Supporting Information

N-Heterocyclic Carbene Catalyzed Chemo- and Enantioselective Cross-Benzoin Reaction of Aldehydes with Isatins

Jianfeng Xu,* Jingyi Peng, Chonglong He, and Hongjun Ren* 

*Department of Chemistry, Zhejiang Sci-Tech University, Hangzhou 310018, P. R. China

e-mail: jfxu@zstu.edu.cn, renhj@zstu.edu.cn

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General Information

Commercially available materials purchased from Energy-Chemical were used as received. Proton nuclear magnetic resonance ($^1$H NMR) spectra were recorded on a Zhongke-Niujin AS 400 (400 MHz) spectrometer. Chemical shifts were recorded in parts per million (ppm, δ) relative to tetramethylsilane (δ 0.00) or chloroform (δ = 7.26, singlet). $^1$H NMR splitting patterns are designated as singlet (s), doublet (d), triplet (t), quartet (q), dd (doublet of doublets); m (multiplets), and etc. All first-order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted are designated as multiplet (m) or broad (br). Carbon nuclear magnetic resonance ($^{13}$C NMR) spectra were recorded on a Zhongke-Niujin AS 400 (100 MHz) spectrometer. High resolution mass spectral analysis (HRMS) was performed on a Waters Xevo G2-S QTof mass spectrometer. The determination of ee was performed via chiral HPLC analysis using Waters Breeze 2 HPLC system. X-ray crystallography analysis was performed on a Bruker X8 APEX X-ray diffractionmeter. Optical rotations were measured using a 2 mL cell with a 1 dm path length on a Rudolph Autopol IV automatic polarimeter and are reported as follows: [α]D ($c$ in g per 100 mL solvent). Infrared spectral analysis (IR) was performed on a Nicolet iS10 infrared spectrometer. Analytical thin-layer chromatography (TLC) was carried out on with GF 254 silica gel coated plates. Flash column chromatography was carried out using 200–300 mesh silica gel. Melting points were uncorrected. N-protected isatins,[1] NHC pre-catalyst[2] D and E were synthesized according to reported method.
General procedure for the cross-benzoin reaction:

To a dry 10 mL Schlenk tube equipped with a magnetic stir bar, were added NHC pre-catalyst D or E (0.02 mmol), isatin 2 (0.1 mmol), 4 Å MS (100 mg, powder), and NaHCO₃ (0.1 mmol). The tube was sealed with a septum, evacuated and refilled with nitrogen (3 cycles). DCE (1 mL) and aldehyde 1 (0.15 mmol) were then added and the reaction mixture was stir for 48 hours (or 24 hours) at room temperature (or at 40 °C). After complete consumption of isatin 2, DCE was removed under reduced pressure and the residue was subjected to column chromatography using petroleum ether/EtOAc = 9/1~5/1 as eluent to afford the desired product 3.

Reduction of 3a to 5:

At -30 °C, to a solution of 3a (48.9 mg, 0.1 mmol, 94% ee) in MeOH (1 mL) was added NaBH₄ (3.8 mg, 0.1 mmol), the reaction mixture was allowed to stir at that temperature for 0.5 hour, then it was warmed to room temperature and directly subjected to column chromatography using petroleum ether/EtOAc = 5/1 as eluent to afford the reductive product 5 (41.2 mg, 84% combined yield, 6:1 dr, 94% ee of the major diastereomer).

Deprotection of 3a to 6. [³]

Under a nitrogen atmosphere, to a solution of 3a (97.8 mg, 0.2 mmol, 94% ee) in DCM (6 mL) were successively added Et₃SiH (128 μL, 0.8 mmol) and TFA (2 mL), the reaction mixture was then stirred at room temperature for 6 hours. After complete consumption of 3a (monitored by TLC), the reaction mixture was quenched with saturated aq. NaHCO₃ (8 mL) and extracted with DCM (6 mL × 3). The combined organic layer was dried over Na₂SO₄, filtered, and concentrated under vacuum. The residue was purified by column chromatography using petroleum ether/EtOAc = 5/1 as eluent to afford the desired product 6 (38.1 mg, 77% yield, 94% ee).

References cited in the SI:
X-ray structure of product 3b

Absolute configurations of the products 3 were assigned based on the crystal X-ray structures of 3b. CCDC 1865244 (3b, obtained as yellow crystals via evaporation of a hexane/DCM solution) contains the supplementary X-ray crystallographic data. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.
Table S1. Crystal data and structure refinement for 3c.

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Characterization of products:

(R)-3-Hexanoyl-3-hydroxy-1-tritylindolin-2-one (3a): 36.7 mg, 75% yield, yellow solid; mp 66-68 °C; 1H NMR (400 MHz, CDCl3) δ 7.48 (d, J = 7.6 Hz, 6H), 7.29-7.19 (m, 9H), 7.07 (d, J = 6.8 Hz, 1H), 7.02-6.95 (m, 2H), 6.36 (d, J = 7.6 Hz, 1H), 4.78 (br, 1H), 1.96 (t, J = 7.6 Hz, 2H), 1.48-1.37 (m, 2H), 1.17-1.10 (m, 2H), 1.06-0.99 (m, 2H), 0.81 (t, J = 7.2 Hz, 3H); 13C NMR (100 MHz, CDCl3) δ 204.0, 173.5, 144.6, 141.5, 129.3, 129.0, 127.8, 127.1, 123.5, 123.1, 116.5, 82.9, 75.1, 36.5, 30.9, 22.9, 22.1, 13.8; IR νmax (KBr, cm⁻¹): 3440, 1736, 1464, 747, 705, 628; HRMS (ESI, m/z): calcd. for C33H31NO3Na⁺ 512.2196, found 512.2203. [α]20⁰D = -6.5 (c = 0.5 in CH2Cl2); HPLC analysis: 94% ee; [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 5.3 min (minor), 8.2 min (major)].

