

Sustainable Electrocatalytic Oxidation of *N*-Alkylamides to Acyclic Imides Using H₂O

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1. General Information

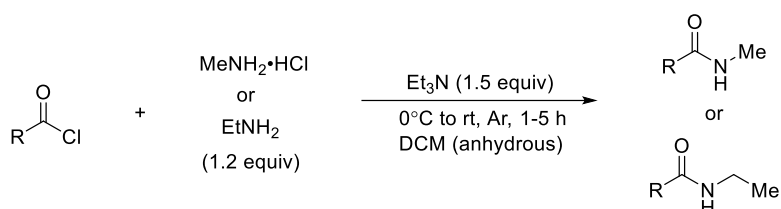
Unless otherwise noted, all chemicals were purchased from commercial suppliers (Sigma Aldrich, TCI, Oakwood) and used without further purification. When required, solvents were dried according to general purification methods. The product mixtures were analyzed by thin layer chromatography using TLC silica gel plates (MerckSchuchardt) with fluorescent indicator ($\lambda = 254$ nm). The purification of the products was performed by flash column chromatography using silica gel 60 (63-200 μm) from SANPONT. ^1H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded on a Bruker AV-III400 (400 MHz) or AMX500 (500 MHz) spectrometer. Chemical shifts were calibrated using residual undeuterated solvent as an internal reference (CDCl_3 : 7.26 ppm ^1H NMR, 77.00 ppm ^{13}C NMR). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), td (triplet of doublets), dt (doublet of triplets), ddd (doublet of doublets of doublets), brs (broad singlet). All high-resolution mass spectra (HRMS) were obtained on a Finnigan/MAT 95XL-T spectrometer. Cyclic voltammetry was performed using an Ametek Versa STAT 3. Absorption spectra were recorded in 1 cm path quartz cuvettes using an Edinburgh FS-5 spectrofluorometer. Continuous wave X-band ESR spectra were obtained with a JEOL (FA200) spectrometer.

2. Synthesis of Amides

2.1. Amides **1a**, **1b**, **1c**, **1d**, **1e**, **1f**, **1g**, **1h**, **1i**, **1j**, **1k**, **1l**, **1m**, **1n**, **1p**, **1s**, **1x**, **1y**, **1aa**, **1ab**, **1ag**, **1ai**, **1ak**, **1al**. were known compounds and prepared according to the literature procedures.

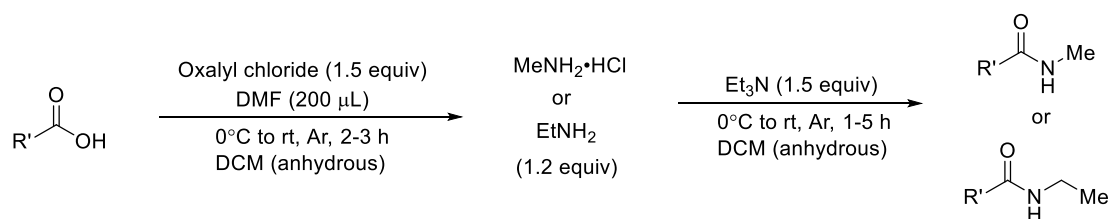
Other Amides used in this work were prepared by the following two procedures:

2.2. General procedure A:



To an ice solution of amine (1.2 equiv) and triethylamine (1.5 equiv) in DCM (anhydrous) (0.25 M). Acyl chloride (1.0 equiv) was added dropwise. The reaction mixture was proceed until TLC indicating reaction complete. The reaction was then quenched with H_2O and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over Na_2SO_4 , and concentrated. The crude residue was purified by flash column chromatography to give the desired amides.

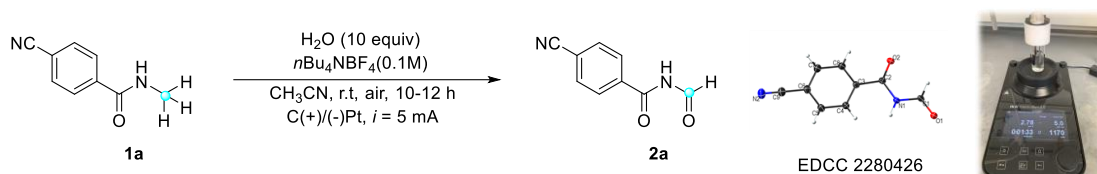
2.3. General procedure B:



To an ice solution of acid (1.0 equiv) and DMF (catalytic) in DCM (anhydrous) (0.25 M), Oxalyl chloride (1.0 equiv) was added dropwise, keep the reaction stirred under 0°C about 30 min, then remove the ice bath. The resulting solution was stirred at room temperature for 2 h. The solvent was evaporated under reduced pressure to afford acyl chloride, which was directly used for the next step without further purification.

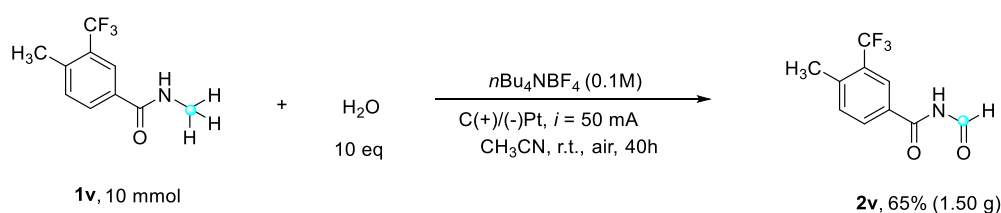
Subsequently, an ice solution of amine (1.2 equiv) and triethylamine (1.5 equiv) in DCM (anhydrous) (0.25 M). Acyl chloride (1.0 equiv) was added dropwise. The reaction mixture was proceeded until TLC indicating reaction complete. The reaction was then quenched with H₂O and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over Na₂SO₄, and concentrated. The crude residue was purified by flash column chromatography to give the desired amides.

3. Typical procedure.



(using ElectroSyn 2.0). The ElectroSyn vial (10 mL volume) was charged with a magnetic stir bar, amide (0.2 mmol, 1.0 equiv.) and H₂O (36 μ L, 10 equiv.) *n*Bu₄NBF₄ (0.5 mmol), CH₃CN (5 mL) (Liquid substrate was added after solvent). The graphite plate anode and Pt plate cathode were adapted on the ElectroSyn vial cap and the vial cap was screwed onto the vial tightly. The vial was adapted onto the vial holder of ElectroSyn 2.0. Parameters were set: “new experiment” at constant current to 5.0 mA, selecting “No” for “use of reference electrode” and adjusting the reaction time “10-12 h” The reaction mixture was quenched by removing from the electricity. Upon completion, the reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (1:20 ethyl acetate/petroleum ether) to afford **2a**

4. A gram scale reaction 1v



The gram scale reaction was conducted in a 100 mL beaker-type cell equipped with a graphite plate anode (6 cm x 4 cm x 0.4 cm) and a platinum plate (5 cm x 5 cm x 0.1 cm) cathode. The two electrodes were placed in parallel. The cell was charged with a magnetic stir bar, *N*, 4-dimethyl-3-(trifluoromethyl)benzamide (2.17 gram, 10 mmol.), H₂O (1.8 mL, 10 equiv.), and *n*Bu₄NBF₄ (5 mmol) and CH₃CN (50 mL) (Liquid substrate was added after solvent). The reaction was carried out at room temperature using a constant current of 50 mA for 40 h (under atmosphere). The reaction mixture was quenched by removing from the electricity. Upon completion, the reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (1:20 ethyl acetate/petroleum ether) to afford **2v** a white solid (1.50g, 65% yield)

5 Mechanistic study experiments

5.1 ¹⁸O-Labeling Experiment

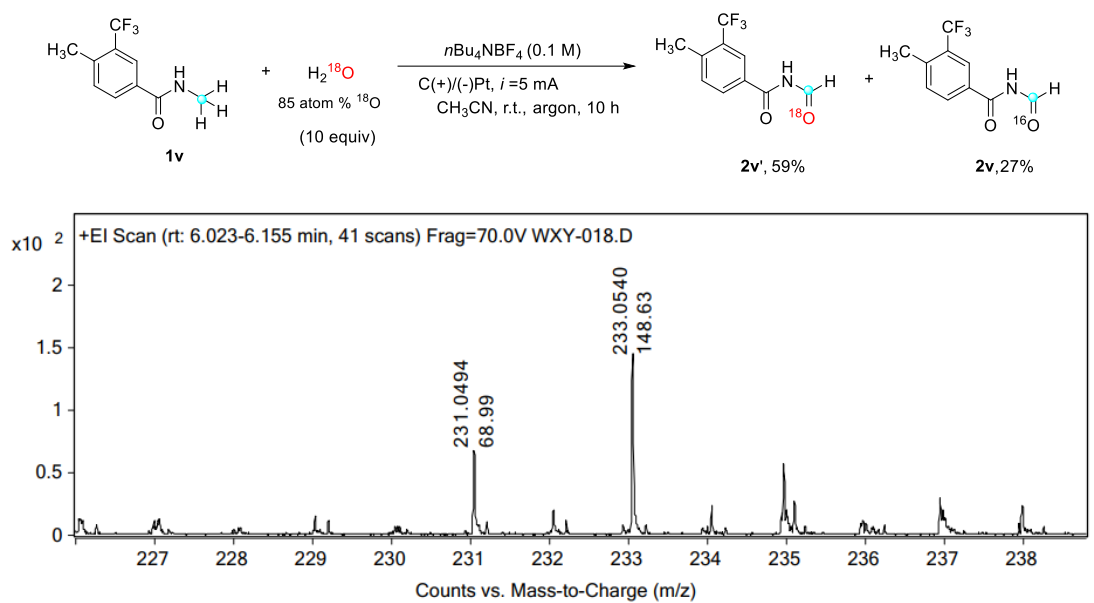
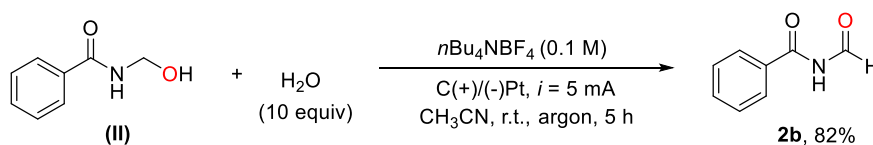


Figure 1. The HRMS determination of the proportion of ¹⁸O

(using ElectraSyn 2.0). The ElectraSyn vial (10 mL volume) was charged with a magnetic stir bar, amide (0.2 mmol, 1.0 equiv.) and H_2^{18}O (36 μL , 10 equiv, 85 atom % ¹⁸O.) $n\text{Bu}_4\text{NBF}_4$ (0.5 mmol), CH_3CN (5 mL) (Liquid substrate was added after solvent). The graphite plate anode and Pt plate cathode were adapted on the ElectraSyn vial cap and the vial cap was screwed onto the vial tightly. An argon filled balloon was adapted through the cap to bubble 3 minutes and then maintain an argon atmosphere. The vial was adapted onto the vial holder of ElectraSyn 2.0. Parameters were set: “new experiment” at constant current to 5.0 mA, selecting “No” for “use of reference electrode” and adjusting the reaction time “10 h” The reaction mixture was quenched by removing from the electricity. Checking the HRMS of the crude reaction mixture, got the **figure 1**. The reaction mixture was concentrated under reduced pressure, the residue was purified by flash column chromatography on silica gel (1:20 ethyl acetate/petroleum ether) to afford the white solid title compound (39.8 mg, 86%). ¹H NMR (400 MHz, CDCl_3) δ 9.18 (d, $J = 8.0 \text{ Hz}$, 1H), 8.83 (br, 1H), 2.56-2.48 (m, 1H), 2.03-1.98 (m, 2H), 1.78-1.71 (m, 1H), 1.14 (s, 3H), 1.11 (s, 3H), 0.98 (s, 3H). ¹³C NMR (126 MHz, CDCl_3) δ 176.8, 168.5, 160.8, 91.18, 55.58, 55.0, 30.50, 28.8, 16.5, 16.5, 9.6.

5.2. *N*-(hydroxymethyl)benzamide(II) Intermediate Reacts Cleanly on the Anode to Form Product



(using ElectraSyn 2.0). The ElectraSyn vial (10 mL volume) was charged with a magnetic stir bar, amide (0.2 mmol, 1.0 equiv.) and H_2O (36 μL , 10 equiv.) $n\text{Bu}_4\text{NBF}_4$ (0.5 mmol), CH_3CN (5 mL)

(Liquid substrate was added after solvent). The graphite plate anode and Pt plate cathode were adapted on the ElectraSyn vial cap and the vial cap was screwed onto the vial tightly. An argon filled balloon was adapted through the cap to bubble 3 minutes and then maintain an argon atmosphere. The vial was adapted onto the vial holder of ElectraSyn 2.0. Parameters were set: “new experiment” at constant current to 5.0 mA, selecting “No” for “use of reference electrode” and adjusting the reaction time “5 h” The reaction mixture was quenched by removing from the electricity. The reaction mixture was concentrated under reduced pressure, the residue was purified by flash column chromatography on silica gel (1:20 ethyl acetate/petroleum ether) to afford the white solid title compound (24.5 mg, 82%).

5.3. The Cyclic Voltammetry of **1b**

Cyclic voltammograms were collected using a VersaSTAT 3 Potentiostat Galvanostat from Princeton Applied Research. Samples were prepared with 0.15 mmol of substrate in 5 mL of 0.1 M tetra-*n*-butylammoniumhexafluorophosphate in dry acetonitrile. The samples were bubbled with argon for 10 min to avoid trace amount of O₂. Measurements were conducted using glassy carbon working electrode, platinum wire counter electrode, and 3.5 M NaCl silver-silver chloride reference electrode in a scan rate of 0.1 V/s.

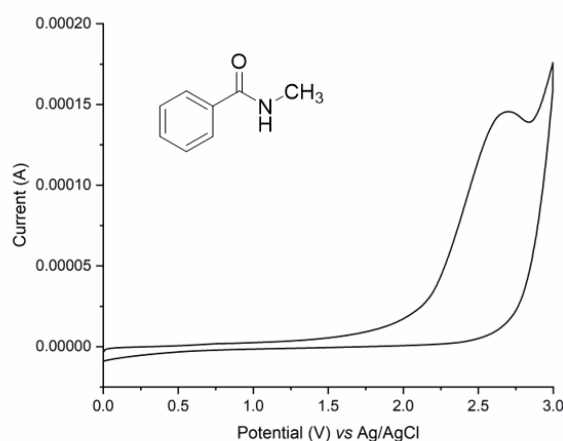
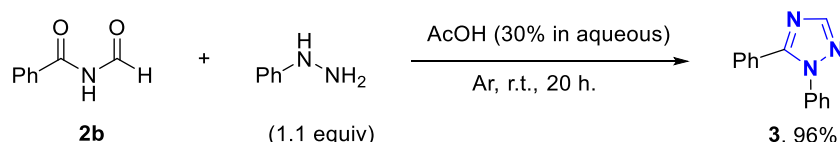


Figure 2. Cyclic voltammogram of **1b** (0.15 mmol) in an electrolyte of *n*Bu₄NBF₄ (0.1 M) in MeCN (5.0 mL). $E_{p/2} = 2.35$ V (vs Ag/AgCl)

6. Application:

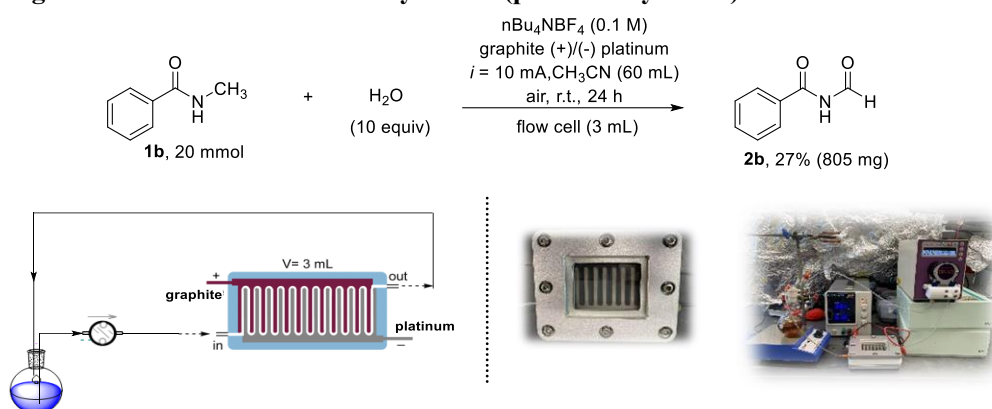
6.1 Synthesis of 1*H*-1, 2, 4-triazole



Two-necked round-bottom glass bottle was charged with a magnetic stir bar, *N*-formylbenzamide **2b** (0.2 mmol, 1.0 equiv.), phenylhydrazine (22 μ L, 1.1 equiv.) and 5 mL AcOH (30% in aqueous) (Liquid substrate was added after solvent). An argon filled balloon was adapted through the cap to bubble 3 minutes and then maintain an argon atmosphere. The reaction mixture was proceed until

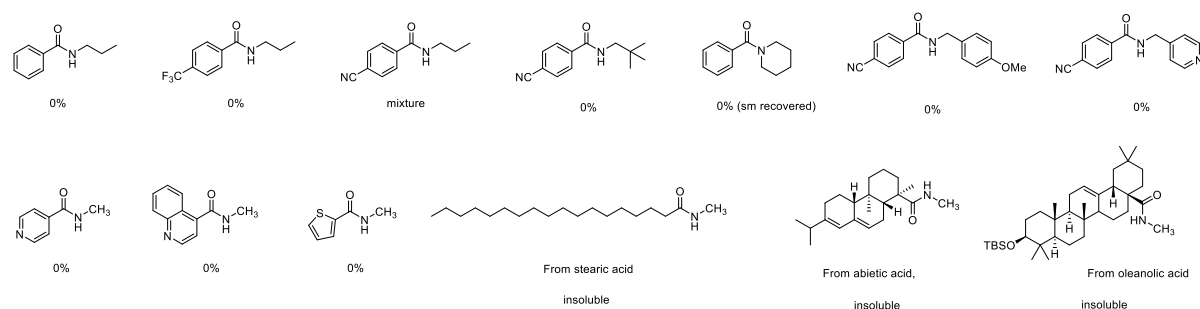
TLC indicating reaction complete. The reaction was then quenched with H₂O and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over Na₂SO₄, and concentrated under reduced pressure, the residue was purified by flash column chromatography on silica gel to afford the title compound **3** (42.5 mg, 96%). ¹H NMR (400 MHz, CDCl₃) δ 7.33-7.39 (m, 5H), 7.43-7.44 (m, 3H), 7.49-7.51 (m, 2H), 8.12 (s, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 125.4, 127.4, 128.6, 128.9, 129.0, 129.4, 130.2, 138.1, 151.2, 153.7

6.2 Large scale in the circulated flow synthesis (preliminary result)



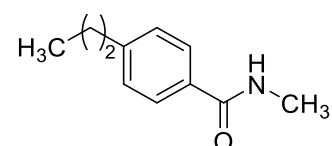
First, assembled and installed the flow electrochemistry device, the anode as graphite electrode, cathode as platinum electrode and the cell volume was 3 mL. Second, **1b** (20 mmol), H₂O (10 equiv), n-Bu₄NBF₄ (0.1 M) were dissolved in CH₃CN (60 mL). The reaction mixture was pumped into the flow cell and electrolyzed at a constant current of 10 mA at room temperature. The flow rate was 10 mL/min and working 24 h. Evaporate the reaction mixture, purified by flash column chromatography, got the **2b** (805mg) in 27% yield.

7. Unsuccessful Substrates



8. Analytical Data of Isolated Compounds

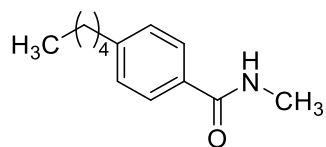
8.1. Analytical Data of Substrates



4-ethyl-N-methylbenzamide (**1o**)

Following the general procedure A to afford product **1o** (797mg, 90 %) as white solid.

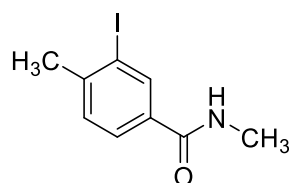
^1H NMR (400 MHz, CDCl_3) δ 7.69 (d, $J = 8.0$ Hz, 2H), 7.14 (d, $J = 8.0$ Hz, 2H), 6.93 (br, 1H), 2.92 (d, $J = 8.0$ Hz, 3H), 2.57 (d, $J = 8.0$ Hz, 2H), 1.65-1.55 (m, 2H), 0.90 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 168.3, 146.1, 131.9, 128.3, 126.8, 37.7, 26.6, 24.1, 13.6. APCI HRMS: Found: m/z 176.1086. Calcd for $\text{C}_{11}\text{H}_{14}\text{NO}$: (M^+) 176.1081. APCI HRMS: Found: m/z 176.1086. Calcd for $\text{C}_{11}\text{H}_{14}\text{NO}$: (M^+) 176.1081.



4-ethyl-N-methylbenzamide (1q)

Following the general procedure A to afford product **1q** (974 mg, 95%) as white solid.

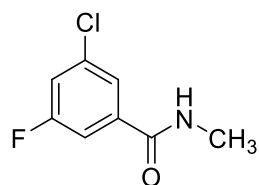
^1H NMR (400 MHz, CDCl_3) δ 7.67 (d, $J = 8.0$ Hz, 2H), 7.19 (d, $J = 8.0$ Hz, 2H), 6.47 (br, 1H), 2.97 (d, $J = 4.0$ Hz, 3H), 2.61 (t, $J = 8.0$ Hz, 2H), 1.63-1.56 (m, 2H), 1.34-1.26 (m, 4H), 0.87 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 168.3, 146.6, 131.9, 128.5, 126.8, 35.7, 31.3, 30.8, 26.7, 22.4, 13.9. APCI HRMS: Found: m/z 204.1402. Calcd for $\text{C}_{13}\text{H}_{18}\text{NO}$: (M^+) 204.1394.



3-iodo-N,4-dimethylbenzamide (1r)

Following the general procedure A to afford product **1r** (1200mg, 88%) as white solid.

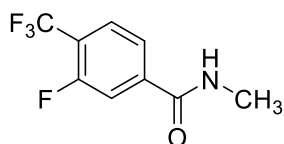
^1H NMR (400 MHz, CDCl_3) δ 8.18 (d, 1H), 7.62 (dd, $J = 4.0, 8.0$ Hz, 1H), 7.23 (d, $J = 8.0$ Hz, 1H), 6.53 (br, 1H), 2.97 (d, $J = 4.0$ Hz, 3H), 2.43 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.6, 144.9, 137.4, 133.7, 129.57, 126.67, 100.87, 28.1, 26.8. APCI HRMS: Found: m/z xx. Calcd for $\text{C}_9\text{H}_{10}\text{INO}$: (M^+) 274.9807.



3-chloro-5-fluoro-N-methylbenzamide (1t)

Following the general procedure A to afford product **1t** (851mg, 91%) as white solid.

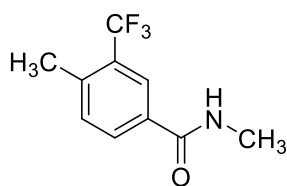
^1H NMR (400 MHz, CDCl_3) δ 7.60-7.56 (m, 1H), 7.08 (dd, $J = 4.0, 12.0$ Hz, 1H), 6.97 (dt, $J = 4.0, 8.0$ Hz, 1H), 6.52 (br, 1H), 2.95 (d, $J = 4.0$ Hz, 3H). ^{19}F NMR (276 MHz, CDCl_3) δ -108.09-(108.11) (m, 1F). ^{13}C NMR (126 MHz, CDCl_3) δ 166.3, 163.0 (d, $J = 253.7$ Hz), 131.8 (d, $J = 10.2$ Hz), 131.7 (d, $J = 9.2$ Hz), 131.3 (d, $J = 3.4$ Hz), 117.4 (d, $J = 24.9$ Hz), 114.3 (d, $J = 21.1$ Hz), 26.7. EI HRMS: Found: m/z 186.0114. Calcd for $\text{C}_8\text{H}_6\text{ClFNO}$: (M^+) 186.0116.



3-fluoro-N-methyl-4-(trifluoromethyl)benzamide (1u)

Following the general procedure A to afford product **1u** (840mg, 80 %) as white solid.

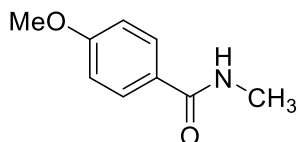
^1H NMR (400 MHz, CDCl_3) δ 8.04 (dd, $J = 4.0, 8.0$ Hz, 1H), 8.01-7.97 (m, 1H), 7.20 (t, $J = 8.0$ Hz, 1H), 7.09 (br, 1H), 2.97 (d, $J = 4.0$ Hz, 3H). ^{19}F NMR (276 MHz, CDCl_3) δ -61.68 (d, $J = 8.3$ Hz, 3F), -109.95-(-110.06) (m, 1F). ^{13}C NMR (126 MHz, CDCl_3) δ 26.9, 117.1 (q, $J = 20.9$ Hz), 122.1 (d, $J = 272.6$ Hz), 126.4, 130.9 (d, $J = 3.4$ Hz), 133.41 – 132.61 (m), 160.3, 162.4 (s), 166.1. EI HRMS: Found: m/z 220.0379. Calcd for $\text{C}_9\text{H}_6\text{F}_4\text{NO}$: (M^+) 220.038.



N, 4-dimethyl-3-(trifluoromethyl)benzamide (1v)

Following the general procedure B to afford product **1v** (890mg, 82%) as white solid.

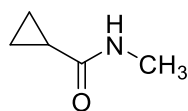
^1H NMR (400 MHz, CDCl_3) δ 7.99 (br, 1H), 7.80 (dd, $J = 4.0, 8.0$ Hz, 1H), 7.27 (d, $J = 8.0$, 1H), 6.93 (br, 1H), 2.97 (d, $J = 4.0$ Hz, 3H), 2.47 (q, $J = 4.0$ Hz, 3H). ^{19}F NMR (276 MHz, CDCl_3) δ -61.94 (s, 3F) ^{13}C NMR (126 MHz, CDCl_3) δ 167.1, 140.11, 132.3, 132.1, 123.0, 129.0 (q, $J = 30.1$ Hz), 124.5 (q, $J = 5.6$ Hz), 124.0 (q, $J = 274.5$ Hz), 26.8, 19.3. APCI HRMS: Found: m/z 216.0654. Calcd for $\text{C}_{10}\text{H}_{10}\text{F}_3\text{NO}$: (M^+) 216.0642.



4-methoxy-N-methylbenzamide (1w)

Following the general procedure A to afford product **1w** (800mg, 97%) as yellowish solid.

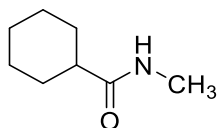
^1H NMR (400 MHz, CDCl_3) δ 7.74-7.71 (m, 2H), 6.91 -6.87 (m, 2H), 6.30 (br, 1H), 3.82 (s, 3H), 2.97 (d, $J = 8.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 167.8, 162.0, 128.6, 126.9, 113.6, 55.3, 26.7. APCI HRMS: Found: m/z 164.072. Calcd for $\text{C}_9\text{H}_{10}\text{NO}_2$: (M^+) 164.0717.



N-methylcyclopropanecarboxamide (1z)

Following the general procedure A to afford product **1z** (475, 96%) as yellowish oil.

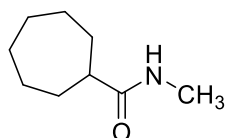
^1H NMR (400 MHz, CDCl_3) δ 5.79 (br, 1H), 2.82 (d, $J = 4.0$ Hz, 3H), 1.36-1.30 (m, 1H), 0.96-0.93 (m, 2H), 0.73-0.68 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 174.2, 26.5, 14.6, 6.9. APCI HRMS: Found: m/z 98.0613. Calcd for $\text{C}_5\text{H}_8\text{NO}$: (M^+) 98.0611.



N-methylcyclohexanecarboxamide (1ac)

Following the general procedure A to afford product **1ac** (691mg, 98%) as white solid

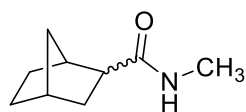
¹H NMR (400 MHz, CDCl₃) δ 5.87 (br, 1H), 2.80 (d, *J* = 4.0 Hz, 3H), 2.13-2.06 (m, 2H), 1.89-1.82 (m, 2H), 1.81-1.75 (m, 2H), 1.70-1.64 (m, 1H), 1.48-1.38 (m, 2H), 1.31-1.21 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 176.8, 45.4, 29.6, 29.3, 26.1, 25.7. EI HRMS: Found: *m/z* 141.1146. Calcd for C₈H₁₅NO: (M⁺) 141.1148.



N-methylcycloheptanecarboxamide (1ad)

Following the general procedure A to afford product **1ad** (736mg, 95%) as white solid

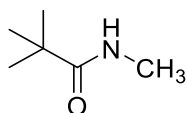
¹H NMR (400 MHz, CDCl₃) δ 5.45 (br, 1H), 2.78 (d, *J* = 4.0 Hz, 3H), 2.24-2.17 (m, 1H), 1.90-1.83 (m, 2H), 1.79-1.73 (m, 2H), 1.69-1.60 (m, 2H), 1.58-1.50 (m, 4H), 1.48-1.39 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 177.9, 47.6, 31.7, 28.1, 26.6, 26.2. APCI HRMS: Found: *m/z* 155.1312. Calcd for C₉H₁₇NO: (M⁺) 155.1310.



(1S, 4R)-N-methylbicyclo[2.2.1]heptane-2-carboxamide (dr = 1:1.4) (1ae)

Following the general procedure B to afford product **1ae** (627mg, 82%) as white solid

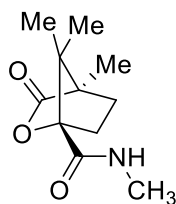
¹H NMR (400 MHz, CDCl₃) δ 5.6 (br, 2.4H), 2.81 (d, *J* = 4.0 Hz, 4.2H), 2.79 (d, *J* = 8.0 Hz, 3H), 2.65-2.59 (m, 1.4H), 2.42-2.36 (m, 2.4H), 2.31-2.25 (m, 2.4H), 2.11 (dd, *J* = 4.0, 8.0 Hz, 1H), 1.88-1.82 (m, 1H), 1.64-1.30 (m, 15.8H), 1.20-1.13 (m, 2.4H). ¹³C NMR (126 MHz, CDCl₃) δ 176.5, 174.7, 47.9, 47.1, 41.4, 40.9, 40.4, 37.0, 36.5, 35.9, 34.4, 31.5, 29.8, 29.2, 28.6, 26.3, 26.3, 24.3. APCI HRMS: Found: *m/z* 153.1153. Calcd for C₉H₁₅NO: (M⁺) 153.1154.



N-methylpivalamide (1af)

Following the general procedure B to afford product **1af** (518mg, 90%) as yellowish oil.

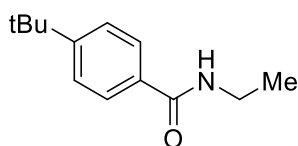
¹H NMR (400 MHz, CDCl₃) δ 5.70 (br, 1H), 2.79 (d, *J* = 8.0 Hz, 3H), 1.18 (s, 9H). ¹³C NMR (126 MHz, CDCl₃) δ 179.1, 29.7, 27.6, 26.5. EI HRMS: Found: *m/z* 115.0993. Calcd for C₆H₁₃NO: (M⁺) 115.0992.



(1S,4R)-N,4,7,7-tetramethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxamide (1ah)

Following the general procedure B to afford product **1ah** (918mg, 87%) as white solid

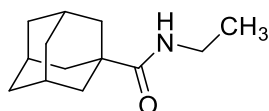
^1H NMR (400 MHz, CDCl_3) δ 6.45 (br, 1H), 2.87 (d, $J = 8.0$ Hz, 3H), 2.57-2.49 (m, 1H), 1.97-1.85 (m, 2H), 1.71-1.65 (m, 1H), 1.11 (s, 3H), 1.10 (s, 3H), 0.89 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 178.2, 167.5, 92.7, 55.28 53.8, 30.3, 29.0, 25.7, 16.7, 16.5, 9.70. APCI HRMS: Found: m/z 210.1144. Calcd for $\text{C}_{11}\text{H}_{16}\text{NO}_3$: (M^+) 210.1136.



4-(tert-butyl)-N-ethylbenzamide (1aj)

Following the general procedure A to afford product **1aj** (953mg, 93%) as white solid

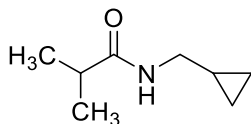
^1H NMR (400 MHz, CDCl_3) δ 7.70 (dt, $J = 4.0, 8.0$ Hz, 2H), 7.42 (dt, $J = 4.0, 8.0$ Hz, 2H), 6.23 (br, 1H), 3.51-3.44 (m, 2H), 1.32 (s, 9H), 1.23 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 167.4, 154.7, 131.9, 126.6, 125.4, 34.8, 31.1, 14.9. APCI HRMS: Found: m/z 205.1465.. Calcd for $\text{C}_{13}\text{H}_{19}\text{NO}$: (M^+) 205.1467.



(3r,5r,7r)-N-ethyladamantane-1-carboxamide (1am)

Following the general procedure A to afford product **1am** (900mg, 87%) as white solid

^1H NMR (400 MHz, CDCl_3) δ 5.65 (br, 1H), 3.25-3.18 (m, 2H), 1.98 (s, 3H), 1.80 (d, $J = 4.0$ Hz, 6H), 1.70-1.62 (m, 6H), 1.07 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 177.7, 40.38, 39.1, 36.48, 34.08, 28.08, 14.8. APCI HRMS: Found: m/z 207.1620. Calcd for $\text{C}_{13}\text{H}_{21}\text{NO}$: (M^+) 207.1623.

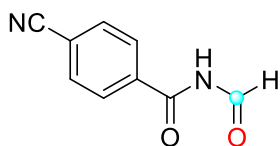


N-(cyclopropylmethyl)isobutyramide (1an)

Following the general procedure A to afford product **1an** (564mg, 80%) as white solid

^1H NMR (400 MHz, CDCl_3) δ 5.59 (br, 1H), 3.20 (dd, $J = 4.0, 8.0$ Hz, 2H), 2.39-2.29 (m, 1H), 1.16 (s, 3H), 1.14 (s, 3H), 0.98-0.88 (m, 1H), 0.51-0.47 (m, 2H), 0.19 (q, $J = 8.0$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 176.8, 44.2, 35.7, 19.6, 10.7, 3.2.. APCI HRMS: Found: m/z xx. Calcd for xx.

8.2. Analytical Data of product



4-cyano-N-formylbenzamide (2a)

Following the typical procedure, the title compound (25.5 mg, white solid) was obtained in 73% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.52 (br, 1H), 9.38 (d, $J = 12.0$ Hz, 1H), 8.06 (dt, $J = 8.0, 4.0$ Hz, 2H), 7.86 (dt, $J = 12.0, 2.0$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 164.9, 163.5, 150.8, 132.9, 128.5, 128.0, 117.4. APCI HRMS: Found: m/z 173.0354. Calcd for $\text{C}_9\text{H}_5\text{N}_2\text{O}_2$: (M^+) 173.0357.

Table 1. Crystal data and structure refinement for M266.

Identification code	M266	
Empirical formula	$\text{C}_9\text{H}_6\text{N}_2\text{O}_2$	
Formula weight	174.16	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	$a = 3.7781(2)$ Å	$\alpha = 94.101(2)^\circ$.
	$b = 7.5804(3)$ Å	$\beta = 93.691(2)^\circ$.
	$c = 14.7776(6)$ Å	$\gamma = 96.948(2)^\circ$.
Volume	$417.90(3)$ Å ³	
Z	2	
Density (calculated)	1.384 Mg/m ³	
Absorption coefficient	0.101 mm ⁻¹	
F(000)	180	
Crystal size	0.188 x 0.117 x 0.099 mm ³	
Theta range for data collection	3.723 to 29.553°.	
Index ranges	$-5 \leq h \leq 5$, $-10 \leq k \leq 10$, $-17 \leq l \leq 20$	
Reflections collected	11723	
Independent reflections	2329 [$R(\text{int}) = 0.0421$]	
Completeness to $\theta = 25.242^\circ$	99.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7459 and 0.7132	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2329 / 0 / 122	
Goodness-of-fit on F^2	1.055	
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0385$, $wR_2 = 0.1091$	
R indices (all data)	$R_1 = 0.0419$, $wR_2 = 0.1135$	

Extinction coefficient	n/a
Largest diff. peak and hole	0.449 and -0.196 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for M266. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	2803(2)	1104(1)	5826(1)	23(1)
O(2)	2254(2)	4745(1)	4049(1)	21(1)
N(1)	3737(2)	2062(1)	4427(1)	18(1)
N(2)	9227(3)	2226(1)	-452(1)	31(1)
C(1)	2489(2)	2214(1)	5278(1)	18(1)
C(2)	3462(2)	3374(1)	3828(1)	16(1)
C(3)	4721(2)	3029(1)	2901(1)	17(1)
C(4)	5226(2)	1338(1)	2536(1)	20(1)
C(5)	6372(2)	1116(1)	1665(1)	21(1)
C(6)	7013(2)	2600(1)	1166(1)	21(1)
C(7)	6509(3)	4298(1)	1525(1)	24(1)
C(8)	5354(2)	4503(1)	2393(1)	21(1)
C(9)	8238(3)	2383(1)	264(1)	24(1)

Table 3. Bond lengths [Å] and angles [°] for M266.

