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Electronic Supplementary Information

## Ligand-Controlled Stereodivergent 1,3-Dipolar Cycloaddition of

### 3-Methyl-4-Nitro-5-Styrylisoxazoles with Azomethine Ylides

Kang Liu, Yong Xiong, Zuo-Fei Wang, Hai-Yan Tao, and Chun-Jiang Wang\*

*E-mail:* cjwang@whu.edu.cn

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### I. General Remarks

<sup>1</sup>H NMR spectra were recorded on a VARIAN Mercury 300 MHz or Bruker 400 MHz spectrometer in CDCl<sub>3</sub>. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = single, d = double, t = triple, q = quarte, m = multiple or unresolved, brs = broad single, coupling constant(s) in Hz, integration). <sup>13</sup>C NMR spectra were recorded on a VARIAN Mercury 75 MHz or Bruker 100 MHz spectrometer in CDCl<sub>3</sub> or DMSO-d<sub>6</sub>. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel-coated plates. Enantiomeric ratios were determined by HPLC, using a chiralpak AS-H column, chiralpak AD-H column, chiralpak IA, IB and IC column with hexane and *i*-PrOH as solvents. The starting materials 3-Methyl-4-Nitro-5-Styrylisoxazoles 1a-1f were prepared according to the literature procedure.<sup>1</sup> All Chiral ligands were prepared according the previous procedure. The absolute configuration of 5 and 6 was determined unequivocally according to the X-ray diffraction analysis, and those of other adducts were deduced on the basis of these results.

### II. Proposed Catalyzed Mechanaism for Ligand-Controlled Diastereodivergent and Stereoselective 1,3-DC of Azomethine Ylides with 4-Nitro-5-Styrylisoxa-Zoles. (Scheme 1)

On the basis of the absolute configurational assignments, the reaction mechanism explaining the observed diastereo-divergent and stereochemistry of the products was proposed (Scheme 1). The in situ formed azomethine ylide is coordinated to the AgOAc/<sup>t</sup>Bu-Phosferrox (L3) complex, in a distorted tetrahedral geometry affording the catalytically active species **A** through the abstraction of a proton by the base, which has been supported by a DFT calculation study performed by Hou and co-workers (left cycle)<sup>2</sup>. The intermediate **B** provides the desired *endo*-cycloaddition product **3** and regenerates the active catalyst and base. The unfavorable steric repulsion between the substituents of dipolarophiles and the bulky *tert*-butyl group of <sup>52</sup>

the Phosferrox ligand inhibits the exo approach, contributing to the good endo selectivity. On the other hand, Nájara and co-workers describes the origins of an unexpected stepwise exo-selective 1,3-DC catalyzed by silver salt and phosphoramidite ligand, which is also proved by DFT calculations<sup>3</sup>. Those calculations show that the silver center is coordinated to both the nitrogen atom and the carboxy group of the azomethine ylide, to the phosphorous atom of chiral ligand L10, furnishing the intermediate A' in a trigonal pyramidal geometry (right cycle). The upper side of the metalloazomethine ylide is shielded by the bulky secondary amine group in the phosphoramidite ligand, and hinders the approach of the dipolarphile 1a from the below side. The intermidiate B' tend to deliver the cycloadduct exo-adduct 4 and regenerate the catalysts. The plausible catalytic process is consistant with the observed stereochemical outcome, and further investigations to explore the mechanism are underway.



Scheme 1 Proposed Mechanism for the AgOAc catalyzed ligand-controlled diastereodivergent and enantioselective annulations of 3-methyl-4-nitro-5-styrylisoxazoles with azomethine ylides.

#### III. General Procedure for Racemic 1,3-DC of Azomethine Ylides with 3-Methyl-

#### 4-Nitro-5-Styrylisoxazoles.

Under argon atmosphere, PPh<sub>3</sub> (8.6 mg, 0.033 mmol) and AgOAc (5.0 mg, 0.030 mmol) were dissolved in 2.0 mL  $CH_2Cl_2$ , and stirred at room temperature for about 30 min. Then, the imino ester 2 (0.39 mmol), Et<sub>3</sub>N (0.045 mmol) and 3-methyl-4-nitro-5styrylisoxazole 1 (0.3 mmol) were added sequentially. Once starting material was consumed (monitored by TLC), the organic solvent was removed and the residue was purified by column chromatography to give the product 3 (10 mol% racemic monophose ligand for the product 4), which was used as the racemic sample for the chiral HPLC analysis.

## IV. General Procedure for Asymmetric 1,3-DC of Azomethine Ylides with 4-Nitro-5-Styrylisoxazoles Catalyzed by AgOAc/ $(S_p,S)$ -Phosferrox ligand or AgOAc/ $(R_a, S, S)$ - phosphoramidite ligand.

Under argon atmosphere,  $(S_p,S)$ -<sup>t</sup>Bu-Phosferrox L<sub>3</sub> (16.3 mg, 0.033 mmol) and AgOAc (5.0 mg, 0.03 mmol) were dissolved in 2.0 mL CH<sub>2</sub>Cl<sub>2</sub>, and stirred at room temperature for about 30 min. Then, the imino ester 2 (0.39 mmol), Et<sub>3</sub>N (0.045 mmol) were added sequentially, the reaction temperature was dropped to 0 °C and then 3-methyl-4-nitro-5-styrylisoxazole 1 (0.3 mmol) was added. Once starting material was consumed (monitored by TLC), the organic solvent was removed and the residue was purified by column chromatography to give the product 3 (10 mol% chiral  $(R_a, S, S)$ -Phosphoramidite L<sub>10</sub> for the product 4), which was then directly analyzed by HPLC to determine the enantiomeric excess.



## Methyl (2S,3R,4S,5R)-5-(4-chlorophenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3phenylpyrrolidine-2-carboxylate:

Yield (90 %); yellow solid. m.p. 136-138 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz) δ

7.34 (d, J = 3.9 Hz, 4H), 7.31-7.27 (m, 1H), 7.27 (d, J = 4.1 Hz, 1H), 7.19 (s, 3H), 5.14 (d, J = 8.9 Hz, 1H), 4.82 (t, J = 9.1 Hz, 1H), 4.18 (d, J = 8.2 Hz, 2H), 3.72 (s, 3H), 2.89 (s, 1H), 2.30 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.28, 172.00, 155.19, 137.94, 137.76, 137.75, 130.52, 128.94, 128.48, 128.15, 127.76, 127.65, 67.12, 64.38, 52.36, 52.09, 50.76, 11.31; HRMS Calcd. For C<sub>22</sub>H<sub>20</sub>ClN<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 442.1164, found: 442.1161. The product was analyzed by HPLC to determine the enantiomeric excess: 87 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 11.94 and 16.44 min.



Methyl (2*S*, 3*R*, 4*S*, 5*R*)-5-(2-chlorophenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3phenyl pyrrolidine-2-carboxylate:

Yield (89 %); yellow solid. m.p. 125-128 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.34-7.20 (m, 6H), 7.14 (s, 3H), 5.12 (d, J = 8.1 Hz, 1H), 4.84 (t, J = 9.2 Hz, 1H), 4.26-4.11 (m, 2H), 3.74 (s, 3H), 2.91 (s, 1H), 2.31 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.34, 171.29, 155.04, 137.84, 136.89, 132.34, 130.90, 129.38, 129.07, 128.96, 128.78, 127.75, 127.54, 126.99, 67.12, 61.26, 52.33, 51.62, 49.58. 11.56; HRMS Calcd. For C<sub>22</sub>H<sub>20</sub>ClN<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 442.1164, found: 442.1163. The product was analyzed by HPLC to determine the enantiomeric excess: 87 % ee (Chiralpak AD-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 11.82 and 15.10 min.



### Methyl (2*S*, 3*R*, 4*S*, 5*R*)-5-(4-bromophenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3phenyl pyrrolidine-2-carboxylate:

Yield (89 %); yellow solid. m.p. 150-153 °C.; <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.34-7.20 (m, 7H), 7.14 (d, J = 8.1 Hz, 2H), 5.12 (d, J = 8.7 Hz, 1H), 4.82 (t, J = 9 Hz, 1H), 4.19-4,13 (m, 2H), 3.72 (s, 3H), 2.89 (s, 1H), 2.30 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.35, 172.00, 158.93, 155.07, 138.14, 133.60, 129.38, 128.61, 128.38, 128.09, 114.21, 113.91, 67.03, 64.05, 55.07, 52.26, 52.12, 49.99, 11.23; HRMS Calcd. For C<sub>22</sub>H<sub>20</sub>BrN<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 486.0659, found: 486.0660. The product was analyzed by HPLC to determine the enantiomeric excess: 87 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 12.52 and 18.21 min.



## Methyl (2*S*, 3*R*, 4*S*, 5*R*)-4-(3-methyl-4-nitroisoxazol-5-yl)-3-phenyl-5-(o-tolyl) pyrrolidine-2-carboxylate:

Yield (75 %); yellow solid. m.p. 110-112 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz) δ 7.45 (d, J = 7.2 Hz, 1H), 7.40-7.20 (m, 5H), 7.17 (t, J = 7.5 Hz, 1H), 6.89 (t, J = 7.5 Hz, 1H), 6.64 (d, J = 8.1 Hz, 1H), 5.33 (t, J = 9.3 Hz, 1H), 4.97 (t, J = 8.7 Hz, 1H), 4.17-4.07 (m, 2H), 3.72 (s, 3H), 3.66 (s, 3H), 3.24 (s, 1H), 2.27 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz) δ 172.75, 172.39, 155.82, 154.80, 138.40, 129.06, 128.82, 128.18, 127.55, 126.15, 120.51, 109.40, 67.76, 61.43, 54.79, 52.38, 52.22, 51.24, 11.28; HRMS Calcd. For C<sub>23</sub>H<sub>23</sub>N<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 422.1710, found: 422.1705. The product was analyzed by HPLC to determine the enantiomeric excess: 82 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 10.82 and 24.13 min.



Methyl (2*S*, 3*R*, 4*S*, 5*R*)-5-(4-methoxyphenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3-phenylpyrrolidine-2-carboxylate:

Yield (57 %); yellow solid. m.p. 100-102 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.38-7.22 (m, 5H), 7.15 (d, J = 8.4 Hz, 2H), 6.72 (d, J = 8.7 Hz, 2H), 5.12 (d, J = 9.0 Hz, 1H), 4.80 (s, 1H), 4.17 (s, 2H), 3.74 (s, 3H), 3.72 (s, 3H), 2.88 (s, 1H), 2.28 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.45, 172.45, 159.15, 155.17, 155.17, 138.16, 138.16, 131.09, 131.09, 128.91, 128.91, 127.88, 127.88, 127.67, 127.67, 113.68, 113.68, 67.34, 65.03, 55.17, 52.67, 52.36, 51.34, 11.38; HRMS Calcd. For C<sub>23</sub>H<sub>23</sub>N<sub>3</sub>O<sub>6</sub> + H<sup>+</sup>: 438.1660, found: 438.1652. The product was analyzed by HPLC to determine the enantiomeric excess: 90 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 15.19 and 29.73 min.



Methyl (2*S*, 3*R*, 4*S*, 5*R*)-4-(3-methyl-4-nitroisoxazol-5-yl)-3,5-diphenylpyrrolidine -2-carboxylate:

Yield (90 %); yellow solid. m.p. 96-98 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.49-7.10 (m, 10H), 5.16 (d, *J* = 9.0 Hz, 1H), 4.84 (t, *J* = 8.4 Hz, 1H), 4.23-4.10 (m, 2H), 3.72 (s, 3H), 2.96 (s, 1H), 2.25 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.38, 155.09, 139.05, 138.08, 128.90, 128.34, 128.05, 127.66, 126.64, 67.39, 65.44, 52.54, 52.32, 51.20, 11.24; HRMS Calcd. For C<sub>22</sub>H<sub>21</sub>N<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 408.1481, found: 408.1479. The product was analyzed by HPLC to determine the enantiomeric excess: 85% ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 10.85 and 18.18 min.



Methyl (2*S*, 3*R*, 4*S*, 5*R*)-5-(furan-2-yl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3-phenyl pyrrolidine-2-carboxylate:

Yield (62 %); yellow solid. m.p. 121-123 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz) δ 7.40-7.20 (m, 5H), 7.20 (s, 1H), 6.30 (d, J = 2.9 Hz, 1H), 6.22 (s, 1H), 5.16 (d, J = 8.2Hz, 1H), 4.80 (t, J = 9.3 Hz, 1H), 4.27-4.07 (m, 2H), 3.70 (s, 3H), 3.04 (s, 1H), 2.39 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz) δ 172.80, 171.21, 155.26, 152.62, 142.55, 137.65, 130.60, 128.85, 128.53, 127.83, 127.67, 110.37, 110.24, 108.30, 66.99, 58.79, 52.35, 51.70, 50.36, 11.56; HRMS Calcd. For C<sub>20</sub>H<sub>19</sub>N<sub>3</sub>O<sub>6</sub> + H<sup>+</sup>: 398.1274, found: 398.1278. The product was analyzed by HPLC to determine the enantiomeric excess: 80 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$ nm); t<sub>r</sub> = 11.76 and 23.40 min.



Methyl (2*S*, 3*R*, 4*S*, 5*R*)-4-(3-methyl-4-nitroisoxazol-5-yl)-5-(naphthalen-1-yl)-3phenylpyrrolidine-2-carboxylate:

Yield (80 %); yellow solid. m.p. 132-134 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$ 8.00-7.89 (m, 2H), 7.71 (m, 2H), 7.50-7.20 (m, 8H), 5.97 (d, J = 8.5 Hz, 1H), 5.22 (t, J = 9.2 Hz, 1H), 4.32 (d, J = 9.7 Hz, 1H), 4.19 (t, J = 9.8 Hz, 1H), 3.76 (s, 3H), 3.07 (s, 1H), 1.98 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.48, 171.49, 154.50, 137.78, 135.08, 133.20, 130.88, 130.08, 128.94, 128.74, 128.34, 127.72, 127.63, 125.68, 125.49, 125.36, 134.66, 121.64, 67.06, 60.31, 52.34, 51.57, 50.91. 11.00; HRMS Calcd. For  $C_{26}H_{23}N_3O_5 + H^+$ : 458.1638, found: 458.1641. The product was analyzed by HPLC to determine the enantiomeric excess: 85% ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 8.73 and 15.33 min.



Methyl (2*S*, 3*R*, 4*S*, 5*R*)-4-(3-methyl-4-nitroisoxazol-5-yl)-5-(naphthalen-2-yl)-3phenylpyrrolidine-2-carboxylate:

Yield (92 %); yellow solid. m.p. 134-136 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz) δ 7.75-7.65 (m, 4H), 7.50-7.20 (m, 8H), 5.33 (d, J = 7.8 Hz, 1H), 4.93 (t, J = 9.2 Hz, 1H), 4.25 (d, J = 5.8 Hz, 2H), 3.76 (s, 3H), 3.06 (s, 1H), 2.13 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz) δ 173.31, 171.55, 155.77, 135.74, 135.48, 133.39, 133.05, 131.47, 129.02, 128.61, 127.91, 127.80, 127.69, 126.62, 126.28, 124.41, 68.58, 65.35, 55.01, 51.78, 48.83, 11.58; HRMS Calcd. For C<sub>26</sub>H<sub>23</sub>N<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 458.1638, found: 458.1635. The product was analyzed by HPLC to determine the enantiomeric excess: 91 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$ nm); t<sub>r</sub> = 13.11 and 36.63 min.



