Supporting Information

Oxidative NHCs catalysis: direct activation of β sp³ carbons of saturated acid chlorides

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1. General information

¹H NMR and ¹³C NMR spectra were recorded on a Bruker AVANCE III 400 spectrometer using tetramethylsilane as internal reference, and chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz, respectively. The HRMS analysis was obtained on a Bruker Apex II FT-ICR mass spectrometer with ESI ionization method. The *ee* value determination was carried out using HPLC with chiral Chirapak column on Agilent 1260 with a UV-detector. Optical rotation was measured by the Perkin Elmer 341 polarimeter. Melting points were taken on an X–4 melting point apparatus and were uncorrected. Dichloromethane was freshly distilled from phosphorous pentoxide. Toluene was freshly distilled from a deep-blue solution of sodium-benzophenone under nitrogen. DBU, TBD, K₂CO₃, Cs₂CO₃, DIPEA and DABCO were purchased from commercial suppliers and used directly. All syntheses and manipulations were carried out under dry nitrogen atmosphere. Flash column chromatography was carried out utilizing 200–300 mesh silica gel.

2. General procedures



2.1 Synthesis of N-heterocyclic carbene catalyst D

A flame-dried 100 mL round-bottom flask was charged with (*S*)-3-methyl-1-(1-phenylcyclobutyl)butan-1-amine (10 mmol) and reprocessed toluene (40 mL). Then benzoyl chloride (10 mmol) was added slowly. After that, the reaction mixture was refluxed and stirred for 16 h. Upon cooling, thionyl chloride (30 mmol) was added to the reaction, the reaction mixture was stirred at 85 °C for 48 h. After the reaction completed, the solvent was removed *in vacuo* and the remains was dissolved in THF. Keeping the solution stirred at 0 °C for 15 min before NEt₃ (30 mmol) and phenylhydrazine (10 mmol) was added in one portion. The reaction mixture was stirred for 16 h at room temperature and then reflux for 5 h. The solvent was removed *in vacuo* and a solution of acetic acid (2%, 20 mL) was added. The mixture was heated to 75 °C and stirred for 1 h. After the reaction completed, the product was precipitated at an ice-water bath.

To a 50 mL round bottom flask equiped with a magnetic stir bar was added (*S*)-*N*-(3-methyl-1-(1-phenylcyclobutyl)butyl)-*N*'-phenylbenzohydrazonamide (5 mmol), $NH_4^+BF_4^-$ (10 mmol) and of triethoxy-methane (30 mmol). The reaction was carried out at 125 °C for 18 h. After the reaction was completed, the

clear dark solution was concentrated. The residue was purified by column chromatography using MeOH/DCM = 1/20 as eluent to yield the desired product **D** in 32% yield as light yellow solid.

¹H NMR (300 MHz, DMSO-*d*₆) δ 10.33 (s, 1H), 7.96 – 7.47 (m, 10H), 7.38 – 7.28 (m, 3H), 7.09 – 6.96 (m, 2H), 4.74 (d, J = 10.1 Hz, 1H), 2.33 (d, J = 6.5 Hz, 2H), 2.13 (d, J = 5.5 Hz, 1H), 1.99 (t, J = 11.9 Hz, 1H), 1.92 – 1.65 (m, 3H), 1.59 – 1.42 (m, 2H), 0.92 (dd, J = 9.3, 6.1 Hz, 6H). ¹³C NMR (75 MHz, DMSO-*d*₆) δ 155.11, 142.17, 140.04, 134.86, 132.48, 130.86, 130.05, 129.84, 129.57, 128.46, 127.52, 127.38, 122.68, 121.05, 65.14, 51.13, 31.29, 30.97, 23.35, 23.22, 21.31, 15.52.

2.2 Procedure for the synthesis of compounds 3a–3f and 3h–3p



A stirred mixture of 3-substituted propanoyl chloride (1) (0.25 mmol), oxidant (0.25 mmol, 102.2 mg), 4Å MS (100 mg), *N*-heterocyclic carbene catalyst **D** (0.02 mmol, 10.2 mg), TBD (0.25 mmol, 34.8 mg), and anhydrous DCM:toluene (1:1) (1 mL) was stirred for 0.5 h under nitrogen atmosphere. Then, a solution of substrate **2** (0.1 mmol) in DCM:toluene (1:1) (0.5 mL) was added and stirred at room temperature. The reaction was monitored by TLC. When the substrate **2** was disappeared, the reaction was quenched by water (2 mL) and the aqueous layer was extracted with ethyl acetate (5 mL×3). The combined organic layer was dried over anhydrous MgSO₄. After removal of the solvent under reduced pressure, the crude residue was purified by flash column chromatography on silica gel using PE/EA (5:1) to afford the desired products **3**.