O(1)-C(1)	1.2192(11)
O(2)-C(2)	1.2173(10)
N(1)-C(1)	1.3737(11)
N(1)-C(2)	1.3870(11)
N(1)-H(1)	0.892(15)
N(2)-C(9)	1.1477(13)
C(1)-H(1A)	0.9500
C(2)-C(3)	1.4934(12)
C(3)-C(4)	1.3941(12)
C(3)-C(8)	1.3963(12)
C(4)-C(5)	1.3900(12)
C(4)-H(4)	0.9500
C(5)-C(6)	1.3939(13)
C(5)-H(5)	0.9500
C(6)-C(7)	1.3966(13)
C(6)-C(9)	1.4438(12)
C(7)-C(8)	1.3853(12)
C(7)-H(7)	0.9500
C(8)-H(8)	0.9500
C(1)-N(1)-C(2)	121.44(7)
C(1)-N(1)-H(1)	116.9(9)
C(2)-N(1)-H(1)	121.7(9)
O(1)-C(1)-N(1)	122.48(8)
O(1)-C(1)-H(1A)	118.8
N(1)-C(1)-H(1A)	118.8
O(2)-C(2)-N(1)	121.30(8)
O(2)-C(2)-C(3)	121.67(8)
N(1)-C(2)-C(3)	117.03(7)
C(4)-C(3)-C(8)	120.14(8)
C(4)-C(3)-C(2)	123.30(8)
C(8)-C(3)-C(2)	116.56(8)
C(5)-C(4)-C(3)	120.09(8)
C(5)-C(4)-H(4)	120.0
C(3)-C(4)-H(4)	120.0
C(4)-C(5)-C(6)	119.21(8)

C(4)-C(5)-H(5)	120.4
C(6)-C(5)-H(5)	120.4
C(5)-C(6)-C(7)	121.14(8)
C(5)-C(6)-C(9)	119.55(8)
C(7)-C(6)-C(9)	119.31(8)
C(8)-C(7)-C(6)	119.14(8)
C(8)-C(7)-H(7)	120.4
C(6)-C(7)-H(7)	120.4
C(7)-C(8)-C(3)	120.27(8)
C(7)-C(8)-H(8)	119.9
C(3)-C(8)-H(8)	119.9
N(2)-C(9)-C(6)	179.35(11)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for M266. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	31(1)	19(1)	21(1)	4(1)	8(1)	9(1)
O(2)	25(1)	18(1)	22(1)	1(1)	4(1)	9(1)
N(1)	21(1)	15(1)	19(1)	1(1)	5(1)	6(1)
N(2)	33(1)	34(1)	24(1)	1(1)	8(1)	1(1)
C(1)	18(1)	16(1)	19(1)	0(1)	4(1)	4(1)
C(2)	15(1)	16(1)	18(1)	0(1)	1(1)	2(1)
C(3)	15(1)	19(1)	17(1)	0(1)	2(1)	4(1)
C(4)	22(1)	19(1)	18(1)	1(1)	2(1)	4(1)
C(5)	22(1)	22(1)	19(1)	-2(1)	2(1)	5(1)
C(6)	17(1)	27(1)	18(1)	1(1)	2(1)	2(1)
C(7)	26(1)	24(1)	22(1)	5(1)	5(1)	3(1)
C(8)	24(1)	19(1)	22(1)	2(1)	4(1)	4(1)
C(9)	23(1)	28(1)	22(1)	1(1)	3(1)	2(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for M266.

	x	y	z	U(eq)
H(1)	4740(40)	1080(20)	4279(10)	33(3)
H(1A)	1339	3223	5447	21
H(4)	4785	337	2882	23
H(5)	6715	-34	1412	25
H(7)	6953	5300	1179	28
H(8)	4991	5651	2643	26

Table 6. Torsion angles [°] for M266.

C(2)-N(1)-C(1)-O(1)	-177.69(8)
C(1)-N(1)-C(2)-O(2)	3.43(13)
C(1)-N(1)-C(2)-C(3)	-176.74(7)
O(2)-C(2)-C(3)-C(4)	-162.53(9)
N(1)-C(2)-C(3)-C(4)	17.64(12)
O(2)-C(2)-C(3)-C(8)	16.66(12)
N(1)-C(2)-C(3)-C(8)	-163.17(8)
C(8)-C(3)-C(4)-C(5)	0.22(13)
C(2)-C(3)-C(4)-C(5)	179.38(8)
C(3)-C(4)-C(5)-C(6)	0.12(13)
C(4)-C(5)-C(6)-C(7)	-0.25(14)
C(4)-C(5)-C(6)-C(9)	179.41(8)
C(5)-C(6)-C(7)-C(8)	0.04(14)
C(9)-C(6)-C(7)-C(8)	-179.62(8)
C(6)-C(7)-C(8)-C(3)	0.31(14)
C(4)-C(3)-C(8)-C(7)	-0.44(14)
C(2)-C(3)-C(8)-C(7)	-179.66(8)

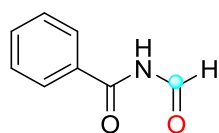
Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for M266 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(1)#1	0.892(15)	1.998(15)	2.8814(10)	170.3(13)
C(1)-H(1A)...O(2)#2	0.95	2.29	3.2266(10)	168.9

Symmetry transformations used to generate equivalent atoms:

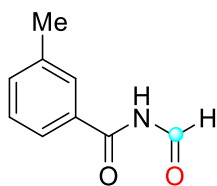
#1 -x+1,-y,-z+1 #2 -x,-y+1,-z+1



N-formylbenzamide (2b)

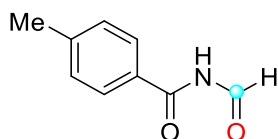
This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (22.4 mg, white solid) was obtained in 75% yield. ¹H NMR

(400 MHz, CDCl₃) δ 9.36 (d, J = 12.0 Hz, 1H), 8.85 (br 1H) 7.90-7.87 (m, 2H), 7.68-7.64 (m, 1H). 7.57-7.52 (m, 2H). APCI HRMS: Found: m/z 148.0406. Calcd for C₈H₆NO₂: (M⁺) 148.0404¹.



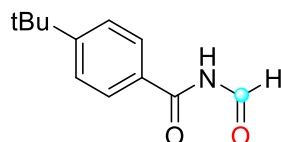
***N*-formyl-4-methylbenzamide (2c)**

This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (16.6 mg, white solid) was obtained in 51% yield. ¹H NMR (400 MHz, CDCl₃) δ 9.36 (d, J = 8.0 Hz, 1H), 8.97 (br, 1H), 7.72 (br, 1H), 7.66 (d, J = 8.0 Hz, 1H), 7.46 (d, J = 8.0 Hz, 1H), 7.42 (t, J = 8.0 Hz, 1H), 2.44 (s, 3H). EI HRMS: Found: m/z 163.0626. Calcd for C₉H₉NO₂: (M⁺) 163.0628².



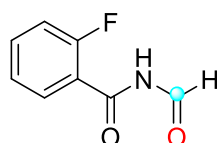
***N*-formyl-3-methylbenzamide (2d)**

This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (13.0 mg, white solid) was obtained in 40% yield. ¹H NMR (400 MHz, CDCl₃) δ 9.36 (d, J = 12.0 Hz, 1H), 8.87(br, 1H) 7.78 (d, J = 8.0 Hz 2H), 7.34 (d, J = 8.0 Hz, 2H), 2.45 (s, 3H). APCI HRMS: Found: m/z 162.0562. Calcd for C₉H₈NO₂: (M⁺) 162.0561³.



4-(tert-butyl)-*N*-formylbenzamide (2e)

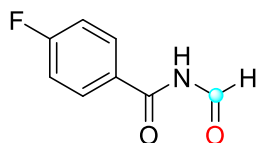
Following the typical procedure, the title compound (30.4 mg, white solid) was obtained in 74% yield. ¹H NMR (400 MHz, CD₃CN) δ 9.45 (br, 1H), 9.38 (d, J = 16.0 Hz, 1H), 7.88 (d, J = 8.0 Hz, 2H) 7.56 (d, J = 12.0 Hz, 2H), 1.35 (s, 9H). ¹³C NMR (126 MHz, CDCl₃) δ 166.4, 164.6, 157.9, 128.1, 128.0, 126.1, 35.2, 31.0. APCI HRMS: Found: m/z 204.1029. Calcd for C₁₂H₁₄NO₂: (M⁺) 204.103.



2-fluoro-*N*-formylbenzamide (2f)

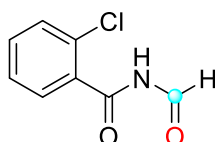
This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (27.1 mg, white solid) was obtained in 81% yield. ¹H NMR (400 MHz, CDCl₃) δ 9.40 (dd, J = 4.0, 8.0 Hz, 1H), 9.06 (br, 1H), 8.14 (dt, J = 4.0, 8.0 Hz, 1H), 7.66-7.61 (m, 1H), 7.35 (dt, J = 4.0, 8.0 Hz, 1H), 7.22 (dd, J = 8.0, 12.0 Hz, 1H). ¹⁹F NMR (376 MHz,

CDCl_3) δ -111.70(-111.59) (m, 1F). EI HRMS: Found: m/z 167.0376. Calcd for $\text{C}_8\text{H}_6\text{FNO}_2$: (M^+) 167.0377⁴.



4-fluoro-*N*-formylbenzamide (2g)

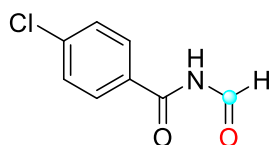
This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (24.4 mg, white solid) was obtained in 73% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.77 (br 1H), 9.04 (d, J = 12.0 Hz, 1H), 8.04-7.99 (m, 2H), 7.26-7.20 (m, 2H). ^{19}F NMR (376 MHz, CDCl_3) δ -103.31(-103.24) (m, 1F). APCI HRMS: Found: m/z 166.0315. Calcd for $\text{C}_8\text{H}_5\text{FNO}_2$: (M^+) 166.031⁵.



2-chloro-*N*-formylbenzamide (2h)

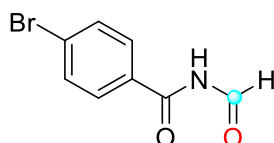
This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (27.9 mg, white solid) was obtained in 76% yield.

^1H NMR (400 MHz, CDCl_3) δ 7.26 (d, J = 12.0 Hz, 1H) 8.90 (br, 1H), 7.35 (t, J = 8.0 Hz, 1H), 7.51-7.39 (m, 3H). EI HRMS: Found: m/z 183.0078. Calcd for $\text{C}_8\text{H}_6\text{ClNO}_2$: (M^+) 183.0082⁴.



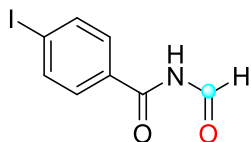
4-chloro-*N*-formylbenzamide (2i)

This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (30.8 mg, white solid) was obtained in 84% yield. ^1H NMR (400 MHz, CD_3CN) δ 9.71 (d, J = 8.0 Hz, 1H), 9.37 (d, J = 8.0 Hz, 1H), 7.92 (dt, J = 4.0, 8.0 Hz, 2H), 7.53(dt, J = 4.0, 8.0 Hz, 2H). EI HRMS: Found: m/z 183.0085. Calcd for $\text{C}_8\text{H}_6\text{ClNO}_2$: (M^+) 183.0082⁴.



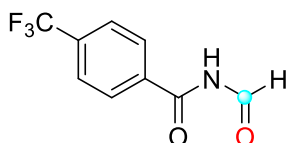
4-bromo-*N*-formylbenzamide (2j)

This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (34.0 mg, white solid) was obtained in 75% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.35(d, J = 8.0 Hz, 1H), 8.98 (br, 1H) 7.77 (dt, J = 8.0, 4.0 Hz, 2H), 7.69 (dt, J = 8.0, 4.0 Hz, 2H). APCI HRMS: Found: m/z 225.951. Calcd for $\text{C}_8\text{H}_5\text{BrNO}_2$: (M^+) 225.9509⁵.



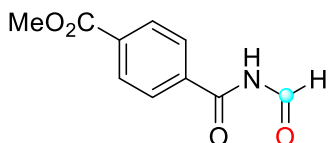
N-formyl-4-iodobenzamide (2k)

Following the typical procedure, the title compound (37.4 mg, white solid) was obtained 68% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.62 (br, 1H), 9.39 (d, $J = 8.0$ Hz, 1H), 8.08 (d, $J = 8.0$ Hz, 2H), 7.82 (d, $J = 8.0$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 165.4, 164.04, 134.3, 128.3, 126.21, 126.18. EI HRMS: Found: m/z 274.9440. Calcd for $\text{C}_8\text{H}_6\text{INO}_2$: (M^+) 274.9443.



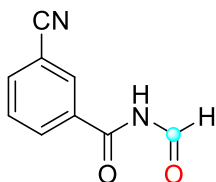
N-formyl-4-(trifluoromethyl)benzamide (2l)

Following the typical procedure, the title compound (33.0 mg, white solid) was obtained in 76% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.35(d, $J = 8.0$ Hz, 1H), 9.21 (br, 1H), 7.91 (dt, $J = 8.0, 4.0$ Hz, 2H), 7.63 (dt, $J = 8.0, 4.0$ Hz, 2H). ^{13}C NMR (126 MHz, d_6 -DMSO) δ 166.7 (s), 164.4 (s), 135.4 (s), 132.8 (q, $J = 32.0$ Hz), 129.3 (s), 125.6 (q, $J = 3.6$ Hz), 123.7 (q, $J = 272.8$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -61.7 (s, 3F). EI HRMS: Found: m/z 217.0348. Calcd for $\text{C}_9\text{H}_6\text{F}_3\text{NO}_2$: (M^+) 217.0345.



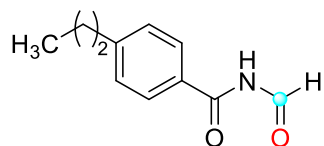
Methyl 4-(formylcarbamoyl)benzoate (2m)

This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (32.3 mg, white solid) was obtained 78% yield. ^1H NMR (400 MHz, CD_3CN) δ 9.62 (d, $J = 8.0$ Hz, 1H), 9.38 (d, $J = 8.0$ Hz, 1H), 8.19 (d, $J = 8.0$ Hz, 2H), 8.01 (d, $J = 8.0$ Hz, 2H), 3.97 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 165.8, 165, 163.7, 134.8, 134.50, 130.20, 128.0, 52.6. APCI HRMS: Found: m/z 206.0466. Calcd for $\text{C}_{10}\text{H}_8\text{NO}_4$: (M^+) 206.0459.



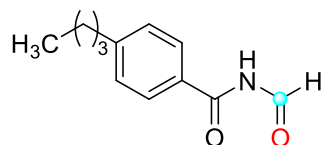
3-cyano-N-formylbenzamide (2n)

Following the typical procedure, the title compound (24.7 mg, white solid) was obtained in 71% yield. ^1H NMR (400 MHz, CD_3CN) δ 9.69 (br, 1H), 9.25 (d, $J = 12.0$ Hz, 1H), 8.27 (t, $J = 4.0$ Hz, 1H), 8.18-8.15 (m, 1H), 7.99 (dt, $J = 4.0, 8.0$ Hz, 1H), 7.73-7.69 (m, 1H). ^{13}C NMR (126 MHz, d_6 -DMSO) δ 166.1, 164.3, 136.6, 133.0, 132.8, 132.2, 130.1, 118.0, 111.9. APCI HRMS: Found: m/z 173.0364. Calcd for $\text{C}_9\text{H}_5\text{N}_2\text{O}_2$: (M^+) 173.0357.



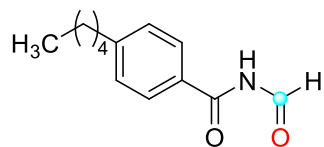
N-formyl-4-propylbenzamide (2o)

Following the typical procedure, the title compound (28.7 mg, white solid) was obtained in 75% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.37 (d, $J = 12.0$ Hz, 1H), 9.16 (d, $J = 12.0$ Hz, 1H), 7.83 (d, $J = 8.0$ Hz, 2H), 7.34 (d, $J = 8.0$ Hz, 2H), 2.67 (t, $J = 8.0$ Hz, 2H), 1.72-1.63 (m, 2H), 0.95 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.2, 163.6, 149.7, 129.3, 128.5, 127.9, 38.0, 24.1, 13.7. APCI HRMS: Found: m/z 190.088. Calcd for $\text{C}_{11}\text{H}_{12}\text{NO}_2$: (M^+) 190.0874.



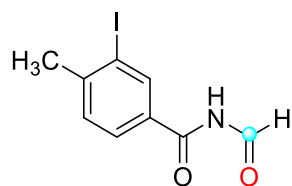
4-butyl-N-formylbenzamide (2p)

Following the typical procedure, the title compound (30.8 mg, white solid) was obtained in 70% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.37 (d, $J = 8.0$ Hz, 1H), 9.29 (d, $J = 12.0$ Hz, 1H), 7.84 (d, $J = 8.0$ Hz, 2H), 7.34 (d, $J = 8.0$ Hz, 2H), 2.71 (t, $J = 8.0$ Hz, 2H), 1.67-1.59 (m, 2H), 1.41-1.32 (m, 2H), 0.94 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.2, 163.7, 149.9, 129.2, 128.4, 127.93, 35.7, 33.1, 22.3, 13.9. APCI HRMS: Found: m/z 204.1033. Calcd for $\text{C}_{12}\text{H}_{14}\text{NO}_2$: (M^+) 204.103.



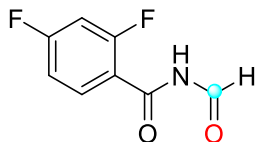
N-formyl-4-pentylbenzamide (2q)

Following the typical procedure, the title compound (23.7 mg, white solid) was obtained 54% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.36 (d, $J = 12.0$ Hz, 1H), 8.90 (d, $J = 8.0$ Hz, 1H), 7.80 (d, $J = 8.0$ Hz, 2H), 7.34 (d, $J = 8.0$ Hz, 2H), 2.69 (t, $J = 8.0$ Hz, 2H), 1.68-1.61 (m, 2H), 1.36-1.30 (m, 4H), 0.89 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.1, 163.3, 150.0, 129.2, 128.5, 127.9, 36.0, 31.4, 30.7, 22.5, 14.0. APCI HRMS: Found: m/z 218.1194. Calcd for $\text{C}_{13}\text{H}_{16}\text{NO}_2$: (M^+) 218.1187.



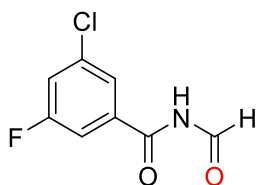
N-formyl-3-iodo-4-methylbenzamide (2r)

Following the typical procedure, the title compound (24.9 mg, white solid) was obtained 43% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.34 (d, $J = 12.0$ Hz, 1H), 8.93 (br, 1H), 8.34 (d, $J = 4.0$ Hz, 1H), 7.75 (dd, $J = 4.0, 8.0$ Hz, 1H), 7.38 (d, $J = 8.0$ Hz, 1H), 2.52 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 164.7, 163.3, 148.3, 138.5, 130.2, 130.1, 127.3, 28.4. APCI HRMS: Found: m/z 288.9603. Calcd for $\text{C}_9\text{H}_8\text{INO}_2$: (M^+) 288.9600.



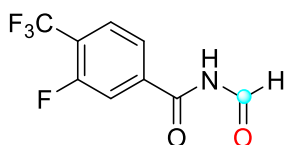
2, 4-difluoro-N-formylbenzamide (2s)

Following the typical procedure, the title compound (24.1 mg, white solid) was obtained in 65% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.39 (dd, $J = 4.0, 8.0$ Hz, 1H), 8.98 (br, 1H), 8.16-8.22 (m, 1H), 7.11-7.06 (m, 1H), 6.96 (ddd, $J = 4.0, 12.0, 16$ Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ . 167.5 (d, $J = 14.3$ Hz), 162.8 (d, $J = 13.1$ Hz), 162.4, 162.1 (d, $J = 3.5$ Hz), 134.3 (dd, $J = 10.6, 2.5$ Hz), 113.4 (dd, $J = 21.6, 3.0$ Hz), 105.0 (dd, $J = 28.1, 26.2$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -98.74(-98.64) (m, 1F), -107.07(-107.02) (m, 1F). APCI HRMS: Found: m/z 184.0221. Calcd for $\text{C}_8\text{H}_4\text{F}_2\text{NO}_2$: (M⁺) 184.0216.



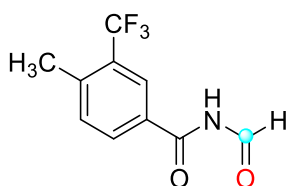
3-chloro-5-fluoro-N-formylbenzamide (2t)

Following the typical procedure, the title compound (31.85 mg, white solid) was obtained 79% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.26 (d, $J = 8.0$ Hz, 1H), 9.07 (br, 1H), 7.84 (dd, $J = 4.0, 8.0$ Hz, 1H), 7.24 (dd, $J = 4.0, 8.0$ Hz, 1H), 7.17-7.12 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 165.5, 164.3 (d, $J = 200.8$ Hz), 162.3, 162.2, 133.1 (d, $J = 9.7$ Hz), 127.9 (d, $J = 3.6$ Hz). 118.5 (d, $J = 25.2$ Hz), 115.3 (d, $J = 21.5$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -103.35 (q, $J = 7.53$ Hz, 1F). APCI HRMS: Found: m/z 199.9926. Calcd for $\text{C}_8\text{H}_4\text{ClFNO}_2$: (M⁺) 199.992.



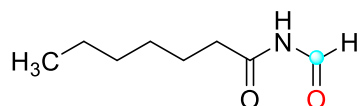
3-fluoro-N-formyl-4-(trifluoromethyl)benzamide (2u)

Following the typical procedure, the title compound (42.8 mg, white solid) was obtained 91% yield. ^1H NMR (400 MHz, CDCl_3) δ 10.54 (d, $J = 8.0$ Hz, 1H), 9.40 (d, $J = 8.0$ Hz, 1H), 8.38 (dd, $J = 4.0, 8.0$ Hz, 1H), 8.30-8.26 (m, 1H), 7.40 (t, $J = 8.0$ Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 118.0 (d, $J = 21.5$ Hz), 119.65 (dd, $J = 34.0, 13.4$ Hz), 121.8 (d, $J = 273.0$ Hz), 127.5 (d, $J = 3.6$ Hz), 128.80 – 127.96 (m), 163.0 (d, $J = 266.3$ Hz), 164.4, 165.1. ^{19}F NMR (376 MHz, CDCl_3) δ -61.7 (d, $J = 11.28$ Hz, 3F), -104.88 (q, $J = 11.28$ Hz, 1F). EI HRMS: Found: m/z 235.0261. Calcd for $\text{C}_9\text{H}_5\text{F}_4\text{NO}_2$: (M⁺) 235.0251.



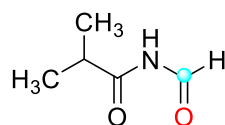
N-formyl-4-methyl-3-(trifluoromethyl)benzamide (2v)

Following the typical procedure, the title compound (37.0 mg, white solid) was obtained in 80% yield. ^1H NMR (400 MHz, CDCl_3) δ 10.2 (d, $J = 8.0$ Hz, 1H), 9.4 (d, $J = 8.0$ Hz, 1H), 8.27 (d, 1H), 8.06 (dd, $J = 8.0$ Hz, 1H), 7.49 (d, $J = 8.0$ Hz, 1H), 2.59 (q, $J = 4.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 165.4 (s), 164.7 (s), 143.5 (s), 132.8 (s), 130.9 (s), 130.1 (q, $J = 30.4$ Hz), 129.0 (s), 126.0 (q, $J = 5.6$ Hz), 123.78 (q, $J = 274.0$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -61.6 (s, 3F). EI HRMS: Found: m/z 231.0498. Calcd for $\text{C}_{10}\text{H}_8\text{NO}_2$: (M^+) 231.0502.



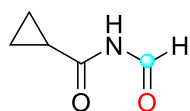
N-formylheptanamide (2x)

Following the typical procedure, the title compound (17.3 mg, white solid) was obtained 55% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.42 (br, 1H), 9.11 (d, $J = 8.0$ Hz, 1H), 2.38 (t, $J = 8.0$ Hz, 2H), 1.70-1.62 (m, 2H), 1.35-1.26 (m, 6H), 0.87 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 173.7, 163.7, 36.6, 31.3, 28.6, 24.1, 22.4, 13.9. APCI HRMS: Found: m/z 156.1035. Calcd for $\text{C}_8\text{H}_{14}\text{NO}_2$: (M^+) 156.103.



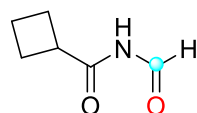
N-formylisobutyramide (2y)

Following the typical procedure, the title compound (16.3 mg, white solid) was obtained 71% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.58 (br, 1H), 9.13 (d, $J = 8.0$ Hz, 1H), 2.60-2.50 (m, 1H), 1.21 (d, $J = 8.0$ Hz, 3H), 1.21 (d, $J = 8.0$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 177.5, 164.2, 35.7, 18.3. APCI HRMS: Found: m/z 114.0563. Calcd for $\text{C}_5\text{H}_8\text{NO}_2$: (M^+) 114.0561.



N-formylcyclopropanecarboxamide (2z)

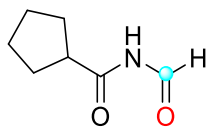
Following the typical procedure, the title compound (14.5 mg, white solid) was obtained 64% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.92 (br, 1H), 9.11 (d, $J = 12.0$ Hz, 1H), 1.65-1.56 (m, 1H), 1.17-1.13 (m, 2H), 1.02-0.98 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 174.9, 163.9, 14.9, 10.1, 8.9. APCI HRMS: Found: m/z 112.0403. Calcd for $\text{C}_5\text{H}_6\text{NO}_2$: (M^+) 112.040



N-formylcyclobutanecarboxamide (2aa)

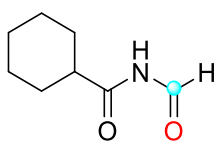
Following the typical procedure, the title compound (16.0 mg, white solid) was obtained in 63% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.11 (d, $J = 12.0$ Hz, 1H), 8.99 (br, 1H), 3.23-3.14 (m, 1H), 2.39-2.32 (m, 2H), 2.30-2.20 (m, 2H), 2.09-2.02 (m, 1H), 1.98-1.87 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 175.1, 163.5, 39.6, 24.5, 18.0. APCI HRMS: Found: m/z 126.0564. Calcd for $\text{C}_6\text{H}_8\text{NO}_2$: (M^+)

126.0561.



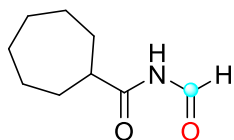
N-formylcyclopentanecarboxamide (2ab)

Following the typical procedure, the title compound (14.4 mg, white solid) was obtained in 51% yield. ¹H NMR (400 MHz, CDCl₃) δ 9.14 (d, *J* = 12.0 Hz, 1H), 8.85 (br, 1H), 2.77-2.69 (m, 1H), 1.98-1.90 (m, 2H), 1.88-1.81 (m, 2H), 1.79-1.70 (m, 2H), 1.68-1.59 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 176.3, 163.0 (s), 45.7, 29.5, 25.9. EI HRMS: Found: *m/z* 141.0783. Calcd for C₇H₁₁NO₂: (M⁺) 141.0784.



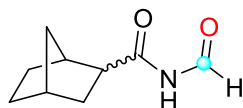
N-formylcyclohexanecarboxamide (2ac)

This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (17.7 mg, white solid) was obtained in 57% yield. ¹H NMR (400 MHz, CDCl₃) δ 9.14 (d, *J* = 12.0 Hz, 1H), 8.88 (br, 1H), 2.30-2.22 (m, 1H), 1.92-1.88 (m, 2H), 1.84-1.80 (m, 2H), 1.52-1.42 (m, 2H), 1.36-1.18 (m, 4H). EI HRMS: Found: *m/z* 155.094. Calcd for C₈H₁₃NO₂: (M⁺) 155.0941⁶.



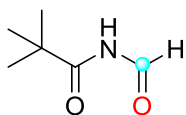
N-formylcycloheptanecarboxamide (2ad)

Following the typical procedure, the title compound (24.4 mg, white solid) was obtained 72% yield. ¹H NMR (400 MHz, CDCl₃) δ 9.13 (d, *J* = 12.0 Hz, 1H), 8.98 (br, 1H), 2.46-2.39 (m, 1H), 1.96-1.90 (m, 2H), 1.80-1.65 (m, 5H), 1.59-1.46 (m, 5H). ¹³C NMR (126 MHz, CDCl₃) δ 177.0, 163.7, 47.0, 30.33, 28.13, 26.3. APCI HRMS: Found: *m/z* 168.1034. Calcd for C₉H₁₄NO₂: (M⁺) 168.103.



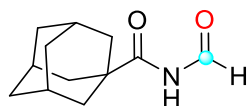
(1S, 4R)-N-formylbicyclo[2.2.1]heptane-2-carboxamide (2ae, dr = 1:2)

Following the typical procedure, the title compound (23.1 mg, white solid) was obtained 69% yield. ¹H NMR (400 MHz, CDCl₃) δ 9.40-9.38 (br 1x1H+2x1H), 9.15 (d, *J* = 8.0 Hz, 1H), 9.10 (d, *J* = 12.0 Hz, 2x1H), 2.81-2.76 (m, 1H), 2.59 (t, *J* = 4.0 Hz, 1H), 2.50 (d, *J* = 4.0 Hz, 2H), 2.35-2.29 (m, 5H), 1.94-1.88 (m, 2H), 1.79-1.74 (m, 1H), 1.66-1.40 (m, 13H), 1.31-1.26 (m, 4H), 1.24-1.18 (m, 4H). ¹³C NMR (126 MHz, CDCl₃) δ 175.7, 174.8, 164.0, 163.9, 48.3, 47.9, 40.9, 40.6, 40.2, 36.9, 36.3, 35.9, 33.0, 30.4, 29.4, 28.9, 28.5, 24.4. APCI HRMS: Found: *m/z* 166.0874. Calcd for C₉H₁₂NO₂: (M⁺) 166.0874.



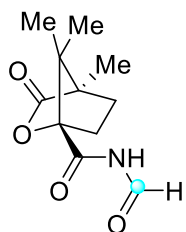
N-formylpivalamide (2af)

Following the typical procedure, the title compound (15.8 mg, white solid) was obtained in 61% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.16 (d, $J = 12.0$ Hz, 1H), 8.66 (br, 1H), 1.26 (s, 9H). ^{13}C NMR (126 MHz, CDCl_3) δ 178.5, 163.5, 29.7, 26.6. APCI HRMS: Found: m/z 128.0715. Calcd for $\text{C}_6\text{H}_{10}\text{NO}_2$: (M^+) 128.0717.



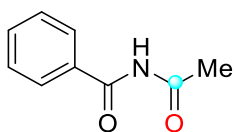
(3r,5r,7r)-N-formyladamantane-1-carboxamide (2ag)

Following the typical procedure, the title compound (24.9 mg, white solid) was obtained 60% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.17 (d, $J = 8.0$ Hz, 1H), 8.58 (br 1H), 2.09-2.10 (m, 3H), 1.88 (d, 6H), 1.70-1.79 (m, 7H). ^{13}C NMR (126 MHz, CDCl_3) δ 177.8, 163.4, 38.7, 38.2, 36.3, 36.1, 27.8, 27.7. EI HRMS: Found: m/z 207.1249 Calcd for $\text{C}_{12}\text{H}_{17}\text{NO}_2$: (M^+) 207.1254.



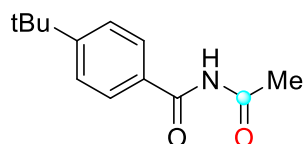
(1S,4R)-N-formyl-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxamide (2ah)

Following the typical procedure, the title compound (33.8 mg, white solid) was obtained in 75% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.18 (d, $J = 8.0$ Hz, 1H), 8.83 (br, 1H), 2.56-2.48 (m, 1H), 2.03-1.98 (m, 2H), 1.78-1.71 (m, 1H), 1.14 (s, 3H), 1.11 (s, 3H), 0.98 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 176.8, 168.5, 160.8, 91.18, 55.58, 55.0, 30.50, 28.8, 16.5, 16.5, 9.6. APCI HRMS: Found: m/z 224.0935. Calcd for $\text{C}_{11}\text{H}_{14}\text{NO}_4$: (M^+) 224.0928.



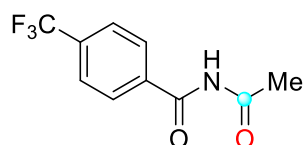
N-acetylbenzamide (2ai)

Following the typical procedure, the title compound (13.4 mg, white solid) was obtained in 41% yield. ^1H NMR (400 MHz, CD_3CN) δ 8.13-8.11 (m, 2H), 7.61-7.57 (m, 1H), 7.55-7.50 (m, 2H), 2.48 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 173.2, 165.5, 133.3, 132.7, 129.1, 127.6, 25.5. EI HRMS: Found: m/z 162.0548. Calcd for $\text{C}_9\text{H}_8\text{NO}_2$: (M^+) 162.055.



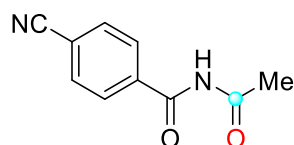
N-acetyl-4-(tert-butyl) benzamide (2aj)

Following the typical procedure, the title compound (18.0 mg, white solid) was obtained in 41% yield. ^1H NMR (400 MHz, CDCl_3) δ 8.58 (br, 1H), 7.9 (dt, $J = 8.0, 4.0$ Hz, 2H), 7.52 (dt, $J = 8.0, 4.0$ Hz, 2H), 2.62 (s, 3H), 1.35 (s, 9H). ^{13}C NMR (126 MHz, CDCl_3) δ 173.2, 165.3, 157.2, 129.7, 127.5, 126.0, 35.2, 31.0, 25.5. EI HRMS: Found: m/z 219.1249. Calcd for $\text{C}_{13}\text{H}_{17}\text{NO}_2$: (M^+) 219.1254.



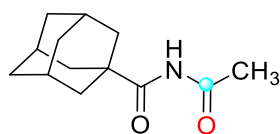
N-acetyl-4-(trifluoromethyl)benzamide (2ak)

This compound is a known compound that has been reported in a previous literature. Following the typical procedure, the title compound (26.4 mg, white solid) was obtained in 57% yield. ^1H NMR (400 MHz, CDCl_3) δ 9.15 (br 1H), 8.02 (d, $J = 8.0$ Hz, 2H), 7.78 (d, $J = 8.0$ Hz, 2H), 2.63 (s, 3H). ^{19}F NMR (376 MHz, CDCl_3) δ -63.2 (s, 3F) EI HRMS: Found: m/z 231.05. Calcd for $\text{C}_{10}\text{H}_8\text{F}_3\text{NO}_2$: (M^+) 231.0502⁷.



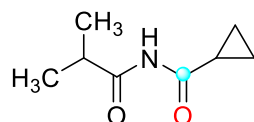
N-acetyl-4-cyanobenzamide (2al)

Following the typical procedure, the title compound (16.2 mg, white solid) was obtained in 43% yield. ^1H NMR (400 MHz, CD_3CN) δ 8.67 (br, 1H), 7.97 (dt, $J = 4.0, 8.0$ Hz, 2H), 7.82 (dt, $J = 4.0, 8.0$ Hz, 2H), 2.62 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 173.5, 164.3, 136.5, 132.7, 128.4, 117.5, 116.7, 25.7. APCI HRMS: Found: m/z 187.0519. Calcd for $\text{C}_{10}\text{H}_7\text{N}_2\text{O}_2$: (M^+) 187.0513.



(3r, 5r, 7r)-N-acetyladamantane-1-carboxamide (2am)

Following the typical procedure, the title compound (27.4 mg, white solid) was obtained 62% yield. ^1H NMR (400 MHz, CDCl_3) δ 8.10 (br 1H), 2.46 (s, 3H), 2.11-2.05 (m, 3H), 1.86 (d, 6H), 1.77-1.67 (m, 6H). ^{13}C NMR (126 MHz, CDCl_3) δ 176.6, 173.5, 41.9, 39.9, 38.6, 38.2, 36.1, 35.7, 27.8, 25.5. EI HRMS: Found: m/z 221.1409. Calcd for $\text{C}_{13}\text{H}_{19}\text{NO}_2$: (M^+) 221.141.