Methyl (2*S*, 3*R*, 4*S*, 5*R*)-3,5-bis(4-chlorophenyl)-4-(3-methyl-4-nitroisoxazol-5-yl) pyrrolidine-2-carboxylate:

Yield (84 %); yellow solid. m.p. 145-147 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta_{S9}$ 

7.29 (m, 4H), 7.18 (m, 4H), 5.13 (d, J = 8.9 Hz, 1H), 4.76 (t, J = 9.0 Hz, 1H), 4.16 (m, 2H), 3.73 (s, 3H), 2.88 (s, 1H), 2.31 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.03, 171.55, 155.22, 137.87, 136.15, 133.81, 133.58, 130.57, 129.11, 129.03, 128.50, 128.10, 66.86, 64.09, 52.41, 52.04, 49.91, 11.26; HRMS Calcd. For C<sub>22</sub>H<sub>19</sub>Cl<sub>2</sub>N<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 476.0702, found: 476.0708. The product was analyzed by HPLC to determine the enantiomeric excess: 85% ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 11.69 and 16.97 min.





Yield (81 %); yellow solid. m.p. 134-136 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.25-7.00 (m, 8H), 5.13 (d, J = 8.6 Hz, 1H), 4.80 (t, J = 9.1 Hz, 1H), 4.21-4.05 (m, 2H), 3.73 (s,3H), 2.88 (s, 1H), 2.33 (s, 3H), 2.30 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.28, 172.07, 155.10, 138.51, 137.94, 137.72, 133.65, 130.47, 128.73, 128.48, 128.40, 128.28, 128.11, 124.66, 67.10, 64.38, 52.27, 52.04, 50.71, 21.33, 11.24; HRMS Calcd. For C<sub>23</sub>H<sub>22</sub>ClN<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 456.1248, found: 456.1250. The product was analyzed by HPLC to determine the enantiomeric excess: 86 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 9.62 and 15.26 min.



Methyl (2S, 3R, 4S, 5R)-5-(4-chlorophenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3-

### (o-tolyl)pyrrolidine-2-carboxylate:

Yield (78 %); yellow solid. m.p. 125-127 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.40 (d, J = 7.6 Hz, 1H), 7.25-7.13 (m, 7H), 5.17 (d, J = 9.0 Hz, 1H), 4.81 (t, J = 9.4 Hz, 1H), 4.49 (t, J = 9.7 Hz, 1H), 4.18 (d, J = 9.0 Hz, 1H), 3.72 (s, 3H), 2.91 (s, 1H), 2.38 (s, 3H), 2.30 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.47, 172.22, 155.23, 137.90, 137.10, 136.23, 133.73, 130.79, 130.42, 128.48, 128.13, 127.33, 126.65, 125.89, 67.59, 64.52, 52.53, 52.37, 45.82, 19.51, 11.29; HRMS Calcd. For C<sub>23</sub>H<sub>22</sub>ClN<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 456.1248, found: 456.1249. The product was analyzed by HPLC to determine the enantiomeric excess: 84 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 9.47 and 13.89 min.



### Methyl (2*S*, 3*R*, 4*S*, 5*R*)-5-(4-chlorophenyl)-3-(4-methoxyphenyl)-4-(3-methyl-4nitroisoxazol-5-yl)pyrrolidine-2-carboxylate:

Yield (65 %); yellow solid. m.p. 136-138 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.26-7.15 (m, 6H), 6.85 (d, J = 8.6 Hz, 2H), 5.12 (d, J = 8.9 Hz, 1H), 4.77 (t, J = 9.5 Hz, 1H), 4.15-4.10 (m, 2H), 3.77 (s, 3H), 3.73 (s, 3H), 2.87 (s, 1H), 2.30 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.35, 172.00, 158.93, 155.07, 138.14, 133.60, 129.38, 128.61, 128.38, 128.09, 114.21, 113.91, 77.43, 77.00, 76.58, 67.03, 64.05, 55.07, 52.26, 52.12, 49.99, 11.23; HRMS Calcd. For C<sub>23</sub>H<sub>22</sub>ClN<sub>3</sub>O<sub>6</sub> + H<sup>+</sup>: 472.1197, found: 472.1180. The product was analyzed by HPLC to determine the enantiomeric excess: 90 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 16.50 and 25.22 min.



Methyl (2*S*, 3*S*, 4*S*, 5*R*)-5-(4-chlorophenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3-((E)-styryl)pyrrolidine-2-carboxylate:

Yield (80 %); yellow solid. m.p. 124-126 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.33-7.20 (m, 5H), 7.17 (m, 4H), 6.56 (d, J = 15.7 Hz, 1H), 6.14 (dd, J = 15.7, 8.5 Hz, 1H), 5.01 (d, J = 5.4 Hz, 1H), 4.59 (t, J = 9.5 Hz, 1H), 3.96 (d, J = 6.0 Hz, 1H), 3.86-3.73 (m, 4H), 2.80 (s, 1H), 2.35 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.65, 172.31, 155.51, 138.25, 136.35, 134.21, 134.07, 129.35, 128.84, 128.41, 128.25, 126.66, 126.08, 105.35, 77.73, 77.31, 76.88, 65.80, 64.37, 52.78, 50.67, 49.55, 11.65; HRMS Calcd. For C<sub>24</sub>H<sub>22</sub>ClN<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 468.1248, found: 468.1250. The product was analyzed by HPLC to determine the enantiomeric excess: 85 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 12.21 and 17.63 min.



Methyl (2*S*, 3*S*, 4*S*, 5*R*)-5-(4-chlorophenyl)-3-cyclopropyl-4-(3-methyl-4-nitroisoxazol-5-yl)pyrrolidine-2-carboxylate:

Yield (87 %); yellow oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.08-7.15 (m, 4H), 4.90 (d, *J* = 8.4 Hz, 1H), 4.45-4.57 (m, 1H), 3.94 (d, *J* = 8.4 Hz, 1H), 3.86 (s, 3H), 2.64-2.75 (m, 1H), 2.36 (s, 3H), 0.81-0.92 (m, 1H), 0.55-0.62 (m, 1H), 0.33-0.45 (m, 1H), 0.21-0.31 (m, 1H), 0.07-0.14 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$ 172.98, 155.10, 137.37, 133.45, 129.43, 128.28, 127.94, 65.75, 64.44, 52.33, 51.54, 49.71, 13.79, 11.32, 4.01, 3.16; HRMS Calcd. For C<sub>19</sub>H<sub>20</sub>ClN<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 406.1091, found: 406.1093. The product was analyzed by HPLC to determine the enantiomeric s12 excess: 64 % ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 11.95 and 16.84 min.



Methyl (2*R*, 3*R*, 4*S*, 5*S*)-5-(4-chlorophenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3phenylpyrrolidine-2-carboxylate:

Yield (95 %); yellow solid. m.p. 134-136 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz) δ 7.48 (d, J = 8.1 Hz, 2H), 7.32 (d, J = 8.1 Hz, 2H), 7.27-7.20 (m, 5H), 4.89-4.96 (m, 1H), 4.66 (d, J = 9.9 Hz, 1H), 4.35-4.44 (m, 2H), 3.25 (s, 3H), 2.41 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz) δ 173.25, 171.19, 155.773, 137.03, 135.49, 134.36, 129.04, 128.59, 127.72, 67.52, 65.09, 54.59, 51.72, 48.97, 11.54; HRMS Calcd. For C<sub>22</sub>H<sub>20</sub>ClN<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 442.1164, found: 442.1158. The product was analyzed by HPLC to determine the enantiomeric excess: 93 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 9.05 and 13.58 min.



Methyl (2*R*, 3*R*, 4*S*, 5*S*)-5-(4-bromophenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3phenylpyrrolidine-2-carboxylate:

Yield (63 %); yellow solid. m.p. 156-158 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$ 7.48 (d, J = 8.4 Hz, 2H), 7.41 (d, J = 8.4 Hz, 2H), 7.19-7.30 (m, 5H), 4.88-4.96 (m, 1H), 4.65 (d, J = 9.9 Hz, 1H), 4.31-4.45 (m, 2H), 3.25 (s, 3H), 2.41 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  173.25, 171.18, 155.81, 137.55, 135.49, 132.02, 131.27, 128.81, 128.63, 127.96, 127.73, 122.56, 67.56, 65.12, 54.63, 51.75, 48.91, 11.57; HRMS Calcd. For C<sub>22</sub>H<sub>20</sub>BrN<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 486.0659, found: 486.0652. The product was analyzed by HPLC to determine the enantiomeric excess: 92 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 9.95 and 15.25 min.



# Methyl (2*R*, 3*R*, *S*, 5*S*)-4-(3-methyl-4-nitroisoxazol-5-yl)-3-phenyl-5-(*p*-tolyl) pyrrolidine-2-carboxylate:

Yield (89 %); yellow solid. m.p. 113-115 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz) δ 7.40 (d, J = 8.1 Hz, 2H), 7.20-7.32 (m, 5H), 7.15 (d, J = 8.1 Hz, 2H), 4.89-4.98 (m, 1H), 4.63 (d, J = 9.9 Hz, 1H), 4.30-4.45 (m, 2H), 3.23 (s, 3H), 2.39 (s, 3H), 2.33 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz) δ 173.25, 171.70, 155.70, 138.43, 135.86, 134.93, 129.56, 128.56, 127.78, 126.96, 68.29, 65.31, 54.96, 51.75, 49.03, 21.12, 11.60; HRMS Calcd. For C<sub>23</sub>H<sub>23</sub>N<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 422.1710, found: 422.1703. The product was analyzed by HPLC to determine the enantiomeric excess: 92 % ee (Chiralpak AD-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 15.92 and 19.87 min.



# Methyl (2*R*, 3*R*, 4*S*, 5*S*)-4-(3-methyl-4-nitroisoxazol-5-yl)-3-phenyl-5-(*m*-tolyl) pyrrolidine-2-carboxylate:

Yield (96 %); yellow solid. m.p. 84-86 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$ 7.09-7.38 (m, 9H), 4.85-4.98 (m, 1H), 4.65 (d, J = 9.9 Hz, 1H), 4.30-4.48 (m, 2H), 3.24 (s, 3H), 2.39 (s, 3H), 2.35 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  173.17, 171.60, 155.67, 138.54, 137.88, 135.85, 129.38, 128.70, 128.52, 127.76, 124.04, states of the states

68.39, 65.24, 54.87, 51.69, 48.98, 21.33, 11.52; HRMS Calcd. For C<sub>23</sub>H<sub>23</sub>N<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 422.1710, found: 422.1703. The product was analyzed by HPLC to determine the enantiomeric excess: 90 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 7.00 and 10.52 min.



Methyl (2*R*, 3*R*, 4*S*, 5*S*)-5-(4-methoxyphenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3-phenylpyrrolidine-2-carboxylate:

Yield (85 %); yellow solid. m.p. 96-98 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.45 (d, J = 8.1 Hz, 2H), 7.18-7.32 (m, 5H), 6.87 (d, J = 8.1 Hz, 2H), 4.89-4.98 (m, 1H), 4.62 (d, J = 10.2 Hz, 1H), 4.30-4.43 (m, 2H), 3.79 (s, 3H), 3.23 (s, 3H), 2.40 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  173.30, 171.67, 159.65, 155.66, 135.80, 129.95, 128.53, 128.27, 127.73, 114.15, 68.00, 65.20, 55.12, 54.78, 51.70, 48.93, 11.57; HRMS Calcd. For C<sub>23</sub>H<sub>23</sub>N<sub>3</sub>O<sub>6</sub> + H<sup>+</sup>: 438.1660, found: 438.1658. The product was analyzed by HPLC to determine the enantiomeric excess: 90 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 11.11 and 16.25 min.



## Methyl (2*R*, 3*R*, 4*S*, 5*S*)-5-(2-methoxyphenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3-phenylpyrrolidine-2-carboxylate:

Yield (80 %); yellow solid. m.p. 148-150 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$ 7.45 (d, J = 7.2 Hz, 1H), 7.20-7.35 (m, 6H), 6.83-6.67 (m, 2H), 4.97-5.08 (m, 1H), 4.79 (d, J = 9.9 Hz, 1H), 4.49 (d, J = 9.6 Hz, 1H), 4.27-4.37 (m, 1H), 3.84 (s, 3H), 3.20 (s, 3H), 2.41 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.64, 172.18, 157.41, 155.51, 137.19, 129.51, 128.36, 127.90, 127.56, 124.96, 120.82, 110.53, 65.47, 65.20, 55.12, 54.81, 51.54, 48.54, 11.55; HRMS Calcd. For C<sub>23</sub>H<sub>23</sub>N<sub>3</sub>O<sub>6</sub> + H<sup>+</sup>: 438.1660, found: 438.1653. The product was analyzed by HPLC to determine the enantiomeric excess: 84 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 7.85 and 16.30 min.



Methyl (2*R*, 3*R*, 4*S*, 5*S*)-5-(3-methoxyphenyl)-4-(3-methyl-4-nitroisoxazol-5-yl)-3-phenylpyrrolidine-2-carboxylate:

Yield (89 %); yellow solid. m.p. 134-136 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.20-7.35 (m, 6H), 7.00-7.15 (m, 2H), 6.85 (d, J = 8.1 Hz, 1H), 4.89-5.05 (m, 1H), 4.65 (d, J = 9.9 Hz, 1H), 4.28-4.57 (m, 2H), 3.83 (s, 3H), 3.25 (s, 3H), 2.41 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  173.16, 171.45, 159.84, 155.63, 139.84, 135.70, 131.18, 129.77, 128.48, 127.70, 119.17, 114.25, 112.26, 68.21, 65.20, 55.05, 54.71, 51.60, 48.80, 22.52, 11.45; HRMS Calcd. For C<sub>23</sub>H<sub>23</sub>N<sub>3</sub>O<sub>6</sub> + H<sup>+</sup>: 438.1660, found: 438.1652. The product was analyzed by HPLC to determine the enantiomeric excess: 87 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 18.54 and 23.49 min.





Yield (90 %); yellow solid. m.p. 146-148 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.15-7.35 (m, 7H), 7.00-7.10 (m, 1H), 6.88-6.98 (m, 1H), 4.89-5.05 (m, 3H), 4.44 (d, J = 9.6 Hz, 1H), 4.25-4.38 (m, 1H), 3.24 (s, 3H), 2.44 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 75 MHz)  $\delta$  172.47, 171.25, 155.77, 141.77, 135.71, 131.40, 128.51, 127.86, 127.75, 126.84, 125.60, 125.11, 65.24, 63.25, 54.76, 51.69, 49.96, 11.53; HRMS Calcd. For C<sub>20</sub>H<sub>19</sub>N<sub>3</sub>O<sub>5</sub>S + H<sup>+</sup>: 414.1118, found: 414.1112. The product was analyzed by HPLC to determine the enantiomeric excess: 85 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 10.18 and 14.51 min.



### Methyl (2*R* , 3*R* ,4*S*, 5*R*)-5-butyl-4-(3-methyl-4-nitroisoxazol-5-yl)-3-phenylpyrrolidine-2-carboxylate:

Yield (75 %); yellow oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz) δ 7.15-7.30 (m, 5H), 4.26-4.40 (m, 2H), 4.10-4.25 (m, 1H), 3.46-3.60 (m, 1H), 3.18 (s, 3H), 2.49 (s, 3H), 1.55-1.80 (m, 2H), 1.36-1.50 (m, 1H), 1.25-1.35 (m, 2H), 0.80-0.90 (m, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 100 MHz) δ 172.46, 155.74, 136.62, 128.84, 128.38, 127.71, 127.62, 127.29, 126.84, 65.61, 65.21, 55.16, 51.60, 49.17, 33.39, 28.80, 22.54, 13.78, 11.62; HRMS Calcd. For C<sub>20</sub>H<sub>25</sub>N<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 388.1794, found: 388.1799. The product was analyzed by HPLC to determine the enantiomeric excess: 86 % ee (Chiralpak AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 12.00 and 22.94 min.



