Supporting Information

Asymmetric Trifluoromethylation of Aromatic Aldehydes by
Cooperative Catalyst with (IPr)CuF and Quinidine-Derived
Quaternary Ammonium Salt

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1. General information

All reactions were carried out under argon atmosphere using typical vacuum-line techniques unless otherwise noted. Solvents were transferred via syringe and were introduced into the reaction vessels through a rubber septum. The $^1$H-NMR (400 MHz) spectra for solution in CDCl$_3$ and DMSO-d$_6$ were recorded on Bruker Avance 400 and Varian Mercury 400. Chemical shifts were reported downfield in ppm from tetramethylsilane (CDCl$_3$, $\delta = 7.26$; DMSO-d$_6$, $\delta = 2.50$). Spectra were reported as follows: chemical shift ($\delta$ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration and assignment. $^{13}$C NMR spectra were collected on Bruker Avance 400 and a Varian Mercury 400 (100 MHz) with complete proton decoupling. Chemical shifts were reported in ppm from the tetramethylsilane (CDCl$_3$, $\delta = 77.0$; DMSO-d$_6$, $\delta = 39.5$). The IR spectra were recorded on Thermo Scientific Nicolet iS10 with KBr pellets. Elemental analyses were performed on an Elementar Vario MICRO CUBE instrument. Enantiomeric excesses were determined by HPLC on Shimadzu LC-20A apparatus with Chiralpak OJ-H, AS-H, OD-H and AD-H. Optical rotations were measured on a Krüss P8000 polarimeter. HRMS was recorded on Bruker Apex IV FTMS. All melting points were determined on a XT4A melting point apparatus without correction. Analytical thin layer chromatography (TLC) was performed using F254 pre-coated silica gel plate. Column chromatography was performed with silica gel (200–300 mesh). Petroleum ether (PE) used had a boiling point range of 60–90 °C.
2 Preparation of Catalysts

2.1 Typical procedure for (IPr)CuF preparation

[1,3-Bis(2,6-di-iso-propylphenyl)imidazol-2-ylidene] copper (I) chloride, (IPr)CuCl

An oven-dried Schlenk flask containing 1,3-bis(2,6-di-iso-propylphenyl) imidazolium chloride (849.0 mg, 2.00 mmol), CuCl (198.0 mg, 2.00 mmol), NaOt-Bu (192.0 mg, 2.00 mmol) was evacuated and refilled with argon three times. THF (10 mL) were added to this Schlenk flask. The resulting suspension was stirred at room temperature for 4 h. Then, it was filtered through Celite in glovebox. The title compound was obtained as a white powder (788.6 mg, 81% yield). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.48 (t, $J = 8.0$ Hz, 2H), 7.29 (d, $J = 7.6$ Hz, 4H), 7.11 (s, 2H), 2.53–2.60 (m, 4H), 1.30 (d, $J = 6.8$ Hz, 12H), 1.22 (d, $J = 6.8$ Hz, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 180.6, 145.6, 134.4, 130.6, 124.2, 123.2, 28.8, 24.9, 23.9; IR (KBr) 3160, 3137, 3070, 2968, 2926, 2869, 2963, 1963, 1577, 1469, 1456, 1405, 1383, 1327, 1114, 1104, 1212, 1058, 937, 946, 809, 765, 742, 699 cm$^{-1}$.

[1,3-Bis(2,6-di-iso-propylphenyl)imidazol-2-ylidene]copper(I) tert-butoxide, (IPr)Cu(Ot-Bu)

In a glovebox, a 30 mL round-bottom flask was charged with (IPr)CuCl (969.0 mg, 2.00 mmol) and NaOt-Bu (192.0 mg, 2.00 mmol). Anhydrous THF (12.0 mL) was added. The resulting opaque brown solution was stirred for 2.0 h. It was filtered through Celite in glovebox and concentrated in vacuo affording (IPr)Cu(Ot-Bu) as an off-white powder (802.2 mg, 79% yield). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.48 (t, $J = 8.0$ Hz, 2H), 7.29 (d, $J = 7.6$ Hz, 4H), 7.11 (s, 2H), 2.53–2.60 (m, 4H), 1.30 (d, $J = 6.8$ Hz, 12H), 1.26 (s, 9H), 1.22 (d, $J = 6.8$ Hz, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 180.6, 145.6, 134.4, 130.6, 124.2, 123.2, 31.2, 28.8, 24.9, 23.9; IR (KBr) 3396, 3136, 3073, 2968, 2926, 2869, 2963, 1963, 1577, 1469, 1456, 1407, 1385, 1364, 1330, 1212, 1058, 935, 805, 758, 745 cm$^{-1}$.

[1,3-Bis(2,6-di-iso-propylphenyl)imidazol-2-ylidene]copper (I) fluoride, (IPr)CuF
In glovebox, (IPr)CuOt-Bu (1043 mg, 2.00 mmol) and benzene (18 mL) were added to a 50 mL round-bottom flask equipped with a Teflon-coated stir bar. The flask was sealed with a rubber septum and took out from the glovebox. Triethylamine tris(hydrofluoride) (110.0 μL, 0.67 mmol) was added via a syringe. The resulting white suspension was stirred for 6 h at room temperature. The solvent was removed under reduced pressure. In the glovebox, the white solid was suspended in hexane (5 mL), filtered, and washed with hexane (5 mL) to afford (IPr)CuF as a white powder (801.0 mg, 85%). ¹H NMR (400 MHz, CDCl₃): ²J 7.48 (t, ²J = 8.0 Hz, 2H), 7.29 (d, ²J = 7.6 Hz, 4H), 7.14 (s, 2H), 2.51–2.58 (m, 4H), 1.30 (d, ²J = 6.8 Hz, 12H), 1.22 (d, ²J = 6.8 Hz, 12H); ¹³C NMR (100 MHz, CDCl₃): ²J 180.5, 145.6, 134.6, 130.5, 124.2, 123.2, 28.8, 24.7, 24.0; IR (KBr) 3166, 3138, 3076, 3031, 1963, 2926, 2869, 1591, 1467, 1402, 1392, 1362, 1329, 1276, 1209, 1181, 1061, 944, 807, 766, 749, 542 cm⁻¹.

2.2 Typical procedure for ammonium bromides of cinchona alkaloids

The ammonium bromides of cinchona alkaloids were synthesized following known procedures.⁴ To a flame-dried flask equipped with a magnetic stirring bar and a condenser was added cinchona alkaloids (1 mmol), toluene (5 mL), and benzyl bromide derivatives (1.2 mmol, 1.2 equiv). The mixture was heated at 80 °C until a TLC analysis showing that the starting material was completely consumed. Cooled to room temperature and poured onto Et₂O (30 mL) with stirring, the resulting suspension was stirred for another 1 h. Then the precipitate was purified by flash chromatography.

N-(4-Methylbenzyl)quinidinium Bromide (3a)

Prepared according to the general procedure, quinidine (324.4 mg, 1.0 mmol) and 4-methylbenzyl bromide (221.1 mg, 1.2 mmol, 1.2 equiv) were stirred for 12 h. The resulting precipitate was purified
by flash chromatography (MeOH/EtOAc = 1/10, V/V) to give a white solid (407.5 mg, 80% yield). 
$[\alpha]_D^{28} = +154.3$ (c 0.35, CH$_3$OH); m.p. 240 °C (decomp.); $^1$H NMR (400 MHz, DMSO–d$_6$): δ 8.81 (d, $J = 4.4$ Hz, 1H), 8.02 (d, $J = 5.2$ Hz, 1H), 7.77 (d, $J = 4.4$ Hz, 1H), 7.60 (t, $J = 3.8$ Hz, 2H), 7.50 (dd, $J = 9.2$, 2.0 Hz, 1H), 7.44 (s, 1H), 7.38 (d, $J = 7.6$ Hz, 2H), 6.84 (s, 1H), 6.53 (s, 1H), 6.03 (ddd, $J = 17.6$, 10.0, 7.2 Hz, 1H), 5.25 (s, 1H), 5.22 (d, $J = 6.0$ Hz, 1H), 5.00 (d, $J = 12.8$ Hz, 1H), 4.72 (d, $J = 12.8$ Hz, 1H), 4.23–4.19 (m, 1H), 4.07 (s, 3H), 3.96–3.94 (m, 1H), 3.85 (t, $J = 9.2$ Hz, 1H), 3.48 (t, $J = 9.2$ Hz, 1H), 2.90 (q, $J = 9.6$ Hz, 1H), 2.66 (q, $J = 8.4$ Hz, 1H), 2.39 (s, 1H), 1.76 (t, $J = 8.8$ Hz, 2H), 1.12–1.05 (m, 1H); $^{13}$C NMR (100 MHz, DMSO–d$_6$): δ 157.9, 147.9, 144.2, 144.0, 140.4, 137.7, 134.0, 131.9, 130.1, 125.9, 125.2, 121.9, 117.5, 102.8, 67.7, 65.1, 63.6, 56.4, 56.1, 54.2, 37.2, 26.9, 23.6, 21.4, 21.1; IR (KBr) 3406, 3054, 1620, 1585, 1508, 1467, 1417, 1353, 1255, 1239, 1227, 1125, 1037, 931, 816 cm$^{-1}$. HRMS calcd for [C$_{28}$H$_{33}$N$_2$O$_2$]+: 429.2536, found 429.2540.

