N-Heterocyclic carbene-catalyzed [3 + 3] cyclocondensation of bromoenals with aldimines: Highly enantioselective synthesis of dihydropyridinones

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Part I General information

Unless otherwise indicated, all reactions were carried out under N₂ atmosphere at room temperature with magnetic stirring. Anhydrous THF and toluene were distilled from sodium and benzophenone. Anhydrous CH₂Cl₂ was distilled from CaH₂. Chiral triazolium salts 4-5¹, aldimines² and α-bromoenals³ were prepared according to literatures. Column chromatography was performed on silica gel 200–300 mesh. All ¹H NMR (300 and 400 MHz), ¹³C NMR (75 and 100 MHz) spectra were recorded on a Bruker-DMX 300 and Bruker Avance 400 spectrometer in CDCl₃, with tetramethylsilane as an internal standard and reported in parts per million (ppm, δ). ¹H NMR Spectroscopy splitting patterns were designated as singlet (s), doublet (d), triplet (t), quartet (q). Splitting patterns that could not be interpreted or easily visualized were designated as multiplet (m) or broad (br). Infrared spectra were recorded on a JASCO FT/IR-480 spectrophotometer and reported as wave number (cm⁻¹). Optical rotations were measured on Perkin Elmer/Model-343 digital polarimeter operating at the sodium D line with a 100 mm path cell, and are reported as follows: [α]₀Τ (concentration (g/100 mL), solvent).
Part II Experimental part

1. NHC catalyzed [3+3] annulation of α-bromoenal 1 and aldimine 2

To the solution of α-bromoenal 1 (0.6 mmol, 3 equiv.), NHC precursor 4 (0.04 mmol, 16.8 mg, 0.2 equiv.), and Et₃N (0.6 mmol, 60.6 mg, 3.0 equiv.) in THF (1 mL), was slowly added aldimine 2 (0.2 mmol, 1 equiv., 2 mL in THF) via a syringe pump in 6 h. The reaction mixture was stirred at room temperature until the full consumption of the aldimine 2 (typically, 12 h). The reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 40:1-10:1) to furnish the corresponding product 3.

Racemic samples for the chiral phase HPLC analysis were prepared using triazolium S1 as the NHC pre-catalyst under the same conditions.

(R)-5-benzyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-281B)

59.0 mg, 71% yield. White solid, mp. 148-149 °C; Rᵣ = 0.2 (petroleum ether/ethyl acetate 5:1); [α]$_{D}^{25}$ -148 (c 0.1, CH₂Cl₂); HPLC analysis: 95% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 80:20, 1.0 mL /min,
254 nm, 11.3 min (minor), 23.3 min (major)]; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 7.87 (d, \(J = 8.3\) Hz, 2H), 7.27 (d, \(J = 8.3\) Hz, 2H), 7.20 (d, \(J = 6.8\) Hz, 2H), 7.18 (t, \(J = 6.8\) Hz, 1H), 7.08-6.98 (m, 6H), 6.71 (d, \(J = 7.4\) Hz, 2H), 3.38 (d, \(J = 15.2\) Hz, 1H), 3.26 (dd, \(J = 7.7, 2.6\) Hz, 1H), 3.10 (d, \(J = 15.2\) Hz, 1H), 2.76 (dd, \(J = 15.6, 7.7\) Hz, 1H), 2.46-2.39 (m, 4H). \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 167.0, 163.4, 160.9, 145.3, 139.6, 138.3, 135.1, 129.5, 128.9, 128.8, 128.8, 128.7, 127.4, 127.0, 126.8, 124.6, 121.0, 41.1, 40.1, 38.9, 21.7. IR (KBr) 2920, 1721, 1266, 1173; HRMS (ESI) calcd for C\(_{25}\)H\(_{23}\)NSO\(_3\)Na [M+Na]\(^+\) 440.12909, found 440.12851.

(GZH-296A)

60.4 mg, 69% yield. Colorless oil; \(R_f = 0.2\) (petroleum ether/ethyl acetate 5:1); [\(\alpha\)]\(^D\) = -72 (c 0.1, CH\(_2\)Cl\(_2\)); HPLC analysis: 98% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 22.5 min (minor), 35.7 min (major)]; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 7.94 (d, \(J = 8.3\) Hz, 2H), 7.37-7.24 (m, 5H), 7.12 (d, \(J = 6.8\) Hz, 2H), 7.07 (s, 1H), 6.81-6.67 (m, 4H), 3.47 (d, \(J = 15.1\) Hz, 1H), 3.33 (dd, \(J = 7.6, 2.7\) Hz, 1H), 3.18 (d, \(J = 15.1\) Hz, 1H), 2.83 (dd, \(J = 15.6, 7.6\) Hz, 1H), 2.54-2.41 (m, 4H). \(^{13}\)C NMR (100 MHz) \(\delta\) 166.7, 149.7, 145.6, 141.9, 138.2, 135.2, 135.0, 130.4, 130.0, 129.9, 129.4, 129.0, 128.8, 128.7, 127.9, 127.8, 127.3, 127.2, 127.0, 125.9, 125.6, 123.7, 121.8, 115.8, 41.4, 39.6, 39.2, 21.9. IR (KBr) 2919, 1732, 1339, 1196; HRMS (ESI) calcd for C\(_{25}\)H\(_{22}\)NSO\(_3\)FNa [M+Na]\(^+\) 448.11966, found 458.11914.
(R)-5-benzyl-4-(4-chlorophenyl)-1-tosyl-3,4-dihydropyridin-2(1H)-one

(GZH-297B)

62.4 mg, 69% yield. Colorless oil. Rf = 0.2 (petroleum ether/ethyl acetate 5:1); \([\alpha]_D^{25} -81 (c 0.1, \text{CH}_2\text{Cl}_2);\) HPLC analysis: 97% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 25.4 min (minor), 37.3 min (major)]; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta 7.93 (d, J = 8.3 \text{ Hz}, 2\text{H}), 7.37-7.24 (m, 5\text{H}), 7.17-6.97 (m, 5\text{H}), 6.71 (d, J = 8.4 \text{ Hz}, 2\text{H}), 3.47 (d, J = 15.2 \text{ Hz}, 1\text{H}), 3.32 (dd, \(J = 7.6, 2.6 \text{ Hz}, 1\text{H}), 3.17 (d, J = 15.2 \text{ Hz}, 1\text{H}), 2.83 (dd, J = 12.5, 15.6 \text{ Hz}, 1\text{H}), 2.57-2.36 (m, 4\text{H}).\) \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta 166.7, 145.5, 138.0, 135.0, 133.2, 129.5, 129.0, 128.3, 126.9, 124.3, 121.4, 41.3, 39.5, 39.0, 21.7.\) IR (KBr) 2919, 1722, 1396, 1196; HRMS (ESI) calcd for C\(_{25}\)H\(_{22}\)NSO\(_3\)ClNa \([\text{M+Na}]^+\) 474.09011, found 474.08946.

(R)-5-benzyl-4-(4-bromophenyl)-1-tosyl-3,4-dihydropyridin-2(1H)-one

(GZH-297A)

70.3 mg, 78% yield. Colorless oil. Rf = 0.2 (petroleum ether/ethyl acetate 5:1); \([\alpha]_D^{25} -87 (c 0.1, \text{CH}_2\text{Cl}_2);\) HPLC analysis: 97% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 26.9 min (minor), 39.3 min (major)]; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta 7.93 (d, J = 8.3 \text{ Hz}, 2\text{H}), 7.37-7.23 (m, 5\text{H}), 7.18 (d, J = 8.3 \text{ Hz}, 2\text{H}), 7.12-7.07 (m, 3\text{H}), 6.65 (d, J = 8.3 \text{ Hz}, 2\text{H}), 3.47 (d, J = 15.2 \text{ Hz}, 1\text{H}), 3.32 (dd, J = 7.6, 2.6 \text{ Hz}, 1\text{H}), 3.17 (d, J = 15.2 \text{ Hz}, 1\text{H}), 2.83 (dd, J = 12.5, 15.6 \text{ Hz}, 1\text{H}), 2.57-2.36 (m, 4\text{H}).\) \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta 166.7, 145.5, 138.0, 135.0, 133.2, 129.5, 129.0, 128.3, 126.9, 124.3, 121.4, 41.3, 39.5, 39.0, 21.7.\) IR (KBr) 2919, 1722, 1396, 1196; HRMS (ESI) calcd for C\(_{25}\)H\(_{22}\)NSO\(_3\)ClNa \([\text{M+Na}]^+\) 474.09011, found 474.08946.
15.2 Hz, 1H), 3.30 (dd, J = 7.6, 2.6 Hz, 1H), 3.17 (d, J = 14.3 Hz, 1H), 2.82 (dd, J = 15.2, 7.6 Hz, 1H), 2.56-2.38 (m, 4H). $^1$H NMR (75 MHz, CDCl$_3$) δ 166.7, 145.5, 138.6, 138.0, 135.0, 132.0, 129.5, 128.8, 128.7, 126.9, 124.2, 121.4, 121.2, 40.9, 39.5, 39.0, 21.7. IR (KBr) 2919, 1722, 1361, 1173; HRMS (ESI) calcd for C$_{25}$H$_{22}$N$_3$O$_3$BrNa [M+Na]$^+$ 518.03960, found 513.03884.

