Supporting Information

Exploring the Structure and Performance of Cd-Chalcogenide Photocatalysts in Selective Trifluoromethylations

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General Information:

Unless otherwise stated, all reactions were carried out under an argon atmosphere in screw cap reaction tubes. All the reagents and solvents were bought from Sigma Aldrich, TCI and Alfa Aesar in a sure-seal bottle and were used as received. Cadmium catalysts were obtained from Sigma Aldrich. For column chromatography, silica gel (100–200 mesh) from Aldrich was used. A gradient elution using *n*-hexane, diethyl ether, DCM and Ethyl Acetate were performed based on Merck aluminum TLC sheets (silica gel 60 F_{254}) and were visualized under UV light (254 nm) or by staining with aqueous potassium permanganate solutions or vanillin alcoholic solution. Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator using a water bath at 25-30 °C.

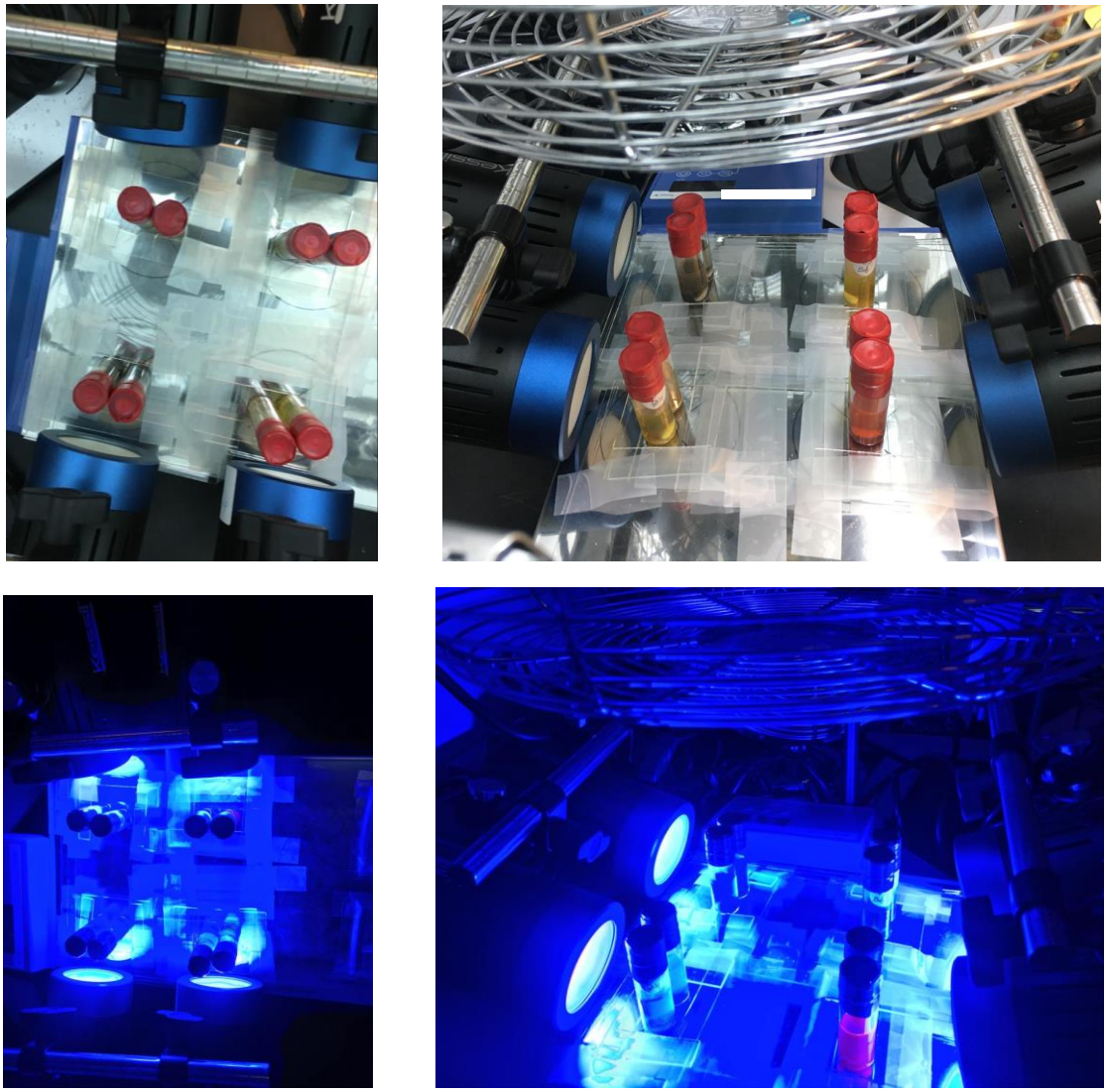
All isolated compounds are characterized by ^{1}H NMR, ^{13}C NMR, ^{19}F NMR spectroscopy, and gas chromatography-mass spectra (GC-MS). Copies of the ^{1}H NMR, ^{13}C NMR and ^{19}F NMR can be found in the supporting information. ^{1}H NMR spectra were recorded in deuterated solvents on Bruker Avance-II spectrometers at 400 or 500 MHz, with residual protic solvent as the internal standard ($CDCl_3$ referenced at 7.26 ppm, and DMSO as referenced at 2.50 ppm, respectively). ^{13}C NMR spectra were recorded in deuterated solvents on Bruker Avance-II spectrometers at 101 or 125 MHz, with the central peak of the deuterated solvent as the internal standard ($CDCl_3$ referenced at 77.16 ppm, and DMSO as referenced at 39.51 ppm, respectively). ^{19}F NMR spectra were recorded in deuterated solvents on Bruker Avance-II at 377 MHz. Chemical shifts (δ) are given in parts per million (ppm), and coupling constants (*J*) are given in Hertz (Hz) rounded to the nearest 0.1 Hz. The ^{1}H NMR spectra are reported as δ/ppm downfield from tetramethylsilane (multiplicity, number of protons, assignment, coupling constant *J*/Hz). The ^{13}C NMR spectra are reported as δ/ppm and were obtained with ^{1}H decoupling and if coupled to fluorine, multiplicity and coupling constant (Hz). Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated s (singlet), b (broad), d (doublet), t (triplet), q = quartet, dd = doublet of doublets and dt = doublet of triplets, m (multiplet); coupling constants (*J*) are in Hertz (Hz). All GCMS analyses were done by Agilent 7890A GC system connected with 5975C inert XL EI/CI MSD (with triple axis detector).

Physical Characterization of CdSe/CdO

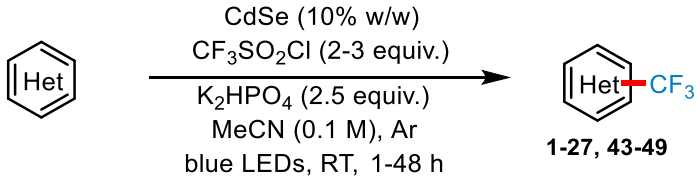
UV-visible spectra were acquired using a JASCO V-670 spectrophotometer with integrating sphere. The Kubelka-Munk function was used to process the data. A Kratos Axis Supra spectrophotometer with monochromatic Al Kα X-ray source (hv = 1486.6 eV) with operating power 300 W, multi-channel plate, and delay line detector was used to collect XPS. The vacuum of the chamber was ~10^{-9} mbar. All spectra were recorded using an aperture slot of 300 μm x 700 μm. Survey spectra were collected using a pass energy of 160 eV and a step size of 1 eV. A pass energy of 20 eV and a step size of 0.1 eV were used for the high-resolution spectra. All binding energies were referenced to the C 1s binding energy of 284.8 eV. Diffraction was carried out using a Bruker D8 ADVANCE X-ray diffractometer at 40 mA/40 kV using Cu Kα (λ = 0.154 nm)

operating conditions. SEM was performed using a Nova NANO SEM 630 (FEI) at an accelerating voltage of 5 kV and working distance of 5 mm. FT-IR spectra were recorded on a Nicolet 6700 FT-IR (ATR) spectrometer and are reported in wavenumbers (cm^{-1}).

Photographs of the photochemical reaction setups



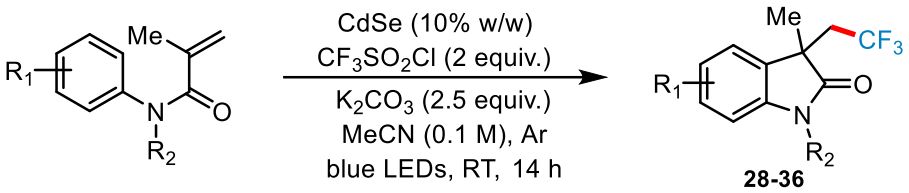
General procedure A for trifluoromethylation of (hetero)arenes



1-27, 43-49

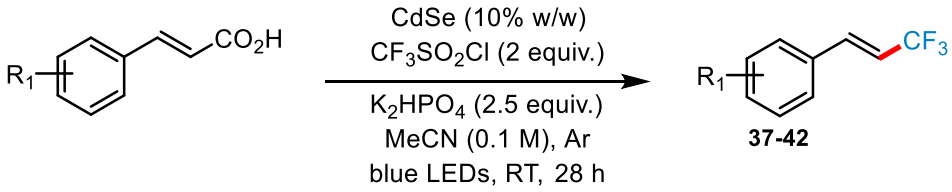
A clean, oven-dried screw cap reaction tube equipped with a Teflon-coated magnetic stir bar was charged with (hetero)arenes (0.3 mmol, 1 equiv.), CdSe (10% w/w) and K_2HPO_4 (130.5 mg, 0.75 mmol, 2.5 equiv.). The reaction tube was capped with a rubber septum, evacuated and backfilled with argon (3 times). Then, dry MeCN (0.1 M, 3.0 mL) and triflyl chloride (64-96 µL, 0.6-0.9 mmol, 2-3 equiv.) were added by syringe and the vial was sealed with parafilm under argon atmosphere. The reaction mixture was stirred at room temperature for 1-48 h under irradiation with 34 W blue LEDs (3 cm away from blue LEDs) with fan cooling. After completion, the reaction mixture was quenched with water and then extracted with diethyl ether or DCM (3 x 10 mL). The organic layer was collected, dried over anhydrous Na_2SO_4, and concentrated *in vacuo*. The residue was purified by column chromatography using silica gel (100-200 mesh size) and diethyl ether/petroleum ether or DCM/petroleum ether as the eluent. (w/w = weight by weight ratio with respect to limiting reagent).

General procedure B for trifluoromethylation of *N*-arylacrylamides



A clean, oven-dried screw cap reaction tube equipped with a Teflon-coated magnetic stir bar was charged with *N*-arylacrylamides (0.3 mmol, 1 equiv.), CdSe (10% w/w) and K_2CO_3 (103.5 mg, 0.75 mmol, 2.5 equiv.). The reaction tube was capped with a rubber septum, evacuated and backfilled with argon (3 times). Then, dry MeCN (0.1 M, 3.0 mL) and triflyl chloride (64 µL, 0.6 mmol, 2 equiv.) were added by syringe and the vial was sealed with parafilm under argon atmosphere. The reaction mixture was stirred at room temperature for 14 h under irradiation with 34 W blue LEDs (3 cm away from blue LEDs) with fan cooling. After completion, the reaction mixture was quenched via exposure to air. The reaction mixture was diluted with DCM and filtered through a small bed of Celite and concentrated *in vacuo*. The residue was purified by column chromatography using silica gel (100-200 mesh size) and EtOAc/petroleum ether as the eluent. (w/w = weight by weight ratio with respect to limiting reagent).

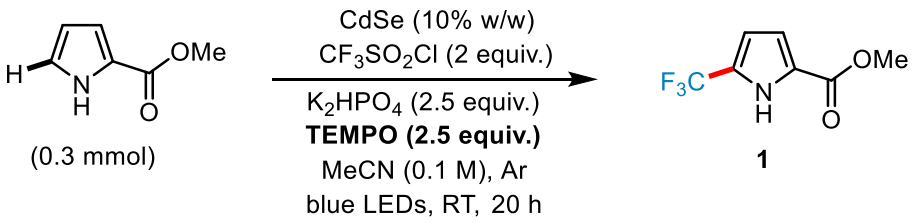
General procedure C for trifluoromethylation of vinylic acids



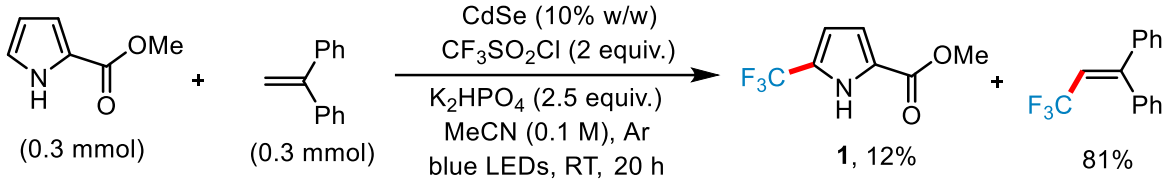
A clean, oven-dried screw cap reaction tube equipped with a Teflon-coated magnetic stir bar was charged with α,β-unsaturated acid (0.3 mmol, 1 equiv.), CdSe (10% w/w) and K_2HPO_4 (130.5 mg, 0.6 mmol, 2.5 equiv.). The reaction tube was capped with a rubber septum, evacuated and

backfilled with argon (3 times). Then, dry MeCN (0.1 M, 3.0 mL) and triflyl chloride (96 µL, 0.9 mmol, 3 equiv.) were added by syringe and the vial was sealed with parafilm under argon atmosphere. The reaction mixture was stirred at room temperature for 28 h under irradiation with 34 W blue LEDs (3 cm away from blue LEDs) with fan cooling. After 28 hours, the reaction mixture was treated with the saturated solution of $NaHCO_3$ and then extracted with diethyl ether (3 x 10 mL). The organic layer was collected, dried over anhydrous Na_2SO_4, and concentrated *in vacuo*. The residue was purified by column chromatography using silica gel (100-200 mesh size) and DCM/petroleum ether or EtOAc/petroleum ether as the eluent. (w/w = weight by weight ratio with respect to limiting reagent).

Radical trapping experiments



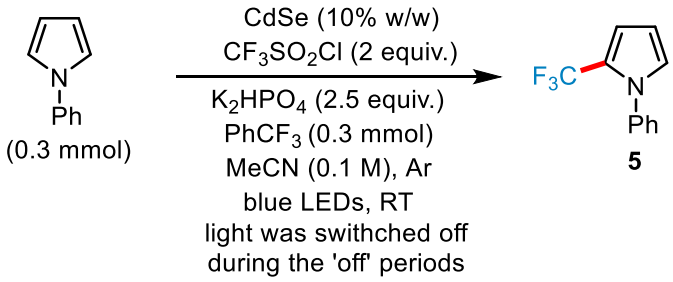
A clean, oven-dried screw cap reaction tube equipped with a Teflon-coated magnetic stir bar was charged with methyl 2-pyrrolecarboxylate (37.5 mg, 0.3 mmol), CdSe (3.7 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol) and TEMPO (117.2 mg, 0.75 mmol). The reaction tube was capped with a rubber septum, evacuated and backfilled with argon (3 times). Then, dry MeCN (0.1 M, 3.0 mL) and triflyl chloride (64 µL, 0.6 mmol, 2 equiv.) were added by syringe and the vial was sealed with parafilm under argon atmosphere. The reaction mixture was stirred at room temperature for 20 h under irradiation with 34 W blue LEDs (3 cm away from blue LEDs) with fan cooling. After 20 h, the reaction was quenched via exposure to air. The GCMS and ^{19}F NMR analysis of the crude reaction mixture did not show the formation of product **1**.

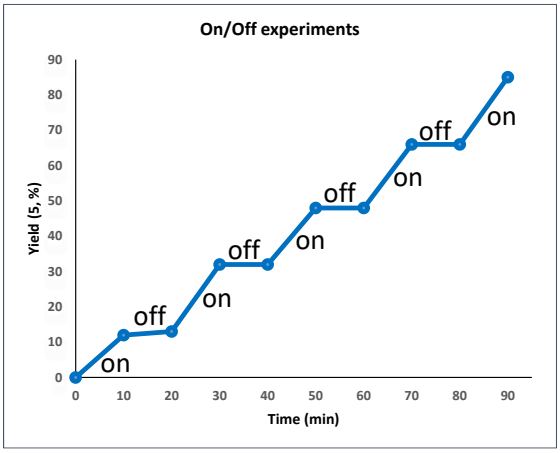


A clean, oven-dried screw cap reaction tube equipped with a Teflon-coated magnetic stir bar was charged with methyl 2-pyrrolecarboxylate (37.5 mg, 0.3 mmol), CdSe (3.7 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol) and 1,1-diphenylethylene (54 mg, 0.3 mmol). The reaction tube was capped with a rubber septum, evacuated and backfilled with argon (3 times). Then, dry MeCN (0.1 M, 3.0 mL) and triflyl chloride (64 µL, 0.6 mmol) were added by syringe and the vial was sealed with parafilm under argon atmosphere. The reaction mixture was stirred at room temperature for 20 h under irradiation with 34 W blue LEDs (3 cm away from blue LEDs) with fan cooling. After 20 h, the reaction mixture was quenched with water and then extracted with diethyl ether (3 x 10 mL). The organic layer was collected, dried over anhydrous Na_2SO_4, and

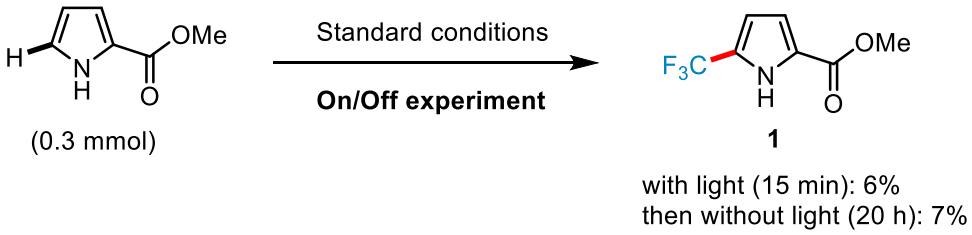
concentrated *in vacuo*. The residue was purified by column chromatography using silica gel (100-200 mesh size) and diethyl ether/petroleum ether as the eluent. Yields were determined by ^{19}F NMR analysis of the crude mixture using trifluoromethylbenzene as an internal standard.

On-off experiments

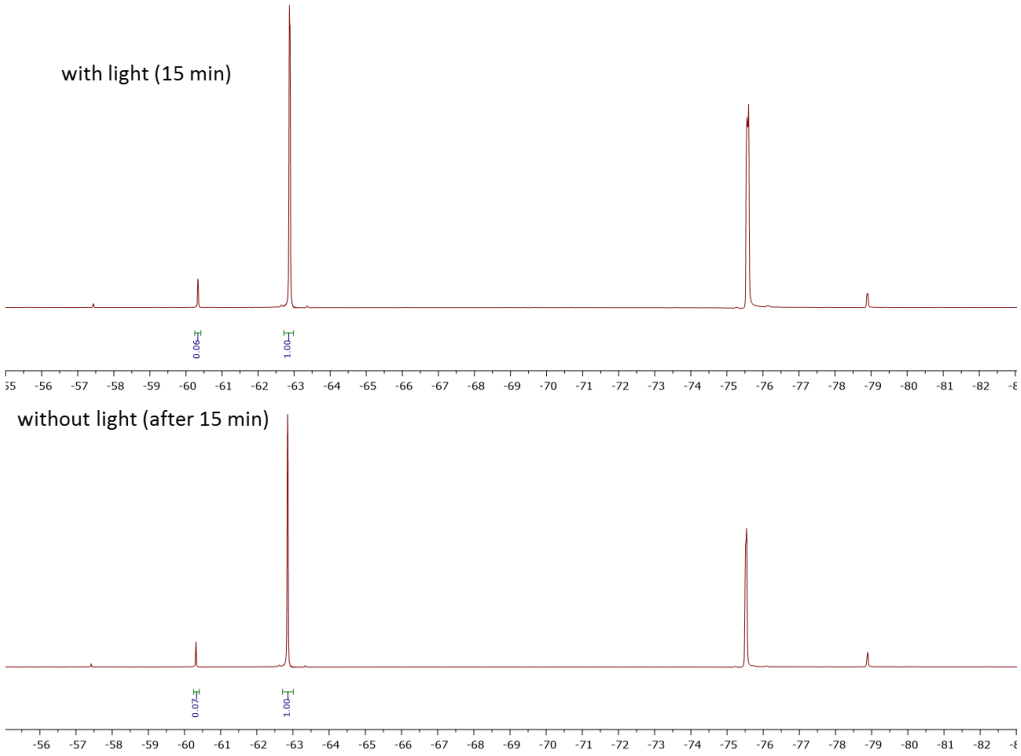




To exclude the possibility of a radical chain process in the reaction, light on-off experiments were carried following the general procedure, with 1-phenyl-1*H*-pyrrole (42.9 mg, 0.3 mmol), CdSe (4.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), triflyl chloride (64 µL, 0.6 mmol) and trifluoromethylbenzene (37 µL, 0.3 mmol). The light was kept off during the off-periods and the yields of the reaction were determined by ^{19}F NMR analysis. No reaction was observed during the light off-cycles which confirms that the reaction is not proceeding by radical chain propagation mechanism.



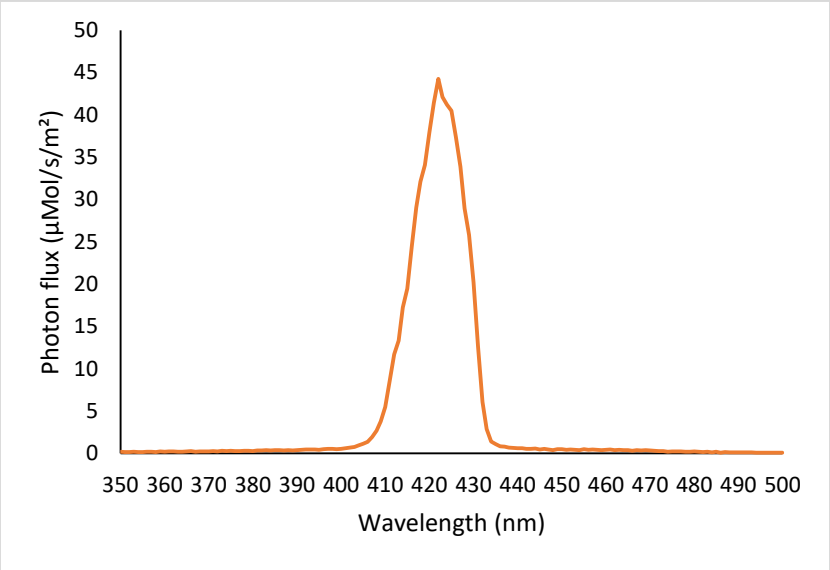
with light (15 min): 6%
then without light (20 h): 7%



The above reaction was performed according to the general procedure A. The yield of the reaction was determined by ^{19}F NMR analysis of the crude mixture using trifluoromethylbenzene as an internal standard.

Quantum yield measurement

The quantum yield was measured according to a published procedure.[1,2] The photon flux was measured using the AvaSpec-3648 spectrometer, AvaLight DHS calibration light source, and FC-UV200-2 fiber-optic cable and the Apparent Quantum Efficiency (AQE) measurement was conducted with a 420 ±5 nm bandpass filter. Photon flux of this spectrophotometer was determined as 3.51348×10^{-7} Einstein/sec using calibrated light source.

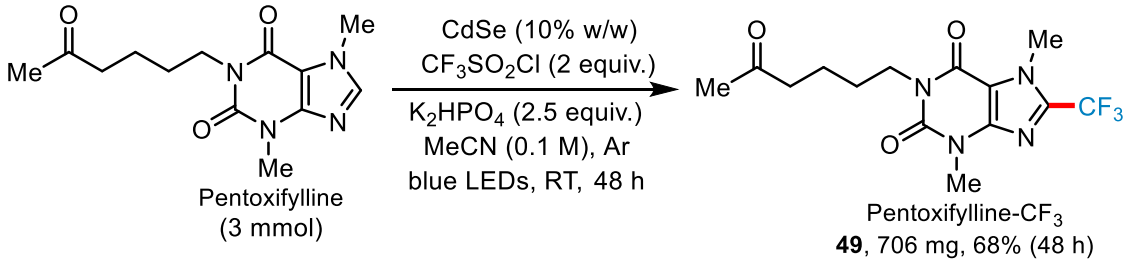


A clean, quartz screw cap reaction tube equipped with a Teflon-coated magnetic stir bar was charged with methyl 2-pyrrolecarboxylate (37.5 mg, 0.3 mmol), CdSe (3.7 mg, 10% w/w), and K_2HPO_4 (130.5 mg, 0.75 mmol). The reaction tube was capped with a rubber septum, evacuated and backfilled with argon (3 times). Then, dry MeCN (0.1 M, 3.0 mL) and triflyl chloride (64 µL, 0.6 mmol) were added by syringe and the vial was sealed with parafilm under argon atmosphere. The reaction mixture was stirred and irradiated at 420 ±5 nm for 900 s with fan cooling. After irradiation, the reaction was quenched with air and yield was determined by ^{19}F NMR analysis using trifluoromethylbenzene as an internal standard. Finally, the amount of **1** was measured as 5.7×10^{-5} mol.

$$\phi = \frac{\text{moles of product}}{\text{moles of absorbed photons}} = \frac{\text{moles of product}}{\text{flux} \bullet t \bullet f}$$

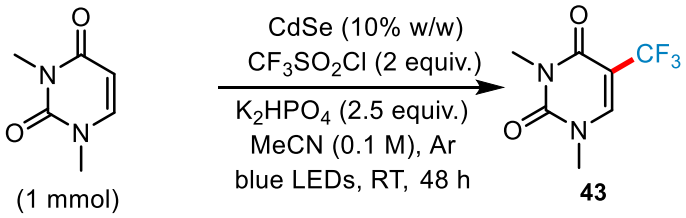
$$\phi_{(420 \pm 5\ \text{nm})} = \frac{5.7 \times 10^{-5}\ \text{mol}}{3.51348 \times 10^{-7}\ \text{Einstein/s} \times 900\ \text{s} \times 1} = 0.18$$

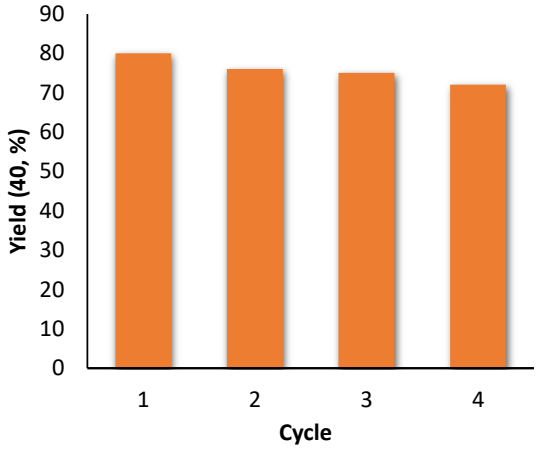
General procedure for high mmol scale reaction



A clean, oven-dried 50 mL screw cap reaction tube equipped with a Teflon-coated magnetic stir bar was charged with pentoxifylline (834 mg, 3 mmol, 1 equiv.), CdSe (83.4 mg, 10% w/w) and K_2HPO_4 (1.30 g, 7.5 mmol, 2.5 equiv.). The reaction tube was capped with a rubber septum, evacuated and backfilled with argon (3 times). Then, dry MeCN (30 mL) and triflyl chloride (639 µL, 6 mmol, 2 equiv.) were added by syringe and the vial was sealed with parafilm under argon atmosphere. The reaction mixture was stirred at room temperature for 48 h under irradiation with 34 W blue LEDs (3 cm away from blue LEDs) with fan cooling. After completion, the reaction mixture was quenched with EA and transferred to a centrifuge tube, and centrifuged (6000 rpm for 10 min) until the solid precipitates. Then, the solid was washed with EA (4 × 15 mL) and centrifuged in order to get complete reaction mixture. The combined organic liquids were collected, and concentrated *in vacuo*. The residue was purified by column chromatography using silica gel (100-200 mesh size) and EA/petroleum ether as the eluent. Finally, the CdSe in the centrifuge tube was washed three times (in the following sequence, 3 × water, 3 × EtOH), and the recovered photocatalyst was dried at 125 ºC for 12 h. The yield of product **49** is 706 mg (68%) and recovered catalyst is 74.6 mg.

General procedure for recycling process-I

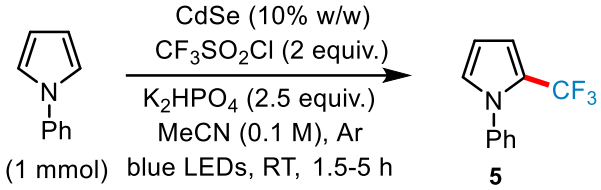


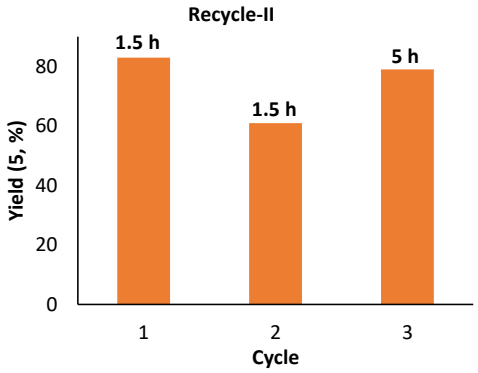


A clean, oven-dried screw cap reaction tube equipped with a Teflon-coated magnetic stir bar was charged with *N*,*N*-dimethyluracil (140 mg, 1 mmol, 1 equiv.), CdSe (14 mg, 10% w/w) and K_2HPO_4 (435 mg, 2.5 mmol, 2.5 equiv.). The reaction tube was capped with a rubber septum, evacuated and backfilled with argon (3 times). Then, dry MeCN (10 mL) and triflyl chloride (213 µL, 2 mmol, 2 equiv.) were added by syringe and the vial was sealed with parafilm under argon

atmosphere. The reaction mixture was stirred at room temperature for 48 h under irradiation with 34 W blue LEDs (3 cm away from blue LEDs) with fan cooling. After completion, yield was determined by ^{19}F NMR analysis of the crude mixture using trifluoromethylbenzene as an internal standard. The reaction mixture was quenched with EA and transferred to a centrifuge tube, and centrifuged (6000 rpm for 5 min) until the solid precipitates. Then, the solid was washed with EA (2 × 5 mL) and centrifuged in order to remove the complete organic compounds. Finally, the solid was washed with brine solution (2 × 5 mL) and EtOH (3 × 5 mL) and centrifuged (6000 rpm for 5 min) until the CdSe precipitates. The recovered photocatalyst was dried at 125 ºC for 12 h and used for next catalytic cycle.

General procedure for recycling process-II

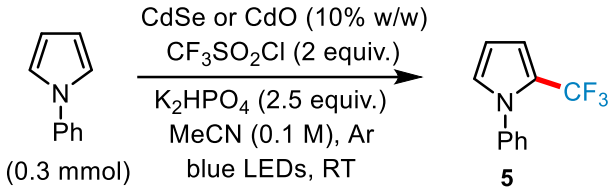


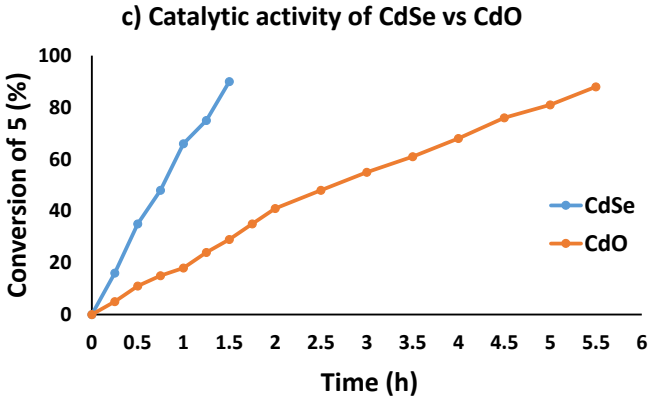


A clean, oven-dried screw cap reaction tube equipped with a Teflon-coated magnetic stir bar was charged with 1-phenyl-1*H*-pyrrole (143 mg, 1 mmol, 1 equiv.), CdSe (14.3 mg, 10% w/w) and K_2HPO_4 (435 mg, 2.5 mmol, 2.5 equiv.). The reaction tube was capped with a rubber septum, evacuated and backfilled with argon (3 times). Then, dry MeCN (10 mL) and triflyl chloride (213 µL, 2 mmol, 2 equiv.) were added by syringe and the vial was sealed with parafilm under argon atmosphere. The reaction mixture was stirred at room temperature for 1.5 h (cycle-1 and cycle-2) under irradiation with 34 W blue LEDs (3 cm away from blue LEDs) with fan cooling. After completion, yield was determined by ^{19}F NMR analysis of the crude mixture using trifluoromethylbenzene as an internal standard. The reaction mixture was quenched with EA and transferred to a centrifuge tube, and centrifuged (6000 rpm for 5 min) until the solid precipitates. Then, the solid was washed with EA (2 × 5 mL) and centrifuged in order to remove the complete organic compounds. Finally, the solid was washed with brine solution (2 × 5 mL) and EtOH (3 × 5 mL) and centrifuged (6000 rpm for 5 min) until the CdSe precipitates. The recovered

photocatalyst was dried at 125 ºC for 12 h and used for next catalytic cycle. The third cycle reaction mixture was carried out for 5 h to get high conversion.

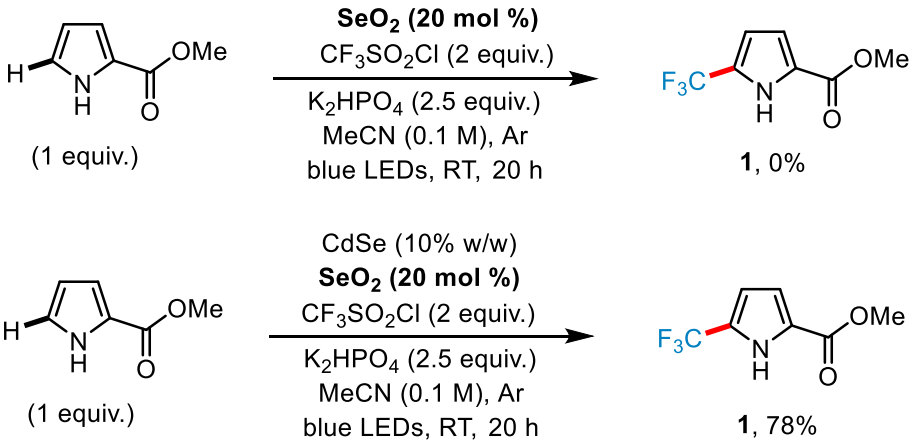
Kinetic profile of CdSe and CdO





A clean, oven-dried screw cap reaction tube equipped with a Teflon-coated magnetic stir bar was charged with 1-phenyl-1*H*-pyrrole (42.9 mg, 0.3 mmol, 1 equiv.), CdSe (4.3 mg, 10% w/w) or CdO (4.3 mg, 10% w/w) and K_2HPO_4 (130.5 mg, 0.75 mmol, 2.5 equiv.). The reaction tube was capped with a rubber septum, evacuated and backfilled with argon (3 times). Then, dry MeCN (3 mL) and triflyl chloride (64 µL, 0.6 mmol, 2 equiv.) were added by syringe and the vial was sealed with parafilm under argon atmosphere. The reaction mixture was stirred at room temperature under irradiation with 34 W blue LEDs (3 cm away from blue LEDs) with fan cooling. Every 15 min time interval, the yield of the reaction was determined by ^{19}F NMR analysis of the crude mixture using trifluoromethylbenzene as an internal standard.

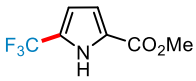
Role of selenium oxide:



Based on the reaction performed with catalytic amount of selenium(IV) oxide in our reaction condition, we observed that selenium oxide does not play any role. The reactions yields were determined by ^{19}F NMR analysis of the crude mixture using trifluoromethylbenzene as an internal standard.

Characterization Data:

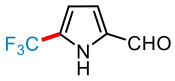
Methyl 5-(trifluoromethyl)-1*H*-pyrrole-2-carboxylate (1)



The title compound was synthesized according to the general procedure A employing CdSe (3.7 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), methyl 1*H*-pyrrole-2-carboxylate (37.5 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 20 h. The product was purified by column chromatography (silica gel, gradient 1 to 10% diethyl ether/petroleum ether). Yield = 70% (40.5 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.50 (s, 1H), 6.90 – 6.86 (m, 1H), 6.60 – 6.58 (m, 1H), 3.92 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.65, 125.24, 125.04 (q, $J_{C\text{-}F}$ = 40.4 Hz), 120.56 (q, $J_{C\text{-}F}$ = 268.6 Hz), 115.12, 111.0 (d, $J_{C\text{-}F}$ = 3.0 Hz), 52.29. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 60.43. **GCMS (EI)** *m/z* calcd. for $C_7H_6F_3NO_2$ [M^+] 193.0, found 193.0, 174.0, 162.0, 154.0, 142.0, 133.0, 123.0, 114.0, 107.0, 88.0, 69.0. The analytical data correspond with those reported in the literature.[3]

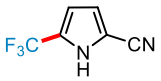
5-(Trifluoromethyl)-1*H*-pyrrole-2-carbaldehyde (2)



The title compound was synthesized according to the general procedure A employing CdSe (2.8 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1*H*-pyrrole-2-carbaldehyde (28.5 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 10 h. The product was purified by column chromatography (silica gel, gradient 1 to 10% diethyl ether/petroleum ether). Yield = 68% (33.2 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.26 (s, 1H), 9.65 (s, 1H), 7.02 – 6.95 (m, 1H), 6.76 – 6.63 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 180.65, 133.97, 127.42 (q, $J_{C\text{-}F}$ = 40.2 Hz), 120.39 (q, $J_{C\text{-}F}$ = 268.2 Hz), 120.12, 111.44 (q, $J_{C\text{-}F}$ = 2.9 Hz). **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 60.92. **GCMS (EI)** *m/z* calcd. for $C_6H_4F_3NO$ [M^+] 163.0, found 163.0, 142.0, 114.0, 107.0, 88.0, 75.0, 69.0, 64.0. The analytical data correspond with those reported in the literature.[4]

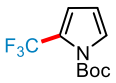
5-(Trifluoromethyl)-1*H*-pyrrole-2-carbonitrile (3)



The title compound was synthesized according to the general procedure A employing CdSe (2.7 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1*H*-pyrrole-2-carbonitrile (27.6 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 10 h. The product was purified by column chromatography (silica gel, gradient 1 to 10% diethyl ether/petroleum ether). Yield = 74% (35.5 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 1H), 6.93 – 6.82 (m, 1H), 6.72 – 6.53 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 125.71 (q, J_{C-F} = 40.9 Hz), 120.29, 119.85 (q, J_{C-F} = 268.0 Hz), 112.84, 110.88 (q, J_{C-F} = 3.0 Hz), 103.90. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 60.56. **GCMS (EI)** *m/z* calcd. for $C_6H_3F_3N_2$ [M^+] 160.0, found 160.0, 140.0, 110.0, 89.0, 69.0, 64.0, 57.0, 52.0, 37.0.

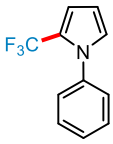
***tert*-Butyl 2-(trifluoromethyl)-1*H*-pyrrole-1-carboxylate (4)**



The title compound was synthesized according to the general procedure A employing CdSe (5.0 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), *tert*-butyl 1*H*-pyrrole-1-carboxylate (50.1 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 3 h. The product was purified by column chromatography (silica gel, gradient 1 to 10% diethyl ether/petroleum ether). Yield = 90% (63.4 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (dd, *J* = 3.3, 1.9 Hz, 1H), 6.73 (dd, *J* = 3.6, 1.9 Hz, 1H), 6.19 (t, *J* = 3.5 Hz, 1H), 1.61 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 147.57, 125.93 (d, J_{C-F} = 2.3 Hz), 121.89 (q, J_{C-F} = 40.2 Hz), 120.68 (q, J_{C-F} = 266.3 Hz), 117.93 (q, J_{C-F} = 4.5 Hz), 109.76, 85.78, 27.85. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 58.31. **GCMS (EI)** *m/z* calcd. for $C_{10}H_{12}F_3NO_2$ [M^+] 235.1, found 235.0, 220.0, 200.0, 176.0, 162.0, 142.0, 135.0, 114.0, 107.0, 88.0, 69.0, 57.1. The analytical data correspond with those reported in the literature.[3]

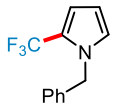
1-Phenyl-2-(trifluoromethyl)-1*H*-pyrrole (5)



The title compound was synthesized according to the general procedure A employing CdSe (4.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1-phenyl-1*H*-pyrrole (42.9 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 1.5 h. The product was purified by column chromatography (silica gel, gradient 1 to 10% diethyl ether/petroleum ether). Yield = 81% (51.2 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.43 (m, 3H), 7.39-7.37 (m, 2H), 6.89 (t, *J* = 2.4 Hz, 1H), 6.74 – 6.73 (m, 1H), 6.28 (t, *J* = 3.3 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 139.24, 129.12, 128.62, 127.38 (q, J_{C-F} = 3.0 Hz), 126.64, 122.35 (q, J_{C-F} = 38.4 Hz), 121.33 (q, J_{C-F} = 267.6 Hz), 112.86 (q, J_{C-F} = 3.0 Hz), 108.36. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 55.87. **GCMS (EI)** *m/z* calcd. for $C_{11}H_8F_3N$ [M^+] 211.0, found 211.1, 191.1, 172.1, 164.1, 158.0, 142.1, 133.1, 115.1, 105.6, 88.0, 77.1, 51.1. The analytical data correspond with those reported in the literature.[5]

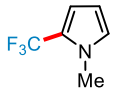
1-Benzyl-2-(trifluoromethyl)-1*H*-pyrrole (6)



The title compound was synthesized according to the general procedure A employing CdSe (4.7 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1-benzyl-1*H*-pyrrole (47.1 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 1 h. The product was purified by column chromatography (silica gel, gradient 1 to 10% diethyl ether/petroleum ether). Yield = 72% (48.6 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.34 (m, 3H), 7.18 (d, *J* = 7.1 Hz, 2H), 6.78 (t, *J* = 2.3 Hz, 1H), 6.70 (d, *J* = 3.7 Hz, 1H), 6.24 (t, *J* = 3.3 Hz, 1H), 5.24 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 137.07, 128.88, 128.02, 127.15, 126.09 (q, $J_{C\text{-}F}$ = 2.3 Hz), 121.71 (q, $J_{C\text{-}F}$ = 266.6 Hz), 121.38 (q, $J_{C\text{-}F}$ = 38.1 Hz), 111.90 (q, $J_{C\text{-}F}$ = 3.7 Hz), 108.13, 51.51. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 57.51. **GCMS (EI)** *m/z* calcd. for $C_{12}H_{10}F_3N$ [M^+] 225.1, found 225.1, 206.1, 154.0, 128.1, 91.1, 89.1, 77.1, 69.1, 65.1, 63.1.

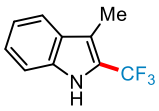
1-Methyl-2-(trifluoromethyl)-1*H*-pyrrole (7)



The title compound was synthesized according to the general procedure A employing CdSe (2.4 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), 1-methyl-1*H*-pyrrole (24.3 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 1 h. The reaction mixture was purified by prep TLC using 15% DCM in petroleum ether to provide the title compound. Yield = 89% (yield determined by ^{19}F NMR analysis due to the high volatility of the desired product).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.71 (d, *J* = 2.3 Hz, 1H), 6.56 (d, *J* = 3.5 Hz, 1H), 6.09 (dd, *J* = 4.2, 2.2 Hz, 1H), 3.72 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 126.73, 121.64 (q, $J_{C\text{-}F}$ = 267.1 Hz), 121.13 (q, $J_{C\text{-}F}$ = 38.1 Hz), 111.67 (q, $J_{C\text{-}F}$ = 3.5 Hz), 107.31, 34.90. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 58.83. **GCMS (EI)** *m/z* calcd. for $C_6H_6F_3N$ [M^+] 149.0, found 149.1, 130.1, 99.1, 78.1, 75.0, 69.0, 64.1, 57.1. The analytical data correspond with those reported in the literature.[6]

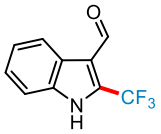
3-Methyl-2-(trifluoromethyl)-1*H*-indole (8)



The title compound was synthesized according to the general procedure A employing CdSe (3.9 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), 3-methyl-1*H*-indole (39.3 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 1 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 75% (44.7 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.66 (d, *J* = 8.0 Hz, 1H), 7.36 (dt, *J* = 15.0, 8.1 Hz, 2H), 7.21 (t, *J* = 7.4 Hz, 1H), 2.46 (q, *J* = 1.9 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 135.31, 128.19, 124.89, 122.25 (q, $J_{C\text{-}F}$ = 269.6 Hz), 121.66 (q, $J_{C\text{-}F}$ = 36.8 Hz), 120.51, 120.22, 114.20 (d, $J_{C\text{-}F}$ = 3.0 Hz), 111.69, 8.46. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 58.61. **GCMS (EI)** *m/z* calcd. for $C_{10}H_8F_3N$ [M^+] 199.1, found 199.1, 178.1, 158.0, 130.1, 102.0, 89.4, 77.0, 65.0, 51.0. The analytical data correspond with those reported in the literature.[7]

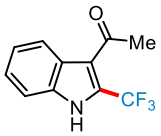
2-(Trifluoromethyl)-1*H*-indole-3-carbaldehyde (9)



The title compound was synthesized according to the general procedure A employing CdSe (4.3 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1*H*-indole-3-carbaldehyde (43.5 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 8 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 58% (37 mg).

^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 13.43 (s, 1H), 10.24 (s, 1H), 8.25 (d, *J* = 8.0 Hz, 1H), 7.61 (d, *J* = 8.2 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 1H), 7.36 (t, *J* = 7.5 Hz, 1H). **^{13}C NMR (101 MHz, DMSO-*d*$_6$)** δ 184.22, 135.35, 130.87 (q, $J_{C\text{-}F}$ = 39.0 Hz), 125.80, 124.32, 123.83, 121.98, 120.78 (q, $J_{C\text{-}F}$ = 270.4 Hz), 115.43, 113.18. **^{19}F NMR (377 MHz, DMSO-*d*$_6$)** δ – 55.46. **GCMS (EI)** *m/z* calcd. for $C_{10}H_6F_3NO$ [M^+] 213.0, found 213.1, 212.1, 192.0, 184.0, 164.0, 144.0, 134.0, 114.0, 107.0, 96.0, 88.0, 75.0. The analytical data correspond with those reported in the literature.[8]

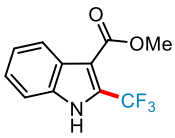
1-(2-(Trifluoromethyl)-1*H*-indol-3-yl)ethan-1-one (10)



The title compound was synthesized according to the general procedure A employing CdSe (4.7 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1-(1*H*-indol-3-yl)ethan-1-one (47.7 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 8 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 60% (40.8 mg).

^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 13.07 (s, 1H), 8.09 (d, *J* = 8.1 Hz, 1H), 7.60 (d, *J* = 8.2 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.33 (t, *J* = 7.6 Hz, 1H), 2.65 (s, 3H). **^{13}C NMR (101 MHz, DMSO-*d*$_6$)** δ 192.60, 134.67, 126.77 (q, $J_{C\text{-}F}$ = 38.3 Hz), 125.26, 124.81, 122.96, 121.86, 120.91 (q, $J_{C\text{-}F}$ = 269.9 Hz), 116.18, 113.24, 30.90. **^{19}F NMR (377 MHz, DMSO-*d*$_6$)** δ – 57.47. **GCMS (EI)** *m/z* calcd. for $C_{11}H_8F_3NO$ [M^+] 227.1, found 227.1, 212.1, 192.0, 178.1, 164.0, 144.0, 128.1, 114.0, 96.0, 75.0, 63.0.

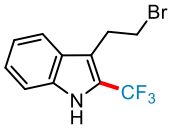
Methyl 2-(trifluoromethyl)-1*H*-indole-3-carboxylate (11)



The title compound was synthesized according to the general procedure A employing CdSe (5.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), methyl 1*H*-indole-3-carboxylate (52.5 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 8 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 64% (46.6 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.09 (s, 1H), 8.26 (d, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.41 – 7.32 (m, 2H), 3.98 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.69, 133.91, 129.04 (q, $J_{C\text{-}F}$ = 38.8 Hz), 126.61, 125.67, 123.38, 123.00, 120.52 (q, $J_{C\text{-}F}$ = 270.0 Hz), 112.07, 108.41, 51.84. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 59.86. **GCMS (EI)** *m/z* calcd. for $C_{11}H_8F_3NO_2$ [M^+] 243.1, found 243.1, 212.1, 192.0, 184.0, 164.0, 144.0, 134.1, 114.0, 107.0, 96.0, 87.0. The analytical data correspond with those reported in the literature.[9]

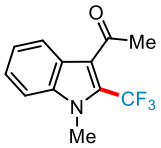
3-(2-Bromoethyl)-2-(trifluoromethyl)-1*H*-indole (12)



The title compound was synthesized according to the general procedure A employing CdSe (6.6 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 3-(2-bromoethyl)-1*H*-indole (66.9 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 1 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 69% (60.2 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (s, 1H), 7.75 (d, *J* = 8.1 Hz, 1H), 7.48 (d, *J* = 8.2 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.37 – 7.25 (m, 1H), 3.73 – 3.59 (m, 2H), 3.56 – 3.45 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 135.22, 127.11, 125.27, 122.38 (q, J_{C-F} = 37.0 Hz), 121.88 (q, J_{C-F} = 268.9 Hz), 121.13, 120.07, 112.01, 115.45 (d, J_{C-F} = 3.0 Hz), 31.40, 27.91. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 58.34. **GCMS (EI)** *m/z* calcd. for $C_{11}H_9BrF_3N$ [M^+] 291.0, found 293.0, 291.0, 272.0, 212.1, 198.1, 190.1, 178.1, 172.1, 158.1, 152.1, 143.1, 128.1, 115.1.

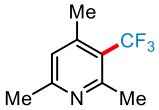
1-(1-Methyl-2-(trifluoromethyl)-1*H*-indol-3-yl)ethan-1-one (13)



The title compound was synthesized according to the general procedure A employing CdSe (5.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1-(1-methyl-1*H*-indol-3-yl)ethan-1-one (51.9 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 8 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 68% (49.1 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.1 Hz, 1H), 7.47 – 7.37 (m, 2H), 7.29 (ddd, *J* = 8.1, 5.4, 2.6 Hz, 1H), 3.90 (q, *J* = 1.3 Hz, 3H), 2.64 (q, *J* = 1.4 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 196.64, 137.26, 125.72 (q, J_{C-F} = 37.5 Hz), 125.60, 124.76, 122.79, 122.09, 121.25 (q, J_{C-F} = 270.3 Hz), 120.04, 110.21, 32.05 (d, J_{C-F} = 3.3 Hz), 31.79 (d, J_{C-F} = 3.5 Hz). **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 54.93. **GCMS (EI)** *m/z* calcd. for $C_{12}H_{10}F_3NO$ [M^+] 241.1, found 241.1, 226.1, 222.1, 211.0, 198.0, 183.0, 178.0, 169.0, 158.0, 151.0, 128.1, 113.0, 101.1, 75.1.

2,4,6-Trimethyl-3-(trifluoromethyl)pyridine (14)

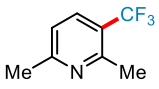


The title compound was synthesized according to the general procedure A employing CdSe (3.6 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 2,4,6-trimethylpyridine (36.3 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 35 h. The reaction mixture was purified by prep TLC using 20% DCM in petroleum ether to provide the title compound. Yield = 80% (yield determined by ^{19}F NMR analysis due to the high volatility of the desired product).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.87 (s, 1H), 2.63 (q, *J* = 3.1 Hz, 3H), 2.47 (s, 3H), 2.40 (q, *J* = 3.1 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 159.98, 156.57, 147.21, 125.64 (q, J_{C-F} = 274.9 Hz), 124.46, 121.42 (q, J_{C-F} = 29.9 Hz), 24.24 (q, J_{C-F} = 3.8 Hz), 23.93, 20.98 (q, J_{C-F} = 3.8 Hz). **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 54.35. **GCMS (EI)** *m/z* calcd. for $C_9H_{10}F_3N$ [M^+] 189.1, found

189.1, 174.0, 169.1, 154.1, 148.0, 139.1, 133.0, 127.0, 120.1, 101.0, 77.0, 40.0. The analytical data correspond with those reported in the literature.[7]

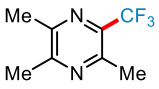
2,6-Dimethyl-3-(trifluoromethyl)pyridine (15)



The title compound was synthesized according to the general procedure A employing CdSe (3.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 2,6-dimethylpyridine (32.1 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 48 h. The reaction mixture was purified by prep TLC using 25% DCM in petroleum ether to provide the title compound. Yield = 75% (yield determined by ^{19}F NMR analysis due to the high volatility of the desired product).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.1 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 1H), 2.67 (s, 3H), 2.58 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.29, 156.45, 134.25 (d, $J_{C\text{-}F}$ = 5.2 Hz), 124.42 (q, $J_{C\text{-}F}$ = 272.2 Hz), 122.24 (q, $J_{C\text{-}F}$ = 31.6 Hz), 120.27, 24.57, 22.72. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 61.90. **GCMS (EI)** *m/z* calcd. for $C_8H_8F_3N$ [M^+] 175.1, found 175.0, 160.0, 156.0, 140.0, 133.0, 125.0, 114.0, 107.1, 92.0, 77.0. The analytical data correspond with those reported in the literature.[7]

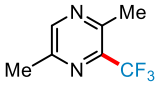
2,3,5-Trimethyl-6-(trifluoromethyl)pyrazine (16)



The title compound was synthesized according to the general procedure A employing CdSe (3.6 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 2,3,5-trimethylpyrazine (36.6 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 48 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 72% (41 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 2.63 (q, *J* = 2.0 Hz, 3H), 2.56 (s, 3H), 2.54 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 154.95, 148.85, 148.63, 138.49 (q, $J_{C\text{-}F}$ = 34.1 Hz), 122.40 (q, $J_{C\text{-}F}$ = 274.3 Hz), 22.09, 21.41, 20.91 (q, $J_{C\text{-}F}$ = 2.5 Hz). **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 65.18. **GCMS (EI)** *m/z* calcd. for $C_8H_9F_3N_2$ [M^+] 190.1, found 190.1, 171.1, 149.0, 121.1, 107.0, 88.0, 80.1, 69.0, 57.0, 42.1.

2,5-Dimethyl-3-(trifluoromethyl)pyrazine (17)



The title compound was synthesized according to the general procedure A employing CdSe (3.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 2,5-dimethylpyrazine (32.4 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 48 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 67% (35.3 mg).

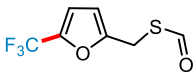
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (s, 1H), 2.67 (d, *J* = 2.3 Hz, 3H), 2.57 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 150.31, 149.37, 146.32, 140.92 (q, $J_{C\text{-}F}$ = 34.1 Hz), 122.05 (q, $J_{C\text{-}F}$ = 274.8 Hz), 21.00 (d, $J_{C\text{-}F}$ = 2.5 Hz), 20.95. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 65.90. **GCMS (EI)** *m/z* calcd. for $C_7H_7F_3N_2$ [M^+] 176.1, found 176.0, 157.1, 149.0, 107.0, 88.0, 80.1, 69.0, 57.0. The analytical data correspond with those reported in the literature.[7]

3,5-Dimethyl-2-(trifluoromethyl)pyrazine (18)

The title compound was synthesized according to the general procedure A employing CdSe (3.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 2,6-dimethylpyrazine (32.4 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 48 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 65% (34.3 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (s, 1H), 2.70 (q, *J* = 1.9 Hz, 3H), 2.61 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 156.37, 151.83, 140.45, 139.31 (q, $J_{C\text{-}F}$ = 34.4 Hz), 122.34 (q, $J_{C\text{-}F}$ = 274.3 Hz), 21.74, 21.49 (q, $J_{C\text{-}F}$ = 2.3 Hz). **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 65.50. **GCMS (EI)** *m/z* calcd. for $C_7H_7F_3N_2$ [M^+] 176.1, found 176.0, 157.0, 149.0, 135.0, 115.0, 107.0, 88.0, 69.0, 66.0. The analytical data correspond with those reported in the literature.[6]

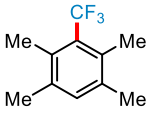
***S*-((5-(Trifluoromethyl)furan-2-yl)methyl)methanethioate (19)**



The title compound was synthesized according to the general procedure A employing CdSe (4.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), *S*-(furan-2-ylmethyl)methanethioate (42.6 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 48 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 68% (42.8 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.16 (s, 1H), 6.70 (dt, *J* = 2.6, 1.3 Hz, 1H), 6.40 – 6.21 (m, 1H), 4.24 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 185.81, 153.00, 141.62 (q, $J_{C\text{-}F}$ = 42.8 Hz), 118.98 (q, $J_{C\text{-}F}$ = 267.0 Hz), 112.77 (q, $J_{C\text{-}F}$ = 2.9 Hz), 109.37, 22.83. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 64.12. **GCMS (EI)** *m/z* calcd. for $C_7H_5F_3O_2S$ [M^+] 210.0, found 210.0, 191.0, 182.0, 149.0, 130.0, 121.0, 111.0, 99.0, 79.0, 69.0, 51.1.

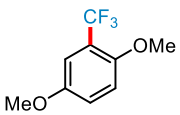
1,2,4,5-Tetramethyl-3-(trifluoromethyl)benzene (20)



The title compound was synthesized according to the general procedure A employing CdSe (4.0 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1,2,4,5-tetramethylbenzene (40.2 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 30 h. The product was purified by column chromatography (silica gel, gradient 100% petroleum ether). Yield = 66% (40 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (s, 1H), 2.33 (q, *J* = 3.3 Hz, 6H), 2.26 (s, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 135.24, 134.68, 133.65, 128.18 (q, $J_{C\text{-}F}$ = 27.0 Hz), 126.48 (q, $J_{C\text{-}F}$ = 277.1 Hz), 20.70, 16.78 (q, $J_{C\text{-}F}$ = 4.7 Hz). **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 51.74. **GCMS (EI)** *m/z* calcd. for $C_{11}H_{13}F_3$ [M^+] 202.1, found 202.1, 187.1, 159.1, 147.1, 133.1, 115.1, 103.1, 91.1, 77.1, 65.0.

