Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Mediator-free electrochemical trifluoromethylation: a cascade approach for the synthesis of trifluoromethylated isoxazolines

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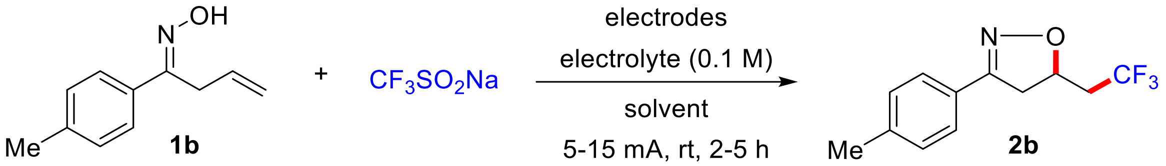
Table of Contents

General Information:

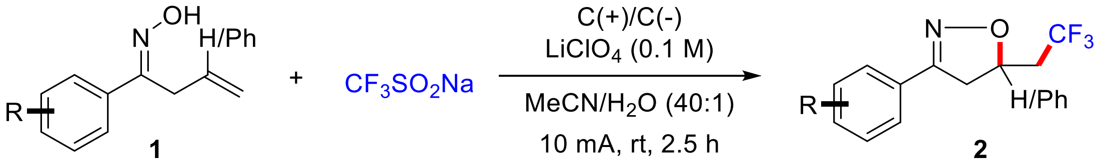
Electrochemical reactions were performed under air using pre-dried glassware. MeCN and other solvents were purchased from Merck Life Science Private Limited and were directly used without further drying or purification. All reagents were obtained from commercial sources and used without further purification. Sodium triflinate as trifluoromethyl source was purchased from TCI. Other chemicals were bought from Avra Chemicals and BLD Pharmatech and used without further purification. Oximes (**1a-1q**)[1] and (**1r-1v**)[2] were synthesized following reported procedures. AXIOMET AX-3003P power supply was used for electrolysis. Platinum electrodes were purchased from Indane Chemical Company, India. Graphite and other electrodes were purchased from IKA. Yields refer to isolated compounds estimated to be >95% pure as determined by ^{1}H NMR. Nuclear magnetic resonance (NMR) spectroscopy was performed using JEOL 400 MHz and Bruker 500 MHz spectrometers, chemical shifts (δ) are provided in ppm. Thin layer chromatography was performed on Merck pre-coated silica gel 60 F254 aluminum sheets with detection under UV light at 254 nm. Chromatographic separations were carried out on Merck silica gel (100−200 mesh) and (230−400 mesh). Cyclic voltammetry was measured on Klyte Research Model 263A. High-resolution mass spectra (HRMS, m/z) were recorded on a Bruker Maxis Impact (TOF) and Waters XEVO G2-XS QTOF mass spectrometers.

Procedure for the Optimization Study:



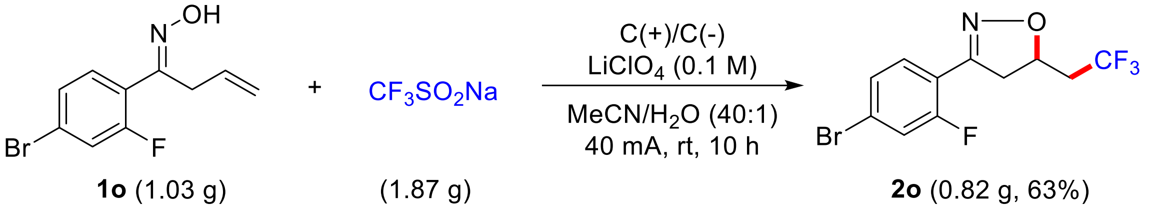
1-phenylbut-3-en-1-one oxime (**1b**, 53 mg, 0.3 mmol), sodium triflinate (1.5-3.0 equiv), and electrolyte (0.1 M) were taken in a dry undivided cell and dissolved in 6 mL of solvent. The cell was equipped with electrodes and the reaction mixture was electrolyzed at 5-15 mA constant current under air atmosphere and room temperature for 2-5 h. After the completion of the reaction, the reaction mixture was concentrated under vacuum and purification was performed by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane to deliver (**2b,** 42-72%) as a white solid.

General Procedure for the Electrochemical Trifluoromethylation (GP1):



Oxime **1** (0.3 mmol, 1.0 equiv), sodium triflinate (140 mg, 0.9 mmol, 3.0 equiv), and lithium perchlorate (64 mg, 0.1 M) were taken in a dry undivided cell and dissolved in 6 mL of $MeCN/H_2O$ (40:1). The cell was equipped with graphite (5.2 cm×0.8 cm×0.2 cm) both as cathode and an anode. Thereafter the reaction mixture was stirred and electrolyzed at a constant current of 10 mA under air atmosphere and room temperature for 2.5 h. After the completion of the reaction, the reaction mixture was concentrated under vacuum and purification was performed by column chromatography in 230-400 silica gel using 1.5-5% ethyl acetate in hexane to deliver trifluoromethylated isoxazoline **2**.

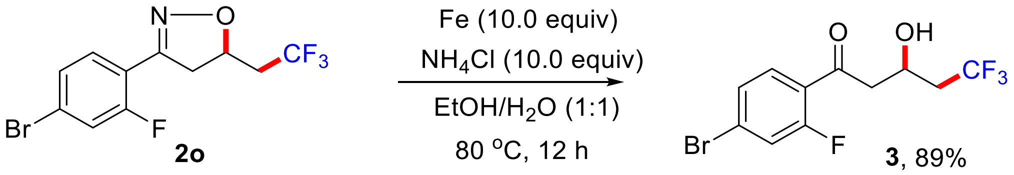
Procedure for the Gram Scale Synthesis of (2o):



1-(4-bromo-2-fluorophenyl)but-3-en-1-one oxime (**1o**, 1.03 g, 3.96 mmol), sodium triflinate (1.85 g, 11.9 mmol, 3.0 equiv), and lithium perchlorate (0.75 g, 0.1 M) were taken in a dry undivided cell and dissolved in 70 mL of MeCN/H_2O (40:1). The cell was equipped with graphite (5.2 cm×3.2 cm×0.2 cm) both as cathode and an anode. Thereafter the reaction mixture was stirred and electrolyzed at a constant current of 40 mA under air atmosphere and room temperature for 10 h. After the completion of the reaction, the reaction mixture was concentrated under vacuum and purification was performed by column chromatography in 230-400 silica gel using 5% ethyl acetate in hexane to deliver 3-(4-bromo-2-fluorophenyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (**2o,** 814 mg, 63%) as a solid.

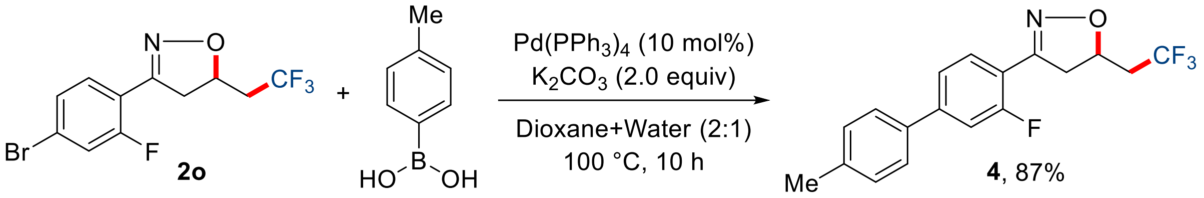
Synthetic Derivatization of 2o:[3, 4]

a)



Fe powder (168 mg, 3 mmol, 10 equiv) was added to isoxazoline **2o** (100 mg, 0.3 mmol, 1 equiv), and NH_4Cl (160 mg, 3 mmol, 10 equiv) in ethanol, and water (1:1, 15 mL) mixture. Then the mixture was stirred at 80 ˚C for 12 hours. After the reaction was completed by TLC monitoring, the reaction mixture was cooled to room temperature, diluted with ethyl acetate, and filtered. The filtrate was washed with water and the organic layer was separated. Then the combined organic layer was dried over Na_2SO_4, evaporated under reduced pressure, and purification of the product by column chromatography in silica gel using ethyl acetate in hexane yielded **3** (87 mg, 89%).

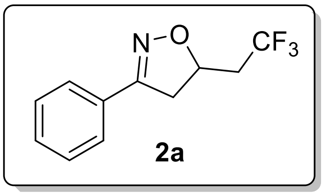
b)



Isoxazoline **2o** (67 mg, 0.2 mmol, 1.0 equiv), *p*-tolylboronic acid (65 mg, 0.24 mmol, 1.2 equiv), and potassium carbonate (55 mg, 0.4 mmol, 2.0 equiv) were taken in an oven-dried 50 ml schlenk round-bottom flask equipped with a stir bar under argon atmosphere. Thereafter 3 ml of degassed dioxane and water (2:1) solvent was added. Then tetrakis(triphenylphosphine)palladium(0) (23 mg, 0.02 mmol, 0.1 equiv) was added under argon atmosphere and the reaction was stirred at 100 °C for 10 h in an oil bath. After the completion of the reaction, the reaction mixture was cooled to room temperature, diluted with ethyl acetate, and washed with water and the organic layer was separated. Then the combined organic layer was dried over Na_2SO_4, evaporated under reduced pressure, and purification of the product by column chromatography in silica gel using ethyl acetate in hexane yielded **4** (59 mg, 87%).

Characterization Data for Synthesized Compounds:

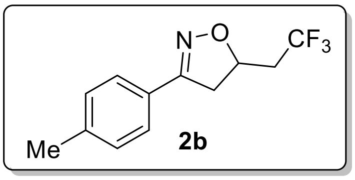
3-phenyl-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2a):[1]



2a

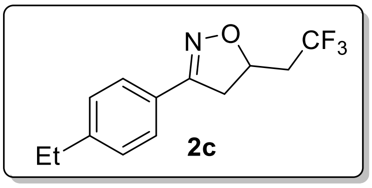
GP 1 was followed using 1-phenylbut-3-en-1-one oxime (**1a**, 48 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2a,** 47 mg, 68%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71 – 7.63 (m, 2H), 7.48 – 7.39 (m, 3H), 5.03 – 4.95 (m, 1H), 3.57 (dd, *J* = 16.7, 10.3 Hz, 1H), 3.17 (dd, *J* = 16.7, 8.0 Hz, 1H), 2.80 – 2.67 (m, 1H), 2.51 – 2.38 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 156.7, 130.6, 129.1, 129.0, 126.9, 125.4 (q, *J* = 276.8 Hz), 74.9 (q, *J* = 3.0 Hz), 40.6, 39.3 (q, *J* = 27.8 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8. HRMS-ESI (m/z): calcd for $C_{11}H_{11}F_3NO$ $[M + H]^+$ 230.0793; found 230.0786.

3-(*p*-tolyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2b): [1]


2b

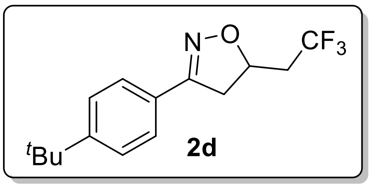
GP 1 was followed using 1-(*p*-tolyl)but-3-en-1-one oxime (**1b**, 53 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2b,** 52 mg, 72%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.2 Hz, 2H), 7.22 (d, *J* = 8.1 Hz, 2H), 5.01 – 4.93 (m, 1H), 3.54 (dd, *J* = 16.7, 10.2 Hz, 1H), 3.14 (dd, J = 16.7, 8.0 Hz, 1H), 2.79 – 2.66 (m, 1H), 2.48 – 2.34 (m, 1H), 2.39 (s, 3H).**^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 156.7, 140.9, 129.7, 126.8, 126.3, 125.5 (q, *J* = 277.0 Hz), 74.8 (q, *J* = 3 Hz), 40.7, 39.3 (q, *J* = 27.7 Hz), 21.6. **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8. HRMS-ESI (m/z): calcd for $C_{12}H_{13}F_3NO$ $[M + H]^+$ 244.0949; found 244.0953.

3-(4-ethylphenyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2c):


2c

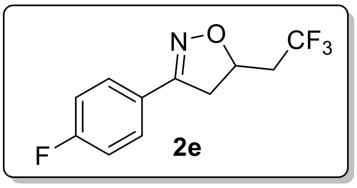
GP 1 was followed using 1-(4-ethylphenyl)but-3-en-1-one oxime (**1c**, 57 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2c,** 49 mg, 64%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.2 Hz, 2H), 7.25 (d, *J* = 8.2 Hz, 2H), 5.01 – 4.93 (m, 1H), 3.55 (dd, *J* = 16.7, 10.2 Hz, 1H), 3.15 (dd, *J* = 16.7, 7.9 Hz, 1H), 2.78 – 2.70 (m, 1H), 2.68 (q, *J* = 7.6 Hz, 2H), 2.54 – 2.35 (m, 1H), 1.25 (t, *J* = 7.6 Hz, 3H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 156.7, 147.2, 128.5, 126.9, 126.5, 125.4 (q, *J* = 277.1 Hz), 74.8 (q, *J* = 3.2 Hz), 40.7, 39.3 (q, *J* = 27.8 Hz), 28.9, 15.5. **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8. HRMS-ESI (m/z): calcd for $C_{13}H_{15}F_3NO$ $[M + H]^+$ 258.1106; found 258.1093.

3-(4-(*tert*-butyl)phenyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2d):


2d

GP 1 was followed using 1-(4-(tert-butyl)phenyl)but-3-en-1-one oxime (**1d**, 65 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2d,** 54 mg, 63%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.4 Hz, 2H), 7.44 (d, *J* = 8.4 Hz, 2H), 4.99 – 4.95 (m, 1H), 3.55 (dd, J = 16.7, 10.2 Hz, 1H), 3.15 (dd, *J* = 16.7, 7.9 Hz, 1H), 2.88 – 2.59 (m, 1H), 2.60 – 2.18 (m, 1H), 1.34 (s, 9H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 156.6, 154.1, 126.7, 126.2, 125.9, 125.5 (q, *J* = 276.8 Hz), 74.8 (q, *J* = 3 Hz), 40.7, 39.3 (q, *J* = 27.6 Hz), 35.0, 31.3. **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8. HRMS-ESI (m/z): calcd for $C_{15}H_{19}F_3NO$ $[M + H]^+$ 286.1419; found 286.1403.

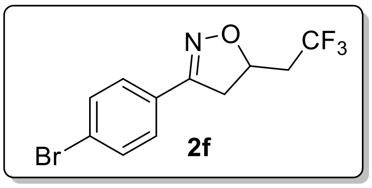
3-(4-fluorophenyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2e):



2e

GP 1 was followed using 1-(4-fluorophenyl)but-3-en-1-one oxime (**1e**, 54 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 2% ethyl acetate in hexane yielded (**2e,** 55 mg, 74%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 – 7.44 (m, 2H), 7.20 – 6.90 (m, 2H), 5.03 – 4.95 (m, 1H), 3.54 (dd, *J* = 16.7, 10.3 Hz, 1H), 3.14 (dd, *J* = 16.7, 8.0 Hz, 1H), 2.81 – 2.63 (m, 1H), 2.57 – 2.31 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 164.1 (d, *J* = 250.7 Hz), 155.8, 128.9 (d, *J* = 7.6 Hz), 125.4 (q, *J* = 278.5 Hz), 125.4 (2, *J* = 2.5 Hz), 116.2 (d, *J* = 22.7 Hz), 75.1 (q, *J* = 2.5 Hz), 40.6, 39.3 (q, *J* = 27.7 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8, -109.3. HRMS-ESI (m/z): calcd for $C_{11}H_{10}F_4NO$ $[M + H]^+$ 248.0699; found 248.0696.

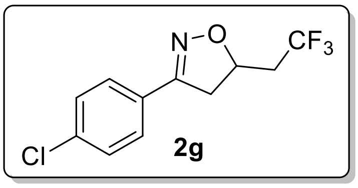
3-(4-bromophenyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2f):



2f

GP 1 was followed using 1-(4-bromophenyl)but-3-en-1-one oxime (**1f**, 72 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2f,** 61 mg, 66%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70 – 7.44 (m, 4H), 5.09 – 4.90 (m, 1H), 3.53 (dd, J = 16.8, 10.4 Hz, 1H), 3.13 (dd, *J* = 16.8, 8.1 Hz, 1H), 2.94 – 2.54 (m, 1H), 2.57 – 2.34 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 155.9, 132.2, 128.3, 128.0, 125.4 (q, *J* = 277 Hz), 124.9, 75.3 (q, *J* = 2.7 Hz), 40.4, 39.2 (q, *J* = 28.5 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8. HRMS-ESI (m/z): calcd for $C_{11}H_{10}BrF_3NO$ $[M + H]^+$ 307.9898; found 307.9892.

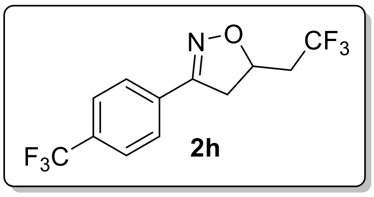
3-(4-chlorophenyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2g):[1]



2g

GP 1 was followed using 1-(4-chlorophenyl)but-3-en-1-one oxime (**1g**, 59 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 2% ethyl acetate in hexane yielded (**2g,** 60 mg, 76%) as a white solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.2 Hz, 2H), 7.38 (d, *J* = 8.2 Hz, 2H), 5.11 – 4.86 (m, 1H), 3.53 (dd, *J* = 16.7, 10.3 Hz, 1H), 3.13 (dd, *J* = 16.7, 8.1 Hz, 1H), 2.77 – 2.67 (m, 1H), 2.55 – 2.37 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 155.8, 136.6, 129.2, 128.1, 127.6, 126.4 (q, *J* = 279.1 Hz) 75.2 (q, *J* = 3 Hz), 40.4, 39.2 (q, *J* = 28 Hz) .**^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.9. HRMS-ESI (m/z): calcd for $C_{11}H_{10}ClF_3NO$ $[M + H]^+$ 264.0403; found 264.0397.

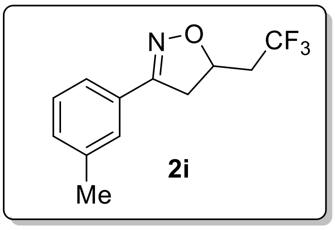
5-(2,2,2-trifluoroethyl)-3-(4-(trifluoromethyl)phenyl)-4,5-dihydroisoxazole (2h):[3]



2h

GP 1 was followed using 1-(4-(trifluoromethyl)phenyl)but-3-en-1-one oxime (**1h**, 69 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 2.5% ethyl acetate in hexane yielded (**2h,** 55 mg, 62%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 5.08 – 5.00 (m, 1H), 3.58 (dd, *J* = 16.8, 10.4 Hz, 1H), 3.18 (dd, *J* = 16.8, 8.2 Hz, 1H), 2.85 – 2.64 (m, 1H), 2.60 – 2.36 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 155.7, 132.5, 132.3 (q, *J* = 32.8 Hz), 127.1, 126.0 (q, *J* = 3.8 Hz), 125.3 (q, *J* = 278.5 Hz), 123.9 (q, *J* = 273.4 Hz), 75.6 (q, *J* = 2.5 Hz), 40.2, 39.2 (q, *J* = 29 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.0, -63.8. HRMS-ESI (m/z): calcd for $C_{12}H_{10}F_6NO$ $[M + H]^+$ 298.0667; found 298.0669.

3-(*m*-tolyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2i):[1]

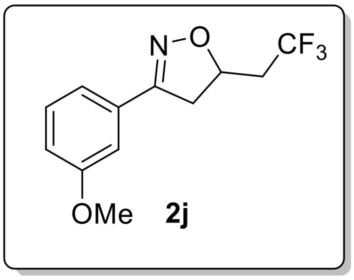


2i

GP 1 was followed using 1-(*m*-tolyl)but-3-en-1-one oxime (**1i**, 53 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2i,** 47 mg, 65%) as solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.45 (d, *J* = 7.6

Hz, 1H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.25 (d, *J* = 7.6 Hz, 1H), 5.01–4.95 (m, 1H), 3.55 (dd, *J* = 16.7, 10.3 Hz, 1H), 3.15 (dd, *J* = 16.7, 7.9 Hz, 1H), 2.78–2.67 (m, 1H), 2.50 – 2.41 (m, 1H), 2.39 (s, 3H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 156.8, 138.7, 131.4, 129.1, 128.9, 127.5, 125.5 (q, *J* = 277.2 Hz), 124.1, 74.9 (q, *J* = 2.5 Hz), 40.7, 39.3 (q, *J* = 28.9 Hz), 21.5. **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8.

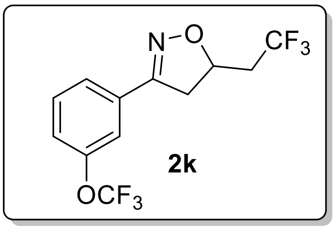
3-(3-methoxyphenyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2j):



2j

GP 1 was followed using 1-(3-methoxyphenyl)but-3-en-1-one oxime (**1j**, 57 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 5% ethyl acetate in hexane yielded (**2j,** 48 mg, 62%) as solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 (t, *J* = 8.0 Hz, 1H), 7.27 (s, 1H), 7.17 (d, *J* = 8.0 Hz, 1H), 6.98 (dd, *J* = 8.1, 2.5 Hz, 1H), 5.02-4.94 (m, 1H), 3.83 (s, 3H), 3.54 (dd, *J* = 16.7, 10.3 Hz, 1H), 3.14 (dd, *J* = 16.7, 7.9 Hz, 1H), 2.78-2.65 (m, 1H), 2.55 – 2.21 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 159.9, 156.7, 130.3, 130.0, 125.4 (q, *J* = 277.2 Hz), 119.5, 116.9, 111.5, 75.0 (q, *J* = 2.5 Hz), 55.5 (q, *J* = 3.8 Hz). 40.6, 39.3 (q, *J* = 27.7 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8. HRMS-ESI (m/z): calcd for $C_{12}H_{13}F_3NO_2$ $[M + H]^+$ 260.0898; found 260.0896.

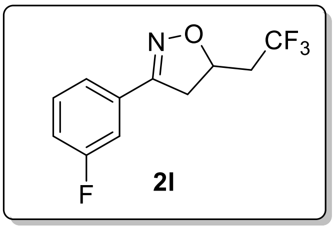
5-(2,2,2-trifluoroethyl)-3-(3-(trifluoromethoxy)phenyl)-4,5-dihydroisoxazole (2k):



2k

GP 1 was followed using 1-(3-(trifluoromethoxy)phenyl)but-3-en-1-one oxime (**1k**, 73 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 5% ethyl acetate in hexane yielded (**2k,** 53 mg, 57%) as solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.8 Hz, 1H), 7.54 (s, 1H), 7.46 (t, *J* = 8.0 Hz, 1H), 7.29 (d, *J* = 8.2 Hz, 1H), 5.07–4.99 (m, 1H), 3.55 (dd, *J* = 16.8, 10.4 Hz, 1H), 3.15 (dd, *J* = 16.8, 8.1 Hz, 1H), 2.76–2.71 (m, 1H), 2.60 – 2.30 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 155.7, 149.7, 131.2, 130.5, 125.3 (q, *J* = 277.2 Hz), 125.2, 122.9, 120.6 (q, *J* = 257 Hz), 119.3, 75.4 (q, *J* = 2.5 Hz), 40.3, 39.2 (q, *J* = 28.9 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -57.9, -63.8. HRMS-ESI (m/z): calcd for $C_{12}H_9F_6NNaO_2$ $[M + Na]^+$ 336.0435; found 336.0410.

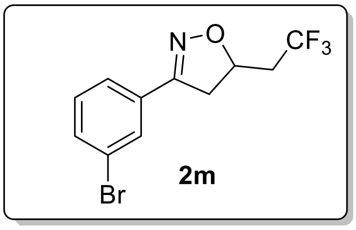
3-(3-fluorophenyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2l):



2l

GP 1 was followed using 1-(3-fluorophenyl)but-3-en-1-one oxime (**1l**, 74 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 2% ethyl acetate in hexane yielded (**2l,** 51 mg, 70%) as solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.47 – 7.31 (m, 3H), 7.19 – 7.07 (m, 1H), 5.05 – 4.97 (m, 1H), 3.54 (dd, J = 16.8, 10.4 Hz, 1H), 3.14 (dd, J = 16.7, 8.1 Hz, 1H), 2.86 – 2.64 (m, 1H), 2.59 – 2.29 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 162.9 (d, J = 246.9 Hz), 155.9 (d, J = 2.5 Hz), 131.2 (d, J = 7.5 Hz), 130.6 (d, J = 8.8 Hz), 125.3 (q, J = 278.4 Hz), 122.6 (d, J = 2.5 Hz), 117.6 (d, J = 21.42 Hz), 113.8 (d, J = 23.9 Hz), 75.3 (q, J = 3.8 Hz), 40.4, 39.2 (q, J = 27.7 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.9, -111.9. HRMS-ESI (m/z): calcd for $C_{11}H_{10}F_4NO$ $[M + H]^+$ 248.0699; found 248.0683.

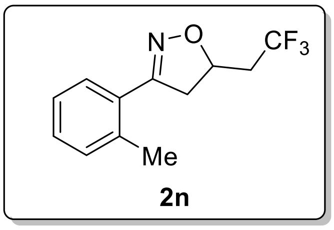
3-(3-bromophenyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2m):



2m

GP 1 was followed using 1-(3-bromophenyl)but-3-en-1-one oxime (**1m**, 72 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 2% ethyl acetate in hexane yielded (**2m,** 59 mg, 64%) as a semi solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.60 (d, J = 7.8 Hz, 1H), 7.56 (d, J = 7.9 Hz, 1H), 7.29 (t, J = 7.9 Hz, 1H), 5.05 – 4.97 (m, 1H), 3.53 (dd, J = 16.8, 10.4 Hz, 1H), 3.13 (dd, J = 16.8, 8.1 Hz, 1H), 2.79 – 2.67 (m, J = 21.3, 10.8, 5.5 Hz, 1H), 2.60 – 2.31 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 155.6, 133.5, 131.1, 130.5, 129.8, 125.4 (q, J = 277.2 Hz), 125.4, 123.1, 75.3 (q, J = 3.8 Hz), 40.3, 39.2 (q, J = 27.7 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8. HRMS-ESI (m/z): calcd for $C_{11}H_{10}BrF_3NO$ $[M + H]^+$ 307.9898; found 307.9891.

3-(*o*-tolyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2n):[3]

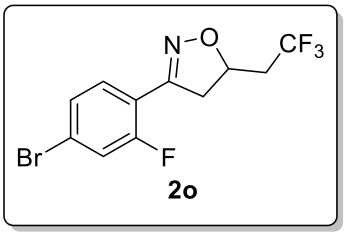


2n

GP 1 was followed using 1-(*o*-tolyl)but-3-en-1-one oxime (**1n**, 53 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2n,** 51 mg, 70%) as solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.27 – 7.21 (m, 2H), 7.20 –

7.16 (m, 2H), 4.87 (m, 1H), 3.53 (dd, *J* = 16.6, 10.2 Hz, 1H), 3.14 (dd, *J* = 16.7, 8.0 Hz, 1H), 2.81 – 2.56 (m, 1H), 2.49 (s, 3H), 2.45 – 2.29 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 157.6, 138.3, 131.9, 129.8, 129.0, 128.2, 126.1, 125.5 (q, *J* = 277.4 Hz), 73.9 (q, *J* = 3.0 Hz), 43.1, 39.2 (q, *J* = 27.8 Hz), 23.1. **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8.

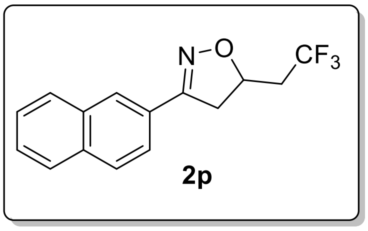
3-(4-bromo-2-fluorophenyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2o):



2o

GP 1 was followed using 1-(4-bromo-2-fluorophenyl)but-3-en-1-one oxime (**1o**, 78 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 5% ethyl acetate in hexane yielded (**2o,** 74 mg, 76%) as solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 – 7.67 (m, 1H), 7.34 – 7.26 (m, 2H), 5.03 – 4.95 (m, 1H), 3.62 (dd, *J* = 17.4, 10.4 Hz, 1H), 3.22 (dd, *J* = 17.5, 8.1 Hz, 1H), 2.84 – 2.57 (m, 1H), 2.60 – 2.30 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 160.1 (d, *J* = 256.9 Hz), 152.8 (d, *J* = 3.3 Hz), 130.0 (d, *J* = 3.7 Hz), 128.3 (d, *J* = 3.2 Hz), 125.3 (q, *J* = 277.4 Hz), 125.1 (d, *J* = 9.6 Hz), 120.3 (d, *J* = 25.3 Hz), 116.4 (d, *J* = 11.6 Hz), 75.5 – 75.3 (m), 42.0 (d, *J* = 7.2 Hz), 39.2 (q, *J* = 28.0 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.9, -110.6. HRMS-ESI (m/z): calcd for $C_{11}H_9BrF_4NO$ $[M + H]^+$ 325.9804; found 325.9790.

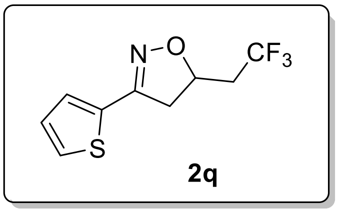
3-(naphthalen-2-yl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2p):[3]



2p

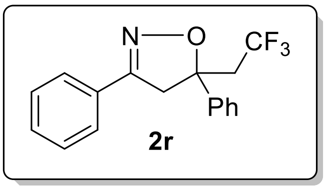
GP 1 was followed using 1-(naphthalen-2-yl)but-3-en-1-one oxime (**1p**, 63 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 3% ethyl acetate in hexane yielded (**2p,** 40 mg, 53%) as solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 – 7.95 (m, 1H), 7.89 – 7.83 (m, 4H), 7.62 – 7.49 (m, 2H), 5.18 – 4.92 (m, 1H), 3.69 (dd, *J* = 16.6, 10.2 Hz, 1H), 3.30 (dd, *J* = 16.6, 8.0 Hz, 1H), 2.97 – 2.64 (m, 1H), 2.63 – 2.34 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 156.9, 134.3, 133.1, 128.9, 128.6, 128.0, 127.5, 127.3, 127.0, 126.7, 125.5 (d, *J* = 262.8 Hz), 123.6, 75.1 (q, *J* = 3.0Hz), 40.6, 39.4 (q, *J* = 27.8 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.9, -110.6.

3-(thiophen-2-yl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2q):[3]


2q

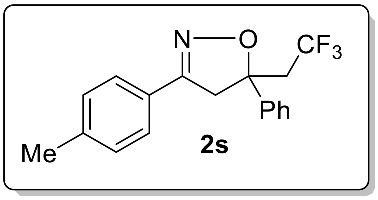
GP 1 was followed using 1-(thiophen-2-yl)but-3-en-1-one oxime (**1q**, 50 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 2% ethyl acetate in hexane yielded (**2q,** 50 mg, 69%) as solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42 (dd, *J* = 4.9, 0.9 Hz, 1H), 7.22 (dd, *J* = 3.7, 0.9 Hz, 1H), 7.08 (dd, *J* = 5.0, 3.7 Hz, 1H), 5.13 – 4.88 (m, 1H), 3.57 (dd, *J* = 16.4, 10.3 Hz, 1H), 3.18 (dd, *J* = 16.5, 8.0 Hz, 1H), 2.80 – 2.67 (m, 1H), 2.51– 2.37 (m, 1H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 152.5, 131.5, 128.9, 128.9, 127.5, 125.4 (d, *J* = 277.0 Hz), 75.2 (q, *J* = 3.0 Hz), 41.4, 39.2 (q, *J* = 27.8 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -63.8.

3,5-diphenyl-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2r):[5]


2r

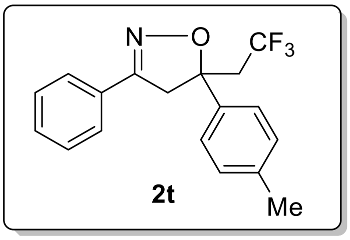
GP 1 was followed using 1,3-diphenylbut-3-en-1-one oxime (**1r**, 71 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2r,** 78 mg, 85%) as solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.68 – 7.64 (m, 2H), 7.51 (d, *J* = 7.7 Hz, 2H), 7.44 – 7.37 (m, 5H), 7.36 – 7.29 (m, 1H), 3.80 (d, *J* = 16.7 Hz, 1H), 3.67 (d, *J* = 16.7 Hz, 1H), 2.96 – 2.90 (m, 2H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 156.6, 142.7, 130.5, 129.2, 128.9, 128.8, 128.3, 126.8, 125.1, δ 125.0 (q, *J* = 278.4 Hz), 86.5 (q, *J* = 1.9 Hz), 46.7 (d, *J* = 1.6 Hz), 44.3 (q, *J* = 27.3 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -60.5.

5-phenyl-3-(*p*-tolyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2s):[5]


2s

GP 1 was followed using 3-phenyl-1-(*p*-tolyl)but-3-en-1-one oxime (**1s**, 75 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2s,** 84 mg, 88%) as solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.2 Hz, 2H), 7.51 (d, *J* = 7.4 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.34 – 7.29 (m, 1H), 7.20 (d, *J* = 8.0 Hz, 2H), 3.77 (d, *J* = 16.7 Hz, 1H), 3.65 (d, *J* = 16.7 Hz, 1H), 2.92 (qd, *J* = 10.3, 0.9 Hz, 2H), 2.37 (s, 3H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 156.5, 142.7, 140.9, 129.6, 128.8, 128.2, 126.8, 126.4, 125.2, δ 125.0 (q, *J* = 276.1 Hz), 86.2 (q, *J* = 2.0 Hz), 46.8 (d, *J* = 1.5 Hz), 44.3 (q, *J* = 27.2 Hz), 21.6. **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -60.4.

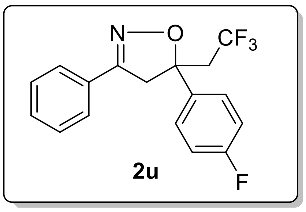
3-phenyl-5-(*p*-tolyl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2t):[5]



2t

GP 1 was followed using 1-phenyl-3-(*p*-tolyl)but-3-en-1-one oxime (**1t**, 75 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2t,** 86 mg, 89%) as solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.6 Hz, 2H), 7.41 – 7.40 (m, 5H), 7.23 (t, *J* = 14.4 Hz, 2H), 3.78 (d, *J* = 16.7 Hz, 1H), 3.66 (d, *J* = 16.7 Hz, 1H), 2.93 (q, *J* = 10.4 Hz, 2H), 2.36 (s, 3H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 156.6, 139.7, 138.0, 130.5, 129.4, 129.3, 128.9, 126.8, 125.0, 125.0 (q, *J* = 278.4 Hz), 86.4 (d, *J* = 1.9 Hz), 46.6, 44.3 (q, *J* = 27.2), 21.1. **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -60.4.

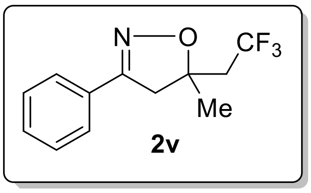
5-(4-fluorophenyl)-3-phenyl-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2u):[5]



2u

GP 1 was followed using 3-(4-fluorophenyl)-1-phenylbut-3-en-1-one oxime (**1u**, 77 mg, 0.2 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded (**2u,** 89 mg, 92%) as solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.69 – 7.60 (m, 2H), 7.54 – 7.45 (m, 2H), 7.45 – 7.36 (m, 3H), 7.11 – 7.03 (m, 2H), 3.79 (d, *J* = 16.7 Hz, 1H), 3.64 (d, *J* = 16.7 Hz, 1H), 3.03 – 2.75 (m, 2H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 162.5 (d, *J* = 247.3 Hz), 156.6, 138.3 (d, *J* = 3.1 Hz), 130.6, 129.0, 128.9, 127.1 (d, *J* = 8.3 Hz), 126.8, 124.9 (q, *J* = 278.3 Hz), 115.7 (d, *J* = 21.7 Hz), 86.1 (d, *J* = 1.9 Hz), 46.9, 44.4 (q, *J* = 27.2 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -60.4, -114.0.

5-methyl-3-phenyl-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2v):[1]

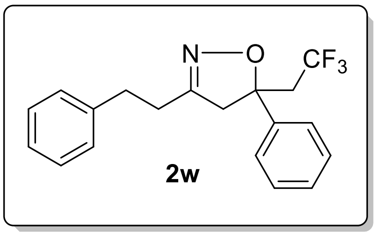


2v

GP 1 was followed using 3-methyl-1-phenylbut-3-en-1-one oxime (**1v**, 53 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 0.5% ethyl acetate in hexane yielded (**2v,** 67 mg, 92%) as solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.71 – 7.58 (m, 2H), 7.48 –

7.34 (m, 3H), 3.40 (d, *J* = 16.8 Hz, 1H), 3.17 (d, *J* = 16.8 Hz, 1H), 2.68 – 2.59 (m, 2H), 1.58 (s, 3H). **^{13}C NMR{^{1}H}** (126 MHz, $CDCl_3$) δ 156.7, 130.4, 129.6, 128.9, 126.7, 125.4 (q, *J* = 277.5 Hz), 83.4 (q, *J* = 1.9 Hz), 45.8 (d, *J* = 1.7 Hz), 43.1 (q, *J* = 27.4 Hz), 25.7 (d, *J* = 1.7 Hz). **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -61.4.

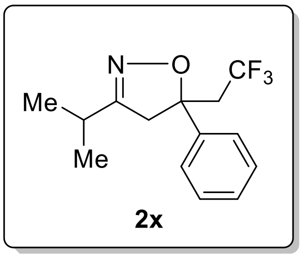
3-phenethyl-5-phenyl-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2w):



2w

GP 1 was followed using 1,5-diphenylhex-5-en-3-one oxime (**1w**, 79 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 0.75% ethyl acetate in hexane yielded (**2w,** 65 mg, 63%) as solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.44 – 7.37 (m, 5H), 7.35 – 7.33 (m, 1H), 7.29 – 7.28 (m, 2H), 7.15 (d, *J* = 7.1 Hz, 2H), 3.32 (d, *J* = 17.1 Hz, 1H), 3.16 (d, *J* = 17.0 Hz, 1H), 2.90 (td, *J* = 8.0, 3.7 Hz, 2H), 2.78 (qd, *J* = 10.3, 2.1 Hz, 2H), 2.67 (t, *J* = 7.9 Hz, 2H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 158.4, 142.9, 140.3, 128.7, 128.7, 128.4, 128.1, 126.5, 125.1, 125.0 (q, *J* = 278.0 Hz), 85.3 (q, *J* = 2.3 Hz), 48.9 (q, *J* = 2.2 Hz), 44.1 (q, *J* = 27.1 Hz), 32.7, 29.6. **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -60.6. HRMS-ESI (m/z): calcd for $C_{19}H_{19}F_3NO$ $[M + H]^+$ 334.1419; found 334.1414.

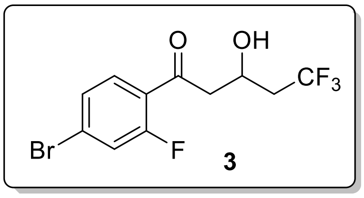
3-isopropyl-5-phenyl-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (2x):



2x

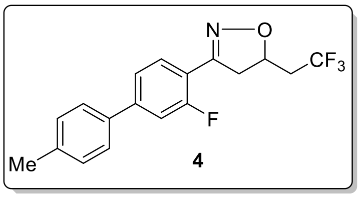
GP 1 was followed using 2-methyl-5-phenylhex-5-en-3-one oxime (**1x**, 60 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol). After 2.5 h, purification by column chromatography in 230-400 silica gel using 0.75% ethyl acetate in hexane yielded (**2x,** 66 mg, 82%) as solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.2 Hz, 2H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.30 (t, *J* = 7.9 Hz, 1H), 3.38 (d, *J* = 16.9 Hz, 1H), 3.22 (d, *J* = 17.0 Hz, 1H), 2.80 (q, *J* = 10.6 Hz, 2H), 2.69 (h, *J* = 7.5 Hz, 1H), 1.16 (d, *J* = 6.9 Hz, 3H), 1.13 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.6, 143.1, 128.7, 128.0, 125.1, 125.1 (q, *J* = 278.2 Hz), 85.1 (q, *J* = 2.0 Hz), 46.6, 44.3 (q, *J* = 26.9 Hz), 28.1, 20.1, 20.0. **^{19}F NMR{^{1}H}** (471 MHz, $CDCl_3$) δ -60.6. HRMS-ESI (m/z): calcd for $C_{14}H_{17}F_3NO$ $[M + H]^+$ 272.1262; found 272.1268.

