Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Electrophilic *N*-Trifluoromethylthiophthalimide as a Fluorinated Reagent in the Synthesis of Acyl Fluorides

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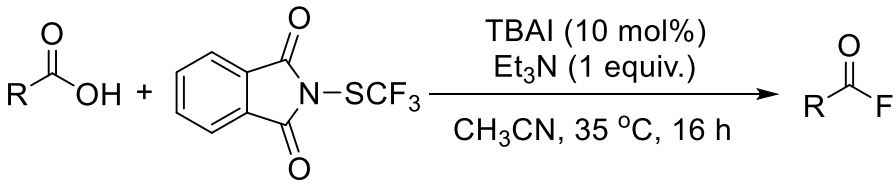
1. General Information

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents for chromatography were HPLC grade. Anhydrous and degassed CH_3CN used in reactions was purchased from Sigma-Aldrich in Sure/Seal™ bottle. Analytical thin-layer chromatography (TLC) was performed on Merck silica gel aluminium plates with F-254 indicator, visualized by irradiation with UV light. Column chromatography was performed on silica gel (particle size 0.043–0.063 mm) by using Interchim PuriFlash®430 automatic purification system. ^{1}H-NMR and ^{13}C-NMR were recorded on Bruker DRX-500 and AMX-400 instruments in $CDCl_3$ and are reported relative to the solvent residual peaks. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated s (singlet), bs (broad singlet), d (doublet), t (triplet), m (multiplet); coupling constants (*J*) are in Hertz (Hz). Mass spectra (EI-MS, 70 eV) were conducted on a Agilent 7890 gas chromatograph equipped with 5975C EI-MSD Triple-Axis Detector using DB5MS and HP5MS columns. HRMS analysis was performed using a Thermo LTQ Velos Orbitrap mass spectrometer (Thermo Scientific, Pittsburgh, PA, USA) equipped with an ESI source.

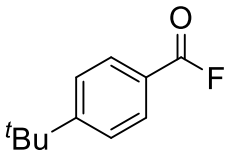
2. General Procedure for the Deoxygenative Fluorination of Carboxylic Acids



A dry 10 mL vial equipped with a stirring bar was charged with *N*-trifluoromethylthiophthalimide (0.2 mmol, 1 equiv.), carboxylic acid (0.4 mmol, 2 equiv.), TBAI (7.4 mg, 0.02 mmol, 10 mol%) in glovebox. Anhydrous and degassed CH_3CN (2.0 mL) and Et_3N (28 uL, 1 equiv.) was added via syringe. The reaction mixture was stirred at 35 ºC for 16 h. After the reaction is completed, the mixture was concentrated under vacuum and the product was purified by flash column chromatography on silica gel using hexane/EtOAc as eluent.

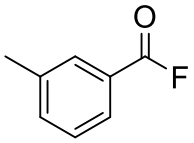
3. Spectroscopic Data of the Products

4-(*tert*-butyl)benzoyl fluoride (3a)



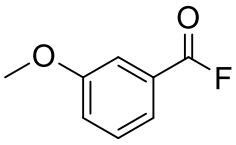
Yield: 89% (32.0 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.4 Hz, 2H), 7.56 (d, *J* = 8.1 Hz, 2H), 1.38 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.5, 157.5 (d, *J* = 342.9 Hz), 131.4 (d, *J* = 4.0 Hz), 126.1, 122.0 (d, *J* = 60.9 Hz), 35.4, 31.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ 17.66. Data in accordance with the literature. [1]

3-methylbenzoyl fluoride (3b)



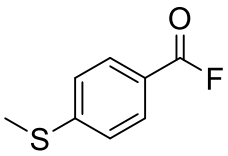
Yield: 83% (22.9 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.5 Hz, 2H), 7.53 (d, *J* = 7.7 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 1H), 2.46 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.6 (d, *J* = 344.4 Hz), 139.1, 136.1, 131.9, 128.9, 128.6, 124.9 (d, *J* = 60.1 Hz), 21.2. ^{19}F NMR (377 MHz, $CDCl_3$) δ 18.25. Data in accordance with the literature.[1]

3-methoxybenzoyl fluoride (3c)



Yield: 97% (29.9 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.6 Hz, 1H), 7.55 (s, 1H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.26 (dd, *J* = 8.3, 2.6 Hz, 1H), 3.89 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.9, 157.3 (d, *J* = 344.6 Hz), 130.1, 126.1 (d, *J* = 60.9 Hz), 123.9 (d, *J* = 3.4 Hz), 122.0, 115.5 (d, *J* = 4.3 Hz), 55.6. ^{19}F NMR (471 MHz, $CDCl_3$) δ 18.60. Data in accordance with the literature. [2]

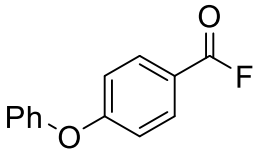
4-(methylthio)benzoyl fluoride (3d)



Yield: 91% (30.9 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.5 Hz, 2H), 7.33 (d, *J* = 8.3 Hz, 2H), 2.56 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.3 (d, *J* = 341.3 Hz), 149.4, 131.6 (d, *J* = 3.8 Hz), 125.1,

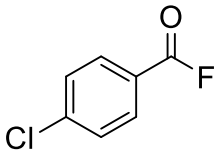
120.5 (d, J = 61.8 Hz), 14.6. ^{19}F NMR (471 MHz, $CDCl_3$) δ 16.63. Data in accordance with the literature.[2]

4-phenoxybenzoyl fluoride (3e)



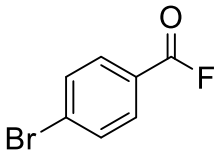
Yield: 92% (39.7 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 (d, J = 8.8 Hz, 2H), 7.46 (t, J = 8.0 Hz, 2H), 7.28 (t, J = 7.4 Hz, 1H), 7.12 (d, J = 7.7 Hz, 2H), 7.05 (d, J = 8.7 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.1, 157.0 (d, J = 340.9 Hz), 154.7, 133.8 (d, J = 4.0 Hz), 130.3, 125.3, 120.6, 118.5 (d, J = 62.0 Hz), 117.4. ^{19}F NMR (377 MHz, $CDCl_3$) δ 16.79. Data in accordance with the literature.[3]

4-chlorobenzoyl fluoride (3f)



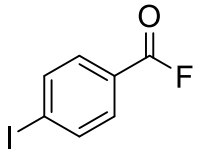
Yield: 44% (14.0 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (d, J = 8.6 Hz, 2H), 7.54 (d, J = 8.6 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.6 (d, J = 343.4 Hz), 142.2, 132.7 (d, J = 3.8 Hz), 129.6, 123.4 (d, J = 62.5 Hz). ^{19}F NMR (471 MHz, $CDCl_3$) δ 18.43. Data in accordance with the literature.[4]

4-bromobenzoyl fluoride (3g)



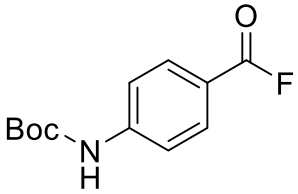
Yield: 62% (25.3 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, J = 8.5 Hz, 2H), 7.71 (d, J = 8.6 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.8 (d, J = 343.7 Hz), 132.7 (d, J = 3.8 Hz), 132.6, 131.0, 123.8 (d, J = 62.5 Hz). ^{19}F NMR (471 MHz, $CDCl_3$) δ 18.43. Data in accordance with the literature.[1]

4-iodobenzoyl fluoride (3h)



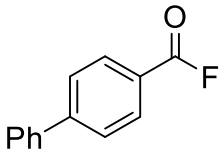
Yield: 50% (25.1 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, J = 7.7 Hz, 8H), 7.76 (d, J = 8.5 Hz, 7H), 7.28 (s, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.1 (d, J = 343.9 Hz), 138.6, 132.5 (d, J = 3.8 Hz), 124.4 (d, J = 62.2 Hz), 104.0. ^{19}F NMR (471 MHz, $CDCl_3$) δ 18.28. Data in accordance with the literature.[5]

***Tert*-butyl (4-(fluorocarbonyl)phenyl)carbamate (3i)**



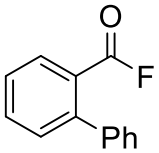
Yield: 99% (47.2 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.7 Hz, 2H), 7.54 (d, *J* = 8.5 Hz, 2H), 1.54 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.2 (d, *J* = 340.6 Hz), 152.0, 145.1, 133.0 (d, *J* = 4.0 Hz), 118.4 (d, *J* = 61.8 Hz), 117.7, 81.8, 28.2. ^{19}F NMR (377 MHz, $CDCl_3$) δ 16.21. HRMS (FDMS) for $C_{12}H_{14}FNO_3$: calculated for $[M]^+$ 239.0952, found 239.0995.

[1,1'-biphenyl]-4-carbonyl fluoride (3j)



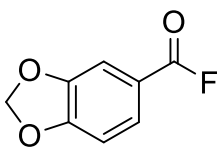
Yield: 52% (20.8 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.3 Hz, 2H), 7.77 (d, *J* = 7.9 Hz, 2H), 7.66 (d, *J* = 7.2 Hz, 2H), 7.52 (t, *J* = 7.4 Hz, 2H), 7.47 (t, *J* = 7.3 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.4 (d, *J* = 343.2 Hz), 148.1, 139.3, 132.0 (d, *J* = 3.9 Hz), 129.1, 128.8, 127.7, 127.4, 123.5 (d, *J* = 61.2 Hz). ^{19}F NMR (471 MHz, $CDCl_3$) δ 18.14. Data in accordance with the literature.[1]

[1,1'-biphenyl]-2-carbonyl fluoride (3k)



Yield: 91% (36.4 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 (d, *J* = 7.9 Hz, 1H), 7.71 (t, *J* = 7.6 Hz, 1H), 7.53 (t, *J* = 7.7 Hz, 1H), 7.46 (q, *J* = 7.2, 6.5 Hz, 4H), 7.38 – 7.35 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.6 (d, *J* = 348.1 Hz), 145.5 (d, *J* = 2.5 Hz), 140.1, 133.9, 132.2 (d, *J* = 2.8 Hz), 131.8 (d, *J* = 2.8 Hz), 128.4, 128.3, 127.9, 127.6, 124.2 (d, *J* = 56.9 Hz). ^{19}F NMR (471 MHz, $CDCl_3$) δ 35.04. Data in accordance with the literature.[1]

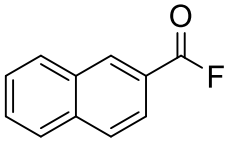
Benzo[d][1,3]dioxole-5-carbonyl fluoride (3l)



Yield: 98% (33.0 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.43 (d, *J* = 1.6 Hz, 1H), 6.92 (d, *J* = 8.2 Hz, 1H), 6.12 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.9 (d, *J* = 340.1 Hz),

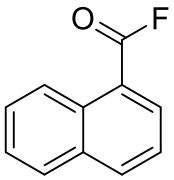
153.8, 148.3 (d, J = 2.0 Hz), 128.2 (d, J = 3.9 Hz), 118.4 (d, J = 62.5 Hz), 110.7 (d, J = 4.2 Hz), 108.6, 102.4. ^{19}F NMR (471 MHz, $CDCl_3$) δ 16.40. Data in accordance with the literature.[6]

2-naphthoyl fluoride (3m)



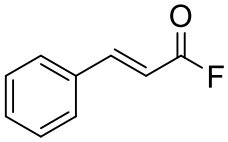
Yield: 95% (33.1 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.65 (s, 1H), 8.02 (d, J = 17.8 Hz, 2H), 7.99 – 7.92 (m, 2H), 7.71 (t, J = 7.5 Hz, 1H), 7.64 (t, J = 7.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.7 (d, J = 343.6 Hz), 136.5, 134.0 (d, J = 3.2 Hz), 132.3, 129.71, 129.66, 129.1, 128.0, 127.4, 125.6 (d, J = 4.2 Hz), 122.0 (d, J = 60.5 Hz). ^{19}F NMR (471 MHz, $CDCl_3$) δ 18.08. Data in accordance with the literature.[1]

1-naphthoyl fluoride (3n)



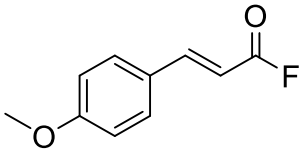
Yield: 92% (32.0 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.05 (d, J = 8.7 Hz, 1H), 8.38 (d, J = 7.4 Hz, 1H), 8.20 (d, J = 8.2 Hz, 1H), 7.97 (d, J = 8.2 Hz, 1H), 7.75 (t, J = 7.2 Hz, 1H), 7.64 (t, J = 7.3 Hz, 1H), 7.59 (t, J = 7.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.4 (d, J = 344.5 Hz), 136.7, 133.9 (d, J = 4.0 Hz), 133.7 (d, J = 2.0 Hz), 132.1 (d, J = 7.2 Hz), 129.2, 129.0, 127.0, 125.2, 124.5, 120.3 (d, J = 55.8 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ 29.91. Data in accordance with the literature.[1]

Cinnamoyl fluoride (5a)



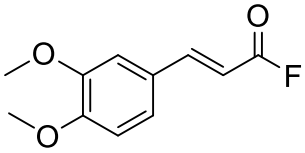
Yield: 72% (21.6 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, J = 16.0 Hz, 1H), 7.59 (dd, J = 7.9, 1.7 Hz, 2H), 7.54 – 7.43 (m, 3H), 6.40 (dd, J = 16.0, 7.3 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.1 (d, J = 338.5 Hz), 151.4 (d, J = 6.1 Hz), 133.1, 131.9, 129.2, 128.8, 112.1 (d, J = 67.2 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ 25.61. Data in accordance with the literature.[7]

(*E*)-3-(4-methoxyphenyl)acryloyl fluoride (5b)



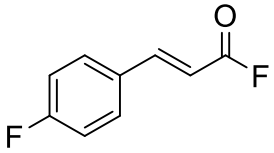
Yield: 97% (34.8 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 15.8 Hz, 1H), 7.54 (d, *J* = 8.8 Hz, 2H), 6.96 (d, *J* = 8.8 Hz, 2H), 6.23 (dd, *J* = 15.9, 7.3 Hz, 1H), 3.88 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.7, 157.6 (d, *J* = 336.6 Hz), 151.1 (d, *J* = 6.3 Hz), 130.7, 125.9, 114.6, 109.1 (d, *J* = 67.1 Hz), 55.5. ^{19}F NMR (377 MHz, $CDCl_3$) δ 24.39. Data in accordance with the literature.[7]

(*E*)-3-(3,4-dimethoxyphenyl)acryloyl fluoride (5c)



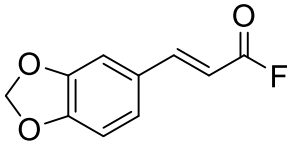
Yield: 93% (39.1 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 15.8 Hz, 1H), 7.17 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.07 (d, *J* = 1.7 Hz, 1H), 6.91 (d, *J* = 8.3 Hz, 1H), 6.23 (dd, *J* = 15.8, 7.3 Hz, 1H), 3.94 (d, *J* = 3.5 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.5 (d, *J* = 336.5 Hz), 152.5, 151.4 (d, *J* = 6.2 Hz), 149.4, 126.1, 124.0, 111.1, 109.9, 109.3 (d, *J* = 67.2 Hz), 56.1, 56.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ 24.49. Data in accordance with the literature.[8]

(*E*)-3-(4-fluorophenyl)acryloyl fluoride (5d)



Yield: 60% (20.2 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 (d, *J* = 16.0 Hz, 1H), 7.60 (dd, *J* = 8.7, 5.3 Hz, 2H), 7.16 (t, *J* = 8.6 Hz, 2H), 6.32 (d, *J* = 23.0 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.8 (d, *J* = 254.1 Hz), 157.0 (d, *J* = 338.3 Hz), 150.0 (d, *J* = 6.1 Hz), 130.8 (d, *J* = 8.8 Hz), 129.5 (d, *J* = 3.3 Hz), 116.5 (d, *J* = 22.1 Hz), 111.9 (dd, *J* = 67.8, 2.5 Hz). ^{19}F NMR (471 MHz, $CDCl_3$) δ 25.61, -106.62. GC-MS (EI): m/z = 168.2 (M^+).

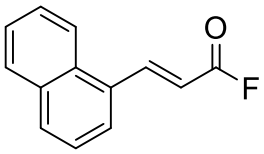
(*E*)-3-(benzo[d][1,3]dioxol-5-yl)acryloyl fluoride (5e)



Yield: 90% (35.0 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 15.8 Hz, 1H), 7.11 – 7.04 (m, 2H),

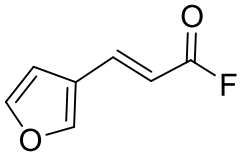
6.87 (d, *J* = 8.2 Hz, 1H), 6.18 (dd, *J* = 15.8, 7.2 Hz, 1H), 6.07 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.4 (d, *J* = 336.9 Hz), 151.1, 151.0, 148.7, 127.6, 126.1, 109.7 (d, *J* = 67.3 Hz), 108.8, 106.7, 102.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ 24.77. Data in accordance with the literature.[9]

(*E*)-3-(naphthalen-1-yl)acryloyl fluoride (5f)



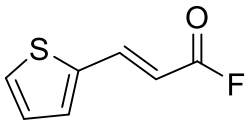
Yield: 75% (30.1 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (d, *J* = 15.7 Hz, 1H), 8.18 (d, *J* = 8.5 Hz, 1H), 8.01 (d, *J* = 8.2 Hz, 1H), 7.94 (d, *J* = 7.5 Hz, 1H), 7.84 (d, *J* = 7.2 Hz, 1H), 7.68 – 7.52 (m, 3H), 6.50 (dd, *J* = 15.7, 7.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.0 (d, *J* = 339.0 Hz), 148.3 (d, *J* = 6.3 Hz), 133.7, 132.2, 131.3, 130.2, 129.0, 127.6, 126.6, 125.9, 125.4, 122.9, 114.3 (d, *J* = 66.8 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ 25.96. Data in accordance with the literature.[10]

(*E*)-3-(furan-3-yl)acryloyl fluoride (5g)



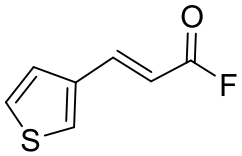
Yield: 60% (16.9 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 15.3 Hz, 2H), 7.51 (s, 1H), 6.65 (s, 1H), 6.10 (dd, *J* = 15.8, 7.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.1 (d, *J* = 337.3 Hz), 146.3, 145.1, 141.4 (d, *J* = 6.3 Hz), 122.2, 111.7 (d, *J* = 67.6 Hz), 107.2. ^{19}F NMR (377 MHz, $CDCl_3$) δ 24.17. Data in accordance with the literature.[11]

(*E*)-3-(thiophen-2-yl)acryloyl fluoride (5h)



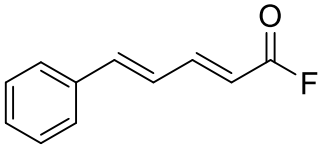
Yield: 70% (21.9 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 15.6 Hz, 1H), 7.55 (d, *J* = 5.0 Hz, 1H), 7.40 (d, *J* = 3.6 Hz, 1H), 7.18 – 7.11 (m, 1H), 6.16 (dd, *J* = 15.6, 7.3 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.1 (d, *J* = 336.4 Hz), 143.4 (d, *J* = 6.5 Hz), 138.3, 133.3, 131.1, 128.6, 110.3 (d, *J* = 68.5 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ 24.30. Data in accordance with the literature.[12]

(*E*)-3-(thiophen-3-yl)acryloyl fluoride (5i)



Yield: 64% (20.1 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 15.8 Hz, 1H), 7.68 (d, *J* = 2.5 Hz, 1H), 7.42 (dd, *J* = 5.0, 3.0 Hz, 1H), 7.35 (d, *J* = 5.1 Hz, 1H), 6.20 (dd, *J* = 15.8, 7.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.5 (d, *J* = 337.6 Hz), 144.5 (d, *J* = 6.2 Hz), 136.6, 131.0, 127.8, 125.1, 111.5 (d, *J* = 67.3 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ 24.70. Data in accordance with the literature.[12]

(2*E*,4*E*)-5-phenylpenta-2,4-dienoyl fluoride (5j)



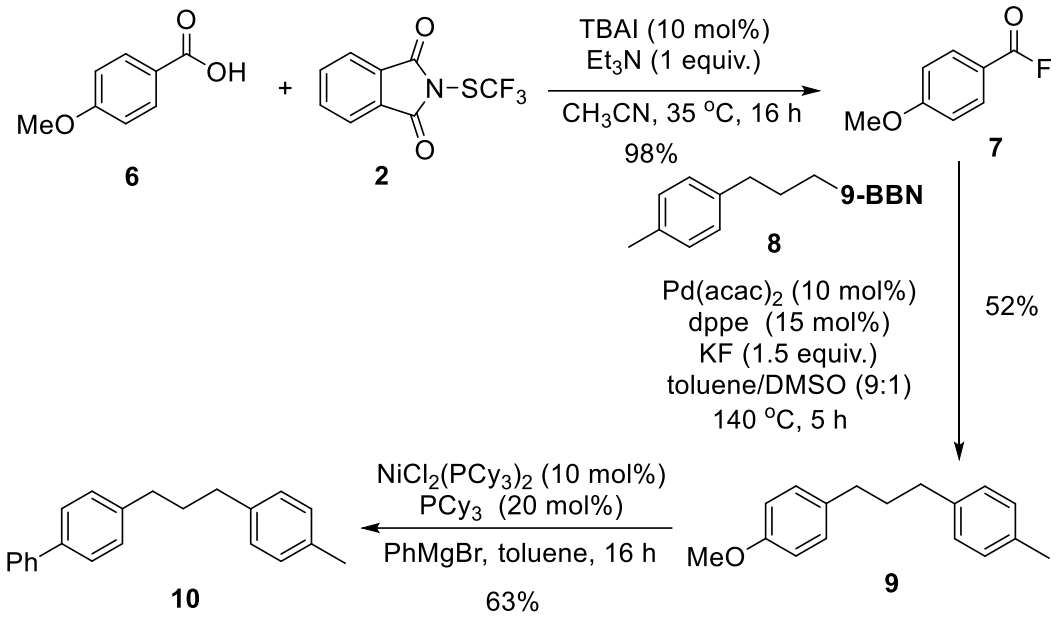
Yield: 64% (22.4 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (dd, *J* = 15.2, 10.9 Hz, 1H), 7.56 – 7.50 (m, 2H), 7.42 (q, *J* = 5.3 Hz, 3H), 7.07 (d, *J* = 15.6 Hz, 1H), 6.95 (dd, *J* = 15.5, 10.9 Hz, 1H), 5.94 (dd, *J* = 15.2, 8.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.2 (d, *J* = 337.2 Hz), 151.2 (d, *J* = 6.0 Hz), 144.2, 135.3, 130.1, 129.0, 127.7, 125.2, 114.6 (d, *J* = 67.0 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ 24.46. Data in accordance with the literature.[13]

4. Gram-Scale Reaction and Synthetic Application

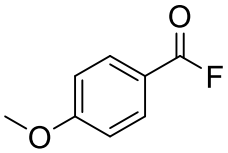
4.1 Gram-Scale Reaction

A dry 100 mL flask equipped with a stirring bar was charged with N-trifluoromethylthiophthalimide (5.0 mmol, 1.23 g, 1 equiv.), carboxylic acid **1i** (10 mmol, 2.37 g, 2 equiv.), TBAI (185.0 mg, 0.5 mmol, 10 mol%) in glovebox. Anhydrous and degassed CH_3CN (50 mL) and Et_3N (0.7 mL, 1 equiv.) was added via syringe. The reaction mixture was stirred at 35 ºC for 16 h. After the reaction is completed, the mixture was concentrated under vacuum and the product was purified by flash column chromatography on silica gel using hexane/EtOAc as eluent to give the product **3i** in 98% yield.

4.2 Synthetic Application

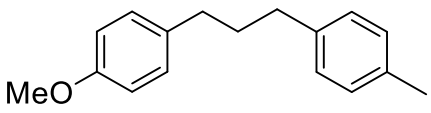


4-methoxybenzoyl fluoride (7)



Acyl fluoride was synthesized according to the general procedure for the deoxygenative fluorination of carboxylic acids. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 8.9 Hz, 2H), 7.01 (d, J = 8.4 Hz, 2H), 3.92 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.2, 157.3 (d, J = 339.8 Hz), 133.8 (d, J = 4.0 Hz), 116.9 (d, J = 61.8 Hz), 114.4, 55.7. ^{19}F NMR (377 MHz, $CDCl_3$) δ 15.98. Data in accordance with the literature.[1]

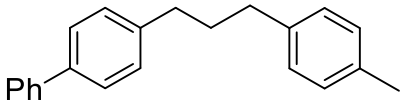
1-methoxy-4-(3-(p-tolyl)propyl)benzene (9)



A dry 20 mL high pressure tube equipped with a stirring bar was charged with $Pd(acac)_2$ (6.1 mg, 0.02 mmol, 10 mol %), dppe (12.0 mg, 0.03 mmol, 15 mol %), KF (17.4 mg, 0.3 mmol, 1.5 equiv.), toluene (0.8 mL), and DMSO (0.2 mL) in glovebox and the reaction mixture was stirred for 2 min at room temperature. Then alkyl 9-BBN **8** (0.32 mmol in 1 mL toluene, 1.6 equiv., generated from the reaction of 0.16 mmol 9-BBN-dimer and 0.32 mmol alkene in dry toluene at 80 °C for 3 h) and acyl fluoride **7** (30.8 mg, 0.2 mmol, 1.0 equiv.) were added sequentially. The reaction mixture was stirred at 140 ºC for 5 h. After the mixture was cooled to room temperature, quenched with saturated NH_4Cl, and extracted with EtOAc. The combined organic extracts were dried over anhydrous Na_2SO4, and

evaporated under vacuum to obtain the crude product which was purified by column chromatography (EtOAc:hexane) on silica gel to afford the desired products **9** in 52% yield (25.1 mg).[14] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 – 7.09 (m, 6H), 6.88 (d, *J* = 8.5 Hz, 2H), 3.84 (s, 3H), 2.64 (td, *J* = 7.7, 4.5 Hz, 4H), 2.37 (s, 3H), 1.96 (p, *J* = 7.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.7, 139.3, 135.1, 134.5, 129.3, 129.0, 128.4, 113.7, 55.3, 35.0, 34.5, 33.3, 21.0. HRMS (APPI FT-ICR MS) for $C_{17}H_{20}O$: calculated for $[M]^+$ 240.15087, found 240.15090.

4-(3-(p-tolyl)propyl)-1,1'-biphenyl (10)



A dry 20 mL high pressure tube equipped with a stirring bar was charged with $NiCl_2(PCy_3)_2$ (13.8 mg, 0.02 mmol, 10 mol %), PCy_3 (11.2 mg, 0.04 mmol, 20 mol %), aryl methyl ether 9 (48.0 mg, 0.2 mmol, 1 equiv.), and toluene (1.4 mL) in glovebox. Then PhMgBr (0.6 ml, 0.6 mmol, 3 equiv., 1 M in THF) was added and the reaction mixture was stirred at 100 ºC for 16 h. After the mixture was cooled to room temperature, quenched with saturated NH_4Cl, and extracted with EtOAc. The combined organic extracts were dried over anhydrous Na_2SO_4, and evaporated under vacuum to obtain the crude product which was purified by column chromatography (EtOAc:hexane) on silica gel to afford the desired products **10** in 63% yield (36.2 mg).[15] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.6 Hz, 2H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.49 (t, *J* = 7.7 Hz, 2H), 7.38 (t, *J* = 7.3 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.16 (s, 4H), 2.73 (dt, *J* = 15.5, 7.7 Hz, 4H), 2.39 (s, 3H), 2.04 (p, *J* = 7.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.6, 141.2, 139.2, 138.7, 135.2, 129.1, 128.92, 128.86, 128.76, 128.4, 127.1, 127.0, 35.1, 33.1, 21.6, 21.1. HRMS (APPI FT-ICR MS) for $C_{22}H_{22}$: calculated for $[M]^+$ 286.1716, found 286.1717.

5. References

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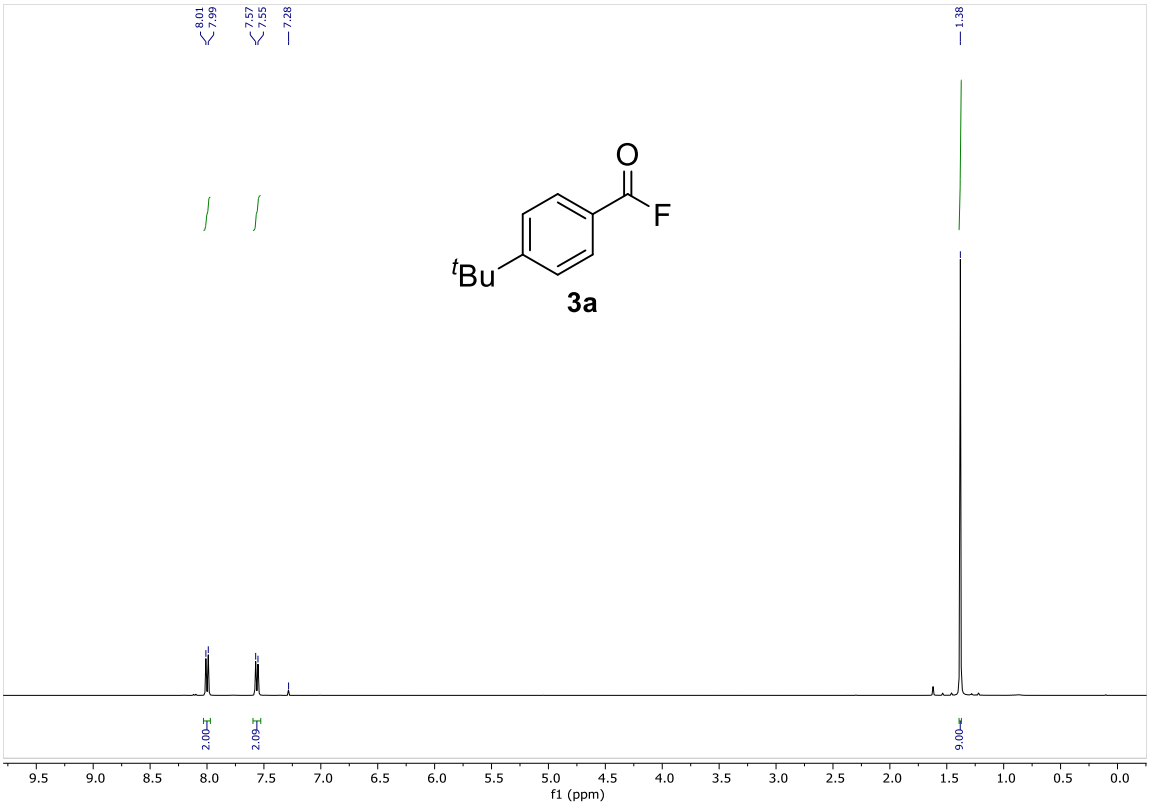
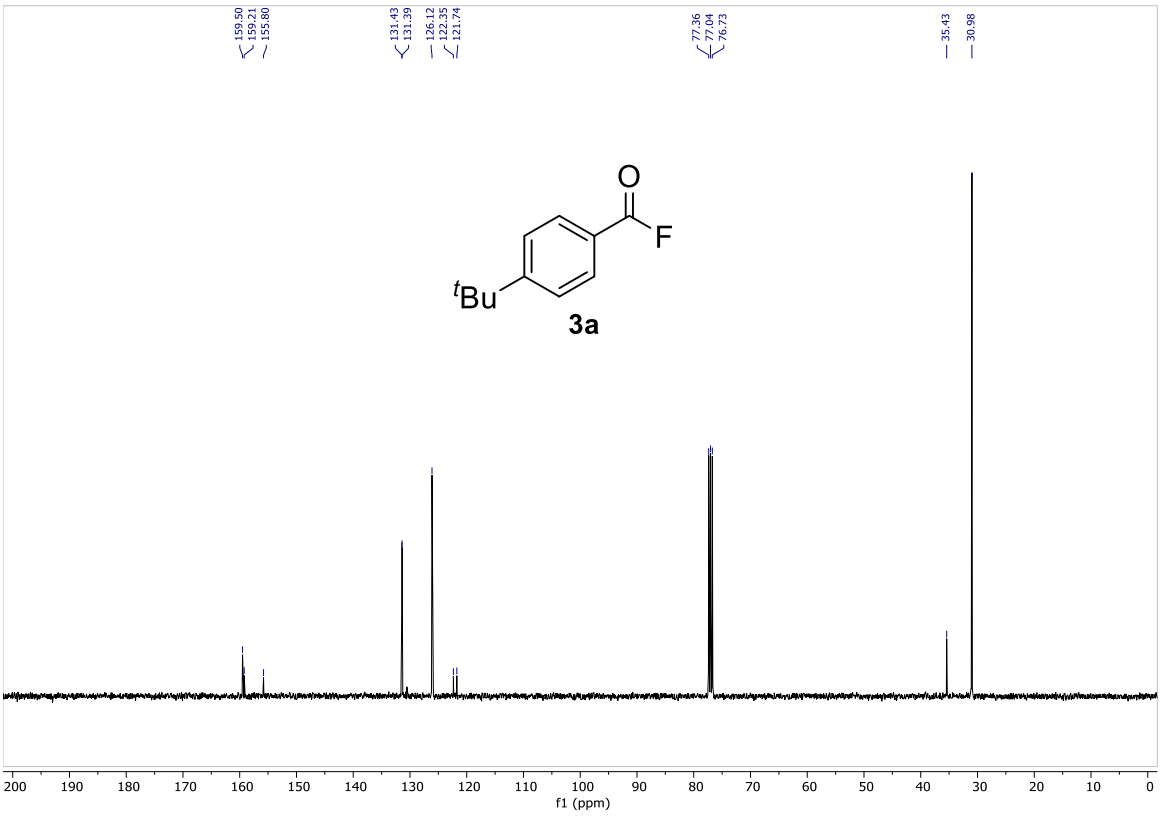
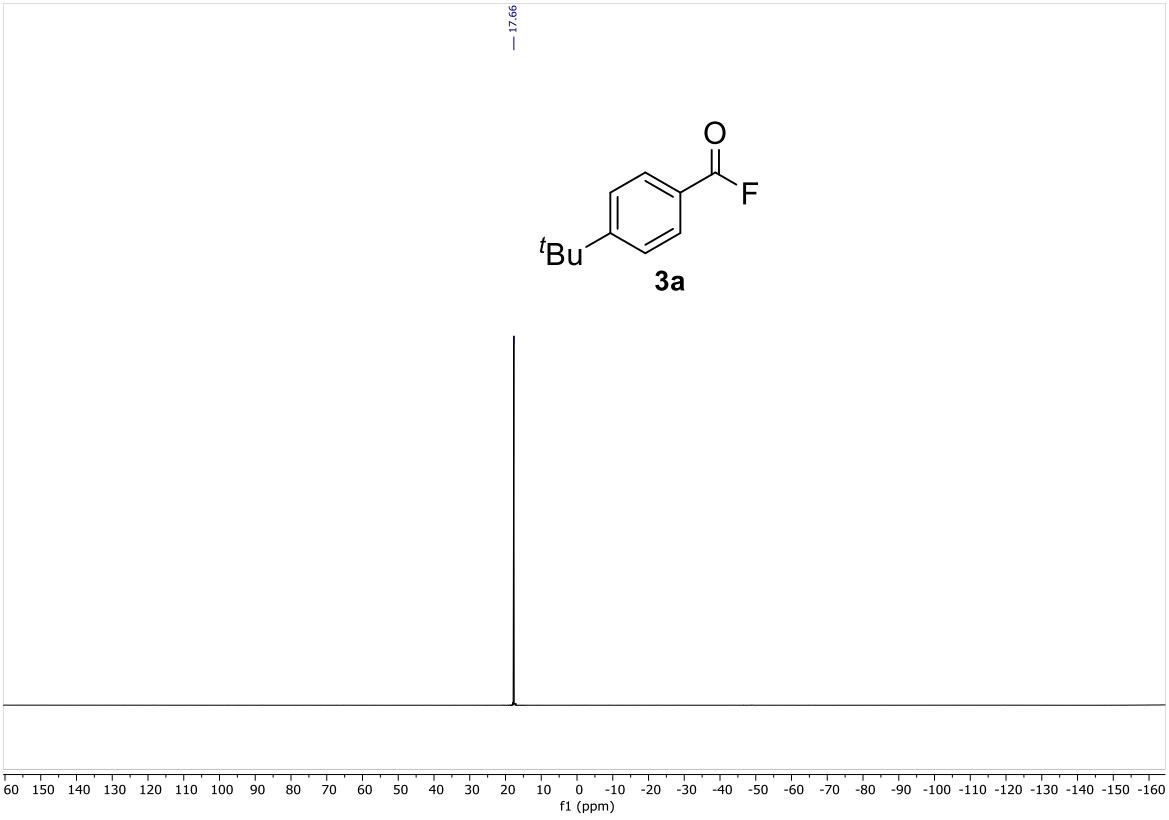
6. Copies of NMR Spectra



