Supporting Information

Asymmetric Michael addition reactions of pyrrolones with chalcones catalyzed by vicinal primary-diamine salts

Xiaolei Du, Dawei Yin, Zemei Ge, Xin Wang* and Runtao Li*

State Key Laboratory of Nature and Biomimetic Drugs, School of Pharmaceutical Science, Peking University, Beijing 100191, China

Contents

A. General information ................................................................................................2
B. Experimental procedures and characterization data of products .........................2
  a General procedure for the synthesis of products .....................................................2
  b Characterization data ............................................................................................3
  c NMR Spectra .........................................................................................................14
  d HPLC Spectra .......................................................................................................32
A. General information

$^1$H NMR spectra were recorded on a Bruker AVANCE III-400 spectrometer. Chemical shifts (in ppm) were referenced to the solvent residual signal ($\delta = 7.26$ ppm) of CDCl$_3$. $^{13}$C NMR spectra were obtained by using the same NMR spectrometer and were calibrated to CDCl$_3$ ($\delta = 77.00$ ppm). High Resolution Mass spectra were recorded using a Fourier Transform Ion Cyclotron Resonance Mass Spectrometer (APEX IV, Bruker). Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Column chromatography was carried out on silica gel (particle size 200–300 mesh ASTM).

B. Experimental procedures and characterization data of products

a. General procedure for the synthesis of products

\[
\begin{align*}
\text{1a } & \text{ Ar}_1=\text{Ph} & 1b & \text{ Ar}_1=4-\text{BrC}_6\text{H}_4 & 1c & \text{ Ar}_1=4-\text{OMeC}_6\text{H}_4
\end{align*}
\]

Pyrroline 1 (0.15 mmol), chalcone 2 (0.225 mmol) was dissolved in toluene (1.5 mL), and the catalyst C1 (0.03 mmol), acid additive (0.06 mmol) was added. The mixture was reacted at 40 \degree C for 5 days. After the completion of the reaction as indicated by TLC, the mixture was cooled to room temperature and the solvent was evaporated. 10 mL of CH$_2$Cl$_2$ was added and the mixture was washed with 5\% HCl, 2\% NaOH, saturated NaCl, the organic phase was dried with anhydrous Na$_2$SO$_4$. The mixture was filtered, the solvent was evaporated in vacuo to afford the crude product. Chromatography on silica gel (ethyl acetate-dichloromethane-petroleum ether=1:1:4) gave the corresponding product 3 as a colourless oil.
b. Characterization data

\[ \text{N-Benzyl-5-(3-oxo-1,3-diphenylpropyl)-4-phenyl-1H-pyrrol-2(5H)-one (3a)} \]

Yield 65%, colourless oil: \(^1\text{H NMR (400 MHz, CDCl}_3\)): (syn) \(\delta 7.89 (d, J = 7.7 \text{ Hz}, 2\text{H}), 7.62 (t, J = 7.4 \text{ Hz}, 1\text{H}), 7.50 (t, J = 7.6 \text{ Hz}, 2\text{H}), 7.30 (m, 4\text{H}), 7.28 - 7.20 (m, 4\text{H}), 7.18 - 7.11 (m, 2\text{H}), 7.09 (d, J = 7.1 \text{ Hz}, 1\text{H}), 7.03 (t, J = 7.4 \text{ Hz}, 2\text{H}), 6.65 (d, J = 7.5 \text{ Hz}, 2\text{H}), 6.22 (s, 1\text{H}), 5.49 (dd, J = 15.3 \text{ Hz}, 1\text{H}), 4.91 (d, J = 1.6 \text{ Hz}, 1\text{H}), 4.34 (d, J = 15.4 \text{ Hz}, 1\text{H}), 4.16 (td, J = 7.3, 2.3 \text{ Hz}, 1\text{H}), 3.46 - 3.29 (m, 2\text{H}). \(^{13}\text{C NMR (100 MHz, CDCl}_3\)): (syn) \(\delta 197.57, 171.86, 160.48, 137.67, 137.00, 136.70, 133.43, 132.48, 129.52, 128.95, 128.71, 128.58, 128.14, 128.08, 128.01, 127.94, 127.79, 127.37, 126.92, 122.43, 64.53, 45.67, 40.94, 37.41\); ES-HRMS: Calcd for \(\text{C}_{32}\text{H}_{28}\text{NO}_2 [M+H]^+\), 458.2114, Found 458.2105.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=85:15, flow rate:1.0 mL/min): \(t_R 9.90\text{min (minor)}, \ t_R 20.71\text{min (major)}, \ ee 95\%\)

\[ \text{N-Benzyl-5-(3-oxo-1,3-diphenylpropyl)-4-(4-bromophenyl)-1H-pyrrol-2(5H)-one (3b)} \]

Yield 40%, colourless oil: \(^1\text{H NMR (400 MHz, CDCl}_3\)): (syn) \(\delta 7.89 (d, J = 7.6 \text{ Hz}, 2\text{H}), 6.80-7.80(m, 15\text{H}), 6.66 (d, J = 7.4 \text{ Hz}, 2\text{H}), 6.22 (s, 1\text{H}), 5.48 (d, J = 15.3 \text{ Hz}, 1\text{H}), 4.87 (m, 1\text{H}), 4.32 (d, J = 15.4 \text{ Hz}, 1\text{H}), 4.18 - 4.06 (m, 1\text{H}), 3.37 (d, J = 7.1 \text{ Hz},
$^{13}$C NMR (100 MHz, CDCl$_3$): (syn) $\delta$ 197.48, 171.52, 159.14, 139.11, 137.51, 137.04, 136.84, 136.64, 133.52, 132.42, 131.79, 128.98, 128.84, 128.75, 128.21, 128.15, 127.98, 127.93, 123.93, 122.89, 64.38, 45.72, 40.91, 37.30; ES-HRMS: Calcd for C$_{32}$H$_{27}$BrNO$_2$ [M+H]$^+$, 536.1219, Found 536.1199.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=85:15, flow rate:1.0 mL/min): $t_R$ 11.41 min (minor), $t_R$ 26.52 min (major), ee 90%

![Chemical structure](image)

$N$-Benzy-5-(3-oxo-1,3-diphenylpropyl)-4-methoxyphenyl-1H-pyrrol-2(5H)-one (3c)

