Proline-catalyzed Dehydrogenative Cross-Coupling Reaction between Chromene and Aldehydes

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1. General Information

Unless otherwise indicated, all reagents were purchased from commercial distributors and used without further purification. $^1$H and $^{13}$C NMR were recorded with $^1$H at 400 MHz and $^{13}$C at 100 MHz, respectively, using tetramethylsilane as an internal reference. Mass spectroscopy data were collected on an HRMS-ESI instrument. Melting points were uncollected. Flash column chromatography was performed over silica gel 200-300. Chromene was prepared according to a reported method.
2. Optimizations of the DCC Reaction

Table S1. Screening of reaction conditions.a

![Chemical Structures](image)

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<th>Additive 2 (75 mg)</th>
<th>Solvent (2mL)</th>
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a Reaction conditions: 1 (0.3 mmol), LiClO₄ (1.2 eq), 4 Å MS (75 mg), and DDQ (0.36 mmol) in solvent (2 mL). 0°C; 2a (0.9 mmol), organocatalyst (20 mol%), 20 °C, 24 h; NaBH₄ (1.0 mmol).

b Under air.

c Isolated yield.
3. Experimental Procedures

General procedure for the DCC reaction of chromene 1 with aldehyde 2a-i.

The mixture of chromene (39.6 mg, 0.3 mmol), LiClO$_4$ (38.16 mg, 0.36 mmol), 4 Å MS (75 mg), DDQ (81.72 mg, 0.36 mmol) was stirred for 0.5 h in freshly distilled acetonitrile (2 mL) at 0°C. Then aldehyde (0.9 mmol) and L-proline (6.9 mg, 0.06 mmol) was added, and the reaction mixture was stirred for 24 h at room temperature (about 20°C). After evaporation of solvent under reduced pressure, NaBH$_4$ (37.83 mg, 1.0 mmol) and ethanol (2 mL) were added to the mixture. The reaction mixture was stirred for 2 h at 0°C. After water (10 mL) was added, the mixture was extracted with EtOAc (10 mL ×3). The combined organic layer was dried over Na$_2$SO$_4$ and evaporated. The residue was purified by column chromatography (silica gel, ethyl acetate/petroleum ether = 1/10 or 1/20 as eluent) to give desired product 3.

4. Characterization of Products:

2-(2H-chromen-2-yl) butan-1-ol 3a

Yield: 84%; light yellow oil. Low polar diastereomer$^3$: $^1$H NMR (400 MHz, CDCl$_3$), δ (ppm): 7.03 (td, $J = 7.6$ Hz, $J = 1.6$ Hz, 1H), 6.90 (dd, $J = 7.4$ Hz, $J = 1.8$ Hz, 1H), 6.79 (td, $J = 7.4$ Hz, $J = 1.1$ Hz, 1H), 6.70 (d, $J = 8.4$ Hz, 1H), 6.38 (dd, $J = 10$ Hz, $J = 1.6$ Hz, 1H), 5.69 (dd, $J = 10$ Hz, $J = 3.2$ Hz 1H), 4.89-4.86 (m, 1H), 3.83-3.80 (m, 1H), 3.75-3.71 (m, 1H), 1.87 (bs, 1H), 1.76-1.71 (m, 1H), 1.54-1.46 (m, 2H), 0.92 (t, $J = 7.4$ Hz, 3H). High polar diastereomer$^3$: $^1$H NMR (400 MHz, CDCl$_3$), δ (ppm): 7.02 (t, $J = 7.6$ Hz, 1H), 6.88 (d, $J = 7.6$ Hz, 1H), 6.77 (t, $J = 7.4$ Hz, 1H), 6.67 (d, $J = 8.4$ Hz, 1H), 6.38 (dd, $J = 10$ Hz, $J = 1.6$ Hz, 1H), 5.60 (dd, $J = 10$ Hz, $J = 2.8$ Hz 1H), 5.08 (d, $J = 2.4$ Hz, 1H), 3.80-3.75 (m, 2H), 1.87 (bs, 1H), 1.75-1.70 (m, 1H), 1.49-1.38 (m, 2H), 0.92 (t, $J = 7.4$ Hz, 3H). MS (EI): $m/z$: 204 [M$^+$], 131(100), 77, 31.