1,4-Dimethoxy-2-(trifluoromethyl)benzene (21)

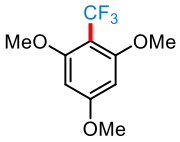


The title compound was synthesized according to the general procedure A employing CdSe (4.1 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1,4-dimethoxybenzene (41.4 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and

CH_3CN (0.1 M, 3 mL) for 35 h. The reaction mixture was purified by prep TLC using 5% DCM in petroleum ether to provide the title compound. Yield = 80% (yield determined by ^{19}F NMR analysis due to the high volatility of the desired product).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, *J* = 3.0 Hz, 1H), 7.02 (dd, *J* = 9.0, 3.0 Hz, 1H), 6.94 (d, *J* = 9.0 Hz, 1H), 3.86 (s, 3H), 3.79 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 152.95, 151.53, 123.43 (q, $J_{C\text{-}F}$ = 272.2 Hz), 119.37 (q, $J_{C\text{-}F}$ = 30.9 Hz), 118.08, 113.57, 112.82 (q, $J_{C\text{-}F}$ = 5.5 Hz), 56.55, 55.87. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 62.41. **GCMS (EI)** *m/z* calcd. for $C_9H_9F_3O_2$ [M^+] 206.1, found 206.1, 191.1, 163.1, 157.0, 148.0, 129.0, 120.1, 101.0, 95.1. The analytical data correspond with those reported in the literature.[7]

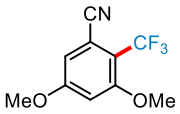
1,3,5-Trimethoxy-2-(trifluoromethyl)benzene (22)



The title compound was synthesized according to the general procedure A employing CdSe (5.0 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1,3,5-trimethoxybenzene (50.4 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 3 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% diethyl ether/petroleum ether). Yield = 81% (57.3 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.12 (s, 2H), 3.83 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.64, 160.51, 124.49 (q, $J_{C\text{-}F}$ = 273.2 Hz), 100.40 (q, $J_{C\text{-}F}$ = 30.1 Hz), 91.33, 56.33, 55.47. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 54.08. **GCMS (EI)** *m/z* calcd. for $C_{10}H_{11}F_3O_3$ [M^+] 236.1, found 236.1, 207.1, 193.1, 176.1, 163.1, 139.1, 120.0, 107.0, 95.1, 69.0. The analytical data correspond with those reported in the literature.[3]

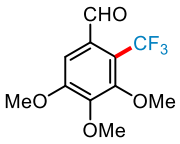
3,5-Dimethoxy-2-(trifluoromethyl)benzonitrile (23)



The title compound was synthesized according to the general procedure A employing CdSe (4.8 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 3,5-dimethoxybenzonitrile (48.9 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 48 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% diethyl ether/petroleum ether). Overall yield: 70% (80:20, r.r., the yield and ratio were determined by ^{19}F NMR analysis). Isolated yield of reported compound **23** is 51% (35.3 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.81 (d, *J* = 2.4 Hz, 1H), 6.73 (s, 1H), 3.90 (s, 3H), 3.88 (s, 3H).**^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.14, 160.04, 122.59 (q, $J_{C\text{-}F}$ = 273.5 Hz), 116.34, 113.29 (q, $J_{C\text{-}F}$ = 31.6 Hz), 112.33 (q, $J_{C\text{-}F}$ = 3.0 Hz), 110.81, 103.86, 56.58, 56.13. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 57.82. **GCMS (EI)** *m/z* calcd. for $C_{10}H_8F_3NO_2$ [M^+] 231.1, found 231.1, 212.0, 201.0, 188.0, 182.0, 171.0, 158.0, 154.0, 145.0, 139.0, 132.0, 126.0, 121.0, 104.1, 88.0.

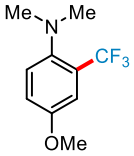
3,4,5-Trimethoxy-2-(trifluoromethyl)benzaldehyde (24)



The title compound was synthesized according to the general procedure A employing CdSe (5.8 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 3,4,5-trimethoxybenzaldehyde (58.8 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 24 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield: 77% (61 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.32 (q, *J* = 2.4 Hz, 1H), 7.34 (s, 1H), 3.95 (s, 6H), 3.93 (s, 3H).**^{13}C NMR (101 MHz, $CDCl_3$)** δ 189.31 (q, $J_{C\text{-}F}$ = 5.7 Hz), 155.92, 152.94, 147.47, 130.98, 124.30 (q, $J_{C\text{-}F}$ = 275.2 Hz), 118.09 (q, $J_{C\text{-}F}$ = 31.1 Hz), 107.40, 62.17, 61.16, 56.39. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 51.13. **GCMS (EI)** *m/z* calcd. for $C_{11}H_{11}F_3O_4$ [M^+] 264.1, found 264.1, 249.0, 229.0, 193.0, 178.0, 163.0, 135.0, 119.0, 107.0, 87.0, 69.0, 57.0. The analytical data correspond with those reported in the literature.[10]

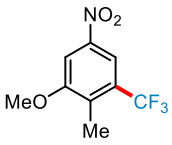
4-Methoxy-*N*,*N*-dimethyl-2-(trifluoromethyl)aniline (25)



The title compound was synthesized according to the general procedure A employing CdSe (4.5 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 4-methoxy-*N*,*N*-dimethylaniline (45.3 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 24 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% diethyl ether/petroleum ether). Yield: 70% (46 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.9 Hz, 1H), 7.12 (s, 1H), 7.04 (dd, *J* = 8.8, 3.0 Hz, 1H), 3.82 (s, 3H), 2.67 (s, 6H).**^{13}C NMR (101 MHz, $CDCl_3$)** δ 156.39, 127.94 (d, $J_{C\text{-}F}$ = 28.8 Hz), 125.07, 123.86 (q, $J_{C\text{-}F}$ = 274.7 Hz),118.20, 112.29 (d, $J_{C\text{-}F}$ = 7.0 Hz), 55.79, 46.59. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 60.68. **GCMS (EI)** *m/z* calcd. for $C_{10}H_{12}F_3NO$ [M^+] 219.1, found 219.1, 204.1, 188.0, 176.1, 160.0, 145.0, 132.0, 115.1, 101.0, 92.1, 75.1, 63.1.

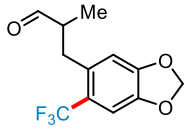
1-Methoxy-2-methyl-5-nitro-3-(trifluoromethyl)benzene (26)



The title compound was synthesized according to the general procedure A employing CdSe (5.0 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 2-methoxy-1-methyl-4-nitrobenzene (50.1 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 24 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield: 43% (30 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (s, 1H), 7.33 (s, 1H), 3.95 (s, 3H), 2.31 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 160.26, 147.34, 132.44, 130.60, 129.54 (q, $J_{C\text{-}F}$ = 5.4 Hz), 122.47 (q, $J_{C\text{-}F}$ = 273.2 Hz), 115.56 (q, $J_{C\text{-}F}$ = 34.1 Hz), 56.36, 16.44. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 58.90. **GCMS (EI)** *m/z* calcd. for $C_9H_8F_3NO_3$ [M^+] 235.0, found 235.0, 189.1, 174.0, 127.0, 109.0, 96.0, 91.0, 77.1, 75.1, 51.1.

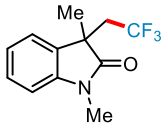
2-Methyl-3-(6-(trifluoromethyl)benzo[*d*][1,3]dioxol-5-yl)propanal (27)



The title compound was synthesized according to the general procedure A employing CdSe (5.7 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 3-(benzo[*d*][1,3]dioxol-5-yl)-2-methylpropanal (57.6 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 24 h. The product was purified by column chromatography (silica gel, gradient 15 to 20% diethyl ether/petroleum ether). Yield: 61% (47.5 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.68 (s, 1H), 7.07 (s, 1H), 6.75 (s, 1H), 6.02 (s, 2H), 3.20 (q, *J* = 7.5 Hz, 1H), 2.70 – 2.62 (m, 2H), 1.12 (d, *J* = 5.8 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 203.71, 150.29, 146.40, 133.13, 124.30 (q, $J_{C\text{-}F}$ = 36.4 Hz), 111.49, 109.71 (q, $J_{C\text{-}F}$ = 252.2 Hz), 106.76 (d, $J_{C\text{-}F}$ = 6.3 Hz), 102.15, 48.10, 32.91, 13.64. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 57.73. **GCMS (EI)** *m/z* calcd. for $C_{12}H_{11}F_3O_3$ [M^+] 260.1, found 260.1, 232.0, 203.0, 190.0, 173.0, 145.0, 133.1, 127.0, 119.0, 103.0.

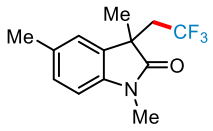
1,3-Dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (28)



The title compound was synthesized according to the general procedure B employing CdSe (5.2 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), *N*-methyl-*N*-phenylmethacrylamide (52.5 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 14 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield: 72% (52.4 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (t, *J* = 7.7 Hz, 1H), 7.26 (d, *J* = 7.3 Hz, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.88 (d, *J* = 7.8 Hz, 1H), 3.23 (s, 3H), 2.82 (dq, *J* = 15.1, 10.7 Hz, 1H), 2.65 (dq, *J* = 15.2, 10.5 Hz, 1H), 1.40 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 178.60, 142.95, 131.10, 128.63, 125.35 (q, $J_{C\text{-}F}$ = 278.3 Hz), 123.65, 122.76, 108.57, 44.50 (d, $J_{C\text{-}F}$ = 2.3 Hz), 40.74 (q, $J_{C\text{-}F}$ = 28.2 Hz), 26.55, 25.11. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 61.96 (t, *J* = 10.2 Hz). **GCMS (EI)** *m/z* calcd. for $C_{12}H_{12}F_3NO$ [M^+] 243.1, found 243.1, 228.1, 208.1, 200.1, 160.1, 145.1, 130.1, 117.1, 103.1, 89.3, 77.1, 65.1. The analytical data correspond with those reported in the literature.[11]

1,3,5-Trimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (29)

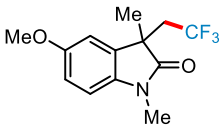


The title compound was synthesized according to the general procedure B employing CdSe (5.6 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), *N*-methyl-*N*-(*p*-tolyl)methacrylamide (56.7 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 14 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield: 93% (71.7 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (d, *J* = 8.0 Hz, 1H), 7.07 (s, 1H), 6.76 (d, *J* = 7.9 Hz, 1H), 3.21 (s, 3H), 2.80 (dq, *J* = 15.1, 10.7 Hz, 1H), 2.62 (dq, *J* = 15.2, 10.5 Hz, 1H), 2.35 (s, 3H), 1.38 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 178.54, 140.58, 132.29, 131.19, 128.87, 125.39 (q, $J_{C\text{-}F}$ = 278.3 Hz), 124.45, 108.27, 44.55 (d, $J_{C\text{-}F}$ = 2.6 Hz), 40.74 (q, $J_{C\text{-}F}$ = 28.2 Hz), 26.57, 25.16, 21.24. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 61.90 (t, *J* = 10.8 Hz). **GCMS (EI)** *m/z* calcd. for $C_{13}H_{14}F_3NO$

[M^+] 257.1, found 257.1, 242.1, 222.1, 174.1, 159.1, 144.1, 130.1, 115.1, 103.1, 91.1, 77.1, 71.7. The analytical data correspond with those reported in the literature.[11]

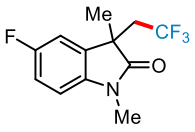
5-Methoxy-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (30)



The title compound was synthesized according to the general procedure B employing CdSe (6.1 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), *N*-(4-methoxyphenyl)-*N*-methylmethacrylamide (61.5 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 14 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield: 86% (70.4 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.86 (d, *J* = 2.3 Hz, 1H), 6.81 (dd, *J* = 8.5, 2.5 Hz, 1H), 6.76 (d, *J* = 8.4 Hz, 1H), 3.77 (s, 3H), 3.18 (s, 3H), 2.79 (dq, *J* = 15.2, 10.7 Hz, 1H), 2.61 (dq, *J* = 15.2, 10.5 Hz, 1H), 1.37 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 178.16, 156.13, 136.42, 132.45, 125.33 (q, J_{C-F} = 278.2 Hz), 112.66, 111.29, 108.78, 55.85, 44.84 (q, J_{C-F} = 2.2 Hz), 40.64 (q, J_{C-F} = 28.3 Hz), 26.53, 25.09.**^{19}F NMR (377 MHz, $CDCl_3$)** δ – 61.88 (t, *J* = 13.3 Hz). **GCMS (EI)** *m/z* calcd. for $C_{13}H_{14}F_3NO_2$ [M^+] 273.1, found 258.1, 238.1, 230.1, 210.1, 190.1, 175.1, 160.1, 147.1, 132.1, 118.1, 104.1, 91.1. The analytical data correspond with those reported in the literature.[11]

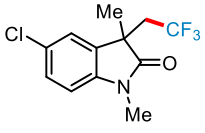
5-Fluoro-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (31)



The title compound was synthesized according to the general procedure B employing CdSe (5.7 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), *N*-(4-fluorophenyl)-*N*-methylmethacrylamide (57.9 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 14 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield: 75% (58.7 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 – 6.97 (m, 2H), 6.79 (dd, *J* = 9.3, 4.2 Hz, 1H), 3.21 (s, 3H), 2.81 (dq, *J* = 15.5, 10.7 Hz, 1H), 2.62 (dq, *J* = 15.4, 10.4 Hz, 1H), 1.39 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 178.22, 159.40 (d, J_{C-F} = 240.9 Hz), 138.92, 132.74 (d, J_{C-F} = 8.1 Hz), 125.23 (q, *J* = 278.2 Hz), 114.95 (d, J_{C-F} = 23.5 Hz), 111.89 (d, J_{C-F} = 24.9 Hz), 109.08 (d, J_{C-F} = 8.1 Hz), 44.93, 40.67 (q, J_{C-F} = 28.4 Hz), 26.68, 25.01. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 61.99 (t, *J* = 10.4 Hz), – 120.43 (d, *J* = 10.2 Hz). **GCMS (EI)** *m/z* calcd. for $C_{12}H_{11}F_4NO$ [M^+] 261.1, found 261.1, 246.1, 226.1, 193.1, 178.1, 163.1, 148.1, 135.1, 124.1, 95.1, 69.1. The analytical data correspond with those reported in the literature.[11]

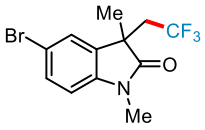
5-Chloro-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (32)



The title compound was synthesized according to the general procedure B employing CdSe (6.2 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), *N*-(4-chlorophenyl)-*N*-methylmethacrylamide (62.7 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 14 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield: 68% (56.5 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.23 (d, *J* = 2.0 Hz, 1H), 6.80 (d, *J* = 8.2 Hz, 1H), 3.21 (s, 3H), 2.82 (dq, *J* = 15.3, 10.6 Hz, 1H), 2.62 (dq, *J* = 15.2, 10.4 Hz, 1H), 1.39 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 178.05, 141.57, 132.80, 128.64, 128.21, 125.18 (q, $J_{C\text{-}F}$ = 278.2 Hz), 124.21, 109.53, 44.70 (d, $J_{C\text{-}F}$ = 2.6 Hz), 40.68 (q, $J_{C\text{-}F}$ = 28.4 Hz), 26.67, 25.03. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 61.96 (t, *J* = 10.8 Hz). **GCMS (EI)** *m/z* calcd. for $C_{12}H_{11}ClF_3NO$ [M^+] 277.0, found 279.1, 277.0, 264.0, 262.0, 244.0, 242.0, 196.0, 194.0, 179.0, 164.0, 159.1, 130.1, 115.1, 103.1, 101.0, 89.1. The analytical data correspond with those reported in the literature.[11]

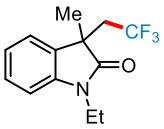
5-Bromo-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (33)



The title compound was synthesized according to the general procedure B employing CdSe (7.5 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), *N*-(4-bromophenyl)-*N*-methylmethacrylamide (75.9 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 14 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield: 72% (69.3 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.36 (s, 1H), 6.75 (d, *J* = 8.3 Hz, 1H), 3.20 (s, 3H), 2.81 (dqd, *J* = 15.2, 10.7, 1.4 Hz, 1H), 2.61 (dq, *J* = 15.2, 10.4 Hz, 1H), 1.38 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 177.90, 142.03, 133.14, 131.51, 126.90, 125.15 (q, $J_{C\text{-}F}$ = 278.3 Hz), 115.39, 110.03, 44.62 (d, $J_{C\text{-}F}$ = 2.4 Hz), 40.65 (q, $J_{C\text{-}F}$ = 28.5 Hz), 26.62, 25.02. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 61.95 (t, *J* = 10.4 Hz). **GCMS (EI)** *m/z* calcd. for $C_{12}H_{11}BrF_3NO$ [M^+] 321.0, found 323.0, 321.0, 308.0, 306.0, 288.0, 286.0, 240.0, 238.0, 208.0, 191.1, 159.1, 130.1, 115.1, 102.1, 89.1. The analytical data correspond with those reported in the literature.[11]

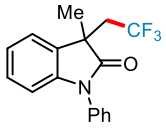
1-Ethyl-3-methyl-3-(2,2,2-trifluoroethyl)indolin-2-one (34)



The title compound was synthesized according to the general procedure B employing CdSe (5.6 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), *N*-ethyl-*N*-phenylmethacrylamide (56.7 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 14 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield: 90% (69.4 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.8 Hz, 1H), 7.25 (d, *J* = 6.8 Hz, 1H), 7.07 (t, *J* = 7.3 Hz, 1H), 6.89 (d, *J* = 7.8 Hz, 1H), 3.87 (dq, *J* = 14.5, 7.3 Hz, 1H), 3.68 (dq, *J* = 14.2, 7.2 Hz, 1H), 2.84 (dq, *J* = 15.2, 10.7 Hz, 1H), 2.63 (dq, *J* = 15.1, 10.4 Hz, 1H), 1.39 (s, 3H), 1.24 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 178.18, 142.00, 131.32, 128.54, 125.36 (q, $J_{C\text{-}F}$ = 278.2 Hz), 123.80, 122.49, 108.71, 44.39 (d, $J_{C\text{-}F}$ = 2.6 Hz), 40.76 (q, $J_{C\text{-}F}$ = 28.2 Hz), 34.87, 25.22, 12.34. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 61.93 (t, *J* = 9.8 Hz). **GCMS (EI)** *m/z* calcd. for $C_{13}H_{14}F_3NO$ [M^+] 257.1, found 257.1, 242.1, 222.1, 214.1, 194.1, 174.1, 146.1, 144.1, 130.1, 128.1, 117.1, 103.1, 89.1, 77.1. The analytical data correspond with those reported in the literature.[11]

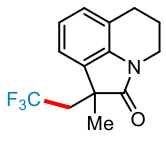
3-Methyl-1-phenyl-3-(2,2,2-trifluoroethyl)indolin-2-one (35)



The title compound was synthesized according to the general procedure B employing CdSe (7.1 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), *N*,*N*-diphenylmethacrylamide (71.1 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 14 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield: 86% (78.7 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (t, *J* = 7.6 Hz, 2H), 7.46 – 7.38 (m, 3H), 7.33 (d, *J* = 7.4 Hz, 1H), 7.24 (t, *J* = 7.5 Hz, 1H), 7.13 (t, *J* = 7.5 Hz, 1H), 6.85 (d, *J* = 7.9 Hz, 1H), 2.98 (dq, *J* = 15.2, 10.7 Hz, 1H), 2.74 (dq, *J* = 15.2, 10.4 Hz, 1H), 1.54 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 178.07, 143.02, 134.43, 130.76, 129.76, 128.54, 128.35, 126.70, 125.41 (q, $J_{C\text{-}F}$ = 278.3 Hz), 123.85, 123.16, 109.85, 44.60 (d, $J_{C\text{-}F}$ = 2.4 Hz), 41.15 (q, $J_{C\text{-}F}$ = 28.1 Hz), 25.54. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 61.83 (t, *J* = 10.4 Hz). **GCMS (EI)** *m/z* calcd. for $C_{17}H_{14}F_3NO$ [M^+] 305.1, found 305.1, 290.1, 270.1, 242.1, 222.1, 200.1, 194.1, 180.1, 167.1, 152.2, 130.1, 117.1, 96.1. The analytical data correspond with those reported in the literature.[11]

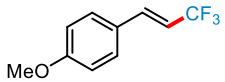
1-Methyl-1-(2,2,2-trifluoroethyl)-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinolin-2(1*H*)-one (36)



The title compound was synthesized according to the general procedure B employing CdSe (6.0 mg, 10% w/w), K_2CO_3 (103.5 mg, 0.75 mmol), 1-(3,4-dihydroquinolin-1(2*H*)-yl)-2-methylprop-2-en-1-one (60.3 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol) and CH_3CN (0.1 M, 3 mL) for 14 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% EA/petroleum ether). Yield: 88% (71 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (d, *J* = 7.4 Hz, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.96 (t, *J* = 7.5 Hz, 1H), 3.79 – 3.66 (m, 2H), 2.90 – 2.56 (m, 4H), 2.00 (p, *J* = 6.0 Hz, 2H), 1.41 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 177.43, 138.70, 129.77, 127.36, 125.51 (q, $J_{C\text{-}F}$ = 278.1 Hz), 122.16, 121.54, 120.59, 45.72 (d, $J_{C\text{-}F}$ = 2.6 Hz), 40.55 (q, $J_{C\text{-}F}$ = 28.3 Hz), 39.13, 24.69, 24.64, 21.21. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 61.81 (t, *J* = 11.8 Hz). **GCMS (EI)** *m/z* calcd. for $C_{14}H_{14}F_3NO$ [M^+] 269.1, found 269.1, 254.1, 234.1, 206.1, 200.1, 186.1, 158.1, 144.1, 130.1, 115.1, 103.0, 92.6, 77.1. The analytical data correspond with those reported in the literature.[11]

(*E*)-1-Methoxy-4-(3,3,3-trifluoroprop-1-en-1-yl)benzene (37)

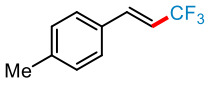


The title compound was synthesized according to the general procedure C employing CdSe (5.3 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), (*E*)-3-(4-methoxyphenyl)acrylic acid (53.4 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 28 h. The product was purified by column chromatography (silica gel, gradient 100% petroleum ether). Yield = 69% (41.8 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.7 Hz, 2H), 7.09 (dd, *J* = 16.1, 2.3 Hz, 1H), 6.91 (d, *J* = 8.7 Hz, 2H), 6.07 (dq, *J* = 16.1, 6.6 Hz, 1H), 3.84 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.18, 137.24 (q, $J_{C\text{-}F}$ = 6.8 Hz), 129.16, 126.18, 124.08 (q, $J_{C\text{-}F}$ = 268.6 Hz), 114.44, 113.50 (q, $J_{C\text{-}F}$ = 33.6 Hz), 55.46. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 62.83 (d, *J* = 5.6 Hz). **GCMS (EI)** *m/z*

calcd. for $C_{10}H_9F_3O$ [M^+] 202.1, found 202.1, 187.0, 183.1, 159.1, 151.1, 139.0, 133.1, 119.0, 109.1, 89.1, 83.1, 75.1, 63.1. The analytical data correspond with those reported in the literature.[12]

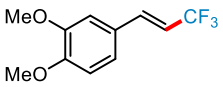
(*E*)-1-Methyl-4-(3,3,3-trifluoroprop-1-en-1-yl)benzene (38)



The title compound was synthesized according to the general procedure C employing CdSe (4.8 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), (*E*)-3-(*p*-tolyl)acrylic acid (48.6 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 28 h. The product was purified by column chromatography (silica gel, gradient 100% petroleum ether). Yield = 58% (32.3 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, *J* = 7.9 Hz, 2H), 7.20 (d, *J* = 7.9 Hz, 2H), 7.12 (dd, *J* = 16.1, 2.2 Hz, 1H), 6.15 (dq, *J* = 16.1, 6.6 Hz, 1H), 2.38 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.43, 137.67 (q, J_{C-F} = 6.8 Hz), 130.78, 129.77, 127.61, 123.92 (q, J_{C-F} = 268.6 Hz), 114.89 (q, J_{C-F} = 33.6 Hz), 21.53. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 63.11. **GCMS (EI)** *m/z* calcd. for $C_{10}H_9F_3$ [M^+] 186.1, found 186.1, 167.1, 151.0, 133.1, 117.1, 115.1, 91.1, 77.1, 69.0, 63.1. The analytical data correspond with those reported in the literature.[12]

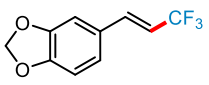
(*E*)-1,2-Dimethoxy-4-(3,3,3-trifluoroprop-1-en-1-yl)benzene (39)



The title compound was synthesized according to the general procedure C employing CdSe (6.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), (*E*)-3-(3,4-dimethoxyphenyl)acrylic acid (62.4 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 28 h. The product was purified by column chromatography (silica gel, gradient 5 to 8% diethyl ether/petroleum ether). Yield = 72% (50.1 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.07 (dd, *J* = 16.1, 2.2 Hz, 1H), 7.01 (dd, *J* = 8.3, 2.0 Hz, 1H), 6.95 (d, *J* = 2.0 Hz, 1H), 6.85 (d, *J* = 8.3 Hz, 1H), 6.06 (dq, *J* = 16.0, 6.5 Hz, 1H), 3.90 (d, *J* = 2.7 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 150.82, 149.32, 137.49 (q, J_{C-F} = 6.8 Hz), 126.38, 123.99 (q, J_{C-F} = 268.6 Hz), 121.71, 113.67 (q, J_{C-F} = 33.8 Hz), 111.12, 109.39, 56.00, 55.96. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 62.81 (d, *J* = 4.6 Hz). **GCMS (EI)** *m/z* calcd. for $C_{11}H_{11}F_3O_2$ [M^+] 232.1, found 232.1, 217.1, 213.1, 189.1, 174.0, 169.1, 149.0, 145.0, 139.1, 125.0, 121.1, 109.1, 101.1, 96.1, 77.1. The analytical data correspond with those reported in the literature.[12]

(*E*)-5-(3,3,3-Trifluoroprop-1-en-1-yl)benzo[*d*][1,3]dioxole (40)

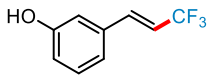


The title compound was synthesized according to the general procedure C employing CdSe (5.7 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), (*E*)-3-(benzo[*d*][1,3]dioxol-5-yl)acrylic acid (57.6 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 28 h. The product was purified by column chromatography (silica gel, gradient 5 to 8% diethyl ether/petroleum ether). Yield = 70% (45.3 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.04 (d, *J* = 17.6 Hz, 1H), 6.95 (s, 1H), 6.92 (d, *J* = 8.2 Hz, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.14 – 5.96 (m, 1H), 6.01 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 149.40,

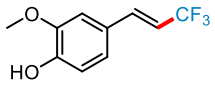
148.52, 137.36 (q, $J_{C\text{-}F}$ = 6.8 Hz), 127.85, 123.94 (q, $J_{C\text{-}F}$ = 268.7 Hz), 123.55, 113.97 (q, $J_{C\text{-}F}$ = 33.7 Hz), 108.64, 106.30, 101.69. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 62.91. **GCMS (EI)** *m/z* calcd. for $C_{10}H_7F_3O_2$ [M^+] 216.0, found 216.1, 215.1, 197.1, 167.0, 157.0, 145.0, 139.1, 132.0, 127.1, 119.1, 114.1, 107.1, 91.1, 89.1, 63.1. The analytical data correspond with those reported in the literature.[13]

(*E*)-3-(3,3,3-Trifluoroprop-1-en-1-yl)phenol (41)



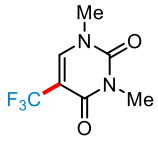
The title compound was synthesized according to the general procedure C employing CdSe (4.9 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), (*E*)-3-(3-hydroxyphenyl)acrylic acid (49.2 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 28 h. The product was purified by column chromatography (silica gel, gradient 3 to 6% DCM/petroleum ether). Yield = 55% (31 mg).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (t, *J* = 7.9 Hz, 1H), 7.08 (dd, *J* = 16.1, 2.3 Hz, 1H), 7.02 (d, *J* = 7.6 Hz, 1H), 6.93 (s, 1H), 6.87 (dd, *J* = 8.1, 2.5 Hz, 1H), 6.16 (dq, *J* = 16.1, 6.5 Hz, 1H), 5.59 (broad, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 156.19, 137.45 (q, $J_{C\text{-}F}$ = 6.8 Hz), 135.17, 130.31, 123.67 (q, $J_{C\text{-}F}$ = 268.8 Hz), 120.39, 117.25, 116.38 (q, $J_{C\text{-}F}$ = 33.9 Hz), 114.16. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 63.37. **GCMS (EI)** *m/z* calcd. for $C_9H_7F_3O$ [M^+] 188.0, found 188.0, 167.0, 149.0, 140.0, 124.0, 119.0, 114.0, 109.1, 101.0, 96.0, 91.1, 75.0, 69.0, 65.1.

(*E*)-2-Methoxy-4-(3,3,3-trifluoroprop-1-en-1-yl)phenol (42)



The title compound was synthesized according to the general procedure C employing CdSe (5.8 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), (*E*)-3-(4-hydroxy-3-methoxyphenyl)acrylic acid (58.2 mg, 0.30 mmol), CF_3SO_2Cl (96 µL, 0.90 mmol), and CH_3CN (0.1 M, 3 mL) for 28 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% DCM/petroleum ether). Yield = 63% (41.2 mg).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.06 (dd, *J* = 16.1, 2.3 Hz, 1H), 7.02 – 6.89 (m, 3H), 6.05 (dq, *J* = 16.1, 6.6 Hz, 1H), 5.86 (s, 1H), 3.93 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 147.62, 146.91, 137.64 (q, $J_{C\text{-}F}$ = 6.8 Hz), 126.03, 124.05 (q, $J_{C\text{-}F}$ = 268.5 Hz), 122.20, 114.84, 113.45 (q, $J_{C\text{-}F}$ = 33.7 Hz), 109.12, 56.07. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 62.78 (d, *J* = 4.3 Hz). **GCMS (EI)** *m/z* calcd. for $C_{10}H_9F_3O_2$ [M^+] 218.1, found 218.1, 203.0, 199.1, 175.0, 167.0, 155.1, 145.1, 139.0, 136.1, 133.0, 127.1, 125.0, 107.0, 101.0, 91.0, 77.0. The analytical data correspond with those reported in the literature.[12]

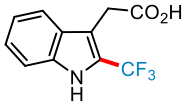
1,3-Dimethyl-5-(trifluoromethyl)pyrimidine-2,4(1*H*,3*H*)-dione (43)



The title compound was synthesized according to the general procedure A employing CdSe (4.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (42.0 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 48 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% diethyl ether/petroleum ether). Yield = 84% (52.4 mg).

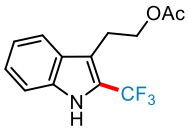
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 1.3 Hz, 1H), 3.47 (s, 3H), 3.32 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 158.84, 150.99, 143.87 (q, J_{C-F} = 5.9 Hz), 122.13 (q, J_{C-F} = 269.7 Hz), 103.95 (q, J_{C-F} = 32.9 Hz), 37.79, 28.05. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 63.81. **GCMS (EI)** *m/z* calcd. for $C_7H_7F_3N_2O_2$ [M^+] 208.0, found 208.1, 188.0, 179.0, 160.0, 150.0, 132.0, 123.0, 103.0, 91.0, 75.0, 60.0. The analytical data correspond with those reported in the literature.[5]

2-(2-(Trifluoromethyl)-1*H*-indol-3-yl)acetic acid (44)



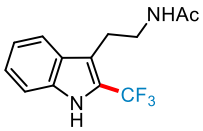
The title compound was synthesized according to the general procedure A employing CdSe (5.2 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 2-(1*H*-indol-3-yl)acetic acid (52.5 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 3 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield = 70% (51 mg).
^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 12.42 (b, 1H), 12.08 (s, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.46 (d, *J* = 8.3 Hz, 1H), 7.29 (t, *J* = 7.6 Hz, 1H), 7.13 (t, *J* = 7.5 Hz, 1H), 3.83 (s, 2H). **^{13}C NMR (101 MHz, DMSO-*d*$_6$)** δ 171.95, 135.63, 126.89, 124.39, 122.17 (q, J_{C-F} = 268.9 Hz), 121.67 (q, J_{C-F} = 36.0 Hz), 120.18, 120.08, 112.24, 110.37 (d, J_{C-F} = 3.0 Hz), 29.31. **^{19}F NMR (377 MHz, DMSO-*d*$_6$)** δ – 56.84. **GCMS (EI)** *m/z* calcd. for $C_{11}H_8F_3NO_2$ [M^+] 243.1, found 243.0, 198.1, 178.0, 158.0, 152.0, 128.0, 101.1, 89.0, 75.0.

2-(2-(Trifluoromethyl)-1H-indol-3-yl)ethyl acetate (45)



The title compound was synthesized according to the general procedure A employing CdSe (6.1 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 2-(1*H*-indol-3-yl)ethyl acetate (60.9 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 3 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield = 79% (64.2 mg).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (s, 1H), 7.71 (d, *J* = 8.1 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.21 (t, *J* = 7.5 Hz, 1H), 4.34 (t, *J* = 6.9 Hz, 2H), 3.25 (td, *J* = 7.0, 1.5 Hz, 2H), 2.03 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.39, 135.39, 127.51, 125.01, 122.54 (q, J_{C-F} = 36.9 Hz), 122.03 (q, J_{C-F} = 240.7 Hz), 120.85, 120.21, 114.04 (d, J_{C-F} = 3.1 Hz), 111.96, 64.26, 23.61, 21.03. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 58.27. **GCMS (EI)** *m/z* calcd. for $C_{13}H_{12}F_3NO_2$ [M^+] 271.1, found 271.1, 211.1, 198.1, 191.1, 178.1, 176.1, 172.1, 158.1, 152.1, 143.1, 140.1, 128.1, 115.1, 101.1, 89.1, 77.1.

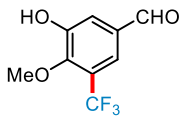
***N*-(2-(2-(Trifluoromethyl)-1*H*-indol-3-yl)ethyl)acetamide (46)**



The title compound was synthesized according to the general procedure A employing CdSe (6.0 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), *N*-(2-(1*H*-indol-3-yl)ethyl)acetamide (60.6 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 3 h. The product was purified by column chromatography (silica gel, gradient 5 to 10% EA/petroleum ether). Yield = 65% (52.6 mg).

^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 11.93 (s, 1H), 7.98 (s, 1H), 7.69 (d, *J* = 8.1 Hz, 1H), 7.44 (d, *J* = 8.1 Hz, 1H), 7.28 (ddd, *J* = 8.2, 6.9, 1.1 Hz, 1H), 7.13 (ddd, *J* = 8.1, 6.9, 1.0 Hz, 1H), 3.24 (q, *J* = 6.7 Hz, 2H), 2.97 (d, *J* = 7.3 Hz, 2H), 1.76 (t, *J* = 1.5 Hz, 3H). **^{13}C NMR (101 MHz, DMSO-*d*$_6$)** δ 169.16, 135.74, 126.81, 124.29, 122.33 (q, $J_{C\text{-}F}$ = 220 Hz), 121.09 (q, $J_{C\text{-}F}$ = 36.1 Hz), 119.90, 114.54 (q, $J_{C\text{-}F}$ = 3.0 Hz), 112.32, 39.67, 23.93, 22.61. **^{19}F NMR (377 MHz, DMSO-*d*$_6$)** δ – 56.41. **GCMS (EI)** *m/z* calcd. for $C_{13}H_{13}F_3N_2O$ [M^+] 270.1, found 270.1, 211.1, 198.0, 189.1, 178.0, 176.0, 166.0, 158.0, 152.0, 128.1, 115.0, 101.1, 77.1, 72.1.

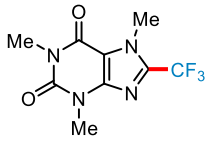
3-Hydroxy-4-methoxy-5-(trifluoromethyl)benzaldehyde (47)



The title compound was synthesized according to the general procedure A employing CdSe (4.5 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), 3-hydroxy-4-methoxybenzaldehyde (45.6 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 48 h. The product was purified by column chromatography (silica gel, gradient 15 to 20% diethyl ether/petroleum ether). Yield = 61% (40.2 mg, 95:5, r.r., the product ratio was determined by ^{19}F NMR analysis).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.24 (q, *J* = 2.1 Hz, 1H), 7.64 (s, 1H), 7.29 (s, 1H), 6.31 (s, 1H), 4.01 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 187.99 (q, $J_{C\text{-}F}$ = 2.9 Hz), 150.34, 148.86, 127.26, 126.54 (q, $J_{C\text{-}F}$ = 33.0 Hz), 123.73 (q, $J_{C\text{-}F}$ = 274.0 Hz), 112.84 (q, $J_{C\text{-}F}$ = 5.9 Hz), 110.30, 56.60. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 56.84. **GCMS (EI)** *m/z* calcd. for $C_9H_7F_3O_3$ [M^+] 220.0, found 220.0, 219.0, 201.0, 191.0, 176.1, 149.0, 129.0, 120.0, 113.0, 101.0, 75.0, 51.0.

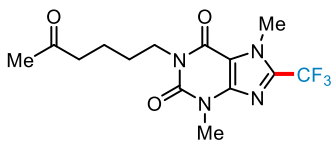
1,3,7-Trimethyl-8-(trifluoromethyl)-3,7-dihydro-1H-purine-2,6-dione (48)



The title compound was synthesized according to the general procedure A employing CdSe (5.8 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), caffeine (58.2 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 48 h. The product was purified by column chromatography (silica gel, gradient 10 to 15% EA/petroleum ether). Yield = 78% (61.3 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.13 (q, *J* = 1.3 Hz, 3H), 3.56 (s, 3H), 3.38 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 155.52, 151.40, 146.59, 138.98 (q, $J_{C\text{-}F}$ = 40.0 Hz), 118.27 (q, $J_{C\text{-}F}$ = 271.4 Hz), 109.72, 33.27 (d, $J_{C\text{-}F}$ = 2.2 Hz), 29.98, 28.27. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 62.43. **GCMS (EI)** *m/z* calcd. for $C_9H_9F_3N_4O_2$ [M^+] 262.1, found 262.1, 243.1, 233.0, 204.0, 193.1, 177.0, 150.0, 108.1, 82.1, 67.0. The analytical data correspond with those reported in the literature.[5]

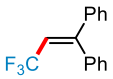
3,7-Dimethyl-1-(5-oxohexyl)-8-(trifluoromethyl)-3,7-dihydro-1*H*-purine-2,6-dione (49)



The title compound was synthesized according to the general procedure A employing CdSe (8.3 mg, 10% w/w), K_2HPO_4 (130.5 mg, 0.75 mmol), pentoxifylline (83.4 mg, 0.30 mmol), CF_3SO_2Cl (64 µL, 0.60 mmol), and CH_3CN (0.1 M, 3 mL) for 48 h. The product was purified by column chromatography (silica gel, gradient 15 to 20% EA/petroleum ether). Yield = 73% (75.7 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.11 (d, *J* = 1.4 Hz, 3H), 4.01 – 3.93 (m, 2H), 3.53 (d, *J* = 1.4 Hz, 3H), 2.46 (t, *J* = 6.7 Hz, 2H), 2.10 (s, 3H), 1.64 – 1.57 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 208.63, 155.35, 151.11, 146.61, 138.96 (q, J_{C-F} = 40.1 Hz), 118.25 (q, J_{C-F} = 271.3 Hz), 109.74, 43.13, 41.18, 33.25 (d, J_{C-F} = 2.2 Hz), 30.03, 29.89, 27.37, 20.94. **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 62.44. **GCMS (EI)** *m/z* calcd. for $C_{14}H_{17}F_3N_4O_3$ [M^+] 346.1, found 346.1, 327.1, 303.1, 289.1, 275.1, 261.0, 249.0, 231.0, 204.0, 177.0, 150.0, 110.0, 82.1, 67.0. The analytical data correspond with those reported in the literature.[5]

(3,3,3-Trifluoroprop-1-ene-1,1-diyl)dibenzene



Yield = 81% (Yield was determined by ^{19}F NMR analysis of the crude mixture using trifluoromethylbenzene as an internal standard).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.33 (m, 6H), 7.33 – 7.25 (m, 4H), 6.17 (qd, *J* = 8.3, 1.6 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 152.61 (q, J_{C-F} = 5.6 Hz), 140.25, 137.40, 129.53, 129.25, 129.23, 128.61, 128.37, 128.17, 128.10, 127.30, 123.25 (q, J_{C-F} = 270.5 Hz), 115.56 (q, J_{C-F} = 33.8 Hz). **^{19}F NMR (377 MHz, $CDCl_3$)** δ – 55.50. **GCMS (EI)** *m/z* calcd. for $C_{15}H_{11}F_3$ [M^+] 248.1, found 248.1, 227.0, 207.1, 196.1, 179.1, 165.1, 151.0, 139.0, 127.0, 114.0, 89.0, 76.0. The analytical data correspond with those reported in the literature.[14]

REFERENCES

(1) Lowry, M. S.; Goldsmith, J. I.; Slinker, J. D.; Rohl, R.; Pascal, R. A.; Malliaras, G. G.; Bernhard, S. Single-Layer Electroluminescent Devices and Photoinduced Hydrogen Production from an Ionic Iridium(III) Complex. *Chem. Mater.* **2005**, *17*, 5712–5719.

(2) Muralirajan, K.; Kancherla, R.; Rueping, M. Dehydrogenative Aromatization and Sulfonylation of Pyrrolidines: Orthogonal Reactivity in Photoredox Catalysis. *Angew. Chem. Int. Ed.* **2018**, *57*, 14787–14791.

(3) Pang, Y.; Lee, J. W.; Kubota, K.; Ito, H. Solid-State Radical C−H Trifluoromethylation Reactions Using Ball Milling and Piezoelectric Materials. *Angew. Chem. Int. Ed.* **2020**, *59*, 22570–22576.

(4) Pitre, S. P.; Tiernan, C. D. M.; Ismaili, H.; Scaiano, J. C. Metal-Free Photocatalytic Radical Trifluoromethylation Utilizing Methylene Blue and Visible Light Irradiation. *ACS Catal.* **2014**, *4*, 2530–2535.

(5) Ghosh, I.; Khamrai, J.; Savateev, A.; Shlapakov, N.; Antonietti, M.; König, B. Organic Semiconductor Photocatalyst can Bifunctionalize Arenes and Heteroarenes. *Science* **2019**, *365*, 360–366.

(6) Nagib, D. A.; MacMillan, D. W. C. Trifluoromethylation of Arenes and Heteroarenes by Means of Photoredox Catalysis. *Nature* **2011**, *480*, 224–228.

(7) Choi, W. J.; Choi, S.; Ohkubo, K.; Fukuzumi, S.; Cho, E. J.; You, Y. Mechanisms and Applications of Cyclometalated Pt(ii) Complexes in Photoredox Catalytic Trifluoromethylation. *Chem. Sci.* **2015**, *6*, 1454–1464.

(8) Ye, Y.; Cheung, K. P. S.; He, L.; Tsu, G. C. Synthesis of 2-(Trifluoromethyl)indoles via Domino Trifluoromethylation/Cyclization of 2-Alkynylanilines. *Org. Lett.* **2018**, *20*, 1676−1679.

(9) Chen, Y.; Wang, Y.; Sun, Z.; Ma, D. Elaboration of 2-(Trifluoromethyl)indoles via a Cascade Coupling/Condensation/Deacylation Process. *Org. Lett.* **2008**, *10*, 625–628.

(10) Seo, S.; Taylor, J. B.; Greaney, M. F. Silver-Catalysed Trifluoromethylation of Arenes at Room Temperature. *Chem. Commun.* **2013**, *49*, 6385–6387.

(11) Guo, J.; Xu, C.; Wang, L.; Huang, W.; Wang, M. Catalyst-Free and Selective Trifluoromethylative Cyclization of Acryloanilides using $PhICF_3Cl$. *Org. Biomol. Chem*. **2019**, *17*, 4593–4599.

(12) Xu, P.; Abdukader, A.; Hu, K.; Cheng, Y.; Zhu, C. Room Temperature Decarboxylative Trifluoromethylation of α,β-Unsaturated Carboxylic Acids by Photoredox Catalysis. *Chem. Commun*. **2014**, *50*, 2308–2310.

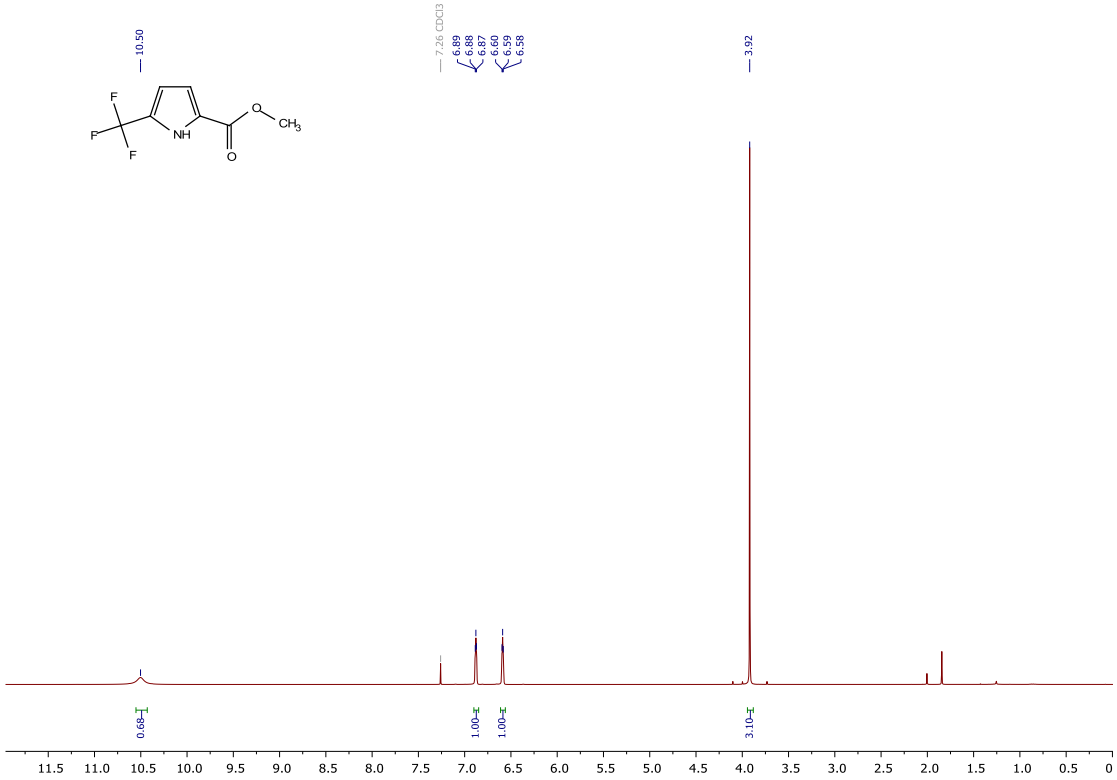
(13) Hong, H.; Li, Y.; Chen, L.; Li, B.; Zhu, Z.; Chen, X.; Chen, L.; Huang, Y. Electrochemical Synthesis Strategy for C_{vinyl}-CF_3 Compounds through Decarboxylative Trifluoromethylation. *J. Org. Chem.* **2019**, *84*, 5980−5986.

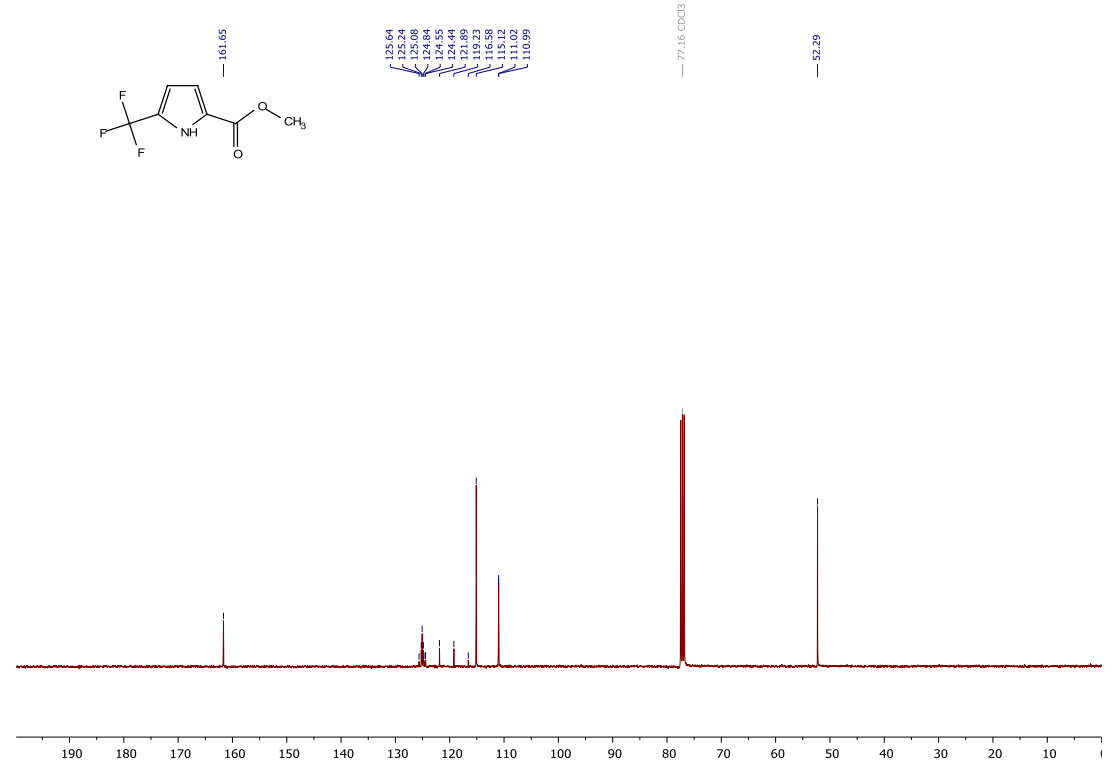
(14) Huang, B.; Bu, X.-S.; Xu, J.; Dai, J.-J.; Feng, Y.-S.; Xu, H.-J. Metal-Free, Visible-Light-Mediated Direct C−H Trifluoromethylation of Hydrazones with NADH Coenzyme Model Catalyst. *Asian J. Org. Chem.* **2018**, *7*, 137 –140.

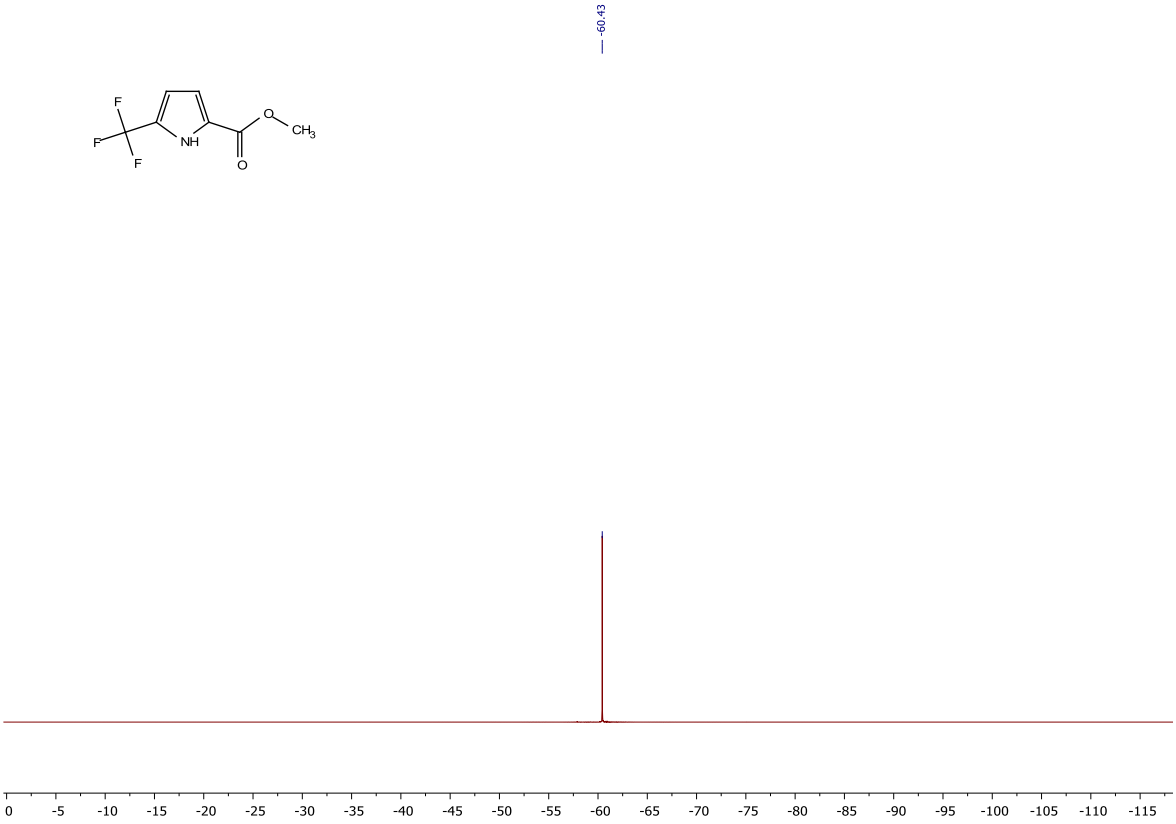
NMR Spectra

^{1}H, ^{13}C and ^{19}F NMR spectra are included below for all isolated compounds.