Yield 55%, colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn) $\delta$ 7.95 – 7.86 (m, 2H), 7.62 (t, $J$ = 7.4 Hz, 1H), 7.50 (t, $J$ = 7.7 Hz, 2H), 7.30 (m, 3H), 7.24 (m, 2H), 7.19 – 7.10 (m, 3H), 7.07 (t, $J$ = 7.2 Hz, 2H), 6.79 (d, $J$ = 8.8 Hz, 2H), 6.69 (d, $J$ = 7.1 Hz, 2H), 6.12 (s, 1H), 5.49 (d, $J$ = 15.4 Hz, 1H), 4.87 (d, $J$ = 2.1 Hz, 1H), 4.34 (d, $J$ = 15.4 Hz, 1H), 4.14 (td, $J$ = 7.2, 2.6 Hz, 1H), 3.83 (s, 3H), 3.41 (d, $J$ = 7.3 Hz, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$): (syn) $\delta$ 197.70, 172.31, 160.73, 160.10, 137.75, 137.09, 136.71, 133.44, 128.93, 128.90, 128.71, 128.11, 128.07, 127.95, 127.72, 126.94, 125.00, 120.41, 114.03, 64.29, 55.36, 45.84, 41.27, 37.86; ES-HRMS: Calcd for C$_{33}$H$_{30}$NO$_3$ [M+H]$^+$, 488.2220, Found 488.2202.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=85:15, flow rate:1.0 mL/min): $t_R$ 12.07 min (minor), $t_R$ 27.83 min (major), ee 90%
**N-Benzyl-5-(1-(3-methoxyphenyl)-3-oxo-3-phenylpropyl)-4-phenyl-1H-pyrrol-2(5H)-one (3d)**

Yield 70%, colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn) $\delta$ 7.79 (d, $J = 7.8$ Hz, 2H), 6.80-7.65 (m, 15H), 6.65 (m, 2H), 6.35 (s, 1H), 5.27 (d, $J = 14.9$ Hz, 1H), 4.90 (m, 1H), 4.09 (m, 1H), 3.91 (d, $J = 14.9$ Hz, 1H), 3.76 (s, 3H), 3.32 (ddd, $J = 24.2$, 17.9, 7.1 Hz, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$): (syn) $\delta$ 197.37, 171.66, 159.71, 159.36, 140.98, 137.22, 136.51, 133.31, 132.45, 130.20, 129.56, 129.18, 128.96, 128.62, 128.49, 128.18, 127.97, 127.33, 123.37, 123.37, 120.26, 114.34, 112.20, 65.11, 55.19, 45.00, 40.65, 35.55; ES-HRMS: Calcd for C$_{33}$H$_{30}$NO$_3$ [M+H]$^+$, 488.2220, Found 488.2220.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=85:15, flow rate:1.0 mL/min): $t_R$ 12.39 min (minor), $t_R$ 30.86 min (major), ee 90%

**N-Benzyl-5-(1-(4-chlorophenyl)-3-oxo-3-phenylpropyl)-4-phenyl-1H-pyrrol-2(5H)-one (3e)**

Yield 62%, colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn) $\delta$ 7.92 – 7.84 (m, 2H), 7.63 (t, $J = 7.4$ Hz, 1H), 7.50 (t, $J = 7.7$ Hz, 2H), 7.35 – 7.30 (m, 4H), 7.29 – 7.24 (m, 4H), 7.20 – 7.15 (m, 2H), 7.00 (t, $J = 5.5$ Hz, 2H), 6.56 (d, $J = 8.4$ Hz, 2H), 6.23 (s, 1H), 5.46 (d, $J = 15.4$ Hz, 1H), 4.87 (d, $J = 2.1$ Hz, 1H), 4.34 (d, $J = 15.4$ Hz, 1H),
4.10 (td, $J = 7.2, 2.7$ Hz, 1H), 3.36 (d, $J = 7.2$ Hz, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$): (syn) $\delta$ 197.23, 171.93, 160.25, 136.90, 136.55, 136.22, 133.56, 132.80, 132.36, 129.74, 129.32, 128.98, 128.76, 128.71, 128.18, 128.11, 127.93, 127.87, 127.36, 122.51, 64.41, 45.96, 40.62, 37.65; ES-HRMS: Calcd for C$_{32}$H$_{27}$ClNO$_2$ [M+H]$^+$, 492.1724, Found 492.1727.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=85:15, flow rate:1.0 mL/min): $t_R$ 10.19 min (minor), $t_R$ 20.38 min (major), ee 81%

$\text{N-Benzyl-5-(1-(3-nitrophenyl)-3-oxo-3-phenylpropyl)-4-phenyl-1H-pyrrrol-2(5H)-one (3f)}$

Yield 75%, colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn) $\delta$ 7.89 (d, $J = 7.4$ Hz, 2H), 7.22-7.55 (m, 15H), 7.14 (m, 2H), 6.23 (s, 1H), 5.37 (d, $J = 15.3$ Hz, 1H), 4.87 (m, 1H), 4.40 (d, $J = 15.4$ Hz, 1H), 4.24 (m, 1H), 3.41 (d, $J = 7.1$ Hz, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$): (syn) $\delta$ 196.82, 171.79, 160.02, 147.77, 140.00, 136.76, 136.27, 134.76, 134.25, 133.80, 129.98, 129.32, 129.14, 128.95, 128.88, 128.84, 128.77, 128.17, 128.06, 127.98, 127.49, 127.27, 123.02, 122.82, 122.05, 64.70, 46.13, 40.78, 36.96; ES-HRMS: Calcd for C$_{32}$H$_{27}$N$_2$O$_4$ [M+H]$^+$, 503.1965, Found 503.1971.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=75:25, flow rate:0.3 mL/min): $t_R$ 48.27 min (minor), $t_R$ 81.21 min (major), ee 92%
**N-Benzyl-5-(1-(3-methoxyphenyl)-3-oxo-3-phenylpropyl)-4-(4-methoxyphenyl)-1H-pyrrolo[2(5H)]-one (3g)**

