Supporting Information for

Rhodium-catalyzed transannulation of N-(per)fluoroalkyl-1,2,3-triazoles in microwave conditions – a general route to N-(per)fluoroalkyl-substituted five-membered heterocycles†

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**General**

Chloroform stabilized with ethanol (~1%) was dried by activated molecular sieves (3 and 4 Å) and stored under argon. All commercially available chemicals were used as received unless stated otherwise. Starting triazoles were prepared according to procedures published in literature.¹ ² Triazole 1q was supplied by CF Plus Chemicals [www.cfplus.cz](http://www.cfplus.cz). Flash column chromatography was performed using silica gel 60 (0.040–0.063 mm). Automated flash column chromatography was performed on Teledyne ISCO CombiFlash Rf+ Lumen Automated Flash Chromatography System with UV/Vis detection. ¹H, ¹³C, and ¹⁹F NMR spectra were measured at ambient temperature using 5 mm diameter NMR tubes. ¹³C spectra were proton decoupled. The chemical shift values (δ) are reported in ppm relative to internal Me₄Si (0 ppm for ¹H and ¹³C NMR) or residual solvents and internal CFCl₃ (0 ppm for ¹⁹F NMR). Coupling constants (J) are reported in Hertz. Structural elucidation was aided by additional acquisition of ¹³C APT and/or various 2D spectra (¹H-¹H COSY, ¹H-¹³C HSQC, ¹H-¹³C HMBC, ¹³C-¹⁹F HMBC). GC-MS spectra were recorded on Agilent 7890A GC (column HP-5MS, 30 m × 0.25 mm × 0.25 μm, 5% phenyl methylpolysiloxane) coupled with 5975C quadrupole mass selective electron impact (EI) detector (70 eV). High resolution MS spectra (HRMS) were recorded on a Waters Micromass AutoSpec Ultima or Agilent 7890A GC coupled with Waters GCT Premier orthogonal acceleration time-of-flight detector using electron impact (EI) ionization. Rhodium catalyst Rh₂(Oct)₄ was used as a 0.01 M solution in dry chloroform. Biotage Initiator EXP EU (300 W power) was used for reactions carried out in a microwave reactor.

**General procedure for synthesis of N-(per)fluoroalkyl-imidazoles 3a-3q.** Initial N-(per)fluoroalkyl-triazole 1a-1q (0.20 mmol) was dissolved in dry CHCl₃ (2 mL) in a 5 mL microwave tube. Nitrile (2 equiv., 0.40 mmol) and a solution of rhodium (II) octanoate (0.002 mmol; 0.01 M in dry CHCl₃) were added. The vial was capped and heated at 140°C for 20 min in a microwave reactor. The resulting mixture was evaporated on silica gel (100 mg) and purified either by filtration through silica gel (washing with CH₂Cl₂) and further evaporation (55°C, 3 Torr) to remove the nitrile or by CombiFlash automatic column chromatography (EtOAc/cyclohexane, 0:100 to 10:90).

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2,4-Diphenyl-1-(trifluoromethyl)-1H-imidazole (3a): Yield: 57%; colorless oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.85–7.88 (m, 1H), 7.71–7.75 (m, 1H), 7.57 (q, \(J_{\text{CF}} = 0.9\) Hz, 1H), 7.52–7.46 (m, 3H), 7.45–7.39 (m, 2H), 7.36–7.30 (m, 1H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 147.2, 142.2, 132.4, 130.2, 129.7, 129.3 (q, \(J_{\text{CF}} = 1.5\) Hz), 128.9, 128.6, 128.1, 125.6, 118.4 (q, \(J_{\text{CF}} = 265.1\) Hz, N-CF\(_3\)), 112.5; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -52.7 (s); HRMS (EI\(^+\)) \(m/z\) calecd for C\(_{19}\)H\(_{13}\)F\(_3\)N\(_2\) [M\(^+\)]: 388.0874, found 388.0875.

2-Phenyl-4-(p-toly1)-1-(trifluoromethyl)-1H-imidazole (3b): Yield: 84%; colorless oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.77–7.72 (m, 2H), 7.72–7.67 (m, 2H), 7.54–7.52 (m, 1H), 7.51–7.44 (m, 3H), 7.25–7.21 (m, 2H), 2.39 (s, 3H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 146.9, 142.2, 137.8, 130.0, 129.7, 129.5, 129.4, 129.2 (q, \(J_{\text{CF}} = 1.5\) Hz), 128.4, 125.3, 118.3 (q, \(J_{\text{CF}} = 265.1\) Hz, N-CF\(_3\)), 111.9, 21.3; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -52.7 (s); HRMS (EI\(^+\)) \(m/z\) calecd for C\(_{17}\)H\(_{13}\)F\(_3\)N\(_2\) [M\(^+\)]: 302.1031, found 302.1032.

4-(4-Methoxyphenyl)-2-phenyl-1-(trifluoromethyl)-1H-imidazole (3c): Yield: 72%; pale yellow oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.80–7.75 (m, 2H), 7.70–7.65 (m, 2H), 6.99–6.91 (m, 2H), 3.85 (s, 3H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 159.7, 147.0, 142.0, 130.2, 129.7, 129.3 (q, \(J_{\text{CF}} = 1.5\) Hz), 128.6, 126.9, 125.1, 118.4 (q, \(J_{\text{CF}} = 265.1\) Hz, N-CF\(_3\)), 114.3, 111.4, 55.5; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -52.7 (s, 3F), -114.5 (s, 1F); HRMS (EI\(^+\)) \(m/z\) calecd for C\(_{16}\)H\(_{10}\)F\(_3\)N\(_2\)O [M\(^+\)]: 318.0980, found 318.0981.