Methyl (2*R*, 3*R*, 4*S*, 5*S*)-5-(4-chlorophenyl)-2-methyl-4-(3-methyl-4-nitro isoxazol-5-yl)-3-phenylpyrrolidine-2-carboxylate:

Yield (67 %); yellow solid. m.p. 135-137 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz) δ 7.48 (d, J = 8.1 Hz, 2H), 7.31 (d, J = 8.1 Hz, 2H), 7.20-7.27 (m, 6H), 5.10-5.21 (m, 1H), 4.71 (d, J = 10.5 Hz, 1H), 3.93 (d, J = 12.6 Hz, 1H), 3.29 (s, 3H), 2.38 (s, 3H), 1.69 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 100 MHz) δ 175.13, 170.97, 155.73, 136.91, 134.82, 134.29, 131.17, 129.02, 128.62, 128.56, 128.11, 127.63, 70.47, 66.33, 62.96, 52.17, 49.35, 25.32, 11.59; HRMS Calcd. For C<sub>23</sub>H<sub>22</sub>ClN<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 456.1248, found: 456.1252. The product was analyzed by HPLC to determine the enantiomeric excess: 82 % ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min,  $\lambda = 220$ nm); t<sub>r</sub> = 7.14 and 9.07 min.



### (2*S*, 3*S*, 4*R*, 5*R*)-3-(3-methyl-4-nitroisoxazol-5-yl)-2,4-diphenyl-7-oxa-1-azaspiro [4.4]nonan-6-one:

Yield (61 %); yellow oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz) δ 7.58-7.65 (m, 2H), 7.26-7.45 (m, 8H), 5.35-5.51 (m, 1H), 4.79 (d, J = 10.2 Hz, 1H), 4.01-4.20 (m, 2H), 3.41-3.58 (m, 1H), 2.61-2.79 (m, 2H), 2.36 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 100 MHz) δ 178.97, 170.48, 155.76, 141.77, 138.40, 132.96, 129.23, 129.00, 128.80, 127.48, 70.04, 67.41, 64.70, 61.14, 49.66, 37.11, 11.60; HRMS Calcd. For C<sub>23</sub>H<sub>21</sub>N<sub>3</sub>O<sub>5</sub> + H<sup>+</sup>: 420.1481, found: 420.1485. The product was analyzed by HPLC to determine the enantiomeric excess: 76 % ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 16.58 and 26.14 min.



Methyl (2*S*, 3*R*, 4*S*, 5*R*)-1-benzoyl-5-(4-chlorophenyl)-4-(3-methyl-4-nitro isoxazol-5-yl)-3-phenylpyrrolidine-2-carboxylate:

Yield (91 %); yellow solid. m.p. 148-150 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  7.11-7.36 (m, 14H), 5.10-5.18 (m, 1H), 4.79-4.86 (m, 1H), 4.14-4.21 (m, 2H), 3.72 (s, 3H), 2.30 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 100 MHz)  $\delta$  171.57, 168.47, 155.03, 134.43, 130.49, 129.13, 128.55, 128.07, 127.93, 127.65, 126.94, 66.56, 64.34, 52.59, 50.69, 46.87, 11.31; HRMS Calcd. For C<sub>29</sub>H<sub>24</sub>ClN<sub>3</sub>O<sub>6</sub> + H<sup>+</sup>: 546.1426, found: 546.1426. The product was analyzed by HPLC to determine the enantiomeric excess: 84 % ee (Chiralpak IC-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 14.67 and 20.57 min.



Methyl (2*S*, 3*R*, 4*S*, 5*R*)-1-benzoyl-5-(4-chlorophenyl)-4-(3-methyl-4-nitro isoxazol-5-yl)-3-phenylpyrrolidine-2-carboxylate:

Yield (85 %); white solid. m.p. 153-155 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, TMS, 300 MHz)  $\delta$  6.83-7.80 (m, 14H), 5.38-5.52 (m, 2H), 5.09-5.28 (m, 1H), 4.31-4.68 (m, 1H), 3.38 (s, 3H), 2.41 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, TMS, 100 MHz)  $\delta$  174.74, 171.88, 147.75, 135.88, 133.61, 130.03, 128.90, 128.40, 127.75, 65.32, 53.71, 49.59, 36.47, 27.52, 11.67; HRMS Calcd. For C<sub>29</sub>H<sub>24</sub>ClN<sub>3</sub>O<sub>6</sub> + H<sup>+</sup>: 546.1422, found: 546.1419. The product was analyzed by HPLC to determine the enantiomeric excess: 86 % ee

(Chiralpak IC-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 16.41 and 18.99 min.

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## VI <sup>1</sup>H NMR and <sup>13</sup>C NMR Spectra












































S38









S40



S41































S48

**VII HPLC Chromatograms** 



Data File D:\LC\D&T&\XY\XY-3-9\XY-3-9 2015-04-18 09-22-02\021-0201.D Sample Wame: XY-3-1&

Acq. Operator :	XY Seq. Line : 2	
Acq. Instrument :	Instrument 1 Location : Vial 21	
Injection Date :	4/18/2015 9:34:44 AM Inj: 1	
	Inj Volume : 5 µl	
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Last changed :	4/18/2015 9:45:50 AM by XY	
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	10 10 20 20 11	
	Area Percent Report	
Sorted By	: Signal	
Multiplier	: 1.0000	
Dilution	: 1.0000	
lise Multinlier a	Ailution Factor with ISTDs	
C	Neurolanath 220	
signal I: VWUI A,	wavelengtn=220 nm	
reak RetTime Type	winth Area Height Area	
# [min]	[min] mau *s [maŭ ] %	
1 11.904 MM	0.7958 5647.07373 118.26422 50.5482	
2 16.326 BB	1.1266 5524.58008 70.28991 49.4518	
Totals :	1.11717e4 188.55413	

Instrument 1 5/12/2015 8:02:34 PM LJ

```
Data File D:\LC\DATA\XY\XY-3-9\XY-3-9 2015-04-18 09-22-02\022-0601.D
Sample Name: XY-3-9A
   _____
   Acq. Operator : XY
                                           Seq. Line :
                                                          б
                                           Location : Vial 22
   Acq. Instrument : Instrument 1
   Injection Date : 4/18/2015 10:54:48 AM
                                                   Inj :
                                                          1
                                            Inj Volume : 5 µl
               : D:\LC\DATA\XY\XY-3-9\XY-3-9 2015-04-18 09-22-02\ASH-20-80-1ML-220MM-30MIN.
   Acq. Method
                  M
   Last changed : 12/20/2014 10:18:35 AM by XY
Analysis Method : D:\LC\DATA\XY\XY-3-9\XY-3-9 2015-04-18 09-22-02\022-0601.D\DA.M (ASH-20-
                  80-1ML-220NM-30MIN.M)
   Last changed
                : 5/12/2015 8:18:33 PM by LJ
          (modified after loading)

WDD1 A, Wavelength=220 nm (DALC/DATAX/YW/Y3-9W/Y3-9 2015-04-18-09-22-02/022-0601.D)
     mAU
      160 -
       140
       120 -
       100 -
       80 -
       60 -
       40
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                                                            20
                                                                                    min
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                                        ------
                        Area Percent Report
   _____
                                         Signal
   Sorted By
                       :
                          1.0000
   Multiplier
                    :
   Dilution
                            1.0000
   Use Multiplier & Dilution Factor with ISTDs
   Signal 1: VWD1 A, Wavelength=220 nm
                                             Area
   Peak RetTime Type Width
                            Area
                                    Height
   # [min] [min] mÅU *5 [mÅU ] %
     1 11.939 BB 0.6995 7898.25195 164.18903 93.5601
2 16.438 MM 1.1514 543.64996 7.86932 6.4399
                         8441.90192 172.05835
   Totals :
```

Instrument 1 5/12/2015 8:18:39 PM LJ



Data File D:\LC\DATA\XY\XY-3-19\XY-3-19-2 2015-04-28 20-51-48\001-0201.D Sample Wame: XY-3-19A

Acq. Operator :	HR		Seq. Line :	2		
Acq. Instrument :	Instrument 1		Location : V	ial l		
Injection Date :	4/28/2015 9:09:32	PM	Inj :	1		
		II	nj Volume : 5	рі		
Acq. Method :	D:\LC\DATA\XY\XY-3 30MIN.M	-19\XY-3-19-2	2015-04-28 2	0-51-48\ID-20-80	- 1ML - 220 MM -	
Last changed :	4/28/2015 8:49:12	PM by HR				
Analysis Method :	D:\LC\DATA\XY\XY-3 20-80-1ML-220MM-30	-19\XY-3-19-2 MIN.M)	2015-04-28 2	0-51-48\001-0201	D\DA.M (ID-	
Last changed :	5/12/2015 9:03:18 (modified after 1c	PM by LJ ading)				
WWD1 A, Waw	alength=220 nm (DALCADATAW)	NX Y-3-19XXY-3-19-2 20	15-04-28 20-51-48'00	1-0201.D)		
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	Area Percen	t Report				
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Multinlier	· 1 0000					
Dilution	1.0000					
Hee Multiplier c	Dilution Fector wit	h ISTDe				
ose nurcipiter a	DILUCION INCCOL WIL	IL IDIDS				
Simual 1: VMD1 A.	. Wavelength=220 nm					
Peak RetTime Type	. Width Area	Height	Area			
# [min]	[min] mAll *s	[mAll ]	8			
~ [·····]	-	-				
1 11.799 VB	0.4765 1.52442e4	462.20883	50.8480			
2 15.093 BB	0.5876 1.47357e4	364.94934	49.1520			
Totals :	2.99799e4	827.15817				

Instrument 1 5/12/2015 9:03:27 PM LJ

Sample Name: XY-3-20A Acq. Operator : HR Acq. Instrument : Instrument 1 Seq. Line : 3 Location : Vial 2 Injection Date : 4/28/2015 9:40:55 PM Inj: 1 Inj Volume: 5 µl : D:\LC\DATA\XY\XY-3-19\XY-3-19-2 2015-04-28 20-51-48\ID-20-80-1ML-220MM-Acq. Method 30MIN.M Last changed : 4/28/2015 8:49:12 PM by HR Analysis Method : D:\LC\DATA\XY\XY-3-19\XY-3-19-2 2015-04-28 20-51-48\002-0301.D\DA.M (ID-20-80-1ML-220MM-30MIN.M) : 5/12/2015 9:05:32 PM by LJ Last changed (modified after loading) W/D1 A, Wavelength=220 nm (D/LC/DATAX/YV/Y-3-19/2 2015-04-28 20-51-48/002-0301.D) mAU 250 -200 -150 -100 ->11.816 50 ٥ 25 10 15 20 min 5 Area Percent Report Sorted By 1 Signal : Multiplier 1.0000 Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm 
 Peak RetTime Type
 Width
 Area
 Height
 Area

 #
 [min]
 [min]
 mAU
 \*s
 [mAU
 %

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

 1
 11.816
 VB
 0.4630
 823.24670
 25.36865
 6.6722

 2
 15.095
 VB
 0.5844
 1.15152e4
 287.13565
 93.3278
 1.23385e4 312.50430 Totals :

Data File D:\LC\DATA\XY\XY-3-19\XY-3-19-2 2015-04-28 20-51-48\002-0301.D

Instrument 1 5/12/2015 9:05:37 PM LJ

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Data File D:\LC\DATA\XY\XY-3-7\XY-3-7 2015-04-15 20-39-16\011-0201.D Sample Wame: XY-2-64A

3c

Acq. Operator : XY	Seg. Line : 2
Acq. Instrument : Instrument 1	Location : Vial 11
Injection Date : 4/15/2015 8:51:40 PM	Inj: 1
	Inj Volume : 5 µl
Acq. Method : D:\LC\DATA\XY\XY-3-7\XY M	-3-7 2015-04-15 20-39-16\ASH-20-80-1ML-220NM-30MIN.
Last changed : 12/20/2014 10:18:35 AM 3	ьу ХҮ
Analysis Method : D:\LC\DATA\XY\XY-3-7\XY	-3-7 2015-04-15 20-39-16\011-0201.D\DA.M (ASH-20-
80-1ML-220MM-30MIN.M)	
Last changed : 3/12/2013 1:22:29 PH by	70 70
(modified after loading W/D1A. Wavelength=220 nm/DALC/DATAW YW Y-3-70	) XY-3-7 2015-04-15 20-39-16'011-0201.01
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Area Percent Rep	15 20 25 min
Area Percent Rep Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000	15 20 25 min
Area Percent Rep Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with IST	15 20 25 min
Area Percent Rep 	0 zt
Area Percent Rep 	15 20 25 min
Area Percent Rep Area Percent Rep Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with IST Signal 1: VWD1 A, Wavelength=220 nm	
Area Percent Rep Area Percent Rep Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with IST. Signal 1: VWD1 A, Wavelength=220 nm	
Area Percent Rep Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with IST. Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area He	
Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with IST Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area He # [min] [min] mAW *s [mAW	15 20 25 mh
Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with IST Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area He # [min] [min] mAU *s [mAU	15 20 25 mh
0       6       10         0       6       10         Area Percent Rep       6       10         Sorted By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Use Multiplier & Dilution Factor with IST         Signal 1: VWD1 A, Wavelength=220 nm         Peak RetTime Type Width Area He         # [min]       [min] mAU *s [mAU	15 20 25 min 
0       6       10         Area Percent Rep       6       10         Sorted By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Use Multiplier & Dilution Factor with IST         Signal 1:       VWD1 Å, Wavelength=220 nm         Peak RetTime Type Width       Area         #       [min]	ight Area 1 % 
0       6       10         Area Percent Rep       6       10         Area Percent Rep       6       10         Multiplier       1.0000       10000         Dilution       1.0000       10000         Use Multiplier & Dilution Factor with IST       1.0000         Signal 1: VWD1 A, Wavelength=220 nm       1         Peak RetTime Type Width Area He       #         #       [min]       [min]         1       12.540 MM       0.8678       4792.93848       92         2       18.129 BE       1.2890       4782.22510       52	ight Area 15 20 25 min 
0       6       10         Area Percent Rep         Area Percent Rep         Sorted By : Signal         Multiplier       1.0000         Dilution       1.0000         Use Multiplier & Dilution Factor with IST         Signal 1: VWD1 A, Wavelength=220 nm         Peak RetTime Type Width Area He         # [min]       [min] mAU *s [mAU	ight Area 15 20 25 min ort DB 15 20 25 min 0 15 0.055 18631 49.9441 .23708
0       6       10         Area Percent Rep       6       10         Area Percent Rep       6       10         Sorted By       :       Signal         Multiplier       :       1.0000         Dilution       :       1.0000         Use Multiplier & Dilution Factor with IST         Signal 1:       VWD1 A, Wavelength=220 nm         Peak RetTime Type Width       Area       He         #       [min]       [min]       mAU         1       12.540 MM       0.8678 4792.93848       92         2       18.129 BB       1.2890 4782.22510       52         Totals :       9575.16357       144	ight Area 15 20 25 min area ort DB 15 20 25 min 25 min 26 min 27 min 26 min 27 min 27 min 28 min 29 min 20 min