***N*-isobutyrylcyclopropanecarboxamide (2an)**

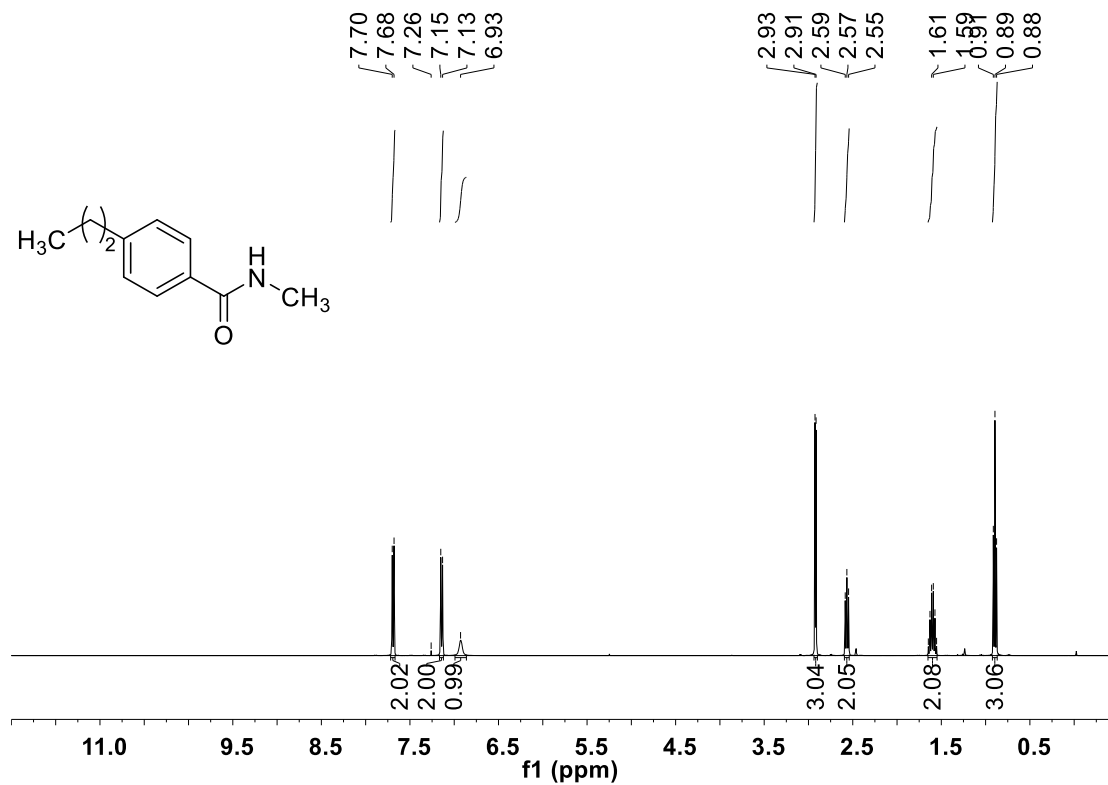
Following the typical procedure, the title compound (18.6mg, white solid) was obtained 60% yield. ¹H NMR (400 MHz, CDCl₃) δ 8.12 (br 1H), 2.90-2.80 (m, 1H), 2.50-2.42 (m, 1H), 1.21 (s, 3H), 1.19 (s, 3H), 1.15-1.11 (m, 2H), 1.00-0.95 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 10.5, 14.8, 18.9, 36.1, 175.4, 177.3). EI HRMS: Found: m/z 155.0950. Calcd for C₈H₁₃NO₂: (M⁺) 155.0946.

9. References

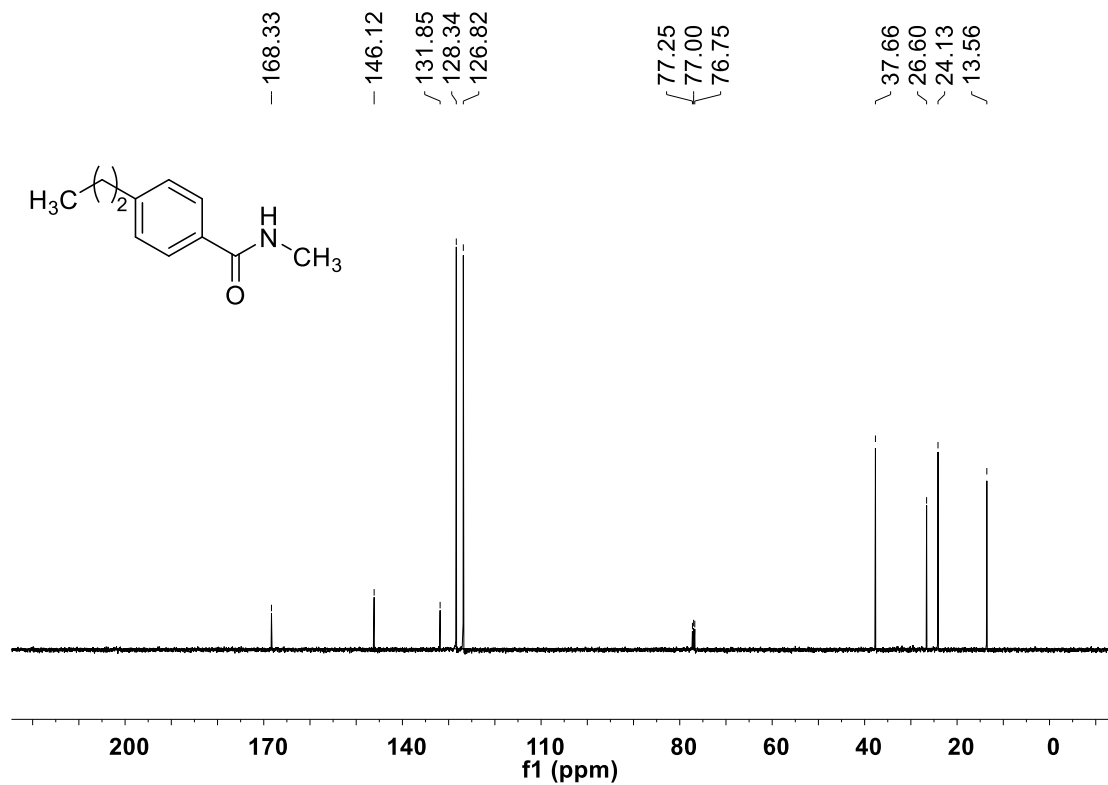
1. Bai, F.; Wang, N.; Bai, Y.; Ma, X.; Gu, C.; Dai, B.; Chen, J., NHPI-Mediated Electrochemical α -Oxygenation of Amides to Benzimides. *The Journal of Organic Chemistry* **2023**, *88* (5), 2985-2998.
2. Aiken, S.; Anozie, K.; de Azevedo, O. D. C. C.; Cowen, L.; Edgar, R. J. L.; Gabbutt, C. D.; Heron, B. M.; Lawrence, P. A.; Mills, A. J.; Rice, C. R.; Urquhart, M. W. J.; Zonidis, D., Expedient synthesis of highly substituted 3,4-dihydro-1,2-oxathiine 2,2-dioxides and 1,2-oxathiine 2,2-dioxides: revisiting sulfene additions to enaminketones. *Org. Biomol. Chem.* **2019**, *17* (44), 9585-9604.
3. Zhang, Q.; Lin, X.-T.; Fukaya, N.; Fujitani, T.; Sato, K.; Choi, J.-C., Selective N-formylation/N-methylation of amines and N-formylation of amides and carbamates with carbon dioxide and hydrosilanes: promotion of the basic counter anions of the zinc catalyst. *Green Chem.* **2020**, *22* (23), 8414-8422.
4. Niu, Z.; Lin, S.; Dong, Z.; Sun, H.; Liang, F.; Zhang, J., Otherwise inert reaction of sulfonamides/carboxamides with formamides via proton transfer-enhanced reactivity. *Org. Biomol. Chem.* **2013**, *11* (15), 2460-2465.
5. Sambaiah, M.; Gudipati, R.; Shiva Kumar, K.; Yennam, S.; Behera, M., An efficient method for the preparation of N-formyl-imide via amidine using propylphosphonic anhydride (T3P®). *Tetrahedron Lett.* **2016**, *57* (3), 403-406.
6. Bhat, S. A.; Bhat, M. Y.; Rather, S. A.; Gani, I.; Bhat, K. A.; Ahmed, Q. N., Iodine and ammonium persulfate mediated activation of DMSO: an approach to N-formylation of amides and synthesis of isatins. *Org. Biomol. Chem.* **2022**, *20* (42), 8197-8202.
7. Cao, M.; Liu, L.; Tang, S.; Peng, Z.; Wang, Y., Palladium-Catalyzed Solvent-Controlled Selective Synthesis of Acyl Isoureas and Imides from Amides, Isocyanides, Alcohols and Carboxylates. *Adv. Synth. Catal.* **2019**, *361* (8), 1887-1895.

10. NMR Spectrum of prepared compounds (^1H NMR; ^{13}C NMR; ^{19}F NMR *etc.*)

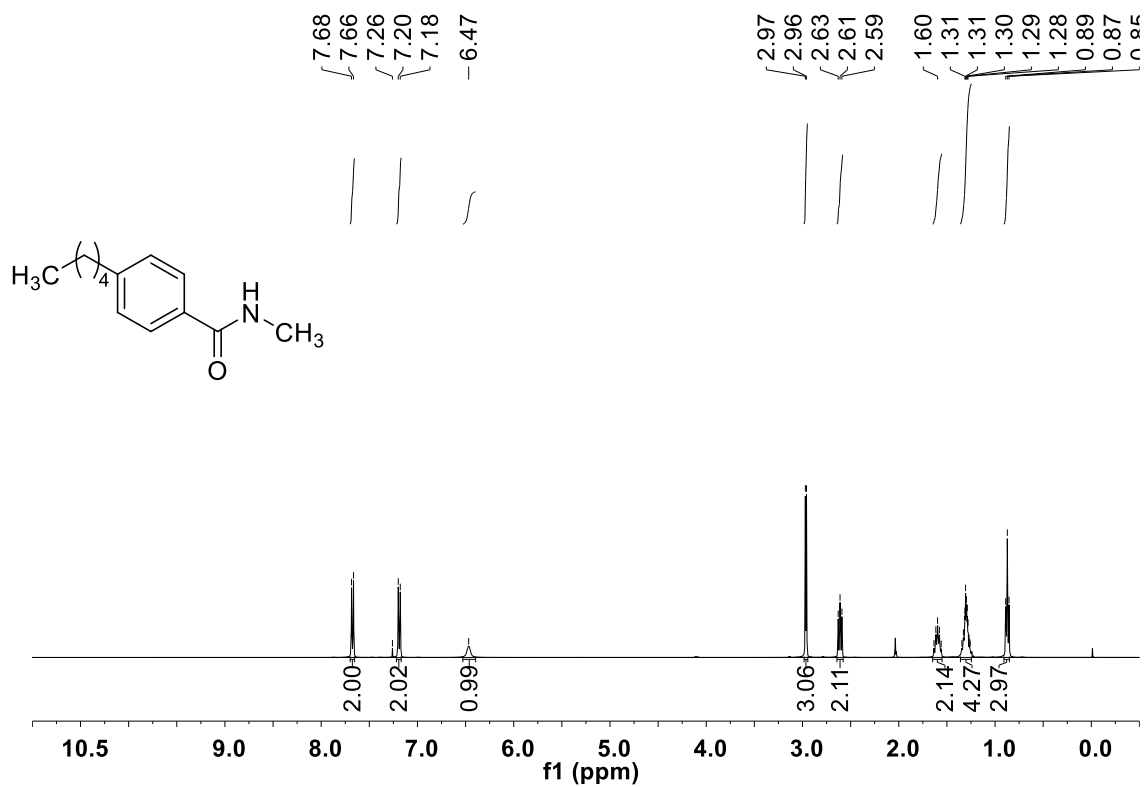
^1H NMR (400 MHz, CDCl_3) spectra for compound **1o**



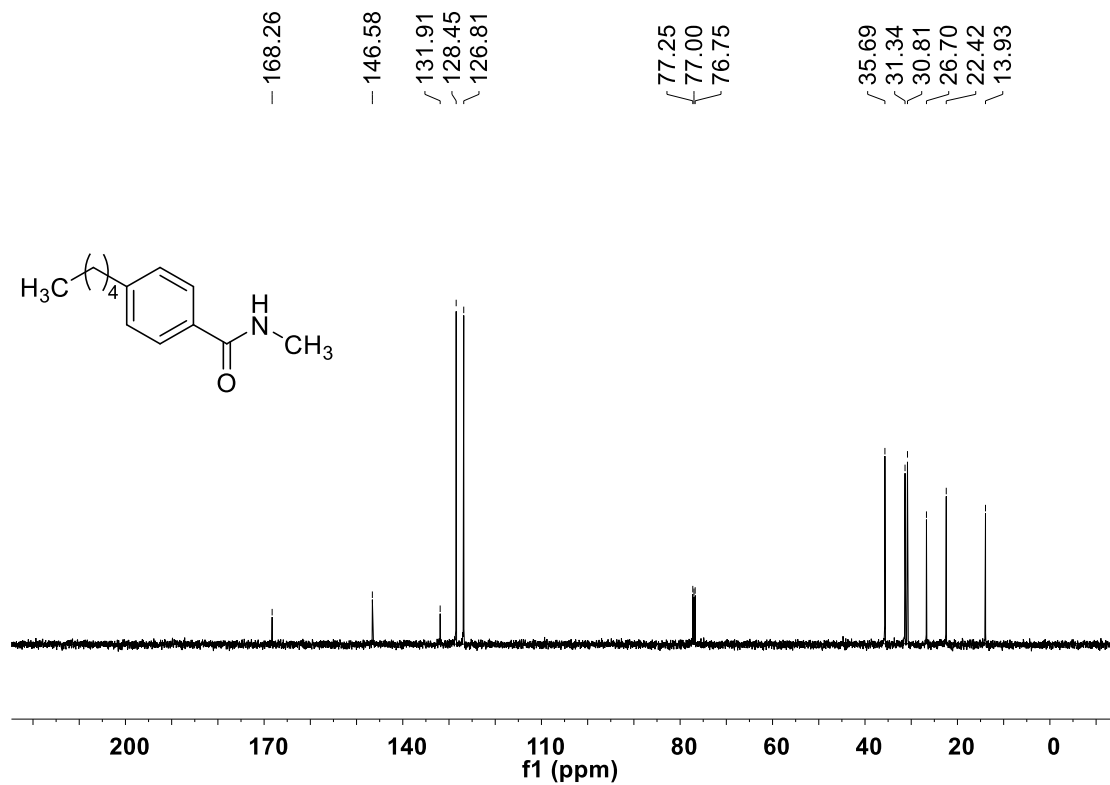
^{13}C NMR (126 MHz, CDCl_3) spectra for compound **1o**



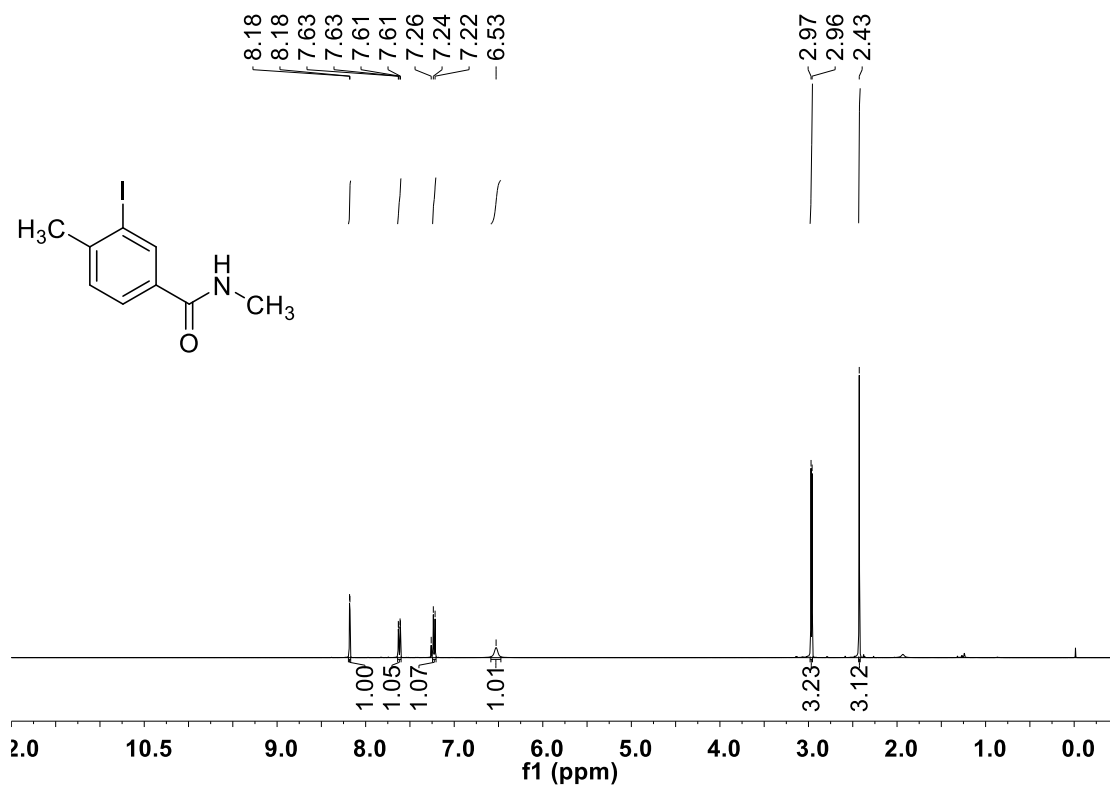
¹H NMR (400 MHz, CDCl₃) spectra for compound **1q**



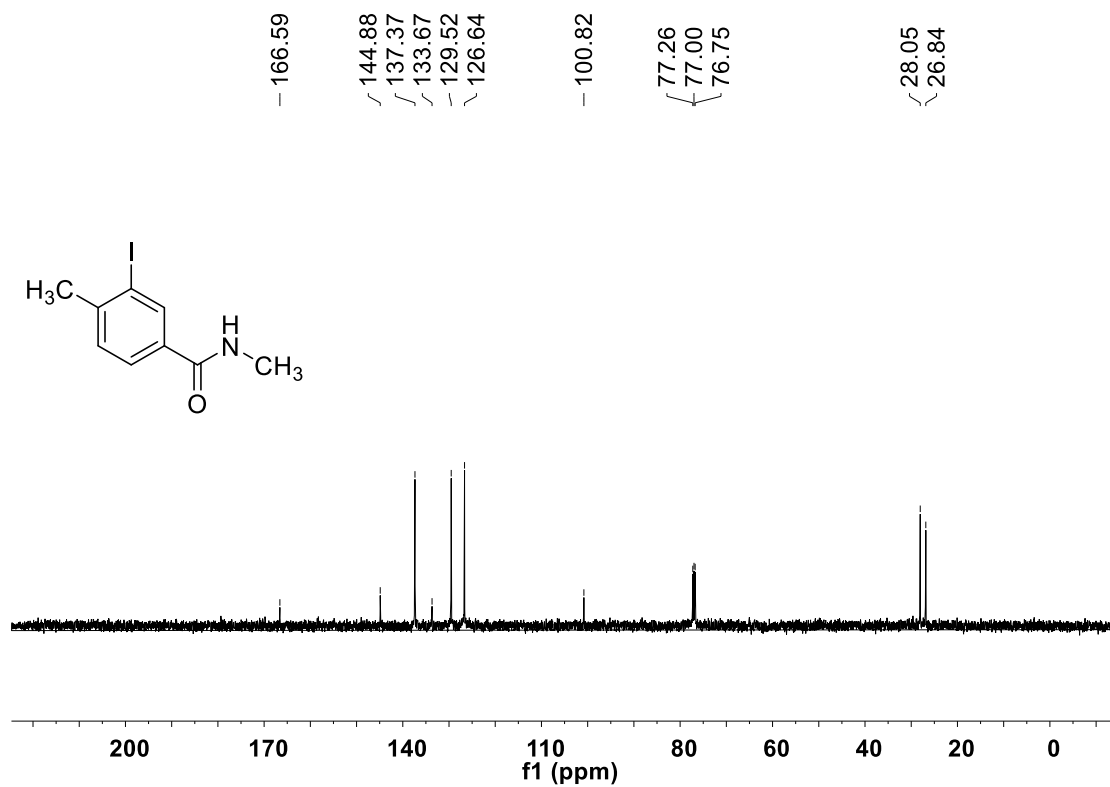
¹³C NMR (126 MHz, CDCl₃) spectra for compound **1q**



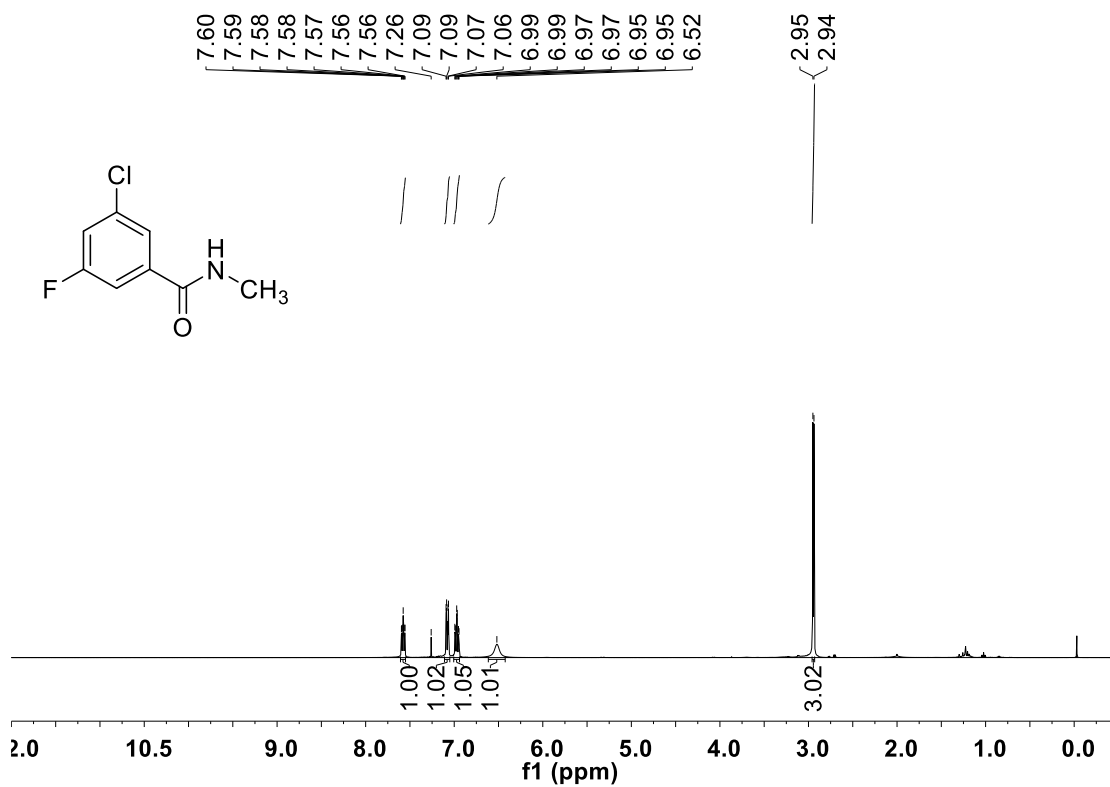
^1H NMR (400 MHz, CDCl_3) spectra for compound **1r**



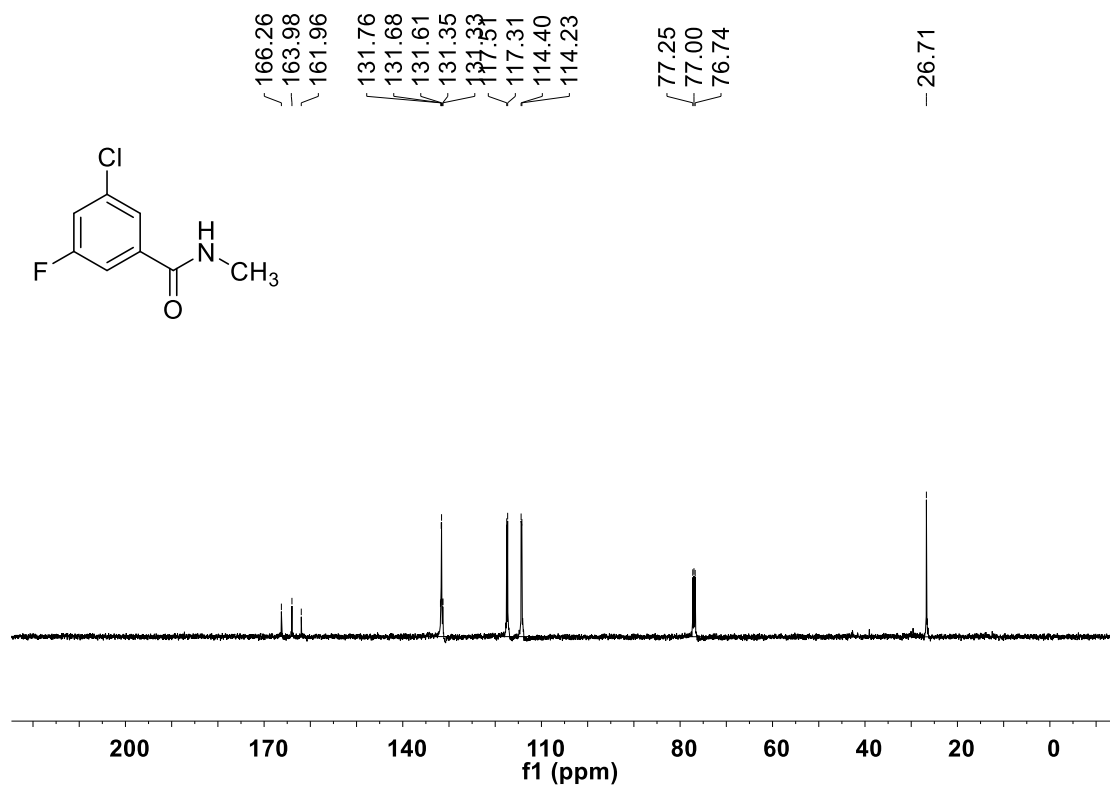
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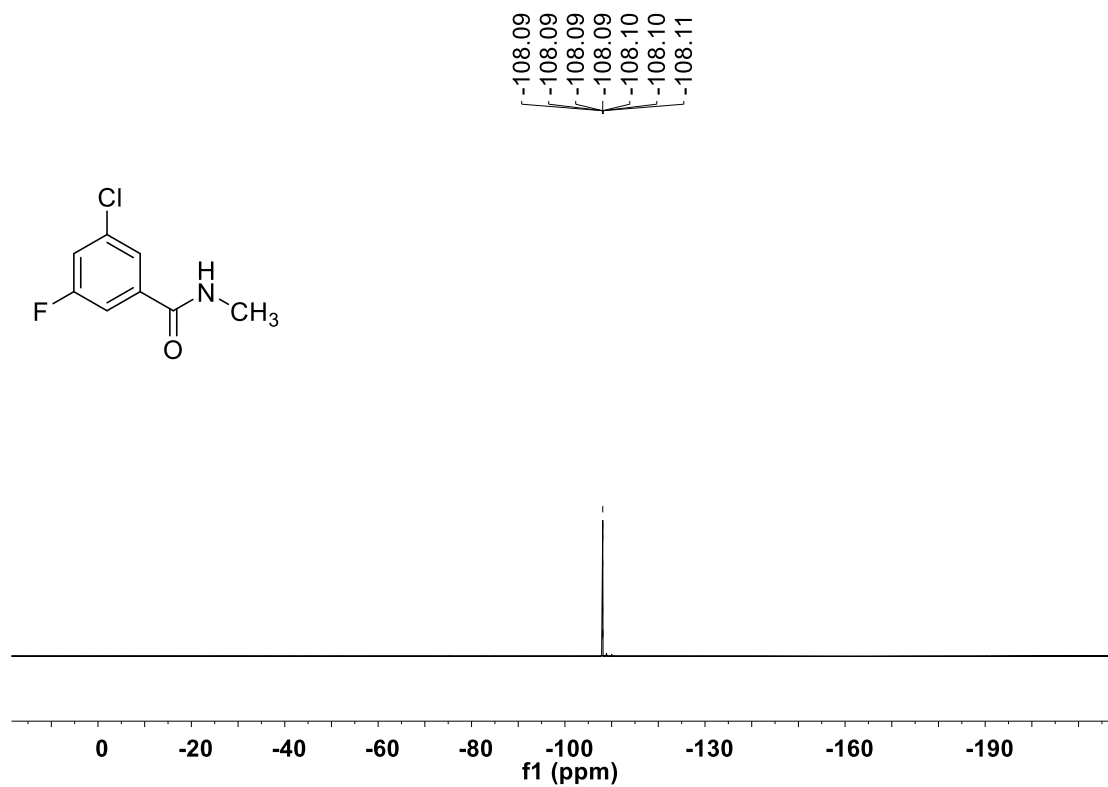
¹H NMR (400 MHz, CDCl₃) spectra for compound **1t**



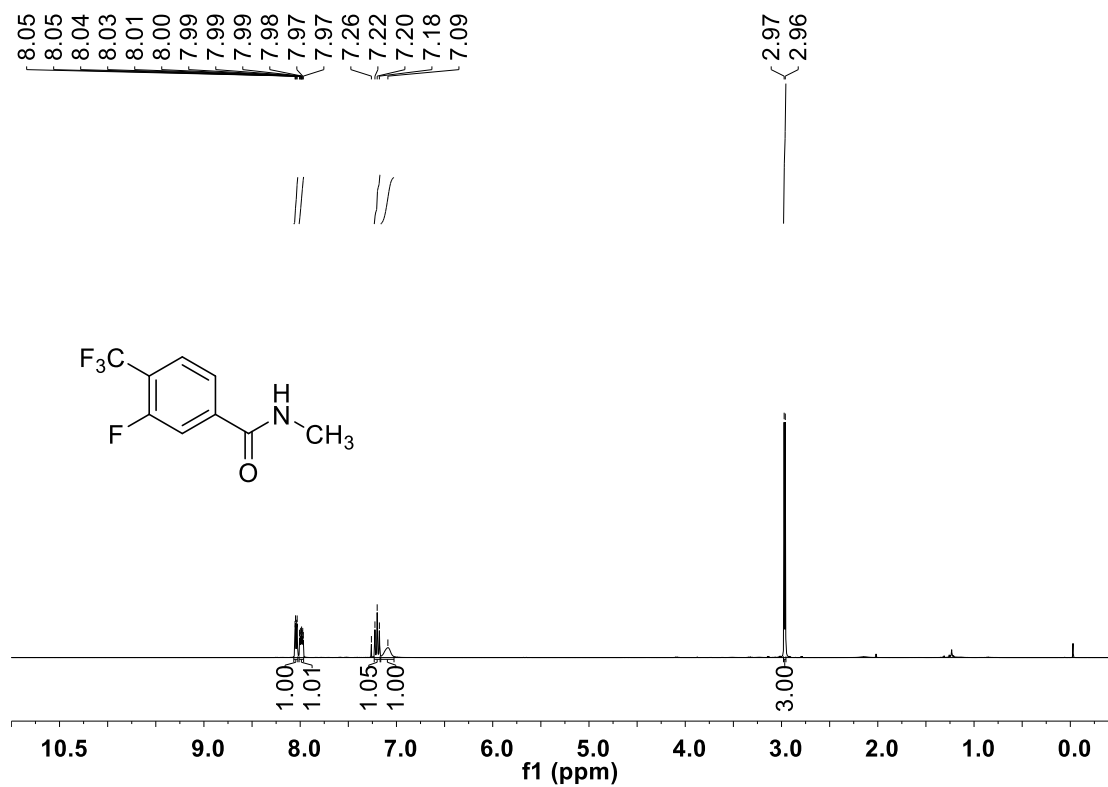
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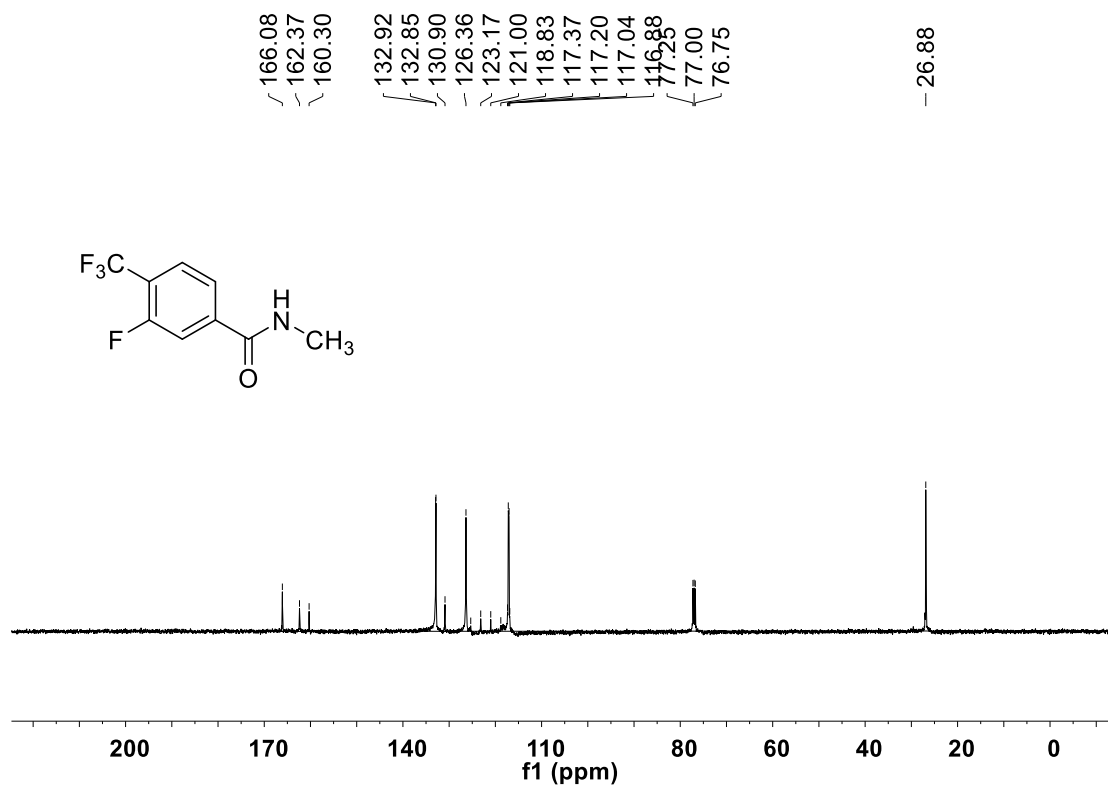
^{19}F NMR (377 MHz, CDCl_3) spectra for compound **1t**



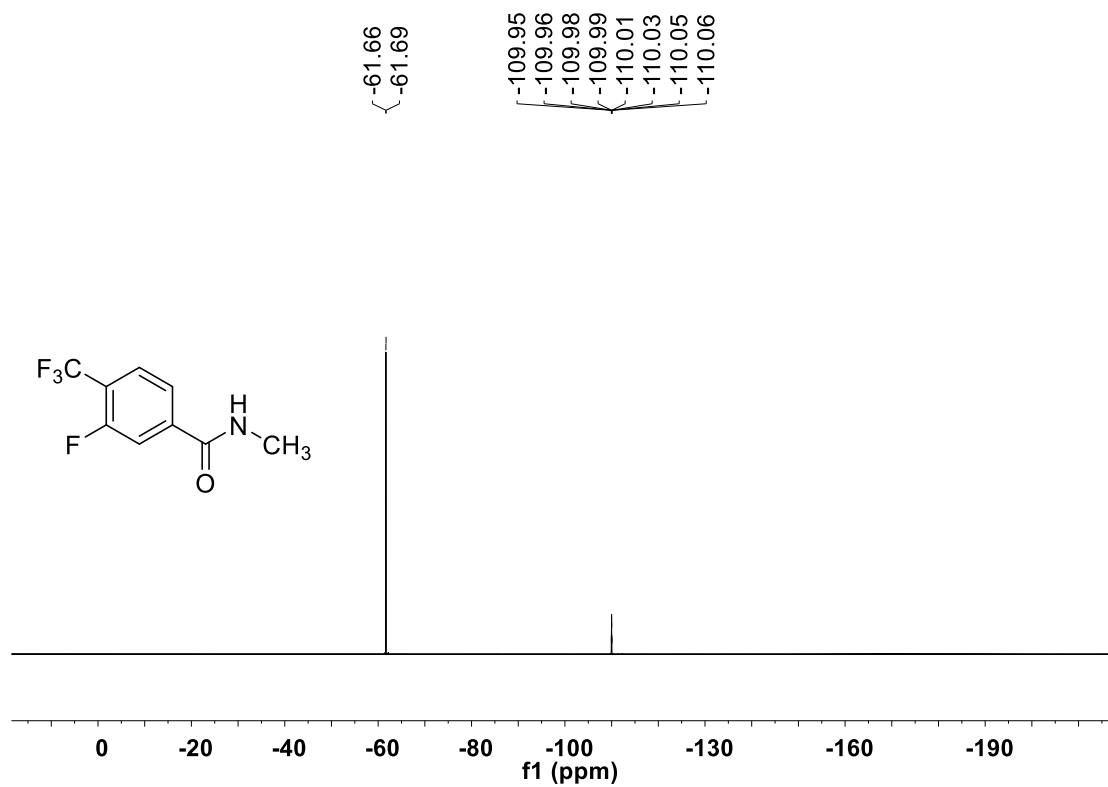
^1H NMR (400 MHz, CDCl_3) spectra for compound **1u**