2.3 Procedure for the synthesis of compounds 5a-5i



A stirred mixture of 3-substituted propanoyl chloride (1a) (0.25 mmol, 42.2 mg), oxidant (0.25 mmol, 102.2 mg), 4Å MS (100 mg), *N*-heterocyclic carbene catalyst **D** (0.02 mmol, 10.2 mg), Cs_2CO_3 (0.25 mmol, 81.5 mg), and anhydrous DCM:toluene (4:1) (1 mL) was stirred for 0.5 h under nitrogen atmosphere. Then,

a solution of substrate 4 (0.1 mmol) in DCM:toluene (4:1) (0.5 mL) was added, and the reaction was stirred for 48 h at room temperature. The reaction was quenched by water (2 mL), and the aqueous layer was extracted with ethyl acetate (5 mL×3). The combined organic layer was dried over anhydrous MgSO₄. After removal of the solvent under reduced pressure, the crude residue was purified by flash column chromatography on silica gel using PE/EA (5:1) to afford the desired products **5**.

2.4 Procedure for the synthesis of compound 7a-7b



A stirred mixture of 3-phenylpropanoyl chloride (1a) (0.25 mmol, 42.2 mg), oxidant (0.25 mmol, 102.2 mg), 4Å MS (100 mg), *N*-heterocyclic carbene catalyst **D** (0.02 mmol, 10.2 mg), DBU (0.25 mmol, 37.4 μ L), and anhydrous toluene (1 mL) was stirred for 0.5 h under nitrogen atmosphere. Then, a solution of enamine **6** (0.1 mmol, 19.1 mg) in toluene (0.5 mL) was added, and the reaction was stirred for 36 h at room temperature. The reaction was quenched by water (2 mL), and the aqueous layer was extracted with ethyl acetate (5 mL×3). The combined organic layer was dried over anhydrous MgSO₄. After removal of the solvent under reduced pressure, the crude residue was purified by flash column chromatography on silica gel using PE/EA (5:1) to afford the desired product **7a**.

3. Characterization of compounds 3a–3f and 3h–3p



(2S, 3R)-1'-methyl-3-phenyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3a)

white solid, mp 148 –151 °C, $[\alpha]_{D}^{20}$ –99 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.54 (d, J = 7.2 Hz, 1H), 7.41 – 7.37 (m, 1H), 7.22 – 7.14 (m, 4H), 6.93 (d, J = 7.2 Hz, 2H), 6.67 (d, J = 7.6 Hz, 1H), 4.07 (dd, J = 14.0, 8.0 Hz, 1H), 3.82 (dd, J = 16.8, 13.6 Hz, 1H), 2.91 (dd, J = 16.8, 8.0 Hz, 1H), 2.82 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 174.79, 172.57, 144.32, 132.07, 131.27, 128.42, 128.26, 127.54, 124.73, 124.12, 123.47, 108.58, 86.43, 50.99, 32.18, 25.78. HRMS (ESI): Exact Mass Calcd. for C₁₈H₁₆NO₃

 $(M+H)^+$: 294.1125, Found: 294.1128. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, retention time: $t_{major} = 18.122 \text{ min}, t_{minor} = 36.708 \text{ min}, 96\% \text{ ee}$).



(2S, 3R)-4'-chloro-1'-methyl-3-phenyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3b)

White solid, mp 127 – 130 °C, $[\alpha]_{D}^{20}$ –95 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.32 (t, *J* = 8.4 Hz, 1H), 7.22 – 7.16 (m, 3H), 7.13 (d, *J* = 8.0 Hz, 1H), 6.96 (d, *J* = 8.0 Hz, 2H), 6.57 (d, *J* = 7.6 Hz, 1H), 4.70 (dd, *J* = 13.6, 8.4 Hz, 1H), 3.80 (dd, *J* = 16.8, 13.6 Hz, 1H), 2.92 (dd, *J* = 16.8, 8.0 Hz, 1H), 2.78 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 174.35, 171.95, 146.00, 132.36, 132.20, 131.96, 128.46, 128.31, 127.59, 124.50, 120.70, 107.06, 86.73, 46.06, 31.33, 25.92. HRMS (ESI): Exact Mass Calcd. for C₁₈H₁₅ClNO₃ (M+H)⁺: 328.0735, Found: 328.0731. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: t_{major} = 27.951 min, t_{minor} = 46.048 min, 95% ee).



(2S, 3R)-5'-chloro-1'-methyl-3-phenyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3c)

white solid, mp 133 – 135 °C, $[\alpha]_{p}^{20}$ –110 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.53 (d, J = 2.0 Hz, 1H), 7.37 – 7.35 (d, J = 8.4, 2.0 Hz, 1H), 7.24 – 7.16 (m, 3H), 6.95 (d, J = 7.2 Hz, 2H), 6.60 (d, J = 8.4 Hz, 1H), 4.04 (dd, J = 13.6, 8.0 Hz, 1H), 3.79 (dd, J = 16.8, 14.0 Hz, 1H), 2.92 (dd, J = 16.8, 8.0 Hz, 1H), 2.80 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 174.24, 172.22, 142.84, 131.67, 131.19, 128.93, 128.54, 128.48, 127.54, 126.47, 124.64, 109.61, 86.07, 51.13, 32.01, 25.91. HRMS (ESI): Exact Mass Calcd. for C₁₈H₁₅ClNO₃ (M+H)⁺: 328.0735, Found: 328.0740. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, retention time: t_{major} = 8.943 min, t_{minor} = 14.912 min, 92% ee).





