Supporting Information

Metal- and base-free synthesis of functionalized α, α-difluoroimines via electrophilic fluorination of N-substituted enamines

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1. General Information

Unless otherwise stated, all commercial reagents were used as received. Reactions were conducted in dry glassware using anhydrous solvents (pass through activated alumina columns). Reaction temperatures were controlled using IKAmag temperature modulator, and unless stated otherwise, reactions were performed at room temperature (rt, approximately, 24 °C). Thin-layer chromatography (TLC) was conducted on plates (GF254) supplied by Yantai Chemicals (China) and visualized using a combination of UV, anisaldehyde, iodine, and potassium permanganate staining. Silica gel (200-300 mesh) supplied by Tsingdao Haiyang Chemicals (China) was used for flash column chromatography. 

$^1$H, $^{13}$C and $^{19}$F NMR spectra were recorded on Bruker spectrometers (400 MHz, 500 MHz). Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference: proton (chloroform $\delta$ 7.26), carbon (chloroform $\delta$ 77.16) or tetramethylsilane (TMS $\delta$ 0.00) was used as a reference. Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), bs (broad singlet). Coupling constants were reported in Hertz (Hz). All high resolution mass spectra were obtained from the Tianjin University Mass Spectrometry Facility.
2. Experimental procedure

(A) Materials
Substrates (Enamines) were prepared according to the known procedures.[1]

(B) Experimental procedure for functionalized α-fluoroimine synthesis

\[ \begin{align*}
R\text{-H} & \quad \text{Conc. NH}_2 \quad \text{EWG} \quad \text{Selectfluor} \\
& \quad \text{CH}_3\text{CN, 0°C} \\
& \quad \text{2} \\
\end{align*} \]

The stirred solution of enamine 1 (0.5 mmol) in CH$_3$CN (5 mL) was cooled to 0 °C, and Selectfluor (443 mg, 1.25 mmol) was added in portions. The resulting mixture was stirred at 0 °C till 1 was completely consumed (1h, monitored by TLC). After that, the reaction mixture was diluted with Et$_2$O and filtered through a pad of Celite. The combined organic mixture was concentrated in vacuo and purified by silica gel column chromatography (Hexane/EtOAc) to afford the desired product 2.

**Ethyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-phenylpropanoate (2a):** 158 mg, 95% yield;

Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.39 – 7.32 (m, 3H), 7.28 – 7.26 (m, 2H), 6.69 (s, 4H), 4.44 (q, $J$ = 7 Hz, 2H), 3.73 (s, 3H), 1.40 (t, $J$ = 7 Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 163.36 (t, $J$ = 31 Hz), 159.81 (t, $J$ = 30 Hz), 157.86, 140.02, 131.00, 130.04, 128.91, 128.72, 123.64, 114.01, 113.13 (t, $J$ = 253 Hz), 63.07, 55.45, 14.19; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 105.11; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{18}$F$_2$NO$_3$) requires $m/z$ 334.1255, found $m/z$ 334.1260
Ethyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-(p-tolyl)propanoate (2c): 153 mg, 88% yield; Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) δ (ppm): 7.17 (d, $J = 8$ Hz, 2H), 7.13 (d, $J = 8$ Hz, 2H), 6.70 (s, 4H), 4.43 (q, $J = 7$ Hz, 2H), 3.73 (s, 3H), 2.33 (s, 3H), 1.40 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) δ (ppm): 163.45 (t, $J = 31$ Hz), 159.94 (t, $J = 30$ Hz), 157.72, 140.28, 140.26, 129.41, 128.87, 127.88, 123.48, 114.01, 113.23 (t, $J = 253$ Hz), 63.01, 55.44, 21.57, 14.18; $^{19}$F NMR (376 MHz, CDCl$_3$) δ (ppm): −105.05; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{19}$H$_{20}$F$_2$NO$_3$) requires m/z 348.1411, found m/z 348.11410
**Ethyl (E)-2,2-difluoro-3-(4-fluorophenyl)-3-((4-methoxyphenyl)imino)propanoate (2d):** 160 mg, 91% yield; Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.32 – 7.29 (m, 2H), 7.07 – 7.02 (m, 2H), 6.75 – 6.69 (m, 4H), 4.47 (q, $J = 7$ Hz, 2H), 3.76 (s, 3H), 1.42 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 163.39 (d, $J = 250$ Hz), 163.07 (t, $J = 31$ Hz), 158.67 (t, $J = 30$ Hz), 157.82, 139.72, 131.09 (d, $J = 9$ Hz), 126.71 (d, $J = 4$ Hz), 123.33, 115.93 (d, $J = 22$ Hz), 114.02, 112.95 (t, $J = 253$ Hz), 63.02, 55.34, 14.05; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): −104.99, −109.24; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{17}$F$_3$NO$_3$) requires m/z 352.1161, found m/z 352.1165;

