# Supporting Information for

# Rhodium-Catalyzed asymmetric hydrogenation of exocyclic α,β-Unsaturated Carbonyl Compounds

Jiaxin Yang, Xiuxiu Li, Cai You, Shuailong Li, Yuqing Guan, Hui Lv, Xumu Zhang

# **Table of Contents**

1. General information 1
2. General preparation of exocyclic $\alpha,\beta$ -Unsaturated carbonyl compounds1
3. General procedure for asymmetric hydrogenation of exocyclic $\alpha,\beta$ -unsaturated
carbonyl compounds
4. Procedure for the chiral piperdine <b>4</b> 15
5. References
6. <sup>1</sup> H NMR and <sup>13</sup> C NMR spectra of substrates and products17
7. HPLC spectra of substrates and products

#### **General information**

All the reactions dealing with air- or moisture-sensitive compounds were carried out in a dry reaction vessel under a positive pressure of nitrogen or in the nitrogen-filled glovebox. Unless otherwise noted, all reagents and solvents were purchased from commercial suppliers and used without further purification. NMR spectra were recorded on Bruker ADVANCE III (400 MHz) spectrometers for <sup>1</sup>H NMR and <sup>13</sup>C NMR. CDCl<sub>3</sub> was the solvent used for the NMR analysis, with tetramethylsilane as the internal standard. Chemical shifts were reported upfield to TMS (0.00 ppm) for <sup>1</sup>H NMR and relative to CDCl<sub>3</sub> (77.0 ppm) for <sup>13</sup>C NMR. Optical rotation was determined using a Perkin Elmer 343 polarimeter. HPLC analysis was conducted on an Agilent 1260 Series instrument. Column Chromatography was performed with silica gel Merck 60 (300-400 mesh).

#### General Preparation of exocyclic *a*,*β*-Unsaturated Carbonyl Compounds



The cyclic carbonyl compound (20 mmol) was dissolved in toluene (25 mL) and the solution was cooled to 0  $^{\circ}$ C, followed by the addition of corresponding aldehyde (10 mmol), the mixture was slowly charged to potassium *t*-butoxide (1.2 equiv.) under an ice/water bath. The mixture was heated to 55  $^{\circ}$ C and stirred therein for 1 h. The reaction mixture was concentrated to a slurry under vacuum, then water (100 mL) was added. The mixture was filtered, and the solid was washed with water and dried in vacuum oven overnight. The crude product was purified by recrystallization from ethyl acetate/petroleum ether or hexane.

# (*E*)-3-benzylidenetetrahydro-2H-pyran-2-one (1a)<sup>1</sup>

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.93 (t, J = 2.2 Hz, 1H), 7.53 – 7.30 (m,

5H), 4.49 – 4.31 (m, 2H), 2.90 – 2.86 (m, 2H), 2.00 – 1.94 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.99, 141.61, 134.91, 130.21, 129.18, 128.55, 125.71, 68.72, 25.91, 22.98.



#### (E)-3-(4-methylbenzylidene)tetrahydro-2H-pyran-2-one (1b)

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.89 (s, 1H), 7.35 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 4.53 – 4.28 (m, 2H), 2.90 – 2.86 (m, 2H), 2.38 (s, 3H), 2.06 – 1.91 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.26, 141.67, 139.58, 132.14, 130.36, 129.31, 124.66, 68.63, 25.99, 22.98, 21.42. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 225.0886, found 225.0883.



#### (E)-3-(4-(tert-butyl)benzylidene)tetrahydro-2H-pyran-2-one (1c)

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.91 (t, *J* = 2.2 Hz, 1H), 7.46 – 7.39 (m, 4H), 4.54 – 4.29 (m, 2H), 2.92 – 2.89 (m, 2H), 1.99 – 1.96 (m, 2H), 1.34 (s, 9H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.21, 152.64, 141.58, 132.15, 130.23, 125.54, 124.78, 68.64, 34.82, 31.15, 25.99, 22.99. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 267.1356, found 267.1358.



#### (*E*)-3-(4-methoxybenzylidene)tetrahydro-2H-pyran-2-one (1d)<sup>2</sup>

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.87 (t, *J* = 2.1 Hz, 1H), 7.43 (d, *J* = 8.8 Hz, 1H), 6.98 – 6.90 (m, 1H), 4.49 – 4.30 (m, 1H), 3.85 (s, 2H), 2.89 – 2.86 (m, 1H), 2.02 – 1.96 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.41, 160.34, 141.40, 132.23, 127.67, 123.10, 114.04, 68.51, 55.36, 26.03, 22.97.



# (*E*)-3-(4-fluorobenzylidene)tetrahydro-2H-pyran-2-one (1e)<sup>3</sup>

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.88 (s, 1H), 7.46 – 7.42 (m, 2H), 7.21 – 6.96 (m, 2H), 4.57 – 4.25 (m, 2H), 2.88 – 2.84 (m, 2H), 2.02 – 1.96 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  166.87, 162.89 (d, *J* = 250 Hz), 140.38, 132.21 (d, *J* = 9 Hz), 131.10 (d, *J* = 2 Hz), 125.36 (d, *J* = 2 Hz), 115.73 (d, *J* = 21 Hz), 68.66, 25.86, 22.91.



# (E)-3-(4-chlorobenzylidene)tetrahydro-2H-pyran-2-one (1f)<sup>4</sup>

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.86 (t, *J* = 2.3 Hz, 1H), 7.42 – 7.33 (m, 4H), 4.61 – 4.16 (m, 2H), 2.88 – 2.84 (m, 2H), 2.02 – 1.96 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.69, 140.16, 135.13, 133.29, 131.41, 128.82, 126.24, 68.69, 25.88, 22.86.



#### (E)-3-(4-(trifluoromethyl)benzylidene)tetrahydro-2H-pyran-2-one (1g)

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.92 (s, 1H), 7.67 (d, J = 8.2 Hz, 2H), 7.53 (d, J = 8.2 Hz, 2H), 4.69 – 4.21 (m, 2H), 2.90 – 2.86 (m, 2H), 2.03 – 1.97 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  166.34, 139.72, 138.31 (d, J = 2 Hz), 130.64 (d, J = 33 Hz), 130.16, 128.09, 125.45 (q, J = 4 Hz), 123.82 (q, J = 270 Hz), 68.85, 25.82, 22.86. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 279.0603, found 279.0603.



methyl (*E*)-4-((2-oxodihydro-2*H*-pyran-3(4H)-ylidene)methyl)benzoate (1h)<sup>5</sup> White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.07 (d, J = 8.4 Hz, 2H), 7.93 (t, J = 2.1 Hz, 1H), 7.49 (d, J = 8.3 Hz, 2H), 4.49 – 4.36 (m, 2H), 3.94 (s, 3H), 2.90 – 2.88 (m, 2H), 2.03 – 1.97 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl3)  $\delta$  166.46, 166.42, 140.21, 139.25, 130.28, 129.94, 129.70, 127.87, 68.79, 52.30, 25.95, 22.91.



#### (E)-3-(2-methylbenzylidene)tetrahydro-2H-pyran-2-one (1i)

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.03 (s, 1H), 7.31 – 7.18 (m, 4H), 4.50 – 4.30 (m, 2H), 2.74 – 2.70 (m, 2H), 2.32 (s, 3H), 1.96 – 1.90 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.67, 140.64, 137.66, 133.91, 130.33, 128.85, 128.56, 126.46, 125.49, 69.09, 25.40, 23.12, 19.95. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 225.0886, found 225.0886.



#### (E)-3-(2-fluorobenzylidene)tetrahydro-2H-pyran-2-one (1j)

White solid. <sup>1</sup>H NMR (400 MHz, CDCl3)  $\delta$  7.98 (s, 1H), 7.47 – 7.30 (m, 2H), 7.15 (m, 2H), 4.55 – 4.28 (m, 2H), 2.79 – 2.75 (m, 2H), 2.00 – 1.94 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  166.29, 160.57 (d, *J* = 250 Hz), 134.24 (d, *J* = 4 Hz), 130.96 (d, *J* = 8 Hz), 130.35 (d, *J* = 2 Hz), 128.25, 123.94 (d, *J* = 4 Hz), 122.85 (d, *J* = 14 Hz), 115.94 (d, *J* = 21 Hz), 69.06, 25.76 (d, *J* = 3 Hz), 22.95. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 229.0635, found 229.0634.



#### (E)-3-(3-methylbenzylidene)tetrahydro-2H-pyran-2-one (1k)

White solid.<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.89 (t, J = 2.1 Hz, 1H), 7.34 – 7.28 (m, 1H), 7.22 (dd, J = 26.4, 6.1 Hz, 3H), 4.49 – 4.27 (m, 2H), 2.91 – 2.87 (m, 2H), 2.38 (s, 3H), 2.00 – 1.94 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.05, 141.80, 138.20, 134.88, 130.95, 129.99, 128.42, 127.26, 125.50, 68.71, 25.95, 22.99, 21.45. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 225.0886, found 225.0883.



# (E)-3-(3-methoxybenzylidene)tetrahydro-2H-pyran-2-one (11)

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.89 (t, *J* = 2.1 Hz, 1H), 7.33 (t, *J* = 7.9 Hz, 1H), 7.03 (d, *J* = 7.7 Hz, 1H), 6.98 – 6.88 (m, 2H), 4.55 – 4.18 (m, 2H), 3.83 (s, 3H), 2.90 – 2.87 (m, 2H), 2.19 – 1.84 (m, 2H) <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  166.92, 159.46, 141.48, 136.18, 129.54, 126.00, 122.59, 115.70, 114.62, 68.72, 55.29, 25.92, 22.95. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 241.0835, found 241.0834.



#### (E)-3-(3-fluorobenzylidene)tetrahydro-2H-pyran-2-one (1m)

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.86 (t, J = 2.2 Hz, 1H), 7.39 (m, 1H), 7.21 (d, J = 7.8 Hz, 1H), 7.17 – 7.01 (m, 2H), 4.57 – 4.15 (m, 2H), 2.90 – 2.86 (m, 2H), 2.04 – 1.96 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  166.52, 162.47 (d, J = 245 Hz), 139.99 (d, J = 3 Hz), 136.84 (d, J = 8 Hz), 130.07 (d, J = 9 Hz), 126.97, 126.06 (d, J = 3 Hz), 116.44 (d, J = 22 Hz), 115.99 (d, J = 21 Hz), 68.69, 25.78, 22.78. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 229.0635, found 229.0632.



