

Oxidative trifluoromethylation and difluoromethylation of unactivated olefins

Ye-bin Wu ^a, Guo-ping Lu ^a, Tao Yuan ^a, Zhu-bing Xu ^a, Li Wan ^c, and Chun Cai^{*a,b}

^a Chemical Engineering college, Nanjing University of Science and Technology, 200 Xiao Ling Wei Street, Nanjing, Jiangsu, People's Republic of China

^b Key Laboratory of Organofluorine Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 345 Lingling Lu, Shanghai 20032.

^c Biotechnology and Pharmaceutical Engineering College, Nanjing Tech University, Nanjing 211816, People's Republic of China

* Corresponding Author E-mail: c.cai@njust.edu.cn

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1. General information

All chemical reagents are obtained from commercial suppliers and used without further purification. All unknown compounds are characterized by ^1H NMR, ^{13}C NMR, MS and elemental analyses. Analytical thin-layer chromatography is performed on glass plates precoated with silica gel impregnated with a fluorescent indicator (254 nm), and the plates are visualized by exposure to ultraviolet light. Mass spectra are taken on a Thermo Scientific ISQ LT GC-MS instrument in the electron ionization (EI) mode. ^1H NMR, ^{13}C NMR and ^{19}F NMR spectra are recorded on an AVANCE 500 Bruker spectrometer operating at 500 MHz, 125 MHz and 470 MHz in CDCl_3 , respectively, and chemical shifts are reported in ppm. GC analyses are performed on an Agilent 7890A instrument (Column: Agilent 19091J-413:30 m \times 320 μm \times 0.25 μm , H, FID detection). GC-MS data was recorded on a 5975C Mass Selective Detector, coupled with a 7890A Gas Chromatograph (Agilent Technologies). High-resolution mass spectra data were obtained on Agilent mass spectrometer using ESI-TOF (electrospray ionization-time of flight) and Waters Micromass GCT Premier spectrometer (electrospray ionization: EI).

2. General procedure

General procedure for the preparation of olefins from corresponding aldehydes by Wittig reaction: Compounds **1** were prepared according to literature.¹ A benzaldehyde derivative (5 mmol) was added to potassium carbonate (1.1 g, 8 mmol) and methyltriphenylphosphonium bromide (2.1 g, 6 mmol) in anhydrous 1,4-dioxane (5 mL) and heated at reflux for 16 h. The reaction mixture was then cooled, filtered and washed with pentane and then concentrated in vacuo.

Two methods of isolation can be used:

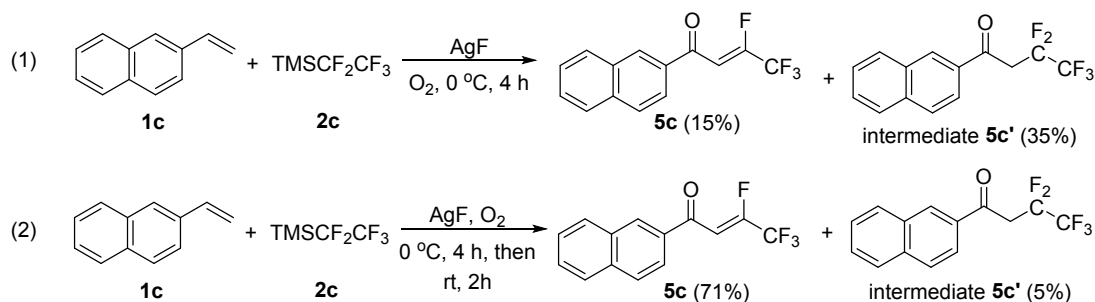
1. The residue was dissolved in hot pentane, cooled to 0 °C, filtered (to remove triphenylphosphine oxide: sparingly soluble in cold pentane) and washed with cold pentane. The filtrate was dried (MgSO_4) and concentrated in vacuo to give the styrene derivative.
2. Isolate via silica gel column chromatography in 40% EtOAc/pentane (ensuring dioxane is removed prior to isolation). All styrene derivatives were then vacuum distilled (may require gentle heating) and degassed before transferring to the glove box.

General procedure for trifluoromethylation of styrenes: A 50 mL oven-dried Schlenk tube with a magnetic stirring bar was equipped with AgF (0.375 mmol), sealed with a septum, and degassed by alternating vacuum evacuation and oxygen backfill (three times). A solution of the olefin (0.25 mmol) and TMSCF_3 (0.25 mmol) in 1 mL anhydrous DMF was added immediately via syringe. Then another 0.25 mmol TMSCF_3 was added by syringe after 1 h. The mixture was stirred at room temperature for 2 h. Upon completion, the reaction mixture was diluted with Et_2O and filtered through a celite pad. Et_2O (20 mL) were added. The organic layer was washed

with water (3 × 5 ml). The combined organic layer was dried over MgSO₄, filtered and concentrated in vacuum. The residue was purified by flash chromatography using petrol ether/ethyl acetate as the eluent. The conditions for chromatography and data for characterization of the products are given below.

General procedure for difluoromethylation of styrenes: A 50 mL oven-dried Schlenk tube with a magnetic stirring bar was equipped with AgF (0.375 mmol), sealed with a septum, and degassed by alternating vacuum evacuation and oxygen backfill (three times). A solution of the olefin (0.25 mmol) and TMSCF₂R (0.25 mmol, R=CF₂COOEt or CF₃) in 1 mL anhydrous DMF was added immediately via syringe. Then another 0.25 mmol TMSCF₂R was added by syringe after 2 h. The mixture was stirred at room temperature for 4 h. Upon completion, the reaction mixture was diluted with ethyl acetate and filtered through a celite pad. Ethyl acetate (20 mL) were added. The organic layer was washed with water (3 × 5 ml). The combined organic layer was dried over MgSO₄, filtered and concentrated in vacuum. The residue was purified by flash chromatography using petrol ether/ethyl acetate as the eluent. The conditions for chromatography and data for characterization of the products are given below.

The procedures of control experiments



Scheme S1 Control experiments

Equation 1: A 50 mL oven-dried Schlenk tube with a magnetic stirring bar was equipped with AgF (0.75 mmol), sealed with a septum, and degassed by alternating vacuum evacuation and oxygen backfill (three times). A solution of 2-Vinylnaphthalene (0.5 mmol) in 2 mL anhydrous DMF was added via syringe. To the resulting suspension, which was precooled to 0 °C was added TMSCF₂CF₃ (0.5 mmol) by syringe. Then another 0.5 mmol TMSCF₂CF₃ was added by syringe after 2 h. The mixture was stirred at 0 °C for 4 h. This procedure was monitored by GC-MS.

Equation 2: A 50 mL oven-dried Schlenk tube with a magnetic stirring bar was equipped with AgF (0.75 mmol), sealed with a septum, and degassed by alternating vacuum evacuation and oxygen backfill (three times). A solution of 2-Vinylnaphthalene (0.5 mmol) in 2 mL anhydrous DMF was added via syringe. To the resulting suspension, which was precooled to 0 °C was added TMSCF₂CF₃ (0.5 mmol) by syringe. Then another 0.5 mmol TMSCF₂CF₃ was added by syringe after 2 h. The mixture was stirred at 0 °C for 4 h. Then the mixture was allowed to warm up to room

temperature and stirring was continued for an additional 2 h. This procedure was monitored by GC-MS.

Oxidative perfluoromethylated intermediate **5c'** was generated in a 35% yield at 0 °C, comparing with a 5% yield when the reaction was warmed up to room temperature. Upon completion, the reaction mixture (equation 1) was diluted with ethyl acetate and filtered through a celite pad. Ethyl acetate (20 mL) were added. The organic layer was washed with water (3 × 10 ml). The combined organic layer was dried over MgSO₄, filtered and concentrated in vacuum. The residue was purified by flash chromatography. We found intermediate **5c'** and product **5c** were hard to separate. The mixture of **5c'** and **5c** was detected by ¹H NMR and ¹⁹F NMR.

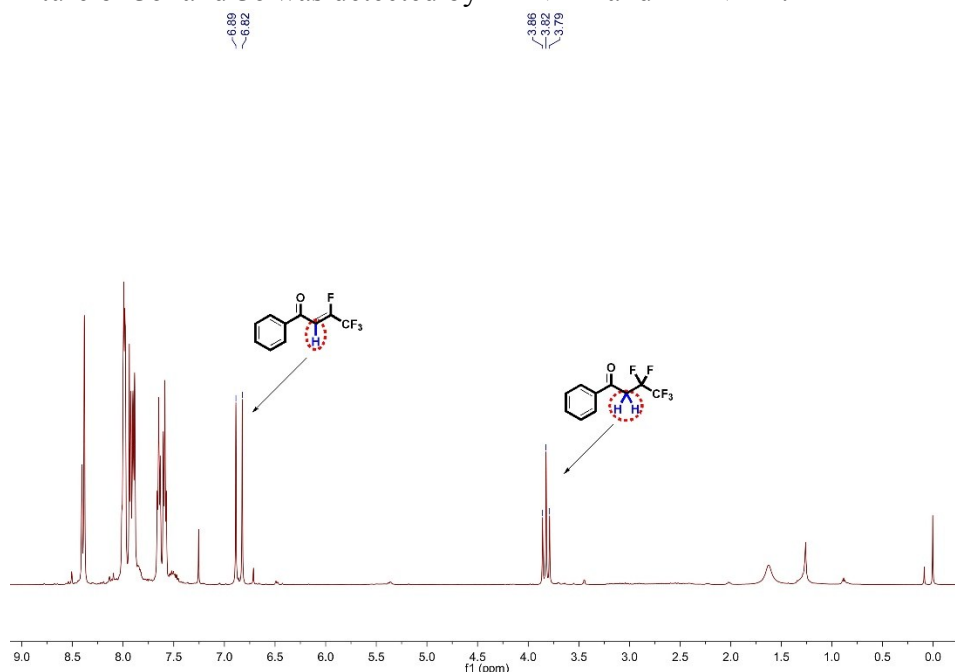


Figure S1 ¹H NMR of the mixture

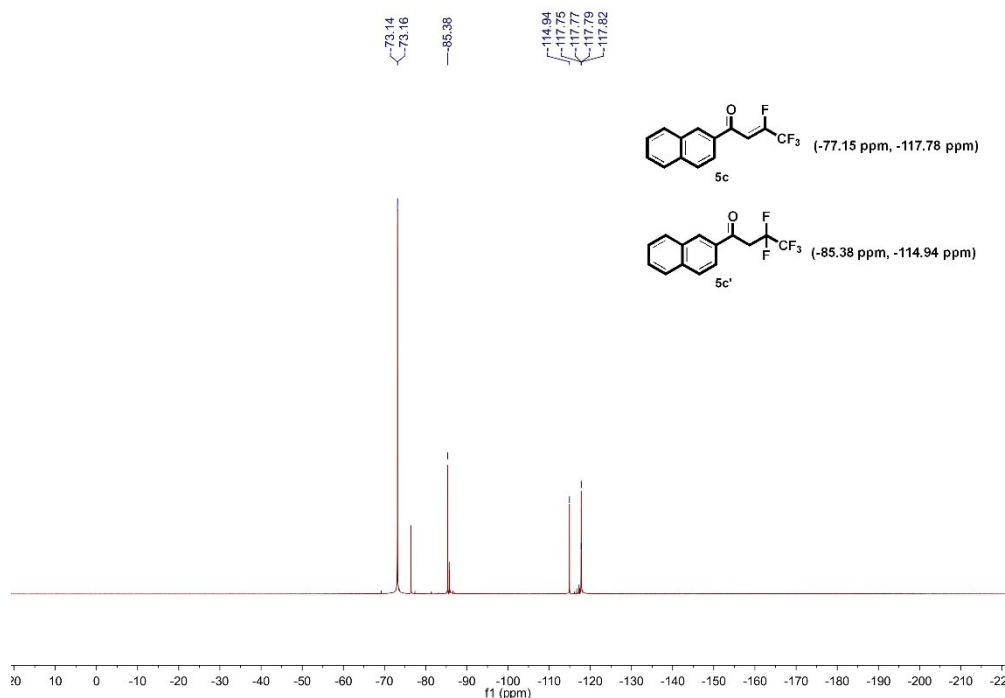
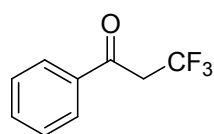


Figure S2 ^{19}F NMR of the mixture

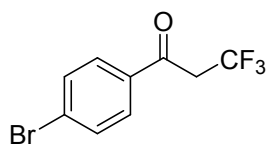
3. Characterization data

3,3,3-trifluoro-1-phenylpropan-1-one (3a)²



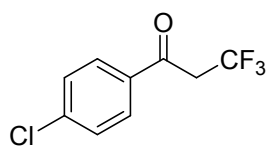
The title compound was isolated as a white solid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 89%. ^1H NMR (500 MHz, CDCl_3) δ 7.95 (d, $J = 7.8$ Hz, 2H), 7.65 (t, $J = 7.4$ Hz, 1H), 7.52 (t, $J = 7.7$ Hz, 2H), 3.81 (q, $J = 10.0$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 189.87 (s), 135.94 (s), 134.34 (s), 129.08 (s), 128.48 (s), 124.15 (q, $J = 277.8$ Hz), 42.22 (q, $J = 27.7$ Hz). ^{19}F NMR (470 MHz, CDCl_3) δ -62.17 (s).

1-(4-bromophenyl)-3,3,3-trifluoropropan-1-one (3b)²



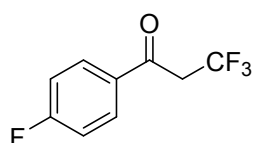
The title compound was isolated as a slightly yellow solid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 83%. ^1H NMR (500 MHz, CDCl_3) δ 7.79 (d, $J = 8.6$ Hz, 2H), 7.65 (d, $J = 8.6$ Hz, 2H), 3.77 (q, $J = 9.9$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 188.94 (s), 134.62 (s), 132.44 (s), 129.93 (s), 129.78 (s), 123.96 (q, $J = 277.2$ Hz), 42.22 (q, $J = 28.1$ Hz). ^{19}F NMR (470 MHz, CDCl_3) δ -61.98 (s).

1-(4-chlorophenyl)-3,3,3-trifluoropropan-1-one (3c)²



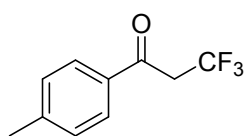
The title compound was isolated as a white solid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 77%. ^1H NMR (500 MHz, CDCl_3) δ 7.88 (d, $J = 8.6$ Hz, 2H), 7.49 (d, $J = 8.6$ Hz, 2H), 3.78 (q, $J = 9.9$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 188.70 (s), 141.03 (s), 134.24 (s), 129.89 (s), 129.45 (s), 123.96 (q, $J = 278.0$ Hz), 42.27 (q, $J = 28.5$ Hz). ^{19}F NMR (470 MHz, CDCl_3) δ -61.96 (s).

3,3,3-trifluoro-1-(4-fluorophenyl)propan-1-one (3d) ²



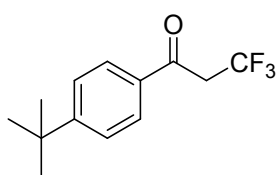
The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 85%. ¹H NMR (500 MHz, CDCl₃) δ 7.99 – 7.96 (m, 2H), 7.19 (t, *J* = 8.5 Hz, 2H), 3.78 (q, *J* = 9.9 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 188.28 (s), 166.49 (d, *J* = 258.3 Hz), 132.41 (s), 131.29 (d, *J* = 9.1 Hz), 124.00 (q, *J* = 277.2 Hz), 116.31 (d, *J* = 21.4 Hz), 42.24 (q, *J* = 28.5 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -61.96 (s), -102.81 (s).

3,3,3-trifluoro-1-(p-tolyl)propan-1-one (3e) ²



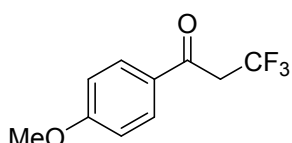
The title compound was isolated as a white solid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 87%. ¹H NMR (500 MHz, CDCl₃) δ 7.84 (d, *J* = 8.1 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.77 (q, *J* = 10.1 Hz, 2H), 2.44 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 189.44 (s), 145.42 (s), 133.58 (s), 129.73 (s), 128.64 (s), 124.19 (q, *J* = 276.2 Hz), 42.13 (q, *J* = 27.5 Hz), 21.82 (s). ¹⁹F NMR (470 MHz, CDCl₃) δ -61.98 (s).

1-(4-(tert-butyl)phenyl)-3,3,3-trifluoropropan-1-one (3f) ³



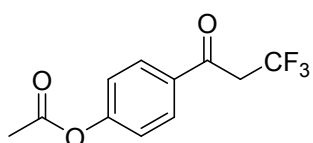
The title compound was isolated as a slightly yellow liquid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 86%. ¹H NMR (500 MHz, CDCl₃) δ 7.89 (d, *J* = 8.5 Hz, 2H), 7.53 (d, *J* = 8.5 Hz, 2H), 3.78 (q, *J* = 10.1 Hz, 2H), 1.36 (s, 9H). ¹³C NMR (126 MHz, CDCl₃) δ 189.43 (s), 158.33 (s), 133.46 (s), 128.52 (s), 126.02 (s), 124.22 (q, *J* = 275.0 Hz), 42.12 (q, *J* = 27.5 Hz), 35.37 (s), 31.11 (s). ¹⁹F NMR (470 MHz, CDCl₃) δ -61.95 (s).

3,3,3-trifluoro-1-(4-methoxyphenyl)propan-1-one (3g) ²



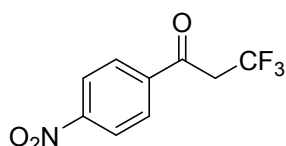
The title compound was isolated as a yellow solid after chromatography on silica with a Combiflash system (100:0-96:4 petrol ether/ethyl acetate). Yield: 75%. ¹H NMR (500 MHz, CDCl₃) δ 7.91 (d, *J* = 8.4 Hz, 2H), 6.97 (d, *J* = 8.4 Hz, 2H), 3.89 (s, 3H), 3.74 (q, *J* = 10.1 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 188.28 (s), 164.49 (s), 140.67 (s), 130.94 (s), 124.26 (q, *J* = 275.6 Hz), 114.23 (s), 55.69 (s), 41.93 (q, *J* = 27.5 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -61.94 (s).

4-(3,3,3-trifluoropropanoyl)phenyl acetate (3h) ⁴



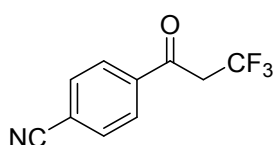
The title compound was isolated as a white solid after chromatography on silica with a Combiflash system (100:0-93:7 petrol ether/ethyl acetate). Yield: 90%. ¹H NMR (500 MHz, CDCl₃) δ 7.97 (d, *J* = 8.7 Hz, 2H), 7.24 (d, *J* = 8.8 Hz, 2H), 3.78 (q, *J* = 10.0 Hz, 2H), 2.33 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 188.64 (s), 168.81 (s), 155.31 (s), 133.44 (s), 130.18 (s), 124.00 (q, *J* = 277.6 Hz), 122.31 (s), 42.21 (q, *J* = 28.5 Hz), 21.23 (s). ¹⁹F NMR (470 MHz, CDCl₃) δ -61.97 (s).

3,3,3-trifluoro-1-(4-nitrophenyl)propan-1-one (3i) ²



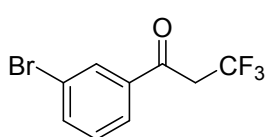
The title compound was isolated as a slightly yellow solid after chromatography on silica with a Combiflash system (100:0-95:5 petrol ether/ethyl acetate). Yield: 70%. ¹H NMR (500 MHz, CDCl₃) δ 8.37 (d, *J* = 8.8 Hz, 2H), 8.12 (d, *J* = 8.8 Hz, 2H), 3.87 (q, *J* = 9.7 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 188.49 (s), 151.07 (s), 140.08 (s), 129.61 (s), 124.31 (s), 123.65 (q, *J* = 277.6 Hz), 42.85 (q, *J* = 29.0 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -61.92 (s).

4-(3,3,3-trifluoropropanoyl)benzotrile (3j) ²



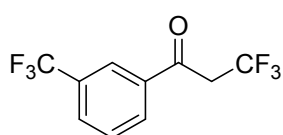
The title compound was isolated as a slightly yellow liquid after chromatography on silica with a Combiflash system (100:0-96:4 petrol ether/ethyl acetate). Yield: 79%. ¹H NMR (500 MHz, CDCl₃) δ 8.04 (d, *J* = 8.4 Hz, 2H), 7.84 (d, *J* = 8.5 Hz, 2H), 3.83 (q, *J* = 9.7 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 188.66 (s), 138.66 (s), 132.94 (s), 128.91 (s), 123.70 (q, *J* = 277.5 Hz), 117.67 (s), 42.61 (q, *J* = 29.0 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -61.92 (s).

1-(3-bromophenyl)-3,3,3-trifluoropropan-1-one (3k) ⁵



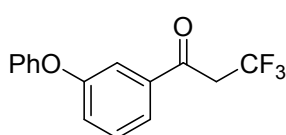
The title compound was isolated as a yellow liquid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 79%. ¹H NMR (500 MHz, CDCl₃) δ 8.07 (s, 1H), 7.86 (d, *J* = 7.8 Hz, 1H), 7.77 (d, *J* = 7.9 Hz, 1H), 7.41 (t, *J* = 7.9 Hz, 1H), 3.78 (q, *J* = 9.8 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 188.56 (s), 137.58 (s), 137.19 (s), 131.50 (s), 130.63 (s), 126.99 (s), 123.88 (q, *J* = 276.3 Hz), 123.45 (s), 42.34 (q, *J* = 28.8 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -62.00 (s).

3,3,3-trifluoro-1-(3-(trifluoromethyl)phenyl)propan-1-one (3l) ⁶



The title compound was isolated as a slightly yellow liquid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 86%. ¹H NMR (500 MHz, CDCl₃) δ 8.19 (s, 1H), 8.13 (d, *J* = 7.9 Hz, 1H), 7.91 (d, *J* = 7.8 Hz, 1H), 7.69 (t, *J* = 7.8 Hz, 1H), 3.84 (d, *J* = 9.8 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 188.60 (s), 136.41 (s), 132.02 (s), 131.75 (s), 131.58 (s), 130.72 (s), 130.72 (s), 125.32 (s), 123.82 (q, *J* = 277.6 Hz), 123.5 (q, *J* = 273.0 Hz), 42.45 (q, *J* = 28.6 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -61.97 (s), -62.93 (s).

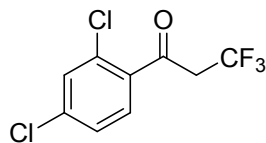
3,3,3-trifluoro-2-methyl-1-(3-phenoxyphenyl)propan-1-one (3m) ²



The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-96:4 petrol ether/ethyl acetate). Yield: 84%. ¹H NMR (500 MHz, CDCl₃) δ 7.65 – 7.63 (m, 1H), 7.56 (t, *J* = 5.0 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.41 – 7.37 (m, 2H), 7.29 – 7.26 (m, 1H), 7.20 – 7.16 (m, 1H), 7.05 – 7.03 (m, 2H), 3.76 (q, *J* = 9.9 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 191.74 (s), 189.24 (s), 158.34 (s), 156.33 (s), 137.61 (s), 130.45 (s), 130.20 (s), 124.32 (s), 124.03 (q, *J* = 278.0 Hz), 123.01 (s), 120.71 (s), 119.55 (d, *J* = 16.5 Hz), 117.94 (s), 42.38 (q, *J* = 28.6 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -62.02 (s). HRMS (EI) Calcd. For

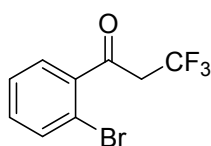
280.0711, C₁₅H₁₁F₃O, found 280.0714.

1-(2,4-dichlorophenyl)-3,3,3-trifluoropropan-1-one (3n)⁷



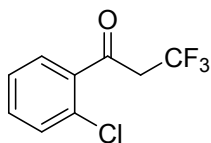
The title compound was isolated as a slightly yellow liquid after chromatography on silica with a Combiflash system (100:0-97:3 petrol ether/ethyl acetate). Yield: 82%. ¹H NMR (500 MHz, CDCl₃) δ 7.54 (d, *J* = 8.4 Hz, 1H), 7.49 (d, *J* = 1.9 Hz, 1H), 7.38 (dd, *J* = 8.4, 1.9 Hz, 1H), 3.86 (q, *J* = 9.9 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 191.17 (s), 139.06 (s), 135.88 (s), 132.49 (s), 131.08 (s), 130.85 (s), 127.94 (s), 123.56 (q, *J* = 278.0 Hz), 46.27 (q, *J* = 28.6 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -62.13 (s).

1-(2-bromophenyl)-3,3,3-trifluoropropan-1-one (3o)²



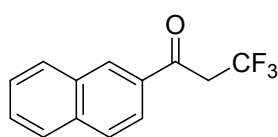
The title compound was isolated as a slightly yellow liquid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 81%. ¹H NMR (500 MHz, CDCl₃) δ 7.68 – 7.64 (m, 1H), 7.47 – 7.41 (m, 2H), 7.39 – 7.36 (m, 1H), 3.85 (q, *J* = 10.0 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 193.40 (s), 140.14 (s), 134.06 (s), 132.82 (s), 129.30 (s), 127.90 (s), 123.58 (q, *J* = 277.2 Hz), 45.98 (q, *J* = 28.1 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -62.09 (s).

1-(2-chlorophenyl)-3,3,3-trifluoropropan-1-one (3p)²



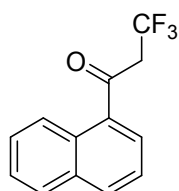
The title compound was isolated as a yellow liquid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 78%. ¹H NMR (500 MHz, CDCl₃) δ 7.59 – 7.53 (m, 1H), 7.48 – 7.44 (m, 2H), 7.40 – 7.37 (m, 1H), 3.87 (q, *J* = 10.0 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 192.50 (s), 137.79 (s), 133.10 (s), 131.33 (s), 130.90 (s), 129.83 (s), 127.45 (s), 123.68 (q, *J* = 278.5 Hz), 46.26 (q, *J* = 27.7 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -62.17 (s).

3,3,3-trifluoro-1-(naphthalen-2-yl)propan-1-one (3q)²



The title compound was isolated as a white solid after chromatography on silica with a Combiflash system (100:0-97:3 petrol ether/ethyl acetate). Yield: 85%. ¹H NMR (500 MHz, CDCl₃) δ 8.41 (s, 1H), 8.03 – 7.96 (m, 2H), 7.93 – 7.89 (m, 2H), 7.68 – 7.63 (m, 1H), 7.62 – 7.57 (m, 1H), 3.94 (q, *J* = 10.0 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 189.78 (s), 136.11 (s), 133.35 (s), 132.48 (s), 130.68 (s), 129.84 (s), 129.34 (s), 129.06 (s), 128.00 (s), 127.34 (s), 125.36 (s), 124.26 (q, *J* = 277.2 Hz), 123.59 (s), 42.28 (q, *J* = 28.5 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -61.86 (s).

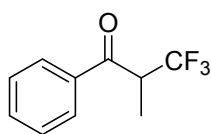
3,3,3-trifluoro-1-(naphthalen-1-yl)propan-1-one (3r)²



The title compound was isolated as a slightly yellow liquid after chromatography on silica with a Combiflash system (100:0-96:4 petrol ether/ethyl acetate). Yield: 63%. ¹H NMR (500 MHz, CDCl₃) δ 8.73 (d, *J* = 8.6 Hz, 1H), 8.07 (d, *J* = 8.2 Hz, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.88 (d, *J* = 7.2 Hz, 1H), 7.66 (t, *J* = 7.5 Hz, 1H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.54 (t, *J* = 7.7 Hz, 1H), 3.92 (q, *J* = 10.0 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 192.99 (s), 134.4 (s), 134.17 (s), 133.97 (s), 130.33 (s),

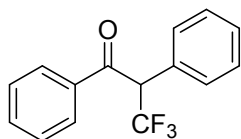
129.21 (s), 128.88 (s), 128.72 (s), 127.06 (s), 125.73 (s), 124.30 (s), 124.13 (q, $J = 277.2$ Hz), 45.10 (q, $J = 28.1$ Hz). ^{19}F NMR (470 MHz, CDCl_3) δ -61.90 (s).

3,3,3-trifluoro-2-methyl-1-phenylpropan-1-one (3s)²



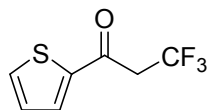
The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 74%. ^1H NMR (500 MHz, CDCl_3) δ 7.97 (d, $J = 8.1$ Hz, 2H), 7.64 (dd, $J = 10.7, 4.1$ Hz, 1H), 7.52 (t, $J = 7.8$ Hz, 2H), 4.32 – 4.20 (m, 1H), 1.49 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 194.53 (s), 135.83 (s), 134.11 (s), 129.04 (s), 128.73 (s), 125.43 (q, $J = 281.0$ Hz), 44.44 (q, $J = 26.5$ Hz), 11.81 (s). ^{19}F NMR (470 MHz, CDCl_3) δ -68.24 (s).

3,3,3-trifluoro-1,2-diphenylpropan-1-one (3t)²



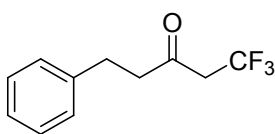
The title compound was isolated as a slightly yellow liquid after chromatography on silica with a Combiflash system (100:0-98:2 petrol ether/ethyl acetate). Yield: 60%. ^1H NMR (500 MHz, CDCl_3) δ 7.91 (dd, $J = 8.4, 1.1$ Hz, 2H), 7.55-7.52 (m, 1H), 7.50 – 7.44 (m, 2H), 7.45 – 7.34 (m, 5H), 5.29 (q, $J = 8.2$ Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 191.23 (s), 135.55 (s), 133.90 (s), 129.97 (s), 129.82 (s), 129.41 (s), 129.32 (s), 129.13 (s), 128.92 (s), 124.37 (q, $J = 275.9$ Hz), 56.70 (s, $J = 26.5$ Hz). ^{19}F NMR (470 MHz, CDCl_3) δ -66.50 (s).

3,3,3-trifluoro-1-(thiophen-2-yl)propan-1-one (3u)⁵



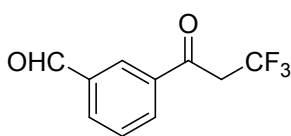
The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 89%. ^1H NMR (500 MHz, CDCl_3) δ 7.76 (dd, $J = 4.9, 1.0$ Hz, 1H), 7.74 (dd, $J = 3.8, 0.8$ Hz, 1H), 7.19 (dd, $J = 4.9, 3.9$ Hz, 1H), 3.72 (q, $J = 10.1$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 182.27 (s), 143.34 (s), 135.82 (s), 133.52 (s), 128.62 (s), 123.77 (q, $J = 278.0$ Hz), 43.21 (q, $J = 29.0$ Hz). ^{19}F NMR (470 MHz, CDCl_3) δ -61.90 (s).

1,1,1-trifluoro-5-phenylpentan-3-one (3v)³



The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-99:1 petrol ether/ethyl acetate). Yield: 71%. ^1H NMR (500 MHz, CDCl_3) δ 7.31 (t, $J = 7.5$ Hz, 2H), 7.23 (t, $J = 7.5$ Hz, 1H), 7.19 (t, $J = 7.3$ Hz, 2H), 3.20 (q, $J = 10.4$ Hz, 2H), 2.95 (t, $J = 7.2$ Hz, 2H), 2.87 (t, $J = 7.4$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 199.31 (s), 140.26 (s), 128.76 (s), 128.42 (s), 126.53 (s), 123.68 (q, $J = 277.6$ Hz), 46.65 (q, $J = 28.6$ Hz), 45.09 (s), 29.34 (s). ^{19}F NMR (470 MHz, CDCl_3) δ -62.32 (s).

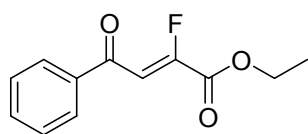
3-(3,3,3-trifluoropropanoyl)benzaldehyde(3w)²



The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-97:3 petrol ether/ethyl acetate). Yield: 84%. ^1H NMR (500 MHz, CDCl_3) δ 10.11 (s, 1H), 8.41 (s, 1H), 8.22 (d, $J = 7.8$ Hz, 1H), 8.16 (d, $J = 7.6$ Hz, 1H), 7.73 (t, $J = 7.7$ Hz, 1H), 3.87 (t, $J = 9.8$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 191.10 (s), 189.00 (s), 136.98 (s), 136.65 (s), 134.87 (s), 133.79 (s), 130.06 (s), 129.31 (s), 123.89 (q, $J = 278.04$ Hz).

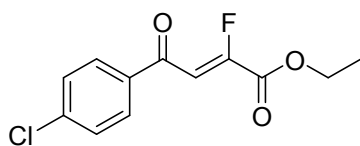
H_z), 42.45 (q, *J* = 28.98 Hz). ¹⁹F NMR (470 MHz, CDCl₃) δ -61.96 (s).

ethyl (Z)-2-fluoro-4-oxo-4-phenylbut-2-enoate (4a) ⁸



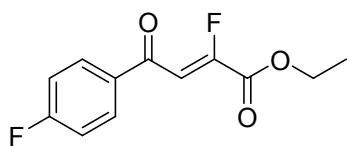
The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-95:5 petrol ether/ethyl acetate). Yield: 85%. ¹H NMR (500 MHz, CDCl₃) δ 7.95 (d, *J* = 7.6 Hz, 2H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 2H), 7.13 (d, *J* = 30.8 Hz, 1H), 4.40 (q, *J* = 7.1 Hz, 2H), 1.40 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 188.33 (s), 160.29 (d, *J* = 34.0 Hz), 152.24 (d, *J* = 286.0 Hz), 136.82 (s), 134.17 (s), 128.95 (s), 128.92 (s), 112.22 (s), 63.00 (s), 14.20 (s). ¹⁹F NMR (470 MHz, CDCl₃) δ -110.39 (s).

ethyl (Z)-4-(4-chlorophenyl)-2-fluoro-4-oxobut-2-enoate (4b)



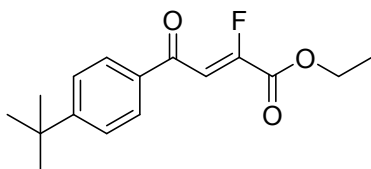
The title compound was isolated as a slightly yellow liquid after chromatography on silica with a Combiflash system (100:0-95:5 petrol ether/ethyl acetate). Yield: 60%. ¹H NMR (500 MHz, CDCl₃) δ 7.88 (d, *J* = 8.6 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.07 (d, *J* = 30.6 Hz, 1H), 4.40 (q, *J* = 7.1 Hz, 2H), 1.40 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 187.10 (s), 160.13 (d, *J* = 34.0 Hz), 152.49 (d, *J* = 287.3 Hz), 140.76 (s), 135.17 (s), 130.28 (s), 129.35 (s), 111.77 (s), 63.08 (s), 14.19 (s). ¹⁹F NMR (470 MHz, CDCl₃) δ -109.34 (s). HRMS (EI) Calcd. For 256.0303, C₁₂H₁₀ClFO₃, found 256.0302.

ethyl (Z)-2-fluoro-4-(4-fluorophenyl)-4-oxobut-2-enoate (4c)



The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-95:5 petrol ether/ethyl acetate). Yield: 75%. ¹H NMR (500 MHz, CDCl₃) δ 7.98 (dd, *J* = 8.7, 5.4 Hz, 2H), 7.18 (t, *J* = 8.5 Hz, 2H), 7.08 (d, *J* = 30.7 Hz, 1H), 4.40 (q, *J* = 7.1 Hz, 2H), 1.40 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 186.79 (s), 166.44 (d, *J* = 257.0 Hz), 160.18 (d, *J* = 34.8 Hz), 152.28 (d, *J* = 286.0 Hz), 133.26 (s), 131.68 (d, *J* = 8.9 Hz), 116.23 (d, *J* = 22.7 Hz), 112.01 (s), 63.05 (s), 14.18 (s). ¹⁹F NMR (470 MHz, CDCl₃) δ -103.11 (s), -109.97 (s). HRMS (EI) Calcd. For 240.0598, C₁₂H₁₀F₂O₃, found 240.0593.

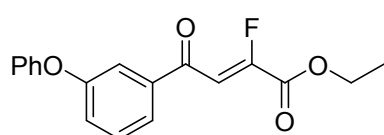
ethyl (Z)-4-(4-(tert-butyl)phenyl)-2-fluoro-4-oxobut-2-enoate (4d)



The title compound was isolated as a slightly yellow liquid after chromatography on silica with a Combiflash system (100:0-95:5 petrol ether/ethyl acetate). Yield: 78%. ¹H NMR (500 MHz, CDCl₃) δ 7.89 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.6 Hz, 2H), 7.12 (d, *J* = 31.0 Hz, 1H), 4.40 (q, *J* = 7.1 Hz, 2H), 1.40 (t, *J* = 7.1 Hz, 3H), 1.36 (s, 9H). ¹³C NMR (126 MHz, CDCl₃) δ 187.92 (s), 160.39 (d, *J* = 35.3 Hz), 158.18 (s), 151.95 (d, *J* = 284.8 Hz), 134.28 (s), 128.94 (s), 125.96 (s), 112.46 (s), 62.92 (s), 35.40 (s), 31.15 (s), 14.20 (s). ¹⁹F NMR (470 MHz, CDCl₃) δ -111.18 (s). HRMS (ESI) Calcd. For 279.1396, C₁₆H₁₉FO₃ [M-H]⁺, found 279.1407.

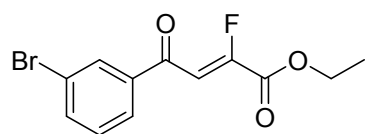
ethyl (Z)-2-fluoro-4-oxo-4-(3-phenoxyphenyl)but-2-enoate (4e)

The title compound was isolated as a colorless liquid after chromatography on silica with a



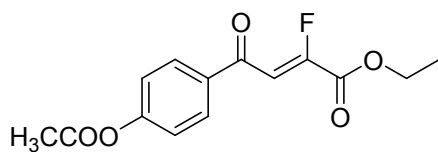
Combiflash system (100:0-92:8 petrol ether/ethyl acetate). Yield: 79%. ^1H NMR (500 MHz, CDCl_3) δ 7.66 (d, $J = 7.6$ Hz, 1H), 7.58 (s, 1H), 7.46 (t, $J = 7.9$ Hz, 1H), 7.38 (t, $J = 7.7$ Hz, 2H), 7.27 – 7.23 (m, 1H), 7.17 (t, $J = 7.4$ Hz, 1H), 7.09 (d, $J = 30.6$ Hz, 1H), 7.04 (d, $J = 8.4$ Hz, 2H), 4.39 (q, $J = 6.8$ Hz, 2H), 1.39 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 187.64 (s), 160.19 (d, $J = 34.7$ Hz), 158.20 (s), 156.46 (s), 152.44 (d, $J = 286.0$ Hz), 138.55 (s), 130.37 (s), 130.15 (s), 124.21 (s), 123.59 (s), 119.41 (s), 118.32 (s), 111.97 (s), 63.03 (s), 14.19 (s). ^{19}F NMR (470 MHz, CDCl_3) δ -109.74 (s). HRMS (ESI) Calcd. For 315.1033, $\text{C}_{18}\text{H}_{15}\text{FO}_4$ $[\text{M}-\text{H}]^+$, found 315.1022.

ethyl (Z)-4-(3-bromophenyl)-2-fluoro-4-oxobut-2-enoate (4f)



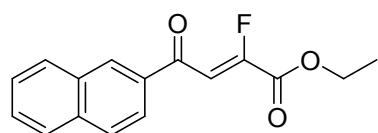
The title compound was isolated as a yellow liquid after chromatography on silica with a Combiflash system (100:0-95:5 petrol ether/ethyl acetate). Yield: 65%. ^1H NMR (500 MHz, CDCl_3) δ 8.07 (s, 1H), 7.87 (d, $J = 7.8$ Hz, 1H), 7.76 (d, $J = 7.0$ Hz, 1H), 7.40 (t, $J = 7.9$ Hz, 1H), 7.08 (d, $J = 30.3$ Hz, 1H), 4.41 (q, $J = 7.1$ Hz, 2H), 1.41 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 186.96 (s), 160.10 (d, $J = 34.0$ Hz), 152.80 (d, $J = 289.8$ Hz), 138.57 (s), 136.96 (s), 131.76 (s), 130.55 (s), 127.42 (s), 123.31 (s), 111.49 (s), 63.15 (s), 14.19 (s). ^{19}F NMR (470 MHz, CDCl_3) δ -108.65 (s). HRMS (ESI) Calcd. For 300.9876, $\text{C}_{12}\text{H}_{10}\text{BrFO}_3$ $[\text{M}-\text{H}]^+$, found 300.9889.

ethyl (Z)-4-(4-acetoxyphenyl)-2-fluoro-4-oxobut-2-enoate (4g)



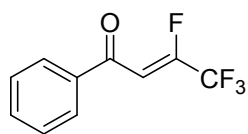
The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-90:10 petrol ether/ethyl acetate). Yield: 70%. ^1H NMR (500 MHz, CDCl_3) δ 7.99 (d, $J = 8.6$ Hz, 2H), 7.25 (d, $J = 8.7$ Hz, 2H), 7.10 (d, $J = 30.7$ Hz, 1H), 4.39 (q, $J = 7.2$ Hz, 2H), 2.34 (s, 3H), 1.40 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 187.03 (s), 168.78 (s), 160.20 (d, $J = 35.1$ Hz), 155.21 (s), 152.33 (d, $J = 287.3$ Hz), 134.33 (s), 130.57 (s), 122.22 (s), 111.96 (s), 63.03 (s), 21.26 (s), 14.18 (s). ^{19}F NMR (470 MHz, CDCl_3) δ -109.98 (s). HRMS (ESI) Calcd. For 303.0645, $\text{C}_{14}\text{H}_{13}\text{FO}_5$ $[\text{M}-\text{Na}]^+$, found 303.0659.

ethyl (Z)-2-fluoro-4-(naphthalen-2-yl)-4-oxobut-2-enoate (4h)



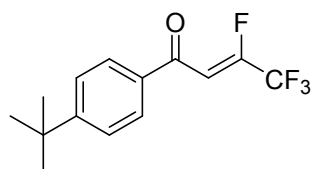
The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-95:5 petrol ether/ethyl acetate). Yield: 72%. ^1H NMR (500 MHz, CDCl_3) δ 8.44 (s, 1H), 8.03 (d, $J = 8.6$ Hz, 1H), 7.99 (t, $J = 7.5$ Hz, 1H), 7.93 (d, $J = 8.5$ Hz, 1H), 7.90 (d, $J = 8.2$ Hz, 1H), 7.64 (t, $J = 7.4$ Hz, 1H), 7.59 (t, $J = 7.5$ Hz, 1H), 7.28 (d, $J = 30.0$ Hz, 1H), 4.43 (q, $J = 7.1$ Hz, 2H), 1.43 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 188.16 (s), 160.41 (d, $J = 35.3$ Hz), 152.25 (d, $J = 286.0$ Hz), 136.14 (s), 134.23 (s), 132.57 (s), 131.37 (s), 129.88 (s), 129.23 (s), 129.02 (s), 128.03 (s), 127.20 (s), 123.85 (s), 112.39 (s), 63.04 (s), 14.23 (s). ^{19}F NMR (470 MHz, CDCl_3) δ -110.67 (s). HRMS (ESI) Calcd. For 273.0927, $\text{C}_{16}\text{H}_{13}\text{FO}_3$ $[\text{M}-\text{H}]^+$, found 273.0929.

