

Ligand-Enabled Stereoselective β -C(sp³)-H Fluorination: Synthesis of Unnatural Enantiopure *anti*- β -Fluoro- α -Amino Acids

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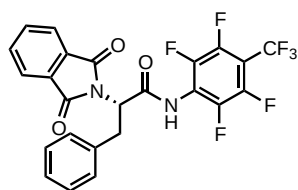
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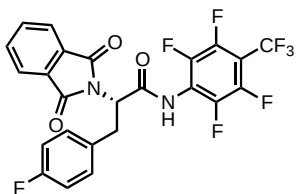
General Information

α -Amino acids and 2,3,5,6-tetrafluoro-4-(trifluoromethyl)aniline were obtained from the commercial sources or synthesized following literature procedures, and used to prepare the corresponding amides. Selectfluor was obtained from Oakwood. Pd(TFA)₂ was obtained from Strem. Ag₂CO₃ was purchased from Alfa-Aesar. Solvents were obtained from Sigma-Aldrich, Alfa-Aesar and Acros and used directly without further purification. Analytical thin layer chromatography was performed on 0.25 mm silica gel 60-F254. Visualization was carried out with UV light and Vogel's permanganate. ¹H NMR was recorded on Bruker AMX-400 instrument (400 MHz) or Bruker DRX-600 instrument (600 MHz). Chemical shifts were quoted in parts per million (ppm) referenced to 0.0 ppm for tetramethylsilane. The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Coupling constants, *J*, were reported in Hertz unit (Hz). ¹³C NMR spectra were recorded on Bruker AMX-400 instrument (100 MHz) or Bruker DRX-600 instrument (150 MHz), and were fully decoupled by broad band proton decoupling. ¹⁹F NMR spectra were recorded on Bruker AMX-400 instrument (376 MHz), and were fully decoupled by broad band proton decoupling. Chemical shifts were reported in ppm referenced to either the center line of a triplet at 77.0 ppm of chloroform-*d* or the center line of a multiplet at 29.84 ppm of acetone-*d*₆. In the ¹³C NMR analysis, peaks that correspond to those of the polyfluoroarylamide auxiliary appeared as nearly invisible, complex sets of multiplets; they were omitted in the following spectroscopic analysis. High-resolution mass spectra (HRMS) were recorded on an Agilent Mass spectrometer using ESI-TOF (electrospray ionization-time of flight).

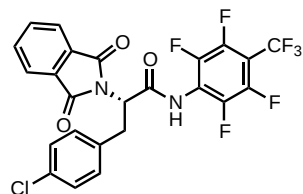
Substrate Structures



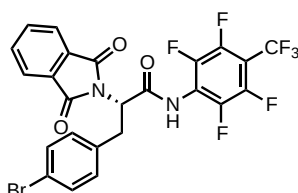
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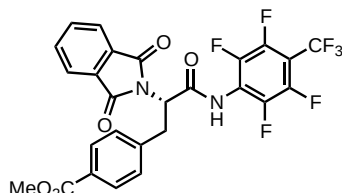
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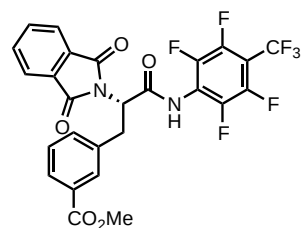
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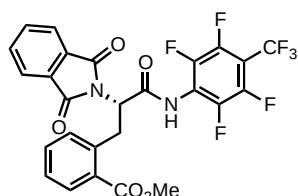
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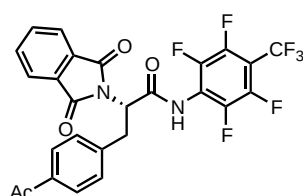
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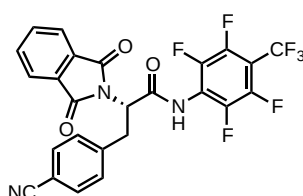
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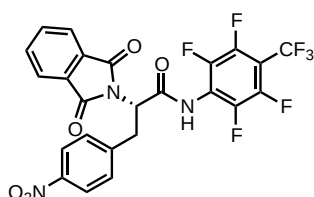
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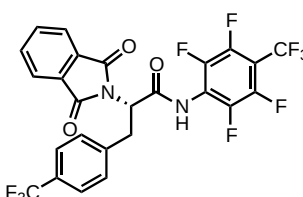
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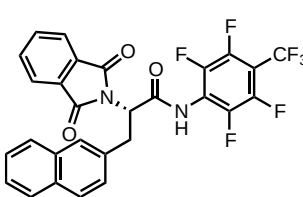
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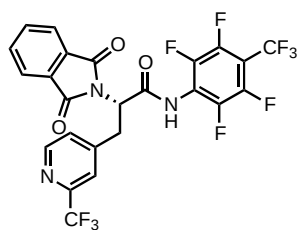
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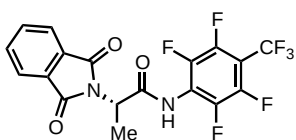
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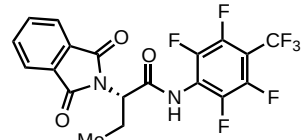
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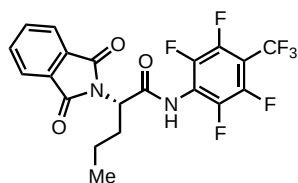
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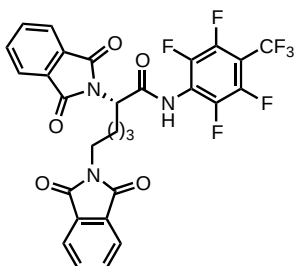
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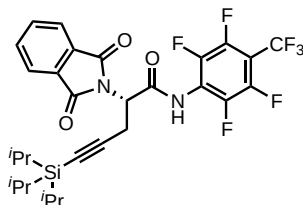
1o



1p



1q

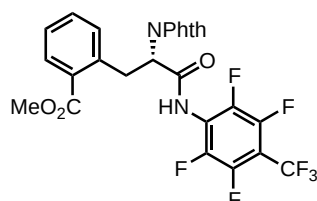


1r

Experimental Section

Substrate Preparation

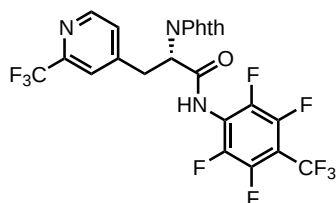
Substrates **1a**, **1n-q** were prepared through traditional synthesis following literature procedure.¹ Substrates **1b-m** were prepared through C-H arylation method following literature procedure.¹ Substrate **1r** was prepared through C-H alkynylation method which is an unpublished result.



(S)-methyl

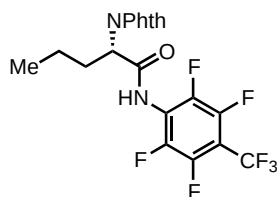
2-(2-(1,3-dioxisoindolin-2-yl)-3-oxo-3-((2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)amino)propyl)benzoate (**1g**)

¹H NMR (400 MHz, CDCl₃) δ 9.23 (br s, 1H), 8.14-8.11 (m, 1H), 7.85-7.83 (m, 2H), 7.74-7.72 (m, 2H), 7.36-7.30 (m, 2H), 7.11-7.09 (m, 1H), 5.38 (dd, *J*₁ = 2.4 Hz, *J*₂ = 10.4 Hz, 1H), 4.17-4.02 (m, 2H), 4.01 (s, 3H). ¹³C NMR (150 MHz, CDCl₃) δ 168.28, 168.08, 166.46, 140.22, 134.28, 133.26, 132.20, 131.55, 127.78, 127.58, 123.65, 56.29, 52.72, 34.28. HRMS (ESI-TOF) Calcd for C₂₆H₁₄F₇N₂O₅ [M-H]⁻: 567.0796, found: 567.0800.



(S)-2-(1,3-dioxisoindolin-2-yl)-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)-3-(2-(trifluoromethyl)pyridin-4-yl)propanamide (**1m**)

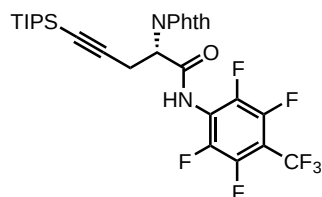
¹H NMR (600 MHz, CDCl₃) δ 8.59 (d, *J* = 5.4 Hz, 1H), 8.21 (br s, 1H), 7.86-7.85 (m, 2H), 7.80-7.79 (m, 2H), 7.51 (s, 1H), 7.38 (d, *J* = 4.8 Hz, 1H), 5.38 (dd, *J*₁ = 6.0 Hz, *J*₂ = 10.2 Hz, 1H), 3.78-3.68 (m, 2H). ¹³C NMR (150 MHz, CDCl₃) δ 167.72, 165.58, 150.42, 148.77 (q, *J* = 34.6 Hz), 146.95, 135.23, 130.69, 126.77, 124.20, 123.01 (q, *J* = 278.2 Hz), 120.98 (q, *J* = 2.7 Hz), 54.72, 34.37. HRMS (ESI-TOF) Calcd for C₂₄H₁₀F₁₀N₃O₃ [M-H]⁻: 578.0568, found: 578.0570.



(S)-2-(1,3-dioxisoindolin-2-yl)-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)pentanamide (**1p**)

¹H NMR (400 MHz, CDCl₃) δ 8.63 (br s, 1H), 7.93-7.91 (m, 2H), 7.82-7.80 (m, 2H), 5.09 (dd,

$J_1 = 6.4$ Hz, $J_2 = 10.0$ Hz, 1H), 2.37-2.19 (m, 2H), 1.44-1.34 (m, 2H), 0.98 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (150 MHz, CDCl_3) δ 168.35, 167.19, 134.84, 131.25, 124.01, 55.59, 31.43, 19.41, 13.34. HRMS (ESI-TOF) Calcd for $\text{C}_{20}\text{H}_{12}\text{F}_7\text{N}_2\text{O}_3$ $[\text{M}-\text{H}]^-$: 461.0742, found: 461.0745.

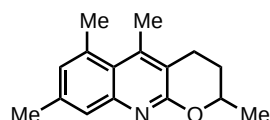


(S)-2-(1,3-dioxoisindolin-2-yl)-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)-5-(triisopropylsilyl)pent-4-ynamide (1r)

^1H NMR (400 MHz, CDCl_3) δ 8.41 (br s, 1H), 7.90-7.88 (m, 2H), 7.79-7.77 (m, 2H), 5.26 (dd, $J_1 = 6.8$ Hz, $J_2 = 10.0$ Hz, 1H), 3.41-3.24 (m, 2H), 0.87-0.85 (m, 21H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.72, 165.78, 134.72, 131.47, 123.95, 101.83, 85.56, 53.22, 21.08, 18.26, 10.95. HRMS (ESI-TOF) Calcd for $\text{C}_{29}\text{H}_{28}\text{F}_7\text{N}_2\text{O}_3\text{Si}$ $[\text{M}-\text{H}]^-$: 613.1763, found: 613.1764.

Ligand Synthesis

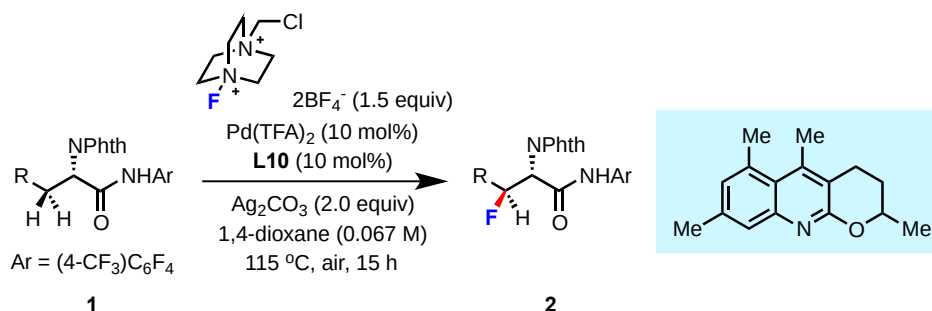
Ligands **4-10** were prepared following literature procedure.²



2,5,6,8-tetramethyl-3,4-dihydro-2H-pyrano[2,3-b]quinoline (L10)

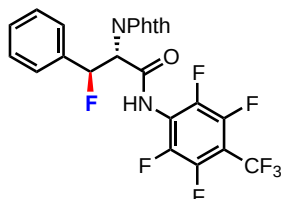
^1H NMR (400 MHz, CDCl_3) δ 7.46 (s, 1H), 6.96 (s, 1H), 4.37-4.29 (m, 1H), 2.96-2.90 (m, 1H), 2.85-2.76 (m, 1H), 2.82 (s, 3H), 2.70 (s, 3H), 2.40 (s, 3H), 2.16-2.10 (m, 1H), 1.84-1.73 (m, 1H), 1.50 (d, $J = 6.0$ Hz, 3H). ^{13}C NMR (150 MHz, CDCl_3) δ 159.22, 147.59, 145.73, 137.88, 134.13, 130.10, 126.25, 123.67, 115.63, 72.74, 29.31, 26.00, 23.98, 21.35, 21.17, 18.83. HRMS (ESI-TOF) Calcd for $\text{C}_{16}\text{H}_{20}\text{NO}$ $[\text{M}+\text{H}]^+$: 242.1539, found: 242.1545.

$\text{C}(\text{sp}^3)\text{-H}$ Fluorination



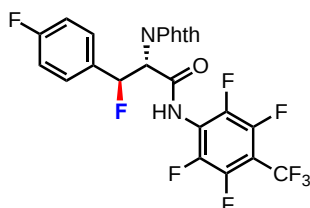
General Procedures for the $\text{C}(\text{sp}^3)\text{-H}$ Fluorination: Substrate **1** (0.10 mmol), Selectfluor (0.15 mmol, 53.2 mg), $\text{Pd}(\text{TFA})_2$ (0.01 mmol, 3.3 mg), **L10** (0.01 mmol, 2.4 mg) and Ag_2CO_3 (0.20 mmol, 55.2 mg) were weighed into a tube (10 mL) with a magnetic stir bar under air. 1,4-Dioxane (1.5 mL) was added, and the tube was sealed with a cap. The reaction mixture was stirred at 115 °C for 15 hours. Upon completion, the reaction mixture was cooled to room temperature and diluted with EtOAc. Then the reaction mixture was filtered through

a short celite tube and purified by preparative thin-layer chromatography using hexane/DCM mixtures as the eluent.



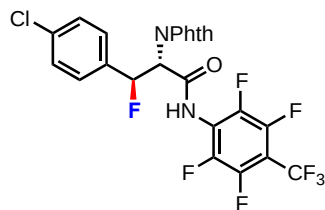
(2R,3S)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-3-phenyl-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)propanamide (2a)

Substrate **1a** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a $> 20:1$ diastereomer ratio. After purification by preparative thin-layer chromatography, **2a** was obtained as a colorless oil (41.9 mg, 79%). ^1H NMR (400 MHz, CDCl_3) δ 8.33 (br d, $J = 11.2$ Hz, 1H), 7.77-7.74 (m, 2H), 7.71-7.67 (m, 2H), 7.43-7.40 (m, 2H), 7.32-7.31 (m, 3H), 6.57 (dd, $J_1 = 10.0$ Hz, $J_2 = 47.6$ Hz, 1H), 5.53 (dd, $J_1 = 9.6$ Hz, $J_2 = 13.2$ Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.01, 163.93 (d, $J = 2.7$ Hz), 134.52, 133.93 (d, $J = 18.9$ Hz), 131.13, 130.34 (d, $J = 2.6$ Hz), 128.90, 127.05 (d, $J = 5.6$ Hz), 123.84, 91.96 (d, $J = 169.2$ Hz), 56.42 (d, $J = 36.2$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.35 (t, $J = 21.8$ Hz, 3F), -140.49-(-140.69) (m, 2F), -142.13-(-142.24) (m, 2F), -164.60 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{24}\text{H}_{11}\text{F}_8\text{N}_2\text{O}_3$ $[\text{M}-\text{H}]^-$: 527.0647, found: 527.0644.



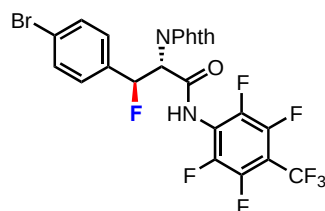
(2R,3S)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-3-(4-fluorophenyl)-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)propanamide (2b)

Substrate **1b** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a $> 20:1$ diastereomer ratio. After purification by preparative thin-layer chromatography, **2b** was obtained as a white solid (38.2 mg, 70%). ^1H NMR (400 MHz, CDCl_3) δ 8.27 (br d, $J = 10.8$ Hz, 1H), 7.79-7.75 (m, 2H), 7.73-7.69 (m, 2H), 7.43-7.40 (m, 2H), 7.01 (t, $J = 8.6$ Hz, 2H), 6.57 (dd, $J_1 = 9.6$ Hz, $J_2 = 47.2$ Hz, 1H), 5.49 (dd, $J_1 = 9.6$ Hz, $J_2 = 13.2$ Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.01, 163.75 (d, $J = 2.6$ Hz), 163.62 (dd, $J_1 = 3.0$ Hz, $J_2 = 249.0$ Hz), 134.66, 131.05, 129.92 (dd, $J_1 = 19.2$ Hz, $J_2 = 3.0$ Hz), 129.20 (dd, $J_1 = 8.7$ Hz, $J_2 = 5.4$ Hz), 123.92, 116.11 (d, $J = 21.8$ Hz), 91.27 (d, $J = 169.2$ Hz), 56.37 (d, $J = 36.3$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.36 (t, $J = 22.2$ Hz, 3F), -109.92 (d, $J = 5.3$ Hz, 1F), -140.42-(-140.62) (m, 2F), -142.17-(-142.29) (m, 2F), -163.03 (d, $J = 4.5$ Hz, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{24}\text{H}_{10}\text{F}_9\text{N}_2\text{O}_3$ $[\text{M}-\text{H}]^-$: 545.0553, found: 545.0554.



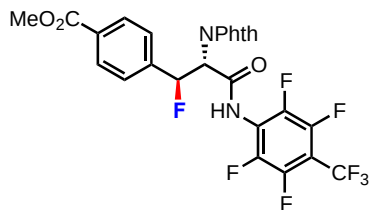
(2R,3S)-3-(4-chlorophenyl)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)propanamide (2c)

Substrate **1c** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. After purification by preparative thin-layer chromatography, **2c** was obtained as a white solid (37.6 mg, 67%). ^1H NMR (400 MHz, CDCl_3) δ 8.25 (br d, J = 10.8 Hz, 1H), 7.79-7.77 (m, 2H), 7.72-7.70 (m, 2H), 7.37 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.8 Hz, 2H), 6.56 (dd, J_1 = 9.2 Hz, J_2 = 47.2 Hz, 1H), 5.48 (dd, J_1 = 10.0 Hz, J_2 = 13.6 Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.03, 163.67 (d, J = 2.7 Hz), 136.41 (d, J = 3.2 Hz), 134.69, 132.49 (d, J = 19.4 Hz), 131.05, 129.26, 128.47 (d, J = 5.4 Hz), 123.97, 91.21 (d, J = 169.5 Hz), 56.30 (d, J = 36.1 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.36 (t, J = 21.4 Hz, 3F), -140.36-(-140.55) (m, 2F), -140.60-(-142.25) (m, 2F), -164.51 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{24}\text{H}_{10}\text{ClF}_8\text{N}_2\text{O}_3$ $[\text{M}-\text{H}]^-$: 561.0258, found: 561.0254.



(2R,3S)-3-(4-bromophenyl)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)propanamide (2d)

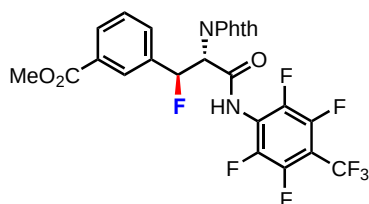
Substrate **1d** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. After purification by preparative thin-layer chromatography, **2d** was obtained as a white solid (41.3 mg, 68%). ^1H NMR (400 MHz, CDCl_3) δ 8.28 (br d, J = 9.2 Hz, 1H), 7.81-7.79 (m, 2H), 7.74-7.72 (m, 2H), 7.48 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 6.8 Hz, 2H), 6.57 (dd, J_1 = 10.4 Hz, J_2 = 46.8 Hz, 1H), 5.50 (dd, J_1 = 9.6 Hz, J_2 = 13.6 Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.04, 163.67 (d, J = 2.7 Hz), 134.70, 133.01 (d, J = 19.4 Hz), 132.21, 131.05, 128.70 (d, J = 5.4 Hz), 124.70 (d, J = 3.6 Hz), 123.98, 91.24 (d, J = 169.8 Hz), 56.25 (d, J = 35.8 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.36 (t, J = 22.2 Hz, 3F), -140.37-(-140.62) (m, 2F), -142.19-(-142.26) (m, 2F), -164.86 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{24}\text{H}_{10}\text{BrF}_8\text{N}_2\text{O}_3$ $[\text{M}-\text{H}]^-$: 604.9753, found: 604.9750.



methyl

4-((1S,2R)-2-(1,3-dioxisoindolin-2-yl)-1-fluoro-3-oxo-3-((2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)amino)propyl)benzoate (2e)

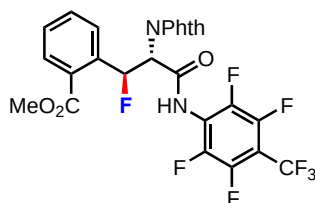
Substrate **1e** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. After purification by preparative thin-layer chromatography, **2e** was obtained as a colorless oil (45.3 mg, 77%). ^1H NMR (400 MHz, CDCl_3) δ 8.31 (br d, J = 10.0 Hz, 1H), 7.98 (d, J = 8.0 Hz, 2H), 7.77-7.75 (m, 2H), 7.71-7.69 (m, 2H), 7.49 (d, J = 8.0 Hz, 2H), 6.62 (dd, J_1 = 9.6 Hz, J_2 = 47.2 Hz, 1H), 5.50 (dd, J_1 = 9.6 Hz, J_2 = 13.6 Hz, 1H), 3.87 (s, 3H). ^{13}C NMR (150 MHz, CDCl_3) δ 166.97, 166.17, 163.60 (d, J = 2.1 Hz), 138.65 (d, J = 18.8 Hz), 134.69, 131.84 (d, J = 2.2 Hz), 130.98, 130.08, 126.98 (d, J = 5.7 Hz), 123.95, 91.13 (d, J = 171.0 Hz), 56.38 (d, J = 35.4 Hz), 52.31. ^{19}F NMR (376 MHz, CDCl_3) δ -56.37 (t, J = 21.8 Hz, 3F), -140.40-(-140.64) (m, 2F), -142.16-(-142.26) (m, 2F), -167.88 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{26}\text{H}_{13}\text{F}_8\text{N}_2\text{O}_5$ $[\text{M}-\text{H}]^-$: 585.0702, found: 585.0703.



methyl

3-((1S,2R)-2-(1,3-dioxisoindolin-2-yl)-1-fluoro-3-oxo-3-((2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)amino)propyl)benzoate (2f)

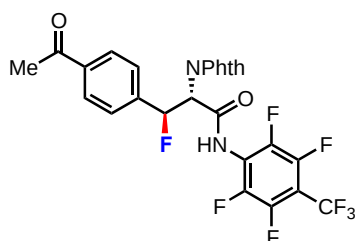
Substrate **1f** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. After purification by preparative thin-layer chromatography, **2f** was obtained as a colorless oil (45.8 mg, 78%). ^1H NMR (400 MHz, CDCl_3) δ 8.34 (br d, J = 10.4 Hz, 1H), 8.10 (s, 1H), 8.00 (d, J = 7.6 Hz, 1H), 7.78-7.76 (m, 2H), 7.70-7.68 (m, 2H), 7.61 (d, J = 7.2 Hz, 1H), 7.41 (t, J = 7.6 Hz, 1H), 6.63 (dd, J_1 = 9.6 Hz, J_2 = 47.2 Hz, 1H), 5.54 (dd, J_1 = 9.6 Hz, J_2 = 12.8 Hz, 1H), 3.88 (s, 3H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.01, 166.02, 163.70 (d, J = 2.7 Hz), 134.60 (d, J = 19.0 Hz), 134.63, 131.38 (d, J = 1.6 Hz), 131.28 (d, J = 5.2 Hz), 131.05, 130.94, 129.08, 128.19 (d, J = 6.0 Hz), 123.93, 91.21 (d, J = 170.6 Hz), 56.45 (d, J = 35.4 Hz), 52.34. ^{19}F NMR (376 MHz, CDCl_3) δ -56.37 (t, J = 21.4 Hz, 3F), -140.45-(-140.65) (m, 2F), -142.14-(-142.23) (m, 2F), -166.36 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{26}\text{H}_{13}\text{F}_8\text{N}_2\text{O}_5$ $[\text{M}-\text{H}]^-$: 585.0702, found: 585.0706.



methyl

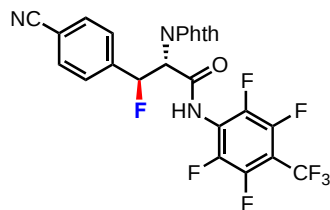
2-((1S,2R)-2-(1,3-dioxoisindolin-2-yl)-1-fluoro-3-oxo-3-((2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)amino)propyl)benzoate (2g)

Substrate **1g** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. After purification by preparative thin-layer chromatography, **2g** was obtained as a colorless oil (44.9 mg, 77%). ^1H NMR (400 MHz, CDCl_3) δ 8.91 (br d, $J = 7.2$ Hz, 1H), 7.87 (d, $J = 8.0$ Hz, 1H), 7.84-7.82 (m, 2H), 7.75-7.73 (m, 2H), 7.68 (d, $J = 7.6$ Hz, 1H), 7.57 (t, $J = 7.6$ Hz, 1H), 7.42 (t, $J = 7.2$ Hz, 1H), 7.16 (dd, $J_1 = 7.2$ Hz, $J_2 = 46.8$ Hz, 1H), 5.74 (dd, $J_1 = 7.6$ Hz, $J_2 = 16.0$ Hz, 1H), 3.90 (s, 3H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.31, 167.10, 163.60, 135.68 (d, $J = 19.8$ Hz), 134.62, 132.56, 131.24, 130.91, 129.66 (d, $J = 1.4$ Hz), 129.03 (d, $J = 3.6$ Hz), 127.56 (d, $J = 9.3$ Hz), 123.89, 88.77 (d, $J = 175.0$ Hz), 58.09 (d, $J = 30.9$ Hz), 52.64. ^{19}F NMR (376 MHz, CDCl_3) δ -56.33 (t, $J = 21.8$ Hz, 3F), -140.76-(-141.00) (m, 2F), -141.97-(-142.08) (m, 2F), -173.97 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{26}\text{H}_{13}\text{F}_8\text{N}_2\text{O}_5$ $[\text{M}-\text{H}]^-$: 585.0702, found: 585.0707.



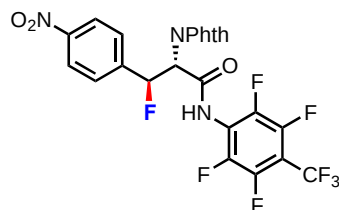
(2R,3S)-3-(4-acetylphenyl)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)propanamide (2h)

Substrate **1h** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. After purification by preparative thin-layer chromatography, **2h** was obtained as a colorless oil (37.9 mg, 66%). ^1H NMR (400 MHz, CDCl_3) δ 8.33 (br d, $J = 10.0$ Hz, 1H), 7.89 (d, $J = 8.0$ Hz, 2H), 7.78-7.76 (m, 2H), 7.71-7.69 (m, 2H), 7.62 (d, $J = 8.0$ Hz, 2H), 7.52 (d, $J = 8.0$ Hz, 2H), 6.63 (dd, $J_1 = 9.6$ Hz, $J_2 = 47.2$ Hz, 1H), 5.51 (dd, $J_1 = 9.6$ Hz, $J_2 = 13.2$ Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 197.34, 167.01, 163.58 (d, $J = 2.5$ Hz), 138.76 (d, $J = 18.6$ Hz), 138.38 (d, $J = 2.5$ Hz), 134.72, 131.00, 128.78, 127.23 (d, $J = 5.7$ Hz), 123.97, 91.08 (d, $J = 171.0$ Hz), 56.38 (d, $J = 35.1$ Hz), 26.63. ^{19}F NMR (376 MHz, CDCl_3) δ -56.36 (t, $J = 21.8$ Hz, 3F), -140.36-(-140.61) (m, 2F), -142.17-(-142.25) (m, 2F), -167.88 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{26}\text{H}_{13}\text{F}_8\text{N}_2\text{O}_4$ $[\text{M}-\text{H}]^-$: 569.0753, found: 569.0757.



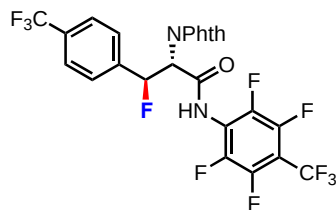
(2R,3S)-3-(4-cyanophenyl)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)propanamide (2i)

Substrate **1i** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. After purification by preparative thin-layer chromatography, **2i** was obtained as a colorless oil (48.0 mg, 87%). ^1H NMR (400 MHz, CDCl_3) δ 8.26 (br d, J = 8.4 Hz, 1H), 7.80-7.78 (m, 2H), 7.74-7.72 (m, 2H), 7.62 (d, J = 8.0 Hz, 2H), 7.54 (d, J = 8.0 Hz, 2H), 6.62 (dd, J_1 = 9.2 Hz, J_2 = 47.2 Hz, 1H), 5.47 (dd, J_1 = 9.6 Hz, J_2 = 14.0 Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 166.98, 163.33 (d, J = 1.6 Hz), 139.02 (d, J = 19.2 Hz), 134.90, 132.66, 130.87, 127.65 (d, J = 6.0 Hz), 124.05, 117.79, 114.16 (d, J = 2.4 Hz), 90.65 (d, J = 172.4 Hz), 56.32 (d, J = 34.6 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.38 (t, J = 21.4 Hz, 3F), -140.32-(-140.52) (m, 2F), -142.19-(-142.28) (m, 2F), -169.60 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{25}\text{H}_{10}\text{F}_8\text{N}_3\text{O}_3$ $[\text{M}-\text{H}]^-$: 552.0600, found: 552.0604.



