Aminolysis of Epoxides Using Iridium Trichloride as an Efficient Catalyst

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Abstract: Iridium trichloride catalyzes the ring opening of epoxides by aryl, heterocyclic, or aliphatic amines under mild conditions. The reactions proceed at room temperature to afford the corresponding β-amino alcohols in excellent yields. In general, the aminolysis of cyclopentene oxide is faster than that of cyclohexene oxide in the presence of iridium trichloride as a catalyst.

Key words: epoxides, iridium trichloride, amines, amino alcohols, aminolysis

β-Amino alcohols are an important class of organic compounds that are widely used in natural products and medicinal chemistry and as chiral auxiliaries and ligands.1,2 The most practical and commonly used method for synthesizing these compounds is the direct aminolysis of an epoxide with an excess of an amine at an elevated temperature. However, the need for high temperatures is disadvantageous for sensitive functional groups and can cause problems of regioselectivity. To eliminate such problems in the ring opening of epoxides by amine nucleophiles, several activators or promoters, such as Lewis acids or metal salts, have been introduced.3–13 Nevertheless, in some cases yields are unsatisfactory, and several of these catalysts fail to bring about the cleavage of epoxides by deactivated or sterically hindered aromatic amines. Here, we report our results on a mild and efficient method for the synthesis of β-amino alcohols by nucleophilic ring opening of epoxides by aryl, heterocyclic, or aliphatic amines with iridium trichloride as a catalyst.

It is generally believed that carbon–metal bonds in third-row transition-metal complexes are more stable than those of first- or second-row transition-metal complexes. Consequently, the third-row transition-metal complexes are expected to be very stable, limiting their use as catalysts in organic transformations.14 Despite this, considerable progress has recently been made in the use of iridium complexes as catalysts in hydrogenation reactions and carbon–carbon and carbon–heteroatom bond-forming reactions.14–17 The most commonly used catalysts for these transformations are dichloro(cyclooctadiene)iridium or dichloro(η5-pentamethylcyclopentadiene)iridium in the presence of a phosphine ligand, or iridium complexes that themselves contain phosphine ligands. Recently, iridium(III) chloride hydrate (IrCl3·xH2O) was used to effect a Meinwald rearrangement for the conversion of styrene oxide and its derivatives into the corresponding aldehydes.18 We therefore wondered whether iridium(III) chloride would catalyze the reaction of epoxides with aryl, heterocyclic, or aliphatic amines to give the corresponding amino alcohols.

Initially, we studied the reaction between cyclohexene oxide (1a) and aniline (2a) (Scheme 1). When cyclohexene oxide was treated with aniline in the presence of 5 mol% of iridium trichloride in dichloromethane at room temperature for 17 h, the amino alcohol 3a was isolated in 98% yield (Table 1, entry 1). Encouraged by this result, we treated cyclohexene oxide with various aniline derivatives in the presence of iridium trichloride at room temperature to afford the corresponding β-amino alcohols in excellent yields in most of the cases that we studied. The results are summarized in Table 1. Even anilines bearing 2,6-dimethyl- or 4-nitro functionalities produced the corresponding arylaminocyclohexan-1-ols 3g and 3h in acceptable yields of 49 and 60%, respectively (entries 7 and 8, respectively).

We then tested the catalytic activity of iridium trichloride for the ring opening of cyclopentene oxide (1b) with aniline (2a). The reaction proceeded smoothly and gave the amino alcohol 4a in 91% yield (Table 1, entry 9). To establish the generality of the methodology, cyclopentene oxide was treated with various aniline derivatives in the presence of iridium trichloride in dichloromethane at room temperature (Scheme 1). The desired amino alcohols 4b–h were obtained in yields comparable to those of products derived from cyclohexene oxide (Table 1, entries 10–16). Note that, in general, the iridium trichloride-catalyzed ring opening of cyclopentene oxide with anilines was faster than that of cyclohexene oxide.

