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 $^1$H NMR and $^{13}$C NMR of known imines 1a, 1b, 1c, 1d, 1h, 1i, 1j, 1k, 1l, 1m, 1n 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); $^1$H NMR (400 MHz, DMSO) $\delta$ 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); $^{13}$C NMR (100 MHz, DMSO) $\delta$ 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); $^1$H NMR (400 MHz, CDCl3) $\delta$ 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); $^{13}$C NMR (100 MHz, CDCl3) $\delta$ 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 C14H19NO2Na [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); $^1$H NMR (400 MHz, DMSO) $\delta$ 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); $^{13}$C NMR (100 MHz, DMSO) $\delta$ 168.9, 149.0, 132.7, 130.8, 129.3, 123.4, 122.0, 88.8, 31.7, 29.4, 23.8, 22.7, 14.2; HRMS Calculated For C14H17NO2Na [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); $^1$H NMR (400 MHz, CDCl3) $\delta$ 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); $^{13}$C NMR (100 MHz, CDCl3) $\delta$ 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 C13H17NO2Na [M+Na]+ 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); $^1$H NMR (400 MHz, CDCl3) $\delta$ 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); $^{13}$C NMR (100 MHz, CDCl3) $\delta$ 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.

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 Hantzsch ester 5d (61.8 mg, 0.20 mmol) were dissolved in CH2Cl2 (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, [α]17D = +30.7 (c 0.55, CHCl3) [lit.2: [α]20D = +53.0 (c 0.8, MeOH) for 92% 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, [α]18D = +10.3 (c 0.67, CHCl3) [lit.2: [α]17D = +39.1 (c 1.0, MeOH) for 97% ee (R)]; 1H NMR (400 MHz, CDCl3) δ 7.83 (d, J = 7.5 Hz, 1H), 7.44 (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 171.6, 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), t1 = 15.4 min (maj), t2 = 16.8 min.

(R)-3-Ethylisoindolin-1-one (2c).2 Pale solid; yield 56% (petroleum ester/EtOAc = 2/1), 86% ee, [α]16D = +22.4 (c 0.63, CHCl3) [lit.2: [α]17D = +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,
[(R)-3-Isopropylisoindolin-1-one (2e).](5) Pale solid, yield 66% (petroleum ester/EtOAc = 2/1), 88% ee, \([\alpha]_{18}^D = +22.6 (c 0.70, CHCl_3) \) [lit.\(^5\): \([\alpha]_{20}^D = -40.0 (c 0.38, CH_2Cl_2)\) for >99% ee (S)]; \(^1\)H NMR (400 MHz, CDCl_3) \(\delta 7.86 (d, J = 7.4 Hz, 1H), 7.56 (t, J = 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, CDCl_3) \(\delta 172.0, 146.9, 132.9, 131.8, 128.1, 123.8, 122.8, 62.6, 31.9, 19.8, 16.1;\) HPLC (OJ-H, elute: Hexanes/i-PrOH = 95/5, detector: 254 nm, flow rate: 0.6 mL/min), \(t_1 = 11.7\) (maj) min, \(t_2 = 13.2\) min.

**(R)-3-Hexylisoindolin-1-one (2f).** (3) Pale solid, yield 71% (petroleum ester/EtOAc = 2/1), 86% ee, \([\alpha]_{18}^D = +26.9 (c 0.77, CHCl_3) \) [lit.\(^2\): \([\alpha]_{20}^D = +53.0 (c 0.8, MeOH)\) for 92% ee (R)]; \(^1\)H NMR (400 MHz, CDCl_3) \(\delta 8.34 (s, 1H), 7.85 (d, J = 7.5 Hz, 1H), 7.55 – 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, CDCl_3) \(\delta 171.7, 148.1, 132.4, 131.8, 128.1, 123.8, 122.6, 57.4, 34.8, 31.8, 29.4, 25.6, 22.7, 14.2;\) HPLC (OJ-H, elute: Hexanes/i-PrOH = 95/5, detector: 254 nm, flow rate: 0.6 mL/min), \(t_1 = 11.2\) min (maj), \(t_2 = 13.8\) min.

