Supporting Information:

Asymmetric Hydrogenolysis of Racemic Tertiary Alcohol,

3-Substituted 3-Hydroxyisoindolin-1-ones

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1. General and Materials

General: All reactions were carried out under an atmosphere of nitrogen using standard Schlenk techniques, unless otherwise noted. ¹H NMR and ¹³C NMR spectra were recorded at room temperature in CDCl₃ and DMSO 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). All reactions were monitored by TLC analysis.

Materials: Commercially available reagents were used throughout without further purification other than those detailed below. The solvents for asymmetric transfer hydrogenolysis reaction were purchased without further purification.

2. Typical Procedure for Synthesis of 3-Hydroxy-Substituted Isoindolin-1-ones 1



Typical procedure:^{1a-g} Solution of Grignard reagent (25 mmol) was added to, under N₂, phthalimide (6) (10 mmol) in THF (10 mL). After being stirred under the room temperature for 3 h, the reaction was quenched by a saturated aqueous solution of NH₄Cl (20 mL). The resulting mixture was extracted with CH₂Cl₂ (3x10 mL). The combined organic phases were washed with brine, then dried (Na₂SO₄), filtered and concentrated. A short silica gel column filtration of the crude mixture [ethyl acetate -petroleum ether = 1:2 as eluent] afforded **1**.

The ¹H NMR and ¹³C NMR of known imines $1a^{1a-b}$, 1b, ^{1a-b} $1c^{1b}$, $1d^{1b}$, $1h^{1g}$, $1i^{1b,1d,1f}$, $1j^{1f}$, $1k^{1f}$, $1l^{1f}$, $1o^{1a-c,1e}$, $1p^{1h}$, $2a^{1i}$ were consistent with the reported literature data.



3-Hydroxy-3-isopropylisoindolin-1-one (1e). Pale solid, mp = 189-191 °C, yield 85% (petroleum ester/ ethyl acetate = 2/1); ¹H NMR (400 MHz, DMSO) δ 8.68 (s, 1H), 7.56 (dt, J = 6.8, 3.5 Hz, 2H), 7.49 – 7.45 (m, 2H), 6.13 (s, 1H), 2.25 – 2.21 (m, 1H), 0.93 (d, J = 6.8 Hz, 3H), 0.60 (d, J = 6.8 Hz, 3H); ¹³C NMR (100 MHz, DMSO) δ 169.1, 149.7, 132.7, 132.6, 129.4, 122.9, 122.8, 90.4, 35.9, 17.7, 17.3;HRMS

Calculated For C₁₁H₁₃NO₂Na [M+Na]⁺ 214.0844, found: 214.0847.

3-Hexyl-3-hydroxyisoindolin-1-one (**1f**). Pale solid, mp = 102-104 °C, yield 66% (petroleum ester/ethyl acetate = 2/1); ¹H NMR (400 MHz, CDCl₃) δ 7.67 – 7.28 (m, 5H), 4.71 – 4.68 (m, 1H), 2.09 – 1.91 (m, 2H), 1.35 – 1.33 (m, 1H), 1.23 – 1.20 (m, 6H), 1.19 – 0.98 (m, 1H), 0.82 (t, *J* = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 170.2, 149.0, 132.7, 130.8, 129.3, 123.4, 122.0, 88.8, 38.6,

31.7, 29.4, 23.8, 22.7, 14.2; HRMS Calculated For $C_{14}H_{19}NO_2Na [M+H]^+$ 256.1313, found: 256.1315.

3-Cyclohexyl-3-hydroxyisoindolin-1-one (1g). Pale solid, mp = 200-202 °C, yield 92% (petroleum ester/ethyl acetate = 2/1); ¹H NMR (400 MHz, DMSO) δ 8.65 (s, 1H), 7.55 (d, *J* = 7.3 Hz, 2H), 7.47 – 7.40 (m, 2H), 6.10 (s, 1H), 1.97 – 1.85 (m, 2H), 1.69 (d, *J* = 12.6 Hz, 1H), 1.56 (d, *J* = 11.0 Hz, 2H), 1.20 – 0.86 (m, 7H); ¹³C NMR (100 MHz, DMSO) δ 168.9, 149.7, 132.7, 132.6, 129.4, 123.0, 122.9,

90.0, 45.8, 27.4, 26.9, 26.6, 26.4, 26.2; HRMS Calculated For $C_{14}H_{17}NO_2Na [M+Na]^+$ 254.1157, found:254.1162.

3-Butyl-3-Hydroxy-4-methylisoindolin-1-one (**1m**). Pale solid, mp = 60-61 °C, yield 40% (petroleum ester/ ethyl acetate = 2/1); ¹H NMR (400 MHz, CDCl₃) δ 7.40 (dd, J = 17.4, 9.9 Hz, 2H), 7.30 (d, J = 7.4 Hz, 1H), 7.06 (d, J = 7.5 Hz, 1H), 4.91 (s, 1H), 2.24 (s, 3H), 2.11 – 1.90 (m, 2H), 1.38 – 1.19 (m, 3H), 1.04 – 0.91 (m, 1H), 0.82 (t, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 171.0, 149.8, 138.0, 132.5, 131.4, 127.6, 119.6, 87.7, 38.4, 26.0, 22.8, 17.2, 14.0; HRMS Calculated For

138.0, 132.5, 131.4, 127.6, 119.6, 87.7, 38.4, 26.0, 22.8, 17.2, 14.0; HRMS Calculated For $C_{13}H_{17}NO_2Na \left[M+Na\right]^+ 242.1157$, found: 242.1156.

3-Butyl-3-Hydroxy-4-methylisoindolin-1-one (1n). Pale solid, mp = 132-134 °C, yield 44%



(petroleum ester/EtOAc = 2/1); ¹H NMR (400 MHz, CDCl₃) δ 7.32 – 7.22 (m, 4H), 4.58 (s, 1H), 2.48 (s, 3H), 2.12 (dd, *J* = 9.6, 6.3 Hz, 2H), 1.35 – 1.13 (m, 3H), 0.81 (t, *J* = 7.0 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 169.8, 145.6, 134.8, 133.8, 131.2, 129.5, 121.0, 89.6, 36.8, 26.0, 22.7, 17.8, 14.0; HRMS

Calculated For C₁₃H₁₇NO₂Na [M+Na]⁺ 242.1157, found: 242.1156.

3. Typical Procedure for Hydrogenolysis of 3-Hydroxy-Substituted Isoindolin-1-ones 1.



Typical procedure: In a dry Schlenk tube, 3-hydroxyisoindolin-1-one (1, 0.20 mmol), and phosphoric acid (*R*)-4 (6.0 mg, 0.01 mmol) and Hanztsch ester **5d** (61.8 mg, 0.20mmol) were dissolved in CH_2Cl_2 (12 mL) at 35°C under a nitrogen atmosphere. The solution was stirred until complete consumption of **1** (monitored by TLC). After removal of the solvent under reduced pressure, the residue was purified by flash chromatography (ethyl acetate/ petroleum ether, 2:1) to afford the desired product.

