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Supporting Information

for

CF3-containing Spiro-epoxyoxindoles via Corey-Chaykovsky Reaction

of N-Alkyl Isatins with $Ph_2S^+CH_2CF_3OTf^-$

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List of content

1. General ·····	••••••\$2
2. General Experimental Procedure ······	······\$2-\$3
3. Characterization Data of the Products	······\$3-\$7
4. NMR spectra	······S8-S33

1. General

All isolated compounds were characterized on Varian 300 and Bruker 400 spectrometers in CDCl₃. Chemical shifts were reported as δ values relative to internal CHCl₃ (δ 7.26 for ¹H NMR and 77.0 for ¹³C NMR). ¹⁹F NMR chemical shifts were determined as δ values relative to external standard PhCF₃ at -63.00. High-resolution mass spectra (HRMS) were obtained on a 4G mass spectrometer by using electrospray ionization (ESI) analyzed by quadrupole time-of- flight (QTof). All melting points were measured with the samples after column chromatography and uncorrected. Column chromatography was performed on silica gel. Anhydrous THF was distilled over sodium benzophenone ketyl under Ar. All other solvents and reagents were used as obtained from commercial sources without further purification.

2. General Experimental Procedure

General Procedure for the Preparation of 3a-n.



To a solution of compound **1a** (81 mg, 0.50 mmol) and compound **2** (418 mg, 1.00 mmol, 2.0 equiv) in THF (5.0 mL) was added TBAT (540 mg, 1.00 mmol, 2.0 equiv) at room temperature. After 12 h, **1a** was completely depleted monitoring on TLC, the solvent was removed and the resulting residue was purified by flash column chromatography (PE:EA = 20:1) to give **3a** (113 mg, 93%) as a white solid.

3b–n were prepared following a similar method. Conditions: **1** (0.50 mmol), **2** (1.00 mmol, 2.0 equiv), TBAT (1.00 mmol, 2.0 equiv), solvent (5 mL), room temperature, air.

Scaled-up experiment of 1a

To a solution of compound **1a** (1.0 g, 6.2 mmol) and compound **2** (5.2 g, 12 mmol, 2.0 equiv) in THF (31.0 mL) was added TBAT (6.7 g, 12 mmol, 2.0 equiv) at room temperature. After 12 h was completely depleted monitoring on TLC, the solvent was removed and the resulting residue was purified by flash column chromatography (PE:EA = 20:1) to give **3a** (1.4 g, 93%) as a white solid. **Procedure for the Preparation of compound 4 and 5.**



3b (160 mg, 0.501 mmol), styrene (78 mg, 0.75 mmol, 1.5 equiv) were dissolved in 50 ml dry DCM in a schlenk quartz tube was degassed, refilled with Ar for three times, and then TPT (10 mg, 0.025 mmol, 0.05 eq) was added. The solution was radiated with 30 W LED UV lamp (365 nm) until **3b** was completely depleted. After the solvent was removed under reduced pressure, the residue was separated by flash column chromatography (PE:EA = 20:1) to give **4a** (74 mg, 35%) as a white solid and **4b** (91 mg, 43%) as a white solid.



To a solution of compound **3a** (49 mg, 0.20 mmol) and trimethylallylsilane (46 mg, 0.40 mmol, 2.0 equiv) in dry DCM (2 mL) was added BF₃ • Et₂O (57 mg, 0.40 mmol, 2.0 equiv) at 0 °C. The resulting solution was stirred at the room temperature and monitored by TLC. After **3a** was completely depleted, the solvent was removed under reduced pressure and the resulting residue was purified by flash column chromatography (PE:EA = 5:1) to give **5** (49 mg, 86%) as a white solid.

3. Characterization Data



1-Methyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3a** (113 mg, Y = 93%, R_f = 0.35 (PE:EA = 5:1)) was isolated as a white solid; mp 75–76 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.44 (td, *J* = 7.6, 0.8 Hz, 1H), 7.37 (d, *J* = 7.6 Hz, 1H), 7.12 (td, *J* = 7.6, 0.4 Hz, 1H), 6.95 (d, *J* = 7.6 Hz, 1H), 4.11 (q, *J* = 5.6 Hz, 1H), 3.26 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.3, 146.0, 131.5, 125.33 (q, *J* = 5.6 Hz), 123.2, 122.1, (q, *J* = 274.8 Hz), 117.5, 109.2, 60.1 (q, *J* = 42.5 Hz), 59.3 (q, *J* = 1.3 Hz), 26.7; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.81 (d, *J* = 5.1 Hz, 3F); ESI-HRMS m/z calcd for C₁₁H₉F₃NO₂[M+H]⁺ 244.0580, found 244.0577.