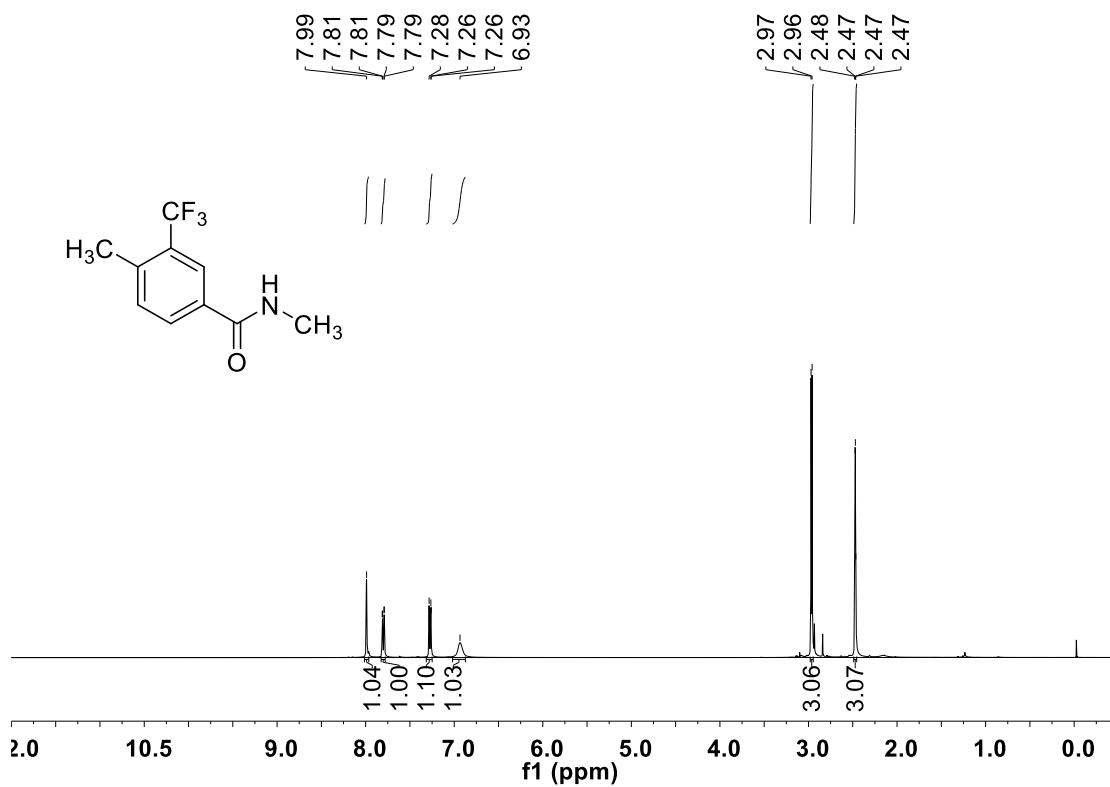
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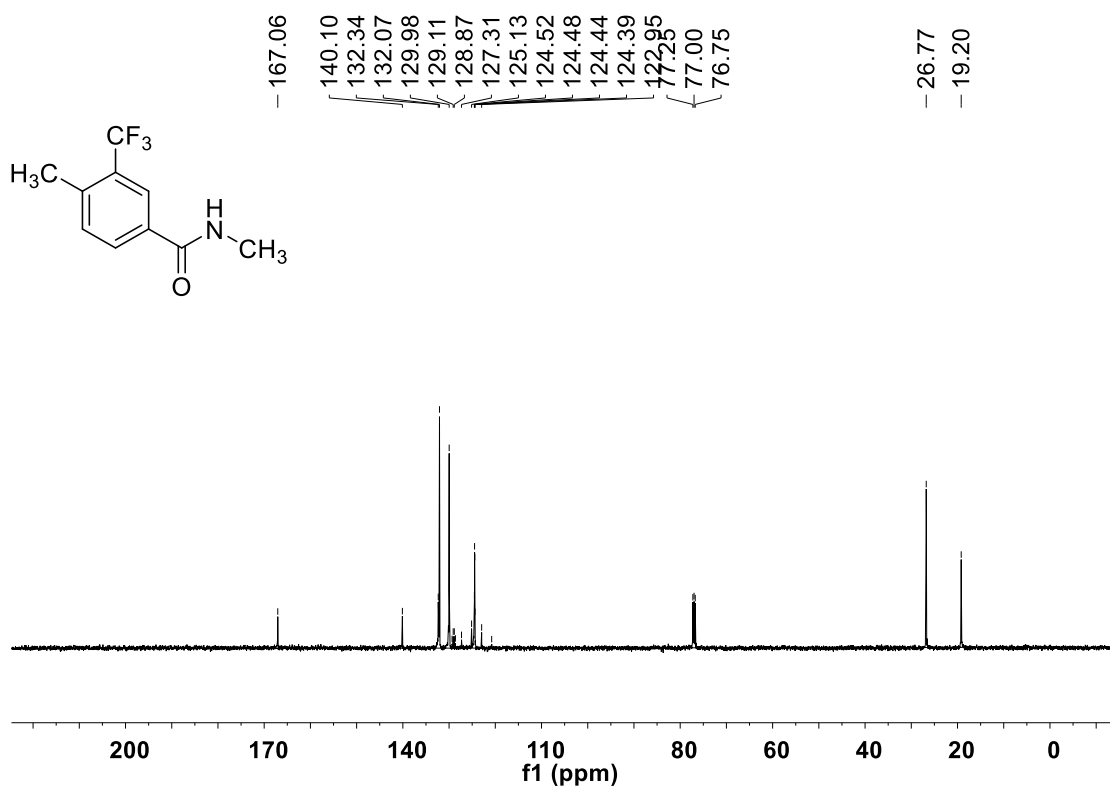
¹⁹F NMR (377 MHz, CDCl₃) spectra for compound **1u**



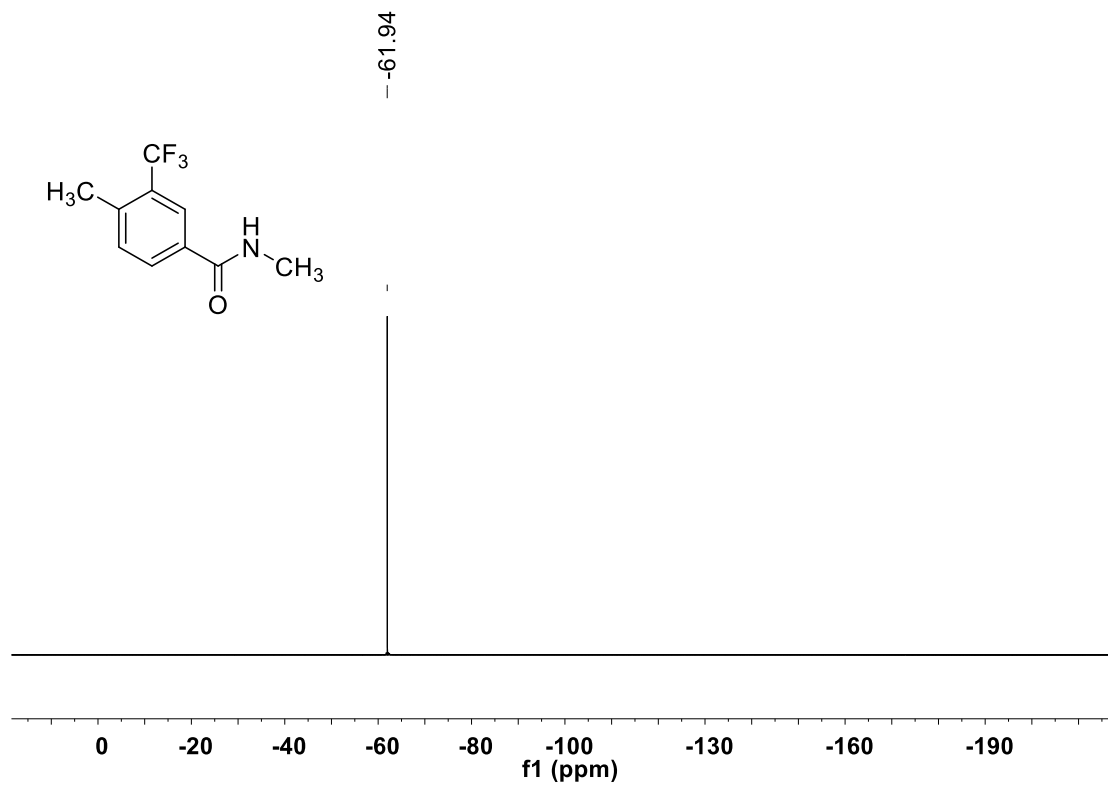
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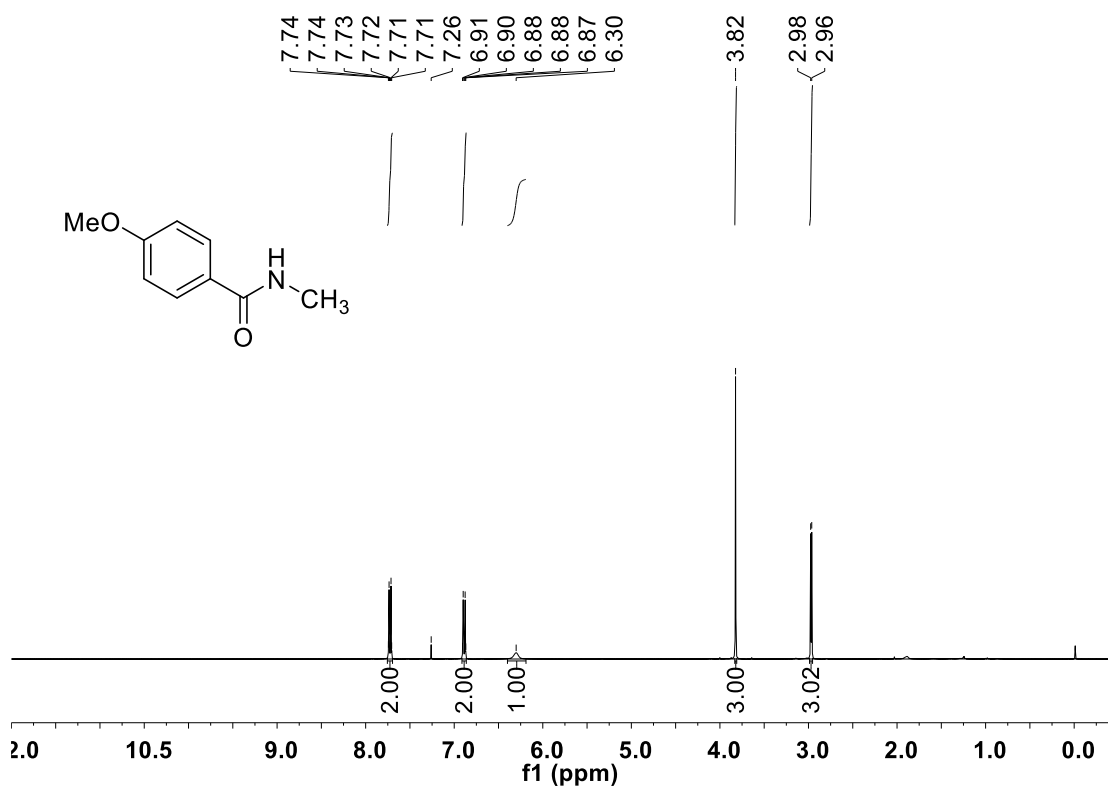
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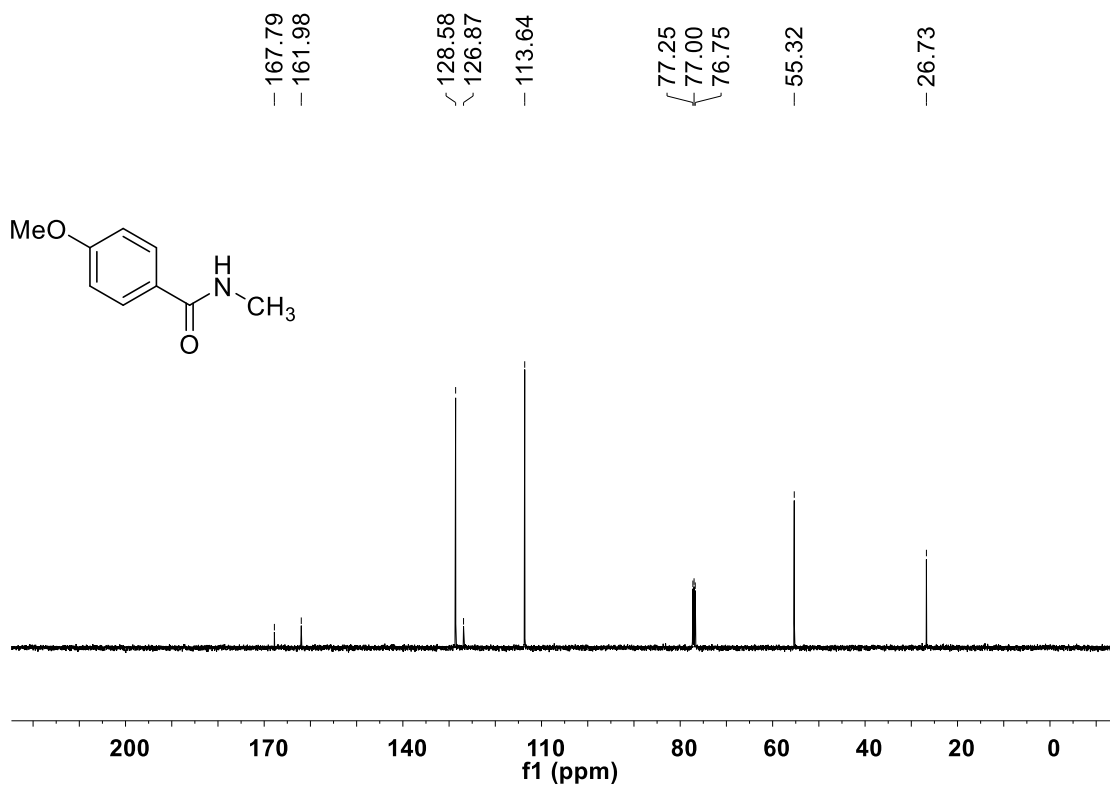
^{19}F NMR (377 MHz, CDCl_3) spectra for compound **1v**



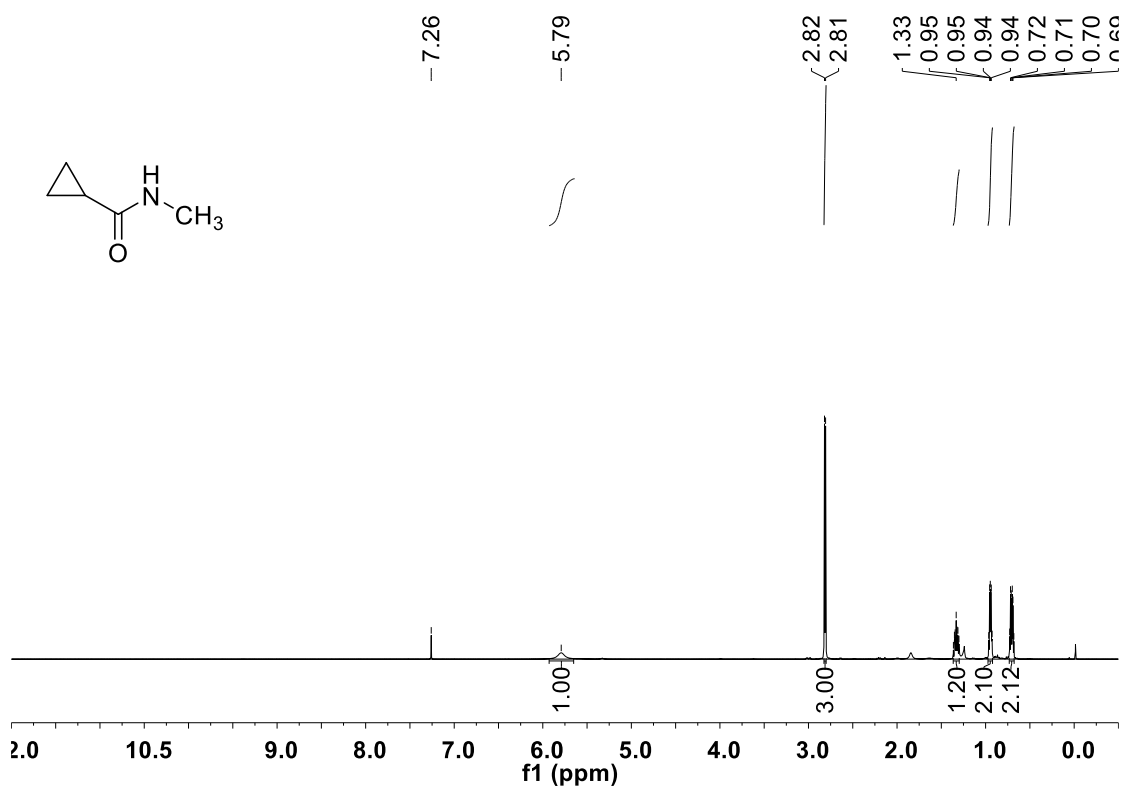
^1H NMR (400 MHz, CDCl_3) spectra for compound **1w**



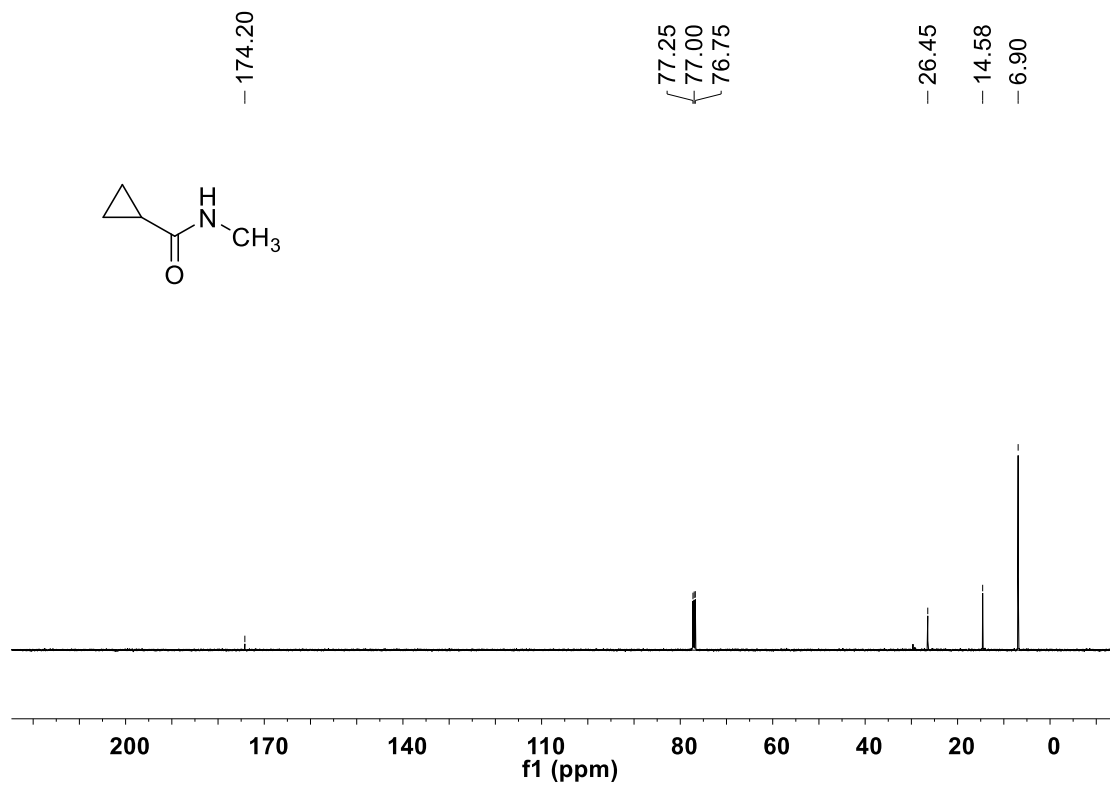
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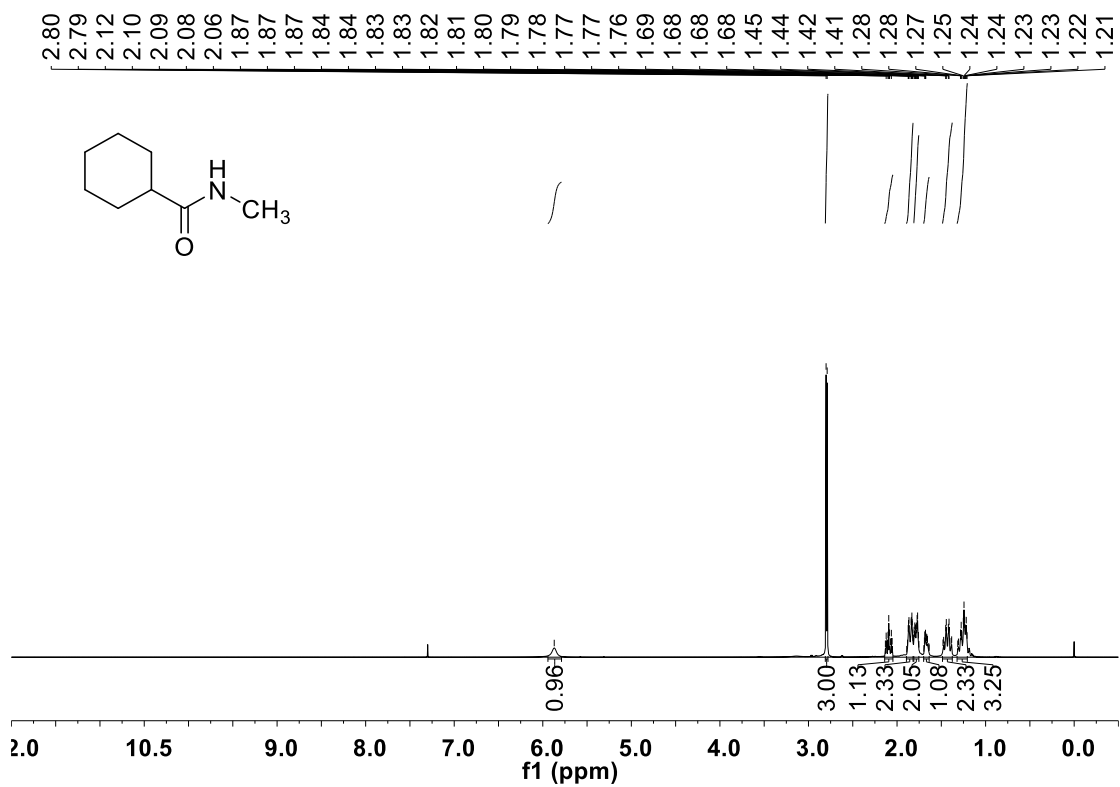
¹H NMR (400 MHz, CDCl₃) spectra for compound **1z**



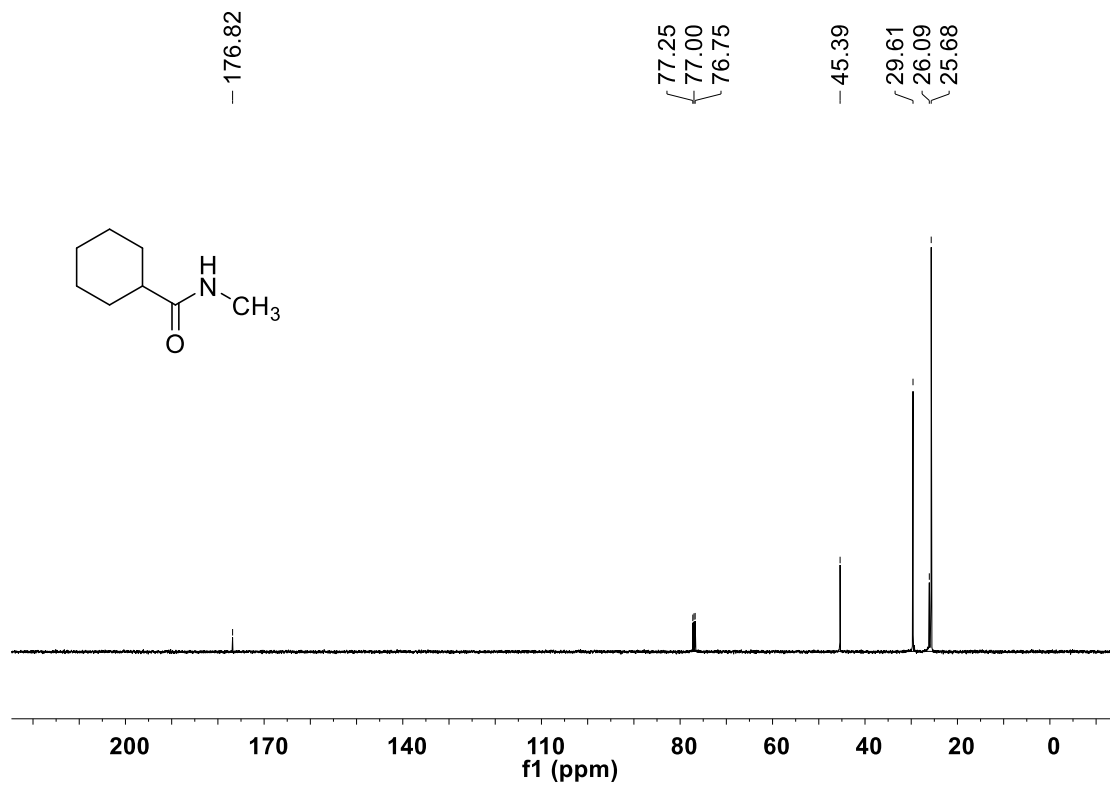
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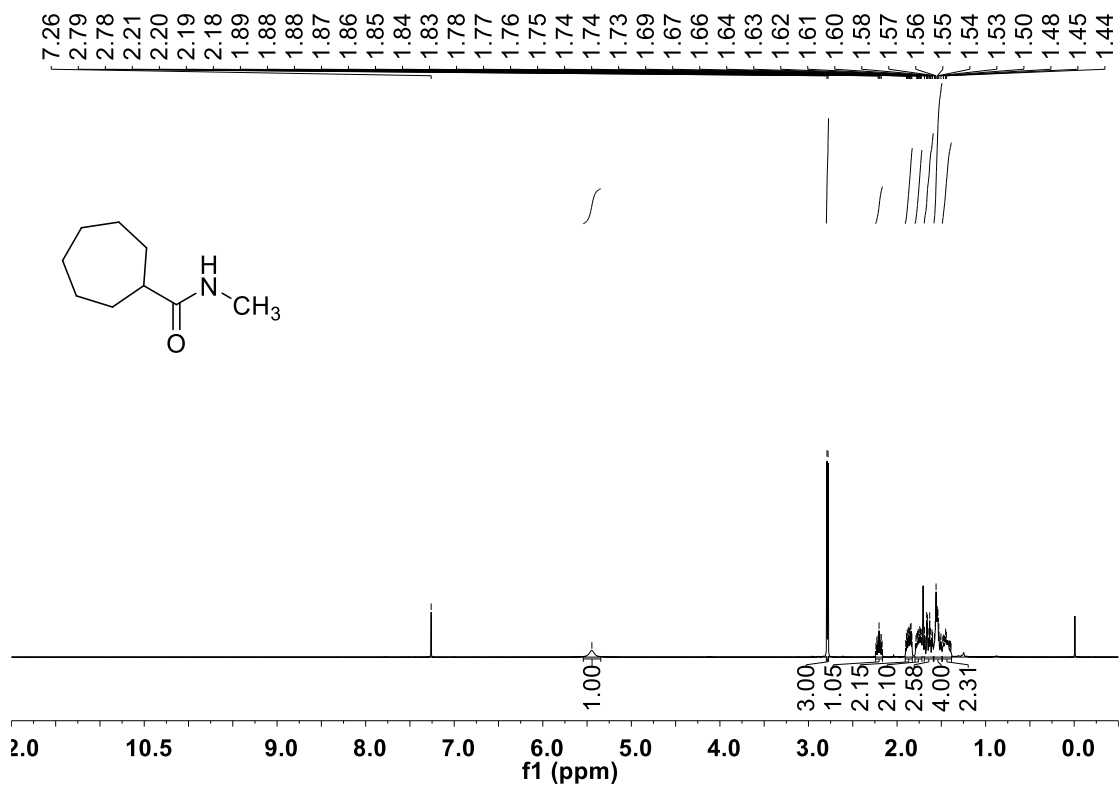
¹H NMR (400 MHz, CDCl₃) spectra for compound **1ac**



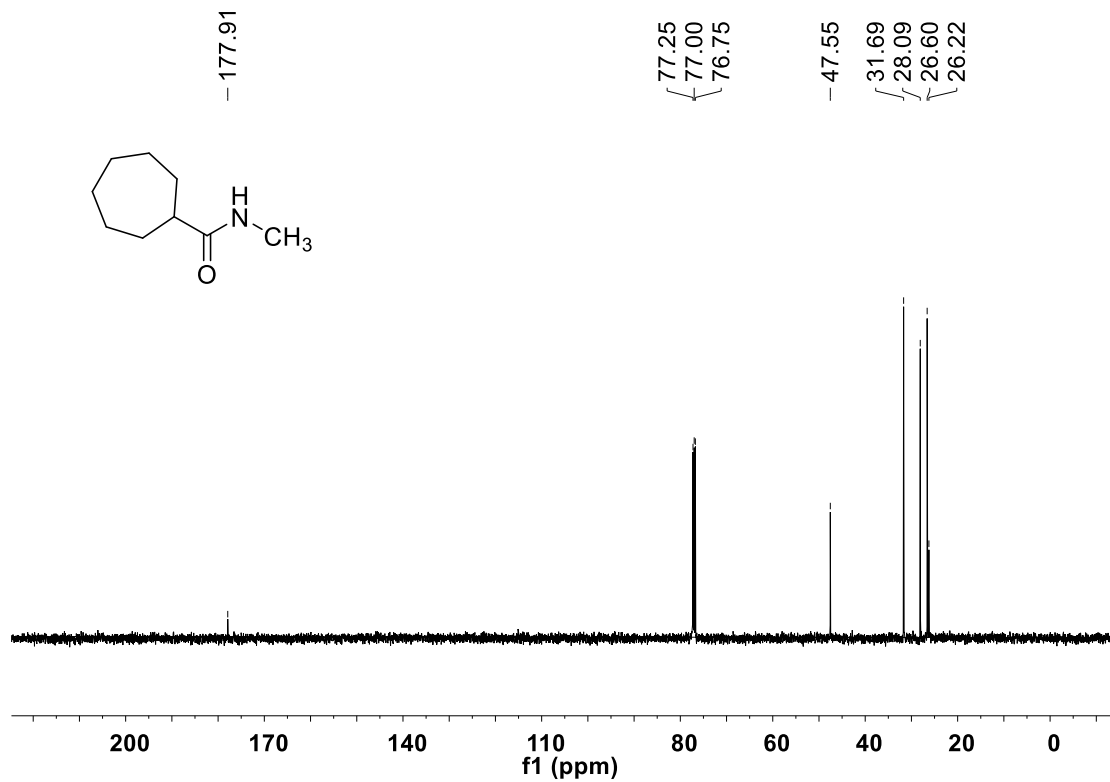
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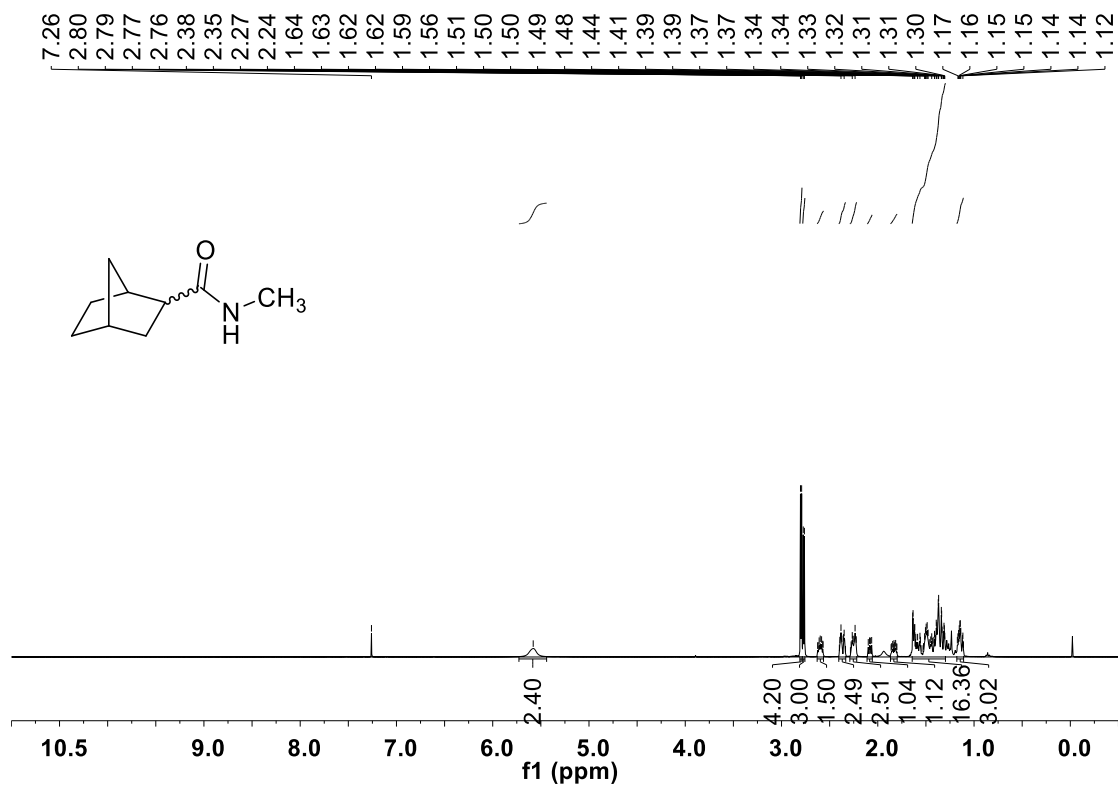
¹H NMR (400 MHz, CDCl₃) spectra for compound **1ad**



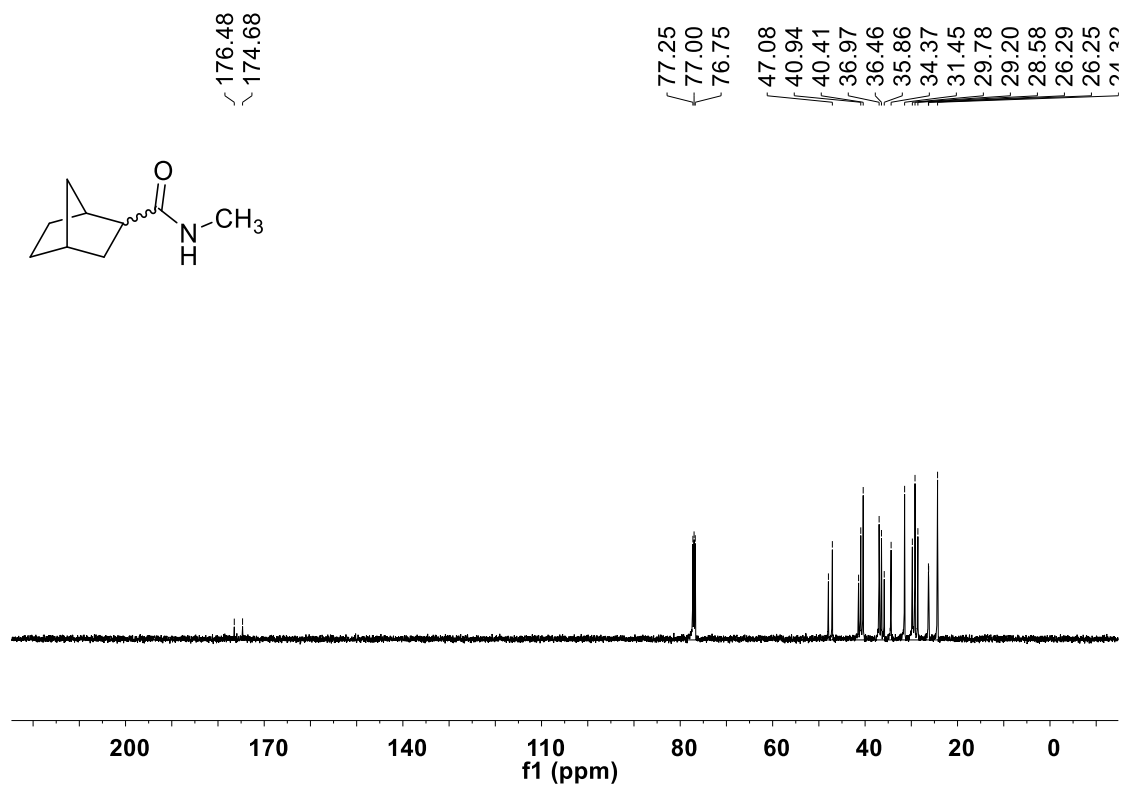
^{13}C NMR (126 MHz, CDCl_3) spectra for compound **1ad**



