Supporting Information

Asymmetric Hydrogenation of α-Hydroxy Ketones with Iridium/f-Amphox Catalyst: Efficient Access to Chiral 1,2-Diols

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I. General Remarks

All reactions and manipulations which are sensitive to moisture or air were performed in an argon-filled glovebox or using standard Schlenk techniques. Hydrogen gas (99.999%) was purchased from Shanghai Regulator Factory Co., Ltd. Simple ketones, were purchased from Aldrich or Alfa Aesar chemical company, and they were further purified by distilled. Anhydrous THF, 1,4-dioxane and toluene was distilled from sodium benzophenone ketyl. Anhydrous \textit{i}-PrOH, EA, CH$_2$Cl$_2$ were freshly distilled from calcium hydride. Anhydrous MeOH and EtOH were freshly distilled from Mg. Anhydrous CF$_3$CH$_2$OH were purchased from Sigma-Aldrich. Solvents were transferred by syringe. \([\text{Ir(COD)Cl}]_2\) was prepared according to the literature. \(\text{1H, 13C and } 31\text{P NMR spectra were recorded with a Bruker ADVANCE III (400 MHz) spectrometer with CDCl}_3 \text{ as the solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts are reported in parts per million (ppm, } \delta \text{ scale) downfield from TMS at 0.00 ppm and referenced to the CDCl}_3 \text{ at 7.26 ppm (for } \text{1H NMR) or 77.0 ppm (for } \text{13C NMR). Data are reported as: multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant in hertz (Hz) and signal area integration in natural numbers. } \text{13C NMR and } 31\text{P NMR analyses were run with decoupling. Optical rotations } [\alpha]_D \text{ were determined using a PERKIN ELMER polarimeter 343 instrument. GC analyses were performed using SHIMADZU Lab Solution instrument. HPLC analyses were performed using Daicel chiral column. Aliphatic } \alpha\text{-hydroxy ketones were purchased from Sigma-Aldrich and all the aromatic } \alpha\text{-hydroxy ketones were prepared according the literature.} \text{The characterization data of compounds } 2a-2c, 2e, 2j, 2l, 2n, 2o, 2r \text{ are in accordance with the reported data in the literature.}\) The characterization data of compounds \(2d, 2g, 2i, 2q, 2m \text{ are in accordance with the reported data in the literature.}\) The characterization data of compounds \(2f, 2h, 2k, 2p, 2s \text{ are in accordance with the reported data in the literature.}\)

II. General procedure for asymmetric hydrogenation

General procedure for S/C = 10,000: To a 4.0 mL vial was added the catalyst precursor \([\text{Ir(COD)Cl}]_2 \text{ (1.4 mg, 2.0×10}^{-3} \text{ mmol), ligand L3 (2.4 mg, 4.2×10}^{-3} \text{ mmol) and anhydrous } \text{PrOH (2.0 mL) under argon atmosphere. The mixture was stirred for 2.0 h at 25 }^\circ\text{C giving orange red solution in the argon-filled glovebox. The resulting solution (10 } \mu\text{L) and a solution of } \text{K}_2\text{CO}_3 \text{ (10 } \mu\text{L, } c = 0.02 \text{ mmol/mL) transferred by syringe into a 5.0 mL vial charged with fresh distilled substrate ketones (0.2 mmol) in 1.0 mL anhydrous } \text{PrOH. The vials were transferred to an autoclave, which was then charged with 20 atm of } \text{H}_2 \text{ and stirred at room temperature for 2 h. The hydrogen gas was released slowly in a well-ventilated hood and the solution was concentrated and passed through a short column of silica gel to remove the metal complex. The product was analyzed by chiral GC or chiral HPLC for ee values. The characterization data of compounds } 2a-2s \text{ are in accordance with the reported data in the literature.}\)
(S)-1-Phenyl-1,2-ethanediol 2a

Colorless solid, 27.4 mg, 99% yield; >99% ee; [α]D25 = +34.8 (c = 0.50, MeOH). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 220 nm; tR(R) = 33.61 min (minor), tR(S) = 37.82 min (major). 1H NMR (400 MHz, CD3OD) δ 7.36-7.24 (m, 4H), 7.23-7.17 (m, 1H), 4.64 (d, J = 7.1, 5.0 Hz, 1H), 3.57 (dd, J = 6.1, 2.6 Hz, 2H); 13C NMR (101 MHz, CD3OD) δ 141.91, 127.88, 127.15, 126.03, 74.59, 67.39.

(S)-1-(2-Methylphenyl)-1,2-ethanediol 2b

Colorless solid, 30.1 mg, 99% yield; 99% ee; [α]D25 = +33.4 (c = 0.35, CHCl3). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 220 nm; tR(R) = 29.23 min (minor), tR(S) = 39.62 min (major). 1H NMR (400 MHz, d-DMSO) δ 7.40 (d, J = 4.0 Hz, 1H), 7.19-7.06 (m, 3H), 5.14 (d, J = 4.0 Hz, 1H), 4.76-4.74 (m, 1H), 3.39 (dt, J = 9.4, 3.8 Hz, 1H), 3.34-3.30 (m, 1H), 2.28 (s, 3H); 13C NMR (101 MHz, d-DMSO) δ 141.84, 134.84, 130.16, 126.99, 126.46, 126.06, 71.0, 67.04, 19.28.

(S)-1-(4-Methylphenyl)-1,2-ethanediol 2c

Colorless solid, 30.1 mg, 99% yield; >99% ee; [α]D25 = +57.8 (c = 0.225, CHCl3). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 210 nm; tR(R) = 29.15 min (minor), tR(S) = 33.59 min (major). 1H NMR (400 MHz, CD3OD) δ 7.26 (d, J = 8.0 Hz, 2H), 7.16 (d, J = 8.0 Hz, 2H), 4.70-4.62 (m, 1H), 3.64-3.57 (m, 2H), 2.33 (s, 3H). 13C NMR (101 MHz, CD3OD) δ 138.82, 136.85, 128.48, 125.97, 74.45, 67.38, 19.79.