**Ethyl (E)-3-(4-chlorophenyl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2e):** 178 mg, 97% yield; Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.31 (d, $J = 8.5$ Hz, 2H), 7.22 (d, $J = 8.5$ Hz, 2H), 6.72 – 6.67 (m, 4H), 4.44 (q, $J = 7$ Hz, 2H), 3.72 (s, 3H), 1.39 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 163.04 (t, $J = 31$ Hz), 158.57 (t, $J = 30$ Hz), 158.00, 139.65, 136.30, 130.36, 129.21, 129.09, 123.44, 114.12, 112.96 (t, $J = 253$ Hz), 63.12, 55.39, 14.11; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): −104.95; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{17}$ClF$_2$NO$_3$) requires m/z 368.0865, found m/z 368.0870
Ethyl (E)-3-(4-bromophenyl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2f): 196 mg, 95% yield; Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.47 (d, $J$ = 8.5 Hz, 2H), 7.22 (d, $J$ = 8.5 Hz, 2H), 6.73 – 6.67 (m, 4H), 4.44 (q, $J$ = 7 Hz, 2H), 3.74 (s, 3H), 1.40 (t, $J$ = 7 Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 163.09 (t, $J$ = 31 Hz), 158.63 (t, $J$ = 30 Hz), 158.05, 139.67, 132.09, 130.57, 129.72, 124.72, 123.50, 114.18, 112.95 (t, $J$ = 253 Hz), 63.18, 55.47, 14.17; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 104.95; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{17}$BrF$_2$NO$_3$) requires m/z 412.0360, found m/z 412.0363

Ethyl (E)-3-(3-bromophenyl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2g): 190 mg, 92% yield; Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.51 (dt, $J$ = 7.5 Hz, 1.5 Hz, 1H), 7.47 (s, 1H), 7.21 – 7.16 (m, 2H), 6.70 (d, $J$ = 2.5 Hz, 4H), 4.44 (q, $J$ = 7 Hz, 2H), 3.72 (s, 3H), 1.40 (t, $J$ = 7 Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 162.96 (t, $J$ = 31 Hz), 158.17, 157.83 (t, $J$ = 30 Hz), 139.35, 133.17, 132.90, 131.54, 130.29, 127.58, 123.68, 122.80, 114.12, 112.84 (t, $J$ = 253 Hz), 63.15, 55.42, 14.12; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 104.98; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{17}$BrF$_2$NO$_3$) requires m/z 412.0360, found m/z 412.0362
Ethyl (E)-3-(2-bromophenyl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2h): 177 mg, 86% yield; Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.56 (d, $J$ = 8 Hz, 1H), 7.41 – 7.36 (m, 2H), 7.31 – 7.27 (m, 1H), 6.81 (d, $J$ = 9 Hz, 2H), 6.71 (d, $J$ = 9 Hz, 2H), 4.54 – 4.43 (m, 2H), 3.74 (s, 2H), 1.44 (t, $J$ = 7 Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 163.09 (t, $J$ = 31 Hz), 158.49, 157.49 (dd, $J$ = 33 Hz, 29 Hz), 139.40, 133.33, 133.28, 131.27, 130.72, 127.47, 124.07, 121.61, 113.65, 112.44 (dd, $J$ = 255 Hz, 251 Hz), 63.15, 55.40, 14.21; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 104.37 (d, $J$ = 277 Hz), – 106.78, (d, $J$ = 277 Hz) ; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{17}$BrF$_2$NO$_3$) requires $m/z$ 412.0360, found $m/z$ 412.0364

Ethyl (E)-2,2-difluoro-3-(4-iodophenyl)-3-((4-methoxyphenyl)imino)propanoate (4i): 220 mg, 96% yield; Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.68 (d, $J$ = 8 Hz, 2H), 7.01 (d, $J$ = 8 Hz, 2H), 6.73 – 6.67 (m, 4H), 4.44 (q, $J$ = 7 Hz, 2H), 3.73 (s, 3H), 1.39 (t, $J$ = 7 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 163.04 (t, $J$ = 31 Hz), 158.67 (t, $J$ = 30 Hz), 158.01, 139.61, 137.96, 130.49, 130.25, 123.48, 114.14, 112.89 (t, $J$ = 253 Hz), 96.77, 63.14, 55.42, 14.14; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 104.93; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{17}$F$_2$INO$_3$) requires $m/z$ 460.0221, found $m/z$ 460.0225
Ethyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-(4-(trifluoromethyl)phenyl)propanoate (2j):

199 mg, 99% yield; Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.61 (d, $J = 8$ Hz, 2H), 7.41 (d, $J = 8$ Hz, 2H), 6.89 (q, $J = 9$ Hz, 4H), 4.46 (q, $J = 7$ Hz, 2H), 3.74 (s, 3H), 1.41 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 162.83 (t, $J = 31$ Hz), 158.12, 158.11 (t, $J = 31$ Hz), 139.22, 134.51, 131.85 (q, $J = 33$ Hz), 129.35, 125.65 (q, $J = 4$ Hz), 123.59 (q, $J = 271$ Hz), 123.50, 114.09, 112.77 (t, $J = 253$ Hz), 63.16, 55.35, 14.05; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 63.07, – 104.94; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{19}$H$_{17}$F$_5$NO$_3$) requires m/z 402.1129, found m/z 402.1131