Instrument 1 5/12/2015 7:22:38 PM LJ

Data File D:\LC\DATA\XY\XY-3-7\XY-3-7 2015-04-15 20-39-16\012-0301.D Sample Name: XY-3-7A \_\_\_\_\_ Seq. Line : Acq. Operator : XY 3 Location : Vial 12 Acq. Instrument : Instrument 1 Injection Date : 4/15/2015 9:23:17 PM Inj: 1 Inj Volume: 5 µl : D:\LC\DATA\XY\XY-3-7\XY-3-7 2015-04-15 20-39-16\ASH-20-80-1ML-220MM-30MIN. Acq. Method M Last changed : 12/20/2014 10:18:35 AM by XV Analysis Method : D:\LC\DATA\XY\XY-3-7\XY-3-7 2015-04-15 20-39-16\012-0301.D\DA.M (ASH-20-80-1ML-220NM-30MIN.M) : 5/12/2015 7:27:35 PM by LJ Last changed (modified after loading) W/DI A, Wavelength=220 nm (DALC/DATAX/ W/Y-3-7WY-3-7 2015-04-15-20-39-16/012-0301.D) mAU 120 100 80 -60 -40 44.7.00 20 206 0 25 10 15 20 min ------Area Percent Report \_\_\_\_\_ Sorted By 1 Simal Multiplier : 1.0000 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Area Height Peak RetTime Type Width Area 1 12.524 BB 0.7662 6907.12549 130.16173 93.9638 2 18.206 MM 1.3247 443.70929 5.58250 6.0362 7350.83478 135.74422 Totals : 

Instrument 1 5/12/2015 7:27:42 PM LJ



Data File D:\LC\DATA\XY\XY-3-9\XY-3-9 2015-04-18 09-22-02\025-0901.D Sample Name: XY-2-64D

Acq. Operator : XY	Seq. Line : 9
Acq. Instrument : Instru:	ment 1 Location : Vial 25
Injection Date : 4/18/2	)15 1:29:53 PM Inj: 1
Acq. Method : D:\LC\;	Inj Volume : 5 µl JATA\XY\XY-3-9\XY-3-9 2015-04-18 09-22-02\ASH-20-80-1ML-220MM-40MIN.
M	215 0.00 M DE 1 50
Last changed : 4/15/2	JIS 2:29:47 PM by 20
Marysis Hernod : D:\Lt\. 80-1ML	-220NM-40MIN.M)
Last changed : 5/12/2 (modif	)15 8:23:45 PM by LJ ied after loading)
VW/D1 A, Wavelength=220 (	m (DALCADATAX YX Y-3-9 XY-3-9 2015-04-18 09-22-02/025-0901.D)
mAU ]	0 0
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0 5	10 15 20 25 min
	Area Percent Report
Sorted By	Simual
Multinlier :	1.0000
Dilution :	1.0000
Use Multiplier & Dilutio:	1 Factor with ISTDs
Signal 1: VWD1 A, Wavele:	ıgth=220 лm
Peak RetTime Type Width	Area Height Area
# [min] [min]	mAU *s [mAU ] %
	•
1 10.836 VB 0.911	3 1.18972e4 200.16354 50.9461
2 23.846 VB 2.108	ł 1.14554e4 74.07942 49.0539
m	0.00F0C.4
IOTALS :	Z.335Zbe4 274.24297

Instrument 1 5/12/2015 8:23:59 PM LJ

\_\_\_\_\_ Acq. Operator : XY Acq. Instrument : Instrument 1 Seq. Line : 10 Location : Vial 26 Injection Date : 4/18/2015 2:01:17 PM Inj: 1 Inj Volume : 5 µl : D:\LC\DATA\XY\XY-3-9\XY-3-9 2015-04-18 09-22-02\ASH-20-80-1ML-220MM-40MIN. Acq. Method M Last changed : 4/15/2015 2:29:47 PM by ZQ Analysis Method : D:\LC\DATA\XY\XY-3-9\XY-3-9 2015-04-18 09-22-02\026-1001.D\DA.M (ASH-20-80-1ML-220NM-40MIN.M) Last changed : 5/12/2015 8:27:02 PM by LJ (modified after loading) W/DIA, Wavelength=220 nm (DALC/DATAX YXY3-9XY3-9 2015-04-18 09-22-02/026-1001.D) mAU 8 175 -150 125 -100 -75 -50 -121.121 101.11 121.121 101.11 25 · D 15 10 20 25 Area Percent Report Sorted By Signal : Multiplier 1.0000 : Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area

Data File D:\LC\DATA\XY\XY-3-9\XY-3-9 2015-04-18 09-22-02\026-1001.D

Sample Name: XY-3-5C

#	[min]		[min]	mAU	*s	[mAU	]	뭡
			-					
1	10.823	VB	0.9005	1.16	932e4	199.	15460	91.3462
2	24.127	MM	2.1345	1107	.76782	8.	64990	8.6538
Total	ls :			1.28	009e4	207.	80450	

Instrument 1 5/12/2015 8:27:07 PM LJ

Page 1 of 1

min



Data File D:\LC\DATA\XY\XY-3-14\XY-3-14 2015-04-23 20-08-44\007-1101.D Sample Wame: XY-2-64E

**3**e

Acg. Operator :	: XV	Seg. Line	: 11
Acq. Instrument :	Instrument ]	Location	. 11 : Vial 7
Injection Date :	4/24/2015 2:58:28 M	Ιουαριοή	
injection babe .	- 2- 2 2 2 2 0 1 0 2 , 5 0 , 2 0 All	Ini Volume	· · ·
Acq. Method :	: D:\LC\DATA\XY\XY-3-14\XY-3-14 60MIN.M	2015-04-23	20-08-44\ASH-20-80-1ML-220MM-
Last changed :	: 1/5/2015 8:51:40 PM by XY		
Analysis Method :	D:\LC\DATA\XY\XY-3-14\XY-3-14	2015-04-23 :	20-08-44\007-1101.D\DA.M (ASH-20-
-	80-1ML-220NM-60MIN.M)		· · ·
Last changed :	: 5/12/2015 8:37:41 PM by LJ		
-	(modified after loading)		
W/D1 A, Wave	elength=220 nm (DALC/DATAW YX Y-3-14XY-3-14 20	15-04-23 20-08-44	N007-1101.D)
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0 5	i 10 15 20	25	30 35 40 45 min
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	Area Percent Report		
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Sorted By	: Signal		
Multiplier	: 1.0000		
Dilution	: 1.0000		
Use Multiplier s	Dilution Factor with ISTDs		
Sigmal 1: VWD1 A,	, Wavelength=220 лт		
Peak RetTime Type	e Width Area Height	Area	
# [min]	[min] mAU *s [mAU ]	8	
	-           -		
1 15.214 VB	0.9206 2641.72363 41.00038	50.6926	
2 29.571 MM	2.8188 2569.54102 15.19267	49.3074	
Totals :	5211.26465 56.19304		

Instrument 1 5/12/2015 8:37:46 PM LJ

```
Data File D:\LC\DATA\XY\XY-3-14\XY-3-14 2015-04-23 20-08-44\008-1201.D
Sample Name: XY-3-9B
   Acq. Operator : XY
                                         Seq. Line : 12
   Acq. Instrument : Instrument 1
                                          Location : Vial 8
   Injection Date : 4/24/2015 3:47:46 AM
                                                Inj: 1
                                          Inj Volume : 5 µl
   Acq. Method
               : D:\LC\DATA\XY\XY-3-14\XY-3-14 2015-04-23 20-08-44\ASH-20-80-1ML-220MM-
                 60MIN.M
   Last changed : 1/5/2015 8:51:40 PM by XY
   Analysis Method : D:\LC\DATA\XY\XY-3-14\XY-3-14 2015-04-23 20-08-44\008-1201.D\DA.M (ASH-20-
                  80-1ML-220NM-60MIN.M)
   Last changed
               : 5/12/2015 8:39:27 PM by LJ
         (modified after loading)

W/D1 A, Wavelength=220 nm (DALCXDATAW YW Y3-14XY-3-142015-0423 20-08-444008-1201.D)
     mAU
                               8
       60 -
       50
       40 -
       30 -
       20 -
       10 -
                                                     29.727
       0
                                                                           46 min
                                                             35
                5
                       10
                               15
                                      20
                                              25
                                                     30
                                                                    40
   -----
                                    Area Percent Report
   Sorted By
                          Signal
                   :
   Multiplier
                          1.0000
   Dilution
                          1.0000
   Use Multiplier & Dilution Factor with ISTDs
   Signal 1: VWD1 A, Wavelength=220 nm
   Peak RetTime Type Width
                          Area
                                  Height
                                           Area
   # [min] [min] mAU *s [mAU ] %
     1 15.193 BB 0.9199 4113.26270 62.80519 94.7375
2 29.727 BV 1.5465 228.48344 1.73510 5.2625
                        4341.74614 64.54029
   Totals :
```

Instrument 1 5/12/2015 8:39:31 PM LJ



Data File D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\031-0201.D Sample Wame: XY-2-70A

3f

Acq. Optical
Avd, Institution, institution i boation, viai si
Acq. Method : D:/LC/DATA/XY/XY-3-8/XY-3-8 2015-04-16 20-35-37/ASH-20-80-1ML-220MM-40MIN. M
Last changed : 4/15/2015 2:29:47 PM by ZQ
Analysis Method : D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\031-0201.D\DA.M (ASH-20-
80-1ML-220NM-40MIN.M) Last changed : 5/12/2015 7:39:33 PM by LJ (modified after loading)
W/D1 A, Wavelength=220 nm (D:\LC\DATAX YX Y3-8X Y-3-8 2015-04-16 20-35-37/031-0201.D)
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1 1 I I I I I I I I I I I I I I I I I I
175-
150
125-
1 100-1 3
50-1
Area Percent Report
Softed by : Signal
miltiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs
Signal 1: VWD1 A, Wavelength=220 nm
Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [mAU ] %
1 10.812 MM 0.6884 8138.98389 197.04778 50.2412
2 17.946 BB 1.3557 8060.82861 85.87557 49.7588
Totals : 1.61998e4 282.92335

Instrument 1 5/12/2015 7:39:38 PM LJ

```
Data File D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\032-0301.D
Sample Name: XY-3-8A
   Acq. Operator : XY
                                        Seq. Line :
                                                     3
                                         Location : Vial 32
   Acq. Instrument : Instrument 1
   Injection Date : 4/16/2015 9:39:50 PM
                                               Inj: 1
                                        Inj Volume : 5 µl
   Acq. Method
              : D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\ASH-20-80-1ML-220MM-40MIN.
                М
   Last changed : 4/15/2015 2:29:47 PM by ZQ
   Analysis Method : D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\032-0301.D\DA.M (ASH-20-
                 80-1ML-220NM-40MIN.M)
   Last changed
              : 5/12/2015 7:41:07 PM by LJ
         (modified after loading)

W/D1 A, Wavelength=220 nm (D:LC/DATAX/YX/Y:3-8X/Y:3-8 2015-04-16 20-35-37/032-0301.D)
     mAU
                                 ę
                                  ł
       80
       60
       40
       20
                                                  18.179
       0
                    5
                               10
                                           15
                                                       20
                                                                  25
                                                                             min
   Area Percent Report
   Sorted By
                    :
                          Signal
                  : 1.0000
: 1.0000
   Multiplier
   Dilution
   Use Multiplier & Dilution Factor with ISTDs
   Signal 1: VWD1 A, Wavelength=220 nm
   Peak RetTime Type Width
                                         Area
                          Area
                                 Height
   1 10.846 BB 0.6069 3907.56885 93.22753 92.5607
2 18.179 BB 1.0539 314.06149 3.51071 7.4393
                       4221.63034 96.73825
  Totals :
   Instrument 1 5/12/2015 7:41:12 PM LJ
                                                                        Page 1 of 1
```



Data File D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\033-0401.D Sample Wame: XY-2-70B

3g

Acq. Operator :	XY Seq. Line : 4
Acq. Instrument :	Instrument 1 Location : Vial 33
Injection Date :	4/16/2015 10:11:08 PM Inj: 1
	Inj Volume : 5 µl
Acq. Method :	D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\ASH-20-80-1ML-220MM-60MIN. M
Last changed :	1/5/2015 8:51:40 PM by XY
Analysis Method :	D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\033-0401.D\DA.M (ASH-20- 80-1ML-220NM-60MIN.M)
Last changed :	5/12/2015 7:42:13 PM by LJ (modified after loading)
WVD1 A, Wave	length=220 nm (DALCUDATAX YX Y-3-8X Y-3-8 2015-04-16 20-35-37'033-0401.D)
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	åres Percent Report
Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000
Use Multiplier &	Dilution Factor with ISTDs
Sigmal 1: VWD1 A,	Wavelength=220 nm
Peak RetTime Type	Width Area Height Area
# [min]	[min] mAU *s [mAU ] %
1 11.765 BB	0.6642 1.38206e4 300.77402 50.1449
2 23.191 BB	1.5911 1.37407e4 123.18302 49.8551
Totals :	2.75613e4 423.95704

Instrument 1 5/12/2015 7:42:19 PM LJ

1								
Acq. Uper	ator :	XY			Seq. Line	: 5		
Acq. Inst Injection	Doto :	J/ 16/201	лс I 5 11+12+Л1	DM	LOCALION	; VIAL 34		
mjection	bare .	40 100 201	5 11, 18, 41	rti	Ini Volume	: 5 ul		
Acq. Meth	.od :	D:\LC\DA M	<b>TA\ XY\ X Y-</b> 3·	-8\XY-3-8 20	)15-04-16 2	0-35-37\ASH-20	-80-1ML-220NM-	60MI
Last chan	.ged :	1/ 5/ 2015	8:51:40 PM	1 by ХҮ				
Analysis	Method :	D:\LC\DA 80-1ML-2	TA\XY\XY-3- 20NM-60MIN.	-8\XY-3-8 20 .M)	15-04-16 2	0-35-37\034-05	01.D\DA.M (ASH	-20-
Last chan	.ged :	5/12/201	5 7:45:15 H	M by LJ				
		(modifie	d after los	ading)	5 0 4 4 9 00 0 5 0 74	004.0504.50		
	UUDIA, Mave	iengin=220 nm ∞	(DRECIDATAX N	x 1-3-8 % 1-3-8 ZU I	о-04-10 д0-30-374	U34U5U1.D)		
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Dilution	1		1.0000					
Use Multi	plier s	Dilution	Factor with	ı ISTDs				
Signal 1:	VWD1 A,	Waveleng	th=220 лm					
Peak RetT	ime Type	Width	Area mau to	Height	Area a			
Turi #		[10111]	JILAU "S	[ 0.201]	<del>ہ</del> ا ا			
1 11.	758 BB	0.6706	1.70407e4	366.45248	90.2479			
2 23.	403 MM	1.6649	1841.40771	18.43321	9.7521			
			. –		_			
Totale :			1.88821e4	384.88570				

Instrument 1 5/12/2015 7:45:22 PM LJ



Data File D:\LC\DATA\XY\XY-3-20\XY-3-20 2015-04-29 15-18-53\001-0201.D Sample Wame: XY-3-19B