The nucleophilic cleavage by aniline of the nonsymmetric epoxides 1c and 1d (Scheme 2) gave the β-amino alcohols 5a and 6a, respectively (Table 1, entries 17 and 18).
### Table 1  Iridium Trichloride Catalyzed Ring Opening of Epoxides with Anilines at Room Temperature

<table>
<thead>
<tr>
<th>Entry</th>
<th>Epoxide</th>
<th>Aniline</th>
<th>Product(^a)</th>
<th>Time (h)</th>
<th>Yield(^b) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>(1a)</td>
<td>PhNH(_2)</td>
<td>(3a)</td>
<td>17</td>
<td>98</td>
</tr>
<tr>
<td>2</td>
<td>4-MeOC(_6)H(_4)NH(_2) (2b)</td>
<td>(3b)</td>
<td>32</td>
<td>96</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>4-ClC(_6)H(_4)NH(_2) (2c)</td>
<td>(3c)</td>
<td>29</td>
<td>89</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>4-BrC(_6)H(_4)NH(_2) (2d)</td>
<td>(3d)</td>
<td>32</td>
<td>86</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>4-F(_3)C(_6)H(_4)NH(_2) (2e)</td>
<td>(3e)</td>
<td>30</td>
<td>89</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>3,5-Me(_2)C(_6)H(_3)NH(_2) (2f)</td>
<td>(3f)</td>
<td>42</td>
<td>90</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>2,6-Me(_2)C(_6)H(_3)NH(_2) (2g)</td>
<td>(3g)</td>
<td>23</td>
<td>49</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>4-O(_2)NC(_6)H(_4)NH(_2) (2h)</td>
<td>(3h)</td>
<td>48</td>
<td>60</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>(1b)</td>
<td>PhNH(_2)</td>
<td>(4a)</td>
<td>23</td>
<td>91</td>
</tr>
<tr>
<td>10</td>
<td>4-MeOC(_6)H(_4)NH(_2) (2b)</td>
<td>(4b)</td>
<td>24</td>
<td>89</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>4-ClC(_6)H(_4)NH(_2) (2c)</td>
<td>(4c)</td>
<td>5</td>
<td>96</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>4-BrC(_6)H(_4)NH(_2) (2d)</td>
<td>(4d)</td>
<td>5</td>
<td>96</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>4-F(_3)C(_6)H(_4)NH(_2) (2e)</td>
<td>(4e)</td>
<td>6</td>
<td>94</td>
<td></td>
</tr>
</tbody>
</table>
To establish the applicability of this catalytic method to heterocyclic and aliphatic amines, amines such as pyrroliidine (2i), piperidine (2j), morpholine (2k), or butan-1-amine (2l), were treated with cyclohexene oxide (1a) or cyclopentene oxide (1b) in the presence of IrCl₃·xH₂O (Scheme 3); the results are shown in Table 2. The corresponding amino alcohols 3i–l and 4i–l were obtained in very high yields (Table 2).

The structures of the β-amino alcohols 3–6 were assigned on the basis of IR spectra, ¹H (500 MHz) and ¹³C (125 MHz) NMR spectra, and mass spectra. The trans-stereochemistry of β-amino alcohols 3 and 4 was deduced from the coupling constants of the protons on C-1 and C-2. For instance, in the ¹H NMR (500 MHz) spectrum of 2-[(4-chlorophenyl)amino]cyclohexan-1-ol (3c), two ¹H signals appeared at δ = 3.09 (ddd, J = 4.0, 9.0, 11.5 Hz, 1 H) and δ = 3.37 (ddd, J = 4.5, 9.5, 10.5 Hz, 1 H) for the CHNH and CHOH protons, respectively, which are indicative of a trans-stereochemistry. Additionally, the ¹H NMR spectra for the 2-(arylamino)cycloalkanols were in agreement with the spectra of these compounds reported in the literature.3a,4c,9,10

We believe that the IrCl₃·xH₂O-mediated epoxide ring opening involves the coordination of the metal salt to the oxygen atom of the epoxide, thereby increasing the electrophilicity at the two carbon atoms. Nucleophilic attack by the nitrogen atom of the amine at a carbon atom followed by oxirane ring opening and proton transfer leads to the formation of an amino alcohol and concurrent release of the metal salt to complete the catalytic cycle.