Ethyl (E)-3-(3-chloro-4-fluorophenyl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2k):

189 mg, 98% yield; Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.40 – 7.38 (m, 1H), 7.15 – 7.07 (m, 2H), 6.74 – 6.67 (m, 4H), 4.44 (q, $J = 7$ Hz, 2H), 3.75 (s, 3H), 1.40 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 162.91 (t, $J = 31$ Hz), 158.96 (d, $J = 252$ Hz), 158.23, 157.17 (t, $J = 30$ Hz), 139.31, 131.37, 129.34 (d, $J = 8$ Hz), 127.86 (d, $J = 4$ Hz), 123.52, 121.98 (d, $J = 18$ Hz), 117.22 (d, $J = 21$ Hz), 114.26, 112.87 (t, $J = 253$ Hz), 63.25, 55.48, 14.16; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 104.90, – 111.49; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{16}$ClF$_3$NO$_3$) requires m/z 386.0771, found m/z 386.0773
Ethyl (E)-3-(benzo[d][1,3]dioxol-5-yl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2l):  
158 mg, 84% yield; Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 6.80 – 6.72 (m, 7H), 5.96 (s, 2H), 4.43 (q, $J = 7$ Hz, 2H), 3.74 (s, 3H), 1.39 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 163.36 (t, $J = 31$ Hz), 158.99 (t, $J = 30$ Hz), 157.80, 157.00, 149.08, 147.90, 140.09, 124.10, 123.69, 123.41, 114.11, 113.19 (t, $J = 253$ Hz), 109.21, 108.73, 101.59, 63.04, 55.45, 14.17; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): –104.83; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{19}$H$_{18}$F$_2$NO$_5$) requires $m/z$ 378.1153, found $m/z$ 378.1157

Ethyl (Z)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-(thiophen-2-yl)propanoate (2m): 132 mg, 78% yield; Colorless oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.57 – 7.56 (m, 1H), 7.42 (dd, $J = 8$ Hz, 1 Hz, 1H), 7.01 (dd, $J = 5$ Hz, 4 Hz, 1H), 6.88 (d, $J = 9$ Hz, 2H), 6.73 (d, $J = 9$ Hz, 2H), 4.41 (q, $J = 7$ Hz, 2H), 3.81 (s, 3H), 1.36 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 163.01 (t, $J = 31$ Hz), 157.76, 153.44 (t, $J = 30$ Hz), 141.47, 133.12 (t, $J = 4$ Hz), 131.71, 129.47 (t, $J = 3$ Hz), 126.77, 120.71, 114.89, 113.74 (t, $J = 256$ Hz), 63.12, 55.58, 14.13; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): –103.98; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{16}$H$_{16}$F$_2$NO$_3$S) requires $m/z$ 340.0819, found $m/z$ 340.0821

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Methyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-phenylpropanoate (2n): 150 mg, 94% yield;

Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.42 – 7.29 (m, 5H), 6.72 (s, 4H), 3.99 (s, 3H), 3.74 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 163.88 (t, $J = 31$ Hz), 159.73 (t, $J = 30$ Hz), 157.88, 139.87, 130.86, 130.08, 128.87, 128.73, 123.70, 113.98, 113.43 (t, $J = 253$ Hz), 55.42, 53.55; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): –105.14; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{17}$H$_{16}$F$_2$NO$_3$) requires $m/z$ 320.1098, found $m/z$ 320.1093

Isopropyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-phenylpropanoate (2o): 160 mg, 92% yield; Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.41 – 7.28 (m, 5H), 6.71 (s, 4H), 5.34 – 5.28 (m, 1H), 3.75 (s, 3H), 1.41 (s, 3H), 1.39 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 162.88 (t, $J = 31$ Hz), 159.83 (t, $J = 30$ Hz), 157.81, 140.06, 131.04, 130.01, 128.91, 128.70, 123.59, 114.00, 112.88 (t, $J = 253$ Hz), 71.32, 55.44, 21.70; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): –104.96; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{19}$H$_{20}$F$_2$NO$_3$) requires $m/z$ 348.1411, found $m/z$ 348.1414
Benzyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-phenylpropanoate (2p): 178 mg, 90% yield;

Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.49 – 7.48 (m, 2H), 7.43 – 7.38 (m, 4H), 7.35 (t, $J$ = 8 Hz, 2H), 7.29 (d, $J$ = 8 Hz, 2H), 6.71 (d, $J$ = 9 Hz, 2H), 6.64 (d, $J$ = 9 Hz, 2H), 5.45 (s, 2H), 3.75 (s, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 163.27 (t, $J$ = 32 Hz), 159.58 (t, $J$ = 30 Hz), 157.86, 139.80, 134.76, 130.84, 130.04, 128.84, 128.74, 128.73, 128.70, 128.67, 123.68, 113.92, 113.28 (t, $J$ = 254 Hz), 68.38, 55.38; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 104.67; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{23}$H$_{20}$F$_2$NO$_3$) requires $m/z$ 396.1411, found $m/z$ 396.1408

(E)-2,2-Difluoro-3-((4-methoxyphenyl)imino)-1,3-diphenylpropan-1-one (2q): 175 mg, 96% yield;

Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 8.07 (d, $J$ = 8 Hz, 2H), 7.62 (t, $J$ = 8 Hz, 1H), 7.50 (t, $J$ = 8 Hz, 2H), 7.43 – 7.34 (m, 5H), 6.64 (d, $J$ = 9 Hz, 2H), 6.54 (d, $J$ = 9 Hz, 2H), 3.70 (s, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 188.48 (t, $J$ = 27 Hz), 161.30 (t, $J$ = 29 Hz), 157.89, 139.91, 133.85, 133.24, 131.05, 130.24, 130.13, 128.95, 128.87, 128.63, 123.38, 114.74 (t, $J$ = 254 Hz), 113.99, 55.42; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 100.73; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{23}$H$_{20}$F$_2$NO$_3$) requires $m/z$ 366.1306, found $m/z$ 366.1310
(E)-2,2-Difluoro-3-((4-methoxyphenyl)imino)-3-phenylpropanenitrile (2r): 142 mg, 99% yield;

Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.47 (t, $J = 8$ Hz, 1H), 7.41 (t, $J = 8$ Hz, 2H), 7.28 (d, $J = 7$ Hz, 2H), 6.86 (d, $J = 9$ Hz, 2H), 6.76 (d, $J = 9$ Hz, 2H), 3.78 (s, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 158.77, 156.06 (t, $J = 30$ Hz), 138.88, 130.74, 129.53, 129.17, 128.74, 124.62, 114.17, 111.52 (t, $J = 44$ Hz), 110.32 (t, $J = 246$ Hz), 55.48; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): –87.19; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{16}$H$_{13}$F$_2$N$_2$O) requires m/z 287.0996, found m/z 287.0996

Ethyl (E)-2,2-difluoro-3-((3-methoxyphenyl)imino)-3-phenylpropane (2s): 157 mg, 94% yield;

Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.37 – 7.27 (m, 5H), 7.05 (t, $J = 9$ Hz, 1H), 6.58 – 6.56 (m, 1H), 6.28 – 6.27 (m, 2H), 4.44 (q, $J = 7$ Hz, 2H), 3.79 (s, 3H), 3.65 (s, 3H), 1.39 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 163.11 (t, $J = 31$ Hz), 161.62 (t, $J = 30$ Hz), 160.02, 148.62, 130.39, 130.20, 129.64, 128.92, 128.51, 113.18, 112.78 (t, $J = 254$ Hz), 111.20, 106.51, 63.20, 55.32, 14.16; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): –105.30; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{16}$H$_{13}$F$_2$NO$_3$) requires m/z 334.1255, found m/z 334.1250
Ethyl (E)-2,2-difluoro-3-((2-methoxyphenyl)imino)-3-phenylpropanoate (2t): 155 mg, 93% yield; Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) δ (ppm): 7.35 – 7.26 (m, 5H), 7.01 (td, $J = 8$ Hz, 2 Hz, 1H), 6.79 (td, $J = 8$ Hz, 1 Hz, 1H), 6.76 (d, $J = 8$ Hz, 1H), 6.65 (dd, $J = 8$ Hz, 2 Hz, 1H), 4.46 (q, $J = 7$ Hz, 2H), 3.63 (s, 3H), 1.42 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) δ (ppm): 163.14 (t, $J = 31$ Hz), 162.80 (t, $J = 30$ Hz), 148.87, 137.27, 131.22, 130.03, 128.20, 128.14, 126.02, 120.93, 120.64, 112.68 (t, $J = 254$ Hz), 111.79, 63.11, 55.44, 14.09; $^{19}$F NMR (376 MHz, CDCl$_3$) δ (ppm): –104.89; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{18}$F$_2$NO$_3$) requires m/z 334.1255, found m/z 334.1258

