Supporting Information
for
Rh(III)-catalyzed C–H activation/desymmetrization of diazabicycles with arenes: facile synthesis of functionalized cyclopentenes

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General Information:

Infrared spectra were obtained on a FTIR spectrometer. $^1$H NMR and $^{13}$C NMR spectra were recorded on BRUKER AVANCE III 500 or BRUKER AVANCE III 400 spectrometer. CD$_3$OD, acetone-$d_6$ and CDCl$_3$ were used as solvent. Chemical shifts were referenced relative to residual solvent signal (CD$_3$OD, $^1$H NMR: $\delta$ 3.31 ppm, $^{13}$C NMR: $\delta$ 49.0 ppm; acetone-$d_6$, $^1$H NMR: $\delta$ 2.05 ppm, $^{13}$C NMR: $\delta$ 29.84 ppm; CDCl$_3$: $^1$H NMR: $\delta$ 7.26 ppm, $^{13}$C NMR: $\delta$ 77.0 ppm). The following abbreviations are used to describe peak patterns where appropriate: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants ($J$) are reported in Hertz (Hz). HRMS were performed on Agilent Technologies 6224 TOF LC/MS apparatus (ESI). Melting points were measured with micro melting point apparatus.

The diazabicycles 1 were prepared according to the literature,$^1$ while heterocycles 2 and oximes 4 were purchased or prepared according to literature report.$^2$ [Cp*RhCl$_2$], AgOAc, anhydrous CH$_3$OH were commercial available.
Typical Procedure:

Typical procedure for desymmetrization: \([\text{Cp}^*\text{RhCl}_2]_2\) (2.5 mg, 2 mol%) and AgOAc (2.7 mg, 8 mol%) were added to a vial. MeOH (0.5 mL) was added and the solution was stirred for 10 min at room temperature. Afterwards, 2-phenylpyridine (31 mg, 0.2 mmol), diazabicycle 1a (52.8 mg, 0.22 mmol), and additional MeOH (1 mL) were added. The reaction was kept at 60 °C under air. After completion, the solution was concentrated and subject to flash column chromatography on silica gel using ethyl acetate/petroleum ether (v/v, 1:3) as eluent to give product 3aa as a colorless oil (64.8 mg, 82% yield).

Characterization of Products 3 and 5:

Diethyl 1-(2-(2-(pyridin-2-yl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (3aa). Colorless oil; \(^1\)H NMR (CD$_3$OD, 500 MHz): \(\delta = 8.63\) (s, 1H), 7.90 (t, \(J = 6.5\) Hz, 1H), 7.50 (d, \(J = 8.0\) Hz, 1H), 7.43-7.31 (m, 5H), 5.78 (m, 1H), 5.58 (s, 1H), 4.97 (s, 1H), 4.31 (s, 1H), 4.11-3.98 (m, 4H), 2.67-2.52 (m, 2H), 1.26-1.10 (m, 6H) ppm; \(^13\)C NMR (CD$_3$OD, 125 MHz): \(\delta = 161.0, 158.6, 157.5, 149.4, 141.5, 138.7, 134.6, 131.0, 130.1, 129.0, 127.6, 126.7, 123.6, 70.1, 63.4, 62.8, 50.9, 36.0, 14.9, 14.7\) ppm; HRMS (ESI) (m/z): calcd for C$_{22}$H$_{25}$N$_3$O$_4$ [M+H]$^+$ 396.1918, found 396.1933.
Diethyl 1-(2-(5-methyl-2-(pyridin-2-yl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (3ba). White solid; m.p. 94-96 °C; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta$ = 8.61 (s, 1H), 7.87 (t, $J$ = 7.5 Hz, 1H), 7.46 (d, $J$ = 7.5 Hz, 1H), 7.39 (t, $J$ = 6.0 Hz, 1H), 7.20-7.12 (m, 3H), 5.77 (s, 1H), 5.55 (s, 1H), 4.98 (s, 1H), 4.30 (s, 1H), 4.11-3.96 (m, 4H), 2.65-2.49 (m, 2H), 2.36 (s, 3H), 1.30-1.10 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 161.1, 158.5, 157.5, 149.3, 141.9, 140.0, 138.7, 138.6, 134.7, 131.1, 130.1, 129.5, 128.3, 126.7, 123.4, 70.0, 63.4, 62.8, 50.6, 36.0, 21.4, 14.9, 14.6; HRMS (ESI) ($m/z$): calcd for C$_{23}$H$_{27}$N$_3$O$_4$ [M+H]$^+$ 410.2075; found 410.2074.

Diethyl 1-(2-(5-methoxy-2-(pyridin-2-yl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (3ca). Colorless oil; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta$ = 8.61 (s, 1H), 7.87 (t, $J$ = 7.5 Hz, 1H), 7.46 (d, $J$ = 7.5 Hz, 1H), 7.37 (t, $J$ = 6.0 Hz, 1H), 7.25 (d, $J$ = 8.5 Hz, 1H), 6.89 (d, $J$ = 7.0 Hz, 2H), 5.78 (m, 1H), 5.56 (s, 1H), 5.00 (s, 1H), 4.35 (s, 1H), 4.12-3.96 (m, 4H), 3.82 (s, 3H), 2.64-2.50 (m, 2H), 1.30-1.11 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 161.7, 160.9, 158.5, 157.5, 149.3, 138.7, 134.5, 134.2, 132.6, 130.3, 126.8, 123.2, 114.2, 113.1, 69.9, 63.4, 62.8, 55.8, 50.9, 35.9, 14.9, 14.6; HRMS (ESI) ($m/z$): calcd for C$_{23}$H$_{27}$N$_3$O$_5$ [M+H]$^+$ 426.2024; found 426.2031.