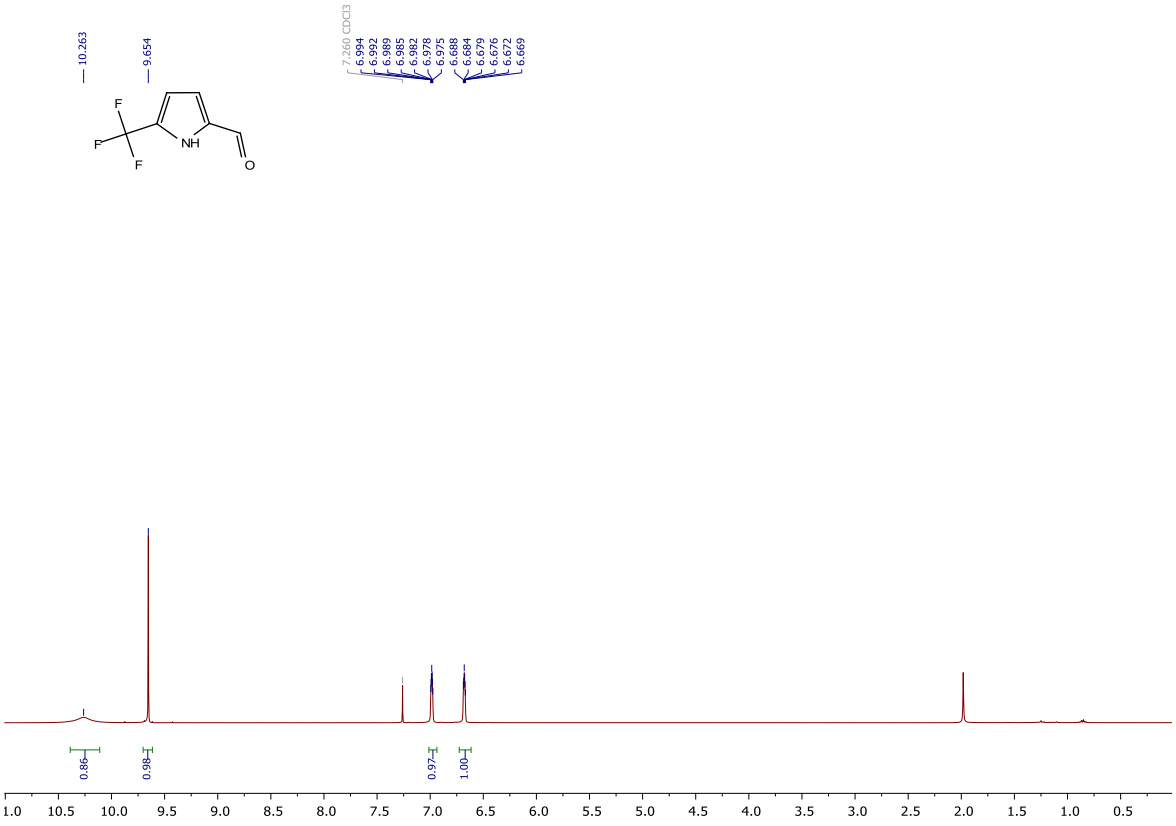
Methyl 5-(trifluoromethyl)-1*H*-pyrrole-2-carboxylate (1)

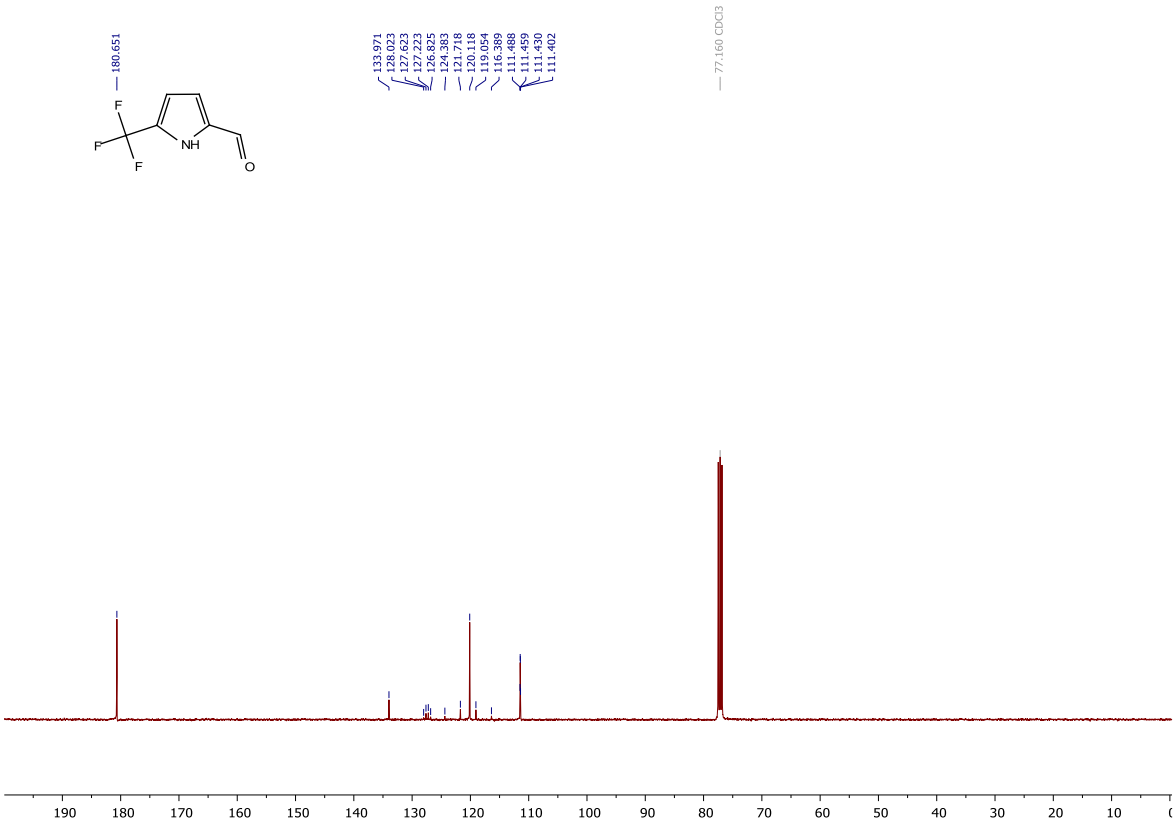






5-(Trifluoromethyl)-1*H*-pyrrole-2-carbaldehyde (2)

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127.223
126.825
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111.459
111.430
111.402
77.160 CDCl3
F
F
F
NH
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190
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150
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80
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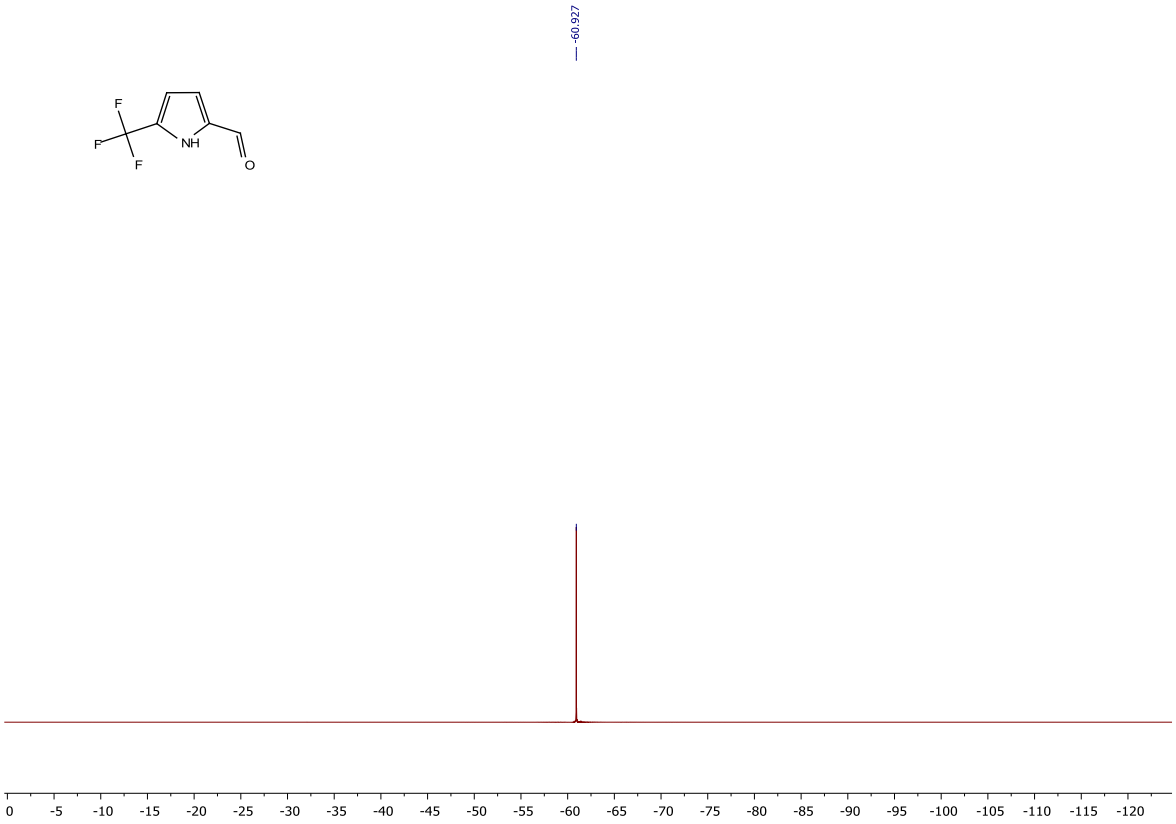
50
40
30
20
10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-50
-55
-60
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-70
-75
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-85
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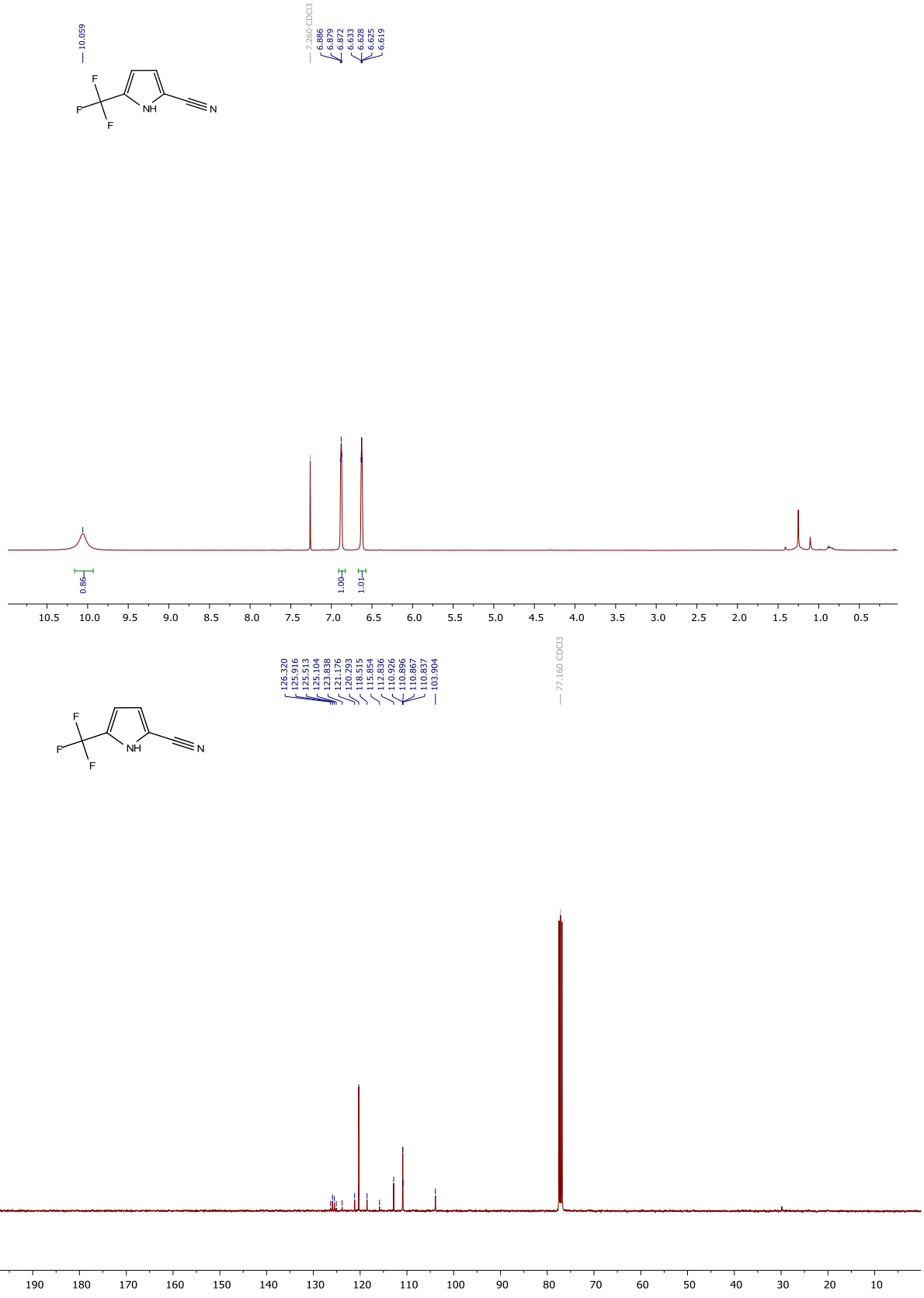
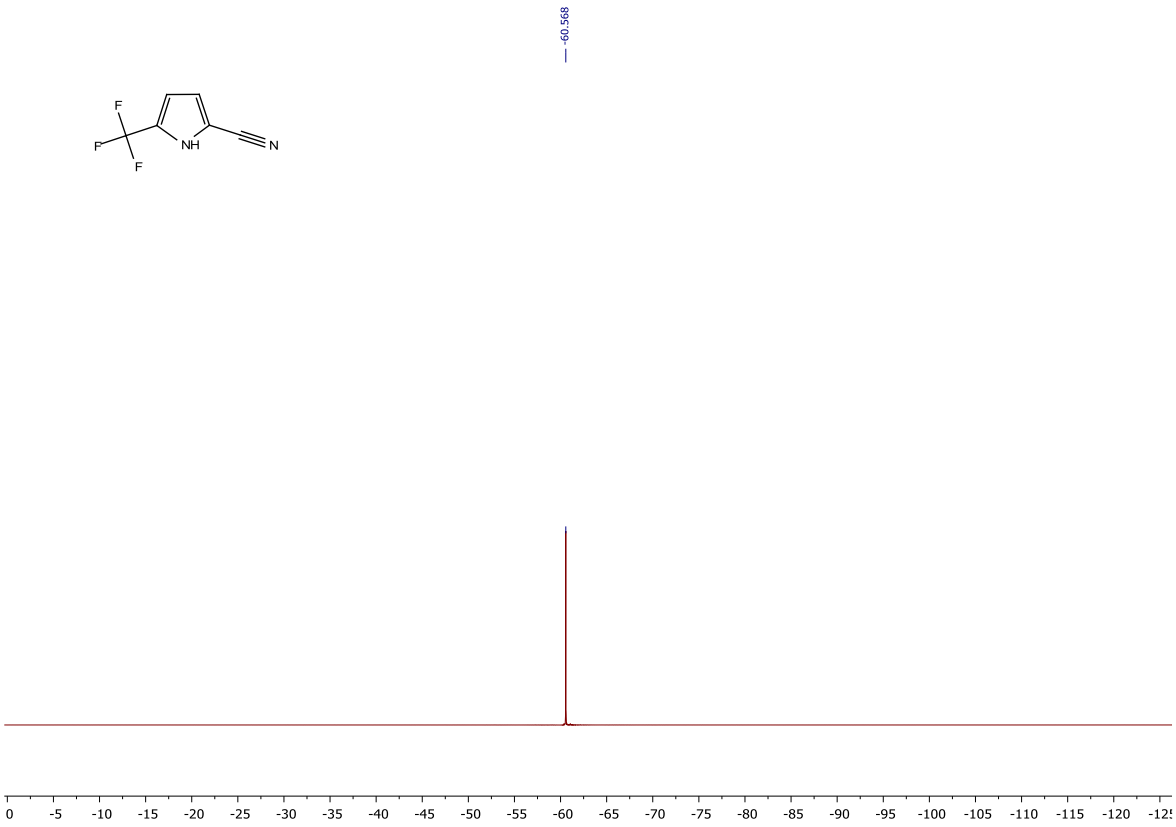
-95
-100
-105
-110
-115
-120

5-(Trifluoromethyl)-1*H*-pyrrole-2-carbonitrile (3)





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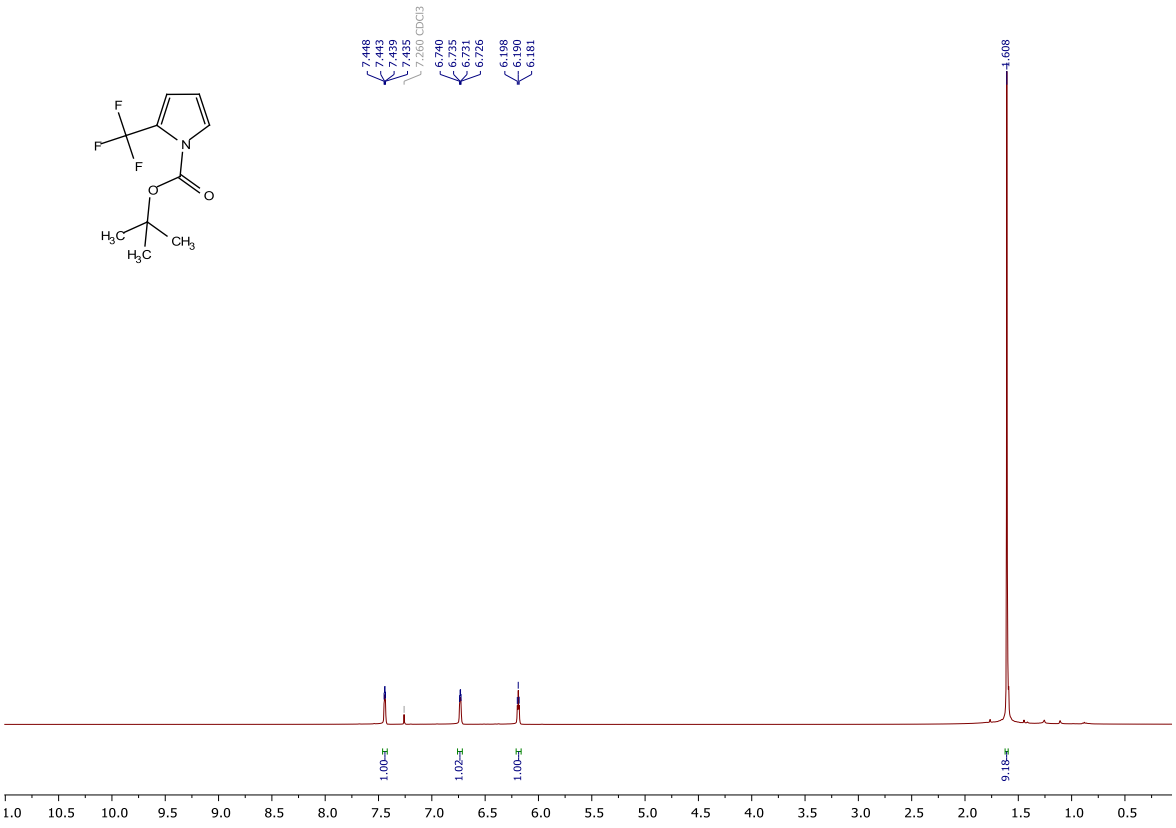

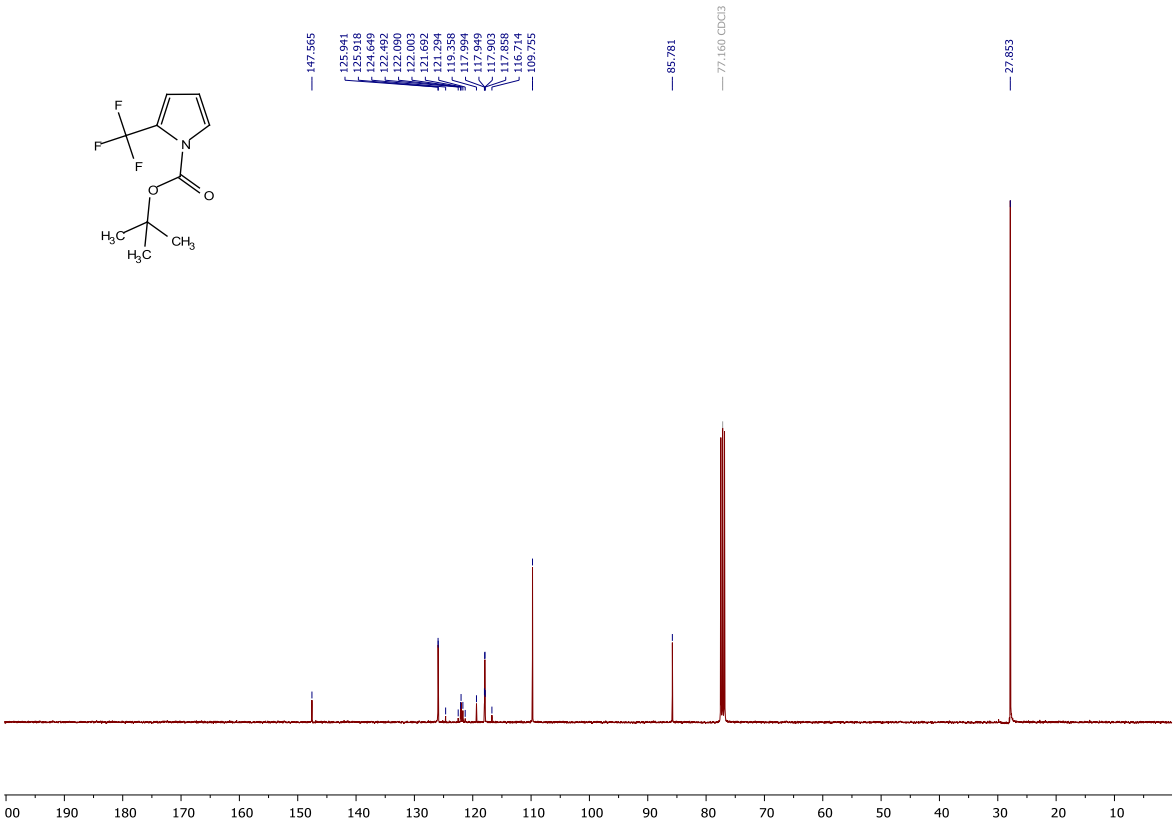
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117.858
116.714
109.755
85.781
77.160 CDCl3
27.853
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F
N
O
O
H3C
CH3
H3C
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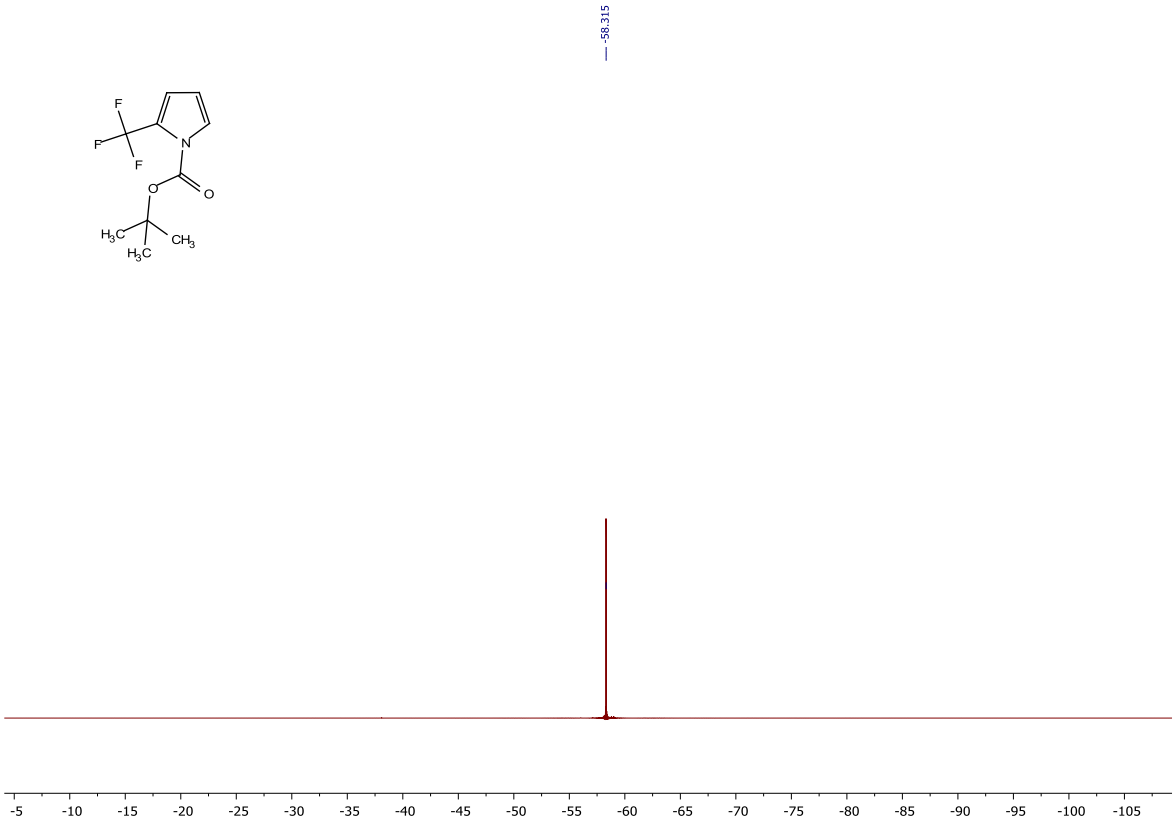
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H3C
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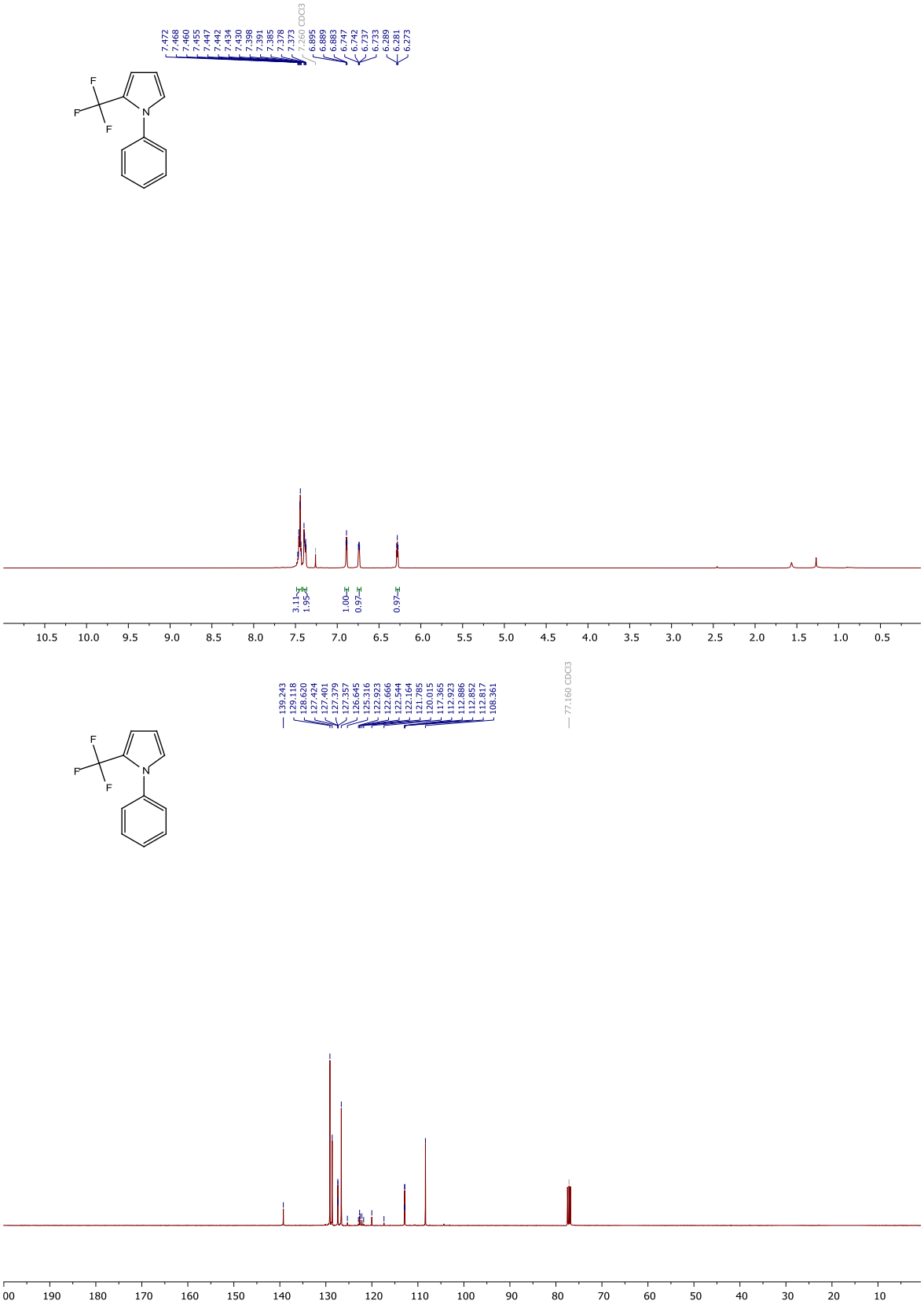
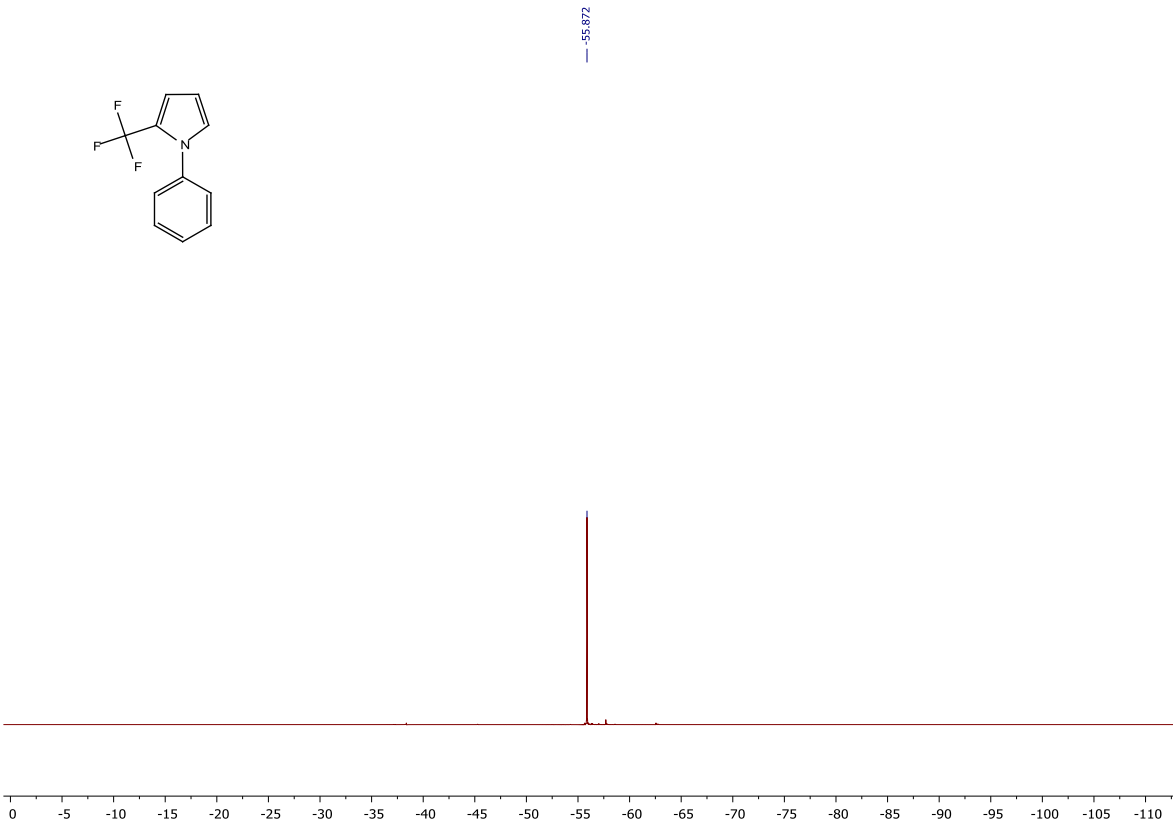
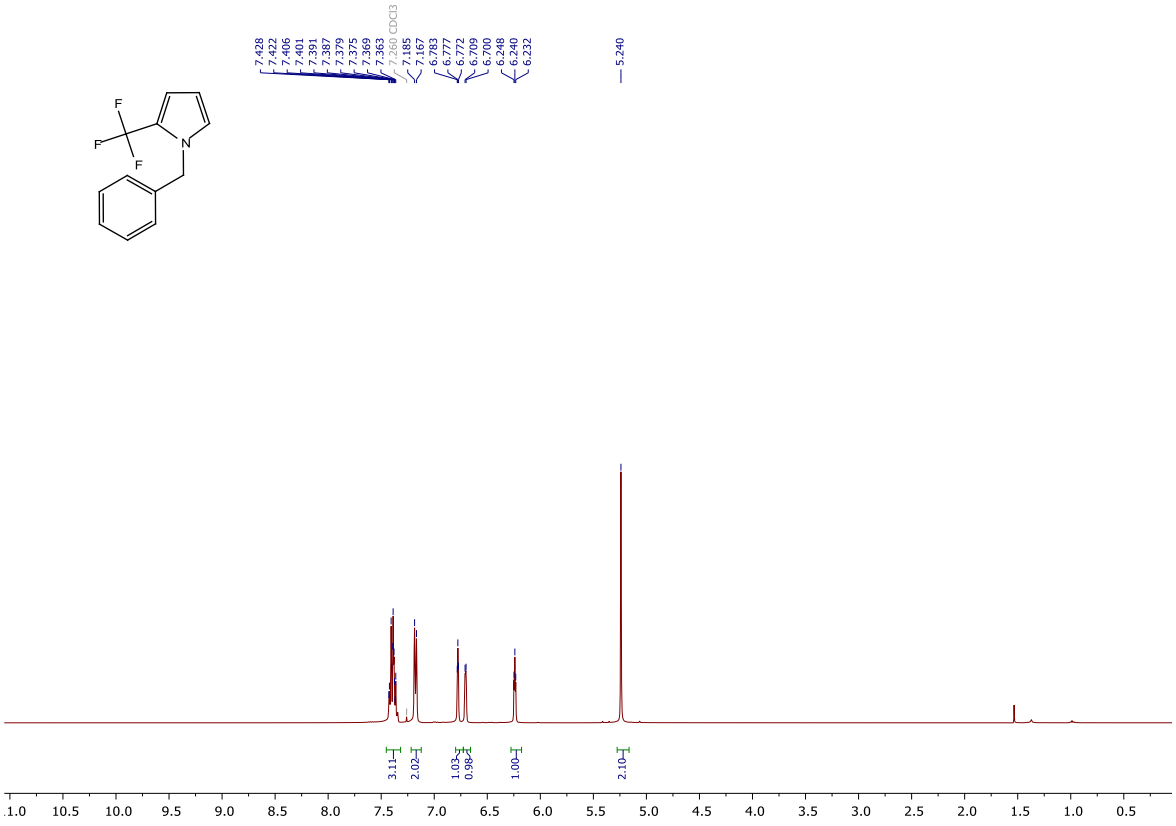
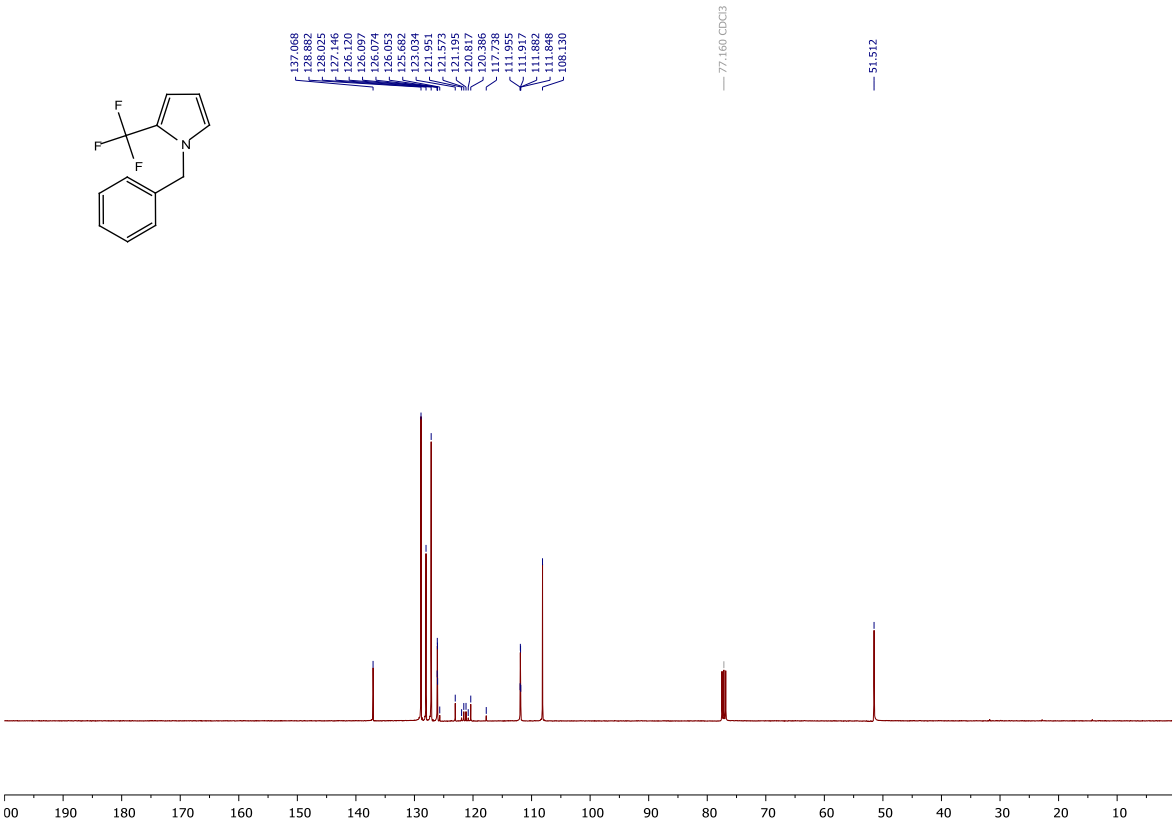
1-Phenyl-2-(trifluoromethyl)-1*H*-pyrrole (5)





1-Benzyl-2-(trifluoromethyl)-1*H*-pyrrole (6)

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111.955
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111.848
108.130
77.160 CDCl3
51.512
F
F
F
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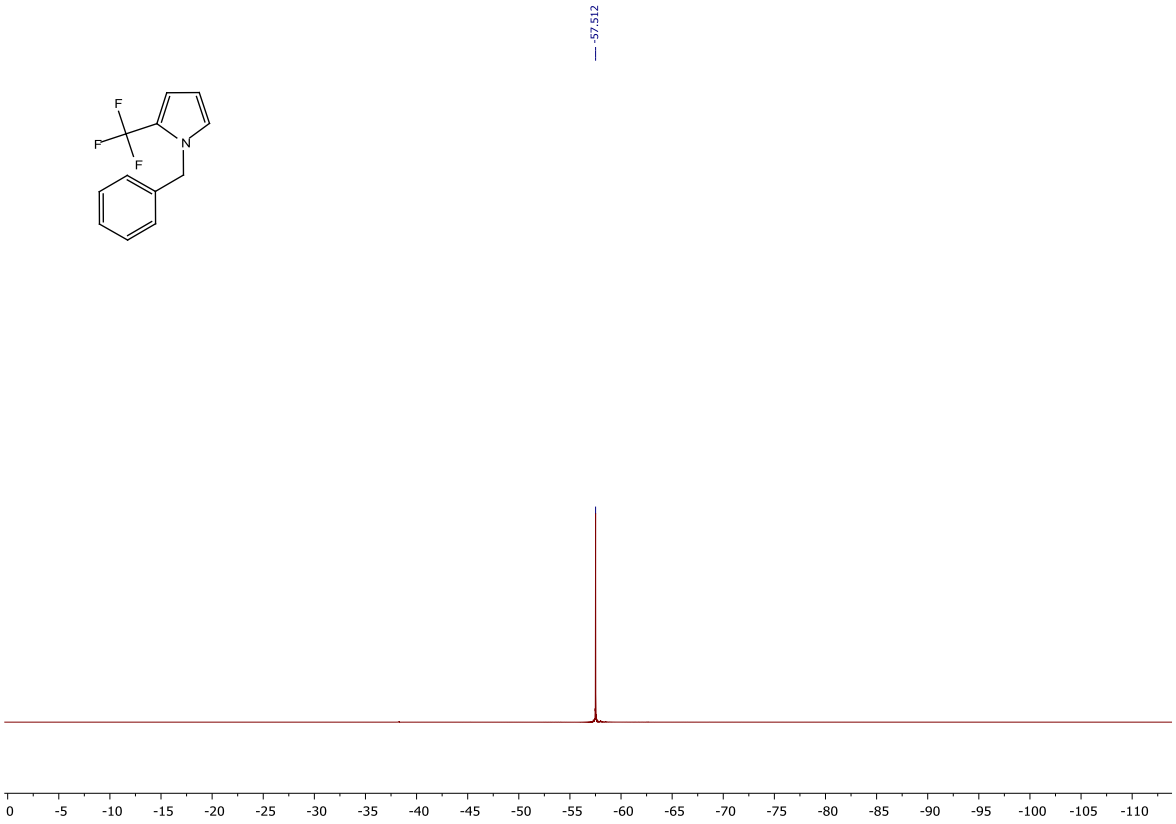
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-55
-60
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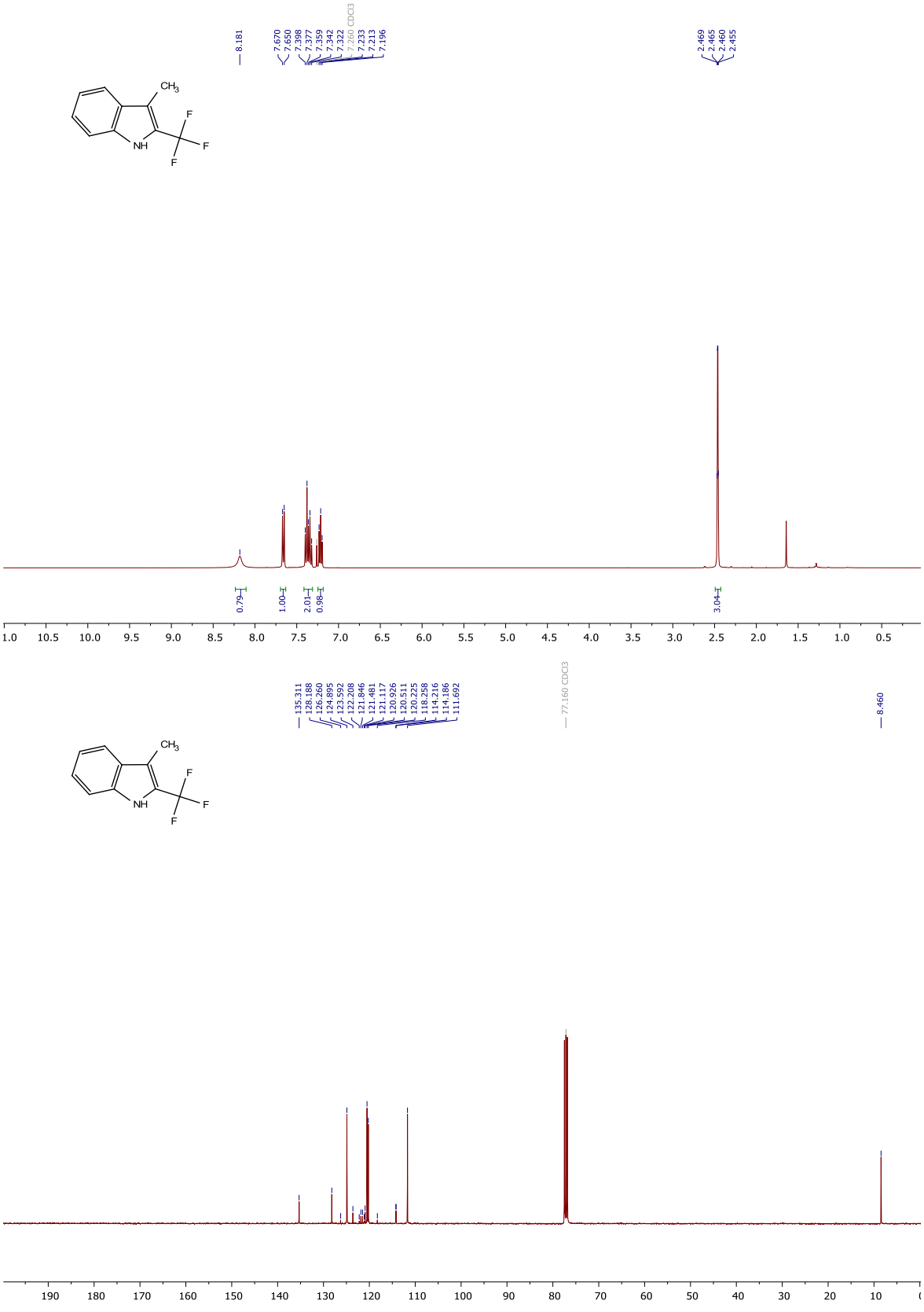
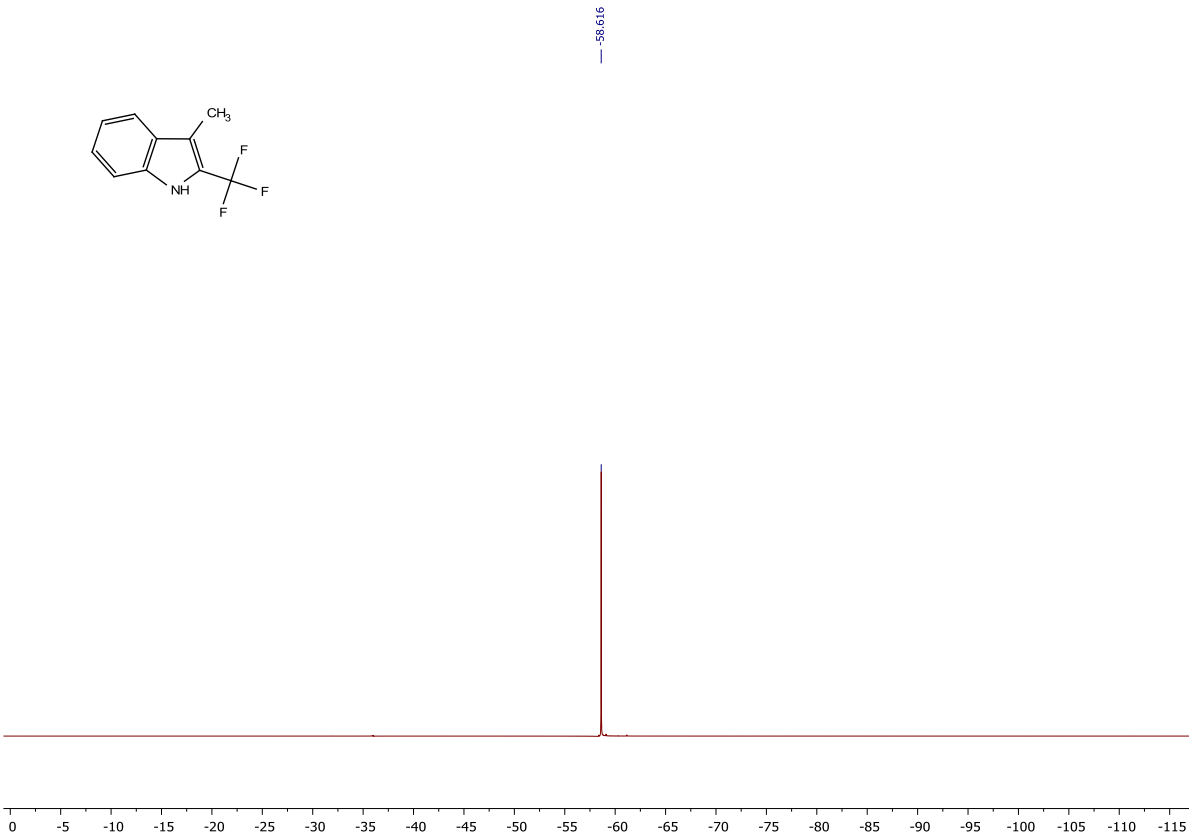
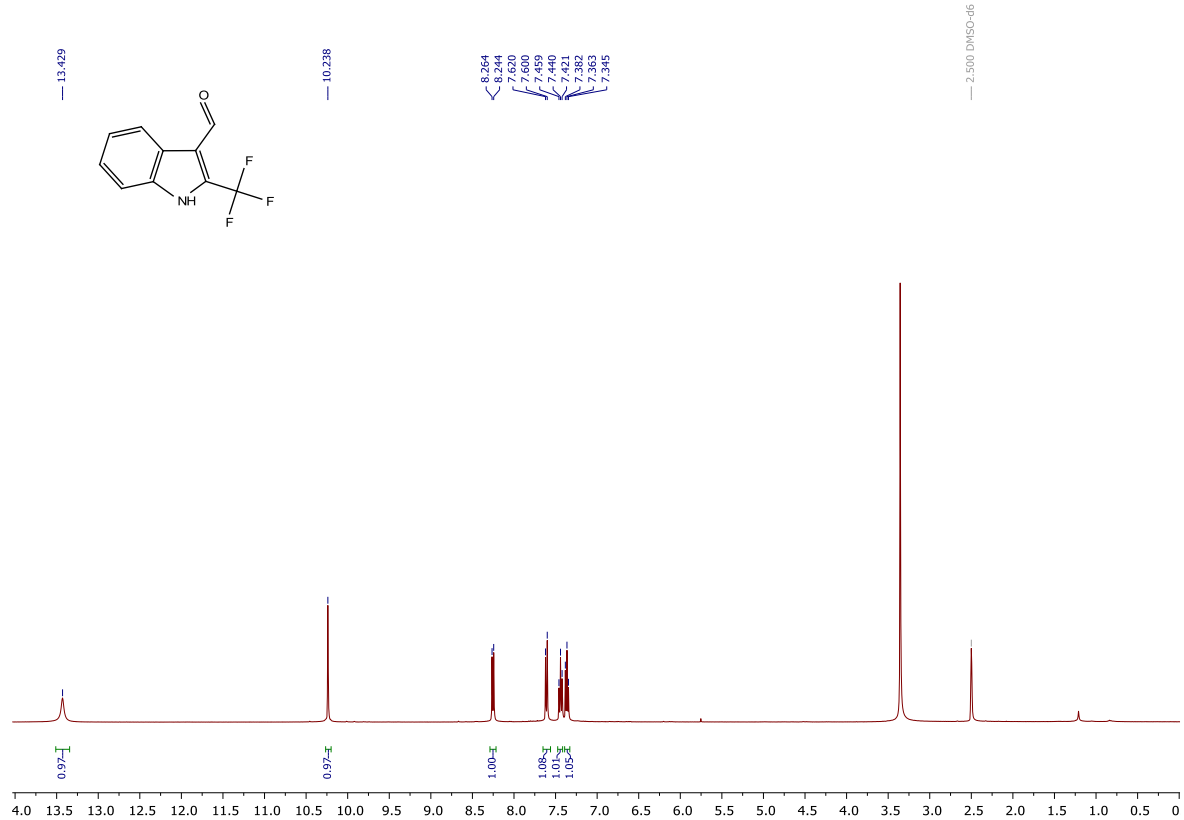
-85
-90
-95
-100
-105
-110

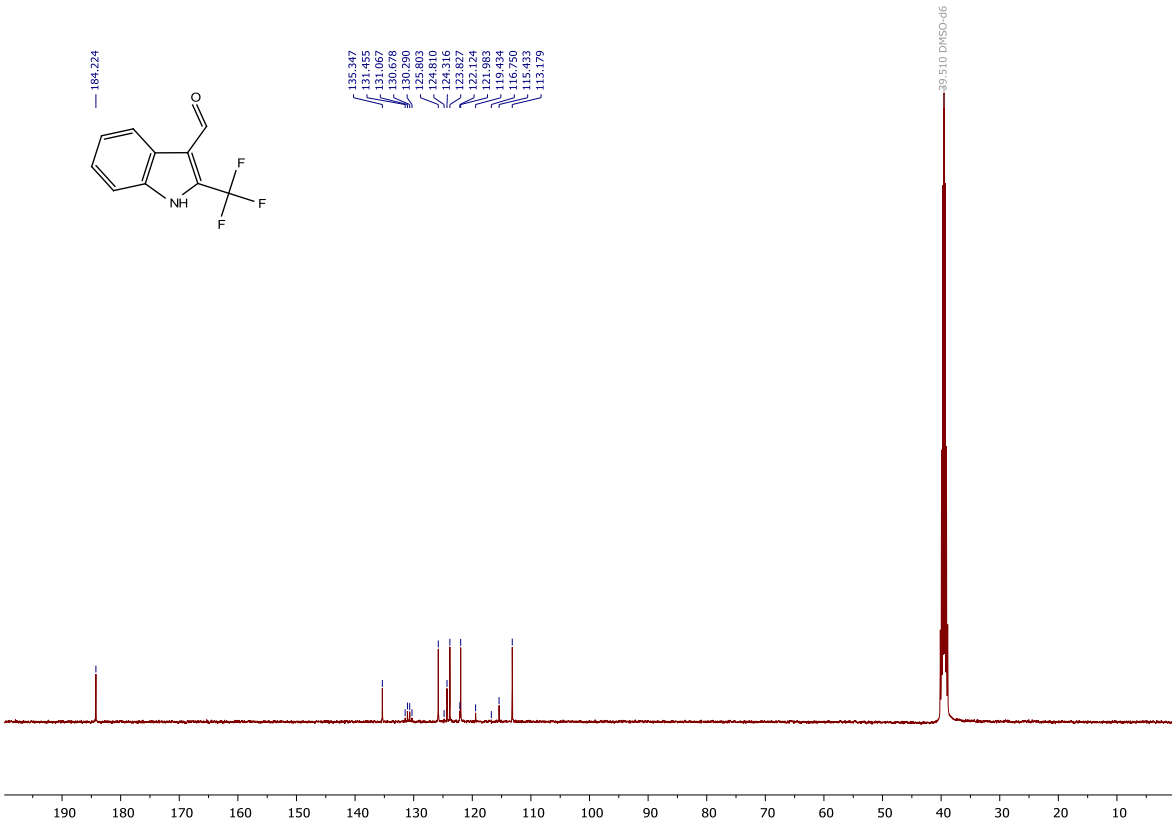
3-Methyl-2-(trifluoromethyl)-1*H*-indole (8)





2-(Trifluoromethyl)-1*H*-indole-3-carbaldehyde (9)

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130.290
125.803
124.810
124.316
123.827
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119.434
116.750
115.433
113.179
39.510 DMSO-d6
O
NH
F
F
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130
120
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40
30
20
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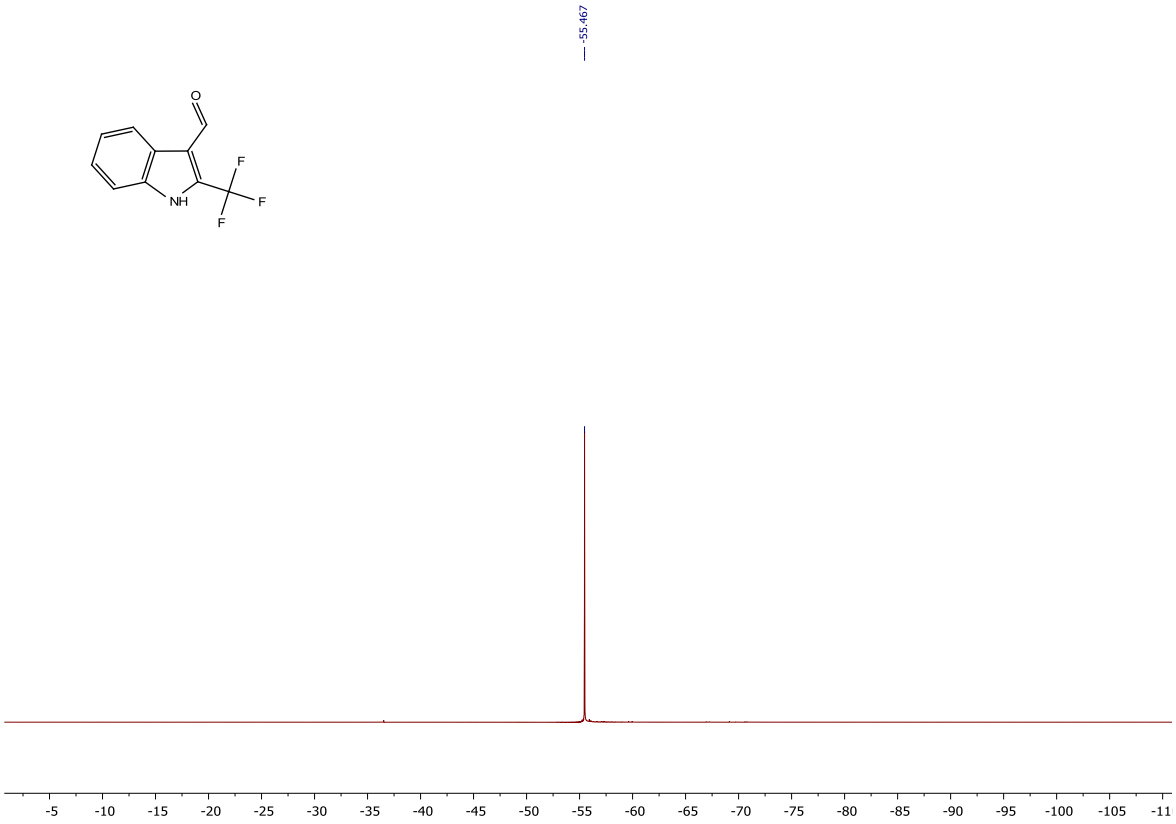
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-65
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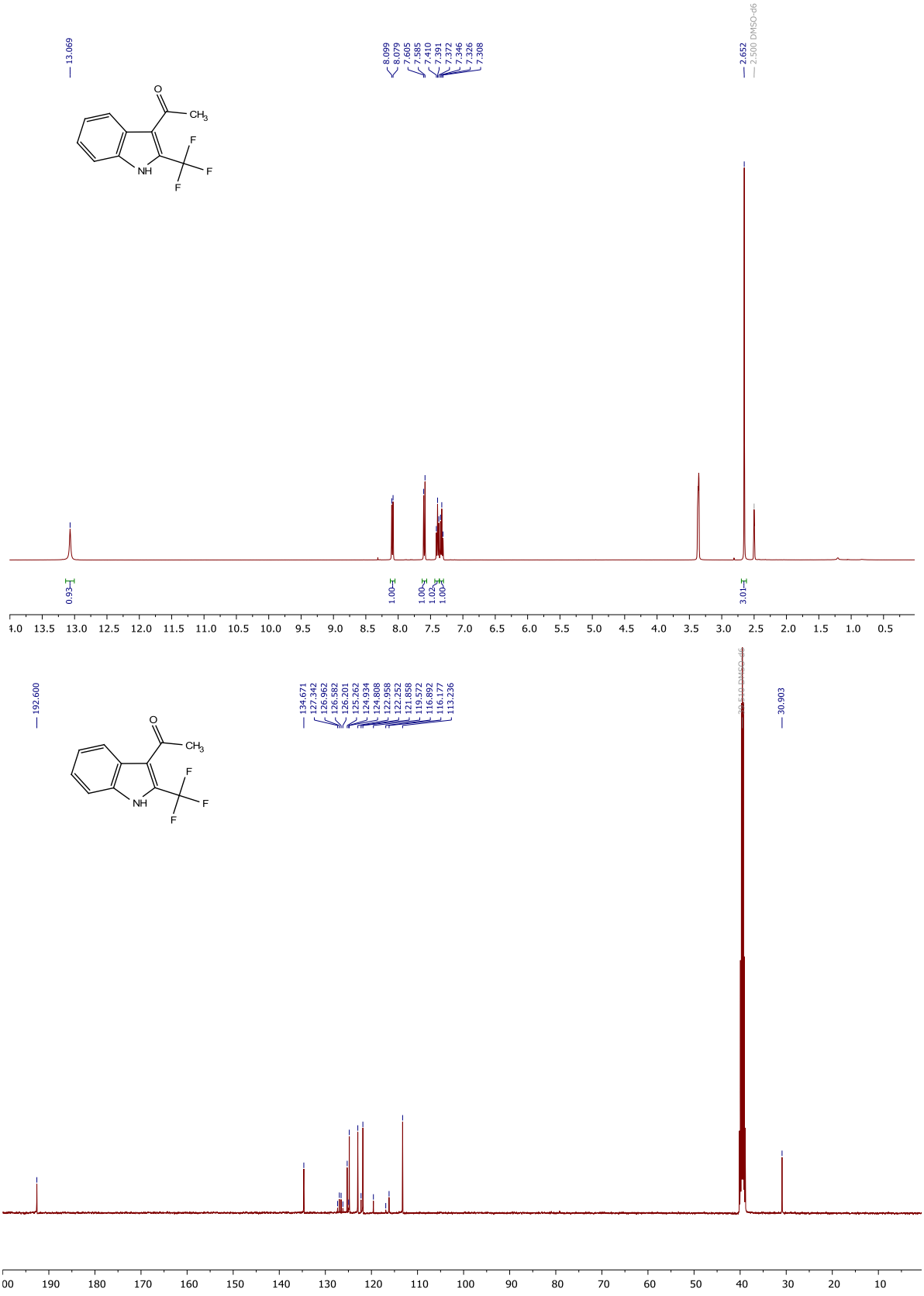
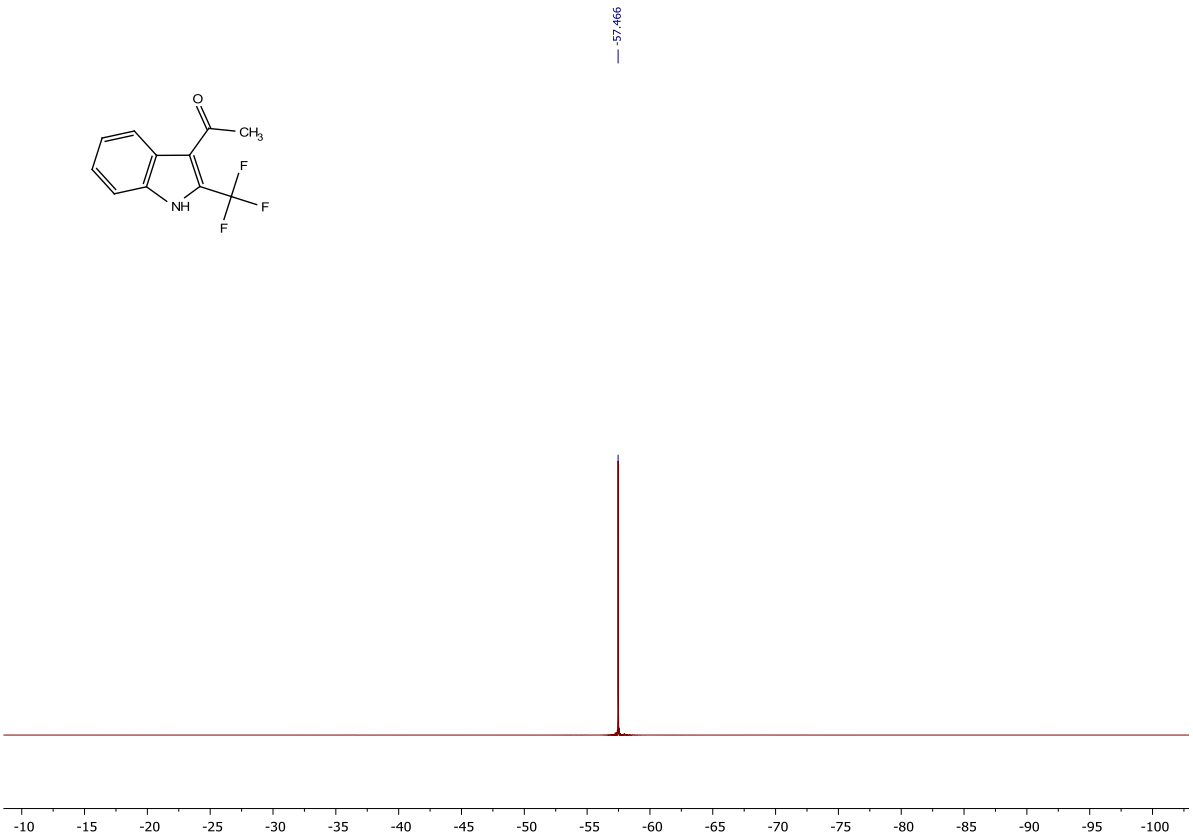
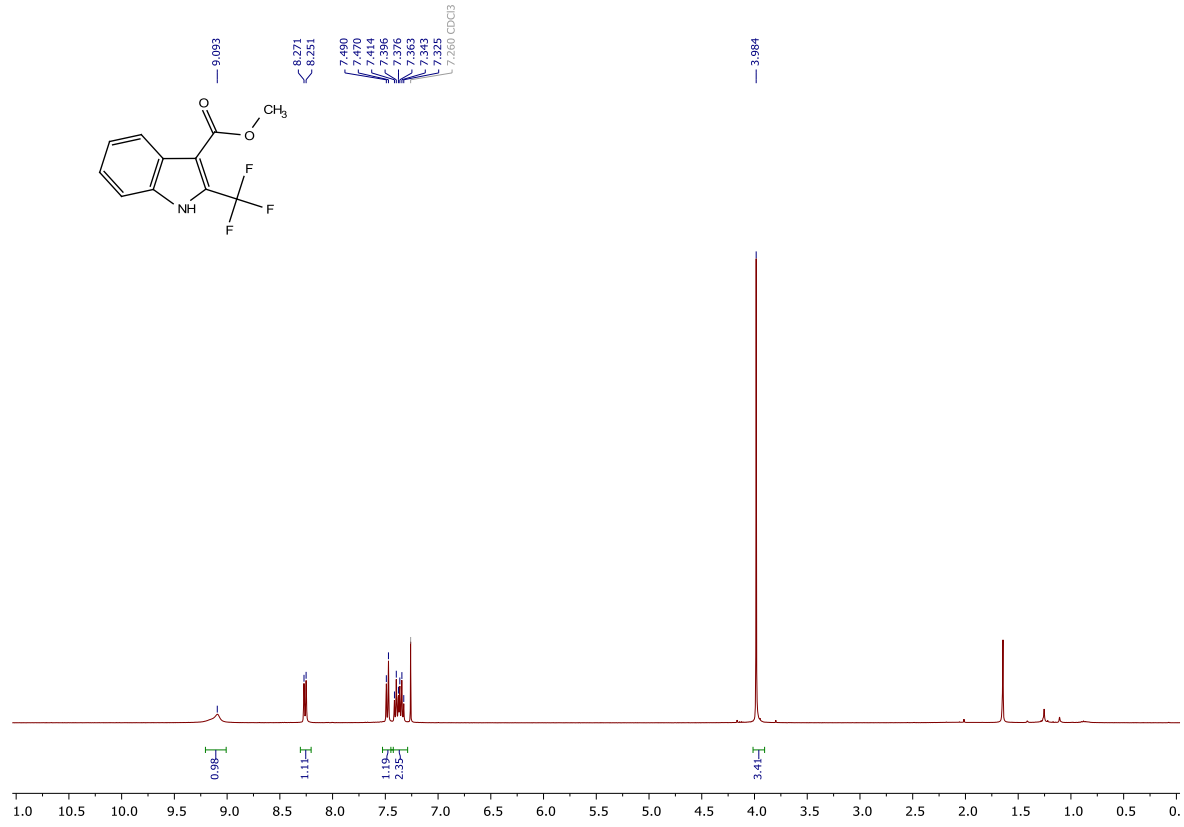
-90
-95
-100
-105

