Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Additive-free regio- and diastereoselective construction of fully-substituted isoxazolidines employing diazo compounds

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SUPPORTING INFORMATION

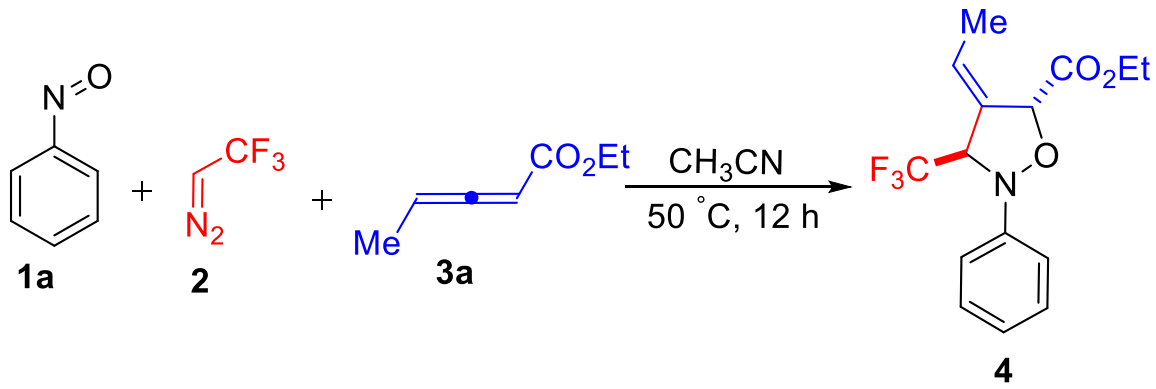
Content:

General experimental information

Unless otherwise specified, all reactions were performed in oven-dried glasswares under nitrogenous atmosphere using dry deoxygenated solvent. The reactions were monitored by TLC visualized by UV (254 nm) and/or with iodine. Column chromatography was performed on 100-200 mesh silica gel using the gradient system ethyl acetate-hexane. NMR data were recorded at Bruker AV 400 MHz in $CDCl_3$/DMSO-d_6 using as internal standards the residual $CHCl_3$ signal for ^1H NMR (δ = 7.26 ppm) and the deuterated solvent signal for ^{13}C NMR (δ = 77.16 ppm). The residual DMSO signal for ^1H NMR (δ = 2.50 ppm) and the deuterated solvent signal for ^{13}C NMR (δ = 39.51 ppm). Coupling constants are given in Hertz (Hz) and the classical abbreviations are used to describe the signal multiplicities. Melting points were measured with a Büchi B-540 apparatus and are uncorrected. High resolution mass spectra were obtained using Q-TOF mass spectrometer. All commercially available reagents were used as received. All allenic esters **(3a-3m)**[1] and nitroarenes[2] were synthesized following literature procedure. Stock solution of the CF_3CHN_2 and Seyferth-Gilbert reagent were preapared according to the literature procedure.[3,4]

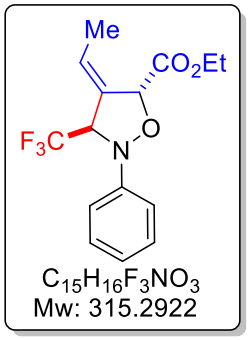
1. L. Rout and A. M. Harned, *Chem. Eur. J.,* 2009, **15**, 12926.
2. S. Chakrabarty, I. Chatterjee, L. Tebben and A. Studer, *Angew. Chem., Int. Ed.,* 2013, **52**, 2968.
3. G. A. Molander and D. Ryu, *Angew. Chem., Int. Ed.,* 2014, **53**, 14181.
4. A. K. Gupta, N. K. Vaishanv, R. Kant and K. Mohanan, *Org. Biomol. Chem.,* 2017, **15,** 6411**.**

General procedure for the synthesis of trifluoromethylated isoxazolidines 4a-4r



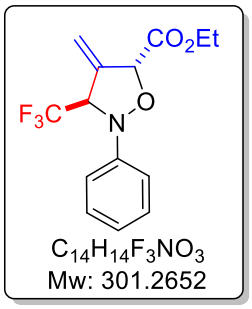
A 10 mL round-bottom flask charged with nitrosobenzene **1a** (54 mg, 0.50 mmol, 1.0 equiv) was sealed, evacuated, backfilled with nitrogen and added dry CH_3CN (2 mL). Subsequently, the requisite amount of CF_3CHN_2 in toluene **2** (1.90 mL, 1.5 mmol) and ethyl penta-2,3-dienoate **3a** (95 mg, 0.75 mmol) were added via a syringe. This reaction mixture was stirred at 50 °C for 12 h. After the completion of reaction, as indicated by TLC, solvent was evaporated under reduced pressure. The residue was purified using column chromatography (100-200 mesh silica gel) using ethyl acetate/hexane as the eluent.

Compound 4a: Ethyl 4-ethylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



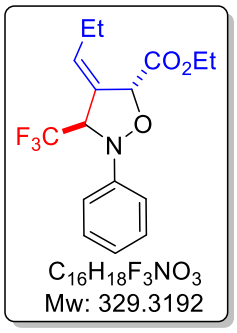
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.5 mmol) and ethyl penta-2,3-dienoate **3a** (95 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4a** as liquid (113 mg, 72%). $\mathbf{R_f}$ (EtOAc/Hexane: 1/9) = 0.40. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.5 (C), 149.6 (C), 133.2 (C), 128.9 (CH), 128.9 (CH), 125.7 (CH), 122.6 (CH), 120.6 (q, $J_{C\text{-}F}$ = 278.6 Hz, C), 114.2 (CH), 114.2 (CH), 78.6 (CH), 67.7 (q, $J_{C\text{-}F}$ = 31.9 Hz, CH), 61.9 (CH_2), 16.3 (CH_3), 13.6 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.27-7.23 (m, 2H), 7.04 (d, J = 7.6 Hz, 2H), 6.97 (t, J = 7.4 Hz, 1H), 6.08 (q, J = 6.8 Hz, 1H), 5.17 (s, 1H), 4.86-4.81 (m, 1H), 4.01-3.87 (m, 2H), 1.72 (d, J = 7.2 Hz, 3H), 1.01 (t, J = 7.2 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.6 (s). **HRMS** for $C_{15}H_{17}F_3NO_3^+$: calcd. $[M+H]^+$: 316.1155, found: 316.1154.

Compound 4b: Ethyl 4-methylene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



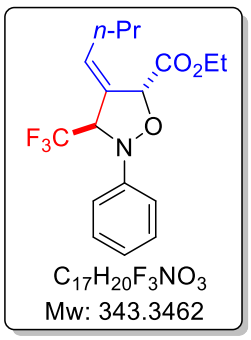
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl buta-2,3-dienoate **3b** (84 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4b** as liquid (90 mg, 60%). $\mathbf{R_f}$ (EtOAc/Hexane: 1/9) = 0.41. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.4 (C), 149.5 (C), 140.3 (C), 128.9 (CH), 128.9 (CH), 124.0 (q, $J_{C\text{-}F}$ = 278.9 Hz, C), 123.4 (CH), 115.7 (CH), 115.7 (CH), 114.7 (CH_2), 79.1 (CH), 69.1 (q, $J_{C\text{-}F}$ = 31.9 Hz, CH), 62.1 (CH_2), 13.9 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.31-7.27 (m, 2H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.03 (t, *J* = 7.2 Hz, 1H), 5.69 (s, 1H), 5.64 (s, 1H), 5.15-5.14 (m, 1H), 4.79-4.34 (m, 1H), 4.11 (q, $J_{H\text{-}F}$ = 7.2 Hz, 2H), 1.17 (t, *J* = 7.0 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.8 (s). **HRMS** for $C_{14}H_{15}F_3NO_3^+$: calcd. $[M+H]^+$: 302.0999, found: 302.1001.

Compound 4c: Ethyl 2-phenyl-4-propylidene-3-(trifluoromethyl)isoxazolidine-5-carboxylate



Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl hexa-2,3-dienoate **3c** (105 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4c** as liquid (107 mg, 65%). $\mathbf{R_f}$ (EtOAc/Hexane: 1/9) = 0.51. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.7 (C), 149.7 (C), 132.4 (C), 131.5 (CH), 128.9 (CH), 128.9 (CH), 124.3 (q, $J_{C\text{-}F}$ = 287.7 Hz, C), 122.5 (CH), 114.0 (CH), 114.0 (CH), 78.5 (CH), 67.6 (q, $J_{C\text{-}F}$ = 31.8 Hz, CH), 61.9 (CH_2), 24.5 (CH_2), 13.6 (CH_3), 13.2 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.28-7.24 (m, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.98 (t, *J* = 7.4 Hz, 1H), 5.98 (t, *J* = 7.4 Hz, 1H), 5.16 (s, 1H), 4.84 (q, *J* = 7.2 Hz, 1H), 3.99-3.84 (m, 2H), 2.11-2.03 (m, 2H), 1.04-0.97 (m, 6H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.6 (s). **HRMS** for $C_{16}H_{19}F_3NO_3^+$: calcd. $[M+H]^+$: 330.1312, found: 330.1307.

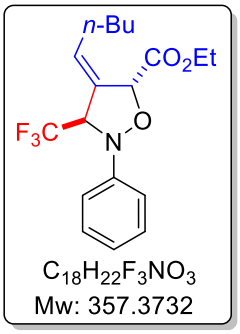
Compound 4d: Ethyl 4-butylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.20 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl hepta-2,3-dienoate **3d** (116 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4d** as liquid (106 mg, 62%). $\mathbf{R_f}$ (EtOAc/Hexane: 1/9) = 0.56. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.7 (C), 149.7 (C), 132.3 (C), 130.9 (CH), 128.9 (CH), 128.9 (CH), 124.4 (q, $J_{C\text{-}F}$ = 278.5 Hz, C), 122.5 (CH), 114.0 (CH), 114.0 (CH), 78.7 (CH), 67.6 (q, $J_{C\text{-}F}$ = 31.8 Hz, CH), 61.8 (CH_2), 33.0 (CH_2), 22.0 (CH_2), 13.7 (CH_3), 13.6 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.27-7.23 (m, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.97 (t, *J* = 7.4 Hz, 1H), 5.98 (t, *J* = 7.4 Hz, 1H), 5.15 (s, 1H), 4.84 (q, *J* = 7.2 Hz, 1H),

3.98-3.83 (m, 2H), 2.07-2.00 (m, 2H), 1.47-1.42 (m, 2H), 0.97 (t, *J* = 7.2 Hz, 3H), 0.88 (t, *J* = 7.2 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.6 (s). **HRMS** for $C_{17}H_{21}F_3NO_3^+$: calcd. $[M+H]^+$: 344.1468, found: 344.1465.

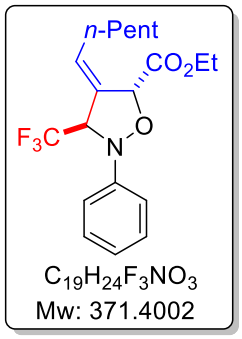
Compound 4e: Ethyl 4-pentylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



$C_{18}H_{22}F_3NO_3$
Mw: 357.3732

Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl octa-2,3-dienoate **3e** (126 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4e** as liquid (109 mg, 61%). **R_f** (EtOAc/Hexane: 1/9) = 0.59. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.7 (C), 149.7 (C), 132.1 (C), 131.0 (CH), 128.9 (CH), 128.9 (CH), 124.4 (q, J_{C-F} = 278.6 Hz, C), 122.5 (CH), 114.0 (CH), 114.0 (CH), 78.7 (CH), 67.6 (q, J_{C-F} = 31.8 Hz, CH), 61.8 (CH_2), 30.8 (CH_2), 30.8 (CH_2), 22.3 (CH_2), 13.9 (CH_3), 13.6 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.28-7.24 (m, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.97 (t, *J* = 7.2 Hz, 1H), 5.98 (t, *J* = 7.4 Hz, 1H), 5.15 (s, 1H), 4.84 (q, *J* = 7.2 Hz, 1H), 4.00-3.83 (m, 2H), 2.08-2.03 (m, 2H), 1.42-1.31 (m, 4H), 0.97 (t, *J* = 7.0 Hz, 3H), 0.89 (t, *J* = 7.2 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.6 (s). **HRMS** for $C_{18}H_{23}F_3NO_3^+$: calcd. $[M+H]^+$: 358.1625, found: 358.1623.

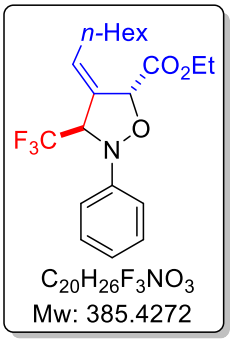
Compound 4f: Ethyl 4-hexylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



$C_{19}H_{24}F_3NO_3$
Mw: 371.4002

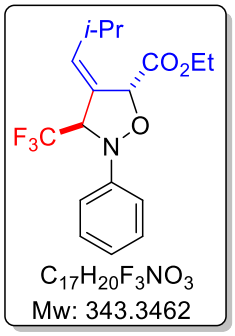
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.9 mL, 1.50 mmol) and ethyl nona-2,3-dienoate **3f** (137 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4f** as liquid (122 mg, 66%). **R_f** (EtOAc/Hexane: 1/9) = 0.67. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.7 (C), 149.8 (C), 132.1 (C), 131.1 (CH), 128.9 (CH), 128.9 (CH), 124.4 (q, J_{C-F} = 278.6 Hz, C), 122.5 (CH), 114.0 (CH), 114.0 (CH), 78.7 (CH), 67.6 (q, J_{C-F} = 31.8 Hz, CH), 61.8 (CH_2), 31.4 (CH_2), 31.1 (CH_2), 28.4 (CH_2), 22.5 (CH_2), 14.1 (CH_3), 13.6 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.28-7.24 (m, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.97 (t, *J* = 7.4 Hz, 1H), 5.98 (t, *J* = 7.4 Hz, 1H), 5.15 (s, 1H), 4.84 (q, *J* = 7.2 Hz, 1H), 3.98-3.83 (m, 2H), 2.07-2.02 (m, 2H), 1.43-1.39 (m, 2H), 1.30-1.23 (m, 4H), 0.97 (t, *J* = 7.2 Hz, 3H), 0.90 (t, *J* = 6.8 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.6 (s). **HRMS** for $C_{19}H_{25}F_3NO_3^+$: calcd. $[M+H]^+$: 372.1781, found: 372.1785.

Compound 4g: Ethyl 4-heptylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



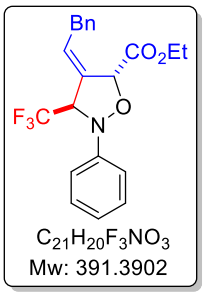
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl deca-2,3-dienoate **3g** (147 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4g** as liquid (117 mg, 61%). $\mathbf{R_f}$ (EtOAc/Hexane: 1/9) = 0.65. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.7 (C), 149.8 (C), 132.1 (C), 131.1 (CH), 128.9 (CH), 128.9 (CH), 124.4 (q, J_{C-F} = 274.5 Hz, C), 122.5 (CH), 114.0 (CH), 114.0 (CH), 78.7 (CH), 67.6 (q, J_{C-F} = 31.6 Hz, CH), 61.8 (CH_2), 31.7 (CH_2), 31.1 (CH_2), 28.9 (CH_2) 28.7 (CH_2), 22.6 (CH_2), 14.1 (CH_3), 13.6 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.27-7.23 (m, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.97 (t, *J* = 7.2 Hz, 1H), 5.97 (t, *J* = 7.4 Hz, 1H), 5.15 (s, 1H), 4.84 (q, *J* = 7.2 Hz, 1H), 3.96-3.84 (m, 2H), 2.12-2.02 (m, 2H), 1.42-1.37 (m, 2H), 1.31-1.23 (m, 6H), 0.97 (t, *J* = 7.0 Hz, 3H), 0.87 (t, *J* = 6.6 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.6 (s). **HRMS** for $C_{20}H_{27}F_3NO_3^+$: calcd. $[M+H]^+$: 386.1938, found: 386.1938.

Compound 4h: Ethyl 4-(2-methylpropylidene)-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



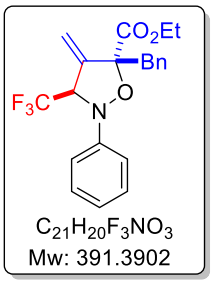
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl 5-methylhexa-2,3-dienoate **3h** (116 mg, 0.75 mmol) and in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4h** as liquid (103 mg, 60%). $\mathbf{R_f}$ (EtOAc/Hexane: 1/9) = 0.45. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.9 (C), 149.8 (C), 137.3 (C), 129.8 (CH), 128.9 (CH), 128.9 (CH), 124.4 (q, J_{C-F} = 278.4 Hz, C), 122.5 (CH), 113.9 (CH), 113.9 (CH), 78.4 (CH), 67.6 (q, J_{C-F} = 31.1 Hz, CH), 61.9 (CH_2), 31.3 (CH), 22.4 (CH_3), 21.8 (CH_3), 13.6 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.27-7.23 (m, 2H), 7.02 (d, *J* = 8.0 Hz, 2H), 6.96 (t, *J* = 7.2 Hz, 1H), 5.78 (d, *J* = 10.4 Hz, 1H), 5.17 (s, 1H), 4.83 (q, *J* = 7.2 Hz, 1H), 4.05-3.79 (m, 2H), 2.34-2.26 (m, 1H), 1.05-0.95 (m, 9H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.6 (s). **HRMS** for $C_{17}H_{21}F_3NO_3^+$: calcd. $[M+H]^+$: 344.1468, found: 344.1466.

Compound 4i: Ethyl 2-phenyl-4-(2-phenylethylidene)-3-(trifluoromethyl)isoxazolidine-5-carboxylate



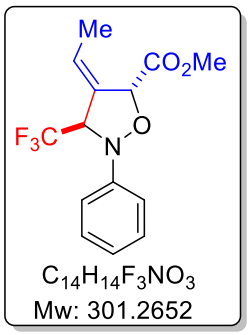
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl 5-phenylpenta-2,3-dienoate **3i** (152 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **4i** as liquid (113 mg, 58%). $\mathbf{R_f}$(EtOAc/Hexane: 1/9) = 0.48. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.5 (C), 149.6 (C), 138.2 (C), 133.4 (C), 129.5 (CH), 128.9 (CH), 128.9 (CH), 128.8 (CH), 128.8 (CH), 128.4 (CH), 128.4 (CH), 126.8 (CH), 124.3 (q, $J_{C\text{-}F}$ = 278.7 Hz, C), 122.6 (CH), 114.1 (CH), 114.1 (CH), 78.6 (CH), 67.6 (q, $J_{C\text{-}F}$ = 31.8 Hz, CH), 63.1 (CH_2), 36.8 (CH_2), 13.5 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.34-7.25 (m, 5H), 7.16 (d, J = 7.6 Hz, 2H), 7.08-7.06 (m, 2H), 7.02-6.99 (m, 1H), 6.23 (t, J = 7.4 Hz, 1H), 5.26 (s, 1H), 4.94 (q, J = 6.8 Hz, 1H), 3.99-3.85 (m, 2H), 3.47-3.44 (m, 2H), 0.99-0.95 (m, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.4. **HRMS** for $C_{21}H_{21}F_3NO_3^+$: calcd. $[M+H]^+$: 392.1468, found: 392.1464.

Compound 4j: Ethyl 5-benzyl-4-methylene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl 2-benzylbuta-2,3-dienoate **3j** (152 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **4j** as liquid (137 mg, 70%). $\mathbf{R_f}$(EtOAc/Hexane: 1/9) = 0.58. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 170.1 (C), 149.0 (C), 143.6 (C), 134.5 (C), 130.8 (CH), 130.8 (CH), 128.8 (CH), 128.8 (CH), 128.2 (CH), 128.2 (CH), 127.2 (CH), 124.1 (q, $J_{C\text{-}F}$ = 279.3 Hz, C), 122.9 (CH), 115.6 (CH_2), 115.3 (CH), 115.3 (CH), 87.8 (C), 68.5 (q, $J_{C\text{-}F}$ = 31.9 Hz, CH), 61.9 (CH_2), 43.3 (CH_2), 13.6 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.26-7.20 (m, 7H), 7.10-7.08 (m, 2H), 6.97-6.93 (m, 1H), 5.64 (s, 1H), 5.52 (s, 1H), 4.83-4.78 (m, 1H), 3.84-3.77 (m, 2H), 3.38-3.30 (m, 2H), 0.88 (t, J = 7.2 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -73.2 (s). **HRMS** for $C_{21}H_{21}F_3NO_3^+$: calcd. $[M+H]^+$: 392.1468, found: 392.1467.

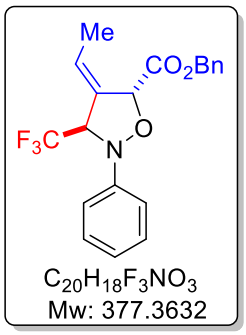
Compound 4k: Methyl 4-ethylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.9 mL, 1.50 mmol) and methyl penta-2,3-dienoate **3k** (84 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **4k** as liquid (92 mg, 61%). $\mathbf{R_f}$ (EtOAc/Hexane: 1/9) = 0.48. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.9 (C), 149.5 (C), 133.0 (C), 128.9 (CH), 128.9 (CH), 126.0 (CH), 124.3 (q, $J_{C\text{-}F}$ = 278.7 Hz, C), 122.8 (CH), 114.2 (CH), 114.2 (CH), 78.2 (CH), 67.8 (q, $J_{C\text{-}F}$ = 31.8 Hz, CH), 52.4 (CH_3), 16.3 (CH_3). **^{1}H**

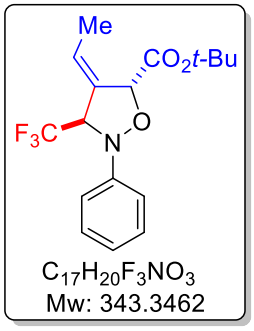
NMR (400 MHz, δppm/$CDCl_3$): 7.29-7.25 (m, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.98 (t, *J* = 7.4 Hz, 1H), 6.09 (q, *J* = 6.8 Hz, 1H), 5.18 (s, 1H), 4.86-4.80 (m, 1H), 3.49 (s, 3H), 1.73 (d, *J* = 6.8 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.7 (s). **HRMS** for $C_{14}H_{15}F_3NO_3^+$: calcd. $[M+H]^+$: 302.0999, found: 302.0999.

Compound 4l: Benzyl 4-ethylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



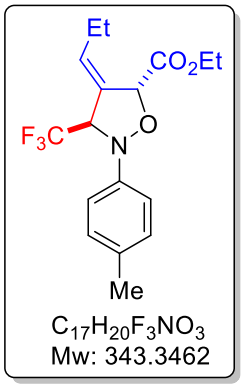
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and benzyl penta-2,3-dienoate **3l** (141 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4l** as liquid (119 mg, 63%). **R_f** (EtOAc/Hexane: 1/9) = 0.43. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.3 (C), 149.5 (C), 134.8 (C), 133.0 (C), 128.9 (CH), 128.9 (CH), 128.7 (CH), 128.7 (CH), 128.6 (CH), 128.6 (CH), 128.6 (CH), 126.0 (CH), 124.3 (q, J_{C-F} = 278.6 Hz, C), 122.7 (CH), 114.2 (CH), 114.2 (CH), 78.5 (CH), 67.8 (q, J_{C-F} = 31.9 Hz, CH), 67.6 (CH_2), 16.3 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.32-7.31 (m, 3H), 7.31 (t, *J* = 6.8 Hz, 2H), 7.14-7.13 (m, 2H), 7.01 (d, J = 6.0 Hz, 2H), 6.96 (t, *J* = 6.0 Hz, 1H), 6.11-6.01 (m, 1H), 5.22 (s, 1H), 5.02 (d, *J* = 9.6 Hz, 1H), 4.81-4.80 (m, 2H), 1.67 (d, *J* = 5.6 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.7 (s). **HRMS** for $C_{20}H_{19}F_3NO_3^+$: calcd. $[M+H]^+$: 378.1312, found: 378.1312.

Compound 4m: *t*-Butyl 4-ethylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate



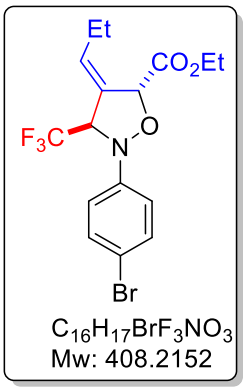
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and *t*-butyl penta-2,3-dienoate **3m** (116 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4m** as white solid (117 mg, 68%). **M.P** 65-67 °C. **R_f** (EtOAc/Hexane: 1/9) = 0.53. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 167.6 (C), 149.8 (C), 133.9 (C), 128.9 (CH), 128.9 (CH), 124.9 (CH), 124.4 (q, J_{C-F} = 278.6 Hz, C), 122.3 (CH), 114.1 (CH), 114.1 (CH), 82.7 (C), 80.2 (CH), 67.4 (q, J_{C-F} = 31.9 Hz, CH), 27.5 (CH_3), 27.5 (CH_3), 27.5 (CH_3), 16.1 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.28-7.24 (m, 2H), 7.04 (d, *J* = 8.0 Hz, 2H), 6.04 (t, *J* = 7.4 Hz, 1H), 6.04 (q, *J* = 6.8 Hz, 1H), 5.07 (s, 1H), 4.82-4.80 (m, 1H), 1.73 (d, *J* = 7.2 Hz, 3H), 1.20 (s, 9H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.7 (s). **HRMS** for $C_{17}H_{21}F_3NO_3^+$: calcd. $[M+H]^+$: 344.1468, found: 344.1468.

Compound 4n: Ethyl 4-propylidene-2-(*p*-tolyl)-3-(trifluoromethyl)isoxazolidine-5-carboxylate



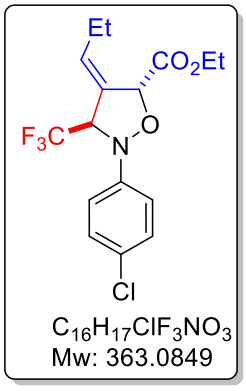
Following the general procedure, treatment of 4-methyl nitrosobenzene **1b** (61 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl hexa-2,3-dienoate **3c** (105 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **4n** as liquid (110 mg, 64%). $\mathbf{R_f}$(EtOAc/Hexane: 1/9) = 0.57. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.8 (C), 147.3 (C), 132.4 (C), 132.3 (C), 131.7 (CH), 129.4 (CH), 129.4 (CH), 124.0 (q, $J_{C\text{-}F}$ = 278.6 Hz, C), 114.6 (CH), 114.6 (CH), 78.4 (CH), 67.8 (q, $J_{C\text{-}F}$ = 31.5 Hz, CH), 61.8 (CH_2), 24.6 (CH_3), 20.6 (CH_2), 13.7 (CH_3), 13.2 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.05 (d, *J* = 6.8 Hz, 2H), 6.97-6.93 (m, 2H), 5.96 (t, *J* = 5.8 Hz, 1H), 5.13 (s, 1H), 4.79 (q, *J* = 6.0 Hz, 1H), 4.00-3.88 (m, 2H), 2.27 (s, 3H), 2.08-2.03 (m, 2H), 1.05-1.01 (m, 6H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.7 (s). **HRMS** for $C_{17}H_{21}F_3NO_3^+$: calcd. $[M+H]^+$: 344.1468, found: 344.1460.

Compound 4o: Ethyl-2-(4-bromophenyl)-4-propylidene-3-(trifluoromethyl)isoxazolidine-5-carboxylate



Following the general procedure, treatment of 4-bromonitrosobenzene **1c** (92 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl hexa-2,3-dienoate **3c** (105 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **4o** as liquid (133 mg, 65%). $\mathbf{R_f}$(EtOAc/Hexane: 1/9) = 0.54. **^{13}C NMR** (100 MHz, δ ppm/$CDCl_3$): 168.6 (C), 148.8 (C), 132.7 (CH), 131.7 (CH), 131.7 (CH), 131.2 (C), 124.2 (q, $J_{C\text{-}F}$ = 222.8 Hz, C), 115.7 (CH), 115.7 (CH), 114.9 (C), 78.6 (CH), 67.5 (q, $J_{C\text{-}F}$ = 25.4 Hz, CH), 62.0 (CH_2), 24.5 (CH_2), 13.7 (CH_3), 13.2 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.35 (d, *J* = 6.8 Hz, 2H), 6.90 (d, *J* = 6.8 Hz, 2H), 5.97 (t, *J* = 6.4 Hz, 1H), 5.16 (s, 1H), 4.76 (q, *J* = 5.6 Hz, 1H), 4.04-3.90 (m, 2H), 2.13-2.03 (m, 2H), 1.06-1.01 (m, 6H). **^{19}F NMR** (376 MHz, δ ppm/$CDCl_3$): -74.6 (s). **HRMS** for $C_{16}H_{18}BrF_3NO_3^+$: calcd. $[M+H]^+$: 408.0417, found: 408.0417.

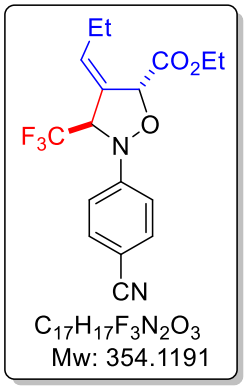
Compound 4p: Ethyl -2-(4-chlorophenyl)-4-propylidene-3-(trifluoromethyl)isoxazolidine-5-carboxylate:



Following the general procedure, treatment of 4-chloronitrosobenzene **1d** (70 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl hexa-2,3-dienoate **3c** (105 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **4p** as liquid (127 mg, 70%). $\mathbf{R_f}$ (EtOAc/Hexane: 1/9) = 0.52. **^{13}C NMR** (100 MHz, δ ppm/$CDCl_3$): 168.6 (C), 148.3 (C), 132.7 (CH), 131.2 (C), 128.8 (CH), 128.8 (CH), 127.6 (C), 124.2 (q, $J_{C\text{-}F}$ = 371.5 Hz, C), 115.4 (CH), 115.4 (CH), 78.6 (CH), 67.6 (q, $J_{C\text{-}F}$ = 42.5 Hz,

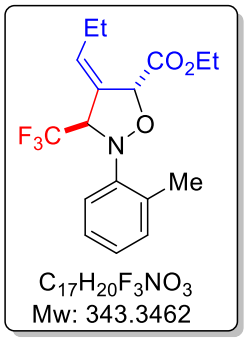
CH), 62.0 (CH_2), 24.5 (CH_2), 13.7 (CH_3), 13.2 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.21 (d, *J* = 7.2 Hz, 2H), 6.96 (d, *J* = 7.2 Hz, 2H), 5.97 (t, *J* = 6.2 Hz, 1H), 5.16 (s, 1H), 4.76 (q, *J* = 6.0 Hz, 1H), 4.01-3.93 (m, 2H), 2.12-2.04 (m, 2H), 1.06-1.01 (m, 6H). **^{19}F NMR** (376 MHz, δ ppm/$CDCl_3$): -74.6 (s). **HRMS** for $C_{16}H_{18}ClF_3NO_3^+$: calcd. $[M+H]^+$: 364.0922, found: 364.0927.

Compound 4q: Ethyl -2-(4-cyanophenyl)-4-propylidene-3-(trifluoromethyl)isoxazolidine-5-carboxylate:



Following the general procedure, treatment of 4-cyanonitrosobenzene **1e** (66 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.90 mL, 1.50 mmol) and ethyl hexa-2,3-dienoate **3c** (105 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4q** as liquid (113 mg, 64%). **R_f** (EtOAc/Hexane: 1/9) = 0.56. **^{13}C NMR** (100 MHz, δ ppm/$CDCl_3$): 168.4 (C), 152.5 (C), 133.1 (CH), 133.1 (CH), 133.1 (CH), 130.5 (C), 124.0 (q, J_{C-F} = 279.2 Hz, C), 119.3 (C), 113.3 (CH), 113.3 (CH), 104.6 (C), 78.6 (CH), 66.1 (q, J_{C-F} = 32.6 Hz, CH), 62.1 (CH_2), 24.4 (CH_2), 13.7 (CH_3), 13.1 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.54 (d, *J* = 7.2 Hz, 2H), 7.04 (d, *J* = 7.2 Hz, 2H), 6.00 (t, *J* = 6.2 Hz, 1H), 5.21 (s, 1H), 4.84 (q, *J* = 5.6 Hz, 1H), 4.04-3.91 (m, 2H), 2.16-2.08 (m, 2H), 1.04-1.00 (m, 6H). **^{19}F NMR** (376 MHz, δ ppm/$CDCl_3$): -74.5 (s). **HRMS** for $C_{17}H_{18}F_3N_2O_3^+$: calcd. $[M+H]^+$: 355.1264, found: 355.1266.

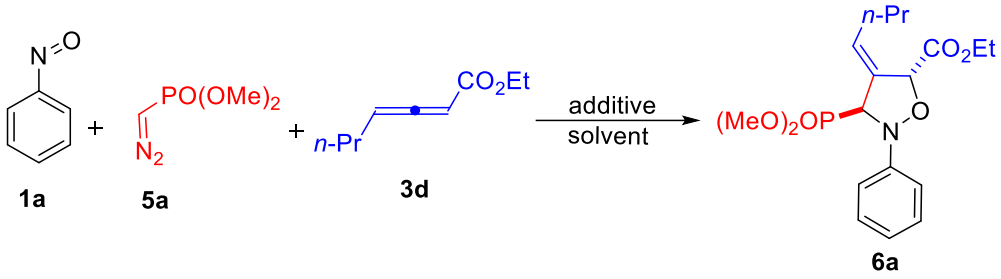
Compound 4r: Ethyl 4-propylidene-2-(*o*-tolyl)-3-(trifluoromethyl)isoxazolidine-5-carboxylate



Following the general procedure, treatment of 2-methyl nitrosobenzene **1f** (61 mg, 0.50 mmol) with CF_3CHN_2 **2** (1.9 mL, 1.50 mmol) and ethyl hexa-2,3-dienoate **3c** (105 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **4r** as liquid (106 mg, 62%) . **R_f** (EtOAc/Hexane: 1/9) = 0.57. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 169.3 (C), 145.6 (C), 136.8 (C), 133.4 (C), 132.8 (CH), 131.3 (CH), 127.2 (CH), 125.8 (CH), 124.2 (q, J_{C-F} = 223.6 Hz, C), 120.0 (CH), 77.2 (CH), 65.6 (q, J_{C-F} = 24.7 Hz, CH), 61.6 (CH_2), 24.8 (CH_3), 18.5 (CH_2), 13.7 (CH_3), 13.4 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.22-7.20 (m, 1H), 7.13-7.02 (m, 3H), 6.08 (t, *J* = 7.6 Hz, 1H), 5.03 (s, 1H), 4.78 (q, *J* = 7.2 Hz, 1H), 3.89-3.66 (m, 2H), 2.50 (s, 3H), 2.28-2.17 (m, 2H), 1.10 (t, *J* = 7.4 Hz, 3H), 0.95 (t, *J* = 7.2 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -74.5 (s). **HRMS** for $C_{17}H_{21}F_3NO_3^+$: calcd. $[M+H]^+$: 344.1468, found: 344.1446.

Optmization study for the synthesis of phosphonyl isoxazolidines

Table S1: Optimization of the reaction conditions[a]

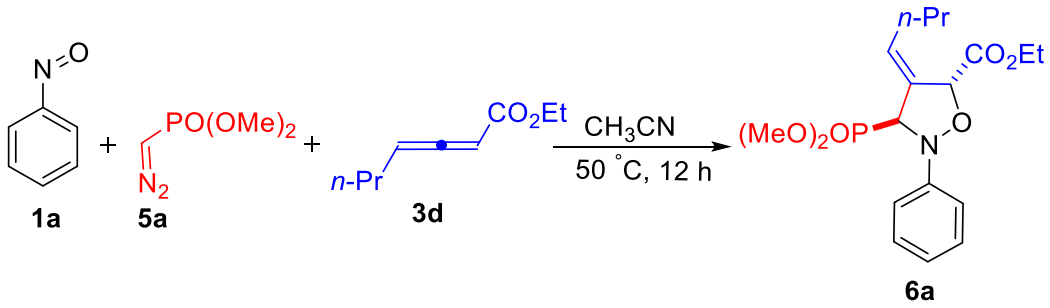


1a 5a 3d 6a

Entry	Solvent	additive	time (h)	yield (%)[b]
1	**CH_3CN**	**-**	**12**	**70**
2	DCM	-	12	48
3	Toluene	-	12	52
4	DMF	-	12	65
5	DMSO	-	12	45
6	1,4 –dioxane	-	12	42
7	DCE	-	12	40
8	THF	-	12	50
9	CH_3CN	CsF	12	58
10	CH_3CN	TBAF	12	50
11	CH_3CN	DBU	12	42
12	CH_3CN	DABCO	12	55
13[c]	CH_3CN	-	72	55
14[d]	CH_3CN	-	12	40

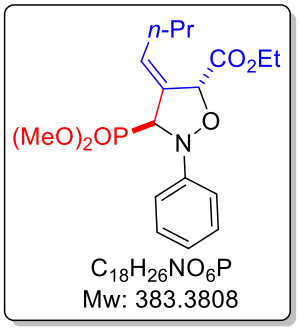
[a]**1a** (0.50 mmol), **5a** (0.50 mmol), **3d** (0.75 mmol) solvent (2.0 mL), 50 ˚C. [b]Isolated yield after silica gel column chromatography. [c]Reaction carried out at 25 ˚C. [d]Reaction carried out at 80 ˚C.

General procedure for the synthesis of phosphonyl isoxazolidines 6a-6m



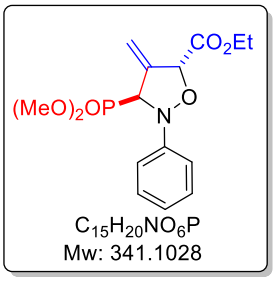
A 10 mL round-bottom flask charged with nitroso benzene **1a** (54 mg, 0.50 mmol) was sealed, evacuated, backfilled with nitrogen and added dry CH_3CN (2 mL). Subsquently, the Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and ethyl hepta-2,3-dienoate **3d** (116 mg, 0.75 mmol) were added via a syringe. This reaction mixture was stirred at 50 °C for 12 h. After the completion of reaction, as indicated by TLC, the solvent was evaporated under reduced pressure. The residue was purified using column chromatography (100-200 mesh silica gel) using ethyl acetate/hexane as the eluent.

Compound 6a: Ethyl 4-butylidene-3-(dimethoxyphosphoryl)-2-phenylisoxazolidine-5-carboxylate



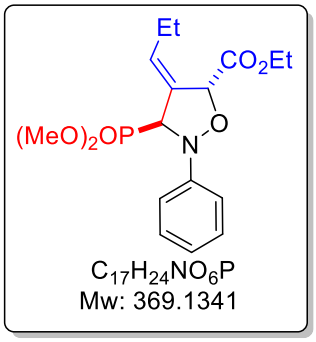
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and ethyl hepta-2,3-dienoate **3d** (116 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **6a** as liquid (134 mg, 70%). $\mathbf{R_f}$ (EtOAc/Hexane: 6/4) = 0.19. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 169.0 (C), 150.9 (d, J_{C-P} = 12.5 Hz, C), 133.4 (d, J_{C-P} = 2.5 Hz, C), 128.7 (CH), 128.7 (CH), 128.3 (d, J_{C-P} = 7.5 Hz, CH), 122.0 (CH), 114.1 (CH), 114.1 (CH), 78.2 (d, J_{C-P} = 2.7 Hz, CH), 64.2 (d, J_{C-P}= 176.7 Hz, CH), 61.6 (CH_2), 54.7 (d, J_{C-P} = 7.2 Hz, CH_3), 53.9 (d, J_{C-P} = 7.4 Hz, CH_3), 32.8 (CH_2) 22.1 (d, J_{C-P} = 2.9 Hz, CH_2), 13.7 (CH_3), 13.4 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.24 (d, J = 7.8 Hz, 2H), 7.03 (d, J = 8.0 Hz, 2H), 6.92 (t, J = 7.2 Hz, 1H), 6.01-5.99 (m, 1H), 5.12 (s, 1H), 4.81 (d, J = 10.0 Hz, 1H), 3.91-3.74 (m, 8H), 2.02-2.00 (m, 2H), 1.44-1.39 (m, 2H), 0.90-0.86 (m, 6H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 20.4 (s). **HRMS** for $C_{18}H_{27}NO_6P^+$: calcd. $[M+H]^+$: 384.1571, found: 384.1570.

Compound 6b: Ethyl 3-(dimethoxyphosphoryl)-4-methylene-2-phenylisoxazolidine-5-carboxylate



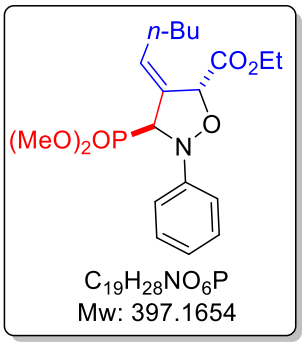
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and ethyl buta-2,3-dienoate **3b** (84 mg, 0.75 mmol) and in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **6b** as liquid (111 mg, 65%). $\mathbf{R_f}$(EtOAc/Hexane: 6/4) = 0.50. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.7 (d, $J_{C\text{-}p}$ = 1.9 Hz, C), 150.5 (d, $J_{C\text{-}P}$ = 12.7 Hz, C), 142.0 (d, $J_{C\text{-}P}$ = 3.5 Hz, C), 128.7 (CH), 128.7 (CH), 123.0 (CH), 115.8 (CH), 115.8 (CH), 111.9 (d, $J_{C\text{-}P$ = 8.2 Hz, CH_2), 79.2 (d, $J_{C\text{-}P$ = 2.6 Hz, CH), 65.9 (d, $J_{C\text{-}P$ = 175.2 Hz, CH), 61.9 (CH_2), 54.7 (d, $J_{C\text{-}P$ = 7.2 Hz, CH_3), 54.1 (d, $J_{C\text{-}P$ = 7.2 Hz, CH_3), 13.9 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.29-7.27 (m, 2H), 7.25-7.21 (m, 2H), 7.00 (t, *J* = 7.2 Hz, 1H), 5.59-5.52 (m, 2H), 5.15-5.14 (m, 1H), 4.78-4.74 (m, 1H), 4.09-4.04 (m, 2H), 3.88 (d, $J_{H\text{-}P$ = 2.4 Hz, 3H), 3.85 (d, $J_{H\text{-}P$ = 2.4 Hz, 3H), 1.12 (t, *J* = 7.2 Hz, 3H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 19.6 (s). **HRMS** for $C_{15}H_{21}NO_6P^+$: calcd. $[M+H]^+$: 342.1101, found: 342.1091.

Compound 6c: Ethyl 3-(dimethoxyphosphoryl)-2-phenyl-4-propylideneisoxazolidine-5-carboxylate



Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and ethyl hexa-2,3-dienoate **3c** (105 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **6c** as liquid (111 mg, 60%). $\mathbf{R_f}$(EtOAc/Hexane: 6/4) = 0.25. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 169.0 (d, $J_{C\text{-}p}$ = 4.3 Hz, C), 150.9 (d, $J_{C\text{-}P}$ = 10.0 Hz, C), 132.8 (d, $J_{C\text{-}P}$ = 2.0 Hz, C), 129.8 (d, $J_{C\text{-}P}$ = 6.1 Hz, CH), 128.7 (CH), 128.7 (CH), 122.2 (CH), 114.3 (CH), 114.3 (CH), 78.1 (d, $J_{C\text{-}P$ = 2.2 Hz, CH), 64.3 (d, $J_{C\text{-}P$ = 141.1 Hz, CH), 61.7 (CH_2), 54.8 (d, $J_{C\text{-}P$ = 5.7 Hz, CH_3), 53.9 (d, $J_{C\text{-}P$ = 5.9 Hz, CH_3), 24.4 (d, $J_{C\text{-}P$ = 4.0 Hz, CH_2), 13.4 (CH_3), 13.4 (d, $J_{C\text{-}P$ = 2.7 Hz, CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.26-7.21 (m, 2H), 7.04 (d, *J* = 7.6 Hz, 2H), 6.93 (t, *J* = 7.2 Hz, 1H), 6.03-5.96 (m, 1H), 5.13 (s, 1H), 4.83-4.80 (m, 1H), 3.92-3.77 (m, 8H), 2.14-1.99 (m, 2H), 1.00 (t, *J* = 7.4 Hz, 3H), 0.92 (t, *J* = 7.2 Hz, 3H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 20.4 (s). **HRMS** for $C_{17}H_{25}NO_6P^+$: calcd. $[M+H]^+$: 370.1414, found: 370.1408.

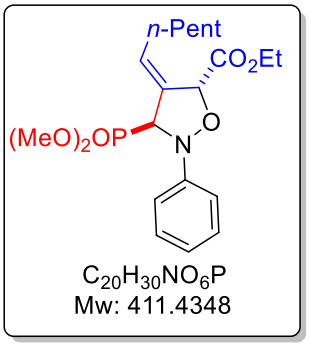
Compound 6d: Ethyl 3-(dimethoxyphosphoryl)-4-pentylidene-2-phenylisoxazolidine-5-carboxylate



$C_{19}H_{28}NO_6P$
Mw: 397.1654

Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and ethyl octa-2,3-dienoate **3e** (126 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **6d** as liquid (141 mg, 71%). $\mathbf{R_f}$ (EtOAc/Hexane: 6/4) = 0.44. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 169.0 (d, $J_{C\text{-}p}$ = 2.2 Hz, C), 150.9 (d, $J_{C\text{-}P}$ = 12.4 Hz, C), 133.2 (d, $J_{C\text{-}P}$ = 2.6 Hz, C), 128.7 (CH), 128.7 (CH), 128.5 (d, $J_{C\text{-}P}$ = 7.6 Hz, CH), 122.1 (CH), 114.1 (CH), 114.1 (CH), 78.2 (d, $J_{C\text{-}P}$ = 2.8 Hz, CH), 64.2 (d, $J_{C\text{-}P}$= 176.7 Hz, CH), 61.6 (CH_2), 54.7 (d, $J_{C\text{-}P}$ = 7.2 Hz, CH_3), 53.9 (d, $J_{C\text{-}P}$ = 7.4 Hz, CH_3), 31.0 (d, $J_{C\text{-}P}$ = 2.9 Hz, CH_2), 30.7 (d, $J_{C\text{-}P}$ = 2.6 Hz, CH_2), 22.3 (CH_2), 14.0 (CH_3), 13.5 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.26-7.21 (m, 2H), 7.04 (d, J = 8.0 Hz, 2H), 6.93 (t, J = 7.2 Hz, 1H), 6.02-6.00 (m, 1H), 5.12 (s, 1H), 4.81 (d, J = 9.6 Hz, 1H), 3.93-3.75 (m, 8H), 2.05-2.03 (m, 2H), 1.37-1.29 (m, 4H), 0.92-0.85 (m, 6H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 20.4 (s). **HRMS** for $C_{19}H_{29}NO_6P^+$: calcd. $[M+H]^+$: 398.1727, found: 398.1724.

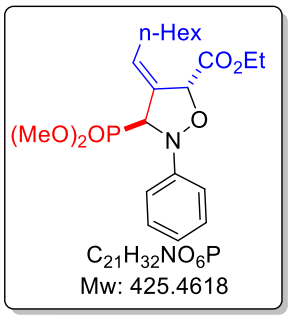
Compound 6e: Ethyl 3-(dimethoxyphosphoryl)-4-hexylidene-2-phenylisoxazolidine-5-carboxylate



$C_{20}H_{30}NO_6P$
Mw: 411.4348

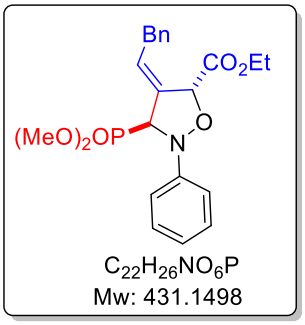
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and ethyl nona-2,3-dienoate **3f** (137 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **6e** as liquid (144 mg, 70%). $\mathbf{R_f}$ (EtOAc/Hexane: 6/4) = 0.39. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 169.0 (d, $J_{C\text{-}p}$ = 2.3 Hz, C), 151.0 (d, $J_{C\text{-}P}$ = 12.4 Hz, C), 133.2 (d, $J_{C\text{-}P}$ = 2.4 Hz, C), 128.7 (CH), 128.7 (CH), 128.6 (d, $J_{C\text{-}P}$ = 7.6 Hz, CH), 122.1 (CH), 114.2 (CH), 114.2 (CH), 78.3 (d, $J_{C\text{-}P}$ = 2.7 Hz, CH), 64.3 (d, $J_{C\text{-}P}$ = 176.6 Hz, CH), 61.7 (CH_2), 54.7 (d, $J_{C\text{-}P}$ = 7.2 Hz, CH_3), 54.0 (d, $J_{C\text{-}P}$ = 7.3 Hz, CH_3), 31.5 (CH_2), 31.0 (d, $J_{C\text{-}P}$ = 2.5 Hz, CH_2), 28.6 (d, $J_{C\text{-}P}$ = 2.9 Hz, CH_2), 22.6 (CH_2), 14.1 (CH_3), 13.5 (CH_3) . **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.26-7.21 (m, 2H), 7.04 (d, J = 8.0 Hz, 2H), 6.93 (t, J = 7.2 Hz, 1H), 6.04-6.00 (m, 1H), 5.12 (s, 1H), 4.82 (d, J = 10.0 Hz, 1H), 3.93-3.75 (m, 8H), 2.06-2.01 (m, 2H), 1.41-1.36 (m, 2H), 1.28-1.25 (m, 4H), 0.92-0.84 (m, 6H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 20.4 (s). **HRMS** for $C_{20}H_{31}NO_6P^+$: calcd. $[M+H]^+$: 412.1884, found: 412.1882.

Compound 6f: Ethyl 3-(dimethoxyphosphoryl)-4-heptylidene-2-phenylisoxazolidine-5-carboxylate



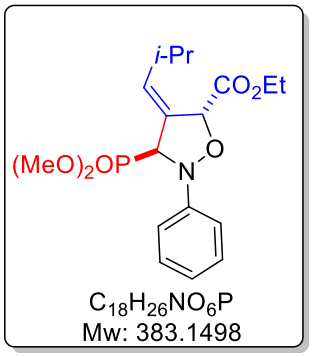
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and ethyl deca-2,3-dienoate **3g** (147 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **6f** as liquid (149 mg, 70%). $\mathbf{R_f}$(EtOAc/Hexane: 6/4) = 0.56. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 169.0 (C), 150.9 (d, $J_{C\text{-}P}$ = 12.4 Hz, C), 133.1 (C), 128.7 (CH), 128.7 (CH), 128.5 (d, $J_{C\text{-}P}$ = 7.6 Hz, CH), 122.0 (CH), 114.1 (CH), 114.1 (CH), 78.2 (d, $J_{C\text{-}P}$ = 2.8 Hz, CH), 64.2 (d, $J_{C\text{-}P}$ = 176.7 Hz, CH), 61.6 (CH_2), 54.7 (d, $J_{C\text{-}P}$ = 7.2 Hz, CH_3), 53.9 (d, $J_{C\text{-}P}$ = 7.4 Hz, CH_3), 31.7 (CH_2), 31.0 (CH_2), 28.9 (CH_2), 28.8 (CH_2), 22.6 (CH_2), 14.0 (CH_3), 13.4 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.26-7.21 (m, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.93 (t, *J* = 7.2 Hz, 1H), 6.03-5.98 (m, 1H), 5.12 (s, 1H), 4.81 (d, *J* = 9.6 Hz, 1H), 3.90-3.78 (m, 8H), 2.07-1.99 (m, 2H), 1.40-1.36 (m, 2H), 1.29-1.21 (m, 6H), 0.91-0.84 (m, 6H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 20.4 (s). **HRMS** for $C_{21}H_{33}NO_6P^+$: calcd. $[M+H]^+$: 426.2040, found: 426.2040.

Compound 6g: Ethyl 3-(dimethoxyphosphoryl)-2-phenyl-4-(2-phenylethylidene)isoxazolidine-5-carboxylate



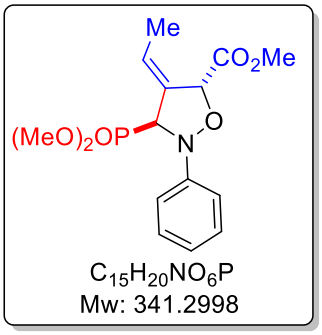
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and ethyl 5-phenylpenta-2,3-dienoate **3i** (152 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **6g** as liquid (129 mg, 60%). $\mathbf{R_f}$ (EtOAc/Hexane: 6/4) = 0.52. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.9 (d, $J_{C\text{-}p}$ = 2.1 Hz, C), 150.9 (d, $J_{C\text{-}P}$ = 12.5 Hz, C), 138.8 (d, $J_{C\text{-}P}$ = 2.8 Hz, C), 134.7 (d, $J_{C\text{-}P}$ = 2.7 Hz, C), 128.8 (CH), 128.8 (CH), 128.7 (CH), 128.7 (CH), 128.4 (CH), 128.4 (CH), 126.6 (CH), 126.5 (d, $J_{C\text{-}P}$ = 7.6 Hz, CH), 122.3 (CH), 114.3 (CH), 114.3 (CH), 78.2 (d, $J_{C\text{-}P}$ = 2.7 Hz, CH), 64.4 (d, $J_{C\text{-}P}$ = 176.8 Hz, CH), 61.8 (CH_2), 54.7 (d, $J_{C\text{-}P}$ = 7.1 Hz, CH_3), 54.0 (d, $J_{C\text{-}P}$ = 7.4 Hz, CH_3), 36.7 (d, $J_{C\text{-}P}$ = 2.6 Hz, CH_2), 13.5 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.28-7.19 (m, 5H), 7.15 (d, *J* = 7.2 Hz, 2H), 7.06 (d, *J* = 8.0 Hz, 2H), 6.95 (t, *J* = 7.2 Hz, 1H), 6.25-6.20 (m, 1H), 5.23 (s, 1H), 4.89 (d, *J* = 10.4 Hz, 1H), 3.88-3.80 (m, 8H), 3.46-3.40, (m, 2H), 0.88 (t, *J* = 7.0 Hz, 3H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 20.3 (s). **HRMS** for $C_{22}H_{27}NO_6P^+$: calcd. $[M+H]^+$: 432.1571, found: 432.1569.

Compound 6h: Ethyl 3-(dimethoxyphosphoryl)-4-(2-methylpropylidene)-2-phenylisoxazolidine-5-carboxylate



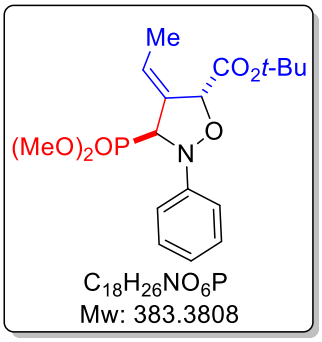
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and ethyl 5-methylhexa-2,3-dienoate **3h** (116 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **6h** as liquid (105 mg, 55%). $\mathbf{R_f}$ (EtOAc/Hexane: 6/4) = 0.44. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 169.1 (d, $J_{C\text{-}p}$ = 2.1 Hz, C), 150.9 (d, $J_{C\text{-}P}$ = 12.3 Hz, C), 134.7 (d, $J_{C\text{-}P}$ = 7.6 Hz, CH), 130.9 (d, $J_{C\text{-}P}$ = 2.4 Hz, C), 128.7 (CH), 128.7 (CH), 122.0 (CH), 114.1 (CH), 114.1 (CH), 77.9 (d, $J_{C\text{-}P}$ = 2.9 Hz, CH), 64.1 (d, $J_{C\text{-}P}$ = 176.6 Hz, CH), 61.6 (CH_2), 54.7 (d, $J_{C\text{-}P}$ = 7.1 Hz, CH_3), 54.0 (d, $J_{C\text{-}P}$ = 7.4 Hz, CH_3), 31.1 (d, $J_{C\text{-}P}$ = 2.4 Hz, CH), 22.5 (d, $J_{C\text{-}P}$ = 3.7 Hz, CH_3), 21.9 (d, $J_{C\text{-}P}$ = 2.8 Hz, CH_3), 13.5 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.26-7.12 (m, 2H), 7.02 (d, *J* = 8.0 Hz, 2H), 6.92 (t, *J* = 7.2 Hz, 1H), 5.85-5.81 (m, 1H), 5.13 (s, 1H), 4.79 (d, *J* = 10.0 Hz, 1H), 3.92-3.72 (m, 8H), 2.33-2.26 (m, 1H), 1.03 (d, *J* = 6.4 Hz, 3H), 0.93-0.87 (m, 6H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 20.3 (s). **HRMS** for $C_{18}H_{27}NO_6P^+$: calcd. $[M+H]^+$: 384.1571, found: 384.1576.

Compound 6i: Methyl 3-(dimethoxyphosphoryl)-4-ethylidene-2-phenylisoxazolidine-5-carboxylate



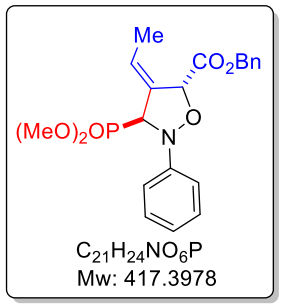
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and methyl penta-2,3-dienoate **3k** (84 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 °C for 12 h followed by column chromatography afforded the product **6i** as liquid (102 mg, 60%). $\mathbf{R_f}$ (EtOAc/Hexane: 6/4) = 0.23. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 169.2 (d, $J_{C\text{-}p}$ = 2.2 Hz, C), 150.6 (d, $J_{C\text{-}P}$ = 12.8 Hz, C), 134.1 (d, $J_{C\text{-}P}$ = 2.9 Hz, C), 128.8 (CH), 128.8 (CH), 123.2 (d, $J_{C\text{-}P}$ = 7.7 Hz, CH), 122.4 (CH), 114.4 (CH), 114.4 (CH), 77.9 (d, $J_{C\text{-}P}$ = 2.8 Hz, CH), 64.5 (d, $J_{C\text{-}P}$ = 176.5 Hz, CH), 54.7 (d, $J_{C\text{-}P}$ = 7.2 Hz, CH_3), 53.9 (d, $J_{C\text{-}P}$ = 8.0 Hz, CH_3), 52.3 (CH_3), 16.2 (d, $J_{C\text{-}P}$ = 2.9 Hz, CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.27-7.23 (m, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 6.95 (t, *J* = 7.2 Hz, 1H), 6.11-6.07 (m, 1H), 5.16 (s, 1H), 4.83-4.80 (m, 1H), 3.87 (d, *J* = 2.8 Hz, 3H), 3.84 (d, J = 2.4 Hz, 3H), 3.47 (s, 3H), 1.73-1.67 (m, 3H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 20.4 (s). **HRMS** for $C_{15}H_{21}NO_6P^+$: calcd. $[M+H]^+$: 342.1101, found: 342.1112.

Compound 6j: *t*-Butyl 3-(dimethoxyphosphoryl)-4-ethylidene-2-phenylisoxazolidine-5-carboxylate



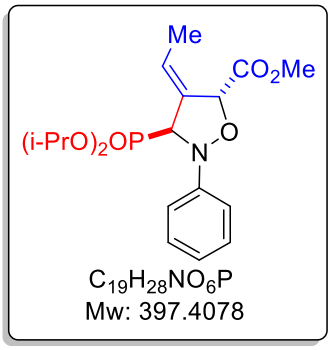
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and *t*-butyl penta-2,3-dienoate **3m** (116 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **6j** as liquid (115 mg, 60%). **M.P** 125-127 ˚C. **R_f** (EtOAc/Hexane: 6/4) = 0.32. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.0 (d, J_{C-p} = 2.1 Hz, C), 151.0 (d, J_{C-P} = 12.1 Hz, C), 135.1 (d, J_{C-P} = 2.7 Hz, C), 128.8 (CH), 128.8 (CH), 122.2 (d, J_{C-P} = 7.6 Hz, CH), 121.8 (CH), 114.3 (CH), 114.3 (CH), 82.4 (C), 79.7 (d, J_{C-P} = 2.9 Hz, CH), 64.0 (d, J_{C-P}= 176.9 Hz, CH), 54.7 (d, J_{C-P} = 7.1 Hz, CH_3), 53.8 (d, J_{C-P} = 7.6 Hz, CH_3), 27.4 (CH_3), 27.4 (CH_3), 27.4 (CH_3), 16.0 (d, J = 2.9 Hz, CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.26-7.21 (m, 2H), 7.06 (d, J = 7.6 Hz, 2H), 6.93 (t, J = 7.2 Hz, 1H), 6.08-6.03 (m, 1H), 5.04 (s, 1H), 4.80-4.77 (m, 1H), 3.83 (d, J = 5.4 Hz, 3H), 1.03 (d, J = 6.4 Hz, 3H), 1.73-1.70 (m, 3H), 1.14 (s, 9H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 20.4 (s). **HRMS** for $C_{18}H_{27}NO_6P^+$: calcd. $[M+H]^+$: 384.1571, found: 384.1570.