(S)-1-(4-Methoxyphenyl)-1,2-ethanediol 2d

Colorless solid, 33.3 mg, 99% yield; >99% ee; [α]D25 = +63.8 (c = 0.24, CHCl3). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate
= 1.0 mL/min; UV detection at 220 nm; \( t_R(R) = 50.37 \text{ min (minor), } t_R(S) = 58.49 \text{ min (major). } \) \(^1\)H NMR (400 MHz, CD\textsubscript{3}OD) \( \delta \) 7.18 (d, \( J = 8.0 \) Hz, 2H), 6.78 (d, \( J = 8.0 \) Hz, 2H), 4.52 (t, \( J = 6.1 \) Hz, 1H), 3.67 (s, 3H), 3.48 (d, \( J = 8.0 \) Hz, 2H). \(^{13}\)C NMR (101 MHz, CD\textsubscript{3}OD) \( \delta \) 159.25, 133.88, 127.21, 113.24, 74.16, 67.31, 54.25.

(S)-1-(4-Ethylphenyl)-1,2-ethanediol 2e

![Structure](image)

Colorless solid, 32.9 mg, 99% yield; >99% ee; \([\alpha]_D^{25} = +20.2 \text{ (c = 0.50, MeOH). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 220 nm; } t_R(R) = 26.75 \text{ min (minor), } t_R(S) = 30.13 \text{ min (major). } \) \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 7.29 (d, \( J = 8.1 \) Hz, 2H), 7.20 (d, \( J = 8.1 \) Hz, 2H), 4.80 (dd, \( J = 8.0, 3.4 \) Hz, 1H), 3.80-3.59 (m, 2H), 2.65 (q, \( J = 7.6 \) Hz, 2H), 2.54 (brs, 1H), 2.17 (brs, 1H), 1.24 (t, \( J = 8.0 \) Hz, 3H). \(^{13}\)C NMR (101 MHz, CDCl\textsubscript{3}) \( \delta \) 144.22, 137.74, 128.10, 126.12, 74.60, 68.10, 15.62.

(S)-1-(3-Fluorophenyl)-1,2-ethanediol 2f

![Structure](image)

Colorless solid, 30.9 mg, 99% yield; >99% ee; \([\alpha]_D^{25} = +19.2 \text{ (c = 0.50, MeOH). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 220 nm; } t_R(R) = 28.39 \text{ min (minor), } t_R(S) = 32.53 \text{ min (major). } \) \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 7.34-7.29 (m, 1H), 7.12-7.08 (m, 2H), 7.01-6.96 (m, 1H), 4.81 (dd, \( J = 8.1, 3.2 \) Hz, 1H), 3.77-3.74 (m, 1H), 3.64-3.59 (m, 1H), 3.07 (brs, 1H), 2.54 (brs, 1H). \(^{13}\)C NMR (101 MHz, CDCl\textsubscript{3}) \( \delta \) 162.97 (d, \( J = 246.3 \) Hz), 143.11 (d, \( J = 6.9 \) Hz), 130.10 (d, \( J = 8.1 \) Hz), 121.62 (d, \( J = 2.9 \) Hz), 114.85 (d, \( J = 21.1 \) Hz), 113.08 (d, \( J = 22.1 \) Hz), 74.04 (d, \( J = 1.6 \) Hz), 67.90.

(S)-1-(4-Fluorophenyl)-1,2-ethanediol 2g

![Structure](image)

Colorless solid, 30.9 mg, 99% yield; >99% ee; \([\alpha]_D^{25} = +56.4 \text{ (c = 0.225, CHCl_3). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 210 nm; } t_R(R) = 31.60 \text{ min (minor), } t_R(S) = 35.53 \text{ min (major). } \) \(^1\)H NMR (400 MHz, CD\textsubscript{3}OD) \( \delta \) 7.30-7.26 (m, 2H), 6.97-6.93 (m, 2H), 4.57 (t, \( J = 8.0 \) Hz, 1H), 3.50-3.48 (m, 2H). \(^{13}\)C NMR (101 MHz,
(S)-1-(2-Chlorophenyl)-1,2-ethanediol 2h

Colorless solid, 34.2 mg, 99% yield; 95% ee; [α]D^25 = +32.4 (c = 0.50, MeOH). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R(R) = 24.34 min (minor), t_R(S) = 33.78 min (major). ¹H NMR (400 MHz, d-DMSO) δ 7.58-7.55 (m, 1H), 7.41-7.30 (m, 2H), 7.29-7.26 (m, 1H), 5.47 (d, J = 4.0 Hz, 1H), 4.92-4.87 (m, 1H), 3.51-3.47 (m, 1H), 3.36-3.26 (m, 1H). ¹³C NMR (101 MHz, d-DMSO) δ 140.82, 131.52, 129.26, 128.99, 128.80, 127.50, 71.02, 66.09.

(S)-1-(3-Chlorophenyl)-1,2-ethanediol 2i

Colorless solid, 34.2 mg, 99% yield; >99% ee; [α]D^25 = +50.8 (c = 0.24, CHCl₃). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R(R) = 33.02 min (minor), t_R(S) = 39.22 min (major). ¹H NMR (400 MHz, CD₃OD) δ 7.43 (s, 1H), 7.38-7.21 (m, 3H), 4.70-4.67 (m, 1H), 3.63-3.61 (m, 2H). ¹³C NMR (101 MHz, CD₃OD) δ 144.61, 133.77, 129.37, 127.07, 126.11, 124.46, 73.76, 67.10.

