Supporting Information

Enantioselective Pd-Catalyzed Dearomative Reductive Heck and Domino Heck-Suzuki Reactions of 2-CF₃-Indoles

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1. General information

Reactions and manipulations involving organometallic or moisture sensitive compounds were carried out under nitrogen atmosphere and glassware was dried by heating gun for 15 min prior to use. ¹H NMR, ¹³C NMR and ¹⁹F NMR spectra were recorded on Bruker AVANCE III 400 MHz or 600 MHz using CDCl₃ as solvent and TMS as internal standard. Anhydrous 1,4-dioxane and toluene were freshly distilled over Na and benzophenone. Anhydrous DCM, Et₃N, and DMF were freshly distilled over CaH₂. Melting points were measured on a Büchi Melting Point B-545 apparatus and uncorrected. Commercial reagents were used as received without further purification unless otherwise noticed. HRMS were recorded on Thermo Scientific LTQ Orbitrap XL. Optical rotations were determined using a Rudolph Autopol IV polarimeter. Chiral HPLC analyses were performed using Agilent 1260 chromatography. Column chromatography was carried out using silica gel (200-300 mesh). Ligands L₁-L₉ were purchased from commercial sources, and ligands L₁₀-L₁₇ were prepared according to the literature procedures and used directly as received.¹ ²

2. Synthesis of 2-CF₃-indole 1

2.1 For compounds 1a-1o

Step 1:³

To a three-neck bottom flask equipped with a condenser was charged with Ph₃P (7.86 g, 30 mmol), CCl₄ (40 mL), and CF₃COOH (10 mmol) at 0 °C under a nitrogen atmosphere. After stirring for 15 min, 2-aminobenzyl alcohol (10 mmol) and Et₃N (1.4 mL, 10 mmol) was then added respectively to the reaction mixture via a syringe. The resulting mixture was allowed to reflux for 2 h. When the reaction was completed, the solution was concentrated under reduced pressure. The residue was washed with EtOAc for three times, and the precipitate was removed by filtration. The filtrate was concentrated and purified by column chromatography on silica gel eluting with EtOAc/petroleum ether 1:50 (v/v) to afford compound S₁.

Step 2:

To a 100 mL flask was charged with benzoic acid derivative (977 mg, 8 mmol), anhydrous DCM (20 mL), and catalytic amount of DMF at 0 °C. The mixture was stirred for 30 minutes. Then, (COCl)₂ (0.89 mL, 1.3 equiv) was added dropwise via a syringe. The resulting mixture was allowed to stir at room temperature for 4 h. The solution was concentrated under reduced pressure to afford the crude acid chloride S₂.
which was used directly for the next step without further purification.

Step 3:
To a suspension of S1 (1.0 equiv), sodium hydroxide (2.5 equiv), and TBAB (20 mol%) in DCM (0.5 M with respect to S1) was added a solution of S2 (1.5 equiv, 2 M) in DCM dropwise at 0 °C. The reaction mixture was stirred at 0 °C for 30 minutes. After adding additional 0.5 equivalent of S2 in DCM (2 M) dropwise, the resulting mixture was allowed to stir at room temperature overnight. The solution was extracted with DCM and the combined organic layers were washed with brine, dried over Na₂SO₄, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel eluting with EtOAc/petroleum ether (1:100) to afford compounds 1a-1o.

2.2 For compounds 1p-1t

Step 1:\[4\]
To a flask equipped with a condenser was charged with Ph₃P (34.5 g, 132 mmol), Et$_3$N (7.3 mL, 53 mmol), CCl₄ (21.1 mL, 220 mmol), and CF₃CO$_2$H (44 mmol) at 0 °C under nitrogen atmosphere. After stirring for 10 min, a solution of o-methylaniline (44 mmol) in CCl₄ (21.1 mL, 220 mmol) was added dropwise to the reaction mixture. Upon completion of the addition, the reaction mixture was allowed to reflux for 3 h. The solution was then concentrated under reduced pressure and petroleum ether was then added. The precipitate was removed via filtration and the filtrate was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel eluting with EtOAc/petroleum ether (1:100) to afford compound S3.

Step 2:\[5\]
To a flask was charged with Pd(dba)$_2$ (11.6 mg, 0.02 mmol, 1.0 mol%), SIPrHCl (8.6 mg, 0.022 mmol, 1.1 mol%), anhydrous KOAc (392 mg, 4.0 mmol, 2.0 equiv) and K$_2$CO$_3$ (884 mg, 6.4 mmol, 3.2 equiv) under N₂ atmosphere. Toluene (2 mL) was introduced via a syringe. The resulting mixture was stirred at room temperature for 10 min, then a solution of S3 (2.0 mmol, 1.0 equiv) in 3 mL toluene were then added. After adding additional 3 mL of toluene, the reaction mixture was allowed to stir at 110 °C (oil bath) for 12 h. When the reaction was completed, the solution was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel eluting with EtOAc/petroleum (1:100) to afford compound S4.
Step 3:

To a suspension of S₄ (1.0 equiv), sodium hydroxide (2.5 equiv), and TBAB (20 mol%) in DCM (0.5 M with respect to S₄) was added a solution of 2-bromobenzoyl chloride (1.5 equiv, 2 M) in DCM dropwise at 0 °C. The reaction mixture was stirred at 0 °C for 30 minutes. After adding additional 0.5 equivalent of 2-bromobenzoyl chloride in DCM (2 M) dropwise, the resulting mixture was allowed to stir at room temperature overnight. The solution was extracted with DCM and the combined organic layers were washed with brine, dried over Na₂SO₄, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel eluting with EtOAc/petroleum ether (1:100) to afford compounds 1p-1t.

3. Preparation of sodium tetraarylborates

Sodium tetraarylborates were prepared according to the literature.[6] To a suspension of Mg powder (6.6 equiv) in THF (20 mL) was added a grain of iodine, then a solution of aryl bromides (6.0 equiv) in THF (20 mL) was slowly added. The resulting mixture was stirred at room temperature for 3 h, giving a dark gray solution of the aryl Grignard reagent. Upon addition of NaBF₄ (1.0 equiv), the heterogeneous reaction mixture was stirred for additional 48-72 h. The reaction mixture was then poured into a solution of Na₂CO₃ in water and stirred for 20 min. After filtering, the filtrate was extracted with ethyl acetate, dried over Na₂SO₄, and concentrated under reduced pressure. The residue was recrystallized from petroleum ether to afford sodium tetraarylborates.