Di-tert-butyl 1-(2-(5-chloro-2-(pyridin-2-yl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (3da). Colorless oil; $^1$H NMR (CD$_3$OD, 400 MHz): $\delta$ = 8.66 (s, 1H), 7.92 (s, 1H), 7.51-7.33 (m, 5H), 5.80 (s, 1H), 5.52 (s, 1H), 4.29 (s, 1H), 3.46 (s, 3H), 2.28-2.12 (m, 4H), 1.18-1.01 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 161.7, 160.9, 158.5, 157.5, 149.3, 138.7, 134.5, 134.2, 132.6, 130.3, 126.8, 123.2, 114.2, 113.1, 69.9, 63.4, 62.8, 55.8, 50.9, 35.9, 14.9, 14.6; HRMS (ESI) ($m/z$): calcd for C$_{23}$H$_{27}$N$_3$O$_4$ [M+H]$^+$ 410.2075; found 410.2074.
2.61-2.48 (m, 2H), 1.50-1.22 (m, 18H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta =$ 159.9, 157.6, 156.6, 149.6, 140.2, 138.9, 136.0, 134.0, 132.8, 131.0, 129.0, 127.6, 126.7, 123.9, 120.1, 82.4, 81.9, 70.7, 67.2, 35.7, 28.6, 28.4; HRMS (ESI) ($m/z$): calcd for C$_{26}$H$_{32}$ClN$_3$O$_4$ [M+H]$^+$ 486.2154; found 486.2156.

Diethyl 1-(2-(2-(pyrimidin-2-yl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (3ea). Colorless oil; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta =$ 8.87 (d, $J =$ 4.5 Hz, 2H), 7.63 (s, 1H), 7.47-7.33 (m, 4H), 5.80 (s, 1H), 5.57 (s, 1H), 4.59 (s, 1H), 4.15-3.93 (m, 4H), 2.71-2.53 (m, 2H), 1.30-1.07 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta =$ 168.4, 158.4, 143.5, 139.6, 134.6, 131.8, 131.0, 129.3, 127.4, 125.9, 125.2, 120.4, 69.1, 63.3, 62.8, 51.1, 36.2, 14.9, 14.6; HRMS (ESI) ($m/z$): calcd for C$_{21}$H$_{24}$N$_4$O$_4$ [M+H]$^+$ 397.1871; found 397.1858.

Diethyl 1-(2-(5-methyl-2-(pyrimidin-2-yl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (3fa). Colorless oil; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta =$ 8.85 (d, $J =$ 4.5 Hz, 2H), 7.56-7.41 (m, 2H), 7.20-7.16 (m, 2H), 5.81 (d, $J =$ 3.5 Hz, 1H), 5.55 (s, 1H), 5.05 (s, 1H), 4.60 (s, 1H), 4.14-3.93 (m, 4H), 2.71-2.51 (m, 2H), 2.38 (s, 3H), 1.34-1.27 (m, 6H); $^{13}$C NMR (CD$_3$OD, 125 MHz): $\delta =$ 168.5, 158.4, 143.4, 141.2, 139.9, 136.8, 134.8, 132.0, 130.2, 129.9, 128.3, 125.9, 125.2, 120.2, 69.4, 63.3, 62.8, 50.8, 35.9, 21.4, 14.9, 14.5; HRMS (ESI) ($m/z$): calcd for C$_{22}$H$_{26}$N$_4$O$_4$ [M+H]$^+$ 411.2027; found 411.2044.
Di-tert-butyl 1-(2-(5-methyl-2-(pyrimidin-2-yl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (3fb). Colorless oil; $^1$H NMR (acetone-$d_6$, 400 MHz): $\delta =$ 8.92 (s, 2H), 8.26-8.06 (m, 1H), 7.73 (s, 1H), 7.47 (s, 1H), 7.26-7.17 (m, 2H), 5.79 (d, $J =$ 2.8 Hz, 1H), 5.51 (s, 1H), 5.10-5.01 (m, 1H), 4.77 (s, 1H), 2.60 (s, 2H), 2.39 (s, 2H), 1.47-1.10 (m, 18H); $^{13}$C NMR (acetone-$d_6$, 100 MHz): $\delta =$ 167.7, 158.0, 156.2, 155.6, 143.2, 140.4, 136.9, 135.0, 132.3, 129.9, 127.9, 119.9, 80.6, 80.3, 67.8, 48.9, 30.5, 28.5, 21.4; HRMS (ESI) (m/z): calcd for C$_{26}$H$_{34}$N$_4$O$_4$ [M+H]$^+$ 467.2653; found 467.2666.

Diethyl 1-(2-(5-methoxy-2-(pyrimidin-2-yl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (3ga). Colorless oil; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta =$ 8.84 (d, $J = 5.0$ Hz, 2H), 7.68 (s, 1H), 7.38 (t, $J = 5.0$ Hz, 1H), 6.93-6.90 (m, 2H), 5.83 (d, $J = 3.5$ Hz, 1H), 5.57 (s, 1H), 5.04 (s, 1H), 4.74 (s, 1H), 4.14-3.91 (m, 4H), 3.84 (s, 3H), 2.71-2.52 (m, 2H), 1.34-1.27 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta =$ 168.1, 162.5, 158.7, 158.3, 157.8, 145.5, 134.6, 133.9, 132.0, 130.5, 125.2, 119.9, 114.6, 113.0, 69.3, 63.4, 62.8, 55.8, 51.1, 36.1, 14.9, 14.6; HRMS (ESI) (m/z): calcd for C$_{22}$H$_{26}$N$_4$O$_5$ [M+H]$^+$ 427.1976; found 427.1993.
Diethyl 1-(2-(2-(1H-pyrazol-1-yl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (3ha). White solid; m.p. 94-96 °C; $^1$H NMR (acetone-$d_6$, 400 MHz): δ = 7.87 (s, 1H), 7.74 (s, 1H), 7.49 (m, 2H), 7.37 (m, 1H), 7.28 (d, $J = 8.0$ Hz, 1H), 6.52 (s, 1H), 5.81 (s, 1H), 5.49 (m, 1H), 4.97 (m, 1H), 4.14-3.95 (m, 5H), 2.59 (br, 2H), 1.24-0.94 (m, 6H); $^{13}$C NMR (acetone-$d_6$, 100 MHz): δ = 157.0, 156.3, 141.1, 140.7, 140.2, 133.6, 132.9, 130.7, 129.9, 129.7, 128.1, 127.3, 107.5, 68.8, 62.3, 61.9, 47.3, 35.8, 14.8, 14.7; HRMS (ESI) (m/z): calcd for C$_{20}$H$_{24}$N$_4$O$_4$ [M+H]$^+$ 385.1871; found 385.1856.