Compound 6k: Benzyl 3-(dimethoxyphosphoryl)-4-ethylidene-2-phenylisoxazolidine-5-carboxylate



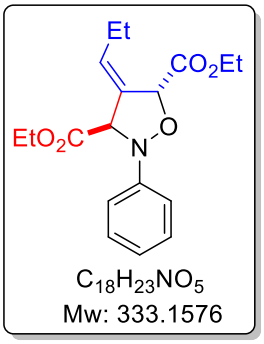
Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5a** (75 mg, 0.50 mmol) and benzyl penta-2,3-dienoate **3l** (141 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **6k** as liquid (127 mg, 61%). **R_f** (EtOAc/Hexane: 6/4) = 0.36. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 168.6 (d, J_{C-p} = 2.2 Hz, C), 150.7 (d, J_{C-P} = 12.8 Hz, C), 134.9 (C), 134.1 (d, J_{C-P} = 2.9 Hz, C), 128.8 (CH), 128.8 (CH), 128.6 (CH), 128.6 (CH), 128.5 (CH),128.5 (CH), 128.5 (CH), 123.3 (d, J_{C-P} = 7.8 Hz, CH), 122.4 (CH), 114.4 (CH), 114.4 (CH), 78.1 (d, J_{C-P} = 2.8 Hz, CH), 67.4 (CH_2), 64.6 (d, J_{C-P} = 176.6 Hz, CH), 54.8 (d, J_{C-P} = 7.2 Hz, CH_3), 53.9 (d, J_{C-P} = 7.4 Hz, CH_3), 16.2 (d, J_{C-P} = 3.0 Hz, CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.30-7.26 (m, 3H), 7.21-7.17 (m, J = 8.0 Hz, 2H), 7.09-7.03 (m, 4H), 6.93 (t, J = 7.4 Hz, 1H), 6.08-6.04 (m, 1H), 5.20 (s, 1H), 4.97 (d, J = 12.4 Hz, 1H), 4.81-4.78 (m, 1H), 4.73 (d, J = 12.4 Hz, 1H), 3.85 (d, J = 2.0 Hz, 3H), 3.83 (d, J = 2.0 Hz, 3H), 1.67-1.66 (m, 3H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 20.4 (s). **HRMS** for $C_{18}H_{25}NO_6P^+$: calcd. $[M+H]^+$: 418.1414, found: 418.1415.

Compound 6l: Methyl 3-(diisopropoxyphosphoryl)-4-ethylidene-2-phenylisoxazolidine-5-carboxylate



Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with Seyferth-Gilbert reagent **5b** (103 mg, 0.50 mmol) and methyl penta-2,3-dienoate **3j** (84 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **6k** as liquid (119 mg, 60%). R_f (EtOAc/Hexane: 6/4) = 0.56. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 169.4 (C), 151.1 (d, J_{C-P} = 13.2 Hz, C), 134.6 (d, J_{C-P} = 2.4 Hz, C), 128.6 (CH), 128.6 (CH), 122.6 (d, J_{C-P} = 7.7 Hz, CH), 122.1 (CH), 114.5 (CH), 114.5 (CH), 77.9 (d, J_{C-P} = 1.8 Hz, CH), 72.7 (d, J_{C-P}= 7.2 Hz, CH), 72.1 (d, J_{C-P} = 7.7 Hz, CH), 65.2 (d, J_{C-P} = 178.6 Hz, CH), 52.2 (CH_3), 24.5 (d, J_{C-P} = 2.8 Hz, CH_3), 24.2 (d, J_{C-P} = 3.1 Hz, CH_3), 24.1 (d, J_{C-P} = 5.2 Hz, CH_3), 23.8 (d, J_{C-P} = 5.5 Hz, CH_3), 16.1 (d, J_{C-P} = 2.2 Hz, CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.26-7.20 (m, 2H), 7.06 (d, *J* = 8.0 Hz, 2H), 6.91 (t, *J* = 7.2 Hz, 1H), 6.09-6.05 (m, 1H), 5.13 (s, 1H), 4.87-4.79 (m, 2H), 4.73-4.69 (m, 1H), 3.43 (s, 3H), 1.70-1.67 (m, 3H), 1.36-1.32 (m, 9H), 1.26 (d, *J* = 6.0 Hz, 3H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 16.9 (s). **HRMS** for $C_{19}H_{29}NO_6P^+$: calcd. $[M+H]^+$: 398.1727, found: 398.1714.

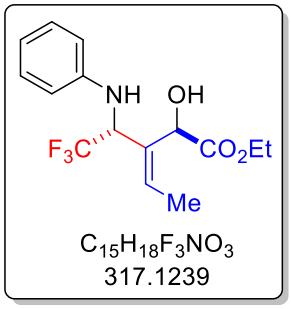
Compound 6m: Diethyl-2-phenyl-4-propylideneisoxazolidine-3,5-dicarboxylate:



Following the general procedure, treatment of nitrosobenzene **1a** (54 mg, 0.50 mmol) with ethyl diazoacetate **5c** (57mg, 0.50 mmol) and ethyl hexa-2,3-dienoate **3c** (105 mg, 0.75 mmol) in CH_3CN (2 mL) at 50 ˚C for 12 h followed by column chromatography afforded the product **6m** as liquid (50 mg, 30%). R_f (EtOAc/Hexane: 1/9) = 0.56. **^{13}C NMR** (100 MHz, δ ppm/$CDCl_3$): 169.5 (C), 169.2 (C), 148.9 (C), 135.5 (C), 129.3 (CH), 128.8 (CH), 128.8 (CH), 122.2 (CH), 115.0 (CH), 115.0 (CH), 76.8 (CH), 68.0 (CH), 61.8 (CH_2), 61.7 (CH_2), 23.7 (CH_2), 14.2 (CH_3), 14.0 (CH_3), 13.5 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.17-7.13 (m, 2H), 6.90 (d, *J* = 7.6 Hz, 2H), 6.85 (t, *J* = 8.0 Hz, 1H), 5.75-5.70 (m, 1H), 5.12 (s, 1H), 4.83-4.81 (m, 1H), 4.14-3.96 (m, 4H), 2.11-2.05 (m, 2H), 1.09-1.03 (m, 6H), 0.93 (t, *J* = 7.6 Hz, 3H). **HRMS** for $C_{18}H_{24}NO_5^+$: calcd. $[M+H]^+$: 334.1649, found: 334.1651.

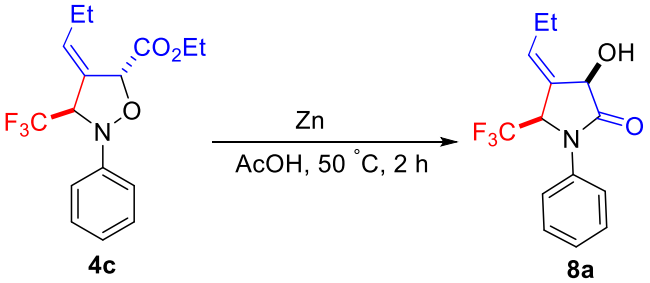
Compound 7: Ethyl-2-hydroxy-3-trifluoromethyl-1-(phenylamino)ethyl)pent-3-enoate

A 10 mL round-bottom flask charged with ethyl-4-ethylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate **4a** (63 mg, 0.20 mmol) was added Zn dust (260 mg, 4.0 mmol, 20 equiv) and acetic acid (2 mL). This reaction mixture was stirred at 25 ˚C for 2 h. After completion of the reaction, as indicated by TLC, the reaction mixture was quenched with water and extracted with ethyl acetate. The combine organic layer was washed with saturated solution of $NaHCO_3$ and brine, dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The residue was purified using column chromatography



(100-200 mesh silica gel) using ethyl acetate/hexane afforded the product **7** as liquid (49 mg, 78%). R_f (EtOAc/Hexane: 3/7) = 0.60. **^{13}C NMR** (100 MHz, δ ppm/$CDCl_3$): 173.5 (C), 145.5 (C), 132.0 (CH), 131.0 (C), 129.4 (CH), 129.4 (CH), 125.1 (q, J_{C-F} = 224.1 Hz, C), 119.1 (CH), 113.7 (CH), 113.7 (CH), 68.0 (CH), 62.5 (CH_2) 56.1 (q, J_{C-F} = 24.2 Hz, CH), 14.2 (CH_3), 14.0 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.21-7.17 (m, 2H), 6.80-6.77 (m, 1H), 6.70-6.67 (m, 2H), 6.16-6.10 (m, 1H), 5.14 (s, 1H), 4.74-4.46 (m, 1H), 4.15-4.10 (m, 2H), 4.01-3.97 (m, 1H), 3.36 (s, 1H), 1.84 (d, J = 5.2 Hz, 3H), 1.18-1.16 (m, 3H). **^{19}F NMR** (376 MHz, δ ppm/$CDCl_3$): -73.4 (s). **HRMS** for $C_{15}H_{19}F_3NO_3^+$: calcd. $[M+H]^+$: 318.1312, found: 318.1309.

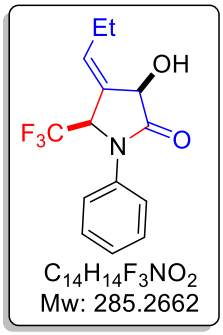
General procedure for the synthesis of γ-lactam 8



4c **8a**

A 10 mL round-bottom flask charged with ethyl-2-phenyl-4-propylidene-3-(trifluoromethyl)isoxazolidine-5-carboxylate **4c** (66 mg, 0.20 mmol) was added Zn dust (260 mg, 4.0 mmol, 20 equiv) and acetic acid (2 mL). This reaction mixture was stirred at 50 °C for 2 h. After completion of the reaction, as indicated by TLC, the reaction mixture was quenched with water and extracted with ethyl acetate. The combine organic layer was washed with saturated solution of $NaHCO_3$ and brine, dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The residue was purified using column chromatography (100-200 mesh silica gel) using ethyl acetate/hexane as the eluent.

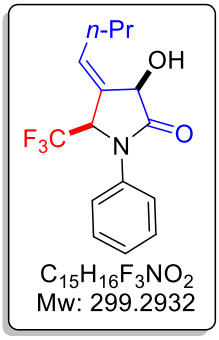
Compound 8a: 3-Hydroxy-1-phenyl-4-propylidene-5-(trifluoromethyl)pyrrolidin-2-one



Following the general procedure, treatment of ethyl 2-phenyl-4-propylidene-3-(trifluoromethyl)isoxazolidine-5-carboxylate **4c** (66 mg, 0.20 mmol) with zinc dust (260 mg, 4.00 mmol) in CH_3COOH (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **8a** as white solid (34 mg, 60%). **Mp** 158-160 °C. R_f (EtOAc/Hexane: 6/4) = 0.38. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 173.3 (C), 140.6 (C), 136.4 (C), 129.4 (CH), 129.4 (CH), 127.8 (CH), 125.2 (CH), 125.2 (CH), 124.4 (CH), 123.7 (q, J_{C-F} = 280.5 Hz, C), 67.8 (CH), 64.0 (q, J_{C-F} = 31.6 Hz, CH), 22.7 (CH_2), 13.6 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.45-7.41 (m, 2H), 7.38-7.35 (m, 2H), 7.34-7.30 (m, 1H), 6.17 (t, J = 7.2 Hz, 1H), 5.06 (q, J_{C-F} = 5.6 Hz, 1H), 4.85 (s, 1H), 3.18 (d, J = 4.4 Hz, 1H), 2.38-

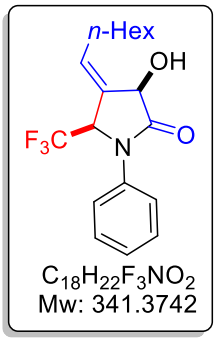
2.35 (m, 2H), 1.12 (t, *J* = 7.6 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -73.7 (s). **HRMS** for $C_{14}H_{15}F_3NO_2^+$: calcd. $[M+H]^+$: 286.1049, found: 286.1050.

Compound 8b: 4-Butylidene-3-hydroxy-1-phenyl-5-(trifluoromethyl)pyrrolidin-2-one



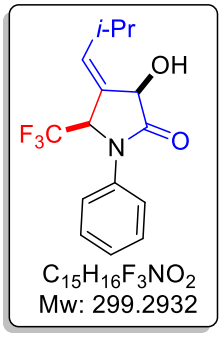
Following the general procedure, treatment of ethyl 4-butylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate **4d** (69 mg, 0.20 mmol) with zinc dust (260 mg, 4.00 mmol) in CH_3COOH (2 mL) at 50 ˚C for 2 h followed by column chromatography afforded the product **8b** as white solid (39 mg, 65%). **Mp** 160-162 ˚C. **R_f** (EtOAc/Hexane: 3/7) = 0.30 **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 173.5 (C), 139.1 (C), 136.4 (C), 129.4 (CH), 129.4 (CH), 127.8 (CH), 125.1 (CH), 125.1 (CH), 125.0 (CH), 123.7 (q, J_{C-F} = 283.0 Hz, C), 67.8 (CH), 64.1 (q, J_{C-F} = 31.7 Hz, CH), 31.1 (CH_2), 22.2 (CH_2), 13.8 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.45-7.41 (m, 2H), 7.38-7.35 (m, 2H), 7.34-7.30 (m, 1H), 6.18 (t, *J* = 7.4 Hz, 1H), 5.02 (q, J_{C-F} = 5.6 Hz, 1H), 4.86 (s, 1H), 3.55 (d, *J* = 4.0 Hz, 1H), 2.38-2.31 (m, 2H), 1.57-1.50 (m, 2H), 0.98 (t, *J* = 7.4 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -73.7 (s). **HRMS** for $C_{15}H_{17}F_3NO_2^+$: calcd. $[M+H]^+$: 300.1206, found: 300.1202.

Compound 8c: 4-Heptylidene-3-hydroxy-1-phenyl-5-(trifluoromethyl)pyrrolidin-2-one



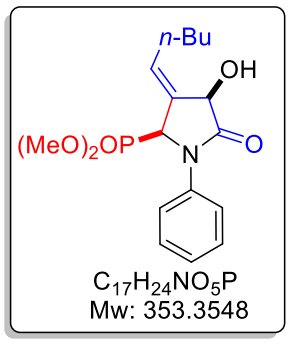
Following the general procedure, treatment of ethyl 4-heptylidene-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate **4g** (77 mg, 0.20 mmol) with zinc dust (260 mg, 4.00 mmol) in CH_3COOH (2 mL) at 50 ˚C for 2 h followed by column chromatography afforded the product **8c** as white solid (47 mg, 69%). **Mp** 170-172 ˚C. **R_f** (EtOAc/Hexane: 3/7) = 0.42 **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 173.5 (C), 139.4 (C), 136.4 (C), 129.4 (CH), 129.4 (CH), 127.8 (CH), 125.1 (CH), 125.1 (CH), 124.8 (CH), 123.7 (q, J_{C-F} = 282.0 Hz, C), 67.8 (CH), 64.0 (q, J_{C-F} = 31.6 Hz, CH), 31.7 (CH_2), 29.2 (CH_2), 29.0 (CH_2), 29.0 (CH_2), 22.7 (CH_2), 14.2 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.45-7.41 (m, 2H), 7.38-7.35 (m, 2H), 7.34-7.30 (m, 1H), 6.17 (t, *J* = 7.4 Hz, 1H), 5.03-4.99 (m, 1H), 4.85 (d, *J* = 3.2 Hz, 1H), 3.28 (d, *J* = 4.4 Hz, 1H), 2.40-2.31 (m, 2H), 1.53-1.49 (m, 2H), 1.39-1.31 (m, 6H), 0.91-0.88 (m, 3H). **^{19}F NMR** (376 MHz, δppm/$CDCl_3$): -73.7 (s). **HRMS** for $C_{18}H_{23}F_3NO_2^+$: calcd. $[M+H]^+$: 342.1675, found: 342.1674.

Compound 8d: 3-Hydroxy-4-(2-methylpropylidene)-1-phenyl-5-(trifluoromethyl)pyrrolidin-2-one



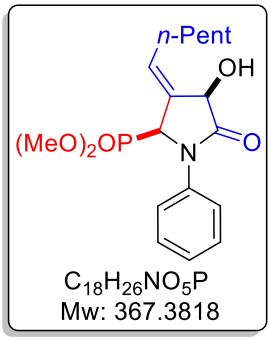
Following the general procedure, treatment of ethyl 4-(2-methylpropylidene)-2-phenyl-3-(trifluoromethyl)isoxazolidine-5-carboxylate **4h** (69 mg, 0.20 mmol) with zinc dust (260 mg, 4.00 mmol) in CH_3COOH (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **8d** as white solid (41 mg, 68%). **Mp** 200-202 °C. **R_f** (EtOAc/Hexane: 3/7) = 0.32. **^{13}C NMR** (100 MHz, δppm/DMSO-d_6): 172.9 (C), 142.4 (C), 136.9 (C), 128.7 (CH), 128.7 (CH), 126.5 (CH), 124.5 (CH), 124.5 (CH), 124.4 (CH), 124.2 (q, J_{C-F} = 282.3 Hz, C), 66.5 (CH), 61.7 (q, J_{C-F} = 31.3 Hz, CH), 27.9 (CH), 22.6 (CH_3), 21.9 (CH_3). **^{1}H NMR** (400 MHz, δppm/DMSO-d_6): 7.50-7.48 (m, 2H), 7.42 (t, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.2 Hz, 1H), 6.30 (d, *J* = 6.0 Hz, 1H), 5.84 (d, J = 10 Hz, 1H), 5.69 (q, J_{C-F} = 6.0 Hz, 1H), 4.71 (d, *J* = 4.8 Hz, 1H), 2.84-2.78 (m, 1H), 1.04 (d, *J* = 2.4 Hz, 3H), 1.02 (d, *J* = 2.4 Hz, 3H). **^{19}F NMR** (376 MHz, δppm/DMSO-d_6): -72.3 **HRMS** for $C_{15}H_{17}F_3NO_2^+$: calcd. $[M+H]^+$: 300.1206, found: 300.1207.

Compound 8e: Dimethyl (4-hydroxy-5-oxo-3-pentylidene-1-phenylpyrrolidin-2-yl)phosphonate



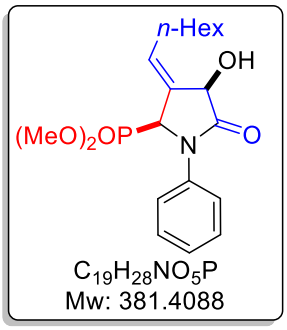
Following the general procedure, treatment of ethyl 3-(dimethoxyphosphoryl)-4-pentylidene-2-phenylisoxazolidine-5-carboxylate **6d** (79 mg, 0.20 mmol) with zinc dust (260 mg, 4.00 mmol) in CH_3COOH (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **8e** as white solid (35 mg, 50%). **Mp** 112-115 °C. **R_f** (EtOAc/Hexane: 6/4) = 0.32. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 172.6 (C), 137.1 (C), 136.1 (d, J_{C-P} = 8.9 Hz, C), 129.1 (CH), 129.1 (CH), 127.5 (d, J_{C-P} = 7.2 Hz, CH), 126.7 (CH), 123.8 (CH), 123.8 (CH), 68.8 (CH), 60.7 (d, J_{C-P} = 155.2 Hz, CH), 54.1 (d, J_{C-P} = 7.7 Hz, CH_3), 53.8 (d, J_{C-P}= 7.4 Hz, CH_3), 31.4 (d, J_{C-P} = 3.3 Hz, CH_2), 28.8 (d, J_{C-P} = 2.7 Hz, CH_2), 22.5 (CH_2), 14.0 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.51 (d, *J* = 8.0 Hz, 2H), 7.40 (t, *J* = 7.8 Hz, 2H), 7.26-7.22 (m, 1H), 6.05-6.00 (m, 1H), 4.97 (d, *J* = 3.6 Hz, 1H), 4.67 (d, *J* = 10.0 Hz, 1H), 4.33 (d, *J* = 10.0 Hz, 1H), 3.70 (d, *J* = 10.8 Hz, 3H), 3.28 (d, *J* = 10.8 Hz, 3H), 2.37-2.34 (m, 2H), 1.50-1.34 (m, 4H), 0.93 (t, *J* = 7.0 Hz, 3H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 24.0 (s). **HRMS** for $C_{17}H_{25}NO_5P^+$: calcd. $[M+H]^+$: 354.1465, found: 354.1465.

Compound 8f: Dimethyl (3-hexylidene-4-hydroxy-5-oxo-1-phenylpyrrolidin-2-yl)phosphonate



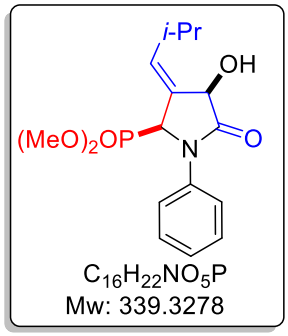
Following the general procedure, treatment of ethyl 3-(dimethoxyphosphoryl)-4-hexylidene-2-phenylisoxazolidine-5-carboxylate **6e** (82 mg, 0.20 mmol) with zinc dust (260 mg, 4.00 mmol) in CH_3COOH (2 mL) at 50 ˚C for 2 h followed by column chromatography afforded the product **8f** as white solid (43 mg, 58%). **Mp** 110-115 ˚C. **R_f** (EtOAc/Hexane: 6/4) = 0.36. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 172.6 (C), 137.1 (C), 136.2 (d, J_{C-P} = 8.5 Hz, C), 129.1 (CH), 129.1 (CH), 127.4 (d, J_{C-P} = 8.6 Hz, CH), 126.6 (CH), 123.8 (CH), 123.8 (CH), 68.8 (CH), 60.7 (d, J_{C-P} = 155.0 Hz, CH), 54.1 (d, J_{C-P} = 7.2 Hz, CH_3), 53.8 (d, J_{C-P}= 6.7 Hz, CH_3), 31.5 (CH_2), 29.0 (CH_2), 29.0 (CH_2), 22.6 (CH_2), 14.1 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.51 (d, *J* = 8.0 Hz, 2H), 7.40 (t, *J* = 7.8 Hz, 2H), 7.26-7.21 (m, 1H), 6.05-6.00 (m, 1H), 4.97 (d, *J* = 3.6 Hz, 1H), 4.67 (d, *J* = 9.6 Hz, 1H), 4.36 (d, *J* = 9.6 Hz, 1H), 3.70 (d, *J* = 10.8 Hz, 3H), 3.27 (d, *J* = 10.8 Hz, 3H), 2.34-2.32 (m, 2H), 1.51-1.45 (m, 2H), 1.34-1.33 (m, 4H), 0.91-0.88 (m, 3H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 24.0 (s). **HRMS** for $C_{18}H_{27}NO_5P^+$: calcd. $[M+H]^+$: 368.1621, found: 368.1620.

Compound 8g: Dimethyl (3-heptylidene-4-hydroxy-5-oxo-1-phenylpyrrolidin-2-yl)phosphonate



Following the general procedure, treatment of ethyl 3-(dimethoxyphosphoryl)-4-heptylidene-2-phenylisoxazolidine-5-carboxylate **6f** (85 mg, 0.20 mmol) with zinc dust (260 mg, 4.00 mmol) in CH_3COOH (2 mL) at 50 ˚C for 2 h followed by column chromatography afforded the product **8g** as white solid (40 mg, 52%). **Mp** 120-122 ˚C. **R_f** (EtOAc/Hexane: 6/4) = 0.36. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 172.6 (C), 137.1 (C), 136.2 (d, J_{C-P} = 7.1 Hz, C), 129.1 (CH), 129.1 (CH), 127.5 (d, J_{C-P} = 5.5 Hz, CH), 126.7 (CH), 123.8 (CH), 123.8 (CH), 68.9 (CH), 60.7 (d, J_{C-P} = 124.0 Hz, CH), 54.1 (d, J_{C-P} = 6.1 Hz, CH_3), 53.5 (d, J_{C-P}= 7.7 Hz, CH_3), 31.8 (CH_2), 29.3 (d, J_{C-P} = 2.6 Hz, CH_2), 29.1 (CH_2), 29.0 (d, J_{C-P} = 2.1 Hz, CH_2), 22.7 (CH_2), 14.2 (CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.51 (d, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 8.0 Hz, 2H), 7.26-7.22 (m, 1H), 6.05-6.00 (m, 1H), 4.98 (d, *J* = 3.6 Hz, 1H), 4.67 (d, *J* = 10.4 Hz, 1H), 4.33 (d, *J* = 10.4 Hz, 1H), 3.70 (d, *J* = 10.8 Hz, 3H), 3.28 (d, *J* = 10.8 Hz, 3H), 2.38-2.31 (m, 2H), 1.51-1.25 (m, 8H), 0.89 (t, *J* = 6.8 Hz, 3H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 24.0 (s). **HRMS** for $C_{19}H_{29}NO_5P^+$: calcd. $[M+H]^+$: 382.1776, found: 382.1776.

Compound 8h: Dimethyl (4-hydroxy-3-(2-methylpropylidene)-5-oxo-1-phenylpyrrolidin-2-yl)phosphonate



Following the general procedure, treatment of ethyl 3-(dimethoxyphosphoryl)-4-(2-methylpropylidene)-2-phenylisoxazolidine-5-carboxylate **6h** (77 mg, 0.20 mmol) with zinc dust (260 mg, 4.00 mmol) in CH_3COOH (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **8h** as white solid (38 mg, 56%). **Mp** 120-122 °C. **R_f** (EtOAc/Hexane: 6/4) = 0.24. **^{13}C NMR** (100 MHz, δppm/$CDCl_3$): 172.5 (C), 142.7 (d, J_{C-P} = 8.8 Hz, C), 137.1 (C), 129.1 (CH), 129.1 (CH), 126.7 (CH), 125.5 (d, J_{C-P} = 6.9 Hz, CH), 123.8 (CH), 123.8 (CH), 68.9 (CH), 60.7 (d, J_{C-P} = 155.1 Hz, CH), 54.4 (d, J_{C-P} = 7.7 Hz, CH_3), 53.7 (d, J_{C-P}= 7.4 Hz, CH_3), 28.8 (d, J_{C-P} = 2.5 Hz, CH), 23.1 (d, J_{C-P} = 4.1 Hz, CH_3), 22.5 (d, J_{C-P} = 2.8 Hz, CH_3). **^{1}H NMR** (400 MHz, δppm/$CDCl_3$): 7.52-7.50 (m, 2H), 7.42-7.38 (m, 2H), 7.26-7.22 (m, 1H), 5.85-5.80 (m, 1H), 4.94-4.93 (m, 1H), 4.68 (d, *J* = 10.8 Hz, 1H), 4.41 (d, *J* = 10.8 Hz, 1H), 3.72 (d, *J* = 10.8 Hz, 3H), 3.28 (d, *J* = 10.8 Hz, 3H), 2.90-2.84 (m, 1H), 1.13 (d, *J* = 6.4 Hz, 3H), 1.07 (d, *J* = 6.8 Hz, 3H). **^{31}P NMR** (161.9 MHz, δppm/$CDCl_3$): 24.2 (s). **HRMS** for $C_{16}H_{23}NO_5P^+$: calcd. $[M+H]^+$: 340.1308, found: 340.1313.

X-ray data collection and structure refinement details of compound 4m:

A good quality colorless single crystal of size 0.20 x 0.14 x 0.07 mm, was selected under a polarizing microscope and was mounted on a glass fiber for data collection. Single crystal X-ray data for compound **4m** were collected on the Rigaku Kappa 3 circle diffractometer equipped with the AFC12 goniometer and enhanced sensitivity (HG) Saturn724+ CCD detector in the 4x4 bin mode using the monochromated Mo-Kα radiation generated from the microfocus sealed tube MicroMax-003 X-ray generator equipped with specially designed confocal multilayer optics. Data collection was performed using ω-scans of 0.5^{o} steps at 293(2) K. Cell determination, data collection and data reduction was performed using the Rigaku CrystalClear-SM Expert 2.1 b24 software. Structure solution and refinement were performed by using SHELXTL-NT. Refinement of coordinates and anisotropic thermal parameters of non-hydrogen atoms were carried out by the full-matrix least-squares method. The hydrogen atoms attached to carbon atoms were generated with idealized geometries and isotropically refined using a riding model.

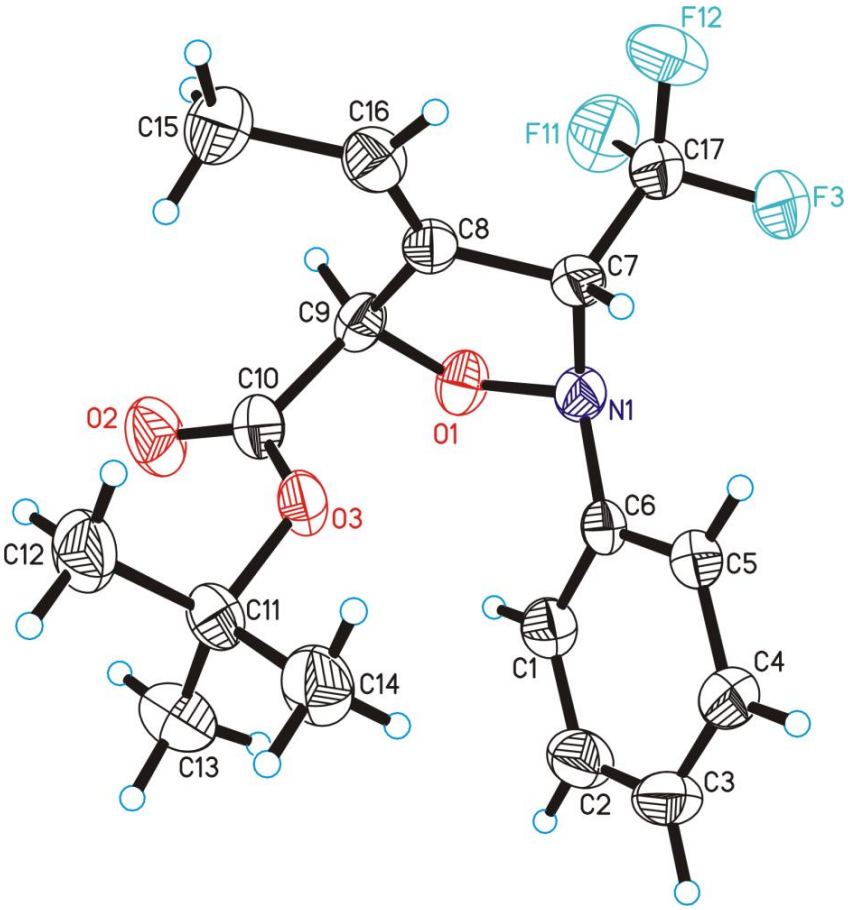


Figure S1 ORTEP diagram drawn with 30% ellipsoid probability for non-H atoms of the crystal structure of compound **4m** determined at 293 K.

Table S2: Crystal data and structure refinement details for compound **4m**

Compound	**4m**
Empirical formula	$C_{17}\ H_{20}\ F_3\ N\ O_3$
Formula weight	343.34
Crystal System	Monoclinic
Space group	$P\ 2_1/c$
a (Å)	11.431(6)
b (Å)	5.931(3)
c (Å)	25.849(14)
α (°)	90.00
β (°)	94.751(10)
γ (°)	90.00
V (Å^3)	1746.5(16)
Z	4
D_c (g/cm^3)	1.306
F_{000}	720
μ (mm^{-1})	0.110
θ_{max} (°)	25.40
Total reflections	8980
Unique reflections	3036
Reflections [$I > 2\sigma(I)$]	1522
Parameters	217
R_{int}	0.0595
Goodness-of-fit	0.912
R [$F^2 > 2\sigma(F^2)$]	0.0596
wR (F^2, all data)	0.1776
CCDC No.	1857117

X-ray data collection and structure refinement details of compound 8a:

A good quality colorless single crystal of size 0.52 x 0.10 x 0.08 mm, was selected under a polarizing microscope and was mounted on a glass fiber for data collection. Single crystal X-ray data for compound **8a** were collected on the Rigaku Kappa 3 circle diffractometer equipped with the AFC12 goniometer and enhanced sensitivity (HG) Saturn724+ CCD detector in the 4x4 bin mode using the monochromated Mo-Kα radiation generated from the microfocus sealed tube MicroMax-003 X-ray generator equipped with specially designed confocal multilayer optics. Data collection was performed using ω-scans of 0.5^o steps at 293(2) K. Cell determination, data collection and data reduction was performed using the Rigaku CrystalClear-SM Expert 2.1 b24 software. Structure solution and refinement were performed by using SHELXTL-NT. Refinement of coordinates and anisotropic thermal parameters of non-hydrogen atoms were carried out by the full-matrix least-squares method. The hydrogen atoms attached to carbon atoms were generated with idealized geometries and isotropically refined using a riding model.

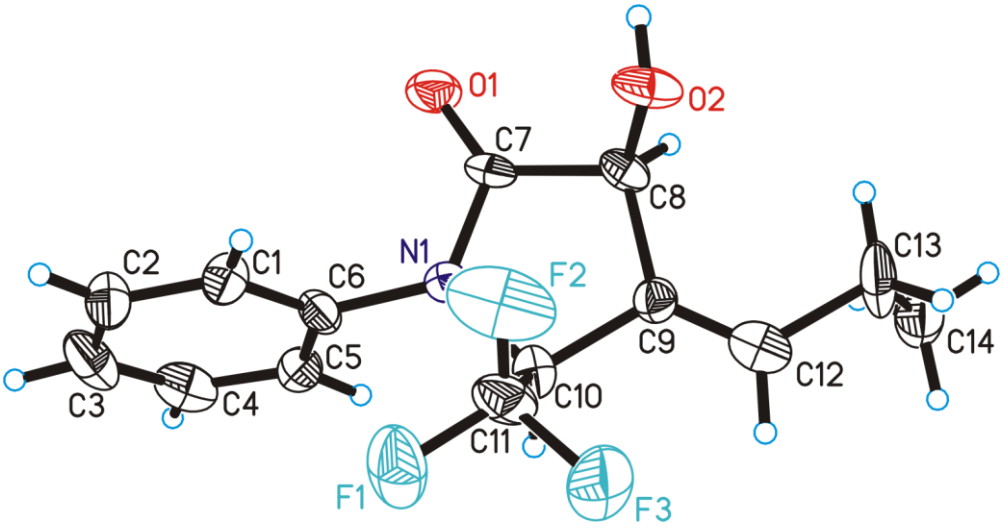
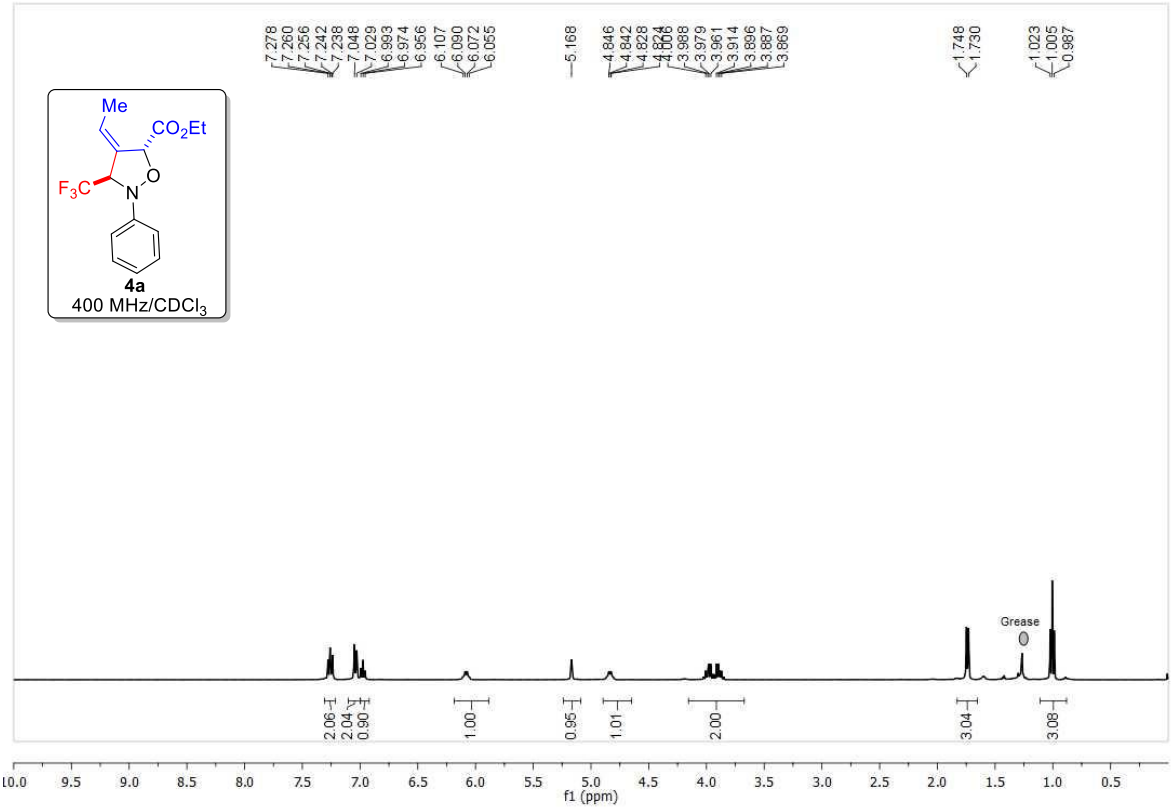


Figure S2 ORTEP diagram drawn with 30% ellipsoid probability for non-H atoms of the crystal structure of compound **8a** determined at 293 K.

Table S3 Crystal data and structure refinement details for compound **8a**

Compound	**8a**
Empirical formula	$C_{14}\ H_{14}\ F_3\ N\ O_2$
Formula weight	285.26
Crystal System	Triclinic
Space group	*P*-1
a (Å)	11.218(3)
b (Å)	12.296(3)
c (Å)	16.820(5)
α (°)	108.32(2)
β (°)	101.98(3)
γ (°)	102.65(2)
V (Å^3)	2051.1(10)
Z	6
D_c (g/cm^3)	1.386
F_{000}	888
μ (mm^{-1})	0.120
θ_{max} (°)	25.44
Total reflections	9236
Unique reflections	5731
Reflections [$I > 2\sigma(I)$]	1340
Parameters	545
R_{int}	0.0800
Goodness-of-fit	0.595
R [$F^2 > 2\sigma(F^2)$]	0.0555
wR (F^2, all data)	0.1588
CCDC No.	1857118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CO2Et
F3C
O
N
4a
400 MHz/CDCl3
7.278
7.260
7.256
7.242
7.238
7.048
7.029
6.993
6.974
6.956
6.107
6.090
6.072
6.055
5.168
4.846
4.842
4.828
4.824
4.008
3.988
3.979
3.961
3.914
3.896
3.887
3.869
1.748
1.730
1.023
1.005
0.987
Grease
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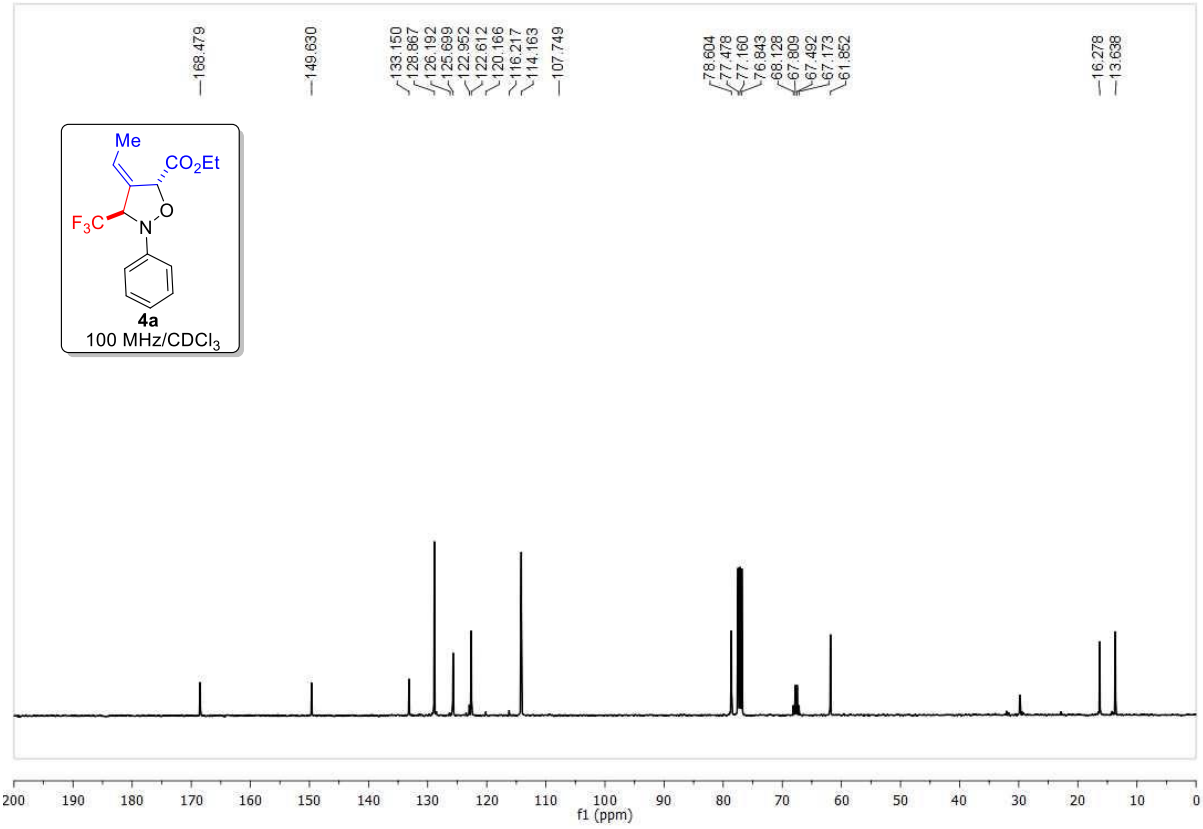
2.04
0.90
1.00
0.95
1.01
2.00
3.04
3.08
f1 (ppm)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O
N
4a
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125.699
122.952
122.612
120.166
116.217
114.163
107.749
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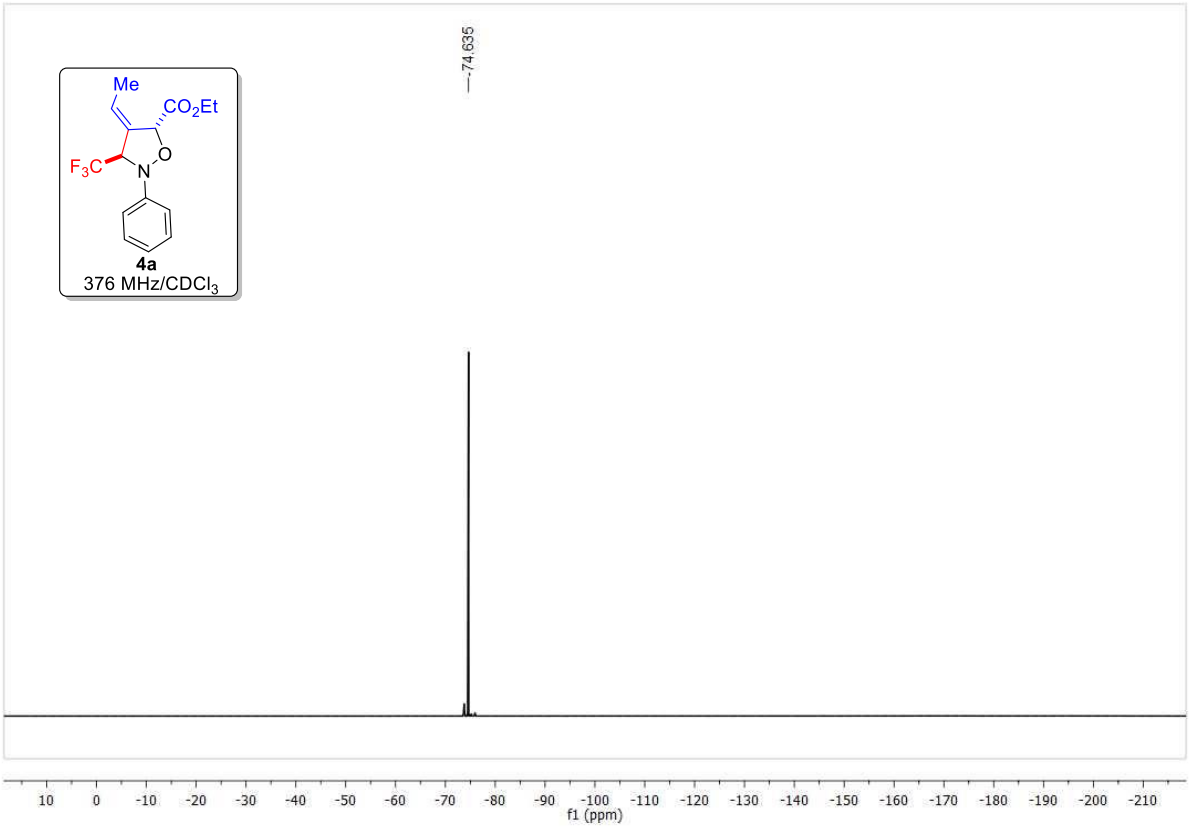
76.843
68.128
67.809
67.492
67.173
61.852
16.278
13.638
f1 (ppm)