(Z)-3,4,4,4-tetrafluoro-1-phenylbut-2-en-1-one (5a)⁹



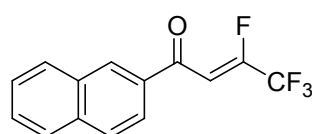
The title compound was isolated as a colorless liquid after chromatography on silica with a Combiflash system (100:0-98:2 petrol ether/ethyl acetate). Yield: 80%. ¹H NMR (500 MHz, CDCl₃) δ 7.94 (d, *J* = 7.4 Hz, 2H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.54 (t, *J* = 7.8 Hz, 2H), 6.73 (d, *J* = 31.4 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 186.65 (s), 136.39 (s), 134.50 (s), 129.13 (s), 128.88 (s), 107.92 (s). ¹⁹F NMR (470 MHz, CDCl₃) δ -73.14 (d, *J* = 10.0 Hz), -116.89 (q, *J* = 10.1 Hz).

(Z)-1-(4-(tert-butyl)phenyl)-3,4,4,4-tetrafluorobut-2-en-1-one (5b)



The title compound was isolated as a slightly yellow liquid after chromatography on silica with a Combiflash system (100:0-98:2 petrol ether/ethyl acetate). Yield: 78%. ¹H NMR (500 MHz, CDCl₃) δ 7.87 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 6.71 (d, *J* = 31.7 Hz, 1H), 1.36 (s, 9H). ¹³C NMR (126 MHz, CDCl₃) δ 185.13 (s), 157.54 (s), 150.90 (q, *J* = 39.9 Hz), 148.65 (q, *J* = 39.9 Hz), 132.76 (s), 127.82 (s), 125.02 (s), 120.35-113.49 (m), 107.04 (s), 34.35 (s), 30.02 (s). ¹⁹F NMR (470 MHz, CDCl₃) δ -73.15 (d, *J* = 10.2 Hz), -117.78 (q, *J* = 11.0 Hz). HRMS (EI) Calcd. For 274.0981, C₁₄H₁₄F₄O₃, found 274.0975.

(Z)-3,4,4,4-tetrafluoro-1-(naphthalen-2-yl)but-2-en-1-one (5c)¹⁰



The title compound was isolated as a slightly yellow solid after chromatography on silica with a Combiflash system (100:0-97:3 petrol ether/ethyl acetate). Yield: 75%. ¹H NMR (500 MHz, CDCl₃) δ 8.41 (s, 1H), 8.01 (d, *J* = 7.9 Hz, 2H), 7.95 (d, *J* = 8.6 Hz, 1H), 7.92 (d, *J* = 8.1 Hz, 1H), 7.67 (t, *J* = 7.3 Hz, 1H), 7.61 (t, *J* = 7.4 Hz, 1H), 6.87 (d, *J* = 31.3 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 186.47 (s), 136.26 (s), 133.80 (s), 132.52 (s), 131.36 (s), 129.90 (s), 129.46 (s), 129.22 (s), 128.07 (s), 127.37 (s), 123.69 (s), 108.03 (s). ¹⁹F NMR (470 MHz, CDCl₃) δ -73.04 (d, *J* = 10.2 Hz), -117.09 (q, *J* = 11.0 Hz).

Reference

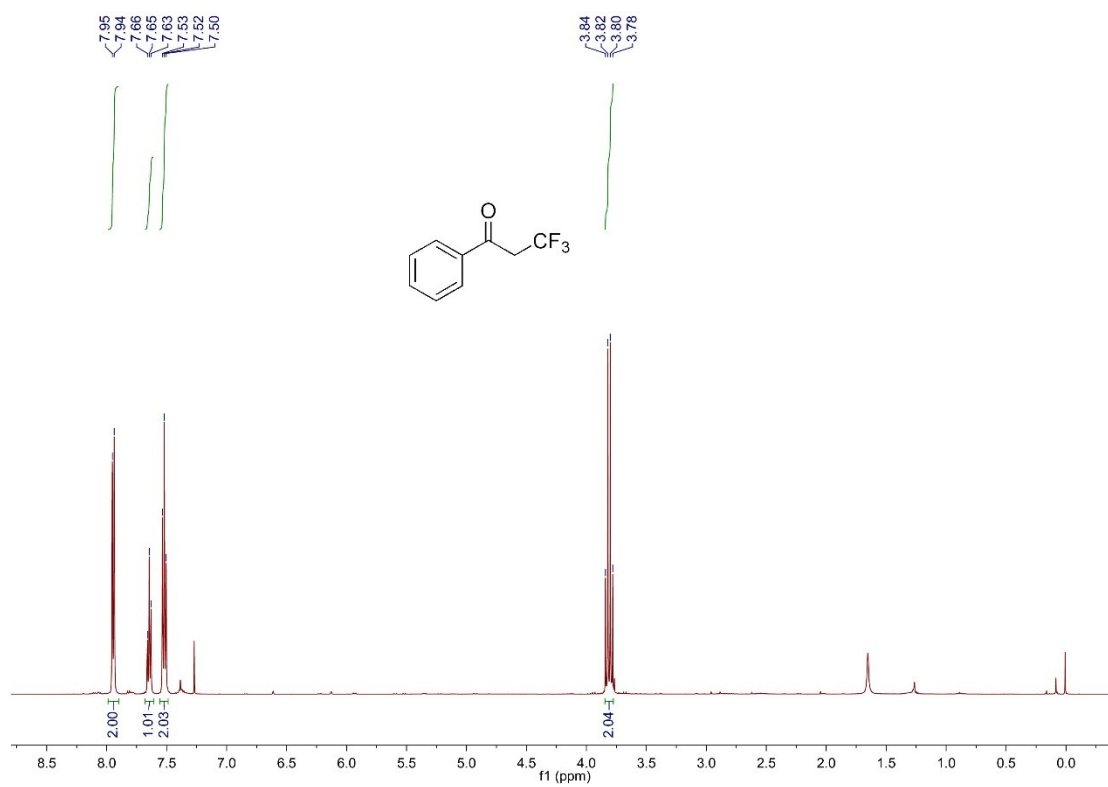
- (1) Gallagher, K. J.; Webster, R. L., Chem. Commun. 2014, 50 (81), 12109-12111..
- (2) Deb, A.; Manna, S.; Modak, A.; Patra, T.; Maity, S.; Maiti, D., Angew. Chem., Int. Ed., 2013, 52 (37), 9747-9750.
- (3) He, Z.; Zhang, R.; Hu, M.; Li, L.; Ni, C.; Hu, J., Chem. Sci., 2013, 4 (9), 3478.
- (4) CAS: 1639446-64-2.
- (5) Lu, Q.; Liu, C.; Huang, Z.; Ma, Y.; Zhang, J.; Lei, A., Chem. Commun., 2014, 50 (91), 14101-4.
- (6) Ohtsuka, Y.; Uruguchi, D.; Yamamoto, K.; Tokuhisa, K.; Yamakawa, T., J. Fluorine Chem., 2016, 181, 1-6.
- (7) Novák, P.; Lishchynskiy, A.; Grushin, V. V., J. Am. Chem. Soc., 2012, 134 (39), 16167-16170.

(8) K. Sato, Y. Ogawa, M. Tamura, M. Harada, T. Ohara, M. Omote, A. Ando and I. Kumadaki, Collect. Czech. Chem. Commun., 2002, 67, 1285;

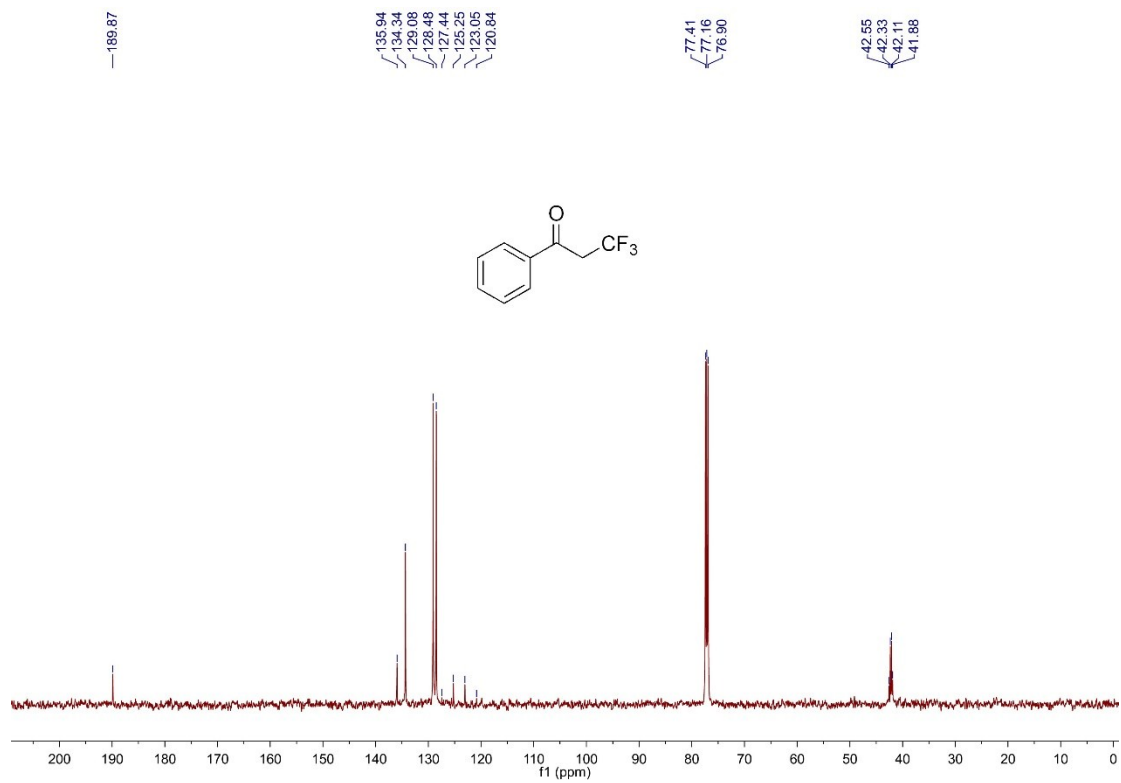
(9) Sano, K.; Fukuhara, T.; Hara, S., J. Fluorine Chem., 2009, 130 (8), 708-713.