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O
F
tBu
3a
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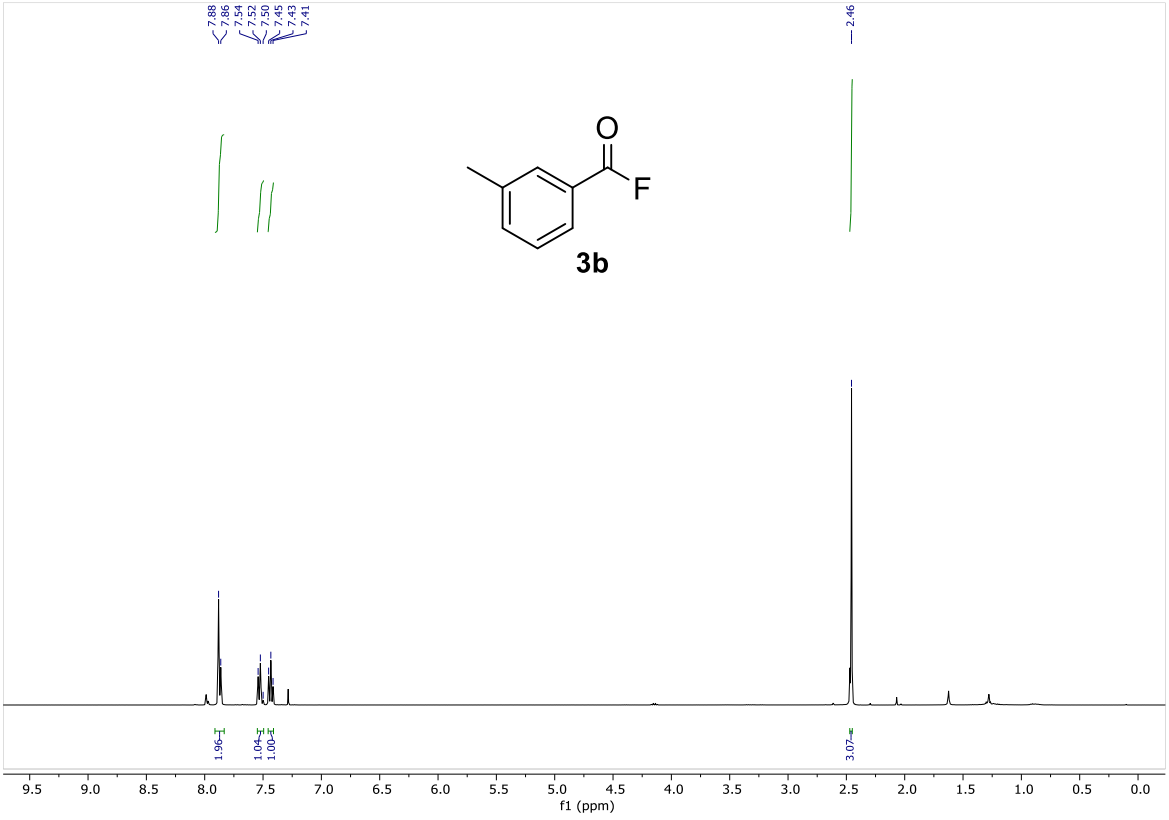
f1 (ppm)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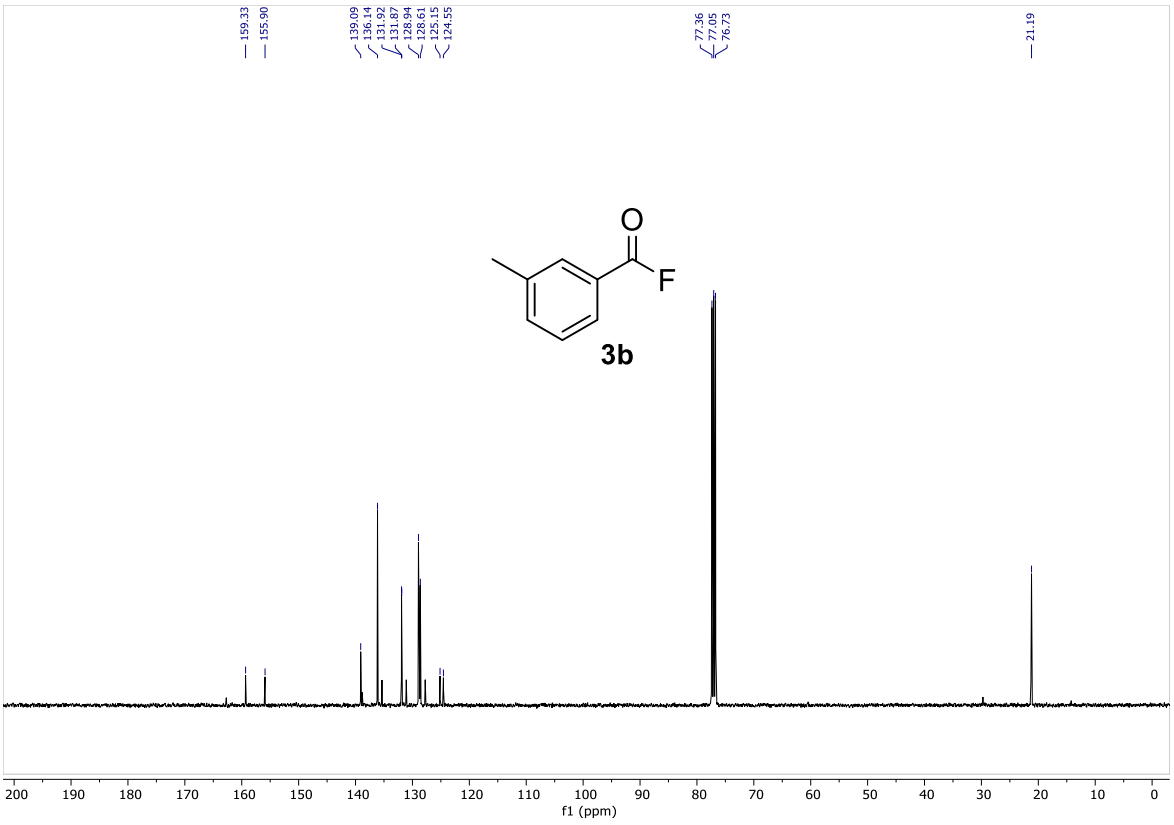
7.86
7.54
7.52
7.50
7.45
7.43
7.41
2.46
O
F
3b
1.96
1.04
1.00
3.07
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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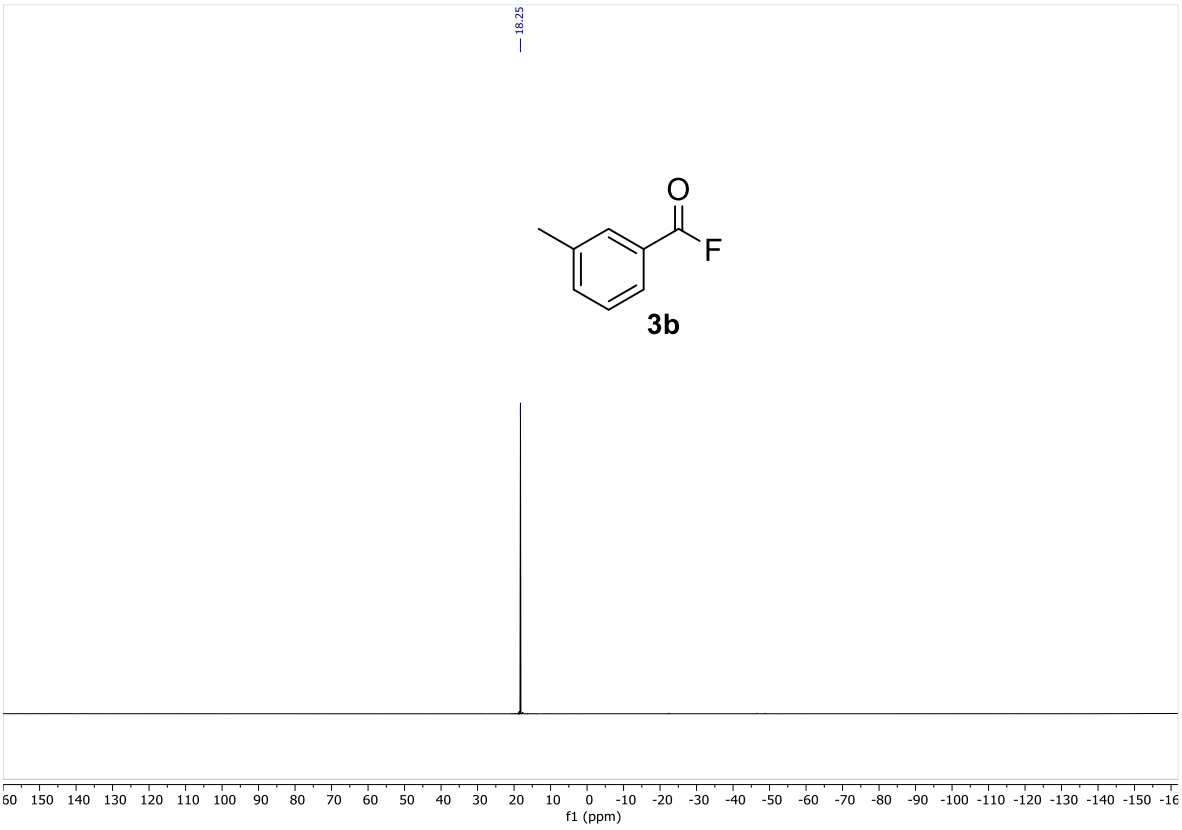
f1 (ppm)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O
F
3b
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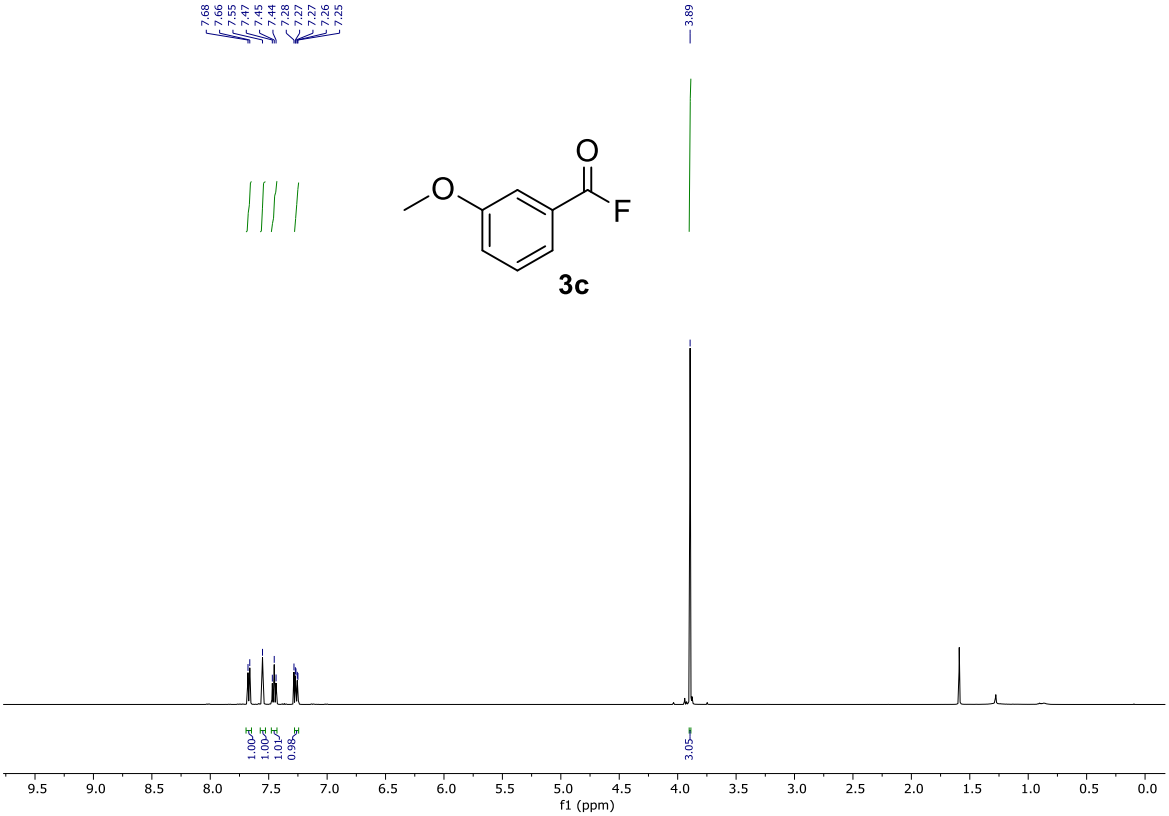
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7.47
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7.44
7.28
7.27
7.27
7.26
7.25
3.89
O
O
F
3c
1.00
1.00
1.01
0.98
3.05
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
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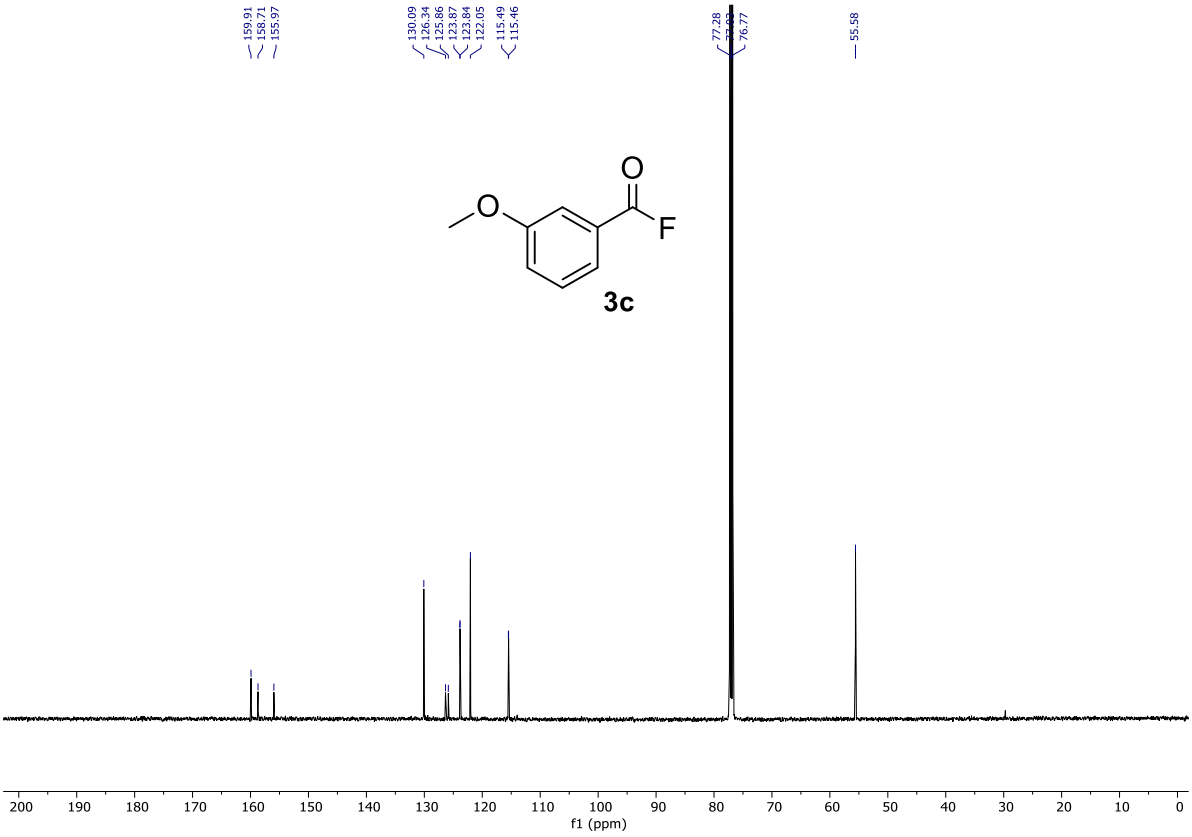
1.0
0.5
0.0
f1 (ppm)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76.77
55.58
O
O
F
3c
200
190
180
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10
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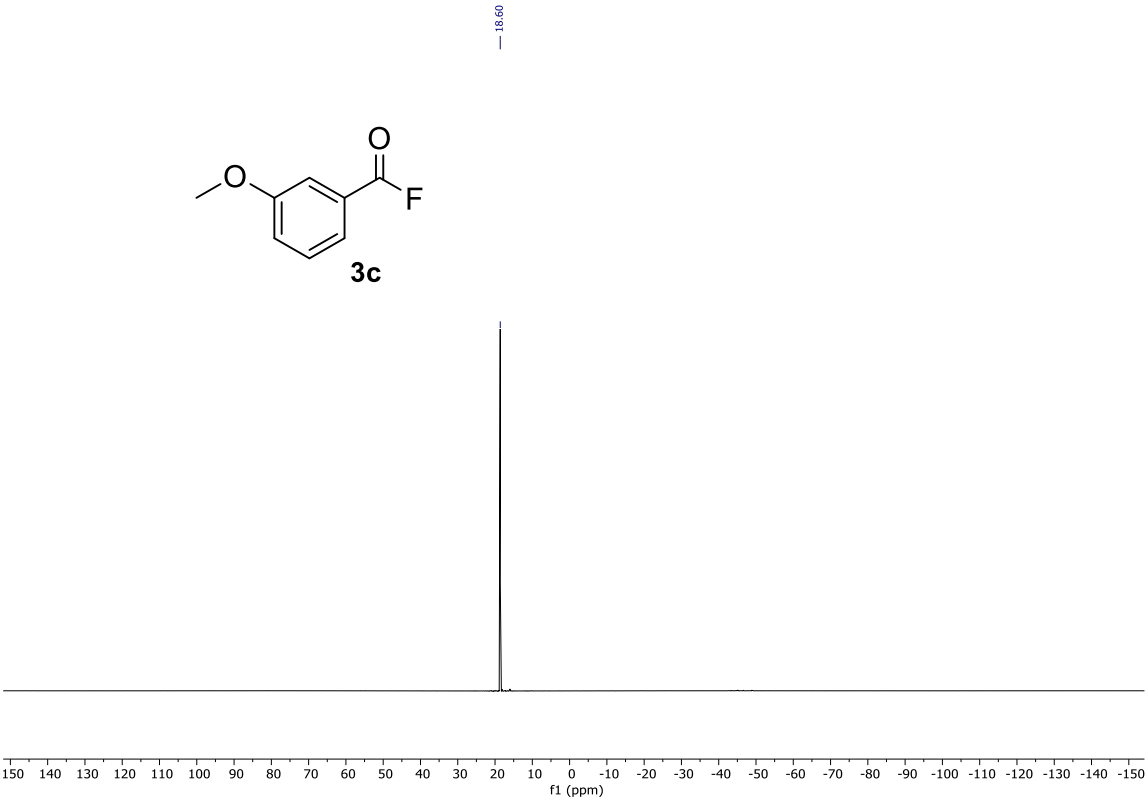
f1 (ppm)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O
F
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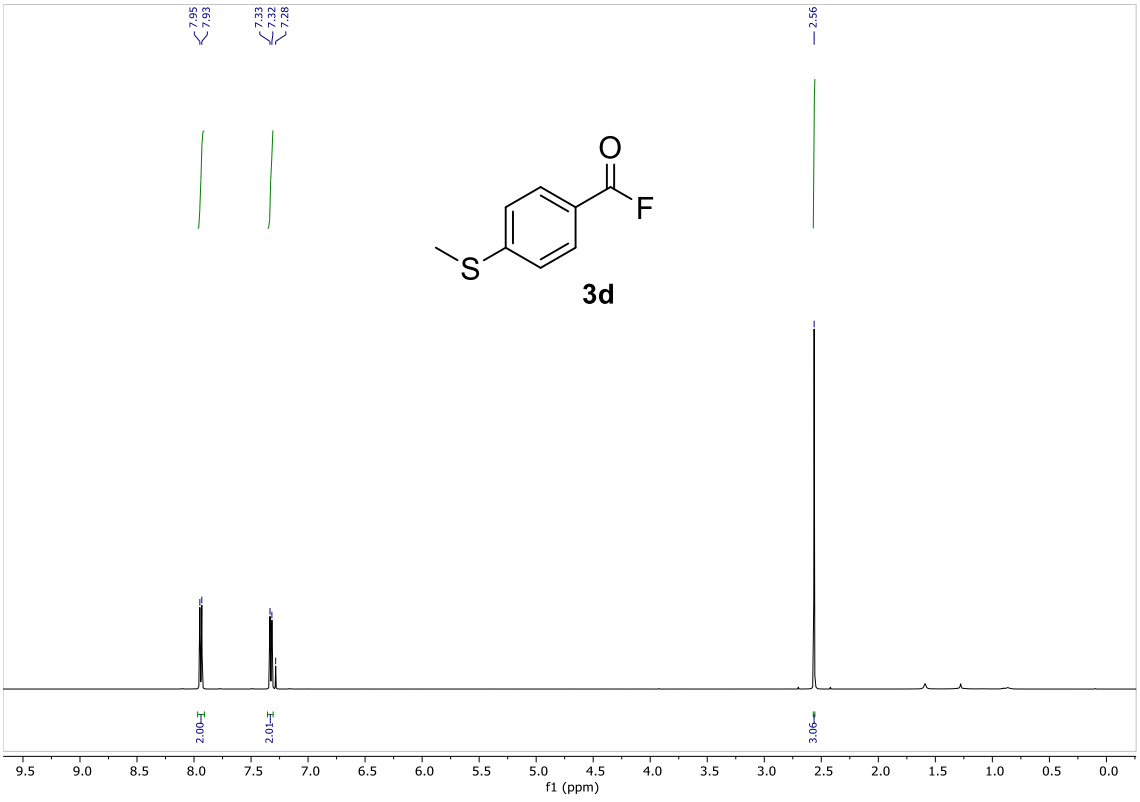
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S
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2.01
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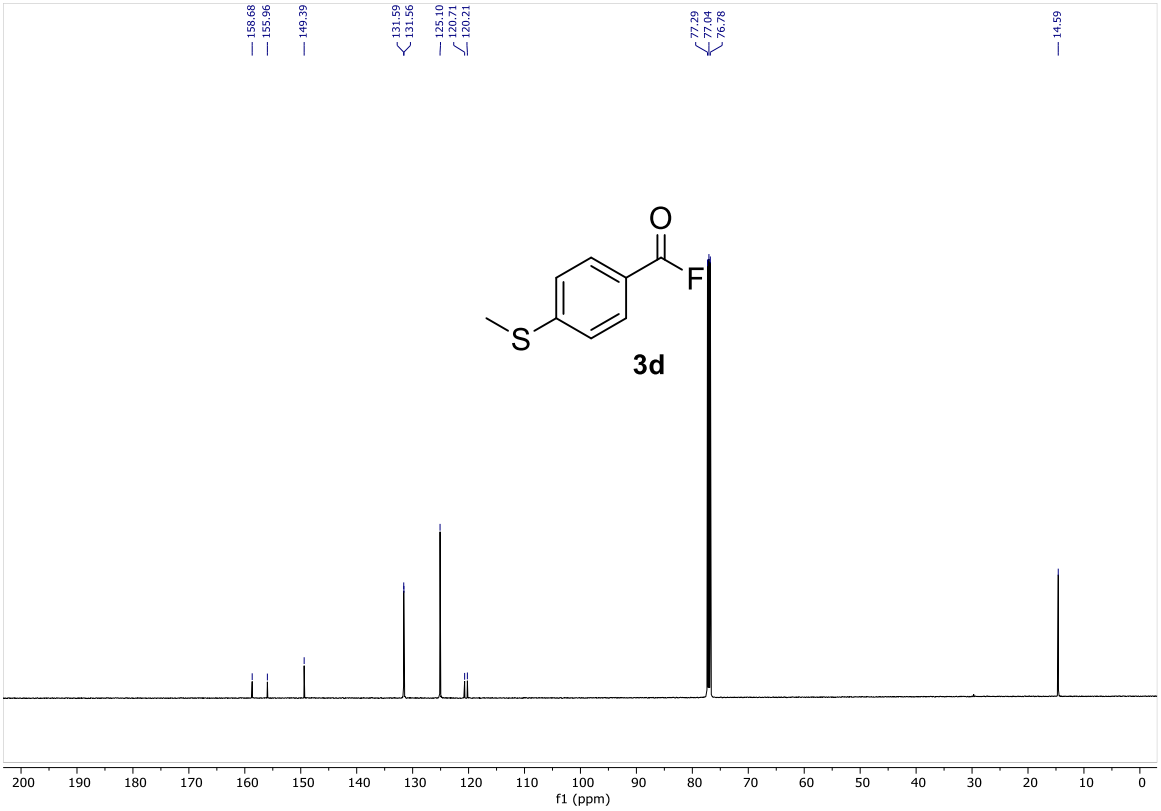
f1 (ppm)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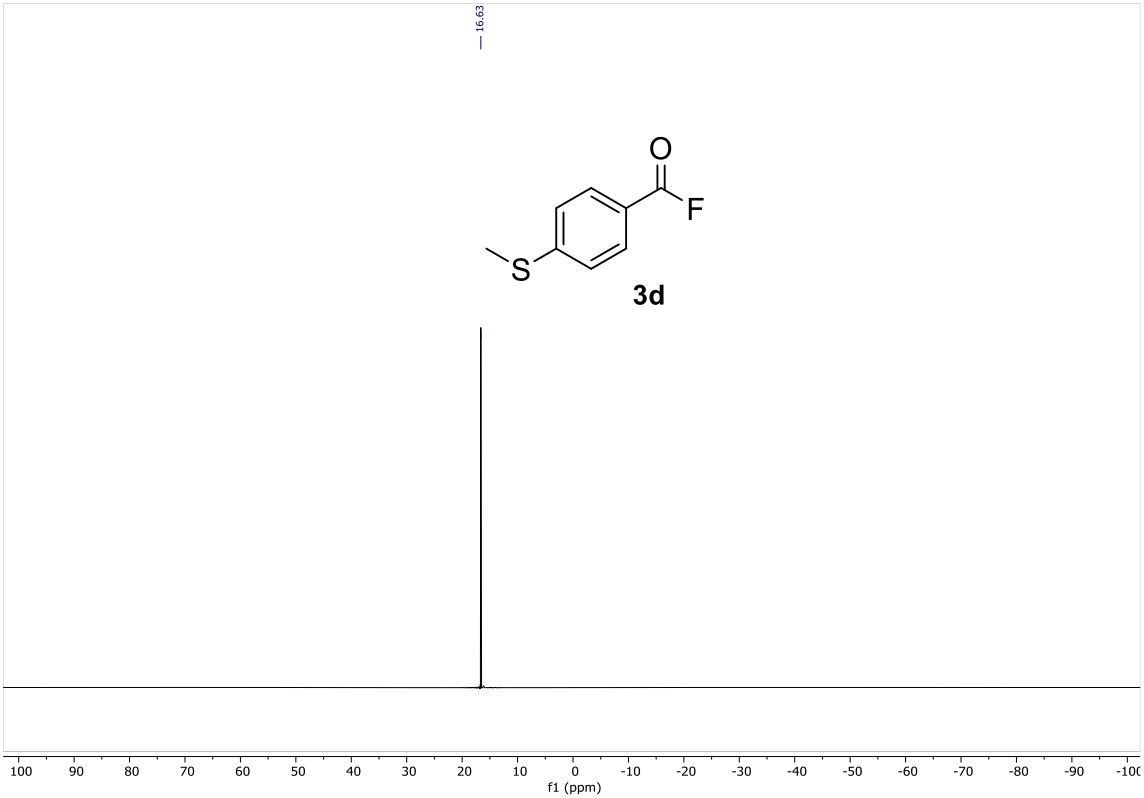
f1 (ppm)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F
S
3d
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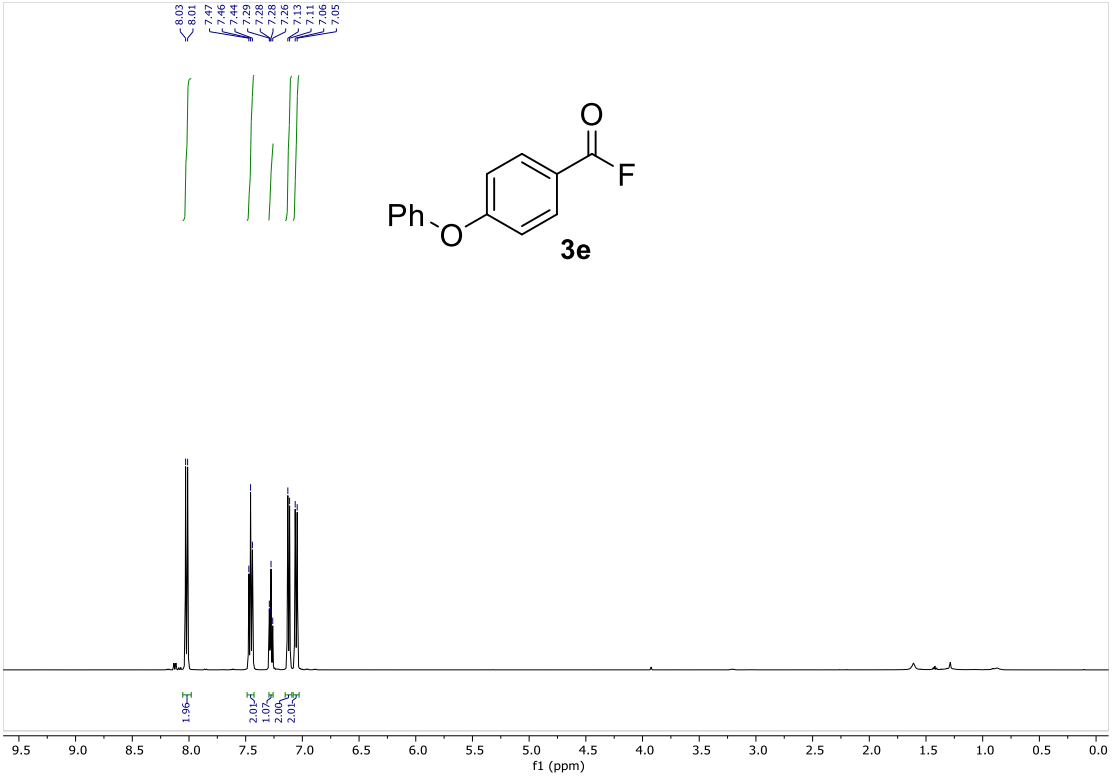
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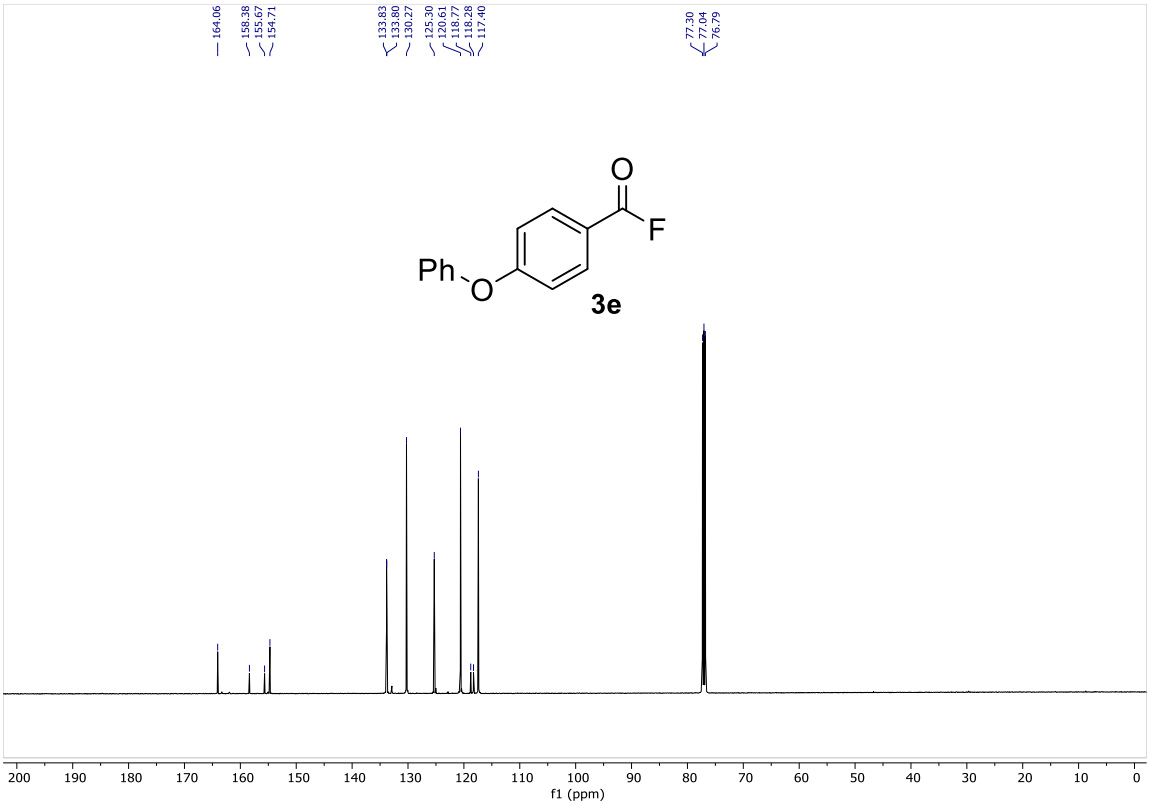
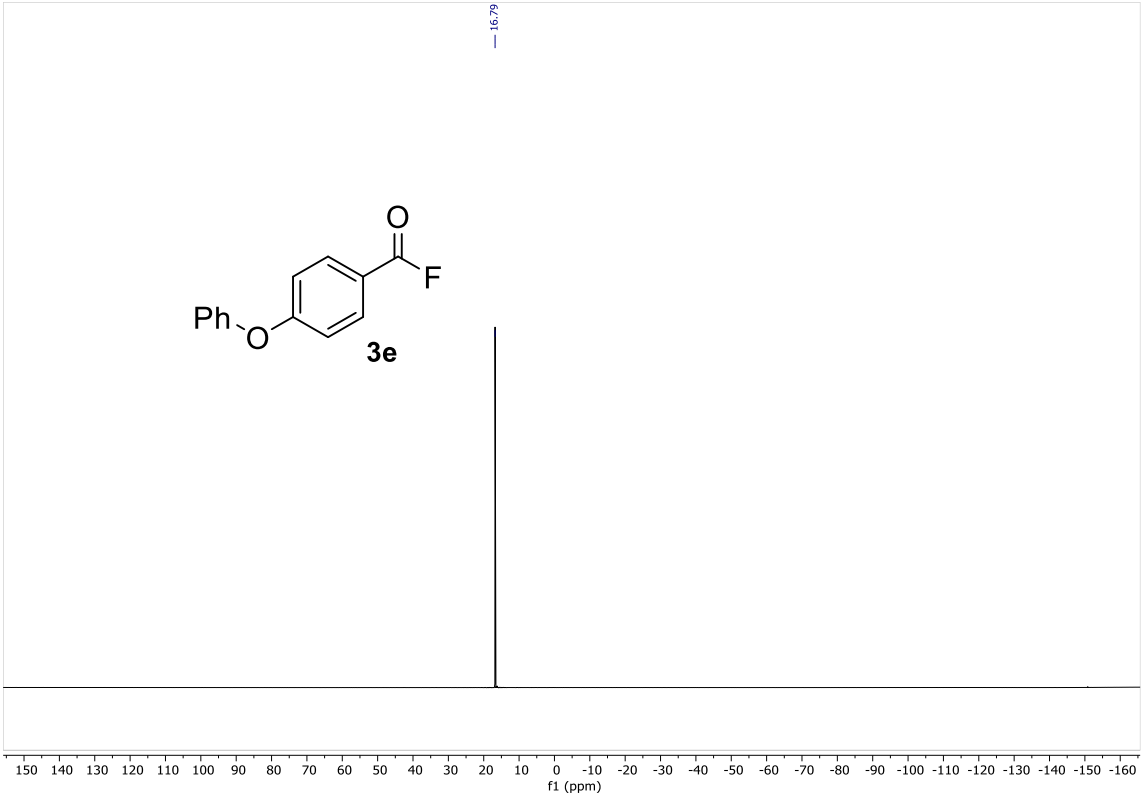
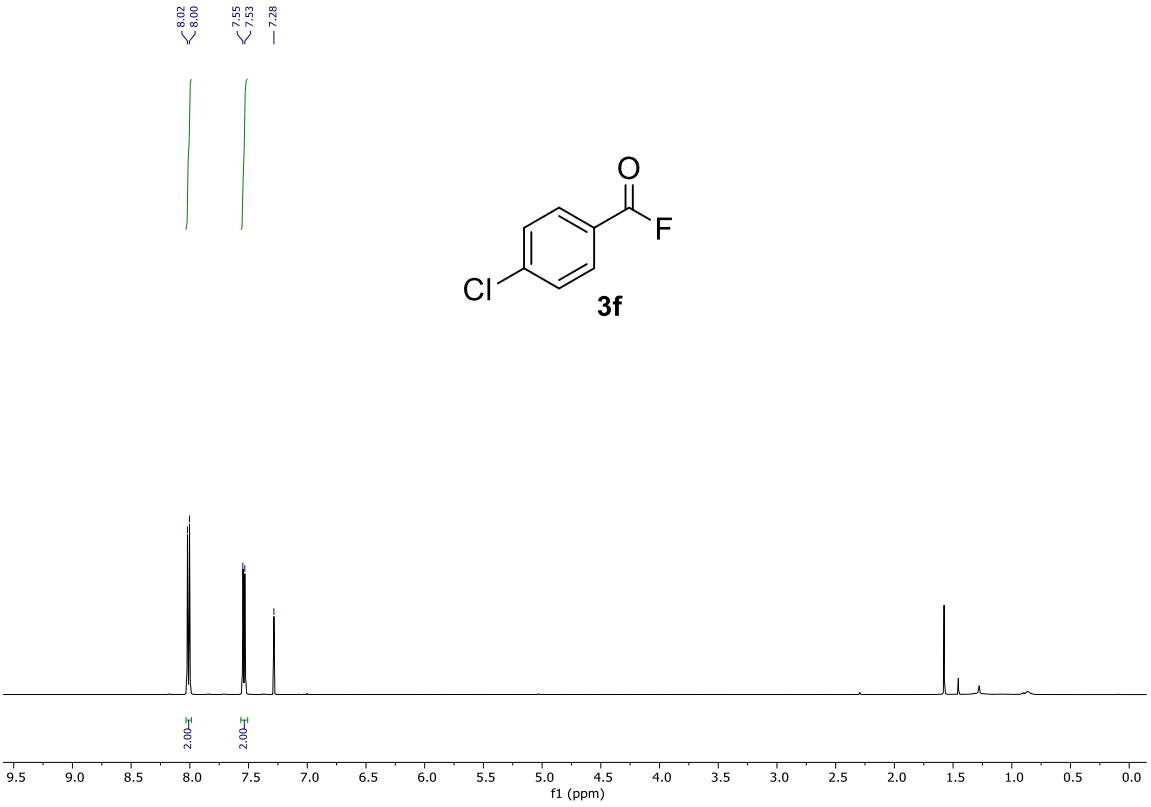
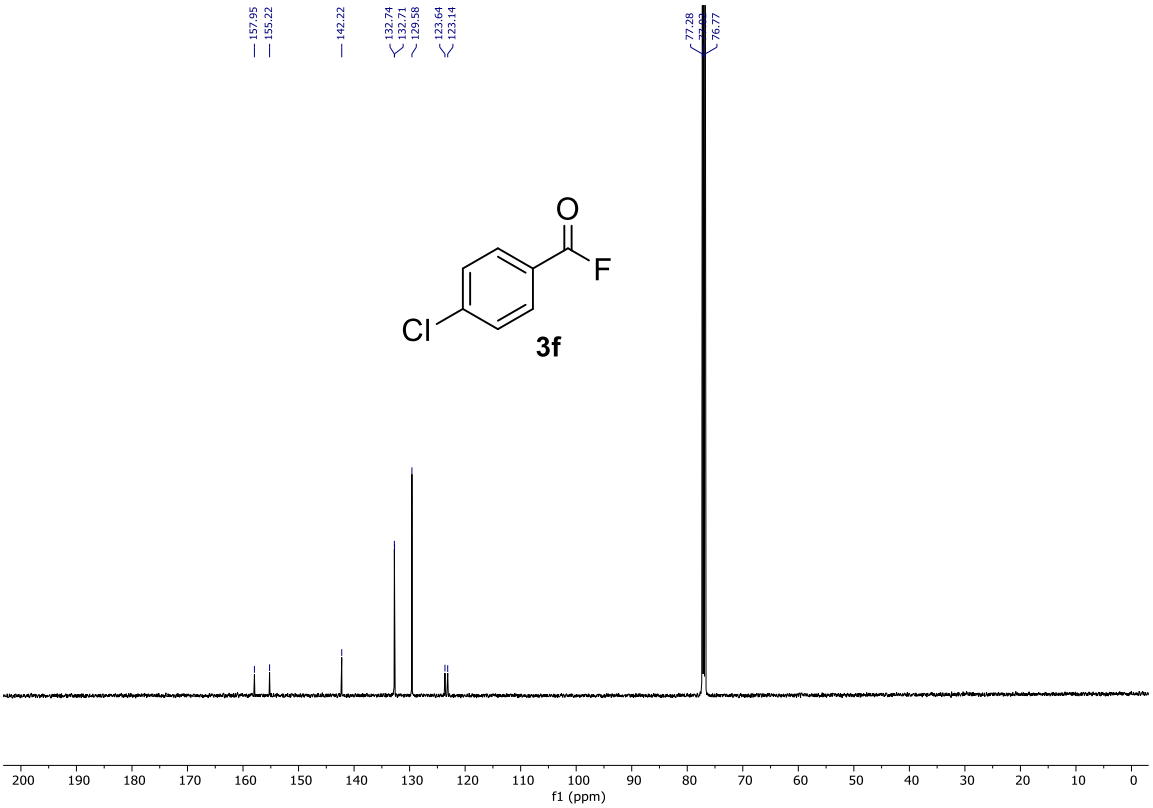
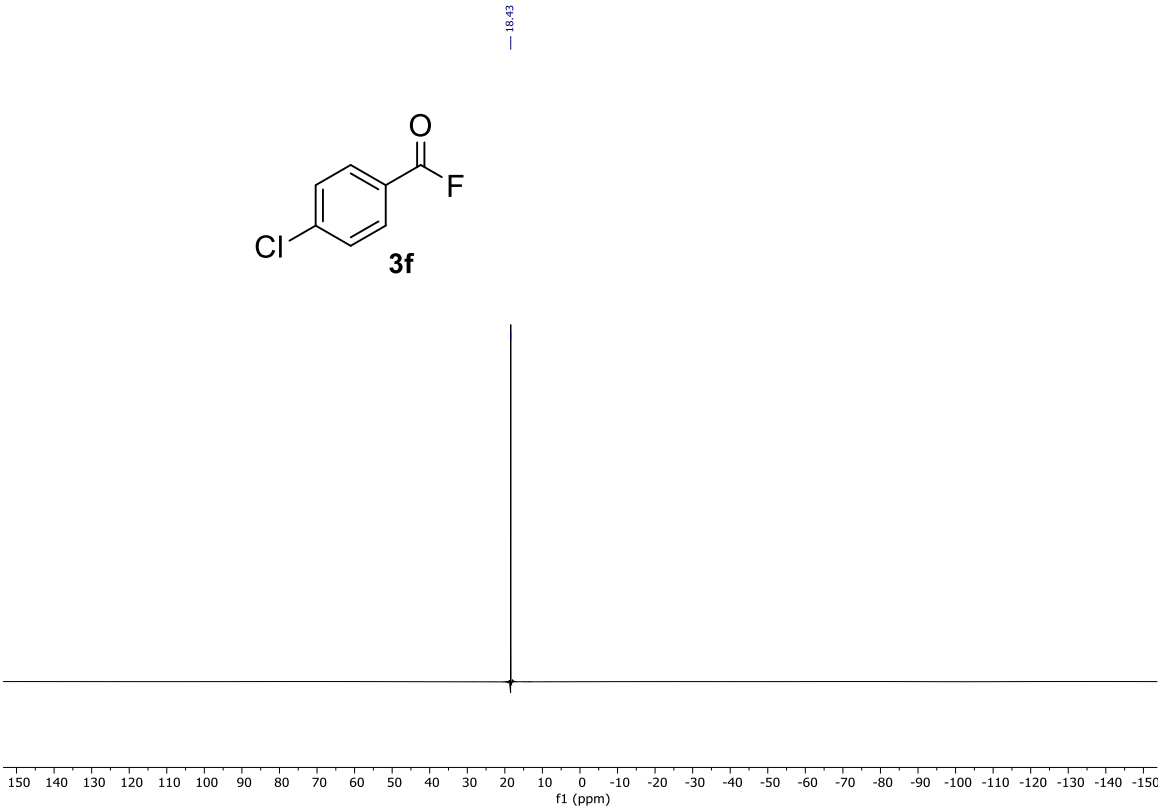
7.06
7.05
O
F
Ph
O
3e
1.96
2.01
1.07
2.00
2.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