Me
CO2Et
F3C
O
N
4a
376 MHz/CDCl3
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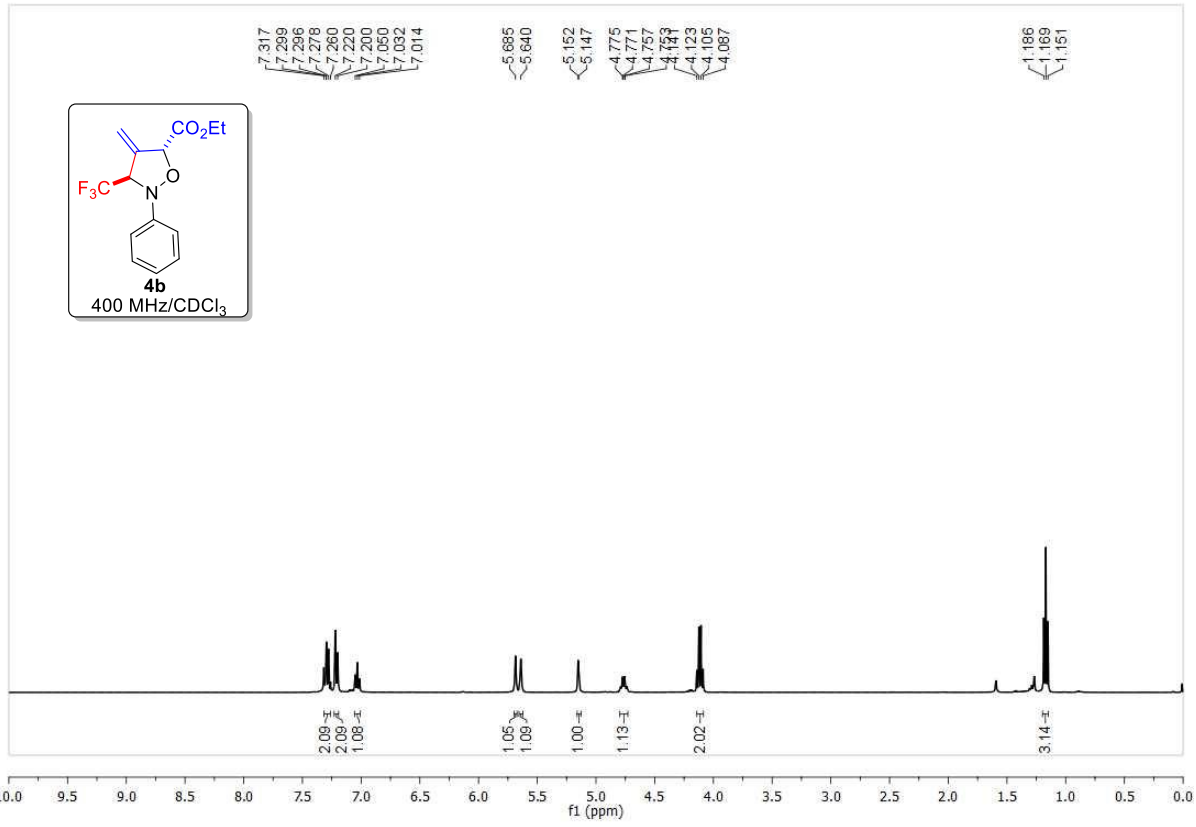
f1 (ppm)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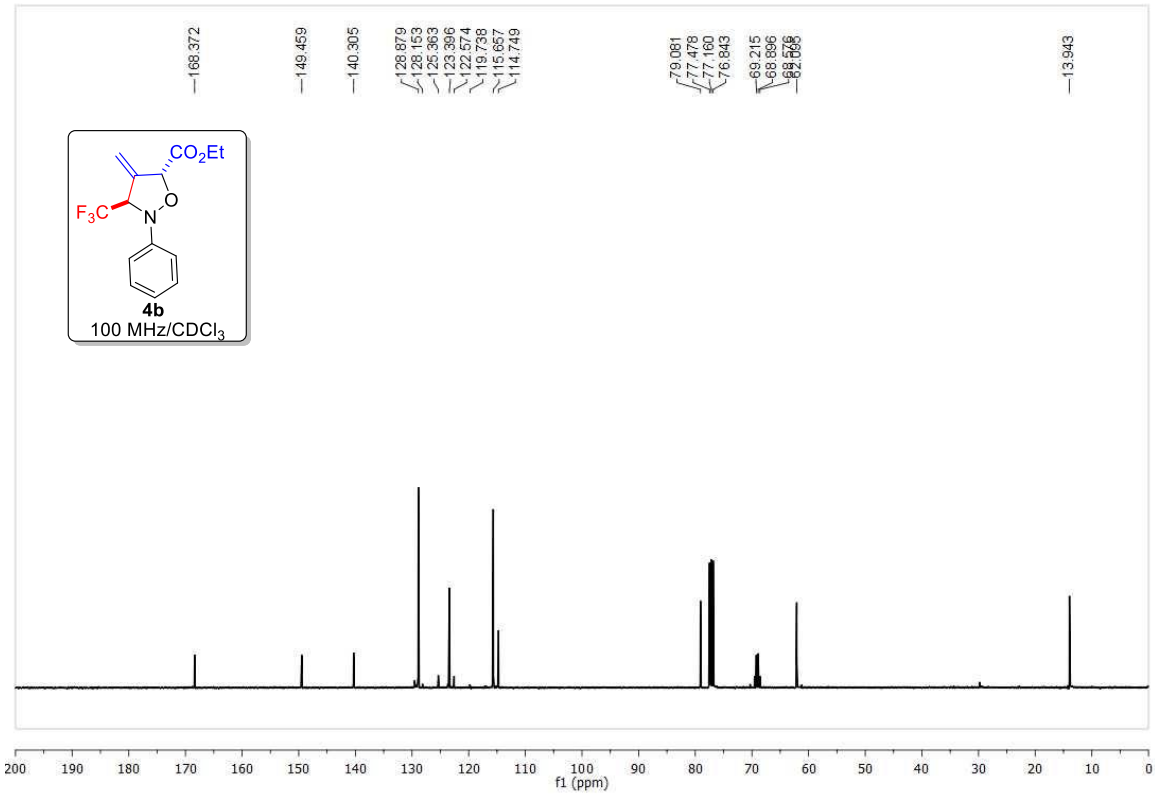
7.220
7.200
7.050
7.032
7.014
5.685
5.640
5.152
5.147
4.775
4.771
4.757
4.123
4.105
4.087
1.186
1.169
1.151
CO2Et
F3C
O
N
4b
400 MHz/CDCl3
2.09
2.09
1.08
1.05
1.09
1.00
1.13
2.02
3.14
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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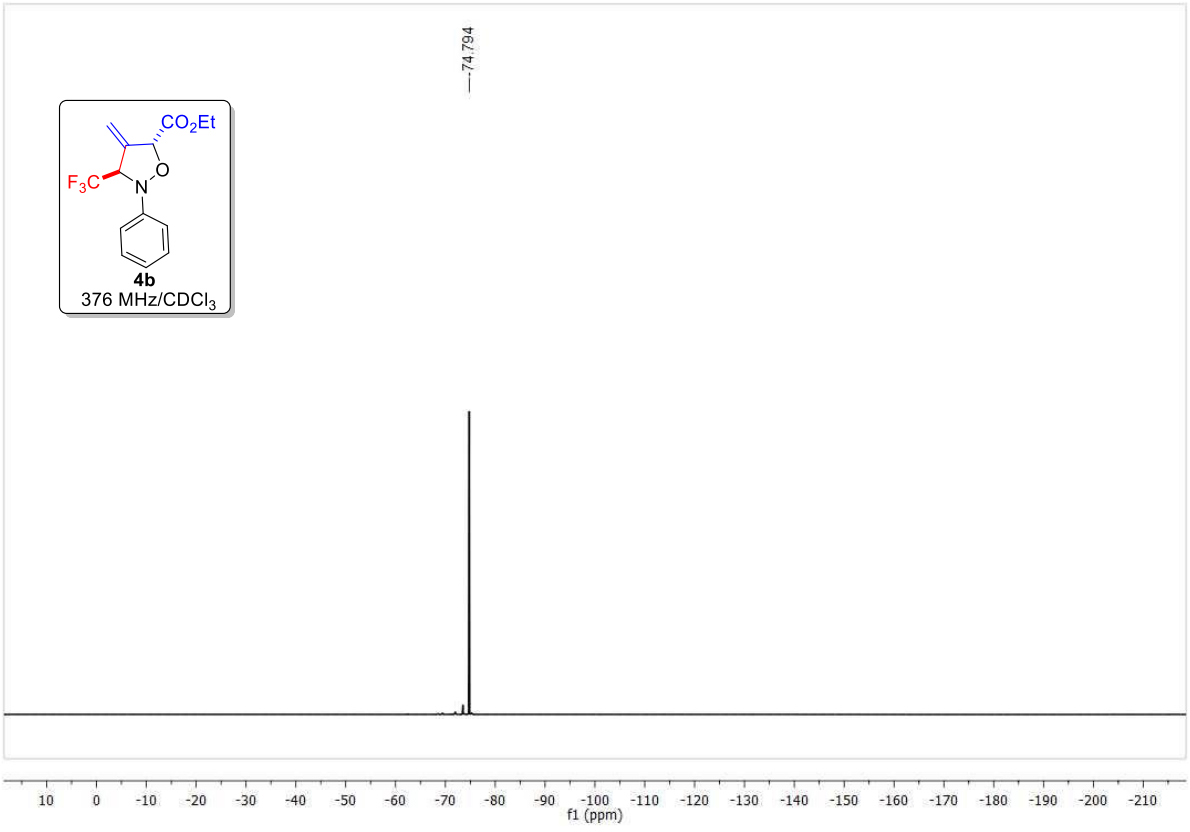
128.153
125.363
123.396
122.574
119.738
115.657
114.749
79.081
77.478
77.160
76.843
69.215
68.896
13.943
CO2Et
F3C
N
O
4b
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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F3C
N
O
4b
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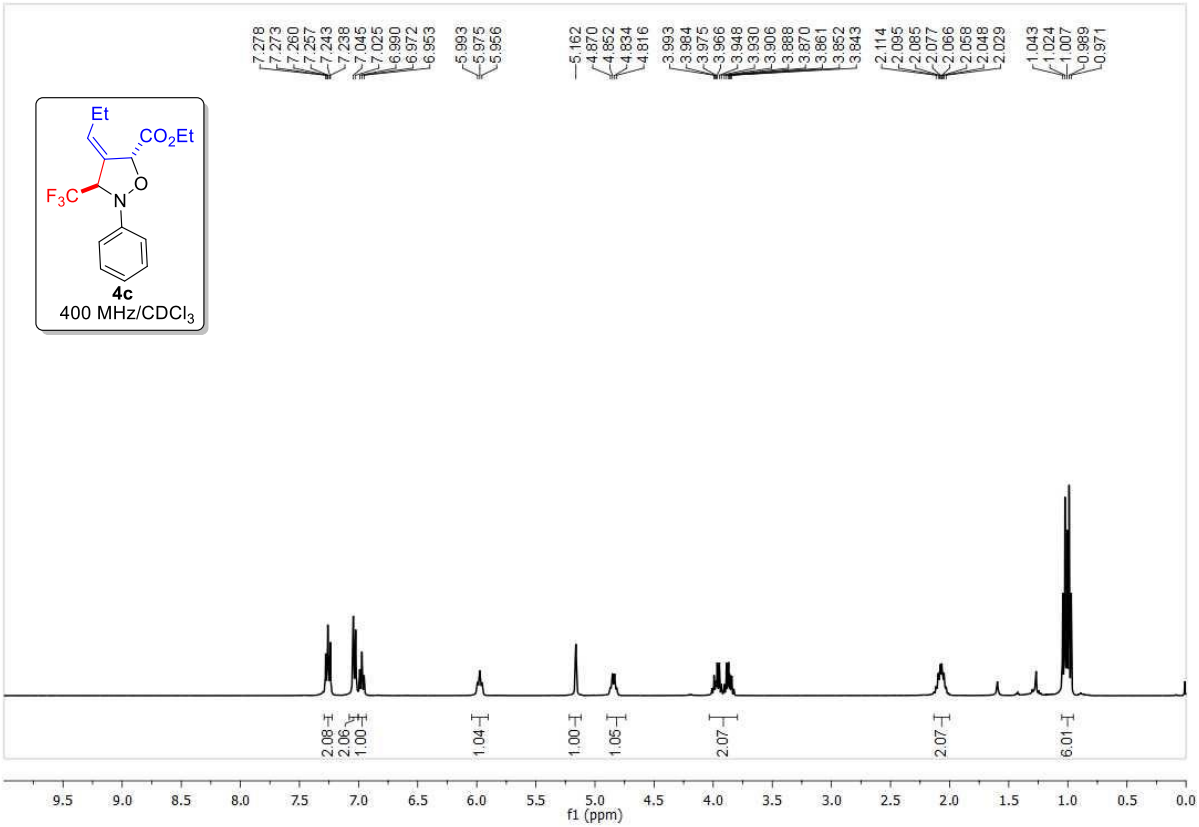
f1 (ppm)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CO2Et
F3C
O
N
4c
400 MHz/CDCl3
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7.045
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6.953
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5.975
5.956
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4.852
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3.948
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3.870
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3.843
2.114
2.095
2.085
2.077
2.066
2.058
2.048
2.029
1.043
1.024
1.007
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2.06
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1.04
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1.05
2.07
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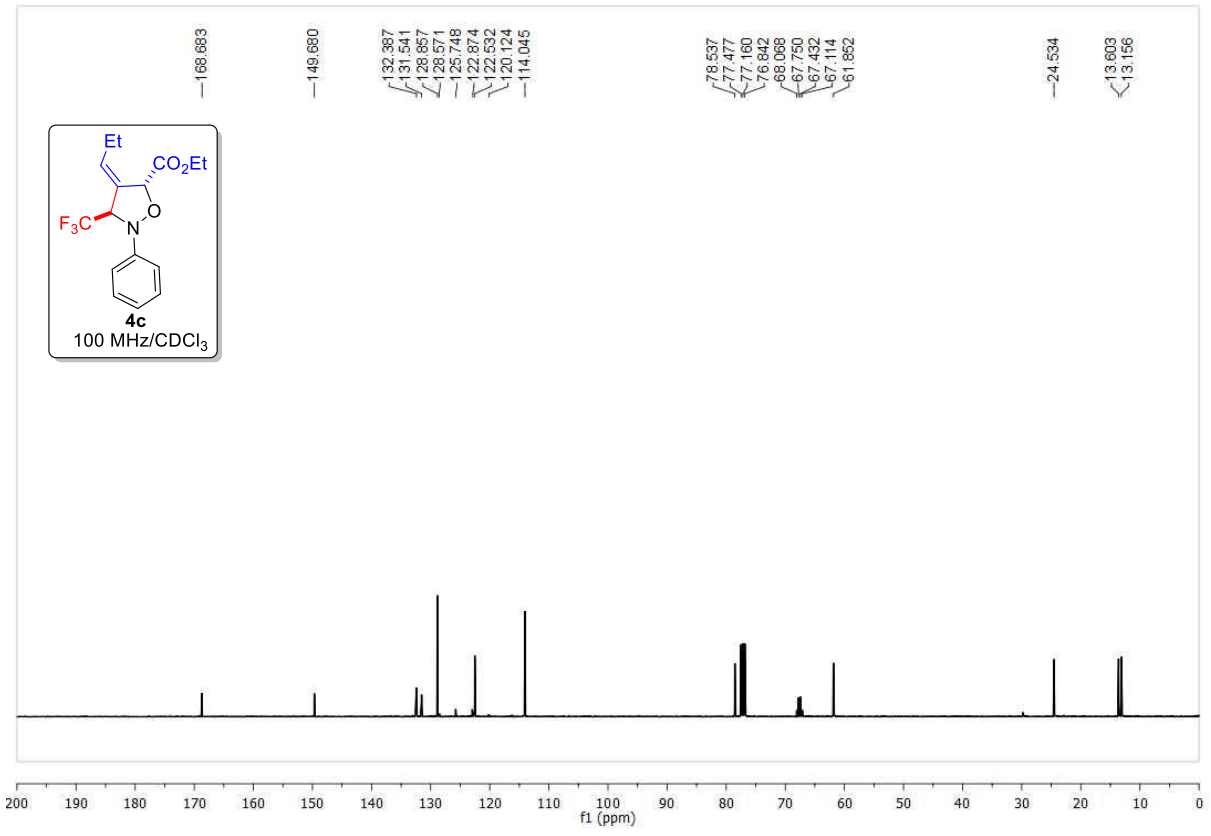
6.01
f1 (ppm)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CO2Et
F3C
O
N
4c
100 MHz/CDCl3
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125.748
122.874
122.532
120.124
114.045
78.537
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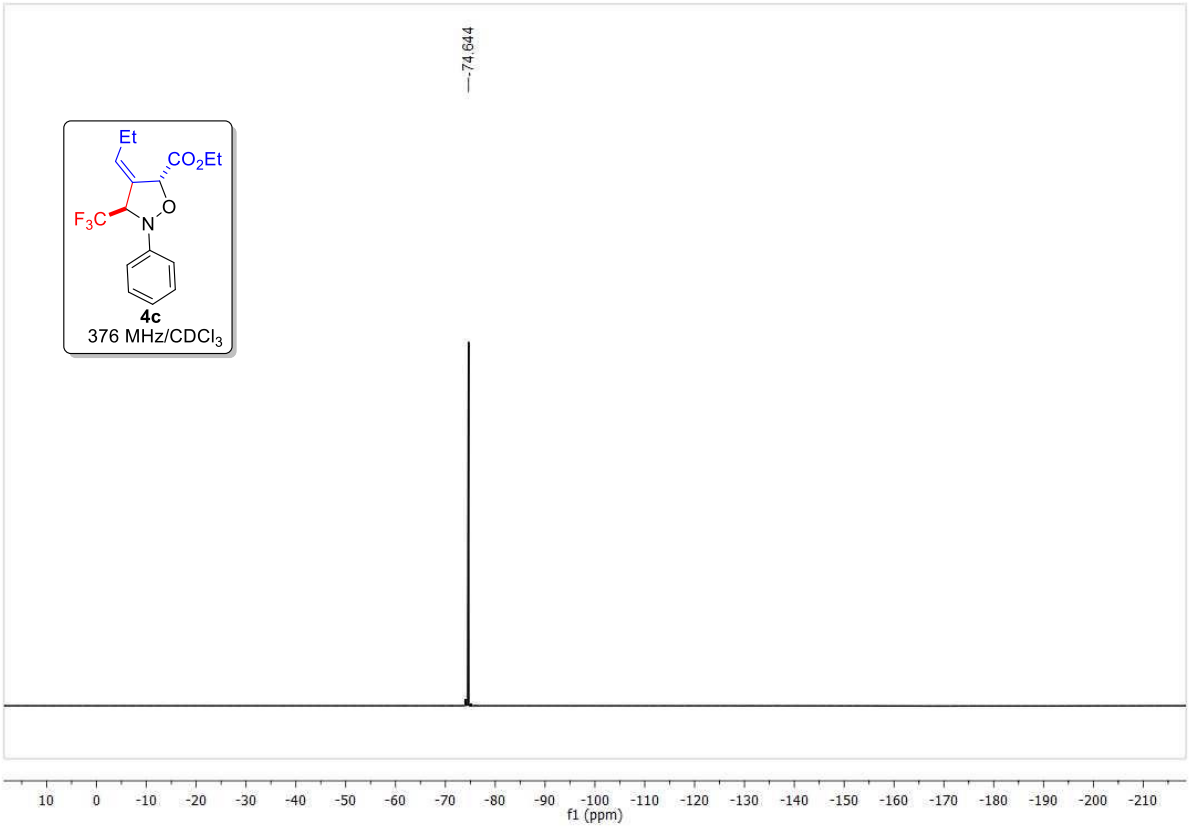
13.156
f1 (ppm)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F3C
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O
4c
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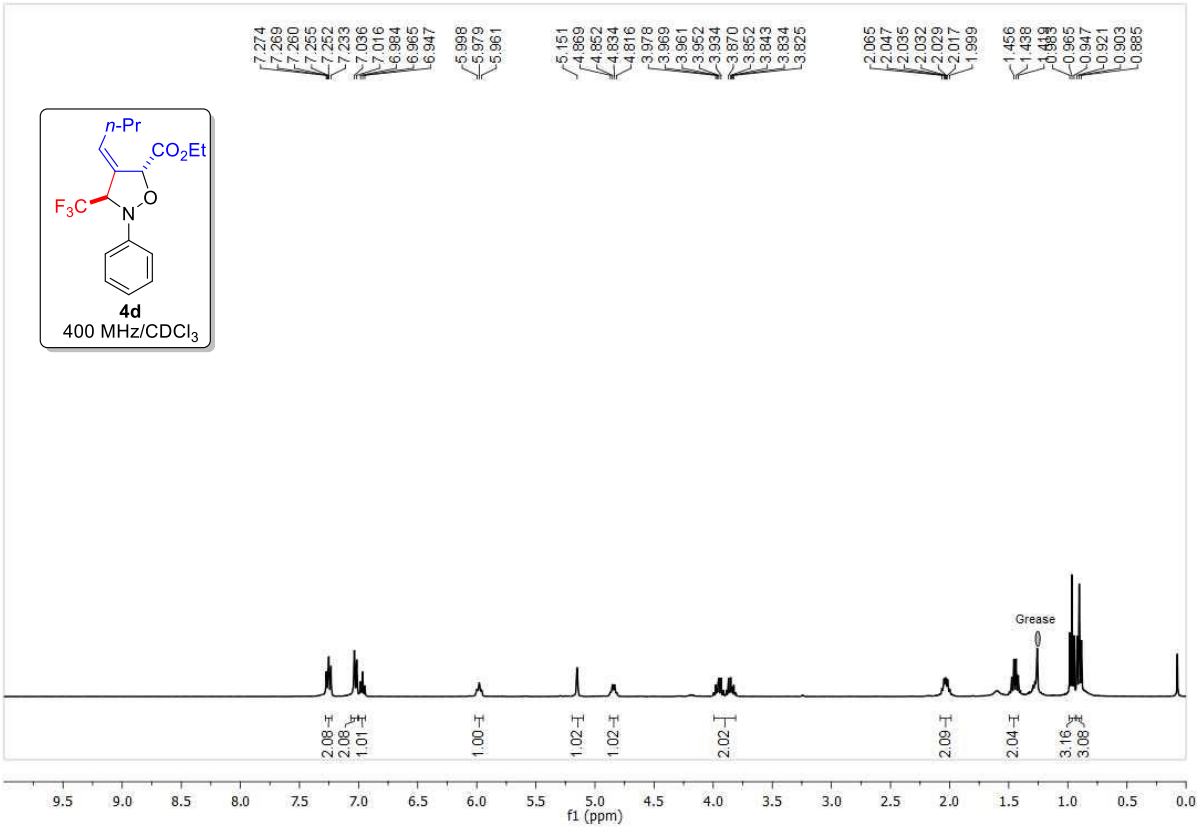
f1 (ppm)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5.998
5.979
5.961
5.151
4.869
4.852
4.834
4.816
3.978
3.969
3.961
3.952
3.934
3.870
3.852
3.843
3.834
3.825
2.065
2.047
2.035
2.032
2.029
2.017
1.999
1.456
1.438
0.965
0.947
0.921
0.903
0.885
n-Pr
CO2Et
F3C
N
O
4d
400 MHz/CDCl3
Grease
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2.08
1.01
1.00
1.02
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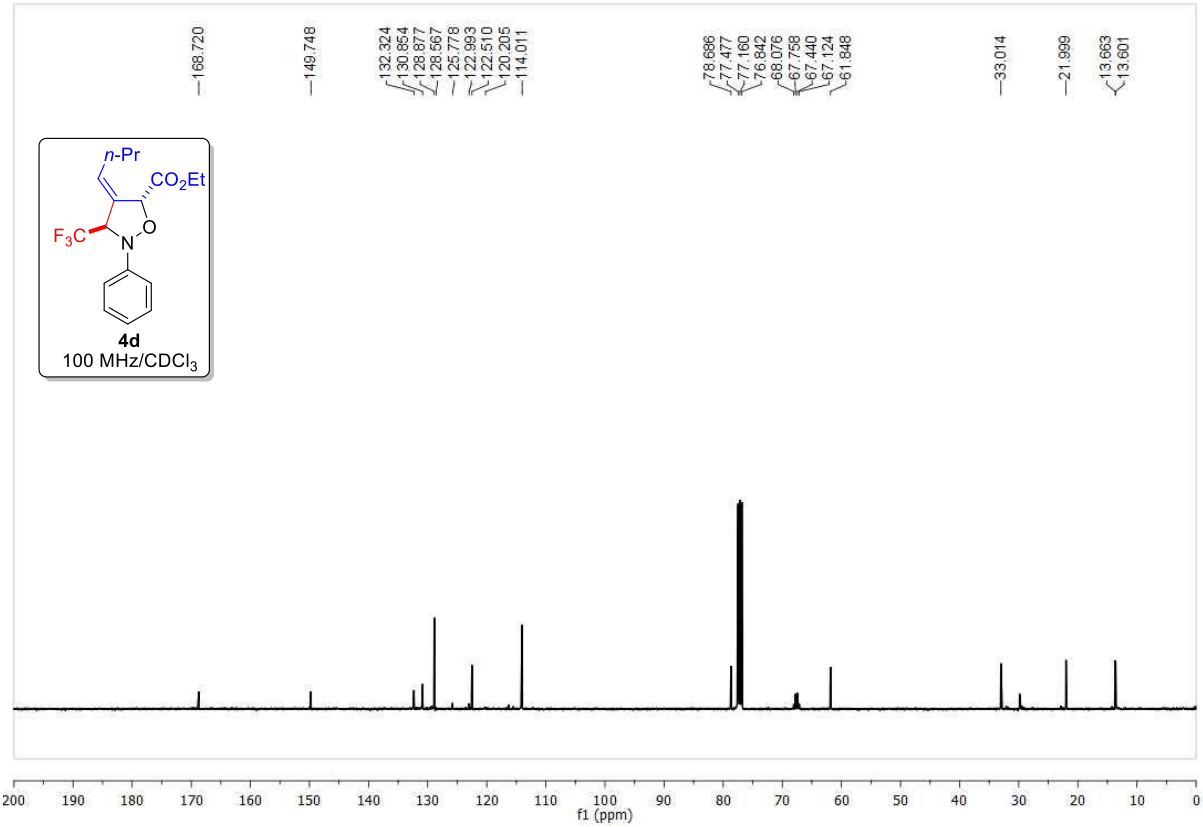
3.16
3.08
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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77.477
77.160
76.842
68.076
67.758
67.440
67.124
61.848
33.014
21.999
13.663
13.601
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N
O
4d
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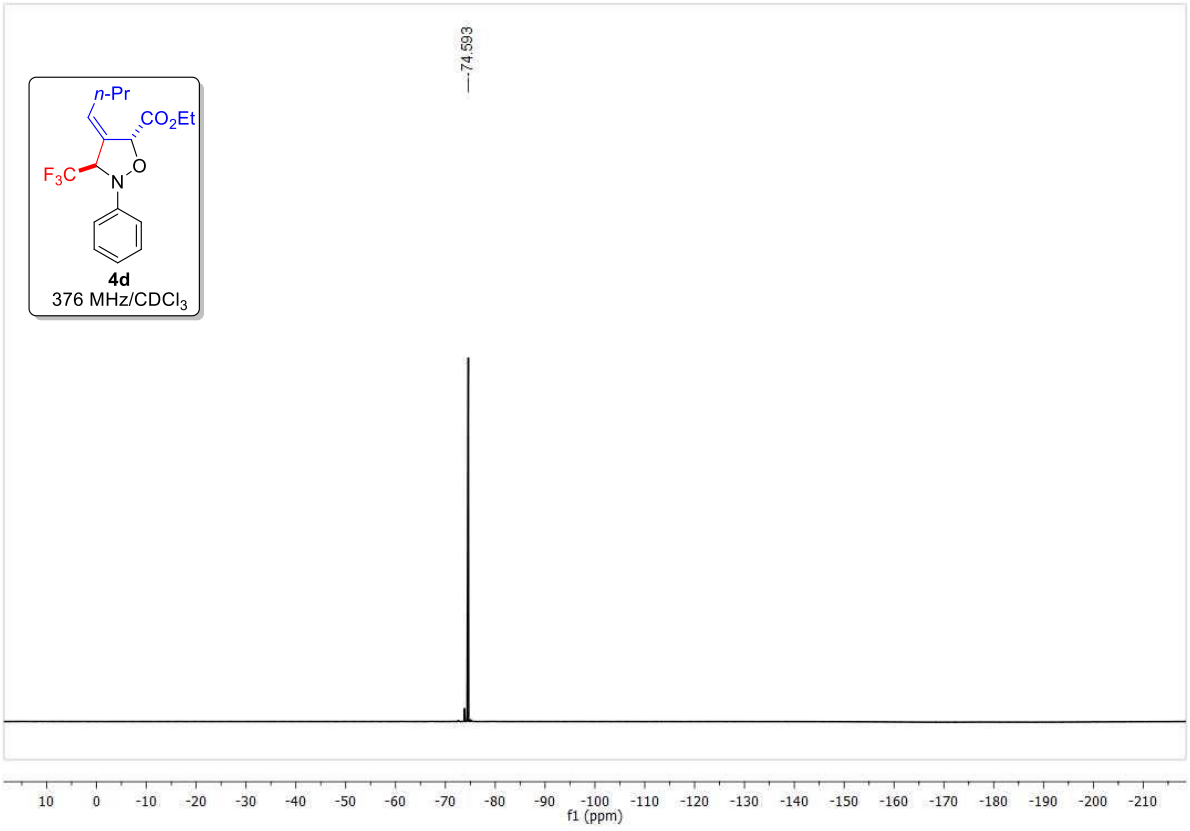
f1 (ppm)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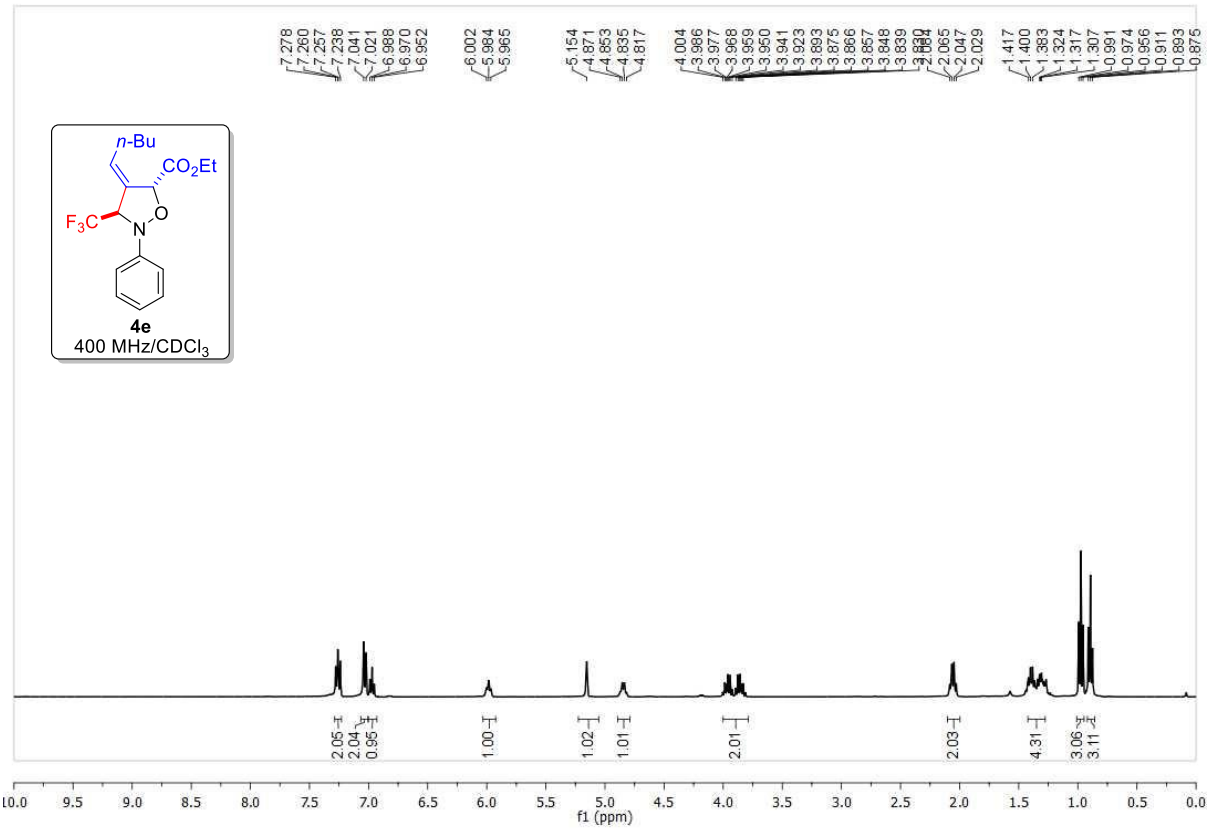
f1 (ppm)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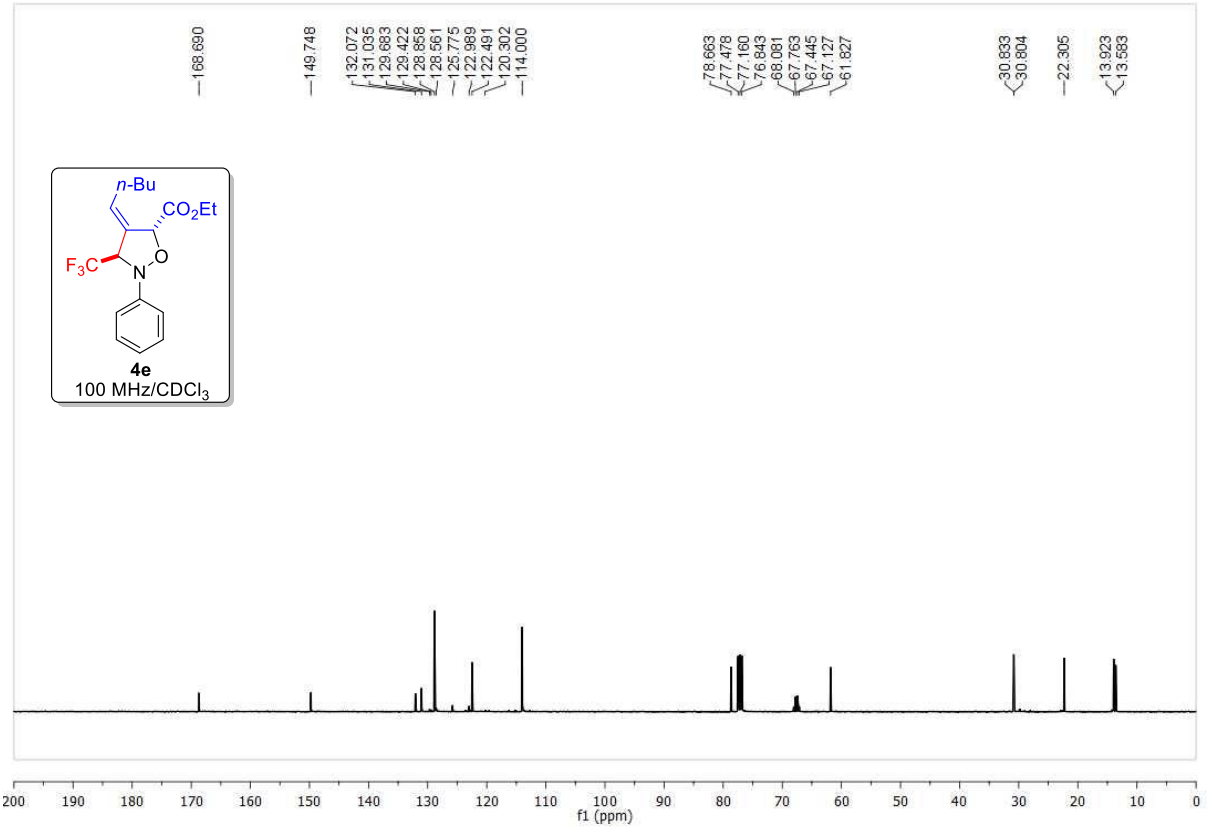
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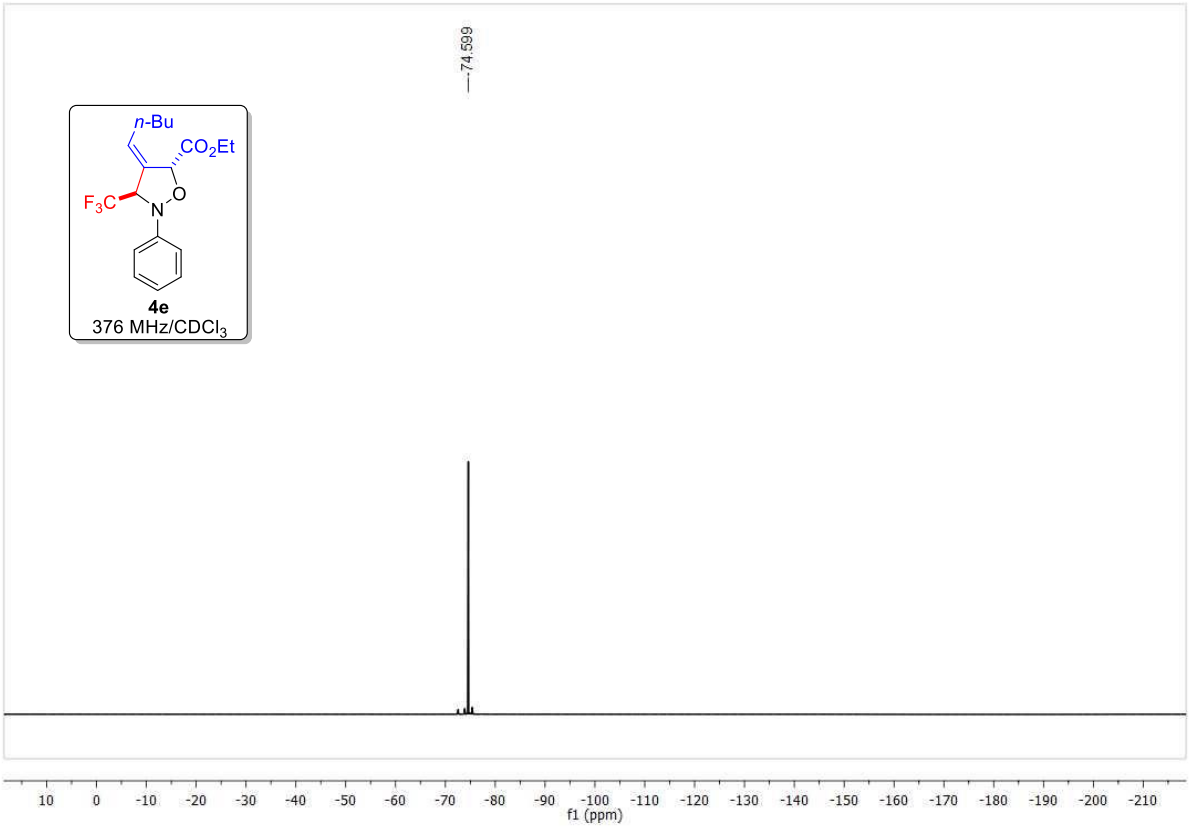
CO2Et
F3C
N
O
4e
100 MHz/CDCl3
f1 (ppm)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n-Bu
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F3C
N
O
4e
376 MHz/CDCl3
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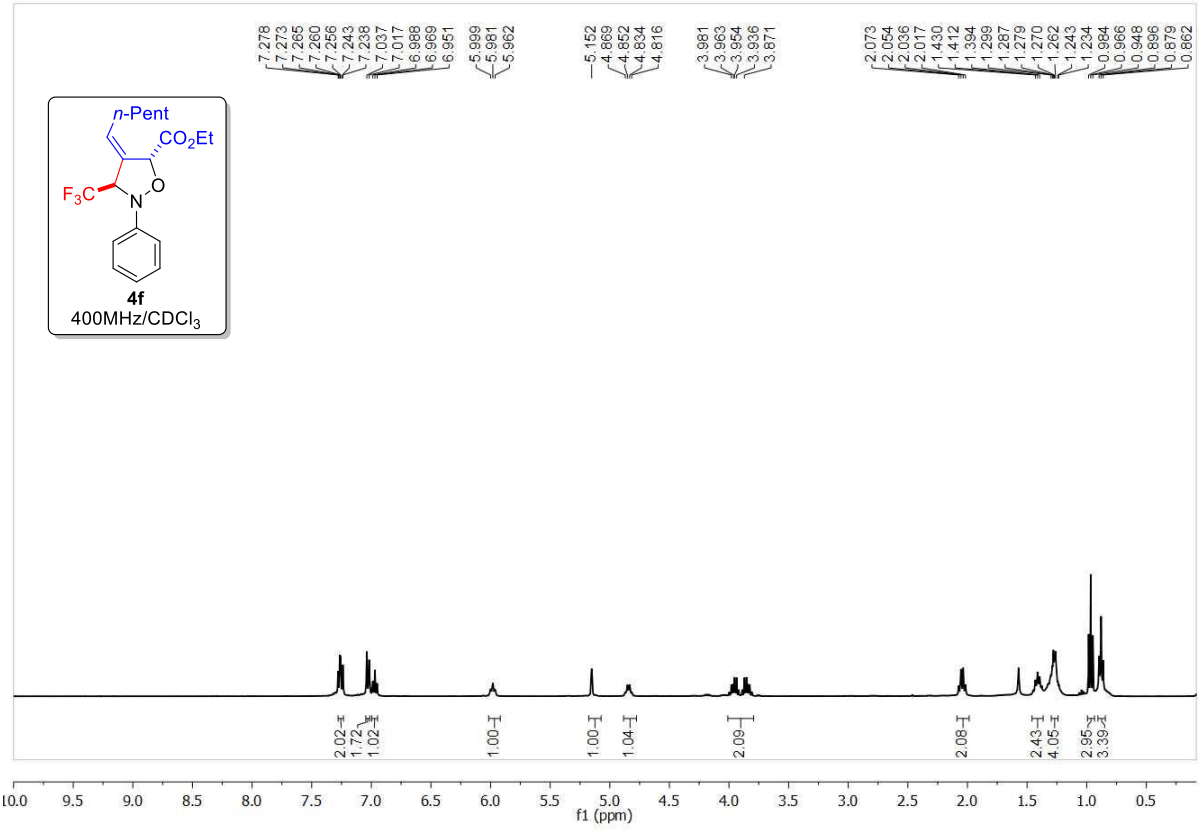
f1 (ppm)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5.981
5.962
5.152
4.869
4.852
4.834
4.816
3.981
3.963
3.954
3.936
3.871
2.073
2.054
2.036
2.017
1.430
1.412
1.394
1.299
1.287
1.279
1.270
1.262
1.243
1.234
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0.966
0.948
0.896
0.879
0.862
n-Pent
CO2Et
F3C
N
O
4f
400MHz/CDCl3
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1.72
1.02
1.00
1.00
1.04
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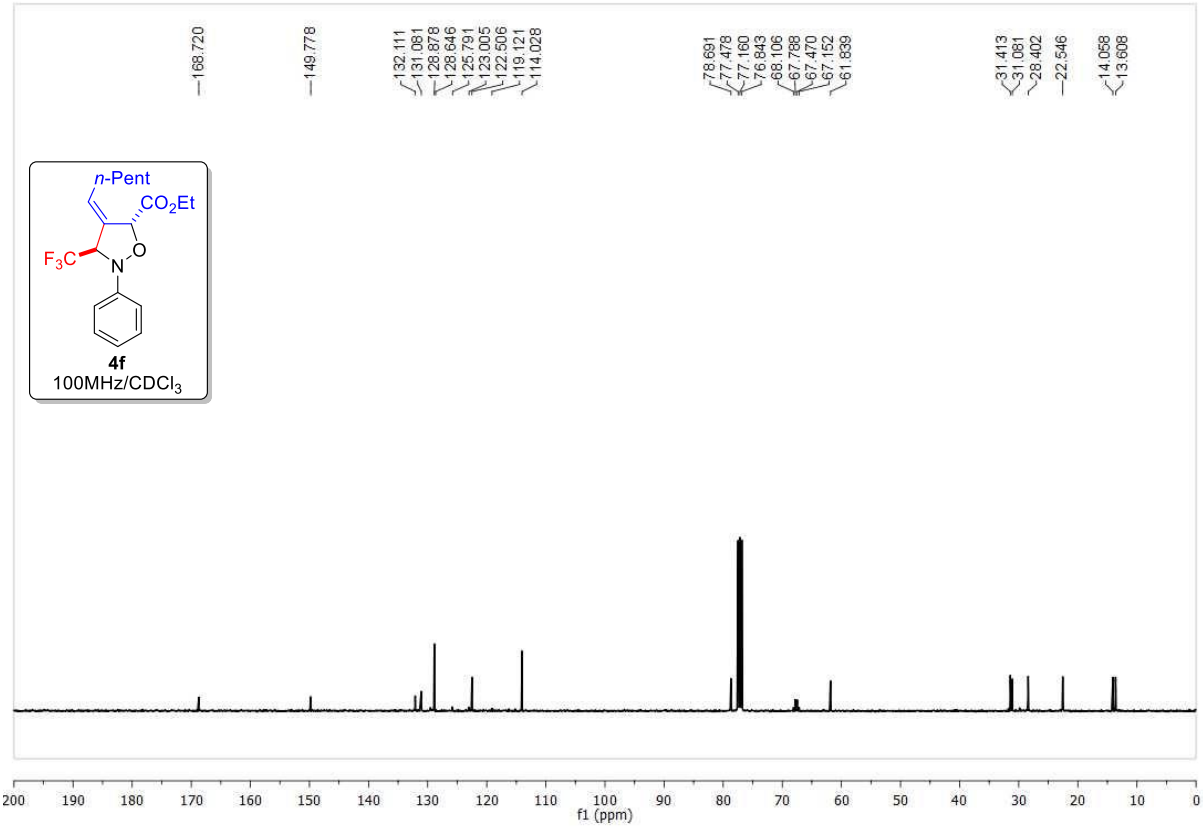
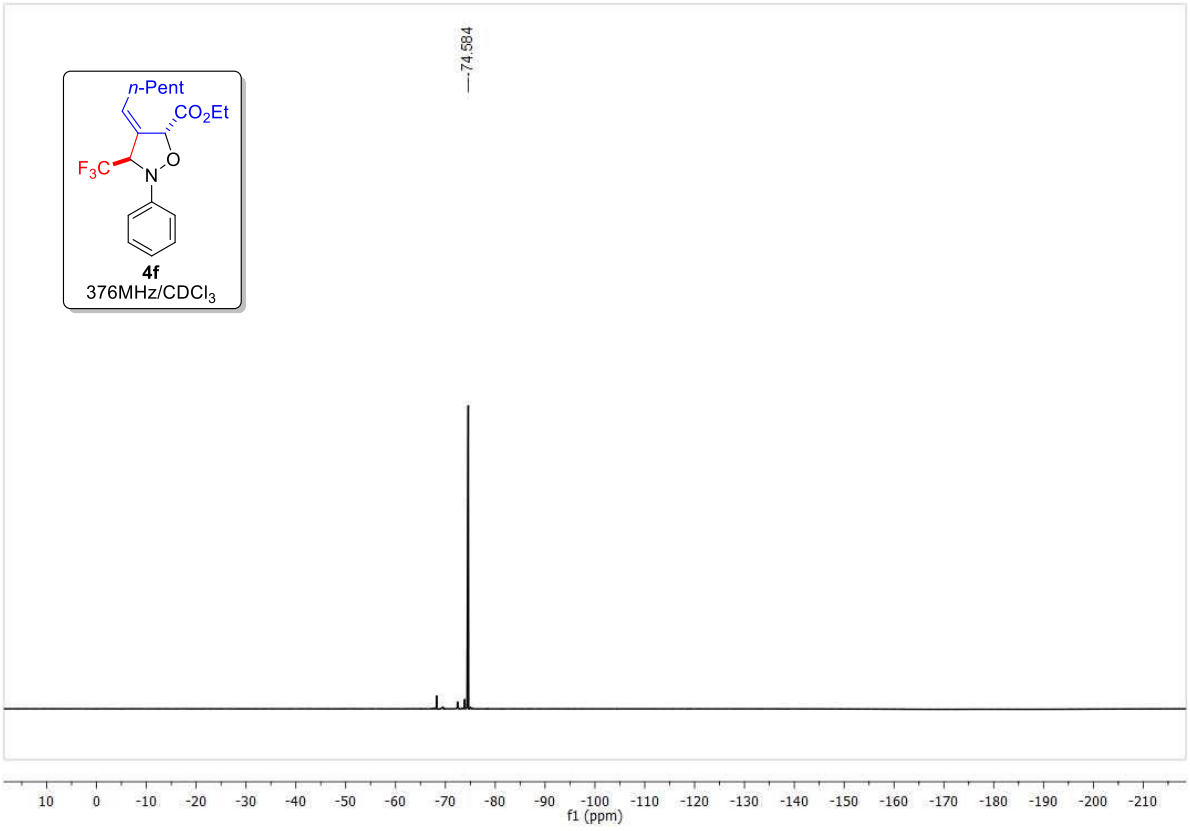
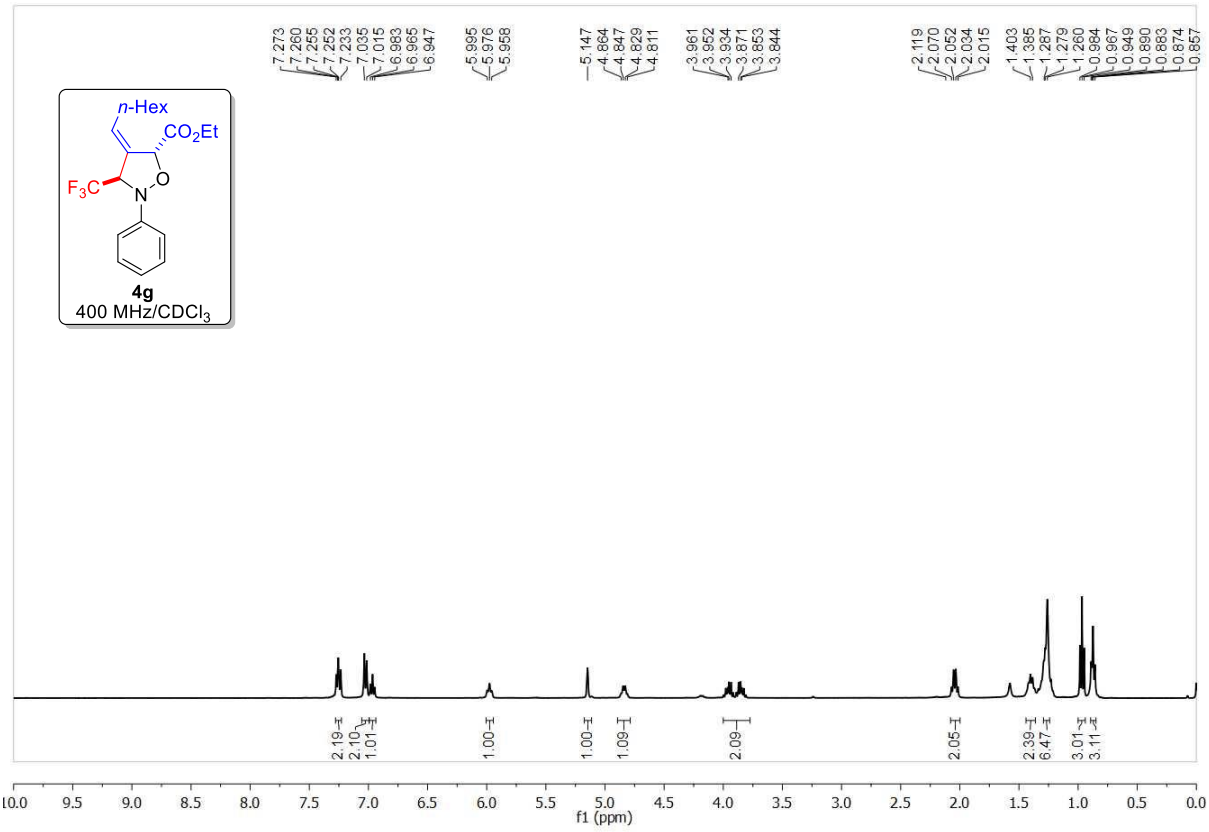
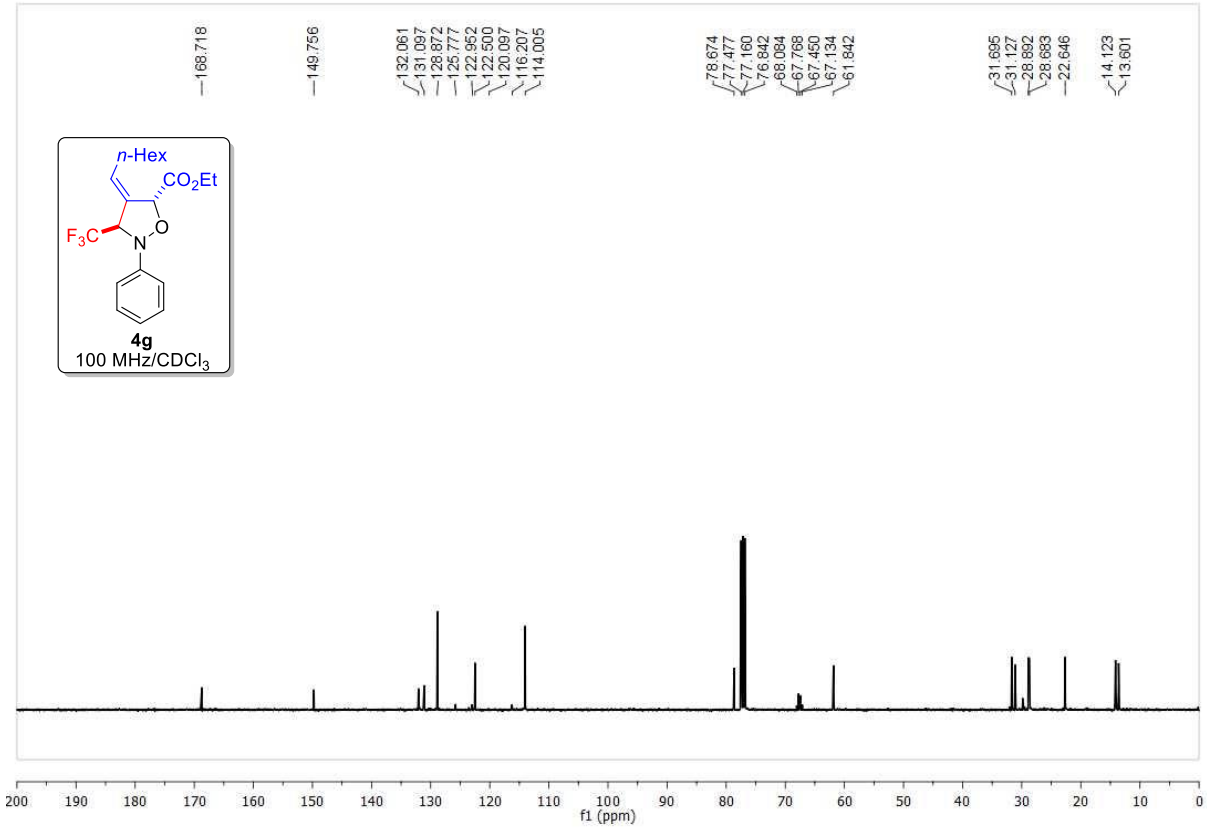
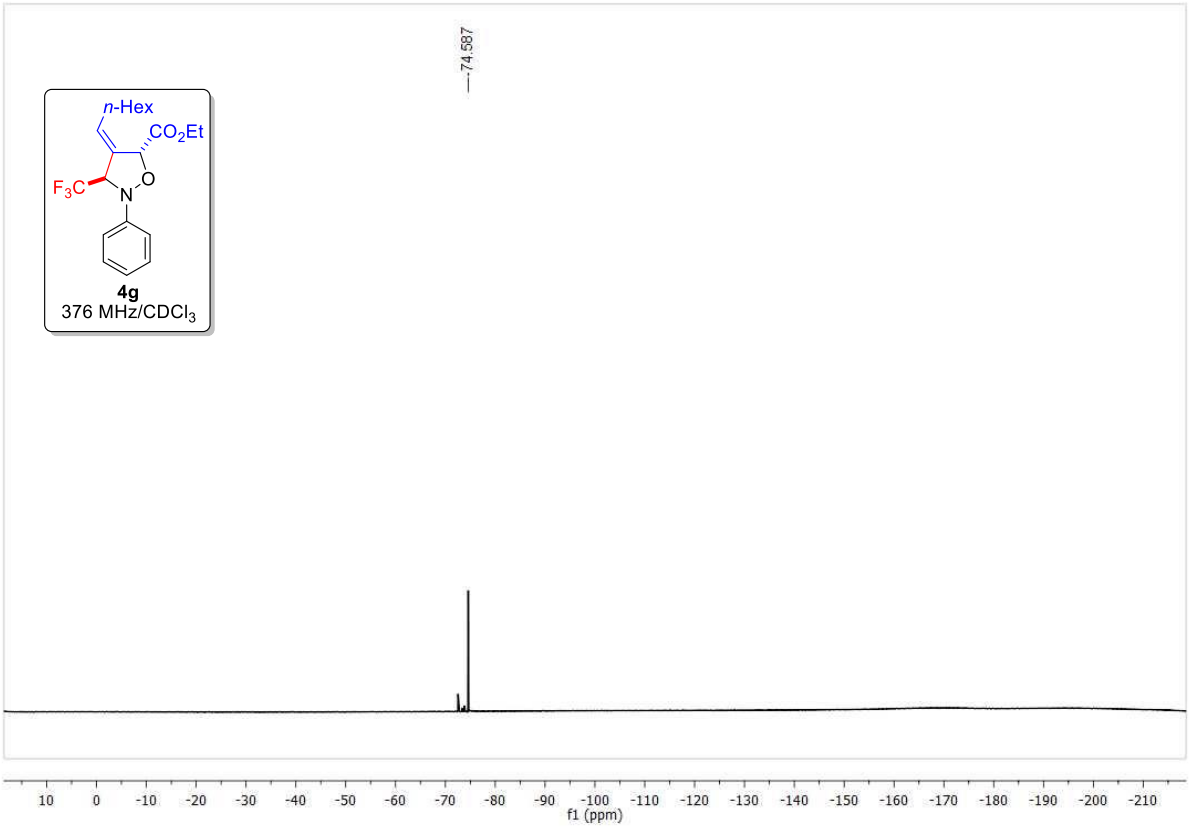
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

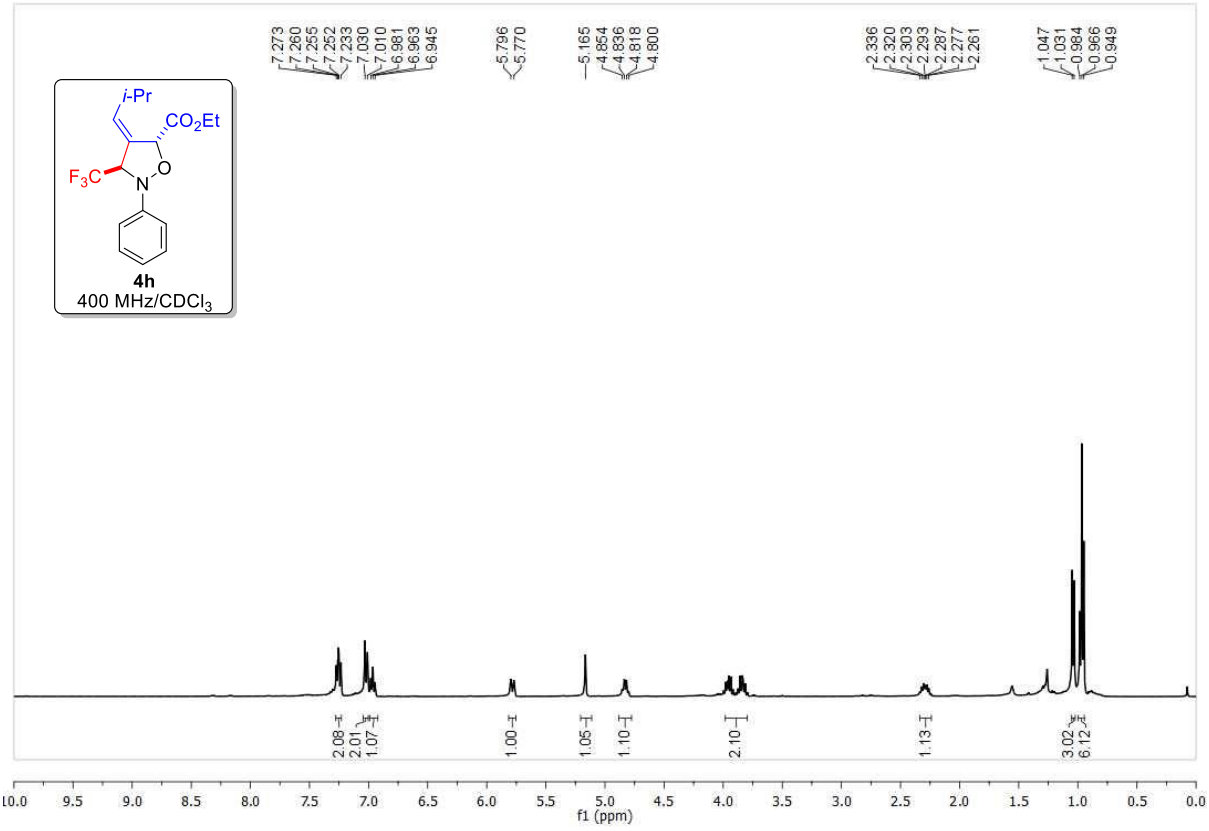

168.720
149.778
132.111
131.081
128.878
128.646
125.791
123.005
122.506
119.121
114.028
78.691
77.478
77.160
76.843
68.106
67.788
67.470
67.152
61.839
31.413
31.081
28.402
22.546
14.058
13.608
n-Pent
CO2Et
F3C
N
O
4f
100MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

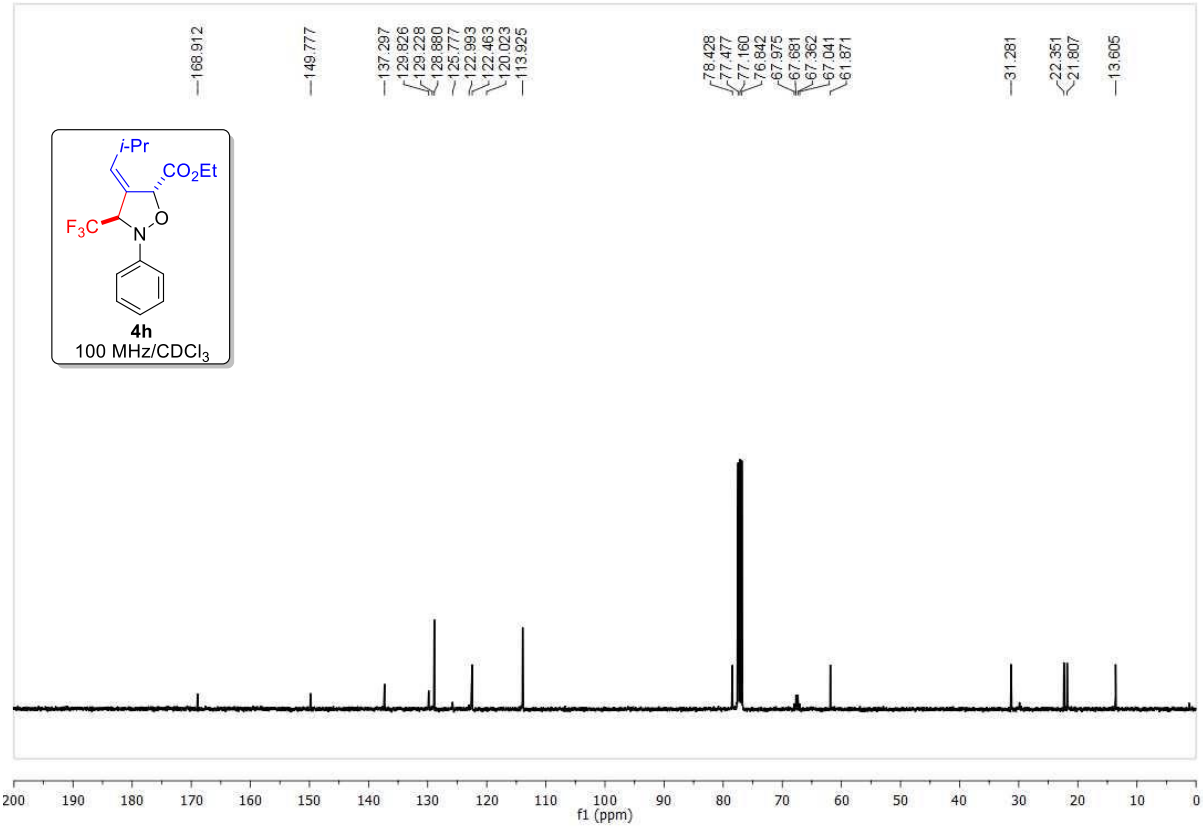

74.584
n-Pent
CO2Et
F3C
N
O
4f
376MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

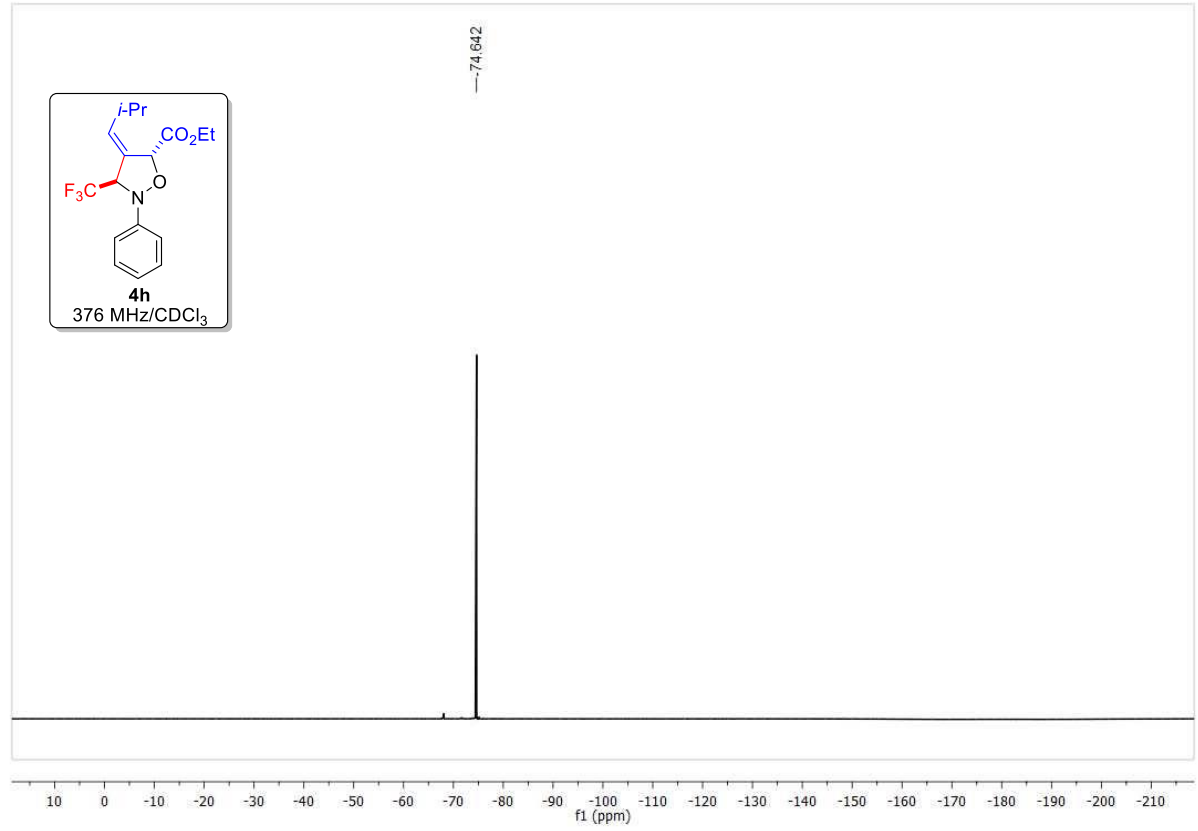

n-Hex
CO2Et
F3C
O
N
4g
400 MHz/CDCl3
f1 (ppm)

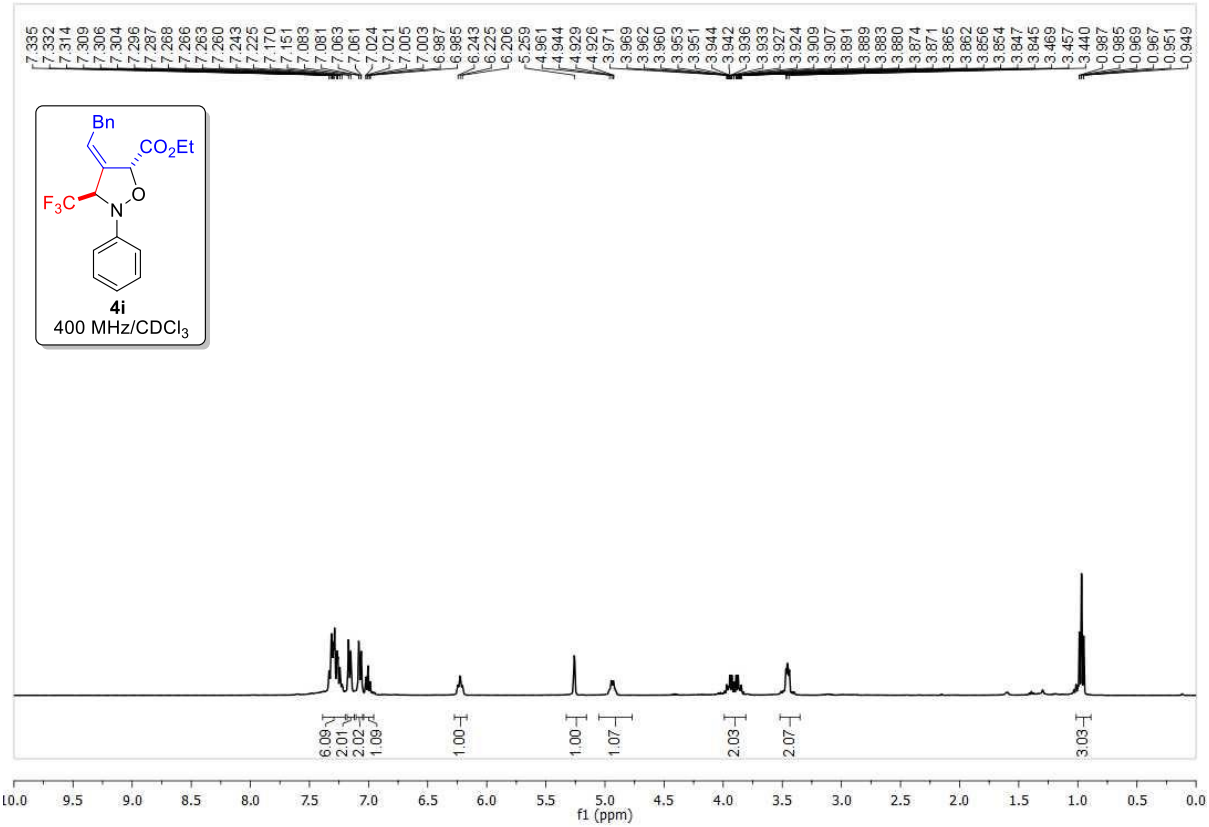

n-Hex
CO2Et
F3C
O
N
4g
100 MHz/CDCl3
f1 (ppm)

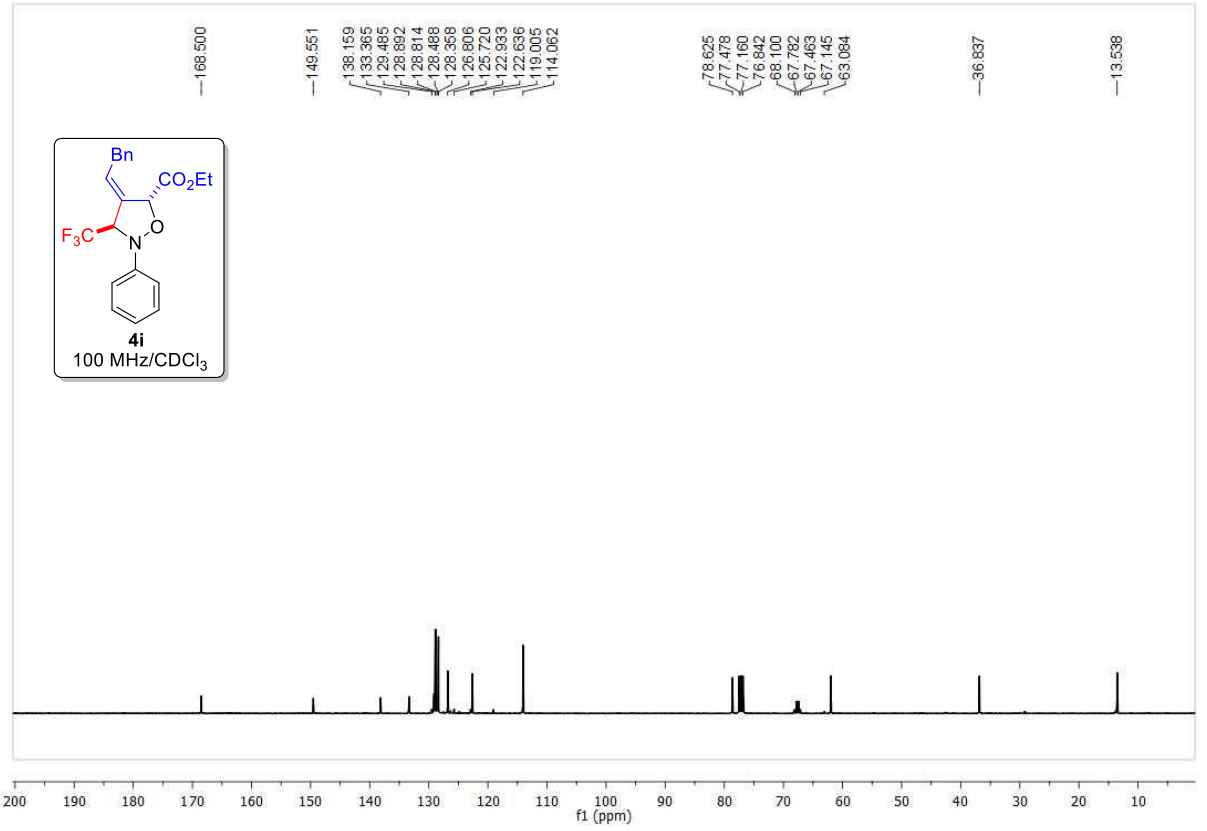

n-Hex
CO2Et
F3C
N
O
4g
376 MHz/CDCl3
-74.587
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