In summary, we have demonstrated a novel and mild method for ring opening of epoxides with aromatic

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Table 1  Iridium Trichloride Catalyzed Ring Opening of Epoxides with Anilines at Room Temperature (continued)

<table>
<thead>
<tr>
<th>Entry</th>
<th>Epoxide</th>
<th>Aniline</th>
<th>Product*</th>
<th>Time (h)</th>
<th>Yieldb (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>14</td>
<td>3,5-Me₂C₆H₄NH₂ (2f)</td>
<td>PhNH₂ (2a)</td>
<td>4f</td>
<td>24</td>
<td>98</td>
</tr>
<tr>
<td>15</td>
<td>2,6-Me₂C₆H₄NH₂ (2g)</td>
<td>PhNH₂ (2a)</td>
<td>4g</td>
<td>24</td>
<td>48</td>
</tr>
<tr>
<td>16</td>
<td>4-O₂NC₆H₄NH₂ (2h)</td>
<td>PhNH₂ (2a)</td>
<td>4h</td>
<td>30</td>
<td>53</td>
</tr>
<tr>
<td>17</td>
<td>(1c)</td>
<td>PhNH₂ (2a)</td>
<td>5a</td>
<td>12</td>
<td>89</td>
</tr>
<tr>
<td>18</td>
<td>(1d)</td>
<td>PhNH₂ (2a)</td>
<td>6a</td>
<td>4</td>
<td>78</td>
</tr>
</tbody>
</table>

*Reactions were carried out with epoxide (1 equiv), aryl amine (1 equiv), and IrCl₃·xH₂O (0.05 equiv, 5 mol%) in CH₂Cl₂ at r.t. (see general procedure).

b Yields are for the pure and isolated products.
Catalytic Aminolysis of Epoxides

Table 2  Iridium Trichloride-Catalyzed Ring Opening of Epoxides with Heterocyclic and Aliphatic Amines at Room Temperature

<table>
<thead>
<tr>
<th>Entry</th>
<th>Epoxide</th>
<th>Amine</th>
<th>Producta</th>
<th>Time Yield (%)b</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>BuNH2</td>
<td>Pyrrolidine (2i)</td>
<td>(3i)</td>
<td>14 87</td>
</tr>
<tr>
<td>2</td>
<td>BuNH2</td>
<td>piperidine (2j)</td>
<td>(3j)</td>
<td>18 85</td>
</tr>
<tr>
<td>3</td>
<td>BuNH2</td>
<td>morpholine (2k)</td>
<td>(3k)</td>
<td>24 79</td>
</tr>
<tr>
<td>4</td>
<td>BuNH2</td>
<td>Pyrrolidine (2i)</td>
<td>(3i)</td>
<td>20 84</td>
</tr>
<tr>
<td>5</td>
<td>BuNH2</td>
<td>piperidine (2j)</td>
<td>(4i)</td>
<td>10 84</td>
</tr>
<tr>
<td>6</td>
<td>BuNH2</td>
<td>morpholine (2k)</td>
<td>(4k)</td>
<td>22 82</td>
</tr>
<tr>
<td>7</td>
<td>BuNH2</td>
<td>(2l)</td>
<td>(4l)</td>
<td>23 86</td>
</tr>
</tbody>
</table>

aReactions were carried out with epoxide (1 equiv), amine (1 equiv), and IrCl3·xH2O (0.05 equiv, 5 mol%) in CH2Cl2 at r.t. (see general procedure).
bYields are for pure and isolated products.

Organic solvents were dried by standard methods when necessary. Commercially available reagents were used without further purification unless mentioned. Aniline was freshly distilled under reduced pressure before use. All reactions were monitored by TLC on aluminum plates coated with silica gel. The TLC plates were visualized with a UV lamp or in an iodine chamber. Column chromatography was performed by using silica gel (100–200 mesh; SD Fine-Chem Ltd.) with EtOAc–hexanes as an eluent system. Melting points are uncorrected. IR spectra of the compounds were recorded on a Thermo Nicolet FT-IR NexusTM and are expressed as wave numbers (cm⁻¹). 1H and 13C NMR spectra were recorded at 500 and 125 MHz, respectively, on a Bruker FT-NMR. The 1H NMR spectra were recorded in CDCl3 using TMS as the internal standard. Chemical shifts of 1H NMR spectra were given in parts per million with respect to TMS, and the coupling constant J was measured in Hz. Mass spectra were recorded by GC-MS (Perkin-Elmer Clarus 500 GC).