$\text{N-(4-Trifluoromethylbenzyl)quinidinium Bromide (3b)}$

Prepared according to the general procedure, quinidine (324.4 mg, 1.0 mmol) and 4-trifluoromethylbenzyl bromide (286.8 mg, 1.2 mmol, 1.2 equiv) were stirred for 12 h. The resulting precipitate was purified by flash chromatography (MeOH/EtOAc = 1/10, v/v) to give a white solid (490.1 mg, 87% yield). $[\alpha]_D^{28} = +184.2$ (c 0.15, CH$_3$OH); m.p. 218 °C (decomp.); $^1$H NMR (400 MHz, DMSO–d$_6$): δ 8.82 (d, $J = 4.4$ Hz, 1H), 8.02 (d, $J = 9.2$ Hz, 1H), 7.97 (dd, $J = 10.4$, 9.2 Hz, 4H), 7.77 (d, $J = 4.4$ Hz, 1H), 7.51 (dd, $J = 9.2$, 2.4 Hz, 1H), 7.44 (d, $J = 2.4$ Hz, 1H), 6.84 (d, $J = 3.6$ Hz, 1H), 6.52 (s, 1H), 6.03 (ddd, $J = 17.4$, 10.5, 6.9 Hz, 1H), 5.25 (s, 1H), 5.10 (d, $J = 8.4$ Hz, 2H), 4.85 (d, $J = 12.8$ Hz, 1H), 4.28–4.22 (m, 1H), 4.06 (s, 3H), 4.02–4.00 (m, 1H), 3.86 (t, $J = 9.4$ Hz, 1H), 3.50 (t, $J = 11.4$ Hz, 1H), 3.00–2.90 (m, 1H), 2.69–2.63 (m, 1H), 2.40 (t, $J = 11.4$ Hz, 1H), 1.91 (s, 1H), 1.79–1.75 (m, 2H), 1.15–1.07 (m, 1H); $^{13}$C NMR (100 MHz, DMSO–d$_6$): δ158.0, 147.9, 144.2, 143.8, 137.7, 135.1, 133.0, 131.9, 130.9 (q, $J = 31.8$ Hz), 126.4, 126.3, 125.9, 123.1, 121.7, 120.8, 117.5, 103.0, 68.1, 65.2, 62.9, 56.5, 56.2, 54.5, 37.2, 26.8, 23.6, 21.1; IR (KBr) 3398, 3209, 2954, 1754, 1594, 1589, 1509, 1533, 1255, 1239, 1227, 1125, 1037, 928, 931, 816 cm$^{-1}$. HRMS calcd for [C$_{28}$H$_{33}$N$_2$O$_2$]+: 429.2536, found 429.2540.

$\text{N-(9-Anthracenylmethyl)quinidinium Chloride (3c)}$

Prepared according to the general procedure, quinidine (324.4 mg, 1.0 mmol) and 9-anthracenylmethyl
chloride (272.1 mg, 1.2 mmol, equiv) were stirred for 12 h. The resulting precipitate was purified by flash chromatography (MeOH/EtOAc = 1/10, V/V) to give a light yellow solid (457.5 mg, 80% yield). $\left[\alpha\right]_D^{28} = +390.0$ (c 0.12, CH$_3$OH); m.p. 161 °C (decomp.) (lit.$^5$ m.p. 160 °C, decomp.); $^1$H NMR (400 MHz, DMSO–d$_6$): $\delta$ 8.98 (s, 1H), 8.86 (d, $J = 4.4$ Hz, 1H), 8.79 (d, $J = 9.2$ Hz, 1H), 8.70 (d, $J = 9.2$ Hz, 1H), 8.28 (dd, $J = 8.2$, 3.0 Hz, 2H), 8.05 (d, $J = 9.2$ Hz, 1H), 7.90 (d, $J = 4.4$ Hz, 1H), 7.82–7.74 (m, 3H), 7.69–7.64 (m, 3H), 7.53 (dd, $J = 7.6$, 2.4 Hz, 1H), 6.98 (s, 1H), 6.33 (d, $J = 14.4$ Hz, 1H), 6.03 (ddd, $J = 17.2$, 10.2, 7.2 Hz, 1H), 5.88 (d, $J = 14.0$ Hz, 1H), 5.18 (d, $J = 10.4$ Hz, 1H), 5.08 (d, $J = 17.2$ Hz, 1H), 4.46 (t, $J = 9.2$ Hz, 2H), 4.21 (s, 4H), 3.18 (t, $J = 11.2$ Hz, 1H), 2.62–2.54 (m, 1H), 2.46–2.35 (m, 2H), 1.78 (s, 1H), 1.69 (d, $J = 8.4$ Hz, 1H), 1.56–1.53 (m, 1H), 1.10–1.04 (m, 1H); $^{13}$C NMR (100 MHz, DMSO–d$_6$): $\delta$ 157.9, 147.9, 144.3, 137.9, 133.5, 133.3, 132.5, 131.8, 131.7, 131.6, 130.2, 128.3, 128.0, 126.1, 125.3, 125.0, 120.5, 119.3, 117.5, 115.5, 103.2, 67.9, 65.7, 56.5, 56.1, 55.8, 55.6, 37.7, 26.1, 24.2, 21.6; IR (KBr) 3394, 3183, 1621, 1508, 1458, 1473, 1431, 1258, 1240, 1227, 1029, 922, 864, 744 cm$^{-1}$.

$N$-(2,4,5-Trifluorobenzyl)quinidinium Bromide (3d)

Prepared according to the general procedure, quinidine (324.4 mg, 1.0 mmol) and 2,4,5-trifluorobenzyl bromide (158.8 μL, 1.2 mmol, 1.2 equiv) were stirred for 12 h. The resulting precipitate was purified by flash chromatography (MeOH/EtOAc = 1/10, V/V) to give a white solid (379.1 mg, 69% yield). $\left[\alpha\right]_D^{28} = +194.8$ (c 0.19, CH$_3$OH); m.p. 182–185 °C (decomp.); $^1$H NMR (400 MHz, DMSO–d$_6$): $\delta$ 8.82 (d, $J = 4.4$ Hz, 1H), 8.16–8.10 (m, 1H), 8.02 (d, $J = 9.2$ Hz, 1H), 7.91–7.85 (m, 1H), 7.76 (d, $J = 4.4$ Hz, 1H), 7.50 (dd, $J = 9.6$, 2.8 Hz, 1H), 7.40 (d, $J = 2.4$ Hz, 1H), 6.84 (d, $J = 2.8$ Hz, 1H), 6.50 (s, 1H), 6.03 (ddd, $J = 17.2$, 10.4, 6.8 Hz, 1H), 5.26–5.21 (m, 2H), 5.01 (d, $J = 12.8$ Hz, 1H), 4.77 (d, $J = 13.6$ Hz, 1H), 4.23–4.18 (m, 1H), 4.06 (s, 3H), 3.94–3.82 (m, 2H), 3.49 (t, $J = 11.4$ Hz, 1H), 3.22–3.15 (m, 1H), 2.67–2.60 (m, 1H), 2.36 (t, $J = 11.8$ Hz, 1H), 1.90 (s, 1H), 1.84–1.75 (m, 2H), 1.10–1.05 (m, 1H); $^{13}$C NMR (100 MHz, DMSO–d$_6$): $\delta$ 158.0 (ddd, $J = 244.1$, 9.9, 2.3 Hz), 157.9, 151.5 (dt, $J = 251.2$, 13.6 Hz), 147.9, 146.7 (ddd, $J = 241.8$, 12.6, 2.4 Hz), 144.2, 143.7, 137.7, 131.9, 125.9, 123.8 (dd, $J = 19.1$, 2.7 Hz), 122.0, 120.7, 117.6, 112.8 (dt, $J = 17.0$, 5.6 Hz), 107.5 (dd, $J = 29.5$, 21.4 Hz), 102.7, 68.0, 65.3, 56.4, 56.1, 56.0, 54.7, 37.5, 26.6, 23.7, 21.0; IR (KBr) 3394, 3198, 3006, 1621, 1520, 1473, 1431, 1258, 1362, 1240, 1227, 1029, 922, 864, 744 cm$^{-1}$; HRMS calcd for [C$_{27}$H$_{28}$F$_3$N$_2$O$_2$]$^+$: 469.2097, found 469.2098.