(2-Bromophenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1a):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid, 95% yield (for the last step); m.p. 74-76 °C. ¹H NMR (600 MHz, CDCl₃) δ 7.69 (dd, J = 7.8, 1.3 Hz, 1H), 7.64 (d, J = 7.8 Hz, 1H), 7.57 (dd, J = 7.4, 1.9 Hz, 1H), 7.54-7.43 (m, 2H), 7.31 (s, 1H), 7.29-7.22 (m, 1H), 7.18-7.08 (m, 1H), 6.44 (d, J = 8.6 Hz, 1H). ¹³C NMR (150 MHz, CDCl₃) δ 165.5, 137.0, 136.6, 133.9, 133.0, 130.3, 128.1, 127.8, 127.2, 126.8, 123.9, 122.6, 120.7 (q, J = 267.0 Hz), 137.8, 115.1 (q, J = 4.5 Hz), 113.9. ¹⁹F NMR (377 MHz, CDCl₃) δ -58.4 ppm. HRMS m/z (ESI+): Calcd for C₁₆H₉BrF₃NONa⁺ (M+Na)⁺ 389.9716, found 389.9717.
(2-Bromo-4-methoxyphenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1b):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 85% yield (for the last step); m.p. 81-83 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.66 (d, $J = 7.7$ Hz, 1H), 7.51 (d, $J = 8.6$ Hz, 1H), 7.28 (s, 1H), 7.23 (d, $J = 7.3$ Hz, 1H), 7.24-7.18 (m, 2H), 7.01 (dd, $J = 8.6$, 2.5 Hz, 1H), 6.58 (dd, $J = 8.5$, 1.3 Hz, 1H), 3.90 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 165.6, 162.8, 137.2, 132.3, 128.3, 128.2, 127.8, 127.1, 126.6, 123.6, 122.5 (d, $J = 5.0$ Hz), 120.7 (q, $J = 267.0$ Hz), 122.0, 119.4 (d, $J = 2.8$ Hz), 114.2 (q, $J = 5.0$ Hz), 113.9 (d, $J = 1.6$ Hz), 55.9. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.1 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{17}$H$_{11}$BrF$_3$NO$_2$Na$^+$ (M+Na)$^+$ 419.9817, found 419.9815.
(2-Bromo-4-methylphenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1c):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 88% yield (for the last step); m.p. 87-89 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.64 (d, $J$ = 7.9 Hz, 1H), 7.54 (d, $J$ = 8.9 Hz, 1H), 7.31 (s, 1H), 7.26 (d, $J$ = 5.9 Hz, 1H), 7.19 (t, $J$ = 7.9 Hz, 1H), 7.13 (d, $J$ = 3.0 Hz, 1H), 7.03 (dd, $J$ = 8.9, 3.1 Hz, 1H), 6.47 (d, $J$ = 8.6 Hz, 1H), 3.84 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 165.7, 144.2, 137.1, 134.5, 133.6, 130.4, 128.8, 127.6 (q, $J$ = 40.0 Hz), 127.2, 126.7, 123.7, 122.5, 120.71, 120.67 (q, $J$ = 267.0 Hz), 114.6 (q, $J$ = 5.0 Hz), 113.9, 21.3. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.2 ppm. HRMS m/z (ESI+): Calcd for C$_{17}$H$_{11}$BrF$_3$NONa$^+$ (M+Na)$^+$ 403.9868, found 403.9870.
(2-Bromo-4-chlorophenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1d):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 77% yield (for the last step); m.p. 94-96 °C. \( ^1 \)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.71 (s, 1H), 7.66 (d, \( J = 7.9 \) Hz, 1H), 7.53-7.48 (m, 2H), 7.30 (t, \( J = 7.4 \) Hz, 2H), 7.21 (t, \( J = 8.4 \) Hz, 1H), 6.58 (d, \( J = 8.4 \) Hz, 1H). \( ^{13} \)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 164.8, 138.7, 136.9, 134.9, 133.8, 131.3, 128.6, 128.3 (q, \( J = 40.0 \) Hz), 127.2, 127.0, 124.1, 122.8, 121.5, 120.6 (q, \( J = 267.0 \) Hz), 115.2 (q, \( J = 5.0 \) Hz), 113.8. \( ^{19} \)F NMR (377 MHz, CDCl\(_3\)) \( \delta \) -58.2 ppm. HRMS \( m/z \) (ESI+): Calcd for C\(_{16}\)H\(_8\)BrClF\(_3\)NONa\(^+\) (M+Na\(^+\)) 423.93221, found 423.9325.
(2-Bromo-4-fluorophenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1e):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 79% yield (for the last step); m.p. 98-100 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.66 (d, $J = 7.8$ Hz, 1H), 7.60 (dd, $J = 8.6$, 5.7 Hz, 1H), 7.44 (dd, $J = 8.1$, 2.4 Hz, 1H), 7.30 (d, $J = 13.1$ Hz, 2H), 7.23-7.18 (m, 2H), 6.57 (d, $J = 8.6$ Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.9, 164.1 (d, $J = 257.0$ Hz), 137.0, 132.8 (d, $J = 4.0$ Hz), 132.3 (d, $J = 9.0$ Hz), 127.2, 127.0, 124.1, 122.8, 122.0 (d, $J = 11.0$ Hz), 121.7, 121.4, 120.5 (q, $J = 267.0$ Hz), 115.8 (d, $J = 22.0$ Hz), 115.6 (q, $J = 5.0$ Hz), 113.8. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.2, -104.2 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{16}$H$_8$BrF$_4$NONa$^+$ (M+Na)$^+$ 419.9818, found 419.9815.
(2-Bromo-4-trifluoromethylphenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1f):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 62% yield (for the last step); m.p. 95-97 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.96 (s, 1H), 7.78 (d, $J$ = 8.0 Hz, 1H), 7.70 (d, $J$ = 8.0 Hz, 1H), 7.67 (d, $J$ = 7.8 Hz, 1H), 7.34 (s, 1H), 7.29 (t, $J$ = 7.5 Hz, 1H), 7.21 (t, $J$ = 7.9 Hz, 1H), 6.58 (d, $J$ = 8.6 Hz, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 164.4, 140.0, 136.9, 134.7 (q, $J$ = 34.5 Hz), 130.8 (q, $J$ = 4.5 Hz), 130.6, 127.8 (q, $J$ = 40.5 Hz), 127.3, 127.2, 125.1 (q, $J$ = 3.0 Hz), 124.4, 122.9, 122.5 (q, $J$ = 272.0 Hz), 121.1, 120.5 (q, $J$ = 267.0 Hz), 115.8 (q, $J$ = 4.5 Hz), 113.8. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.3, -63.1 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{17}$H$_8$BrF$_9$NONa$^+$ (M+Na)$^+$ 457.9586, found 457.9586.
(2-Bromo-5-methoxyphenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1g):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 81% yield (for the last step); m.p. 106-108 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.64 (d, $J = 7.8$ Hz, 1H), 7.55 (d, $J = 8.2$ Hz, 1H), 7.38 (s, 1H), 7.30-7.28 (m, 2H), 7.24 (d, $J = 7.2$ Hz, 1H), 7.14 (t, $J = 7.2$ Hz, 1H), 6.46 (d, $J = 8.0$ Hz, 1H), 2.39 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 165.7, 138.5, 137.0, 136.3, 133.8 (d, $J = 28.7$ Hz), 130.6, 128.0 (q, $J = 40.0$ Hz), 127.3, 126.8, 123.9, 122.6, 120.7 (q, $J = 267.0$ Hz), 119.4, 117.2, 115.0 (q, $J = 5.0$ Hz), 113.9, 20.9. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.4 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{17}$H$_{11}$BrF$_3$NO$_2$Na$^+$ (M+Na)$^+$ 419.9818, found 419.9818.
(2-Bromo-5-methylphenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1h):
Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 90% yield (for the last step); m.p. 89-91 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.64 (d, $J$ = 7.8 Hz, 1H), 7.55 (d, $J$ = 8.2 Hz, 1H), 7.38 (s, 1H), 7.30-7.28 (m, 2H), 7.24 (d, $J$ = 7.6 Hz, 1H), 7.14 (t, $J$ = 7.9 Hz, 1H), 6.46 (d, $J$ = 8.8 Hz, 1H), 2.39 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 165.7, 138.5, 137.0, 136.3, 133.9, 133.7, 130.7, 128.0 (q, $J$ = 40.0 Hz), 127.3, 126.8, 123.9, 122.6, 120.7 (q, $J$ = 267.0 Hz), 117.2, 115.0 (q, $J$ = 5.0 Hz), 113.9, 20.9. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.4 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{17}$H$_{11}$BrF$_3$NONa$^+$ (M+Na)$^+$ 403.9868, found 403.9870.
(2-Bromo-5-chlorophenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1i):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 79% yield (for the last step); m.p. 105-107 °C. \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 7.66 (d, \(J = 7.8\) Hz, 1H), 7.62 (d, \(J = 8.6\) Hz, 1H), 7.55 (d, \(J = 2.5\) Hz, 1H), 7.45 (dd, \(J = 8.6, 2.5\) Hz, 1H), 7.32 (s, 1H), 7.28 (t, \(J = 7.5\) Hz, 1H), 7.21 (t, \(J = 7.9\) Hz, 1H), 6.59 (d, \(J = 8.6\) Hz, 1H). \(^1\)^C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 164.2, 137.9, 136.9, 135.0, 134.4, 133.0, 130.1, 127.9 (q, \(J = 40.5\) Hz), 127.3, 127.1, 124.2, 122.8, 120.5 (q, \(J = 267.0\) Hz) 118.6, 115.5 (q, \(J = 4.5\) Hz), 113.8. \(^1\)^F NMR (377 MHz, CDCl\(_3\)) \(\delta\) -58.3 ppm. HRMS \(m/z\) (ESI+): Calcd for C\(_{16}\)H\(_8\)BrClF\(_3\)NONa\(^+\) (M+Na\(^+\)) 423.9322, found 423.9321.
(2-Bromo-5-fluorophenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1j):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 70% yield (for the last step); m.p. 53-55 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.67-7.64 (m, 2H), 7.32-7.30 (m, 2H), 7.28 (t, $J = 7.5$ Hz, 1H), 7.23-7.18 (m, 2H), 6.56 (d, $J = 8.6$ Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.3, 161.9 (d, $J = 250.0$ Hz), 137.9 (d, $J = 7.0$ Hz), 136.8, 135.6 (d, $J = 8.0$ Hz), 127.9 (d, $J = 39.0$ Hz), 127.3, 127.1, 124.2, 122.8, 120.5 (q, $J = 267.0$ Hz), 120.4 (d, $J = 22.0$ Hz), 117.6 (d, $J = 24.0$ Hz), 115.6 (q, $J = 5.0$ Hz), 115.0 (d, $J = 3.0$ Hz), 113.8. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.4, -111.9 ppm. HRMS m/z (ESI+): Calcd for C$_{16}$H$_8$BrF$_4$NONa$^+$ (M+Na)$^+$ 407.9618, found 407.9621.
(2-Bromo-6-fluorophenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1k):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 64% yield (for the last step); m.p. 94-96 °C. 1H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.67-7.65 (m, 1H), 7.51 (dd, \(J = 8.2, 1.0\) Hz, 1H), 7.45 (td, \(J = 8.1, 5.6\) Hz, 1H), 7.32-7.27 (m, 2H), 7.24-7.19 (m, 2H), 6.80 (d, \(J = 7.3\) Hz, 1H). 13C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 160.8, 159.9 (d, \(J = 255.0\) Hz), 136.8, 133.4 (d, \(J = 9.0\) Hz), 129.2 (d, \(J = 4.0\) Hz), 127.9, 127.4 (d, \(J = 4.0\) Hz), 126.3, 126.1, 124.4, 122.8, 121.1 (d, \(J = 3.0\) Hz), 120.5 (q, \(J = 267.0\) Hz), 116.5 (q, \(J = 5.0\) Hz), 115.5 (d, \(J = 21.0\) Hz), 113.7. 19F NMR (377 MHz, CDCl\(_3\)) \(\delta\) -58.5, -110.9 ppm. HRMS \textit{m/z} (ESI+): Calcd for C\(_{16}\)H\(_8\)BrF\(_4\)NONa\(^+\) (M+Na\(^+\)) 407.9618, found 407.9621.
(2-Bromo-6-chlorophenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1l):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 66% yield (for the last step); m.p. 77-79 °C. $^1$H NMR (600 MHz, CDCl$_3$) δ 7.66 (d, $J = 8.2$ Hz, 2H), 7.51 (d, $J = 6.9$ Hz, 1H), 7.38 (d, $J = 29.1$ Hz, 2H), 7.27 (d, $J = 14.0$ Hz, 1H), 7.14 (s, 1H), 6.20 (s, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) δ 161.8, 159.0, 136.7, 136.10, 132.5 (d, $J = 4.5$ Hz), 131.9, 131.2, 129.3, 128.6, 127.7 (d, $J = 4.0$ Hz), 124.3, 122.9, 120.7 (q, $J = 267$ Hz), 116.4, 115.0 (q, $J = 4.5$ Hz), 112.9. $^{19}$F NMR (377 MHz, CDCl$_3$) δ -59.2 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{16}$H$_8$BrClF$_3$NONa$^+$ (M+Na)$^+$ 423.9322, found 423.9321.
(2-Bromo-4,5-dimethoxyphenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1m): Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 75% yield (for the last step); m.p. 115-117 ºC. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.64 (d, $J = 7.8$ Hz, 1H), 7.29 (s, 1H), 7.24 (dd, $J = 7.8$, 0.9 Hz, 1H), 7.17 (t, $J = 7.4$ Hz, 1H), 7.08 (d, $J = 4.8$ Hz, 2H), 6.57 (d, $J = 8.5$ Hz, 1H), 3.97 (s, 3H), 3.87 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 165.6, 152.4, 149.0, 137.1, 128.0 (q, $J = 40.0$ Hz), 127.98, 127.2, 126.7, 123.7, 122.5, 120.7 (q, $J = 267.0$ Hz), 116.3, 114.4 (q, $J = 5.0$ Hz), 113.8, 113.0, 112.7, 56.5, 56.4. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.2 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{18}$H$_{14}$BrF$_3$NO$_3^+$ (M+H)$^+$ 428.0104, found 428.0108.
(2-Bromo-4,5-difluorophenyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1n):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 59% yield (for the last step); m.p. 98-100 °C. \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 7.67 (d, \(J = 7.8\) Hz, 1H), 7.53 (t, \(J = 8.0\) Hz, 1H), 7.45 (t, \(J = 8.5\) Hz, 1H), 7.30 (t, \(J = 7.8\) Hz, 2H), 7.26-7.23 (m, 1H), 6.68 (d, \(J = 8.5\) Hz, 1H). \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 163.7, 152.1 (dd, \(J = 259.5, 12.0\) Hz), 149.9 (dd, \(J = 265.5, 12.0\) Hz), 136.9, 133.0 (d, \(J = 4.5\) Hz), 127.8 (q, \(J = 39.0\) Hz), 127.3, 127.2, 124.3, 123.3, 123.1, 122.9, 120.5 (q, \(J = 267.0\) Hz), 119.5 (d, \(J = 21.0\) Hz), 115.5 (q, \(J = 4.5\) Hz), 113.7. \(^{19}\)F NMR (377 MHz, CDCl\(_3\)) \(\delta\) -58.2, -127.9, -135.3 ppm. HRMS \(m/\ell\) (ESI+): calcd for C\(_{16}\)H\(_7\)BrF\(_5\)NONa\(^+\) (M+Na\(^+) 425.9523, found 425.9522.
(1-Bromonaphthyl)(2-(trifluoromethyl)-1H-indol-1-yl)methanone (1o):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); reddish brown liquid 49% yield (for the last step). $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 8.30 (s, 1H), 7.92 (d, $J$ = 29.7 Hz, 2H), 7.65-7.52 (m, 4H), 7.32 (s, 1H), 7.17 (s, 1H), 6.98 (s, 1H), 6.35 (t, $J$ = 6.0 Hz, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 166.1, 136.9, 135.2, 134.5, 132.0, 129.1, 128.8, 128.7, 128.6, 128.2 (q, $J$ = 39.0 Hz), 128.1, 127.3, 127.0, 125.0, 124.0, 122.6, 122.0, 120.8 (q, $J$ = 266.0 Hz), 115.4 (q, $J$ = 4.5 Hz), 114.0. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.0 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{20}$H$_{11}$BrF$_3$NONa$^+$ (M+Na)$^+$ 439.9868, found 439.9871.
(2-Bromophenyl)(6-methoxy-2-(trifluoromethyl)-1H-indol-1-yl)methanone (1p):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); reddish brown solid 45% yield (for the last step); m.p. 116-118 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.71 (d, $J = 7.8$ Hz, 1H), 7.59 (dd, $J = 7.5$, 1.9 Hz, 1H), 7.55-7.46 (m, 3H), 7.23 (s, 1H), 6.87 (dd, $J = 8.7$, 2.2 Hz, 1H), 5.97 (d, $J = 2.1$ Hz, 1H), 3.48 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 165.5, 159.3, 138.2, 136.9, 133.8, 132.8, 130.1, 128.1, 126.7, 123.1, 121.0, 120.7 (q, $J = 267.7$ Hz), 120.69, 115.2 (q, $J = 5.0$ Hz), 113.6, 98.0, 55.1. $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -58.0 ppm. Calcd for C$_{17}$H$_{11}$BrF$_3$NO$_2$Na+ (M+Na)$^+$ 419.9818, found 419.9818.
(2-Bromophenyl)(6-fluoro-2-(trifluoromethyl)-1H-indol-1-yl)methanone (1q):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); light yellow solid 43% yield (for the last step); m.p. 79-81 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.70 (d, $J$ = 7.7 Hz, 1H), 7.60-7.57 (m, 2H), 7.55-7.49 (m, 2H), 7.27 (s, 1H), 7.01 (t, $J$ = 8.4 Hz, 1H), 6.19 (d, $J$ = 10.5 Hz, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 165.4, 161.9 (d, $J$ = 244.5 Hz), 137.4 (d, $J$ = 12.0 Hz), 136.0, 134.0, 133.3, 130.3, 128.5, (d, $J$ = 4.5 Hz), 128.2, 123.7, 123.6, 120.6, 120.4 (q, $J$ = 267.0 Hz), 114.6 (q, $J$ = 4.5 Hz), 112.9 (d, $J$ = 24.0 Hz), 101.5 (d, $J$ = 30.0 Hz). $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.2, -112.0 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{16}$H$_8$BrF$_4$NONa$^+$ (M+Na)$^+$ 407.9618, found 407.9619.
(2-Bromophenyl)(6-chloro-2-(trifluoromethyl)-1H-indol-1-yl)methanone (1r):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); white solid 41% yield (for the last step); m.p. 74-76 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.71 (dd, \(J = 7.1, 1.9\) Hz, 1H), 7.61-7.51 (m, 4H), 7.26-7.23 (m, 2H), 6.48 (s, 1H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 165.4, 137.3, 135.9, 134.0, 133.4, 132.9, 130.4, 128.7, 128.3, 125.7, 124.8, 124.4, 122.9, 120.4 (q, \(J = 267.0\) Hz), 120.7, 114.5 (q, \(J = 5.0\) Hz), 114.4, 77.4. \(^{19}\)F NMR (377 MHz, CDCl\(_3\)) \(\delta\) -58.3 ppm.