(R)-3-Hydroxy-3-propionyl-1-tritylindolin-2-one (3b): 34.4 mg, 77% yield, yellow solid; mp 184-186 °C; 1H NMR (400 MHz, CDCl3) δ 7.49 (d, J = 7.6 Hz, 6H), 7.29-7.19 (m, 9H), 7.08 (d, J = 6.8 Hz, 1H), 7.02-6.94 (m, 2H), 6.37 (d, J = 7.6 Hz, 1H), 4.76 (br, 1H), 2.09-1.95 (m, 2H), 0.91 (t, J = 7.2 Hz, 3H); 13C NMR (100 MHz, CDCl3) δ 204.7, 173.8, 144.6, 141.5, 129.3, 129.0, 127.8, 127.2, 127.1, 123.5, 123.2, 116.6, 82.9, 75.2, 29.9, 7.5; IR νmax (KBr, cm⁻¹): 3466, 1740, 1465, 751, 738, 700; HRMS (ESI, m/z): calcd. for C30H23NO3Na⁺ 470.1727, found 470.1736. [α]24⁰D = +0.3 (c = 0.5 in CH2Cl2); HPLC analysis: 96% ee; [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 6.1 min (minor), 8.3 min (major)].

(R)-3-Decanoyl-3-hydroxy-1-tritylindolin-2-one (3c): 38.7 mg, 71% yield, yellow solid; mp 87-89 °C; 1H NMR (400 MHz, CDCl3) δ 7.49 (d, J = 7.6 Hz, 6H), 7.28-7.21 (m, 9H), 7.07 (d, J = 7.2 Hz, 1H), 7.02-6.94 (m, 2H), 6.36 (d, J = 7.6 Hz, 1H), 4.82 (br, 1H), 1.98 (t, J = 7.6 Hz, 2H), 1.45-1.36 (m, 2H), 1.28-1.04 (m, 12H), 0.87 (t, J = 6.8 Hz, 3H); 13C NMR (100 MHz, CDCl3) δ 204.0, 173.6, 144.5, 141.5, 129.3, 129.0, 127.8, 127.0, 126.9, 123.5, 123.1, 116.5, 82.9, 75.0, 36.5, 31.8, 29.2, 29.1, 29.0, 28.7, 23.2, 22.6, 14.0; IR νmax (KBr, cm⁻¹): 3448, 1736, 1464, 774, 748, 705; HRMS(ESI, m/z): calcd. for C37H39NO3Na⁺ 568.2822, found 568.2823. [α]20⁰D = -1.9 (c = 0.5 in CH2Cl2); HPLC analysis: 94% ee; [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 5.3 min (minor), 7.7 min (major)].

(R)-3-Hydroxy-3-(3-phenylpropanoyl)-1-tritylindolin-2-one (3d): 31.9 mg, 61% yield, yellow solid; mp 61-63 °C; 1H NMR (400 MHz, CDCl3) δ 7.46 (d, J = 7.2 Hz, 6H), 7.25-7.16 (m, 12H), 7.00-6.88 (m, 5H), 6.33 (d, J = 8.0 Hz, 1H), 4.73 (br, 1H), 2.74 (t, J = 7.6 Hz, 2H), 2.39-2.23 (m, 2H); 13C NMR (100 MHz, CDCl3) δ 203.2, 173.6, 144.5, 141.4, 139.9, 129.3, 128.9, 128.4, 128.1, 127.8, 127.1, 126.7, 126.2, 123.5, 123.2, 116.5, 83.0, 75.1, 38.2, 29.3; IR νmax (KBr, cm⁻¹): 3433, 1736, 1464, 803, 747,
704; HRMS (ESI, m/z): calcd. for C_{36}H_{29}NO_3Na^+ 546.2040, found 546.2053. \([\alpha]_{D}^{27} = +0.8 \quad (c = 0.5 \text{ in CH}_2Cl_2); \) HPLC analysis: 92% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: \(i\text{-PrOH/hexane} = 20/80; \) retention times: 6.8 min (minor), 11.0 min (major)].