1-Benzyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3b** (137 mg, Y = 86%, R_f = 0.45 (PE:EA = 5:1)) was isolated as a white solid; mp 100–101 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.38 (d, *J* = 7.6 Hz, 1H), 7.36–7.25 (m, 6H), 7.07 (td, *J* = 7.6, 0.8 Hz, 1H), 6.85 (d, *J* = 8.0 Hz, 1H), 5.04–4.85 (m, 2H), 4.19 (q, *J* = 6.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 169.6, 145.2, 134.6, 131.4, 128.9, 128.0, 127.3, 125.5 (q, *J* = 5.5 Hz), 123.3, 122.1 (q, *J* = 274.9 Hz), 117.5, 110.2, 60.3 (q, *J* = 41.5 Hz), 59.4 (q, *J* = 1.3 Hz), 44.4; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.72 (s, 3F); ESI-HRMS m/z calcd for C₁₇H₁₃F₃NO₂ [M+H]⁺ 320.0893, found 320.0890.



5-Fluoro-1-methyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3c** (116 mg, Y = 89%, $R_f = 0.29$ (PE:EA = 5:1)) was isolated as a white solid; mp 113–114 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.22-7.08 (m, 2H), 6.90 (dd, J = 8.4, 4.0 Hz, 1H), 4.12 (q, J = 5.6 Hz, 1H), 3.27 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.0, 159.2 (d, J = 240.7 Hz), 142.0 (d, J = 2.1 Hz), 121.9 (d, J = 274.8 Hz), 119.1 (d, J = 8.8 Hz), 118.0 (d, J = 23.3 Hz), 113.6 (dq, J = 26.7, 5.9 Hz), 109.9 (d, J = 8.0 Hz), 60.1 (q, J = 42.5 Hz), 59.2, 26.8; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.82 (d, J = 4.8 Hz, 3F), -119.15 (dd, J = 9.4, 6.0 Hz, 1F); ESI-HRMS m/z calcd for C₁₁H₈F₄NO₂ [M+H]⁺ 262.0486, found 262.0485.



4-Chloro-1-methyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3d** (108 mg, Y = 78%, $R_f = 0.33$ (PE:EA = 5:1)) was isolated as a white solid; mp 166–167 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.36 (t, J = 7.6 Hz, 1H), 7.01 (d, J = 8.4 Hz, 1H), 6.84 (d, J = 7.6 Hz, 1H), 4.81 (q, J = 6.4 Hz, 1H), 3.26 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.6, 146.7, 132.4, 130.6, 124.5, 121.6 (q, J = 276.9 Hz) 116.2, 107.6, 59.8 (q, J = 1.8 Hz), 57.6 (q, J = 44.2 Hz), 27.0; ¹⁹F NMR (282 MHz, CDCl₃) δ -64.05 (d, J = 5.6 Hz, 3F); ESI-HRMS m/z calcd for C₁₁H₈ClF₃NO₂ [M+H]⁺ 278.0190, found 278.0190.

5-Chloro-1-methyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3e** (132 mg, Y = 95%, $R_f = 0.23$ (PE:EA = 5:1)) was isolated as a white solid; mp 131–132 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.42 (dd, J = 8.4, 2.0 Hz, 1H), 7.32 (s, 1H), 6.89 (d, J = 8.4 Hz, 1H), 4.12 (q, J = 5.6 Hz, 1H), 3.26 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 168.8, 144.5, 131.4, 128.8, 125.7 (q, J = 6.0 Hz), 121.9 (q, J = 275.7 Hz), 119.1, 110.2, 60.2 (q, J = 42.5 Hz), 59.0 (q, J = 1.2 Hz), 26.8; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.72 (d, J = 4.8 Hz, 3F); ESI-HRMS m/z calcd for C₁₁H₈ClF₃NO₂ [M+H]⁺ 278.0190, found 278.0186.



4-Bromo-1-methyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3f** (112 mg, Y = 70%, $R_f = 0.32$ (PE:EA = 5:1)) was isolated as a white solid; mp 158–160 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.28 (t, J = 8.0 Hz, 1H), 7.18 (d, J = 8.0 Hz, 1H), 6.88 (d, J = 7.6 Hz, 1H), 4.90 (q, J = 6.0 Hz, 1H), 3.26 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.7, 146.9, 132.5, 127.6, 121.6 (q, J = 276.9 Hz), 118.0, 117.8, 108.2, 60.1 (q, J = 1.6 Hz), 57.3 (q, J = 44.2 Hz), 26.9; ¹⁹F NMR (282 MHz, CDCl₃) δ - 63.81 (d, J = 5.6 Hz, 3F); ESI-HRMS m/z calcd for C₁₁H₈BrF₃NO₂ [M+H]⁺ 321.9685, found 321.9683.