**(R)-3-Cyclohexylisoindolin-1-one (2g).** (6) Pale solid, yield 54% (petroleum ester/EtOAc = 2/1), 76 % ee, \([\alpha]_{16}^D = +19.5 (c 0.55, CHCl_3); \) \(^1\)H NMR (400 MHz, CDCl_3) \(\delta 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, CDCl_3) \(\delta 171.6, 146.7, 132.7, 131.7, 128.1, 123.9, 122.9, 62.1, 42.0, 30.5, 26.6, 26.5, 26.3, 26.1;\) HPLC (OJ-H, elute: Hexanes/i-PrOH = 90/10, detector: 254 nm, flow rate: 0.8 mL/min), \(t_1 = 6.1\) min (maj), \(t_2 = 7.6\) min.

**[(R)-3-Phenethylisoindolin-1-one (2h).]** (7) Pale solid, yield 38% (petroleum ester/EtOAc = 2/1), 78% ee, \([\alpha]_{17}^D = +20.3 (c 0.53, CHCl_3); \) \(^1\)H NMR (400 MHz, CDCl_3) \(\delta 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 = 3.7, 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, CDCl_3) \(\delta 171.7, 147.7, 141.2, 132.3, 132.0, 128.8, 128.6, 128.3, 126.4, 124.0, 122.6, 56.8, 36.6, 32.1;\) 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). Pale solid, yield 47% (petroleum ester/EtOAc = 2/1), 95% ee, \( [\alpha]_{D}^{17} = +42.7 \text{ (c 0.43, CHCl}_3 \text{, lit.}^{5} [\alpha]_{D}^{17} = -65.0 \text{ (c 0.53, CH}_2\text{Cl}_2 \text{, for >99% ee (S))}; {^1}H \text{ NMR (400 MHz, CDCl}_3 \text{) \( \delta \) 7.85 (d, \( J = 7.5 \text{ Hz, 1H}\), 7.35 – 7.33 (m, 2H), 7.32 – 7.23 (m, 6H), 6.50 (s, 1H), 4.80 (dd, \( J = 9.2, 5.1 \text{ Hz, 1H}\), 3.24 (dd, \( J = 13.6, 5.1 \text{ Hz, 1H}\), 2.79 (dd, \( J = 13.6, 9.2 \text{ Hz, 1H}\)); \(^{13}C \text{ NMR (100 MHz, CDCl}_3 \text{) \( \delta \) 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), \( t_1 = 6.6 \text{ min (maj)}\), \( t_2 = 7.5 \text{ min (maj)}\).}

(R)-3-(4-methylbenzyl)isoindolin-1-one (2j). Pale solid, yield 57% (petroleum ester/EtOAc = 2/1), 94% ee, \( [\alpha]_{D}^{17} = +69.8 \text{ (c 0.55, CHCl}_3 \text{); {^1}H \text{ NMR (400 MHz, CDCl}_3 \text{) \( \delta \) 7.83 (d, \( J = 7.5 \text{ Hz, 1H}\), 7.74 (t, \( J = 7.4 \text{ Hz, 1H}\), 7.46 (t, \( J = 7.4 \text{ Hz, 1H}\), 7.32 – 7.30 (m, 1H), 7.14 – 7.09 (m, 4H), 6.64 (s, 1H), 4.76 (dd, \( J = 8.5, 5.5 \text{ Hz, 1H}\), 3.19 – 3.17 (m, 1H), 2.79– 2.76 (m, 1H), 2.33 (s, 3H); \(^{13}C \text{ NMR (100 MHz, CDCl}_3 \text{) \( \delta \) 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 (OJ-H, elute: Hexanes/i-PrOH = 95/5, detector: 254 nm, flow rate: 1.0 mL/min), \( t_1 = 19.8 \text{ min, } t_2 = 25.9 \text{ min (maj).}