(S)-1-(4-Chlorophenyl)-1,2-ethanediol 2j

Colorless solid, 34.2 mg, 99% yield; >99% ee; [α]D^25 = +11.8 (c = 0.50, MeOH). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R(R) = 33.41 min (minor), t_R(S) = 37.63 min (major). ¹H NMR (400 MHz, CD₃OD) δ 7.36-7.25 (m, 4H), 4.64-4.61 (m, 1H), 3.59-3.51 (m, 2H). ¹³C NMR (101 MHz, CD₃OD) δ 140.91, 132.72, 127.89, 127.68, 73.75, 67.11.

(S)-1-(3-Bromophenyl)-1,2-ethanediol 2k
(S)-1-(4-Bromophenyl)-1,2-ethanediol 2l

Colorless solid, 42.6 mg, 98% yield; >99% ee; [α]D25 = +31.8 (c = 0.50, CHCl3). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 220 nm; tR(R) = 39.54 min (minor), tR(S) = 42.78 min (major). 1H NMR (400 MHz, CD3OD) δ 7.46–7.43 (m, 2H), 7.27–7.25 (m, 2H), 4.62–4.60 (m, 1H), 3.56–3.54 (m, 2H). 13C NMR (101 MHz, CD3OD) δ 141.41, 130.90, 128.02, 120.70, 73.79, 67.06.

(S)-1-(3,5-bis(trifluoromethyl)phenyl)-1,2-ethanediol 2m

Colorless solid, 54.3 mg, 99% yield; >99% ee; [α]D25 = +35.8 (c = 0.50, CHCl3). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 220 nm; tR(R) = 18.64 min (minor), tR(S) = 20.74 min (major). 1H NMR (400 MHz, CD3OD) δ 7.90 (s, 2H), 7.76 (s, 1H), 4.75 (t, J = 8.0 Hz, 1H), 3.57 (d, J = 8.0 Hz, 2H). 13C NMR (101 MHz, CD3OD) δ 143.03, 131.80 (q, J = 33.0 Hz), 126.27, 121.86, 99.99, 73.39, 67.58. 19F NMR (377 MHz, CDCl3) δ -62.89.

(S)-1-(1-Naphthyl)-1,2-ethanediol 2n

Colorless solid, 37.3 mg, 99% yield; 97% ee; [α]D25 = +35.8 (c = 0.50, MeOH). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 70:30; flow rate
= 0.5mL/min; UV detection at 254 nm; \( t_{R}(R) = 10.54 \text{ min (minor)} \), \( t_{R}(S) = 14.83 \text{ min (major)} \). \(^1\)H NMR (400 MHz, CD\textsubscript{3}OD) \( \delta \) 8.17 (d, \( J = 8.0 \text{ Hz, 1H} \)), 7.92-7.90 (m, 1H), 7.83-7.81 (m, 1H), 7.59 (d, \( J = 7.1 \text{ Hz, 1H} \)), 7.74-7.72 (m, 1H), 7.56-7.49 (m, 3H), 5.57-5.55 (m, 1H), 3.90-3.86 (m, 1H), 3.73-3.68 (m, 1H). \(^{13}\)C NMR (101 MHz, CD\textsubscript{3}OD) \( \delta \) 137.30, 133.83, 130.65, 128.47, 127.53, 125.62, 125.09, 125.01, 123.29, 122.60, 71.37, 67.02.

\((S)-1-(2-Naphtyl)-1,2-ethanediol 2o\)

\[
\text{Colorless solid, 37.3 mg, 99\% yield; 99\% ee; } [\alpha]_{D}^{25} = +13.6 \text{ (c = 0.50, MeOH). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 70:30; flow rate = 0.5 mL/min; UV detection at 254 nm; } t_{R}(R) = 11.18 \text{ min (minor)}, t_{R}(S) = 12.39 \text{ min (major).} \]

\(^1\)H NMR (400 MHz, CD\textsubscript{3}OD) \( \delta \) 7.87-7.84 (m, 4H), 7.48-7.46 (m, 3H), 4.91-4.83 (m, 1H), 3.75-3.72 (m, 2H).

\(^{13}\)C NMR (101 MHz, CD\textsubscript{3}OD) \( \delta \) 139.43, 133.39, 133.12, 127.53, 127.51, 127.24, 125.66, 125.38, 124.17, 74.65, 67.24.

\((R)-1-(2-Furyl)-1,2-ethanediol 2p\)

\[
\text{Colorless, 25.4 mg, 99\% yield; >99\% ee; } [\alpha]_{D}^{25} = +4.4 \text{ (c = 0.50, MeOH). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 95:5; flow rate = 0.8 mL/min; UV detection at 230 nm; } t_{R}(S) = 25.36 \text{ min (minor)}, t_{R}(R) = 29.82 \text{ min (major).} \]

\(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 7.37 (s, 1H), 6.34-6.29 (m, 2H), 4.79-4.77 (m, 1H), 3.87-3.79 (m, 2H), 3.74 (brs, 1H), 3.29 (brs, 1H).

\(^{13}\)C NMR (101 MHz, CDCl\textsubscript{3}) \( \delta \) 153.55, 142.32, 110.34, 107.02, 68.34, 65.04.

\((R)-1-(2-Thienyl)-1,2-ethanediol 2q\)

\[
\text{Colorless solid, 28.6 mg, 99\% yield; >99\% ee; } [\alpha]_{D}^{25} = +36.9 \text{ (c = 0.52, CHCl\textsubscript{3}). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 220 nm; } t_{R}(S) = 7.59 \text{ min (minor)}, t_{R}(R) = 9.02 \text{ min (major).} \]

\(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 7.27-7.25 (m, 1H), 6.99-6.97 (m, 2H), 5.01 (dd, \( J = 7.6, 3.6 \text{ Hz, 1H} \)), 3.81-3.68 (m, 2H), 3.68 (brs, 1H), 3.20 (brs, 1H).