#### (E)-3-(naphthalen-2-ylmethylene)tetrahydro-2H-pyran-2-one (1n)

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.07 (t, *J* = 2.1 Hz, 1H), 7.91 (s, 1H), 7.85 (dd, *J* = 8.8, 5.7 Hz, 3H), 7.58 – 7.47 (m, 3H), 4.62 – 4.28 (m, 2H), 2.99 (m, 2H), 2.03 – 1.97 (m, 2H).<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.01, 141.69, 133.25, 132.99, 132.46, 130.35, 128.51, 128.18, 127.68, 127.22, 127.18, 126.64, 125.91, 68.73, 26.08, 23.03. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 261.0886, found 261.0886.



#### *tert*-butyl (*E*)-3-benzylidene-2-oxopiperidine-1-carboxylate (10)<sup>6</sup>

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.88 (t, *J* = 2.0 Hz, 1H), 7.39 (d, *J* = 4.4 Hz, 4H), 7.36 – 7.28 (m, 1H), 3.92 – 3.61 (m, 2H), 2.95 – 2.60 (m, 2H), 1.91 – 1.85 (m, 2H), 1.57 (s, 9H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.77, 153.18, 138.51, 135.67, 130.74, 130.00, 128.57, 128.40, 82.99, 45.98, 28.08, 26.27, 22.57.



*tert*-butyl (*E*)-3-(4-fluorobenzylidene)-2-oxopiperidine-1-carboxylate (1p)<sup>6</sup>

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.83 (s, 1H), 7.40 – 7.36 (m, 2H), 7.08 (t, *J* = 8.6 Hz, 2H), 3.84 – 3.69 (m, 2H), 2.77 (t, *J* = 5.7 Hz, 2H), 1.98 – 1.84 (m, 2H), 1.63 (s, 1H), 1.57 (s, 9H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.68, 162.63 (d, *J* =249 Hz), 153.15, 137.39, 131.93 (d, *J* =8 Hz), 131.82 (d, *J* =4 Hz), 130.49 (d, *J* =1 Hz), 115.56 (d, *J* =22 Hz), 83.10, 45.95, 28.11, 26.29, 22.57.



#### (*E*)-2-benzylidenecyclohexan-1-one (1q)<sup>6</sup>

Yellow solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.50 (t, *J* = 2.1 Hz, 1H), 7.43 – 7.35 (m, 4H), 7.34 – 7.30 (m, 1H), 2.86 – 2.82 (m, 2H), 2.54 (t, *J* = 6.7 Hz, 2H), 1.96 – 1.90 (m, 2H), 1.81 – 1.69 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 201.88, 136.66, 135.61, 135.59, 130.30, 128.53, 128.33, 40.34, 28.94, 23.87, 23.38.



#### (*E*)-3-benzylidenedihydrofuran-2(*3H*)-one (1r)<sup>6</sup>

Yellow solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.58 (t, J = 2.9 Hz, 1H), 7.52 – 7.41 (m, 5H), 4.47 (t, J = 7.3 Hz, 2H), 3.28 – 3.24 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  172.53, 136.67, 134.64, 130.00, 129.86, 128.94, 123.53, 65.45, 27.43.

# General Procedure for Asymmetric Hydrogenation of exocyclic $\alpha,\beta$ -Unsaturated Carbonyl Compounds

$$X \xrightarrow{n} R \frac{Rh(NBD)_2BF_4 (2 \text{ mol}\%), ZhaoPhos (2.2 \text{ mol}\%)}{H_2 (50 \text{ atm}), DCE, 35 ^{\circ}C, 24 \text{ h}} X \xrightarrow{n} R$$

In a nitrogen-filled glovebox, the Rh(NBD)<sub>2</sub>BF<sub>4</sub> (0.02 mmol) and ZhaoPhos (0.022 mmol) were dissolved in 1.0 mL anhydrous DCE, and the solution was stirred for 30 min at rt. Then the solution was equally divided into 10 vials charged with substrates (0.1 mmol) in anhydrous DCE solution (1.0 mL). The resulting vials were transferred to an autoclave, which was charged with 50 atm H<sub>2</sub>, and the reaction was stirred at 35  $\degree$  for 24h. When the reaction was completed, the H<sub>2</sub> was released slowly and the solution was passed through a short column of silica gel with EA. The solution was concentrated under reduced pressure to get the target compound. The chiral compounds were the analyzed by using HPLC on a chiral stationary phase to determine the ee.



# (*R*)-3-benzyltetrahydro-2H-pyran-2-one (2a)<sup>1</sup>

White solid; Isolated yield: 98%, 18.6 mg; 97% ee;  $[\alpha]_D^{25} = + 61.1$  (c =1, MeOH); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 97:3, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 20.0 min (major), 21.3 min (minor). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 – 7.29 (m, 2H), 7.26 – 7.18 (m, 3H), 4.31 – 4.24 (m, 2H), 3.51 – 3.21 (m, 1H), 2.83 – 2.58 (m, 2H), 1.96 – 1.85 (m, 2H), 1.81 (dd, *J* = 12.3, 6.1 Hz, 1H), 1.58 – 1.46 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  174.15, 138.93, 129.19, 128.54, 126.52, 68.57, 41.52, 37.18, 24.04, 21.91.



#### (*R*)-3-(4-methylbenzyl)tetrahydro-2H-pyran-2-one (2b)

White solid; Isolated yield: 98%, 19.9 mg; 99% ee;  $[\alpha]_D^{25} = +76.5$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 97:3, flow rate = 0.5 mL/min; UV detection at 210 nm; t<sub>R</sub> = 45.4 min (major), 50.1 min (minor).<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.16 – 7.05 (m, 4H), 4.53 – 4.02 (m, 2H), 3.45 – 3.12 (m, 1H), 2.79 – 2.55 (m, 2H), 2.33 (s, 3H), 1.97 – 1.85 (m, 2H), 1.83 – 1.74 (m, 1H), 1.58 – 1.44 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  174.23, 136.03, 135.76, 129.19, 129.04, 68.56, 41.55, 36.72, 23.98, 21.88, 21.03. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 227.1043, found 227.1038.



#### (*R*)-3-(4-(*tert*-butyl)benzyl)tetrahydro-2H-pyran-2-one (2c)

White solid. Isolated yield: 97%, 23.9 mg; 99% ee;  $[\alpha]_D^{25} = +76.5$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 97:3, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 16.8 min (major), 24.7 min (minor). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 (d, *J* = 8.3 Hz, 1H), 7.13 (d, *J* = 8.2 Hz, 1H), 4.39 – 4.15 (m, 1H), 3.35 (dd, *J* = 12.9, 3.1 Hz, 1H), 2.74 – 2.61 (m, 1H), 1.95 – 1.85 (m, 1H), 1.85 – 1.77 (m, 1H), 1.56 – 1.52 (m, 1H), 1.31 (s, 4H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  174.31, 149.34, 135.82, 128.84, 125.41, 68.55, 41.52, 36.64, 34.43, 31.39, 24.07, 21.89. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 269.1512, found 269.1512.



# (*R*)-3-(4-methoxybenzyl)tetrahydro-2H-pyran-2-one (2d)<sup>2</sup>

White solid; Isolated yield: 96%, 21.1 mg; 98% ee;  $[\alpha]_D^{25} = +71.6$  (c = 1, CH<sub>3</sub>Cl); The

enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 97:3, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 31.0 min (major), 33.7 min (minor). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.12 (d, *J* = 8.6 Hz, 1H), 6.84 (d, *J* = 8.7 Hz, 1H), 4.47 – 4.08 (m, 1H), 3.79 (s, 2H), 3.28 (d, *J* = 9.7 Hz, 1H), 2.85 – 2.59 (m, 1H), 1.96 – 1.85 (m, 1H), 1.84 – 1.80 (m, 1H), 1.57 – 1.46 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  174.21, 158.24, 130.86, 130.18, 113.89, 68.62, 55.28, 41.70, 36.30, 24.00, 21.93.



#### (*R*)-3-(4-fluorobenzyl)tetrahydro-2H-pyran-2-one (2e)<sup>3</sup>

White solid; Isolated yield: 93%, 19.4 mg; 97% ee;  $[\alpha]_D^{25} = -12.6$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 97:3, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 22.1 min (major), 24.4 min (minor). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.20 – 7.12 (m, 1H), 7.01 – 6.96 (m, 1H), 4.31 – 4.24 (m, 1H), 3.39 – 3.14 (m, 1H), 2.88 – 2.50 (m, 1H), 1.96 – 1.86 (m, 1H), 1.86 – 1.78 (m, 1H), 1.59 – 1.43 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  173.95, 161.64 (d, *J* = 243 Hz), 134.53 (d, *J* = 3 Hz), 130.64 (d, *J* = 8 Hz), 115.32 (d, *J* = 21 Hz), 68.58, 41.56, 36.32, 24.02, 21.92.

# ° C

#### (*R*)-3-(4-chlorobenzyl)tetrahydro-2H-pyran-2-one (2f)<sup>4</sup>

White solid; Isolated yield: 95%, 21.3 mg; 98% ee;  $[\alpha]_{25}^{D} = +45.3$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 97:3, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 23.3 min (major), 26.7 min (minor).<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.28 (t, *J* = 2.3 Hz, 1H), 7.25 (d, *J* = 2.5 Hz, 1H), 7.18 – 7.12 (m, 2H), 4.34 – 4.24 (m, 2H), 3.41 – 3.19 (m, 1H), 2.71 (m, 2H), 1.97 – 1.86 (m, 2H), 1.94 – 1.79 (m, 1H), 1.55 – 1.48 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  173.81, 137.36, 132.31, 130.57, 128.62, 68.56, 41.39, 36.46, 24.04,

21.91.