Catalytic Ring Cleavage of Epoxides by Amines; General Procedure

IrCl3·xH2O (21 mg, 0.05 mM, 5 mol%) was added to a stirred mixture of the epoxide (1 mM) and amine (1 mM) in CH2Cl2 (2 mL) at r.t. The reaction was monitored by TLC at regular intervals. When the reaction was complete, the solvent was evaporated in a rotary evaporator, and the residue was purified by column chromatography (silica gel [EtOAc–hexanes, 10:90 for aromatic amine products, 60:40 for heterocyclic and aliphatic amine products]).

trans-2-[(Phenylamino)cyclohexanol (3a)

Purple solid; yield: 187 mg (98%); mp 60–61 °C (Lit.3b 58–59 °C).
IR (KBr): vmax = 3388 cm⁻¹.
1H NMR (CDCl3, 500 MHz): δ = 7.18 (t, J = 8.0 Hz, 2 H), 6.73 (dd, J = 8.0, 19.0 Hz, 3 H), 3.36 (ddd, J = 4.0, 9.5, 13.0 Hz, 1 H), 3.15 (ddd, J = 4.0, 9.5, 13.0 Hz, 1 H), 2.70 (br s, 1 H), 2.11 (d, J = 12.5 Hz, 2 H), 1.81–1.68 (m, 2 H), 1.45–1.34 (m, 2 H), 1.31 (t, J = 10.5 Hz, 2 H), 1.05 (dq, J = 3.5, 12.5 Hz, 1 H).
13C NMR (CDCl3, 125 MHz): δ = 146.7, 128.3, 117.3, 113.4, 73.4, 59.1, 32.2, 30.5, 24.0, 23.3.
MS (EI, 70 eV): m/z = 191 [M⁺].

trans-2-[(4-Methoxyphenyl)amino]cyclohexanol (3b)

Brown liquid; yield: 212 mg (96%).
IR (KBr): vmax = 3419 cm⁻¹.
1H NMR (CDCl3, 500 MHz): δ = 6.78 (d, J = 9.0 Hz, 2 H), 6.69 (d, J = 9.0 Hz, 2 H), 3.75 (s, 3 H), 3.33 (ddd, J = 4.5, 10.0, 13.5 Hz, 1 H), 3.01 (ddd, J = 4.0, 9.5, 13.0 Hz, 1 H), 2.15–2.06 (m, 2 H), 1.80–1.68 (m, 2 H), 1.43–1.24 (m, 3 H), 1.01 (dq, J = 3.5, 11.5 Hz, 1 H).
13C NMR (CDCl3, 125 MHz): δ = 153.2, 141.0, 116.8, 114.9, 74.2, 62.0, 55.8, 33.1, 31.3, 25.1, 24.3.
MS (EI, 70 eV): m/z = 221 [M⁺].

trans-2-[(4-Chlorophenyl)amino]cyclohexanol (3c)

Brown solid; yield: 200 mg (89%); mp 74–75 °C.
IR (KBr): vmax = 3408 cm⁻¹.
1H NMR (CDCl3, 500 MHz): δ = 7.12 (td, J = 2.0, 8.5 Hz, 2 H), 6.65 (td, J = 2.5, 9.0 Hz, 2 H), 3.37 (s, J = 4.0, 9.5, 10.5 Hz, 1 H), 3.09 (ddd, J = 4.0, 9.0, 11.5 Hz, 1 H), 2.14–2.06 (m, 2 H), 1.82–1.69 (m, 2 H), 1.43–1.28 (m, 2 H), 1.14–1.02 (m, 2 H).
13C NMR (CDCl3, 125 MHz): δ = 147.7, 129.3, 118.3, 113.4, 74.4, 60.1, 33.2, 31.5, 24.9, 24.3.
MS (EI, 70 eV): m/z = 225 [M⁺].

trans-2-[(4-Bromophenyl)amino]cyclohexanol (3d)

Wheat-colored solid; yield: 232 mg (86%); mp 110–111 °C.
IR (KBr): vmax = 3485 cm⁻¹.
1H NMR (CDCl3, 500 MHz): δ = 7.12 (d, J = 8.5 Hz, 2 H), 6.64 (d, J = 8.5 Hz, 2 H), 3.36 (ddd, J = 4.5, 10.0, 14.0 Hz, 1 H), 3.09 (ddd, J = 4.0, 9.5, 11.5 Hz, 1 H), 2.60 (br s, 2 H), 2.15–2.06 (m, 2 H), 2.01–1.98 (m, 2 H), 1.90–1.86 (m, 1 H).
13C NMR (CDCl3, 125 MHz): δ = 148.8, 129.3, 118.3, 113.4, 74.4, 60.1, 33.2, 31.5, 24.9, 24.3.
MS (EI, 70 eV): m/z = 229 [M⁺].