Acq. Operator : HR	Seq. Line : 2
Acq. Instrument : Instrument 1	Location : Vial 1
Injection Date : 4/29/2015 3:31:16 PM	Inj: 1
Acq. Method : D:\LC\DATA\XY\XY-3-20 40MIU M	1nj Volume : 5 µl 0\XY-3-20 2015-04-29 15-18-53\&SH-20-80-1ML-220WM-
Last changed : 4/26/2015 10:30:07 PM	M by HR
Analysis Method : D:\LC\DATA\XY\XY-3-20	0\XY-3-20 2015-04-29 15-18-53\001-0201.D\DA.M (ASH-20-
80-1ML-220NM-40MIN.M)	)
Last changed : 5/12/2015 9:08:16 PM	by LJ
(modified after load)	ing)
W/D1 A, Wavelength=220 nm (DALCADATAX YX Y:	-3-20XY-3-20 2015-04-29 15-18-53001-0201.D)
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200 100 0 5 10 Area Percent F Sorted By : Signal	15 20 25 30 35 mit
200 100 0 5 10 Area Percent F Sorted By : Signal Multiplier : 1.0000	
200 100 0 5 10 Area Percent F Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000	15 20 25 30 35 mi 
200 100 0 5 10 Area Percent F Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with 1	15 20 25 30 35 mm Report ISTDs
200 100 0 5 10 Area Percent F Area Percent F Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with D	15         20         25         30         35         mir           Report
200 100 0 5 10 Area Percent F Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with 1	15         20         25         30         35         mir           Report
200 100 0 5 10 Area Percent F Area Percent F Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with 1 Signal 1: VWD1 A, Wavelength=220 nm	15         20         25         30         35         mir           Report
200       100         0       0         <	15 20 25 30 35 mir
Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with 1 Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area	Height Area
200 100 0 5 10 Area Percent F Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with D Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area # [min] [min] mAU *s [n	15         20         25         30         35         mit           15         20         25         30         35         mit           Report
Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with D Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area # [min] [min] mAU *s [m 	Interview         Interview <t< td=""></t<>
200       100         100       0         0       6         100       10         Area Percent F         Area Percent F         Sorted By       :         Signal Multiplier       :         1.0000         Dilution       :         Signal 1:       VWD1 Å, Wavelength=220 nm         Peak RetTime Type Width       Årea         #       [min]         1       8.653 VB       0.6478 3.84295e4         2       15.146 VB       1.4802 3.87200-0	Ist         20         25         30         35         mir           Report         IstDs
200 100 0 5 10 Area Percent F Area Percent F Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with D Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area # [min] [min] mAW *s [n 	Ist
200       100       100         0       5       10         Area Percent F         Area Percent F         Sorted By : Signal         Multiplier       1.0000         Dilution       1.0000         Use Multiplier & Dilution Factor with 1         Signal 1: VWD1 Å, Wavelength=220 nm         Peak RetTime Type Width Area         # [min]       [min] mAU *s [n	Ist Description         Ist Description           Height Area         MAU ]           Barrier         Barrier           ISTDs         Barrier           ISTOS         Barrier

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Instrument 1 5/12/2015 9:08:23 PM LJ

Data File D:\LC\DATA\XY\XY-3-20\XY-3-20 2015-04-29 15-18-53\002-0301.D Sample Name: XY-3-20B \_\_\_\_\_ Acq. Operator : HR Acq. Instrument : Instrument 1 Seq. Line : 3 Location : Vial 2 Injection Date : 4/29/2015 4:12:48 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\XY\XY-3-20\XY-3-20 2015-04-29 15-18-53\ASH-20-80-1ML-220MM-Acg. Method 40MIN.M Last changed : 4/26/2015 10:30:07 PM by HR Analysis Method : D:\LC\DATA\XY\XY-3-20\XY-3-20 2015-04-29 15-18-53\002-0301.D\DA.M (ASH-20-80-1ML-220NM-40MIN.M) Last changed : 5/12/2015 9:09:58 PM by LJ (modified after loading) W/DI A. Wavelength=220 nm (D/LC/DATAX YX V3-20X V-3-20 2015-04-29 15-18-53/002-0301.D) mAU 8 2 500 400 -300 200 100 15.327 0 35 15 25 ΞÓ. 10 20 min 5 Area Percent Report Sorted By : Signal 1.0000 Multiplier : Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 Å, Wavelength=220 nm Area Peak RetTime Type Width Area Height 1 8.733 VB 0.6643 2.37975e4 530.77020 92.8499 2 15.327 VB 1.1667 1832.58887 18.62792 7.1501 2.56301e4 549.39812 Totals : \_\_\_\_\_

Instrument 1 5/12/2015 9:10:03 PM LJ



Data File D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\037-0801.D Sample Name: XY-2-70D

Acg. Operator :	XY	Seg. Line : 8
Aco. Instrument :	Instrument 1	Location : Vial 37
Injection Date :	4/17/2015 2:17:25 AM	Ini: 1
		Ini Volume : 5 ul
Acq. Method	D:\LC\DATA\XY\XY-3-8\XY-3-8 M	2015-04-16 20-35-37\ASH-20-80-1ML-220MM-60MIN.
Last changed :	1/5/2015 8:51:40 PM by XY	
Analysis Method :	D:\LC\DATA\XY\XY-3-8\XY-3-8	2015-04-16 20-35-37\037-0801.D\DA.M (ASH-20-
	80-1ML-220NM-60MIN.M)	
Last changed :	5/12/2015 7:53:41 PM by LJ	
	(modified after loading)	
WVD1 A, Wav	elength=220 nm (DALCADATAW YW Y-3-8WY-3-8 2	D15-04-16 20-35-37'037-08D1.D)
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1 13 067 00	0 8856 0 530000 750 7360	7 50 0200
1 13.001 VD	0.0000 4.0044464 107.1400 0.0000 4.0044664 107.1400	0 00 0701
2 30.1(1 MM	3.3330 4.3306464 149.9291	0 42.7171
Totola	0.06500.04 000 6565	7
IOLAIS ;	3.0030364 309.0303	1

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Instrument 1 5/12/2015 7:53:46 PM LJ

Data File D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\038-0901.D Sample Name: XY-3-8D \_\_\_\_\_ Acq. Operator : XY Seq. Line : - 9 Location : Vial 38 Acq. Instrument : Instrument 1 Injection Date : 4/17/2015 3:19:09 AM Inj: 1 Inj Volume: 5 µl : D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\ASH-20-80-1ML-220MM-60MIN. Acq. Method M Last changed : 1/5/2015 8:51:40 PM by XY Analysis Method : D:\LC\DATA\XY\XY-3-8\XY-3-8 2015-04-16 20-35-37\038-0901.D\DA.M (ASH-20-80-1ML-220NM-60MIN.M) : 5/12/2015 7:56:58 PM by LJ Last changed (modified after loading) W/DI A, Wavelength=220 nm (DALC/DATAX/ W/Y3-8W/Y3-8 2015-04-16 20-35-37/038-0901.D) mAU a 250 200 150 100 50 18ª (S<sup>1,9</sup>) 628 ٥ 50 10 20 30 40 min Area Percent Report \_\_\_\_\_ Sorted By 1 Simul Multiplier : 1.0000 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Area Height Peak RetTime Type Width Area 1.68307e4 272.55617 Totals : 

Instrument 1 5/12/2015 7:57:09 PM LJ



Data File D:\LC\DATA\XY\XY-3-14\XY-3-14-1 2015-04-25 09-01-49\011-0201.D Sample Wame: XY-3-14A

\_\_\_\_\_ Acq. Operator : HR Acq. Instrument : Instrument 1 Injection Date : 4/25/2015 9:14:16 AM Seq. Line : 2 Location : Vial 11 Ілј: 1 Ілј Volume: 5 µl : D:\LC\DATA\XY\XY-3-14\XY-3-14-1 2015-04-25 09-01-49\ASH-20-80-1ML-220MM-Acq. Method 30MIN.M Last changed : 12/20/2014 10:18:35 AM by XY Analysis Method : D:\LC\DATA\XY\XY-3-14\XY-3-14-1 2015-04-25 09-01-49\011-0201.D\DA.M (ASH-20-80-1ML-2201MM-30MIN.M) : 5/12/2015 8:42:39 PM by LJ Last changed (modified after loading) W/DIA, Wavelength=220 nm (DSLC/DATAX/ YX Y3-14X/Y-3-14-12015-04-25 09-01-49/011-0201.D) mAU 10,540 80 60 16.891 40 20 0. -20 15 10 20 25 min \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By Signal . 1.0000 Multiplier : 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Height Peak RetTime Type Width Area Area # [min] [min] mAU \*5 [mAU ] % 1 11.701 MM 0.8796 5813.72217 110.15289 50.7020 2 16.891 BB 1.4065 5652.73389 56.05727 49.2980 Totals : 1.14665e4 166.21015 \_\_\_\_\_

Instrument 1 5/12/2015 8:42:48 PM LJ

Data File D:\LC\DATA\XY\XY-3-14\XY-3-14-1 2015-04-25 09-01-49\012-0301.D Sample Name: XY-3-16A Acq. Operator : HR Seq. Line : 3 Location : Vial 12 Acq. Instrument : Instrument 1 Injection Date : 4/25/2015 9:45:47 AM Inj: 1 Inj Volume: 5 µl : D:\LC\DATA\XY\XY-3-14\XY-3-14-1 2015-04-25 09-01-49\ASH-20-80-1ML-220MM-Acq. Method 30MIN.M Last changed : 12/20/2014 10:18:35 AM by XY Analysis Method : D:\LC\DATA\XY\XY-3-14\XY-3-14-1 2015-04-25 09-01-49\012-0301.D\DA.M (ASH-20-80-1ML-220MM-30MIN.M) : 5/12/2015 8:45:25 PM by LJ Last changed (modified after loading) WD1 A, Wavelength=220 nm (DALC/DATAX/YXY3-14412016-04-26:09-01-49/012-0301.D) mAU T a 200 175 150 -125 100 -75 -50 -16.966 25 ٥ 15 25 20 5 10 min Area Percent Report ------Sorted By Signal : : Multiplier 1.0000 Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Height Area Area 1 11.692 VB 0.7856 1.14211e4 214.52846 92.6269 2 16.966 BB 1.1714 909.11847 9.47822 7.3731 Totals : 1.23302e4 224.00668 

Instrument 1 5/12/2015 8:45:29 PM LJ



Data File D:\LC\D&T&\XY\XY-3-14\XY-3-14 2015-04-23 20-08-44\045-1401.D Sample Wame: XY-3-14C

Acq. Operator :	XY Seq. Line : 14
Acq. Instrument :	Instrument 1 Location : Vial 45
Injection Date :	4/24/2015 5:27:16 AM Inj: 1
	Inj Volume : 5 µl
Acq. Method :	D:\LC\DATA\XY\XY-3-14\XY-3-14 2015-04-23 20-08-44\ASH-20-80-1ML-220MM- 60MIM.M
Last changed :	1/5/2015 8:51:40 PM by XY
Analysis Method :	D:\LC\DATA\XY\XY-3-14\XY-3-14 2015-04-23 20-08-44\045-1401.D\DA.M (ASH-20-
	80-1MI-220MM-60MIN-M)
Last changed :	5/12/2015 9:25:31 PM by LJ
	(modified after Loading)
W/D1 A. Wave	(incath = 220 nm (DALCODATAX YX Y-3-14 XY-3-14 2015-04-23 20-08-440/45-1401.D)
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Signal 1: VWD1 A,	Wavelength=220 nm
Peak RetTime Type	Width Area Height Area
# [min]	[min] mAU *s [mAU ] %
1 9.632 MM	0.6438 6875.23975 177.99336 50.4050
2 15.195 MM	1.4944 6764.76660 75.44424 49.5950
a averavo tat	
Totals '	1.36400+4 253.43761
ICDUID .	T.00100C1 000.1010T

Instrument 1 5/12/2015 9:25:36 PM LJ

Data File D:\LC\DATA\XY\XY-3-14\XY-3-14 2015-04-23 20-08-44\046-1501.D Sample Name: XY-3-16C Acq. Operator : XY Seq. Line : 15 Acq. Instrument : Instrument 1 Location : Vial 46 Injection Date : 4/24/2015 6:16:34 AM Inj: 1 Inj Volume : 5 µl : D:\LC\DATA\XY\XY-3-14\XY-3-14 2015-04-23 20-08-44\ASH-20-80-1ML-220MM-Acq. Method 60MIN.M Last changed : 1/5/2015 8:51:40 PM by XY Analysis Method : D:\LC\DATA\XY\XY-3-14\XY-3-14 2015-04-23 20-08-44\046-1501.D\DA.M (ASH-20-80-1ML-220NM-60MIN.M) Last changed : 5/12/2015 9:26:55 PM by LJ (modified after loading) WDD1 A, Wavelength=220 nm (DALCODATAW YW Y3-14XY-3-142015-0423 20-08-44046-1501.D) mAU 🗋 œ 300 250 200 150 100 -50 15.258 Û 35 15 40 46 10 ź 25 30 5 min \_\_\_\_\_ Area Percent Report - -- -- --- -- -- -- -- -- -- -- -- --Sorted By Simual 1 : 1.0000 Multiplier 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Height Area Area 1 9.618 VB 0.5792 1.33708e4 342.49857 92.8853 2 15.258 BB 1.0144 1024.15283 11.95092 7.1147 Totals : 1.43949e4 354.44948 \_\_\_\_\_

Instrument 1 5/12/2015 9:27:00 PM LJ



Data File D:\LC\DATA\XY\XY-3-18\XY-3-18 2015-04-26 19-14-56\021-0201.D Sample Wame: XY-3-17A

Acq. Operator :	HR	Seq. Line : 2	
Aco. Instrument :	Instrument 1	Location : Vial 21	
Injection Date	A/26/2015 7:37:40 DM	Ind 1	
injection bate .	4) 20) 2013 1.31.49 FM		
		ілј voiume : 5 µi	
Acq. Method :	D:\LC\DATA\XY\XY-3-18\XY-3-18	2015-04-26 19-14-56\A	SH-20-80-1ML-220NM-
	40MIN.M		
Last changed '	4/26/2015 7·44·03 PM by HR		
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Analysis Method :	D: \LU\DATA\XY\XY-3-18\XY-3-18	2015-04-26 19-14-56\0	21-0201.D\DA.M (ASH-20-
	80-1ML-220NM-40MIN.M)		
Last changed :	5/12/2015 8:53:26 PM by LJ		
	(modified after loading)		
VIII/D1 A VIAve	elenth=220 pm (DN CDATAX XX X-3-18X X-3-18)	2015-04-26 19-14-56021-0201 D)	
		(D10-D+2010-1+00D21-D201.D)	
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Dilution	: 1,0000		
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Signal 1: VWD1 A.	Wavelength=220 nm		
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	•		
1 9.484 MM	0.6651 1.51974e4 380.82593	50.3148	
2 13 814 BB	1 1198 1 5007264 100 72105	49 6852	
2 13.014 DD	1.11/0 1.000/264 199.12190	47.0002	
m · ·	0 000 45 4 500 5000		

Instrument 1 5/12/2015 8:53:30 PM LJ

Data File D:\LC\DATA\XY\XY-3-18\XY-3-18 2015-04-26 19-14-56\022-0301.D Sample Name: XY-3-18A Acq. Operator : HR Seq. Line : 3 Location : Vial 22 Acq. Instrument : Instrument 1 Injection Date : 4/26/2015 8:19:26 PM Inj: 1 Inj Volume: 5 µl : D:\LC\DATA\XY\XY-3-18\XY-3-18 2015-04-26 19-14-56\ASH-20-80-1ML-220MM-Acg. Method 40MIN.M : 4/26/2015 8:17:59 PM by HR Last changed (modified after loading) Analysis Method : D:\LC\DATA\XY\XY-3-18\XY-3-18 2015-04-26 19-14-56\022-0301.D\DA.M (ASH-20-80-1ML-220NM-40MIN.M) : 5/12/2015 8:55:28 PM by LJ Last changed (modified after loading) W/DIA Wavelength=220 nm (DXLC/DATAX/YX/Y3-18X/Y3-18 2015-0426 19-14-56022-0301.D) 99 1 mAU 500 400 300 200 action of the second 100 ٥ зb 35 20 25 5 10 15 min \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By Signal : Multiplier 1.0000 : 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area [min] mAU \*s [mAU ] # [min] ł 1 9.466 VB 0.6193 2.26895e4 553.69000 92.0986 2 13.889 MM 1.2082 1946.60217 26.85242 7.9014 Totals : 2.46361e4 580.54242

Instrument 1 5/12/2015 8:55:33 PM LJ


Data File D:\LC\DATA\XY\XY-3-14\XY-3-14-1 2015-04-25 09-01-49\013-0401.D Sample Wame: XY-3-14B