Ethyl (E)-2,2-difluoro-3-phenyl-3-(phenylimino)propanoate (2u): 141 mg, 93% yield; Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) δ (ppm): 7.36 – 7.25 (m, 5H), 7.17 (t, $J = 8$ Hz, 2H), 7.02 (t, $J = 7.5$ Hz, 1H), 6.71 (d, $J = 7.5$ Hz, 2H), 4.45 (q, $J = 7$ Hz, 2H), 1.40 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) δ (ppm): 163.14 (t, $J = 31$ Hz), 161.51 (t, $J = 30$ Hz), 147.39, 130.36, 130.15, 129.01, 128.14, 126.02, 120.86, 112.82 (t, $J = 254$ Hz), 111.79, 63.11, 55.44, 14.09; $^{19}$F NMR (376 MHz, CDCl$_3$) δ (ppm): –105.28; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{17}$H$_{16}$F$_2$NO$_2$) requires m/z 304.1149, found m/z 304.1150
Ethyl (E)-2,2-difluoro-3-((4-fluorophenyl)imino)-3-phenylpropanoate (2v): 133 mg, 83% yield;

Yellow oil; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.39 – 7.36 (m, 1H), 7.33 – 7.30 (m, 2H), 7.25 – 7.24 (m, 2H), 6.88 – 6.84 (m, 2H), 6.70 – 6.67 (m, 2H), 4.44 (q, \(J = 7\) Hz, 2H), 1.40 (t, \(J = 7\) Hz, 3H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) (ppm): 163.09 (t, \(J = 31\) Hz), 161.88 (t, \(J = 31\) Hz), 160.57 (d, \(J = 244\) Hz), 143.26 (d, \(J = 3\) Hz), 130.32, 128.92, 128.70, 122.95 (d, \(J = 8\) Hz), 115.70 (d, \(J = 23\) Hz), 112.76 (t, \(J = 254\) Hz), 63.21, 14.17; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) (ppm): – 105.31, – 117.18; HRMS (ESI) exact mass calculated for [M+H]\(^+\) (C\(_{17}\)H\(_{15}\)F\(_3\)NO\(_2\)) requires \(m/z\) 322.1055, found \(m/z\) 322.1059

Ethyl (E)-3-((4-chlorophenyl)imino)-2,2-difluoro-3-phenylpropanoate (2w): 145 mg, 86% yield;

Yellow oil; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.39 – 7.36 (m, 1H), 7.33 – 7.30 (m, 2H), 7.25 – 7.23 (m, 2H), 7.14 (d, \(J = 9\) Hz, 2H), 6.65 (d, \(J = 9\) Hz, 2H), 4.44 (q, \(J = 7\) Hz, 2H), 1.39 (t, \(J = 7\) Hz, 3H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) (ppm): 163.00 (t, \(J = 31\) Hz), 162.30 (t, \(J = 31\) Hz), 145.83, 130.99, 130.44, 130.05, 129.02, 128.93, 128.71, 122.41, 112.63 (t, \(J = 254\) Hz), 63.27, 14.17; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) (ppm): – 105.31; HRMS (ESI) exact mass calculated for [M+H]\(^+\) (C\(_{17}\)H\(_{15}\)ClF\(_2\)NO\(_2\)) requires \(m/z\) 338.0759, found \(m/z\) 338.0763
Ethyl (E)-2,2-difluoro-3-phenyl-3-((4-(trifluoromethyl)phenyl)imino)propanoate (2x). 167 mg, 90% yield; Yellow oil; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 7.47 (d, $J = 8$ Hz, 2H), 7.40 – 7.38 (m, 1H), 7.34 – 7.32 (m, 2H), 7.28 – 7.26 (m, 2H), 6.82 (d, $J = 8$ Hz, 2H), 4.47 (q, $J = 7$ Hz, 2H), 1.41 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 163.12 (t, $J = 30$ Hz), 162.82 (t, $J = 31$ Hz), 150.55, 130.64, 129.66, 128.90, 128.69, 127.21 (q, $J = 33$ Hz), 126.16 (q, $J = 34$ Hz), 124.14 (q, $J = 270$ Hz), 120.61, 112.40 (t, $J = 254$ Hz), 63.36, 14.11; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 62.25, – 105.37; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{15}$F$_5$NO$_2$) requires $m/z$ 372.1023, found $m/z$ 372.1024

Ethyl (E)-2,2-difluoro-3-((4-nitrobenzyl)imino)-3-phenylpropanoate (2y): 125 mg, 69% yield; White solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 8.17 (d, $J = 9$ Hz, 2H), 7.53 – 7.47 (m, 3H), 7.39 (d, $J = 9$ Hz, 2H), 7.31 – 7.29 (m, 2H), 4.65 (s, 2H), 4.41 (q, $J = 7$ Hz, 2H), 1.35 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ (ppm): 164.91 (t, $J = 30$ Hz), 163.17 (t, $J = 31$ Hz), 147.18, 146.03, 130.54, 129.95, 129.06, 128.03, 127.79, 123.77, 112.58 (t, $J = 253$ Hz), 63.18, 55.69, 14.13; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm): – 105.92; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{17}$F$_2$N$_2$O$_4$)
requires m/z 363.1156, found m/z 363.1159

![Chemical Structure](image)