(10) CAS: 1214712-41-0

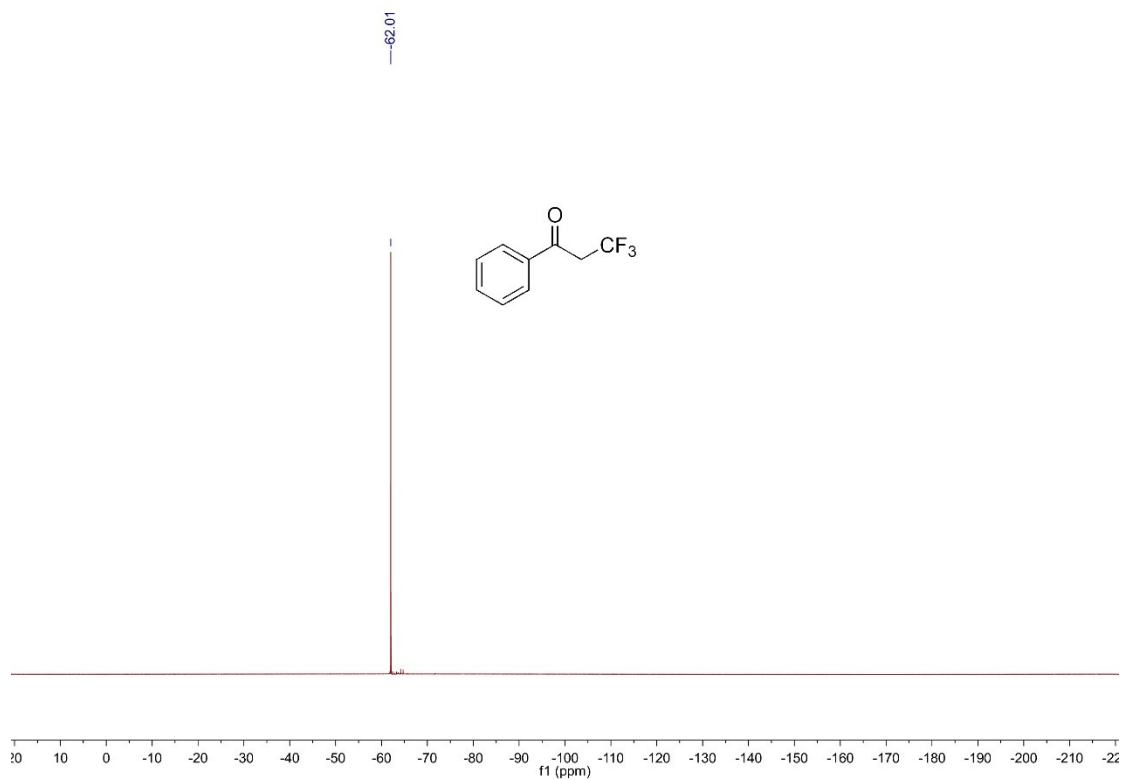
5. NMR Spectra of all products



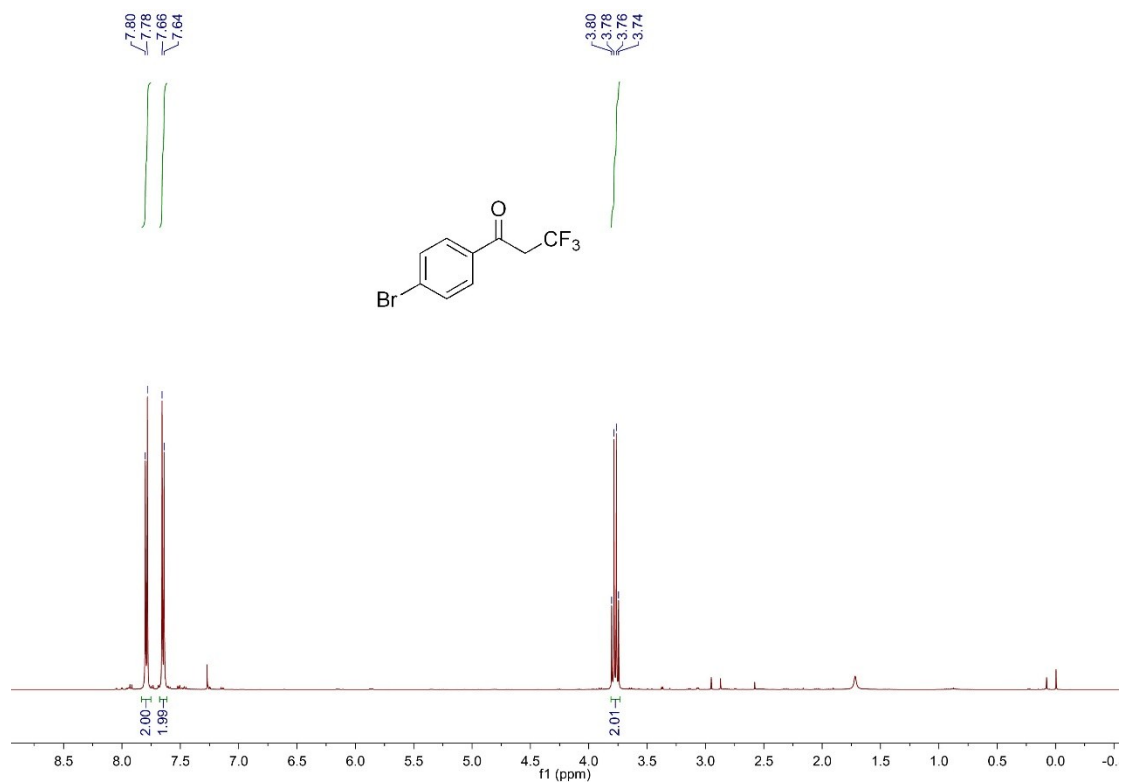
^1H NMR **3a**



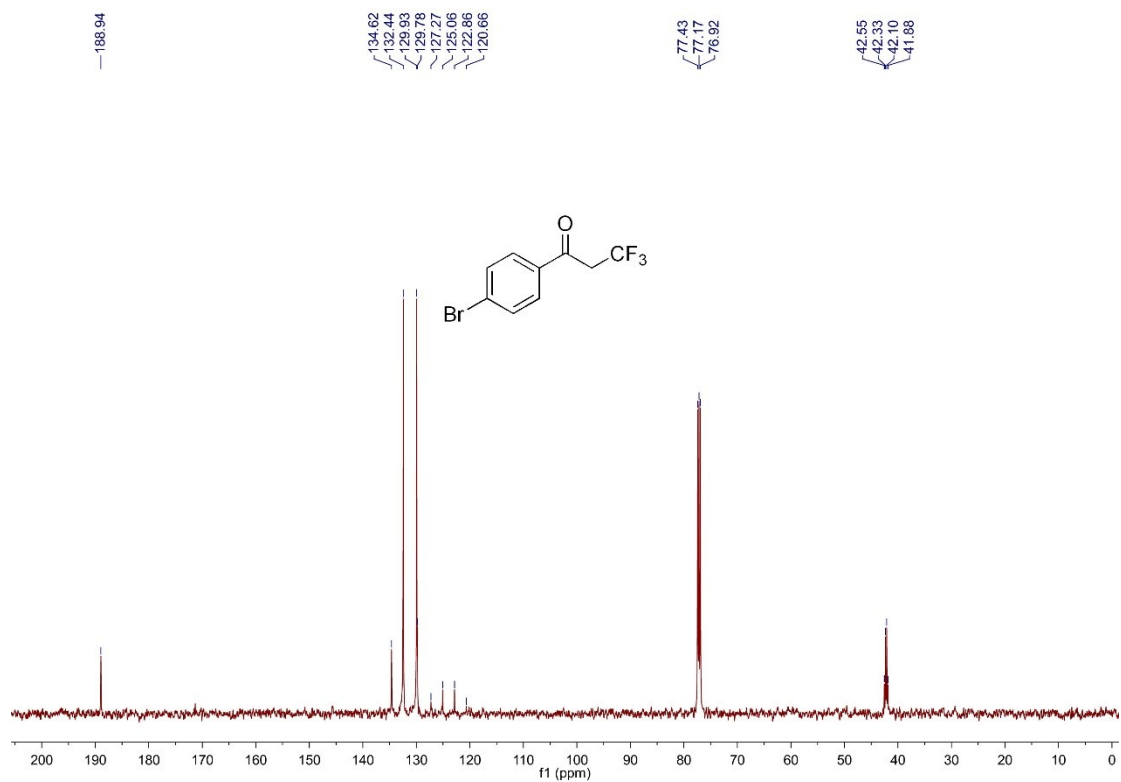
¹³C NMR **3a**



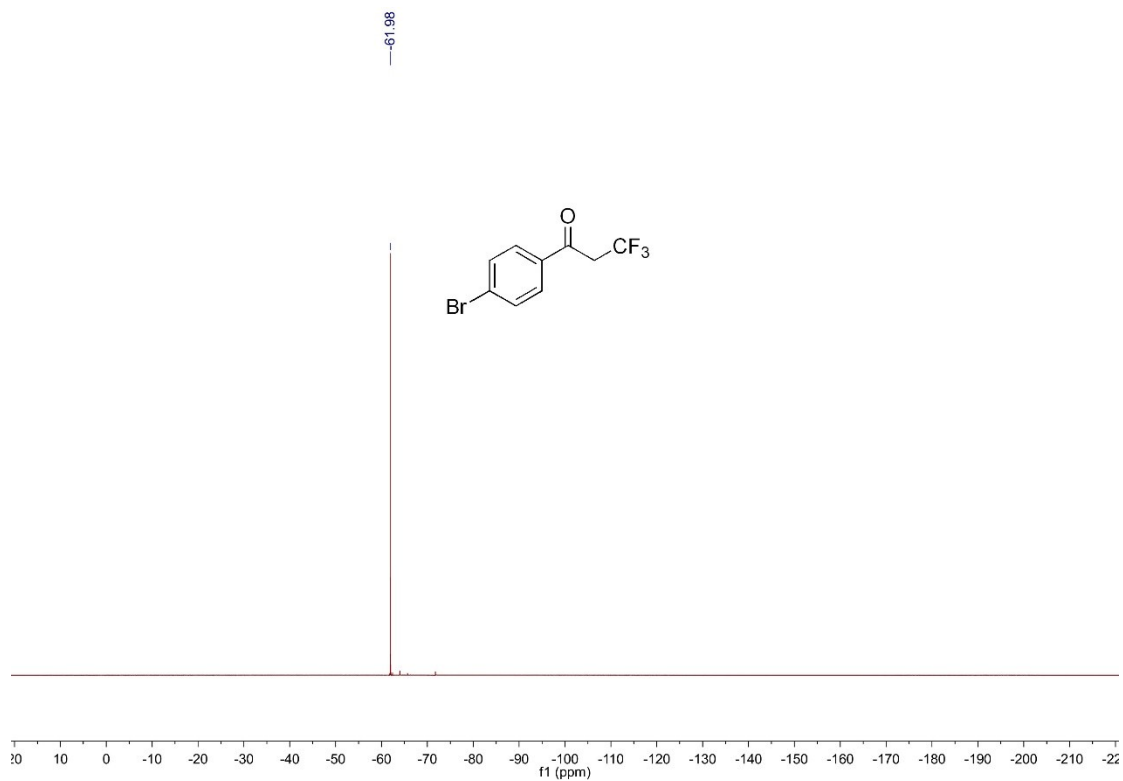
¹⁹F NMR **3a**



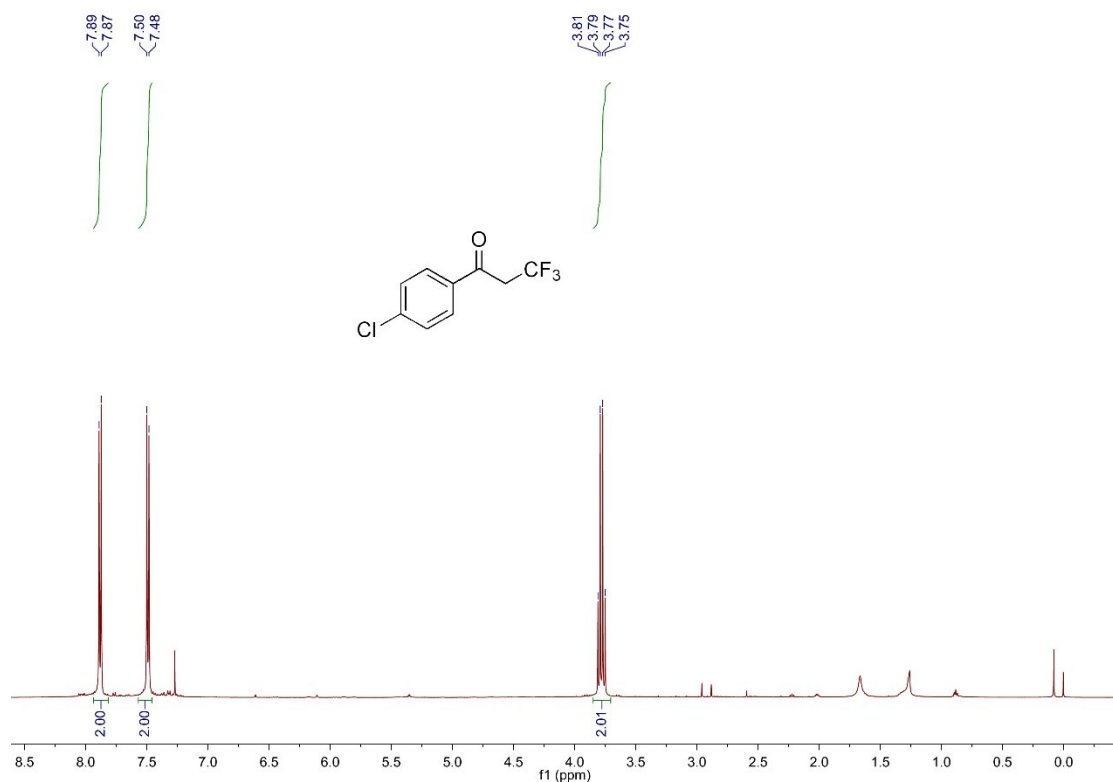
¹H NMR 3b



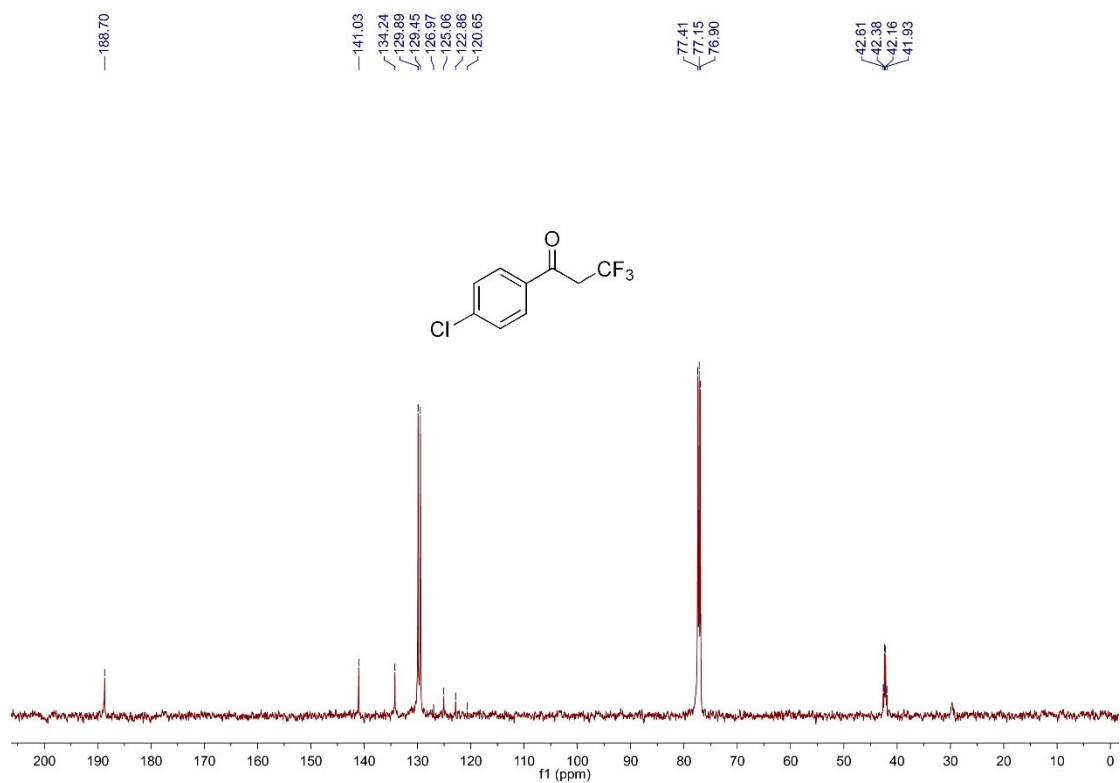
¹³C NMR 3b



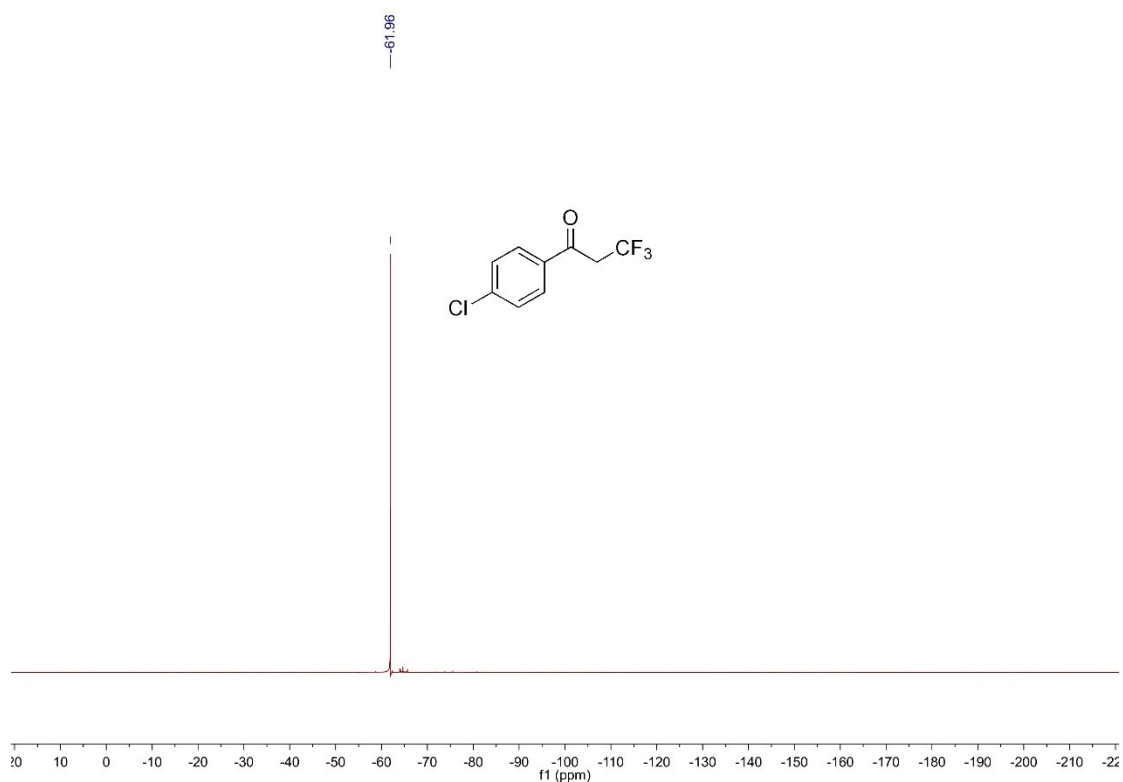
^{19}F NMR 3b



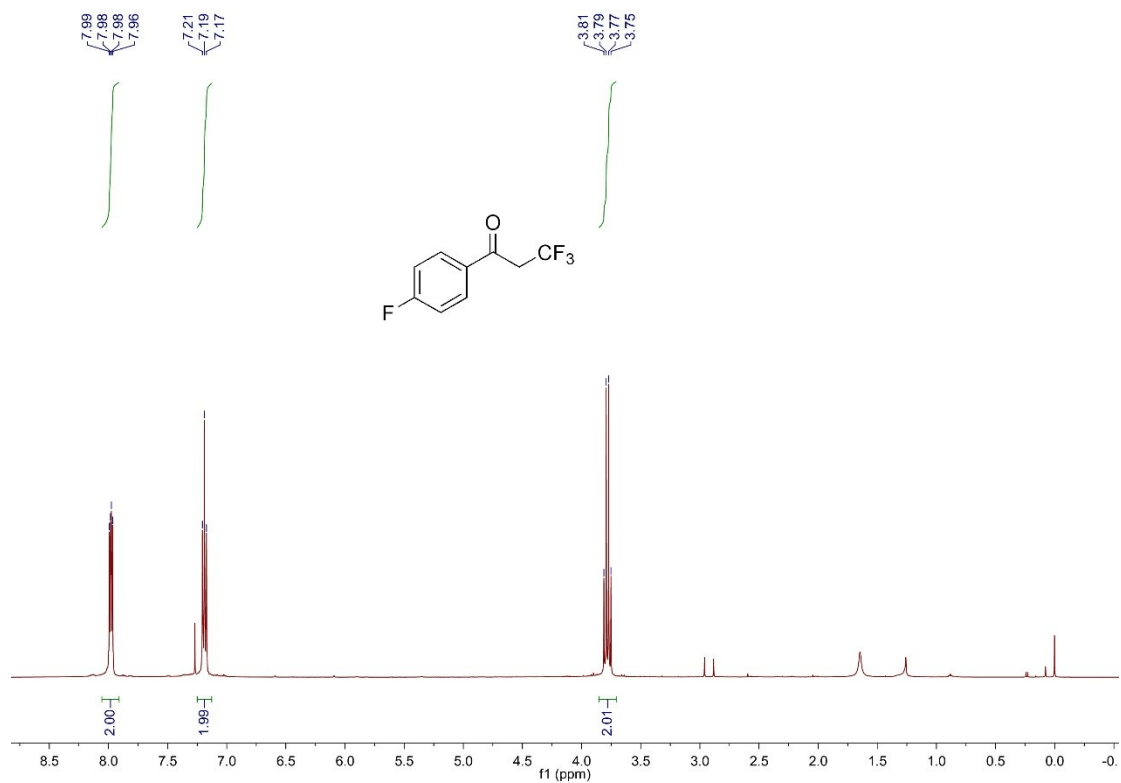
^1H NMR 3c



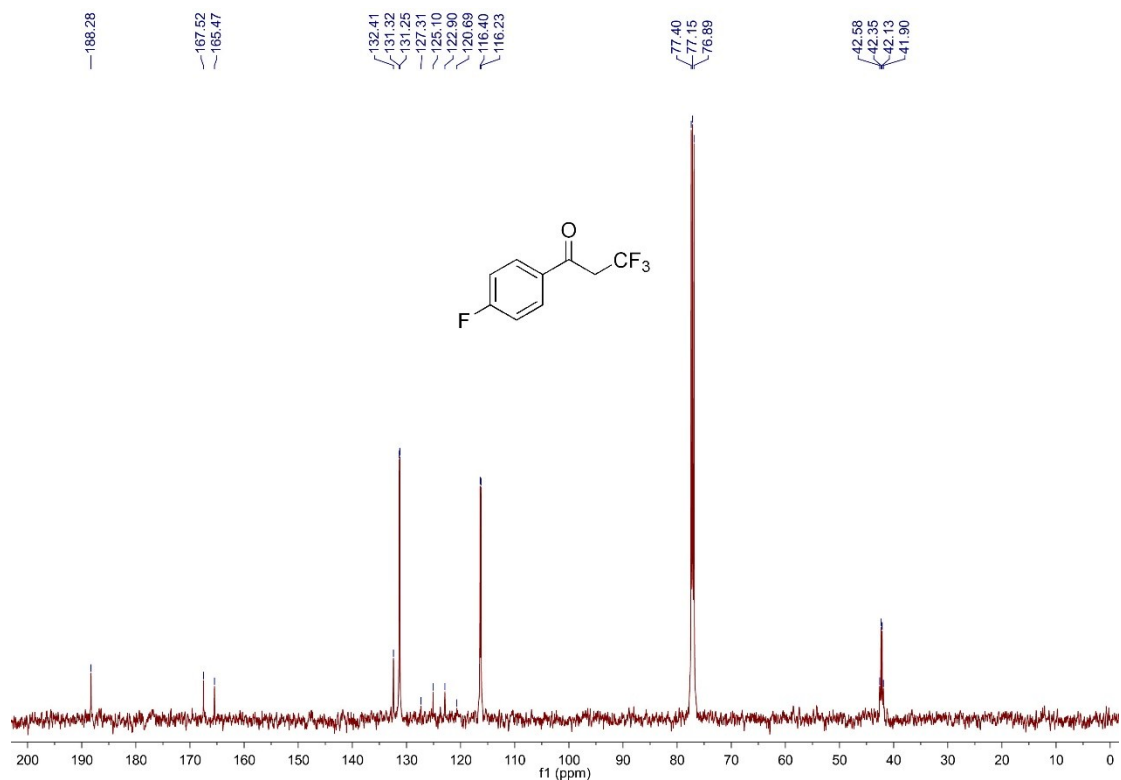
^{13}C NMR **3c**



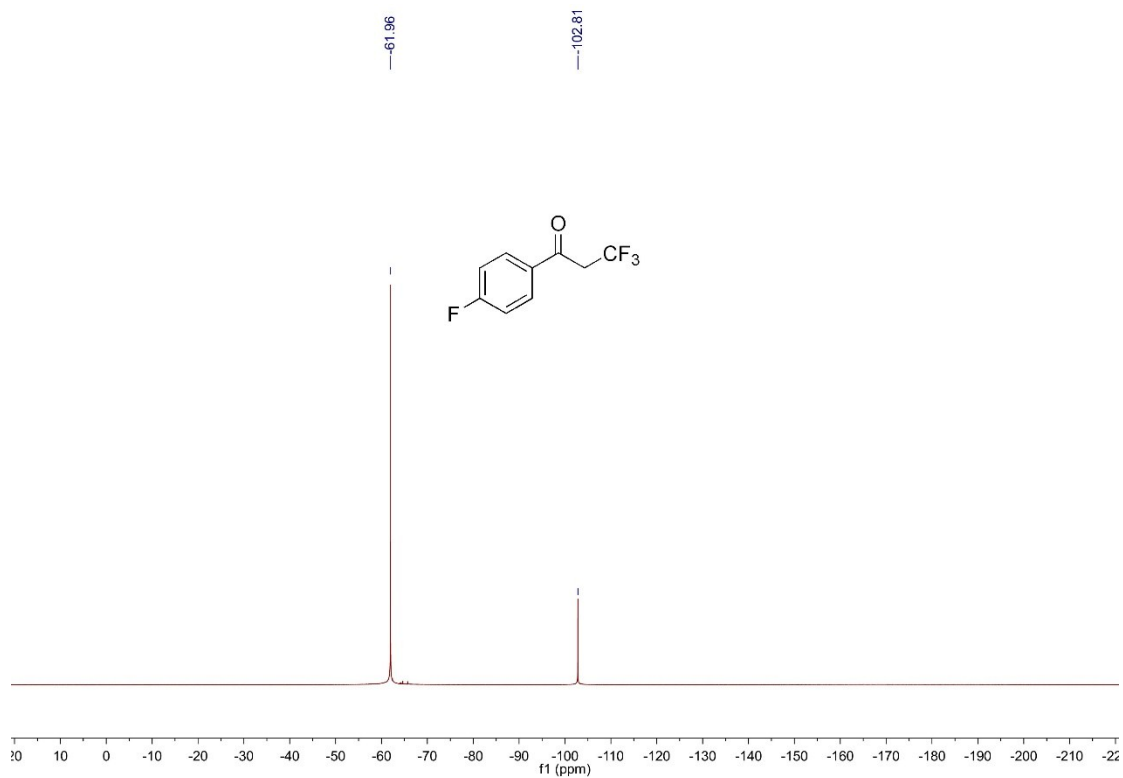
^{19}F NMR **3c**



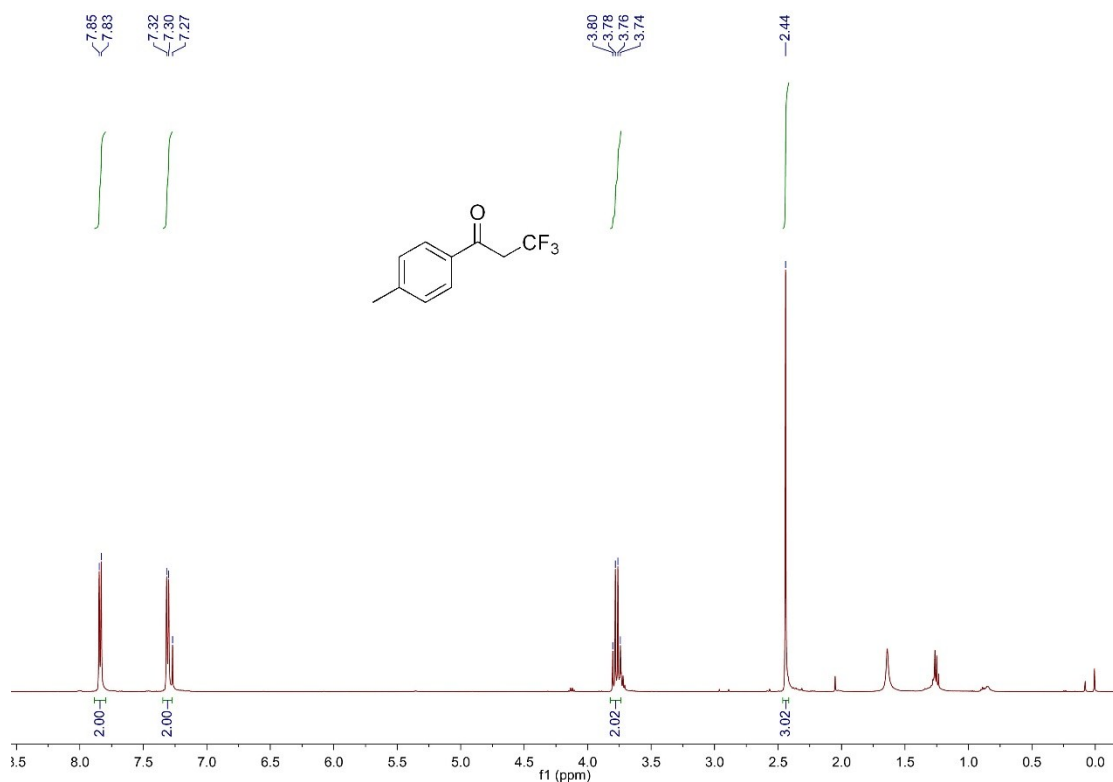
¹H NMR 3d



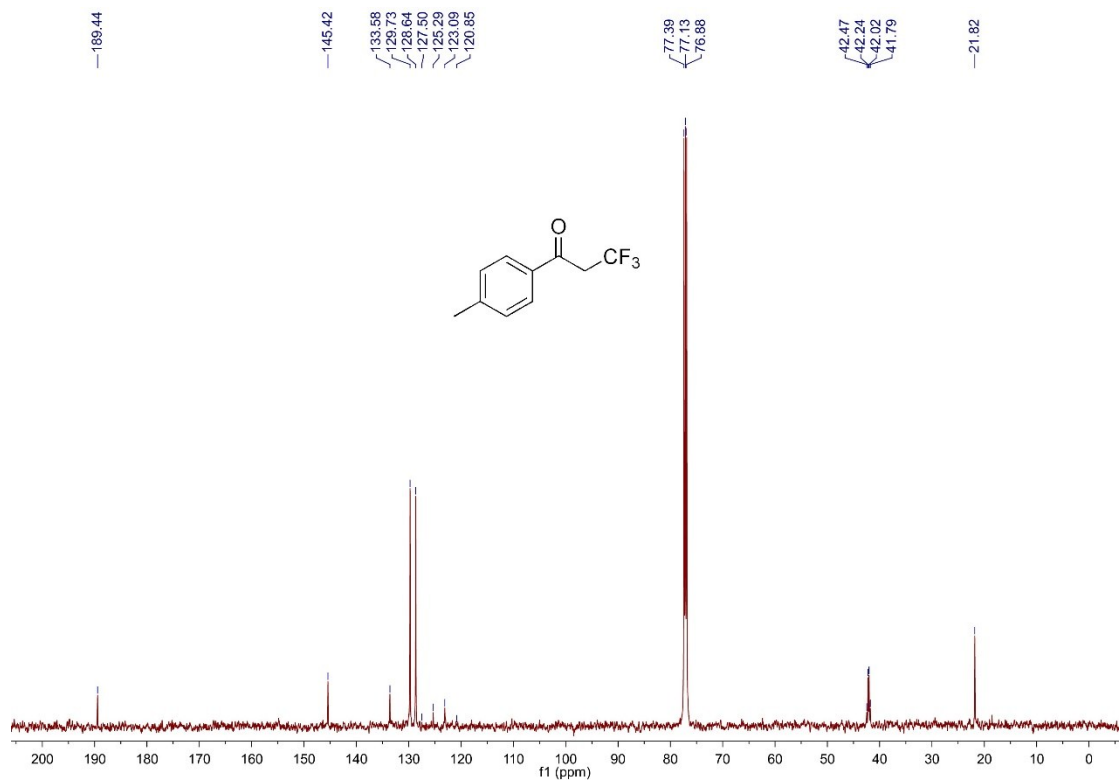
¹³C NMR 3d



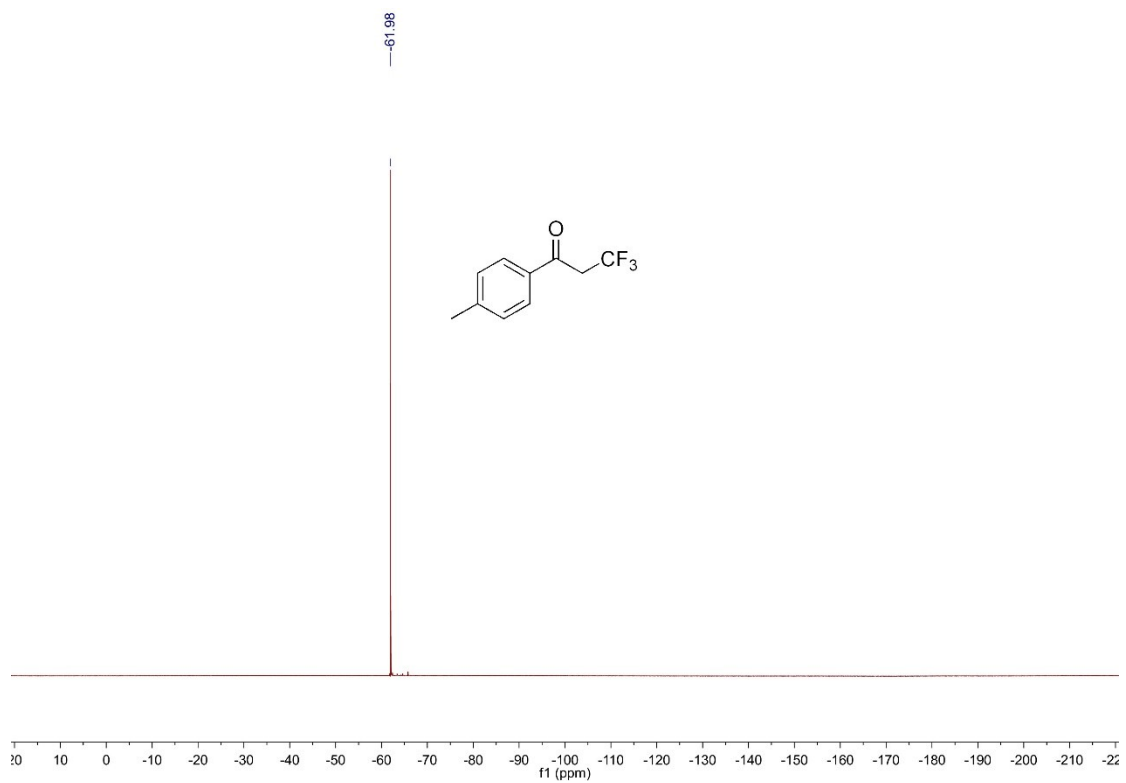
¹⁹F NMR 3d



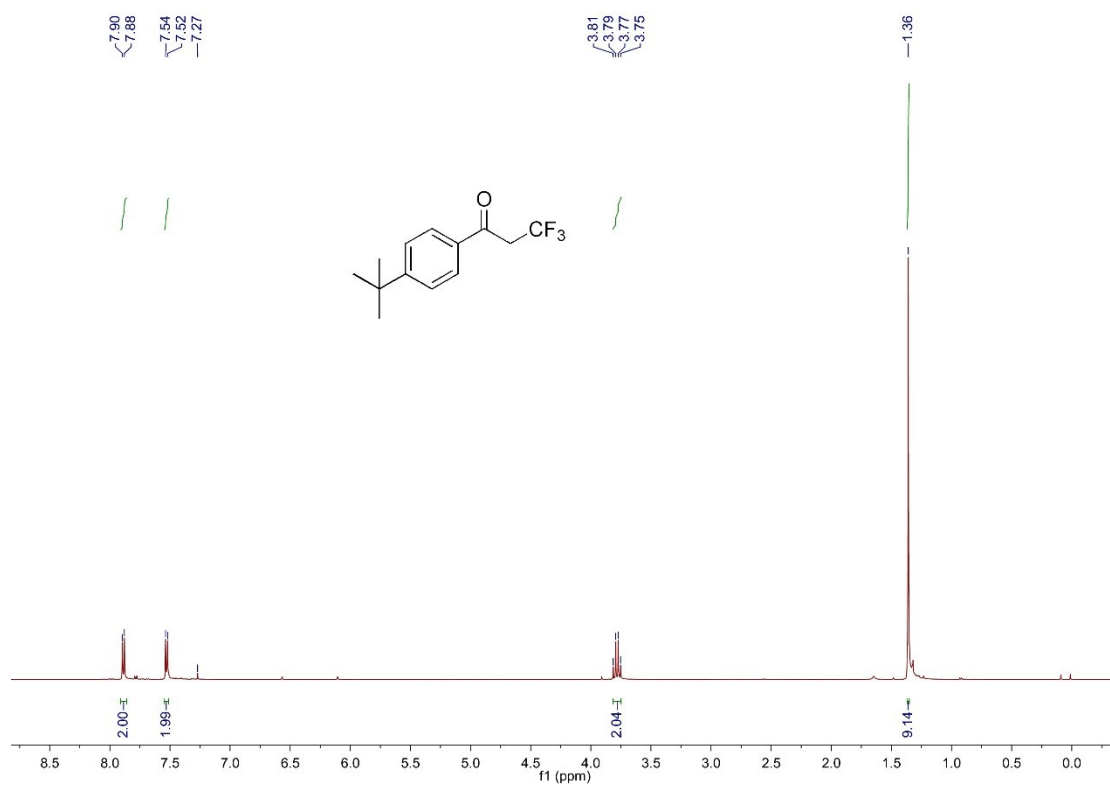
¹H NMR 3e



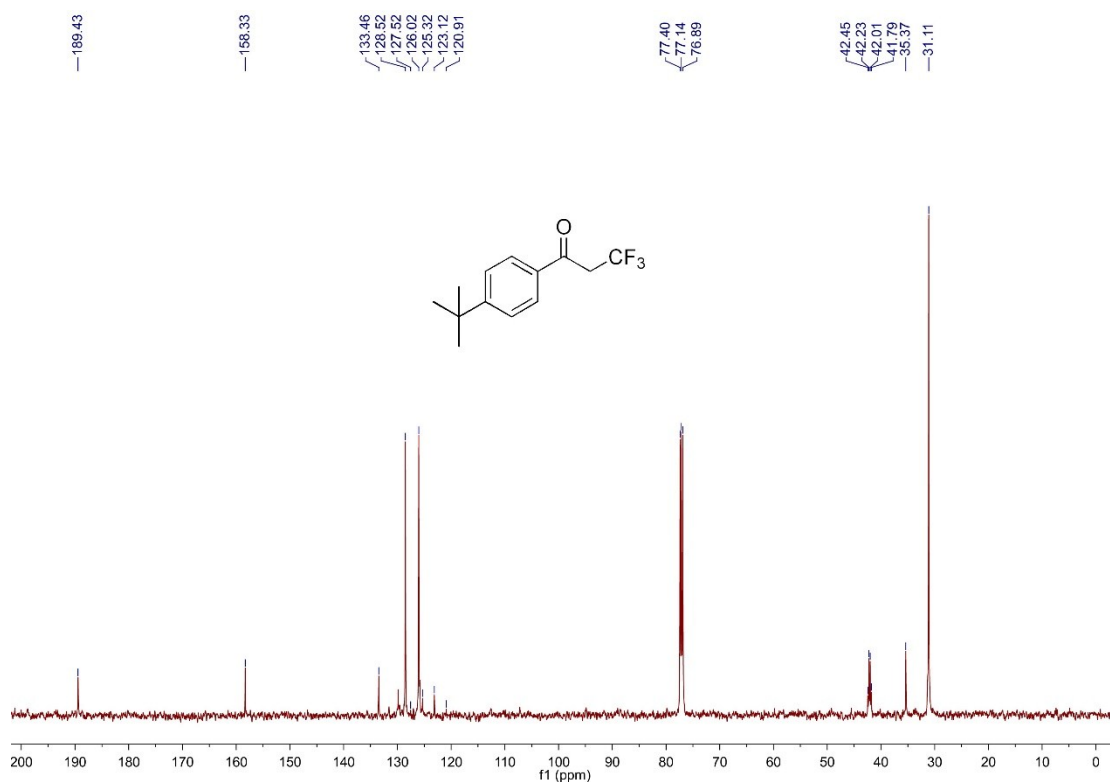
¹³C NMR 3e



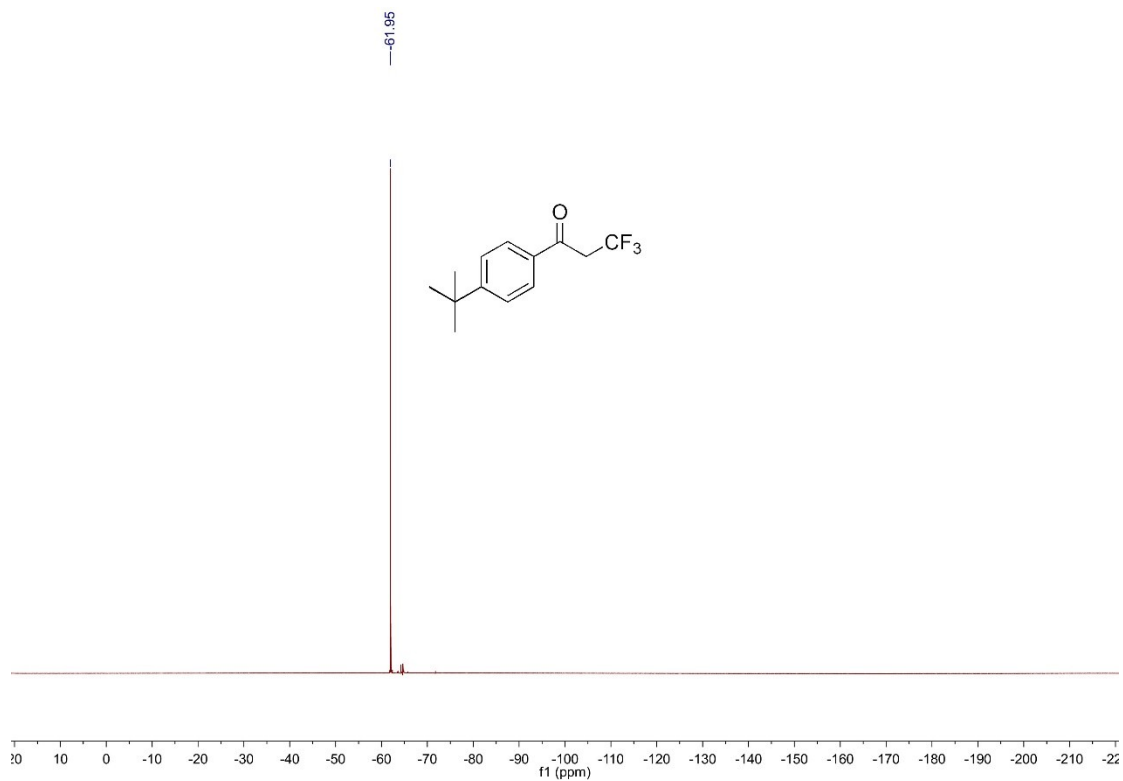
¹⁹F NMR 3e



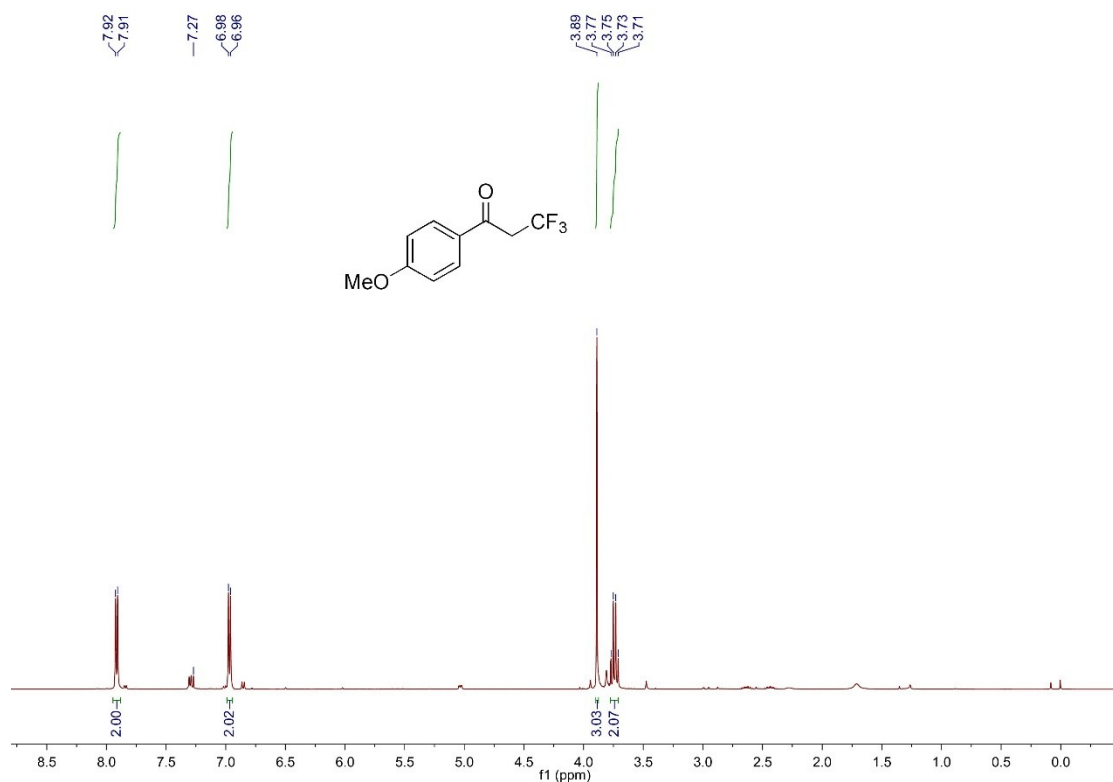
$^1\text{H NMR}$ 3f



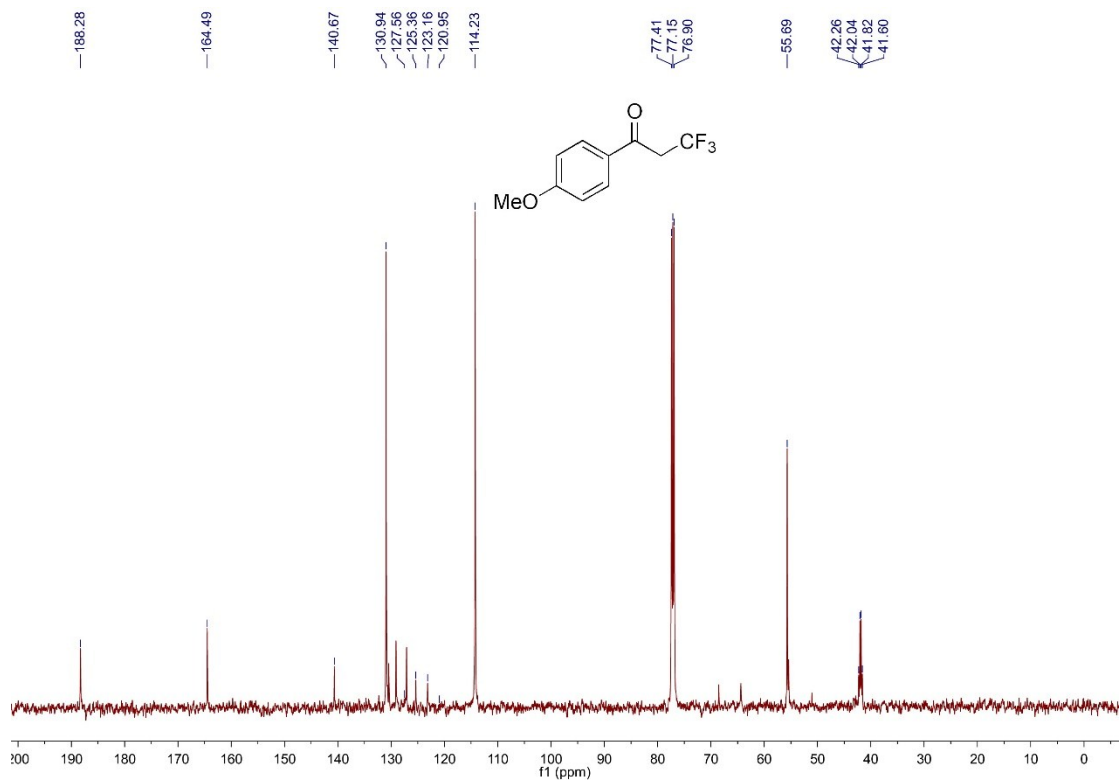
$^{13}\text{C NMR}$ 3f



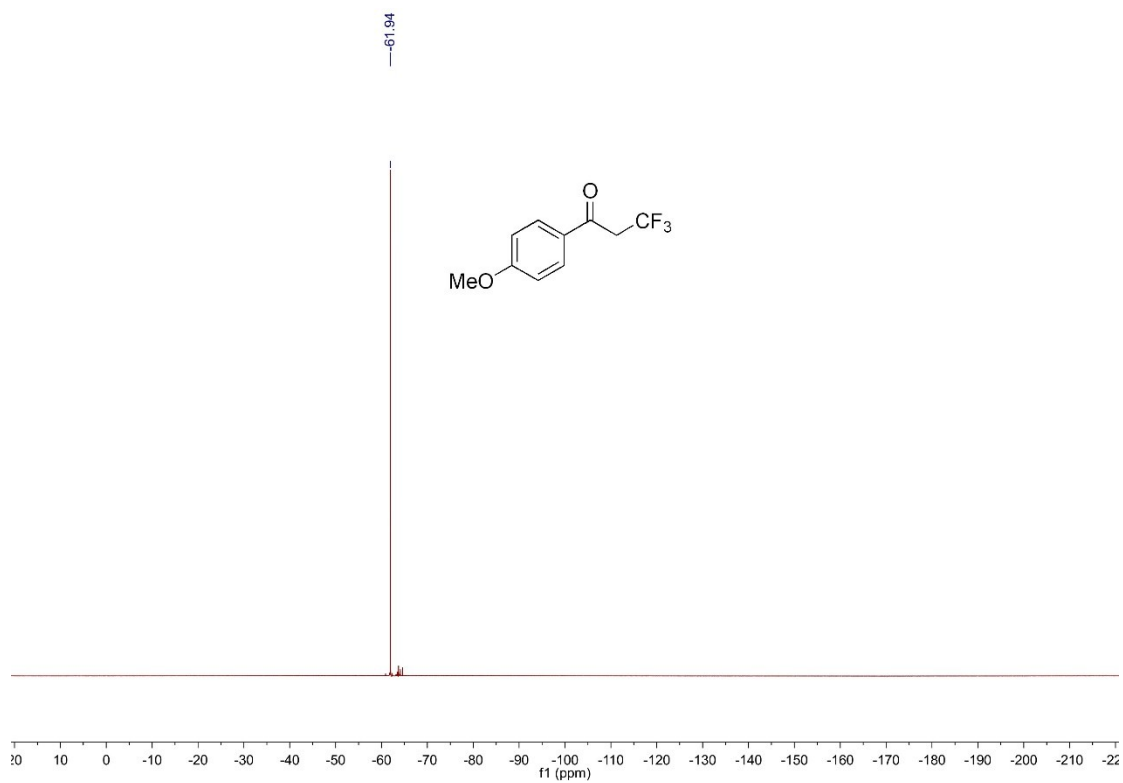
^{19}F NMR 3f



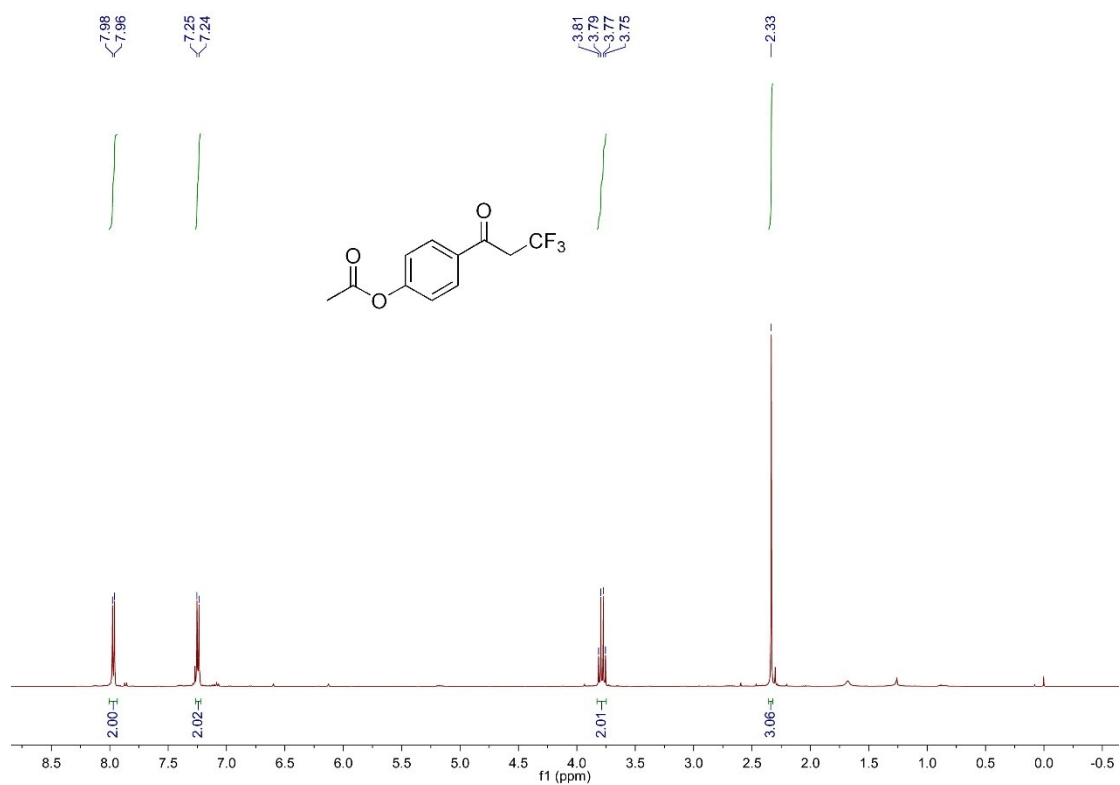
^1H NMR 3g



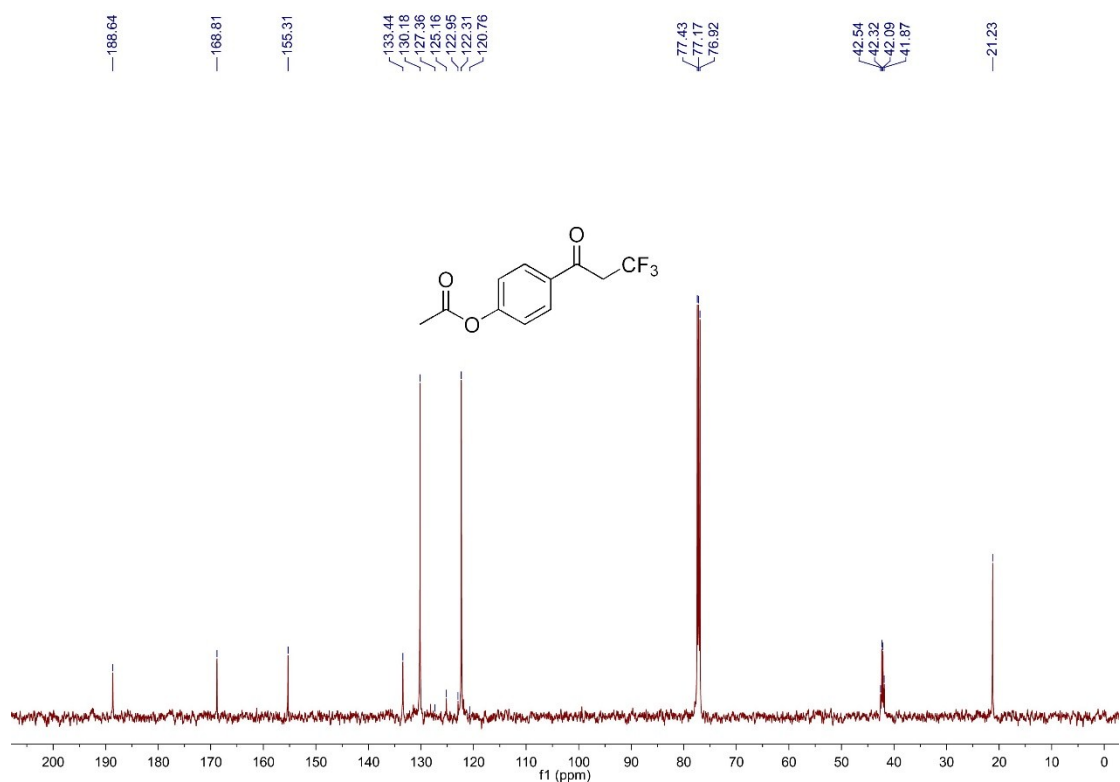
