Supporting Information

Organocatalytic Enantioselective aza-Henry reaction of Ketimines Derived from Isatins: Access to Optically Active 3-Aminooxindoles

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General Note and procedure

Spectral data

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**General Note**

All reactions were performed in oven-dried glassware. All solvents and commercially available chemical were used without further purification. The molecular sieves were activated at 200 °C for 2 hours in an oven. The column chromatography was carried out on a column packed with silica gel 60-120. $^1$H NMR spectra were recorded in CDCl$_3$ on a BRUKER AVANCE III (500 MHz), JNM-ECS400 (400 MHz), BRUKER AVANCE II (400 MHz) and JEOL (300 MHz) spectrometer. $^{13}$C NMR spectra were recorded in CDCl$_3$ on BRUKER AVANCE III (125 MHz), JNM- ECS400 (100 MHz), BRUKER AVANCE II (100 MHz) and JEOL (75 MHz). Chemical shifts (δ) are expressed in ppm downfield from internal TMS. MS were recorded on micrOTOF-Q II 10356 Mass Spectrometer. Optical rotation was determined with AUTOPOL IV polarimeter at 25 °C using sodium D light. HPLC analyses were performed on a Shimadzu LC-20AD using Daicel Chiralpak OD-H, IA, IB and AS-H columns.

**General Procedure**

To the solution of ketimines derived from isatins (0.1 mmol), nitroalkane (0.25 mmol), 4Å MS (50 mg) in 0.3 mL of THF, the catalyst BnCPN (VI, 20 mol%) was added at 25 °C. The reaction mixture was stirred for 24 hours and the progress of the reaction was monitored at regular intervals by thin layer chromatography (tlc). After the completion of reaction, the crude reaction mixture was purified by column chromatography on silica gel (mesh 60–120) using hexane–ethyl acetate (1:1) as eluent. The enantiomeric excess of the purified 3 were determined using Daicel Chiralpak columns. The racemic standards were prepared using triethylamine (20 mol%) as a catalyst.
(R)-tert-Butyl 1-benzyl-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3a)

![Chemical structure of 3a](image)

Orange oil; 80% yield; $[\alpha]_{20}^D = -6.79$ (c 0.25, CHCl$_3$); 84% ee; HPLC [Chiralpak OD-H, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, $t_R = 16.3$ min (major) and $t_R = 29.3$ min (minor)];

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.28-7.76 (m, 8H), 6.65 (d, $J = 10.0$ Hz, 1H), 5.94 (s, 1H), 4.90-5.04 (m, 3H), 4.69 (d, $J = 15.0$ Hz, 1H), 1.41 (s, 9H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 28.13, 44.56, 59.91, 77.80, 81.32, 109.9, 123.5, 124.5, 125.8, 127.4, 127.9, 128.9, 130.3, 135.0, 142.5, 153.7, 172.8; HRMS calcd. for C$_{21}$H$_{23}$N$_3$O$_5$ [M+Na]$^+$ 404.1263; found 404.1233.

(R)-tert-Butyl 1-benzyl-5-chloro-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3b)

![Chemical structure of 3b](image)

Yellow semi-solid; yield 74%; $[\alpha]_{20}^D = -8.79$ (c 0.25, CHCl$_3$); 67% ee; [Chiralpak OD-H, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, $t_R = 8.83$ min (minor) and $t_R = 9.38$ min (major)];

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.23-7.48 (m, 7H), 6.70 (d, $J = 10.0$ Hz, 1H), 5.93 (s, 1H), 4.90-5.05 (m, 3H), 4.69 (d, $J = 15.0$ Hz, 1H), 1.41 (s, 9H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 28.15, 44.69, 59.81, 77.46, 81.68, 111.0, 125.1, 127.3, 128.0, 129.0, 130.3, 134.6, 141.1, 172.5; HRMS calcd. for C$_{21}$H$_{22}$ClN$_3$O$_5$ [M+Na]$^+$ 454.1146; found 454.1167.