(R)-5-benzyl-4-(3-chlorophenyl)-1-tosyl-3,4-dihydropyridin-2(1H)-one
(GZH-391A)

58.0 mg, 64% yield. White solid, mp. 92-93 °C. R$_f$ = 0.2 (petroleum ether/ethyl acetate 5:1); [α]$_D$$^{35}$ -84 (c 0.1, CH$_2$Cl$_2$); HPLC analysis: 97% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH/MeOH = 90:6:4, 1.0 mL/min, 254 nm, 16.8 min (minor), 19.4 min (major)]; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.93 (d, J = 8.2 Hz, 2H), 7.34 (d, J = 8.2 Hz, 2H), 7.29 (t, J = 7.2 Hz, 2H), 7.23 (d, J = 5.6 Hz, 1H), 7.11 (d, J = 7.0 Hz, 2H), 7.06 (s, 1H), 6.80 – 6.69 (m, 3H), 3.45 (d, J = 15.1 Hz, 1H), 3.32 (dd, J = 7.6, 2.7 Hz, 1H), 3.16 (d, J = 15.1 Hz, 1H), 2.81 (dd, J = 15.6, 7.6 Hz, 1H), 2.55–2.36 (m, 4H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 166.7, 145.6, 141.9, 138.2 135.0, 130.4, 130.0, 129.0, 129.0, 128.9, 128.7, 127.9, 127.2, 125.9, 115.9, 41.0, 40.0, 39.2, 22.0. IR (KBr) 2919, 1722, 1362, 1173; HRMS (ESI) calcd for C$_{25}$H$_{22}$N$_3$O$_3$ClNa [M+Na]$^+$ 474.09011, found 474.08953.

(S)-5-benzyl-4-(2-methoxyphenyl)-1-tosyl-3,4-dihydropyridin-2(1H)-one
(GZH-295A)
72.7 mg, 81% yield. White solid, mp. 95-96 °C. R_f = 0.3 (petroleum ether/ethyl acetate 5:1); [α]_D^25 = -77 (c 0.1, CH₂Cl₂); HPLC analysis: 97% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 17.8 min (minor), 29.3 min (major)]; ^1^H NMR (300 MHz, CDCl₃) δ 7.86 (d, J = 8.3 Hz, 2H), 7.26-7.13 (m, 5H), 7.09-7.04 (m, 3H), 6.99 (s, 1H), 6.70 (d, J = 8.2 Hz, 2H), 6.62 (dd, J = 7.5, 1.6 Hz, 1H), 6.55 (t, J = 7.3 Hz, 1H), 3.65 (dd, J = 8.2, 2.2 Hz, 1H), 3.54 (s, 3H), 3.33 (t, J = 15.0 Hz, 1H), 3.07 (d, J = 15.0 Hz, 1H), 2.68 (dd, J = 15.9, 8.2 Hz, 1H), 2.49 (dd, J = 15.9, 2.4 Hz, 1H), 2.37 (s, 3H). ^13^C NMR (75 MHz, CDCl₃) δ 167.6, 157.1, 145.1, 138.6, 135.4, 129.4, 128.8, 128.6, 127.8, 127.2, 126.7, 123.2, 121.4, 120.4, 110.8, 54.9, 39.1, 38.9, 34.8, 21.7. IR (KBr) 2920, 1716, 1361, 1174; HRMS (ESI) calcd for C_{26}H_{25}NSO_{4}Na [M+Na]^⁺ 474.13965, found 470.13913.

(S)-5-benzyl-4-(2-bromo-5-methoxyphenyl)-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-295C)

81.0 mg, 77% yield. White crystal, mp. 115-116 °C. R_f = 0.3 (petroleum ether/ethyl acetate 5:1); [α]_D^25 = -115 (c 0.1, CH₂Cl₂); HPLC analysis: 95% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 14.1 min (minor), 23.5 min (major)]; ^1^H NMR (300 MHz, CDCl₃) δ 7.94 (d, J = 8.4 Hz, 2H), 7.38 (d, J = 8.7 Hz, 1H), 7.35-7.19 (m, 5H), 7.18 (s, 1H), 7.13 (d, J = 6.8 Hz, 2H), 6.60 (dd, J = 8.8, 3.0 Hz, 1H), 6.48 (d, J = 3.0 Hz, 1H), 3.87 (dd, J = 8.0, 2.5 Hz, 1H), 3.63 (s, 3H), 3.43 (d, J = 4.8 Hz, 1H), 3.18 (d, J = 14.8 Hz, 1H), 2.79 (dd, J = 15.9, 8.1 Hz, 1H), 2.58 (dd, J = 15.9, 2.5 Hz, 1H), 2.43 (s, 3H). ^13^C NMR (75 MHz, CDCl₃) δ 166.7, 159.4, 145.2, 138.7, 137.9, 135.2, 134.0, 129.6, 128.9, 128.8, 128.7, 126.9, 123.4, 122.1, 114.5, 114.4, 114.0, 55.3, 39.5, 39.2, 39.1, 21.7. IR (KBr) 2919, 1722, 1362, 1174; HRMS (ESI) calcd for C_{26}H_{24}NSO_{4}BrNa [M+Na]^⁺ 550.04866, found 550.04738.
(R)-methyl 4-(5-benzyl-2-oxo-1-tosyl-1,2,3,4-tetrahydropyridin-4-yl)benzoate (GZH-392A)

27.2 mg, 57% yield. Colorless oil. \( R_f = 0.3 \) (petroleum ether/ethyl acetate 5:1); [\( \alpha \)]\textsubscript{D}\textsuperscript{-94} (c 0.1, CH\textsubscript{2}Cl\textsubscript{2}); HPLC analysis: 92% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH/MeOH = 90:6:4, 1.0 mL /min, 254 nm, 25.2 min (minor), 31.9 min (major)]; \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 7.94 (d, \( J = 8.0 \) Hz, 2H), 7.72 (d, \( J = 8.0 \) Hz, 1H), 7.34 (d, \( J = 8.0 \) Hz, 1H), 7.32-7.26 (m, 3H), 7.13 – 7.11 (m, 2H), 6.82 (d, \( J = 8.0 \) Hz, 1H), 3.92 (s, 3H), 3.50-3.39 (m, 2H), 3.17 (d, \( J = 15.1 \) Hz, 1H), 2.87 (dd, \( J = 15.1, 7.7 \) Hz, 1H), 2.52-2.48 (m, 4H). \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \( \delta \) 166.6, 166.5, 145.6, 144.7, 137.9, 134.9, 130.2, 129.6, 129.3, 128.8, 127.0, 127.0, 124.0, 121.6, 52.2, 40.8, 40.0, 39.1, 21.7. IR (KBr) 2920, 1722, 1361, 751; HRMS (ESI) calcd for C\textsubscript{27}H\textsubscript{25}NSO\textsubscript{5}Na [M+Na]\(^+\) 498.13456, found 498.13464.

(R)-5-benzyl-4-(4-(pyrrolidine-1-carbonyl)phenyl)-1-tosyl-3,4-dihydropyridin-2(1\(H\))-one (GZH-395C)

71.8 mg, 70% yield. White solid, mp. 72-74 °C. \( R_f = 0.2 \) (petroleum ether/ethyl acetate 1:1); [\( \alpha \)]\textsubscript{D}\textsuperscript{-105} (c 0.1, CH\textsubscript{2}Cl\textsubscript{2}); HPLC analysis: 99% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH/MeOH = 70:20:10, 1.0 mL /min, 254 nm, 15.2 min (minor), 23.9 min (major)]; \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \( \delta \) 169.1, 166.8, 145.4, 141.5, 138.1, 136.4, 135.0, 129.5, 128.8, 128.7, 127.8, 126.9,
126.9, 124.0, 121.3, 49.6, 46.2, 40.8, 40.0, 38.9, 26.4, 24.4, 21.8. IR (KBr) 2920, 1716, 1623, 1173; HRMS (ESI) calcd for C\textsubscript{30}H\textsubscript{30}N\textsubscript{2}SO\textsubscript{4}Na \[M+Na\]^+ 537.18185, found 537.18060.

\[(\mathit{R})\text{-}4\text{-(5-benzyl-2-oxo-1-tosyl-1,2,3,4-tetrahydropyridin-4-yl)benzonitrile (GZH-395A)}\]

69.1 mg, 78\% yield. White solid, mp. 56-57 °C. \(R_f = 0.3\) (petroleum ether/ethyl acetate 5:1); \([\alpha]_D^{25} -94\) (c 0.1, CH\textsubscript{2}Cl\textsubscript{2}); HPLC analysis: 91\% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH/MeOH = 90:6:4, 1.0 mL /min, 254 nm, 37.3 min (minor), 55.5 min (major)]; \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta 7.94\) (d, \(J = 8.2\) Hz, 1H), 7.38-7.35 (m, 3H), 7.31-7.22 (m, 4H), 7.15 (s, 1H), 7.10 (d, \(J = 7.1\) Hz, 1H), 6.89 (d, \(J = 8.1\) Hz, 1H), 3.49 (d, \(J = 15.2\) Hz, 1H), 3.40 (dd, \(J = 7.6, 2.4\) Hz, 1H), 3.18 (d, \(J = 15.2\) Hz, 1H), 2.88 (dd, \(J = 15.7, 7.6\) Hz, 1H), 2.54-2.43 (m, 4H). \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta 166.3, 145.7, 145.1, 137.6, 134.9, 132.7, 129.6, 128.8, 128.7, 127.8, 127.1, 123.3, 121.9, 118.4, 111.4, 40.6, 40.1, 39.1, 21.8. IR (KBr) 2920, 2850, 2228, 1717, 1633, 1173; HRMS (ESI) calcd for C\textsubscript{26}H\textsubscript{22}N\textsubscript{2}SO\textsubscript{3}Na \[M+Na\]^+ 465.12433, found 465.12404.

\[(\mathit{S})\text{-}5\text{benzyl-4-propyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-296C)}\]

16.7 mg, 22\% yield. Colorless oil. \(R_f = 0.6\) (petroleum ether/ethyl acetate 5:1); \([\alpha]_D^{25} -6\) (c 0.1, CH\textsubscript{2}Cl\textsubscript{2}); HPLC analysis: 90\% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 19.7 min (minor), 21.7 min (major)]; \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta 7.89\) (d, \(J = 8.0\) Hz, 2H), 7.33-7.30 (m,
4H), 7.24-7.21 (m, 3H), 6.83 (s, 1H), 3.50 (d, \(J = 15.2\) Hz, 1H), 3.34 (d, \(J = 15.2\) Hz, 1H), 2.50-2.43 (m, 4H), 2.32 (dd, \(J = 15.5, 2.0\) Hz, 1H), 2.11 (m, 1H), 1.18-1.13 (m, 2H), 1.00-0.98 (m, 2H), 0.72 (t, \(J = 6.3\) Hz, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 168.3, 145.1, 138.5, 135.3, 129.3, 128.7, 128.7, 128.6, 127.0, 126.8, 119.9, 39.4, 38.2, 34.1, 33.4, 21.7, 19.4, 13.9. IR (KBr) 2922, 1722, 1362, 1174; HRMS (ESI) calcd for C\(_{22}\)H\(_{25}\)NSO\(_3\)Na [M+Na\(^+\)] 406.14474, found 406.14450.