white solid, mp 99 – 102 °C, $[\alpha]_{D}^{20}$ –119 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.30 (dd, J = 7.2, 2.4 Hz, 1H), 7.24 – 7.16 (m, 3H), 7.12 – 7.07 (m, 1H), 6.95 (d, J = 7.2 Hz, 2H), 6.60 (dd, J = 8.8, 4.0 Hz, 1H), 4.03 (dd, J = 13.6, 8.0 Hz, 1H), 3.81 (dd, J = 16.8, 13.6 Hz, 1H), 2.92 (dd, J = 16.8, 7.6 Hz, 1H), 2.80 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 174.34, 172.38, 160.70, 158.29, 140.25, 131.70, 128.52, 128.46, 127.54, 126.35, 126.27, 117.74, 117.51, 112.44, 112.19, 109.40, 109.32, 86.27, 51.23, 32.05, 25.93. HRMS (ESI): Exact Mass Calcd. for C₁₈H₁₅FNO₃ (M+H)⁺: 312.1035, Found: 312.1037. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: t_{major} = 18.068 min, t_{minor} = 24.745 min, 93% ee).



(2S, 3R)-1',5'-dimethyl-3-phenyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3e)

white solid, mp 127 – 130 °C, $[\alpha]_{D}^{20}$ –132 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.36 (s, 1H), 7.22 – 7.14 (m, 4H), 6.93 (d, *J* = 7.2 Hz, 2H), 6.55 (d, *J* = 8.0 Hz 1H), 4.06 (dd, *J* = 13.6, 8.0 Hz, 1H), 3.81 (dd, *J* = 16.8, 13.6 Hz, 1H), 2.90 (dd, *J* = 16.8, 8.0 Hz, 1H), 2.79 (s, 3H), 2.41 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 174.80, 172.51, 141.94, 133.17, 132.24, 131.50, 128.38, 128.19, 127.52, 124.81, 124.74, 108.33, 86.55, 50.87, 32.21, 25.78, 21.10. HRMS (ESI): Exact Mass Calcd. for C₁₉H₁₈NO₃ (M+H)⁺: 308.1281, Found: 308.1277. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: t_{major} = 18.146 min, t_{minor} = 36.620 min, 97% ee).



(2S, 3R)-6'-bromo-1'-methyl-3-phenyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3f)

white solid, mp 189 – 192 °C, $[\alpha]_{D}^{20}$ –131 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.40 (d, J = 8.0Hz, 1H), 7.35 (dd, J = 8.0, 1.6 Hz, 1H), 7.23 – 7.17 (m, 3H), 6.94 (d, J = 8.0 Hz, 2H), 6.83 (d, J = 1.6 Hz, 1H), 4.04 (dd, J = 13.6, 8.0 Hz, 1H), 3.80 (dd, J = 16.8, 13.6 Hz, 1H), 2.92 (dd, J = 16.8, 8.0 Hz, 1H), 2.80 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 174.44, 172.44, 145.51, 131.65, 128.58, 128.47, 127.50, 126.35, 125.38, 125.11, 123.63, 112.24, 85.95, 50.90, 32.04, 25.92. HRMS (ESI): Exact Mass Calcd. for C₁₈H₁₅BrNO₃ (M+H)⁺: 372.0230, Found: 372.0238. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, retention time: t_{major} = 8.964 min, t_{minor} = 12.685 min, 97% ee).



(2S, 3R)-1'-allyl-3-phenyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3h)

white solid, mp 163 – 165 °C, $[\alpha]_{D}^{20}$ –113 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.56 (d, *J* = 7.2 Hz, 1H), 7.35 (t, *J* = 7.6 Hz 1H), 7.23 – 7.15 (m, 4H), 6.95 (d, *J* = 7.2 Hz, 2H), 6.63 (d, *J* = 8.0 Hz 1H), 5.26 – 5.16 (m, 1H), 4.82 (d, *J* = 10.4 Hz, 1H), 4.35 (d, *J* = 17.2 Hz, 1H), 4.28 (dd, *J* = 4.0, 2.0 Hz, 1H), 4.24 (dd, *J* = 4.0, 2.0 Hz, 1H), 4.11 (dd, *J* = 14.0, 8.0 Hz, 1H), 3.85 (dd, *J* = 16.8, 14.0 Hz, 1H), 3.71 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.92 (dd, *J* = 16.8, 8.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 174.78, 172.33, 143.57, 131.94, 131.16, 129.96, 128.54, 128.30, 127.76, 124.61, 124.17, 123.41, 116.98, 109.52, 86.41, 51.10, 41.95, 32.06. HRMS (ESI): Exact Mass Calcd. for C₂₀H₁₈NO₃ (M+H)⁺: 320.1281, Found: 320.1285. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: t_{minor} = 15.607 min, t_{major} = 17.554 min, 94% ee).