Di-$ tert$-butyl 1-(2-(2-(1H-pyrazol-1-yl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (3hb). Colorless oil; $^1$H NMR (CD$_3$OD, 500 MHz): δ = 7.82 (s, 1H), 7.74 (s, 1H), 7.47 (t, $J = 4.0$ Hz, 2H), 7.37 (s, 1H), 7.28 (d, $J = 7.5$ Hz, 1H), 6.54 (s, 1H), 5.81 (s, 1H), 5.51(s, 1H), 4.93 (s, 1H), 3.98 (s, 1H), 2.62-2.48(m, 2H), 1.47-1.39(m, 18H); $^{13}$C NMR (CD$_3$OD, 100 MHz): δ = 157.7, 156.5, 152.6, 141.4, 140.8, 133.6, 131.0, 130.6, 130.1, 129.8, 128.4, 127.8, 107.8, 84.6, 82.1, 70.7, 67.2, 35.9, 28.6, 28.4; HRMS (ESI) (m/z): calcd for C$_{24}$H$_{32}$N$_4$O$_4$ [M+H]$^+$ 441.2497; found 441.2484.
Diethyl 1-(2-(2-(1-methyl-1H-imidazol-2-yl)phenyl)cyclopent-3-en-1-yl)hydrazine 1,2-dicarboxylate (3ia). Colorless oil; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta$= 7.50-7.27 (m, 4H), 7.20-7.06 (m, 2H), 5.78 (s, 1H), 5.58 (s, 1H), 4.11-3.98 (m, 5H), 3.50 (s, 3H), 2.62-2.48 (m, 2H), 1.31-1.15 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$= 158.6, 157.4, 148.3, 144.9, 133.9, 131.6, 131.2, 130.9, 130.5, 129.3, 127.7, 127.6, 123.0, 69.9, 63.4, 62.7, 50.9, 35.7, 34.1, 14.9, 14.7; HRMS (ESI) (m/z): calcd for C$_{21}$H$_{26}$N$_4$O$_4$ [M+H]$^+$ 399.2027; found 399.2024.

Diethyl 1-(2-(2-(1-(hydroxyimino)ethyl)phenyl)cyclopent-3-en-1-yl)hydrazine 1,2-dicarboxylate (5aa). Colorless oil; $^1$H NMR (CD$_3$OD, 400 MHz): $\delta$= 7.36-7.20 (m, 4H), 5.85 (s, 1H), 5.62 (s, 1H), 4.31 (s, 1H), 4.11 (m, 4H), 2.73-2.59 (m, 2H), 2.20 (s, 3H), 1.30 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100MHz): $\delta$= 159.0, 158.8, 157.6, 142.7, 139.2, 134.5, 130.4, 129.8, 129.4, 128.9, 127.4, 69.1, 63.4, 62.9, 51.7, 36.4, 17.1, 14.9, 14.8; HRMS (ESI) (m/z): calcd for C$_{19}$H$_{25}$N$_3$O$_5$ [M+H]$^+$ 376.1867; found 376.1869.

Di-tert-butyl 1-(2-(2-(1-(hydroxyimino)ethyl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5ab). White solid; m.p. 59-61 °C; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta$= 7.33-7.19 (m, 4H), 5.83 (m, 1H), 5.57 (br, 1H), 4.24 (br, 1H), 2.66-2.52 (m, 2H), 2.18 (s, 3H), 1.50-1.29 (m, 18H); $^{13}$C NMR (CD$_3$OD, 100MHz): $\delta$= 158.6, 157.8, 156.6, 152.6, 143.0, 139.3, 134.4, 130.5, 129.8, 129.2, 127.4, 82.2, 70.3, 51.6, 36.2,
28.6, 28.4, 17.2; HRMS (ESI) (m/z): calcd for C_{23}H_{33}N_{3}O_{5} [M+H]^+ 432.2493; found 432.2478.

Diethyl 1-(2-(2-(1-(hydroxyimino)ethyl)-5-methylphenyl)cyclopent-3-en-1-yl)-hydrazine-1,2-dicarboxylate (5ba). Colorless oil; \( ^1H \) NMR (CD_{3}OD, 500 MHz): \( \delta = 7.06 \) (m, 3H), 5.82 (m, 1H), 5.58 (br, 1H), 4.28-4.01 (m, 5H), 2.70-2.50 (m, 2H), 2.31 (s, 3H), 2.16 (s, 3H), 1.34-1.18 (m, 6H); \( ^{13}C \) NMR (CD_{3}OD, 100 MHz): \( \delta = 159.0, 157.6, 142.5, 139.7, 136.3, 134.6, 130.3, 129.3, 128.2, 69.1, 63.4, 62.9, 51.5, 36.3, 21.4, 17.1, 14.8, 14.6; \) HRMS (ESI) (m/z): calcd for C_{20}H_{27}N_{3}O_{5} [M+H]^+ 390.2024; found 390.2033.