Racemates of **2** were prepared by the reduction of the corresponding 3-Hydroxy-Substituted Isoindolin-1-ones **1** using NaBH₃CN and concd. HCl (3 drops) in MeOH.^{1a}

(*R*)-3-Butylisoindolin-1-one (2a).² Pale solid, yield 62% (petroleum ester/ EtOAc = 2/1), 86% ee,



 $[\alpha]^{17}{}_{\rm D} = +30.7 \ (c \ 0.55, \text{CHCl}_3) \ [\text{lit.}^2: \ [\alpha]^{20}{}_{\rm D} = +53.0 \ (c \ 0.8, \text{MeOH}) \ \text{for } 92\% \ \text{ee}$ (*R*)]; ¹H NMR (400 MHz, CDCl₃) δ 8.04 (s, 1H), 7.86 (d, *J* = 7.5 Hz, 1H), 7.56 (td, *J* = 7.5, 1.1 Hz, 1H), 7.50 - 7.39 (m, 2H), 4.63 (dd, *J* = 7.4, 4.7 Hz, 1H), 1.96 (dd, *J* = 18.9, 5.4 Hz, 1H), 1.68 (dd, *J* = 15.8, 5.4 Hz, 1H), 1.53 -

1.26 (m, 4H), 0.90 (t, J = 7.1 Hz, 3H);.¹³C NMR (100 MHz, CDCl₃) δ 171.6, 148.0, 132.3, 131.9, 128.1, 123.9, 122.6, 57.3, 34.5, 27.8, 22.8, 14.1; HPLC (AD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 0.8 mL/min), t₁ = 8.9 min (maj), t₂ = 12.2 min.

(*R*)-3-Methylisoindolin-1-one (2b).^{2,4} Pale solid, yield 64% (petroleum ester/EtOAc = 2/1), 65% ee, $[\alpha]^{18}_{D}$ = +10.3 (*c* 0.67, CHCl₃) [lit.²: $[\alpha]^{20}_{D}$ = +39.1 (*c* 1.0, MeOH) for 97% ee (*R*)]; ¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, *J* = 7.5 Hz, 1H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.44 (dd, *J* = 15.2, 7.5 Hz, 2H), 4.70 (q, *J* = 6.7 Hz, 1H), 1.50 (d, *J* = 6.7 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 171.4, 149.2, 132.0, 128.2, 123.8 (d, *J* = 1.7

Hz), 122.4, 52.9, 20.4; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 95/5, detector: 254 nm, flow rate: 0.6 mL/min), $t_1 = 15.4$ min (maj), $t_2 = 16.8$ min.

(*R*)-3-Ethylisoindolin-1-one (2c).² Pale solid; yield 56% (petroleum ester/ EtOAc = 2/1), 86% ee,



 $[α]^{18}_{D}$ = +22.4 (c 0.63, CHCl₃) [lit.²: $[α]^{20}_{D}$ = +52.0 (*c* 0.6, MeOH) for 92% ee (*R*)]; ¹H NMR (400 MHz, CDCl₃) δ 7.83 (t, *J* = 13.9 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (dd, *J* = 14.7, 7.5 Hz, 2H), 4.61 (dd, *J* = 6.6, 5.0 Hz, 1H), 2.08 – 1.98 (m, 1H), 1.73 (dt, *J* = 14.2, 7.2 Hz, 1H), 0.97 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz,

CDCl₃) δ 171.6, 147.7, 132.4, 131.9, 128.2, 123.9, 122.6, 58.3, 27.5, 9.7; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 95/5, detector: 254 nm, flow rate: 0.7 mL/min), t₁ = 12.0 min (maj), t₂ = 13.4 min.

(*R*)-3-Propylisoindolin-1-one (2d).² Pale solid, yield 60% (petroleum ester/EtOAc = 2/1), 83% ee,



 $[\alpha]_{D}^{18} = +19.9 \ (c \ 0.67, \ CHCl_3) \ [lit.^2: \ [\alpha]_{D}^{20} = +57.2 \ (c \ 0.7, \ MeOH) \ for \ 97\% \ ee$ (*R*)]; ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 7.5 Hz, 1H), 7.66 (s, 1H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.45 (dd, *J* = 13.1, 7.4 Hz, 2H), 4.63 (dd, *J* = 7.6, 4.6 Hz, 1H), 1.99 - 1.87 (m, 1H), 1.70 - 1.59 (m, 1H), 1.58 - 1.33 (m, 2H), 0.97 (t, *J* = 1.51 + 1.51

7.3 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 171.8, 148.1, 132.4, 131.8, 128.1, 123.8, 122.6, 57.3, 36.9, 19.1, 14.2; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 95/5, detector: 254 nm, flow rate: 0.6 mL/min), t₁ = 12.7 (maj) min, t₂ = 15.5 min.

(*R*)-3-isopropylisoindolin-1-one (2e).⁵ Pale solid, yield 66% (petroleum ester/EtOAc = 2/1), 88%



ee, $[\alpha]^{18}{}_{D}$ = +22.6 (*c* 0.70, CHCl₃) [lit.⁵: $[\alpha]^{rt}{}_{D}$ = -40.0 (*c* 0.38, CH₂Cl₂) for >99% ee (*S*)]; ¹H NMR (400 MHz, CDCl₃) δ 7.86 (d, *J* = 7.4 Hz, 1H), 7.56 (t, *J* = 7.3 Hz, 2H), 7.45 (dd, *J* = 13.6, 7.3 Hz, 2H), 4.57 (d, *J* = 2.6 Hz, 1H), 2.26 (ddd, *J* = 13.6, 8.6, 5.3 Hz, 1H), 1.10 (d, *J* = 6.9 Hz, 3H), 0.73 (d, *J* = 6.8 Hz, 3H); ¹³C

NMR (100 MHz, CDCl₃) δ 172.0, 146.9, 132.9, 131.8, 128.1, 123.8, 122.8, 62.6, 31.9, 19.8, 16.1; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 95/5, detector: 254 nm, flow rate: 0.6 mL/min), t₁ = 11.7 (maj) min, t₂ = 13.2 min.