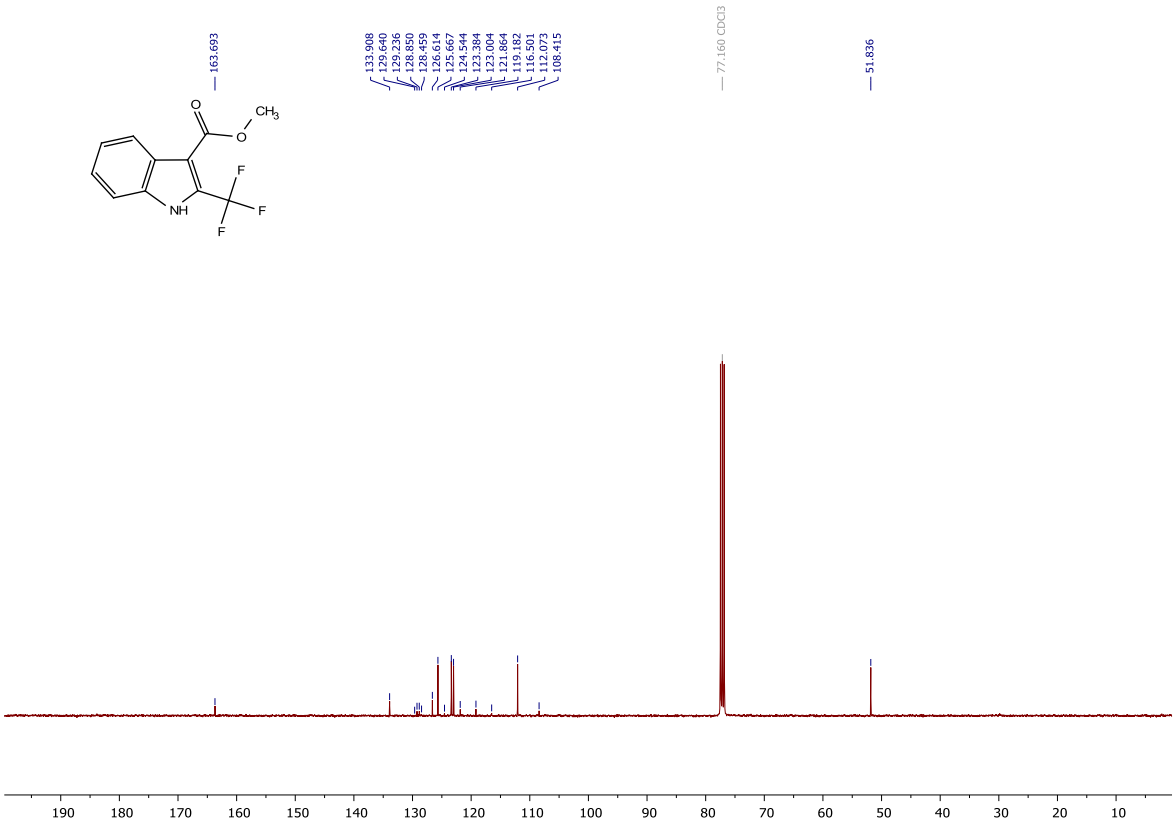
1-(2-(Trifluoromethyl)-1*H*-indol-3-yl)ethan-1-one (10)





Methyl 2-(trifluoromethyl)-1*H*-indole-3-carboxylate (11)

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125.667
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123.384
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116.501
112.073
108.415
77.160 CDCl3
51.836
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F
F
F
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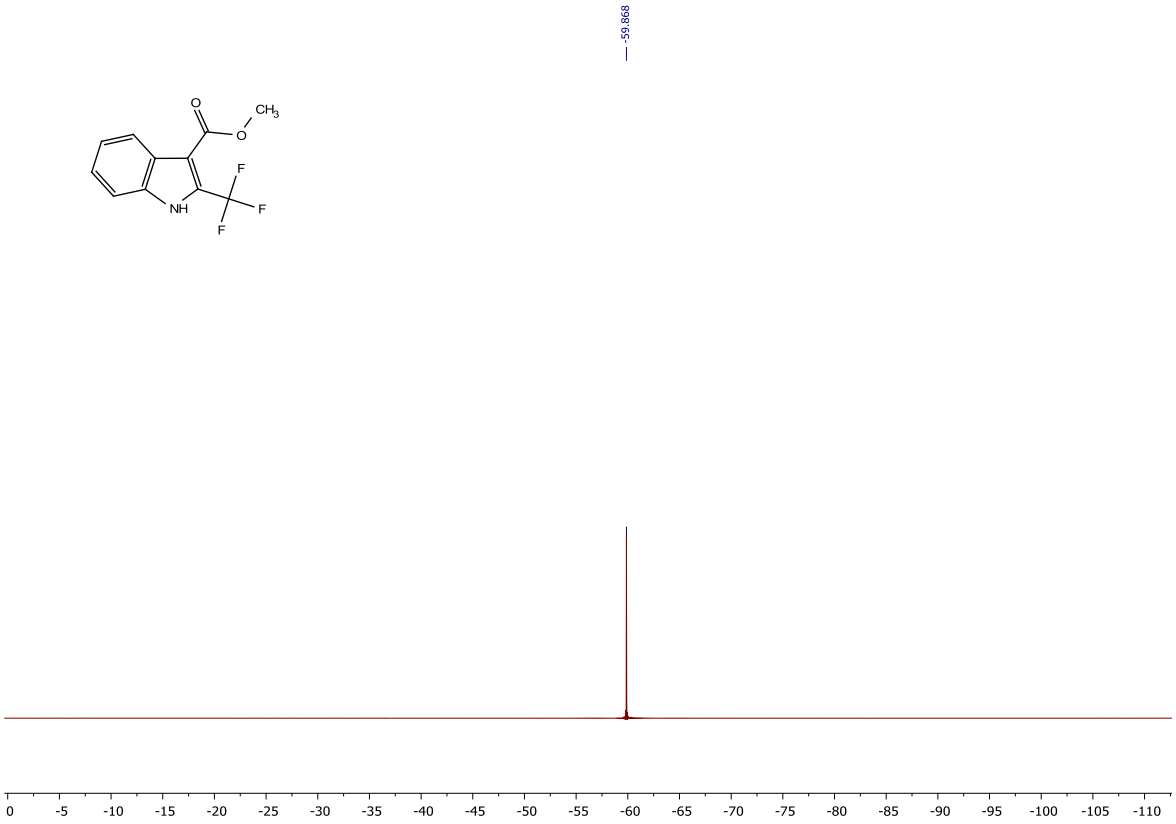
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-50
-55
-60
-65
-70
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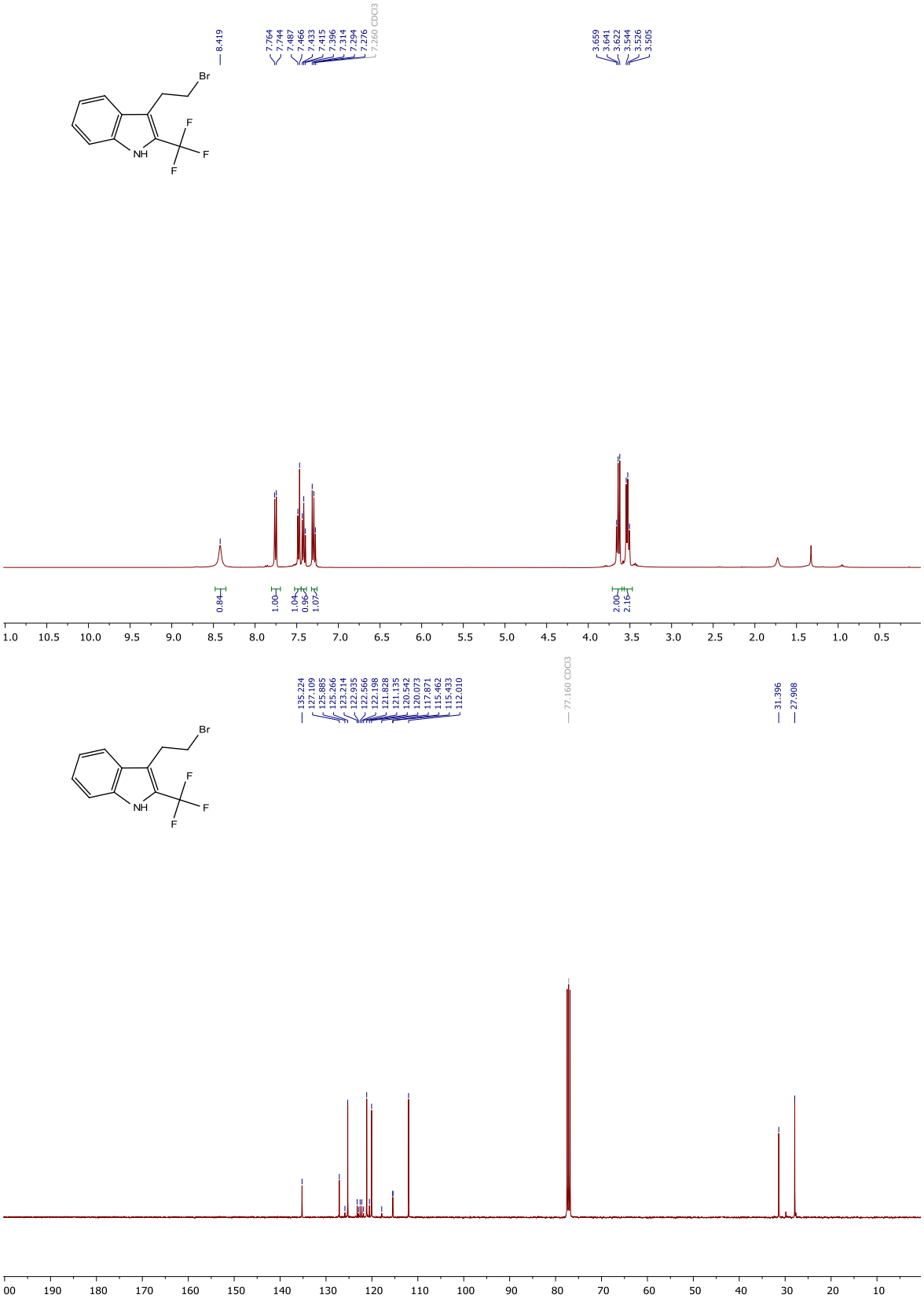
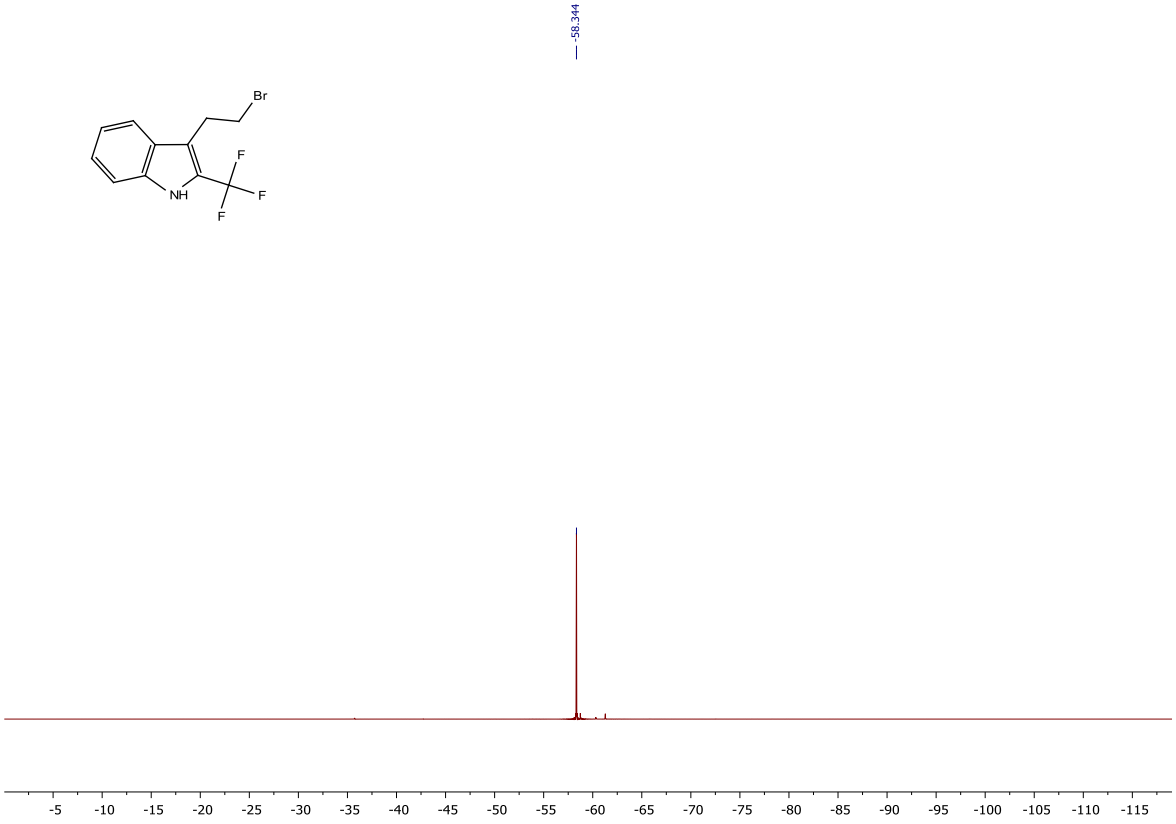
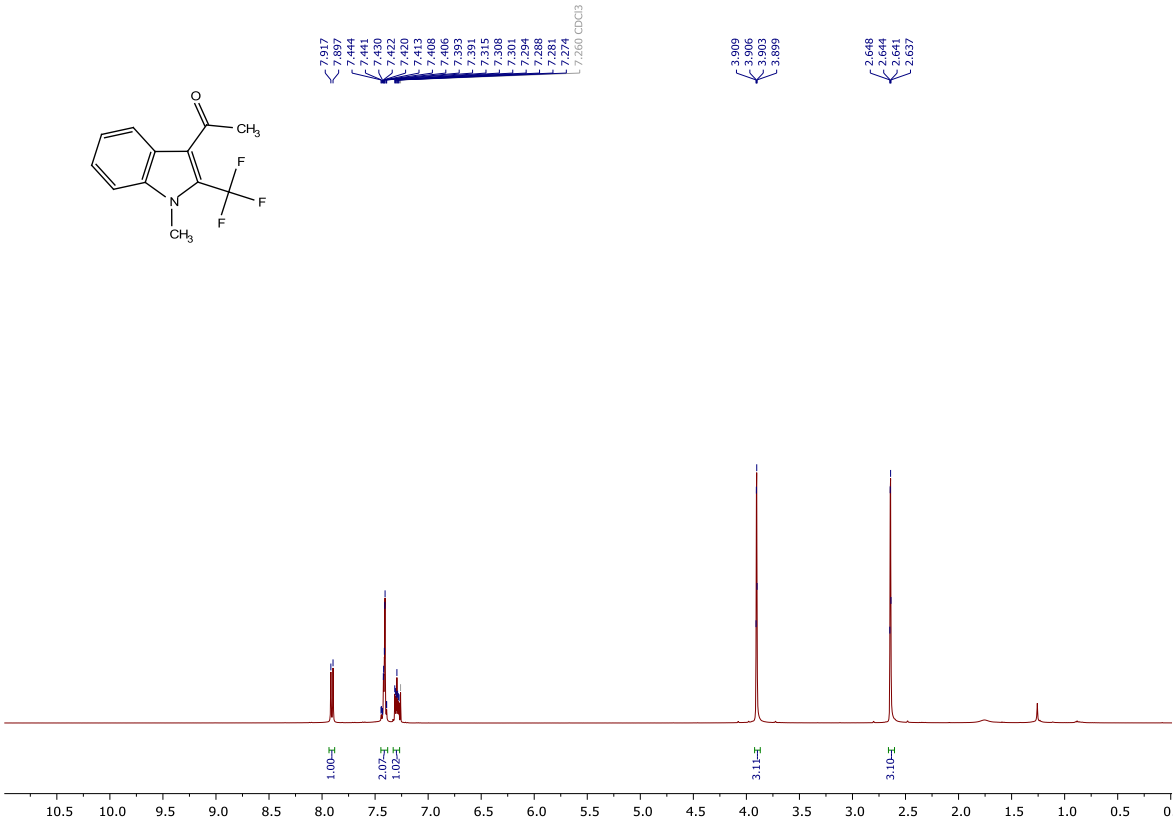
-90
-95
-100
-105
-110

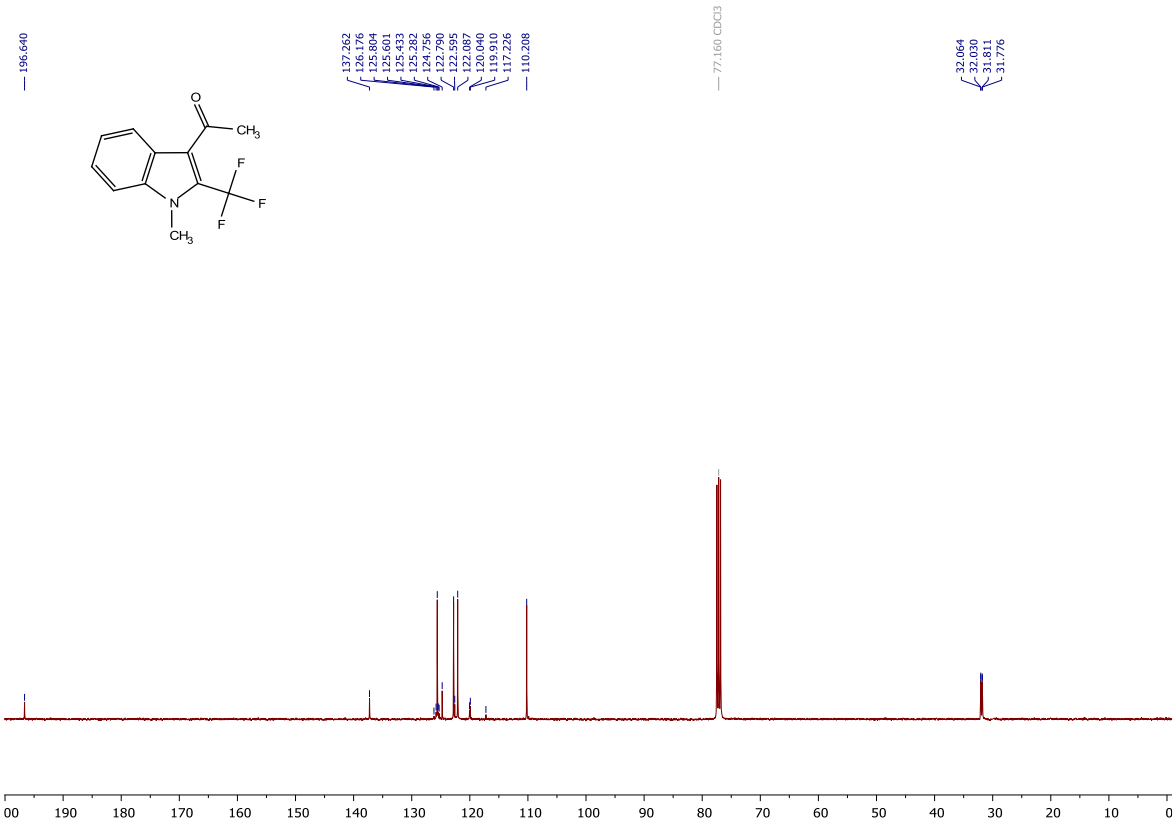
3-(2-Bromoethyl)-2-(trifluoromethyl)-1*H*-indole (12)





1-(1-Methyl-2-(trifluoromethyl)-1*H*-indol-3-yl)ethan-1-one (13)

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119.910
117.226
110.208
77.160 CDCl3
32.064
32.030
31.811
31.776
O
CH3
F
F
F
N
CH3
00
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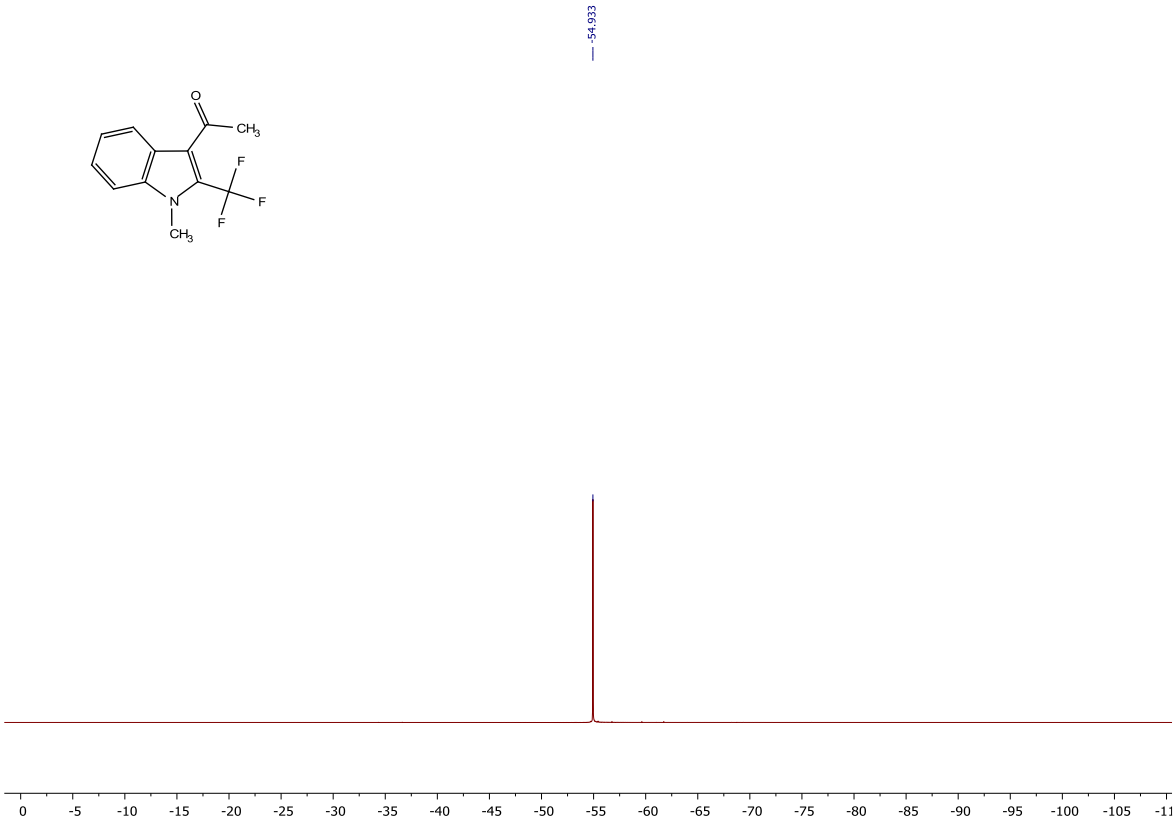
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-40
-45
-50
-55
-60
-65
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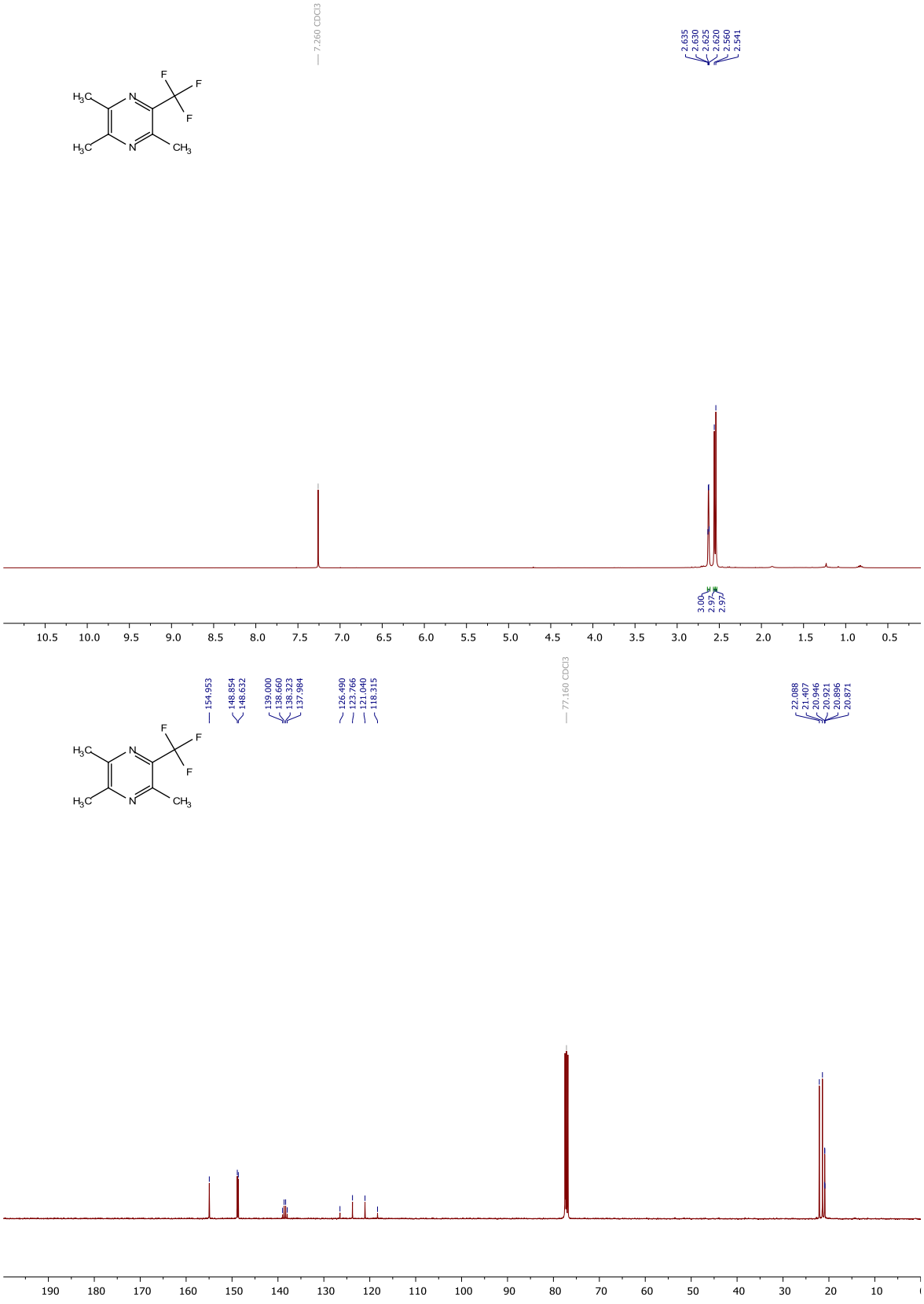
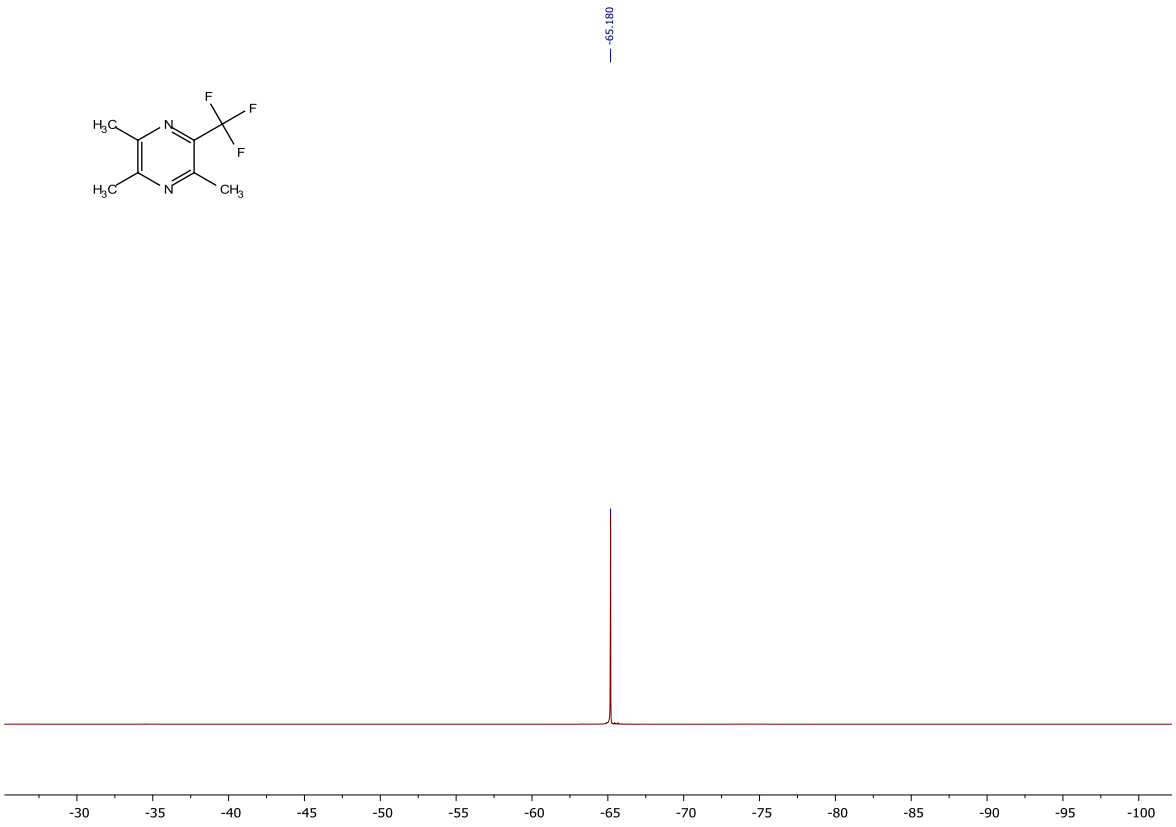
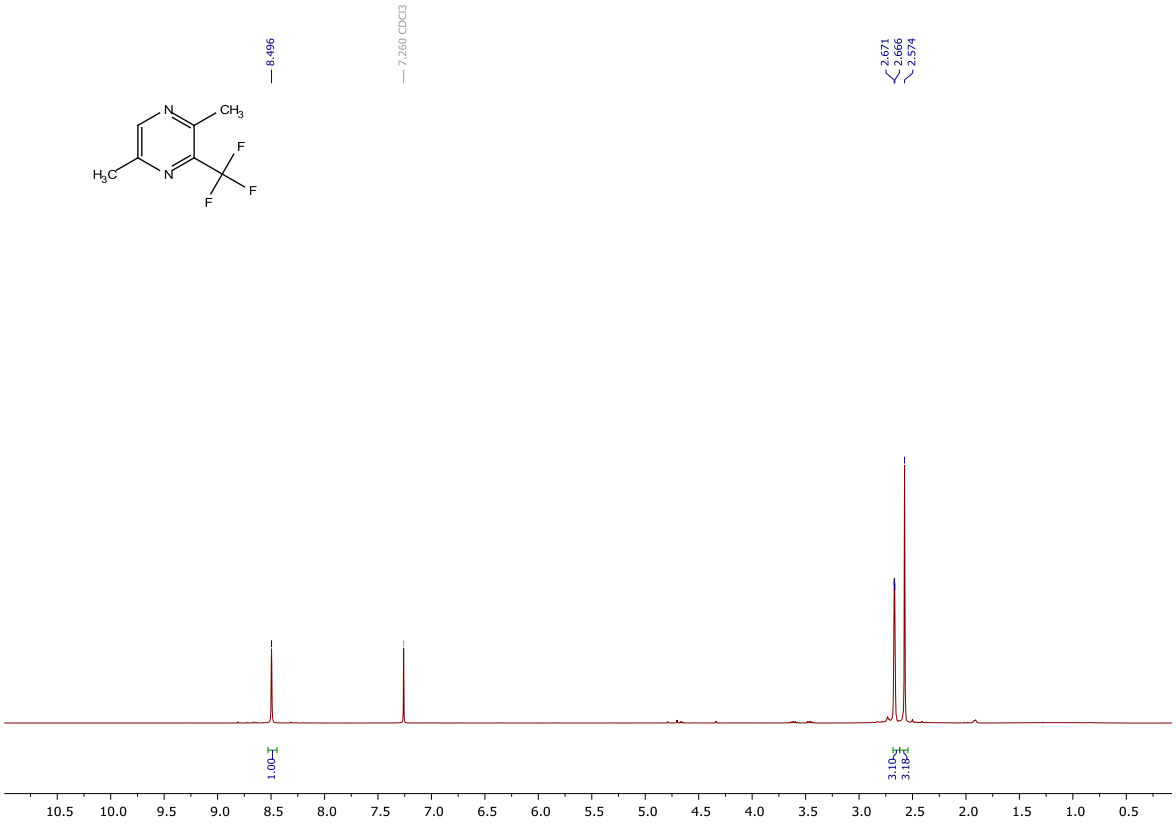
-105
-11

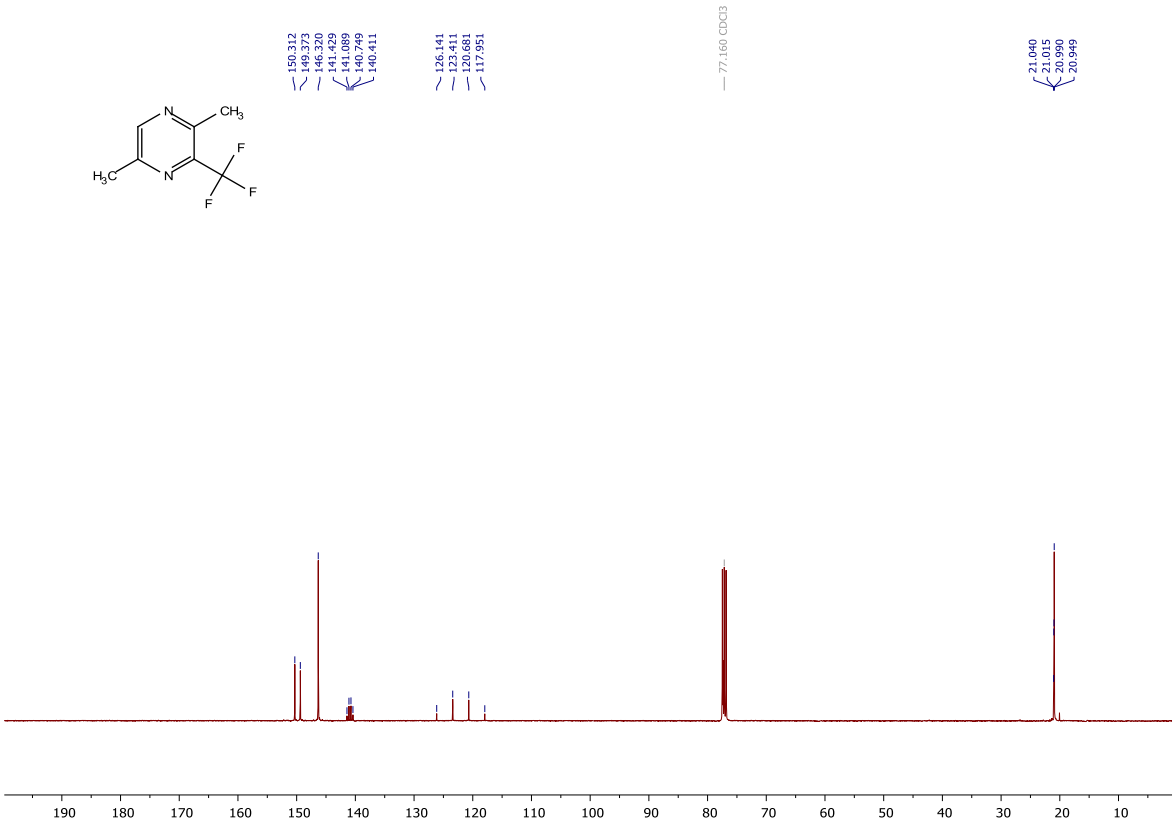
2,3,5-Trimethyl-6-(trifluoromethyl)pyrazine (16)





2,5-Dimethyl-3-(trifluoromethyl)pyrazine (17)

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77.160 CDCl3
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21.015
20.990
20.949
N
CH3
H3C
N
F
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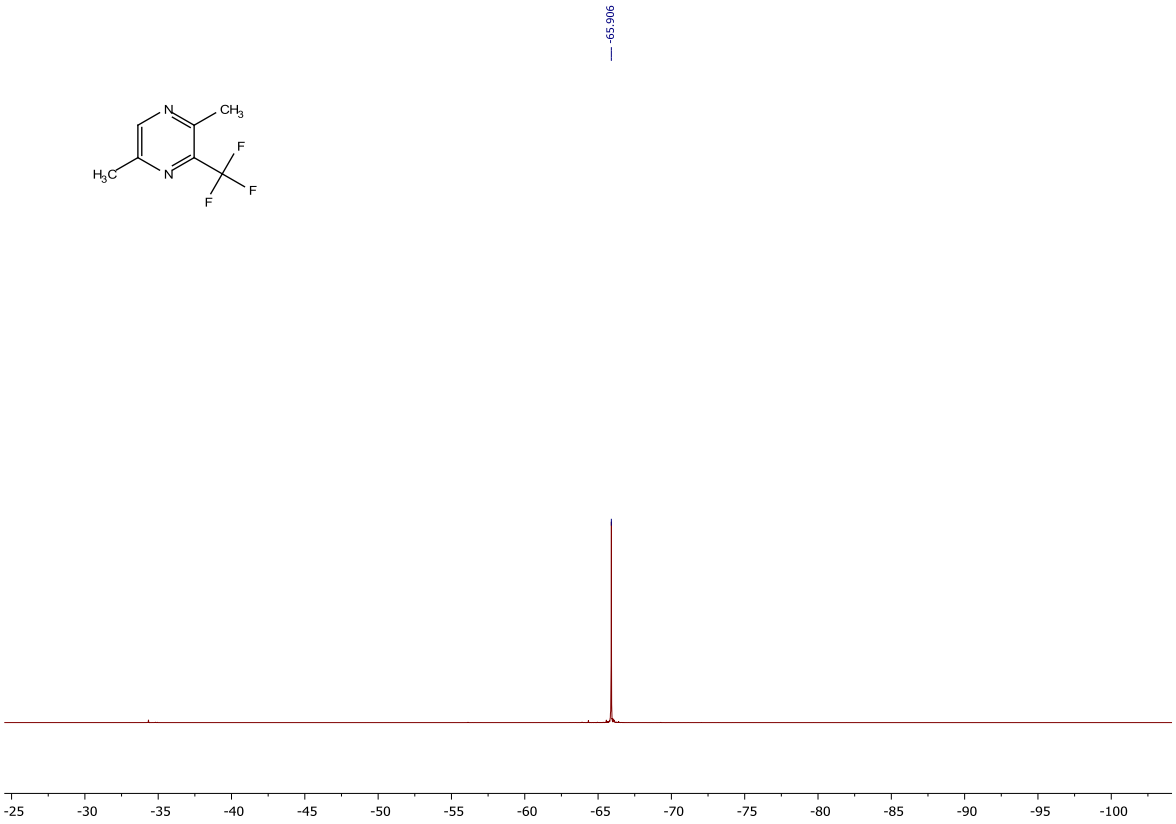
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-80
-85
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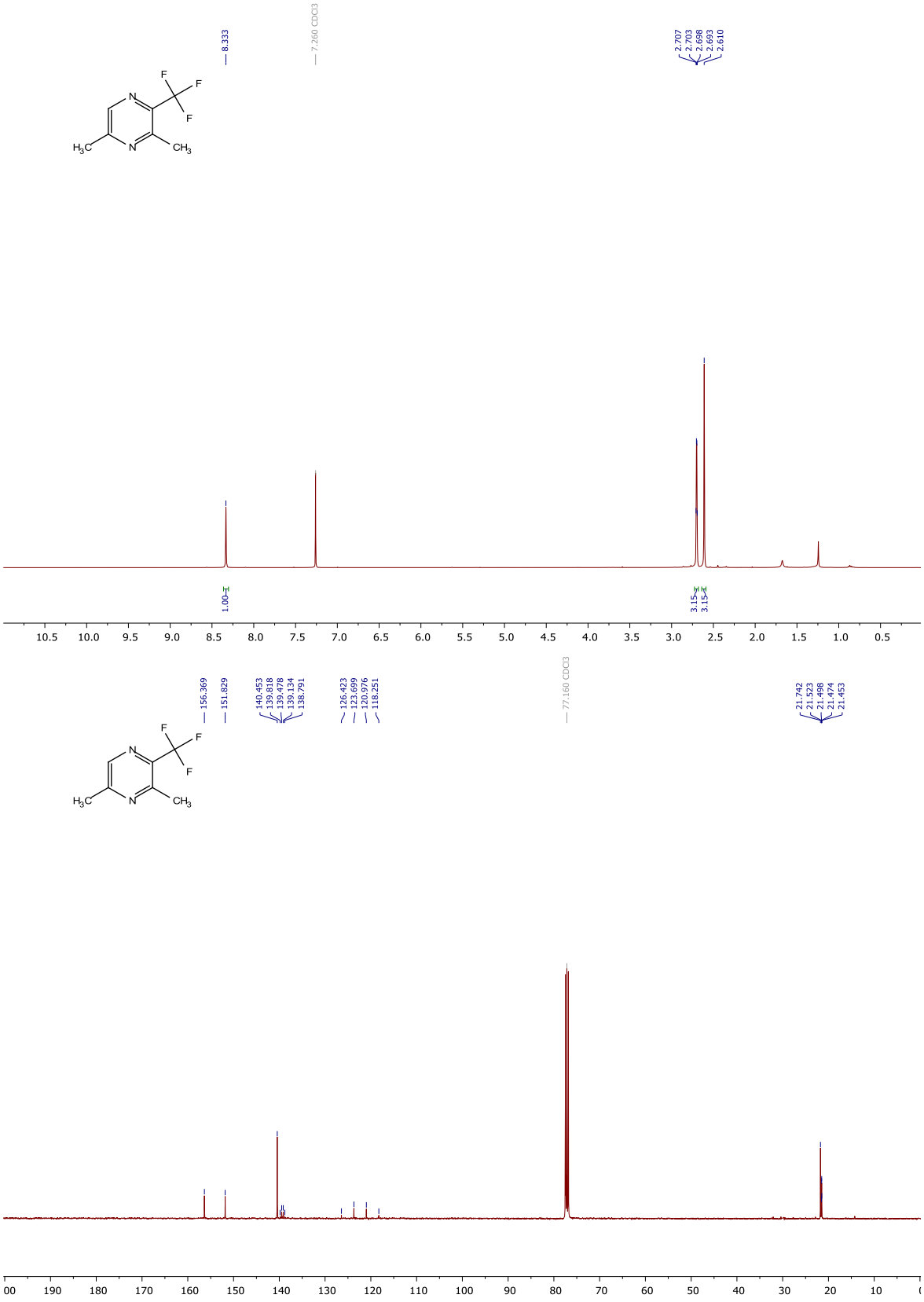
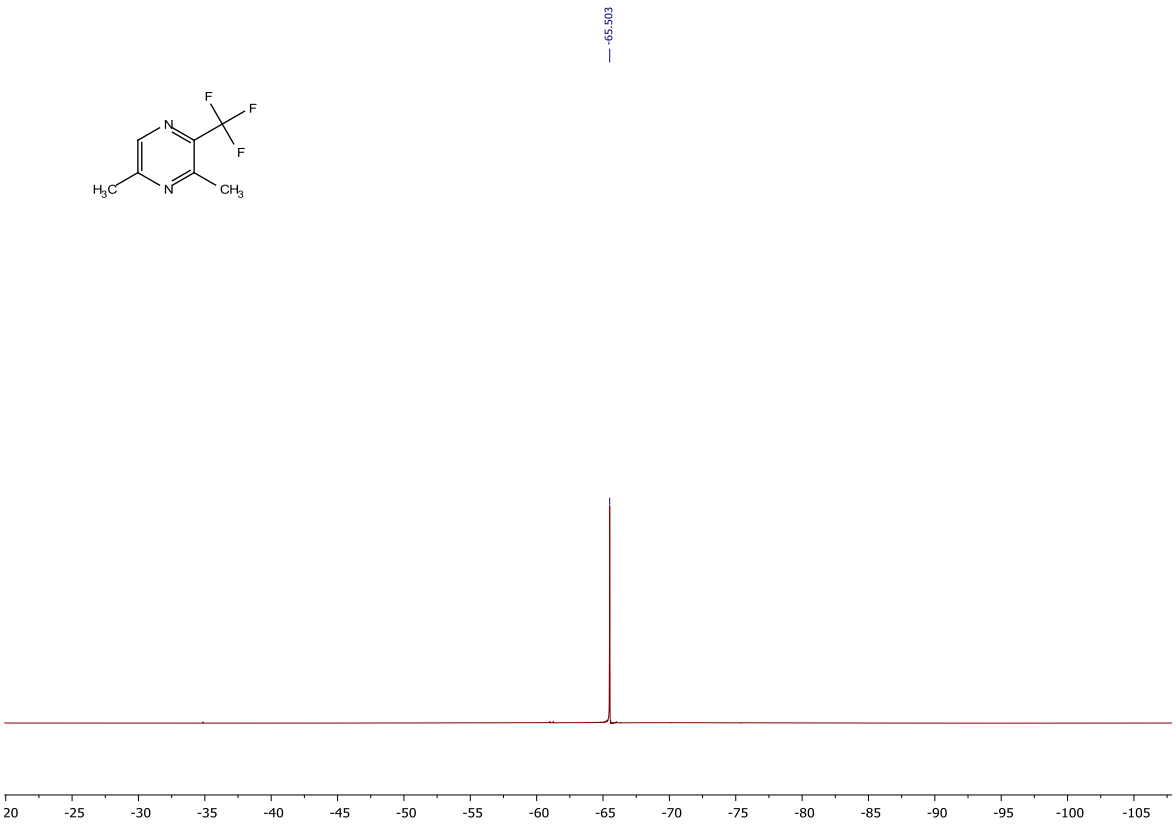
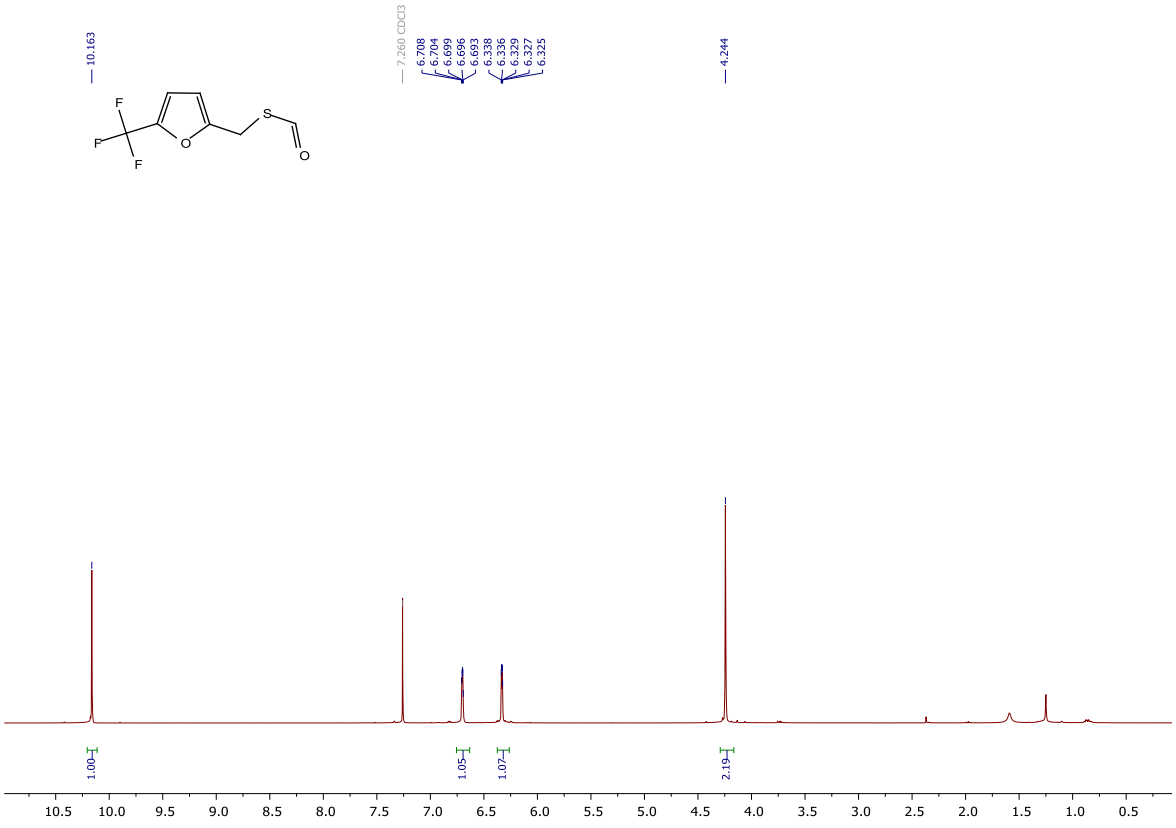
-95
-100

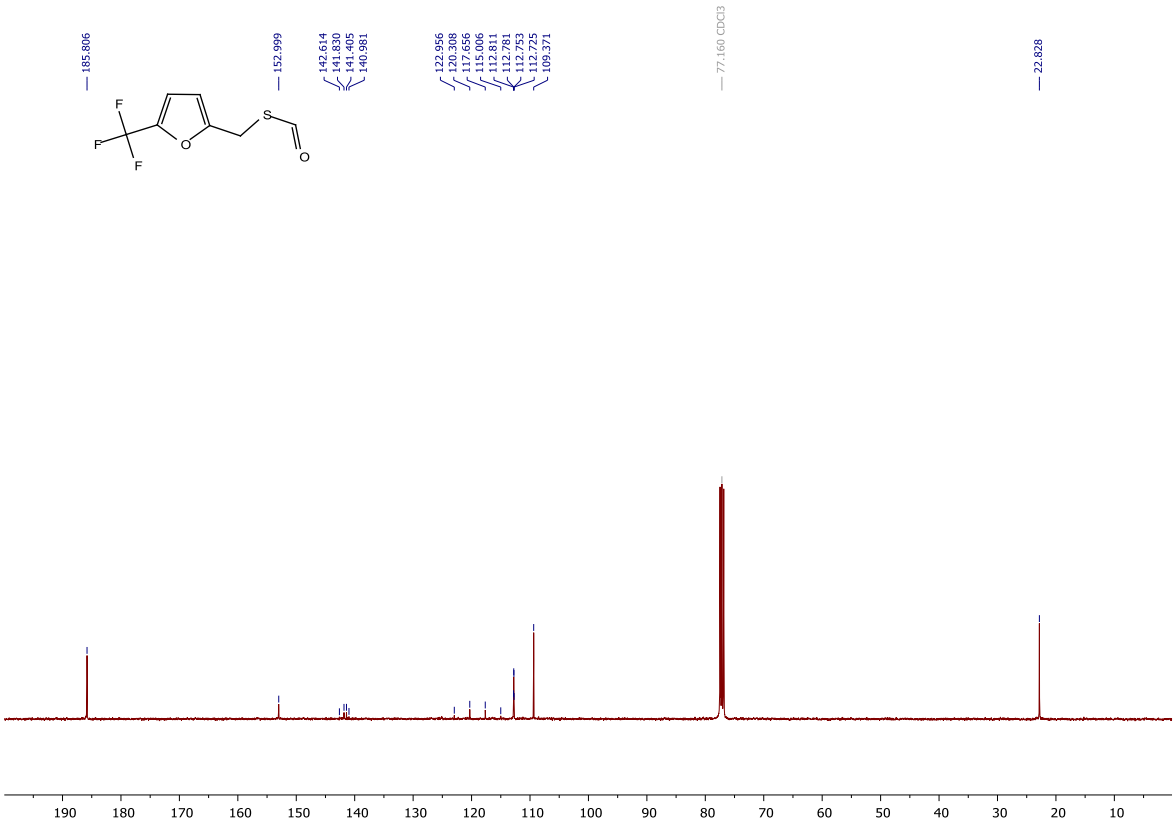
3,5-Dimethyl-2-(trifluoromethyl)pyrazine (18)

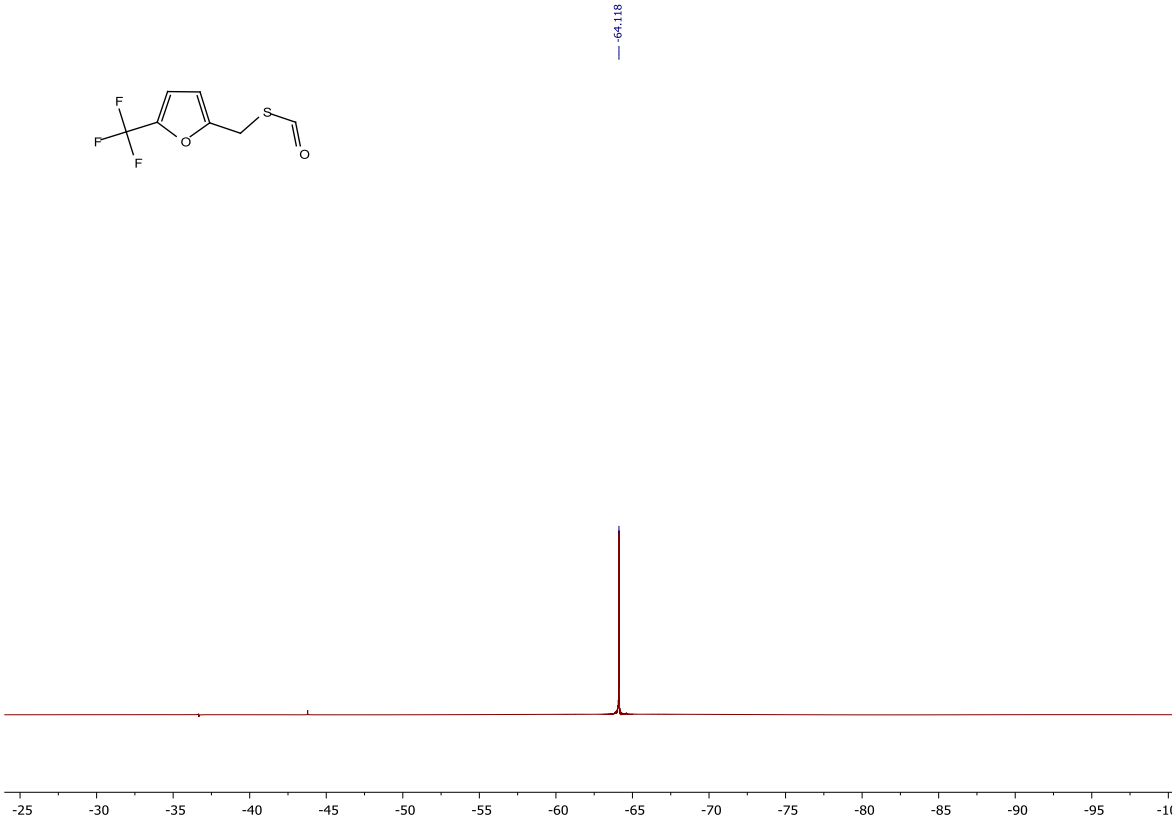




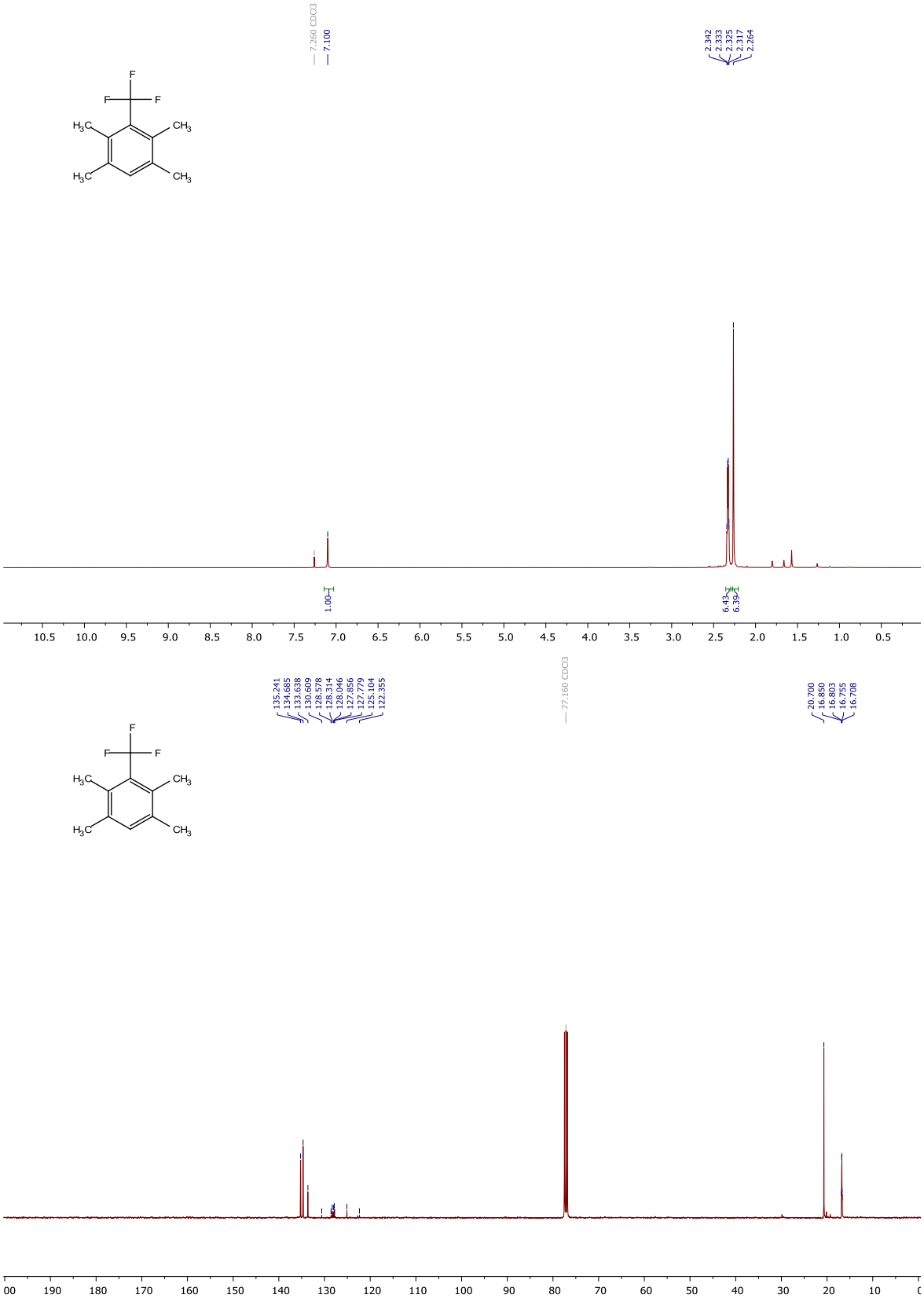
S-((5-(Trifluoromethyl)furan-2-yl)methyl)methanethioate (19)

