740.13, 584 cm$^{-1}$. $^1$H NMR (400 MHz, Chloroform-d) $\delta$ = 7.36 – 7.19 (m, 5H), 7.15 (t, $J$ = 7.8 Hz, 2H), 6.64 (t, $J$ = 7.3 Hz, 1H), 6.50 (d, $J$ = 8.0 Hz, 2H), 4.73 (d, $J$ = 8.3 Hz, 1H), 3.70 (d, $J$ = 8.6 Hz, 1H), 3.42 (q, $J$ = 8.4 Hz, 1H), 2.38 (tt, $J$ = 11.0, 7.9, 5.9 Hz, 1H), 2.00 (m, Hz, 3H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 147.1, 144.6, 128.9, 128.4, 126.6, 125.9, 115.7, 112.3, 62.9, 49.1, 36.0, 23.0 ppm. HRMS (ESI) calcd. for C$_{16}$H$_{17}$NNa [M+Na]$^+$ m/z 246.1261 found m/z 246.1251.

The enantiomeric ratio of 2b was determined by HPLC analysis using Daicel Chiralcel OJ-H column: n-Hexane: isopropanol = 95:5, flow rate 0.5 mL/min, $\lambda$ = 254 nm (channel 1), 232 nm (channel 2): $t_1$ (major) = 8.3 min, $t_2$ (minor) = 9.9 min.

(R)-2-(4-fluorophenyl)-1-(4-methoxyphenyl)pyrrolidine (2c)

Alcohol 1c (144.5 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)$_3$ (25.05 mg, 0.05 mmol) were treated as described for 1a for 24 h and purified as described for 1a to obtain
2c (128.7 mg, 0.474 mmol, 95% yield) as a yellowish oil. IR (neat) 3061, 3045, 2963, 2911, 2815, 1611, 1525, 1493, 1451, 1362, 1281, 1229, 1178, 1177, 1044, 965, 812.23, 771.19, 737.12, 701.38, 523.21 cm\(^{-1}\). \(^1\)H NMR (400 MHz, Chloroform-d) \(\delta = 7.20\) (s, 2H), 7.03 – 6.89 (m, 2H), 6.81 – 6.70 (m, 2H), 6.42 (d, \(J = 8.4\) Hz, 2H), 4.60 (d, \(J = 8.5\) Hz, 1H), 3.71 (s, 3H), 3.64 – 3.70 (m, 1H), 2.38 (t, \(J = 10.3\) Hz, 1H), 1.68 (s, 1H) ppm. \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta = 151.0, 143.7, 141.9, 132.2, 128.6, 127.3, 114.9, 113.1, 127.3 (J\(_{CF} = 10\) Hz), 115.2 (J\(_{CF} = 20\) Hz), 114.8, 113.0, 62.8, 55.9, 49.7, 36.2, 23.3 ppm. HRMS (ESI) calcd. for C\(_{17}\)H\(_{19}\)NFO [M+H] \(m/z\) 272.1447 found \(m/z\) 272.1445. The enantiomeric ratio of 2c was determined by HPLC analysis using Daicel Chiralcel OJ-H column: n-Hexane : isopropanol = 95:5, flow rate 0.5 mL/min, \(\lambda = 254\) nm (channel 1), 232 nm (channel 2): \(t_1\) (minor) = 10.4 min, \(t_2\) (major) = 19.9 min.

\((R)-2-(4\text{-chlorophenyl})-1-(4\text{-methoxyphenyl})\text{pyrrolidine (2d)}\)

![Chemical structure](image)

Alcohol 1d (152.5 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)_3 (25.05 mg, 0.05 mmol) were treated as described for 1a for 24 h and purified as described for 1a to obtain 2d (140.6 mg, 0.489 mmol, 98% yield) as a yellowish oil. IR (neat) 3061, 3045, 2963, 2911, 2815, 1611, 1531, 1490, 1450, 1356, 1280, 1232.45, 1188, 1167.35, 1063.54, 960, 811.33, 770.29, 717.11, 701.41, 520.83 cm\(^{-1}\). \(^1\)H NMR (400 MHz, Chloroform-d) \(\delta = 7.33 – 7.23\) (m, 2H), 7.19 (s, 2H), 6.82 – 6.70 (m, 2H), 6.41 (d, \(J = 8.4\) Hz, 2H), 4.59 (dd, \(J = 8.6, 2.7\) Hz, 1H), 3.71 (s, 3H), 3.70 (s, 1H), 3.34 (q, \(J = 8.3\) Hz, 1H), 2.44 – 2.28 (m, 1H), 1.98 (s, 2H), 1.87 (s, 1H). \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta = 151.0, 143.7, 141.9, 132.2, 128.6, 127.3, 114.9, 113.1, 62.9, 55.9, 49.7, 36.2, 23.3 ppm. HRMS (ESI) calcd. for C\(_{17}\)H\(_{19}\)ClNO [M+H] \(m/z\) 288.1158
found \( m/z \) 288.1150. The enantiomeric ratio of \( 2d \) was determined by HPLC analysis using Daicel Chiralcel OJ-H column: \( n \)-Hexane : isopropanol = 95:5, flow rate 0.5 mL/min, \( \lambda \) = 254 nm (channel 1), 232 nm (channel 2): \( t_1 \) (minor) = 24.0 min, \( t_2 \) (major) = 33.4 min.

\((R)-1-(4\text{-methoxyphenyl})-2-(3\text{-}(\text{ trifluoromethoxy})\text{phenyl})\text{pyrrolidine (2e)}\)

Alcohol \( 1e \) (177.5 mg, 0.5 mmol), MS (3\AA) (300 mg), and the catalyst Fe(OTf)_3 (25.05 mg, 0.05 mmol) were treated as described for \( 1a \) for 24 h and purified as described for \( 1a \) to obtain \( 2d \) (165.5 mg, 0.485 mmol, 91\% yield) as a yellowish oil. IR (neat) 3061, 3053, 2921, 2822, 1611, 1521, 1493, 1451, 1352, 1271, 1222.35, 1178, 1060.57, 963, 801.34, 771, 701.41 cm^{-1}.