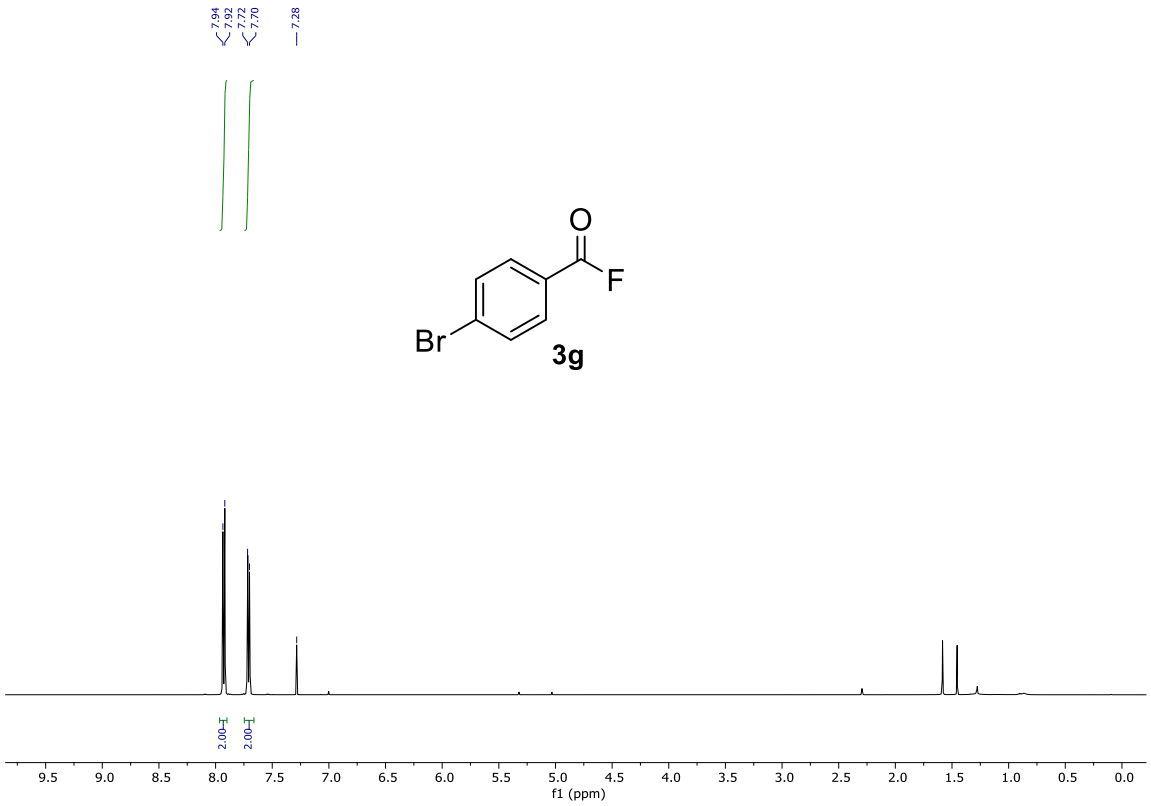

164.06
158.38
155.67
154.71
133.83
133.80
130.27
125.30
120.61
118.77
118.28
117.40
77.30
77.04
76.79
O
F
Ph
O
3e
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

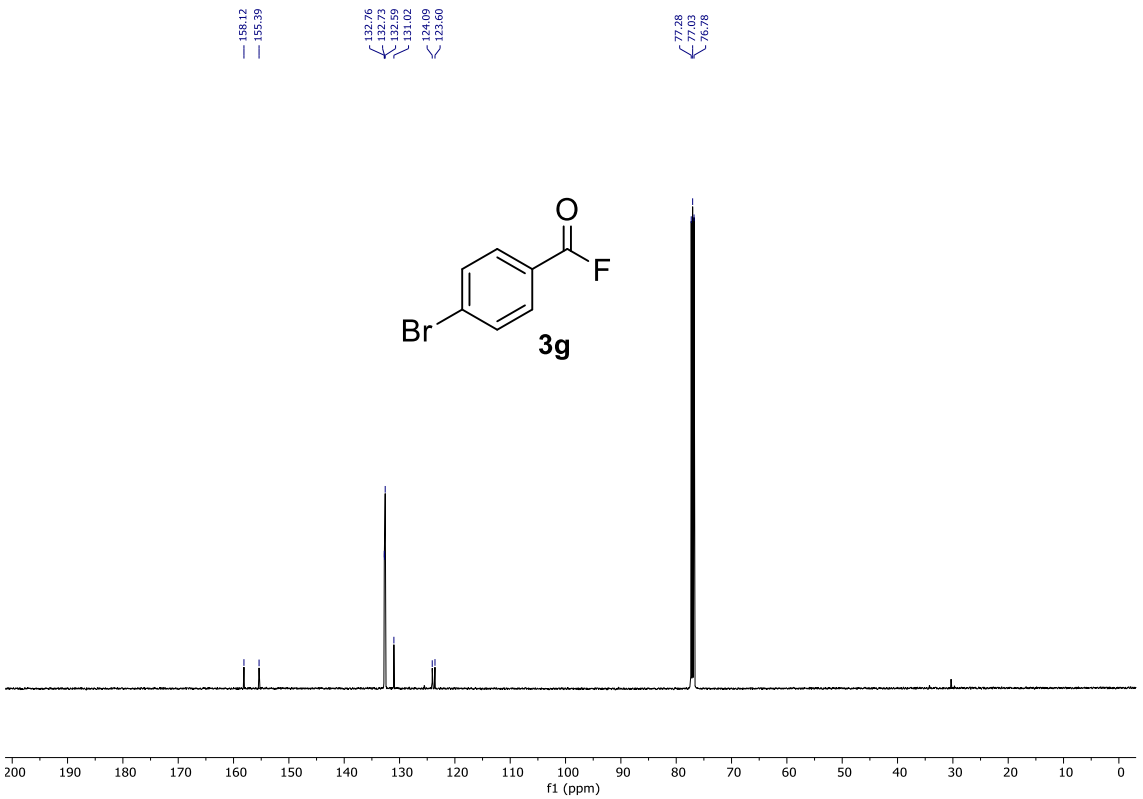

16.79
O
F
Ph
O
3e
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

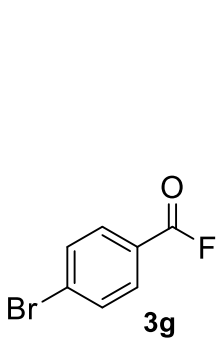

8.02
8.00
7.55
7.53
7.28
O
F
Cl
3f
2.00
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


157.95
155.22
142.22
132.74
132.71
129.58
123.64
123.14
77.28
76.77
O
F
Cl
3f
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


18.43
O
F
Cl
3f
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

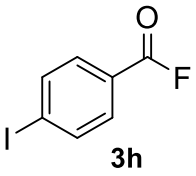

7.94
7.92
7.72
7.70
7.28
O
F
Br
3g
2.00
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


158.12
155.39
132.76
132.73
132.59
131.02
124.09
123.60
77.28
77.03
76.78
O
F
Br
3g
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
F
Br

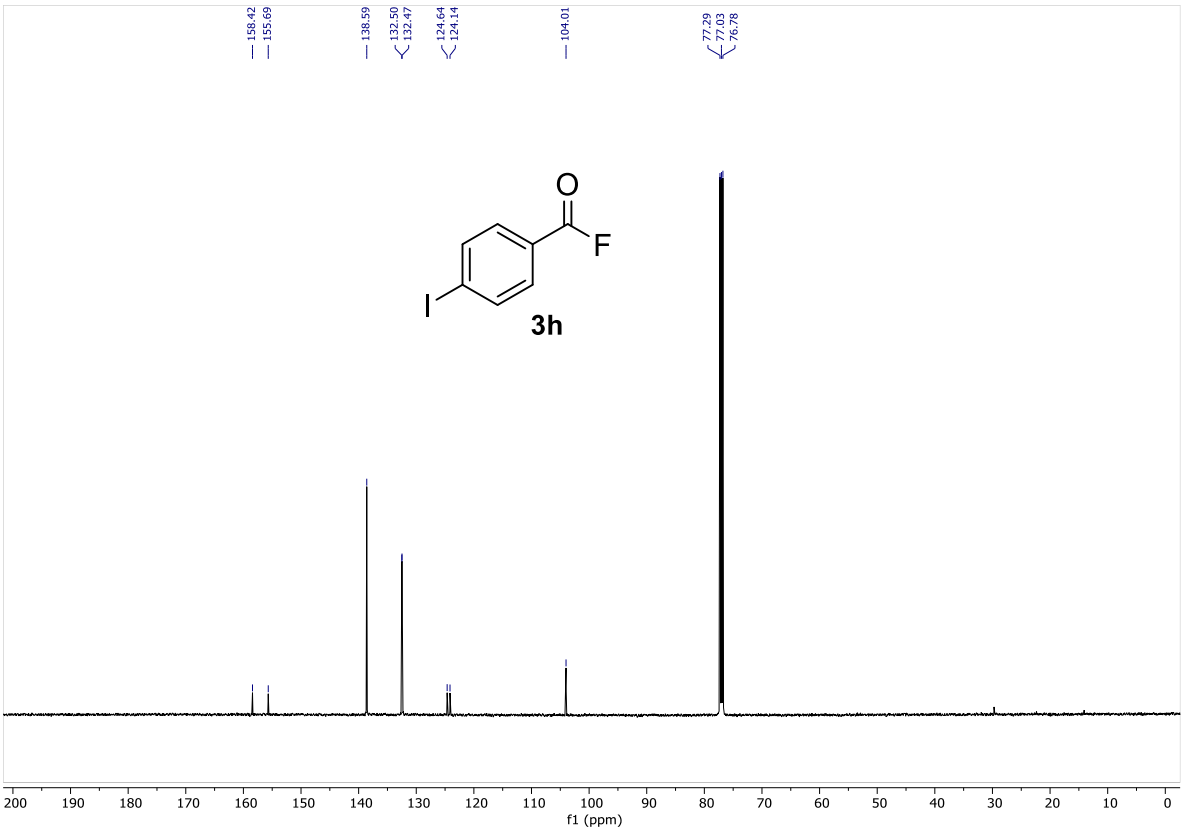
3g

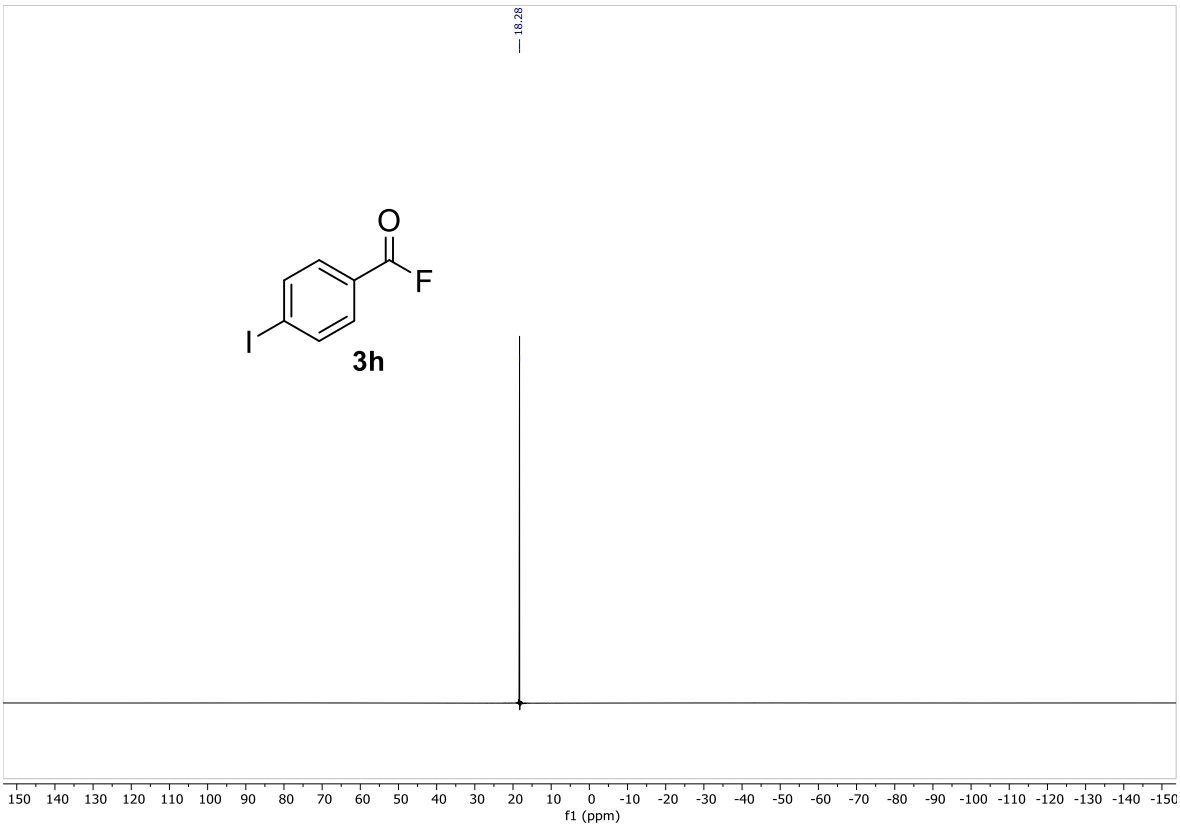
18.43
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

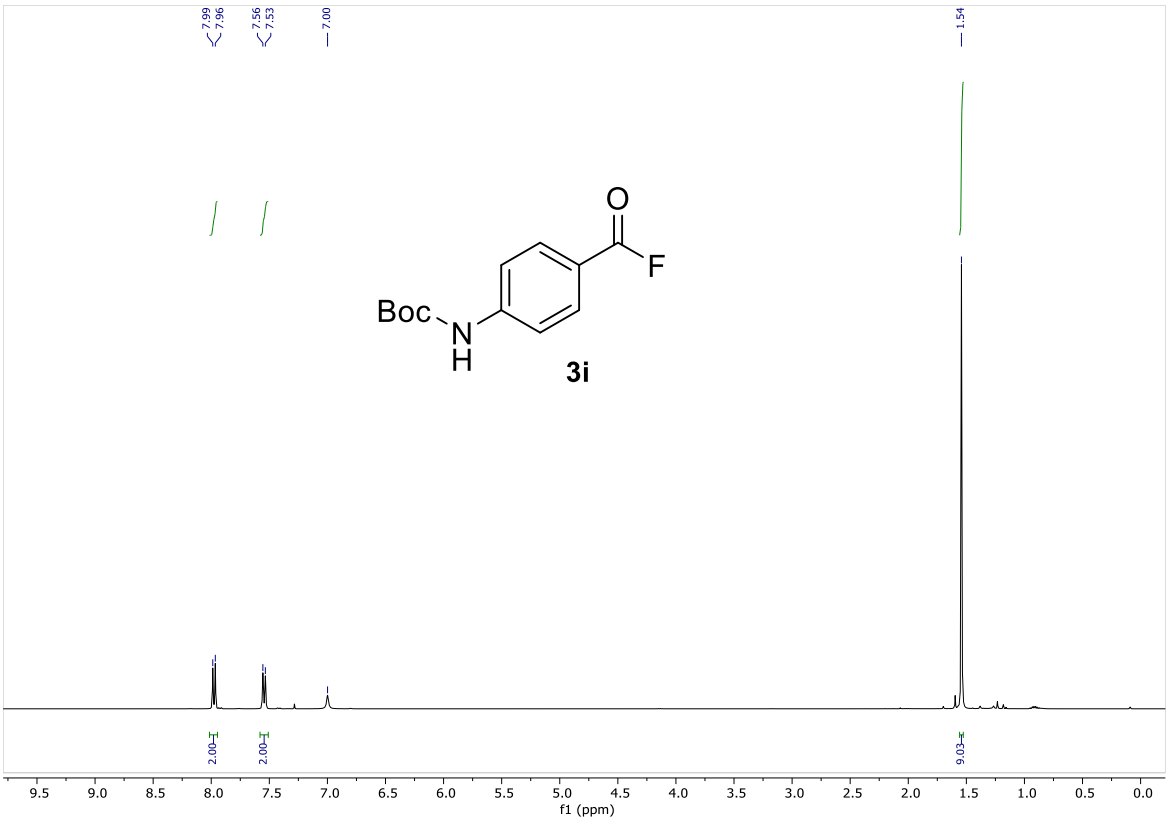

O
F
I

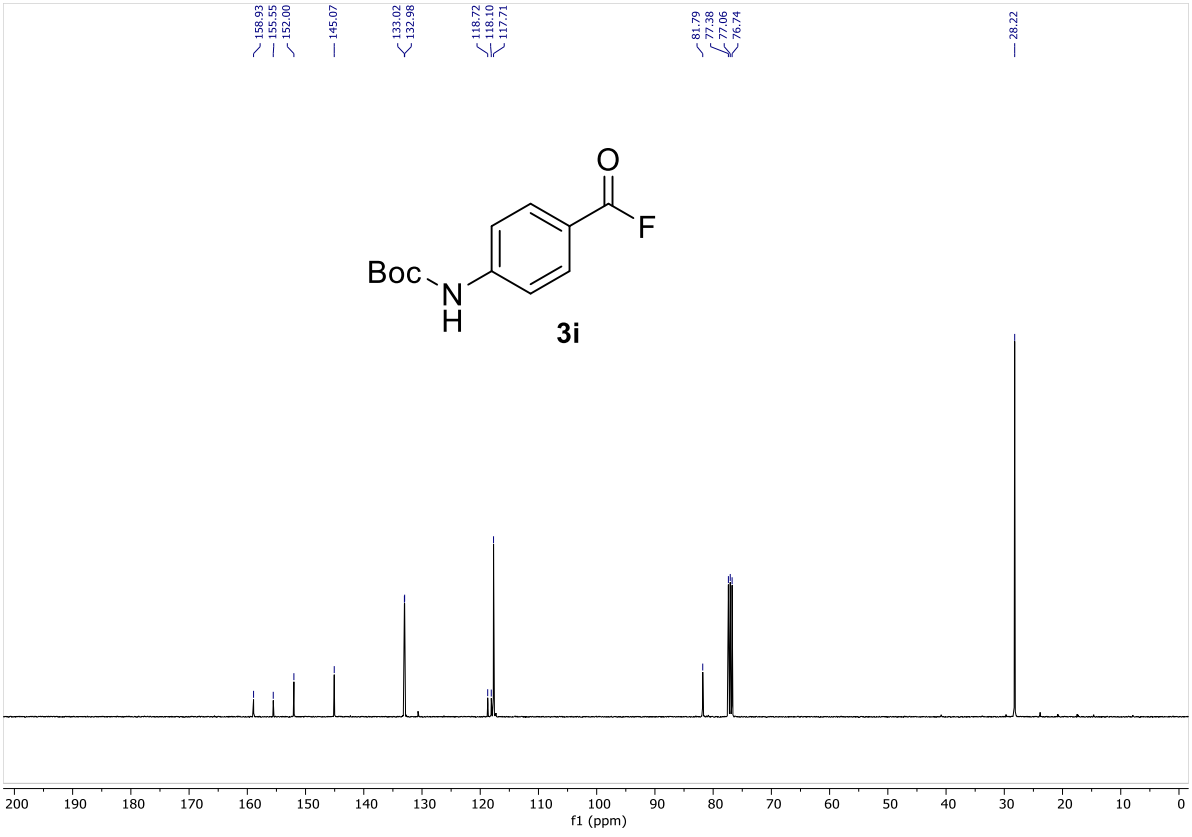
3h

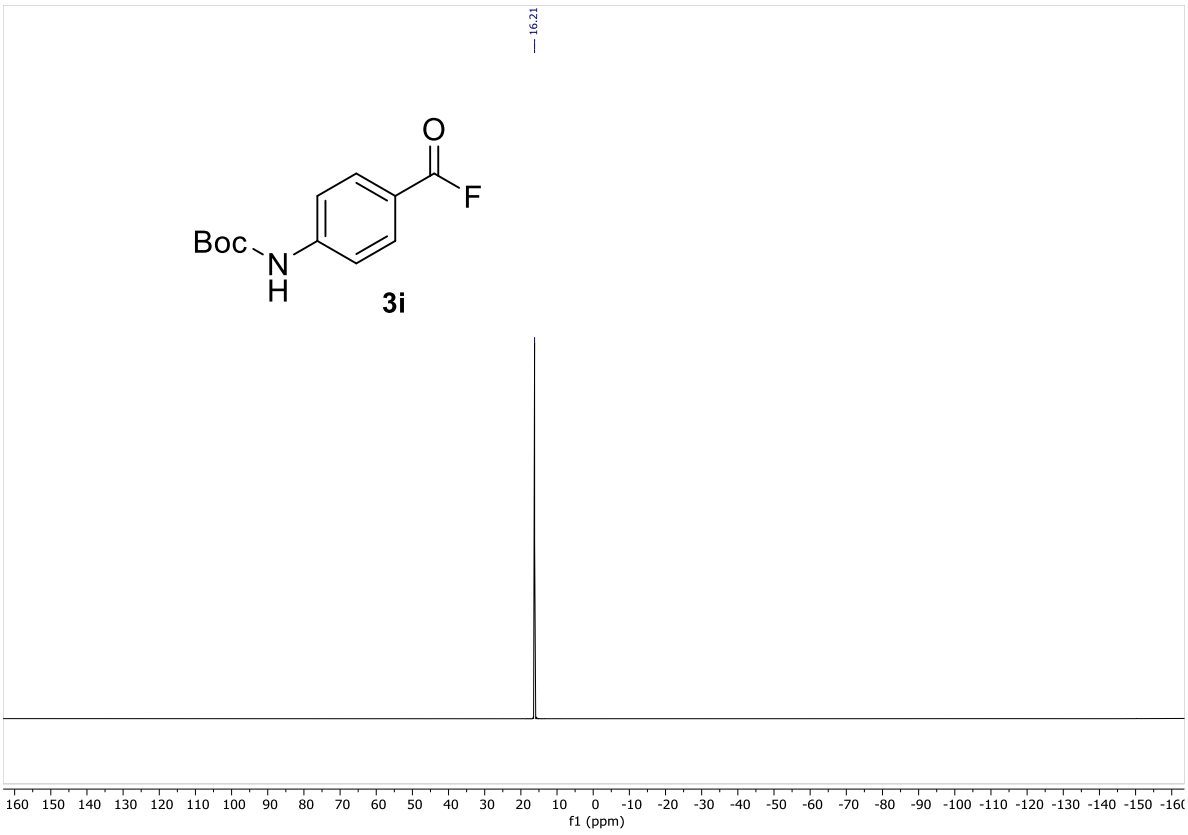
7.94
7.92
7.77
7.75
7.28
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

