

## Supporting Information

### Synthesis of Five-, Six-, and Seven-Membered Ring Lactams by Cp\*Rh Complex-Catalyzed Oxidative *N*-Heterocyclization of Amino Alcohols

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#### Experimental Section:

**General:** All reactions and manipulations were carried out under an atmosphere of argon by means of standard Schlenk techniques. <sup>1</sup>H- and <sup>13</sup>C-NMR spectra were recorded on JEOL A-500 and EX-270 spectrometers. Infrared spectra were recorded on a HORIBA FT-300 spectrometer. Gas chromatography (GC) analyses were performed on a Shimadzu GC-14A gas chromatograph with capillary column (Shimadzu CBP1-M25-025). Column chromatography was carried out by using Wako-gel C-200. Melting points were determined under air on a Yanagimoto micro melting point apparatus. Elemental analyses were carried out at the Microanalysis Center of Kyoto University. Solvents were dried by using standard procedures and distilled prior to use. The catalysts Cp\*Rh(OAc)<sub>2</sub>•H<sub>2</sub>O,<sup>1</sup> RhCl(PPh<sub>3</sub>)<sub>3</sub>,<sup>2</sup> and [Cp\*IrCl<sub>2</sub>]<sub>2</sub><sup>3</sup> were prepared according to literature methods. 2-Aminophenethyl alcohol (**3a**) was commercial material.

3-(2-Aminophenyl)-1-propanol (1a),<sup>4</sup> 3-(2-amino-5-chlorophenyl)-1-propanol (1b),<sup>5</sup>  
3-(2-amino-5-methoxyphenyl)-1-propanol (1f),<sup>5</sup> 4-(2-aminophenyl)-1-butanol (1h),<sup>5</sup>  
2-(2-aminophenyl)-1-propanol (3b),<sup>6</sup> 2-amino-6-chlorophenethyl alcohol (3c),<sup>6</sup>  
2-iodo-4-(methoxycarbonyl)aniline,<sup>7</sup> and 4-cyano-2-iodoaniline<sup>7</sup> were prepared according to literature methods. Other reagents were used as obtained from commercial sources.

**[Cp\*RhCl<sub>2</sub>]<sub>2</sub>:** The known title complex<sup>1</sup> was prepared as follows. To RhCl<sub>3</sub>•3H<sub>2</sub>O (1.02 g, 3.89 mmol) in 25 mL of methanol was added excess pentamethylcyclopentadiene (1.0 mL). The mixture was stirred under reflux for 21 h. After the mixture was cooled to room temperature, the product was isolated by filtration and washing with ether. Yield 1.02 g (85%).

**4-Acetyl-2-iodoaniline:** A solution of CaCO<sub>3</sub> (4.7 g, 47 mmol) in water (12 mL) was added to a solution of 4-acetylaniline (4.05 g, 30.0 mmol) in methanol (20 mL), followed by a solution of ICl (5.16 g, 31.8 mmol) in methanol (20 mL). The mixture was stirred at room temperature for 15 h, and then diluted with ether and quenched with water. The aqueous layer was extracted with ether and the combined organic phases were dried with Na<sub>2</sub>SO<sub>4</sub>. After filtration and removal of the solvent in vacuo, the residue was purified by column chromatography. Yield 7.59 g (97%). This product (4-acetyl-2-iodoaniline) was identified by spectral comparison with literature data.<sup>8</sup>

**2-Iodo-4-methylaniline:** A solution of CaCO<sub>3</sub> (6.0 g, 60 mmol) in water (16 mL) was added to a solution of p-toluidine (4.31 g, 40.3 mmol) in methanol (30 mL), followed by a solution of ICl (6.65 g, 41.0 mmol) in methanol (32 mL). The mixture was stirred at room temperature for 15 h, and then diluted with ether and quenched with water. The aqueous layer was extracted with ether and the combined organic phases were dried with Na<sub>2</sub>SO<sub>4</sub>. After filtration and removal of the

solvent in vacuo, the residue was purified by column chromatography. Yield 6.36 g (68%). This product (2-iodo-4-methylaniline) was identified by spectral comparison with literature data.<sup>9</sup>