1-(4-bromo-2-fluorophenyl)-5,5,5-trifluoro-3-hydroxypentan-1-one (3):



^{1}H NMR (400 MHz, CDCl$_3$) δ 7.79 (t, *J* = 8.2 Hz, 1H), 7.40 (ddd, *J* = 12.3, 9.5, 1.7 Hz, 2H), 4.71 – 4.49 (m, 1H), 3.26 – 3.16 (m, 2H), 2.59 – 2.42 (m, 1H), 2.42 – 2.25 (m, 1H), 1.63 (s, 1H). **^{13}C NMR{^{1}H}** (126 MHz, CDCl$_3$) δ 196.4 (d, *J* = 4.4 Hz), 162.0 (d, *J* = 259.8 Hz), 131.7 (d, *J* = 3.0 Hz), 129.4 (d, *J* = 10.0 Hz), 128.5 (d, *J* = 3.4 Hz), 126.1 (q, *J* = 276.9 Hz), 123.8 (d, *J* = 12.5 Hz), 120.7 (d, *J* = 27.1 Hz), 63.0 – 61.6 (m), 49.7 (d, *J* = 7.6 Hz), 40.3 (q, *J* = 27.6 Hz). **^{19}F NMR{^{1}H}** (471 MHz, CDCl$_3$) δ -63.42, -106.38. HRMS-ESI (m/z): calcd for $C_{11}H_{10}BrF_4O_2$ [M + H]$^+$ 328.9800; found 328.9787.

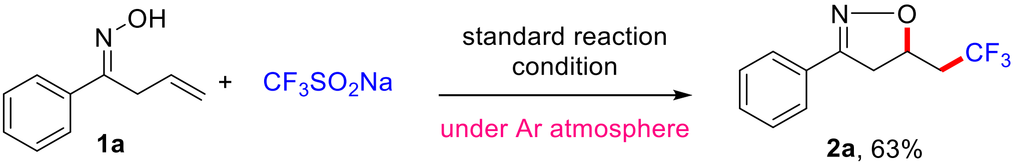
3-(3-fluoro-4'-methyl-[1,1'-biphenyl]-4-yl)-5-(2,2,2-trifluoroethyl)-4,5-dihydroisoxazole (4):



^{1}H NMR (500 MHz, CDCl$_3$) δ 7.90 (t, *J* = 8.0 Hz, 1H), 7.50 (d, *J* = 8.2 Hz, 2H), 7.42 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.34 (dd, *J* = 12.7, 1.7 Hz, 1H), 7.27 (d, *J* = 7.9 Hz, 2H), 5.08 – 4.91 (m, 1H), 3.67 (ddd, *J* = 17.4, 10.3, 2.2 Hz, 1H), 3.27 (ddd, *J* = 17.4, 8.1, 1.8 Hz, 1H), 2.73 (dqd, *J* = 21.2, 10.6, 5.7 Hz, 1H), 2.52 – 2.42 (m, 1H), 2.41 (s, 3H). **^{13}C NMR{^{1}H}** (126 MHz, CDCl$_3$) δ 160.8 (d, *J* = 252.1 Hz), 153.4 (d, *J* = 3.2 Hz), 145.5 (d, *J* = 8.5 Hz), 138.7, 136.0 (d, *J* = 1.9 Hz), 129.3 (d, *J* = 3.6 Hz), 129.0, 126.9, 125.4 (q, *J* = 277.1 Hz), 123.0 (d, *J* = 2.8 Hz), 115.5 (d, *J* = 11.9 Hz), 114.6 (d, *J* = 23.0 Hz), 75.3 – 75.0 (m), 42.3 (d, *J* = 7.1 Hz), 39.3 (q, *J* = 27.9 Hz), 21.3. **^{19}F NMR{^{1}H}** (471 MHz, CDCl$_3$) δ -63.8, -112.7. HRMS-ESI (m/z): calcd for $C_{18}H_{16}F_4NO$ [M + H]$^+$ 338.1163; found 338.1152.

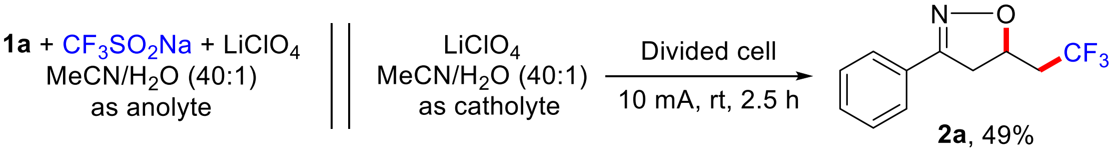
Mechanistic Studies:

a) Electrochemical trifluoromethylation under argon atmosphere:



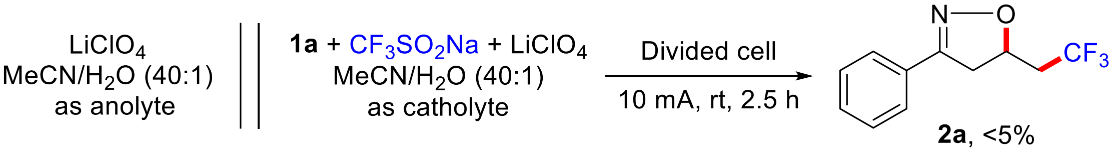
1-phenylbut-3-en-1-one oxime (**1a**, 48 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol), and lithium perchlorate (64 mg, 0.1 M) were taken in a dry undivided cell and dissolved in 6 mL of MeCN/H_2O (40:1). The cell was equipped with graphite (5.2 cm×0.8 cm×0.2 cm) both as cathode and an anode. Thereafter the reaction mixture was stirred and electrolyzed at a constant current of 10 mA under argon atmosphere and room temperature for 2.5 h. After the completion of the reaction, the reaction mixture was concentrated under vacuum and purification was performed by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane to deliver (**2a,** 43 mg, 63%) as a white solid.

b) Divided cell electrolysis at anode:



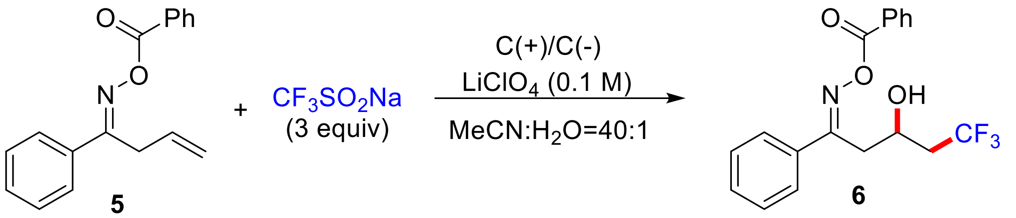
1-phenylbut-3-en-1-one oxime (**1a**, 48 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol), and lithium perchlorate (32 mg, 0.1 M) in 3 mL of MeCN/H_2O (40:1) were taken in the anodic chamber of a dry divided cell (separated by G3 sintered). Lithium perchlorate (32 mg, 0.1 M) and 3 mL of MeCN/H_2O (40:1) were taken in the cathodic chamber of the cell. The cell was equipped with graphite (5.2 cm × 0.8 cm × 0.2 cm) electrodes both as anode and cathode. Thereafter, the reaction mixture was continuously stirred and electrolyzed with an AXIOMET AX-3003P power supply of 10 mA under air atmosphere and room temperature for 2.5 h. After the completion of the reaction, the reaction mixture was concentrated under vacuum and purification by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane yielded **2a** (34 mg, 49%) as a white solid.

c) Divided cell electrolysis at cathode:

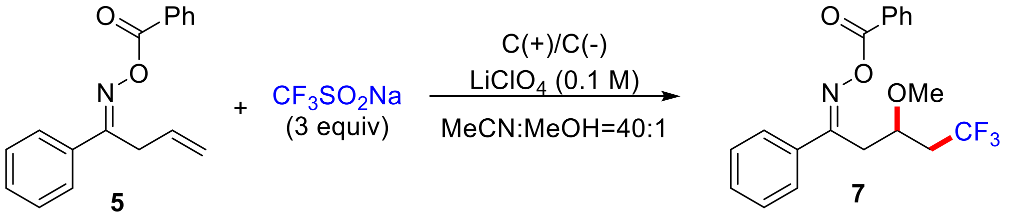


1-phenylbut-3-en-1-one oxime (**1a**, 48 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol), and lithium perchlorate (32 mg, 0.1 M) in 3 mL of MeCN/H_2O (40:1) were taken in the cathodic chamber of a dry divided cell (separated by G3 sintered). Lithium perchlorate (32 mg, 0.1 M) and 3 mL of MeCN/H_2O (40:1) were taken in the anodic chamber of the cell. The cell was equipped with graphite (5.2 cm × 0.8 cm × 0.2 cm) electrodes both as anode and cathode. Thereafter, the reaction mixture was continuously stirred and electrolyzed with an AXIOMET AX-3003P power supply of 10 mA under air atmosphere and room temperature for 2.5 h. After 2.5 h, solvent was evaporated and the crude mixture was analyzed using ^{1}H NMR, where **2a** was not detected.

d) Control experiments involving 5:



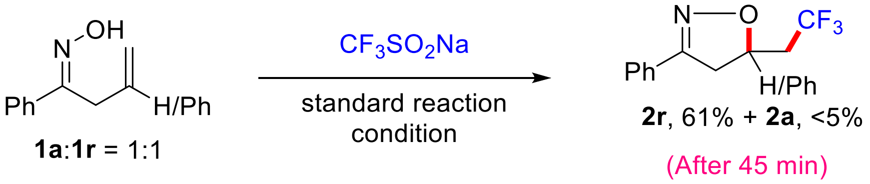
[M + Na$^+$] = 374.1010 was detected by HRMS



[M + Na$^+$] = 388.1146 was detected by HRMS

1-phenylbut-3-en-1-one *O*-benzoyl oxime (**5**, 79 mg, 0.3 mmol), sodium triflinate (140 mg, 0.9 mmol), and lithium perchlorate (64 mg, 0.1 M), were taken in an undivided cell and dissolved in 6 mL of MeCN/H_2O (40:1) or MeCN/MeOH (40:1). The cell was equipped with graphite (5.2 cm×0.8 cm×0.2 cm) both as cathode and anode. Thereafter the reaction mixture was stirred and electrolyzed at a constant current of 10 mA under air atmosphere and room temperature. After 2.5 h, HRMS measurement confirms the formation of **6** and **7** respectively and **2a** was not observed.

e) Control experiments involving 1a and 1r:



2r, 61% + **2a**, <5%

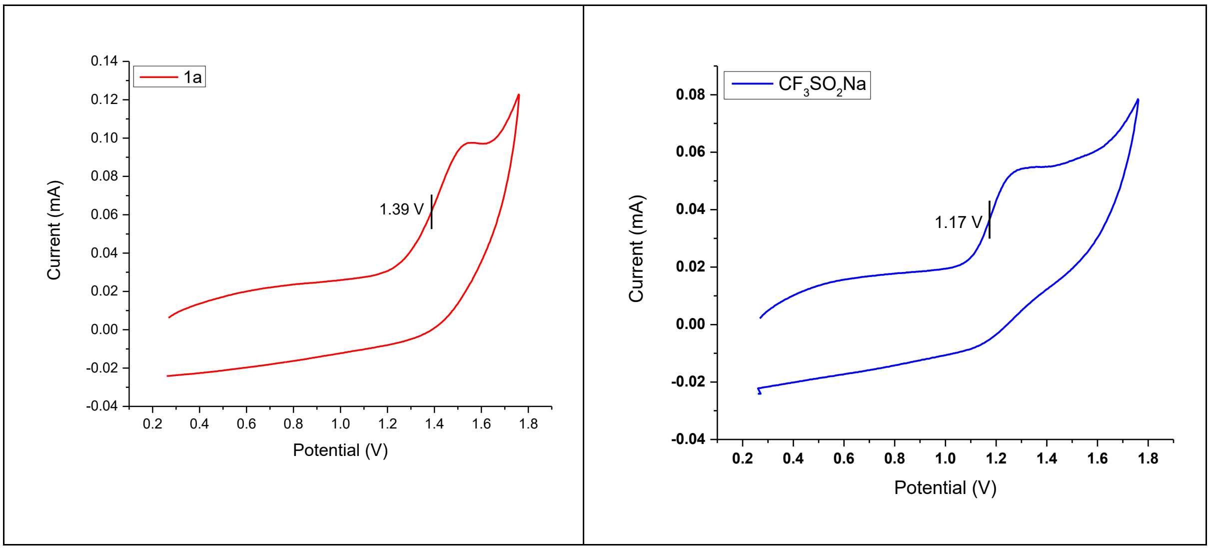
(After 45 min)

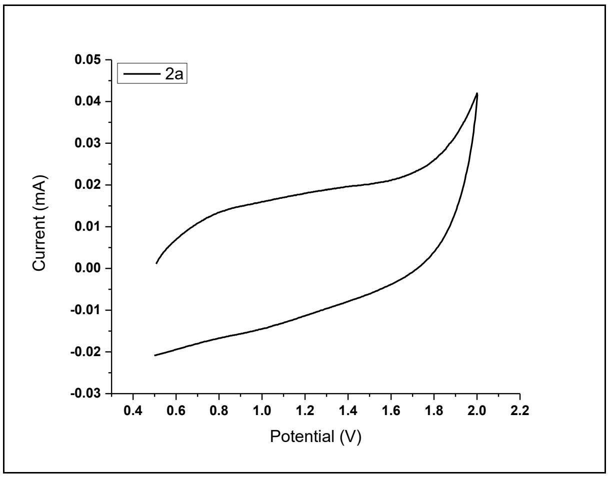
1-phenylbut-3-en-1-one oxime (**1a**, 16 mg, 0.1 mmol), 1,3-diphenylbut-3-en-1-one oxime (**1r**, 24 mg, 0.1 mmol), sodium triflinate (47 mg, 0.3 mmol), and lithium perchlorate (64 mg, 0.1

M) were taken in a dry undivided cell and dissolved in 6 mL of MeCN/H_2O (40:1). The cell was equipped with graphite (5.2 cm×0.8 cm×0.2 cm) both as cathode and an anode. Thereafter the reaction mixture was stirred and electrolyzed at a constant current of 10 mA under argon atmosphere and room temperature for 2.5 h. After 2.5 h, the reaction mixture was monitored by TLC and, **2a** was not detected. Then the reaction mixture was concentrated under vacuum and purification was performed by column chromatography in 230-400 silica gel using 1.5% ethyl acetate in hexane to deliver **2r** (19 mg, 61%) as a white solid.

General Procedure for Cyclic Voltammetry (CV):

Cyclic voltammetry experiment was carried out in a three-electrode cell at room temperature under air using a glassy carbon disk as working electrode and a Pt wire as the counter electrode. MeCN/H_2O (40:1, 5 mL) was employed as the solvent in presence of $LiClO_4$ (0.1 M) as the electrolyte. Reference electrode Ag/AgCl was used. The scan rate was 0.1 V/s. Initially, CV of (**1a**) was measured and exhibited half peak potential ($E_{p/2}$) at 1.39 V. Thereafter CV measurement was done for CF_3SO_2Na which showed the oxidation peaks at 1.17 V. Afterthat CV experiment was done for our desired product **2a** where we did not observed any peak in the operated potential window.





Crystallographic Data

Sample Preparation: The synthesized solid of **2g** was dissolved in a binary mixture of Methanol-Hexane (2:1) into a vial. The nice quality blocked-shaped crystals suitable for SCXRD were obtained after 3 days by the slow evaporation of the solvents.

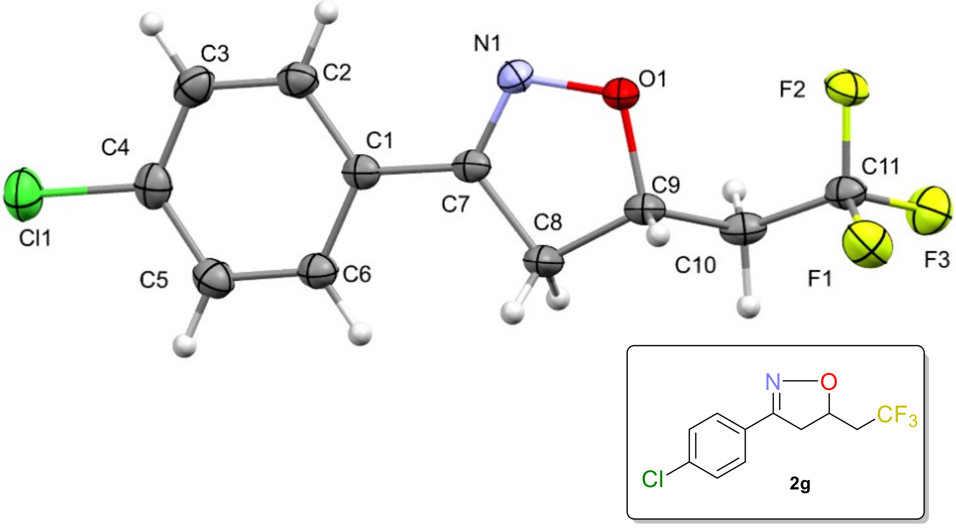


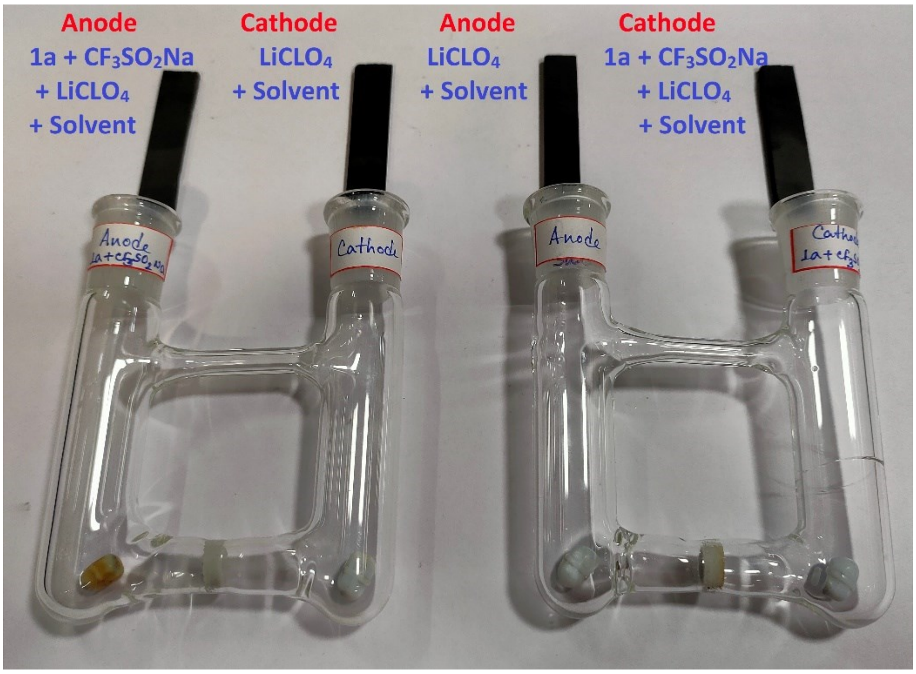
Figure S1: ORTEP diagram of compound 2g.

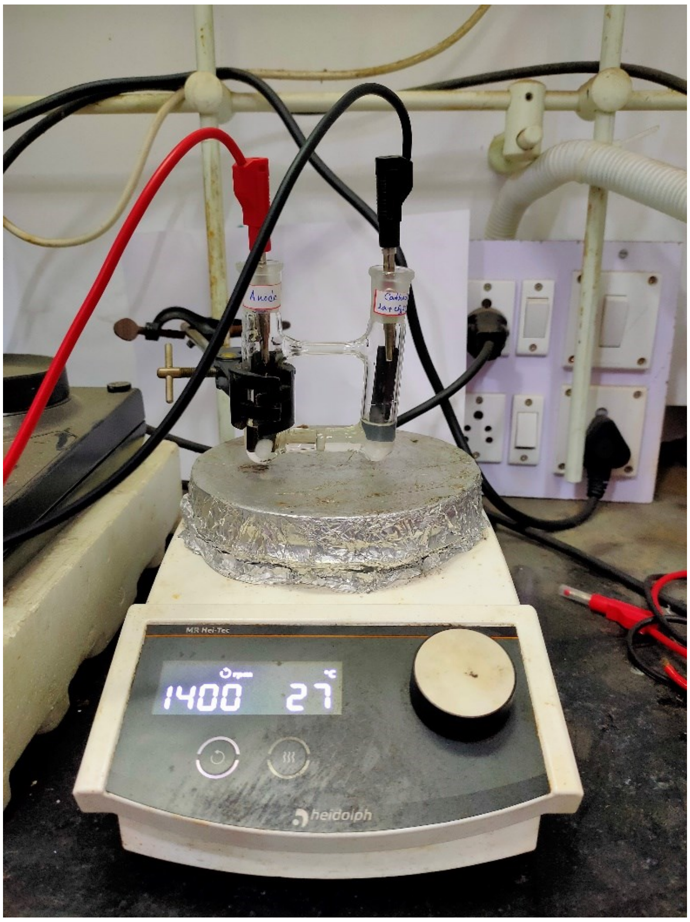
Table S1. Crystallographic and Structural Refinement Parameters of 2g

Compound Name	**2g**
Temperature (K)	100 K

Formula	$C_{11}H_9ClF_3NO$
Formula Weight	263.64
Crystal System	Monoclinic
Space group	$P2_1/c$
a/ Å	14.0286(3)
b/Å	10.8221(2)
c/Å	7.36612(15)
α/°	90.00
β/°	102.292(2)
γ/°	90.00
V/ Å^3	1092.68(4)
Z	4
D_c/ g cm^{-3}	1.603
μ /mm^{-1}	3.370
F(000)	536
θ range/°	3.224-68.182
Measured Refl.	9412
Independent Refl.	1981
R_{int}	0.0491
Goodness of fit (F^2)	1.043
$R1$ (all data)	0.0399
$wR2$ (all data)	0.1049
CCDC No.	2208817

Image of Divided Cell Setup:

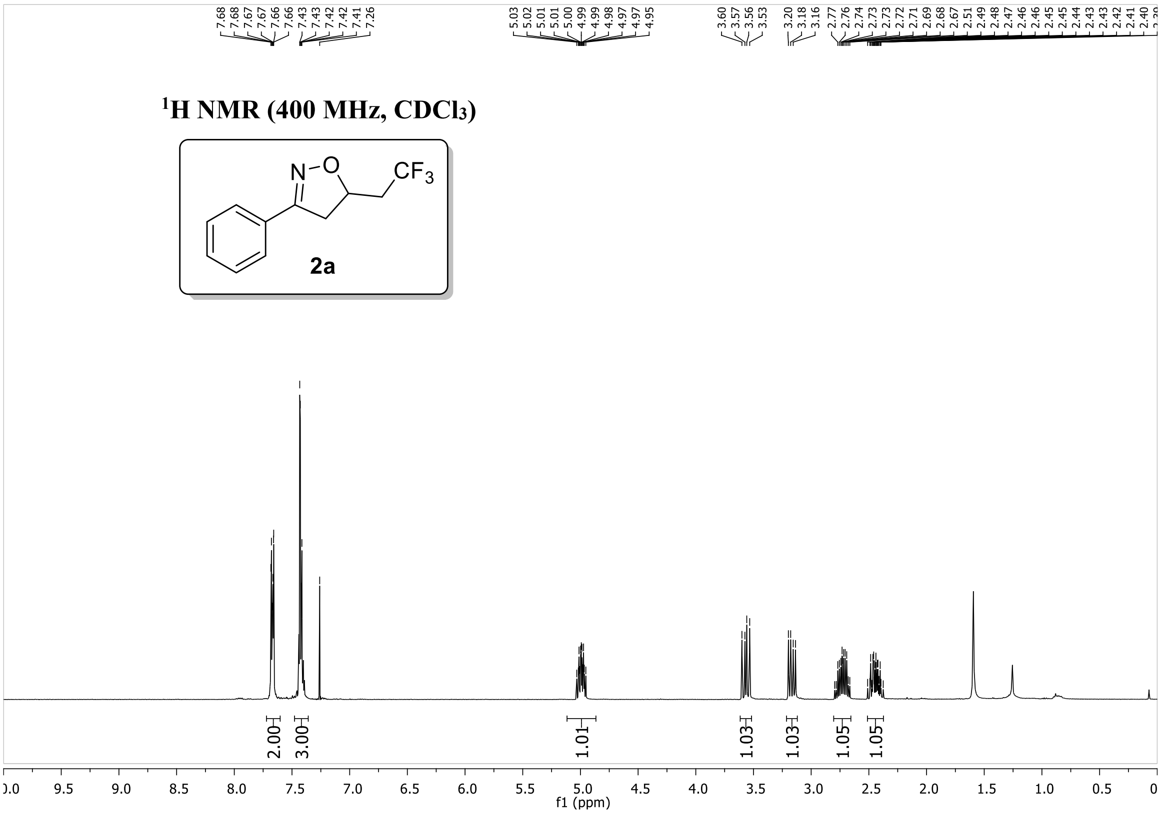


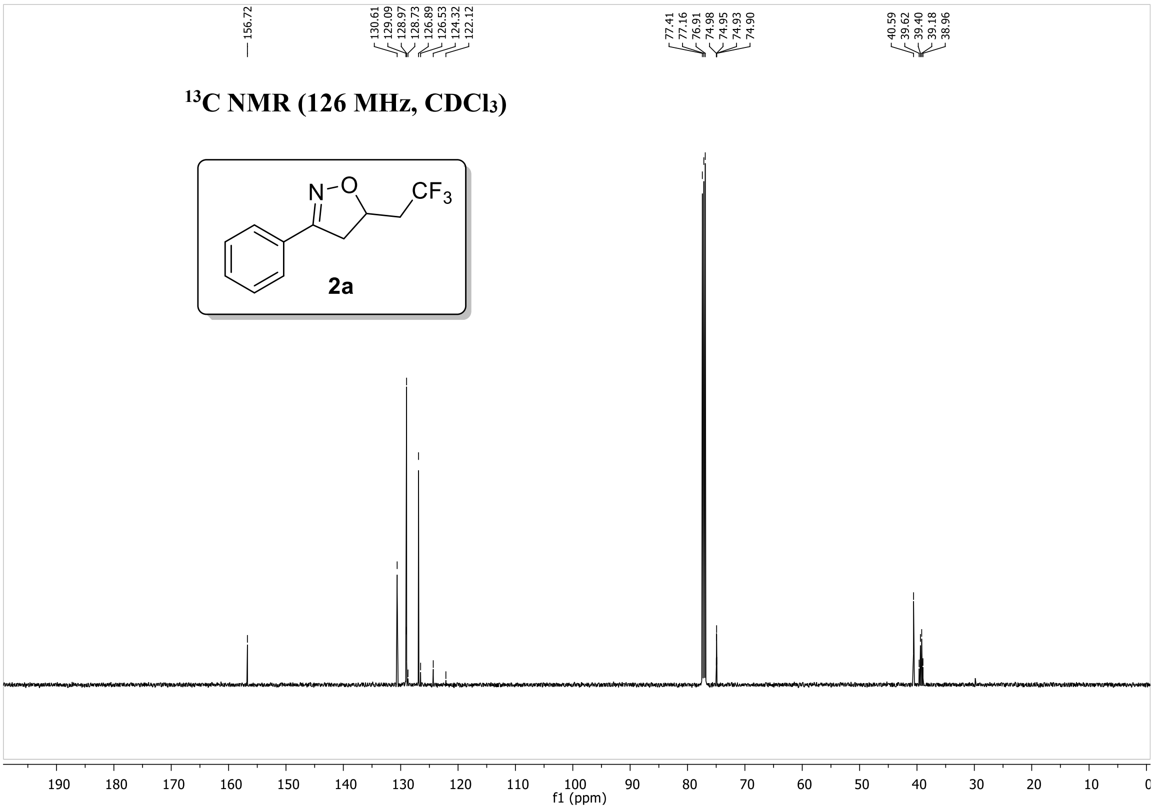


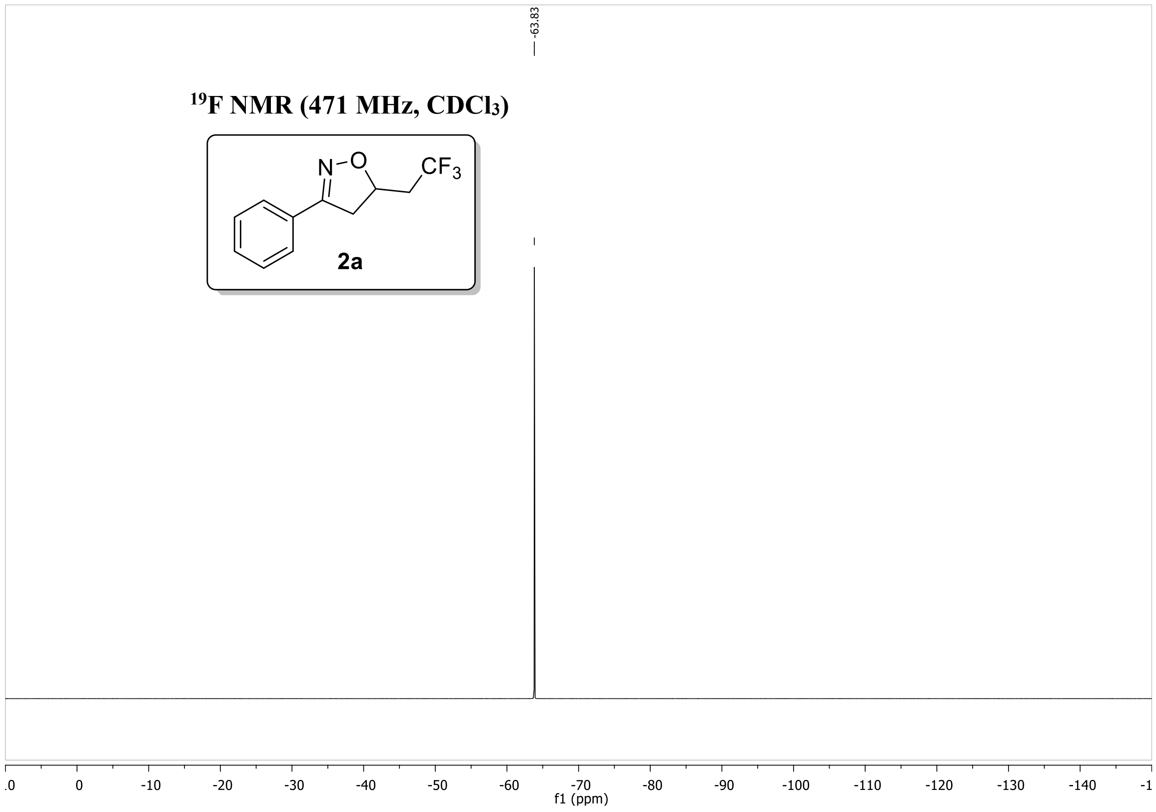
Reference:

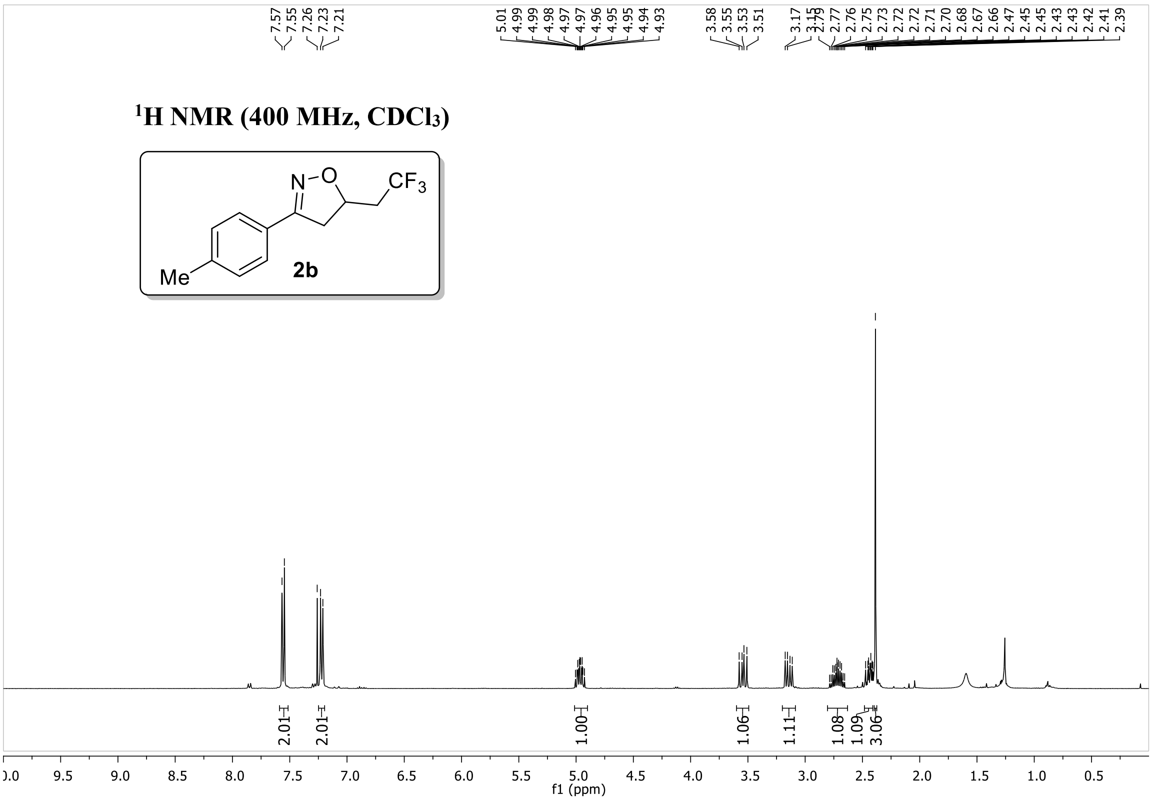
1) Y. T. He, L. H. Li, Y. F. Yang, Y. Q. Wang, J. Y. Luo, X. Y. Liu, Y. M. Liang, *Chem. Commun.* **2013**, *49*, 5687-5689.
2) C. B. Tripathi, S. Mukherjee, *Angew. Chem. Int. Ed.* **2013**, *52*, 8450 –8453.
3) W. Zhang, Y. Su, K. H. Wang, L. Wu, B. Chang, Y. Shi, D. Huang, Y. Hu, *Org. Lett.* **2017**, *19*, 376−379.
4) M. Christakakou, M. Schön, M. Schnürch, M. D. Mihovilovic, *Synlett* **2013**, *24*, 2411–2418.
5) Q. Wei, J. R. Chen, X. Q. Hu, X. C. Yang, B. Lu, W. J. Xiao, *Org. Lett.* **2015**, *17*, 4464−4467.