4-(4-Fluorophenyl)-2-phenyl-1-(trifluoromethyl)-1H-imidazole (3d): Yield: 64%; colorless oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.86–7.78 (m, 2H), 7.72–7.63 (m, 2H), 7.54–7.44 (m, 4H), 6.91–6.85 (m, 2H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 157.1, 147.3, 141.4, 130.3, 129.6, 129.3 (q, \(J_{\text{CF}} = 1.4\) Hz), 128.7 (d, \(J_{\text{CF}} = 3.2\) Hz), 128.6, 127.3 (d, \(J_{\text{CF}} = 8.1\) Hz), 118.3 (q, \(J_{\text{CF}} = 265.5\) Hz, N-CF\(_3\)), 115.8 (d, \(J_{\text{CF}} = 21.7\) Hz), 112.1; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -52.7 (s, 3F), -114.5 (s, 1F); HRMS (EI\(^+\)) \(m/z\) calecd for C\(_{16}\)H\(_{10}\)F\(_3\)N\(_2\) [M\(^+\)]: 306.0780, found 306.0778.

2-Phenyl-1-(trifluoromethyl)-4-(4- trifluoromethyl)phenyl)-1H-imidazole (3e): Yield: 63%; pale yellow oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.99–7.93 (m, 2H), 7.74–7.63 (m, 5H), 7.55–7.45 (m, 3H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 147.6, 140.8, 135.9 (q, \(J_{\text{CF}} = 1.4\) Hz), 130.4, 130.0 (q, \(J_{\text{CF}} = 32.8\) Hz), 129.4, 129.3 (q, \(J_{\text{CF}} = 1.4\) Hz), 128.7, 125.9 (q, \(J_{\text{CF}} = 3.8\) Hz), 125.7 (q, \(J_{\text{CF}} = 272.0\) Hz), 125.7, 118.3 (q, \(J_{\text{CF}} = 265.7\) Hz, N-CF\(_3\)), 113.6; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -52.7 (s, 3F), -63.0 (s, 3F); HRMS (EI\(^+\)) \(m/z\) calecd for C\(_{19}\)H\(_{10}\)F\(_3\)N\(_2\) [M\(^+\)]: 356.0748, found 356.0746.

4-(4-Nitrophenyl)-2-phenyl-1-(trifluoromethyl)-1H-imidazole (3f): Yield: 52%; yellow solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.33–8.25 (m, 2H), 8.05–7.99 (m, 2H), 7.76–7.73 (m, 1H), 7.71–7.67 (m, 2H), 7.57–7.46 (m, 3H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 148.0, 147.4, 140.0, 138.7, 130.6, 129.3 (q, \(J_{\text{CF}} = 1.5\) Hz), 129.1, 128.7, 126.0, 124.4, 118.2 (q, \(J_{\text{CF}} = 266.2\) Hz, N-CF\(_3\)), 114.7; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -52.8 (s); HRMS (EI\(^+\)) \(m/z\) calecd for C\(_{16}\)H\(_{10}\)F\(_3\)N\(_2\)O [M\(^+\)]: 333.0725, found 333.0726.

2-(4-Methoxyphenyl)-4-(p-toly1)-1-(trifluoromethyl)-1H-imidazole (3g): Yield: 78%; white solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.77–7.69 (m, 2H), 7.67–7.58 (m, 2H), 7.49 (q, \(J_{\text{CF}} = 0.8\) Hz, 1H), 7.24–7.20 (m, 2H), 7.05–6.94 (m, 2H), 3.87 (s, 3H), 2.38 (s, 3H);
$^{13}$C NMR (101 MHz, CDCl$_3$) δ 161.1, 147.0, 142.0, 137.8, 130.8 (q, $J_{C,F} = 1.5$ Hz), 129.7, 129.5, 125.4, 122.2, 118.4 (q, $J_{C,F} = 264.9$ Hz, N-CF$_3$), 114.0, 111.8, 55.5, 21.4; $^{19}$F NMR (376 MHz, CDCl$_3$) δ -52.7 (s); HRMS (EI+) m/z calcd for C$_{18}$H$_{15}$F$_3$N$_2$O [M]+: 332.1136, found 332.1134.

2-(3-Methoxyphenyl)-4-(p-tolyl)-1-(trifluoromethyl)-1H-imidazole (3h): Yield: 94%; pale yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.79–7.70 (m, 2H), 7.51 (q, $^4J_{H,F} = 0.9$ Hz, 1H), 7.41–7.35 (m, 1H), 7.28–7.21 (m, 4H, signal overlap with solvent), 7.04 (ddd, $^3J = 8.3$, 2.6, 1.0 Hz, 1H), 3.86 (s, 3H), 2.38 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 159.6, 146.9, 142.2, 137.9, 130.9, 129.6 (3C), 125.4, 121.7 (q, $J_{C,F} = 1.5$ Hz), 118.4 (q, $J_{C,F} = 265.2$, N-CF$_3$), 116.3, 114.6 (q, $J_{C,F} = 1.2$ Hz), 112.0 (q, $J_{C,F} = 1.2$ Hz), 55.5, 21.4; $^{19}$F NMR (376 MHz, CDCl$_3$) δ -52.6 (s); HRMS (EI+) m/z calcd for C$_{18}$H$_{15}$F$_3$N$_2$O [M]+: 332.1136, found 332.1133.

2-(4-Chlorophenyl)-4-(p-tolyl)-1-(trifluoromethyl)-1H-imidazole (3i): Yield: 82%; colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.75–7.68 (m, 2H), 7.67–7.59 (m, 2H), 7.52 (q, $^4J_{H,F} = 0.9$ Hz, 1H), 7.50–7.42 (m, 2H), 7.25–7.21 (m, 2H), 2.38 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 145.8, 142.5, 138.1, 136.5, 130.7 (q, $J_{C,F} = 1.4$ Hz), 129.6, 129.4, 128.9, 128.2, 125.4, 118.3 (q, $J_{C,F} = 265.1$ Hz, N-CF$_3$), 112.2, 21.4; $^{19}$F NMR (376 MHz, CDCl$_3$) δ -52.7 (s); HRMS (EI+) m/z calcd for C$_{17}$H$_{12}$ClF$_3$N$_2$O [M]+: 336.0641, found 336.0642.