Acg. Operator :	HR	Sea.	Line: 4		
Acg. Instrument :	Instrument 1	Loc	ation : Vial	13	
Injection Date :	4/25/2015 10:17:36	M	Ιπή: 1		
-		Ιπή V	olume : 5 ul	L	
Acq. Method :	D:\LC\DATA\XY\XY-3- 30MIN.M	14\XY-3-14-1 201	5-04-25 09-0	)1-49\ASH-20-80-1MI	-220 <b>NM</b> -
Last changed :	12/20/2014 10:18:35	ам бүхү			
Analysis Method :	D:\LC\DATA\XY\XY-3- 20-80-1ML-220MM-30M	·14\XY-3-14-1 201 [IN.M)	5-04-25 09-0	)1-49\013-0401.D\D;	A.M (ASH-
Last changed :	5/12/2015 8:47:40 P (modified after los	'M by LJ (ding)			
VM/D1 A, Wavele	ngth=220 nm (DALCADATAW YV)	(Y-3-14XY-3-14-1 2015-04	25 09-01-49/013-04	101.D)	
mAU .			Ø. 18		
100 -					
60 -				24.911	
40 -				( \	\
20 -		ļ			
0 <u> </u>	······	/	<u>`</u>		
	·····		· · · · ·	· · · · · ·	· · · · · ·
U	<u> </u>	) 15		20 25	mn
	Årea Percent	Report			
Sorted By	: Sional				
Multiplier	; 1.0000				
Dilution	: 1.0000				
Use Multiplier & D	ilution Factor with	l ISTDs			
Signal 1: VWD1 A,	Wavelength=220 nm				
Deals DotTime Turne	Nidth dree	Height lya	•		
feak Kebillie Type # [min]	(min) mall te	ופוקונ אופ (האוד א א	a		
# [min]	[min] mao	[maxo ] 8	1		
1 16.487 MM 2 24.911 BB	1.2787 7726.15576 1.7912 7521.01221	100.70462 50.6 49.87806 49.3	727 273		
Totals :	1.52472e4	150.58268			

Instrument 1 5/12/2015 8:47:48 PM LJ

Data File D:\LC\DATA\XY\XY-3-14\XY-3-14-1 2015-04-25 09-01-49\014-0501.D Sample Name: XY-3-16A -----Acq. Operator : HR Seq. Line : 5 Acq. Instrument : Instrument 1 Location : Vial 14 Injection Date : 4/25/2015 10:49:03 AM Ілј: 1 Ілј Volume: 5 µl : D:\LC\DATA\XY\XY-3-14\XY-3-14-1 2015-04-25 09-01-49\ASH-20-80-1ML-220MM-Acq. Method 30MIN.M Last changed : 12/20/2014 10:18:35 AM by XY Analysis Method : D:\LC\DATA\XY\XY-3-14\XY-3-14-1 2015-04-25 09-01-49\014-0501.D\DA.M (ASH-20-80-1ML-220NM-30MIN.M) Last changed : 5/12/2015 8:51:22 PM by LJ (modified after loading) WWD1 A, Wavelength=220 nm (D3LC/DATAX/YW/Y3-144/Y-3-14-1-2015-04-25-09-01-49/014-0501.D) mAU 60 50 40 30 -20 -25.218 25.218 25. 50 25 10 -D 17.5 ່າວ່ 25 27.5 75 10 15 22.5 12.5 min \_\_\_\_\_ Area Percent Report Sorted By Simual : : 1.0000 Multiplier Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Height Area Area 1 16.497 BB 1.1171 5026.63135 64.72860 95.0991 2 25.218 MM 2.0150 259.04449 2.14267 4.9009 Totals : 5285.67584 66.87127 \_\_\_\_\_

Instrument 1 5/12/2015 8:51:35 PM LJ



Data File D:\LC\DATA\XY\XY-3-18\XY-3-18 2015-04-26 19-14-56\023-0401.D Sample Wame: XY-3-17B

Acq. Opticalof - Inc	
Arde Installand a Mattheward I Mathematical Science (1975)	
Acq. Method : D:\LC\DATA\XY\XY-3-18\XY-3-18 2015-04-26 19-14-56\ASH-20-80-1ML-220MM- 40MTW.M	
Last changed : 4/26/2015 8:59:29 PM by HR (modified after loading)	
Analysis Method : D:LC/DATA/XY/XY-3-18/2015-04-26 19-14-56/023-0401.D/DA.M (ASH	-20-
Last changed : 5/12/2015 8:57:25 PM by LJ	
[modilied after loading]	
Wild A, Wateregn=220 http://www.rkits-tokit-3-tokit-3-tokit-3-to-2015-14-2013-14-50023-0401-0.0	
1 <sup>20</sup> 7	
40 ]	
	min
Area Percent Report	
Sorted By : Signal	
Multiplier : 1.0000	
Dilution : 1.0000	
Use Multiplier & Dilution Factor with ISTDs	
obe Manaparter & Balabion record with 19195	
Circuit I. UTBLI I. Discriber 200	
signal I: vwDI A, wavelengtn=220 nm	
D 1 D 1 D 1 D 1 D 1 D 1 D 1 D 1 D 1 D 1	
Peak RetTime Type Width Area Height Area	
Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU ] %	
Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU ] ዬ 	
Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU ] %        1 12.230 BB 0.8072 2793.07056 50.13667 50.7390	
Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] % 	
Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU ] %      1 12.230 BB 0.8072 2793.07056 50.13667 50.7390 2 17.667 MM 1.7951 2711.71167 25.17738 49.2610	

Instrument 1 5/12/2015 8:57:30 PM LJ

Data File D:\LC\DATA\XY\XY-3-18\XY-3-18 2015-04-26 19-14-56\024-0501.D Sample Name: XY-3-18B Acq. Operator : HR Seq. Line : - 5 Location : Vial 24 Acq. Instrument : Instrument 1 Injection Date : 4/26/2015 9:42:30 PM Inj: 1 Inj Volume: 5 µl : D:\LC\DATA\XY\XY-3-18\XY-3-18 2015-04-26 19-14-56\ASH-20-80-1ML-220MM-Acg. Method 40MIN.M : 4/26/2015 9:41:02 PM by HR Last changed (modified after loading) Analysis Method : D:\LC\DATA\XY\XY-3-18\XY-3-18 2015-04-26 19-14-56\024-0501.D\DA.M (ASH-20-80-1ML-220NM-40MIN.M) Last changed : 5/12/2015 8:58:54 PM by LJ (modified after loading) W/DIA Wavelength=220 nm(DXLC/DATAX/YX/Y3-18X/Y3-18 2015-0426 19-14-56024-0501.D) mAU 613**4**# 200 150 100 50 17.632 ٥ зb 35 10 15 20 25 5 min \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By Signal : Multiplier 1.0000 : 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] mAU \*s [mAU ] ł 1 12.213 BB 0.8532 1.28109e4 226.60112 92.5058 2 17.632 W 1.2523 1037.85962 9.96990 7.4942 Totals : 1.38488e4 236.57102

Instrument 1 5/12/2015 8:58:59 PM LJ



Data File D:\LC\DATA\LK\LK-14-34\LK-14-34 2016-06-03 14-29-13\063-0201.D Sample Wame: LK-14-34

Acq. Operator	: HR Seq. Line : 2
Acq. Instrument	: Instrument l Location : Vial 63
Injection Date	: 6/3/2016 2:41:58 PM Inj: 1
	Inj Volume : 5 µl
Acq. Method	: D:\LC\DATA\LK\LK-14-34\LK-14-34 2016-06-03 14-29-13\ASH-10-90-1ML-220MM.M
Last changed	: 3/17/2015 9:21:18 AM by HR
Analysis Method	: D:\LC\DATA\LK\LK-14-34\LK-14-34 2016-06-03 14-29-13\063-0201.D\DA.M (ASH-
	10-90-1ML-220WM.M)
Last changed	: 6/6/2016 8:56:11 AM by HR
	(modified after loading)
VIVD1 A, Wax	elength=220 nm (D\LC\DATA\LK\LK-14-34\LK-14-34 2016-06-03 14-29-13\063-0201.D)
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	ke
400	
200	
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200	
100	
100-	
l 0	
5	7.5 10 12.5 15 17.5 20 22.5 min
	Area Percent Report
Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000
Use Multiplier &	Dilution Factor with ISTDs
Sigmal 1: VWD1 A	, Wavelength=220 лm
Peak RetTime Typ	e Width Area Height Area
# [min]	[min] mAU *s [mAU ] %
	-
1 12.017 MM	0.7610 2.19080e4 479.78107 51.6117
2 16.722 MM	1.5270 2.05397e4 224.17654 48.3883
Totals :	4.24477e4 703.95761

\*\*\* End of Report \*\*\*

Instrument 1 6/6/2016 8:56:16 AM HR

Sample Name: LK-14-37 \_\_\_\_\_ Acq. Operator : HR Acq. Instrument : Instrument 1 Seq. Line : 1 Location : Vial 64 Injection Date : 6/3/2016 3:12:17 PM Ini : 1 Inj Volume : 5 µl : D:\LC\DATA\LK\LK-14-37-38A\LK-14-37-38A 2016-06-03 15-10-32\ASH-10-90-1ML-Acq. Method 220NM-28MIN.M Last changed : 6/3/2016 3:09:46 PM by HR Analysis Method : D:\LC\DATA\LK\LK-14-37-38A\LK-14-37-38A 2016-06-03 15-10-32\064-0101.D\DA. M (ASH-10-90-1ML-220MM-28MIN.M) Last changed : 6/6/2016 8:58:07 AM by HR (modified after loading) WWD1 A, Wavelength=220 nm (DALC/DATALK/LK/1437-38A/LK-1437-38A/2016-06-03 15-10-32/064-0101.D) mAU œ 500 400 300 200 16.841 100 D 12.5 15 17.5 22.5 25 75 10 20 min Area Percent Report Sorted By Signal : : Multiplier 1.0000 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Area Peak RetTime Type Width Height Area # [min] [min] mAU \*5 [mAU ] % 1 11.948 VB 0.6641 2.71785e4 575.97791 82.2189 2 16.841 BB 1.2770 5877.77979 63.81124 17.7811 Totals : 3.30563e4 639.78915 \_\_\_\_\_ \*\*\* End of Report \*\*\*

Data File D:\LC\DATA\LK\LK-14-37-38A\LK-14-37-38A 2016-06-03 15-10-32\064-0101.D

Instrument 1 6/6/2016 8:58:12 AM HR

Page 1 of 1

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Data File D:\LC\DATA\WZF\WZF-1-2B\WZF-1-2B 2015-06-05 10-13-29\055-0201.D Sample Wame: WZF-1-2B

4a

Aco. Operator :	LJ Seg. Line : 2
Acg. Instrument :	Instrument 1 Location : Vial 55
Injection Date :	6/5/2015 10:26:18 AM Inj: 1
	Ini Volume : 5 ul
Acq. Method :	D:\LC\DATA\WZF\WZF-1-2B\WZF-1-2B 2015-06-05 10-13-29\ASH-20-80-1ML-220AM- 25MIA.M
Last changed :	6/4/2015 10:05:34 AM by LJ
Analysis Method :	D:\LC\DATA\WZF\WZF-1-2B\WZF-1-2B 2015-06-05 10-13-29\055-0201.D\DA.M (ASH- 20-80-1ML-220MM-25MIN.M)
Last changed :	10/30/2015 11:16:00 AM by LK
	(modified after loading)
W/D1 A, Wave	length=220 nm (DALCIDATAW/ZFW/ZF-1-28W/ZF-1-28 2015-06-05 10-13-29/055-0201.D)
mAU -	&, <sup>2</sup>
200 -	
150 -	
100 -	
50 -	
0	
6	8 10 12 14 16 18 20 22 min
	Area Percent Report
Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000
Use Multiplier s	Dilution Factor with ISTDs
Signal 1: VWDl A,	Wavelength=220 nm
Peak RetTime Tune	Midth brea Height brea
# [min]	
" [""TIL	
1 9.422 MM	0.6239 9085.66016 242.72263 50.2988
2 13.644 MM	1.0756 8977.69922 139.10770 49.7012
5 10:011 (MI	2
Totals :	1.80634e4 381.83032

Instrument 1 10/30/2015 11:16:05 AM LK

Sample Name: WZF-1-35B Seq. Line : 2 Location : Vial 78 Acq. Operator : LJ Acq. Instrument : Instrument 1 Injection Date : 7/9/2015 10:06:23 AM Ini : 1 Inj Volume : 5 µl : D:\LC\DATA\WZF\WZF-1-35B\WZF-1-35B 2015-07-09 09-53-46\ASH-20-80-1ML-Acq. Method 220NM-25MIN.M Last changed : 6/8/2015 2:39:29 PM by LJ Analysis Method : D:\LC\DATA\WZF\WZF-1-35B\WZF-1-35B 2015-07-09 09-53-46\078-0201.D\DA.M ( ASH-20-80-1ML-220MM-25MIN.M) Last changed : 10/30/2015 11:20:28 AM by LK (modified after loading) W/D1 A, Wavelength=220 nm (DALCODATAW/ZFW/ZF-1-358 W/ZF-1-358 2015-07-09 09-53-466078-0201.D) mAU ] 400 300 -200 100 053 9 ۵ 12 14 16 18 20 min 10 Ŕ \_\_\_\_\_ Area Percent Report Sorted Bv . Signal Multiplier : 1.0000 Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Area Peak RetTime Type Width Height Area 1 9.053 VB 0.6828 1215.55664 26.75987 3.4051 2 13.583 VB 1.0423 3.44824e4 491.73355 96.5949 3.56979e4 518.49342 Totals :

Data File D:\LC\DATA\WZF\WZF-1-35B\WZF-1-35B 2015-07-09 09-53-46\078-0201.D

Instrument 1 10/30/2015 11:20:37 AM LK

\_\_\_\_\_



Br H 4b Data File D:\LC\DATA\WZF\WZF-1-10B\WZF-1-10B 2015-06-08 14-42-55\021-0201.D Sample Wame: WZF-1-10B

lag Operator :	· I.I · · · · · · · · · · · · · · · · ·	
Acq. Operator .	· Lo Sey	estion : Wisl 21
Injection Date :	· 6/8/2015 2:55:10 DM	
injection bate .	. 0,0,2013 2.33.19 PM	Volume : 5 ul
log Method .	• D•\1 C\D AT A\ 107 E\ 107 E_ 1_10B\107 E_ 1_10B	2015_06_08_14_42_55\ &\$H_20_80_1MT_
Acq. accard ,	220 <b>UM</b> .M	2010 00 00 14 42 00(ADN 20 00 10D
Last changed :	: 6/8/2015 2:39:56 PM by LJ	
Analysis Method :	: D:\LC\D&T&\WZF\WZF-1-10B\WZF-1-10B	2015-06-08 14-42-55\021-0201.D\D&.M (
interfore inconce :	ASH-20-80-1ML-220MM.M)	
Last changed :	: 10/30/2015 11:34:40 AM by LK	
· <b>,</b> · - · ·	(modified after loading)	
WWD1 A, Wave	elength=220 nm (DALCADATAW/ZFW/ZF-1-10BW/ZF-1-10B	2015-06-08 14-42-55/021-0201.D)
mAU	ç.	
	β.	
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	10 12 14	16 18 min
		=============
	Area Percent Report	
		===============
Sorted By	: Signal	
Multiplier	: 1.0000	
Dilution	: 1.0000	
Vse Multiplier s	Dilution Factor with ISTDs	
Sigmai I: VWDI A,	, Wavelength=220 nm	
Peak RetTime Type	e width Area Height Ar	ea
# [min] 	[minj mau *s [mau ]	3
1 0 040 75	-	1
1 9.043 DB	0.3091 1000.34034 194.03119 50.	0070
4 15.335 MM		
	1.2186 1659.31646 99.83111 49.	3310
Totale :	1.2186 1659.31646 99.83111 49.	a 310
Totals :	1.2786 7659.37646 99.83777 49. 1.53399e4 294.68956	3310
Totals :	1.2786 7659.37646 99.83777 49. 1.53399e4 294.68956	3310