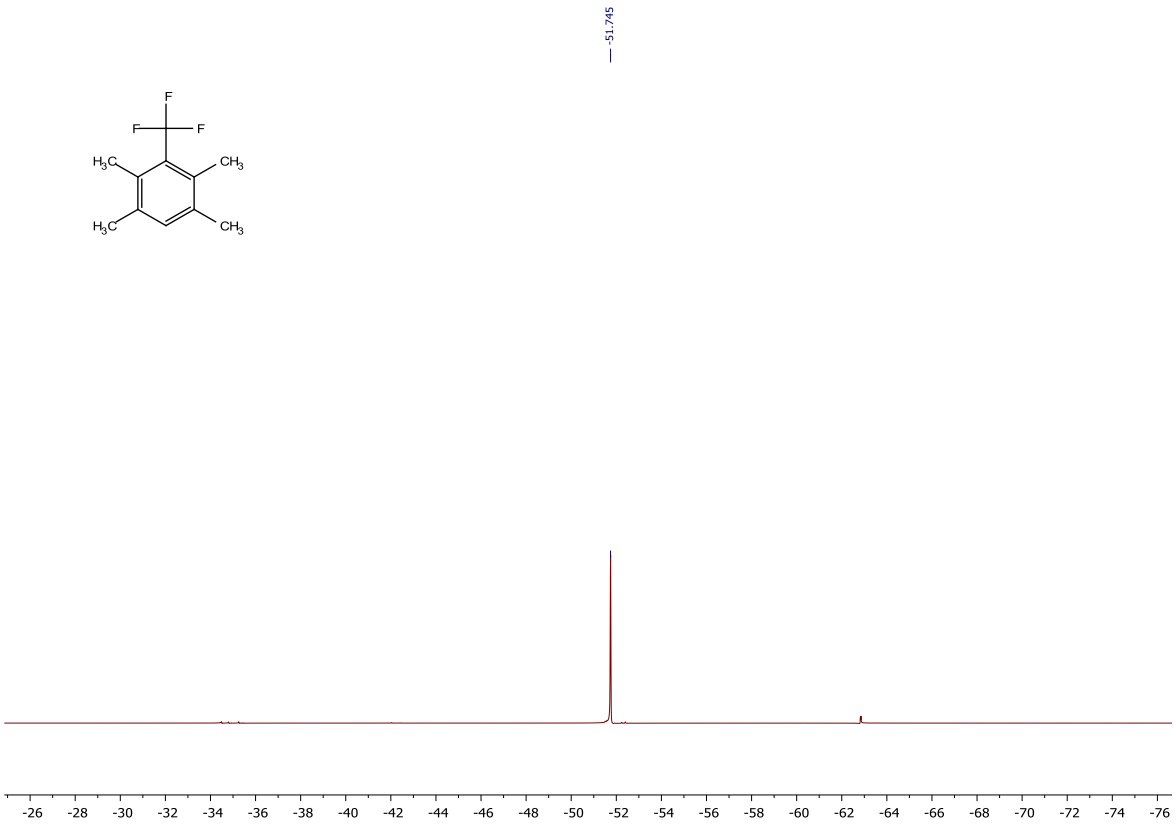



185.806
152.999
142.614
141.830
141.405
140.981
122.956
120.308
117.656
115.006
112.811
112.781
112.753
112.725
109.371
77.160 CDCl3
22.828
F
F
F
O
S
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

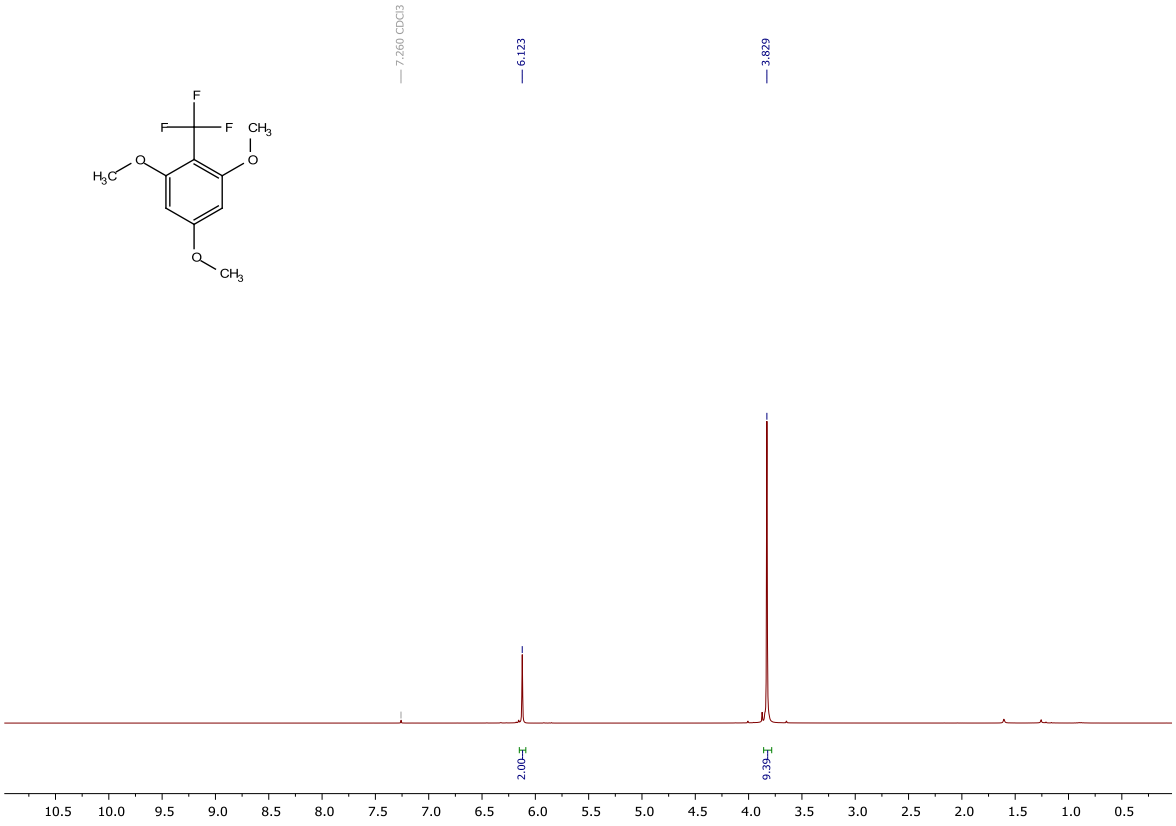

-64.118
F
F
F
O
S
O
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95

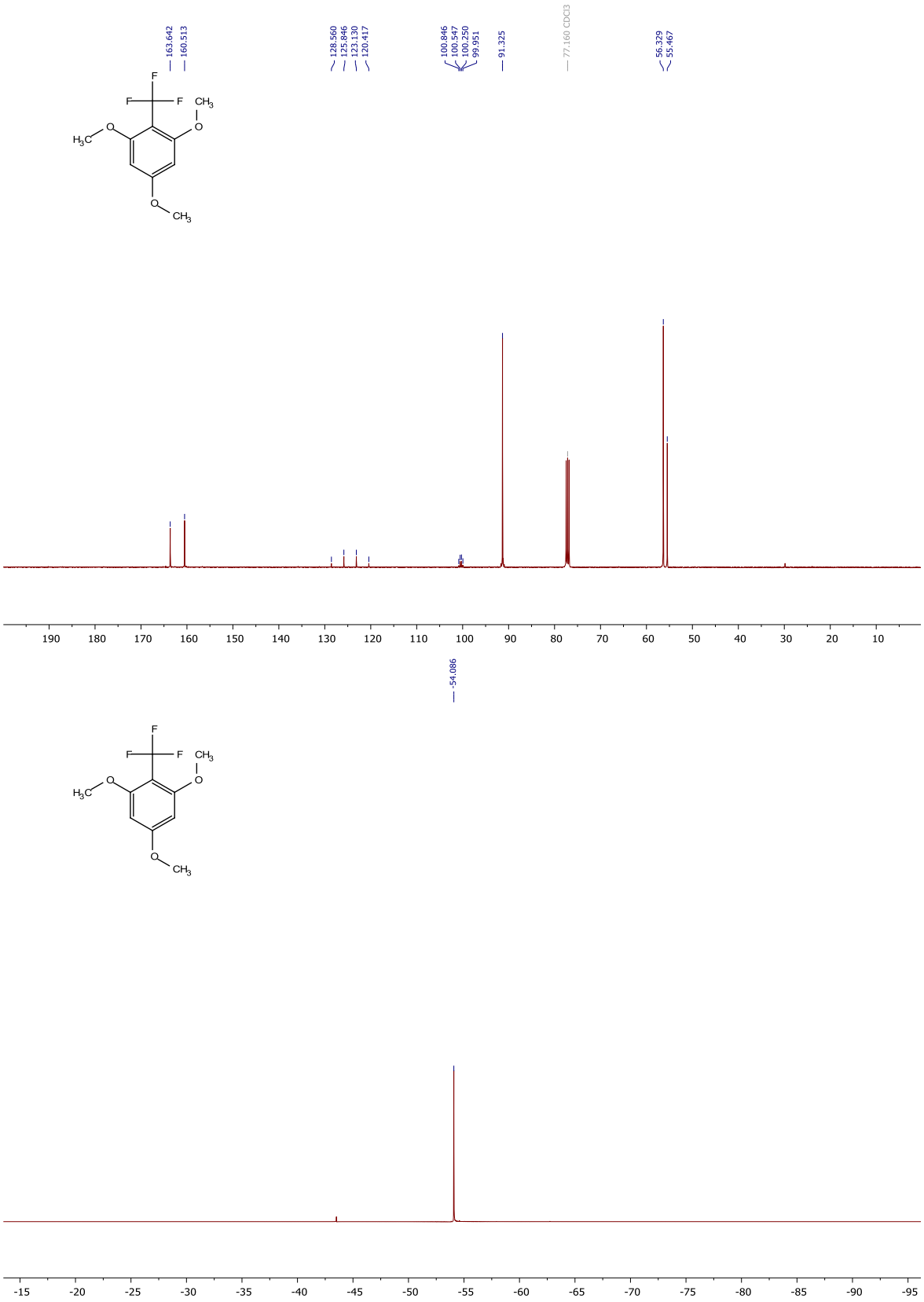
1,2,4,5-Tetramethyl-3-(trifluoromethyl)benzene (20)



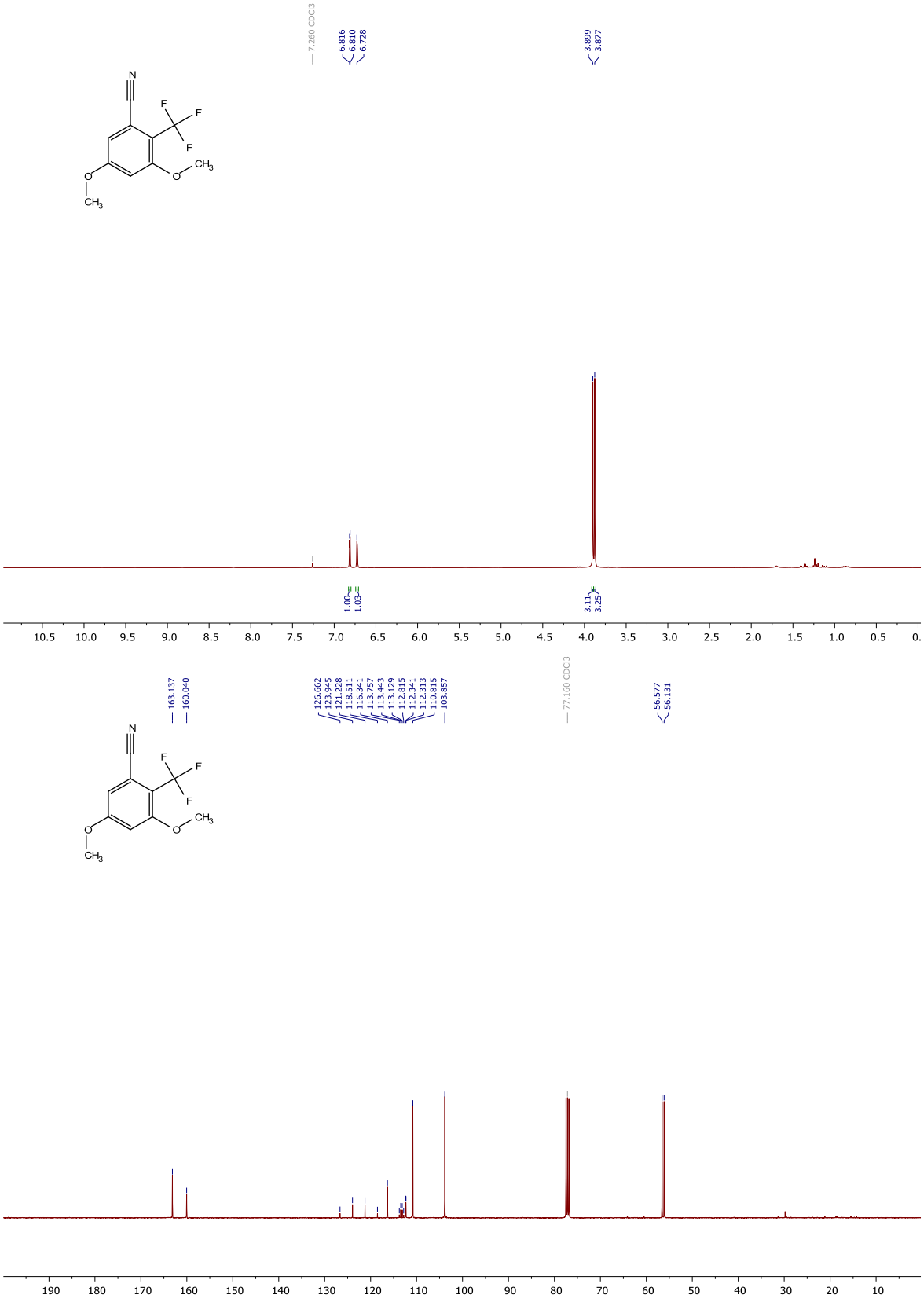


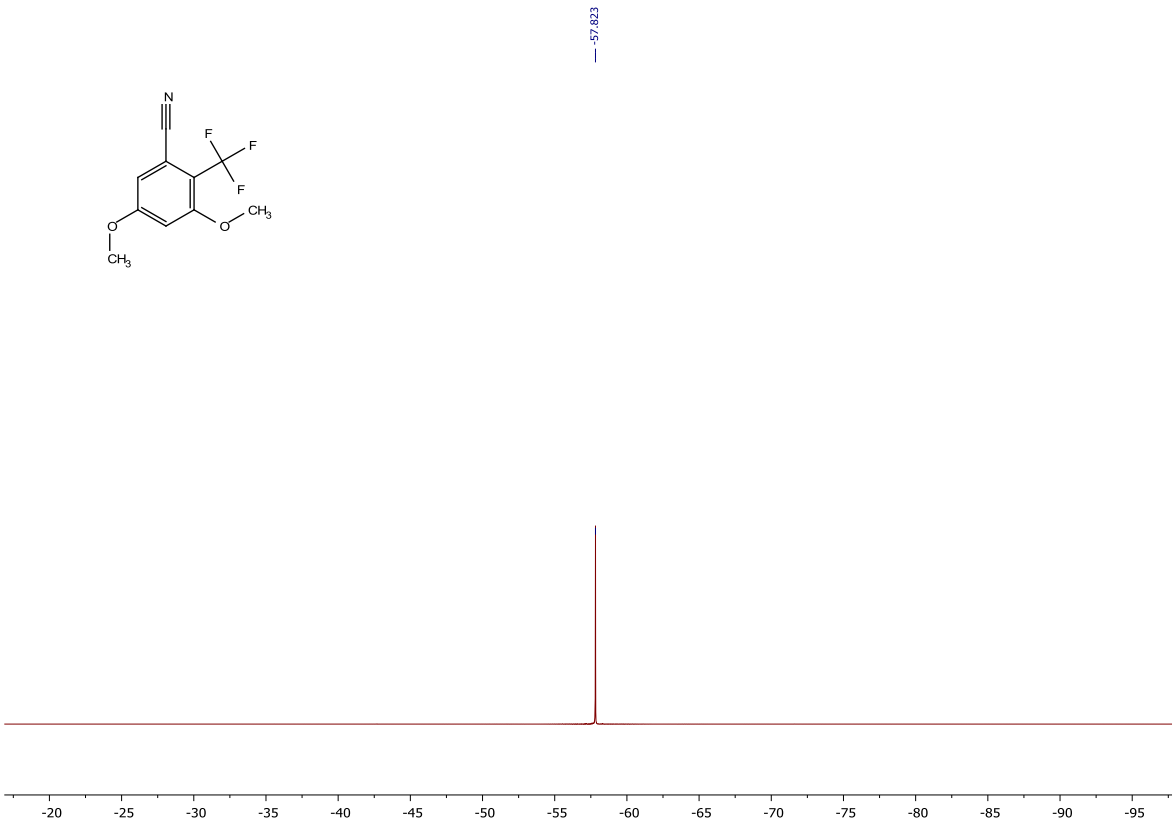
1,3,5-Trimethoxy-2-(trifluoromethyl)benzene (22)



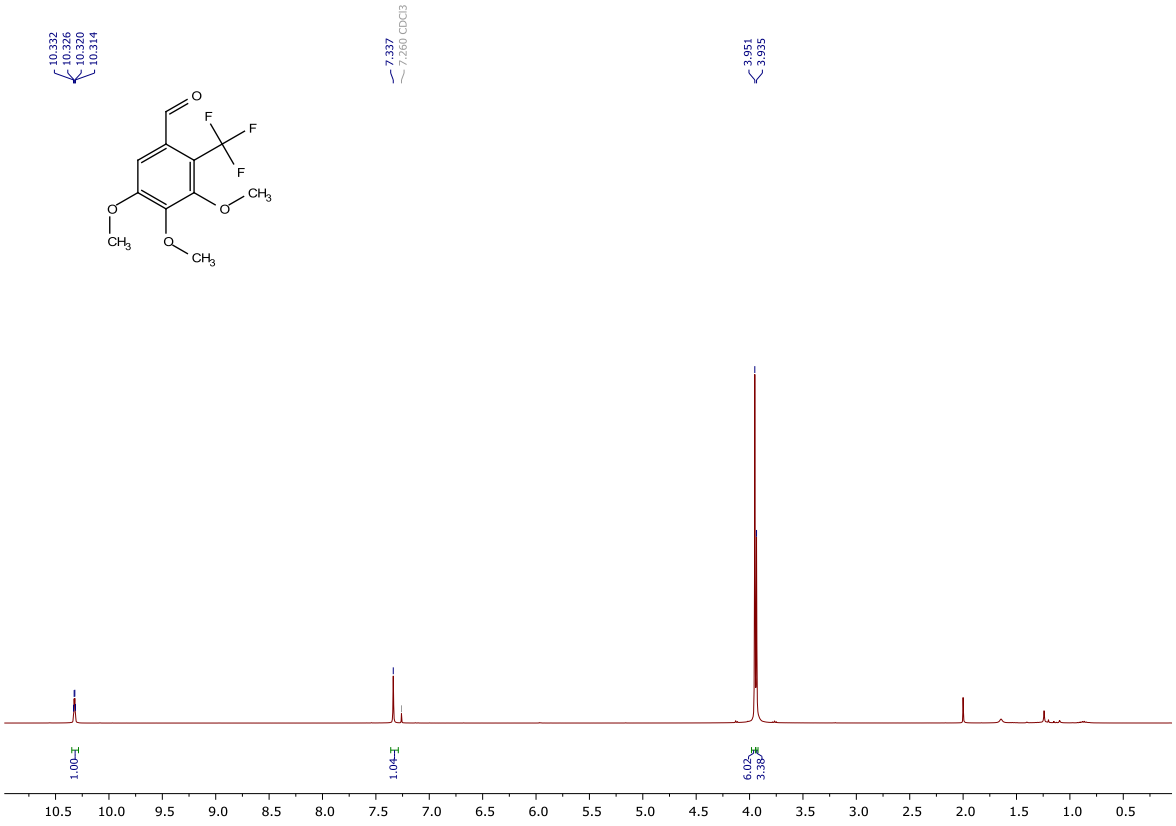

163.642
160.513
128.560
125.846
123.130
120.417
100.846
100.547
100.250
99.951
91.325
77.160 CDCl3
56.329
55.467
F
F
F
CH3
H3C
O
O
O
CH3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
-54.086
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95

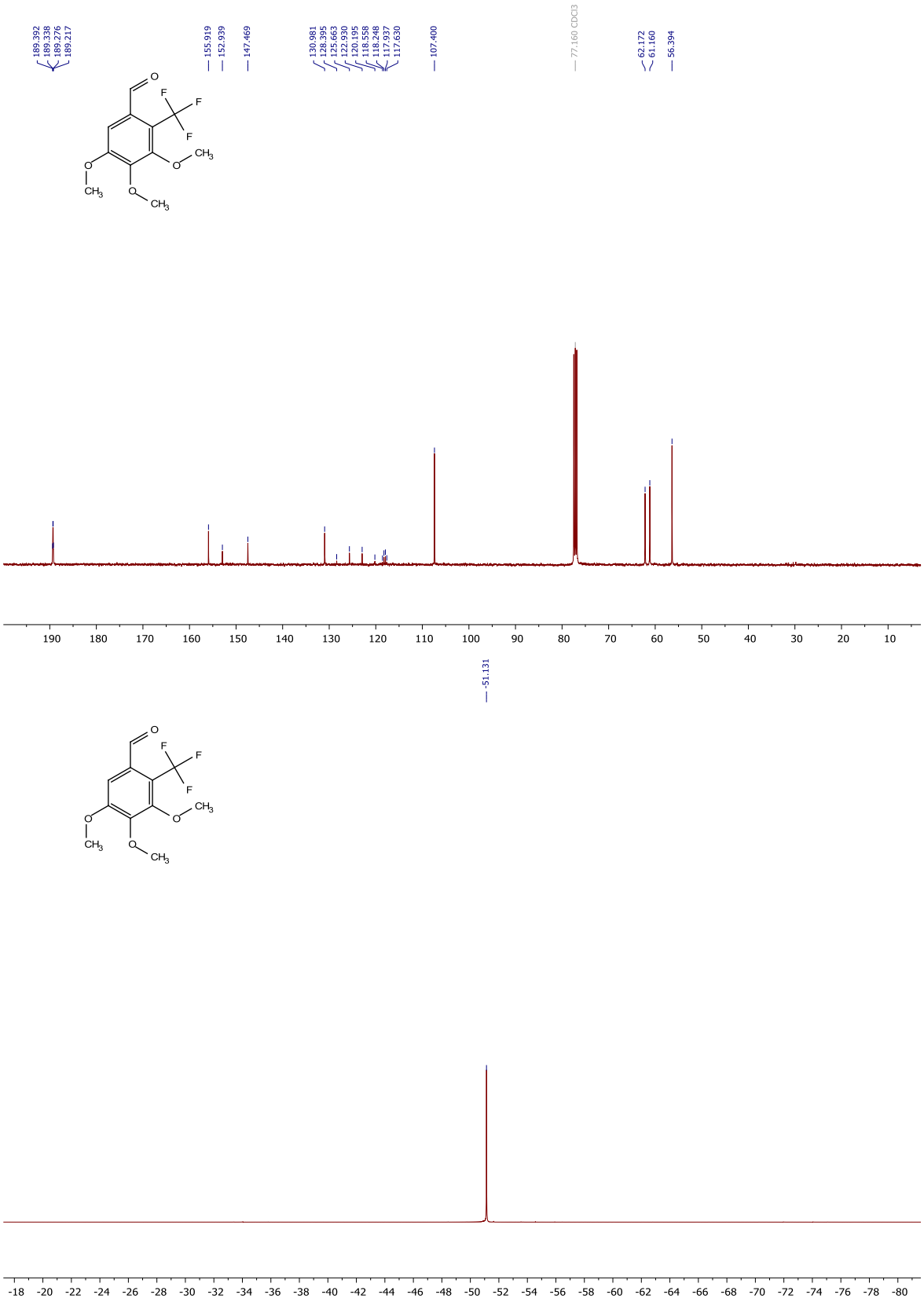
3,5-Dimethoxy-2-(trifluoromethyl)benzonitrile (23)



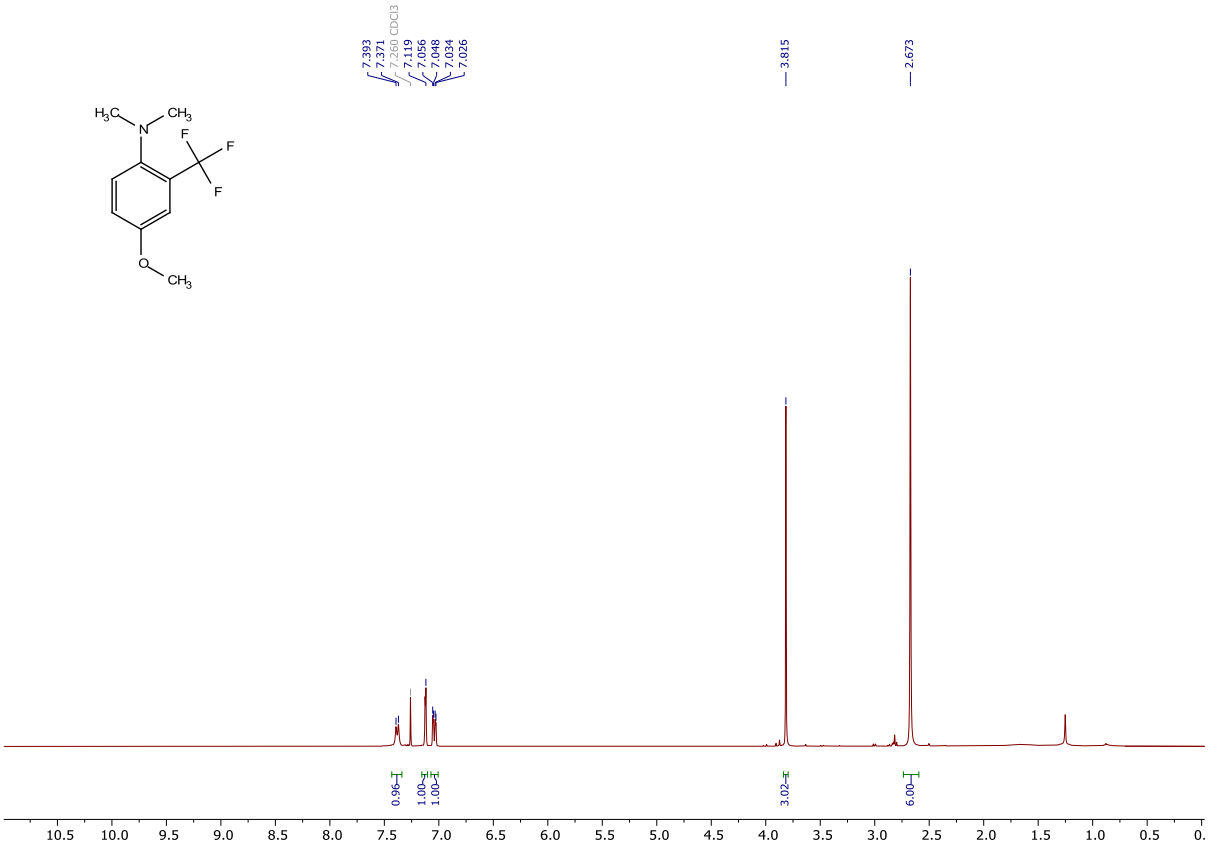


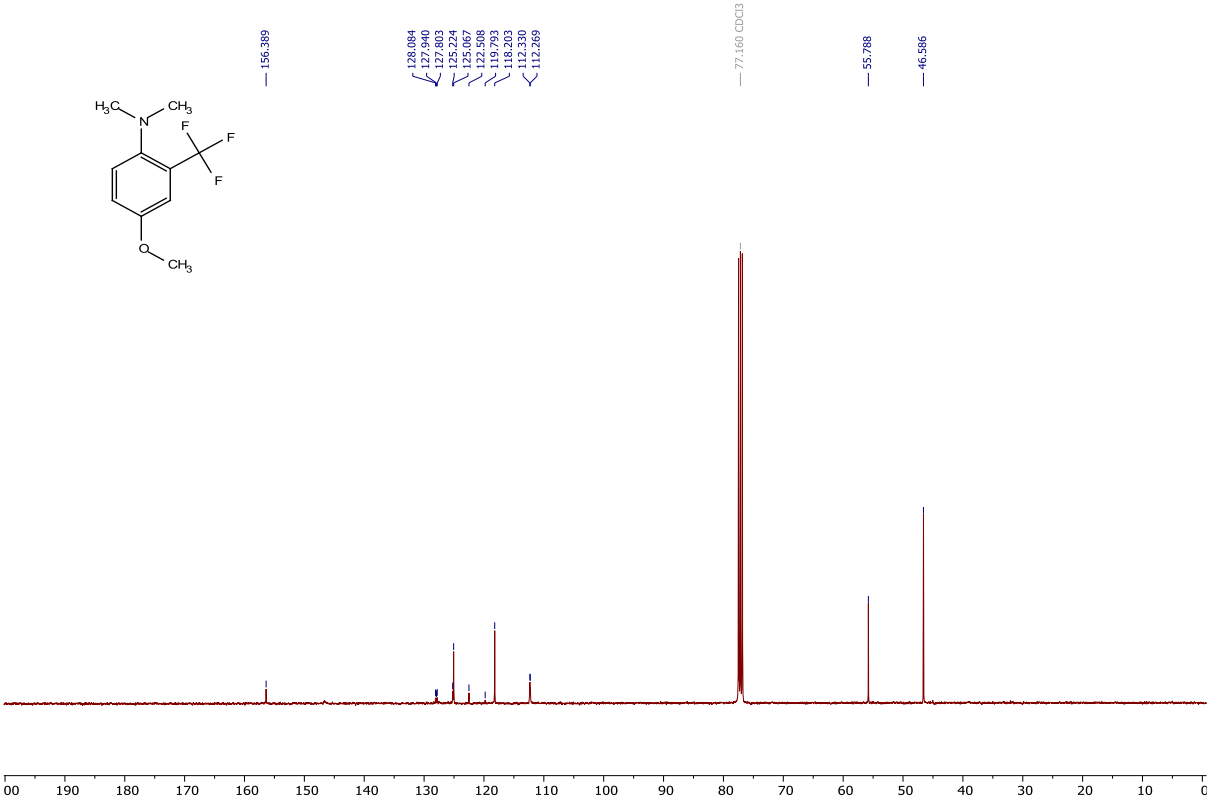
3,4,5-Trimethoxy-2-(trifluoromethyl)benzaldehyde (24)

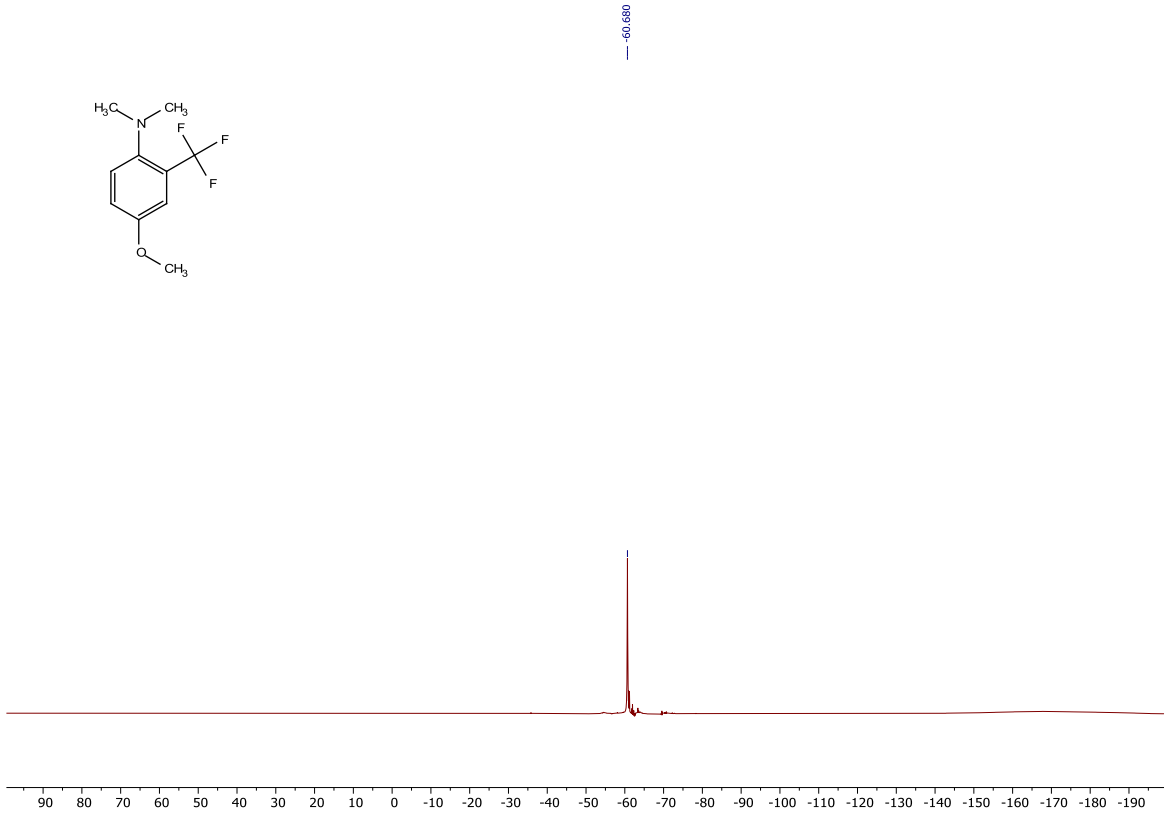



189.392
189.338
189.276
189.217
155.919
152.939
147.469
130.981
128.395
125.663
122.930
120.195
118.558
118.248
117.937
117.630
107.400
77.160 CDCl3
62.172
61.160
56.394
O
F
F
F
CH3
O
CH3
O
CH3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
-51.131
-18 -20 -22 -24 -26 -28 -30 -32 -34 -36 -38 -40 -42 -44 -46 -48 -50 -52 -54 -56 -58 -60 -62 -64 -66 -68 -70 -72 -74 -76 -78 -80

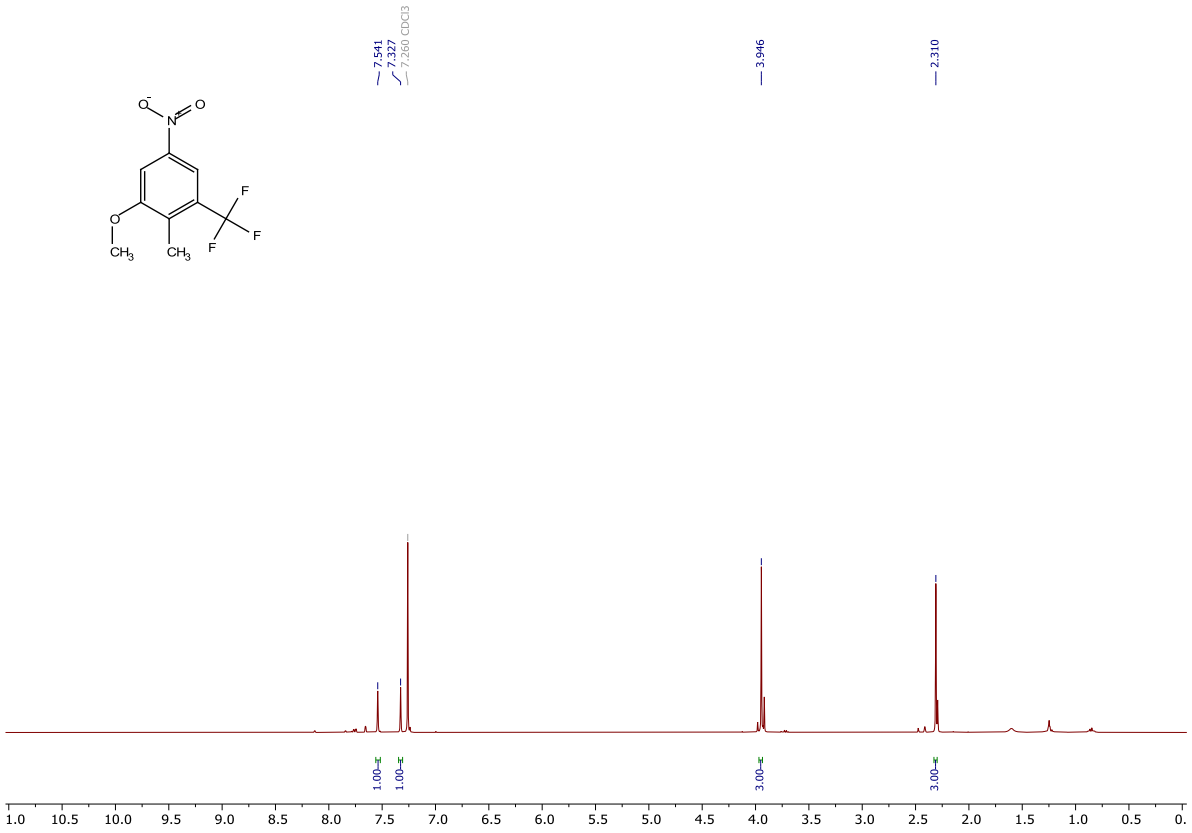
4-Methoxy-N,N-dimethyl-2-(trifluoromethyl)aniline (25)

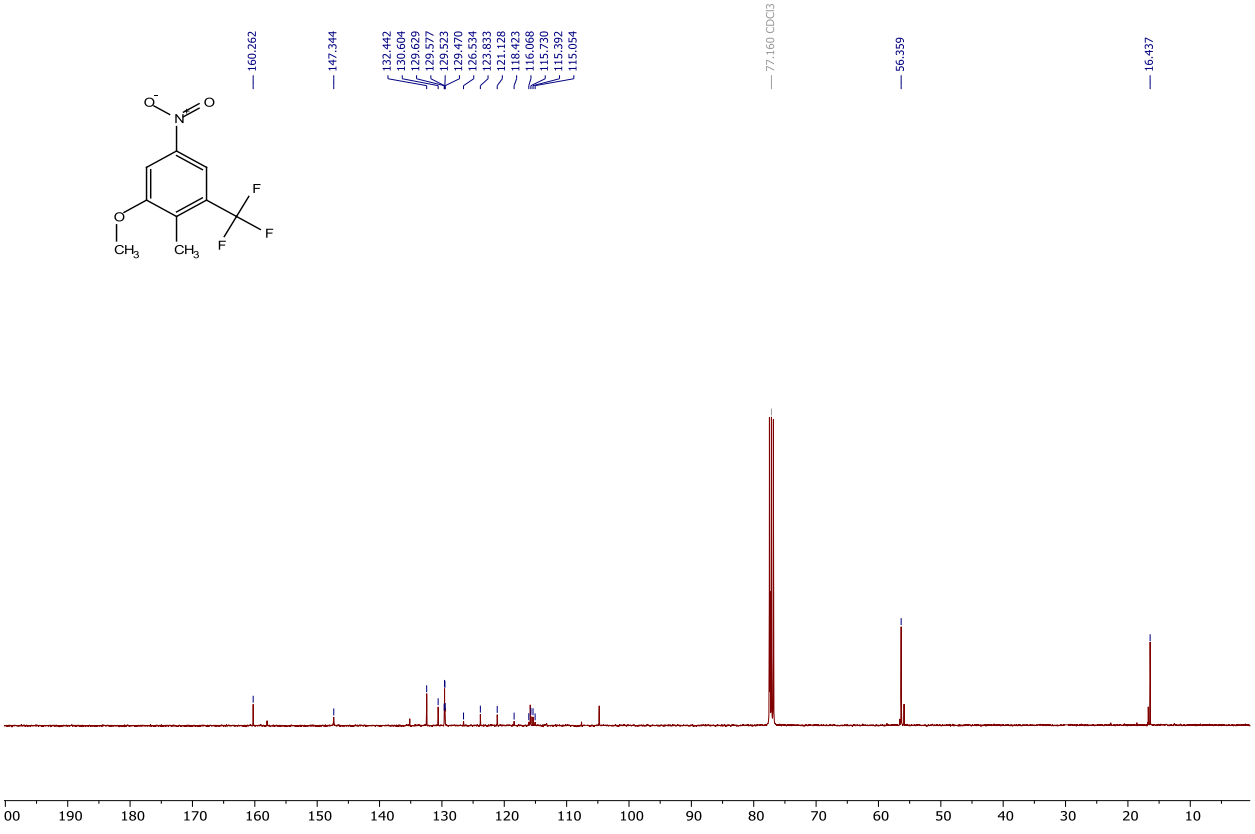


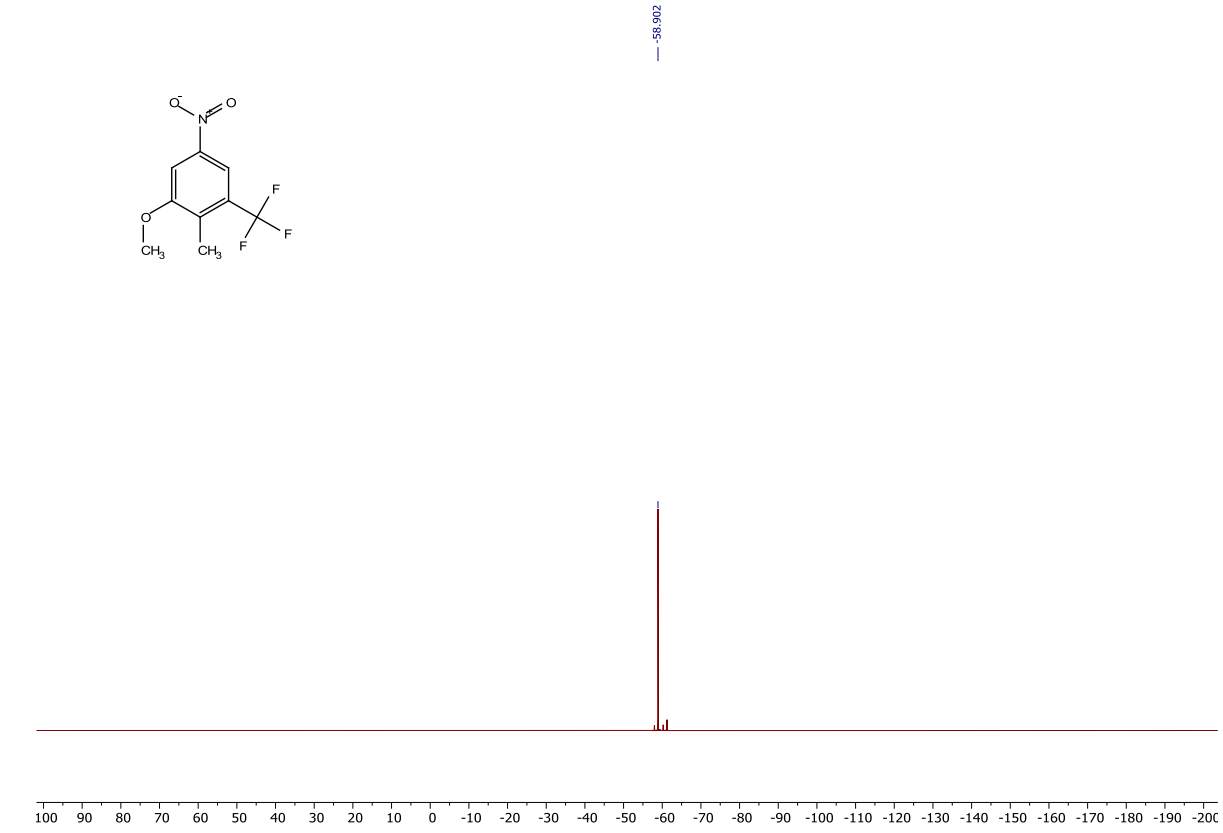




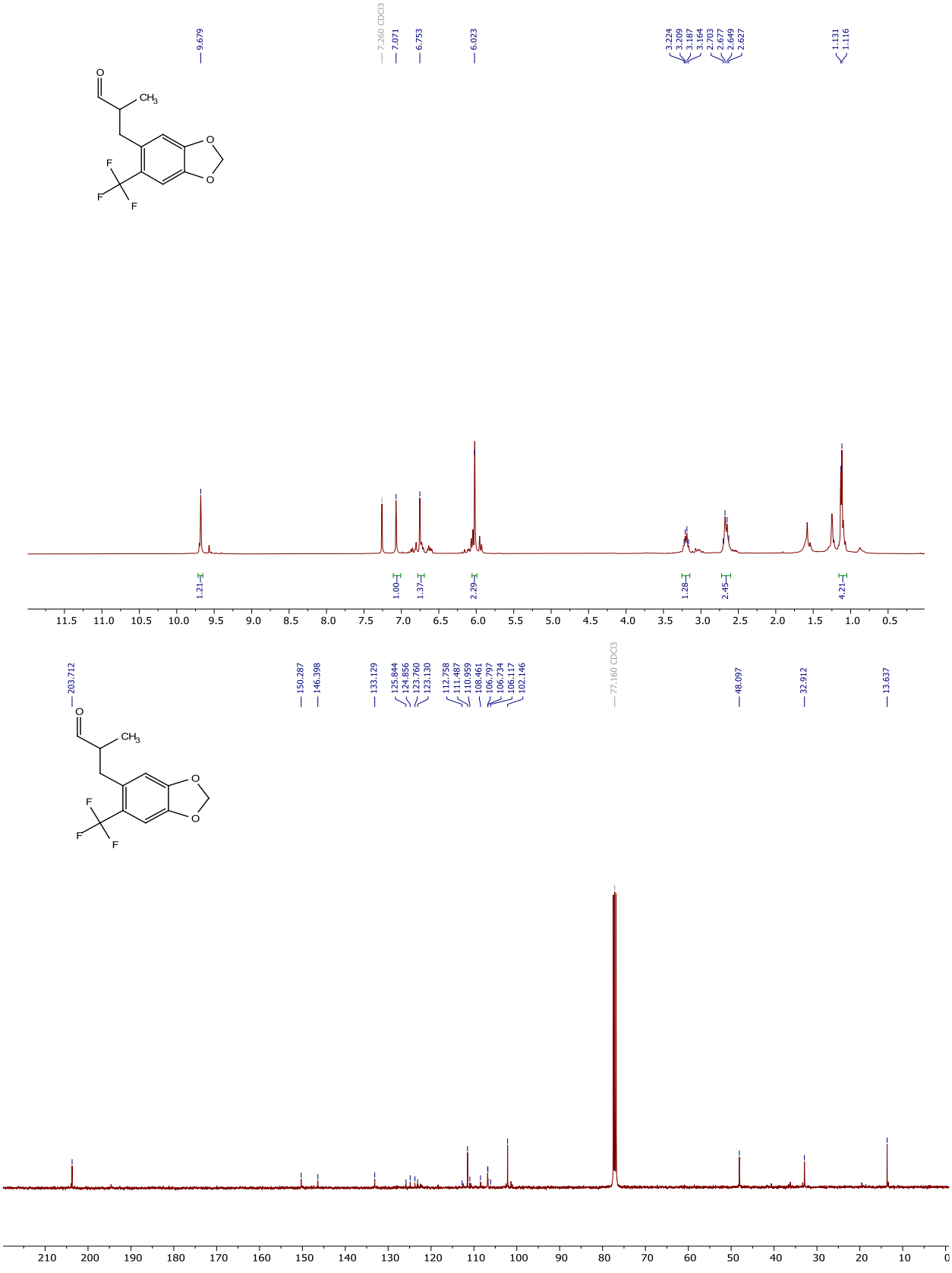
1-Methoxy-2-methyl-5-nitro-3-(trifluoromethyl)benzene (26)

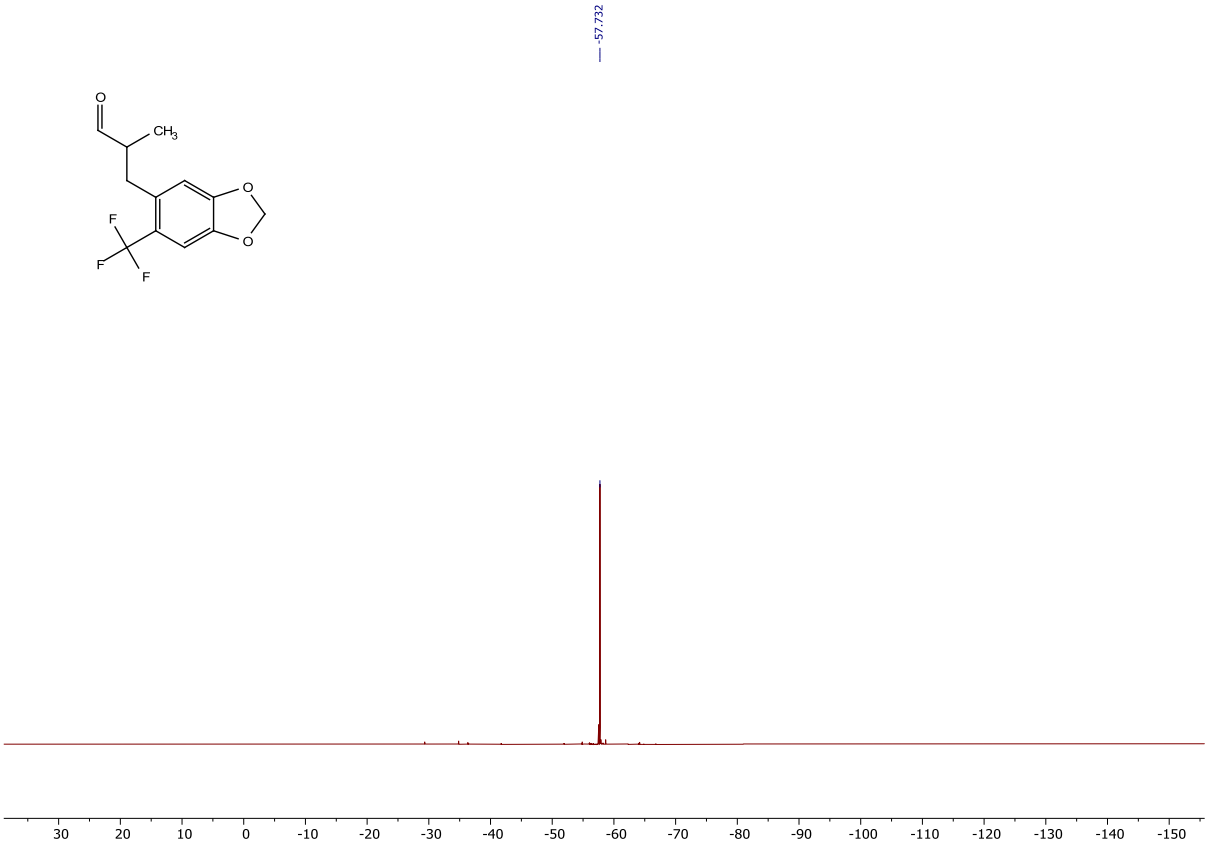



160.262
147.344
132.442
130.604
129.629
129.577
129.523
129.470
126.534
123.833
121.128
118.423
116.068
115.730
115.392
115.054
77.160 CDCl3
56.359
16.437
O
N
O
F
F
F
O
CH3
CH3
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

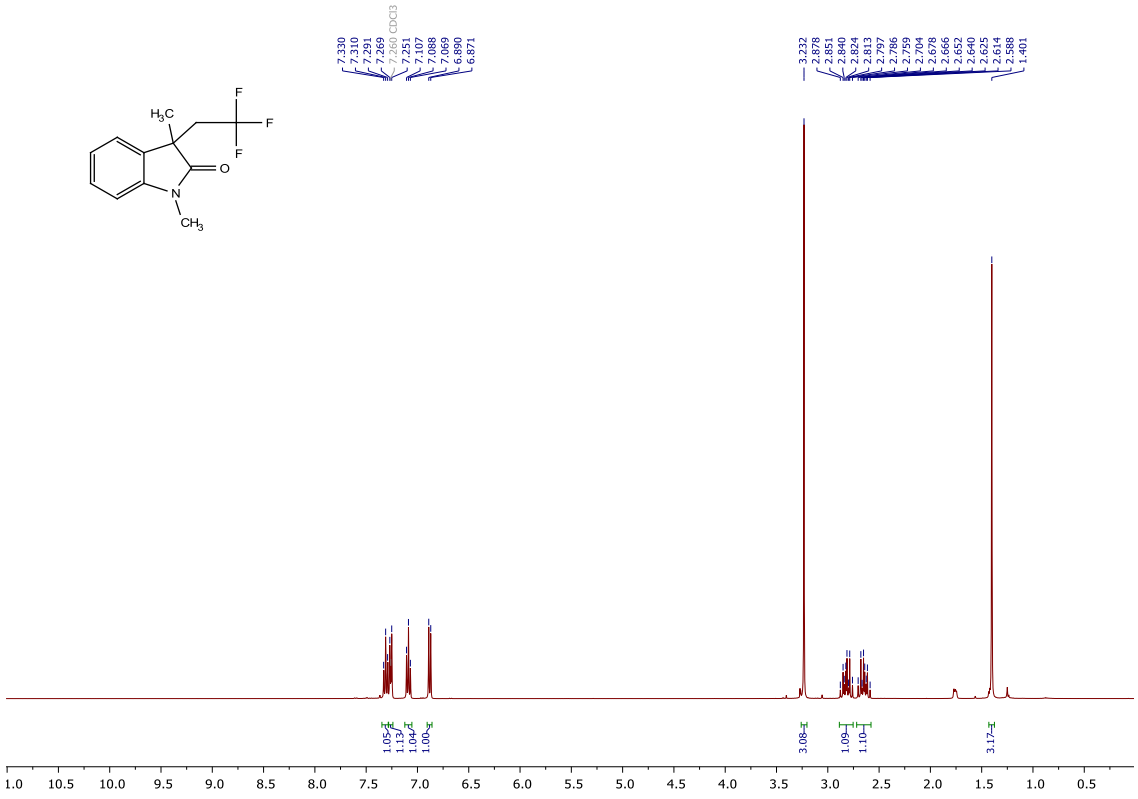

-58.902
O
N
O
F
F
F
O
CH3
CH3
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200

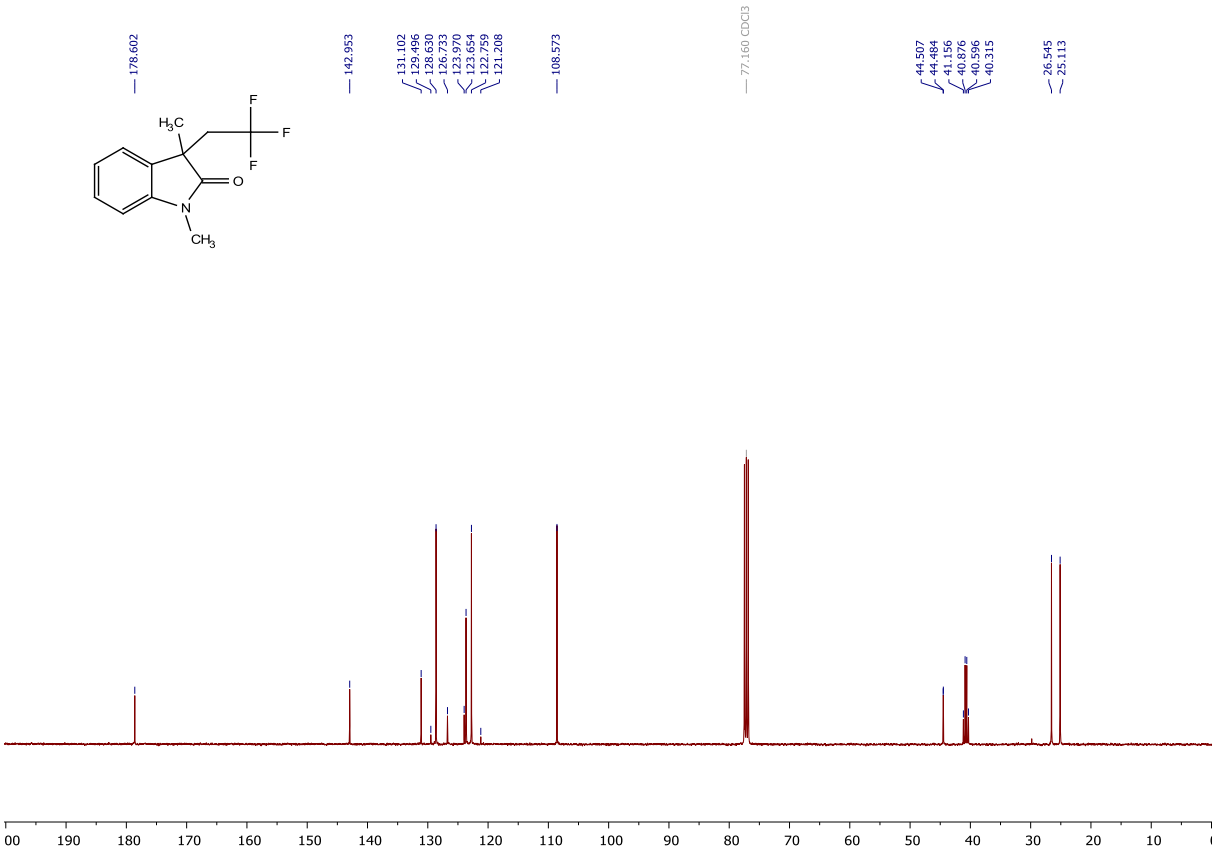
2-Methyl-3-(6-(trifluoromethyl)benzo[d][1,3]dioxol-5-yl)propanal (27)