^{13}C NMR **3g**



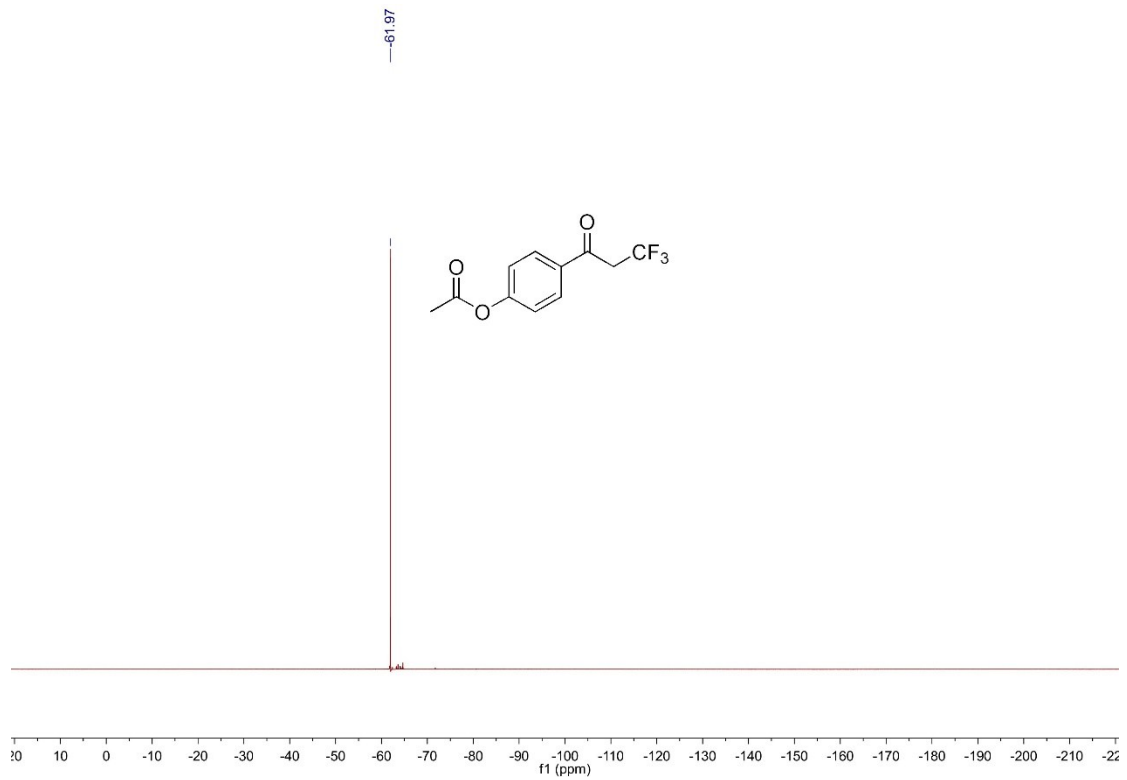
^{19}F NMR **3g**



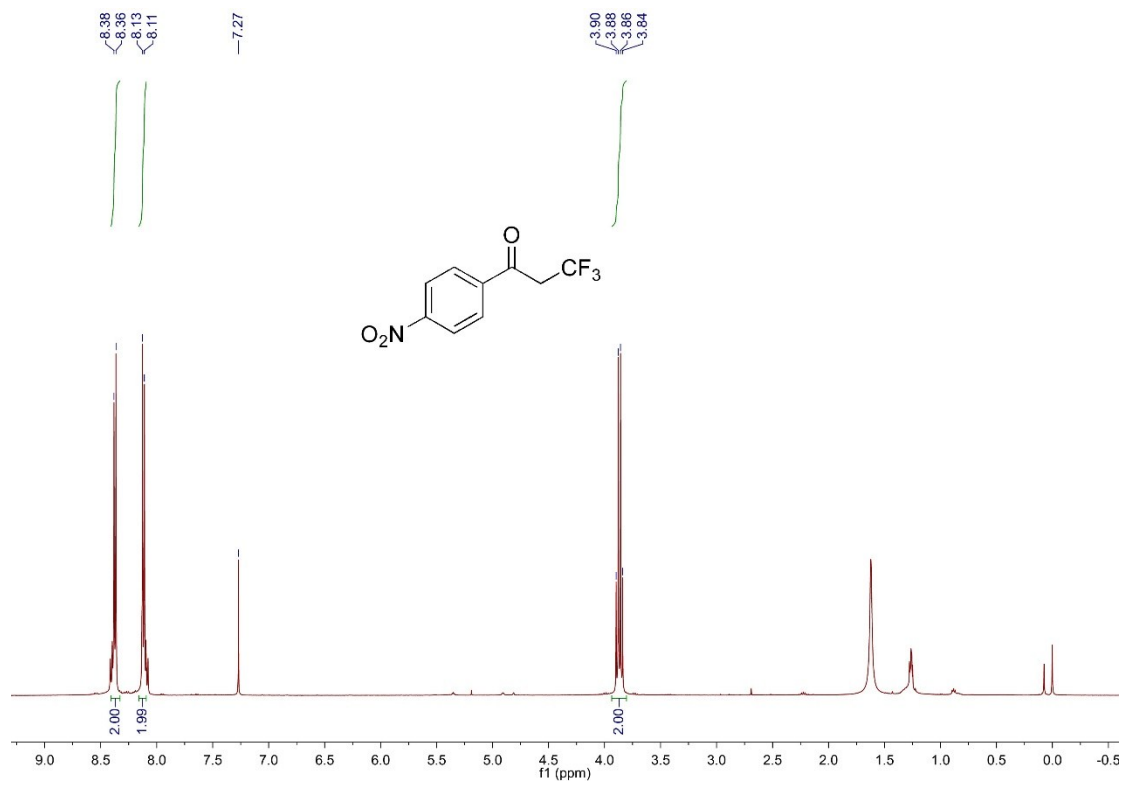
¹H NMR 3h



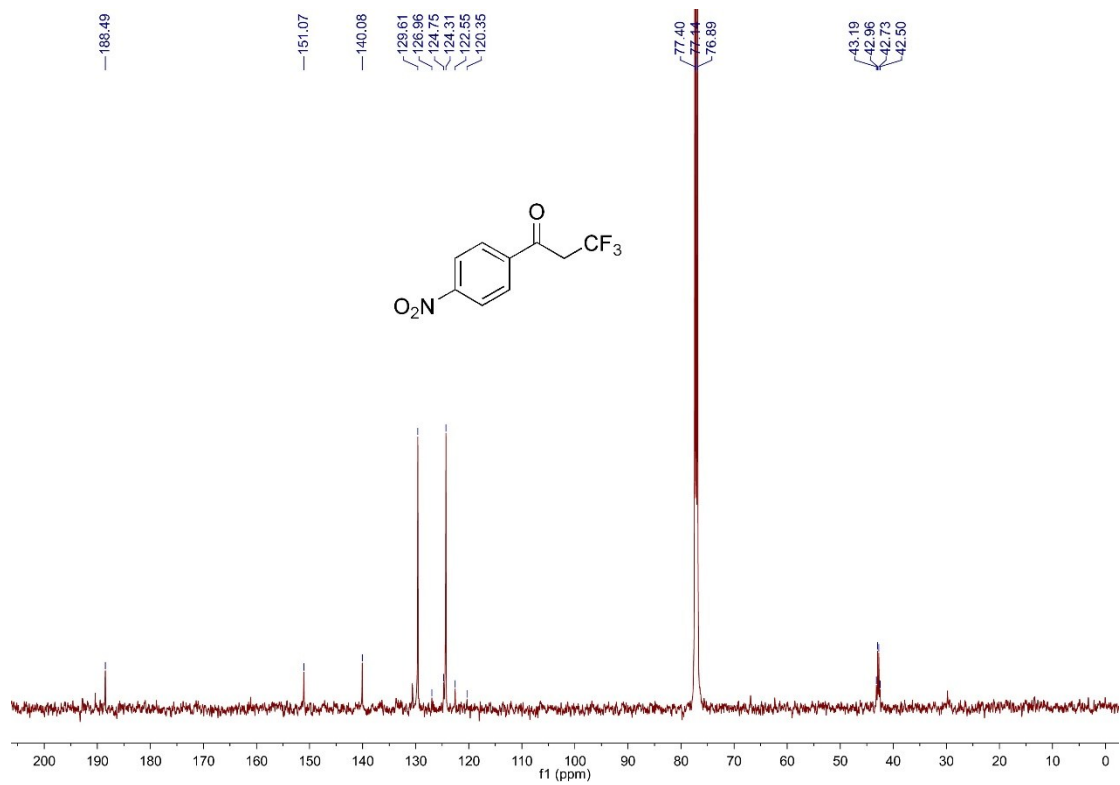
¹³C NMR 3h



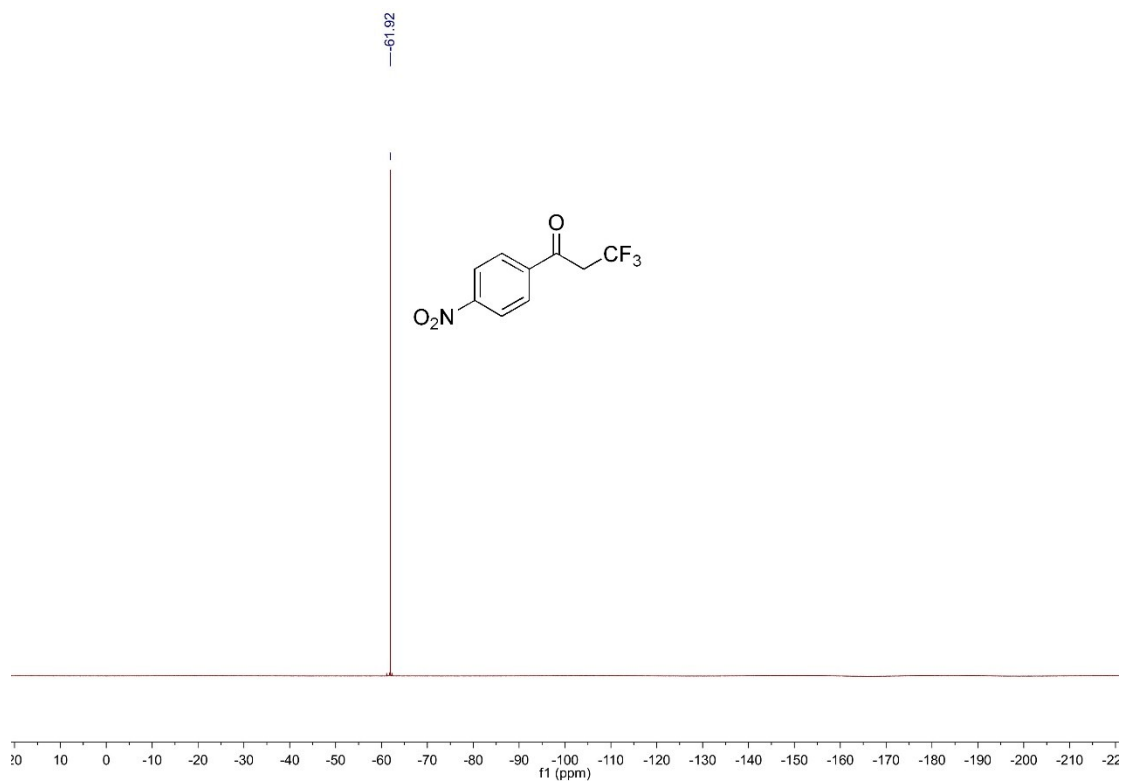
¹⁹F NMR 3h



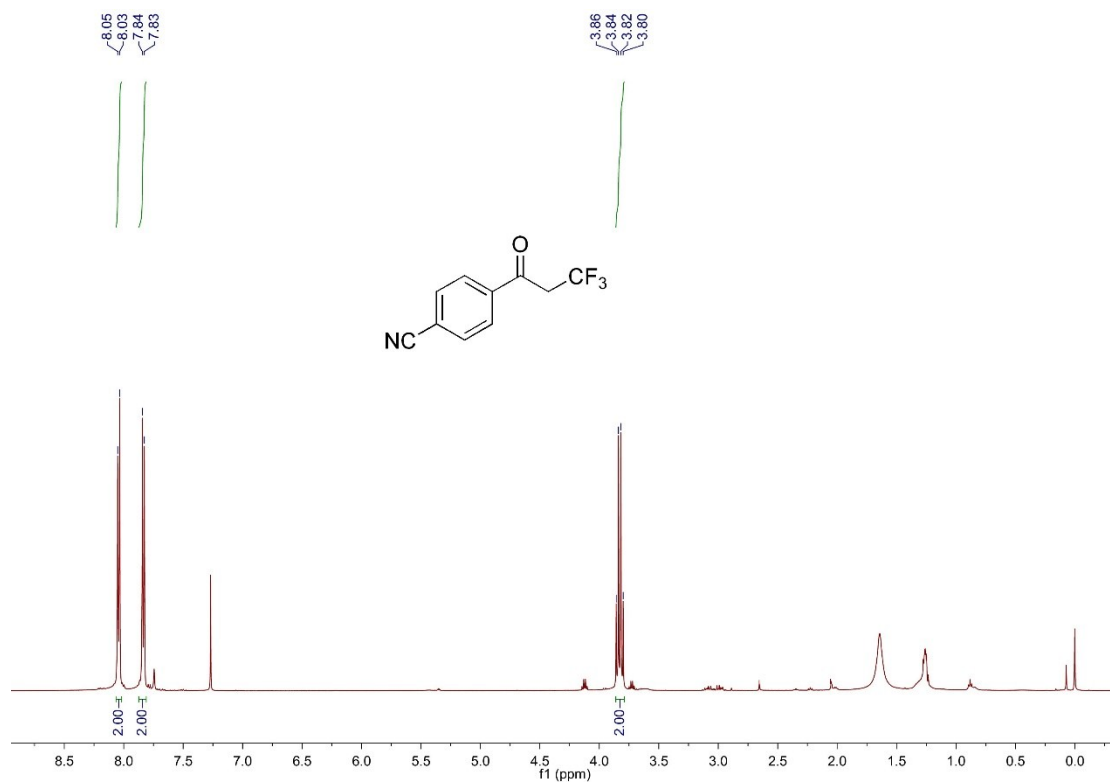
¹H NMR 3i



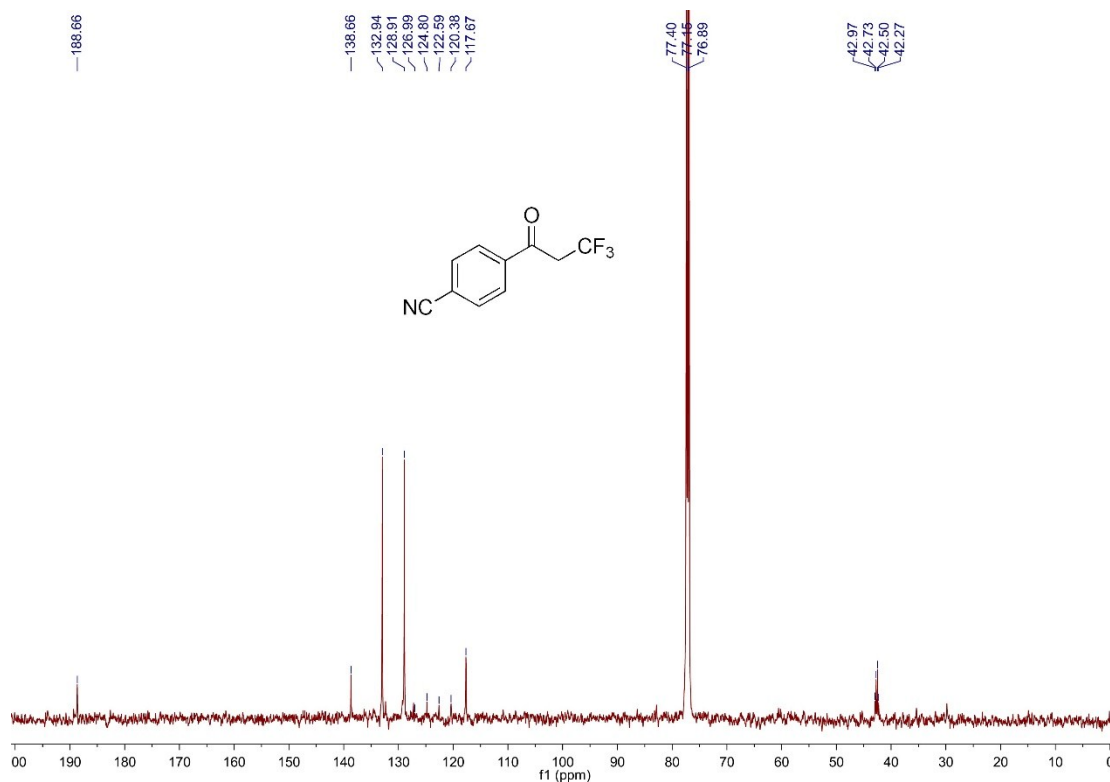
¹³C NMR 3i



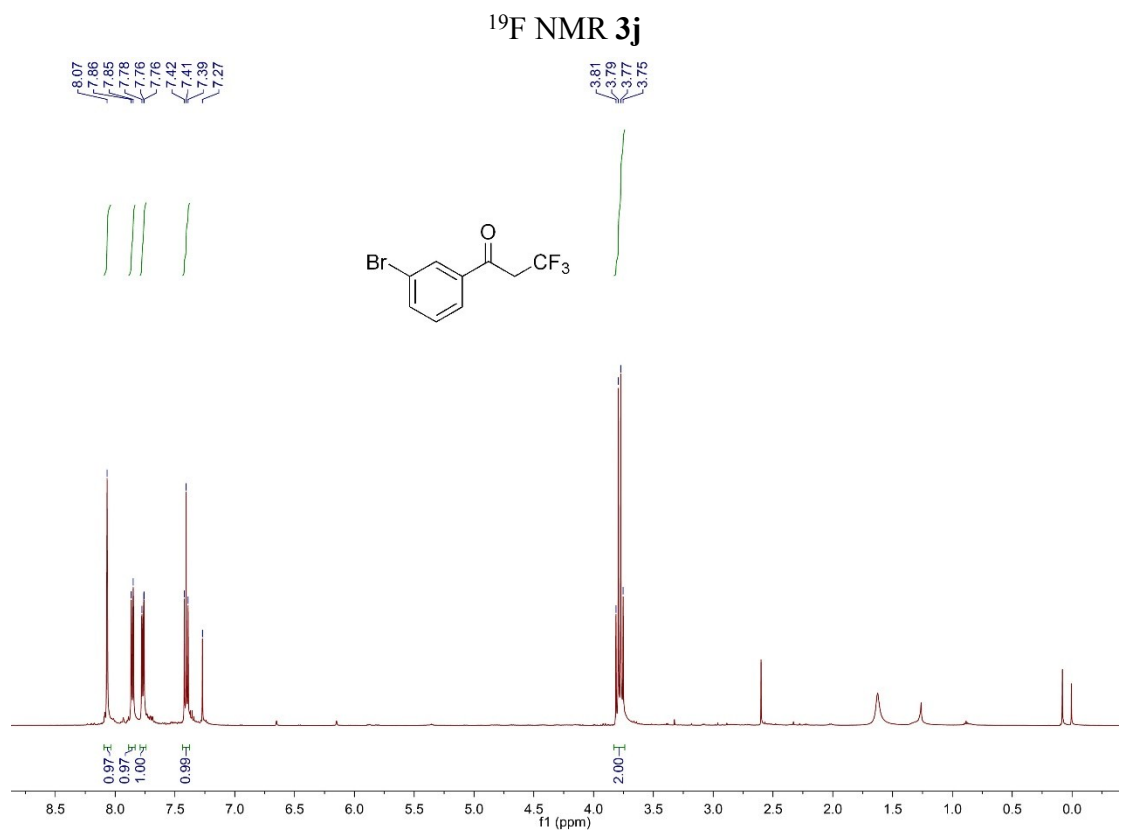
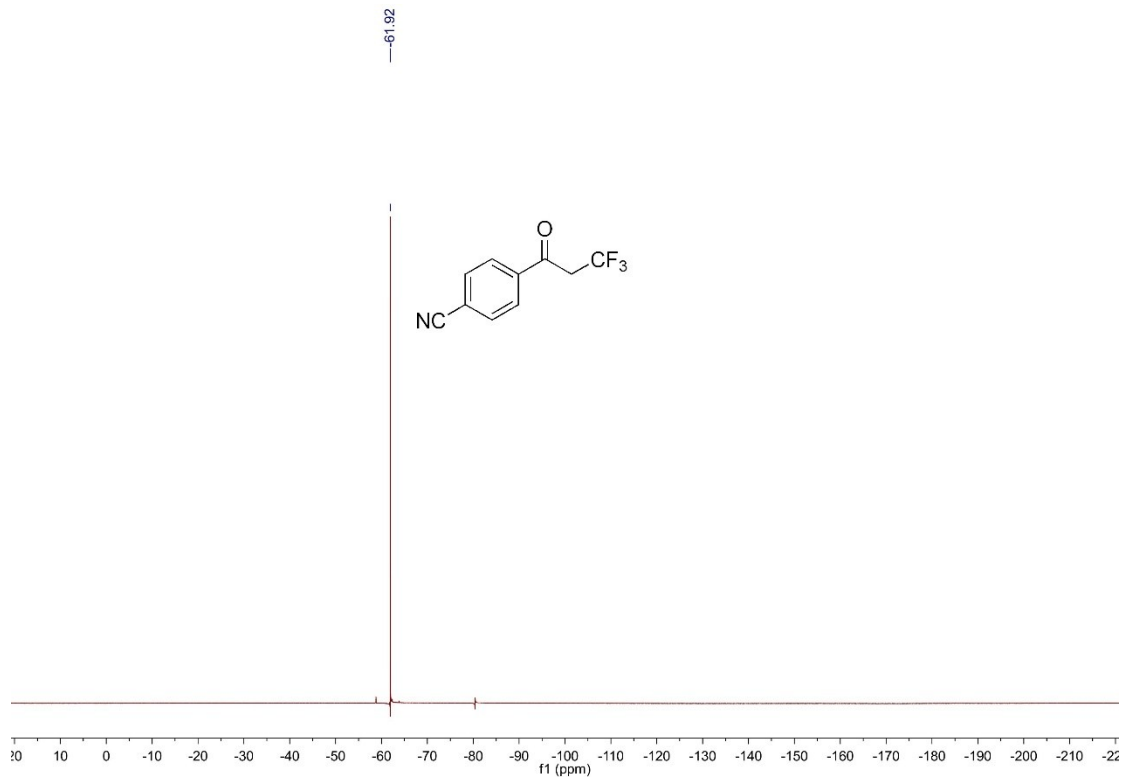
¹⁹F NMR 3i

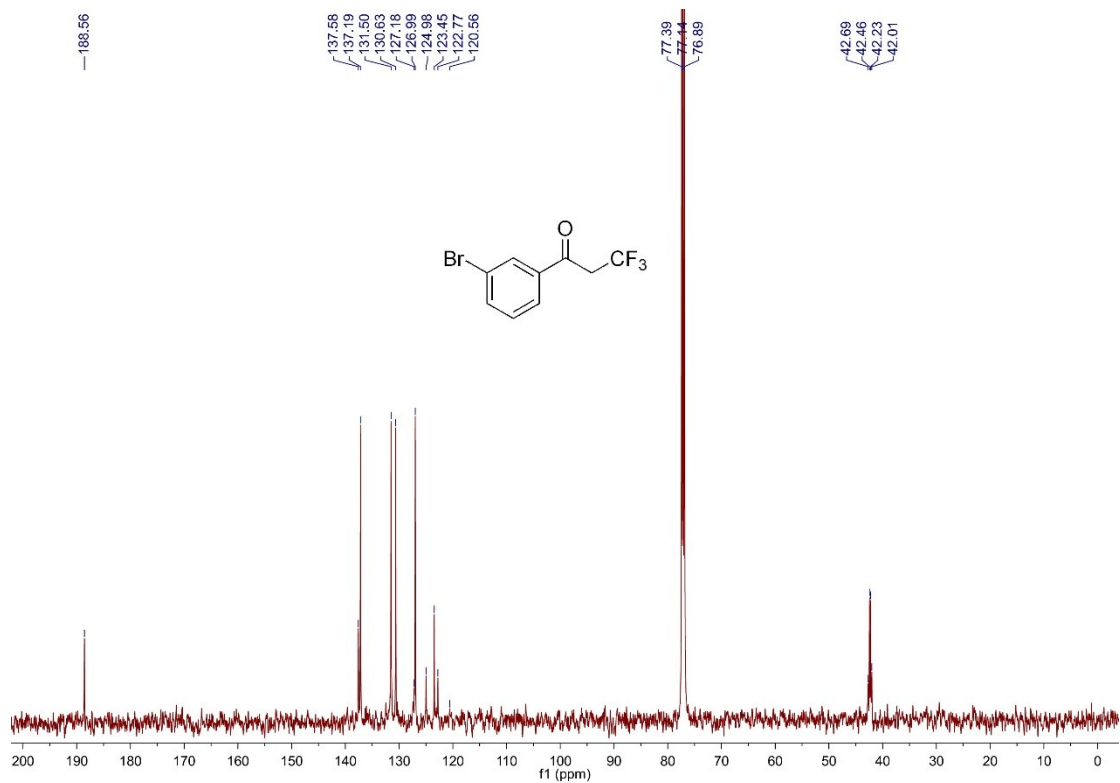


¹H NMR 3j

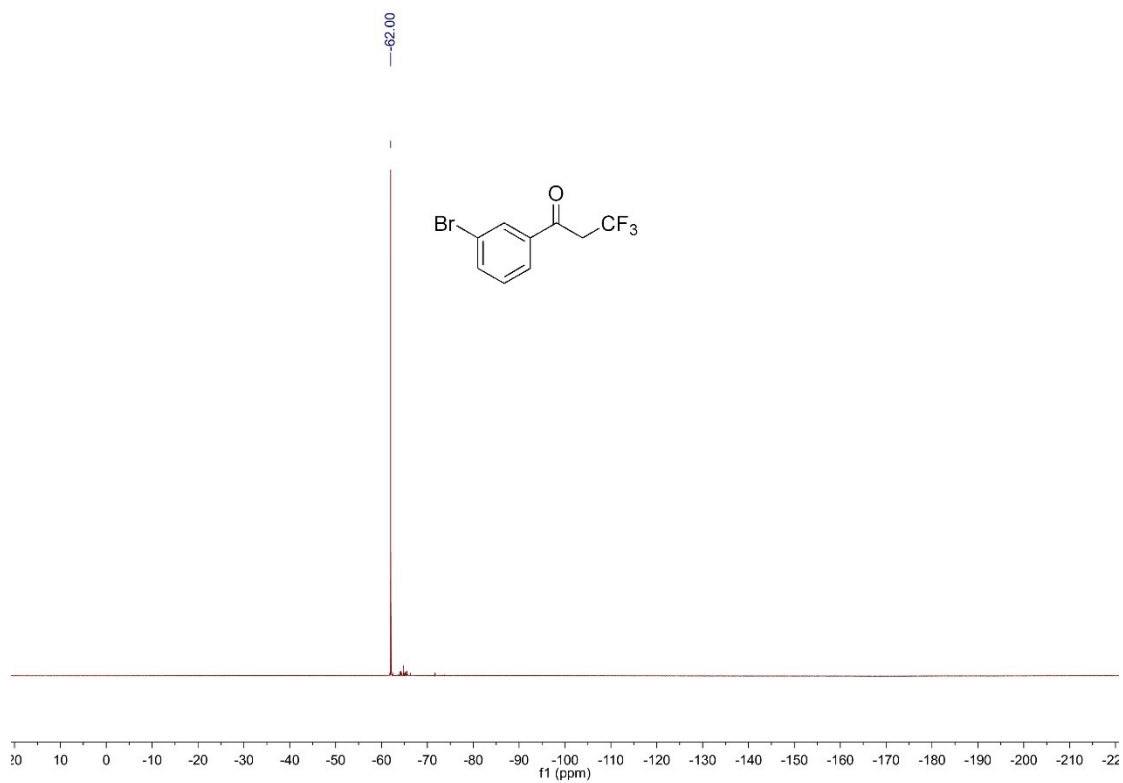


¹³C NMR 3j

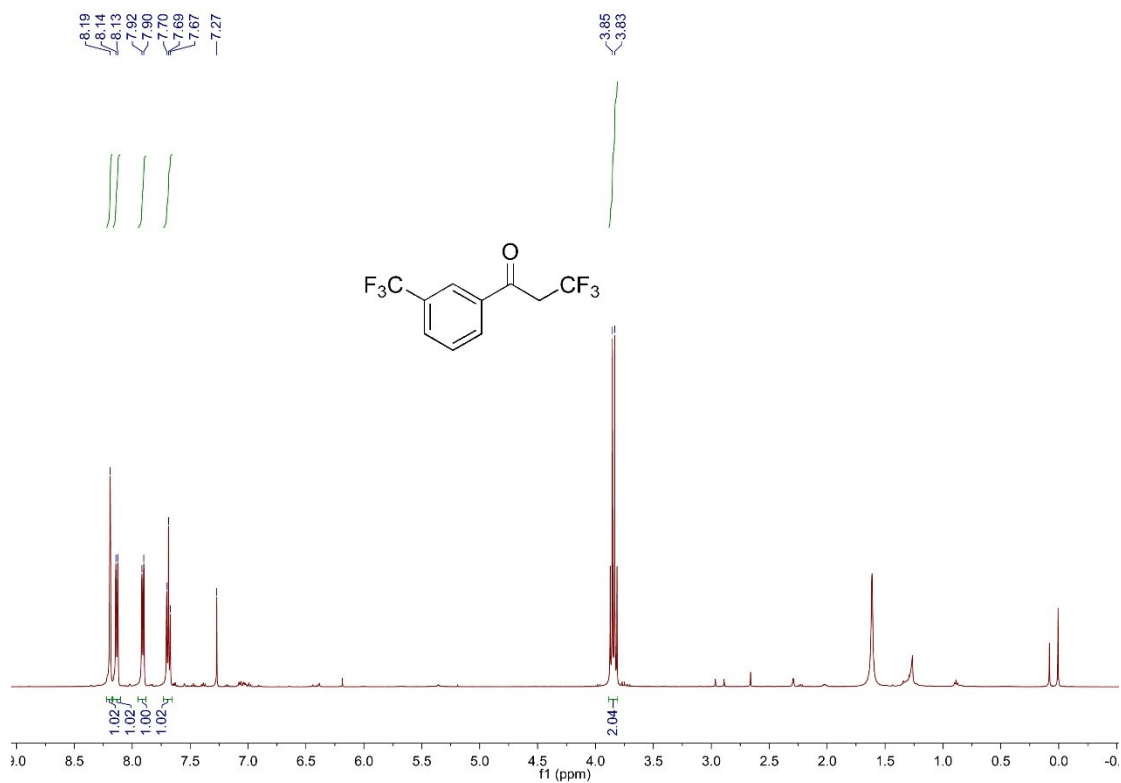




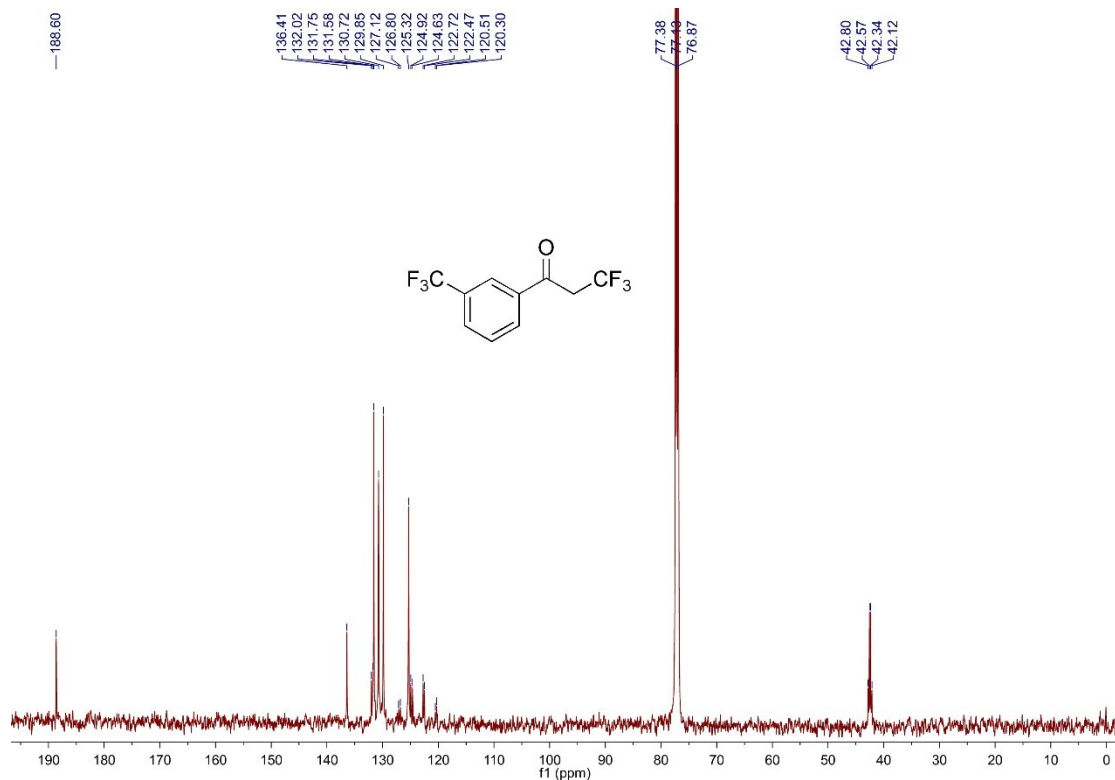
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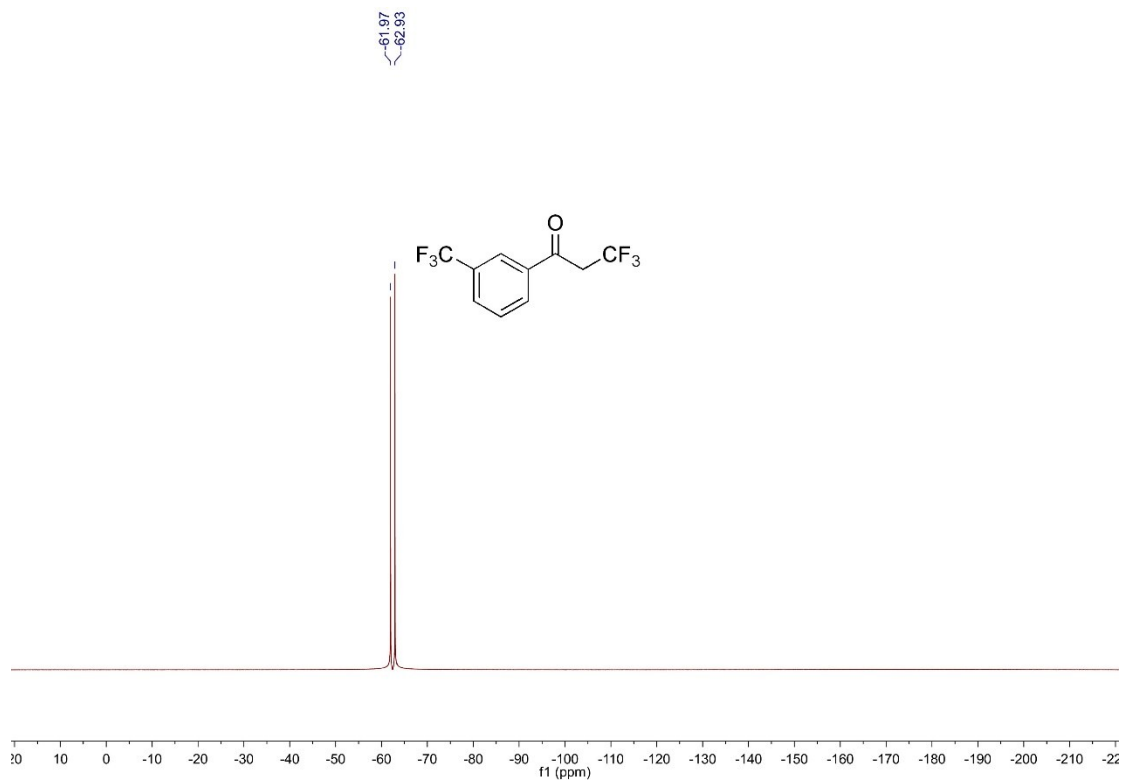
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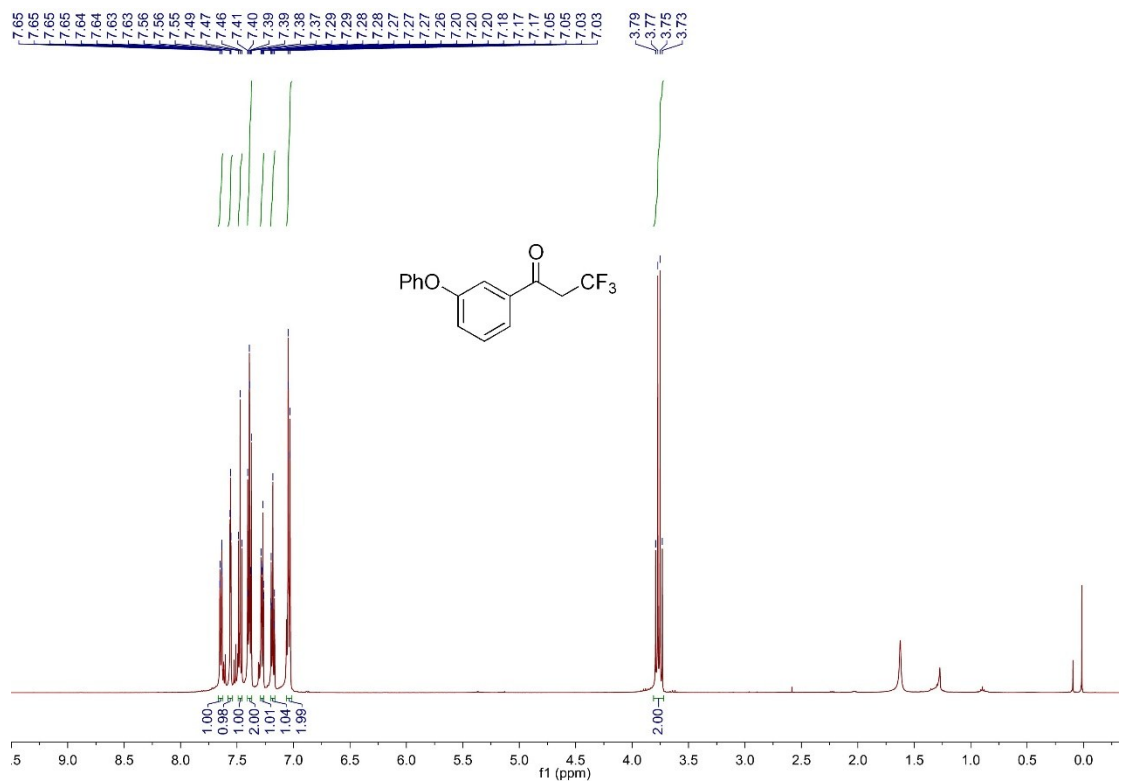
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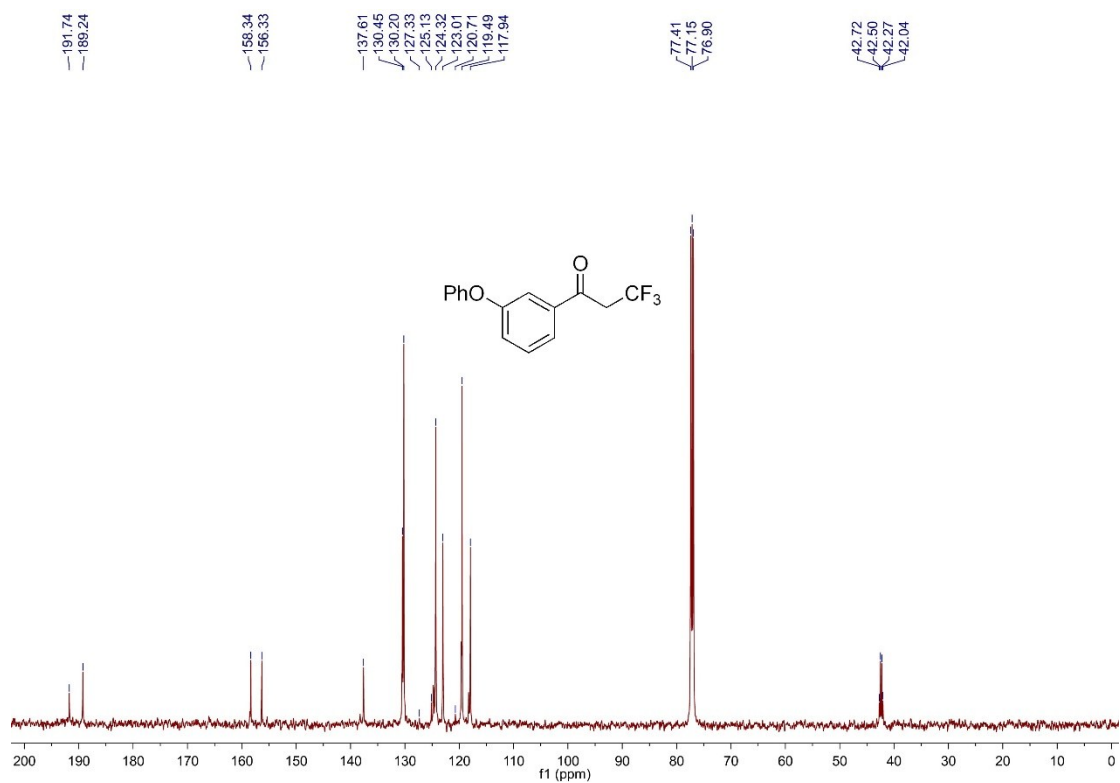
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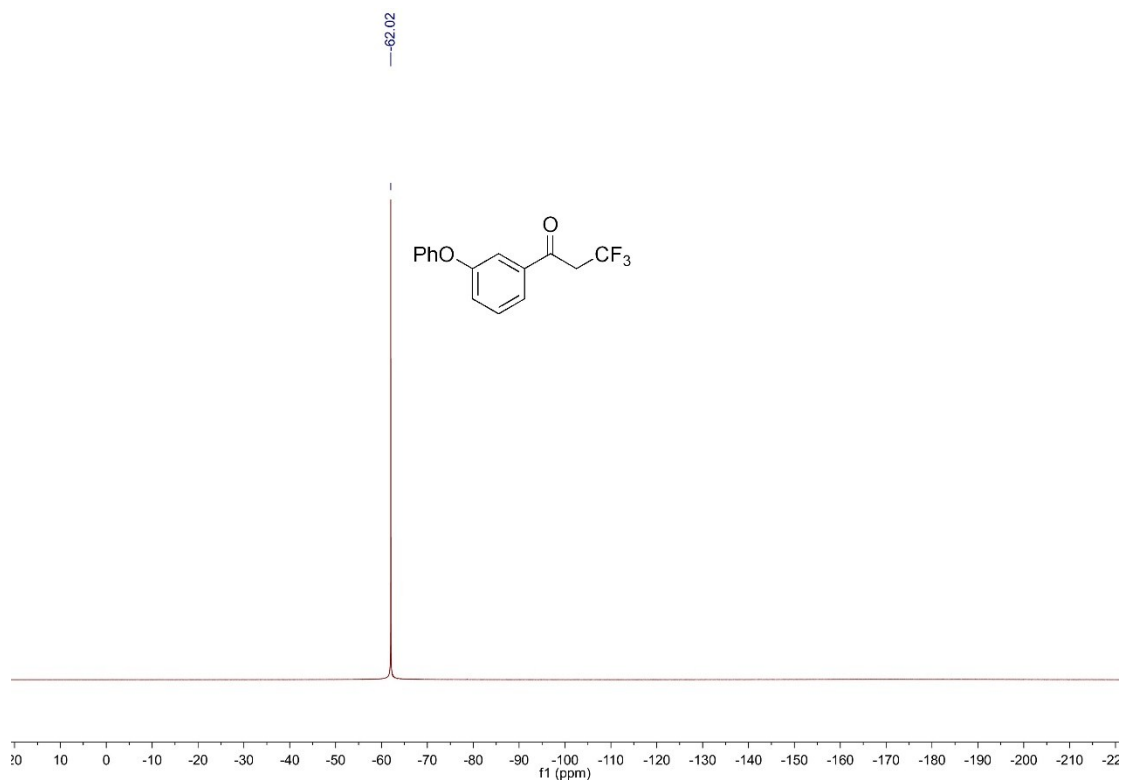
¹⁹F NMR 3l



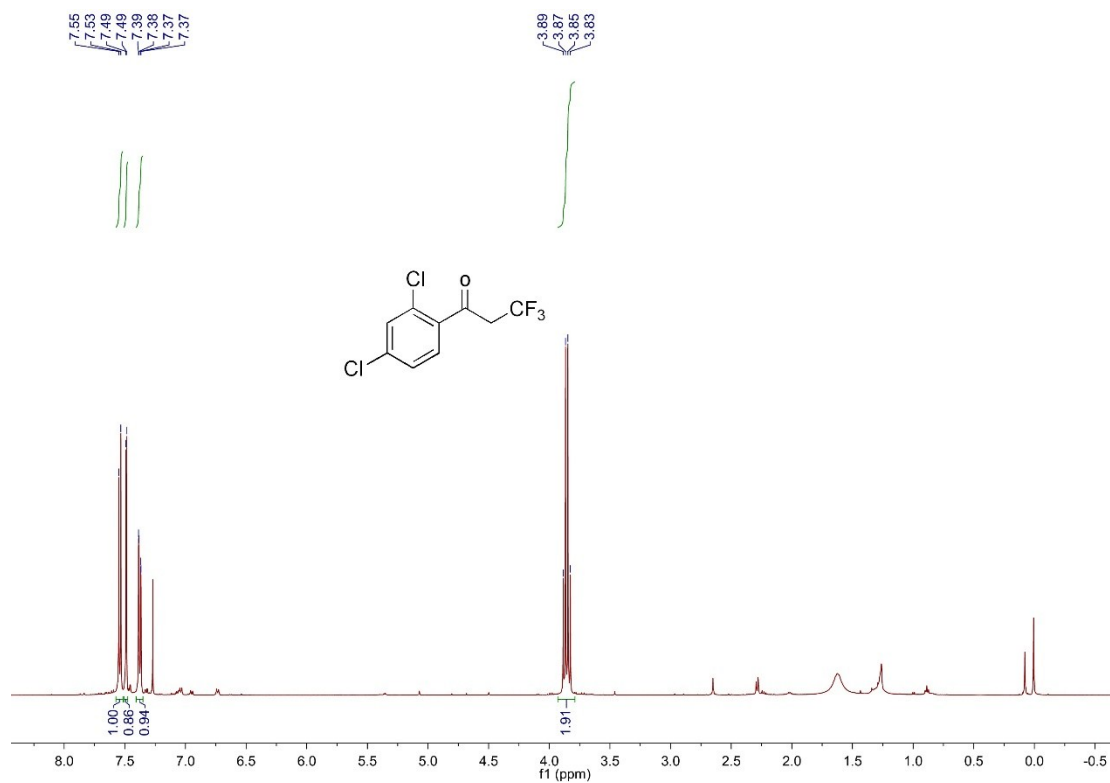
¹H NMR 3m



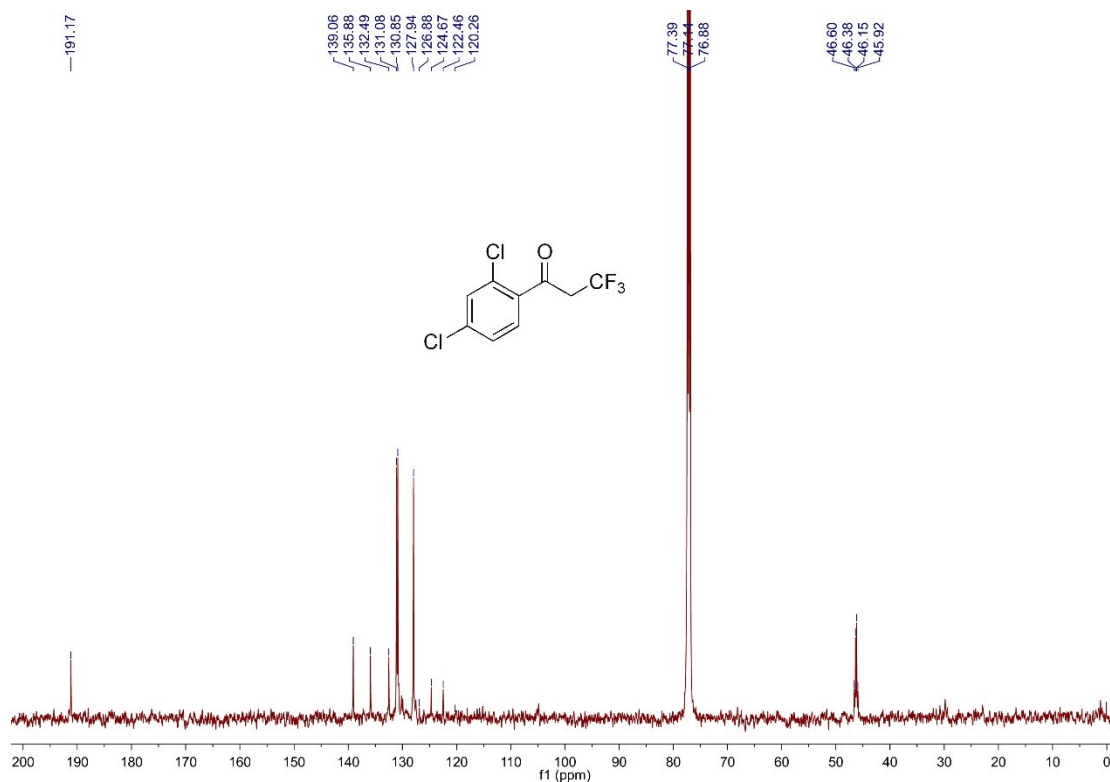
^{13}C NMR **3m**



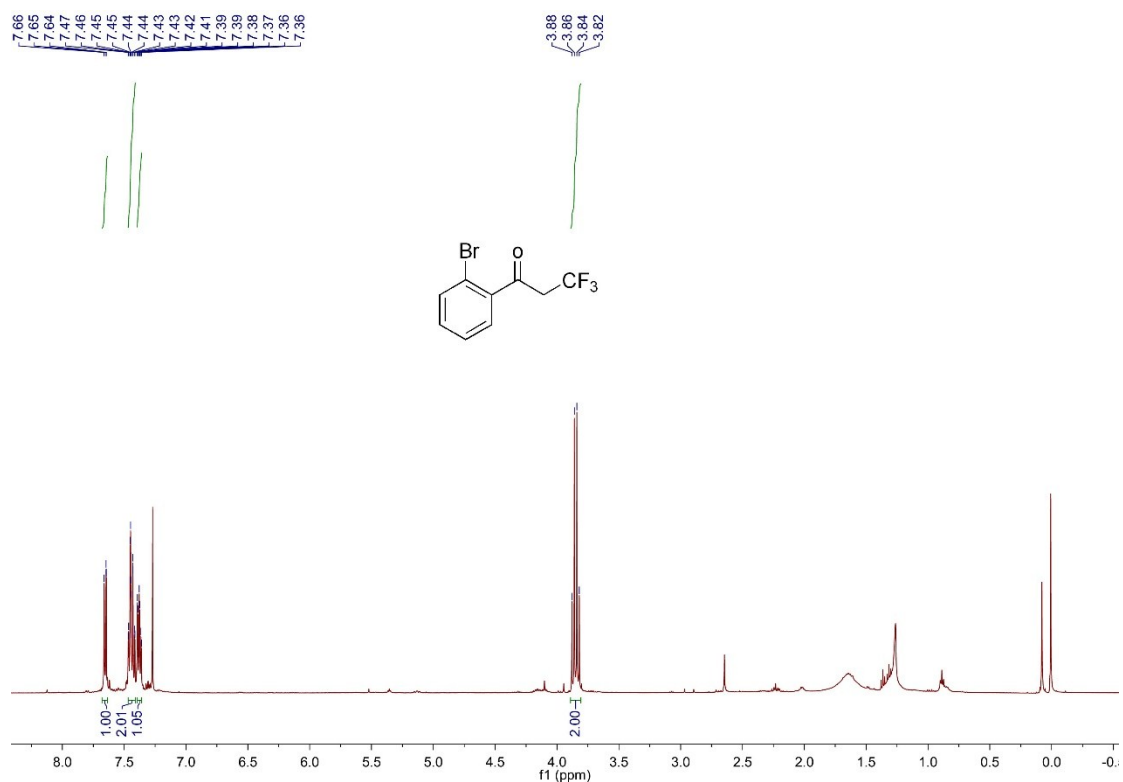
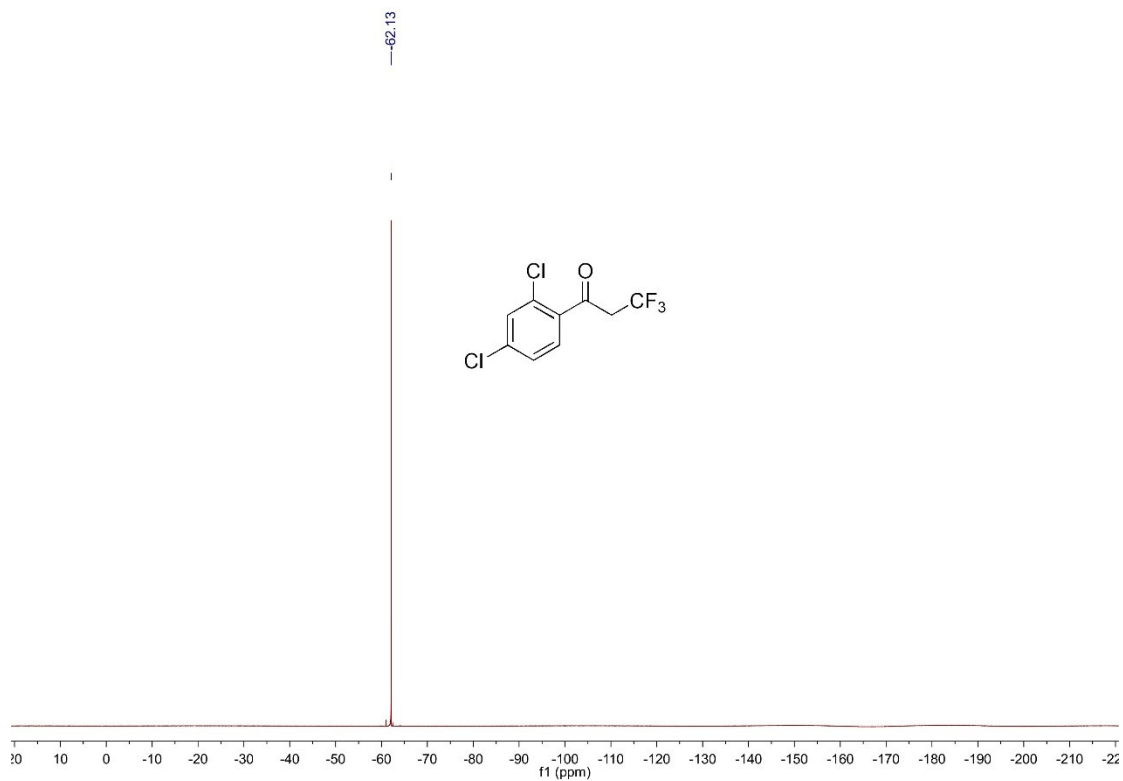
^{19}F NMR **3m**