\(^1H \text{ NMR} \) (400 MHz, Chloroform-\( d \)) \( \delta = 7.31 \) (t, \( J = 7.8 \) Hz, 1H), 7.18 (dd, \( J = 7.7 \), 1.3 Hz, 1H), 7.13 – 7.04 (m, 2H), 6.81 – 6.71 (m, 2H), 6.47 – 6.38 (m, 2H), 4.62 (dd, \( J = 8.5 \), 2.5 Hz, 1H), 3.72 (s, 3H), 3.71 – 3.63 (m, 1H), 3.35 (td, \( J = 8.7 \), 7.1 Hz, 1H), 2.40 (m, 1H), 2.00 (m, 2H), 1.91 (m, 1H). \(^{13}C \text{ NMR} \) (101 MHz, CDCl_3) \( \delta = 151.1, 149.6, 147.9, 141.8, 129.8, 124.2, 119.2, 118.8, 118.5, 114.8, 113.1, 63.1, 55.9, 49.7, 36.1, 23.3 \) ppm HRMS (ESI) calcd. for \( \text{C}_{18}\text{H}_{18}\text{F}_3\text{NO}_2 \) [M+H] \( m/z \) 338.1323 found \( m/z \) 338.1320. The enantiomeric ratio of \( 2e \) was determined by HPLC analysis using Daicel Chiralcel OJ-H column: \( n \)-Hexane : isopropanol = 90:10, flow rate 1.0 mL/min, \( \lambda \) = 254 nm (channel 1), 232 nm (channel 2): \( t_1 \) (major) = 7.18 min, \( t_2 \) (minor) = 9.5 min.
(R)-1-(4-methoxyphenyl)-2-(p-tolyl)pyrrolidine (2f)

Alcohol 1f (143.5 mg, 0.5 mmol), MS (3 Å) (300 mg), and the catalyst Fe(OTf)$_3$ (25.05 mg, 0.05 mmol) were treated as described for 1a for 24 h and purified as described for 1a to obtain 2f (124.1 mg, 0.464 mmol, 93% yield) as a colorless oil. IR (neat) 3068, 3050, 2911, 2821, 1609, 1530, 1491, 1453, 1372, 1273, 1231.42, 1137, 1166.32, 1064.43, 961, 816.43, 771.49, 707.13, 521.93 cm$^{-1}$. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ = 7.27 – 7.17 (m, 4H), 6.91 – 6.81 (m, 2H), 6.60 – 6.51 (m, 2H), 4.72 (dd, $J$ = 8.3, 2.5 Hz, 1H), 3.81 (s, 3H), 3.80 – 3.75 (m, 1H), 3.45 (td, $J$ = 8.9, 6.6 Hz, 1H), 2.53 – 2.45 (m, 1H), 2.44 (s, 3H), 2.21 – 2.09 (m, 1H), 2.08 – 1.96 (m, 2H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 150.7, 142.2, 142.1, 136.0, 129.1, 125.8, 114.8, 112.9, 63.1, 55.9, 49.6, 43.4, 36.3, 23.3, 21.0 ppm. HRMS (ESI) calcd. for C$_{18}$H$_{22}$NO [M+H] $m/ z$ 268.1705 found $m/ z$ 268.1696.

The enantiomeric ratio of 2f was determined by HPLC analysis using Daicel Chiralcel OJ-H column: n-Hexane : isopropanol = 95:5, flow rate 1.0 mL/min, $\lambda$ = 254 nm (channel 1), 232 nm (channel 2): $t_1$ (major) = 11.4 min, $t_2$ (major) = 12.5 min.

(R)-2-(3-methoxyphenyl)-1-(4-methoxyphenyl)pyrrolidine (2g)

Alcohol 1g (150.5 mg, 0.5 mmol), MS (3 Å) (300 mg), and the catalyst Fe(OTf)$_3$ (25.05 mg, 0.05 mmol) were treated as described for 1a for 24 h and purified as described for 1a to obtain 2g (119.4 mg, 0.421 mmol, 88% yield) as a colorless oil. IR (neat) 3068, 3050, 2911, 2821,
1609, 1530, 1491, 1453, 1372, 1273, 1231.42, 1137, 1166.32, 1064.43, 961, 816.43, 771.49, 707.13, 521.93 cm\(^{-1}\). \(^1\text{H NMR}\) (400 MHz, Chloroform-\(d\)) \(\delta = 7.22\) (t, \(J = 7.8\) Hz, 1H), 6.94 – 6.64 (m, 5H), 6.44 (d, \(J = 8.5\) Hz, 2H), 4.65 – 4.50 (m, 1H), 3.77 (s, 3H), 3.71 (s, 3H), 3.67 (d, \(J = 10.1\) Hz, 1H), 3.34 (q, \(J = 8.3\) Hz, 1H), 2.37 (tt, \(J = 11.5, 7.8\) Hz, 1H), 2.12 – 1.80 (m, 3H). \(^{13}\text{C NMR}\) (101 MHz, CDCl\(_3\)) \(\delta = 159.8, 150.9, 147.2, 142.2, 129.4, 118.3, 114.8, 113.0, 111.9, 111.6, 63.5, 55.9, 55.1, 49.7, 36.2, 23.4\) ppm. HRMS (ESI) calcd. for C\(_{18}\)H\(_{22}\)NO\(_2\) [M+H] \textit{m/z} 284.1649 found \textit{m/z} 284.1645.

The enantiomeric ratio of 2g was determined by HPLC analysis using Daicel Chiralcel OJ-H column: \textit{n}-Hexane : isopropanol = 95:05, flow rate 0.5 mL/min, \(\lambda = 254\) nm (channel 1), 232 nm (channel 2): \(t_1\) (minor) = 27.8 min, \(t_2\) (major) = 35.4 min.

\(\text{(R)-2-phenyltetrahydrofuran (2h)}\)\(^7\)

\[
\begin{align*}
\text{OH} & \quad \text{MS (3Å)} \\
\text{1h} & \quad 10 \text{ mol% Fe(OTf)}_3, \\
\text{DEC : n-Hexane (1:1)} & \quad 30 ^\circ \text{C, 12h}
\end{align*}
\]

\[
\begin{align*}
\text{2h} & \quad \text{H}_2\text{O} \\
\text{S/R = 07:93} & \quad \text{S/R = 88:12}
\end{align*}
\]

Alcohol 1h (83 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)\(_3\) (25.05 mg, 0.05 mmol) were treated as described for 1a for 12 h. After completion of reaction (TLC), the crude was concentrated under vacuum and purified by a fast column chromatographic using silica gel (mess 100-200) and dichloromethane eluent obtain pure 2h (82 mg, 0.98.8 mmol, 99% yield) as colorless oil. \(^1\text{H NMR}\) (400 MHz, Chloroform-\(d\)) \(\delta = 7.37 – 7.30\) (m, 4H), 7.28 – 7.22 (m, 1H), 4.90 (t, \(J = 7.2\) Hz, 1H), 4.10 (dt, \(J = 8.3, 6.8\) Hz, 1H), 3.94 (td, \(J = 7.8, 6.4\) Hz, 1H), 2.38 – 2.26 (m, 1H), 2.07–1.95 (m, 2H), 1.87 – 1.76 (m, 1H) ppm. \(^{13}\text{C NMR}\) (101 MHz, CDCl\(_3\)) \(\delta = 143.4, 128.2, 127.1, 125.6, 80.7, 68.6, 34.6, 26.0\) ppm. The enantiomeric ratio of 2h was determined by HPLC analysis using Daicel Chiralcel OJ-H column: \textit{n}-Hexane : isopropanol = 95:05, flow rate 0.5 mL/min, \(\lambda = 254\) nm (channel 1): \(t_1\) (minor) = 9.2 min, \(t_2\) (major) = 10.1 min.
(R)-2-(4-fluorophenyl)tetrahydrofuran (2i)