**3-(2-Amino-5-methoxycarbonylphenyl)-2-propyn-1-ol:** To a solution of 2-iodo-4-(methoxycarbonyl)aniline (1.39 g, 5.00 mmol) in triethylamine (20 mL) was added PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (72.9 mg, 0.103 mmol), CuI (9.6 mg, 0.050 mmol), and propargyl alcohol (0.44 mL, 7.6 mmol). The mixture was stirred at room temperature for 4.5 h. After filtration and evaporation of the solvent, the residue was extracted with ethyl acetate and purified by column chromatography. Yield 0.677 g (66%). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.97 (d, *J* = 2 Hz, 1H, aromatic), 7.79 (dd, *J* = 9, 2 Hz, 1H, aromatic), 6.65 (d, *J* = 9 Hz, 1H, aromatic), 4.67 (br, 2H, NH<sub>2</sub>), 4.54 (s, 2H, CH<sub>2</sub>), 3.85 (s, 3H, CO<sub>2</sub>Me), 2.28 (br, 1H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.7 MHz): δ 166.6 (C), 151.7 (C), 134.6 (CH), 131.6 (CH), 119.2 (C), 113.3 (CH), 106.3 (C), 93.1 (C), 81.2 (C), 51.8 (CH<sub>3</sub>), 51.6 (CH<sub>2</sub>). IR (nujol): ν 1705, 1693, 1637, 1597, 1506, 1294, 1234, 1018, 955 cm<sup>-1</sup>.

**3-(2-Amino-5-acetylphenyl)-2-propyn-1-ol:** To a solution of 4-acetyl-2-iodoaniline (7.59 g, 29.1 mmol) in triethylamine (113 mL) was added PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (410 mg, 0.585 mmol), CuI (58.6 mg, 0.307 mmol), and propargyl alcohol (2.54 mL, 43.6 mmol). The mixture was stirred at room temperature under hydrogen atmosphere for 30 h. After evaporation of the solvent, the residue was extracted with dichloromethane and purified by column chromatography. Yield 4.12 g (73%). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.91 (d, *J* = 2 Hz, 1H, aromatic), 7.77 (dd, *J* = 9, 2 Hz, 1H, aromatic), 6.67 (d, *J* = 9 Hz, 1H, aromatic), 4.71 (br, 2H, NH<sub>2</sub>), 4.56 (d, *J* = 5 Hz, 2H, CH<sub>2</sub>), 2.50 (s, 3H, COMe), 2.07 (brt, 1H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.7 MHz): δ 195.9 (C), 151.9 (C), 134.0 (CH), 130.6 (CH), 127.4 (C), 113.4 (CH), 106.1 (C), 93.2 (C), 81.2 (C), 51.6 (CH<sub>2</sub>), 26.0 (CH<sub>3</sub>).

IR (nujol):  $\nu$  1645, 1622, 1581, 1331, 1302, 1261, 1041, 821  $\text{cm}^{-1}$ .

**3-(2-Amino-5-cyanophenyl)-2-propyn-1-ol:** To a solution of 4-cyano-2-iodoaniline (4.89 g, 20.0 mmol) in triethylamine (80 mL) was added  $\text{PdCl}_2(\text{PPh}_3)_2$  (283 mg, 0.403 mmol), CuI (38.2 mg, 0.201 mmol), and propargyl alcohol (1.75 mL, 30.1 mmol). The mixture was stirred at room temperature for 4.5 h. After filtration and evaporation of the solvent, the residue was extracted with ethyl acetate and purified by column chromatography. Yield 1.96 g (57%).  $^1\text{H}$  NMR (acetone- $\text{d}_6$ , 500 MHz):  $\delta$  7.51 (d,  $J = 2$  Hz, 1H, aromatic), 7.39 (dd,  $J = 9, 2$  Hz, 1H, aromatic), 6.86 (d,  $J = 9$  Hz, 1H, aromatic), 5.93 (br, 2H,  $\text{NH}_2$ ), 4.46 (d,  $J = 6$  Hz, 2H,  $\text{CH}_2$ ), 4.32 (t,  $J = 6$  Hz, 1H, OH).  $^{13}\text{C}$  NMR (acetone- $\text{d}_6$ , 125.7 MHz):  $\delta$  153.8 (C), 136.6 (CH), 133.9 (CH), 119.9 (C), 114.7 (CH), 107.9 (C), 99.1 (C), 96.4 (C), 79.5 (C), 51.1 ( $\text{CH}_2$ ). IR (nujol):  $\nu$  2222, 1630, 1504, 1022, 822  $\text{cm}^{-1}$ .