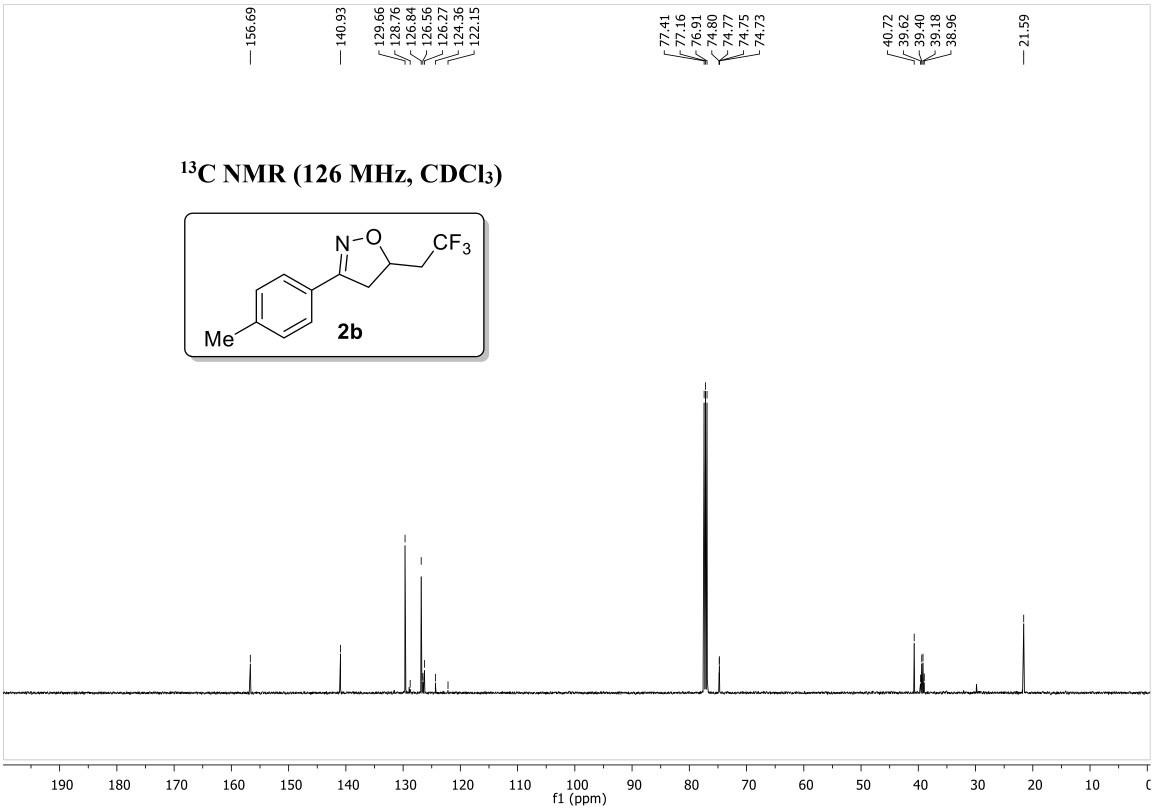
NMR Spectra of Synthesized Compounds:

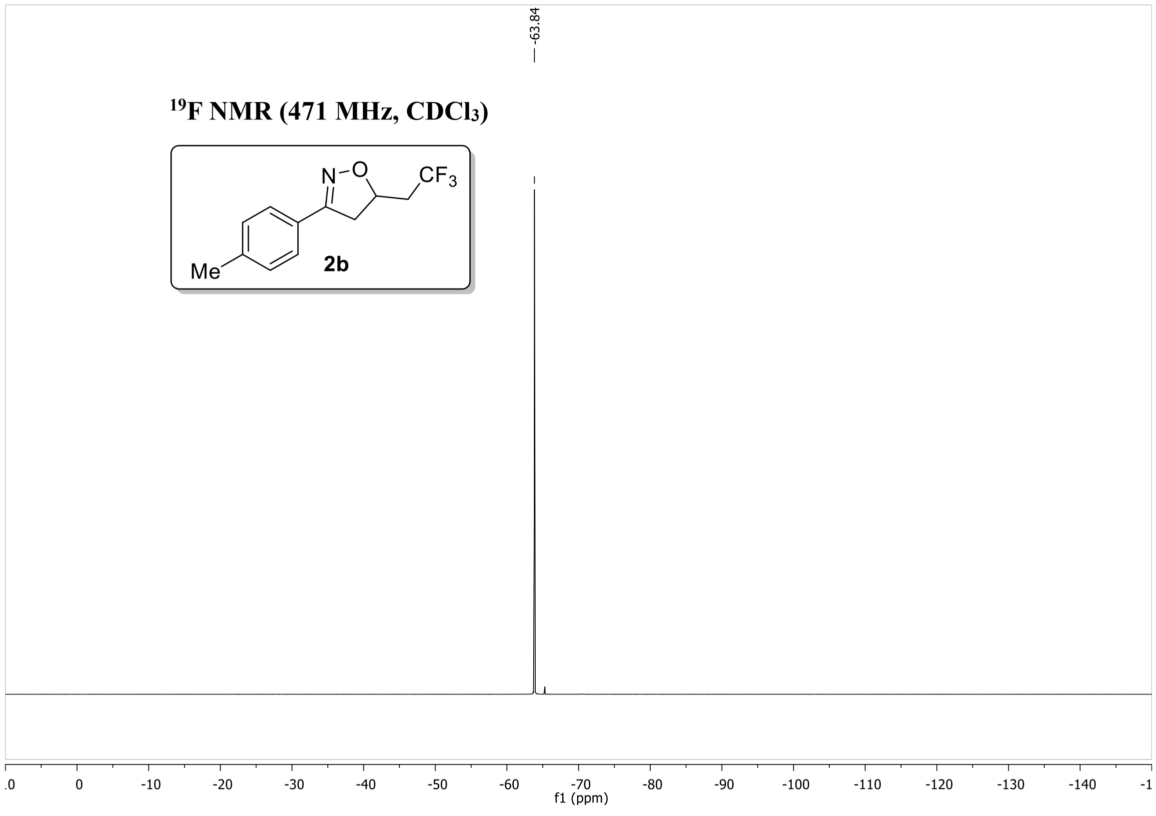




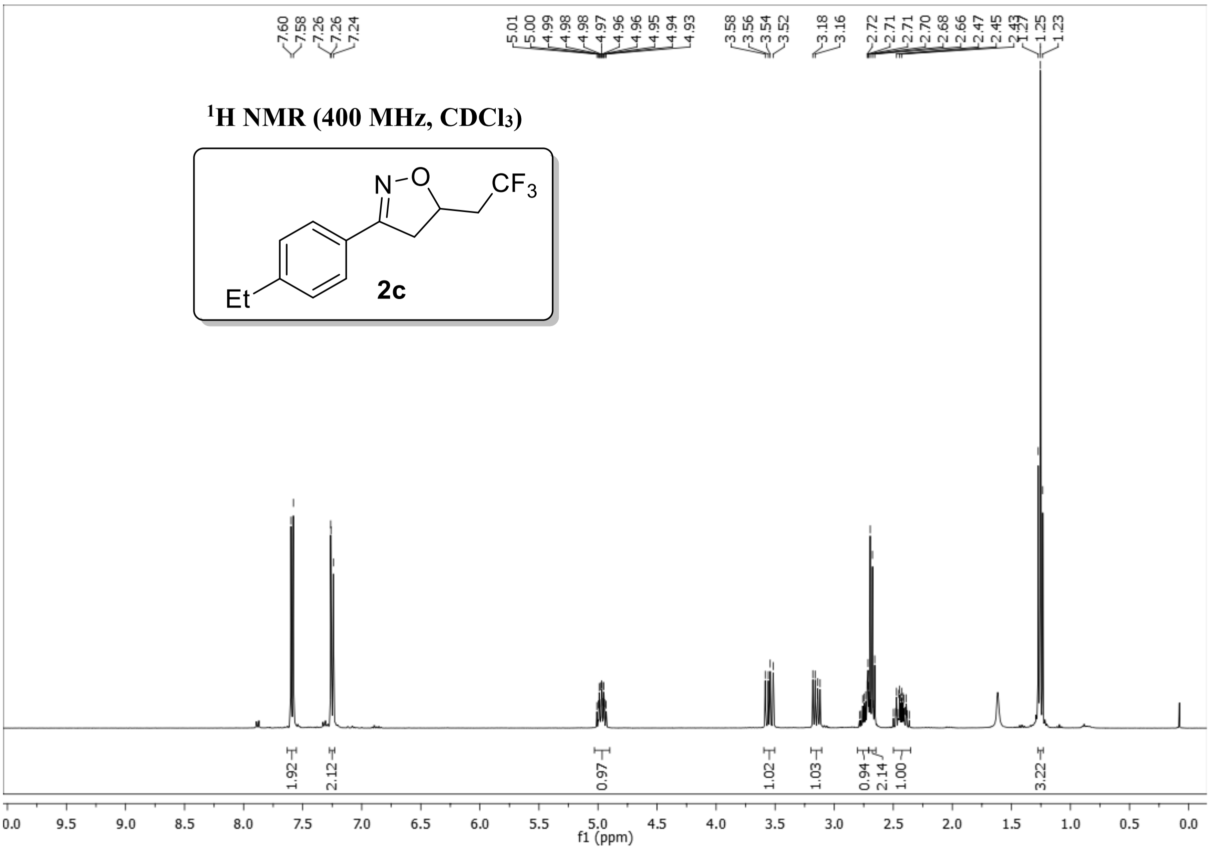

-63.83
19F NMR (471 MHz, CDCl3)
N
O
CF3
2a
.0
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-1
f1 (ppm)


¹H NMR (400 MHz, CDCl₃)
N–O
CF₃
Me
2b
7.57
7.55
7.26
7.23
7.21
5.01
4.99
4.99
4.98
4.97
4.97
4.96
4.95
4.95
4.94
4.93
3.58
3.55
3.53
3.51
3.17
3.15
3.13
2.77
2.76
2.75
2.75
2.73
2.72
2.72
2.71
2.70
2.68
2.67
2.66
2.47
2.45
2.45
2.43
2.43
2.42
2.41
2.39
2.01
2.01
1.00
1.06
1.11
1.08
1.09
3.06
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)

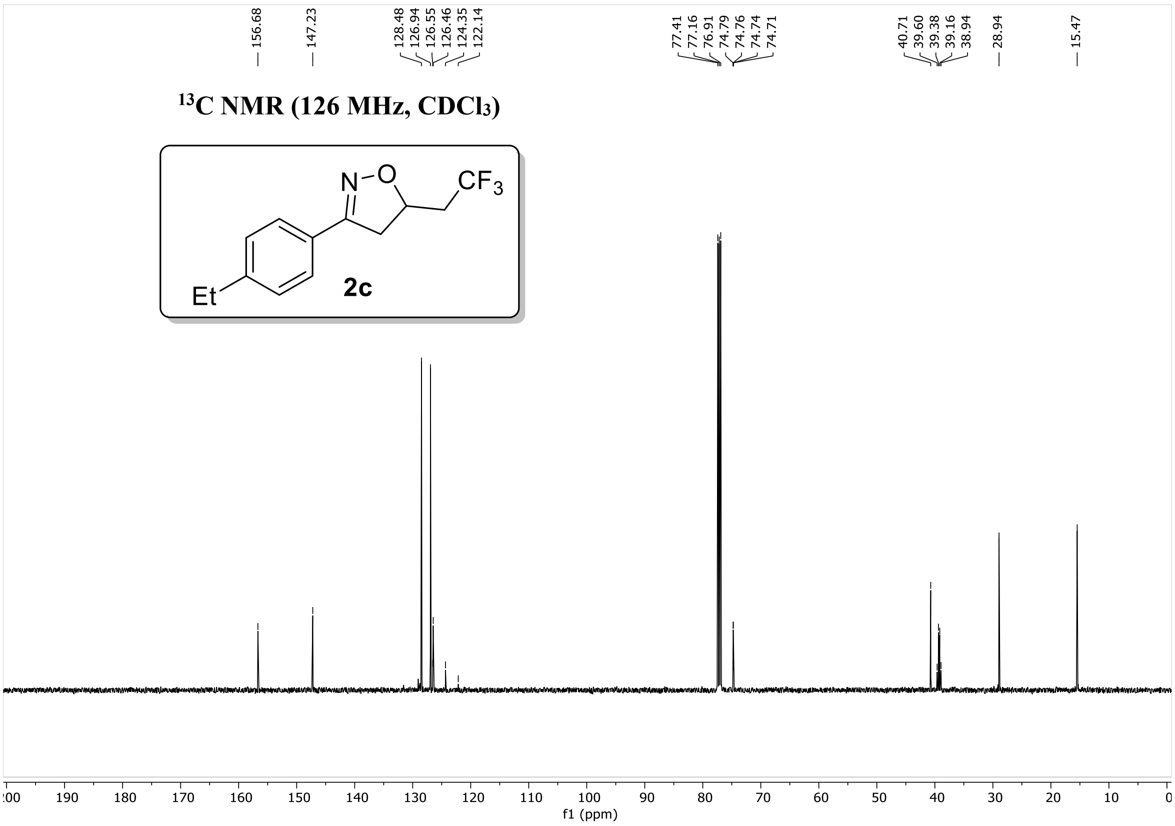

¹³C NMR (126 MHz, CDCl₃)
N–O
CF₃
Me
2b
156.69
140.93
129.66
128.76
126.84
126.56
126.27
124.36
122.15
77.41
77.16
76.91
74.80
74.77
74.75
74.73
40.72
39.62
39.40
39.18
38.96
21.59
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


^{19}F NMR (471 MHz, CDCl$_3$)

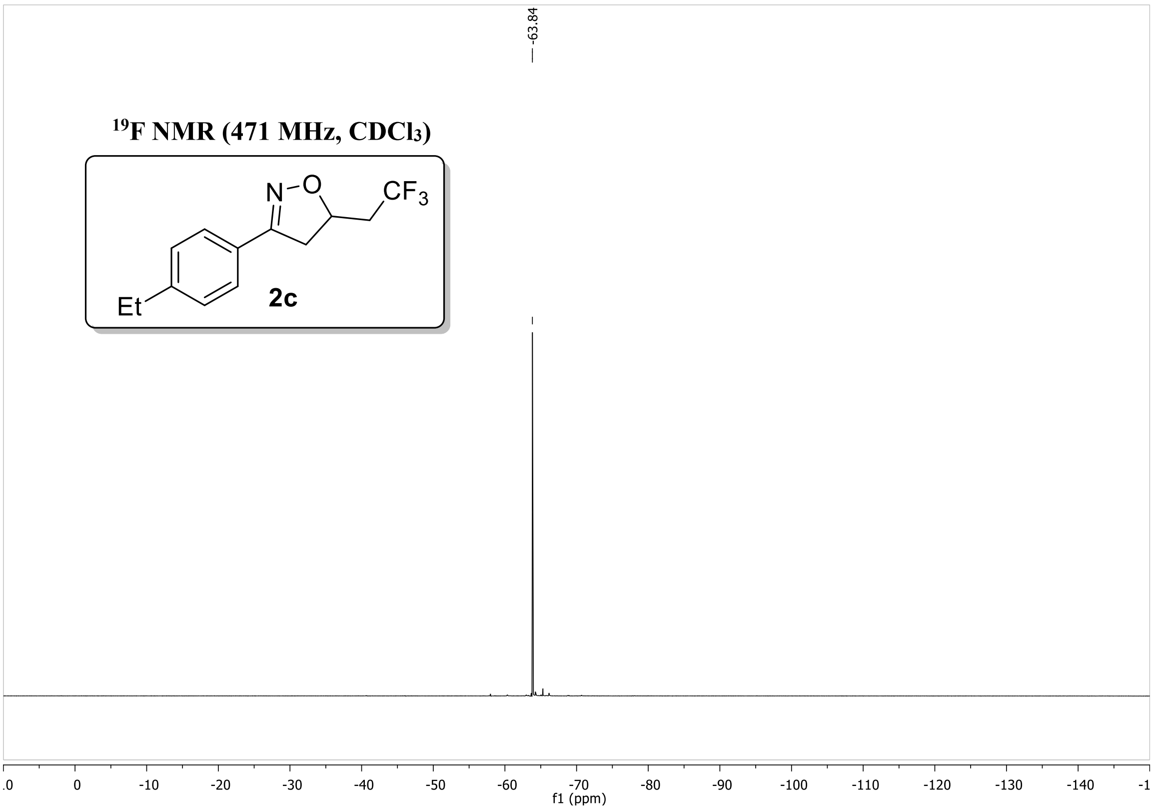
2b

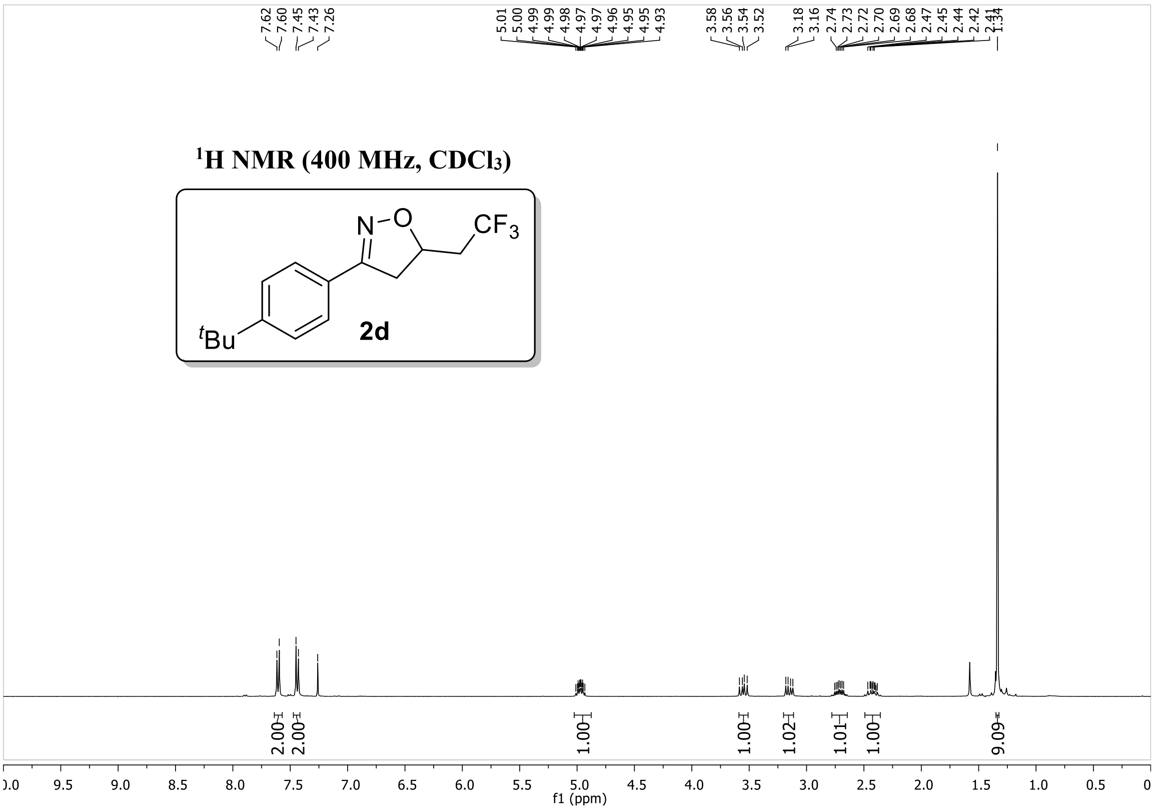


1H NMR (400 MHz, CDCl3)
N
O
CF3
Et
2c
7.60
7.58
7.26
7.26
7.24
5.01
5.00
4.99
4.98
4.98
4.97
4.96
4.96
4.95
4.94
4.93
3.58
3.56
3.54
3.52
3.18
3.16
2.72
2.71
2.71
2.70
2.68
2.66
2.47
2.45
2.43
1.27
1.25
1.23
1.92
2.12
0.97
1.02
1.03
0.94
2.14
1.00
3.22
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



13C NMR (126 MHz, CDCl3)
N
O
CF3
Et
2c
156.68
147.23
128.48
126.94
126.55
126.46
124.35
122.14
77.41
77.16
76.91
74.79
74.76
74.74
74.71
40.71
39.60
39.38
39.16
38.94
28.94
15.47
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-63.84
19F NMR (471 MHz, CDCl3)
N
O
CF3
Et
2c
.0
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-1
f1 (ppm)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4.99
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4.96
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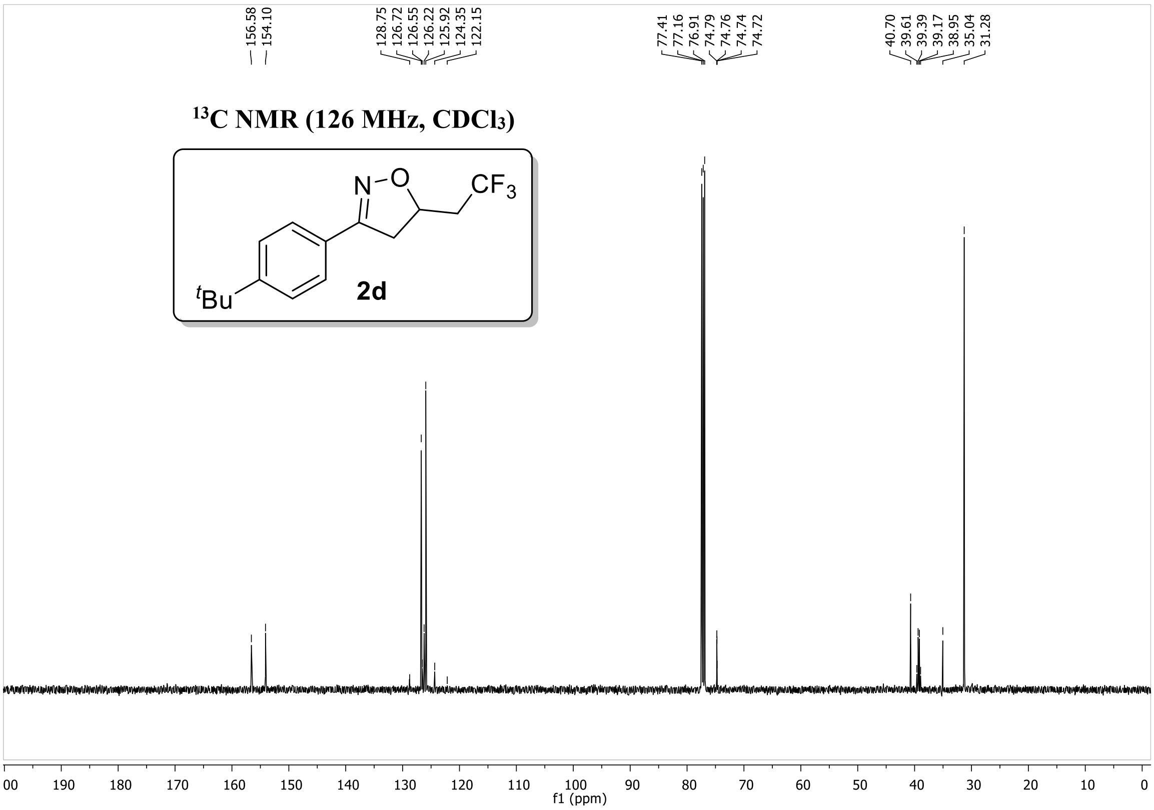
2.73
2.72
2.70
2.69
2.68
2.47
2.45
2.44
2.42
2.41
1.34
¹H NMR (400 MHz, CDCl₃)
N
O
CF3
tBu
2d
2.00
2.00
1.00
1.00
1.02
1.01
1.00
9.09
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)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126.55
126.22
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124.35
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77.41
77.16
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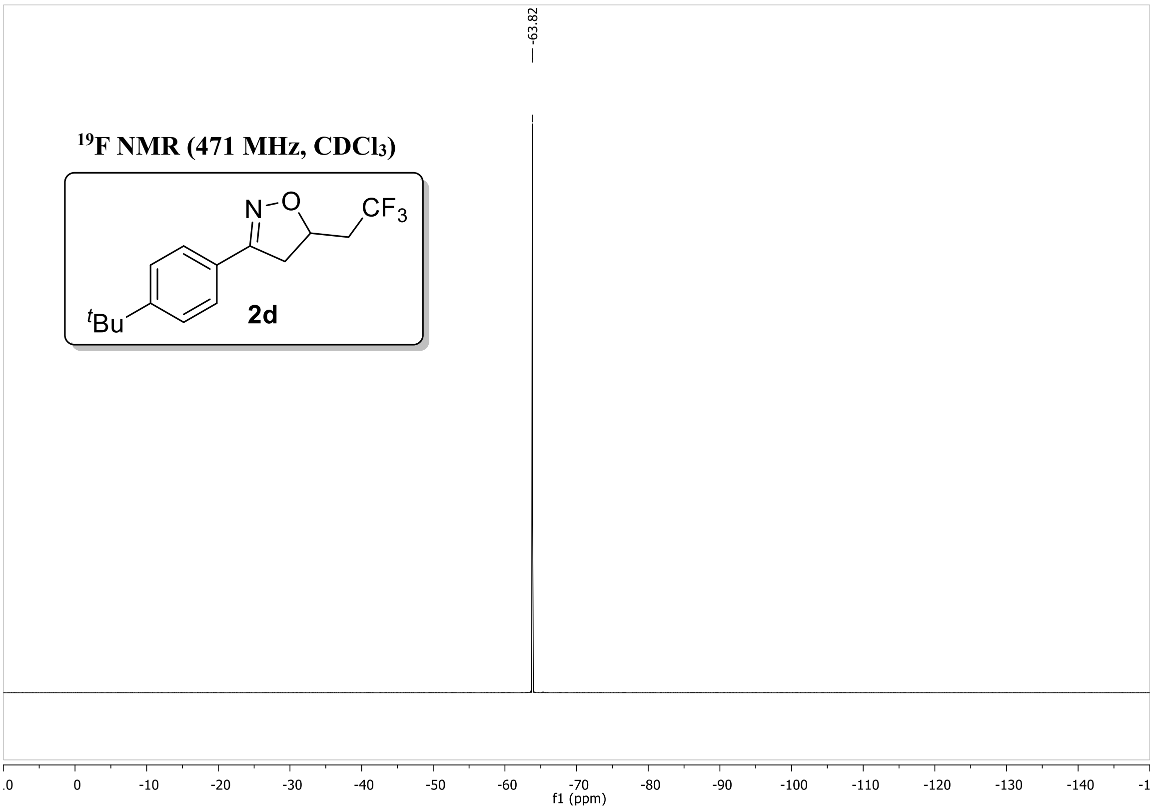
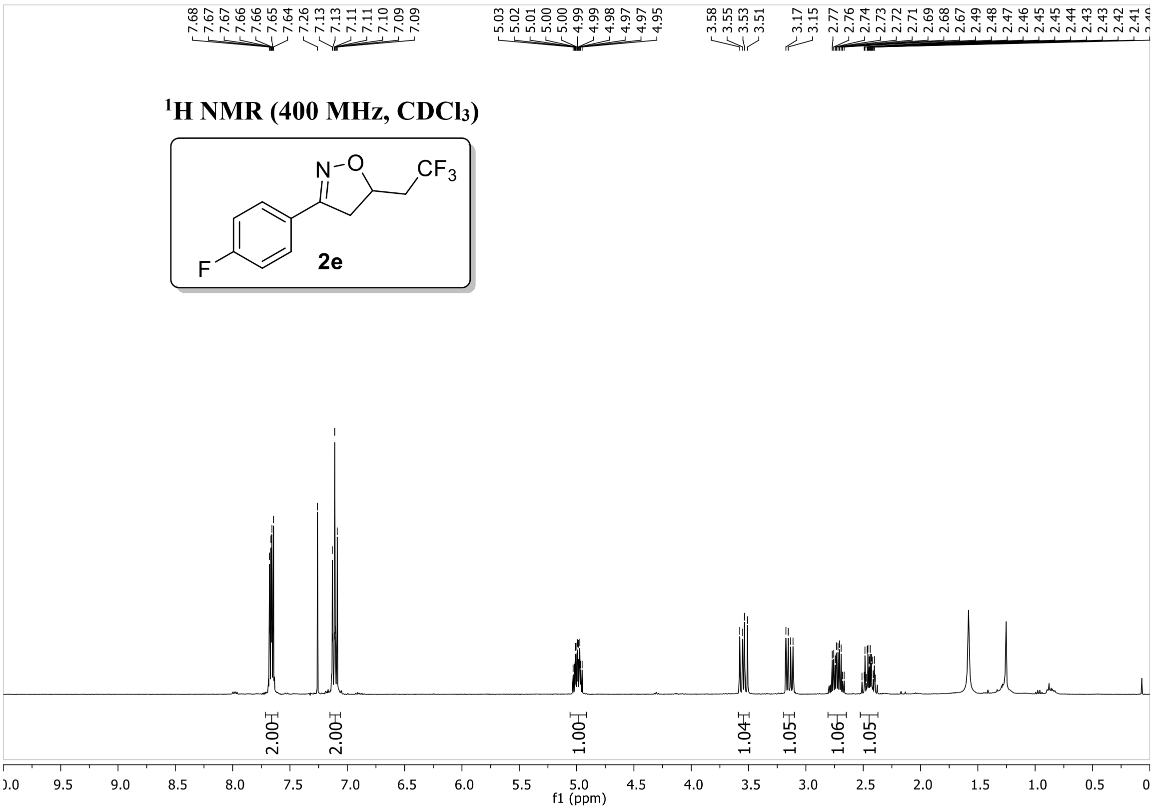
74.79
74.76
74.74
74.72
40.70
39.61
39.39
39.17
38.95
35.04
31.28
¹³C NMR (126 MHz, CDCl₃)
N
O
CF3
tBu
2d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



^{19}F NMR (471 MHz, CDCl$_3$)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7.09
7.09
5.03
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5.01
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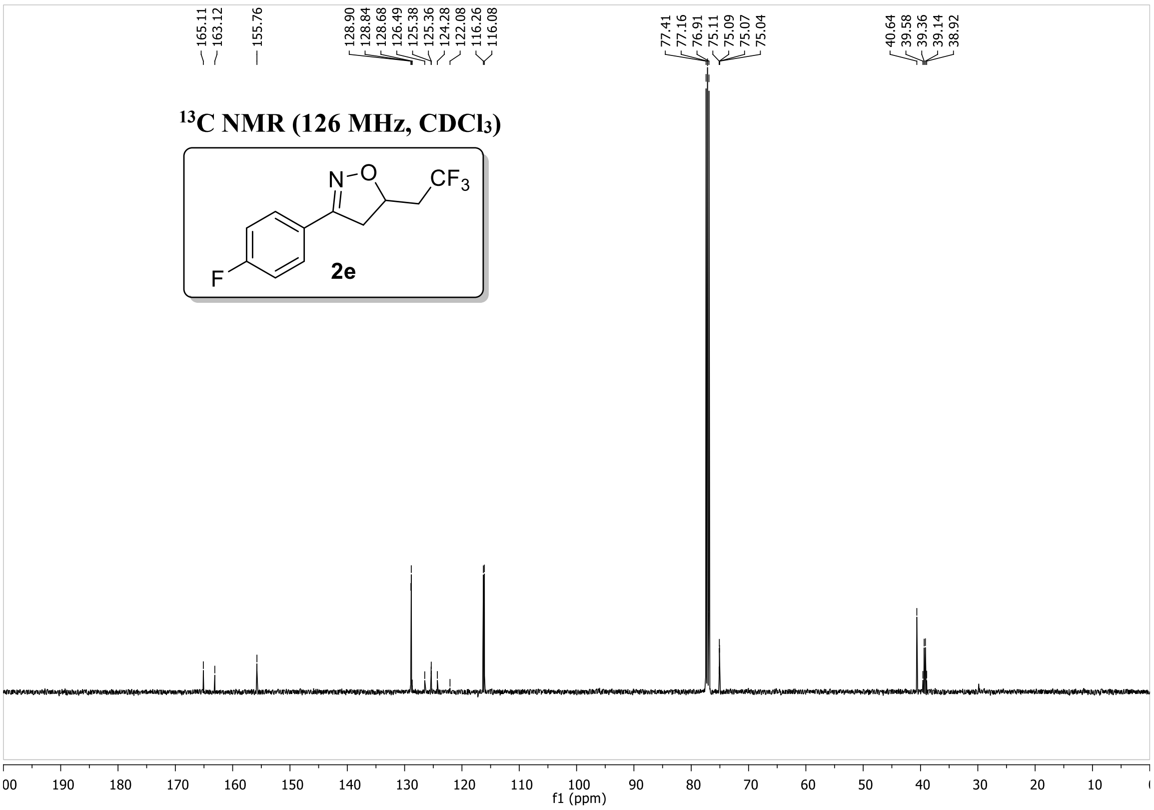
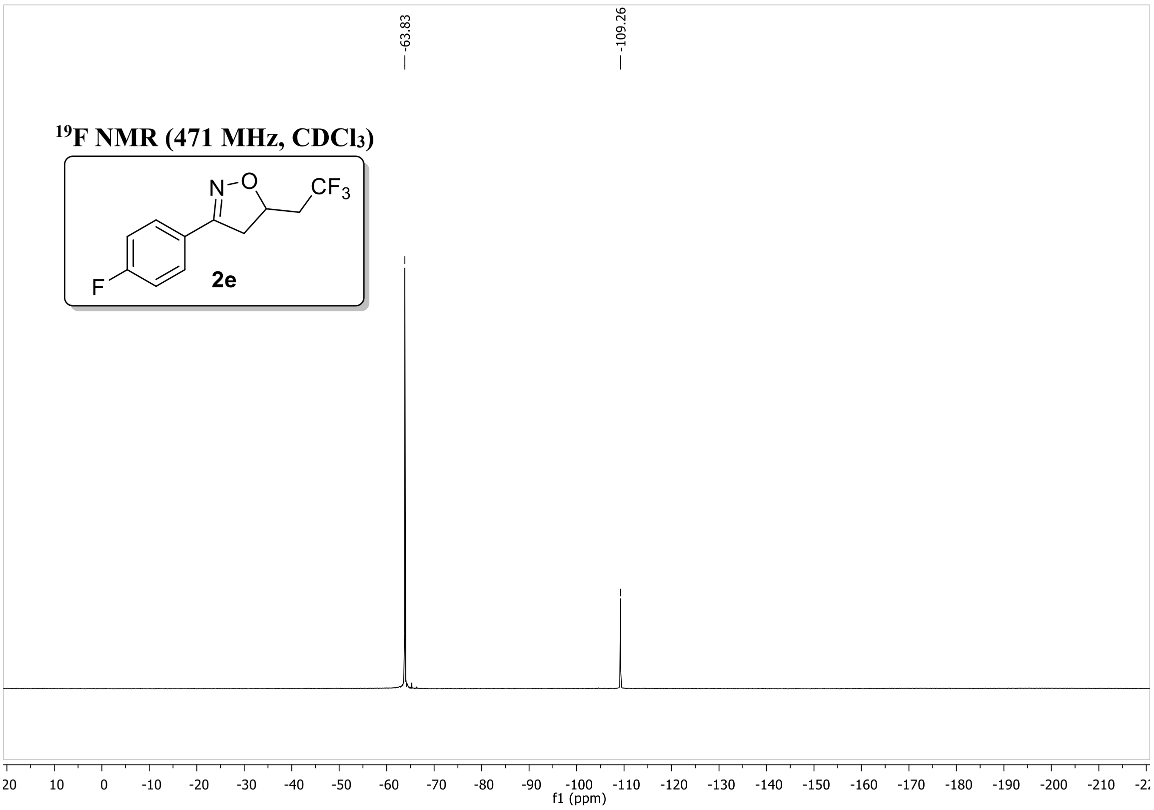
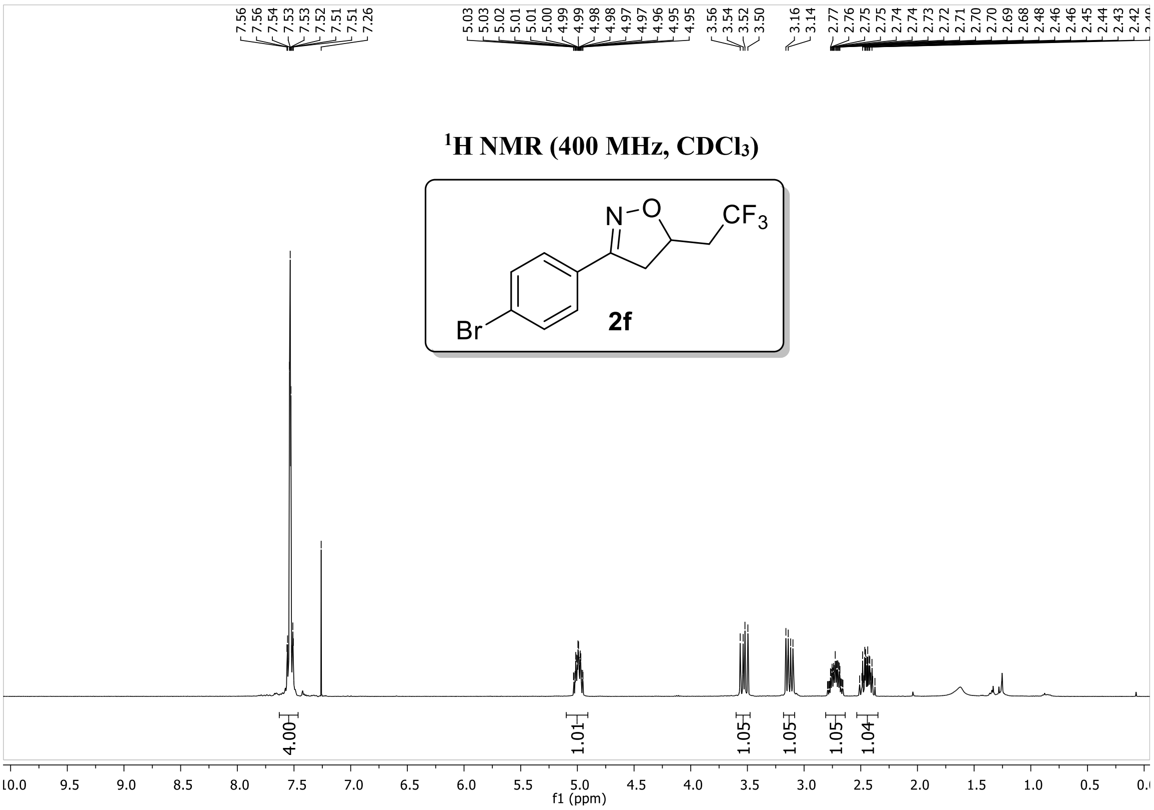
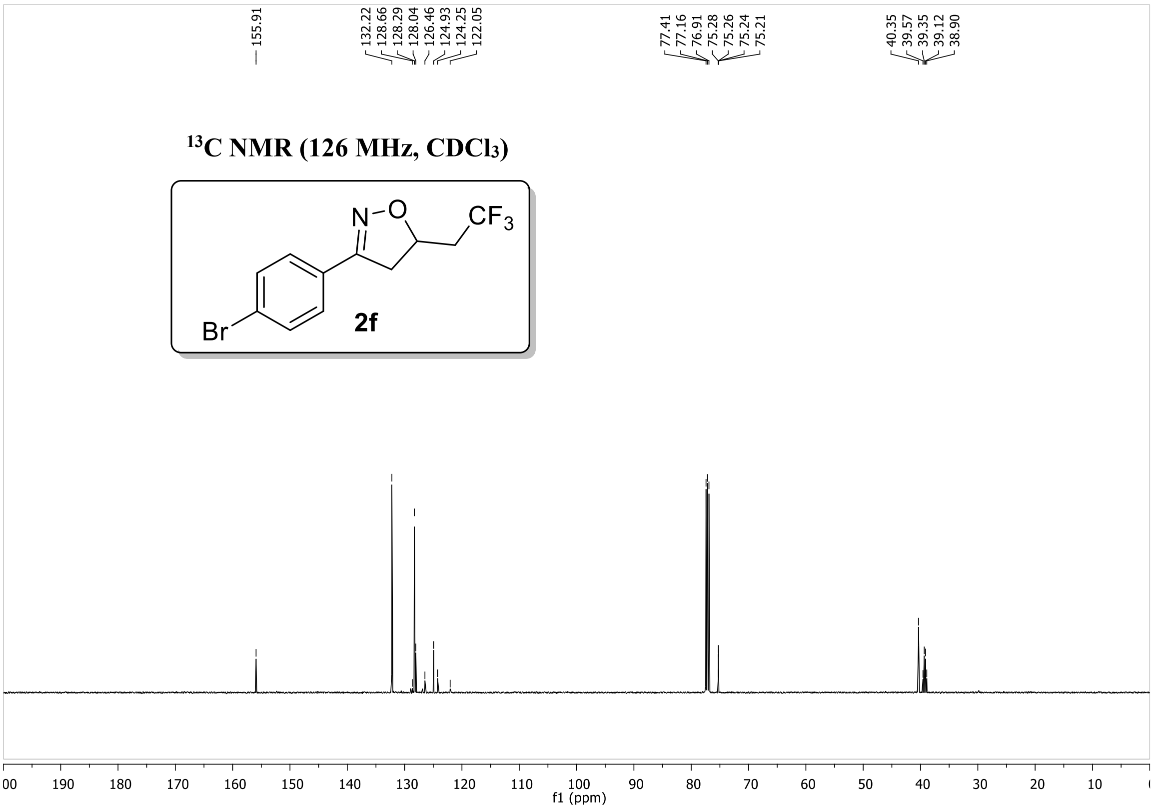
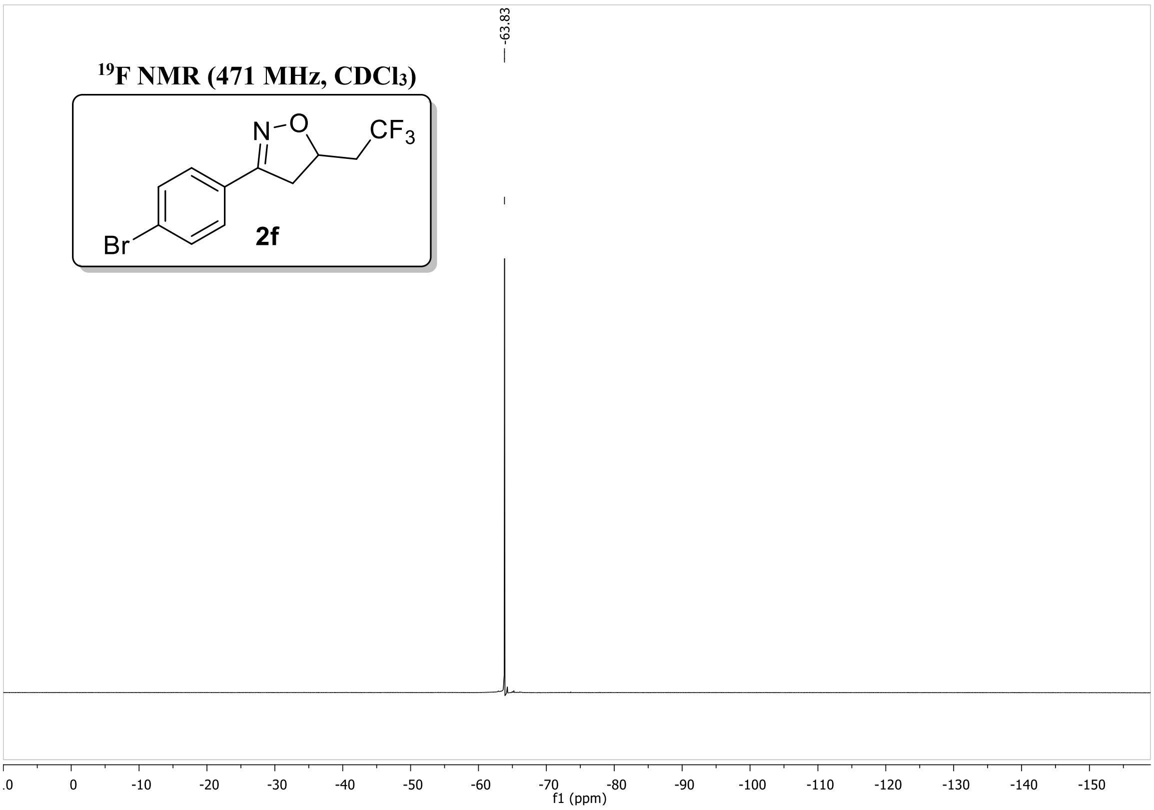
2.48
2.47
2.46
2.45
2.44
2.43
2.43
2.42
2.41
^{1}H NMR (400 MHz, CDCl$_3$)
N
O
CF$_3$
F
2e
2.00
2.00
1.00
1.04
1.05
1.06
1.05
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

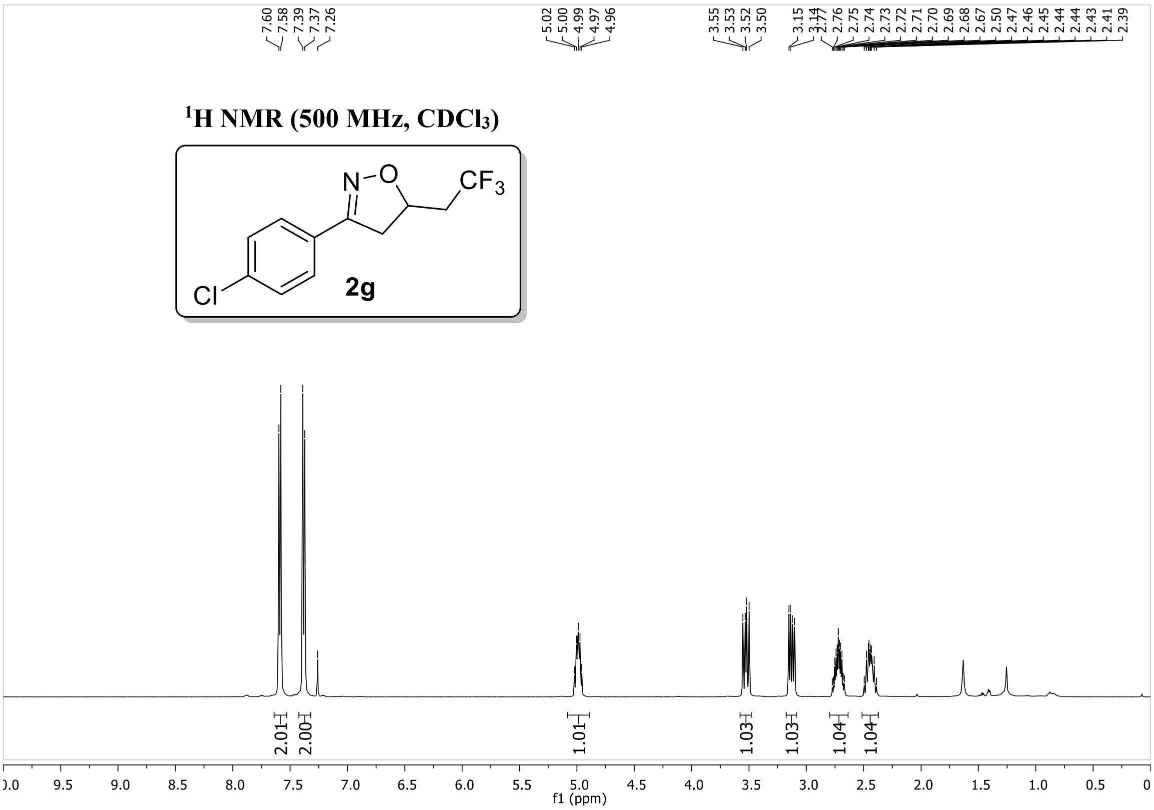

165.11
163.12
155.76
128.90
128.84
128.68
126.49
125.38
125.36
124.28
122.08
116.26
116.08
77.41
77.16
76.91
75.11
75.09
75.07
75.04
40.64
39.58
39.36
39.14
38.92
^{13}C NMR (126 MHz, CDCl$_3$)
N
O
CF$_3$
F
2e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