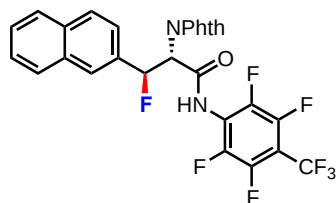
(2R,3S)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-3-(4-nitrophenyl)-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)propanamide (2j)

Substrate **1j** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. After purification by preparative thin-layer chromatography, **2j** was obtained as a white solid (29.8 mg, 52%). ^1H NMR (400 MHz, CDCl_3) δ 8.22 (br d, J = 8.8 Hz, 1H), 8.19 (d, J = 8.8 Hz, 2H), 7.81-7.78 (m, 2H), 7.75-7.72 (m, 2H), 7.63 (d, J = 8.0 Hz, 2H), 6.69 (dd, J_1 = 9.6 Hz, J_2 = 47.2 Hz, 1H), 5.50 (dd, J_1 = 9.6 Hz, J_2 = 14.0 Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.02, 163.19 (d, J = 1.6 Hz), 148.90 (d, J = 2.1 Hz), 140.76 (d, J = 19.2 Hz), 134.94, 130.88, 128.01 (d, J = 6.0 Hz), 124.13, 124.10, 90.48 (d, J = 172.4 Hz), 56.42 (d, J = 34.6 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.37 (t, J = 21.4 Hz, 3F), -140.18-(-140.43) (m, 2F), -142.16-(-142.25) (m, 2F), -169.29 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{24}\text{H}_{10}\text{F}_8\text{N}_3\text{O}_5$ $[\text{M}-\text{H}]^-$: 572.0498, found: 572.0502.



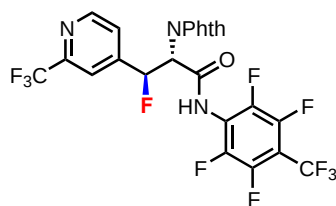
(2R,3S)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)propanamide (2k)

Substrate **1k** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a $> 20:1$ diastereomer ratio. After purification by preparative thin-layer chromatography, **2k** was obtained as a white solid (45.0 mg, 76%). ^1H NMR (400 MHz, CDCl_3) δ 8.25 (br d, $J = 10.4$ Hz, 1H), 7.80-7.78 (m, 2H), 7.73-7.71 (m, 2H), 7.61-7.55 (m, 4H), 6.71 (dd, $J_1 = 9.2$ Hz, $J_2 = 47.2$ Hz, 1H), 5.51 (dd, $J_1 = 10.0$ Hz, $J_2 = 13.6$ Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.09, 163.49 (d, $J = 2.2$ Hz), 137.86 (d, $J = 19.4$ Hz), 134.79, 132.34 (qd, $J_1 = 2.8$ Hz, $J_2 = 32.7$ Hz), 131.01, 127.47 (d, $J = 5.6$ Hz), 125.97 (q, $J = 3.4$ Hz), 124.03, 123.48 (q, $J = 270.8$ Hz), 91.00 (d, $J = 170.7$ Hz), 56.40 (d, $J = 35.1$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.37 (t, $J = 21.8$ Hz, 3F), -63.26 (s, 3F), -140.31-(-140.52) (m, 2F), -142.18-(-142.25) (m, 2F), -167.26 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{25}\text{H}_{10}\text{F}_{11}\text{N}_2\text{O}_3$ $[\text{M-H}]^-$: 595.0521, found: 595.0519.



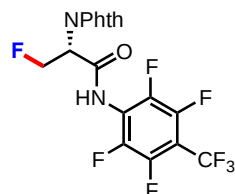
(2R,3S)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-3-(naphthalen-2-yl)-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)propanamide (2l)

Substrate **1l** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a $> 7:1$ diastereomer ratio. After purification by preparative thin-layer chromatography, **2l** was obtained as a major diastereoisomer and as a white solid (30.7 mg, 53%). ^1H NMR (400 MHz, CDCl_3) δ 8.37 (br d, $J = 11.2$ Hz, 1H), 7.89 (s, 1H), 7.84-7.77 (m, 3H), 7.72-7.70 (m, 2H), 7.63-7.61 (m, 2H), 7.55 (d, $J = 8.4$ Hz, 1H), 7.51-7.45 (m, 2H), 6.76 (dd, $J_1 = 10.0$ Hz, $J_2 = 48.0$ Hz, 1H), 5.68 (dd, $J_1 = 10.0$ Hz, $J_2 = 13.2$ Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.10, 164.01 (d, $J = 2.6$ Hz), 134.48, 133.99 (d, $J = 1.8$ Hz), 132.69, 131.14 (d, $J = 18.4$ Hz), 131.12, 129.19, 128.39, 128.14 (d, $J = 7.0$ Hz), 127.74, 127.27, 126.71, 123.85, 123.02 (d, $J = 4.4$ Hz), 92.31 (d, $J = 168.3$ Hz), 56.24 (d, $J = 36.2$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.33 (t, $J = 21.8$ Hz, 3F), -140.44-(-140.72) (m, 2F), -142.11-(-142.20) (m, 2F), -162.59 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{28}\text{H}_{13}\text{F}_8\text{N}_2\text{O}_3$ $[\text{M-H}]^-$: 557.0804, found: 557.0806.



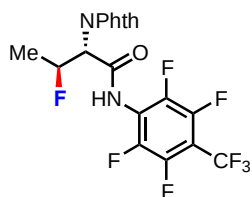
(2R,3S)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)-3-(2-(trifluoromethyl)pyridin-4-yl)propanamide (2m)

Substrate **1m** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. A 2:1 mixture of **2m** to elimination byproduct was obtained after purification by preparative thin-layer chromatography,³ **2m** and elimination byproduct were obtained as a colorless oil (25.9 mg, 43%). ^1H NMR (600 MHz, CDCl_3) δ 8.73 (d, J = 4.8 Hz, 1H), 8.15 (br d, J = 7.2 Hz, 1H), 7.83-7.82 (m, 2H), 7.77-7.76 (m, 2H), 7.72 (s, 1H), 7.55 (d, J = 4.8 Hz, 1H), 6.67 (dd, J_1 = 9.0 Hz, J_2 = 47.4 Hz, 1H), 5.47 (dd, J_1 = 9.0 Hz, J_2 = 14.4 Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.08, 165.92, 150.78, 149.20 (qd, J_1 = 7.6 Hz, J_2 = 35.0 Hz), 144.96 (d, J = 20.1 Hz), 135.13, 130.81, 124.24, 123.92 (d, J = 6.0 Hz), 120.93 (q, J = 273.0 Hz), 118.05 (qd, J_1 = 3.0 Hz, J_2 = 5.6 Hz), 89.61 (d, J = 174.8 Hz), 56.37 (d, J = 33.4 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.38 (t, J = 21.4 Hz, 3F), -68.49 (s, 3F), -140.06-(-140.33) (m, 2F), -142.14-(-142.26) (m, 2F), -175.06 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{24}\text{H}_9\text{F}_{11}\text{N}_3\text{O}_3$ $[\text{M}-\text{H}]^-$: 596.0474, found: 596.0471.



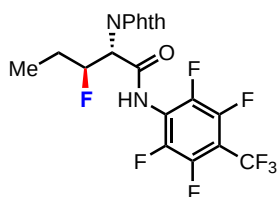
(R)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)propanamide (2n)

Substrate **1n** was fluorinated following this procedure: Substrate **1n** (0.10 mmol, 43.4 mg), Selectfluor (0.125 mmol, 44.3 mg), $\text{Pd}(\text{OAc})_2$ (0.01 mmol, 2.3 mg) and **L5** (0.02 mmol, 5.4 mg) were weighed into a tube (10 mL) with a magnetic stir bar under air. 1,4-Dioxane (1.25 mL) was added, and the tube was sealed with a cap. The reaction mixture was stirred at 115 $^\circ\text{C}$ for 15 hours. Upon completion, the reaction mixture was cooled to room temperature and diluted with EtOAc. Then the reaction mixture was filtered through a short celite tube and purified by preparative thin-layer chromatography using toluene/EtOAc mixtures as the eluent. After purification by preparative thin-layer chromatography, **2n** was obtained as a colorless oil (29.4 mg, 65%). ^1H NMR (400 MHz, CDCl_3) δ 8.03 (br d, J = 3.6 Hz, 1H), 7.94-7.92 (m, 2H), 7.82-7.80 (m, 2H), 5.44-5.23 (m, 2H), 5.05 (ddd, J_1 = 6.8 Hz, J_2 = 9.6 Hz, J_3 = 46.0 Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.47, 164.04 (d, J = 4.8 Hz), 134.92, 131.38, 124.12, 80.06 (d, J = 171.8 Hz), 53.08 (d, J = 24.0 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.38 (t, J = 21.8 Hz, 3F), -140.29-(-140.55) (m, 2F), -142.36-(-142.45) (m, 2F), -222.21 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{18}\text{H}_7\text{F}_8\text{N}_2\text{O}_3$ $[\text{M}-\text{H}]^-$: 451.0334, found: 451.0336.



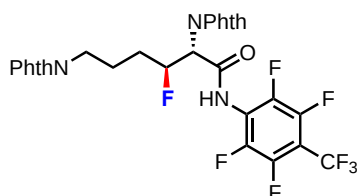
(2R,3S)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)butanamide (2o)

Substrate **1o** was fluorinated following the general fluorination procedure. Analysis of crude reaction mixture by ^1H NMR showed a $> 20:1$ diastereomer ratio. After purification by preparative thin-layer chromatography, **2o** was obtained as a white solid (24.7 mg, 53%). ^1H NMR (600 MHz, CDCl_3) δ 8.20 (br d, $J = 12.0$ Hz, 1H), 7.92-7.91 (m, 2H), 7.80-7.79 (m, 2H), 5.87-5.75 (m, 1H), 5.10 (dd, $J_1 = 9.0$ Hz, $J_2 = 13.8$ Hz, 1H), 1.50 (dd, $J_1 = 6.0$ Hz, $J_2 = 25.8$ Hz, 3H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.46, 164.08 (d, $J = 2.0$ Hz), 134.76, 131.47, 124.06, 88.87 (d, $J = 165.0$ Hz), 57.13 (d, $J = 30.2$ Hz), 18.77 (d, $J = 21.9$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.36 (t, $J = 22.2$ Hz, 3F), -140.52-(-140.81) (m, 2F), -142.23-(-142.34) (m, 2F), -172.07 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{19}\text{H}_9\text{F}_8\text{N}_2\text{O}_3$ $[\text{M}-\text{H}]^-$: 465.0491, found: 465.0490.



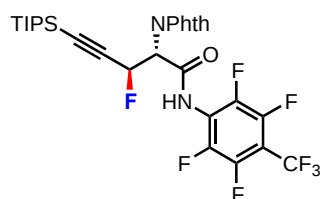
(2R,3S)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)pentanamide (2p)

Substrate **1p** was fluorinated following the general fluorination procedure but using 20 mol% **L10** and adding 1.0 equiv of KHCO_3 . Analysis of crude reaction mixture by ^1H NMR showed a $> 20:1$ diastereomer ratio. After purification by preparative thin-layer chromatography, **2p** was obtained as a white solid (14.4 mg, 30%). ^1H NMR (600 MHz, CDCl_3) δ 8.24 (br d, $J = 12.6$ Hz, 1H), 7.92-7.91 (m, 2H), 7.80-7.79 (m, 2H), 5.64 (dt, $J_1 = 8.4$ Hz, $J_2 = 49.2$ Hz, 1H), 5.16 (dd, $J_1 = 10.2$ Hz, $J_2 = 13.2$ Hz, 1H), 1.89-1.66 (m, 2H), 1.08 (t, $J = 7.8$ Hz, 3H). ^{13}C NMR (150 MHz, CDCl_3) δ 167.49, 164.34 (d, $J = 2.0$ Hz), 134.74, 131.50, 124.04, 92.56 (d, $J = 167.4$ Hz), 55.57 (d, $J = 30.4$ Hz), 25.48 (d, $J = 20.8$ Hz), 8.06 (d, $J = 4.4$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.36 (t, $J = 21.8$ Hz, 3F), -140.57-(-140.82) (m, 2F), -142.24-(-142.34) (m, 2F), -183.02 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{20}\text{H}_{11}\text{F}_8\text{N}_2\text{O}_3$ $[\text{M}-\text{H}]^-$: 479.0647, found: 479.0644.



(2R,3S)-2,6-bis(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)hexanamide (2q)

Substrate **1q** was fluorinated following the general fluorination procedure but using 20 mol% **L10** and adding 1.0 equiv of KHCO_3 . Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. After purification by preparative thin-layer chromatography, **2q** was obtained as a white solid (16.0 mg, 25%). ^1H NMR (600 MHz, CDCl_3) δ 8.27 (br d, J = 11.4 Hz, 1H), 7.89-7.88 (m, 2H), 7.80-7.77 (m, 4H), 7.71-7.69 (m, 2H), 5.76-5.64 (m, 1H), 5.15 (dd, J_1 = 9.0 Hz, J_2 = 13.2 Hz, 1H), 3.70 (t, J = 7.2 Hz, 2H), 2.01-1.76 (m, 4H). ^{13}C NMR (150 MHz, CDCl_3) δ 168.22, 167.46, 164.01, 134.73, 134.00, 131.90, 131.45, 124.07, 123.25, 91.07 (d, J = 168.6 Hz), 55.89 (d, J = 29.7 Hz), 37.12, 29.59 (d, J = 20.6 Hz), 23.28 (d, J = 3.2 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -56.36 (t, J = 21.8 Hz, 3F), -140.57-(-140.81) (m, 2F), -142.15-(-142.22) (m, 2F), -181.85 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{29}\text{H}_{16}\text{F}_8\text{N}_3\text{O}_5$ $[\text{M}-\text{H}]^-$: 638.0968, found: 638.0967.



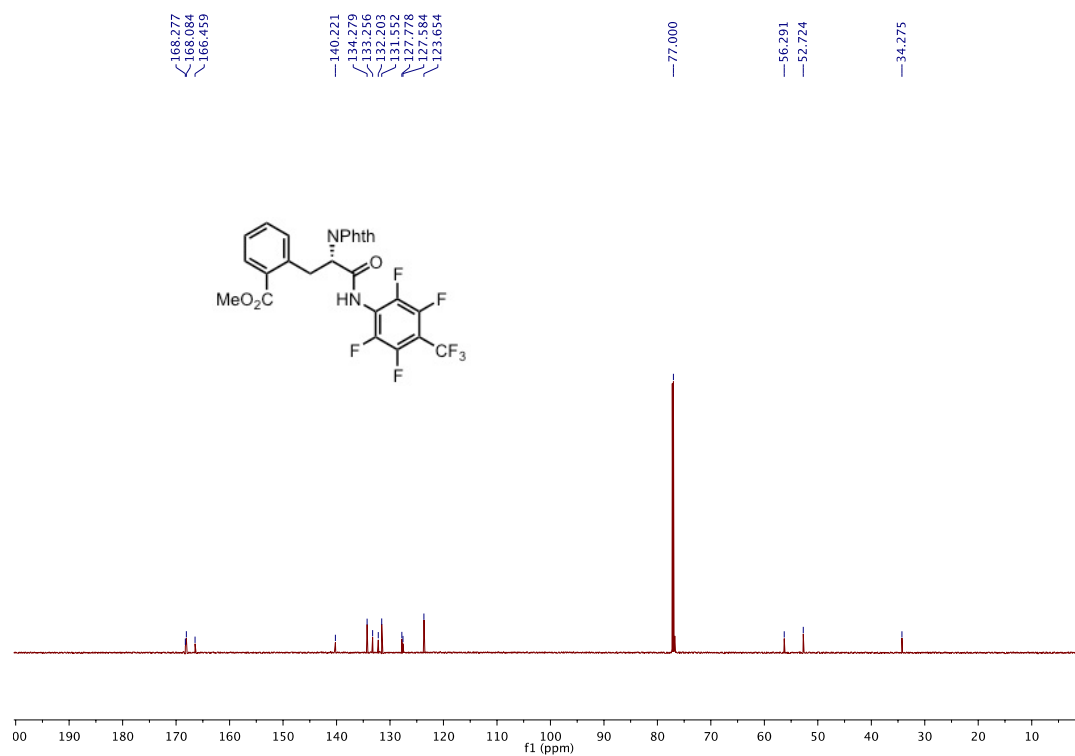
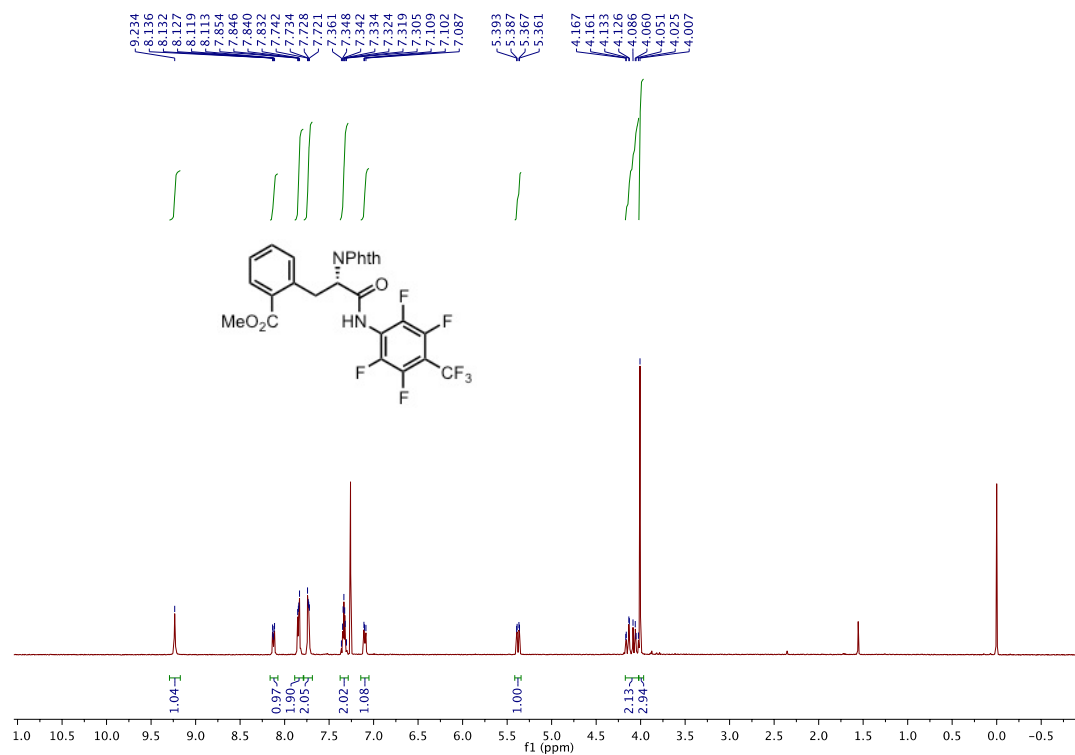
(2R,3S)-2-(1,3-dioxoisindolin-2-yl)-3-fluoro-N-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)-5-(triisopropylsilyl)pent-4-ynamide (2r)

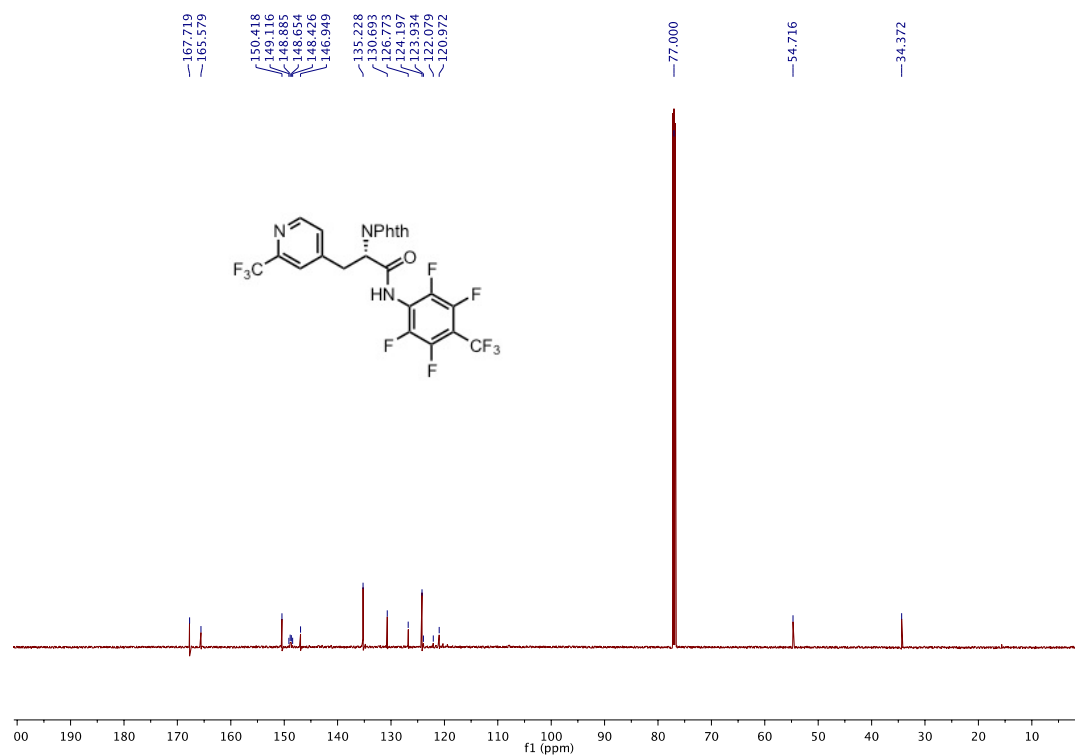
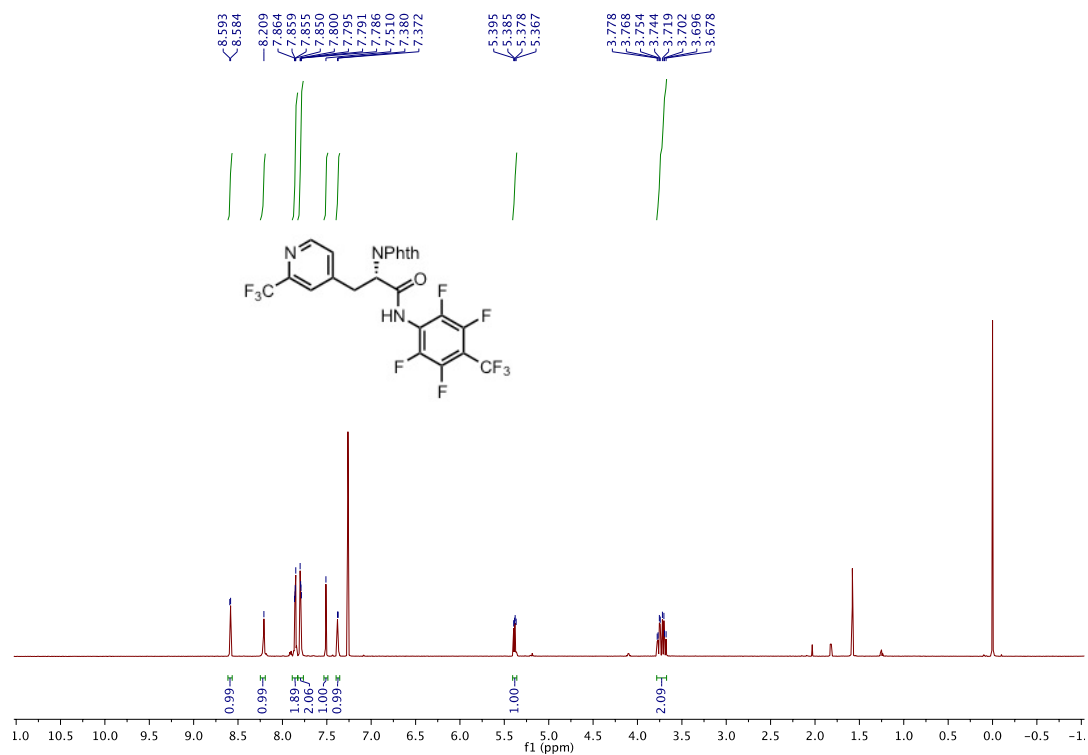
Substrate **1r** was fluorinated following this procedure: Substrate **1r** (0.10 mmol, 61.6 mg), Selectfluor (0.125 mmol, 44.3 mg), $\text{Pd}(\text{OAc})_2$ (0.01 mmol, 2.3 mg) and **L5** (0.02 mmol, 5.4 mg) were weighed into a tube (10 mL) with a magnetic stir bar under air. 1,4-Dioxane (1.25 mL) was added, and the tube was sealed with a cap. The reaction mixture was stirred at 115 °C for 15 hours. Upon completion, the reaction mixture was cooled to room temperature and diluted with EtOAc. Then the reaction mixture was filtered through a short celite tube and purified by preparative thin-layer chromatography using toluene/EtOAc mixtures as the eluent. Analysis of crude reaction mixture by ^1H NMR showed a > 20:1 diastereomer ratio. After purification by preparative thin-layer chromatography, **2r** was obtained as a colorless oil (31.7 mg, 50%). ^1H NMR (400 MHz, CDCl_3) δ 8.00 (br d, J = 8.4 Hz, 1H), 7.91-7.89 (m, 2H), 7.80-7.76 (m, 2H), 6.30 (dd, J_1 = 9.2 Hz, J_2 = 49.6 Hz, 1H), 5.31 (dd, J_1 = 9.6 Hz, J_2 = 13.2 Hz, 1H), 0.86 (s, 21H). ^{13}C NMR (150 MHz, CDCl_3) δ 166.88, 162.90 (d, J = 2.6 Hz), 134.69, 131.57, 123.98, 97.53 (d, J = 22.5 Hz), 95.37 (d, J = 8.8 Hz), 80.43 (d, J = 170.0 Hz), 55.70 (d, J = 32.2 Hz), 18.15, 10.71. ^{19}F NMR (376 MHz, CDCl_3) δ -56.38 (t, J = 21.4 Hz, 3F), -140.39-(-140.59) (m, 2F), -142.18-(-142.28) (m, 2F), -170.11 (s, 1F). HRMS (ESI-TOF) Calcd for $\text{C}_{29}\text{H}_{27}\text{F}_8\text{N}_2\text{O}_3\text{Si}$ $[\text{M}-\text{H}]^-$: 631.0742, found: 631.0745.