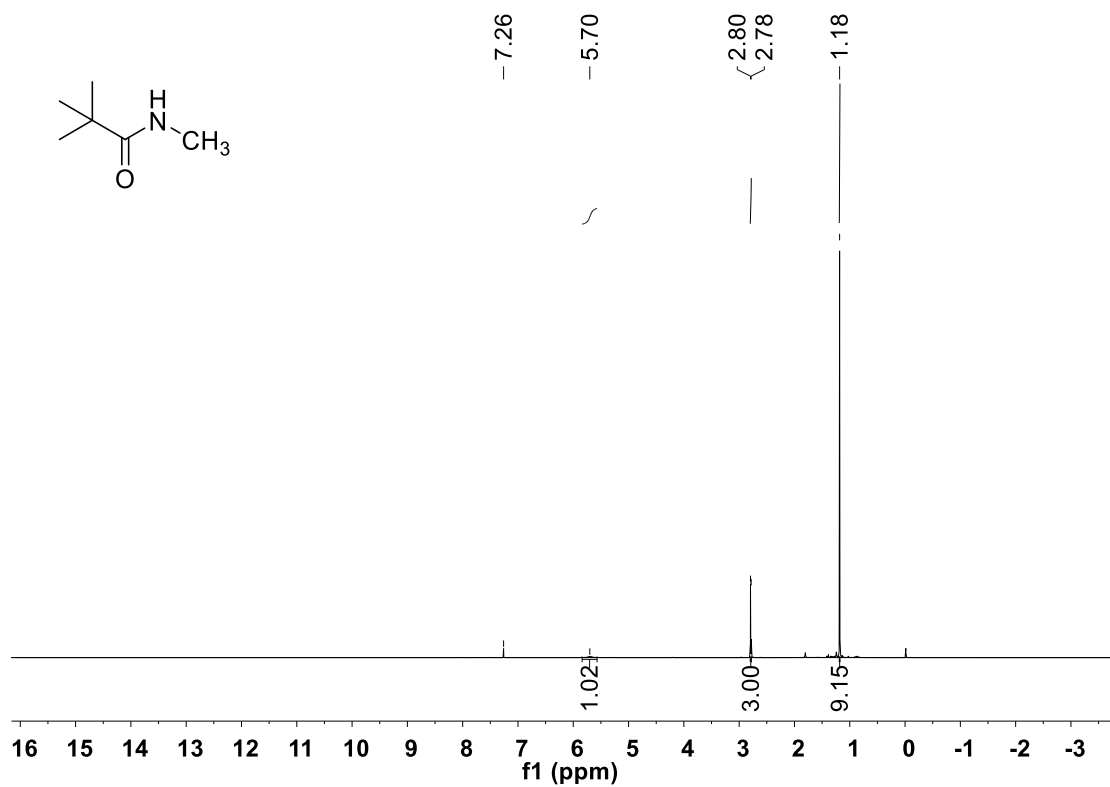
^1H NMR (400 MHz, CDCl_3) spectra for compound **1ae**



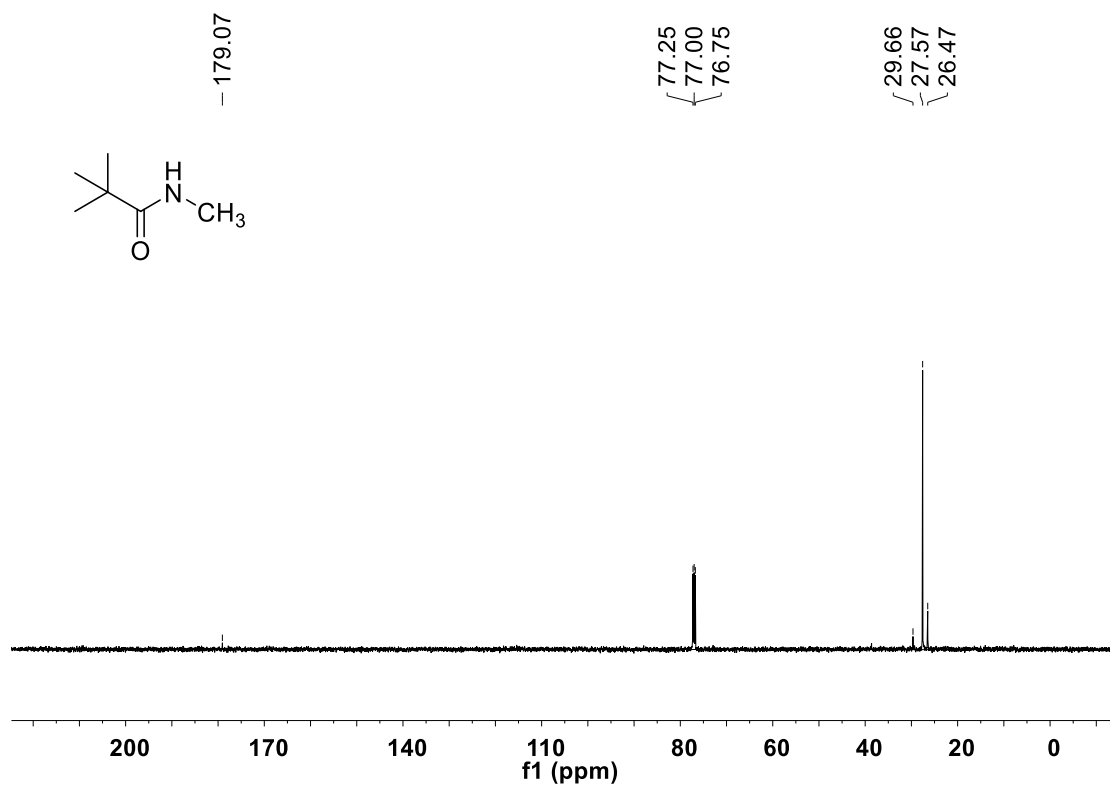
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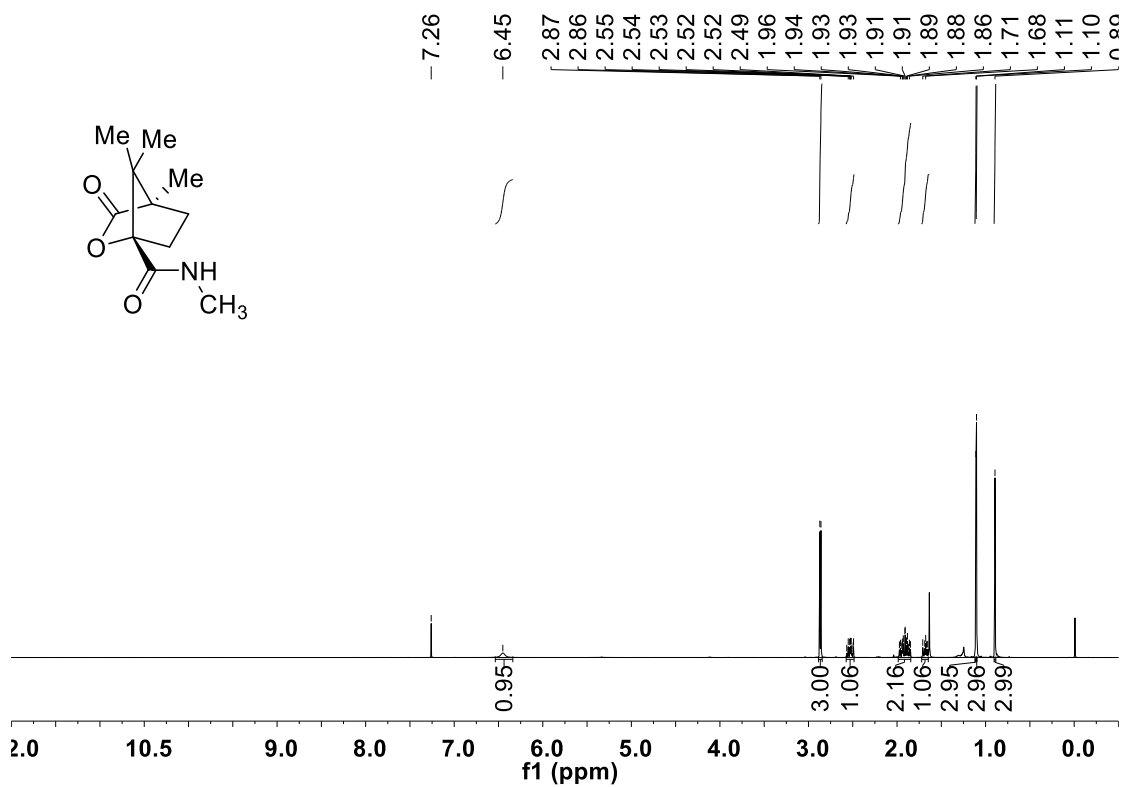
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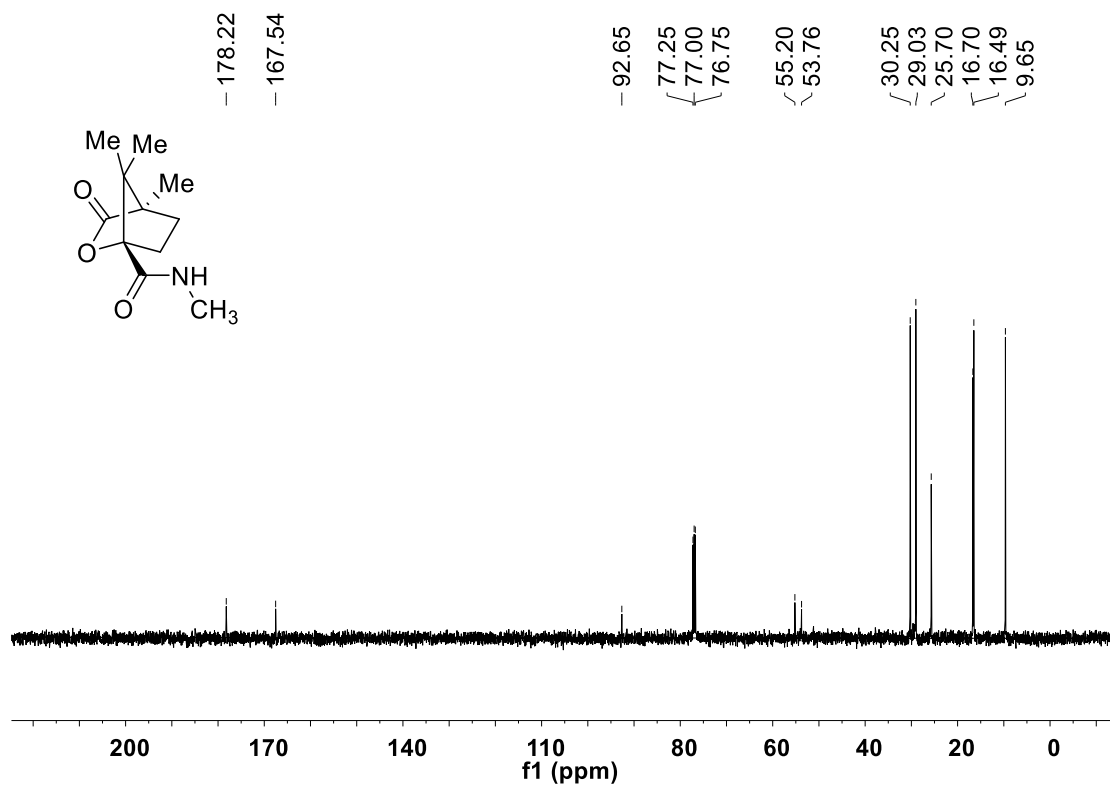
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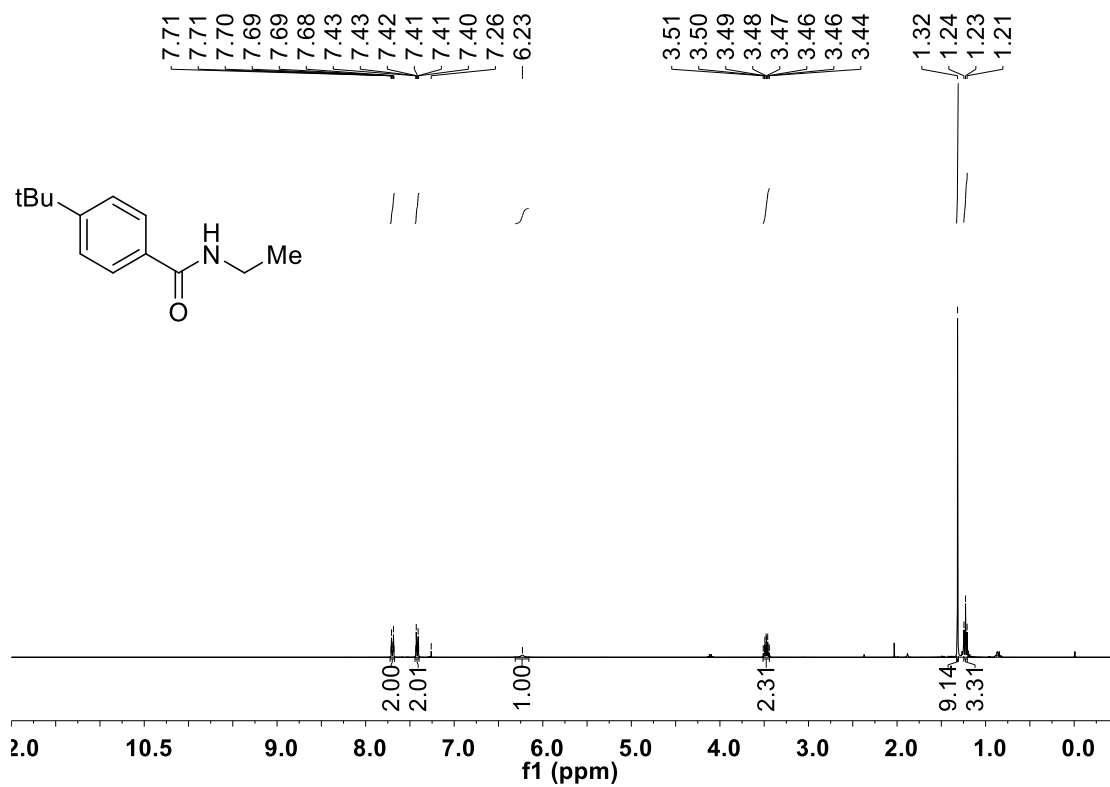
¹H NMR (400 MHz, CDCl₃) spectra for compound **1ah**



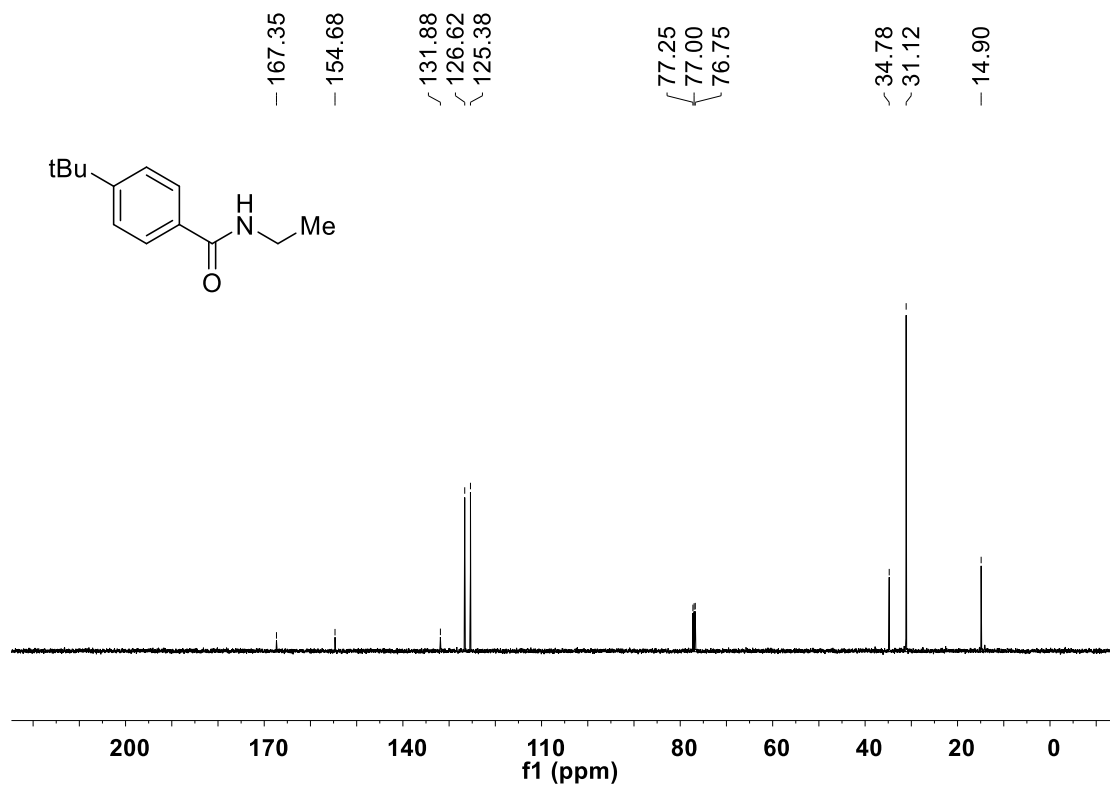
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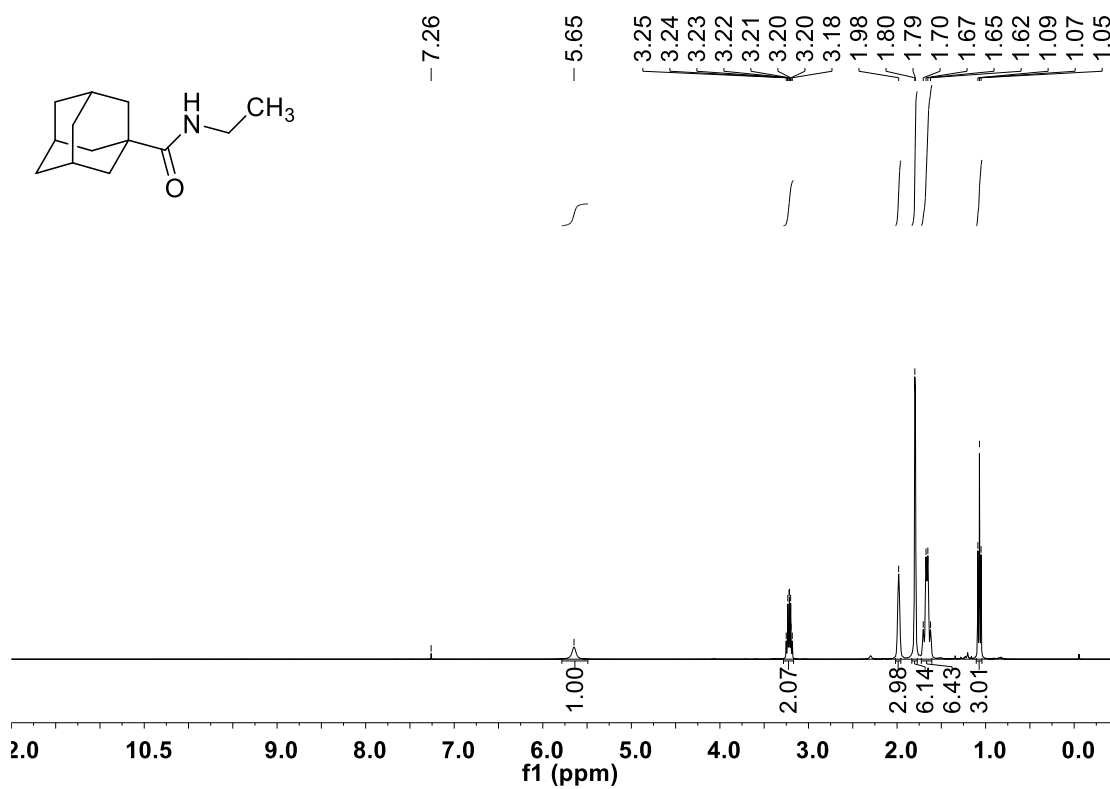
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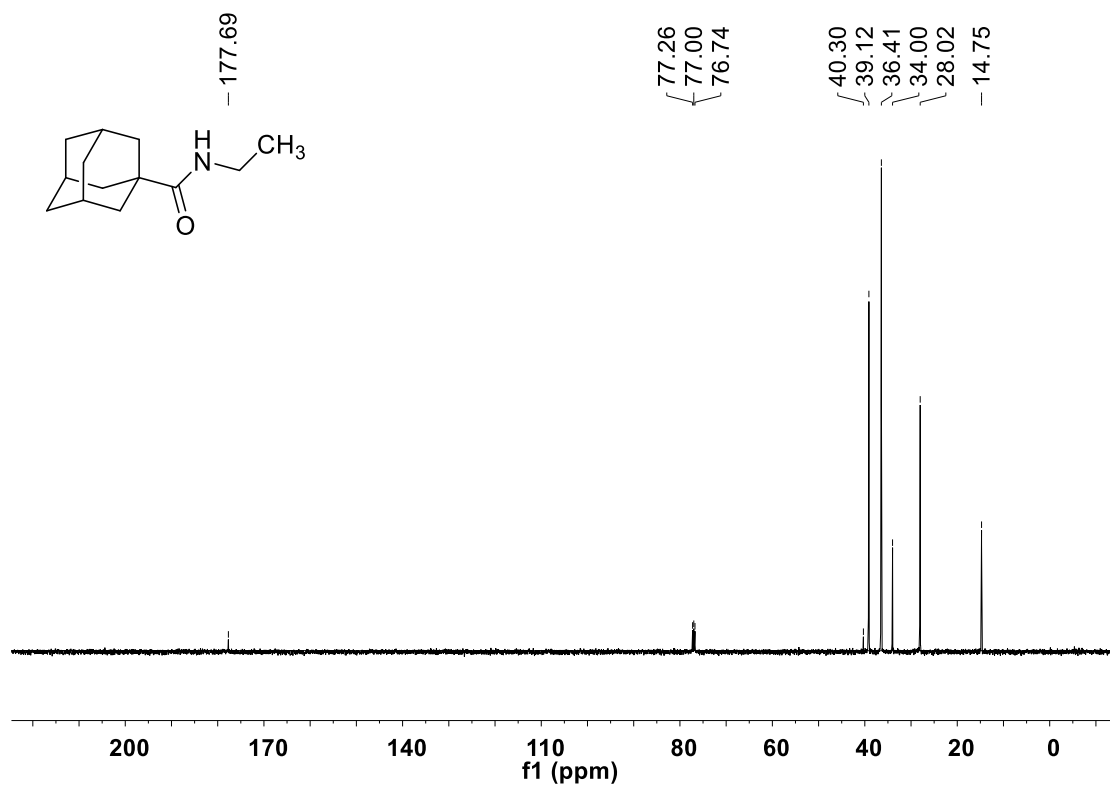
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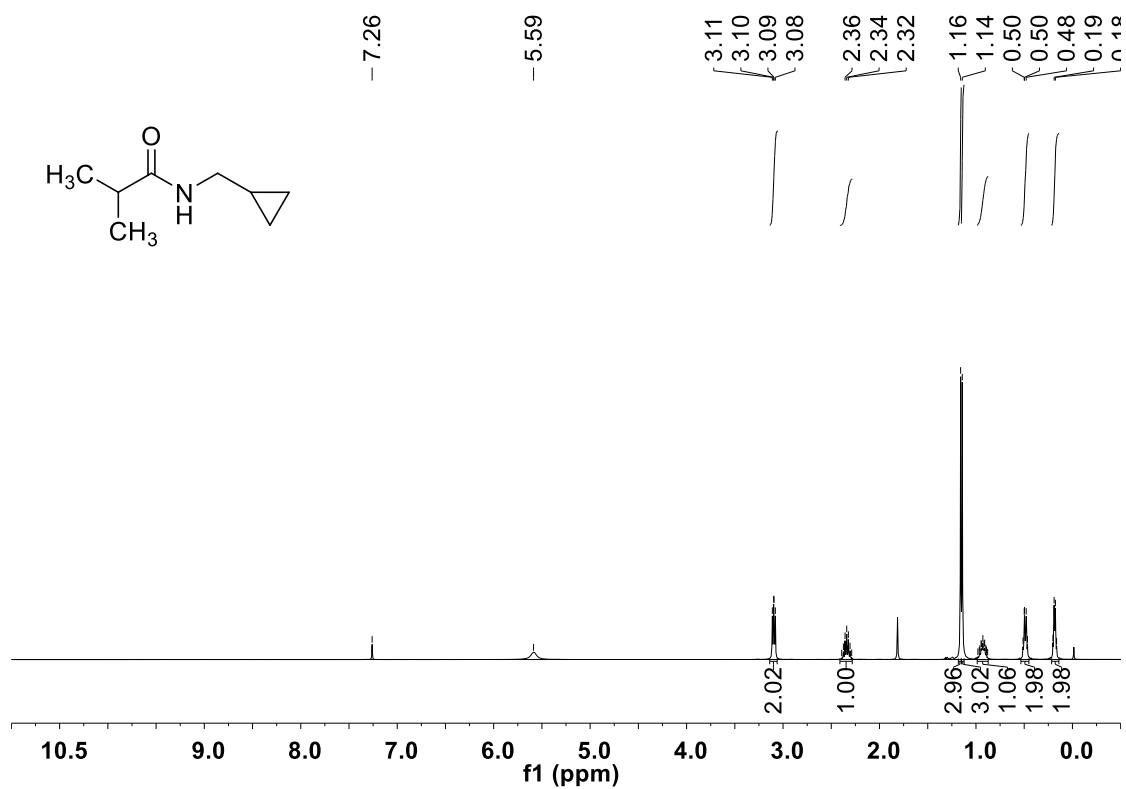
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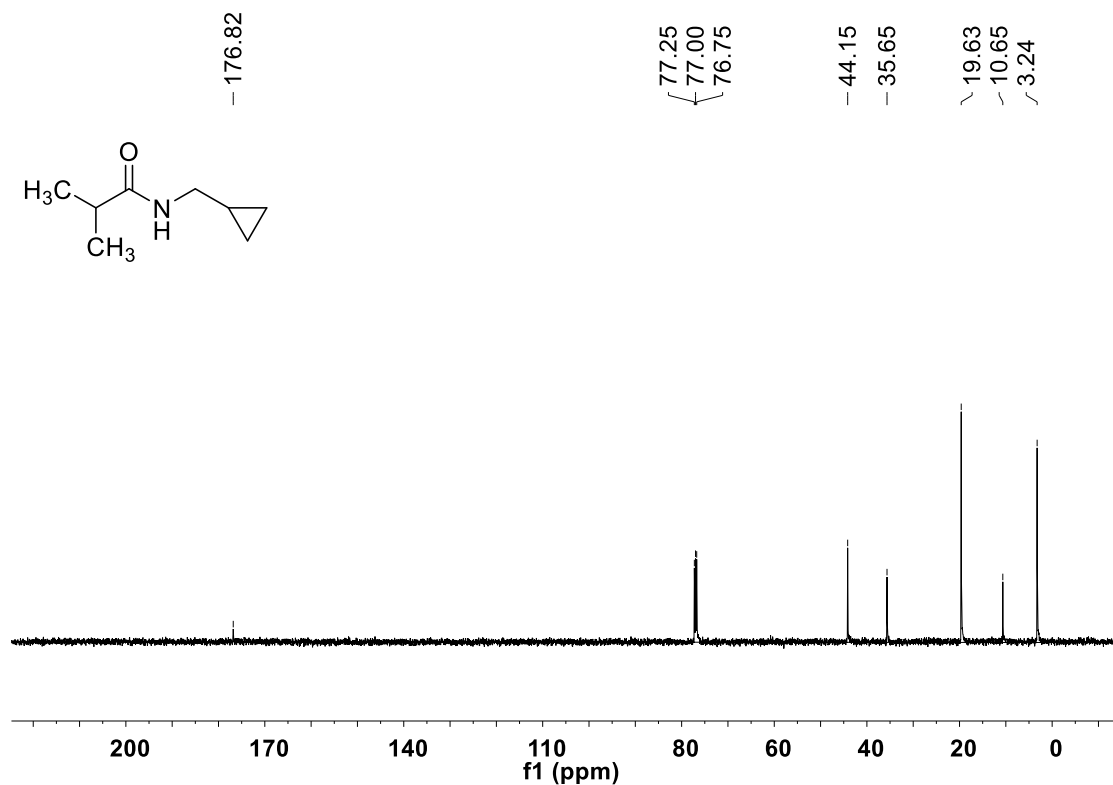
¹³C NMR (126 MHz, CDCl₃) spectra for compound **1am**



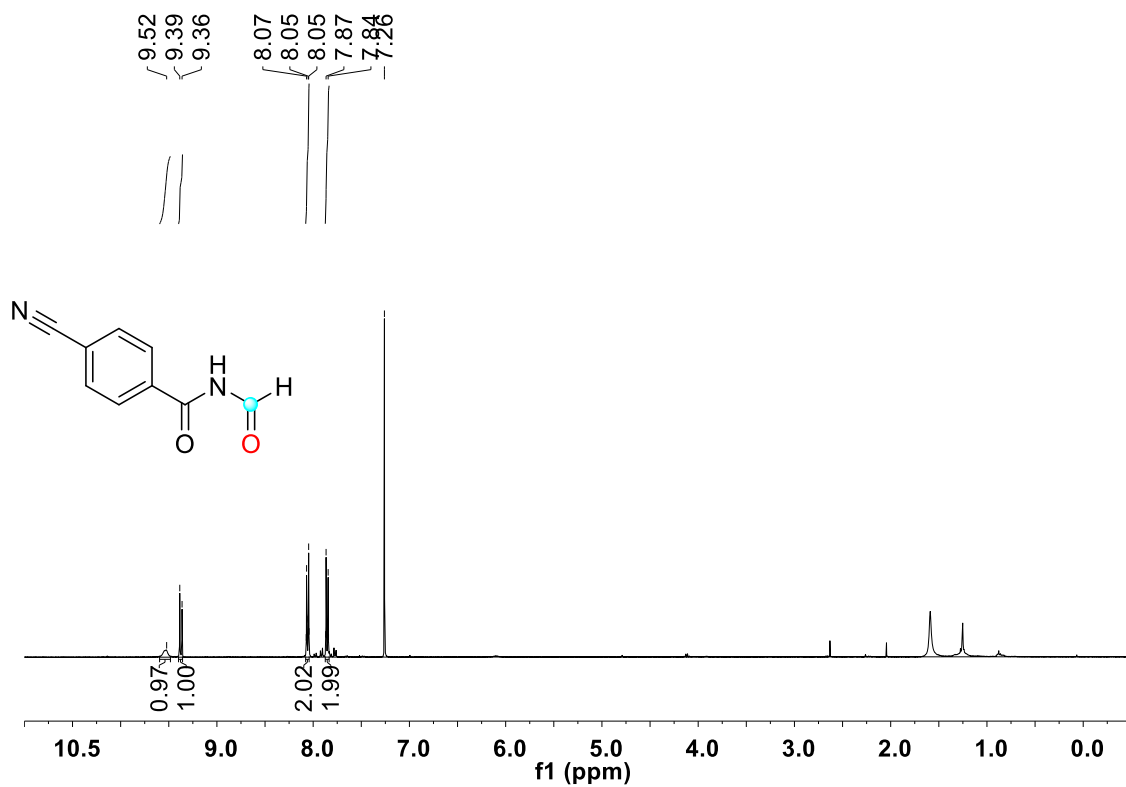
¹H NMR (400 MHz, CDCl₃) spectra for compound **1an**