(*R*)-3-Hexylisoindolin-1-one (2f).³ Pale solid, yield 71% (petroleum ester/EtOAc = 2/1), 86% ee,



 $[\alpha]^{18}{}_{\rm D}$ = +26.9 (*c* 0.77, CHCl₃) [lit.²: $[\alpha]^{20}{}_{\rm D}$ = +53.0 (*c* 0.8, MeOH) for 92% ee (*R*)]; ¹H NMR (400 MHz, CDCl₃) δ 8.34 (s, 1H), 7.85 (d, *J* = 7.5 Hz, 1H), 7.55 (td, *J* = 7.4, 0.9 Hz, 1H), 7.53 – 7.43 (m, 2H), 4.63 (dd, *J* = 7.3, 4.7 Hz, 1H), 1.99 – 1.91 (m, 1H), 1.69 – 1.61 (m, 1H), 1.47 (dd, *J* =

16.3, 15.5 Hz, 1H), 1.47 – 1.26 (m, 7H), 0.85 (t, J = 6.9 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 171.7 , 148.1, 132.4, 131.8, 128.1, 123.8, 122.6, 57.4, 34.8, 31.8, 29.4, 25.6, 22.7, 14.2; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 95/5, detector: 254 nm, flow rate: 0.6 mL/min), t₁ = 11.2 min (maj), t₂ =13.8 min.

(*R*)-3-Cyclohexylisoindolin-1-one (2g).⁶ Pale soild, yield 54% (petroleum ester/EtOAc = 2/1), 76% ee, $[\alpha]^{16}_{D}$ = +19.5 (*c* 0.55, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, *J* = 7.4 Hz, 1H), 7.66 (s, 1H), 7.61 (td, *J* = 7.6, 1.1 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 4.59 (d, *J* = 3.2 Hz, 1H), 1.98 – 1.91 (m, 2H), 1.72 (d, *J* = 9.2 Hz, 2H), 1.38 – 1.10 (m, 7H); ¹³C NMR (100 MHz, CDCl₃) δ 171.6, 146.7, 132.7, 131.7, 128.1, 123.9, 122.9, 62.1, 42.0, 30.5, 26.6, 26.5, 26.3, 26.1; HPLC (OJ-H, elute: Hexanes/*i*-PrOH

= 90/10, detector: 254 nm, flow rate: 0.8 mL/min), $t_1 = 6.1 \text{ min (maj)}$, $t_2 = 7.6 \text{ min}$.

(*R*)-3-Phenethylisoindolin-1-one (2h).⁷ Pale solid, yield 38% (petroleum ester/EtOAc = 2/1), 78% ee, $[\alpha]^{17}_{D}$ = +20.3 (*c* 0.53, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.22 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 1H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (dd, *J* = 16.9, 7.6 Hz, 2H), 7.35 – 7.11 (m, 5H), 4.67 (dd, *J* = 7.9, 3.9 Hz, 1H), 2.77 (dt, *J* = 37.9, 19.8 Hz, 2H), 2.39 – 2.23 (m, 1H), 1.97 (dt, *J* = 18.5, 10.5 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 171.7, 147.7, 141.2, 132.3, 132.0,

128.8, 128.6, 128.3, 126.4, 124.0, 122.6, 56.8, 36.6, 32.1; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 95/5, detector: 254 nm, flow rate: 0.8 mL/min), $t_1 = 18.9$ min (maj), $t_2 = 23.1$ min.

(*R*)-3-benzylisoindolin-1-one (2i).⁵ Pale soild, yield 47% (petroleum ester/EtOAc = 2/1), 95% ee,



 $[\alpha]^{17}_{D}$ = +42.7 (*c* 0.43, CHCl₃) [lit.⁵: $[\alpha]^{rt}_{D}$ = -65.0 (*c* 0.53, CH₂Cl₂) for >99% ee (*S*)]; ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 7.5 Hz, 1H), 7.35 – 7.33 (m, 2H), 7.32 – 7.23 (m, 6H), 6.50 (s, 1H), 4.80 (dd, *J* = 9.2, 5.1 Hz, 1H), 3.24 (dd, *J* = 13.6, 5.1 Hz, 1H), 2.79 (dd, *J* = 13.6, 9.2 Hz, 1H); ¹³C NMR

(100 MHz, CDCl₃) δ 170.4, 147.0 137.2, 132.1, 132.0, 129.4, 129.1, 128.6, 127.4, 124.1, 122.8, 58.2, 41.6; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 80/20, detector: 254 nm, flow rate: 1.0 mL/min), t₁ = 6.6 min (maj), t₂ = 7.5 min

(*R*)-3-(4-methylbenzyl)isoindolin-1-one (2j).⁸ Pale solid, yield 57% (petroleum ester/EtOAc = 2/1), 94% ee, $[\alpha]^{16}_{D}$ = +69.8 (*c* 0.55, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, *J* = 7.5 Hz, 1H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 1H), 7.32 - 7.30 (m, 1H), 7.14 - 7.09 (m, 4H), 6.64 (s, 1H), 4.76 (dd, *J* = 8.5, 5.5 Hz, 1H), 3.19 - 3.17 (m, 1H), 2.79 - 2.76 (m, 1H), 2.33 (s, 3H); ¹³C

NMR (100 MHz, CDCl₃) δ 170.6, 147.1, 136.9, 134.0, 132.1, 131.9, 129.7, 129.3, 128.5, 124.0, 122.9, 58.3, 41.1, 21.3; HPLC (OD-H, elute: Hexanes/*i*-PrOH = 95/5, detector: 254 nm, flow rate: 1.0 mL/min), t₁ = 19.8 min, t₂ = 25.9 min (maj).

(+)-3-(3-methylbenzyl)isoindolin-1-one (2k). Pale yellow oil, yield 49 % (petroleum ester/EtOAc



= 2/1), 91% ee, $[\alpha]^{17}_{D}$ = +59.9 (*c* 0.57, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, *J* = 7.5 Hz, 1H), 7.55 (t, *J* = 7.3 Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 1H), 7.23 (dd, *J* = 13.6, 6.4 Hz, 1H), 7.06 (dd, *J* = 19.9, 9.5 Hz, 3H), 6.69 (s, 1H), 4.77 (dd, *J* = 9.1, 5.0 Hz, 1H), 3.22 – 3.18 (m, 1H), 2.72 (d, *J* = 8.9 Hz, 1H),

2.33 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 170.8, 147.2, 138.6, 137.1, 132.2, 131.8, 130.3, 128.8, 128.5, 128.0, 126.4, 124.0, 123.0, 58.3, 41.5, 21.6; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 0.8 mL/min), t₁ = 10.4 min (maj), t₂ = 12.8 min; HRMS Calculated For C₁₆H₁₅NONa [M+Na]⁺ 260.1051, found: 260.1058.

(+)-3-(4-fluorobenzyl)isoindolin-1-one (2l). Pale solid, mp = 114-116 °C, yield 50% (petroleum ester/EtOAc = 2/1), 93% ee, $[\alpha]^{18}{}_{D}$ = +93 (c 0.50, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.81 (d, J = 7.4 Hz, 1H), 7.55 – 7.46 (m, 3H), 7.28 – 7.21

(m, 2H), 7.00 – 6.90 (m, 3H), 4.81 (t, J = 6.7 Hz, 1H), 3.17 (dd, J = 13.6, 5.5 Hz, 1H), 2.91 (dd, J = 13.5, 8.1 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃)

 δ 170.92 (s), 163.0 (d, J = 246.5) , 146.7, 139.4 (d, J = 7.3 Hz), 132.2, 132.0, 130.4 (d, J = 8.3 Hz), 128.6, 125.2 (d, J = 2.8 Hz), 124.1, 122.9, 116.5 (d, J = 21Hz), 114.2 (d, J = 21 Hz), 57.9, 41.0 (d, J = 1.5 Hz); HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 0.8 mL/min), t₁ = 16.2 min (maj), t₂ = 19.9 min; HRMS Calculated For C₁₅H₁₂NONaF [M+Na]⁺ 264.0801, found: 264.0805.