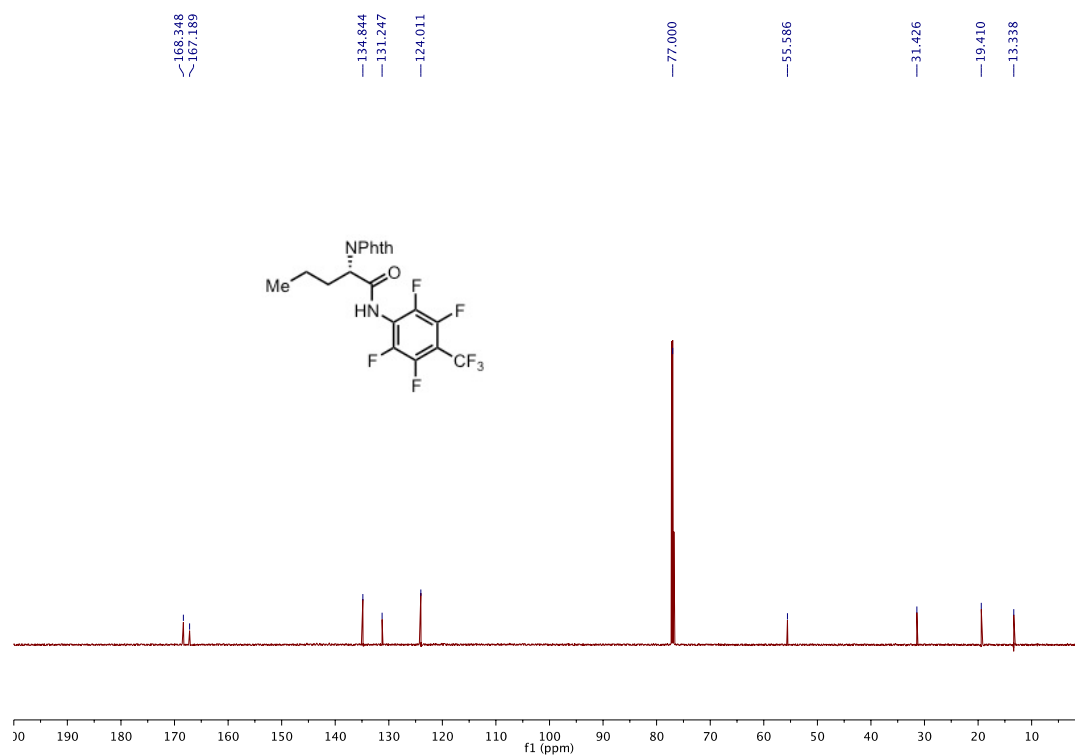
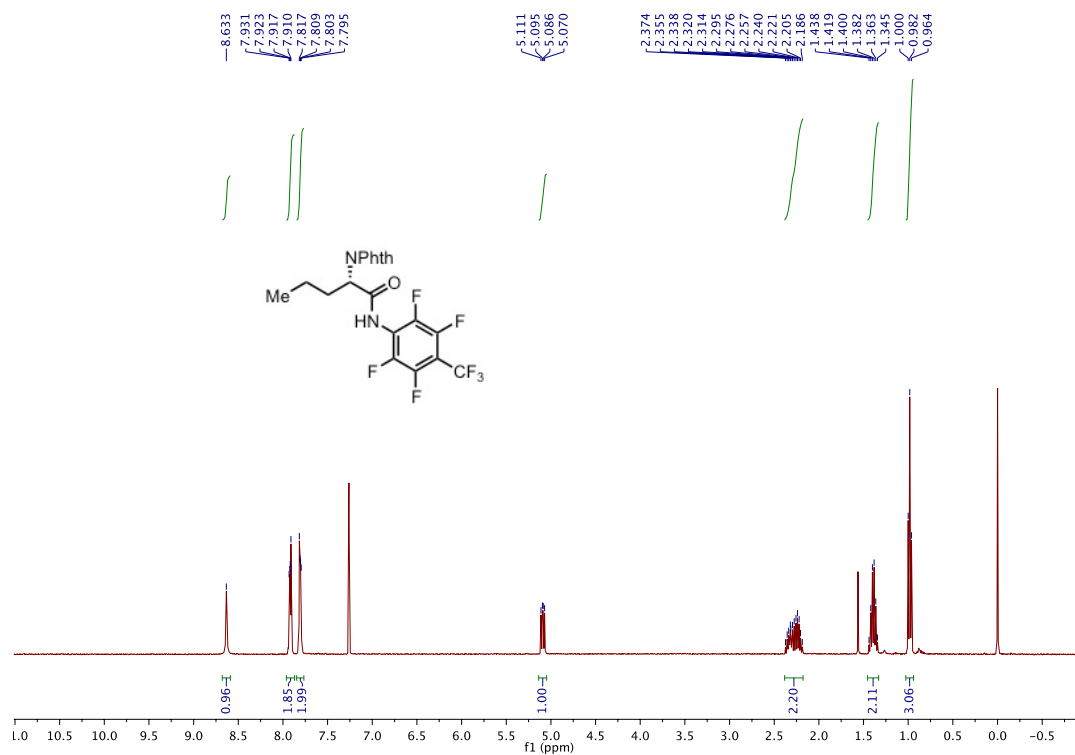
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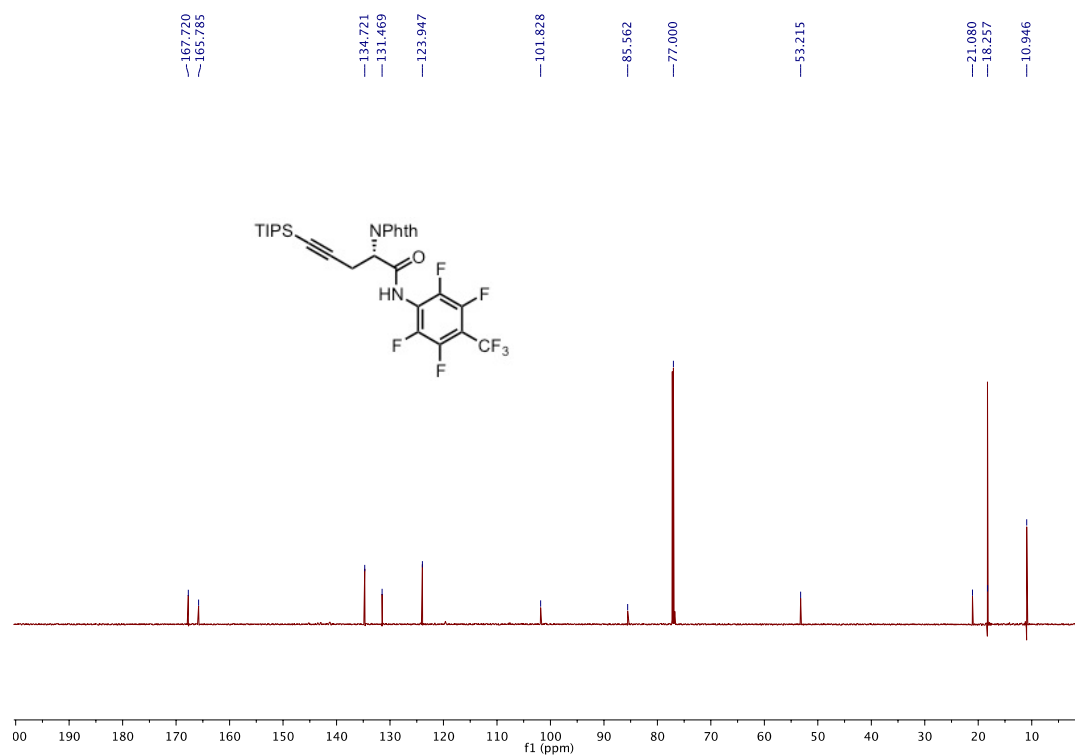
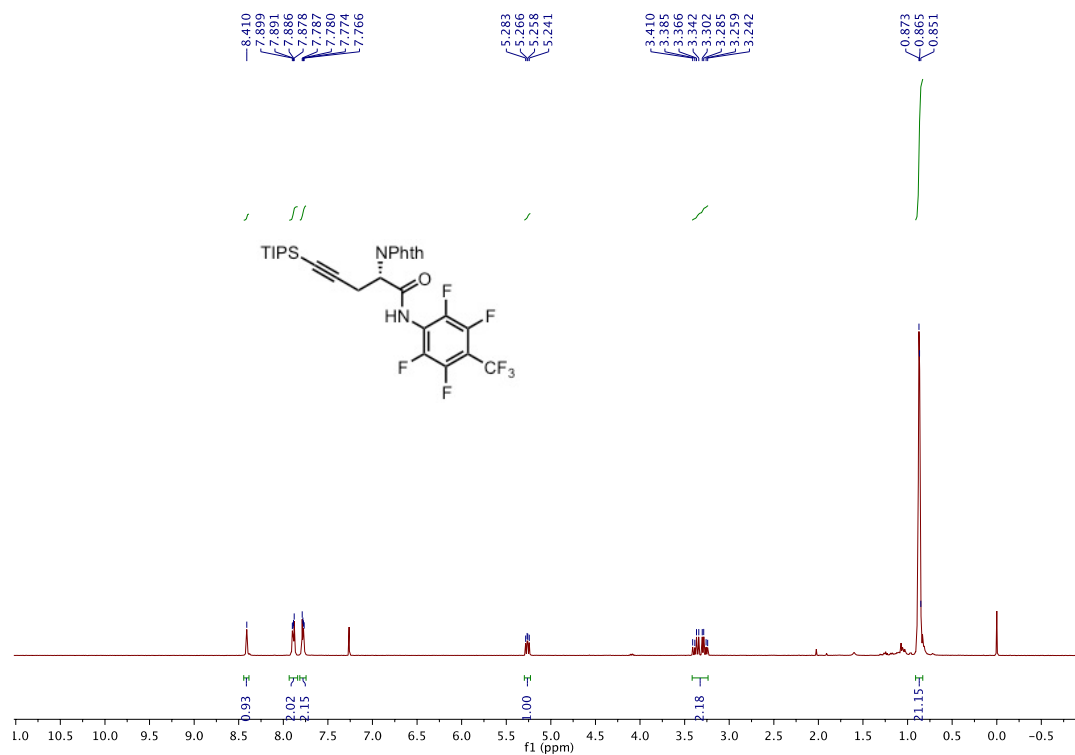
- (1) He, J.; Li, S.-H.; Deng, Y.-Q.; Fu, H.-Y.; Laforteza, B. N.; Spangler, J. E.; Homs, A.; Yu, J.-Q. *Science* **2014**, *343*, 1216.
- (2) Li, S.-H.; Chen, G.; Feng, C.-G.; Gong, W.; Yu, J.-Q. *J. Am. Chem. Soc.* **2014**, *136*, 5267.
- (3) Xia, J.-B.; Zhu, C.; Chen, C. *J. Am. Chem. Soc.* **2013**, *135*, 9342.

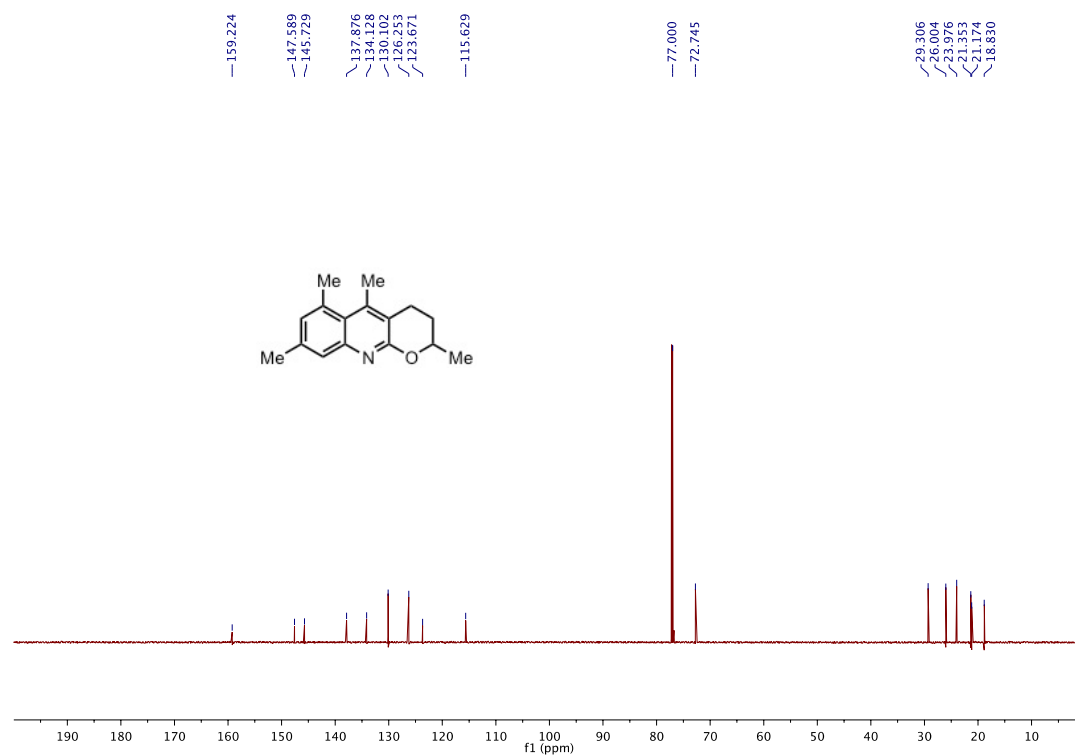
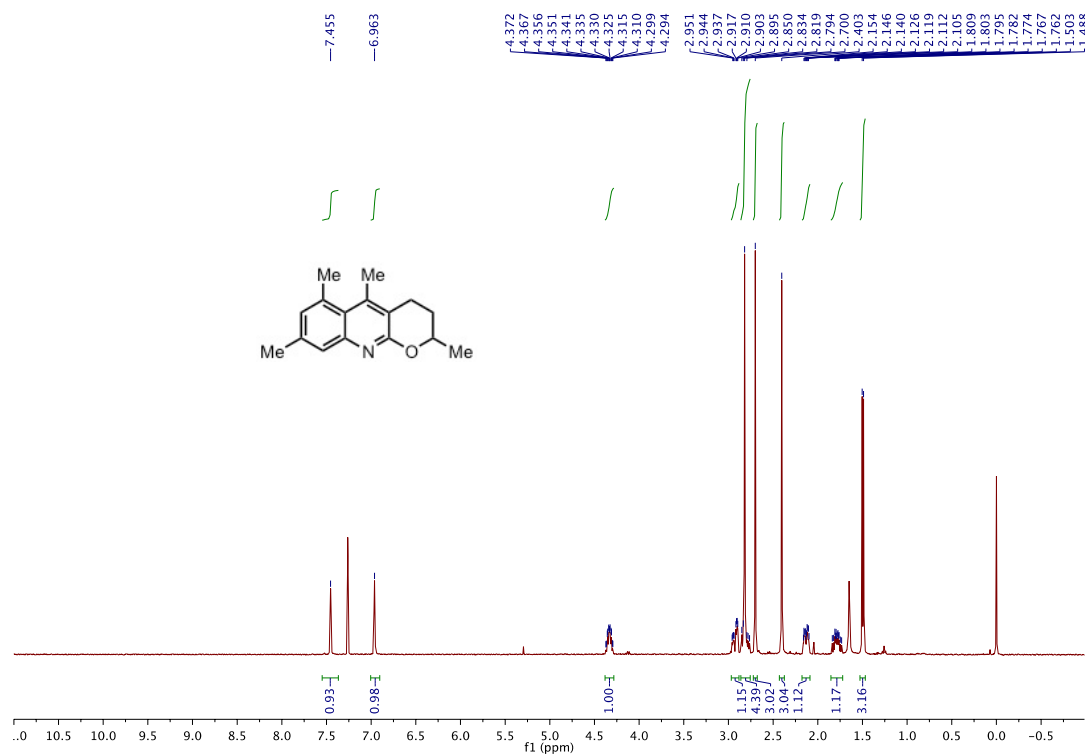
^1H and ^{13}C NMR Spectra

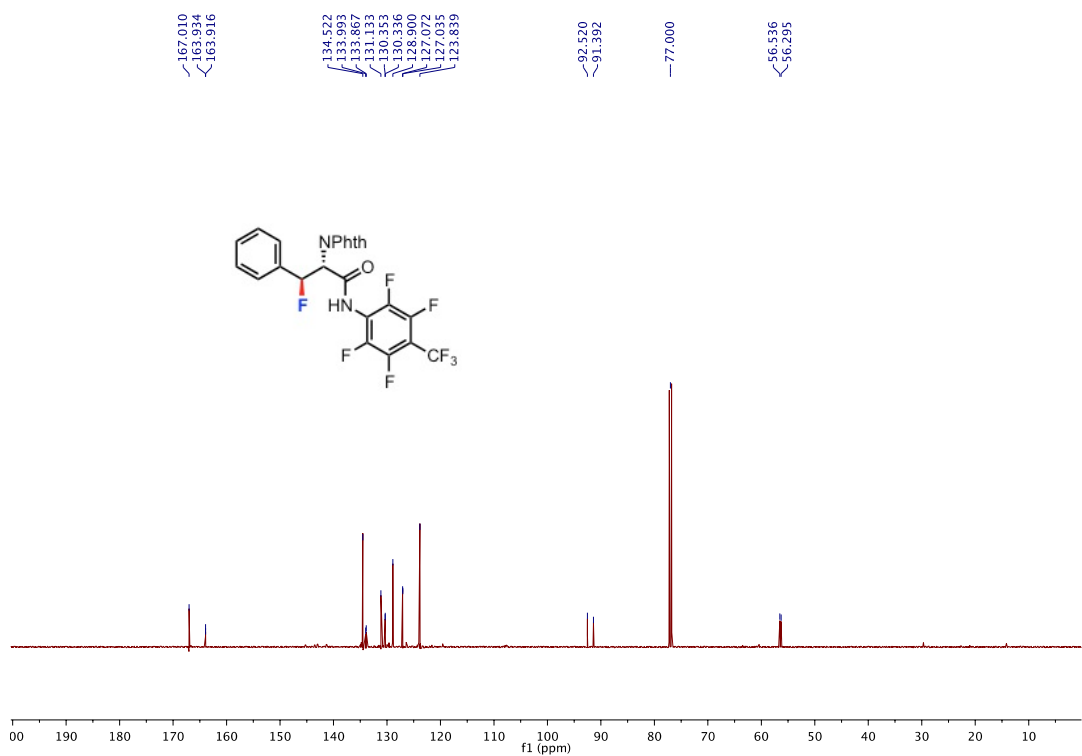
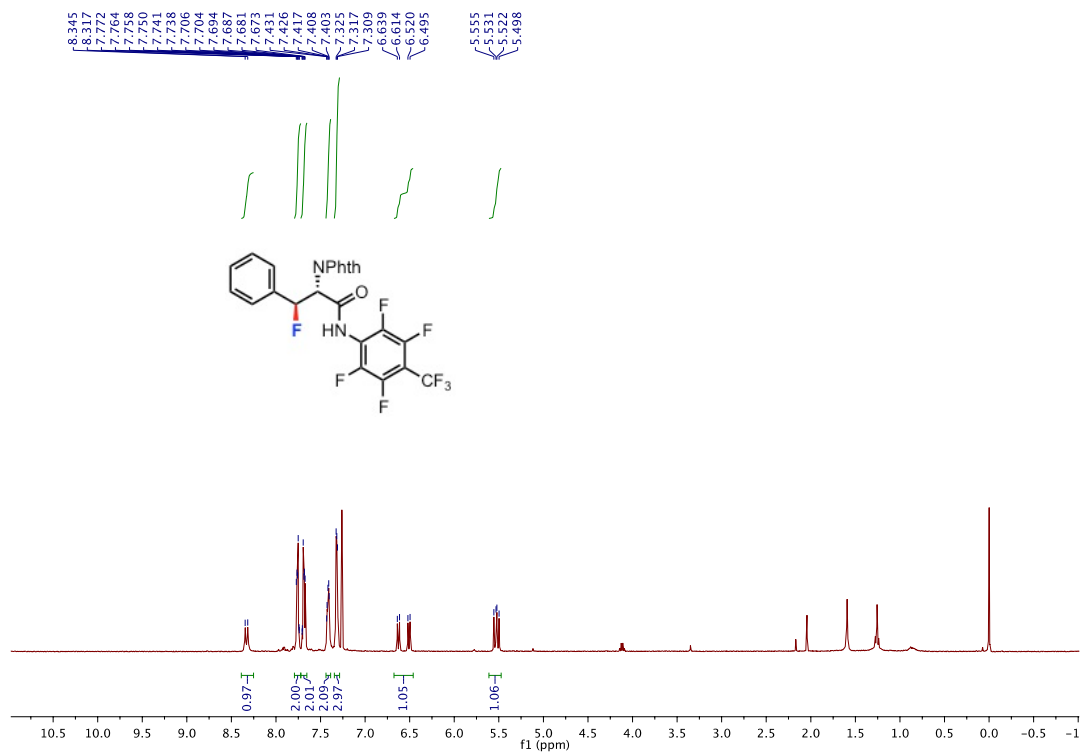


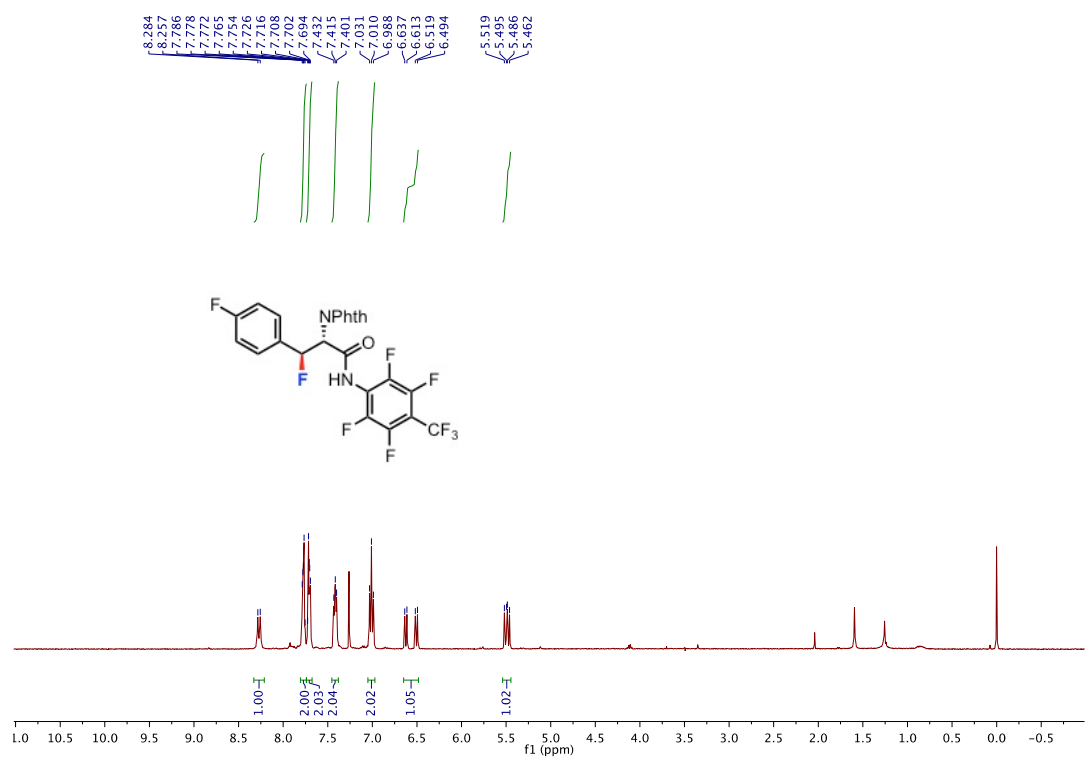
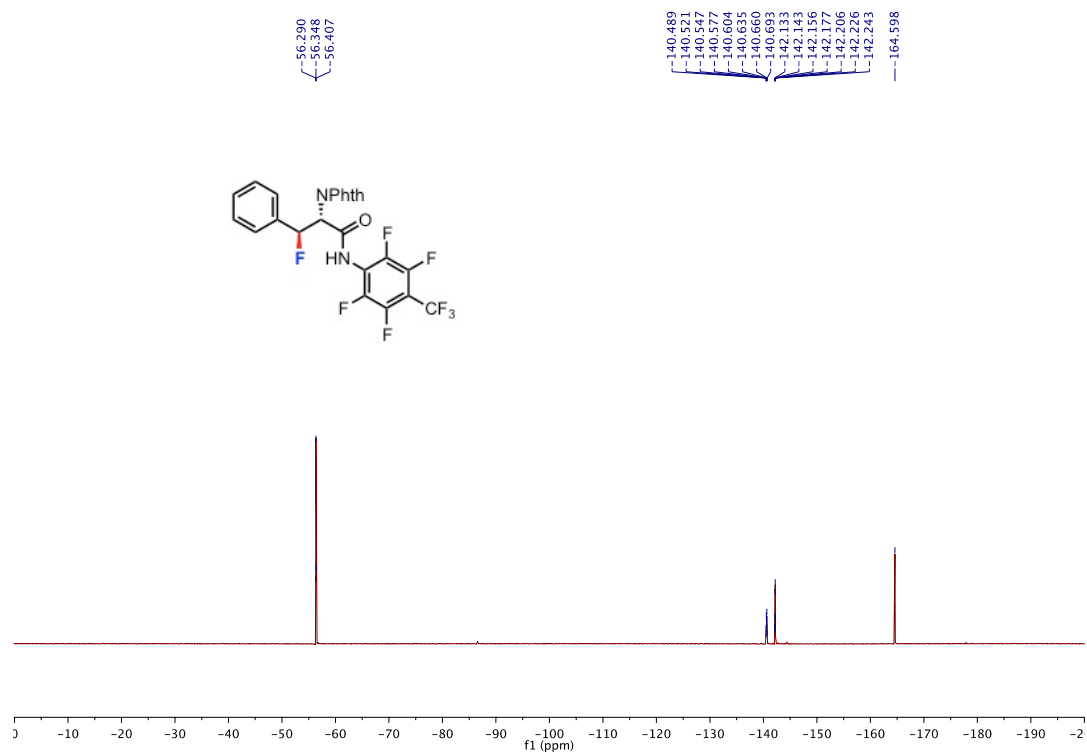


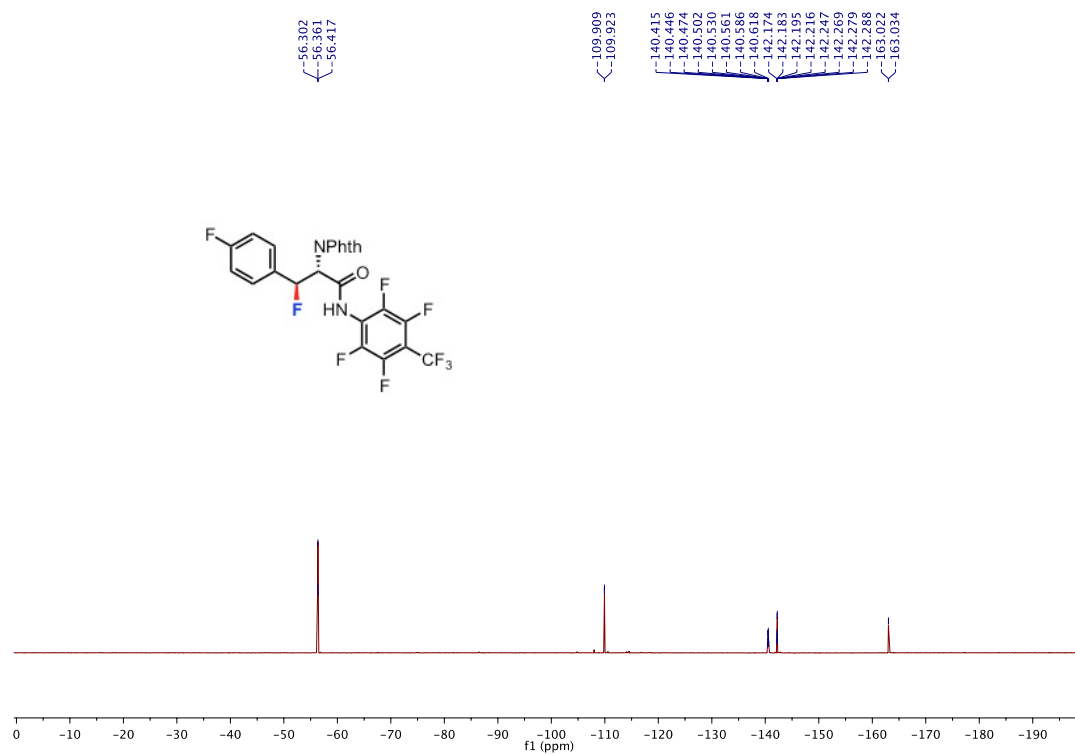
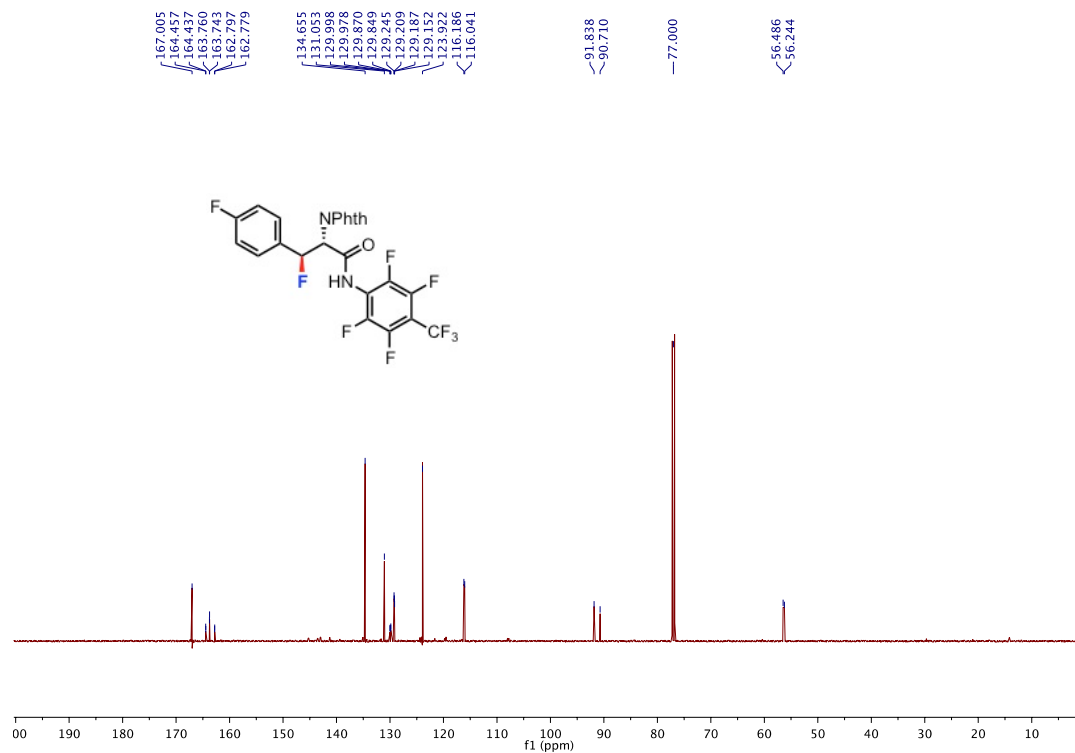