(R)-3-Hydroxy-3-(3-(4-methoxyphenyl)propanoyl)-1-trityl indolin-2-one (3e): 32.1 mg, 58% yield, yellow solid; mp 79-81 °C; \(^1H\) NMR (400 MHz, CDCl_3) \(\delta 7.45 \quad (d, \quad J = 7.2 \text{ Hz}, \quad 6H), \quad 7.24\) 7.15 (m, 9H), 6.99-6.87 (m, 3H), 6.84 (d, \(J = 8.4 \text{ Hz}, \quad 2H), \quad 6.74 \quad (d, \quad J = 8.8 \text{ Hz}, \quad 2H), \quad 6.33 \quad (d, \quad J = 8.0 \text{ Hz}, \quad 1H), \quad 4.71 \quad (br, \quad 1H), \quad 3.77 \quad (s, \quad 3H), \) 2.69 (t, \(J = 8.0 \text{ Hz}, \quad 2H), \quad 2.37-2.20 \quad (m, \quad 2H); \) \(^{13}C\) NMR (100 MHz, CDCl_3) \(\delta 203.2, \quad 173.6, \quad 158.1, \quad 144.6, \quad 141.5, \quad 132.0, \quad 129.3, \quad 129.0, \quad 129.0, \quad 127.8, \quad 127.1, \quad 126.7, \quad 123.5, \quad 123.1, \quad 116.5, \quad 113.8, \quad 83.0, \quad 75.2, \quad 55.3, \quad 38.5, \quad 28.5; \) IR (KBr, cm\(^{-1}\)): 3408, 1736, 1513, 824, 747, 705; HRMS (ESI, m/z): calcd. for C_{33}H_{31}NO_4H^+ 554.2326, found 554.2325. \([\alpha]_{D}^{25} = -1.0 \quad (c = 0.5 \text{ in CH}_2Cl_2); \) HPLC analysis: 92% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: \(i\text{-PrOH/hexane} = 20/80; \) retention times: 8.3 min (minor), 14.2 min (major)].

(R)-3-(3-(4-Bromophenyl)propanoyl)-3-hydroxy-1-tritylindolin-2-one (3f): 30.7 mg, 51% yield, yellow solid; mp 82-84 °C; \(^1H\) NMR (400 MHz, CDCl_3) \(\delta 7.46 \quad (d, \quad J = 7.6 \text{ Hz}, \quad 6H), \quad 7.31 \quad (d, \quad J = 8.4 \text{ Hz}, \quad 2H), \quad 7.25-7.17 \quad (m, \quad 9H), \quad 7.01-6.97 \quad (m, \quad 1H), \quad 6.93-6.88 \quad (m, \quad 2H), \quad 6.76 \quad (d, \quad J = 8.0 \text{ Hz}, \quad 2H), \quad 6.32 \quad (d, \quad J = 8.0 \text{ Hz}, \quad 1H), \quad 4.65 \quad (br, \quad 1H), \quad 2.67 \quad (t, \quad J = 7.6 \text{ Hz}, \quad 2H), \quad 2.30-2.13 \quad (m, \quad 2H); \) \(^{13}C\) NMR (100 MHz, CDCl_3) \(\delta 202.9, \quad 173.4, \quad 144.5, \quad 141.4, \quad 138.8, \quad 131.4, \quad 129.8, \quad 129.4, \quad 129.2, \quad 128.9, \quad 127.8, \quad 127.1, \quad 123.5, \quad 123.2, \quad 120.0, \quad 116.5, \quad 82.9, \quad 75.1, \quad 37.9, \quad 28.6; \) IR (KBr, cm\(^{-1}\)): 3434, 1735, 1488, 812, 746, 705; HRMS (ESI, m/z): calcd. for C_{33}H_{31}NO_3BrNa^+ 624.1145, found 624.1150. \([\alpha]_{D}^{24} = -0.6 \quad (c = 0.5 \text{ in CH}_2Cl_2); \) HPLC analysis: 90% ee, [CHIRALPAK IA column; 0.5 mL/min; solvent system: \(i\text{-PrOH/hexane} = 20/80; \) retention times: 12.4 min (minor), 29.9 min (major)].