(+)-3-butyl-4-methylisoindolin-1-one (2m). Pale solid, mp = 79-80 °C, yield 62% (petroleum



ester/EtOAc = 2/1), 82% ee, $[\alpha]^{18}{}_{D}$ = +15.3 (*c* 0.73, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.40 (t, *J* = 7.5 Hz, 1H), 7.20 (dd, *J* = 17.2, 7.6 Hz, 2H), 7.02 (s, 1H), 4.53 (dd, *J* = 7.9, 4.2 Hz, 1H), 2.72 (s, 3H), 1.93 (ddd, *J* = 10.5, 9.6, 4.4 Hz, 1H), 1.64 - 1.53 (m, 1H), 1.50 - 1.25 (m, 4H), 0.90 (t, *J* = 7.1 Hz, 3H);

¹³C NMR (100 MHz, CDCl₃) δ 172.1, 148.7, 138.1, 131.5, 130.1, 129.1, 119.9, 56.2, 34.8, 27.9,

22.8, 17.5, 14.1; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 95/5, detector: 254 nm, flow rate: 0.6 mL/min), $t_1 = 8.3 \text{ min (maj)}$, $t_2 = 9.2 \text{ min}$; HRMS Calculated For $C_{13}H_{17}NONa [M+Na]^+ 226.1208$, found: 226.1204.

(+)-3-butyl-7-methylisoindolin-1-one (2n). Pale solid, mp = 83-85 °C, yield 71% (petroleum ester/EtOAc = 2/1), 90% ee, $[\alpha]^{16}{}_{D}$ = +36.9 (c 0.68, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.93 (s, 1H), 7.69 (d, J = 7.2 Hz, 1H), 7.38 – 7.31 (m, 2H), 4.68 (dd, J = 7.6, 2.7 Hz, 1H), 2.40 (s, 3H), 2.17 – 2.03 (m, 1H), 1.67 (ddd, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 1.43 – 1.23 (m, 3H), 1.22 – 1.05 (m, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9, 6.1, 3.2 Hz, 1H), 0.86 (t, J = 10.9

7.0 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 171.7, 145.9, 133.4, 132.8, 132.5, 128.3, 121.4, 56.9, 32.4, 27.1, 22.7, 18.5, 14.0; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 1.0 mL/min), t₁ = 4.9 min (maj), t₂ = 9.2 min; HRMS Calculated For C₁₃H₁₇NONa [M+Na]⁺ 226.1208, found: 226.1203.

(*R*)-3-phenylisoindolin-1-one (2o).^{4a} Pale solid, yield 43% (petroleum ester/ EtOAc = 2/1), 61% ee, $[\alpha]^{17}{}_{\rm D}$ = -81.1 (*c* 0.34, CHCl₃) [lit^{4a}: $[\alpha]^{25}{}_{\rm D}$ = -193.3 (*c* 0.73, DMSO) for > 96% ee (*R*).]; ¹H NMR (400 MHz, CDCl₃) δ 7.88 (d, *J* = 7.1 Hz, 1H), 7.56 – 7.39 (m, 2H), 7.28 (ddd, *J* = 20.0, 13.8, 7.2 Hz, 6H), 6.93 (s, 1H), 5.63 (s, 1H); ¹³C NMR (100 MHz, DMSO) δ 170.4, 148.8, 140.2, 132.5, 131.9, 129.4, 128.8, 128.5, 127.2, 124.1, 123.5, 60.2; HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 0.8 mL/min), t₁ = 11.9 min (maj), t₂ = 17.8 min.

(E)-3-butylideneisoindolin-1-one (2a'). ¹¹ Pale solid, (petroleum ester/EtOAc = 3/1), ¹H NMR



(400 MHz, CDCl₃) δ 8.78 (s, 1H), 7.85 (d, J = 7.6 Hz, 1H), 7.66 (d, J = 7.7 Hz, 1H), 7.58 (d, J = 7.4 Hz, 1H), 7.47 (d, J = 7.5 Hz, 1H), 5.65 (t, J = 7.9 Hz, 1H), 2.36 (q, J = 7.5 Hz, 2H), 1.59 (dd, J = 14.6, 7.3 Hz, 2H), 1.02 (t, J = 7.4 Hz, 3H). The (**Z**)-3-butylideneisoindolin-1-one is trace ammount, we can not isolate the pure compound.

4. References

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Page 1

Single Mass Analysis Tolerance = 50.0 PPM / DBE: min = -20.0, max = 200.0 Selected filters: None

Monoisotopic Mass, Even Electron Ions 6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass) Elements Used: C: 0-100 H: 0-120 N: 1-1 O: 2-2 Na: 1-1 MC-4-38B 11051903 31 (0.805) AM (Cen,6, 80.00, Ar,5000.0,475.27,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (24:32) 100 1 214.0847

1: TOF MS ES+ 4.54e3









Page 1

2

Single Mass Analysis Tolerance = 50.0 PPM / DBE: min = -20.0, max = 200.0 Selected filters: None

Monoisotopic Mass, Even Electron Ions 6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass) Elements Used: C: 0-100 H: 0-120 N: 1-1 O: 2-2 Na: 1-1 MC-4-56 11051904 42 (1.057) AM (Cen,6, 80.00, Ar,5000.0,475.27,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (41:42) 400 . 489,2732 1: TOF MS ES+ 1.82e3 256.1315 % 471.2594 490.2728 338.1359 571.2700 722,4083 491.2733 572.2894 723.4200,804.4036 886.4255 968.4181 1037.5261 257.1364 420.1453 1201.6311 m/z 0 400 200 600 700 300 500 1000 800 900 1100 1200 -20.0 200.0 Minimum: 5.0 50.0 Maximum: Mass Calc. Mass mDa PPM DBE i-FIT Formula 256.1315 256.1313 0.8 5.5 11.3 C14 H19 N O2 Na 0.2



1f - HRMS



Page 1

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Single Mass Analysis Tolerance = 50.0 PPM / DBE: min = -20.0, max = 200.0 Selected filters: None

 Monoisotopic Mass, Even Electron Ions

 6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

 Elements Used:

 C: 0-100
 H: 0-120

 N: 1-1
 O: 2-2

 Na: 1-1

 MC-4-61B

 11051905 16 (0.402) AM (Cen,6, 80.00, Ar,5000.0,475.27,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (13:16)

