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Supporting Information

Chiral Phosphoric Acid-Catalyzed Asymmetric Transfer Hydrogenation of 3-Trifluoromethylthioquinoline

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1. General:

Commercially available reagents were used without further purification. Solvents were treated prior to use according to the standard methods. ¹H NMR, ¹³C NMR and ¹⁹F NMR spectra were recorded at room temperature in CDCl₃ on 400 MHz instrument with tetramethylsilane (TMS) as internal standard. Enantiomeric excess was determined by HPLC analysis, using chiral column described below in detail. Optical rotations were measured by polarimeter. Flash column chromatography was performed on silica gel (200-300 mesh).

2. Synthesis of 3-Trifluoromethylthioquinolines

3-Trifluoromethylthioquinoline derivatives can be synthesized by **Path A** and **B**. The **Path A** starts from quinoline *N*-oxide, which can be transformed to 2-chloro-3-aminoquinolines **1** according to the known literatures.¹⁻³ The compound **1** was then successfully converted to 2-chloro-3-trifluoromethylthioquinoline **2** following the report by Goossen's group.⁴ Lastly, we coupled compound **2** with boronic acids to give the 2-substituted 3-trifluoromethylthioquinoline **3**. The **Path B** starts from the 3-aminoquinoline **4**, formal substitution of amino by SCF₃ group to give the 3-trifluoromethylthioquinoline **3k**. The compound **3k** was oxidized by 3-choroperbenzoic to get 3-trifluoromethylthioquinoline *N*-oxide and then reacted with aryl boronic acids in dimethyl sulfoxide to give 2-substituted 3-trifluoromethylthioquinoline **3k**,⁴ 3-aminoquinoline **4**⁷ are known.

2.1. Synthesis of 2-Substituted 3-Trifluoromethylthioquinolines via Path A





In a 50 mL round-bottom flask, 2-chloro-3-aminoquinoline **1a** (7.081 g, 40 mmol) was dissolved in a mixture of absolute ethanol (12.0 mL) and the aqueous solution of HBF₄ (40%, 13.9 mL, 80 mmol), then the *tert*-butyl nitrite (10.6 mL, 5.250g, 80 mmol) was added dropwise to the mixture at 0 °C. The reaction was stirred at room temperature for 1 h and 30 mL diethyl ether was added to precipitate the arenediazonium tetrafluoroborate which was filtered off and washed with diethyl ether (10 mL×5). The arenediazonium tetrafluoroborate was dried in *vacuo* and was then directly used without further purification.

Under nitrogen atmosphere, to a 250 mL Schlenk flask was added copper thiocyanate (2.156 g, 17.6 mmol), sodium thiocyanate (4.356 g, 52.7 mmol), cesium carbonate (23.104 g, 70.2 mmol) and acetonitrile (90 mL), the resulting suspension was stirred at room temperature for 20 minutes. The solution of the arenediazonium tetrafluoroborate (9.720 g, 35.1 mmol) in acetonitrile (110 mL) was added dropwise and the reaction mixture was stirred for 1 h. Trifluoromethyltrimethylsilane (10.3 mL, 70.2 mmol) was added at once and the mixture was stirred at room temperature for 36 h. The resulting mixture was filtered and rinsed with diethyl ether. The organic solution was then washed with water (50 mL) and brine (50 mL). The organic layer was dried with sodium sulfate, filtered and concentrated. The residue was further purified by silica gel column chromatography to get the 2-chloro-3-trifluoromethylthioquinoline 2a 2.795 g in 26% yield (eluent: petroleum

ether/ethyl acetate).

2-Chloro-3-(trifluoromethyl)quinoline (2a): unknown compound, 2.795 g, 26% yield, white solid, mp 76-77 °C, $R_f = 0.60$ (petroleum ether/ethyl acetate 10:1). ¹H NMR (400 MHz, CDCl₃) δ SCF₃ $\begin{cases} 8.59 \text{ (s, 1H), 8.05 (d, J = 8.4 Hz, 1H), 7.93-7.77 (m, 2H), 7.72-7.56 (m, 1H);} \\ {}^{13}C \text{ NMR (100 MHz, CDCl₃)} \delta = 153.2, 148.2, 148.0, 132.9, 129.2 (q, {}^{1}J_{FC} = 308 \text{ Hz}), 128.8, 128.3, 128.1, 127.2, 119.1 (q, {}^{3}J_{FC} = 2.0 \text{ Hz}); {}^{19}\text{F NMR (376)} \end{cases}$

MHz, CDCl₃) δ = -42.0; HRMS (ESI) *m*/*z* Calculated for C₁₀H₆ClF₃NS [M+H]⁺ 263.9856, found 263.9860.

6-Methoxy-2-chloro-3-(trifluoromethylthio)quinoline (2b): unknown compound, 190 mg, 18% yield, white solid, mp 83-84 °C, $R_f = 0.65$ (petroleum ether/ethyl acetate 10:1). ¹H NMR (400

MHz, CDCl₃) δ 8.47 (s, 1H), 7.94 (d, J = 9.2 Hz, 1H), 7.47 (dd, J = 9.2, 2.8 Hz, 1H), 7.09 (d, J = 2.8 Hz, 1H), 3.95 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 159.0, 150.4, 146.4, 144.3, 130.1, 129.2 (q, ¹ $J_{FC} = 308$ Hz),

128.5, 125.7, 119.2 (d, ${}^{3}J_{FC} = 2.0 \text{ Hz}$), 105.2, 55.9; ${}^{19}\text{F}$ NMR (376 MHz, CDCl₃) $\delta = -41.9$. HRMS (ESI) m/z Calculated for C₁₁H₈ClF₃NOS [M+H]⁺ 293.9967, found 293.9970.

A mixture of 2-chloro-3-trifluoromethylthioquinoline **2a** (263.7 mg, 1.0 mmol), boronic acid (1.2 mmol, 1.2 equiv), Pd(PPh₃)₄ (58 mg, 0.05 mmol, 5 mol%) and potassium carbonate (415 mg, 3.0 mmol, 3.0 equiv) in DME (12 mL) was stirred at reflux. When the reaction was complete (determined by TLC), the reaction mixture was diluted with water (15 mL), then extracted with dichloromethane (50 mL×4). The combined organic layer was dried with sodium sulfate. After filtration, the solvent was removed under the reduced pressure and the residue was purified by flash chromatography on silica gel to provide the product 3-trifluoromethylthioquinolines **3** (eluent: petroleum ether/dichloromethane).

2-Phenyl-3-(trifluoromethyl)quinoline (3a): unknown compound, 371 mg, 81% yield, white solid, mp 87-88 $^{\circ}$ C, R_f = 0.60 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃)



δ 8.66 (s, 1H), 8.19 (d, J = 8.5 Hz, 1H), 7.91 (d, J = 8.1 Hz, 1H), 7.84 (ddd, J = 8.4, 7.0, 1.4 Hz, 1H), 7.71-7.57 (m, 3H), 7.57-7.41 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 162.1, 148.3, 146.1, 139.6, 132.0, 129.8, 129.8, 129.4 (q, ¹ $J_{\rm FC} = 307$ Hz), 129.1, 128.3, 127.9, 127.8, 127.3, 118.2 (d, ³ $J_{\rm FC} = 1.8$ Hz); ¹⁹F

NMR (376 MHz, CDCl₃) δ = -42.0; HRMS (ESI) *m*/*z* Calculated for C₁₆H₁₁F₃NS [M+H]⁺ 306.0559, found 306.0561.

2-*m***-Tolyl-3-(trifluoromethylthio)quinoline (3b):** unknown compound, 242 mg, 76% yield, white solid, mp 43-44 $^{\circ}$ C, R_f = 0.60 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz,



CDCl₃) δ 8.65 (s, 1H), 8.20 (d, *J* = 8.5 Hz, 1H), 7.90 (d, *J* = 8.2 Hz, 1H), 7.87-7.79 (m, 1H), 7.64 (t, *J* = 7.5 Hz, 1H), 7.41 (dd, *J* = 16.1, 11.2 Hz, 3H), 7.31 (d, *J* = 4.3 Hz, 1H), 2.47 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 162.2, 148.2, 145.7, 139.5, 138.1, 131.9, 130.3, 129.8, 129.8, 129.4 (q,

 ${}^{1}J_{\text{FC}} = 308 \text{ Hz}$), 128.1, 127.8, 127.7, 127.3, 126.9, 118.4 (d, ${}^{3}J_{\text{FC}} = 1.8 \text{ Hz}$), 21.7; ¹⁹F NMR (376 MHz, CDCl₃) $\delta = -41.9$. HRMS (ESI) *m*/*z* Calculated for C₁₇H₁₃F₃NS [M+H]⁺ 320.0715, found 330.0716.