(R)-3-Hydroxy-1-trityl-3-(undec-10-enoyl)indolin-2-one (3g): 42.3 mg, 76% yield, orange solid; mp 137-139 °C; \(^1H\) NMR (400 MHz, CDCl_3) \(\delta 7.48 \quad (d, \quad J = 7.2 \text{ Hz}, \quad 6H), \quad 7.28-7.19 \quad (m, \quad 9H), \quad 7.07 \quad (d, \quad J = 6.8 \text{ Hz}, \quad 1H), \quad 7.02-6.94 \quad (m, \quad 2H), \quad 6.36 \quad (d, \quad J = 8.4 \text{ Hz}, \quad 1H), \quad 5.85-5.74 \quad (m, \quad 1H), \quad 5.01-4.91 \quad (m, \quad 2H), \quad 4.79 \quad (br, \quad 1H), \quad 2.03-1.95 \quad (m, \quad 4H), \quad 1.43-1.03 \quad (m, \quad 12H); \) \(^{13}C\) NMR (100 MHz, CDCl_3) \(\delta 204.0, \quad 173.5, \quad 144.6, \quad 141.5, \quad 139.1, \quad 129.3, \quad 129.0, \quad 127.8, \quad 127.0, \quad 127.0, \quad 123.5, \quad 123.1, \quad 116.5, \quad 114.1, \quad 82.9, \quad 75.1, \quad 36.5, \quad 33.7, \quad 29.1, \quad 29.0, \quad 28.9, \quad 28.8, \quad 28.7, \quad 23.2; \) IR (KBr, cm\(^{-1}\)): 3440, 1737, 1464, 746, 705, 629; HRMS (ESI, m/z): calcd. for C_{33}H_{31}NO_3Na^+ 580.2822, found 580.2833. \([\alpha]_{D}^{27} = -0.4 \quad (c = 0.5 \text{ in CH}_2Cl_2); \) HPLC analysis: 92% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: \(i\text{-PrOH/hexane} = 20/80; \) retention times: 5.2 min (minor), 7.9 min (major)].
(R)-3-(Cyclopropanecarbonyl)-3-hydroxy-1-tritylindolin-2-one (3b): 32.1 mg, 70% yield, yellow solid; mp 191-193 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.50 (d, J = 7.6 Hz, 6H), 7.27-7.13 (m, 10H), 7.04-6.96 (m, 2H), 6.39 (d, J = 8.8 Hz, 1H), 4.88 (br, 1H), 1.28-1.24 (m, 1H), 1.16-1.05 (m, 2H), 0.85-0.74 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 203.9, 173.6, 144.7, 141.6, 129.2, 129.0, 127.8, 127.1, 127.0, 123.9, 123.2, 116.4, 83.1, 75.1, 16.4, 13.9, 12.0; IR νₘₐₓ (KBr, cm⁻¹): 3444, 1734, 1470, 763, 739, 706; HRMS (ESI, m/z): calcd. for C₃₉H₅₂NO₃Na⁺ 582.1727, found 582.1732. [α]°₂⁶ = -0.7 (c = 0.5 in CH₂Cl₂); HPLC analysis: 65% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 6.6 min (minor), 18.3 min (major)].

(R)-3-benzoyl-3-hydroxy-1-tritylindolin-2-one (3j): 15.8 mg, 32% yield, yellow slurry; ¹H NMR (400 MHz, CDCl₃) δ 7.46-7.45 (m, 8H), 7.24-7.20 (m, 10H), 6.92 (t, J = 7.6 Hz, 3H), 7.03 (t, J = 8.0 Hz, 1H), 6.92 (t, J = 7.6 Hz, 1H), 6.44 (d, J = 8.0 Hz, 1H), 5.42 (br, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 194.9, 173.3, 144.4, 141.3, 133.6, 133.5, 130.2, 129.4, 129.1, 128.4, 127.8, 127.0, 124.4, 123.2, 116.6, 81.5, 74.6; IR νₘₐₓ (KBr, cm⁻¹): 3415, 1733, 1340, 740, 704, 629; HRMS (ESI, m/z): calcd. for C₃₉H₅₂NO₃Na⁺ 518.1727, found 518.1730. [α]°₂⁶ = +1.3 (c = 0.5 in CH₂Cl₂); HPLC analysis: 28% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 7.3 min (major), 8.9 min (minor)].

(R)-3-hydroxy-3-(4-methoxybenzoyl)-1-tritylindolin-2-one (3k): 23.6 mg, 45% yield, yellow slurry; ¹H NMR (400 MHz, CDCl₃) δ 7.52 (d, J = 7.2 Hz, 6H), 7.28-7.17 (m, 11H), 7.07-7.00 (m, 2H), 6.90 (t, J = 7.6 Hz, 1H), 6.51 (d, J = 8.8 Hz, 2H), 6.45 (d, J = 8.0 Hz, 1H), 5.57 (br, 1H), 3.77 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 192.7, 173.4, 163.8, 144.2, 141.6, 132.3, 131.8, 129.3, 129.0, 127.8, 126.9, 125.0, 124.4, 123.1, 116.5, 113.7, 80.6, 74.4, 55.4; IR νₘₐₓ (KBr, cm⁻¹): 3415, 1735, 1400, 1259, 738, 706; HRMS (ESI, m/z): calcd. for C₃₉H₃₇NO₄Na⁺ 548.1832, found 548.1841. [α]°₂⁶ = +0.8 (c = 0.5 in CH₂Cl₂); HPLC analysis: 34% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 9.0 min (major), 11.0 min (minor)].