Instrument 1 10/30/2015 11:35:30 AM LK

Data File D:\LC\DATA\WZF\WZF-1-31B\WZF-1-31B-1 2015-07-07 12-57-37\078-0201.D Sample Wame: WZF-1-31B



Instrument 1 10/30/2015 11:38:26 AM LK



Data File D:\LC\DATA\W2F\W2F-1-44B-1\W2F-1-44B-1 2015-07-22 09-06-46\033-0201.D Sample Wame: W2F-1-44B-1

**4**c

Acq. Operator :	ZQ Seq. Line : 2
Acq. Instrument :	Instrument 1 Location : Vial 33
Injection Date :	7/22/2015 9:29:12 AM Inj: 1
Acq. Method :	Inj Volume : 5 µl D:\LC\DATA\WZF\WZF-1-44B-1\WZF-1-44B-1 2015-07-22 09-06-46\ADH-20-80-1ML-
T	22UNM.M - 7/22/2015 0.02.21 NW h 70
hast thanged :	. 1/66/6013 9:03:61 AM DY 60 Divitev Division M7 F. 1. 440 - 1:07F. 1. 440 - 1:2016 - 07 - 22:00 - 06 - 46:022 - 020 1:0:Division M
Anarysis Nethod :	D. (DC(DAIA(W21(W21-1-44D-1(W21-1-44D-1 2013-01-22 03-00-40(033-0201.D(DA.M /ADH_20_80_1MI_2200NM_M)
Last changed '	(ADR-20-00-III) - 220MAR)
hast charged .	(modified after loading)
WVD1 A. Wave	elength=220 nm (DXLCVDATAW/ZFW/ZF-1-448-1\W/ZF-1-448-1 2015-07-22 09-06-461033-0201,D)
	N N N N N N N N N N N N N N N N N N N
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75-	
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14	15 16 17 18 19 20 21 22 mb
	area Percent Report
Control Dec	
Sorted By	: Simal
multipiler	: 1.0000
Dilution	: 1.0000
Use Multiplier &	Dilution Factor with ISTDs
Signal 1: VWD1 A,	, Wavelength=220 nm
Peak RetTime Type	: Width Area Height Area
# [min]	[min] mAU *s [mAU ] %
	•
1 15.952 BB	0.6545 8749.16309 192.83839 49.9245
2 19.704 BB	0.8382 8775.64258 150.67863 50.0755
Totals :	1.75248e4 343.51703

Instrument 1 10/30/2015 11:55:43 AM LK

Data File D:\LC\DATA\W2F\W2F-1-47B-1\W2F-1-47B-1 2015-07-27 12-34-20\066-0201.D Sample Wame: W2F-1-47B-1

\_\_\_\_\_ Acq. Operator : WZF Acq. Instrument : Instrument 1 Seg. Line : 2 Location : Vial 66 Injection Date : 7/27/2015 12:46:49 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\WZF\WZF-1-47B-1\WZF-1-47B-1 2015-07-27 12-34-20\ADH-20-80-1ML-Acq. Method 220NM.M Last changed : 7/22/2015 9:03:21 AM by ZQ Analysis Method : D: \LC\DATA\W2F\W2F-1-47B-1\W2F-1-47B-1 2015-07-27 12-34-20\066-0201.D\DA.M (ADH-20-80-1ML-220NM.M) Last changed : 10/30/2015 11:50:23 AM by LK (modified after loading) \W/D1 A, Wavelength=220 nm (D\LC\DATAW/ZFW/ZF-1-47B-1\W/ZF-1-47B-1 2015-07-27 12-3420'066-0201.D) mAU æ 700 600 -500 400 300 -200 -100 -19.870 0 17 21 22 15 16 18 19 20 min 14 \_\_\_\_\_ Area Percent Report Sorted By . Signal Multiplier : 1.0000 Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm 
 Peak RetTime Type
 Width
 Area
 Height
 Area

 # [min]
 [min]
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 1
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 2
 1
 9.870
 BB
 0.7736
 1
 33
 5
 43
 018
 2
 4.19370
 3
 7.574
 Peak RetTime Type Width 3.55412e4 773.89169 Totals : \_\_\_\_\_

Instrument 1 10/30/2015 11:50:27 AM LK

Page 1 of 1

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Data File D:\LC\DATA\W2F\W2F-1-54B-3\W2F-1-56B 2015-09-04 16-57-30\069-0201.D Sample Name: W2F-1-54B-3

4d



Instrument 1 10/30/2015 1:02:01 PM LK



Instrument 1 10/30/2015 1:05:21 PM LK



MeO H 4e Data File D:\LC\DATA\WZF\WZF-1-24B-1\WZF-1-24B-1 2015-06-27 10-35-03\034-0201.D Sample Name: WZF-1-24B-1

lag Operator	
Acq. Uperator	; LU DEQ. LLIE ; 2 ; Instrument ]
Injection Date	· 6/27/2015 10·47·51 M Tuti · 1
Injection babe	Ini Volume : 5 ul
Acq. Method	: D:\LC\DATA\WZF\WZF-1-24B-1\WZF-1-24B-1 2015-06-27 10-35-03\ASH-20-80-1ML- 220MM-25MIN.M
Last changed	: 6/8/2015 2:39:29 PM by LJ
Analysis Method	: D:\LC\DATA\WZF\WZF-1-24B-1\WZF-1-24B-1 2015-06-27 10-35-03\034-0201.D\DA.M
	(ASH-20-80-1ML-220MM-25MIN.M)
Last changed	: 10/30/2015 11:59:08 AM by LK
	(modified after loading)
	elengm=220 nm(UXLC/UATAW/2FW/2F-1-248-1W/2F-1-248-12010-06-27 10-36-03/034-0201.0)
m.AU	а ж_ж
250 -	A
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200 -	] ]
150 -	
100-	34
100	$( ) \qquad \land \qquad  $
50 -	
10	12 14 16 18 20 22 mm
	Area Percent Report
Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000
Use Multiplier &	Dilution Factor with ISTDs
Cimel I. UIRI I	Nevel emoth=220 mm
signal I: VWDI A	, wavelength=220 lum
Doold DotTime Turn	a Nidth broa Vaight broa
feak Kerinne iyp # [min]	c winth Afea height Afea [min] màll te [màll ] 9
	-
1 10.750 MM	0.7694 1.18346e4 256.37186 49.9984
2 16.756 MM	1.9811 1.18354e4 99.56952 50.0016
Totals :	2.36700e4 355.94138

Instrument 1 10/30/2015 11:59:13 AM LK





Instrument 1 10/30/2015 12:06:05 PM LK

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Data File D:\LC\DATA\WZF\WZF-1-50B\WZF-1-50B 2015-07-31 20-20-24\055-0201.D Sample Wame: WZF-1-50B



Instrument 1 10/30/2015 1:07:58 PM LK

Data File D:\LC\DATA\W2F\W2F-1-53B-2\W2F-1-53B-2 2015-08-21 14-01-37\077-0201.D Sample Wame: W2F-1-53B-2

Acq. Operator : ZQ Seq. Line : 2
Acq. Instrument : Instrument 1 Location : Vial 77
Injection Date : 8/21/2015 2:23:19 PM Inj: 1
ותן עסועשר : 5 או Acq. Method : D:\LC\DATA\WZF\WZF-1-53B-2\WZF-1-53B-2 2015-08-21 14-01-37\ASH-20-80-1ML- 220MM-25MIN.M
Last changed : 6/8/2015 2:39:29 PM by LJ
Analysis Method : D:\LC\DATA\WZF\WZF-1-53B-2\WZF-1-53B-2 2015-08-21 14-01-37\077-0201.D\DA.M (ASH-20-80-1ML-220WM-25MIW.M)
Last changed : 10/30/2015 1:10:28 PM by LK (modified after loading)
Wi/D1 A, Vilavelength=220 nm (D3LCDA1AW/2F-W/2F-1-33B-2AW/2F-1-53B-22U16-08-2114-01-37/07-0201.D)
m <sup>2</sup>
800
700-
600 -
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200 -
100 - / /
8 10 12 14 16 18 20 min
Area Percent Report
Control Texas and Control
auteu by : alguar Multiniar - 1 0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs
······································
Signal 1: VWD1 Å, Wavelength=220 nm
Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU ] %
т 7.848 vв   0.4715 8581.46680 265.66718   7.8378 2 16.299 VB   1.8707 1.00907e5   845.90961  92.1622
Totals : 1.09489e5 1111.57678

Instrument 1 10/30/2015 1:10:33 PM LK

Page 1 of 1

S90



Data File D:\LC\DATA\WZF\WZF-1-45B-4\WZF-1-45B-4 2015-07-28 20-39-18\065-0201.D Sample Name: WZF-1-45B-4

4g

log Operator '	W7F Serr Line 2
Acq. Operator :	Instrument   Location : Vial 65
Injection Date :	7/28/2015 8:51:53 PM Ind : 1
ingeobion babe .	י, 20, 2010 0,01,00 ווו וחז Volume : 5 עו
Aca. Method :	D: \L C\ DATA\ WZ F\ WZ F- 1-45B-4\ WZ F- 1-45B-4 2015-07-28 20-39-18\ ASH-20-80-06ML-
	220MM.M
Last changed :	7/28/2015 8:36:18 PM by WZF
Analysis Method :	D: \LC\ DATA\ WZ F\ WZ F-1-45B-4\ WZF-1-45B-4 2015-07-28 20-39-18\ 065-0201. D\ DA.M
-	(ASH-20-80-06ML-220MM.M)
Last changed :	10/30/2015 12:55:07 PM by LK
	(modified after loading)
VII/D1 A, Wave	elength=220 nm (DALC/DATAW/ZFW/ZF-1-46B-4W/ZF-1-46B-4 2015-07-28 20-39-18'065-0201.D)
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14	16 18 20 22 24 26 min
	Area Percent Report
Sorted By	: Simual
Multiplier	: 1.0000
Dilution	: 1.0000
Use Multinlier 6	Dilution Factor with ISTDs
Signal 1: VWD1 A,	Wavelength=220 nm
Peak RetTime Type	Width Area Height Area
# [min]	[min] mAU *s [mAU ] %
	·
1 17.182 BB	0.8679 5.99297e4 1002.37305 49.9319
2 22.871 VB	1.4211 6.00932e4 631.88171 50.0681
Totals :	1.20023e5 1634.25476

Instrument 1 10/30/2015 12:55:12 PM LK





Totals : 1.85693e5 1674.83798

Instrument 1 10/30/2015 12:57:43 PM LK

Page 1 of 2

S92

:



Data File D:\LC\DATA\W2F\W2F-1-56B\W2F-1-56B 2015-09-04 16-16-04\067-0201.D Sample Wame: W2F-1-56B

4h

Acg. Operator :	нв	Seg. Line : 2	
Acg. Instrument :	Instrument 1	Location : Vial 67	
Injection Date :	9/4/2015 4:33:39 PM	Іпј: 1	
		Inj Volume : 5 µl	
Acq. Method :	D: \LC\DATA\WZF\WZF-1-56B	\WZF-1-56B 2015-09-04 16-16-04\ASH	-20-80-1ML-
	220 <b>NM-25MIN.M</b>		
Last changed :	6/8/2015 2:39:29 PM by L	J	
Analysis Method :	D: \LU\DATA\WZF\WZF-1-56B	\WZF-1-56B 2015-09-04 16-16-04\067 T M	-0201.D\DA.M (
lest chenged .	10/30/2015 1:15:38 DM bar	N.(1)	
hast changed .	(modified after loading)	LR .	
VIVD1 A, Wave	length=220 nm (DALCADATAW/ZFW/ZF-1-	56BW/ZF-1-56B 2015-09-04 16-16-04067-0201.D)	
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Sorted By	: Signal		
Multiplier	: 1.0000		
Dilution	: 1.0000		
Use Multiplier s	Dilution Factor with ISTD	5	
Simmal 1, VIIID 1	Mavelength=220 nm		
bighti 1, 1, 1, 1,	waterengon and ian		
Peak RetTime Type	Width Area Hei	oht Area	
# [min]	[min] mAU *s [mAU	~] %	
1 10.946 VB	0.5517 1.04888e4 272.	96948 50.0735	
2 15.979 VB	0.9282 1.04580e4 165.	77760 49.9265	
T	0.00450-4 400	24200	
IOTAIS :	2.09469e4 438.	14109	

Instrument 1 10/30/2015 1:15:42 PM LK

Sample Name: WZF-1-66B Acq. Operator : HR Acq. Instrument : Instrument 1 Seq. Line : 2 Location : Vial 66 Injection Date : 9/9/2015 12:45:23 PM Ini : 1 Inj Volume : 5 µl : D:\LC\DATA\WZF\WZF-1-66B\YL-12-22 2015-09-09 12-32-41\ASH-20-80-1ML-220MM-Acq. Method 25MIN.M Last changed : 6/8/2015 2:39:29 PM by LJ Analysis Method : D:\LC\DATA\WZF\WZF-1-66B\YL-12-22 2015-09-09 12-32-41\066-0201.D\DA.M ( ASH-20-80-1ML-220MM-25MIN.M) : 10/30/2015 1:21:30 PM by LK Last changed (modified after loading) WDD1A, Wavelength=220 nm(D/LC/DATAW/ZFW/ZF-1-66BV/L12-22 2015-09-09 12-32-41/066-0201.D) mAU 400 350 300 -250 200 150 A BOOM 100 178 50 D 18 1n 12 14 18 min Area Percent Report Sorted By . Signal Multiplier : 1.0000 Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Area Peak RetTime Type Width Height Area # [min] [min] mAU \*5 [mAU ] % 1 10.178 MM 0.5589 2063.26489 61.52769 7.4593 2 14.511 BB 0.8760 2.55972e4 436.70819 92.5407 2.76605e4 498.23588 Totals : \_\_\_\_\_

Data File D:\LC\DATA\WZF\WZF-1-66B\YL-12-22 2015-09-09 12-32-41\066-0201.D

Instrument 1 10/30/2015 1:21:34 PM LK



Data File D:\LC\DATA\LK\LK-14-25\LK-14-25 2016-05-28 09-59-26\084-0201.D Sample Wame: LK-14-25

4i

Aca. One	rator : WZF			Seg. Line :	2		
Aca. Ins	trument : Instru	ment l		Location :	Vial 84		
Injectio	π Date : 5/28/2	016 10:12:00	AM	Ini :	1		
				Inj Volume :	5 ul		
Aca. Met	hod : D:\LC\)	DATAVLKVLK-14	-25\LK-14-2	5 2016-05-28		\ASH-2-98-1ML-	220 MM .M
Last cha	nged : 5/26/2	016 8:38:14 A	M by WZF				
Analysis	Method : D:\LC\	DATA\LK\LK-14	-25\LK-14-2	5 2016-05-28	09-59-26	\084-0201.D\DA	.M (ASH-
•	2-98-1	ML-220NM.M)					·
Last cha	nged : 6/6/20	16 8:52:41 AM	by HR				
	(modif	ied after loa	ding)				
	W/D1 A, Wavelength=220 (	Im (DALCADATA/LKAL	K14-25VLK-14-25	2016-05-28 09-59-26	N084-0201.D)		
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		Area Percent	Report				
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Sorted B	у :	Signal					
Multipli	er :	1.0000					
Dilution	. :	1.0000					
Use Mult	iplier & Dilutio:	n Factor with	ISTDs				
	-						
Signal l	: VWD1 A, Wavele:	ngth=220 nm					
Peak Ret	Time Type Width	Area	Height	Area			
# [л	in] [min]	mAU *s	[mAU]	8			
		-		I			
1 12	.041 BV 0.733	9 1.70413e4	337.12213	50.5433			
2 21	.859 MM 1.828	5 1.66750e4	151.99010	49.4567			
Totals :		3.37163e4	489.11223				
== == == ==	= == == == == == == ==			:	== == == ====		