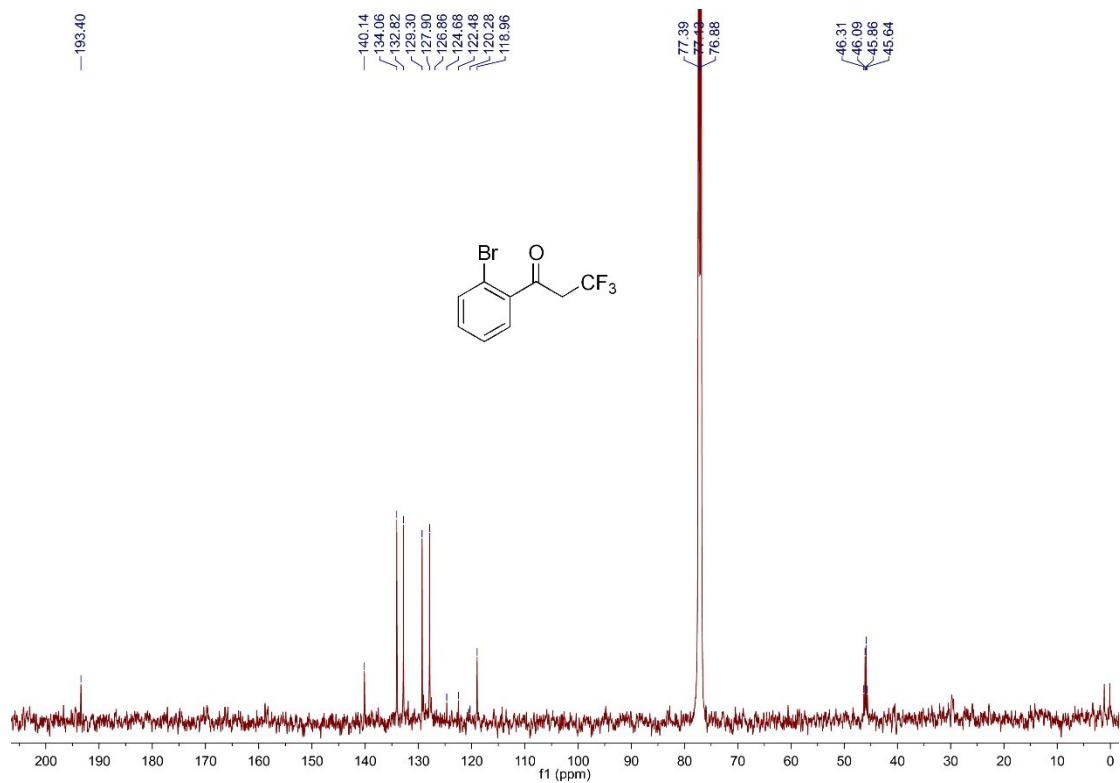


¹H NMR 3n

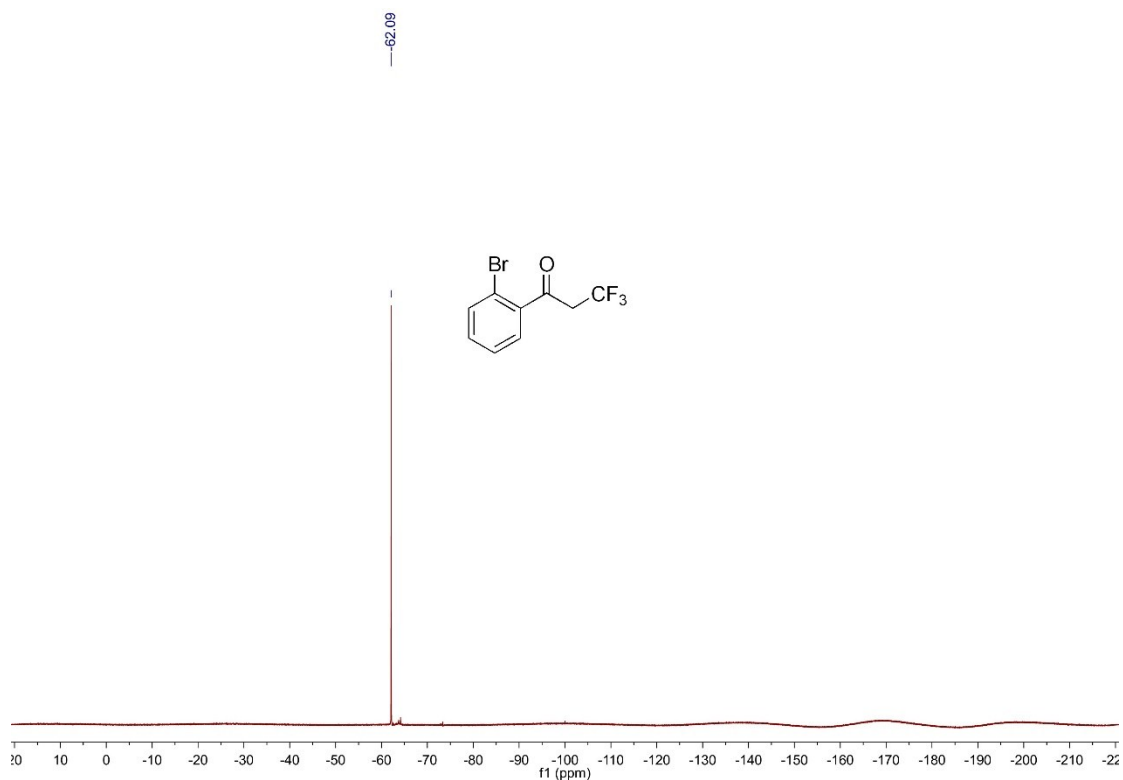


¹³C NMR 3n

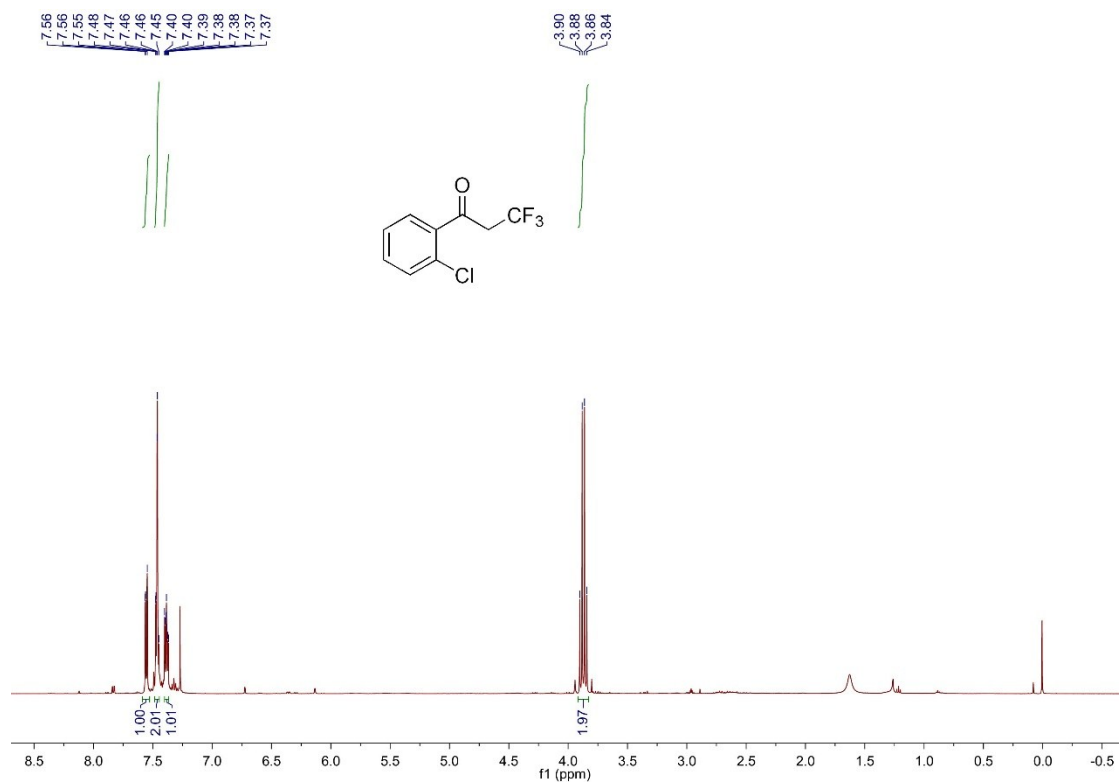




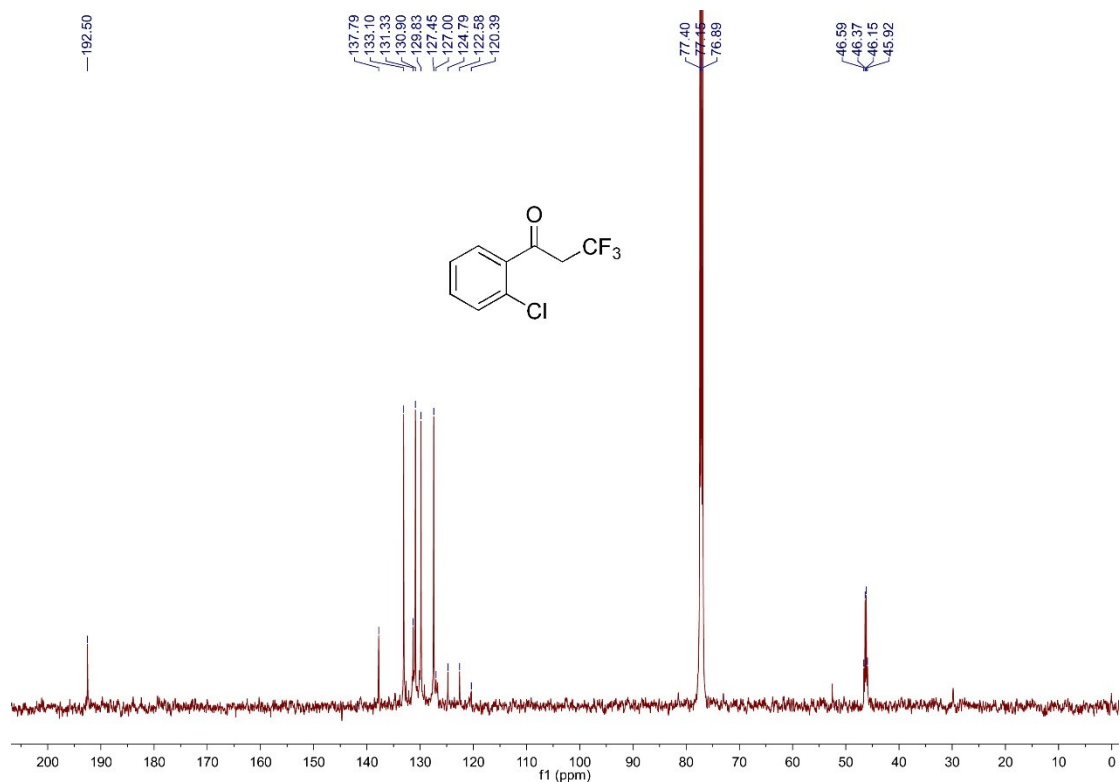
¹³C NMR 3o



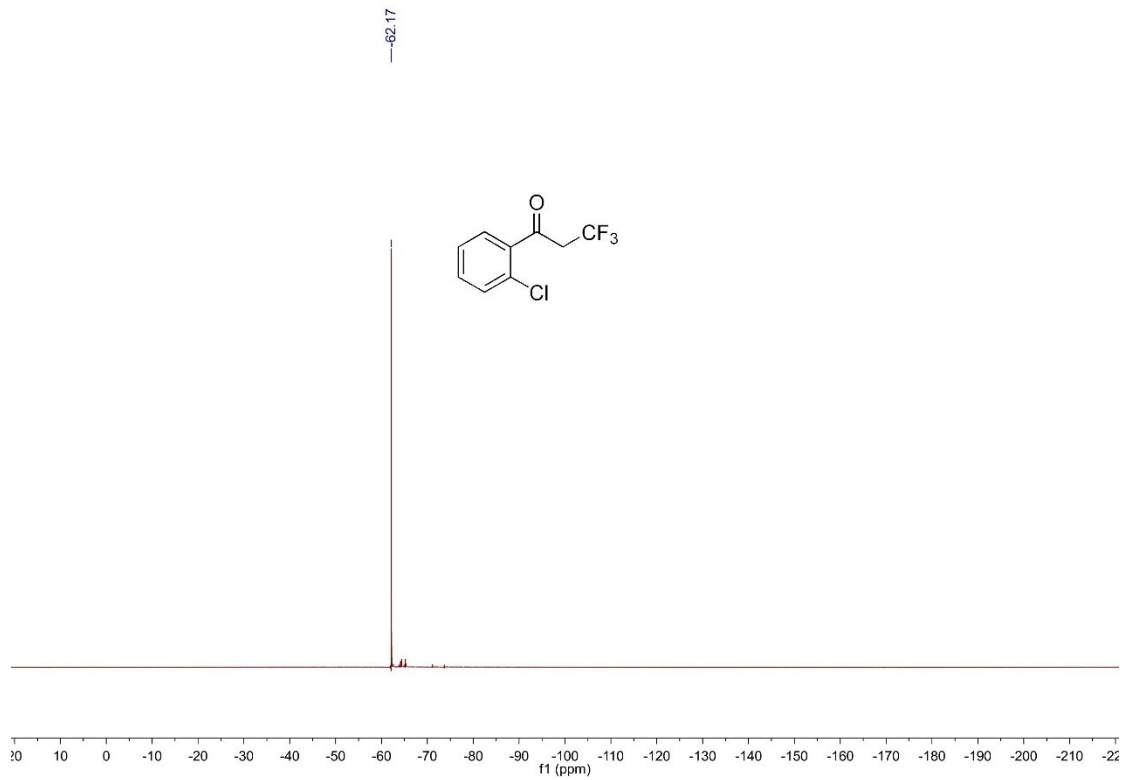
¹⁹F NMR 3o



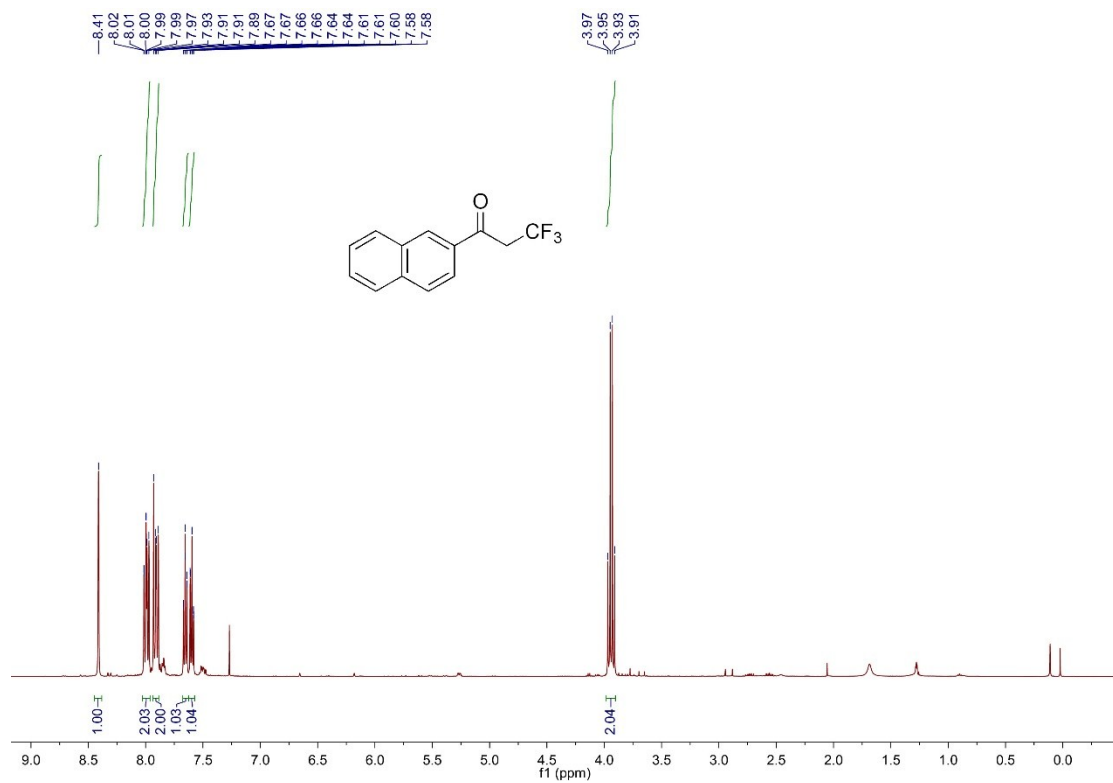
¹H NMR 3p



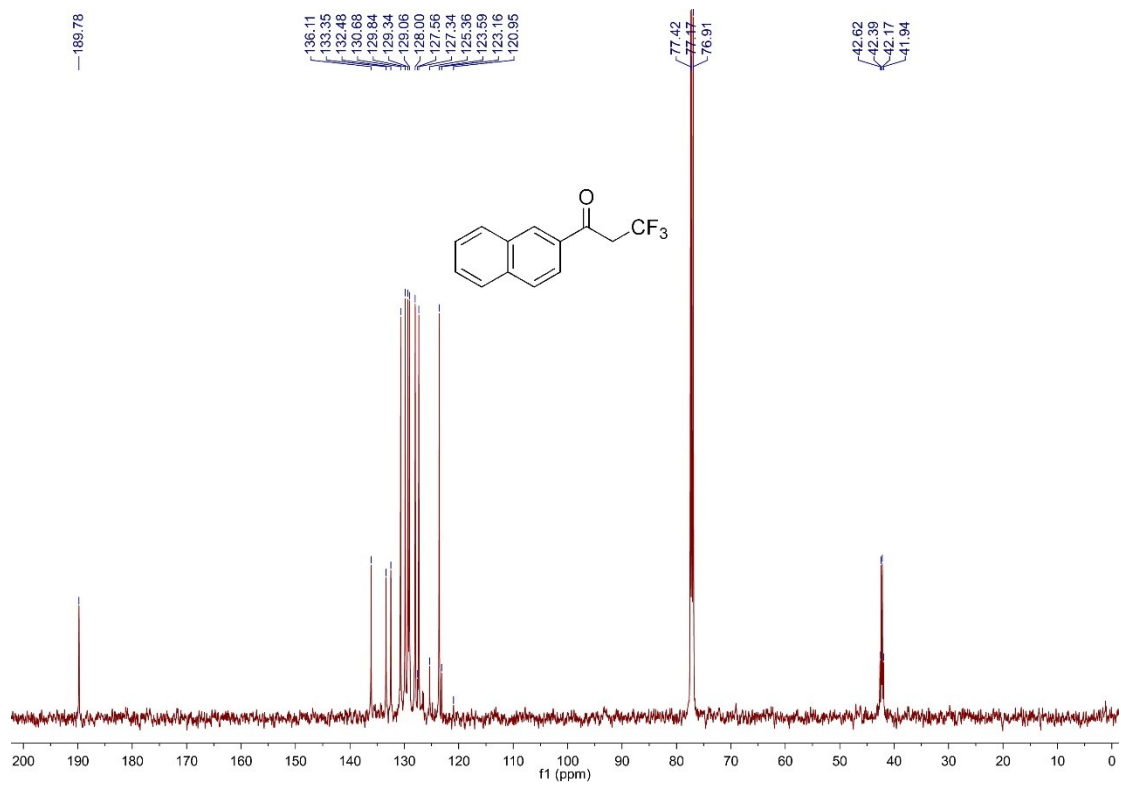
¹³C NMR 3p



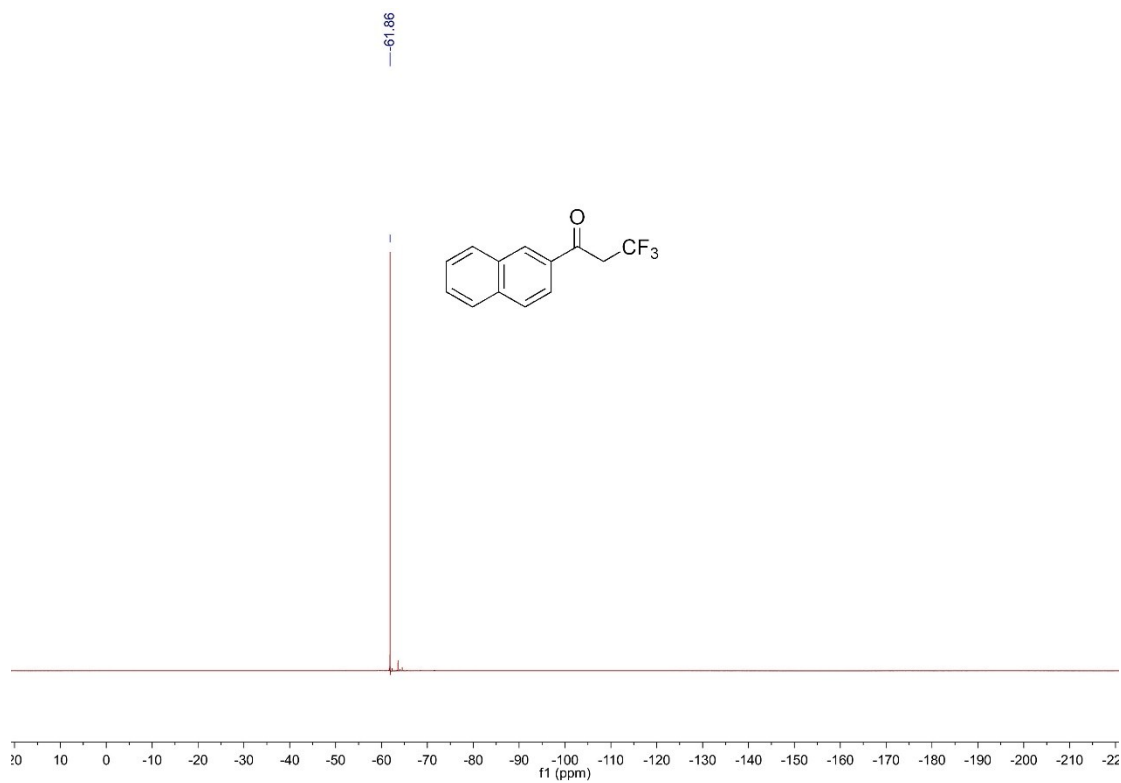
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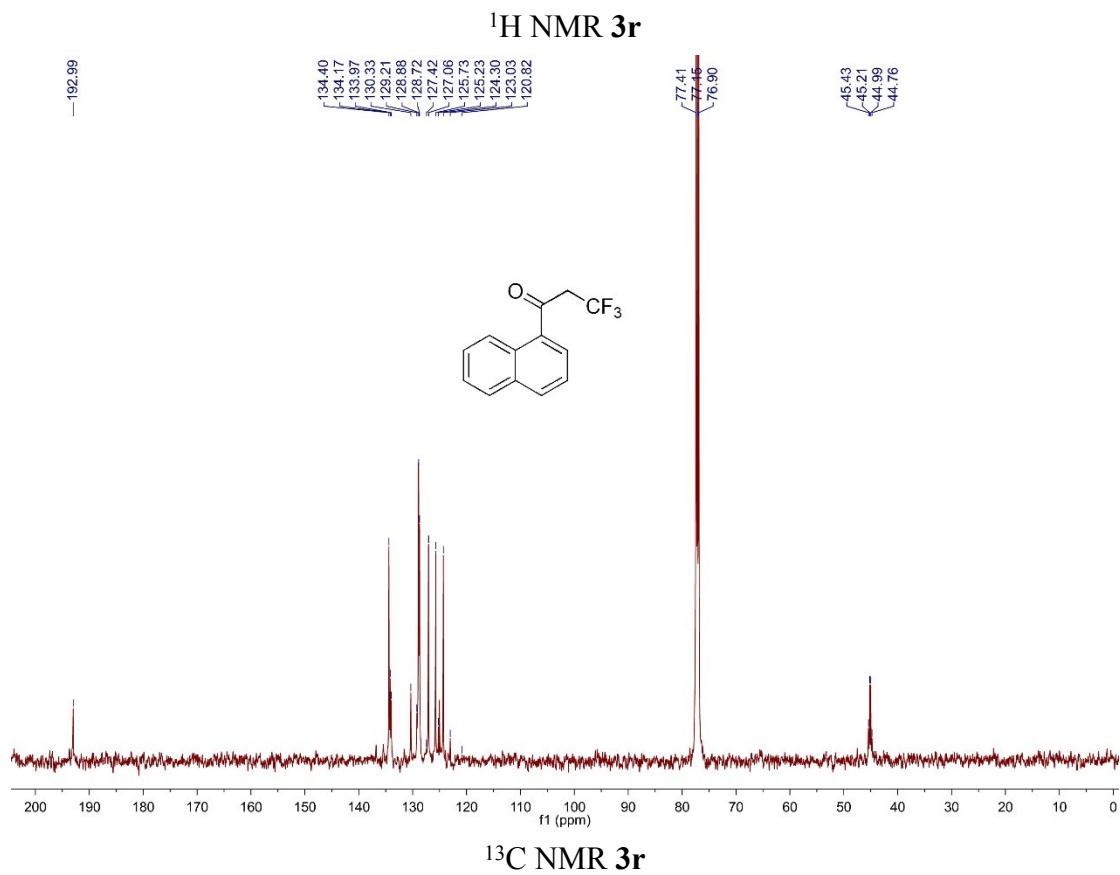
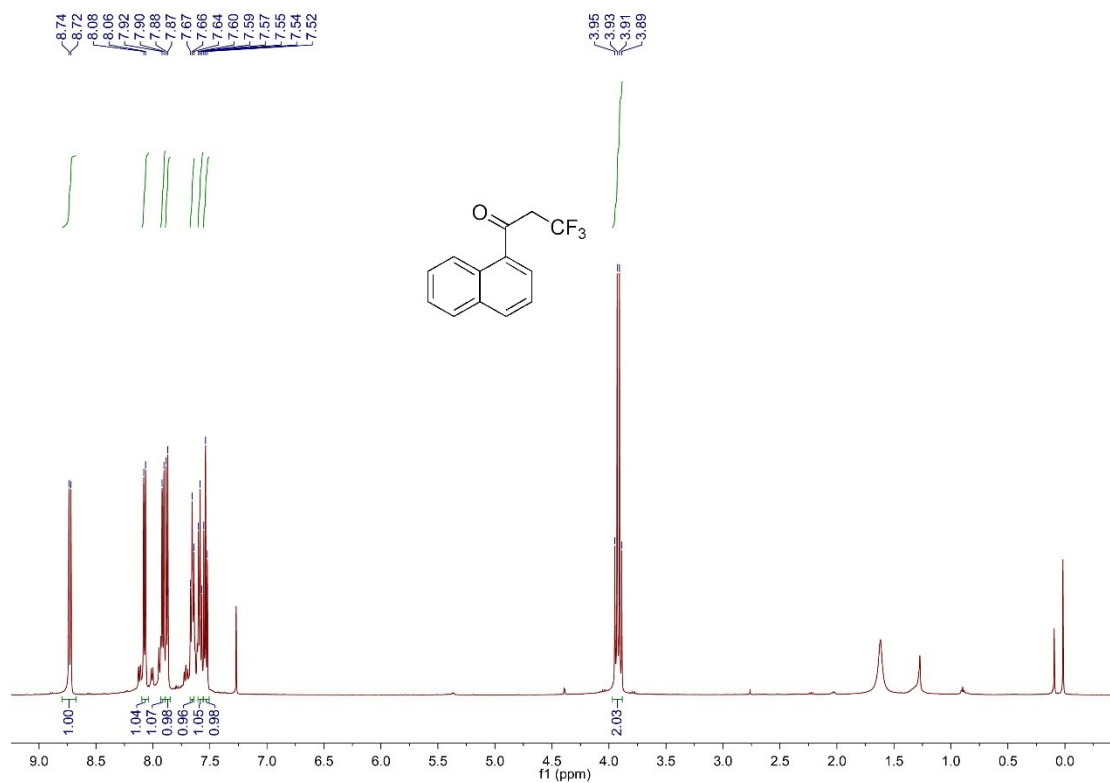
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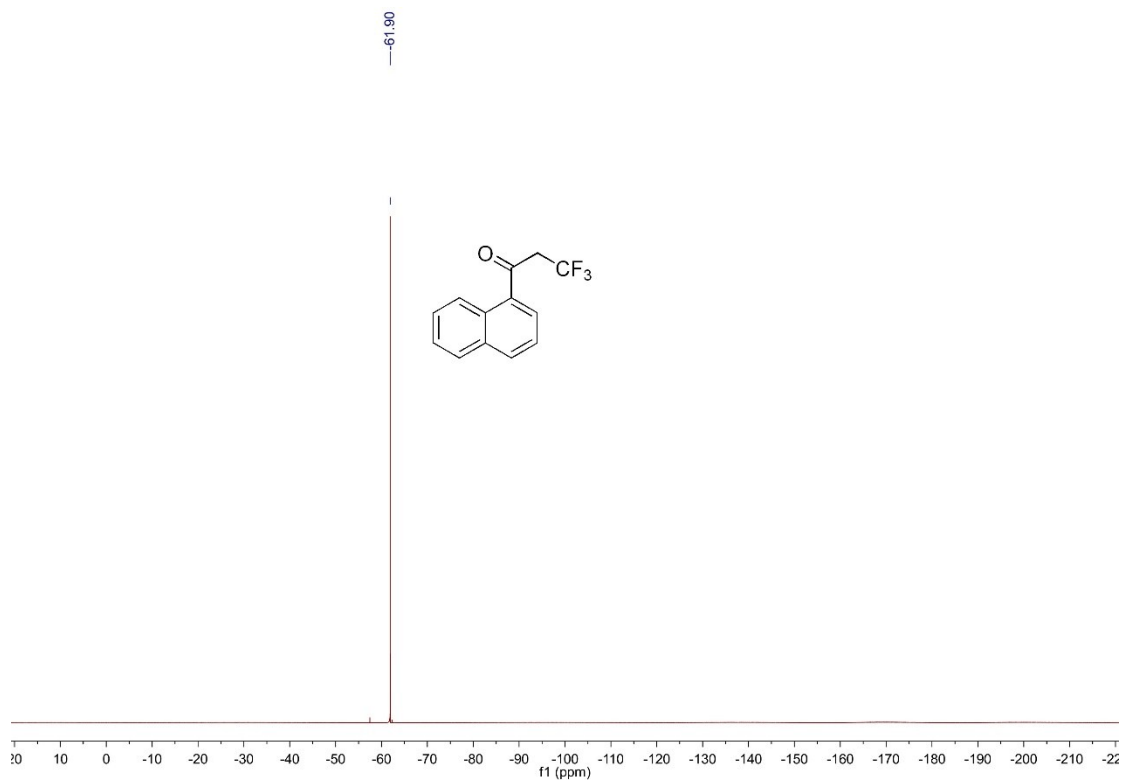