Alcohol 1i (83 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)₃ (25.05 mg, 0.05 mmol) were treated as described for 1a for 12 h. After completion of reaction (TLC), the crude was concentrated under vacuum and purified by a fast column chromatographic using silica gel (mess 100-200) and dichloromethane eluent obtain pure 2i (82 mg, 0.988 mmol, 99% yield) as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ = 7.31-7.26 (m, 2H), 7.03-6.98 (m, 2H), 4.85 (t, J = 6.8 Hz), 4.08 (q, J = 8.0 Hz, 1H), 3.94 (q, J = 7.6 Hz, 1H), 2.34-2.26 (m, 1H), 2.04-1.96 (m, 1H), 1.80-1.71 (m, 1H) ppm. ¹³C NMR (100 MHz, CDCl₃) δ = 162.0 (J_C-F = 250 Hz), 139.1, 139.0, 127.2 (J_C-F = 10 Hz), 115.0 (J_C-F = 20 Hz), 80.1, 68.6, 34.6, 25.9 ppm.

The enantiomeric ratio of 2i was determined by HPLC analysis using Daicel Chiralcel OJ-H column: n-Hexane : isopropanol = 80:20, flow rate 0.5 mL/min, λ = 254 nm (channel 1): t₁ (major) = 12.3 min, t₂ (minor) = 13.8 min.

(R)-1-(4-methoxyphenyl)-2-vinylpyrrolidine (2j)

Alcohol 1j (110.5 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)₃ (25.05 mg, 0.05 mmol) were treated as described for 1a at 100 °C for 48 h. After completion of reaction (TLC), the crude was concentrated under vacuum and purified by the column chromatographic using silica gel (mess 100-200) to obtain pure 2j (89 mg, 0.438 mmol, 88% yield) as colorless oil. IR (neat) cm⁻¹ 3040, 2955.9, 2890, 2875, 2821, 1616.5, 1575.6, 1513,
1460, 1365, 1274, 1181, 1045, 970, 820, 591 cm\(^{-1}\). \(^1\)H NMR (400 MHz, Chloroform-d) \(\delta = 6.87 – 6.81\) (m, 2H), 6.61 – 6.55 (m, 2H), 5.84 (ddd, \(J = 17.1, 10.2, 5.4\) Hz, 1H), 5.19 – 5.08 (m, 2H), 4.10 (ddddd, \(J = 6.7, 5.4, 2.6, 1.3\) Hz, 1H), 3.77 (s, 3H), 3.49 (dd, \(J = 8.4, 7.0, 2.8\) Hz, 1H), 3.22 (td, \(J = 8.7, 6.9\) Hz, 1H), 2.21 – 1.91 (m, 4H), 1.83 (ddt, \(J = 8.7, 6.1, 3.0\) Hz, 1H) ppm. \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta = 150.8, 142.5, 139.9, 114.8, 114.3, 112.9, 61.4, 55.9, 49.2, 32.7, 23.3\) ppm. HRMS (ESI) calcd. for C\(_{13}\)H\(_{18}\)NO [M+H] 204.1383 \(m/z\) found 204.1386 \(m/z\). The enantiomeric ratio of \(2j\) was determined by HPLC analysis using Daicel Chiralcel OJ-H column: \(\text{n-Hexane: isopropanol = 99.5:0.5, flow rate 0.5 mL/min, }\lambda = 254 \text{ nm (channel 1): } t_1 \text{ (major) = 60.5 min, } t_2 \text{ (minor) = 63.1 min.}

(S)-1-(4-methoxyphenyl)-2-methylpyrrolidine (2k)

\[
\begin{align*}
1k & \quad \text{Me} \\
\text{OH} & \quad \text{N} \\
\text{Me} & \quad \text{OMe}, \\
\end{align*}
\]

\[
\begin{align*}
10 \text{ mol\% Fe(OTf)}_3, & \quad \text{MS (3Å)} \\
1,2-\text{Dichloroethane} & \quad 100 \text{ °C, 48h} \\
2k & \quad + \text{H}_2\text{O} \\
R/S = 2:98 & \quad S/R = 2:98
\end{align*}
\]

Alcohol \(1k\) (104.5 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)\(_3\) (25.05 mg, 0.05 mmol) were treated as described for \(1a\) at 100 °C for 48 h. After completion of reaction (TLC), the crude was concentrated under vacuum and purified by the column chromatographic using silica gel (mess 100-200) to obtain pure \(2k\) (76.4 mg, 0.4 mmol, 80% yield) as colorless oil. IR (neat) cm\(^{-1}\) 3045, 2961, 2930, 2874, 2824, 1616, 1575, 1464, 1329, 1275, 1245, 1181, 1164, 1041, 970, 811.6, 591 cm\(^{-1}\). \(^1\)H NMR (400 MHz, Chloroform-d) \(\delta = 6.97 – 6.87\) (m, 2H), 6.68 – 6.56 (m, 2H), 3.85 (td, \(J = 6.5, 2.2\) Hz, 1H), 3.82 (s, 3H), 3.52 – 3.43 (m, 1H), 3.17 (td, \(J = 8.8, 7.0\) Hz, 1H), 2.21 – 1.91 (m, 3H), 1.75 (dp, \(J = 5.3, 2.6, 2.2\) Hz, 1H), 1.23 (d, \(J = 6.2\) Hz, 3H) ppm. \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta = 150.6, 142.2, 114.9, 112.7, 55.9, 54.0, 48.9, 33.1, 23.3, 19.5\) ppm. The enantiomeric ratio of \(2k\) was determined by
HPLC analysis using Daicel Chiralcel OJ-H column: n-Hexane : isopropanol = 99.5:0.5, flow rate 0.5 mL/min, $\lambda = 254$ nm (channel 1): $t_1$ (minor) = 30.0 min, $t_2$ (major) = 60.6 min.