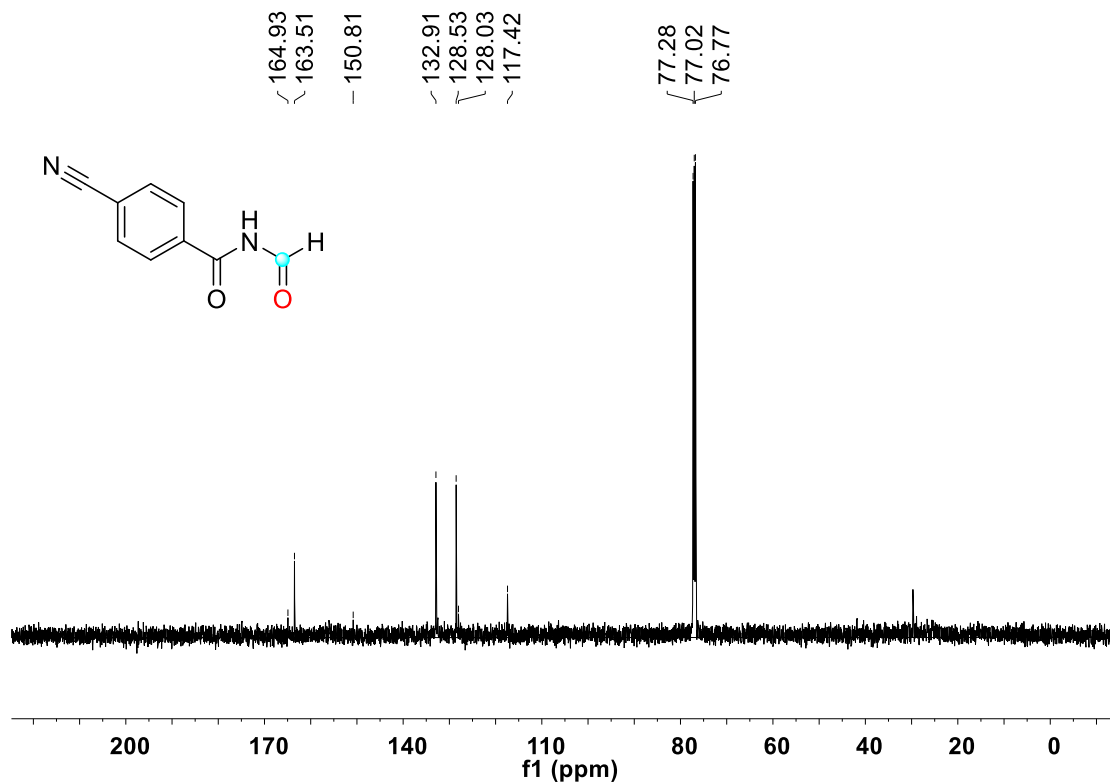
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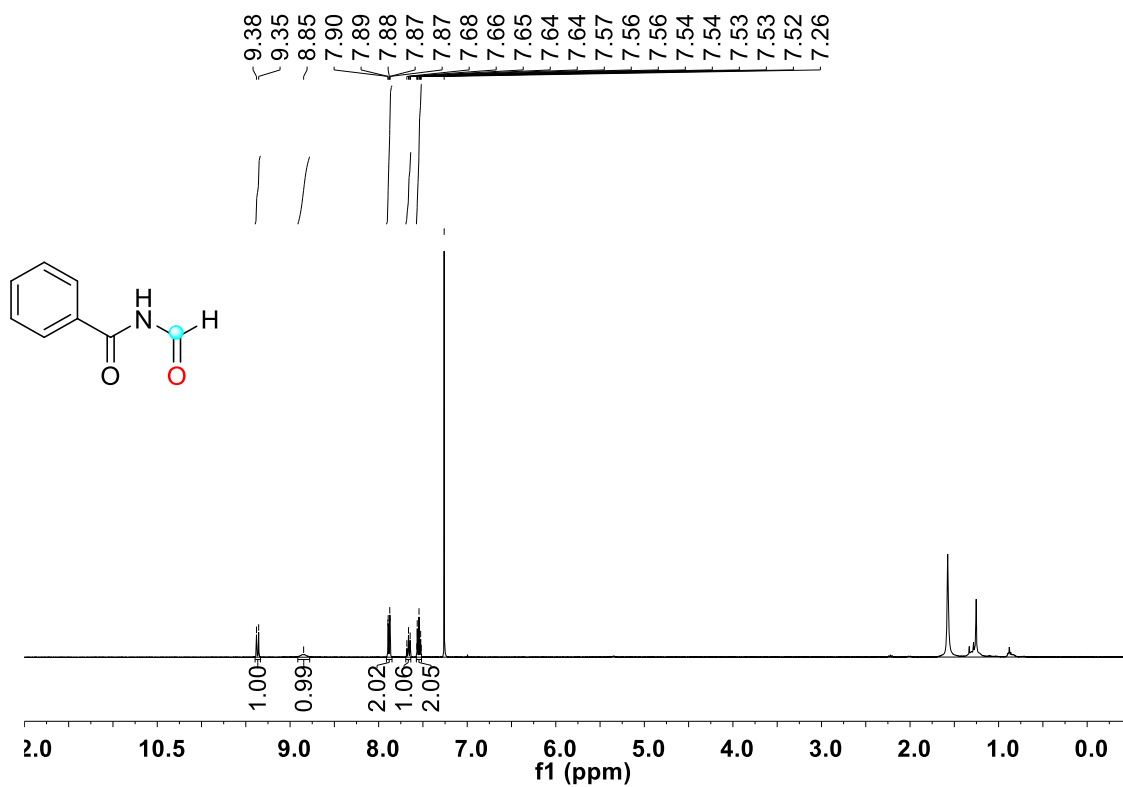
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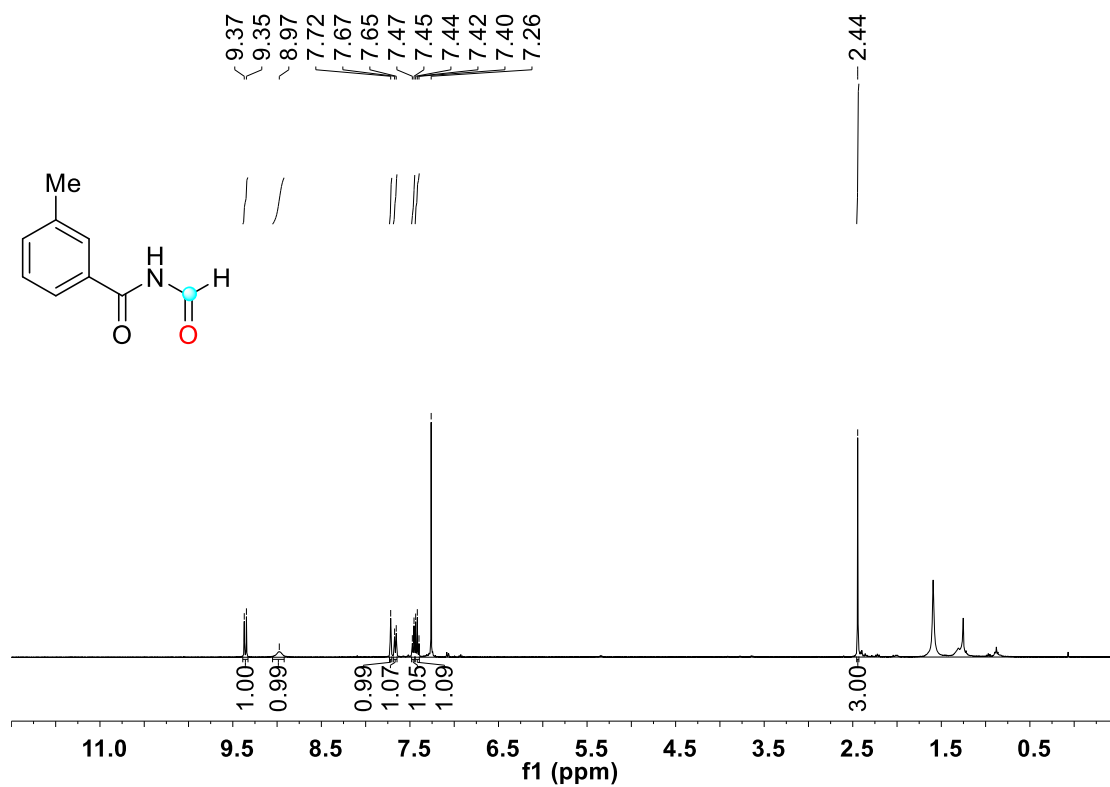
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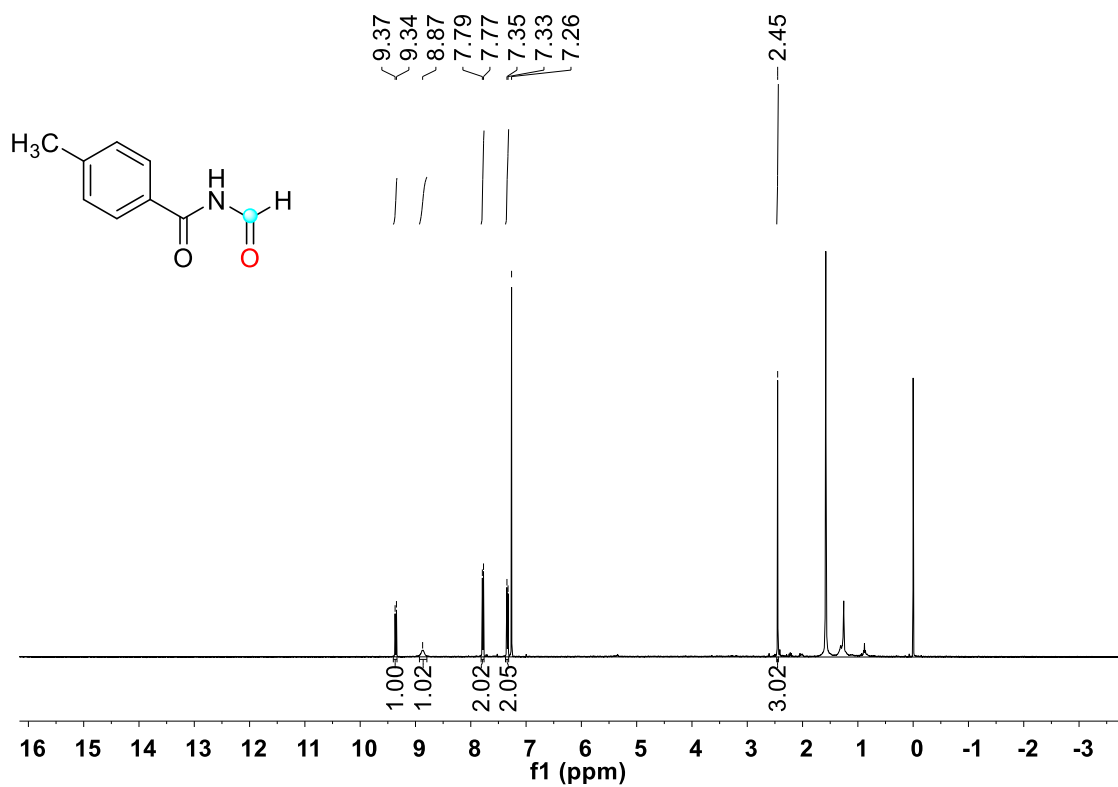
¹H NMR (400 MHz, CDCl₃) spectra for compound **2b**



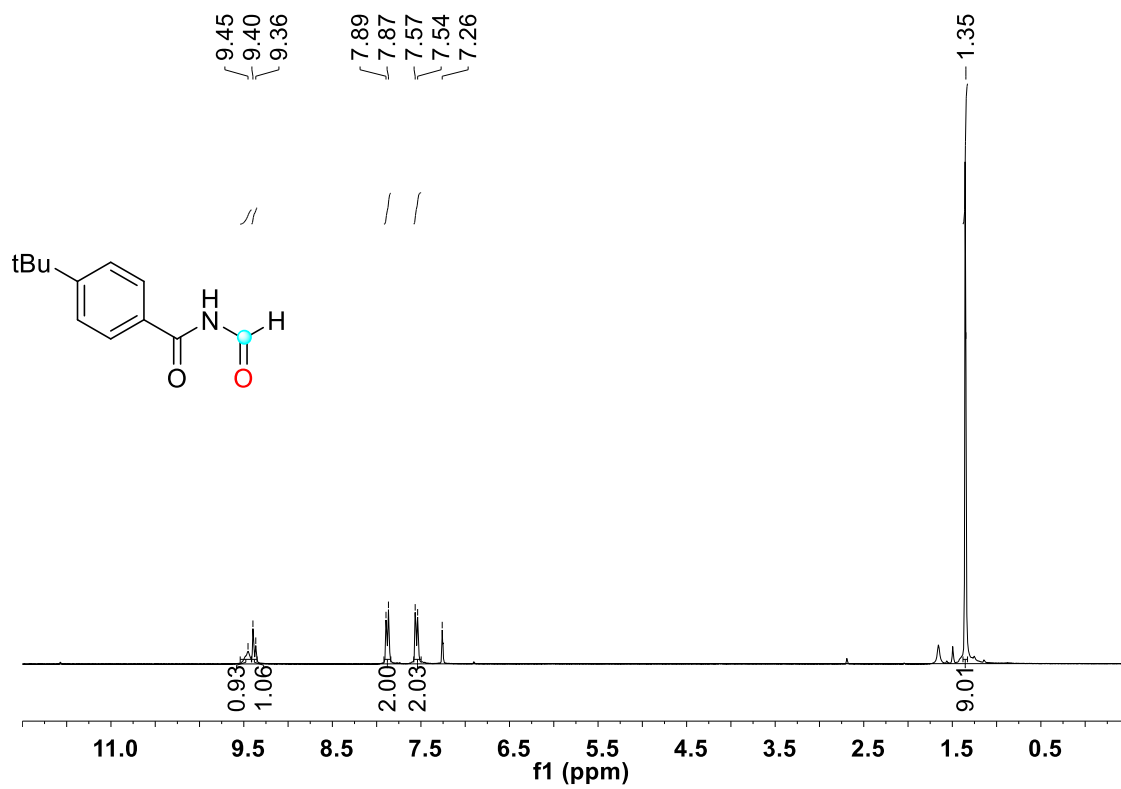
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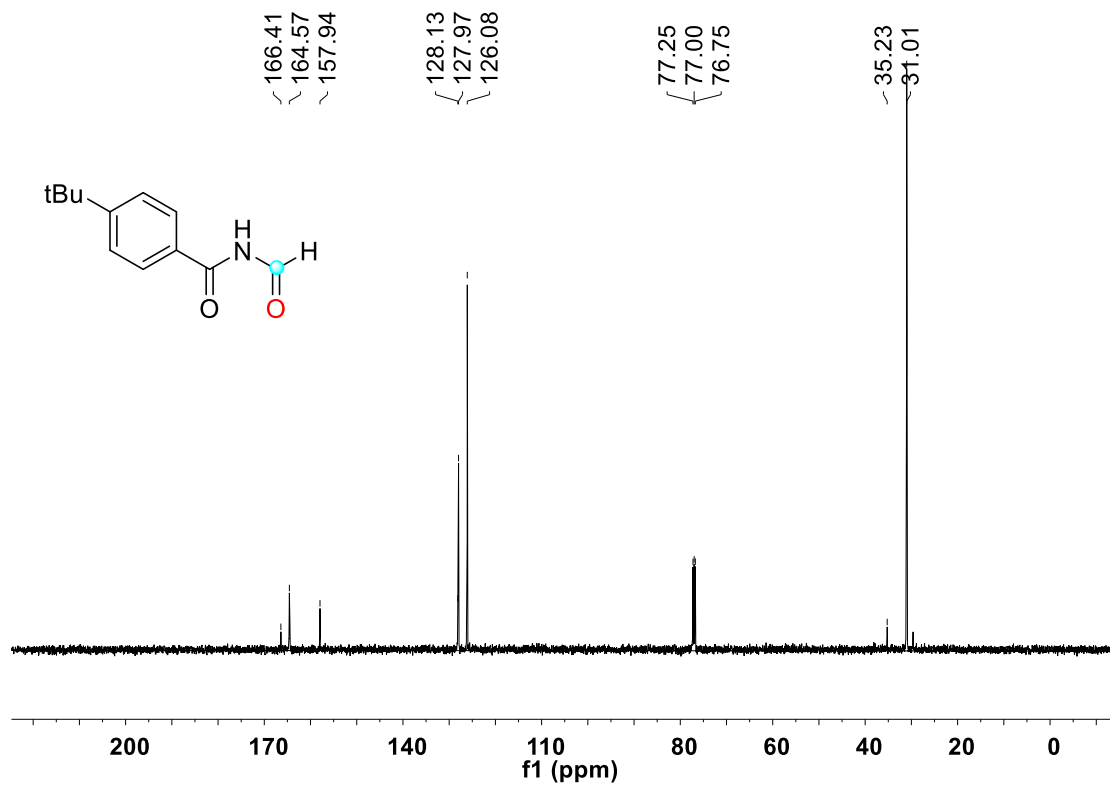
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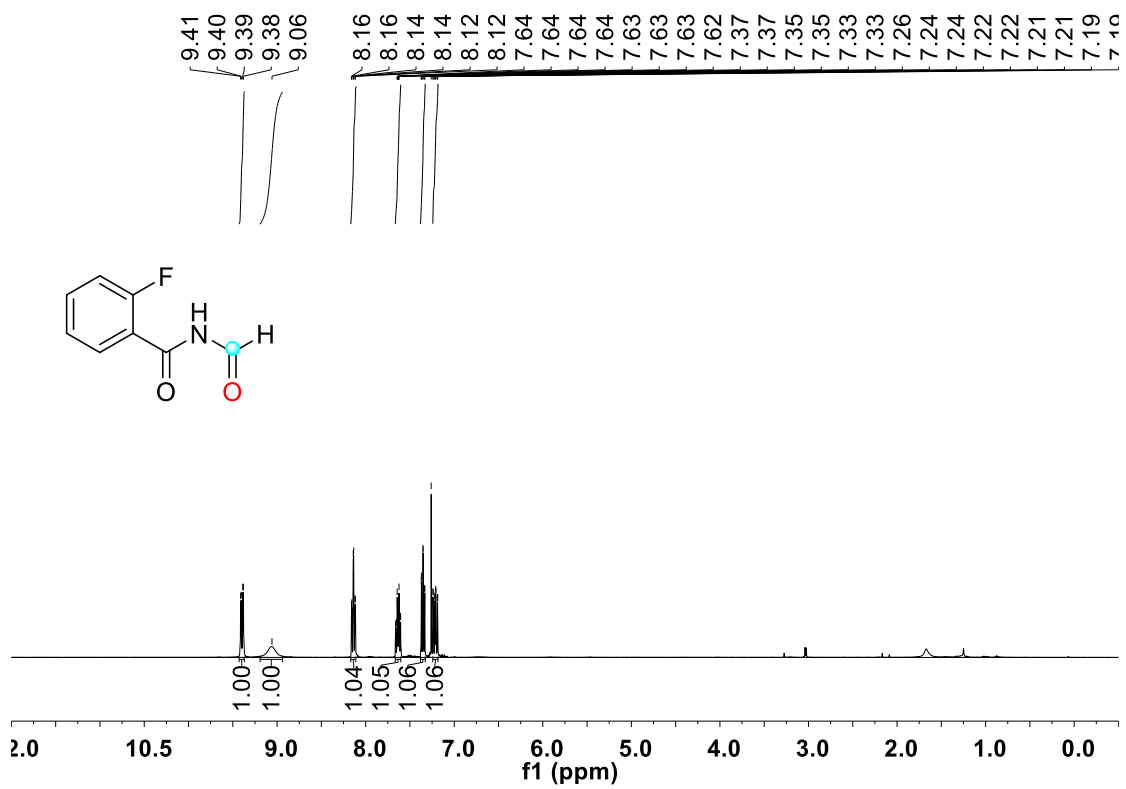
¹H NMR (400 MHz, CDCl₃) spectra for compound **2e**



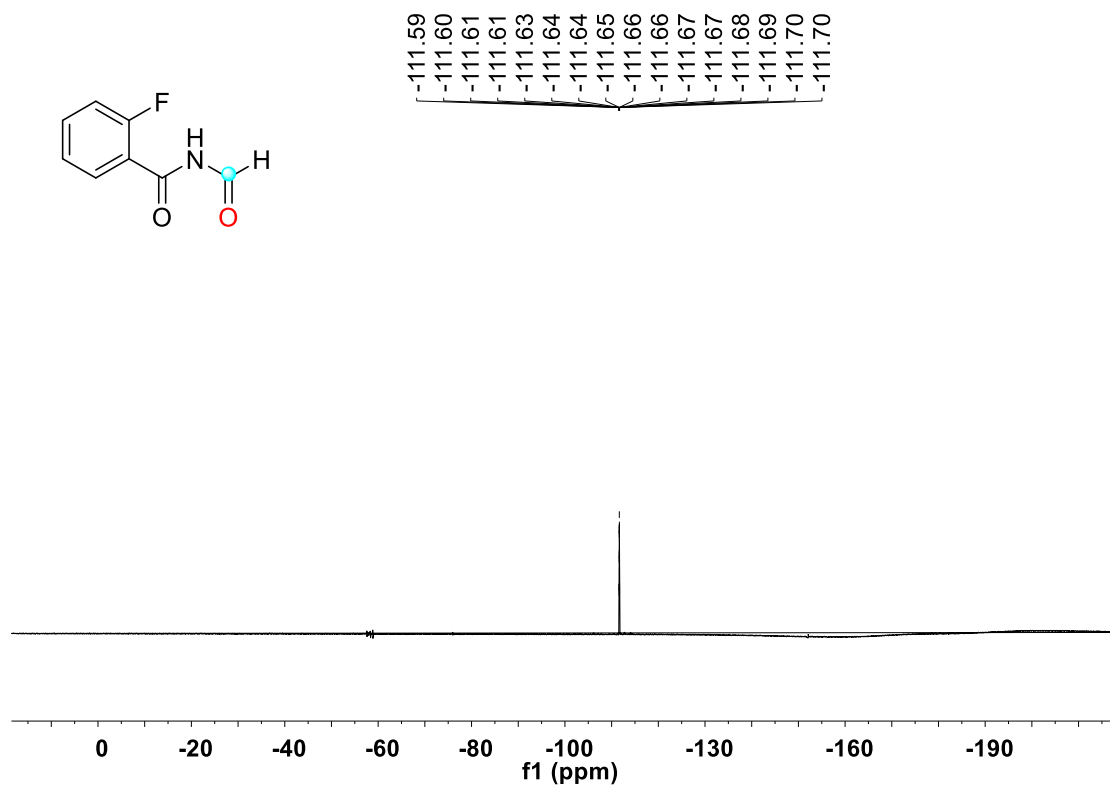
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2e**



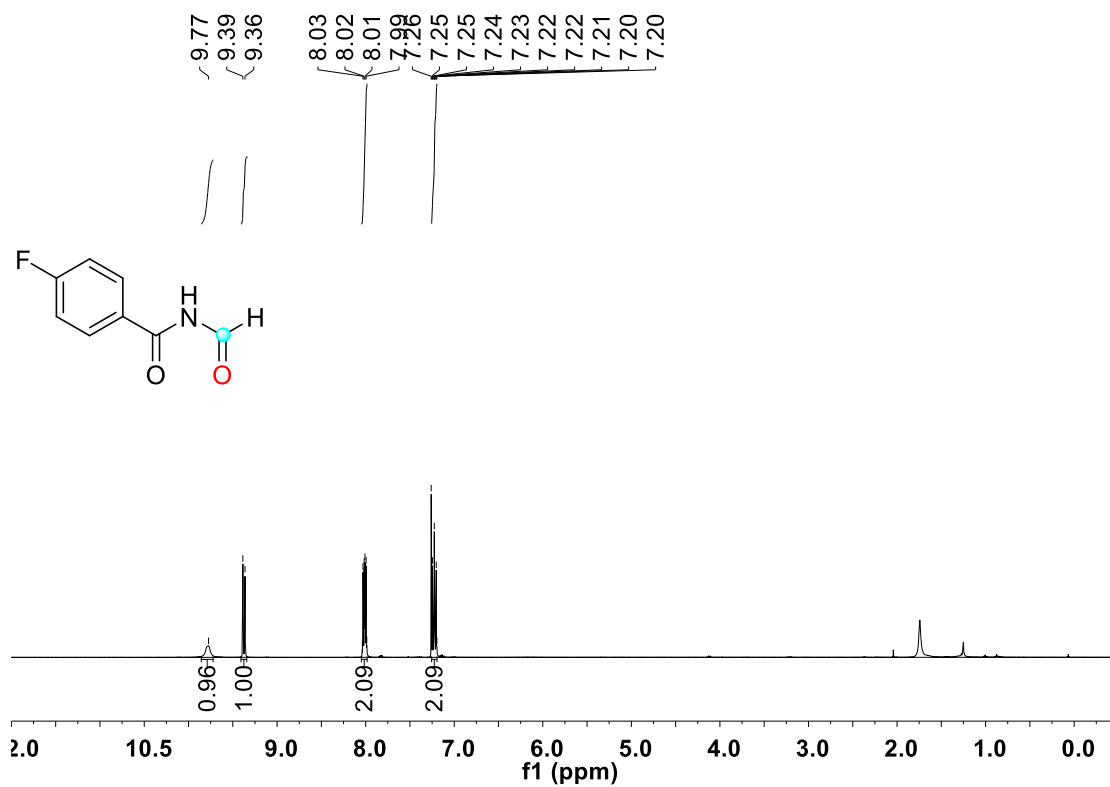
¹H NMR (400 MHz, CDCl₃) spectra for compound **2f**



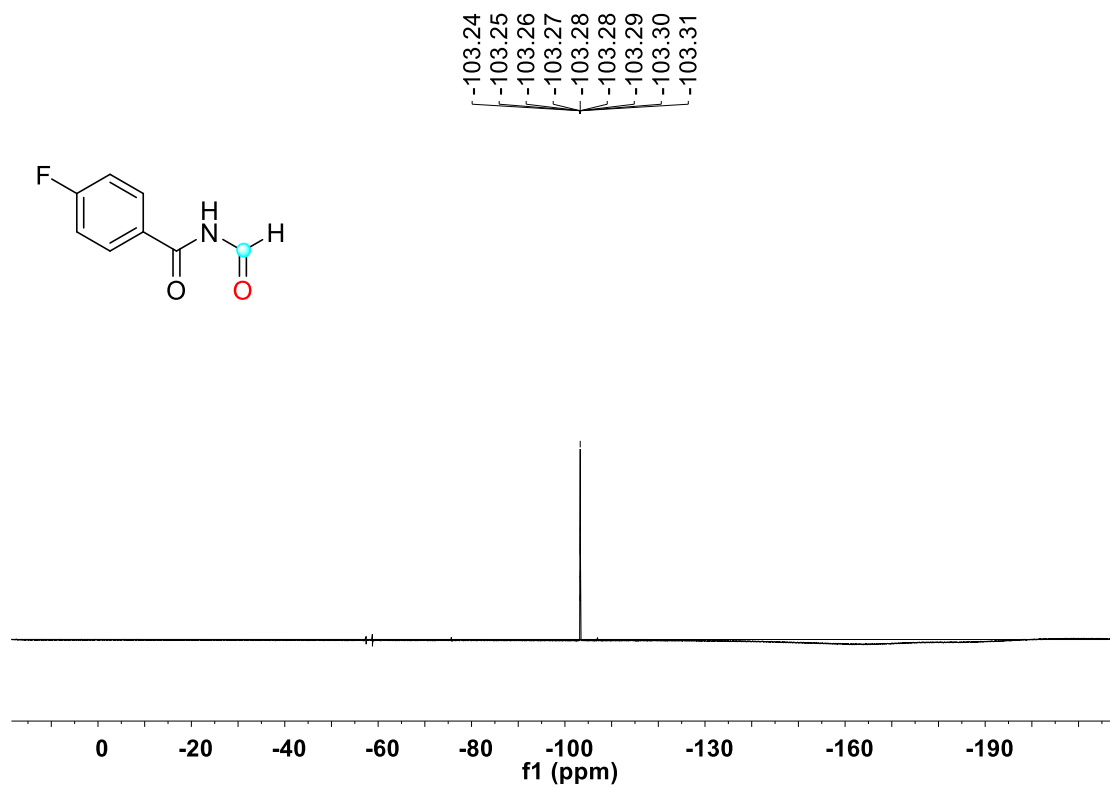
¹⁹F NMR (377 MHz, CDCl₃) spectra for compound **2f**



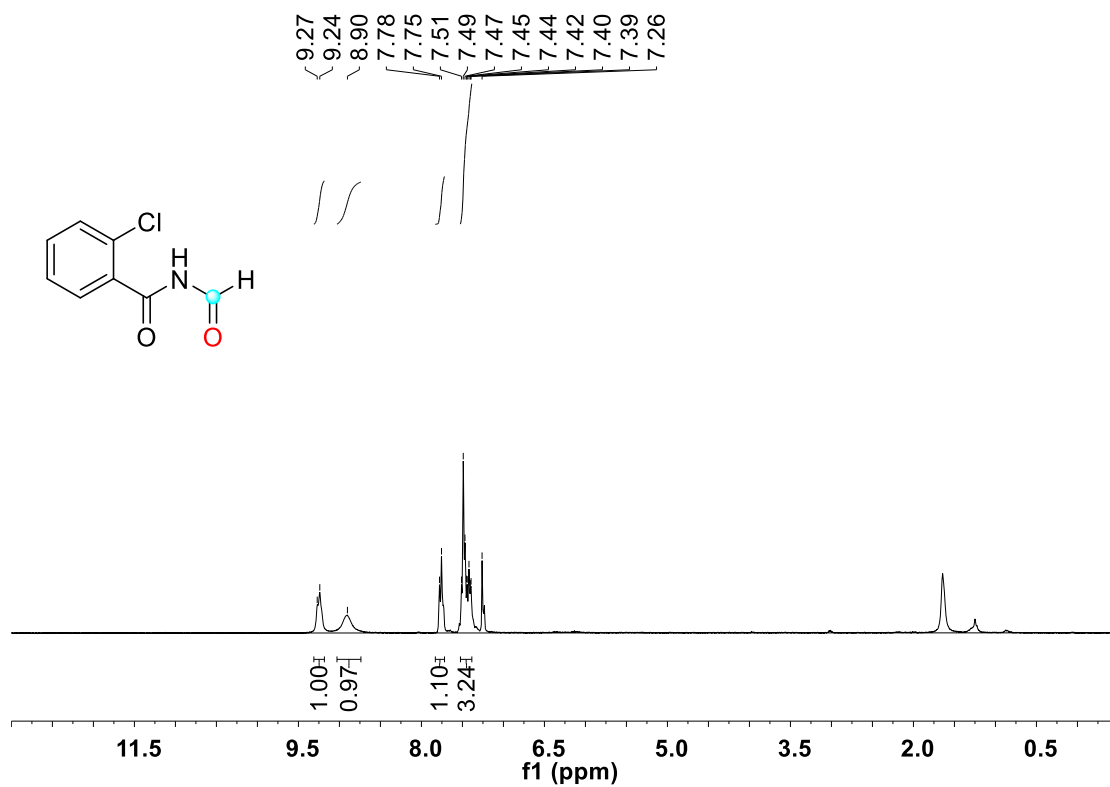
^1H NMR (400 MHz, CDCl_3) spectra for compound **2g**



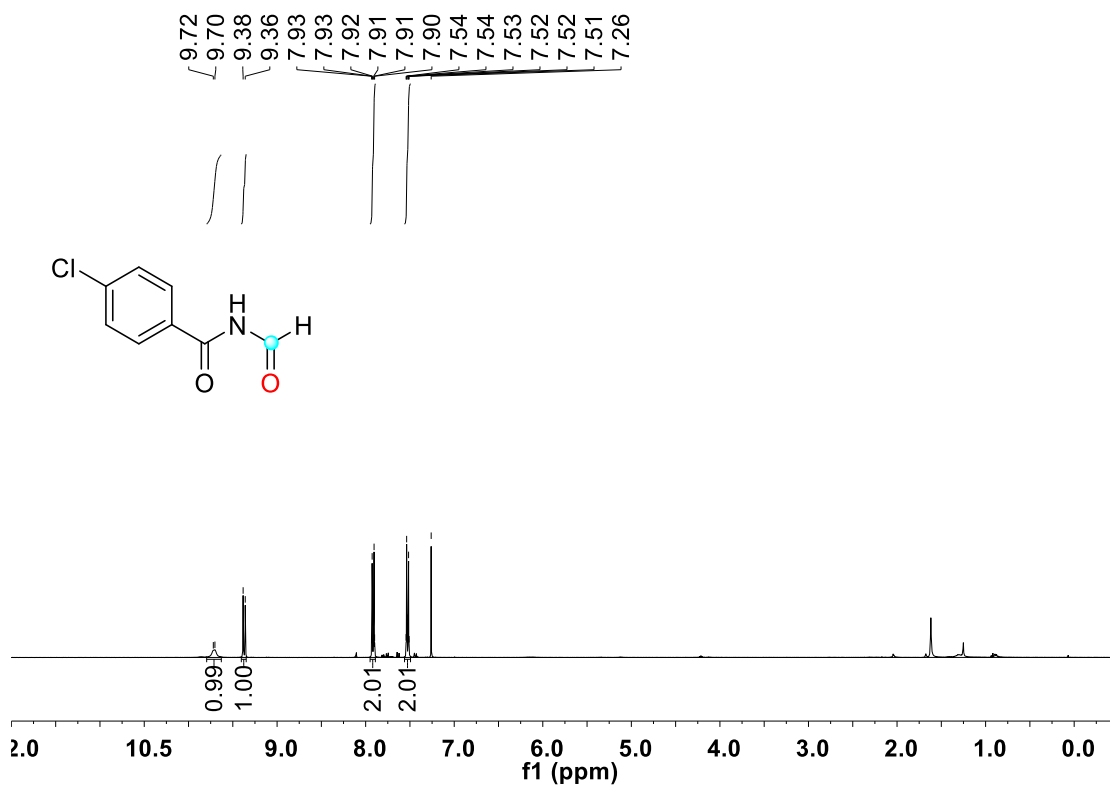
^{19}F NMR (377 MHz, CDCl_3) spectra for compound **2g**



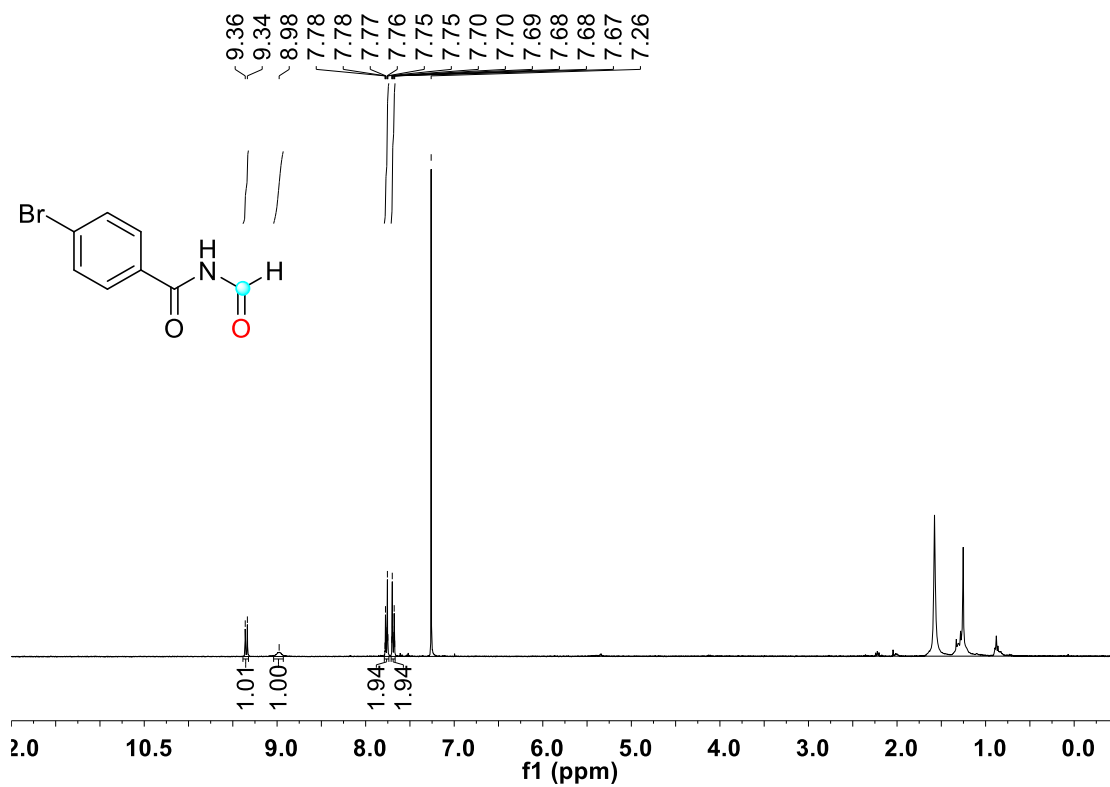
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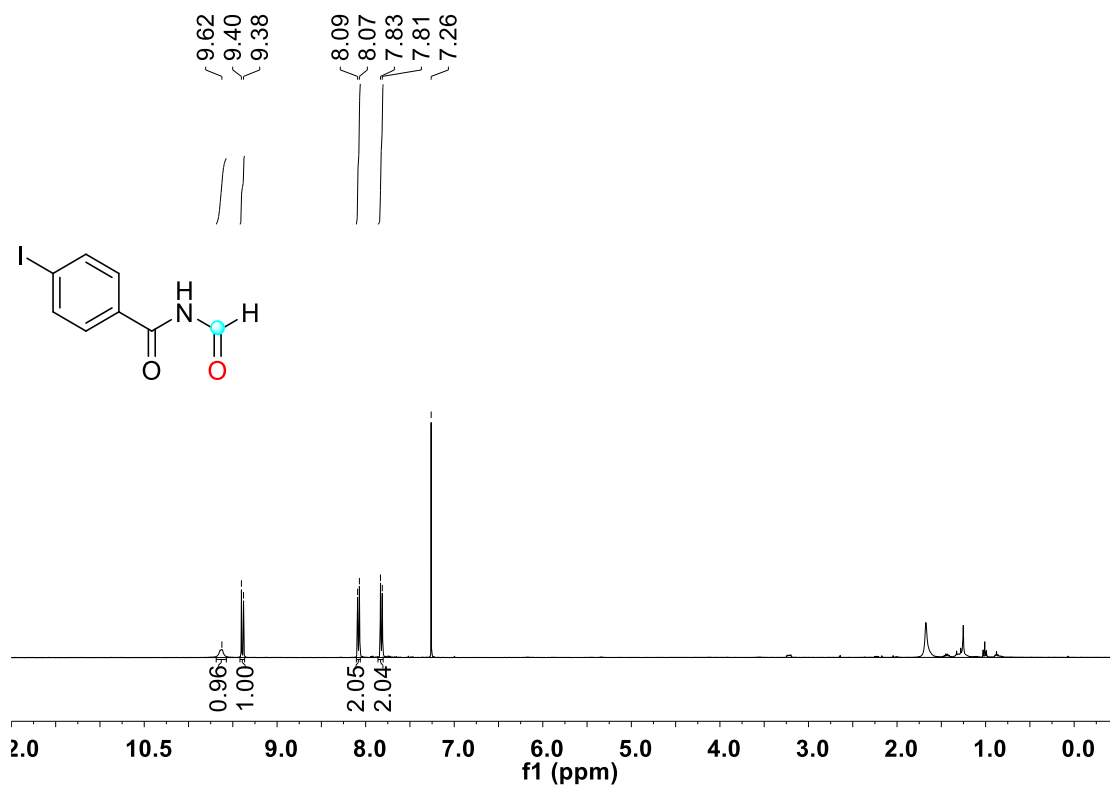
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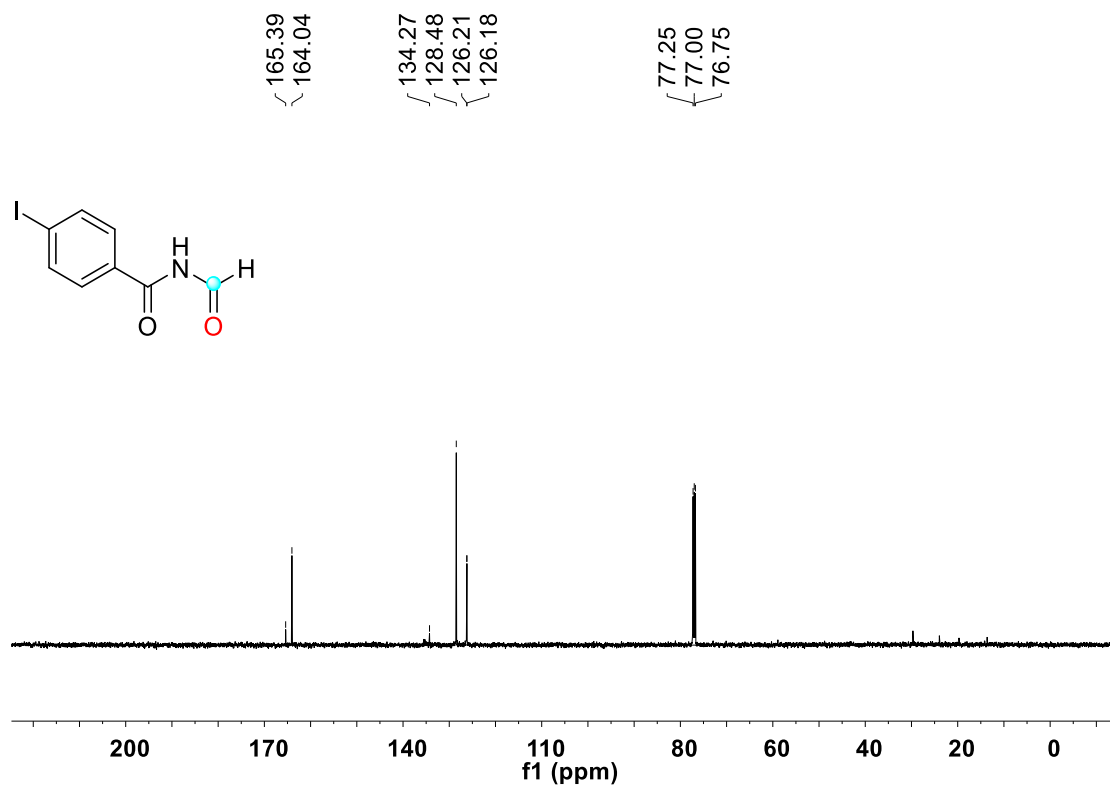
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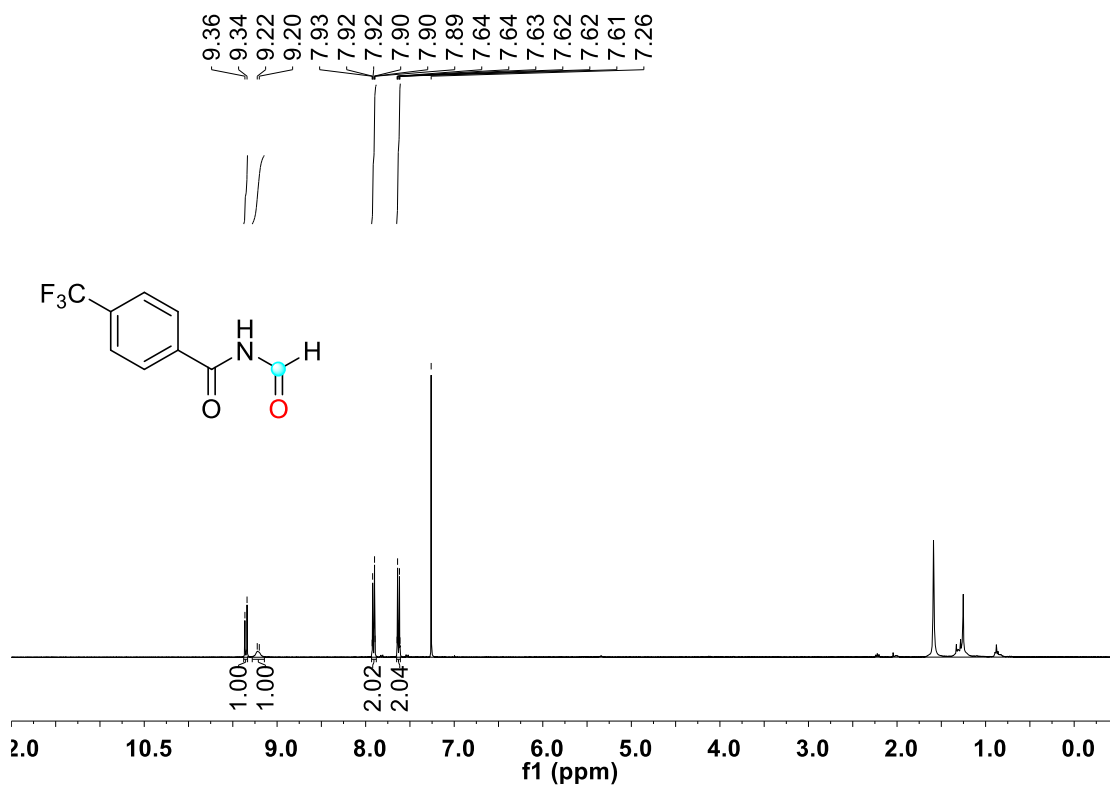
¹H NMR (400 MHz, CDCl₃) spectra for compound **2k**