HRMS \(m/z\) (ESI+): Calcd for C\(_{16}\)H\(_8\)BrClF\(_3\)NONa\(^+\) (M+Na\(^+\)) \(423.9322\), found 423.9320.
(2-Bromophenyl)(5-methoxy-2-(trifluoromethyl)-1H-indol-1-yl)methanone (1s):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); reddish brown liquid 44% yield (for the last step). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.68 (d, $J = 7.5$ Hz, 1H), 7.58-7.46 (m, 3H), 7.23 (s, 1H), 7.05 (s, 1H), 6.74 (d, $J = 9.2$ Hz, 1H), 6.27 (d, $J = 9.2$ Hz, 1H), 3.81 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 165.3, 156.4, 136.6, 133.8, 133.0, 131.6, 130.1, 128.3 (q, $J = 40.0$ Hz), 128.2, 128.1, 120.6, 120.5 (q, $J = 266.0$ Hz), 116.3, 115.1 (q, $J = 5.0$ Hz), 114.8, 104.1, 55.6. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -58.5 ppm. HRMS $m/z$ (ESI+): Calcd for $C_{17}H_{11}BrF_3NO_2Na^+$ (M+Na)$^+$ 419.9818, found 419.9819.
(2-Bromophenyl)(2-(Difluoromethyl)-1H-indol-1-yl)methanone (1t):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:100 (v/v); reddish brown liquid 35% yield (for the last step). $^1$H NMR (400 MHz, CDCl$_3$) δ 7.72 (d, J = 7.0 Hz, 1H), 7.61 (d, J = 7.8 Hz, 1H), 7.53-7.50 (m, 3H), 7.39 (s, 1H), 7.24-7.20 (m, 2H), 7.06 (t, J = 8.1 Hz, 1H), 6.26 (d, J = 8.5 Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 166.6, 136.9, 136.5, 133, 9 (t, J = 29 Hz), 133.8, 132.8, 129.4, 128.7, 128.3, 126.0, 124.0, 122.4, 120.1, 113.8, 112.4 (t, J = 7 Hz), 109.80 (t, J = 235 Hz). $^{19}$F NMR (376 MHz, CDCl$_3$) δ -114.09 (dd, J = 799.2, 293.0 Hz, 2F). HRMS m/z (ESI+): Calcd for C$_{16}$H$_{10}$BrF$_2$NONa$^+$ (M+Na)$^+$ 371.9806, found 371.9807.
4. Procedure for enantioselective dearomative reductive Heck of 1

To a dried Schlenk tube were charged with Pd(OAc)$_2$ (2.3 mg, 0.01 mmol) and (R)-Synphos (7.6 mg, 0.012 mmol) under N$_2$ atmosphere, 2.0 mL CH$_3$CN was then introduced via a syringe and the tube was sealed using Teflon cap. After stirring at 40 ºC for 0.5 h, substrate 1 (0.2 mmol) and Et$_3$SiH (0.4 mmol) were added to the reaction mixture. The resulting mixture was then allowed to stir at 100 ºC until the reaction was complete (monitored by TLC). The solution was then concentrated under reduced pressure. The residue was purified by column chromatography on silica gel, eluting with ethyl/petroleum ether 1:50 (v/v) to afford the products 2.

(R)-10b-(Trifluoromethyl)-10b,11-dihydro-6H-isooindolo[2,1-a]indol-6-one (2a):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (68% yield), m.p. 99-101 ºC. [α]$_D^{20}$ = +196.1 (c 0.5, CH$_2$Cl$_2$), 99% ee Chiralpak AD-H column (25 cm × 0.46 cm ID), $^n$hexane/$^n$PrOH = 80/20, 0.7 mL/min, 280 nm; $t_{major}$ = 13.4 min, $t_{minor}$ = 17.3 min). $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.92 (d, $J$ = 6.6 Hz, 1H), 7.71-7.58 (m, 4H), 7.31-7.21 (m, 2H), 7.10 (t, $J$ = 7.1 Hz, 1H), 3.72 (dd, $J$ = 16.7, 4.9 Hz, 1H), 3.43 (dd, $J$ = 16.4, 4.7 Hz, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 169.7, 142.7, 140.8, 133.7, 133.4, 133.2, 130.5, 128.3, 125.3 (q, $J$ = 282.0 Hz) 125.4,
125.1, 124.8, 123.4, 116.7, 74.2 (q, $J = 30.0$ Hz), 34.7. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -79.9 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{16}$H$_{10}$F$_3$NONa$^+$ (M+Na)$^+$ 312.0607, found 312.0606.
(R)-9-Methoxy-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2b):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); colorless oil (58% yield). $[\alpha]_D^{20} = +294.4$ (c 0.5, CH$_2$Cl$_2$), 99% ee [Daicel Chiralpak C1 column (25 cm × 0.46 cm ID), $^n$hexane/PrOH = 90/10, 0.7 mL/min, 280 nm; $t_{major} = 11.7$ min, $t_{minor} = 13.0$ min]. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.81 (d, $J = 9.2$ Hz, 1H), 7.67 (d, $J = 8.0$ Hz, 1H), 7.29 (t, $J = 7.9$ Hz, 1H), 7.21 (d, $J = 7.4$ Hz, 1H), 7.10-7.07 (m, 3H), 3.92 (s, 3H), 3.69 (d, $J = 16.8$ Hz, 1H), 3.42 (d, $J = 16.5$ Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.8, 164.4, 145.1, 141.2, 132.9, 128.3, 127.0, 125.4, 125.3 (q, $J = 282.0$ Hz), 124.9, 124.8, 123.9, 116.68 (d, $J = 12.0$ Hz), 108.7, 73.8 (q, $J = 30.0$ Hz), 55.9, 34.8. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -80.0 ppm. HRMS m/z (ESI+): Calcd for C$_{17}$H$_{12}$F$_3$NONa$^+$ (M+Na)$^+$ 342.0712, found 342.0714.
(R)-9-Methyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2c):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); colorless oil (50% yield). 

\[\alpha\]$_D^{20}$ = +264.4 (c 0.5, CH$_2$Cl$_2$), 99% ee [Daicel Chiralpak AS column (25 cm × 0.46 cm ID), hexane/iPrOH = 90/10, 0.6 mL/min, 280 nm; t$_{major}$ = 8.1 min, t$_{minor}$ = 8.9 min]. 

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.78 (d, $J$ = 7.8 Hz, 1H), 7.68 (d, $J$ = 7.8 Hz, 1H), 7.40 (t, $J$ = 8.0 Hz, 2H), 7.30 (t, $J$ = 7.7 Hz, 1H), 7.22 (d, $J$ = 7.5 Hz, 1H), 7.11-7.07 (m, 1H), 3.70 (d, $J$ = 16.5 Hz, 1H), 3.41 (d, $J$ = 16.5 Hz, 1H), 2.51 (s, 3H). 

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.9, 145.0, 143.1, 141.0, 133.2, 131.5, 130.7, 128.3, 125.3 (q, $J$ = 282.0 Hz), 125.2, 125.0, 124.1, 124.8, 116.7, 74.0 (q, $J$ = 30.1 Hz), 34.7, 22.1.

$^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -79.9 ppm. 

HRMS m/z (ESI+): Calcd for C$_{17}$H$_{12}$F$_3$NONa$^+$ (M+Na)$^+$ 326.0763, found 326.0764.
(R)-9-chloro-10b-(trifluoromethyl)-10b,11-dihydro-6H-isooindolo[2,1-a]indol-6-one (2d):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (41% yield), m.p. 103-105 °C. [α]D^{20} = +177.3 (c 0.5, CH₂Cl₂), 99% ee [Daicel Chiralpak C4 column (25 cm x 0.46 cm ID), hexane/iPrOH = 90/10, 0.7 mL/min; t_{minor} = 7.8 min, t_{major} = 11.6 min]. ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, J = 8.1 Hz, 1H), 7.68 (d, J = 7.9 Hz, 1H), 7.63 (s, 1H), 7.59 (d, J = 8.2 Hz, 1H), 7.32 (t, J = 7.7 Hz, 1H), 7.26-7.23 (m, 1H), 7.13 (t, J = 7.8 Hz, 1H), 3.71 (d, J = 16.5 Hz, 1H), 3.44 (d, J = 16.5 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 168.7, 144.0, 140.5, 140.3, 132.9, 131.9, 131.2, 128.5, 126.6, 125.3, 125.0 (q, J = 282.0 Hz), 124.9, 124.2, 116.7, 73.9 (q, J = 30.0 Hz), 34.6. ¹⁹F NMR (377 MHz, CDCl₃) δ -79.8 ppm. HRMS m/z (ESI+): Calcd for C₁₆H₉ClF₃NONa⁺ (M+Na)⁺ 346.0217, found 346.0216.