(R)-3-(Furan-2-carbonyl)-3-hydroxy-1-tritylindolin-2-one (3l): 30.6 mg, 63% yield, yellow solid; mp 198-200 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.52 (d, J = 7.6 Hz, 6H), 7.27-7.21 (m, 10H), 7.09 (d, J = 7.2 Hz, 1H), 7.01 (t, J = 7.6 Hz, 1H), 6.90 (t, J = 7.6 Hz, 1H), 6.57 (d, J = 3.6 Hz, 1H), 6.43 (d, J = 7.6 Hz, 1H), 6.25 (d, J = 2.0 Hz, 1H), 5.12 (br, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 182.4, 173.2, 148.5, 147.7, 144.7, 141.6, 129.3, 129.2, 128.0, 127.8, 126.9, 124.2, 122.9, 121.2, 116.4, 112.5, 80.4, 74.7; IR νₘₐₓ (KBr, cm⁻¹): 3385, 1726, 1463, 746, 706, 699; HRMS (ESI, m/z): calcd. for C₃₂H₂₃NO₄Na⁺ 508.1519, found 508.1531. [α]°₂⁶ = -1.2 (c = 0.5 in CH₂Cl₂); HPLC analysis: 43% ee,
(R)-3-Hexanoyl-3-hydroxy-5-methyl-1-tritylindolin-2-one (3m):
40.7 mg, 81% yield, yellow solid; mp 117-119 °C; 1H NMR (400 MHz, CDCl₃) δ 7.48 (d, J = 7.6 Hz, 6H), 7.28-7.18 (m, 9H), 6.88 (s, 1H), 6.80 (d, J = 8.4 Hz, 1H), 6.23 (d, J = 8.0 Hz, 1H), 4.77 (br, 1H), 2.20 (s, 3H), 1.98 (t, J = 7.2 Hz, 2H), 1.47-1.38 (m, 2H), 1.18-1.11 (m, 2H), 1.07-1.01 (m, 2H), 0.82 (t, J = 6.8 Hz, 3H); 13C NMR (100 MHz, CDCl₃) δ 204.1, 173.6, 142.1, 141.6, 132.9, 129.7, 129.0, 127.9, 127.8, 127.0, 124.1, 116.3, 83.0, 75.0, 36.5, 30.9, 22.9, 22.1, 20.6, 13.8; IR νmax (KBr, cm⁻¹): 3435, 1737, 1488, 743, 706; HRMS (ESI, m/z): calcd. for C₃₆H₃₇NO₃Na⁺, found 526.2362. [α][D] = -1.6 (c = 0.5 in CH₂Cl₂); HPLC analysis: 92% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 5.8 min (minor), 8.4 min (major)].

(R)-3-Hexanoyl-3-hydroxy-6-methyl-1-tritylindolin-2-one (3n):
36.2 mg, 72% yield, yellow solid; mp 99-101 °C; 1H NMR (400 MHz, CDCl₃) δ 7.48 (d, J = 8.4 Hz, 6H), 7.28-7.19 (m, 9H), 6.94 (d, J = 7.6 Hz, 1H), 6.77 (d, J = 7.6 Hz, 1H), 6.09 (s, 1H), 4.75 (br, 1H), 2.07 (s, 3H), 1.96-1.91 (m, 2H), 1.44-1.35 (m, 2H), 1.17-1.10 (m, 2H), 1.06-0.98 (m, 2H), 0.81 (t, J = 7.2 Hz, 3H); 13C NMR (100 MHz, CDCl₃) δ 204.2, 173.8, 144.8, 141.6, 139.5, 129.1, 127.7, 127.0, 124.2, 123.7, 123.2, 117.3, 82.7, 75.0, 36.5, 30.9, 22.9, 22.1, 22.0, 13.7; IR νmax (KBr, cm⁻¹): 3436, 1736, 1612, 1449, 1146, 706; HRMS (ESI, m/z): calcd. for C₃₆H₃₇NO₃Na⁺, found 526.2357. [α][D] = +0.1 (c = 0.5 in CH₂Cl₂); HPLC analysis: 95% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 5.5 min (minor), 8.0 min (major)].

(R)-3-Hexanoyl-3-hydroxy-5-methoxy-1-tritylindolin-2-one (3p):
41.0 mg, 79% yield, red solid; mp 92-94 °C; 1H NMR (400 MHz, CDCl₃) δ 7.48 (d, J = 7.6 Hz, 6H), 7.29-7.19 (m, 9H), 6.64 (d, J = 2.8 Hz, 1H), 6.53 (d, J = 8.8 Hz, 1H), 6.25 (d, J = 9.2 Hz, 1H), 4.84 (br, 1H), 3.67 (s, 3H), 1.98 (t, J = 7.2 Hz, 2H), 1.47-1.38 (m, 2H), 1.19-1.11 (m, 2H), 1.08-1.01 (m, 2H), 0.82 (t, J = 7.2 Hz, 3H); 13C NMR (100 MHz, CDCl₃) δ 204.0, 173.5, 155.9, 141.6, 137.6, 129.0, 128.2, 127.8, 127.0, 117.3, 114.6, 109.4, 83.2, 75.1, 55.6, 36.4, 30.9, 22.9, 22.1, 13.8; IR νmax (KBr, cm⁻¹): 3428, 1733, 1488, 807, 742, 706; HRMS (ESI, m/z): calcd. for C₃₆H₃₈NO₃Na⁺, found 542.2302, found 542.2317. [α][D] = -2.5 (c = 0.5 in CH₂Cl₂); HPLC analysis: 92% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 6.3 min (minor), 9.4 min (major)].