(S)-2-ethyl-1-(4-methoxyphenyl)pyrrolidine (2l)

Alcohol 1l (111.5 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)$_3$ (25.05 mg, 0.05 mmol) were treated as described for 1a at 100 °C for 48 h. After completion of reaction (TLC), the crude was concentrated under vacuum and purified by the column chromatographic using silica gel (mess 100-200) to obtain pure 2l (85.07 mg, 0.419 mmol, 83% yield) as colorless oil. IR (neat) cm$^{-1}$: 3044, 2960.59, 2931, 2873, 2829, 1619.75, 1574.68, 1512.9, 1464, 1363, 1327, 1274, 1240.55, 1180, 1163, 1044, 969, 810.9, 590, 525 cm$^{-1}$. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ = 7.01 – 6.93 (m, 2H), 6.69 – 6.60 (m, 2H), 3.87 (s, 3H), 3.62 (tt, $J = 7.2$, 2.6 Hz, 1H), 3.57 – 3.48 (m, 1H), 3.25 – 3.14 (m, 1H), 2.20 – 1.99 (m, 3H), 1.99 – 1.81 (m, 2H), 1.50 – 1.33 (m, 1H), 1.06 (t, $J = 7.5$ Hz, 3H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 150.5, 142.4, 114.9, 112.5, 60.4, 55.7, 49.0, 29.8, 26.0, 23.5, 10.5 ppm. HRMS (ESI) calcd. For C$_{13}$H$_{20}$NO [M+H] 206.1539 m/z found 206.1546 m/z. The enantiomeric ratio of 2l was determined by HPLC analysis using Daicel Chiralcel OJ-H column: n-Hexane : isopropanol = 99.5:0.5, flow rate 0.5 mL/min, $\lambda = 254$ nm (channel 1): $t_1$ (minor) = 25.8 min, $t_2$ (minor) = 63.8 min.
(R)-1-(4-methoxyphenyl)-2-phenylpiperidine (2m)

```
  OMe
  \   \      Ph
  OH \   \      Ph
  \    \      \      OMe
    \     \    \ 2m
  \     \   \   |      \  
    \     \   \   |      \ 
 S/R= 88:12
          \   \    
        1m
```

Alcohol 1m (142.58 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)₃ (25.05 mg, 0.05 mmol) were treated as described for 1a for 24 h and purified as described for 1a to obtain 2m (116.1 mg, 0.434 mmol, 87% yield) as a colorless oil. IR (neat) 3060, 3044, 2956, 2915, 2830, 1611, 1512, 1491, 1451, 1360, 1261, 1245, 1180, 1171, 961.56, 811.75, 771.69, 748.15, 590.1, 521.25 cm⁻¹. ¹H NMR (400 MHz, Chloroform-d) δ = 7.19 – 7.14 (m, 2H), 7.13 – 7.06 (m, 2H), 7.03 – 6.96 (m, 1H), 6.87 – 6.76 (m, 2H), 6.63 – 6.52 (m, 2H), 3.96 (dd, J = 9.5, 3.3 Hz, 1H), 3.61 (s, 3H), 3.35 – 3.25 (m, 1H), 2.81 (ddd, J = 12.0, 10.1, 3.5 Hz, 1H), 1.90 – 1.81 (m, 1H), 1.72 (dddd, J = 15.0, 13.4, 10.5, 6.4 Hz, 4H), 1.47 – 1.38 (m, 1H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 144.7, 128.0, 127.4, 126.1, 123.7, 113.7, 99.9, 92.9, 64.4, 56.4, 55.3, 36.1, 26.5, 24.1 ppm. HRMS (ESI) calcd. for C₁₈H₂₂NO [M+H] m/z 268.1702 found m/z 268.1696. The enantiomeric ratio of 2m was determined by HPLC analysis using Daicel Chiralcel OJ-H column: n-Hexane : isopropanol = 95:05, flow rate 0.5 mL/min, λ = 254 nm (channel 1), 232 nm (channel 2): t₁ (minor) = 13.6 min, t₂ (major) = 20.5 min.

(R)-1-(4-methoxyphenyl)-2-phenyl-1,2,3,4-tetrahydroquinoline (2n)

```
  OMe
  \   \      Ph
  OH \   \      Ph
  \    \      \      OMe
    \     \    \ 2m
  \     \   \   |      \  
    \     \   \   |      \ 
 S/R = 99:1
          \   \    
        1n
```

10 mol% Fe(OTf)₃, 1,2-Dichloroethane 90 °C, 24h

R/S = 95:5
Alcohol 1n (166.58 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)$_3$ (25.05 mg, 0.05 mmol) were treated as described for 1a for 24 h and purified as described for 1a to obtain 2n (157.5 mg, 0.5 mmol, 100% yield) as a colorless oil. IR (neat) 3013, 2931, 2852, 1602, 1508, 1451, 1289.97, 1238.08, 1211, 1179, 1101, 933.6, 827.75, 756.69, 700.08, 552.2 cm$^{-1}$. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ = 7.21 (d, $J$ = 4.4 Hz, 4H), 7.15 (dd, $J$ = 4.8, 3.7 Hz, 1H), 7.07 – 7.01 (m, 2H), 6.99 – 6.94 (m, 1H), 6.93 – 6.85 (m, 1H), 6.78 – 6.70 (m, 2H), 6.60 (td, $J$ = 7.3, 1.2 Hz, 1H), 6.52 (dd, $J$ = 8.3, 1.1 Hz, 1H), 4.78 (d, $J$ = 4.3 Hz, 1H), 3.71 (s, 3H), 2.71 – 2.52 (m, 2H), 2.25 (ddt, $J$ = 12.9, 11.4, 4.9 Hz, 1H), 2.14 – 2.02 (m, 1H) ppm. $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ = 156.8, 145.2, 144.2, 140.5, 129.2, 128.3, 128.2, 126.7, 126.7, 126.7, 122.4, 116.8, 114.7, 114.1, 63.7, 55.4, 28.9, 23.7 ppm. HRMS (ESI) calcd. for C$_{22}$H$_{21}$NNaO [M+Na]$^+$ m/z 338.1508 found m/z 338.1515.

The enantiomeric ratio of 2n was determined by HPLC analysis using Daicel Chiralcel OJ-H column: n-Hexane : isopropanol = 96:4, flow rate 1.0 mL/min, $\lambda$ = 254 nm (channel 1), 232 nm (channel 2): $t_1$ (minor) = 10.5 min, $t_2$ (major) = 11.9 min.

(R)-2-phenylchromane (2o)

Alcohol 1o (114 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)$_3$ (25.05 mg, 0.05 mmol) were treated as described for 1a at -20 °C for 48 h. After completion of reaction (TLC), the crude was concentrated under vacuum and purified by the column chromatographic using silica gel (mess 100-200) to obtain pure 2o (95 mg, 0.454 mmol, 91% yield) as colorless oil. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ = 7.50 – 7.38 (m, 4H), 7.38 – 7.31 (m, 1H), 7.21 – 7.08 (m, 2H), 6.99–6.85 (m, 2H), 5.10 (dd, $J$ = 10.1, 2.5 Hz, 1H), 3.03
(ddddd, J = 17.3, 11.2, 6.0, 1.1 Hz, 1H), 2.83 (dd, J = 16.5, 5.3, 3.4 Hz, 1H), 2.25 (dd, J = 13.7, 5.9, 3.3, 2.5 Hz, 1H), 2.13 (ddddd, J = 13.7, 11.3, 10.1, 5.3 Hz, 1H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$) δ = 155.1, 141.7, 129.5, 128.5, 127.8, 127.3, 125.9, 121.8, 120.3, 116.9, 76.7, 29.9, 25.1 ppm. The enantiomeric ratio of 2o was determined by HPLC analysis using Daicel Chiralcel JM column: n-Hexane: isopropanol = 98:2, flow rate 0.5 mL/min, λ = 254 nm (channel 1): $t_1$ (major) = 8.9 min, $t_2$ (minor) = 14.2 min.