2-*p***-Tolyl-3-(trifluoromethylthio)quinoline (3c):** unknown compound, 200 mg, 63% yield, white solid, mp 97-98 °C, $R_f = 0.50$ (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃)



δ 8.65 (s, 1H), 8.19 (d, J = 8.5 Hz, 1H), 7.89 (d, J = 8.1 Hz, 1H), 7.83 (t, J = 7.5 Hz, 1H), 7.62 (t, J = 7.4 Hz, 1H), 7.55 (d, J = 7.7 Hz, 2H), 7.33 (d, J = 7.7 Hz, 2H), 2.46 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 162.1, 148.3, 145.8, 139.0, 136.7, 131.8, 129.8, 129.8, 129.4 (q, ¹J_{FC} = 307 Hz), 129.0,

127.8, 127.6, 127.2, 118.2 (d, ${}^{3}J_{FC} = 1.8$ Hz), 21.6; ${}^{19}F$ NMR (376 MHz, CDCl₃) $\delta = -42.0$; HRMS (ESI) *m*/*z* Calculated for C₁₇H₁₃F₃NS [M+H]⁺ 320.0715, found 320.0718.

2-(3-Methoxyphenyl)-3-(trifluoromethylthio)quinoline (3d): unknown compound, 168 mg, 79% yield, white solid, mp 53-54 °C, $R_f = 0.40$ (petroleum ether/dichloromethane 1:1). ¹H NMR (400



MHz, CDCl₃) δ 8.65 (s, 1H), 8.19 (d, J = 8.5 Hz, 1H), 7.89 (d, J = 8.2 Hz, 1H), 7.83 (t, J = 7.7 Hz, 1H), 7.63 (t, J = 7.5 Hz, 1H), 7.41 (t, J = 7.9 Hz, 1H), 7.17 (d, J = 7.6 Hz, 2H), 7.03 (d, J = 8.5 Hz, 1H), 3.87 (s, 3H++); ¹³C NMR (100 MHz, CDCl₃) $\delta = 161.8$, 159.5, 148.1, 145.8,

140.8, 131.9, 129.7, 129.4, 129.4 (q, ${}^{1}J_{FC} = 307$ Hz), 127.8, 127.8, 127.3, 122.2, 118.2 (d, ${}^{3}J_{FC} = 1.8$ Hz), 115.1, 115.0, 55.5; ${}^{19}F$ NMR (376 MHz, CDCl₃) $\delta = -41.9$. HRMS (ESI) *m*/*z* Calculated for C₁₇H₁₃F₃NOS [M+H]⁺ 336.0664, found 336.0666.

2-(4-Methoxyphenyl)-3-(trifluoromethylthio)quinoline (3e): unknown compound, 281 mg, 84% yield, white solid, mp 136-137 $^{\circ}$ C, R_f = 0.50 (petroleum ether/dichloromethane 1:1). ¹H NMR



(400 MHz, CDCl₃) δ 8.63 (s, 1H), 8.18 (d, *J* = 8.5 Hz, 1H), 7.88 (d, *J* = 8.2 Hz, 1H), 7.82 (t, *J* = 7.7 Hz, 1H), 7.62 (dd, *J* = 12.4, 5.7 Hz, 3H), 7.04 (d, *J* = 8.7 Hz, 2H), 3.89 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 161.6, 160.4, 148.3, 146.0, 132.0, 131.8, 131.4, 129.7, 127.8, 127.5,

129.4 (q, ${}^{1}J_{FC}$ = 308 Hz), 127.1, 118.2 (d, ${}^{3}J_{FC}$ = 1.7 Hz), 113.8, 55.5; 19 F NMR (376 MHz, CDCl₃) δ = -42.0. HRMS (ESI) *m*/*z* Calculated for C₁₇H₁₃F₃NOS [M+H]⁺ 336.0664, found 336.0666.

2-(4-*tert***-Butylphenyl)-3-(trifluoromethylthio)quinoline (3f):** unknown compound, 286 mg, 79% yield, white solid, mp 124-125 °C, $R_f = 0.55$ (petroleum ether/dichloromethane 1:1). ¹H NMR



(400 MHz, CDCl₃) δ 8.63 (s, 1H), 8.17 (d, J = 8.5 Hz, 1H), 7.88 (d, J = 8.2 Hz, 1H), 7.80 (ddd, J = 8.4, 7.0, 1.4 Hz, 1H), 7.63-7.55 (m, 3H), 7.54-7.49 (m, 2H), 1.38 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) $\delta = 161.2$, 152.1, 148.3, 145.7, 136.7, 131.8, 129.8, 129.6, 129.5 (q, ¹ $J_{FC} = 307$ Hz),

127.8, 127.6, 127.2, 125.3, 118.3, 35.0, 31.5; ¹⁹F NMR (376 MHz, CDCl₃) δ = -41.9. HRMS (ESI) *m*/*z* Calculated for C₂₀H₁₉F₃NS [M+H]⁺ 362.1185, found 362.1187.

3-(Trifluoromethylthio)-2+-(4-(trifluoromethyl)phenyl)-quinoline (3g): unknown compound, 517 mg, 92% yield, white solid, mp 118-119 $^{\circ}$ C, R_f = 0.65 (petroleum ether/dichloromethane 1:1).



¹H NMR (400 MHz, CDCl₃) δ 8.71 (s, 1H), 8.19 (d, J = 8.5 Hz, 1H), 7.94 (d, J = 8.1 Hz, 1H), 7.88 (t, J = 7.5 Hz, 1H), 7.77 (s, 4H), 7.69 (d, J = 7.5 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ = 160.9, 148.4, 147.1, 143.1, 131.2, 130.9, 129.9, 129.3 (q, ¹ $J_{FC} = 206$ Hz), 129.2 (q, ¹ $J_{FC} = 308$ Hz),

128.0, 127.5, 125.7, 125.3 (q, ${}^{1}J_{FC} = 3.8 \text{ Hz}$), 123.0, 117.6 (d, ${}^{3}J_{FC} = 1.8 \text{ Hz}$); ${}^{19}\text{F}$ NMR (376 MHz, CDCl₃) $\delta = -42.1$, -62.7. HRMS (ESI) *m*/*z* Calculated for C₁₇H₁₀F₆NS [M+H]⁺ 374.0433, found 374.0435.

2-(4-Fluorophenyl)-3-(trifluoromethylthio)quinoline (3h): unknown compound, 268 mg, 83% yield, white solid, mp 86-87 °C, $R_f = 0.45$ (petroleum ether/dichloromethane 1:1). ¹H NMR (400



MHz, CDCl₃) δ 8.66 (s, 1H), 8.17 (d, J = 8.5 Hz, 1H), 7.90 (d, J = 8.2 Hz, 1H), 7.84 (t, J = 7.7 Hz, 1H), 7.70-7.53 (m, 3H), 7.19 (t, J = 8.7 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ = 164.7, 162.2, 161.1, 148.4, 146.6, 135.6, 132.0, 131.9, 131.0 (q, ¹J_{FC} = 242 Hz), 129.3 (q, ¹J_{FC} = 308 Hz), 127.9,

127.9, 117.9 (d, ${}^{3}J_{\text{FC}} = 1.7 \text{ Hz}$), 115.5; ${}^{19}\text{F}$ NMR (376 MHz, CDCl₃) $\delta = -42.1$, -112.5. HRMS (ESI) m/z Calculated for C₁₆H₁₀F₄NS [M+H]⁺ 324.0465, found 324.0467.

2-(Naphthalen-2-yl)-3-(trifluoromethylthio)quinoline (3i): unknown compound, 432 mg, 81% yield, white solid, mp 114-115 $^{\circ}$ C, R_f = 0.50 (petroleum ether/dichloromethane 1:1). ¹H NMR



(400 MHz, CDCl₃) δ 8.69 (s, 1H), 8.21 (d, J = 8.3 Hz, 1H), 8.11 (s, 1H), 8.03-7.80 (m, 5H), 7.75 (d, J = 8.1 Hz, 1H), 7.64 (t, J = 7.3 Hz, 1H), 7.59-7.43 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) $\delta = 162.0$, 148.4, 146.1, 137.0, 133.5, 133.1, 132.0, 129.8, 129.7, 129.4 (q, ¹ $_{JFC} = 308$ Hz), 128.8,

128.0, 128.0, 127.9, 127.3, 127.3, 127.0, 126.6, 125.3, 118.4 (d, ${}^{3}J_{FC} = 1.8$ Hz); ${}^{19}F$ NMR (376 MHz, CDCl₃) $\delta = -42.0$. HRMS (ESI) *m*/*z* Calculated for C₂₀H₁₃F₃NS [M+H]⁺ 356.0715, found 356.0712.

6-Methoxy-2-phenyl-3-(trifluoromethylthio)quinoline (3j): unknown compound, 128 mg, 80% yield, white solid, mp 143-144 $^{\circ}$ C, R_f = 0.50 (petroleum ether/dichloromethane 1:1). ¹H NMR (400



MHz, CDCl₃) δ 8.53 (s, 1H), 8.07 (d, J = 9.2 Hz, 1H), 7.60 (d, J = 6.3 Hz, 2H), 7.48 (t, J = 6.1 Hz, 4H), 7.13 (d, J = 2.2 Hz, 1H), 3.96 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) $\delta = 159.6$, 158.7, 144.5, 144.4, 139.6, 131.2, 129.9, 129.4 (q, ¹ $J_{FC} = 308$ Hz), 128.8, 128.4, 128.3, 125.0, 118.4

(d, ${}^{3}J_{FC} = 1.7$ Hz), 104.8, 55.9; 19 F NMR (376 MHz, CDCl₃) $\delta = -42.0$. HRMS (ESI) m/zCalculated for C₁₇H₁₃F₃NOS [M+H]⁺ 336.0664, found 336.0666.