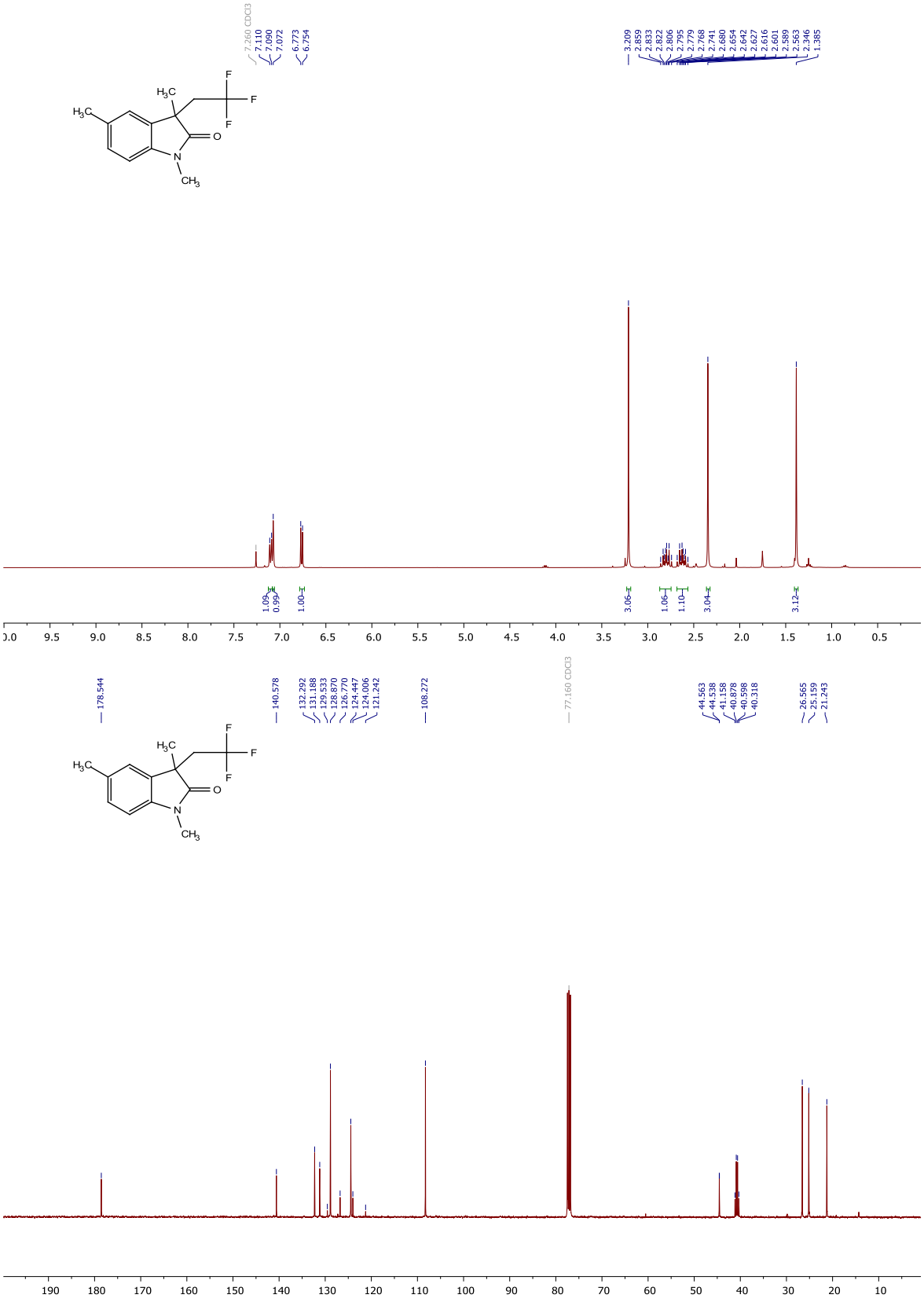
1,3-Dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (28)

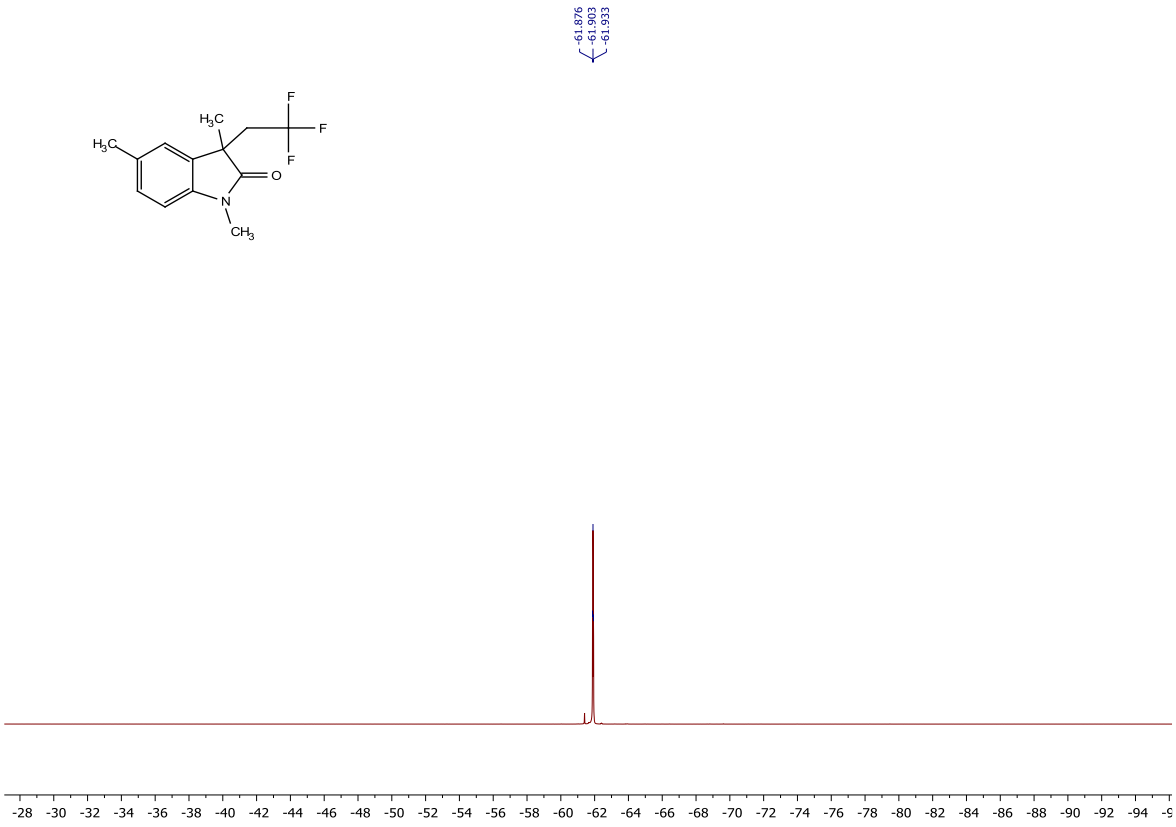



178.602
142.953
131.102
129.496
128.630
126.733
123.970
123.654
122.759
121.208
108.573
77.160 CDCl3
44.507
44.484
41.156
40.876
40.596
40.315
26.545
25.113
F
F
F
H3C
O
N
CH3
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

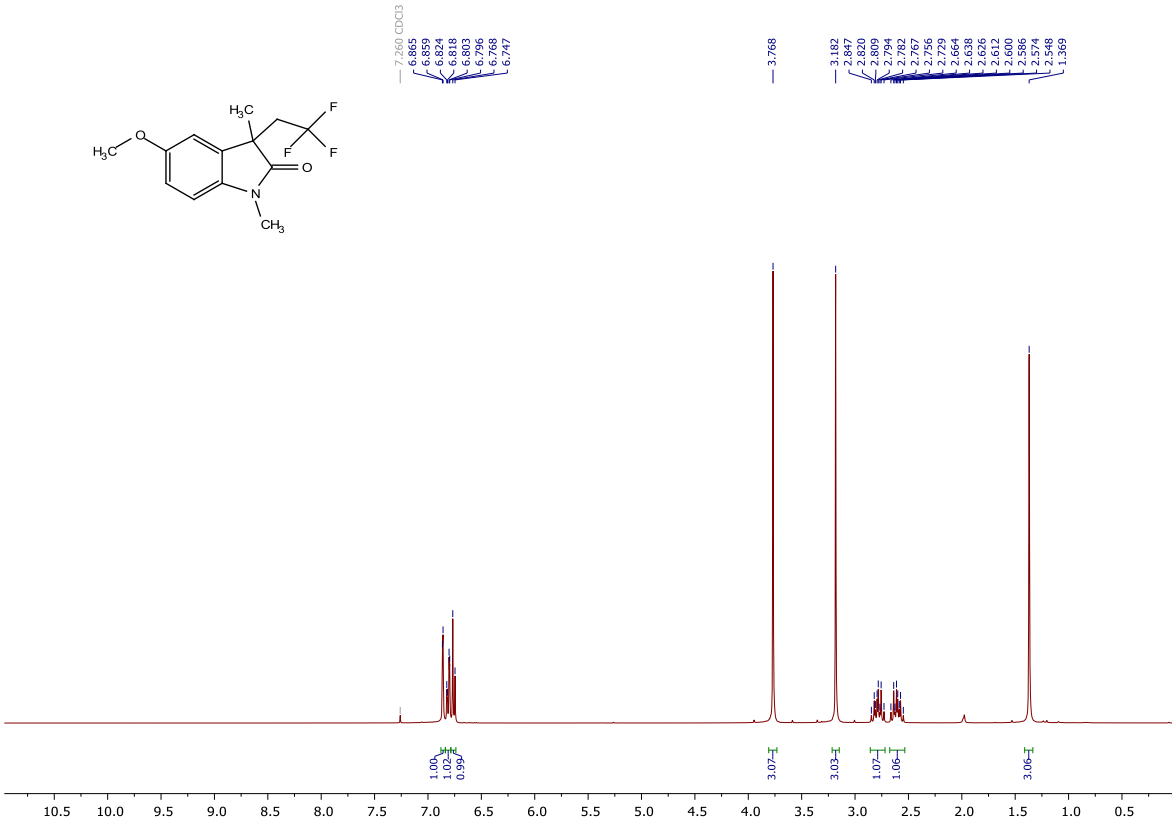

-61.931
-61.958
-61.995
F
F
F
H3C
O
N
CH3
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95

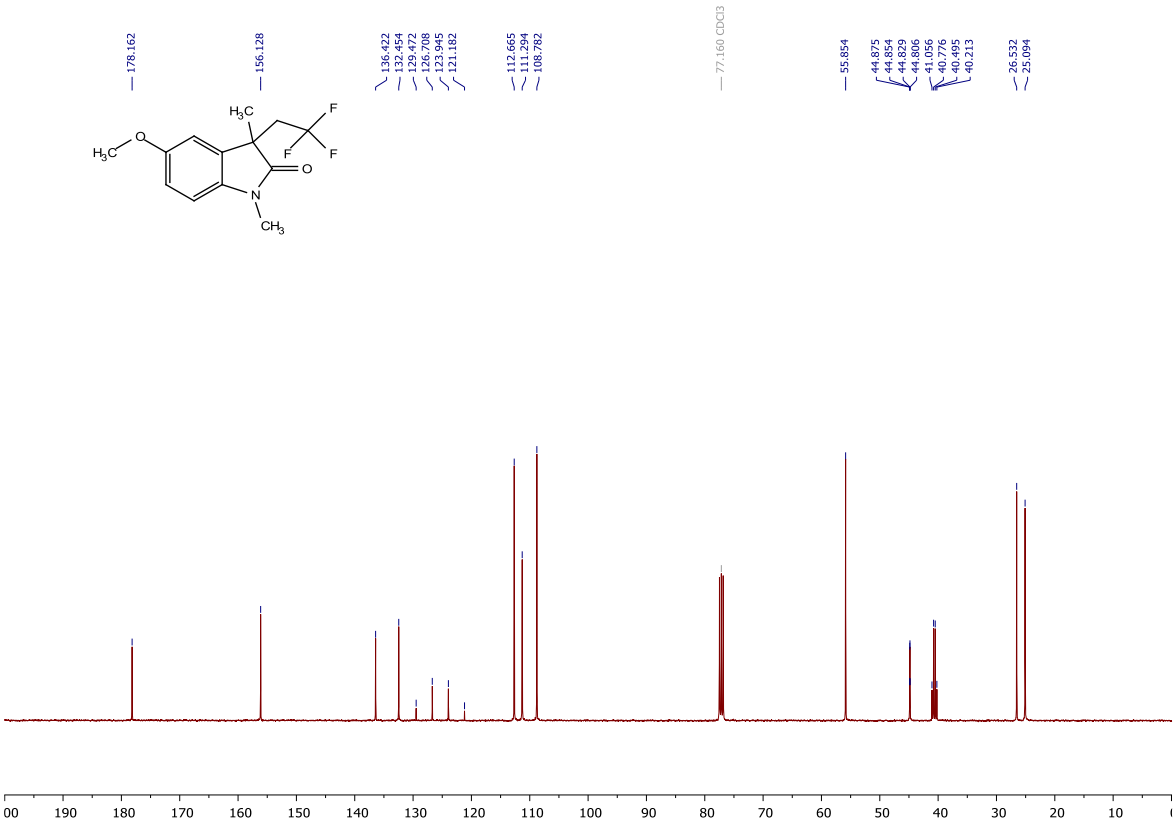
1,3,5-Trimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (29)

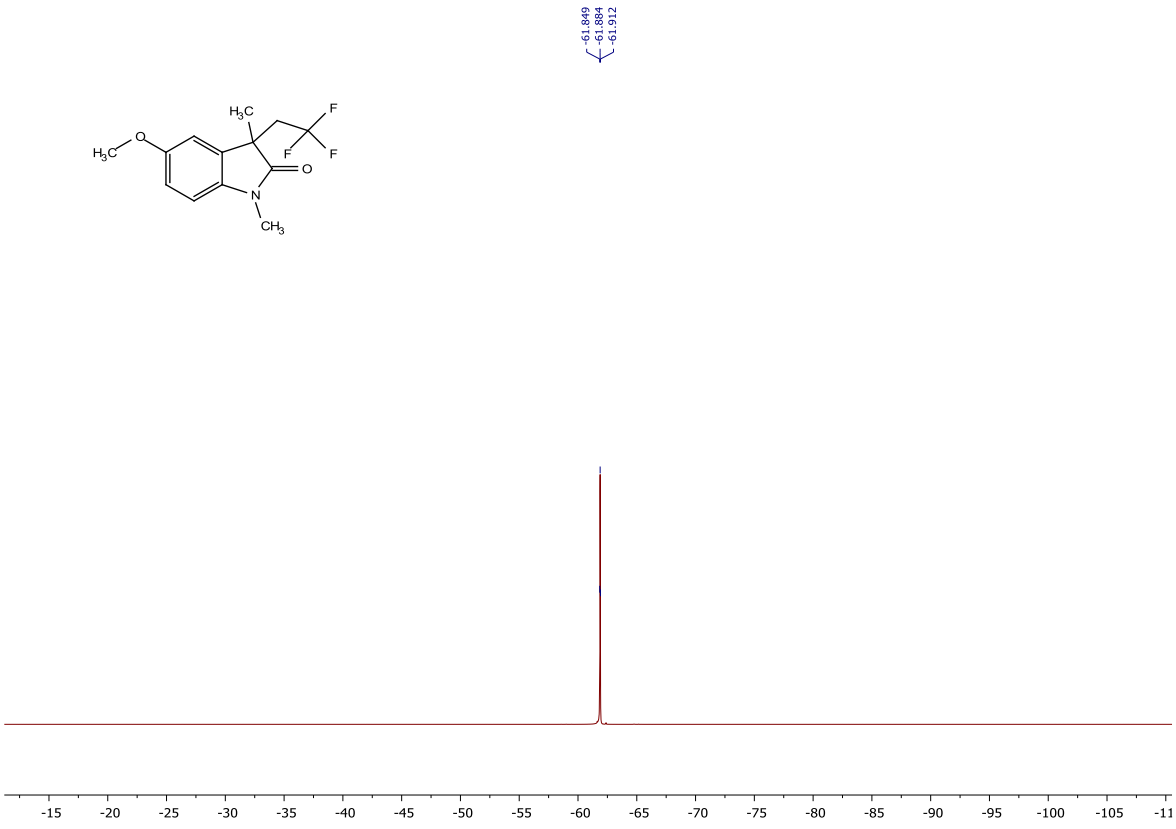




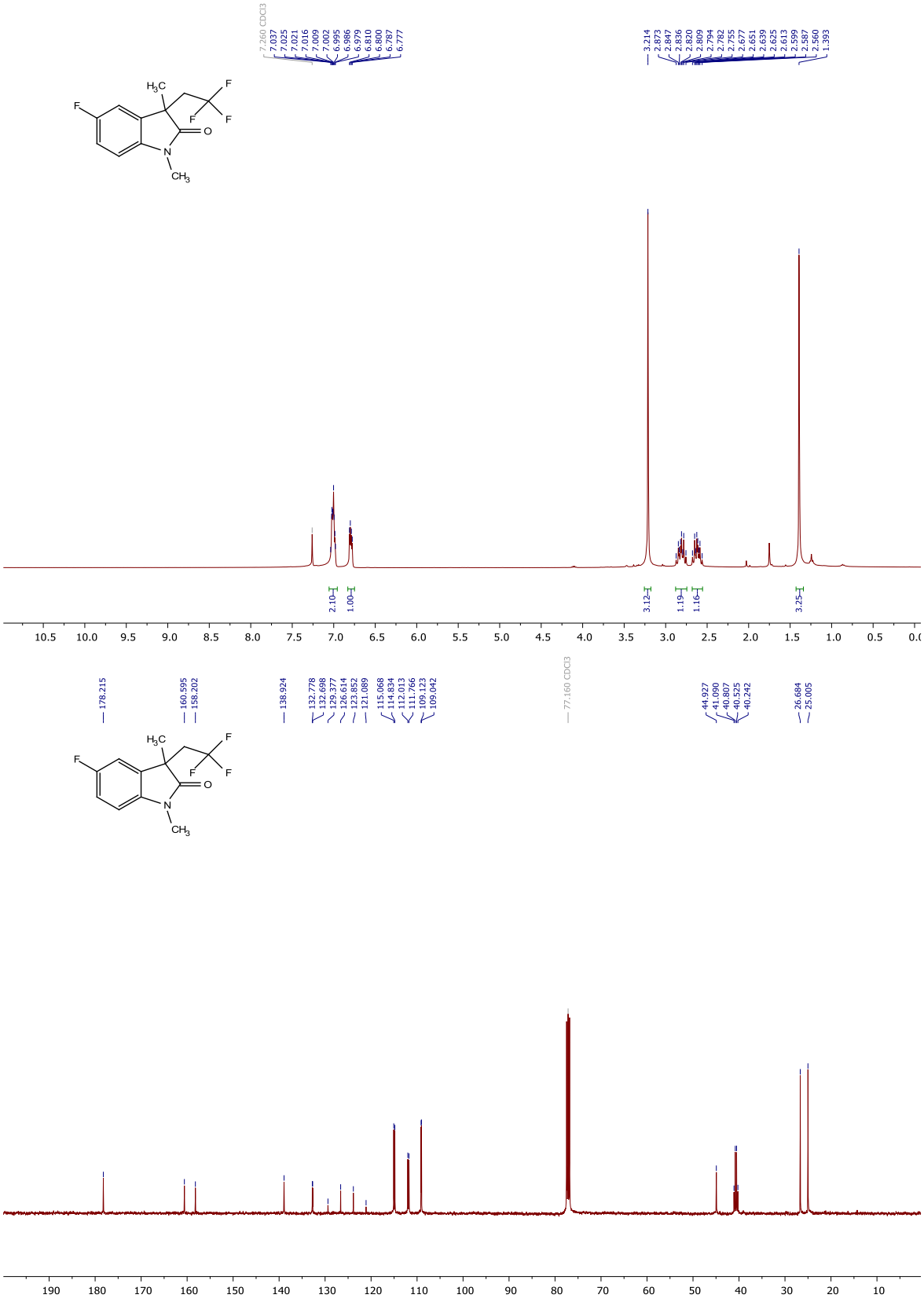
5-Methoxy-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (30)

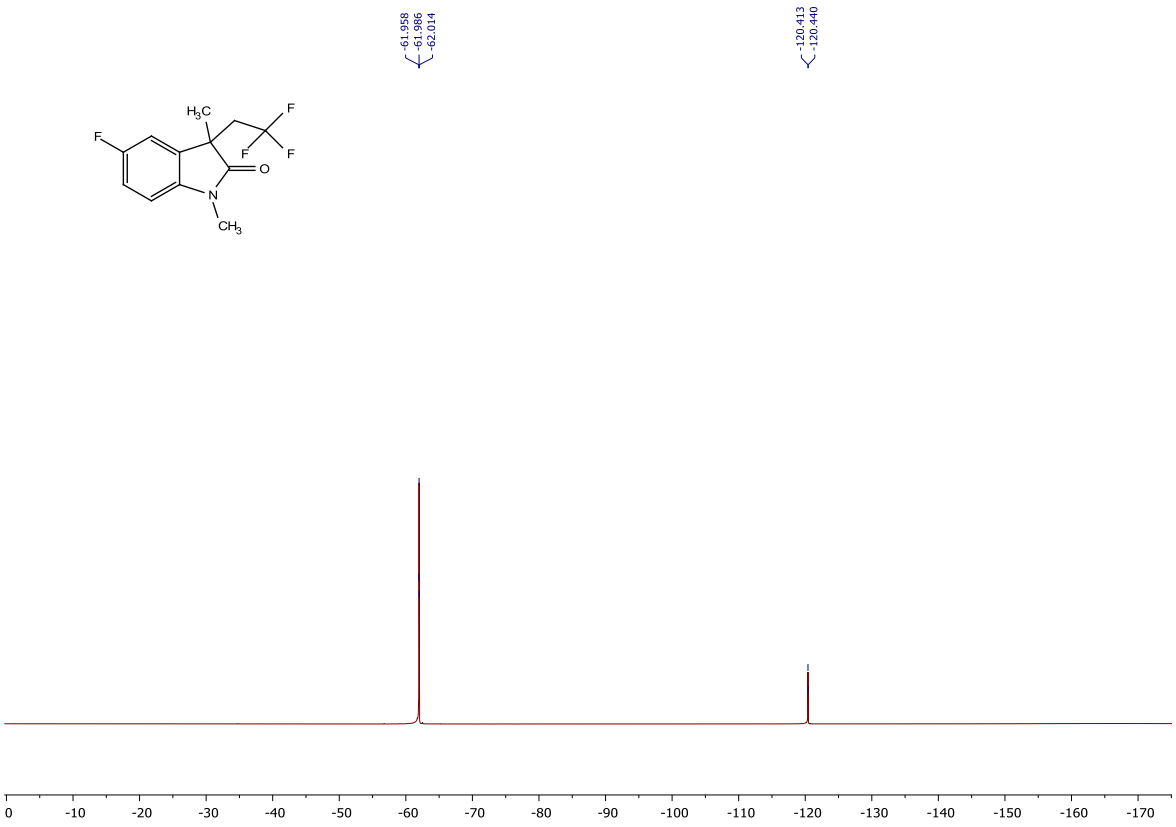



178.162
156.128
136.422
132.454
129.472
126.708
123.945
121.182
112.665
111.294
108.782
77.160 CDCl3
55.854
44.875
44.854
44.829
44.806
41.056
40.776
40.495
40.213
26.532
25.094
H3C
O
H3C
F
F
F
O
N
CH3
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

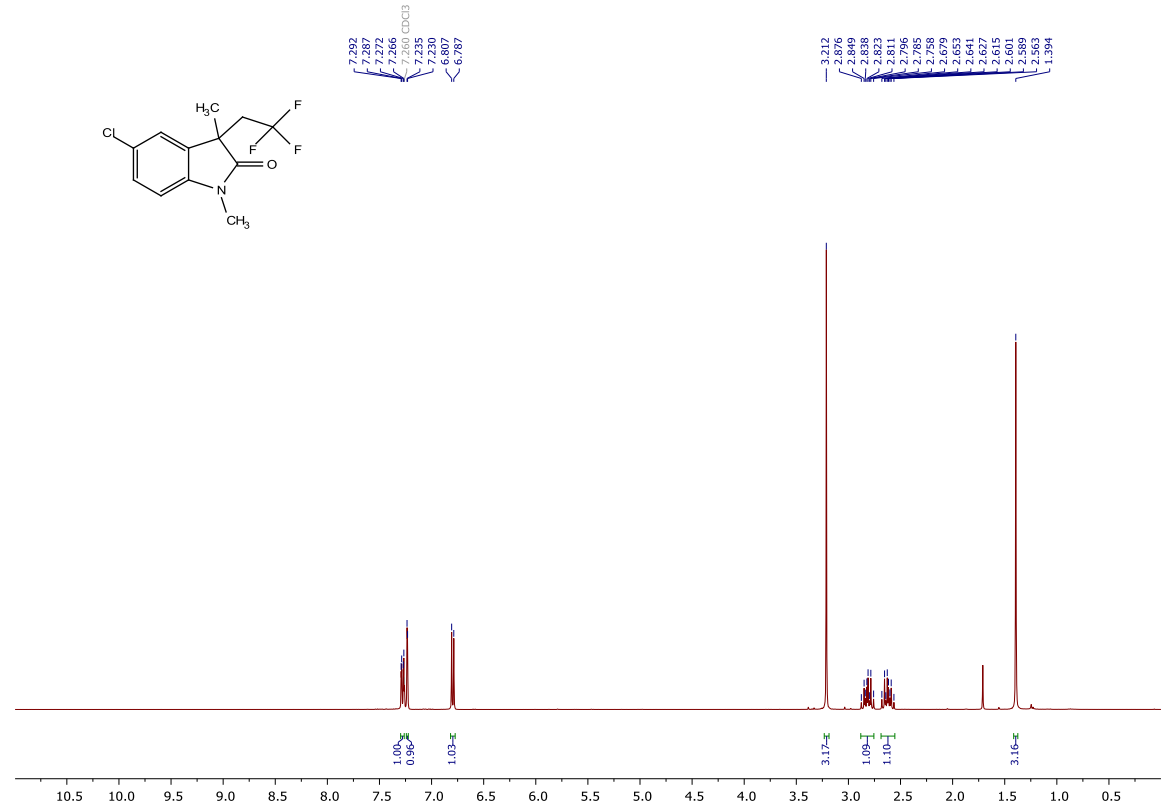

-61.849
-61.884
-61.912
H3C
O
H3C
F
F
F
O
N
CH3
-15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -11

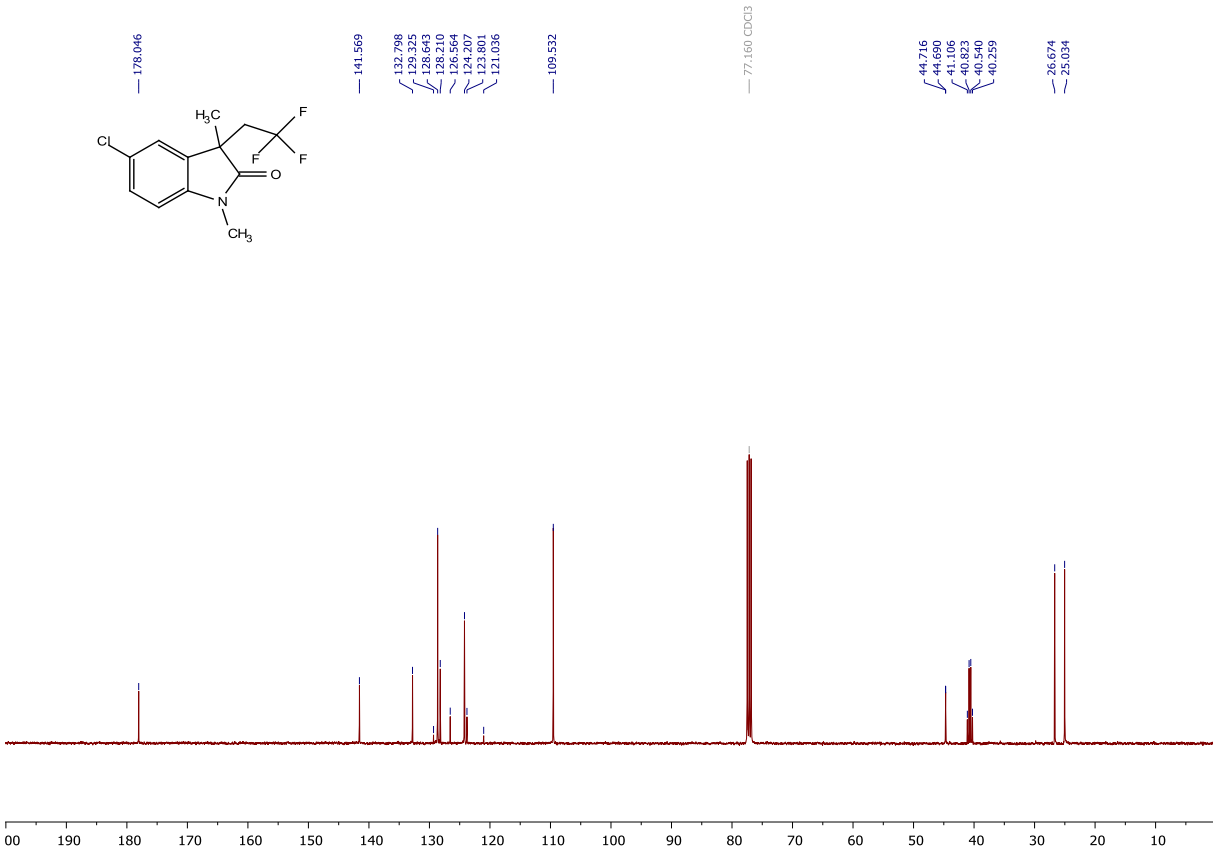
5-Fluoro-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (31)

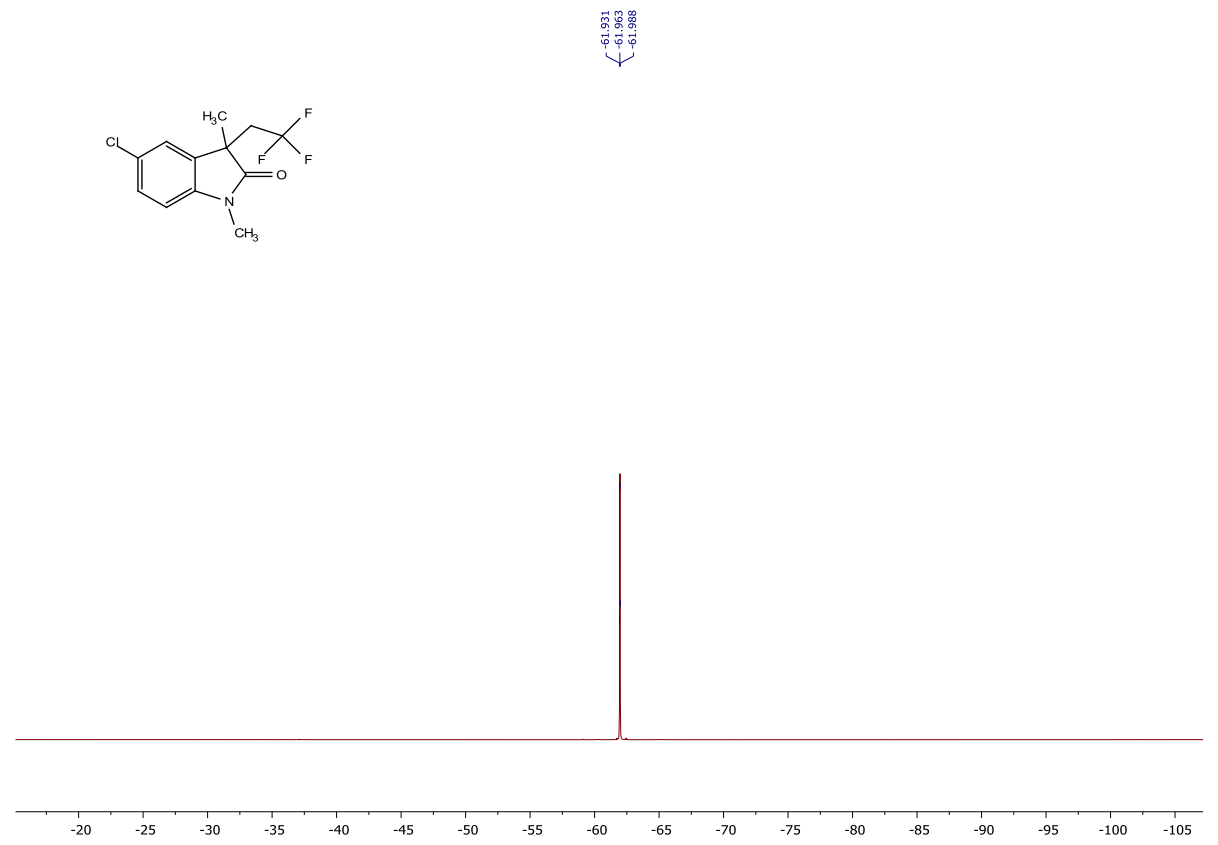




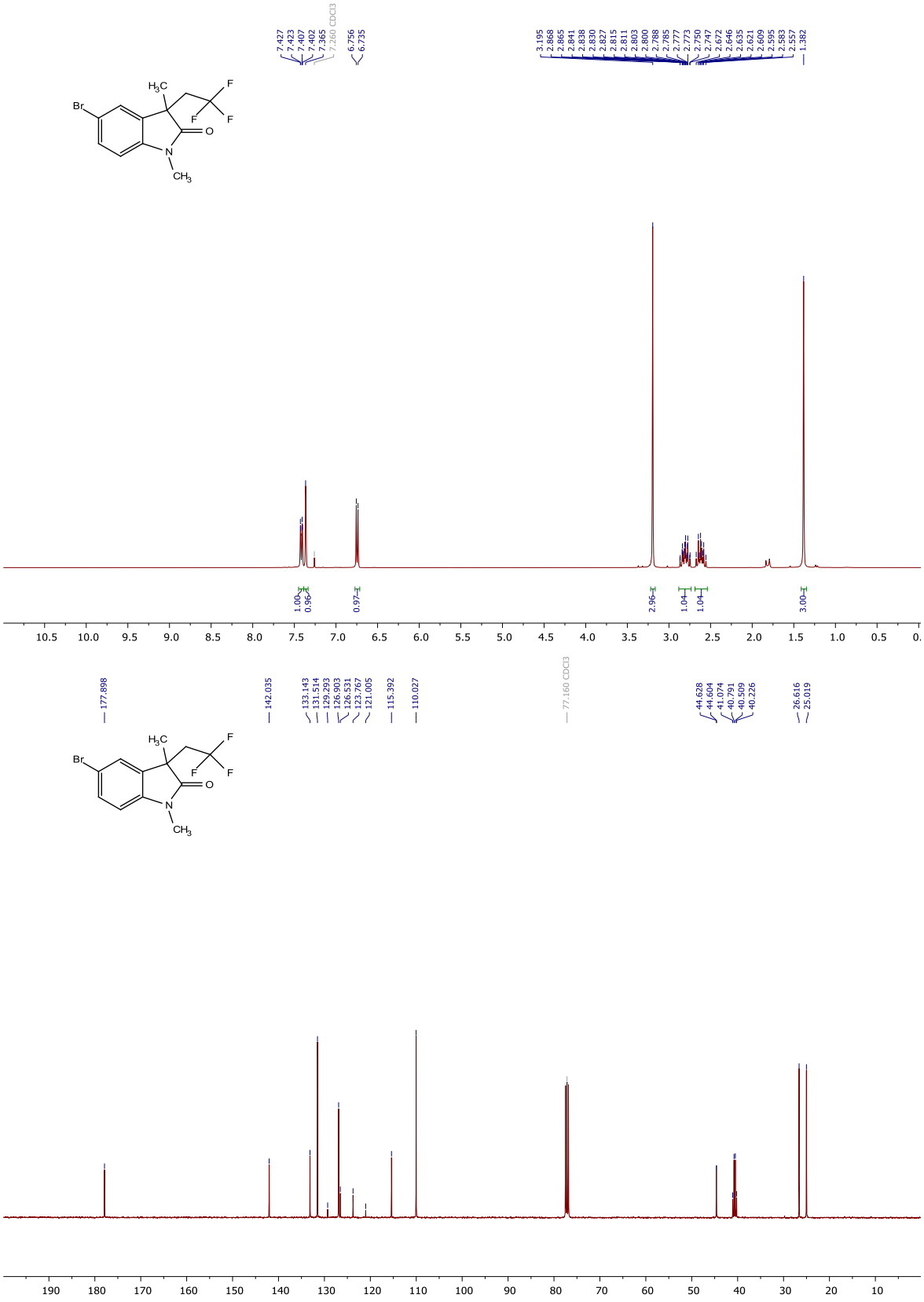
5-Chloro-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (32)

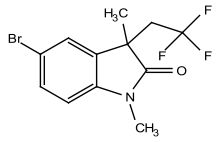



178.046
141.569
132.798
129.325
128.643
128.210
126.564
124.207
123.801
121.036
109.532
77.160 CDCl3
44.716
44.690
41.106
40.823
40.540
40.259
26.674
25.034
Cl
H3C
F
F
F
O
N
CH3
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

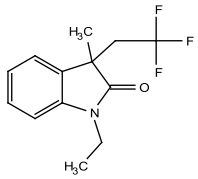

-61.931
-61.963
-61.988
Cl
H3C
F
F
F
O
N
CH3
-20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105

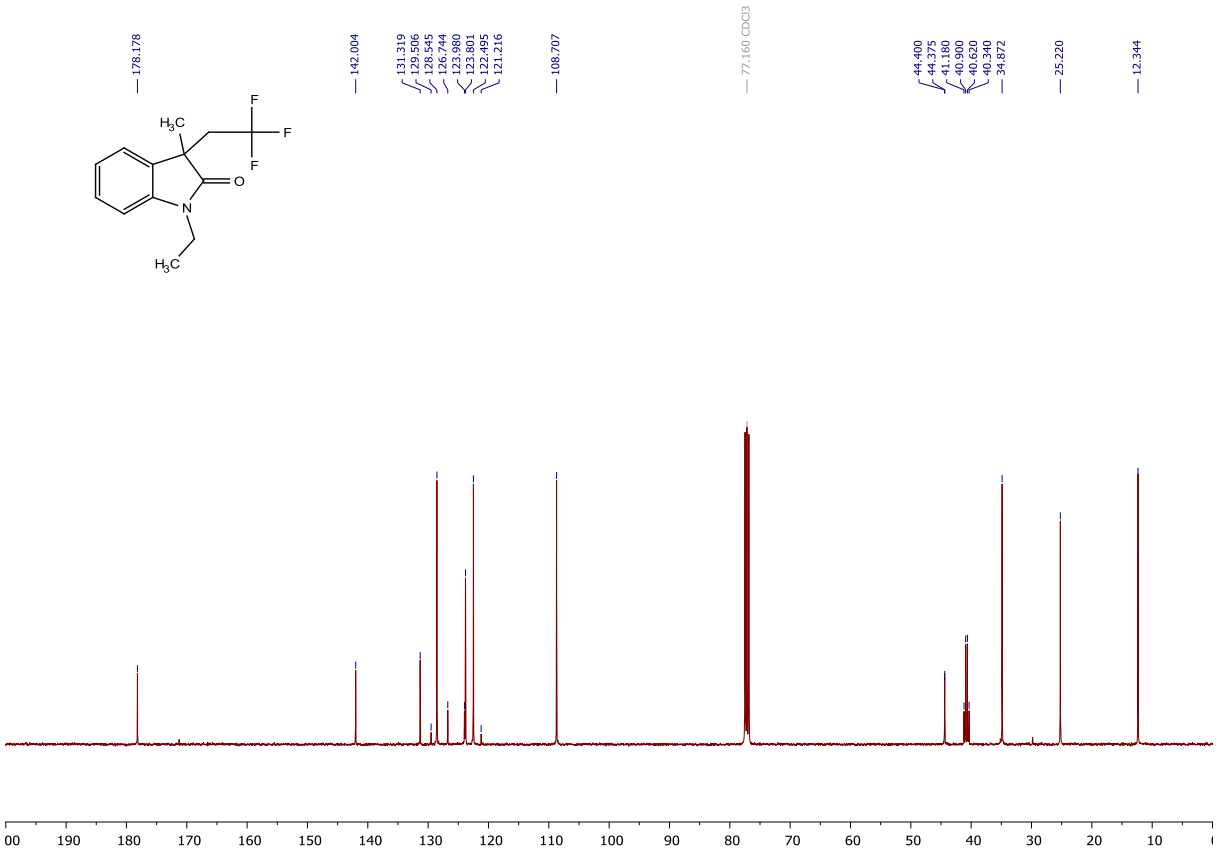
5-Bromo-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (33)





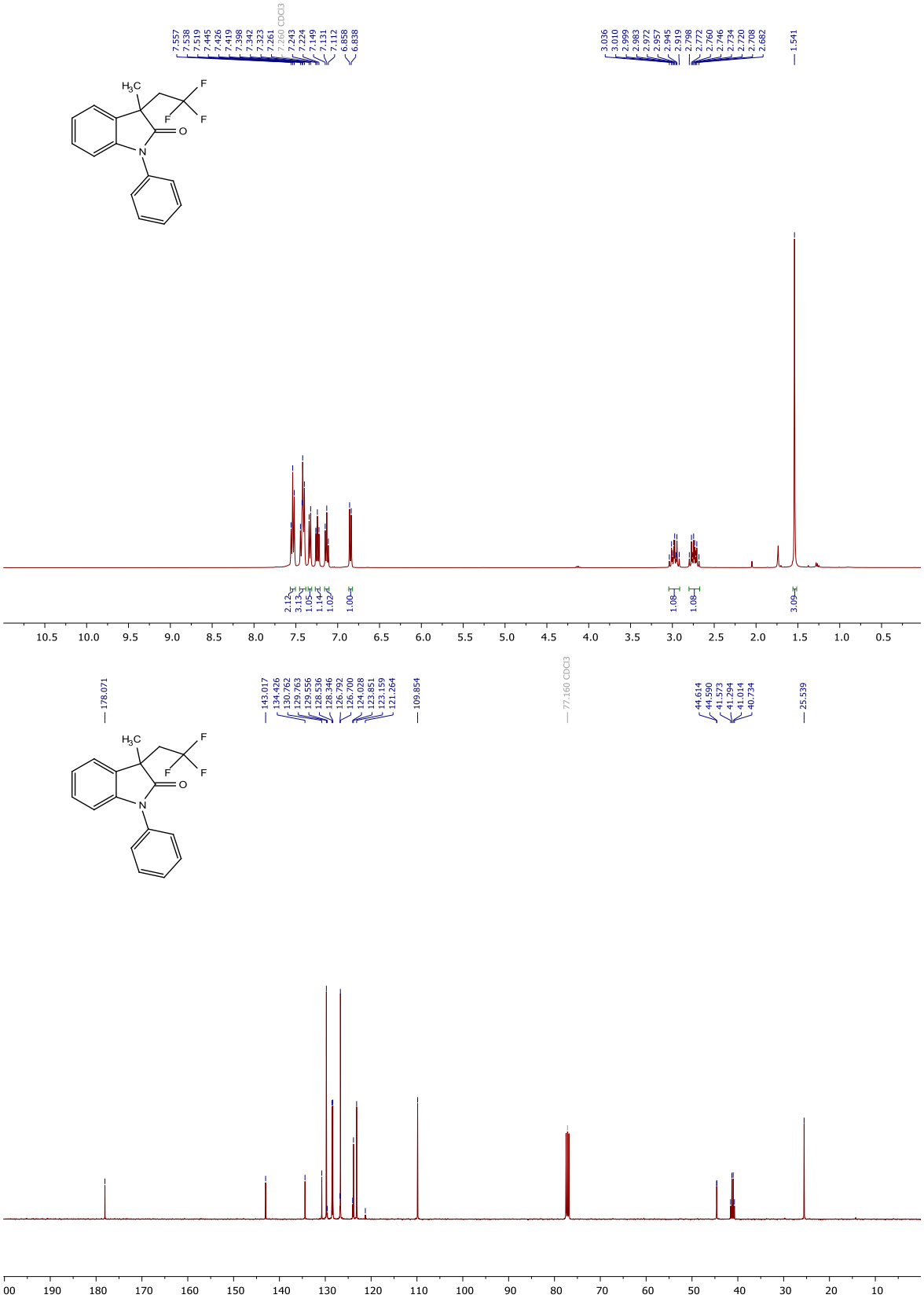
1-Ethyl-3-methyl-3-(2,2,2-trifluoroethyl)indolin-2-one (34)

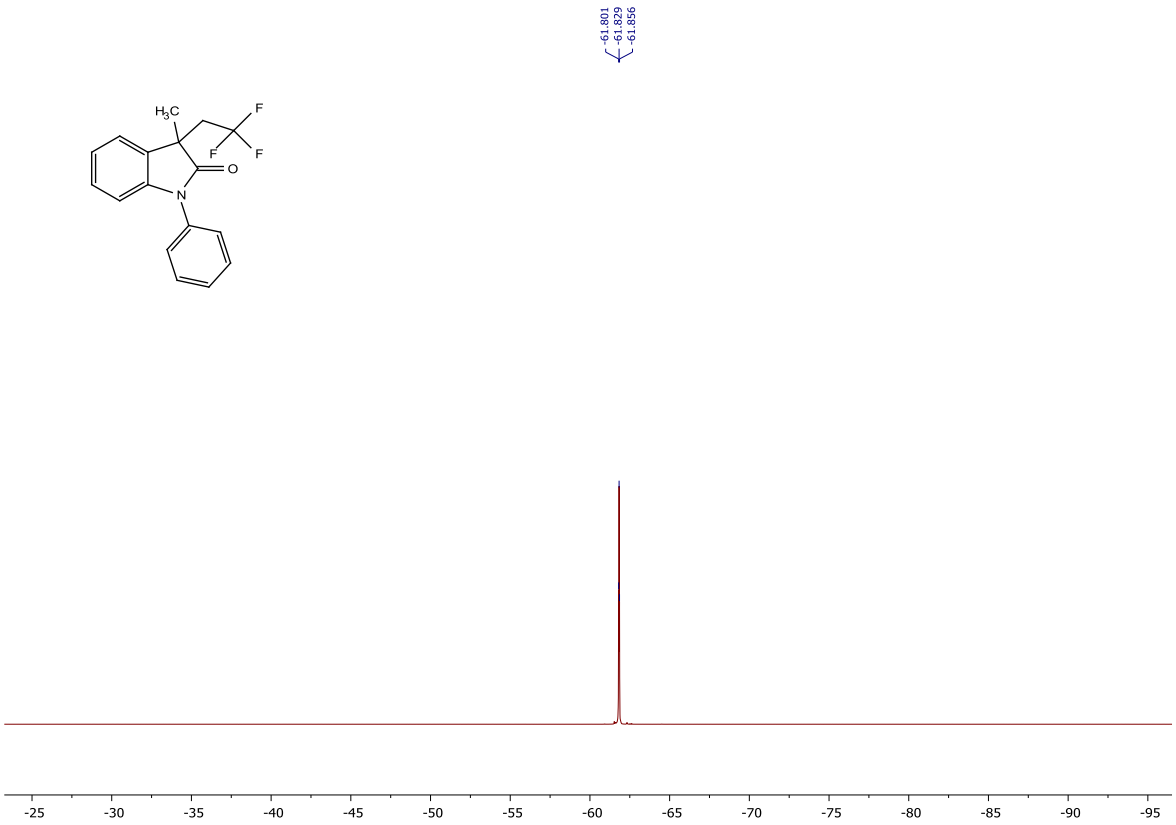



178.178
142.004
131.319
129.506
128.545
126.744
123.980
123.801
122.495
121.216
108.707
77.160 CDCl3
44.400
44.375
41.180
40.900
40.620
40.340
34.872
25.220
12.344
F
F
F
H3C
O
N
H3C
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

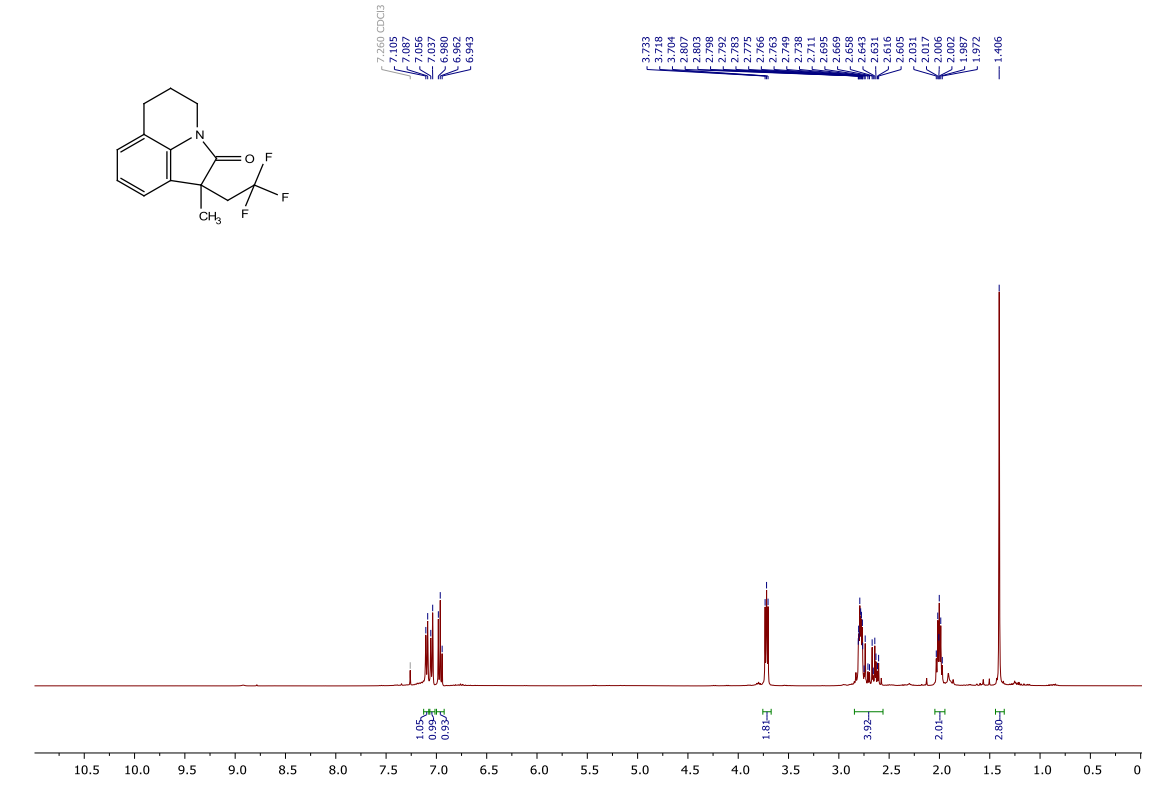

-61.899
-61.925
-61.951
F
F
F
H3C
O
N
H3C
-20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105

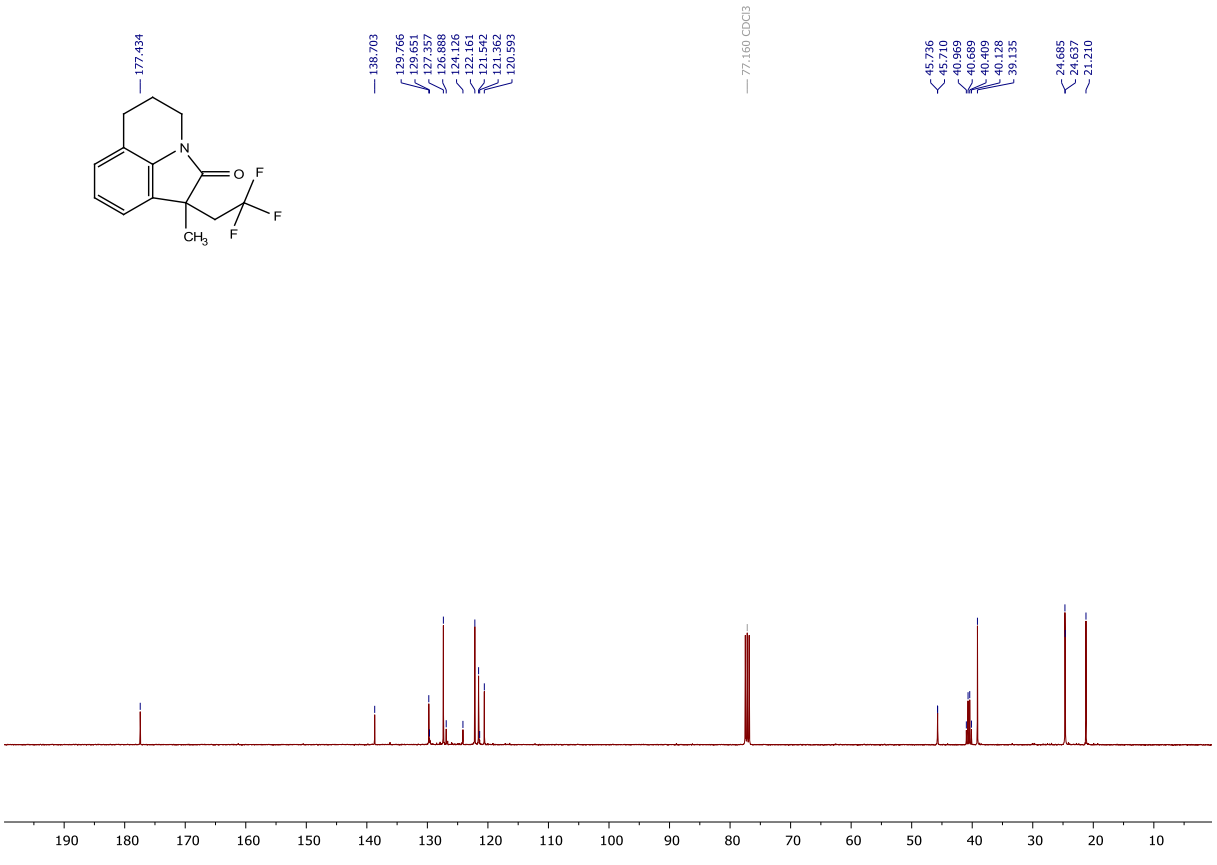
3-Methyl-1-phenyl-3-(2,2,2-trifluoroethyl)indolin-2-one (35)

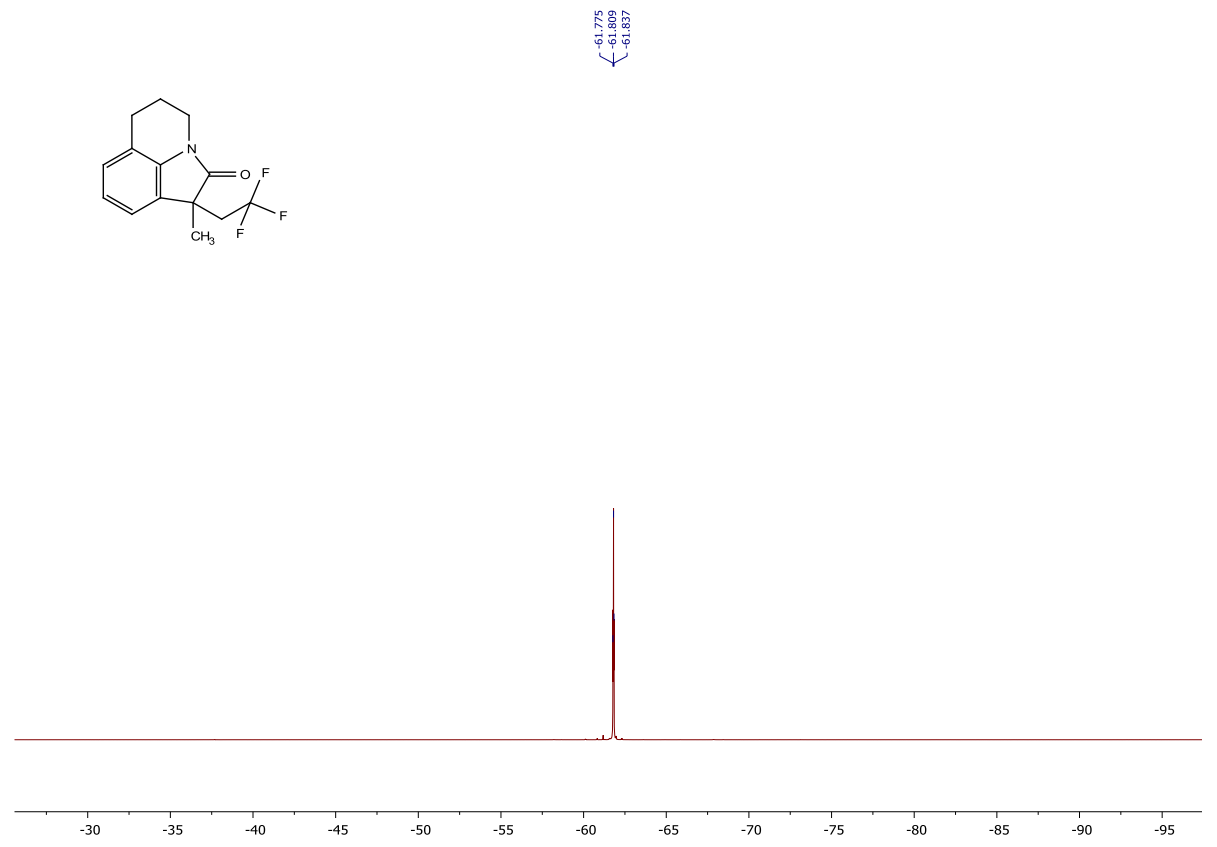




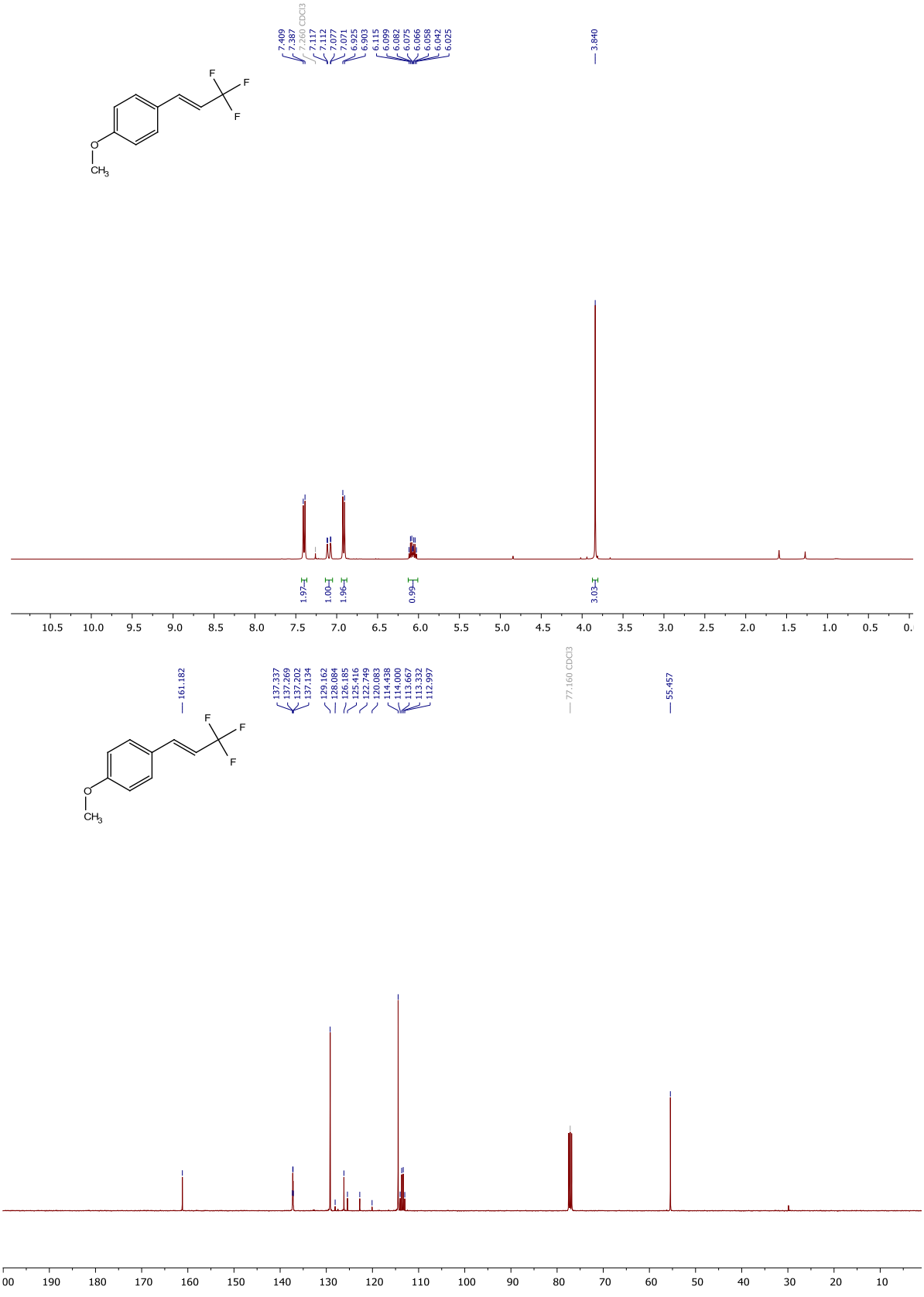
1-Methyl-1-(2,2,2-trifluoroethyl)-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinolin-2(1*H*)-one (36)