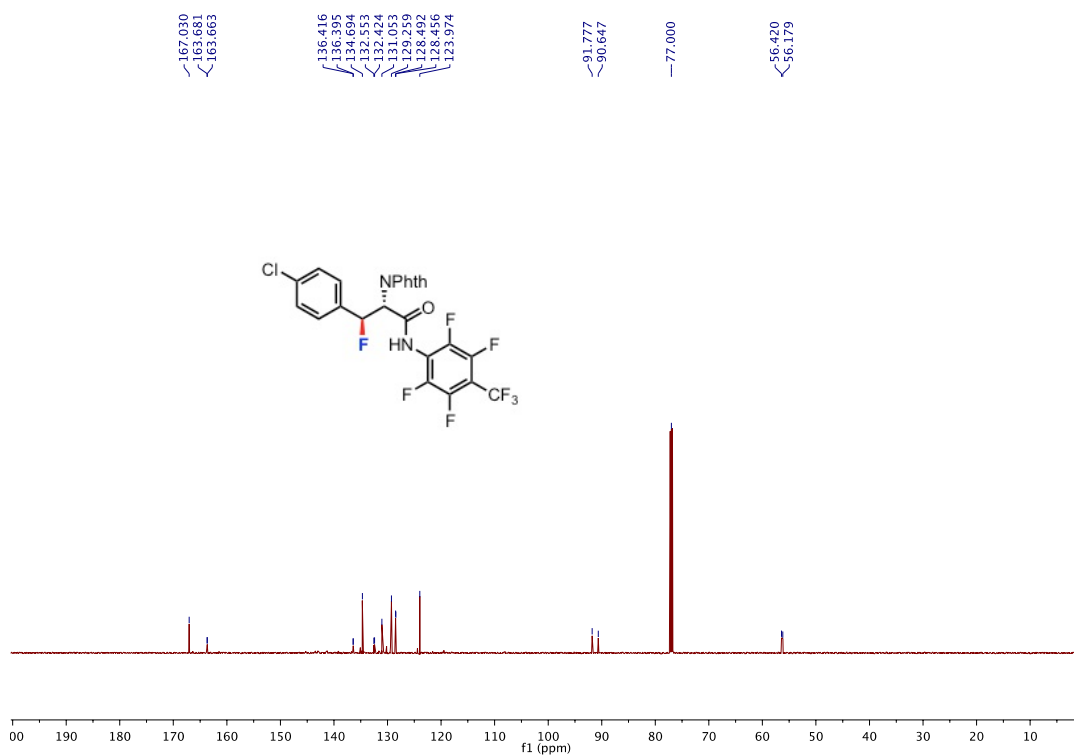
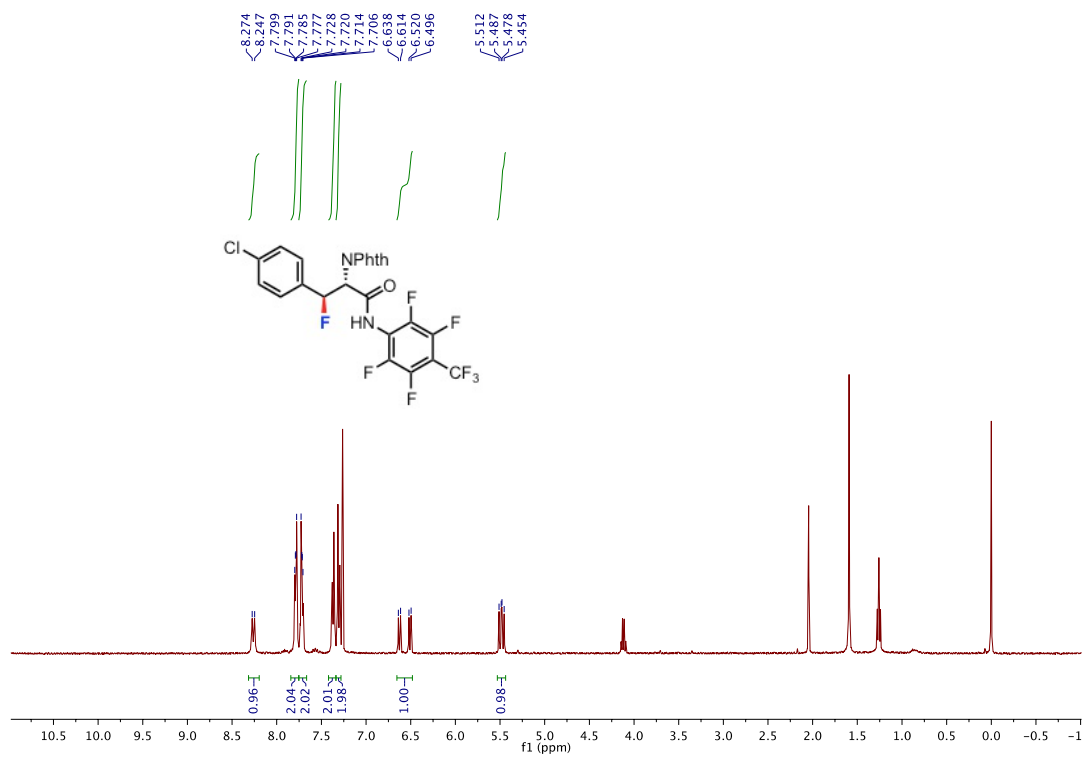


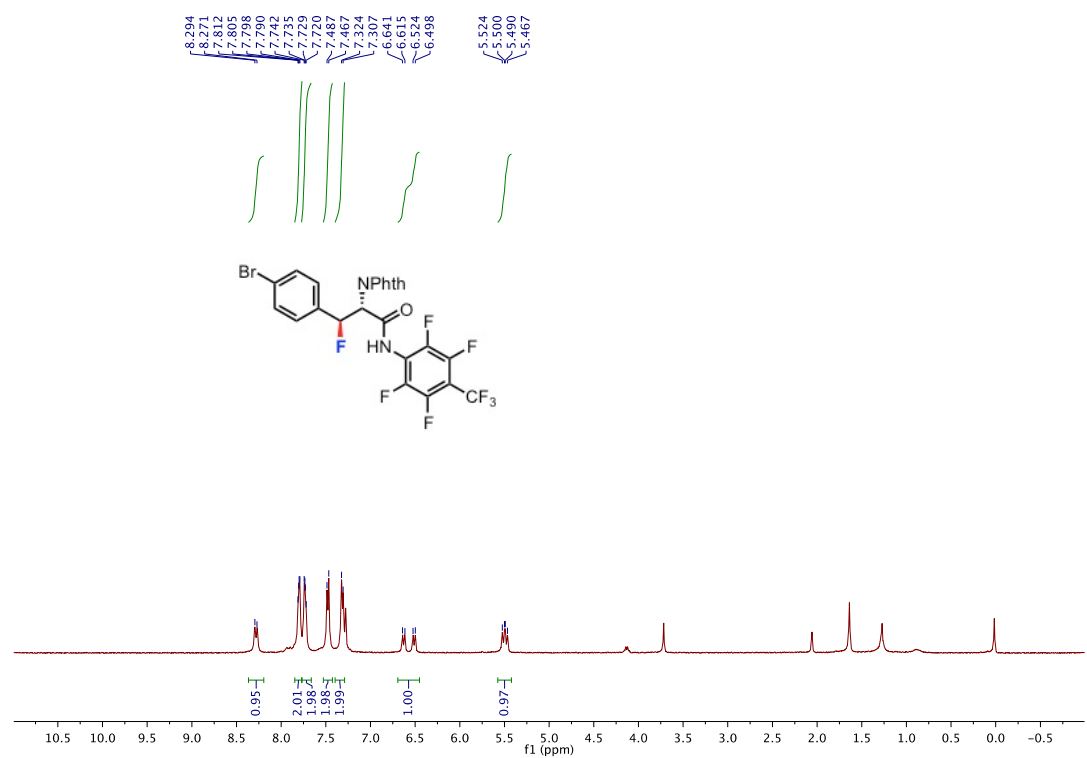
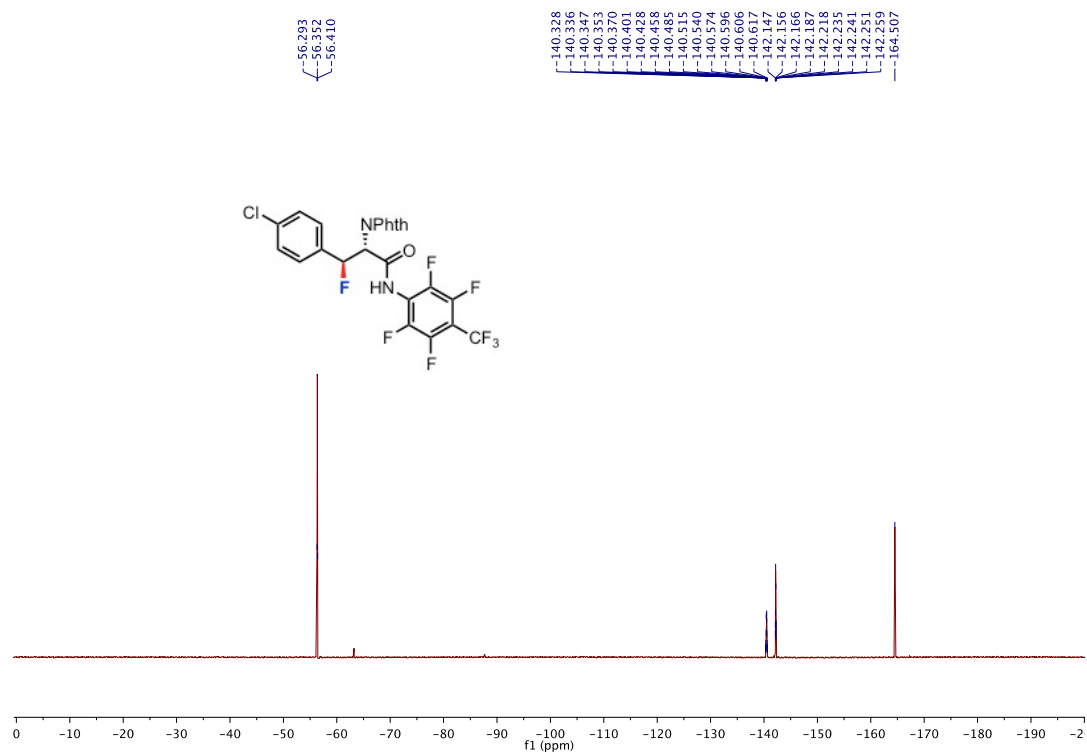


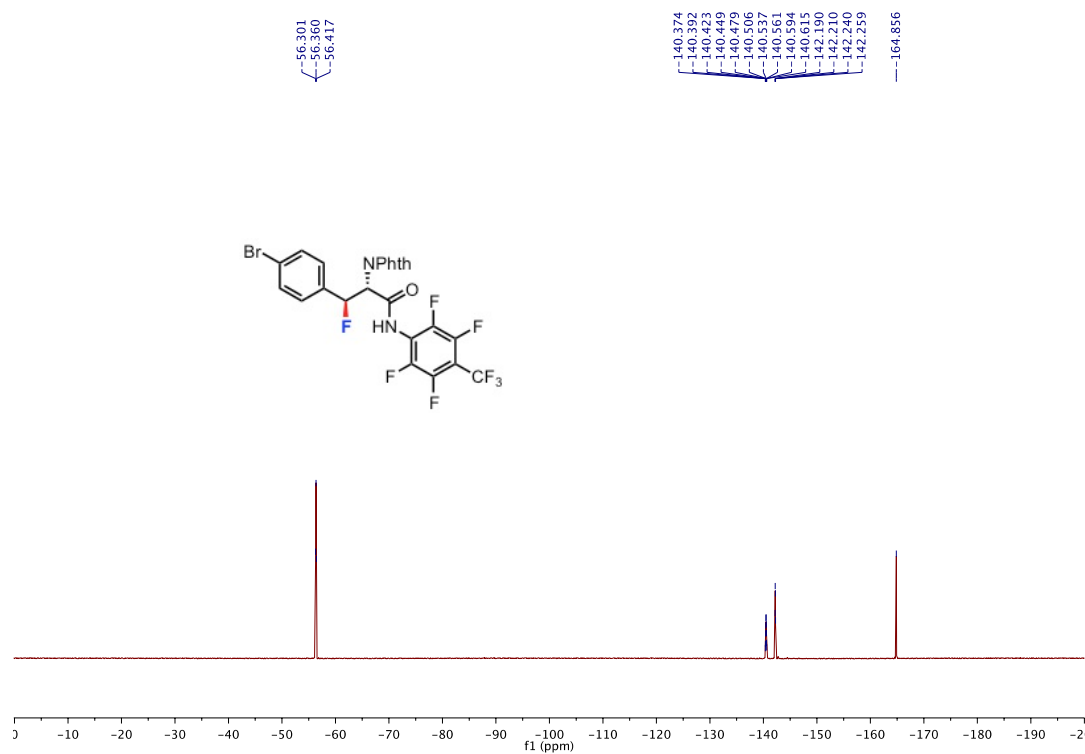
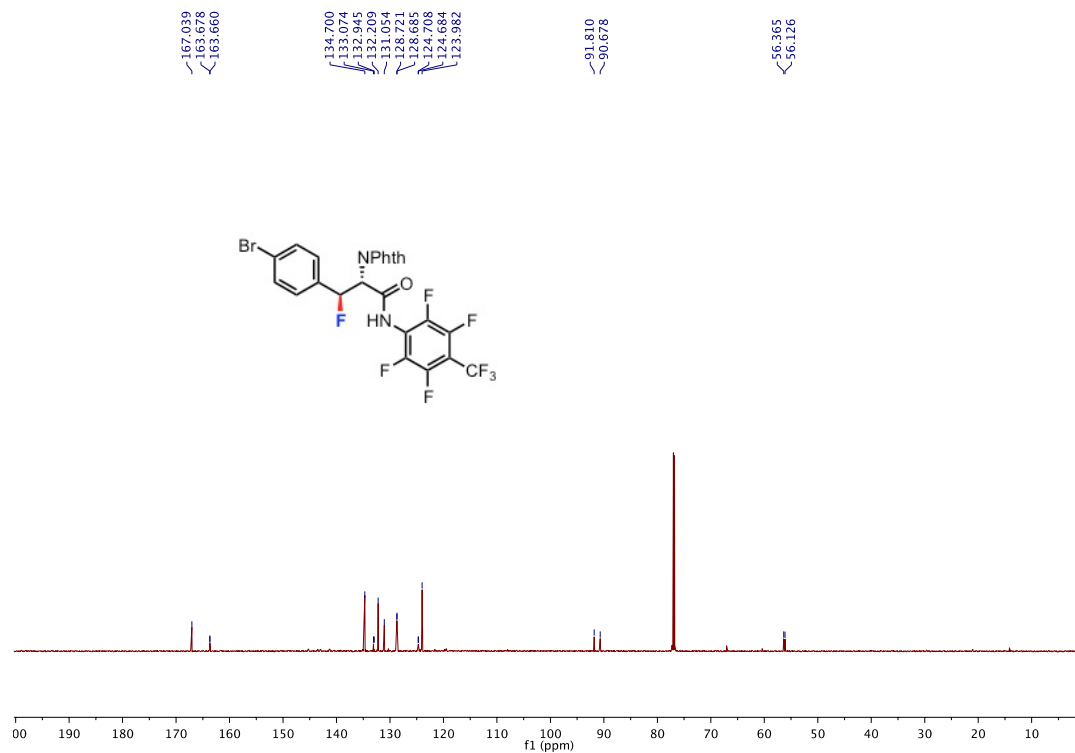


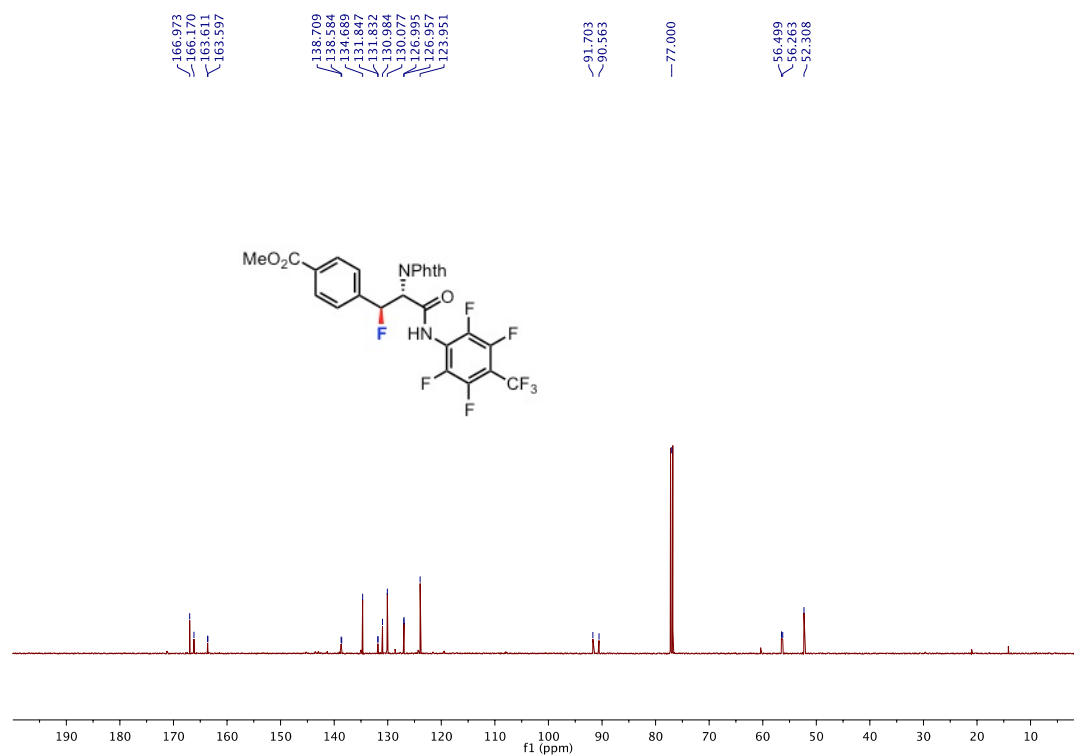
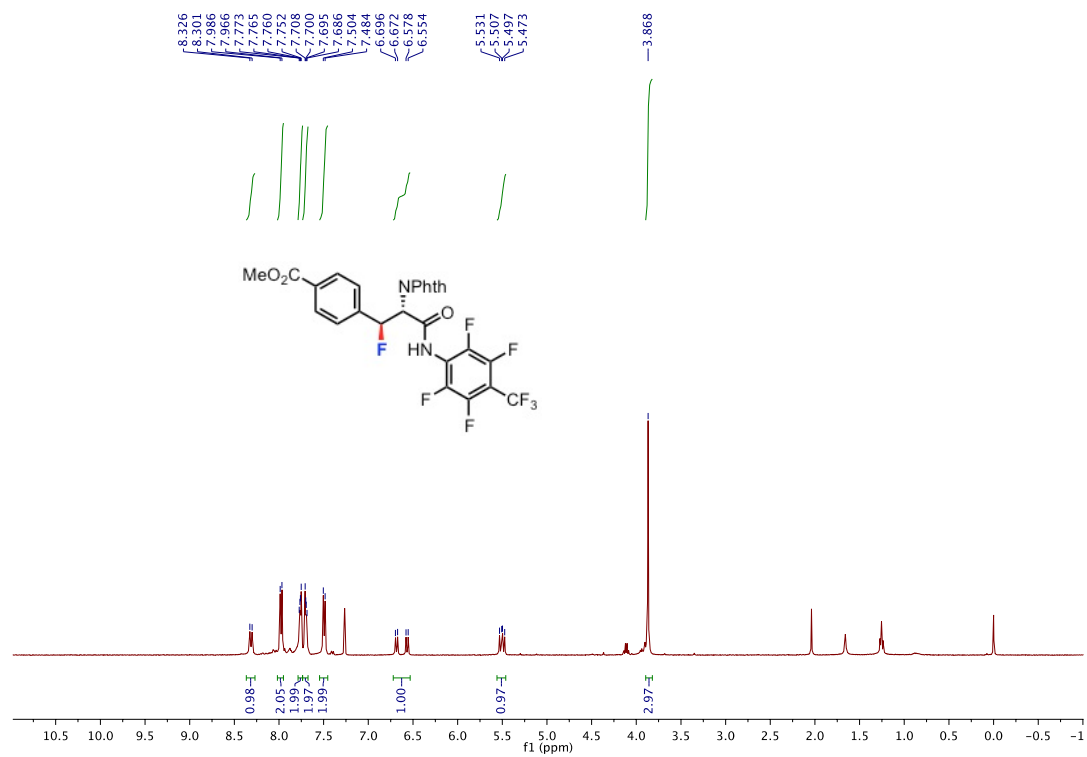


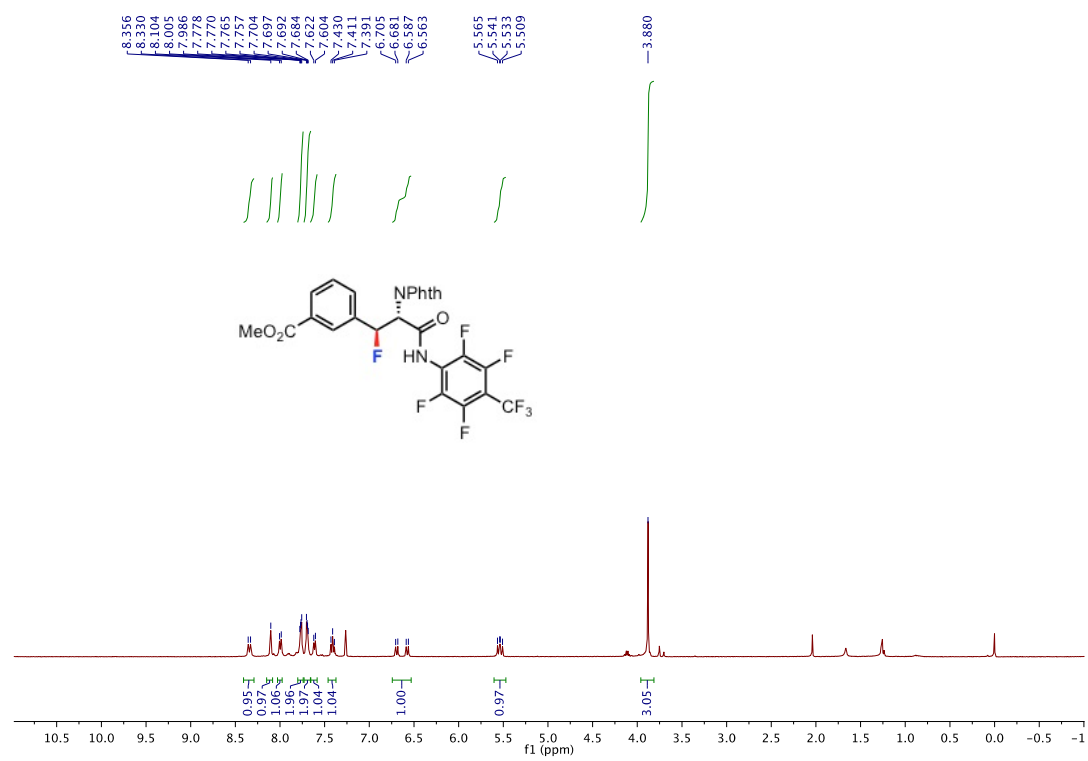
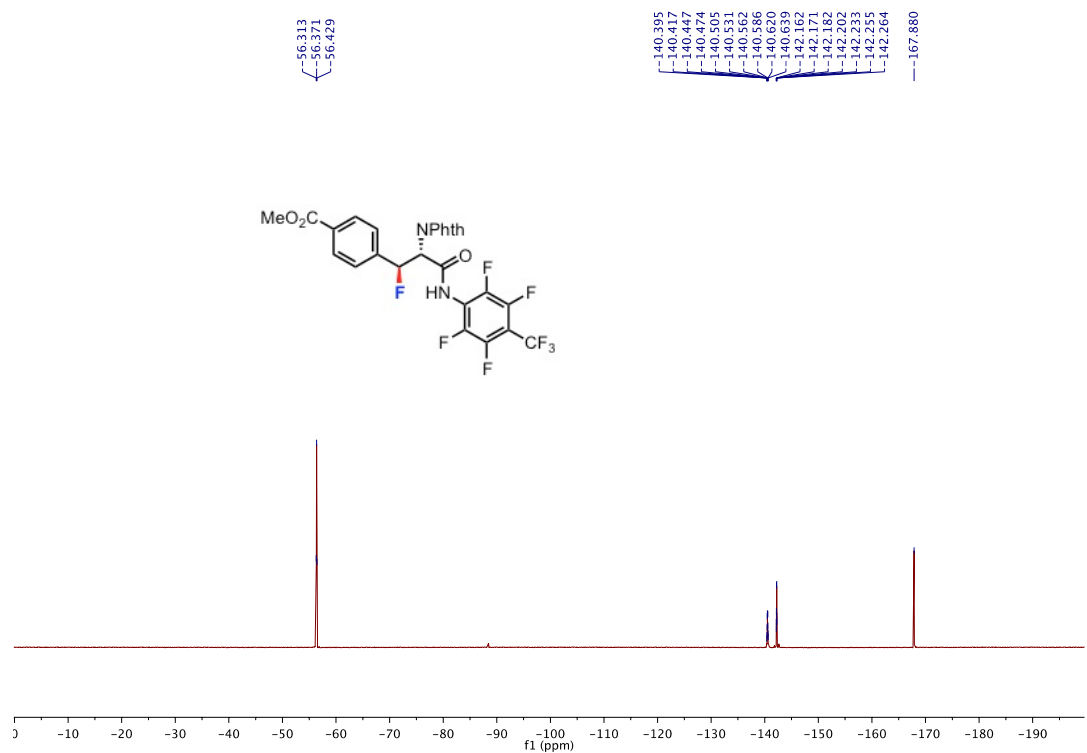


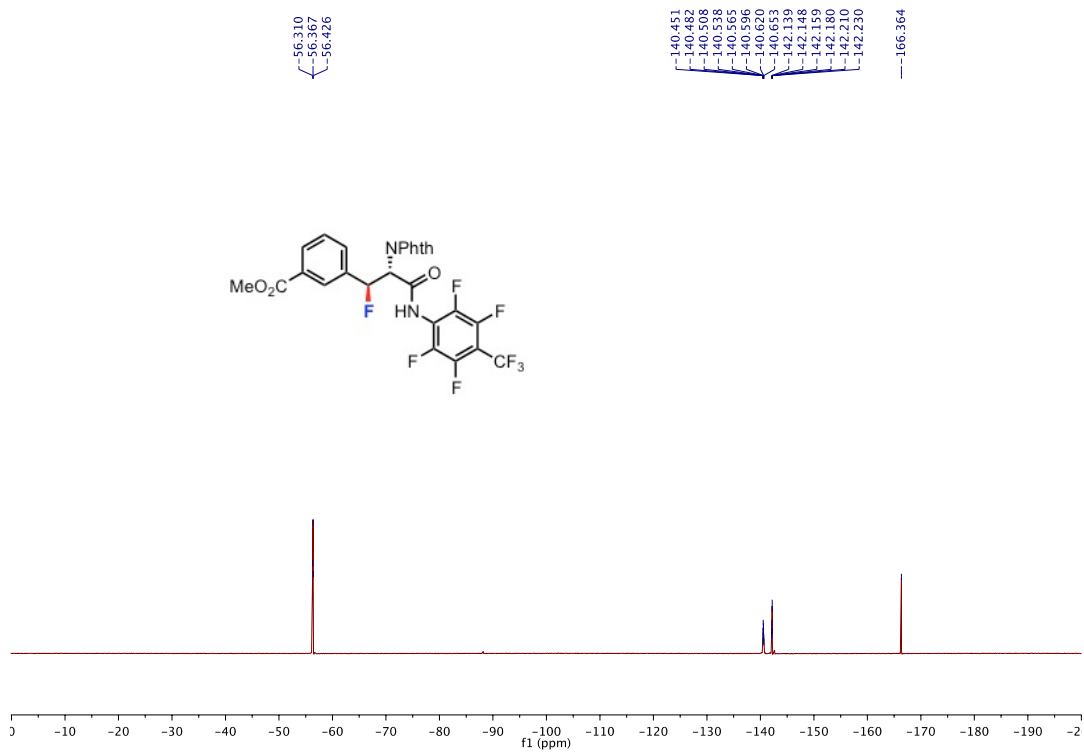
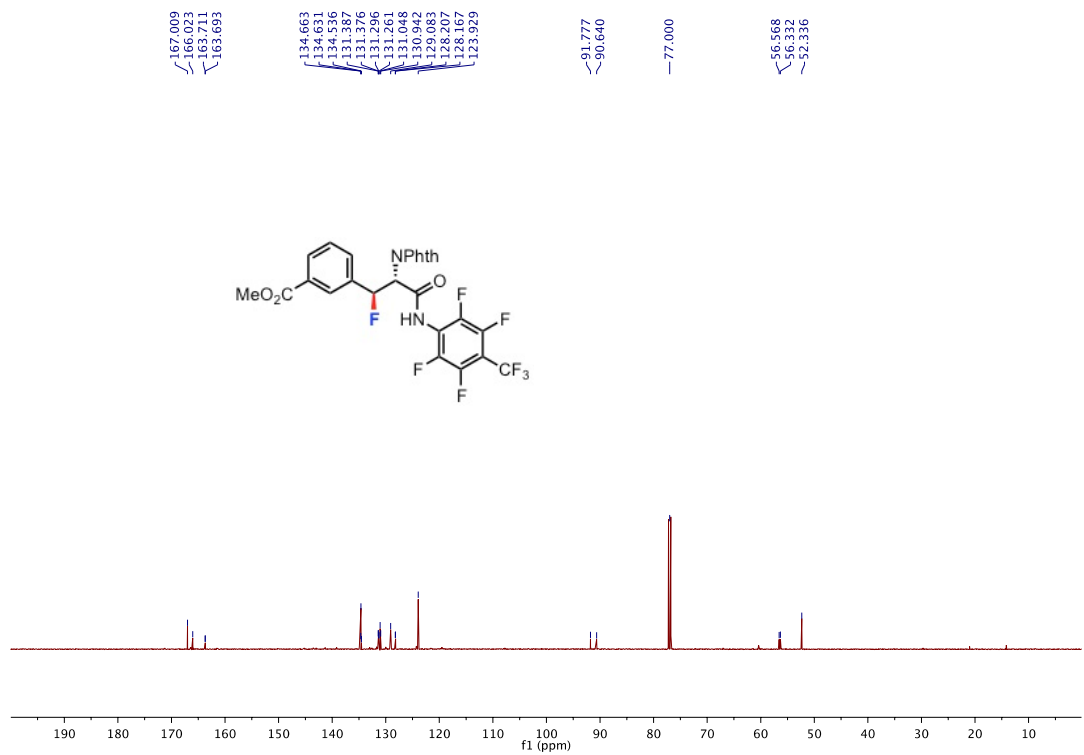