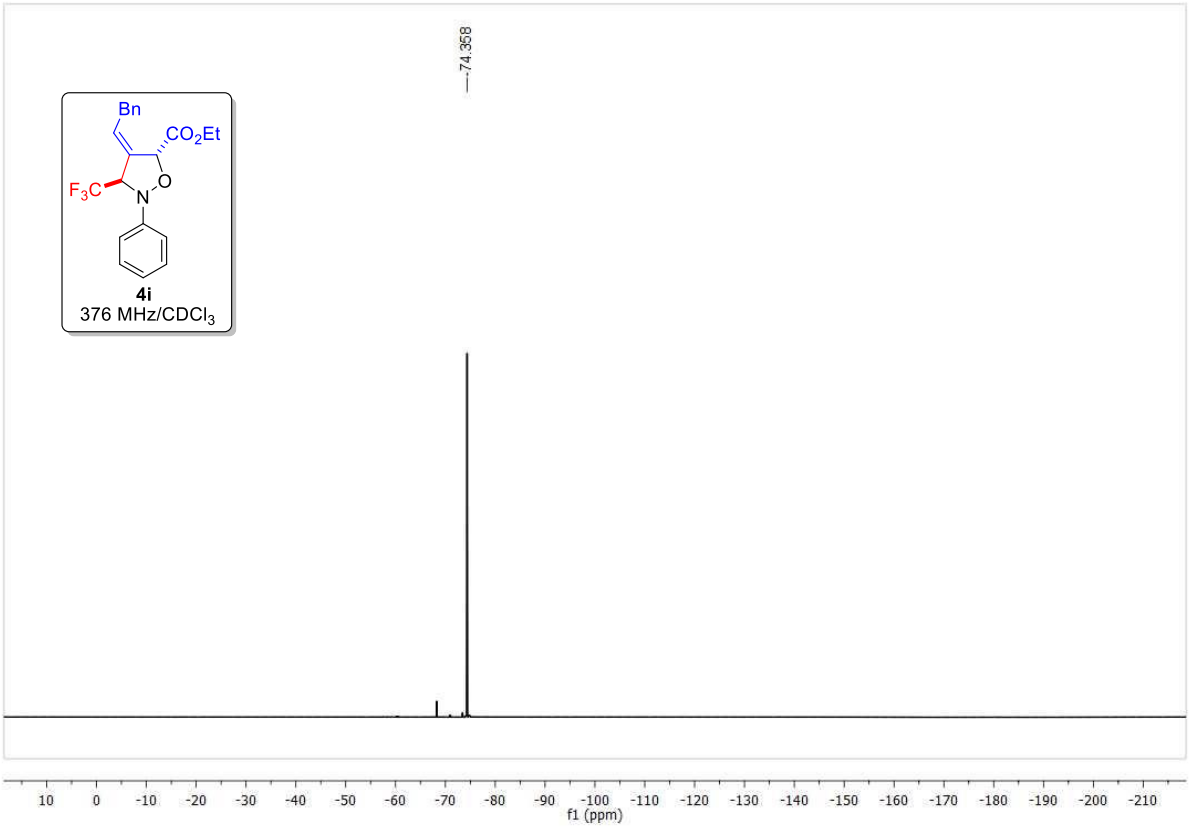

i-Pr
CO2Et
F3C
N
O
4h
400 MHz/CDCl3
7.273
7.260
7.255
7.252
7.233
7.030
7.010
6.981
6.963
6.945
5.796
5.770
5.165
4.854
4.836
4.818
4.800
2.336
2.320
2.303
2.293
2.287
2.277
2.261
1.047
1.031
0.984
0.966
0.949
2.08
2.01
1.07
1.00
1.05
1.10
2.10
1.13
3.02
6.12
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

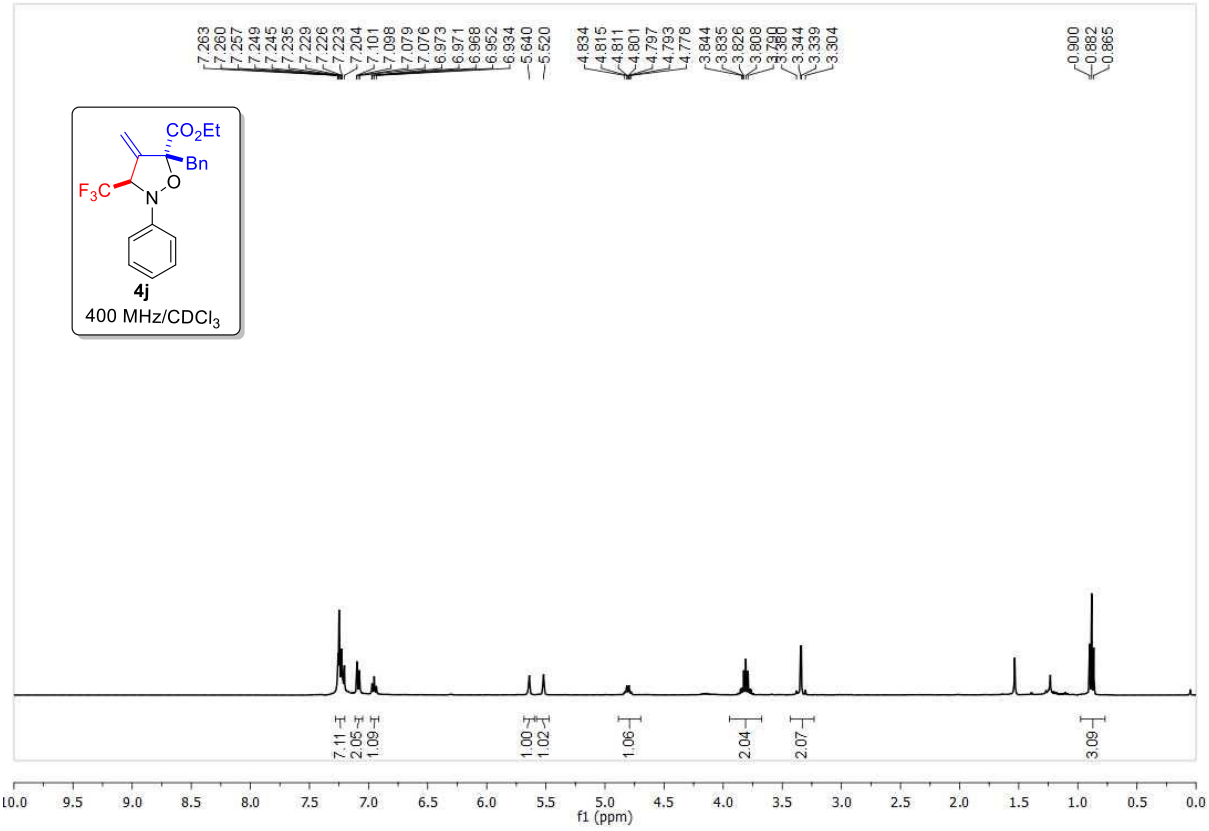

168.912
149.777
137.297
129.826
129.228
128.880
125.777
122.993
122.463
120.023
113.925
78.428
77.477
77.160
76.842
67.975
67.681
67.362
67.041
61.871
31.281
22.351
21.807
13.605
i-Pr
CO2Et
F3C
O
N
4h
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

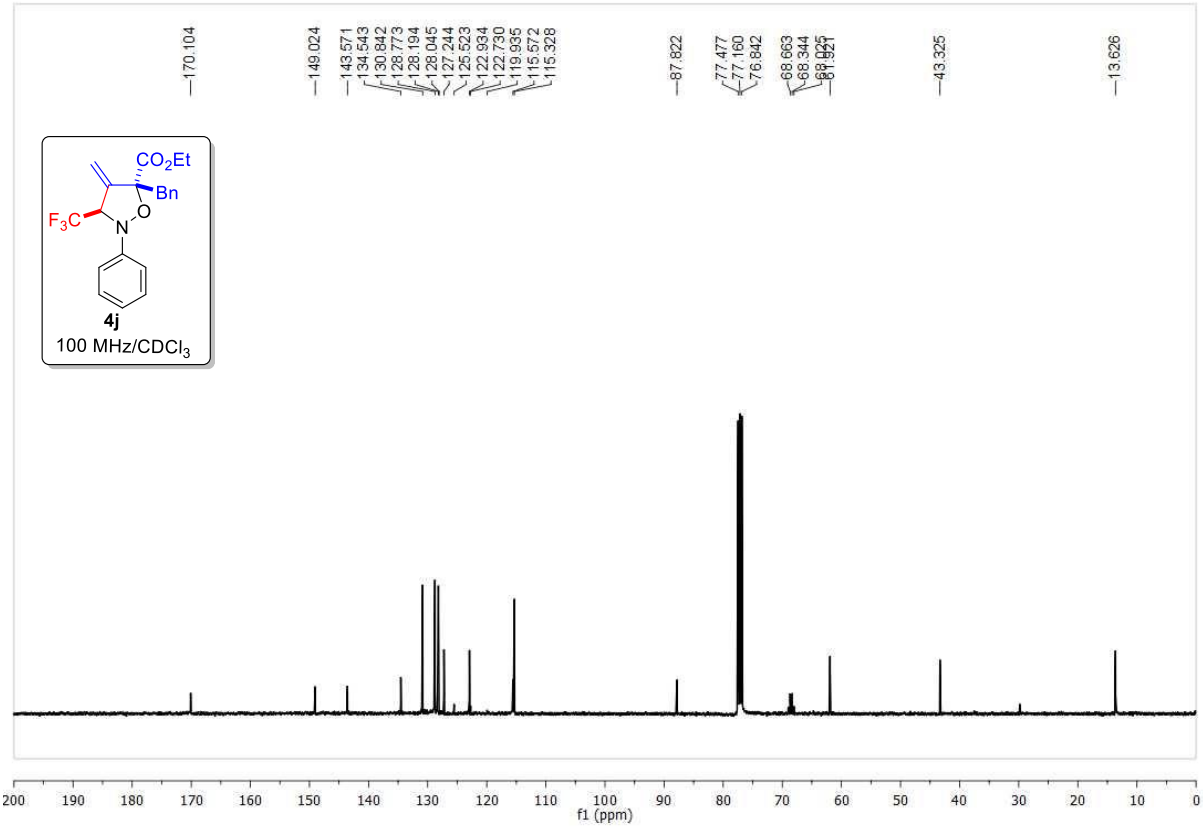

-74.642
i-Pr
CO2Et
F3C
O
N
4h
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

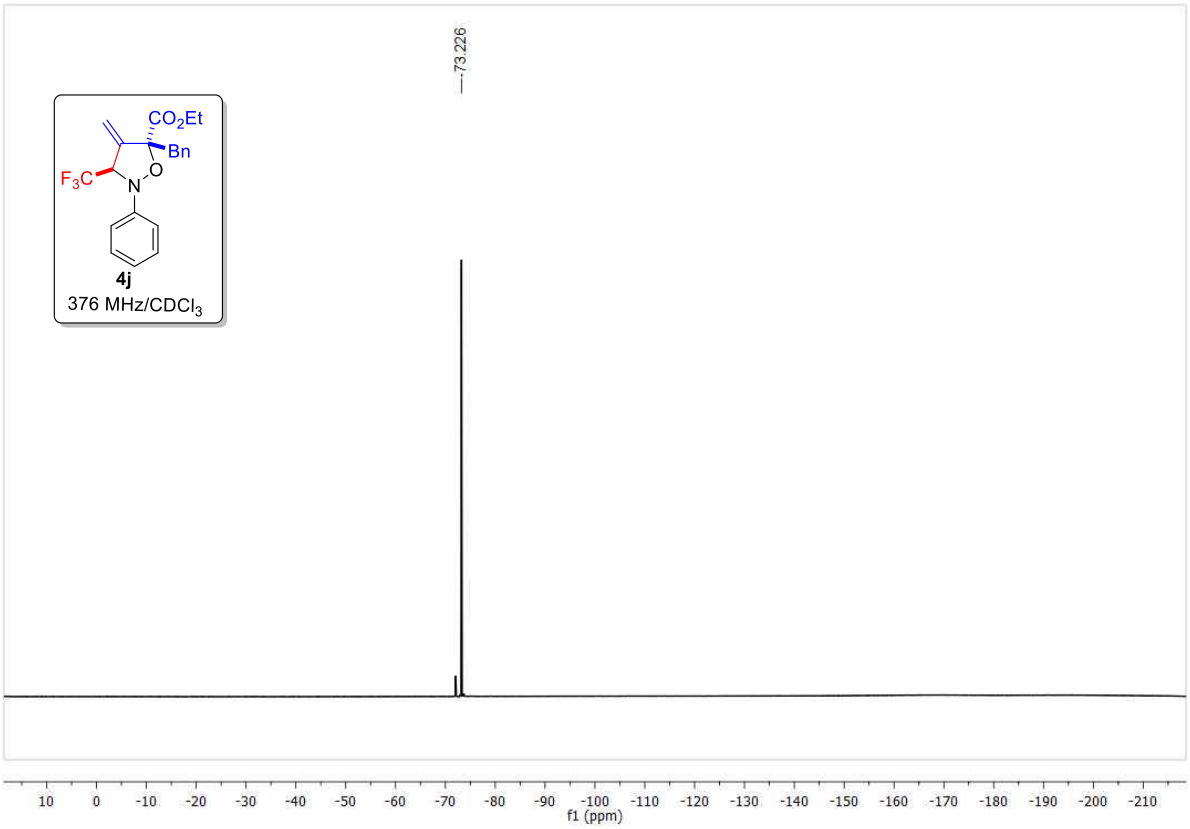

Bn
CO2Et
F3C
N
O
4i
400 MHz/CDCl3
f1 (ppm)

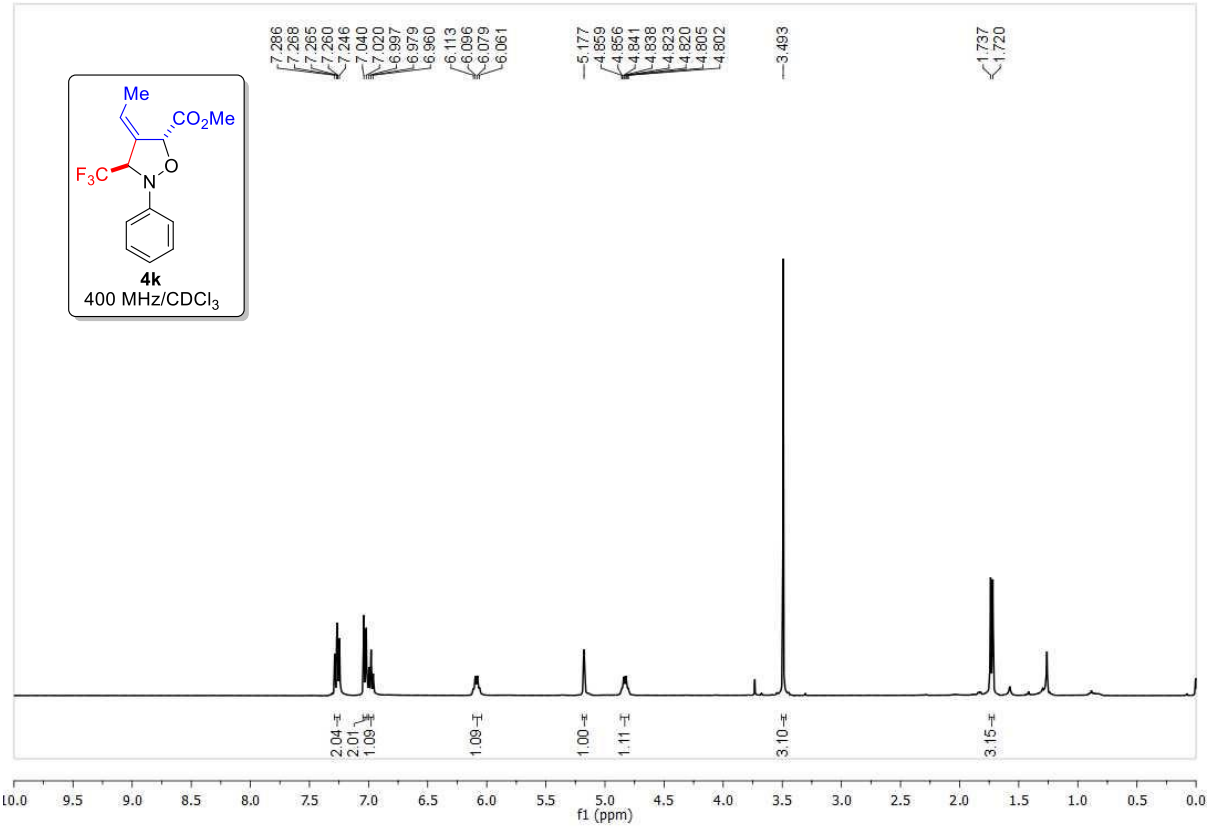

Bn
CO2Et
F3C
N
O
4i
100 MHz/CDCl3
f1 (ppm)

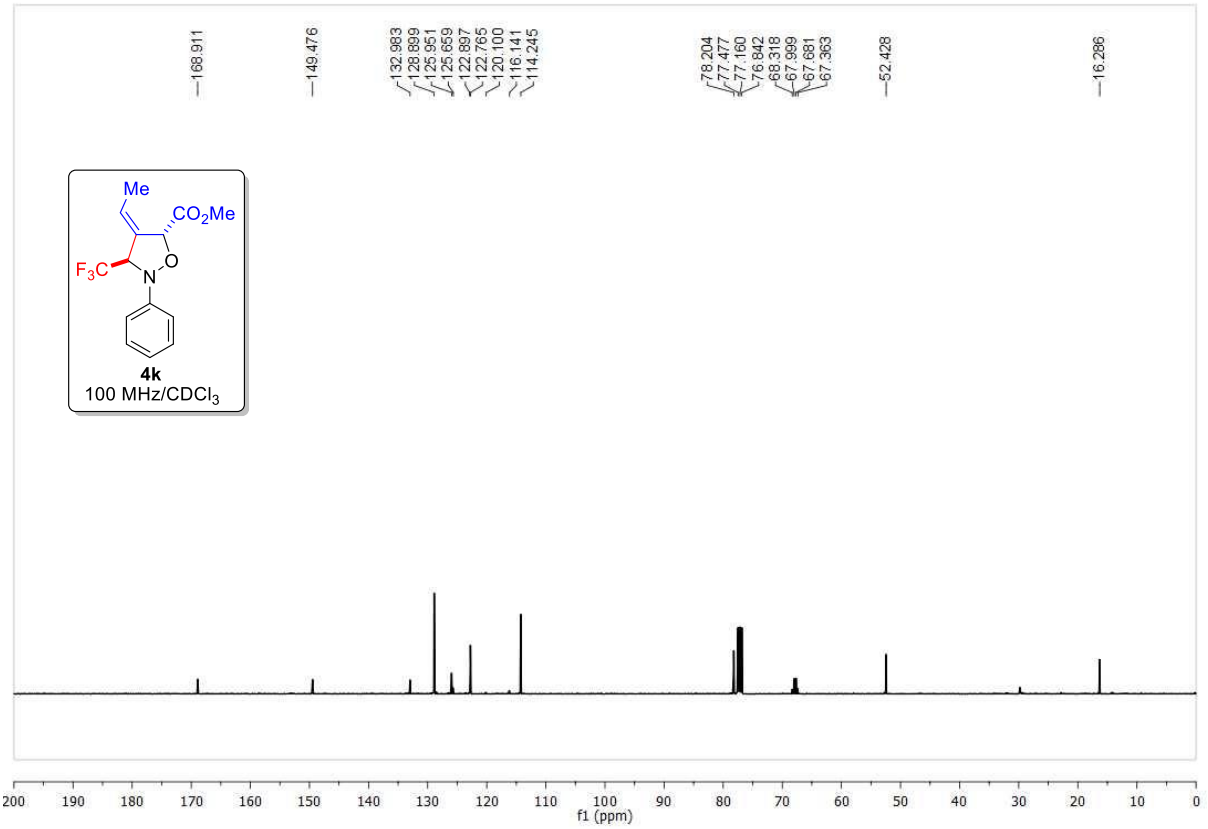

-74.358
Bn
CO2Et
F3C
N
O
4i
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

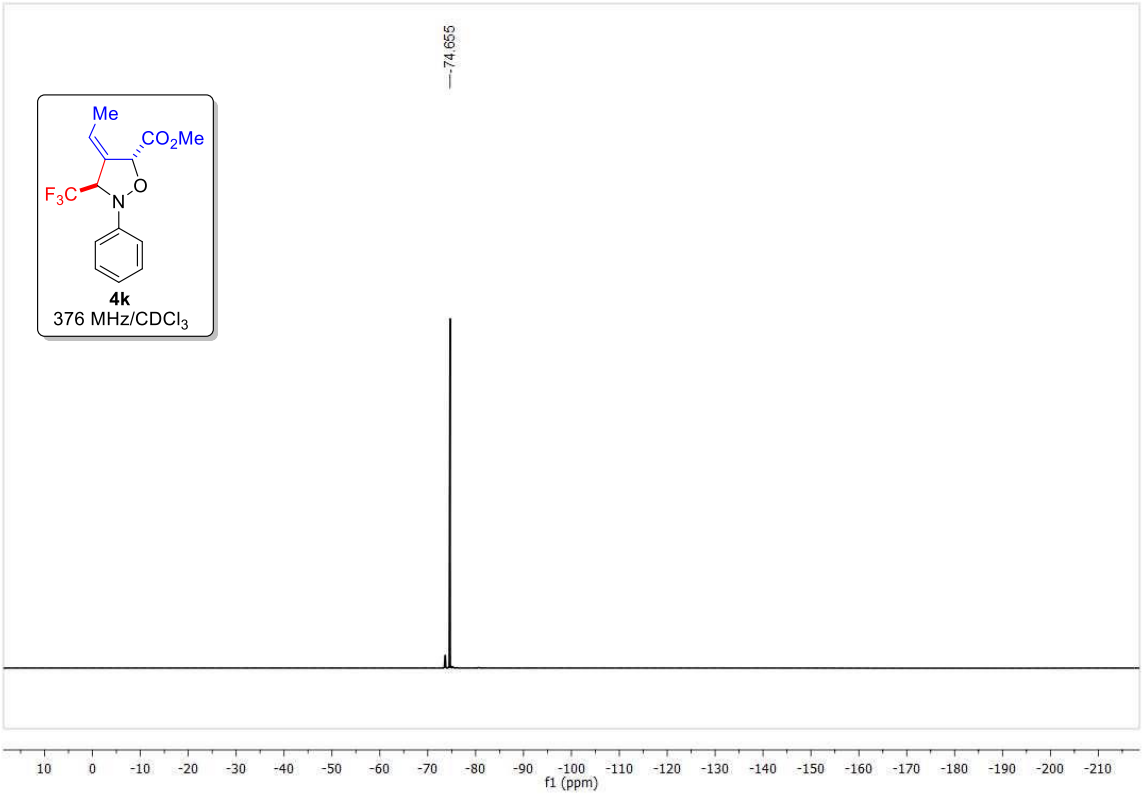

7.263
7.260
7.257
7.249
7.245
7.235
7.229
7.226
7.223
7.204
7.101
7.098
7.079
7.076
7.076
6.973
6.971
6.968
6.952
6.934
5.640
5.520
4.834
4.815
4.811
4.801
4.797
4.793
4.778
3.844
3.835
3.826
3.808
3.790
3.380
3.344
3.339
3.304
0.900
0.882
0.865
CO2Et
Bn
F3C
N
O
4j
400 MHz/CDCl3
7.11
2.05
1.09
1.00
1.02
1.06
2.04
2.07
3.09
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
f1 (ppm)

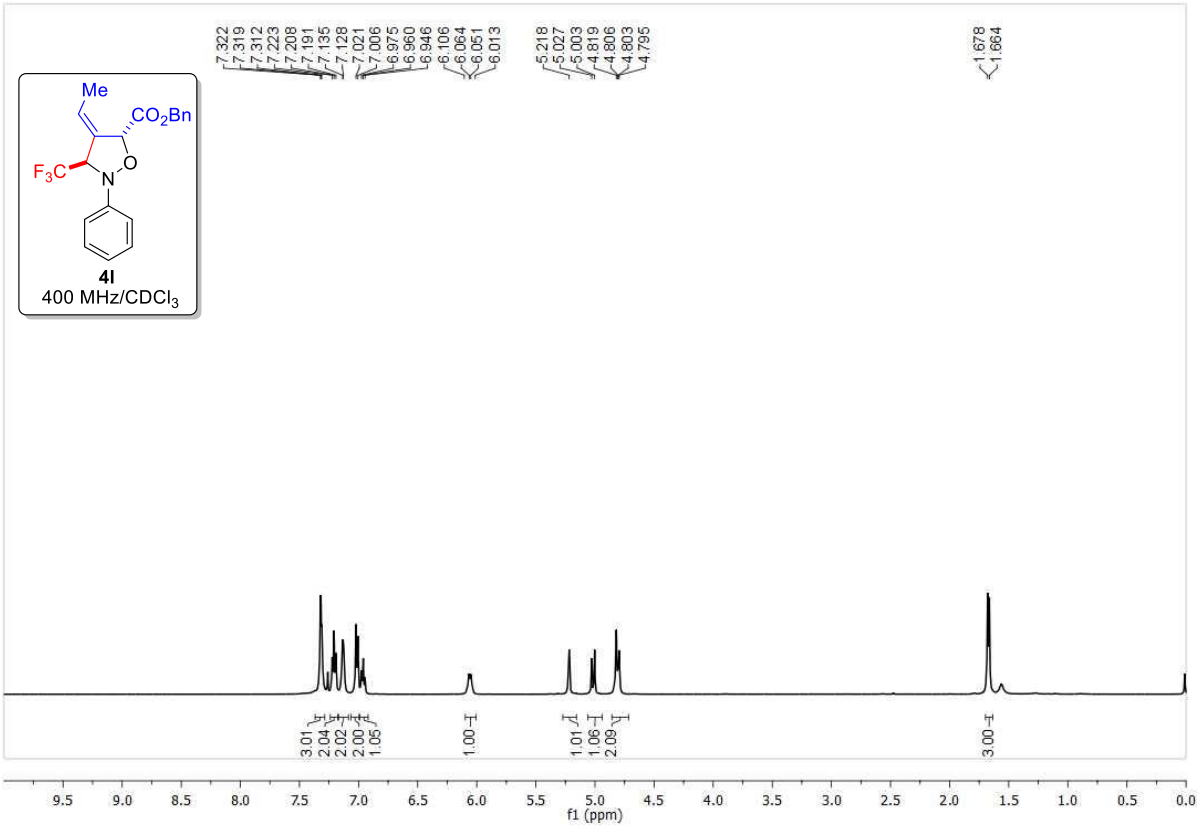

170.104
149.024
143.571
134.543
130.842
128.773
128.194
128.045
127.244
125.523
122.934
122.730
119.935
115.572
115.328
87.822
77.477
77.160
76.842
68.663
68.344
61.921
43.325
13.626
CO2Et
Bn
F3C
N
O
4j
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

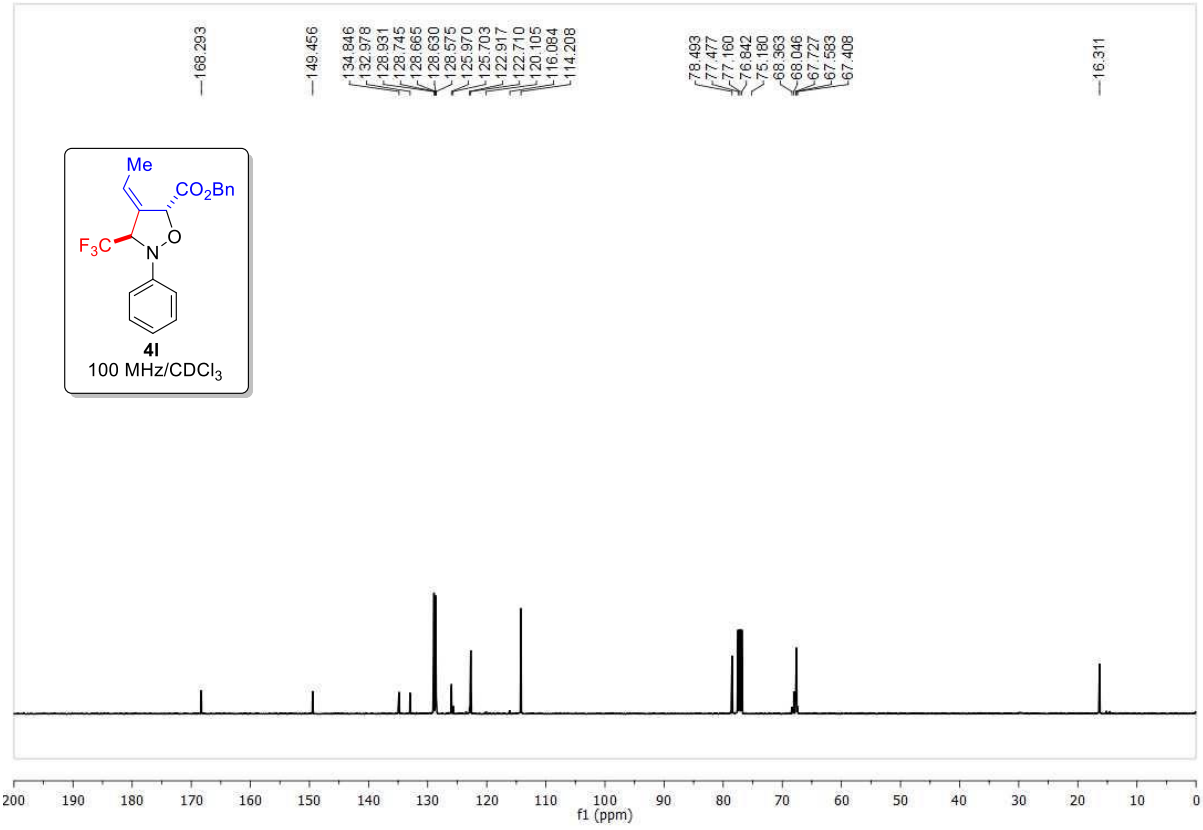

73.226
CO2Et
Bn
F3C
N
O
4j
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

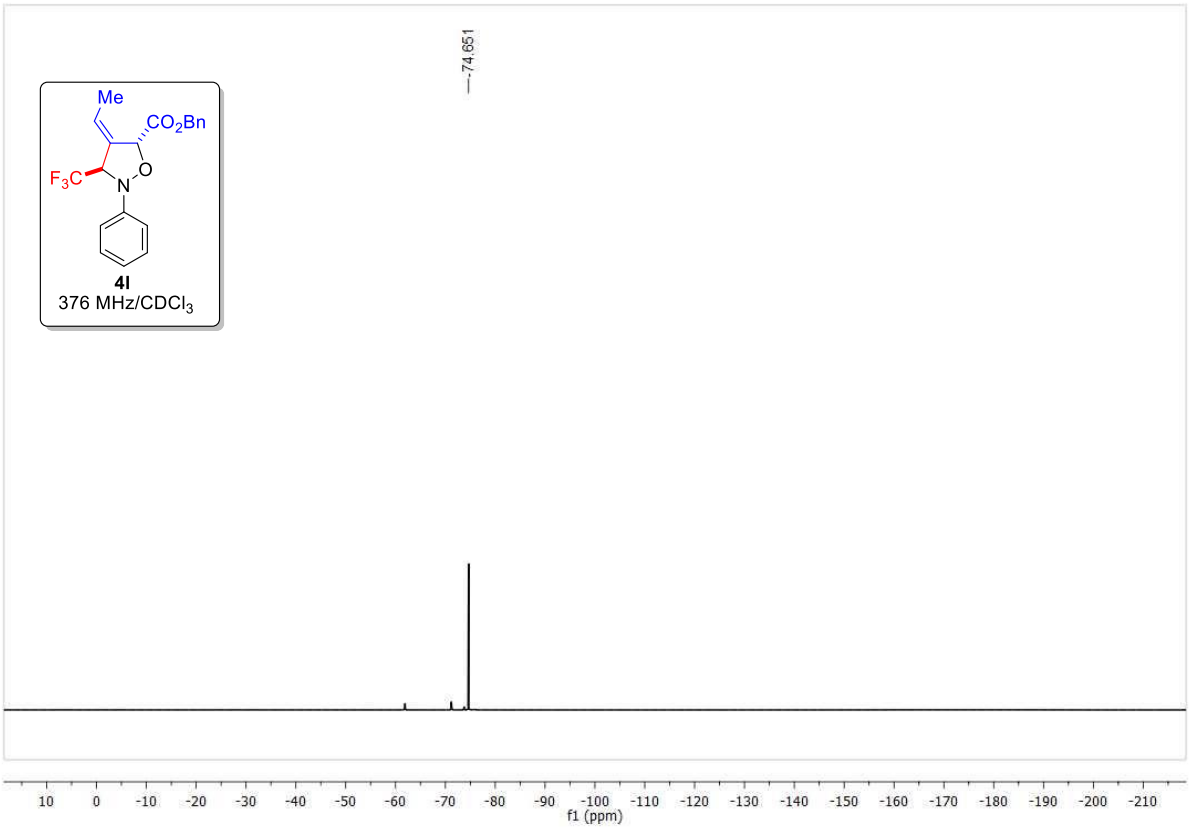

Me
CO2Me
F3C
N
O
4k
400 MHz/CDCl3
7.286
7.268
7.265
7.260
7.246
7.040
7.020
6.997
6.979
6.960
6.113
6.096
6.079
6.061
5.177
4.859
4.856
4.841
4.838
4.823
4.820
4.805
4.802
3.493
1.737
1.720
2.04
2.01
1.09
1.09
1.00
1.11
3.10
3.15
f1 (ppm)

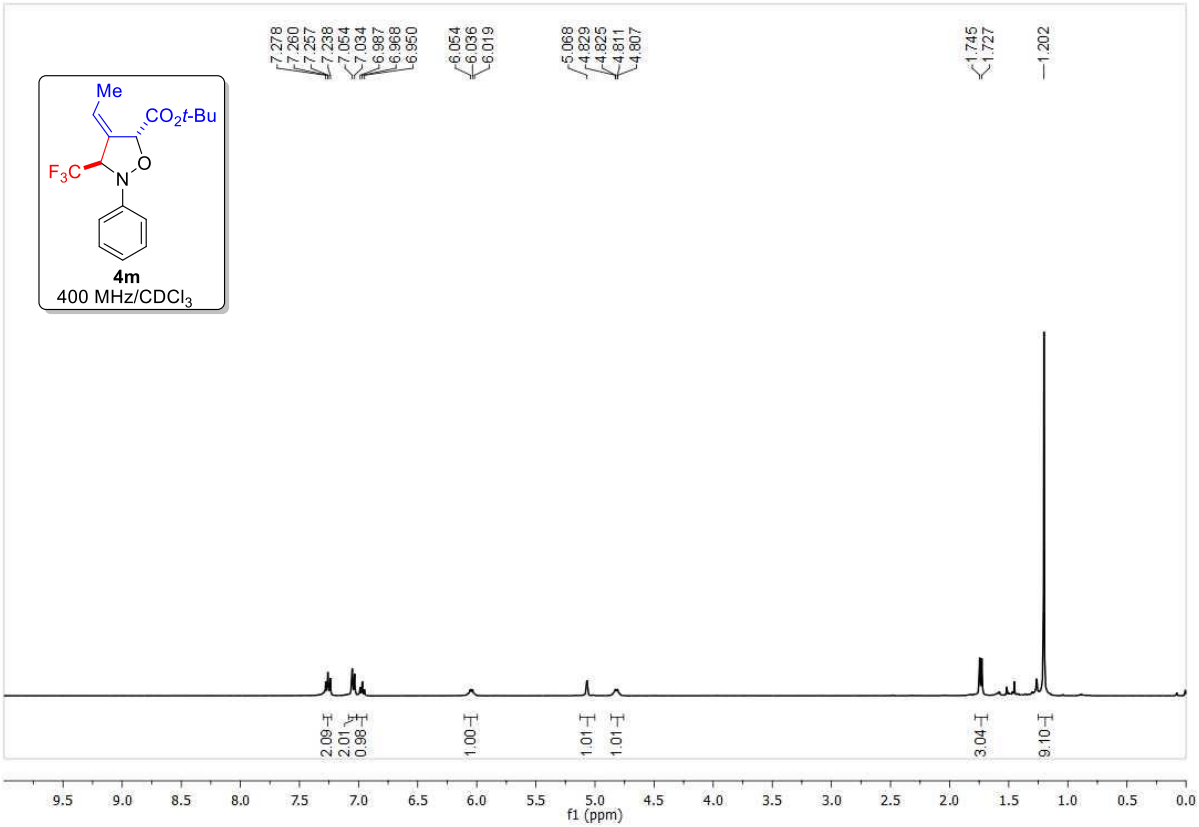

Me
CO2Me
F3C
N
O
4k
100 MHz/CDCl3
168.911
149.476
132.983
128.899
125.951
125.659
122.897
122.765
120.100
116.141
114.245
78.204
77.477
77.160
76.842
68.318
67.999
67.681
67.363
52.428
16.286
f1 (ppm)

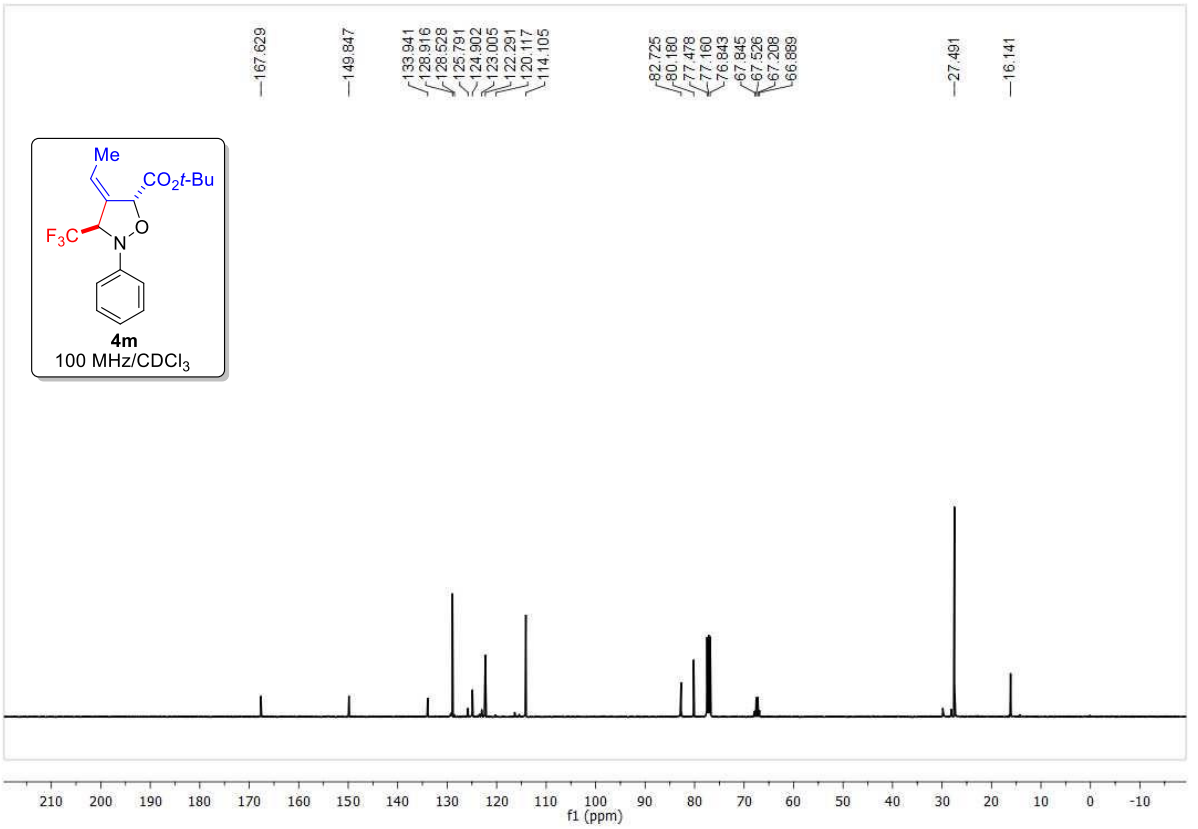

-74.655
Me
CO2Me
F3C
N
O
4k
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

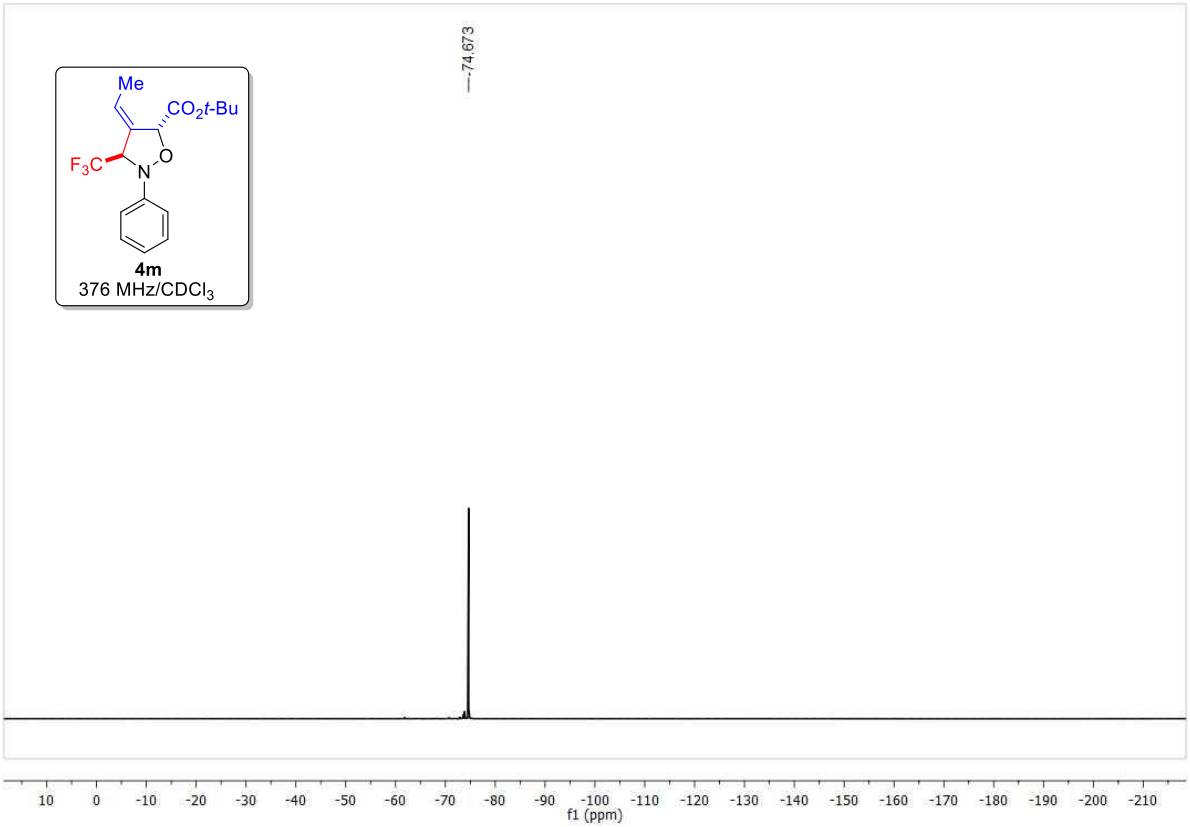

7.322
7.319
7.312
7.223
7.208
7.191
7.135
7.128
7.021
7.006
6.975
6.960
6.946
6.106
6.064
6.051
6.013
5.218
5.027
5.003
4.819
4.806
4.803
4.795
1.678
1.664
Me
CO2Bn
F3C
N
O
4l
400 MHz/CDCl3
3.01
2.04
2.02
2.00
1.05
1.00
1.01
1.06
2.09
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

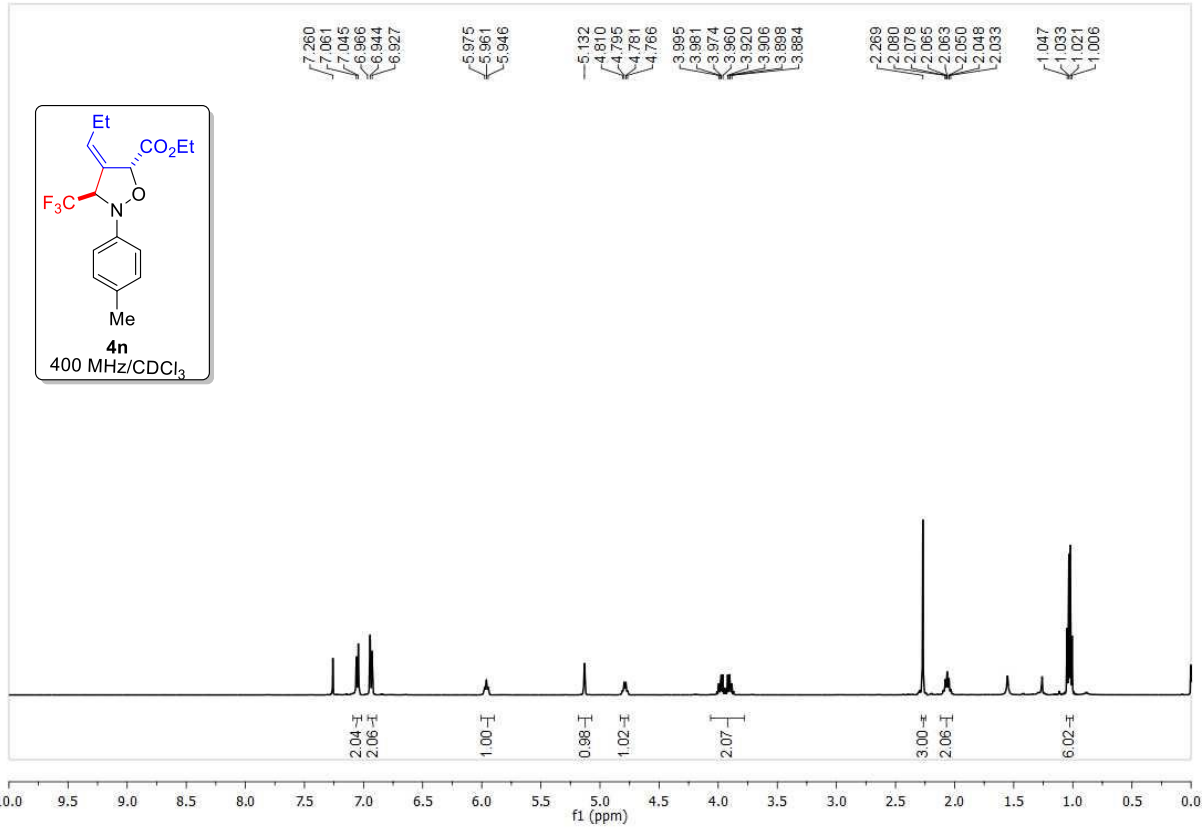

168.293
149.456
134.846
132.978
128.931
128.745
128.665
128.630
128.575
125.970
125.703
122.917
122.710
120.105
116.084
114.208
78.493
77.477
77.160
76.842
75.180
68.363
68.046
67.727
67.583
67.408
16.311
Me
CO2Bn
F3C
N
O
4l
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

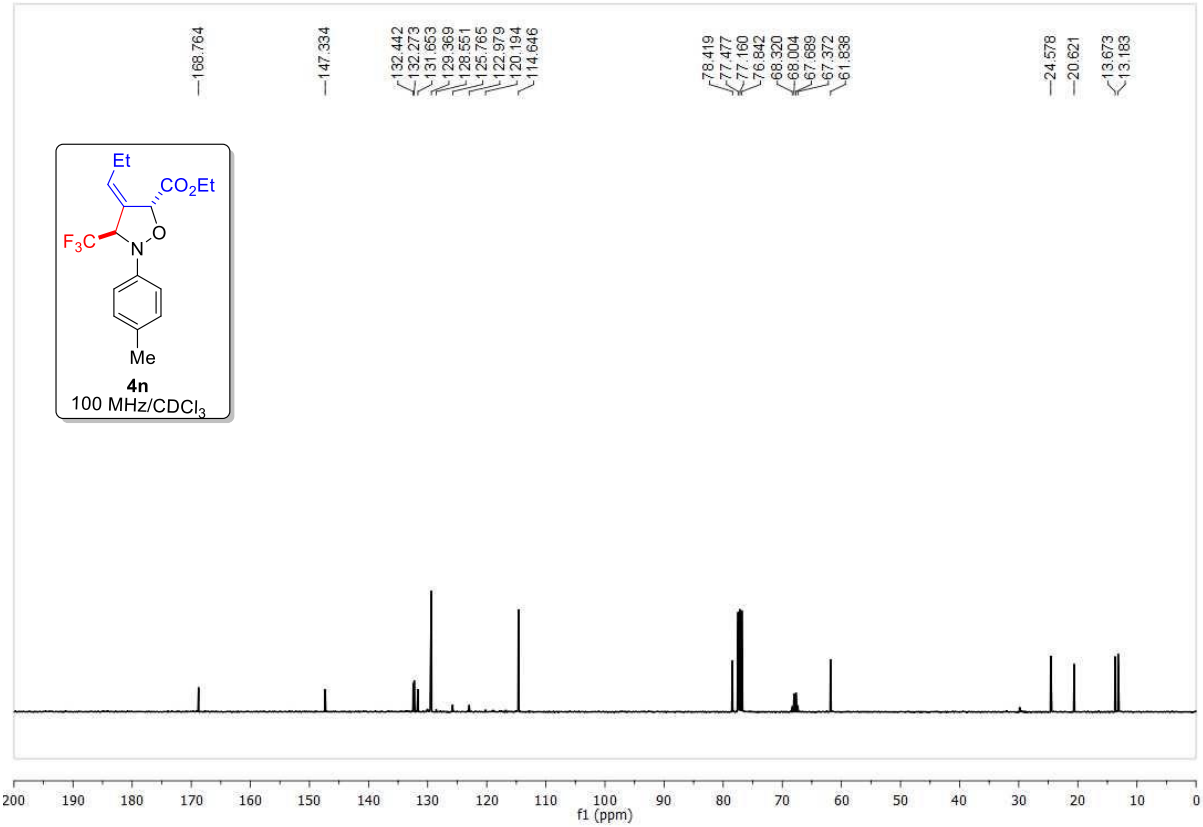

-74.651
Me
CO2Bn
F3C
N
O
4l
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

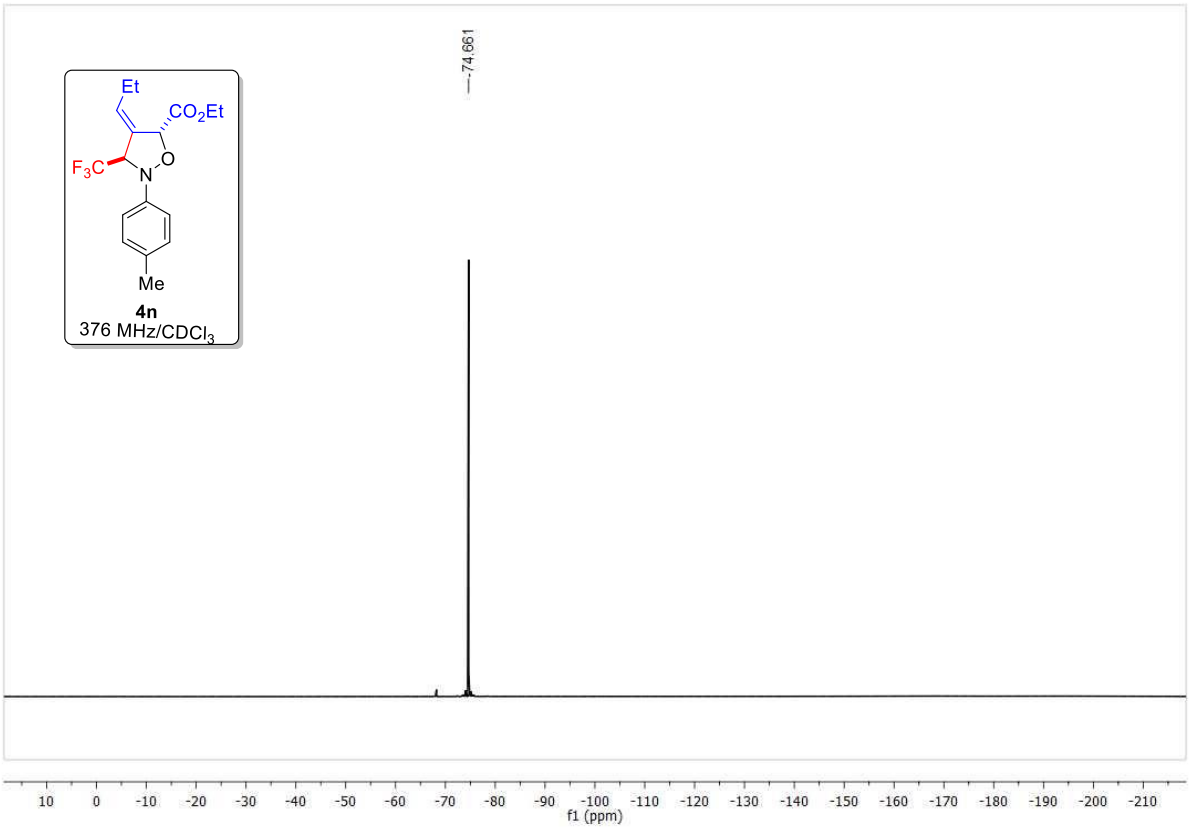

Me
CO2t-Bu
F3C
O
N
4m
400 MHz/CDCl3
7.278
7.260
7.257
7.238
7.054
7.034
6.987
6.968
6.950
6.054
6.036
6.019
5.068
4.829
4.825
4.811
4.807
1.745
1.727
1.202
2.09
2.01
0.98
1.00
1.01
1.01
3.04
9.10
f1 (ppm)

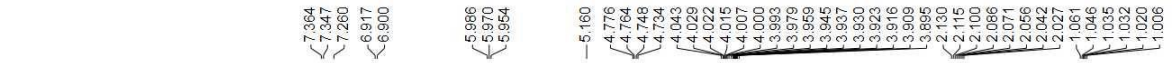

Me
CO2t-Bu
F3C
O
N
4m
100 MHz/CDCl3
167.629
149.847
133.941
128.916
128.528
125.791
124.902
123.005
122.291
120.117
114.105
82.725
80.180
77.478
77.160
76.843
67.845
67.526
67.208
66.889
27.491
16.141
f1 (ppm)

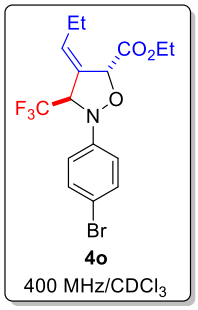

Me
CO2t-Bu
F3C
N
O
4m
376 MHz/CDCl3
-74.673
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

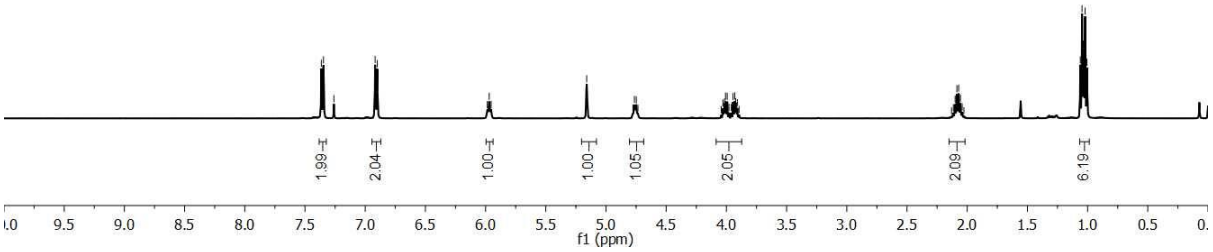

7.260
7.061
7.045
6.966
6.944
6.927
5.975
5.961
5.946
5.132
4.810
4.795
4.781
4.766
3.995
3.981
3.974
3.960
3.920
3.906
3.898
3.884
2.269
2.080
2.078
2.065
2.063
2.050
2.048
2.033
1.047
1.033
1.021
1.006
Et
CO2Et
F3C
N
O
Me
4n
400 MHz/CDCl3
2.04
2.06
1.00
0.98
1.02
2.07
3.00
2.06
6.02
10.0 9.5 9.0 8.5 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
f1 (ppm)


168.764
147.334
132.442
132.273
131.653
129.369
128.551
125.765
122.979
120.194
114.646
78.419
77.477
77.160
76.842
68.320
68.004
67.689
67.372
61.838
24.578
20.621
13.673
13.183
Et
CO2Et
F3C
N
O
Me
4n
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-74.661
Et
CO2Et
F3C
N
O
Me
4n
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

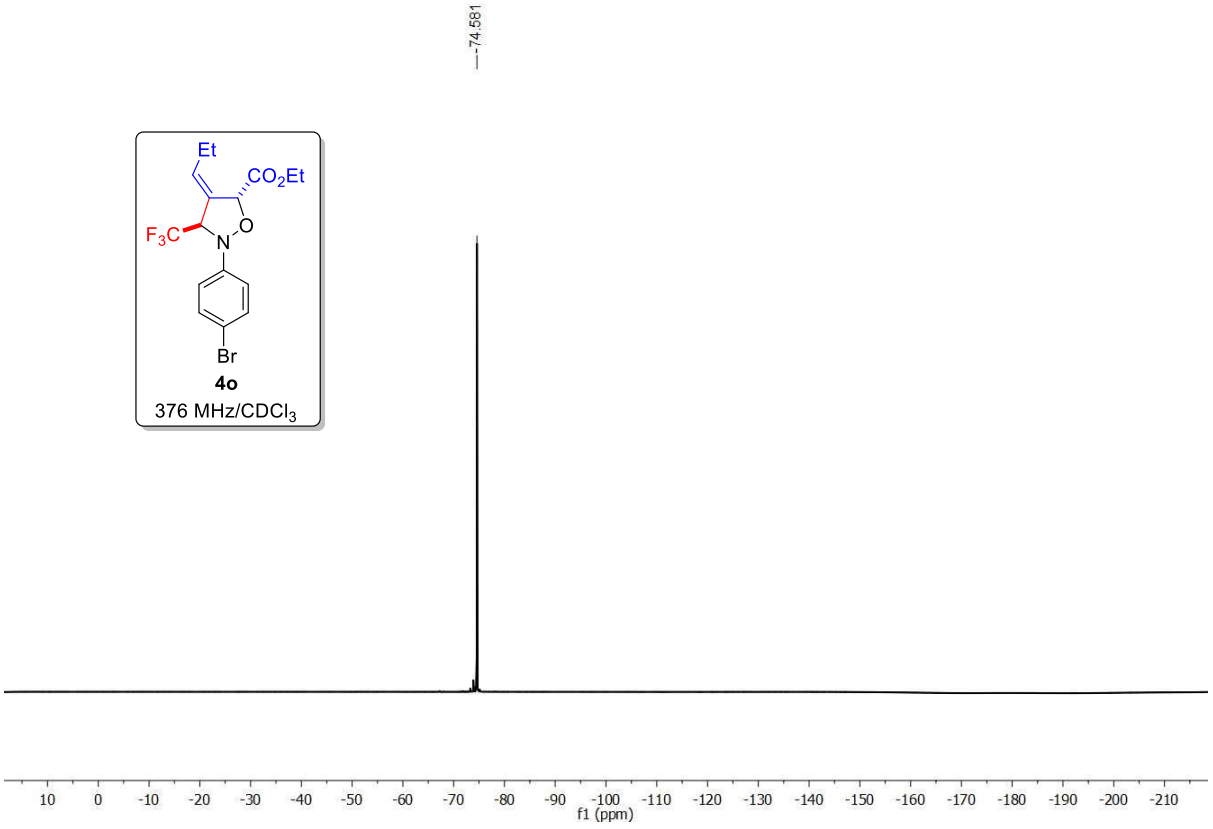

7.364
7.347
7.260
6.917
6.900
5.986
5.970
5.954
5.160
4.776
4.764
4.748
4.734
4.043
4.029
4.022
4.015
4.007
4.000
3.993
3.979
3.959
3.945
3.937
3.930
3.923
3.916
3.909
3.895
2.130
2.115
2.100
2.086
2.071
2.056
2.042
2.027
1.061
1.046
1.035
1.032
1.020
1.006