 100

 254_1162
 1: TOF MS ES+ 2.93e3 100 485.2397 % 336.1443 567.2808 255.1187 418.1298 500.1259 568.2883 649.2787 716.3685 798.4281 925.0284 1008.0565 1108.8820 0 700 800 1000 1100 200 300 400 500 600 900 ~~ ^

Minimum: Maximum:		5.0	50.0	200.0							
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Form	ula				
254.1162	254.1157	0.5	2.0	6.5	1.0	C14	H17	N	02	Na	



1g - HRMS



S14

Page 1

Single Mass Analysis Tolerance = 50.0 PPM / DBE: min = -20.0, max = 200.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions 6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass) Elements Used: C: 0-100 H: 0-120 N: 1-1 O: 2-2 Na: 1-1 MC-4-62A 11051810 50 (1.288) AM (Cen,6, 80.00, Ar,5000.0,475.27,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (50) 680.3693

1: TOF MS ES+ 176

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100							6	80.3693		176
-	242 .1	165			461.2383					
%-	28	3.1340			-			696.3595		
-		301 31	l.1541 15.1812	443.227	76 477. 2	2393	662.3542	697.3671	38 4061	899.4963
0_1	250		350	400	450 50	0 550	600 650	700 75	0 800	m/z 850 900
Mini Maxi	mum: mum:			5.0	50.0	-20.0 200.0				
Mass		Calc.	Mass	mDa	PPM	DBE	i-FIT	Formula		
242.	1165	242.11	57	0.8	3.3	5.5	5546066.0	C13 H17 1	N O2 Na	

IH HO

1m - HRMS



Page 1

.4

Single Mass Analysis Tolerance = 50.0 PPM / DBE: min = -20.0, max = 200.0 Selected filters: None

Monoisotopic Mass, Even Electron Ions 6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass) Elements Used: C: 0-100 H: 0-120 N: 1-1 O: 2-2 Na: 1-1 MC-4-62B 11051809 24 (0.597) AM (Cen,6, 80.00, Ar,5000.0,475.27,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (22:24) 100-1 461.2431 1: TOF MS ES+ 4.15e3 100-680.3701 % 681.3735 462.2483

242.1156 258.0894		403.2331	477.2169 478.2240 _{559.2460}		696.3430 899.5 778.3724		99.5032	5162	1118.6311	1268.5508	1338.7582	
0	300	400	500	600	700	800	900	1000	1100	1200	1300	
Minimum Maximum			5.0	50.0	-20. 200.	0 0						
Mass	Calc	. Mass	mDa	PPM	DBE	i -1	TIT	Formul	La			
242.115	6 242.	1157	-0.1	-0.4	5.5	2.	3	C13 H	117 N O	2 Na		

ΙH ΗÓ

1n - HRMS









S21









Electronic Supplementary Material (ESI) for Chemical Communications This journal is The Royal Society of Chemistry 2012







Page 1⁻

Single Mass Analysis Tolerance = 50.0 PPM / DBE: min = -20.0, max = 200.0 Selected filters: None

Monoisotopic Mass, Even Electron lons 6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass) Elements Used: C: 0-100 H: 0-120 N: 1-1 O: 1-1 Na: 1-1 MC-4-818 11051907 17 (0.421) AM (Cen,6, 80.00, Ar,5000.0,475.27,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (16:19) 100 260,1058 497.2197 1: TOF MS ES+ 3.59e3 % 498.2200 261.1094 342.1105 579.2243 734.3359 202.1034 437.1903 896.3972 971.4505 m/z 661.2300 816.3645 0-600 750 450 650 200 250 350 550 700 950 300 400 500 800 850 900 -20.0 Minimum: Maximum: 50.0 5.0 Calc. Mass mDa PPM DBE i-FIT Formula Mass 3.3 C16 H15 N O Na 260.1058 260.1051 0.7 2.7 9.5



2k - HRMS



Single Mass Analysis Tolerance = 50.0 PPM / DBE: min = -20.0, max = 200.0 Selected filters: None Monoisotopic Mass, Even Electron lons 6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass) Elements Used: C: 0-100 H: 0-120 N: 1-1 O: 1-1 Na: 1-1 F: 1-1 MC-4-82A 11051906 55 (1.424) AM (Cen,6, 80.00, Ar,5000.0,475.27,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (55:73) 100-, 264.0805 1: TOF MS ES+ 8.10e3 505.1686 % 506.1711 346.0851 613.2032 614.2099 746.2595 854.2960 372.1218 1252.9576 1316.0220 m/z 962.3275 1089.0516 0 1100 1200 1300 400 900 1000 200 300 500 600 700 800 Minimum: Maximum: -20.0 5.0 50.0 200.0 i-FIT DBE Formula PPM Mass Calc. Mass mDa 264.0801 0.4 1.5 9.5 3.8 C15 H12 N O Na F 264.0805

Page 1

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2I - HRMS

S31



Page 1

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Single Mass Analysis Tolerance = 50.0 PPM / DBE: min = -20.0, max = 200.0 Selected filters: None

Monoisotopic Mass, Even Electron lons 6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass) Elements Used: C: 0-100 H: 0-120 N: 1-1 O: 1-1 Na: 1-1
 MC-4-65A

 11051812 31 (0.813) AM (Cen,6, 80.00, Ar,5000.0,475.28,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (28:33)

 429,2551

 632,3860
 1: TOF MS ES+ 1.29e3 633.3907 % 226.1204 430.2603 634.3945 267.1475 301.1443 431.2641 504.2752 714.3915 917.5280 224,1052 835.5356 999.5479 m/z Ш 0 200 300 400 500 600 700 800 900 1000 Minimum: Maximum: -20.0 200.0 5.0 50.0





2m - HRMS



Elemental	Composition	Report								Page 1
Single Ma Tolerance Selected fi	ss Analysis = 50.0 PPM / iters: None	DBE: m	iin = -20).0, max =	200.0					7
Monoisotopic 6 formula(e) Elements Us C: 0-100 H MC-4-658 11051811 41 (1 100-1	: Mass, Even Elect evaluated with 1 re ed: : 0-120 N: 1-1 .035) AM (Cen,6, 80.0 429	ron lons esults withi O: 1-1 N 0, Ar,5000.0, .2551	n limits (a a: 1-1 475.28,0.70 632	all results (up),LS 10); Sm (S 3883	o to 1000) fo G, 2x3.00); Sb	r eac (1,40	:h mass) .00); Cm (38:	:41)	1: TC	F MS ES+ 975
			032							
226.	1203									
%-		430.2550		633.3904						
146.0236	301.1416 390.1304	504.268	31	634.3972 _{714.}	3967835.5246	917.53	393 999.5201	1120.6539	1202.7	7083
0-4	بېرون <u>لومېرون اومېرون د مېرون</u> 300 400	500 500	600	700	800	900	1000	1100	1200	1300
Minimum: Maximum:		5.0	50.0	-20.0 200.0						
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	E	Formula			
226.1203	226.1208	-0.5	-2.2	5.5	2773040.	0 0	C13 H17	N O Na		