Instrument 1 6/6/2016 8:52:49 AM HR

Data File D:\LC\DATA\LK\LK-14-28-29\LK-14-28-29 2016-05-28 10-53-37\086-0301.D Sample Name: LK-14-29

\_\_\_\_\_ Acq. Operator : WZF Acq. Instrument : Instrument 1 Seq. Line : 3 Location : Vial 86 Injection Date : 5/28/2016 11:37:49 AM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\LK\LK-14-28-29\LK-14-28-29 2016-05-28 10-53-37\ASH-2-98-1ML-Acq. Method 220NM-30MIN.M Last changed : 5/28/2016 10:52:47 AM by WZF Analysis Method : D:\LC\DATA\LK\LK-14-28-29\LK-14-28-29 2016-05-28 10-53-37\086-0301.D\DA.M (ASH-2-98-1ML-220NM-30MIN.M) Last changed : 6/6/2016 8:54:05 AM by HR (modified after loading) \WVD1 A, Wavelength=220 nm (DALCADATALKALK 14-28-29/LK 14-28-29 2016-05-28 10-53-37/086-0301.D) mAU -200 -175 -150 -125 -100 -75 -12.002 50 · 25 0 10 15 2ΰ 25 min Area Percent Report -Sorted By Signal : : 1.0000 : 1.0000 Multiplier Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Area Peak RetTime Type Width Height Area # [min] [min] mAU \*5 [mAU ] % 1 12.002 BB 0.7173 2086.61621 41.21986 7.1615 2 22.944 VB 1.7982 2.70499e4 219.59877 92.8385 Totals : 2.91365e4 260.81863 \_\_\_\_\_ \*\*\* End of Report \*\*\*

Instrument 1 6/6/2016 8:54:09 AM HR

Page 1 of 1

:



CI H 4j Data File D:\LC\DATA\LK\LK-14-20B\LK-14-20B 2016-05-10 14-44-27\083-0201.D Sample Name: LK-14-20B

Acq. Operator : LHC Seq. Line : 2
Acq. Instrument : Instrument 1 Location : Vial 83
Injection Date : 5/10/2016 2:57:05 PM Inj : 1
Inj Volume : 5 pl Acq. Method : D:\LC\DATA\LK\LK-14-20B\LK-14-20B 2016-05-10 14-44-27\ASH-20-80-1ML-220MM. M
Last changed : 10/17/2015 3:54:53 PM by LJ Analysis Method : D:/LC/DATA/LK/LK-L4-20B/LK-14-20B 2016-05-10 14-44-27/083-0201.D/DA.M (
ASH-20-80-1ML-2200M.M) Last changed : $6/6/2016$ 8:41:25 AM by HR (modified after loading)
W/D1 A, Wavelength=220 nm (D/LC/DATA/LK/LK-14-208/LK-14-208 2016-05-10 14-44-27/083-0201.D)
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500
300 -
200 -
0 2 4 6 8 10 12 14 min
Area Percent Report
Control Terror Control
sorrea by ; signal Multinlier ; 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs
Signal 1: VWD1 A, Wavelength=220 nm
Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] % 
1 5.781 MF 0.3595 1.21737e4 564.30969 50.7366
z 7.001 MM 0.5453 1.18202e4 361.25134 49.2634
Totals : 2.39938e4 925.56104
THE OF HERE

Instrument 1 6/6/2016 8:41:33 AM HR

Data File D:\LC\DATA\LK\LK-14-23B\LK-14-23B 2016-05-24 19-24-23\093-0201.D Sample Name: LK-14-23B Acq. Operator : WZF Seq. Line : 2 Acq. Instrument : Instrument 1 Location : Vial 93 Injection Date : 5/24/2016 7:36:58 PM Ini : 1 Inj Volume : 5 µl : D:\LC\DATA\LK\LK-14-23B\LK-14-23B 2016-05-24 19-24-23\ASH-10-90-1ML-220MM-Acq. Method 22MIN.M Last changed : 11/9/2015 3:28:40 PM by LHC Analysis Method : D:\LC\DATA\LK\LK-14-23B\LK-14-23B 2016-05-24 19-24-23\093-0201.D\DA.M ( ASH-10-90-1ML-220MM-22MIN.M) Last changed : 6/6/2016 8:45:06 AM by HR (modified after loading) W/D1 A, Wavelength=220 nm (DALCXDATANLK1K1423BLK-1423B 2016-05-24 19-2423'093-0201.D) mAU ] 0 1200 1000 800 600 400 200 9.D71 D-10 12 14 6 ś min Area Percent Report -Sorted By Signal 1 1.0000 : Multiplier 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Area Peak RetTime Type Width Height Area # [min] [min] mAU \*5 [mAU] % 1 7.140 W 0.4786 4.50153e4 1383.45776 91.0685 2 9.071 VB 0.6749 4414.85547 96.51945 8.9315 Totals : 4.94301e4 1479.97721 \_\_\_\_\_ \*\*\* End of Report \*\*\*

Instrument 1 6/6/2016 8:45:12 AM HR



Data File D:\LC\DATA\LK\LK-14-21\LK-14-21 2016-05-24 20-07-29\094-0201.D Sample Wame: LK-14-21

Acq. Operator : 027 Seq. Line : 2	
Acq. Instrument : Instrument I Location : Vial 94	
1/1 ματροποία τη την την την την την την την την την	м
A. Q. MCLIND . D. (D.(DALA)DA DA 14-21(DA 14-21 2010-03-24 20-01-23(A)H-20-00-HH-220M. Last channed . 10/17/2015 3:54.53 DM by L.	
hast changed (1, 10,17,2013) (3,34,35,17,17,10,2013) and by ho hast we is Mathinal (1,11,17,11,11,11,11,11,11,11,11,11,11,11	{_
20.80-10122000 M	
Last changed : $6/6/2016$ 8:47:12 M by HR	
(modified after loading)	
WVD1 A, Wavelength=220 nm (D:\LCDATA\LK1K1421\LK-1421 2016-05-2420-07-29094-0201.D)	
mAU 1	
1 A 20 <sup>0</sup>	
20-1 (1/4*)*	
15 –	
6-1 ) \ \ \	
<u>6 10 15 20 25 30 38 44</u>	) min
Area Percent Report	
Sorted By : Signal	
Multiplier : 1.0000	
Dilution : 1.0000	
Use Multiplier & Dilution Factor with ISTDs	
Signal 1: VWD1 A, Wavelength=220 nm	
Peak RetTime Type Width Area Height Area	
# [min] [min] m&U *s [m&U ] %	
1 16.041 MM 2.3034 3129.03149 22.64103 50.0559	
2 26.387 MM 3.6600 3122.04028 14.21681 49.9441	
Totals : 6251.07178 36.85784	
*** End of Report ***	

Instrument 1 6/6/2016 8:47:18 AM HR

Data File D:\LC\DATA\LK\LK-14-27B\LK-14-27B 2016-05-27 14-39-00\011-0201.D Sample Wame: LK-14-27B

\_\_\_\_\_ Acq. Operator : W2F Acq. Instrument : Instrument 1 Seq. Line : 2 Location : Vial 11 Injection Date : 5/27/2016 2:51:43 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\LK\LK-14-27B\LK-14-27B 2016-05-27 14-39-00\ASH-20-80-1ML-220MM-Acq. Method 40MIN.M Last changed : 5/25/2016 8:53:10 AM by WZF Analysis Method : D:\LC\DATA\LK\LK-14-27B\LK-14-27B 2016-05-27 14-39-00\011-0201.D\DA.M ( ASH-20-80-1ML-220MM-40MIN.M) Last changed : 6/6/2016 8:49:16 AM by HR (modified after loading) W/D1 A Wavelength=220 nm(DALCXDATAVLK14/14/278/LK-14/278/2016-05-27/14/39-00/011-0201.D) 1.00 N mAU 50 -40 30 -16.583 20 10 -0 15 25 35 10 20 зb min Area Percent Report -Sorted By Signal 1 1.0000 : Multiplier 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Area Peak RetTime Type Width Height Area # [min] [min] mAU \*5 [mAU] % 1 16.583 BB 1.1739 1453.69763 14.59158 12.0415 2 26.141 MM 3.0553 1.06187e4 57.92581 87.9585 Totals : 1.20724e4 72.51739 \_\_\_\_\_ \*\*\* End of Report \*\*\*

Instrument 1 6/6/2016 8:49:22 AM HR



Data File D:\LC\DATA\LK\LK-12-72A-110\LK-12-72A-110 2015-12-09 21-41-22\044-0101.D Sample Wame: LK-12-72A

Acq. Operator :	LHC Seq. Line : 1
Acq. Instrument :	Instrument 1 Location : Vial 44
Injection Date :	12/9/2015 9:42:59 PM Inj: 1
	Inj Volume : 5 µl
Acq. Method	D:\LC\DATA\LK\LK-12-72A-110\LK-12-72A-110 2015-12-09 21-41-22\ICH-50-50-
	1ML-220NM.M
Last changed :	12/9/2015 10:08:17 PM by LHC
	(modified after loading)
Analysis Method :	D: \LC\DATA\LK\LK-12-72A-110\LK-12-72A-110\2015-12-09\21-41-22\044-0101.D\
	DA.M (ICH-50-50-IML-220MM.M)
Last changed :	5/24/2016 9:16:26 PM by W2F
VI0/D1 A 306-	(modified after loading) North-220 pm/DM (CDATA) M/ K12 72A 1100 K 12 72A 110 2015 12 00 21 41 22044 0101 FD
	sergui=220 mm(DXEC4041AEN12472AE11042N124724E110201341240821441-2204440101.0)
mAU -	
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	Area Percent Report
Control Dec	01
Sorted By	s signal
Mult ipiler	
Dilution	: 1.0000
Use Multiplier &	Dilution Factor with ISTDs
Signal I: VWDI A,	wavelength=220 nm
Deals Date: 5	This has a straight have
Peak RetTime Type	Width Area Height Area
# [min]	iminj mau *5 [mau j %
1 14.777 VB	0.1663 9901.50586 197.21677 50.1775
2 20.756 VB	1.1565 9831.43555 125.17695 49.8225
Totals :	1.97329e4 322.39371

Instrument 1 5/24/2016 9:16:33 PM WZF

Page l of l

Data File D:\LC\DATA\LK\LK-12-72A-110\LK-12-72A-110 2015-12-09 22-10-37\045-0101.D Sample Wame: LK-12-110

Acq. Operator	: LHC Seq. Line : 1
Acq. Instrument	: Instrument l Location : Vial 45
Injection Date	: 12/9/2015 10:12:39 PM Inj: 1
	Inj Volume : 5 µl
Acq. Method	: D:\LC\DATA\LK\LK-12-72A-110\LK-12-72A-110 2015-12-09 22-10-37\ICH-50-50- 1ML-220MM-30MIN.M
Last changed	: 12/9/2015 10:09:50 PM by LHC
Analysis Method	: D:\LC\DATA\LK\LK-12-72A-110\LK-12-72A-110 2015-12-09 22-10-37\045-0101.D\
Last changed	: 6/6/2016 10:21:36 AM by HR
VI0/D1 A 3065	(modified after loading)
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Dilution	: 1.0000
Use Multiplier &	Dilution Factor with ISTDs
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Simual 1: VMD1 A	. Wavelenαth=220 nm
bight in 1.001 in	, werezengen beo ian
Peak RetTime Typ	e Width Area Height Area
# [min]	[min] mAU *s [mAU ] %
1 14.071 MM 2 20 572 MM	U.0014 1113.00091 - 66.11333 - 1.0000 1 3218 1 378366 - 173 20756 - 02 1320
2 20.312 PH	1.3210 1.3143464 113.27130 72.1320
Totals :	1.4917le4 196.01291
	*** Fnd of Deport ***
	THE OF REPORT

Instrument 1 6/6/2016 10:21:41 AM HR

Page 1 of 1

S102



Data File D:\LC\DATA\LK\LK-12-72B-ICH\LK-12-72B-ICH 2015-12-09 15-53-59\036-0201.D Sample Wame: LK-12-72B-ICH

```
_____
                                              Seq. Line : 2
   Acq. Operator : LHC
   Acq. Instrument : Instrument 1
                                               Location : Vial 36
   Injection Date : 12/9/2015 4:06:50 PM
                                                    Inj: 1
                                              Inj Volume : 5 µl
               : D:\LC\DATA\LK\LK-12-72B-ICH\LK-12-72B-ICH 2015-12-09 15-53-59\ICH-30-70-
   Acg. Method

        IML-220WM.M

        Last changed
        : 12/9/2015 3:42:20 PM by LHC

        Analysis Method
        : D:\LC\DATA\LK\LK-12-72B-ICH\LK-12-72B-ICH 2015-12-09 15-53-59\036-0201.D\

                  DA.M (ICH-30-70-1ML-220NM.M)
   Last changed
                 : 5/24/2016 9:21:36 PM by WZF
          (modified after loading)

W/D1 A. Wavelergth=220 nm (DALC/DATALKLK:12-728-ICHLK-12-728-ICH2015-12-09 15-53-59/036-0201.D)
     mAU
                                          8
                                                         18,987
       250
       200
       150
       100
       50
        0
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                              14
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                                                                        22
                    12
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                                                                                      min
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                         Area Percent Report
   _____
   Sorted By
                       :
                             Simal
   Multiplier
                      :
                             1.0000
                      ;
                             1.0000
   Dilution
   Use Multiplier & Dilution Factor with ISTDs
   Signal 1: VWD1 A, Wavelength=220 nm
   Peak RetTime Type Width
   Area
     1 16.229 BB 0.6656 1.31428e4 300.75595 49.9097
2 18.987 BB 0.8255 1.31903e4 240.76797 50.0903
                          2.63331e4 541.52393
   Totals :
   -------
                          *** End of Report ***
Instrument 1 5/24/2016 9:21:41 PM WZF
                                                                                 Page 1 of 1
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Data File D:\LC\DATA\LK\LK-12-76B\LK-12-76B 2015-12-09 16-35-21\044-0101.D Sample Name: LK-12-76B \_\_\_\_\_ Acq. Operator : LHC Acq. Instrument : Instrument 1 Seq. Line : 1 Location : Vial 44 Injection Date : 12/9/2015 4:36:53 PM Ini : 1 Inj Volume : 5 µl : D:\LC\DATA\LK\LK-12-76B\LK-12-76B 2015-12-09 16-35-21\ICH-30-70-1ML-220MM-Acq. Method 25MIM.M Last changed : 12/9/2015 4:34:16 PM by LHC Analysis Method : D:\LC\DATA\LK\LK-12-76B\LK-12-76B 2015-12-09 16-35-21\044-0101.D\DA.M ( ICH-30-70-1ML-220MM-25MIN.M) Last changed : 5/24/2016 9:19:55 PM by WZF (modified after 1 oading) W/DI A, Wavelength=220 nm (D/LC/DATA/LKLK12-768/LK12-768/2015-12-09 16-35-21/0440101.D) mAU . They're 400 350 300 -250 200 150 -17 100<sup>-1402<sup>4</sup></sup> 100 50 ۵ 14 16 18 20 22 24 12 min Area Percent Report Sorted By Signal 1 1.0000 : Multiplier 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Area Peak RetTime Type Width Height Area # [min] [min] mAU \*5 [mAU] % 1 16.409 MM 0.6876 1746.23743 42.32442 7.0978 2 18.992 MM 0.9128 2.28564e4 417.33026 92.9022 Totals : 2.46027e4 459.65469 \_\_\_\_\_ \*\*\* End of Report \*\*\*

Instrument 1 5/24/2016 9:20:00 PM WZF