Di-tert-butyl 1-(2-(2-(1-(hydroxyimino)ethyl)-5-methylphenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5bb). White solid; m.p. 58-60 °C; \( ^1H \) NMR (CD_{3}OD, 500 MHz): \( \delta = 7.07 \) (m, 3H), 5.82 (br, 1H), 5.56 (br, 1H), 4.24 (br, 1H), 2.65-2.49 (m, 2H), 2.32 (s, 3H), 2.16 (s, 3H), 1.52-1.12 (m, 18H); \( ^{13}C \) NMR (CD_{3}OD, 100MHz): \( \delta = 158.6, 158.0, 156.7, 142.7, 139.6, 136.5, 134.6, 130.3, 129.6, 129.2, 128.1, 82.2, 70.4, 36.2, 28.6, 21.4, 17.2; \) HRMS (ESI) (m/z): calcd for C_{24}H_{35}N_{3}O_{5} [M+H]^+ 446.2650; found 446.2655.
**Diethyl 1-(2-(4-(1-(hydroxyimino)ethyl)-[1,1′-biphenyl]-3-yl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5ca).** White solid; m.p.147-149 °C; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta$ = 7.59 (d, $J$ = 7.5 Hz, 2H), 7.50 (d, $J$ = 6.5 Hz, 2H), 7.43 (t, $J$ = 7.5Hz, 2H), 7.35-7.28 (m, 2H), 5.87 (s, 1H), 5.66 (s, 1H), 4.97 (br, 1H), 4.39 (br, 1H), 4.18-4.02 (m, 4H), 2.73-2.59 (m, 2H), 2.22 (s, 3H), 1.30-1.27 (m, 6H); $^{13}$C NMR (CD$_3$OD, 125 MHz): $\delta$ = 159.0, 158.5, 157.7, 143.1, 142.8, 141.9, 138.2, 134.4, 130.6, 130.1, 129.9, 128.6, 128.0, 127.5, 126.1, 69.0, 63.5, 62.9, 51.7, 36.2, 16.8, 14.8, 14.6; HRMS (ESI) ($m/z$): calcd for C$_{25}$H$_{29}$N$_3$O$_5$ [M+H]$^+$ : 452.2180, found: 452.2173.

**Di-tert-butyl 1-(2-(5-hydroxy-2-(1-(hydroxyimino)ethyl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5da).** White solid; m.p.127-129 °C; $^1$H NMR (CD$_3$OD, 400 MHz): $\delta$ = 7.03 (d, $J$ = 8.4 Hz, 1H), 6.88-6.33 (m, 2H), 5.81 (br, 1H), 5.55 (br, 1H), 4.25 (br, 1H), 2.62-2.48 (m, 2H), 2.15 (s, 3H), 1.48-1.29 (m, 18H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 159.0, 157.9, 156.5, 144.5, 134.6, 130.6, 130.3, 115.4, 114.6, 113.4, 82.3, 70.4, 51.5, 36.1, 28.5, 17.3; HRMS (ESI) ($m/z$): calcd for C$_{23}$H$_{33}$N$_3$O$_6$ [M+H]$^+$ 448.2442; found 448.2437.
Diethyl 1-(2-(1-(hydroxyimino)ethyl)-5-methoxyphenyl)cyclopent-3-en-1-yl)-
hydrazine-1,2-dicarboxylate (5ea). Colorless oil; $^1$H NMR (CD$_3$OD, 400 MHz): $\delta$ = 7.13 (d, $J$ = 8.4 Hz, 1H), 6.80 (m, 2H), 5.83 (d, $J$ = 3.2 Hz, 1H), 5.60 (br, 1H), 4.33 (s, 1H), 4.17 (m, 4H), 3.77 (s, 3H), 2.69-2.55 (m, 2H), 2.16 (s, 3H), 1.30-1.25 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 161.3, 159.0, 158.7, 157.6, 144.3, 134.3, 131.7, 130.8, 130.5, 114.1, 112.9, 69.0, 63.7, 62.9, 55.7, 51.6, 36.2, 17.2, 14.8, 14.6; HRMS (ESI) (m/z): calcd for C$_{20}$H$_{27}$N$_3$O$_6$ [M+H]$^+$ 406.1973; found 406.1972.

Diethyl 1-(2-(5-chloro-2-(1-(hydroxyimino)ethyl)phenyl)cyclopent-3-en-1-yl)-
hydrazine-1,2-dicarboxylate (5fa). Yellow solid; m.p. 119-120 °C; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta$ = 7.25-7.18 (m, 3H), 5.88 (s, 1H), 5.58 (s, 1H), 4.84 (m, 1H), 4.32 (s, 1H), 4.16 (m, 4H), 2.71-2.57 (m, 2H), 2.16 (s, 3H), 1.34-1.17 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 159.0, 157.6, 145.2, 137.9, 135.5, 133.7, 131.1, 128.9, 127.6, 68.1, 63.5, 62.9, 51.9, 36.4, 16.8, 14.7, 14.6; HRMS (ESI) (m/z): calcd for C$_{19}$H$_{24}$ClN$_3$O$_5$ [M+H]$^+$ 410.1477; found 410.1487.