**3-(2-Amino-5-methylphenyl)-2-propyn-1-ol:** To a solution of 2-iodo-4-methylaniline (3.50 g, 15.0 mmol) in triethylamine (60 mL) was added  $\text{PdCl}_2(\text{PPh}_3)_2$  (227 mg, 0.323 mmol), CuI (35.2 mg, 0.185 mmol), and propargyl alcohol (1.75 mL, 30.1 mmol). The mixture was stirred at room temperature for 4.5 h. After filtration and evaporation of the solvent, the residue was extracted with dichloromethane and purified by column chromatography. Yield 1.17 g (48%).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.06 (s, 1H, aromatic), 6.92 (d,  $J = 8$  Hz, 1H, aromatic), 6.60 (d,  $J = 8$  Hz, 1H, aromatic), 4.49 (s, 2H,  $\text{CH}_2$ ), 3.99 (br,  $\text{NH}_2$  or OH), 2.18 (s, 3H,  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.7 MHz):  $\delta$  145.5 (C), 132.3 (CH), 130.7 (CH), 127.3 (C), 114.7 (CH), 107.4 (C), 92.5 (C), 82.3 (C), 51.4 ( $\text{CH}_2$ ), 20.2 ( $\text{CH}_3$ ). IR (nujol):  $\nu$  1620, 1504, 1317, 1246, 1151, 1020, 1010, 816  $\text{cm}^{-1}$ .

**3-(2-Amino-5-methoxycarbonylphenyl)-1-propanol (1c):** A mixture of 3-(2-amino-5-methoxycarbonylphenyl)-2-propyn-1-ol (0.897 g, 4.37 mmol), Pd on carbon (5%Pd) (441 mg), and methanol (10 mL) was placed in a flask. The atmosphere was changed to hydrogen, and the mixture was stirred at room temperature for 48 h. After filtration, the product **1c** was isolated by Kugelrohr distillation (180 °C, 0.5 mmHg). Yield 0.561 g (61 %). m.p. 83.1-84.1 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.75 (s, 1H, aromatic), 7.72 (d, *J* = 9 Hz, aromatic), 6.63 (d, *J* = 8 Hz, 1H, aromatic), 3.85 (s, 3H, CO<sub>2</sub>Me), 3.67 (t, *J* = 6 Hz, 2H, -CH<sub>2</sub>OH), 2.64 (t, *J* = 7 Hz, 2H, ArCH<sub>2</sub>-), 1.88 (m, 2H, -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>-). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.7 MHz): δ 167.4 (C), 149.2 (C), 131.6 (CH), 129.3 (CH), 124.9 (C), 119.6 (C), 114.5 (CH), 61.4 (CH<sub>2</sub>), 51.6 (CH<sub>3</sub>), 31.6 (CH<sub>2</sub>), 26.7 (CH<sub>2</sub>). IR (nujor): ν 3400, 3390, 1684, 1648, 1600, 1295, 1263, 1232, 1076, 999, 933, 839 cm<sup>-1</sup>. Anal Calcd for C<sub>11</sub>H<sub>15</sub>NO<sub>3</sub>: C, 63.14; H, 7.23; N, 6.70. Found: C, 63.39; H, 7.14; N, 6.46.

**3-(2-Amino-5-acetylphenyl)-1-propanol (1d):** A mixture of 3-(2-amino-5-acetylphenyl)-2-propyn-1-ol (1.14 g, 6.04 mmol), Pd on carbon (5%Pd) (603 mg), and methanol (12 mL) was placed in a flask. The atmosphere was changed to hydrogen, and the mixture was stirred at room temperature for 50 h. After filtration, the product **1d** was isolated by column chromatography. Yield 0.556 g (48 %). m.p. 80.3-80.9 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.72 (d, *J* = 2 Hz, 1H, aromatic), 7.67 (dd, *J* = 8, 2 Hz, 1H, aromatic), 6.64 (d, *J* = 8 Hz, 1H, aromatic), 3.69 (t, *J* = 6 Hz, 2H, -CH<sub>2</sub>OH), 2.66 (t, *J* = 8 Hz, 2H, ArCH<sub>2</sub>-), 2.51 (s, 3H, COMe), 1.88 (m, 2H, -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>-). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.7 MHz): δ 196.9 (C), 149.7 (C), 130.7 (CH), 128.8 (CH), 127.8 (C), 124.9 (C), 114.2 (CH), 61.4 (CH<sub>2</sub>), 31.6 (CH<sub>2</sub>), 26.8 (CH<sub>2</sub>), 26.0 (CH<sub>3</sub>). IR (nujor): ν 3383, 3219, 1651, 1587, 1504, 1282, 1255, 1051, 820 cm<sup>-1</sup>. Anal Calcd for C<sub>11</sub>H<sub>15</sub>NO<sub>2</sub>: C, 68.37; H, 7.82; N, 7.25. Found: C, 68.50; H, 7.66; N, 6.96.