(2S, 3R)-3-phenyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3i)

white solid, mp 111 – 114 °C, $[\alpha]_{D}^{20}$ –89 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.54 (d, J = 7.2 Hz, 1H), 7.35 – 7.31 (m, 1H), 7.23 – 7.16 (m, 4H), 7.04 (br s, 1H), 6.97 (d, J = 7.2 Hz, 2H), 6.71 (d, J = 7.6 Hz, 1H), 4.09 (dd, J = 13.6, 8.0 Hz, 1H), 3.79 (dd, J = 16.8, 13.6 Hz, 1H), 2.92 (dd, J = 16.8, 8.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 174.67, 173.98, 141.28, 132.00, 131.31, 128.62, 128.37, 127.65, 125.07, 124.67, 123.58, 110.38, 86.32, 50.94, 32.11. HRMS (ESI): Exact Mass Calcd. for C₁₇H₁₄NO₃ (M+H)⁺: 280.0968, Found: 280.0964. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: t_{major} = 8.706 min, t_{minor} = 24.000 min, 95% ee).



(2S, 3R)-3-phenyl-1'-tosyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3j)

white solid, mp 202 – 204 °C, $[\alpha]_{D}^{20}$ –102 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, *J* = 8.0 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.49 – 7.45 (m, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.23 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 7.6 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 2H), 6.81 (d, *J* = 7.6 Hz, 2H), 4.01 (dd, *J* = 13.6, 8.0 Hz, 1H), 3.67 (dd, *J* = 17.2, 13.6 Hz, 1H), 2.90 (dd, *J* = 17.2, 8.0 Hz, 1H), 2.42 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 173.80, 171.40, 145.87, 139.94, 134.46, 131.97, 130.57, 129.90, 128.81, 128.68, 127.77, 127.29, 125.67, 124.52, 123.89, 113.79, 85.77, 51.94, 31.66, 21.70. HRMS (ESI): Exact Mass Calcd. for C₂₄H₂₀NO₅S (M+H)⁺: 434.1057, Found: 434.1066. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, retention time: t_{major} = 13.383 min, t_{minor} = 18.311 min, 87% ee).



(2S, 3R)-3-(2-chlorophenyl)-1'-methyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3k)

white solid, mp 122 – 125 °C, $[\alpha]_{D}^{20}$ –141 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.63 (d, J = 8.0 Hz, 1H), 7.57 (d, J = 7.6 Hz, 1H), 7.36 – 7.32 (m, 1H), 7.25 – 7.21 (m, 1H), 7.19 – 7.12 (m, 3H), 6.65 (d, J = 7.6 Hz, 1H), 4.83 (dd, J = 13.6, 8.4 Hz, 1H), 3.66 (dd, J = 16.8, 13.6 Hz, 1H), 2.97 (dd, J = 16.8, 8.4 Hz, 1H), 2.91 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 174.31, 172.93, 143.91, 134.91, 131.23, 130.18, 129.83, 129.34, 129.03, 126.98, 125.67, 123.82, 123.22, 108.47, 86.37, 45.24, 34.21, 25.98. HRMS (ESI): Exact Mass Calcd. for C₁₈H₁₅CINO₃ (M+H)⁺ : 328.0735, Found: 328.0739. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, retention time: t_{minor} = 6.835 min, t_{major} = 10.468 min, 90% ee).



(2S, 3R)-3-(3-chlorophenyl)-1'-methyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3l)

white solid, mp 134 – 137 °C, $[\alpha]_{D}^{20}$ –127 (c 1.0, CH₂Cl₂), $[\alpha]_{D}^{20}$ -97(c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.53 (d, J = 7.2 Hz, 1H), 7.42 (t, J = 8.0 Hz, 1H), 7.24 – 7.18 (m, 2H), 7.11 (t, J = 8.0 Hz, 1H), 6.89 (s, 1H), 6.84 (d, J = 7.6 Hz, 1H), 6.72 (d, J = 7.6 Hz, 1H), 4.04 (dd, J = 14.0, 7.6 Hz, 1H), 3.77 (dd, J = 16.8, 14.0 Hz, 1H), 2.92 (dd, J = 16.8, 8.0 Hz, 1H), 2.87 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 174.20, 172.31, 144.28, 134.39, 134.35, 131.55, 129.70, 128.50, 127.83, 125.67, 124.35, 124.12, 123.68, 108.77, 86.05, 50.42, 32.17, 25.90. HRMS (ESI): Exact Mass Calcd. for C₁₈H₁₅ClNO₃ (M+H)⁺: 328.0735, Found:

328.0732. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, retention time: $t_{major} = 47.098 \text{ min}, t_{minor} = 52.457 \text{ min}, 94\%$ ee).



(2S, 3R)-3-(4-chlorophenyl)-1'-methyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3m)

white solid, mp 193 – 196 °C, $[\alpha]_{D}^{20}$ –133 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.53 (d, J = 7.2 Hz, 1H), 7.42 – 7.38 (m, 1H), 7.21 (t, J = 7.6 Hz, 1H), 7.14 (d, J = 8.8 Hz, 2H), 6.87 (d, J = 8.4 Hz, 2H), 6.70 (d, J = 8.0 Hz, 1H), 4.04 (dd, J = 13.6, 8.0 Hz, 1H), 3.76 (dd, J = 16.8, 14.0 Hz, 1H), 2.91 (dd, J = 16.8, 8.0 Hz, 1H), 2.86 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 174.31, 172.40, 144.27, 134.23, 131.46, 130.67, 128.96, 128.64, 124.36, 124.12, 123.60, 108.76, 86.11, 50.31, 32.27, 25.90. HRMS (ESI): Exact Mass Calcd. for C₁₈H₁₅CINO₃ (M+H)⁺: 328.0735, Found: 328.0730. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, retention time: t_{major} = 25.633 min, t_{minor} = 38.990 min, 94% ee).