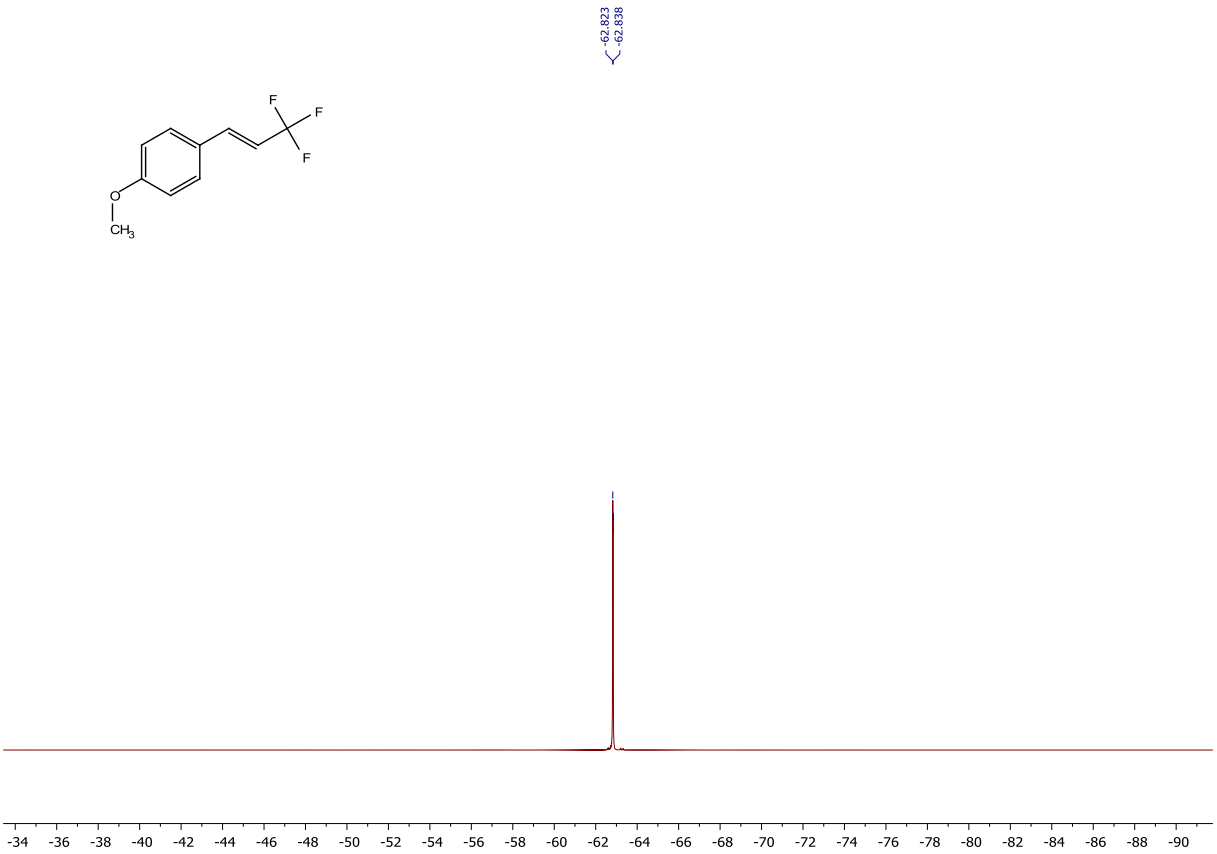



177.434
138.703
129.766
129.651
127.357
126.888
124.126
122.161
121.542
121.362
120.593
77.160 CDCl3
45.736
45.710
40.969
40.689
40.409
40.128
39.135
24.685
24.637
21.210
O
F
F
F
N
CH3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

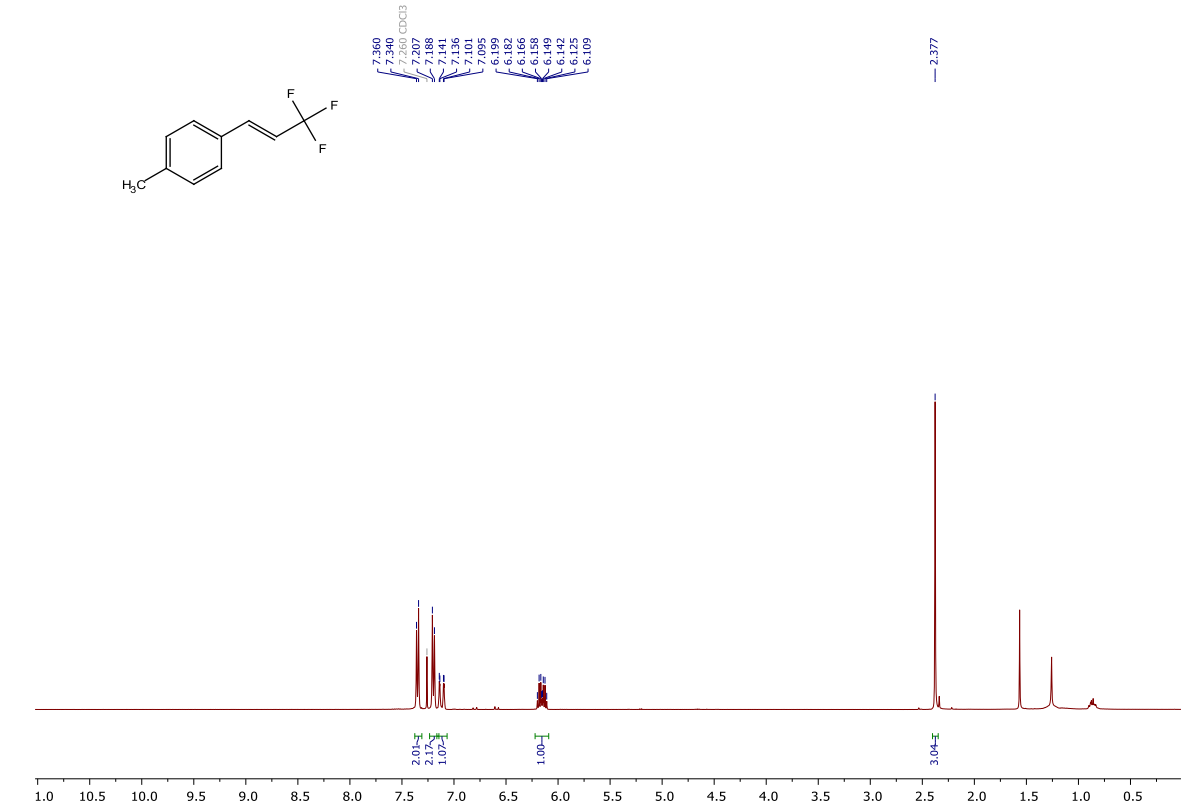

-61.775
-61.809
-61.837
O
F
F
F
N
CH3
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95

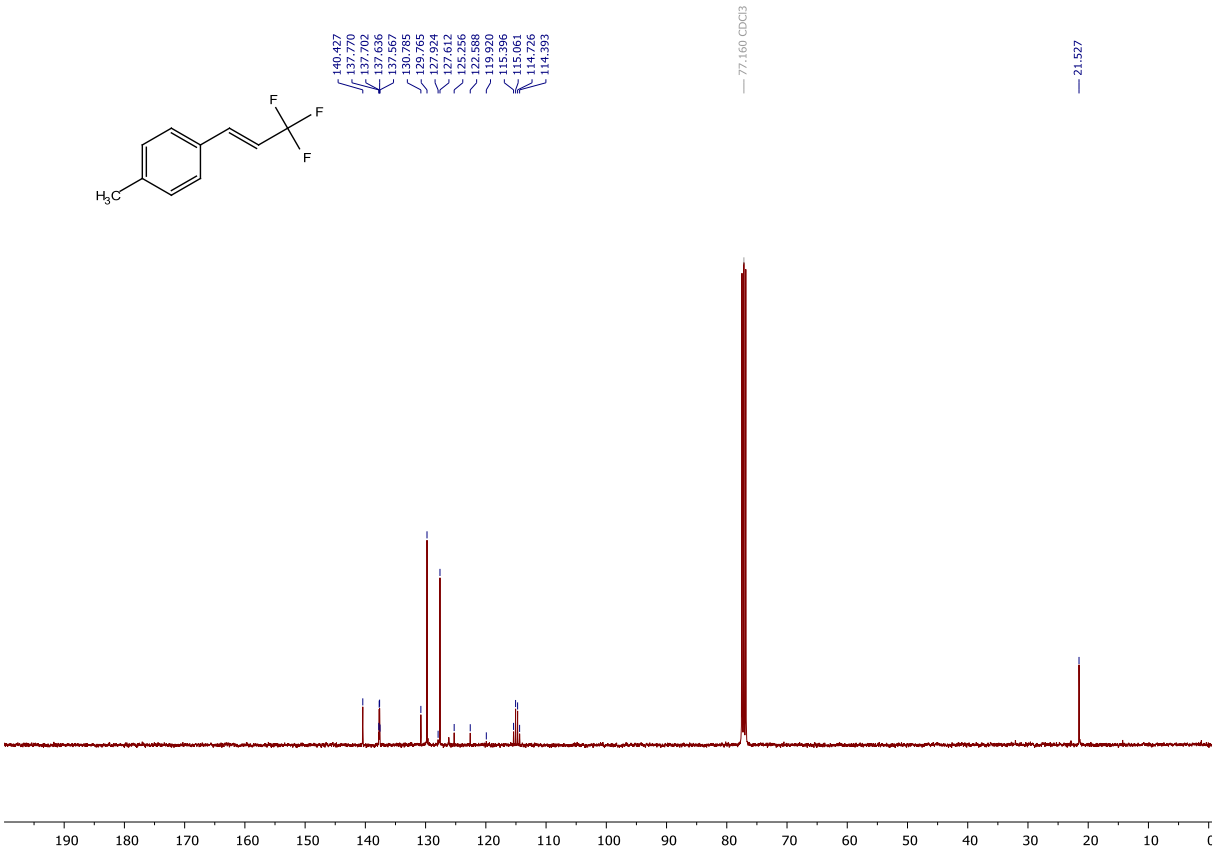
(*E*)-1-Methoxy-4-(3,3,3-trifluoroprop-1-en-1-yl)benzene (37)

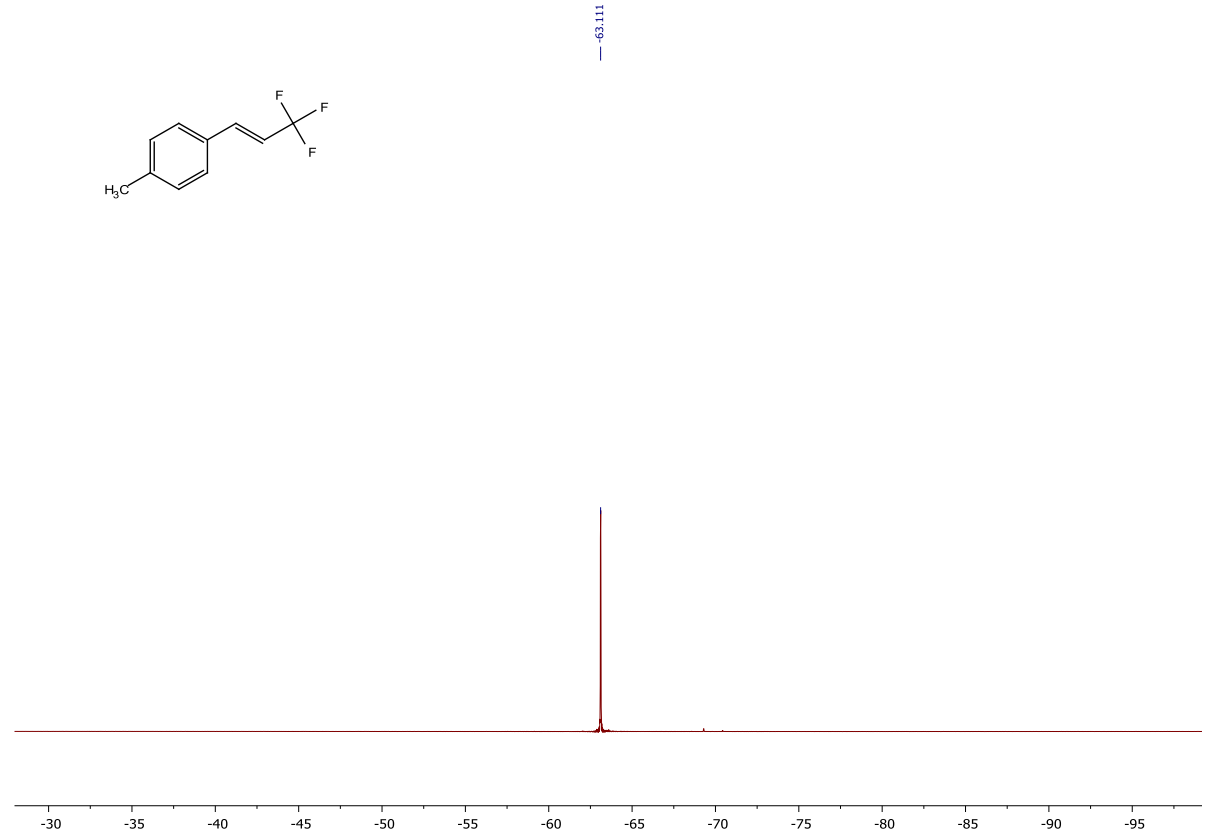




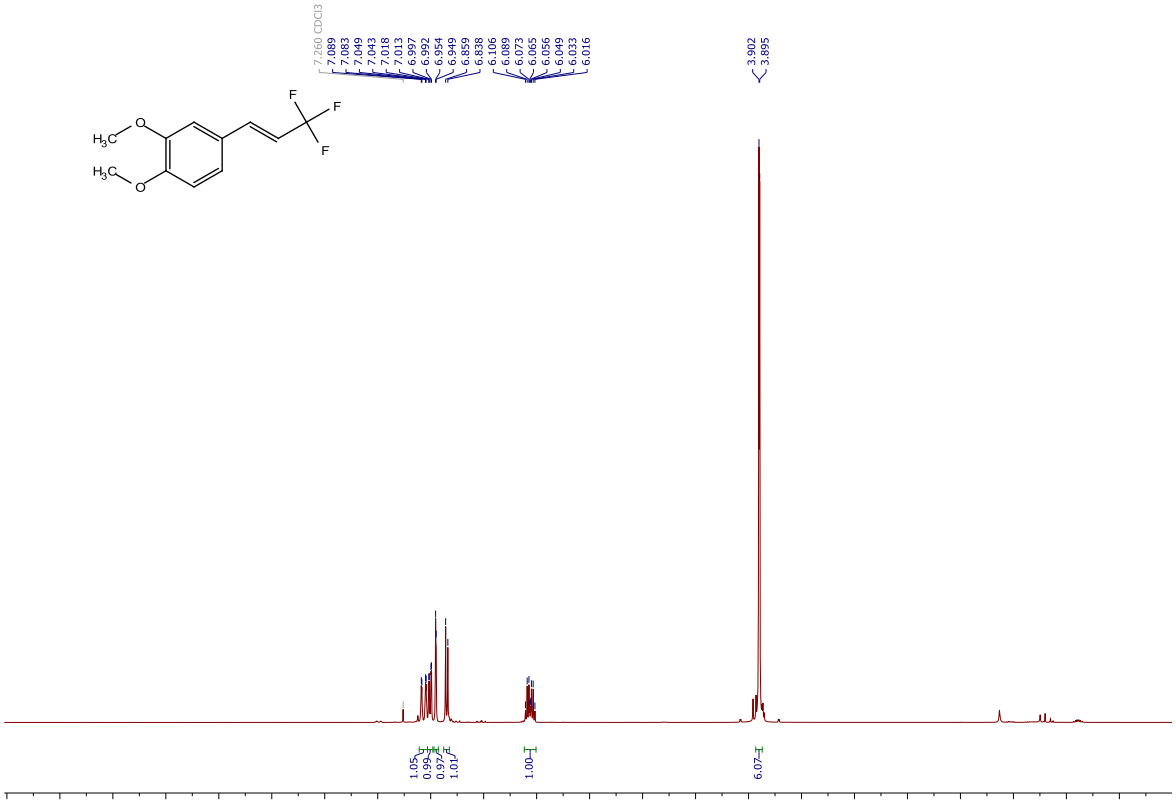
(*E*)-1-Methyl-4-(3,3,3-trifluoroprop-1-en-1-yl)benzene (38)

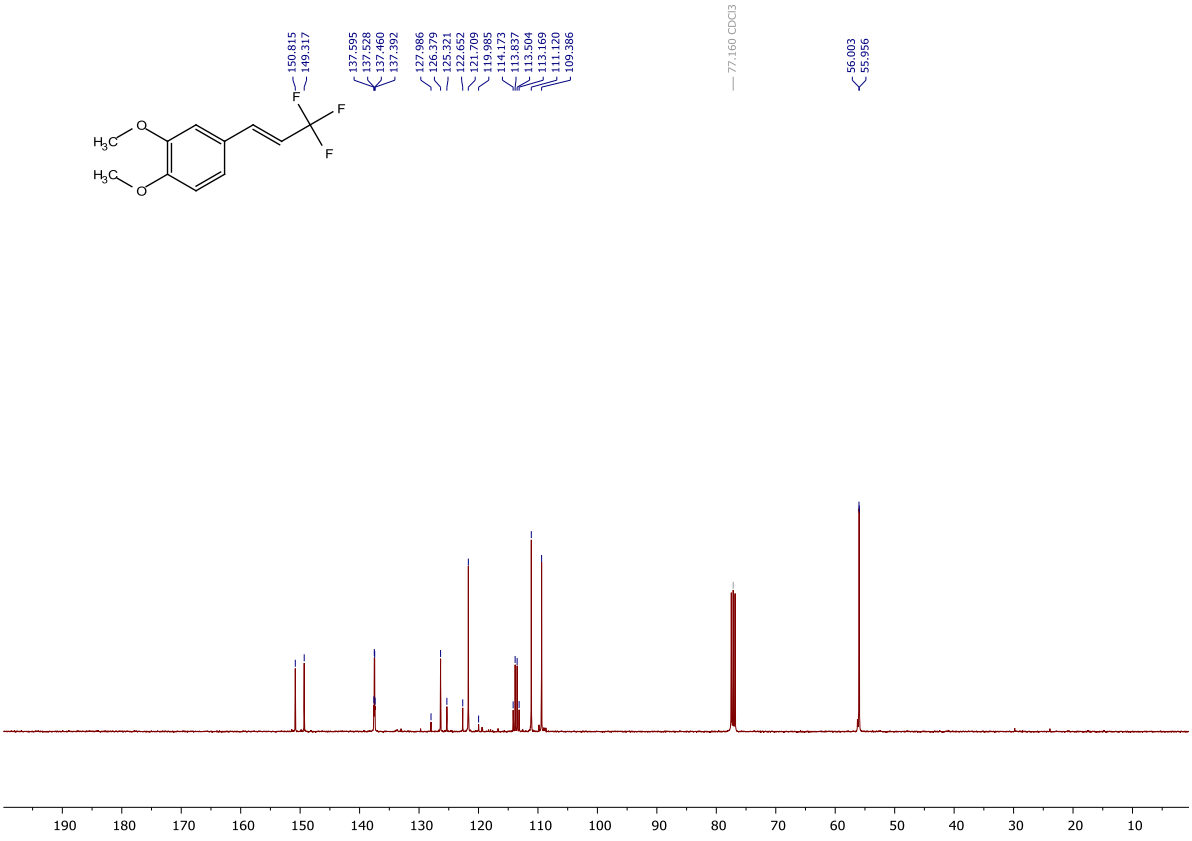


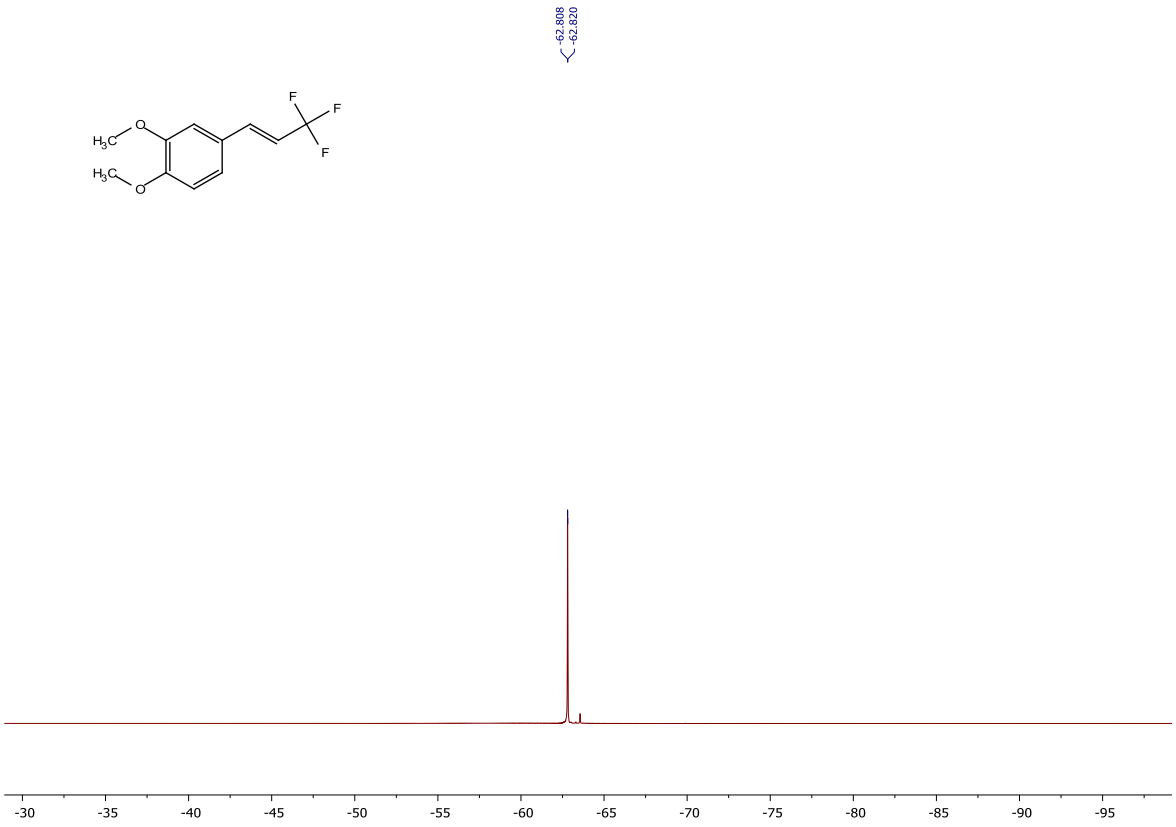

140.427
137.770
137.702
137.636
137.567
130.785
129.765
127.924
127.612
125.256
122.588
119.920
115.396
115.061
114.726
114.393
77.160 CDCl3
21.527
F
F
F
H3C
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


-63.111
F
F
F
H3C
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95

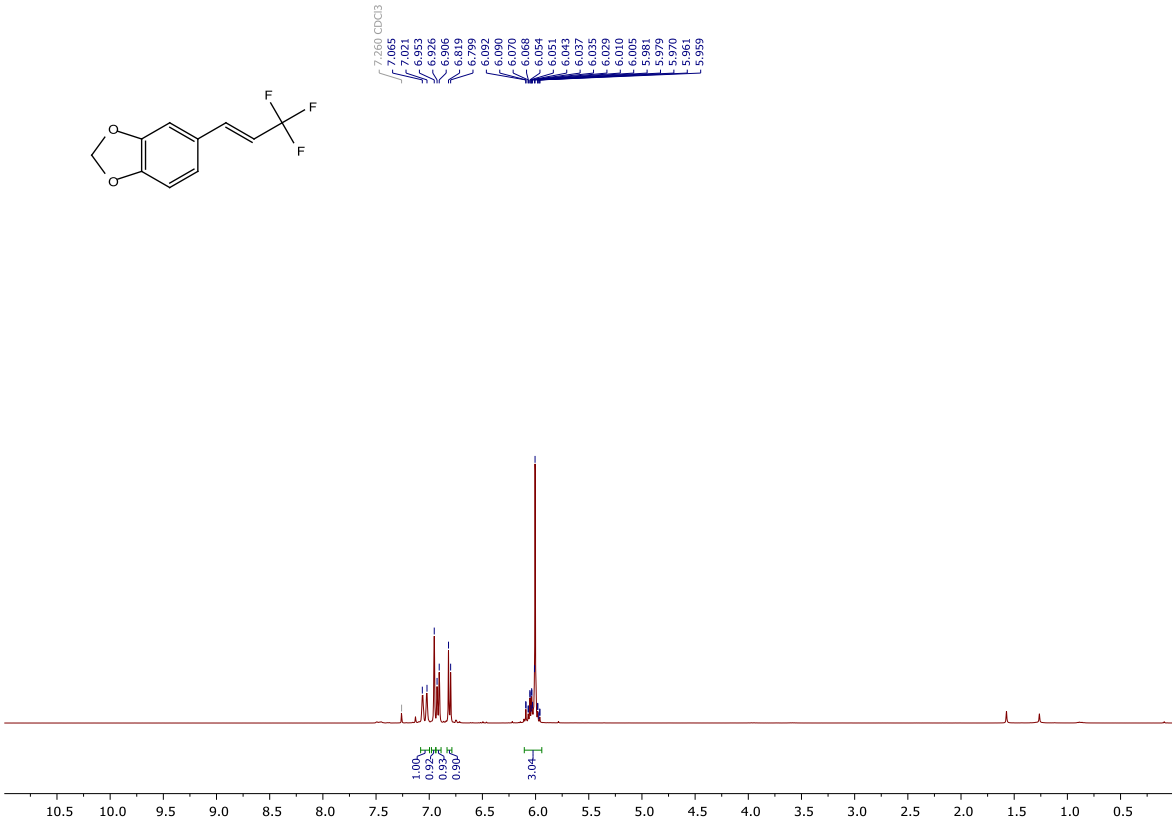
(*E*)-1,2-Dimethoxy-4-(3,3,3-trifluoroprop-1-en-1-yl)benzene (39)

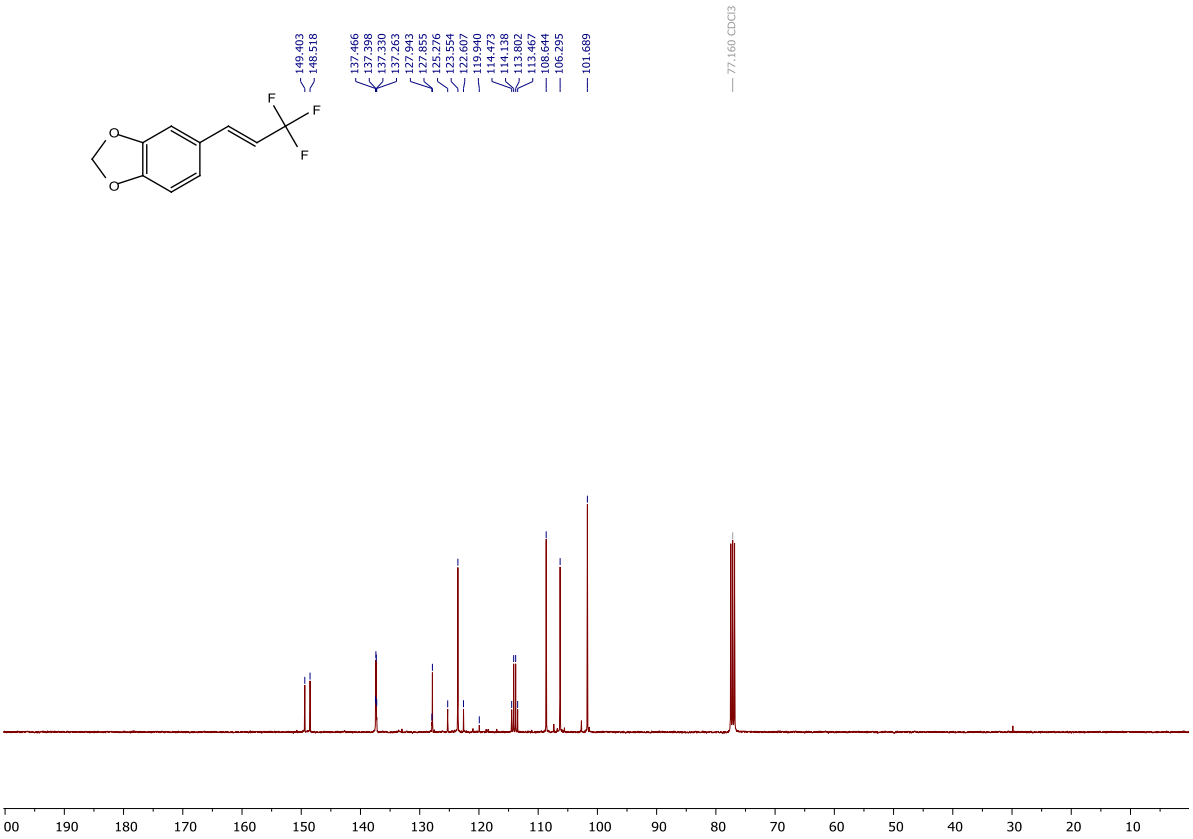


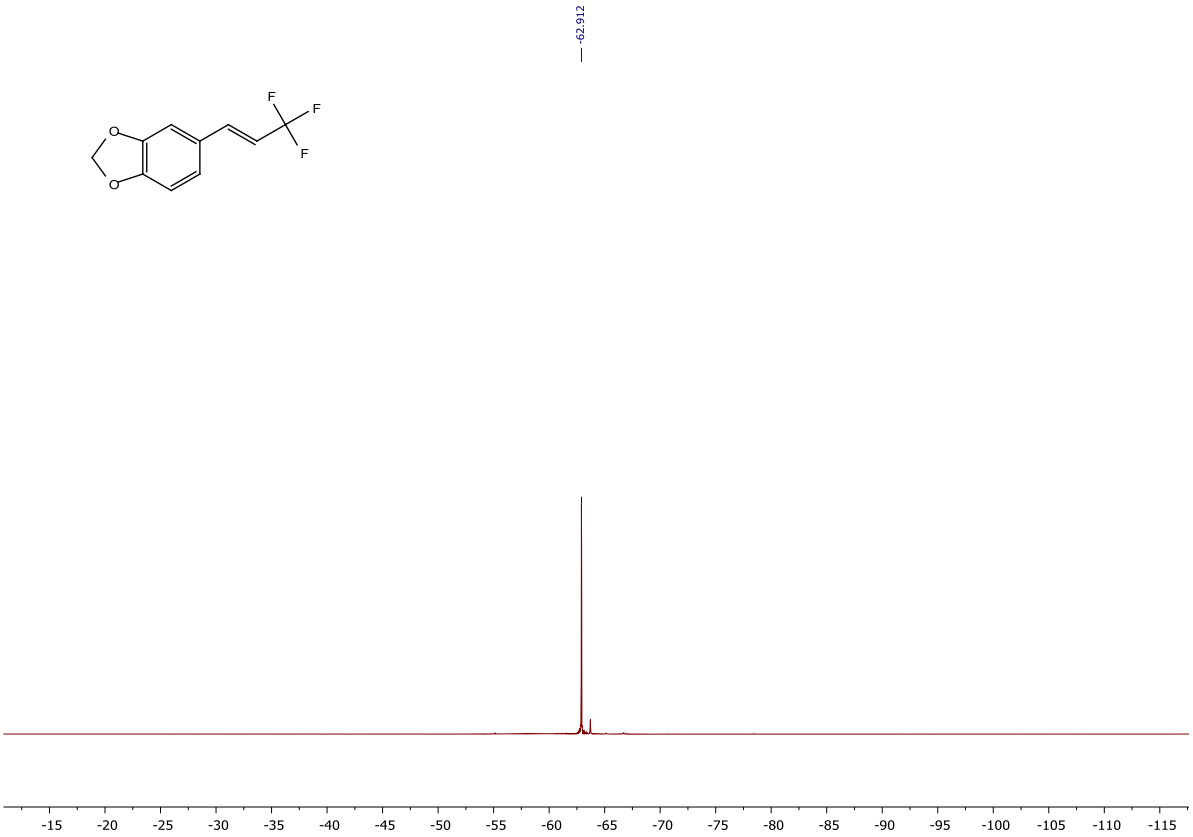




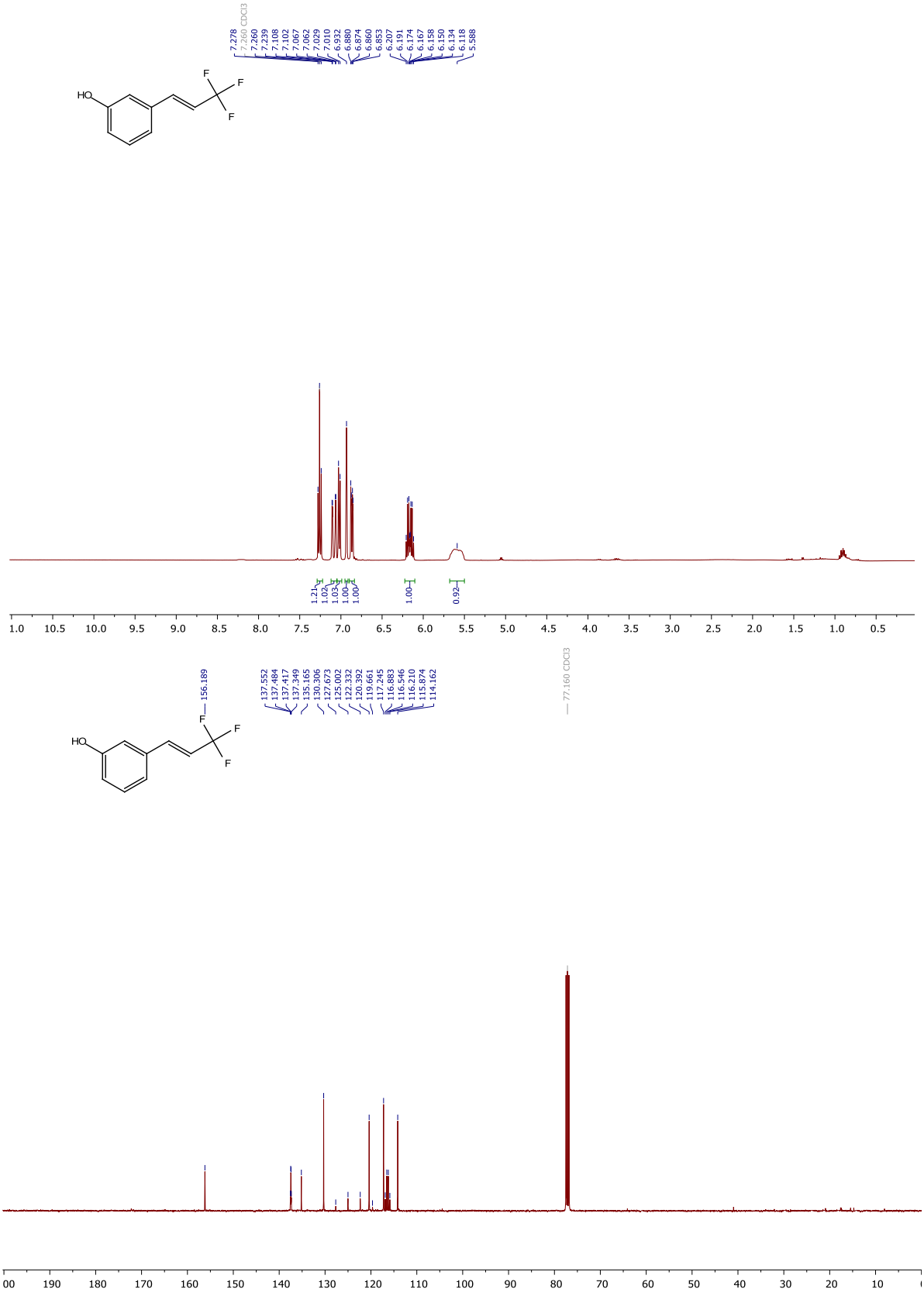
(*E*)-5-(3,3,3-Trifluoroprop-1-en-1-yl)benzo[*d*][1,3]dioxole (40)

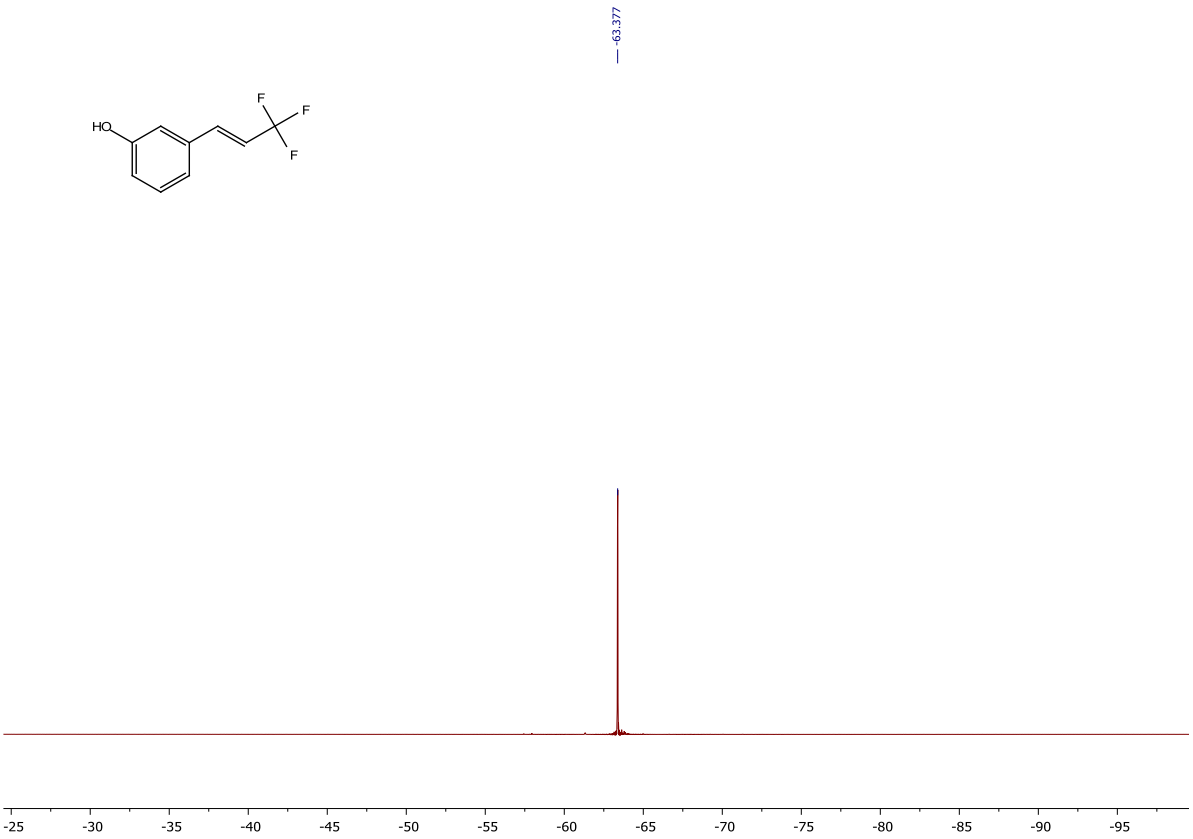



149.403
148.518
137.466
137.398
137.330
137.263
127.943
127.855
125.276
123.554
122.607
119.940
114.473
114.138
113.802
113.467
108.644
106.295
101.689
77.160 CDCl3
F
F
F
O
O
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

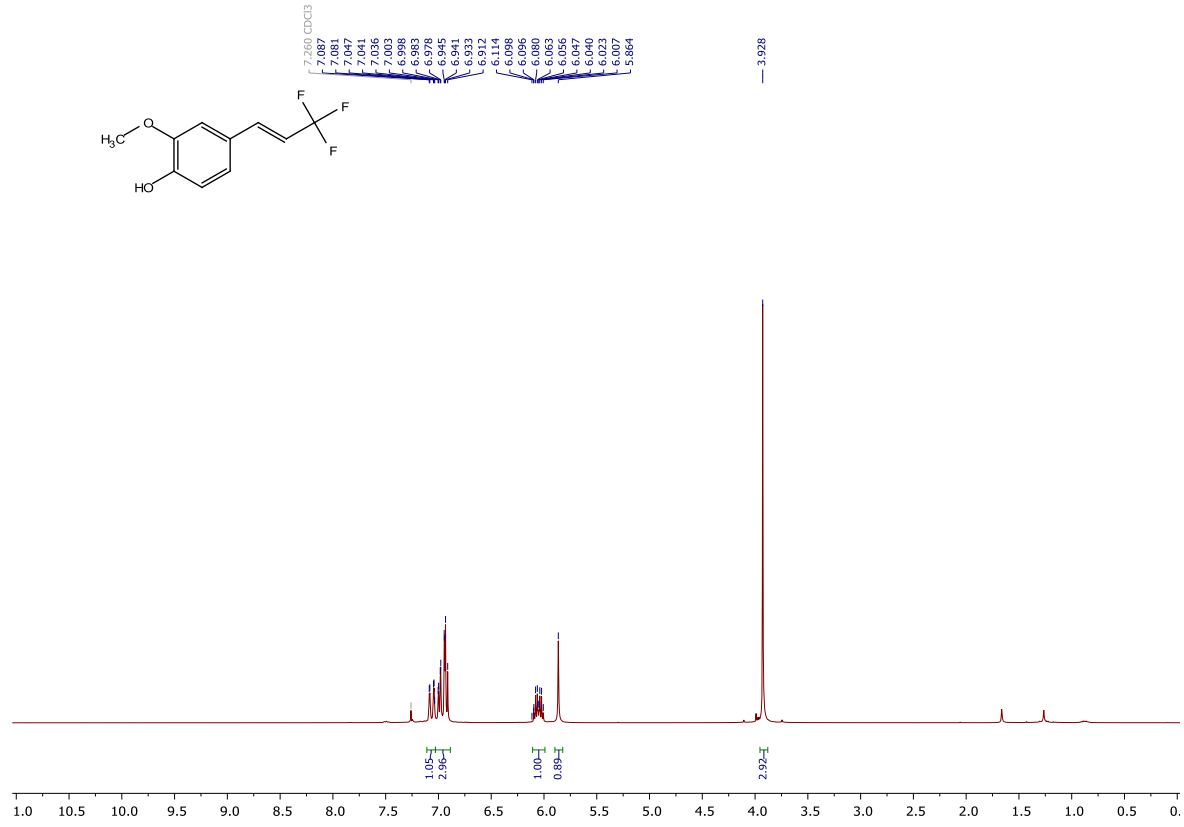

-62.912
F
F
F
O
O
-15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115

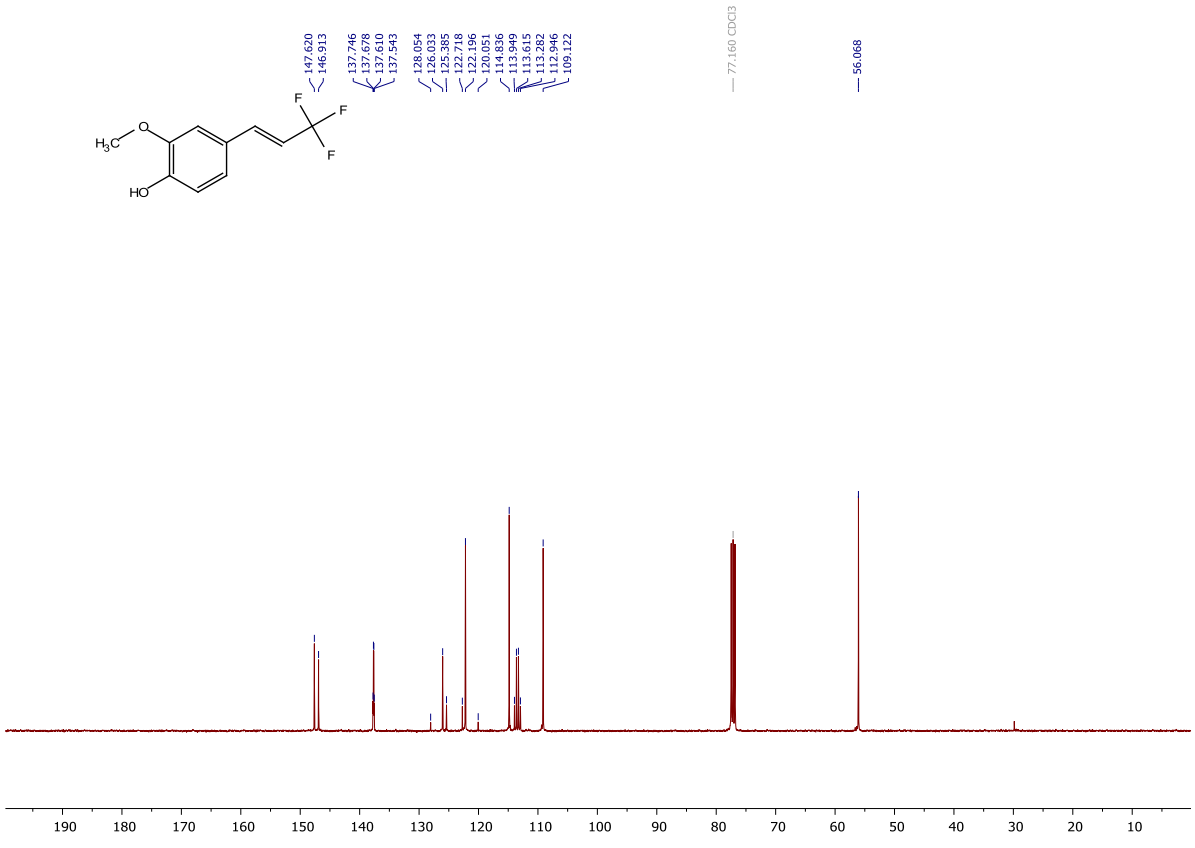
(*E*)-3-(3,3,3-Trifluoroprop-1-en-1-yl)phenol (41)

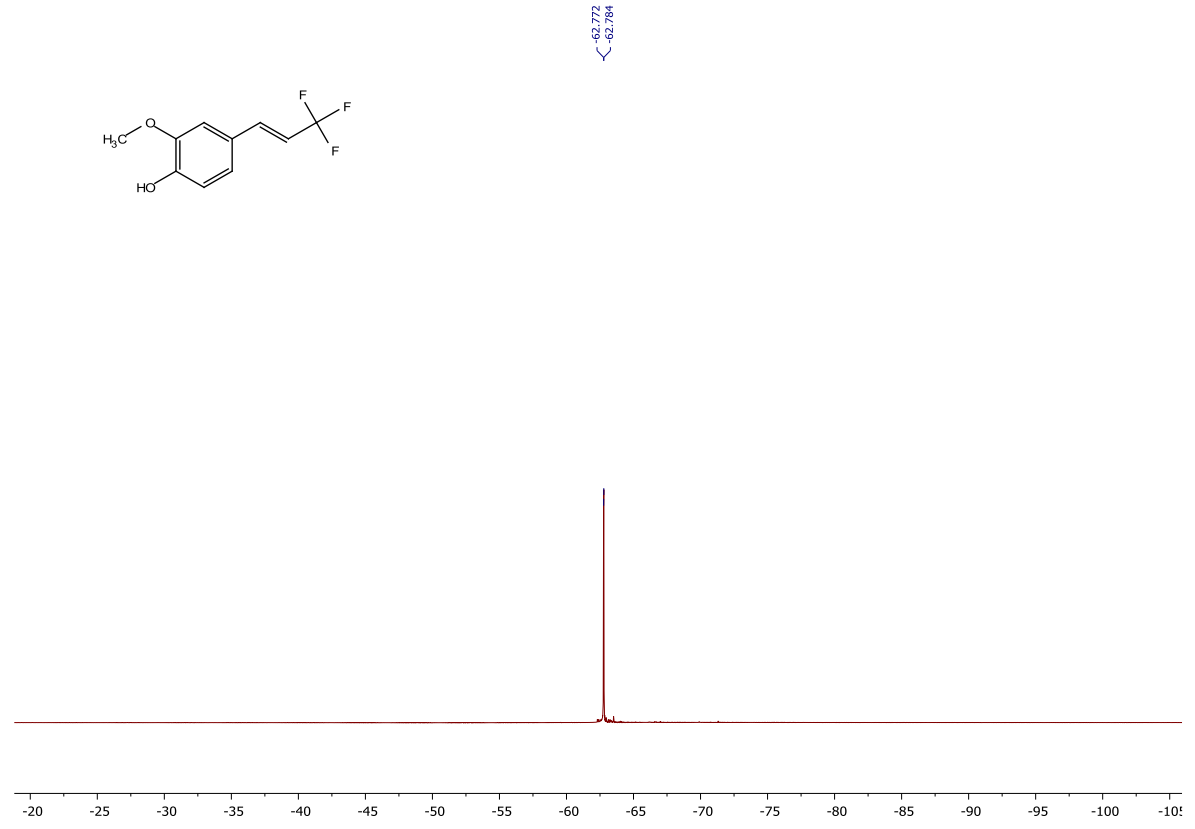




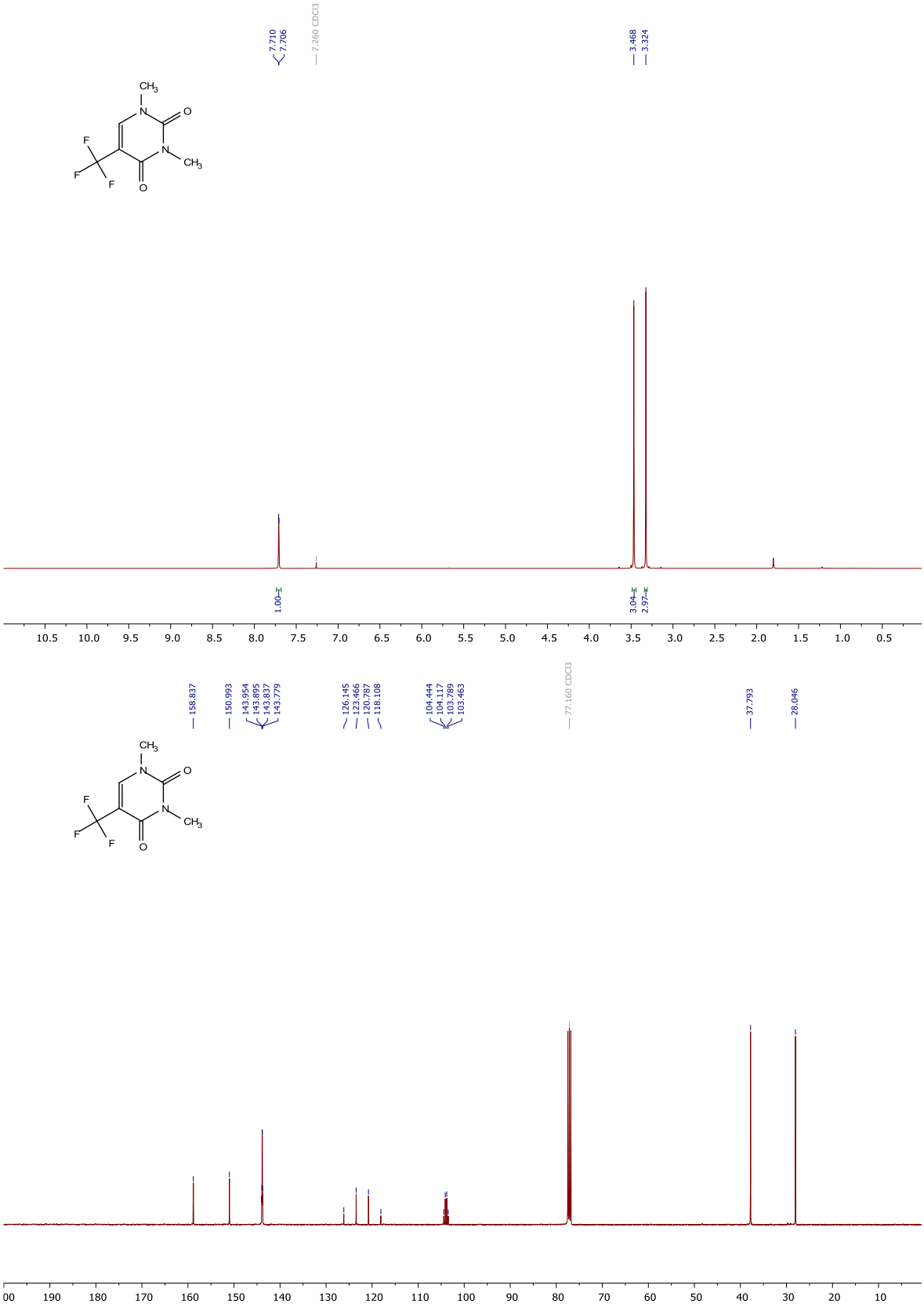
(*E*)-2-Methoxy-4-(3,3,3-trifluoroprop-1-en-1-yl)phenol (42)

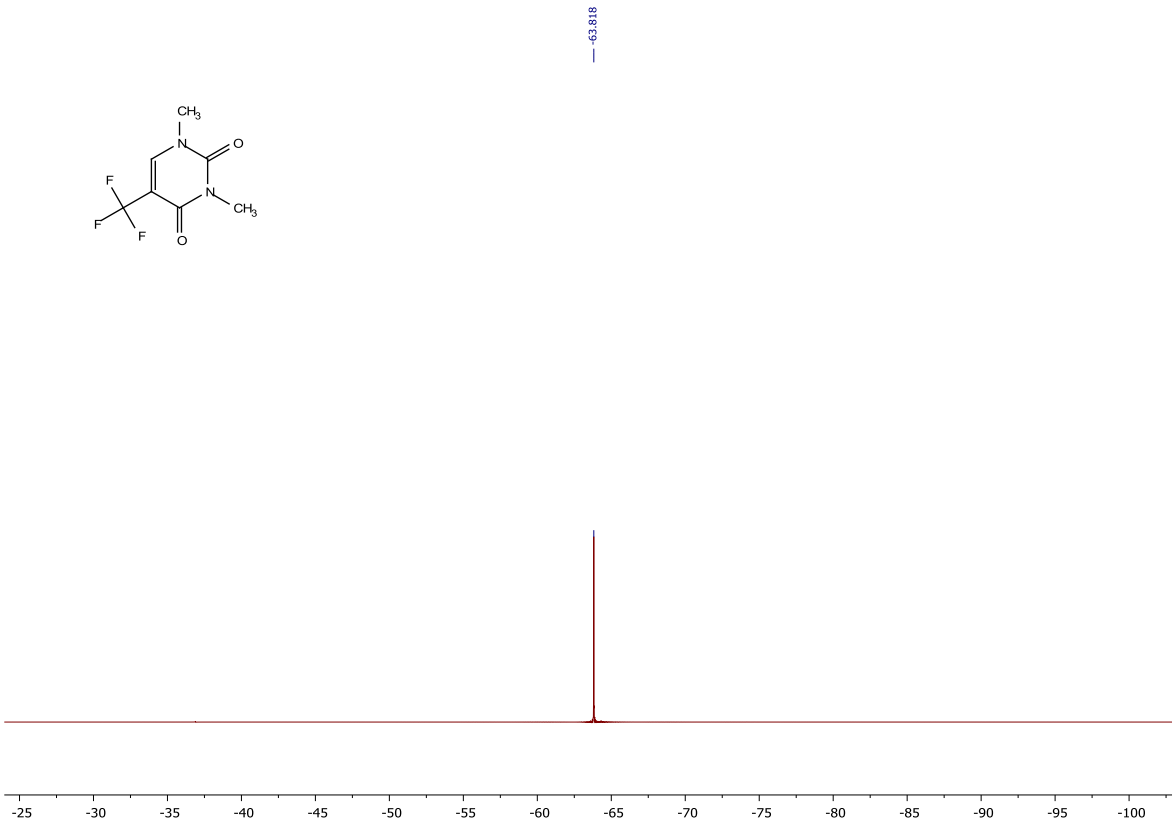



147.620
146.913
137.746
137.678
137.610
137.543
128.054
126.033
125.385
122.718
122.196
120.051
114.836
113.949
113.615
113.282
112.946
109.122
77.160 CDCl3
56.068
F
F
F
O
H3C
HO
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