$N$-(3,5-Ditrifluoromethylbenzyl)quinidinium Bromide (3e)

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Prepared according to the general procedure, quinidine (324.4 mg, 1.0 mmol) and 3,5-ditrifluoromethylbenzyl bromide (368.4 mg, 1.2 mmol, 1.2 equiv) were stirred for 12 h. The resulting precipitate was purified by flash chromatography (MeOH/EtOAc = 1/10, V/V) to give a white solid (536.7 mg, 85% yield). \( [\alpha]^2_D = +176.1 \) (c 0.19, CH\(_3\)OH); m.p. 177 °C (decomp.); \(^1\)H NMR (400 MHz, DMSO–d\(_6\)): \( \delta \) 8.82 (d, \( J = 4.8 \) Hz, 1H), 8.56 (s, 2H), 8.38 (s, 1H), 8.04 (d, \( J = 9.2 \) Hz, 1H), 7.77 (d, \( J = 4.4 \) Hz, 1H), 7.53 (dd, \( J = 7.2, 2.4 \) Hz 1H), 7.44 (d, \( J = 2.4 \) Hz, 1H), 6.78 (d, \( J = 3.2 \) Hz, 1H), 6.48 (s, 1H), 6.04 (ddd, \( J = 17.4, 10.2, 7.2 \) Hz, 1H), 5.28 (d, \( J = 2.8 \) Hz, 1H), 5.22 (d, \( J = 12.4 \) Hz, 2H), 5.01 (d, \( J = 12.8 \) Hz, 1H), 4.34 (t, \( J = 10.0 \) Hz, 1H), 4.10–4.13 (m, 1H), 4.06 (s, 3H), 3.80 (m, t, \( J = 9.4 \) Hz, 1H), 3.48 (t, \( J = 11.4 \) Hz, 1H), 3.04 (q, \( J = 9.6 \) Hz, 1H), 2.62 (q, \( J = 8.4 \) Hz, 1H), 2.42 (t, \( J = 11.6 \) Hz, 1H), 1.91 (s, 1H), 1.85–1.72 (m, 2H), 1.20–1.13 (m, 1H); \(^{13}\)C NMR (100 MHz, DMSO–d\(_6\)): \( \delta \) 158.1, 147.9, 144.2, 143.7, 137.8, 135.1, 132.0, 131.7, 131.3 (q, \( J = 33.1 \) Hz), 130.1, 126.0, 125.0, 124.6 (q, \( J = 4.1 \) Hz), 123.7 (q, \( J = 271.3 \) Hz), 121.5, 120.9, 117.6, 103.1, 68.4, 65.2, 61.8, 56.3, 56.2, 54.7, 37.4, 26.9, 23.6, 21.1; IR (KBr) 3394, 3201, 2954, 2664, 1622, 1509, 1473, 1432, 1374, 1281, 1214, 1178, 1135, 1027, 1005, 866, 905, 843, 828, 709, 682 cm\(^{-1}\).

\( N-(3,5\text{-Ditrifluoromethylbenzyl})\text{quinidinium Fluoride (3f)}^8 \)

The column of Amberlyst A-26 (OH\(^-\) form, 500 mg) was washed with methanol. A solution of the \( N-(3,5\text{-ditrifluoromethylbenzyl})\text{quinidinium bromide (126.2 mg, 0.2 mmol) in methanol (5 mL) was slowly passed through the column and the column then washed with methanol. The eluent was neutralized until pH = 7 with HF and the solvents were removed in vacuo. The residue was coevaporated with toluene three times and dried under vacuum overnight and the chiral ammonium fluoride was used without further purification.} \( [\alpha]^2_D = +127.2 \) (c 0.14, CH\(_3\)OH); m.p. 190 °C (decomp.); \(^1\)H NMR (400 MHz, DMSO–d\(_6\)): \( \delta \) 8.83 (d, \( J = 4.4 \) Hz, 1H), 8.53 (s, 2H), 8.38 (s,1H), 8.05 (d, \( J = 9.2 \) Hz, 1H), 7.78 (d, \( J = 4.4 \) Hz, 1H), 7.55 (dd, \( J = 9.2, 2.4 \) Hz, 1H), 7.44 (d, \( J = 2.4 \) Hz, 1H), 6.75 (d, \( J = 2.8 \) Hz, 1H), 6.46 (s, 1H), 6.05 (dd, \( J = 17.6, 10.4, 7.2 \) Hz, 1H), 5.24–5.29 (m, 2H), 5.14 (d, \( J = 12.8 \) Hz, 1H), 4.97 (d, \( J = 12.8 \) Hz, 1H), 4.32 (t, \( J = 9.2 \) Hz, 1H), 4.05 (s, 3H), 4.03 (t, \( J = 12.0 \) Hz, 1H), 3.78 (t, \( J = 10.0 \) Hz, 1H), 3.49 (t, \( J = 11.4 \) Hz, 1H), 3.13–2.99 (m, 1H), 2.62 (q, \( J = 2.4 \) Hz, 1H), 2.43 4.34 (t, \( J = 11.4 \) Hz, 1H), 1.92 (s, 1H), 1.83–1.76 (m, 2H), 1.46–1.20 (m, 1H); \(^{13}\)C NMR (100 MHz, DMSO–d\(_6\)): \( \delta \) 158.1, 147.9, 144.1, 143.8, 137.7, 135.1, 132.0, 131.7, 131.3 (q, \( J = 33.1 \) Hz), 126.0, 124.7 (q, \( J = 19.0 \) Hz), 123.7 (q, \( J = 271.8 \) Hz), 121.4, 120.9, 117.6, 103.2, 68.5, 65.3, 61.9, 56.2, 54.8,
N-(3,3''',5,5''''-tetrakis(trifluoromethyl)-1,1':3',1'''-terbenzyl)quinidinium Bromide (3g)

Prepared according to the general procedure, quinidine (324.4 mg, 1.0 mmol) and 3,3''',5,5''''-tetrakis(trifluoromethyl)-1,1':3',1'''-terbenzyl bromide (714.0 mg, 1.2 mmol, 1.2 equiv) were stirred for 12 h. The resulting precipitate was purified by flash chromatography (MeOH/EtOAc = 1/10, V/V) to give a white solid (919.4 mg, 71% yield). [α]D28 = +129.5 (c 0.14, CH3OH); m.p. 186 °C (decomp.); 1H NMR (400 MHz, CDCl3): δ 8.39 (d, J = 4.4 Hz, 1H), 8.29 (s, 2H), 8.00 (s, 4H), 7.84 (s, 2H), 7.69 (d, J = 6.0 Hz, 2H), 7.60 (s, 1H), 6.94 (d, J = 8.8 Hz, 1H), 6.71 (d, J = 5.6 Hz, 1H), 6.44 (s, 1H), 6.00 (s, 2H), 5.88 (ddd, J = 17.0, 10.2, 7.0 Hz, 1H), 5.23–5.17 (m, 2H), 4.66 (t, J = 10.0 Hz, 1H), 4.36 (t, J = 10.0 Hz, 1H), 3.77 (s, 3H), 3.34 (t, J = 11.2 Hz, 1H), 2.98 (q, J = 10.0 Hz, 1H), 2.28–2.43 (m, 2H), 1.82 (s, 1H), 1.78 (d, J = 8.4 Hz, 2H), 0.90–0.83 (m, 1H); 13C NMR (100 MHz, DMSO–d6): δ 157.8, 147.0, 143.9, 142.1, 139.8, 135.1, 133.1, 132.5 (q, J = 33.4 Hz), 131.5, 130.2, 127.3, 127.2, 127.1, 126.0, 123.1 (q, J = 272.0 Hz), 122.1 (q, J = 3.4 Hz), 120.4, 120.1, 118.4, 102.8, 68.2, 66.8, 61.5, 56.9, 56.3, 54.5, 38.1, 27.1, 24.0, 21.7; IR (KBr) 3402, 3196, 2946, 1711, 1621, 1509, 1432, 1280, 1239, 1126, 1134, 1029, 1002, 900, 885, 844, 827, 718, 704, 683, 640 cm–1.