Yield 64%, colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn) $\delta$ 7.92 – 7.86 (m, 2H), 7.50 (t, $J = 7.7$ Hz, 2H), 7.41 (dd, $J = 17.8, 8.3$ Hz, 3H), 7.28 – 7.20 (m, 5H), 7.02 – 6.91 (m, 2H), 6.87 – 6.80 (m, 2H), 6.29 (s, 1H), 6.16 (s, 2H), 5.48 (d, $J = 15.4$ Hz, 1H), 4.84 (d, $J = 2.1$ Hz, 1H), 4.30 (d, $J = 15.4$ Hz, 1H), 4.14 (td, $J = 7.4, 2.7$ Hz, 1H), 3.82 (s, 3H), 3.58 (s, 3H), 3.34 (qd, $J = 18.0, 7.2$ Hz, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$): (syn) $\delta$ 197.65, 172.10, 160.73, 160.05, 159.24, 139.44, 137.09, 136.73, 133.41, 129.05, 128.91, 128.70, 128.43, 128.17, 127.94, 127.73, 125.10, 120.58, 120.46, 114.62, 113.94, 112.54, 64.53, 55.34, 54.92, 45.54, 41.00, 37.24; ES-HRMS: Calcd for C$_{34}$H$_{32}$NO$_4$ [M+H]$^+$, 518.2325, Found 518.2326.

The enantiomeric excess was determined by HPLC analysis (OD-H, hexane:IPA=85:15, flow rate:1.0 mL/min): $t_R$ 14.63 min (minor), $t_R$ 40.45 min (major), ee 93%

**N-Benzyl-5-(1-(3-methylphenyl)-3-oxo-3-phenylpropyl)-4-(4-methoxyphenyl)-1H-pyrrolo[2(5H)]-one (3h)**

Yield 55%, colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn) $\delta$ 7.89 (d, $J = 7.3$ Hz, 2H), 7.50 (t, $J = 7.7$ Hz, 2H), 7.30 (m, 5H), 6.95 (m, 4H), 6.78 (t, $J = 6.8$ Hz, 2H), 6.38
(s, 1H), 6.14 (s, 1H), 5.43 (d, $J = 15.4$ Hz, 1H), 4.83 (m, 1H), 4.34 (d, $J = 15.4$ Hz, 1H), 4.12 (td, $J = 7.1, 2.7$ Hz, 1H), 3.82 (s, 3H), 3.35 (m, 2H), 2.11 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$): (syn) $\delta$ 197.74, 172.15, 160.70, 160.19, 139.56, 137.73, 137.46, 137.22, 136.79, 133.37, 128.96, 128.89, 128.68, 128.38, 128.21, 127.96, 127.70, 127.31, 124.78, 120.60, 113.88, 64.80, 55.33, 45.67, 40.60, 37.33, 21.28; ES-HRMS: Calcd for C$_{34}$H$_{32}$NO$_3$ [M+H]$^+$, 502.2376, Found 502.2373.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA= 85:15, flow rate:1.0 mL/min): $t_R$ 10.72min (minor), $t_R$ 22.10min (major), ee 85%.

$N$-BenzyI-5-(1-(p-tolyl)-3-oxo-3-phenylpropyl)-4-(4-methoxyphenyl)-1H-pyrrole-2(5H)-one (3i)

Yield 60%, colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn) $\delta$ 7.89 (d, $J = 8.0$ Hz, 2H), 7.62 (t, $J = 7.3$ Hz, 1H), 7.50 (t, $J = 7.6$ Hz, 2H), 7.28 (m, 3H), 7.20 (m, 4H), 6.88 (d, $J = 7.8$ Hz, 2H), 6.81 (d, $J = 8.6$ Hz, 2H), 6.58 (d, $J = 7.8$ Hz, 2H), 6.11 (s, 1H), 5.48 (d, $J = 15.4$ Hz, 1H), 4.86 (d, $J = 1.0$ Hz, 1H), 4.33 (d, $J = 15.4$ Hz, 1H), 4.07 (t, $J = 6.2$ Hz, 1H), 3.84 (s, 3H), 3.50 – 3.32 (m, 2H), 2.25 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$): (syn) $\delta$ 197.79, 172.40, 166.75, 160.19, 137.14, 136.48, 134.58, 133.37, 128.97, 128.85, 128.71, 128.68, 128.07, 127.98, 127.95, 127.66, 125.08, 120.31, 114.01, 64.22, 55.34, 45.95, 41.13, 38.35, 20.96; ES-HRMS: Calcd for C$_{33}$H$_{32}$NO$_3$ [M+H]$^+$, 502.2376, Found 502.2374.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA= 85:15, flow rate:1.0 mL/min): $t_R$ 10.20min (minor), $t_R$ 22.10min (major), ee 85%.
**N-Benzyl-5-(1-(3-chloroophenyl)-3-oxo-3-phenylpropyl)-4-(4-methoxyphenyl)-1H-pyrrol-2(5H)-one (3j)**

Yield 82%, colourless oil; $^1$H NMR (400 MHz, CDCl$_3$): (syn) $\delta$ 7.88 (d, $J = 7.7$ Hz, 2H), 7.63 (t, $J = 7.2$ Hz, 1H), 7.51 (t, $J = 7.5$ Hz, 2H), 7.32 (m, 3H), 7.28 (m, 2H), 7.10 (t, $J = 7.7$ Hz, 3H), 7.02 (t, $J = 7.7$ Hz, 1H), 6.81 (d, $J = 8.2$ Hz, 3H), 6.62 (d, $J = 7.6$ Hz, 1H), 6.53 (s, 1H), 6.15 (s, 1H), 5.39 (d, $J = 15.4$ Hz, 1H), 4.81 (s, 1H), 4.34 (d, $J = 15.3$ Hz, 1H), 4.11 (t, $J = 6.6$ Hz, 1H), 3.84 (s, 3H), 3.38 – 3.27 (m, 2H).