(R)-5-Fluoro-3-hexanoyl-3-hydroxy-1-tritylindolin-2-one (3q):
19.8 mg, 39% yield, yellow solid; mp 87-89 °C; 1H NMR (400 MHz, CDCl₃) δ 7.54 (d, J = 7.9 Hz, 6H), 7.28-7.18 (m, 9H), 6.88 (s, 1H), 6.79 (d, J = 8.4 Hz, 1H), 6.23 (d, J = 8.0 Hz, 1H), 4.77 (br, 1H), 2.20 (s, 3H), 1.98 (t, J = 7.2 Hz, 2H), 1.47-1.38 (m, 2H), 1.18-1.11 (m, 2H), 1.07-1.01 (m, 2H), 0.80 (t, J = 6.8 Hz, 3H); 13C NMR (100 MHz, CDCl₃) δ 204.1, 173.6, 142.1, 141.6, 132.9, 129.7, 129.0, 127.9, 127.8, 127.0, 124.1, 116.3, 83.0, 75.0, 36.5, 30.9, 22.9, 22.1, 20.6, 13.8; IR νmax (KBr, cm⁻¹): 3435, 1737, 1488, 743, 706; HRMS (ESI, m/z): calcd. for C₃₆H₃₇NO₃Na⁺, found 526.2362. [α][D] = -1.6 (c = 0.5 in CH₂Cl₂); HPLC analysis: 92% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 5.8 min (minor), 8.4 min (major)].
\(\text{MHz, } \text{CDCl}_3 \delta 7.46 \ (d, J = 7.2 \text{ Hz}, 6H), 7.29-7.20 \ (m, 9H), 6.83-6.81 \ (m, 1H), 6.74-6.69 \ (m, 1H), 6.32-6.29 \ (m, 1H), 4.78 \ (br, 1H), 2.02 \ (t, J = 7.2 \text{ Hz}, 2H), 1.49-1.42 \ (m, 2H), 1.20-1.13 \ (m, 2H), 1.10-1.03 \ (m, 2H), 0.83 \ (t, J = 7.2 \text{ Hz}, 3H), \) \(^{13}\text{C NMR} \) (100 MHz, \(\text{CDCl}_3 \)) \(\delta 203.4, 173.5, 158.9 \ (d, J_{\text{C-F}} = 243.1 \text{ Hz}), 141.3, 140.5, 129.0, 128.6 \ (d, J_{\text{C-F}} = 7.4 \text{ Hz}), 127.9, 127.2, 117.3 \ (d, J_{\text{C-F}} = 7.3 \text{ Hz}), 115.8 \ (d, J_{\text{C-F}} = 22.8 \text{ Hz}), 111.2 \ (d, J_{\text{C-F}} = 24.5 \text{ Hz}), 82.9, 75.2, 36.6, 30.9, 22.9, 22.1, 13.8; IR \nu_{\text{max}} \) (KBr, cm\(^{-1}\)): 3442, 1740, 1467, 813, 740, 705; HRMS (ESI, m/z): calcd. for \(C_{33}H_{30}NO_3Na^+ \) 530.2102, found 530.2106. [\(\alpha\)]\(^{23}\)\(_D\) = +1.0 \(c = 0.5\) in \(\text{CH}_2\text{Cl}_2\); HPLC analysis: 90% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: \(i\)-PrOH/hexane = 20/80; retention times: 4.7 min (minor), 6.5 min (major)].

\(\text{(R)-5-Chloro-3-hexanoyl-3-hydroxy-1-tritylldolin-2-one (3r): 24.6 mg, 47% yield, yellow solid; mp 65-67 °C; }^{1}\text{H NMR} \) (400 MHz, \(\text{CDCl}_3 \)) \(\delta 7.45 \ (d, J = 8.4 \text{ Hz}, 6H), 7.29-7.21 \ (m, 9H), 7.06 \ (d, J = 2.4 \text{ Hz}, 1H), 6.97 \ (d, J = 8.8 \text{ Hz}, 1H), 6.29 \ (d, J = 8.8 \text{ Hz}, 1H), 4.77 \ (br, 1H), 2.03 \ (t, J = 7.6 \text{ Hz}, 2H), 1.50-1.41 \ (m, 2H), 1.21-1.14 \ (m, 2H), 1.10-1.03 \ (m, 2H), 0.84 \ (t, J = 7.2 \text{ Hz}, 3H); \) \(^{13}\text{C NMR} \) (100 MHz, \(\text{CDCl}_3 \)) \(\delta 203.3, 173.3, 143.2, 141.2, 129.3, 129.0, 128.8, 128.7, 127.9, 127.3, 123.8, 117.4, 82.7, 75.3, 36.6, 30.9, 22.8, 22.2, 13.8; IR \nu_{\text{max}} \) (KBr, cm\(^{-1}\)): 3427, 1739, 1468, 812, 740, 705; HRMS (ESI, m/z): calcd. for \(C_{33}H_{30}NO_3ClNa^+ \) 546.1806, found 546.1812. [\(\alpha\)]\(^{23}\)\(_D\) = +1.8 \(c = 0.5\) in \(\text{CH}_2\text{Cl}_2\); HPLC analysis: 84% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: \(i\)-PrOH/hexane = 20/80; retention times: 4.8 min (minor), 6.5 min (major)].