2-Methyl-3-(trifluoromethyl)quinoline (3l): unknown compound, 168 mg, 70% yield, white solid, mp 70-71 $^{\circ}$ C, R_f = 0.25 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ

8.49 (s, 1H), 8.05 (d, J = 8.4 Hz, 1H), 7.79 (dd, J = 14.1, 7.7 Hz, 2H), 7.55 (t, J = 7.5 Hz, 1H), 2.94 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 161.8, 148.7, 147.0, 131.9, 129.6 (q, ¹ $J_{FC} = 307$ Hz), 128.9, 127.9, 127.2, 127.0, 118.4 (q, ³ $J_{FC} = 1.9$

Hz), 24.8; ¹⁹F NMR (376 MHz, CDCl₃) δ = -42.3. HRMS (ESI) *m*/*z* Calculated for C₁₁H₉F₃NS [M+H]⁺ 244.0402, found 244.0402.

2.2. Synthesis of 2-Substituted 3-Trifluoromethylthioquinolines via Path B



3-Trifluoromethylthioquinoline 3k was prepared according to the known procedure starting from the 3-aminoquinoline 4.⁴

To the solution of 3-trifluoromethylthioquinoline 3k (1.000 g, 4.4 mmol) in dichloromethane (40 mL), 3-chloroperbenzoic acid (1.307 g, 5.3 mmol) was added and the reaction was stirred for 24 h at room temperature. Afterwards residual 3-chloroperbenzoic acid was removed by adding sodium hydroxide solution (1.0 M, 30 mL×3). The aqueous phase was extracted three times with dichloromethane and the combined organic layers were dried over sodium sulfate. The mixture

was treated in a usual manner to give a crude product and the resulting solid was purified by silica gel column chromatography to provide the 3-(trifluoromethylthio)quinoline *N*-oxide 936 mg in 87% yield (eluent: ethyl acetate/methanol).

Under nitrogen atmosphere, to the solution of 3-(trifluoromethylthio)quinoline *N*-oxide (0.6 mmol, 147 mg) in dimethyl sulfoxide (1.0 mL), aryl boronic acid (1.8 mmol, 3.0 eq.) was added and the reaction was stirred at 110 $^{\circ}$ C. Afterwards, the reaction was cooled to room temperature and diluted with dichloromethane. Column chromatography of the reaction mixture provided the pure product **3** (eluent: petroleum ether/dichloromethane).

| Entry | R | Weight (mg) | Yield (%) |
|-------|------------------------------------|-------------|------------------|
| 1 | Ph | 50 | 28 (3a) |
| 2 | $3-MeC_6H_4$ | 68 | 36 (3b) |
| 3 | $4-\text{MeC}_6\text{H}_4$ | 70 | 37 (3c) |
| 4 | 3-MeOC ₆ H ₄ | 25 | 12 (3d) |
| 5 | $4-MeOC_6H_4$ | 78 | 39 (3e) |
| 6 | 4 - $^{t}BuC_{6}H_{4}$ | 98 | 45 (3f) |
| 7 | $4-CF_3C_6H_4$ | 93 | 42 (3g) |
| 8 | $4-FC_6H_4$ | 80 | 42 (3h) |
| 9 | 2-Naphthyl | 87 | 41 (3i) |

Table S1. The Results for the Final Step of Path B

3. Asymmetric Transfer Hydrogenation of 3-Trifluoromethylthioquinlines



The mixture of 3-trifluoromethylthioquinolines **3** (0.15 mmol), Hantzsch ester **5b** (81 mg, 0.36 mmol, 2.4 equiv), and chiral phosphoric acid (*S*)-**6b** (5.6 mg, 0.005 mmol, 5 mol%) in diethyl ether (4.5 mL) was stirred under nitrogen for 36-72 h at 50 $^{\circ}$ C. After the reaction was complete (determined by TLC), the solvent was removed under reduced pressure. Purification was performed by silica gel column eluted with petroleum ether/dichloromethane to give the desired products **7**. The enantiomeric excesses were determined by chiral HPLC.

(25,35)-(+)-2-Phenyl-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7a): 44 mg, 94%

SCF₃ yield, unknown compound, 99% ee, white solid, mp 69-70 °C, $[\alpha]^{20}_{D} = +38.30$ (*c* 0.53, CHCl₃), R_f = 0.65 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.52-7.28 (m, 5H), 7.07 (dd, *J* = 16.0, 7.8 Hz, 2H), 6.76 (t, *J* = 7.4 Hz, 1H), 6.61 (d, *J* = 7.9 Hz, 1H), 4.80 (s, 1H), 4.13 (s, 1H), 3.89

(dd, J = 7.0, 4.5 Hz, 1H), 3.51 (dd, J = 16.6, 4.3 Hz, 1H), 3.16 (dd, J = 16.6, 5.1 Hz, 1H); ¹³C

NMR (100 MHz, CDCl₃) δ = 143.7, 140.1, 130.2, 128.6, 128.6 (q, ${}^{1}J_{FC}$ = 306 Hz), 128.5, 127.9, 127.4, 119.0, 118.1, 114.9, 59.0, 46.0, 35.5; ${}^{19}F$ NMR (376 MHz, CDCl₃) δ = -39.6. HRMS (ESI) *m*/*z* Calculated for C₁₆H₁₅F₃NS [M+H]⁺ 310.0872, found 310.0866. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 75/25, flow = 0.7 mL/min, retention time 14.4 min and 21.5 min (major).

(2S,3S)-(+)-2-*m*-Tolyl-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7b): 46 mg, 95% yield, unknown compound, 96% ee, white solid, mp 95-96 °C, $[\alpha]^{20}_{D} = +41.74$ (*c* 0.92, CHCl₃), R_f



= 0.80 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.29-7.22 (m, 1H), 7.22-7.12 (m, 3H), 7.11-7.01 (m, 2H), 6.76 (td, J = 7.4, 1.0 Hz, 1H), 6.62 (t, J = 8.4 Hz, 1H), 4.76 (s, 1H), 4.09 (s, 1H), 3.88 (dd, J = 7.2, 4.5 Hz, 1H), 3.51 (dd, J = 16.6, 4.3 Hz, 1H), 3.16

(dd, J = 16.6, 4.8 Hz, 1H), 2.37 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) $\delta = 143.8$, 140.0, 138.3, 130.2, 129.2, 128.7 (q, ¹ $J_{FC} = 305$ Hz), 128.5, 128.0, 127.9, 124.4, 119.0, 118.2, 114.9, 59.0, 46.0, 35.7, 21.8; ¹⁹F NMR (376 MHz, CDCl₃) $\delta = -39.5$. HRMS (ESI) m/z Calculated for C₁₇H₁₇F₃NS [M+H]⁺ 324.1028, found 324.1021. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 75/25, flow = 0.7 mL/min, retention time 11.3 min and 14.1 min (major).

(2S,3S)-(+)-2-*p*-Tolyl-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7c): 47 mg, 97% yield, unknown compound, 99% ee, colorless oil, $[\alpha]_{D}^{20} = +31.50$ (*c* 0.80, CHCl₃), R_f = 0.80



(petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.26 (d, *J* = 7.8 Hz, 2H), 7.17 (d, *J* = 7.8 Hz, 2H), 7.06 (dd, *J* = 15.7, 7.8 Hz, 2H), 6.74 (t, *J* = 7.3 Hz, 1H), 6.58 (d, *J* = 7.8 Hz, 1H), 4.74 (s, 1H), 4.07 (s, 1H), 3.86 (dd, *J* = 7.2, 4.6 Hz, 1H), 3.48 (dd, *J* = 16.6, 4.3 Hz,

1H), 3.14 (dd, J = 16.6, 5.3 Hz, 1H), 2.35 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) $\delta = 143.8$, 138.2, 137.1, 130.1, 129.3, 128.7 (q, ¹ $J_{FC} = 305$ Hz), 127.9, 127.3, 118.9, 118.2, 114.8, 58.7, 45.8, 35.4, 21.3; ¹⁹F NMR (376 MHz, CDCl₃) $\delta = -39.5$. HRMS (ESI) m/z Calculated for C₁₇H₁₇F₃NS [M+H]⁺ 324.1028, found 324.1032. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 75/25, flow = 0.7 mL/min, retention time 11.4 min and 19.5 min (major).