(+)-3-(3-methylbenzyl)isoindolin-1-one (2k). Pale yellow oil, yield 49 % (petroleum ester/EtOAc = 2/1), 91% ee, \( [\alpha]_{D}^{17} = +59.9 \text{ (c 0.57, CHCl}_3 \text{); {^1}H \text{ NMR (400 MHz, CDCl}_3 \text{) \( \delta \) 7.84 (d, \( J = 7.5 \text{ Hz, 1H}\), 7.55 (t, \( J = 7.3 \text{ Hz, 1H}\), 7.47 (t, \( J = 7.4 \text{ Hz, 1H}\), 7.23 (dd, \( J = 13.6, 6.4 \text{ Hz, 1H}\), 7.06 (dd, \( J = 19.9, 9.5 \text{ Hz, 3H}\), 6.69 (s, 1H), 4.77 (dd, \( J = 9.1, 5.0 \text{ Hz, 1H}\), 3.22 – 3.18 (m, 1H), 2.72 (d, \( J = 8.9 \text{ Hz, 1H}\), 2.33 (s, 3H); \(^{13}C \text{ NMR (100 MHz, CDCl}_3 \text{) \( \delta \) 170.8, 147.2, 138.6, 137.1, 132.2, 131.8, 130.3, 128.8, 128.5, 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), \( t_1 = 10.4 \text{ min (maj)}\), \( t_2 = 12.8 \text{ min; HRMS Calculated For C}_{16}H_{15}NONa [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]_{D}^{17} = +93 \text{ (c 0.50, CHCl}_3 \text{); {^1}H \text{ NMR (400 MHz, CDCl}_3 \text{) \( \delta \) 7.81 (d, \( J = 7.4 \text{ Hz, 1H}\), 7.55 – 7.46 (m, 3H), 7.28 – 7.21 (m, 2H), 7.00 – 6.90 (m, 3H), 4.81 (t, \( J = 6.7 \text{ Hz, 1H}\), 3.17 (dd, \( J = 13.6, 5.5 \text{ Hz, 1H}\), 2.91 (dd, \( J = 13.5, 8.1 \text{ Hz, 1H}\); \(^{13}C \text{ NMR (100 MHz, CDCl}_3 \text{) \( \delta \) 170.92 (s), 163.0 (d, \( J = 246.5 \text{ Hz, 1H}\), 146.7, 139.4 (d, \( J = 7.3 \text{ Hz, 1H}\), 132.2, 132.0, 130.4 (d, \( J = 8.3 \text{ Hz, 128.6, 125.2 (d, \( J = 2.8 \text{ Hz, 1H}\), 124.1, 122.9, 116.5 (d, \( J = 21 \text{ Hz, 1H}\), 114.2 (d, \( J = 21 \text{ 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), } t_1 = 16.2 \text{ min (maj), } t_2 = 19.9 \text{ min}; \text{ HRMS Calculated For C}_{16}H_{12}ONaF [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]_{D}^{17} = +15.3 \text{ (c 0.73, CHCl}_3 \text{); {^1}H \text{ NMR (400 MHz, CDCl}_3 \text{) \( \delta \) 7.40 (t, \( J = 7.5 \text{ Hz, 1H}\), 7.20 (dd, \( J = 17.2, 7.6 \text{ Hz, 2H}\), 7.02 (s, 1H), 4.53 (dd, \( J = 7.9, 4.2 \text{ Hz, 1H}\), 2.72 (s, 3H), 1.93 (ddd, \( J = 10.5, 9.6, 4.4 \text{ Hz, 1H}\), 1.64 – 1.53 (m, 1H), 1.50 – 1.25 (m, 4H), 0.90 (t, \( J = 7.1 \text{ Hz, 3H}; \(^{13}C \text{ NMR (100 MHz, CDCl}_3 \text{) \( \delta \) 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), t₁ = 8.3 min (maj), t₂ = 9.2 min; HRMS Calculated For C₁₃H₁₇NONa [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, [α]D²⁰ = +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 (dd, 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 = 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). Pale solid, yield 43% (petroleum ester/ EtOAc = 2/1), 61% ee, [α]D²⁰ = -81.1 (c 0.34, CHCl₃) [lit⁴⁺: [α]²⁵® = -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'). 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 amount, we can not isolate the pure compound.
4. References