N-(3,5-Ditrifluoromethylbenzyl)quininium Bromide (4)

Prepared according to the general procedure, quinine (324.4 mg, 1.0 mmol) and 3,5-ditrifluoromethylbenzyl bromide (368.4 mg, 1.2 mmol, 1.2 equiv) were stirred for 12 h. The resulting precipitate was purified by flash chromatography (MeOH/EtOAc = 1/10, V/V) to give a white solid (372.3 mg, 59% yield). [α]D28 = –164.5 (c 0.15, CH3OH); m.p. 192 °C (decomp.); 1H NMR (400 MHz, DMSO–d6): δ 8.82 (d, J = 4.4 Hz, 1H), 8.52 (s, 2H), 8.37 (s, 1H), 8.04 (d, J = 9.2 Hz, 1H), 7.76 (d, J = 4.4 Hz, 1H), 7.52 (ddd, J = 9.2, 2.4 Hz, 1H), 7.40 (d, J = 2.8 Hz, 1H), 6.71 (d, J = 4.4 Hz, 1H), 6.56 (d, J = 4.4 Hz, 1H), 5.77 (ddd, J = 17.4, 10.5, 6.8 Hz, 1H), 5.67 (d, J = 12.4 Hz, 1H), 5.15 (d, J = 17.2 Hz, 1H), 5.03 (d, J = 4.4 Hz, 1H), 5.00 (d, J = 6.0 Hz, 1H), 4.41 (t, J = 11.2 Hz, 1H), 4.04 (s, 3H), 3.82 (s, 3H), 3.44 (t, J = 11.2 Hz, 1H), 2.98 (q, J = 10.0 Hz, 1H), 2.28–2.43 (m, 2H), 1.82 (s, 1H), 1.75 (d, J = 8.4 Hz, 2H), 0.90–0.83 (m, 1H); 13C NMR (100 MHz, DMSO–d6): δ 157.8, 147.0, 143.9, 142.1, 139.8, 135.1, 133.1, 132.5 (q, J = 33.4 Hz), 131.5, 130.2, 127.3, 127.2, 127.1, 126.0, 123.1 (q, J = 272.0 Hz), 122.1 (q, J = 3.4 Hz), 120.4, 120.1, 118.4, 102.8, 68.2, 66.8, 61.5, 56.9, 56.3, 54.5, 38.1, 27.1, 24.0, 21.7; IR (KBr) 3402, 3196, 2946, 1711, 1621, 1509, 1432, 1280, 1239, 1126, 1134, 1029, 1002, 900, 885, 844, 827, 718, 704, 683, 640 cm–1.
3.83 (dd, \( J = 16.8, 10.0 \) Hz, 2H), 3.47 (t, \( J = 11.4, 1H \)), 3.30–3.23 (m, 1H), 2.67–2.65 (m, 1H),
2.28–2.24 (m, 1H), 2.19–2.12 (m, 1H), 2.03–1.99 (m, 1H), 1.82 (t, \( J = 10 \) Hz, 1H), 1.47–1.53 (m, 1H);
\(^{13}\)C NMR (100 MHz, DMSO–d\(_6\)): \( \delta = 158.1, 147.9, 144.2, 144.0, 138.5, 135.0, 132.0, 131.7, 131.3 \) (q, \( J = 33.0 \) Hz), 125.9, 124.7 (q, \( J = 3.4 \) Hz), 123.6 (q, \( J = 272.0 \) Hz), 121.8, 120.8, 117.2, 102.5, 69.1, 64.1, 61.8, 59.7, 56.3, 51.2, 37.7, 26.6, 24.7, 21.0; IR (KBr) 3406, 3218, 2983, 1706, 1622, 1509, 1473, 1432, 1374, 1280, 1241, 1178, 1137, 1028, 904, 843, 827, 683 cm\(^{-1}\).

1,3-Bis(quinidinium-N-methylene)benzene Dibromide (5)

A mixture of quinidine (324.4 mg, 1.0 mmol) with \(\alpha\),\(\alpha'\)-dibromo-m-xylene (132.0 mg, 0.5 mmol) in a
mixture of ethanol (1 mL), DMF (1.2 mL), and chloroform (0.4 mL) was stirred at 100 °C for 8 h. After
cooling the reaction mixture to room temperature, the reaction mixture was diluted with methanol (40
mL) and then added to ether (200 mL) dropwise with stirring. The solid precipitated was filtered. The
resulting precipitate was purified by flash chromatography (MeOH/EtOAc = 1/10, V/V) to give a white
solid (456.4 mg, 75% yield). \([\alpha]_D^{28} = +197.2 \) (c 0.14, CH\(_3\)OH); m.p. 218 °C (decomp.); \(^1\)H NMR (400
MHz, DMSO–d\(_6\)): \( \delta = 8.82 \) (d, \( J = 4.0 \) Hz, 2H), 8.07 (s, 1H), 8.03 (d, \( J = 8.8 \) Hz, 2H), 7.90 (d, \( J = 7.6 \) Hz,
2H), 7.78 (d, \( J = 4.4 \) Hz, 2H), 7.74 (d, \( J = 7.6 \) Hz, 1H), 7.52 (d, \( J = 2.0 \) Hz, 1H), 7.50 (d, \( J = 2.0 \) Hz,
1H), 7.44 (d, \( J = 2.0 \) Hz, 2H), 6.83 (d, \( J = 3.2 \) Hz, 2H), 6.56 (s, 2H), 6.03 (ddd, \( J = 16.8, 10.4, 6.8 \) Hz,
2H), 5.22–5.12 (m, 6H), 4.82 (d, \( J = 12.4 \) Hz, 2H), 4.24–4.19 (m, 2H), 4.09–3.99 (m, 8H), 3.85–3.80
(m, 2H), 3.53–3.47 (m, 2H), 3.27–3.22 (m, 2H), 2.77–2.70 (m, 2H), 2.42 (t, \( J = 11.4 \) Hz, 2H), 1.91 (s,
2H), 1.76–1.74 (m, 4H), 1.16–1.12 (m, 2H); \(^{13}\)C NMR (100 MHz, DMSO–d\(_6\)): \( \delta = 158.0, 147.9, 144.2,
143.9, 139.2, 137.7, 135.7, 131.9, 130.2, 129.0, 125.9, 121.8, 120.9, 117.5, 102.8, 68.0, 65.1, 63.3, 56.2,
54.1, 37.3, 26.9, 23.5, 21.1; IR (KBr) 3386, 2951, 2361, 1621, 1590, 1509, 1473, 1459, 1431, 1358,
1241, 1227, 1207, 1026, 1001, 934, 866, 828, 717, 855 cm\(^{-1}\).

N-(3,5-Ditrifluoromethyl)benzyl-O(9)-3,5-ditrifluoromethylbenzyl)quinidinium Bromide (6)

To a suspension of \( N \)-(3,5-ditrifluoromethylbenzyl)quinidinium bromide (632.5 mg, 1.0 mmol) in
dichloromethane (3 mL) was added 3,5-ditrifluoromethylbenzyl bromide (920.0 \( \mu \)L, 5.0 mmol) and
50% aqueous KOH (560.0 \( \mu \)L, 5.0 mmol). The resulting mixture was stirred vigorously at room
temperature for 4 h. The mixture was diluted with water (5 mL) and extracted with dichloromethane (3
x 20 mL). The combined organic extracts were dried over Na₂SO₄, filtered and concentrated in vacuo.

The crude solid was purified by flash chromatography (MeOH/EtOAc = 1/10, V/V) to give a white solid (540.3 mg, 63% yield). \([\alpha]_D^{28} = +78.1 \text{ (c 0.31, CH₂Cl₂)}\); m.p. 150 °C (decomp.); \(^1\)H NMR (400 MHz, DMSO–d₆): \(\delta 8.80 \text{ (d, } J = 2.8 \text{ Hz, 1H), 8.53} \text{ (s, 2H), 8.39} \text{ (s, 1H), 8.27} \text{ (s, 2H), 8.09} \text{ (s, 1H), 8.04} \text{ (d, } J = 9.2 \text{ Hz, 1H), 7.75} \text{ (d, } J = 1.2 \text{ Hz, 1H), 7.56–7.48} \text{ (m, 2H), 6.57} \text{ (s, 1H), 6.01–5.96} \text{ (m, 1H), 5.19–5.02} \text{ (m, 5H), 4.68} \text{ (d, } J = 12.0 \text{ Hz, 1H), 4.07} \text{ (s, 4H), 3.86–3.83} \text{ (m, 1H), 3.58–3.52} \text{ (m, 1H), 3.00–2.93} \text{ (m, 1H), 2.64} \text{ (d, } J = 9.2 \text{ Hz, 2H), 1.98} \text{ (s, 1H), 1.81–1.74} \text{ (m, 2H),1.45–1.42} \text{ (m, 1H), 0.86–0.74} \text{ (m, 1H); } ^{13}\text{C NMR (100 MHz, CDCl}₃): \(\delta 184.6, 158.8, 146.7, 146.6, 146.5, 145.0, 141.3, 139.4, 137.8, 134.9, 134.5, 134.4, 134.3, 132.5 \text{ (q, } J = 33.0 \text{ Hz), 131.6, 130.2, 128.2} \text{ (q, } J = 33.0 \text{ Hz), 124.6, 124.5, 124.4, 123.1} \text{ (q, } J = 271.7 \text{ Hz), 122.7} \text{ (q, } J = 271.9 \text{ Hz), 122.3} \text{ (q, } J = 2.8 \text{ Hz), 118.2, 101.7, 73.1, 69.4, 67.5, 61.5, 61.4, 56.1, 55.1, 37.7, 26.9, 23.6, 21.9, 11.0; IR (KBr) 3420, 2961, 1622, 1508, 1475, 1433, 1372, 1280, 1241, 1227, 1177, 1135, 1030, 902, 843, 828, 707, 683 \text{ cm}^{-1}; \) HRMS calcd for [C₃₈H₃₃F₁₂N₂O₂]⁺: 777.2344, found 777.2331.