(2*S*,3*S*)-(+)-2-(3-Methoxyphenyl)-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7d): 46 mg, 90% yield, unknown compound, 99% ee, colorless oil, $[\alpha]^{20}{}_{\rm D}$ = +56.31 (*c* 0.82, CHCl₃), R_f



= 0.85 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.29 (t, *J* = 7.9 Hz, 1H), 7.07 (dd, *J* = 16.1, 7.8 Hz, 2H), 6.97 (d, *J* = 7.9 Hz, 2H), 6.87 (dd, *J* = 8.3, 1.8 Hz, 1H), 6.76 (t, *J* = 7.4 Hz, 1H), 6.61 (d, *J* = 8.1 Hz, 1H), 4.76 (s, 1H), 4.12 (s, 1H), 3.89 (dd, *J* =

7.2, 4.5 Hz, 1H), 3.81 (s, 3H), 3.51 (dd, J = 16.6, 4.3 Hz, 1H), 3.16 (dd, J = 16.6, 5.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) $\delta = 159.8$, 143.7, 141.7, 130.2, 129.7, 128.7 (q, ¹ $J_{FC} = 305$ Hz), 127.9, 119.7, 119.1, 118.2, 114.9, 113.6, 113.3, 58.9, 55.5, 45.9, 35.6; ¹⁹F NMR (376 MHz, CDCl₃) $\delta = -39.5$. HRMS (ESI) m/z Calculated for C₁₇H₁₇F₃NOS [M+H]⁺ 340.0977, found 340.0976. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 85/15, flow = 0.7 mL/min, retention time 37.9 min and 40.6 min (major).

(2*S*,3*S*)-(+)-2-(4-Methoxyphenyl)-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7e): 50 mg, 98% yield, unknown compound, 99% ee, white solid, mp 65-66 °C, $[\alpha]^{20}{}_{\rm D}$ = +26.80 (*c* 1.0, CHCl₃), R_f = 0.65 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.35-7.27 (m, 2H), 7.12-7.01 (m, 2H), 6.93-6.86 (m, 2H), 6.75 (td, *J* = 7.5, 1.1 Hz, 1H), 6.59 (dd,



 $C_{17}H_{17}F_3NOS [M+H]^+$ 340.0977, found 340.0978. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 75/25, flow = 0.7 mL/min, retention time 15.7 min and 21.1 min (major).

(2*S*,3*S*)-(+)-2-(4-*tert*-Butylphenyl)-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7f): 53 mg, 97% yield, unknown compound, 98% ee, colorless oil, $[\alpha]^{20}{}_{\rm D}$ = +37.37 (*c* 0.80, CHCl₃), R_f



= 0.95 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.43-7.27 (m, 4H), 7.06 (dd, *J* = 14.9, 7.3 Hz, 2H), 6.75 (td, *J* = 7.5, 1.0 Hz, 1H), 6.59 (d, *J* = 7.9 Hz, 1H), 4.77 (s, 1H), 4.12 (s, 1H), 3.89 (dd, *J* = 7.4, 4.8 Hz, 1H), 3.48 (dd, *J* = 16.6, 4.4 Hz, 1H), 3.15 (dd,

J = 16.6, 5.4 Hz, 1H), 1.33 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) $\delta = 151.5, 143.8, 137.1, 133.3, 130.1, 128.7$ (q, ¹ $J_{FC} = 306$ Hz), 127.9, 127.1, 125.5, 118.9, 114.8, 58.7, 45.8, 35.3, 34.8, 31.5; ¹⁹F NMR (376 MHz, CDCl₃) $\delta = -39.5$. HRMS (ESI) m/z Calculated for C₂₀H₂₃F₃NS [M+H]⁺ 366.1498, found 366.1503. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 75/25, flow = 0.7 mL/min, retention time 8.3 min and 9.7 min (major).

(2*S*,3*S*)-(+)-3-(trifluoromethylthio)-2-(4-(trifluoromethyl)phenyl)-1,2,3,4-tetrahydroquinoline (7g): 52 mg, 92% yield, unknown compound, 99% ee, colorless oil, $[\alpha]_{D}^{20} = +45.71$ (*c* 1.0,



CHCl₃), $R_f = 0.90$ (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.64 (d, J = 8.2 Hz, 2H), 7.54 (t, J = 6.8 Hz, 2H), 7.16-6.98 (m, 2H), 6.79 (t, J = 7.4 Hz, 1H), 6.64 (d, J = 7.9 Hz, 1H), CF₃ 4.87 (s, 1H), 4.13 (s, 1H), 3.89 (d, J = 2.4 Hz, 1H), 3.53 (dd, J = 16.6,

4.4 Hz, 1H), 3.14 (dd, J = 16.7, 5.1 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) $\delta = 144.0, 143.3, 129.9, 129.8$ (q, ¹ $J_{FC} = 210$ Hz), 129.7, 129.0 (q, ¹ $J_{FC} = 307$ Hz), 128.1, 127.9, 125.6 (q, ² $J_{FC} = 4$ Hz), 119.4, 118.0, 115.0, 58.7, 45.6, 35.4; ¹⁹F NMR (376 MHz, CDCl₃) $\delta = -39.8, -62.6$. HRMS (ESI) m/z Calculated for C₁₇H₁₄F₆NS [M+H]⁺ 378.0746, found 378.0758. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 75/25, flow = 0.7 mL/min, retention time 14.5 min and 22.1 min (major).

(2*S*,3*S*)-(+)-2-(4-Fluorophenyl)-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7h): 46 mg, 94% yield, unknown compound, 99% ee, colorless oil, $[\alpha]^{20}{}_{\rm D}$ = +38.89 (*c* 0.72, CHCl₃), R_f =



0.85 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.45-7.32 (m, 2H), 7.06 (dd, J = 9.6, 7.8 Hz, 4H), 6.78 (dd, J = 7.5, 1.1 Hz, 1H), 6.61 (dd, J = 7.9, 0.7 Hz, 1H), 4.78 (s, 1H), 4.08 (s, 1H), 3.83 (dd, J = 7.3, 4.7 Hz, 1H), 3.51 (dd, J = 16.6, 4.4 Hz, 1H), 3.13 (dd, J = 16.6, 5.0 Hz,

1H); ¹³C NMR (100 MHz, CDCl₃) δ = 143.6, 131.5 (q, ¹*J*_{FC} = 294 Hz), 129.2, 129.1, 128.6 (q, ¹*J*_{FC} = 305 Hz), 128.0, 119.2, 118.0, 115.6, 115.4, 114.9, 58.4, 46.0, 35.4; ¹⁹F NMR (376 MHz, CDCl₃) δ = -39.7, -113.7. HRMS (ESI) *m*/*z* Calculated for C₁₆H₁₄F₄NS [M+H]⁺ 328.0778, found 328.0776. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 75/25, flow = 0.7 mL/min, retention time 13.8 min and 21.6 min (major).

(2S,3S)-(+)-2-(Naphthalen-2-yl)-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7i): 50 mg, 93% yield, unknown compound, 99% ee, colorless oil, $[\alpha]^{20}{}_{D} = +32.19$ (*c* 0.92, CHCl₃), R_f =

0.90 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.98-7.73 (m, 4H), 7.56-7.37 (m, 3H), 7.10 (dd, J = 16.0, 7.7 Hz, 2H), 6.79 (dd, J = 10.8, 4.0 Hz, 1H), 6.66 (d, J = 10.8, 4.0 Hz, 1H), 1H, 1H), 1H Hz, 1H), 1H, 1



7.9 Hz, 1H), 4.95 (s, 1H), 4.22 (s, 1H), 3.98 (dd, J = 7.2, 4.5 Hz, 1H), 3.56 (dd, J = 16.6, 4.3 Hz, 1H), 3.21 (dd, J = 16.7, 4.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) $\delta = 143.8$, 137.5, 134.1, 133.4, 130.3, 128.7 (q, ¹ $J_{FC} = 307$ Hz), 128.5, 128.4, 128.0, 127.9, 126.6, 126.5, 126.3,

125.3, 119.2, 118.2, 115.0, 59.1, 46.1, 35.7; ¹⁹F NMR (376 MHz, CDCl₃) δ = -39.5. HRMS (ESI) *m*/*z* Calculated for C₂₀H₁₇F₃NS [M+H]⁺ 360.1034, found 360.1031. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 75/25, flow = 0.7 mL/min, retention time 22.0 min and 33.1 min (major).

(2*S*,3*S*)-(+)-6-Methoxy-2-phenyl-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7j): 41 mg, 81% yield, unknown compound, 99% ee, white solid, mp 80-81 °C, $[\alpha]_{D}^{20} = +28.71$ (*c* 0.80,

MeO

CHCl₃), $R_f = 0.85$ (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.28 (dd, J = 11.9, 5.0 Hz, 4H), 7.17-7.07 (m, 2H), 6.85 (dd, J = 9.0, 2.8 Hz, 1H), 6.74 (d, J = 2.7 Hz, 1H), 6.61 (s, 1H), 4.32 (dd, J = 15.0, 6.0 Hz, 1H), 3.83 (s, 3H), 3.43 (dd, J = 16.8, 6.1 Hz, 1H), 3.08

(dd, J = 16.8, 9.5 Hz, 1H), 1.25 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) $\delta = 158.5$, 129.1, 129.0, 128.9, 128.6 (q, ¹ $J_{FC} = 305$ Hz), 128.3, 114.0, 114.0, 113.9, 113.9, 113.8, 66.6, 55.7, 43.3, 33.1; ¹⁹F NMR (376 MHz, CDCl₃) $\delta = -40.1$. HRMS (ESI) m/z Calculated for C₁₇H₁₇F₃NOS [M+H]⁺ 340.0983, found 340.0981. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 90/10, flow = 0.7 mL/min, retention time 8.5 min (major) and 9.4 min.