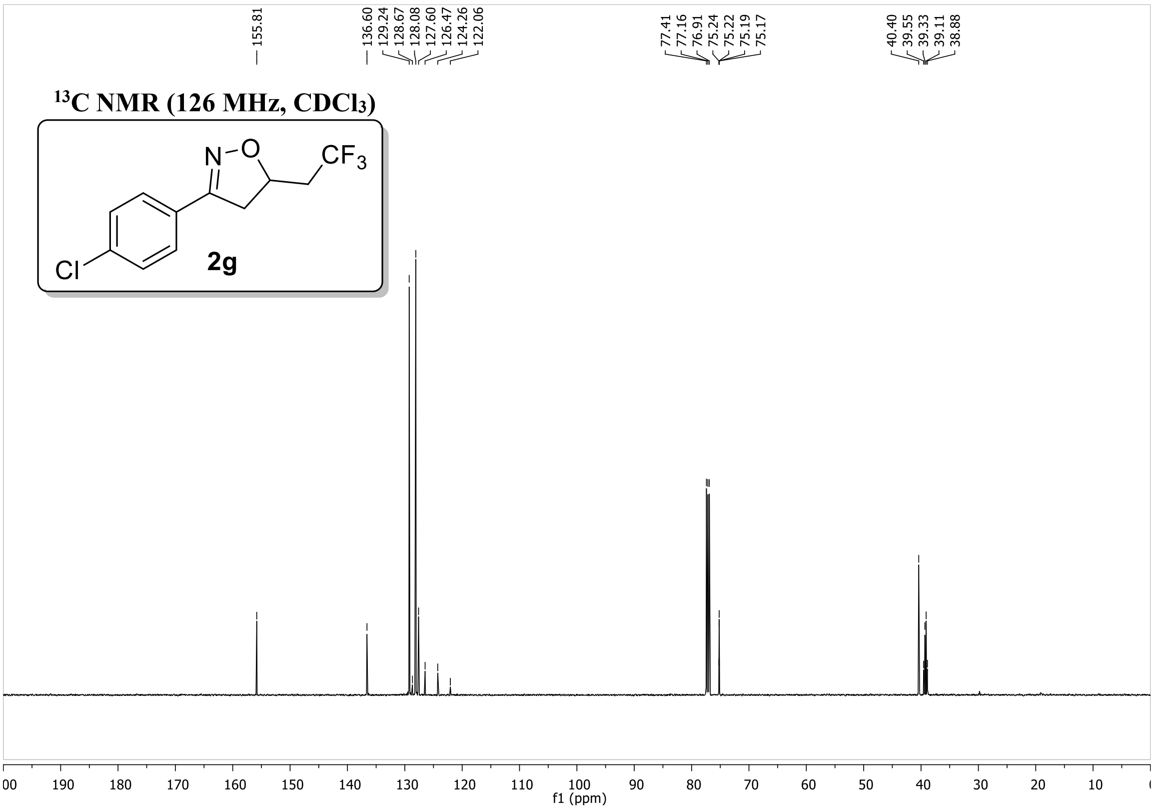

-63.83
-109.26
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
F
2e
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

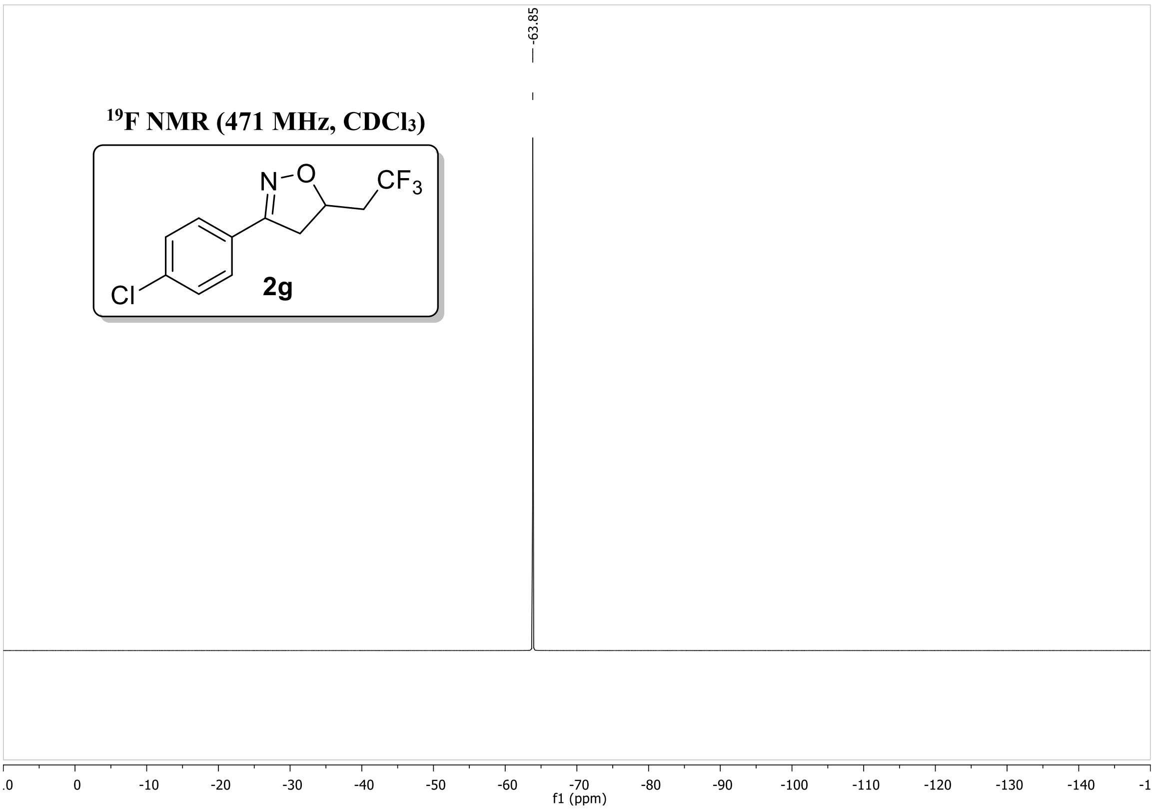

7.56
7.56
7.54
7.53
7.53
7.52
7.51
7.51
7.26
5.03
5.03
5.02
5.01
5.01
5.00
4.99
4.99
4.98
4.98
4.97
4.97
4.96
4.95
4.95
3.56
3.54
3.52
3.50
3.16
3.14
2.77
2.76
2.75
2.75
2.74
2.74
2.73
2.72
2.71
2.70
2.70
2.69
2.68
2.48
2.46
2.46
2.45
2.44
2.43
2.42
¹H NMR (400 MHz, CDCl₃)
N–O
CF₃
Br
2f
4.00
1.01
1.05
1.05
1.05
1.04
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
f1 (ppm)

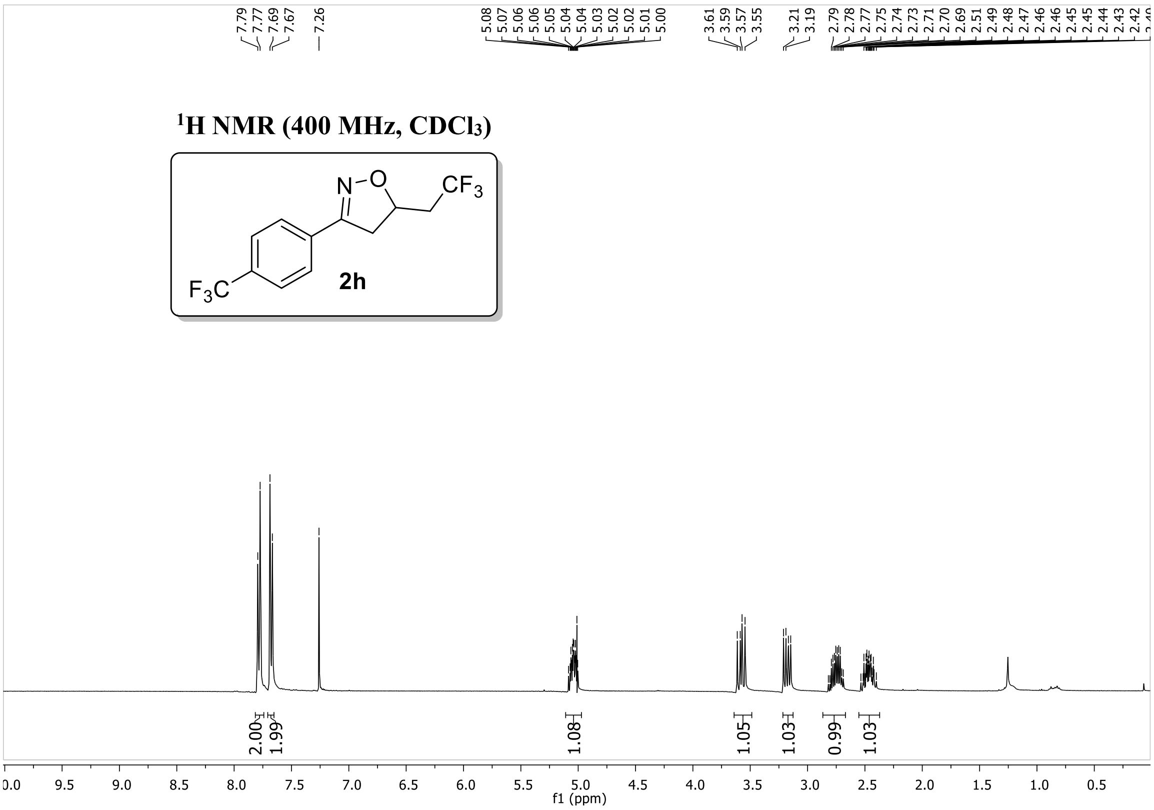

155.91
132.22
128.66
128.29
128.04
126.46
124.93
124.25
122.05
77.41
77.16
76.91
75.28
75.26
75.24
75.21
40.35
39.57
39.35
39.12
38.90
¹³C NMR (126 MHz, CDCl₃)
N–O
CF₃
Br
2f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

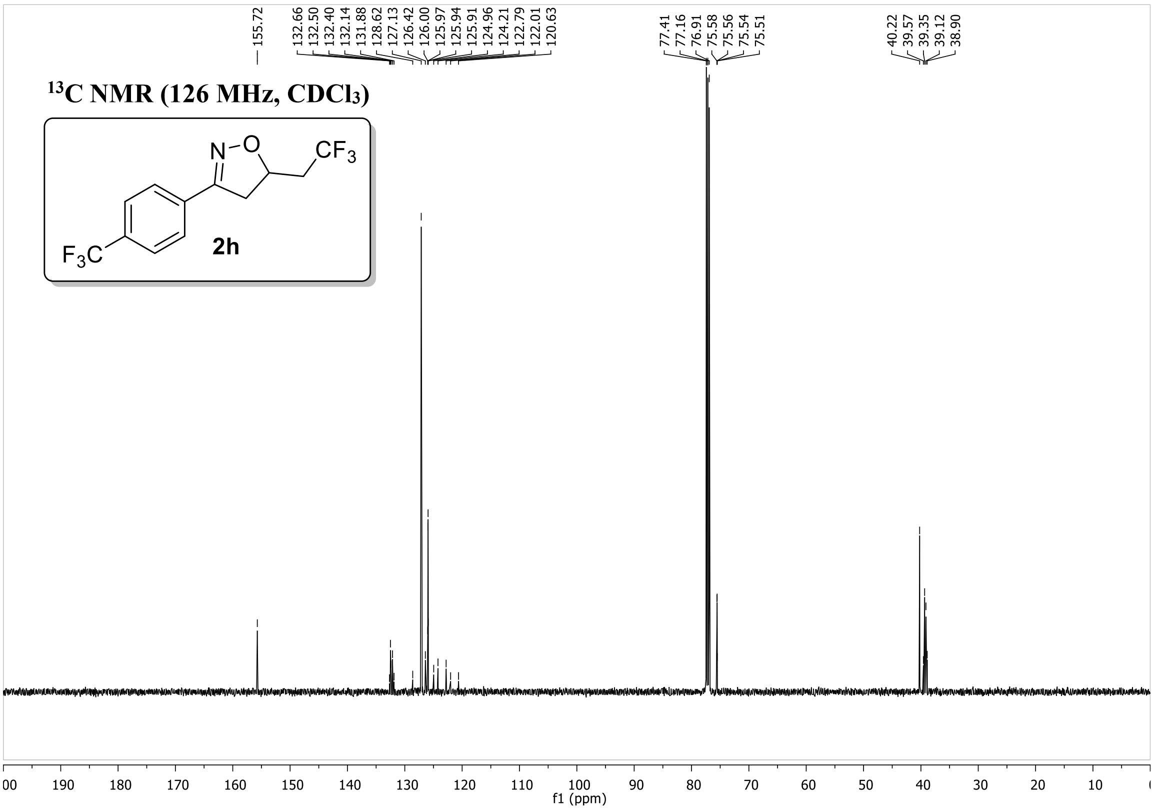

-63.83
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
Br
2f
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)

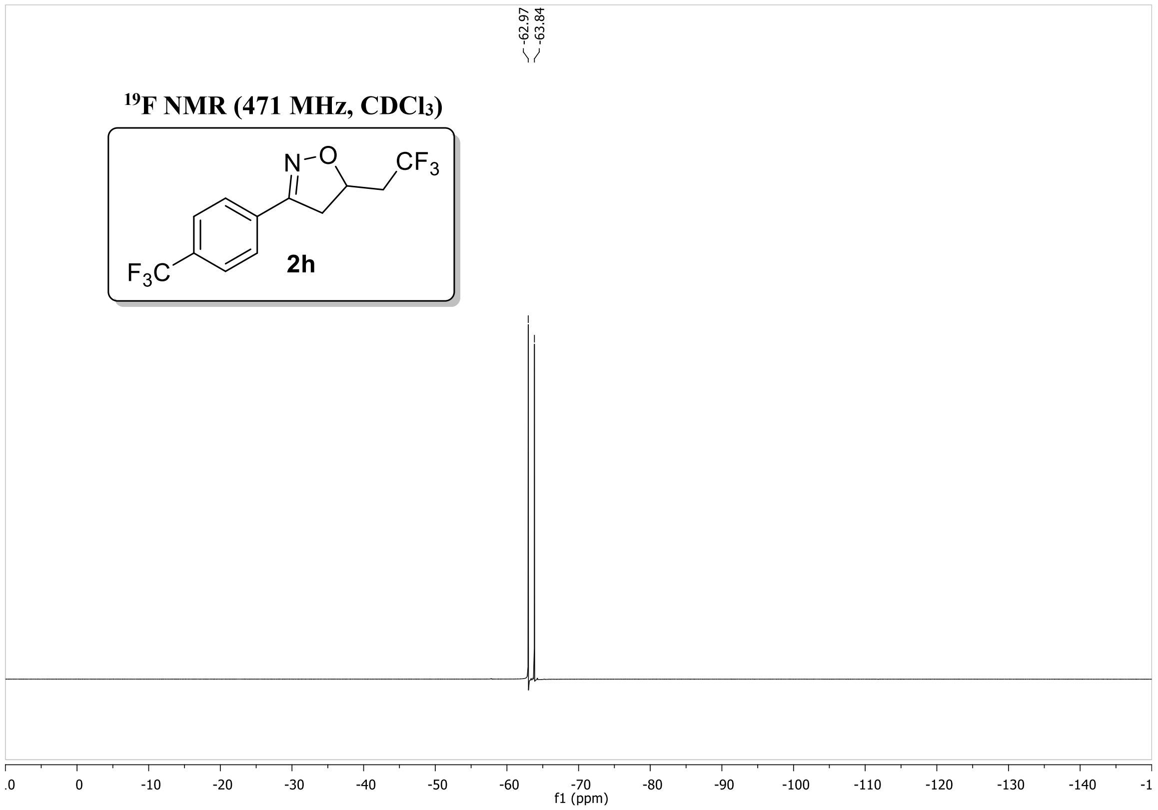

7.60
7.58
7.39
7.37
7.26
5.02
5.00
4.99
4.97
4.96
3.55
3.53
3.52
3.50
3.15
3.14
2.77
2.76
2.75
2.74
2.73
2.72
2.71
2.70
2.69
2.68
2.67
2.50
2.47
2.46
2.45
2.44
2.44
2.43
2.41
2.39
¹H NMR (500 MHz, CDCl₃)
N–O
CF₃
Cl
2g
2.01
2.00
1.01
1.03
1.03
1.04
1.04
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

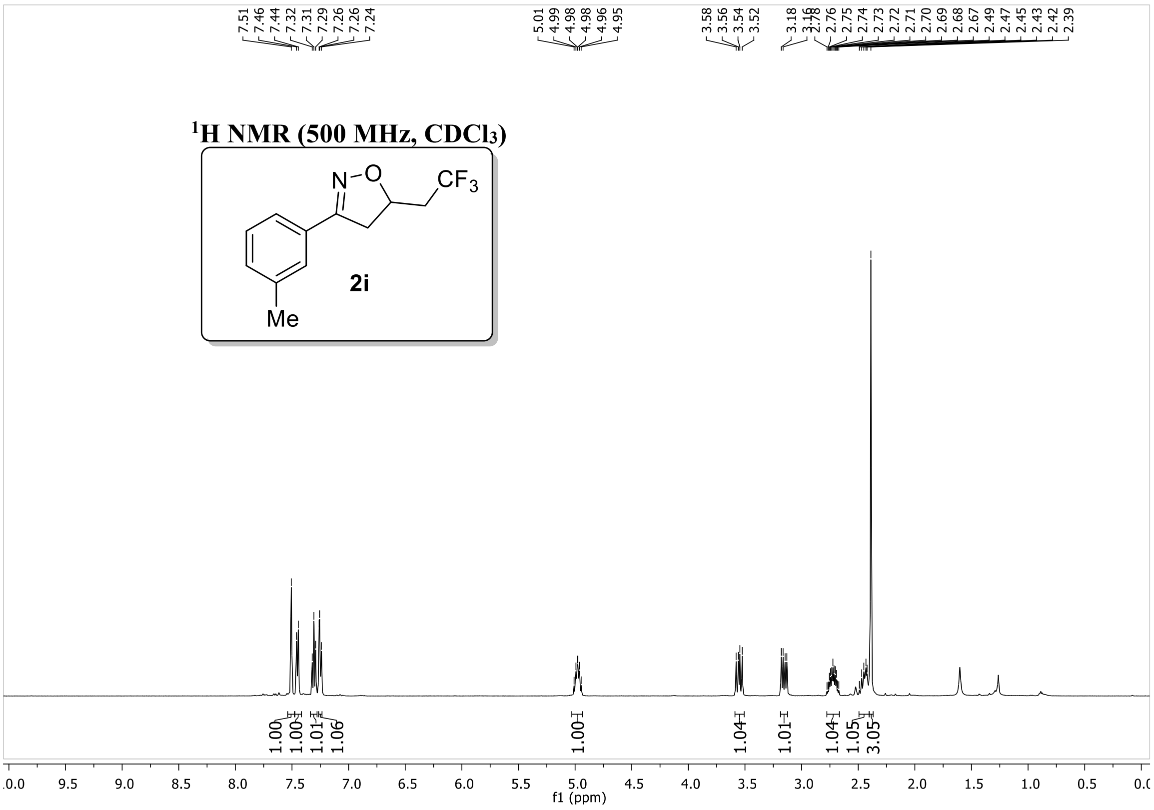

155.81
136.60
129.24
128.67
128.08
127.60
126.47
124.26
122.06
77.41
77.16
76.91
75.24
75.22
75.19
75.17
40.40
39.55
39.33
39.11
38.88
¹³C NMR (126 MHz, CDCl₃)
N–O
CF₃
Cl
2g
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


-63.85
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
Cl
2g
.0
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-1
f1 (ppm)


7.79
7.77
7.69
7.67
7.26
5.08
5.07
5.06
5.06
5.05
5.04
5.04
5.03
5.02
5.01
5.00
3.61
3.59
3.57
3.55
3.21
3.19
2.79
2.78
2.77
2.75
2.74
2.73
2.71
2.70
2.69
2.51
2.49
2.48
2.47
2.46
2.46
2.45
2.45
2.44
2.43
2.42
^{1}H NMR (400 MHz, CDCl$_3$)
N
O
CF$_3$
F$_3$C
2h
2.00
1.99
1.08
1.05
1.03
0.99
1.03
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)

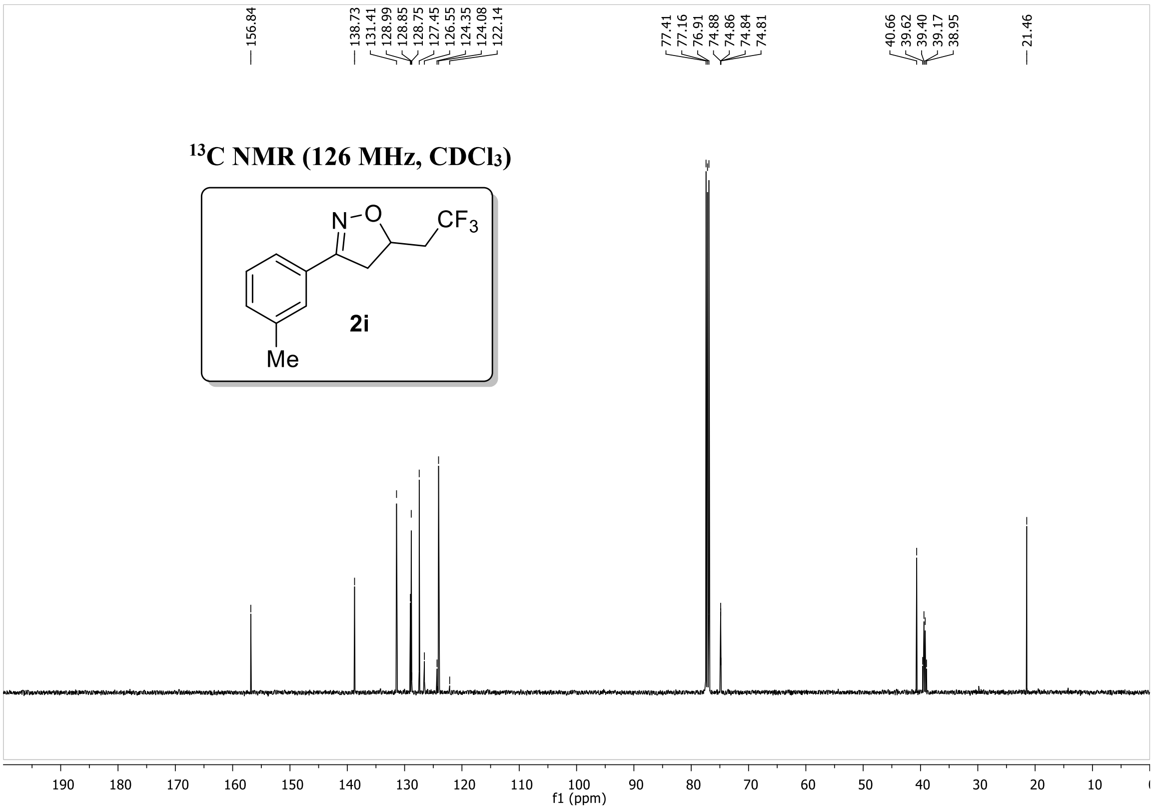

155.72
132.66
132.50
132.40
132.14
131.88
128.62
127.13
126.42
126.00
125.97
125.94
125.91
124.96
124.21
122.79
122.01
120.63
77.41
77.16
76.91
75.58
75.56
75.54
75.51
40.22
39.57
39.35
39.12
38.90
^{13}C NMR (126 MHz, CDCl$_3$)
N
O
CF$_3$
F$_3$C
2h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


-62.97
-63.84
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
F$_3$C
2h
.0
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-1
f1 (ppm)



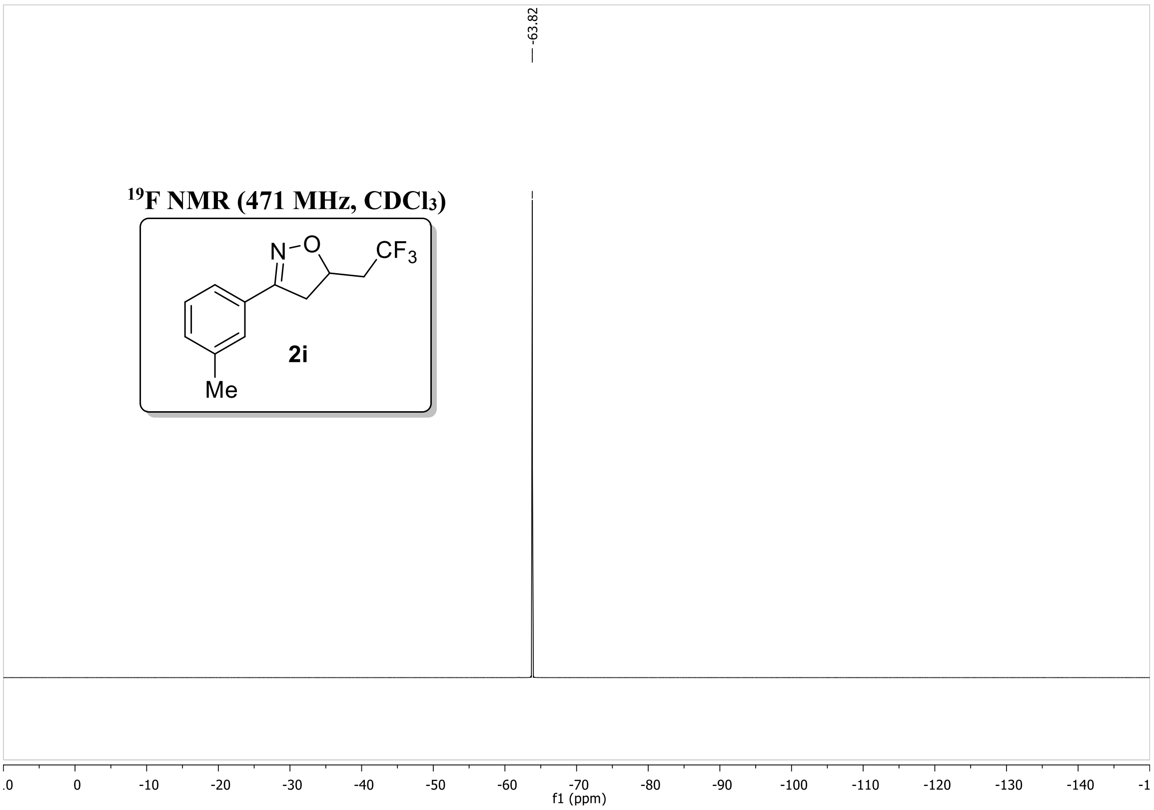
^{1}H NMR (500 MHz, CDCl$_3$)

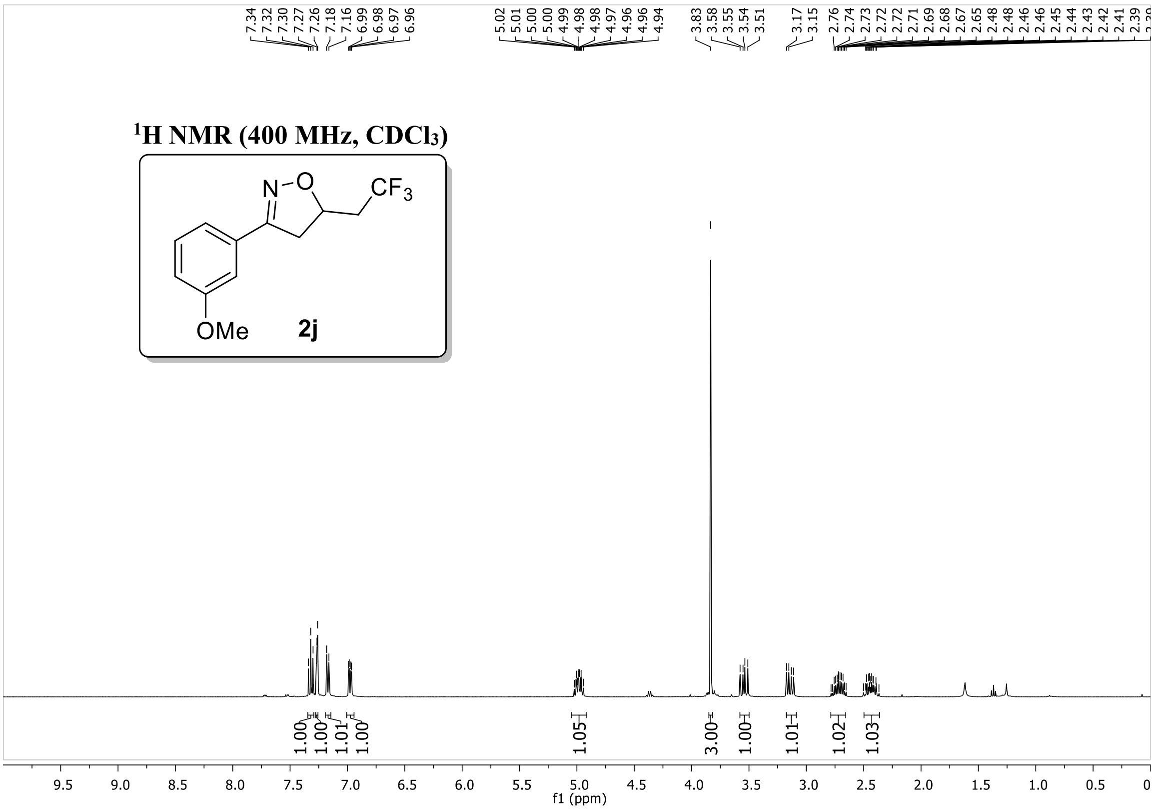
2i

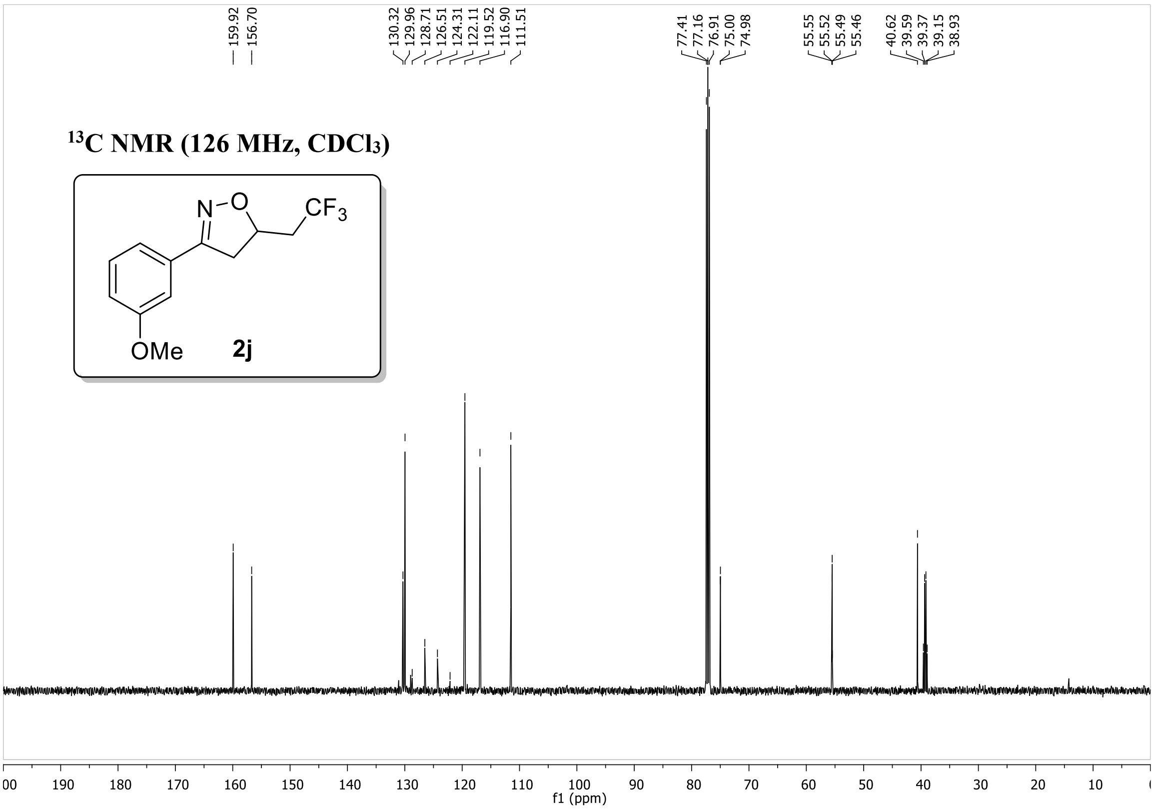


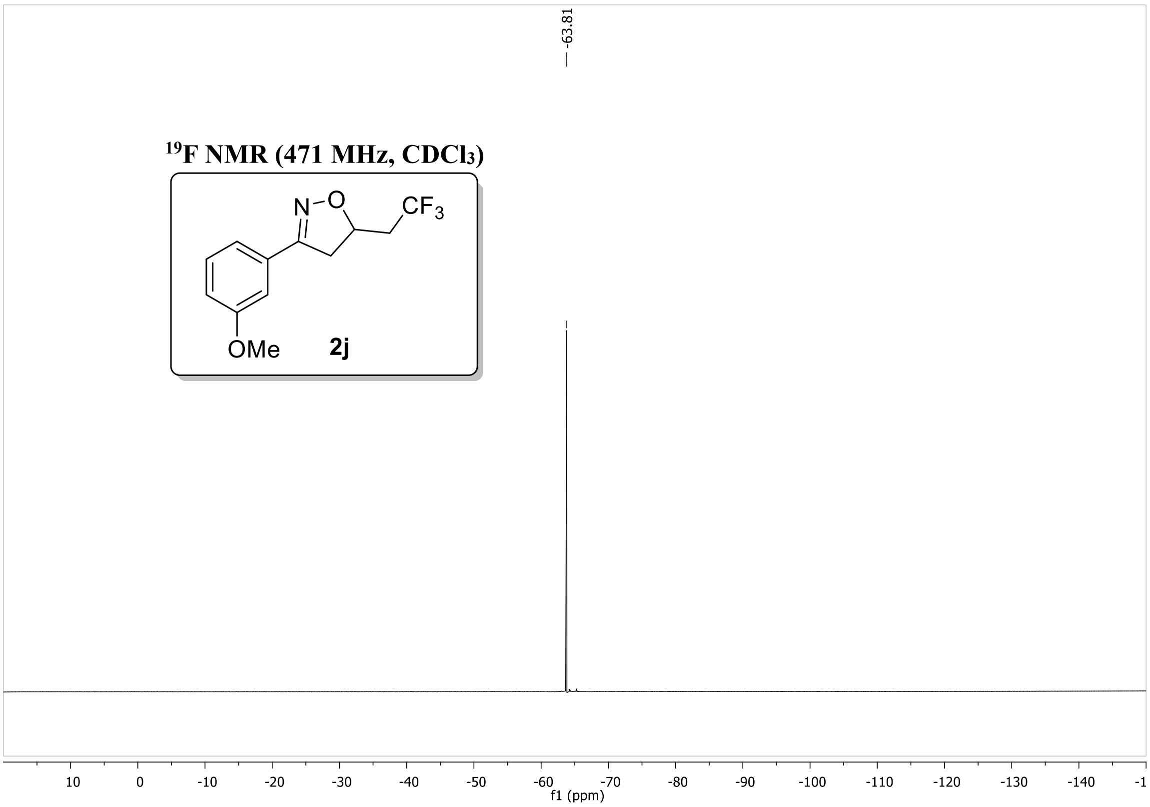
^{13}C NMR (126 MHz, CDCl$_3$)

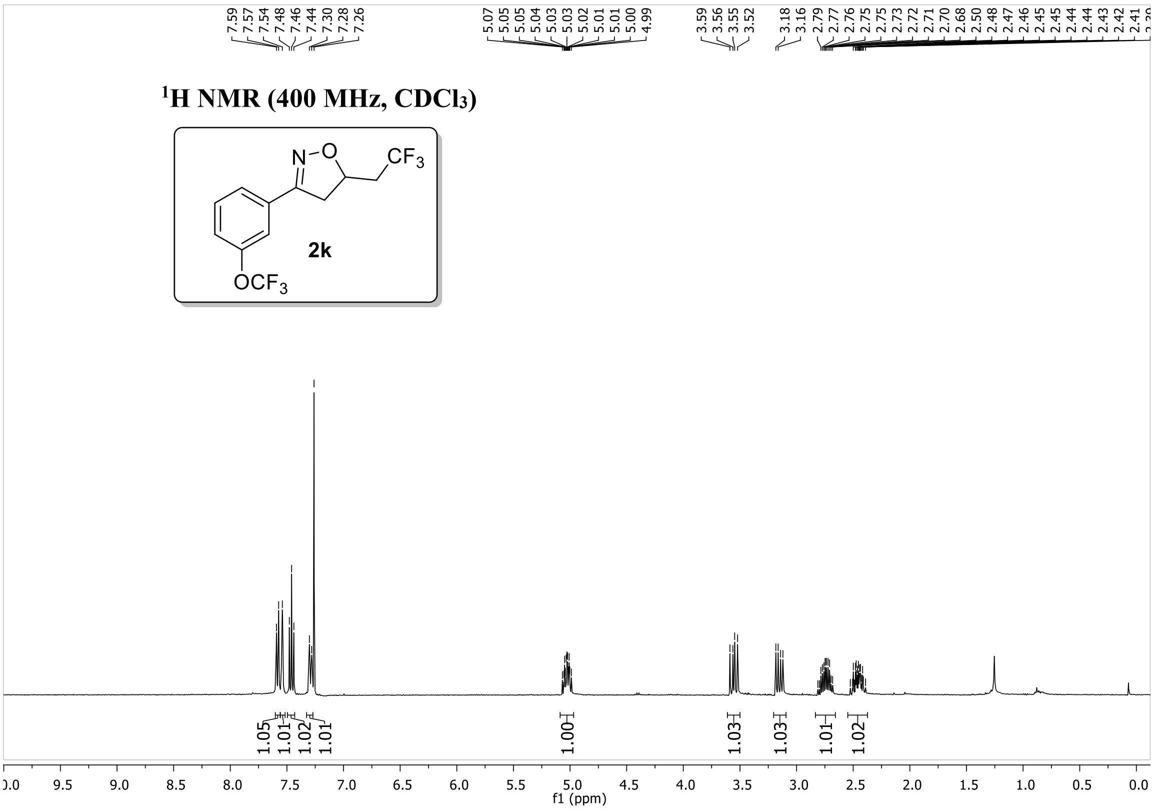
2i

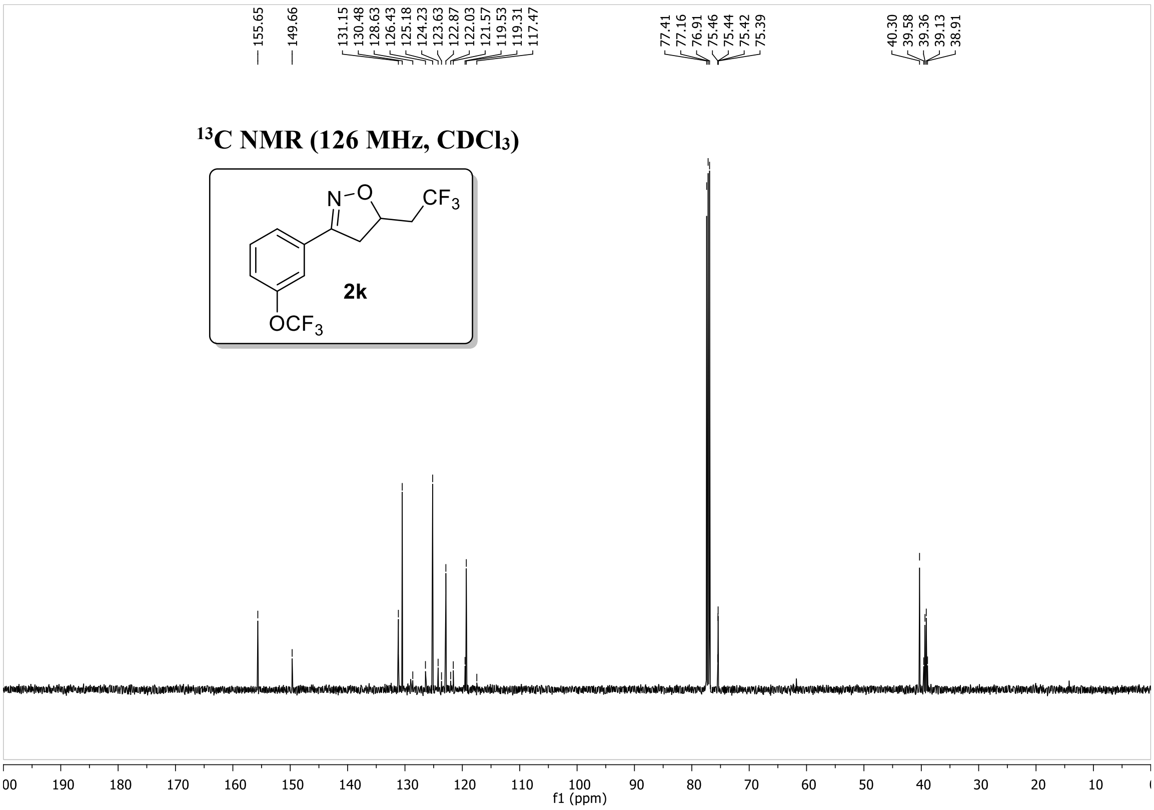

-63.82
19F NMR (471 MHz, CDCl3)
N
O
CF3
2i
Me
.0
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-1
f1 (ppm)