**3-(2-Amino-5-cyanophenyl)-1-propanol (1e):** A mixture of 3-(2-amino-5-cyanophenyl)-2-propyn-1-ol (1.40 g, 8.13 mmol), Pd on carbon (5%Pd) (715 mg), and methanol (18 mL) was placed in a flask. The atmosphere was changed to hydrogen, and the mixture was stirred at room temperature for 50 h. After filtration, the product **1e** was isolated by column chromatography. Yield 1.08 g (76 %). m.p. 80.9-81.5 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.29 (m, 2H, aromatic), 6.63 (d, *J* = 8 Hz, 1H, aromatic), 4.40 (br, 2H, NH<sub>2</sub>), 3.69 (t, *J* = 6 Hz, 2H, -CH<sub>2</sub>OH), 2.62 (t, *J* = 7 Hz, 2H, ArCH<sub>2</sub>-), 1.85 (m, 2H, -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>-), 1.72 (br, 1H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.7 MHz): δ 148.9 (C), 133.7 (CH), 131.5 (CH), 125.9 (C), 120.3 (C), 114.9 (CH), 100.1 (C), 61.2 (CH<sub>2</sub>), 31.3 (CH<sub>2</sub>), 26.4 (CH<sub>2</sub>). IR (nujor): ν 3402, 3338, 3219, 2214, 1647, 1604, 1504, 1300, 1215, 1061, 1022, 818 cm<sup>-1</sup>. Anal Calcd for C<sub>10</sub>H<sub>12</sub>N<sub>2</sub>O: C, 68.16; H, 6.86; N, 15.90. Found: C, 68.43; H, 6.88; N, 15.62.

**3-(2-Amino-5-methylphenyl)-1-propanol (1g):** A mixture of 3-(2-amino-5-methylphenyl)-2-propyn-1-ol (0.806 g, 5.00 mmol), Pd on carbon (5%Pd) (602 mg), and methanol (10 mL) was placed in a flask. The atmosphere was changed to hydrogen, and the mixture was stirred at room temperature for 16 h. After filtration, the product **1g** was isolated by Kugelrohr distillation (160 °C, 0.7 mmHg) followed by recrystallization with diisopropyl ether. Yield 0.580 g (70 %). m.p. 67.9-68.6 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 270 MHz): δ 6.85-6.82 (m, 2H, aromatic), 6.59 (d, *J* = 8 Hz, 1H, aromatic), 3.59 (t, *J* = 6 Hz, 2H, -CH<sub>2</sub>OH), 3.30 (br, NH<sub>2</sub> or OH), 2.59 (t, *J* = 7 Hz, 2H, ArCH<sub>2</sub>-), 2.23 (s, 3H, CH<sub>3</sub>), 1.84 (m, 2H, -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>-). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 67.8 MHz): δ 141.3 (C), 130.3 (CH), 128.5 (C), 127.4 (CH), 126.8 (C), 116.3 (CH), 61.3 (CH<sub>2</sub>), 32.4 (CH<sub>2</sub>), 26.8 (CH<sub>2</sub>), 20.5 (CH<sub>3</sub>). IR (nujor): ν 1643, 1583, 1504, 1279, 1157, 1055, 903, 812 cm<sup>-1</sup>. Anal Calcd for C<sub>10</sub>H<sub>15</sub>NO: C, 72.69; H, 9.15; N, 8.48. Found: C, 72.68; H, 9.18; N,

8.44.

***N*-Methyl-2-aminophenethyl alcohol (3d):** To a solution of 2-aminophenethyl alcohol (0.823 g, 6.00 mmol) in formic acid (12 mL) was added acetic anhydride (5 mL) at 55 °C. After the addition was complete, the mixture was stirred at room temperature for 1 h. Then ice-water (4.8 mL) was added and the solution was concentrated by evaporation. Extraction with ethyl acetate, washing with HCl<sub>aq</sub>, drying with Na<sub>2</sub>SO<sub>4</sub>, and evaporation of the solvent gave a crude product of *N*-formyl-2-aminophenethyl alcohol, which was used in the next reaction without further purification.