2-(2H-chromen-2-yl) propan-1-ol 3b

Yield: 76%; light yellow oil. Low polar diastereomer$^3$: $^1$H NMR (400 MHz, CDCl$_3$), δ (ppm): 7.10 (td, $J = 7.6$ Hz, $J = 1.6$ Hz, 1H), 6.96 (dd, $J = 8.8$ Hz, $J = 1.4$ Hz, 1H), 6.85 (td, $J = 7.4$ Hz, $J = 1.1$ Hz, 1H), 6.77 (d, $J = 8$ Hz, 1H), 6.44 (dd, $J = 10$ Hz, $J = 1.2$ Hz, 1H), 5.76 (dd, $J = 10$ Hz, 1H).
Hz, \( J = 3.2 \) Hz (1H), 4.83-4.80 (m, 1H), 3.75 (d, \( J = 5.2 \) Hz, 2H), 2.16-2.10 (m, 1H), 2.03 (bs, 1H), 1.02 (d, \( J = 6.8 \) Hz, 3H). High polar diastereomer: \(^1\)H NMR (400 MHz, CDCl\(_3\)), \( \delta \) (ppm): 7.08 (td, \( J = 7.6 \) Hz, \( J = 1.6 \) Hz, 1H), 6.94 (dd, \( J = 7.6 \) Hz, \( J = 1.6 \) Hz, 1H), 6.83 (td, \( J = 7.6 \) Hz, \( J = 1.2 \) Hz, 1H), 6.74 (\( d \), \( J = 8 \) Hz, 1H), 6.45 (dd, \( J = 9.8 \) Hz, \( J = 1.8 \) Hz, 1H), 5.63 (dd, \( J = 10 \) Hz, \( J = 2.8 \) Hz, 1H), 5.10 (\( d \), \( J = 2.4 \) Hz, 1H), 3.83-3.70 (m, 2H), 2.03-1.99 (m, 1H), 1.04 (d, \( J = 7.2 \) Hz, 3H). MS (EI): \( m/z \): 190 [M\(^+\)], 131 (100), 77, 31.

2-(2H-chromen-2-yl) pentan-1-ol 3c

Yield: 66%; yellow oil. Mixture of two diastereomers: \(^1\)H NMR (400 MHz, CDCl\(_3\)), \( \delta \) (ppm): 7.12-7.06 (m, 1H), 6.98-6.93 (m, 1H), 6.88-6.82 (m, 1H), 6.78 (\( d \), \( J = 8.0 \) Hz, 0.54H), 6.74 (\( d \), \( J = 8.0 \) Hz, 0.46H), 6.47 (\( d \), \( J = 10 \) Hz, 1H), 5.76 (dd, \( J = 10 \) Hz, \( J = 3.2 \) Hz, 0.54H), 5.65 (dd, \( J = 10 \) Hz, \( J = 2.8 \) Hz, 0.46H), 5.15-5.13 (m, 0.46H), 4.94-4.91 (m, 0.54H), 3.89-3.75 (m, 2H), 2.03 (bs, 1H), 1.9-1.88 (m, 1H), 1.51-1.31 (m, 4H), 0.95-0.90 (m, 3H). MS (EI): \( m/z \): 218 [M\(^+\)], 131 (100), 77, 31.

2-(2H-chromen-2-yl) hexan-1-ol 3d

Yield: 80%; yellow oil. Mixture of two diastereomers: \(^1\)H NMR (400 MHz, CDCl\(_3\)), \( \delta \) (ppm): 7.04-6.99 (m, 1H), 6.90-6.86 (m, 1H), 6.80-6.74 (m, 1H), 6.70 (\( d \), \( J = 8.0 \) Hz, 0.53H), 6.67 (\( d \), \( J = 8.0 \) Hz, 0.47H), 6.37 (\( d \), \( J = 10 \) Hz, 1H), 5.69 (dd, \( J = 10 \) Hz, \( J = 3.2 \) Hz, 0.52H), 5.58 (dd, \( J = 10 \) Hz, \( J = 2.8 \) Hz, 0.48H), 5.06 (dd, \( J = 6.0 \) Hz, \( J = 3.2 \) Hz, 0.47H), 4.87-4.84 (m, 0.53H), 3.82-3.68 (m, 2H), 1.932 (bs, 1H), 1.81-1.78 (m, 1H), 1.43-1.40 (m, 2H), 1.30-1.22 (m, 4H), 0.85-0.80 (m, 3H). MS (EI): \( m/z \): 232 [M\(^+\)], 131 (100), 77, 31.