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trans-2-[(4-Trifluoromethyl)phenyl]amino)cyclohexanol (3e)
Brown viscous liquid; yield: 230 mg (89%).

IR (KBr): ν \text{max} = 3383 cm⁻¹.

\(^1^H\) NMR (CDCl₃, 500 MHz): δ = 7.39 (d, J = 8.5 Hz, 2 H), 6.71 (d, J = 8.5 Hz, 2 H), 3.73 (br s, 1 H), 3.40 (ddd, J = 4.5, 9.5, 13.5 Hz, 1 H), 3.21 (dd, J = 4.0, 9.0, 11.0 Hz, 1 H), 2.36 (br s, 1 H), 2.15–2.08 (m, 2 H), 1.83–1.71 (m, 2 H), 1.45–1.20 (m, 3 H), 1.18–1.02 (m, 1 H).

\(^1^3^C\) NMR (CDCl₃, 125 MHz): δ = 150.6, 126.7, 124.9 (q, J = 268.8 Hz), 119.4 (q, J = 43.8 Hz), 113.0, 74.6, 59.4, 33.5, 31.6, 24.8, 24.2.

MS (EI, 70 eV): m/z = 259 [M⁺].

trans-2-[(3,5-Dimethylphenyl)amino)cyclohexanol (3f)
Brown solid; yield: 197 mg (90%); mp 34–36 °C.

IR (KBr): ν \text{max} = 3384 cm⁻¹.

\(^1^H\) NMR (CDCl₃, 500 MHz): δ = 6.42 (s, 1 H), 6.37 (s, 2 H), 3.33 (ddd, J = 4.0, 9.5, 13.5 Hz, 1 H), 3.12 (dd, J = 4.0, 9.5 Hz, 1 H), 2.83 (br s, 2 H), 2.23 (s, 6 H), 2.15–2.07 (m, 2 H), 1.80–1.70 (m, 2 H), 1.45–1.27 (m, 3 H), 1.04 (dq, J = 4.0, 12.5 Hz, 1 H).

\(^1^3^C\) NMR (CDCl₃, 125 MHz): δ = 147.8, 139.0, 120.3, 112.3, 74.5, 60.1, 33.0, 31.7, 25.0, 24.2, 21.4.

MS (EI, 70 eV): m/z = 219 [M⁺].

trans-2-[(2,6-Dimethylphenyl)amino)cyclohexanol (3g)
Brown viscous liquid; yield: 107 mg (49%).

IR (KBr): ν \text{max} = 3432 cm⁻¹.

\(^1^H\) NMR (CDCl₃, 500 MHz): δ = 7.00 (d, J = 7.5 Hz, 2 H), 6.84 (t, J = 7.5 Hz, 1 H), 3.41 (dt, J = 4.0, 10.0 Hz, 1 H), 2.87 (ddd, J = 4.0, 9.5, 11.0 Hz, 1 H), 2.29 (s, 6 H), 1.85–1.79 (m, 1 H), 1.76–1.71 (m, 1 H), 1.68–1.62 (m, 1 H), 1.42–1.23 (m, 3 H), 1.19–1.06 (m, 2 H).

\(^1^3^C\) NMR (CDCl₃, 125 MHz): δ = 144.0, 129.8, 129.1, 122.3, 75.1, 63.4, 33.1, 32.5, 25.3, 24.3, 19.2.

MS (EI, 70 eV): m/z = 219 [M⁺].

trans-2-[(4-Nitrophenyl)amino)cyclohexanol (3h)
Brown viscous liquid; yield: 230 mg (89%).

IR (KBr): ν \text{max} = 3384 cm⁻¹.