(3S)-(+)-3-(Trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7k): unknown compound, 33 mg, 94% yield, 67% ee, colorless oil, $[\alpha]^{20}_{D} = +11.23$ (*c* 0.60, CHCl₃), R_f = 0.65 (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.04 (ddd, *J* = 8.0, 1.3, 0.7 Hz, 1H), 6.97 (d, *J* = 7.5 Hz, 1H), 6.70 (td, *J* = 7.4, 1.0 Hz, 1H), 6.54 (d, *J* = 8.0 Hz, 1H), 3.93 (s, 1H), 3.87-3.76 (m, 1H), 3.66 (d, *J* = 11.7 Hz, 1H), 3.39 (ddd, *J* = 11.7, 7.2, 1.1 Hz, 1H), 3.26 (dd, *J* = 16.4, 4.9 Hz, 1H), 2.94 (dd, *J* = 16.4, 7.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ = 143.3, 130.0, 128.4 (q, ¹*J*_{FC} = 307 Hz), 127.9, 118.4, 118.2, 114.7, 47.6, 38.7, 34.2; ¹⁹F NMR (376 MHz, CDCl₃) δ = -39.4. HRMS (ESI) *m/z* Calculated for C₁₀H₁₁F₃NS [M+H]⁺ 234.0559, found 234.0566. HPLC: Chiracel OD-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 70/30, flow = 0.7 mL/min, retention time 9.1 min and 12.4 min (major).

2-Methyl-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (7l): unknown compound, 31 mg, 84% yield (inseparable mixture of two diastereoisomers), 72/28 d.r., 67% and 50% ee, colorless

SCF₃ oil, $R_f = 0.75$ (petroleum ether/dichloromethane 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.03 (dq, J = 14.1, 7.2 Hz, 2H), 6.71 (dtd, J = 8.3, 7.4, 1.0 Hz, 1H), 6.52 (d, J = 8.1 Hz, 1H), 4.00-3.80 (m, 1H), 3.80-3.70 (m, 1H), 3.67 (dd, J = 6.3, 3.4 Hz, 1H), 3.44-3.36 (m, 0.8H), 3.37-3.28 (m, 0.3H), 3.12 (dd, J = 16.7, 3.5 Hz, 0.72H), 3.05 (dd, J = 16.8, 6.9 Hz, 0.28H), 1.35 (d, J = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 143.7, 143.6, 130.3, 129.7, 129.1 (q, ¹ $J_{FC} = 306$ Hz), 129.0 (q, ¹ $J_{FC} = 306$ Hz), 127.9, 127.7, 118.8, 118.1, 117.9, 117.8, 114.6, 114.4, 51.7, 50.4, 45.5, 44.6, 36.2, 33.0, 21.6, 19.4; ¹⁹F NMR (376 MHz, CDCl₃) δ = -39.1, -39.3. HRMS (ESI) *m*/*z* Calculated for C₁₁H₁₃F₃NS [M+H]⁺ 248.0715, found 248.0718. HPLC: Chiracel OG-H column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 90/10,

flow = 0.7 mL/min, retention time (for 67% ee) 6.7 min and 7.5 min (major); retention time (for 50% ee) 8.8 min and 10.9 min (major).

4. The Determination of the Absolute Configuration of (+)-7b

In order to determine the absolute configuration of the product 2-*m*-tolyl-3-(trifluoromethyl-thio)-1,2,3,4-tetrahydroquinoline (+)-**7b** (50 mg, >99% ee) was dissolved in dichloromethane (0.1 mL) at 50 °C, then *n*-hexane (3.0 mL) was added and the solution was cooled down to room temperature. The single crystal of (+)-**7b** was grown from the solution, the absolute configuration of which was determined to be *cis*-(2*S*,3*S*) based on single-crystal X-ray diffraction analysis. CCDC 1439307 contains the supplementary crystallographic data for this paper. These can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac. uk.



Figure S1. The Absolute Configuration of 2-*m*-Tolyl-3-(trifluoromethylthio)-1,2,3,4-tetrahydroquinoline (+)-**7b**

5. References

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6. Copy of NMR and HPLC for Racemic and Chiral Compounds



1H NMR JZ-2-42 in CDCI3





13C NMR JZ-2-42 in CDCI3











S14





1H NMR JZ-2-45 in CDCI3













-41.93



1H NMR JZ-2-59 in CDCI3



-2.4595











-41.92







--42.03

19F NMR JZ-2-48B in CDCI3



1H NMR JZ-2-53B in CDCI3



-1.3812









1H NMR JZ-2-46B in CDCI3





S35




1H NMR JZ-2-53A in CDCI3





S38





1H NMR JZ-2-46A in CDCI3





S41





S43





-41.95











1H NMR JZ-2-61B in CDCI3







S51



1H NMR JZ-2-70B in CDCI3









1H NMR JZ-2-68C in CDCI3









1H NMR JZ-2-84 in CDCI3





S59



19F NMR JZ-2-84 in CDCI3







1H NMR JZ-2-70A in CDCI3





S62



19F NMR JZ-2-70A in CDCI3

Te OMe





1H NMR JZ-2-68A in CDCI3





S65







1H NMR JZ-2-72B in CDCI3











1H NMR JZ-2-68B in CDCI3






S72



1H NMR JZ-2-72A in CDCI3





----39.49

19F NMR JZ-2-72A in CDCI3







1H NMR JZ-2-101 in CDCI3







10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 f1 (ppm)



1H NMR JZ-3-5 in CDCI3











1H NMR JZ-2-73 In CDCI3







Data File C:\CHEM32\1\61B+-.D Sample Name: JZ-1-97(+-)

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| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | | |
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| | | (modified after loading) | | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | | |
| Last changed | : | 11/14/2015 9:29:08 AM | | | | | |
| | | (modified after loading) | | | | | |
| Sample Info | : | OD, H/i-PrOH = 75/25, 0.7 mL/min | n, 30 | οC, | 25 | 54 nm | |
| | | | | | | | |



| Sorted By | : | Signal | | | |
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| Multiplier: | | : | 1.0000 | | |
| Dilution: | | : | 1.0000 | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) |
| Use Multiplier 🛛 | Dilution | Factor wi | th ISTDs | | |
| | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm



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Instrument 1 11/14/2015 9:31:09 AM

Page 1 of 1

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(+/-)-7a

"SCF₃



| Acq. Operator | : | ZHOU | | | | |
|-----------------|---|----------------------------------|-----------|-----|-------|------|
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| Last changed | : | 11/14/2015 9:29:08 AM | | | | |
| | | (modified after loading) | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/m | nin, 30 c | С, | 254 n | ITO. |



| | | Area Per | cent Report | | | | | | | | |
|------------------|----------|----------|-------------|---------|---------------------|--|--|--|--|--|--|
| | | | | | | | | | | | |
| Sorted By | : | Sign | nal | | | | | | | | |
| Multiplier: | | : | 1.0000 | | | | | | | | |
| Dilution: | | : | 1.0000 | | | | | | | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) | | | | | | |
| Use Multiplier a | Dilution | Factor | with ISTDs | | | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm

| Peak | RetTime | Type | Width | A1 | ea | Hei | aht | Area |
|------|---------|------|--------|-------|-------|------|-------|---------|
| # | [min] | | [min] | mAU | *s | [mAU |] | % |
| 1 | 14.427 | BB | 0.3001 | 20. | 66220 | 1. | 07444 | 0.3784 |
| 2 | 21.470 | BB | 0.4697 | 5439. | 21191 | 179. | 65277 | 99.6216 |
| Tota | ls : | | | 5459. | 87411 | 180. | 72721 | |
| | | | | | | | | |

*** End of Report ***

Instrument 1 11/14/2015 9:29:34 AM

Page 1 of 1

"SCF₃

Data File C:\CHEM32\1\70B+-.D Sample Name: JZ-2-65B

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| | | (modified after loading) | | | | |
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| Last changed | : | 11/14/2015 9:48:53 AM | | | | |
| | | (modified after loading) | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/m | min, 30 | οC, | 254 1 | n n |
| | | | | | | |



Area Percent Report

| Sorted By | : | Siqnal | | | |
|----------------|------------|-----------|----------|---------|---------------------|
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| Dilution: | | : | 1.0000 | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) |
| Use Multiplier | & Dilution | Factor wi | th ISTDs | | |
| | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm





*** End of Report ***

Instrument 1 11/14/2015 9:49:08 AM

Page 1 of 1



| Acq. Operator : | ZHOU | |
|-------------------|---------------------------------|--------------------|
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| Injection Date : | 6/8/2015 12:31:59 PM | |
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| | (modified after loading) | |
| Analysis Method : | C:\CHEM32\1\METHODS\DEF_LC.M | |
| Last changed : | 11/14/2015 9:49:34 AM | |
| | (modified after loading) | |
| Sample Info : | OD-H, H/i-PrOH = 75/25, 0.7 mL/ | min, 30 oC, 254 nm |



| Area Percent Report | | | | | | | | | | | |
|---------------------|----------|------------|----------|---------|---------------------|--|--|--|--|--|--|
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| Sorted By | : | Signal | | | | | | | | | |
| Multiplier: | | : | 1.0000 | | | | | | | | |
| Dilution: | | : | 1.0000 | | | | | | | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) | | | | | | |
| Use Multiplier & | Dilution | Factor wit | th ISTDs | | | | | | | | |
| | | | | | | | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm

| Peak # | RetTime [min] | Type | Width [min] | Aı mAU | ea *s | Hei [mAU | .ght] | Area % | |
|-----------|------------------|------|----------------|-----------|----------|-------------|-----------|-----------|----------------|
| | | | | | | | | | |
| 1 | 11.262 | BB | 0.2377 | 58. | 84858 | 3. | 88809 | 2.1096 | |
| 2 | 14.110 | BB | 0.3010 | 2730. | 67871 | 141. | 38191 | 97.8904 | Ň Ň Í |
| Total | ls : | | | 2789. | 52729 | 145. | 27000 | | |
| | | | | | | | | | (+) -7b |
| | | | | | | | | | |

*** End of Report ***

Instrument 1 11/14/2015 9:49:44 AM

Page 1 of 1

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Me

Data File C:\CHEM32\1\68C+-.D Sample Name: JZ-2-63C(+-)

| Acq. Operator | : | ZHOU | | | | |
|-----------------|---|----------------------------------|---------|-----|--------|----------|
| Acq. Instrument | : | Instrument 1 | Locatio | n : | Vial 1 | L |
| Injection Date | : | 5/30/2015 12:23:57 AM | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | |
| Last changed | : | 5/29/2015 11:04:01 PM by ZHOU | | | | |
| | | (modified after loading) | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | |
| Last changed | : | 11/14/2015 9:44:16 AM | | | | |
| | | (modified after loading) | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/m | min, 30 | oC, | 254 ni | <u>n</u> |
| | | | | | | |



| Sorted By | : | Signal | | | |
|------------------|------------|-----------|----------|---------|---------------------|
| Multiplier: | | : | 1.0000 | | |
| Dilution: | | : | 1.0000 | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) |
| Use Multiplier (| a Dilution | Factor wi | th ISTDs | | |
| | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm



*** End of Report ***

Instrument 1 11/14/2015 9:44:24 AM

Page 1 of 1

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| Acq. Operator | : | ZHOU | | | | |
|-----------------|---|----------------------------------|-----------|-----|-------|---|
| Acq. Instrument | : | Instrument 1 | Location | 1: | Vial | 1 |
| Injection Date | : | 6/5/2015 12:21:55 PM | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | |
| Last changed | : | 6/5/2015 11:07:54 AM by ZHOU | | | | |
| | | (modified after loading) | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | |
| Last changed | : | 11/14/2015 9:45:09 AM | | | | |
| | | (modified after loading) | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/m | nin, 30 c | οC, | 254 r | m |



| Area Percent Report | | | | | | | | |
|---|----------------------------|--|--|--|--|--|--|--|
| | | | | | | | | |
| Sorted By Multiplier: Dilution: Sample Amount: Use Multiplier & | : Dilution | Signal : 1.0000 : 1.0000 : 1.00000 [ng/ul] (not used in calc.) Factor with ISTDs | | | | | | |
| Signal 1: VWD1 A, Peak RetTime Type # [min] | Waveleng Width [min] | Area Height AreaSCF3 | | | | | | |

| 1 2 | 11.358 19.485 | BB BB | 0.2442 0.4396 | 16.92536 4509.24023 | 1.07896 159.92871 | 0.3739 99.6261 | N ···· | |
|--------|------------------|----------|------------------|------------------------|----------------------|-------------------|-------------------|----------|
| Total | з: | | | 4526.16560 | 161.00767 | | (+)- 7c Me |) |
| | | | | | | | | |

*** End of Report ***

Instrument 1 11/14/2015 9:45:32 AM

Data File C:\CHEM32\1\84+-.D Sample Name: JZ-2-84(+-)

| lag Operator | | 70011 | | == | | |
|-----------------|---|---------------------------------|------------|----|-------|----|
| Acq. Operator | • | 21100 | | | | |
| Acq. Instrument | : | Instrument 1 | Location | : | Vial | 1 |
| Injection Date | : | 7/5/2015 6:10:17 AM | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | |
| Last changed | : | 7/5/2015 5:55:49 AM by ZHOU | | | | |
| | | (modified after loading) | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | |
| Last changed | : | 11/14/2015 10:15:03 AM | | | | |
| | | (modified after loading) | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 85/15, 0.7 mL/ | /min, 30 o | с, | 254 1 | nm |



Area Percent Report

| Sorted By | : | Signal | L | | |
|----------------|------------|-----------|----------|---------|---------------------|
| Multiplier: | | : | 1.0000 | | |
| Dilution: | | : | 1.0000 | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) |
| Use Multiplier | & Dilution | Factor wi | th ISTDs | | |
| | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm



*** End of Report ***

Instrument 1 11/14/2015 10:15:44 AM

Page 1 of 1

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| Acq. Operator | : | ZHOU | | | | |
|-----------------|---|----------------------------------|------------|----|-------|------|
| Acq. Instrument | : | Instrument 1 | Location | : | Vial | 1 |
| Injection Date | : | 7/5/2015 6:56:33 AM | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | |
| Last changed | : | 7/5/2015 5:55:49 AM by ZHOU | | | | |
| | | (modified after loading) | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | |
| Last changed | : | 11/14/2015 10:15:03 AM | | | | |
| | | (modified after loading) | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 85/15, 0.7 mL/m | nin, 30 o(| Ζ, | 254 n | III. |



| | Ar | sa Percent Repoi | rt | | |
|---|---------------------|---|-------------------------------|--------------------|------------------|
| | | | | | |
| Sorted By Multiplier: Dilution: Sample Amount: Use Multiplier « | : Dilution F | Signal : 1.0000 : 1.0000 : 1.00007 actor with ISTD: |) 0 0 [ng/ul] (r s | not used in calc.) | |
| Signal 1: VWD1 A, Peak RetTime Type | Wavelengt) Width | h=254 nm Area Hei | ght Area | | SCF ₃ |
| # Emainel | լաուլ ար | мо "з [шмо | ll | | |
| # [min] 1 37.927 BV 2 40.558 VB | 0.6723 0.9318 3 | 22.67941 4.1704 407.24023 56.8 | 46e-1 0.6612 88216 99.3388 | N N | |

*** End of Report ***

Instrument 1 11/14/2015 10:15:11 AM

Data File C:\CHEM32\1\70A+-.D Sample Name: JZ-2-52A(+-)

| Acq. Operator | : | ZHOU | | | | |
|-----------------|---|-----------------------------------|--------|------|-------|-----|
| Acq. Instrument | : | Instrument 1 L | ocatio | on : | Vial | 1 |
| Injection Date | : | 5/16/2015 12:08:00 PM | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | |
| Last changed | : | 5/16/2015 11:47:32 AM by ZHOU | | | | |
| | | (modified after loading) | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | |
| Last changed | : | 11/14/2015 9:47:08 AM | | | | |
| | | (modified after loading) | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/mi | n, 30 | οC, | 254 1 | nm. |



..... Area Percent Report

| Sorted By | : | Signal | | | |
|----------------|------------|-----------|----------|---------|---------------------|
| Multiplier: | | : | 1.0000 | | |
| Dilution: | | : | 1.0000 | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) |
| Use Multiplier | & Dilution | Factor wi | th ISTDs | | |
| | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm



*** End of Report ***

Instrument 1 11/14/2015 9:47:35 AM

Page 1 of 1

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Data File C:\CHEM32\1\70A.D Sample Name: JZ-2-70A

| Acq. Operator | : | ZHOU | | |
|-----------------|---|---------------------------------|---------------|--------|
| Acq. Instrument | : | Instrument 1 | Location : 1 | Vial l |
| Injection Date | : | 6/8/2015 11:44:42 AM | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | |
| Last changed | : | 6/8/2015 11:22:52 AM by ZHOU | | |
| | | (modified after loading) | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | |
| Last changed | : | 11/14/2015 9:48:09 AM | | |
| | | (modified after loading) | | |
| Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/ | min, 30 oC, 3 | 254 nm |



| Signal : : Factor with gth=254 nm Area | 1.0000 1.0000 .00000 [n .ISTDs Height. | ng/ul] (: |
|---|---|-----------------------|
| Signal : : 1 Factor with gth=254 nm Area | 1.0000 1.0000 .00000 [n .ISTDs | ng/ul] (: |
| gth=254 nm Area | Height. | |
| mAU *s | [mAU] | Area % |
| 36.44908 6482.07031 | 1.31395 207.12946 | 5 0.5592 5 99.4408 |
| 6518.51939 | 208.44341 | L |
| - | 36.44908 6482.07031 6518.51939 **** End of | |