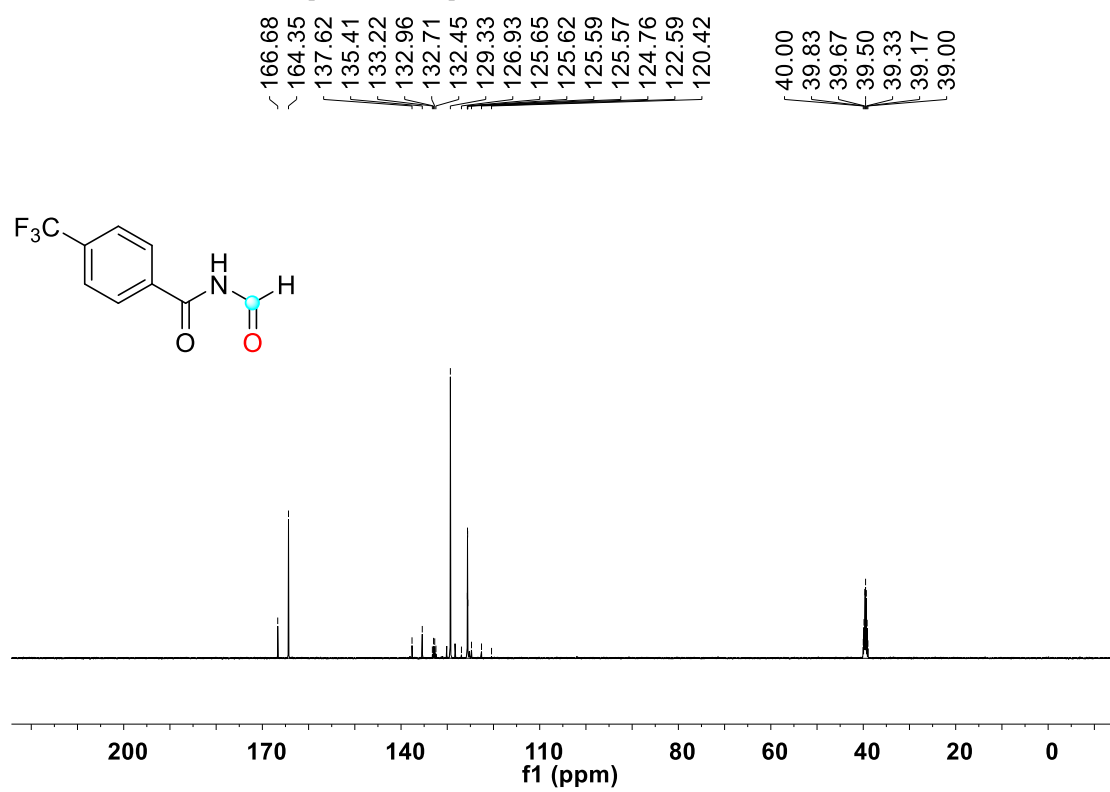
^{13}C NMR (126 MHz, CDCl_3) spectra for compound **2k**



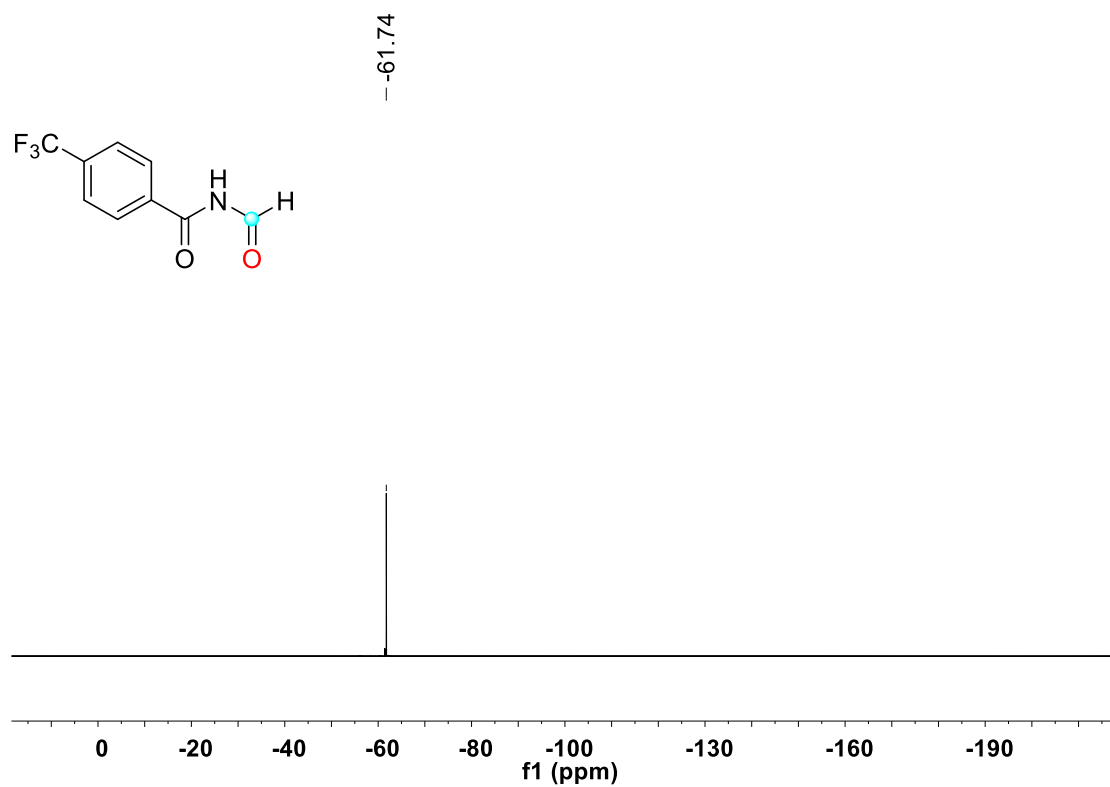
^1H NMR (400 MHz, CDCl_3) spectra for compound **2l**



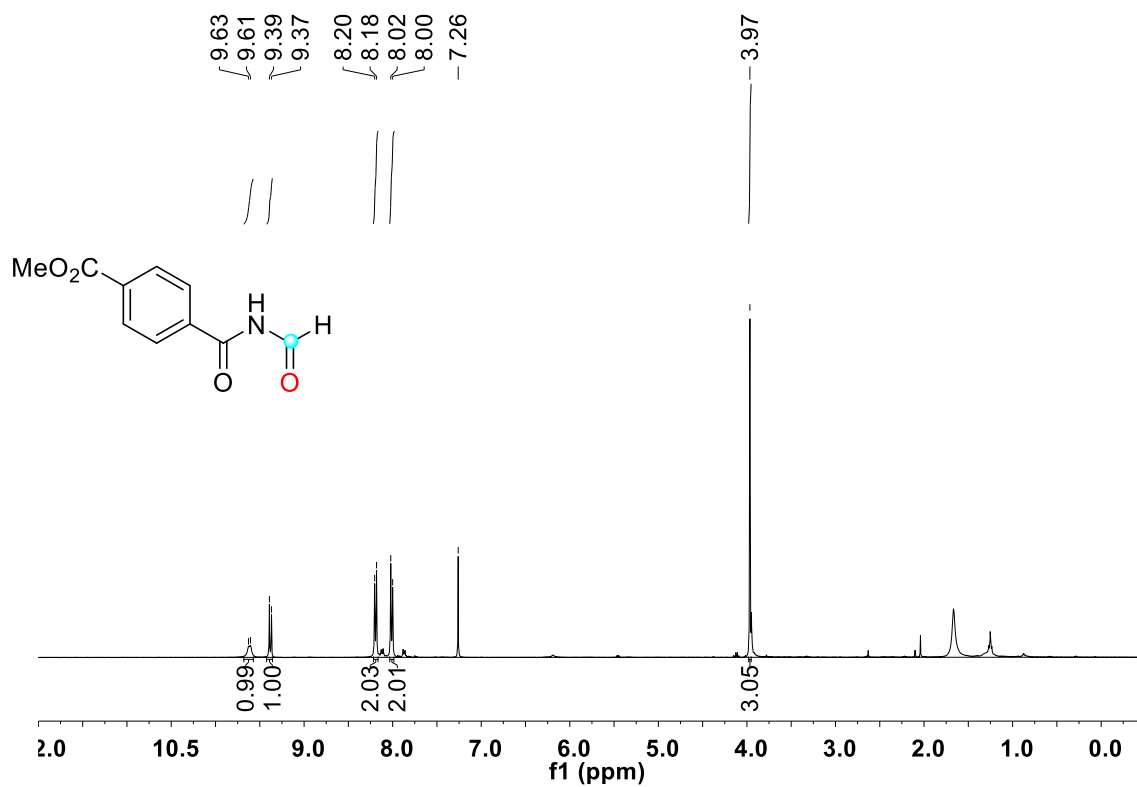
¹³C NMR (126 MHz, CDCl₃) spectra for compound **21**



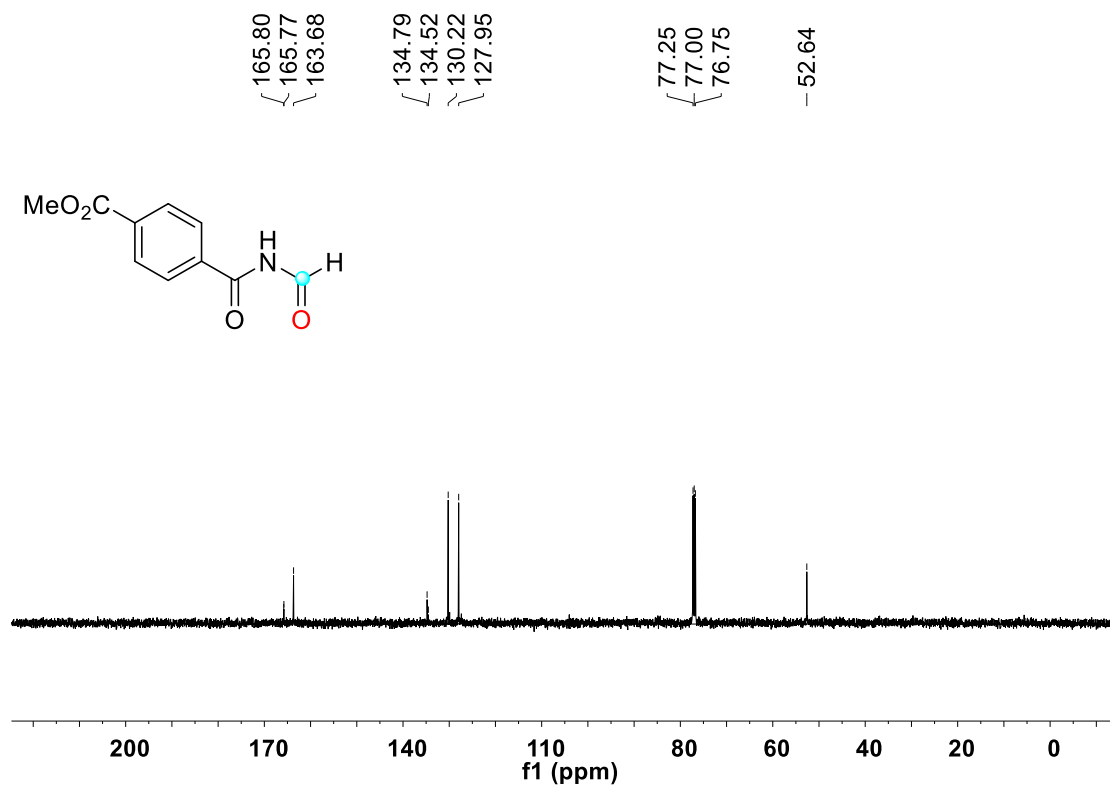
¹⁹F NMR (377 MHz, CDCl₃) spectra for compound **21**



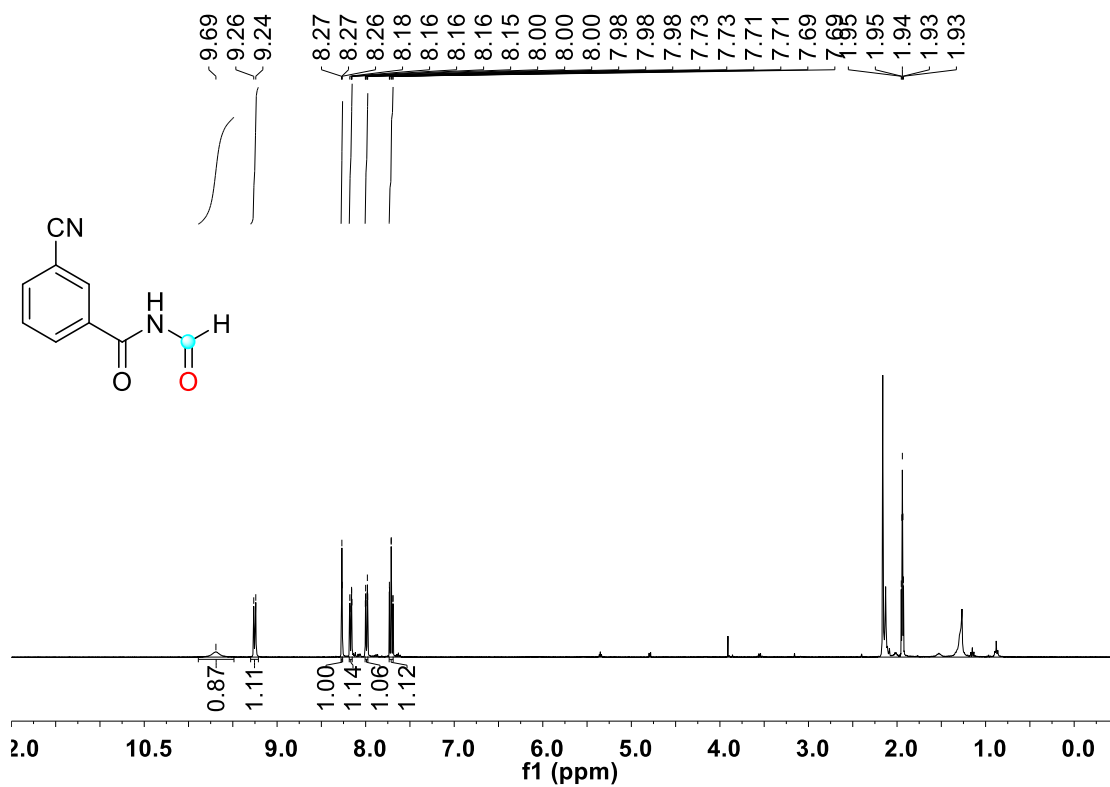
¹H NMR (400 MHz, CDCl₃) spectra for compound **2m**



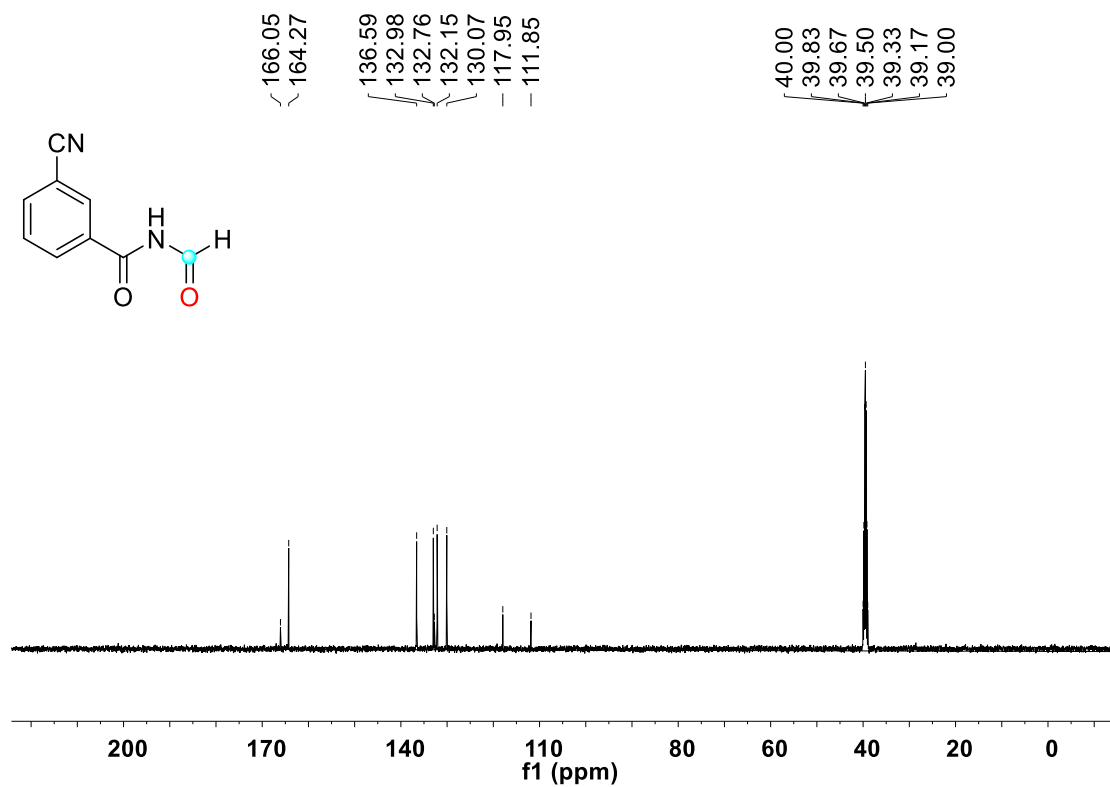
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2m**



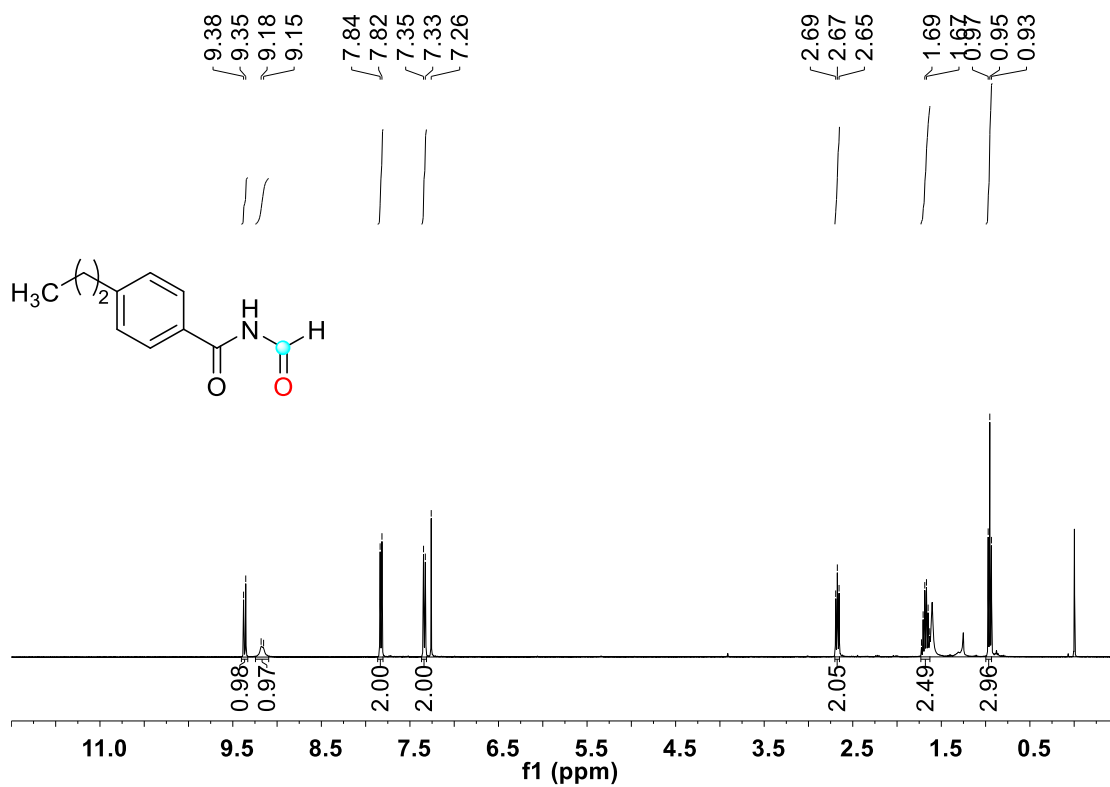
¹H NMR (400 MHz, CDCl₃) spectra for compound **2n**



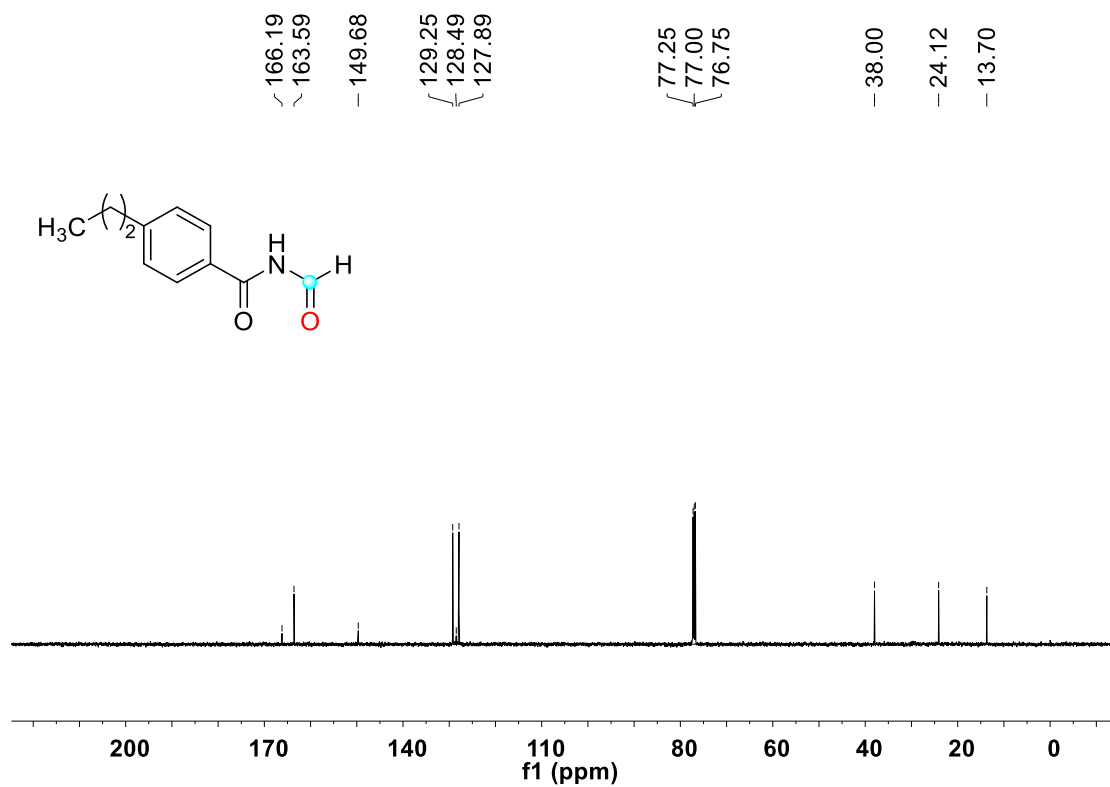
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2n**



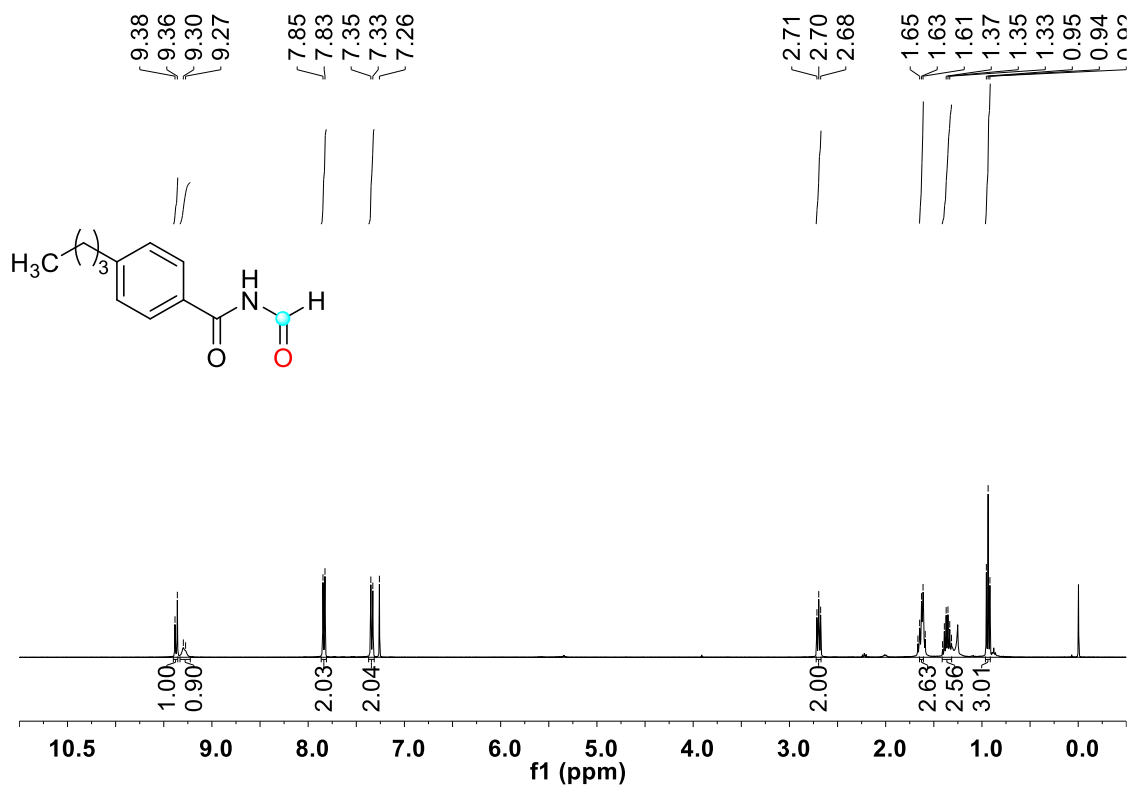
¹H NMR (400 MHz, CDCl₃) spectra for compound **2o**



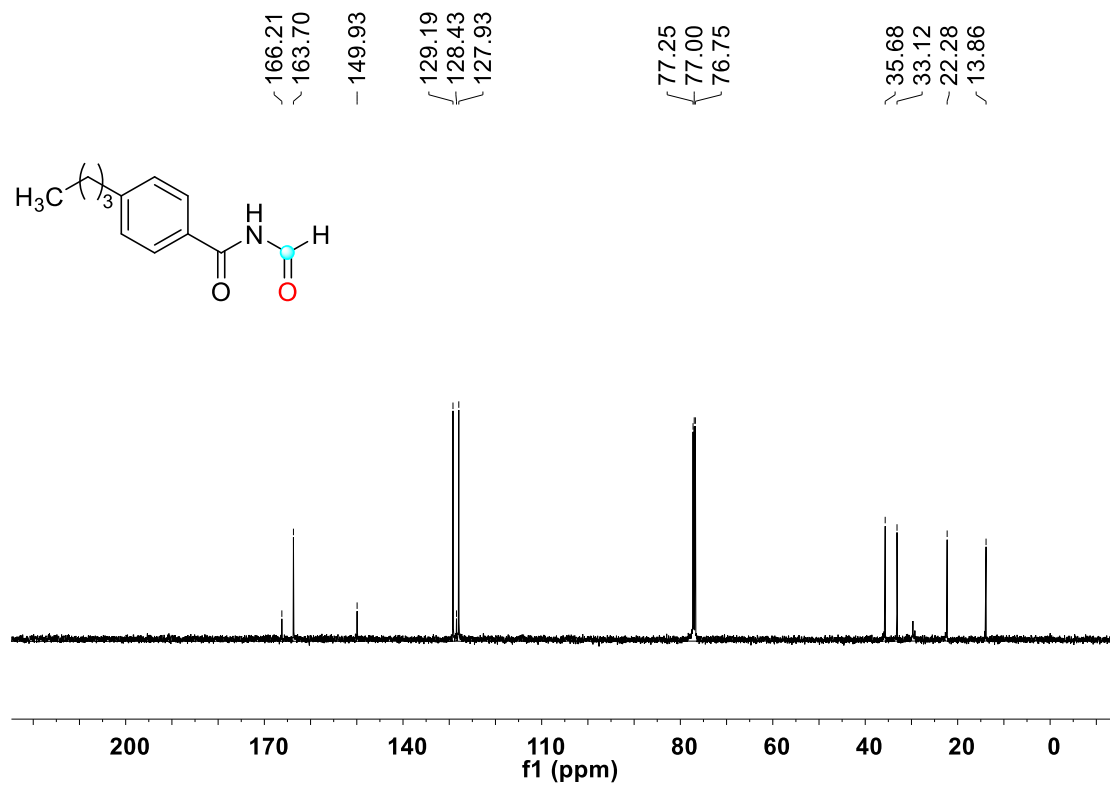
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2o**



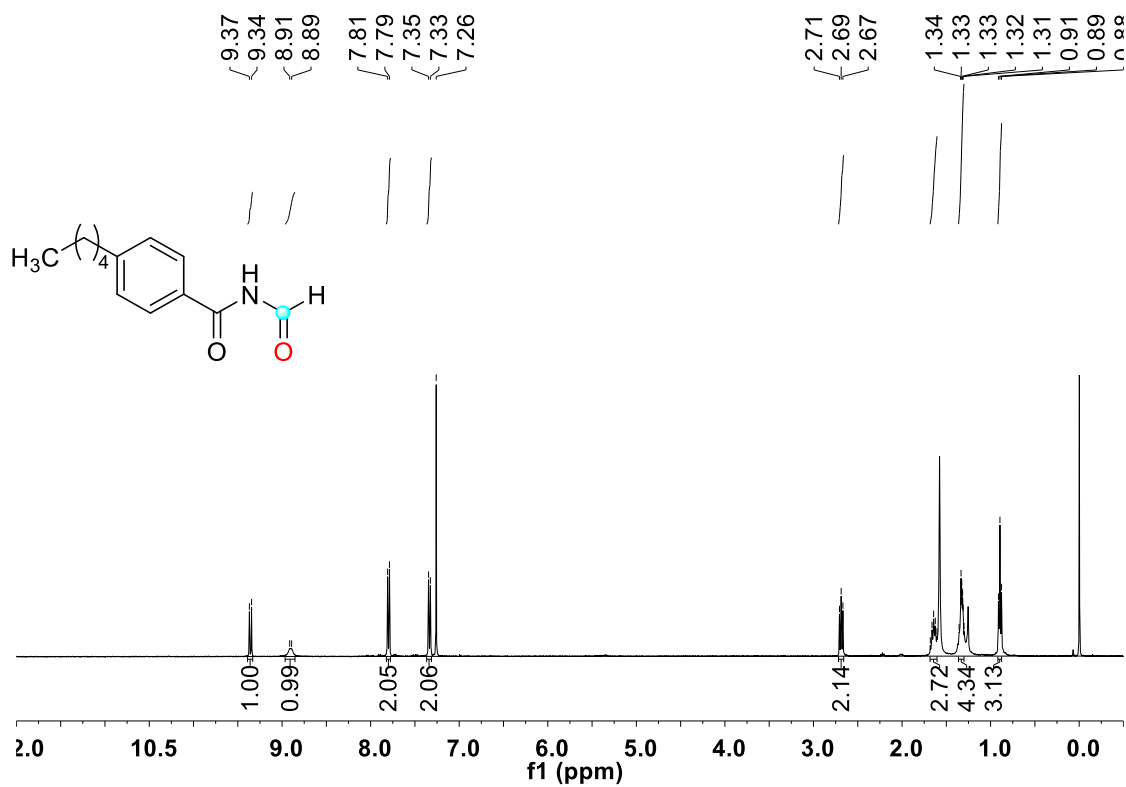
¹H NMR (400 MHz, CDCl₃) spectra for compound **2p**