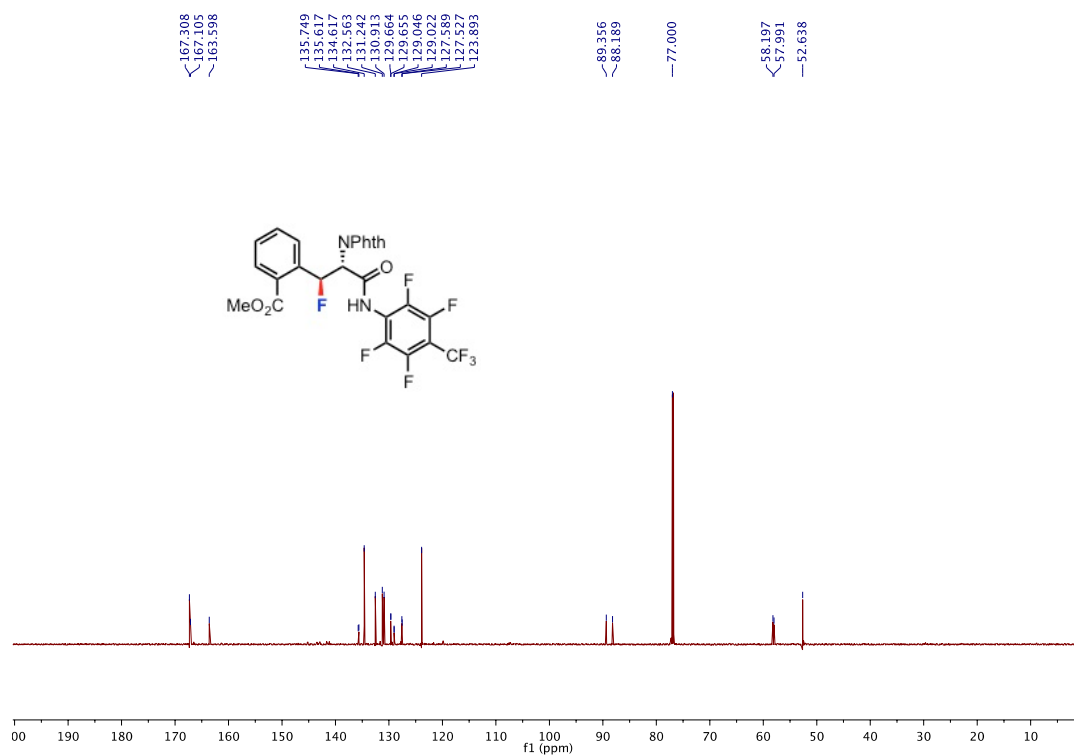
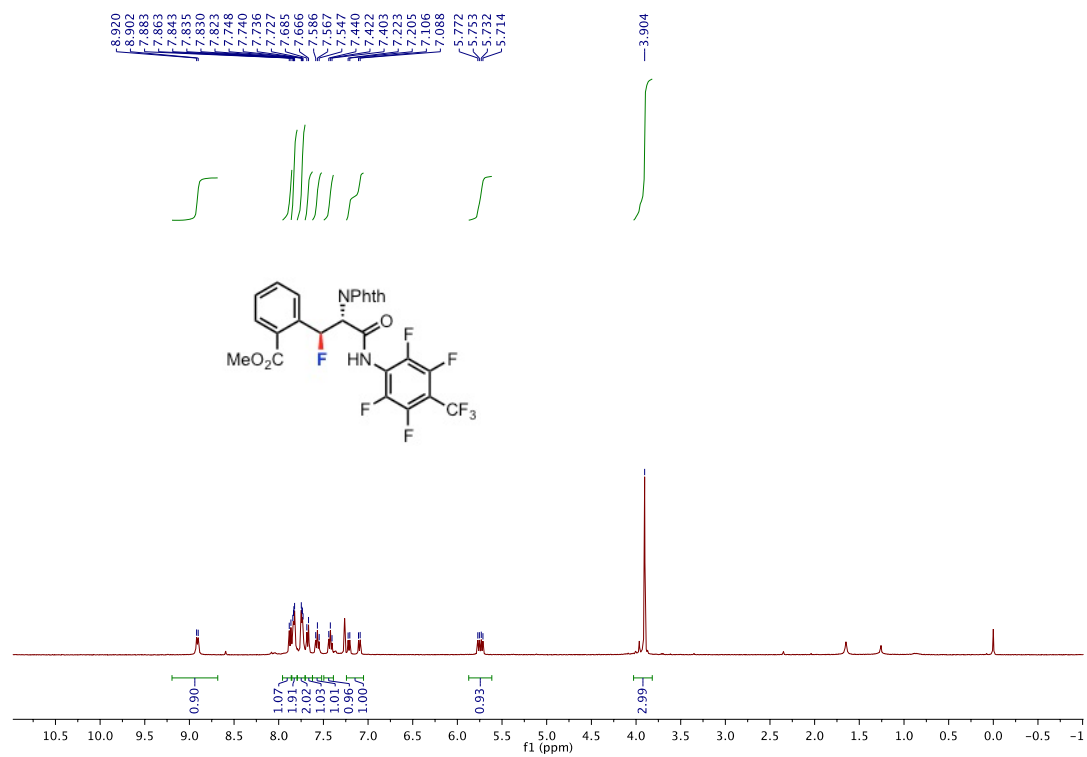


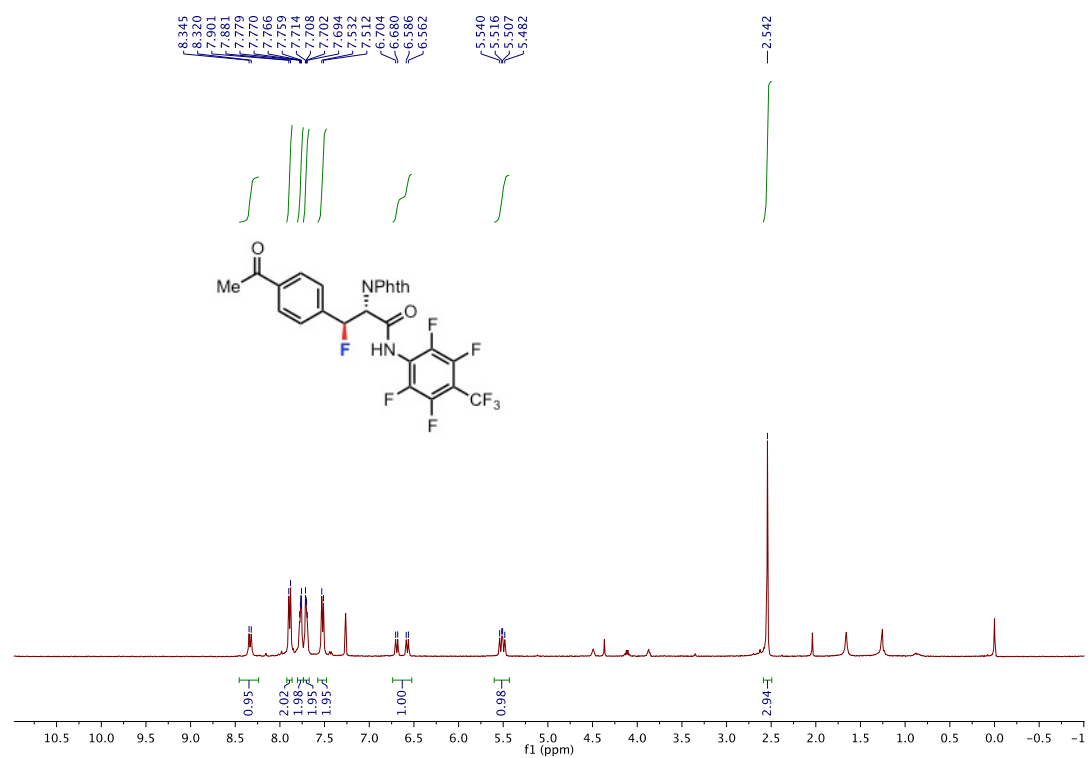
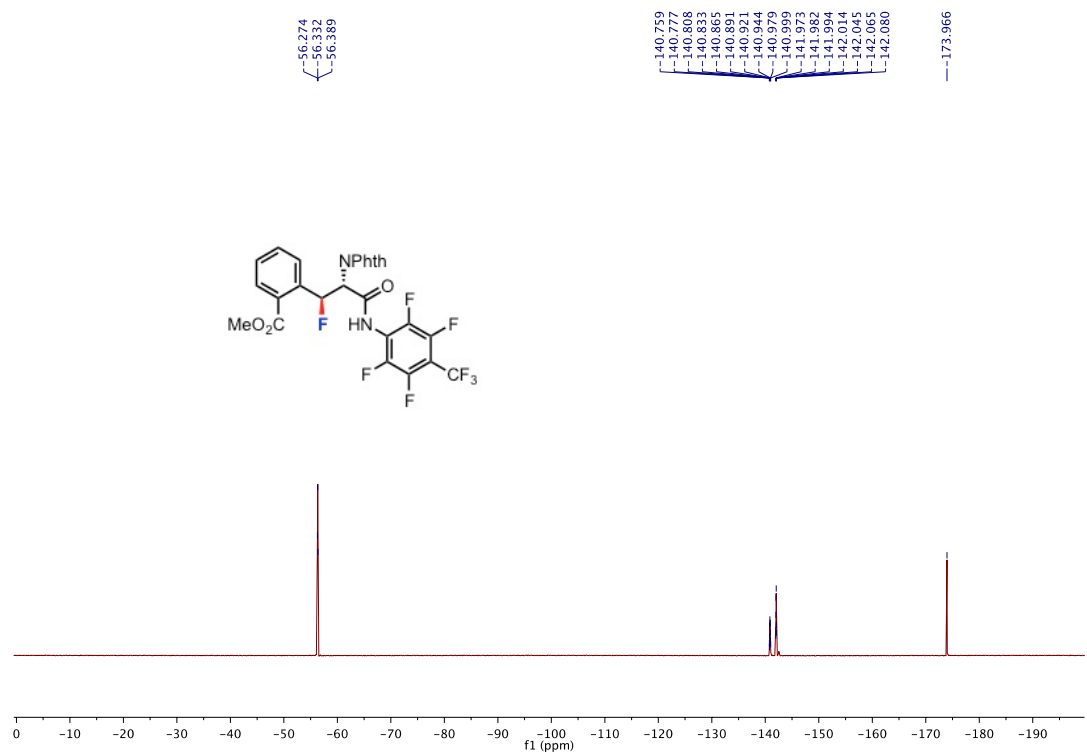


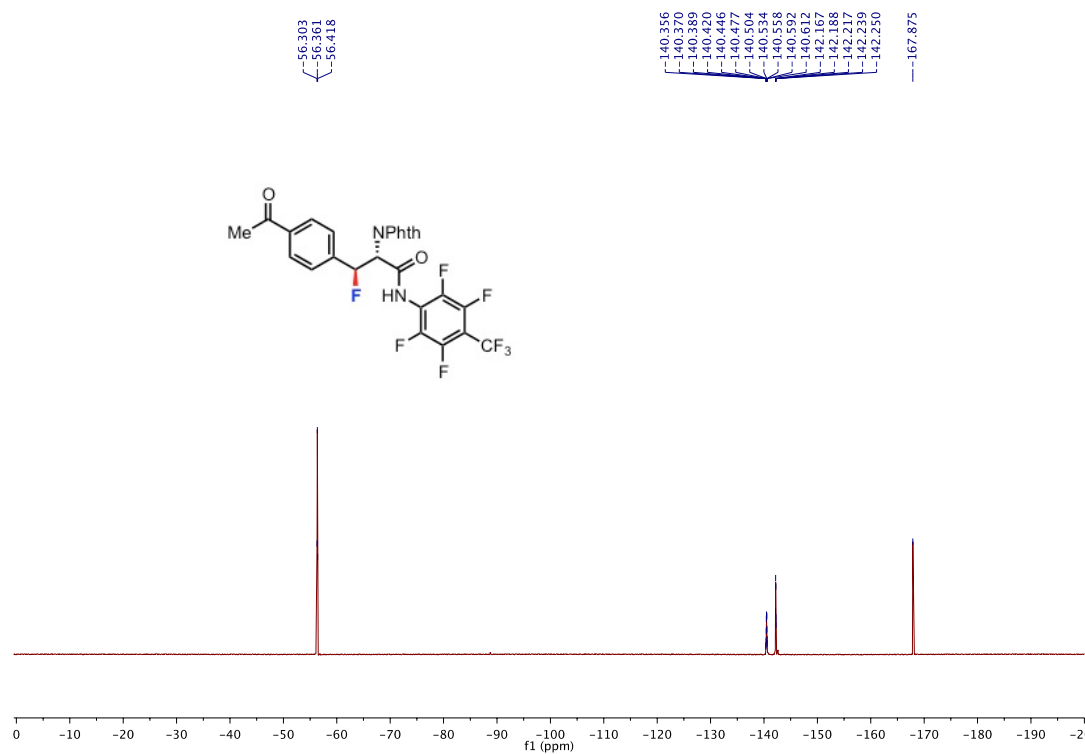
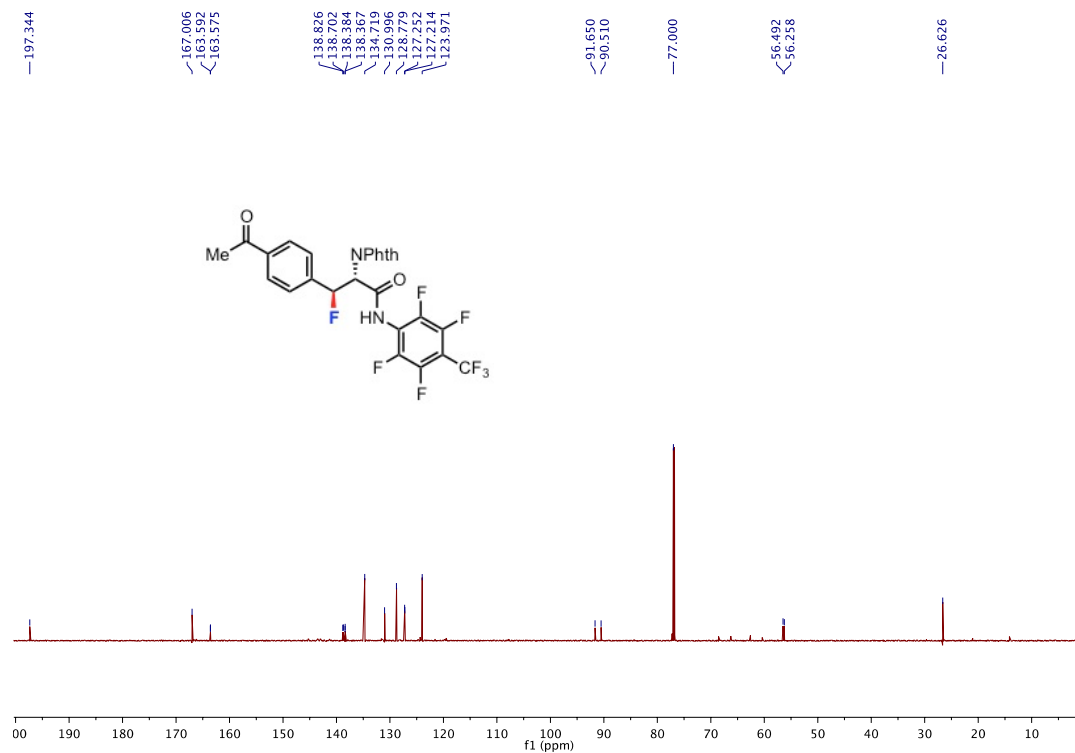


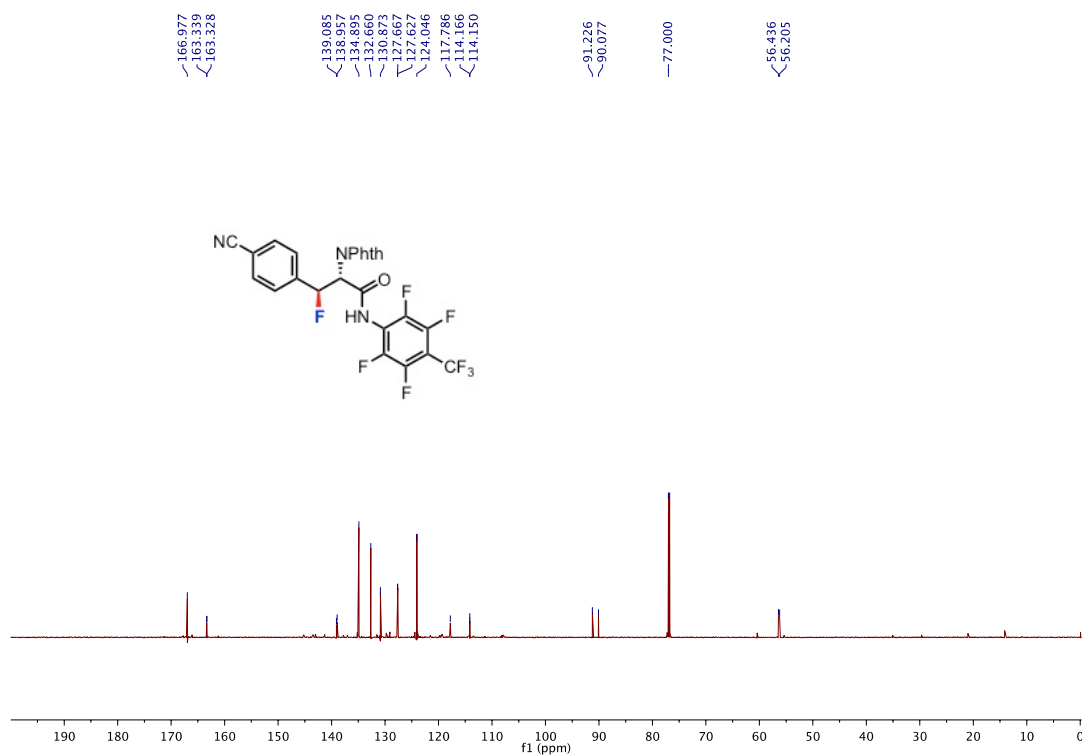
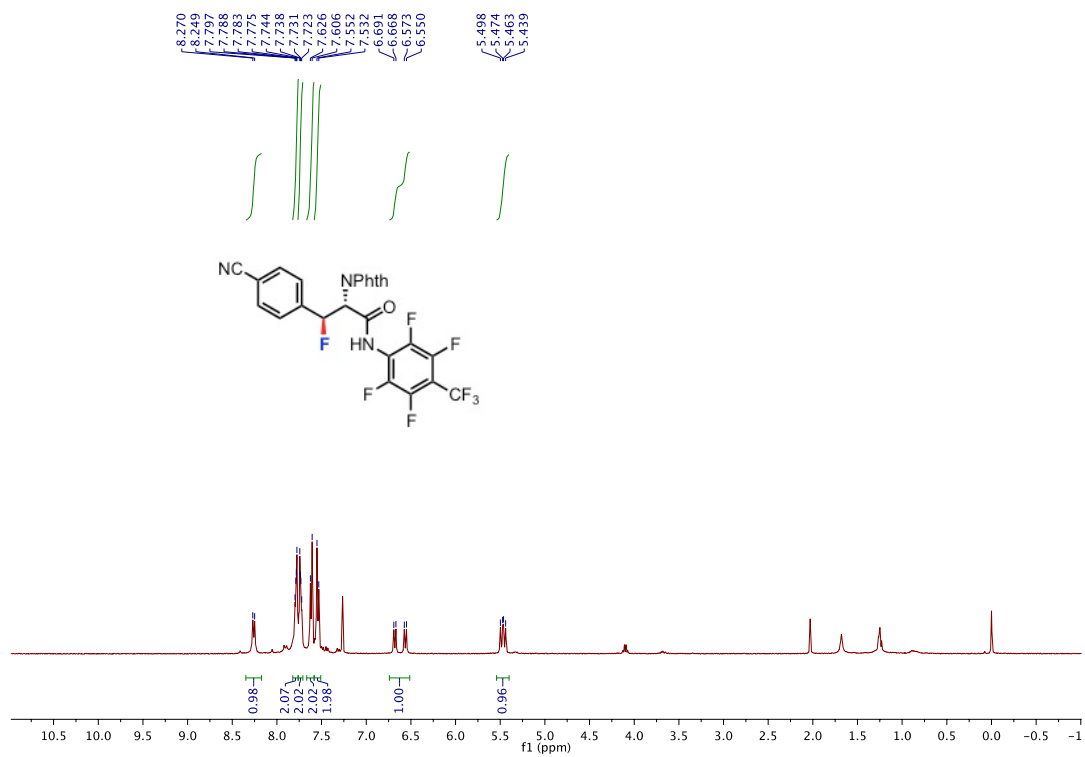


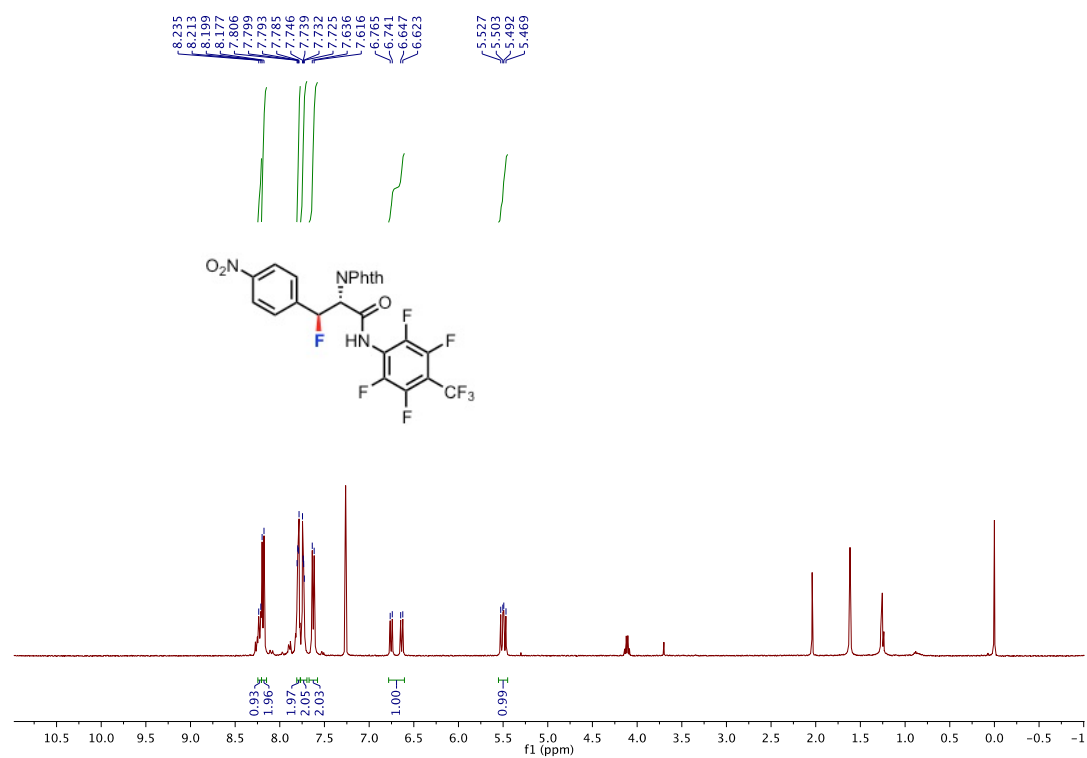
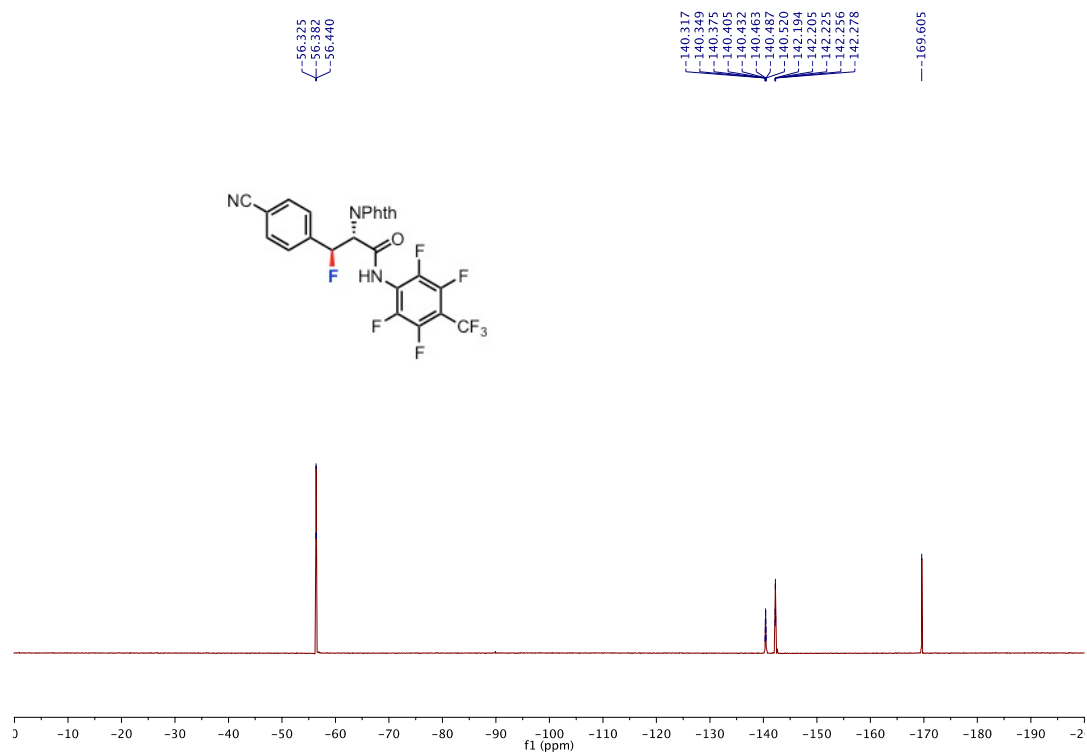


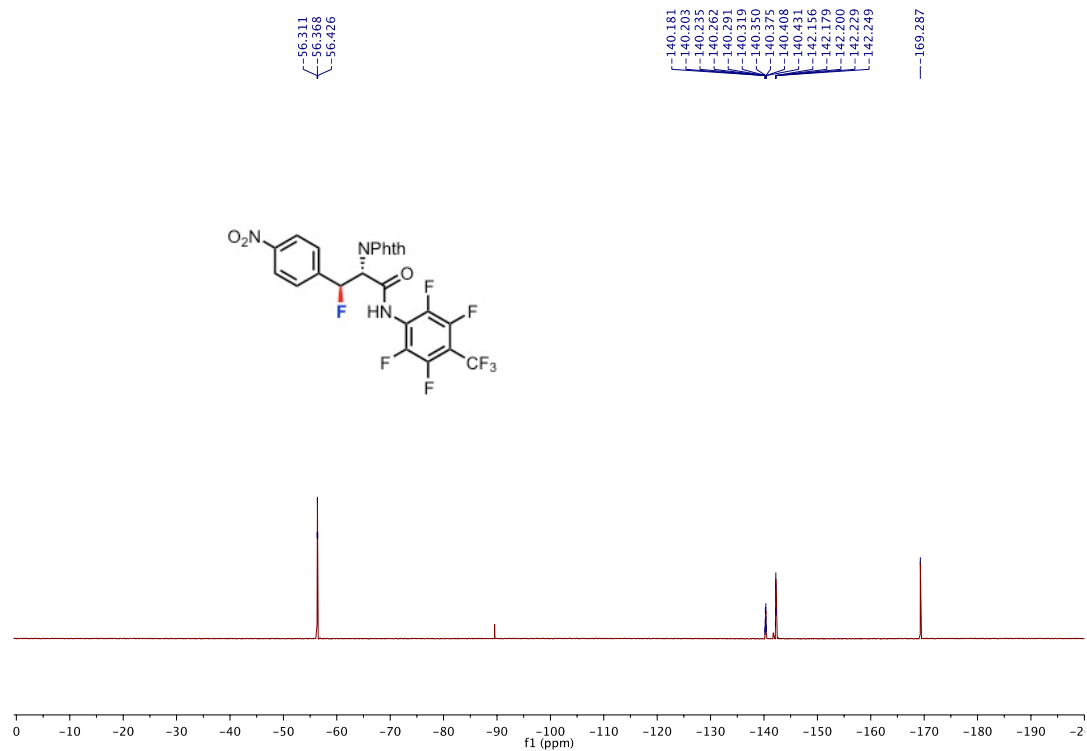
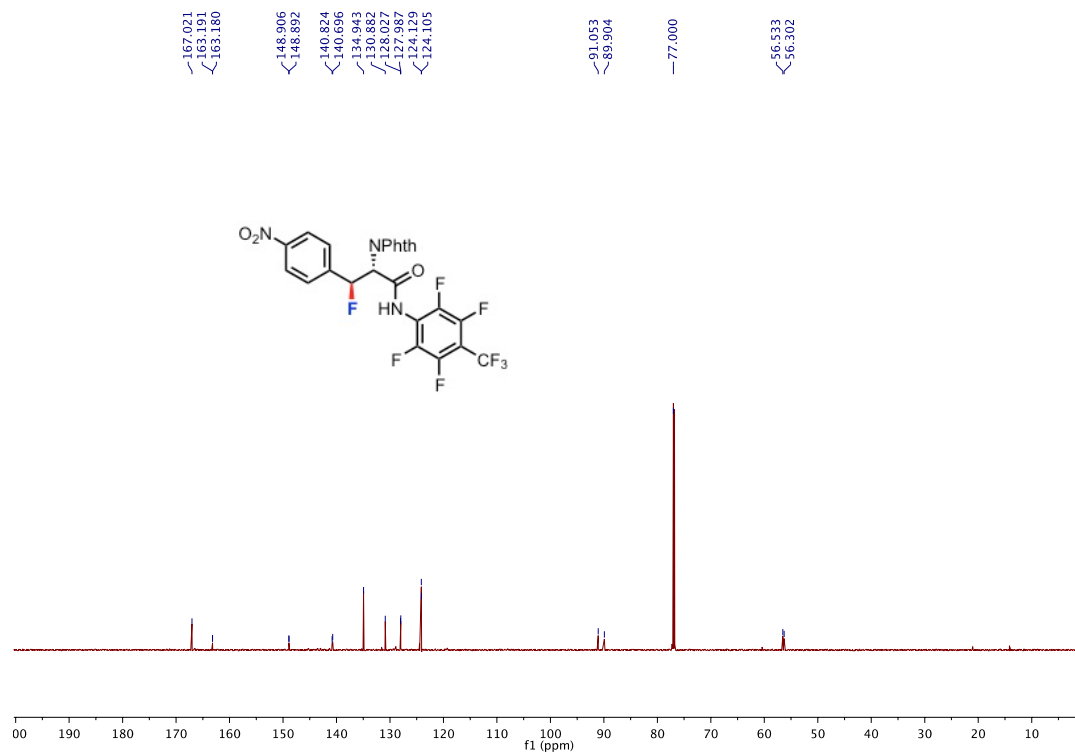