¹³C NMR 3q

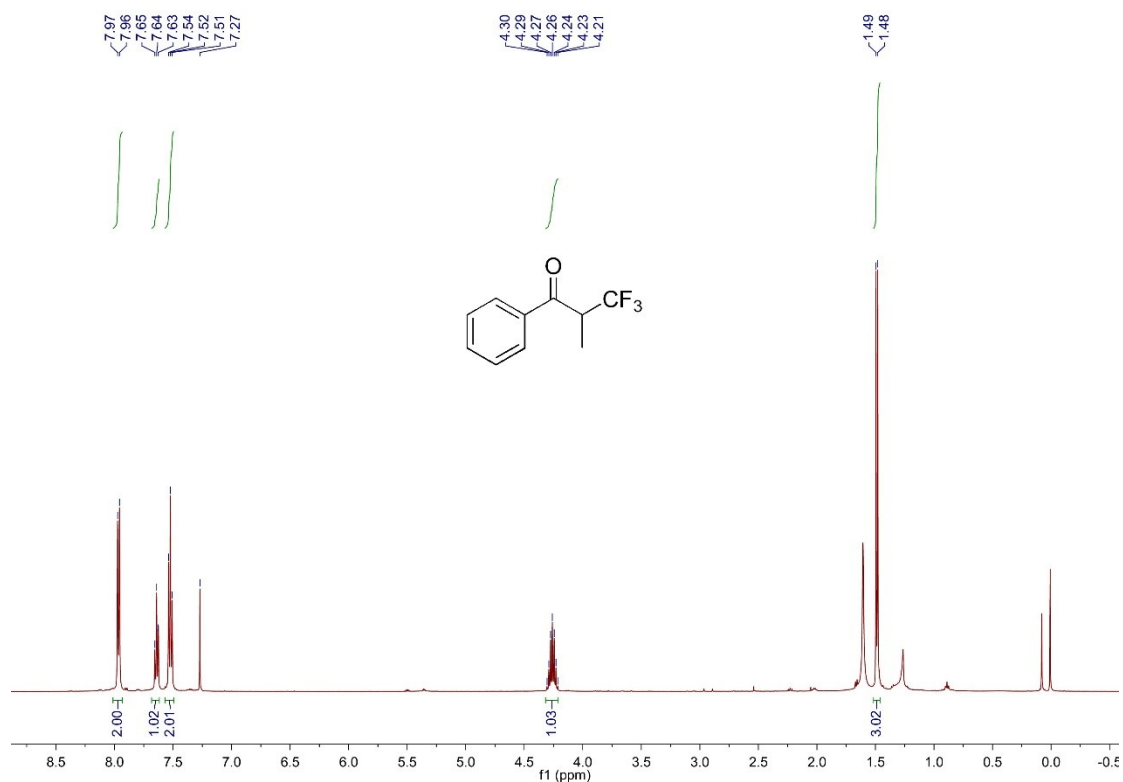


¹⁹F NMR 3q

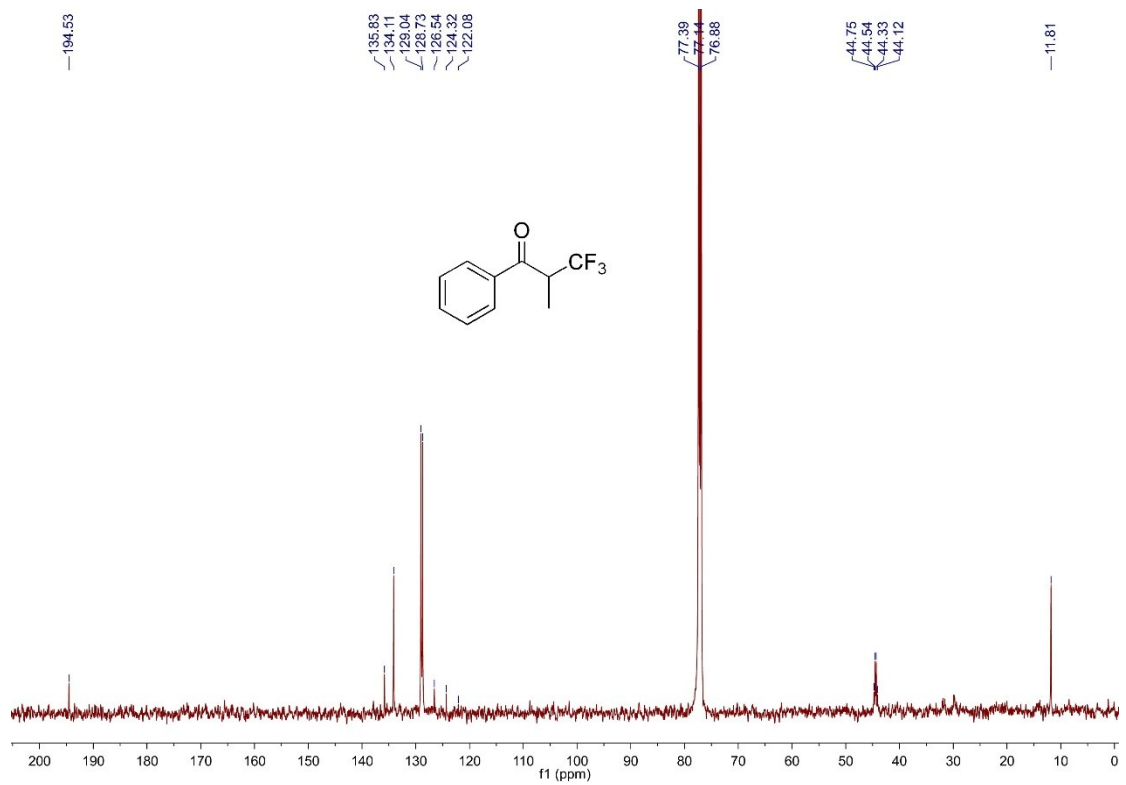




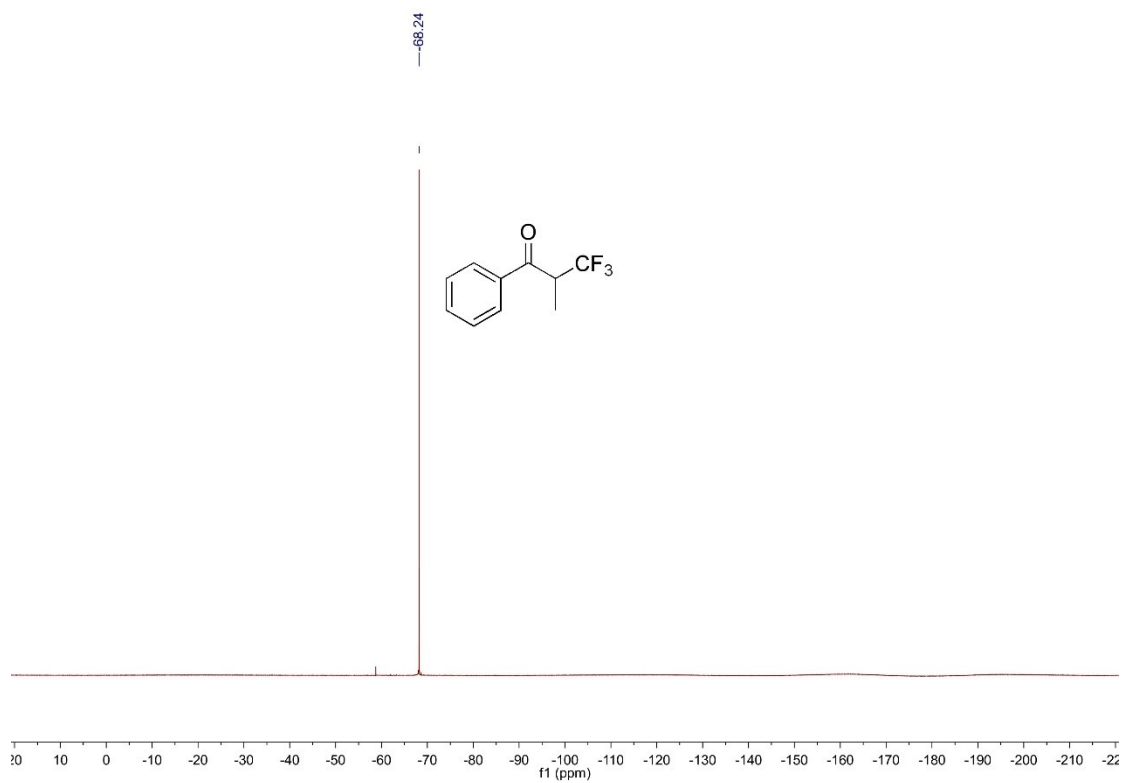
¹⁹F NMR 3r



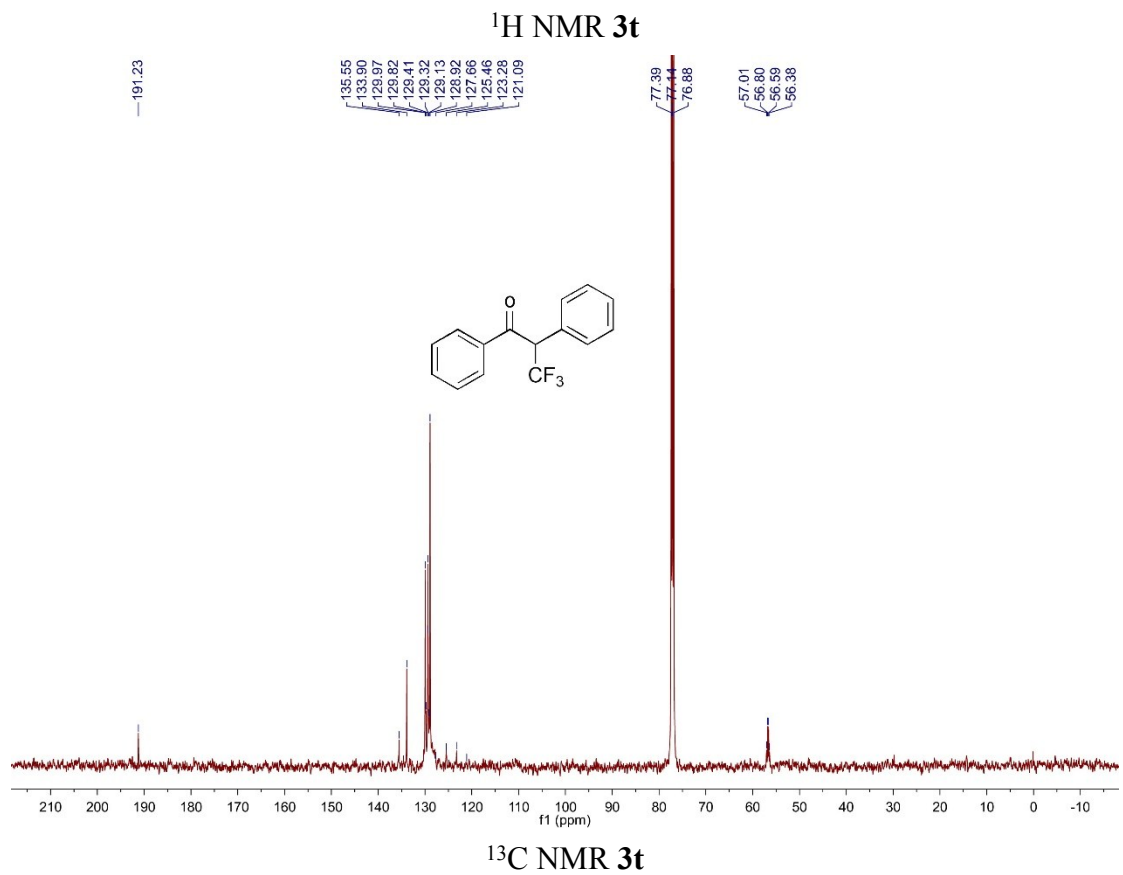
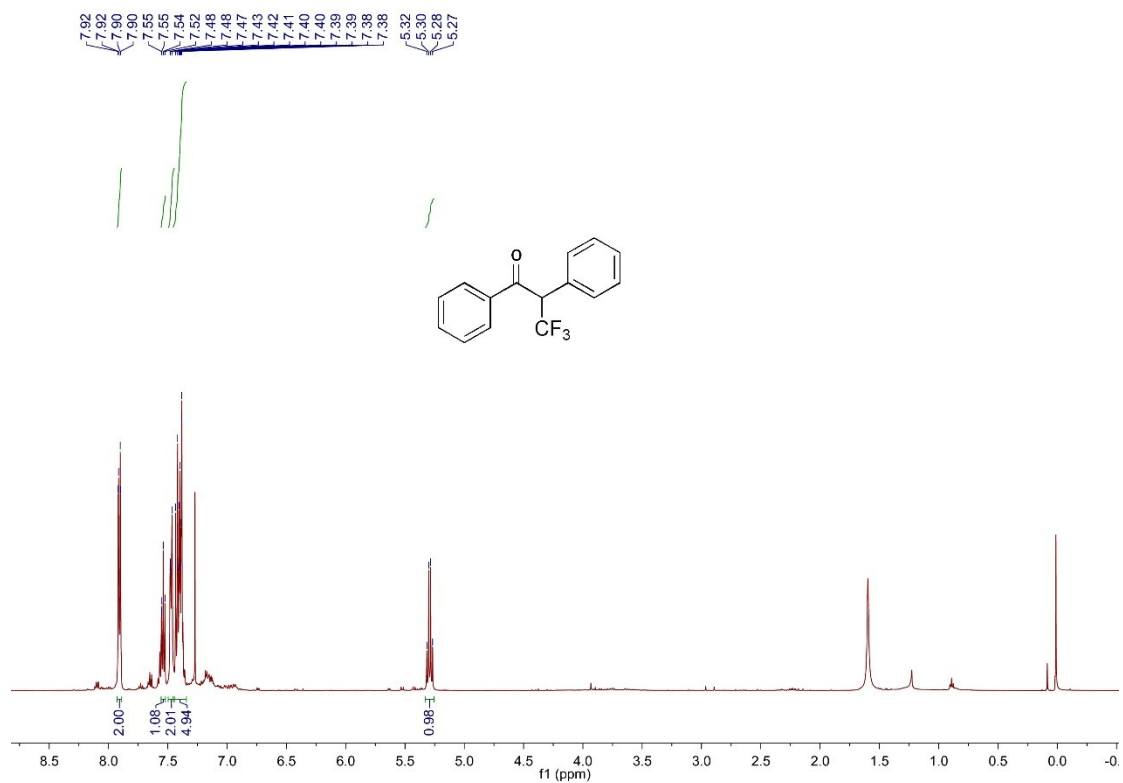
¹H NMR 3s

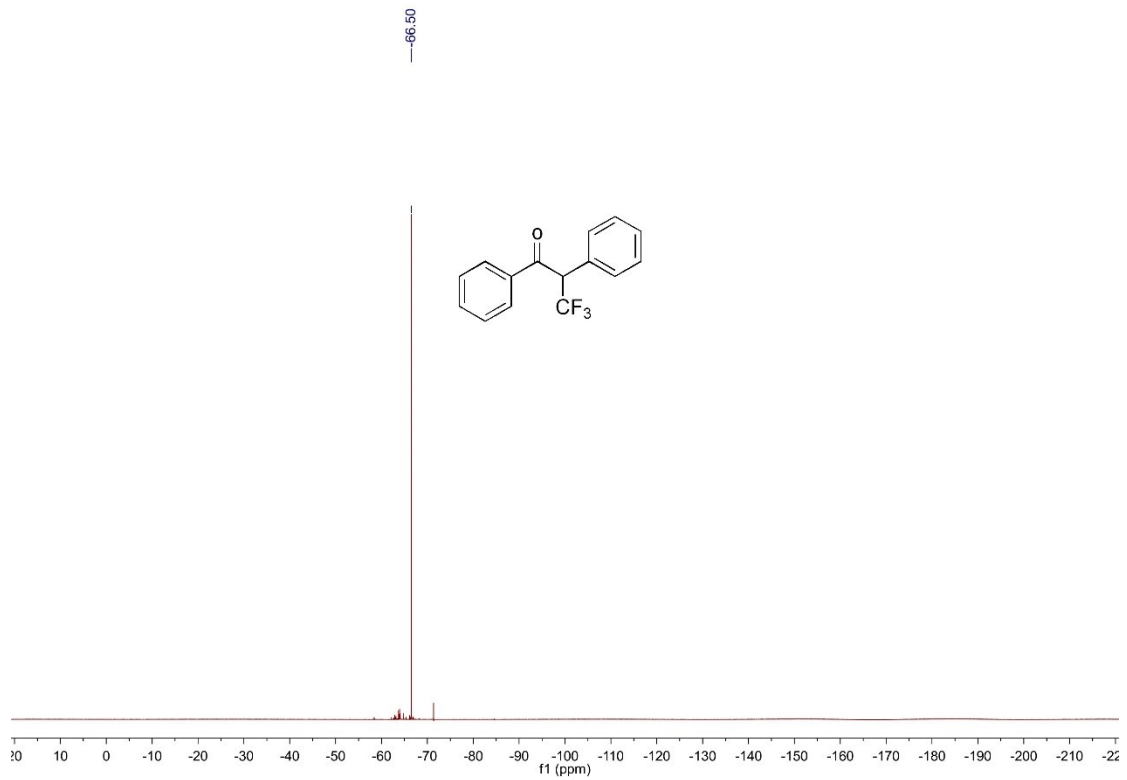


¹³C NMR 3s

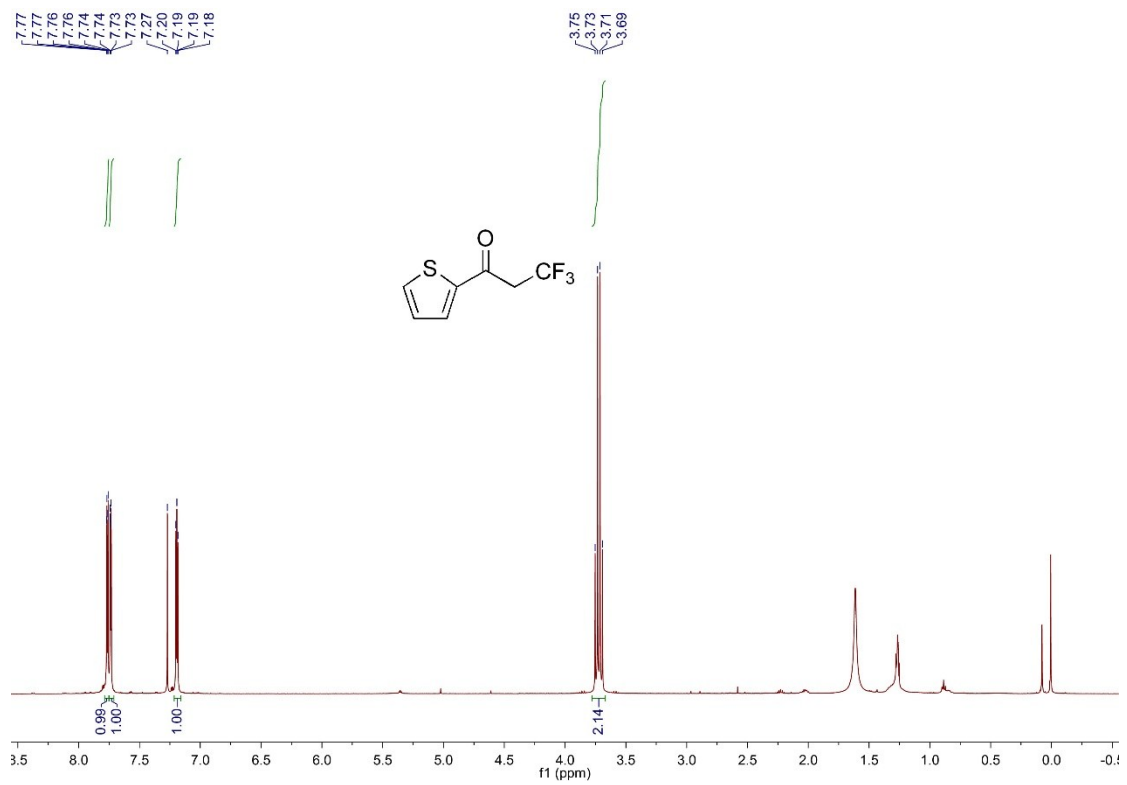


¹⁹F NMR 3s

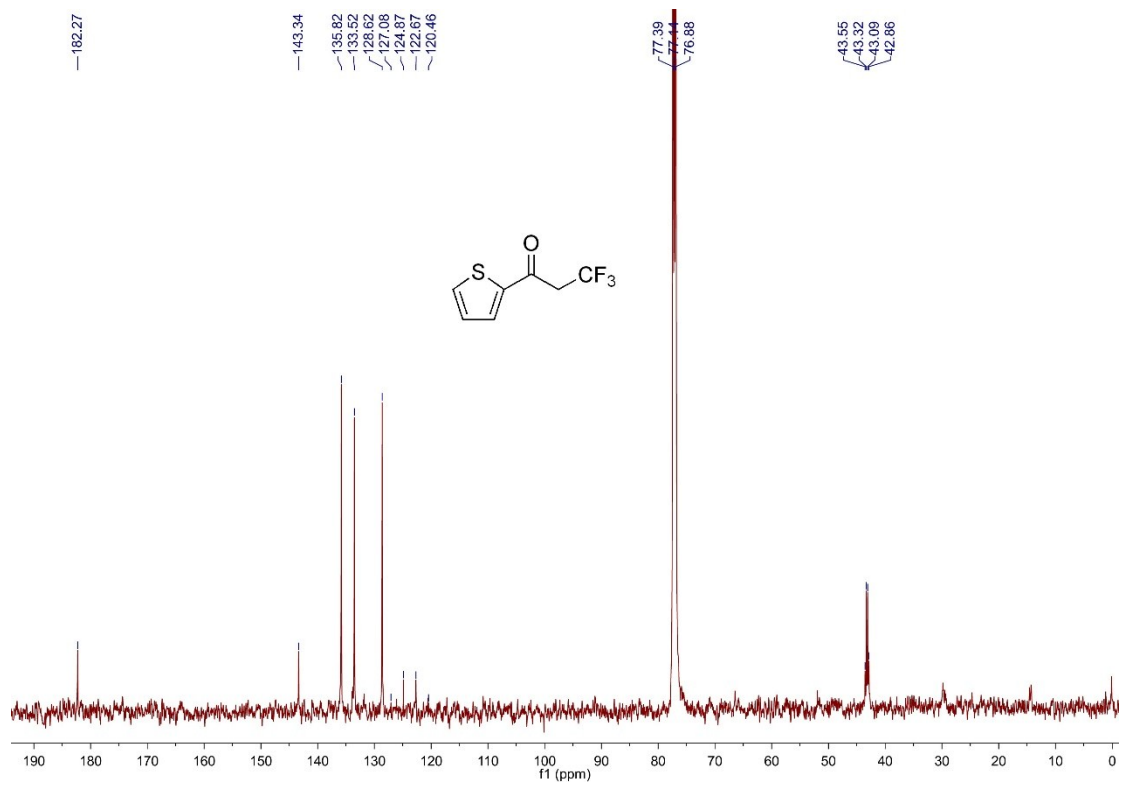




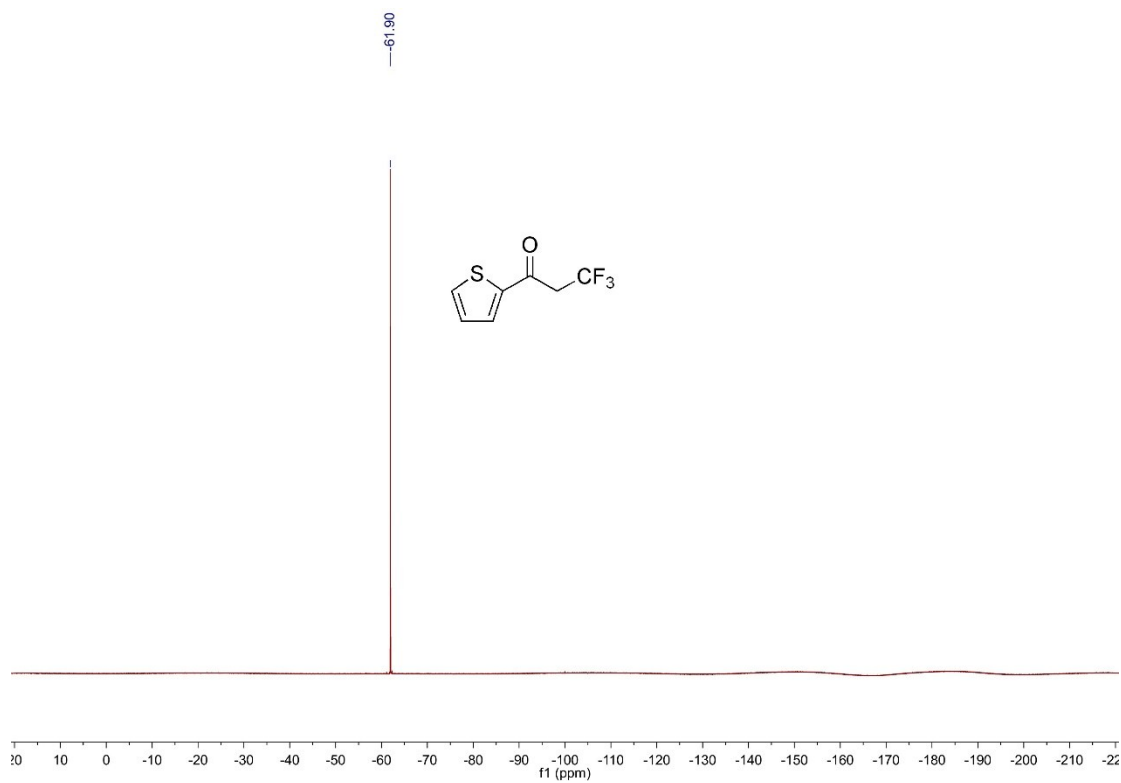
^{19}F NMR 3t



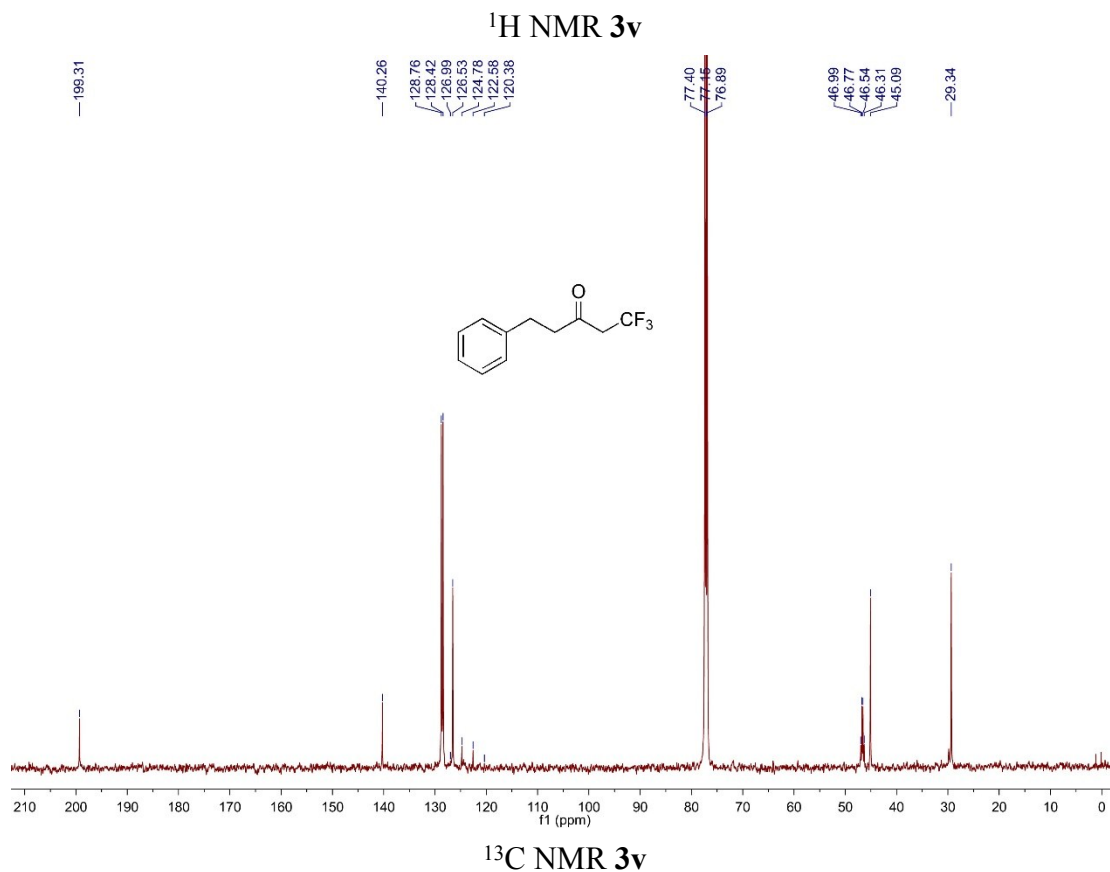
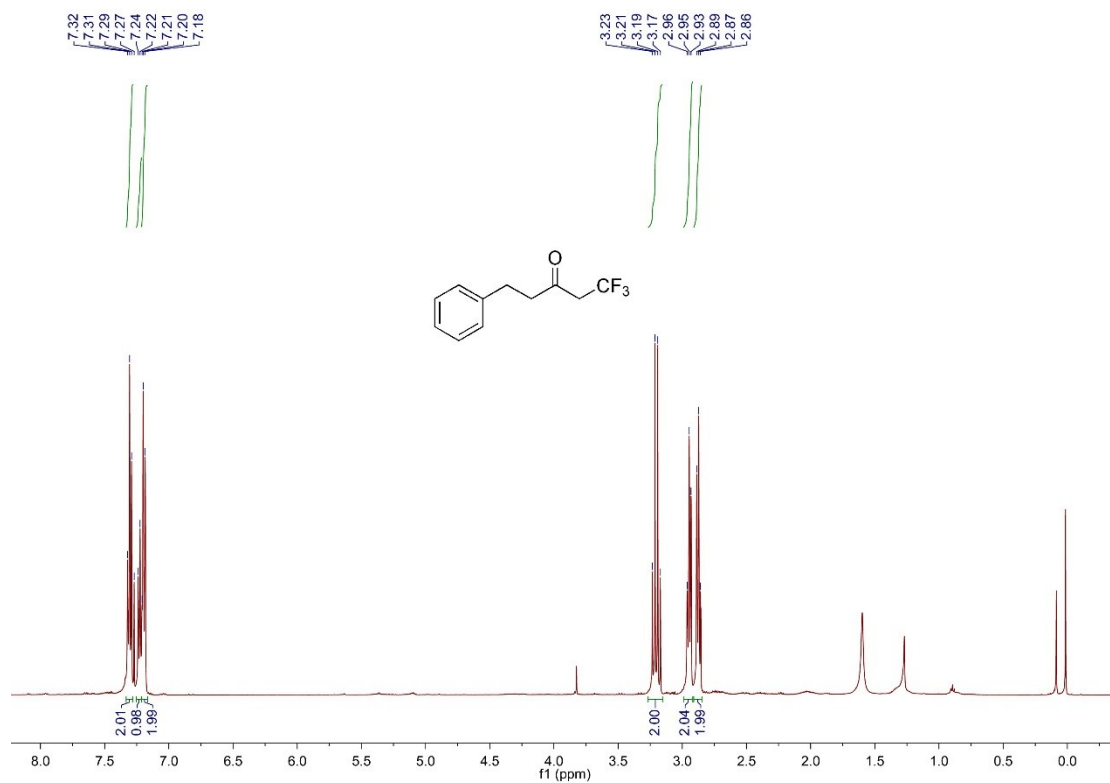
^1H NMR 3u

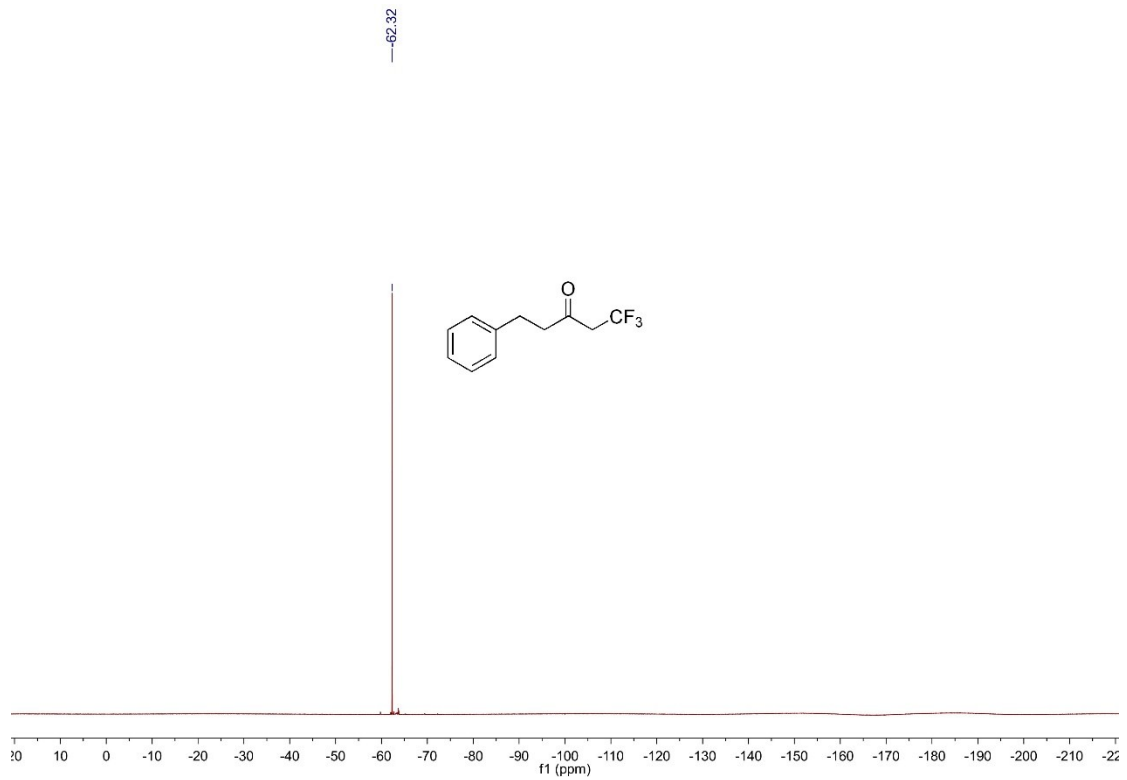


¹³C NMR **3u**

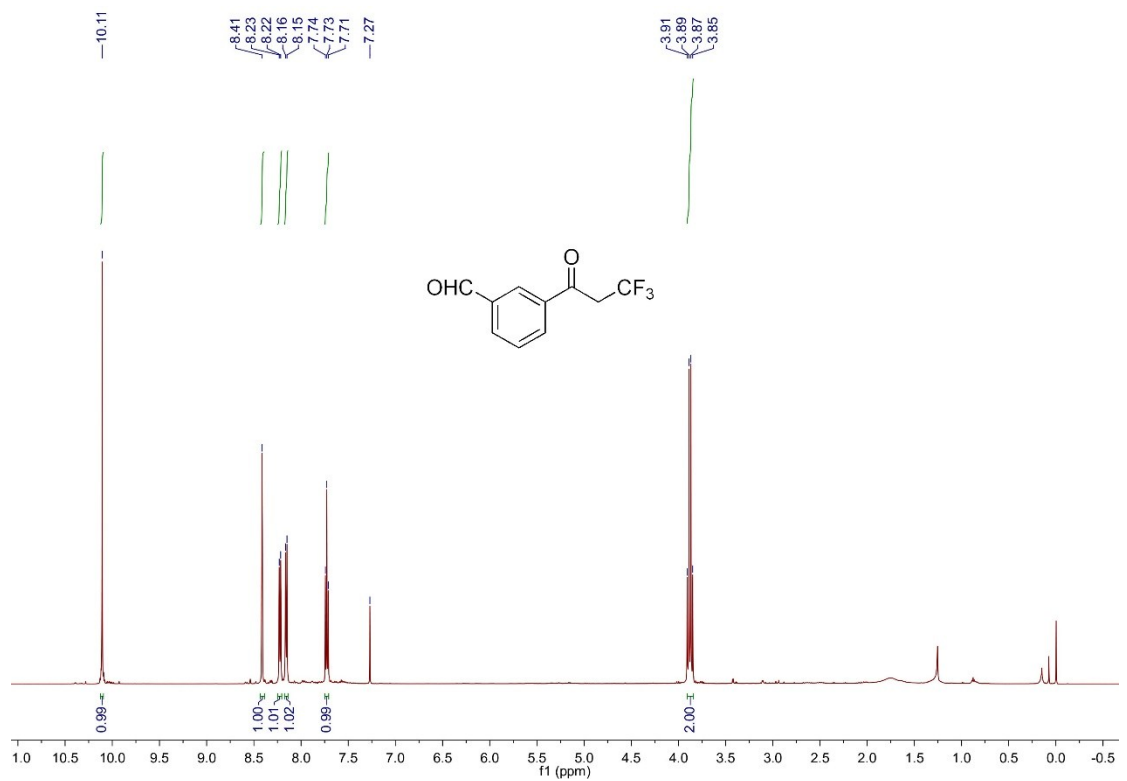


¹⁹F NMR **3u**

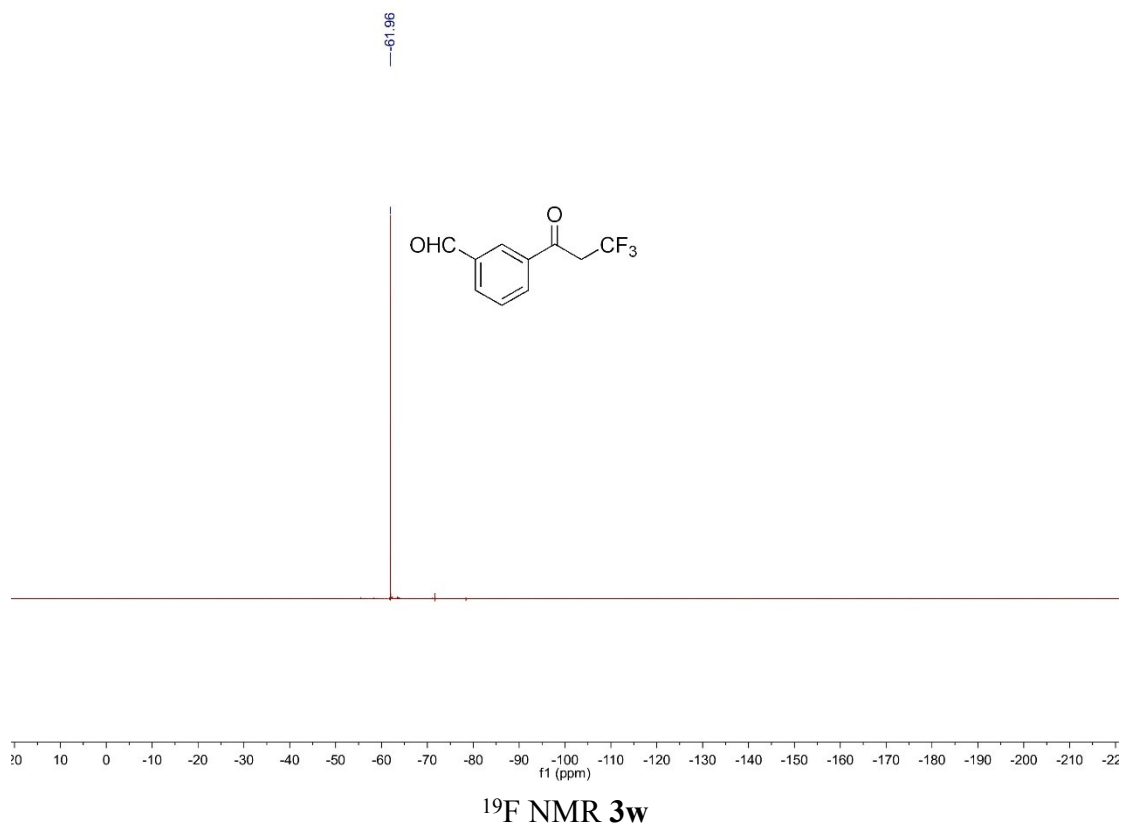
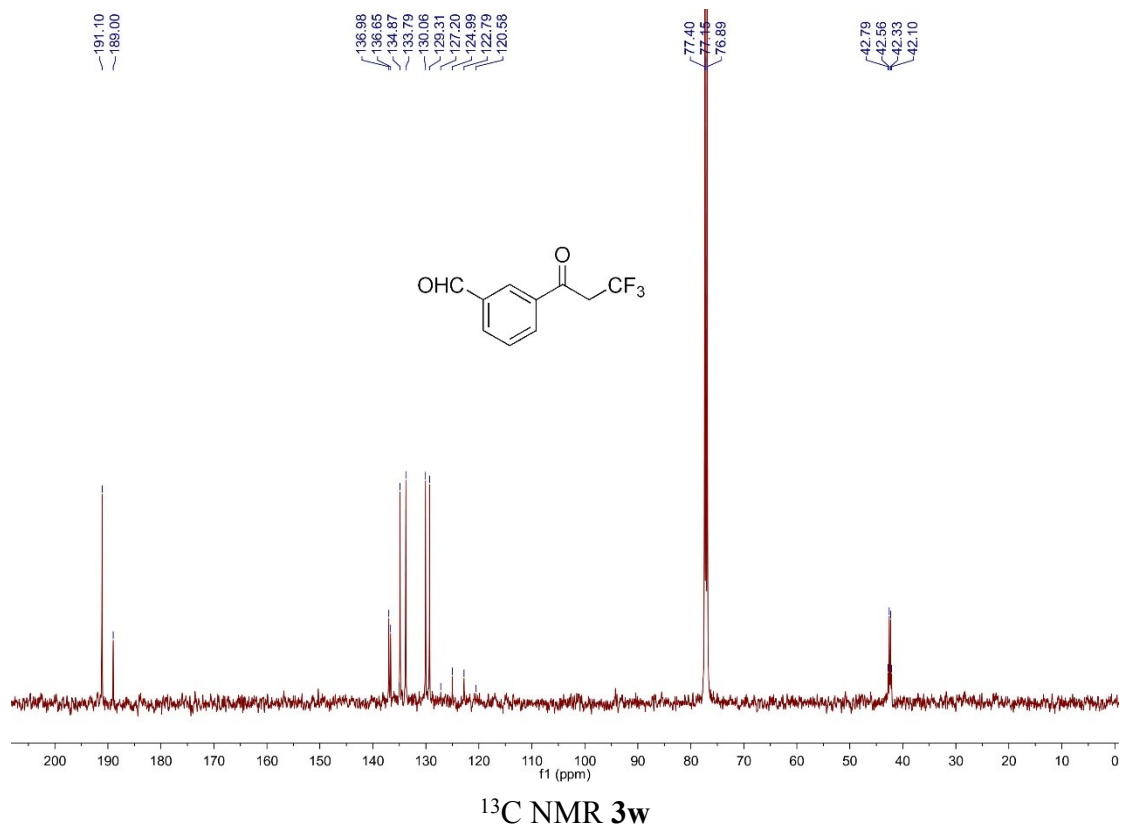