#### (*R*)-3-(4-(trifluoromethyl)benzyl)tetrahydro-2H-pyran-2-one (2g)

White solid; Isolated yield: 94%, 24.5 mg; 97% ee;  $[\alpha]_D^{25} = +31.1$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95:5, flow rate = 0.5 mL/min; UV detection at 210 nm; t<sub>R</sub> = 26.0 min (major), 28.6 min (minor). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.56 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 4.46 – 4.16 (m, 2H), 3.41 – 3.37 (m, 1H), 2.94 – 2.63 (m, 2H), 2.00 – 1.88 (m, 2H), 1.87 – 1.78 (m, 1H), 1.62 – 1.46 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  173.69, 143.14 (d, *J* = 1 Hz), 129.56, 128.88 (q, *J* = 33 Hz), 125.45 (q, *J* = 4 Hz), 124.22 (q, *J* = 270 Hz), 68.54, 41.26, 36.93, 24.14, 21.91. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 281.0760, found 281.0756.



#### methyl (R)-4-((2-oxotetrahydro-2H-pyran-3-yl)methyl)benzoate (2h)<sup>5</sup>

White solid; Isolated yield: 94%, 23.3 mg; 90% ee;  $[\alpha]_D^{25} = + 11.4$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 23.1 min (minor), 28.3 min (major). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.97 (d, J = 8.3 Hz, 2H), 7.28 (d, J = 8.2 Hz, 2H), 4.35 – 3.91 (m, 2H), 3.91 (s, 3H), 3.40 (dd, J = 12.8, 3.5 Hz, 1H), 2.84 – 2.71 (m, 2H), 1.94 – 1.86 (m, 2H), 1.86 – 1.77 (m, 1H), 1.58 – 1.48 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  173.67, 166.98, 144.46, 129.87, 129.26, 128.59, 68.55, 52.11, 41.27, 37.19, 24.19, 21.96.



#### (R)-3-(2-methylbenzyl)tetrahydro-2H-pyran-2-one (2i)

White solid; Isolated yield: 98%, 20.0 mg; 97% ee;  $[\alpha]_D^{25} = + 8.1$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralcel OJ-H column, hexane: isopropanol = 95:5, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 29.6 min (major), 32.6 min (minor). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.17 – 7.13 (m, 4H), 4.31 (t, J = 5.9 Hz, 2H), 3.66 – 3.31 (m, 1H), 2.69 – 2.60 (m, 2H), 2.34 (s, 3H), 1.95 – 1.87 (m, 2H), 1.86 – 1.75 (m, 1H), 1.61 – 1.49 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  174.25, 137.21, 136.34, 130.56, 129.90, 126.64, 125.95, 68.47, 40.31, 34.37, 24.17, 21.89, 19.53. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 227.1043, found 227.1042.



#### (R)-3-(2-fluorobenzyl)tetrahydro-2H-pyran-2-one (2j)

White solid; Isolated yield: 95%, 19.7 mg; 99% ee;  $[\alpha]_D^{25} = -4.4$  (c =1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralcel OJ-H column, hexane: isopropanol = 95:5, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 28.2 min (minor), 32.6 min (major). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.27 – 7.17 (m, 2H), 7.08 – 7.01 (m, 2H), 4.43 – 4.17 (m, 2H), 3.37 (q, *J* = 9.0 Hz, 1H), 2.88 – 2.66 (m, 2H), 1.91 (m, 2H), 1.87 – 1.78 (m, 1H), 1.55 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  174.06, 161.30 (d, *J* = 243 Hz), 131.67 (d, *J* = 5 Hz), 128.35 (d, *J* = 8 Hz), 125.83 (d, *J* = 15 Hz), 124.17 (d, *J* = 4 Hz), 115.31 (d, *J* = 22 Hz), 68.41, 40.28 (d, *J* = 1 Hz), 30.37 (d, *J* = 2 Hz), 23.92, 21.81. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 231.0792, found 231.0789.



#### (*R*)-3-(3-methylbenzyl)tetrahydro-2H-pyran-2-one (2k)

White solid; Isolated yield: 98%, 19.9 mg; 99% ee;  $[\alpha]_D^{25} = +91.8$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralcel OJ-H column, hexane: isopropanol = 95:5, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 30.7 min (minor), 38.7 min (major). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.19 (t, J = 7.5 Hz, 1H), 7.07 – 6.96 (m, 3H), 4.35 – 4.22 (m, 2H), 3.36 – 3.33 (m, 1H), 2.76 – 2.64 (m, 2H), 2.33 (s,

3H), 1.92 - 1.87 (m, 2H), 1.83 - 1.73 (m, 1H), 1.51 (dd, J = 16.0, 6.5 Hz, 1H). <sup>13</sup>C NMR (101 MHz, CDCl3)  $\delta$  174.27, 138.84, 138.13, 129.91, 128.39, 127.22, 126.15, 68.55, 41.48, 37.07, 24.02, 21.86, 21.41. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 227.1043, found 227.1041.



#### (R)-3-(3-methoxybenzyl)tetrahydro-2H-pyran-2-one (2l)

White solid; Isolated yield: 96%, 21.2 mg; 98% ee;  $[\alpha]_D^{25} = +65.7$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralpak AS-H column, hexane: isopropanol = 90:10, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 43.8 min (minor), 47.7 min (major).<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.22 (t, *J* = 7.8 Hz, 1H), 6.88 – 6.59 (m, 3H), 4.49 – 4.08 (m, 2H), 3.80 (s, 3H), 3.49 – 3.25 (m, 1H), 2.74 – 2.65 (m, 2H), 1.96 – 1.86 (m, 2H), 1.83 (dd, *J* = 11.4, 5.1 Hz, 1H), 1.60 – 1.44 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  174.13, 159.66, 140.51, 129.49, 121.50, 114.88, 111.67, 68.57, 55.17, 41.41, 37.19, 24.02, 21.87. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 243.0992, found 243.0987.



#### (*R*)-3-(3-fluorobenzyl)tetrahydro-2H-pyran-2-one (2m)

White solid; Isolated yield: 98%, 20.4 mg; 99% ee;  $[\alpha]_D^{25} = + 61.4$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralcel OJ-H column, hexane: isopropanol = 95:5, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 40.0 min (major), 49.0 min (minor).<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.27 – 7.20 (m, 1H), 7.03 – 6.83 (m, 3H), 4.32 – 4.25 (m, 3H), 3.35 (q, *J* = 8.5 Hz, 1H), 2.87 – 2.48 (m, 3H), 1.99 – 1.76 (m, 4H), 1.60 – 1.44 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  173.76, 162.87 (d, *J* = 245 Hz), 141.49 (d, *J* = 9 Hz), 129.98 (d, *J* = 8 Hz), 127.85 (d, *J* = 3 Hz), 116.02 (d, *J* = 21 Hz), 113.44 (d, *J* = 21 Hz), 68.52, 41.29, 36.88, 36.87, 24.10, 21.92. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 231.0792, found 231.0788.



#### (R)-3-(naphthalen-2-ylmethyl)tetrahydro-2H-pyran-2-one (2n)

White solid; Isolated yield: 98%, 23.5 mg; 99% ee;  $[\alpha]_D^{25} = +29.2$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralcel OJ-H column, hexane: isopropanol = 90:10, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 58.5 min (major), 65.3 min (minor).<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.83 – 7.78 (m, 3H), 7.64 (s, 1H), 7.55 – 7.39 (m, 2H), 7.34 (dd, *J* = 8.4, 1.7 Hz, 1H), 4.42 – 4.18 (m, 2H), 3.55 – 3.51 (dd, *J* = 13.2, 3.6 Hz, 1H), 2.99 – 2.74 (m, 2H), 1.96 – 1.83 (m, 2H), 1.83 – 1.73 (m, 1H), 1.62 – 1.52 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  174.11, 136.42, 133.47, 132.24, 128.23, 127.73, 127.66, 127.51, 127.40, 126.16, 125.57, 68.63, 41.48, 37.35, 24.11, 21.93. HRMS (ESI): [M+Na<sup>+</sup>] Calc. 263.1043, found 263.1040.



# tert-butyl (R)-3-benzyl-2-oxopiperidine-1-carboxylate (20)<sup>6</sup>

White solid; Isolated yield: 74%, 21.4 mg; 95% ee;  $[\alpha]_D^{25} = +33.7$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 97:3, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 12.5 min (major), 13.7 min (minor).<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.30 (d, *J* = 7.1 Hz, 1H), 7.23 – 7.18 (m, 3H), 3.77 – 3.65 (m, 1H), 3.61 – 3.54 (m, 1H), 3.42 (d, *J* = 9.8 Hz, 1H), 2.71 – 2.56 (m, 2H), 1.87 – 1.79 (m, 2H), 1.77 – 1.70 (m, 1H), 1.54 (s, 3H), 1.47 – 1.39 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  173.78, 152.97, 139.62, 129.26, 128.42, 126.28, 82.91, 45.77, 45.60, 37.08, 28.06, 25.27, 21.55.



(*R*)-1-((l1-methyl)(l1-oxidanyl)boranyl)-3-(4-fluorobenzyl)piperidin-2-one (2p)<sup>6</sup> White solid; Isolated yield: 73%, 22.5 mg; 91% ee;  $[\alpha]_D^{25} = +48.2$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95:5, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 9.5 min (major), 11.3 min (minor).<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.16 – 7.13 (m, 2H), 6.97 (t, J = 8.7 Hz, 2H), 3.74 – 3.67 (m, 1H), 3.69 – 3.57 (m, 1H), 3.36 – 3.33 (m, 1H), 2.65 – 2.59 (m, 2H), 1.85 – 1.79 (m, 2H), 1.77 – 1.72 (m, 1H), 1.71 – 1.62 (m, 1H), 1.54 (s, 9H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  173.56, 161.55 (d, J = 243 Hz), 152.91, 135.21 (d, J = 3 Hz), 130.69 (d, J = 8 Hz), 115.21 (d, J = 21 Hz), 82.97, 45.78, 45.65, 36.27, 28.07, 25.33, 21.60.