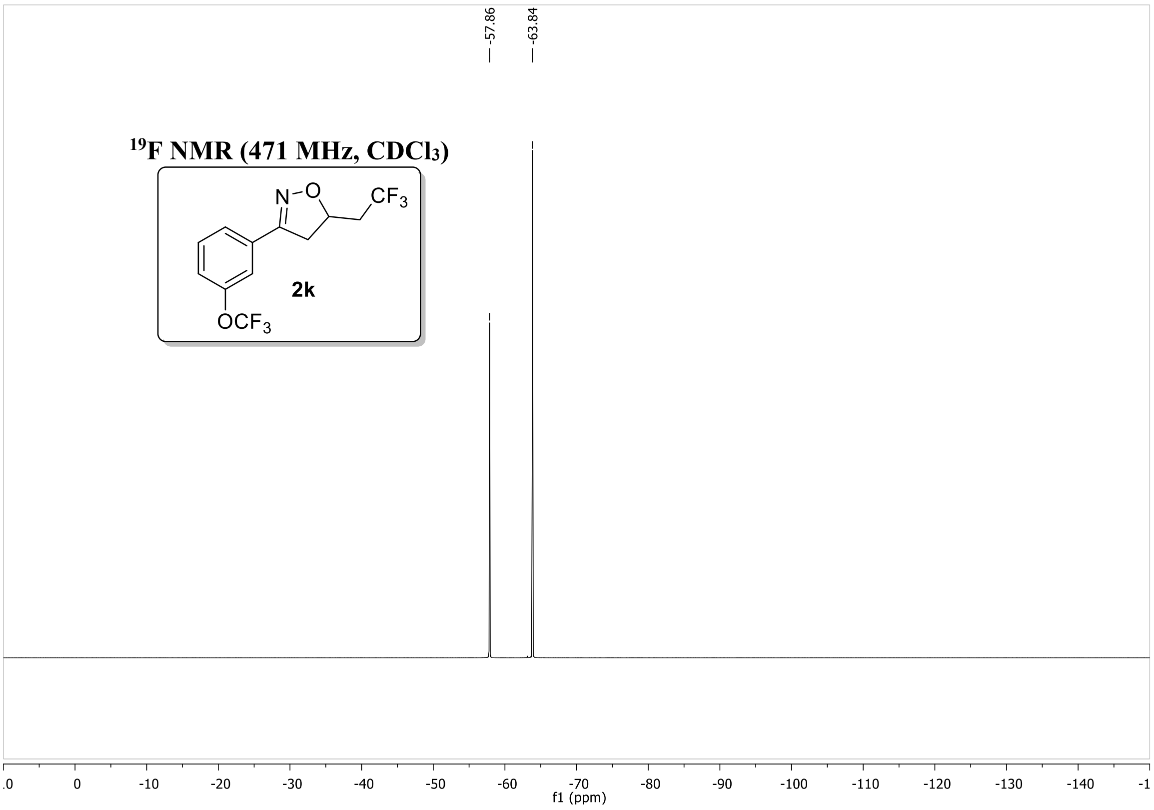

1H NMR (400 MHz, CDCl3)
N–O
CF3
OMe
2j
7.34 7.32 7.30 7.27 7.26 7.18 7.16 6.99 6.98 6.97 6.96
5.02 5.01 5.00 4.99 4.98 4.98 4.97 4.96 4.96 4.94
3.83 3.58 3.55 3.54 3.51
3.17 3.15
2.76 2.74 2.73 2.72 2.72 2.71 2.69 2.68 2.67 2.65 2.48 2.48 2.46 2.45 2.44 2.43 2.42 2.41 2.39
1.00 1.00 1.01 1.00
1.05
3.00
1.00
1.01
1.02
1.03
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
f1 (ppm)

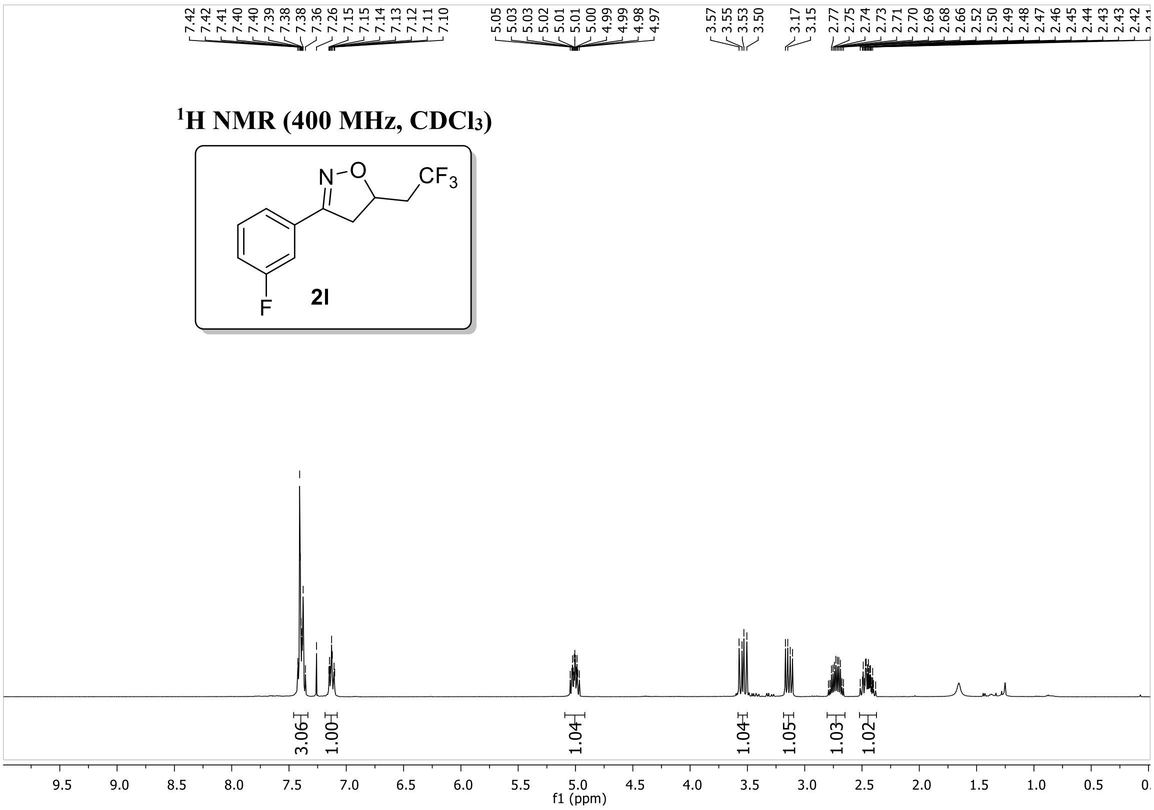

13C NMR (126 MHz, CDCl3)
N–O
CF3
OMe
2j
159.92 156.70
130.32 129.96 128.71 126.51 124.31 122.11 119.52 116.90 111.51
77.41 77.16 76.91 75.00 74.98
55.55 55.52 55.49 55.46
40.62 39.59 39.37 39.15 38.93
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

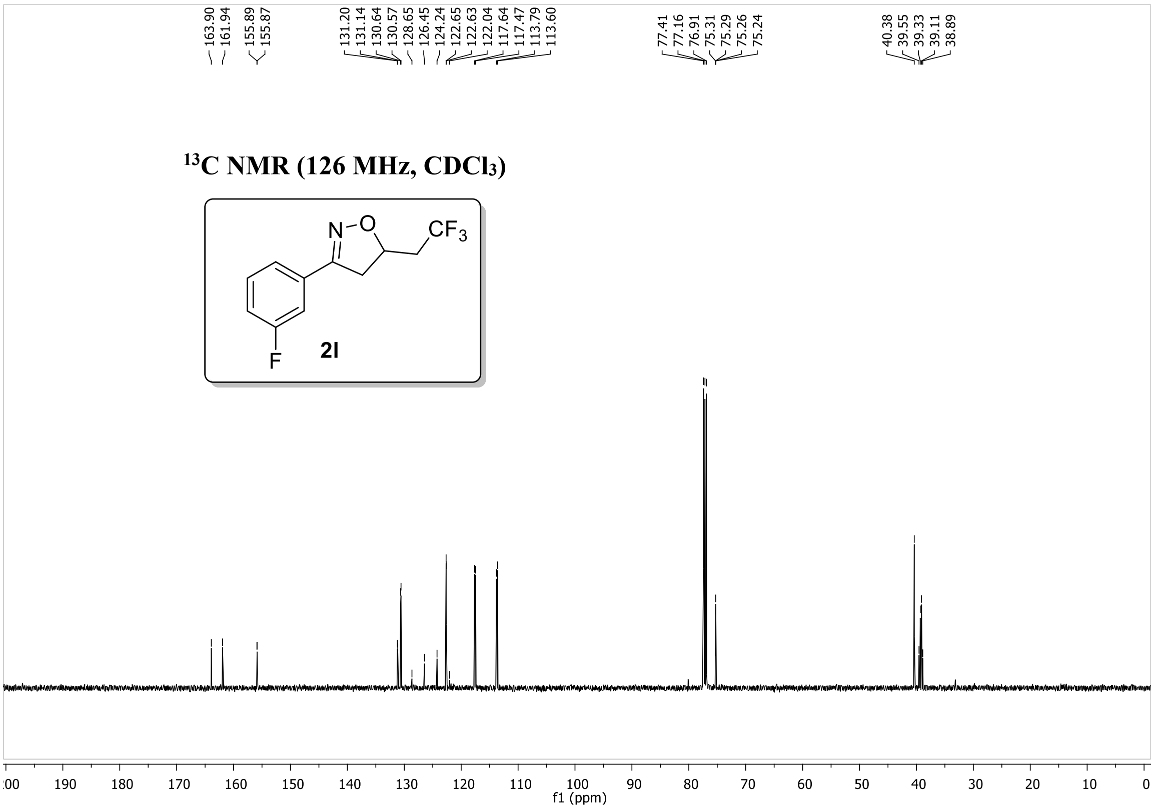

-63.81
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
OMe
2j
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-1
f1 (ppm)

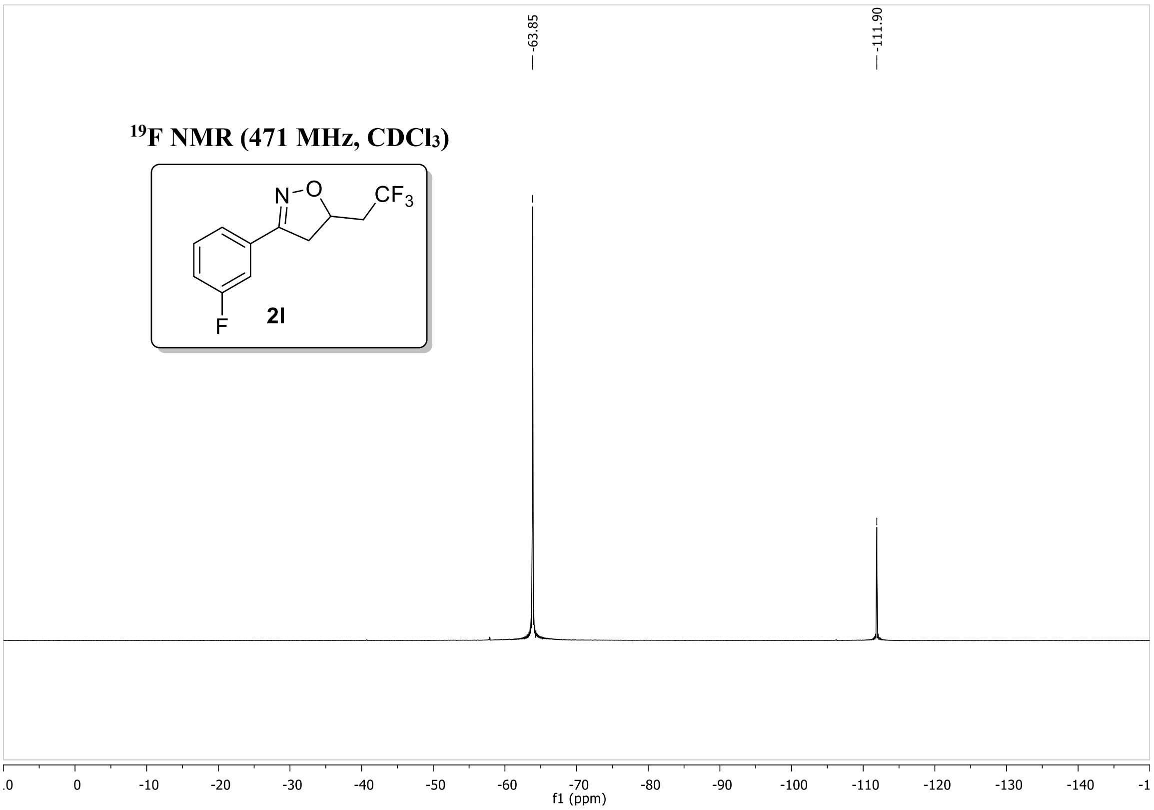

7.59
7.57
7.54
7.48
7.46
7.44
7.30
7.28
7.26
5.07
5.05
5.04
5.03
5.03
5.02
5.01
5.01
5.00
4.99
3.59
3.56
3.55
3.52
3.18
3.16
2.79
2.77
2.76
2.75
2.75
2.73
2.72
2.71
2.70
2.68
2.50
2.48
2.47
2.46
2.45
2.44
2.44
2.43
2.42
2.41
^{1}H NMR (400 MHz, CDCl$_3$)
N–O
CF$_3$
OCF$_3$
2k
1.05
1.01
1.02
1.01
1.00
1.03
1.03
1.01
1.02
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)


155.65
149.66
131.15
130.48
128.63
126.43
125.18
124.23
123.63
122.87
122.03
121.57
119.53
119.31
117.47
77.41
77.16
76.91
75.46
75.44
75.42
75.39
40.30
39.58
39.36
39.13
38.91
^{13}C NMR (126 MHz, CDCl$_3$)
N–O
CF$_3$
OCF$_3$
2k
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

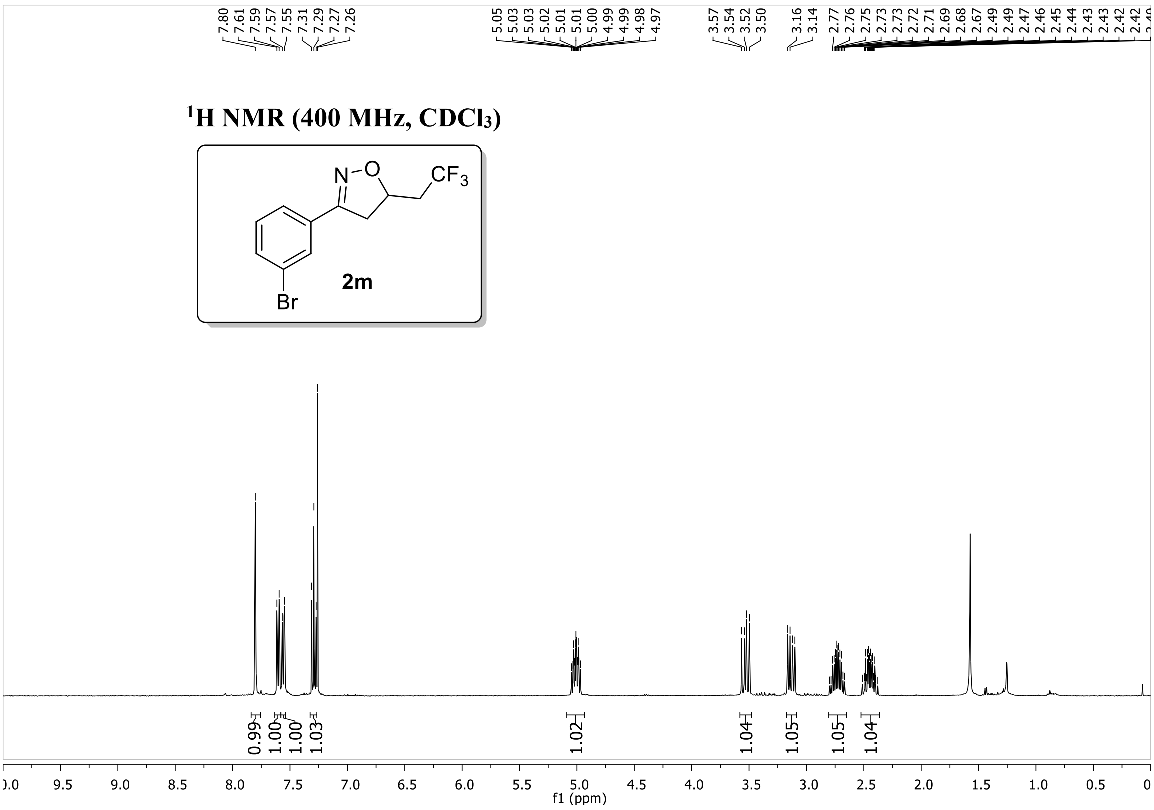

-57.86
-63.84
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
OCF$_3$
2k
.0
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-1
f1 (ppm)

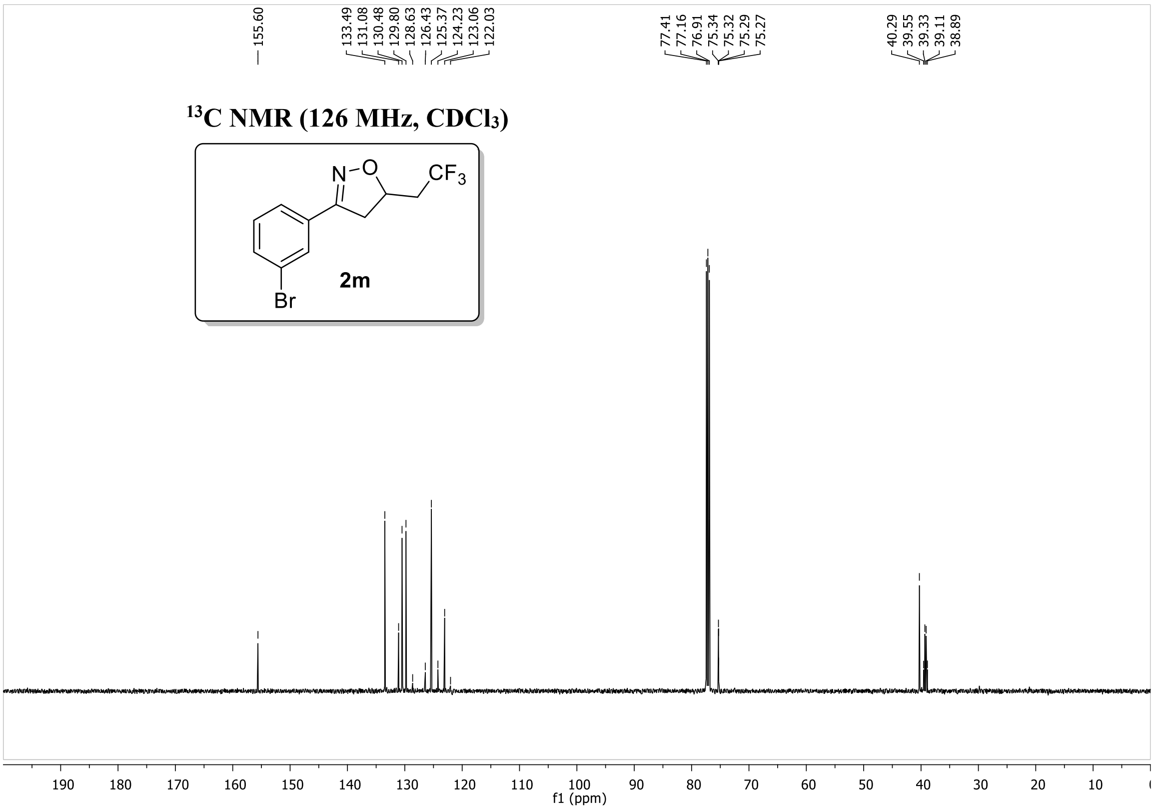

7.42
7.42
7.41
7.40
7.40
7.39
7.38
7.38
7.36
7.26
7.15
7.15
7.14
7.13
7.12
7.11
7.10
5.05
5.03
5.03
5.02
5.01
5.01
5.00
4.99
4.99
4.98
4.97
3.57
3.55
3.53
3.50
3.17
3.15
2.77
2.75
2.74
2.73
2.71
2.70
2.69
2.68
2.66
2.66
2.52
2.50
2.49
2.48
2.47
2.46
2.45
2.44
2.43
2.43
2.42
^{1}H NMR (400 MHz, CDCl$_3$)
N
O
CF$_3$
F
2l
3.06
1.00
1.04
1.04
1.05
1.03
1.02
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)


163.90
161.94
155.89
155.87
131.20
131.14
130.64
130.57
128.65
126.45
124.24
122.65
122.63
122.04
117.64
117.47
113.79
113.60
77.41
77.16
76.91
75.31
75.29
75.26
75.24
40.38
39.55
39.33
39.11
38.89
^{13}C NMR (126 MHz, CDCl$_3$)
N
O
CF$_3$
F
2l
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

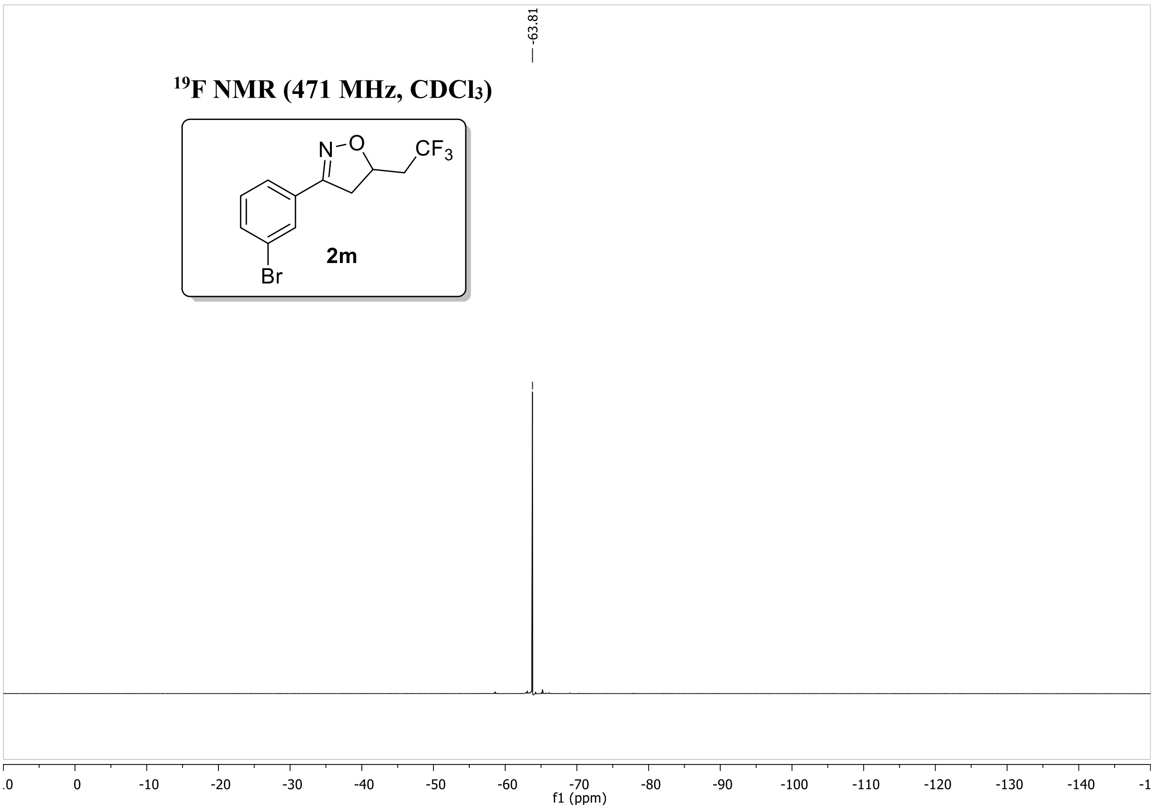


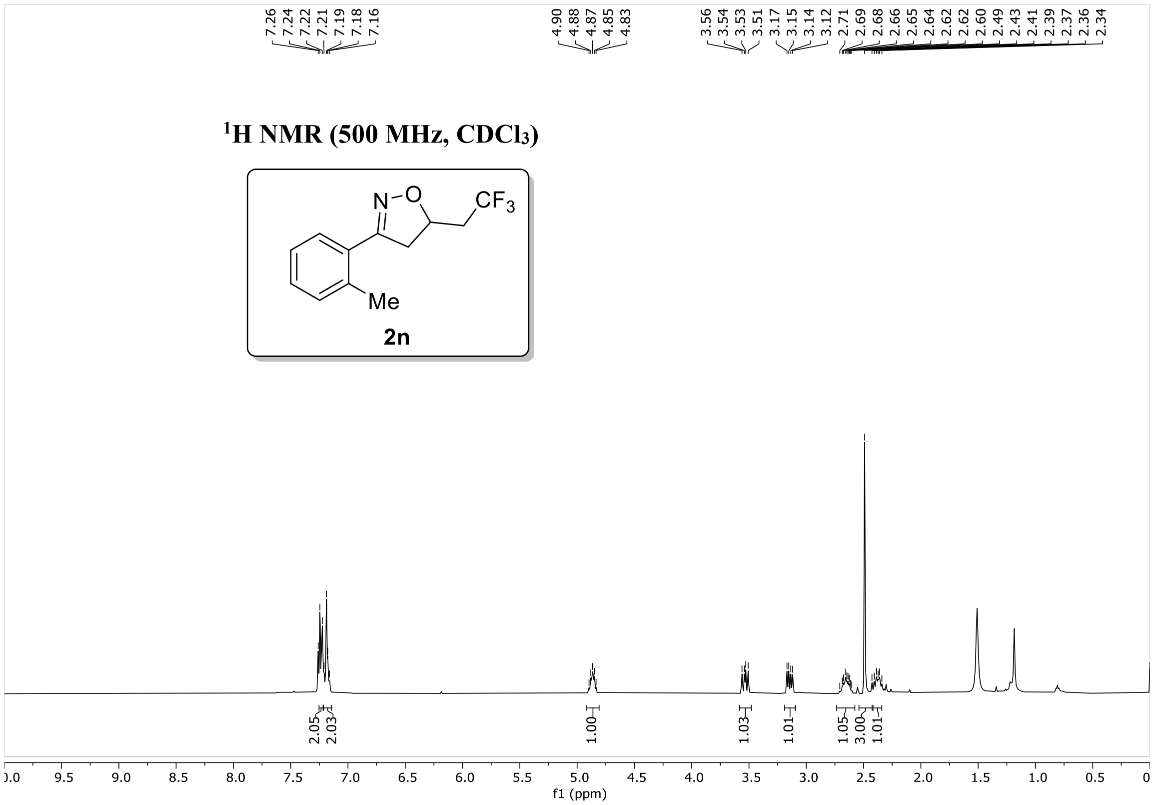
-63.85
-111.90
19F NMR (471 MHz, CDCl3)
N-O
CF3
F
2l
.0
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-1
f1 (ppm)


7.80
7.61
7.59
7.57
7.55
7.31
7.29
7.27
7.26
5.05
5.03
5.03
5.02
5.01
5.01
5.00
4.99
4.99
4.98
4.97
3.57
3.54
3.52
3.50
3.16
3.14
2.77
2.76
2.75
2.73
2.73
2.72
2.71
2.69
2.68
2.67
2.49
2.47
2.46
2.45
2.44
2.43
2.43
2.42
¹H NMR (400 MHz, CDCl₃)
N
O
CF₃
Br
2m
0.99
1.00
1.00
1.03
1.02
1.04
1.05
1.05
1.04
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)


155.60
133.49
131.08
130.48
129.80
128.63
126.43
125.37
124.23
123.06
122.03
77.41
77.16
76.91
75.34
75.32
75.29
75.27
40.29
39.55
39.33
39.11
38.89
¹³C NMR (126 MHz, CDCl₃)
N
O
CF₃
Br
2m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

^{19}F NMR (471 MHz, $CDCl_3$)

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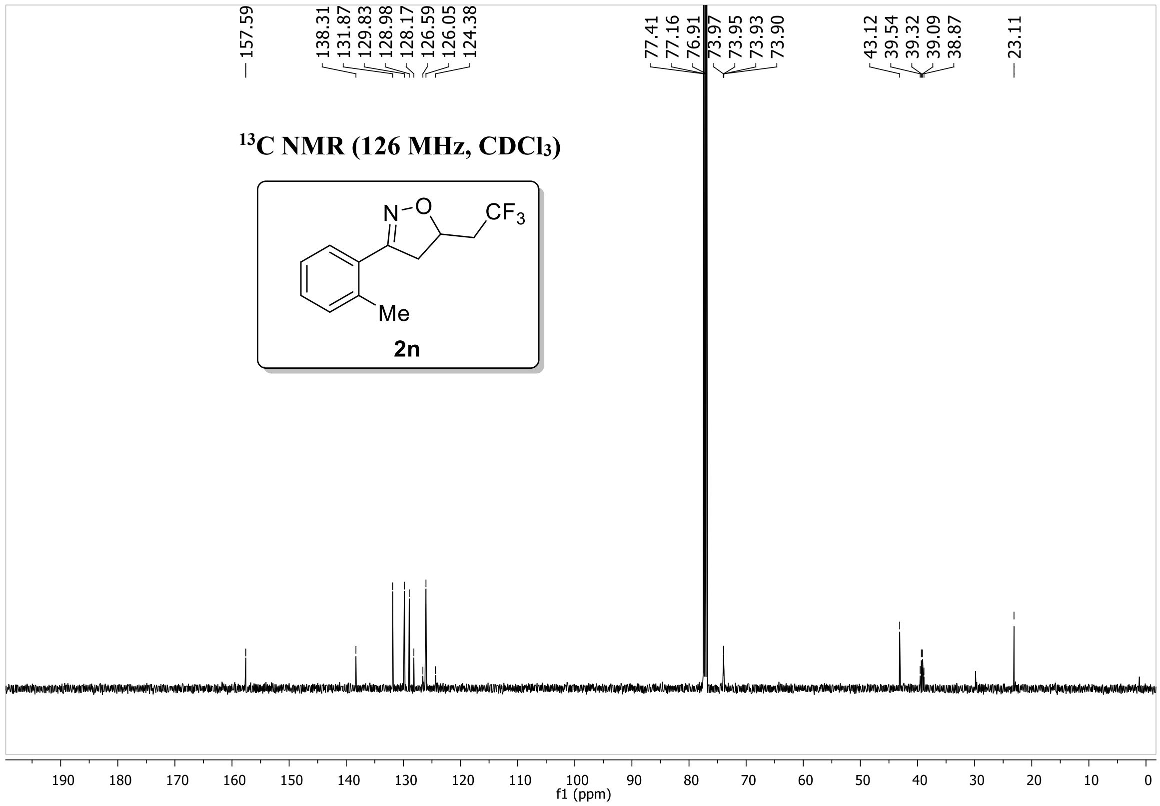
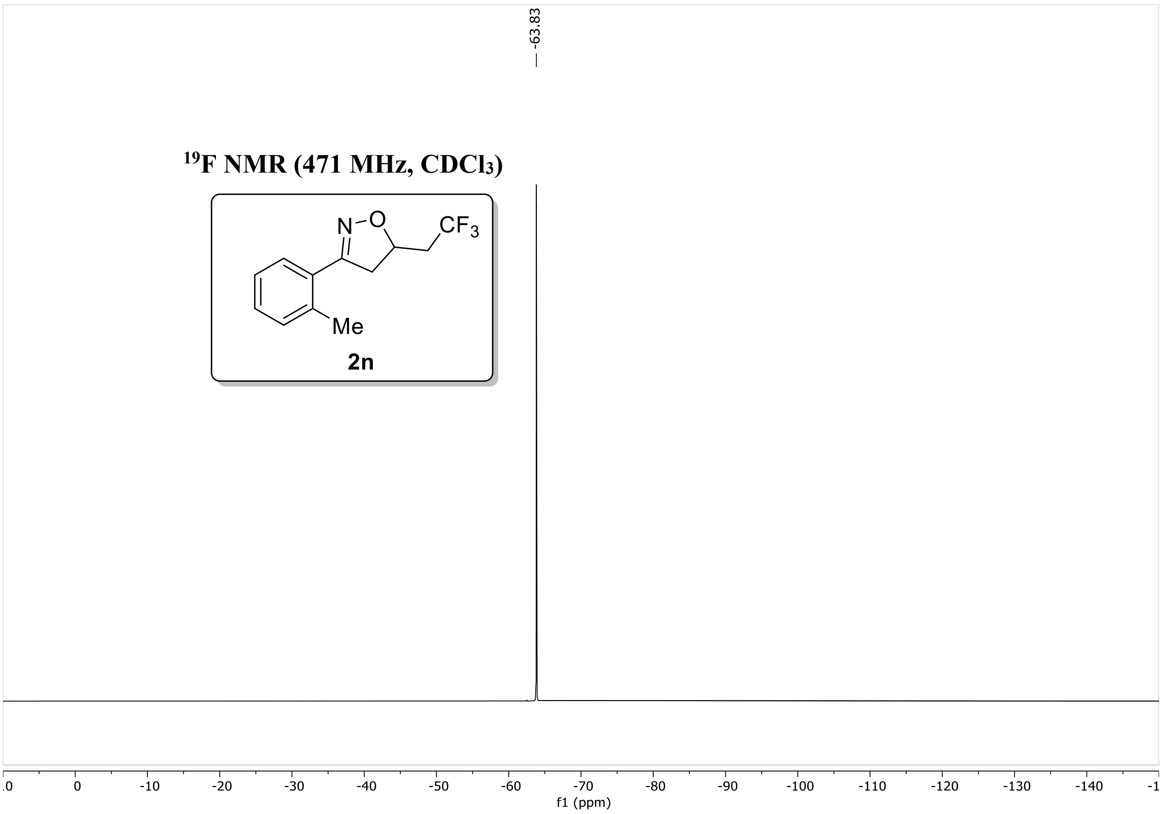
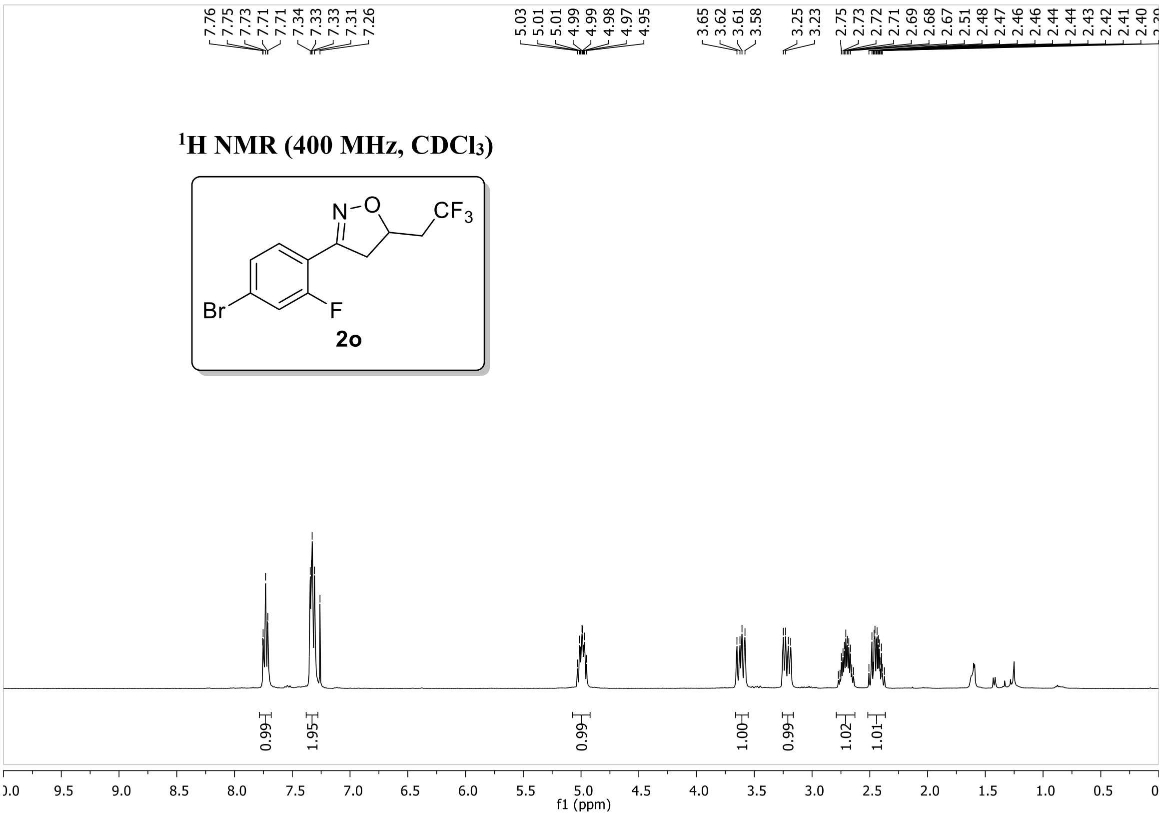
7.24
7.22
7.21
7.19
7.18
7.16
4.90
4.88
4.87
4.85
4.83
3.56
3.54
3.53
3.51
3.17
3.15
3.14
3.12
2.71
2.69
2.68
2.66
2.65
2.64
2.62
2.62
2.60
2.49
2.43
2.41
2.39
2.37
2.36
2.34
^{1}H NMR (500 MHz, CDCl$_3$)
N
O
CF$_3$
Me
2n
2.05
2.03
1.00
1.03
1.01
1.05
3.00
1.01
f1 (ppm)


157.59
138.31
131.87
129.83
128.98
128.17
126.59
126.05
124.38
77.41
77.16
76.91
73.97
73.95
73.93
73.90
43.12
39.54
39.32
39.09
38.87
23.11
^{13}C NMR (126 MHz, CDCl$_3$)
N
O
CF$_3$
Me
2n
f1 (ppm)