$^{13}$C NMR (100 MHz, CDCl$_3$): (syn) $\delta$ 197.25, 172.22, 160.95, 159.96, 140.01, 137.02, 136.52, 133.88, 133.58, 129.28, 128.99, 128.91, 128.75, 128.39, 128.16, 127.96, 127.84, 127.13, 126.38, 124.83, 120.69, 114.14, 64.60, 55.38, 45.92, 40.86, 37.23; ES-HRMS: Calcd for C$_{33}$H$_{29}$ClNO$_3$ [M+H]$^+$, 522.1830, Found 522.1830.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA= 92:8, flow rate:1.0 mL/min): $t_R$ 25.87 min (minor), $t_R$ 57.63 min (major), ee 83%

**N-Benzyl-5-(1-(4-chlorophenyl)-3-oxo-3-phenylpropyl)-4-(4-methoxyphenyl)-1H-pyrrol-2(5H)-one (3k)**
Yield 64%, colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn) δ 7.88 (d, $J = 7.6$ Hz, 2H), 7.63 (t, $J = 7.4$ Hz, 1H), 7.51 (t, $J = 7.7$ Hz, 2H), 7.30 (m, 3H), 7.26 – 7.20 (m, 2H), 7.15 (d, $J = 8.7$ Hz, 2H), 7.03 (d, $J = 8.4$ Hz, 2H), 6.82 (d, $J = 8.7$ Hz, 2H), 6.59 (d, $J = 8.4$ Hz, 2H), 6.12 (s, 1H), 5.45 (d, $J = 15.4$ Hz, 1H), 4.83 (d, $J = 2.3$ Hz, 1H), 4.33 (d, $J = 15.4$ Hz, 1H), 4.08 (td, $J = 7.1$, 2.4 Hz, 1H), 3.84 (s, 3H), 3.46 – 3.30 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$): (syn) δ 197.37, 172.39, 160.89, 159.91, 136.98, 136.54, 136.29, 133.58, 132.76, 129.41, 128.94, 128.76, 128.14, 128.08, 127.94, 127.81, 124.81, 120.47, 114.14, 64.21, 55.40, 46.10, 40.92, 37.99; ES-HRMS: Calcd for C$_{33}$H$_{29}$ClNO$_3$ [M+H]$^+$, 522.1830, Found 522.1833.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=90:10, flow rate:1.0 mL/min): $t_R$ 18.24 min (minor), $t_R$ 38.63 min (major), ee 87%

![Chemical structure](image)

$N$-Benzyl-5-(1-(4-fluorophenyl)-3-oxo-3-phenylpropyl)-4-(4-methoxyphenyl)-1H-pyrrol-2(5H)-one (3l)

Yield 55%, colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn) δ 7.90 – 7.86 (m, 2H), 7.63 (t, $J = 7.4$ Hz, 1H), 7.51 (t, $J = 7.7$ Hz, 2H), 7.30 (m, 3H), 7.23 (m, 2H), 7.17 (d, $J = 8.8$ Hz, 2H), 6.83 (d, $J = 8.8$ Hz, 2H), 6.77 (t, $J = 8.7$ Hz, 2H), 6.63 (m, 2H), 6.12 (s, 1H), 5.46 (d, $J = 15.4$ Hz, 1H), 4.84 (d, $J = 1.9$ Hz, 1H), 4.34 (d, $J = 15.4$ Hz, 1H), 4.09 (td, $J = 7.2$, 2.3 Hz, 1H), 3.84 (s, 3H), 3.43 (dd, $J = 18.2$, 7.1 Hz, 1H), 3.36 (dd, $J = 18.1$, 7.7 Hz, 1H), $^{13}$C NMR (100 MHz, CDCl$_3$): (syn) δ 197.49, 172.44, 161.71 (d, $J = 244.0$ Hz), 160.88, 160.00, 137.02, 136.60, 133.54, 129.63, 129.55, 128.92 (d, $J = 1.5$ Hz), 128.75, 128.07, 127.94, 127.78, 124.88, 120.42, 114.97, 114.76, 114.14,
ES-HRMS: Calcd for C_{33}H_{29}FNO_{3} [M+H]^+, 506.2126, Found 506.2126.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=85:15, flow rate:1.0 mL/min): t_R 13.20 min (minor), t_R 29.05 min (major), ee 85%

\[ \text{N-Benzyl-5-(3-oxo-1-phenyl-3-(p-tolyl)propyl)-4-(4-methoxyphenyl)-1H-pyrrolo-2(5H)-one (3m)} \]

Yield 69%; colourless oil; \(^1^H\) NMR (400 MHz, CDCl\(_3\)): (syn) \(\delta\) 7.81 (d, \(J = 8.2\) Hz, 2H), 7.39 (d, \(J = 8.7\) Hz, 2H), 7.34 – 7.04 (m, 12H), 6.81 (m, 2H), 6.11 (s, 1H), 5.48 (d, \(J = 15.4\) Hz, 1H), 4.89 (d, \(J = 1.9\) Hz, 1H), 4.35 (d, \(J = 15.4\) Hz, 1H), 4.15 (m, 1H), 3.82 (s, 3H), 3.38 (m, 2H), 2.45 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): (syn) \(\delta\) 197.33, 172.37, 160.75, 160.17, 144.28, 137.86, 137.18, 134.31, 129.38, 128.94, 128.88, 128.45, 128.16, 128.09, 127.96, 127.70, 126.91, 125.04, 120.37, 114.05, 64.31, 55.35, 45.91, 41.41, 37.85, 21.70; ES-HRMS: Calcd for C\(_{34}\)H\(_{32}\)NO\(_3\) [M+H]^+, 502.2376, Found 502.2373.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=95:5, flow rate:1.0 mL/min): t_R 39.43 min (minor), t_R 93.11 min (major), ee 84%
**N-Benzyl-5-(1-(3-bromophenyl)-3-oxo-3-(p-tolyl)propyl)-4-(4-methoxyphenyl)-1H-pyrrol-2(5H)-one (3n)**