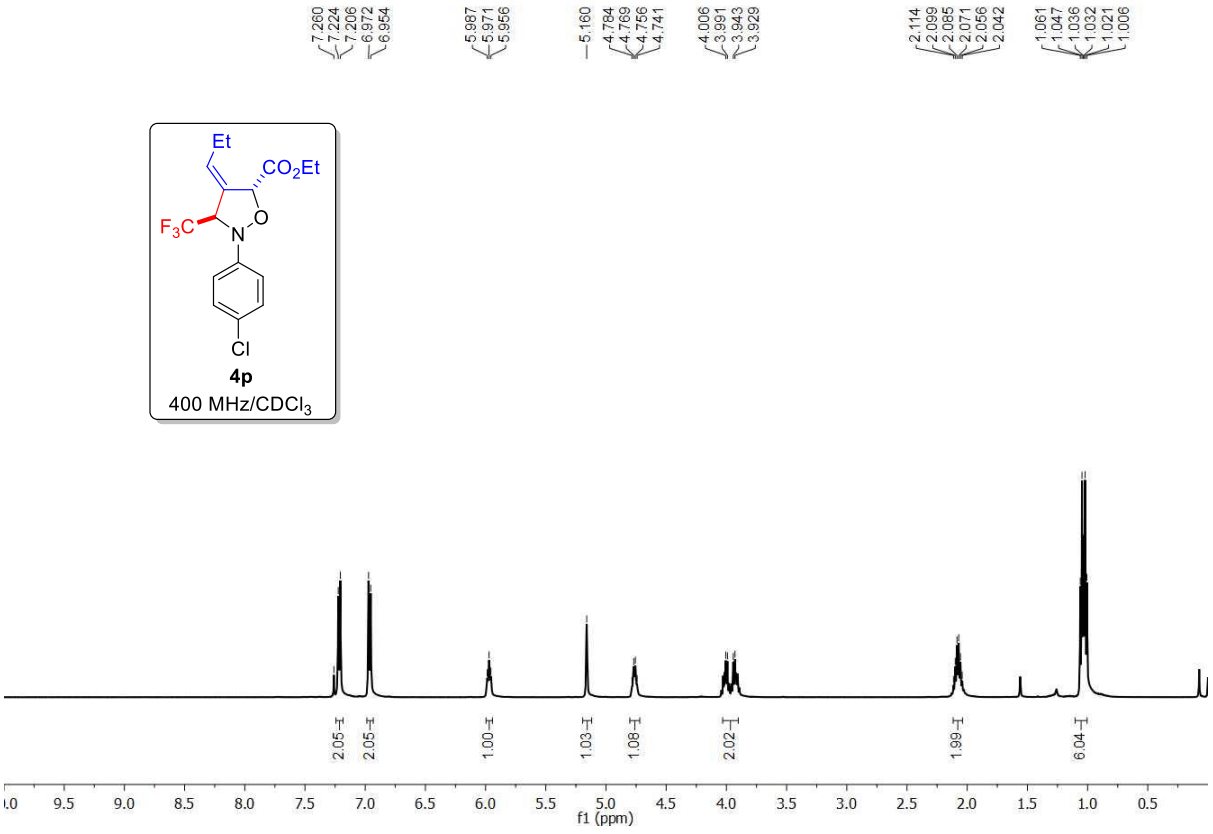

Et
CO2Et
F3C
N
O
Br
4o
400 MHz/CDCl3

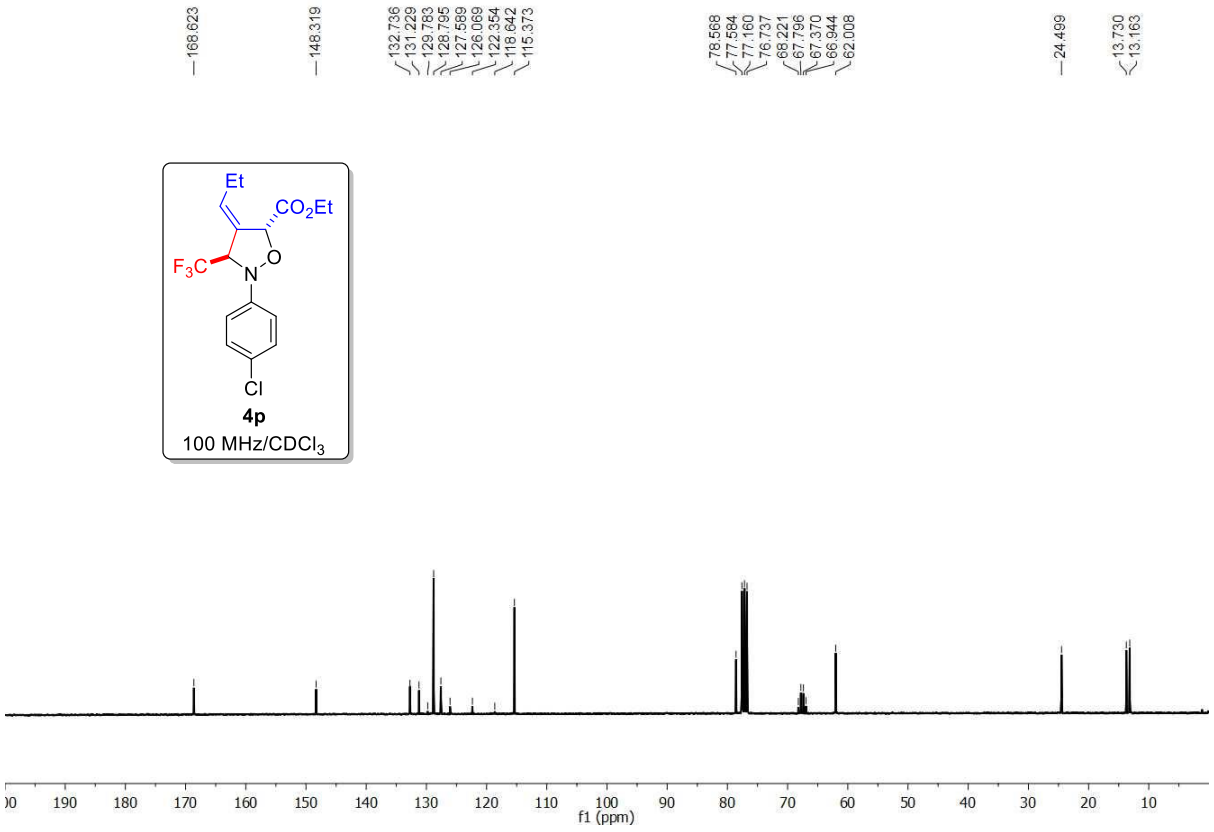

1.99
2.04
1.00
1.00
1.05
2.05
2.09
6.19
f1 (ppm)

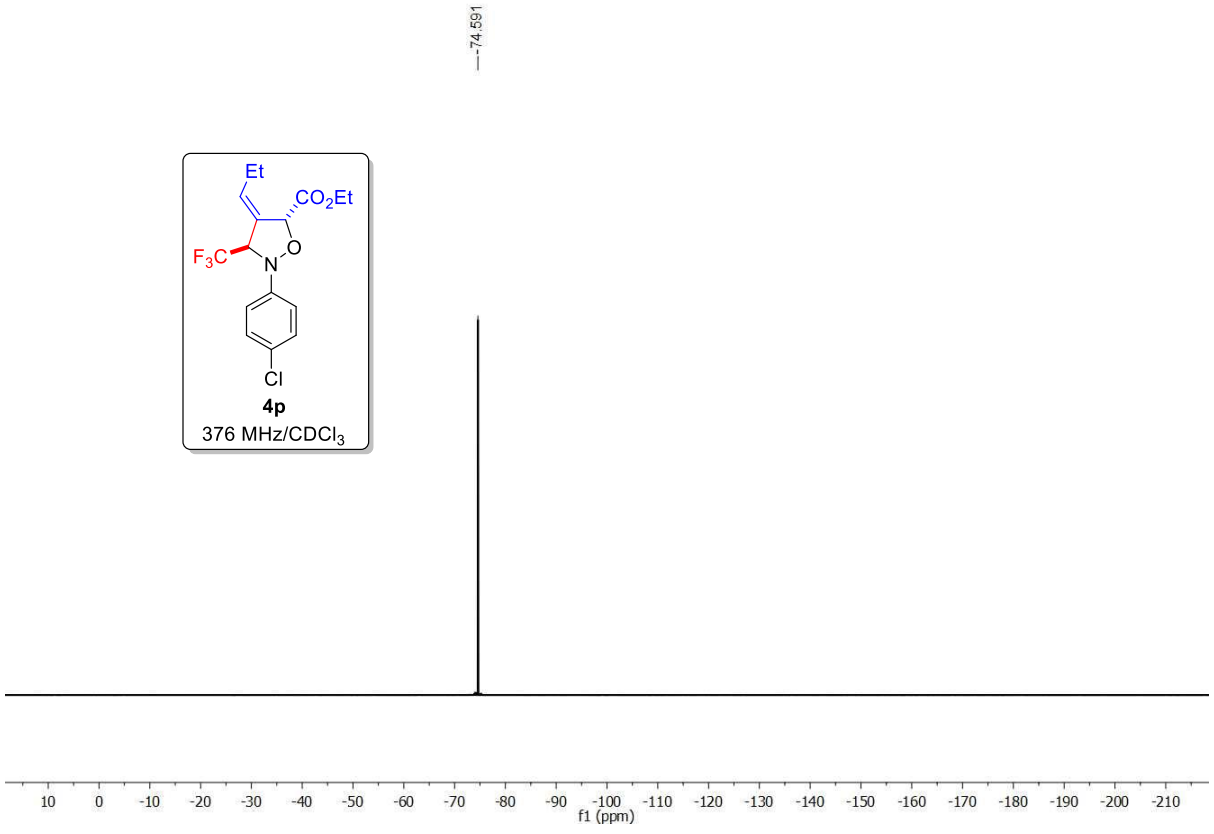

168.607
148.810
132.725
131.703
131.173
127.532
125.305
123.077
120.788
115.686
114.924
78.561
77.413
77.160
76.906
67.865
67.610
67.354
67.100
62.030
24.500
13.727
13.172
f1 (ppm)

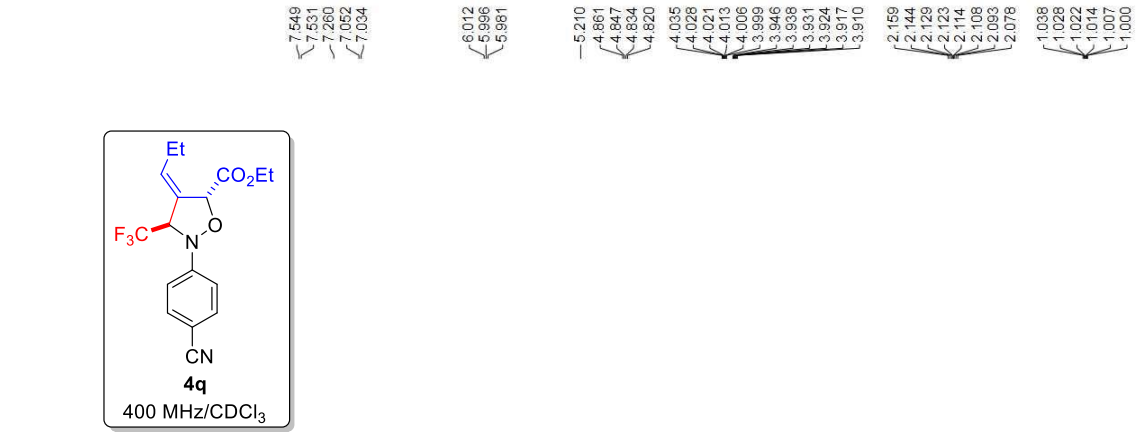
Et
CO2Et
F3C
N
O
Br
4o
100 MHz/CDCl3


-74.581
Et
CO2Et
F3C
N
O
Br
4o
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

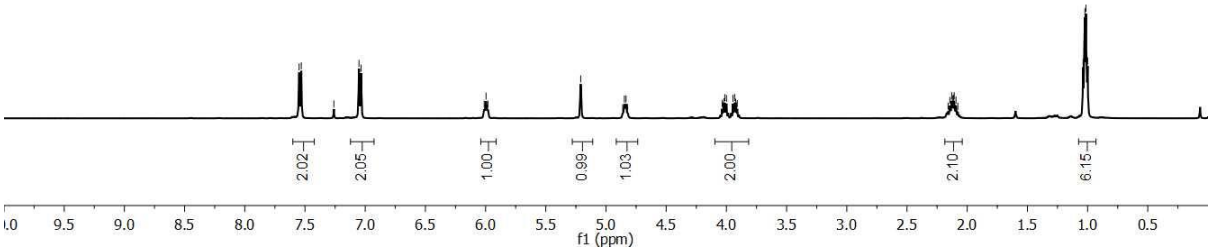

7.260
7.224
7.206
6.972
6.954
5.987
5.971
5.956
5.160
4.784
4.769
4.756
4.741
4.006
3.991
3.943
3.929
2.114
2.099
2.085
2.071
2.056
2.042
1.061
1.047
1.036
1.032
1.021
1.006
Et
CO2Et
F3C
N
O
Cl
4p
400 MHz/CDCl3
2.05
2.05
1.00
1.03
1.08
2.02
1.99
6.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


168.623
148.319
132.736
131.229
129.783
128.795
127.589
126.069
122.354
118.642
115.373
78.588
77.584
77.160
76.737
68.221
67.796
67.370
66.944
62.008
24.499
13.730
13.163
Et
CO2Et
F3C
N
O
Cl
4p
100 MHz/CDCl3
f1 (ppm)

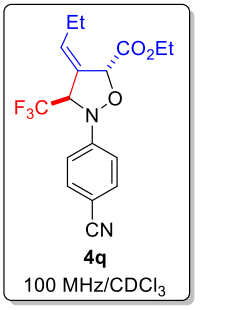

-74.591
Et
CO2Et
F3C
N
O
Cl
4p
376 MHz/CDCl3
f1 (ppm)

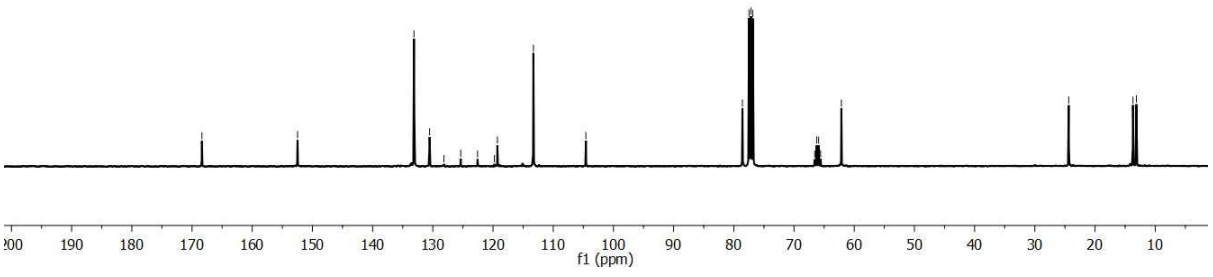

7.549
7.531
7.260
7.052
7.034
6.012
5.996
5.981
5.210
4.861
4.847
4.834
4.820
4.035
4.028
4.021
4.013
4.006
3.999
3.946
3.938
3.931
3.924
3.917
3.910
2.159
2.144
2.129
2.123
2.114
2.108
2.093
2.078
1.038
1.028
1.022
1.014
1.007
1.000

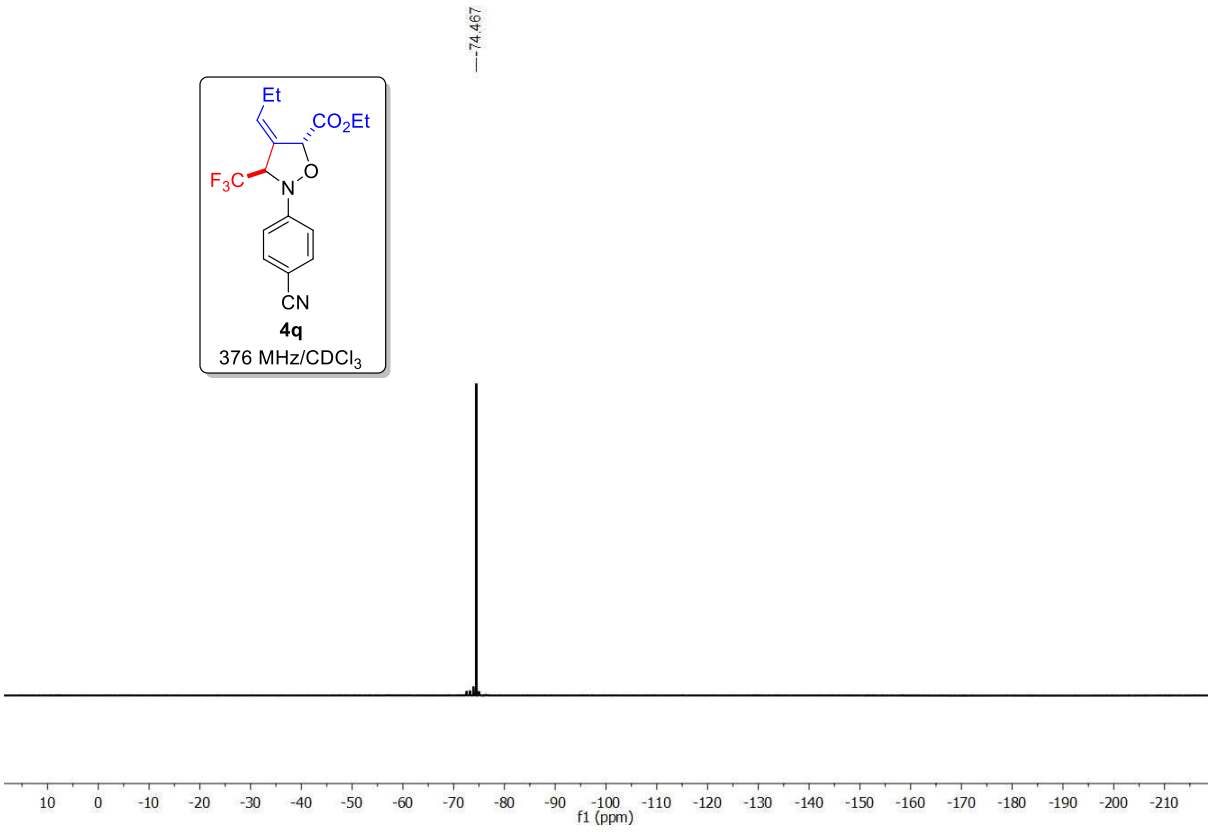
Et
CO2Et
F3C
O
N
CN
4q
400 MHz/CDCl3

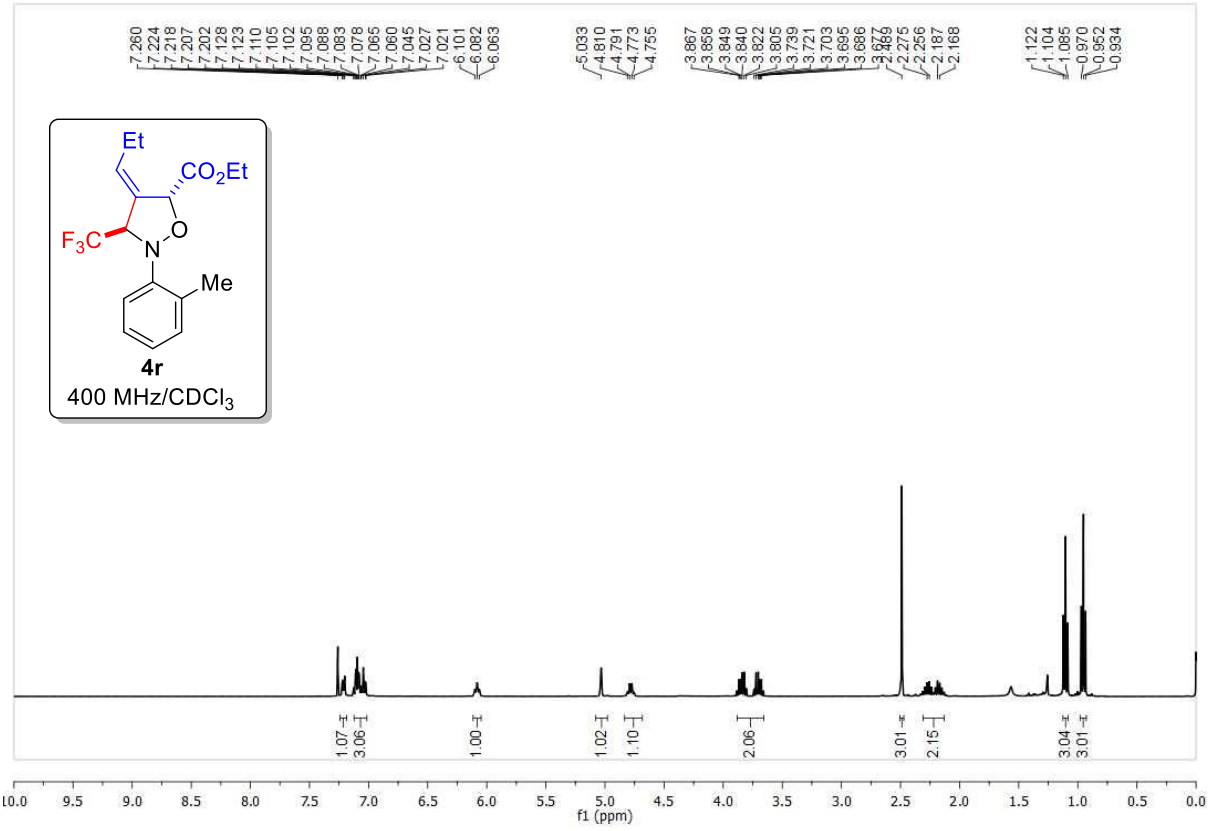

2.02
2.05
1.00
0.99
1.03
2.00
2.10
6.15
f1 (ppm)

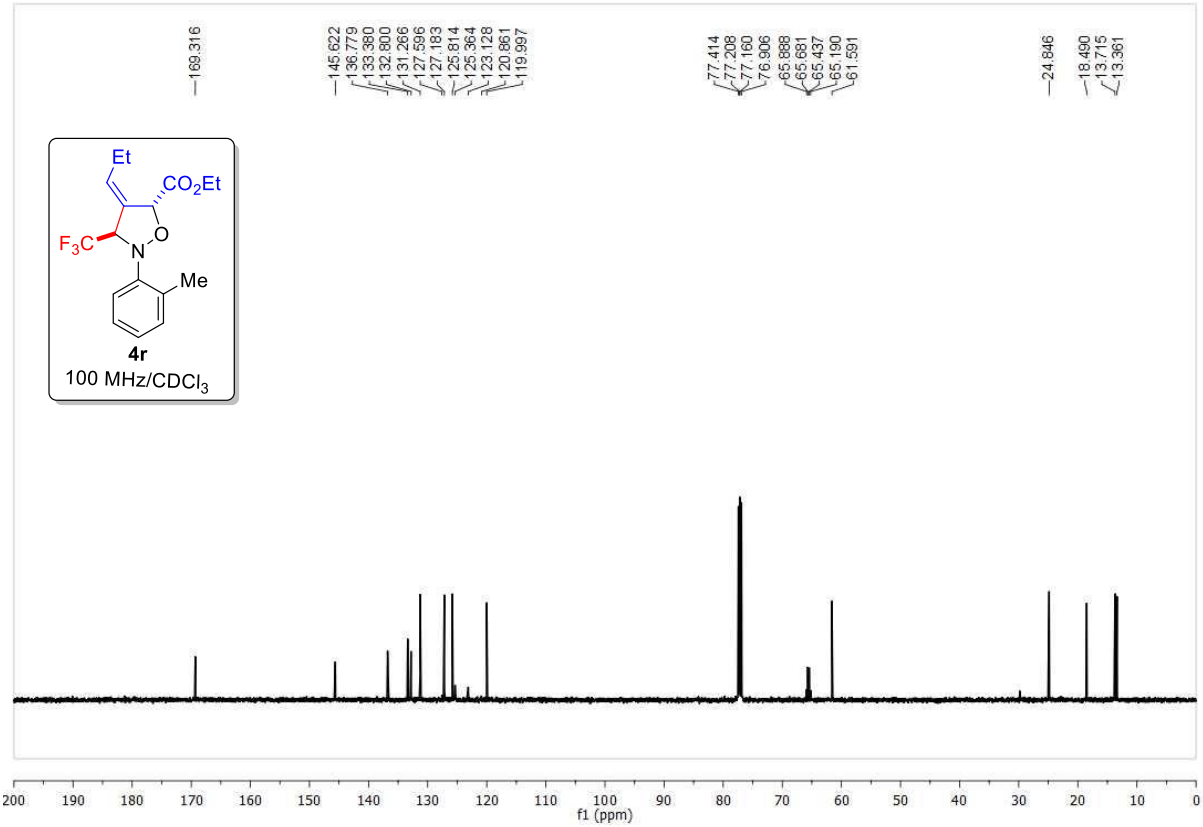

168.361
152.469
133.131
133.069
130.539
128.155
125.362
122.570
119.781
119.274
113.294
104.571
78.568
77.477
77.160
76.843
66.559
66.235
65.908
65.582
62.134
24.371
13.699
13.126

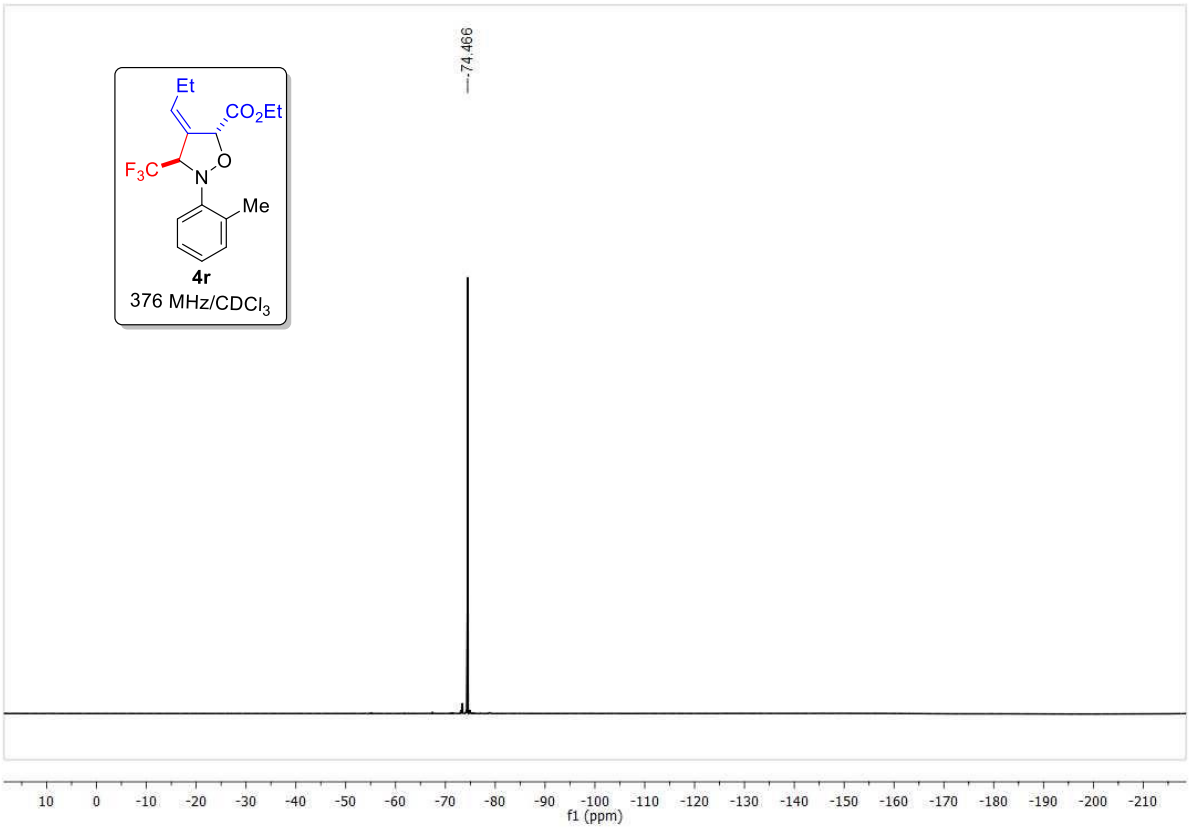

Et
CO2Et
F3C
O
N
CN
4q
100 MHz/CDCl3

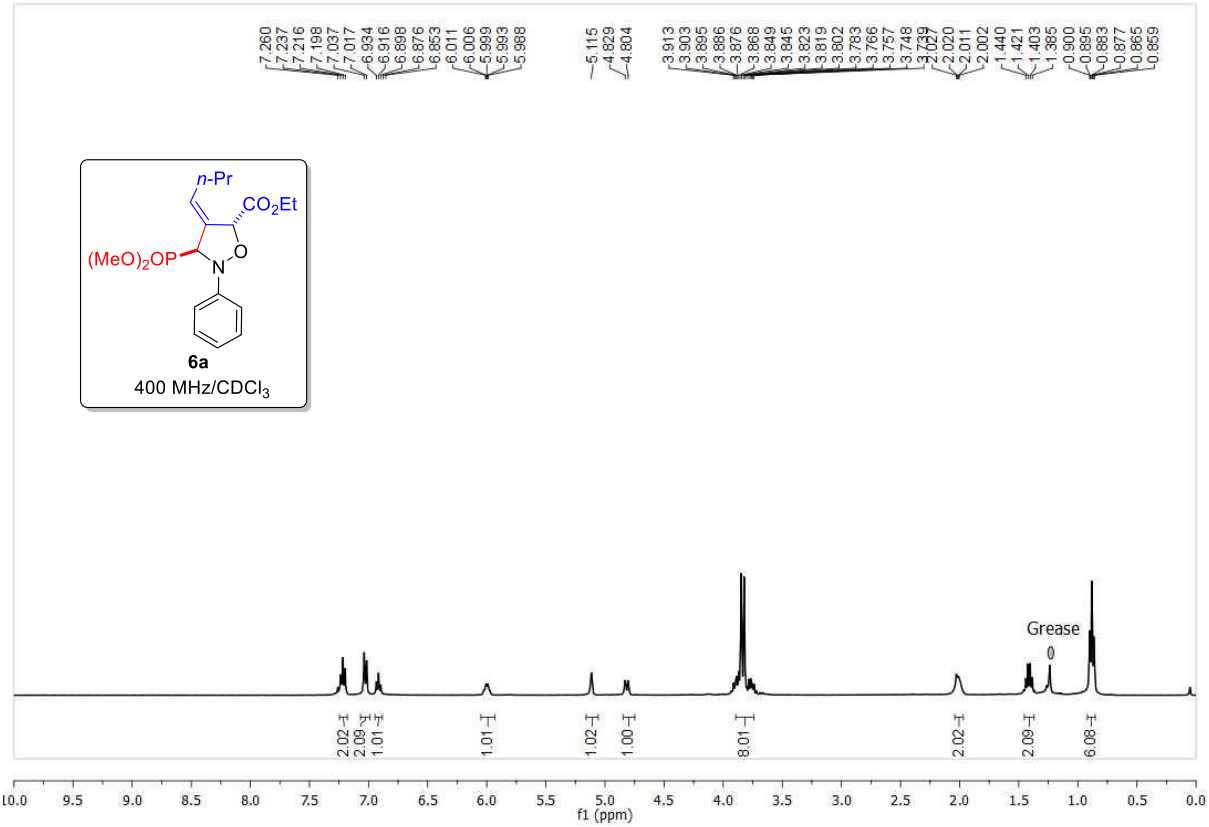

f1 (ppm)

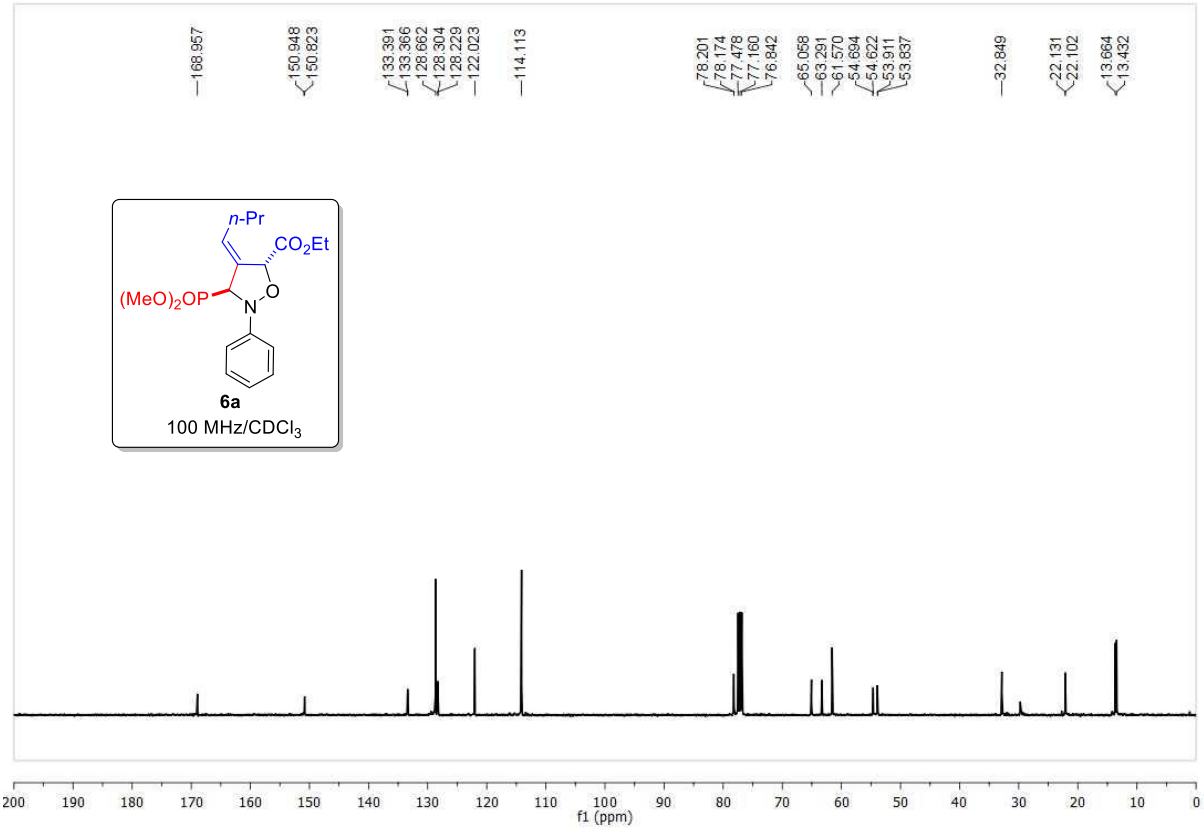

Et
CO2Et
F3C
N
O
CN
4q
376 MHz/CDCl3
-74.467
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

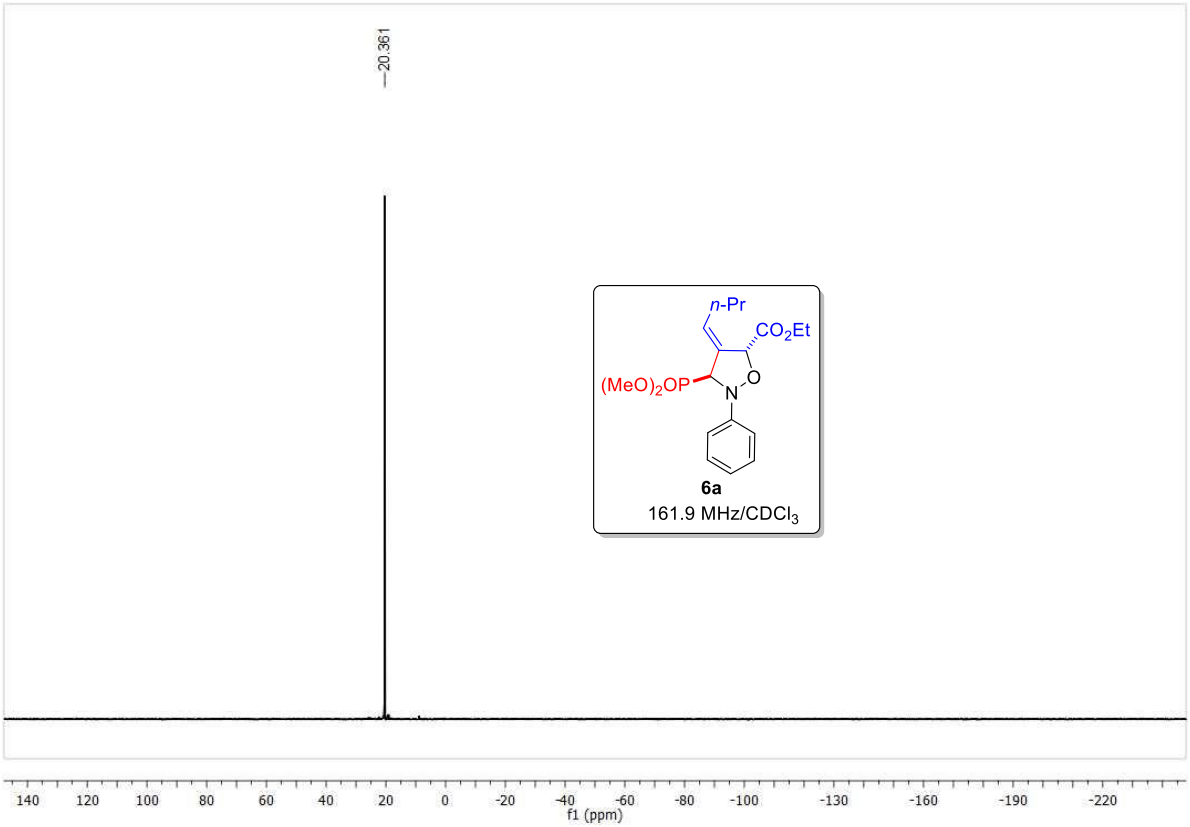

7.260 7.224 7.218 7.207 7.202 7.128 7.123 7.110 7.105 7.102 7.095 7.088 7.083 7.078 7.065 7.060 7.045 7.027 7.021 6.101 6.082 6.063
5.033 4.810 4.791 4.773 4.755
3.867 3.858 3.849 3.840 3.822 3.805 3.739 3.721 3.703 3.695 3.686 3.677 2.489 2.275 2.256 2.187 2.168
1.122 1.104 1.085 0.970 0.952 0.934
Et
CO2Et
F3C
N
O
Me
4r
400 MHz/CDCl3
1.07 3.06 1.00 1.02 1.10 2.06 3.01 2.15 3.04 3.01
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
f1 (ppm)

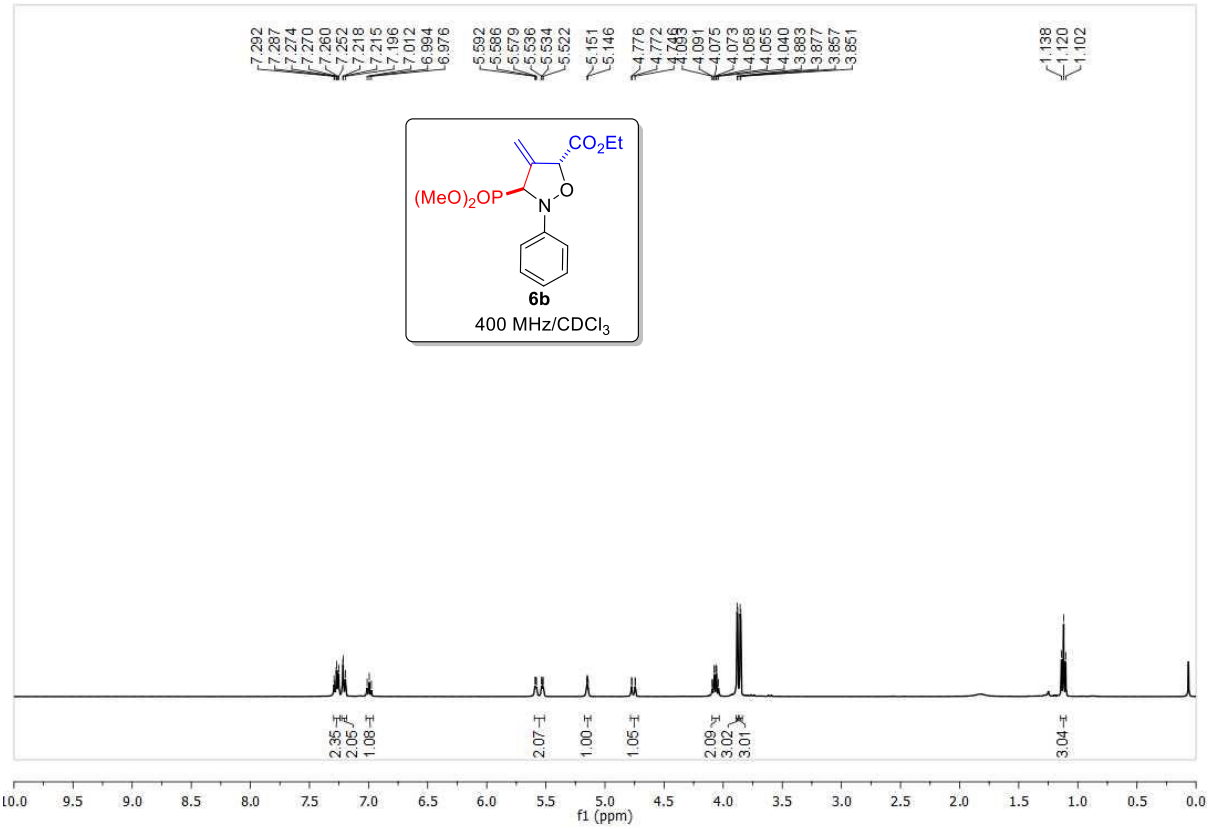

169.316
145.622
136.779
133.380
132.800
131.266
127.596
127.183
125.814
125.364
123.128
120.861
119.997
77.414
77.208
77.160
76.906
65.888
65.681
65.437
65.190
61.591
24.846
18.490
13.715
13.361
Et
CO2Et
F3C
N
O
Me
4r
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

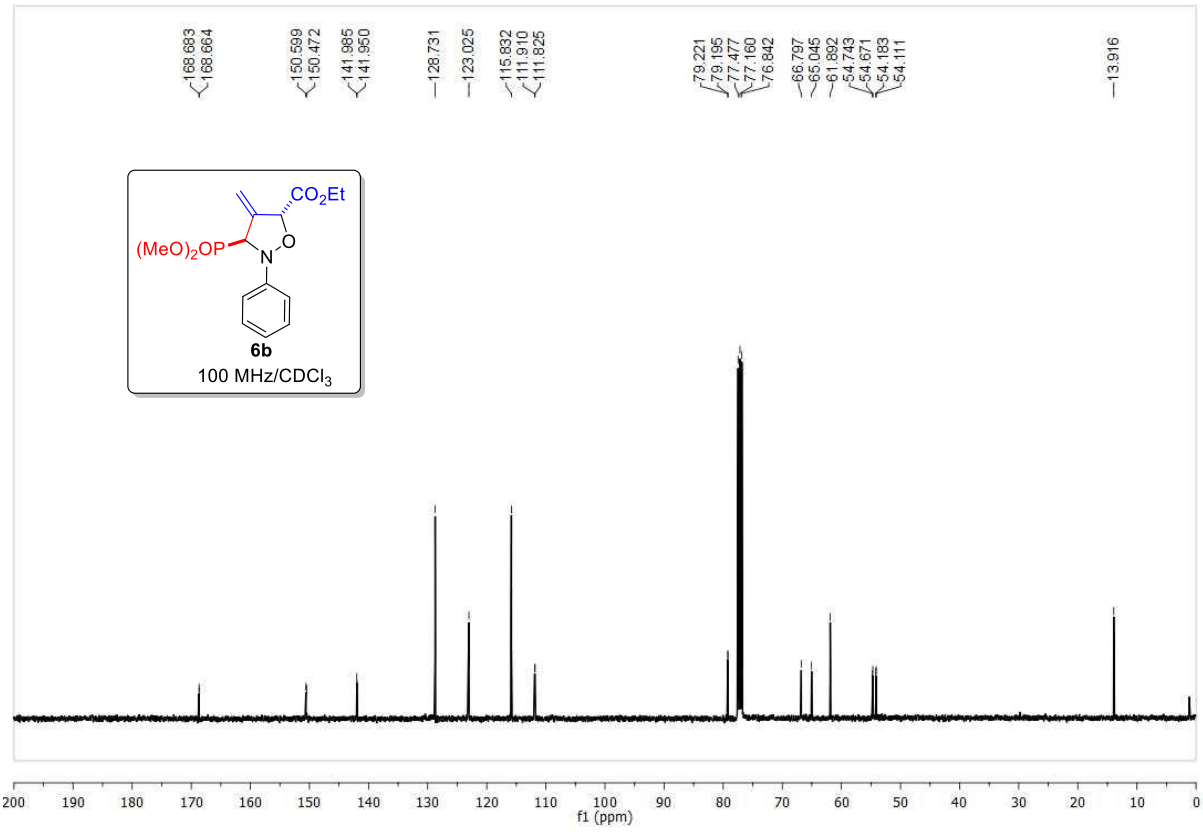

74.466
Et
CO2Et
F3C
N
O
Me
4r
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

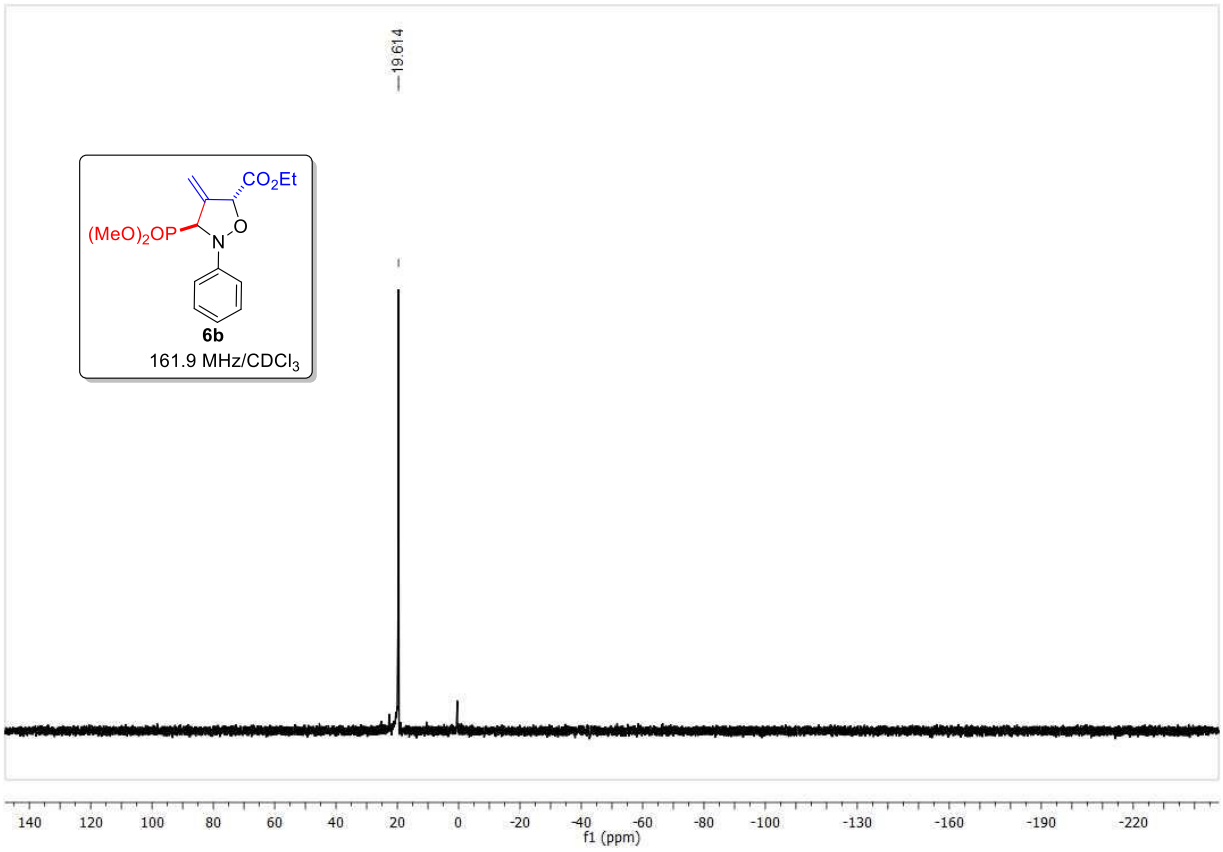

7.260
7.237
7.216
7.198
7.037
7.017
6.934
6.916
6.898
6.876
6.853
6.011
6.006
5.999
5.993
5.988
5.115
4.829
4.804
3.913
3.903
3.895
3.886
3.876
3.868
3.849
3.845
3.823
3.819
3.802
3.783
3.766
3.757
3.748
3.739
2.027
2.020
2.011
2.002
1.440
1.421
1.403
1.385
0.900
0.895
0.883
0.877
0.865
0.859
n-Pr
CO2Et
(MeO)2OP
N
O
6a
400 MHz/CDCl3
Grease
2.02
2.09
1.01
1.01
1.02
1.00
8.01
2.02
2.09
6.08
f1 (ppm)

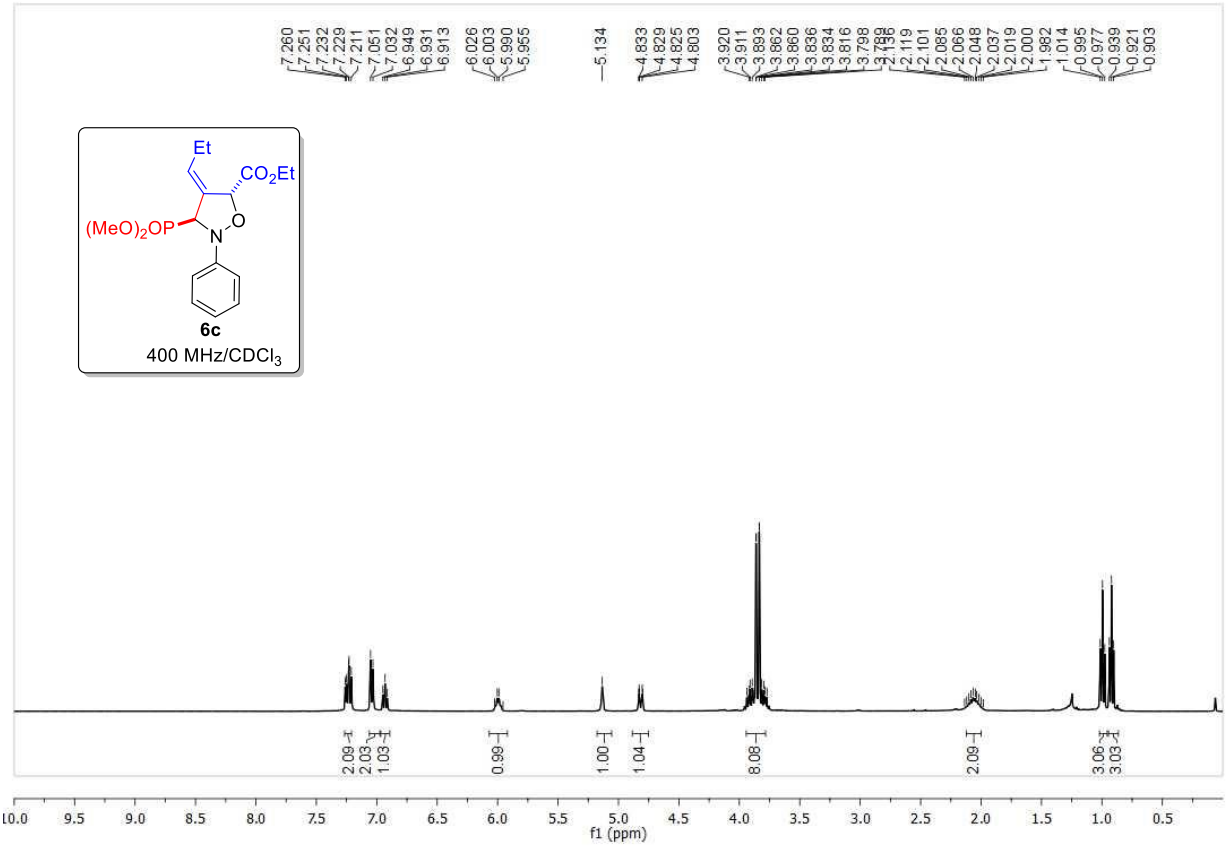

168.957
150.948
150.823
133.391
133.366
128.662
128.304
128.229
122.023
114.113
78.201
78.174
77.478
77.160
76.842
65.058
63.291
61.570
54.694
54.622
53.911
53.837
32.849
22.131
22.102
13.664
13.432
n-Pr
CO2Et
(MeO)2OP
N
O
6a
100 MHz/CDCl3
f1 (ppm)

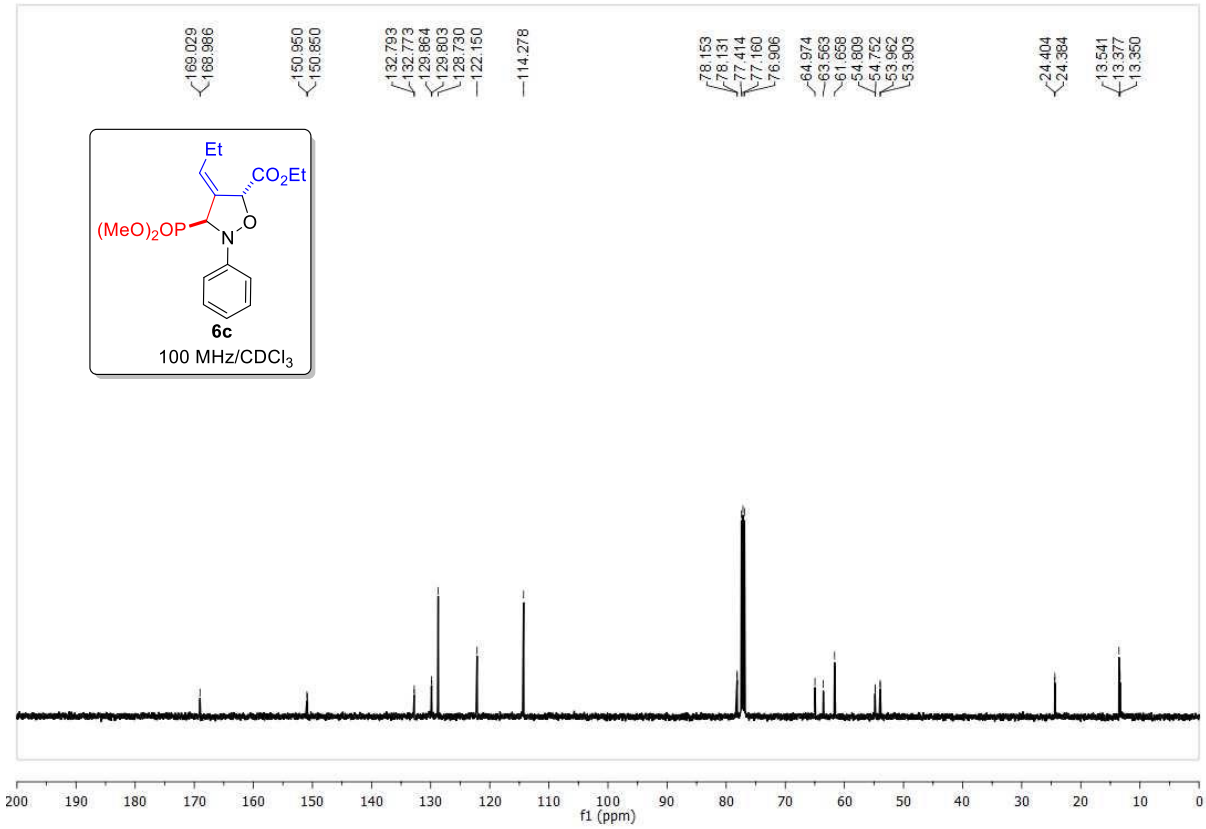

20.361
n-Pr
CO2Et
(MeO)2OP
N
O
6a
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

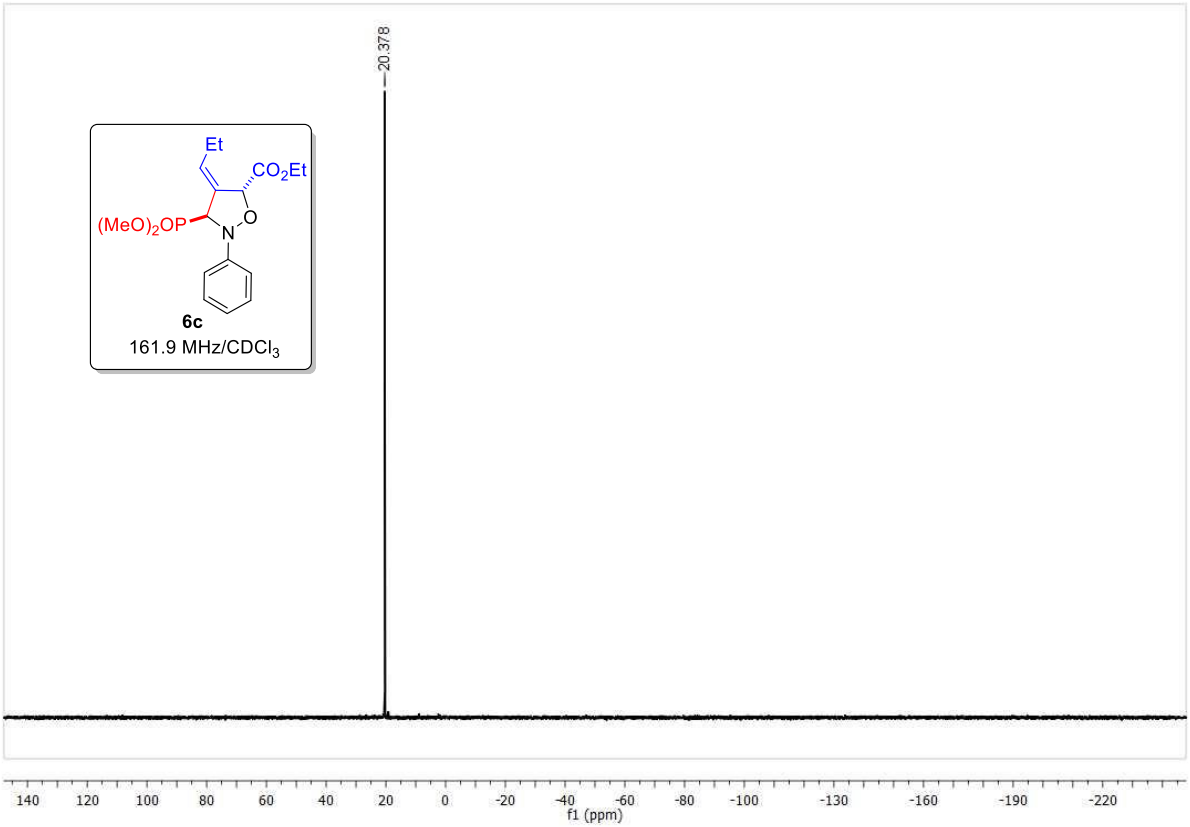

7.292 7.287 7.274 7.270 7.260 7.252 7.218 7.215 7.196 7.012 6.994 6.976
5.592 5.586 5.579 5.536 5.534 5.522
5.151 5.146
4.776 4.772 4.746 4.093 4.091 4.075 4.073 4.058 4.055 4.040 3.883 3.877 3.857 3.851
1.138 1.120 1.102
CO2Et
(MeO)2OP
N
O
6b
400 MHz/CDCl3
2.35 2.05 1.08 2.07 1.00 1.05 2.09 3.02 3.01 3.04
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
f1 (ppm)

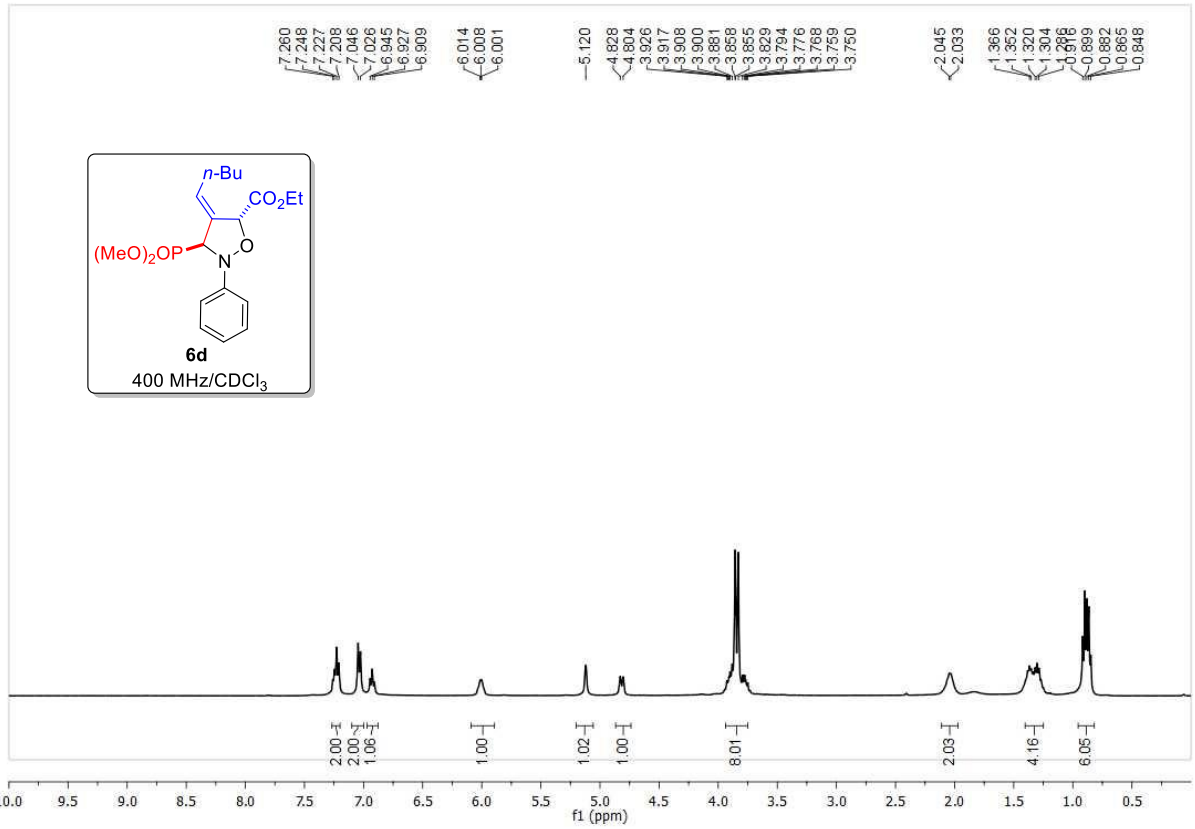

168.683
168.664
150.599
150.472
141.985
141.950
128.731
123.025
115.832
111.910
111.825
79.221
79.195
77.477
77.160
76.842
66.797
65.045
61.892
54.743
54.671
54.183
54.111
13.916
CO2Et
(MeO)2OP
N
O
6b
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