**Ethyl (E)-3-(cyclopropylimino)-2,2-difluoro-3-phenylpropanoate (2z):** 122 mg, 91% yield; Yellow oil; ¹H NMR (500 MHz, CDCl₃) δ (ppm): 7.48 – 7.44 (m, 3H), 7.41 – 7.39 (m, 2H), 4.35 (q, J = 7 Hz, 2H), 2.92 – 2.88 (m, 1H), 1.33 (t, J = 7 Hz, 3H), 0.97 – 0.86 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ (ppm): 163.40 (t, J = 31 Hz), 159.65 (t, J = 30 Hz), 130.85, 129.81, 128.70, 128.50, 113.15 (t, J = 251 Hz), 62.68, 35.07, 14.02, 10.65; ¹⁹F NMR (376 MHz, CDCl₃) δ (ppm): – 105.63; HRMS (ESI) exact mass calculated for [M+H]+ (C₁₄H₁₆F₂NO₂) requires m/z 268.1149, found m/z 268.1150

![Chemical Structure](image)

**Ethyl (E)-2-fluoro-3-phenyl-3-(phenylamino)acrylate (8):** 109 mg, 44% yield; colorless oil; ¹H NMR (400 MHz, CDCl₃) δ (ppm): 9.13 (brs, 1H), 7.45 – 7.43 (m, 2H), 7.36 – 7.32 (m, 3H), 7.05 (t, J = 8 Hz, 2H), 6.87 (t, J = 8 Hz, 1H), 6.62 (d, J = 8 Hz, 2H), 4.37 (q, J = 7 Hz, 2H), 1.40 (t, J = 7 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 164.85 (d, J = 26 Hz), 143.56 (d, J = 24 Hz), 140.82, 132.34 (d, J = 230 Hz), 130.84, 129.79, 129.75, 128.84, 128.56, 122.59, 121.45, 60.93, 14.54; ¹⁹F NMR (376 MHz, CDCl₃) δ (ppm): – 170.71; HRMS (ESI) exact mass calculated for [M+H]+ (C₁₄H₁₆F₂NO₂) requires m/z 286.1243, found m/z 286.1234
3. Late-stage Modification

a) Synthesis of compound 3 \(^{[2]}\)

To a solution of \(1a\) (168 mg, 0.5 mmol) in THF (2 mL) was added 1 N HCl (2 mL) at 0 °C, and the resulting mixture was stirred at room temperature for 1h. Then the reaction mixture was extracted with Et\(_2\)O (3*5 mL), and the combined organic layer was washed with Na\(_2\)SO\(_4\), filtered and concentrated in vacuo. The residue was purified by column chromatography (1:10, EA : Hexane) to afford 3 as colorless oil (95 mg, 83%). \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm): 8.07 (d, \(J = 7.5\) Hz, 2H), 7.67 (t, \(J = 7.5\) Hz, 1H), 7.51 (t, \(J = 7.5\) Hz, 2H), 6.71 (s, 4H), 4.38 (q, \(J = 7\) Hz, 2H), 1.31 (t, \(J = 7\) Hz, 3H); \(^1\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) (ppm): 185.58 (t, \(J = 27\) Hz), 161.92 (t, \(J = 30\) Hz), 135.20, 131.20, 130.02 (t, \(J = 2.6\) Hz), 129.08, 109.91 (t, \(J = 253\) Hz), 63.86, 13.91; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) (ppm): –107.64

b) Synthesis of compound 4\(^{[3]}\)

To a solution of \(1a\) (168 mg, 0.5 mmol) in EtOH (5 mL) was added NaBH\(_3\)CN (130 mg, 2 mmol) and AcOH (120 mg, 2 mmol), the resulting mixture was stirred at room temperature overnight. After the reaction was finished, sat. NaHCO\(_3\) was added and the aqueous was extracted with EA (3*5 mL). The combined organic layer was washed with brine, dried with Na\(_2\)SO\(_4\), filtered and concentrated in vacuo. The residue was purified by column chromatography (1: 20, EA : Hexane) to afford 4 as white solid (151 mg, 90%). White solid; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.42 – 7.32 (m, 5H), 6.73 – 6.70 (m, 2H), 6.62 – 6.60 (m, 2H), 5.03 (dd, \(J = 19.5\) Hz, 7.5 Hz, 1H), 4.29 (q, \(J = 7\) Hz, 2H), 4.17 (brs, 1H), 3.70 (s, 3H), 1.26 (t, \(J = 7\) Hz, 3H); \(^1\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) (ppm): 163.75 (t, \(J = 31\) Hz), 63.86, 13.91;
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153.36, 139.43, 134.20, 128.89, 128.75, 128.47, 158.05, 139.67, 132.09, 130.57, 129.72, 124.72, 123.50, 114.18, 112.95 (t, \( J = 253 \) Hz), 63.18, 55.47, 14.17; \(^{19}\text{F} \) NMR (376 MHz, CDCl\(_3\)) \( \delta \) (ppm): – 108.84 (d, \( J = 257 \) Hz), – 119.81 (d, \( J = 257 \) Hz); HRMS (ESI) exact mass calculated for \([\text{M+Na}]^+ (\text{C}_{15}\text{H}_{19}\text{FO}_{2}\text{Na})\) requires \( m/\ell \) 358.1231, found \( m/\ell \) 358.1233

c) Synthesis of compound 5\(^4\)