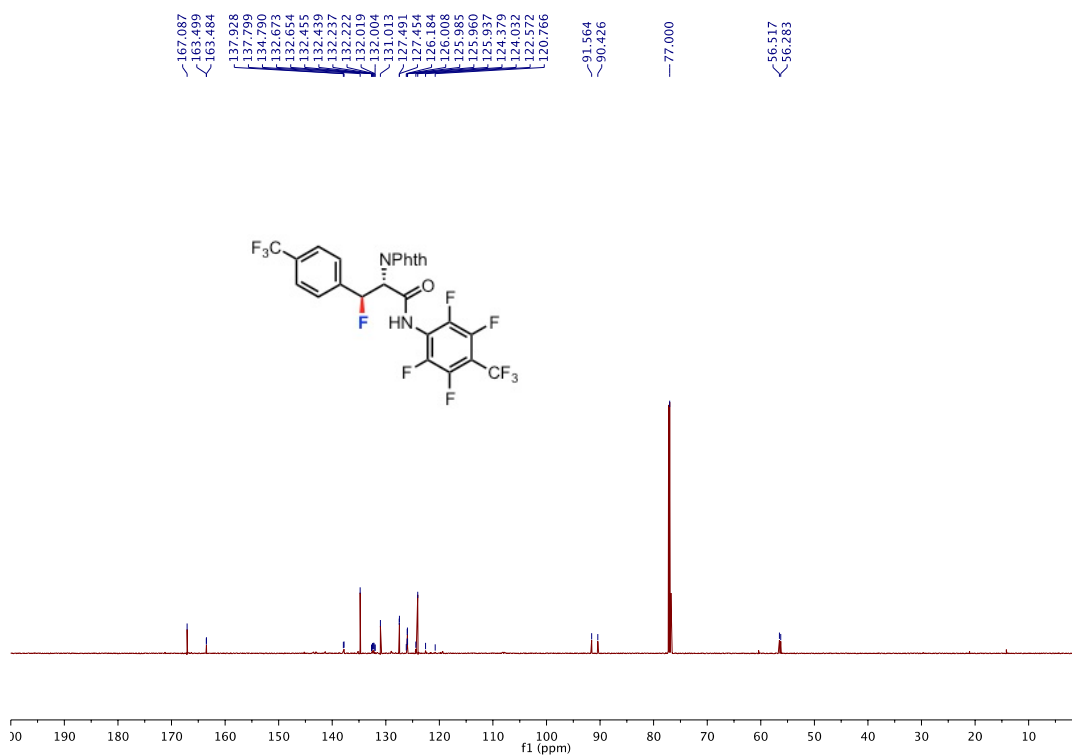
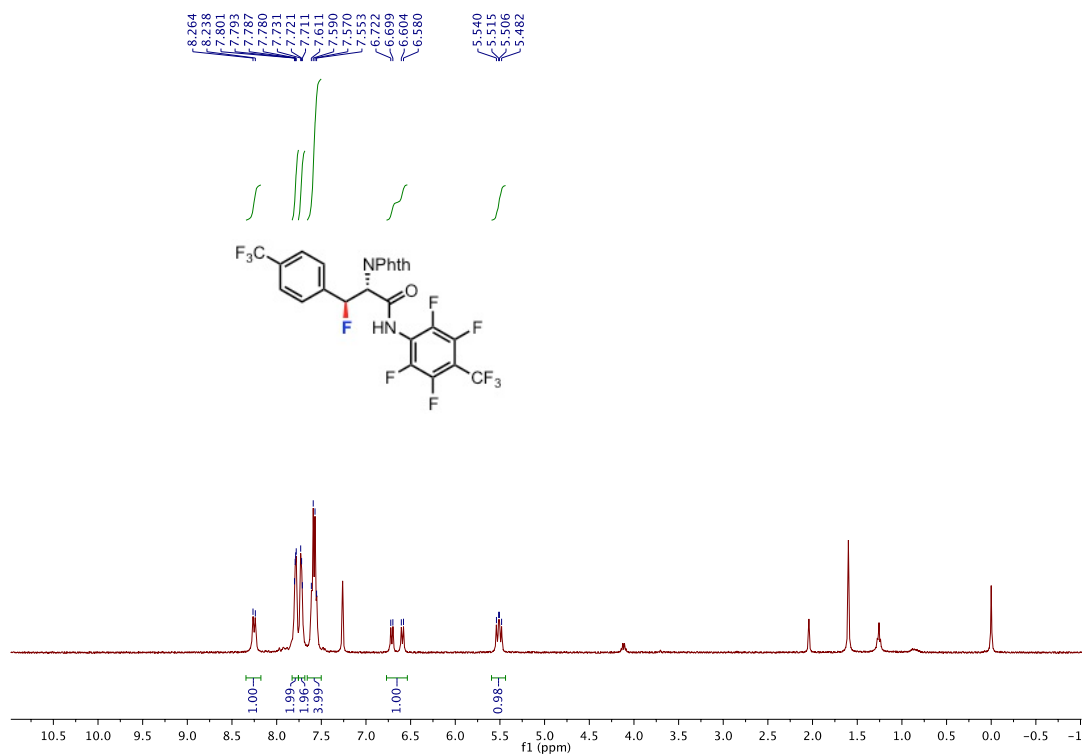


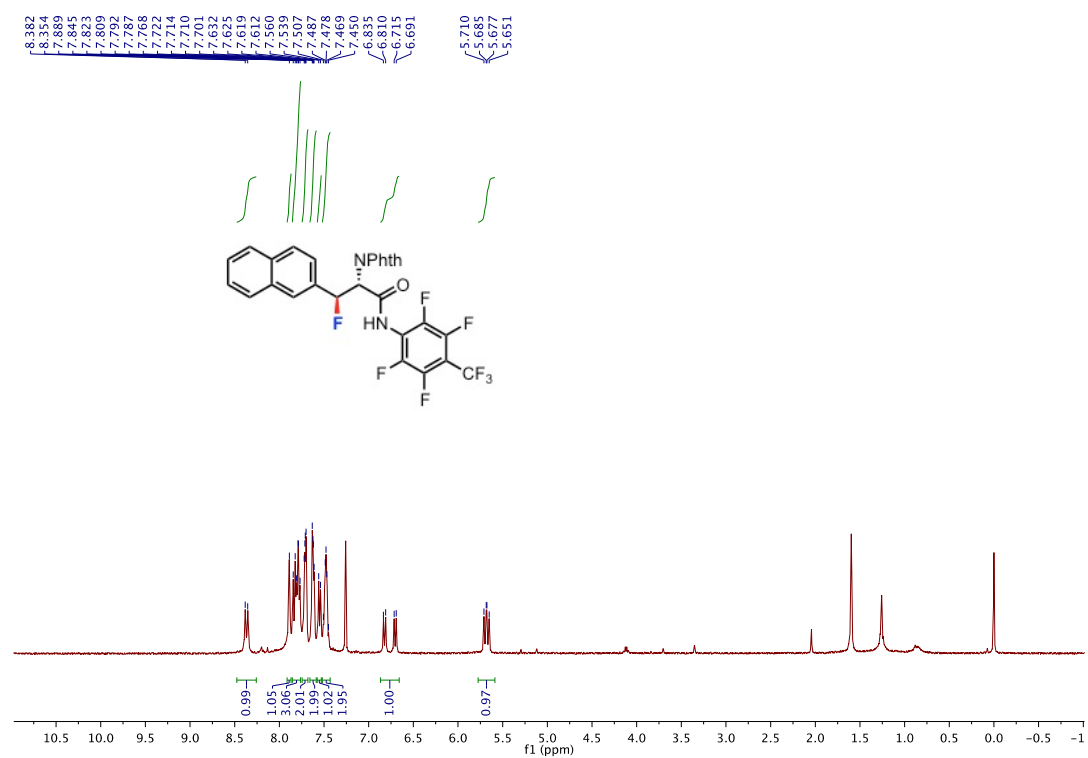
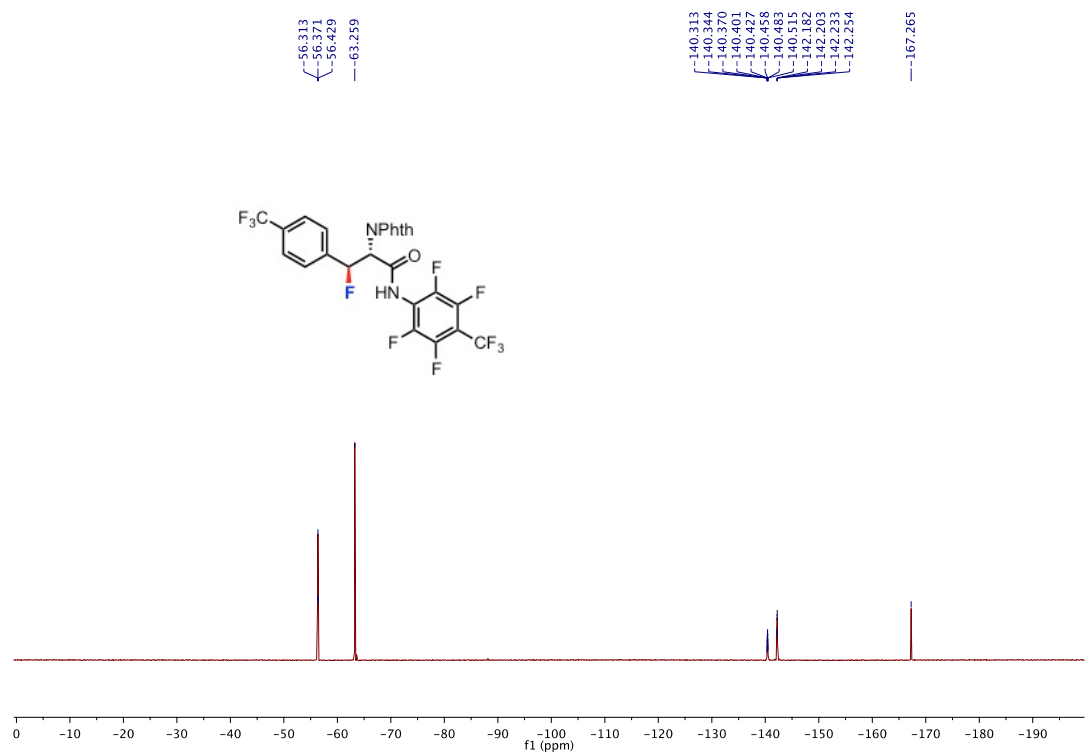


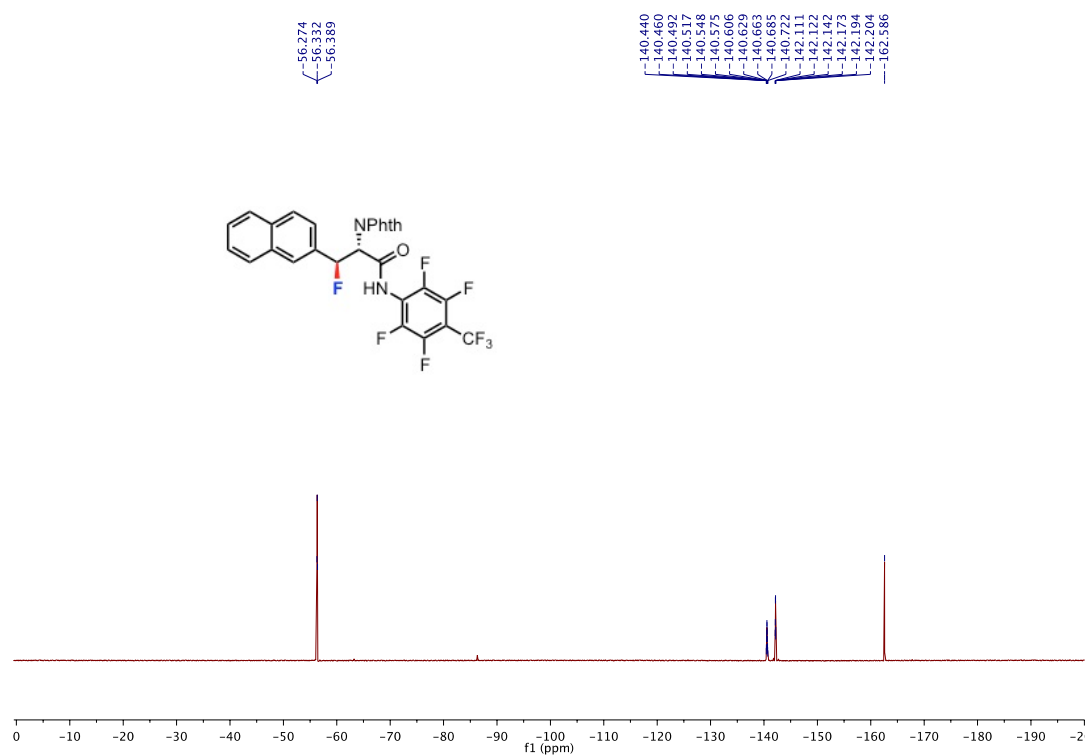
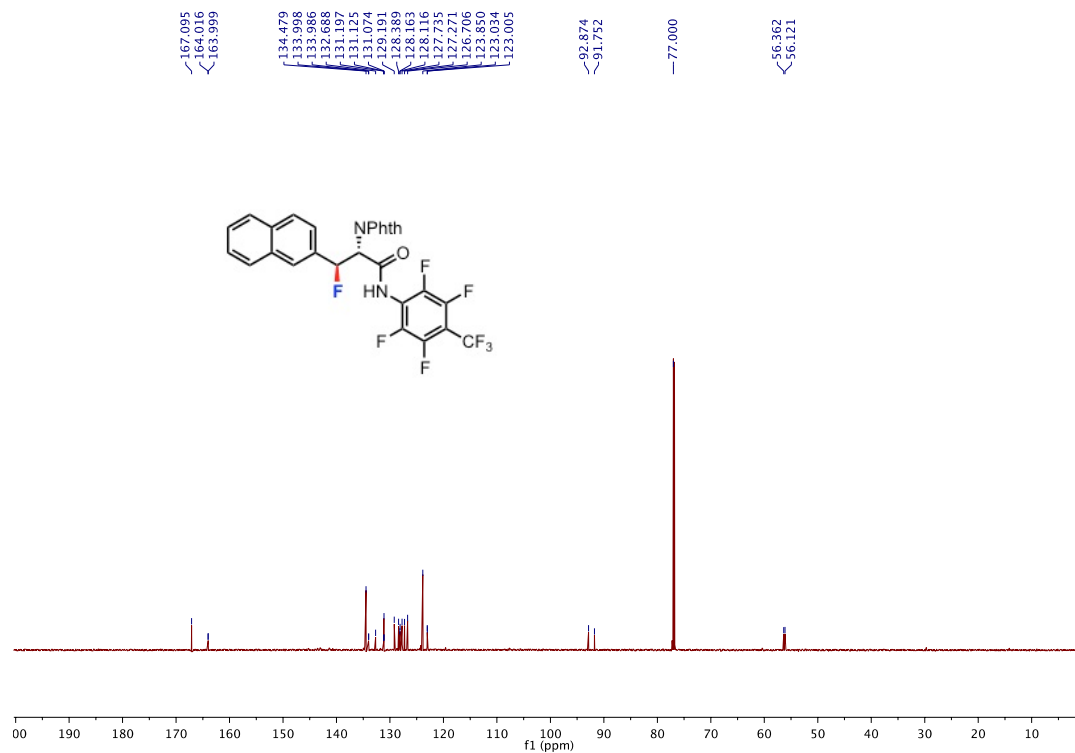


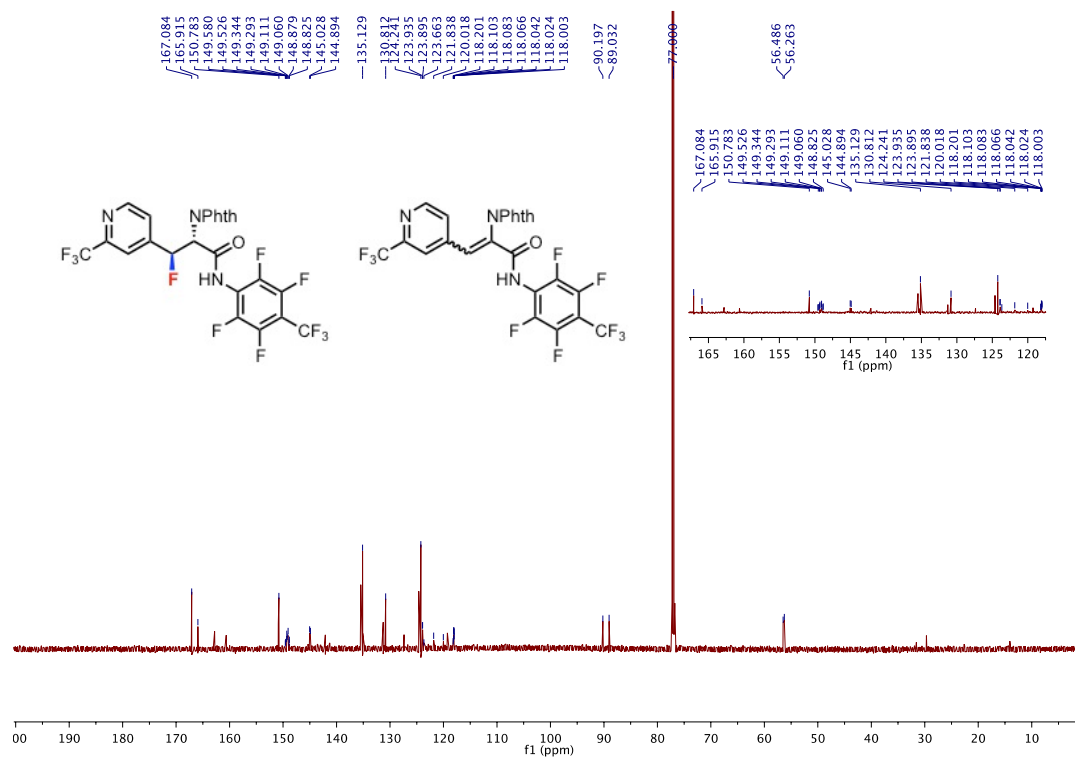
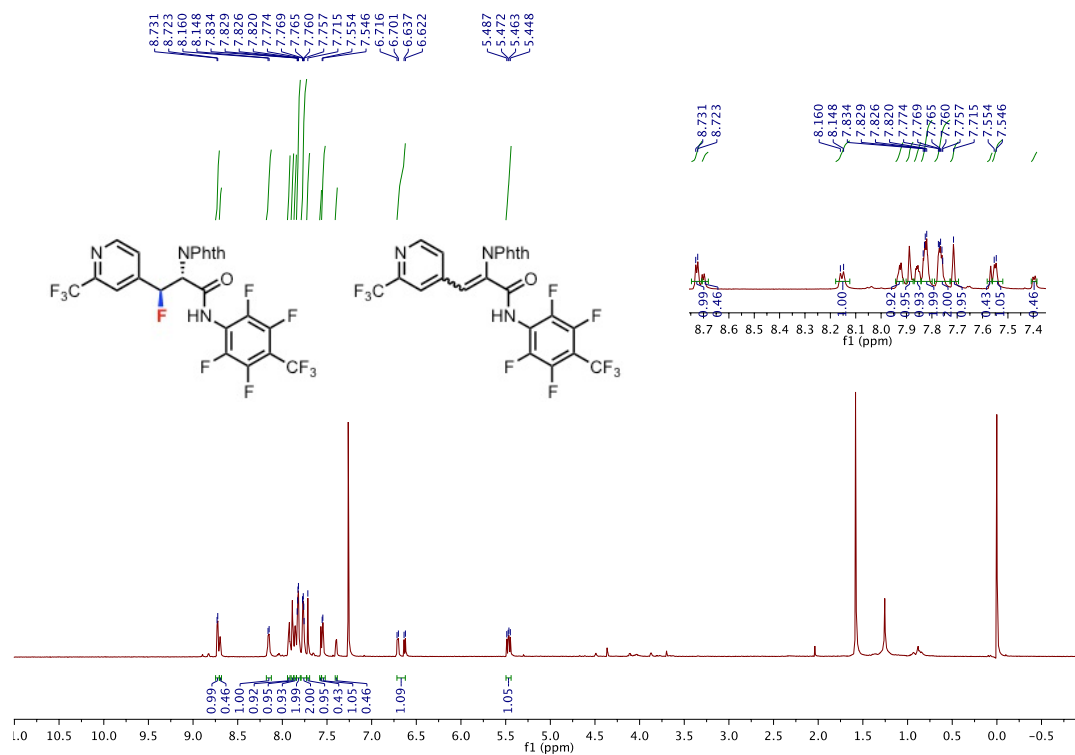


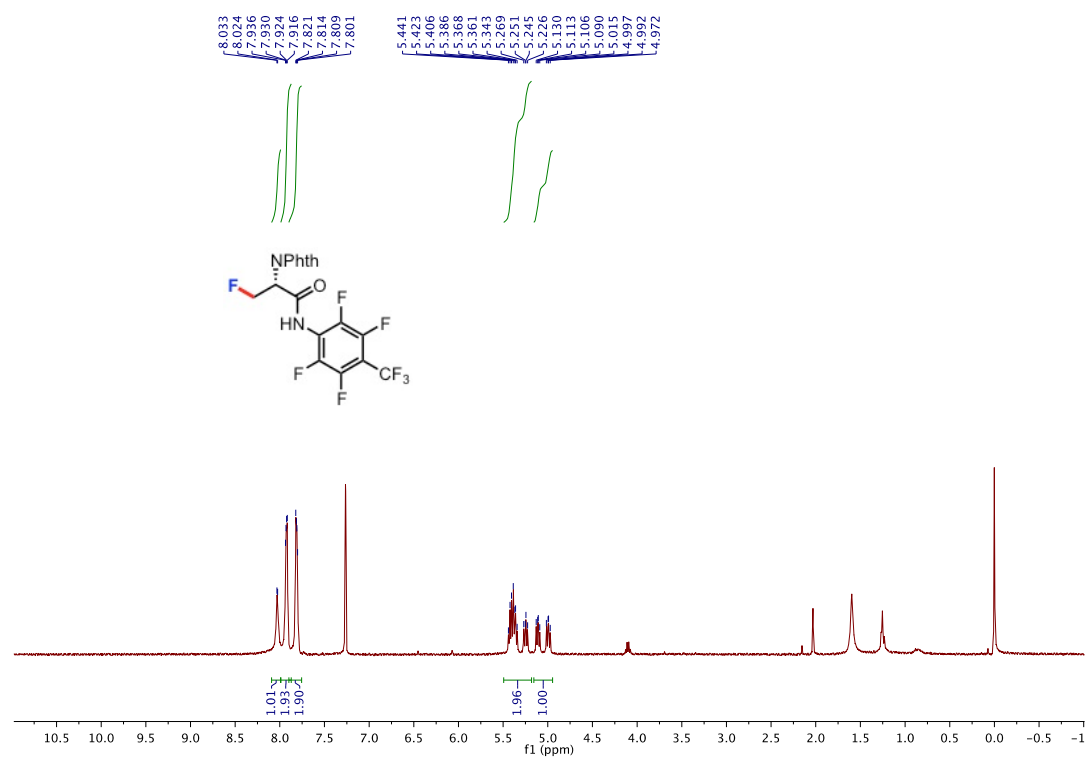
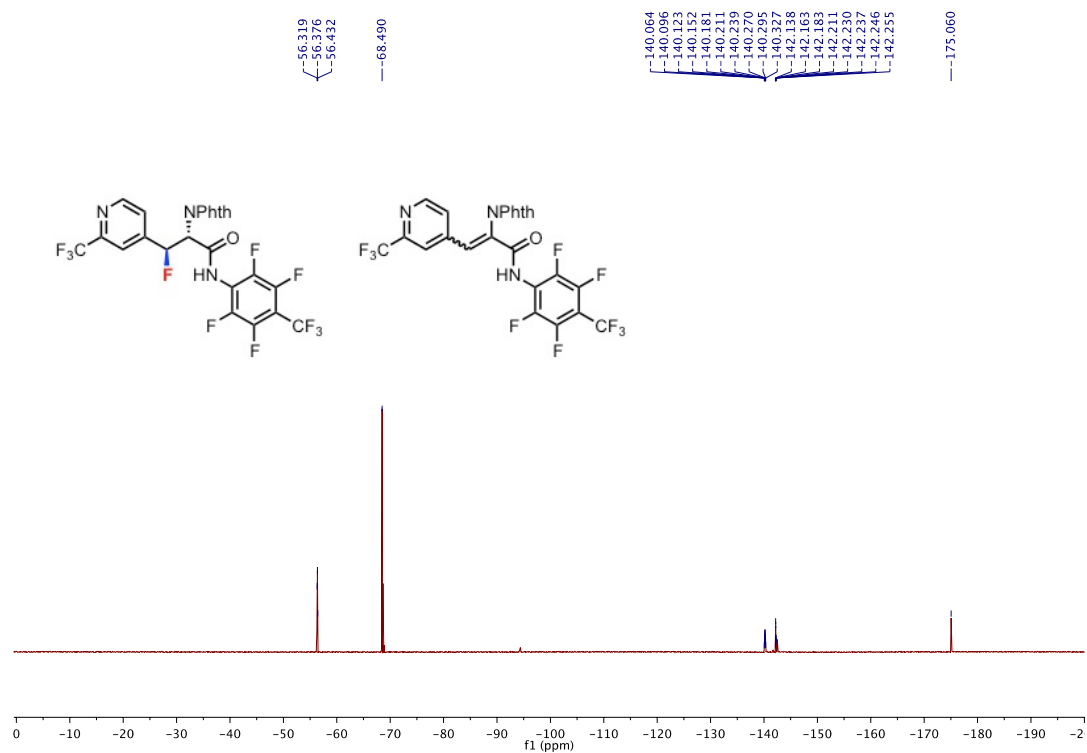


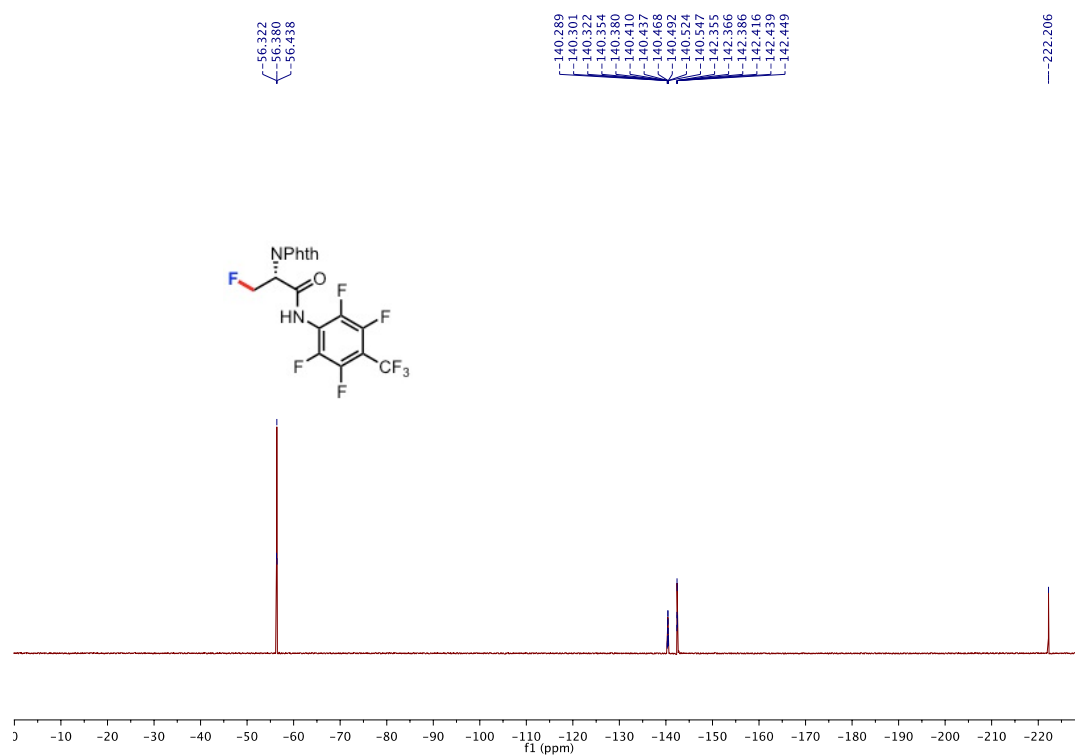
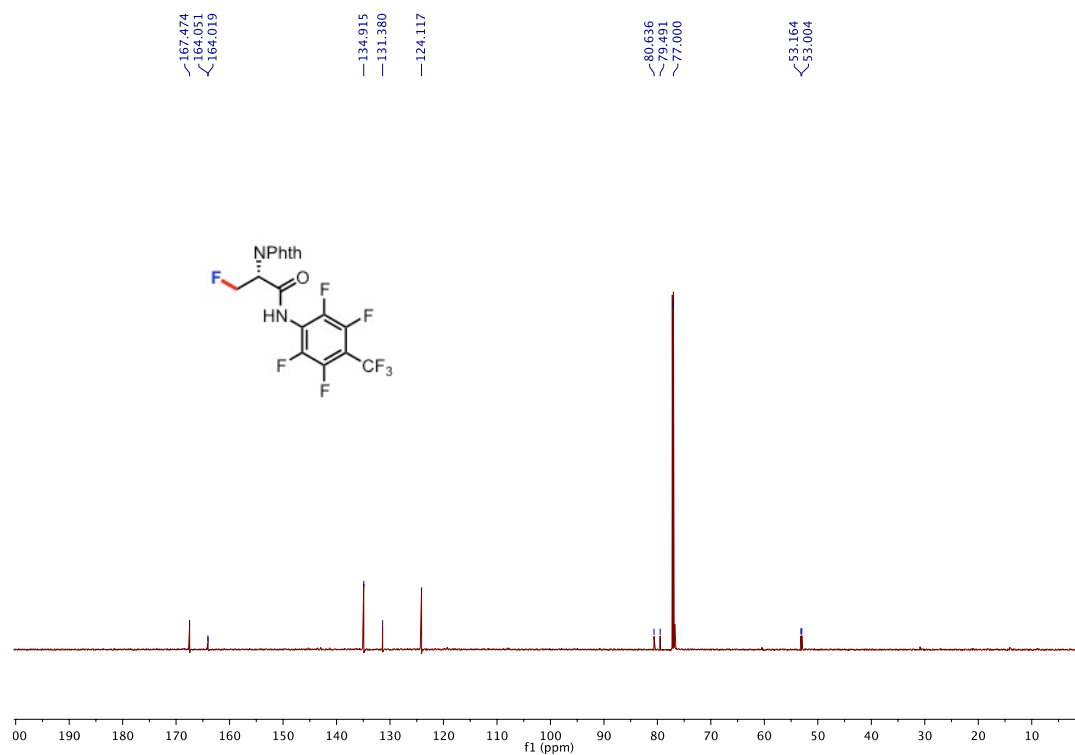