(2S, 3R)-1'-methyl-3-(p-tolyl)-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3n)

white solid, mp 148 – 151 °C, $[\alpha]_{D}^{20}$ –143 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.52 (d, J = 7.2 Hz, 1H), 7.40 – 7.36 (m, 1H), 7.21 – 7.17 (m, 1H), 6.96 (d, J = 8.0 Hz, 2H), 6.81 (d, J = 8.4 Hz, 2H), 6.67 (d, J = 8.0 Hz, 1H), 4.04 (dd, J = 13.6, 8.0 Hz, 1H), 3.79 (dd, J = 16.8, 13.6 Hz, 1H), 2.89 (dd, J =16.8, 8.0 Hz, 1H), 2.84 (s, 3H), 2.24 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 174.89, 172.67, 144.36, 137.98, 131.19, 129.10, 128.99, 127.39, 124.84, 124.11, 123.41, 108.57, 86.41, 50.63, 32.36, 25.82, 20.97. HRMS (ESI): Exact Mass Calcd. for C₁₉H₁₈NO₃ (M+H)⁺: 308.1281, Found: 308.1288. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, retention time: t_{major} = 8.964 min, t_{minor} = 12.685 min, 97% ee).



(2S, 3R)-3-(4-methoxyphenyl)-1'-methyl-3,4-dihydro-5H-spiro[furan-2,3'-indoline]-2',5-dione (3o)

white solid, mp 172 – 174 °C, $[\alpha]_{D}^{20}$ –101 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.52 (d, J = 7.6 Hz, 1H), 7.40 – 7.36 (m, 1H), 7.19 (t, J = 7.6 Hz, 1H), 6.85 (d, J = 8.8 Hz, 2H), 6.69 – 6.66 (m, 3H), 4.02 (dd, J = 14.0, 8.0 Hz, 1H), 3.80 – 3.72 (m, 4H), 2.91 – 2.85 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 174.86, 172.71, 159.39, 144.34, 131.20, 128.68, 124.81, 124.09, 123.92, 123.42, 113.77, 108.57, 86.44, 55.15, 50.36, 32.46, 25.85. HRMS (ESI): Exact Mass Calcd. for C₁₉H₁₈NO₄ (M+H)⁺: 324.1230, Found: 324.1233. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, retention time: t_{major} = 13.343 min, t_{minor} = 18.061 min, 95% ee).



(2R, 3R)-1',3-dimethyl-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione (3p)

Off-white solid, $[\alpha]_{D}^{20}$ +107 (c 0.31, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.42 (td, J = 8.0, 1.2 Hz, 1H), 7.35 (d, J = 7.6 Hz, 1H), 7.14 (td, J = 7.6, 0.4 Hz, 1H), 6.86 (d, J = 8.0 Hz, 1H), 3.20 (s, 3H), 3.03 (dd, J = 16.4, 12.8 Hz, 1H), 2.94 – 2.84 (m, 1H), 2.72 (dd, J = 16.4, 8.0 Hz, 1H), 1.00 (d, J = 6.4 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 175.40, 173.05, 144.57, 131.15, 124.84, 124.22, 123.49, 108.60, 86.07, 40.26, 34.98, 26.18, 12.58. HRMS (ESI): Exact Mass Calcd. for C₁₃H₁₃NO₃Na (M+Na)⁺: 254.0788, Found: 254.0791. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, retention time: t_{minor} = 19.954 min, t_{major} = 22.269 min, 95% ee).

4. Characterization of compounds 5a-5i and 7a-7b



(R)-5-acetyl-4,6-diphenyl-3,4-dihydro-2H-pyran-2-one (5a)

white solid, mp 136 – 140 °C, $[\alpha]_{D}^{20}$ –176 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.62 (d, J = 8.4 Hz, 2H), 7.54 – 7.50 (m, 1H), 7.39 (t, J = 7.6 Hz, 2H), 7.29 – 7.20 (m, 3H), 7.14 (d, J = 8.4 Hz, 2H), 4.32 (dd, J = 7.2, 3.2 Hz, 1H), 3.07 (dd, J = 16.0, 7.6 Hz, 1H), 2.93 (dd, J = 16.0, 3.6 Hz, 1H), 1.90 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 195.78, 166.49, 154.74, 139.97, 138.42, 133.06, 129.13, 128.78, 128.71, 127.62, 126.76, 117.75, 39.41, 36.19, 18.99. HRMS (ESI): Exact Mass Calcd. for C₁₉H₁₇O₃ (M+H)⁺: 293.1172, Found: 293.1181. HPLC (Chiralpak OD–H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: t_{minor} = 10.650 min, t_{major} = 12.607 min, 95% ee).