5-Bromo-1-methyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3g** (128 mg, Y = 80%, $R_f = 0.27$ (PE:EA = 5:1)) was isolated as a white solid; mp 122–123 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.57 (dd, J = 8.4, 2.0 Hz, 1H), 7.46 (d, J = 0.4 Hz, 1H), 6.84 (d, J = 8.4 Hz, 1H), 4.12 (q, J = 5.6 Hz, 1H), 3.26 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 168.7, 145.0, 134.4, 128.5 (q, J = 6.1 Hz), 121.9 (q, J = 274.8 Hz), 119.5, 116.0, 110.6, 60.2 (q, J = 42.6 Hz), 58.9 (q, J = 1.3 Hz), 26.8; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.76 (d, J = 4.8 Hz, 3F); ESI-HRMS m/z calcd for C₁₁H₈BrF₃NO₂ [M+H]⁺ 321.9685, found 321.9686.

6-Bromo-1-methyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3h** (145 mg, Y = 90%, $R_f = 0.57$ (PE:EA = 5:1)) was isolated as a white solid; mp 127–128 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.31–7.16 (m, 2H), 7.11 (d, J = 1.6 Hz, 1H), 4.11 (q, J = 6.0 Hz, 1H), 3.26 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 169.2, 147.2, 126.6 (q, J = 5.7 Hz), 126.2, 125.6, 122.0 (d, J = 274.9 Hz), 116.4, 112.9, 60.1 (q, J = 42.5 Hz), 59.0 (q, J = 1.3 Hz), 26.9; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.60 (s, 3F); ESI-HRMS m/z calcd for C₁₁H₈BrF₃NO₂ [M+H]⁺ 321.9685, found 321.9683.



7-Bromo-1-methyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3i** (145 mg, Y = 90%, $R_f = 0.62$ (PE:EA = 5:1)) was isolated as a white solid; mp 119–120 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.54 (dd, J = 8.0, 1.2 Hz, 1H), 7.31 (d, J = 7.6 Hz, 1H), 6.96 (t, J = 7.6 Hz, 1H), 4.12 (q, J = 6.0 Hz, 1H), 3.66 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 170.0, 143.1, 137.1,124.3 (q, J = 5.6 Hz), 124.3, 121.9 (q, J = 275.1 Hz), 120.8, 103.4, 60.9 (q, J = 42.6 Hz), 58.8 (q, J = 1.4 Hz), 30.6; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.10 (s, 3F); ESI-HRMS m/z calcd for C₁₁H₈BrF₃NO₂ [M+H]⁺ 321.9685, found 321.9682.



5-Iodo-1-methyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3j** (162 mg, Y = 88%, $R_f = 0.29$ (PE:EA = 5:1)) was isolated as a white solid; mp 124–125 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.76 (dd, J = 8.4, 1.6 Hz, 1H), 7.61 (s, 1H), 6.73 (d, J = 8.4 Hz, 1H), 4.10 (q, J = 6.0 Hz, 1H), 3.25 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 168.5, 145.6, 140.2, 134.0 (q, J = 6.1 Hz), 121.9 (q, J = 274.9 Hz), 119.7, 111.1, 85.6, 60.2 (q, J = 42.5 Hz), 58.7 (q, J = 1.3 Hz), 26.8; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.85 (d, J = 4.2 Hz, 3F); ESI-HRMS m/z calcd for C₁₁H₈F₃INO₂ [M+H]⁺ 369.9546, found 369.9546.



1,5-Dimethyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3k** (108 mg, Y = 84%, $R_f = 0.41$ (PE:EA = 5:1)) was isolated as a white solid; mp 135–136 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.24 (d, J = 7.8 Hz, 1H), 7.18 (s, 1H), 6.84 (d, J = 8.0 Hz, 1H), 4.10 (q, J = 6.0 Hz, 1H), 3.24 (s, 3H), 2.34 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.3, 143.6, 133.0, 131.7, 126.0 (q, J = 5.4 Hz), 122.2 (q, J = 274.8 Hz), 117.4, 109.0, 60.1 (q, J = 42.4 Hz), 59.4, 26.7, 21.0; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.69 (d, J = 4.8 Hz, 3F); ESI-HRMS m/z calcd for C₁₂H₁₁F₃NO₂ [M+H]⁺ 258.0736, found 258.0733.