\(N-(3,5\text{-Ditrifluoromethyl)benzyl)-6’-hydroxyquinidinium Bromide (7)\)

Ethanethiol (2.30 mL, 30.8 mmol) was added under argon atmosphere to a stirred suspension of sodium hydride (370.0 mg, 15.4 mmol) in dry DMF (15 ml). Quinidine (500 mg, 1.5mmol) in dry DMF (7.5 mL) was added dropwise and the reaction mixture was stirred at 110 °C for 13 h. The solvent and excess ethanethiol were removed under reduced pressure. Then the 3,5-ditrifluoromethylbenzyl bromide (675.4 mg, 2.2 mmol) was added in THF (9 mL). The reaction mixture was refluxed and monitored by TLC analysis. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (MeOH/EtOAc = 1/20, V/V). The product was obtained as pale white solid (481.6 mg, 52 %). \([\alpha]_D^{28} = +182.3 \text{ (c 0.16, CH₃OH)}; \) m.p. 258 °C (decomp.); \(^1\)H NMR (400 MHz, DMSO–d₆): \(\delta 10.06 \text{ (s, 1H), 8.75} \text{ (d, } J = 4.4 \text{ Hz, 1H), 8.63} \text{ (s, 2H), 8.37} \text{ (s, 1H), 7.95} \text{ (d, } J = 8.8 \text{ Hz, 1H), 7.70} \text{ (d, } J = 4.4 \text{ Hz, 1H), 7.67} \text{ (d, } J = 2.4 \text{ Hz, 1H), 7.38} \text{ (dd, } J = 9.0 \text{ Hz, 2H), 6.68} \text{ (d, } J = 3.6 \text{ Hz, 1H), 6.32} \text{ (s, 1H), 6.02} \text{ (ddd, } J = 17.4, 10.5, 6.9 \text{ Hz, 1H), 5.38} \text{ (d, } J = 12.4 \text{ Hz, 1H), 5.26–5.19} \text{ (m, 3H), 4.32} \text{ (t, } J = 9.6 \text{ Hz, 1H), 4.13} \text{ (t, } J = 9.6 \text{ Hz, 1H), 3.92} \text{ (t, } J = 9.6 \text{ Hz, 1H), 3.49} \text{ (t, } J = 11.2 \text{ Hz, 1H), 3.12–3.05} \text{ (m, 1H), 2.65–2.59} \text{ (m, 1H), 2.33} \text{ (t, } J = 11.6 \text{ Hz, 1H), 1.89} \text{ (s, 1H), 1.83–1.78} \text{ (m, 2H), 1.17–1.09} \text{ (m, 1H); } ^{13}\text{C NMR (100 MHz, DMSO–d₆): } \delta 156.5, 147.2, 143.4, 143.1, 137.7, 135.2, 131.9, 131.7, 131.2 \text{ (q, } J = 33.0 \text{ Hz), 126.1, 124.6} \text{ (q, } J = 3.2 \text{ Hz), 123.7} \text{ (q, } J = 271.8 \text{ Hz), 122.2, 120.4, 117.6, 105.1, 68.2, 65.3, 60.9, 56.3, 54.7, 37.3, 26.9, 23.6, 21.0; \) IR (KBr) 3369, 3234, 1622, 1531, 1469, 1217, 1181, 1135, 1003, 927, 905, 864, 736, 736, 709, 683 \text{ cm}^{-1}; \) HRMS calcd for [C₂₈H₂₇F₆N₂O₂]⁺: 537.1971, found 537.1959.

\(N-(3,5\text{-Ditrifluoromethyl)benzyl)-9-O-benzyl-6’-hydroxyquinidinium Bromide (8)\)
Sodium hydride (96.0 mg, 4.0 mmol) was added to a solution of quinidine (324.4 mg, 1.0 mmol) in dry DMF (5 mL). Benzyl chloride (173 μL, 1.5 mmol) was added dropwise. The reaction mixture was stirred at room temperature for 20 h and quenched by water. The aqueous phase was extracted with ethyl acetate. The organic layer was washed with brine, dried over Na₂SO₄, concentrated in vacuo to afford yellowish oil, which was used without purification. Ethanethiol (434.0 μL, 5.8 mmol) was added to a stirred suspension of sodium hydride (139.3 mg, 5.8 mmol) in dry DMF (3 mL). The yellowish oil (300 mg) in dry DMF (3 mL) was added dropwise and the reaction mixture was stirred at 110 °C for 15 h. The solvent and excess ethanethiol were removed under reduced pressure. The crude product was added the 3,5-ditrifluoromethylbenzyl bromide (336.2 mg, 1.1 mmol) in THF (6 mL). The reaction mixture was refluxed and monitored by TLC analysis. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (MeOH/EtOAc = 1/20, V/V). The product was obtained as pale white solid (240.9 mg, 46 %). 

\[ \alpha \text{D}^{2} = +68.9 \ (c \ 0.33, \text{CH}_3\text{OH}) \]; m.p. 246 °C (decomp.); \(^1\)H NMR (400 MHz, CDCl₃): δ 9.26 (s, 1 H), 8.83 (d, J = 3.6 Hz, 1H), 8.27 (s, 1H), 8.05 (d, J = 9.2 Hz, 2H), 7.97 (s,1H), 7.72–7.70 (m, 1H), 7.58–7.39 (m, 7H), 6.34 (d, J = 12.0 Hz, 1H), 6.05 (s, 1H), 5.93 (ddd, J = 17.0, 10.0, 7.2 Hz, 1H), 5.37 (d, J = 10.4 Hz, 1H), 5.24–5.13 (m, 2H), 5.07 (d, J = 12.4 Hz, 1H), 4.53 (t, J = 9.4 Hz, 1H), 4.33–4.29 (m, 2H), 4.16–4.11 (m, 1H), 3.75 (d, J = 12.0 Hz, 1H), 3.06 (t, J = 11.4 Hz, 1H), 2.75–2.67 (m, 1H), 2.51–2.36 (m, 2H), 2.02 (s, 1H), 1.95 (t, J = 10.4 Hz, 1H), 1.71–1.12 (m, 1H); \(^1^3\)C NMR (100 MHz, CDCl₃): δ 167.7, 157.4, 146.4, 146.4, 144.1, 136.7, 135.8, 134.9, 134.0, 132.6 (q, J = 34.3 Hz), 132.3, 131.9, 130.9, 129.9, 129.8, 129.5, 129.4, 128.9, 125.9, 123.8, 122.7 (q, J = 274.2 Hz), 118.7, 104.8, 73.5, 72.0, 66.5, 59.4, 56.1, 54.4, 37.7, 27.0, 23.5, 21.7; IR (KBr) 3419, 3113, 2958, 2921, 2357, 1727, 1618, 1465, 1371, 1270, 1232, 1186, 1143, 1026, 903, 752, 706, 682 cm⁻¹; HRMS calcd for [C₃₅H₃₃F₆N₂O₂]⁺: 627.2440, found 627.2434.

3 General procedure for the enantioselective trifluoromethylation reaction of aldehydes

To a stirred solution of (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%) and N-(3,5-ditrifluoromethylbenzyl)quinidinium bromide (3e) (2.6 mg, 0.004 mmol, 2 mol%) in toluene (0.6 mL) was added aryl
aldehydes (1a–p 0.2 mmol) at –78 °C under argon atmosphere. Then this mixture was kept at –78 °C for 10 min. After that, Me$_3$SiCF$_3$ (60 μL, 0.4 mmol, 2 equiv) was added dropwise. After stirring for 1–2 h at –78 °C, the reaction was quenched with water. Aqueous layer was extracted with EtOAc (15 mL x 3), and the combined organic layers was washed with brine, dried over Na$_2$SO$_4$ and concentrated under reduced pressure. Then the crude product trimethylsilyl ether was treated with n-Bu$_4$NF (0.2 mL, 1 M in THF, 0.2 mmol) in THF (2.0 mL) at room temperature until all protected intermediate converted to final product. The resulting mixture was concentrated under reduced pressure, and purified by column chromatography on silica gel (PE/EtOAc = 20/1, V/V) to give trifluoromethylated alcohols 2a–p.