(R)-tert-Butyl 1-benzyl-5-bromo-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3c)
Orange semi-solid; 79% yield; \([\alpha]_{20}^{D} = -9.19\) (c 0.25, CHCl\(_3\)); 73% ee; HPLC [Chiralpak OD-H, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, \(t_R = 13.6\) min (minor) and \(t_R = 15.6\) min (major)]; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.28-7.61 (m, 7H), 6.65 (d, \(J = 10.0\) Hz, 1H), 5.94 (s, 1H), 4.90-5.04 (m, 3H), 4.68 (d, \(J = 15.0\) Hz, 1H), 1.41 (s, 9H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 28.15, 44.66, 59.74, 77.47, 81.70, 111.5, 116.2, 127.3, 127.7, 127.8, 128.1, 129.0, 133.2, 134.5, 141.6, 153.7, 172.4; HRMS calcd. for C\(_{21}\)H\(_{22}\)BrN\(_3\)O\(_5\) [M+Na]\(^+\) 498.0635; found 498.0664.

\((R)-\text{tert-Butyl 1-benzyl-5-iodo-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3d)}\)

Yellow semi solid; yield 77%; \([\alpha]_{20}^{D} = -8.79\) (c 0.25, CHCl\(_3\)); 72% ee; [Chiralpak OD-H, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, \(t_R = 13.6\) min (minor) and \(t_R = 23.8\) min (major)]; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 7.28-7.74 (m, 7H), 6.55 (d, \(J = 9.0\) Hz, 1H), 5.95 (s, 1H), 4.87-5.04 (m, 3H), 4.66 (d, \(J = 12.0\) Hz, 1H), 1.41 (s, 9H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 28.16, 44.61, 59.56, 77.51, 81.70, 86.02, 112.0, 127.3, 128.1, 128.9, 133.1, 134.5, 139.2, 142.2, 153.7, 172.2; HRMS calcd. for C\(_{21}\)H\(_{22}\)IN\(_3\)O\(_5\) [M+Na]\(^+\) 546.0496; found 546.0545.

\((R)-\text{tert-Butyl 1-allyl-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3e)}\)
Yellow semi solid; yield 73%; $\left[\alpha\right]_{20}^{D} = -6.79$ (c 0.25, CHCl$_3$); 70% ee; [Chiralpak OD-H, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, $t_R = 12.6$ min (minor) and $t_R = 13.9$ min (major)]; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.28-7.63 (m, 3H), 6.80 (d, J = 5.0 Hz, 1H), 5.81-5.87 (m, 2H), 5.31-5.38 (m, 2H), 5.01 (d, J = 15.0 Hz, 1H), 4.66 (d, J = 15.0 Hz, 1H), 4.32-4.44 (m, 2H), 1.40 (s, 9H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 28.12, 43.03, 59.77, 77.76, 81.24, 109.8, 118.3, 123.4, 130.3, 130.7, 142.6, 172.5; HRMS calcd. for C$_{17}$H$_{21}$N$_3$O$_5$ [M + Na]$^+$ 370.1378; found 370.1389.

(R)-tert-Butyl 1-allyl-5-chloro-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3f)

Yellow oil; yield 76%; $[\alpha]_{20}^{D} = -9.03$ (c 0.25, CHCl$_3$); 76% ee; [Chiralpak OD-H, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, $t_R = 9.67$ min (major) and $t_R = 10.5$ min (minor)]; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.28-7.51 (m, 2H), 6.84 (d, J = 10.0 Hz, 1H), 5.83-5.86 (m, 2H), 5.29-5.38 (m, 2H), 5.02 (d, J = 15.0 Hz, 1H), 4.66 (d, J = 10.0 Hz, 1H), 4.32-4.47 (m, 2H), 1.40 (s, 9H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 28.13, 43.14, 59.66, 77.41, 81.62, 110.9, 118.5, 125.2, 127.4, 128.9, 130.3, 141.2, 153.7, 172.2; HRMS calcd. for C$_{17}$H$_{20}$ClN$_3$O$_5$ [M + Na]$^+$ 404.0984; found 404.0996.