\(^{13}\)C NMR (101 MHz, CDCl\textsubscript{3}) \( \delta \) 143.86, 126.89, 125.10, 124.46, 70.70, 67.69.

\((S)-1,2-Propanediol 2r\)
Colorless oil, 15.1 mg, 99% yield; 93% ee; $[\alpha]_D^{25} = +7.0$ (c = 0.50, MeOH). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 205 nm; $t_R(S) = 43.34$ min (major), $t_R(R) = 41.36$ min (minor). HPLC conditions (To the corresponding p-Toluenesulfonyl derivatives). $^1$H NMR (400 MHz, d-DMSO) $\delta$ 4.39-4.37 (m, 1H), 4.32 (d, $J = 4.5$ Hz, 1H), 3.50-3.38 (m, 1H), 3.20-3.11 (m, 1H), 3.07-3.03 (m, 1H), 0.89 (d, $J = 6.3$ Hz, 3H). $^{13}$C NMR (101 MHz, d-DMSO) $\delta$ 67.69, 67.64, 20.42.

(R)-1,2-Butanediol 2s

Colorless oil, 17.8 mg, 99% yield; 96% ee; $[\alpha]_D^{25} = +0.8$ (c = 0.28, MeOH). The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 205 nm; $t_R(S) = 28.2$ min (minor), $t_R(R) = 32.37$ min (major). HPLC conditions (To the corresponding p-Toluenesulfonyl derivatives). $^1$H NMR (400 MHz, CD$_3$OD) $\delta$ 3.41-3.22 (m, 3H), 1.45-1.43 (m, 1H), 1.32-1.26 (m, 1H), 0.86 (t, $J = 8.0$ Hz, 3H). $^{13}$C NMR (101 MHz, CD$_3$OD) $\delta$ 73.30, 65.60, 25.81, 8.94.

Asymmetric Hydrogenation of acetophenone at S/C = 1 000 000

To a 4 mL vial was added the catalyst precursor [Ir(COD)Cl]$_2$ (1.4 mg, 2.0×10$^{-3}$ mmol), ligand L3 (2.4 mg, 4.2×10$^{-3}$ mmol) and anhydrous ¹PrOH (2.0 mL) under argon atmosphere. The mixture was stirred for 2.0 h at 25 °C giving orange red solution in the argon-filled glovebox. The resulting solution (50 µL) and K$_2$CO$_3$ (13.8 mg) transferred by syringe into a 100 mL vial charged with recrystallized α-hydroxyacetophenone (100 mmol) in 25 mL anhydrous ¹PrOH. The vial was transferred to an autoclave, which was then charged with 50 atm of H$_2$ and stirred at room temperature for 24 h. The hydrogen gas was released slowly in a well-ventilated hood and the solution was concentrated and passed through a column of silica gel to remove the metal complex. The product (S)-1-Phenyl-1,2-ethanediol 2a was analyzed by chiral HPLC, >99% yield and >99% ee.
III. GC and HPLC spectra

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Acq. Operator : SYSTEM                   Seq. Line : 12
Acq. Instrument : 1260HPLC-DAD            Location : Vial 41
Injection Date : 12/15/2015 1:03:43 AM   Inj : 1
Inj Volume : 1.000 uL
Acq. Method : E:\DATA\2X8-2062X8-206\206\RAC-00| 2015-12-14 19-57-50\DAD-OD(1-2)-98-2-1.000-95055-50555.D
Last changed : 12/14/2015 9:13:46 PM by SYSTEM
Analysis Method : E:\DATA\2X8-2062X8-206\206\RAC-00| 2015-12-14 19-57-50\DAD-OD(1-2)-98-2-1.000-95055-50555.D (Sequence Method)
Last changed : 12/21/2015 12:50:11 PM by SYSTEM
(modified after loading)
Additional Info : Peak(s) manually integrated

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

Area Percent Report

=================================================================================================
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD 1D, Sig=220.4, Pef-off

Peak Ret Time Type Width Area Height Area
# [min] [min] [N/AUs] [N/Au] %
---|--------|--------|-----------|--------|
1 29.226 M 0.4300 17.38722 6.61577e-1 0.4149
Totals : 41.9063349 57.36381

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

*** End of Report ***

1260HPLC-DAD 1/21/2016 12:50:19 PM SYSTEM

Page 1 of 1
Data File E:\DATA\YF\YF-2-75\YF-2-75-DAD-00-95-5-1 2015-05-31 16-06-23\063-1901.D
Sample Name: WUL-P-CH3-OH

Acq. Operator : SYSTEM
Seq. Line : 19
Acq. Instrument : 1260HPLC-DAD
Location : Vial 63
Injection Date : 5/31/2015 10:38:59 PM
Inj. Volume : 5.000 µl

Acq. Method : E:\DATA\YF\YF-2-75\YF-2-75-DAD-00-95-5-1 2015-05-31 16-06-23\DAD-8D\98-2-ML-50MIL(1-2).X
Last changed : 5/31/2015 4:09:31 PM by SYSTEM
Analysis Method : E:\DATA\YF\YF-2-75\YF-2-75-DAD-00-95-5-1 2015-05-31 16-06-23\DAD-8D\98-2-ML-50MIL(1-2).X (Sequence Method)
Last changed : 12/6/2015 7:40:10 PM by SYSTEM
(modified after loading)

Additional Info : Peak(s) manually integrated

--- Area Percent Report ---

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal : DAD 1 C, Sig=2;0,4 Ref=off

<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>26.179</td>
<td>BB</td>
<td>0.6220</td>
<td>2.62215e4</td>
<td>473.53427</td>
<td>49.9787</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>30.634</td>
<td>BB</td>
<td>1.0955</td>
<td>2.62438e4</td>
<td>350.51547</td>
<td>35.0213</td>
<td></td>
</tr>
</tbody>
</table>