#### (R)-2-benzylcyclohexan-1-one $(2q)^6$

Yellow solid; Isolated yield: 98%, 18.4 mg; 91% ee;  $[\alpha]_D^{25} = + 31.2$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralcel OJ-H column, hexane: isopropanol = 99:1, flow rate = 1.0 mL/min; UV detection at 210 nm; t<sub>R</sub> = 12.9 min (minor), 14.3 min (major).<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.29 – 7.25 (m, 2H), 7.21 – 7.11 (m, 3H), 3.26 – 3.21 (m, 1H), 2.57-2.54 (m, 1H), 2.48 – 2.39 (m, 2H), 2.39 – 2.28 (m, 1H), 2.11 – 1.99 (m, 2H), 1.86 – 1.79 (m, 1H), 1.72 – 1.58 (m, 2H), 1.39 – 1.30 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  212.66, 140.40, 129.17, 128.32, 125.99, 52.51, 42.21, 35.49, 33.43, 28.09, 25.10.



#### (*R*)-3-benzyldihydrofuran-2(3H)-one (2r)<sup>6</sup>

White solid; Isolated yield: 93%, 16.4 mg; 79% ee;  $[\alpha]_D^{25} = +23.5$  (c = 1, CH<sub>3</sub>Cl); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 95:5, flow rate = 0.5 mL/min; UV detection at 210 nm; t<sub>R</sub> = 22.8 min (major), 25.4 min (minor).<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.31 (t, *J* = 7.2 Hz, 2H), 7.27 – 7.18 (m, 3H), 4.24 – 4.11 (m, 2H), 3.27 – 3.23 (m, 1H), 2.98 – 2.58 (m, 2H), 2.32 – 2.16 (m, 1H), 2.08 – 1.92 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  178.77, 138.42, 128.88, 128.71, 126.76, 66.58, 41.12, 36.12, 28.05.

**Procedure for the chiral piperdine 4**<sup>7</sup>



In a nitrogen-filled glovebox, the Rh(NBD)<sub>2</sub>BF<sub>4</sub> (2 mol%) and ZhaoPhos (2.2 mol%) were dissolved in anhydrous DCE (1 M), and the solution was stirred for 1h at rt. Then the benzylidenelactam **1p** (1.5g) was added. The resulting vial was transferred to an autoclave, which was charged with 70 atm H<sub>2</sub>, and the reaction was stirred at 35 °C for 48h. When the reaction was completed, the H<sub>2</sub> was released slowly and the solution was passed through a short column of silica gel with EA. The solution was concentrated under reduced pressure to get benzyllactam **2p** in 71% yield. Benzyllactam **2p** (1.68 g) was dissolved in a mixture of CH<sub>2</sub>Cl<sub>2</sub> (5 ml) and TFA (5 ml). The mixture was stirred at room temperature for 1 h and the evaporated *in vacuo*. The resulting residue was used for reduction. A dry round bottomed flask was charged with the residue followed by THF (2 M). The reaction flask was purged with N<sub>2</sub> for three times, then cooled to 0 °C. 30 minutes later, LiAlH<sub>4</sub> (1.0 M in THF, 2 eq) was added dropwise. The mixture was reflux for 12 h and quenched with water. The overall yield of piperidine **4** was 68.4 %.



#### (*R*)-3-(4-fluorobenzyl)piperidine (4)

colourless liquid. <sup>1</sup>H NMR (400 MHz, CDCl3)  $\delta$  7.08 (dd, J = 8.4, 5.5 Hz, 2H), 6.95 (t, J = 8.7 Hz, 2H), 3.05 (t, J = 12.9 Hz, 2H), 2.58 (t, J = 12.0 Hz, 1H), 2.50 – 2.44 (m, 2H), 2.33 (t, J = 11.2 Hz, 2H), 1.74 (m, 3H), 1.52 (d, J = 12.9 Hz, 1H), 1.08 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl3)  $\delta$  161.42 (d, J = 242 Hz), 135.28 (d, J = 3 Hz), 130.33 (d, J = 8 Hz), 115.09 (d, J = 21 Hz), 51.37, 46.02, 40.02, 37.94, 30.56, 25.20.

#### Reference

- 1. F. Tian, D. Yao, Y. Liu, F. Xie and W. Zhang, Adv. Synth. Catal. 2010, 352, 1841.
- 2. E. D. Beaulieu, L. Voss and D. Trauner, Org. Lett. 2008, 10, 869.
- 3. W. Zhang, F. Tian and D. Yao, Patent CN102212086.
- 4. M. Ferreira, T. B. Bisol, H. P. da Conceicao, T. V. C. Russo, A. J. Bortoluzzi and M. M. Sa, *Synthesis*, 2017, **49**, 667.
- 5. S. M. Raders and J. G. Verkade, J. Org. Chem. 2009, 74, 5417.
- 6. X. Liu, Z. Han, Z. Wang and K. Ding, Angew. Chem. Int. Ed. 2014, 53, 1978.
- T.-Y. Yue, D. D. McLeod, K. B. Albertson, S. R. Beck, J. Deerberg, J. M. Fortunak, W. A. Nugent, L. A. Radesca, L. Tang and C. D. Xiang, *Org. Process Res. Dev.* 2006, 10, 262-271.



# <sup>1</sup>H,<sup>13</sup>C NMR spectra of substrates and products





































4,45 4,44 4,44 7,290 7,290 7,290 7,203 7,203 1,97 1,97












































































7.57 7.57 7.57 7.57 7.557.55





 $\begin{array}{c} 7.99\\ 7.96\\$ 



























































## HPLC of substrates and products

## HPLC-2a-rac

Data File E:\DATA\WSW\YZL-1-101-102\YZL 2018-05-03 17-39-35\071-1001.D Sample Name: YJX-1-124-1-Rac



<sup>1260</sup>HPLC-DAD 5/4/2018 4:30:34 PM SYSTEM

#### HPLC-2a-cat

Data File E:\DATA\WSW\YZL-1-101-102\YZL 2018-05-03 17-39-35\072-1101.D Sample Name: YJX-1-124-1

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 11 Location : Vial 72 Injection Date : 5/3/2018 10:09:48 PM Inj: 1 Inj Volume : 3.000 µl : E:\DATA\WSW\YZL-1-101-102\YZL 2018-05-03 17-39-35\VWD-AD(1-2)-97-3-1ML-Acq. Method 3UL-210NM-60MIN.M Last changed : 5/3/2018 6:39:00 PM by SYSTEM Analysis Method : E:\DATA\WSW\YZL-1-101-102\YZL 2018-05-03 17-39-35\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M (Sequence Method) : 5/4/2018 4:34:55 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak (s) manually integrated VWD1A, Wavelength=210 nm (E:\DATAW SW\\YZL-1-101-102\\YZL 2018-05-03 17-39-35\072-1101.D) mAU 500 400 19.996 300 200 100 21.307 ο 12.5 17.5 7.5 10 20 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 19.996 BV 0.4266 9040.19434 323.95499 98.6751 2 21.307 VB 0.4797 121.37720 3.72931 1.3249 0.4797 121.37720 3.72931 9161.57153 327.68430 Totals : \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-DAD 5/4/2018 4:35:00 PM SYSTEM

55

## HPLC-2b-rac

Data File E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\043-0501.D Sample Name: YJX-1-124-2-R2

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-DAD Seq. Line : 5 Location : Vial 43 Injection Date : 5/4/2018 11:44:10 AM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\DAD-0D(1-2)-97-3-0.5ML-3UL-ALL-60MIN.M Last changed : 5/4/2018 11:21:21 AM by SYSTEM Analysis Method : E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\DAD-0D(1-2)-97-3-0.5ML-3UL-ALL-60MIN.M (Sequence Method) : 5/4/2018 4:09:57 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak (s) manually integrated DAD1A Sig=220.4 Re+380.100 (E:DATAYLXYLX:1.124YLX:1.1240504.2018.05.04.08.51.550043.0501.D) mAU О 700 600 -44.872 500 1421320 **1**9.916 400 300 -200 100 o 42 44 ஸ் 52 46 54min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=220,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 44.972 BB 1.0657 4.03312e4 507.19556 48.9077 2 49.916 MM 2.0120 4.21326e4 349.01953 51.0923 8.24638e4 856.21509 Totals : \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-DAD 5/4/2018 4:10:27 PM SYSTEM

#### HPLC-2b-cat

Data File E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\042-0401.D Sample Name: YJX-1-124-2

Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-DAD Seq. Line : - 4 Location : Vial 42 Injection Date : 5/4/2018 10:43:13 AM Inj: 1 Inj Volume : 3.000 µl : E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\DAD-0D(1-2)-97-Acq. Method 3-0.5ML-3UL-ALL-60MIN.M Last changed : 5/4/2018 11:21:21 AM by SYSTEM (modified after loading) Analysis Method : E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\DAD-0D(1-2)-97-3-0.5ML-3UL-ALL-60MIN.M (Sequence Method) : 5/4/2018 4:05:21 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak (s) manually integrated DAD1A Sig=220.4 Re= 380.100 (E:\DATAYKIXYIX-1.124\YIX-1.1240504.2018.05.04.08.51.550042.0401.D) mALL 0 46.357 400 300 200 100 Hand Siller 0 50 10 20 30 40 min Area Percent Report \_\_\_\_\_ Sorted By Signal : Multiplier 1.0000 : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=220,4 Ref=360,100 Peak RetTime Type Width Area Height Area [mAU\*s] # [min] [min] [mAU] ÷ 1 45.357 BB 1.0533 2.89318e4 367.97626 99.9820 2 50.126 MM 0.2078 5.22047 2.99043e-1 0.0180 Totals : 2.89371e4 368.27530 

1260HPLC-DAD 5/4/2018 4:05:33 PM SYSTEM

## HPLC-2c-rac

Data File E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\051-6101.D Sample Name: YJX-1-124-3-R2

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-DAD Seq. Line : 61 Location : Vial 51 Injection Date : 5/5/2018 9:12:41 PM Inj: 1 Inj Volume : 3.000 µl : E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\DAD-0D(1-2)-97-Acq. Method 3-1ML-3UL-210NM-60MIN.M Last changed : 5/5/2018 3:33:50 PM by SYSTEM Analysis Method : E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\DAD-0D(1-2)-97-3-1ML-3UL-210NM-60MIN.M (Sequence Method) : 5/10/2018 10:40:51 AM by SYSTEM Last changed (modified after loading) Additional Info : Peak (s) manually integrated DAD1 C. Sig=210.4 Re=360,100 (E:\DATA(YJX)YJX 1-124YJX 1-124-05042018-05-04-08-51-55'051-8101.D) mAU O 400 16.830 300 200 24,692 100 0 15 20 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 16.830 BB 0.4280 7988.02246 284.14053 50.6680 2 24.692 VB 0.8598 7777.39307 116.95254 49.3320 1.57654e4 401.09307 Totals : \*\*\* End of Report \*\*\*