ΝH H 2n -HRMS

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Electronic Supplementary Material (ESI) for Chemical Communications This journal is The Royal Society of Chemistry 2012





Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000139.D Sample Name: MC-4-51B

 Acq. Operator
 :

 Acq. Instrument : Instrument 1
 Location : Vial 1

 Injection Date
 : 2/21/2011 2:23:15 PM

 Acq. Method
 : D:\D7-3-78B.M

 Last changed
 : 1/6/2011 10:33:39 AM

 Analysis Method
 : C:\CHEM32\1\METHODS\SW.M

 Last changed
 : 5/6/2011 11:33:07 AM

 Sample Info
 : AD-H, H/1-PtOH =90/10,0.8 mL/min, 30 oC, 254nm



Area Percent Report

Sorteo	i By	:	Signal			
Multip	plier:		:	1.0000		
Diluti	ion:		:	1.0000		
Use Mu	ultiplier «	Dilution	Factor wit	h ISTDs		
						6
Signal	L 1: VWD1 A,	Wavelenç	gth=254 nm			Ľ
Peak H	RetTime Type	Width	Area	Height	Area	
#	[min]	[min]	mAU *s	[mAU]	*	
-						
1	8.809 BB	0.1910	3432.94385	271.89182	49.8527	
2	12.107 BB	0.2587	3453.23682	203.91774	50.1473	
Totals	s :		6886.18066	475.80956		

*** End of Report ***

Data File C:\CHEM32\1\DATA\ZHOU-10\YZNOO1165.D Sample Name: MC-4-29A



Area Percent Report



Sorted By	:	Signal			
Multiplier:		:	1.0000		0
Dilution:		:	1.0000		Ų,
Use Multiplier & I	llution	Factor with	n ISTDs		$\wedge \mathcal{A}$
Signal 1: VWD1 A,	Wavelen	gth=254 nm			
					4 11/
Peak RetTime Type	Width	Area	Height	Area	н
# [min]	[min]	mAU *s	[mAU]	*	
					(+) - 2a
1 8.940 BB	0.1916	360.95905	28.47867	92.9576	()
2 12.216 MM R	0.2671	27.34583	1.70631	7.0424	
Totals :		388.30488	30.18498		

*** End of Report ***

Instrument 1 5/7/2011 2:04:20 PM

Page 1 of 1

(+/-) - 2a

Instrument 1 12/21/2010 4:40:26 PM

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000145.D Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000157.D Sample Name: MC-4-51A(+-) Sample Name: MC-4-54A _____ _____ Acq. Operator : Acq. Operator : Acq. Instrument : Instrument 1 Location : Vial 1 Acq. Instrument : Instrument 1 Location : Vial 1 Injection Date : 2/22/2011 3:15:25 PM Injection Date : 2/24/2011 3:29:44 PM Acq. Method : D:\DY-3-78B.M Acq. Method : D:\DY-3-78B.M Last changed : 2/22/2011 3:01:39 PM Last changed : 2/24/2011 3:28:01 PM (modified after loading) (modified after loading) Analysis Method : C:\CHEM32\1\METHODS\SW.M Analysis Method : C:\CHEM32\1\METHODS\SW.M Last changed : 5/7/2011 2:08:57 PM Last changed : 5/7/2011 2:35:25 PM (modified after loading) (modified after loading) Sample Info : OJ-H, H/i-PrOH =95/5, 0.6 mL/min, 30 oC, 254nm Sample Info : 0J-H, H/i-PrOH =95/5, 0.6 mL/min, 30 oC, 254 nm VWD1A, Wavelength=254 nm (ZHOU-11\YZN000145.D) VWD1A, Wavelength=254 nm (ZHOU-11\\YZN000157.D) mAU mAU 100 -40 80 30 -60 20 40 10 20 _____ Area Percent Report Area Percent Report Sorted By Sorted By : Signal : Signal Multiplier: : 1.0000 Multiplier: : 1.0000 Dilution: : 1.0000 Dilution: : 1.0000 Use Multiplier & Dilution Factor with ISTDs Use Multiplier & Dilution Factor with ISTDs NΗ Signal 1: VWD1 A, Wavelength=254 nm Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area Height Area Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] ÷ # [min] [min] mAU *s [mAU] * (+/-) - 2b (+) - 2b 1 15.513 VV 0.4428 3340.56421 117.86552 49.9245 1 15.410 VV 0.4223 1368.89856 50.30894 82.2748 2 16.881 VB 0.4873 3350.66650 107.11401 50.0755 2 16.830 VB 0.4521 294.91418 10.07535 17.7252 Totals : 6691.23071 224.97952 1663.81274 60.38430 Totals : _____ *** End of Report *** *** End of Report *** Page 1 of 1 Page 1 of 1 Instrument 1 5/7/2011 2:09:35 PM Instrument 1 5/7/2011 2:35:28 PM

Data File C:\CHEM32\1\DATA\2H0U-11\YZN000147.D
Sample Name: MC-4-52B(+-)

 Acq. Operator
 :

 Acq. Instrument :
 Instrument :
 Location : Vial 1

 Injection Date :
 :
 2/22/2011 4:18:51 PM

 Acq. Method :
 D:\DY-3-78B.M
 Location :
 Vial 1

 Last changed :
 :
 2/22/2011 4:18:52 PM
 (modified after loading)

 Analysis Method :
 :
 C:\CHEM3211/METHODS\SW.M

 Last changed :
 :
 5/6/2011 1:33:07 AM

 Sample Info
 :
 0/3-H, H/i-PrOH =95/5, 0.7 mL/min, 30 oC, 254nm



Area Percent Report



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*** End of Report ***

Instrument 1 5/7/2011 2:42:32 PM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000173.D Sample Name: MC-4-57A

Acq. Operator : Acq. Instrument : Instrument 1 Location : Vial 1 Injection Date : 3/1/2011 3:36:20 PM Acq. Method : D:\DY-3-78B.M Last changed : 3/1/2011 3:22:58 PM (modified after loading) Analysis Method : C:\CHEM32\1\METHODS\SU.M Last changed : 5/7/2011 2:45:11 PM (modified after loading)

Sample Info : 0J-H, H/i-PrOH =95/5, 0.7 mL/min, 30 oC, 254 nm



Area Percent Report



Instrument 1 5/7/2011 2:45:17 PM

Data File C:\CHEM32\1\DATA\ZHOU-11\YZNO00158.D Sample Name: MC-4-53A

Acq. Operator	:	
Acq. Instrument	:	Instrument l Location : Vial 1
Injection Date	:	2/24/2011 4:03:20 PM
Acq. Method	:	D:\DY-3-78B.M
Last changed	:	2/24/2011 3:59:24 PM
		(modified after loading)
Analysis Method	:	C:\CHEM32\1\METHODS\SW.M
Last changed	:	5/7/2011 2:47:00 PM
		(modified after loading)
Sample Info	:	OJ-H, H/i-PrOH =95/5, 0.6 mL/min, 30 oC, 254 nm



Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000174.D Sample Name: MC-4-57B



Area Percent Report

Sorted By	:	Signal			Q						
Multiplier:		:	1.0000		$\sim \parallel$						
Dilution:		:	1.0000								
Use Multiplier & I	lution	Factor with	n ISTDs		NH						
Signal 1: VWD1 A,	Waveleng	th=254 nm			Ĥ Ű						
Peak RetTime Type	Width	Area	Height	Area	(+) - 2d						
# [min]	[min]	mAU *s	[mAU]	*	(+) - 2u						
1 12.716 BB	0.3403	813.72662	36.89865	91.7049							
2 15.470 BB	0.3975	73.60495	2.83517	8.2951							
Totals :		887.33157	39.73382								
	*** End of Report ***										

Instrument 1 3/3/2011 7:33:26 PM



Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000181.D Sample Name: MC-4-57C

 Acq. Operator
 :

 Acq. Instrument :
 Instrument 1
 Location : Vial 1

 Injection Date :
 3/3/2011 4:42:44 PM
 Acq. Nethod :
 C:\CHEM32\1\METHODS\SW.M

 Last changed :
 3/3/2011 4:30:20 PM
 (modified after loading)

 Analysis Method :
 C:\CHEM32\1\METHODS\SW.M

 Last changed :
 12/21/2010 4:56:35 PM

 Sample Info :
 0/3-H, H/1-POH =95/5, 0.6 mL/min, 30 oC, 254 nm



Area Percent Report





Instrument 1 5/7/2011 2:50:17 PM

Page 1 of 1

Instrument 1 3/4/2011 2:21:48 PM

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000179.D Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000183.D Sample Name: MC-4-58B+-Sample Name: MC-4-58A _____ Acq. Operator : Acq. Operator : Acq. Instrument : Instrument 1 Acq. Instrument : Instrument 1 Location : Vial 1 Location : Vial 1 Injection Date : 3/3/2011 3:18:08 PM Injection Date : 3/3/2011 7:28:53 PM Acq. Method : D:\DY-3-78B.M Acq. Method : C:\CHEM32\1\METHODS\SW.M Last changed : 3/3/2011 3:15:10 PM Last changed : 3/3/2011 7:24:01 PM (modified after loading) (modified after loading) Analysis Method : C:\CHEM32\1\METHODS\SW.M Analysis Method : C:\CHEM32\1\METHODS\SW.M Last changed : 3/3/2011 4:36:20 PM Last changed : 5/7/2011 2:50:11 PM (modified after loading) (modified after loading) Sample Info : 0J-H, H/i-PrOH =95/5, 0.6 mL/min, 30 oC, 254 nm Sample Info : OJ-H, H/i-PrOH =95/5, 0.6 mL/min, 30 oC, 254 nm VWD1A, Wavelength=254 nm (ZHOU-11\\YZN000179.D) VWD1A, Wavelength=254 nm (ZHOU-11\\YZN000183.D) mAU mAU 80 80 -70 -70 -60 -60 -<u>50</u> -50 -40 -4n -30 -30 -20 20.4 10 Area Percent Report Area Percent Report Sorted By : Signal Sorted By Signal : \cap Multiplier: : 1.0000 Multiplier: : 1.0000 Dilution: : 1.0000 Dilution: . 1.0000 Use Multiplier & Dilution Factor with ISTDs Use Multiplier & Dilution Factor with ISTDs NH ٧Н Signal 1: VWD1 A, Wavelength=254 nm Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area Height Peak RetTime Type Width Area Height Area Area (+/-) - 2f # [min] [min] mAU *s [mAU] * # [min] [min] mAU *s [mAU] ÷ (+) - 2f 1 11.073 VB 0.3149 1748.23645 85.77927 50.0444 1 11.246 VB 0.3184 1829.72522 88.47999 92.7892 2 13.523 VB 0.3812 1745.13184 70.69690 49.9556 2 13.803 VB 0.3793 142.19164 5.79792 7.2108 3493.36829 156.47617 1971.91685 94.27791 Totals : Totals : _____ _____ *** End of Report *** *** End of Report *** Page 1 of 1 Page 1 of 1 Instrument 1 3/3/2011 4:36:24 PM Instrument 1 5/7/2011 2:55:56 PM

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000236.D Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000302.D Sample Name: MC-4-62B(+-) Sample Name: MC-4-73A _____ Acq. Operator : Acq. Operator : Acq. Instrument : Instrument 1 Location : Vial 1 Acq. Instrument : Instrument 1 Location : Vial 1 Injection Date : 3/24/2011 2:16:31 PM Injection Date : 4/1/2011 4:50:34 PM Acq. Method : C:\CHEM32\1\METHODS\SW.M Acq. Method : C:\CHEM32\1\METHODS\SW.M Last changed : 3/24/2011 2:14:36 PM Last changed : 4/1/2011 4:49:44 PM (modified after loading) (modified after loading) Analysis Method : C:\CHEM32\1\METHODS\SW.M Analysis Method : C:\CHEM32\1\METHODS\SU.M Last changed : 5/7/2011 3:07:59 PM Last changed : 3/31/2011 4:32:02 PM (modified after loading) Sample Info : 0J-H, H/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 254nm Sample Info : 0J-H, H/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 254 nm VWD1A, Wavelength=254 nm (ZHOU-11\\YZN000302.D) VWD1A, Wavelength=254 nm (ZHOU-11\YZN000236.D) mAU mAU 160 50 · 140 40-120 -100 -30 -80 · 20 -60 10 40 20Area Percent Report Area Percent Report Sorted By : Signal \cap Sorted By Multiplier: : 1.0000 : Signal Multiplier: : 1.0000 Dilution: : 1.0000 Dilution: : 1.0000 Use Multiplier & Dilution Factor with ISTDs Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area Height Area Peak RetTime Type Width Area Height # [min] [min] mAU *s [mAU] Area ÷ # [min] [min] mAU *s [mAU] ÷ (+/-) - 2g 1 6.050 VB 0.1787 733.46393 61.40973 87.7768 (+) - 2g 1 6.159 VB 0.1793 2024.83948 174.15790 49.3632 2 7.637 BB 0.2607 102.13696 6.01494 12.2232 2 7.685 BB 0.2771 2077.08472 116.11961 50.6368 Totals : 835.60088 67.42467 4101.92419 290.27750 Totals : _____ *** End of Report *** *** End of Report *** Page 1 of 1 Page 1 of 1 Instrument 1 5/7/2011 3:08:03 PM Instrument 1 4/2/2011 10:03:33 AM