4-(4-Methoxyphenyl)-2-(4-nitrophenyl)-1-(trifluoromethyl)-1H-imidazole (3j): Yield: 33%; purification by column chromatography on C18 reverse-phase silica (H$_2$O/MeCN, 80:20 to 20:80); yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.37–8.32 (m, 2H), 7.95–7.88 (m, 2H), 7.81–7.73 (m, 2H), 7.53 (q, $^4J_{H,F} = 0.9$ Hz, 1H), 7.03–6.92 (m, 2H), 3.85 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 160.0, 148.7, 144.4, 142.9, 135.7, 130.3, 126.9, 124.6, 123.8 (m), 118.3 (q, $J_{C,F} = 265.6$ Hz, N-CF$_3$), 114.4 (m), 112.4, 55.5 (q, $J_{C,F} = 10.8$ Hz); $^{19}$F NMR (376 MHz, CDCl$_3$) δ -52.4 (s); HRMS (EI+) m/z calcd for C$_{17}$H$_{12}$F$_3$N$_3$O$_3$ [M]+: 363.0831, found 363.0828.

2-Methyl-4-(p-tolyl)-1-(trifluoromethyl)-1H-imidazole (3k): Yield: 71%; colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.67–7.57 (m, 2H), 7.33 (s, 1H), 7.23–7.16 (m, 2H), 2.59 (q, $^3J_{H,F} = 1.4$ Hz, 3H), 2.37 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 144.6, 141.4, 137.7, 129.7, 129.5, 125.2, 118.5 (q, $J_{C,F} = 263.8$ Hz, N-CF$_3$), 110.8, 21.4, 14.4 (q, $J_{C,F} = 2.4$ Hz); $^{19}$F NMR (376 MHz, CDCl$_3$) δ -56.2 (s); HRMS (EI+) m/z calcd for C$_{12}$H$_{11}$F$_3$N$_2$ [M]+: 240.0874, found 240.0876.
2-(3,4-Dimethoxybenzyl)-4-(p-toly1)-1-(trifluoromethyl)-1H-imidazole (3I): Yield: 56%; yellow oil; 1H NMR (400 MHz, CDCl3) δ 7.71–7.65 (m, 2H), 7.35 (d, JHH = 0.8 Hz, 1H), 7.23–7.19 (m, 2H), 6.86 (s, 1H), 6.79 (d, JHH = 1.0 Hz, 2H), 4.20 (d, JHH = 1.1 Hz, 2H), 3.85 (s, 3H), 3.84 (s, 3H), 2.37 (s, 3H); 13C NMR (101 MHz, CDCl3) δ 149.0, 148.1, 146.6, 141.7, 137.8, 129.8, 129.6, 129.5, 128.5, 125.3, 120.9–120.5 (m), 118.4 (q), 1F NMR (264.6 Hz, N-CF3), 111.9 (m), 111.2 (m), 56.9 (q, JCF = 11.2 Hz), 34.5–33.8 (m), 21.4 (q, JCF = 7.9 Hz); 19F NMR (376 MHz, CDCl3) δ -55.1 (s); HRMS (EI) m/z caleed for C20H19F3N2O [M]+: 376.1399, found 376.1397.

Ethyl 2-phenyl-1-(trifluoromethyl)-1H-imidazole-4-carboxylate (3m): Yield: 65%, colorless oil; 1H NMR (400 MHz, CDCl3) δ 7.97 (q, JHF = 0.9 Hz, 1H), 7.67–7.59 (m, 2H), 7.55–7.41 (m, 3H), 4.42 (q, JHF = 7.2 Hz, 2H), 1.39 (t, JHH = 7.2 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 161.9, 147.6, 134.2, 130.7, 129.4 (q, JCF = 1.3 Hz), 128.6, 128.5, 122.9 (q, JCF = 1.2 Hz), 117.9 (q, JCF = 267.1 Hz, N-CF3), 61.4, 14.5; 19F NMR (376 MHz, CDCl3) δ -55.1 (s); HRMS (EI) m/z caleed for C10H13F3N2O [M]+: 284.0773, found 284.0770.

4-(4-Methoxyphenyl)-1-(perfluoroethyl)-1H-imidazole (3n): Yield: 92%; yellow solid; 1H NMR (400 MHz, CDCl3) δ 7.82–7.73 (m, 2H), 7.62–7.54 (m, 2H), 7.52–7.41 (m, 3H), 7.39–7.35 (m, 1H), 7.01–6.90 (m, 2H), 3.84 (s, 3H); 13C NMR (101 MHz, CDCl3) δ 159.8, 148.2, 142.8, 130.5, 130.0 (2C), 128.2, 126.9, 125.0, 117.6 (q, JCF = 288.0 Hz, 2JCF = 44.9 Hz, CF3), 114.3, 111.6, 110.6 (tq, JCF = 269.2 Hz, 2JCF = 44.9 Hz, N-CF3), 55.5; 19F NMR (376 MHz, CDCl3) δ 84.8 (s, 3F), -93.9 (s, 2F); HRMS (EI) m/z caleed for C18H15F3N2O [M]+: 368.0948, found 368.0954.