1260HPLC-VWD 5/10/2018 10:41:00 AM SYSTEM

## HPLC-2c-cat

Data File E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\052-6201.D Sample Name: YJX-1-124-3

Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-DAD Seq. Line : 62 Location : Vial 52 Injection Date : 5/5/2018 10:13:35 PM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\DAD-0D(1-2)-97-3-1ML-3UL-210NM-60MIN.M Last changed : 5/5/2018 3:33:50 PM by SYSTEM Analysis Method : E:\DATA\YJX\YJX-1-124\YJX-1-124-0504 2018-05-04 08-51-55\DAD-0D(1-2)-97-3-1ML-3UL-210NM-60MIN.M (Sequence Method) : 5/10/2018 10:42:42 AM by SYSTEM Last changed (modified after loading) Additional Info : Peak (s) manually integrated DAD1 C. Sig=210.4 Re=360,100 (E:\DATA(YJX)YJX 1-124YJX 1-124-0504 2018-05-04-08-51-55'052-6201.D) mAU  $\cap$ 800 600 16.821 400 200 0 15 25 20 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 16.821 BB 0.4386 1.41313e4 486.95718 100.0000 Totals : 1.41313e4 486.95718 \*\*\* End of Report \*\*\*

1260HPLC-VWD 5/10/2018 10:42:53 AM SYSTEM

#### HPLC-2d-rac

Data File E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\083-0201.D Sample Name: YJX-1-124-6-R2

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 2 Location : Vial 83 Injection Date : 5/4/2018 4:38:23 PM Inj: 1 Inj Volume : 3.000 µl : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-Acq. Method 3-1ML-3UL-210NM-60MIN.M Last changed : 5/4/2018 4:21:45 PM by SYSTEM Analysis Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M (Sequence Method) : 5/4/2018 7:54:06 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated VWD1A,Wavelength=210 nm (E:\DATA\VJX\VJX +124-0504\VJX +1242018-05-04 16-21-444083-0201.D) mAU  $\cap$ 120 100 OMe 80 886.00 846 60 · 40 20 o 30 32 36 34 min Area Percent Report \_\_\_\_\_ Sorted By : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 30.988 BB 0.6696 2837.98364 64.33804 50.0257 2 33.476 BB 0.7179 2835.06323 59.39242 49.9743 5673.04688 123.73046 Totals : \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-VWD 5/4/2018 7:57:34 PM SYSTEM

## HPLC-2d-cat

Data File E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\084-0301.D Sample Name: YJX-1-124-6

Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 3 Location : Vial 84 Injection Date : 5/4/2018 5:39:08 PM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M Last changed : 5/4/2018 4:21:45 PM by SYSTEM Analysis Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M (Sequence Method) : 5/4/2018 8:02:28 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated VWD1A,Wavelength=210 nm (E\DATA\VJX\VJX 1.124-0504\VJX 1.1242018-05-0418-21-44084-0301.D) mAU റ 160 140 · OMe 8 120 · Б 100 -80 -60 40 N.OAA 20 -678 0 25 зΰ 10 15 35 20 min Area Percent Report \_\_\_\_\_ Sorted By Signal : Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area [mAU\*s] # [min] [min] [mAU\*s] [mAU] \* -!----| 1 31.021 BB 0.6737 4889.56299 109.55953 99.1676 2 33.676 MM 0.5666 41.04440 1.20737 0.8324 4930.60738 110.76690 Totals : \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-VWD 5/4/2018 8:02:34 PM SYSTEM

#### HPLC-2e-rac

Data File E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\081-4001.D Sample Name: YJX-1-124-9-Rac

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 40 Location : Vial 81 Injection Date : 5/5/2018 8:11:56 PM Inj: 1 Inj Volume : 10.000 µl Acq. Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-10UL-210NM-60MIN.M Last changed : 5/5/2018 4:23:57 PM by SYSTEM Analysis Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-10UL-210NM-60MIN.M (Sequence Method) : 5/6/2018 8:10:04 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak (s) manually integrated

 WWD1 A, W avelength=210 nm (E:\DATAYYJXY)XYX 1-124-0504YYJX 1-124-2018-05-04 16-21-444081-4001.D)
 mAU 140  $\cap$ 120 100 -ਸ਼ੇ 80 -60 -40 20 0 20 22 24 28 30 18 28 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 22.617 BB 0.6036 3412.14331 86.80087 50.7139 2 24.863 BB 0.7639 3316.08398 66.70898 49.2861 6728.22729 153.50985 Totals : \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-VWD 5/6/2018 8:10:10 PM SYSTEM

#### HPLC-2e-cat

Data File E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\082-4101.D Sample Name: YJX-1-124-9

Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 41 Location : Vial 82 Injection Date : 5/5/2018 9:12:45 PM Inj: 1 Inj Volume : 10.000 µl Acq. Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-10UL-210NM-60MIN.M Last changed : 5/5/2018 4:23:57 PM by SYSTEM Analysis Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-10UL-210NM-60MIN.M (Sequence Method) : 5/6/2018 8:13:16 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak (s) manually integrated

 VWD1A, Wavelength=210 nm (E:\DATAYYJXYJX 1-124-0524YJX 1-124-2018-05-04-18-21-44/082-4101.D)
 mAU 0 800 · 700 -126 600 -S. 500 -400 300 200 100 24.362 0 15 20 min Area Percent Report \_\_\_\_\_ Sorted By : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area # [min] [mAU\*s] [mAU] \* -1----1 1 22.126 BB 0.5443 1.86659e4 529.49677 98.4185 2 24.362 BB 0.7127 299.94257 6.05787 1.5815 1.89658e4 535.55463 Totals : \*\*\* End of Report \*\*\*

1260HPLC-VWD 5/6/2018 8:13:20 PM SYSTEM

## HPLC-2f-rac

Data File E:\DATA\LONGJIA0\LJ-3-117-1\LJ-3-117-1 2018-05-06 20-26-26\071-0801.D Sample Name: YJX-1-124-10-R2

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 8 Location : Vial 71 Injection Date : 5/7/2018 12:07:34 AM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\LONGJIAO\LJ-3-117-1\LJ-3-117-1 2018-05-06 20-26-26\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M Last changed : 5/6/2018 8:41:25 PM by SYSTEM Analysis Method : E:\DATA\LONGJIAO\LJ-3-117-1\LJ-3-117-1 2018-05-06 20-26-26\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M (Sequence Method) : 5/7/2018 3:58:49 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated WWD1A, W avelength=210 nm (E\DATA\LONGJIAC\LL3-117-1\LJ-3-117-1 2018-05-06 20-26-26'071-0801.D) 80 - $\cap$ 70 -CI , 1880 A 60 -23862 50 · 27.507 40 -30 20 10 ο - 10 15 20 25 30 10 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 23.852 MM 0.5440 1665.41882 51.02635 49.7081 2 27.507 BB 0.6541 1684.98132 38.76582 50.2919 3350.40015 89.79217 Totals : \*\*\* End of Report \*\*\*

1260HPLC-VWD 5/7/2018 3:58:56 PM SYSTEM

## HPLC-2f-cat

Data File E:\DATA\LONGJIA0\LJ-3-117-1\LJ-3-117-1 2018-05-06 20-26-26\072-0901.D Sample Name: YJX-1-124-10

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 9 Location : Vial 72 Injection Date : 5/7/2018 1:08:15 AM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\LONGJIAO\LJ-3-117-1\LJ-3-117-1 2018-05-06 20-26-26\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M Last changed : 5/6/2018 8:41:25 PM by SYSTEM Analysis Method : E:\DATA\LONGJIAO\LJ-3-117-1\LJ-3-117-1 2018-05-06 20-26-26\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M (Sequence Method) : 5/7/2018 4:04:06 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak (s) manually integrated VWD1A, Wavelength=210 nm (E:\DATA\LONGJIAO\LL\_3-117-1\Ll-3-117-1 2018-05-06 20-26-26'072-0901.D) 0 mAU 1200  $\cap$ CI 1000 23.307 800 600 400 200 26.712 0 10 15 5 20 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] \* - | - - - - - - - | 1 23.307 BB 0.5083 2.52120e4 759.48804 98.9540 2 26.712 BB 0.5482 266.49527 7.21090 1.0460 2.54785e4 766.69894 Totals : \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-VWD 5/7/2018 4:04:20 PM SYSTEM

## HPLC-2g-rac

Data File D:\DATA\LGY\LGY-2-73-RE-2\LGY-2-73-RE-2 2018-09-11 21-44-11\041-0701.D Sample Name: YJX-P-CF3



Instrument 1 9/12/2018 3:14:18 PM

## HPLC-2g-cat

Data File D:\DATA\YANG JIAXIN\YJX-P-CF3\LGY-2-73-RE-2 2018-09-12 09-55-24\042-0201.D Sample Name: YJX-P-CF3



Instrument 1 9/12/2018 3:10:23 PM

## HPLC-2h-rac

Data File D:\DATA\YANG JIAXIN\YJX-3-77\YJX-3-77-1218 2019-12-18 10-17-17\062-0301.D Sample Name: YJX-3-77-2



Instrument 1 12/22/2019 8:26:27 PM

#### HPLC-2h-cat

Data File D:\DATA\YANG JIAXIN\YJX-3-77\YJX-3-77-1218 2019-12-18 10-17-17\061-0201.D Sample Name: YJX-3-77-1



Instrument 1 12/22/2019 8:23:58 PM

## HPLC-2i-rac

Data File E:\DATA\YJX\YJX-1-105\YJX-1-105-2 2018-04-02 10-19-04\021-0601.D Sample Name: YJX-1-105-2-0J



# HPLC-2i-cat

Data File E:\DATA\WEF\XX-A-40\GYQA-180407-F-3-ME0-EE 2018-04-08 08-53-49\071-2501.D Sample Name: YJX-1-107-4