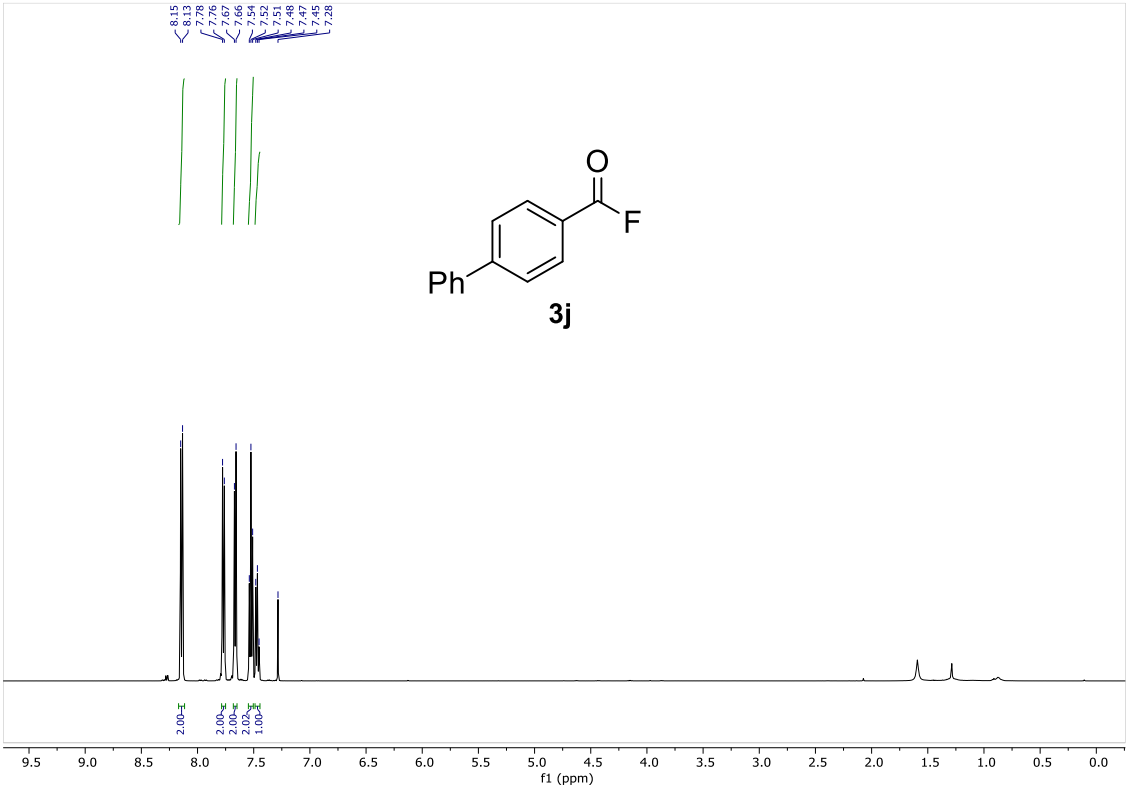

158.42
155.69
138.59
132.50
132.47
124.64
124.14
104.01
77.39
77.03
76.78
O
F
I
3h
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

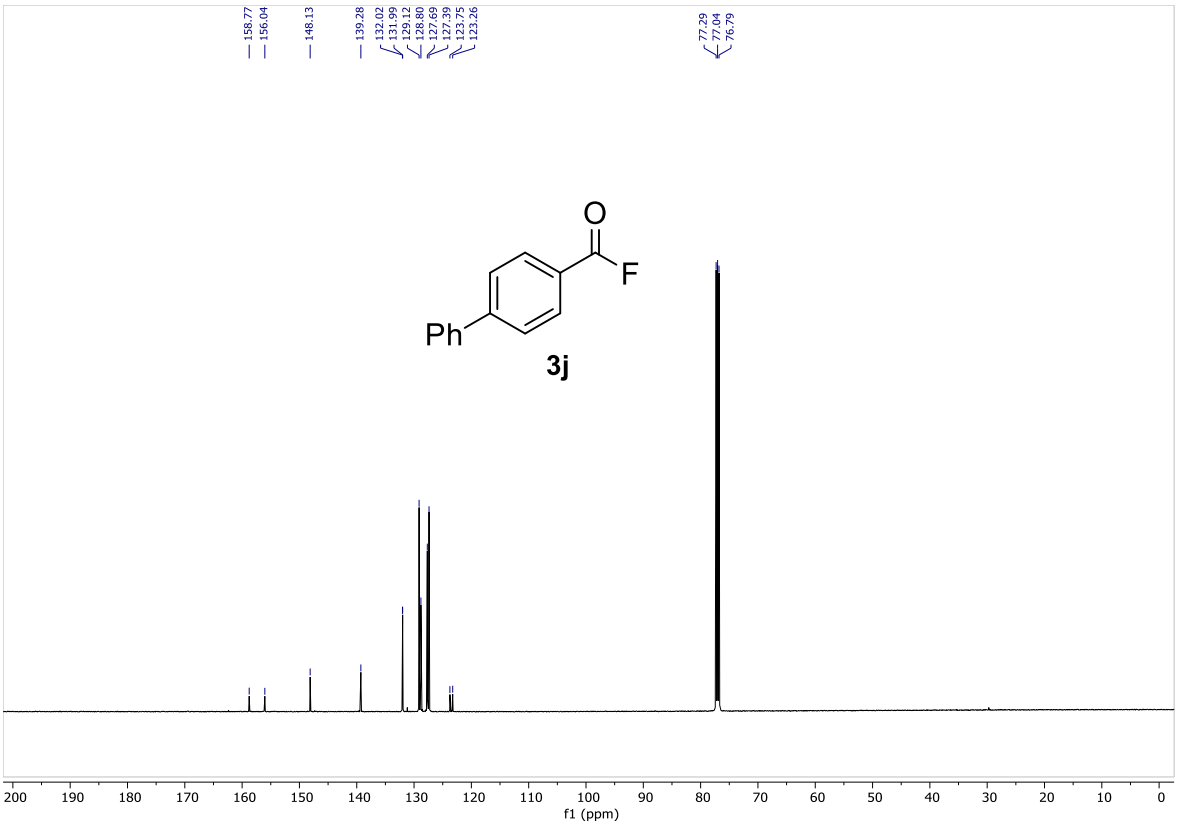

18.28
O
F
I
3h
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

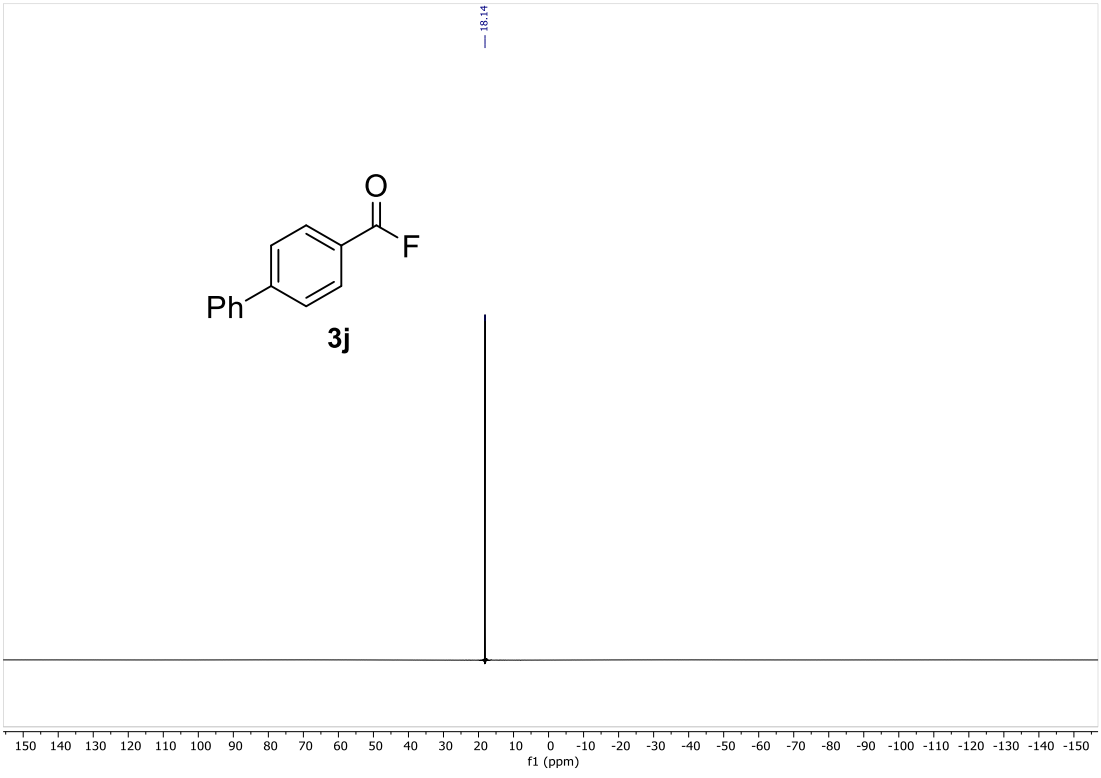

7.99
7.96
7.56
7.53
7.00
1.54
O
F
Boc
N
H
3i
2.00
2.00
9.03
f1 (ppm)

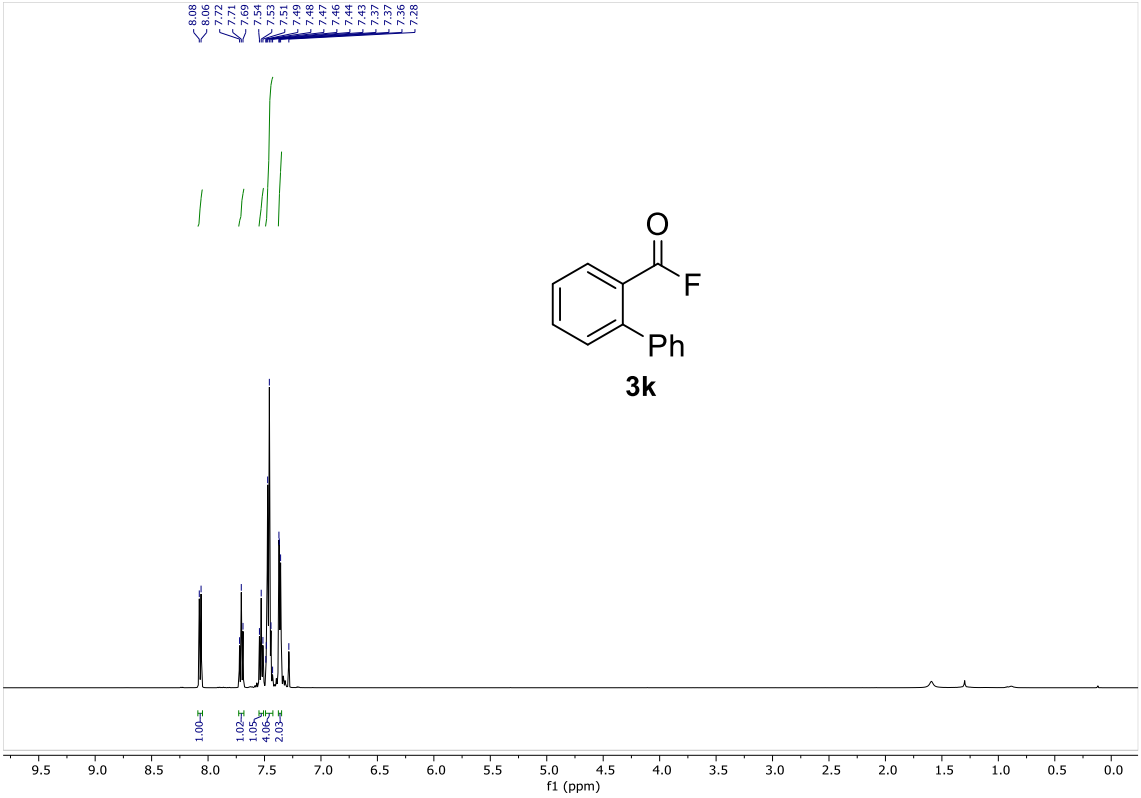

158.93
155.55
152.00
145.07
133.02
132.98
118.72
118.10
117.71
81.79
77.38
77.06
76.74
28.22
O
F
Boc
N
H
3i
f1 (ppm)

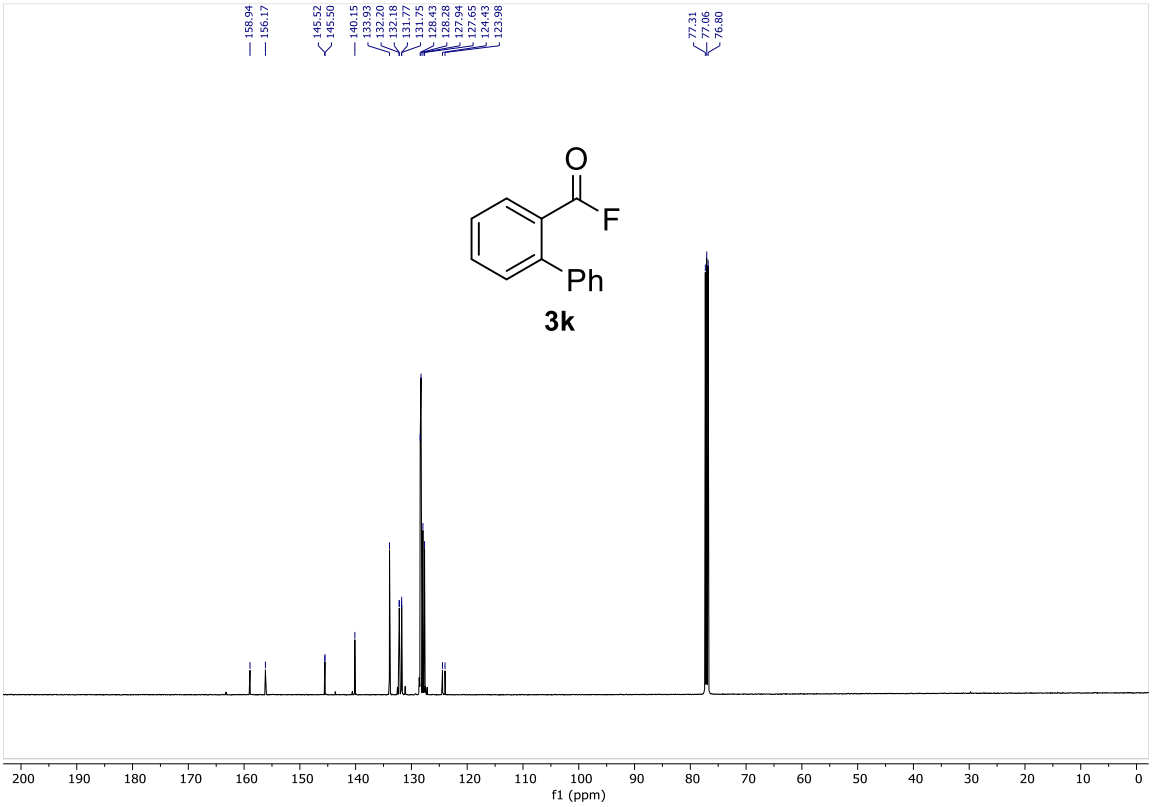

16.21
O
F
Boc
N
H
3i
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

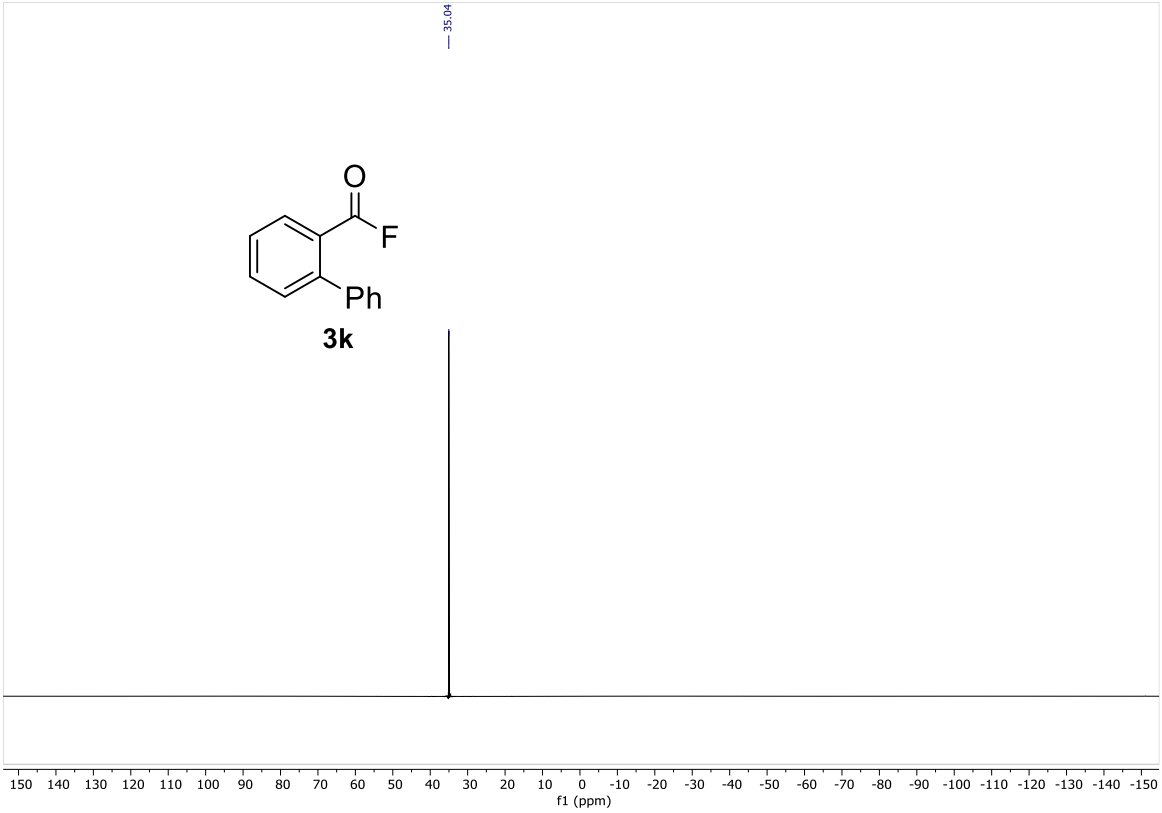

8.15
8.13
7.78
7.76
7.67
7.66
7.54
7.52
7.51
7.48
7.47
7.45
7.28
O
F
Ph
3j
2.00
2.00
2.02
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

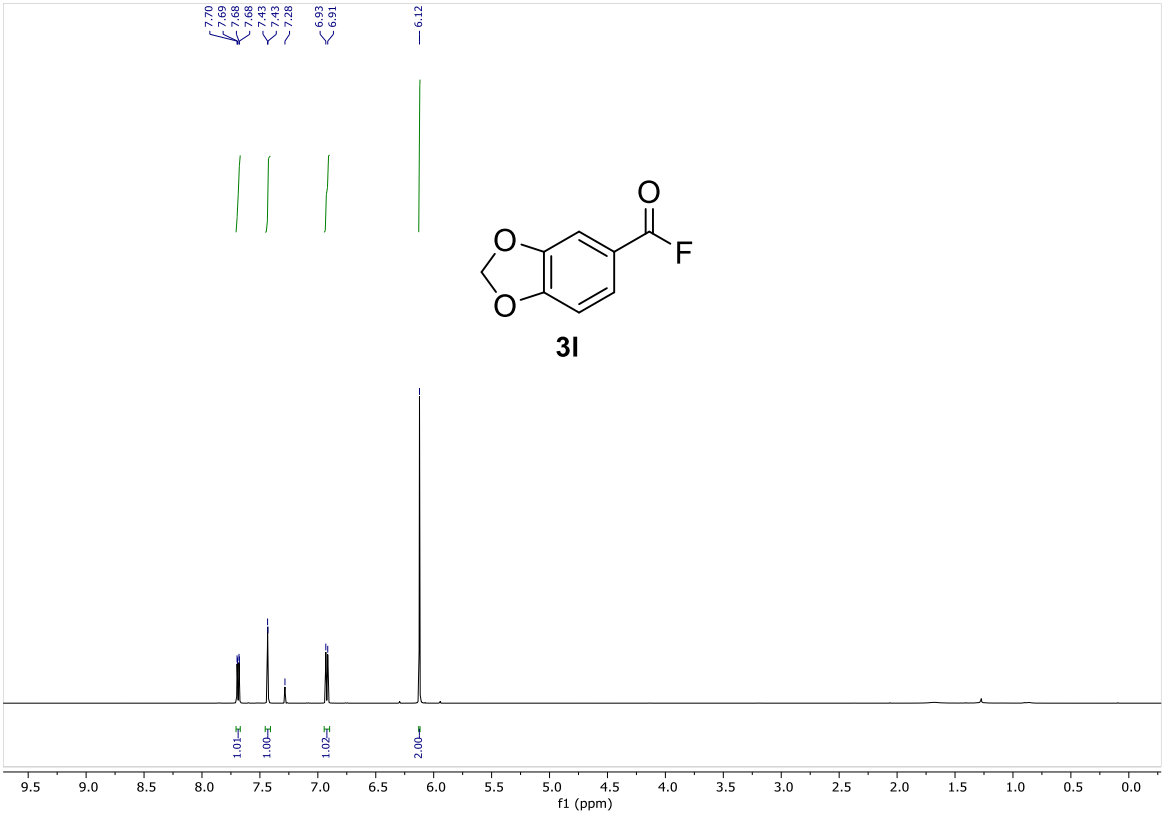

158.77
156.04
148.13
139.28
132.02
131.99
129.12
128.80
127.69
127.39
123.75
123.26
77.29
77.04
76.79
O
F
Ph
3j
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

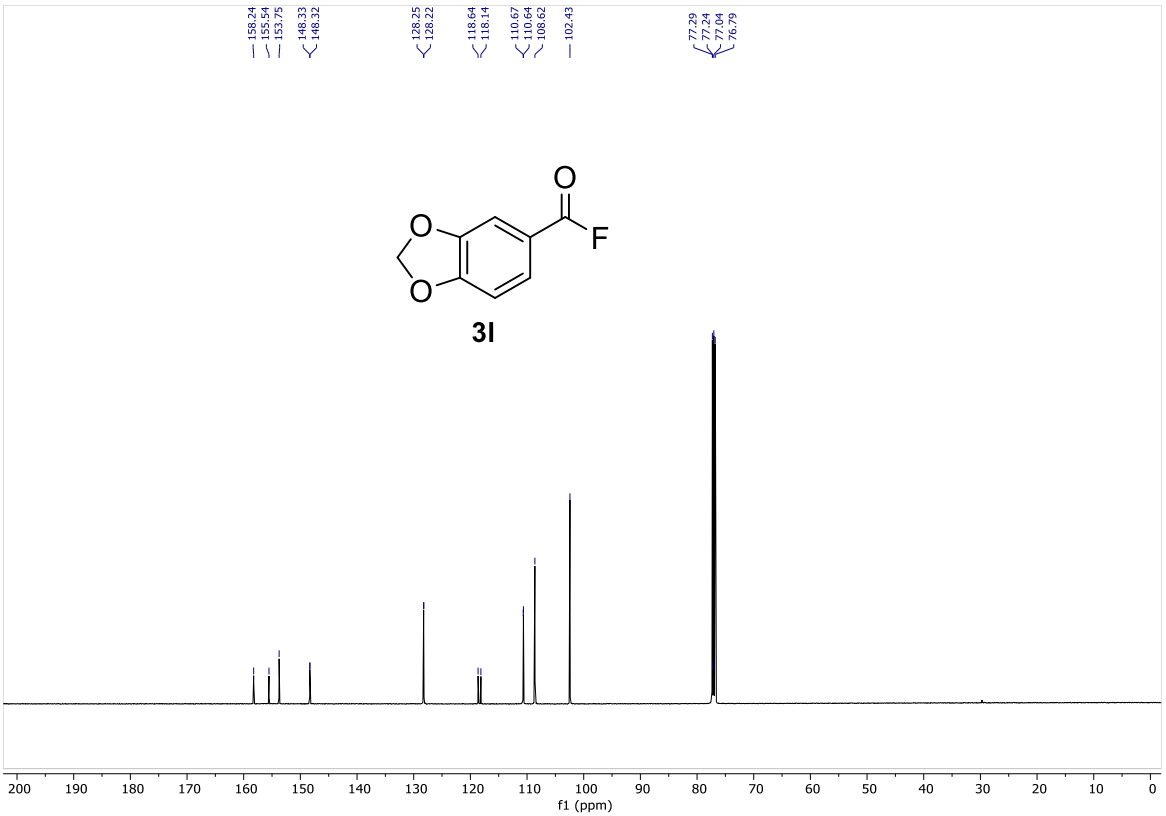

18.14
O
F
Ph
3j
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

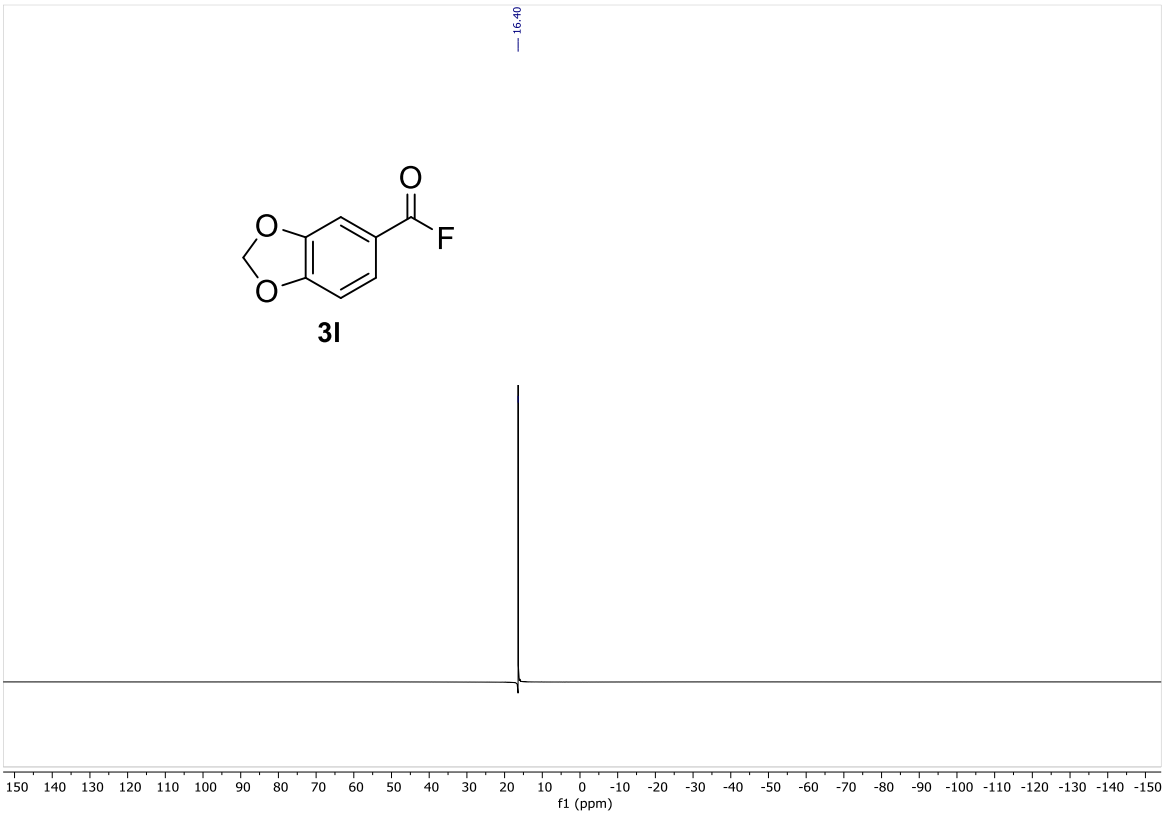

8.08
8.06
7.72
7.71
7.69
7.54
7.53
7.51
7.49
7.48
7.47
7.46
7.44
7.43
7.37
7.37
7.36
7.28
O
F
Ph
3k
1.00
1.02
1.05
4.06
2.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

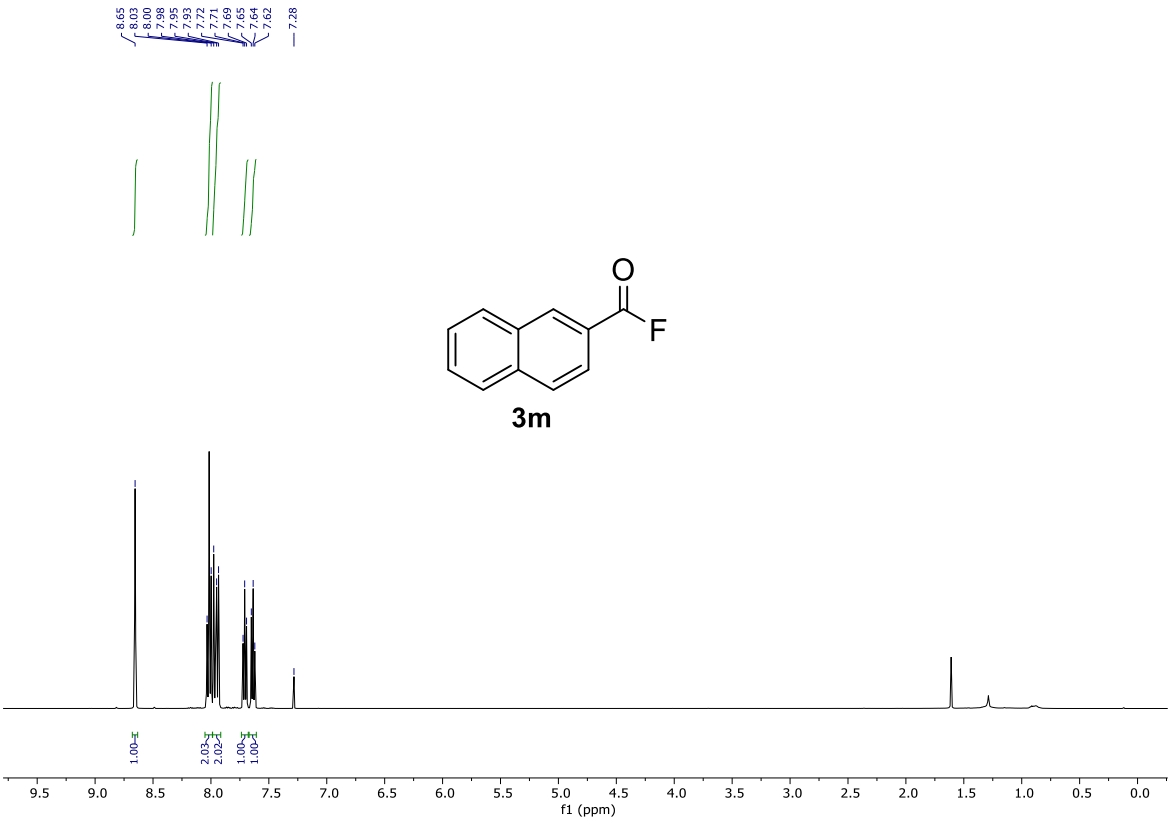

158.94
156.17
145.52
145.50
140.15
133.93
132.20
132.18
131.77
131.75
128.43
128.28
127.94
127.65
124.43
123.98
77.31
77.06
76.80
O
F
Ph
3k
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