(R)-tert-Butyl 1-allyl-5-bromo-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3g)

White solid; yield 77%; $[\alpha]_{20}^{D} = -9.99$ (c 0.25, CHCl$_3$); 73% ee; [Chiralpak OD-H, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, $t_R = 12.4$ min (minor) and $t_R = 13.5$ min (major)]; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 7.46-7.60 (m, 2H), 6.78 (d, J= 8.4 Hz, 1H), 5.79-5.85 (m, 2H), 5.28 (d, J= 11.1 Hz, 2H), 4.98 (d, J= 12.3 Hz, 1H), 4.63 (d, J= 12.6 Hz, 1H), 4.29-4.47 (m, 2H), 1.37 (s, 9H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 28.13, 43.14, 59.66, 77.41, 81.61,
(R)-tert-Butyl 1-allyl-5-iodo-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3h)

Yellow oil; yield 78%; \([\alpha]_{20}^D = -7.99\) (c 0.25, CHCl\(_3\)); 89% ee; HPLC [Chiralpak IB, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, \(t_R = 10.1\) min (minor) and \(t_R = 15.4\) min (major)]; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 7.26-7.75 (m, 2H), 6.67 (d, \(J = 9.0\) Hz, 1H), 5.79-5.87 (m, 2H), 5.25-5.77 (m, 2H), 4.95 (d, \(J = 12.0\) Hz, 1H), 4.61 (d, \(J = 12.0\) Hz, 1H), 4.28-4.44 (m, 2H), 1.37 (s, 9H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 28.13, 43.05, 50.90, 59.41, 77.46, 81.64, 85.89, 111.9, 118.5, 128.0, 130.3, 133.2, 139.2, 142.4, 171.8; HRMS calcd. for C\(_{17}\)H\(_{20}\)BrN\(_3\)O\(_5\) [M+Na]\(^+\) 448.0479; found 448.0502

(R)-tert-Butyl 5-chloro-1-(2-methylallyl)-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3i)

Yellow oil; yield 71%; \([\alpha]_{20}^D = -11.6\) (c 0.25, CHCl\(_3\)); 75% ee; HPLC [Chiralpak OD-H, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, \(t_R = 7.34\) min (minor) and \(t_R = 8.24\) min (major)]; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 7.26-7.47 (m, 2H), 6.83 (d, \(J = 9.0\) Hz, 1H), 5.85 (s, 1H), 4.93-4.98 (m, 3H), 4.63 (d, \(J = 12.0\) Hz, 1H), 4.21-4.38 (m, 2H), 1.77 (s, 3H), 1.37 (s, 9H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 19.90, 28.13, 46.78, 50.90, 59.71, 77.43, 81.61, 111.0, 113.5, 125.1, 127.4, 128.9, 130.3, 138.2, 141.4, 172.2; HRMS calcd. for C\(_{18}\)H\(_{22}\)ClN\(_3\)O\(_5\) [M+Na]\(^+\) 418.1140; found 418.1156
(R)-tert-Butyl 1-((E)-but-2-enyl)-5-chloro-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3j)

Yellow oil; yield 70%; $[\alpha]_D^{20} = -7.13$ (c 0.25, CHCl$_3$); 79% ee; [Chiralpak IB, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, $t_R = 6.24$ min (minor) and $t_R = 6.91$ min (major)]; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.32-7.50 (m, 2H), 6.85 (d, $J = 10.0$ Hz, 1H), 5.85 (s, 1H), 4.97-5.01 (m, 3H), 4.66 (d, $J = 15.0$ Hz, 1H), 4.33 (dd, $J = 55.0$ Hz, $J = 15.0$ Hz, 2H), 1.80 (s, 3H), 1.40 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 20.05, 28.26, 46.89, 59.83, 81.74, 111.2, 113.7, 125.2, 127.5, 129.1, 130.5, 138.4, 141.5, 153.8, 172.5; HRMS calcd. for C$_{18}$H$_{22}$ClN$_3$O$_5$ [M+Na]$^+$ 418.1140; found 418.1149