Instrument 1 11/14/2015 9:48:17 AM

Data File C:\CHEM32\1\DATA\YZ007947.D Sample Name: JZ-2-63A(+-)

| Acq. Operator | : | ZHOU | | | | | |
|-----------------|---|----------------------------------|-----------|----|-------|---|--|
| Acq. Instrument | : | Instrument 1 | Location | 1: | Vial | 1 | |
| Injection Date | : | 5/29/2015 11:35:28 PM | | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | | |
| Last changed | : | 5/29/2015 11:04:01 PM by ZHOU | | | | | |
| | | (modified after loading) | | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF LC.M | | | | | |
| Last changed | : | 3/14/2016 10:02:21 PM | | | | | |
| | | (modified after loading) | | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/m | min, 30 d | c, | 254 n | m | |



-----Area Percent Report _____

| Sorted By | : | Signa | 11 | | |
|----------------|------------|----------|-----------|---------|---------------------|
| Multiplier: | | : | 1.0000 | | |
| Dilution: | | : | 1.0000 | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) |
| Use Multiplier | & Dilution | Factor w | ith ISTDs | | |
| | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm





*** End of Report ***

Instrument 1 3/14/2016 10:02:27 PM

Page 1 of 1

```
Data File C:\CHEM32\1\DATA\YZ008015.D
Sample Name: JZ-2-68A
    .....
    Acq. Operator : ZHOU
    Acq. Instrument : Instrument 1
Injection Date : 6/5/2015 11:37:24 AM
Acq. Method : C:\HPCHEM\1\METHODS\DEF LC.M
                                                       Location : Vial 1
    Last changed : 6/5/2015 11:07:54 AM by ZHOU
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed : 3/14/2016 10:00:14 PM
                   (modified after loading)
: OD-H, H/i-PrOH = 75/25, 0.7 mL/min, 30 oC, 254 nm
    Sample Info
```





*** End of Report ***

Instrument 1 3/14/2016 10:01:49 PM

Data File C:\CHEM32\1\72B+-.D Sample Name: JZ-2-65A(+-)

| Acq. Operator | : | ZHOU | | | | |
|-----------------|---|----------------------------------|----------|---|-------|---|
| Acq. Instrument | : | Instrument 1 | Location | : | Vial | 1 |
| Injection Date | : | 6/1/2015 11:37:20 PM | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | |
| Last changed | : | 6/1/2015 11:03:19 PM by ZHOU | | | | |
| | | (modified after loading) | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | |
| Last changed | : | 11/14/2015 9:59:03 AM | | | | |
| | | (modified after loading) | | | | |
| Sample Info | : | OD, H/i-PrOH = 75/25, 0.7 mL/min | , 30 oC, | 2 | 54 nm | |
| | | | | | | |



| Sorted By | | Signal | | | | |
|------------------|----------|-----------|----------|---------|--------------|--------|
| Multiplier: | | : | 1.0000 | | | |
| Dilution: | | : | 1.0000 | | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in | calc.) |
| Use Multiplier a | Dilution | Factor wi | th ISTDs | | | |
| | | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm



*** End of Report ***

Instrument 1 11/14/2015 9:59:14 AM

Page 1 of 1

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| Acq. Operator : | ZHOU | | | |
|-------------------|----------------------------------|-------------|--------|--|
| Acq. Instrument : | Instrument 1 | Location : | Vial l | |
| Injection Date : | 6/11/2015 11:30:56 PM | | | |
| Acq. Method : | C:\HPCHEM\1\METHODS\DEF LC.M | | | |
| Last changed : | 6/11/2015 11:14:23 PM by ZHOU | | | |
| | (modified after loading) | | | |
| Analysis Method : | C:\CHEM32\1\METHODS\DEF_LC.M | | | |
| Last changed : | 11/14/2015 9:59:49 AM | | | |
| | (modified after loading) | | | |
| Sample Info : | OD-H, H/i-PrOH = 75/25, 0.7 mL/m | ain, 30 oC, | 254 nm | |







| Peak RetTi # [mir | me Type] | Width [min] | Area mAU *s | Height [mAU] | Area % | |
|----------------------|----------------|----------------|------------------------|-------------------------|----------------|----------------|
| 1 14.4 | 66 VB 20 BB | 0.3482 | 16.63230 4585.54883 | 7.27673e-1 131.16261 | 0.3614 99.6386 | |
| Totals : | | | 4602.18113 | 131.89029 | | CF |
| | | | | | | (+) -7g |
| | | | *** End of | Report *** | | |

Instrument 1 11/14/2015 10:00:05 AM

Page 1 of 1

Data File C:\CHEM32\1\68B+-.D Sample Name: JZ-2-63B(+-)

| Acq. Operator | : | ZHOU | |
|-----------------|---|----------------------------------|--------------------|
| Acq. Instrument | : | Instrument 1 | Location : Vial l |
| Injection Date | : | 5/29/2015 11:53:13 PM | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | |
| Last changed | : | 5/29/2015 11:04:01 PM by ZHOU | |
| | | (modified after loading) | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | |
| Last changed | : | 11/14/2015 9:41:19 AM | |
| | | (modified after loading) | |
| Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/m | ain, 30 oC, 254 nm |
| | | | |



| Sorted By | : | Signa | 1 | | |
|------------------|------------|----------|-----------|---------|---------------------|
| Multiplier: | | : | 1.0000 | | |
| Dilution: | | : | 1.0000 | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) |
| Use Multiplier - | & Dilution | Factor w | ith ISTDs | | |
| | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm





*** End of Report ***

Instrument 1 11/14/2015 9:42:27 AM

Page 1 of 1



| | Acq. Operator | : | ZHOU | | | | |
|--|-----------------|---|----------------------------------|-----------|-----|-------|-----|
| | Acq. Instrument | : | Instrument 1 | Location | 1 : | Vial | 1 |
| | Injection Date | : | 6/5/2015 12:59:57 PM | | | | |
| | Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | |
| | Last changed | : | 6/5/2015 11:07:54 AM by ZHOU | | | | |
| | | | (modified after loading) | | | | |
| | Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | |
| | Last changed | : | 11/14/2015 9:41:19 AM | | | | |
| | | | (modified after loading) | | | | |
| | Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/m | ain, 30 c | ъC, | 254 r | TD. |



| Area Percent Report | | | | | | | | | |
|---|--|--|--|--|--|--|--|--|--|
| | | | | | | | | | |
| Sorted By : Signal Multiplier: : 1.0000 Dilution: : 1.0000 Sample Amount: : 1.00000 [ng/ul] (not used in calc.) Use Multiplier & Dilution Factor with ISTDs | | | | | | | | | |
| Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] * | | | | | | | | | |

| 1 | 13.840 | BB | 0.3183 | 24.75752 | 1.16264 | 0.3675 | |
|-------|--------|----|--------|------------|-----------|---------|--|
| 2 | 21.578 | BB | 0.4886 | 6711.12158 | 212.04829 | 99.6325 | |
| Total | ls : | | | 6735.87911 | 213.21094 | | |





Instrument 1 11/14/2015 9:41:32 AM

Data File C:\CHEM32\1\72A+-.D Sample Name: JZ-2-52B(+-)

| Acq. Operator | : | ZHOU | |
|-----------------|---|----------------------------------|--------------------|
| Acq. Instrument | : | Instrument 1 | Location : Vial l |
| Injection Date | : | 5/16/2015 11:54:22 PM | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | |
| Last changed | : | 5/16/2015 11:18:59 PM by ZHOU | |
| | | (modified after loading) | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | |
| Last changed | : | 11/14/2015 9:50:47 AM | |
| | | (modified after loading) | |
| Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/m | min, 30 oC, 254 nm |



..... Area Percent Report

| Sorted By | | Signal | L | | |
|----------------|------------|-----------|-----------|---------|---------------------|
| Multiplier: | | : | 1.0000 | | |
| Dilution: | | : | 1.0000 | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) |
| Use Multiplier | & Dilution | Factor wi | ith ISTDs | | |
| | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm

| Peak # | RetTime [min] | Type | Width [min] | A mAU | rea *s | Hei [mAU | qht] | Area % | |
|-----------|------------------|----------|----------------|--------------|------------------|-------------|----------------|--------------------|--|
| 1 2 | 22.302 33.085 | BB BB | 0.6095 | 2000 2000 | .68127 .75012 | 50. 37. | 63852 88595 | 49.9991 50.0009 | |
| Total | ls : | | | 4001 | .43140 | 88. | 52448 | | |
| | | | | | | | | | |