Elemental Composition Report

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-4.98B
1105/1903.31 (8.085) AM (Clm6, 80.00, Ar:5000, 0.475, 0.70, 0.71 10), Sb (BG, 2x3.00), Sb (1,40.00); Cm (24:32)
1: TOF MS ES+
4.54e3

Minimum:

Maximum:

Mass  Calc. Mass  pPM  DBE  i-FFT  Formula
214.0847  214.0844  0.3  1.4  5.5  7.0  C11 H13 N O2 Na

1e - HRMS
1H NMR (CDCl₃, 400 MHz)

13C NMR (CDCl₃, 100 MHz)
Elemental Composition Report

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: 0-150  H: 0-120  N: 1-1  O: 2-2  Na: 1-1

<table>
<thead>
<tr>
<th>Mass</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>256.1315</td>
<td>256.1313</td>
<td>0.2</td>
<td>0.8</td>
<td>5.5</td>
<td>11.3</td>
<td>C14 H19 N O2 Na</td>
</tr>
</tbody>
</table>

![Structural diagram](image_url)

1f - HRMS
$^{1}H$ NMR (DMSO, 400 MHz)

$^{13}C$ NMR (DMSO, 100 MHz)

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Elemental Composition Report

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

Mass  Calc. Mass  mDa  PPM  DBE  i-FT  Formula
254.1192 254.1157 0.5 2.0 6.5 1.0  C14 H17 N O2 Na

1g - HRMS
Elemental Composition Report

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: 4-42A
11501810 80 (1.280) AM (Cen, 80, 80.0, Av: 5000.0, 475.275.70, LS 10); Sm (SO, 2x3.00); Sb (1.40.00); Cm (50)

176

Minimum: 5.0 50.0 200.0
Maximum: 5.0 50.0 200.0

Mass  Calc. Mass  m/z  PPM  DBE  i-FIT  Formula
242.1365 242.1157 0.8  3.3  5.5  5546066.0 C13 N17 N O2 Na

1m - HRMS
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1H NMR MC-4-61A in CDCl3

13C NMR MC-4-61A in CDCl3
Elemental Composition Report

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:4.628
1101689 54 (0.597) AM (Cen, 50 0.00, Ar:50000,475,27,0,70,LS 10); Sm (SS, 2x0.00); Sb (1,40,00 ; Cem (22,24)

1: TOF MS(ES) 4.19e3

Minimum:
Mass Calc. Mass mDa PPM DBE i-FIT7 Formula
242.1156 242.1157 -0.1 -0.4 5.5 2.3 C13 H17 N O2 Na

1n - HRMS
1H NMR (CDCl₃, 400 MHz)

13C NMR (CDCl₃, 100 MHz)
2c - $^1$H NMR (CDCl$_3$, 400 MHz)
$^{13}$C NMR (CDCl$_3$, 100 MHz)
CQA-2011-03
1H NMR MC-4,53A in CDCl3.
NHz本性混合

2d - 1H NMR (CDCl3, 400 MHz)
13C NMR (CDCl3, 100 MHz)
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2e - $^1$H NMR (CDCl$_3$, 400 MHz)
$^{13}$C NMR (CDCl$_3$, 100 MHz)

S22
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\[ 2f \cdot ^1H\text{NMR (CDCl}_3, 400 MHz) \]

\[ ^{13}C\text{NMR (CDCl}_3, 100 MHz) \]
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CQA-2011-33
H-NMR NOE-4.62 in CDCl3