\(\text{(R)-5-methyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-292A)}\)

48.1 mg, 71% yield. Colorless oil. \(R_f = 0.2\) (petroleum ether/ethyl acetate 10:1); [\(\alpha\)\(_D\)]\(^{25}\) -143 (c 0.1, CH\(_2\)Cl\(_2\)); HPLC analysis: 97% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 17.4 min (minor), 45.7 min (major)]; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 7.93 (d, \(J = 8.4\) Hz, 2H), 7.33 (s, 1H), 6.91-6.80 (m, 2H), 3.39 (d, \(J = 7.6, 3.5\) Hz, 1H), 2.96 (dd, \(J = 15.6, 7.6\) Hz, 1H), 2.58 (dd, \(J = 15.6, 3.5\) Hz, 1H), 2.45 (s, 3H), 1.74 (d, \(J = 1.2\) Hz, 3H). \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 167.1, 145.2, 139.7, 135.2, 129.4, 128.8, 128.7, 127.3, 126.9, 121.0, 119.6, 42.9, 40.8, 21.7, 19.0. IR (KBr) 2920, 1716, 1355, 1170; HRMS (ESI) calcd for C\(_{19}\)H\(_{19}\)NSO\(_3\)Na [M+Na\(^+\)] 364.09779, found 364.09732.

\(\text{(R)-5-ethyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-292C)}\)

57.1 mg, 80% yield. Colorless oil. \(R_f = 0.2\) (petroleum ether/ethyl acetate 5:1); [\(\alpha\)\(_D\)]\(^{25}\) -164 (c 0.1, CH\(_2\)Cl\(_2\)); HPLC analysis: 98% ee [Daicel CHIRALPAK OD-H column,
20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL/min, 254 nm, 14.5 min (minor), 54.1 min (major)]; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 7.93 (d, $J = 8.3$ Hz, 2H), 7.33 (d, $J = 8.3$ Hz, 1H), 7.19-7.00 (m, 3H), 6.95 (s, 1H), 6.84 (d, $J = 7.0$ Hz, 2H), 3.46 (dd, $J = 7.6$, 2.9 Hz, 1H), 2.94 (dd, $J = 15.5$, 7.6 Hz, 1H), 2.55 (dd, $J = 15.5$, 2.9 Hz, 1H), 2.45 (s, 2H), 2.19-1.94 (m, 2H), 1.04 (t, $J = 7.4$ Hz, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 167.2, 145.1, 140.0, 135.2, 129.4, 128.8, 128.7, 127.3, 126.9, 126.7, 119.0, 41.4, 41.2, 26.0, 21.7, 12.4. IR (KBr) 2919, 1716, 1362, 1171; HRMS (ESI) calcd for C$_{20}$H$_{21}$NSO$_3$Na [M+Na]$^+$ 378.11344, found 378.11289.

(R)-5-propyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-291A)

44.7 mg, 61% yield. Colorless oil. $R_f = 0.3$ (petroleum ether/ethyl acetate 10:1); [$\alpha$]$^\text{D}_{19}$ -115 (c 0.1, CH$_2$Cl$_2$); HPLC analysis: 96% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 80:20, 1.0 mL/min, 254 nm, 8.4 min (minor), 26.9 min (major)]; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.93 (d, $J = 8.4$ Hz, 2H), 7.33 (d, $J = 8.4$ Hz, 2H), 7.16-7.12 (m, 3H), 6.96 (s, 1H), 6.83 (d, $J = 7.2$ Hz, 2H), 3.45 (dd, $J = 7.2$, 2.8 Hz, 1H), 2.93 (dd, $J = 15.6$, 7.6 Hz, 1H), 2.54 (dd, $J = 15.6$, 2.8 Hz, 1H), 2.46 (s, 3H), 2.03 (t, $J = 7.2$ Hz), 1.50-1.38 (m, 2H), 0.89 (t, $J = 7.4$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 167.2, 145.2, 139.9, 135.1, 129.4, 128.8, 128.7, 127.3, 126.9, 125.1, 119.7, 41.3, 41.2, 35.0, 21.7, 21.0, 13.7. IR (KBr) 2920, 1722, 1362, 1172; HRMS (ESI) calcd for C$_{21}$H$_{23}$NSO$_3$Na [M+Na]$^+$ 392.12909, found 392.12842.

(R)-5-butyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-293D)

511
51.0 mg, 67% yield. White solid, mp. 120-121 °C. Rf = 0.3 (petroleum ether/ethyl acetate 10:1); [α]25° -85 (c 0.1, CH2Cl2); HPLC analysis: 98% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 12.4 min (minor), 51.8 min (major)]; 1H NMR (300 MHz, CDCl3) δ 7.93 (d, J = 8.3 Hz, 2H), 7.32 (t, J = 7.7 Hz, 2H), 7.17-7.05 (m, 3H), 6.96 (s, 1H), 6.83 (d, J = 7.2 Hz, 2H), 3.46 (dd, J = 7.5, 2.8 Hz, 1H), 2.93 (dd, J = 15.5, 7.6 Hz, 1H), 2.56 (dd, J = 15.5, 2.8 Hz, 1H), 2.54 (s, 3H), 2.13-1.94 (m, 2H), 1.52-1.08 (m, 4H), 0.87 (t, J = 7.1 Hz, 3H). 13C NMR (75 MHz, CDCl3) δ 167.2, 145.1, 139.9, 135.2, 129.4, 128.8, 128.7, 127.3, 126.9, 125.3, 119.6, 41.3, 41.2, 32.6, 29.9, 22.3, 21.7, 13.8. IR (KBr) 2920, 1716, 1362, 1172; HRMS (ESI) calcd for C22H25NSO3Na [M+Na]+ 406.14474, found 406.14423.

(R)-5-pentyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(/H)-one (GZH-292E)

57.1 mg, 72% yield. Colorless oil. Rf = 0.4 (petroleum ether/ethyl acetate 10:1); [α]25° -84 (c 0.1, CH2Cl2); HPLC analysis: 98% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH/MeOH = 90:6:4, 0.5 mL /min, 254 nm, 16.7 min (minor), 39.0 min (major)]; 1H NMR (300 MHz, CDCl3) δ 7.93 (d, J = 8.3 Hz, 2H), 7.33 (d, J = 8.3 Hz, 2H), 7.10 (m, 3H), 6.95 (s, 1H), 6.88-6.75 (m, 2H), 3.45 (dd, J = 7.5, 2.9 Hz, 1H), 2.93 (dd, J = 15.5, 7.6 Hz, 1H), 2.55 (dd, J = 15.5, 2.9 Hz, 1H), 2.46 (s, 3H), 2.11-1.94 (m, 2H), 1.53-1.09 (m, 6H), 0.86 (t, J =8.1 Hz, 3H). 13C NMR (75 MHz, CDCl3) δ 167.2, 145.1, 139.9, 135.2, 129.4, 128.8, 128.7, 127.3, 126.9, 125.3, 119.6, 41.3, 41.2, 32.9, 31.4, 27.4, 22.4, 21.7, 14.0. IR (KBr) 2920, 1716, 1362, 1172; HRMS (ESI) calcd for C23H27NSO3Na [M+Na]+ 420.16039, found 420.15991.
(R)-5-hexyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-282C)

52.6 mg, 64% yield. Colorless oil. R_f = 0.5 (petroleum ether/ethyl acetate 5:1); [α]_D^{25} -59 (c 0.1, CH₂Cl₂); HPLC analysis: 96% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH/MeOH = 90:6:4, 0.5 mL /min, 254 nm, 16.1 min (minor), 36.7 min (major)]; ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, J = 8.3 Hz, 2H), 7.33 (d, J = 8.3 Hz, 2H), 7.14 (t, J = 7.3 Hz, 1H), 7.07 (t, J = 7.4 Hz, 2H), 6.95 (s, 1H), 6.82 (d, J = 7.2 Hz, 2H), 3.45 (dd, J = 7.5, 2.8 Hz, 1H), 2.93 (dd, J = 15.5, 7.6 Hz, 1H), 2.56 (dd, J = 15.5, 2.8 Hz, 1H), 2.46 (s, 3H), 2.11-1.97 (m, 2H), 1.49-1.12 (m, 8H), 0.87 (t, J = 6.9 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 167.2, 145.1, 139.9, 135.2, 129.4, 128.8, 128.7, 127.3, 126.9, 125.3, 119.6, 41.3, 41.2, 32.9, 31.5, 28.8, 27.7, 22.6, 21.7, 14.1. IR (KBr) 2921, 1717, 1362, 1172; HRMS (ESI) calcd for C₂₄H₂₉NSO₃Na [M+Na]⁺ 434.17604, found 434.17566.