4. Analytical and spectral characterization data for the trifluoromethyl alcohols (2a–p)

2,2,2-trifluoro-1-(naphthalen-2-yl)ethanol (2a)

The reaction of 1a (31.2 mg, 0.20 mmol), Me$_3$SiCF$_3$ (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 1 h gave 2a as a white solid (40.7 mg, 90%) by flash chromatography (PE/EtOAc = 20/1). m.p. 76–78 °C; [α]$_D^{25} = –24.7$ (c 0.21, CH$_2$Cl$_2$) [lit.10 [α]$_D^{20} = –23.7$ (c 0.16, CH$_2$Cl$_2$, 71% ee)]; $^1$H NMR (400 MHz, CDCl$_3$): δ 7.96–7.86 (m, 4H), 7.59–7.51 (m, 3H), 5.19 (m, 1H), 2.77 (d, $J = 4.4$ Hz, 1H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): δ 133.8, 132.9, 131.3, 128.5, 128.3, 127.8, 127.4, 126.9, 126.6, 124.4 (q, $J = 281.2$ Hz), 124.3, 73.0 (q, $J = 31.9$ Hz) ppm; IR (KBr) 3374, 3062, 1602, 1507, 1341, 1247, 1262, 1193, 1124, 1085, 822, 792, 763, 752, 702, 486 cm$^{-1}$. The ee was determined by HPLC on Chiralpak OJ-H (n-hexane/i-PrOH = 80/20, 1.0 mL/min, 254 nm), $t_R$(major) = 10.7 min, $t_S$(minor) = 15.4 min, 75% ee.
The reaction of 1b (27.2 μL, 0.20 mmol), Me₃SiCF₃ (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 2 h gave 2b as a colorless oil (39.8 mg, 88%) by flash chromatography (PE/EtOAc = 20/1). [α]D²⁵ = −14.6 (c 0.47, CH₂Cl₂); ¹H NMR (400 MHz, CDCl₃): δ 8.06 (d, J = 8.4 Hz, 1 H), 7.93–7.91 (m, 2H), 7.84 (d, J = 7.2 Hz, 1H), 7.60–7.53 (m, 3H), 5.90 (m, 1H), 2.78 (d, J = 4.0 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 131.1, 130.3, 129.1, 126.1, 125.8, 125.2, 124.7 (q, J = 281.7 Hz), 122.8, 69.0 (q, J = 32.0 Hz) ppm; IR (KBr) 3421, 3046, 2927, 2847, 1515, 1399, 1355, 1264, 1167, 1126, 1093, 1030, 877, 781, 698, 632, 538 cm⁻¹. The ee was determined by HPLC on Chiralpak AS-H (n-hexane/i-PrOH = 98/2, 1.0 mL/min, 254 nm), tᵣₘ (major) = 16.7 min, tᵣₘ (minor) = 19.8 min, 60% ee.

2,2,2-trifluoro-1-(naphthalen-1-yl)ethanol (2b)¹¹

The reaction of 1b (27.2 μL, 0.20 mmol), Me₃SiCF₃ (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 2 h gave 2b as a colorless oil (39.8 mg, 88%) by flash chromatography (PE/EtOAc = 20/1). [α]D²⁵ = −14.6 (c 0.47, CH₂Cl₂); ¹H NMR (400 MHz, CDCl₃): δ 8.06 (d, J = 8.4 Hz, 1 H), 7.93–7.91 (m, 2H), 7.84 (d, J = 7.2 Hz, 1H), 7.60–7.53 (m, 3H), 5.90 (m, 1H), 2.78 (d, J = 4.0 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 131.1, 130.3, 129.1, 126.1, 125.8, 125.2, 124.7 (q, J = 281.7 Hz), 122.8, 69.0 (q, J = 32.0 Hz) ppm; IR (KBr) 3421, 3046, 2927, 2847, 1515, 1399, 1355, 1264, 1167, 1126, 1093, 1030, 877, 781, 698, 632, 538 cm⁻¹. The ee was determined by HPLC on Chiralpak AS-H (n-hexane/i-PrOH = 98/2, 1.0 mL/min, 254 nm), tᵣₘ (major) = 16.7 min, tᵣₘ (minor) = 19.8 min, 60% ee.
The reaction of 1c (20.3 μL, 0.20 mmol), Me3SiCF3 (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) at −20°C for 2 h gave 2c as a colorless oil (28.1 mg, 88%) by flash chromatography (PE/EtOAc = 20/1). \([\alpha]_D^{25} = -13.1 (c \ 0.28, CH_2Cl_2) \text{[lit.10} \ [\alpha]_D^{20} = -12.5 (c \ 0.40, CH_2Cl_2, 56\% \text{ee})\]; ^1H NMR (400 MHz, CDCl3): 67.48–7.47 (m, 2H), 7.42–7.40 (m, 3H), 5.10 (q, J = 6.6 Hz, 1H), 2.73 (s, 1H) ppm; ^13C NMR (100 MHz, CDCl3): δ 134.0, 129.6, 128.7, 127.5, 124.3 (q, J = 281.0 Hz), 72.9 (q, J = 32.0 Hz) ppm; IR (KBr) 3410, 2927, 2851, 2360, 2342, 1457, 1206, 1171, 1127, 1093, 1063, 760, 705, 633 cm⁻¹. The ee was determined by HPLC on Chiralpak OJ-H (n-hexane/i-PrOH = 95/5, 1.0 mL/min, 254 nm), t_R(major) = 21.3 min, t_S(minor) = 29.4 min, 60% ee.
The reaction of 1d (37.0 mg, 0.20 mmol), Me$_3$SiCF$_3$ (60.0 μL, 0.40 mmol, 2 equiv), (iPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 2 h gave 2d as a colorless oil (41.3 mg, 81%) by flash chromatography (PE/EtOAc = 20/1). [$\alpha$]$_D^{25}$ = –11.5 (c 0.24, CH$_2$Cl$_2$); $^1$H NMR (400 MHz, CDCl$_3$): δ 7.55–7.52 (m, 2H), 7.34 (d, $J$ = 8.4 Hz, 2H), 4.97 (q, $J$ = 6.4 Hz, 1H), 2.98 (s, 1H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): δ 132.8, 131.9, 129.1, 124.0 (q, $J$ = 281.1 Hz), 123.8, 72.2 (q, $J$ = 32.1 Hz) ppm; IR (KBr) 3420, 2921, 2353, 1596, 1491, 1406, 1356, 1267, 1097, 1012, 871, 847, 726, 667, 586 cm$^{-1}$. The ee was determined by HPLC on Chiralpak OD-H (n-hexane/i-PrOH = 95/5, 1.0 mL/min, 254 nm), $t_R$(major) = 9.1 min, $t_R$(minor) = 12.6 min, 57% ee.
The reaction of 1e (23.4 μL, 0.20 mmol), Me3SiCF3 (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 2 h gave 2e as a colorless oil (41.7 mg, 82%) by flash chromatography (PE/EtOAc = 20/1). [α]D\textsubscript{25} = −7.3 (c 0.11, CH2Cl2); \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): δ 7.64 (s, 1H), 7.55–7.52 (m, 1H), 7.40–7.38 (d, J = 7.6 Hz, 1H), 7.27 (t, J = 7.8 Hz, 1H), 5.00–4.95 (m, 1H), 3.01 (d, J = 4.4 Hz, 1H) ppm; \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): δ 136.3, 130.3, 124.0 (q, J = 281.1 Hz), 123.2, 116.7, 114.7, 114.5, 72.2 (q, J = 32.1 Hz) ppm; IR (KBr) 3409, 3062, 2925, 1574, 1430, 1355, 1255, 1178, 1076, 997, 838, 784, 630, 532, 453 cm\textsuperscript{−1}. The ee was determined by HPLC on Chiralpak OD-H (n-hexane/i-PrOH = 95/5, 1.0 mL/min, 254 nm), t\textsubscript{R}(major) = 8.8 min, t\textsubscript{R}(minor) = 14.4 min, 51% ee.
The reaction of 1f (28.1 mg, 0.20 mmol), Me3SiCF3 (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) at –20 °C for 2 h gave 2f as a colorless oil (35.0 mg, 83%) by flash chromatography (PE/EtOAc = 20/1). \([\alpha]_D^{25} = –8.3 \ (c \ 0.13, \ CH_2Cl_2) \ [lit.10 \ [\alpha]_D^{20} = –8.8 \ (c \ 0.10, \ CH_2Cl_2, \ 50\% \ ee)]; \) \(^1H\) NMR (400 MHz, CDCl3): \(\delta \ 7.43–7.38 \ (m, \ 4H), \ 5.04–4.98 \ (m, \ 1H), \ 2.72 \ (d, J = 4.4 \ Hz, \ 1H) \ ppm; \) \(^13C\) NMR (100 MHz, CDCl3): \(\delta \ 135.7, \ 132.4, \ 129.0, \ 128.9, \ 124.1 \ (q, J = 281.1 \ Hz), \ 72.3 \ (q, J = 32.1 \ Hz) \ ppm; \) IR (KBr) 3431, 2919, 1576, 1492, 1346, 1267, 1197, 1097, 870, 847, 811, 667, 811 cm\(^{-1}\). The ee was determined by HPLC on Chiralpak OD-H (n-hexane/i-PrOH = 95/5, 1.0 mL/min, 254 nm), \(t_d(major) = 8.0 \ min, \ t_d(minor) = 10.9 \ min, \ 52\% \ ee.\)
The reaction of 1g (23.6 μL, 0.20 mmol), Me₃SiCF₃ (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 1 h gave 2g as a colorless oil (33.4 mg, 88%) by flash chromatography (PE/EtOAc = 20/1). [α]D²⁵ = −12.7 (c 0.18, CH₂Cl₂) [lit.¹⁰ [α]D²⁰ = −18.4 (c 0.43, CH₂Cl₂, 60% ee)]; ¹H NMR (400 MHz, CDCl₃): δ 7.36 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 7.6 Hz, 2H), 4.97 (m, 1H), 2.73 (s, 1H), 2.38 (s, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 139.6, 131.1, 129.4, 127.4, 124.3 (q, J = 280.9 Hz), 72.8 (q, J = 31.9 Hz), 21.3 ppm; IR (KBr) 3421, 3046, 2927, 2847, 1515, 1399, 1355, 1264, 1167, 1126, 1093, 1030, 1001, 978, 877, 781, 698, 632, 538, 525, 455 cm⁻¹. The ee was determined by HPLC on Chiralpak OD-H (n-hexane/i-PrOH = 97/3, 1.0 mL/min, 254 nm), tR(major) = 13.3 min, tR(minor) = 17.7 min, 68% ee.