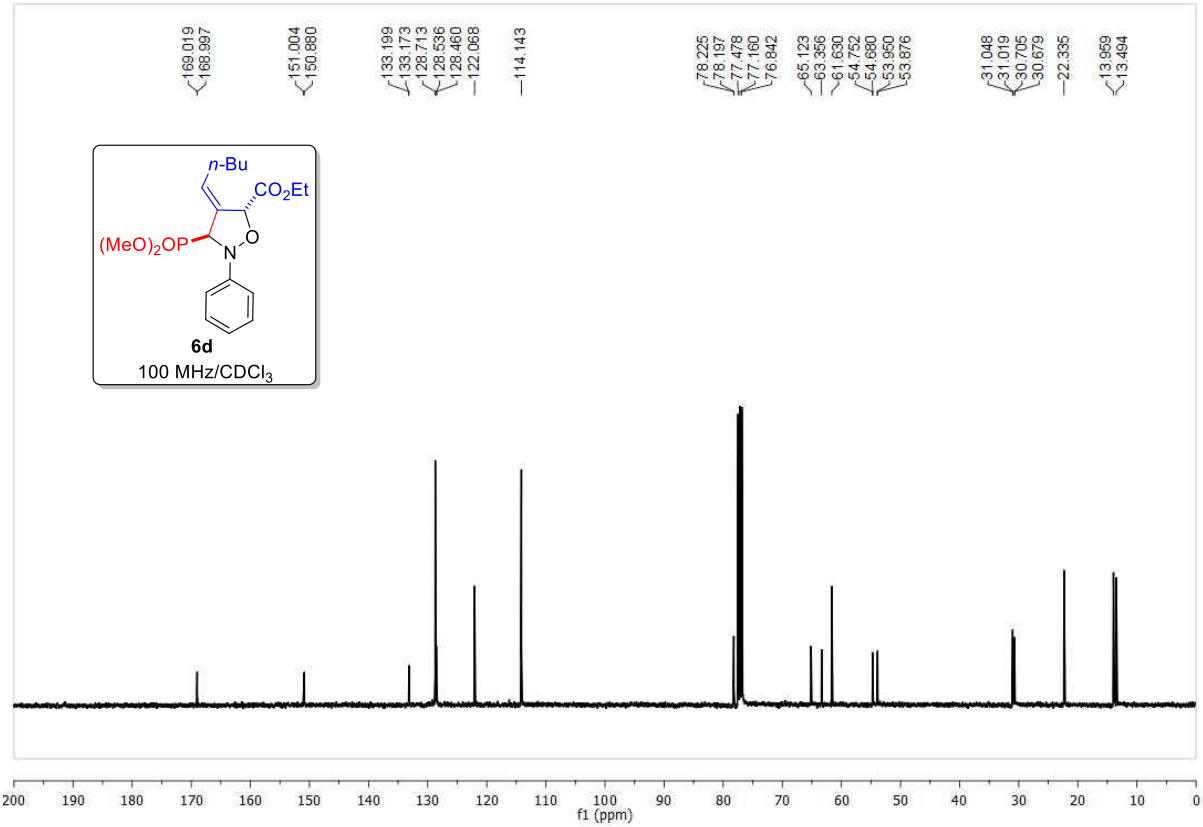

19.614
CO2Et
(MeO)2OP
N
O
6b
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

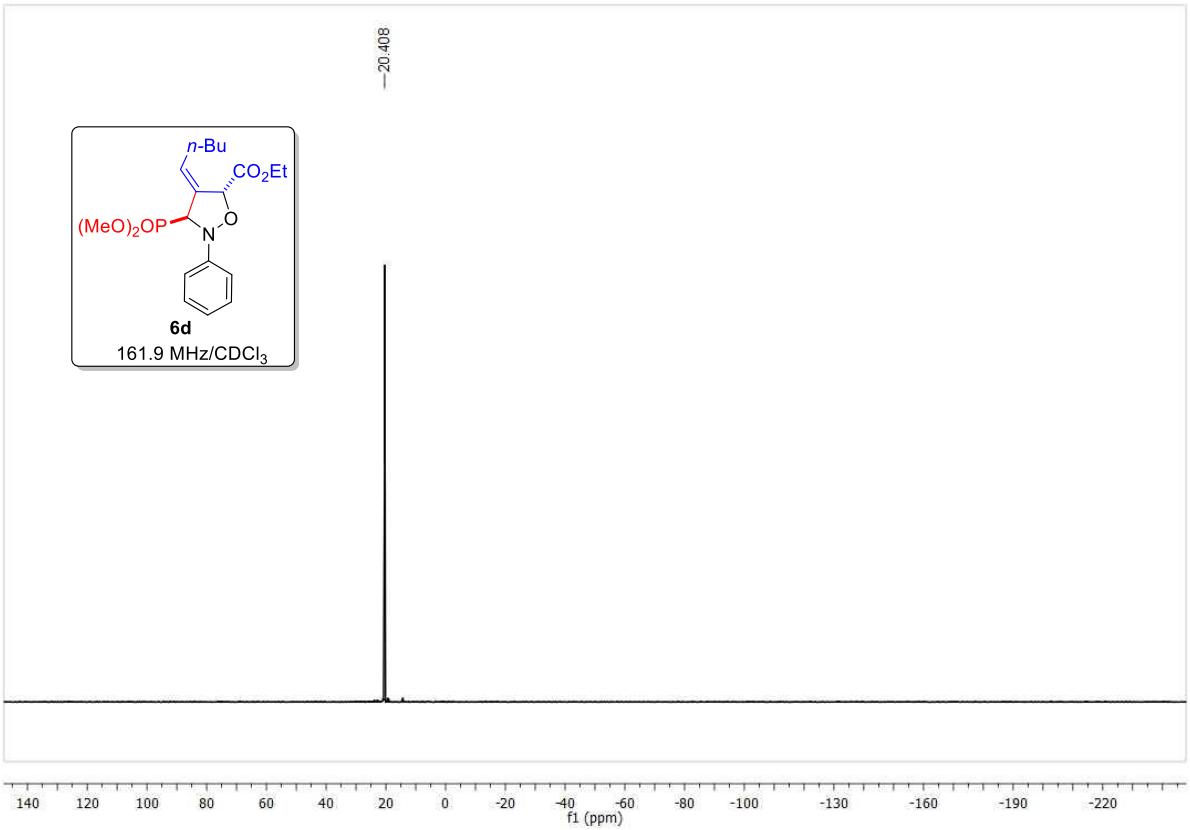

Et
CO2Et
(MeO)2OP
N
O
6c
400 MHz/CDCl3
f1 (ppm)

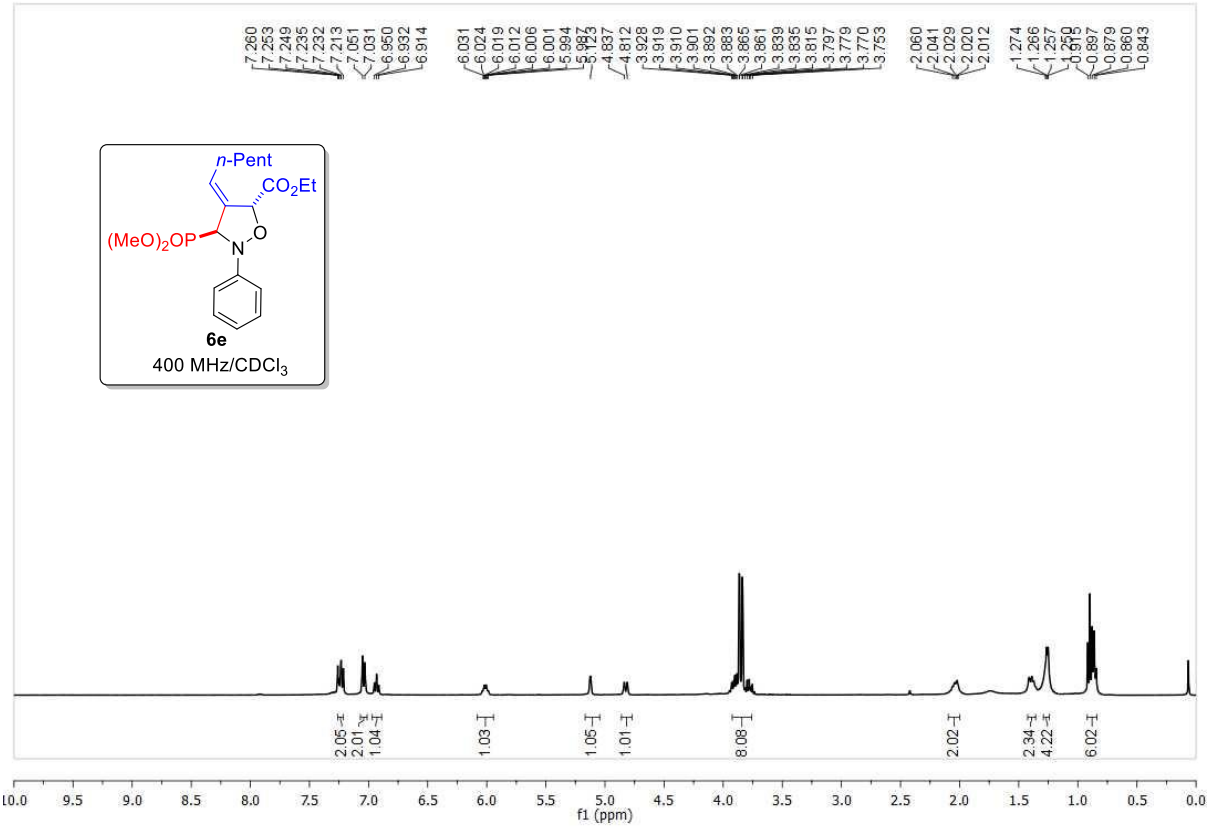

Et
CO2Et
(MeO)2OP
N
O
6c
100 MHz/CDCl3
f1 (ppm)

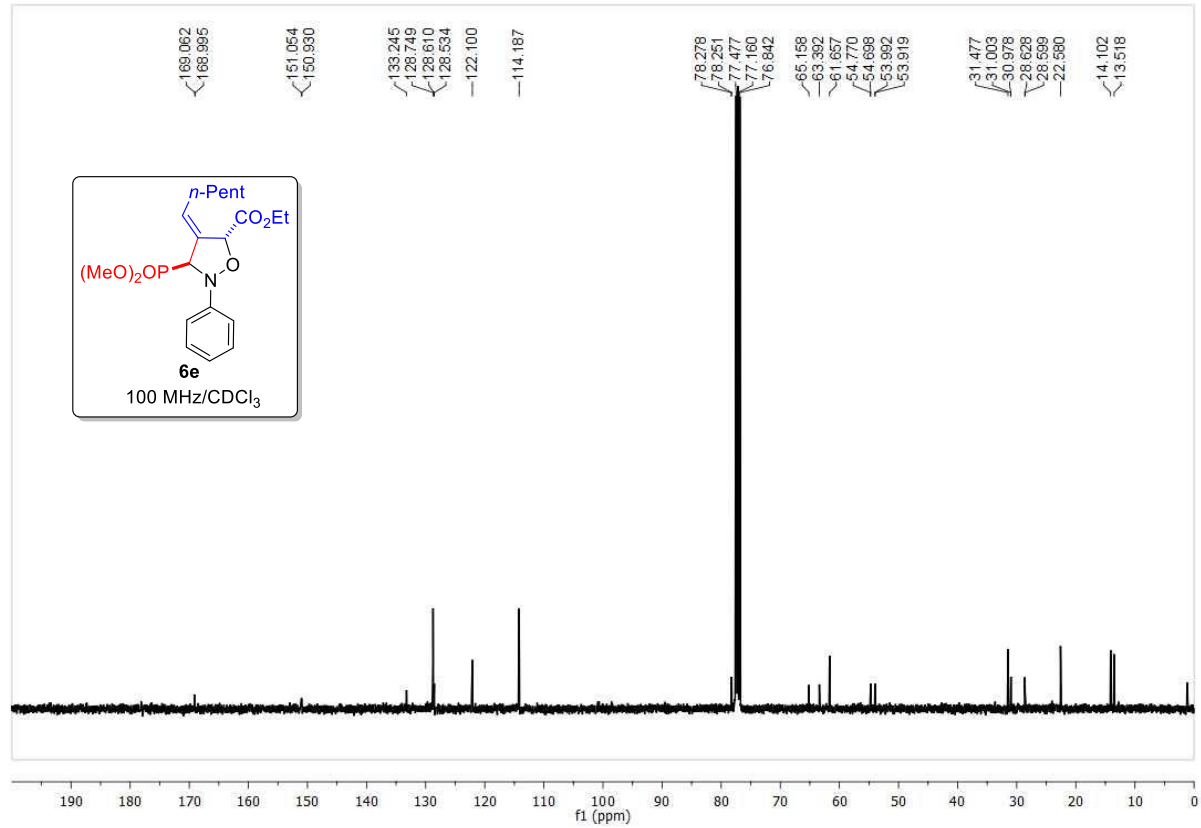

20.378
Et
CO2Et
(MeO)2OP
N
O
6c
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

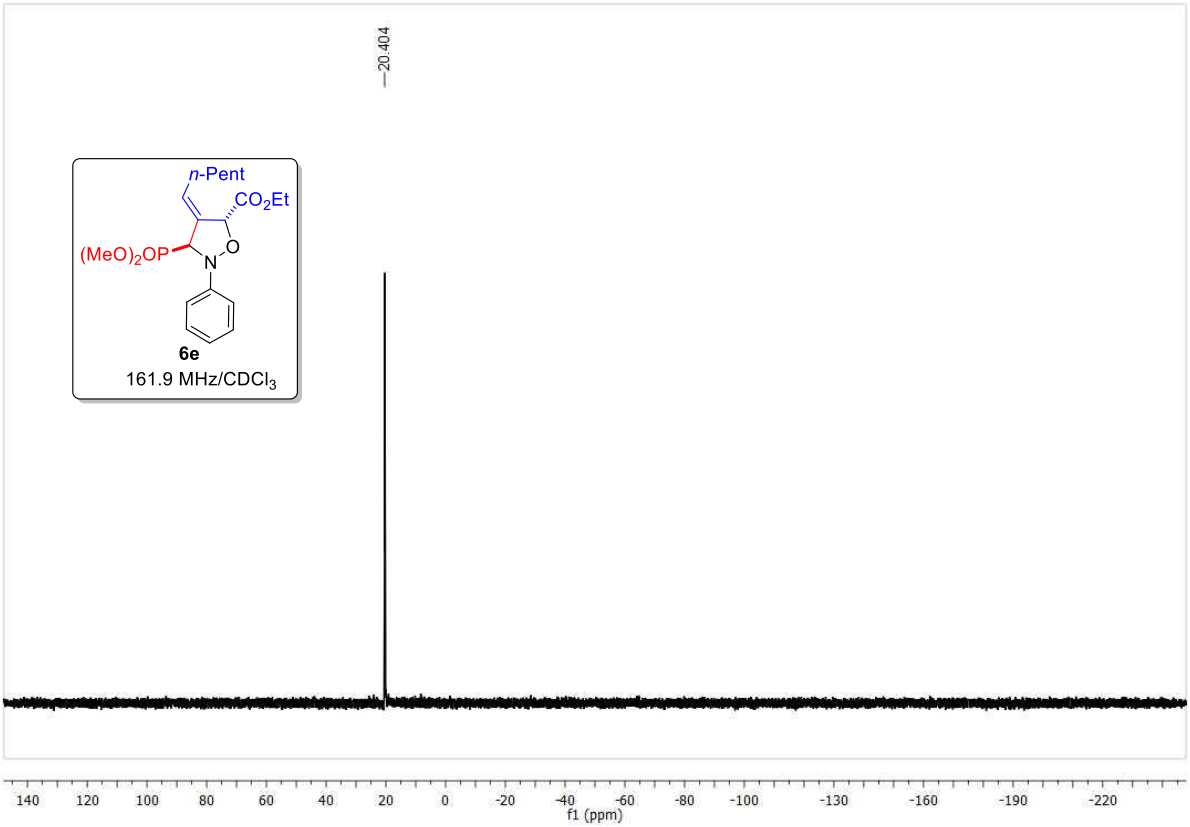

7.260 7.248 7.227 7.208 7.046 7.026 6.945 6.927 6.909
6.014 6.008 6.001
5.120
4.828 4.804
3.926 3.917 3.908 3.900 3.881 3.858 3.855 3.829 3.794 3.776 3.768 3.759 3.750
2.045 2.033
1.366 1.352 1.320 1.304 1.286 0.916 0.899 0.882 0.865 0.848
n-Bu
CO2Et
(MeO)2OP
N
O
6d
400 MHz/CDCl3
2.00 2.00 1.06 1.00 1.02 1.00 8.01 2.03 4.16 6.05
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
f1 (ppm)

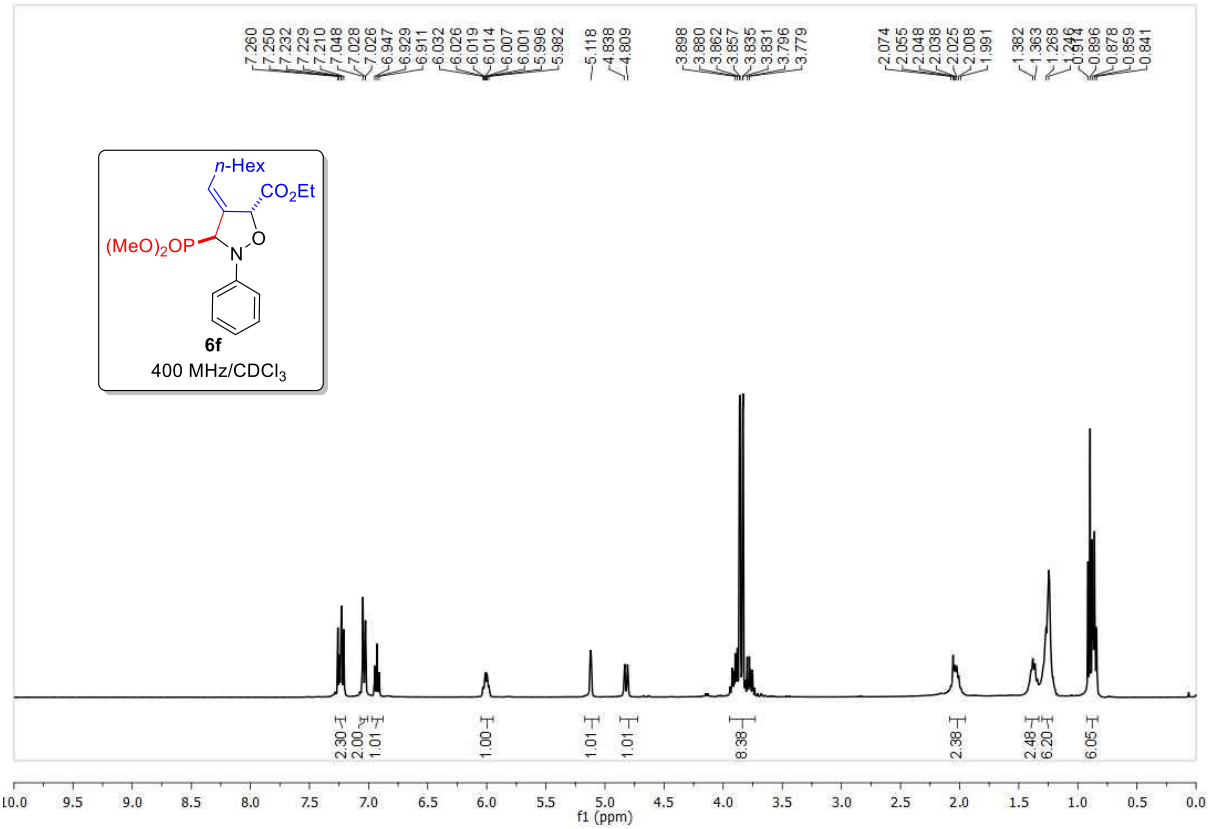

169.019
168.997
151.004
150.880
133.199
133.173
128.713
128.536
128.460
122.068
114.143
78.225
78.197
77.478
77.160
76.842
65.123
63.356
61.630
54.752
54.680
53.950
53.876
31.048
31.019
30.705
30.679
22.335
13.959
13.494
n-Bu
CO2Et
(MeO)2OP
N
O
6d
100 MHz/CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

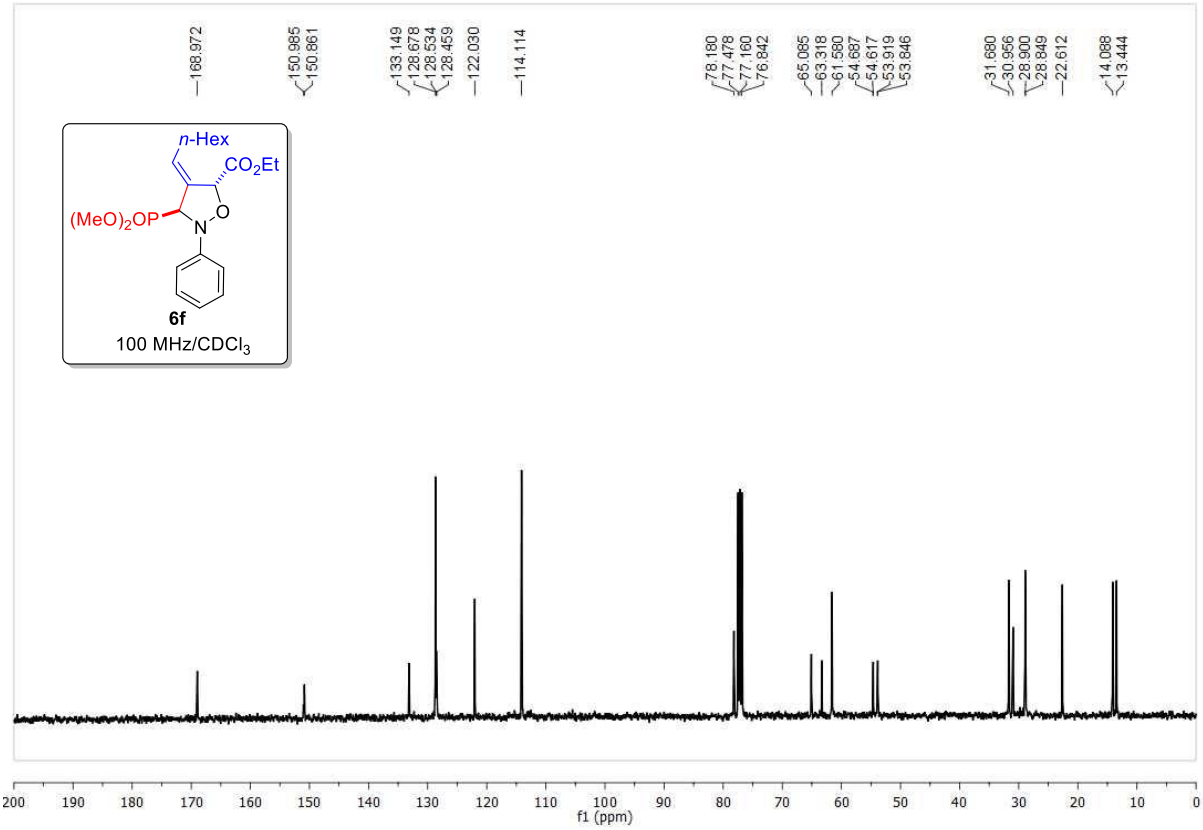

20.408
n-Bu
CO2Et
(MeO)2OP
N
O
6d
161.9 MHz/CDCl3
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

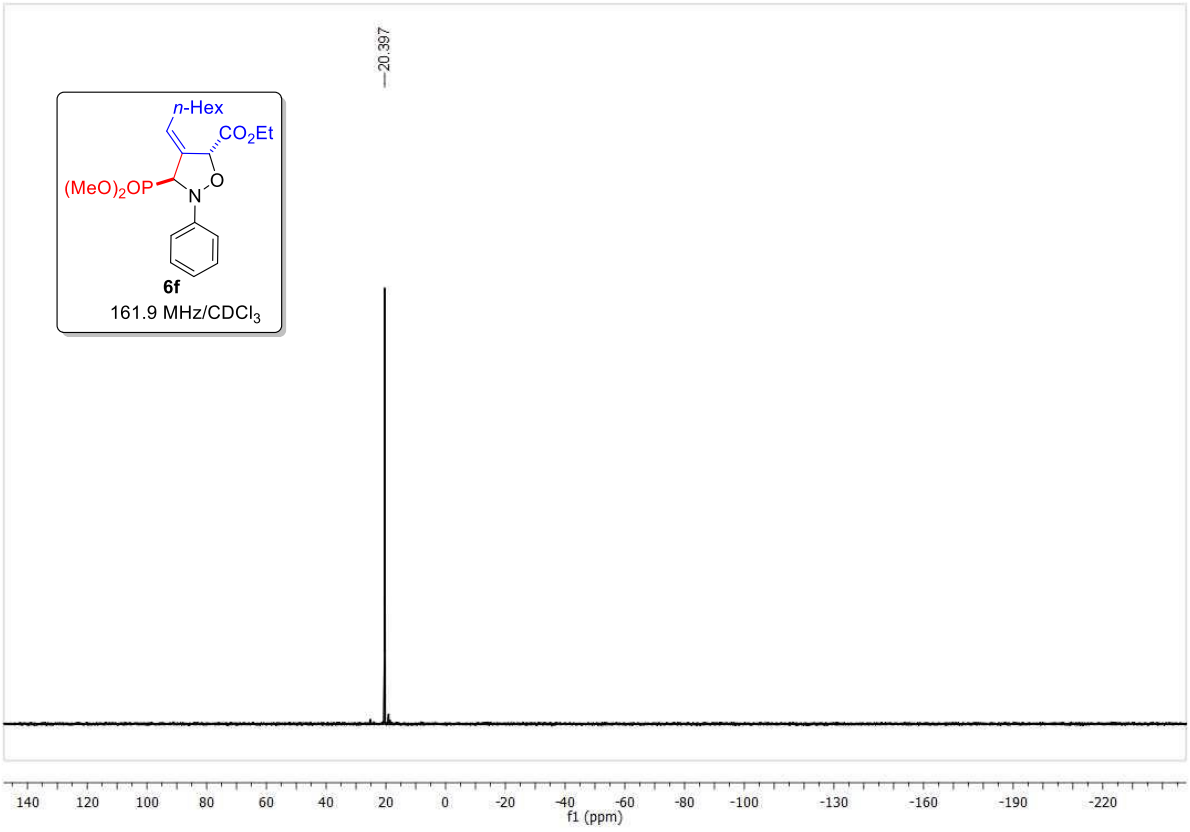

n-Pent
CO2Et
(MeO)2OP
N
O
6e
400 MHz/CDCl3
f1 (ppm)

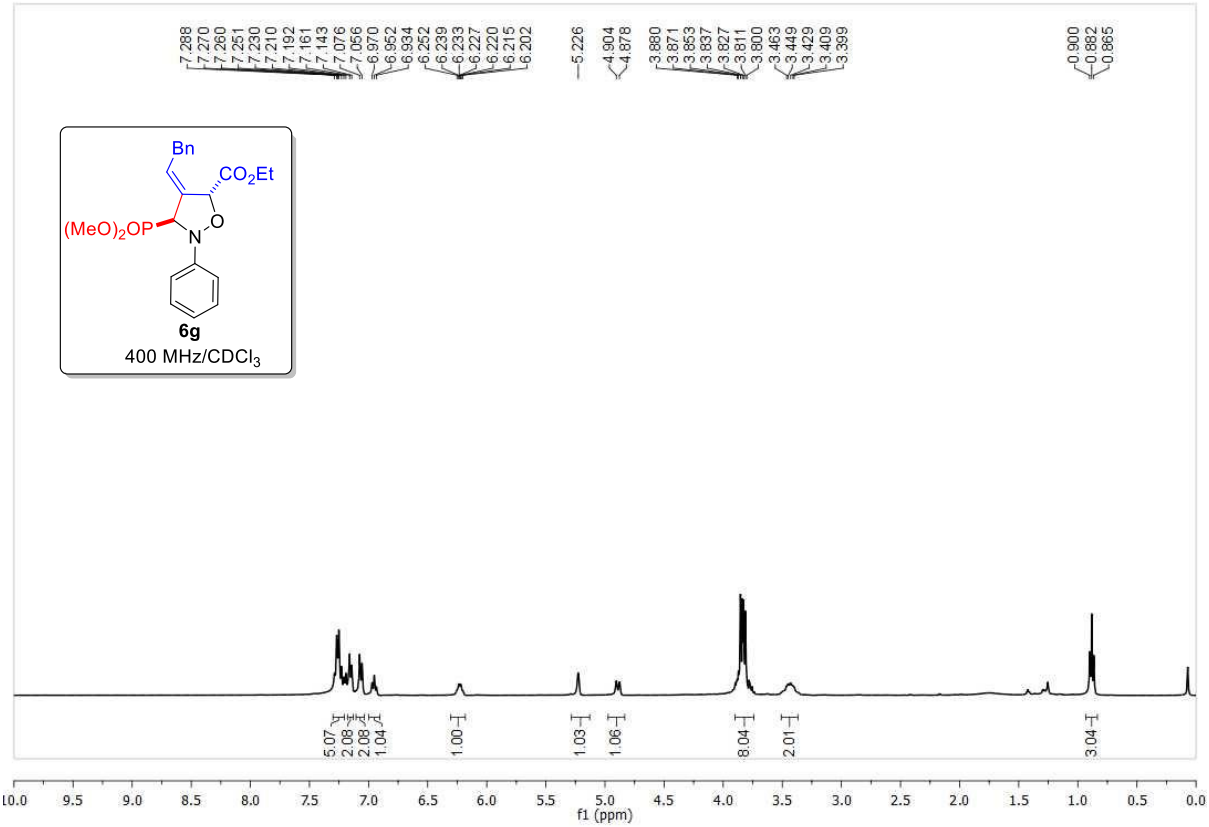

n-Pent
CO2Et
(MeO)2OP
N
O
6e
100 MHz/CDCl3
f1 (ppm)

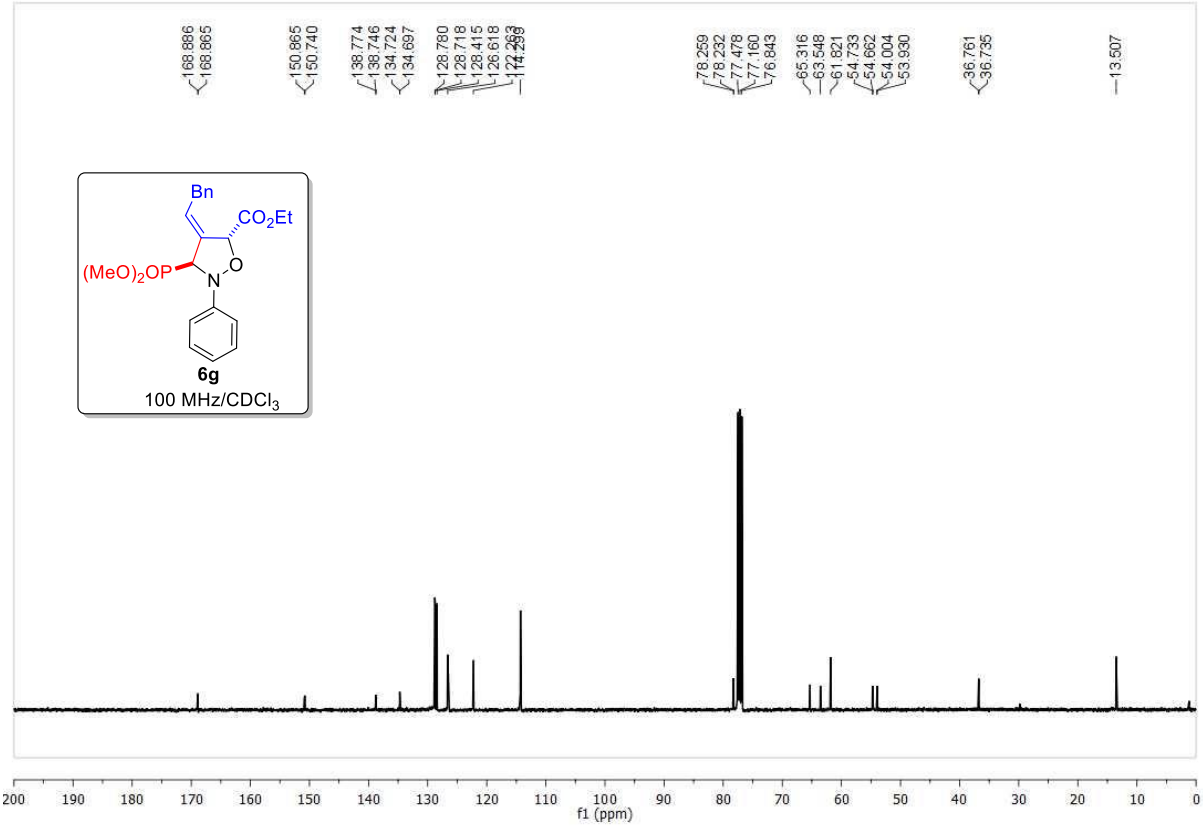

20.404
n-Pent
CO2Et
(MeO)2OP
N
O
6e
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

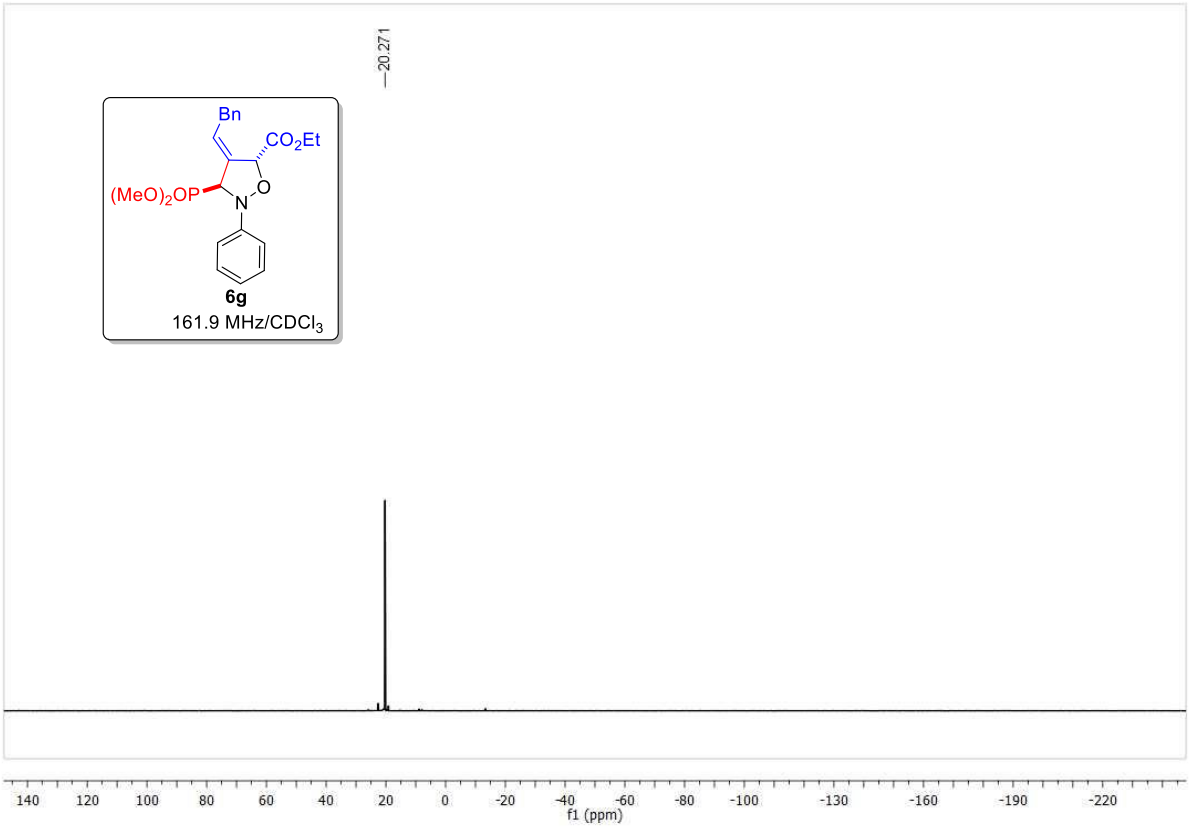

7.260 7.250 7.232 7.229 7.210 7.048 7.028 7.026 6.947 6.929 6.911 6.032 6.026 6.019 6.014 6.007 6.001 5.996 5.982
5.118 4.838 4.809
3.898 3.880 3.862 3.857 3.835 3.831 3.796 3.779
2.074 2.055 2.048 2.038 2.025 2.008 1.991
1.382 1.363 1.268 0.896 0.878 0.859 0.841
n-Hex
CO2Et
(MeO)2OP
N
O
6f
400 MHz/CDCl3
2.30 2.00 1.01 1.00 1.01 1.01 8.38 2.38 2.48 6.20 6.05
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
f1 (ppm)

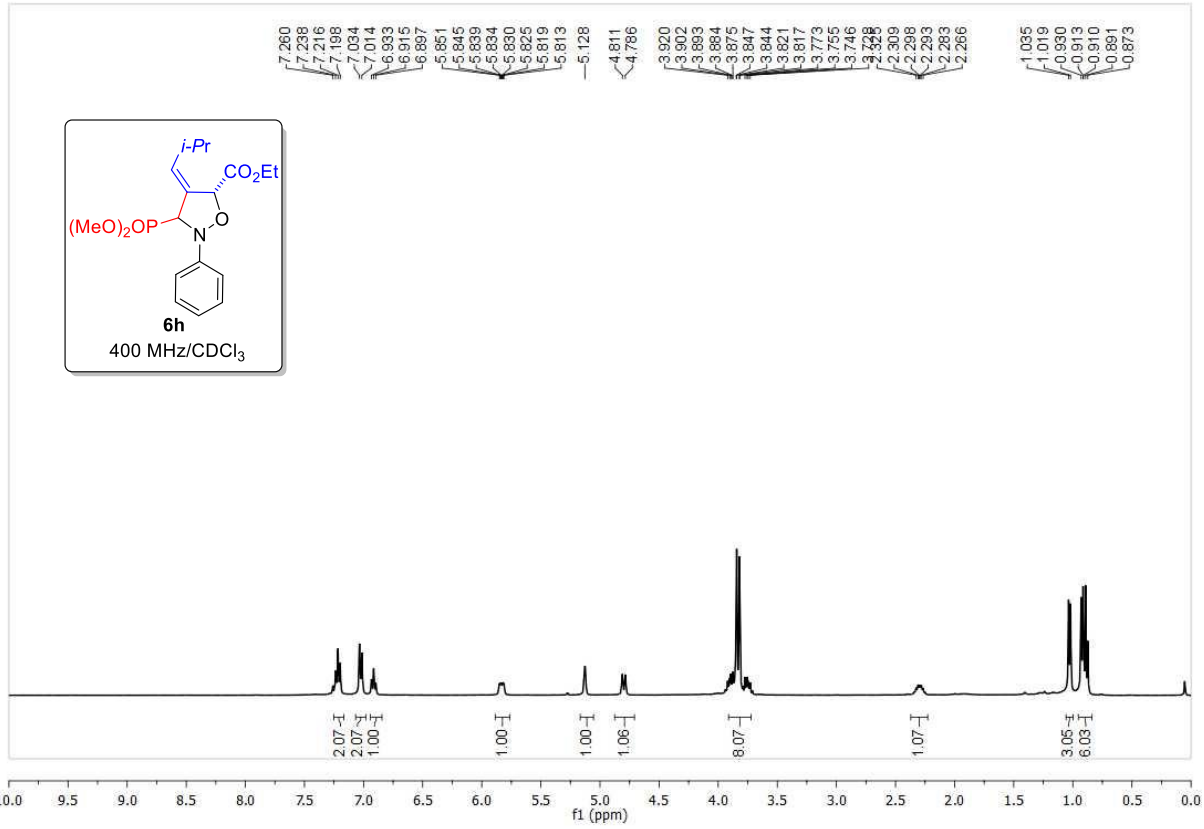

168.972
150.985
150.861
133.149
128.678
128.534
128.459
122.030
114.114
78.180
77.478
77.160
76.842
65.085
63.318
61.580
54.687
54.617
53.919
53.846
31.680
30.956
28.900
28.849
22.612
14.088
13.444
n-Hex
CO2Et
(MeO)2OP
N
O
6f
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

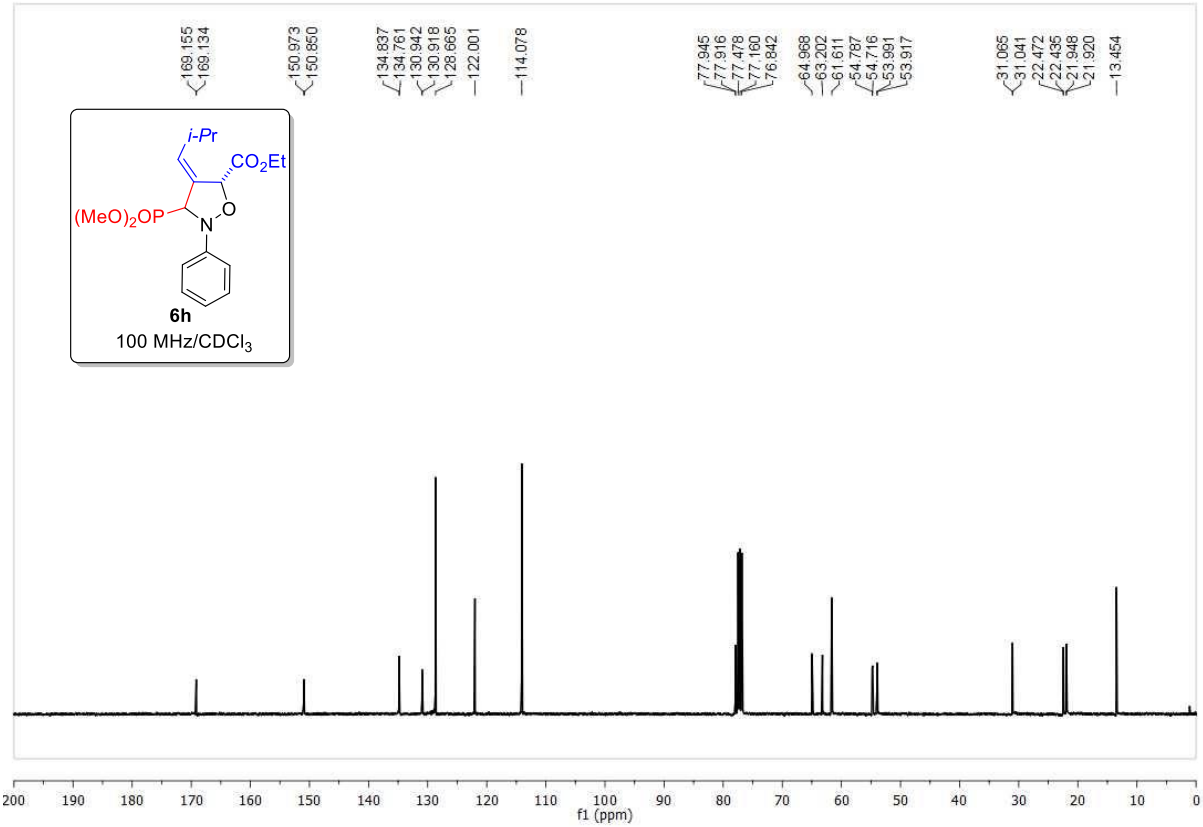

20.397
n-Hex
CO2Et
(MeO)2OP
N
O
6f
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

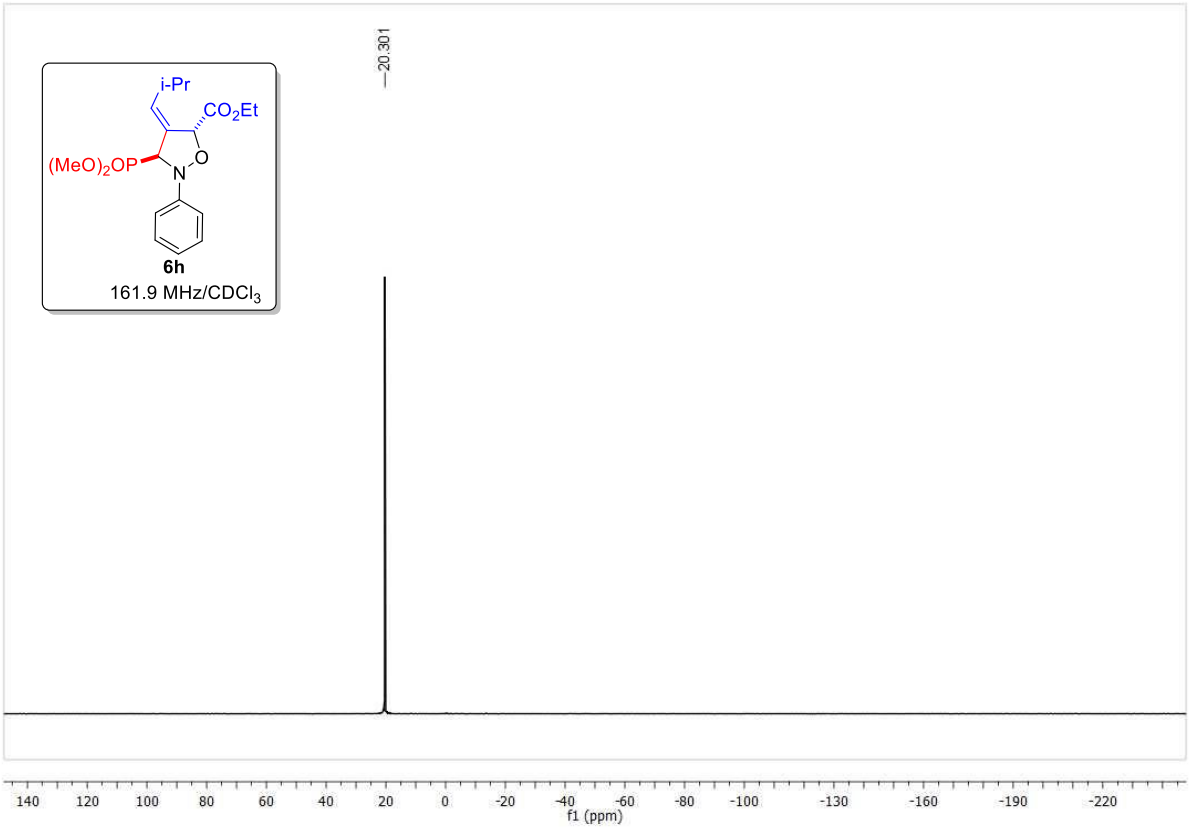

7.288
7.270
7.260
7.251
7.230
7.210
7.192
7.161
7.143
7.076
7.056
6.970
6.952
6.934
6.252
6.239
6.233
6.227
6.220
6.215
6.202
5.226
4.904
4.878
3.880
3.871
3.853
3.837
3.827
3.811
3.800
3.463
3.449
3.429
3.409
3.399
0.900
0.882
0.865
Bn
CO2Et
(MeO)2OP
N
O
6g
400 MHz/CDCl3
5.07
2.08
2.08
1.04
1.00
1.03
1.06
8.04
2.01
3.04
f1 (ppm)

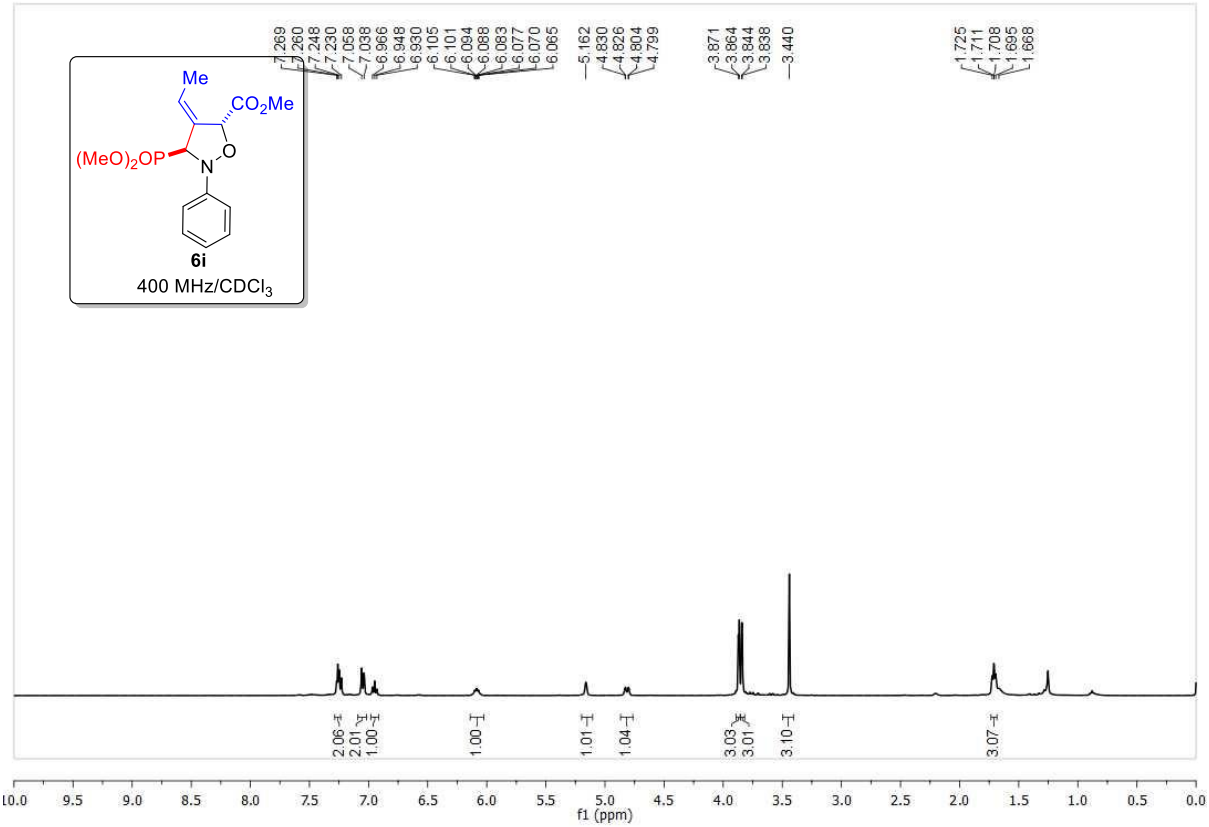

168.886
168.865
150.865
150.740
138.774
138.746
134.724
134.697
128.780
128.718
128.415
126.618
78.259
78.232
77.478
77.160
76.843
65.316
63.548
61.821
54.733
54.662
54.004
53.930
36.761
36.735
13.507
Bn
CO2Et
(MeO)2OP
N
O
6g
100 MHz/CDCl3
f1 (ppm)

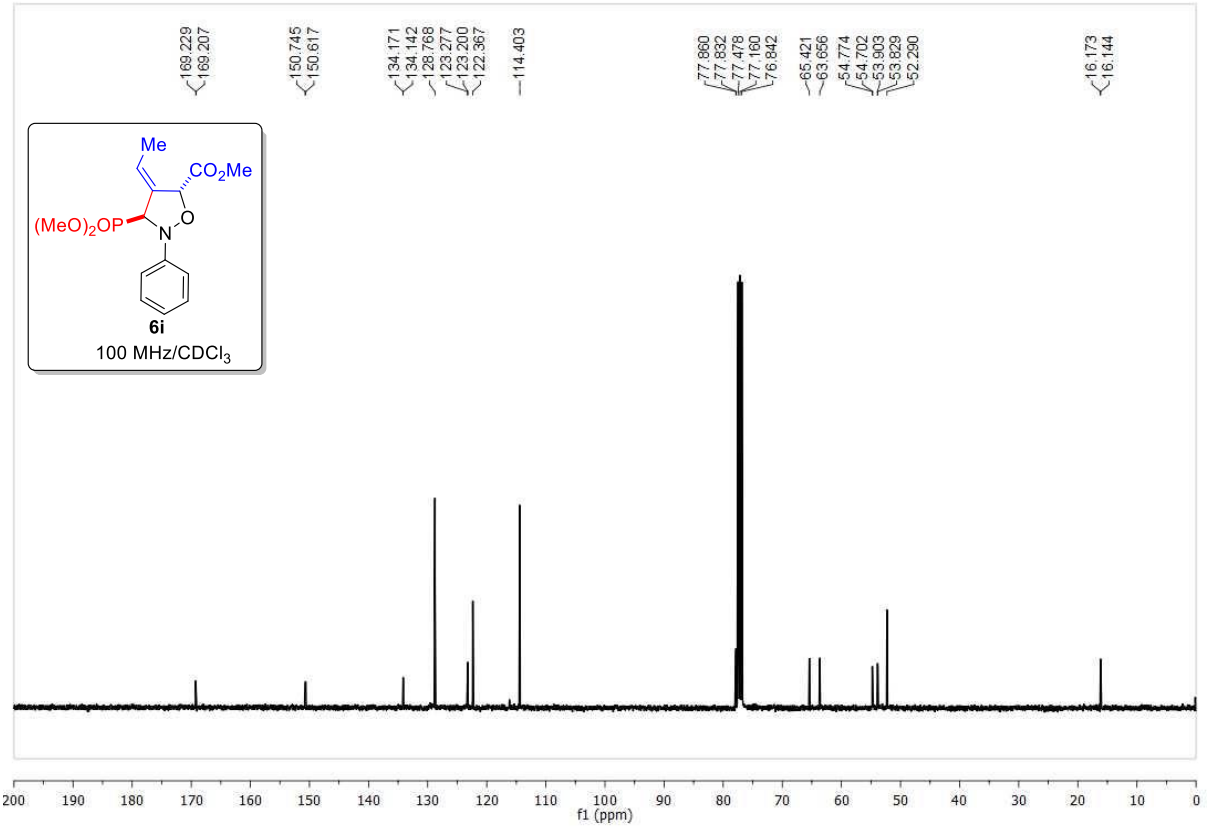

20.271
Bn
CO2Et
(MeO)2OP
N
O
6g
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

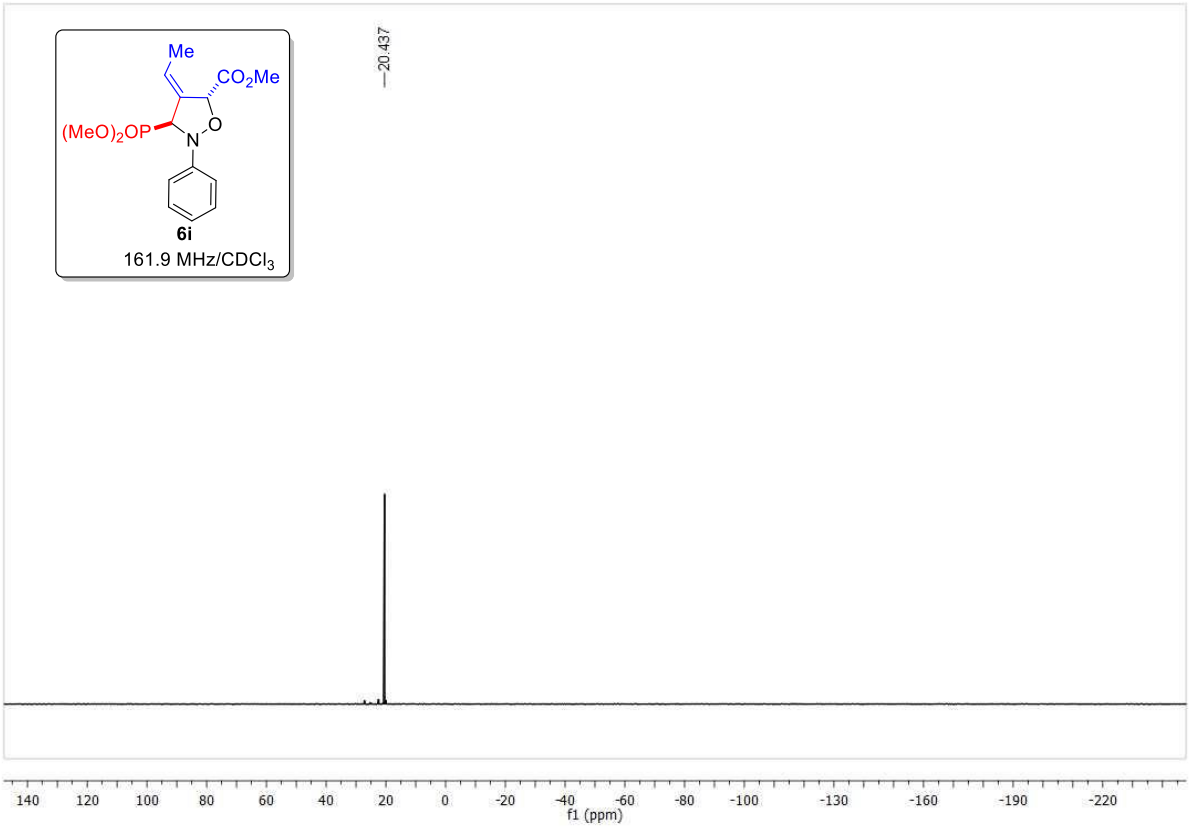

7.260 7.238 7.216 7.198 7.034 7.014 6.933 6.915 6.897 5.851 5.845 5.839 5.834 5.830 5.825 5.819 5.813 5.128 4.811 4.786 3.920 3.902 3.893 3.884 3.875 3.847 3.844 3.821 3.817 3.773 3.755 3.746 2.309 2.298 2.293 2.283 2.266 1.035 1.019 0.930 0.913 0.910 0.891 0.873
i-Pr
CO2Et
(MeO)2OP
N
O
6h
400 MHz/CDCl3
2.07 2.07 1.00 1.00 1.00 1.06 8.07 1.07 3.05 6.03
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
f1 (ppm)