Yield 66%; colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn+anti) δ 7.81 (d, $J = 8.0$ Hz, 1H), 7.74 (d, $J = 8.0$ Hz, 1H), 7.38 (t, $J = 12.4$ Hz, 1H), 7.35 – 7.27 (m, 3H), 7.22 (dd, $J = 11.2$, 8.6 Hz, 2H), 7.10 (d, $J = 8.8$ Hz, 1H), 6.99 – 6.87 (m, 2H), 6.78 (t, $J = 7.3$ Hz, 2H), 6.53 (d, $J = 7.4$ Hz, 0H), 6.38 (s, 0H), 6.27 (s, 0H), 6.13 (s, 0H), 5.43 (d, $J = 15.3$ Hz, 0H), 5.23 (d, $J = 14.9$ Hz, 0H), 4.84 (s, 1H), 4.35 (d, $J = 15.3$ Hz, 0H), 4.21 – 4.03 (m, 1H), 3.82 (dd, $J = 13.1$, 8.6 Hz, 3H), 3.50 (dd, $J = 17.8$, 9.3 Hz, 0H), 3.33 (d, $J = 7.2$ Hz, 1H), 2.94 (dd, $J = 17.8$, 5.0 Hz, 0H), 2.45 (s, 1H), 2.42 (s, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): (syn+anti) δ 197.37, 197.13, 172.23, 172.15, 161.15, 160.69, 160.26, 159.15, 144.22, 144.10, 139.65, 138.16, 137.81, 137.41, 137.36, 137.27, 134.34, 134.17, 129.36, 129.28, 128.97, 128.91, 128.88, 128.86, 128.44, 128.39, 128.24, 128.18, 128.15, 128.10, 127.92, 127.90, 127.69, 127.61, 127.31, 125.28, 125.22, 124.80, 121.21, 120.53, 114.58, 113.89, 65.35, 64.79, 55.40, 55.33, 45.73, 45.05, 41.04, 40.80, 37.28, 34.95, 21.70, 21.65, 21.52, 21.29; ES-HRMS: Calcd for C$_{34}$H$_{31}$BrNO$_3$ [M+H]$^+$, 516.2533, Found 516.2534.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=85:15, flow rate:1.0 mL/min): $t_r$ 9.88 min (minor), $t_r$ 18.34 min (major), ee 85%

**N-Benzyl-5-(1-(3-bromophenyl)-3-oxo-3-(p-tolyl)propyl)-4-(4-methoxyphenyl)-1H-pyrrol-2(5H)-one (3o)**

Yield 64%, colourless oil: $^1$H NMR (400 MHz, CDCl$_3$): (syn) δ 7.78 (d, $J = 8.0$ Hz, 2H), 7.37 – 7.20 (m, 8H), 7.11 (d, $J = 8.5$ Hz, 2H), 6.96 (t, $J = 8.0$ Hz, 1H), 6.81 (d, $J$
= 8.6 Hz, 2H), 6.66 (d, J = 6.5 Hz, 2H), 6.14 (s, 1H), 5.37 (d, J = 15.3 Hz, 1H), 4.81 (s, 1H), 4.38 – 4.27 (d, J = 15.3 Hz, 1H), 4.08 (td, J = 7.0, 2.2 Hz, 1H), 3.84 (s, 3H), 3.37 – 3.21 (m, 2H), 2.46 (s, 3H). 13C NMR (100 MHz, CDCl3): (syn) δ 196.85, 172.26, 160.94, 160.01, 144.47, 140.40, 137.07, 134.08, 131.31, 130.01, 129.54, 129.42, 128.98, 128.91, 128.20, 128.09, 127.82, 126.84, 124.85, 122.12, 120.69, 114.14, 64.66, 55.37, 45.95, 40.90, 37.05, 21.71, 1.02; ES-HRMS: Calcd for C34H31BrNO3 [M+H]+, 580.1481, Found 580.1478.

The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=85:15, flow rate:1.0 mL/min): tR 13.07min (minor), tR 23.90min (major), ee 86%

![Chemical structure](image)

**N-Benzyl-5-(1-(4-fluorophenyl)-3-oxo-3-(p-tolyl)propyl)-4-(4-methoxyphenyl)-1H-pyrrol-2(5H)-one (3p)**

Yield 90%, colourless oil; 1H NMR (400 MHz, CDCl3): (syn) δ 7.77 (t, J = 16.7 Hz, 2H), 7.30 (m, 5H), 7.26 – 7.21 (m, 2H), 7.18 (d, J = 8.8 Hz, 2H), 6.83 (d, J = 8.8 Hz, 2H), 6.76 (m, J = 8.6 Hz, 2H), 6.63 (m, 2H), 6.11 (s, 1H), 5.45 (d, J = 15.4 Hz, 1H), 4.84 (d, J = 2.2 Hz, 1H), 4.33 (d, J = 15.4 Hz, 1H), 4.07 (dt, J = 20.2, 10.1 Hz, 1H), 3.84 (s, 3H), 3.37 (m, 2H), 2.46 (s, 3H). 13C NMR (100 MHz, CDCl3): (syn) δ 197.12, 172.50, 160.87, 160.06, 144.44, 137.06, 134.16, 133.55, 129.64, 129.56, 129.42, 128.91, 128.10, 128.07, 127.76, 124.90, 120.37, 114.93, 114.72, 114.12, 64.25, 55.38, 46.15, 40.87, 38.24, 21.70; ES-HRMS: Calcd for C34H31FNO3 [M+H]+, 520.2282, Found 520.2283.
The enantiomeric excess was determined by HPLC analysis. (OD-H, hexane:IPA=92:8, flow rate:1.0 mL/min): $t_R$ 20.47 min (minor), $t_R$ 50.49 min (major), ee 87%

c NMR Spectra

$^1$H NMR and $^{13}$C NMR spectra of 3a
$^{1}H$ NMR and $^{13}C$ NMR spectra of 3b
$^{1}$H NMR and $^{13}$C NMR spectra of 3c
1H NMR and 13C NMR spectra of 3d
$^1$H NMR and $^{13}$C NMR spectra of 3e
$^1$H NMR and $^{13}$C NMR spectra of 3f
$^1$H NMR and $^{13}$C NMR spectra of 3g
$^1$H NMR and $^{13}$C NMR spectra of 3h

$^1$H NMR and $^{13}$C NMR spectra of 3i
$^1$H NMR and $^{13}$C NMR spectra of 3j
$^1$H NMR and $^{13}$C NMR spectra of 3k
$^1$H NMR and $^{13}$C NMR spectra of 3l