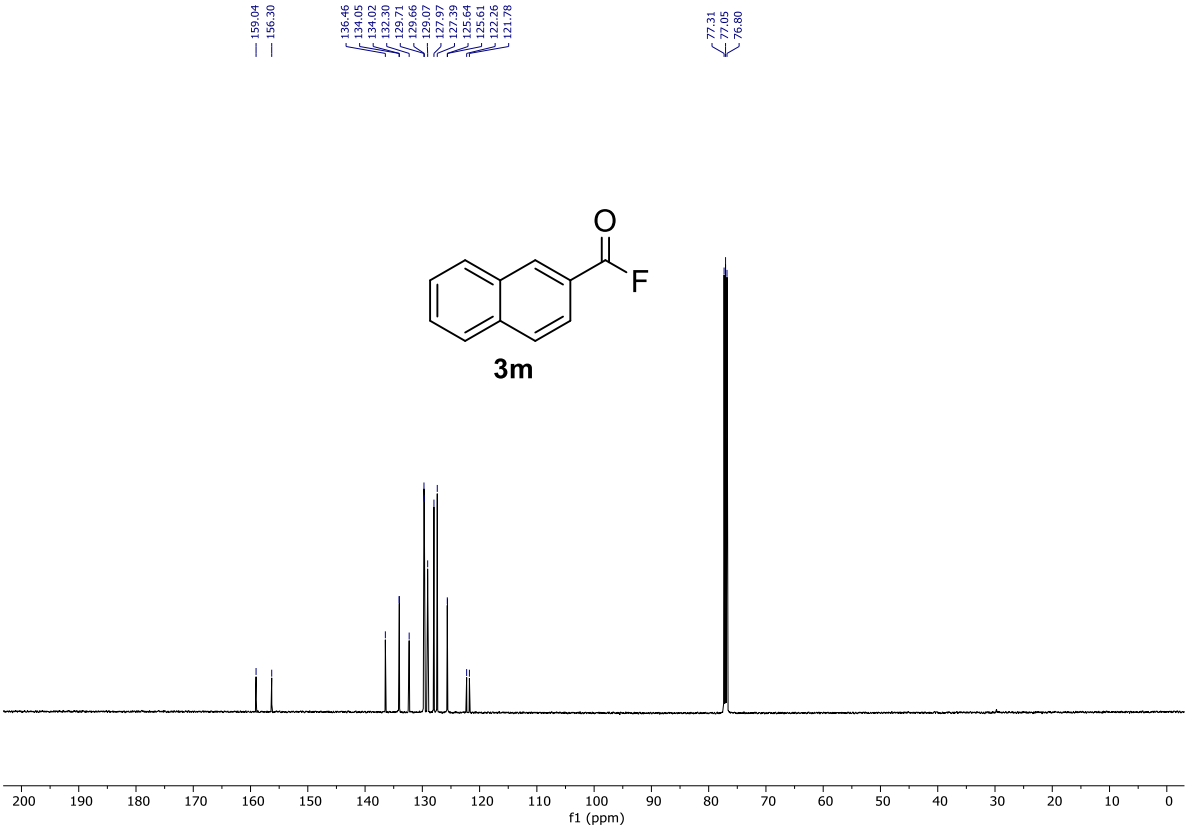

35.04
O
F
Ph
3k
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

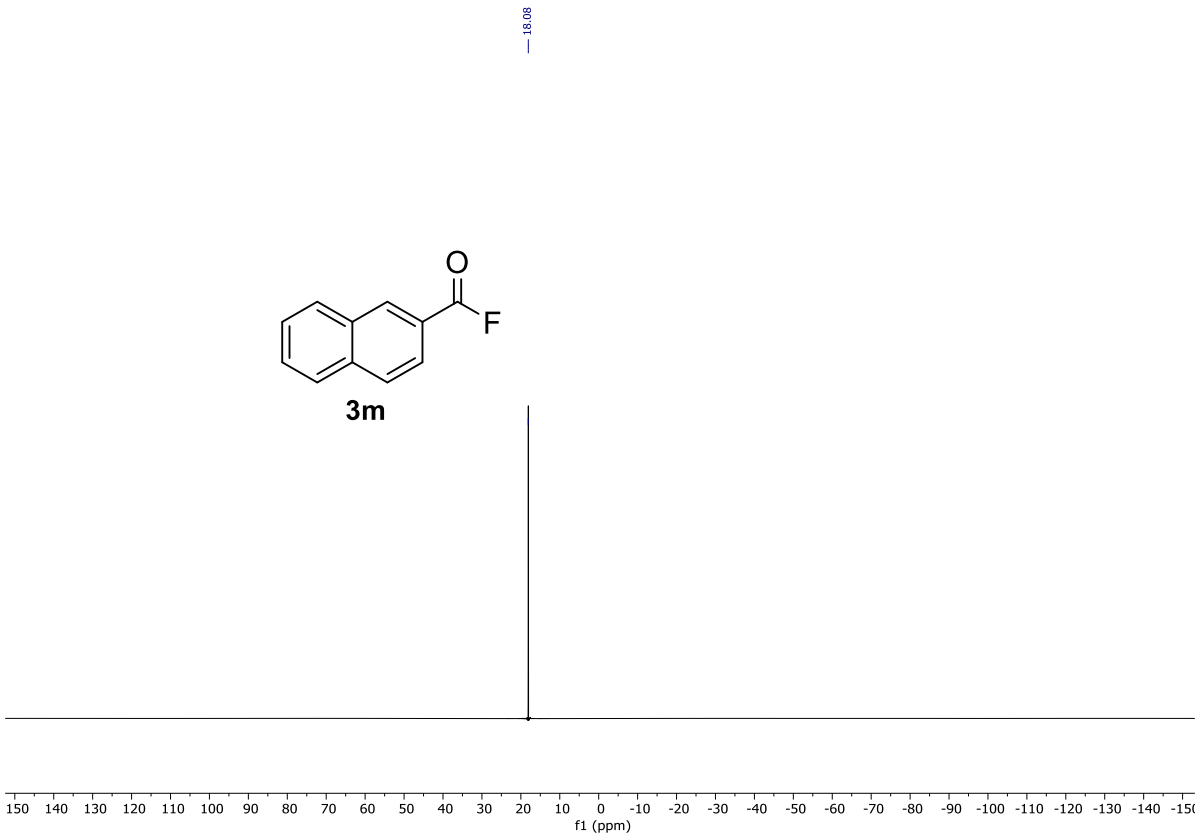

7.70
7.69
7.68
7.68
7.43
7.43
7.28
6.93
6.91
6.12
O
O
O
F
3l
1.01
1.00
1.02
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

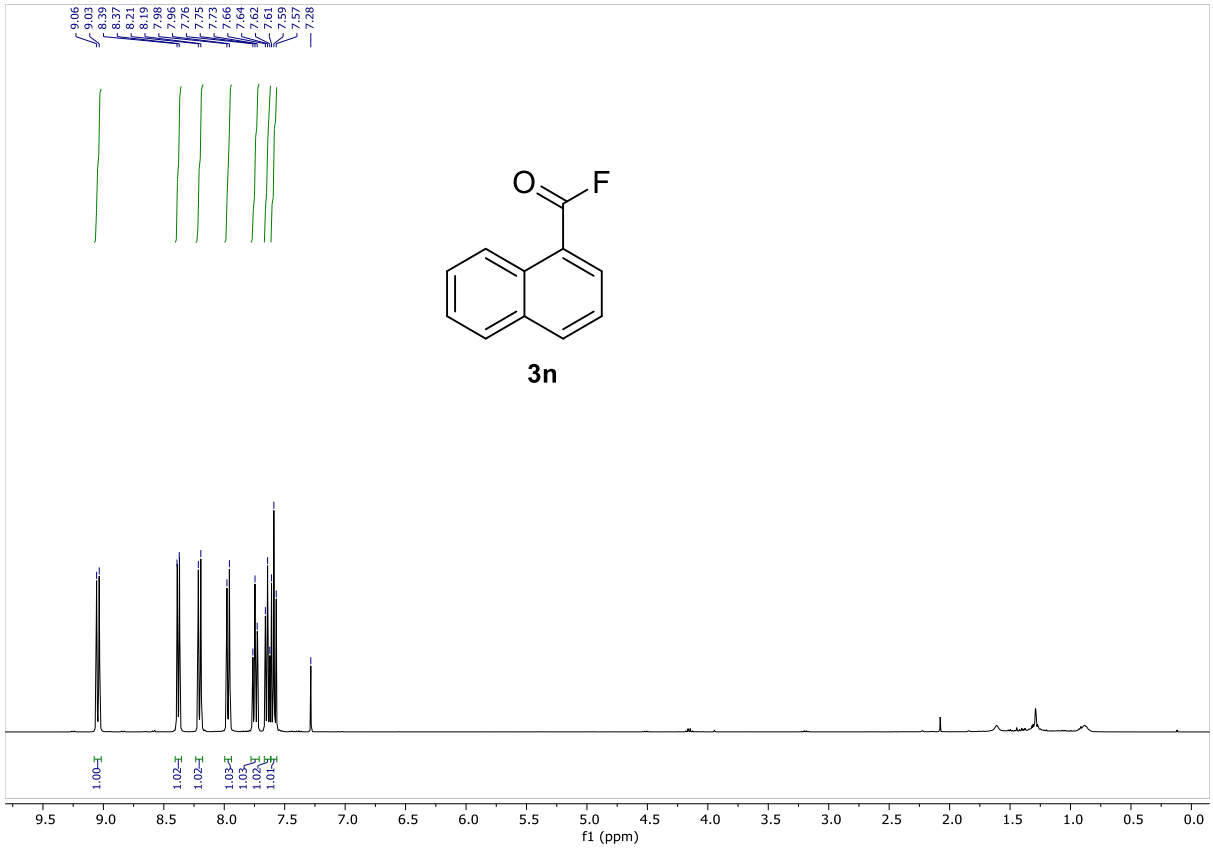

158.24
155.54
153.75
148.33
148.32
128.25
128.22
118.64
118.14
110.67
110.64
108.62
102.43
77.39
77.24
77.04
76.79
O
O
O
F
3l
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

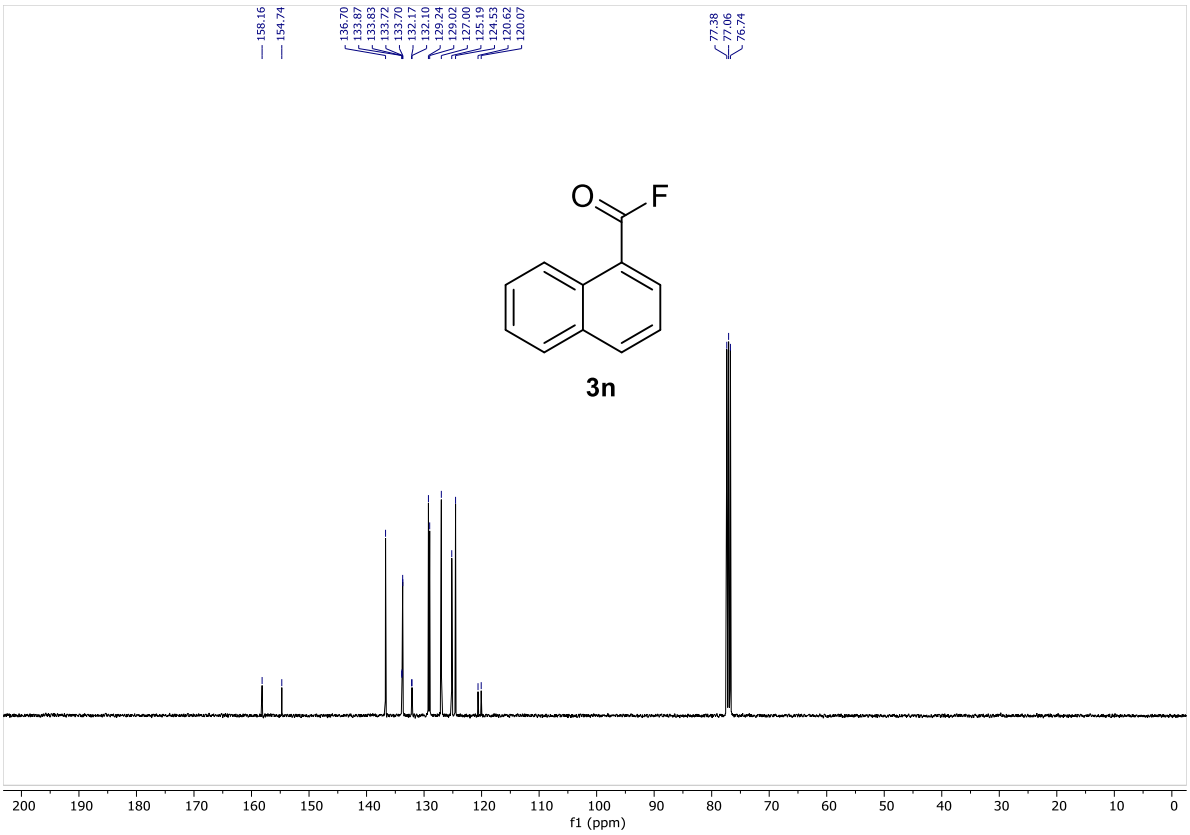

16.40
O
O
O
F
3l
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

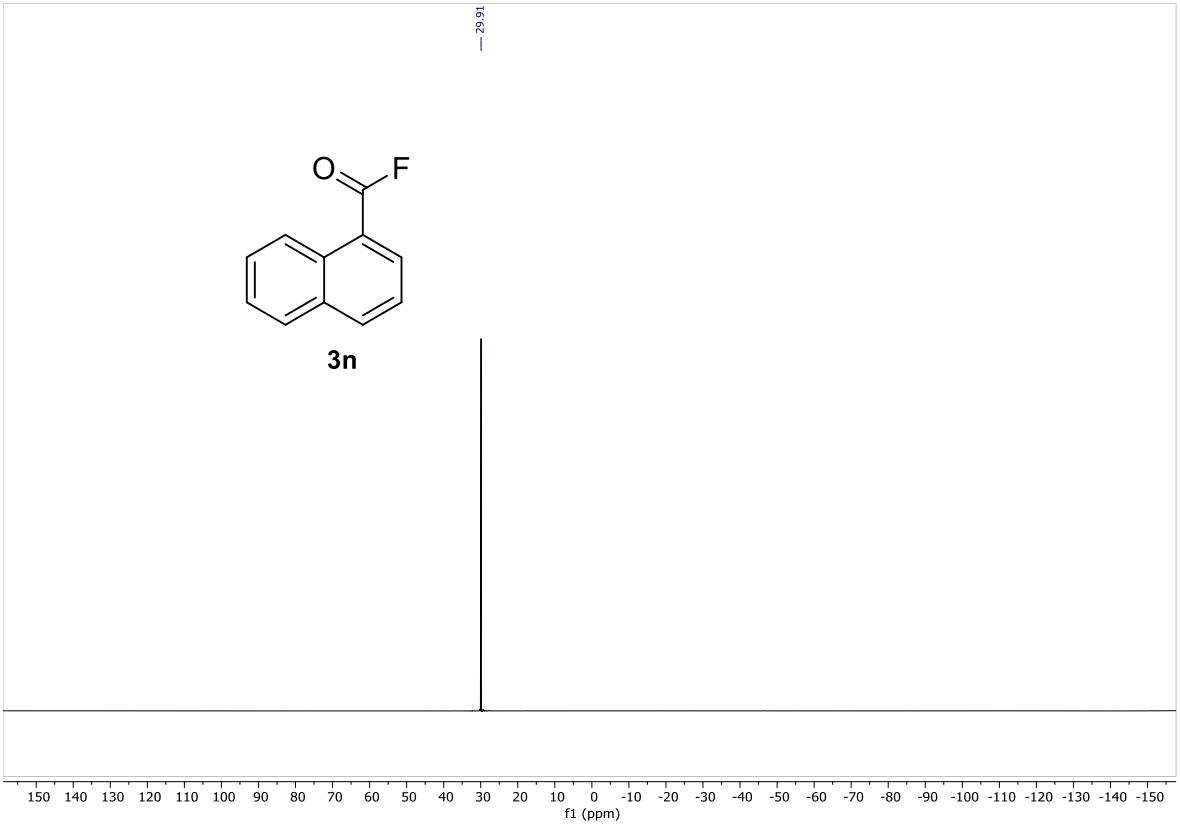

8.65
8.03
8.00
7.98
7.95
7.93
7.72
7.71
7.69
7.65
7.64
7.62
7.28
O
F
3m
1.00
2.03
2.02
1.00
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

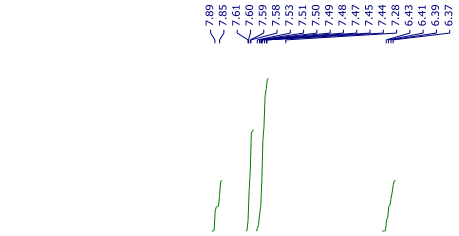

159.04
156.30
136.46
134.05
134.02
132.30
129.71
129.66
129.07
127.97
127.39
125.64
125.61
122.26
121.78
77.31
77.05
76.80
O
F
3m
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

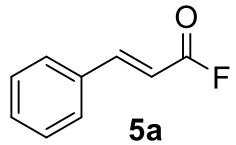

18.08
O
F
3m
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140
f1 (ppm)


9.06
9.03
8.39
8.37
8.21
8.19
7.98
7.96
7.76
7.75
7.73
7.66
7.64
7.62
7.61
7.59
7.57
7.28
O
F
3n
1.00
1.02
1.02
1.03
1.03
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 0.0
f1 (ppm)

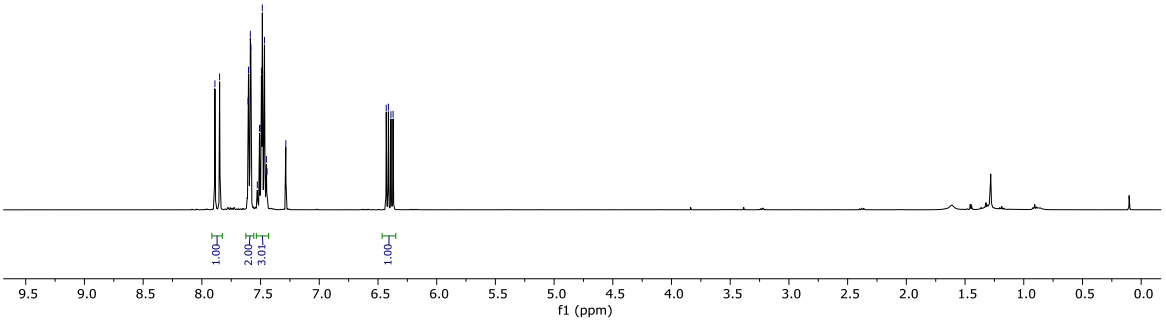

158.16
154.74
136.70
133.87
133.83
133.72
133.70
132.17
132.10
129.24
129.02
127.00
125.19
124.53
120.62
120.07
77.38
77.06
76.74
O
F
3n
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


29.91
O
F
3n
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

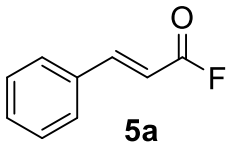

7.89
7.85
7.61
7.60
7.59
7.58
7.53
7.51
7.50
7.49
7.48
7.47
7.45
7.44
7.28
6.43
6.41
6.39
6.37


O
F

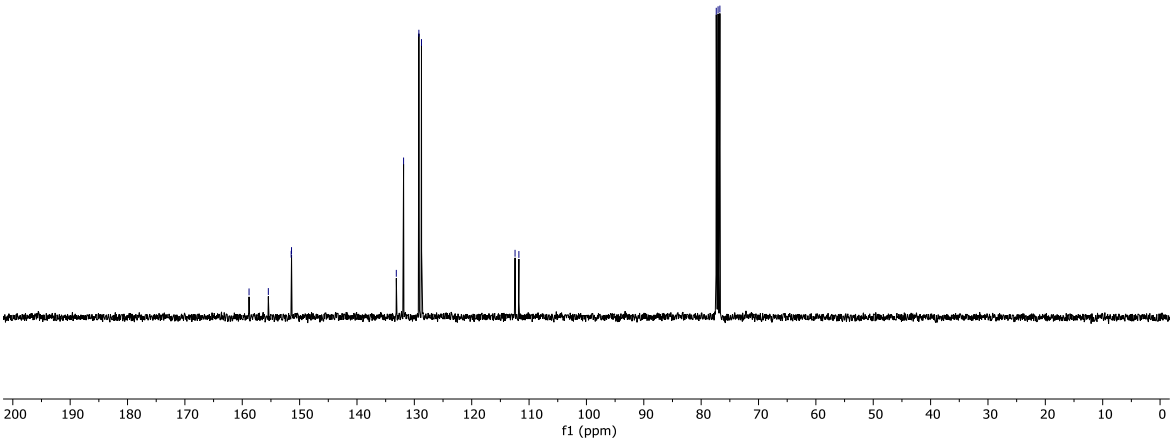
5a

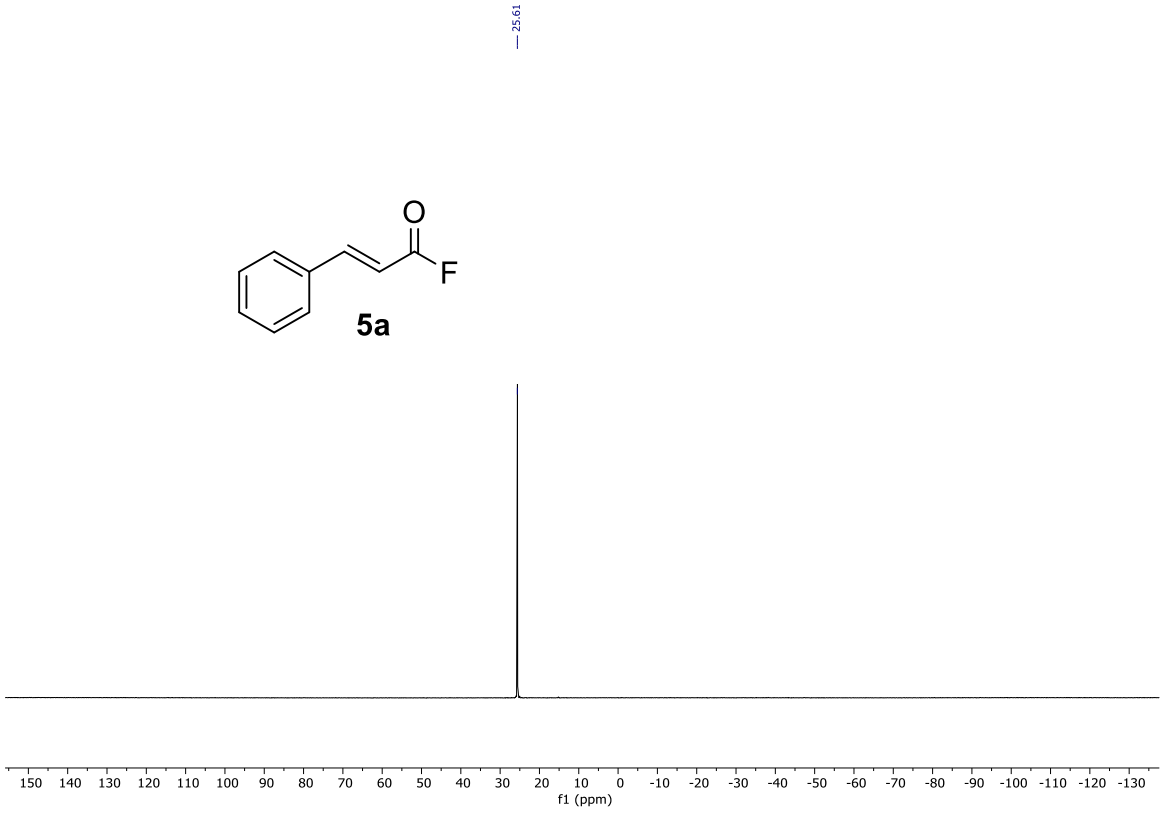

1.00
2.00
3.01
1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

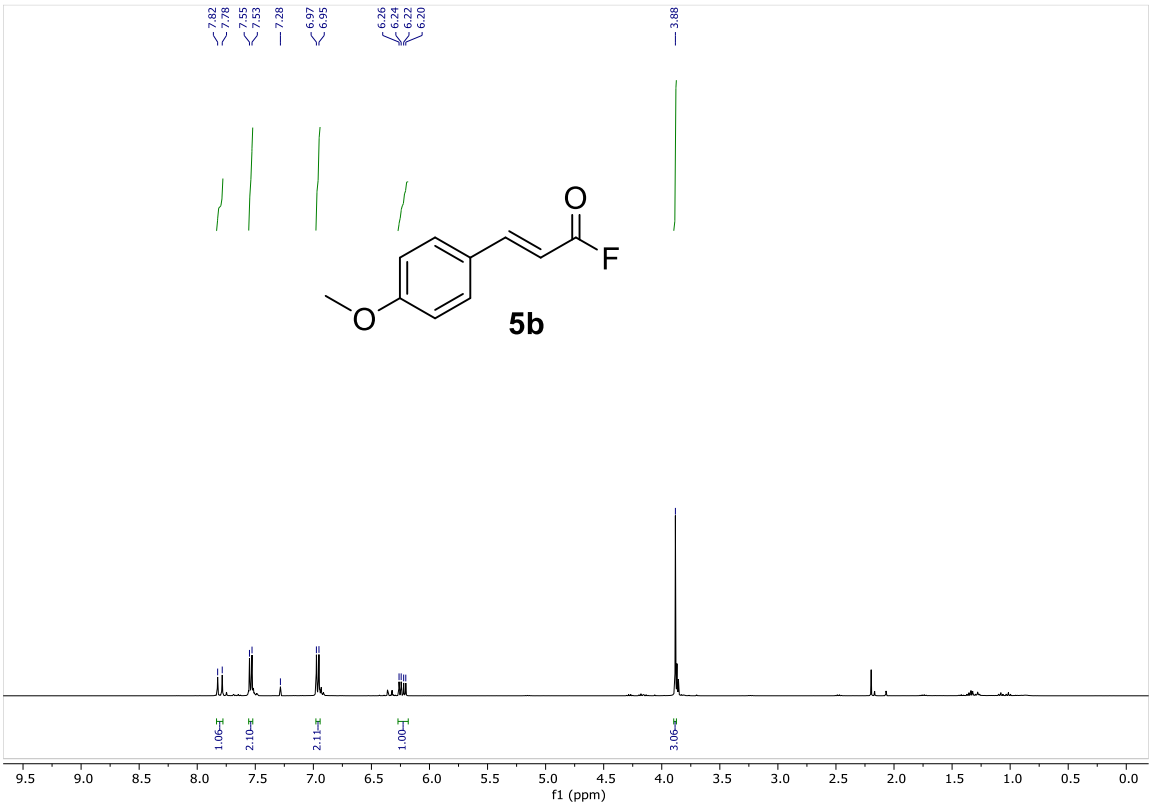

158.82
155.45
151.47
151.41
133.14
131.88
129.21
128.75
112.44
111.78
77.37
77.05
76.73