2,2,2-trifluoro-1-(p-tolyl)ethanol (2g)¹⁰

The reaction of 1g (23.6 μL, 0.20 mmol), Me₃SiCF₃ (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 1 h gave 2g as a colorless oil (33.4 mg, 88%) by flash chromatography (PE/EtOAc = 20/1). [α]D²⁵ = −12.7 (c 0.18, CH₂Cl₂) [lit.¹⁰ [α]D²⁰ = −18.4 (c 0.43, CH₂Cl₂, 60% ee)]; ¹H NMR (400 MHz, CDCl₃): δ 7.36 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 7.6 Hz, 2H), 4.97 (m, 1H), 2.73 (s, 1H), 2.38 (s, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 139.6, 131.1, 129.4, 127.4, 124.3 (q, J = 280.9 Hz), 72.8 (q, J = 31.9 Hz), 21.3 ppm; IR (KBr) 3421, 3046, 2927, 2847, 1515, 1399, 1355, 1264, 1167, 1126, 1093, 1030, 1001, 978, 877, 781, 698, 632, 538, 525, 455 cm⁻¹. The ee was determined by HPLC on Chiralpak OD-H (n-hexane/i-PrOH = 97/3, 1.0 mL/min, 254 nm), tR(major) = 13.3 min, tR(minor) = 17.7 min, 68% ee.
The reaction of 1h (36.0 mg, 0.20 mmol), Me3SiCF3 (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 1 h gave 2h as a white solid (45.4 mg, 90%) by flash chromatography (PE/EtOAc = 10/1). m.p. 113–114 °C; [α]D28 = −18.5 (c 0.40, CH2Cl2) [lit.10 [α]D20 = −7.1 (c 0.14, CH2Cl2, 56% ee)]; 1H NMR (400 MHz, CDCl3): δ 7.65–7.54 (m, 6H), 7.47–7.43 (m, 2H), 7.39–7.35 (m, 1H), 5.07 (m, 1H), 2.70 (d, J = 4.4 Hz, 1H) ppm; 13C NMR (100 MHz, CDCl3): δ 142.5, 140.3, 132.9, 128.9, 127.9, 127.7, 127.4, 127.2, 124.3 (q, J = 281.0 Hz), 72.7 (q, J = 32.0 Hz) ppm; IR (KBr) 3364, 1490, 1408, 1352, 1256, 1198, 1173, 1158, 1130, 1075, 1006, 855, 823, 764, 743, 726, 696 cm⁻¹. The ee was determined by HPLC on Daicel Chiralpak OD-H (hexane/iPrOH = 90/10, 1.0 mL/min, 254 nm), tR(major) = 10.0 min, tR(minor) = 12.8 min, 66% ee.
The reaction of 1i (34.5 μL, 0.20 mmol), Me₃SiCF₃ (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 1 h gave 2i as a colorless oil (46.6 mg, 87%) by flash chromatography (petroleum ether/EtOAc = 20/1). [α]₂⁵D = –8.8 (c 0.50, CH₂Cl₂); ¹H NMR (400 MHz, CDCl₃): δ 7.37–7.32 (m, 3H), 7.18 (d, J = 8.0 Hz, 1H), 7.14–7.10 (m, 2H), 7.04–7.00 (m, 3H), 4.96 (m, 1H), 2.75 (d, J = 4.0 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 157.3, 156.4, 135.6, 129.7, 129.6, 123.8 (q, J = 281.0 Hz), 123.4, 121.8, 119.3, 118.8, 117.5, 72.2 (q, J = 32.0 Hz) ppm; IR (KBr) 3447, 2926, 2843, 2357, 1606, 1491, 1458, 1438, 1262, 1170, 1126, 1044, 839, 788, 710, 556 cm⁻¹. The ee was determined by HPLC on Chiralpak OD-H (n-hexane/i-PrOH = 95/5, 1.0 mL/min, 254 nm), t₀(major) = 13.9 min, t₀(minor) = 28.6 min, 60% ee.
The reaction of 1j (24.3 μL, 0.20 mmol), Me3SiCF3 (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 1 h gave 2j as a colorless oil (35.0 mg, 85%) by flash chromatography (PE/EtOAc = 20/1). [α]D25 = –13.6 (c 0.27, CH2Cl2) [lit.10 [α]D20 = –8.9 (c 1.00, CH2Cl2, 41% ee)]; 1H NMR (400 MHz, CDCl3): δ 7.40 (d, J = 8.4 Hz, 2H), 6.93 (d, J = 8.4 Hz, 2H), 4.97 (q, J = 3.2 Hz, 1H), 3.82 (s, 3H), 2.58 (s, 1H) ppm; 13C NMR (100 MHz, CDCl3): δ 160.5, 128.8, 126.2, 124.4 (q, J = 281.0 Hz), 114.1, 72.5 (q, J = 32.0 Hz), 55.3 ppm; IR (KBr) 3445, 3008, 2918, 2842, 1614, 1587, 1517, 1465, 1444, 1356, 1252, 1207, 1171, 1127, 1075, 1031, 850, 819, 695, 589, 520 cm–1. The ee was determined by HPLC on Chiralpak OJ-H (n-hexane/i-PrOH = 95/5, 1.0 mL/min, 254 nm), tR(major) = 34.8 min, tR(minor) = 38.5 min, 67% ee.