ethyl (R)-2-oxo-4,6-diphenyl-3,4-dihydro-2H-pyran-5-carboxylate (5b)

white solid, mp 131– 134 °C, $[\alpha]_{D}^{20}$ –134 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.52 – 7.50 (m, 2H), 7.47 – 7.39 (m, 3H), 7.36 – 7.32 (m, 2H), 7.29 – 7.28 (m, 2H), 7.27 – 7.25 (m, 1H), 4.41 (dd, *J* = 7.6 , 2.4 Hz ,1H), 3.93 (q, *J* = 2.8 Hz, 2H), 3.10 (dd, *J* = 16.0, 7.6 Hz ,1H), 2.94 (dd, *J* = 16.0, 2.4 Hz ,1H), 0.88 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 166.35, 165.96, 158.50, 139.90, 133.11, 130.11, 129.19, 128.63, 127.97, 127.73, 126.75, 111.71, 60.98, 38.87, 36.30, 13.43. HRMS (ESI): Exact Mass Calcd. for C₂₀H₁₉O₄ (M+H)⁺: 323.1278, Found: 323.1273. HPLC (Chiralpak OD–H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: t_{minor} = 12.276 min, t_{major} = 13.809 min, 91% ee).



ethyl (R)-6-methyl-2-oxo-4-phenyl-3,4-dihydro-2H-pyran-5-carboxylate (5c)

brown solid, mp 111 – 114 °C, $[\alpha]_{D}^{20}$ –170 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.31 – 7.28 (m, 2H), 7.25 – 7.21 (m, 1H), 7.14 (d, *J* = 7.2 Hz, 2H), 4.26 (d, *J* = 7.2 Hz, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 2.95 (dd, *J* = 15.6, 7.6 Hz, 1H), 2.82 (dd, *J* = 15.6, 2.4 Hz, 1H), 2.47 (s, 3H), 1.19 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 166.09, 165.93, 161.27, 140.63, 128.99, 127.46, 126.57, 110.00, 60.82, 37.82, 36.34, 18.84, 14.02. HRMS (ESI): Exact Mass Calcd. for C₁₅H₁₇O₄ (M+H)⁺: 261.1121, Found: 261.1127. HPLC (Chiralpak OD–H column, *n*-hexane/*i*-PrOH = 80/20), flow rate = 1.0 mL/min, retention time: t_{minor} = 9.105 min, t_{major} = 16.704 min, 95% ee).



methyl (R)-6-methyl-2-oxo-4-phenyl-3,4-dihydro-2H-pyran-5-carboxylate (5d)

white solid, mp 99 – 102 °C, $[\alpha]_{D}^{20}$ –183 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.32 – 7.22 (m, 3H), 7.13 (d, *J* = 7.2 Hz, 2H), 4.26 (d, *J* = 7.2 Hz, 1H), 3.68 (s, 3H), 2.94 (dd, *J* = 16.0, 7.6 Hz, 1H), 2.83 (dd, *J* = 16.0, 2.4 Hz, 1H), 2.48 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 166.42, 165.98, 161.72, 140.39, 129.06, 127.54, 126.55, 109.71, 51.89, 37.73, 36.44, 18.90. HRMS (ESI): Exact Mass Calcd. for C₁₄H₁₅O₄

 $(M+H)^+$: 247.0965, Found: 247.0972. HPLC (Chiralpak OD-H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: $t_{minor} = 7.831 \text{ min}, t_{major} = 16.611 \text{ min}, 91\%$ ee).



(R)-5-benzoyl-4,6-diphenyl-3,4-dihydro-2H-pyran-2-one (5e)

brown solid, mp 201 – 203 °C, $[\alpha]_D^{20}$ –86 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.49 (d, *J* = 7.2 Hz, 2H), 7.37 (d, *J* = 6.8 Hz, 2H), 7.30 – 7.21 (m, 6H), 7.18 – 7.06 (m, 5H), 4.56 (dd, *J* = 7.6, 2.4 Hz, 1H), 3.21 (dd, *J* = 16.0, 7.6 Hz, 1H), 3.06 (dd, *J* =16.0, 2.4 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 195.91, 166.65, 154.79, 139.70, 137.08, 132.57, 131.97, 130.27, 129.22, 128.98, 128.04, 127.98, 127.77, 126.84, 118.28, 40.38, 35.93. HRMS (ESI): Exact Mass Calcd. for C₂₄H₁₉O₃ (M+H)⁺: 355.1329, Found: 355.1334. HPLC (Chiralpak OD–H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: t_{minor} = 19.703 min, t_{major} = 28.502 min, 91% ee).