Totals : 5.24653e4 $24,069.74$

--- End of Report ---

1260HPLC-DAD 12/9/2015 7:40:16 PM SYSTEM
Acq. Operator : SYSTEM
Seq. Line : 15
Acq. Instrument : 1260HPLC-DAD
Location : Vial 44
Injection Date : 12/15/2015 3:38:21 AM
Inj. Volume : 1.000 μl
Acq. Method : E:\DATA\XEB-266\XEB-266(RAC-OD) 2015-12-14 19-57-50\DAD-OD(1-2)-98-2-1.OPL-205-250NM-40MIN.M
Last changed : 12/14/2015 8:13:44 PM by SYSTEM
Analysis Method : E:\DATA\XEB-266\XEB-266(RAC-OD) 2015-12-14 19-57-50\DAD-OD(1-2)-98-2-1.OPL-205-250NM-40MIN.M (Sequence Method)
Last changed : 12/15/2015 9:48:41 AM by SYSTEM
(modified after loading)
Additional Info : Peak(s) manually integrated

Signal 1: DAB1 C, Sig=2.10,4 Ref=off
Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*sec] [mAU] %
---- ---- ---- ---- ---- ---- ---- ----
1 29.110 MN 0.5399 29.93672 9.39864e-1 0.1175
2 33.567 EE 1.1571 2.54515e4 293.1012 99.8925
Totals : 2.5401e4 294.0410

*** End of Report ***
Sorted By Signal
Multiplier  1.0000
Dilution  1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD 0, Sig-220,4 Ref-off

Peak PeakTime Type Width Area Height Area
# [min] [min] [nAU*s] [nAU] [nAU]
---- ------ ------ --------- -------- -------
1 50.368 MN 1.1124 11.26849 1.668362 1 0.4512
2 50.486 MN 2.9108 2466.1293 17.93087 59.5488
Totals : 2497.3841 18.0991

*** End of Report ***
Acq. Instrument: LS20HPLC-DAD  
Injection Date: 9/22/2015 1:43:57 PM  
Volume: 1.000 μL  
Method: E:\DATA\OFF\UFFY-1-1701\20150921-ZXH-81  
Analysis Method: E:\DATA\OFF\UFFY-1-1701\20150921-ZXH-81  
Last changed: 9/22/2015 11:07:21 AM by SYSTEM  
Additional Info: Peaks normally integrated

---

Area Percent Report

Sorted By: Signal  
Multiplier: 1.0000  
Do not use Multiplier for Dilution Factor with ISTDs

Signal: DA01 A, Sig-210.4 Ret-off

Peak RetTime Type Width Area Height Area
# [min] [min] [min] [μAU] [μAU]
--- --- --- --- --- ---
1 25.913 BB 0.6348 4594.16797 169.6684 45.5386
2 29.224 BB 0.7202 4661.23632 97.4692 50.4684

Totals: 9275.40479 207.14677

--- End of Report ---
<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>26.749</td>
<td>NM</td>
<td>0.4236</td>
<td>17.2080</td>
<td>6.76984e+1</td>
<td>6.153</td>
</tr>
<tr>
<td>2</td>
<td>30.132</td>
<td>HD</td>
<td>0.8581</td>
<td>1.10626e+4</td>
<td>192.36777</td>
<td>99.845</td>
</tr>
</tbody>
</table>

Totals: 1.16998e+4 173.04475

--- End of Report ---

L2689PFC-DAD 12/13/2015 3:11:50 PM SYSTEM
Signal 1: DAD1 A, Sigm=20,4 Ref=off

Peak RetTime Type Width Area Height Area %
# [min] [min] [mAU*s] [mAU] [mAU] %
------ ---- ---- ---- ---- ---- ---- ---- ---- ----
1 26.966 EB 0.7436 1.4766624 297.22147 49.0024
2 33.640 EB 0.6449 1.485324 269.35419 50.1966

Totals :
2.96498e4 557.57565

*** End of Report ***
Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD 1 A, 3190-210, 4 Ref-off

| Peak RefTime Type Width Area Height Area % |
|-----|----------|------|--------|----------|------|
| 1   | 29.387   | MM   | 0.4601 | 7.50856 | 2.6733e-1 | 0.3045 |
| 2   | 32.526   | BB   | 0.6184 | 24589.4750 | 43.17194 | 99.6955 |
| Totals: | 2465.98366 | 43.43928 |

*** End of Report ***
Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAB C, Sig=2,0,4 Ref=off

Peak RetTime Type Width Area Height Area
# [min] [min] [nAU*s] [nAU] %
--- --- --- --- --- ---
1 31.595 MM 0.558 24.62478 7.35022e-1 0.3609
2 35.531 BR 1.018 6799.45313 90.47634 99.6391

Totals: 6824.07790 91.21136

*** End of Report ***
Data File: E:\DATA\WUL\XY-1-23\XY-1-23 2015-12-12 13-39-51\044-0601.D
Sample Name: WUL-2-o-c1--d1ol-rac

---

Acq. Operator : SYSTEM
Acq. Instrument : 1260HPLC-DAD
Injection Date : 12/12/2015 3:23:59 PM
Inj. Volume : 1.000 µL

Acq. Method : E:\DATA\WUL\XY-1-23\XY-1-23 2015-12-12 13-39-51\DAD-00\(1-2)-98-2-1.0ML-205-250MM-60MIN.M

Last changed : 12/12/2015 3:23:25 PM by SYSTEM
Analysis Method : E:\DATA\WUL\XY-1-23\XY-1-23 2015-12-12 13-39-51\DAD-00\(1-2)-98-2-1.0ML-205-250MM-60MIN.M (Sequence Method)

Additional Info : Peak(s) manually integrated

---

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD D, Sig=220,4 Ret=off

<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>25.702</td>
<td>0.050</td>
<td>1.242</td>
<td>100.000</td>
</tr>
<tr>
<td>2</td>
<td>36.328</td>
<td>1.431</td>
<td>1.000</td>
<td>49.698</td>
</tr>
</tbody>
</table>