To a suspension of LiAlH<sub>4</sub> (0.680 g, 17.9 mmol) in THF (6 mL) was added a solution of *N*-formyl-2-aminophenethyl alcohol (crude product prepared as above) in THF (12 mL) at 0 °C. The solution was stirred under reflux for 15 h. Water (2.7 mL) and 4 N NaOH<sub>aq</sub> (0.7 mL) was added at 0 °C. Filtration, extraction with ethyl acetate, drying with Na<sub>2</sub>SO<sub>4</sub>, and evaporation of the solvent gave a crude product of **3d**, which was purified by column chromatography. Yield 0.660 g (75%). This product (*N*-methyl-2-aminophenethyl alcohol) was identified by spectral comparison with literature data.<sup>10</sup>

**General procedure for the synthesis of 3,4-dihydro-2(1*H*)-quinolinone under various conditions shown in Table 1:** Under an atmosphere of argon in a heavy-walled glass reactor, 3-(2-aminophenyl)-1-propanol (0.50 mmol), catalyst (5.0%Rh), base (10%), and acetone (12.5 mL) were placed. The reactor was sealed, and the mixture was stirred at 100 °C (oil bath temp.) for 20 h. The yield of 3,4-dihydro-2(1*H*)-quinolinone was determined by GC analysis using undecane as an internal standard.

**General procedure for the synthesis of 3,4-dihydro-2(1*H*)-quinolinones and 1,3,4,5-tetrahydro-2*H*-1-benzazepin-2-one catalyzed by [Cp\**RhCl*<sub>2</sub>]<sub>2</sub> shown in Table 2:**

Under an atmosphere of argon in a heavy-walled glass reactor, substrate (0.50 mmol), [Cp\**RhCl*<sub>2</sub>]<sub>2</sub> (0.0125 mmol, 5.0%*Rh*), K<sub>2</sub>CO<sub>3</sub> (0.050 mmol, 10%), and acetone (12.5 mL) were placed. The reactor was sealed, and the mixture was stirred at 100 °C (oil bath temp.) for 20 h. After evaporation of the solvent, the products were isolated by column chromatography. The products **2a**,<sup>11</sup> **2b**,<sup>12</sup> **2c**,<sup>13</sup> **2e**,<sup>11</sup> **2f**,<sup>11</sup> **2g**,<sup>11</sup> and **2h**<sup>14</sup> were identified by spectral comparison with literature data.

**6-Acetyl-3,4-dihydro-2(1*H*)-quinolinone (2d):** m.p. 161.0-162.0 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 9.0 (br, 1H, NH), 7.82-7.79 (m, 2H, aromatic), 6.88 (d, *J* = 8 Hz, 1H, aromatic), 3.05 (t, *J* = 8 Hz, 2H, -COCH<sub>2</sub>-), 2.69 (m, 2H, Ar-CH<sub>2</sub>-), 2.57 (s, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.7 MHz): δ 196.7 (C), 171.8 (C), 141.4 (C), 132.3 (C), 128.6 (CH), 128.4 (CH), 123.5 (C), 115.2 (CH), 30.4 (CH<sub>2</sub>), 26.4 (CH<sub>3</sub>), 25.1 (CH<sub>2</sub>). IR (nujor): ν 1680, 1606, 1504, 1324, 1313, 1279, 1250, 1182, 972, 827 cm<sup>-1</sup>. Anal. Calcd for C<sub>11</sub>H<sub>15</sub>NO<sub>2</sub>: C, 69.83; H, 5.86; N, 7.40. Found: C, 69.73; H, 5.83; N, 7.11.

**General procedure for the synthesis of oxindoles catalyzed by [Cp\**RhCl*<sub>2</sub>]<sub>2</sub> shown in Table**

**3:** Under an atmosphere of argon in a glass reactor with reflux condenser, substrate (1.0 mmol), [Cp\**RhCl*<sub>2</sub>]<sub>2</sub> (0.015 mmol, 3.0%*Rh*), K<sub>2</sub>CO<sub>3</sub> (0.10 mmol), and acetone (20 mL) were placed. The mixture was stirred under reflux for 8 h. After evaporation of the solvent, the products were isolated by column chromatography. The products **4a**,<sup>15</sup> **4b**,<sup>16</sup> **4c**,<sup>17</sup> and **4d**<sup>18</sup> were identified by spectral comparison with literature data.

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