(R)-5-acetyl-6-(3-bromophenyl)-4-phenyl-3,4-dihydro-2H-pyran-2-one (5f)

brown solid, mp 131 – 134 °C, $[\alpha]_{D}^{20}$ –98 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.53 (d, J = 8.4 Hz, 2H), 7.48 (d, J = 8.4 Hz, 2H), 7.28 – 7.21 (m, 3H), 7.11 (d, J = 8.4 Hz, 2H), 4.29 (dd, J = 7.2, 3.6 Hz, 1H), 3.06 (dd, J = 16.0, 7.2 Hz, 1H), 2.93 (dd, J = 16.0, 4.0 Hz, 1H), 1.91 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 194.37, 166.16, 155.52, 140.22, 139.76, 135.82, 131.56, 130.25, 129.24, 127.78, 127.22, 126.73, 122.97, 117.46, 39.47, 36.19, 19.09. HRMS (ESI): Exact Mass Calcd. for C₁₉H₁₆BrO₃ (M+H)⁺: 372.0277, Found: 372.0281. HPLC (Chiralpak OD–H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: t_{minor} = 15.840 min, t_{major} = 19.112 min, 87% ee).



(R)-5-acetyl-6-(4-bromophenyl)-4-phenyl-3,4-dihydro-2H-pyran-2-one (5g)

brown solid, mp 142 – 145 °C, $[\alpha]_{D}^{20}$ –109 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.53 (q, J = 8.8 Hz, 2H),, 7.48 (q, J = 8.4 Hz, 2H), 7.28 – 7.20 (m, 3H), 7.11 (d, J = 8.4 Hz, 2H), 4.28 (dd, J = 7.2, 3.6 Hz, 1H), 3.06 (dd, J = 16.0, 7.2 Hz, 1H), 2.93 (dd, J = 16.0, 4.0 Hz, 1H), 1.91 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 194.72, 166.25, 154.81, 139.84, 137.10, 132.07, 130.25, 129.21, 128.29, 127.74, 126.69, 117.46, 39.46, 36.10, 19.06. HRMS (ESI): Exact Mass Calcd. for C₁₉H₁₆BrO₃ (M+H)⁺: 372.0277, Found: 372.0274. HPLC (Chiralpak OD–H column, *n*-hexane/*i*-PrOH = 80/20), flow rate = 1.0 mL/min, retention time: t_{minor} = 16.105 min, t_{major} = 21.732 min, 87% ee).



(R)-5-acetyl-6-methyl-4-phenyl-3,4-dihydro-2H-pyran-2-one (5h)

white solid, mp 109 – 112 °C, $[\alpha]_{D}^{20}$ –182 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.36 – 7.32 (m, 2H), 7.29 – 7.28 (m, 1H), 7.15 (d, *J* = 7.2 Hz, 2H), 4.15 (d, *J* = 6.0 Hz, 1H), 2.97 (dd, *J* =15.6, 7.2 Hz, 1H), 2.84 (dd, *J* =15.6, 2.4 Hz, 1H), 2.43 (s, 3H), 2.12 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 197.88, 165.56, 160.23, 139.70, 129.43, 127.94, 126.65, 117.28, 38.86, 37.16, 29.75, 19.06. HRMS (ESI): Exact Mass Calcd. for C₁₄H₁₅O₃ (M+H)⁺: 231.1016, Found: 231.1019. HPLC (Chiralpak OD–H column, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, retention time: t_{minor} = 10.954 min, t_{major} = 13.941 min, 88% ee).

ethyl (R)-4-methyl-2-oxo-6-phenyl-3,4-dihydro-2H-pyran-5-carboxylate (5i)

colorless oil, $[\alpha]_{D}^{20}$ -101 (c 1.0, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.44 – 7.35 (m, 5H), 4.05 (q, J = 7.2 Hz, 2H), 3.24 – 3.17 (m, 1H), 2.85 (dd, J = 15.6, 6.8 Hz, 1H), 2.66 (dd, J = 15.6, 2.4 Hz, 1H), 1.25 (d, J = 6.8 Hz, 3H), 0.99 (t, J = 6.8 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 166.92, 166.66, 157.35, 133.19, 129.88, 128.50, 113.91, 60.88, 35.55, 28.25, 19.17, 13.56. HRMS (ESI): Exact Mass Calcd. for C₁₅H₁₇O₄ (M+H)⁺: 261.1121, Found: 261.1123. HPLC (Chiralpak OD–H column, *n*-hexane/*i*-PrOH = 95/5, flow rate = 1.0 mL/min, retention time: t_{minor} = 11.237 min, t_{major} = 14.681 min, 83% ee).



ethyl (R)-6-oxo-2,4-diphenyl-1,4,5,6-tetrahydropyridine-3-carboxylate (7a)

white solid, mp 122 – 124 °C, $[\alpha]_{D}^{20}$ –144 (c 1.0, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 7.42 – 7.30 (m, 9H), 7.25 – 7.23 (m, 1H), 7.19 (br s, 1H), 4.34 (dd, *J* = 8.0, 2.4 Hz, 1H), 3.86 (q, *J* = 7.2 Hz, 2H), 3.06 (dd, *J* = 16.4, 8.0 Hz, 1H), 2.80 (dd, *J* = 16.4, 2.4 Hz, 1H), 0.83 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 169.53, 166.58, 146.50, 141.71, 135.76, 129.51, 128.87, 128.44, 127.82, 127.11, 126.73, 108.73, 60.16, 38.59, 38.01, 13.49. HRMS (ESI): Exact Mass Calcd. for C₂₀H₂₀NO₃ (M+H)⁺: 322.1438, Found: 322.3844. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 80/20), flow rate = 1.0 mL/min, retention time: t_{major} = 15.822 min, t_{minor} = 29.715 min, 86% ee).