Di-tert-butyl 1-(2-(5-chloro-2-(1-(hydroxyimino)ethyl)phenyl)cyclopent-3-en-1-yl)
hydrazine-1,2-dicarboxylate (5fb). White solid; m.p. 143-145 °C; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta$ = 7.25-7.20 (m, 3H), 5.89 (m, 1H), 5.56 (br, 1H), 4.26 (br, 1H), 2.66-2.56 (m, 2H), 2.17 (s, 3H), 1.50-1.29 (m, 18H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 157.9, 156.6, 152.6, 145.3, 138.1, 135.5, 133.7, 131.1, 129.2, 127.5, 82.4, 70.0,
66.4, 51.7, 36.1, 28.6, 28.4, 16.8; HRMS (ESI) (m/z): calcd for C_{23}H_{32}ClN_{3}O_{5} [M+H]^+ 466.2103; found 466.2127.

**Di-tert-butyl 1-(2-(5-bromo-2-(1-(hydroxyimino)ethyl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5ga).** White solid; m.p.161-163 °C; \(^1\)H NMR (CD\(_3\)OD, 500 MHz): \(\delta = 7.40\) (d, \(J = 6.0\) Hz, 2H), 7.13 (d, \(J = 5.5\) Hz, 1H), 5.86 (m, 1H), 5.56 (br, 1H), 4.22 (br, 1H), 2.64-2.55 (m, 2H), 2.17 (s, 3H), 1.48-1.17 (m, 18H); \(^{13}\)C NMR (CD\(_3\)OD, 100 MHz): \(\delta = 157.9, 156.5, 145.7, 138.5, 133.7, 132.1, 131.3, 130.5, 123.7, 82.5, 82.0, 70.1, 51.7, 36.1, 28.6, 28.4, 16.9\); HRMS (ESI) (m/z): calcd for C\(_{23}\)H\(_{32}\)BrN\(_{3}\)O\(_{5}\) [M+H]^+ 510.1598; found 510.1601.

**Di-tert-butyl 1-(2-(2-(1-(hydroxyimino)ethyl)-5-nitrophenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5ha).** Yellow solid; m.p.177-179 °C; \(^1\)H NMR (CD\(_3\)OD, 400 MHz): \(\delta = 8.120\) (m, 2H), 7.47 (m, 1H), 5.92 (m, 1H), 5.62 (br, 1H), 4.40 (br, 1H), 2.72-2.57 (m, 2H), 2.22 (s, 3H), 1.48-1.29 (m, 18H); \(^{13}\)C NMR (CD\(_3\)OD, 100MHz): \(\delta = 156.6, 155.6, 154.4, 148.0, 144.7, 143.5, 131.8, 131.6, 129.1, 123.2, 122.8, 121.1, 81.6, 81.1, 69.3, 50.9, 35.6, 28.1, 16.5, 16.2\); HRMS (ESI) (m/z): calcd for C\(_{23}\)H\(_{32}\)N\(_{4}\)O\(_{7}\) [M+H]^+ 477.2344; found 477.2351.

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Di-tert-butyl 1-(2-(5-acetamido-2-(1-(hydroxyimino)ethyl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5ia). White solid; m.p.139-141 °C; $^1$H NMR (acetone-$d_6$, 400 MHz): $\delta = 9.24$ (s, 1H), 8.01 (s, 1H), 7.66 (s, 1H), 7.41-7.35 (m, 1H), 7.16 (br, 1H), 5.81 (br, s, 1H), 5.57 (br, s, 1H), 4.78 (br, 1H), 4.49-4.36 (m, 1H), 2.58 (br, 2H), 2.17 (s, 3H), 2.08 (s, 3H), 1.44-1.29 (m, 18H); $^{13}$C NMR (CDCl$_3$, 125 MHz): $\delta = 169.0, 156.6, 156.4, 156.0, 139.0, 132.8, 130.1, 128.7, 118.6, 117.8, 81.9, 81.0, 28.2, 24.3, 16.8, 16.2$; HRMS (ESI) ($m/z$): calcd for C$_{25}$H$_{36}$N$_4$O$_6$ [M+H]$^+$ 489.2708; found 489.2704.

Di-tert-butyl 1-(2-(2-(1-(hydroxyimino)ethyl)-4-nitrophenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5ja). Colorless oil; $^1$H NMR (CD$_3$OD, 400 MHz): $\delta = 8.17$ (d, $J = 8.0$ Hz, 1H), 8.07 (s, 1H), 7.52 (br, 1H), 5.90 (m, 1H), 5.59 (br, s, 1H), 4.38 (m, 1H), 2.74-2.56 (m, 2H), 2.24 (s, 3H), 1.48-1.28 (m, 18H); $^{13}$C NMR (CD$_3$OD, 100MHz): $\delta = 158.0, 156.5, 151.4, 147.6, 140.6, 133.2, 131.8, 130.6, 124.4, 124.3, 82.3, 66.6, 52.6, 36.0, 28.2, 16.2$; HRMS (ESI) ($m/z$): calcd for C$_{23}$H$_{32}$N$_4$O$_7$ [M+H]$^+$ 477.2344; found 477.2339.
Diethyl 1-(2-(3-bromo-2-(1-(hydroxyimino)ethyl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5ka). White solid; m.p. 146-147 °C; \(^1\)H NMR (CD\(_3\)OD, 500 MHz): \(\delta = 7.49\) (d, \(J = 5.0\) Hz, 1H), 7.25 (m, 2H), 5.84 (m, 1H), 5.56 (m, 1H), 4.96 (s, 1H), 4.16-4.04 (m, 5H), 2.80-2.60 (m, 2H), 2.13 (s, 3H), 1.34-1.17 (m, 6H); \(^{13}\)C NMR (CD\(_3\)OD, 100 MHz): \(\delta = 159.0, 158.1, 157.4, 146.2, 139.6, 139.3, 133.5, 131.8, 131.2, 127.9, 124.1, 66.9, 63.5, 62.8, 54.8, 36.7, 16.8, 14.8, 14.7; HRMS (ESI) (m/z): calcd for C\(_{19}\)H\(_{24}\)BrN\(_3\)O\(_5\) [M+H]\(^+\) 454.0972; found 454.0943.