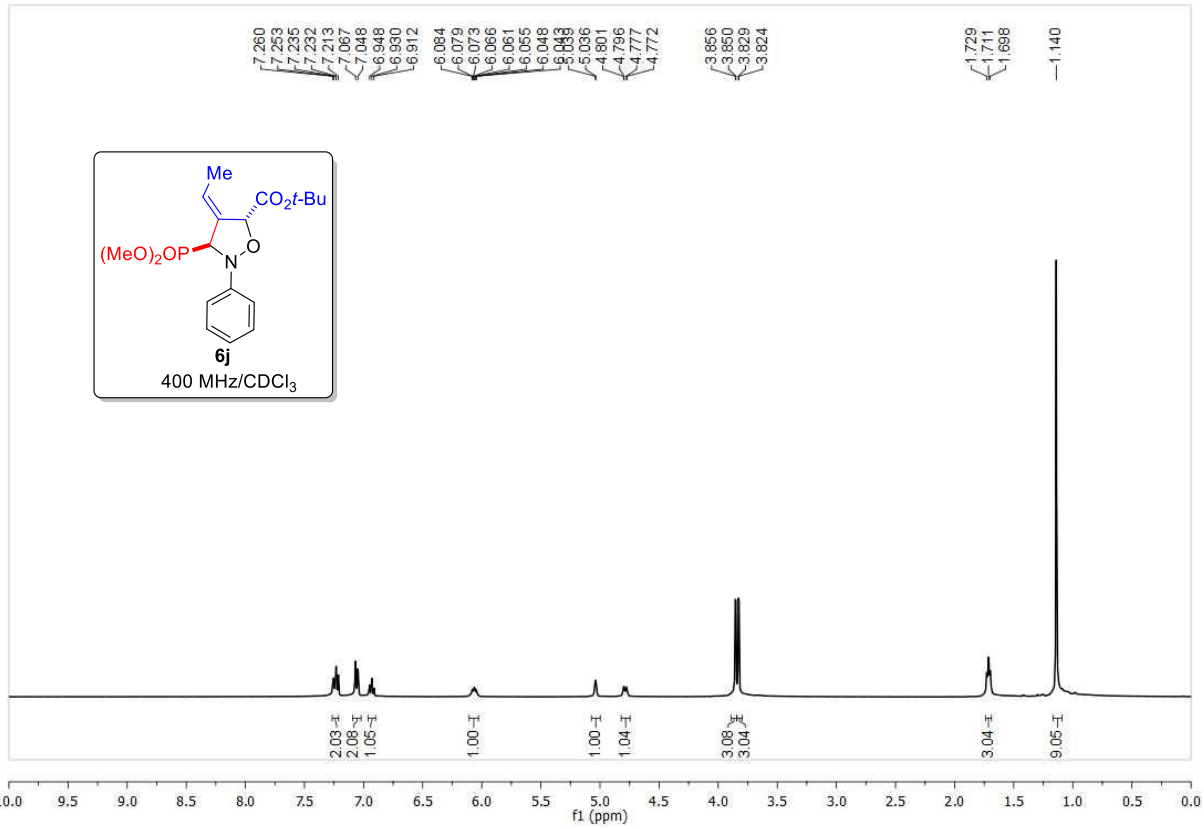

169.155
169.134
150.973
150.850
134.837
134.761
130.942
130.918
128.665
122.001
114.078
77.945
77.916
77.478
77.160
76.842
64.968
63.202
61.611
54.787
54.716
53.991
53.917
31.065
31.041
22.472
22.435
21.948
21.920
13.454
i-Pr
CO2Et
(MeO)2OP
N
O
6h
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

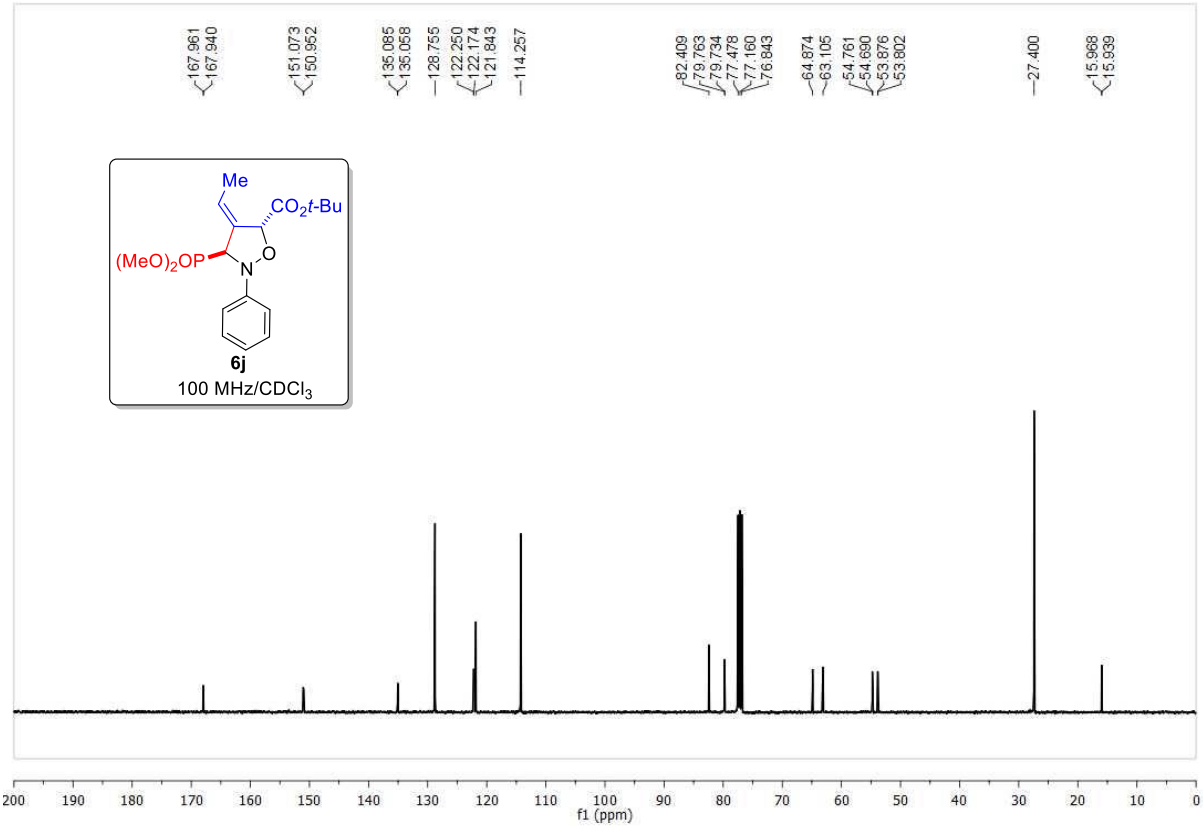

20.301
i-Pr
CO2Et
(MeO)2OP
N
O
6h
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

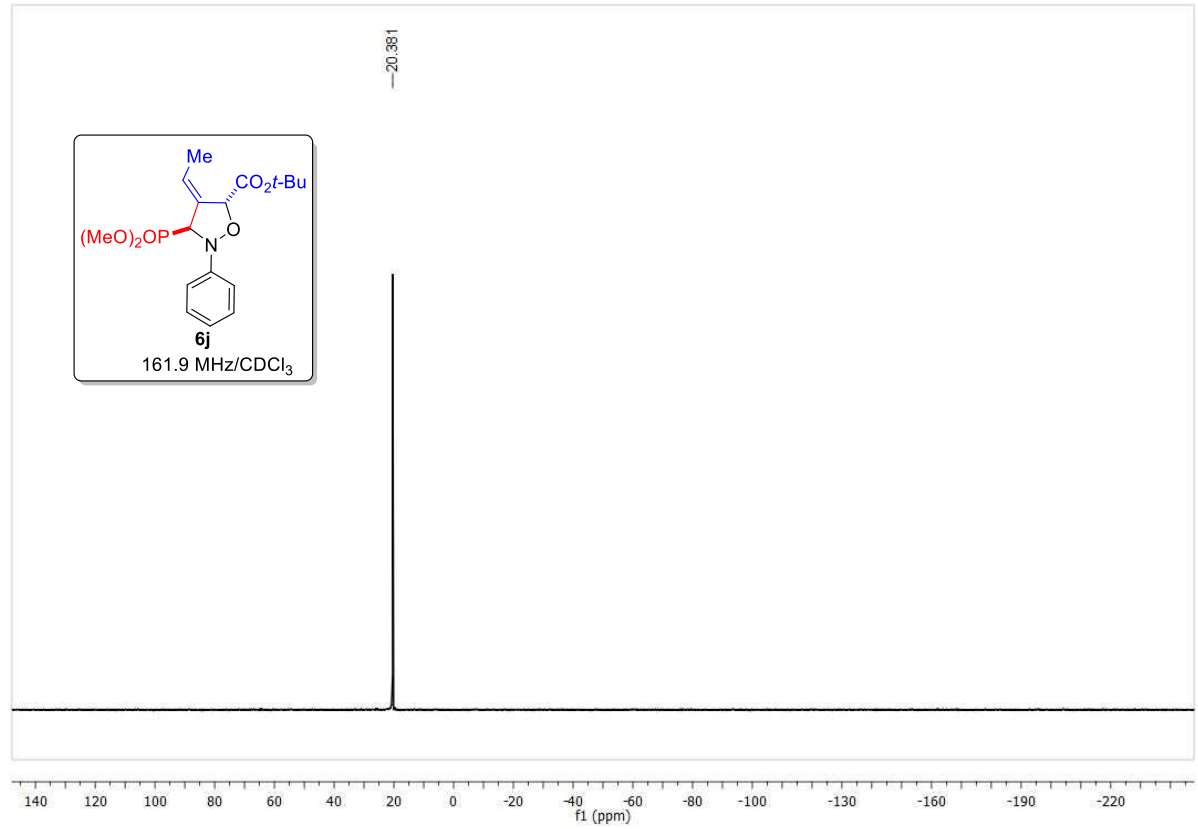

Me
CO2Me
(MeO)2OP
N
O
6i
400 MHz/CDCl3
7.269
7.260
7.248
7.230
7.058
7.038
6.966
6.948
6.930
6.105
6.101
6.094
6.088
6.083
6.077
6.070
6.065
5.162
4.830
4.826
4.804
4.799
3.871
3.864
3.844
3.838
3.440
1.725
1.711
1.708
1.695
1.668
2.06
2.01
1.00
1.00
1.01
1.04
3.03
3.01
3.10
3.07
f1 (ppm)

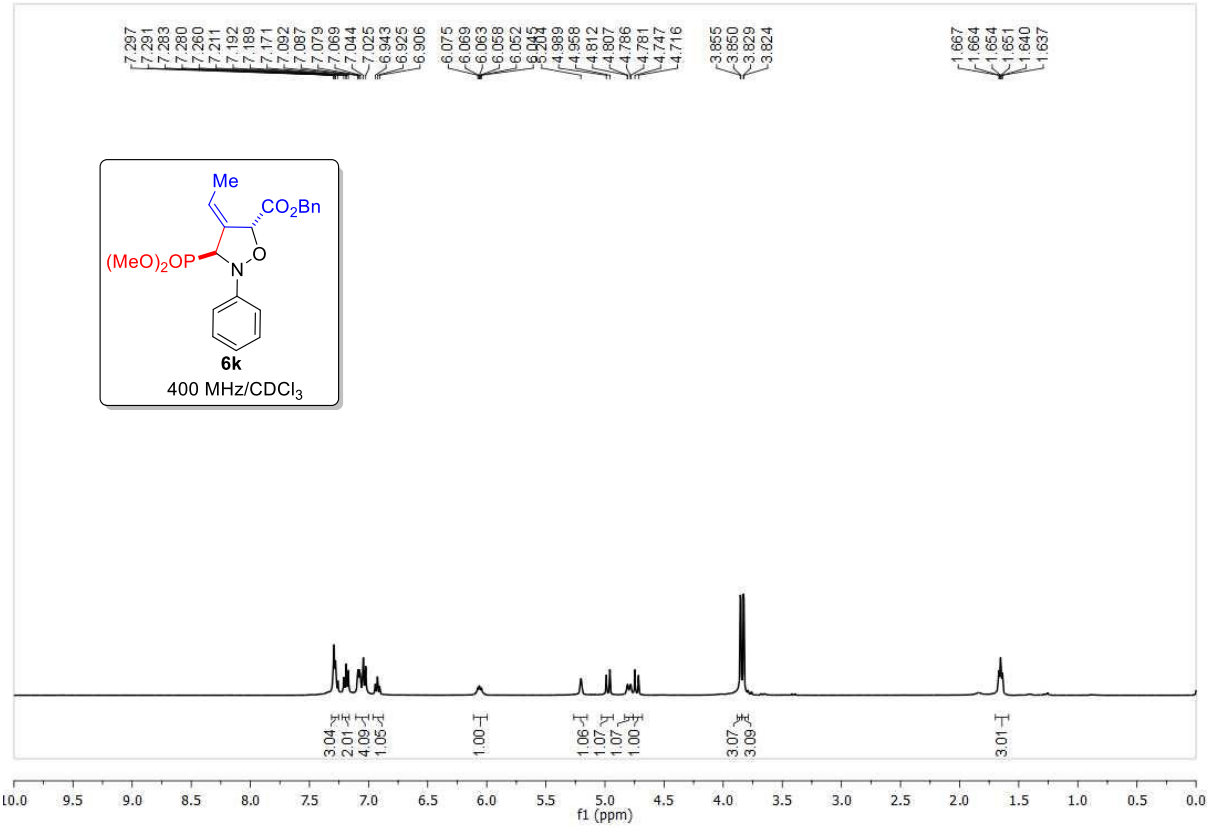

Me
CO2Me
(MeO)2OP
N
O
6i
100 MHz/CDCl3
169.229
169.207
150.745
150.617
134.171
134.142
128.768
123.277
123.200
122.367
114.403
77.860
77.832
77.478
77.160
76.842
65.421
63.656
54.774
54.702
53.903
53.829
52.290
16.173
16.144
f1 (ppm)

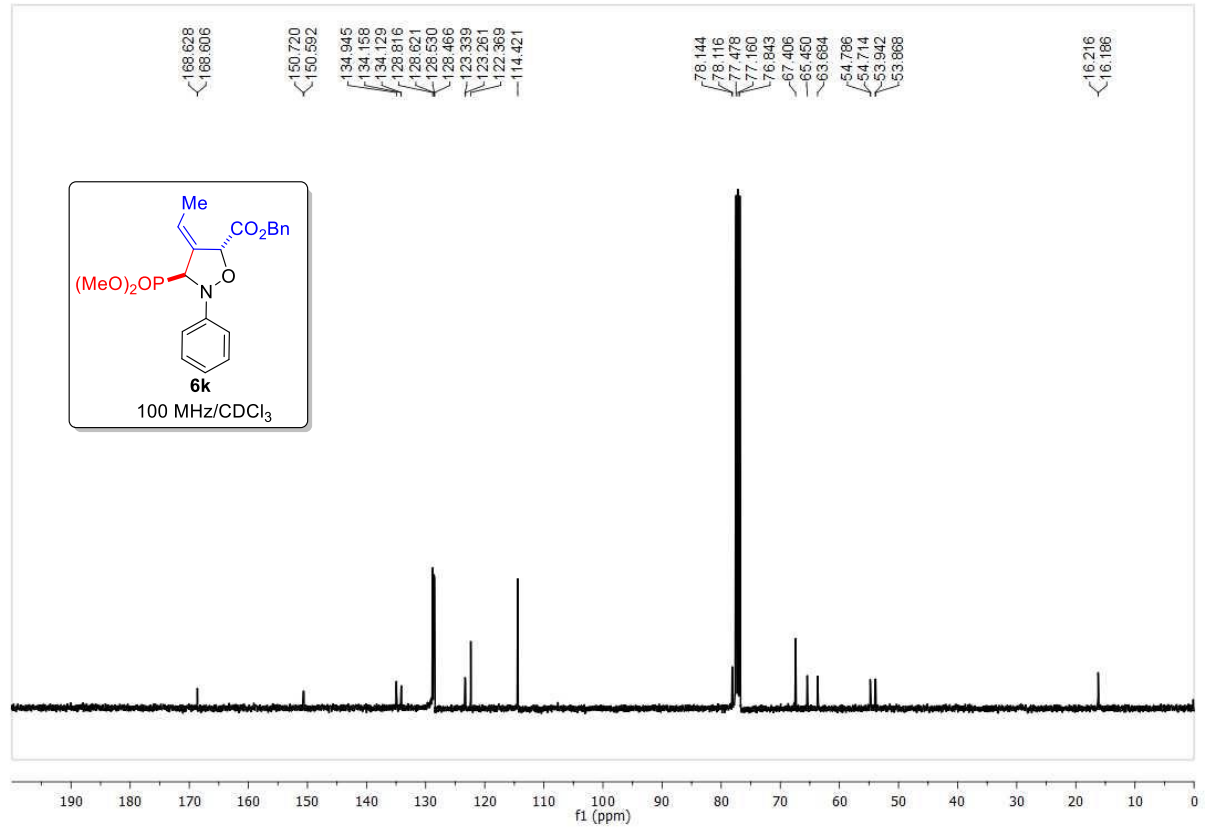

Me
CO2Me
(MeO)2OP
N
O
6i
161.9 MHz/CDCl3
20.437
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

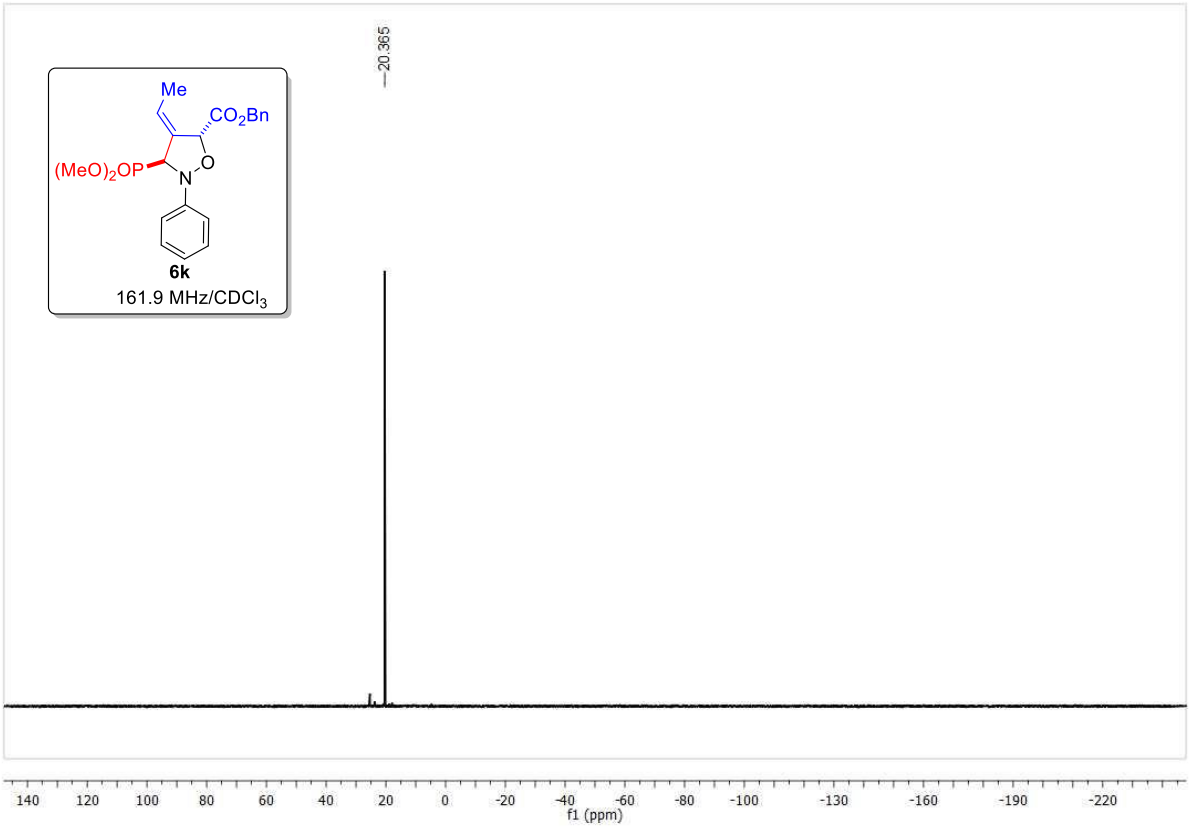

7.260
7.253
7.235
7.232
7.213
7.067
7.048
6.948
6.930
6.912
6.084
6.079
6.073
6.066
6.061
6.055
6.048
5.036
4.801
4.796
4.777
4.772
3.856
3.850
3.829
3.824
1.729
1.711
1.698
1.140
Me
CO2t-Bu
(MeO)2OP
N
O
6j
400 MHz/CDCl3
2.03
2.08
1.05
1.00
1.00
1.04
3.08
3.04
3.04
9.05
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 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
f1 (ppm)

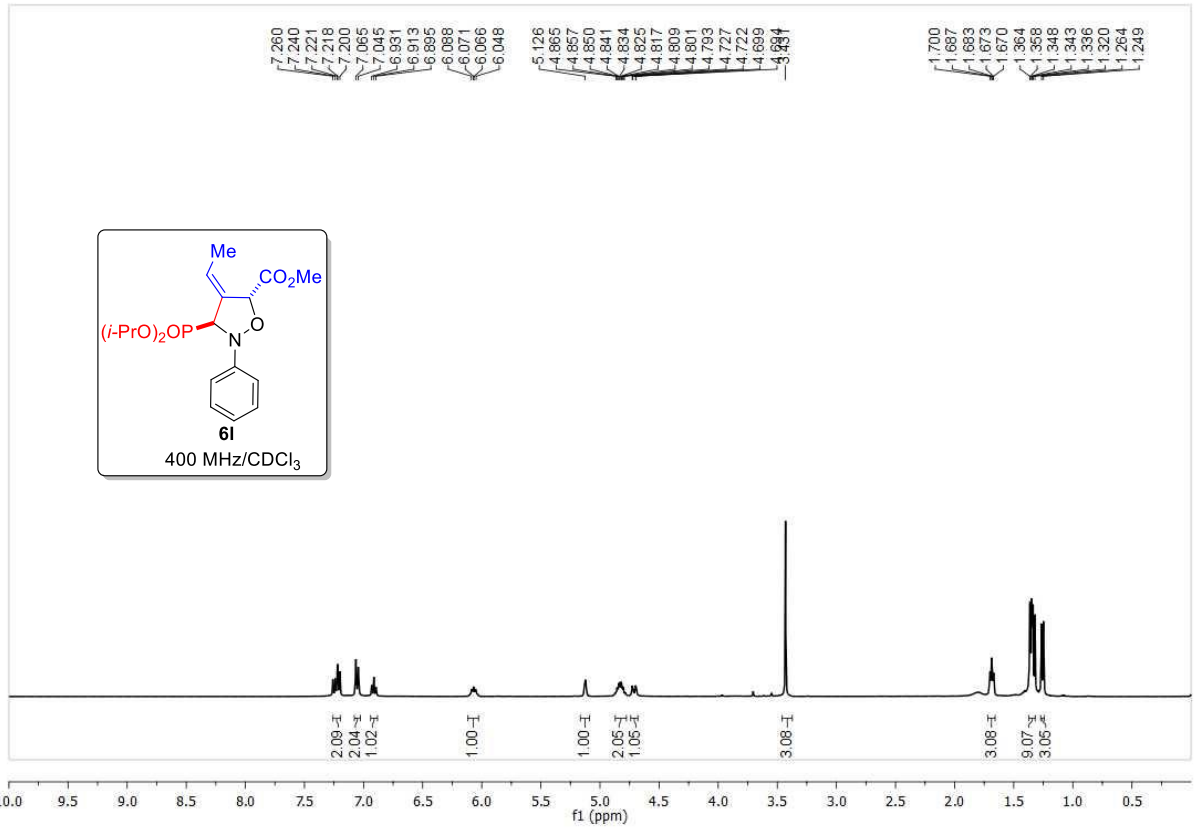

167.961
167.940
151.073
150.952
135.085
135.058
128.755
122.250
122.174
121.843
114.257
82.409
79.763
79.734
77.478
77.160
76.843
64.874
63.105
54.761
54.690
53.876
53.802
27.400
15.968
15.939
Me
CO2t-Bu
(MeO)2OP
N
O
6j
100 MHz/CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

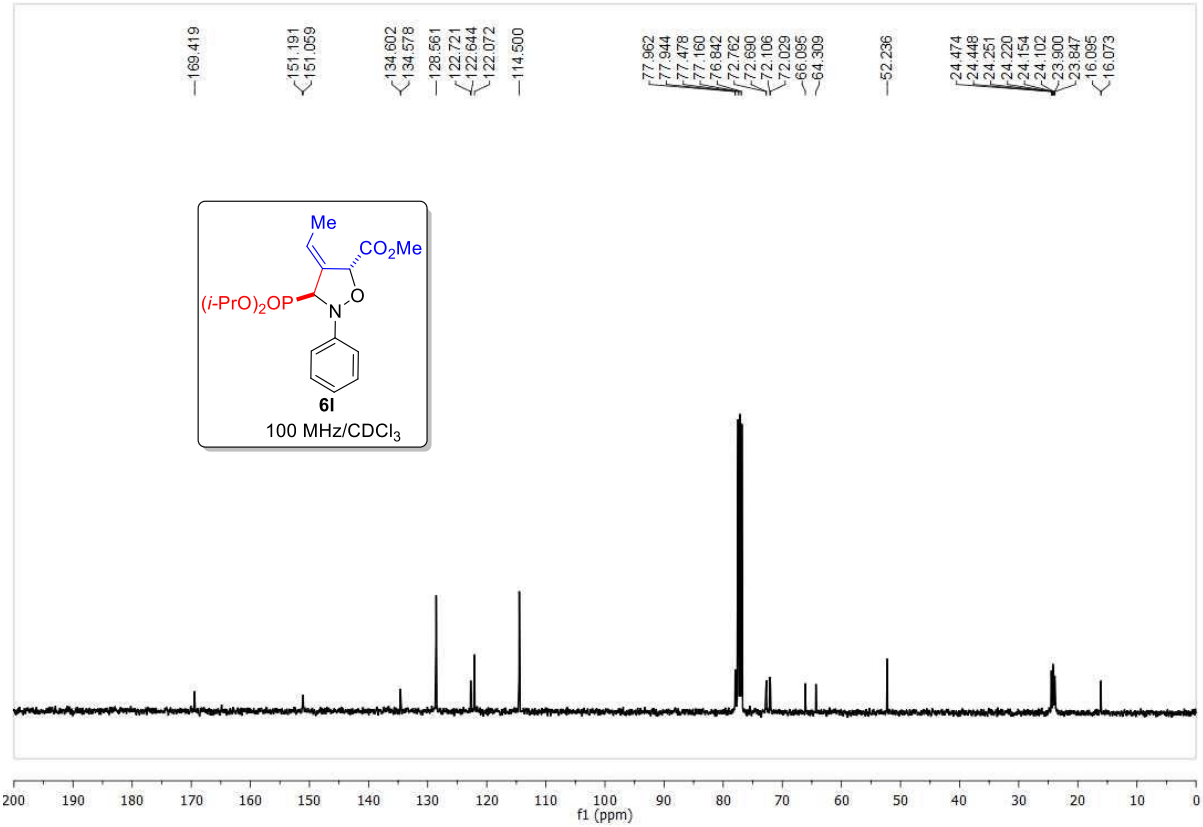

20.381
Me
CO2t-Bu
(MeO)2OP
N
O
6j
161.9 MHz/CDCl3
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

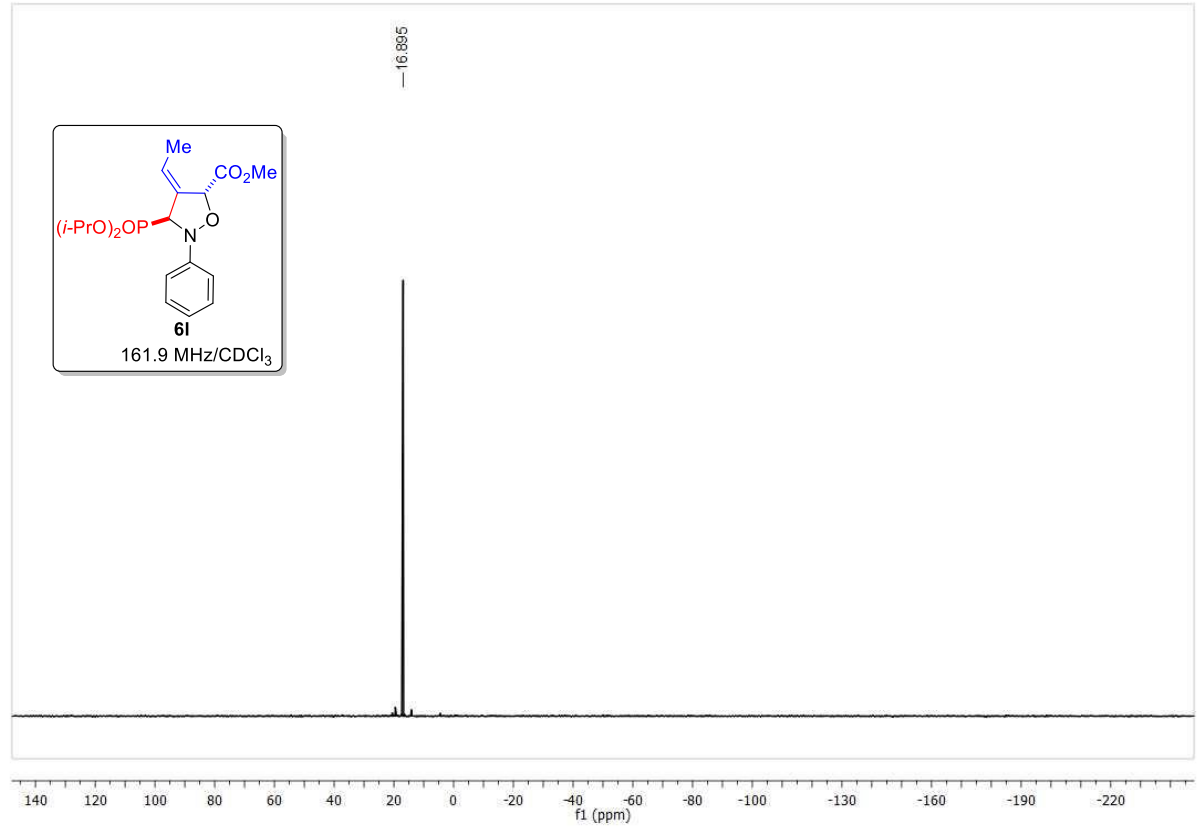

7.297
7.291
7.283
7.280
7.260
7.211
7.192
7.189
7.171
7.092
7.087
7.079
7.069
7.044
7.025
6.943
6.925
6.906
6.075
6.069
6.063
6.058
6.052
6.045
5.204
4.989
4.968
4.812
4.807
4.786
4.781
4.747
4.716
3.855
3.850
3.829
3.824
1.667
1.664
1.654
1.651
1.640
1.637
Me
CO2Bn
(MeO)2OP
N
O
6k
400 MHz/CDCl3
3.04
2.01
4.09
1.05
1.00
1.06
1.07
1.07
1.00
3.07
3.09
3.01
f1 (ppm)

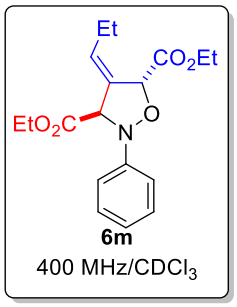

168.628
168.606
150.720
150.592
134.945
134.158
134.129
128.816
128.621
128.530
128.466
123.339
123.261
122.369
114.421
78.144
78.116
77.478
77.160
76.843
67.406
65.450
63.684
54.786
54.714
53.942
53.868
16.216
16.186
Me
CO2Bn
(MeO)2OP
N
O
6k
100 MHz/CDCl3
f1 (ppm)

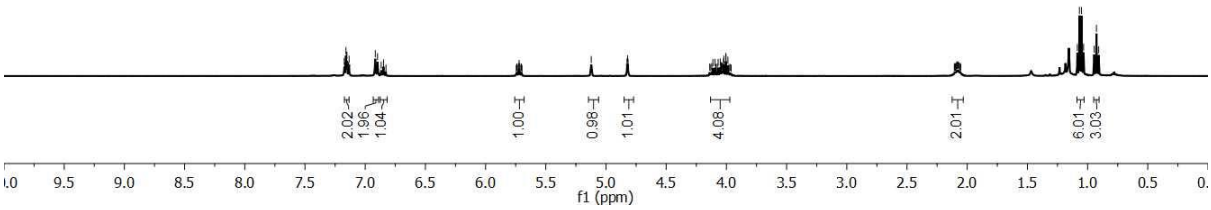

Me
CO2Bn
(MeO)2OP
N
O
6k
161.9 MHz/CDCl3
20.365
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

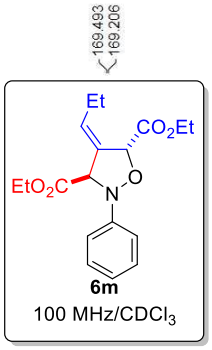

7.260 7.240 7.221 7.218 7.200 7.065 7.045 6.931 6.913 6.895 6.088 6.071 6.066 6.048
5.126 4.865 4.857 4.850 4.841 4.834 4.825 4.817 4.809 4.801 4.793 4.727 4.722 4.699 3.431
1.700 1.687 1.683 1.673 1.670 1.364 1.358 1.348 1.343 1.336 1.320 1.264 1.249
Me
CO2Me
(i-PrO)2OP
N
O
6l
400 MHz/CDCl3
2.09 2.04 1.02 1.00 1.00 2.05 1.05 3.08 3.08 9.07 3.05
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
f1 (ppm)


169.419
151.191
151.059
134.602
134.578
128.561
122.721
122.644
122.072
114.500
77.962
77.944
77.478
77.160
76.842
72.762
72.690
72.106
72.029
66.095
64.309
52.236
24.474
24.448
24.251
24.220
24.154
24.102
23.900
23.847
16.095
16.073
Me
CO2Me
(i-PrO)2OP
N
O
6l
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

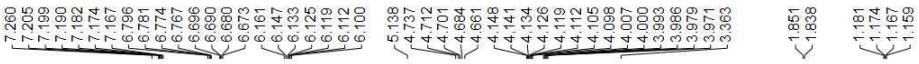

16.895
Me
CO2Me
(i-PrO)2OP
N
O
6l
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

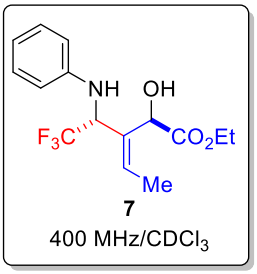

Et
CO2Et
EtO2C
N
O
6m
400 MHz/CDCl3

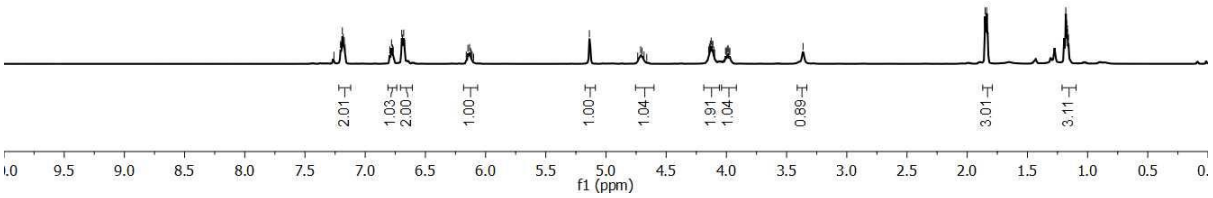

2.02
1.96
1.04
1.00
0.98
1.01
4.08
2.01
6.01
3.03
f1 (ppm)


Et
CO2Et
EtO2C
N
O
6m
100 MHz/CDCl3

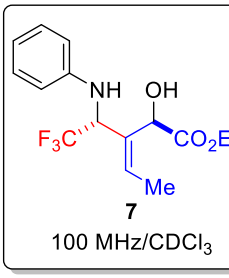
169.493
169.206
148.852
135.460
129.349
128.811
122.172
115.006
77.477
77.160
76.843
68.007
61.798
61.685
23.739
14.175
13.982
13.478
f1 (ppm)

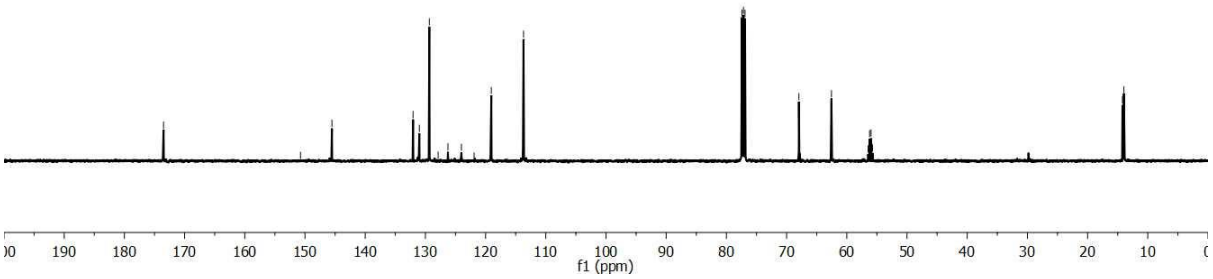

7.260
7.205
7.199
7.190
7.182
7.174
7.167
6.796
6.781
6.774
6.767
6.696
6.690
6.680
6.673
6.161
6.147
6.133
6.125
6.119
6.112
6.100
5.138
4.737
4.712
4.701
4.684
4.661
4.148
4.141
4.134
4.126
4.119
4.112
4.105
4.098
4.007
4.000
3.993
3.986
3.979
3.971
3.363
1.851
1.838
1.181
1.174
1.167
1.159

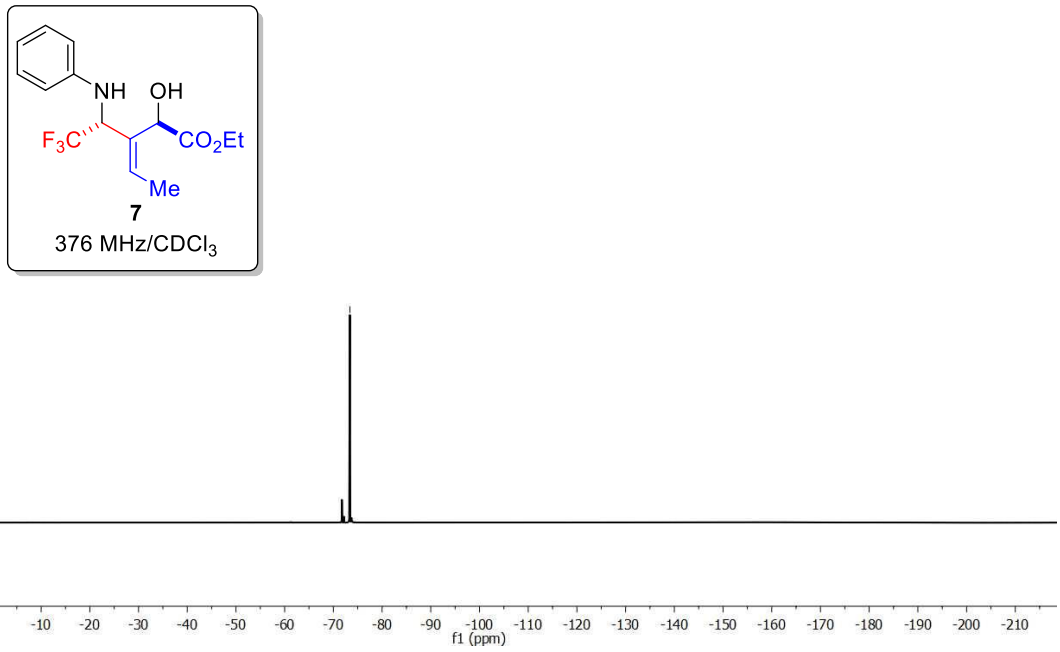

NH
OH
F3C
CO2Et
Me
7
400 MHz/CDCl3

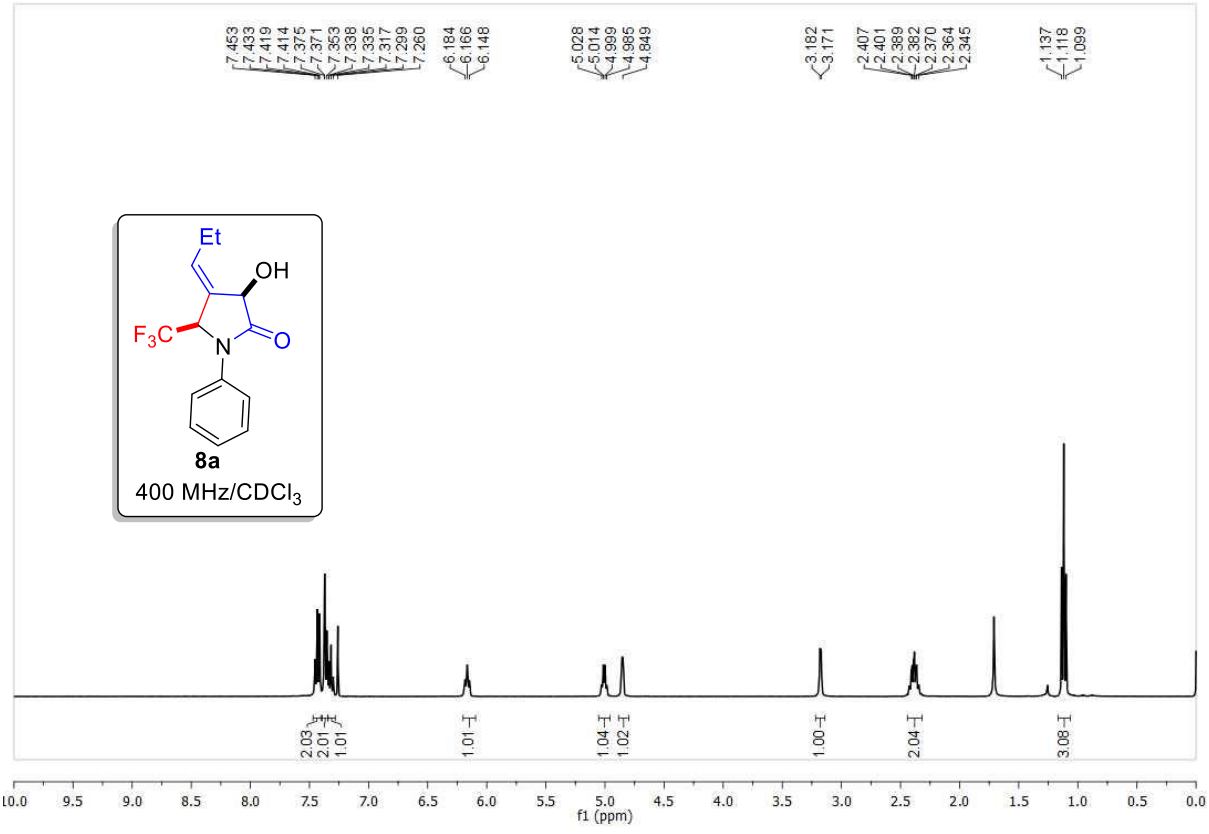

2.01
1.03
2.00
1.00
1.00
1.04
1.91
1.04
0.89
3.01
3.11
f1 (ppm)

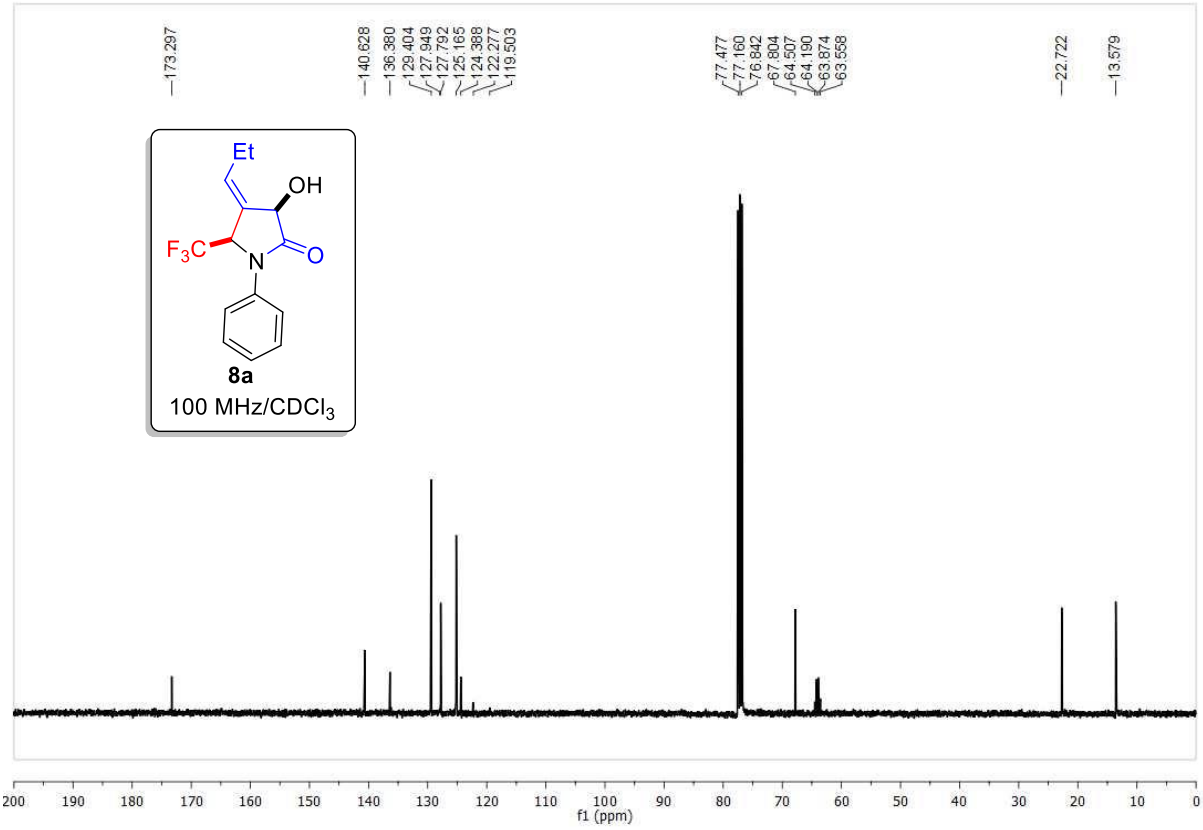

173.497
150.747
145.525
132.049
131.006
129.401
129.350
127.885
126.259
124.018
121.911
119.052
113.701
77.414
77.160
76.906
67.958
62.549
56.455
56.213
55.972
55.729
14.211
13.968

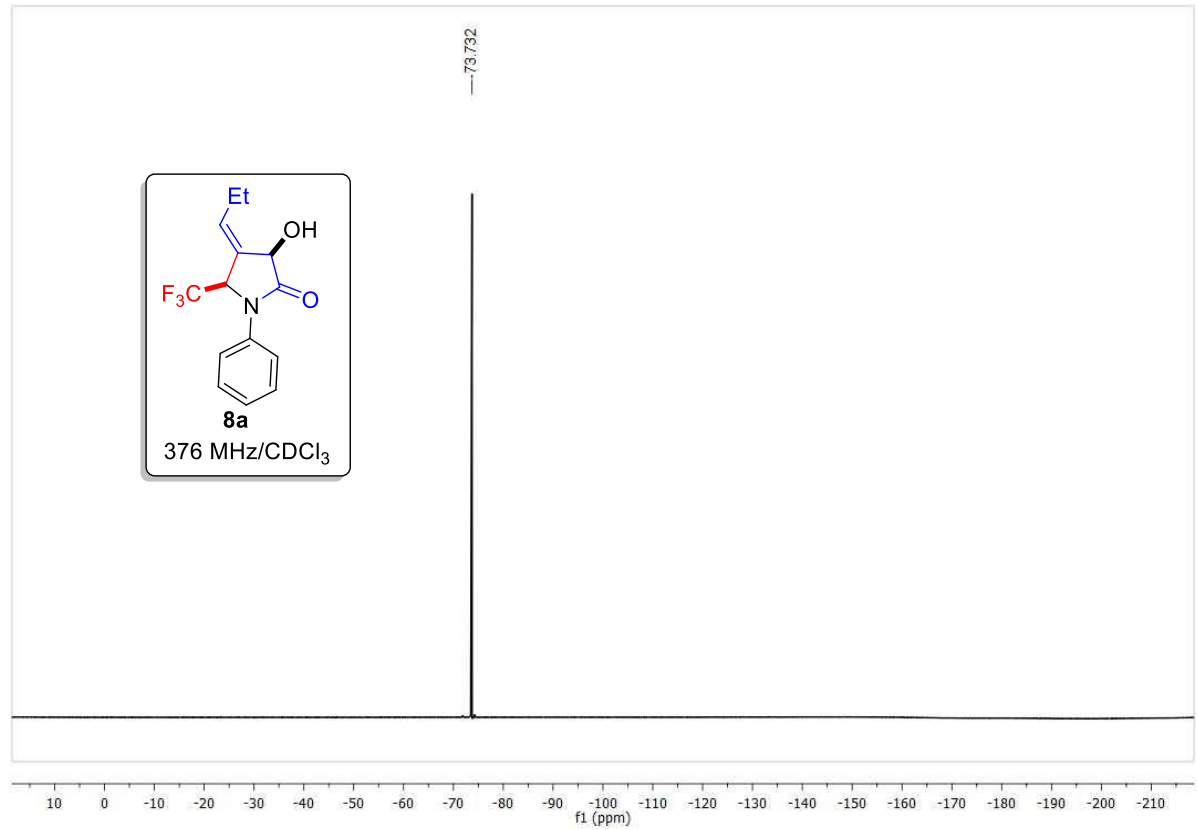

NH
OH
F3C
CO2Et
Me
7
100 MHz/CDCl3

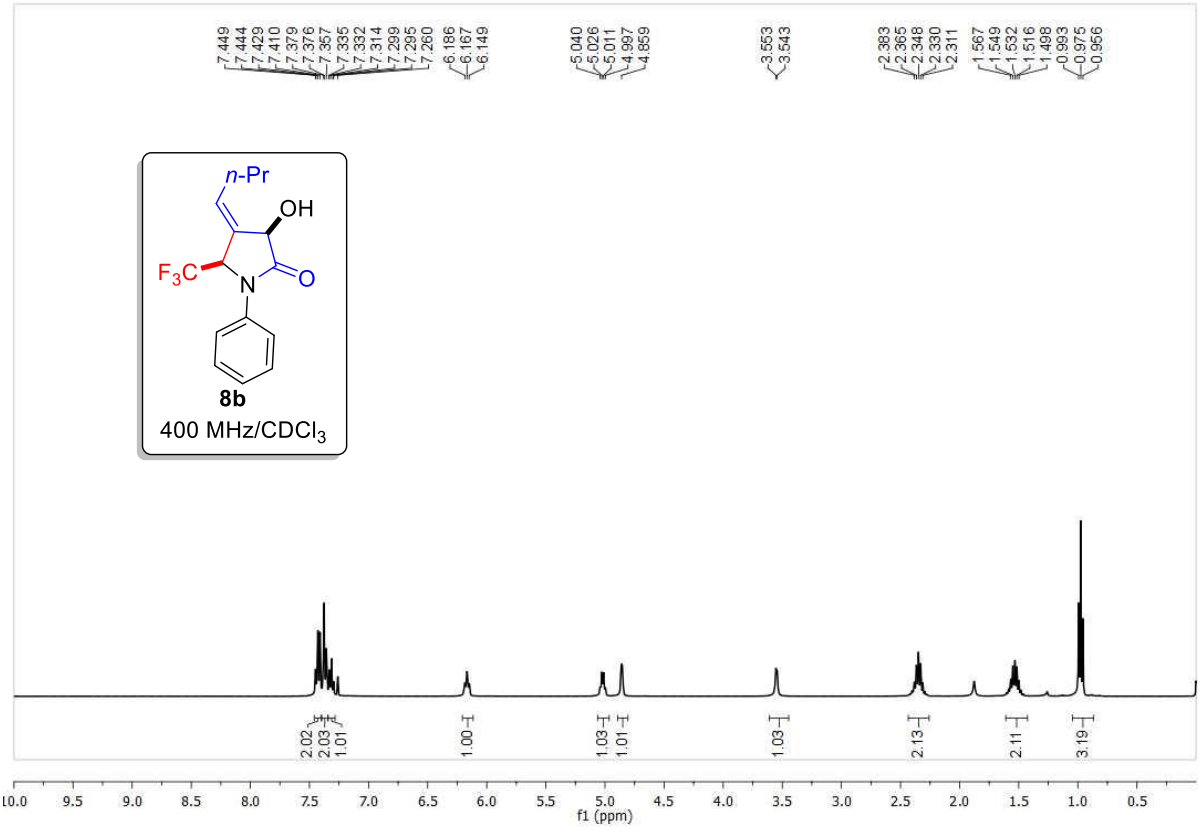

f1 (ppm)

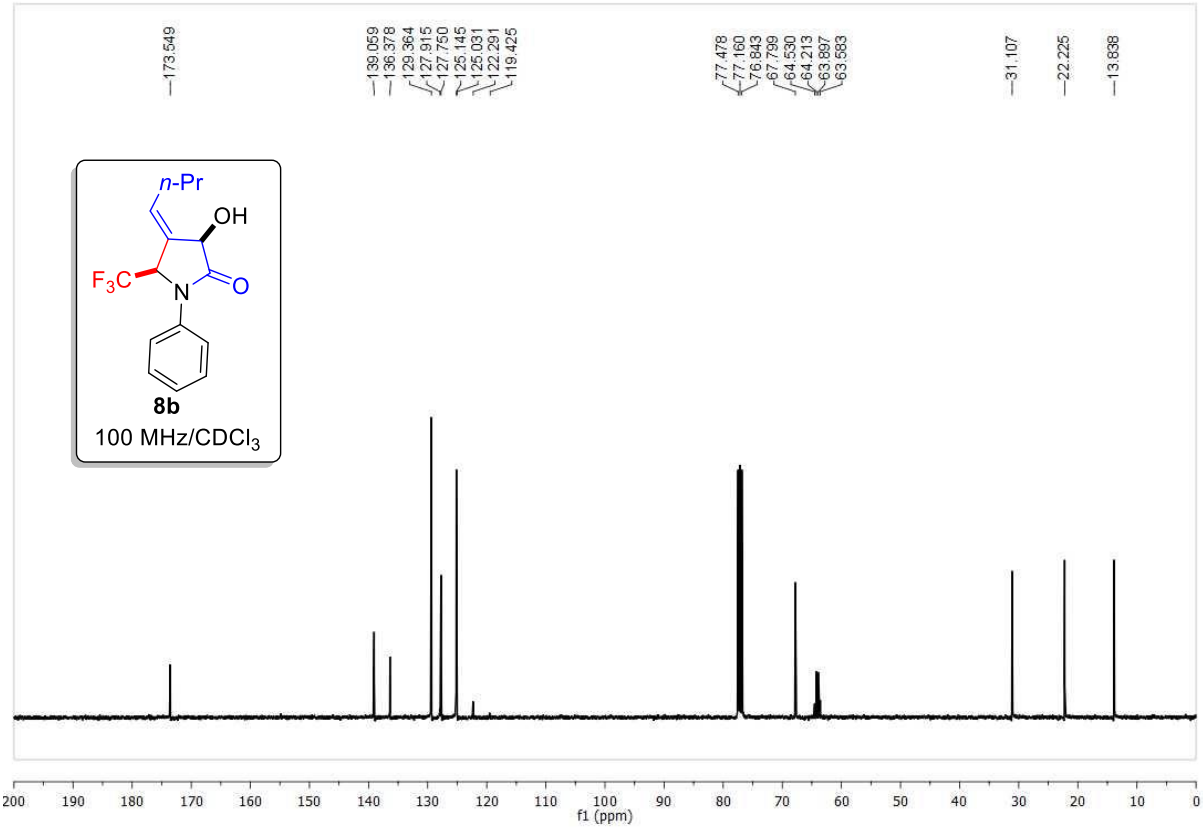

-73.388
NH
OH
F3C
CO2Et
Me
7
376 MHz/CDCl3
f1 (ppm)

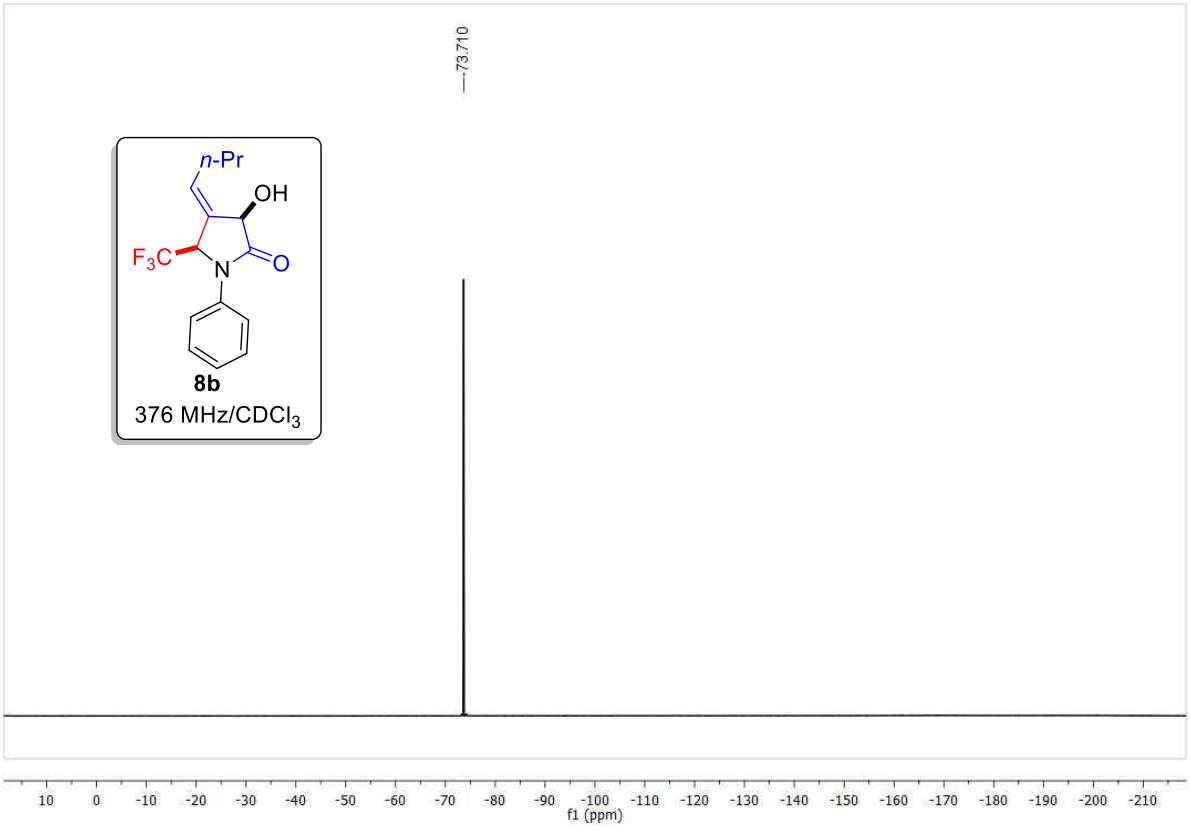

7.453
7.433
7.419
7.414
7.375
7.371
7.353
7.338
7.335
7.317
7.299
7.260
6.184
6.166
6.148
5.028
5.014
4.999
4.985
4.849
3.182
3.171
2.407
2.401
2.389
2.382
2.370
2.364
2.345
1.137
1.118
1.099
Et
OH
F3C
N
O
8a
400 MHz/CDCl3
2.03
2.01
1.01
1.01
1.04
1.02
1.00
2.04
3.08
f1 (ppm)

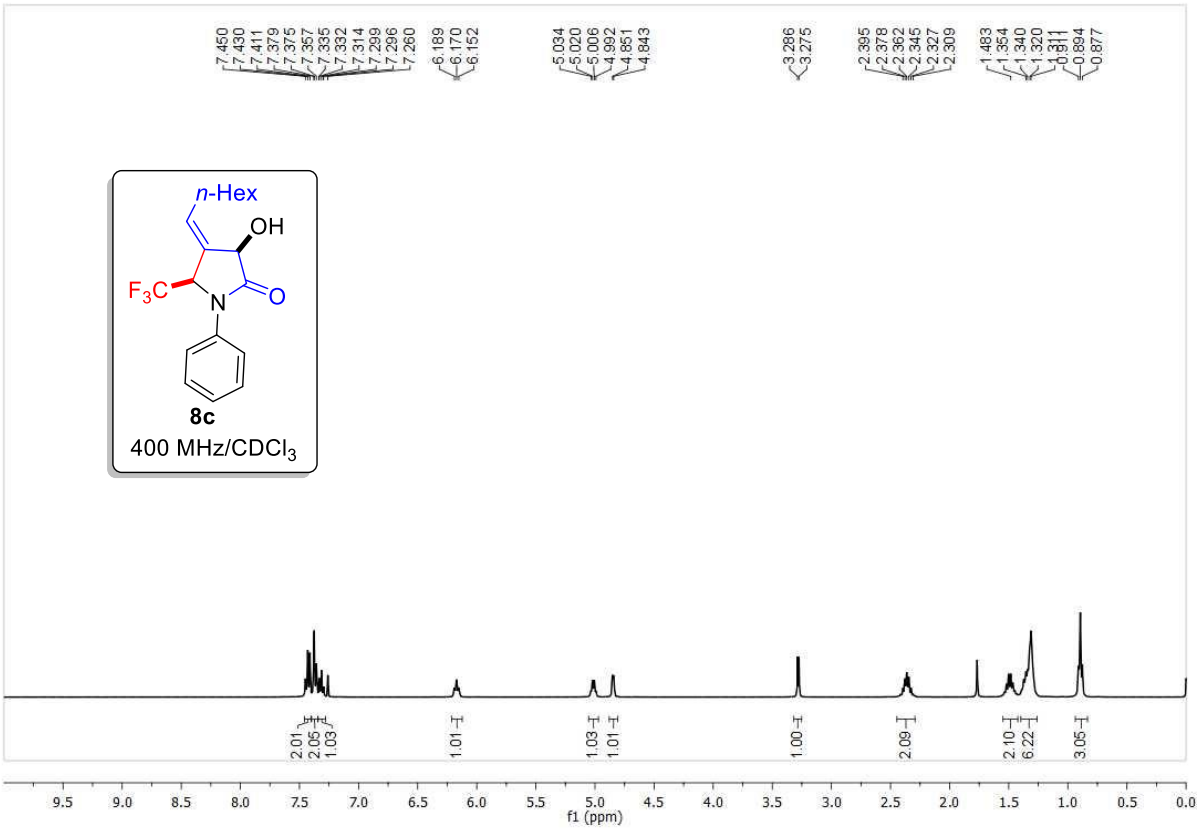

173.297
140.628
136.380
129.404
127.949
127.792
125.165
124.388
122.277
119.503
77.477
77.160
76.842
67.804
64.507
64.190
63.874
63.558
22.722
13.579
Et
OH
F3C
N
O
8a
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