2-(2H-chromen-2-yl) heptan-1-ol 3e

Yield: 90%; yellow oil. Mixture of two diastereomers: \(^1\)H NMR (400 MHz, CDCl\(_3\)), \( \delta \) (ppm): 7.11-7.05 (m, 2H), 6.97-6.92 (m, 2H), 6.87-6.81 (m, 2H), 6.76 (\( d \), \( J = 8.4 \) Hz, 1H), 6.73 (\( d \), \( J = 8.0 \) Hz, 1H), 6.44 (\( d \), \( J = 10 \) Hz, 2H), 5.75 (dd, \( J = 9.8 \) Hz, \( J = 3.0 \) Hz, 1H), 5.65 (dd, \( J = 10.0 \) Hz, \( J = 3.2 \) Hz, 1H), 5.14-5.12 (m, 1H), 4.94-4.91 (m, 1H), 3.88-3.74 (m, 4H), 2.25 (bs, 2H), 1.89-1.84 (m, 2H), 1.52-1.46 (m, 4H), 1.44-1.26 (m, 12H), 0.90-0.85 (m, 6H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)), \( \delta \) (ppm): 153.7, 153.4, 129.2, 126.6, 125.1, 125.0, 124.7, 124.3, 122.2, 121.8, 121.4, 121.2, 115.8, 115.6, 77.3, 76.9, 63.0,
62.6, 45.5, 45.4, 32.1 (2 C), 27.2, 27.0, 26.9 (2 C), 25.9(2 C), 22.6(2 C), 14.1(2 C).

HRMS (TOF MS EI\(^+\)): m/z calcd for C\(_{16}\)H\(_{22}\)O\(_2\), 246.1620; found 246.1621.

2-(2H-chromen-2-yl) octan-1-ol 3f

Yield: 82%; yellow oil. Mixture of two diastereomers: \(^1\)H NMR (400 MHz, CDCl\(_3\)), \(\delta\) (ppm): 7.12-7.06 (m, 1H), 7.00-6.93 (m, 1H), 6.88-6.82(m, 1H), 6.77 (d, \(J = 8.0\) Hz, 0.54H), 6.74 (d, \(J = 8.4\) Hz, 0.48H), 6.45 (d, \(J = 10.0\) Hz, 1H), 5.76 (dd, \(J = 9.8\) Hz, \(J = 6.0\) Hz, 0.54H), 5.66 (dd, \(J = 10.0\) Hz, \(J = 2.8\) Hz,0.48H), 5.15-5.13(m, 0.49H), 4.94-4.91 (m, 0.54H), 3.90-3.75 (m, 2H), 2.02(bs, 1H),1.90-1.85 (m, 1H),1.52-1.46 (m,2H), 1.28-1.26 (m,8H), 0.88-0.86 (m, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)), \(\delta\) (ppm): 153.7, 153.4, 129.2, 126.6, 125.2, 125.1, 124.7, 124.2, 122.2, 121.8, 121.4, 121.2 115.8, 115.6, 77.4, 77.0, 63, 2, 62.7, 45.4 (2 C), 31.8, 31.7, 29.6, 29.5, 27.5 (2 C), 27.2, 27.1, 25.9(2 C), 22.6 (2C), 14.1 (2 C). HRMS (TOF MS EI\(^+\)): m/z calcd for C\(_{17}\)H\(_{24}\)O\(_2\), 260.1776; found 260.1780.

2-(2H-chromen-2-yl) nonan-1-ol 3g

Yield: 90%; light yellow oil. Mixture of two diastereomers: \(^1\)H NMR (400 MHz, CDCl\(_3\)), \(\delta\) (ppm): 7.12-7.06 (m, 2H), 6.97-6.93 (m, 2H), 6.87-6.81 (m, 2H), 6.77 (d, \(J = 8.0\) Hz, 1H), 6.74 (d, \(J = 8.0\) Hz, 1H), 6.44(d, \(J = 10\) Hz,2H), 5.76 (dd, \(J = 10.0\) Hz, \(J = 3.2\)Hz, 1H), 5.66 (dd, \(J = 9.8\) Hz, \(J = 3.0\) Hz, 1H), 5.15-5.12 (m, 1H), 4.94-4.91(m,1H), 3.89-3.75 (m, 4H), 2.05 (bs, 2H), 1.90-1.86 (m, 2H), 1.51-1.45 (m, 4H), 1.31-1.24 (m, 18H), 0.90-0.85 (m, 6H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)), \(\delta\) (ppm): 153.7, 153.4, 129.2, 126.6, 125.2, 125.1, 124.7, 124.2, 122.2, 121.8, 121.4, 121.2, 115.8, 115.6, 77.4, 77.0, 63.2, 62.7, 45.4 (2 C), 31.8, 31.7, 29.6, 29.5, 27.5 (2 C), 27.2, 27.1, 25.9 (2C), 22.6 (2C), 14.1 (2 C). HRMS (TOF MS EI\(^+\)): m/z calcd for C\(_{18}\)H\(_{26}\)O\(_2\), 274.1933; found 260.1936.

2-(2H-chromen-2-yl)-3-methylbutan-1-ol 3h

Yield: 93%; yellow oil. Low polar diastereomer: \(^1\)H NMR (CDCl\(_3\), 400 MHz), \(\delta\) (ppm): 7.09 (td, \(J = 7.6\) Hz, \(J = 1.5\) Hz, 1H), 6.97 (dd, \(J = 7.6\) Hz, \(J = 1.6\) Hz, 1H), 6.86 (td, \(J = 7.4\) Hz, \(J = 1.1\)Hz, 1H), 6.76 (d, \(J = 8.0\) Hz, 1H), 6.45 (dd, \(J = 10.0\) Hz, \(J = 2.0\) Hz,1H), 5.72 (dd, \(J = 10.0\) Hz, \(J = 2.8\) Hz, 1H), 5.13-5.11 (m, 1H), 3.97-3.88 (m, 2H), 2.28-2.06 (m, 2H), 1.62-1.57 (m, 1H), 1.04 (d, \(J = 6.8\) Hz, 3H), 1.00 (d, \(J = 6.8\) Hz, 3H). \(^{13}\)C NMR (100 MHz,
CDCl₃), δ (ppm): 153.2, 129.3, 126.6, 126.0, 124.9, 122.2, 121.6, 115.9, 76.6, 61.4, 51.2, 26.0, 21.1, 19.8; High polar diastereomer: ¹H NMR (CDCl₃, 400 MHz), δ (ppm): 7.09 (td, J = 3.6 Hz, J = 1.6 Hz, 1H), 6.96 (dd, J = 7.6 Hz, J = 1.6 Hz, 1H), 6.85 (td, J = 7.4 Hz, J = 1.1 Hz, 1H), 6.76 (d, J = 8.0 Hz, 1H), 6.65 (dd, J = 10 Hz, J = 2.0 Hz, 1H), 5.78 (dd, J = 9.8 Hz, J = 3.0 Hz, 1H), 5.17 (dd, J = 5.2 Hz, J = 2.4 Hz,1H), 3.91-3.84 (m, 2H), 2.07-1.99 (m, 1H), 1.90 (bs , 1H), 1.82-1.76 (m, 1H), 1.02 (d, J = 6.8 Hz, 3H), 1.00 (d, J = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 153.6, 129.2, 126.5, 125.0, 124.5, 122.0, 121.2, 115.8, 76.1, 61.1, 51.0, 25.9, 21.5, 20.2; HRMS (TOF MS El⁺): m/z calcd for C₁₄H₁₈O₂, 218.1307; found 218.1304.

2-(2H-chromen-2-yl)-3-phenylpropan-1-ol 3i²

Yield: 64%; yellow oil. Mixture of two diastereomers: ¹H NMR (400 MHz, CDCl₃), δ (ppm): 7.21-7.17 (m, 3H), 7.15-7.10 (m, 2H), 7.06-7.01(m, 1H), 6.91-6.87 (m, 1H), 6.81-6.77(m, 1H), 6.75 (d, J = 8.8 Hz, 0.55H), 6.71 (d, J = 8 Hz, 0.5H) 6.42-6.37 (m, 1H), 5.67 (t, J = 2.8 Hz, 0.52H), 5.65 (t, J = 2.8 Hz, 0.48H), 5.10-5.08 (m, 0.51H), 4.88-4.86 (m, 0.49H), 3.67-3.66 (m, 1H), 2.90-2.83 (m, 1H), 2.79-2.73 (m, 0.53H), 2.70-2.64 (m, 0.62H), 2.09-2.07 (m, 1H), 1.79 (bs, 1H); MS (EI): m/z: 266 [M⁺], 131(100), 91, 77, 57, 31.

4. References


[3] There is no literature on anti- and syn-configurations of diastereomers 3a-i.
5. $^1$H NMR, $^{13}$C NMR and HR-MS Spectra of Products.

$^1$H NMR of 2-(2H-chromen-2-yl) butan-1-ol 3a (low polar)
$^1$H NMR of 2-(2H-chromen-2-yl) butan-1-ol 3a (high polar)
MS of 2-(2H-chromen-2-yl) butan-1-ol 3a
$^1$H NMR of 2-(2H-chromen-2-yl) propan-1-ol 3b (low polar)
$^1$H NMR of 2-(2H-chromen-2-yl) propan-1-ol 3b (high polar)
MS of 2-(2H-chromen-2-yl) propan-1-ol 3b
$^1$H NMR of 2-(2H-chromen-2-yl) pentan-1-ol 3c (mixture of two diastereomers)
MS of 2-(2H-chromen-2-yl) pentan-1-ol 3c
$^1$H NMR of 2-(2H-chromen-2-yl) hexan-1-ol 3d (mixture of two diastereomers)
MS of 2-(2H-chromen-2-yl) hexan-1-ol 3d
$^1$H NMR of 2-(2H-chromen-2-yl) heptan-1-ol 3e (mixture of two diastereomers)
$^{13}$C NMR of 2-(2H-chromen-2-yl) heptan-1-ol 3e (mixture of two diastereomers)
Elemental Composition Report

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
20 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
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w(14-7 112 (1.867)
TOF MS El+

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Maximin:

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$^1$H NMR of 2-(2H-chromen-2-yl) octan-1-ol 3f (mixture of two diastereomers)

$^{13}$C NMR of 2-(2H-chromen-2-yl) octan-1-ol 3f (mixture of two diastereomers)
HR-MS of 2-(2H-chromen-2-yl) octan-1-ol 3f
Elemental Composition Report

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 60.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
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TOF MS EI+

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$^{13}$C NMR of 2-(2H-chromen-2-yl) nonan-1-ol 3g (mixture of two diastereomers)
HR-MS of 2-(2H-chromen-2-yl) nonan-1-ol 3g
**Elemental Composition Report**

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
22 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100  H: 0-200  O: 0-5

\[ \text{MW (140.152, 1.594)} \]

**TOF MS EI**

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**Diagram**

A graph showing mass distribution with a peak at 274.1936 m/z.
$^{13}$C NMR of 2-(2H-chromen-2-yl)-3-methylbutan-1-ol 3h (low polar)
$^1$H NMR of 2-(2H-chromen-2-yl)-3-methylbutan-1-ol 3h (high polar)
$^{13}$C NMR of 2-(2H-chromen-2-yl)-3-methylbutan-1-ol 3h (high polar)
HR-MS of 2-(2H-chromen-2-yl)-3-methylbutan-1-ol 3h
Elemental Composition Report

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
18 formula(s) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:
C: 0-100  H: 0-200  O: 0-5

w2/14 iso-s 86 (1.433)
TOF MS El+

<table>
<thead>
<tr>
<th>Mass</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>218.1304</td>
<td>218.1307</td>
<td>-0.3</td>
<td>-1.4</td>
<td>6.0</td>
<td>5549107.5</td>
<td>C14 H18 O2</td>
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