(R)-5-heptyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-391B)

55.5 mg, 65% yield. Colorless oil. R_f = 0.5 (petroleum ether/ethyl acetate 5:1); [α]_D^{25} -46 (c 0.1, CH₂Cl₂); HPLC analysis: 98% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH/MeOH = 90:6:4, 0.5 mL /min, 254 nm, 15.6 min (minor), 34.4 min (major)]; ¹H NMR (300 MHz, CDCl₃) δ 7.93 (d, J = 8.3 Hz, 2H), 7.33 (d, J = 8.3 Hz, 2H), 7.18-7.01 (m, 3H), 6.95 (s, 1H), 6.86-6.75 (m, 2H), 3.45 (dd, J = 7.5, 2.9 Hz, 1H), 2.93 (dd, J = 15.5, 7.6 Hz, 1H), 2.56 (dd, J = 15.5, 2.9 Hz, 1H), 2.46 (s, 3H), 2.11-1.96 (m, 2H), 1.52-1.12 (m, 10H), 0.88 (t, J = 6.8 Hz, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 167.2, 145.1, 139.9, 135.2, 129.4, 128.8, 128.7, 126.9, 125.3,
(R)-5-octyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-294C)

68.2 mg, 78% yield. Colorless oil. Rₜ = 0.5 (petroleum ether/ethyl acetate 5:1); [α]₂⁵° -112 (c 0.1, CH₂Cl₂); HPLC analysis: 98% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 10.0 min (minor), 33.0 min (major)]; ¹H NMR (300 MHz, CDCl₃) δ 7.93 (d, J = 8.3 Hz, 2H), 7.33 (d, J = 8.3 Hz, 2H), 7.20-6.99 (m, 3H), 6.95 (s, 1H), 6.87-6.75 (m, 2H), 3.45 (dd, J = 7.5, 2.8 Hz, 1H), 2.93 (dd, J = 15.5, 7.6 Hz, 1H), 2.56 (dd, J = 15.5, 2.9 Hz, 1H), 2.45 (s, 3H), 2.04 (t, J = 7.7 Hz, 2H), 1.54-1.10 (m, 12H), 0.88 (t, J = 6.7 Hz, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 167.2, 145.1, 139.9, 135.2, 129.4, 128.8, 128.7, 127.2, 126.9, 125.3, 119.6, 41.3, 41.2, 32.9, 31.8, 29.3, 29.2, 29.2, 27.7, 22.7, 21.7, 14.1. IR (KBr) 2920, 1716, 1362, 1173; HRMS (ESI) calcd for C₂₅H₃₁NSO₃Na [M+Na]⁺ 448.19169, found 448.119125.

(R)-5-nonyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-294E)

73.3 mg, 81% yield. Colorless oil. Rₜ = 0.5 (petroleum ether/ethyl acetate 5:1); [α]₂⁵° -42 (c 0.1, CH₂Cl₂); HPLC analysis: 97% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 9.6 min (minor), 33.6 min (major)]; ¹H NMR (300 MHz, CDCl₃) δ 7.93 (d, J = 8.3 Hz, 2H), 7.34 (t, J = 8.3 Hz,
2H), 7.15-7.04 (m, 3H), 6.95 (s, 1H), 6.82 (d, $J = 7.0$ Hz, 1H), 3.45 (dd, $J = 7.5$, 2.8 Hz, 1H), 2.93 (dd, $J = 15.5$, 7.6 Hz, 1H), 2.56 (dd, $J = 15.5$, 2.9 Hz, 1H), 2.45 (s, 3H), 2.04 (t, $J = 7.7$ Hz, 2H), 1.53-1.11 (m, 14H), 0.88 (t, $J = 6.7$ Hz, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 167.2, 145.1, 139.9, 135.2, 129.4, 128.8, 128.7, 127.2, 126.9, 125.3, 119.6, 41.3, 41.2, 32.9, 31.9, 29.5, 29.3, 29.2, 27.7, 22.7, 21.7, 14.1. IR (KBr) 2922, 1716, 1362, 1172; HRMS (ESI) calcd for C$_{27}$H$_{35}$NSO$_3$Na [M+Na]$^+$ 476.22299, found 476.22263.

(R)-5-isopropyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-293A)
54.9 mg, 74% yield. Colorless oil. R$_f$ = 0.3 (petroleum ether/ethyl acetate 10:1); $[\alpha]_D^{25}$ -97 (c 0.1, CH$_2$Cl$_2$); HPLC analysis: 95% ee [Daicel CHIRALPAK OD-H column, 20 $^\circ$C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 12.6 min (minor), 63.0 min (major)]; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 7.94 (d, $J = 8.3$ Hz, 2H), 7.33 (d, $J = 8.3$ Hz, 2H), 7.17-6.96 (m, 4H), 6.88-6.76 (m, 2H), 3.54 (dd, $J = 7.3$, 2.2 Hz, 1H), 2.90 (dd, $J = 15.4$, 7.3 Hz, 1H), 2.52 (dd, $J = 15.4$, 2.2 Hz, 1H), 2.45 (s, 3H), 2.34 (m, 1H), 1.07 (d, $J = 6.8$ Hz, 3H), 1.01 (t, $J = 6.8$ Hz, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 167.2, 145.1, 140.1, 135.2, 131.1, 129.4, 128.8, 128.7, 127.2, 126.8, 119.1, 41.9, 39.9, 31.8, 22.0, 21.7, 21.5. IR (KBr) 2920, 1720, 1361, 1173; HRMS (ESI) calcd for C$_{21}$H$_{23}$NSO$_3$Na [M+Na]$^+$ 392.12909, found 392.12869.

(S)-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one (GZH-391C)
55.4 mg, 57% yield. Colorless oil. Rf = 0.3 (petroleum ether/ethyl acetate 5:1); [α]D
-107 (c 0.1, CH2Cl2); HPLC analysis: 99% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/i-PrOH/MeOH = 90:6:4, 1.0 mL /min, 254 nm, 15.8 min (minor), 18.0 min (major)]; 1H NMR (300 MHz, CDCl3) δ 7.93 (d, J = 8.4 Hz, 2H), 7.34 (d, J = 8.4 Hz, 2H), 7.24-7.17 (m, 2H), 7.15 (dd, J = 8.2, 1.7 Hz, 1H), 7.08-6.97 (m, 2H), 5.48 (dd, J = 8.2, 4.2 Hz, 1H), 3.80-3.59 (m, 1H), 2.84 (dd, J = 15.7, 6.7 Hz, 1H), 2.63 (dd, J = 15.7, 8.6 Hz, 1H), 2.45 (s, 3H). 13C NMR (75 MHz, CDCl3) δ 167.4, 145.3, 141.1, 135.2, 129.5, 128.9, 128.7, 127.3, 126.8, 124.7, 112.6, 41.3, 37.8, 21.7. IR (KBr) 2920, 1730, 1362, 1172; HRMS (ESI) calcd for C18H17NSO3Na [M+Na]+ 350.08214, found 350.08176.

2. Chemical transformations of compound 3a

To a stirred solution of 3a (208.5 mg, 0.5 mmol) in ethanol/ethyl acetate (1:1, 4 ml) was added Pd/C (10 mol%), H2 (30 atm). After stirring for 24 h, the solvent was removed in vacuo and the product re-dissolved in ethyl acetate. Then, the solvent was filtered through a plug of celite, evaporated, and purified by column chromatography to afford 6 (209 mg, 99%) as an inseparable cis (major) and trans (minor) mixture in 99% ee (major isomer) and 4:1 dr.

(4S,5S)-5-benzyl-4-phenyl-1-tosylpiperidin-2-one (GZH-393A)

209 mg, 99% yield. White solid, mp. > 250 °C. Rf = 0.1 (petroleum ether/ethyl acetate 5:1); [α]D
= -8 (c 0.1, CH2Cl2); HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 °C, 254 nm, hexane/i-PrOH/MeOH = 60:30:10, 1.0 mL /min, 254 nm, 13.0 min (minor), 21.3 min (major)]; 1H NMR (400 MHz, CDCl3) δ 7.88 (d, J = 8.3 Hz, 2H), 7.33-7.25 (m, 8H), 7.09 (t, J = 8.2 Hz, 4H), 4.02 (dd, J = 12.6, 4.1 Hz, 1H), 3.61-3.47 (m, 1H), 2.89-2.77 (m, 1H), 2.74-2.68 (m, 2H), 2.66-2.59
(m, 1H), 2.44-2.32 (m, 5H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 169.8, 144.9, 141.8, 138.4, 135.8, 129.4, 129.3, 129.1, 128.8, 128.7, 127.4, 127.2, 126.7, 49.5, 44.0, 42.3, 41.0, 37.9, 21.7. IR (KBr) 2921, 1635, 1454; 1169. HRMS (ESI) calcd for C$_{25}$H$_{25}$NSO$_3$Na [M+Na]$^+$ 442.14474, found 442.14398.

To a stirred solution of 3a (41.7 mg, 0.1 mmol) in THF (1.0 mL) at 0 °C, under N$_2$ atmosphere, was dropwise added LiAlH$_4$ (300 µL, 1 M in THF, 0.3 mmol, 3 equiv.). The reaction was stirred at 0 °C for 1 h. Then the solution was quenched with 0.1 M HCl solution. The aqueous phase was extracted three times with DCM. The combined organic phases were dried over Na$_2$SO$_4$, filtered, and concentrated under reduced pressure. The residue was purified via silica gel flash chromatography (petroleum ether/ethyl acetate 8:1-3:1) to afford 7 (41.5 mg, 99%) as a white solid.