To a solution of 1a (168 mg, 0.5 mmol) in MeOH/THF (v/v = 1/1, 4 mL) was added NaBH\(_4\) (76 mg, 2 mmol) slowly at 0 °C, the reaction mixture was stirred at 0 °C for 1 h. TLC indicated the reaction was completion, sat. NH\(_4\)Cl was added to quench the reaction. The resulting mixture was extracted with EA (3*5 mL), the combined organic layers were washed with brine, dried with MgSO\(_4\), filtered and concentrated in vacuo. The residue was purified by column chromatography (1:2, EA : Hexane) to afford 5 as yellow solid (141 mg, 96%). \(^1\text{H} \) NMR (500 MHz, CDCl\(_3\)) \( \delta \) (ppm): 7.43 – 7.31 (m, 5H), 6.71 (d, \( J = 9 \) Hz, 2H), 6.60 (d, \( J = 9 \) Hz, 2H), 4.88 – 4.83 (m, 1H), 4.30 (brs, 1H), 3.99 – 3.91 (m, 1H), 3.83 – 3.76 (m, 1H), 3.70 (s, 3H), 2.54 (brs, 3H); \(^{13}\text{C} \) NMR (125 MHz, CDCl\(_3\)) \( \delta \) (ppm): 153.17, 140.00, 135.98, 128.76, 128.54, 128.38, 121.57 (t, \( J = 247 \) Hz), 116.00, 114.98, 63.01 (t, \( J = 31 \) Hz), 60.94 (dd, \( J = 26 \) Hz, 3 Hz), 55.80; \(^{19}\text{F} \) NMR (376 MHz, CDCl\(_3\)) \( \delta \) (ppm): – 113.97 (d, \( J = 256 \) Hz), – 117.86 (d, \( J = 256 \) Hz); HRMS (ESI) exact mass calculated for \([\text{M+H}]^+ (\text{C}_{16}\text{H}_{18}\text{F}_{2}\text{NO}_{2})\) requires \( m/\ell \) 294.1306, found \( m/\ell \) 294.1308

d) Synthesis of compound 6
To a Schlenk tube was added 1f (85 mg, 0.2 mmol), CuI (2 mg), Pd(PPh3)2Cl2 (14 mg), Et3N (1 mL) and THF (1 mL). Then the tube was charged with N2, and was stirred at room temperature for 15 h until complete consumption of starting material as monitored by TLC. After the reaction was finished, the mixture was filtered through a pad of Celite, washed with EA. The combined organic mixture was concentrated in vacuo and purified by silica gel column chromatography (1:20, EA:Hexane) to afford compound 6 as yellow oil (73 mg, 84%). 1H NMR (400 MHz, CDCl3) δ (ppm): 7.56 – 7.50 (m, 4H), 7.39 – 7.37 (m, 3H), 7.31 – 7.28 (m, 2H), 6.73 (s, 4H), 4.47 (q, J = 7 Hz, 2H), 3.76 (s, 3H), 1.43 (t, J = 7 Hz, 3H); 13C NMR (100 MHz, CDCl3) δ (ppm): 163.23 (t, J = 31 Hz), 159.07 (t, J = 30 Hz), 158.01, 139.85, 131.85, 131.82, 130.55, 129.04, 128.80, 128.55, 125.24, 123.61, 122.89, 114.13, 113.08 (t, J = 253 Hz), 91.54, 88.64, 63.16, 55.47, 14.20; 19F NMR (376 MHz, CDCl3) δ (ppm): – 104.86; HRMS (ESI) exact mass calculated for [M+H]+ (C26H22F2NO3) requires m/z 434.1568, found m/z 434.1571

e) Synthesis of compound 7[^5]

Under N2 atmosphere, to a solution of 1f (103 mg, 0.25 mmol) and Pd(PPh3)4 (15 mg, 0.0125 mmol) in dry toluene (5 mL) was added K2CO3 (69 mg, 0.5 mmol), Ph(OH)2 (61 mg, 0.5 mmol). The vigorously stirred mixture was heated to 90 °C for 1 h (monitored by TLC). After cooling, the mixture was filtered through a pad of Celite, the filter cake was washed with EA. The organic mixture was concentrated in vacuo and the residue was purified by column chromatography (1:20, EA : Hexane) to afford 7 as
yellow oil (81 mg, 79%). $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.59 – 7.56 (m, 4H), 7.44 (t, $J = 8$ Hz, 2H), 7.37 (d, $J = 8$ Hz, 3H), 6.78 – 6.71 (m, 4H), 4.47 (q, $J = 7$ Hz, 2H), 3.74 (s, 3H), 1.42 (t, $J = 7$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 163.37 (t, $J = 31$ Hz), 159.48 (d, $J = 30$ Hz), 157.86, 142.73, 140.09, 139.98, 129.60, 129.46, 129.01, 128.06, 127.29, 127.19, 123.57, 114.08, 113.24 (t, $J = 253$ Hz), 63.09, 55.43 14.19; $^{19}$F NMR (376 MHz, CDCl$_3$) δ (ppm): –104.90; HRMS (ESI) exact mass calculated for [M+H]$^+$ (C$_{24}$H$_{22}$F$_2$NO$_3$) requires m/z 410.1568, found m/z 410.1571;