[Chemical structure image]
Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (48% yield), m.p. 118-120 °C. \([\alpha]_{D}^{20} = +137.4 \text{ (c 0.5, CH}_2\text{Cl}_2)\), 95% ee [Daicel Chiralpak C1 column (25 cm × 0.46 cm ID), 
hexane/iPrOH = 90/10, 0.7 mL/min, \(t_{\text{minor}} = 8.7\) min, \(t_{\text{major}} = 9.6\) min]. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.90\) (dd, \(J = 8.4, 5.0\) Hz, 1H), 7.68 (d, \(J = 7.8\) Hz, 1H), 7.33-7.27 (m, 3H), 7.23 (d, \(J = 7.5\) Hz, 1H), 7.12 (t, \(J = 7.5\) Hz, 1H), 3.71 (d, \(J = 16.5\) Hz, 1H), 3.44 (d, \(J = 16.5\) Hz, 1H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta 168.6, 166.1\) (d, \(J = 254.0\) Hz), 145.1, 145.0, 140.7, 132.8, 129.4 (d, \(J = 10.0\) Hz), 128.5, 127.7 (d, \(J = 40.0\) Hz), 125.3 (d, \(J = 37.0\) Hz), 125.0 (q, \(J = 283.0\) Hz), 118.6 (d, \(J = 23.0\) Hz), 116.7, 111.5 (d, \(J = 25.0\) Hz), 74.8 (q, \(J = 30.0\) Hz), 34.6. \(^{19}\)F NMR (377 MHz, CDCl\(_3\)) \(\delta -79.9, -102.7\) ppm. HRMS \(m/z\) (ESI+): Calcd for C\(_{16}\)H\(_9\)F\(_4\)NONa\(^+\) (M+Na\(^+\)) 330.0513, found 330.0513.
(R)-9-Trifluoromethyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2f):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (51% yield), m.p. 111-113 °C. [α]D<sup>20</sup> = +164.5 (c 0.5, CH<sub>2</sub>Cl<sub>2</sub>). 99% ee [Daicel Chiralpak OJ-H column (25 cm × 0.46 cm ID), "hexane/PrOH = 90/10, 0.6 mL/min, 280 nm; t<sub>major</sub> = 9.2 min, t<sub>minor</sub> = 11.1 min]. <sup>1</sup>H NMR (400 MHz, S55
CDCl$_3$ $\delta$ 8.04 (d, $J = 8.3$ Hz, 1H), 7.90-7.88 (m, 2H), 7.70 (d, $J = 7.8$ Hz, 1H), 7.33 (t, $J = 7.7$ Hz, 1H), 7.26 (d, $J = 7.3$ Hz, 1H), 7.14 (t, $J = 7.4$ Hz, 1H), 3.77 (d, $J = 16.5$ Hz, 1H), 3.48 (d, $J = 16.5$ Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 167.9, 142.9, 140.2, 136.8, 135.5 (q, $J = 33.0$ Hz), 133.0, 129.2, 128.6, 128.0 (q, $J = 4.0$ Hz), 126.1, 125.6, 125.0, 124.9 (q, $J = 282.0$ Hz), 123.3 (q, $J = 271.0$ Hz), 116.7, 74.5 (q, $J = 30.0$ Hz), 34.6. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -62.6, -79.7 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{17}$H$_9$F$_6$NONa$^+$ (M+Na)$^+$ 380.0481, found 380.0481.
(R)-8-methyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2g):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); colorless oil (68% yield). \([\alpha]_D^{20} = +119.2\) (c 0.5, CH\(_2\)Cl\(_2\)), 91% ee [Daicel Chiralpak C1 column (25 cm × 0.46 cm ID), \(\text{Hexane/PrOH} = 90/10\), 0.7 mL/min, 280 nm; \(t_{\text{minor}} = 9.2\) min, \(t_{\text{major}} = 10.9\) min]. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.68
(d, $J = 8.0$ Hz, 1H), 7.50 (d, $J = 8.4$ Hz, 1H), 7.36 (d, $J = 2.8$ Hz, 1H), 7.30 (t, $J = 7.7$ Hz, 1H), 7.22-7.18 (m, 2H), 7.10 (t, $J = 7.2$ Hz, 1H), 3.88 (s, 3H), 3.68 (d, $J = 16.5$ Hz, 1H), 3.40 (d, $J = 16.4$ Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.7, 161.7, 140.7, 135.0, 134.8, 133.5, 128.3, 125.3 (q, $J = 282.0$ Hz), 125.1, 124.9, 124.5, 121.7, 116.6, 108.1, 73.8 (q, $J = 30.0$ Hz), 55.9, 34.8. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -80.1 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{17}$H$_{12}$F$_3$NONa$^+$ (M+Na)$^+$ 342.0712, found 342.0714.
(R)-8-methyl-10b-(trifluoromethyl)-11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2h):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); colorless oil (71% yield). $[\alpha]_D^{20} = +179.0$ (c 0.5, CH$_2$Cl$_2$), 98% ee [Daicel Chiralpak C1 column (25 cm × 0.46 cm ID), hexane/iPrOH = 90/10, 0.7 mL/min, 280 nm; t$_\text{minor}$ = 8.2 min, t$_\text{major}$ = 10.0 min]. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.69 (t, $J$ = 7.8 Hz, 2H), 7.50 (d, $J$ = 7.8 Hz, 1H), 7.47 (d, $J$ = 7.8 Hz, 1H), 7.29 (t, $J$ = 7.8 Hz, 2H).
Hz, 1H), 7.21 (d, J = 7.8 Hz, 1H), 7.09 (t, J = 7.5 Hz, 1H), 3.69 (d, J = 16.4 Hz, 1H), 3.40 (d, J = 16.4 Hz, 1H), 2.46 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.0, 141.0, 140.8, 140.0, 134.7, 133.5, 133.4, 128.3, 125.6, 125.3 (q, J = 282.0 Hz), 125.0, 124.8, 123.3, 116.6, 74.0 (q, J = 30.0 Hz), 34.8, 21.4. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -80.0 ppm. HRMS m/z (ESI+): Calcd for C$_{17}$H$_{12}$F$_3$NONa$^+$ (M+Na)$^+$ 326.0763, found 326.0764.
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(R)-8-chloro-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2i):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (65% yield), m.p. 102-104 °C. [α]D20 = +82.4 (c 0.5, CH2Cl2), 99% ee [Daicel Chiralpak AD-H column (25 cm x 0.46 cm ID), hexane/iPrOH = 80/20, 0.6 mL/min, 280 nm; tminor = 10.5 min, tmajor ≈ 13.5 min]. 1H NMR (400 MHz, CDCl3) δ 7.88 (s, 1H), 7.69-7.63 (m, 2H), 7.57 (d, J = 8.2 Hz, 1H), 7.31 (t, J = 7.7 Hz, 1H), 7.23 (d, J = 7.5 Hz, 1H), 7.12 (d, J = 7.5 Hz, 1H), 3.71 (d, J = 16.5 Hz, 1H), 3.43 (d, J = 16.5 Hz, 1H). 13C NMR (100 MHz, CDCl3) δ 168.2, 140.7, 140.4, 137.1, 135.3, 133.9, 133.1, 128.5, 125.5, 125.4, 125.0 (q, J = 282.0 Hz), 124.9, 124.8, 116.7, 74.0 (q, J = 30.0 Hz), 34.6. 19F NMR (377 MHz, CDCl3) δ -79.9 ppm. HRMS m/z (ESI+): Calcd for C16H9ClF3NONa+ (M+Na)+ 346.0217, found 346.0216.
(R)-8-Fluoro-10b-(trifluoromethyl)-10b,11-dihydro-6H-isouindolo[2,1-a]indol-6-one (2j):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); colorless oil (67% yield). [α]D^20 = +92.0 (c 0.5, CH2Cl2), 98% ee [Daicel Chiralpak C1 column (25 cm × 0.46 cm ID), nhexane/iPrOH = 90/10, 0.7 mL/min, 280 nm; t_minor = 7.5 min, t_major = 9.3 min]. 1H NMR (600 MHz, CDCl3) δ 7.68 (dd, J = 7.9, 1.0 Hz, 1H), 7.62-7.60 (m, 1H), 7.58 (dd, J = 7.2, 2.5 Hz, 1H), 7.38 (td, J = 8.5, 2.5 Hz, 1H), 7.33-7.30 (m, 1H), 7.13 (td, J = 7.5, 1.1 Hz, 1H), 3.72 (d, J = 16.4 Hz, 1H), 3.43 (d, J = 16.4 Hz, 1H). 13C NMR (100 MHz, CDCl3) δ 168.4 (d, J = 3.0 Hz), 161.1 (d, J = 250.0 Hz), 140.5, 138.1 (d, J = 2.0 Hz), 135.9 (d, J = 8.0 Hz), 133.2, 128.4, 125.4, 125.3, 125.1 (q, J = 282.0 Hz), 124.9, 121.2 (d, J = 24.0 Hz), 116.7, 112.4 (d, J = 24.0 Hz), 74.4 (q, J = 30.0 Hz), 34.7. 19F NMR (377 MHz, CDCl3) δ -80.0, -108.9 ppm. HRMS m/z (ESI+): Calcd for C16H9F4NONa+ (M+Na)^+ 330.0513, found 330.0513.
(R)-7-Fluoro-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2k):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (57% yield), m.p. 105-107 °C. [α]D^20 = +117.5 (c 0.5, CH₂Cl₂), 99% ee [Daicel Chiralpak C1 column (25 cm × 0.46 cm ID), hexane/IPrOH = 90/10, 0.7 mL/min, 280 nm; t_minor = 10.3 min, t_major = 11.8 min]. ^1H NMR (400 MHz, CDCl₃) δ 7.69-7.64 (m, 2H), 7.42 (d, J = 7.6 Hz, 1H), 7.31 (t, J = 7.7 Hz, 1H), 7.24 (t, J = 8.0 Hz, 2H), 7.11 (t, J = 7.6 Hz, 1H), 3.73 (d, J = 16.5 Hz, 1H), 3.46 (d, J = 16.5 Hz, 1H). ^13C NMR (100 MHz, CDCl₃) δ 166.3 (d, J = 3.0 Hz), 159.4 (d, J = 262.0 Hz), 144.9, 140.7, 135.9 (d, J = 8.0 Hz), 132.8, 128.4, 125.0 (q, J = 282.0 Hz), 125.3, 124.8, 120.6 (d, J = 14.0 Hz), 119.7 (d, J = 4.0 Hz), 118.1 (d, J = 19.0 Hz), 116.8, 74.0 (q, J = 30.0 Hz), 34.8. ^19F NMR (377 MHz, CDCl₃) δ -80.0, -114.2 ppm. HRMS m/z (ESI+): Calcd for C₁₆H₁₀F₄NONa⁺ (M+Na)⁺ 330.0513, found 330.0513.
(R)-7-chloro-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2b):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); colorless oil (61% yield), m.p. 138-140 °C. \([\alpha]D^{20} = +165.5\) (c 0.5, CH\(_2\)Cl\(_2\)), 90% ee [Daicel Chiralpak C1 column (25 cm × 0.46 cm ID), hexane/iPrOH = 90/10, 0.7 mL/min, 210 nm; \(t_{\text{minor}} = 9.6\) min, \(t_{\text{major}} = 10.7\) min]. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\)
7.70 (d, $J = 7.9$ Hz, 1H), 7.64-7.52 (m, 3H), 7.30 (t, $J = 7.7$ Hz, 1H), 7.22 (d, $J = 7.5$ Hz, 1H), 7.11 (td, $J = 7.5$, 1.1 Hz, 1H), 3.73 (d, $J = 16.5$ Hz, 1H), 3.43 (d, $J = 16.5$ Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 167.2, 144.9, 140.8, 134.4, 133.1 (q, $J = 41.0$ Hz), 132.3, 130.5, 129.3, 128.4 (d, $J = 7.0$ Hz), 125.3 (d, $J = 36.0$ Hz), 125.0 (q, $J = 282.0$ Hz), 124.7, 122.1 (d, $J = 2.0$ Hz), 116.8 (d, $J = 24.0$ Hz), 73.1 (q, $J = 30.0$ Hz), 34.9. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -79.8 ppm. HRMS m/z (ESI+): Calcd for C$_{16}$H$_9$CIF$_3$NONa$^+$ (M+Na)$^+$ 346.0217, found 346.0218.
(R)-8,9-Dimethoxy-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2m):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); colorless oil (72% yield). \([\alpha]_D^{20} = +196.0\) (c 0.5, CH\(_2\)Cl\(_2\)), 99% ee [Daicel Chiralpak AD-H column (25 cm × 0.46 cm ID), \(\theta\)hexane/PrOH = 85/15, 0.6
mL/min, 254 nm; $t_{\text{minor}} = 11.6$ min, $t_{\text{major}} = 12.7$ min. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.67 (d, $J = 7.8$ Hz, 1H), 7.33-7.28 (m, 2H), 7.22 (d, $J = 7.8$ Hz, 1H), 7.10 (t, $J = 7.5$ Hz, 1H), 7.03 (s, 1H), 4.01 (s, 3H), 3.96 (s, 3H), 3.68 (d, $J = 16.5$ Hz, 1H), 3.43 (d, $J = 16.4$ Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.4, 154.2, 151.3, 141.1, 136.7, 133.1, 128.3, 125.3 (q, $J = 282.0$ Hz), 125.4, 124.9, 124.8, 116.6, 106.3, 105.2, 73.8 (q, $J = 30.0$ Hz), 56.5, 56.4, 34.7. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -79.9 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{18}$H$_{14}$F$_3$NO$_3$Na$^+$ (M+Na)$^+$ 372.0818, found 372.0817.
(R)-8,9-Difluoro-10b-(trifluoromethyl)-10b,11-dihydro-6H isoindolo[2,1-afindol-6-one (2n):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (50% yield), m.p. 135-137 ºC. \([\alpha]_D^{20} = +107.4\, (c\ 0.5, \text{CH}_2\text{Cl}_2)\), 99% ee [Daicel Chiralpak AD-H column (25 cm × 0.46 cm ID), \(\text{hexane/PrOH} = 80/20\), 0.6 mL/min, 280 nm; \(t_{\text{major}} = 8.6\, \text{min}, t_{\text{minor}} = 10.9\, \text{min}\)]. \(^1\text{H NMR} (400\, \text{MHz, CDCl}_3) \delta 7.71\ (t, \(J = 7.9\, \text{Hz, 1H}), 7.67\ (d, \(J = 7.9\, \text{Hz, 1H}), 7.47\ (t, \(J = 7.9\, \text{Hz, 1H}),
7.33 (t, J = 7.7 Hz, 1H), 7.25 (d, J = 7.6 Hz, 2H), 7.14 (t, J = 7.5 Hz, 1H), 3.71 (d, J = 16.5 Hz, 1H), 3.44 (d, J = 16.5 Hz, 1H). 1H NMR (100 MHz, CDCl₃) δ 167.8 (d, J = 3.0 Hz), 154.6 (dd, J = 179.0, 14.0 Hz), 152.0 (dd, J = 175.0, 14.0 Hz), 140.4, 138.9 (d, J = 8.0 Hz), 132.8, 130.1 (dd, J = 7.0, 6.0 Hz), 128.6, 125.5, 125.0, 120.6 (q, J = 283.0 Hz), 116.7, 114.4 (dd, J = 19.3, 1.9 Hz) 113.2 (d, J = 20.0 Hz), 74.0 (q, J = 30.0 Hz), 34.6. 19F NMR (377 MHz, CDCl₃) δ -79.9, -125.7, -131.6 ppm. HRMS m/z (ESI+): Calcd for C16H9F5NO+ (M+H)+ 326.0599, found 326.0600.
(R)-13a-(Trifluoromethyl)-13,13a-dihydro-7H-benzo[6,7]isoindolo[2,1-a]indol-7-one (2o):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); colorless liquid (23% yield), 
$[\alpha]_D^{20} = +149.0$ (c 0.5, CH$_2$Cl$_2$), 89% ee [Daicel Chiralpak AD-H column (25 cm × 0.46 cm ID), hexane/iPrOH = 80/20, 0.6 mL/min, 280 nm; $t_{\text{major}} = 32.8$ min, $t_{\text{minor}} = 54.3$ min]. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 8.06 (t, $J = 8.6$ Hz, 2H), 8.02 (dd, $J = 8.0$, 1.5 Hz, 1H), 7.91 (d, $J = 8.3$ Hz,
1H), 7.76 (d, J = 7.9 Hz, 1H), 7.72-7.67 (m, 2H), 7.33 (t, J = 7.7 Hz, 1H), 7.27 (d, J = 7.5 Hz, 1H), 7.11 (t, J = 7.5 Hz, 1H), 4.02 (d, J = 16.3 Hz, 1H), 3.72 (d, J = 16.3 Hz, 1H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 169.7, 141.6, 140.4, 136.4, 132.4, 132.3, 132.0, 129.5, 128.6, 128.4, 128.1, 127.5, 125.4 (q, J = 282.0 Hz), 124.92, 124.89, 124.87, 120.5, 116.0, 75.1 (q, J = 30.0 Hz), 34.2. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -78.1 ppm. HRMS m/z (ESI+): Calcd for C$_{20}$H$_{12}$F$_3$NONa$^+$ (M+Na)$^+$ 362.0763, found 362.0760.
(R)-3-Methoxy-10b-(difluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2p):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (62% yield), m.p. 140-142 °C. [α]D20 = +300.0 (c 0.5, CH2Cl2), 98% ee [Daicel Chiralpak AD-H column (25 cm × 0.46 cm ID), n-hexane/iPrOH = 90/10, 0.6 mL/min, 210 nm; tminor = 15.9 min, tmajor = 23.2 min]. 1H NMR (600 MHz,
CDCl$_3$ $\delta$ 7.91 (s, 1H), 7.63-7.60 (m, 3H), 7.30 (s, 1H), 7.11 (q, $J$ = 4.1, 3.4 Hz, 1H), 6.65 (d, $J$ = 7.2, 1H), 3.85 (s, 3H), 3.65 (d, $J$ = 15.8 Hz, 1H), 3.37 (d, $J$ = 15.8 Hz, 1H).