Totals : 2.1654e4 257.42062

---

End of Report

---

1260HPLC-DAD 12/12/2015 8:48:15 PM SYSTEM
Acq. Operator : SYSTEM  
Acq. Instrument : LC20HPLC-DAD  
Injection Date : 2015-11-20 11:29:00 AM  
Inj Volume : 1.000 µL  
OSR-205-2500R-50MIN. H  
Last changed : 11/20/2015 10:50:22 PM by SYSTEM  
OSR-205-2500R-50MIN. H (Sequence Method)  
Last changed : 12/15/2015 10:34:37 AM by SYSTEM  
(modified after loading)  
Additional Info : Peak(s) manually integrated

---

**Axes Percent Report**

---

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Do not use Multiplier & Dilution Factor with ISSTDs

**Signal 1: DAD 1, Sig-205, 4 Ret-off**

**Peak Percent Type | Width | Area | Height | Area**

<table>
<thead>
<tr>
<th>#</th>
<th>[min]</th>
<th>[min]</th>
<th>[AU]²</th>
<th>[AU]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>24.33</td>
<td>MM</td>
<td>0.59</td>
<td>271.76</td>
</tr>
<tr>
<td>2</td>
<td>35.98</td>
<td>MM</td>
<td>1.00</td>
<td>1.18</td>
</tr>
</tbody>
</table>

Totals : 1.6579e+04 197.0141

*** End of Report ***

**LC20HPLC-DAD 12/15/2015 10:34:46 AM SYSTEM**
Acq. Operator : SYSTEM  Seq. Line : 7
Acq. Instrument : 1260HPLC-DAD  Location : Vial 75
Injection Date : 12/6/2015 11:37:37 PM  Inj : 1
Inj Volume : 1.000 μl
Acq. Method : E:\DATA\WUL\XY-20151208-20\XY-20151208-20(D) 2015-12-08 19-45-04\DAD-OD (1-2)-98-2-1.0ML-265-250NM-50MIN.M
Last changed : 12/6/2015 9:36:56 PM by SYSTEM
Analysis Method : E:\DATA\WUL\XY-20151208-20\XY-20151208-20(D) 2015-12-08 19-45-04\DAD-OD (1-2)-98-2-1.0ML-265-250NM-50MIN.M (Sequence Method)
Last changed : 12/11/2015 10:00:02 AM by SYSTEM
(modified after loading)
Additional Info : Peak(s) manually integrated

[Graph showing chromatogram with peaks labeled (2l)-rac]

Signal 1: DAD D, Sig=220,4 Ref=off

Peak RetTime Type Width Area Height Area # [min] [min] [mAU] [%]
---- [min] [min] [mAU] [%]
1 32.613 BR 0.9388 1.86265e4 294.94156 49.8364 3
2 39.830 BR 1.1078 1.87480e4 227.90480 50.1636 2
Totals : 3.73753e4 522.34656

*** End of Report ***
Data File: E:\DATA\WL\XY-2015\220\XY-20151208-20(09) 2015-12-08 19-45-04\076-0801.D
Sample Name: XY-20-m-Cl

Acq. Operator: SYSTEM
Seq. Line: 0
Acq. Instrument: 1260HPLC-DAD
Location: Vial 76
Injection Date: 12/9/2015 12:28:31 AM
Inj: 1
Inj Volume: 1.000 µl
Acq. Method: E:\DATA\WL\XY-20151208-20\XY-20151208-20(09) 2015-12-08 19-45-04\DAD-0D
(1-2)-98-2-1.0M-205-250W-5MIN.M
Last changed: 12/6/2015 9:36:56 PM by SYSTEM
Analysis Method: E:\DATA\WL\XY-20151208-20\XY-20151208-20(09) 2015-12-08 19-45-04\DAD-0D
(1-2)-98-3-1.0M-205-250W-5MIN.M (Sequence Method)
Last changed: 12/13/2015 3:56:56 PM by SYSTEM
(modified after loading)
Additional Info: Peak(s) manually integrated

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAB 1, D Sig=220.4 Ref=off

Peak RetTime Type Width Area Height Area
# [min] [min] [aAU*2] [aAU] %
--- --|--|--|---|--|--|
1 33.018 NN 0.7659 109.41030 2.38079 0.4751
2 39.224 NN 1.3679 2.2519646 279.26505 99.5249

Totals: 2.3029064 201.64503

*** End of Report ***
Acq. Operator: SYSTEM  
Seq. Line: 11
Injection Date: 12/19/2015 5:02:42 PM  
Inj. Volume: 1.000 µL  
Acq. Method: E:\DATA\LXX90-ER-B2120151218 2015-12-19 10:59-42\DAD-06(1-2)-90-2-1.0ML-205-250HM-70PIX.N  
Last changed: 12/19/2015 11:42:07 AM by SYSTEM
Analysis Method: E:\DATA\LXX90-ER-B2120151218 2015-12-19 10:59-42\DAD-06(1-2)-90-2-1.0ML-205-250HM-70PIX.N (Sequence Method)  
Last changed: 12/19/2015 11:42:07 AM by SYSTEM (modified after loading)
Additional Info: Peak(s) manually integrated

---

AXIS PERCENT REPORT

---

Sorted By: Signal  
Multiplier: 1.0000  
Dilution: 1.0000  
Do not use Multiplier & Dilution Factor with INAlDs

Signal 1: DAD1 8, Sig=205,4 Ref-off

Peak RetTime Type Width Area Height Area
# [min] [min] [kAU's] [kAU] [%]
--- --- --- --- --- ---
 1 36.617 BS 1.0250 3.62355 4 486.48805 49.7070
 2 46.417 BS 1.5001 3.66637 4 373.55541 50.2930