-63.83
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
Me
2n
.0
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-1
f1 (ppm)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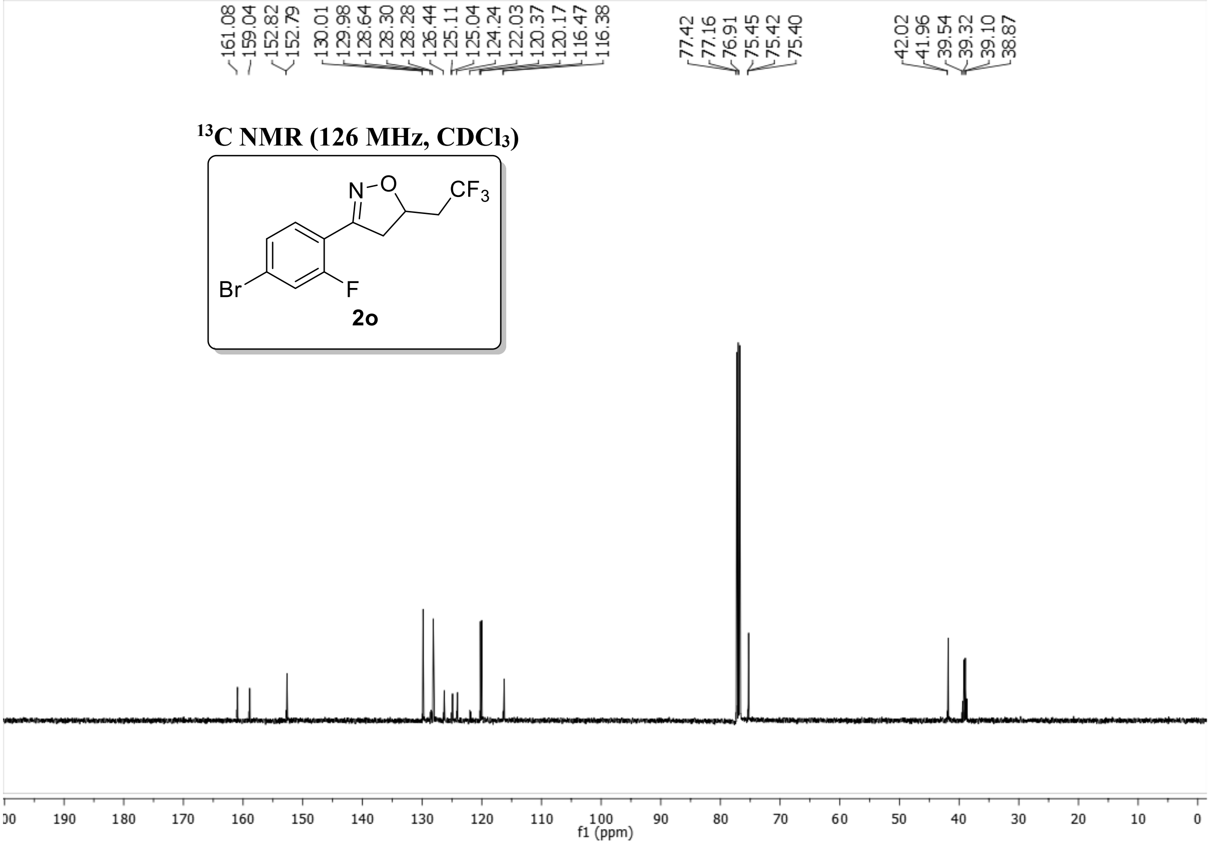
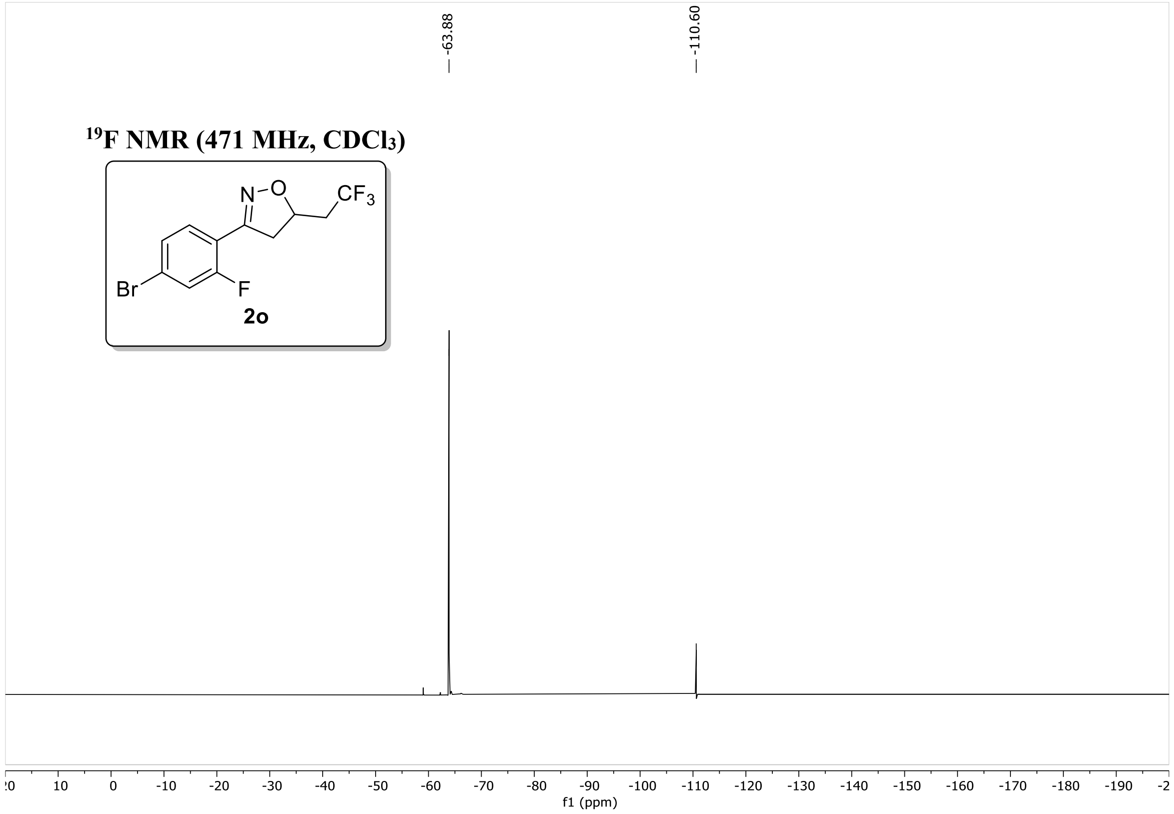
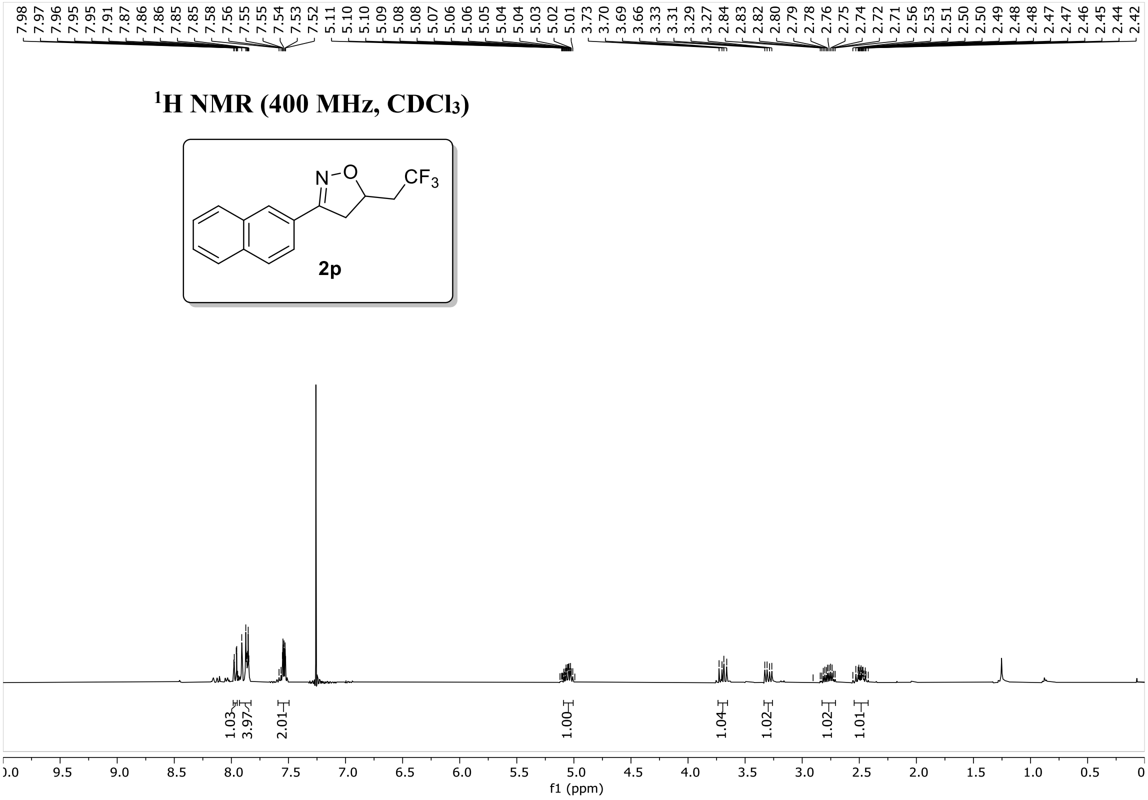
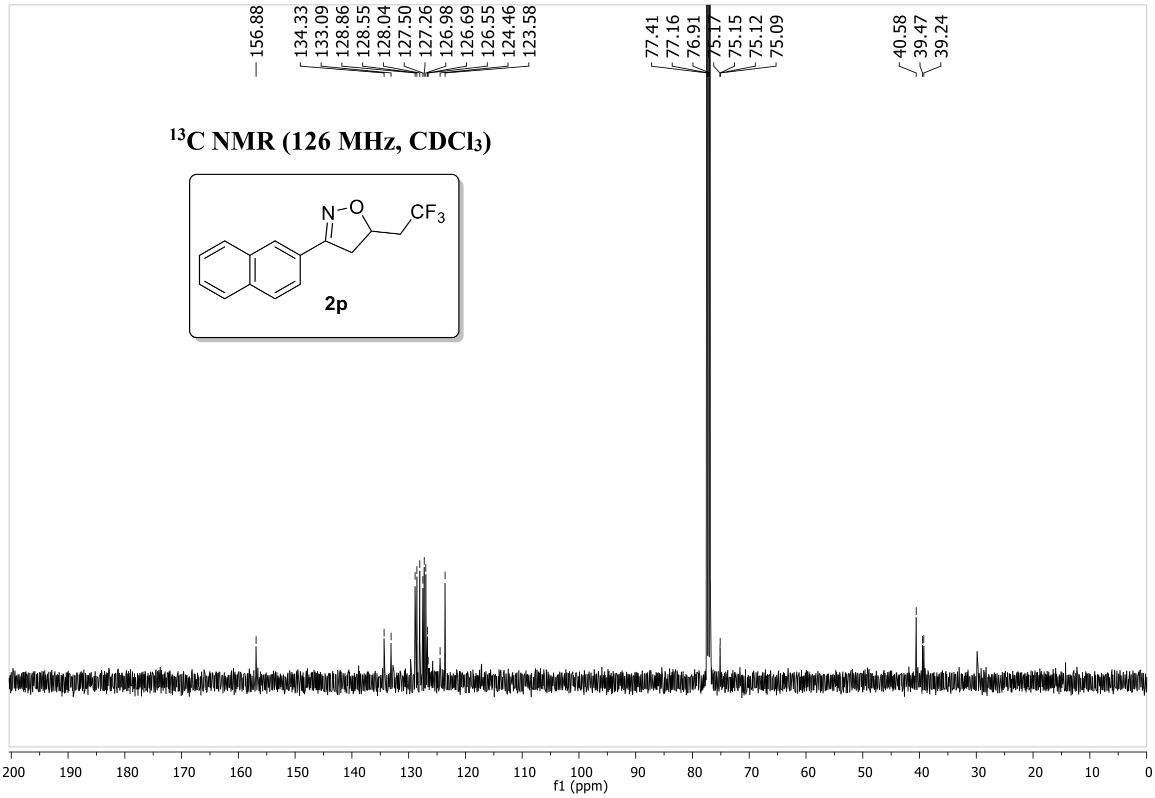
7.75
7.73
7.71
7.71
7.34
7.33
7.33
7.31
7.26
5.03
5.01
5.01
4.99
4.99
4.98
4.97
4.95
3.65
3.62
3.61
3.58
3.25
3.23
2.75
2.73
2.72
2.71
2.69
2.68
2.67
2.51
2.48
2.47
2.46
2.44
2.44
2.43
2.42
2.41
2.40
¹H NMR (400 MHz, CDCl₃)
N
O
CF₃
Br
F
2o
0.99
1.95
0.99
1.00
0.99
1.02
1.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
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
f1 (ppm)

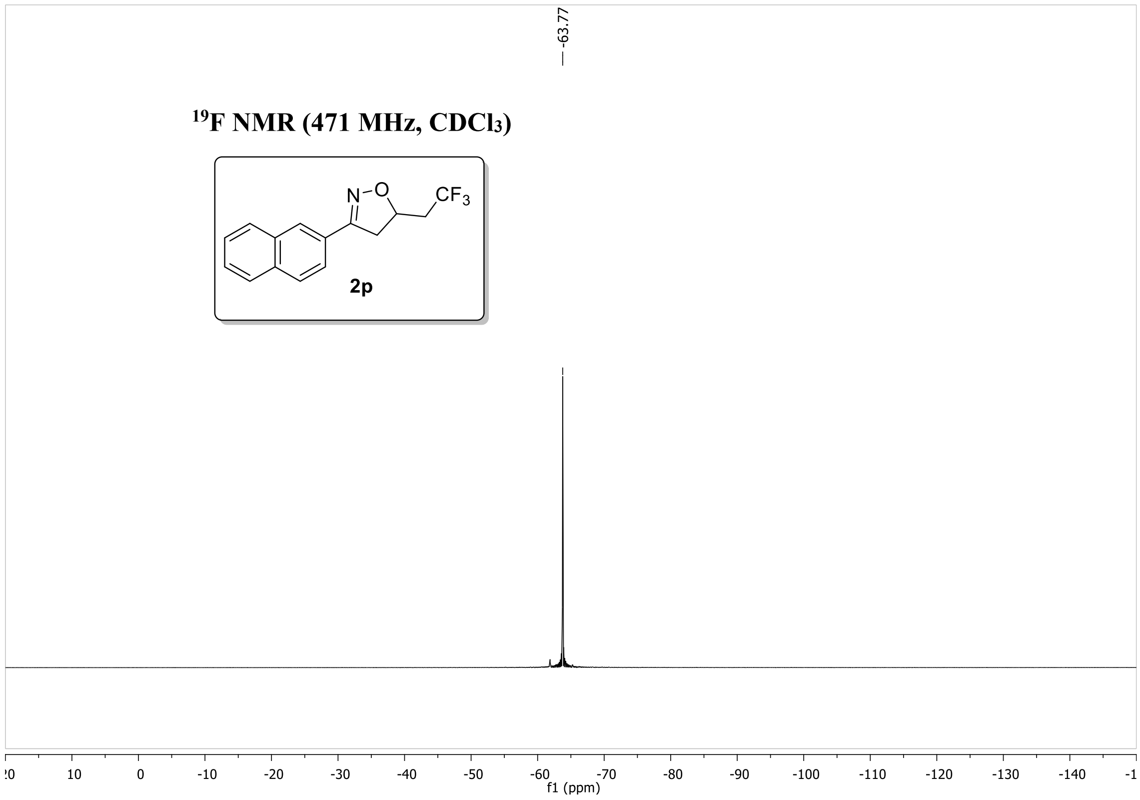

161.08
159.04
152.82
152.79
130.01
129.98
128.64
128.30
128.28
126.44
125.11
125.04
124.24
122.03
120.37
120.17
116.47
116.38
77.42
77.16
76.91
75.45
75.42
75.40
42.02
41.96
39.54
39.32
39.10
38.87
¹³C NMR (126 MHz, CDCl₃)
N
O
CF₃
Br
F
2o
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



^{19}F NMR (471 MHz, $CDCl_3$)

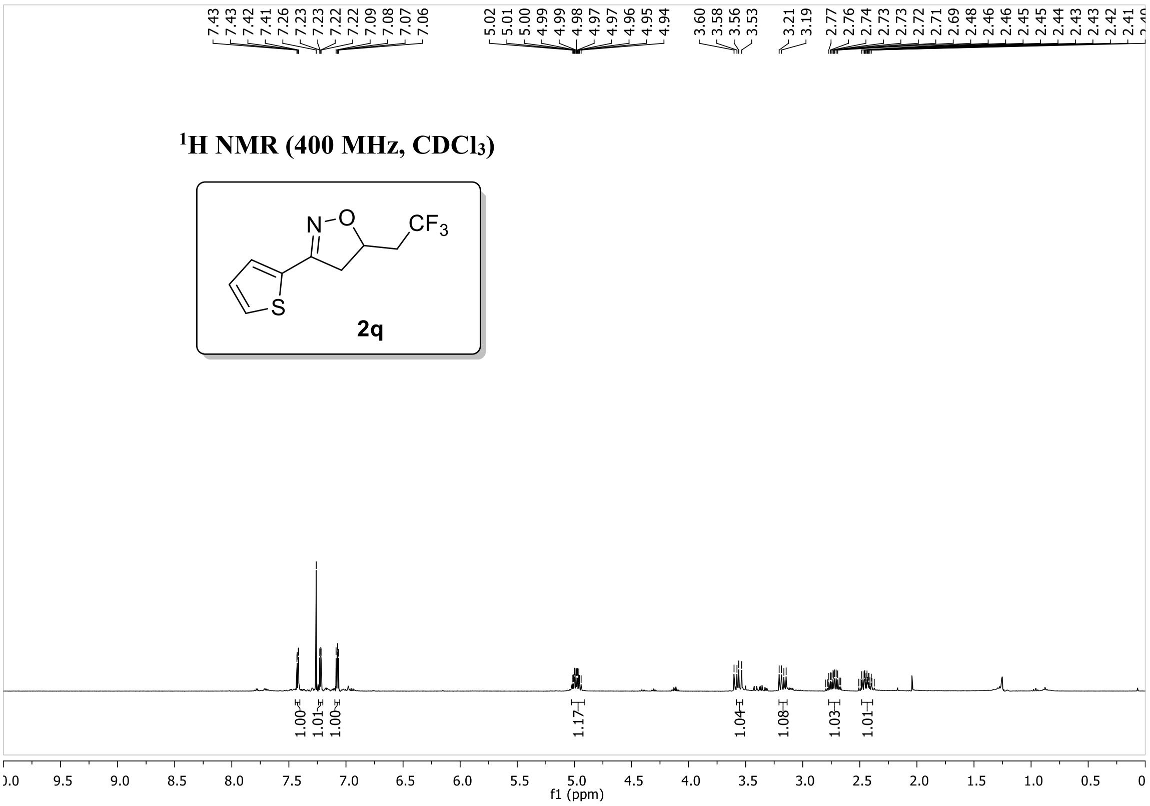

1H NMR (400 MHz, CDCl3)
N–O
CF3
2p
7.98 7.97 7.96 7.95 7.95 7.91 7.87 7.86 7.86 7.85 7.85 7.58 7.56 7.55 7.55 7.54 7.53 7.52 5.11 5.10 5.09 5.08 5.08 5.07 5.06 5.06 5.05 5.04 5.04 5.03 5.02 5.01 3.73 3.70 3.69 3.66 3.33 3.31 3.29 3.27 2.84 2.83 2.82 2.80 2.79 2.78 2.76 2.75 2.74 2.72 2.71 2.56 2.53 2.51 2.50 2.50 2.49 2.48 2.48 2.47 2.47 2.46 2.45 2.44 2.42
1.03
3.97
2.01
1.00
1.04
1.02
1.02
1.01
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
f1 (ppm)

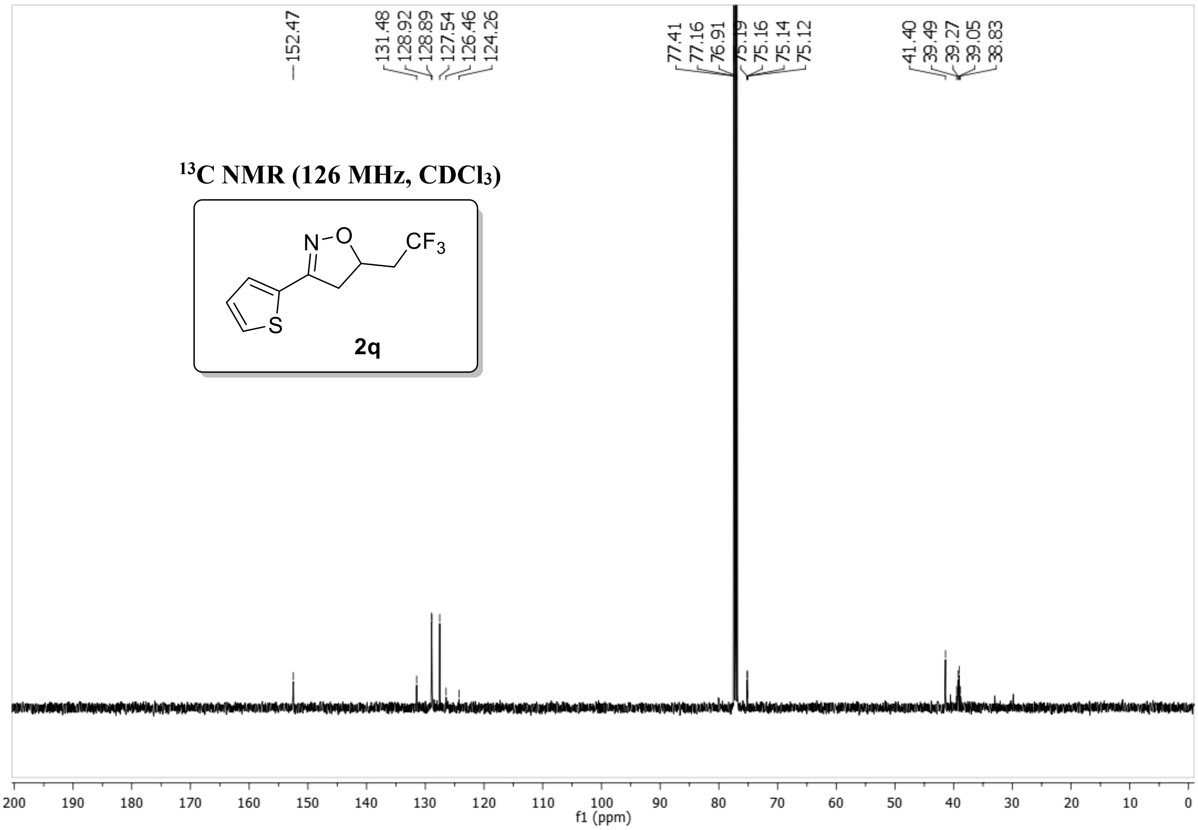

13C NMR (126 MHz, CDCl3)
N–O
CF3
2p
156.88
134.33 133.09 128.86 128.55 128.04 127.50 127.26 126.98 126.69 126.55 124.46 123.58
77.41 77.16 76.91 75.17 75.15 75.12 75.09
40.58 39.47 39.24
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

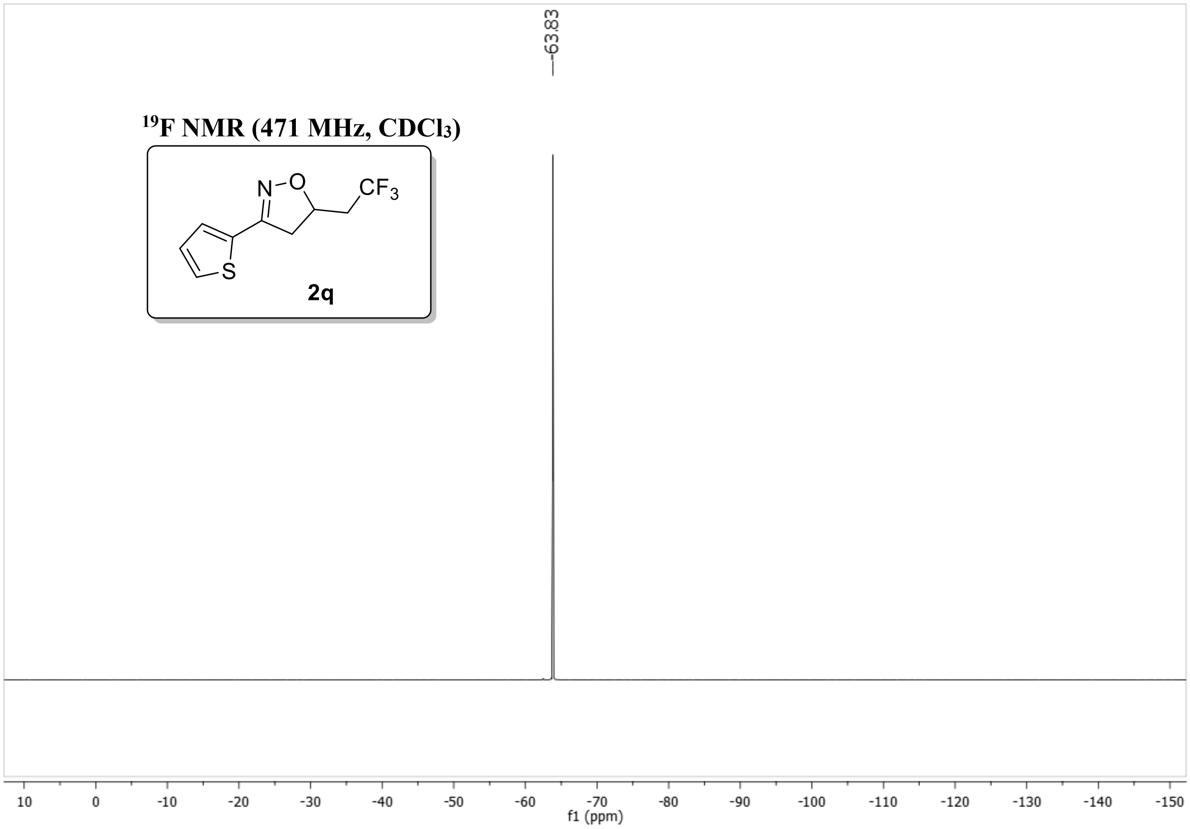


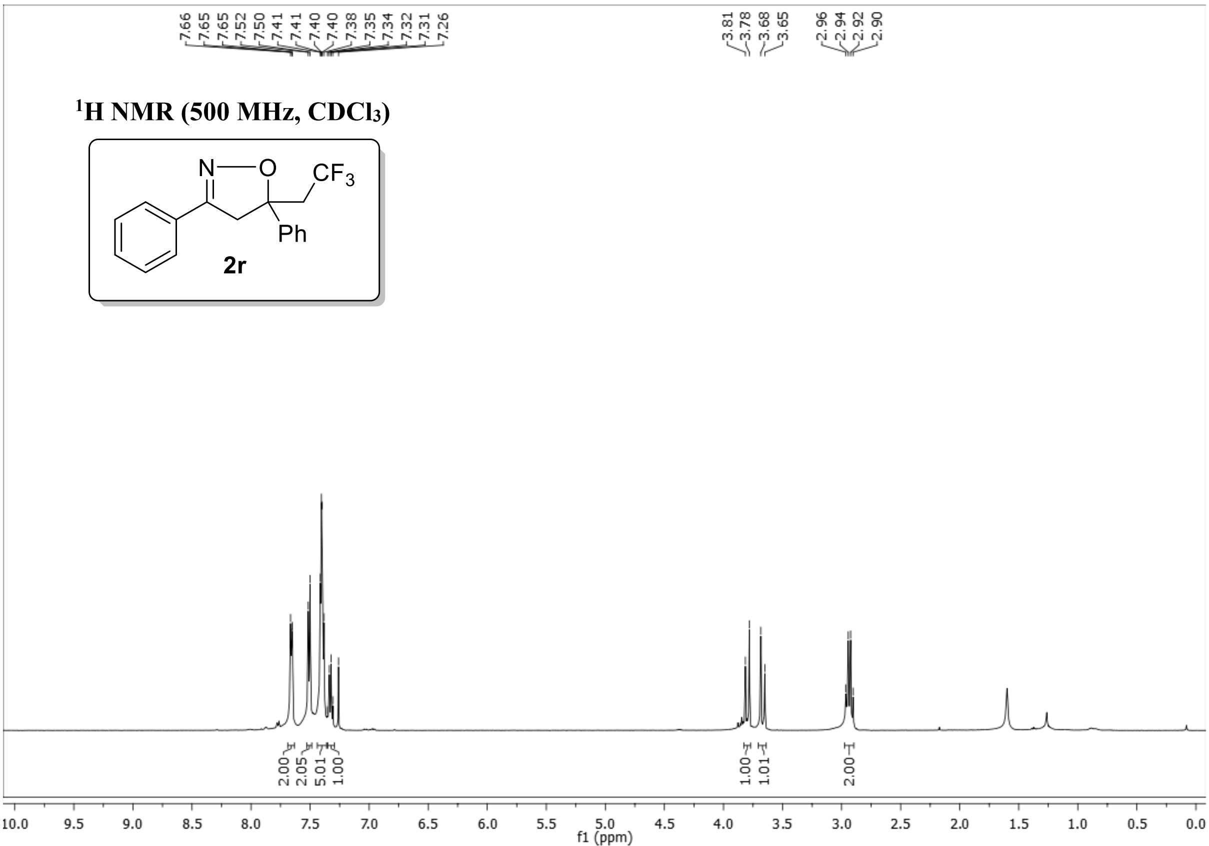
^{19}F NMR (471 MHz, CDCl$_3$)

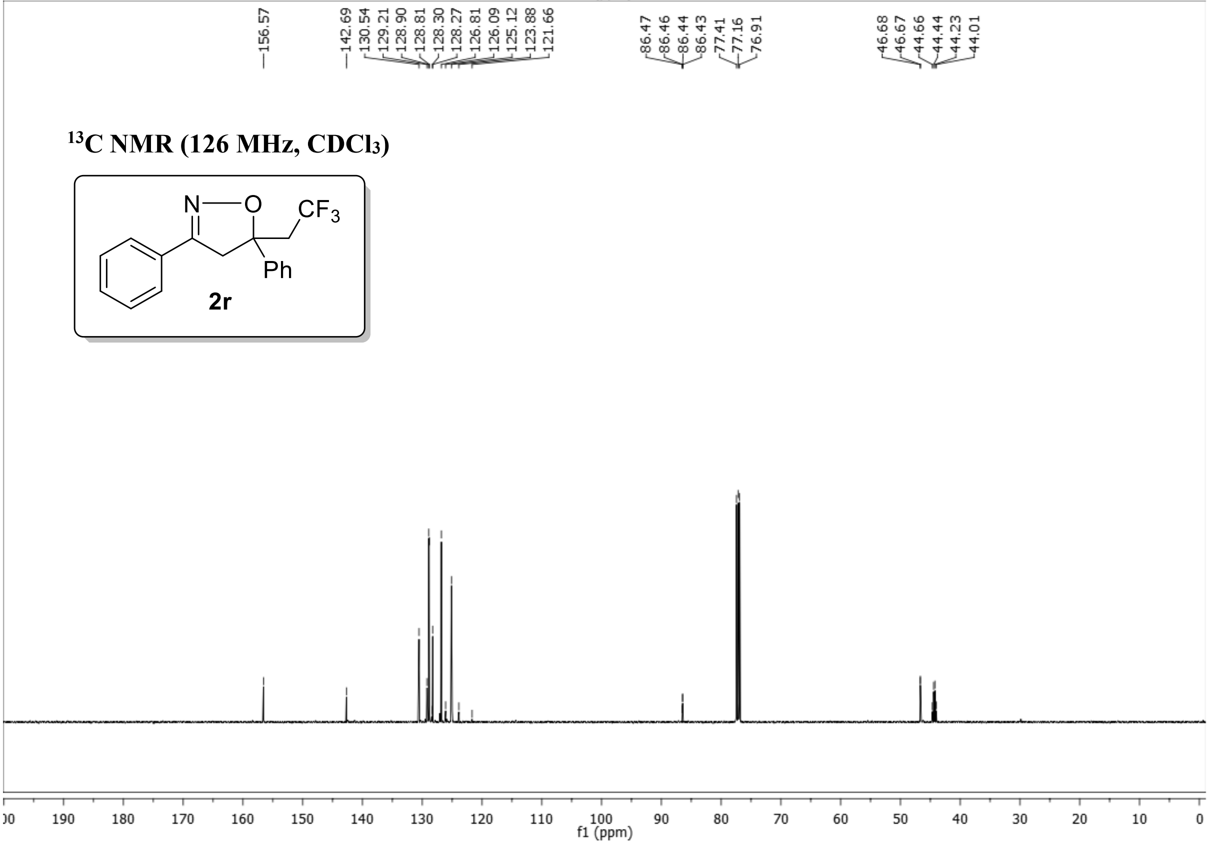
2p

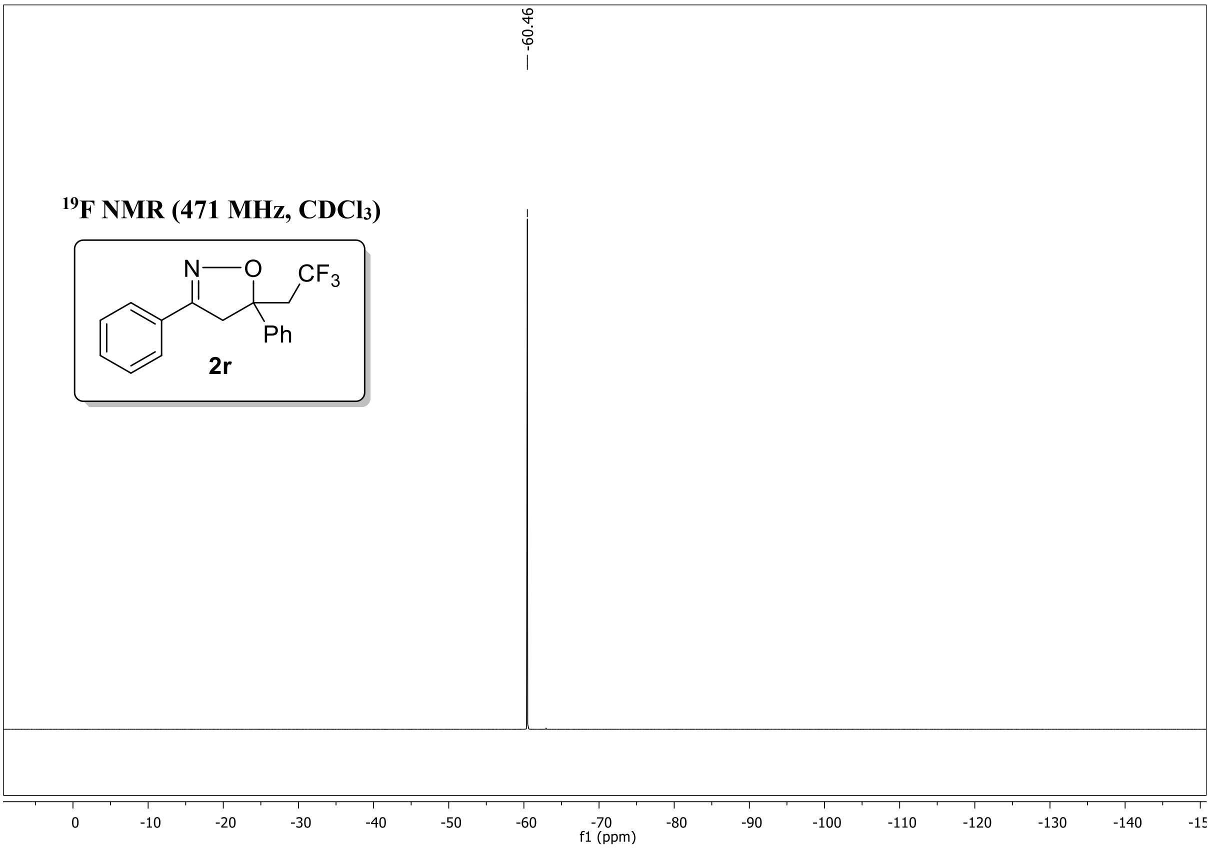

7.43
7.43
7.42
7.41
7.26
7.23
7.23
7.22
7.22
7.09
7.08
7.07
7.06
5.02
5.01
5.00
4.99
4.99
4.98
4.97
4.97
4.96
4.95
4.94
3.60
3.58
3.56
3.53
3.21
3.19
2.77
2.76
2.74
2.73
2.73
2.72
2.71
2.69
2.48
2.46
2.45
2.45
2.44
2.43
2.43
2.42
2.41
^{1}H NMR (400 MHz, CDCl$_3$)
N
O
CF$_3$
S
2q
1.00
1.01
1.00
1.17
1.04
1.08
1.03
1.01
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
f1 (ppm)

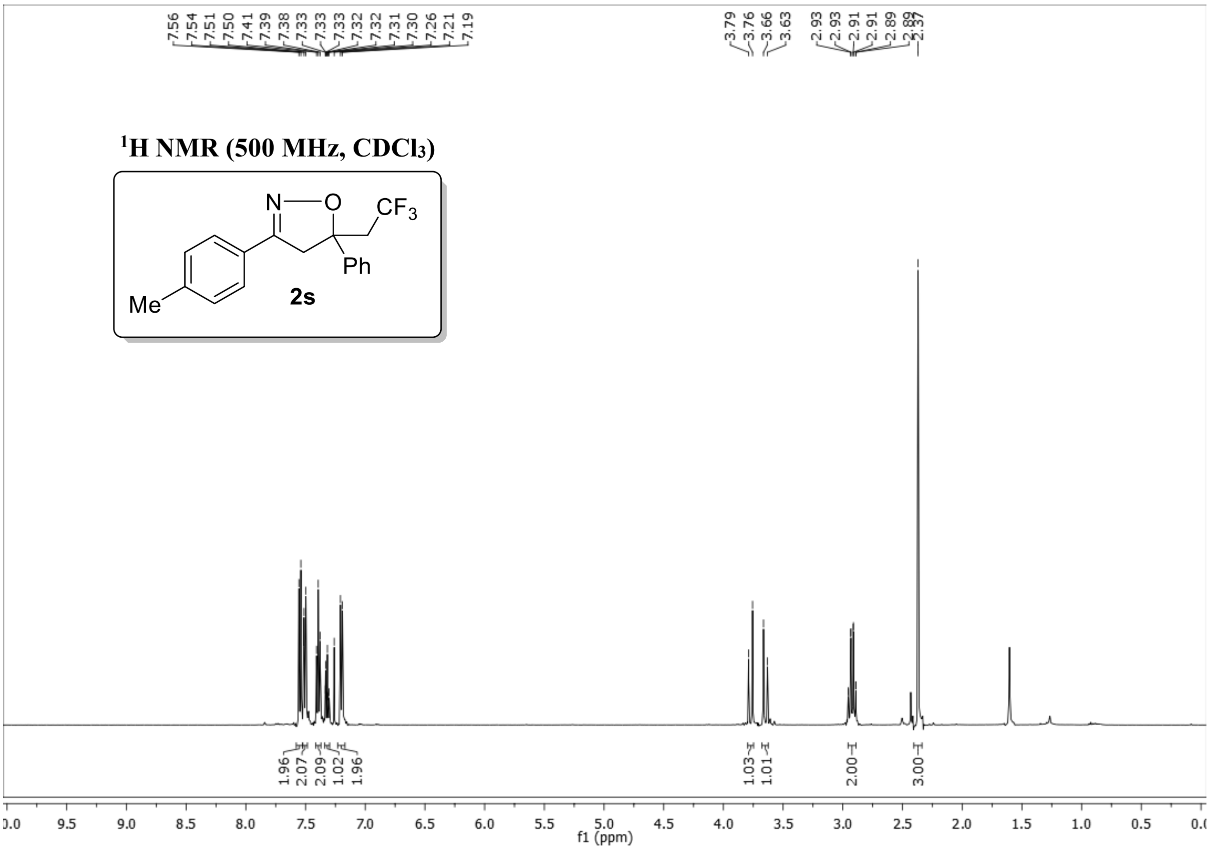

152.47
131.48
128.92
128.89
127.54
126.46
124.26
77.41
77.16
76.91
75.19
75.16
75.14
75.12
41.40
39.49
39.27
39.05
38.83
^{13}C NMR (126 MHz, CDCl$_3$)
N
O
CF$_3$
S
2q
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