(R)-2-(4-fluorophenyl)chromane (2p)

Alcohol 1p (123 mg, 0.5 mmol), MS (3Å) (300 mg), and the catalyst Fe(OTf)$_3$ (25.05 mg, 0.05 mmol) were treated as described for 1a at -20 °C for 48 h. After completion of reaction (TLC), the crude was concentrated under vacuum and purified by the column chromatographic using silica gel (mess 100-200) to obtain pure 2p (92 mg, 0.419 mmol, 81% yield) as colorless oil. $^1$H NMR (400 MHz, Chloroform-d) δ = 7.47 – 7.39 (m, 2H), 7.21 – 7.06 (m, 4H), 6.97 – 6.87 (m, 2H), 5.08 (dd, J = 10.2, 2.5 Hz, 1H), 3.15 – 2.94 (m, 1H), 2.84 (dd, J = 16.5, 5.3, 3.2 Hz, 1H), 2.23 (ddddd, J = 13.7, 5.8, 3.2, 2.4 Hz, 1H), 2.10 (ddddd, J = 13.7, 11.3, 10.2, 5.3 Hz, 1H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$) δ = 162.4 (J$_{C,F}$ = 250 Hz), 154.9, 137.5, 137.5, 129.5, 127.73 (J$_{C,F}$ = 10 Hz), 127.4, 121.7, 120.4, 116.9, 115.3 (J$_{C,F}$ = 20 Hz), 30.00, 25.04 ppm. ppm. The enantiomeric ratio of 2p was determined by HPLC analysis using Daicel Chiralcel JM-column: n-Hexane : isopropanol = 95:5, flow rate 1 mL/min, λ = 254 nm (channel 1): $t_1$ (major) = 6.5 min, $t_2$ (minor) = 9.7 min.
(R)-2-methyl-1,2-diphenyl-1,2,3,4-tetrahydroquinoline (4a)

Alcohol 3a (158.5 mg, 0.5 mmol) and the catalyst Fe(OTf)₃ (25.05 mg, 0.05 mmol) in DCE + n-Hexane (1:1) were treated as described for 1a at room temperature for 48 h and purified as described for 1a to obtain 4a (146.51 mg, 0.5 mmol, 98% yield) as a white color solid. IR (neat) 3028, 3057.11, 29.79.30, 2935.16, 2845, 1602, 1591, 1575.59, 1492, 1455.32, 1378, 1319, 1236.83, 1156, 1132, 1072, 1026, 1003, 933.49, 841, 763, 746, 699.48 cm⁻¹. ¹H NMR (400 MHz, Chloroform-d) δ = 7.47 – 7.36 (m, 2H), 7.29 (tt, J = 4.0, 3.3 Hz, 4H), 7.24 – 7.18 (m, 2H), 7.18 – 7.13 (m, 2H), 6.98 – 6.84 (m, 2H), 6.61 (td, J = 7.3, 1.1 Hz, 1H), 6.33 (dd, J = 8.3, 1.2 Hz, 1H), 2.71 (dt, J = 16.3, 4.2 Hz, 1H), 2.40 (ddd, J = 16.7, 12.3, 5.4 Hz, 1H), 2.25 (ddd, J = 12.8, 5.3, 3.6 Hz, 1H), 2.15 (td, J = 12.6, 4.8 Hz, 1H), 1.44 (s, 3H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 147.6, 146.3, 144.3, 131.0, 129.2, 129.1, 128.2, 126.5, 126.5, 126.3, 125.9, 121.7, 116.4, 114.7, 61.3, 37.8, 29.4, 24.8 ppm. HRMS (ESI) calcd. for C₂₂H₂₁NNa [M+Na] m/z 322.1570 found m/z 322.1574.

The enantiomeric ratio of 4a was determined by HPLC analysis using Daicel Chiralcel OD-H column: n-Hexane : isopropanol = 99:1, flow rate 1.0 mL/min, λ = 254 nm (channel 1), 232 nm (channel 2): t₁ (major) = 4.7 min, t₂ (minor) = 5.6 min.

(R)-2-methyl-2-phenylchromane (4b)
Alcohol 3b (121.5 mg, 0.5 mmol) and the catalyst Fe(OTf)$_3$ (25.05 mg, 0.05 mmol) in DCE + $n$-Hexane (1:1) were treated as described for 1a at -15 °C temperature for 48 h and purified as described for 1a to obtain 4b (122 mg, 0.5 mmol, 100% yield) as a white color solid. IR (neat) 3060, 3024.55, 2977.79, 2929, 2849, 16010.53, 1582, 1522.56, 1488, 1456, 1446, 1373, 1340.54, 1243.57, 1167, 1119, 1069, 1029, 971, 945.49, 826, 753, 699.68, 548 cm$^{-1}$. $^1$H NMR (400 MHz, Chloroform-$_d$) $\delta$ = 7.38 (dd, $J$ = 8.4, 1.3 Hz, 2H), 7.30 (ddd, $J$ = 7.8, 6.8, 1.2 Hz, 2H), 7.24 - 7.18 (m, 1H), 7.17 - 7.10 (m, 1H), 7.04 - 6.91 (m, 2H), 6.81 (td, $J$ = 7.4, 1.3 Hz, 1H), 2.67 (dt, $J$ = 16.1, 4.9 Hz, 1H), 2.51 - 2.34 (m, 2H), 2.09 (ddd, $J$ = 13.7, 10.5, 5.4 Hz, 1H), 1.66 (s, 3H) ppm. $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ = 154.1, 145.6, 129.3, 128.3, 127.3, 126.7, 124.9, 121.6, 119.9, 116.9, 78.3, 32.9, 30.1, 22.6 ppm. HRMS (ESI) calcd. for C$_{16}$H$_{16}$NNa [M+Na]$^+$ m/z 247.1231 found m/z 322.1225.

The enantiomeric ratio of 4b was determined by HPLC analysis using Daicel Chiralcel OD-H column: $n$-Hexane: isopropanol = 99:1, flow rate 0.5 mL/min, $\lambda$ = 254 nm (channel 1), 232 nm (channel 2): $t_1$ (major) = 20.1 min, $t_2$ (minor) = 22.2 min.