1-(Perfluoropropyl)-2,4-diphenyl-1H-imidazole (3o): Yield: 71%, white solid; 1H NMR (500 MHz, CDCl3) δ 7.91–7.84 (m, 2H), 7.61–7.55 (m, 2H), 7.51–7.39 (m, 6H), 7.37–7.31 (m, 1H); 13C NMR (101 MHz, CDCl3) δ 148.4, 142.7, 132.2, 130.4, 130.1, 128.9, 122.2 (2C), 125.6, 117.4 (qtt, 1JCF = 287.7 Hz, 2JCF = 33.3 Hz, 3JCF = 2.1 Hz, CF3), 112.9, 112.4 (t, 1JCF = 269.8 Hz, 2JCF = 32.1 Hz, N-CF3), 110.2–105.6 (m); 19F NMR (376 MHz, CDCl3) δ -80.6 (t, 3JCF = 9.7 Hz, 3F), -89.8 (q, 1JCF = 9.7 Hz, 2F), -126.1 (s, 2F); HRMS (EI) m/z caleed for C18H15F3N2O [M]+: 388.0810, found 388.0809.

2-Phenyl-1-(1,1,2,2-tetrafluoro-2-phenoxymethyl)-4-(p-toly1)-1H-imidazole (3p): Yield: 57%; brown oil; 1H NMR (400 MHz, CDCl3) δ 7.81–7.73 (m, 2H), 7.68–7.62 (m, 2H), 7.56 (s, 1H), 7.52–7.38 (m, 3H), 7.40–7.30 (m, 2H), 7.30–7.19 (m, 3H, signal overlap with solvent), 7.10–7.02 (m, 2H), 2.38 (s, 3H); 13C NMR (101 MHz, CDCl3) δ 148.6, 148.4, 142.3, 137.7, 131.2, 130.1, 129.9 (2C), 129.7, 129.5, 128.0, 127.0, 125.4, 121.5, 116.3 (tt, 1JCF = 277.3 Hz, 2JCF = 40.8 Hz), 113.2, 111.9 (tt, 1JCF = 268.8 Hz, 2JCF = 40.8 Hz), 21.4; 19F NMR (376 MHz, CDCl3) δ -86.3 (t, 3JCF = 4.2 Hz, 2F), -93.7 (t, 3JCF = 4.2 Hz, 2F); HRMS (EI) m/z caleed for C24H18F3N2O [M]+: 426.1355, found 426.1356.

1-(2-(2,4-Diphenyl-1H-imidazol-1-yl)-1,1,2,2-tetrafluoroethyl)-1H-pyrazole (3q): Yield: 70%; red oil; 1H NMR (400 MHz, CDCl3) δ 7.83–7.76 (m, 2H), 7.72 (qd, J = 1.5, 0.6 Hz, 1H), 7.64–7.58 (m, 1H), 7.54–7.35 (m, 1H), 7.35–7.26 (m, 1H), 7.25 (s, 1H, signal overlap with solvent), 6.42 (dd, J = 2.7, 1.7 Hz, 1H); 13C NMR (101 MHz, CDCl3) δ 148.3, 143.9, 142.4, 132.4, 130.5, 130.0, 129.8, 129.1, 128.8, 128.0 (2C), 125.5, 113.1, 112.6 (tt, 1JCF = 271.3 Hz, 2JCF = 42.1 Hz), 112.5 (tt, 1JCF = 269.2 Hz, 2JCF = 42.1 Hz), 108.9; 19F NMR (376 MHz, CDCl3) δ -92.1 (t, 3JCF = 4.7 Hz, 2F), -98.2 (t, 3JCF = 4.7 Hz, 2F); HRMS (EI) m/z caleed for C20H14F4N4 [M]+: 386.1155, found 386.1156.
General procedure for synthesis of N-(per)fluoroalkyl-pyrroles 4a-4i. N-(per)fluoroalkyl-triazole I (0.20 mmol) was dissolved in dry CHCl₃ (2 mL) in a 5 mL microwave tube. Vinyl ether (10 equiv., 2.0 mmol) and a solution of rhodium (II) octanoate (0.002 mmol; 0.01 M in dry CHCl₃) were added. The vial was capped and heated at 140°C for 20 min in a microwave reactor. The resulting mixture was evaporated on silica gel (100 mg) and purified by CombiFlash automatic column chromatography (cyclohexane).

In case of derivatives 4e and 4g the non-eliminated products were observed. For preparation of the desired pyrroles was developed one-pot two-step procedure.

![Diagram](https://via.placeholder.com/150)

One-pot two-step procedure for preparation of pyrroles 4e and 4g. N-perfluoroalkyl-triazole (0.20 mmol) was dissolved in dry CHCl₃ (2 mL) in a 5 mL microwave tube. Vinyl ether (10 equiv., 2.0 mmol) and a solution of rhodium (II) octanoate (0.002 mmol; 0.01 M in dry CHCl₃) were added. The vial was capped and heated at 140°C for 20 min in a microwave reactor. Then TsOH·H₂O (0.40 mmol; 76.1 mg) was added. The resulting suspension was stirred at rt for 2 h filtered, evaporated on silica gel (100 mg) and purified by CombiFlash automatic column chromatography (cyclohexane).

3-Phenyl-1-(trifluoromethyl)-1H-pyrrole (4a): Yield: 96%; white solid; ¹H NMR (400 MHz, CDCl₃) δ 7.55–7.48 (m, 2H), 7.41–7.34 (m, 2H), 7.30–7.21 (m, 2H), 7.03 (dd, J = 3.3, 2.3 Hz, 1H), 6.63 (ddq, J = 3.3, 1.6, 0.8 Hz, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 133.9, 128.9, 128.4, 127.0, 125.8, 119.5 (q, ¹JCF = 260.1 Hz, N-CF₃), 118.8, 113.9, 110.6; ¹⁹F NMR (376 MHz, CDCl₃) δ -57.5 (s); HRMS (EI⁺) m/z calcd for C₁₁H₈F₃N [M⁺]: 211.0609, found 211.0611.