$^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 169.6, 160.1, 142.8, 141.9, 133.7, 133.4, 130.5, 125.4, 125.3 (q, $J$ = 282.0 Hz), 125.0, 124.7, 123.6, 111.0, 102.8, 75.0 (q, $J$ = 30.0 Hz), 55.7, 34.0. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -79.9 ppm. HRMS m/z (ESI+): Calcd for C$_{17}$H$_{12}$F$_3$NONa$^+$ (M+Na)$^+$ 342.0712, found 342.0711.
(R)-3-fluoro-10b-(difluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2q):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (47% yield), m.p. 83-85 °C. [α]$_D^{20}$ = +175.9 (c 0.5, CH$_2$Cl$_2$), 99% ee [Daicel Chiralpak OJ column (25 cm × 0.46 cm ID), n-hexane/i-PrOH = 80/20, 0.6 mL/min, 280 nm; t$_{major}$ = 9.5 min, t$_{minor}$ = 10.9 min]. $^1$H NMR (600 MHz, CDCl$_3$) δ 7.92 (d, J = 7.6 Hz, 1H), 7.70 (t, J = 7.5 Hz, 1H), 7.64-7.61 (m, 2H), 7.43 (dd,
$J = 8.7, 2.5$ Hz, 1H), 7.17-7.14 (m, 1H), 6.82-6.78 (m, 1H), 3.69 (d, $J = 16.3$ Hz, 1H),
3.39 (d, $J = 16.3$ Hz, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 169.5, 162.8 (d, $J = 244.5$
Hz), 142.6, 142.0 (d, $J = 12.0$ Hz), 134.0, 133.0, 130.7, 128.5 (d, $J = 3.0$ Hz), 125.6,
125.3 (d, $J = 10.5$ Hz), 125.2 (q, $J = 282.0$ Hz), 123.6, 111.6 (d, $J = 7.5$ Hz), 105.1(d, $J$
= 27.0 Hz), 75.0 (d, $J = 30.0$ Hz), 34.1. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -80.0, -112.9
ppm. HRMS m/z (ESI+): Calcd for C$_{16}$H$_9$F$_4$NO$_3$Na$^+$ (M+Na)$^+$ 330.0513, found
330.0514.
(R)-3-chloro-10b-(difluoromethyl)-10b,11-dihydro-6H-isoindolof2,1-alindol-6-one (2r):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (57% yield), m.p. 175-177 ºC. [α]_D^{20} = +154.3 (c 0.5, CH₂Cl₂), 97% ee [Daicel Chiralpak OJ column (25 cm × 0.46 cm ID), "hexane/iPrOH = 80/20, 0.6 mL/min, 280 nm; t_major = 9.6 min, t_minor = 11.2 min]. ^1H NMR (600 MHz,
CDCl₃ δ 7.92 (d, J = 7.5 Hz, 1H), 7.72 (d, J = 7.7 Hz, 2H), 7.61 (dd, J = 13.0, 7.0 Hz, 2H), 7.14 (d, J = 7.9 Hz, 1H), 7.08 (d, J = 8.0 Hz, 1H), 3.70 (d, J = 16.6 Hz, 1H), 3.39 (d, J = 16.6 Hz, 1H). ¹³C NMR (150 MHz, CDCl₃) δ 169.4, 142.5, 141.8, 134.0 (d, J = 30.0 Hz), 132.9, 131.7, 130.7, 129.9 (d, J = 4.5 Hz), 125.6, 125.5, 125.11, 125.09 (q, J = 282.0 Hz), 123.6, 117.2, 74.6 (q, J = 30.0 Hz), 34.34. ¹⁹F NMR (377 MHz, CDCl₃) δ -80.0 ppm. HRMS m/z (ESI+): Calcd for C₁₆H₉ClF₃NONa⁺ (M+Na)⁺ 346.0217, found 346.0219.
(R)-2-Methoxy-10b-(difluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (2s):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); colorless oil (53% yield). 

$[\alpha]_D^{20} = +94.4$ (c 0.5, CH$_2$Cl$_2$), 99% ee [Daicel Chiralpak AD-H column (25 cm × 0.46 cm ID), hexane/iPrOH = 80/20, 0.6 mL/min, 280 nm; $t_{major} = 26.2$ min, $t_{minor} = 36.1$ min]. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.91 (d, $J = 7.5$ Hz, 1H), 7.70-7.58 (m, 4H), 6.84-6.80 (m, 2H), 3.79 (s, 3H), 3.68 (d, $J = 16.5$ Hz, 1H), 3.41 (d, $J = 16.5$ Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$)) $\delta$ 169.9, 157.4, 142.5, 134.9, 134.3, 133.6, 133.4, 130.5, 125.4, 125.3 (q, $J = 283.0$ Hz), 123.5, 117.1, 112.6, 111.5, 74.5 (q, $J = 30.0$ Hz), 55.7, 34.9. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -79.8 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{17}$H$_{12}$F$_3$NO$_3$ (M+Na)$^+$ 342.0712, found 362.0713.
(R)-10b-(Difluoromethyl)-10b,11-dihydro-6H-isoinol[2,1-a]indol-6-one (2):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); colorless liquid (51% yield). \([\alpha]_D^{20} = +206.5 \text{ (c 0.5, CH}_2\text{Cl}_2), 98\% \text{ ee [Daicel Chiralpak OJ-H column (25 cm} \times \text{ 0.46 cm ID), } ^\text{\hexane/PrOH} = 90/10, \text{ 0.6 mL/min, 280 nm; } t_{\text{major}} = 21.5 \text{ min, } t_{\text{minor}} = 26.3 \text{ min}. \] 1H NMR (600 MHz, CDCl$_3$) \(\delta\) 7.90 (d, \(J = 7.5 \text{ Hz, 1H}), 7.69-7.61 \text{ (m, 3H), 7.57 (t, } J = 7.4 \text{ Hz, 1H}), 7.29 \text{ (t, } J = 7.7 \text{ Hz, 1H), 7.23 (d, } J = 7.4 \text{ Hz, 1H), 7.10 (t, } J = 7.5 \text{ Hz, 1H}), 5.76 (t, } J = 55.8 \text{ Hz, 1H), 3.65 (d, } J = 16.2 \text{ Hz, 1H), 3.31 (d, } J = 16.2 \text{ Hz, 1H).} \] 13C NMR (150 MHz, CDCl$_3$) \(\delta\) 169.4, 143.6 (d, \(J = 3.0 \text{ Hz}), 140.4, 133.9 (d, } J = 34.5.0 \text{ Hz), 133.4, 130.1, 128.3, 125.2, 125.1 (d, } J = 13.5.0 \text{ Hz), 124.2, 117.1, 116.7, 115.4, 113.7, 74.1 \text{ (t, } J = 24.0 \text{ Hz), 33.2.} \] 19F NMR (565 MHz, CDCl$_3$) \(\delta\) -128.67 (ddd, \(J = 706.6, 281.1, 55.6 \text{ Hz, 2F).} \) HRMS \(m/z\) (ESI+): Calcd for C$_{16}$H$_{11}$F$_2$NONa$^+$ (M+Na$^+$) 294.0701, found 294.0700.
5. Procedure for enantioselective Heck-Suzuki reaction of 1 with Ar₄BNa

To a dried Schlenk tube were charged with Pd(dba)₂ (5 mol%, 5.8 mg), chiral ligand L₁₆ (10 mol%, 19.2 mg), substrate 1 (0.2 mmol), K₂CO₃ (0.4 mmol, 55.2 mg), and Ar₄BNa (0.4 mmol) under N₂ atmosphere. 1,4-Dioxane (2.0 mL) was then introduced via a syringe and the tube was sealed using Teflon cap. The resulting mixture was stirred at 100 °C until the reaction was completed (monitored by TLC). The solution was then concentrated under reduced pressure and the residue was purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether (v/v = 1/80) to afford product 3.