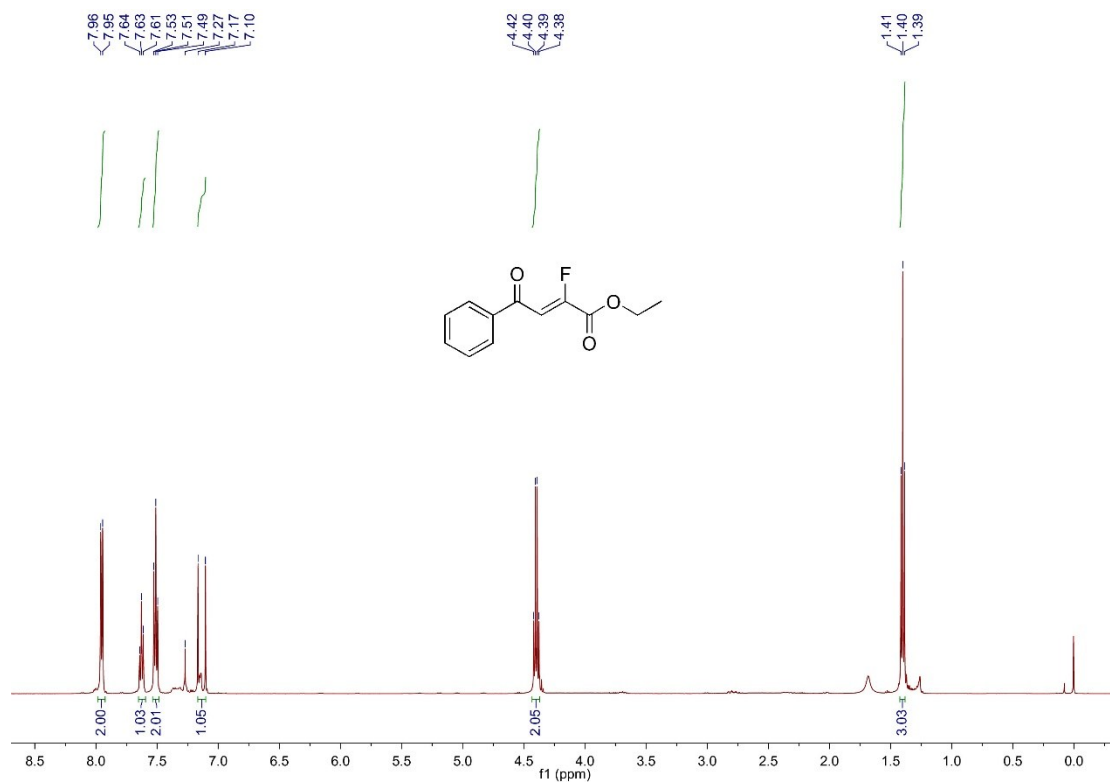


¹⁹F NMR **3v**

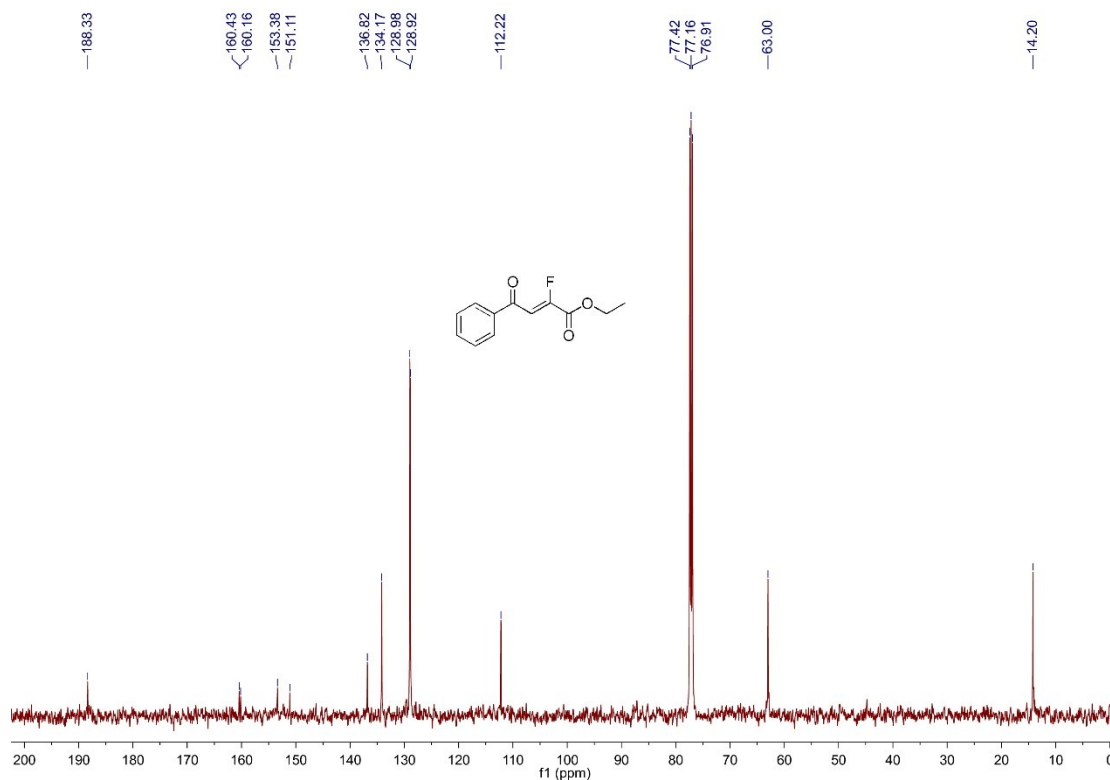


¹H NMR **3w**

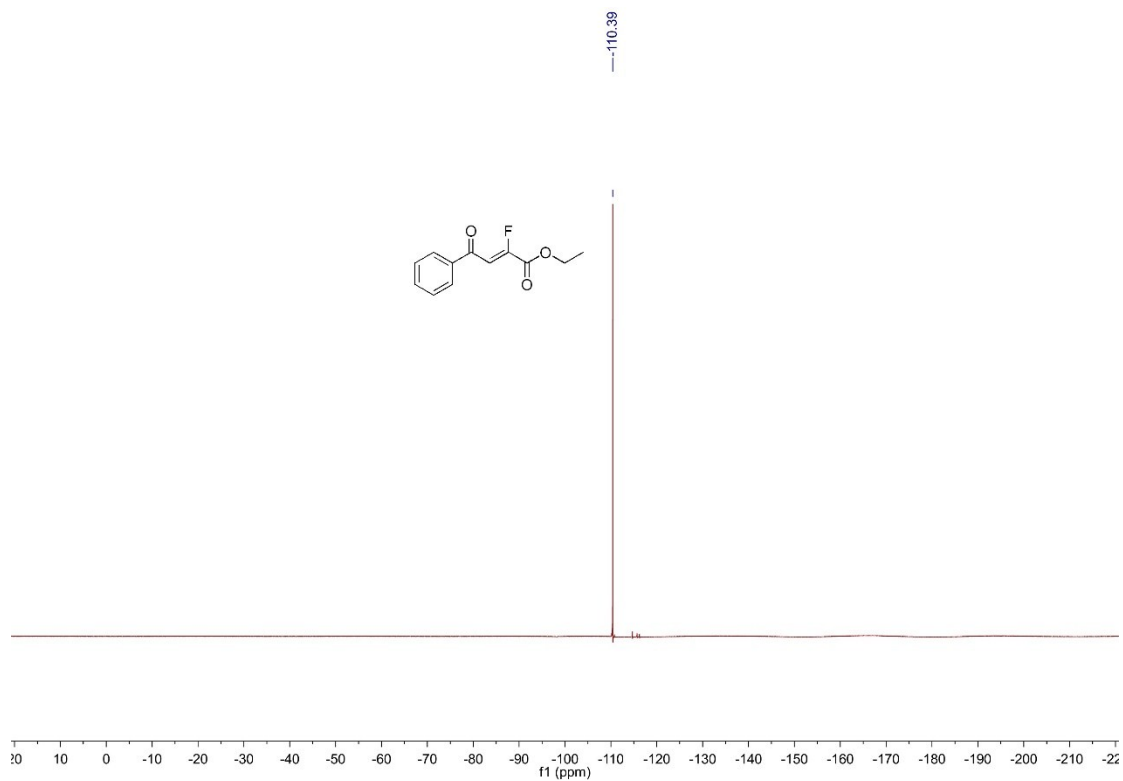




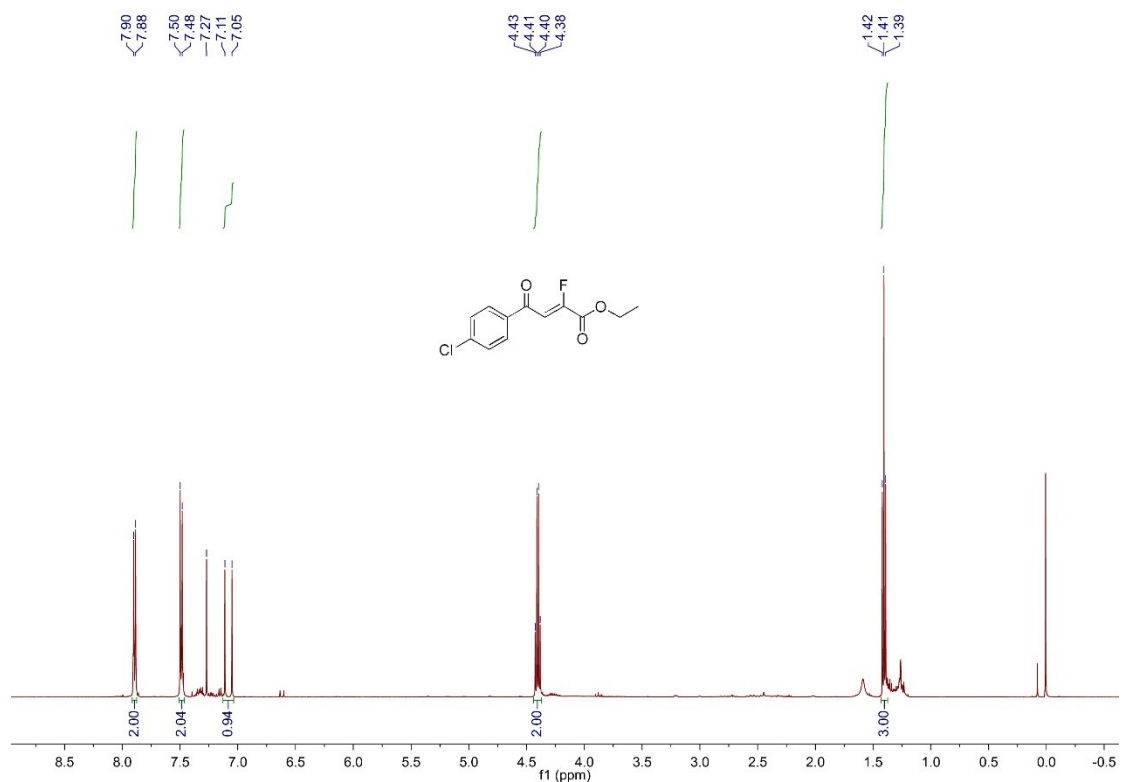
$^1\text{H NMR}$ 4a



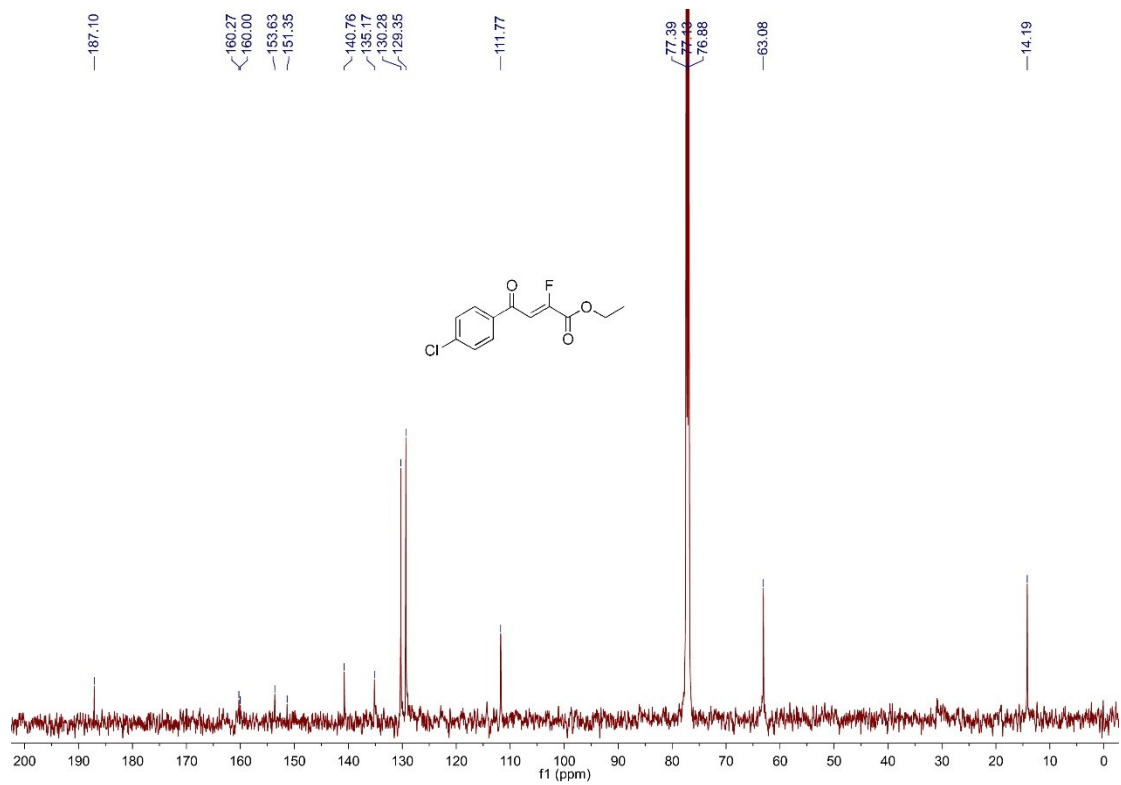
$^{13}\text{C NMR}$ 4a



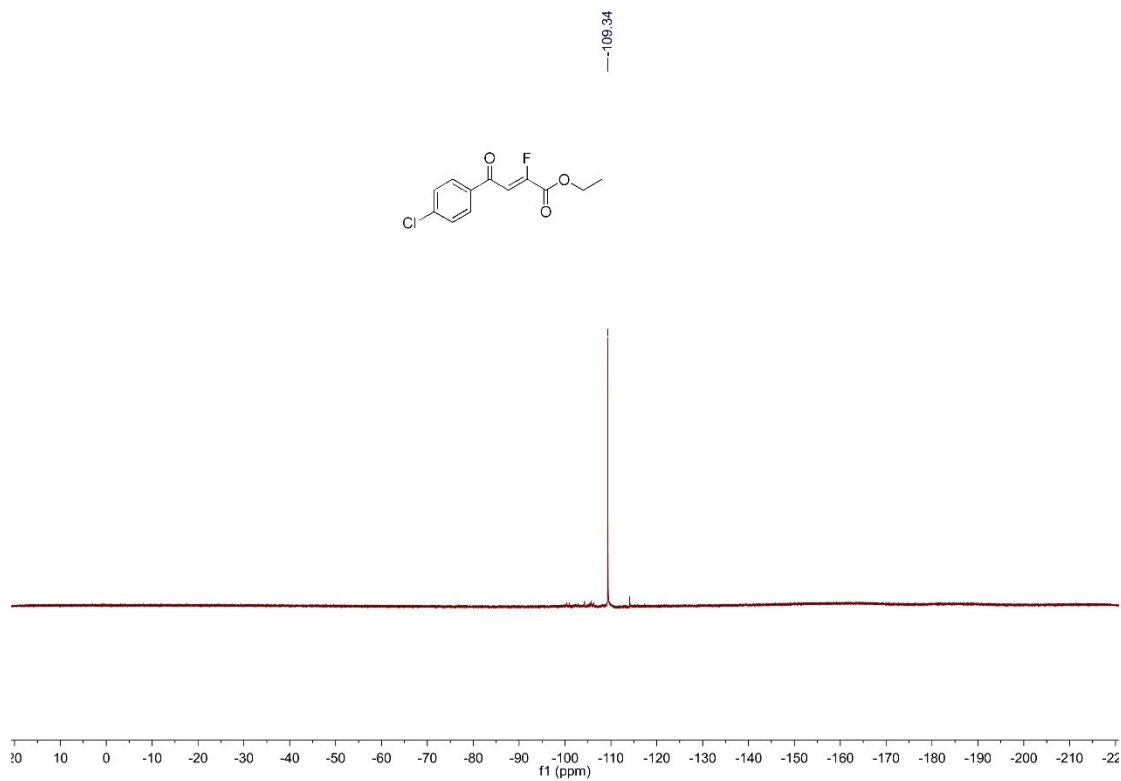
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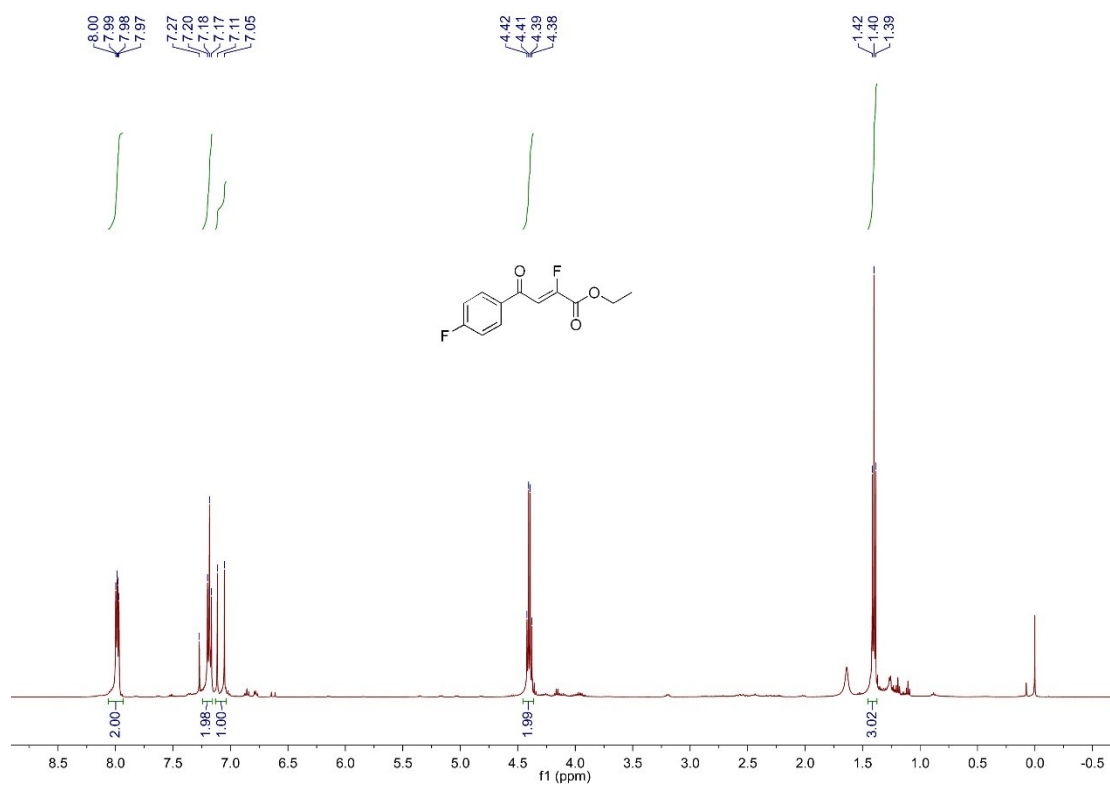
^1H NMR 4b



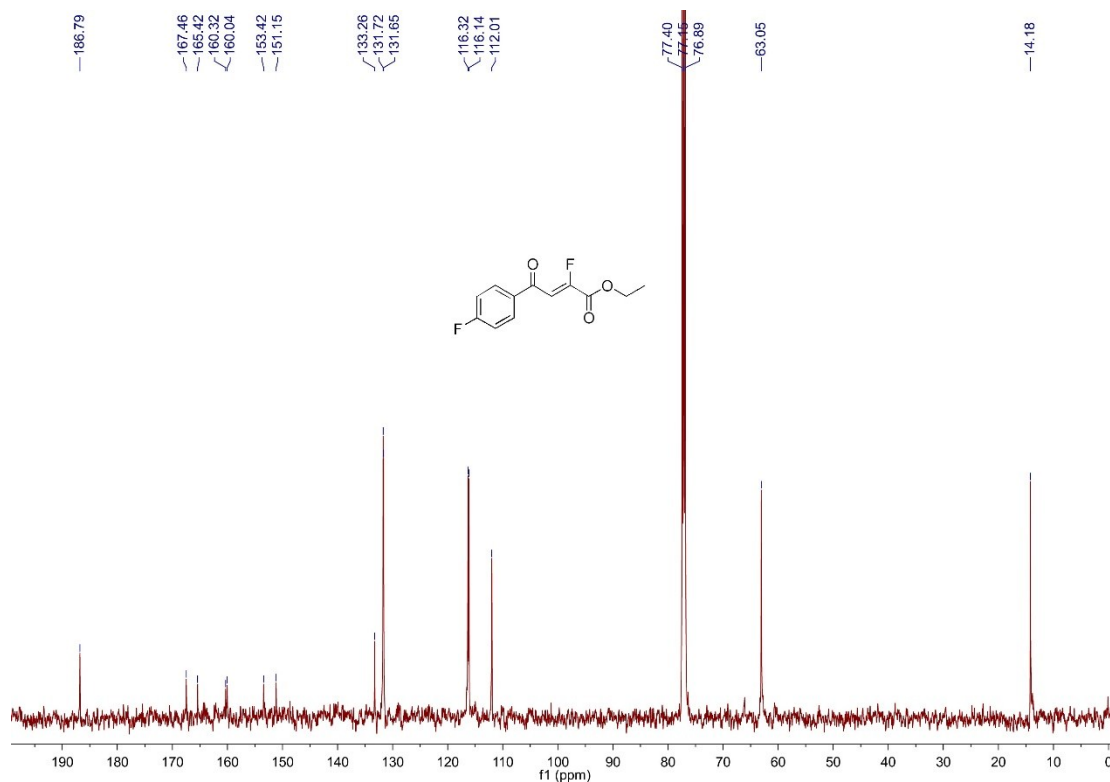
¹³C NMR 4b



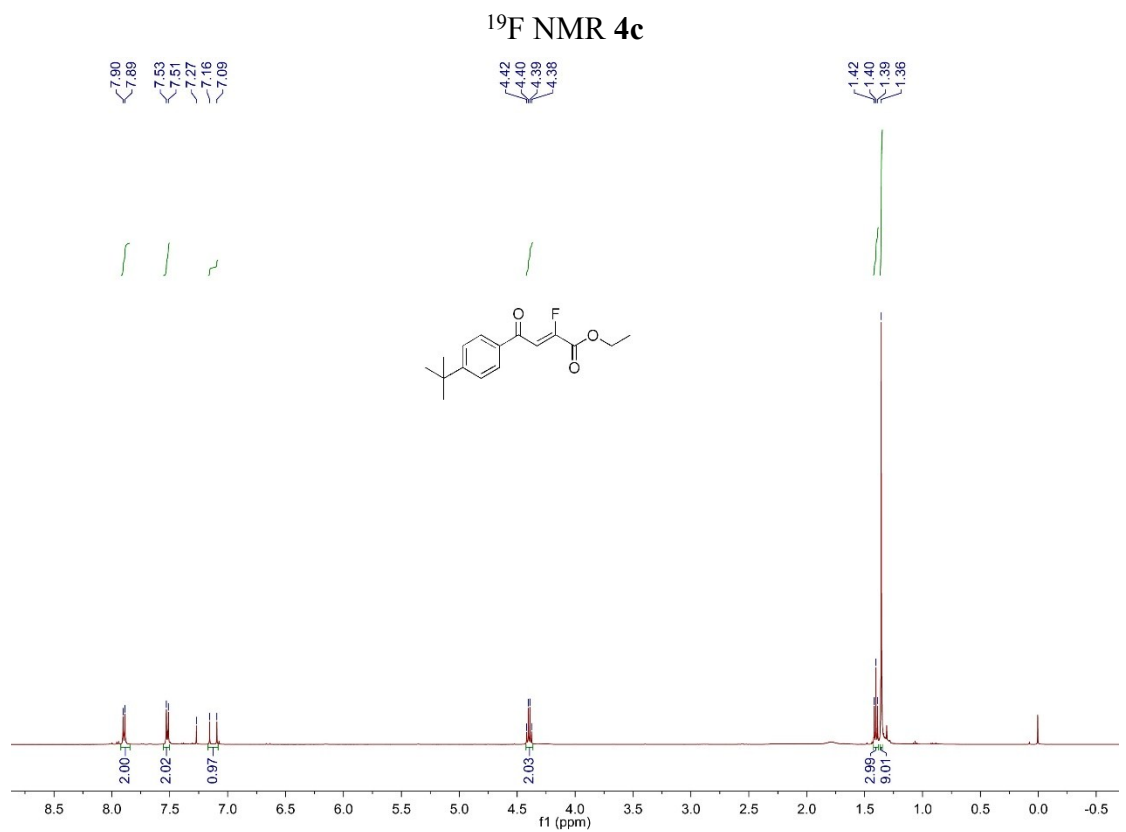
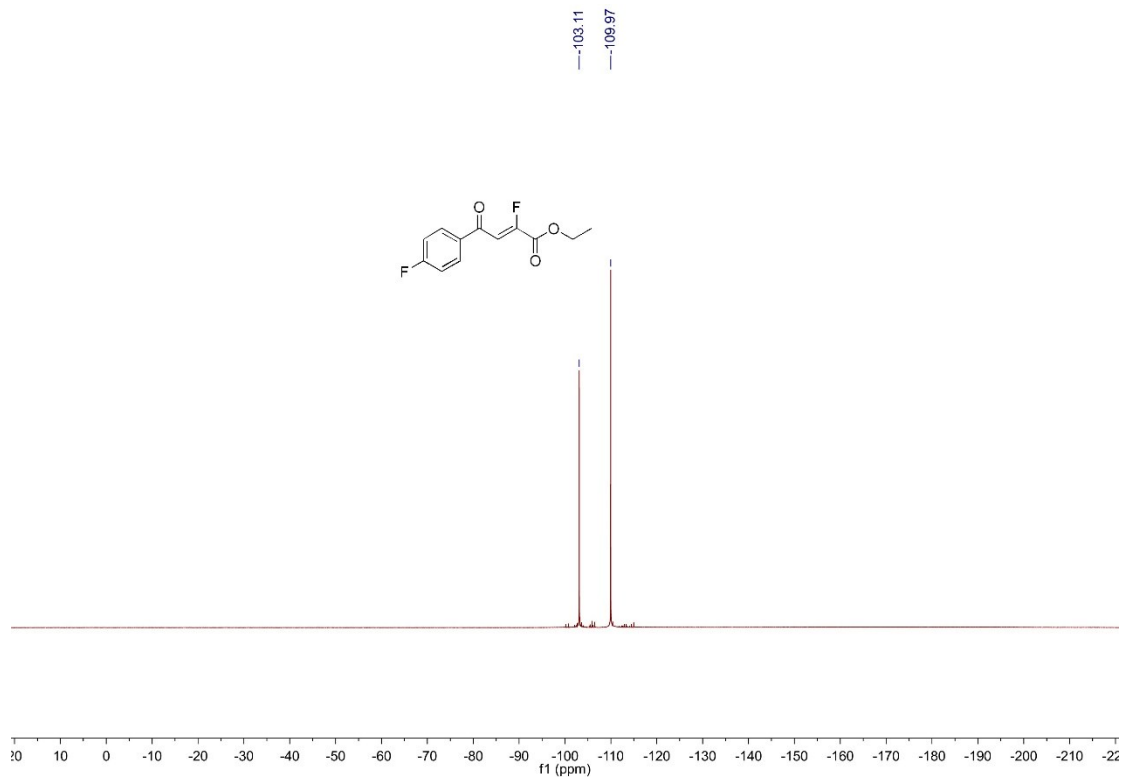
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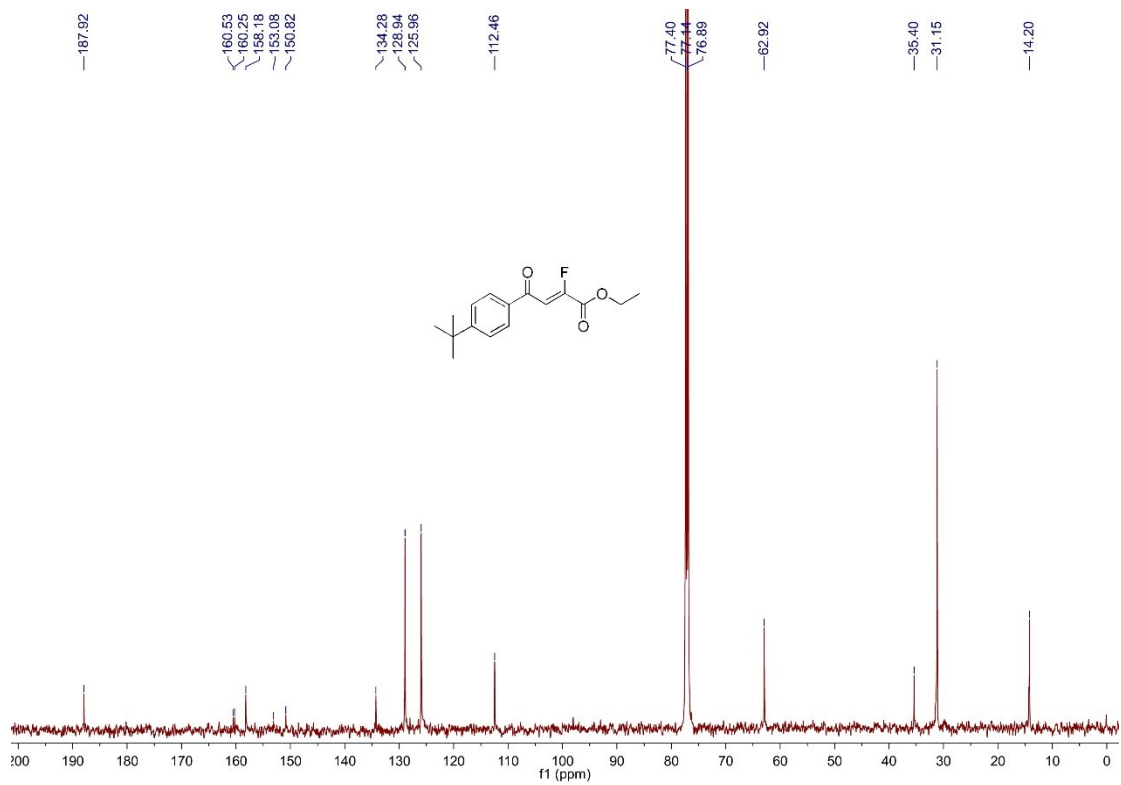