\(^1^H\) NMR (CDCl₃, 500 MHz): δ = 3.16 (dt, J = 4.5, 9.5 Hz, 1 H), 2.83–2.76 (m, 1 H), 2.51–2.44 (m, 1 H), 2.20 (ddd, J = 4.0, 9.5, 11.5 Hz, 1 H), 2.13–2.01 (m, 2 H), 1.19–1.15 (m, 2 H), 1.51–1.42 (m, 2 H), 1.37 (sextet, J = 7.5 Hz, 2 H), 1.32–1.21 (m, 3 H), 1.18–0.94 (m, 1 H), 0.93 (t, J = 7.5 Hz, 3 H).

\(^1^3^C\) NMR (CDCl₃, 125 MHz): δ = 73.3, 68.5, 46.4, 33.8, 32.6, 30.3, 25.0, 24.5, 20.4, 13.9.

MS (EI, 70 eV): m/z = 171 [M⁺].

trans-2-[(3,5-Dimethylphenyl)amino)cyclohexanol (3i)
Colourless liquid; yield: 144 mg (84%).

IR (KBr): ν \text{max} = 3411 cm⁻¹.

\(^1^H\) NMR (CDCl₃, 500 MHz): δ = 3.16 (dt, J = 4.5, 9.5 Hz, 1 H), 2.83–2.76 (m, 1 H), 2.51–2.44 (m, 1 H), 2.20 (ddd, J = 4.0, 9.5, 11.5 Hz, 1 H), 2.13–2.01 (m, 2 H), 1.19–1.15 (m, 2 H), 1.51–1.42 (m, 2 H), 1.37 (sextet, J = 7.5 Hz, 2 H), 1.32–1.21 (m, 3 H), 1.18–0.94 (m, 1 H), 0.93 (t, J = 7.5 Hz, 3 H).

\(^1^3^C\) NMR (CDCl₃, 125 MHz): δ = 147.7, 129.3, 117.6, 113.3, 78.3, 62.2, 32.9, 32.1, 21.1.

MS (EI, 70 eV): m/z = 177 [M⁺].

trans-2-[(4-Methoxyphenyl)amino)cyclopentanol (4a)
Brown viscous liquid; yield: 184 mg (89%).

IR (KBr): ν \text{max} = 3381 cm⁻¹.

\(^1^H\) NMR (CDCl₃, 500 MHz): δ = 6.78 (dd, J = 3.5, 8.5 Hz, 2 H), 6.67 (dd, J = 3.5, 7.0 Hz, 2 H), 4.07 (q, J = 5.0 Hz, 1 H), 3.75 (s, 3 H), 3.56–3.51 (m, 1 H), 2.68 (br s, 2 H), 2.28–2.18 (m, 1 H), 2.03–1.94 (m, 1 H), 1.86–1.58 (m, 3 H), 1.41 (sextet, J = 6.5 Hz, 1 H).

\(^1^3^C\) NMR (CDCl₃, 125 MHz): δ = 152.7, 141.1, 115.5, 114.9, 77.9, 63.7, 55.8, 32.7, 30.9, 20.9.
trans-2-[(4-Chlorophenyl)amino]cyclopentanol (4c)
Brown viscous liquid; yield: 202 mg (96%).
IR (KBr): ν\text{max} = 3366 cm\text{−1}.
1^1\text{H} NMR (CDCl\text{3}, 500 MHz): δ = 8.51–7.95 (m, 2 H), 6.59 (d, J = 9.5 Hz, 1 H), 4.60 (br s, 1 H), 4.18–4.08 (m, 1 H), 3.69 (quintet, J = 6.5 Hz, 1 H), 2.36–2.22 (m, 1 H), 1.99 (quintet, J = 7.0 Hz, 1 H), 1.92–1.69 (m, 3 H), 1.46 (sextet, J = 7.0 Hz, 1 H).
1^13\text{C} NMR (CDCl\text{3}, 125 MHz): δ = 153.7, 137.2, 126.5, 111.6, 78.0, 61.6, 33.0, 32.7, 21.0.
MS (EI, 70 eV): m/\text{z} = 222 [M^+].

trans-2-[(4-Bromophenyl)amino]cyclopentanol (4d)
Brown liquid; yield: 140 mg (82%).
IR (KBr): ν\text{max} = 3371 cm\text{−1}.
1^1\text{H} NMR (CDCl\text{3}, 500 MHz): δ = 4.21 (dt, J = 4.5, 6.5 Hz, 1 H), 3.30 (br s, 2 H), 2.81–2.71 (m, 4 H), 2.63 (dt, J = 5.5, 8.0 Hz, 1 H), 2.06–1.94 (m, 2 H), 1.89–1.82 (m, 4 H), 1.78–1.58 (m, 4 H).
1^13\text{C} NMR (CDCl\text{3}, 125 MHz): δ = 76.5, 73.3, 52.4, 34.2, 29.1, 23.3, 21.3.
MS (EI, 70 eV): m/\text{z} = 169 [M^+].