(10bR,11S)-11-Phenyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-af]indol-6-one (3a):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (85% yield), m.p. 169-171 °C. [α]D²⁰ = +63.0 (c 0.5, CH₂Cl₂), 95% ee [Daicel Chiralpak AD-H column (25 cm × 0.46 cm ID), nhexane/iPrOH = 80/20, 0.6 mL/min, 280 nm; t_major = 7.5 min, t_minor = 16.2 min]. ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, J = 7.9 Hz, 1H), 7.77 (d, J = 7.8 Hz, 1H), 7.43-7.35 (m, 2H), 7.29 (t, J = 7.8 Hz, 1H), 7.14 (q, J = 6.4, 5.3 Hz, 2H), 7.07 (d, J = 7.6 Hz, 1H), 7.02-7.00 (m, 3H), 6.65 (s, 2H), 4.98 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 170.1, 140.5, 140.1, 139.0, 137.0, 133.3, 132.8, 130.0, 128.9, 128.6, 128.4, 127.6, 125.8, 125.6, 125.4 (q, J = 282.0 Hz), 124.9, 124.7, 116.5, 78.5 (q, J = 30.0 Hz), 51.0. ¹⁹F NMR (377 MHz, CDCl₃) δ -78.7 ppm. HRMS m/z (ESI+): Calcd for C₂₂H₁₄F₃NONa⁺ (M+Na)⁺ 388.0919, found 388.0915.
(10bR,11S)-9-Methoxy-11-phenyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolof2,1-alindol-6-one (3b):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (68% yield), m.p. 124-126 °C. \([\alpha]_{D}^{20} = +44.5\) (c 0.5, CH2Cl2), 83% ee [Daicel Chiralpak OD-H column (25 cm × 0.46 cm ID), hexane/iPrOH = 80/20, 0.6 mL/min, 280 nm; t\text{minor} = 7.3 min, t\text{major} = 8.3 min].
$^{1}$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.81 (d, $J = 7.9$ Hz, 1H), 7.66 (d, $J = 8.5$ Hz, 1H), 7.39 (ddd, $J = 8.2$, 6.9, 2.0 Hz, 1H), 7.13-7.09 (m, 2H), 7.04 (t, $J = 2.9$ Hz, 3H), 6.86 (dd, $J = 8.5$, 2.3 Hz, 1H), 6.68 (s, 2H), 6.46 (s, 1H), 4.95 (s, 1H), 3.65 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.2, 163.4, 142.7, 141.0, 139.0, 136.5, 134.7, 128.9, 128.6, 128.5, 127.7, 126.1, 125.7, 125.5, 125.4 (q, $J = 282.0$ Hz), 125.3, 116.9, 116.4, 109.6, 77.9 (q, $J = 30.0$ Hz), 55.6, 51.1. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -78.7 ppm. HRMS m/z (ESI+): Calcd for C$_{23}$H$_{16}$F$_3$NO$_2$Na$^+$ (M+Na)$^+$ 418.1025, found 418.1025.
(10bR,11S)-9-Methyl-11-phenyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (3c): Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); colorless liquid (74% yield). [α]D20 = +22.2 (c 0.5, CH2Cl2), 94% ee [Daicel Chiralpak AD-H column (25 cm × 0.46 cm ID), hexane/PrOH = 80/20, 0.6 mL/min, 254 nm; tminor = 7.2 min, tmajor = 13.7 min]. 1H NMR (600 MHz, CDCl3) δ 7.83 (d, J = 7.9 Hz, 1H), 7.64 (d, J = 7.8 Hz, 1H), 7.41-7.38 (m, 1H), 7.16-7.12 (m, 3H), 7.01 (t, J = 2.5 Hz, 2H), 6.83 (s, 1H), 6.64 (s, 2H), 4.95 (s, 1H), 2.20 (s, 3H). 13C NMR (151 MHz, CDCl3) δ 170.2, 143.8, 140.8, 140.5, 139.0, 136.8, 130.8, 130.7, 128.8, 128.6, 128.5, 128.4, 125.7, 125.5, 125.4, 125.37 (q, J = 282.0 Hz), 124.5, 116.5, 78.2 (q, J = 30.0 Hz), 51.0, 21.8. 19F NMR (377 MHz, CDCl3) δ -78.7 ppm. HRMS m/z (ESI+): Calcd for C23H16F3NONa+ (M+Na)+ 402.1076, found 402.1077.
(10bR,11S)-9-Phenyl-11-Phenyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (3d);

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (80% yield). $[\alpha]_D^{20} = -36.8$ (c 0.5, CH$_2$Cl$_2$), 99% ee [Daicel Chiralpak A2 column (25 cm × 0.46 cm ID), hexane/iPrOH = 90/10, 0.6 mL/min, 210 nm; $t_{\text{minor}} = 9.8$ min, $t_{\text{major}} = 14.3$ min]. $^1$H NMR (600 MHz, CDCl$_3$) δ 7.99 (d, $J = 1.7$ Hz, 1H), 7.86 (d, $J = 7.9$ Hz, 1H), 7.52 (dt, $J = 8.1$, 1.6 Hz, 3H), 7.43-7.41 (m, 3H), 7.37-7.35 (m, 1H), 7.16-7.11 (m, 3H), 7.05-7.01 (m, 3H), 6.68 (s, 2H), 5.00 (s, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) $^{13}$C NMR (150 MHz, CDCl$_3$) δ 167.0, 143.0, 140.5, 139.1, 139.0, 138.8, 136.9, 134.1, 131.6, 129.0, 128.9, 128.6, 128.5, 128.2, 127.7, 127.2, 125.8, 125.6, 125.3 (q, $J = 282$ Hz), 125.2, 122.9, 116.53, 78.4 (q, $J = 30$ Hz), 51.05. $^{19}$F NMR (377 MHz, CDCl$_3$) δ -78.7 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{28}$H$_{18}$F$_3$NONa$^+$ (M+Na)$^+$ 464.1233, found 464.1233.

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (56% yield), m.p. 150-152 °C. [α]D20  = +67.8 (c 0.5, CH2Cl2), 77% ee [Daicel Chiralpak C2 column (25 cm × 0.46 cm ID), hexane/iPrOH = 95/05, 0.5 mL/min, 280 nm; tminor = 9.2 min, tmajor = 10.1 min].

H NMR (600 MHz, CDCl3) δ 7.82 (d, J = 7.9 Hz, 1H), 7.76 (dd, J = 8.4, 4.9 Hz, 1H), 7.41 (t, J = 8.2 Hz, 1H), 7.20-7.10 (m, 2H), 7.06 (m, 4H), 6.75 (d, J = 7.7 Hz, 2H), 6.65 (s, 2H), 4.96 (s, 1H).

13C NMR (150 MHz, CDCl3) δ 168.9, 165.3 (d, J = 253.5 Hz), 142.7 (d, J = 10.5 Hz), 140.5, 138.5, 136.6, 129.4 (d, J = 1.5 Hz), 129.0, 128.7, 128.3, 127.9, 126.8 (d, J = 10.5 Hz), 125.8 (d, J = 7.5 Hz), 125.0 (q, J = 282 Hz), 117.9, 117.80, 116.5, 112.7, 112.5, 78.1 (q, J = 30.0 Hz), 51.06.

19F NMR (377 MHz, CDCl3) δ -78.7, -103.9 ppm. HRMS m/z (ESI+): Calcd for C22H13F4NONa+ (M+Na)+ 406.0826, found 406.0826.
**(10bR,11S)-8,9-Dimethoxy-11-phenyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-α]indol-6-one(3f):**

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (51% yield), m.p. 150-152 °C. [α]_D^{20} = +93.0 (c 0.5, CH₂Cl₂), 82% ee [Daicel Chiralpak OD-H column (25 cm × 0.46 cm ID), hexane/iPrOH = 80/20, 0.6 mL/min, 280 nm; t_{minor} = 8.0 min, t_{major} = 9.0 min].

**1H NMR (400 MHz, CDCl₃) δ 7.81 (d, J = 7.9 Hz, 1H), 7.40 (ddd, J = 8.3, 6.5, 2.2 Hz, 1H), 7.19 (s, 1H), 7.15-7.10 (m, 2H), 7.07-7.05 (m, 3H), 6.70 (s, 2H), 6.38 (s, 1H), 4.96 (s, 1H), 3.87 (s, 3H), 3.68 (s, 3H).**

**13C NMR (100 MHz, CDCl₃) δ 170.8, 153.2, 150.8, 141.0, 139.3, 136.4, 134.5, 128.9, 128.7, 128.5, 127.7, 125.8, 125.5, 125.4 (q, J = 282.0 Hz), 125.3, 116.4, 106.7, 105.6, 78.9 (q, J = 30.0 Hz), 56.2, 50.9.**

**19F NMR (377 MHz, CDCl₃) δ -79.0 ppm.**

**HRMS m/z (ESI+):** Calcd for C₂₄H₁₈F₃NO₃Na⁺ (M+Na)⁺ 448.1131, found 448.1130.
(10bR,11S)-8-Methoxy-11-phenyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-
isoindolo[2,1-a]indol-6-one (3g):
Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); colorless liquid (76% yield). 
$[\alpha]_D^{20} = +62.7$ (c 0.5, CH$_2$Cl$_2$), 92% ee [Daicel Chiralpak A2 column (25 cm × 0.46 cm ID), $\nu$hexane/PrOH = 90/10, 0.6 mL/min, 280 nm; $t_{\text{minor}} = 12.3$ min, $t_{\text{major}} = 14.7$ min]. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.83 (d, $J = 7.9$ Hz, 1H), 7.40 (ddd, $J = 8.3$, 6.4, 2.3 Hz, 1H), 7.23 (d, $J = 2.4$ Hz, 1H), 7.16-7.11 (m, 2H), 7.05-7.02 (m, 3H), 6.95 (d, $J = 8.5$ Hz, 1H), 6.83 (dd, $J = 8.5$, 2.5 Hz, 1H), 6.65 (s, 2H), 4.95 (s, 1H), 3.76 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.0, 161.1, 140.5, 139.2, 137.1, 134.9, 132.3, 128.9, 128.6, 128.4, 127.6, 125.8, 125.7, 125.5, 125.4 (q, $J = 282.0$ Hz), 121.0, 116.4, 107.3, 78.2 (q, $J = 30.0$ Hz), 55.6, 50.9. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -78.9 ppm. HRMS $m/z$ (ESI$^+$): Calcd for C$_{22}$H$_{16}$F$_3$NO$_2$Na$^+$ (M+Na)$^+$ 418.1025, found 418.1025.
(10bR,11S)-8-Methyl-11-phenyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoiindo[2,1-a]indol-6-one (3h):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (83% yield), m.p. 142-144 °C. [α]D20 = +48.8 (c 0.5, CH2Cl2), 96% ee [Daicel Chiralpak OD-H column (25 cm × 0.46 cm ID), n-hexane/iPrOH = 80/20, 0.6 mL/min, 280 nm; tminor = 7.2 min, tmajor = 13.1 min].