(R,Z)-N-(2-benzyl-5-hydroxy-3-phenylpent-1-enyl)-4-methylbenzenesulfonamide (GZH-S230A)

41.5 mg, 99% yield. White solid, 106-107 °C. R$_f$ = 0.1 (petroleum ether/ethyl acetate 5:1); [$\alpha$]$^\circ$ = -39 (c 0.1, CH$_2$Cl$_2$); HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/i-PrOH = 90:10, 1.0 mL /min, 254 nm, 13.8 min (minor), 15.3 min (major)]; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.17 (d, $J$ = 9.1 Hz, 1H), 7.72 (d, $J$ = 8.1 Hz, 2H), 7.32 (d, $J$ = 8.0 Hz, 2H), 7.17-7.12 (m, 5H), 6.87 (d, $J$ = 6.6 Hz, 2H), 6.72 (m, 2H), 5.96 (d, $J$ = 9.3 Hz, 1H), 3.90 (dd, $J$ = 9.3 Hz, 1H), 3.61 (m, 1H), 3.40-3.13 (m, 1H), 2.86 (d, $J$ = 12.0 Hz, 1H), 2.77 (d, $J$ = 15.2 Hz, 1H), 2.48 (s, 3H), 2.14 (brs, 1H), 1.98-1.81 (m, 1H), 1.48 (t, $J$ = 13.0 Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 143.4, 141.6, 139.2, 137.3, 129.7, 129.0, 128.2, 128.2, 128.0, 127.3, 127.0, 126.5, 126.3, 122.1, 59.4, 39.4, 37.1, 31.9, 21.6. IR (KBr) 3446, 2922, 2851, 1633, 1162. HRMS (ESI) calcd for C$_{25}$H$_{27}$NSO$_3$Na [M+Na]$^+$ 444.15970, found 444.16039.
3. X-ray structure of compound 3g

The crystal of compound 3g was prepared from its solution in petroleum ether/CH$_2$Cl$_2$ by slow evaporation. The structure of compound 3g was established by the X-ray analysis of its crystal (Figure S1).

![Figure S1. X-ray structure of 3g.](image)

4. References


Part III NMR Spectra
3m
Part IV HPLC Spectra

rac-3a

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Acq. Method : C:\CHEM32\METHODS\JWQ20121205.M
Last changed : 2014-7-1 19:01:51 by gzh
(modified after loading)
Analysis Method : C:\CHEM32\METHODS\JWQ20121205.M
Last changed : 2014-9-6 17:09:12 by skq
(modified after loading)
Sample Info : GC-H N/1=90:10 1.0ml/min 250nm

![UV chromatogram with peaks at 330 and 3149 mAU](image)

### Area Percent Report

<table>
<thead>
<tr>
<th>Sorted By</th>
<th>Signal</th>
<th>Multiplier</th>
<th>Dilution</th>
<th>Use Multiplier &amp; Dilution Factor with ISTDs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Signal 1</td>
<td>VWD1 A, Wavelength=250 nm</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area Height</th>
<th>Area</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>[min]</td>
<td>[mAU]</td>
<td>[mAU]</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>24.338</td>
<td>1.0225</td>
<td>6735.62842</td>
<td>99.52602</td>
</tr>
<tr>
<td>2</td>
<td>37.149</td>
<td>1.5250</td>
<td>6767.04785</td>
<td>67.83276</td>
</tr>
</tbody>
</table>

Totals : 1.35027e4  167.35877

*** End of Report ***
Acq. Operator : ghb
Acq. Instrument : Instrument 1
Location : Vial 1
Injection Date : 2014-7-1 17:10:33
Last changed : 2014-7-1 17:12:10 by ghb
(modified after loading)
Analysis Method : C:\CHEN32\METHODS\JWQ20121205.M
Last changed : 2014-9-6 17:07:07 by ghb
(modified after loading)
Sample Info : OD-M H/I=90:10 1.0nl/min 250nm

Signal 1: WVD1 A, Wavelength=250 nm
Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [mAU ] %
------- -------- -------- -------- ------- ------- -------
1 25.408 BV 0.9998 632.42457 9.65557 1.3082
2 37.291 BB 1.5618 4.77120e4 461.36322 98.6918
Totals : 4.83444e4 471.06388

*** End of Report ***
Acq. Operator : gzh
Acq. Instrument : Instrument 1
Location : Vial 1
Injection Date : 2014-7-1 16:15:14
Acq. Method : C:\CHEM32\METHODS\JWQ20121205.M
Last changed : 2014-7-1 16:16:38 by gzh
(modified after loading)
Analysis Method : C:\CHEM32\METHODS\JWQ20121205.M
Last changed : 2014-9-17 10:31:52 by ckg
(modified after loading)
Sample Info : OD-H H/I=90:10 1.0ml/Min 250nm

VWD1 A, Wavelength=250 nm (520H000901D)

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=250 nm

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25.364</td>
<td>BB</td>
<td>1.1255</td>
<td>1.57761e4</td>
<td>211.73230</td>
<td>49.7106</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>38.590</td>
<td>BB</td>
<td>1.6422</td>
<td>1.59571e4</td>
<td>150.09341</td>
<td>50.2852</td>
<td></td>
</tr>
</tbody>
</table>

Totals : 3.17332e4 361.82571

*** End of Report ***
Acq. Operator : gzh  Location : Vial 1
Acq. Instrument : Instrument 1
Injection Date : 2014-7-1 15:27:59
Acq. Method : C:\CHEM32\METHODS\JWQ20121205.M
Last changed : 2014-7-1 15:24:52 by gzh
(modified after loading)
Analysis Method : C:\CHEM32\METHODS\JWQ20121205.M
Last changed : 2014-9-6 17:01:43 by ckg
(modified after loading)
Sample Info : CD-H H/I=90:10 1.0ml/min 250nm

Area Percent Report

Signal 1: VWD1 A, Wavelength=250 nm

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime [min]</th>
<th>Width [min]</th>
<th>Area [mAU]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>26.903</td>
<td>1.1558</td>
<td>426.71927</td>
<td>5.71926</td>
<td>1.2842</td>
</tr>
<tr>
<td>2</td>
<td>39.324</td>
<td>1.6597</td>
<td>3.28019e4</td>
<td>300.08530</td>
<td>98.7158</td>
</tr>
</tbody>
</table>

Totals : 3.3286e4 305.80455

*** End of Report ***
Data File C:\CHEM32\DATA\GSH\000257.D
Sample Name: GSH-2908 3-Chloro racemic

---

Acq. Operator: gzh
Acq. Instrument: Instrument 1
Location: Viel 1
Injection Date: 2015-5-4 20:13:31
Acq. Method: C:\CHEM32\DATA\METHODS\JWQ20121208.M
Last changed: 2015-5-4 20:13:31 by gzh
(modified after loading)
Analysis Method: C:\CHEM32\DATA\METHODS\JWQ20121208.M
Last changed: 2015-5-4 21:36:48 by gzh
(modified after loading)
Sample Info: OD=H N/H=90:64 1 ml/min 254 nm

---

Areas Percent Report

---

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VW01 A, Wavelength=254 nm

---

Peak RetTime Type Width Area % Height Area %
----- ------ ------ ------ ------ ------ ------ ------
1 16.421 s1 0.5965 1.22500e4 375.9166 64.9466 2 19.406 s1 0.8787 1.22822e4 333.1941 50.0534

Totals: 2.45381e4 708.2108

---

*** End of Report ***
Sample Name: gzh-275a

Acq. Operator : gzh
Acq. Instrument : Instrument 1
Location : Vial 1
Injection Date : 2014-6-27 19:40:15
Acq. Method : C:\CHEM33\METHODS\JWQ20121205.M
Last changed : 2014-6-27 16:55:36 by gzh
(modified after loading)
Analysis Method : C:\CHEM33\METHODS\JWQ20121205.M
Last changed : 2014-6-6 16:50:39 by ckg
(modified after loading)
Sample Info : GC-H R/=98:10 1.0ml/min 250nm

Signal 1: VW01 A, Wavelength=250 nm

<table>
<thead>
<tr>
<th>Ret Time</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>17.813</td>
<td>0.8932</td>
<td>163.3909</td>
<td>2.74198</td>
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<tr>
<td>2</td>
<td>29.349</td>
<td>1.2825</td>
<td>9832.20703</td>
<td>117.09649</td>
</tr>
</tbody>
</table>

Totals : 9995.60339 119.83847

--- End of Report ---

SS4
Acq. Operator : qzh
Acq. Instrument : Instrument 1
Injection Date : 2014-9-11 10:35:54
Acq. Method : C:\CHEM32\1\METHODS\JWQ20121205.M
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
(modified after loading)
Sample Info : O-D H R/I=90:10 mL/min 254nm

---

VWD1 A, Wavelength=254 nm (GZCHC090131.D)

---

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area [mAU]</th>
<th>Height [mA]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>13.245</td>
<td>BB</td>
<td>0.678</td>
<td>4200.19213</td>
<td>96.13531</td>
<td>49.806</td>
</tr>
<tr>
<td>24.162</td>
<td>BB</td>
<td>1.093</td>
<td>4206.79932</td>
<td>59.06753</td>
<td>50.0394</td>
</tr>
</tbody>
</table>

Totals : 8406.98145 155.20284

---

*** End of Report ***
Acq. Operator : gzh
Acq. Instrument : Instrument 1
Injection Date : 2014-9-11 11:15:32
Last changed : 2014-9-11 10:26:54 by gzh
Analysis Method : C:\\CHEN321\METHODS\MwQ20121205.M
Last changed : 2014-9-11 10:26:54 by gzh
Sample Info : OD-K H/I=90:10 l/mL/min 254nm

Sorted By : Signal
Multiplier : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type Width Area Height Area
--- | [min] | [min] | mAU | a | [mAU | b
1  14.076 BB 0.6656 108.17165 2.51140 2.6053
2  23.547 BB 1.3781 4043.85938 57.40792 97.3947

Totals : 4152.03102 59.91932

*** End of Report ***
Data File C:\\CHEM321\DATA\GEH\000274.D
Sample Name: gzh-395D 4-amide racemic

Acq. Operator : gzh
Acq. Instrument : Instrument 1
Location : Vial 1
Injection Date : 2015-3-18 19:04:00
Acq. Method : C:\\CHEM321\METHODS\JWQ20121205.M
Last changed : 2015-3-18 19:34:21 by gzh
(modified after loading)
Analysis Method : C:\\CHEM321\METHODS\JWQ20121205.M
Last changed : 2015-3-18 19:34:43 by gzh
(modified after loading)
Sample Info : OD-H R/1/M=70;20:10 1.0 ml/min 254 nm

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime [min]</th>
<th>Width [min]</th>
<th>Area [mAU]</th>
<th>Height [mAU]</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15.166 MM</td>
<td>0.8898</td>
<td>2851.37305</td>
<td>52.86636</td>
<td>49.3365</td>
</tr>
<tr>
<td>2</td>
<td>23.925 MM</td>
<td>1.3614</td>
<td>2928.02173</td>
<td>35.84447</td>
<td>50.6631</td>
</tr>
</tbody>
</table>

Totals : 5779.39478 88.71083

*** End of Report ***
Data File: C:\\CHEM32\\DATA\\GSH\\000275.D
Sample Name: gzh-395C 4-amide

Acq. Operator: gzh
Acq. Instrument: Instrument 1
Injection Date: 2015-5-18 19:39:53
Acq. Method: C:\\CHEM32\\METHODS\\JWQ0111208.M
Last Changed: 2015-5-18 20:15:09 by gzh
Analysis Method: C:\\CHEM32\\METHODS\\JWQ0111208.M
Last Changed: 2015-5-18 20:17:53 by gzh
Sample Info: OD-8 R/7/N=70.20:10 1.0 ml/min 254 nm

MWDA A. Wavelength=254 nm (Q5=000275.D)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: MWDA A, Wavelength=254 nm

Peak RetTime Type Width Area Height Area %
<table>
<thead>
<tr>
<th>[min]</th>
<th>[min]</th>
<th>mAU</th>
<th>[mAU]</th>
</tr>
</thead>
<tbody>
<tr>
<td>23.81</td>
<td>1.2164</td>
<td>1.9988e4</td>
<td>245.85287</td>
</tr>
</tbody>
</table>

Totals: 1.9984e4 245.85287

*** End of Report ***
Data File: C:\CHEM32\DATA\GZH\GZH0273.D
Sample Name: gzh-3998 4-CN racemic

Acq. Operator: gzh
Acq. Instrument: Instrument 1
Injection Date: 2015-5-18 16:57:44
Injection Name: C:\CHEM32\DATA\GZH\GZH0273.D
Injection Method: C:\CHEM32\DATA\GZH\GZH0273.D
Last changed: 2015-5-18 18:40:16 by gzh
(modified after loading)
Analysis Method: C:\CHEM32\DATA\GZH\GZH0273.D
Last changed: 2015-5-18 18:42:42 by gzh
(modified after loading)
Sample Info: OD-E Rt/Ret=99:16 1.0 ml/min 254 nm

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISIDs

Signal 1: VWD1 A, Wavelength=254 nm
Peak RetTime Type Width Area Height Area
# [min] [min] mAU [mAU] [mAU] [-][-]
1 37.460 m 1.2089 1.21436e4 161.08490 48.4187
2 55.477 m 1.7379 1.29431e4 113.02414 51.5813
Totals: 2.50927e4 274.10704

*** End of Report ***
Sample Name: gzh-296D racemic

Data collected as follows:

Acq. Operator: gzh
Acq. Instrument: Instrument 1
Injection Date: 2014-9-23 18:28:39
Acq. Method: C:\\CHEM32\\METHODS\\JWQ20112105.M
Last Changed: 2014-9-23 18:19:42 by gzh
(modified after loading)
Analysis Method: C:\\CHEM32\\METHODS\\JWQ20112105.M
Last Changed: 2014-9-8 16:59:28 by c4q
(modified after loading)
Sample Info: OD-H L=1/M=95:1 1mL/min 254nm

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VM1 A, Wavelength=254 nm

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.800</td>
<td>VV</td>
<td>0.7640</td>
<td>3865.22583</td>
<td>79.21175</td>
</tr>
<tr>
<td>2</td>
<td>20.304</td>
<td>VB</td>
<td>0.7646</td>
<td>3546.31350</td>
<td>71.55048</td>
</tr>
</tbody>
</table>

Totals: 7411.54541 150.76224

End of Report
Sample Name: gzh-296C

Acq. Operator : gzh
Acq. Instrument : Instrument 1
Injection Date : 2014-8-23 19:08:36
Injection Vol : 20.0000
Injection Time : 1.0000
Analysis Method : C:\CHEM32\METHODS\JNW20121205.M
Analysis Date : 2014-9-6 17:00:14
Analysis Time : 1.0000

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type Width Area Height Area %
---|---------|--------|----------|----------|-------|-------|
1 19.661 UV 0.7577 522.25433 10.45223 5.5787
2 21.869 VB 0.8678 8839.24805 152.62944 94.4213
Totals : 9361.50239 163.08167

*** End of Report ***
Sample Name: gzh-292b

Acq. Operator : gzh
Acq. Instrument : Instrument 1
Location : Vial 1
Injection Date : 2014-6-25 22:02:57
Acq. Method : C:\CHEM32\METHODS\JWQ20121205.M
Last changed : 2014-6-25 22:44:13 by gzh
(modified after loading)
Analysis Method : C:\CHEM32\METHODS\JWQ20121205.M
Last changed : 2014-8-6 16:22:37 by ckq
(modified after loading)
Sample Info : OJ-18 A1•D019 1.0mL/min 250nm

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=250 nm

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width [min]</th>
<th>Area [mAU]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>17.471</td>
<td>689.24324</td>
<td>17.18647</td>
<td>50.6892</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>46.139</td>
<td>661.76538</td>
<td>17.05870</td>
<td>49.3108</td>
<td></td>
</tr>
</tbody>
</table>

Totals : 1342.02663 24.17225

*** End of Report ***
Sample Name: gzh-292a

Acq. Operator : gzh
Location : Vial 1
Injection Date : 25-Jun-14, 22:55:54
Acq. Method : JWQ20121125.M
Analysis Method : C:\\CHEN32\\METHODS\\JWQ20121125.M
Last Changed : 2014-9-6 16:24:05 by ckq
(modified after loading)
Sample Info : OD=M H/I=9E10 1.0ml/min 250nm

VWD1 A, Wavelength=250 nm (322900008345)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=250 nm

Peak RetTime Type Width Area Height Area %
--- ----------- ------- ------- ------- ------- ------- -------
1 17.440 BR 0.6966 98.732558 2.17613 1.3914
2 45.684 BR 1.5176 6997.31641 70.84755 98.6086
Totals : 7096.04899 73.12368

*** End of Report ***
Data File C:\CHEM32\1\DATA\GEH\000005.D
Sample Name: gsh-292c

Acq. Operator : gsh
Acq. Instrument : Instrumet 1
Injection Date : 2014-5-26 11:07:11
Acq. Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed : 2014-5-26 12:06:29 by gsh
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed : 2014-9-6 16:26:46 by ckq
Sample Info : OD-H R/I=59:10 1.0mL/min 250mm

Area Percent Report

Signal 1: VWD1 A, Wavelength=250 nm

Peak RetTime Type Width Area Height Area %
# [min] [min] nAU [nAU] [
1 14.511 MW 0.6333 153.81590 4.04805 1.1777
2 54.070 RH 1.8674 1.29064e4 102.33691 98.8223
Totals : 1.00630e4 106.38396

--- End of Report ---
Sample Name: gzh-291b

Acq. Operator: gzh
Acq. Instrument: Instrument 1
Injection Date: 2014-6-25 16:44:56
Acq. Method: C:\CHEMS\\METHODS\JWQ20121203.M
Last changed: 2014-6-25 15:41:19 by lzp
(modified after loading)
Analysis Method: C:\CHEMS\\METHODS\JWQ20121203.M
Last changed: 2014-9-6 16:07:06 by ckg
(modified after loading)
Sample Info: OD H/1=80:20 1.0ml/min 250nm

---

VWD1 A, Wavelength=250 nm (G2H=00095E 000)

---

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISIDs

Signal 1: VWD1 A, Wavelength=250 nm

Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [mAU ] %
--- ------- ---- ----------- ---------
1 8.490 VB 0.3093 4349.6016 317.26871 50.9967
2 27.051 BB 1.0445 4180.30713 61.87226 49.0073

Totals: 6529.96720 279.16098

---

*** End of Report ***
Sample Name: qzh-293b

Acq. Operator : qzh  
Location : Vial 1

Injection Date : 26-Jun-14, 20:33:36
Acq. Method : JNQ20121205.M
Analysis Method : C:\CHRM32\METHODS\JNQ20121205.M
Last changed : 2014-3-6 16:12:00 by cqg
(modified after loading)
Sample Info : 00-H A/1=80:20 1.0mL/min 250nm

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with STDs

Signal 1: VWD1 A, Wavelength=250 nm

<table>
<thead>
<tr>
<th>Peak Ref</th>
<th>T1</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.416</td>
<td>0.4704</td>
<td>669.62103</td>
<td>19.79294</td>
<td>1.9014</td>
</tr>
<tr>
<td>2</td>
<td>26.863</td>
<td>1.1492</td>
<td>3.45478e4</td>
<td>454.35645</td>
<td>96.0396</td>
</tr>
</tbody>
</table>

Totals : 3.52174e4 474.14939

*** End of Report ***
Sample Name: gsh-291d

Acq. Operator : gsh
Acq. Instrument : Instrument 1
Injection Date : 2014-6-27 16:55:48
Acq. Method : C:\CHEM32\METHODS\JWQ20121205.M
Last Changed : 2014-6-27 16:55:16 by gsh
(modified after loading)
Analysis Method : C:\CHEM32\METHODS\JWQ20121205.M
Last Changed : 2014-9-6 16:17:16 by ckg
(modified after loading)
Sample Info : 00-M A/l=90:10 1.0ml/min 250mm

---

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VW01 A, Wavelength=250 nm

Peak RetTime Type Width Area Height Area
# [min] [min] nAU s [nAU] %
---|---|---|---|---|---|---|
1 12.297 VV 0.4680 784.77809 26.04533 49.3015
2 53.284 VB 1.9000 807.01434 6.59215 50.6985

Totals : 1591.79242 32.63747

---

*** End of Report ***
Sample Name: gzh-293d (2910)

Acq. Operator : gzh
Acq. Instrument : Instrument 1
Injection Date : 2014-6-27 10:42:03
Injection Time : 2014-6-27 10:30:09 by gzh
Injection Volume : 25 µL
Analysis Method : C:\\CHEM32\\1\METHODS\\JWQ20121205.M
Last Charged : 2014-9-6 16:19:26 by ckq
Sample Info : OD-H H/L=56.10.1.0mL/min 250nm

VWD1 A, Wavelength=250 nm (02M10000971.D)

== Aera Percent Report ==

Signal 1: VWD1 A, Wavelength=250 nm

<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>12.352</td>
<td>0.4752</td>
<td>99.84862</td>
<td>3.30137</td>
<td>0.9561</td>
</tr>
<tr>
<td>2</td>
<td>51.827</td>
<td>1.9163</td>
<td>1.0344Da4</td>
<td>82.54314</td>
<td>99.0439</td>
</tr>
</tbody>
</table>

Totals : 1.04438e4 85.84452

*** End of Report ***
Data File \C:\CHEM32\DATA\021\000246.D
Sample Name: S44-292F C7 racemic

Acq. Operator : ghj
Acq. Instrument : Instrument 1
Injection Date : 2015-5-4 11:46:57
Acq. Method : \C:\\CHEM32\METHODS\JWQ20121205.N
Last changed : 2015-5-4 11:46:55 by ghj
(modified after loading)
Analysis Method : \C:\\CHEN32\METHODS\JWQ20121205.N
Last changed : 2015-5-4 21:53:03 by ghj
(modified after loading)
Sample info : OC-H N1/H=901618 0.2 ml/min 254 nm

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with IS7D

Signal 1: VWD1 A, Wavelength=254 nm

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width [min]</th>
<th>Area [mAU]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 16.452</td>
<td>UV</td>
<td>0.4905</td>
<td>3759.93677</td>
<td>144.70047</td>
<td>50.7704</td>
</tr>
<tr>
<td>2 38.181</td>
<td>BB</td>
<td>0.9318</td>
<td>3626.43164</td>
<td>55.94635</td>
<td>49.2296</td>
</tr>
</tbody>
</table>
Totals : | | | | | 7366.36841 | 204.24682

*** End of Report ***
Data File C:\CHEM32\DATA\Q2X\000245.D
Sample Name: 06A-2928 CT

Acq. Operator : gzh
Acq. Instrument : Instrument 1
Injection Date : 2015-5-4 10:58:15
Acq. Method : C:\\CHEM32\\METHODS\\JWQ20121208.M
Last changed : 2015-5-4 10:36:59 by gzh
(modified after loading)
Analysis Method : C:\\CHEM32\\METHODS\\JWQ20121208.M
Last changed : 2015-5-4 21:54:20 by gzh
(modified after loading)
Sample info : 06-H\1/3-991614 0.2 ml/min 254 nm

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with IS07s

Signal 1: VWD1 A, Wavelength=254 nm

<table>
<thead>
<tr>
<th>Peak RetTime Type Width Area Height Area</th>
<th>#</th>
<th>(min)</th>
<th>(min)</th>
<th>mAU</th>
<th>mAU</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>16.664</td>
<td>0.4115</td>
<td>644.17</td>
<td>24.03</td>
<td>0.989</td>
</tr>
</tbody>
</table>

Totals : 6.481104 908.4306

*** End of Report ***
Data File C:\CHEM32\DATA\GZK\000248.D
Sample Name: 214-282D C# racemic

-----------------------------------------------------------------------------

Acq. Operator : gzh
Acq. Instrument : Instrument 1
Injection Date : 2015-5-4 15:07:58
Acq. Method : C:\CHEM32\WORK\METHD5\JNWQ20121208.M
Last changed : 2015-5-4 15:12:58 by gzh
(modified after loading)
Analysis Method : C:\CHEM32\WORK\METHD5\JNWQ20121208.M
Last changed : 2015-5-4 21:50:02 by gzh
(modified after loading)
Sample info : DD-H N/10=901614 0.2 ml/min 254 nm

-----------------------------------------------------------------------------

Area Percent Report

---

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISSTDs

Signal 1: VWD 1 A, Wavelength=254 nm

<table>
<thead>
<tr>
<th>Peak 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>36.955 min</td>
<td>0.9236</td>
<td>6569.3354</td>
<td>95.1601</td>
<td>49.1969</td>
</tr>
<tr>
<td>2</td>
<td>36.129 min</td>
<td>0.4332</td>
<td>6569.6240</td>
<td>211.6600</td>
<td>50.8031</td>
</tr>
</tbody>
</table>

Totals : 1.15340e4 307.12694

---

*** End of Report ***
Data File C:\CHEN321\DATA\GZH\000247.D
Sample Name: 654-282C

Acq. Operator : gzh
Acq. Instrument : Instrument 1
Injection Date : 2015-5-4 14:16:48
Acq. Method : C:\CHEN321\VMSTHOGS\JMQJ20121205.M
Last changed : 2015-5-4 14:13:34 by gzh
(modified after loading)
Analysis Method : C:\CHEN321\VMSTHOGS\JMQJ20121205.M
Last changed : 2015-5-4 21:51:53 by gzh
(modified after loading)
Sample info : OC- H N 1/1=90161 0.2 ml/min 254 nm

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with IS7Ds

Signal 1: VWD1 A, Wavelength=254 nm

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Width [min]</th>
<th>Area [mAU]</th>
<th>Area [%]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>16.061</td>
<td>0.6255</td>
<td>480.09085</td>
<td>17.45236</td>
<td>2.0665</td>
</tr>
<tr>
<td>2</td>
<td>36.714</td>
<td>0.9608</td>
<td>2.270516e4</td>
<td>367.62164</td>
<td>97.9335</td>
</tr>
</tbody>
</table>

Totals : 2.32316e4 385.07400

*** End of Report ***
Data File C:\CHEN32\DATA\02H\000250.D
Sample Name: 024-281P CA racemic

Acq. Operator : gzh
Acq. Instrument : Instrument 1
Injection Date : 2015-5-4 16:35:40
Acq. Method : C:\CHEN32\METHODS\JWQ20121005.M
Last changed : 2015-5-4 15:12:58 by gzh
(modified after loading)
Analysis Method : C:\CHEN32\METHODS\JWQ20121005.M
Last changed : 2015-5-4 21:46:16 by gzh
(modified after loading)
Sample info : 02-H-017=00160 0.2 ml/min 254 nm

VWD1 A, Wavelength=254 nm (254000250.D)

Area Percent Report

Sorted By : Signal multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with IS7Ds

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type Width Area Height Area % %
--- [min] [min] [mAU] [mAU] [mAU]
1 15.593 UV 0.4142 6643.55322 245.9225 50.6811
2 34.289 BB 0.8933 6464.99072 114.22908 49.3189
Totals : 1.31026e+6 360.1413

*** End of Report ***
Data File C:\CHEN32\DATA\G2H\000249.D
Sample Name: 014-1518 39

-----------------------------------------------------------------------------
Acq. Operator : gzh
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 2015-5-4 15:51:23
Acq. Method : C:\CHEN32\METHODS\JWQ20121208.M
Last changed : 2015-5-4 15:12:58 by gzh
(modified after loading)
Analysis Method : C:\CHEN32\METHODS\JWQ20121208.M
Last changed : 2015-5-4 21:47:39 by gzh
(modified after loading)
Sample info : DD-M H/1/M=90184 0.2 ml/min 254 nm

-----------------------------------------------------------------------------

Area Percent Report

-----------------------------------------------------------------------------
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with IS7Ds

Signal 1: VWD1 A, Wavelength=254 nm

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>34.447</td>
<td>0.9182</td>
<td>2.50227e4</td>
<td>426.05383</td>
</tr>
<tr>
<td>Totals</td>
<td></td>
<td></td>
<td></td>
<td>2.52844e4</td>
</tr>
</tbody>
</table>

-----------------------------------------------------------------------------

*** End of Report ***
Sample Name: ghz-294D

Acq. Operator : ghz
Acq. Instrument : Instrument 1
Location : Vial 1
Injection Date : 2014-6-30 15:54:53
Acq. Method : C:\CHEM32\METHODS\JM20121205.M
Last changed : 2014-6-30 17:30:49 by ghz
(modified after loading)
Analysis Method : C:\CHEM32\METHODS\JM20121205.M
Last changed : 2014-9-6 16:48:40 by ckg
(modified after loading)
Sample Info : CD-H H/1=90:10 1.0ml/min 250nm

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=250 nm

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.855</td>
<td>BV</td>
<td>0.4005</td>
<td>6726.27002</td>
<td>260.26773</td>
<td>48.2242</td>
<td></td>
</tr>
<tr>
<td>33.413</td>
<td>BB</td>
<td>1.2834</td>
<td>7221.64209</td>
<td>85.92634</td>
<td>51.7758</td>
<td></td>
</tr>
</tbody>
</table>

Totals : 1.3947e4 346.19407

*** End of Report ***
Sample Name: gzh-294C

Acq. Operator: gzh
Acq. Instrument: Instrument: Location: Vial 1
Injection Date: 2014-6-30 16:05:29
Last changed: 2014-6-30 16:30:48 by gzh
(modified after loading)
Analysis Method: C:\\CHEM32\\METHODS\\JWQ20121205.M
Last changed: 2014-9-6 16:45:33 by ckq
(modified after loading)
Sample Info: OD-H R/I=50:10 1.0m/1min 250nm

VWD1 A, Wavelength=250 nm (GZH\000083.D)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=250 nm

Peak RetTime Type Width Area Height Area%
# [min] [min] nAU *% [nAU ] %
---|--------|--------|---------|--------|--------|
1  9.975 BB  0.416  379.36035  14.15743  1.1670
2 32.975 BB  1.3427  3.21271e4  346.83667  98.8330
Totals: 3.25064e4 360.99910

*** End of Report ***
Sample Name: gzh-294f

Acq. Operator : gzh
Acq. Instrument : Instrument 1  Location : Vial 1
Injection Date : 2014-6-30 19:24:39
Acq. Method : C:\CH32\METHODS\JWQ20121205.M
Last changed : 2014-6-30 18:33:42 by gzh
(modified after loading)
Analysis Method : C:\CH32\METHODS\JWQ20121205.M
Last changed : 2014-9-6 16:47:40 by ckq
(modified after loading)
Sample Info : COD-H H/I=99:10 1.0ml/min 250nm

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with TSTDs

Signal 1: VWD1 A, Wavelength=250 nm

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 9.815 VUV</td>
<td>0.4194</td>
<td>0.08678e4</td>
<td>404.61176</td>
<td>50.4017</td>
<td></td>
</tr>
<tr>
<td>2 34.871 BB</td>
<td>1.4438</td>
<td>1.07340e4</td>
<td>116.28735</td>
<td>49.5983</td>
<td></td>
</tr>
</tbody>
</table>

Totals : 2.16419e4 520.89911

*** End of Report ***
Acq. Operator : gzh
Acq. Instrument : Instrument 1
Injection Date : 2014-6-30 17:33:50
Last changed : 2014-6-30 17:30:49 by gzh
(modified after loading)
Analysis Method : C:\CHEMS3\METHODS\JNQ20121205.M
Last changed : 2014-6-6 16:46:19 by ckq
(modified after loading)
Sample Info : 0.5 mL/min 1.0 mAU/min 250 mm

Area Percent Report

Signal 1: VWD1 A, Wavelength=250 nm

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Width</th>
<th>Height</th>
<th>Area</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>[min]</td>
<td>[min]</td>
<td>mAU *s</td>
<td>[mAU ]</td>
</tr>
<tr>
<td>1 9.619 Vb</td>
<td>0.5981</td>
<td>543.0852</td>
<td>15.4510</td>
<td>2.6534</td>
</tr>
<tr>
<td>2 33.599 Hz</td>
<td>1.4865</td>
<td>3.23039e4</td>
<td>328.1005</td>
<td>98.3466</td>
</tr>
</tbody>
</table>

Totals : 3.28469e4 343.8515

*** End of Report ***
Sample Name: gzh-293b

Acq. Operator : gzh
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 2014-6-26 19:08:57
Acq. Method : C:\CHEM32\METHODS\JWQ20121205.M
Last changed : 2014-6-26 20:30:56 by gzh
(modified after loading)
Analysis Method : C:\CHEM32\METHODS\JWQ20121205.M
Last changed : 2014-9-6 16:38:02 by ckq
(modified after loading)
Sample info : Q/H H/t=90:10 1.0ml/min 250nm

==================================================================================================================================
Area Percent Report
==================================================================================================================================

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=250 nm

<table>
<thead>
<tr>
<th>Peak RetTime Type Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12.470</td>
<td>0.4540</td>
<td>2394.19214</td>
</tr>
<tr>
<td>2</td>
<td>61.628</td>
<td>2.1336</td>
<td>2452.36699</td>
</tr>
</tbody>
</table>

Totals : 4846.55713 98.47033

==================================================================================================================================

*** End of Report ***
Sample Name: gzh-293a

---

Acq. Operator : gzh
Acq. Instrument : Instrument 1
Injection Date : 2014-6-26 17:51:33
Acq. Method : C:\CHEM32\METHODS\WQ20121205.M
Last Changed : 2014-6-26 17:52:06 by gzh
(modified after loading)
Analysis Method : C:\CHEM32\METHODS\WQ20121205.M
Last changed : 2014-9-5 16:39:02 by ckq
(modified after loading)
Sample Info : 00-M III-90110 1.0ML/Min 250nm

---

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VMD1 A, Wavelength=250 nm

<table>
<thead>
<tr>
<th>Peak</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>12.585</td>
<td>BB</td>
<td>0.466</td>
<td>213.54277</td>
<td>6.43359</td>
<td>2.4870</td>
</tr>
<tr>
<td>2</td>
<td>63.029</td>
<td>BB</td>
<td>2.0018</td>
<td>8372.88426</td>
<td>56.84404</td>
<td>97.5130</td>
</tr>
</tbody>
</table>

Totals : 8586.40703 62.27758

---

*** End of Report ***
Data File C:\CHEN32\DATA\GSH\000255.D
Sample Name: 248-2948 C-2 racemic

Acq. Operator  :  gsh
Acq. Instrument :  Instrument 1
Injection Date  :  2015-5-4 19:51:55
Acq. Method     :  C:\CHEN32\VSMETHODS\JMQ20121205.M
Last changed   :  2015-5-4 17:17:41 by gsh
(modified after loading)
Analysis Method :  C:\CHEN32\VSMETHODS\JMQ20121205.M
Last changed   :  2015-5-4 21:37:50 by gsh
(modified after loading)
Sample info    :  OD-H N1/1=M-191614 1 ml/min 254 nm

---

Area Percent Report
---

Sorted By  :  Signal
Multiplier  :  1.0000
Dilution    :  1.0000
Use Multiplier & Dilution Factor with IS7Ds

Signal 1: VWD1 A, Wavelength=254 nm

<table>
<thead>
<tr>
<th>RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>16.911</td>
<td>0.425</td>
<td>1.8353</td>
<td>44.922</td>
<td>152.93</td>
</tr>
<tr>
<td>18.018</td>
<td>0.475</td>
<td>1.8410</td>
<td>60.751</td>
<td>50.07</td>
</tr>
</tbody>
</table>

Totals : 3.6764, 1285.86932

---

*** End of Report ***

Instrument 1 2015-5-4 21:37:55  gsh
Page 1 of 1
Data File C:\CHEM321\DATA\GZH\000254.D
Sample Name: GZH-391C C-2

===============================================================================
<table>
<thead>
<tr>
<th>Acq. Operator</th>
<th>gzh</th>
<th>Location</th>
<th>Vial 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acq. Instrument</td>
<td>Instrument 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Injection Date</td>
<td>2015-5-4 19:22:03</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acq. Method</td>
<td>C:\CHEM321\METHODS\JMP20121205.M</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Last changed</td>
<td>2015-5-4 17:17:41 by gzh</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Analysis Method</td>
<td>C:\CHEM321\METHODS\JMP20121205.M</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Last changed</td>
<td>2015-5-4 21:41:23 by gzh</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample Info</td>
<td>00E61 E/H/M-50.614 1 ml/min 254 nm</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

===============================================================================

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VW01 A, Wavelength=254 nm

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Ares</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(min)</td>
<td>(mAU)</td>
<td>(mAU)</td>
<td>(mAU)</td>
</tr>
<tr>
<td>1 15.771 min</td>
<td>0.4000</td>
<td>72.91888</td>
<td>5.89385</td>
<td>0.6010</td>
</tr>
<tr>
<td>2 17.967 min</td>
<td>0.4970</td>
<td>1.2661046</td>
<td>404.47421</td>
<td>99.3990</td>
</tr>
</tbody>
</table>

Totals : 1.21339e4 407.36806

===============================================================================

*** End of Report ***
Data File C:\CHEM32\DATA\GSH\000285.D
Sample Name: gsh=393A

Acq. Operator : gsh
Acq. Instrument : Instrument 1
Location : Vial 1
Injection Date : 2015-5-19 21:34:44
Acq. Method : C:\CHEM32\\METHODS\JHQ20121205.M
Last changed : 2015-5-19 21:30:45 by gsh
(modified after loading)
Analysis Method : C:\CHEM32\\METHODS\JHQ20121205.M
Last changed : 2015-5-19 22:08:32 by gsh
(modified after loading)
Sample info : IC H/13=60:30:10 1 ml/min 254 nm

Area Percent Report

Sorted by : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISIDs

Signal 1: WVD1 A, Wavelength=254 nm

<p>| Peak RetTime Type Width Area Height Area |
|-----|-----|-----|-----|-----|</p>
<table>
<thead>
<tr>
<th></th>
<th>[min]</th>
<th>[min]</th>
<th>mAU *s</th>
<th>mAU</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12.987</td>
<td>0.4044</td>
<td>3.01796e4</td>
<td>1153.05273</td>
</tr>
<tr>
<td>2</td>
<td>17.421</td>
<td>0.5221</td>
<td>7028.93066</td>
<td>205.03394</td>
</tr>
<tr>
<td>Totals</td>
<td></td>
<td></td>
<td>3.72055e4</td>
<td>1358.08667</td>
</tr>
</tbody>
</table>

*** End of Report ***
Data File \C:\CHEM32\DATA\G1H\000279.D
Sample Name: gsh-S230C L1A1K4 racemic

Acq. Operator : gsh
Acq. Instrument : instrument 1
Location : Vial 1
Injection Date : 2015-5-19 11:18:15
Acq. Method : \C:\CHEM32\METHODS\JNQ20121208.M
Last changed : 2015-5-19 11:38:17 by gsh
(modified after loading)
Analysis Method : \C:\CHEM32\METHODS\JNQ20121208.M
Last changed : 2015-5-19 11:40:16 by gsh
(modified after loading)
Sample info : IA H/H<90:10 1.0 ml/min 254 nm

Area Percent Report

Sorted by : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISIDs

Signal 1: VWD1 A, Wavelength=254 nm
Peak RetTime Type Width Area Height Area
# [min] [min] mAU s [mAU] %
------------------------------------|----------------|----------------|----------------|
1 13.845 VV 0.4879 5816.49269 179.95856 49.0179
2 15.271 VB 0.5361 6051.63672 166.74712 50.9821
Totals : 1.1870e4 346.7067

*** End of Report ***