*Parameter* | *Value*
---|---
1 Solvent | CDCl$_3$
2 Temperature | 298.6 K
3 Number of Scans | 6
4 Pulse Width | 2 ms
5 Spectrometer Frequency | 400.13 MHz
6 Spectral Width | 8821.7 Hz
7 Lowest Frequency | -1953.4 Hz
8 Number | 32
$^1$H NMR and $^{13}$C NMR spectra of 3n
$^{1}$H NMR and $^{13}$C NMR spectra of 3o

$^{1}$H NMR and $^{13}$C NMR spectra of 3p
HSQC of $3p_1$
HMBC of 3p₁
d HPLC Spectra

3a (racemate)

---

3a (chiral synthesis)

---
3b (racemate)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Sample amount: 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with IS/IDs

Signal 1: DAD B, Sig=238,4 Ref=380,100

Peak RetTime Type Width Area Height Area
# [min] [min] [mm^2] [a.u.] [a.u.] [%]

1 11.510 std 0.6364 1.8496x4 957.3827 95.3923
2 16.698 std 1.1671 1.8996x4 293.8439 18.8977
3 19.368 std 1.4673 1.7576x4 270.0923 11.4130
4 21.228 BBA 2.8375 1.8482x4 284.2065 10.3290

3b (chiral synthesis)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Sample amount: 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with IS/IDs

Signal 1: DAD B, Sig=238,4 Ref=380,100

Peak RetTime Type Width Area Height Area
# [min] [min] [mm^2] [a.u.] [a.u.] [%]

1 11.432 PM 0.6212 1.6352x3 155.7905 5.2063
2 26.512 PM 2.4560 6.6189x4 408.2053 94.7937

34
3c (racemate)

```
+---------------------------------------------+----------+----------+----------+
|            Area Percent Report              |          |          |          |
+---------------------------------------------+----------+----------+----------+
| Sorted by:                                  | Signal   |          |          |
| Multiplier:                                 | 1.0000   |          |          |
| Dilution:                                   | 1.0000   |          |          |
| Sample Amount:                              | 1.0000 [μg/μl] (not used in calc.) |          |          |
| Use Multiplier & Dilution factor with ISTDs |          |          |          |
| Signal 1: DAD: B, Sise=20,4 Res=368,108    |          |          |          |
| Peak RetTime | Type | Width | Area   | Weight | Area   |           |           |
| [min]       | [min] | [mmHg]  | [micro] | [mmHg] | [micro] | [4]       | [4]       |
+---------------------------------------------+----------+----------+----------+----------+----------+----------+----------|
| 3 13.368 | TIC   | 2.2464   | 2.22958B | 119.84956  | 27.7245  | 4 24.306 | TIC   | 2.7999 | 1.69323B | 146.16570  | 32.0099  |
+---------------------------------------------+----------+----------+----------+----------+----------+----------+----------+
```

3c (chiral synthesis) (TLC)

```
+---------------------------------------------+----------+----------+----------+
|            Area Percent Report              |          |          |          |
+---------------------------------------------+----------+----------+----------+
| Sorted by:                                  | Signal   |          |          |
| Multiplier:                                 | 1.0000   |          |          |
| Dilution:                                   | 1.0000   |          |          |
| Sample Amount:                              | 1.0000 [μg/μl] (not used in calc.) |          |          |
| Use Multiplier & Dilution factor with ISTDs |          |          |          |
| Signal 1: DAD: B, Sise=20,4 Res=368,108    |          |          |          |
| Peak RetTime | Type | Width | Area   | Weight | Area   |           |           |
| [min]       | [min] | [mmHg]  | [micro] | [mmHg] | [micro] | [4]       | [4]       |
+---------------------------------------------+----------+----------+----------+----------+----------+----------+----------|
| 1 12.873 | TIC   | 1.9706   | 0.99068 | 57.13 | 8.3323  | 2 27.854 | TIC   | 2.9717 | 1.6035E9 | 613.12599 | 94.3214  |
+---------------------------------------------+----------+----------+----------+----------+----------+----------+----------+
```
3d (racemate)

![Chemical structure and chromatogram]

Area Percent Report

Signal 1: DAD 1 B, Spline=2,0 A Ref=368,100

<table>
<thead>
<tr>
<th>#</th>
<th>Ret Time</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>11.709</td>
<td>0.255</td>
<td>306.50</td>
<td>368.100</td>
<td>32.604</td>
</tr>
<tr>
<td>2</td>
<td>17.797</td>
<td>0.370</td>
<td>396.44</td>
<td>368.100</td>
<td>19.809</td>
</tr>
<tr>
<td>3</td>
<td>21.550</td>
<td>0.317</td>
<td>384.83</td>
<td>368.100</td>
<td>19.198</td>
</tr>
<tr>
<td>4</td>
<td>31.116</td>
<td>1.917</td>
<td>653.78</td>
<td>368.100</td>
<td>18.907</td>
</tr>
</tbody>
</table>

3d (chiral synthesis)

![Chemical structure and chromatogram]

Area Percent Report

Signal 1: DAD 1 B, Spline=2,0 A Ref=488,100

<table>
<thead>
<tr>
<th>#</th>
<th>Ret Time</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>11.390</td>
<td>0.952</td>
<td>432.87</td>
<td>488.100</td>
<td>94.159</td>
</tr>
<tr>
<td>2</td>
<td>38.846</td>
<td>2.862</td>
<td>449.36</td>
<td>488.100</td>
<td>5.841</td>
</tr>
</tbody>
</table>
3e (racemate) (TLC)

3e (chiral synthesis) (TLC)
3f (racemate)

Area Percent Report

<table>
<thead>
<tr>
<th>Signal</th>
<th>Area</th>
<th>Width</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1211.886</td>
<td>4.312</td>
<td>568.356</td>
<td>565.856</td>
</tr>
<tr>
<td>2</td>
<td>940.845</td>
<td>4.893</td>
<td>470.346</td>
<td>490.347</td>
</tr>
<tr>
<td>3</td>
<td>73.183</td>
<td>5.729</td>
<td>10.120</td>
<td>10.120</td>
</tr>
<tr>
<td>4</td>
<td>79.554</td>
<td>5.189</td>
<td>10.120</td>
<td>10.120</td>
</tr>
</tbody>
</table>

3f (chiral synthesis)

Area Percent Report

<table>
<thead>
<tr>
<th>Signal</th>
<th>Area</th>
<th>Width</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>471.192</td>
<td>5.954</td>
<td>568.356</td>
<td>565.856</td>
</tr>
<tr>
<td>2</td>
<td>81.217</td>
<td>6.572</td>
<td>10.120</td>
<td>10.120</td>
</tr>
<tr>
<td>3</td>
<td>104.442</td>
<td>5.387</td>
<td>10.120</td>
<td>10.120</td>
</tr>
<tr>
<td>4</td>
<td>104.442</td>
<td>5.387</td>
<td>10.120</td>
<td>10.120</td>
</tr>
</tbody>
</table>
3g (racemate)

3g (chiral synthesis)
3h (racemate)

3h (chiral synthesis) (TLC)

---

**Area Percent Report**

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Sample Amount: 1.00000 [g/mL] (not used in calc.)