1H NMR (600 MHz, CDCl3) δ 7.83 (d, J = 7.9 Hz, 1H), 7.56 (s, 1H), 7.41-7.38 (m, 1H), 7.14-7.08 (m, 3H), 7.03 (dd, J = 5.0, 2.0 Hz, 3H), 6.94 (d, J = 7.9 Hz, 1H), 6.65 (s, 2H), 4.95 (s, 1H), 2.31 (s, 3H). 13C NMR (150 MHz, CDCl3) δ 170.2, 140.6, 140.3, 139.1, 137.4, 137.0, 133.8, 128.8, 128.5, 127.7, 127.5, 125.8, 125.44, 125.38 (q, J = 282.0 Hz), 124.9, 124.6, 116.5, 78.3 (q, J = 30.0 Hz), 50.9, 21.3. 19F NMR (377 MHz, CDCl3) δ -78.9 ppm. HRMS m/z (ESI+): Calcd for C23H16F3NONa+ (M+Na)+ 402.1076, found 402.1075.

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(10bR,11S)-8-Phenyl-11-phenyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isouindolo[2,1-a]indol-6-one (3i):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (72% yield). [α]D^20 = -89.6 (c 0.5, CH₂Cl₂), 90% ee [Daicel Chiralpak C1 column (25 cm × 0.46 cm ID), hexane/iPrOH = 90/10, 0.6 mL/min, 254 nm; t_minor = 7.9 min, t_major = 10.8 min]. \(^1\)H NMR (600 MHz, CDCl₃) δ 7.99 (d, J = 1.2 Hz, 1H), 7.86 (d, J = 7.9 Hz, 1H), 7.52 (dd, J = 8.1, 1.7 Hz, 3H), 7.42 (t, J = 7.5 Hz, 3H), 7.36 (t, J = 7.2 Hz, 1H), 7.17-7.11 (m, 3H), 7.05-7.01 (m, 3H), 6.69 (s, 2H), 5.00 (s, 1H). \(^13\)C NMR (150 MHz, CDCl₃) δ 170.0, 143.2, 140.5, 139.1, 139.0, 138.8, 136.9, 134.1, 131.6, 129.0, 128.9, 128.6, 128.4, 128.2, 127.7, 127.1, 125.8, 125.6, 125.2, 125.4 (q, J = 282.0 Hz), 122.9, 116.5, 78.2 (q, J = 30.0 Hz), 51.0. \(^19\)F NMR (377 MHz, CDCl₃) δ -78.7 ppm. HRMS m/z (ESI+): Calcd for C₂₈H₁₈F₃NONa⁺ (M+Na)⁺ 464.1233, found 464.1233.
(10bR,11S)-11-(Benzo[d][1,3]dioxo-5-yl)-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]Indole-6-one (3j):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (57% yield). [α]D20 = +21.9 (c 0.5, CH2Cl2), 80% ee [Daicel Chiralpak AS column (25 cm × 0.46 cm ID), hexane/iPrOH = 90/10, 0.6 mL/min, 280 nm; t minor = 10.9 min, t major = 12.2 min]. 1H NMR (600 MHz, CDCl3) δ 7.82 (d, J = 7.9 Hz, 1H), 7.79 (d, J = 7.2 Hz, 1H), 7.43-7.36 (m, 3H), 7.16-7.11 (m, 3H), 6.49 (d, J = 8.4 Hz, 1H), 6.23 (s, 1H), 6.01 (s, 1H), 5.76 (dd, J = 13.7, 1.5 Hz, 2H), 4.90 (s, 1H). 13C NMR (150 MHz, CDCl3) δ 170.0, 147.8, 146.9, 140.3, 140.1, 136.9, 133.4, 132.9, 132.9, 130.0, 128.9, 125.7, 125.5, 125.4 (q, J = 282.0 Hz), 124.9, 124.8, 122.0, 116.5, 108.7, 108.1, 101.0, 78.4 (q, J = 30.0 Hz), 50.7. 19F NMR (377 MHz, CDCl3) δ -78.8 ppm. HRMS m/z (ESI+): calcd for C23H14F3NO3Na+ (M+Na)+ 432.0818, found 432.0818.
(10bR,11S)-11-(3-Methoxyphenyl)-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoinindolo[2,1-a]indol-6-one (3k):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); colorless liquid (90% yield). \([\alpha]_D^{20} = +62.2 \text{ (c 0.5, CH}_2\text{Cl}_2)\), 79% ee [Daicel Chiralpak A2 column (25 cm × 0.46 cm ID), \(n\)hexane/PrOH = 95/05, 0.6 mL/min, 210 nm; \(t_{\text{minor}} = 17.5 \text{ min}, t_{\text{major}} = 27.9 \text{ min}\)]. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.84 (d, \(J = 7.9 \text{ Hz, 1H}\), 7.68 (d, \(J = 7.8 \text{ Hz, 1H}\), 7.42-7.36 (m, 2H), 7.31 (td, \(J = 7.5, 1.3 \text{ Hz, 1H}\), 7.14-7.11 (m, 3H), 6.92 (t, \(J = 7.9 \text{ Hz, 1H}\), 6.54 (ddd, \(J = 8.3, 2.6, 0.9 \text{ Hz, 1H}\), 6.21 (d, \(J = 29.7 \text{ Hz, 2H}\), 4.93 (s, 1H), 3.52 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 170.0, 159.6, 140.5, 140.4, 140.1, 136.8, 133.3, 132.8, 130.0, 129.6, 128.9, 125.8, 125.6, 125.4 (q, \(J = 282.0 \text{ Hz}\), 124.9, 124.6, 120.8, 116.5, 114.1, 113.0, 78.5 (q, \(J = 30.0 \text{ Hz}\), 55.1, 50.9. \(^{19}\)F NMR (377 MHz, CDCl\(_3\)) \(\delta\) -78.7 ppm. HRMS \(m/z\) (ESI+): Calcd for C\(_{23}\)H\(_{16}\)F\(_3\)NO\(_2\)Na\(^+\) (M+Na\(^+\)) 418.1025, found 418.1025.
Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (86% yield), m.p. 188-190 °C. $[\alpha]_D^{20} = +79.9$ (c 0.5, CH$_2$Cl$_2$), 87% ee [Daicel Chiralpak A2 column (25 cm × 0.46 cm ID), hexane/iPrOH = 90/10, 0.6 mL/min, 280 nm; $t_{\text{minor}} = 11.3$ min, $t_{\text{major}} = 16.7$ min]. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.84 (d, $J = 7.9$ Hz, 1H), 7.68 (d, $J = 7.8$ Hz, 1H), 7.42-7.35 (m, 2H), 7.30 (t, $J = 7.8$ Hz, 1H), 7.14 (d, $J = 6.3$ Hz, 2H), 7.07 (d, $J = 7.6$ Hz, 1H), 6.68 (t, $J = 7.5$ Hz, 1H), 6.82 (d, $J = 7.5$ Hz, 1H), 6.44 (d, $J = 32.4$ Hz, 2H), 4.93 (s, 1H), 2.09 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.1, 140.5, 140.2, 138.9, 138.2, 137.0, 133.3, 132.7, 129.9, 129.1, 128.8, 128.4, 128.3, 125.8, 125.5, 125.4 (q, $J = 30.0$ Hz), 124.9, 124.6, 116.5, 78.5 (q, $J = 30.0$ Hz), 51.0, 21.2. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -78.7 ppm. HRMS $m/z$ (ESI+): Calcd for C$_{23}$H$_{16}$F$_3$NONa$^+$ (M+Na)$^+$ 402.1076, found 402.1076.
(10bR,11S)-11-(4-Methoxyphenyl)-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (3m):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); colorless liquid (82% yield). [α]D20 = +119.1 (c 0.5, CH2Cl2), 89% ee [Daicel Chiralpak A2 column (25 cm × 0.46 cm ID), n hexane/iPrOH = 80/20, 0.6 mL/min, 280 nm; tminor = 10.5 min, tmajor = 17.0 min]. 1H NMR (400 MHz, CDCl3) δ 7.83 (d, J = 7.9 Hz, 1H), 7.77 (d, J = 7.8 Hz, 1H), 7.38 (q, J = 6.9 Hz, 2H), 7.32 (t, J = 7.8 Hz, 1H), 7.15-7.08 (m, 3H), 6.55 (s, 4H), 4.94 (s, 1H), 3.62 (s, 3H). 13C NMR (100 MHz, CDCl3) δ 170.1, 158.8, 140.3, 140.2, 137.3, 133.3, 132.9, 131.3, 129.9, 129.5, 128.8, 125.7, 125.5, 125.4 (q, J = 282.0 Hz), 125.0, 124.7, 116.5, 113.8, 78.5 (q, J = 30.0 Hz), 55.1, 50.3. 19F NMR (377 MHz, CDCl3) δ -78.7 ppm. HRMS m/z (ESI+): Calcd for C23H16F3NO2Na+ (M+Na)+ 418.1025, found 418.1023.
(10bR,11S)-11-(4-Ethylphenyl)-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (3n):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (79% yield). \([\alpha]_D^{20} = +27.8 \text{ (c 0.5, CH}_2\text{Cl}_2)\), 91% ee [Daicel Chiralpak A2 column (25 cm \times 0.46 cm ID), \(n\)-hexane/iPrOH = 90/10, 0.6 mL/min, 280 nm; \(t_{\text{minor}} = 9.9 \text{ min, } t_{\text{major}} = 15.0 \text{ min}\)].

\(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta 7.83 (d, J = 7.9 \text{ Hz, } 1H), 7.76 (d, J = 7.8 \text{ Hz, } 1H), 7.36 (dt, J = 20.9, 6.6 \text{ Hz, } 2H), 7.26 (dd, J = 15.3, 7.8 \text{ Hz, } 1H), 7.14-7.10 \text{ ppm. HRMS } m/z (ESI+):\) Calcd for C\(_{24}\)H\(_{18}\)F\(_3\)NONa\(^+\) (M+Na)\(^+\) 416.1233, found 416.1232.
(10bR,11S)-11-(((1',1'-Biphenyl)-4-yl)-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indole Dol-6-one (3o):}

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); colorless liquid (51% yield). 

$[\alpha]_D^{20} = +20.8$ (c 0.5, CH$_2$Cl$_2$), 93% ee [Daicel Chiralpak A2 column (25 cm × 0.46 cm ID), hexane/PrOH = 90/10, 0.6 mL/min, 254 nm; $t_{\text{minor}} = 15.7$ min, $t_{\text{major}} = 31.5$ min].

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.86 (d, $J = 7.9$ Hz, 1H), 7.78 (d, $J = 7.8$ Hz, 1H), 7.43-7.33 (m, 6H), 7.35-7.23 (m, 4H), 7.16 (d, $J = 4.2$ Hz, 2H), 7.12 (d, $J = 7.8$ Hz, 1H), 6.71 (d, $J = 7.2$ Hz, 2H), 5.02 (s, 1H).