(S)-2-methyl-2-(4-methylpentyl)chromane (4c)

Alcohol 3c (125.15 mg, 0.5 mmol) and the catalyst Fe(OTf)$_3$ (25.05 mg, 0.05 mmol) in DCE + $n$-Hexane (1:1) were treated as described for 1a at -15 °C temperature for 48 h and purified as described for 1a to obtain 4c (106 mg, 0.456 mmol, 92% yield) as a colorless oil. IR (neat) 3374, 3041, 2928, 2865, 1603, 1515, 1430, 1319, 1254, 1214, 1170, 1155, 1063, 1023, 824, 693 cm$^{-1}$. $^1$H NMR (400 MHz, Chloroform-$_d$) $\delta$ 7.12 - 7.02 (m, 2H), 6.84 - 6.75 (m, 2H), 2.75 (t, $J$ = 6.8 Hz, 2H), 1.86 - 1.72 (m, 2H), 1.60 - 1.50 (m, 2H), 1.37 (ddd, $J$ = 14.4, 9.5, 7.6
Hz, 2H), 1.28 (s, 3H), 1.24 – 1.08 (m, 3H), 0.86 (d, J = 6.6 Hz, 6H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ = 153.8, 129.4, 127.2, 121.1, 119.4, 117.2, 76.2, 39.8, 39.3, 30.6, 27.9, 24.2, 22.6, 22.6, 22.1, 21.4 ppm. HRMS (ESI) calcd. for C$_{16}$H$_{24}$O$^+_{}$Na $m/z$ 255.1719 found $m/z$ 255.1713. The enantiomeric ratio of 4c was determined by HPLC analysis using Daicel Chiralcel OD-H column: n-Hexane : isopropanol = 99:1, flow rate 1.0 mL/min, $\lambda$ = 254 nm (channel 1), 232 nm (channel 2): t$_1$ (major) = 6.3 min, t$_2$ (minor) = 3.9 min.

30. Characterization data of synthesized intermediates

3-(hydroxymethyl)-1-phenylbutane-1,4-diol (1h’

\[
\text{IR (neat) 3337.11, 3063, 3031, 2931.01, 1603, 1493, 1453, 1348, 1205, 1218, 1156, 1028, 913.78, 849, 757.33, 700.74, 553.70 cm}^{-1}. \]

$^1$H NMR (400 MHz, Chloroform-$d$) δ = 7.38 – 7.30 (m, 4H), 7.29 – 7.22 (m, 1H), 4.80 (dd, J = 8.7, 4.0 Hz, 1H), 3.80 – 3.58 (m, 4H), 2.75 (s, 3H), 1.93 (q, J = 5.8 Hz, 1H), 1.86 – 1.69 (m, 2H) ppm. $^{13}$C NMR (101 MHz, CDCl$_3$) δ = 144.8, 128.5, 127.6, 125.6, 72.8, 65.4, 65.3, 40.5, 38.6 ppm. HRMS (ESI) calcd. for C$_{11}$H$_{16}$NaO$_3$ [M+Na] $m/z$ 219.0994 found $m/z$ 219.0992.

2-(hydroxy(phenyl)methyl)-1-phenylbutane-1,4-diol (1h’’

\[
\text{IR (neat) 3314.70, 3027, 2924.74, 1603, 1493, 1450, 1342, 1202, 1217, 1089, 1047.97, 1028.09, 913.28, 744.08, 701.12, 655 cm}^{-1}. \]

$^1$H NMR (400 MHz, Chloroform-$d$) δ = 7.45 – 7.35 (m, 4H), 7.34 – 7.24 (m, 3H), 7.23 – 7.11 (m, 3H), 5.05 (d, J = 4.4 Hz, 1H), 4.96 (d, J = 2.2 Hz, 1H), 3.55 (t, J = 6.1 Hz, 2H), 2.15 (dtd, J = 7.2, 4.6, 2.2 Hz, 1H), 1.80 (ddt, J = 14.2, 8.0, 6.1 Hz, 1H), 1.60 – 1.46 (m, 1H) ppm. $^{13}$C NMR (101 MHz, CDCl$_3$) δ = 143.1, 142.7,
HRMS (ESI) calcd. for C\textsubscript{17}H\textsubscript{20}NaO\textsubscript{3} [M+Na] \textit{m/z} 295.1305 found \textit{m/z} 295.1298.

1-(4-methoxyphenyl)piperidin-2-one (K2)

\[
\text{IR (neat) 2953, 2905, 2839, 1683, 1652, 1601, 1510, 1492, 1459, 1360.65, 1331, 1295, 1268, 1247.94, 1223, 1178, 1105, 1031, 830, 755, 730.61, 601, 575, 556.49 cm}^{-1}. \quad \text{\textsuperscript{1}H NMR (400 MHz, Chloroform-}d\textit{)} \delta = 7.19 – 7.10 (m, 2H), 6.93 – 6.87 (m, 2H), 3.80 (s, 3H), 3.62 – 3.56 (m, 2H), 2.58 – 2.50 (m, 2H), 1.97 – 1.87 (m, 4H). \quad \text{\textsuperscript{13}C NMR (101 MHz, CDCl}_3\text{)} \delta = 153.8, 129.4, 127.2, 121.1, 119.4, 117.2, 76.2, 39.8, 39.3, 30.6, 27.9, 24.2, 22.6, 22.6, 22.0, 21.4 ppm.

(S)-1-phenylethan-1-ol (N)

\[
\text{\textsuperscript{1}H NMR (400 MHz, Chloroform-}d\textit{)} \delta = 7.40 – 7.32 (m, 4H), 7.31 – 7.24 (m, 1H), 4.89 (q, J = 6.4 Hz, 1H), 1.50 (d, J = 6.5 Hz, 3H) ppm. \quad \text{\textsuperscript{13}C NMR (101 MHz, CDCl}_3\text{)} \delta = 145.8, 128.4, 127.4, 125.3, 70.4, 25.1 ppm.

(S)-1-phenylethyl diisopropylcarbamate (O)
**1H NMR** (400 MHz, Chloroform-d) $\delta = 7.40 - 7.29$ (m, 4H), 7.31 – 7.22 (m, 1H), 5.85 (q, $J = 6.6$ Hz, 1H), 1.55 (d, $J = 6.6$ Hz, 3H), 1.20 (d, $J = 7.8$ Hz, 12H) ppm. **13C NMR** (101 MHz, CDCl$_3$) $\delta = 155.0, 142.8, 128.4, 127.4, 126.0, 72.7, 46.1$ (br), 22.8, 21.3 (br) ppm.