*** End of Report ***

Instrument 1 11/14/2015 9:50:58 AM

Page 1 of 1

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| Acq. Operator | : | ZHOU | | | | | |
|-----------------|---|----------------------------------|-----------|-----|-------|------|--|
| Acq. Instrument | : | Instrument 1 | Location | . : | Vial | 1 | |
| Injection Date | : | 6/11/2015 11:23:52 AM | | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | | |
| Last changed | : | 6/11/2015 10:48:11 AM by ZHOU | | | | | |
| | | (modified after loading) | | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | | |
| Last changed | : | 11/14/2015 9:50:47 AM | | | | | |
| | | (modified after loading) | | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 75/25, 0.7 mL/m | nin, 30 o | С, | 254 r | 110. | |



| Area Percent Report | | | | | | | | |
|---|-----------------|-------------------------------|--|----------|----------|-------------|----------|--|
| Sorted By Multiplier: Dilution: Sample Amount: Use Multiplier & | : Dilution F | Siqmal : : actor wit | 1.0000 1.0000 1.00000 h ISTDs | [ng/ul] | (not use | d in calc.) | | |
| Signal 1: VWD1 A, | Wavelengt | h=254 nm | | | | | <_ "SCF₃ | |
| Peak RetTime Type | Width | Area | Heigh | t Are | a | | 7 | |
| # [min] | [min] m | LAU ^S | . [10740 | 1 * | | | | |
| 1 21 985 BB | 0 5986 | 25 31996 | 5 77861 | e-1 0.6 | 427 | | | |
| 2 33.096 BB | 0.8532 3 | 914.20264 | 71.60 | 965 99.3 | 573 | - N H | | |

Totals : 3939.52259 72.18751



*** End of Report ***

Instrument 1 11/14/2015 9:51:38 AM

Data File C:\CHEM32\92+-.D Sample Name: JZ-2-92(+-)

| Acq. Operator | : | ZHOU | | | | | |
|-----------------|---|----------------------------------|---------|-----|-------|---|--|
| Acq. Instrument | : | Instrument 1 | Locatio | n : | Vial | 1 | |
| Injection Date | : | 9/23/2015 12:39:25 AM | | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | | |
| Last changed | : | 9/23/2015 12:31:52 AM by ZHOU | | | | | |
| | | (modified after loading) | | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | | |
| Last changed | : | 3/5/2016 4:24:24 PM | | | | | |
| | | (modified after loading) | | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 90/10, 0.7 mL/m | min, 30 | οC, | 254 i | m | |



Area Percent Report

| Sorted By : Multiplier: Dilution: Sample Amount: Use Multiplier & Dilution | Signal : : : Factor wit | 1.0000 1.0000 1.00000 th ISTDs | [ng/ul] | (not used | in calc.) |
|--|-------------------------------------|---|---------|-----------|-----------|
|--|-------------------------------------|---|---------|-----------|-----------|

Signal 1: VWD1 A, Wavelength=254 nm



*** End of Report ***

Instrument 1 3/5/2016 4:24:47 PM

Page 1 of 1

SCF₃

```
Data File C:\CHEM32\101.D
Sample Name: JZ-2-92
Acr. Operator : 2HOU
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 9/23/2015 1:09:46 AM
Acq. Method : C:\HPCHEM1\METHODSNDFF LC.M
Last changed : 9/23/2015 1:2:31:52 AM by ZHOU
(modified after Loading)
Analysis Method : C:\CHEM32\1\METHODSNDFF LC.M
Last changed : 3/5/2016 4:27:41 PM
(modified after Loading)
Sample Info : OP-H, H/1-PrOH = 90/10, 0.7 mL/min, 30 oC, 254 nm
```





Totals: 1130.79573 112.60890

*** End of Report ***

Instrument 1 3/5/2016 4:28:12 PM

Page 1 of 1

(+)**-7j**

Data File C:\CHEM32\1\3-5+-.D Sample Name: JZ-2-100(+-)

| Acq. Operator | : | ZHOU | | | | | |
|-----------------|---|----------------------------------|---------|-----|-------|---|--|
| Acq. Instrument | : | Instrument 1 | Locatio | n : | Vial | 1 | |
| Injection Date | : | 9/29/2015 11:22:31 PM | | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | | |
| Last changed | : | 9/29/2015 11:09:10 PM by ZHOU | | | | | |
| | | (modified after loading) | | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | | |
| Last changed | : | 11/14/2015 10:16:46 AM | | | | | |
| | | (modified after loading) | | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 70/30, 0.7 mL/m | min, 30 | oC, | 254 n | m | |
| | | | | | | | |



| g/ul] (not used in calc.) |
|---------------------------|
| 9 |

Signal 1: VWD1 A, Wavelength=254 nm





*** End of Report ***

Instrument 1 11/14/2015 10:16:52 AM

Page 1 of 1



| Acq. Operator | : | ZHOU | | | | | |
|-----------------|---|----------------------------------|-----------|----|-------|---|--|
| Acq. Instrument | : | Instrument 1 | Location | : | Vial | 1 | |
| Injection Date | ÷ | 10/13/2015 10:39:56 AM | | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | | |
| Last changed | ÷ | 10/13/2015 10:09:20 AM by ZHOU | | | | | |
| | | (modified after loading) | | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | | |
| Last changed | : | 11/14/2015 10:16:46 AM | | | | | |
| | | (modified after loading) | | | | | |
| Sample Info | : | OD-H, H/i-PrOH = 70/30, 0.7 mL/m | nin, 30 o | С, | 254 r | m | |



Area Percent Report Sorted By : Signal Multiplier: : 1.0000

| Dilution: | | 1.0000 | | |
|---------------------------|--------|------------|---------|---------------------|
| Sample Amount: | : | 1.00000 | [ng/ul] | (not used in calc.) |
| Use Multiplier & Dilution | Factor | with ISTDs | | |
| | | | | |

Signal 1: VWD1 A, Wavelength=254 nm

| Peak # | RetTime [min] | Type | Width [min] | Ar mAU | ea *s | Hei [mAU | .ght] | Area % | SCF ₃ |
|-----------|------------------|----------|------------------|---------------|----------------|-------------|----------------|--------------------|------------------|
| 1 2 | 9.075 12.419 | VB BB | 0.1608 0.2210 | 500. 2548. | 14172 30054 | 48. 179. | 61657 47720 | 16.4065 83.5935 | |
| Total | ls : | | | 3048. | 44226 | 228. | 09377 | | (+)- 7k |
| | | | | *** <u>F</u> | nd of | Repor | t *** | | |

Instrument 1 11/14/2015 10:17:12 AM

Data File C:\CHEM32\1\73+-.D Sample Name: JZ-2-65C(+-)

| Acq. Operator | : | ZHOU | | | | | | |
|-----------------|---|----------------------------------|----------------|------|--|--|--|--|
| Acq. Instrument | : | Instrument 1 | Location : Vi | al 1 | | | | |
| Injection Date | : | 6/3/2015 7:19:02 AM | | | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | | | |
| Last changed | : | 6/3/2015 7:04:58 AM by ZHOU | | | | | | |
| | | (modified after loading) | | | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | | | |
| Last changed | : | 11/14/2015 10:01:02 AM | | | | | | |
| | | (modified after loading) | | | | | | |
| Sample Info | : | OG-H, H/i-PrOH = 90/10, 0.7 mL/m | min, 30 oC, 25 | 4 nm | | | | |
| | | | | | | | | |



| Sorted By | : | Sign | al | | |
|----------------|------------|--------|------------|---------|---------------------|
| Multiplier: | | : | 1.0000 | | |
| Dilution: | | : | 1.0000 | | |
| Sample Amount: | | : | 1.00000 | [ng/ul] | (not used in calc.) |
| Use Multiplier | & Dilution | Factor | with ISTDs | | |
| | | | | | |

Signal 1: VWD1 A, Wavelength=254 nm



Instrument 1 11/14/2015 10:01:23 AM

Page 1 of 1

(+/-)-7I

_SCF₃

Ме



| Acq. Operator | : | ZHOU | | | | |
|-----------------|---|----------------------------------|-----------|----|-------|---|
| Acq. Instrument | : | Instrument 1 | Location | : | Vial | 1 |
| Injection Date | : | 6/15/2015 11:59:18 AM | | | | |
| Acq. Method | : | C:\HPCHEM\1\METHODS\DEF LC.M | | | | |
| Last changed | : | 6/15/2015 11:02:01 AM by ZHOU | | | | |
| | | (modified after loading) | | | | |
| Analysis Method | : | C:\CHEM32\1\METHODS\DEF_LC.M | | | | |
| Last changed | : | 11/14/2015 10:01:02 AM | | | | |
| | | (modified after loading) | | | | |
| Sample Info | : | OG, H/i-PrOH = 90/10, 0.7 mL/min | 1, 30 oC, | 2. | 54 nm | |





Instrument 1 11/14/2015 10:12:37 AM