3-(4-Methoxyphenyl)-1-(trifluoromethyl)-1H-pyrrole (4b): Yield: 93%; white solid; ¹H NMR (400 MHz, CDCl₃) δ 7.49–7.40 (m, 2H), 7.16 (t, J = 2.0 Hz, 1H), 7.01 (dd, J = 3.3, 2.3 Hz, 1H), 6.96–6.88 (m, 2H), 6.57 (ddq, J = 3.2, 1.7, 0.7 Hz, 1H), 3.83 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 158.8, 128.1, 126.9, 126.7, 119.1 (q, ¹JC,F = 260.2 Hz, N-CF₃), 118.7, 114.4, 113.1, 110.5, 55.5; ¹⁹F NMR (376 MHz, CDCl₃) δ -57.5 (s); HRMS (EI⁺) m/z calcd for C₁₃H₁₀F₃NO [M⁺]: 241.0714, found 241.0712.

3-(p-Tolyl)-1-(trifluoromethyl)-1H-pyrrole (4c): Yield: 82%; white solid; ¹H NMR (400 MHz, CDCl₃) δ 7.44–7.39 (m, 2H), 7.25–7.16 (m, 3H), 7.02 (dd, J = 3.2, 2.3 Hz, 1H), 6.60 (ddq, J = 3.2, 1.5, 0.7 Hz, 1H).
Hz, 1H), 2.37 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 136.7, 131.1, 129.6, 128.4, 125.6, 119.1 (q, $^1J_{C,F}$ = 260.2 Hz, N-CF$_3$), 118.7, 113.5, 110.6, 21.3; $^{19}$F NMR (376 MHz, CDCl$_3$) δ -57.5 (s); HRMS (EI+) m/z calcd for C$_{12}$H$_{10}$F$_3$N [M$^+$]: 225.0765, found 225.0762.

3-(4-Fluorophenyl)-1-(trifluoromethyl)-1H-pyrazole (4d): Yield: 80%; white solid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.50–7.42 (m, 2H), 7.19 (t, $J$ = 2.0 Hz, 1H), 7.10–7.04 (m, 2H), 7.03 (dd, $J$ = 3.2, 2.3 Hz, 1H), 6.57 (ddq, $J$ = 3.3, 1.5, 0.7 Hz, 1H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 162.1 (d, $^1J_{C,F}$ = 245.7 Hz), 130.1 (d, $^1J_{C,F}$ = 3.3 Hz), 127.5, 127.3 (d, $^3J_{C,F}$ = 8.0 Hz), 119.2 (q, $^1J_{C,F}$ = 260.8 Hz, N-CF$_3$), 118.9, 115.8 (d, $^2J_{C,F}$ = 21.6 Hz), 113.7, 110.5; $^{19}$F NMR (376 MHz, CDCl$_3$) δ -57.5 (s, 3F), -116.2 (s, 1F); HRMS (EI+) m/z calcd for C$_{12}$H$_{10}$F$_3$N [M$^+$]: 229.0515, found 229.0514.

2-Ethoxy-1-(perfluoroethyl)-4-phenyl-2,3-dihydro-1H-pyrrrole (4e'): not isolated; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.38–7.28 (m, 4H), 7.24–7.18 (m, 1H), 6.56 (s, 1H), 5.45 (d, $J$ = 7.6 Hz, 1H), 3.62 (dq, $J$ = 9.2, 7.0 Hz, 1H), 3.52 (dq, $J$ = 9.2, 7.0 Hz, 1H), 3.32–3.20 (m, 1H), 2.85 (m, 1H), 1.22 (t, $J$ = 7.0 Hz, 3H); $^{19}$F NMR (376 MHz, CDCl$_3$) δ -83.3 (s, 3F), -93.4 (d, $^2J_{F,F}$ = 212.9 Hz, 1F), -95.8 (d, $^3J_{F,F}$ = 212.9 Hz, 1F).

1-(Perfluoroethyl)-3-phenyl-1H-pyrrrole (4f): Yield: 89%; white solid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.58–7.48 (m, 2H), 7.42–7.34 (m, 2H), 7.31–7.22 (m, 1H), 7.20 (tt, $J$ = 1.7, 0.8 Hz, 1H), 7.02–6.95 (m, 1H), 6.67 (ddt, $J$ = 3.4, 1.7, 0.9 Hz, 1H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 133.9, 129.0, 128.6, 127.0, 125.8, 119.3, 117.8 (qt, $^1J_{C,F}$ = 287.6 Hz, $^2J_{C,F}$ = 47.0 Hz, CF$_3$), 114.4 110.9, 110.8 (tq, $^1J_{C,F}$ = 263.8 Hz, $^2J_{C,F}$ = 41.8 Hz, N-CF$_2$); $^{19}$F NMR (376 MHz, CDCl$_3$) δ -85.9 (s, 3F), -99.1 (s, 2F); HRMS (EI+) m/z calcd for C$_{12}$H$_{10}$F$_3$N [M$^+$]: 261.0577, found 261.0578.