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000185.D Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000203.D Sample Name: MC-4-55+-Sample Name: MC-4-59A _____ _____ Acq. Operator : Acq. Operator : Acq. Instrument : Instrument 1 Location : Vial 1 Acq. Instrument : Instrument 1 Location : Vial 1 Injection Date : 3/3/2011 8:24:14 PM Injection Date : 3/5/2011 3:20:25 PM Acq. Method : C:\CHEM32\1\METHODS\SW.M Acq. Method : C:\CHEM32\1\METHODS\SW.M Last changed : 3/3/2011 8:09:43 PM Last changed : 3/5/2011 3:08:42 PM (modified after loading) (modified after loading) Analysis Method : C:\CHEM32\1\METHODS\SW.M Analysis Method : C:\CHEM32\1\METHODS\SW.M Last changed : 5/7/2011 3:15:54 PM Last changed : 5/7/2011 3:12:17 PM (modified after loading) (modified after loading) Sample Info : OJ-H, H/i-PrOH =90/10, 0.8 mL/min, 30 oC, 254 nm Sample Info : 0J-H, H/i-PrOH =90/10, 0.8mL/min, 30 oC, 254 nm VWD1A, Wavelength=254 nm (ZHOU-11\\YZN000185.D) W/D1A, W avelength=254 nm (ZHOU-11\\\/ZN000203.D) mAU mAU 25 25 20 -20 15 15 10 10 22 _____ Area Percent Report Area Percent Report Sorted By : Signal Sorted By Signal . Multiplier: : 1.0000 Multiplier: : 1.0000 Dilution: : 1.0000 Dilution: : 1.0000 Use Multiplier & Dilution Factor with ISTDs Use Multiplier & Dilution Factor with ISTDs NΗ Signal 1: VWD1 A, Wavelength=254 nm Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area Height Peak RetTime Type Width Area Area Height Area # [min] [min] mAU *s [mAU] ÷ # [min] [min] mAU *s [mAU] ÷ (+) - 2h (+/-) 1 19.247 BB 0.6737 1273.27771 29.17627 50.1470 1 18.969 BB 0.6437 1208.26550 28.98074 88.8729 2 23.081 BB 0.8311 1265.81335 23.43452 49.8530 2 23.104 BB 0.7506 151.27776 2.94579 11.1271 2539.09106 52.61079 1359.54326 31.92653 Totals : Totals : _____ _____ *** End of Report *** *** End of Report *** Page 1 of 1 Page 1 of 1 Instrument 1 5/7/2011 3:15:57 PM Instrument 1 5/7/2011 3:12:36 PM





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Instrument 1 5/7/2011 3:54:00 PM ZX





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Instrument 1 5/7/2011 3:58:43 PM ZX

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000238.D Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000287.D Sample Name: MC-4-65A(+-) Sample Name: MC-4-72A _____ Acq. Operator : Acq. Operator : Acq. Instrument : Instrument 1 Location : Vial 1 Acq. Instrument : Instrument 1 Location : Vial 1 Injection Date : 3/24/2011 2:54:55 PM Injection Date : 3/31/2011 3:41:17 PM Acq. Method : C:\CHEM32\1\METHODS\SW.M Acq. Method : C:\CHEM32\1\METHODS\SW.M Last changed : 3/24/2011 2:45:16 PM Last changed : 3/31/2011 3:29:23 PM (modified after loading) (modified after loading) Analysis Method : C:\CHEM32\1\METHODS\SW.M Analysis Method : C:\CHEM32\1\METHODS\SW.M Last changed : 5/7/2011 3:24:32 PM Last changed : 3/29/2011 3:55:42 PM (modified after loading) Sample Info : 0J-H, H/i-PrOH = 95/5, 0.6 mL/min, 30 oC, 254nm Sample Info : 0J-H, H/i-PrOH = 95/5, 0.6 mL/min, 30 oC, 254 nm VWD1A, W avelength=254 nm (ZHOU-11\YZN000287.D) VWD1A, Wavelength=254 nm (ZHOU-11\YZN000238.D) mAU mAU 60 600 50 500 · 40 400 -30 -300 -20 200 -100 _____ Area Percent Report Area Percent Report -----Sorted By : Signal O \cap Sorted By Multiplier: : 1.0000 : Signal Multiplier: : 1.0000 Dilution: : 1.0000 Dilution: : 1.0000 Use Multiplier & Dilution Factor with ISTDs NH Use Multiplier & Dilution Factor with ISTDs NH Signal 1: VWD1 A, Wavelength=254 nm Signal 1: VWD1 A, Wavelength=254 nm (+/-) - 2m Peak RetTime Type Width Area Height Area (+) - 2m Peak RetTime Type Width Area Height # [min] [min] mAU *s [mAU] Area * # [min] [min] mAU *s [mAU] * 1 8.258 VV 0.2400 1043.08484 65.91434 91.1652 1 8.300 VV 0.2462 1.13361e4 714.80652 49.7936 2 9.248 VV 0.2688 101.08498 5.71957 8.8348 2 9.225 VB 0.2925 1.14301e4 606.97162 50.2064 1144.16982 71.63391 Totals : 2.27662e4 1321.77814 Totals : -----*** End of Report *** *** End of Report *** Page 1 of 1 Page 1 of 1 Instrument 1 5/7/2011 3:24:37 PM Instrument 1 3/31/2011 3:53:54 PM



Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000433.D Sample Name: MC-3-62(+-)

Acq. Operator	:							
Acq. Instrument	:	Instrument 1 Location : Vial 1						
Injection Date	:	5/4/2011 11:08:34 AM						
Acq. Method	:	C:\CHEM32\1\METHODS\SW.M						
Last changed	:	5/4/2011 11:01:26 AM						
		(modified after loading)						
Analysis Method	:	C:\CHEM32\1\METHODS\SW.M						
Last changed	:	5/7/2011 3:46:05 PM						
		(modified after loading)						
Sample Info	:	0J-H, H/i-PrOH =90/10, 0.8 mL/min, 30 oC, 254 nm						



Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000432.D Sample Name: MC-4-89B

Acq. Operator	:					
Acq. Instrument	:	Instrument 1	Location	a :	Vial	1
Injection Date	:	5/4/2011 10:38:42 AM				
Acq. Method	:	C:\CHEM32\1\METHODS\SW.M				
Last changed	:	5/4/2011 10:34:43 AM				
		(modified after loading)				
Analysis Method	:	C:\CHEM32\1\METHODS\SW.M				
Last changed	:	5/7/2011 3:46:05 PM				
		(modified after loading)				
Sample Info	:	0J-H, H/i-PrOH =90/10, 0.8 mL/	min, 30 (ъC,	254 1	nm



**** End of Report ***

Instrument 1 5/7/2011 3:46:50 PM

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Instrument 1 5/7/2011 3:46:17 PM