$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 170.1, 140.5, 140.3, 140.08, 140.06, 138.0, 136.9, 133.3, 132.9, 130.0, 129.0, 128.9, 128.8, 127.5, 127.1, 126.8, 125.8, 125.6, 125.4 (q, $J = 282.0$ Hz), 125.0, 124.8, 116.6, 78.5 (q, $J = 30.0$ Hz), 50.7. $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -78.7 ppm. HRMS m/z (ESI+): Calcd for C$_{28}$H$_{19}$F$_3$NONa$^+$ (M+Na)$^+$ 464.1233, found 464.1234.
(10bR,11S)-11-(4-fluorophenyl)-10b-(trifluoromethyl)-10b,11-dihydro-6H-
isoindolo[2,1-a]indol-6-one (3p):
Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (45% yield), m.p. 216-218 °C. [α]D20 = +58.8 (c 0.5, CH2Cl2), 77% ee [Daicel Chiralpak A2 column (25 cm × 0.46 cm ID), n-hexane/iPrOH = 90/10, 0.6 mL/min, 280 nm; t_minor = 9.7 min, t_major = 13.5 min]. 1H NMR (600 MHz, CDCl3) δ 7.84 (d, J = 7.9 Hz, 1H), 7.78 (d, J = 7.2 Hz, 1H), 7.41 (dd, J = 7.5, 1.2 Hz, 2H), 7.34 (t, J = 8.0 Hz, 1H), 7.15 (t, J = 7.4 Hz, 1H), 7.11 (d, J = 7.4 Hz, 1H), 7.07 (d, J = 7.7 Hz, 1H), 6.72 (t, J = 8.5 Hz, 2H), 6.62 (s, 2H), 4.97 (s, 1H). 13C NMR (150 MHz, CDCl3) δ 169.9, 161.9 (d, J = 244.5 Hz), 140.4, 139.9, 136.7, 134.9 (d, J = 4.5 Hz), 133.3, 132.9, 130.1 (d, J = 9.0 Hz), 130.0, 129.1, 125.7 (d, J = 7.5 Hz), 125.3 (q, J = 282.0 Hz), 124.8, 116.6, 115.6, 115.4, 78.4 (q, J = 30.0 Hz), 50.2. 19F NMR (377 MHz, CDCl3) δ -78.7, -114.0 ppm. HRMS m/z (ESI+): Calcd for C22H13F4NONa+ (M+Na)+ 406.0826, found 406.0824.
(10bR,11S)-3-fluoro-11-phenyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoinindolo[2,1-a]indol-6-one (3q):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (58% yield), m.p. 155-157 °C. [α]D²⁰ = +89.1 (c 0.5, CH₂Cl₂), 90% ee [Daicel Chiralpak OD-H column (25 cm x 0.46 cm ID), hexane/PrOH = 90/10, 0.6 mL/min, 230 nm; $t_{major} = 7.3$ min, $t_{minor} = 7.7$ min]. $^1$H NMR (400 MHz, CDCl₃) δ 7.77 (d, $J = 7.5$ Hz, 1H), 7.57 (dd, $J = 8.8$, 2.5 Hz, 1H), 7.37 (td, $J = 7.5$, 1.0 Hz, 1H), 7.30 (td, $J = 7.6$, 1.3 Hz, 1H), 7.13-6.96 (m, 5H), 6.83 (td, $J = 8.7$, 2.5 Hz, 1H), 6.67-6.55 (m, 2H), 4.94 (s, 1H). $^{13}$C NMR (100 MHz, CDCl₃) δ 169.9, 163.1 ($d$, $J = 245.0$ Hz), 141.7 (d, $J = 13.0$ Hz), 140.1, 138.8, 133.0, 132.9, 132.4 (d, $J = 3.0$ Hz), 130.1, 128.6, 128.4, 127.8, 126.6 (d, $J = 7.0$ Hz), 124.9, 124.8, 125.2 (q, $J = 282.0$ Hz), 112.4 (d, $J = 23.0$ Hz), 104.7 (d, $J = 27.0$ Hz), 79.2 (q, $J = 30.0$ Hz), 50.44. $^{19}$F NMR (377 MHz, CDCl₃) δ -78.8, -111.9 ppm. HRMS $m/z$ (ESI+): Calcd for C₂₂H₁₄F₄NO⁺ (M+H)⁺ 384.1006, found 384.1011.
(10bR,11S)-2-methoxy-11-phenyl-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indol-6-one (3r):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:80 (v/v); white solid (68% yield), m.p. 108-110 °C. [α]D20 = +53.4 (c 0.5, CH2Cl2), 95% ee [Daicel Chiralpak OD-H column (25 cm x 0.46 cm ID), "hexane/PrOH = 85/15, 0.6 mL/min, 280 nm; tminor = 7.8 min, tmajor = 8.7 min]. 1H NMR (400 MHz, CDCl3) δ 7.74 (dd, J = 8.1, 4.0 Hz, 2H), 7.39-7.32 (m, 1H), 7.31-7.22 (m, 1H), 7.13-6.97 (m, 4H), 6.93 (dd, J = 8.6, 2.6 Hz, 1H), 6.66 (dd, J = 9.6, 4.7 Hz, 3H), 4.92 (s, 1H), 3.73 (s, 3H). 13C NMR (125 MHz, CDCl3) δ 170.1, 157.8, 140.0, 138.7, 138.4, 134.1, 133.5, 132.5, 129.9, 128.5, 128.4, 127.6, 125.3 (q, J = 282.0 Hz), 124.8, 124.6, 117.1, 114.0, 111.7, 78.7 (q, J = 282.0 Hz), 55.6, 51.3. 19F NMR (377 MHz, CDCl3) δ -78.8, -111.9 ppm. HRMS m/z (ESI+): Calcd for C23H17F3NO2 (M+H)+ 396.1206, found 396.1210.
6 Synthetic transformations
6.1 Bromination of 2a

A reported procedure in the literature was followed for the synthesis of 4[7]. To a suspension of 2a (0.2 mmol, 99% ee) in AcOH (2 mL) was added Br₂ (160 mg, 2 mmol) at room temperature, and the mixture was stirred for 0.5 h. CH₂Cl₂ (4 mL) was then introduced via a syringe. The resulting mixture was allowed to stir at room temperature overnight. The reaction was then quenched with saturated Na₂S₂O₃ and extracted with CH₂Cl₂. The combined organic layers were washed with brine, dried over MgSO₄, and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v), to afford 4 (80% yield, 98% ee).

(R)-2-bromo-10b-(trifluoromethyl)-11-dihydro-6H-isoindolo[2,1-al]indol-6-one (4):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (80% yield), m.p. 106-108 °C. [α]D$^20$ = +171.2 (c 0.5, CH₂Cl₂), 98% ee [Daicel Chiralpak C1 column (25 cm × 0.46 cm ID), nhexane/iPrOH = 90/10, 0.6 mL/min, 280 nm; t_minor = 10.3 min, t_major = 10.7 min]. $^1$H NMR (400 MHz, CDCl₃) δ 7.91 (d, J = 7.5 Hz, 1H), 7.76-7.67 (m, 1H), 7.65-7.53 (m, 3H), 7.43 (d, J = 8.3 Hz, 1H), 7.37 (s, 1H), 3.71 (d, J = 16.7 Hz, 1H), 3.43 (d, J = 16.7 Hz, 1H). $^{13}$C NMR (100 MHz, CDCl₃) δ 169.5, 142.3, 139.9, 135.5, 134.0, 133.0, 131.3, 130.7, 128.0, 125.6, 125.1 (q, J = 282.0 Hz), 123.68, 123.61, 117.9, 78.4 (q, J = 30.0 Hz), 34.56. $^{19}$F NMR (377 MHz, CDCl₃) δ -79.1 ppm. HRMS m/z (ESI+): Calcd for C₁₆H₁₀BrF₃NO⁺ (M+H)⁺ 369.9862, found 369.9865.
6.2 Reduction of 2a

A reported procedure in the literature was followed for the synthesis of 5. A solution of 2a (0.2 mmol, 1 equiv) in THF (2 mL) was added BH$_3$·DMS (45.5 μL, 0.4
mol, 2 equiv) dropwise. The resulting mixture was refluxed for 1 hour until the starting material was all consumed. The mixture was cooled to 0 °C and MeOH (1 mL) was added. After the evolution of gas ceased, the solution was concentrated under reduced pressure, the residue was purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v), to afford 5 (94% yield, 98% ee).

(R)-10b-(trifluoromethyl)-10b,11-dihydro-6H-isoindolo[2,1-a]indole (5):

Purified by chromatography on silica gel, eluting with ethyl acetate/petroleum ether 1:50 (v/v); white solid (94% yield), m.p. 77-79 °C. [α]_D^20 = +212.0 (c 0.5, CH₂Cl₂), 98% ee [Daicel Chiralpak A2 column (25 cm × 0.46 cm ID), hexane/iPrOH = 90/10, 0.6 mL/min, 230 nm; ť_major = 6.4 min, ť_minor = 6.7 min]. ^1H NMR (400 MHz, CDCl₃) δ 7.36-7.31 (m, 1H), 7.25-7.18 (m, 2H), 7.16-7.10 (m, 1H), 7.07 (t, J = 7.6 Hz, 1H), 6.98 (d, J = 6.8 Hz, 1H), 6.79-6.68 (m, 2H), 4.72 (d, J = 14.7 Hz, 1H), 4.34 (d, J = 14.6 Hz, 1H), 3.64 (d, J = 16.4 Hz, 1H), 3.44 (d, J = 16.4 Hz, 1H). ^13C NMR (100 MHz, CDCl₃) δ 153.4, 141.0, 138.3, 129.5, 128.3, 128.0, 127.9, 126.6 (q, J = 282.0 Hz), 124.7, 123.8, 122.9, 121.5, 112.6, 80.4 (q, J = 30.0 Hz), 59.97, 37.26. ^19F NMR (377 MHz, CDCl₃) δ -79.9 ppm. HRMS m/z (ESI+): Calcd for C₁₆H₁₃F₃N⁺ (M+H)⁺ 276.0990, found 276.0993.
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7 Crystal report of compounds 2f and 3a

7.1 Crystal report of compound 2f (grown in a mixed solvent of DCM and n-hexane)

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**checkCIF/PLATON report**

Structure factors have been supplied for datablock(s) cu_210317_cjf_1_4.cf3_0m

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

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The following ALERTS were generated. Each ALERT has the format test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.
### Alert level G

**PLAT742_ALERT_2_G Low ‘MainMol’ Ueq as Compared to Neighbors of C16 Check**

**PLAT742_ALERT_2_G Low ‘MainMol’ Ueq as Compared to Neighbors of C17 Check**

**PLAT742_ALERT_2_G Short Intermol X...Y Contact 01**

1-x, -1/2-y, 1-z = 2.646 Check

**PLAT791_ALERT_4_G Model has Chirality at C7**

(Schneider Script) R Verify

**PLAT792_ALERT_3_G Percentage of Disagreement Data at Theta(Max) Still**

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**PLAT797_ALERT_4_G Missing # of PSC Reflections Above STh/L= 0.600**

2 Note

**PLAT798_ALERT_2_G Number C=C Bonds with Positive Residual Density.**

2 Info

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It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" field of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

**Publication of your CIF in IUCr journals**

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation); however, if you intend to submit to Acta Crystallographica Section C or E or IUCrData, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

**Publication of your CIF in other journals**

Please refer to the Notes for Authors of the relevant journal for any special instructions relating to CIF submission.
7.2 Crystal report of compound 3a (grown in a mixed solvent of DCM and n-hexane)

**checkCIF/PLATON report**

Structure factors have been supplied for datablock(s) cu_210316_cjf_2_bd_0m

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

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test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

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Publication of your CIF in other journals

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PLATON version of 05/12/2020; check.def file version of 05/12/2020
7. References