1-(1,1,2,2-Tetrafluoro-2-(3-phenyl-1H-pyrrol-1-yl)ethyl)-1H-pyrazole (4f): Yield: 92%; colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.77 (tq, $J$ = 1.5, 0.7 Hz, 1H), 7.58–7.51 (m, 1H), 7.48–7.43 (m, 2H), 7.39–7.32 (m, 2H), 7.28–7.19 (m, 1H), 6.96 (ddt, $J$ = 2.3, 1.6, 0.7 Hz, 1H), 6.77 (ddd, $J$ = 3.3, 1.9, 0.6 Hz, 1H), 6.57 (ddt, $J$ = 3.2, 1.8, 1.0 Hz, 1H), 6.41 (ddt, $J$ = 2.8, 1.7, 0.6 Hz, 1H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 143.6, 134.0, 129.1, 128.9, 128.1, 126.8, 119.3, 114.4, 112.8 (tt, $^1J_{C,F}$ = 268.1 Hz, $^2J_{C,F}$ = 40.9 Hz), 112.7 (tt, $^1J_{C,F}$ = 269.2 Hz, $^3J_{C,F}$ = 43.5 Hz), 110.3, 108.6; $^{19}$F NMR (376 MHz, CDCl$_3$) δ -97.8 (t, $^1J_{F,F}$ = 5.6 Hz, 2F), -100.1 (t, $^3J_{F,F}$ = 5.6 Hz, 2F); HRMS (EI+) m/z calcd for C$_{12}$H$_{10}$F$_3$N$_3$ [M$^+$]: 309.0889, found 309.0888.

Ethyl 5-ethyl-1-(trifluoromethyl)-4,5-dihydro-1H-pyrrrole-3-carboxylate (4g'): not isolated; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.12–7.08 (m, 1H), 5.41 (ddq, $J$ = 8.1, 2.3, 0.9 Hz, 1H), 4.19 (q, $J$ = 7.2 Hz, 2H), 3.68–3.56 (m, 1H), 3.54–3.44 (m, 1H), 3.11–2.99 (m, 1H), 2.83–2.74 (m, 1H), 1.28 (t, $^1J_{H,F}$ = 7.1 Hz, 3H), 1.20 (t, $^3J_{H,F}$ = 7.0 Hz, 3H); $^{19}$F NMR (376 MHz, CDCl$_3$) δ -57.9 (s).
**Ethyl 1-(trifluoromethyl)-1H-pyrole-3-carboxylate (4g):** Yield: 92%; colorless oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.60 (dd, \(J = 2.3, 1.6\) Hz, 1H), 6.96 (dd, \(J = 3.3, 2.3\) Hz, 1H), 6.72 (ddq, \(J = 3.3, 1.7, 0.9\) Hz, 1H), 4.30 (q, \(J_{CH} = 7.1\) Hz, 2H), 1.35 (t, \(J_{CH} = 7.1\) Hz, 3H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 163.6, 122.5, 118.5, 118.5 (q, \(J_{CF} = 262.6\) Hz, N-CF\(_3\)), 112.6, 60.6, 14.5; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -57.9 (s); HRMS (EI+) \(m/z\) calcd for C\(_8\)H\(_3\)F\(_3\)NO\(_2\) [M]+: 207.0507, found 207.0506.

**2-Methyl-4-(p-tolyl)-1-(trifluoromethyl)-1H-pyrole (4i):** Yield: 63%; white solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.42–7.33 (m, 2H), 7.17 (dddd, \(J = 7.6, 2.0, 1.2, 0.6\) Hz, 2H), 7.13 (dt, \(J = 1.9, 0.6\) Hz, 1H), 6.34–6.28 (m, 1H), 2.38 (dq, \(J = 2.0, 1.4\) Hz, 3H), 2.36 (s, 3H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 136.4, 131.3, 129.7, 125.4, 119.5 (q, \(J_{CF} = 261.0\) Hz, N-CF\(_3\)), 113.5 (q, \(J = 2.1\) Hz), 110.4 (q, \(J = 1.6\) Hz), 21.3, 12.7 (q, \(J = 2.4\) Hz); \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -55.7 (s); HRMS (EI+) \(m/z\) calcd for C\(_{13}\)H\(_{12}\)F\(_3\)N [M]+: 239.0922, found 239.0924.

**Preparation of imidazolone 6**

N-perfluoroalkyl-triazole 1b (0.20 mmol) was dissolved in dry CHCl\(_3\) (2 mL) in a 5 mL microwave tube. Phenyl isocyanate (2 equiv., 0.4 mmol) and a solution of rhodium (II) octanoate (0.002 mmol; 0.01 M in dry CHCl\(_3\)) were added. The vial was capped and heated at 120°C for 20 min in a microwave reactor. The resulting mixture was evaporated on silica gel (100 mg) and purified by CombiFlash automatic column chromatography using the (EtOAc/cyclohexane).

**3-Phenyl-4-(p-tolyl)-1-(trifluoromethyl)-1,3-dihydro-2H-imidazol-2-one (6):** Yield: 75%; brown oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.38–7.28 (m, 3H), 7.21–7.15 (m, 2H), 7.08–7.02 (m, 2H), 6.98–6.92 (m, 2H), 6.56 (s, 1H), 2.31 (s, 3H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 149.6, 138.9, 134.2, 129.5, 129.2, 128.0, 127.7, 127.4, 127.1, 125.0, 118.5 (q, \(J_{CF} = 263.0\) Hz, N-CF\(_3\)), 103.3 (q, \(J = 1.4\) Hz), 21.4; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -59.1 (s); HRMS (EI+) \(m/z\) calcd for C\(_{17}\)H\(_{13}\)F\(_3\)N\(_2\)O [M]+: 318.0980, found 318.0979.