Totals: 7.25002e4 860.32346

---

End of Report ***

L280MIC-DAD 12/19/2015 2:32:55 PM SYSTEM
Acq. Operator : SYSTEM  |  Seq. Line : 20
Acq. Instrument : L260HPLC-DAD  |  Location : Vial 02
Injection Date : 12/24/2015 7:41:59 PM  |  Inj. Volume : 1.000 µl
Injection Time : 12/24/2015 8:30:00 AM
Analysis Method : L260HPLC-DAD 12/24/2015 8:30:00 AM 60min
Last changed : 12/24/2015 8:30:01 AM by SYSTEM
(modified after loading)

Additional Info : Peak(s) manually integrated

---

Axes Percent Report

---

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD C, Sig=210,4 Ref-off

<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>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[AU*S]</td>
<td>[AU]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>39.178</td>
<td>NN</td>
<td>0.7118</td>
<td>66.2319</td>
<td>1.4108</td>
<td>4.2220</td>
</tr>
<tr>
<td>2</td>
<td>45.835</td>
<td>HH</td>
<td>1.5403</td>
<td>2.969994</td>
<td>275.99332</td>
<td>99.7780</td>
</tr>
</tbody>
</table>

Totals : 2.71402e4 277.0246

---

*** End of Report ***
Acq. Operator: SYSTEM  Seq. Line: 9
Acq. Instrument: 1260HPLC-DAD  Location: Vial 47
Injection Date: 12/12/2015 5:00:43 PM  Inj: 1
Injection Volume: 1.000 µl
Acq. Method: E:\DATA\WL\XY-1-23\XY-1-23 2015-12-12 13-39-51\DAD-08\(1-2)-98-2-1.0ML-205-250MM-60MIN.M
Last changed: 12/12/2015 1:39:51 PM by SYSTEM
Analysis Method: E:\DATA\WL\XY-1-23\XY-1-23 2015-12-12 13-39-51\DAD-08\(1-2)-98-2-1.0ML-205-250MM-60MIN.M (Sequence Method)
Last changed: 12/12/2015 8:52:24 PM by SYSTEM
(modified after loading)
Additional Info: Peak(s) manually integrated

Signal 1: DAD C, Sig=210,4 Ref=off

<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>40.929</td>
<td>BR</td>
<td>1.0949</td>
<td>7029.29102</td>
<td>91.64509</td>
<td>49.8686</td>
</tr>
<tr>
<td>2</td>
<td>45.732</td>
<td>BR</td>
<td>1.2421</td>
<td>7966.34926</td>
<td>77.47664</td>
<td>50.1314</td>
</tr>
</tbody>
</table>

Totals:
1.40956e4 169.12173

*** End of Report ***

1260HPLC-DAD 12/12/2015 8:52:29 PM SYSTEM
Data File: E:\DATA\YC\YC-20150530\YC-RAC-0530 2015-05-30 21-53-45\088-1001.D
Sample Name: wul-d1-CF3-OH

Acq. Operator: SYSTEM
Seq. Line: 10
Acq. Instrument: 126HPLC-DAD
Location: Vial 06
Injection Date: 5/31/2015 2:30:44 AM
Inj: 1
Injection Volume: 5.000 µl

Last changed: 5/30/2015 10:38:28 PM by SYSTEM
Analysis Method: E:\DATA\YC\YC-20150530\YC-RAC-0530 2015-05-30 21-53-45\DAD-ODH-98-2-1ML-3MIN[1-2].M (Sequence Method)
Last changed: 12/6/2015 9:45:29 PM by SYSTEM
(modified after loading)

Additional Info: Peak(s) manually integrated

Area Percent Report

---

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DADl C, Sig=2.10,4 Ref=off

<table>
<thead>
<tr>
<th>Peak Type</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>BB</td>
<td>14.912</td>
<td>0.6045 s</td>
<td>5325.47266</td>
</tr>
<tr>
<td>2</td>
<td>BB</td>
<td>17.465</td>
<td>0.6486 s</td>
<td>5245.19385</td>
</tr>
</tbody>
</table>

Totals: 1.0517 µg 265.6725 µg

---

*** End of Report ***

126HPLC-DAD 12/9/2015 5:45:34 PM SYSTEM
Area Percent Report

Signal 1: DAB1C, Sig=310,4 Ref=off
Peak RetTime Type Width Area Height Area
  #  [min]  [min]  [mAU*s]  [mAU]  
  1  10.751  YV  0.2765  2.0994e4  1355.38237  45.4756
  2  16.562  HH  0.9230  2.8846e4  919.91034  50.8424
  Totals:  5.0541e+4  2279.29071

*** End of Report ***
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DA01 B, Sig=254.4 Ref=off

<table>
<thead>
<tr>
<th>Peak Ret Time Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU²]</td>
<td>[mAU]</td>
</tr>
<tr>
<td>1</td>
<td>11.428</td>
<td>BB</td>
<td>0.2963</td>
<td>1137.25781</td>
</tr>
<tr>
<td>2</td>
<td>12.910</td>
<td>BB</td>
<td>0.3399</td>
<td>1141.6985</td>
</tr>
</tbody>
</table>

Totals :
2678.95667 129.50200

*** End of Report ***

1260HPLC-DAD 12/12/2015 9:44:24 PM SYSTEM
Signal 1: DAB 1 A, Sig=254,4 Ref=off

Peak RetTime Type Width Area Height Area
# [min] [min] [nAU*s] [nAU] %
--- --- --- --- --- --- --- --- --- --- --- --- --- ---
1 11.178 MH 0.1897 12.47672 1.09637 0.5572
2 12.385 MH 0.3240 2226.56763 114.23113 99.4428

Totals:
2239.04434 115.32750

Area Percent Report

0
10
20
30
40
50
60
70
80
90
100

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

Signal(s) manually integrated

Additional Info: Peak(s) manually integrated

Sort by: Signal
Multiplier: 1.0000
Dilution: 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