\(\text{(R)-5-Bromo-3-hexanoyl-3-hydroxy-1-tritylldolin-2-one (3s): 24.9 mg, 44% yield, yellow solid; mp 86-88 °C; }^{1}\text{H NMR} \) (400 MHz, \(\text{CDCl}_3 \)) \(\delta 7.45 \ (d, J = 6.8 \text{ Hz}, 6H), 7.29-7.19 \ (m, 10H), 7.12 \ (d, J = 8.8 \text{ Hz}, 1H), 6.24 \ (d, J = 8.8 \text{ Hz}, 1H), 4.77 \ (br, 1H), 2.04 \ (t, J = 6.8 \text{ Hz}, 2H), 1.50-1.41 \ (m, 2H), 1.21-1.14 \ (m, 2H), 1.10-1.03 \ (m, 2H), 0.84 \ (t, J = 7.2 \text{ Hz}, 3H); \) \(^{13}\text{C NMR} \) (100 MHz, \(\text{CDCl}_3 \)) \(\delta 203.3, 173.2, 143.7, 141.2, 132.2, 129.1, 128.9, 127.9, 127.3, 126.6, 117.9, 116.1, 82.7, 75.3, 36.6, 30.9, 22.9, 22.2, 13.8; IR \nu_{\text{max}} \) (KBr, cm\(^{-1}\)): 3431, 1740, 1466, 812, 742, 706; HRMS (ESI, m/z): calcd. for \(C_{33}H_{30}NO_3BrNa^+ \) 590.1301, found 590.1303. [\(\alpha\)]\(^{26}\)\(_D\) = -1.6 \(c = 0.5\) in \(\text{CH}_2\text{Cl}_2\); HPLC analysis: 82% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: \(i\)-PrOH/hexane = 20/80; retention times: 4.9 min (minor), 6.7 min (major)].

\(\text{(R)-3-hexanoyl-3-hydroxy-5-iodo-1-tritylldolin-2-one (3t): 26.4 mg, 43% yield, yellow solid; mp 98-100 °C; }^{1}\text{H NMR} \) (400 MHz, \(\text{CDCl}_3 \)) \(\delta 7.45 \ (d, J = 7.6 \text{ Hz}, 6H), 7.35 \ (s, 1H), 7.32-7.20 \ (m, 10H), 6.12 \ (d, J = 8.4 \text{ Hz}, 1H), 4.76 \ (br, 1H), 2.03 \ (t, J = 7.2 \text{ Hz}, 2H), 1.50-1.41 \ (m, 2H), 1.21-1.14 \ (m, 2H), 1.10-1.03 \ (m, 2H), 0.84 \ (t, J = 7.2 \text{ Hz}, 3H); \) \(^{13}\text{C NMR} \) (100 MHz, \(\text{CDCl}_3 \)) \(\delta 203.3, 173.0, 144.4, 141.2, 138.1, 132.2, 129.3, 129.0, 127.9, 127.3, 118.3, 86.1, 82.5, 75.3, 36.6, 30.9, 22.9, 22.2, 13.8; IR \nu_{\text{max}}\)
(R)-1-(bis(4-Methoxyphenyl)(phenyl)methyl)-3-hexanoyl-3-hydroxyindolin-2-one (3v): 39.5 mg, 72% yield, pale yellow solid; mp 146-148 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.43-7.37 (m, 6H), 7.27-7.17 (m, 3H), 7.07 (d, \(J = 6.8\) Hz, 1H), 7.03-6.94 (m, 2H), 6.79 (d, \(J = 8.8\) Hz, 4H), 6.37 (d, \(J = 8.0\) Hz, 1H), 4.80 (br, 1H), 3.76 (s, 6H), 2.01 (t, \(J = 7.2\) Hz, 2H), 1.48-1.38 (m, 2H), 1.19-1.12 (m, 2H), 1.08-1.00 (m, 2H), 0.82 (t, \(J = 7.2\) Hz, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 204.0, 173.6, 158.3, 144.8, 142.3, 133.8, 133.7, 130.3, 129.3, 128.6, 127.7, 127.0, 126.7, 123.4, 123.0, 116.6, 113.0, 83.0, 74.4, 55.1, 36.5, 30.9, 22.9, 22.1, 13.8; IR \(\nu\) \(\text{max}\) (KBr, cm\(^{-1}\))): 3466, 1728, 1254, 835, 773, 726; HRMS (ESI, m/z): calcd. for C\(_{33}\)H\(_{30}\)NO\(_3\)Na\(^+\) 572.2407, found 572.2410. \([\alpha]^{25}_D = +0.1\) (c = 0.5 in CH\(_2\)Cl\(_2\)); HPLC analysis: 95% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: \(i\)-PrOH/hexane = 20/80; retention times: 8.8 min (minor), 25.7 min (major)].

(R)-1-Benzyl-3-hexanoyl-3-hydroxyindolin-2-one (3w): 20.9 mg, 62% yield, viscous solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.35-7.28 (m, 6H), 7.13 (d, \(J = 7.2\) Hz, 1H), 7.07 (t, \(J = 7.6\) Hz, 1H), 6.86 (d, \(J = 8.0\) Hz, 1H), 5.08 (d, \(J = 15.6\) Hz, 1H), 5.06 (br, 1H), 4.79 (d, \(J = 15.2\) Hz, 1H), 2.25-2.07 (m, 2H), 1.55-1.45 (m, 2H), 1.15-1.05 (m, 4H), 0.79 (t, \(J = 7.2\) Hz, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 203.2, 173.1, 143.9, 135.1, 130.7, 128.9, 128.0, 127.5, 126.5, 124.0, 123.7, 110.0, 83.4, 44.4, 35.9, 30.8, 23.1, 22.0, 13.7; IR \(\nu\) \(\text{max}\) (KBr, cm\(^{-1}\))): 3421, 1736, 1613, 1468, 752, 698; HRMS (ESI, m/z): calcd. for C\(_{31}\)H\(_{29}\)NO\(_3\)Na\(^+\) 360.1570, found 360.1574. \([\alpha]^{25}_D = -2.6\) (c = 0.5 in CH\(_2\)Cl\(_2\)); HPLC analysis: 82% ee, [CHIRALPAK OJ-H column; 1 mL/min; solvent system: \(i\)-PrOH/hexane = 10/90; retention times: 10.5 min (minor), 12.8 min (major)].