Diethyl 1-(2-(2-((hydroxyimino)(phenyl)methyl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5la). White solid; m.p. 70-72 °C; \(^1\)H NMR (CD\(_3\)OD, 400 MHz): \(\delta = 7.56\) (d, \(J = 3.6\) Hz, 2H), 7.40-7.30 (m, 5H), 7.24 (t, \(J = 7.6\) Hz, 1H), 7.17 (s, 1H), 5.73 (br, 1H), 5.40 (br, 1H), 4.25 (br, 1H), 4.13 (m, 4H), 2.69-2.53 (m, 2H), 1.34-1.03 (m, 6H); \(^{13}\)C NMR (CD\(_3\)OD, 100MHz): \(\delta = 158.9, 157.7, 157.4, 144.0, 138.3, 135.3, 134.4, 131.5, 131.3, 130.5, 130.3, 130.2, 129.7, 129.1, 128.9, 127.3, 69.1, 63.5, 63.0, 51.5, 36.5, 14.8, 14.6; HRMS (ESI) (m/z): calcd for C\(_{24}\)H\(_{27}\)N\(_3\)O\(_5\) [M+H]\(^+\) : 438.2024; found 438.2023.

Di-tert-butyl 1-(2-(2-((hydroxyimino)(phenyl)methyl)phenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5lb). White solid; m.p. 80-82 °C; \(^1\)H NMR
(CD$_3$OD, 400 MHz): δ= 7.88 (s, 0.2 H, NH), 7.57 (br, 2H), 7.43-7.36 (m, 5H), 7.26-7.14 (m, 2H), 5.73(br, 1H), 5.39 (m, 1H), 4.89 (m, 1H), 4.16 (br, 1H), 2.60-2.50 (m, 2H), 1.54-1.24 (m, 18H); $^{13}$C NMR (CD$_3$OD, 100MHz): δ= 157.6, 156.5, 152.6, 144.0, 138.4, 135.3, 134.4, 131.4, 131.2, 130.3, 130.2, 129.6, 129.3, 129.0, 127.3, 82.1, 70.1, 66.8, 51.2, 36.3, 28.6, 28.4; HRMS (ESI) (m/z): calcd for C$_{28}$H$_{35}$N$_3$O$_5$ [M+H]$^+$ 494.2650; found 494.2634.

Diethyl 1-(2-(2-(1-(hydroxyimino)propyl)phenyl)cyclopent-3-en-1-yl) hydrazine-1,2-dicarboxylate (5ma). White solid; m.p. 87-89 °C; $^1$H NMR (CD$_3$OD, 500 MHz): δ= 7.33 (t, $J$=7.5 Hz, 1H), 7.23 (m, 2H), 7.14 (d, $J$=7.5Hz, 1H), 5.82 (d, $J$=3.5 Hz, 1H), 5.57 (m, 1H), 4.93 (m, 1H), 4.15-3.99 (m, 5H), 2.76-2.62 (m, 4H), 1.34-1.21 (m, 6H), 0.98 (t, $J$= 7.5Hz, 3H); $^{13}$C NMR (CD$_3$OD, 100 MHz): δ= 163.6, 159.0, 157.6, 143.2, 137.7, 134.5, 130.4, 129.9, 129.6, 128.8, 127.3, 69.5, 63.4, 62.9, 51.9, 36.4, 24.3, 14.8, 14.6, 9.8; HRMS (ESI) (m/z): calcd for C$_{20}$H$_{27}$N$_3$O$_5$ [M+H]$^+$ 390.2024; found 390.2012.

Diethyl 1-(2-(2-(1-(hydroxyimino)-2-phenylethyl)phenyl)cyclopent-3-en-1-yl)-hydrazine-1,2-dicarboxylate (5na). Colorless oil; $^1$H NMR (CD$_3$OD, 400 MHz): δ= 7.28 (t, $J$= 7.2 Hz, 1H), 7.16-7.00 (m, 8H), 5.70 (s, 1H), 5.11 (m, 1H), 4.42 (d, $J$= 14.0
Hz, 1H), 4.18-3.70 (m, 6H), 2.65-2.54 (m, 2H), 1.40-1.14 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta =$ 160.2, 159.0, 157.9, 143.5, 137.6, 134.7, 130.7, 129.9, 129.4, 128.8, 127.5, 127.1, 69.8, 63.5, 63.1, 51.3, 37.3, 36.1, 30.8, 15.0, 14.6; HRMS (ESI) ($m/z$): calcd for C$_{25}$H$_{29}$N$_3$O$_5$ [M+H]$^+$ 452.2180; found 452.2181.

Diethyl 1-(2-(8-(hydroxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5oa). White solid; m.p. 144-146 °C; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta =$ 7.17 (t, $J = 7.0$ Hz, 2H), 7.03 (d, $J = 6.5$ Hz, 1H), 5.80 (s, 1H), 5.59 (br, 1H), 5.11(br, 1H), 4.82 (m, 1H), 4.18-3.91 (m, 4H), 2.79-2.53 (m, 6H), 1.80-1.66 (m, 2H), 1.34-1.02 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta =$ 159.1, 157.7, 143.6, 143.3, 135.1, 131.5, 129.5, 127.7, 126.9, 69.2, 63.4, 63.0, 50.3, 36.2, 32.2, 26.2, 22.5, 14.8, 14.5; HRMS (ESI) ($m/z$): calcd for C$_{21}$H$_{27}$N$_3$O$_5$ [M+H]$^+$ 402.2024; found 402.2029.