**Preparation of pyrrolone 7**

N-perfluoroalkyl-triazole 1b (0.20 mmol) was dissolved in dry CHCl\(_3\) (2 mL) in a 5 mL microwave tube. Ketene t-butyldimethylsilyl methyl acetal (2 equiv., 0.4 mmol) and a solution of rhodium (II) octanoate (0.002 mmol; 0.01 M in dry CHCl\(_3\)) were added. The vial was capped and heated at 120°C for 15 min in microwave reactor. Then 1M solution of TBAF (5 equiv., 1 mmol) in THF was added and resulting solution was stirred for 1 h, evaporated on silica gel (100 mg) and purified by CombiFlash automatic column chromatography (EtOAc/cyclohexane).
4-(p-Tolyl)-1-(trifluoromethyl)-1,5-dihydro-2H-pyrrrol-2-one (7): Yield: 63%; slightly yellow crystals; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.46–7.39 (m, 2H), 7.30–7.22 (m, 2H, signal overlap with solvent), 6.39–6.33 (m, 1H), 4.64–4.59 (m, 2H), 2.41 (s, 3H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 169.5, 157.2, 142.2, 130.0, 127.7, 126.1, 119.6 (q, \(^1\)J\(_{\text{C-F}}\) = 261.3 Hz, N-CF\(_3\)), 118.0 (q, \(J = 2.0\) Hz), 49.5, 21.6; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -57.7 (s); HRMS (EI+) m/z calcd for C\(_{12}\)H\(_{10}\)F\(_3\)NO [M]+: 241.0714, found 241.0711.

Copies of \(^1\)H, \(^{13}\)C and \(^{19}\)F NMR Spectra

\(^1\)H NMR (400 MHz, CDCl\(_3\)) of 3a

\(^{13}\)C NMR (101 MHz, CDCl\(_3\)) of 3a

\(^{19}\)F NMR (376 MHz, CDCl\(_3\)) of 3a
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3a
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3a
$^1$H NMR (400 MHz, CDCl$_3$) of 3b
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3b
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3b
$^1$H NMR (400 MHz, CDCl$_3$) of 3c

![NMR Spectrum]

3c
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3c
$^{19}\text{F NMR (376 MHz, CDCl\textsubscript{3}) of 3c}$
$^1$H NMR (400 MHz, CDCl$_3$) of 3d
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3d
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3d

![NMR Spectrum](image_url)
$^1$H NMR (400 MHz, CDCl$_3$) of 3e
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3e
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3e
$^1$H NMR (400 MHz, CDCl$_3$) of 3f
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3f
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3f
$^1$H NMR (400 MHz, CDCl$_3$) of 3g

![NMR spectrum of 3g]
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3g
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3g
$^1$H NMR (400 MHz, CDCl$_3$) of 3h
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3h
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3h
$^1$H NMR (400 MHz, CDCl$_3$) of $3i$
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3i
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3i
$^1$H NMR (400 MHz, CDCl$_3$) of 3j

![NMR spectrum of 3j](image-url)
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3j
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3j

![NMR Spectrum]
$^{1}$H NMR (400 MHz, CDCl$_3$) of 3k
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3k

![Chemical Structure](image)

- 14.40
- 14.43
- 14.45
- 14.47
- 21.38
- 110.83
- 114.54
- 117.15
- 119.77
- 122.39
- 125.21
- 129.53
- 129.73
- 137.70
- 141.43
- 144.55

N
N
C
H
3
C
H
3
F
F
3k
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3k

![Chemical structure of 3k](image)
$^1$H NMR (400 MHz, CDCl$_3$) of 31
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3l
NMR (376 MHz, CDCl$_3$) of 3l

![NMR Spectrum of 3l](image)

**Chemical Structure of 3l**

![Chemical Structure](image)
$^1$H NMR (400 MHz, CDCl$_3$) of 3m
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3m

![NMR spectrum of 3m](image)
$^{19}$F NMR (376 MHz, CDCl$_3$) of $3m$
$^1$H NMR (400 MHz, CDCl$_3$) of 3n
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3n
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3n
$^1$H NMR (500 MHz, CDCl$_3$) of 3o
APT $^{13}$C NMR (101 MHz, CDCl$_3$) of 3o
$^{19}$F NMR (376 MHz, CDCl$_3$) of 3o
$^1$H NMR (400 MHz, CDCl₃) of 3p

3p

14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3p
$^{19}\text{F NMR (376 MHz, CDCl}_3\text{)}$ of 3p

![Chemical Structure of 3p](image)

$\delta$ (ppm): 2.03, 2.04

$\text{N}$

$\text{F}$

$\text{O}$

$\text{F}$

$\text{F}$

$\text{C}$

$\text{H}_3$

$\text{3p}$
$^1$H NMR (400 MHz, CDCl$_3$) of 3q

![Chemical structure of 3q](image)
$^{13}$C NMR (101 MHz, CDCl$_3$) of 3q
$^{19}\text{F NMR (376 MHz, CDCl}_3\text{)}$ of 3q
$^1$H NMR (400 MHz, CDCl$_3$) of 4a

![NMR Spectrogram](image-url)
$^{13}$C NMR (101 MHz, CDCl$_3$) of 4a
$^{19}\text{F NMR (376 MHz, CDCl}_3\text{) of 4a}$

![Chemical structure of 4a with fluorine atoms indicated](image)
$^1$H NMR (400 MHz, CDCl$_3$) of 4b

![NMR spectrum of 4b](image_url)
$^{13}$C NMR (101 MHz, CDCl$_3$) of 4b
$^{19}$F NMR (376 MHz, CDCl$_3$) of 4b
$^1$H NMR (400 MHz, CDCl$_3$) of 4c
$^{13}$C NMR (101 MHz, CDCl$_3$) of 4c
$^{19}$F NMR (376 MHz, CDCl$_3$) of 4c

![NMR spectrum of 4c](image)
$^1$H NMR (400 MHz, CDCl$_3$) of 4d
$^{13}$C NMR (101 MHz, CDCl$_3$) of 4d

![Chemical Structure of 4d]
$^{19}$F NMR (376 MHz, CDCl$_3$) of 4d
$^1$H NMR (400 MHz, CDCl$_3$) of 4e$^-$
$\text{19F NMR (376 MHz, CDCl}_3\text{) of 4e}'$
$^{1}$H NMR (400 MHz, CDCl₃) of 4e
$^{13}$C NMR (101 MHz, CDCl$_3$) of 4e