(R)-1- Allyl-3-hexanoyl-3-hydroxyindolin-2-one (3x): 18.1 mg, 63% yield, viscous solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.38 (t, \(J = 8.0\) Hz, 1H), 7.16-7.08 (m, 2H), 6.93 (d, \(J = 8.0\) Hz, 1H), 5.92-5.82 (m, 1H), 5.35-5.28 (m, 2H), 5.02 (br, 1H), 4.48 (dd, \(J_1 = 16.0\) Hz, \(J_2 = 5.2\) Hz, 1H), 4.29 (dd, \(J_1 = 16.0\) Hz, \(J_2 = 5.2\) Hz, 1H), 2.30-2.12 (m, 2H), 1.54-1.48 (m, 2H), 1.19-1.06 (m, 4H), 0.80 (t, \(J = 7.2\) Hz, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 203.3, 172.8, 144.0, 130.7, 130.6, 126.5, 124.0, 123.7, 118.4, 109.9, 83.3, 42.9, 35.7, 30.9, 23.1, 22.1, 13.7; IR \(\nu\) \(\text{max}\) (KBr, cm\(^{-1}\))): 3333, 1721, 1686, 1469, 760, 683; HRMS (ESI, m/z): calcd. for C\(_{17}\)H\(_{20}\)NO\(_3\)Na\(^+\) 310.1414, found 310.1418. \([\alpha]^{26}_D = -3.7\) (c = 0.5 in CH\(_2\)Cl\(_2\)); HPLC analysis: 50% ee, [CHIRALPAK AS-H column; 1 mL/min; solvent system: \(i\)-PrOH/hexane = 10/90; retention times: 14.3 min (minor), 20.1 min (major)].
(3R)-3-Hydroxy-3-(1-hydroxyhexyl)-1-tritylindolin-2-one (5): 41.2 mg, 84% combined yield, 6:1 dr, viscous solid; $^1$H NMR (400 MHz, CDCl$_3$) of major diastereomer: $\delta$ 7.46 (d, $J$ = 7.2 Hz, 6H), 7.27-7.20 (m, 10H), 6.99-6.92 (m, 2H), 6.27 (d, $J$ = 7.2 Hz, 1H), 4.07 (br, 1H), 3.82-3.76 (m, 1H), 2.62 (d, $J$ = 10.8 Hz, 1H), 1.45-1.39 (m, 1H), 1.17-1.07 (m, 5H), 0.97-0.92 (m, 2H), 0.82 (t, $J$ = 6.8 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) of major diastereomer: $\delta$ 179.0, 144.1, 141.6, 129.2, 128.5, 128.0, 127.7, 127.6, 127.0, 123.5, 116.2, 76.0, 74.6, 31.5, 31.3, 25.7, 22.3, 13.9; IR $\nu_{\text{max}}$ (KBr, cm$^{-1}$) of major diastereomer: 3448, 1717, 1464, 739, 704, 654; HRMS (ESI, m/z): calcd. for C$_{33}$H$_{33}$NO$_3$Na$^+$ 514.2353, found 514.2360. [a]$^{24}_D$ = -3.9 (c = 0.5 in CH$_2$Cl$_2$); HPLC analysis of major diastereomer: 94% ee, [CHIRALPAK IC column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 8.1 min (minor), 11.5 min (major)].

(R)-3-Hexanoyl-3-hydroxyindolin-2-one (6): 38.1 mg, 77% yield, viscous solid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.14 (br, 1H), 7.33 (t, $J$ = 7.6 Hz, 1H), 7.13 (d, $J$ = 7.2 Hz, 1H), 7.08 (t, $J$ = 7.6 Hz, 1H), 6.99 (d, $J$ = 7.6 Hz, 1H), 5.17 (br, 1H), 2.41-2.33 (m, 1H), 2.29-2.21 (m, 1H), 1.57-1.49 (m, 2H), 1.21-1.08 (m, 4H), 0.80 (t, $J$ = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 203.3, 175.8, 141.9, 130.9, 127.0, 124.2, 123.7, 111.1, 83.9, 35.6, 30.8, 23.1, 22.1, 13.7; IR $\nu_{\text{max}}$ (KBr, cm$^{-1}$): 3285, 1736, 1619, 1472, 1191, 754; HRMS (ESI, m/z): calcd. for C$_{14}$H$_7$NO$_3$Na$^+$ 270.1101, found 270.1102. [a]$^{25}_D$ = -5.8 (c = 0.5 in CH$_2$Cl$_2$); HPLC analysis: 94% ee, [CHIRALPAK IA column; 1 mL/min; solvent system: i-PrOH/hexane = 20/80; retention times: 6.6 min (major), 9.2 min (minor)].
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