ethyl (R)-4-methyl-6-oxo-2-phenyl-1,4,5,6-tetrahydropyridine-3-carboxylate (7b)

white solid, mp 141 – 144 °C, $[\alpha]_{D}^{20}$ –128 (c 1.0, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.41 – 7.37 (m, 3H), 7.30 – 7.28 (m, 2H), 7.06 (br s, 1H), 3.95 (q, *J* = 6.8 Hz, 2H), 3.18 – 3.10 (m, 1H), 2.79 (dd, *J* = 16.4, 6.8 Hz, 1H), 2.43 (dd, *J* = 16.4, 0.8 Hz, 1H), 1.25 (d, *J* = 6.8 Hz, 3H), 0.91 (t, *J* = 6.8 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 170.16, 166.90, 144.83, 135.86, 129.35, 128.38, 127.77, 111.06, 60.03, 37.56, 28.48, 18.72, 13.60. HRMS (ESI): Exact Mass Calcd. for C₁₅H₁₇NO₃Na (M+Na)⁺: 282.1101, Found: 282.1102. HPLC (Chiralpak AD–H column, *n*-hexane/*i*-PrOH = 95/5, flow rate = 1.0 mL/min, retention time: t_{minor} = 13.041 min, t_{major} = 11.553 min, 75% ee).

5. X-Ray crystal structure of compound 3b



6. NMR spectra of compounds 3a–3f, 3h–3p, 5a–5i and 7a–7b

¹H NMR spectrum of compound **3a** (CDCl₃, 400 MHz)







¹H NMR spectrum of compound **3c** (CDCl₃, 400 MHz)









¹H NMR spectrum of compound **3f** (CDCl₃, 400 MHz)







¹H NMR spectrum of compound **3i** (CDCl₃, 400 MHz)



¹H NMR spectrum of compound **3j** (CDCl₃, 400 MHz)



¹H NMR spectrum of compound **3k** (CDCl₃, 400 MHz)



¹H NMR spectrum of compound **3l** (CDCl₃, 400 MHz)



¹H NMR spectrum of compound **3m** (CDCl₃, 400 MHz)



¹H NMR spectrum of compound **3n** (CDCl₃, 400 MHz)



¹H NMR spectrum of compound **30** (CDCl₃, 400 MHz)



¹H NMR spectrum of compound **3p**











¹H NMR spectrum of compound **5c** (CDCl₃, 400 MHz)















¹H NMR spectrum of compound **5i** (CDCl₃, 400 MHz)



¹H NMR spectrum of compound 7a (CDCl₃, 400 MHz)





7. HPLC spectra for compounds 3a–3f, 3h–3p, 5a–5i and 7a–7b

















2567.60522

33.54792

3.64

24.745

2

PDA 230.16 nm









Peak	Processed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 254 nm	15.882	3081.38013	92.98299	49.96
2	PDA 254 nm	17.913	3086.50464	82.17327	50.04
			0		









S49





Peak	Processed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 280 nm	46.725	3.16768e4	219.19832	49.92
2	PDA 280 nm	52.079	3.17813e4	212.05476	50.08









S53







Pea	rocessed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 254 nm	20.150	2371.76123	60.13734	50.52
2	PDA 254 nm	22.430	2322.74219	51.78837	49.48











Peak	Processed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 280 nm	12.210	3.72243e4	817.08600	50.08
2	PDA 280 nm	13.897	3.71014e4	894.90759	49.92







Peak	Processed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 230 nm	8.901	2.76678e4	1083.27173	48.95
2	PDA 230 nm	16.394	2.88580e4	740.59827	51.05









Peak	Processed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 254 nm	20.187	2.25771 e4	297.19433	50.03
2	PDA 254 nm	28.813	2.25511 e4	228.92552	49.97









Peak	Processed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 254 nm	15.335	3.65230e4	726.63184	52.6592
2	PDA 254 nm	18.775	3.28342e4	614.07043	47.3408







Peak	Processed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 254 nm	16.031	3.13770e4	625.71527	49.82
2	PDA 254 nm	22.197	3.16042e4	449.75967	50.18
			<u> </u>		









Peak	Processed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 254 nm	10.766	1.85695e4	617.98260	49.23
2	PDA 254 nm	13.971	1.91513e4	535.67426	50.77







Peak	Processed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 254 nm	11.253	4749.16016	227.04272	49.10
2	PDA 254 nm	14.649	4923.09961	178.28705	50.90







Peak	Processed	Retention	Peak area	Peak height	Peak area
	channel	time (min)	(mAU*s)	(mAU)	(%)
1	PDA 254 nm	16.999	2.35951e4	360.10287	51.26
2	PDA 254 nm	31.294	2.24315e4	195.12534	48.74







Ph	CO ₂ Et
HN	
0	7b

13.117

8032.83691

215.71977

50.44

2

PDA 254 nm