Acq. nptrator : 3131M Acq. Instrument : 1800HELO-DAD Injection Date : 4/9/2018 2:44:42 AM Inj Pituwe : 3.000 µl Acq. Method : E:\DATA\WEFYXX-A-0\GYQA-180407-F-3-HEO-EE 2018-04-08 08-53-49\ 6)-95-5-1ML-3UL-60HIN.M Analysis Method : E:\DATA\WEFYXX-A-40\GYQA-180407-F-3-HEO-EE 2018-04-08 08-53-49\ 6)-95-5-1ML-3UL-60HIN.M (Sequence Method) Last changed : 5/4/2018 4:45:33 PM by SYSTEM (aodified after loading) Additional Info : Peek (3) manually integrated DADIC Sig=204.Ref-300.00(EUATAWE_XX-4000YDA-180407-F-3-MEO-EE 2018-04-08 08-53-400'1-2010) meU additional Info : Peek (3) manually integrated DADIC Sig=204.Ref-300.00(EUATAWE_XA400YDA-180407-F-3-MEO-EE 2018-04-08 08-53-400'1-2010) meU additional Info : Peek (3) manually integrated DADIC Sig=204.Ref-300.00(EUATAWE_XA400YDA-180407-F-3-MEO-EE 2018-04-08 08-53-400'1-2010) meU additional Info : Peek (3) manually integrated DADIC Sig=204.Ref-300.00(EUATAWE_XA400YDA-180407-F-3-MEO-EE 2018-04-08 08-53-400'1-2010) meU additional Info : Peek (3) manually integrated Area Percent Report sorted By : Sigmal Multiplier : 1.0000 Dilution : 1.0000 Do not use Multiplier 4 Dilution Factor with ISTDs Sigmal 1: DADIC, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [main] [main] [main] [main] [main] [main] = [main] =	
Acq. Hold Mark 1 Another PAC. Injection Date: 4/9/2016 2:444 22 M Inj Volume: 3,000 µl Acq. Method : E:\DATA\WEF\XX-A-40\GYQA-100407-F-3-MED-EE 2018-04-08 08-53-49\ 6)-95-5-HL-3UL-60HLM M (Sequence Method) Last changed : 5/4/2016 4:2:3:3 PH by SYSTEM Analysis Method : E:\DATA\WEF\XX-A-40\GYQA-100407-F-3-MED-EE 2018-04-08 08-53-49\ (a)-55-5-HL-3UL-60HLM M (Sequence Method) Last changed : 5/4/2018 4:2:4:33 PH by SYSTEM (mail ice after loading) Additional Info : Peak(s) manually integrated DADIC Sup2004Rem300.000(EVANAWE XA400VQA 10007F-3-MEDEE 2018-0408 08-53 4007 + 20010) mAU 0 0 0 0 0 0 0 0 0 0 0 0 0	
<pre>Injoint For : 0.0000 First In In Volume : 3.000 µl Acq. Method : E:\DATA\WEFYX-A-40\GYDA-180407-F-3-MEO-EE 2018-04-08 08-53-49\</pre>	
Acq. Method : F:\DATA\WFYXX-A-40\(V7QA-180407-F-3-HEO-EE 2018-04-08 08-53-49\) Last changed : 4/8/2018 4:02:29 PH by SYSTEM Analysis Method : E:\DATA\WFYXX-A-40\(V7QA-180407-F-3-HEO-EE 2018-04-08 08-53-49\) (b) 535-51H-30\(-CONTA) M (Sequence Method) Last changed : 5/4/2018 4:54:33 PH by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DATO(C.Sup2104Ref=300.00(E:DATAWFE.X-A40\0000 4007-F-3-MEO-EE 2018-04-08 08-53-49\) (c) -55HH-30(C) Additional Info : Peak(s) manually integrated DATO(C.Sup2104Ref=300.00(E:DATAWFE.X-A40\0000 4007-F-3-MEO-EE 2018-0400.0863 4007+2201.D) (mAU) (mod) (m	
S)-95-5-IIL-3UL-6ULIN.M     Last changed     S)-95-5-IIL-3UL-6UNIN.M     Last changed     S)-4018 4:02:29 PL by SYSTEM     Analysis Method:     S: UDATA/WEP/XX-A-40/GTCA-180407-F-3-MED-EE 2018-04-08 08-53-49/     Last changed     S)-402-10-4018 4:53:31 PL by SYSTEM     Ladditional Info:     Peak (s) manually integrated     DADIC.Sig=2104Ref=300.000 (EDATAWEL-X-4400/VDA 480407-F-3-MEDEE 2018-04-08 08-63 4907+201.D)     ImAU     DADIC.Sig=2104Ref=360.100     Feak RetTime Type Width Area     F [min] [min] [mAU <sup>3</sup> ] [mAU] %     ImAU 1 %     Im3 [min] [mAU <sup>3</sup> ] [mAU] %     ImaU 1 %     Im3 [min] [mAU <sup>3</sup> ] [mAU] %     Im3 [mAU <sup>3</sup> ] Iso.38323     ImaU 1 %     Im3 [min] [mAU <sup>3</sup> ] Iso.38323     ImaU	49\DAD-0J()
Last changed : 4/8/2016 4/02:29 PH Dy SYSTEM Analysis Method : E:\DATA\WEFYX-A 40\GTOA-160407-F-3.HE0-EE 2018-04-08 08-53-49\ (6)-95-5-IHL-30L-60HLM. (Sequence Method) Last changed : S/4/2018 4:54:33 PH by SYSTEM (modified after loading) Additional Info : Pesk(s) manually integrated	10,010 00,0
Analysis Method : E::DATA/MEFXX-A-40/070A-100407-F-3-MED-EE 2018-04-08 08-53-49\ 6)-95-5-IML-3UL-60MIN.M (Sequence Method) Last changed : 5/4/2018 4:54:33 PH by SYSTEM (modified after loading) Additional Info : Pre4(s) manually integrated DADIC.Sige2104R#-300,000(EDATAWE.XA400/024 80407-F-3-MED-EE 2018-04000863.4007+2010) meU 200 0 0 0 0 0 0 0 0 0 0 0 0	
6)-95-5-1ML-30L-60/HR.M (Sequence Method) Last changed : 5/4/2018 4:54:33 PM by SYSTEM (modified after loading) Additional Info : Peak(3) manually integrated DAULC Sp22H0.4R#=300.000(EUA/AWE.X-AWAUVDA HOUD/F-3-MEDCE 2018-04:08:08:63-49071-201.0) mAU 	49\DAD-0J()
Last changed : 5/4/2018 41:54:33 PM by SYSTEM (additional Info : Peak (3) manually integrated DADIC.Sym210.4 Ref=300.000(EUA1200E_XA400/02A 80007.F-3.MEC.EE 2018.04.08.0863.40071.2201.D) mau 200 100 00 00 00 00 00 00 00 00	,-
(modified after loading) Additional Info : Peak (s) manually integrated DADIC Sign210.4R #= 380.100 (EVALAWE .X A480/02A 10007.F.3 MED EE 2018.04.08 08653 4007.1.201.D) mau au au au au au au au au au	
Additional Info : Peak (s) manually integrated DADIC.SuperIO.4Ref 300.100 (ENATAWE XA40.070A 100407.F.3.MED EE 20 18-04-08 08-63 4007 + 201.D) m44 200 100 100 0 0 0 0 0 0 0 0 0 0 0 0	
DADIC. Sig=210.4 Ref=380.400 (E:0ATAWE_XA40xY04 180407-F-3-MED.EE 2018-04.08 08:53.4007+2201.D)	
mwu       0	))
$\begin{aligned} \begin{array}{c} & \end{array}{} \\ & \begin{array}{c} & \end{array}{} \\ & \end{array}{} \\ & \end{array}{} \\ \hline \\ \\ \\ & \end{array}{} \\ \\ \hline \\ \\ \\ & \end{array}{} \\ \\ \\ \hline \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \end{array}{} \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\$	
200	
200 100 100 100 100 100 100 100	
200 100 100 100 100 100 100 100	
100       100         0       0         <	
100 100 100 100 100 100 100 100	
100 100 100 100 100 100 100 100	
<pre>     100</pre>	
100       0	12
100       60         60       6         6       10         45       20         25    Area Percent Report    Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU] \$  1 29.555 EB 0.6260 5788.57617 129.87875 98.2866 2 32.587 EB 0.4821 100.9105 2.50448 1.7134 Totals : 5889.48712 132.38323 **** End of Report ***	8 0
100       1	N A
100       0       0       10       15       20       25         Area Percent Report         Area Percent Report         Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] * 	Ű.
<pre>     100     10     10     1     10     1</pre>	Ц
add       a	
Area Percent Report Area Percent Report Area Percent Report Multiplier : 1.0000 Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDS Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAUT*s] [mAU] % 	J
60       0       10       15       20       25         Area Percent Report         Area Percent Report         Sorted Ey : Signal Multiplier : 1.0000 Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] %         1       29.555 BB       0.6260 5788.57617 129.87875 98.2866         2       32.587 BB       0.4821 100.91095 2.50448 1.7134         Totals : 5889.48712 132.38323	
s0       0       0       15       20       25         Area Percent Report         Area Percent Report         Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [NAU*s] [NAU] * 	
Area Percent Report Area Percent Report Sorted By : Signal Multiplier : 1.0000 Do not use Multiplier & Dilution Factor with ISTDS Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU] * 	- 1 1
Area Percent Report         Area Percent Report         Sorted By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width Area Height Area         # [min]       [min] [miN]*	
Area Percent Report         Area Percent Report         Sorted By       :       Signal         Multiplier       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width       Area         #       [min]         [min]       [mAU*s]         [mAU]       %	- 1 \ .
Area Percent Report         Area Percent Report         Sorted By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width       Area         # [min]       [mAU]         1       29.555 BB       0.6260 5788.57617         12       32.587 BB       0.4821         0.4821       100.91095       2.50448         1.7134       Totals :       5889.48712         **** End of Report ****	
0       6       10       15       20       25         Area Percent Report         Area Percent Report         Multiplier       :       1.0000         Dilution       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width       Area         #       [min]       [mAU]         *       [mAU]         1       29.555 BB       0.6260 5788.57617         2       32.587 BB       0.4821         Totals :       5889.48712       132.38323         **** 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:     DAD1 C, Sig=210,4 Ref=360,100       Peak RetTime Type Width     Area       #     [min]       [min]     [mAU*s]       1     29.555 BB       0.6260     5788.57617       129.555 BB     0.6260       2     32.587 BB       0.4821     100.91095       2.50448     1.7134	
Area Percent Report         Sorted By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width       Area         # [min]       [mAU]	30 '
Area Percent Report         Sorted By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width       Area         # [min]       [mAU*s]       [mAU]	
Area Percent Report         Sorted By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width       Area         # [min]       [mAU*s]       [mAU]	
Area Percent Report         Sorted By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width       Area         # [min]       [mAU]	
Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] % 	
Sorted By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width       Area         # [min]       [mAU*s]       [mAU]	
Softed By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width       Area         # [min]       [mAU*s]         [mAU]       %	
Multiplier : 1.0000 Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] % 	
Dilution       :       1.0000         Do not use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1 C, Sig=210,4 Ref=360,100         Peak RetTime Type Width       Area         # [min]       [mAU*s]         [mAU]       %	
Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] % 	
Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] %       1 29.555 BB 0.6260 5788.57617 129.87875 98.2866 2 32.587 BB 0.4821 100.91095 2.50448 1.7134 Totals : 5889.48712 132.38323 	
Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] % 	
Signal 1: DADI C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] % 	
Peak RetTime Type Width       Area       Height       Area         # [min]       [mAU*s]       [mAU]       %                         1       29.555 BB       0.6260       5788.57617       129.87875       98.2866         2       32.587 BB       0.4821       100.91095       2.50448       1.7134         Totals :       5889.48712       132.38323         **** End of Report ***	
<pre>reak Ketline Type W10th Area height Area # [min] [mAU*s] [mAU] %     1 29.555 BB 0.6260 5788.57617 129.87875 98.2866 2 32.587 BB 0.4821 100.91095 2.50448 1.7134 Totals : 5889.48712 132.38323 *** End of Report ***</pre>	
<pre># [minj [minj [mAU*sj [mAU] % </pre>	
1 29.555 BB 0.6260 5788.57617 129.87875 98.2866 2 32.587 BB 0.4821 100.91095 2.50448 1.7134 Totals : 5889.48712 132.38323 **** End of Report ***	
2 32.587 BB 0.4821 100.91095 2.50448 1.7134 Totals : 5889.48712 132.38323	
Totals : 5889.48712 132.38323	
*** End of Report ***	
*** End of Report ***	
*** End of Report ***	
""" End of Report """	
# HPLC-2j-rac