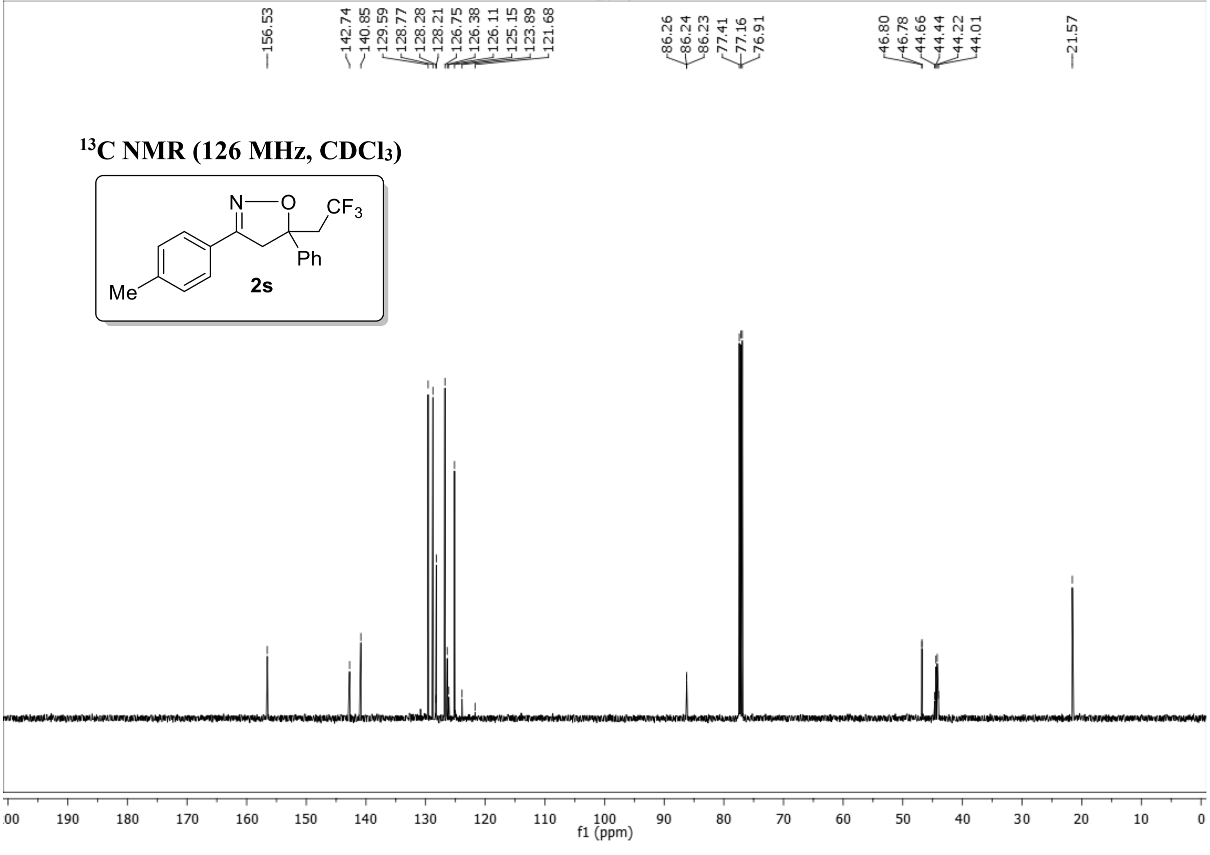

—63.83
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
S
2q
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)

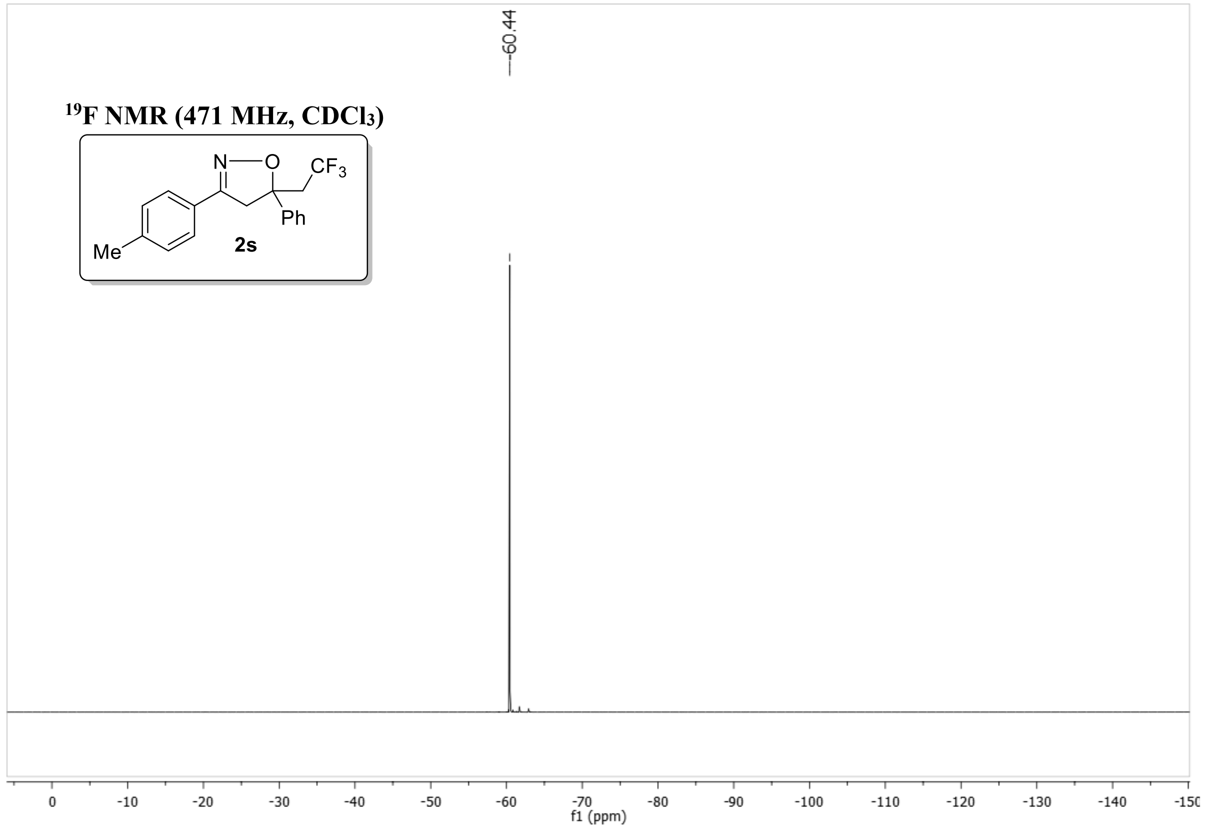

7.66
7.65
7.65
7.52
7.50
7.41
7.41
7.40
7.40
7.38
7.35
7.34
7.32
7.31
7.26
3.81
3.78
3.68
3.65
2.96
2.94
2.92
2.90
1H NMR (500 MHz, CDCl3)
N
O
CF3
Ph
2r
2.00
2.05
5.01
1.00
1.00
1.01
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

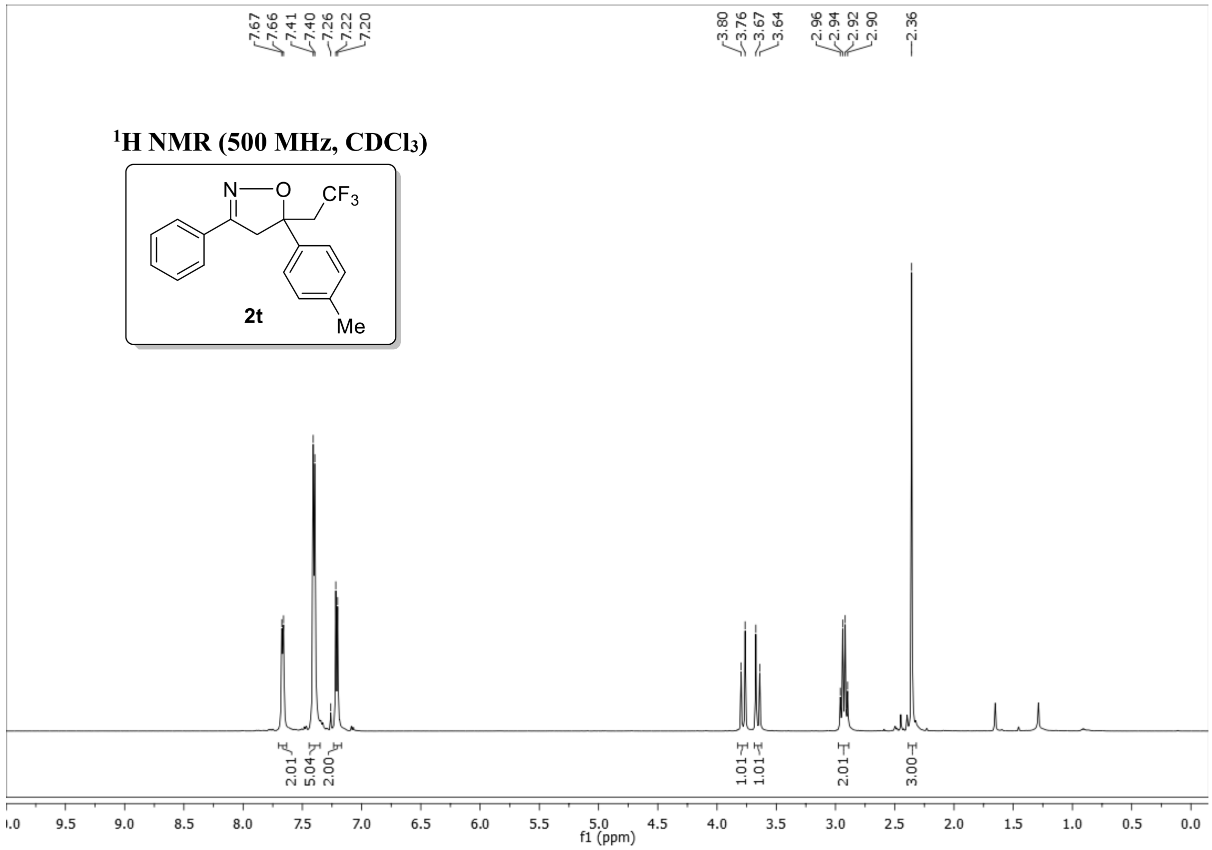

156.57
142.69
130.54
129.21
128.90
128.81
128.30
128.27
126.81
126.09
125.12
123.88
121.66
86.47
86.46
86.44
86.43
77.41
77.16
76.91
46.68
46.67
44.66
44.44
44.23
44.01
13C NMR (126 MHz, CDCl3)
N
O
CF3
Ph
2r
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

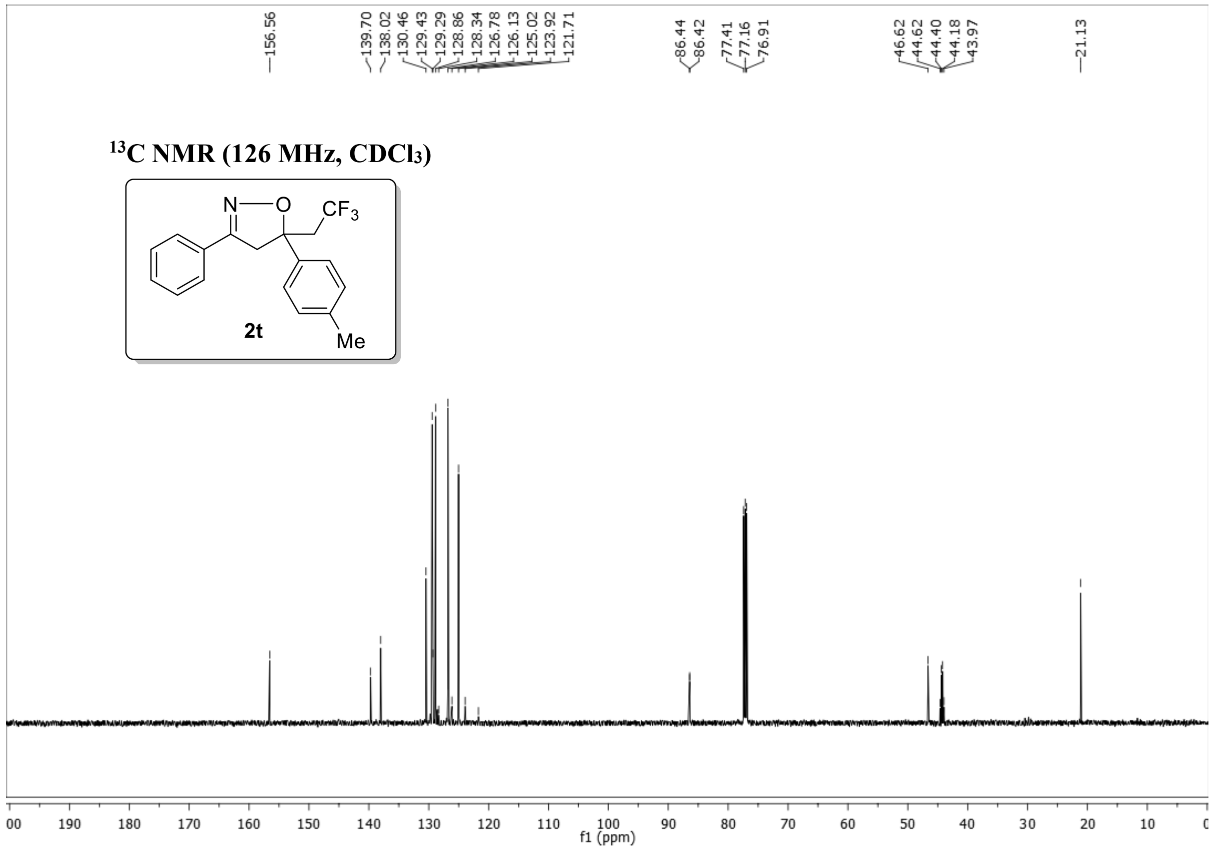

-60.46
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
Ph
2r
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-15
f1 (ppm)

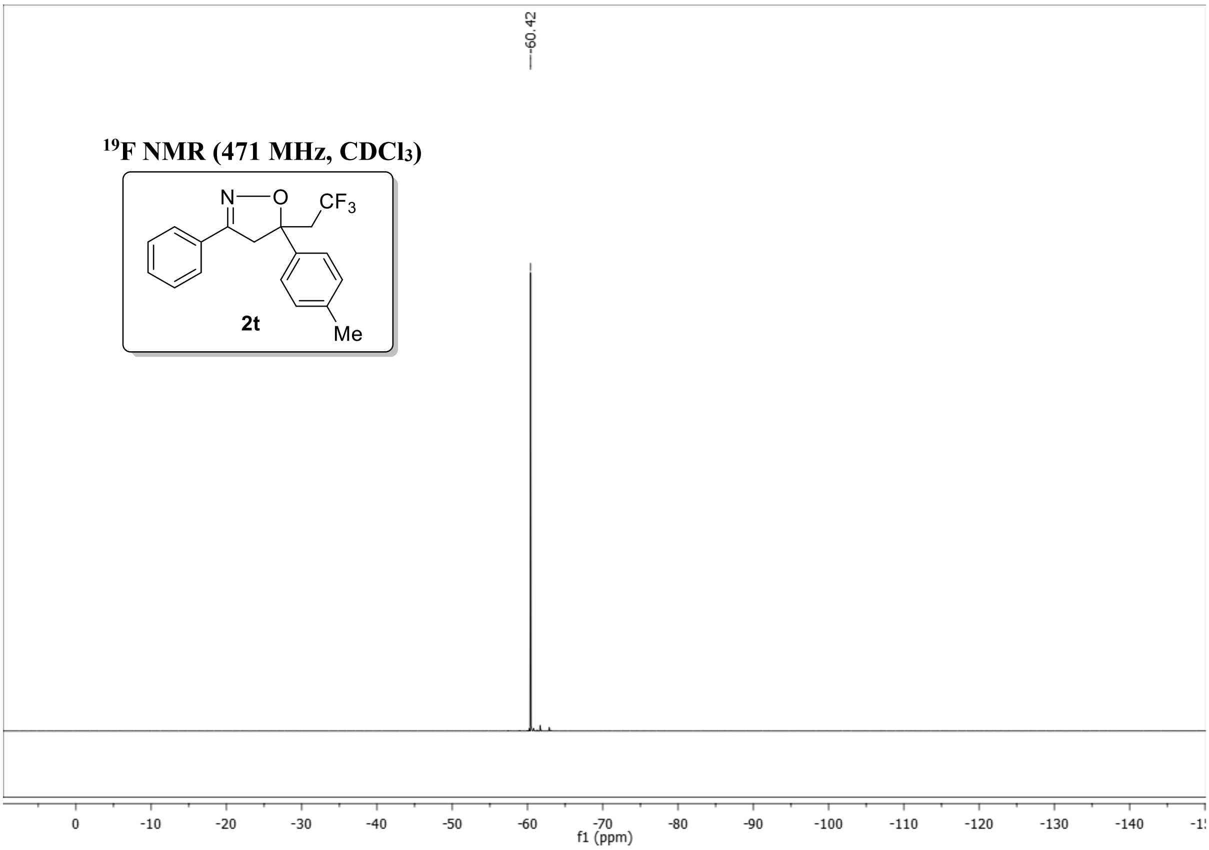

7.56
7.54
7.51
7.50
7.41
7.39
7.38
7.33
7.33
7.33
7.32
7.32
7.31
7.30
7.26
7.21
7.19
3.79
3.76
3.66
3.63
2.93
2.93
2.91
2.91
2.89
2.89
2.37
1H NMR (500 MHz, CDCl3)
N
O
CF3
Ph
Me
2s
1.96
2.07
2.09
1.02
1.96
1.03
1.01
2.00
3.00
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


156.53
142.74
140.85
129.59
128.77
128.28
128.21
126.75
126.38
126.11
125.15
123.89
121.68
86.26
86.24
86.23
77.41
77.16
76.91
46.80
46.78
44.66
44.44
44.22
44.01
21.57
13C NMR (126 MHz, CDCl3)
N
O
CF3
Ph
Me
2s
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-60.44
19F NMR (471 MHz, CDCl3)
N
O
CF3
Ph
Me
2s
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)

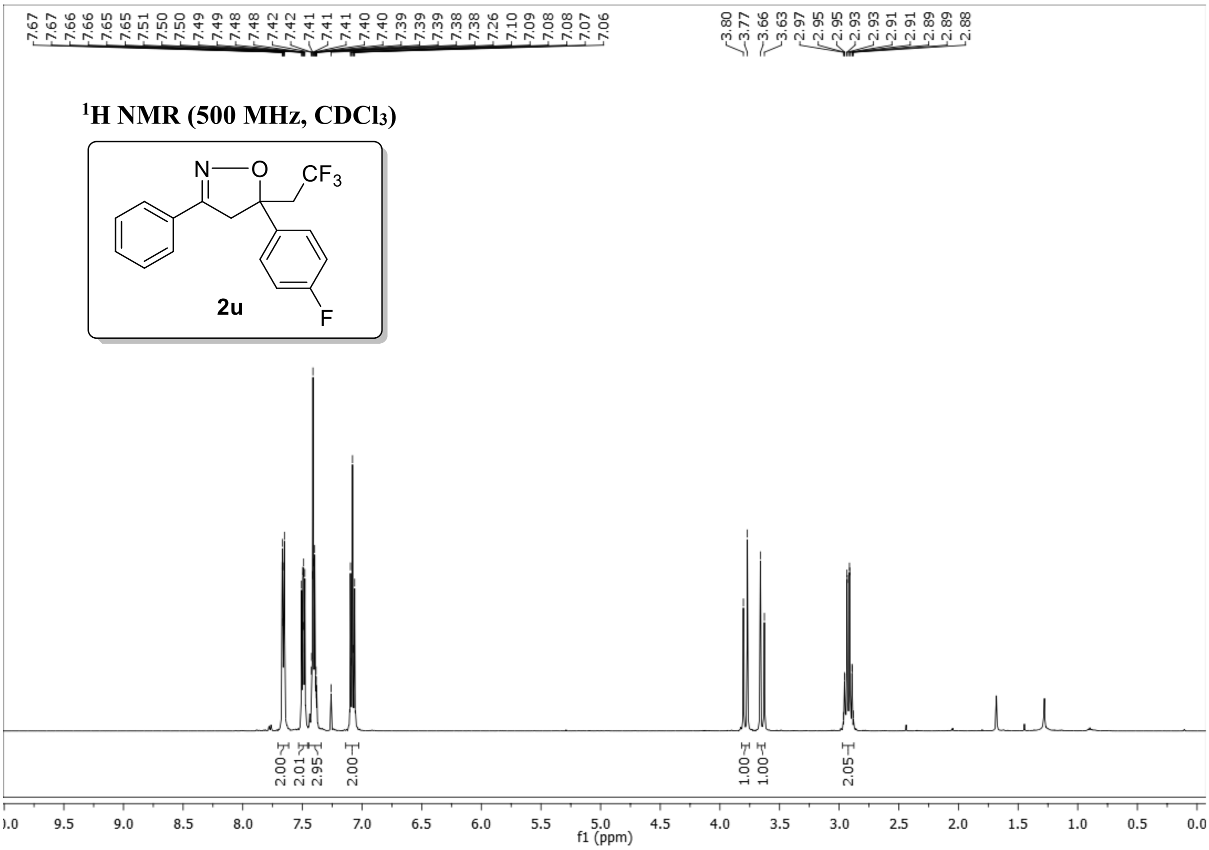

7.67
7.66
7.41
7.40
7.26
7.22
7.20
3.80
3.76
3.67
3.64
2.96
2.94
2.92
2.90
2.36
^{1}H NMR (500 MHz, CDCl$_3$)
N
O
CF$_3$
2t
Me
2.01
5.04
2.00
1.01
1.01
2.01
3.00
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


156.56
139.70
138.02
130.46
129.43
129.29
128.86
128.34
126.78
126.13
125.02
123.92
121.71
86.44
86.42
77.41
77.16
76.91
46.62
44.62
44.40
44.18
43.97
21.13
^{13}C NMR (126 MHz, CDCl$_3$)
N
O
CF$_3$
2t
Me
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

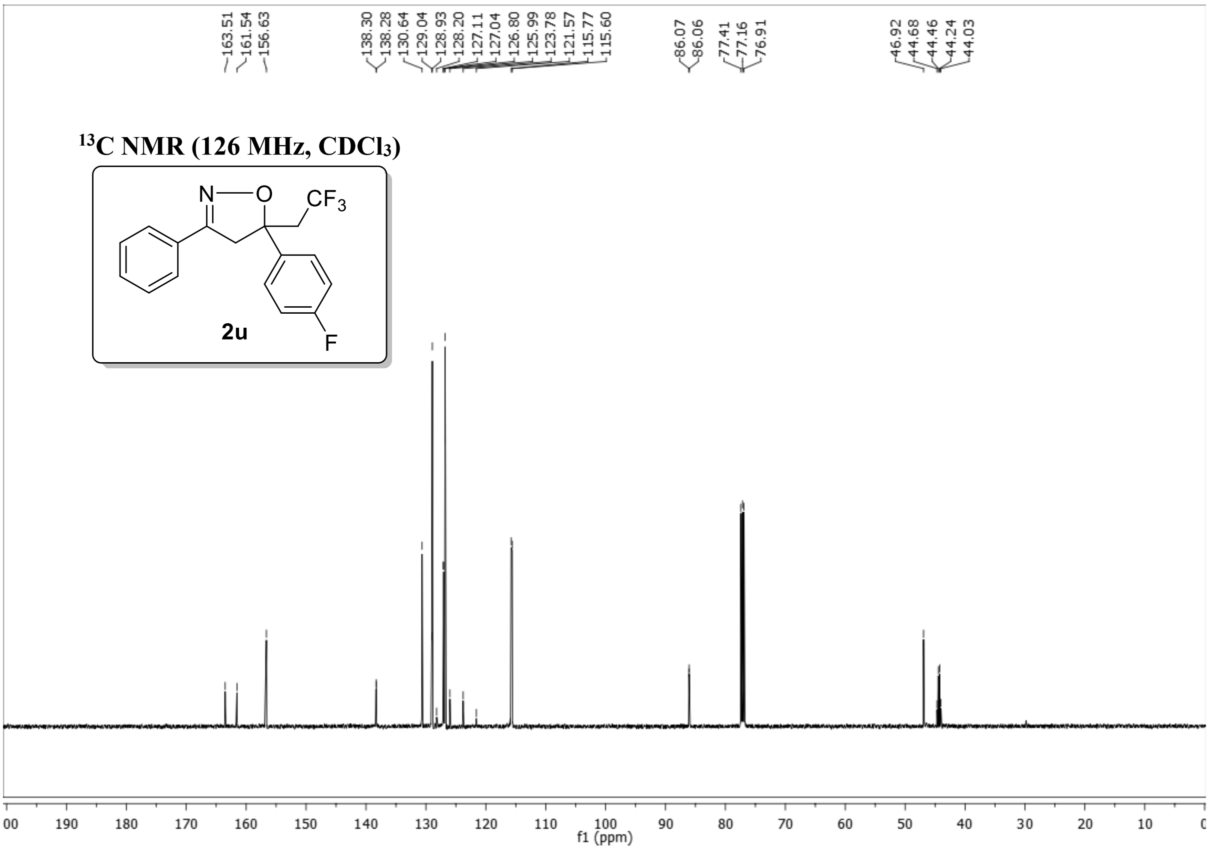


^{19}F NMR (471 MHz, CDCl$_3$)

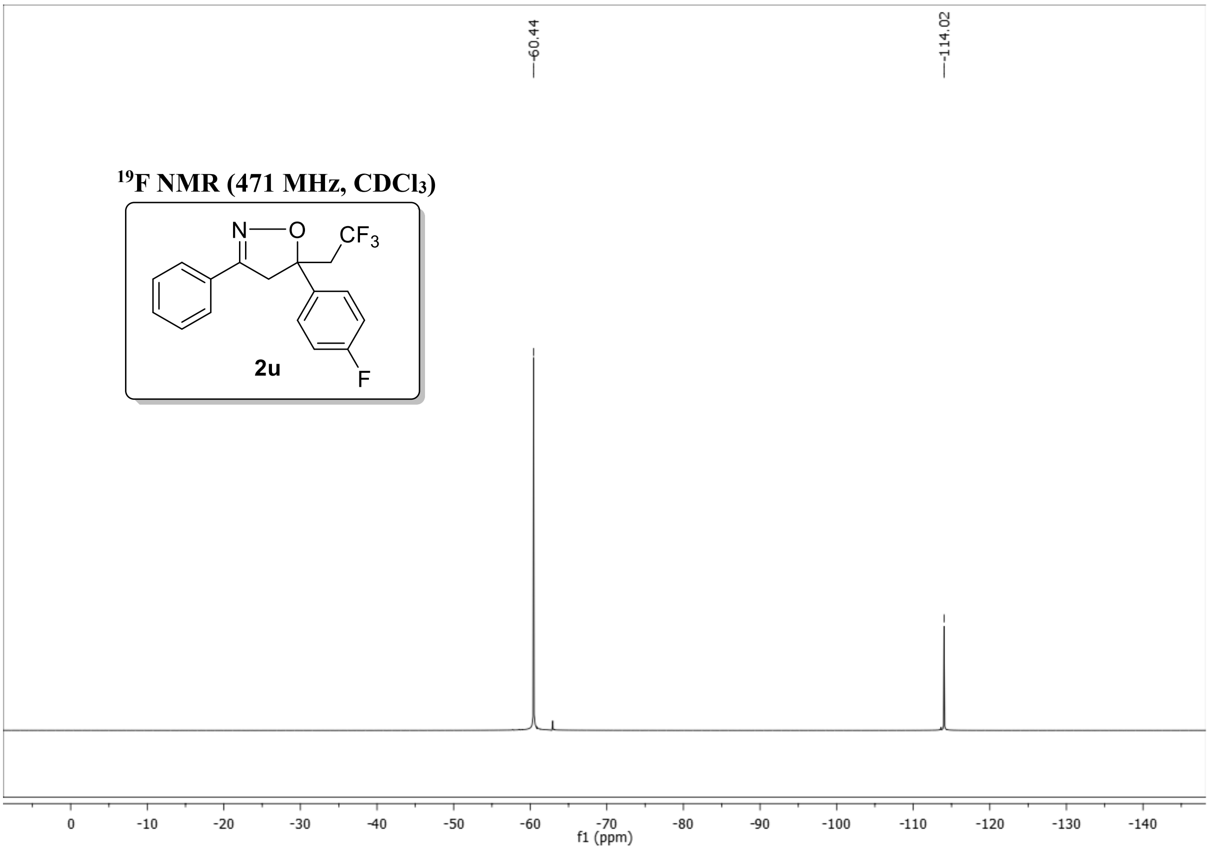
2t

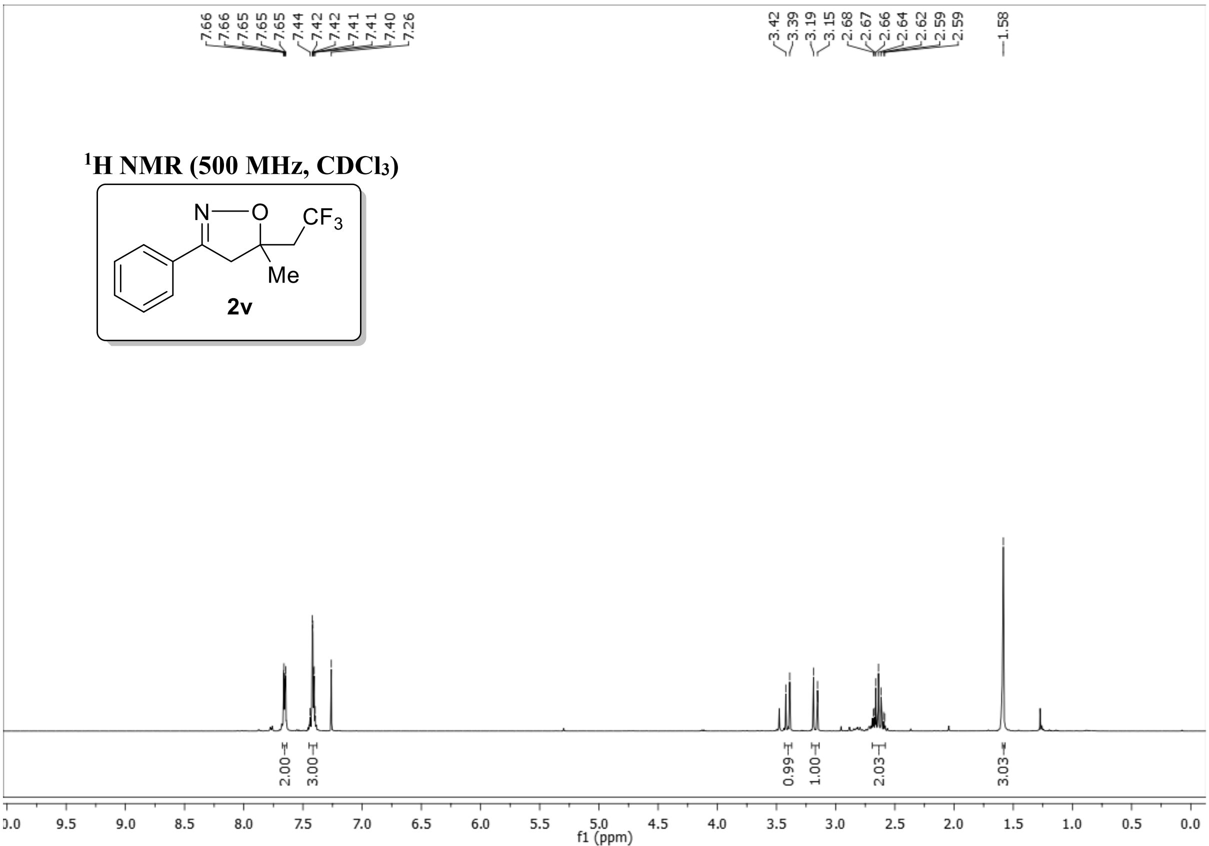


^{1}H NMR (500 MHz, CDCl$_3$)

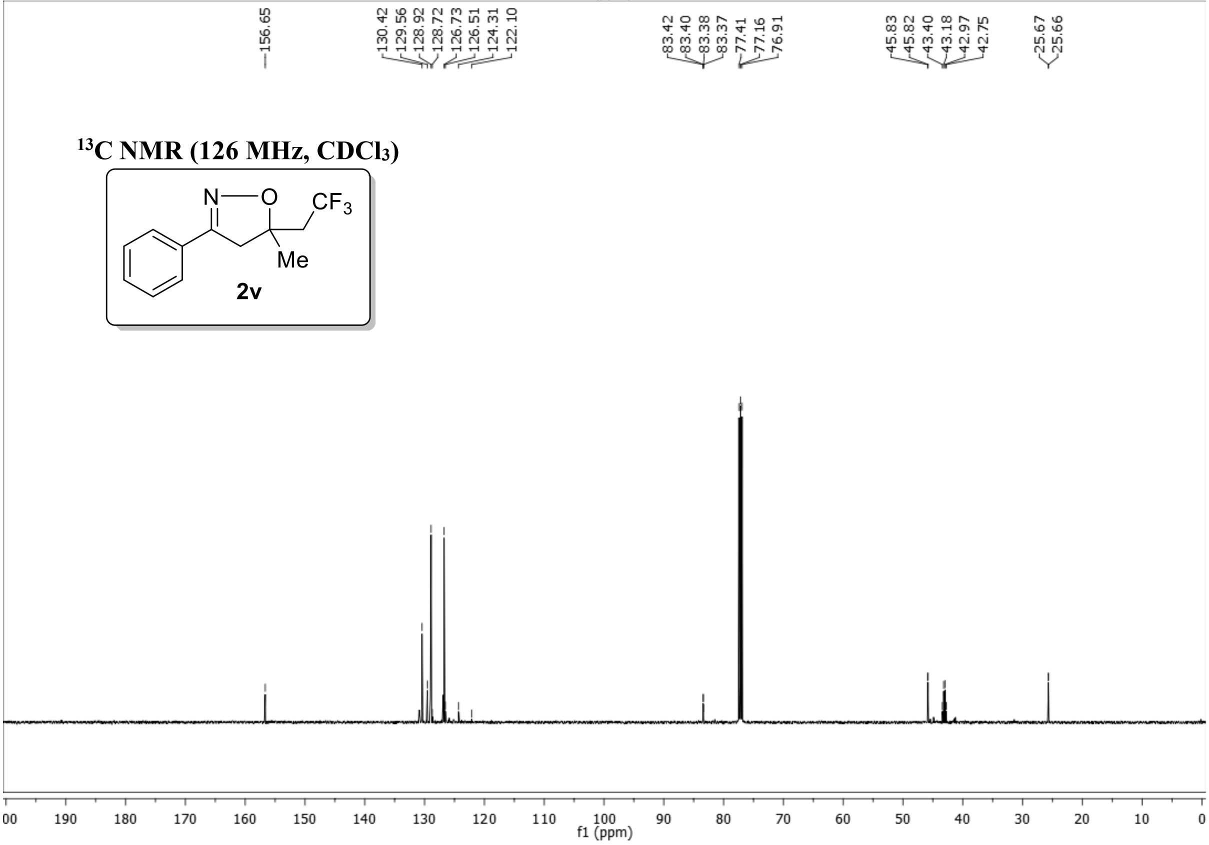


^{13}C NMR (126 MHz, CDCl$_3$)

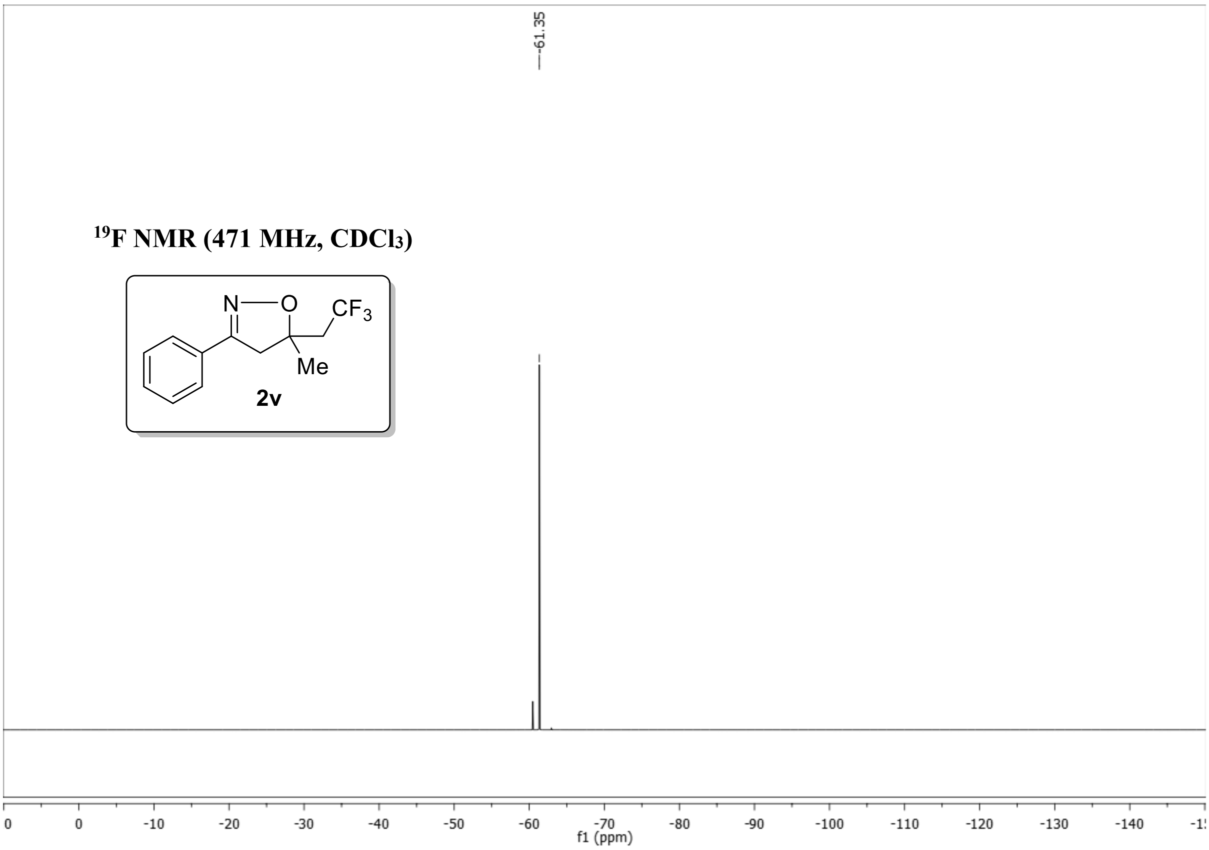

19F NMR (471 MHz, CDCl3)
2u
N
O
CF3
F
-60.44
-114.02
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
f1 (ppm)



^{1}H NMR (500 MHz, CDCl$_3$)