![Diagram of 4e molecule]
$^{19}$F NMR (376 MHz, CDCl$_3$) of 4e
$^1$H NMR (400 MHz, CDCl$_3$) of 4f
$^{13}$C NMR (101 MHz, CDCl$_3$) of 4f
$^{19}$F NMR (376 MHz, CDCl$_3$) of 4f
$^1$H NMR (400 MHz, CDCl$_3$) of 4g'
$^{19}$F NMR (376 MHz, CDCl$_3$) of 4g$^-$
$^1$H NMR (400 MHz, CDCl$_3$) of 4g
$^{13}$C NMR (101 MHz, CDCl$_3$) of 4g
$^{19}$F NMR (376 MHz, CDCl$_3$) of 4g
$^1$H NMR (400 MHz, CDCl$_3$) of 4h
$^{13}$C NMR (101 MHz, CDCl$_3$) of 4h
$^{19}$F NMR (376 MHz, CDCl$_3$) of 4h
$^1$H NMR (400 MHz, CDCl$_3$) of 4i
$^{13}$C NMR (101 MHz, CDCl$_3$) of 4i
$^{19}$F NMR (376 MHz, CDCl$_3$) of 4i
$^1$H NMR (400 MHz, CDCl$_3$) of 6

![Diagram of molecule 6]
$^{13}$C NMR (101 MHz, CDCl$_3$) of 6
$^{19}$F NMR (376 MHz, CDCl$_3$) of 6

![NMR spectrum of compound 6](image-url)
$^1$H NMR (400 MHz, CDCl$_3$) of 7

C$_3$H$_7$NOF$_3$F
$^{13}$C NMR (101 MHz, CDCl$_3$) of 7

![Chemical Structure of 7](image-url)
\textbf{\( ^{19}\text{F} \) NMR (376 MHz, CDCl\(_3\)) of 7}

\begin{center}
\includegraphics[width=0.4\textwidth]{figure.png}
\end{center}

\textit{Competition experiment}

N-perfluoroalkyl triazole 1d (0.1 mmol; 1 equiv.), N-tosyl triazole 8 (0.1 mmol; 1 equiv.) and benzonitrile (0.1 mmol; 1 equiv.) were dissolved in dry CHCl\(_3\) (2 mL) and a solution of rhodium (II) octanoate (0.001 mmol; 0.01 M in dry CHCl\(_3\)) was added. The vial was capped and mixture was heated at 140°C for 20 min in microwave reactor followed by measurement of \( ^{19}\text{F} \{^{1}\text{H}\} \) NMR spectra.
Stability of N-CF$_3$ imidazole 3g and pyrrole 4b in acidic and basic conditions

Stability of imidazole 3g

Imidazole 3g (18 mg; 0.05 mmol) was dissolved in CD$_3$OD (1.06 mL) and PhCF$_3$ was added as an internal standard. Then $^{19}$F and $^1$H NMR spectra were measured. For stability experiment in basic condition, NaOH (10 mg; 0.25 mmol) was added to the prepared solution (500 µL) and after 18 h at 25 °C $^{19}$F and $^1$H NMR spectra were measured. In case of experiment in acidic conditions, 98% H$_2$SO$_4$ in D$_2$O (40 µL) was added to the prepared solution (560 µL) and after 18 h at room temperature $^{19}$F and $^1$H NMR spectra were measured.
$^1$H NMR (400 MHz, CD$_3$OD) of 3g and PhCF$_3$ (as a standard) before experiment
\(^{19}\text{F NMR (376 MHz, CD}_2\text{OD)}\) of \(3g\) and PhCF\(_3\) (as a standard) before experiment.
1H NMR (400 MHz, CD$_3$OD) of 3g and PhCF$_3$ (as a standard) after addition of acid 18 h
$^{19}$F NMR (376 MHz, CD$_3$OD) of $3g$ and PhCF$_3$ (as a standard) after addition of acid 18 h
$^{1}$H NMR (400 MHz, CD$_3$OD) of 3g and PhCF$_3$ (as a standard) after addition of base 18 h

ratio H/D = 81:19
$^{19}$F NMR (376 MHz, CD$_3$OD) of $3g$ and PhCF$_3$ (as a standard) after addition of base 18 h

Stability of pyrrole 4b

Pyrrole 4b (2.4 mg; 0.01 mmol) was dissolved in CD$_3$OD (1.06 mL) and PhCF$_3$ was added as an internal standard. Then $^{19}$F and $^1$H NMR spectra were measured. For stability experiment in basic condition, NaOH (10 mg; 0.25 mmol) was added to the prepared solution (500 µL) and after 18 h at 25 °C $^{19}$F and $^1$H NMR spectra were measured. In case of experiment in acidic conditions, 98% H$_2$SO$_4$ in D$_2$O (40 µL) was added to the prepared solution (560 µL) and after 18 h at room temperature $^{19}$F and $^1$H NMR spectra were measured.
$^1$H NMR (400 MHz, CD$_3$OD) of 4b and PhCF$_3$ (as a standard) before experiment.
$^{19}$F NMR (376 MHz, CD$_3$OD) of 4b and PhCF$_3$ (as a standard) before experiment
\textsuperscript{1}H NMR (400 MHz, CD\textsubscript{3}OD) of 4b and PhCF\textsubscript{3} (as a standard) after addition of acid 18 h
$^{19}$F NMR (376 MHz, CD$_3$OD) of 4b and PhCF$_3$ (as a standard) after addition of acid 18 h
$^1$H NMR (400 MHz, CD$_3$OD) of 4b and PhCF$_3$ (as a standard) after addition of base 18 h

![NMR Spectrum](image-url)
$^{19}$F NMR (376 MHz, CD$_3$OD) of 4b and PhCF$_3$ (as a standard) after addition of base 18 h