Data File E:\DATA\YJX\YJX-1-105\YJX-1-105-2 2018-04-02 10-19-04\021-1201.D Sample Name: YJX-1-105-3

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-DAD Seq. Line : 12 Location : Vial 21 Injection Date : 4/2/2018 4:04:49 PM Inj: 1 Inj Volume : 3.000 µl : E:\DATA\YJX\YJX-1-105\YJX-1-105-2 2018-04-02 10-19-04\DAD-0J(1-6)-95-5-Acq. Method 1ML-3UL-60MIN.M Last changed : 4/2/2018 11:35:29 AM by SYSTEM Analysis Method : E:\DATA\YJX\YJX-1-105\YJX-1-105-2 2018-04-02 10-19-04\DAD-0J(1-6)-95-5-1ML-3UL-60MIN.M (Sequence Method) : 5/4/2018 4:58:48 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DAD1C, Sig=210,4 Re=360,100 (E:\DATAYJXYJX-1-105YJX-1-105-2 2018-04-02 10-19-04021-1201.D) mAU  $\cap$ 400 28.243 32.587 300 200 100 0 28 30 32 34 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 28.243 BB 0.5875 1.34749e4 327.84149 50.0700 2 32.567 BB 0.6489 1.34372e4 294.19751 49.9300 2.69120e4 622.03900 Totals : \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-DAD 5/4/2018 4:59:00 PM SYSTEM

# HPLC-2j-cat

Data File E:\DATA\WEF\XX-A-40\GYQA-180407-F-3-ME0-EE 2018-04-08 08-53-49\072-2601.D Sample Name: YJX-1-107-5

Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-DAD Seq. Line : 26 Location : Vial 72 Injection Date : 4/9/2018 3:45:35 AM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\WEF\XX-A-40\GYQA-180407-F-3-ME0-EE 2018-04-08 08-53-49\DAD-0J(1-6)-95-5-1ML-3UL-60MIN.M Last changed : 4/8/2018 4:02:29 PM by SYSTEM Analysis Method : E:\DATA\WEF\XX-A-40\GYQA-180407-F-3-ME0-EE 2018-04-08 08-53-49\DAD-0J(1-6)-95-5-1ML-3UL-60MIN.M (Sequence Method) : 5/4/2018 5:04:58 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DAD1C, Sig=210,4 Ref=360,100 (E:\DATAWE...X, A40(SYQA 180407-F-3-MEO-EE 2018-04-08 08-53-49007-2-2801.D) mAU 0 F 1000 32.696 800 600 400 200 0 15 10 25 зΰ 35 źò min Area Percent Report \_\_\_\_\_ Sorted By : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 32.696 BB 0.7750 4.17057e4 722.93329 100.0000 Totals : 4.17057e4 722.93329 \*\*\* End of Report \*\*\*

1260HPLC-DAD 5/4/2018 5:05:03 PM SYSTEM

# HPLC-2k-rac

Data File E:\DATA\GYQ\GYQ-A\GYQA-180502-F-2-NAI 2018-05-02 14-28-28\011-1101.D Sample Name: YJX-1-124-4

Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-DAD Seq. Line : 11 Location : Vial 11 Injection Date : 5/2/2018 8:10:06 PM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\GYQ\GYQ-A\GYQA-180502-F-2-NAI 2018-05-02 14-28-28\DAD-0J(1-6)-95 -5-1ML-3UL-60MIN.M Last changed : 5/2/2018 3:52:26 PM by SYSTEM Analysis Method : E:\DATA\GYQ\GYQ-A\GYQA-180502-F-2-NAI 2018-05-02 14-28-28\DAD-0J(1-6)-95 -5-1ML-3UL-60MIN.M (Sequence Method) : 5/4/2018 4:40:45 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DAD1C, Sig=210,4 Re=360,100 (E:\DATA\GYD\GYD\GYD\A-180502-F-2-NAI2018-05-02 14-28-28\011-1101.D) mAU Ο 175 150 125 100 · 8 ŝ 39.234 75 · 50 25 0 38 40 з4 зò 32 36 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier : 1.0000 Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 30.564 BB 0.5664 3027.59424 78.42869 49.9931 2 39.234 BB 0.6603 3028.42505 63.51556 50.0069 6056.01929 141.94425 Totals : \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-DAD 5/4/2018 4:40:52 PM SYSTEM

## HPLC-2k-cat

Data File E:\DATA\WEF\XX-A-40\GYQA-180407-F-3-ME0-EE 2018-04-08 08-53-49\071-2501.D Sample Name: YJX-1-107-4

Seq. Line : 25 Acq. Operator : SYSTEM Location : Vial 71 Acq. Instrument : 1260HPLC-DAD Injection Date : 4/9/2018 2:44:42 AM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\WEF\XX-A-40\GYQA-180407-F-3-ME0-EE 2018-04-08 08-53-49\DAD-0J(1-6)-95-5-1ML-3UL-60MIN.M Last changed : 4/8/2018 4:02:29 PM by SYSTEM Analysis Method : E:\DATA\WEF\XX-A-40\GYQA-180407-F-3-ME0-EE 2018-04-08 08-53-49\DAD-0J(1-6)-95-5-1ML-3UL-60MIN.M (Sequence Method) : 5/4/2018 4:54:33 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DAD1C, Sig=210,4 Ref=360,100 (E:\DATAWE...X, A40(SYQA 180407-F-3-MEO-EE 2018-04-08 08-53-49007-1-2501.D) mAU О 200 29.665 150 100 50 32.587 o 15 25 20 зò 10 min Area Percent Report \_\_\_\_\_ Sorted By : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=360,100 Peak RetTime Type Width Height Area Area [mAU\*s] # [min] [min] [mAU\*s] [mAU] % 1 29.555 BB 0.6260 5788.57617 129.87875 98.2866 2 32.587 BB 0.4821 100.91095 2.50448 1.7134 0.4821 100.91095 2.50448 5889.48712 132.38323 Totals : \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-DAD 5/4/2018 4:55:34 PM SYSTEM

### HPLC-21-rac

Data File D:\DATA\YANG JIAXIN\YJX-1-124-7\YJX-1-124-7 2018-05-25 09-11-47\041-0201.D Sample Name: YJX-7-R

\_\_\_\_\_ Acq. Operator : Seq. Line : 2 Location : Vial 41 Acq. Instrument : Instrument 1 Injection Date : 5/25/2018 9:28:34 AM Inj: 1 Inj Volume : 5.000 µl : D:\DATA\YANG JIAXIN\YJX-1-124-7\YJX-1-124-7 2018-05-25 09-11-47\VWD-AS(1-6) Acq. Method -90-10-1.0ML-5UL-210NM-60MIN.M Last changed : 5/25/2018 9:09:09 AM Analysis Method : D:\METHOD\YANG JIAXIN\VWD-AD(1-2)-97-3-1ML-1UL-210NM-15MIN.M Last changed : 5/25/2018 3:35:07 PM (modified after loading) Additional Info : Peak(s) manually integrated
WD1 A Wavelength=210 nm (D:\DATAYANG JIAXIN\YJX-1-1247\YJX-1-1247 2018-05-25 09-11-47041-0201.D) mALI  $\cap$ .OMe - 20 - 40 220 - 60 - 80 -100 -120 45 50 55 35 40 з'n min Area Percent Report Sorted By : Signal : 1.0000 Multiplier Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] \* ----|-----|----|-----|------| 1 43.759 BB 1.1632 4027.68628 53.14627 50.0237 2 48.275 BB 1.2755 4023.86914 47.48991 49.9763 8051.55542 100.63618 Totals :