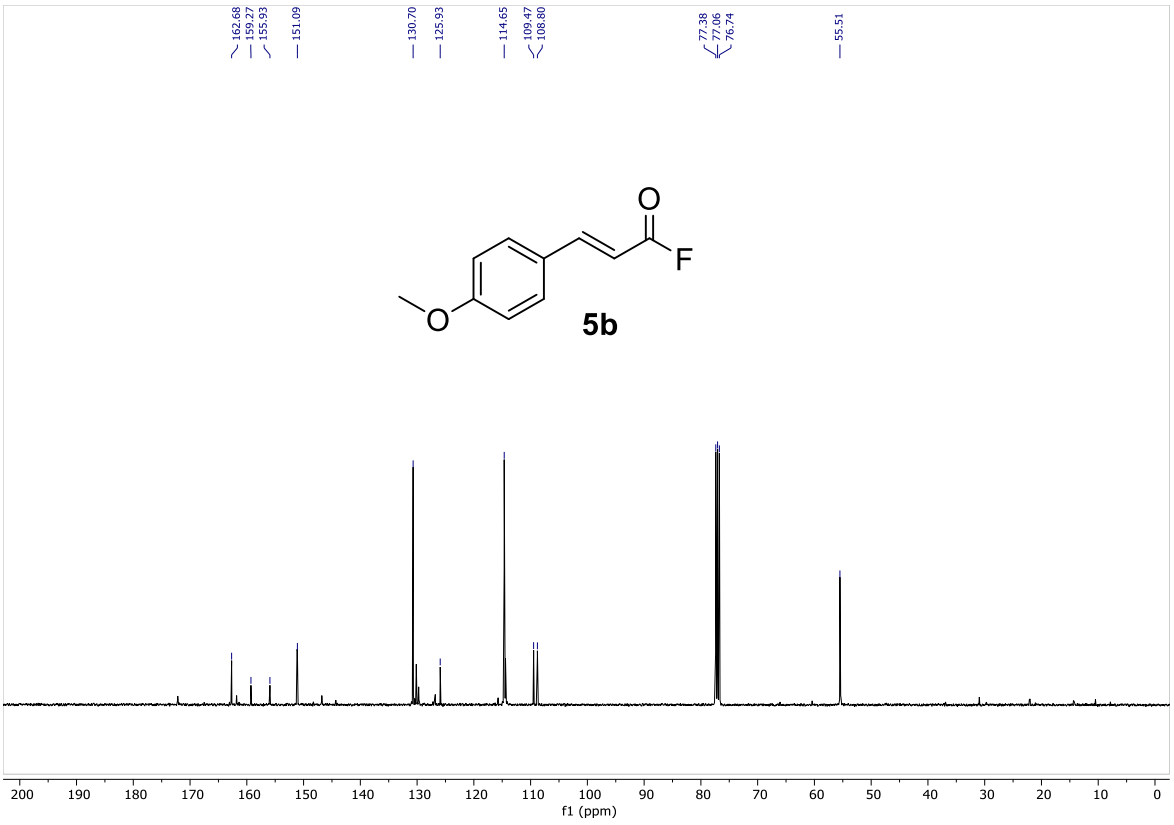

O
F

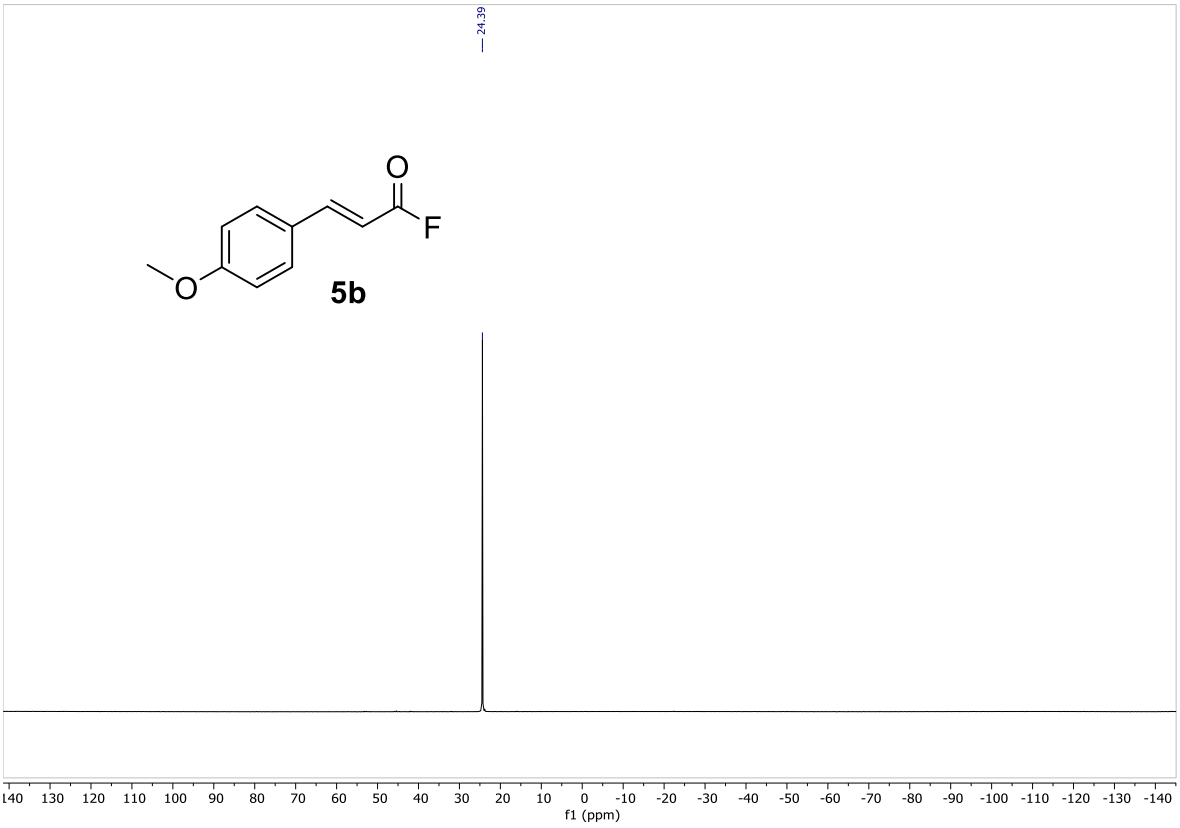
5a

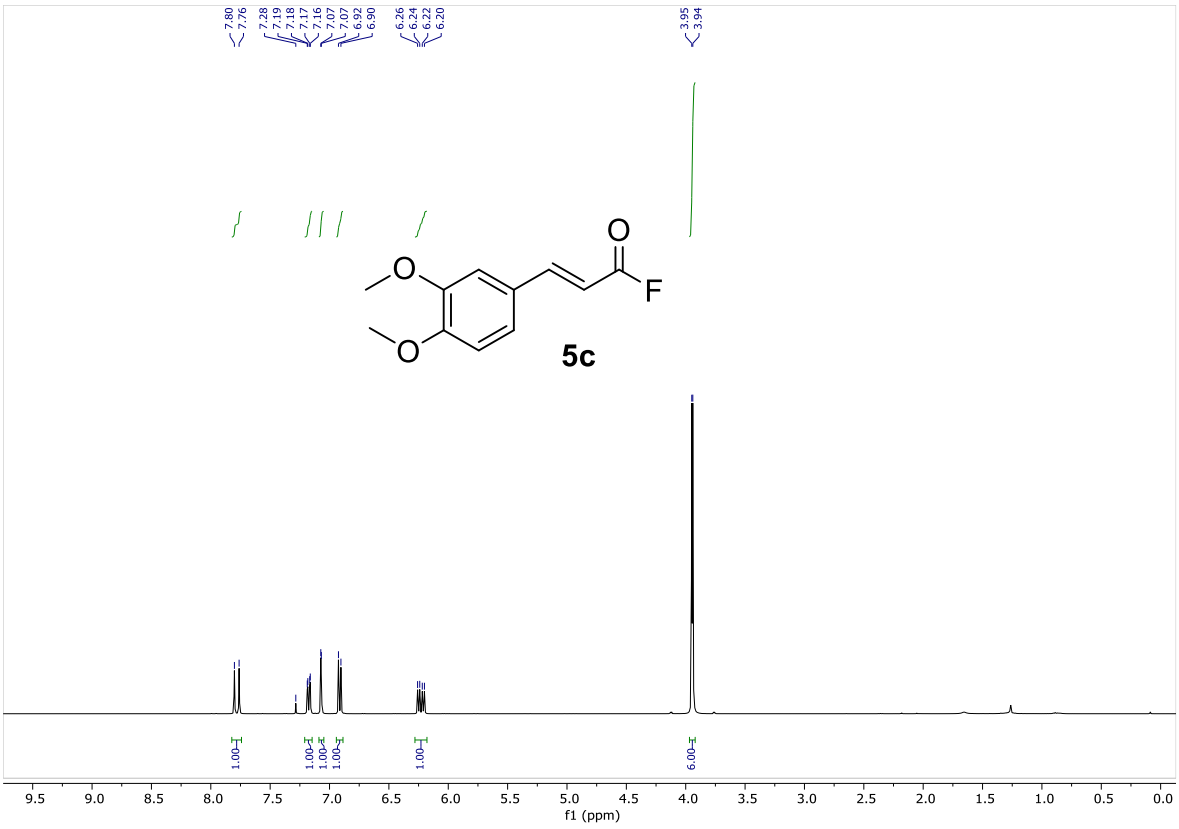

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

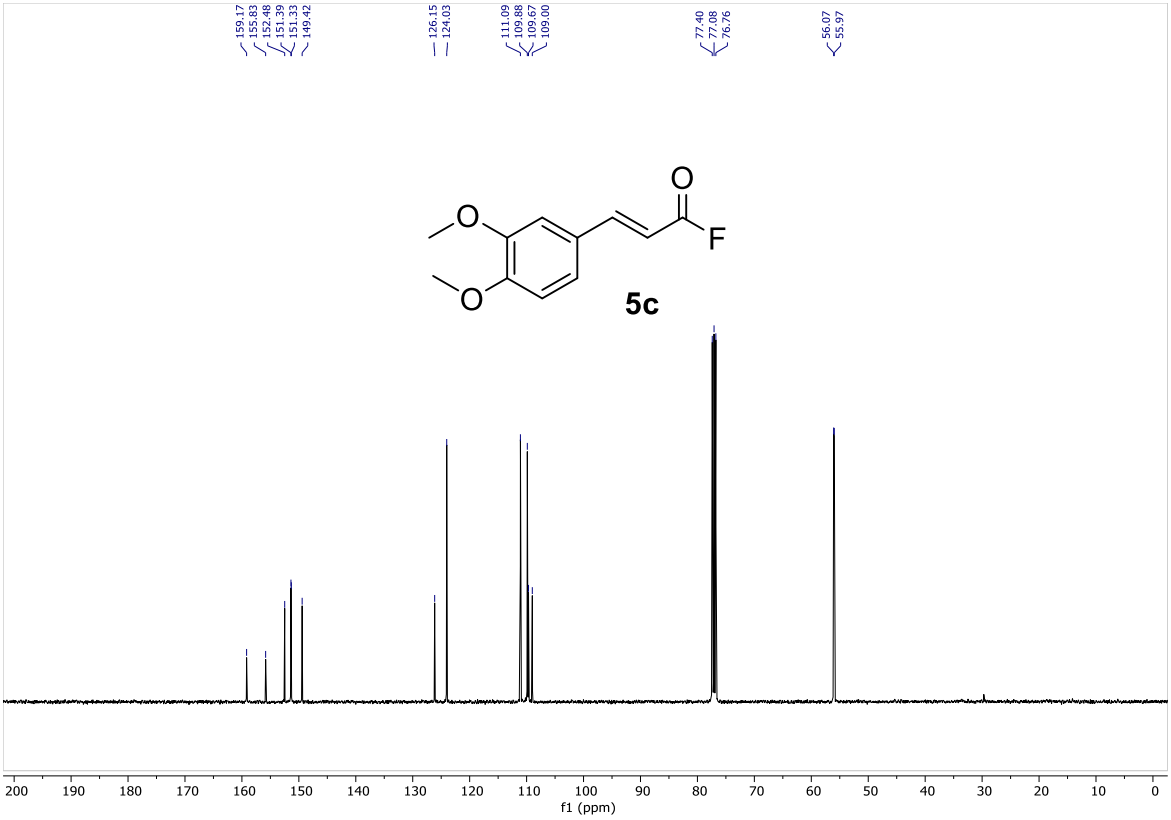

25.61
O
F
5a
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130
f1 (ppm)

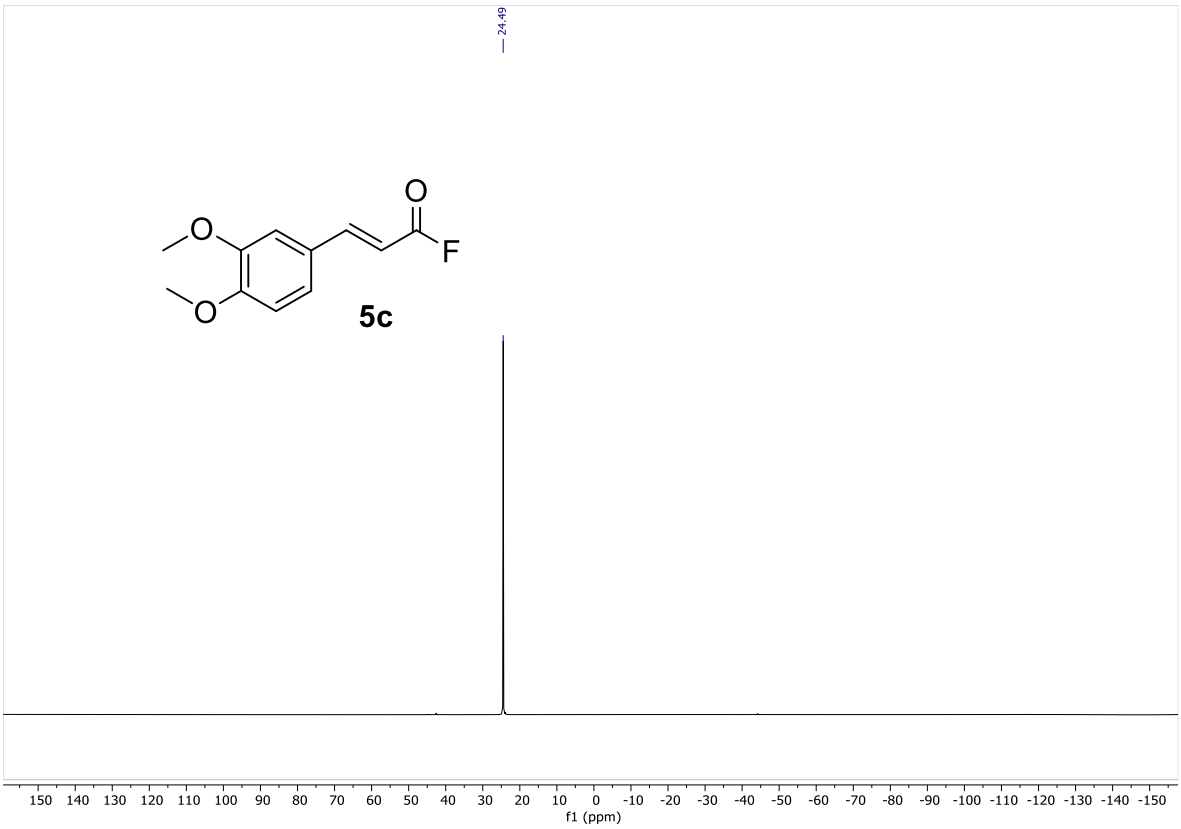

7.82
7.78
7.55
7.53
7.28
6.97
6.95
6.26
6.24
6.22
6.20
3.88
O
F
O
5b
1.06
2.10
2.11
1.00
3.06
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

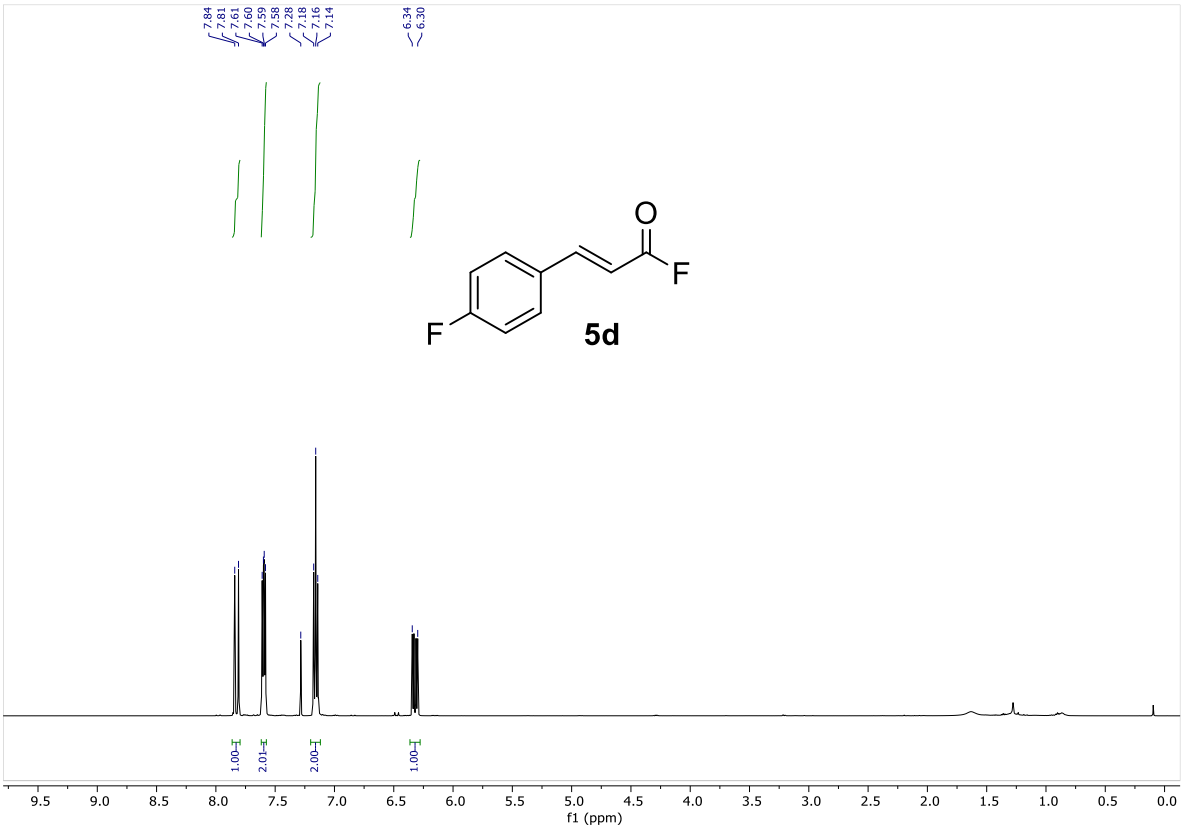

162.68
159.27
155.93
151.09
130.70
125.93
114.65
109.47
108.80
77.38
77.06
76.74
55.51
O
F
O
5b
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

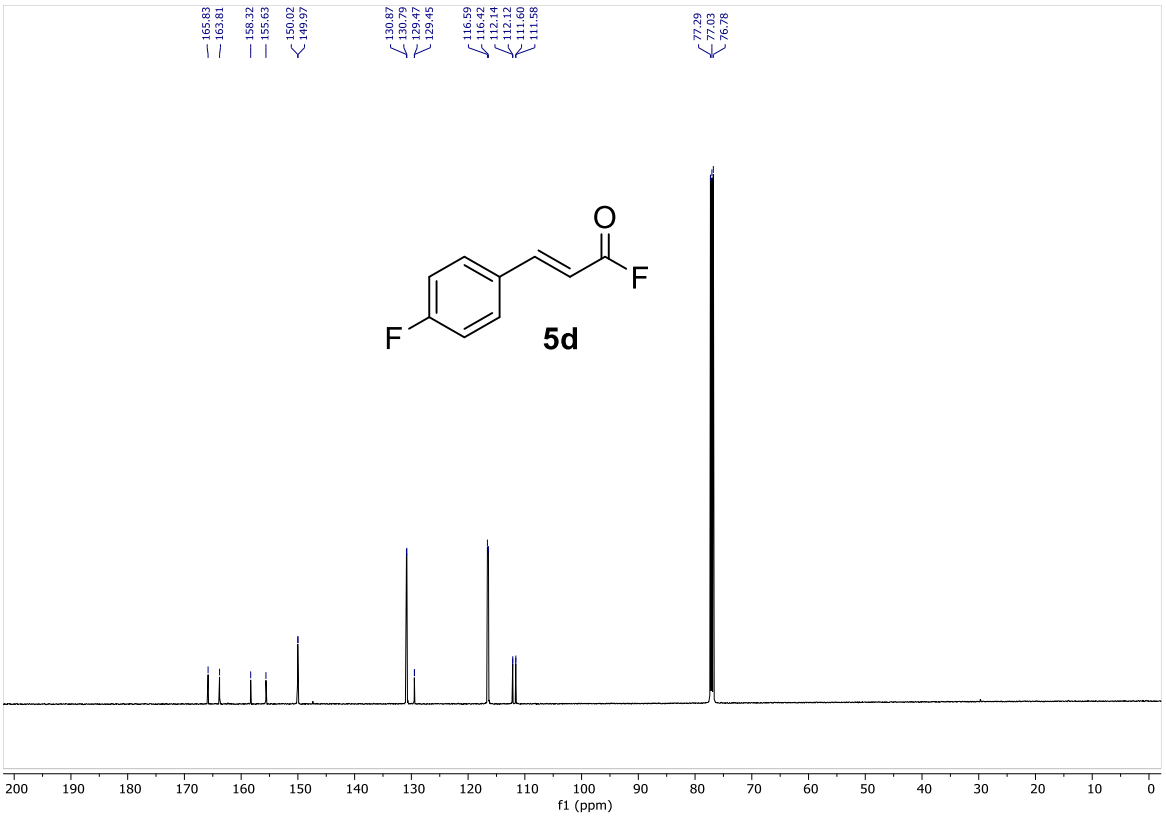

24.39
O
F
O
5b
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140
f1 (ppm)

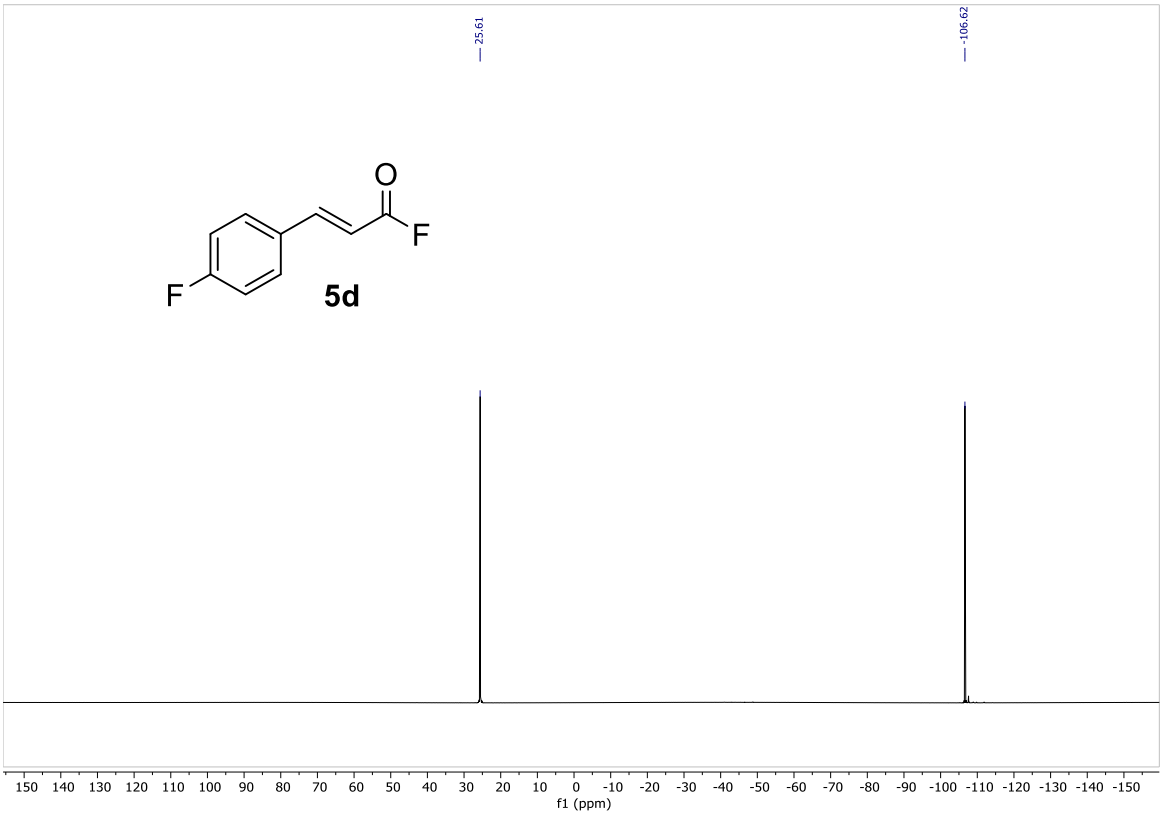

7.80
7.76
7.28
7.19
7.18
7.17
7.16
7.07
7.07
6.92
6.90
6.26
6.24
6.22
6.20
3.95
3.94
O
O
O
F
5c
1.00
1.00
1.00
1.00
1.00
6.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

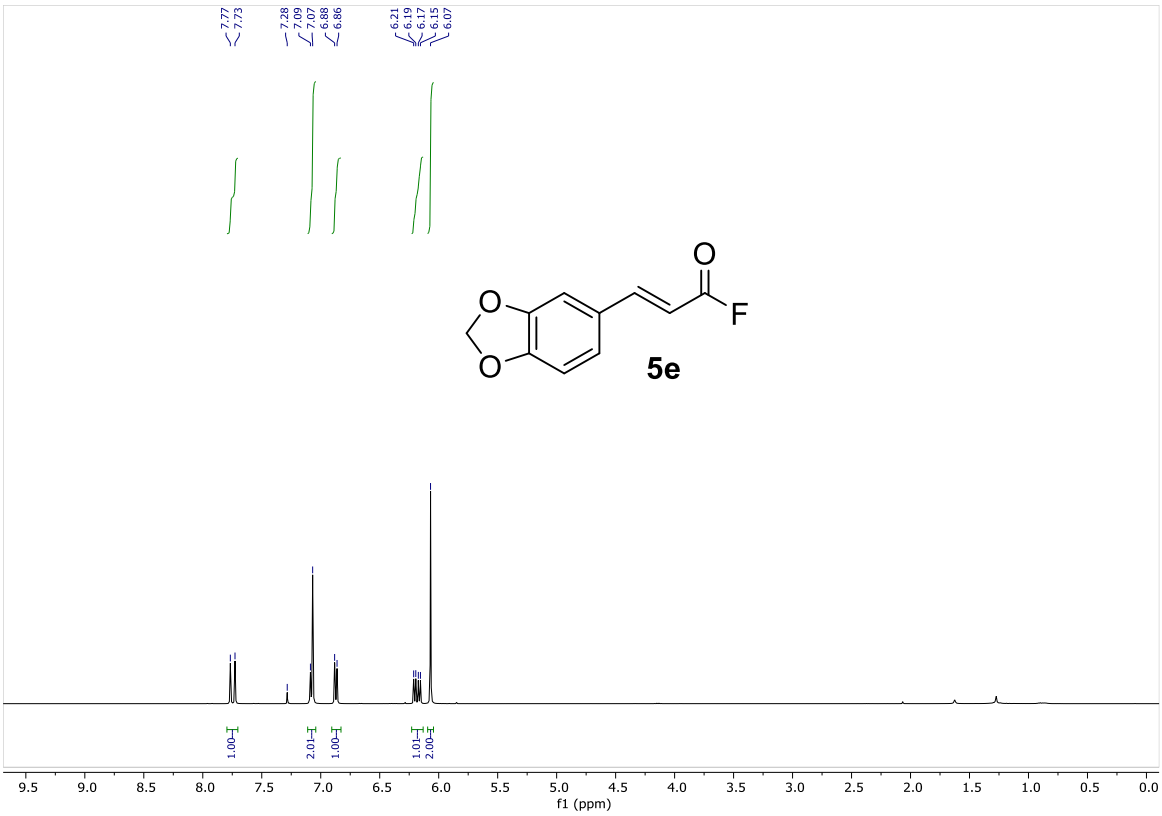

159.17
155.83
152.48
151.39
151.33
149.42
126.15
124.03
111.09
109.88
109.67
109.00
77.40
77.08
76.76
56.07
55.97
O
O
O
F
5c
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

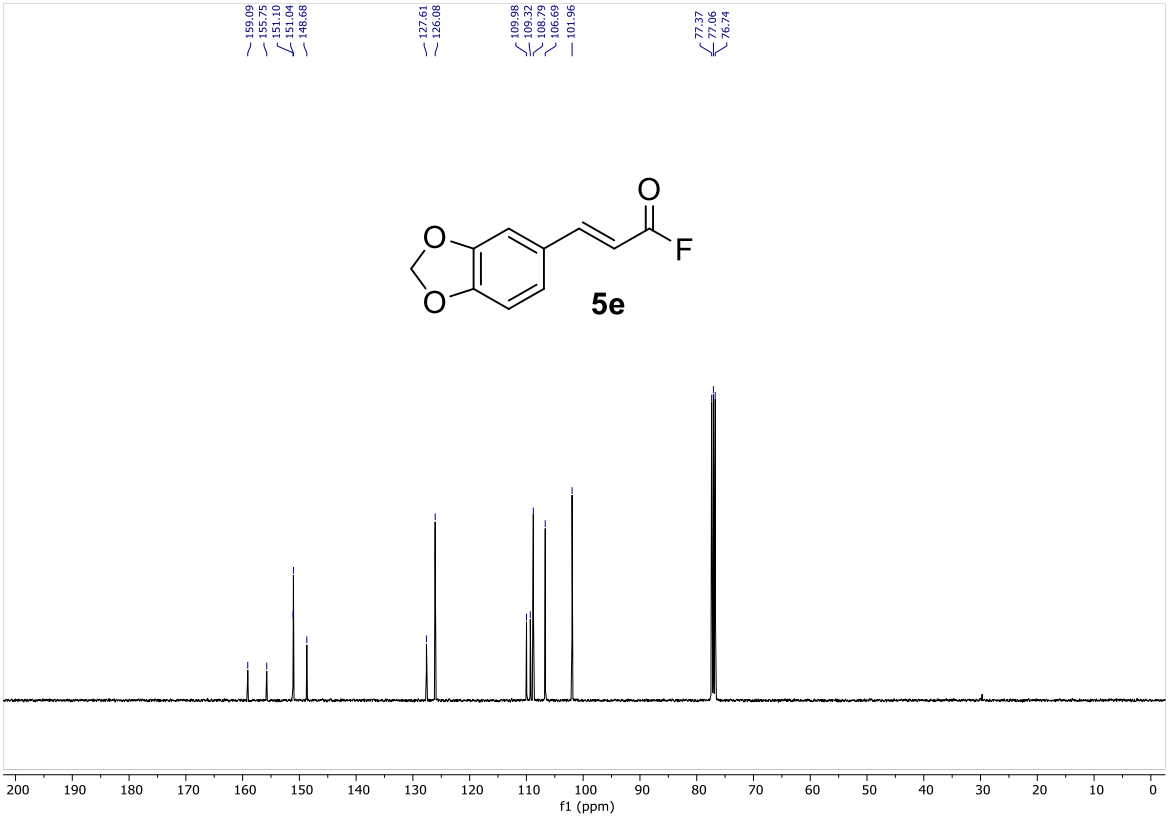

24.49
O
O
O
F
5c
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

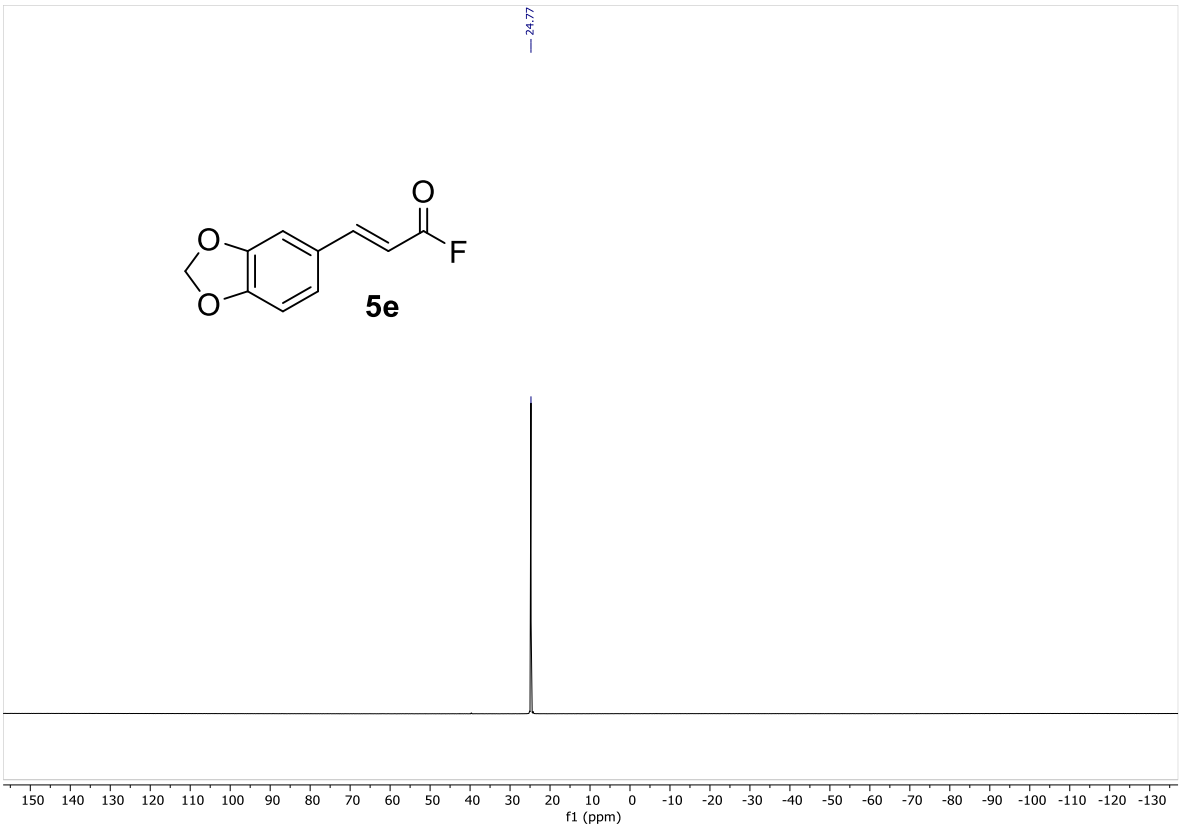

7.84
7.81
7.61
7.60
7.59
7.58
7.28
7.18
7.16
7.14
6.34
6.30
O
F
F
5d
1.00
2.01
2.00
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

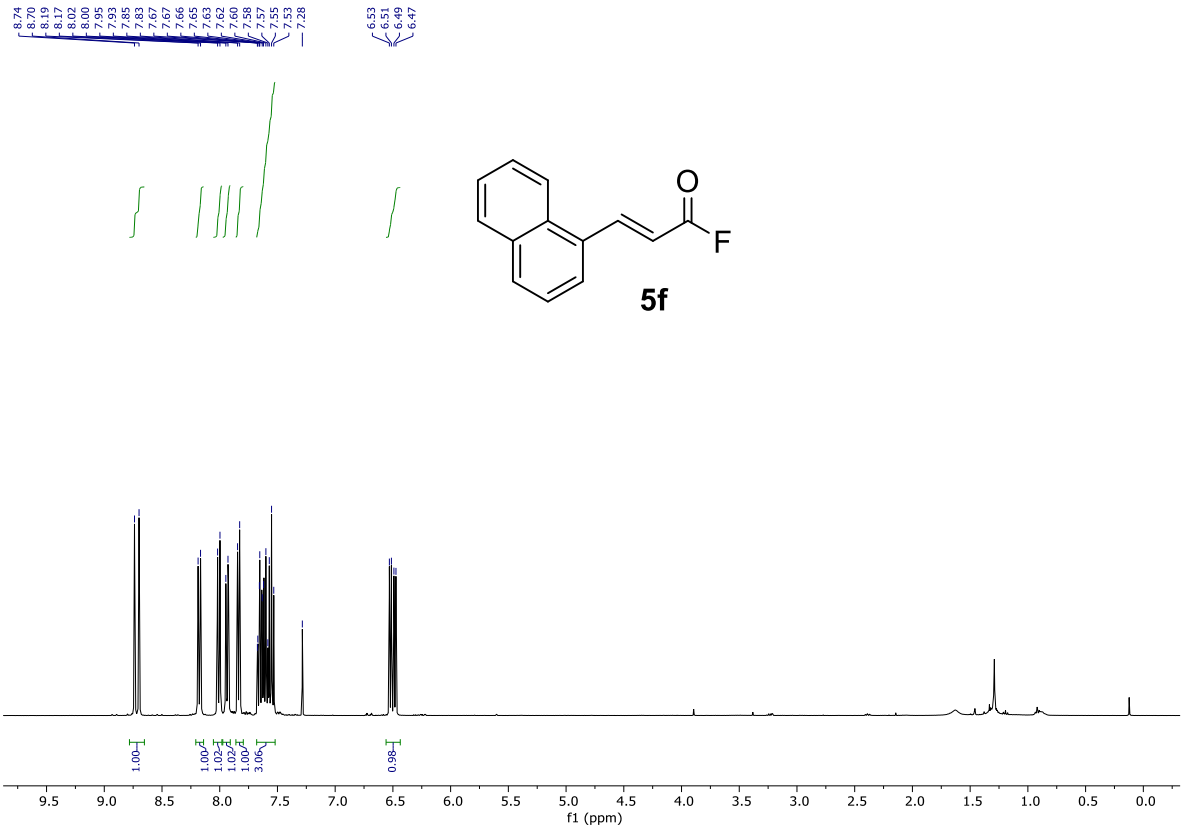

165.83
163.81
158.32
155.63
150.02
149.97
130.87
130.79
129.47
129.45
116.59
116.42
112.14
112.12
111.60
111.58
77.29
77.03
76.78
O
F
F
5d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