Use Multiplier & Dilution factor with [TLC]

---

**Signal 1: [TLC B, Sig=210, x Ref=350,100]**

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>30.518</td>
<td>EV</td>
<td>0.782</td>
<td>1.317</td>
<td>84449</td>
<td>27.304</td>
</tr>
<tr>
<td>2</td>
<td>33.976</td>
<td>VB</td>
<td>1.040</td>
<td>1.807</td>
<td>55887</td>
<td>22.027</td>
</tr>
<tr>
<td>3</td>
<td>36.766</td>
<td>EV</td>
<td>1.316</td>
<td>1.876</td>
<td>54734</td>
<td>22.866</td>
</tr>
<tr>
<td>4</td>
<td>43.171</td>
<td>EB</td>
<td>1.810</td>
<td>1.433</td>
<td>52657</td>
<td>28.481</td>
</tr>
</tbody>
</table>

---

**Signal 1: [TLC B, Sig=210, x Ref=350,100]**

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.879</td>
<td>EB</td>
<td>1.273</td>
<td>0.843</td>
<td>1.319</td>
<td>35.823</td>
</tr>
<tr>
<td>2</td>
<td>21.187</td>
<td>EB</td>
<td>1.508</td>
<td>1.385</td>
<td>1.466</td>
<td>32.458</td>
</tr>
</tbody>
</table>

---

40
3i (racemate)

![Chromatogram](image1)

Area Percent Report

<table>
<thead>
<tr>
<th>#</th>
<th>Run Time</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>30.274</td>
<td>EV</td>
<td>1.188</td>
<td>1.188</td>
<td>599.763</td>
<td>52.187</td>
</tr>
<tr>
<td>2</td>
<td>15.498</td>
<td>BV</td>
<td>1.416</td>
<td>1.416</td>
<td>271.465</td>
<td>17.938</td>
</tr>
<tr>
<td>3</td>
<td>15.838</td>
<td>BA</td>
<td>1.657</td>
<td>1.657</td>
<td>277.526</td>
<td>19.376</td>
</tr>
<tr>
<td>4</td>
<td>21.211</td>
<td>BA</td>
<td>2.209</td>
<td>2.209</td>
<td>350.593</td>
<td>31.568</td>
</tr>
</tbody>
</table>

3i (chiral synthesis) (TLC)

![Chromatogram](image2)

Area Percent Report

<table>
<thead>
<tr>
<th>#</th>
<th>Run Time</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.351</td>
<td>MN</td>
<td>1.144</td>
<td>1.144</td>
<td>517.405</td>
<td>7.648</td>
</tr>
<tr>
<td>2</td>
<td>20.412</td>
<td>MN</td>
<td>2.109</td>
<td>2.109</td>
<td>2983.065</td>
<td>82.195</td>
</tr>
</tbody>
</table>
3j (racemate)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Sample Amount: 1.000000 [mg/mL] (not used in calc.)
See Multiplier & Dilution factor with A UD

Signal 1: DAD, S=210.4 Ref=300.1000

Peak RetTime Type Width Area Height Area %
-----------|--|----------|--|----------|--|
1 26.771 R 1.2734 5.84328e4 828.99569 27.2569
2 36.846 R 1.6728 4.47616e4 623.12999 24.1853
3 43.317 R 1.7364 1.89524e4 167.16836 20.1389
4 69.962 R 4.8209 5.14833e4 142.15873 27.7484

3j (chiral synthesis)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Sample Amount: 1.000000 [mg/mL] (not used in calc.)
See Multiplier & Dilution factor with A UD

Signal 1: DAD, S=210.4 Ref=300.1000

Peak RetTime Type Width Area Height Area %
-----------|--|----------|--|----------|--|
1 25.876 R 2.9809 4.10616e4 36.4813 39.3789
2 57.033 R 4.8209 4.75732e4 136.8479 91.6222
3k (racemate)

---

3k (chiral synthesis) (TLC)
3l (racemate) (TLC)

---

Area Percent Report

---

Sorted By:  Signal
Multiplier:  1.0000
Dilution:  1.0000
Sample Amount:  1.00000 [mg/mL] (not used in calc.)

the Multiplier & Dilution factor with 107th

Signal 1: DADI B, Sig=208.4 Ref=360,100

Peak RetTime Type Width Area Height Area
# [min] [mm] [mm] [mm] [mm] [mm]

1 12.775 RB 1.2417 1.4023465 1681.9932 49.6332
2 28.915 VBA 2.3518 1.4653865 775.9552 50.3668

3l (chiral synthesis) (TLC)

---

Area Percent Report

---

Sorted By:  Signal
Multiplier:  1.0000
Dilution:  1.0000
Sample Amount:  1.00000 [mg/mL] (not used in calc.)

the Multiplier & Dilution factor with 107th

Signal 1: DADI B, Sig=208.4 Ref=360,100

Peak RetTime Type Width Area Height Area
# [min] [mm] [mm] [mm] [mm] [mm]

1 12.208 HR 1.4396 1.6169364 1096.6065 7.6801
2 25.093 HR 3.7005 1.3535865 556.8980 87.3805
3m (racemate)

![Graph of racemate](image)

**Area Percent Report**

<table>
<thead>
<tr>
<th>Sorted by</th>
<th>Signal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Multiplier</td>
<td>1.0000</td>
</tr>
<tr>
<td>Dilution</td>
<td>1.0000</td>
</tr>
<tr>
<td>Sample amount</td>
<td>1.00000 [ng/ul] (not used in calcs.)</td>
</tr>
</tbody>
</table>