Instrument 1 5/25/2018 3:35:15 PM

# HPLC-21-cat

Data File D:\DATA\YANG JIAXIN\YJX-1-124-7\YJX-1-124-7 2018-05-25 09-11-47\042-0301.D Sample Name: YJX-7



Instrument 1 5/25/2018 3:36:43 PM

## HPLC-2m-rac

Data File D:\DATA\LWD\WSW-6-1\WSW-6-1 2018-06-01 21-09-03\011-0601.D Sample Name: YJX-1-5-R

\_\_\_\_\_ Acq. Operator : Seq. Line : 6 Location : Vial 11 Acq. Instrument : Instrument 2 Injection Date : 6/2/2018 2:00:50 AM Inj: 1 Inj Volume : 3.000 µl Acq. Method : D:\DATA\LWD\WSW-6-1\WSW-6-1 2018-06-01 21-09-03\DAD-0J(1-6)-95-5-1ML-3UL-ALL-60MIN.M Last changed : 5/24/2018 8:19:20 PM Analysis Method : D:\METHOD\WEN SONGWEI\DAD-0J(1-6)-100-0-0.3ML-10UL-ALL-60MIN.M Last changed : 6/2/2018 9:12:31 AM (modified after loading) Additional Info : Peak(s) manually integrated DADIC, Sig=210.4 Ref-off (D:DATALWD:WSW-8-11WSW-8-1 2018-06-01 21-09-03011-0601.D) mALL 🗌 Ω 175 -150 -125 -40 D17 100 49 D41 75 -50 25 ۵ .25 35 37.5 40 42.5 45 50 52.5 55 min 47.5 Area Percent Report -----Sorted By Signal : 1.0000 : Multiplier 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 C, Sig=210,4 Ref=off Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] \* ----|-----|-----|------|------|------| 1 40.017 BB 0.6699 6393.43799 114.77940 50.3187 2 49.041 BB 0.7988 6312.45117 95.54179 49.6813 Totals : 1.27059e4 210.32119

Instrument 2 6/2/2018 9:12:39 AM

## HPLC-2m-cat

Instrument 2 6/2/2018 7:12:40 PM

Data File D:\DATA\YANG JIAXIN\YJX-1-5\YJX-1-5 2018-06-02 17-36-03\012-0301.D Sample Name: YJX-1-5



79

### HPLC-2n-rac

Data File D:\DATA\LG\COOH-HCL\20180529-COOH 2018-05-29 08-11-05\011-1701.D Sample Name: YJX-1-17-R



Instrument 2 5/30/2018 9:42:25 PM

## HPLC-2n-cat

Data File D:\DATA\H2Y\SOLVENT\SOLVENT 2018-05-30 10-42-03\012-1101.D Sample Name: YJX-1-17



Instrument 2 5/30/2018 9:39:23 PM

## HPLC-2o-rac

Data File E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\071-1301.D Sample Name: YJX-1-124-8-R2

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 13 Location : Vial 71 Injection Date : 5/5/2018 12:04:15 AM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M Last changed : 5/4/2018 4:21:45 PM by SYSTEM Analysis Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M (Sequence Method) : 5/5/2018 9:41:31 AM by SYSTEM Last changed (modified after loading) Additional Info : Peak (s) manually integrated

 VWD1A, Wavelength=210 nm (E:\DATAYYJXYJX 1-124-0504YYJX 1-1242018-05-04-16-21-444071-1301.D)
 mAU Boc N 12.676 800 13.889 600 400 200 0 12 14 10 16 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 12.676 BB 0.2957 1.39866e4 726.12280 49.9505 2 13.899 BB 0.3207 1.40143e4 670.91998 50.0495 Totals : 2.80009e4 1397.04279 \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-VWD 5/5/2018 9:41:36 AM SYSTEM

## HPLC-2o-cat

Data File E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\072-1401.D Sample Name: YJX-1-124-8

Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 14 Location : Vial 72 Injection Date : 5/5/2018 1:05:04 AM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M Last changed : 5/4/2018 4:21:45 PM by SYSTEM Analysis Method : E:\DATA\YJX\YJX-1-124-0504\YJX-1-124 2018-05-04 16-21-44\VWD-AD(1-2)-97-3-1ML-3UL-210NM-60MIN.M (Sequence Method) : 5/5/2018 9:44:12 AM by SYSTEM Last changed (modified after loading) 0 mAU Boc N 1400 12,484 1200 1000 800 600 · 400 200 13.729 0 4 16 10 14 12 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 12.484 VV 0.2974 2.22944e4 1153.93469 97.4783 2 13.729 VB 0.3319 576.73810 25.98991 2.5217 2.28711e4 1179.92460 Totals : \*\*\* End of Report \*\*\*

1260HPLC-VWD 5/5/2018 9:44:17 AM SYSTEM

## HPLC-2p-rac

Data File D:\DATA\LYH\LYH-3-558\LYH-3-558 2018-10-18 20-39-10\023-0801.D Sample Name: YJX-1-207

\_\_\_\_\_ Acq. Operator : Seq. Line : 8 Location : Vial 23 Acq. Instrument : Instrument 1 Injection Date : 10/19/2018 2:20:44 AM Inj: l Inj Volume : 3.000 µl : D:\DATA\LYH\LYH-3-558\LYH-3-558 2018-10-18 20-39-10\VWD-AD(1-2)-95-5-1ML-Acq. Method 3UL-210NM-40MIN.M Last changed : 6/20/2018 10:03:06 PM Analysis Method : D:\METHOD\CZY\DAD-OD(1-2)-98-2-1ML-3UL-ALL-70MIN.M Last changed : 2/19/2019 8:56:19 PM (modified after loading) Additional Info : Peak(s) manually integrated WWD1 A Wavelength=210nm (D:\DATALYH\LYH-3-558\LYH-3-558 2018-10-18 20-39-10/023-0801.D) mALL 🗌 Ο Boc 1750 1500 1250 927 1.106 1000 750 500 250 0 12 10 mir Area Percent Report -----Sorted By Signal : 1.0000 . : Multiplier 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] \* ----|-----|----|-----|-----|-----| 1 9.271 BB 0.2138 1.54250e4 1091.92212 50.3644 2 11.106 BV 0.2509 1.52018e4 919.54590 49.6356 Totals : 3.06269e4 2011.46802

Instrument 2 2/19/2019 8:58:21 PM

# HPLC-2p-cat

Data File D:\DATA\YANG JIAXIN\YJX-2-34-2\YJX-2-34-2 2018-12-04 09-36-40\081-0201.D Sample Name: YJX-2-34-2



Instrument 2 2/19/2019 8:38:33 PM

# HPLC-2q-rac

Data File D:\DATA\GU...NG\GYQB-INDOLE\GYQB-180723-TRI-RAC-1 2018-07-23 14-42-13\041-4801.D Sample Name: YJX-1-167-1-Rac



Instrument 2 8/19/2018 4:38:56 PM

# HPLC-2q-cat

Data File D:\DATA\GU...NG\GYQB-INDOLE\GYQB-180723-TRI-RAC-1 2018-07-23 14-42-13\042-4901.D Sample Name: YJX-1-167-1



Instrument 2 8/19/2018 4:36:35 PM

## HPLC-2r-rac

Data File E:\DATA\LYH\LYH-3-464\LYH-3-464 2018-05-08 17-35-18\054-3301.D Sample Name: YJX-1-124-19-R2

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 33 Location : Vial 54 Injection Date : 5/9/2018 10:38:51 AM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\LYH\LYH-3-464\LYH-3-464 2018-05-08 17-35-18\VWD-AD(1-2)-95-0.5ML -3UL-210NM-60MIN.M Last changed : 5/8/2018 10:25:53 PM by SYSTEM Analysis Method : E:\DATA\LYH\LYH-3-464\LYH-3-464 2018-05-08 17-35-18\VWD-AD(1-2)-95-0.5ML -3UL-210NM-60MIN.M (Sequence Method) : 5/9/2018 2:58:06 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated VWD1A, Wavelength=210 nm (E:\DATAL\YH\LYH-3-484LYH-3\_4842018-05-08 17-35-18'054-3301.D) 0 mAU 1750 1500 1250 292 25.359 22 1000 -750 -500 · 250 0 20 10 25 15 min Area Percent Report \_\_\_\_\_ Sorted By : Signal Multiplier 1.0000 : : Dilution 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 22.767 BB 0.4843 3.39700e4 1093.96716 49.9255 2 25.359 BV 0.5060 3.40713e4 1054.36829 50.0745 Totals : 6.80413e4 2148.33545 \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-VWD 5/9/2018 2:59:56 PM SYSTEM

## HPLC-2r-cat

Data File E:\DATA\LYH\LYH-3-464\LYH-3-464 2018-05-08 17-35-18\055-3401.D Sample Name: YJX-1-124-19

\_\_\_\_\_ Acq. Operator : SYSTEM Acq. Instrument : 1260HPLC-VWD Seq. Line : 34 Location : Vial 55 Injection Date : 5/9/2018 11:39:48 AM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\LYH\LYH-3-464\LYH-3-464 2018-05-08 17-35-18\VWD-AD(1-2)-95-0.5ML -3UL-210NM-60MIN.M Last changed : 5/8/2018 10:25:53 PM by SYSTEM Analysis Method : E:\DATA\LYH\LYH-3-464\LYH-3-464 2018-05-08 17-35-18\VWD-AD(1-2)-95-0.5ML -3UL-210NM-60MIN.M (Sequence Method) : 5/9/2018 3:01:14 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated VWD1A, Wavelength=210 nm (E:\DATAL\YH\LYH-3-484LYH-3-4842018-05-08 17-35-18'055-3401.D) mAU 1200 1000 208 8 800 600 400 26.362 200 0 20 15 10 min Area Percent Report \_\_\_\_\_ Sorted Bv : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=210 nm Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] % 1 22.798 BB 0.4757 2.66837e4 872.87067 89.2262 2 25.352 BB 0.4920 3221.97681 101.58834 10.7738 2.99057e4 974.45901 Totals : \*\*\* End of Report \*\*\* Page 1 of 1 1260HPLC-VWD 5/9/2018 3:01:32 PM SYSTEM