*** End of Report ***
Data File: E:\DATA\WFY\WFY-1-170\DX20150921-2X8-01 2015-09-22 11-07-21\063-0801.D
Sample Name: WUL-FYAN-RAC

Acq. Operator: SYSTEM
Seq. Line: 0
Acq. Instrument: 1260HPLC-DA
Location: Vial 63
Injection Date: 9/22/2015 12:35:52 PM
Injection Volume: 1.000 μl
Last changed: 9/22/2015 11:24:11 AM by SYSTEM
Analysis Method: E:\DATA\WFY\WFY-1-170\DX20150921-2X8-01 2015-09-22 11-07-21\DAD-OD\D1-2-95-5-0.8M-210MM-254MM-50MIN.M
Last changed: 12/11/2015 10:46:41 AM by SYSTEM
(modified after loading)
Additional Info: Peak(s) manually integrated

---

Signal 1: DAD1 A, Sig=230,4 Ref=off

Peak RetTime Type Width Area Height Area
# [min] [min] [nA] [nA] [nA]
---|---|---|---|---|---|
1 24.000 BR 0.7753 3085.60278 57.08909 49.8095
2 28.401 BR 0.9044 3978.96948 50.14202 50.1905

Totals:
6134.57227 107.15011

---

*** End of Report ***

1260HPLC-DA 12/11/2015 10:46:46 AM SYSTEM
Signal 1: DAB A, Sig=230.4 Ref=off

Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*sec] [mAU] %  
1 25.363 MIN 1.1004 48.39669 7.32993e-1 0.3545
2 29.819 BB 1.0627 1.3603664 177.35224 99.6455

Totals: 1.3552064 170.12624

*** End of Report ***
Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal: DAD 1 B, Sig-210,4 Ref-off

Peak RetTime Type Width Area Height Area

<table>
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<tr>
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<th>[min]</th>
<th>[mAU*s]</th>
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<td>1</td>
<td>9.174</td>
<td>0.3088</td>
<td>7663.2094</td>
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<td>10.685</td>
<td>0.8597</td>
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<td>326.6755</td>
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Totals: 1.5556e+4 609.91280

End of Report
Data File: E:\DATA\UWL\XY-20151202-17\XY-20151202-17 2015-12-02 15-57-33\081-0001.D
Spread Name: wv1-2-199-1

Acq. Operator: SYSTEM
Seq. Line: 6
Acq. Instrument: 1260HPLC-DAD
Location: Vial 01
Injection Date: 12/2/2015 15:49:23 PM
Injection Volume: 1.000 µL

Acq. Method: E:\DATA\UWL\XY-20151202-17\XY-20151202-17 2015-12-02 15-57-33\DAD-08(1-2)
Last changed: 12/2/2015 15:39:03 PM by SYSTEM
Analysis Method: E:\DATA\UWL\XY-20151202-17\XY-20151202-17 2015-12-02 15-57-33\DAD-08(1-2)
Last changed: 12/30/2015 12:39:51 PM by SYSTEM
(modified after loading)

Additional Info: Peaks equally integrated

---

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Do not use Multiplier & Dilution Factor with ISSTs

Signal 1: DA01, SIG=220p4 Ret-off

<table>
<thead>
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<th>Type Width</th>
<th>Area</th>
<th>Height</th>
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<td>[AU]</td>
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<td>0.9643</td>
<td>0.01836644</td>
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<td>49.3577</td>
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<tr>
<td>2</td>
<td>1.0989</td>
<td>1.0419364</td>
<td>139.02203</td>
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Totals: 2.05029e+4 232.44112

---

*** End of Report ***
Signal 1: DAD1 B, SIG=205.4 Ret-off

Peak RetTime Type Width Area Height Area
# [min] [min] [AUC\mu g] [AUC] q
----- ----- ------- ------ ------ ------ ------
1 41.356 0.9203 383.09811 8.93803 3.7211
2 42.339 1.3254 9912.14844 123.7126 96.2789

Totals:
1.02952e4 130.5656

*** End of Report ***

1260HPLC-DAD 12/19/2015 2:58:12 PM SYSTEM
---

**Acq. Operator:** SYSTEM  
**Seq. Line:** 7  
**Acq. Instrument:** 1280HPLC-DAD  
**Location:** Vial 02  
**Injection Date:** 12/3/2015 7:40:15 PM  
**Inj. Volume:** 1.000 μL  
**Acq. Method:** E:\DATA\UL\XY-20151202-17\XY-20151202-17 2015-12-02 15-57-33\DAD-OPID-2  
**Last changed:** 12/3/2015 4:53:35 PM by SYSTEM  
**Analysis Method:** E:\DATA\UL\XY-20151202-17\XY-20151202-17 2015-12-02 15-57-33\DAD-OPID-2  
**Last changed:** 1/21/2016 1:07:12 PM by SYSTEM  
**Additional Info:** Peak(s) manually integrated

---

**Axes Percent Report**

---

**Signal 1:** DAD 0, Sig-220,4 Ref-off  
**Multiplier:** 1.0000  
**Dilution:** 1.0000  
**Do not use Multiplier & Dilution Factor with ISTDs**

---

**Peak Percent Type**  
**Width**  
**Area**  
**Height**  
**Area**  
---  
1  
30.628  
8512.81739  
157.75935  
30.628  
2  
34.696  
8436.16496  
142.45229  
49.7798  
**Totals:**  
1.69510e4  
300.21634

---

*** End of Report ***

---

**1280HPLC-STD 1/21/2016 1:07:30 PM SYSTEM**  
Page 1 of 1
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<td>0.9228</td>
<td>1.73618e4</td>
<td>262.76511</td>
<td>99.0021</td>
</tr>
</tbody>
</table>

Totals: 1.91239e4 271.26337
IV. NMR spectra
V. References: