Diastereoselective Pd-Catalyzed Conjugate Addition of Arylboronic Acids to α-Substituted Cyclic Enones

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1. General methods

The reactions were carried out in flame-dried glassware under a dry argon atmosphere. All solvents were purified and dried by using standard methods prior to use. Commercially available reagents were used without further purification. \(^1\)H NMR spectra were recorded on a NMR instrument operated at 400 MHz. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (CDCl\(_3\): \(\delta 7.26\) ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet or unresolved), coupling constants (Hz), and integration. \(^1\)C NMR spectra were recorded on a NMR instrument operated at 100 MHz with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (CDCl\(_3\): \(\delta 77.1\) ppm). Infrared spectra were recorded from thin films of pure samples. Mass and HRMS spectra were measured in EI or ESI mode and the mass analyzer type used for the HRMS was TOF. Thin layer chromatography was performed on pre-coated glassback plates and visualized with UV light at 254 nm. Flash column chromatography was performed on silica gel. Substrates \(1b, 1c, 1e, \) and \(1f\) were synthesized according to literature.\(^1\)

2. More screening of reaction conditions for Pd-catalyzed conjugate addition of phenylboronic acid (2a) to 2-methyl-cyclopentenone (1a)\(^a\)

![Chemical reaction diagram]

\[
\begin{array}{c}
\text{1a} \quad \text{2a} \\
\text{Solvent, } \text{Ar} \\
\text{T, 24 h} \\
\end{array}
\]

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<th>solvent</th>
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<th>yield(%) (^b)</th>
<th>dr (^c)</th>
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<td>L</td>
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<td>Ratio</td>
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$^a$ Molar ratio of [Pd]/L/1a/2a = 5:6:100:200.

$^b$ Isolated yield.

$^c$ Determined by GC chromatography with crude product.

### 3. General experimental procedure and characterization of products

To a flame dried 25mL Schlenk tube were added Pd(TFA)$_2$ (4.2 mg, 0.0125 mmol), ligand L2 (2.3 mg, 0.015 mmol), anhydrous DMAc (1.0 mL). The resulting mixture was allowed to stir for 60 mins. The arylboronic acids 2 (2 equiv, 0.5 mmol) and unsaturated ketone 1 (1 equiv, 0.25 mmol) were added subsequently. The resulting reaction mixture was stirred at 80 °C for 24 hours. Water (10 mL) was added and the aqueous was extracted by ethyl acetate (2 × 10 mL). The combined organic phase was washed with water (3 × 10 mL) and brine (10 mL). The organic phase was dried over anhydrous Na$_2$SO$_4$, filtered. After the volatile was removed in vacuo, the ratio of two diastereoisomers was determined by $^1$H NMR or GC chromatography. Then the resulting residue was subjected to flash chromatography on silica gel with petroleum ether and ethyl acetate as eluent to give product 3.

GC chromatography **condition A**: (SPB$^{TM}$-5 column, 30 m × 0.32 mm × 0.25 μm, carrier gas nitrogen), injector temperature 280 °C, split ratio 20, linear velocity flow control, column flow 1.0 mL/min, column temperature 80 °C (2 min), 80-200 °C (15 °C/min), 200 °C (1 min), 200-280 °C (20 °C/min), 280 °C (10 min), FID detector
temperature 280 ºC.

GC chromatography condition B: (SPB™-5 column, 30 m × 0.32 mm × 0.25 μm, carrier gas nitrogen), injector temperature 280 ºC, split ratio 20, linear velocity flow control, column flow 1.0 mL/min, column temperature 80 ºC (2 min), 80-200 ºC (2 ºC/min), 200 ºC (1 min), 200-280 ºC (20 ºC/min), 280 ºC (10 min), FID detector temperature 280 ºC.

trans-2-methyl-3-phenylcyclopentan-1-one (3a)

![trans-2-methyl-3-phenylcyclopentan-1-one (3a)](image)

Colorless oil, yield: 98%, dr: >20/1 (GC, condition A): T_R = 9.97 min (major), 10.23 min (minor).

1H NMR (400 MHz, CDCl3): δ 7.39–7.30 (m, 2H), 7.25 (dt, J = 6.2, 1.8 Hz, 3H), 2.79 (td, J = 12.0, 5.6 Hz, 1H), 2.60–2.46 (m, 1H), 2.35–2.15 (m, 3H), 2.02–1.87 (m, 1H), 1.03 (d, J = 6.9 Hz, 3H).

13C NMR (101 MHz, CDCl3): δ 219.7, 142.3, 128.7, 127.1, 126.9, 51.4, 51.0, 37.7, 29.6, 12.2.

MS (EI) m/z (rel): 174 (M+, 60), 117 (100), 91.05 (49), 77 (16).

trans-2-methyl-3-(o-tolyl)cyclopentan-1-one (3b)

![trans-2-methyl-3-(o-tolyl)cyclopentan-1-one (3b)](image)

Colorless oil, yield: 85%, dr: >20/1 (GC, condition B): T_R = 23.29 min (major), 25.34 min (minor).

1H NMR (400 MHz, CDCl3): δ 7.33–7.01 (m, 5H), 3.10 (td, J = 12.1, 5.2 Hz, 1H), 2.61–2.48 (m, 1H), 2.43–2.17 (m, 6H), 1.87–1.71 (m, 1H), 1.01 (dd, J = 6.8, 1.3 Hz, 3H).
13C NMR (101 MHz, CDCl3): δ 219.7, 140.3, 136.4, 130.6, 126.5, 126.4, 124.7, 51.0, 46.0, 37.6, 29.1, 19.8, 12.1.

IR (neat): 2962, 1738, 1492, 1459, 1406, 1260, 1147, 1090, 1021, 798, 761, 732

MS (EI) m/z (rel): 188 (M⁺, 60), 132 (90), 114 (100), 90 (50).

HRMS (EI): Calcd for C13H16O [M⁺]: 188.1201; found 188.1199.

trans-2-methyl-3-(m-tolyl)cyclopentan-1-one (3c)

Colorless oil, yield: 82%, dr: >20/1 (GC, condition B): T_R = 23.03 min (major)
24.26 min (minor).

1H NMR (400 MHz, CDCl3): δ 7.25 (t, J = 7.7 Hz, 1H), 7.13–7.01 (m, 3H), 2.78 (td, J = 12.0, 5.6 Hz, 1H), 2.60–2.49 (m, 1H), 2.36 (s, 3H), 2.33–2.19 (m, 3H), 2.02–1.89 (m, 1H), 1.04 (d, J = 6.8 Hz, 3H).

13C NMR (101 MHz, CDCl3): δ 219.8, 142.3, 138.3, 128.6, 127.9, 127.6, 124.1, 51.4, 50.9, 37.7, 29.6, 21.5, 12.2.

IR (neat): 2962, 2873, 1737, 1607, 1491, 1454, 1405, 1373, 1284, 1143, 1092, 1060, 950, 879, 823, 785, 727, 700.

MS (EI) m/z (rel): 188 (M⁺, 53), 134 (100), 101 (50), 62 (40).

HRMS (EI): Calcd for C13H16O [M⁺]: 188.1201; found 188.1205.

trans-2-methyl-3-(p-tolyl)cyclopentan-1-one (3d)

Colorless oil, yield: 84%, dr: >20/1(GC, GC condition B): T_R = 23.32 min (major),
25.37 min (minor).
\textbf{trans-3-(4-bromophenyl)-2-methylcyclopentan-1-one (3e)}

\begin{center}
\begin{tikzpicture}
\draw (0,0) circle (0.5 cm);
\draw (0,0) -- (0.5,0.5);
\draw (0,0) -- (0.5,-0.5);
\draw (0.5,-0.5) -- (0,-1);
\draw (0,0) -- (-0.5,-0.5);
\draw (-0.5,-0.5) -- (0,-1);
\draw (-0.5,-0.5) -- (-0.5,0.5);
\draw (-0.5,0.5) -- (0,1);
\draw (0,1) -- (0.5,0.5);
\draw (-0.5,0.5) -- (-0.5,-0.5);
\node at (1.2,0) {Br};
\end{tikzpicture}
\end{center}

Yellow oil, yield: 75%, dr: >20/1 (GC, condition B): $T_R = 34.26$ min (major), 35.66 min (minor).

\textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): $\delta$ 7.53–7.36 (m, 2H), 7.17–7.05 (m, 2H), 2.76 (td, $J = 12.1, 5.6$ Hz, 1H), 2.60–2.47 (m, 1H), 2.34–2.11 (m, 3H), 1.98–1.84 (m, 1H), 1.02 (d, $J = 6.9$ Hz, 3H).

\textsuperscript{13}C NMR (101 MHz, CDCl\textsubscript{3}): $\delta$ 218.9, 141.4, 131.8, 128.9, 120.5, 51.3, 50.4, 37.6, 29.5, 12.1.

IR (neat): 2963, 2874, 1736, 1490, 1454, 1405, 1373, 1276, 1163, 1143, 1059, 1027, 1008, 950, 894, 820, 731, 678.

MS (El) $m/z$ (rel): 254 (M\textsuperscript{+}, 50), 253 (10), 252 (60), 238 (90), 168 (100).

HRMS (El): Calcd for C\textsubscript{12}H\textsubscript{13}OBr [M\textsuperscript{+}]: 252.0150; found 252.0158.
Colorless oil, yield: 89%, dr: >20/1 (GC, condition B): $T_R = 27.01$ min (major), 27.59 min (minor).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.92–7.78 (m, 3H), 7.71 (d, $J = 2.0$ Hz, 1H), 7.55–7.45 (m, 2H), 7.41 (dd, $J = 8.5, 1.9$ Hz, 1H), 2.98 (td, $J = 12.0, 5.9$ Hz, 1H), 2.65–2.53 (m, 1H), 2.44–2.26 (m, 3H), 2.12–1.99 (m, 1H), 1.09 (d, $J = 6.9$ Hz, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 219.6, 139.8, 133.6, 132.6, 128.5, 127.7, 127.6, 126.3, 125.8, 125.7, 125.2, 51.4, 51.2, 37.7, 29.6, 12.3.

IR (neat): 3053, 2962, 2873, 1735, 1632, 1599, 1507, 1453, 1404, 1373, 1291, 1270, 1139, 1059, 1021, 948, 891, 856, 817, 747, 652, 629

MS (El) $m/z$ (rel): 224 (M$^+$, 60), 196 (70), 130 (100), 73 (40).

HRMS (El): Calcd for C$_{16}$H$_{16}$O [M$^+$]: 224.1201; found 224.1209.

*trans*-2-methyl-3-(4-(trifluoromethyl)phenyl)cyclopentan-1-one (3g)

![Diagram of trans-2-methyl-3-(4-(trifluoromethyl)phenyl)cyclopentan-1-one (3g)](image)

Yellow oil, yield: 50%, dr: 18/1 ($^1$H NMR).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.62 (d, $J = 8.4$ Hz, 2H), 7.42–7.35 (m, 2H), 2.88 (td, $J = 12.0, 5.5$ Hz, 1H), 2.58 (ddd, $J = 18.7, 8.3, 1.6$ Hz, 1H), 2.39–2.19 (m, 3H), 2.03–1.90 (m, 1H), 1.04 (d, $J = 6.8$ Hz, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 218.6, 146.4, 129.4 (d, $J = 31.8$ Hz), 127.5, 125.8 (q, $J = 3.7$ Hz), 125.5 (d, $J = 271.2$ Hz), 51.3, 50.8, 37.6, 29.4, 12.1.

$^{19}$F NMR (376 MHz, CDCl$_3$): $\delta$ -62.46.

IR (neat): 2967, 1740, 1618, 1456, 1422, 1323, 1161, 1117, 1066, 1016, 954, 840,
trans-3-(4-methoxyphenyl)-2-methylcyclopentan-1-one (3h)

Yellow oil, yield: 60%, dr: 15/1 (1H NMR).

\[
\begin{align*}
\text{1H NMR (400 MHz, CDCl}_3&\text{): }\delta 7.16 (dt, J = 8.3, 2.3 \text{ Hz}, 2\text{H}), 6.88 (dt, J = 8.2, 2.2 \\
&\text{Hz}, 2\text{H}), 3.79 (d, J = 1.3 \text{ Hz}, 3\text{H}), 2.73 (td, J = 12.1, 5.5 \text{ Hz}, 1\text{H}), 2.57–2.45 (m, 1\text{H}), \\
&2.32–2.11 (m, 3\text{H}), 1.96–1.80 (m, 1\text{H}), 1.01 (d, J = 6.9 \text{ Hz}, 3\text{H}).
\end{align*}
\]

\[
\begin{align*}
\text{13C NMR (101 MHz, CDCl}_3&\text{): }\delta 219.8, 158.4, 134.3, 128.0, 114.1, 55.3, 51.6, 50.2, \\
&37.7, 29.7, 12.1.
\end{align*}
\]

IR (neat): 2961, 2836, 1736, 1611, 1583, 1512, 1455, 1405, 1371, 1178, 1144, 1105, 949, 896, 828, 770, 737, 638.

MS (EI) m/z (rel): 204 (M⁺, 9), 146 (100), 120 (58), 90 (22).

HRMS (EI): Calcd for C₁₃H₁₆O₂ [M⁺]: 204.1150; found 204.1147.

trans-2-butyl-3-phenylcyclopentan-1-one (3i)

Colorless oil, yield: 56%, dr: >20/1 (GC, condition B): \(T_R = 30.74\) min (major), 32.42 min (minor).

\[
\begin{align*}
\text{1H NMR (400 MHz, CDCl}_3&\text{): }\delta 7.34 (t, J = 7.6 \text{ Hz}, 2\text{H}), 7.30–7.18 (m, 3\text{H}), 2.99 (td, \\
&J = 11.9, 11.3, 5.4 \text{ Hz}, 1\text{H}), 2.51 (dd, J = 16.8, 8.4 \text{ Hz}, 1\text{H}), 2.36–2.16 (m, 3\text{H}),
\end{align*}
\]
1.97–1.82 (m, 1H), 1.54–1.45 (m, 2H), 1.33–1.00 (m, 4H), 0.77 (t, \( J = 7.1 \) Hz, 3H).

\(^{13}\text{C} \) NMR (101 MHz, CDCl\(_3\)): \( \delta \) 219.7, 143.0, 128.7, 127.1, 126.8, 55.8, 48.5, 38.4, 30.2, 28.8, 27.6, 22.8, 13.8.

IR (neat): 3029, 2957, 2931, 2872, 1737, 1712, 1601, 1494, 1454, 1406, 1260, 1155, 1073, 1030, 798, 763, 700.

MS (EI) \( m/z \) (rel): 216 (M\(^+\), 10), 172 (30), 92 (100), 68 (80).

HRMS(EI): Calcd for C\(_{15}\)H\(_{20}\)O \([\text{M}]^{+}\): 216.1514; found 216.1519.

\textit{trans}-2,3-diphenylcyclopentan-1-one (3j)

White solid, yield: 80\%, dr: >20/1 (\textsuperscript{1}H NMR).

\( ^{1}\text{H} \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.37–7.14 (m, 8H), 7.12–7.03 (m, 2H), 3.61–3.44 (m, 2H), 2.77–2.66 (m, 1H), 2.58–2.44 (m, 2H), 2.20–2.06 (m, 1H).

MS (EI) \( m/z \) (rel): 236 (M\(^+\), 20), 235 (100), 178 (33), 117 (72), 90 (41).

\textit{trans}-2-methyl-3-phenylcyclohexan-1-one (3k)

Colorless oil, yield: 90\%, dr: 16/1 (GC, condition B): \( T_{R} = 23.49 \) min (major), 25.09 min (minor).

\( ^{1}\text{H} \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.36–7.27 (m, 2H), 7.26–7.13 (m, 3H), 2.67–2.39 (m, 4H), 2.14 (dq, \( J = 11.9, 3.3 \) Hz, 1H), 2.08–1.88 (m, 2H), 1.81–1.69 (m, 1H), 0.86–0.68 (m, 3H).

MS (EI) \( m/z \) (rel): 188 (M\(^+\), 5), 187 (34), 116 (100), 90 (28), 76 (10).

\textit{trans}-2-methyl-3-(4-(trifluoromethyl)phenyl)cyclohexan-1-one (3l)

\textit{trans}-2,3-diphenylcyclopentan-1-one (3j)

\textit{trans}-2-methyl-3-phenylcyclohexan-1-one (3k)

\textit{trans}-2-methyl-3-(4-(trifluoromethyl)phenyl)cyclohexan-1-one (3l)
Colorless oil, yield: 50%, dr: 10/1 (1H NMR).

1H NMR (400 MHz, CDCl3): major isomer: δ 7.59 (d, J = 8.3 Hz, 2H), 7.32 (d, J = 8.2 Hz, 2H), 2.70–2.58 (m, 2H), 2.57–2.42 (m, 2H), 2.17 (ddt, J = 12.2, 6.5, 3.2 Hz, 1H), 2.02–1.90 (m, 2H), 1.85–1.71 (m, 1H), 0.80 (d, J = 6.1 Hz, 3H).

13C NMR (101 MHz, CDCl3): δ 211.6, 147.8, 128.3, 127.6, 126.4, 125.6 (q, J = 3.5 Hz), 52.9, 50.2, 41.7, 34.3, 26.4, 12.2.

19F NMR (376 MHz, CDCl3): δ -62.40.

IR (neat): 2932, 1711, 1618, 1449, 1421, 1323, 1258, 1220, 1162, 1067, 1017, 932, 835, 802, 667, 625, 605.

MS (EI) m/z (rel): 256 (M+, 20), 184 (30), 130 (50), 97 (100).


trans-3-(4-methoxyphenyl)-2-methylcyclohexan-1-one (3m)

Colorless oil, yield: 49%, dr: 19/1 (GC, condition B): T_R = 36.91 min (major), 38.83 min (minor).

1H NMR (400 MHz, CDCl3): δ 7.15–7.07 (m, 2H), 6.89–6.83 (m, 2H), 3.80 (s, 3H), 2.61–2.40 (m, 4H), 2.19–2.07 (m, 1H), 1.99–1.85 (m, 2H), 1.81–1.70 (m, 1H), 0.80 (d, J = 6.2 Hz, 3H).

13C NMR (101 MHz, CDCl3): δ 212.6, 158.2, 136.1, 128.1, 113.9, 55.3, 52.4, 50.9, 41.9, 34.7, 26.5, 12.3.

IR (neat): 2932, 2865, 1705, 1610, 1584, 1511, 1445, 1375, 1328, 1302, 1285, 1243, 1178, 1133, 1107, 1076, 1033, 964, 930, 890, 827, 809, 751, 724, 700, 650.
trans-2-butyl-3-phenylcyclohexan-1-one (3n)

Colorless oil, yield: 69%, dr: >20/1 (1H NMR).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.32 (t, $J = 7.4$ Hz, 2H), 7.27–7.16 (m, 3H), 2.69 (td, $J = 11.4$, 3.9 Hz, 1H), 2.58 (ddd, $J = 11.4$, 8.3, 2.5 Hz, 1H), 2.51–2.42 (m, 2H), 2.16–2.07 (m, 1H), 2.01–1.85 (m, 2H), 1.78–1.72 (m, 1H), 1.54–1.48 (m, 1H), 1.31–1.23 (m, 1H), 1.16–0.91 (m, 4H), 0.74 (t, $J = 7.2$ Hz, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 212.3, 144.0, 128.6, 127.2, 126.6, 55.6, 51.5, 42.4, 34.8, 29.8, 26.6, 26.4, 22.8, 13.9.


MS (EI) m/z (rel): 218 (M$^+$, 10), 160 (100), 143 (60), 114 (80), 92 (63).

HRMS (EI) m/z (rel) Calcd for C$_{14}$H$_{18}$O$_2$ [M]: 218.1307; found 218.1304.

trans-2,3-diphenylcyclohexan-1-one (3o)

White solid, yield: 60%, dr: >20/1 (1H NMR).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.23–7.00 (m, 8H), 6.99–6.87 (m, 2H), 3.82 (d, $J = 12.1$ Hz, 1H), 3.24 (td, $J = 11.9$, 4.3 Hz, 1H), 2.70–2.56 (m, 2H), 2.30–2.21 (m, 1H), 2.21–2.06 (m, 2H), 2.03–1.89 (m, 1H).

MS (EI) m/z (rel): 250 (M$^+$, 56), 158 (17), 132 (53), 116 (100), 90 (27).
4. References


3i