$^1\text{H NMR}$ 4c

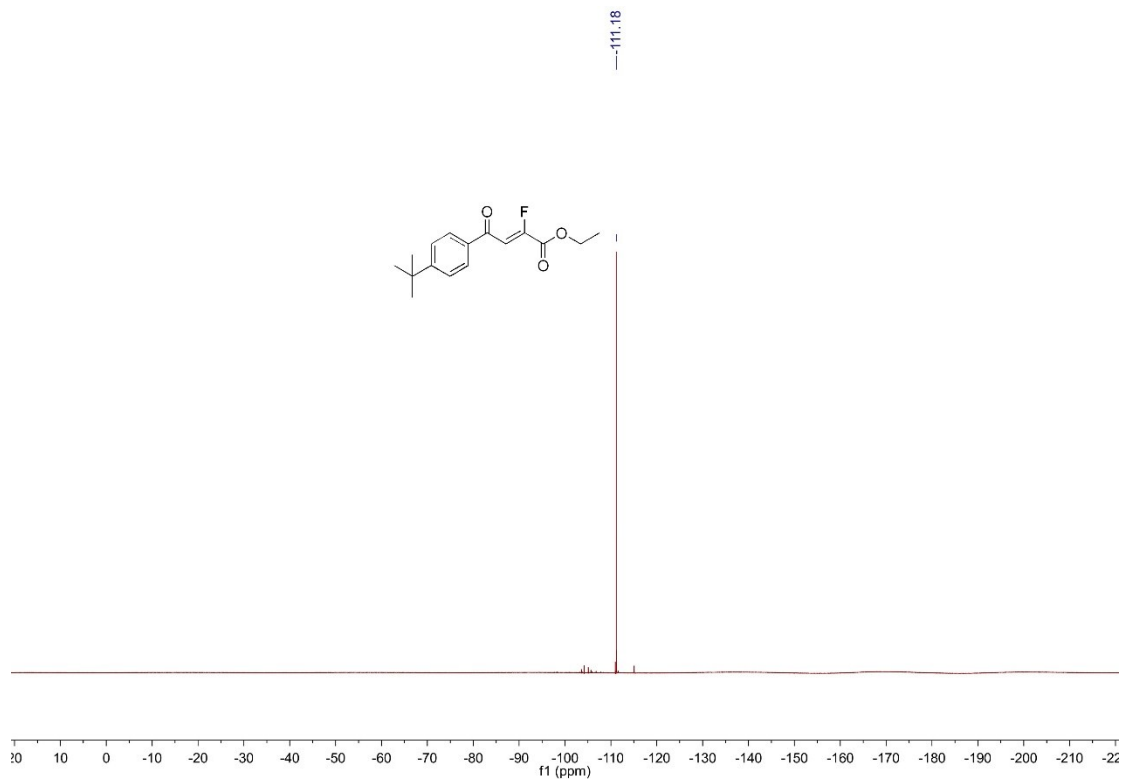


$^{13}\text{C NMR}$ 4c

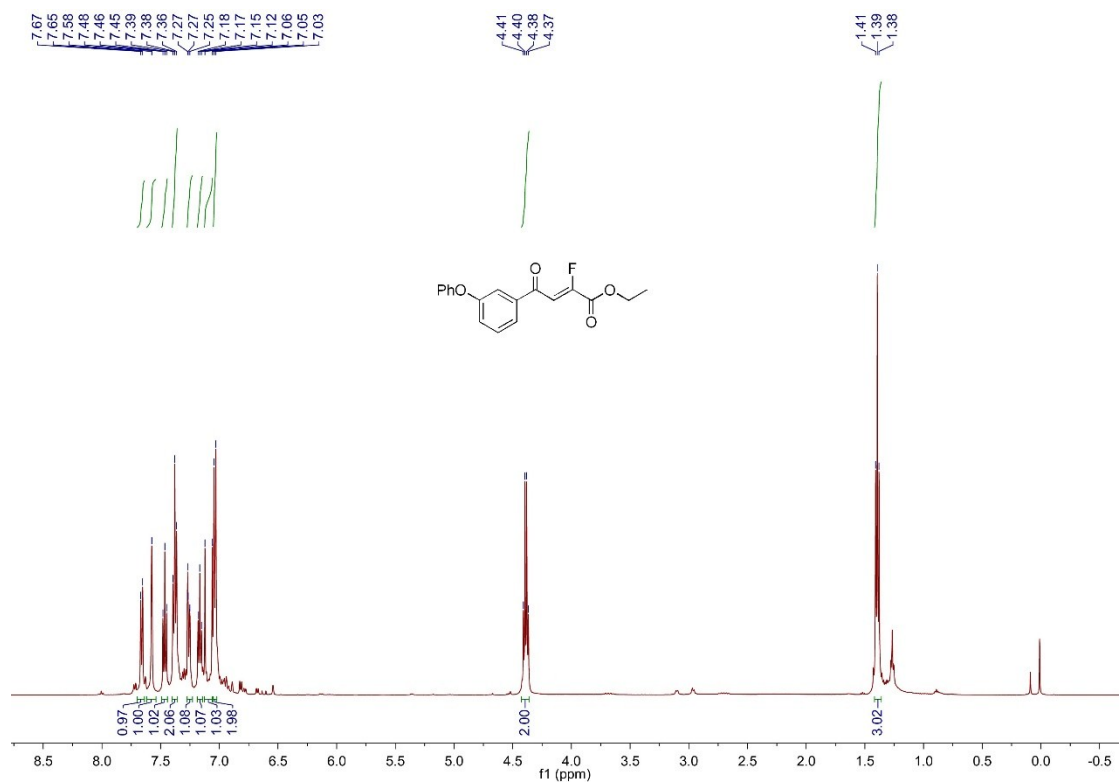




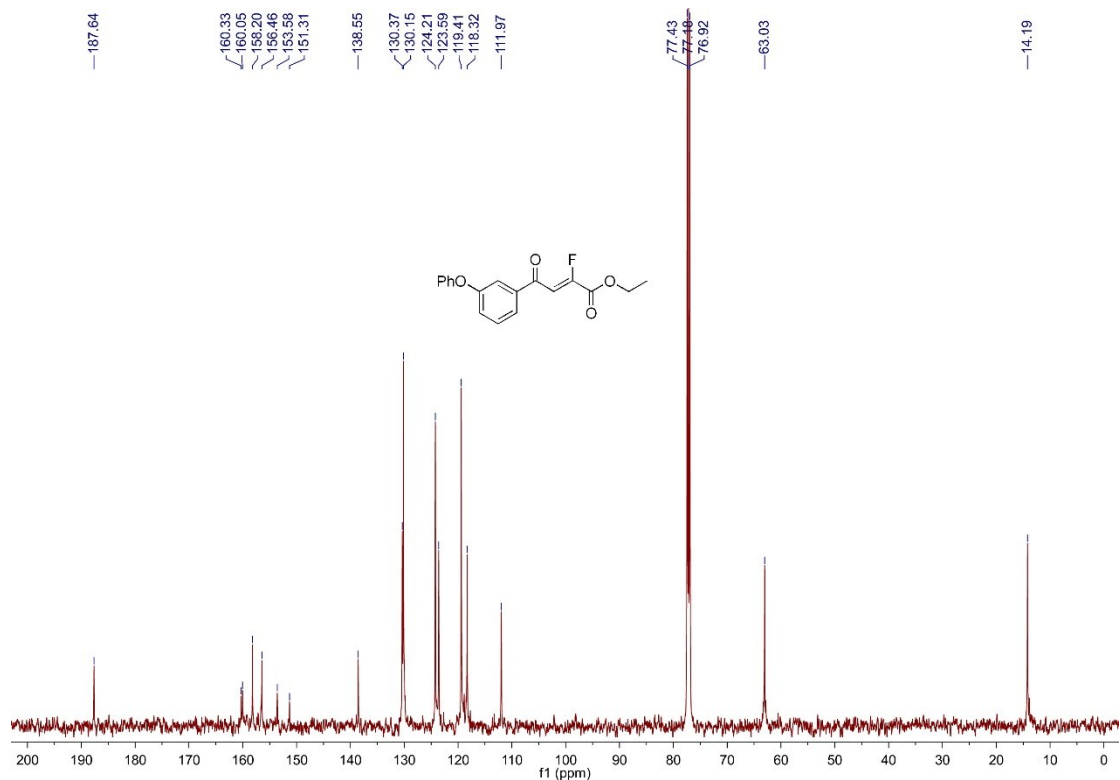
¹³C NMR 4d



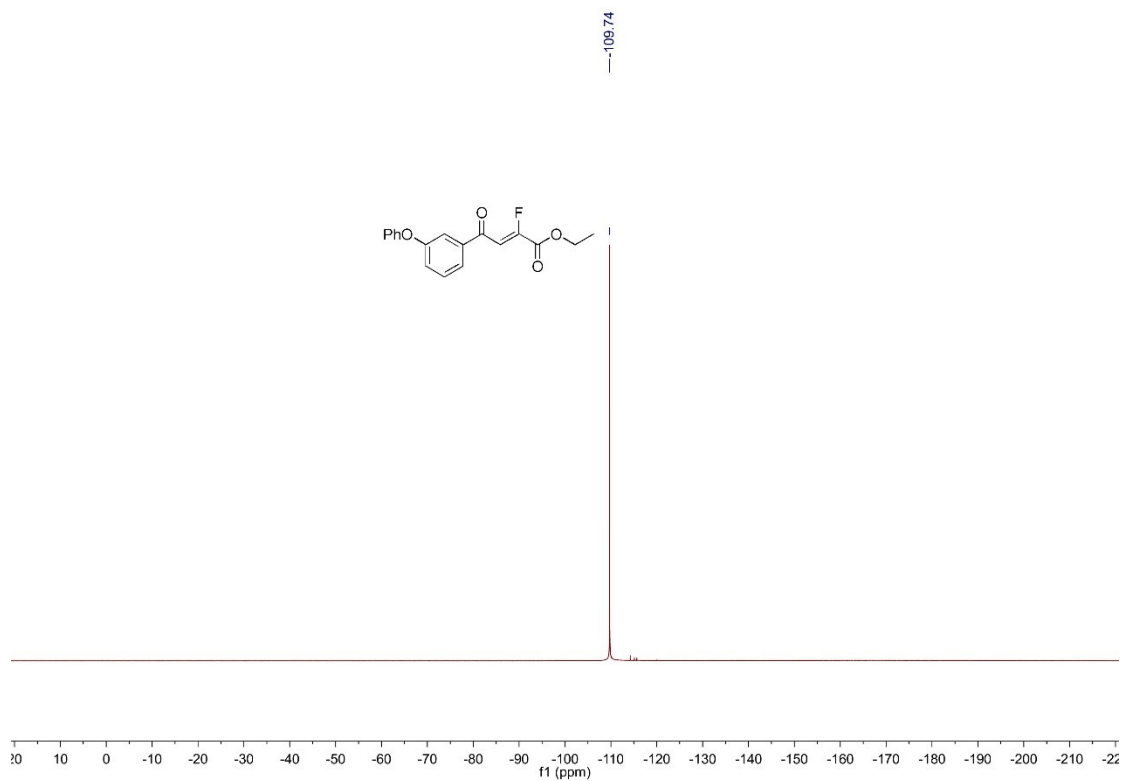
¹⁹F NMR 4d



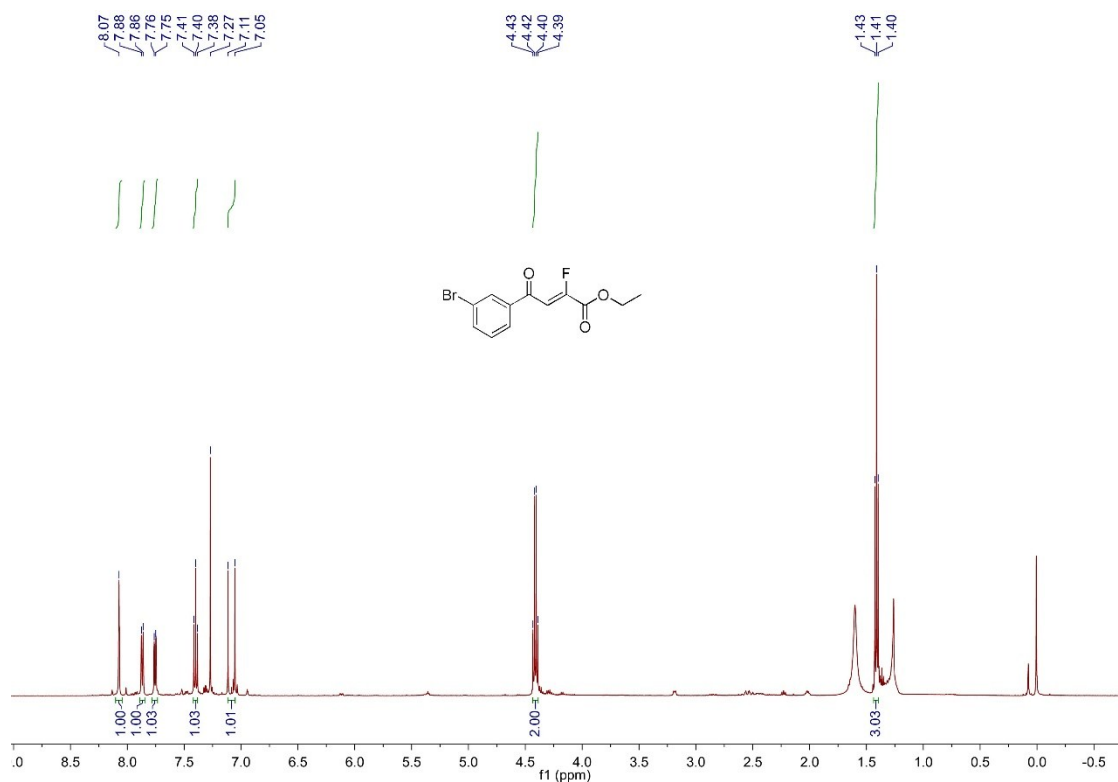
¹H NMR 4e



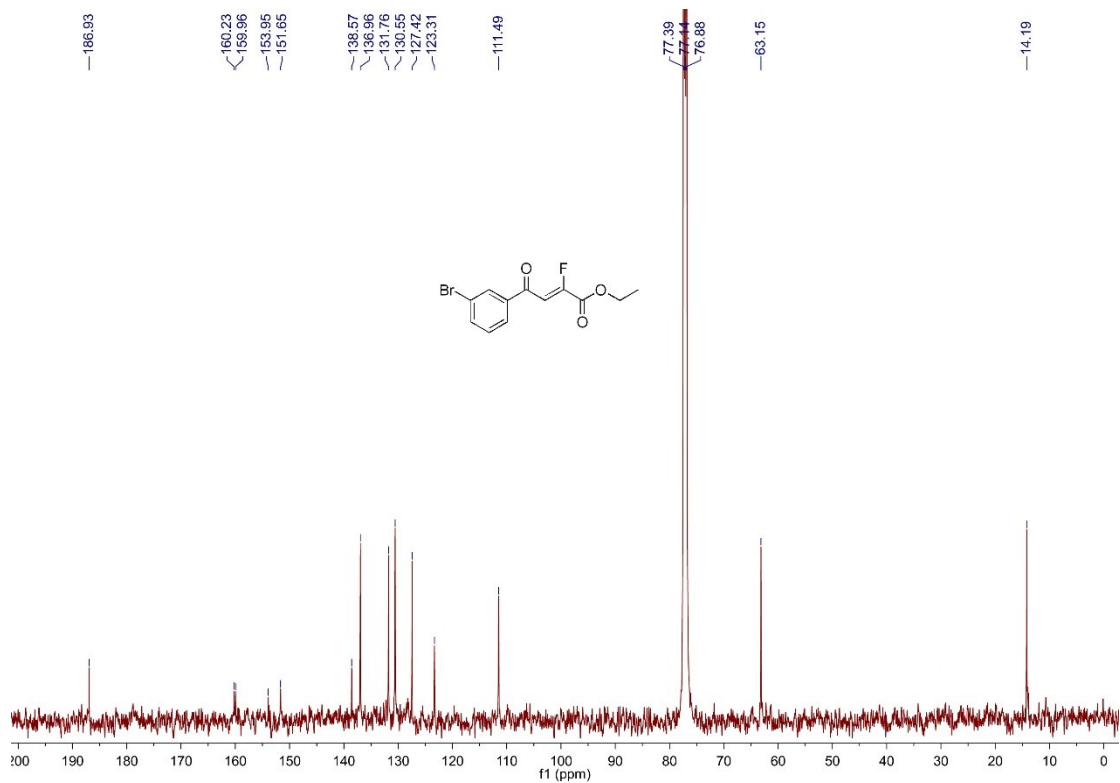
¹³C NMR 4e



^{19}F NMR 4e



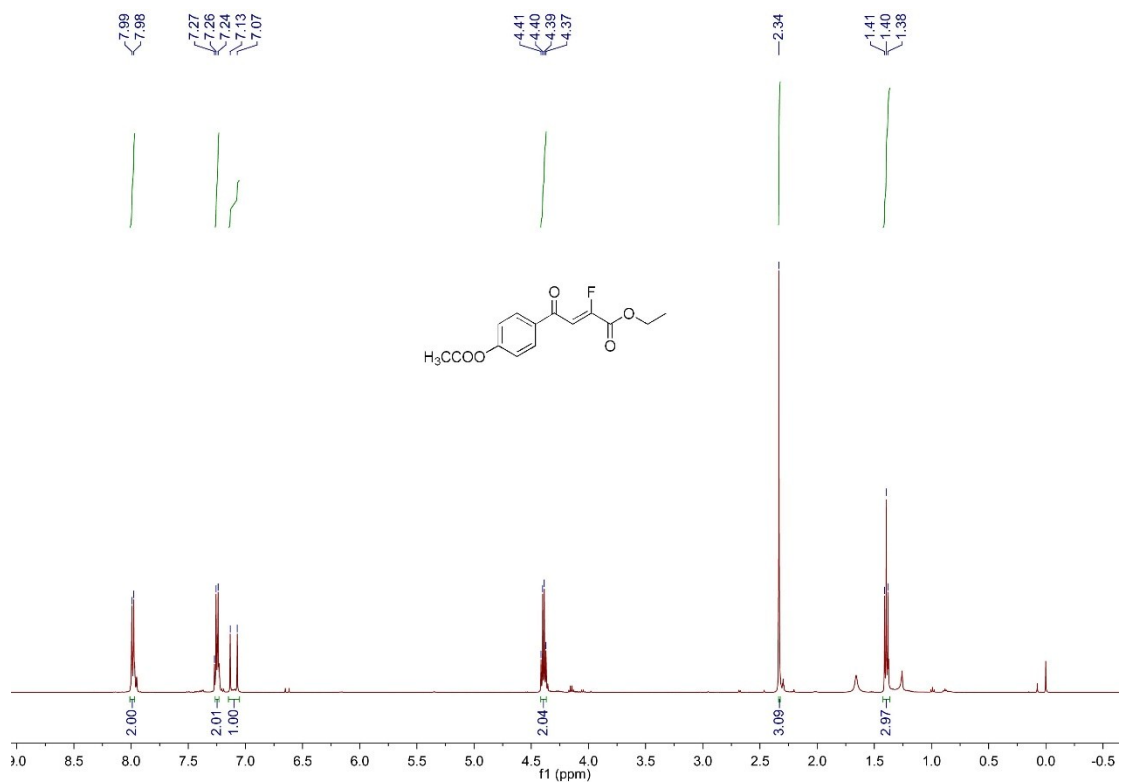
^1H NMR 4f



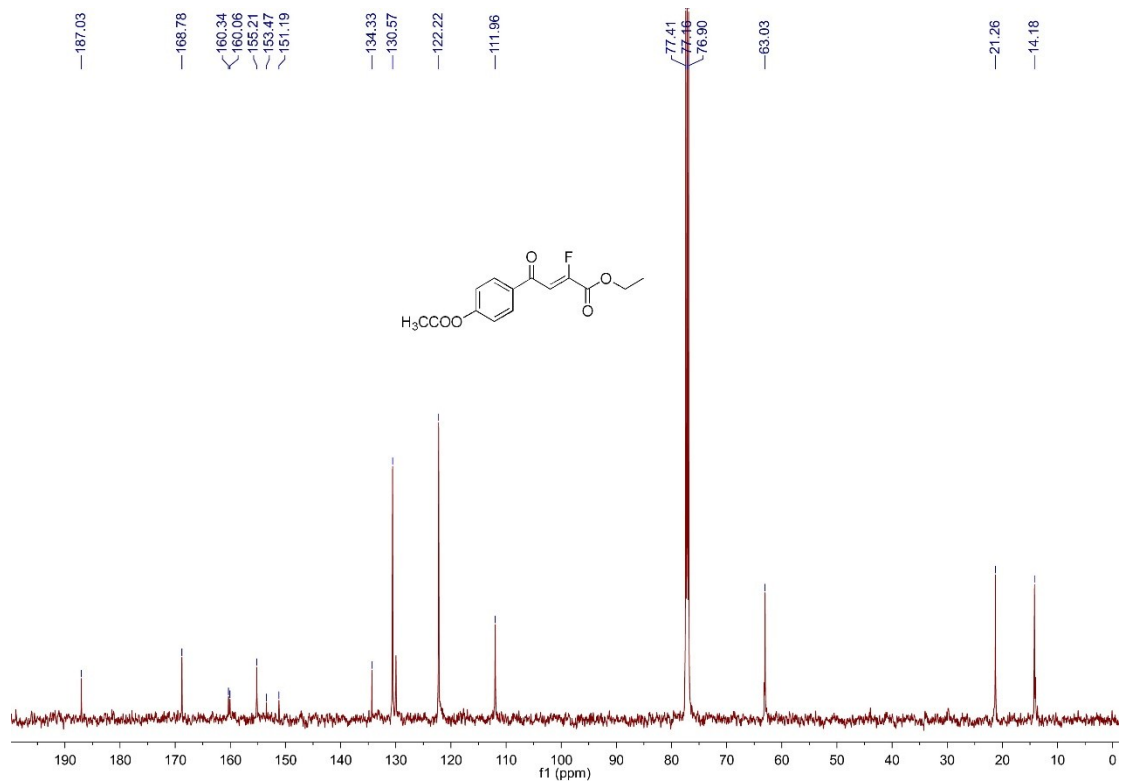
¹³C NMR 4f



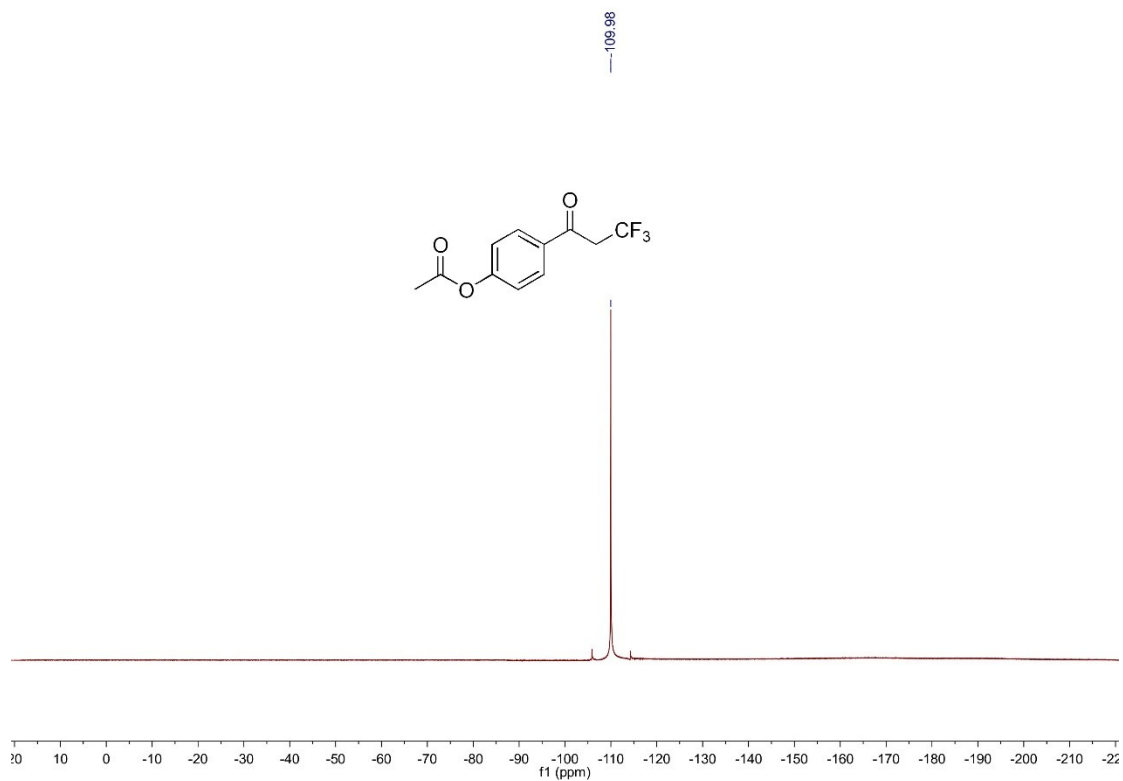
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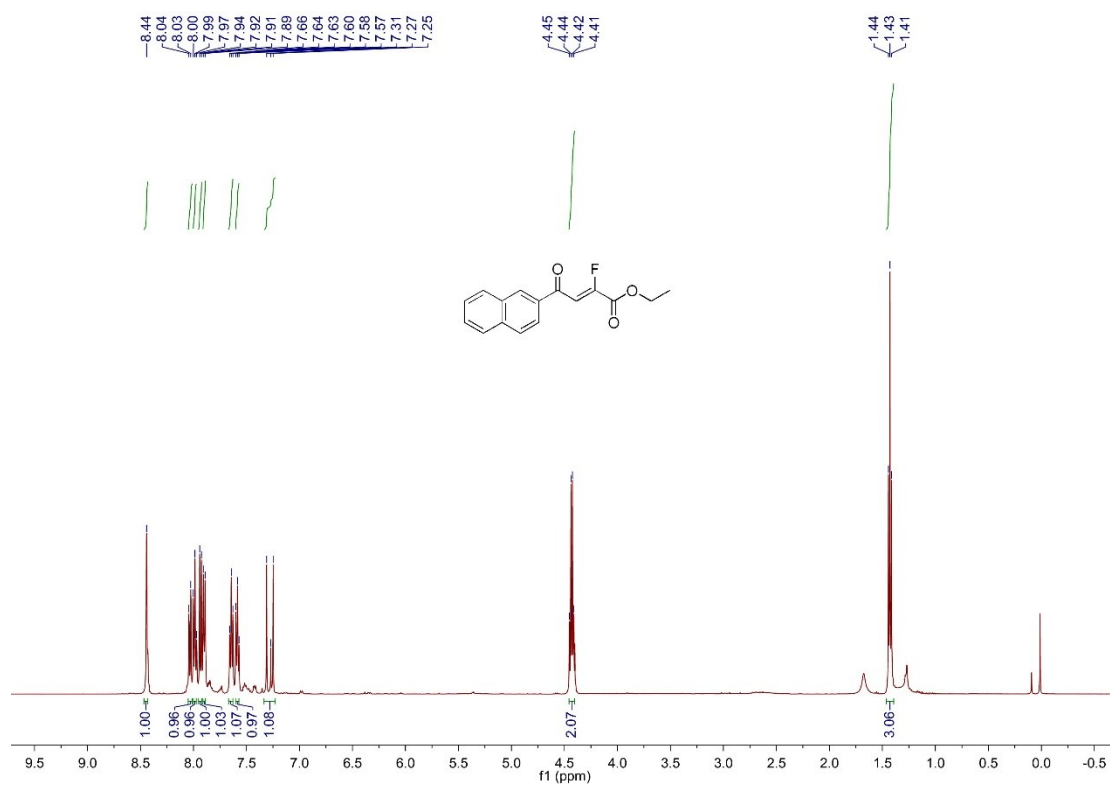
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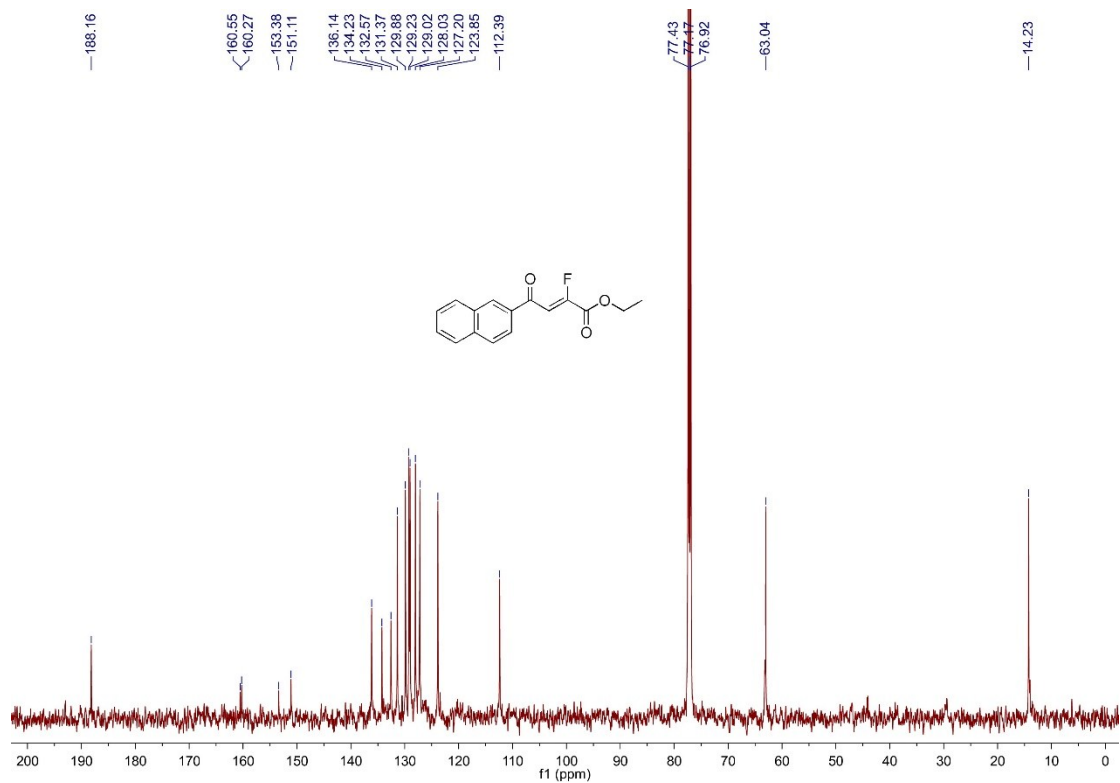
¹³C NMR 4g



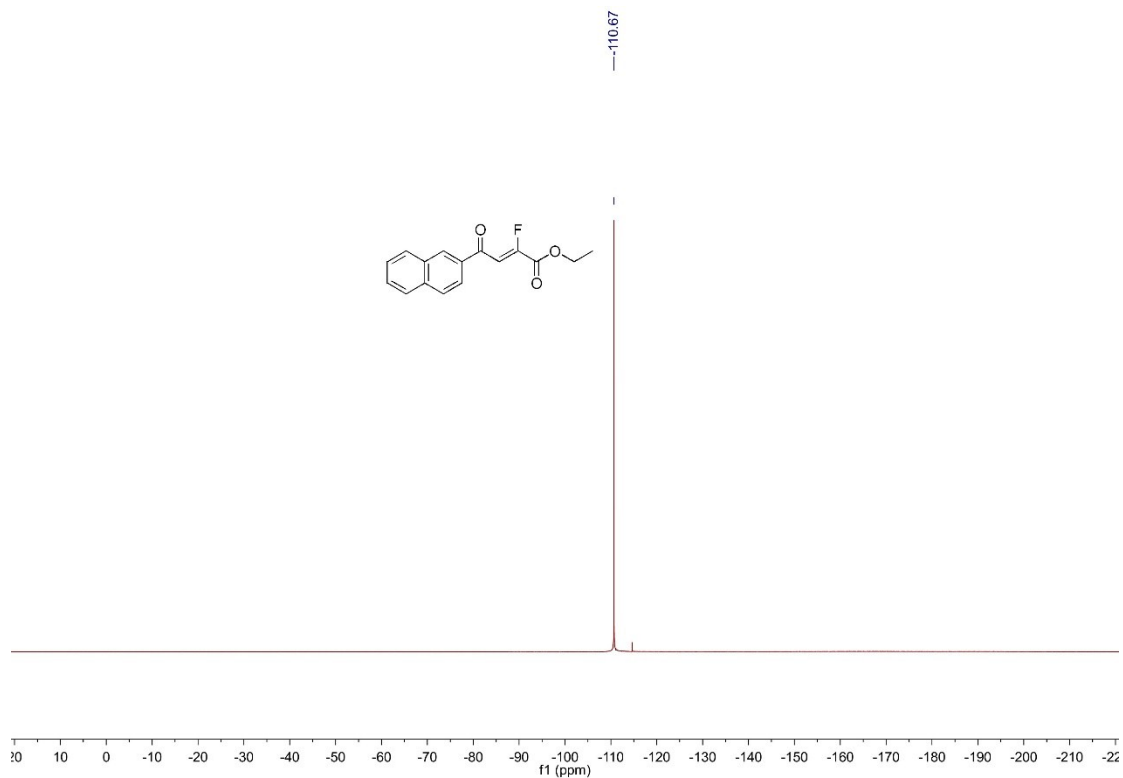
^{19}F NMR 4g



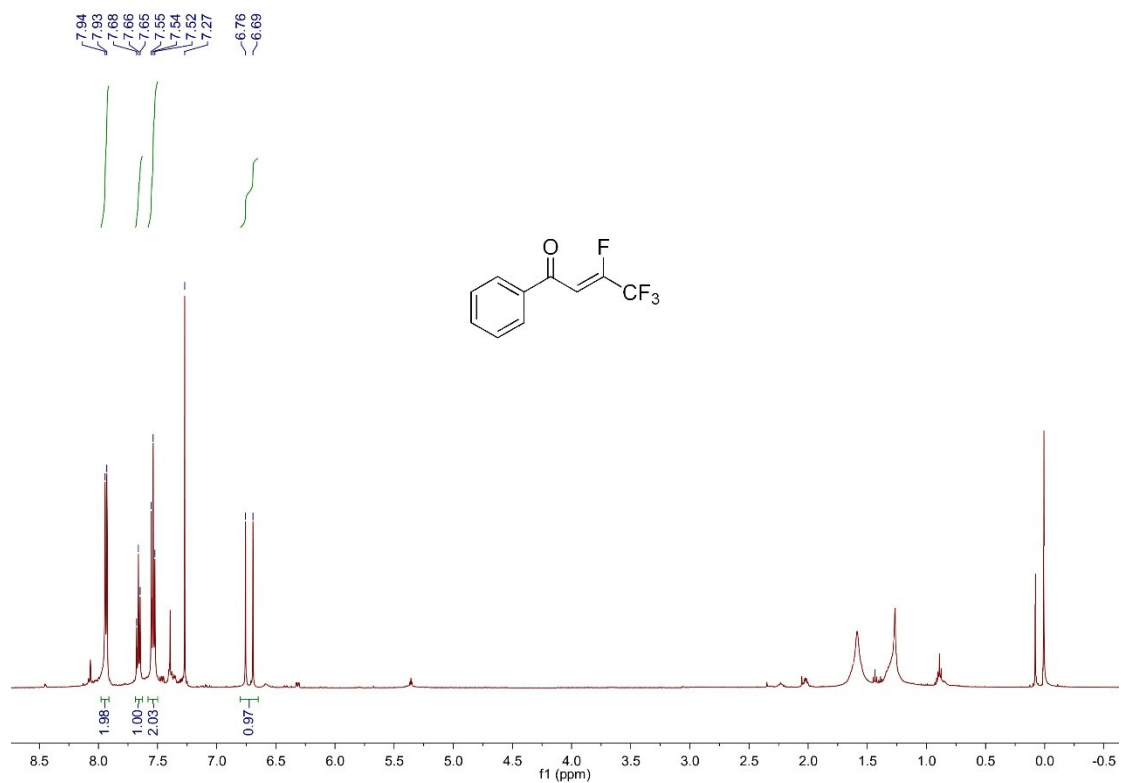
^1H NMR 4h



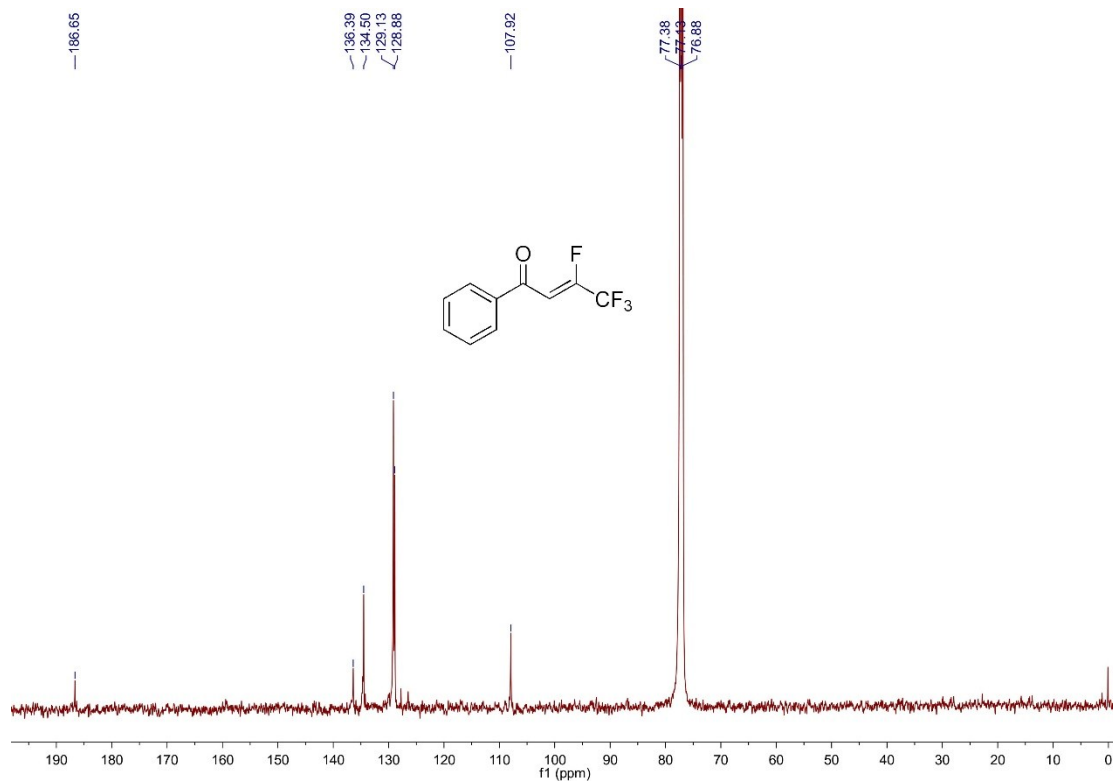
¹³C NMR 4h



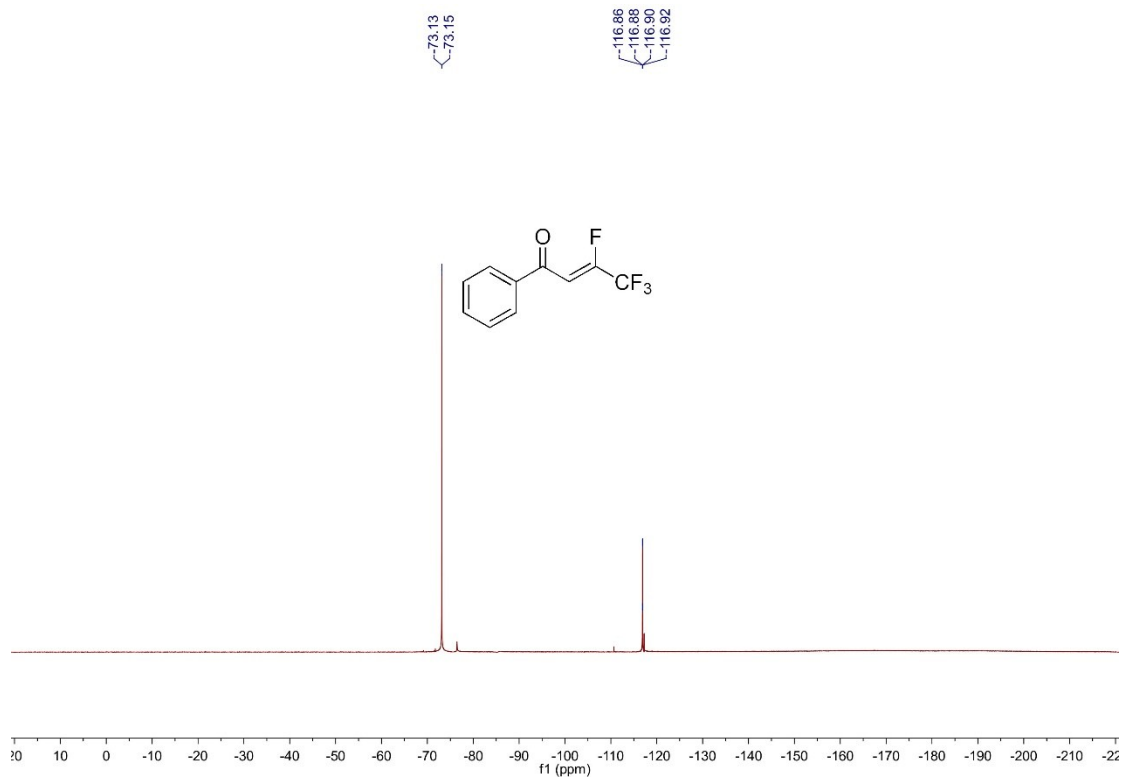
¹⁹F NMR 4h



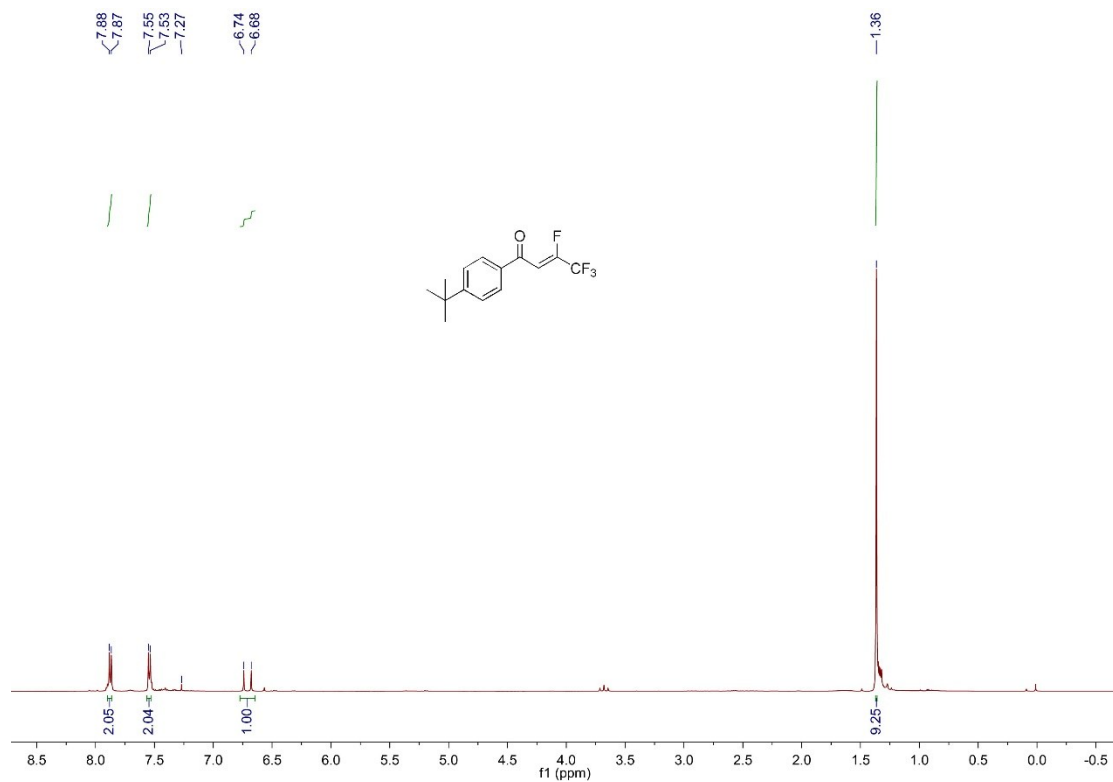
¹H NMR 5a



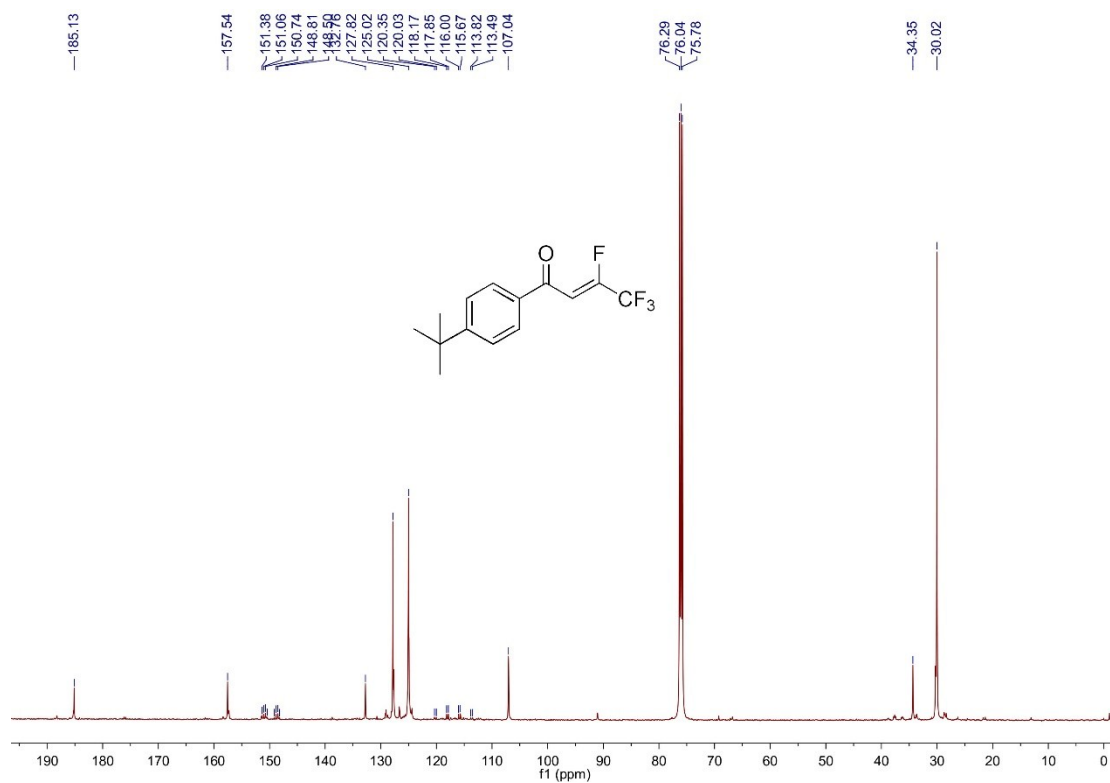
¹³C NMR 5a



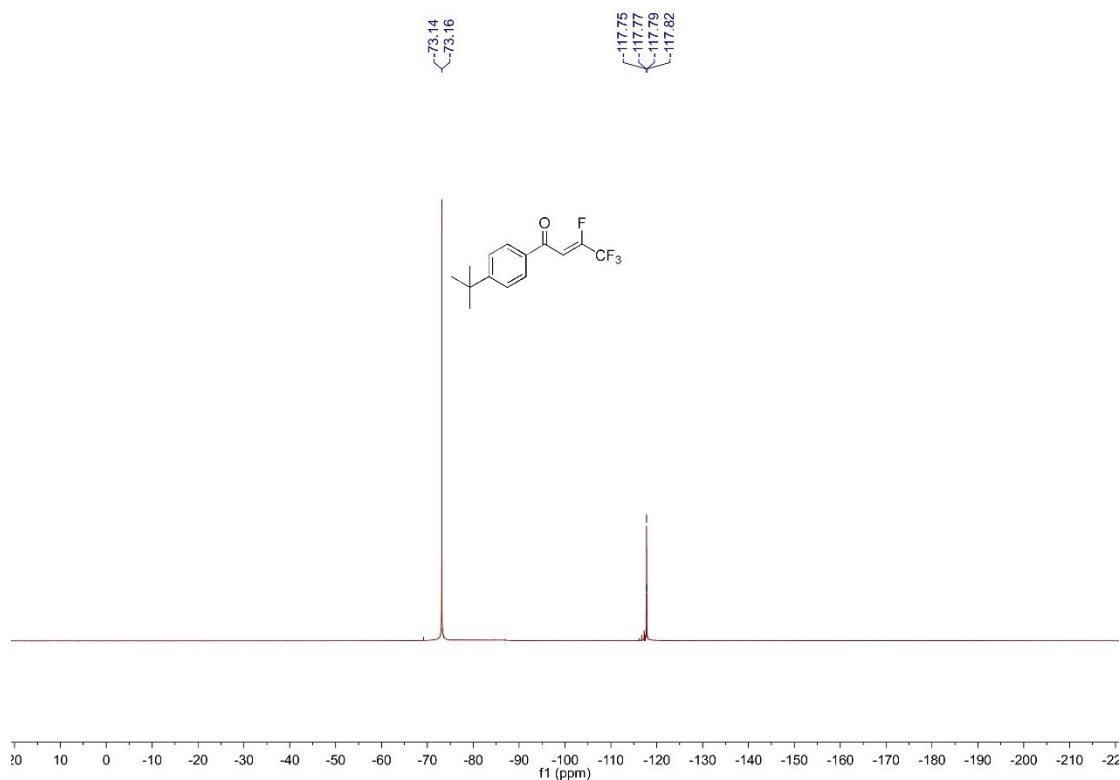
¹⁹F NMR 5a



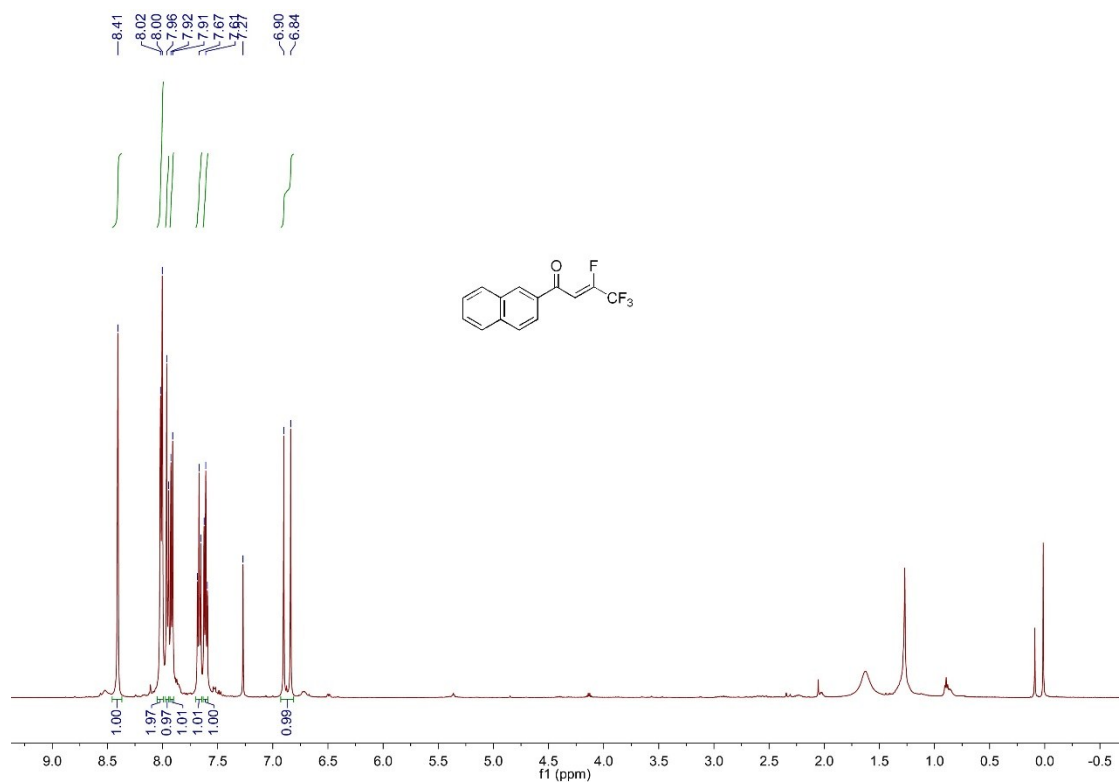
¹H NMR 5b



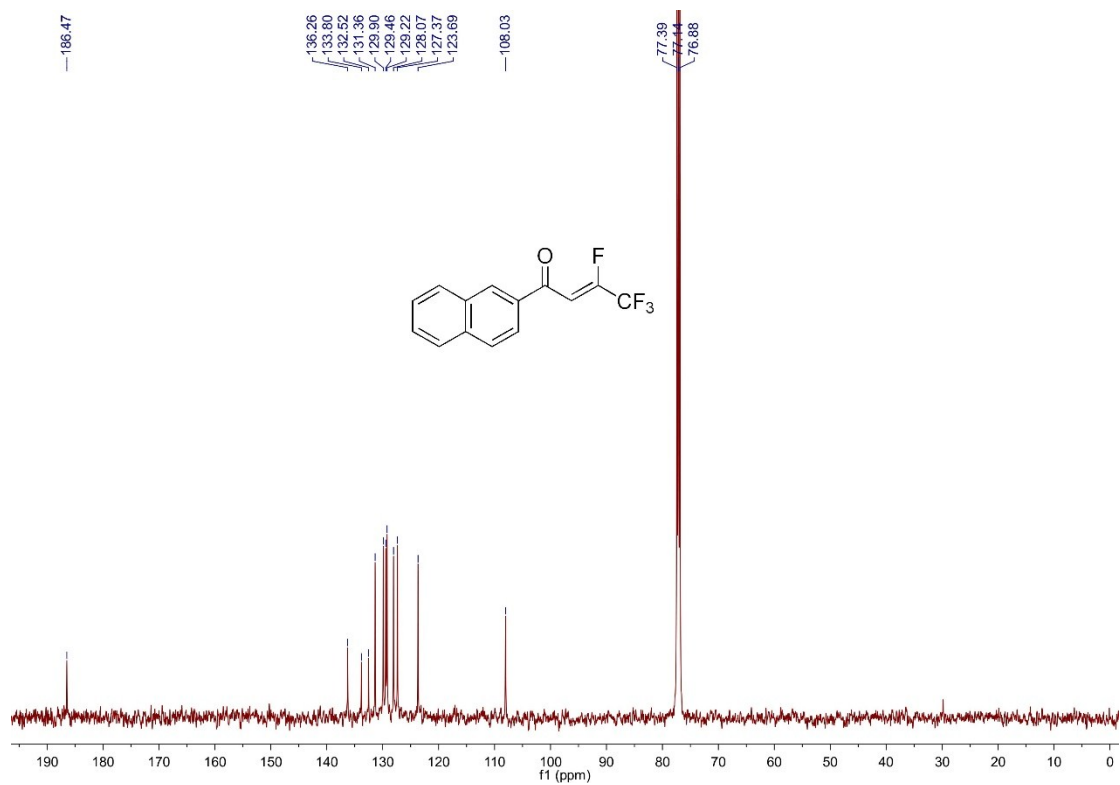
¹³C NMR 5b



¹⁹F NMR 5b



¹H NMR 5c



¹³C NMR 5c



^{19}F NMR **5c**