Diethyl 1-(2-(4-(hydroxyimino)chroman-5-yl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5pa). White solid; m.p. 151-152 °C; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta =$ 7.16 (s, 1H), 6.89 (s, 1H), 6.75 (d, $J = 6.0$ Hz, 1H), 5.81 (s, 1H), 5.58 (s, 1H), 5.39 (br, 1H), 4.19-4.00 (m, 6H), 3.10-2.98 (m, 2H), 2.69-2.54 (m, 2H), 1.31-1.19 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta =$ 159.4, 158.0, 152.9, 145.2, 134.7, 130.8, 129.9, 122.7, 119.8, 117.1, 66.1, 63.4, 63.0, 50.8, 36.2, 26.3, 14.8, 14.5; HRMS (ESI) ($m/z$): calcd for C$_{20}$H$_{25}$N$_3$O$_6$ [M+H]$^+$ 404.1816; found 404.1829.
Diethyl 1-(2-(2-(1-(hydroxyimino)ethyl)furan-3-yl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5qa). Colorless oil; $^1$H NMR (CD$_3$OD, 400 MHz): $\delta$ = 7.47 (m, 1H), 6.39 (s, 1H), 5.81 (m, 1H), 5.58 (m, 1H), 4.82 (m, 1H), 4.19-4.00 (m, 4H), 2.57-2.44 (m, 2H), 2.16 (s, 3H), 1.34-0.99 (m, 6H); $^{13}$C NMR (CD$_3$OD, 125 MHz): $\delta$ = 160.5, 157.9, 143.5, 140.2, 135.4, 133.3, 130.8, 128.3, 112.5, 66.9, 63.7, 63.4, 63.0, 48.9, 35.5, 14.8, 14.7; HRMS (ESI) (m/z): calcd for C$_{17}$H$_{23}$N$_3$O$_6$ [M+H]$^+$ 366.1660; found 366.1643.

Diethyl 1-(2-(2-(1-(hydroxyimino)ethyl)thiophen-3-yl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (5ra). Colorless oil; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta$ = 7.34 (m, 1H), 6.93 (m, 1H), 5.78 (m, 1H), 5.58 (d, $J$ = 4.0 Hz, 1H), 4.86 (m, 1H), 4.59 (s, 1H), 4.17-3.98 (m, 4H), 2.64-2.48 (m, 2H), 2.23 (s, 3H), 1.38-1.08 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 159.1, 157.8, 152.6, 142.9, 136.4, 134.0, 130.2, 129.3, 126.0, 67.3, 63.5, 63.0, 35.8, 16.3, 14.7, 14.6; HRMS (ESI) (m/z): calcd for C$_{17}$H$_{23}$N$_3$O$_5$S [M+H]$^+$ 382.1431; found 382.1437.
Di tert-butyl 1-(2-(2-(1-(hydroxyimino)ethyl)thiophen-3-yl)cyclopent-3-en-1-yl)-hydrazine-1,2-dicarboxylate (5rb). Colorless oil; $^1$H NMR (CD$_3$OD, 400 MHz): $\delta$ = 7.37 (m, 1H), 6.93 (m, 1H), 5.81 (m, 1H), 5.58 (br, 1H), 4.57 (m, 1H), 2.62-2.50 (m, 2H), 2.24 (s, 3H), 1.49-1.18 (m, 18H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 158.1, 156.8, 152.5, 143.3, 134.0, 130.4, 129.5, 126.5, 126.0, 82.3, 69.0, 47.2, 35.8, 28.6, 28.4, 16.4; HRMS (ESI) (m/z): calcd for C$_{21}$H$_{31}$N$_3$O$_5$S [M+H]$^+$ 438.2057; found 438.2052.

Derivation of Product 5fa to 6 and 7:

To a vial was added 5fa (82 mg, 0.2 mmol) and NaHCO$_3$ (34 mg, 0.4 mmol), DCM (2 mL) was then added. The solution was cooled to 0 °C and m-CPBA (81 mg, 85% purity, 0.4 mmol) was added in one portion. Afterwards, the reaction solution was slowly warmed to 15 °C and kept for 24 hours. After that, saturated Na$_2$SO$_3$ solution (2 mL) was added and the mixture was stirred for 15 min. The solution was diluted with DCM (10 mL) and water (5 mL) and then subject to separatory funnel. The aqueous layer was extracted with DCM for three times and the organic layer was combined, washed with saturated Na$_2$SO$_3$ solution, NaHCO$_3$ solution, brine, dried over anhydrous MgSO$_4$, concentrated under vacuum and subject to flash chromatography on silica gel using ethyl acetate/petroleum ether (v/v, 1:3) as eluent to give product 6 as colorless oil (72 mg, 84.5% yield).

To a vial was added 5fa (82 mg, 0.2 mmol) and NaHCO$_3$ (34 mg, 0.4 mmol), DCM (2 mL) was then added. The solution was cooled to 0 °C and m-CPBA (81 mg, 85% purity, 0.4 mmol) was added in one portion. Afterwards, the reaction solution was slowly warmed to 15 °C and kept for 24 hours. After that, saturated Na$_2$SO$_3$ solution (2 mL) was added and the mixture was stirred for 15 min. The solution was diluted with DCM (10 mL) and water (5 mL) and then subject to separatory funnel. The aqueous layer was extracted with DCM for three times and the organic layer was combined, washed with saturated Na$_2$SO$_3$ solution, NaHCO$_3$ solution, brine, dried over anhydrous MgSO$_4$, concentrated under vacuum and subject to flash chromatography on silica gel using ethyl acetate/petroleum ether (v/v, 1:3) as eluent to give product 6 as colorless oil (72 mg, 84.5% yield).
Diethyl 1-(2-(5-chloro-2-(1-(hydroxyimino)ethyl)phenyl)-6-oxabicyclo[3.1.0]-hexan-3-yl)hydrazine-1,2-dicarboxylate (6). Colorless oil; $^1$H NMR (CD$_3$OD, 400 MHz): $\delta$ = 7.64 (s, 1H), 7.28 (m, 2H), 4.58 (m, 1H), 4.15-3.96 (m, 4H), 3.65 (m, 2H), 3.31 (m, 1H), 2.39 (q, $J$ = 7.6 Hz, 1H), 2.22 (s, 3H), 1.96 (br, 1H), 1.45-1.11 (m, 6H); $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 158.9, 157.4, 140.4, 138.9, 135.1, 131.0, 129.4, 127.9, 63.6, 63.1, 60.6, 54.9, 44.6, 14.8, 14.6; HRMS (ESI) ($m/z$): calcd for C$_{19}$H$_{24}$ClN$_3$O$_6$ [M+H]$^+$ 426.1427; found 426.1420.