**2-iodo-N-phenylaniline (Q)**

$\text{NH}$

**1H NMR** (400 MHz, Chloroform-d) $\delta = 7.83 - 7.76$ (m, 1H), 7.34 (dd, $J = 8.5, 7.4$ Hz, 2H), 7.24 – 7.20 (m, 2H), 7.15 (dt, $J = 7.8, 1.1$ Hz, 2H), 7.09 – 7.02 (m, 1H), 6.64 (ddd, $J = 7.9, 5.4, 3.3$ Hz, 1H), 5.93 (s, 1H) ppm. **13C NMR** (101 MHz, CDCl$_3$) $\delta = 143.9, 141.9, 139.5, 129.4, 129.0, 122.5, 121.9, 119.9, 115.9, 88.8$ ppm.

**1-(4-methoxyphenyl)pyrrolidin-2-one (A2)**

$\text{OMe}$

**1H NMR** (400 MHz, Chloroform-d) $\delta = 7.52 - 7.44$ (m, 2H), 6.93 – 6.82 (m, 2H), 3.80 (d, $J = 6.9$ Hz, 2H), 3.78 (s, 3H), 2.56 (t, $J = 8.1$ Hz, 2H), 2.18 – 2.05 (m, 2H) ppm. **13C NMR** (101 MHz, CDCl$_3$) $\delta = 173.8, 156.5, 132.6, 121.7, 113.9, 55.4, 49.1, 32.4, 17.9$ ppm.

**(5-phenyltetrahydrofuran-3-yl)methanol (2h)**

$\text{OH}$

**1H NMR** (400 MHz, Chloroform-d) $\delta = 7.41 - 7.32$ (m, 10H), 7.29 (dq, $J = 6.0, 2.9$ Hz, 3H), 5.00 (t, $J = 7.2$ Hz, 1H), 4.88 (dd, $J = 9.4, 6.4$ Hz, 1H), 4.24 (dd, $J = 8.8, 7.1$ Hz, 1H), 4.04 (dd, $J = 8.8, 7.5$ Hz, 1H), 3.95 (dd, $J = 8.8, 5.6$ Hz, 1H), 3.77 (dd, $J = 8.8, 5.9$ Hz, 2H), 3.69 (dd, $J = 10.9, 7.0$ Hz, 2H), 3.65 (dd, $J = 6.9, 1.9$ Hz, 2H), 2.72 – 2.54 (m, 2H), 2.48 (ddd, $J = 11.9$ Hz, 2H) ppm.
12.5, 8.1, 6.4 Hz, 1H), 2.19 (ddd, $J = 12.3, 7.2, 5.0$ Hz, 1H), 2.00 (ddd, $J = 12.7, 8.5, 7.3$ Hz, 1H), 1.90 – 1.80 (m, 2H), 1.77 (s, 1H), 1.55 (ddd, $J = 12.4, 9.4, 7.8$ Hz, 1H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$) δ = 143.1, 142.3, 128.3, 128.3, 127.3, 127.2, 125.7, 125.5, 81.2, 80.1, 71.1, 70.9, 65.1, 64.5, 42.4, 41.7, 37.8, 37.3 ppm. HRMS (ESI) calcd. for C$_{11}$H$_{14}$NaO$_2$ [M+Na]$^+$ m/z 201.0886 found m/z 201.0881.

Phenyl(2-phenyltetrahydrofuran-3-yl)methyl acetate (2h’’

IR (neat) 3063, 3032, 2947, 2875.58, 1738, 1494, 1455, 1371, 1233.84, 1063, 1024, 962, 913, 756.71, 700 cm$^{-1}$. $^1$H NMR (400 MHz, Chloroform-$d$) δ = 7.38 – 7.33 (m, 8H), 7.32 – 7.27 (m, 2H), 7.25 – 7.18 (m, 4H), 7.08 (ddd, $J = 7.9, 1.6, 0.6$ Hz, 3H), 5.79 (d, $J = 9.7$ Hz, 1H), 4.78 (d, $J = 6.3$ Hz, 1H), 4.02 – 3.98 (m, 1H), 3.98 – 3.92 (m, 1H), 2.88 – 2.77 (m, 1H), 2.11 (s, 3H), 2.09 – 2.03 (m, 1H), 1.91 – 1.83 (m, 1H) ppm. $^{13}$C NMR (101 MHz, CDCl$_3$) δ = 173.8, 156.5, 132.6, 121.7, 113.9, 55.4, 49.1, 32.4, 17.9 ppm. HRMS (ESI) calcd. for C$_{19}$H$_{20}$NaO$_3$ [M+Na]$^+$ m/z 319.1305 found m/z 319.1310.

31. Reference
32. Copies of HPLC chromatograms for all starting alcohols and products:

1a:

![HPLC Chromatogram 1a](image1)

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<tr>
<th>Retention Time (min)</th>
<th>Area [mV.s]</th>
<th>Height [mV]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>33359.214</td>
<td>797.494</td>
<td>97.8</td>
</tr>
<tr>
<td>2</td>
<td>1218.906</td>
<td>17.761</td>
<td>2.2</td>
</tr>
<tr>
<td>Total</td>
<td>35673.134</td>
<td>815.255</td>
<td>100.0</td>
</tr>
</tbody>
</table>

1a: racemates

![HPLC Chromatogram 1a racemates](image2)

<table>
<thead>
<tr>
<th>Retention Time (min)</th>
<th>Area [mV.s]</th>
<th>Height [mV]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15885.688</td>
<td>225.898</td>
<td>50.9</td>
</tr>
<tr>
<td>2</td>
<td>15332.894</td>
<td>191.768</td>
<td>48.1</td>
</tr>
<tr>
<td>Total</td>
<td>31218.583</td>
<td>417.666</td>
<td>100.0</td>
</tr>
</tbody>
</table>
1b: racemates
1c: racemates
1d: racemates
1e: racemates
1f: racemates
1g: racemates
1h: racemates
ii: racemates
1j: racemates
1k: racemates
11: racemates
1m:

**Image**: A graph showing retention time vs. area with peaks at 29.2 min, with areas 794.7 mV and 12.3 mV.

<table>
<thead>
<tr>
<th>Retention Time (min)</th>
<th>Area (mV.s)</th>
<th>Height (mV)</th>
<th>Area (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>31,980</td>
<td>61,930</td>
<td>87.6</td>
</tr>
<tr>
<td>Total</td>
<td>5945.478</td>
<td>74200</td>
<td>100.0</td>
</tr>
</tbody>
</table>

**Legend**: Racemates

---

1m: racemates

**Image**: A graph showing retention time vs. area with peaks at 29.0 min and 31.2 min, with areas 48,738 mV and 43,700 mV respectively.