2g - 1H NMR (CDCl3, 400 MHz)
13C NMR (CDCl3, 100 MHz)

CQA-2011-33
13C NMR NOE-4.62 in CDCl3

S24
Elemental Composition Report

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

Monoisotopic Mass, Even Electron Ions
6 formula(s) 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-4106
11651607 17 (0.421) AM (C9H9, 80.00, As,5000.0,475.27,0.70,1.5 10); Sm (SG, 2×3.00; Sb (1.40 00); Ce (16:19) 1: TOF MS ESI
3.59%3

Minimum:
-20.0
Maximum:
5.0 50.0 200.0

Mass  Calc. Mass  mDa  PPM  DBE  i-VIT  Formula
260.1058 260.1051 0.7 2.7 9.5 3.3 C16 H15 N O Na

2k - HRMS
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2fl- $^1$H NMR (CDCl$_3$, 400 MHz)

$^{13}$C NMR (CDCl$_3$, 100 MHz)

S30
Elemental Composition Report

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:

MC-4.02A
110519065 (1.424) AM (Cen.8, 80.00, Ar:5000.0,4.75,270.70,LS 10); Sm (SO, 2×0.00); Sb (1,40.00); Clm (55.73)
264.0805

Minimum: 5.0  Maximum: 50.0  -20.0  200.0
Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
264.0805  264.0801  0.4  1.5  9.5  3.0  C15 H12 N O Na F

2l - HRMS
Elemental Composition Report

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

Monosotopic 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: 1-1  Na: 1-1

MC/4.65A
1: TOF MS ES+

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<th>Mass</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>FPPM</th>
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<td>5.5</td>
<td>3.6</td>
<td>C13 H17 N O Me</td>
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</tbody>
</table>

2m - HRMS
Electronic Supplementary Material (ESI) for Chemical Communications

1H NMR (CDCl₃, 400 MHz)

13C NMR (CDCl₃, 100 MHz)

S34
Elemental Composition Report

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

Monoisotopic Mass, Even Electron Ions
6 forms 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-4-626

110/1911 41 (1.035) AM (C35.6, 80.00, At:5000:0.475:28.70:LS:10): Sm (SG, 2x3.00); Sb (1.40.00); Cm (38.41)
1: TOF MS ESI+

0 100  226.1203  632.3883
%  301.1416  354.3972  1.5393
146.0236  400.2500  999.5201
0 200  300  400  500  600  700  800  900  1000  1100  1200  1300

Minimum:  5.0  50.0  -20.0
Maximum:  260.0

Mass  Calc. Mass | mDa  FPM  DBE  1-FIT  Formula
226.1203  226.1208  -0.3  -2.2  5.5  2773040.0  C13 H17 N O Na

2n -HRMS
2o - $^1$H NMR (CDCl$_3$, 400 MHz)
$^{13}$C NMR (DMSO, 100 MHz)
S37

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SW-10-08-10
1H NMR of 2a' in CDCl₃

2a' - ¹H NMR (CDCl₃, 400 MHz)
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**Sample Name:** MC-4-01A

**Injection Date:** 4/15/2011 4:50:03 PM

**Sample Size:** 0.5 µl, 0.05 µl/min, 20 sec, 254 nm

---

**Sample Name:** MC-4-02B

**Injection Date:** 4/15/2011 4:50:03 PM

**Sample Size:** 0.5 µl, 0.05 µl/min, 20 sec, 254 nm

---

**Signal:**

**Wavelength:** 254 nm

**Peak RetTime Tube Width Area Height Area**

<table>
<thead>
<tr>
<th></th>
<th>Tube Width</th>
<th>Area</th>
<th>Height</th>
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</table>

**Totals:** 566.0144 181.01615

**Results obtained with enhanced integrator!***

---

**Instrument:** 5/7/2011 3:57:22 PM EZ

---

**Instrument:** 5/7/2011 3:58:43 PM EZ

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