1,7-Dimethyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3I** (117 mg, Y = 91%, R_f = 0.45 (PE:EA = 5:1)) was isolated as a white solid; mp 129–130 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.20 (d, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.6 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 4.08 (q, *J* = 6.0 Hz, 1H), 3.54 (s, 3H), 2.59 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 170.3, 143.6, 135.3, 123.1, 123.0 (q, *J* = 5.6 Hz), 122.1 (q, *J* = 275.0 Hz), 121.0, 118.2, 60.7 (q, *J* = 42.4 Hz), 59.1 (q, *J* = 1.4 Hz), 30.3, 19.0; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.37 (s, 3F); ESI-HRMS m/z calcd for C₁₂H₁₁F₃NO₂ [M+H]⁺ 258.0736, found 258.0732.



5-Methoxy-1-methyl-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3m** (70 mg, Y = 51%, $R_f = 0.24$ (PE:EA = 5:1)) was isolated as a white solid; mp 150–152 °C. ¹H NMR (400 MHz,

CDCl₃) δ 7.04–6.92 (m, 2H), 6.85 (d, J = 8.0 Hz, 1H), 4.11 (q, J = 6.0 Hz, 1H), 3.79 (s, 3H), 3.25 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.1, 156.2, 139.3, 122.1 (q, J = 274.9 Hz), 118.7, 116.1, 112.5 (q, J = 5.8 Hz), 109.7, 60.1 (q, J = 42.5 Hz), 59.6 (q, J = 1.1 Hz), 55.8, 26.8; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.80 (d, J = 5.1 Hz, 3F); ESI-HRMS m/z calcd for C₁₂H₁₁F₃NO₃ [M+H]⁺ 274.0686, found 274.0682.



1-Methyl-5-nitro-3'-(trifluoromethyl)spiro[indoline-3,2'-oxiran]-2-one. Compound **3n** (94 mg, Y = 65%, $R_f = 0.18$ (PE:EA = 2:1)) was isolated as a white solid; mp 135–136 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.42 (dd, J = 8.8, 2.4 Hz, 1H), 8.25 (d, J = 1.6 Hz, 1H), 7.11 (d, J = 8.8 Hz, 1H), 4.19 (q, J = 5.6 Hz, 1H), 3.38 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.4, 151.2, 143.8, 128.2, 121.7 (q, J = 274.9 Hz), 121.3 (q, J = 6.2 Hz), 118.5, 109.1, 60.5 (q, J = 42.8 Hz), 58.6 (q, J = 1.3 Hz), 27.3; ¹⁹F NMR (282 MHz, CDCl₃) δ -67.69 (s, 3F); ESI-HRMS m/z calcd for C₁₁H₈F₃N₂O₄ [M+H]⁺ 289.0431, found 289.0428.



Compound **4a** (74 mg, Y = 35%, $R_f = 0.29$ (PE:EA = 5:1)) was isolated as a white solid; mp 119–120 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.54 (d, *J* = 7.6 Hz, 1H), 7.47 (d, J = 7.2 Hz, 2H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.37–7.20 (m, 7H), 7.11 (t, *J* = 7.6 Hz, 1H), 6.79 (d, *J* = 8.0 Hz, 1H), 5.70 (dd, *J* = 10.8, 5.6 Hz, 1H), 5.17–5.00 (m, 2H), 4.83 (d, *J* = 15.6 Hz, 1H), 2.78 (t, *J* = 12.0 Hz 1H), 2.52 (dd, *J* = 12.8, 5.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 174.7, 142.0, 140.1, 135.2, 129.0, 128.8, 128.7, 128.3, 128.0, 127.8, 127.0, 125.8, 125.2 (d, *J* = 1.8 Hz), 123.4 (q, *J* = 279.1 Hz), 122.8, 109.6, 82.8 (q, *J* = 32.2 Hz), 82.7, 56.1, 47.8, 44.2; ¹⁹F NMR (282 MHz, CDCl₃) δ -72.52 (d, *J* = 6.2 Hz, 3F); ESI-HRMS m/z calcd for C₂₅H₂₁F₃NO₂ [M+H]⁺ 424.1519, found 424.1511.