2,2,2-trifluoro-1-(4-methoxyphenyl)ethanol (2j)10

The reaction of 1j (24.3 μL, 0.20 mmol), Me3SiCF3 (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 1 h gave 2j as a colorless oil (35.0 mg, 85%) by flash chromatography (PE/EtOAc = 20/1). [α]D25 = –13.6 (c 0.27, CH2Cl2) [lit.10 [α]D20 = –8.9 (c 1.00, CH2Cl2, 41% ee)]; 1H NMR (400 MHz, CDCl3): δ 7.40 (d, J = 8.4 Hz, 2H), 6.93 (d, J = 8.4 Hz, 2H), 4.97 (q, J = 3.2 Hz, 1H), 3.82 (s, 3H), 2.58 (s, 1H) ppm; 13C NMR (100 MHz, CDCl3): δ 160.5, 128.8, 126.2, 124.4 (q, J = 281.0 Hz), 114.1, 72.5 (q, J = 32.0 Hz), 55.3 ppm; IR (KBr) 3445, 3008, 2918, 2842, 1614, 1587, 1517, 1465, 1444, 1356, 1252, 1207, 1171, 1127, 1075, 1031, 850, 819, 695, 589, 520 cm–1. The ee was determined by HPLC on Chiralpak OJ-H (n-hexane/i-PrOH = 95/5, 1.0 mL/min, 254 nm), tR(major) = 34.8 min, tR(minor) = 38.5 min, 67% ee.
The reaction of 1k (24.3 μL, 0.20 mmol), Me₃SiCF₃ (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 1 h gave 2k as a colorless oil (36.7 mg, 89%) by flash chromatography (PE/EtOAc = 20/1). [α]D²⁵ = −13.6 (c 0.13, CH₂Cl₂); ¹H NMR (400 MHz, CDCl₃): δ 7.38–7.30 (m, 1H), 7.04 (d, J = 7.6 Hz, 2H), 6.95–6.92 (m, 1H), 4.98 (m, 1H), 3.82 (s, 3H), 2.93 (s, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 159.8, 135.5, 129.7, 124.2 (q, J = 281.1 Hz), 119.8, 115.1, 113.0, 72.8 (q, J = 31.8 Hz), 55.4 ppm; IR (KBr) 3442, 2926, 2843, 1357, 1605, 1589, 1491, 1458, 1438, 1262, 1170, 1044, 933, 788, 760, 710, 631, 556 cm⁻¹. The ee was determined by HPLC on Chiralpak OD-H (n-hexane/i-PrOH = 95/5, 1.0 mL/min, 254 nm), tR(major) = 20.7 min, tR(minor) = 28.0 min, 74% ee.
The reaction of 1l (27.2 mg, 0.20 mmol), Me₃SiCF₃ (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 1 h gave 2l as a colorless oil (36.1 mg, 88%) by flash chromatography (PE/EtOAc = 20/1). [α]D²⁵ = −11.7 (c 0.24, CH₂Cl₂); ¹H NMR (400 MHz, CDCl₃): δ 7.39–7.34 (m, 2H), 7.00 (dt, J = 7.6, 1.2 Hz, 1H), 6.94 (d, J = 8.4 Hz, 1H), 5.27 (m, 1H), 3.86 (s, 3H), 3.75 (q, J = 4.0 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 157.6, 130.6, 129.3, 124.7 (q, J = 282.0 Hz), 122.1, 121.1, 111.3, 69.9 (q, J = 32.5 Hz), 55.8 ppm; IR (KBr) 3441, 3012, 2946, 2845, 1605, 1591, 1496, 1466, 1442, 1358, 1249, 1171, 1131, 1027, 871, 829, 781, 627, 593, 574, 538, 498 cm⁻¹. The ee was determined by HPLC on Chiralpak OJ-H (n-hexane/i-PrOH = 97/3, 1.0 mL/min, 254 nm), tₛ(minor) = 23.5 min, tᵣ(major) = 27.4 min, 74% ee.

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The reaction of 1m (30.0 mg, 0.20 mmol), Me3SiCF3 (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 2 h gave 2m as a colorless oil (40.5 mg, 92%) by flash chromatography (PE/EtOAc = 10/1). \[\alpha\]D25 = –16.7 (c 0.48, CH2Cl2) [lit.10 \[\alpha\]D20 = –7.8 (c 0.55, CH2Cl2, 46% ee)]; 1H NMR (400 MHz, CDCl3): δ 6.96 (s, 1H), 6.91 (d, J = 8.4 Hz, 1H), 6.81 (d, J = 8.0 Hz, 1H), 5.97 (s, 2H), 4.90 (m, 1H), 2.90 (d, J = 2.8 Hz, 1H) ppm; 13C NMR (100 MHz, CDCl3): δ 148.6, 148.0, 127.8, 124.3 (q, J = 281.0 Hz), 121.6, 108.3, 107.7, 101.4, 72.6 (q, J = 32.0 Hz) ppm; IR (KBr) 3454, 2906, 2772, 1855, 1602, 1506, 1492, 1448, 1351, 1251, 1170, 1124, 1040, 930, 872, 810, 793, 730, 704, 636, 654, 560 cm⁻¹. The ee was determined by HPLC on Chiralpak OD-H (n-hexane/i-PrOH = 97/3, 1.0 mL/min, 254 nm), tR(major) = 27.9 min, tR(minor) = 34.7 min, 81% ee.
The reaction of 1n (32.8 mg, 0.20 mmol), Me₃SiCF₃ (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 2 h gave 2n as a colorless oil (43.1 mg, 92%) by flash chromatography (PE/EtOAc = 10/1). [α]D²⁵ = -16.3 (c 0.54, CH₂Cl₂); ¹H NMR (400 MHz, CDCl₃): δ 7.00 (s, 1H), 6.94 (d, J = 8.4 Hz, 1H), 6.88 (d, J = 8.4 Hz, 1H), 4.90 (m, 1H), 4.27 (s, 4H), 2.90 (d, J = 4.0 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 144.6, 143.6, 127.2, 124.3 (q, J = 281.0 Hz), 120.6, 117.5, 116.6, 72.4 (q, J = 32.0 Hz), 64.4, 64.3 ppm; IR (KBr) 3458, 2928, 1539, 1511, 1461, 1437, 1314, 1291, 1260, 1171, 1125, 1067, 921, 888, 846, 816, 725, 703, 664 cm⁻¹. The ee was determined by HPLC on Chiralpak OD-H (n-hexane/i-PrOH = 97/3, 1.0 mL/min, 254 nm), tₗ(minor) = 44.9 min, tₗ(major) = 50.4 min, 79% ee.
The reaction of 1o (30.7 μL, 0.20 mmol), Me3SiCF3 (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 2 h gave 2o as a white solid (37.1 mg, 80%) by flash chromatography (PE/EtOAc = 20/1). m.p. 49–50 °C; [α]D25 = –19.3 (c 0.20, CH2Cl2); 1H NMR (400 MHz, CDCl3): δ 7.38 (d, J = 8.4 Hz, 2H), 6.95–6.92 (m, 2H), 6.10–6.00 (m, 1H), 5.41(qd, J = 1,6, 17.2 Hz, 1H), 5.29 (qd, J = 1.2, 10.8 Hz, 1H), 4.98–4.92 (m, 1H), 4.54 (td, J = 1.6, 5.2 Hz, 2H), 2.61 (br, 1H) ppm; 13C NMR (100 MHz, CDCl3): δ 159.5, 133.0, 128.8, 126.3, 124.4 (q, J = 281.0 Hz), 117.9, 114.9, 72.5 (q, J = 32.0 Hz), 68.9 ppm; IR (KBr) 3408, 2929, 2863, 1888, 1515, 1428, 1324, 1247, 1178, 1117, 1070, 1013, 940, 871, 802, 589 cm−1; Anal. Calcd. for C11H11F3O2: C, 56.90; H, 4.77; Found: C, 57.12; H, 4.87; The ee was determined by HPLC on Chiralpak AD-H (n-hexane/i-PrOH = 97/3, 1.0 mL/min, 254 nm), tS(minor) = 18.0 min, tR(major) = 20.2 min, 66% ee.
The reaction of 1p (30.2 μL, 0.20 mmol), Me3SiCF3 (60.0 μL, 0.40 mmol), (IPr)CuF (1.9 mg, 0.004 mmol, 2 mol%), quaternary ammonium salts (3e) (2.6 mg, 0.004 mmol, 2 mol%) for 1 h gave 2p as a white solid (40.1 mg, 85%) by flash chromatography (PE/EtOAc = 10/1). m.p. 47–49 °C; [α]D25 = –16.7 (c 0.63, CH2Cl2); 1H NMR (400 MHz, CDCl3): δ 7.37 (d, J = 8.4 Hz, 2H), 7.32 (d, J = 8.8 Hz, 2H), 4.93–4.50 (m, 1H), 2.96 (q, J = 7.2 Hz, 2H), 2.70 (s, 1H), 1.33 (t, J = 7.4 Hz, 3H) ppm; 13C NMR (100 MHz, CDCl3): δ 138.9, 131.1, 128.3, 127.9, 124.2 (q, J = 281.0 Hz), 72.5 (q, J = 32.0 Hz), 72.1, 14.2 ppm; IR (KBr) 3356, 2974, 2928, 2873, 1601, 1496, 1406, 1351, 1255, 1135, 1093, 1071, 848, 811, 793, 735, 680, 586, 496 cm⁻¹; Anal. Calcd. for C10H11F3OS: C, 50.84; H, 4.69; Found: C, 51.23; H, 4.76; The ee was determined by HPLC on Chiralpak OJ-H (n-hexane/i-PrOH = 95/5, 1.0 mL/min, 254 nm), tR(major) = 26.2 min, tR(minor) = 30.2 min, 73% ee.
5 References

6 Copies of NMR spectra
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