(R)-tert-Butyl 5-chloro-3-(nitromethyl)-2-oxoindolin-3-ylcarbamate (3k)

Yellow oil; yield 68%; $[\alpha]_D^{20} = +21.4$ (c 0.25, CHCl$_3$); 74% ee; HPLC [Chiralpak IA, hexane/i-PrOH, 9:1, 1 mL/min, 254 nm, $t_R = 11.3$ min (major) and $t_R = 15.6$ min (minor)]; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 8.48 (s, 1H), 7.26-7.31 (m, 2H), 6.83 (d, $J = 8.4$ Hz, 1H), 6.19 (s, 1H), 4.75 (dd, $J = 58.8$ Hz and $J = 12.6$ Hz, 2H), 1.38 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 22.69, 44.00, 55.81, 80.71, 110.5, 112.0, 133.1, 114.8, 115.3, 118.1, 121.6, 127.9, 128.1, 128.9, 129.0, 134.6, 135.3, 136.1, 144.6, 172.0, 183.6; HRMS calcd. for C$_{14}$H$_{16}$ClN$_3$O$_5$ [M+Na]$^+$ 364.0671; found 364.0686
3(R),1'(R/S)-tert-Butyl 5-chloro-1-(2-methylallyl)-3-(1'-nitroethyl)-2-oxoindolin-3-ylcarbamate (3l)

Brown solid; yield 75%; [α]_20^D = -9.19 (c 0.25, CHCl₃); dr 72:28; [Chiralpak IA, hexane/i-PrOH, 90:10, 1 mL/min, 254 nm, ee = 80% of major diastereomer and ee = 82% of minor diastereomer, t_R = 5.60 min (major) , t_R = 8.56 min (major) and t_R = 6.31 min (minor), t_R = 16.3 min (minor)]; \(^1\)H NMR (500 MHz, CDCl₃) δ 6.82-7.39 (m, 3H), 6.09 (d, J= 25.0 Hz, 1H), 5.00-5.06 (m, 3H), 4.31 (dd, J= 65.0 Hz, J= 20.0 Hz, 2H), 1.74-1.81 (m, 3H), 1.58 (s, 3H), 1.35 (s, 9H); \(^{13}\)C NMR (125 MHz, CDCl₃) δ 13.19, 20.13, 28.11, 46.68, 62.61, 81.33, 84.56, 110.6, 113.3, 113.6, 123.6, 124.8, 128.7, 130.1, 130.2, 138.5, 142.4, 172.9; HRMS calcd. for C₁₉H₂₄ClN₃O₅ [M+Na]^+ 432.1297; found 432.1300.

3(R),1'(R/S)-tert-Butyl-5-chloro-1-(2-methylallyl)-3-(1'-nitropropyl)-2-oxoindolin-3-ylcarbamate (3m)