Compound **4b** (103 mg, Y = 43%, $R_f = 0.26$ (PE:EA = 5:1)) was isolated as a white solid; mp 134–135 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.49 (d, J = 7.2 Hz, 2H), 7.41 (t, J = 7.6 Hz, 2H), 7.37–7.23 (m, 7H), 7.18 (t, J = 7.6 Hz, 1H), 7.02 (t, J = 7.6 Hz, 1H), 6.73 (d, J = 8.0 Hz, 1H), 5.55 (dd, J = 8.4, 7.6 Hz, 1H), 5.05 (d, J = 15.6 Hz, 1H), 4.85 (d, J = 15.6 Hz, 1H), 4.75 (q, J = 6.9 Hz, 1H), 2.94 (dd, J = 12.8, 6.8 Hz, 1H), 2.37 (dd, J = 12.8, 9.5 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 177.5, 142.4, 139.4, 135.3, 128.8, 128.8, 128.6, 128.2, 128.1, 127.8, 127.0, 125.8, 125.7 (q, J = 2.7 Hz), 123.2 (q, J = 278.5 Hz), 122.9, 109.3, 82.9 (q, J = 31.7 Hz), 81.1, 55.4, 47.2, 44.0; ¹⁹F NMR (282 MHz, CDCl₃) δ -71.80 (dd, J = 22.0, 6.8 Hz, 3F); ESI-HRMS m/z calcd for C₂₅H₂₁F₃NO₂ [M+H]⁺ 424.1519, found 424.1510.



Compound **5** (49 mg, Y = 86%, $R_f = 0.33$ (PE:EA = 3:1)) was isolated as a white solid; mp 118–119 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.51 (d, *J* = 7.2 Hz, 1H), 7.33 (td, *J* = 7.6, 0.8 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 1H), 6.87 (d, *J* = 7.6 Hz, 1H), 5.38–5.24 (m, 1H), 5.04 (d, *J* = 16.8 Hz, 1H), 4.92 (d, *J* = 10.8 Hz, 1H), 4.53 (q, *J* = 6.8 Hz, 1H), 4.09 (br s, 1H), 3.21 (s, 3H), 2.93 (dd, *J* = 13.6, 6.4 Hz, 1H), 2.76 (dd, *J* = 13.2, 8.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 176.2, 143.1, 130.8, 128.6, 127.4, 125.3, 124.1 (q, *J* = 282.1 Hz), 122.8, 119.8, 108.2, 73.5 (q, *J* = 30.2 Hz), 53.2, 39.4, 26.2; ¹⁹F NMR (282 MHz, CDCl₃) δ -74.40 (d, *J* = 6.5 Hz, 3F); ESI-HRMS m/z calcd for C₁₄H₁₅F₃NO₂ [M+H]⁺ 286.1049, found 286.1042.



Fig. S2. ¹³C NMR of compound 3a (100 MHz, CDCl₃).







Fig. S6. ¹⁹F NMR of compound 3b (282 MHz, CDCl₃).



Fig. S8. ¹³C NMR of compound 3c (100 MHz, CDCl₃).



Fig. S10. ¹H NMR of compound 3d (400 MHz, CDCl₃).



Fig. S12. ¹⁹F NMR of compound 3d (282 MHz, $CDCl_3$).



Fig. S14. ¹³C NMR of compound 3e (100 MHz, CDCl₃).



 $< \frac{-67.707}{-67.724}$



Fig. S18. ¹⁹F NMR of compound 3f (282 MHz, CDCl₃).



Fig. S20. ¹³C NMR of compound 3g (100 MHz, CDCl₃).





 $< \frac{-67.751}{-67.768}$



-50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145

Fig. S24. ¹⁹F NMR of compound 3h (282 MHz, CDCl₃).



Fig. S26. ¹³C NMR of compound 3i (100 MHz, CDCl₃).







Fig. S30. ¹⁹F NMR of compound 3j (282 MHz, CDCl₃).



Fig. S32. ¹³C NMR of compound 3k (100 MHz, CDCl₃).





Fig. S34. ¹H NMR of compound 3l (400 MHz, CDCl₃).





Fig. S36. ¹⁹F NMR of compound 3l (282 MHz, CDCl₃).



Fig. S38. ¹³C NMR of compound 3m (100 MHz, CDCl₃).







50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -1 fl (ppm)

Fig. S42. ¹⁹F NMR of compound 3n (282 MHz, CDCl₃).



Fig. S44. ¹³C NMR of compound 4a (100 MHz, CDCl₃).



 $< \frac{72.546}{72.568}$



Fig. S46. ¹H NMR of compound 4b (400 MHz, CDCl₃).





Fig. S48. ¹⁹F NMR of compound 4b (282 MHz, CDCl₃).



Fig. S50. ¹³C NMR of compound 5 (100 MHz, CDCl₃).



50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -1 f1 (ppm)

Fig. S51. ¹⁹F NMR of compound 5 (282 MHz, CDCl₃).