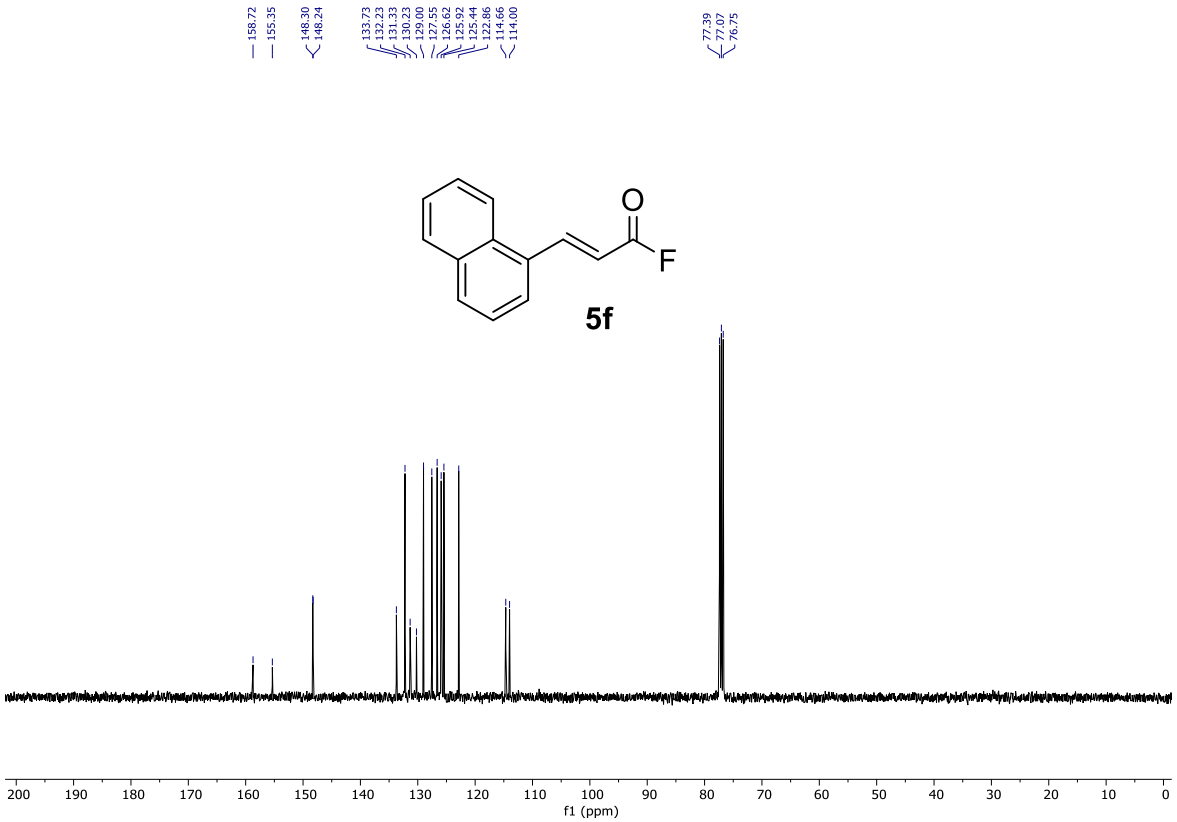

25.61
-106.62
O
F
F
5d
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

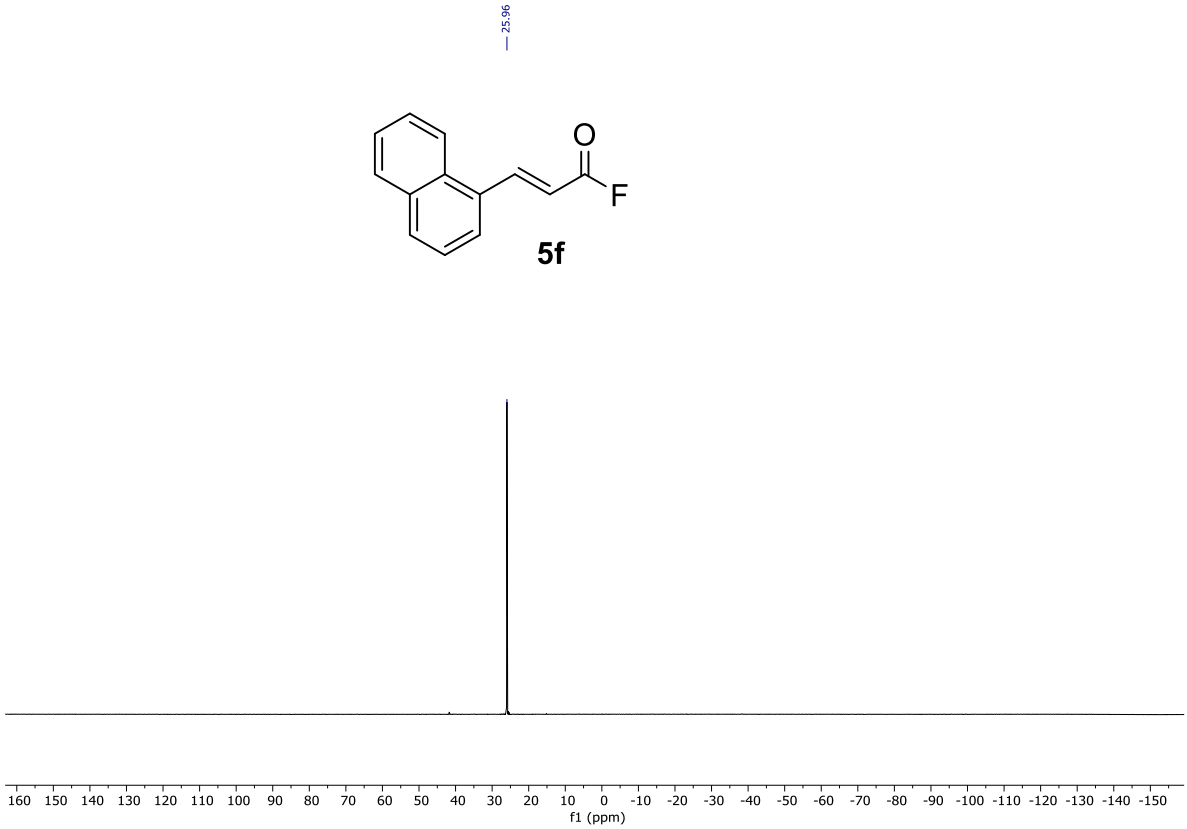

7.77
7.73
7.28
7.09
7.07
6.88
6.86
6.21
6.19
6.17
6.15
6.07
O
O
O
F
5e
1.00
2.01
1.00
1.01
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

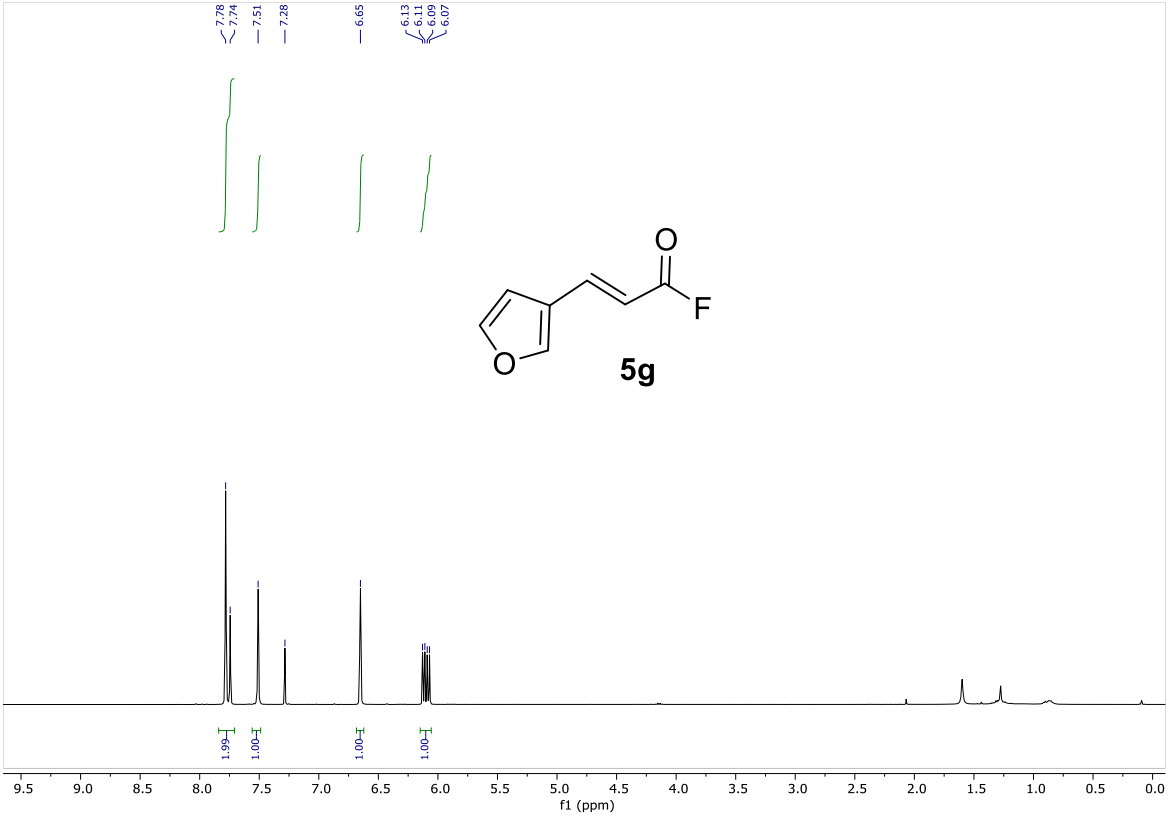

159.09
155.75
151.10
151.04
148.68
127.61
126.08
109.98
109.32
108.79
106.69
101.96
77.37
77.06
76.74
O
O
O
F
5e
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

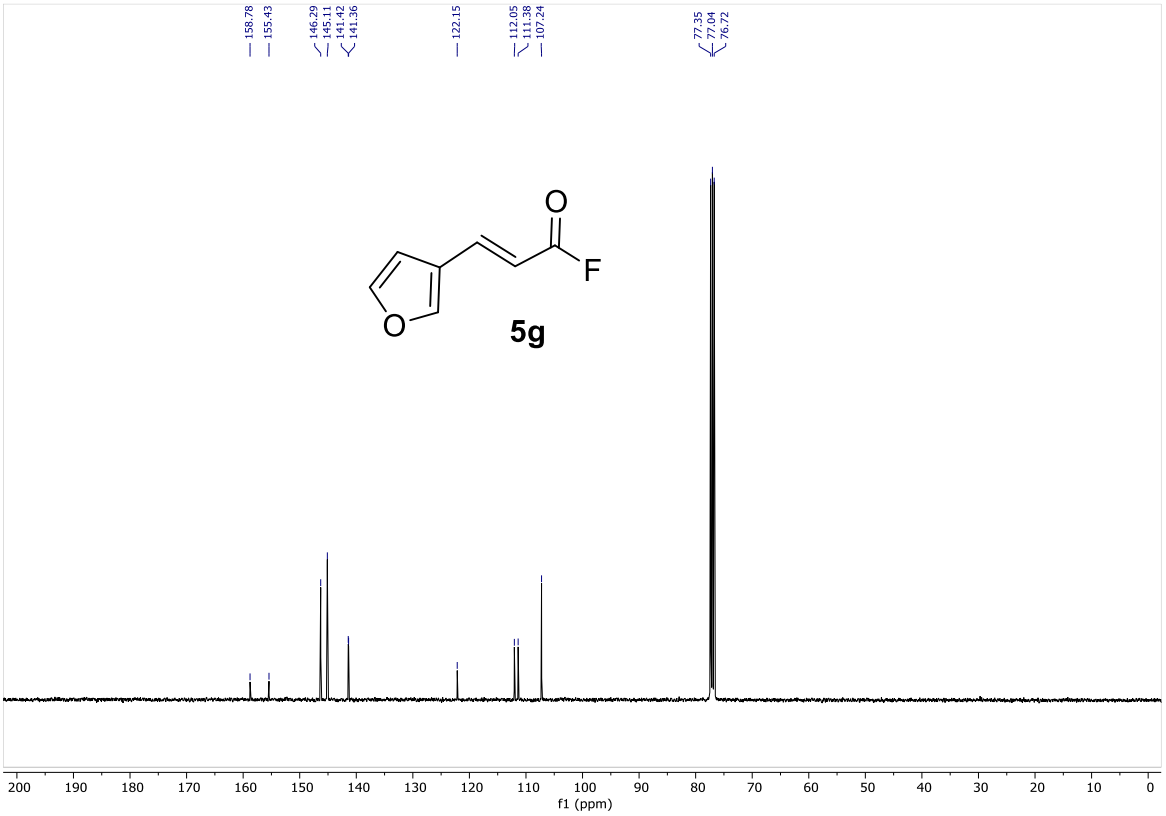

24.77
O
O
O
F
5e
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130
f1 (ppm)

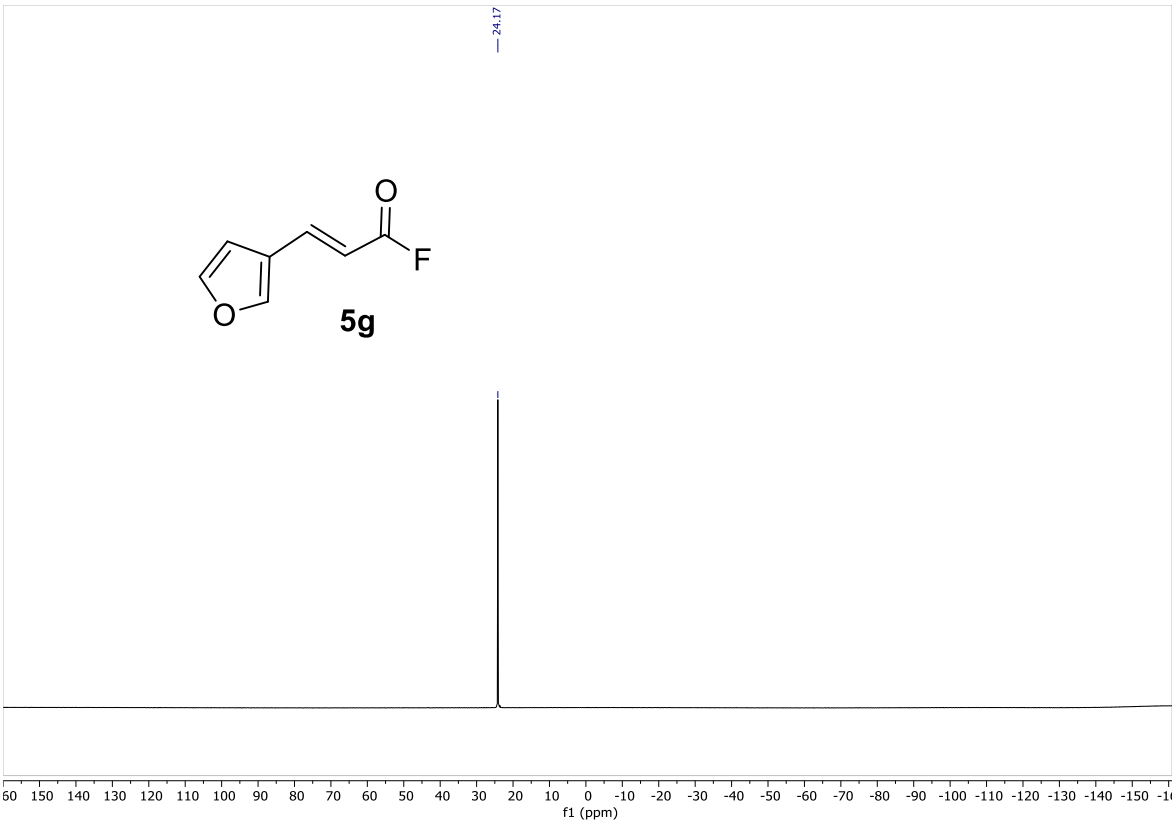

8.74
8.70
8.19
8.17
8.02
8.00
7.95
7.93
7.85
7.83
7.67
7.67
7.66
7.65
7.63
7.62
7.60
7.58
7.57
7.55
7.53
7.28
6.53
6.51
6.49
6.47
O
F
5f
1.00
1.00
1.02
1.02
1.00
3.86
0.98
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

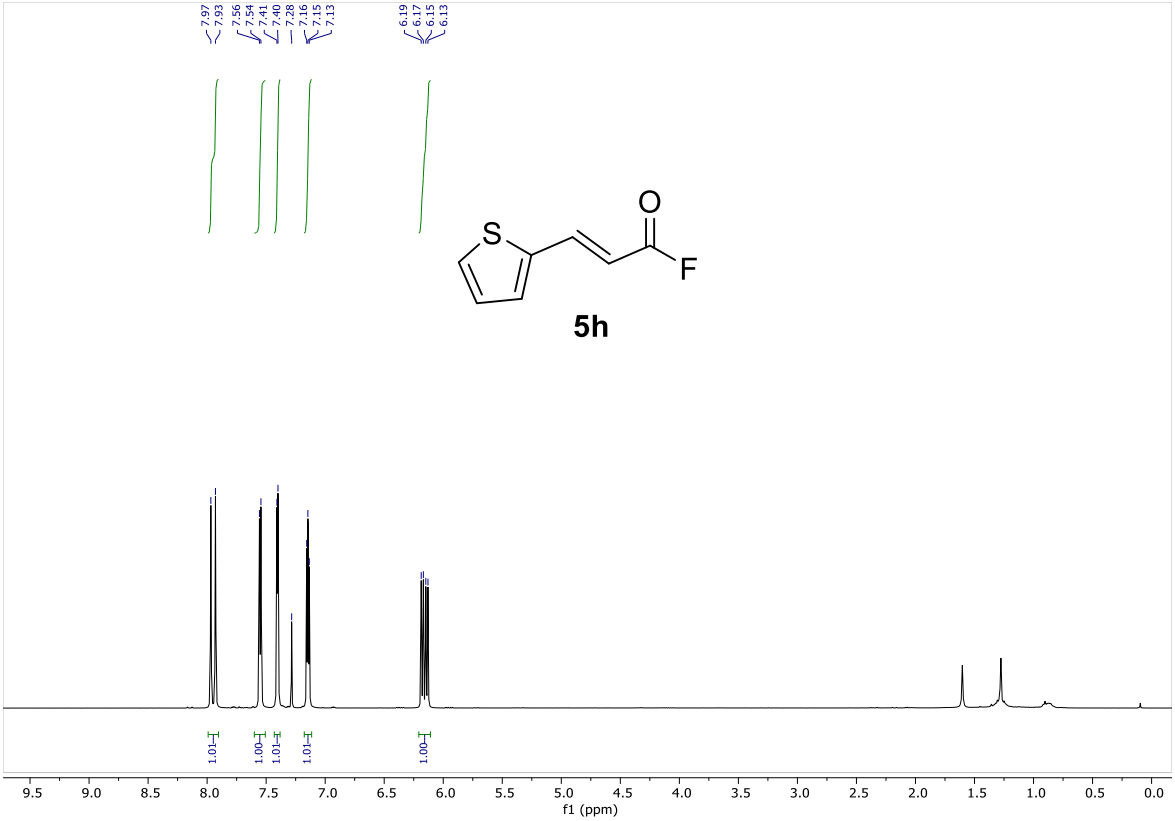

158.72
155.35
148.30
148.24
133.73
132.23
131.33
130.23
129.00
127.55
126.62
125.92
125.44
122.86
114.66
114.00
77.39
77.07
76.75
O
F
5f
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

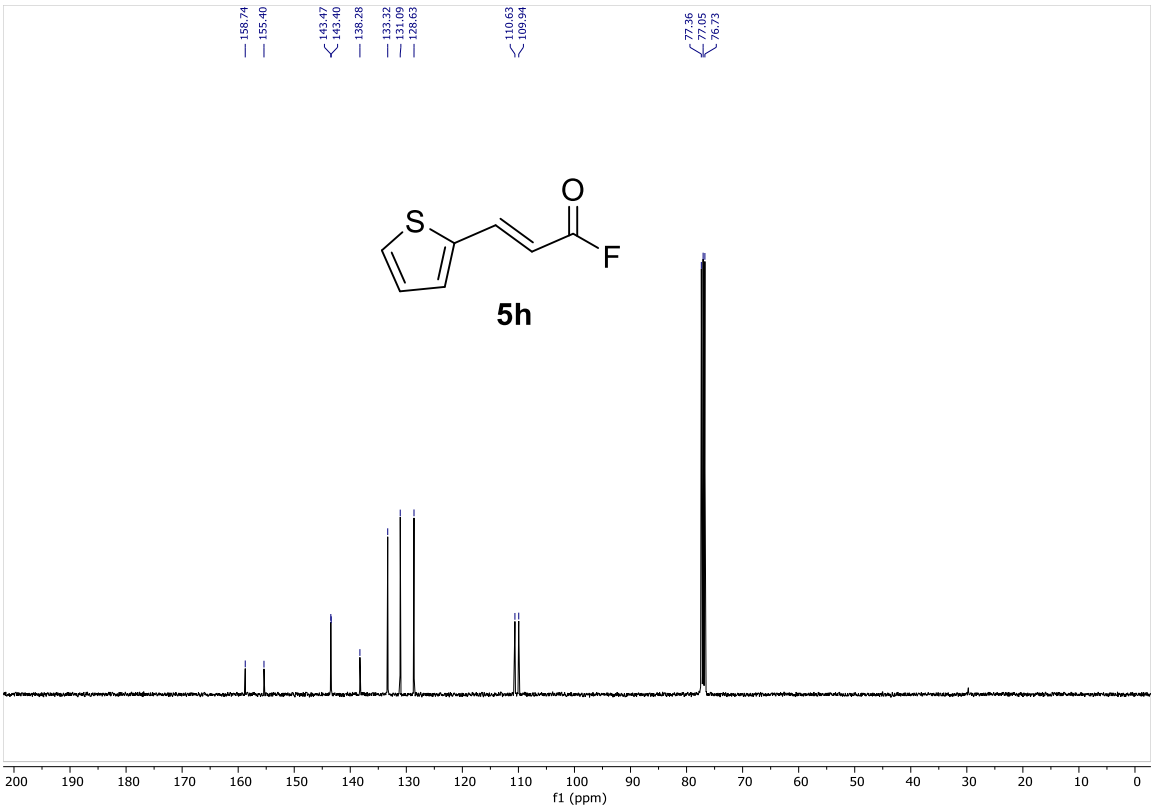

25.96
O
F
5f
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

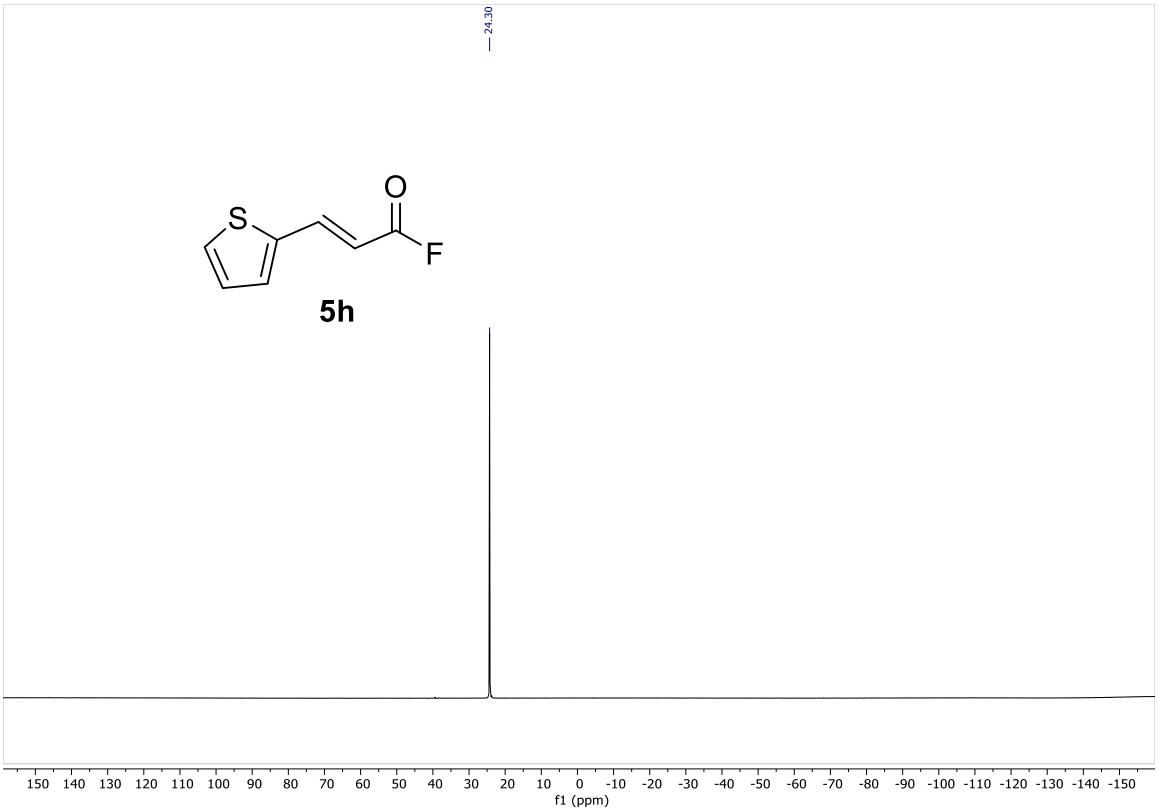

7.78
7.74
7.51
7.28
6.65
6.13
6.11
6.09
6.07
O
F
O
5g
1.99
1.00
1.00
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

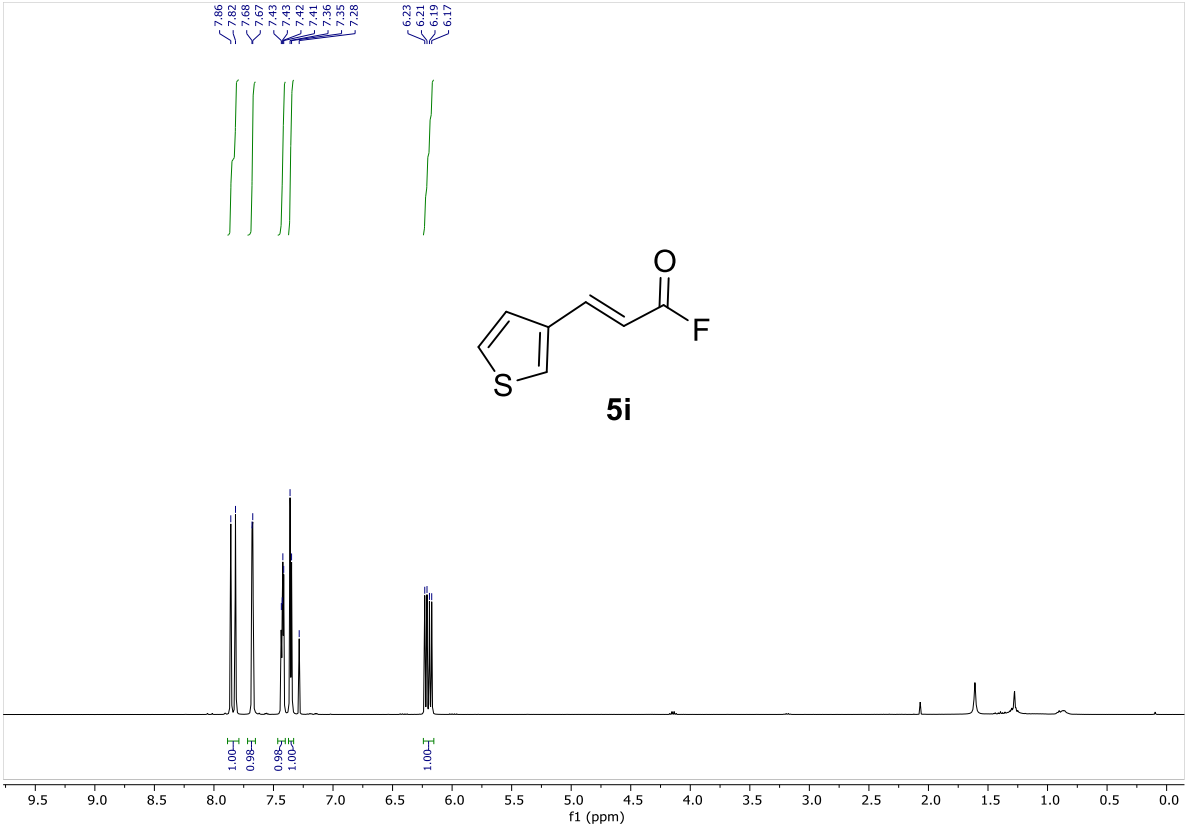

158.78
155.43
146.29
145.11
141.42
141.36
122.15
112.05
111.38
107.24
77.35
77.04
76.72
O
F
O
5g
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

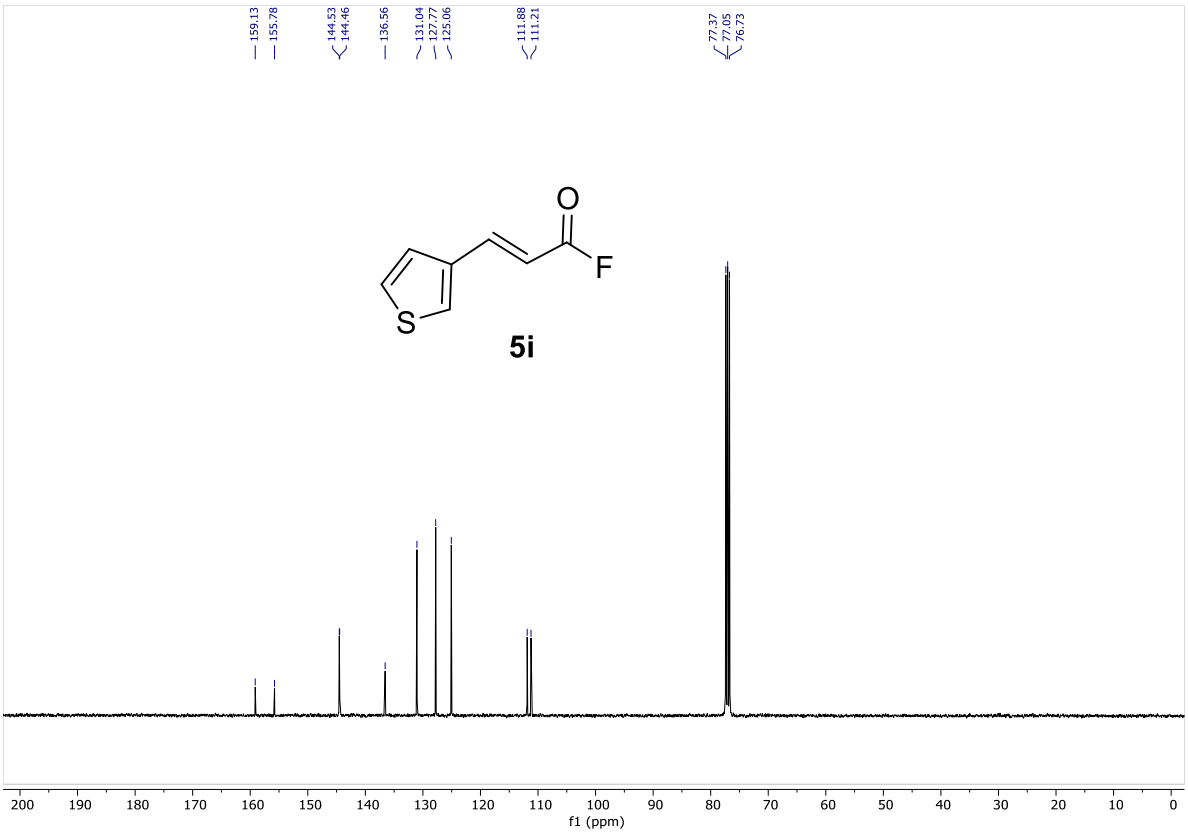

24.17
O
F
O
5g
60 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

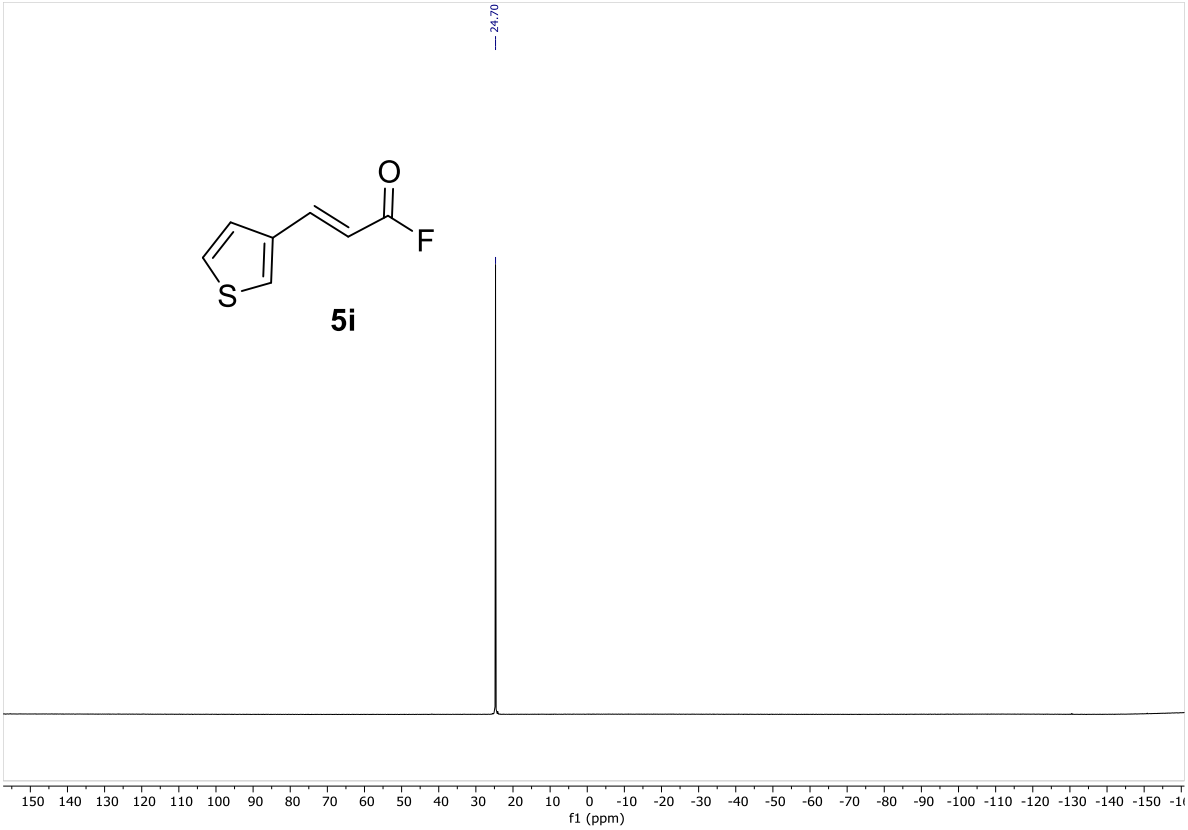

7.97
7.93
7.56
7.54
7.41
7.40
7.28
7.16
7.15
7.13
6.19
6.17
6.15
6.13
S
O
F
5h
1.01
1.00
1.01
1.01
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