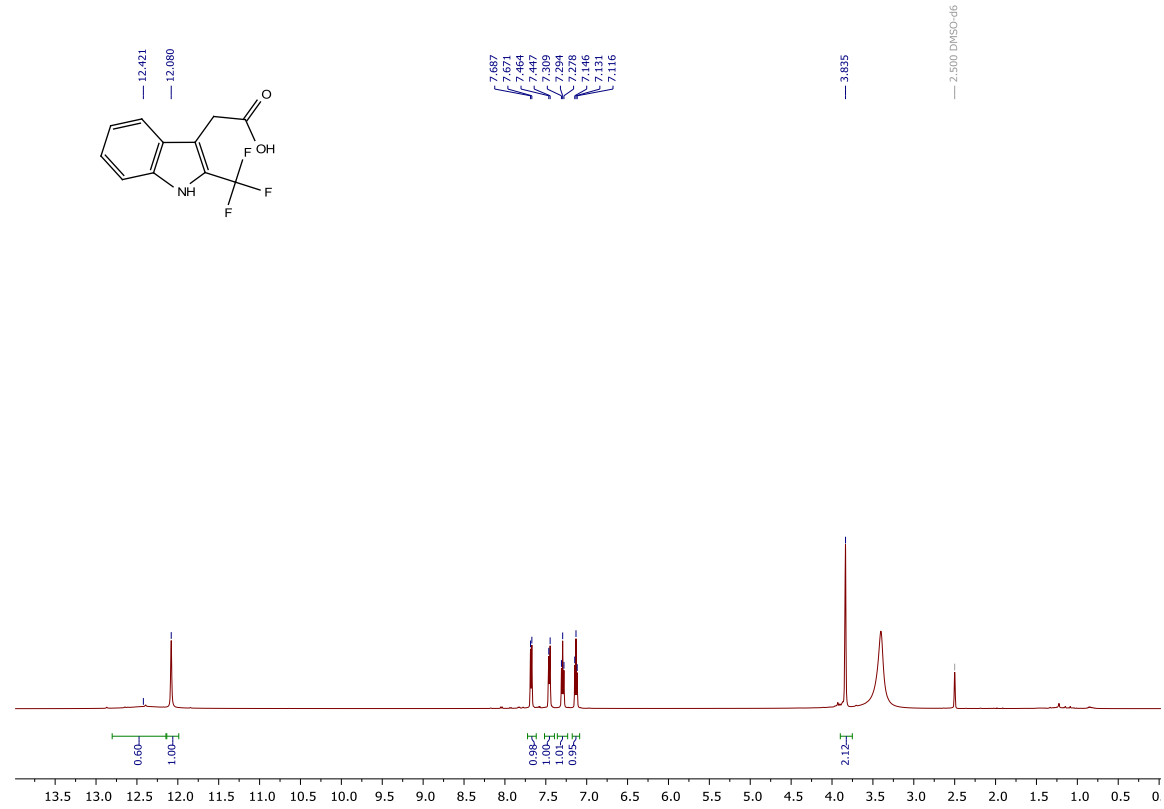

-62.772
-62.784
F
F
F
O
H3C
HO
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105

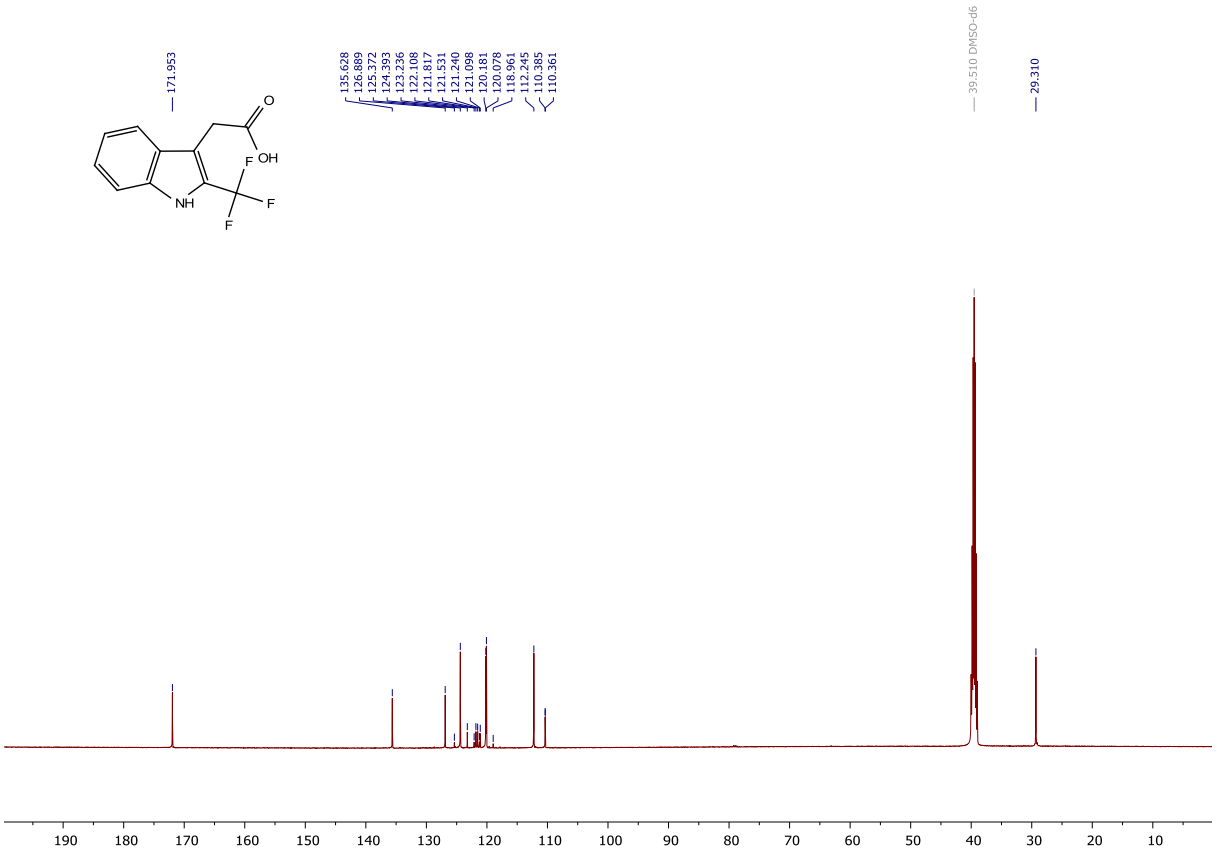
1,3-Dimethyl-5-(trifluoromethyl)pyrimidine-2,4(1*H*,3*H*)-dione (43)

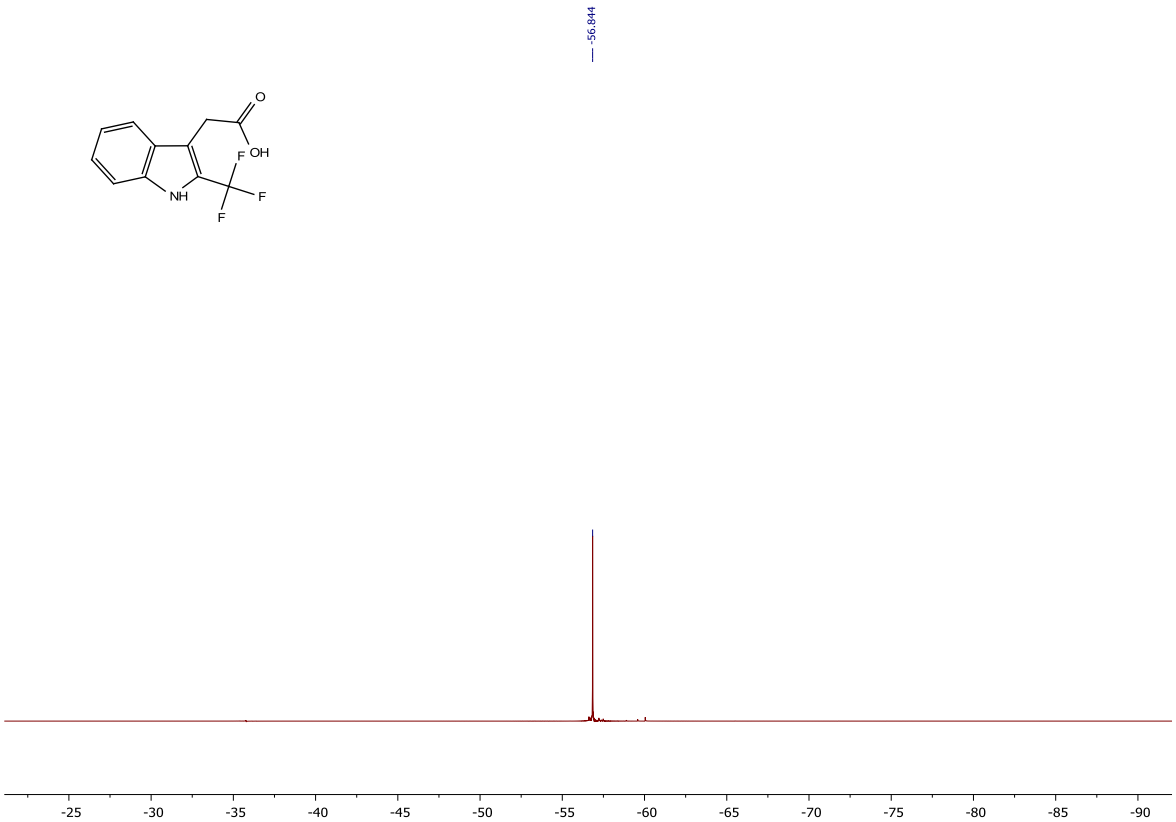




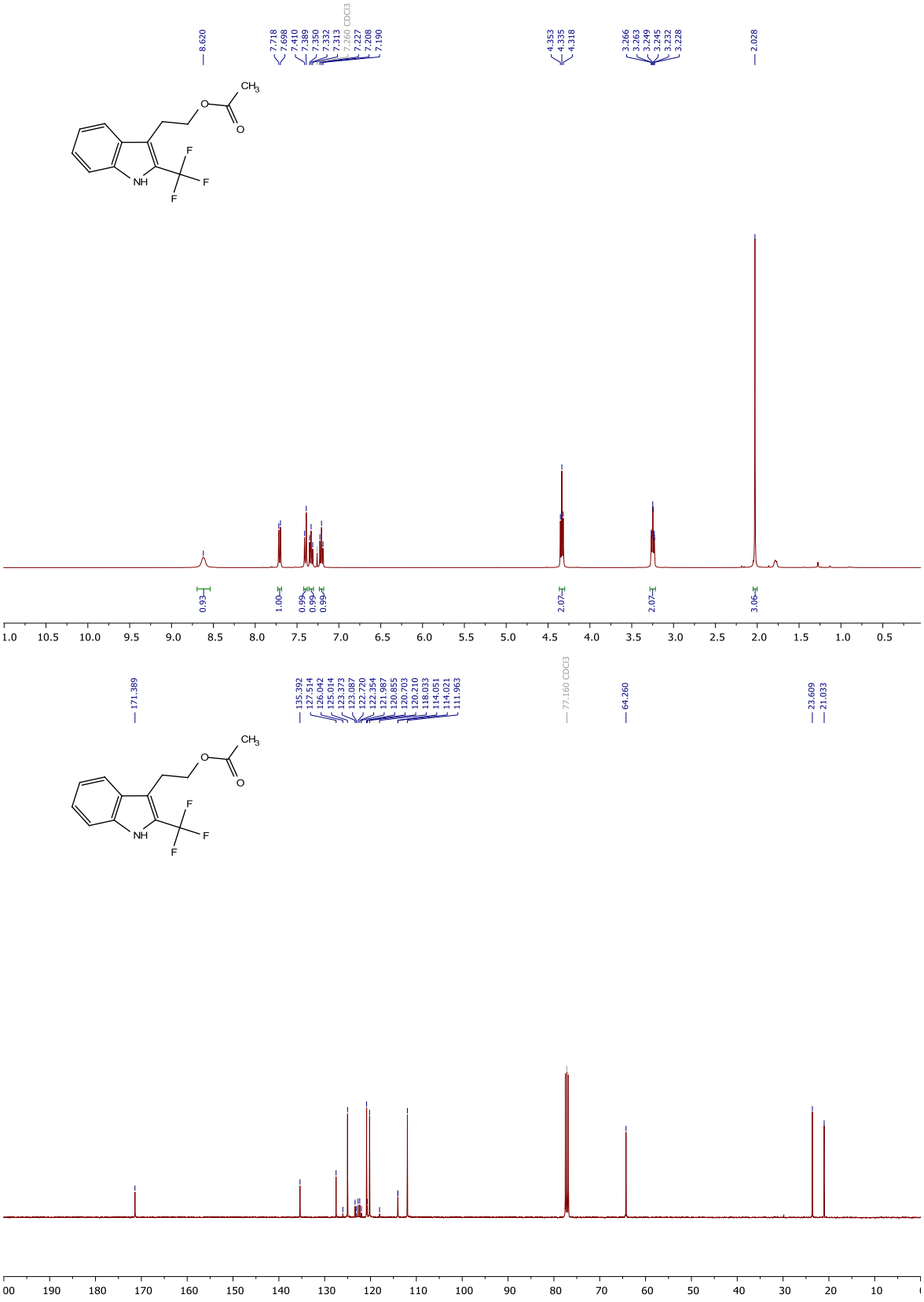
2-(2-(Trifluoromethyl)-1*H*-indol-3-yl)acetic acid (44)

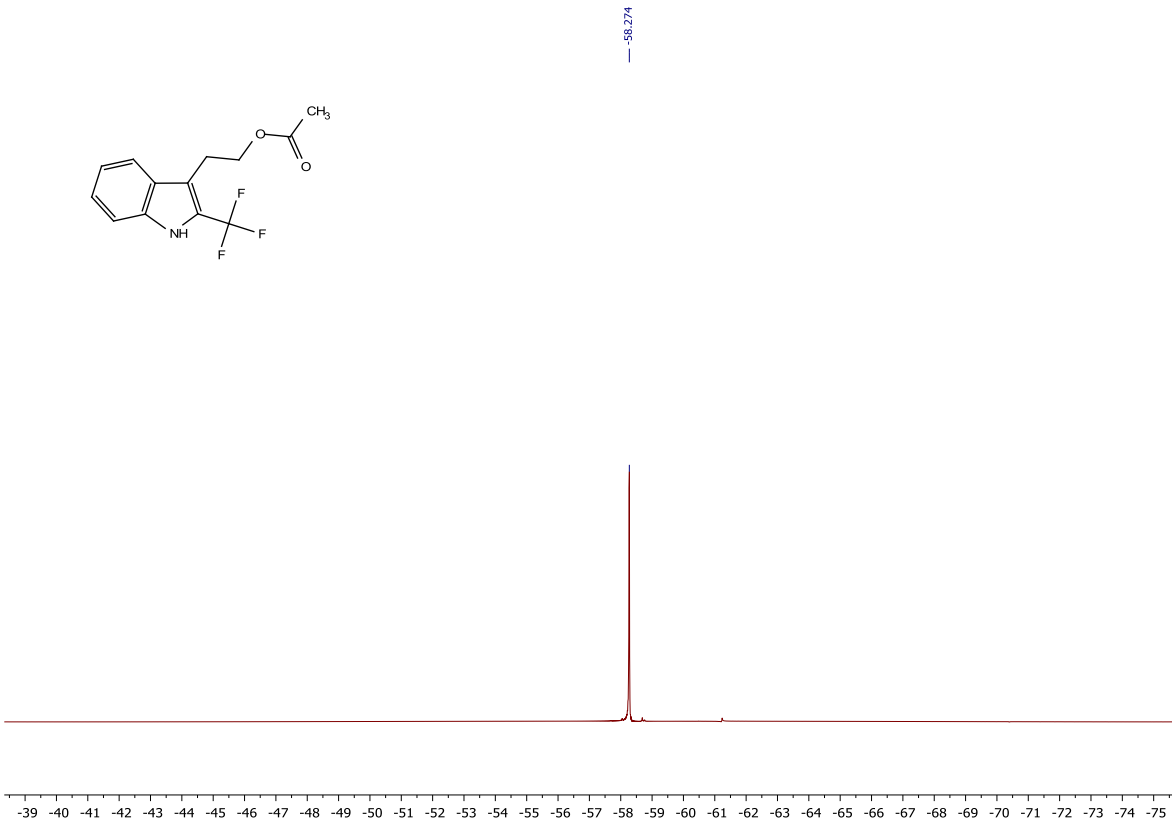



171.953
135.628
126.889
125.372
124.393
123.236
122.108
121.817
121.531
121.240
121.098
120.181
120.078
118.961
112.245
110.385
110.361
39.510 DMSO-d6
29.310
O
OH
F
F
F
NH
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

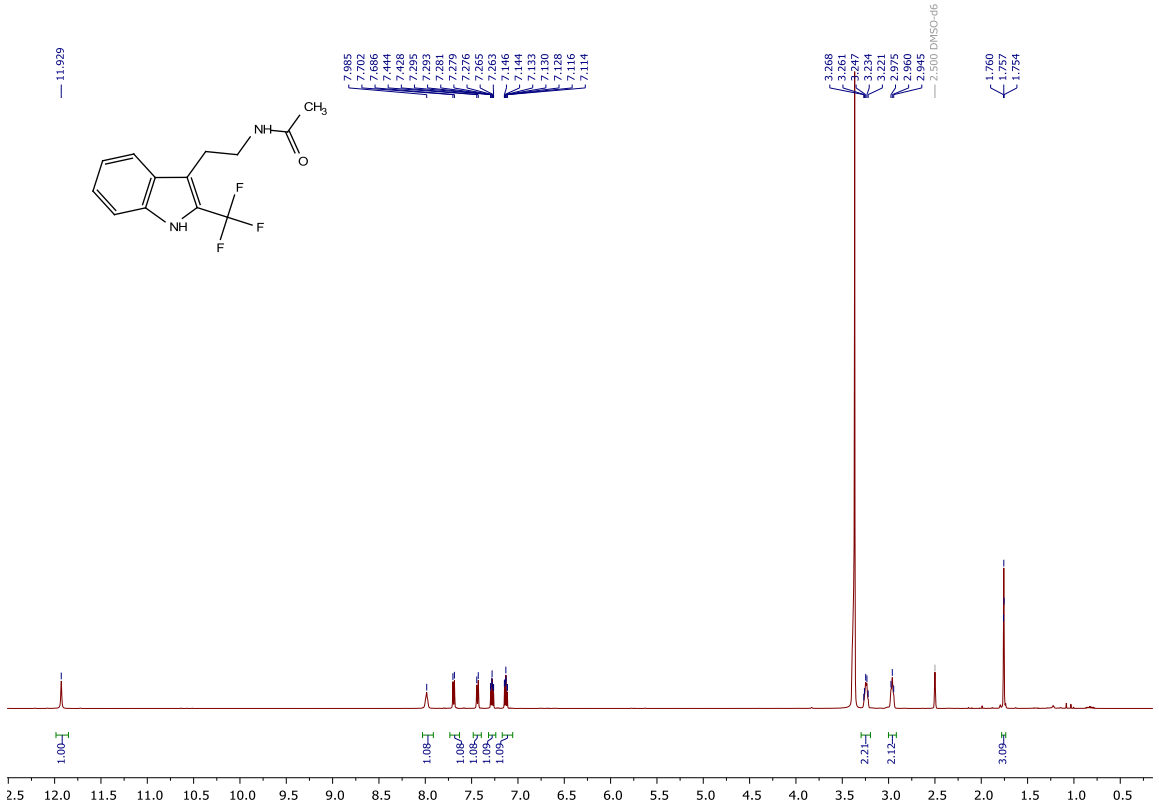

-56.844
O
OH
F
F
F
NH
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90

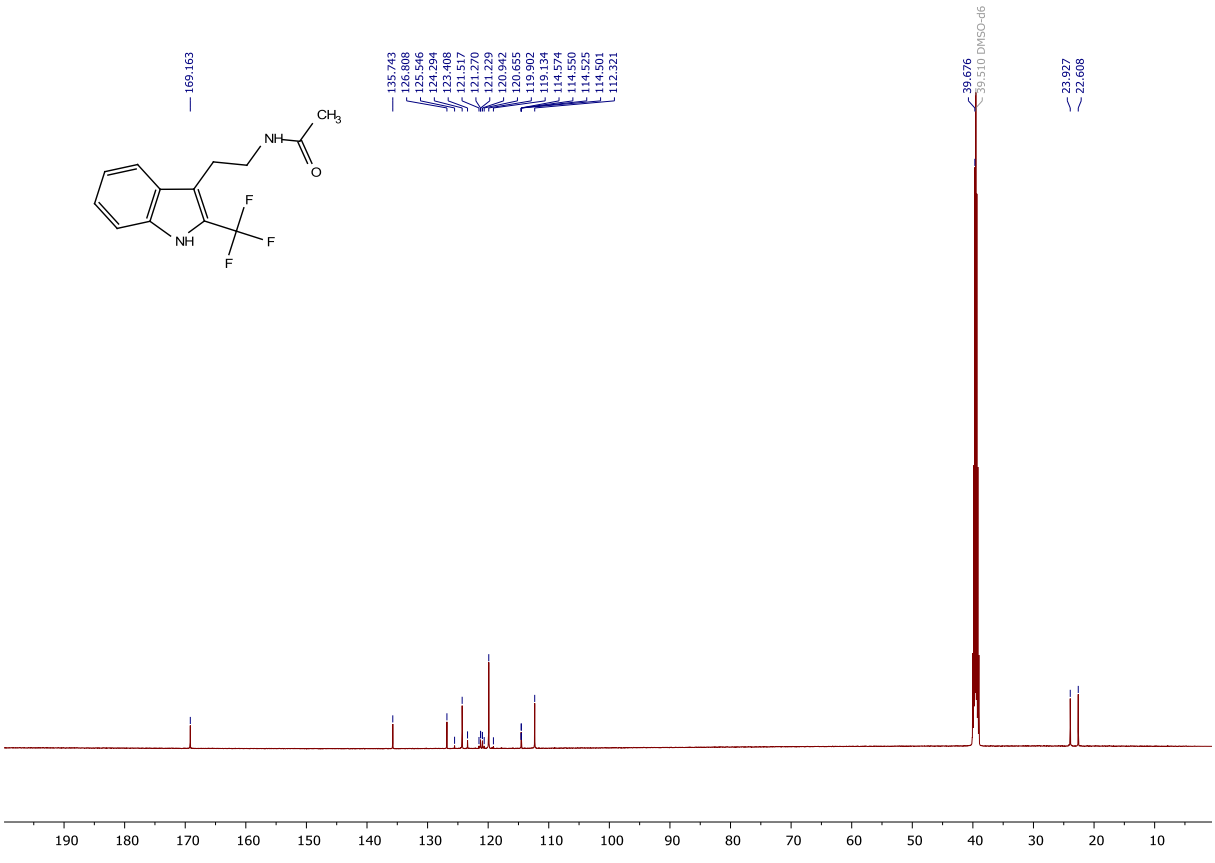
2-(2-(Trifluoromethyl)-1H-indol-3-yl)ethyl acetate (45)

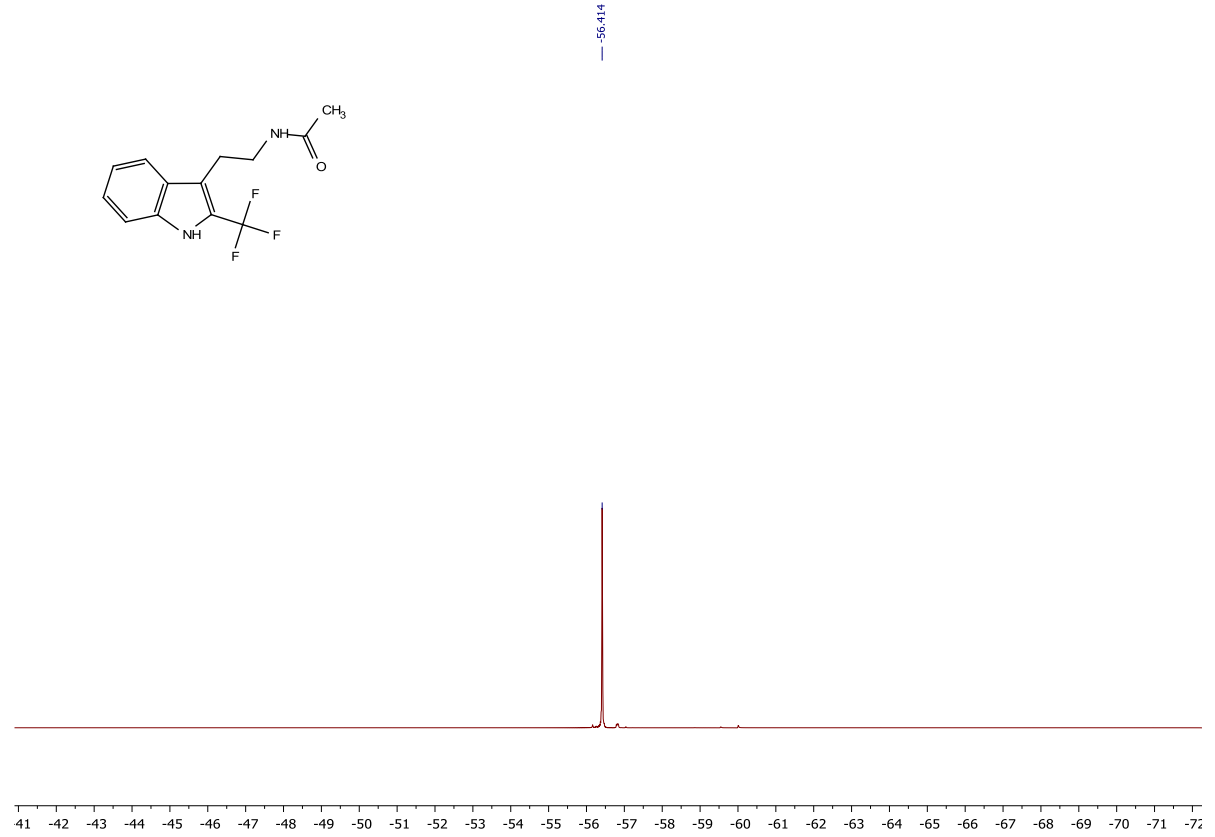




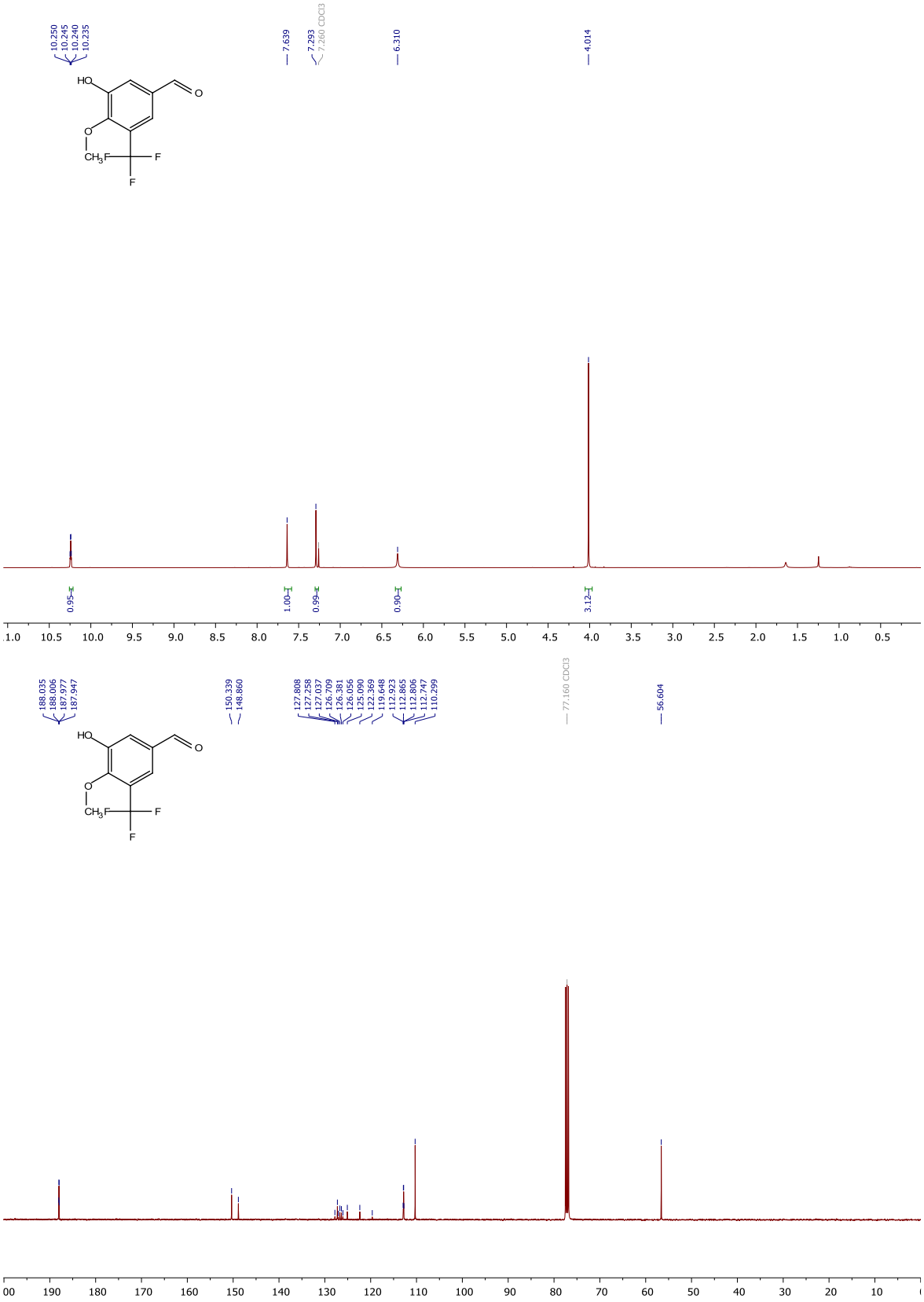
N-(2-(2-(Trifluoromethyl)-1*H*-indol-3-yl)ethyl)acetamide (46)

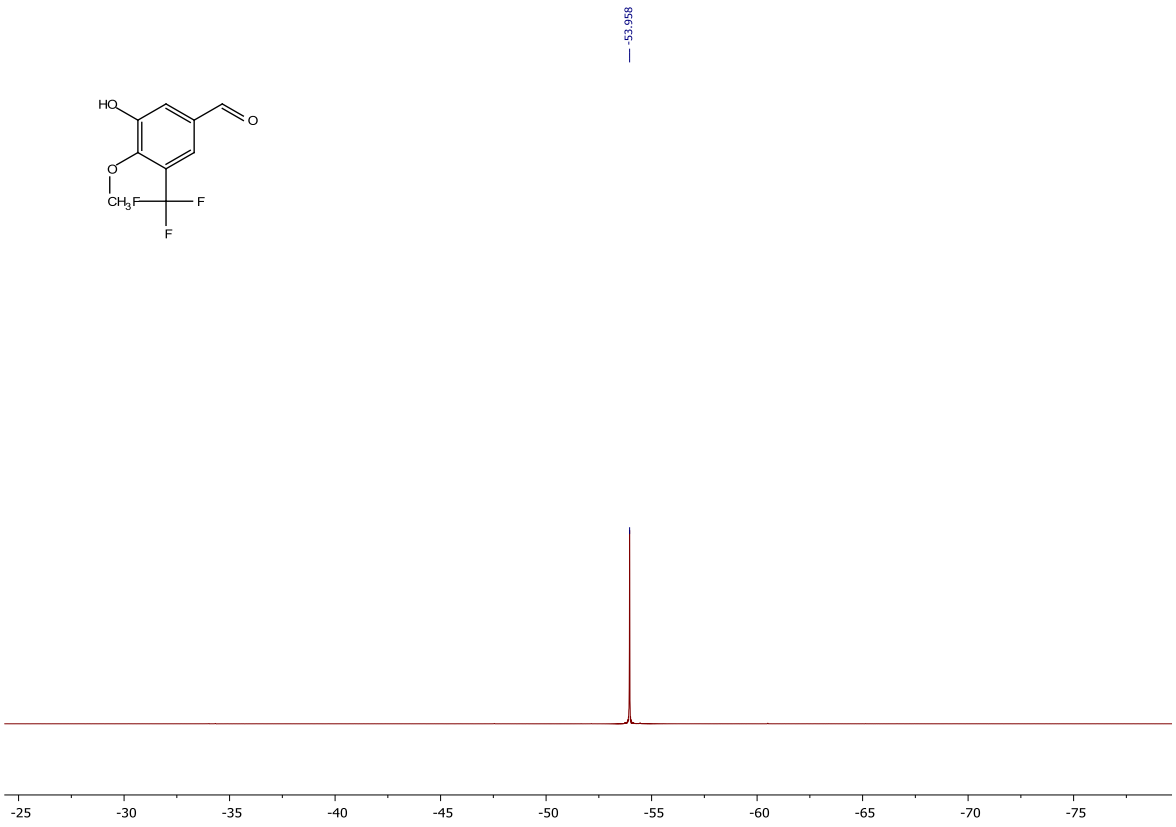



169.163
135.743
126.808
125.546
124.294
123.408
121.517
121.270
121.229
120.942
120.655
119.902
119.134
114.574
114.550
114.525
114.501
112.321
39.676
39.510 DMSO-d6
23.927
22.608
NH
CH3
O
F
F
F
NH
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

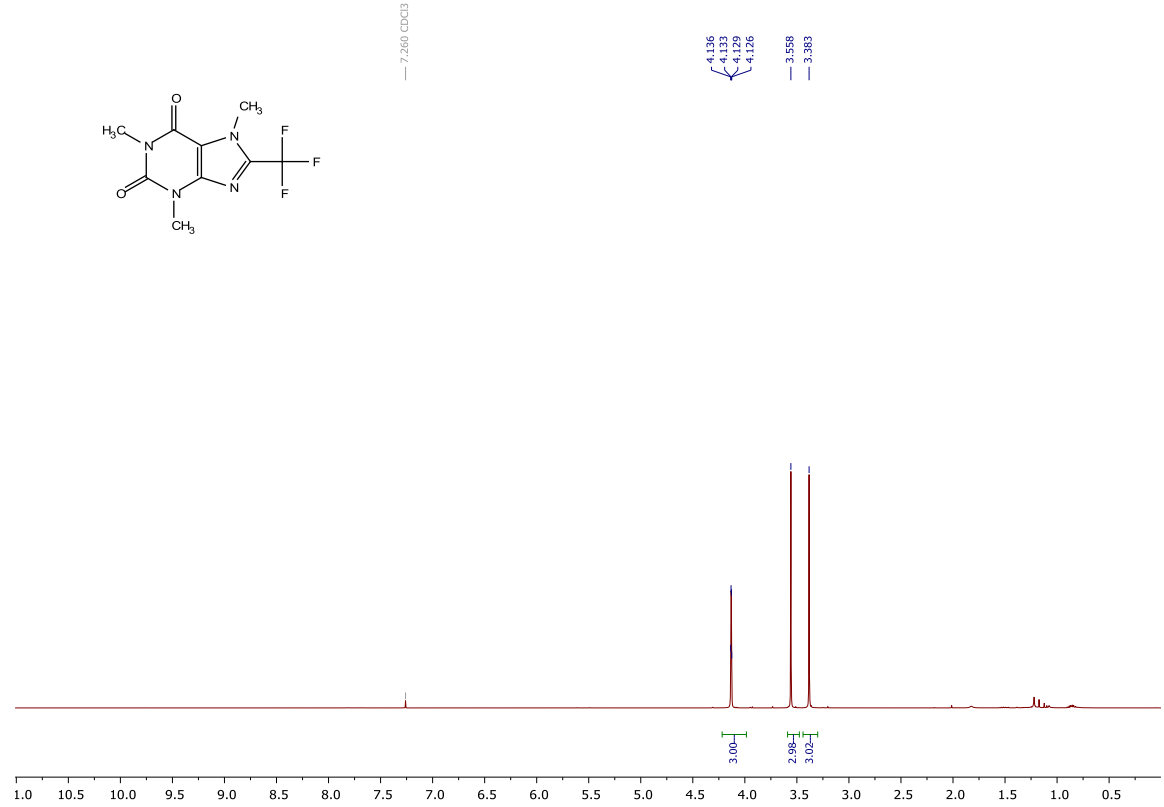

-56.414
NH
CH3
O
F
F
F
NH
-41
-42
-43
-44
-45
-46
-47
-48
-49
-50
-51
-52
-53
-54
-55
-56
-57
-58
-59
-60
-61
-62
-63
-64
-65
-66
-67
-68
-69
-70
-71
-72

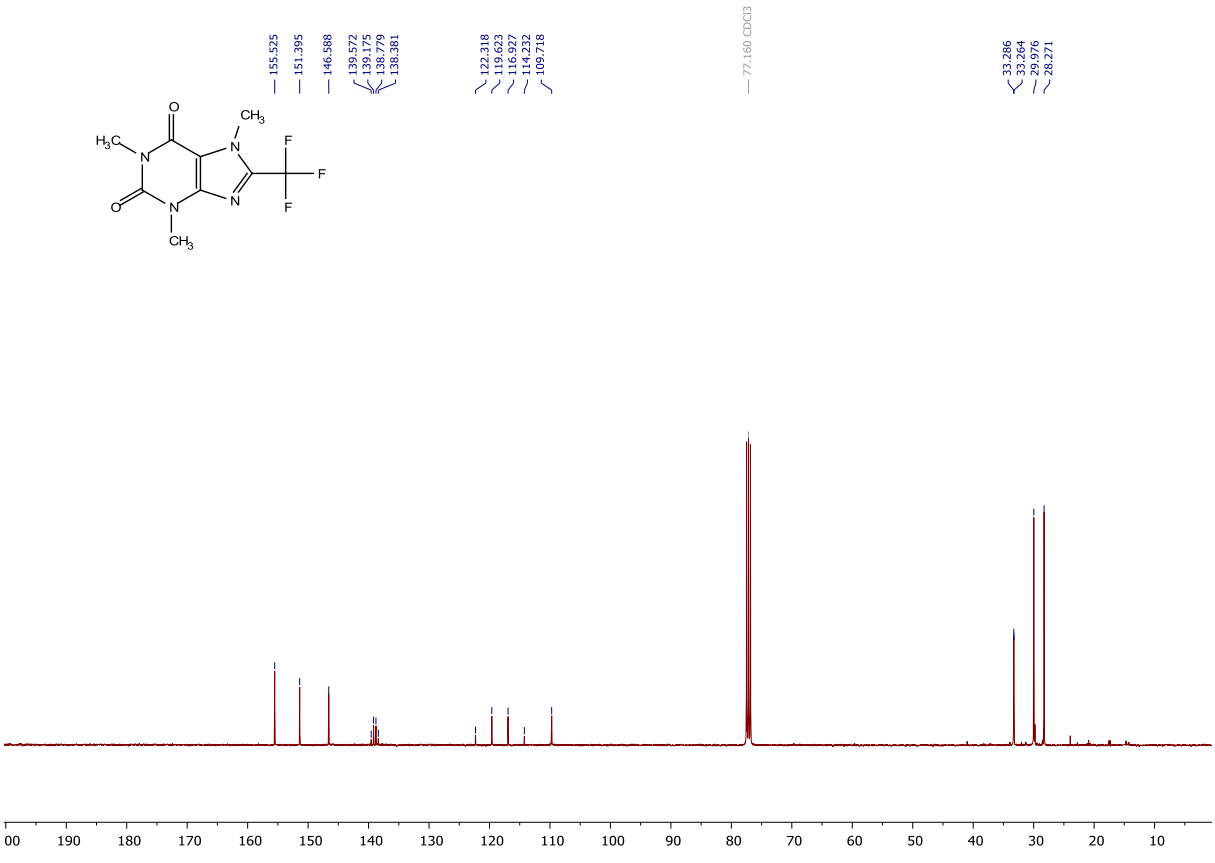
3-Hydroxy-4-methoxy-5-(trifluoromethyl)benzaldehyde (47)

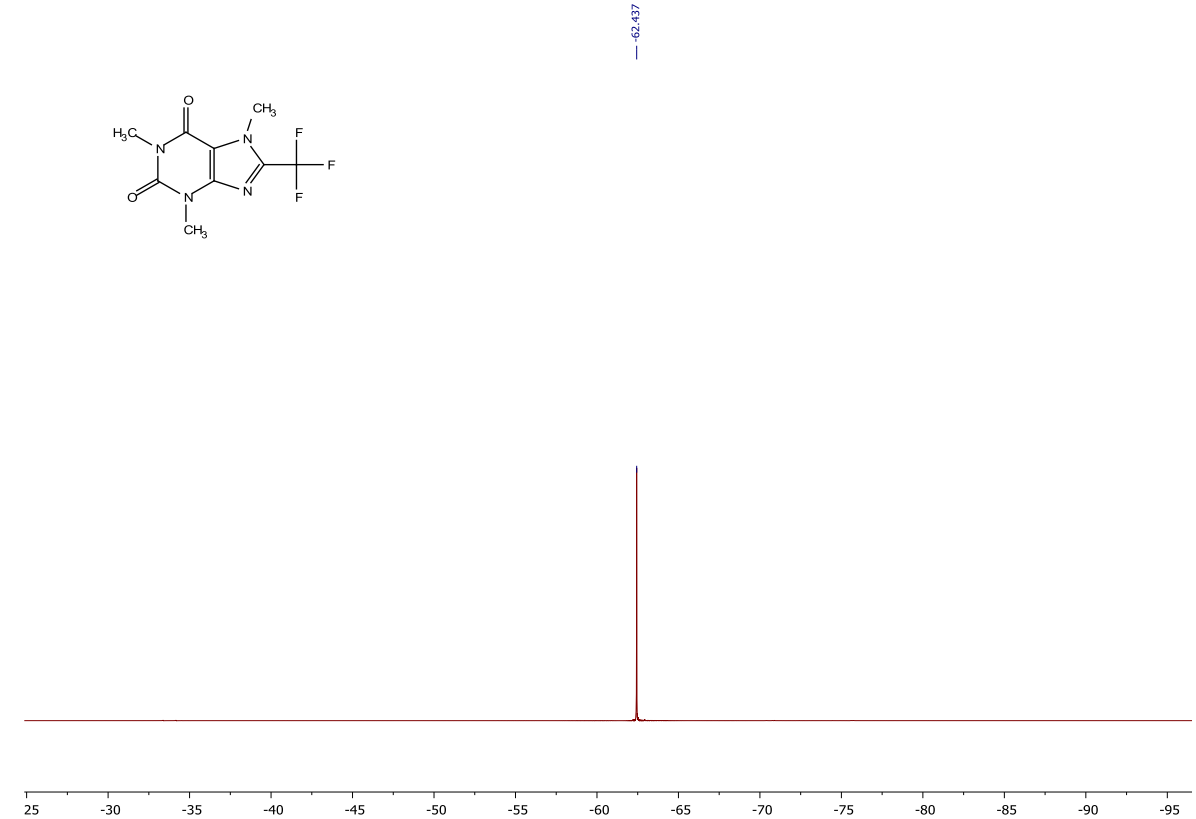




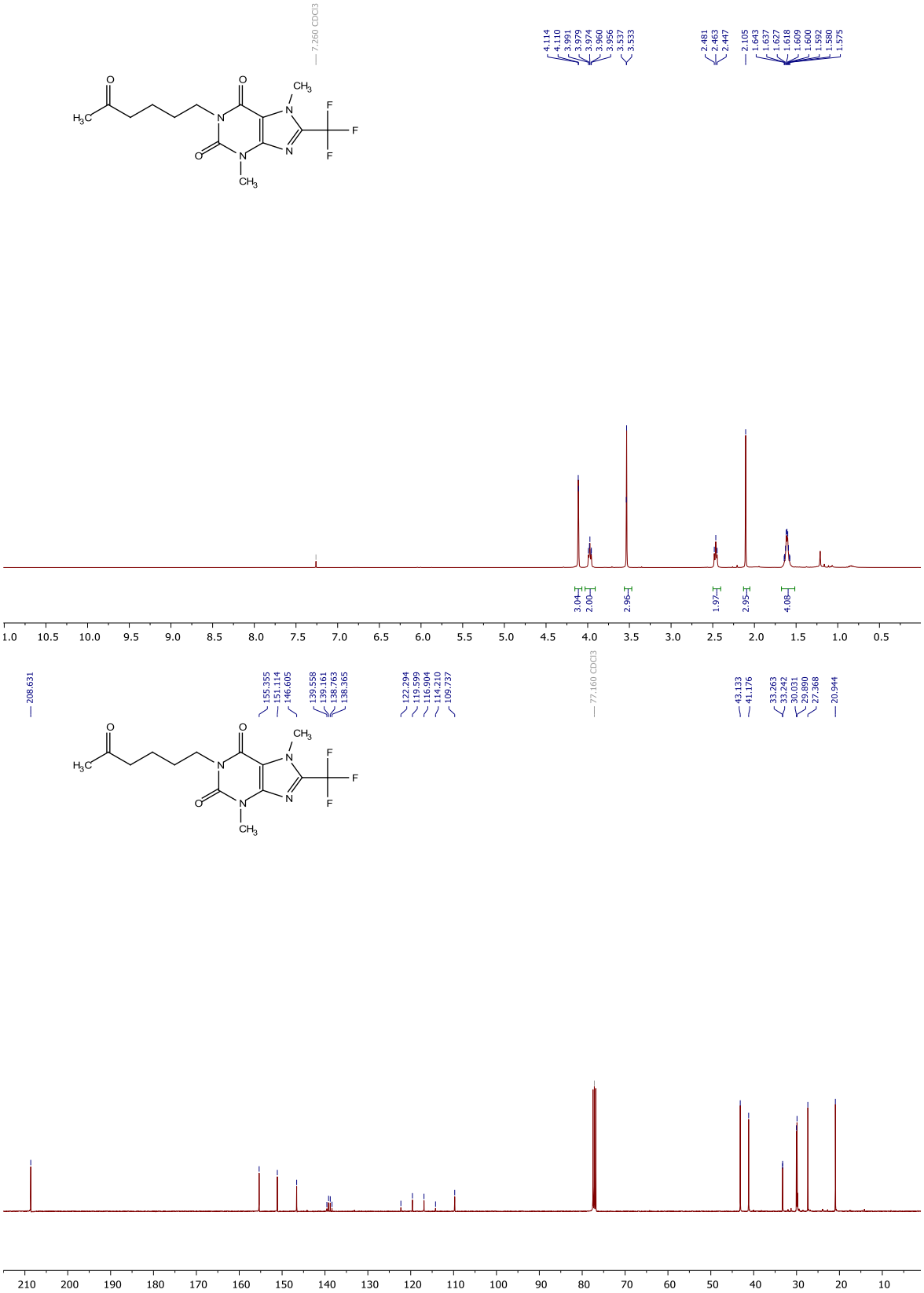
1,3,7-Trimethyl-8-(trifluoromethyl)-3,7-dihydro-1H-purine-2,6-dione (48)

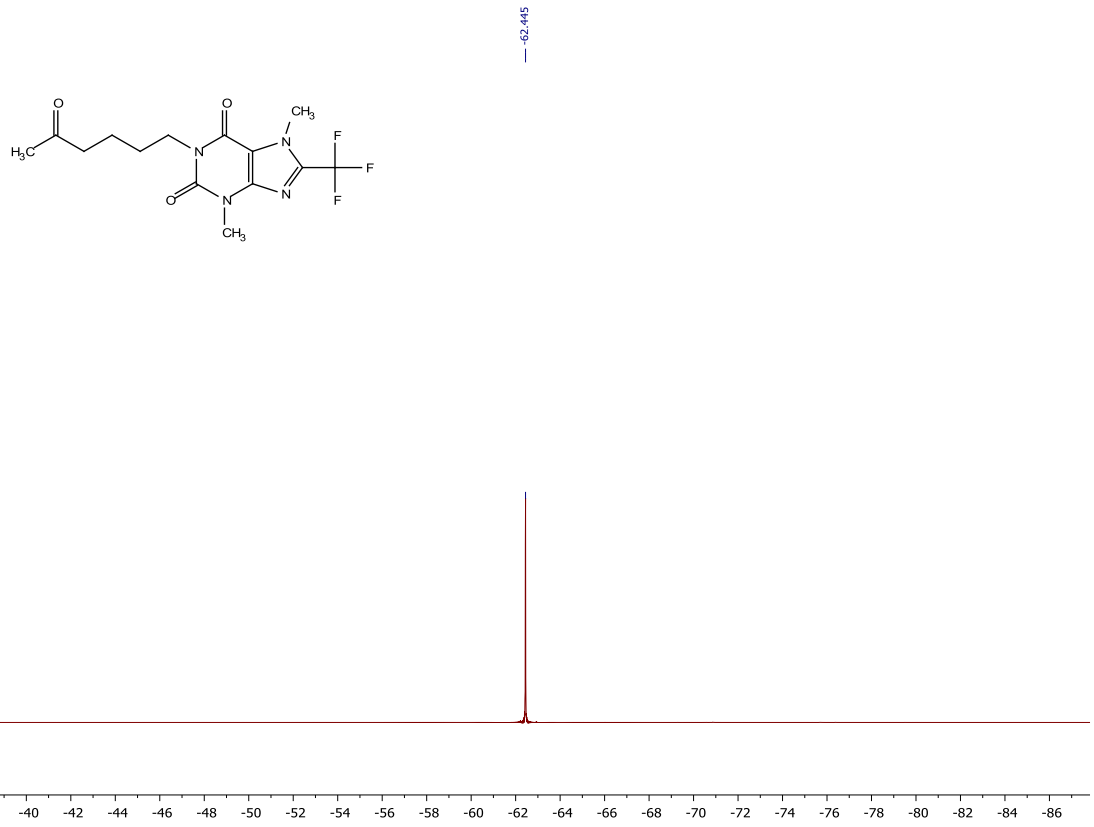



155.525
151.395
146.588
139.572
139.175
138.779
138.381
122.318
119.623
116.927
114.232
109.718
77.160 CDCl3
33.286
33.264
29.976
28.271
O
CH3
H3C
N
F
F
F
O
N
N
CH3
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

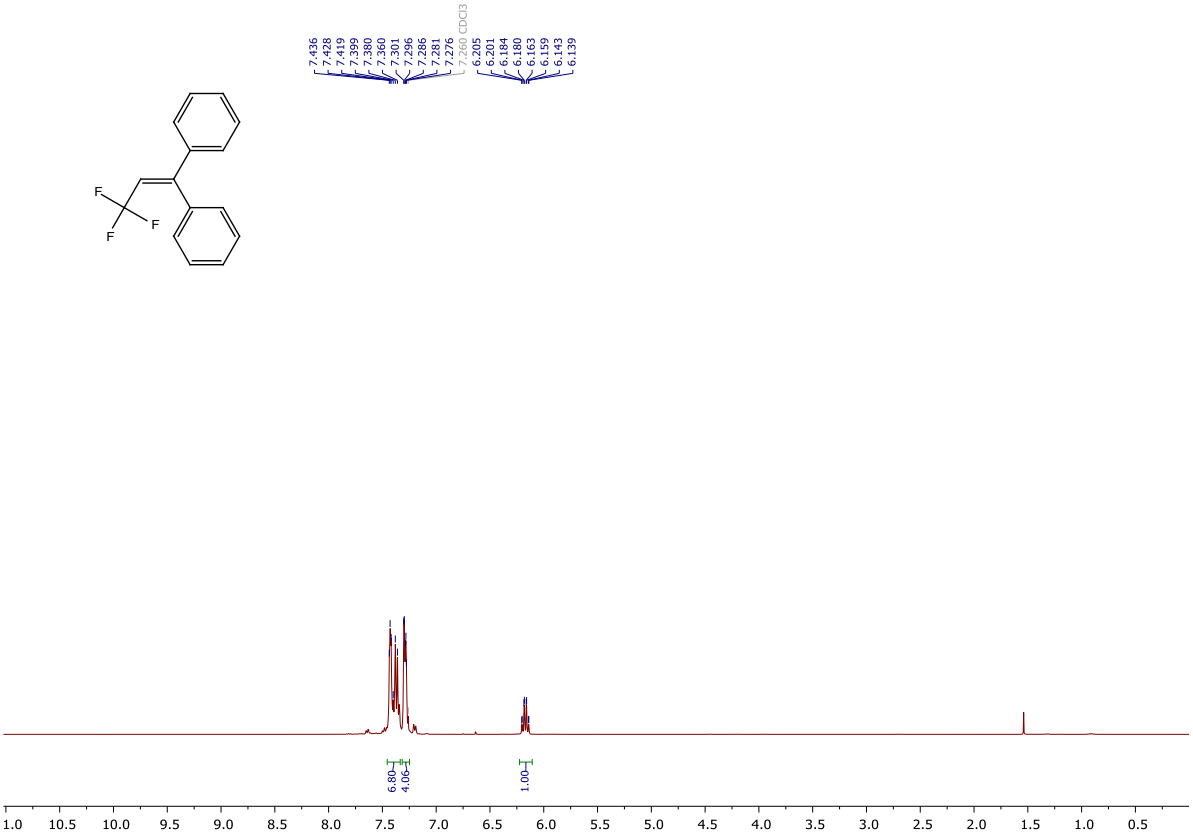

-62.437
O
CH3
H3C
N
F
F
F
O
N
N
CH3
25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95

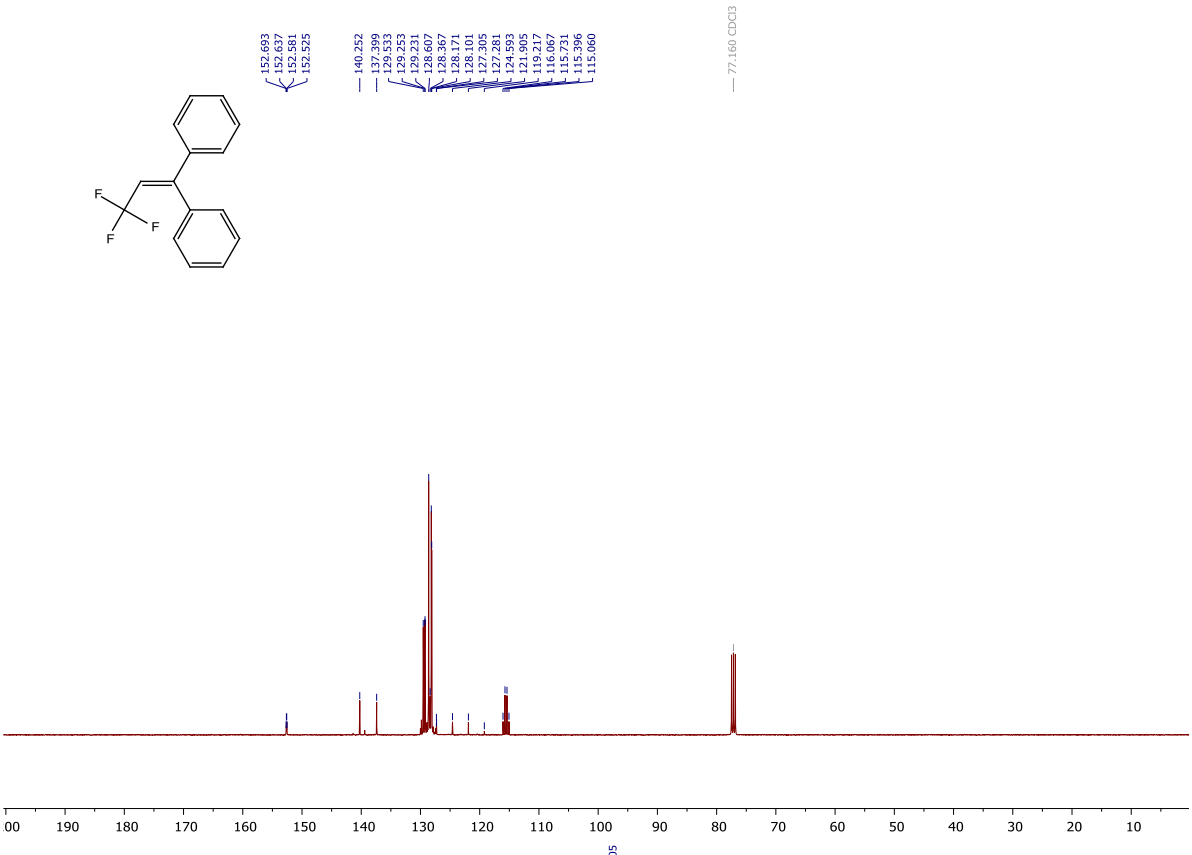
3,7-Dimethyl-1-(5-oxohexyl)-8-(trifluoromethyl)-3,7-dihydro-1*H*-purine-2,6-dione (49)

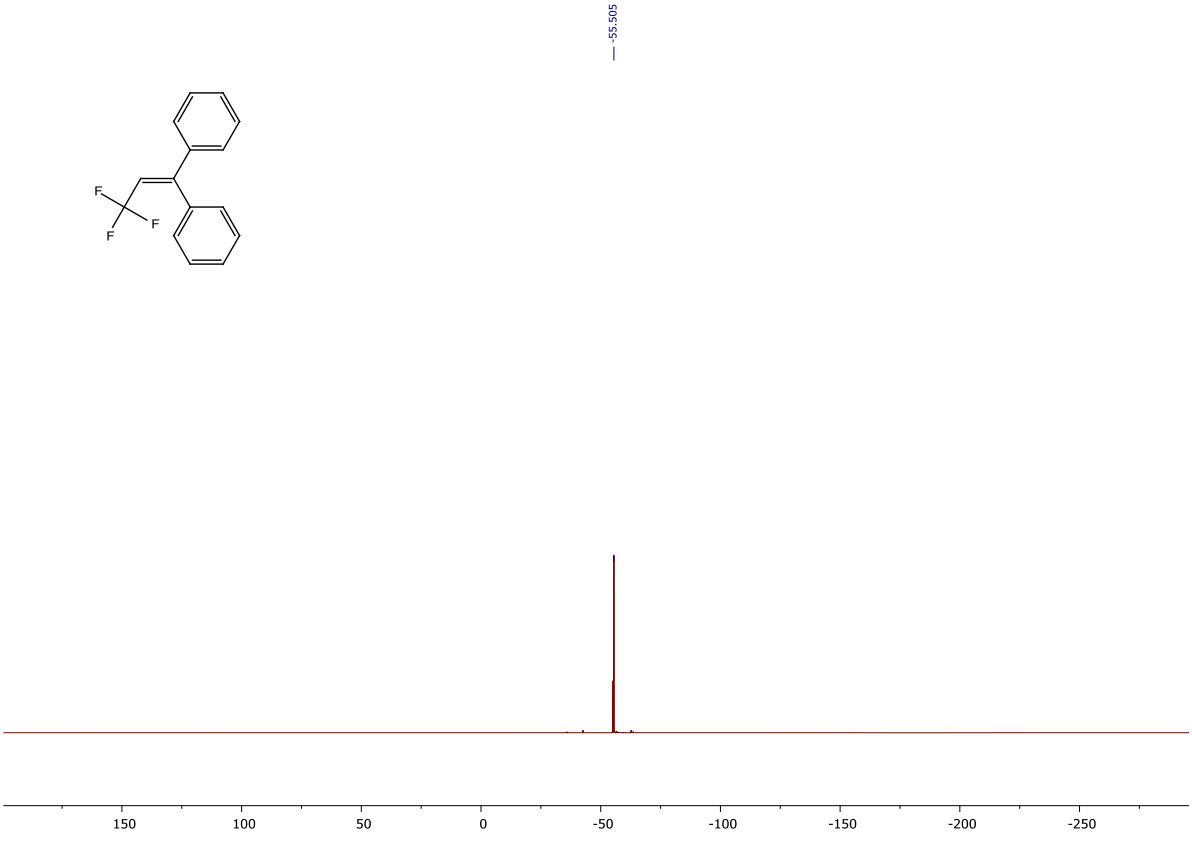




(3,3,3-Trifluoroprop-1-ene-1,1-diyl)dibenzene




152.693
152.637
152.581
152.525
140.252
137.399
129.533
129.253
129.231
128.607
128.367
128.171
128.101
127.305
127.281
124.593
121.905
119.217
116.667
115.731
115.396
115.060
77.160 CDCl3
F
F
F
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


-55.505
F
F
F
150
100
50
0
-50
-100
-150
-200
-250