trans-2-[(Piperidin-1-yl)cyclopentanol (4j)
Colourless liquid; yield: 139 mg (82%).
IR (KBr): ν\text{max} = 3405 cm\text{−1}.
1^1\text{H} NMR (CDCl\text{3}, 500 MHz): δ = 3.83 (dd, J = 7.5, 8.0 Hz, 1 H), 1.96–1.88 (m, 1 H), 1.80–1.73 (m, 2 H), 1.72–1.57 (m, 2 H), 1.54–1.46 (m, 1 H).
1^13\text{C} NMR (CDCl\text{3}, 125 MHz): δ = 75.3, 74.9, 67.0, 52.0, 34.4, 27.3, 21.4.
MS (EI, 70 eV): m/\text{z} = 171 [M^+].

trans-2-[(Butylamino)cyclopentanol (4l)
Colourless liquid; yield: 135 mg (86%).
IR (KBr): ν\text{max} = 3415 cm\text{−1}.
1^1\text{H} NMR (CDCl\text{3}, 500 MHz): δ = 3.85 (q, J = 6.5 Hz, 1 H), 2.81 (q, J = 7.5 Hz, 1 H), 2.66–2.53 (m, 2 H), 2.04–1.90 (m, 2 H), 1.76–1.59 (m, 2 H), 1.56–1.42 (m, 3 H), 1.32 (sextet, J = 7.0 Hz, 2 H), 1.28–1.22 (m, 1 H), 0.90 (t, J = 7.0 Hz, 3 H).
1^13\text{C} NMR (CDCl\text{3}, 125 MHz): δ = 77.5, 66.7, 48.3, 32.6, 32.3, 30.0, 20.5, 20.3, 13.9.
MS (EI, 70 eV): m/\text{z} = 157 [M^+].

2-Phenyl-2-(phenylamino)ethanol (5a)
Brown liquid; yield: 190 mg (89%).
IR (KBr): ν\text{max} = 3397 cm\text{−1}.
1^1\text{H} NMR (CDCl\text{3}, 500 MHz): δ = 7.31–7.24 (m, 4 H), 7.21–7.17 (m, 1 H), 7.06–7.01 (m, 2 H), 6.64–6.60 (m, 1 H), 6.50 (dd, J = 1.0, 8.5, 2 H), 4.41 (q, J = 4.0 Hz, 1 H), 3.83 (dd, J = 4.0, 11.0 Hz, 1 H), 3.64 (dd, J = 7.0, 11.0 Hz, 1 H).

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13C NMR (CDCl3, 125 MHz): δ = 147.1, 140.1, 129.1, 128.7, 127.5, 126.6, 117.8, 113.8, 67.2, 59.8.

MS (EI, 70 eV): m/z = 213 [M⁺].

1-Phenoxy-3-(phenylamino)propan-2-ol (6a)
Brown liquid; yield: 189 mg (78%).

IR (KBr): νmax = 3558 cm⁻¹.

1H NMR (CDCl3, 500 MHz): δ = 7.32–7.27 (m, 2 H), 7.21–7.16 (m, 2 H), 7.00–6.96 (m, 1 H), 6.94–6.90 (m, 2 H), 6.76–6.71 (m, 1 H), 6.69–6.65 (m, 2 H), 4.28–4.20 (m, 1 H), 4.15–3.99 (m, 2 H), 3.43 (dd, J = 4.5, 13.0 Hz, 1 H), 3.29 (dd, J = 7.0, 13.0 Hz, 1 H).

13C NMR (CDCl3, 125 MHz): δ = 158.3, 147.9, 129.5, 129.2, 121.2, 117.9, 114.4, 113.2, 69.9, 68.7, 46.5.

MS (EI, 70 eV): m/z = 243 [M⁺].

Supporting Information for this article is available online at http://www.thieme-connect.com/ejournals/toc/synthesis.

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