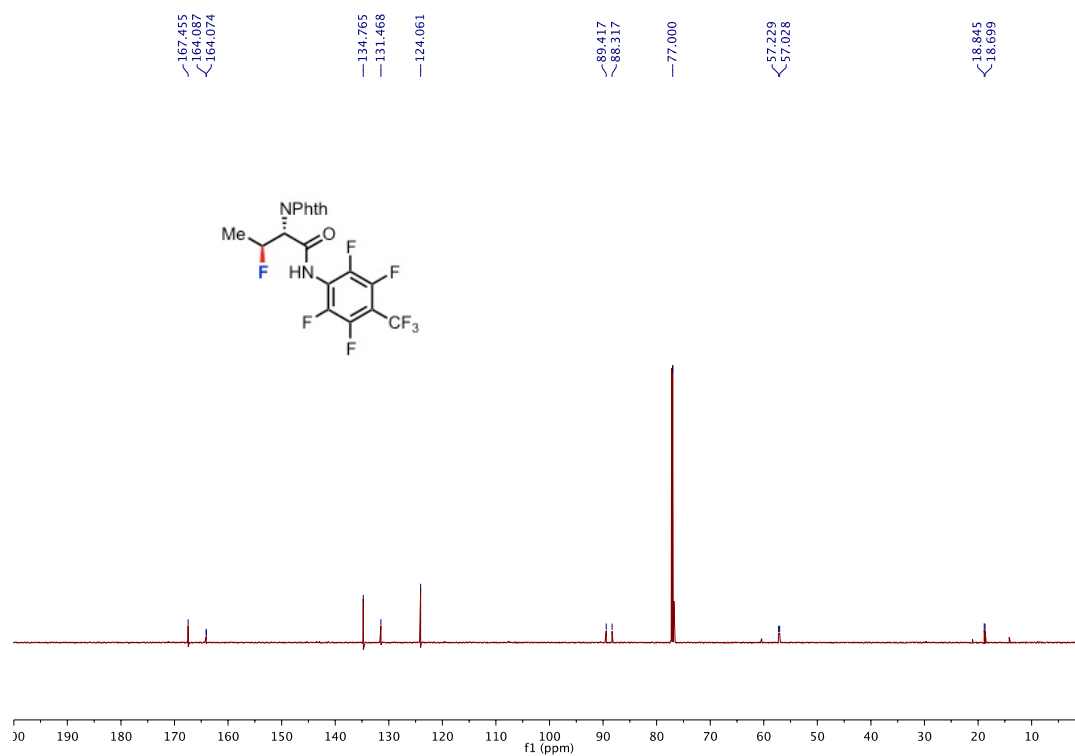
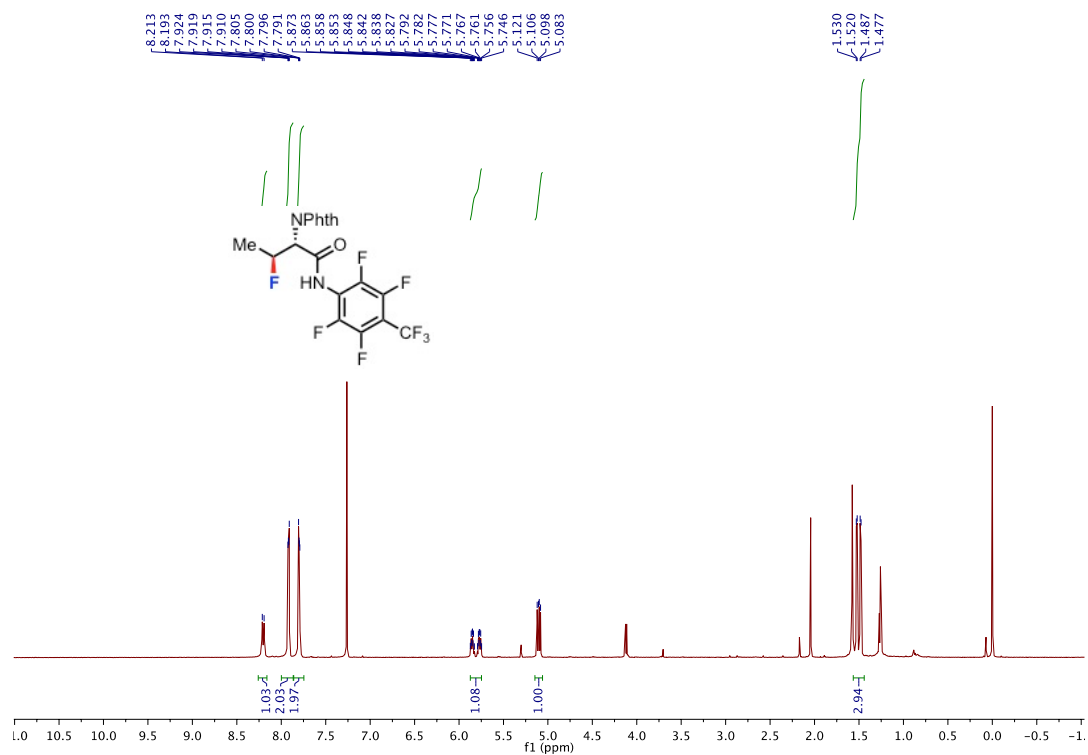


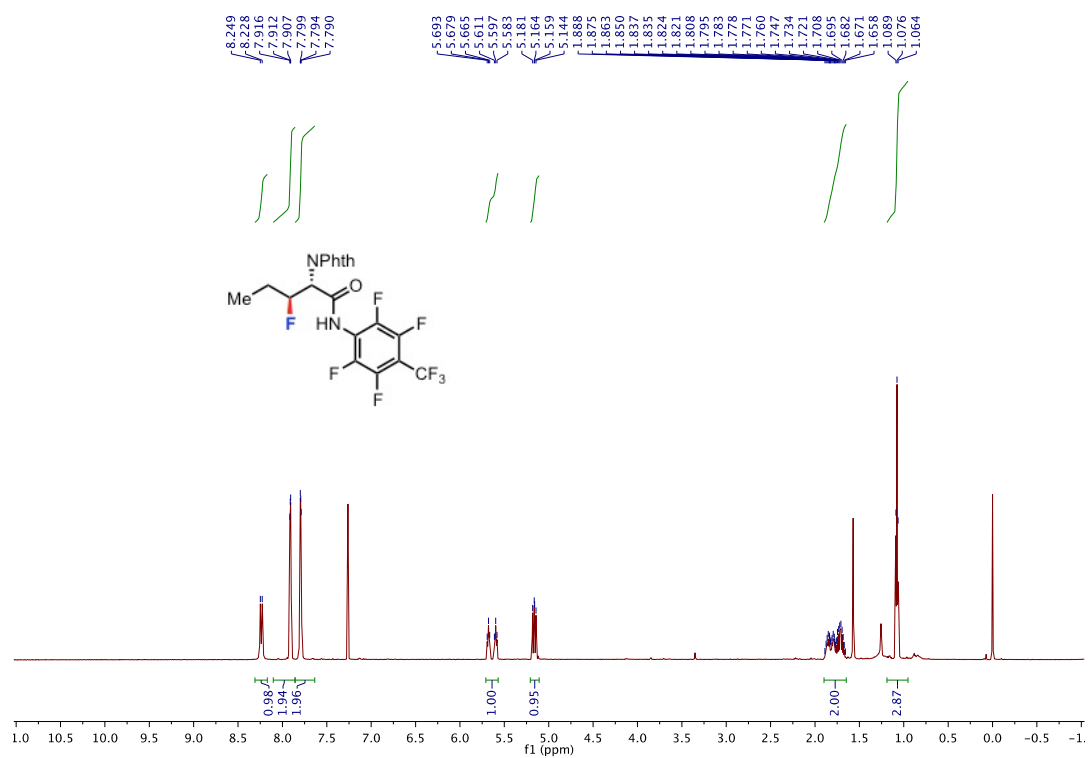
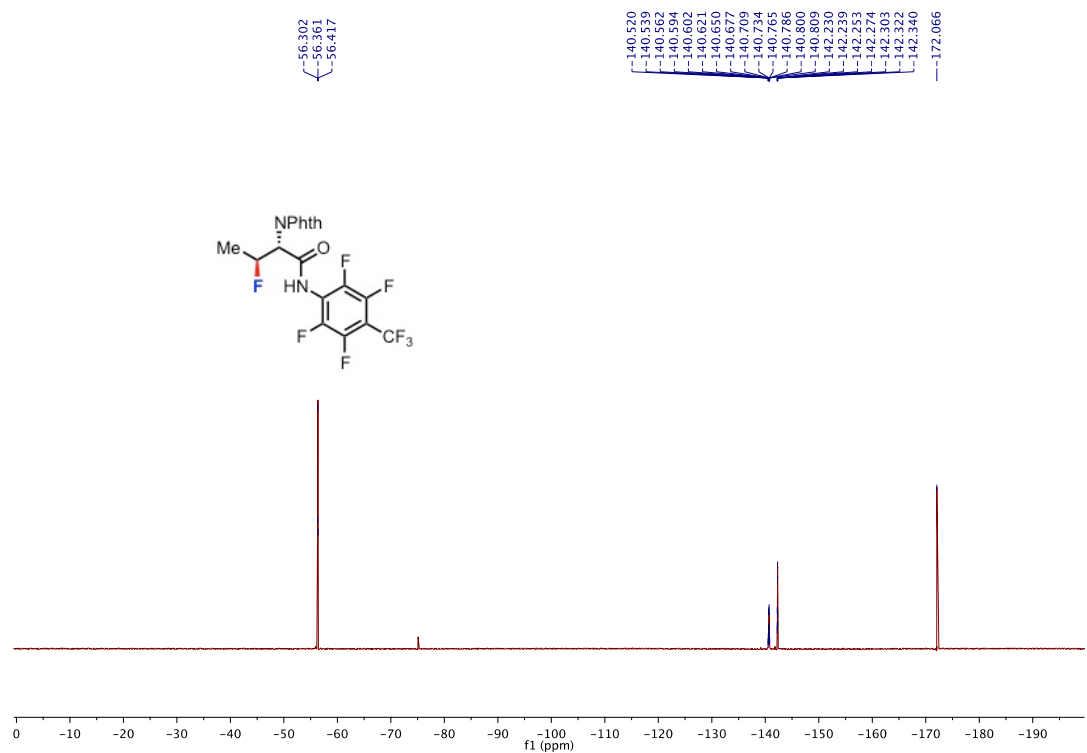


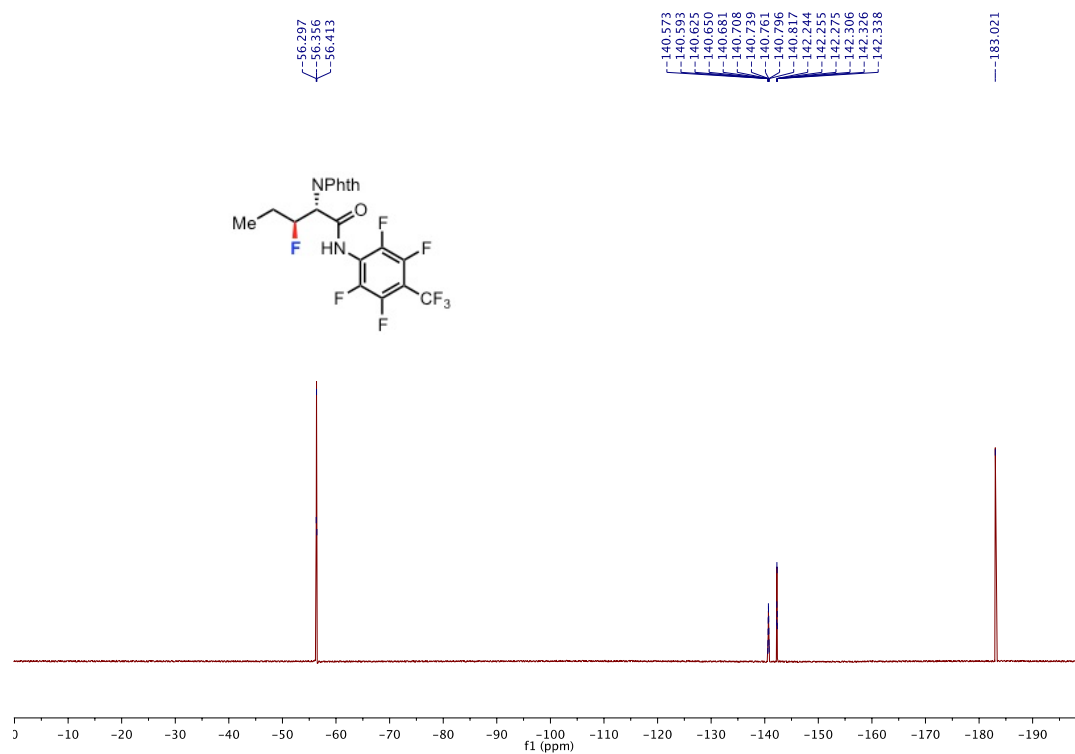
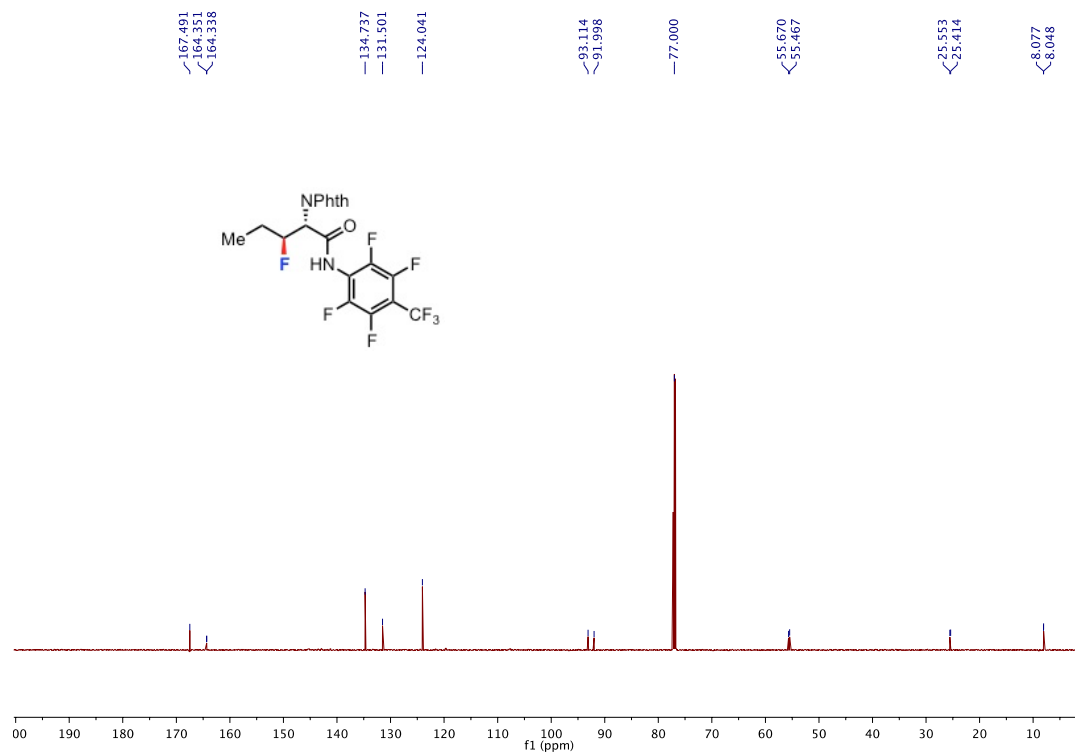


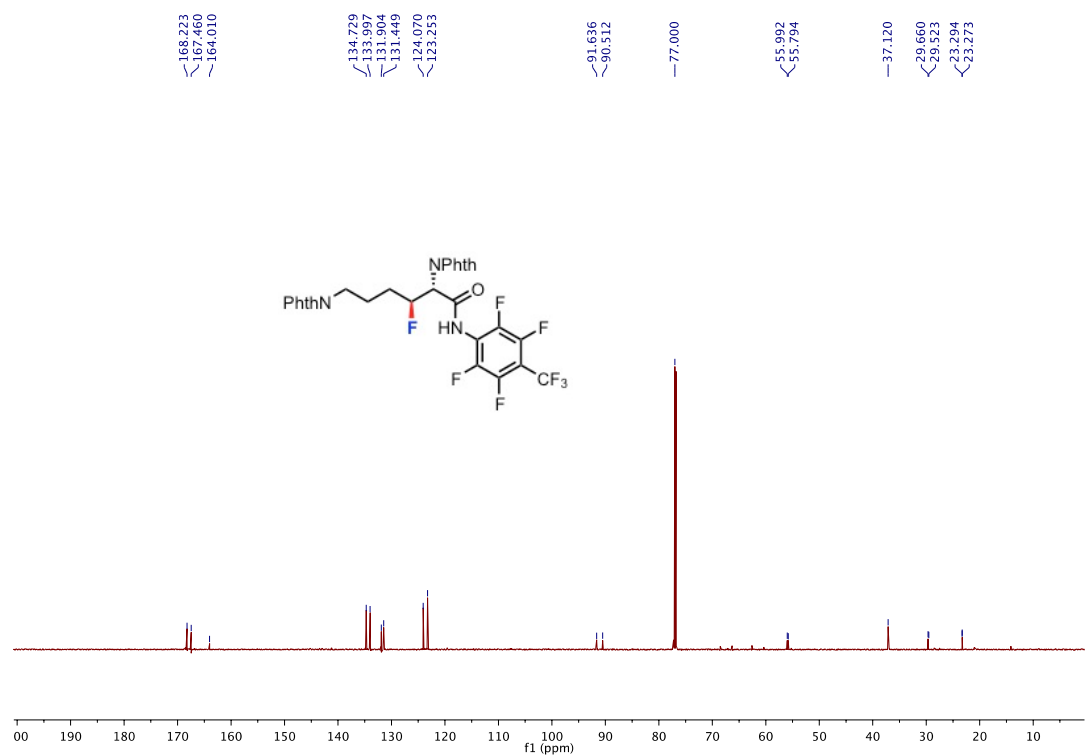
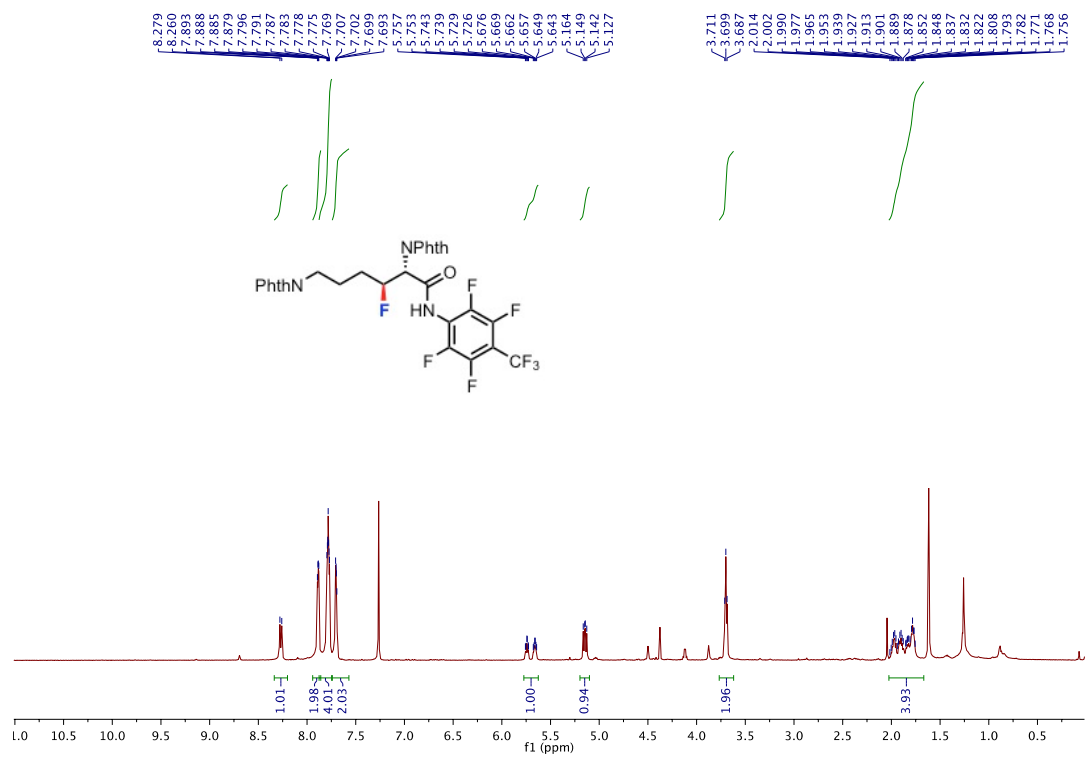


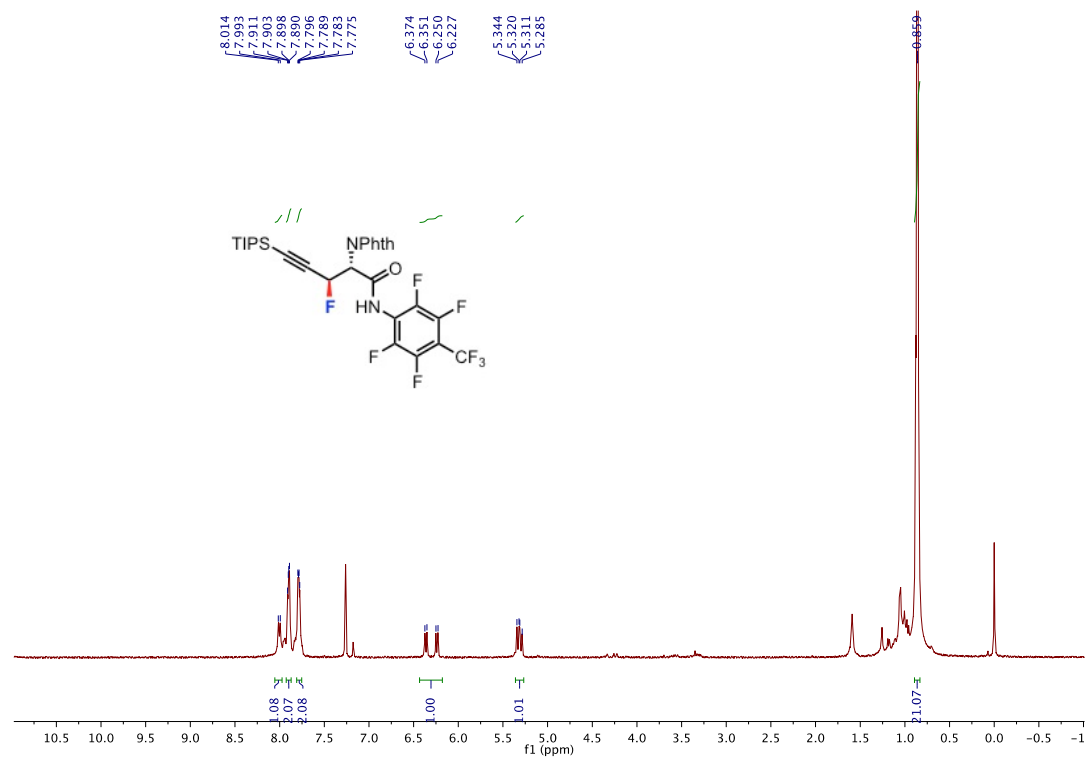
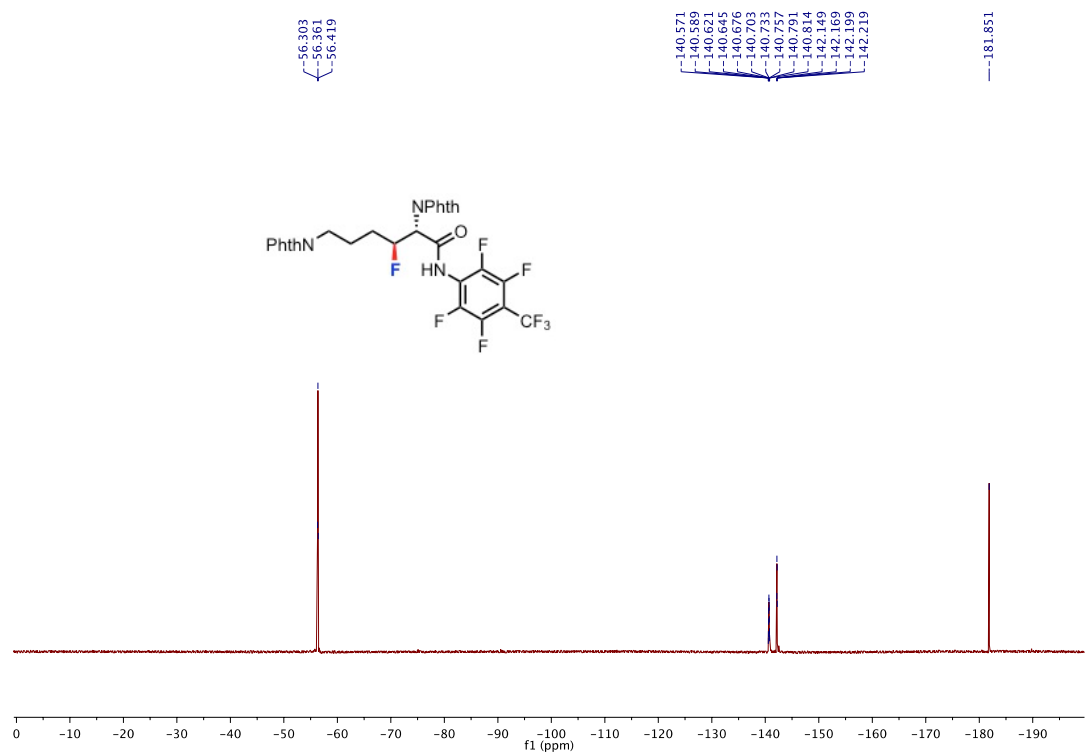


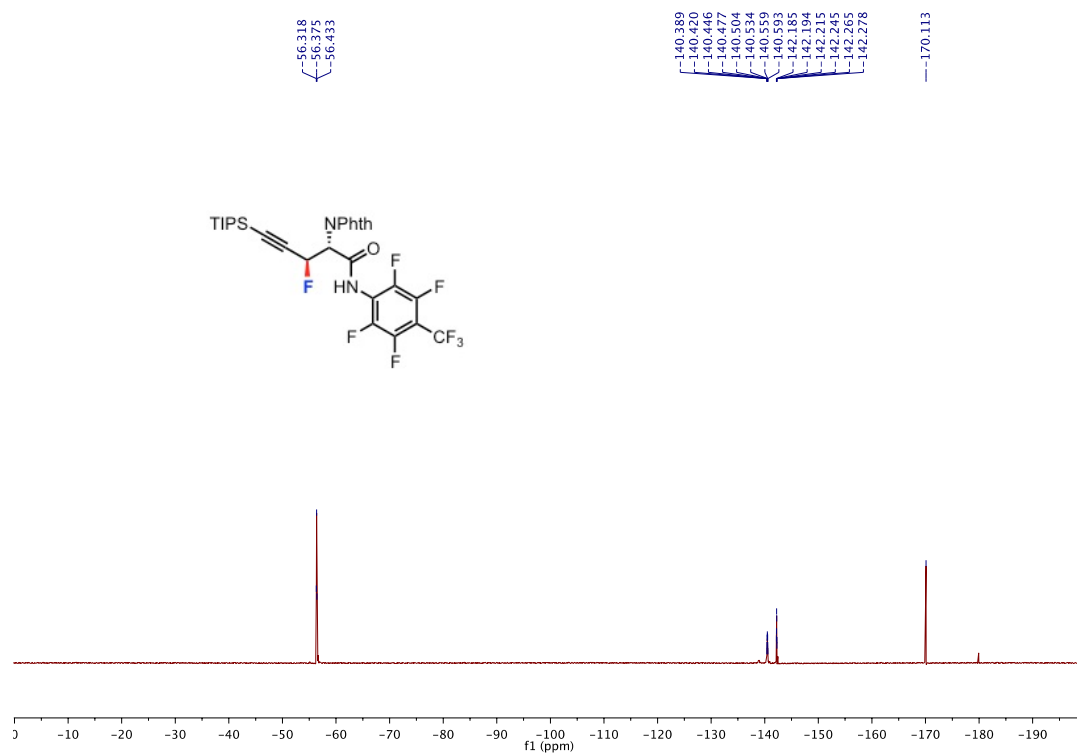
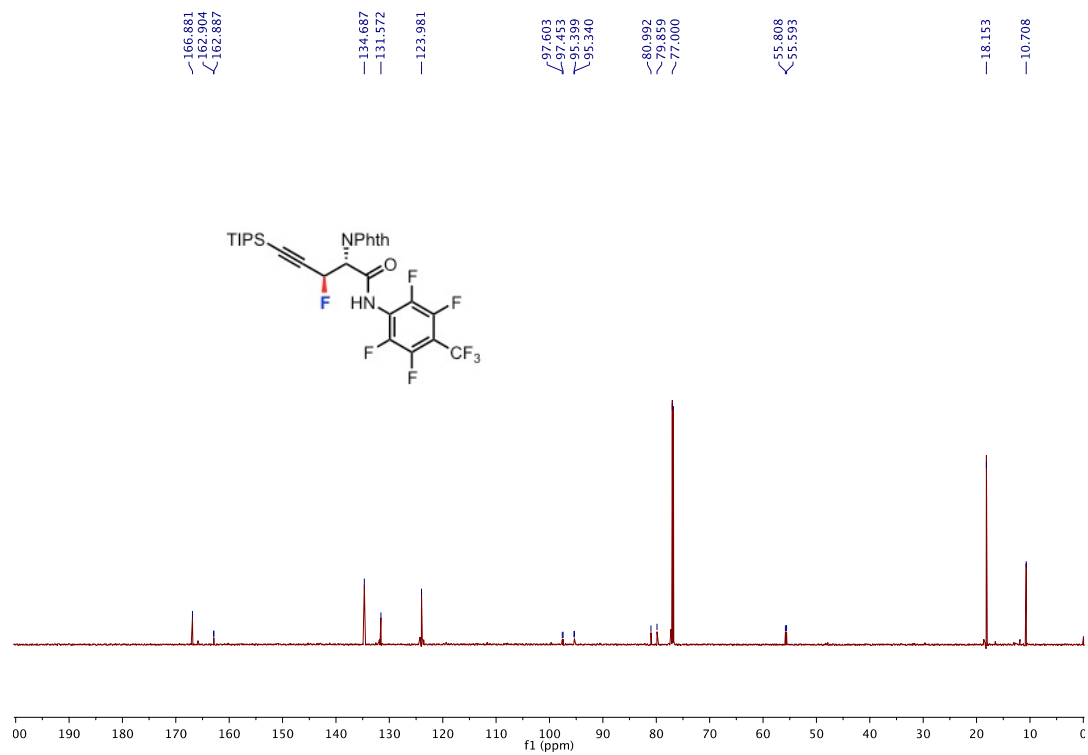












X-ray Crystallographic Data of 2c

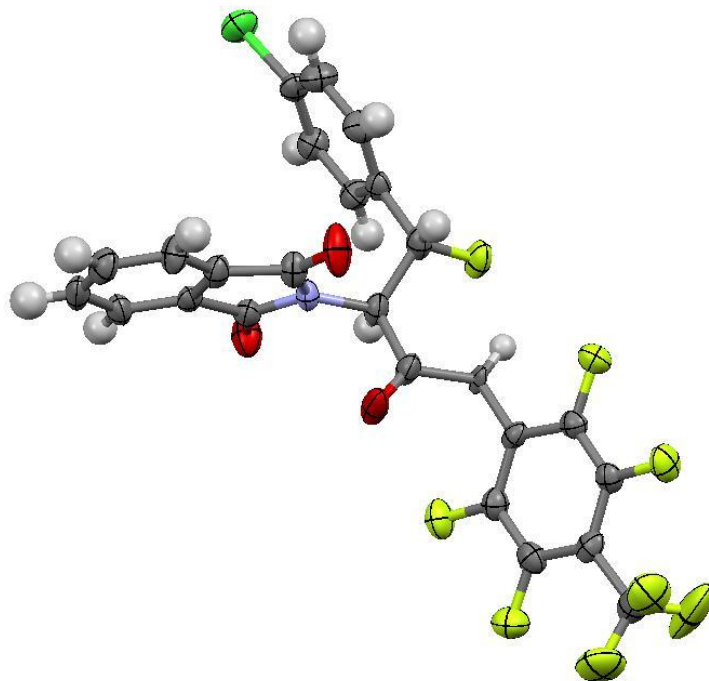


Table 1. Crystal data and structure refinement for **2c**.