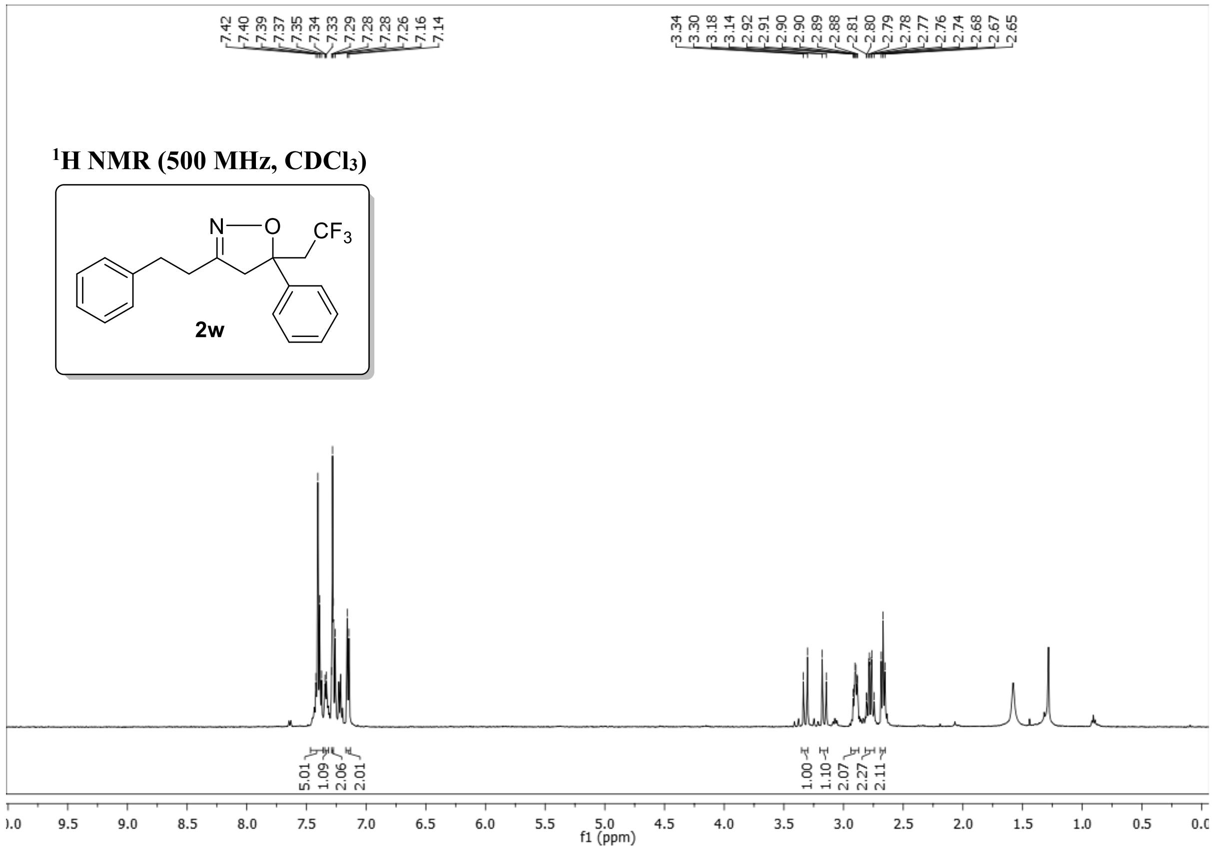


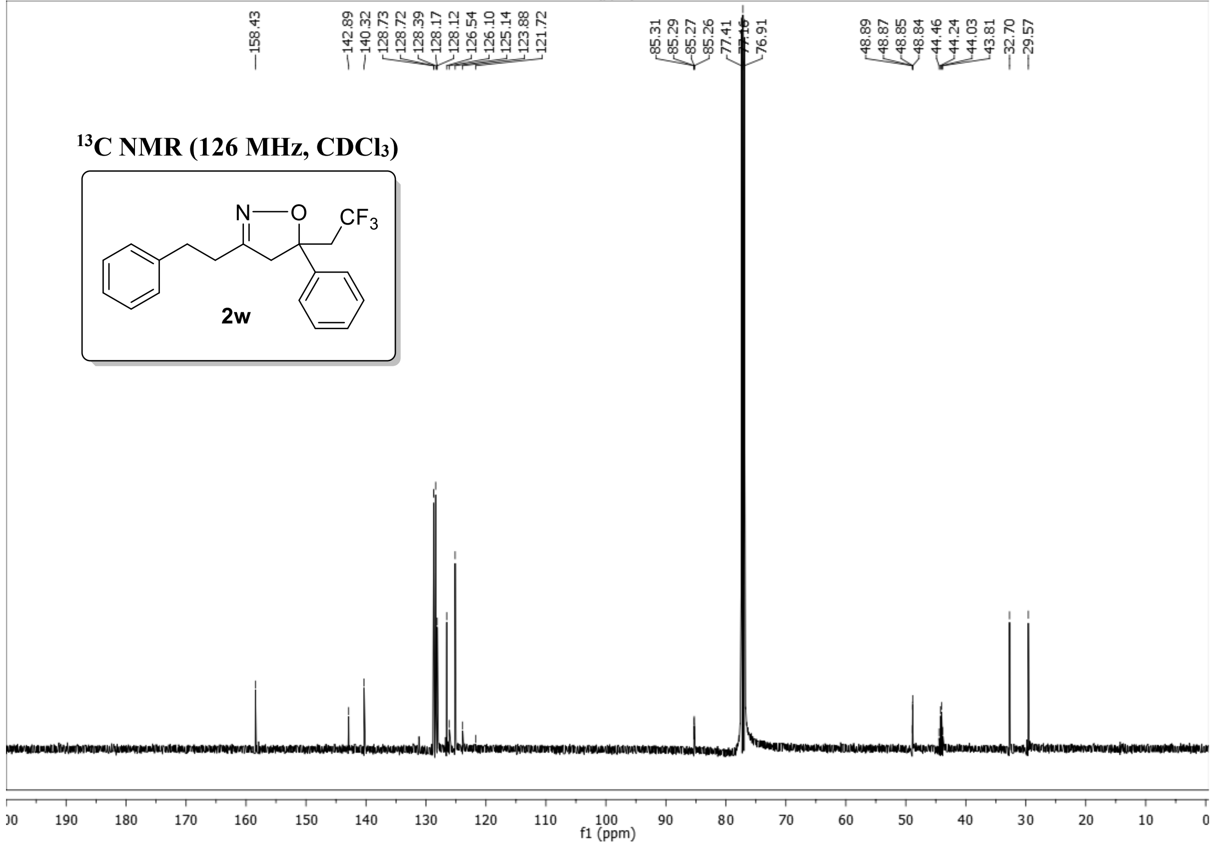
^{13}C NMR (126 MHz, CDCl$_3$)

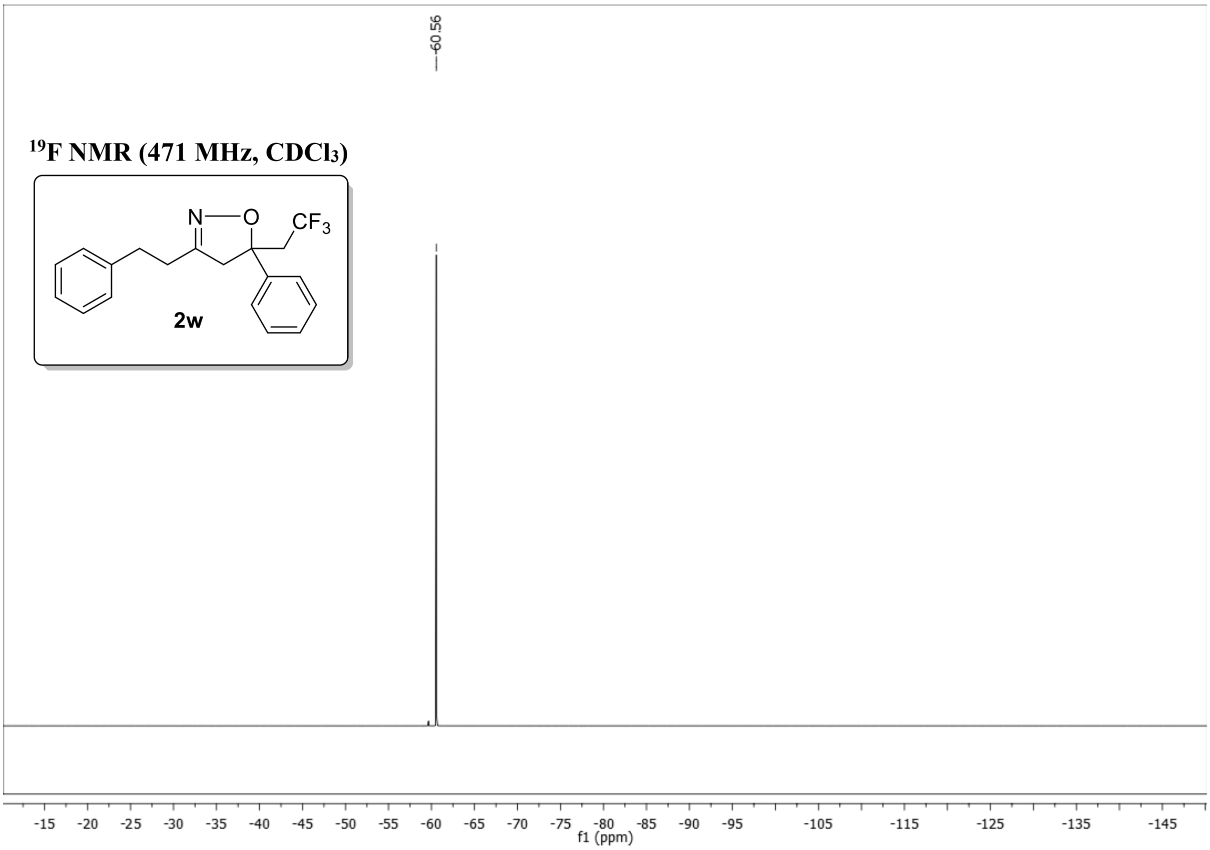


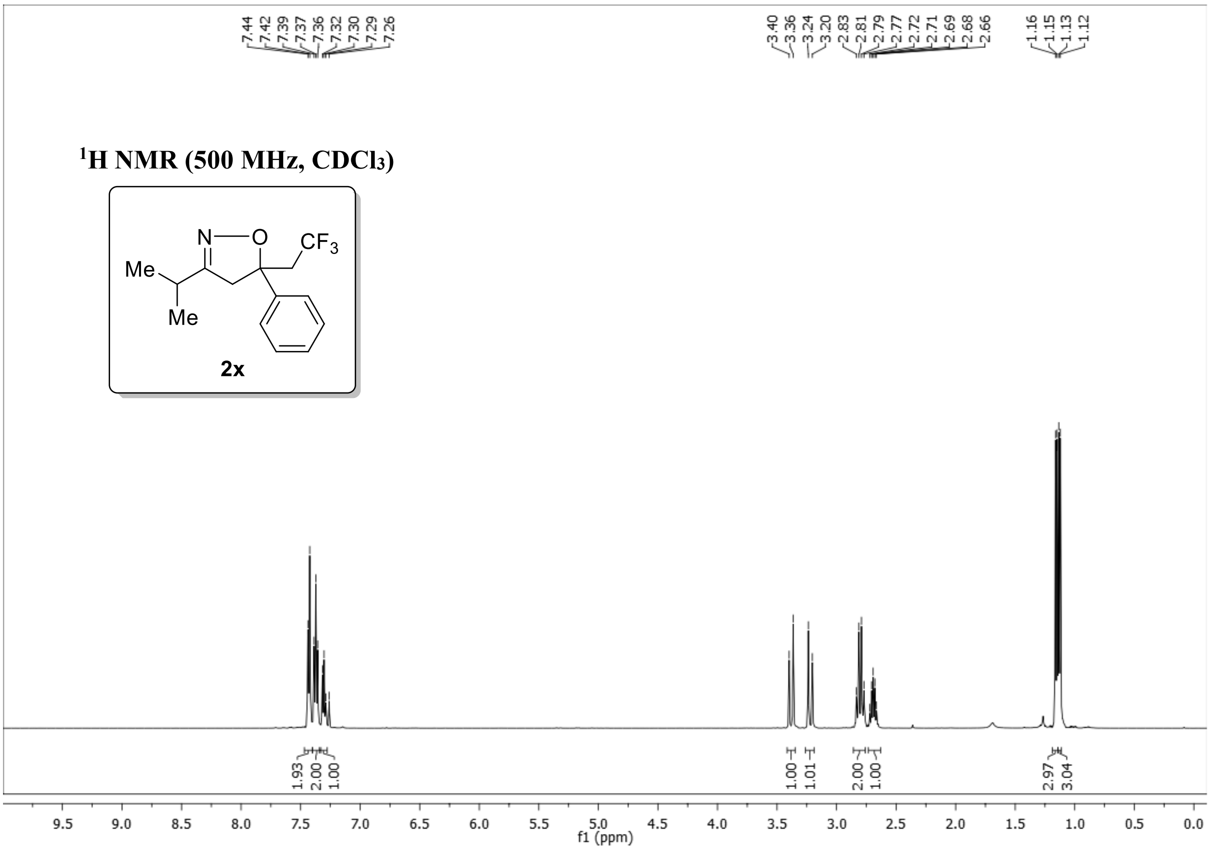
^{19}F NMR (471 MHz, $CDCl_3$)

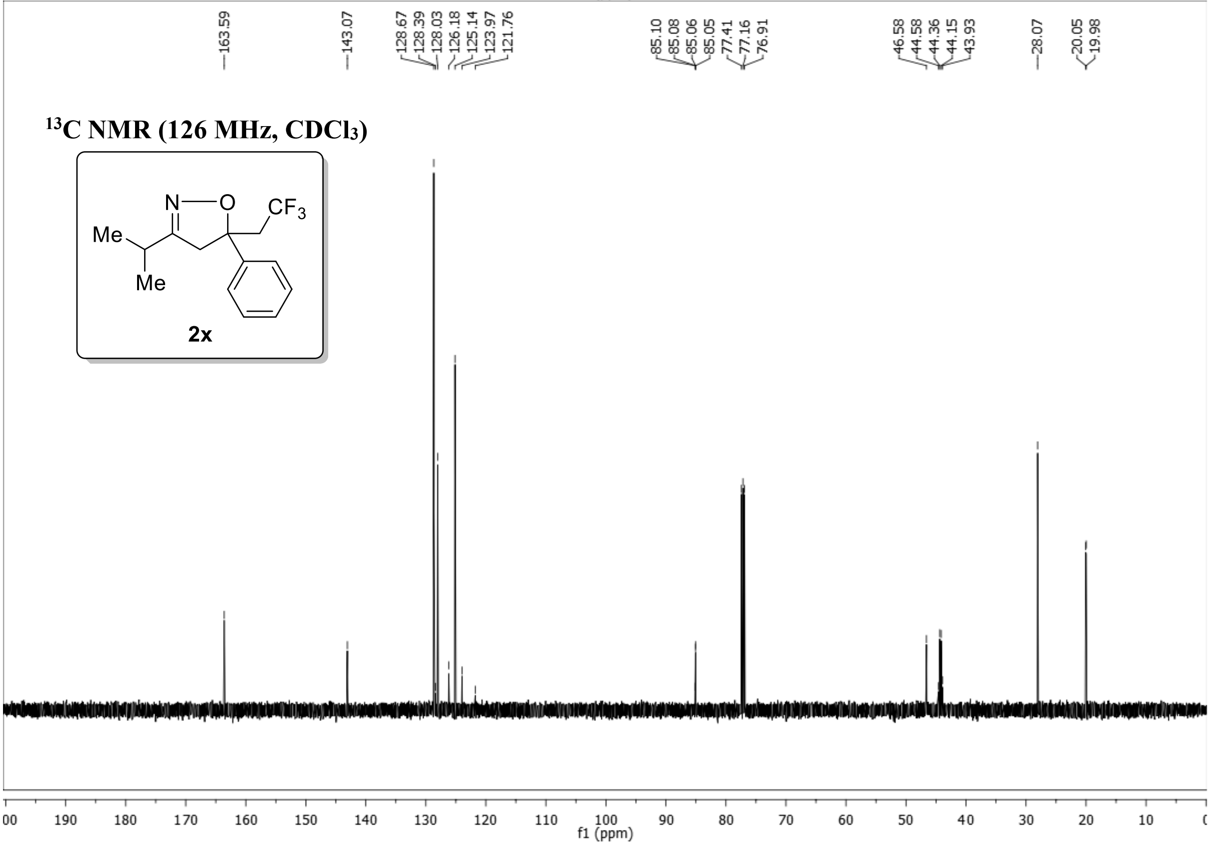
2v

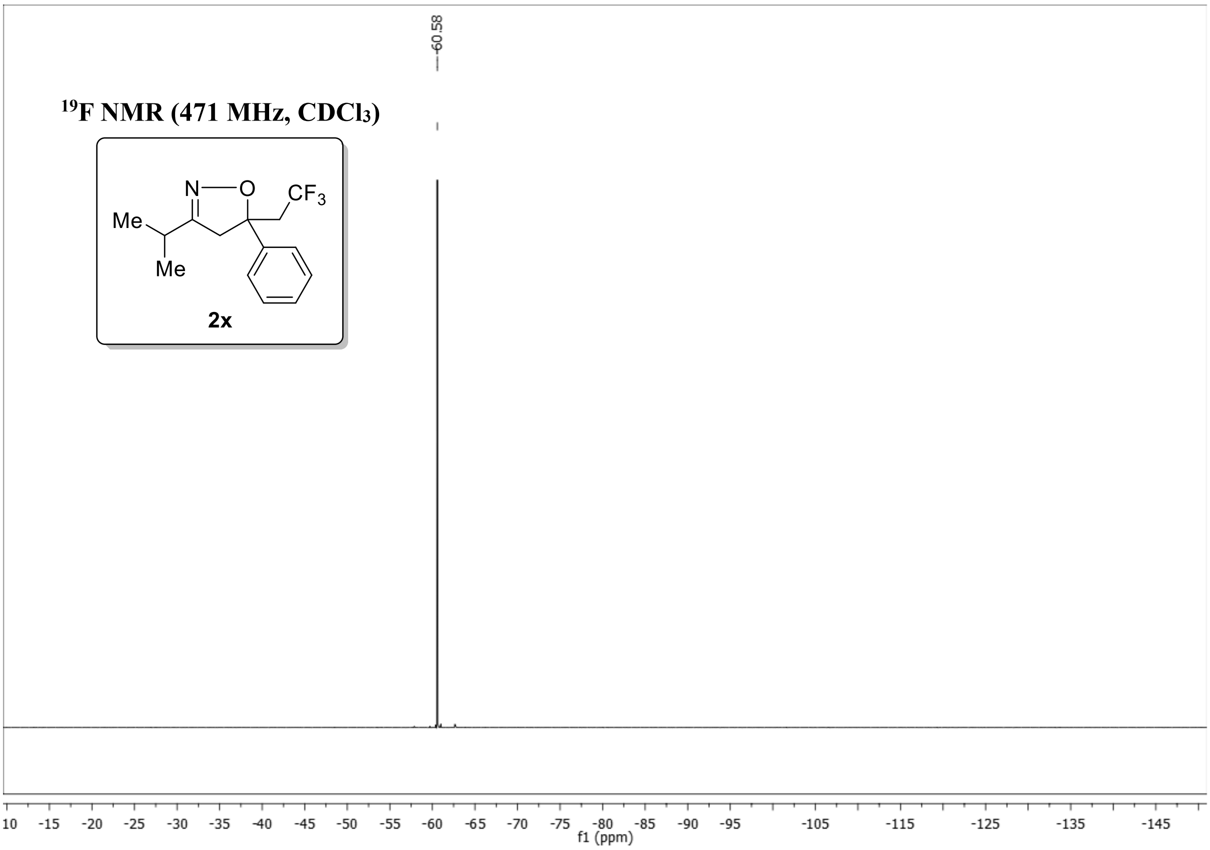

7.42
7.40
7.39
7.37
7.35
7.34
7.33
7.29
7.28
7.28
7.26
7.16
7.14
3.34
3.30
3.18
3.14
2.92
2.91
2.90
2.90
2.89
2.88
2.81
2.80
2.79
2.78
2.77
2.76
2.74
2.68
2.67
2.65
^{1}H NMR (500 MHz, CDCl$_3$)
N
O
CF$_3$
2w
5.01
1.09
2.06
2.01
1.00
1.10
2.07
2.27
2.11
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


158.43
142.89
140.32
128.73
128.72
128.39
128.17
128.12
126.54
126.10
125.14
123.88
121.72
85.31
85.29
85.27
85.26
77.41
77.16
76.91
48.89
48.87
48.85
48.84
44.46
44.24
44.03
43.81
32.70
29.57
^{13}C NMR (126 MHz, CDCl$_3$)
N
O
CF$_3$
2w
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

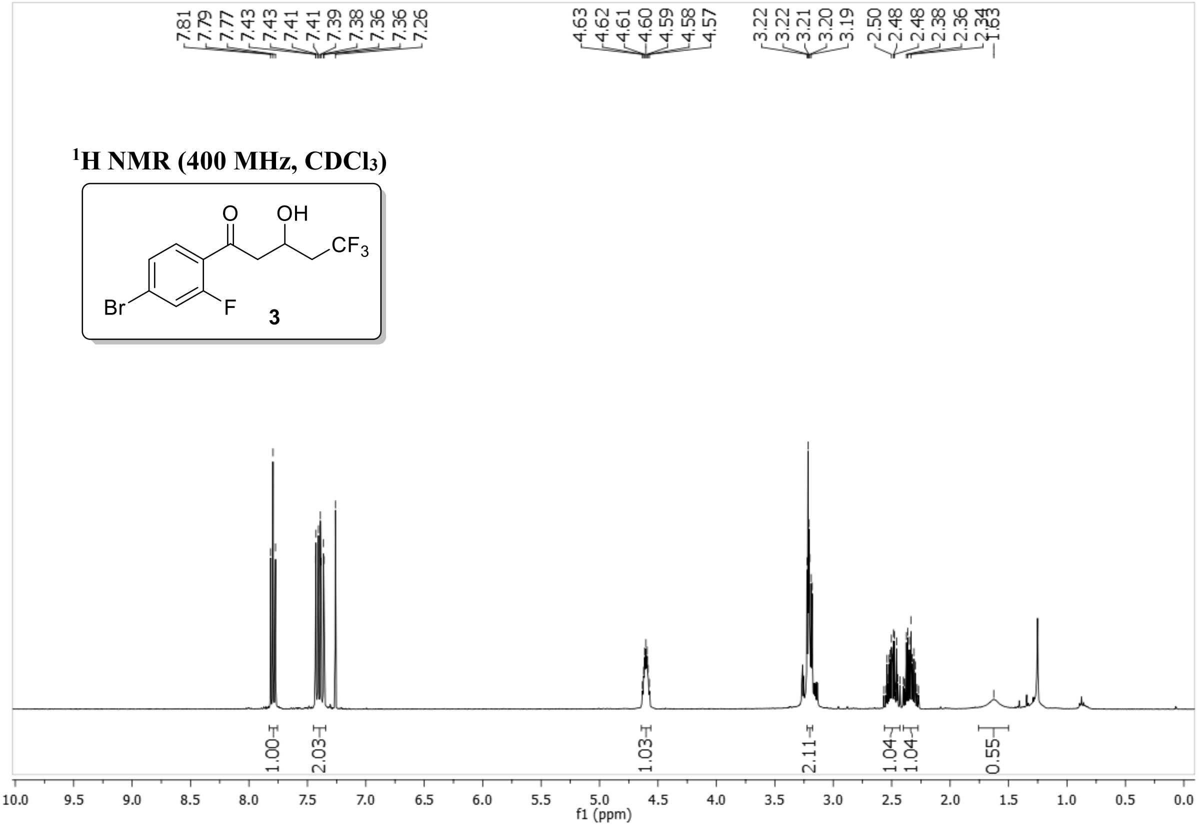

-60.56
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
2w
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-105
-115
-125
-135
-145
f1 (ppm)

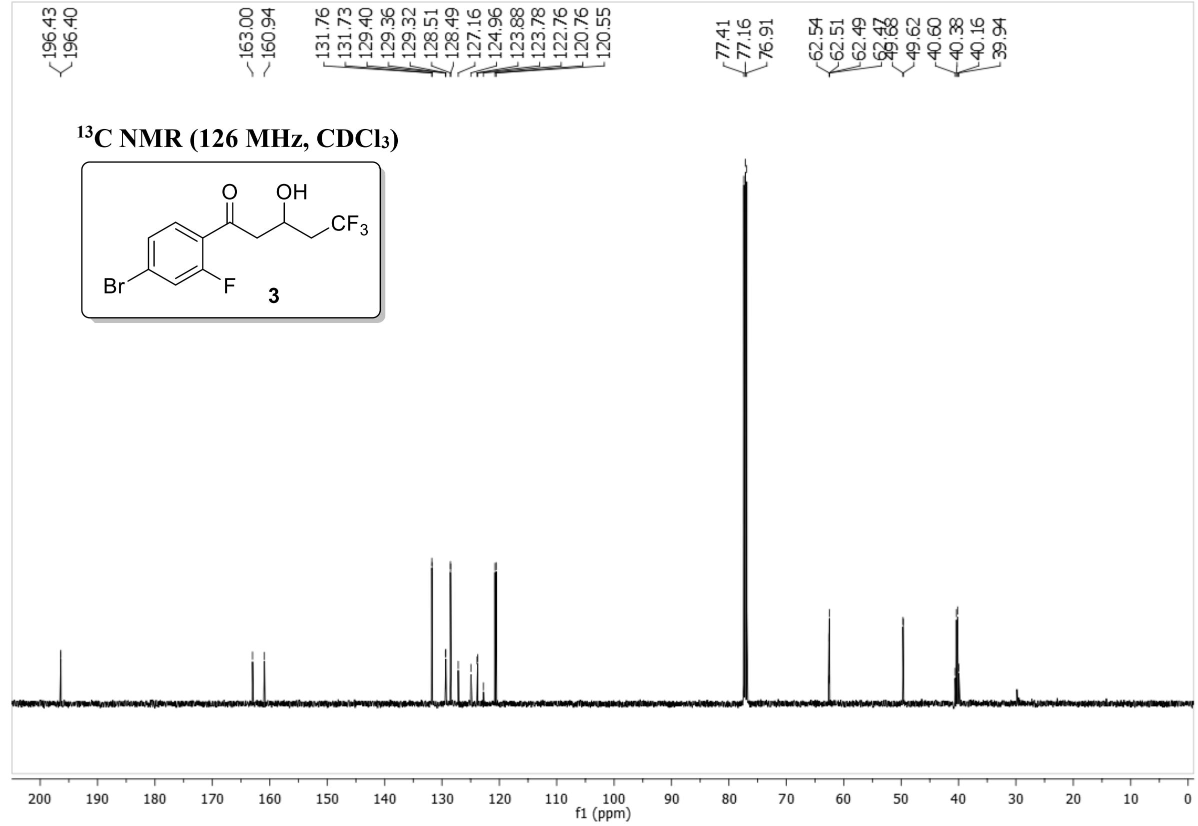

7.44
7.42
7.39
7.37
7.36
7.32
7.30
7.29
7.26
3.40
3.36
3.24
3.20
2.83
2.81
2.79
2.77
2.72
2.71
2.69
2.68
2.66
1.16
1.15
1.13
1.12
^{1}H NMR (500 MHz, CDCl$_3$)
N
O
CF$_3$
Me
Me
2x
1.93
2.00
1.00
1.00
1.01
2.00
1.00
2.97
3.04
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

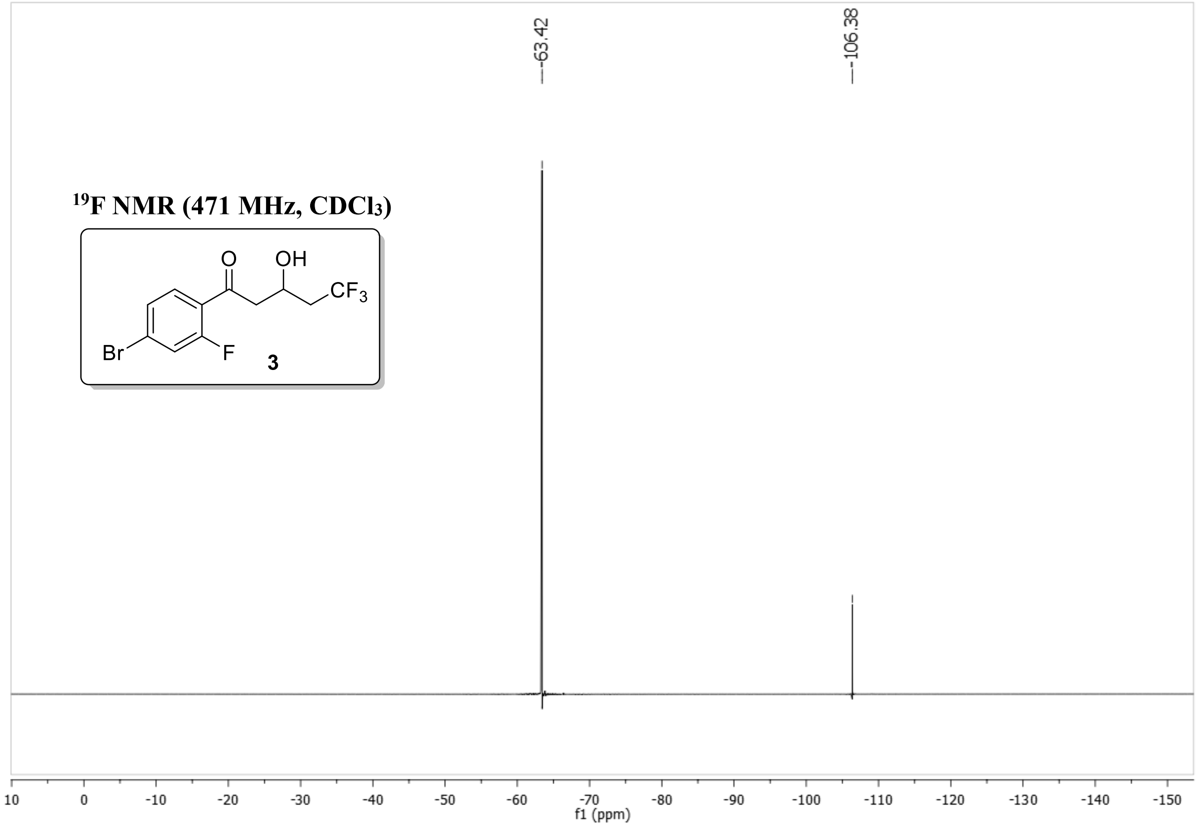

163.59
143.07
128.67
128.39
128.03
126.18
125.14
123.97
121.76
85.10
85.08
85.06
85.05
77.41
77.16
76.91
46.58
44.58
44.36
44.15
43.93
28.07
20.05
19.98
^{13}C NMR (126 MHz, CDCl$_3$)
N
O
CF$_3$
Me
Me
2x
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

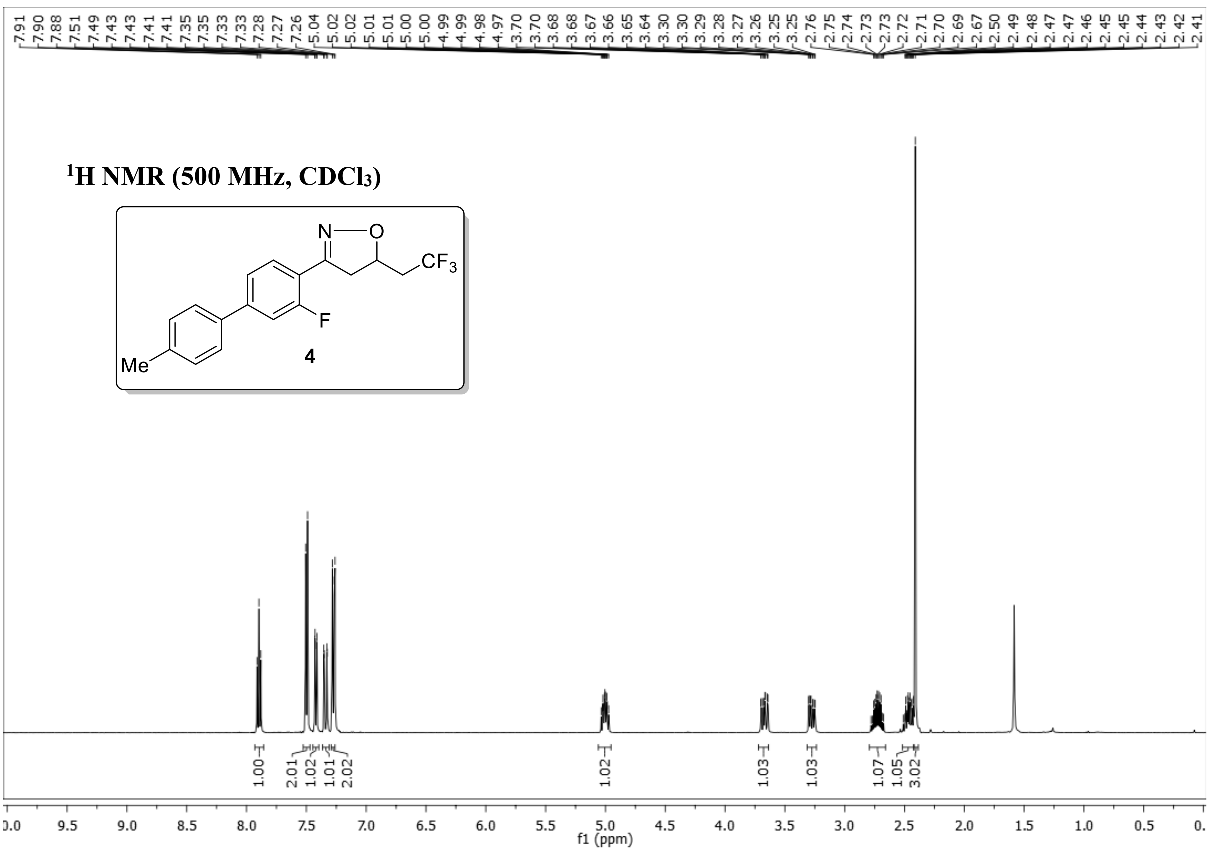


-60.58
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
Me
Me
2x
10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-105
-115
-125
-135
-145
f1 (ppm)

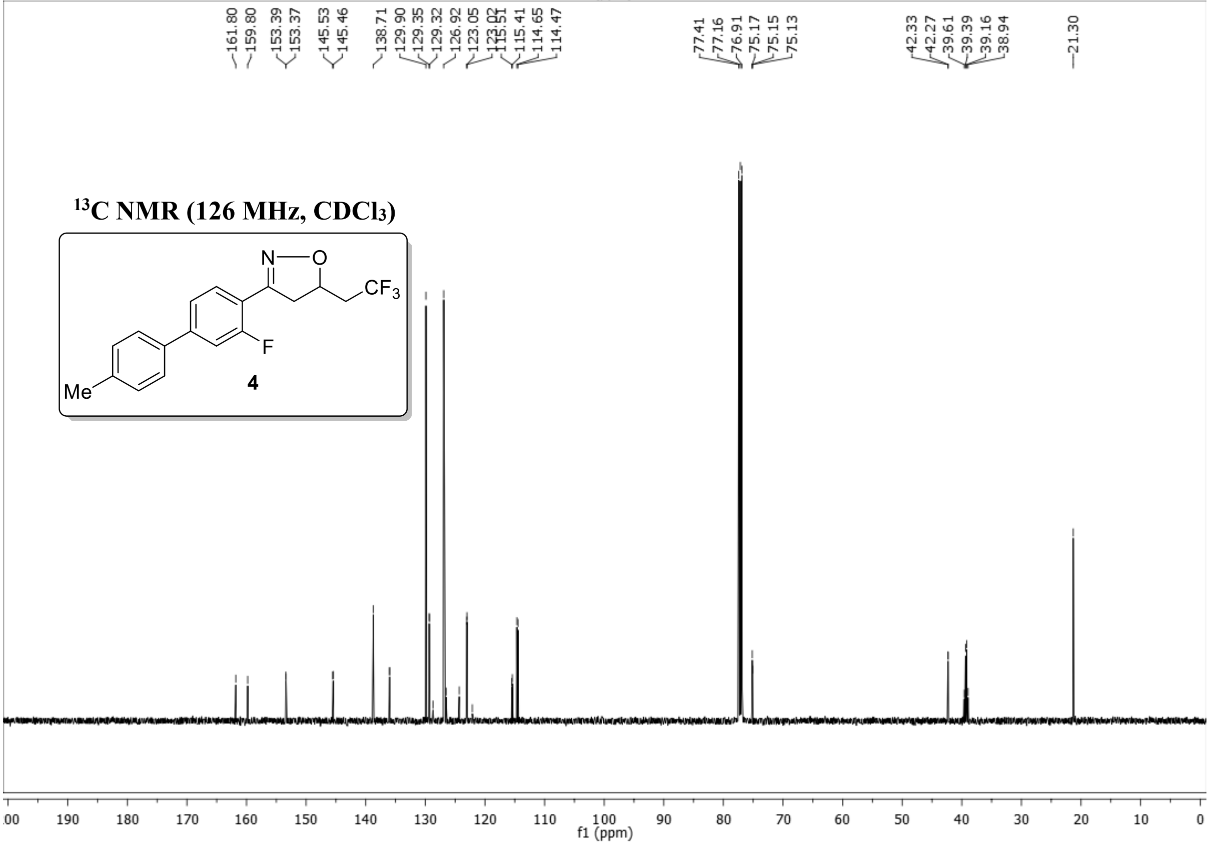

7.81
7.79
7.77
7.43
7.43
7.41
7.41
7.39
7.38
7.36
7.36
7.26
4.63
4.62
4.61
4.60
4.59
4.58
4.57
3.22
3.22
3.21
3.20
3.19
2.50
2.48
2.48
2.38
2.36
2.34
1.63
1H NMR (400 MHz, CDCl3)
O
OH
CF3
Br
F
3
1.00
2.03
1.03
2.11
1.04
1.04
0.55
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


196.43
196.40
163.00
160.94
131.76
131.73
129.40
129.36
129.32
128.51
128.49
127.16
124.96
123.88
123.78
122.76
120.76
120.55
77.41
77.16
76.91
62.54
62.51
62.49
62.47
49.68
49.62
40.60
40.38
40.16
39.94
13C NMR (126 MHz, CDCl3)
O
OH
CF3
Br
F
3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

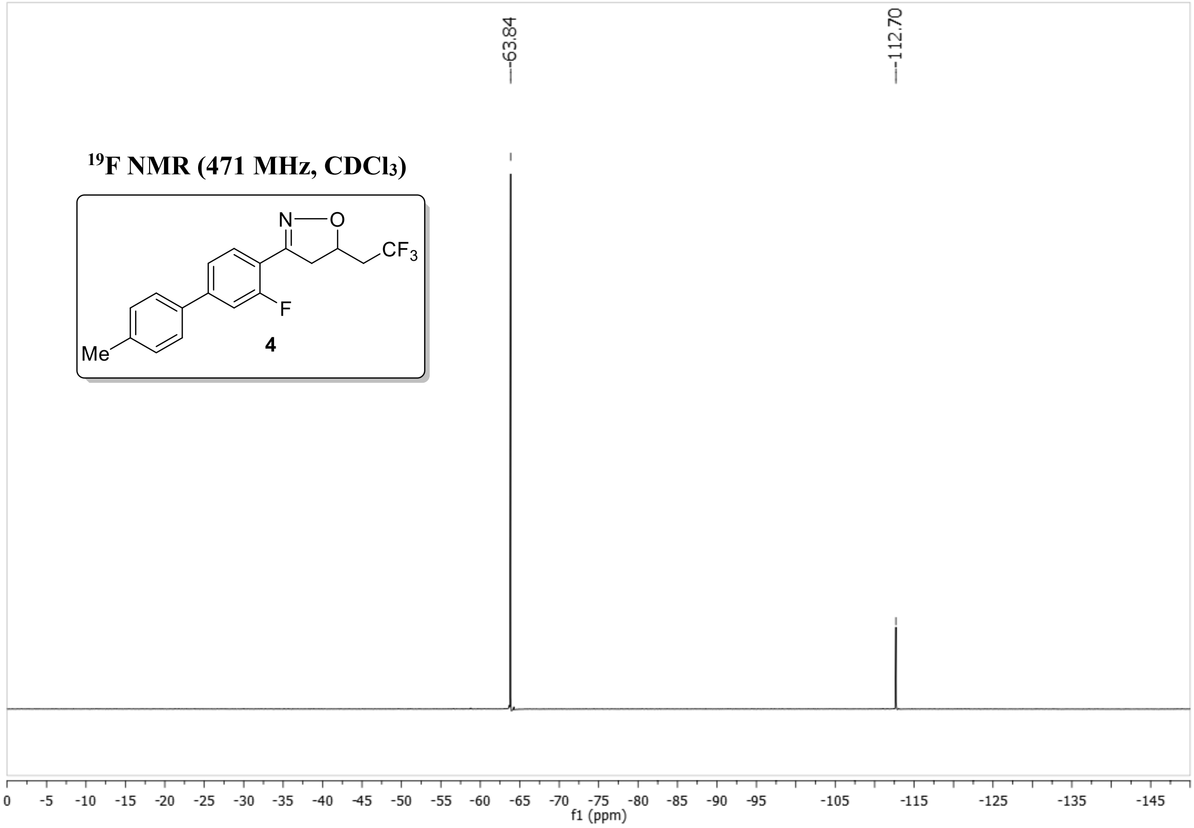

-63.42
-106.38
^{19}F NMR (471 MHz, CDCl$_3$)
O
OH
CF$_3$
Br
F
3
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)



^{1}H NMR (500 MHz, CDCl$_3$)



^{13}C NMR (126 MHz, CDCl$_3$)


-63.84
-112.70
^{19}F NMR (471 MHz, CDCl$_3$)
N
O
CF$_3$
F
Me
4
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-105
-115
-125
-135
-145
f1 (ppm)