Reference:
4. NMR Spectra

Ethyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-phenylpropanoate (2a)
Ethyl (E)-2,2-difluoro-3-(4-methoxyphenyl)-3-(4-methoxyphenylimino)propanoate (2b)
Ethyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-(p-tolyl)propanoate (2c)
Ethyl (E)-2,2-difluoro-3-(4-fluorophenyl)-3-((4-methoxyphenyl)imino)propanoate (2d)
Ethyl (E)-3-(4-chlorophenyl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2e)
Ethyl \((E)\)-3-(4-bromophenyl)-2,2-difluoro-3-\((4\text{-methoxyphenyl})\text{imino}\)propanoate (2f)
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Ethyl (E)-3-(3-bromophenyl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2g)
Ethyl \((E)\)-3-(2-bromophenyl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2h)
Ethyl (E)-2,2-difluoro-3-(4-iodophenyl)-3-((4-methoxyphenyl)imino)propanoate (2i)
Ethyl \((E)-2,2\text{-difluoro-3-((4-methoxyphenyl)imino)-3-(4-(trifluoromethyl)phenyl)propanoate}\) (2j)
Ethyl (E)-3-(3-chloro-4-fluorophenyl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2k)
Ethyl (E)-3-((benzo[d][1,3]dioxol-5-yl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (2l)
Ethyl (Z)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-(thiophen-2-yl)propanoate (2m)
Methyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-phenylpropanoate (2n)
Isopropyl (E)-2,2-difluoro-3-[(4-methoxyphenyl)imino]-3-phenylpropanoate (2o)
Benzyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-phenylpropanoate (2p)
(E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-1,3-diphenylpropan-1-one (2q)
(E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-phenylpropanenitrile (2r)
Ethyl (E)-2,2-difluoro-3-((3-methoxyphenyl)imino)-3-phenylpropanoate (2s)
Ethyl (E)-2,2-difluoro-3-((2-methoxyphenyl)imino)-3-phenylpropanoate (2t)
Ethyl (E)-2,2-difluoro-3-phenyl-3-(phenylimino)propanoate (2u)
Ethyl (E)-2,2-difluoro-3-((4-fluorophenyl)imino)-3-phenylpropanoate (2v)
Ethyl (E)-3-((4-chlorophenyl)imino)-2,2-difluoro-3-phenylpropanoate (2w)
Ethyl (E)-2,2-difluoro-3-phenyl-3-((4-(trifluoromethyl)phenyl)imino)propanoate (2x)
Ethyl (E)-2,2-difluoro-3-((4-nitrobenzyl)imino)-3-phenylpropanoate (2y)
Ethyl (E)-3-(cyclopropylimino)-2,2-difluoro-3-phenylpropanoate (2z)
Ethyl 2,2-difluoro-3-oxo-3-phenylpropanoate (3)
Ethyl 2,2-difluoro-3-((4-methoxyphenyl)amino)-3-phenylpropanoate (4)
Ethyl (E)-2,2-difluoro-3-((4-methoxyphenyl)imino)-3-(4-(phenylethynyl)phenyl)propanoate (6)
Ethyl (E)-3-((1,1'-biphenyl)-4-yl)-2,2-difluoro-3-((4-methoxyphenyl)imino)propanoate (7)
Ethyl (E)-2-fluoro-3-((4-methoxyphenyl)amino)-3-phenylacrylate (8)
5. Determination of the configuration of the imine C=N bond

NOESY experiment of 2a
NOESY experiment of 2r
NOESY experiment of 2y
NOESY experiment of compound 6
NOESY experiment of compound 7
6. HRMS spectrum of the reaction solution of Scheme 4b
Display Report

Acquisition Parameter
- Source Type: ESI
- Ion Polarity: Positive
- Focus: Active
- Scan Begin: 100 m/z
- Scan End: 1700 m/z
- Set Capillary: 4500 V
- Set End Plate Offset: -500 V
- Set Collision Cell RF: 150.0 Vpp
- Set Nebulizer: 0.8 Bar
- Set Dry Heater: 100 °C
- Set Dry Gas: 4.0 l/min
- Set Divert Valve: Source

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