Identification code	2c	
Empirical formula	$\text{C}_{24}\text{H}_{11}\text{ClF}_8\text{N}_2\text{O}_3$	
Formula weight	562.80	
Temperature	100.0 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P1	
Unit cell dimensions	$a = 8.4169(5)$ Å	$\alpha = 100.935(2)^\circ$.
	$b = 9.1537(5)$ Å	$\beta = 101.544(2)^\circ$.
	$c = 15.6960(8)$ Å	$\gamma = 104.301(2)^\circ$.
Volume	$1111.12(11)$ Å ³	
Z	2	
Density (calculated)	1.682 Mg/m ³	
Absorption coefficient	0.272 mm ⁻¹	
F(000)	564	
Crystal size	0.3 x 0.25 x 0.2 mm ³	
Theta range for data collection	1.368 to 26.373°.	
Index ranges	$-6 \leq h \leq 10$, $-11 \leq k \leq 11$, $-19 \leq l \leq 19$	
Reflections collected	11968	

Independent reflections	6275 [R(int) = 0.0352]
Completeness to theta = 26.000°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.0932 and 0.0596
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	6275 / 3 / 685
Goodness-of-fit on F ²	1.040
Final R indices [I>2sigma(I)]	R1 = 0.0419, wR2 = 0.1199
R indices (all data)	R1 = 0.0526, wR2 = 0.1347
Absolute structure parameter	0.15(5)
Extinction coefficient	n/a
Largest diff. peak and hole	0.390 and -0.379 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2c**.
 $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(2)	11029(2)	-1359(2)	9375(1)	50(1)
Cl(1)	-751(2)	11527(2)	636(1)	55(1)
F(9)	6400(4)	378(3)	5951(2)	40(1)
F(12)	2588(4)	4621(4)	3527(2)	47(1)
F(10)	616(5)	1261(4)	5824(2)	51(1)
F(13)	4836(4)	3678(4)	4559(2)	42(1)
F(5)	5349(4)	6778(3)	5493(2)	43(1)
F(4)	7391(4)	5658(3)	6548(2)	47(1)
F(11)	-1676(4)	2092(4)	4758(3)	60(1)
F(1)	4103(4)	10014(4)	4079(2)	49(1)
F(3)	11842(4)	7694(4)	5272(2)	58(1)
F(2)	9777(5)	8708(4)	4211(2)	51(1)
O(6)	4056(5)	500(4)	8204(2)	43(1)
O(5)	8432(6)	4764(4)	8442(3)	53(1)
N(3)	6248(5)	2497(4)	8113(2)	29(1)
O(1)	5977(6)	9957(4)	1445(3)	53(1)
O(4)	3720(6)	3226(5)	7110(2)	53(1)
F(6)	10115(6)	4597(5)	6605(3)	75(1)
O(3)	6624(6)	6981(5)	2942(2)	54(1)
N(1)	4376(5)	7972(4)	1900(3)	30(1)
N(4)	4048(6)	2050(5)	5778(3)	38(1)
O(2)	2580(7)	5648(4)	1931(3)	60(1)
F(15)	-2219(6)	3980(7)	3786(4)	101(2)
F(14)	-322(6)	5223(6)	3343(3)	89(2)
F(8)	12237(6)	5970(6)	6345(3)	80(1)
C(2)	4360(6)	7444(5)	413(3)	31(1)
N(2)	6365(6)	8293(5)	4255(3)	36(1)
C(32)	7415(7)	3874(5)	8680(3)	35(1)
F(7)	11249(8)	6913(6)	7402(3)	116(2)
C(15)	678(7)	8811(6)	2154(4)	40(1)
C(10)	2337(7)	9677(5)	2620(3)	33(1)
C(31)	7067(7)	3933(6)	9573(3)	33(1)
C(25)	5173(7)	1695(6)	8556(3)	32(1)

C(22)	8020(7)	6424(5)	5977(3)	34(1)
C(6)	2484(8)	4810(6)	-132(3)	46(1)
C(3)	4648(7)	7453(7)	-421(4)	40(1)
C(23)	6952(6)	6981(5)	5431(3)	32(1)
C(1)	5044(7)	8643(6)	1270(3)	34(1)
C(35)	7281(6)	-90(5)	7393(3)	31(1)
C(45)	367(7)	3405(6)	4104(3)	34(1)
C(44)	-61(7)	2548(6)	4707(4)	38(1)
C(33)	6029(7)	2100(5)	7155(3)	32(1)
C(13)	420(7)	10824(6)	1422(3)	38(1)
C(46)	2051(7)	3785(5)	4080(3)	33(1)
C(43)	1124(7)	2119(5)	5267(3)	35(1)
C(7)	3302(7)	6132(6)	553(3)	37(1)
C(26)	5744(6)	2636(6)	9511(3)	32(1)
C(18)	7476(7)	7726(5)	4810(3)	31(1)
C(39)	7891(7)	-1722(6)	8367(4)	39(1)
F(16)	-1473(9)	2852(7)	2709(4)	161(4)
C(48)	-935(8)	3849(7)	3471(4)	50(2)
C(40)	6747(6)	-1316(6)	7766(4)	37(1)
C(38)	9589(7)	-897(6)	8592(3)	38(1)
C(19)	9145(7)	7937(6)	4772(3)	36(1)
C(42)	2799(7)	2489(5)	5234(3)	32(1)
C(21)	9688(7)	6611(6)	5942(3)	34(1)
C(16)	6030(7)	7878(6)	3333(3)	37(1)
C(29)	7213(9)	4829(7)	11113(4)	50(2)
C(47)	3228(6)	3306(6)	4619(3)	31(1)
C(11)	2994(7)	11134(6)	2483(3)	38(1)
C(41)	4470(7)	2519(6)	6696(3)	38(1)
C(28)	5915(8)	3523(7)	11050(3)	48(1)
C(34)	6007(6)	401(5)	6792(3)	31(1)
C(12)	2038(7)	11727(5)	1895(3)	36(1)
C(8)	4906(7)	8743(6)	2859(3)	34(1)
C(9)	3408(7)	8980(6)	3209(3)	38(1)
C(14)	-272(7)	9392(6)	1558(4)	47(1)
C(01W)	3308(8)	6467(5)	1522(3)	36(1)
C(37)	10162(7)	300(6)	8207(4)	44(1)
C(20)	10236(7)	7410(6)	5335(3)	38(1)
C(4)	3846(8)	6111(7)	-1102(4)	47(1)

C(27)	5141(7)	2386(7)	10239(4)	43(1)
C(5)	2788(8)	4833(7)	-973(4)	46(1)
C(36)	9004(7)	704(6)	7608(4)	40(1)
C(24)	10847(9)	6072(8)	6589(4)	54(2)
C(30)	7828(8)	5046(6)	10380(3)	43(1)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for **2c**.

Cl(2)-C(38)	1.736(5)
Cl(1)-C(13)	1.742(5)
F(9)-C(34)	1.422(5)
F(12)-C(46)	1.335(6)
F(10)-C(43)	1.345(6)
F(13)-C(47)	1.339(6)
F(5)-C(23)	1.342(6)
F(4)-C(22)	1.348(6)
F(11)-C(44)	1.343(6)
F(1)-C(9)	1.421(5)
F(3)-C(20)	1.339(7)
F(2)-C(19)	1.340(6)
O(6)-C(25)	1.200(6)
O(5)-C(32)	1.198(7)
N(3)-C(32)	1.400(6)
N(3)-C(25)	1.399(7)
N(3)-C(33)	1.440(6)
O(1)-C(1)	1.209(6)
O(4)-C(41)	1.203(7)
F(6)-C(24)	1.345(8)
O(3)-C(16)	1.198(7)
N(1)-C(1)	1.397(6)
N(1)-C(8)	1.462(6)
N(1)-C(01W)	1.387(6)
N(4)-H(4)	0.8800
N(4)-C(42)	1.405(6)
N(4)-C(41)	1.368(6)
O(2)-C(01W)	1.210(6)
F(15)-C(48)	1.297(8)
F(14)-C(48)	1.306(8)
F(8)-C(24)	1.320(8)
C(2)-C(3)	1.379(7)
C(2)-C(1)	1.471(7)
C(2)-C(7)	1.391(7)
N(2)-H(2)	0.8800
N(2)-C(18)	1.411(6)

N(2)-C(16)	1.376(6)
C(32)-C(31)	1.482(7)
F(7)-C(24)	1.289(7)
C(15)-H(15)	0.9500
C(15)-C(10)	1.392(7)
C(15)-C(14)	1.377(8)
C(10)-C(11)	1.387(7)
C(10)-C(9)	1.499(7)
C(31)-C(26)	1.385(7)
C(31)-C(30)	1.384(7)
C(25)-C(26)	1.496(7)
C(22)-C(23)	1.374(7)
C(22)-C(21)	1.386(8)
C(6)-H(6)	0.9500
C(6)-C(7)	1.375(7)
C(6)-C(5)	1.398(8)
C(3)-H(3)	0.9500
C(3)-C(4)	1.383(8)
C(23)-C(18)	1.372(7)
C(35)-C(40)	1.381(7)
C(35)-C(34)	1.495(7)
C(35)-C(36)	1.391(7)
C(45)-C(44)	1.387(8)
C(45)-C(46)	1.383(8)
C(45)-C(48)	1.514(8)
C(44)-C(43)	1.377(8)
C(33)-H(33)	1.0000
C(33)-C(41)	1.537(7)
C(33)-C(34)	1.545(6)
C(13)-C(12)	1.377(7)
C(13)-C(14)	1.372(8)
C(46)-C(47)	1.381(7)
C(43)-C(42)	1.380(8)
C(7)-C(01W)	1.492(7)
C(26)-C(27)	1.375(8)
C(18)-C(19)	1.385(8)
C(39)-H(39)	0.9500
C(39)-C(40)	1.380(7)

C(39)-C(38)	1.378(7)
F(16)-C(48)	1.277(7)
C(40)-H(40)	0.9500
C(38)-C(37)	1.380(8)
C(19)-C(20)	1.383(8)
C(42)-C(47)	1.380(7)
C(21)-C(20)	1.389(7)
C(21)-C(24)	1.503(8)
C(16)-C(8)	1.545(7)
C(29)-H(29)	0.9500
C(29)-C(28)	1.378(9)
C(29)-C(30)	1.379(8)
C(11)-H(11)	0.9500
C(11)-C(12)	1.380(7)
C(28)-H(28)	0.9500
C(28)-C(27)	1.400(8)
C(34)-H(34)	1.0000
C(12)-H(12)	0.9500
C(8)-H(8)	1.0000
C(8)-C(9)	1.518(8)
C(9)-H(9)	1.0000
C(14)-H(14)	0.9500
C(37)-H(37)	0.9500
C(37)-C(36)	1.384(8)
C(4)-H(4A)	0.9500
C(4)-C(5)	1.360(9)
C(27)-H(27)	0.9500
C(5)-H(5)	0.9500
C(36)-H(36)	0.9500
C(30)-H(30)	0.9500

C(32)-N(3)-C(33)	122.5(4)
C(25)-N(3)-C(32)	112.6(4)
C(25)-N(3)-C(33)	124.3(4)
C(1)-N(1)-C(8)	122.4(4)
C(01W)-N(1)-C(1)	112.4(4)
C(01W)-N(1)-C(8)	124.9(4)
C(42)-N(4)-H(4)	118.8

C(41)-N(4)-H(4)	118.8
C(41)-N(4)-C(42)	122.4(4)
C(3)-C(2)-C(1)	130.7(5)
C(3)-C(2)-C(7)	120.8(5)
C(7)-C(2)-C(1)	108.4(4)
C(18)-N(2)-H(2)	118.9
C(16)-N(2)-H(2)	118.9
C(16)-N(2)-C(18)	122.2(4)
O(5)-C(32)-N(3)	124.2(5)
O(5)-C(32)-C(31)	130.7(5)
N(3)-C(32)-C(31)	105.1(4)
C(10)-C(15)-H(15)	120.0
C(14)-C(15)-H(15)	120.0
C(14)-C(15)-C(10)	120.1(5)
C(15)-C(10)-C(9)	119.7(4)
C(11)-C(10)-C(15)	118.8(5)
C(11)-C(10)-C(9)	121.4(5)
C(26)-C(31)-C(32)	109.2(4)
C(30)-C(31)-C(32)	130.0(5)
C(30)-C(31)-C(26)	120.7(5)
O(6)-C(25)-N(3)	124.6(4)
O(6)-C(25)-C(26)	130.0(5)
N(3)-C(25)-C(26)	105.4(4)
F(4)-C(22)-C(23)	118.0(5)
F(4)-C(22)-C(21)	120.3(5)
C(23)-C(22)-C(21)	121.7(5)
C(7)-C(6)-H(6)	121.5
C(7)-C(6)-C(5)	117.0(5)
C(5)-C(6)-H(6)	121.5
C(2)-C(3)-H(3)	121.4
C(2)-C(3)-C(4)	117.2(5)
C(4)-C(3)-H(3)	121.4
F(5)-C(23)-C(22)	119.5(5)
F(5)-C(23)-C(18)	118.8(5)
C(18)-C(23)-C(22)	121.6(5)
O(1)-C(1)-N(1)	123.8(5)
O(1)-C(1)-C(2)	130.3(5)
N(1)-C(1)-C(2)	105.8(4)

C(40)-C(35)-C(34)	119.9(4)
C(40)-C(35)-C(36)	119.2(5)
C(36)-C(35)-C(34)	120.8(4)
C(44)-C(45)-C(48)	122.4(5)
C(46)-C(45)-C(44)	116.4(5)
C(46)-C(45)-C(48)	121.1(5)
F(11)-C(44)-C(45)	120.4(5)
F(11)-C(44)-C(43)	117.5(5)
C(43)-C(44)-C(45)	122.1(5)
N(3)-C(33)-H(33)	106.8
N(3)-C(33)-C(41)	109.1(4)
N(3)-C(33)-C(34)	112.2(4)
C(41)-C(33)-H(33)	106.8
C(41)-C(33)-C(34)	114.6(4)
C(34)-C(33)-H(33)	106.8
C(12)-C(13)-Cl(1)	118.8(4)
C(14)-C(13)-Cl(1)	120.0(4)
C(14)-C(13)-C(12)	121.2(5)
F(12)-C(46)-C(45)	120.8(5)
F(12)-C(46)-C(47)	117.8(5)
C(47)-C(46)-C(45)	121.5(5)
F(10)-C(43)-C(44)	119.0(5)
F(10)-C(43)-C(42)	119.7(5)
C(44)-C(43)-C(42)	121.2(5)
C(2)-C(7)-C(01W)	107.8(4)
C(6)-C(7)-C(2)	121.6(5)
C(6)-C(7)-C(01W)	130.5(5)
C(31)-C(26)-C(25)	107.7(4)
C(27)-C(26)-C(31)	122.1(5)
C(27)-C(26)-C(25)	130.2(5)
C(23)-C(18)-N(2)	120.9(5)
C(23)-C(18)-C(19)	117.4(5)
C(19)-C(18)-N(2)	121.6(5)
C(40)-C(39)-H(39)	120.3
C(38)-C(39)-H(39)	120.3
C(38)-C(39)-C(40)	119.3(5)
F(15)-C(48)-F(14)	103.4(6)
F(15)-C(48)-C(45)	112.8(5)

F(14)-C(48)-C(45)	112.1(5)
F(16)-C(48)-F(15)	109.1(7)
F(16)-C(48)-F(14)	108.5(6)
F(16)-C(48)-C(45)	110.6(5)
C(35)-C(40)-H(40)	119.6
C(39)-C(40)-C(35)	120.8(5)
C(39)-C(40)-H(40)	119.6
C(39)-C(38)-Cl(2)	119.7(4)
C(39)-C(38)-C(37)	121.0(5)
C(37)-C(38)-Cl(2)	119.3(4)
F(2)-C(19)-C(18)	121.2(5)
F(2)-C(19)-C(20)	117.6(5)
C(20)-C(19)-C(18)	121.1(5)
C(43)-C(42)-N(4)	123.3(5)
C(43)-C(42)-C(47)	117.1(5)
C(47)-C(42)-N(4)	119.6(5)
C(22)-C(21)-C(20)	116.7(5)
C(22)-C(21)-C(24)	120.1(5)
C(20)-C(21)-C(24)	123.0(6)
O(3)-C(16)-N(2)	122.7(5)
O(3)-C(16)-C(8)	123.5(5)
N(2)-C(16)-C(8)	113.7(4)
C(28)-C(29)-H(29)	119.4
C(28)-C(29)-C(30)	121.1(5)
C(30)-C(29)-H(29)	119.4
F(13)-C(47)-C(46)	119.2(4)
F(13)-C(47)-C(42)	119.0(5)
C(42)-C(47)-C(46)	121.7(5)
C(10)-C(11)-H(11)	119.4
C(12)-C(11)-C(10)	121.2(5)
C(12)-C(11)-H(11)	119.4
O(4)-C(41)-N(4)	123.4(5)
O(4)-C(41)-C(33)	122.7(4)
N(4)-C(41)-C(33)	113.9(4)
C(29)-C(28)-H(28)	119.2
C(29)-C(28)-C(27)	121.5(5)
C(27)-C(28)-H(28)	119.2
F(9)-C(34)-C(35)	110.0(4)

F(9)-C(34)-C(33)	104.4(3)
F(9)-C(34)-H(34)	109.7
C(35)-C(34)-C(33)	113.1(4)
C(35)-C(34)-H(34)	109.7
C(33)-C(34)-H(34)	109.7
C(13)-C(12)-C(11)	118.7(5)
C(13)-C(12)-H(12)	120.7
C(11)-C(12)-H(12)	120.7
N(1)-C(8)-C(16)	107.6(4)
N(1)-C(8)-H(8)	106.2
N(1)-C(8)-C(9)	111.3(4)
C(16)-C(8)-H(8)	106.2
C(9)-C(8)-C(16)	118.5(4)
C(9)-C(8)-H(8)	106.2
F(1)-C(9)-C(10)	109.6(4)
F(1)-C(9)-C(8)	106.3(4)
F(1)-C(9)-H(9)	109.6
C(10)-C(9)-C(8)	112.0(4)
C(10)-C(9)-H(9)	109.6
C(8)-C(9)-H(9)	109.6
C(15)-C(14)-H(14)	120.0
C(13)-C(14)-C(15)	119.9(5)
C(13)-C(14)-H(14)	120.0
N(1)-C(01W)-C(7)	105.5(4)
O(2)-C(01W)-N(1)	124.7(5)
O(2)-C(01W)-C(7)	129.8(5)
C(38)-C(37)-H(37)	120.4
C(38)-C(37)-C(36)	119.2(5)
C(36)-C(37)-H(37)	120.4
F(3)-C(20)-C(19)	117.5(5)
F(3)-C(20)-C(21)	121.1(5)
C(19)-C(20)-C(21)	121.4(5)
C(3)-C(4)-H(4A)	118.9
C(5)-C(4)-C(3)	122.2(5)
C(5)-C(4)-H(4A)	118.9
C(26)-C(27)-C(28)	116.7(5)
C(26)-C(27)-H(27)	121.7
C(28)-C(27)-H(27)	121.7

C(6)-C(5)-H(5)	119.5
C(4)-C(5)-C(6)	121.1(5)
C(4)-C(5)-H(5)	119.5
C(35)-C(36)-H(36)	119.8
C(37)-C(36)-C(35)	120.4(5)
C(37)-C(36)-H(36)	119.8
F(6)-C(24)-C(21)	111.0(5)
F(8)-C(24)-F(6)	102.4(5)
F(8)-C(24)-C(21)	113.2(6)
F(7)-C(24)-F(6)	107.0(6)
F(7)-C(24)-F(8)	109.7(6)
F(7)-C(24)-C(21)	112.9(5)
C(31)-C(30)-H(30)	121.0
C(29)-C(30)-C(31)	117.9(6)
C(29)-C(30)-H(30)	121.0

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2c**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2}U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(2)	43(1)	58(1)	50(1)	14(1)	-1(1)	25(1)
Cl(1)	52(1)	69(1)	56(1)	30(1)	9(1)	32(1)
F(9)	57(2)	49(2)	23(1)	5(1)	12(1)	31(2)
F(12)	56(2)	52(2)	41(2)	26(1)	15(2)	15(2)
F(10)	65(2)	50(2)	43(2)	22(1)	21(2)	11(2)
F(13)	33(2)	51(2)	44(2)	12(1)	13(1)	14(1)
F(5)	36(2)	52(2)	44(2)	12(1)	14(1)	18(1)
F(4)	59(2)	50(2)	46(2)	27(2)	21(2)	25(2)
F(11)	34(2)	75(2)	67(2)	18(2)	17(2)	5(2)
F(1)	64(2)	55(2)	28(2)	1(1)	6(2)	33(2)
F(3)	36(2)	80(2)	60(2)	20(2)	13(2)	15(2)
F(2)	62(2)	55(2)	42(2)	21(2)	23(2)	15(2)
O(6)	33(2)	50(2)	37(2)	2(2)	9(2)	1(2)
O(5)	71(3)	37(2)	37(2)	5(2)	14(2)	-6(2)
N(3)	31(2)	31(2)	23(2)	3(2)	7(2)	9(2)
O(1)	61(3)	42(2)	44(2)	6(2)	18(2)	-2(2)
O(4)	70(3)	69(3)	28(2)	0(2)	8(2)	51(2)
F(6)	91(3)	80(3)	82(3)	48(2)	28(2)	50(2)
O(3)	76(3)	69(3)	28(2)	3(2)	7(2)	50(2)
N(1)	39(2)	31(2)	21(2)	7(2)	7(2)	12(2)
N(4)	53(3)	43(2)	24(2)	7(2)	8(2)	30(2)
O(2)	99(4)	40(2)	39(2)	15(2)	28(2)	5(2)
F(15)	60(3)	145(4)	137(4)	79(4)	33(3)	62(3)
F(14)	81(3)	100(3)	107(4)	68(3)	11(3)	42(3)
F(8)	61(3)	105(3)	99(3)	47(3)	18(2)	53(3)
C(2)	31(2)	36(2)	32(2)	12(2)	7(2)	20(2)
N(2)	51(3)	39(2)	24(2)	2(2)	5(2)	29(2)
C(32)	45(3)	30(2)	30(2)	9(2)	8(2)	13(2)
F(7)	165(5)	134(4)	42(2)	-22(2)	-37(3)	115(4)
C(15)	37(3)	36(2)	49(3)	16(2)	10(2)	9(2)
C(10)	42(3)	36(2)	29(2)	6(2)	12(2)	22(2)
C(31)	39(3)	38(2)	23(2)	4(2)	2(2)	19(2)
C(25)	33(3)	40(3)	25(2)	6(2)	6(2)	15(2)

C(22)	42(3)	30(2)	28(2)	7(2)	8(2)	11(2)
C(6)	58(4)	40(3)	35(3)	3(2)	8(3)	11(3)
C(3)	43(3)	51(3)	36(3)	15(2)	15(2)	21(3)
C(23)	29(2)	34(2)	32(2)	0(2)	9(2)	13(2)
C(1)	35(3)	42(3)	32(2)	14(2)	9(2)	20(2)
C(35)	31(2)	32(2)	28(2)	4(2)	7(2)	11(2)
C(45)	33(3)	39(3)	23(2)	1(2)	1(2)	9(2)
C(44)	30(3)	37(3)	39(3)	1(2)	4(2)	10(2)
C(33)	40(3)	36(2)	21(2)	4(2)	7(2)	16(2)
C(13)	40(3)	43(3)	36(3)	13(2)	8(2)	23(2)
C(46)	45(3)	32(2)	24(2)	7(2)	9(2)	13(2)
C(43)	46(3)	30(2)	30(2)	9(2)	16(2)	6(2)
C(7)	45(3)	39(3)	30(2)	10(2)	8(2)	18(2)
C(26)	32(2)	43(3)	24(2)	9(2)	6(2)	21(2)
C(18)	39(3)	31(2)	22(2)	3(2)	2(2)	15(2)
C(39)	35(3)	35(2)	45(3)	16(2)	5(2)	9(2)
F(16)	211(7)	138(5)	78(3)	-60(3)	-97(4)	129(5)
C(48)	47(4)	59(4)	41(3)	5(3)	-1(3)	28(3)
C(40)	27(2)	38(3)	47(3)	11(2)	7(2)	11(2)
C(38)	35(3)	42(3)	40(3)	9(2)	8(2)	21(2)
C(19)	45(3)	31(2)	31(3)	8(2)	10(2)	7(2)
C(42)	42(3)	28(2)	26(2)	2(2)	7(2)	16(2)
C(21)	38(3)	35(2)	29(2)	3(2)	2(2)	17(2)
C(16)	47(3)	44(3)	22(2)	5(2)	6(2)	21(3)
C(29)	63(4)	55(3)	28(3)	-1(2)	1(3)	25(3)
C(47)	29(3)	35(2)	25(2)	5(2)	6(2)	8(2)
C(11)	41(3)	35(2)	34(2)	7(2)	7(2)	11(2)
C(41)	47(3)	37(3)	29(2)	8(2)	3(2)	18(2)
C(28)	52(3)	74(4)	26(3)	13(3)	16(3)	29(3)
C(34)	33(2)	37(2)	25(2)	7(2)	11(2)	14(2)
C(12)	39(3)	31(2)	39(3)	10(2)	10(2)	11(2)
C(8)	46(3)	34(2)	22(2)	5(2)	8(2)	15(2)
C(9)	52(3)	38(3)	25(2)	5(2)	10(2)	20(2)
C(14)	36(3)	46(3)	53(3)	15(3)	3(3)	10(3)
C(01W)	52(3)	30(2)	29(2)	8(2)	12(2)	15(2)
C(37)	28(3)	47(3)	57(3)	17(3)	9(3)	11(2)
C(20)	35(3)	40(3)	37(3)	4(2)	9(2)	14(2)
C(4)	60(4)	66(4)	27(3)	14(3)	13(3)	39(3)

C(27)	35(3)	57(3)	38(3)	12(2)	6(2)	16(3)
C(5)	52(3)	47(3)	32(3)	-2(2)	-1(3)	19(3)
C(36)	34(3)	44(3)	52(3)	22(2)	16(2)	15(2)
C(24)	59(4)	57(4)	40(3)	2(3)	-4(3)	30(3)
C(30)	52(3)	42(3)	31(3)	1(2)	5(2)	16(3)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for **2c**.

	x	y	z	U(eq)
H(4)	4578	1450	5518	45
H(2)	5869	8935	4507	44
H(15)	201	7817	2247	48
H(6)	1745	3922	-37	55
H(3)	5367	8345	-522	49
H(33)	7042	2787	7036	38
H(39)	7514	-2563	8623	47
H(40)	5577	-1885	7606	45
H(29)	7694	5591	11670	60
H(11)	4124	11735	2800	45
H(28)	5536	3393	11570	57
H(34)	4845	-319	6698	37
H(12)	2486	12739	1818	43
H(8)	5668	9807	2922	40
H(9)	2697	7961	3254	45
H(14)	-1406	8802	1242	56
H(37)	11339	841	8352	52
H(4A)	4041	6081	-1680	56
H(27)	4244	1486	10194	52
H(5)	2248	3941	-1463	56
H(36)	9387	1528	7341	49
H(30)	8743	5932	10428	52