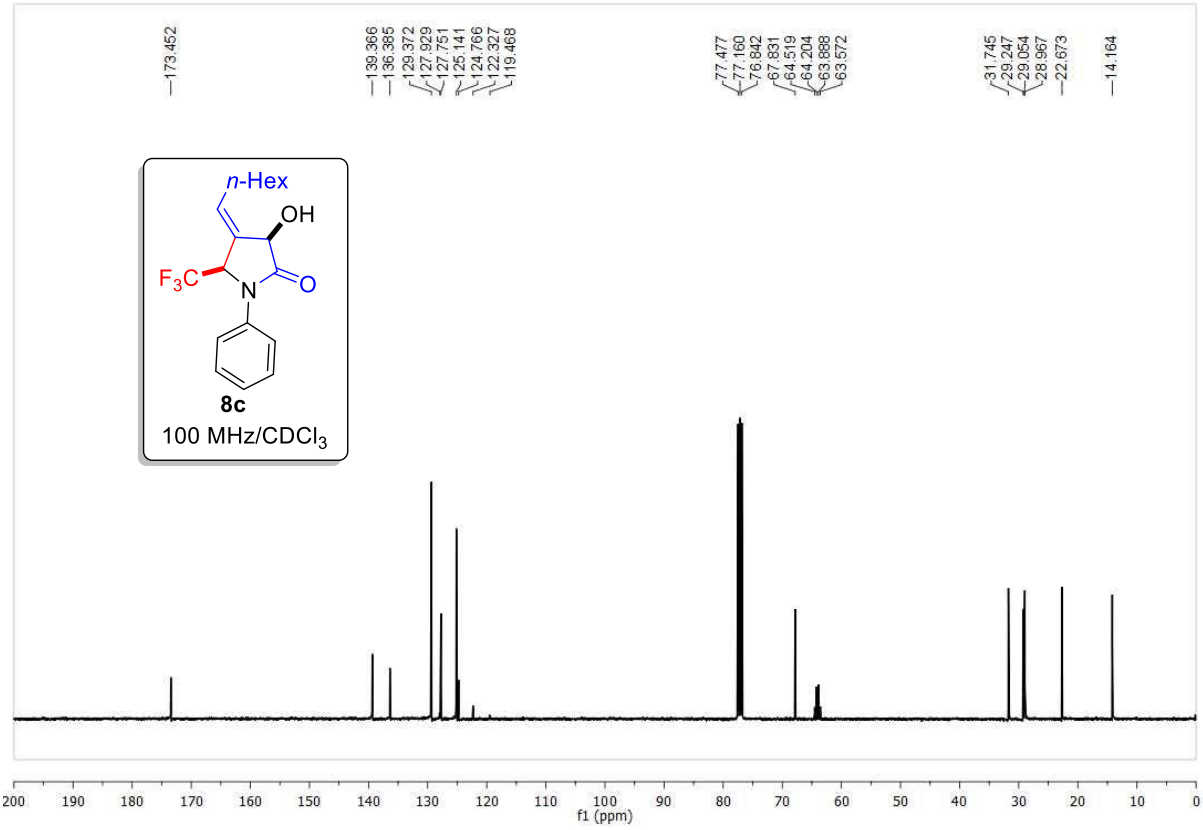

73.732
Et
OH
F3C
N
O
8a
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

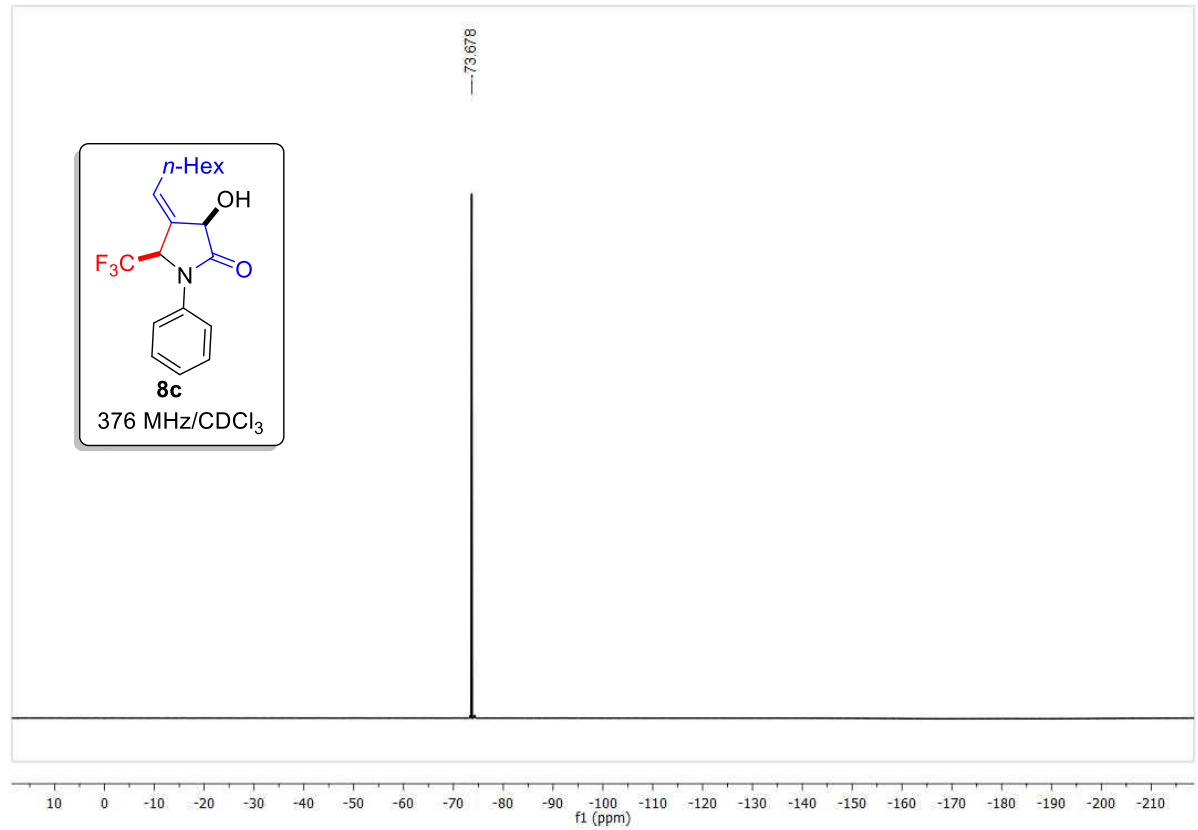

7.449
7.444
7.429
7.410
7.379
7.376
7.357
7.335
7.332
7.314
7.299
7.295
7.260
6.186
6.167
6.149
5.040
5.026
5.011
4.997
4.859
3.553
3.543
2.383
2.365
2.348
2.330
2.311
1.567
1.549
1.532
1.516
1.498
0.993
0.975
0.956
n-Pr
OH
F3C
N
O
8b
400 MHz/CDCl3
2.02
2.03
1.01
1.00
1.03
1.01
1.03
2.13
2.11
3.19
f1 (ppm)

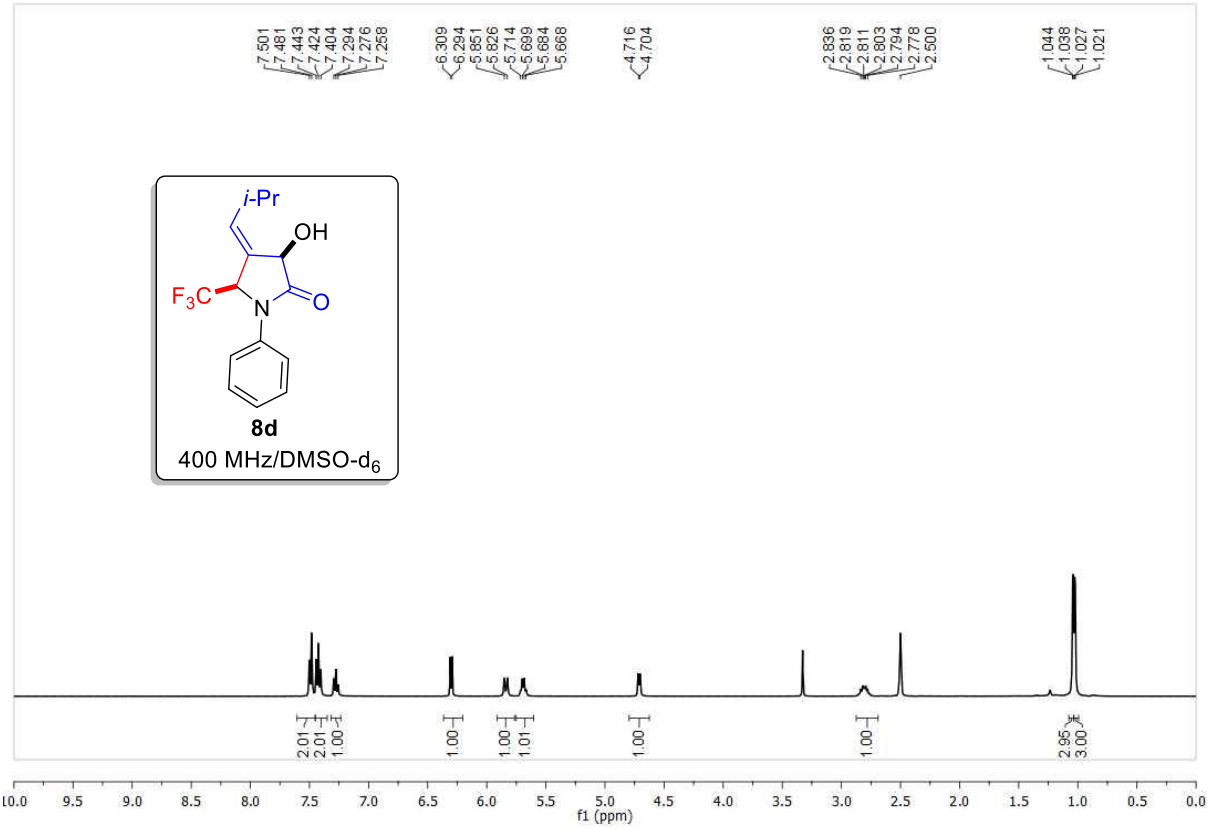

173.549
139.059
136.378
129.364
127.915
127.750
125.145
125.031
122.291
119.425
77.478
77.160
76.843
67.799
64.530
64.213
63.897
63.583
31.107
22.225
13.838
n-Pr
OH
F3C
N
O
8b
100 MHz/CDCl3
f1 (ppm)

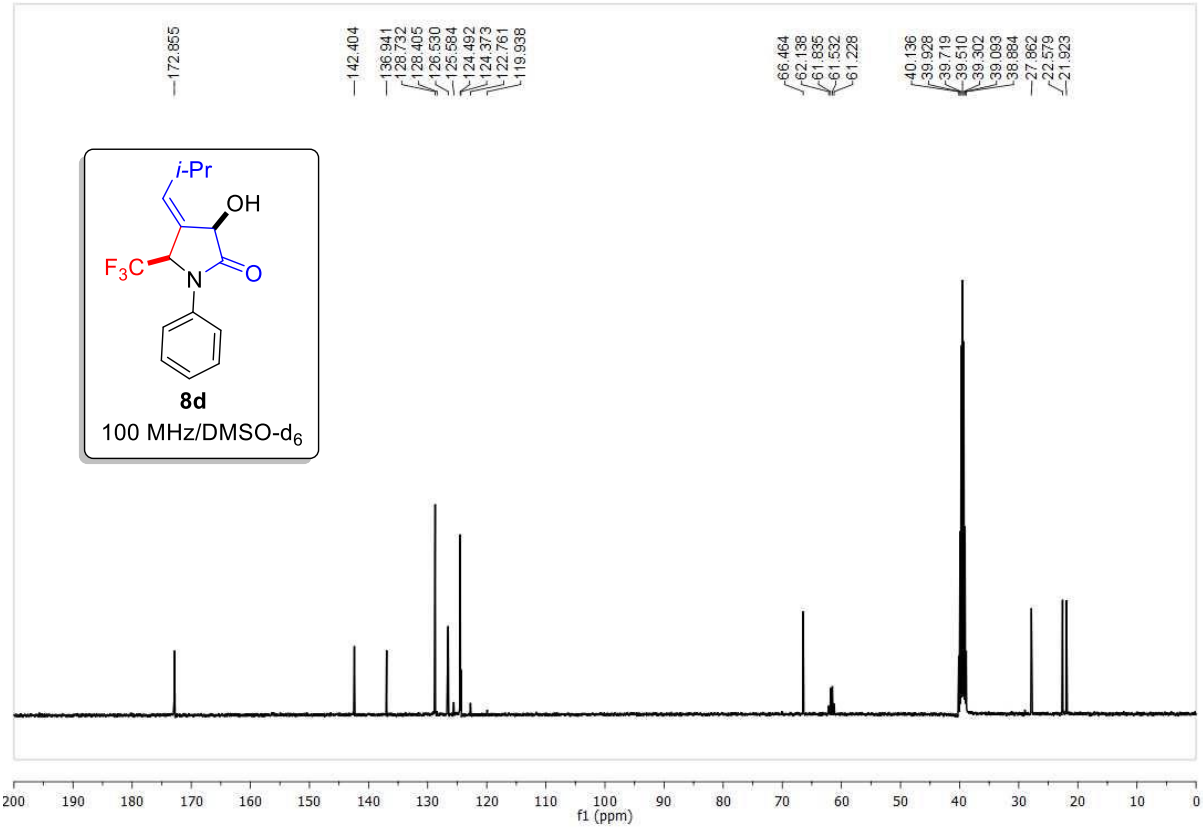

-73.710
n-Pr
OH
F3C
N
O
8b
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

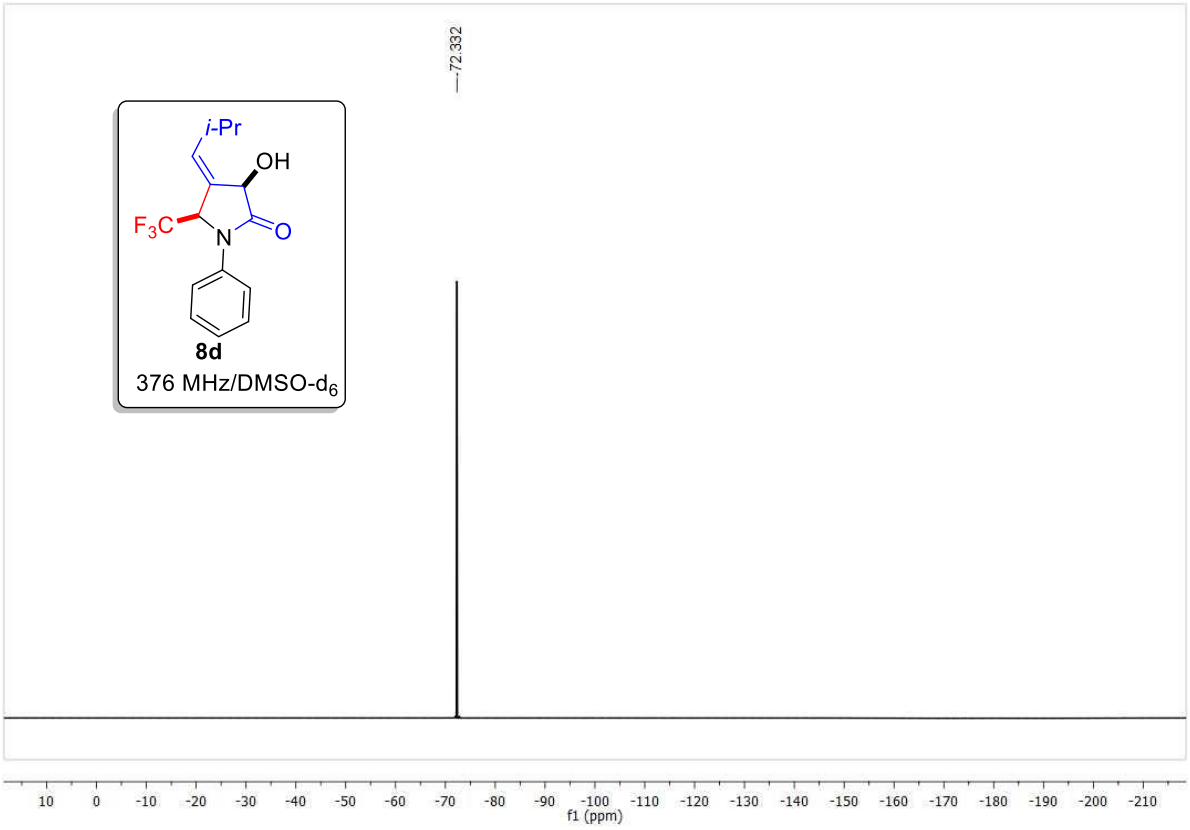

7.450 7.430 7.411 7.379 7.375 7.357 7.335 7.332 7.314 7.299 7.296 7.260
6.189 6.170 6.152
5.034 5.020 5.006 4.992 4.851 4.843
3.286 3.275
2.395 2.378 2.362 2.345 2.327 2.309
1.483 1.354 1.340 1.320 1.311 0.911 0.894 0.877
n-Hex
OH
F3C
N
O
8c
400 MHz/CDCl3
2.01 2.05 1.03 1.01 1.03 1.01 1.00 2.09 2.10 6.22 3.05
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
f1 (ppm)

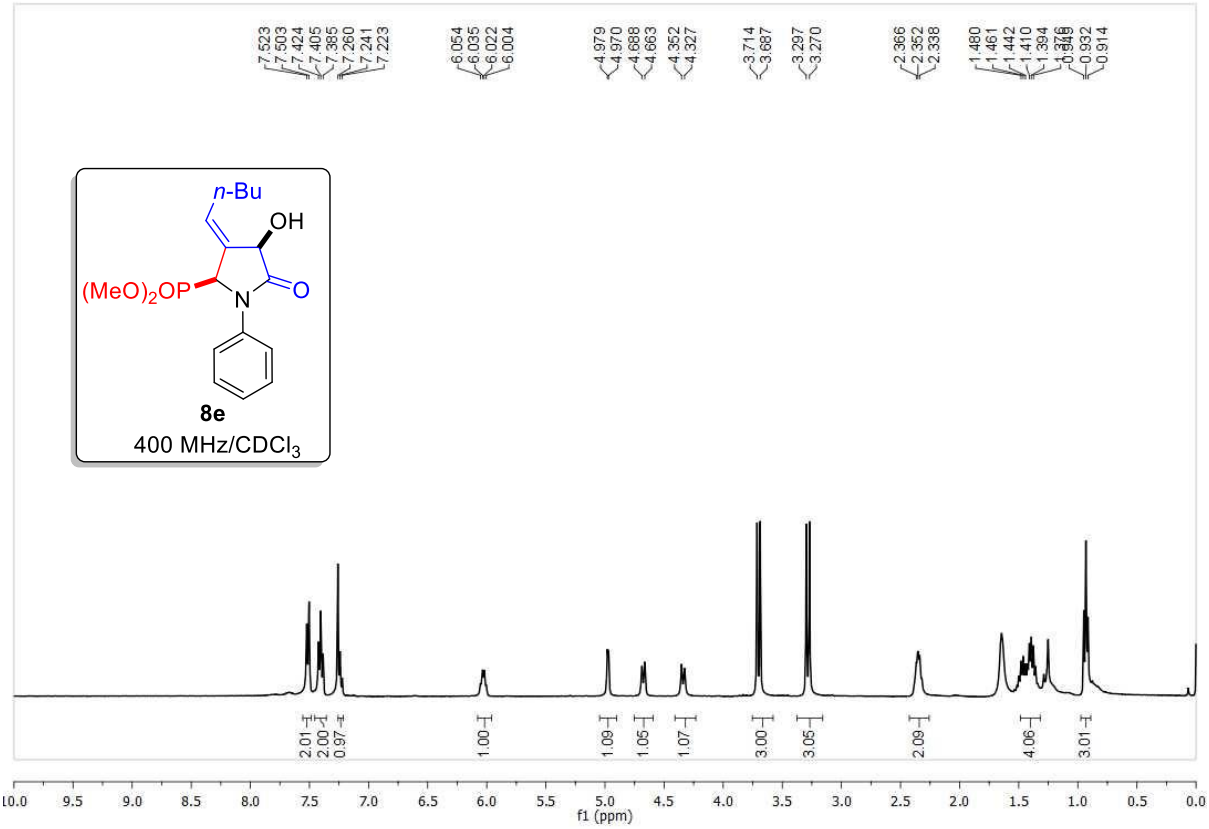

173.452
139.366
136.385
129.372
127.929
127.751
125.141
124.766
122.327
119.468
77.477
77.160
76.842
67.831
64.519
64.204
63.888
63.572
31.745
29.247
29.054
28.967
22.673
14.164
n-Hex
OH
F3C
N
O
8c
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

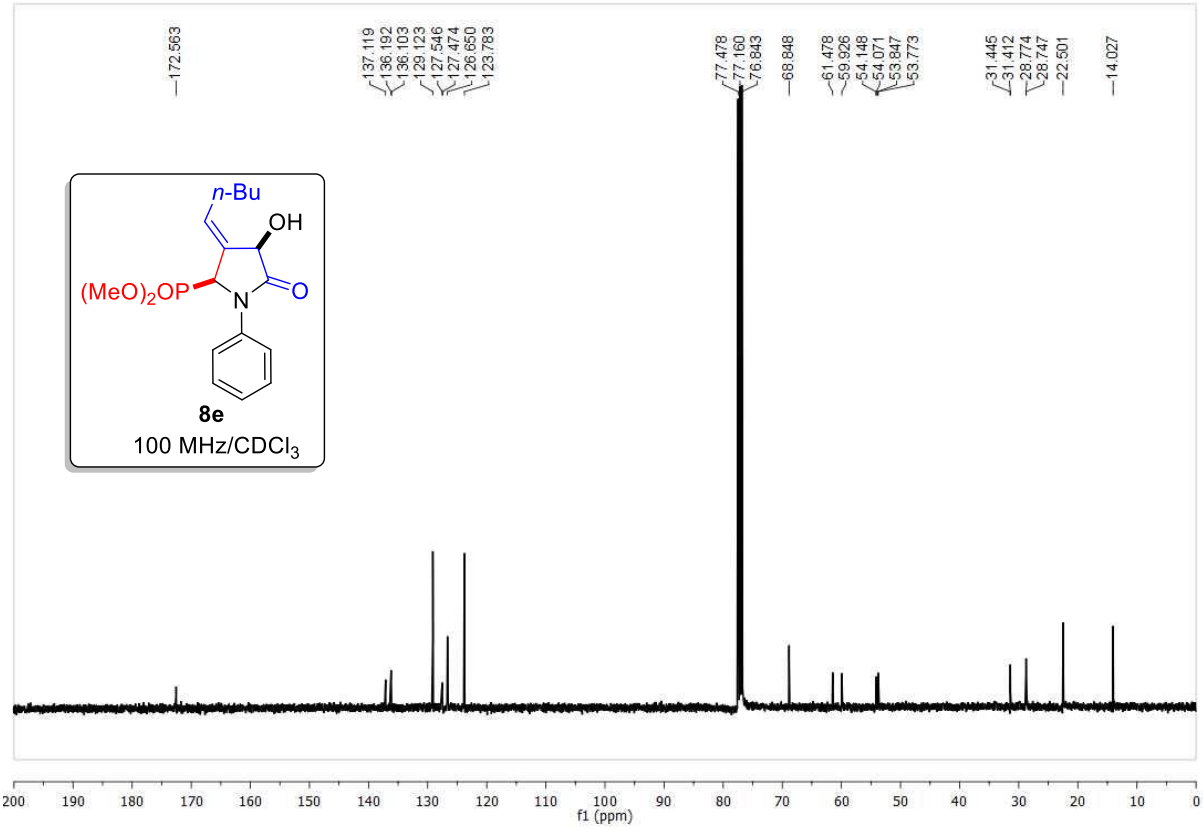

73.678
n-Hex
OH
F3C
N
O
8c
376 MHz/CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

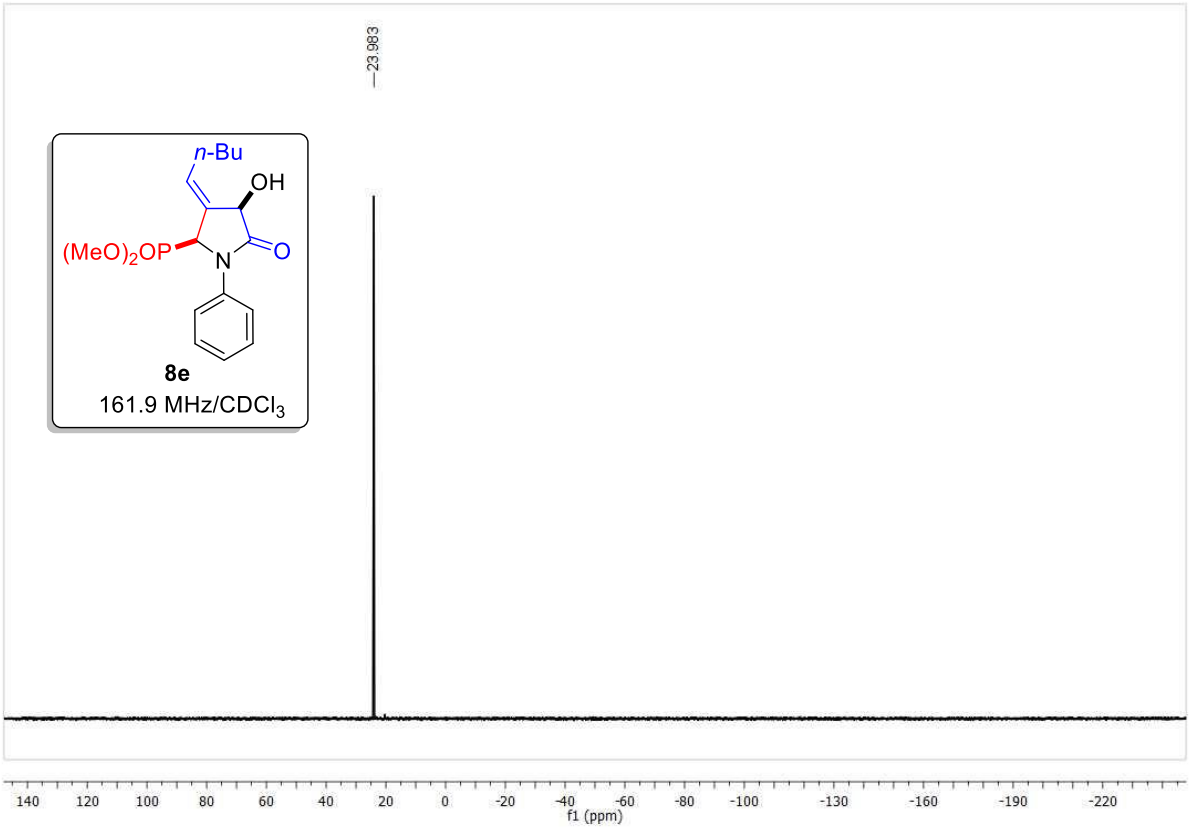

7.501
7.481
7.443
7.424
7.404
7.294
7.276
7.258
6.309
6.294
5.851
5.826
5.714
5.699
5.684
5.668
4.716
4.704
2.836
2.819
2.811
2.803
2.794
2.778
2.500
1.044
1.038
1.027
1.021
i-Pr
OH
F3C
N
O
8d
400 MHz/DMSO-d6
2.01
2.01
1.00
1.00
1.00
1.01
1.00
1.00
2.95
3.00
f1 (ppm)

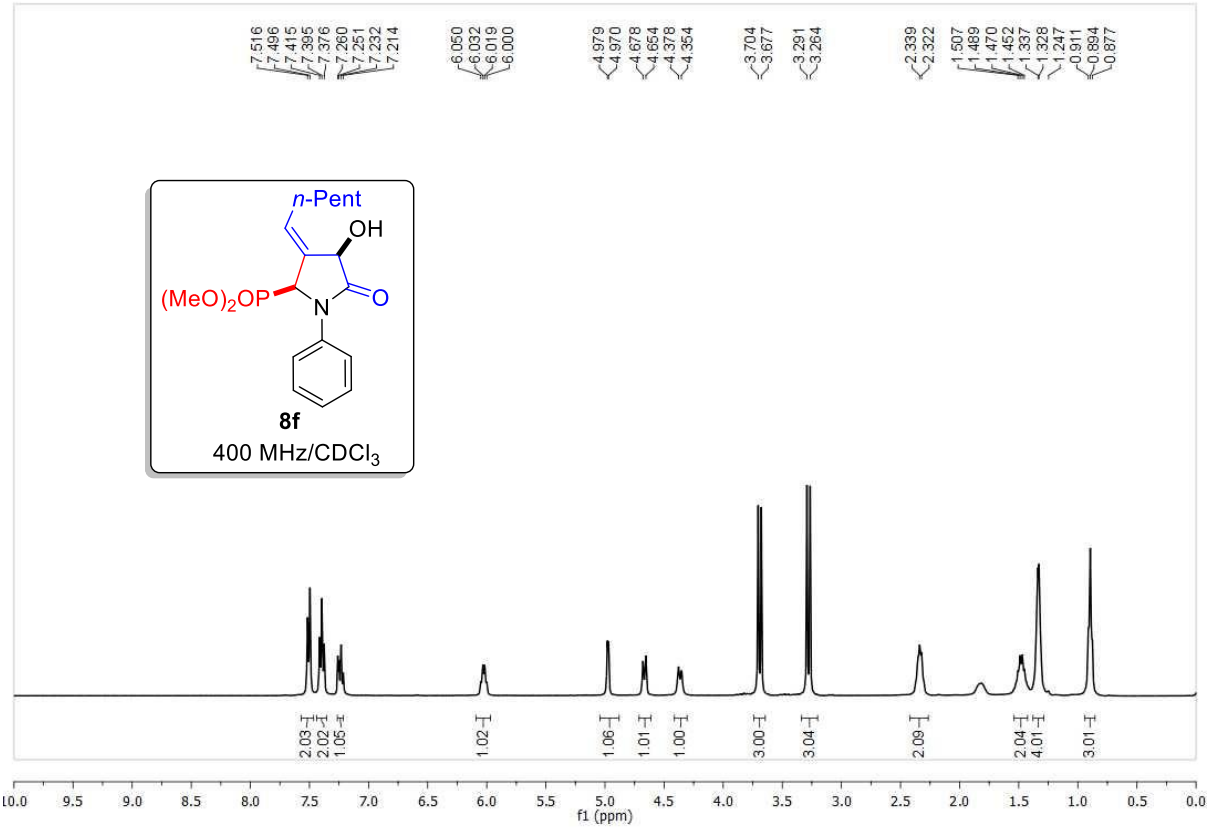

172.855
142.404
136.941
128.732
128.405
126.530
125.584
124.492
124.373
122.761
119.938
66.464
62.138
61.835
61.532
61.228
40.136
39.928
39.719
39.510
39.302
39.093
38.884
27.862
22.579
21.923
i-Pr
OH
F3C
N
O
8d
100 MHz/DMSO-d6
f1 (ppm)

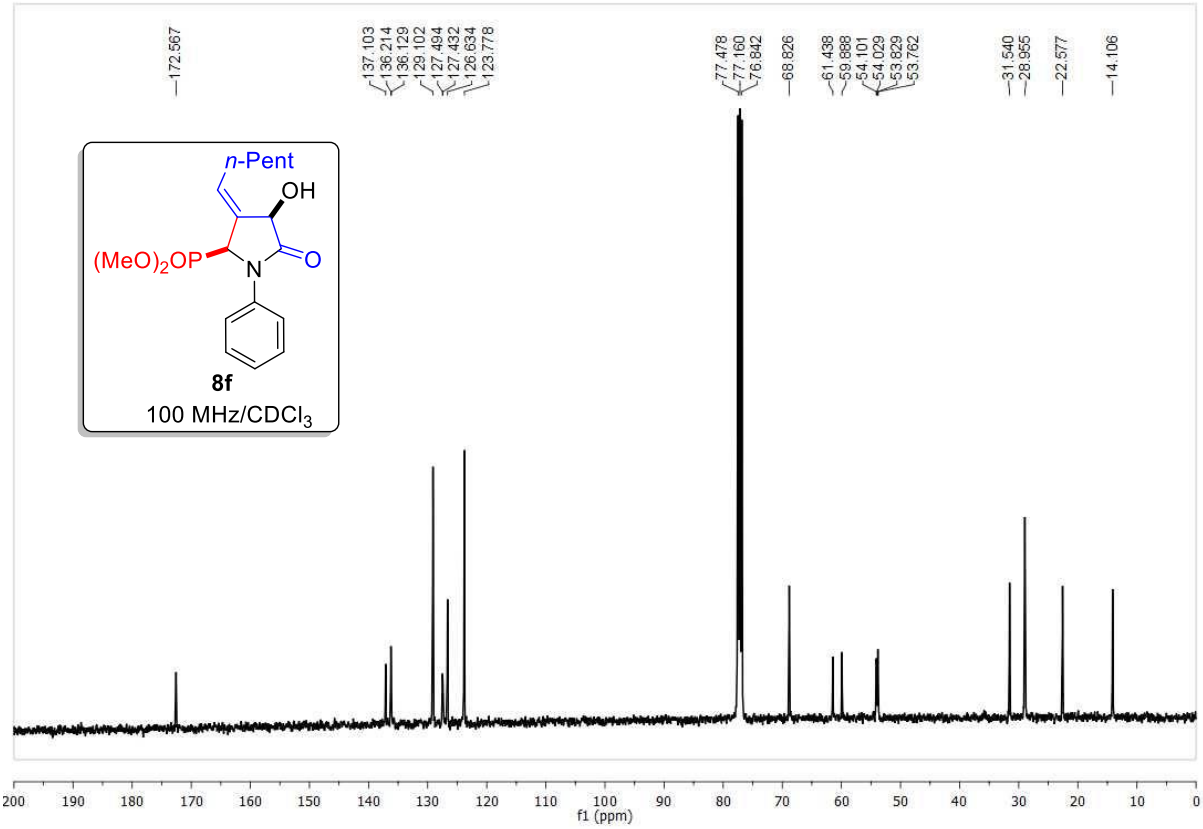

-72.332
i-Pr
OH
F3C
N
O
8d
376 MHz/DMSO-d6
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

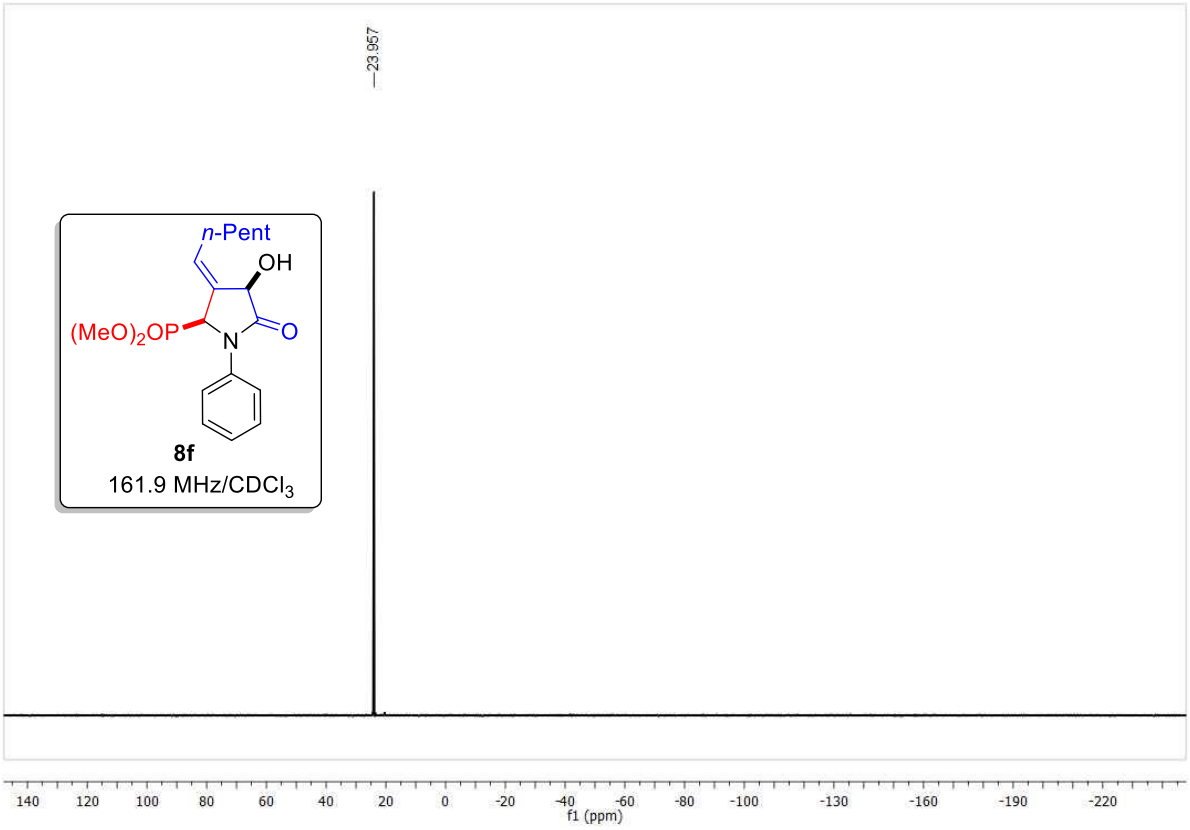

7.523
7.503
7.424
7.405
7.385
7.260
7.241
7.223
6.054
6.035
6.022
6.004
4.979
4.970
4.688
4.663
4.352
4.327
3.714
3.687
3.297
3.270
2.366
2.352
2.338
1.480
1.461
1.442
1.410
1.394
0.932
0.914
n-Bu
OH
(MeO)2OP
N
O
8e
400 MHz/CDCl3
2.01
2.00
0.97
1.00
1.09
1.05
1.07
3.00
3.05
2.09
4.06
3.01
10.0 9.5 9.0 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
f1 (ppm)

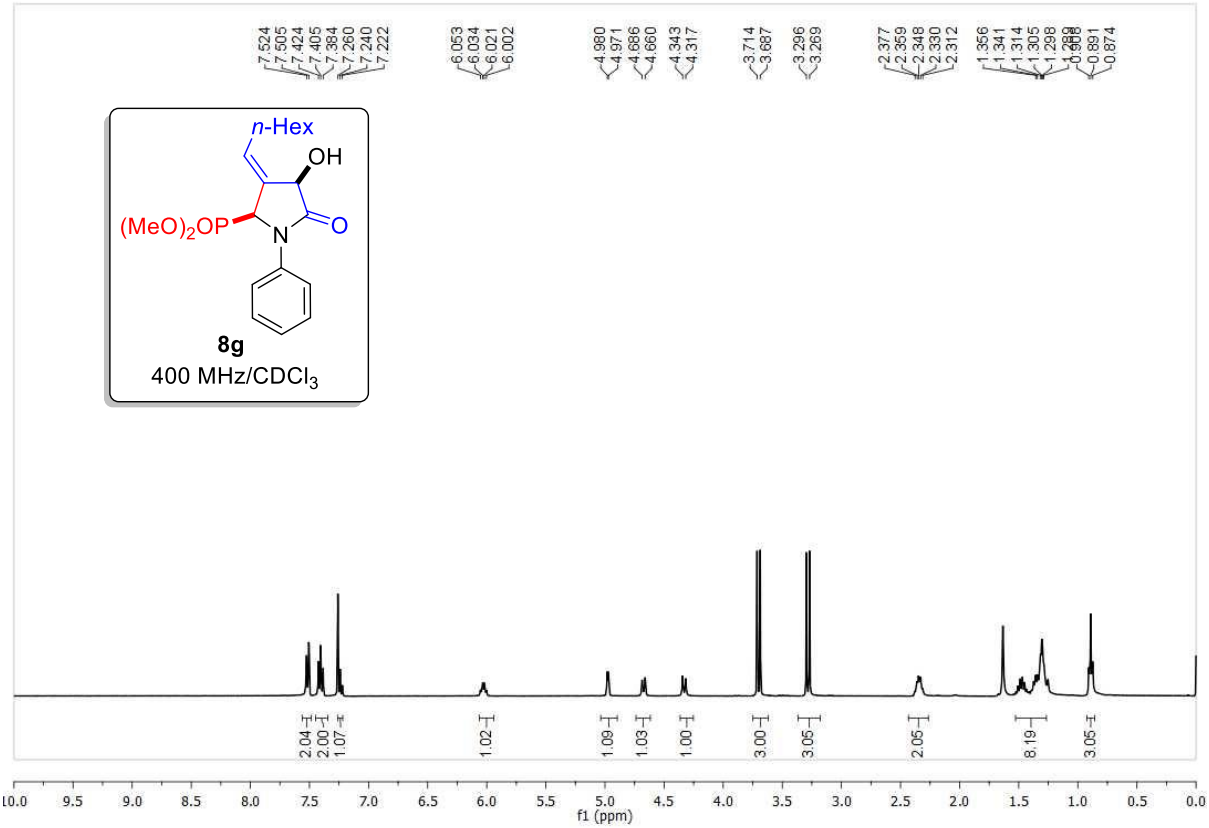

172.563
137.119
136.192
136.103
129.123
127.546
127.474
126.650
123.783
77.478
77.160
76.843
68.848
61.478
59.926
54.148
54.071
53.847
53.773
31.445
31.412
28.774
28.747
22.501
14.027
n-Bu
OH
(MeO)2OP
N
O
8e
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

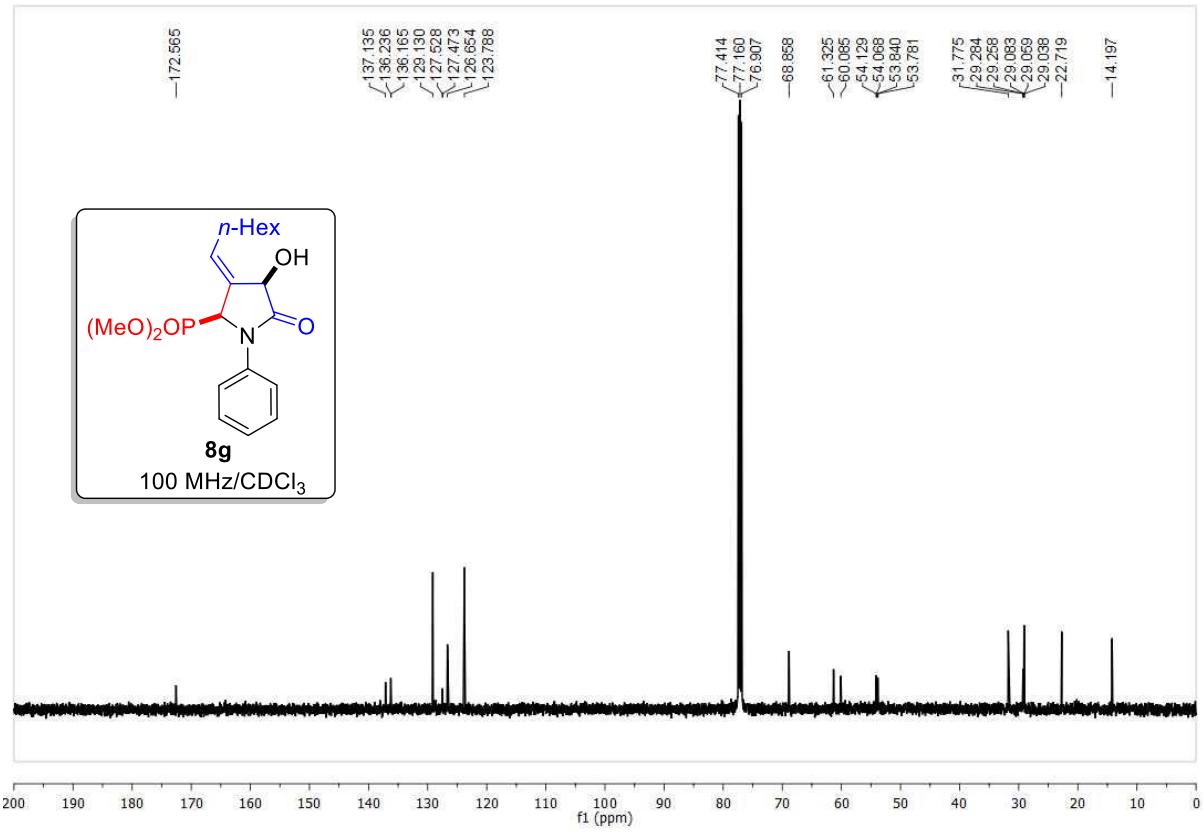

23.983
n-Bu
OH
(MeO)2OP
N
O
8e
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

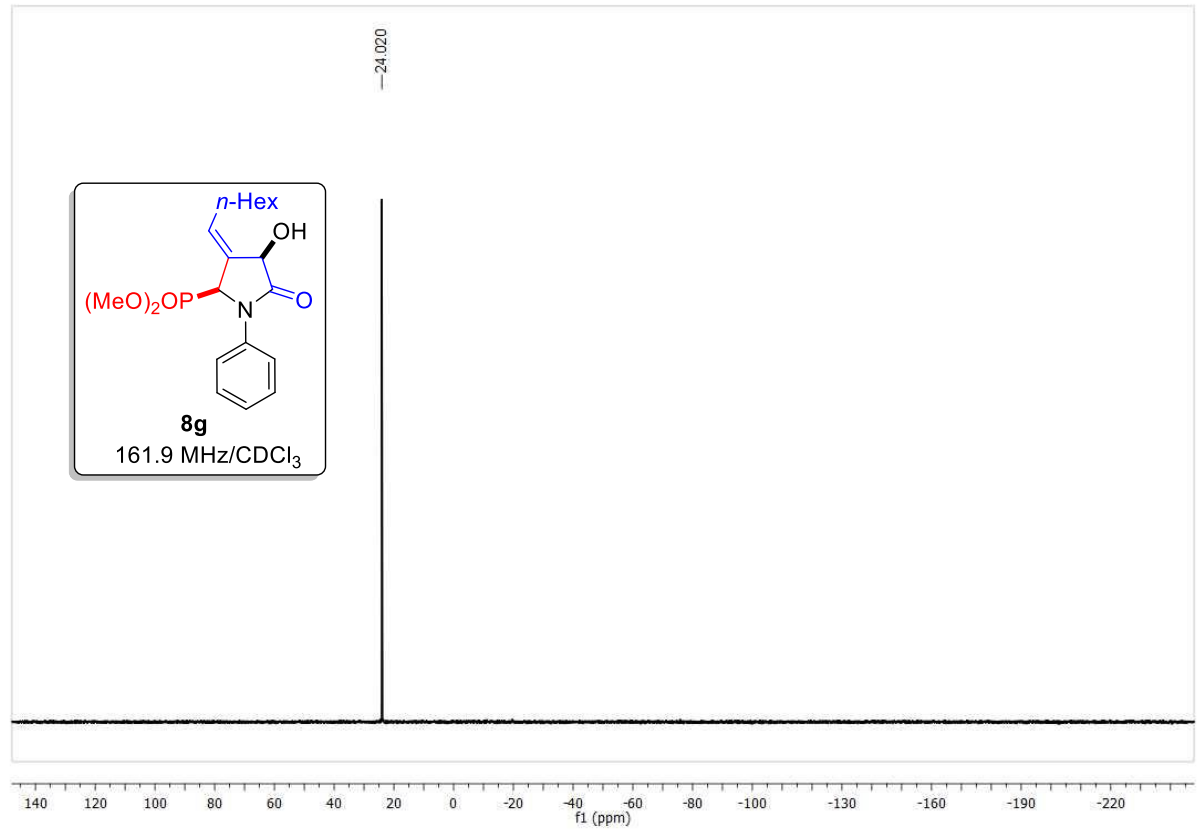

n-Pent
OH
(MeO)2OP
N
O
8f
400 MHz/CDCl3
f1 (ppm)

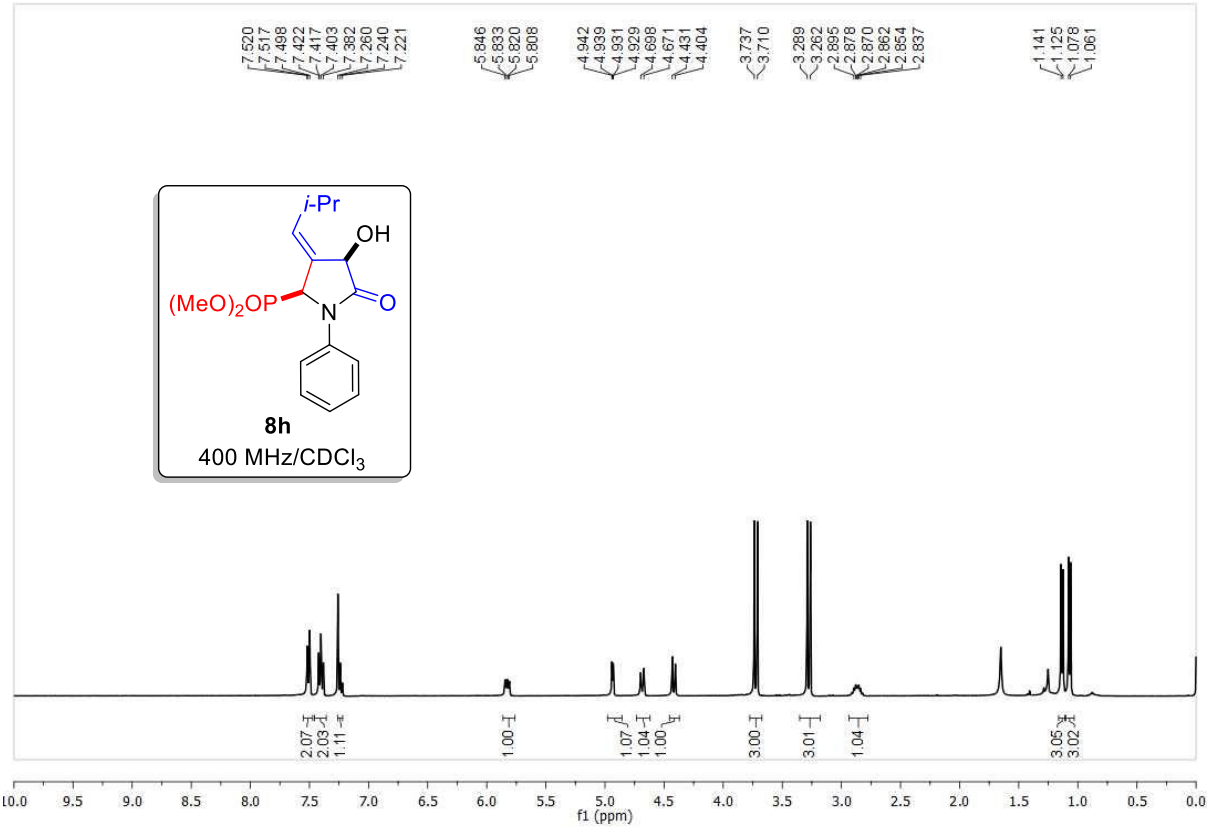

n-Pent
OH
(MeO)2OP
N
O
8f
100 MHz/CDCl3
f1 (ppm)

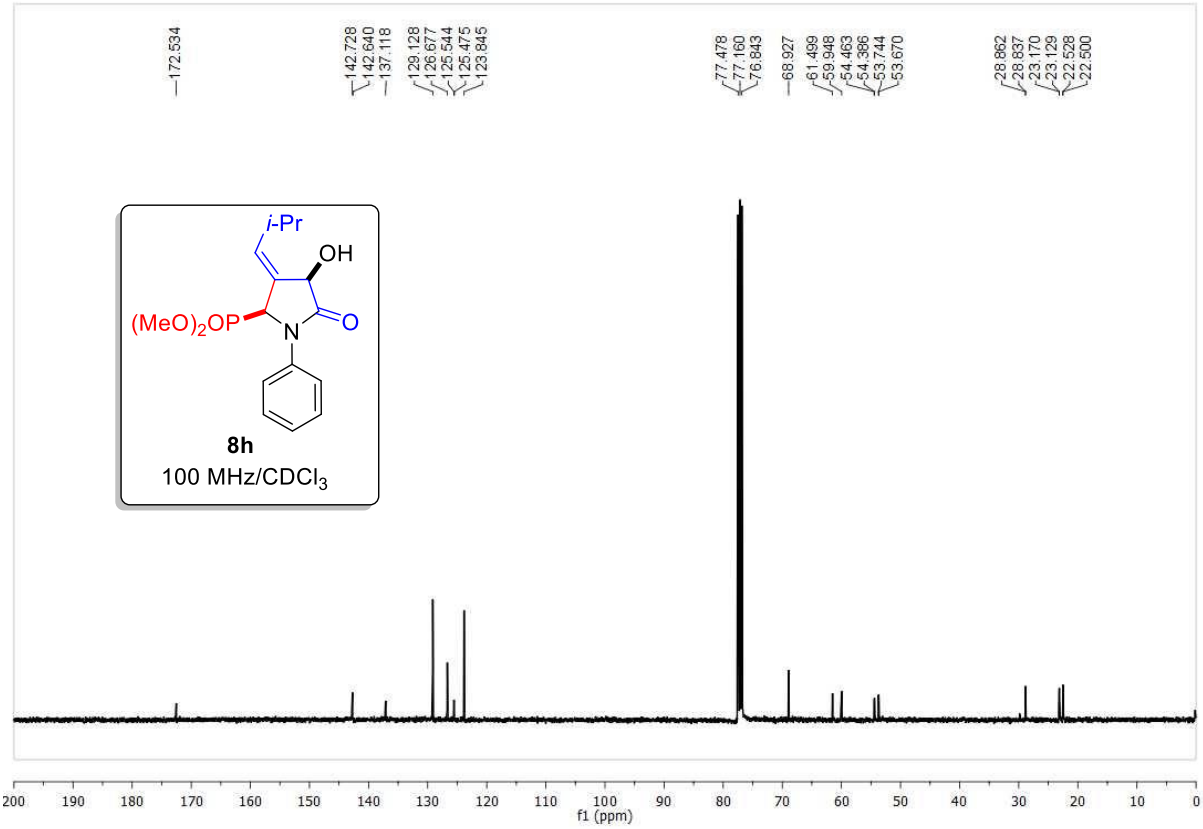

23.957
n-Pent
OH
(MeO)2OP
N
O
8f
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

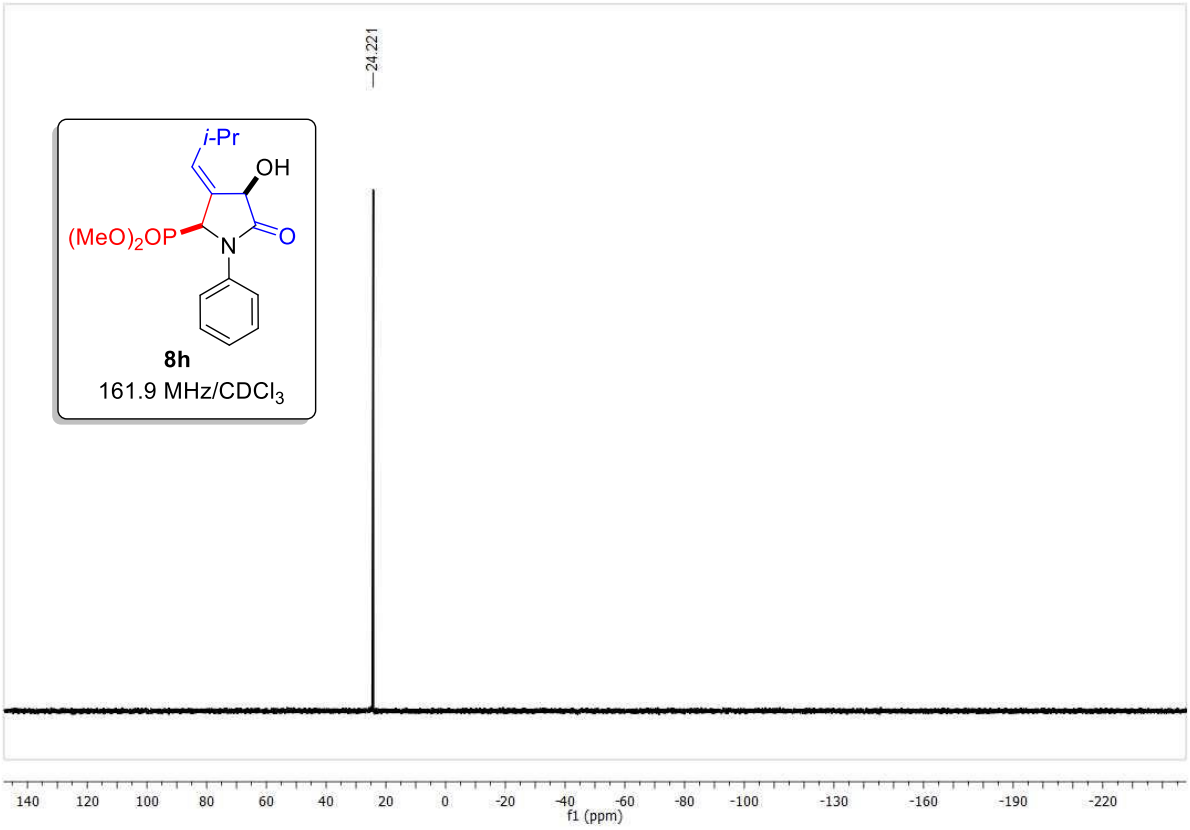

7.524 7.505 7.424 7.405 7.384 7.260 7.240 7.222
6.053 6.034 6.021 6.002
4.980 4.971 4.686 4.660 4.343 4.317
3.714 3.687 3.296 3.269
2.377 2.359 2.348 2.330 2.312
1.356 1.341 1.314 1.305 1.298
0.891 0.874
n-Hex
OH
(MeO)2OP
N
O
8g
400 MHz/CDCl3
2.04 2.00 1.07 1.02 1.09 1.03 1.00 3.00 3.05 2.05 8.19 3.05
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
f1 (ppm)


172.565
137.135
136.236
136.165
129.130
127.528
127.473
126.654
123.788
77.414
77.160
76.907
68.858
61.325
60.085
54.129
54.068
53.840
53.781
31.775
29.284
29.258
29.083
29.059
29.038
22.719
14.197
n-Hex
OH
(MeO)2OP
N
O
8g
100 MHz/CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


24.020
n-Hex
OH
(MeO)2OP
N
O
8g
161.9 MHz/CDCl3
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)


7.520
7.517
7.498
7.422
7.417
7.403
7.382
7.260
7.240
7.221
5.846
5.833
5.820
5.808
4.942
4.939
4.931
4.929
4.698
4.671
4.431
4.404
3.737
3.710
3.289
3.262
2.895
2.878
2.870
2.862
2.854
2.837
1.141
1.125
1.078
1.061
i-Pr
OH
(MeO)2OP
N
O
8h
400 MHz/CDCl3
2.07
2.03
1.11
1.00
1.07
1.04
1.00
3.00
3.01
1.04
3.05
3.02
f1 (ppm)


172.534
142.728
142.640
137.118
129.128
126.677
125.544
125.475
123.845
77.478
77.160
76.843
68.927
61.499
59.948
54.463
54.386
53.744
53.670
28.862
28.837
23.170
23.129
22.528
22.500
i-Pr
OH
(MeO)2OP
N
O
8h
100 MHz/CDCl3
f1 (ppm)


24.221
i-Pr
OH
(MeO)2OP
N
O
8h
161.9 MHz/CDCl3
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)