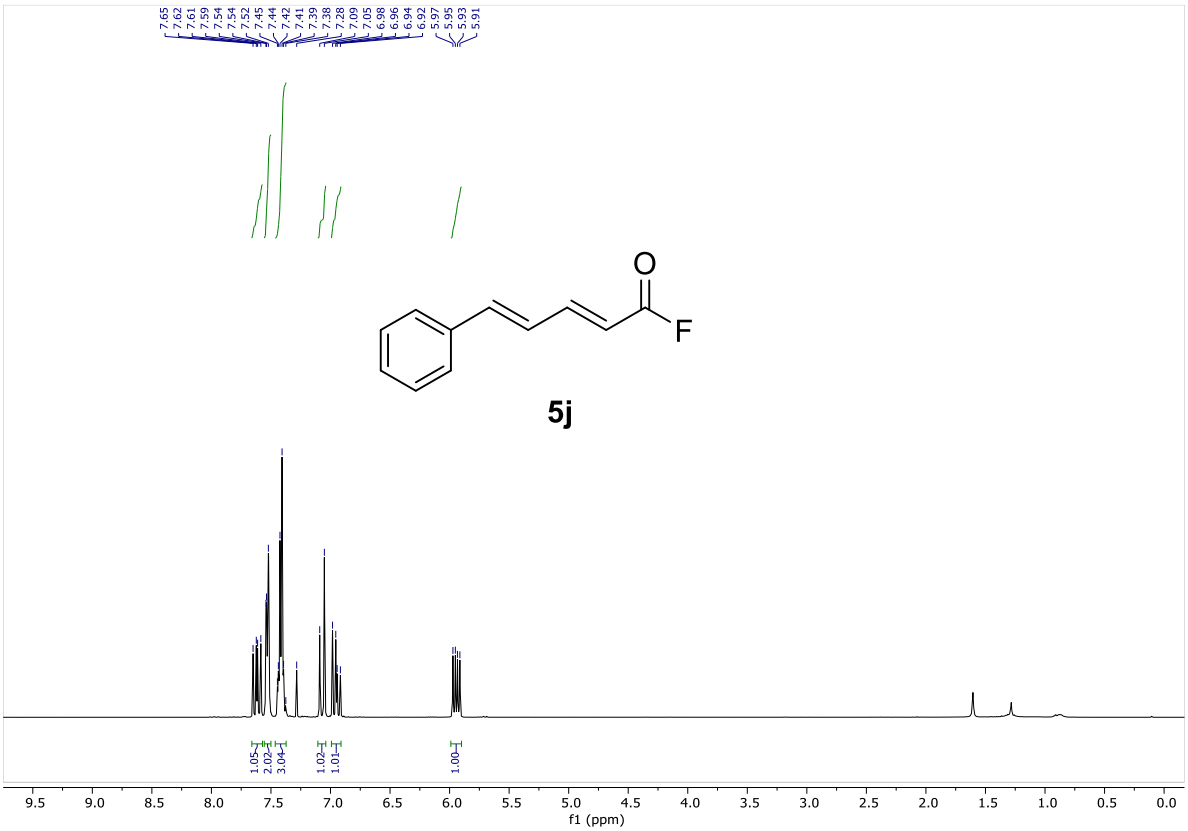

158.74
155.40
143.47
143.40
138.28
133.32
131.09
128.63
110.63
109.94
77.36
77.05
76.73
O
S
F
5h
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

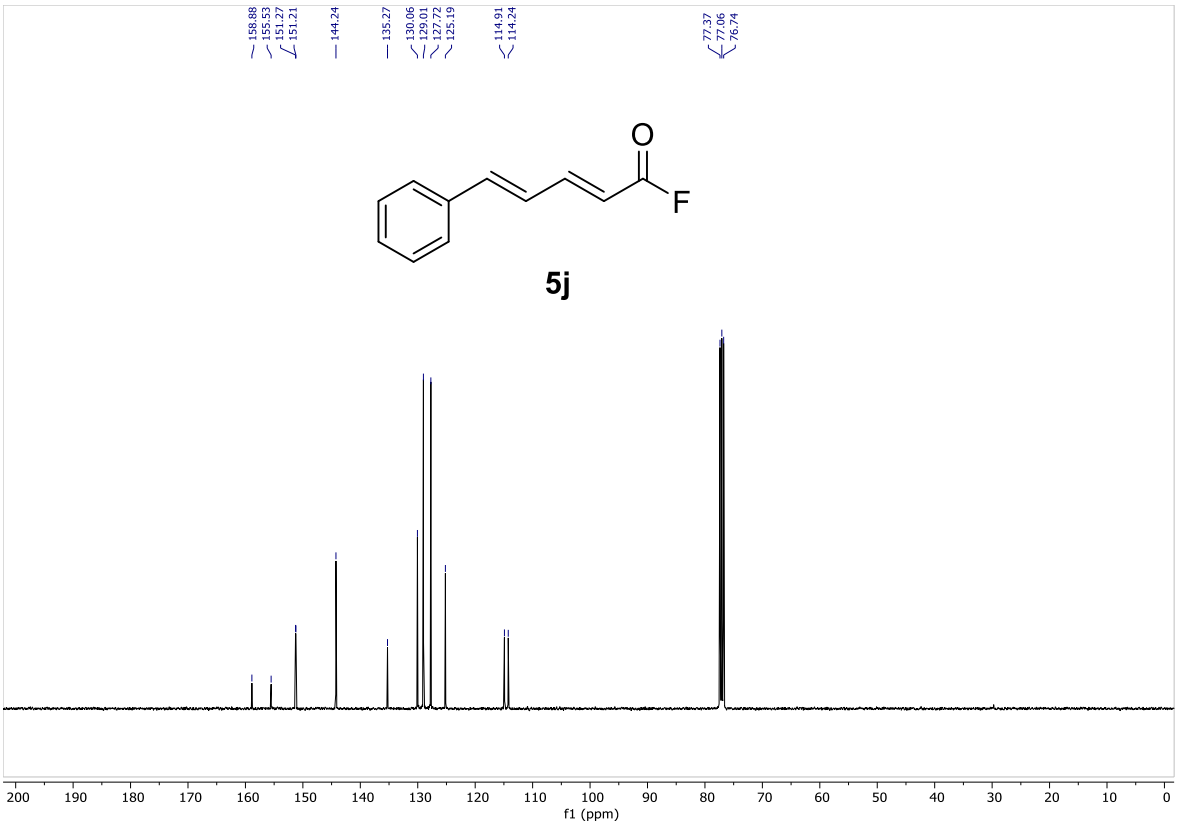

24.30
O
S
F
5h
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

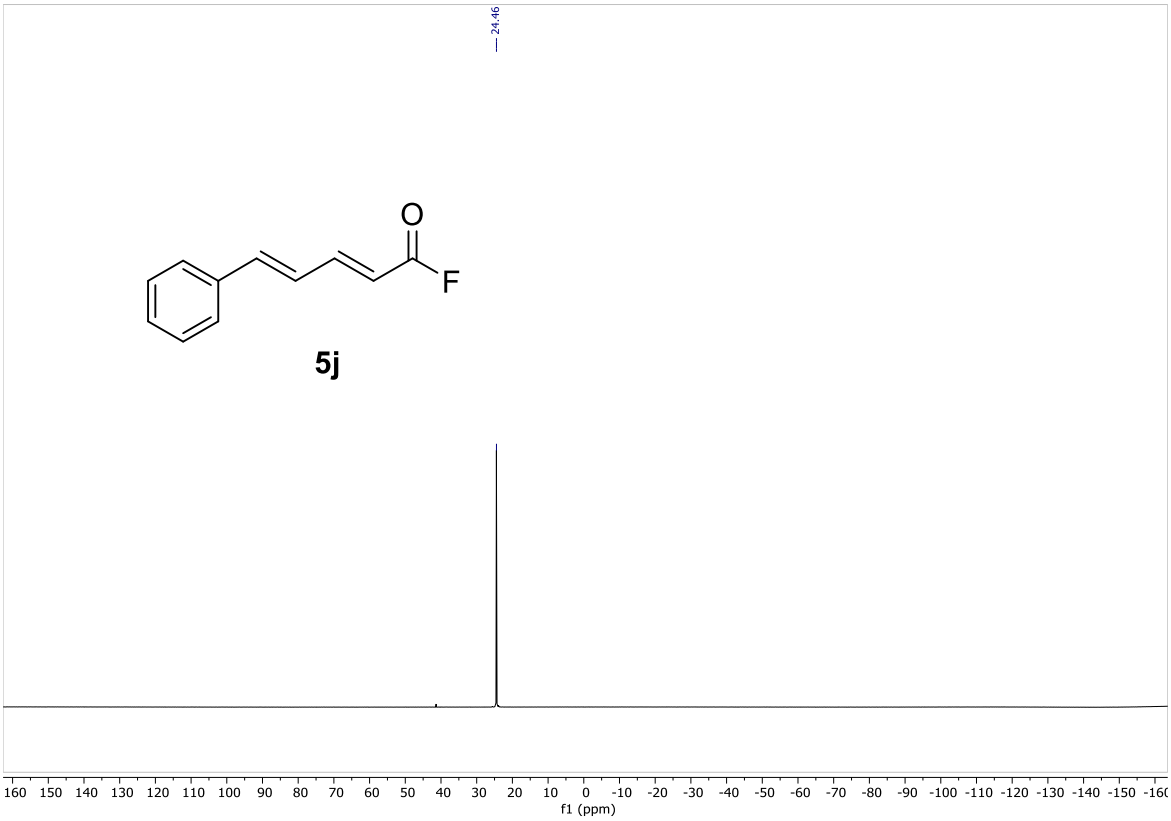

7.86
7.82
7.68
7.67
7.43
7.42
7.41
7.36
7.35
7.28
6.23
6.21
6.19
6.17
O
F
S
5i
1.00
0.98
0.98
1.00
1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

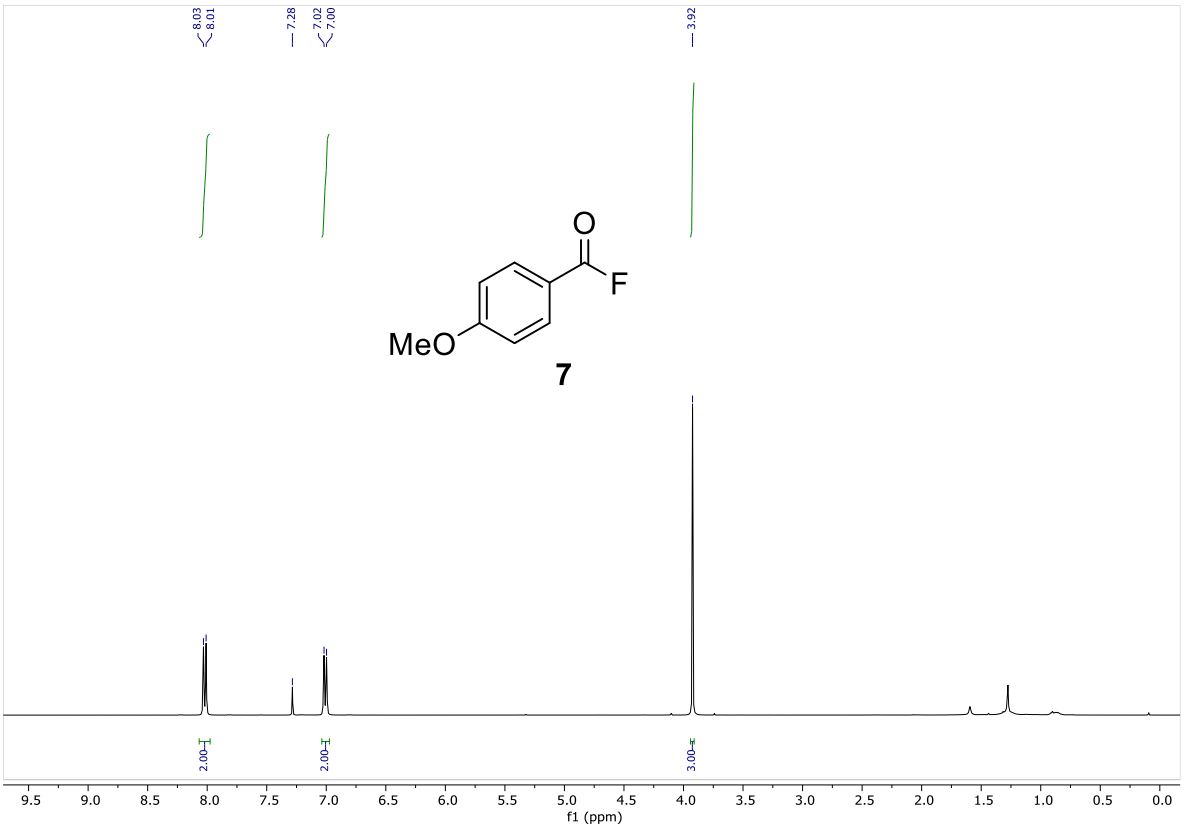

159.13
155.78
144.53
144.46
136.56
131.04
127.77
125.06
111.88
111.21
77.37
77.05
76.73
O
F
S
5i
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

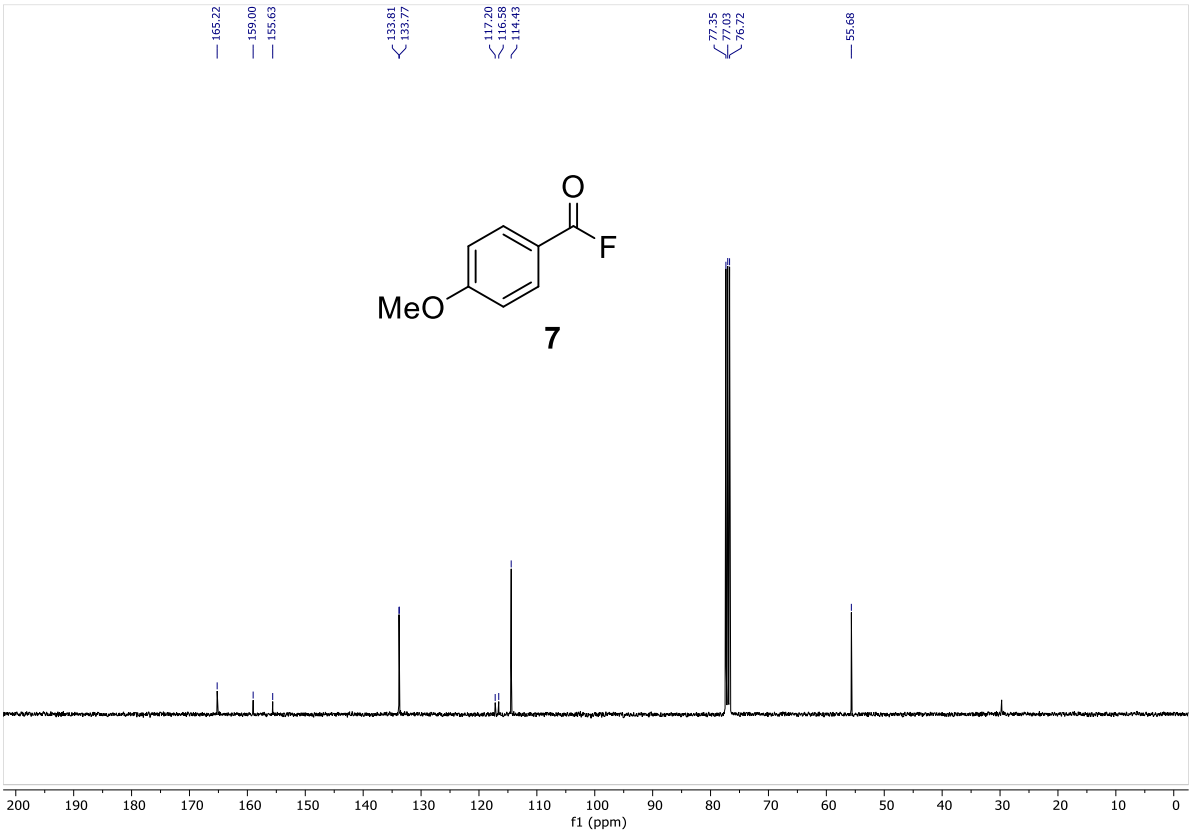

24.70
O
F
S
5i
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

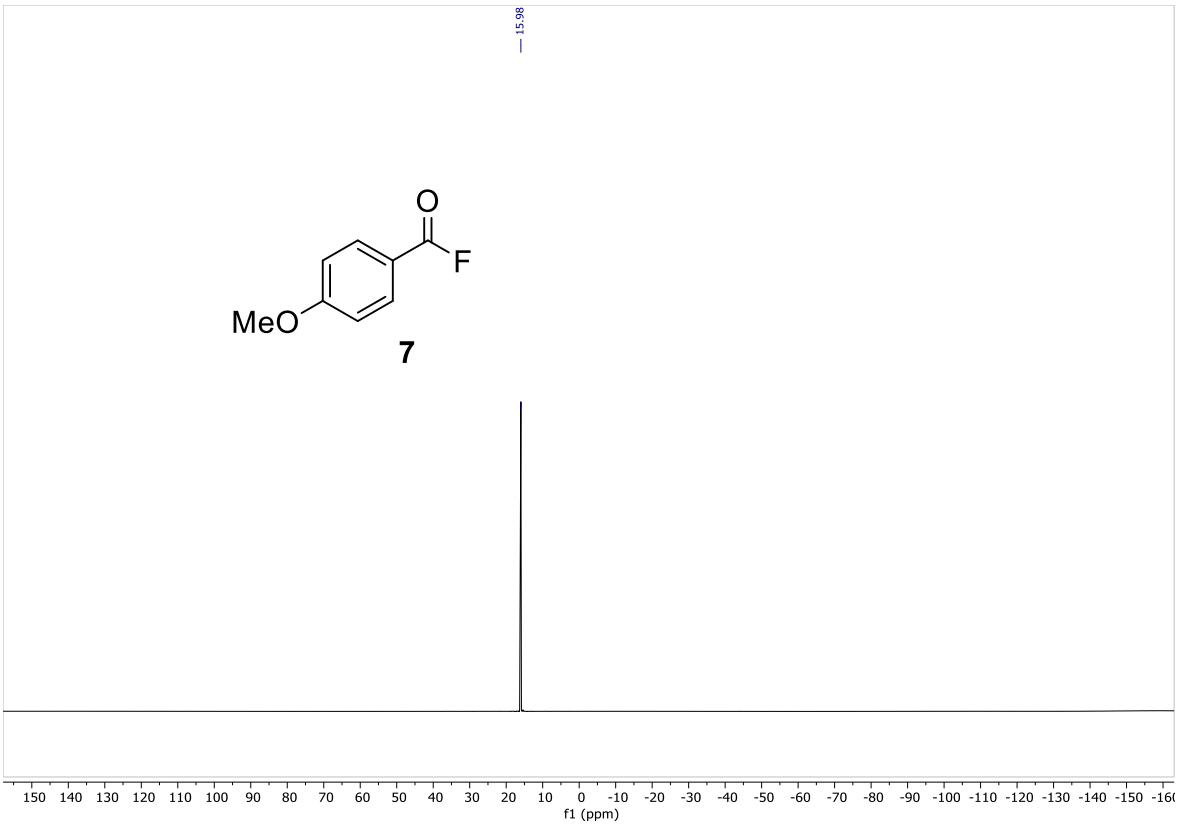

7.65
7.62
7.61
7.59
7.54
7.54
7.52
7.45
7.44
7.42
7.41
7.39
7.38
7.28
7.09
7.05
6.98
6.96
6.94
6.92
5.97
5.95
5.93
5.91
O
F
5j
1.05
2.02
3.04
1.02
1.01
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

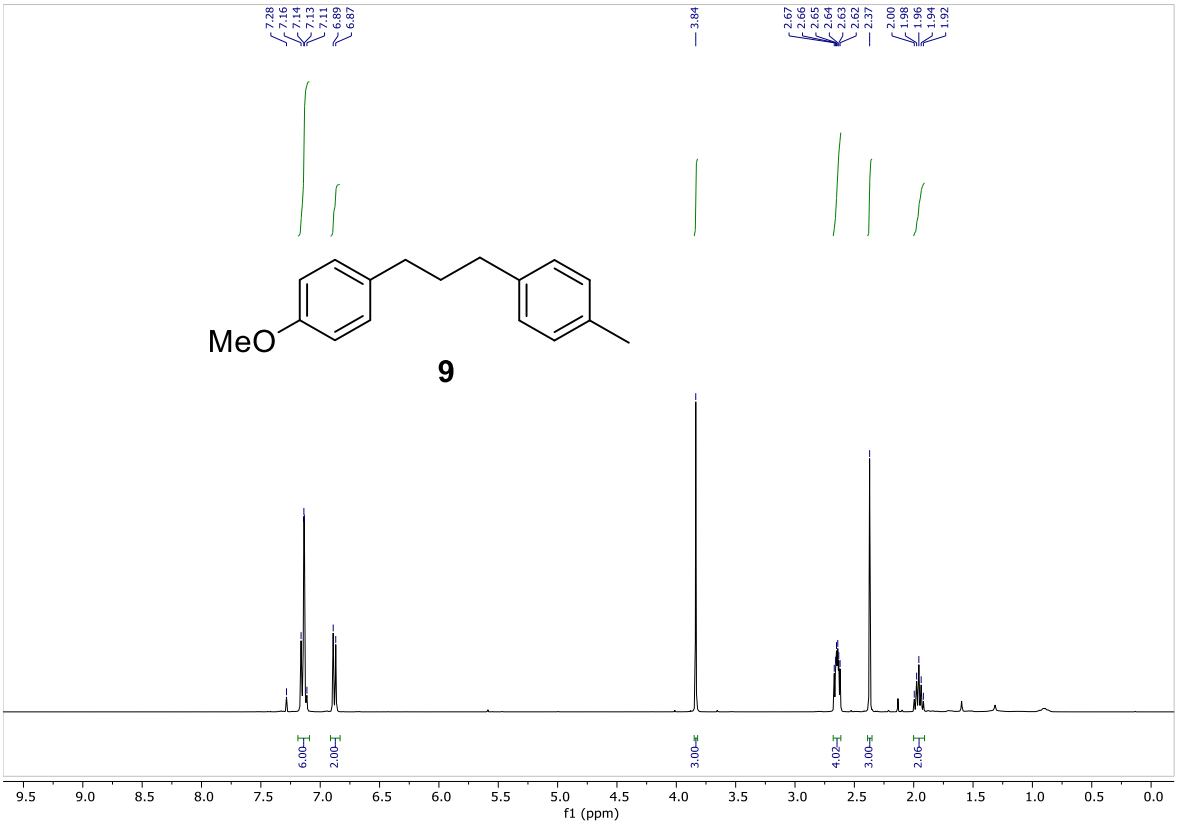

158.88
155.53
151.27
151.21
144.24
135.27
130.06
129.01
127.72
125.19
114.91
114.24
77.37
77.06
76.74
O
F
5j
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

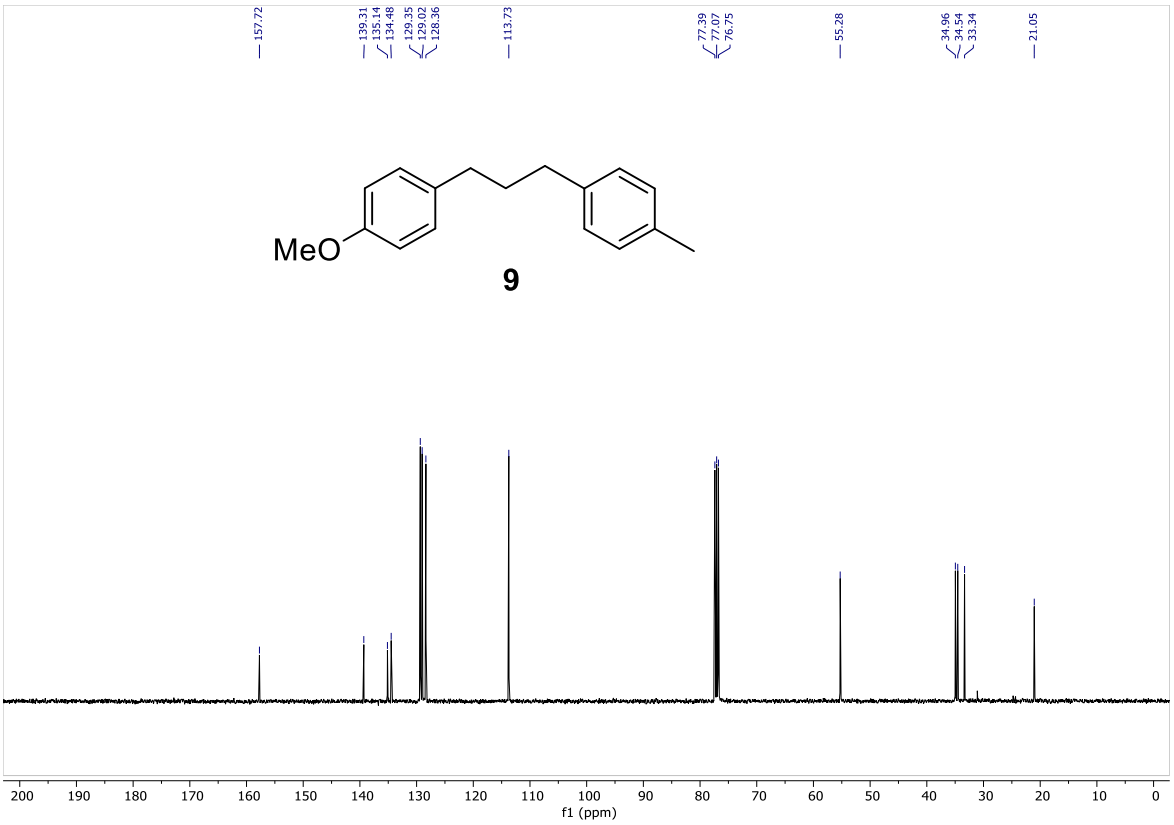

24.46
O
F
5j
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -16
f1 (ppm)

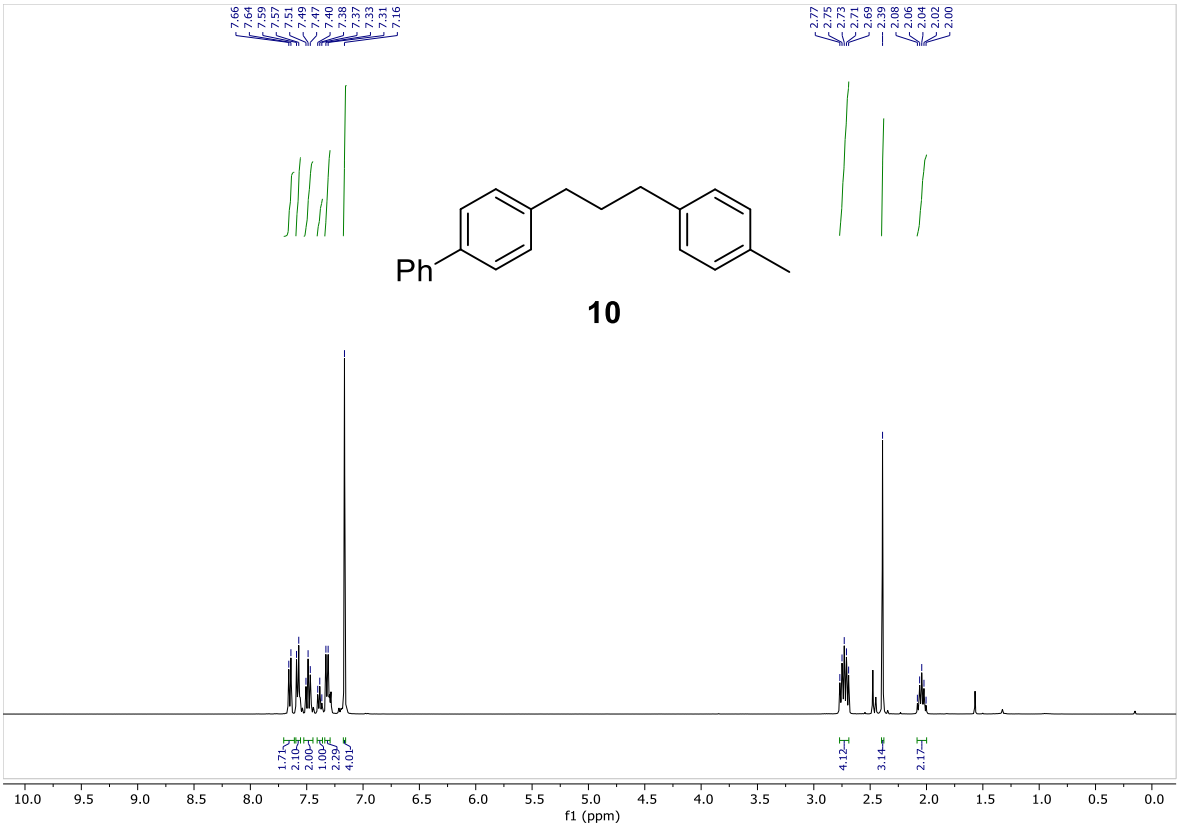

8.03
8.01
7.28
7.02
7.00
3.92
O
F
MeO
7
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

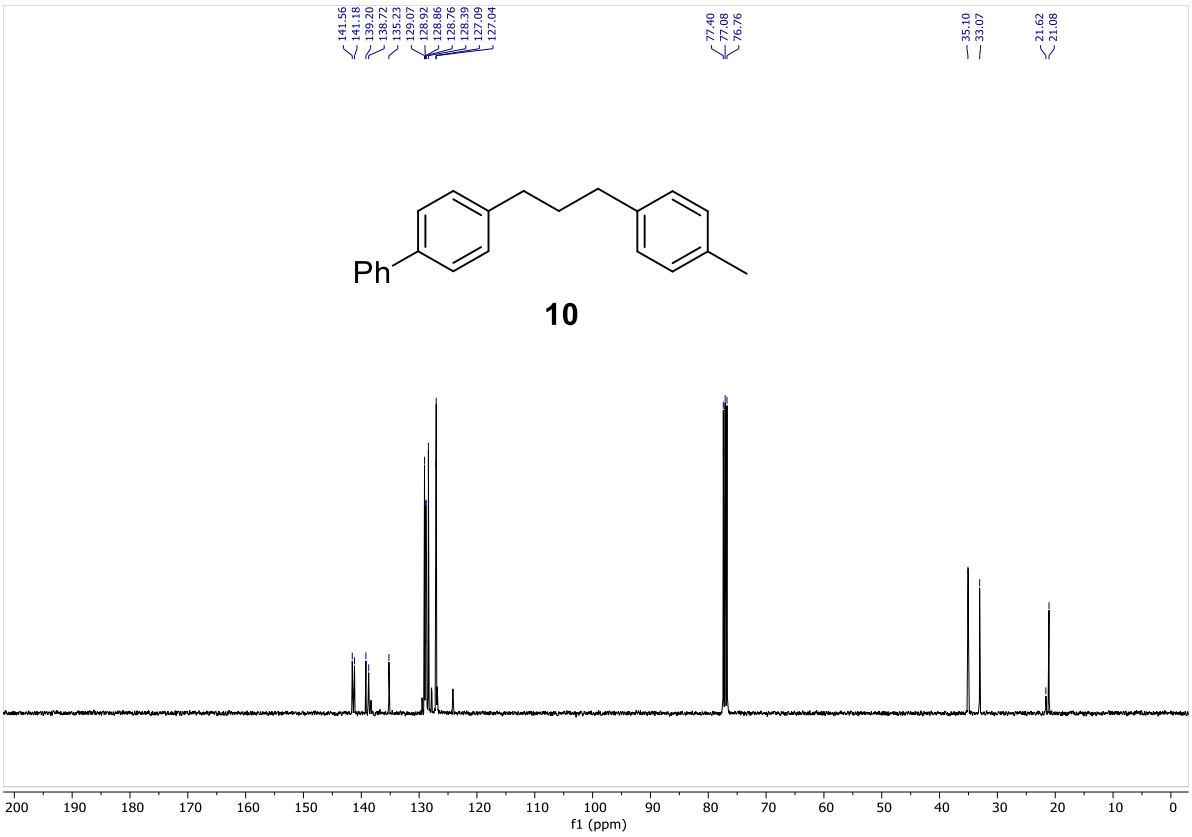

165.22
159.00
155.63
133.81
133.77
117.20
116.58
114.43
77.35
77.03
76.72
55.68
O
F
MeO
7
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


15.98
O
F
MeO
7
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)


7.28
7.16
7.14
7.13
7.11
6.89
6.87
3.84
2.67
2.66
2.65
2.64
2.63
2.62
2.37
2.00
1.98
1.96
1.94
1.92
MeO
9
6.00
2.00
3.00
4.02
3.00
2.06
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)


157.72
139.31
135.14
134.48
129.35
129.02
128.36
113.73
77.39
77.07
76.75
55.28
34.96
34.54
33.34
21.05
MeO
9
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.66
7.64
7.59
7.57
7.51
7.49
7.47
7.40
7.38
7.37
7.33
7.31
7.16
2.77
2.75
2.73
2.71
2.69
2.39
2.08
2.06
2.04
2.02
2.00
Ph
10
1.71
2.10
2.00
1.00
2.29
4.01
4.12
3.14
2.17
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)


141.56
141.18
139.20
138.72
135.23
129.07
128.92
128.86
128.76
128.39
127.09
127.04
77.40
77.08
76.76
35.10
33.07
21.62
21.08
Ph
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)