Use Multiplier & Dilution Factor with ISTDs

Signal | Data B, Sig=20, Data=1000

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>[min]</td>
<td>[min]</td>
<td>[amu]</td>
<td>[amu]</td>
<td>[amu]</td>
</tr>
<tr>
<td>1</td>
<td>10.470</td>
<td>0.0070</td>
<td>1050.13788</td>
<td>100.0874</td>
</tr>
<tr>
<td>2</td>
<td>11.415</td>
<td>0.0096</td>
<td>1244.03769</td>
<td>79.5952</td>
</tr>
<tr>
<td>3</td>
<td>11.915</td>
<td>0.0090</td>
<td>4516.91147</td>
<td>68.74288</td>
</tr>
<tr>
<td>4</td>
<td>20.155</td>
<td>1.1172</td>
<td>3691.12982</td>
<td>61.7926</td>
</tr>
</tbody>
</table>

3m (chiral synthesis)

![Graph of chiral synthesis](image)

**Area Percent Report**

<table>
<thead>
<tr>
<th>Sorted by</th>
<th>Signal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Multiplier</td>
<td>1.0000</td>
</tr>
<tr>
<td>Dilution</td>
<td>1.0000</td>
</tr>
<tr>
<td>Sample amount</td>
<td>1.00000 [ng/ul] (not used in calcs.)</td>
</tr>
</tbody>
</table>

Use Multiplier & Dilution Factor with ISTDs

Signal | Data B, Sig=20, Data=1000

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>[min]</td>
<td>[min]</td>
<td>[amu]</td>
<td>[amu]</td>
<td>[amu]</td>
</tr>
<tr>
<td>1</td>
<td>10.189</td>
<td>0.00155</td>
<td>5528.80286</td>
<td>61.31767</td>
</tr>
<tr>
<td>2</td>
<td>20.165</td>
<td>1.7384</td>
<td>1.027846</td>
<td>808.28459</td>
</tr>
</tbody>
</table>
3n (racemate)

---

**Area Percent Report**

**Sorted By**: Signal  
**Multiplier**: 1.0000  
**Dilution**: 1.0000  
**Sample Amount**: 1.000000 mg/mL  
*Use Multiplier & Dilution Factor with 100%*

**Signal 1**:  
<table>
<thead>
<tr>
<th>Retention Time</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.639 min</td>
<td>0.625</td>
<td>7.996</td>
<td>5.301</td>
<td>12.635</td>
</tr>
<tr>
<td>10.738 min</td>
<td>0.846</td>
<td>1.313</td>
<td>5.709</td>
<td>11.190</td>
</tr>
<tr>
<td>15.928 min</td>
<td>1.061</td>
<td>1.155</td>
<td>5.881</td>
<td>17.372</td>
</tr>
<tr>
<td>18.950 min</td>
<td>1.392</td>
<td>0.318</td>
<td>7.134</td>
<td>10.573</td>
</tr>
</tbody>
</table>

3n (chiral synthesis) (TLC)

---

**Area Percent Report**

**Sorted By**: Signal  
**Multiplier**: 1.0000  
**Dilution**: 1.0000  
**Sample Amount**: 1.000000 mg/mL  
*Use Multiplier & Dilution Factor with 100%*

**Signal 1**:  
<table>
<thead>
<tr>
<th>Retention Time</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.885 min</td>
<td>0.797</td>
<td>1.304</td>
<td>5.232</td>
<td>7.468</td>
</tr>
<tr>
<td>16.149 min</td>
<td>1.671</td>
<td>1.290</td>
<td>5.260</td>
<td>8.394</td>
</tr>
</tbody>
</table>

---
4-3o (racemate) (TLC)

```
<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12.787</td>
<td>1.690</td>
<td>1.920</td>
<td>1.72925</td>
<td>55.429</td>
</tr>
<tr>
<td>2</td>
<td>13.372</td>
<td>1.690</td>
<td>1.920</td>
<td>1.10316</td>
<td>48.571</td>
</tr>
</tbody>
</table>
```

4-3o (chiral synthesis) (TLC)

```
<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>11.977</td>
<td>0.895</td>
<td>1.20564</td>
<td>218.0568</td>
<td>95.984</td>
</tr>
<tr>
<td>2</td>
<td>21.002</td>
<td>0.673</td>
<td>1.175120</td>
<td>124.7652</td>
<td>83.444</td>
</tr>
</tbody>
</table>
```
3p (racemate)

![Graph showing a chromatogram with labeled peaks and areas.]

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Sample Amount: 1.00000 [ug/ul] (not used in calc.)
Use Multiplier & Dilution Factor with Split

Signal 1: DAD1 spectra=210,4 ref=58U,1000

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20.645</td>
<td>2.5669</td>
<td>1.9561×10⁴</td>
<td>94.27399</td>
<td>33.1224</td>
</tr>
<tr>
<td>2</td>
<td>27.935</td>
<td>3.5375</td>
<td>1.5379×10³</td>
<td>54.99404</td>
<td>17.47955</td>
</tr>
<tr>
<td>3</td>
<td>41.602</td>
<td>3.7936</td>
<td>1.8008×10⁴</td>
<td>37.48130</td>
<td>13.18445</td>
</tr>
<tr>
<td>4</td>
<td>56.637</td>
<td>6.6889</td>
<td>2.7151×10³</td>
<td>56.65451</td>
<td>35.16405</td>
</tr>
</tbody>
</table>

3p (chiral synthesis) (TLC)

![Graph showing a chromatogram with labeled peaks and areas.]

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Sample Amount: 1.00000 [ug/ul] (not used in calc.)
Use Multiplier & Dilution Factor with Split

Signal 1: DAD1 spectra=210,4 ref=58U,1000

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20.475</td>
<td>3.4647</td>
<td>4.8850×10⁴</td>
<td>23.33111</td>
<td>6.7256</td>
</tr>
<tr>
<td>2</td>
<td>58.490</td>
<td>10.6080</td>
<td>6.7263×10⁴</td>
<td>105.95723</td>
<td>35.27544</td>
</tr>
</tbody>
</table>

48