$5fa$ (62 mg, 0.15 mmol), TsCl (2 mg) and AlCl$_3$ (1 mg) were added to Schlenk tube, vacuumed and refilled with argon for three times. Anhydrous CH$_3$CN (4 mL) was added and the solution was heated to reflux and kept for 1 hour. Then it was transferred to a round bottom flask. Silica was added to the flask and volatiles were evaporated under vacuum. The purification was performed by flash column chromatography on silica gel using ethyl acetate/petroleum ether (v/v, 1:1) as eluent to give product 7 as colorless oil (46 mg, 74% yield).

Diethyl 1-(2-(2-acetamido-5-chlorophenyl)cyclopent-3-en-1-yl)hydrazine-1,2-dicarboxylate (7).
**dicarboxylate (7).** Colorless oil; $^1$H NMR (CD$_3$OD, 500 MHz): $\delta$ = 7.71 (br, 1H), 7.47-7.45 (m, 0.25H), 7.23-7.14 (m, 2H), 5.98 (s, 1H), 5.58 (s, 1H), 4.72 (s, 1H), 4.26-3.94 (m, 5H), 2.74-2.54 (m, 2H), 2.19 (s, 3H), 1.34-1.29 (m, 6H), $^{13}$C NMR (CD$_3$OD, 100 MHz): $\delta$ = 172.5, 160.1, 157.5, 149.0, 139.3, 135.5, 132.6, 131.8, 129.5, 128.4, 127.8, 66.5, 63.8, 63.3, 50.5, 36.2, 23.6, 14.8, 14.6; HRMS (ESI) ($m/z$): calcd for C$_{19}$H$_{24}$ClN$_3$O$_5$ [M+H]$^+$ 410.1477; found 410.1460.

**Control Experiments:**

![Diagram of Reaction](image)

[Cp*RhCl$_2$]$_2$ (1.2 mg, 2 mol%), AgOAc (1.4 mg, 8 mol%) were added to a vial and MeOH (0.2 mL) was then added. The solution was stirred for 10 min, afterwards, diazabicycle 1b (29.6 mg, 0.1 mmol) was added and the solution was kept at 60 °C for 8h. TLC analysis showed it was recovered and no new product was observed.

![Diagram of Reaction](image)

[Cp*RhCl$_2$]$_2$ (6.2 mg, 0.01 mmol), AgOAc (6.7 mg, 0.04 mmol) and 4a (2.7 mg, 0.02 mmol) were added to a NMR tube and CD$_3$OD (0.5 mL) was then added. The NMR tube was kept at 60 °C in oil bath for 1h. $^1$H NMR analysis showed 4a was consumed and the intermediate 4a-I was formed.

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To the NMR tube was then added diazabicycle 1b (6 mg, 0.02 mmol) and it was kept at 60 °C in oil bath for 1h. 1H NMR analysis showed 4a-I was consumed and the product 5ab was formed.
Kinetic Isotope Effect Study:

[D₄]-5aa was prepared according to general procedure in 87% yield.

White solid; m.p. 132-134 °C; ¹H NMR (CD₃OD, 500 MHz): δ = 5.83 (s, 1H), 5.60 (s, 1H), 4.29-4.01 (m, 5H), 2.71-2.52 (m, 2H), 2.18 (s, 3H), 1.31-1.26 (m, 6H); ¹³C NMR (CD₃OD, 100 MHz): δ = 159.1, 158.9, 157.6, 142.6, 139.1, 134.5, 130.4, 129.1, 127.1, 125.2, 68.9, 63.5, 62.9, 51.7, 36.4, 17.1, 14.8, 14.6; HRMS (ESI) (m/z): calcd for C₁₉H₂₁D₄N₃O₅ [M+H]+ 380.2114; found 380.2123.

Following general procedure, [Cp*RhCl₂]₂ (1.2 mg, 2 mol%), AgOAc (1.3 mg, 8 mmol%) were added to a vial. MeOH (0.2 mL) was added and the solution was stirred at RT for 10 min. Afterwards, diazabicycle 1a (24 mg, 0.1 mmol) and oxime 4a (13.5 mg, 0.1 mmol) was added, followed by addition of MeOH (0.6 mL). In another reaction vial, [D₅]-4a (14 mg, 0.1 mmol) was used instead of 4a. The two reactions were kept at 60 °C for 5 min. Then they were diluted with DCM and combined. Silica gel was added and all the solvent were concentrated under vacuum. The residue was subject to flash chromatography to isolate the products (70% for combing yield) and the value of K_H/K_D was obtained based on ¹H NMR.
Reference:


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$KIE = 2.7$

$\text{H}_2/\text{D}_2$-$\text{D}4$-$\text{Saa}/[\text{D}_4]$-$\text{Saa}$