<table>
<thead>
<tr>
<th>Retention Time (min)</th>
<th>Area (mV.s)</th>
<th>Height (mV)</th>
<th>Area (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>28,990</td>
<td>80,678</td>
<td>40.6</td>
</tr>
<tr>
<td>2</td>
<td>31,977</td>
<td>74,933</td>
<td>50.4</td>
</tr>
<tr>
<td>Total</td>
<td>123,967</td>
<td>155,612</td>
<td>100.0</td>
</tr>
</tbody>
</table>
1n: racemates
10: racemates
1p: racemates
3a: racemates
3b: racemates
3c: racemates
2a: racemates
2b: racemates
2c: racemates
2d: racemates
2e: racemates
2f: racemates
2g: racemates
2h: racemates
2i: racemates
2j: racemates
2k

2k: racenates
2m

![Graph](image1)

2m: racemates

![Graph](image2)
2n:

<table>
<thead>
<tr>
<th>Reten. Time [min]</th>
<th>Area [mV.s]</th>
<th>Height [mV]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9,979</td>
<td>151,077</td>
<td>6,312</td>
</tr>
<tr>
<td>2</td>
<td>10,707</td>
<td>997,627</td>
<td>189,426</td>
</tr>
<tr>
<td>Total</td>
<td>9946,704</td>
<td></td>
<td>190,738</td>
</tr>
</tbody>
</table>

2n: racemates

<table>
<thead>
<tr>
<th>Reten. Time [min]</th>
<th>Area [mV.s]</th>
<th>Height [mV]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9,707</td>
<td>13999,292</td>
<td>244,281</td>
</tr>
<tr>
<td>2</td>
<td>11,040</td>
<td>15936,947</td>
<td>262,238</td>
</tr>
<tr>
<td>Total</td>
<td>30336,199</td>
<td></td>
<td>603,516</td>
</tr>
</tbody>
</table>
2o

2o : racemates
2p: racemates
4a: racemates
4b: racemates
4c:

4c: racemates
33. Copies of NMR for all starting alcohols and products:

1a (\(^1^H\) NMR and \(^{13}^C\) NMR, CDCl\(_3\))

[Image of NMR spectra]
1b (\textsuperscript{1}H NMR and \textsuperscript{13}C NMR, CDCl\textsubscript{3})
$1c$ ($^1H$ NMR and $^{13}C$ NMR, CDCl$_3$)
1d (\(^1\)H NMR and \(^{13}\)C NMR, CDCl\(_3\))
**1e** (\(^{1}H\) NMR and \(^{13}C\) NMR, CDCl\(_3\))

![NMR Spectrum Image]
1f (\(^1\)H NMR and \(^{13}\)C NMR, CDCl\(_3\))
$1g$ ($^1H$ NMR and $^{13}C$ NMR, CDCl$_3$)
$\textbf{1h} \quad \left( ^1\text{H NMR and } ^{13}\text{C NMR, CDCl}_3 \right)$

![NMR Spectra Image]

**NMR Spectra Details**
- **$^1$H NMR**
  - Chemical Shifts: 7.360, 7.298, 7.260, 7.222 ppm

- **$^{13}$C NMR**
  - Chemical Shifts: 128.418, 125.769 ppm

**Structural Formula**

![Chemical Structure Image]
1i (\(^1\)H NMR and \(^{13}\)C NMR, CDCl\(_3\))
$^{1}J$ (H NMR and $^{13}$C NMR, CDCl$_3$)
1k (\(^1\)H NMR and \(^{13}\)C NMR, CDCl₃)
$^{11}(^1H\text{ NMR and }^{13}C\text{ NMR, CDCl}_3)$
1m (¹H NMR and ¹³C NMR, CDCl₃)
$\textbf{1n (}^{1}\text{H NMR and }^{13}\text{C NMR, CDCl}_3\text{)}$

[Diagram of NMR spectra and structural formula]
**10** ($^1$H NMR and $^{13}$C NMR, CDCl$_3$)
1p (¹H NMR and ¹³C NMR, CDCl₃)
$\text{1h} \ (^1\text{H NMR and } ^{13}\text{C NMR, CDCl}_3)$
$1h^\prime\prime$ ($^1$H NMR and $^{13}$C NMR, CDCl$_3$)
3a (\textsuperscript{1}H NMR and \textsuperscript{13}C NMR, CDCl\textsubscript{3})
3b (\(^1^H\) NMR and \(^1^3^C\) NMR, CDCl\(_3\))
$3c\ (\text{H NMR and } ^{13}\text{C NMR, CDCl}_3)$
2a (1H NMR and 13C NMR, CDCl3)
2b (\(^1H\) NMR and \(^{13}C\) NMR, CDCl\(_3\))
$2e \ (^{1}H \text{ NMR and } ^{13}C \text{ NMR, CDCl}_3)$
$2d\ (^{1}H\ \text{NMR\ and\ }^{13}C\ \text{NMR, CDCl}_3)$
2e (\textsuperscript{1}H NMR and \textsuperscript{13}C NMR, CDCl\textsubscript{3})
2f (\(^1\)H NMR and \(^{13}\)C NMR, CDCl\(_3\))
2g (¹H NMR and ¹³C NMR, CDCl₃)
$2h$ ($^1$H NMR and $^{13}$C NMR, CDCl$_3$)

\[ \text{Diagram showing NMR spectra with peak assignments.} \]
$2i\ (^{1}H\ NMR\ and\ ^{13}C\ NMR,\ CDCl_3)$

![NMR Spectra](image)
2j (\textsuperscript{1}H NMR and \textsuperscript{13}C NMR, CDCl\textsubscript{3})
2k ($^1$H NMR and $^{13}$C NMR, CDCl$_3$)
2I (\(^1\)H NMR and \(^{13}\)C NMR, CDCl\(_3\))
$2m \left( ^1H \text{NMR and } ^{13}C \text{NMR, CDCl}_3 \right)$
$2n$ ($^1H$ NMR and $^{13}C$ NMR, CDCl$_3$)
$2o \left( ^1H \text{NMR and } ^{13}C \text{NMR, CDCl}_3 \right)$
2p ($^1$H NMR and $^{13}$C NMR, CDCl$_3$)
$2h^\prime$ ($^1$H NMR and $^{13}$C NMR, CDCl$_3$)
$2h^{''}$ ($^1$H NMR and $^{13}$C NMR, CDCl$_3$)
$4a$ ($^1$H NMR and $^{13}$C NMR, CDCl$_3$)
4b (\(^1\)H NMR and \(^{13}\)C NMR, CDCl\(_3\))
4c (\(^{1}H\) NMR and \(^{13}C\) NMR, CDCl\(_3\))
A1 ($^1$H NMR and $^{13}$C NMR, CDCl$_3$)
K ($^1$H NMR and $^{13}$C NMR, CDCl$_3$)
M (\(^1\)H NMR and \(^{13}\)C NMR, CDCl\(_3\))
J (\textsuperscript{1}H NMR and \textsuperscript{13}C NMR, CDCl\textsubscript{3})

\begin{center}
\begin{figure}
\includegraphics[width=\textwidth]{J_NMR}
\end{figure}
\end{center}