Yellow semi-solid; yield 75%; dr 55:45, [Chiralpak IA, hexane/i-PrOH, 90:10, 1 mL/min, 210 nm, ee = 56% of major diastereomer and ee = 45% of minor diastereomer, t_R = 4.72 min (major) , t_R = 6.79 min (major) and t_R = 5.16 min (minor), t_R = 8.62 min (minor)]; \(^1\)H NMR (300 MHz, CDCl₃) δ 7.10-7.30 (m, 2H), 6.80 (d, J= 12.3 Hz, 1H), 6.11 (s, 1H), 4.78-4.98 (m, 3H), 4.77-4.82 (m, 1H), 4.27 (dd, J= 33.8 Hz and J= 15.6 Hz, 2H), 2.16-2.42 (m, 2H), 1.78 (s, 3H), 1.33 (s, 9H), 0.94 (t, J= 14.4 Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl₃) δ 10.63, 20.70, 28.08, 46.65, 62.43, 81.40, 91.74, 92.81, 110.7, 112.4, 113.3, 113.6, 123.3, 125.3, 128.6, 128.8, 130.1, 138.4, 141.0, 142.0, 153.4, 171.9, 173.2.
3\((R)\),1'\((R/S)\)-tert-Butyl 1-allyl-5-iodo-3-(1'-nitropropyl)-2-oxoindolin-3-ylcarbamate (3n)

Yellow semi-solid; yield 80%; dr 54:46, [Chiralpak IA, hexane/i-PrOH, 90:10, 1 mL/min, 208 nm, ee = 64% of major diastereomer, \(t_R = 6.15\) min (major), \(t_R = 7.63\) min (minor); and ee = 67% of minor diastereomer, \(t_R = 10.6\) min (major) \(t_R = 12.5\) min (minor)]; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.37-7.68 (m, 3H), 6.66-6.73 (m, 1H), 6.10 (s, 1H), 5.82-5.84 (m, 1H), 5.28-5.40 (m, 2H), 4.27-4.44 (m, 3H), 2.15-2.42 (m, 2H), 1.41 (s, 9H), 0.94-0.99 (m, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 10.57, 20.59, 29.71, 43.09, 62.18, 81.46, 85.79, 91.78, 92.62, 111.5, 113.1, 118.7, 119.1, 130.4, 130.6, 131.5, 133.5, 133.9, 138.9, 142.0, 142.9, 153.0, 172.5.
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Shimadzu LCsolution Analysis Report

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Shimadzu LCsolution Analysis Report

---Graph 1---

D:/HPLC/Akshay/Shiff Base derivatives (ch3no2)/scff bases/crotylist+CH3NO2, 1ml, 10% IB rec.lcd

---Graph 2---

D:/HPLC/Akshay/Shiff Base derivatives (ch3no2)/scff bases/crotylist+CH3NO2, 1ml, 10% IB chiral.lcd

---Table 1---

<table>
<thead>
<tr>
<th>Peak#</th>
<th>Ret. Time</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
<th>Height %</th>
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---Table 2---

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== Shimadzu LCsolution Analysis Report ==

D:\HPLC\Akshay\niroethane\l-Nallylscff base+NP, 1ml,10%, IA reclcd

1 PDA Multi 1/209nm 4nm

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D:\HPLC\Akshay\niroethane\l-Nallylscff base+NP, 1ml,10%, IA.lcd

1 PDA Multi 1/208nm 4nm

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</thead>
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NHO

BOCN

Cl

NO₂

CH

3j

--- PROCESSING PARAMETERS ---

Date: 2023-03-21

Sample: NHO

Solvent: DMSO

Creation time: 2023-03-21 15:00:00

Current time: 2023-03-21 15:00:19

Comment: Simple pulse decoupling

Data format: NMR

Dim: 1024

Dim axis: 100C

Dimensions: 1D

Site: ECN 400

Spectrometer: NMR 400

Field strength: 5.29776T (400 MHz)

Avg. duration: 1.0934993[ms]

G protein: 100 (gpm)

Teckels: 100

Mez: 1

Total scans: 2048

X 1D width: 7.67 (hertz)

X 1D time: 1.04229012[s]

X 1D size: 1024

X 1D exit: 1024

X 1D exit: 1024

Decoupling: TRNS

Temp exit: 293(K)

Peltier gain: 60

Peltier delay: 2.00

Repeat time: 1.5433312[ms]

Temp set: 19.5(°C)
May03-2014
AKS-B-14

N
O
HN
Boc

I
3
N
O
2

3n