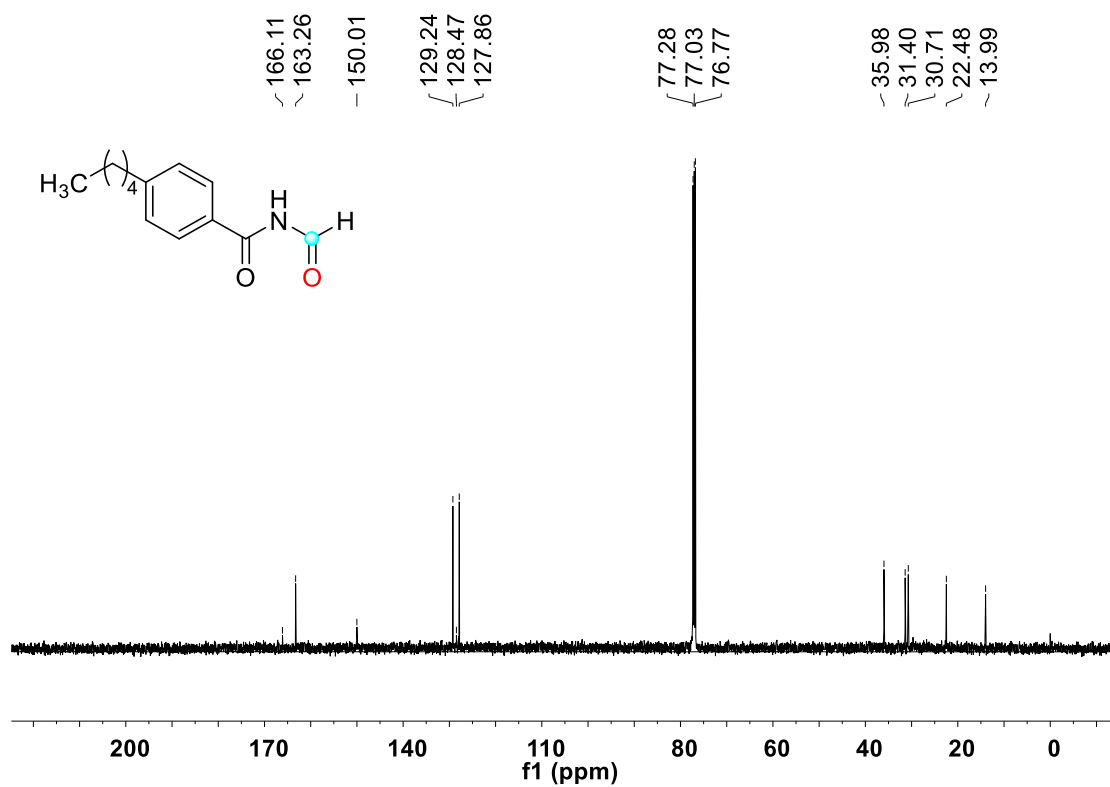
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2p**



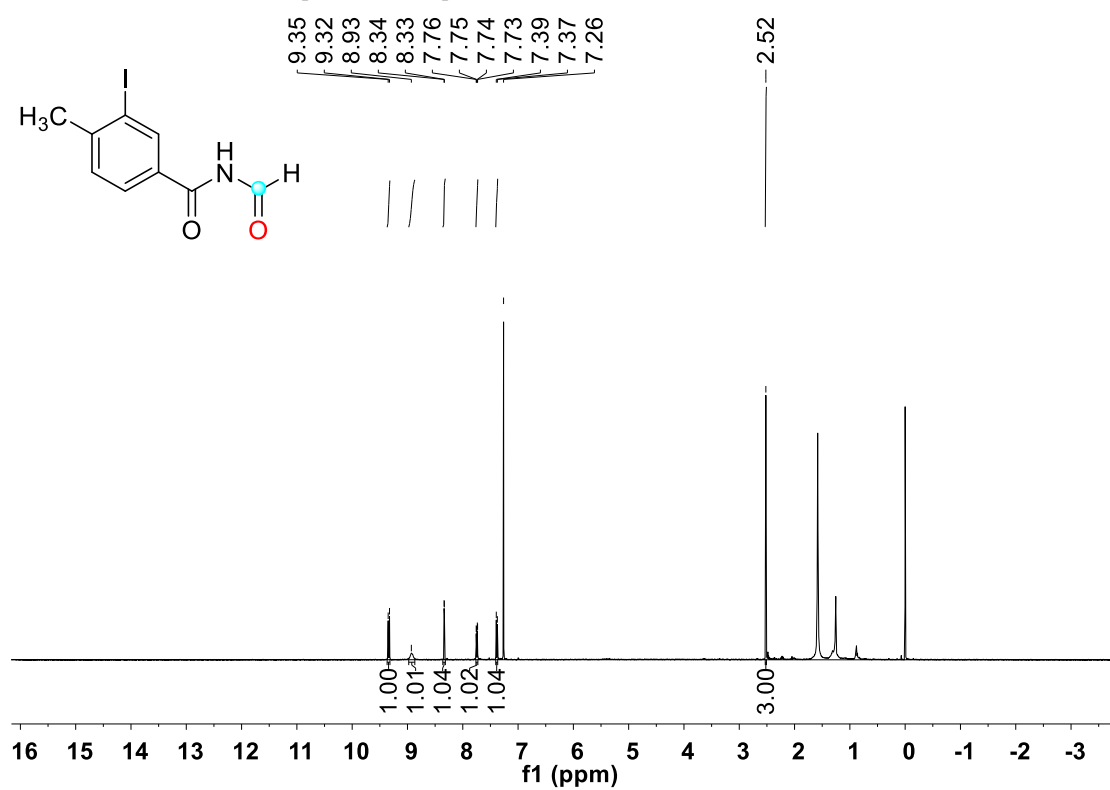
¹H NMR (400 MHz, CDCl₃) spectra for compound **2q**



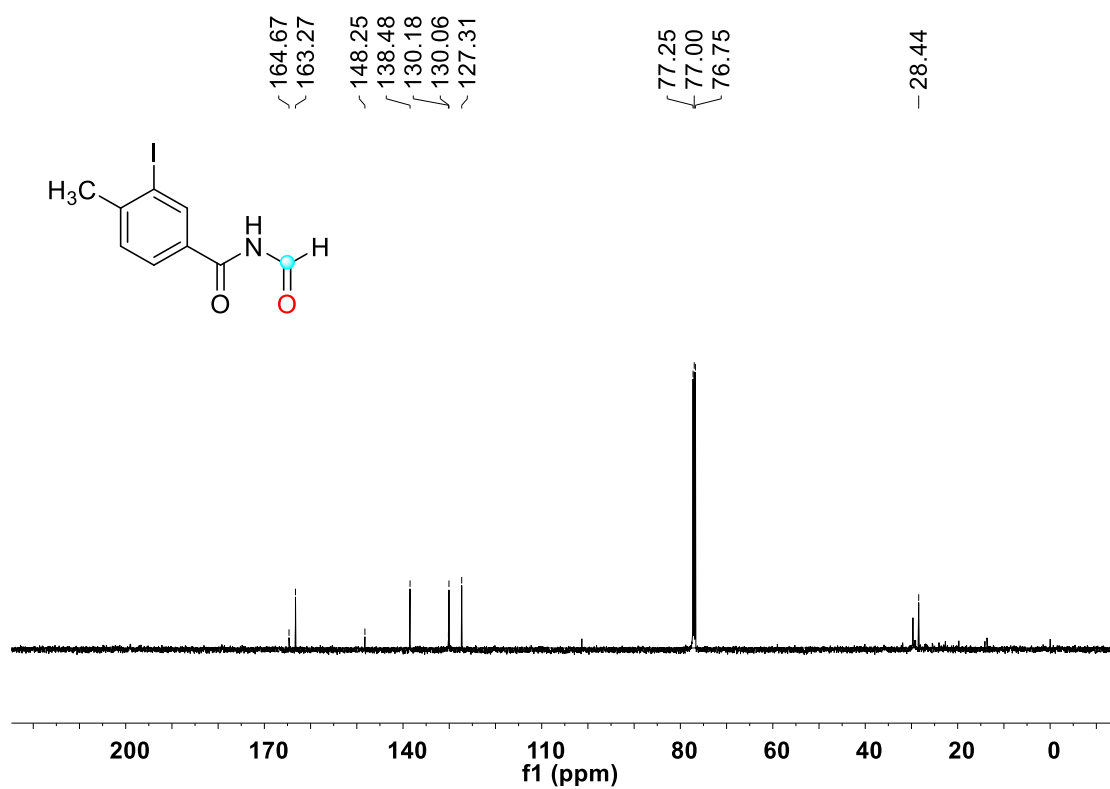
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2q**



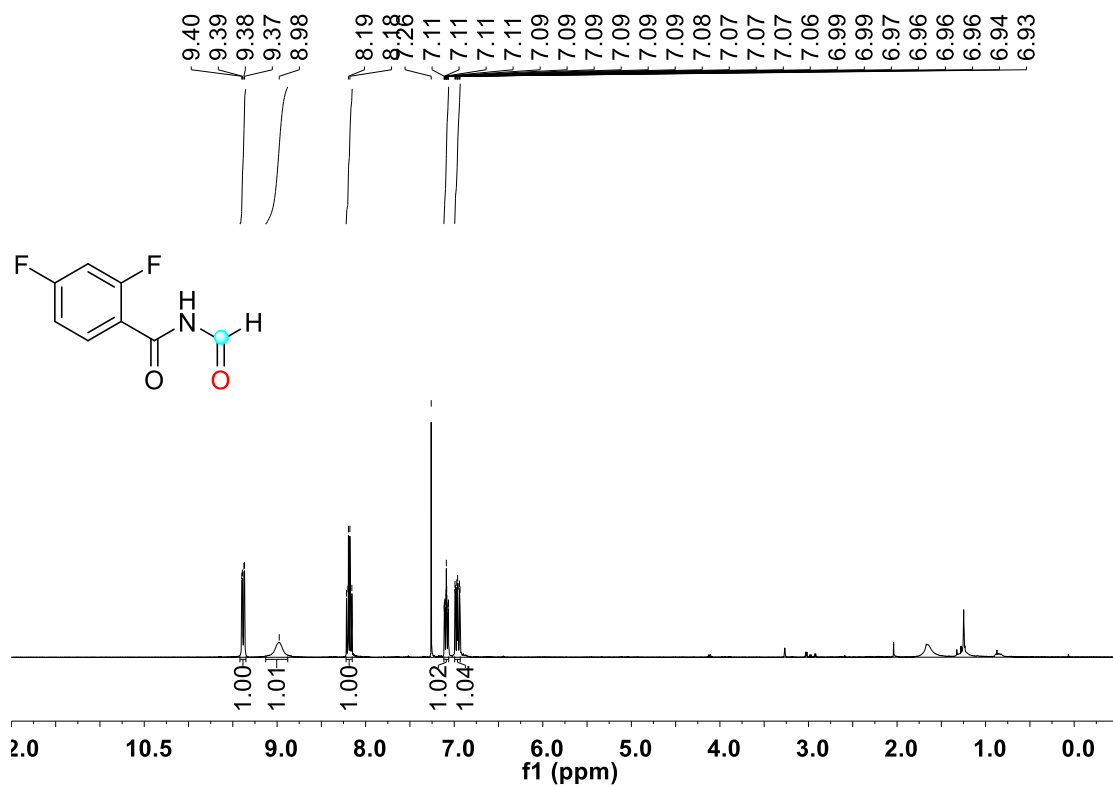
¹H NMR (400 MHz, CDCl₃) spectra for compound **2r**



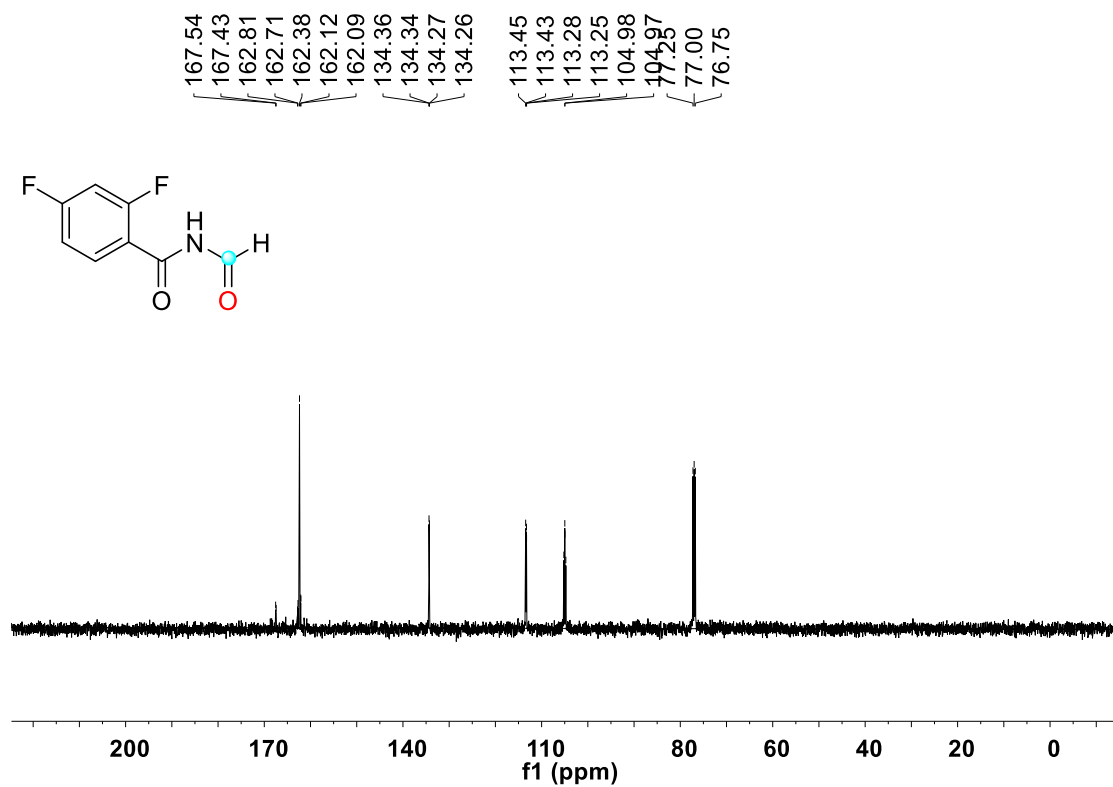
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2r**



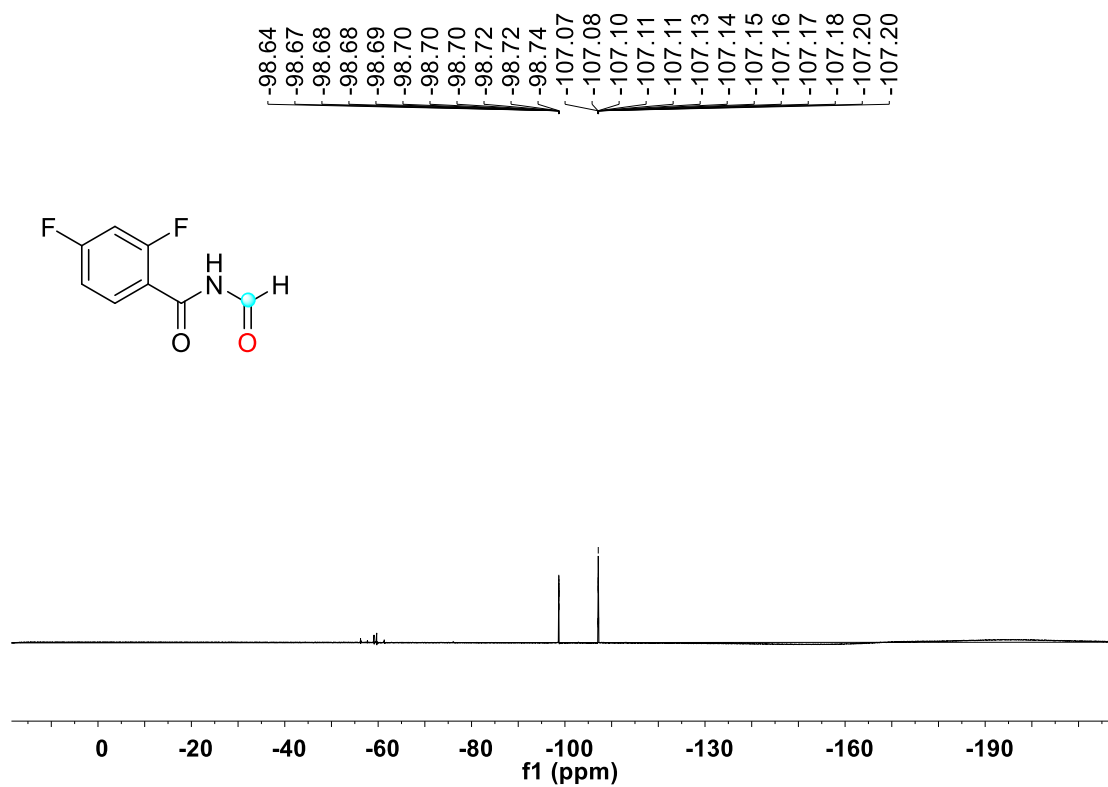
¹H NMR (400 MHz, CDCl₃) spectra for compound 2s



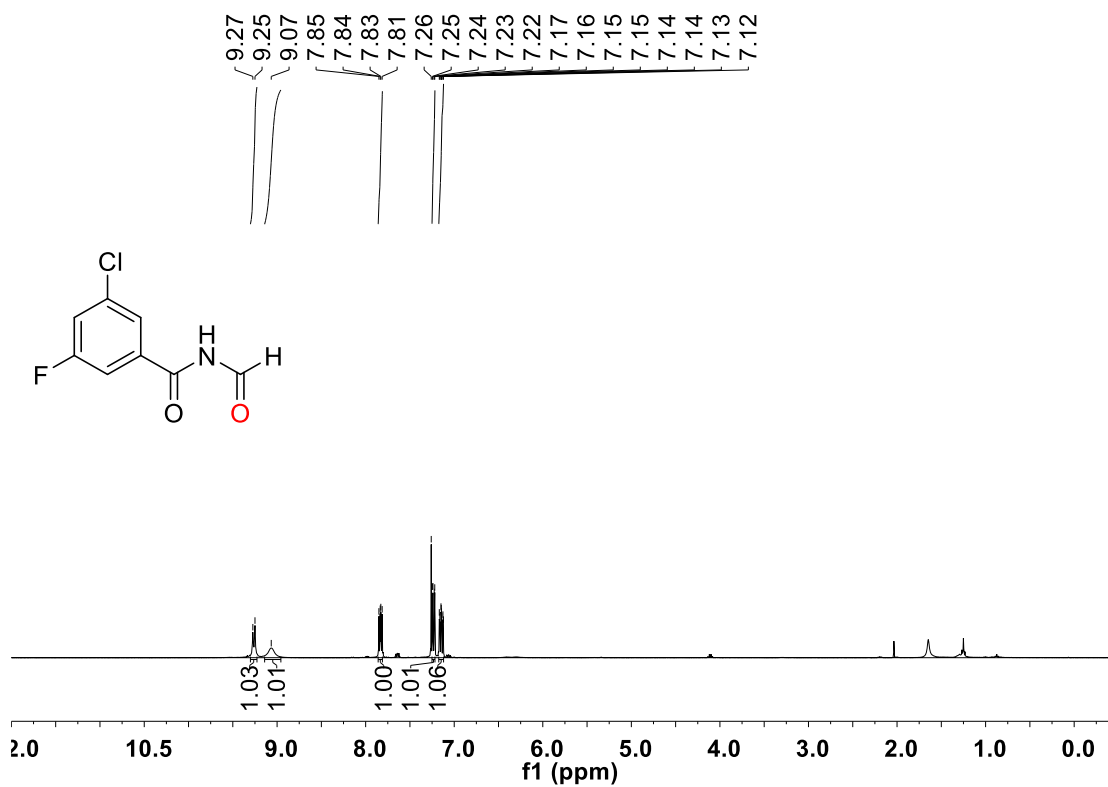
¹³C NMR (126 MHz, CDCl₃) spectra for compound 2s



^{19}F NMR (377 MHz, CDCl_3) spectra for compound **2s**

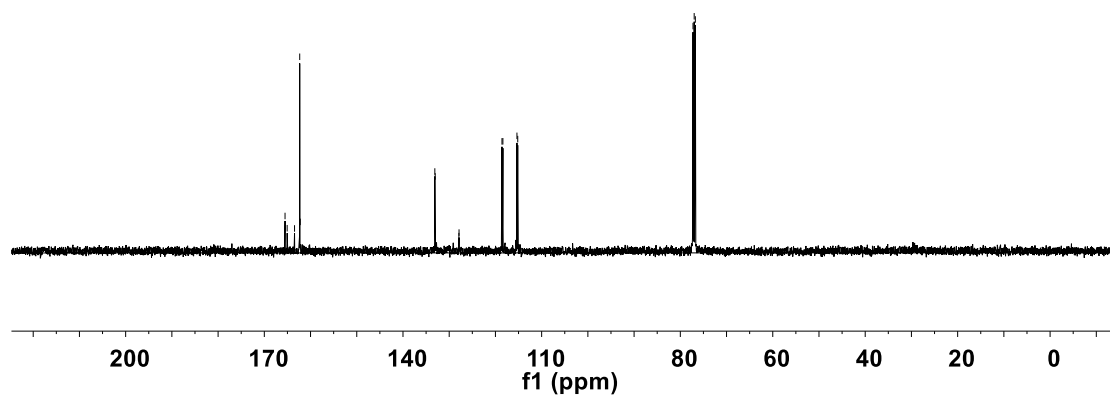
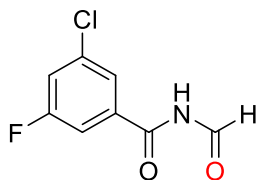


^1H NMR (400 MHz, CDCl_3) spectra for compound **2t**



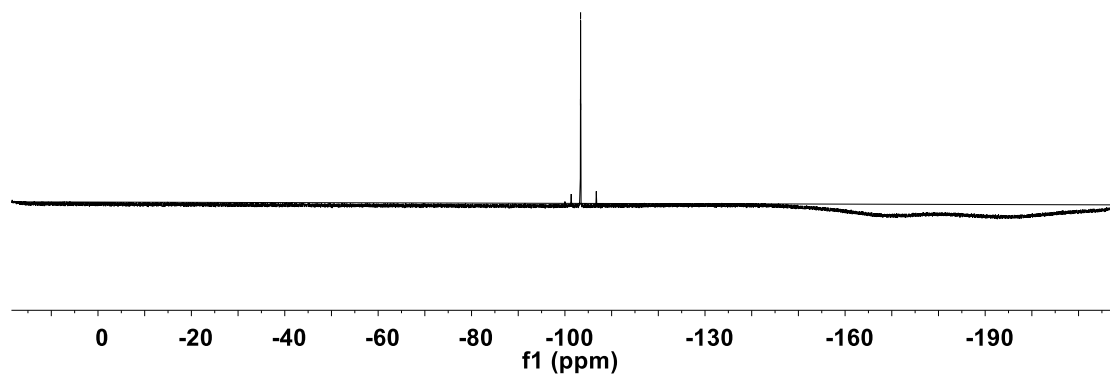
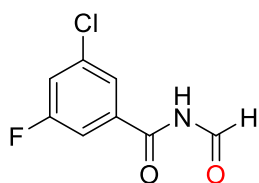
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2t**

165.52
165.06
163.46
162.34
162.23
133.11
133.03
127.90
127.87
118.58
118.38
115.34
115.17
77.25
77.00
76.75

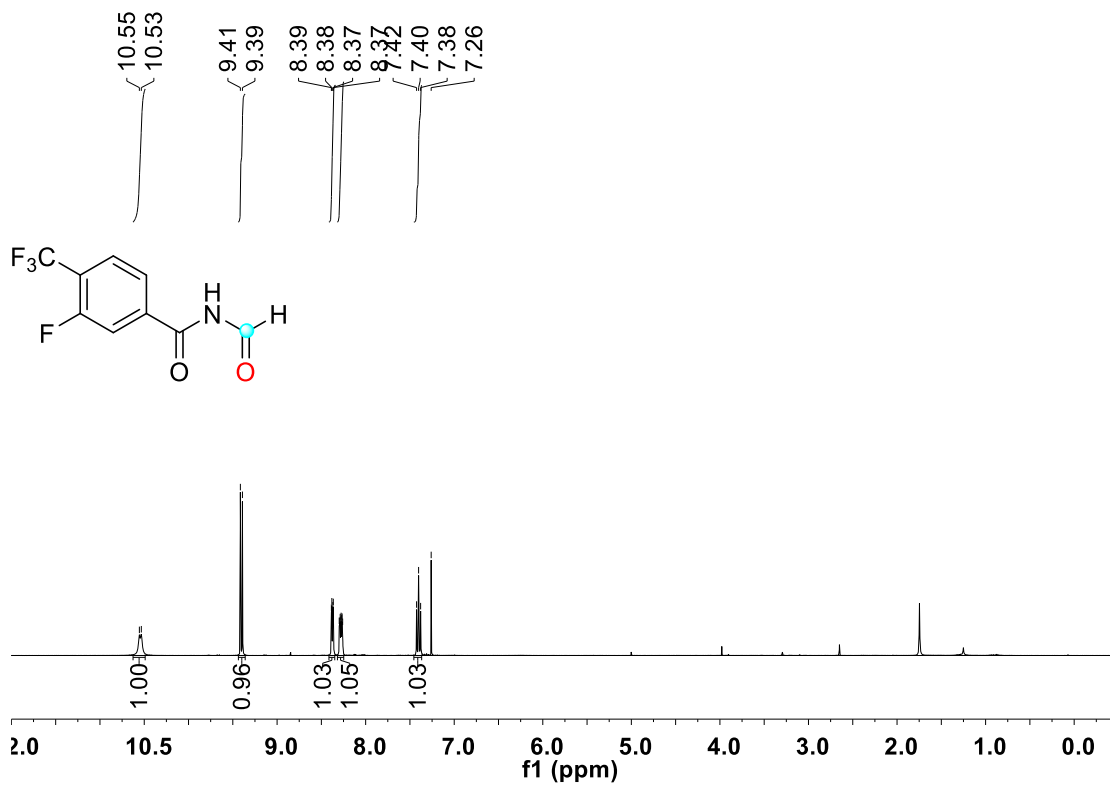


¹⁹F NMR (377 MHz, CDCl₃) spectra for compound **2t**

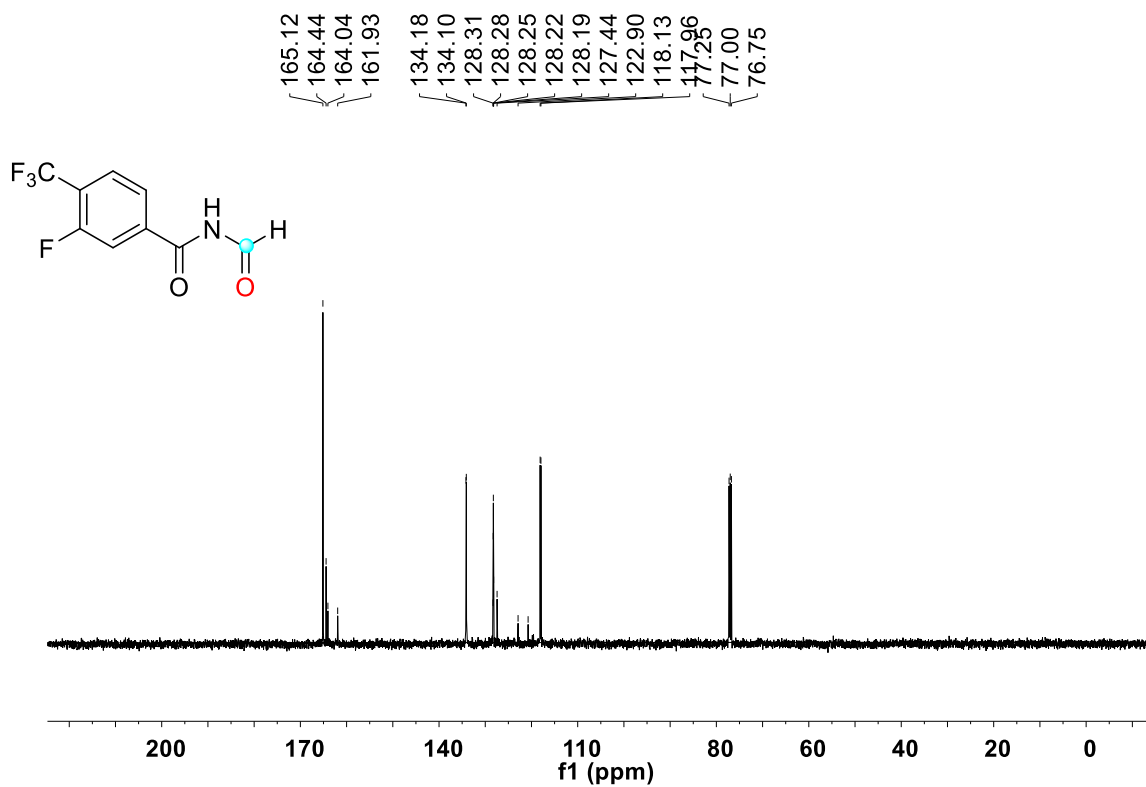
-103.32
-103.34
-103.36
-103.38



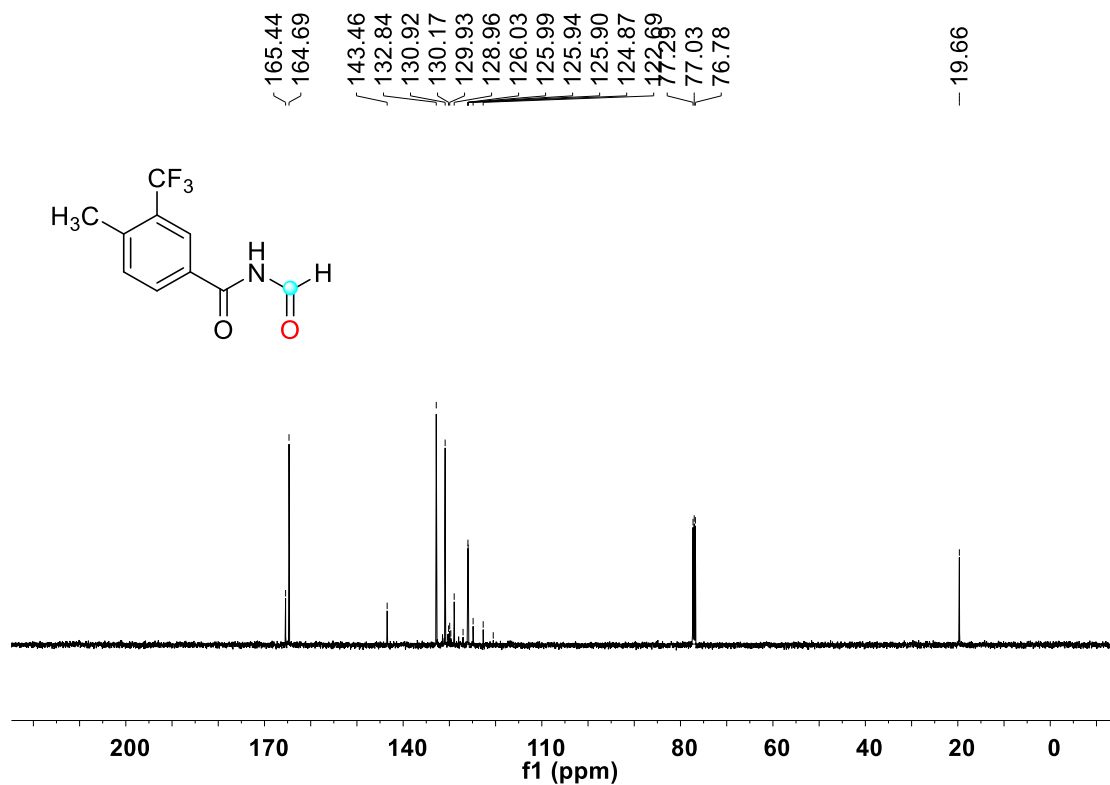
¹H NMR (400 MHz, CDCl₃) spectra for compound **2u**



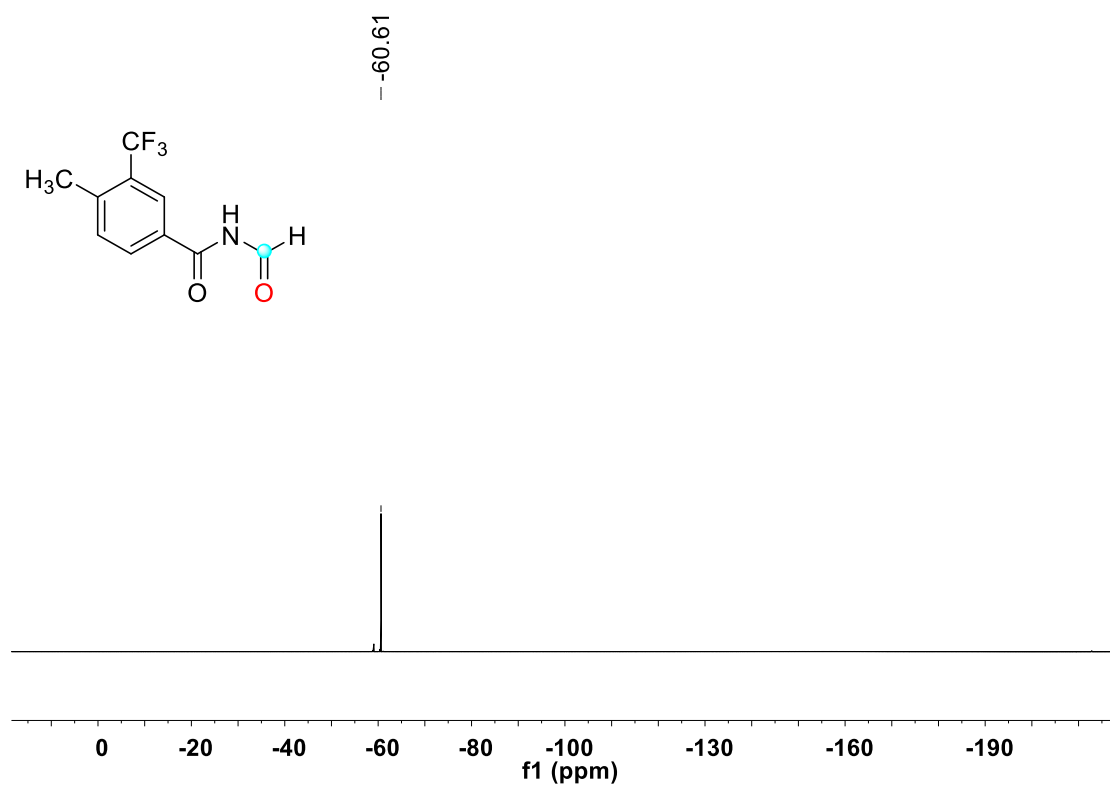
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2u**



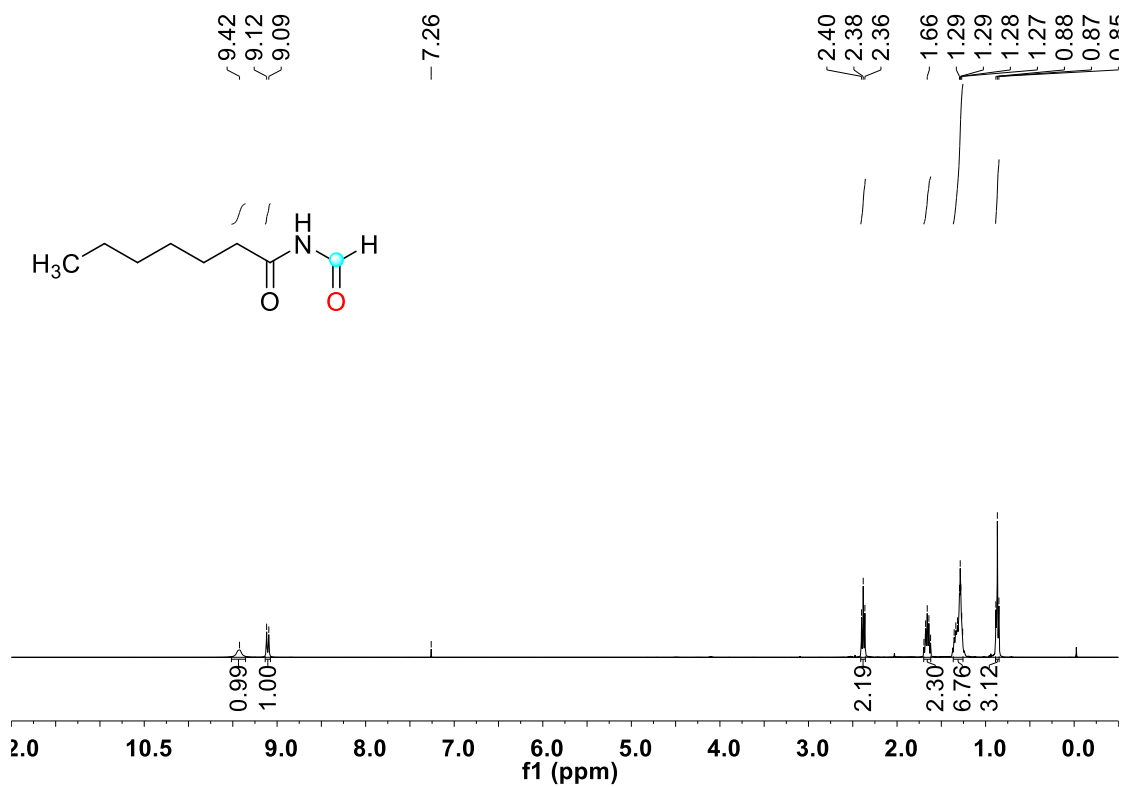
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2v**



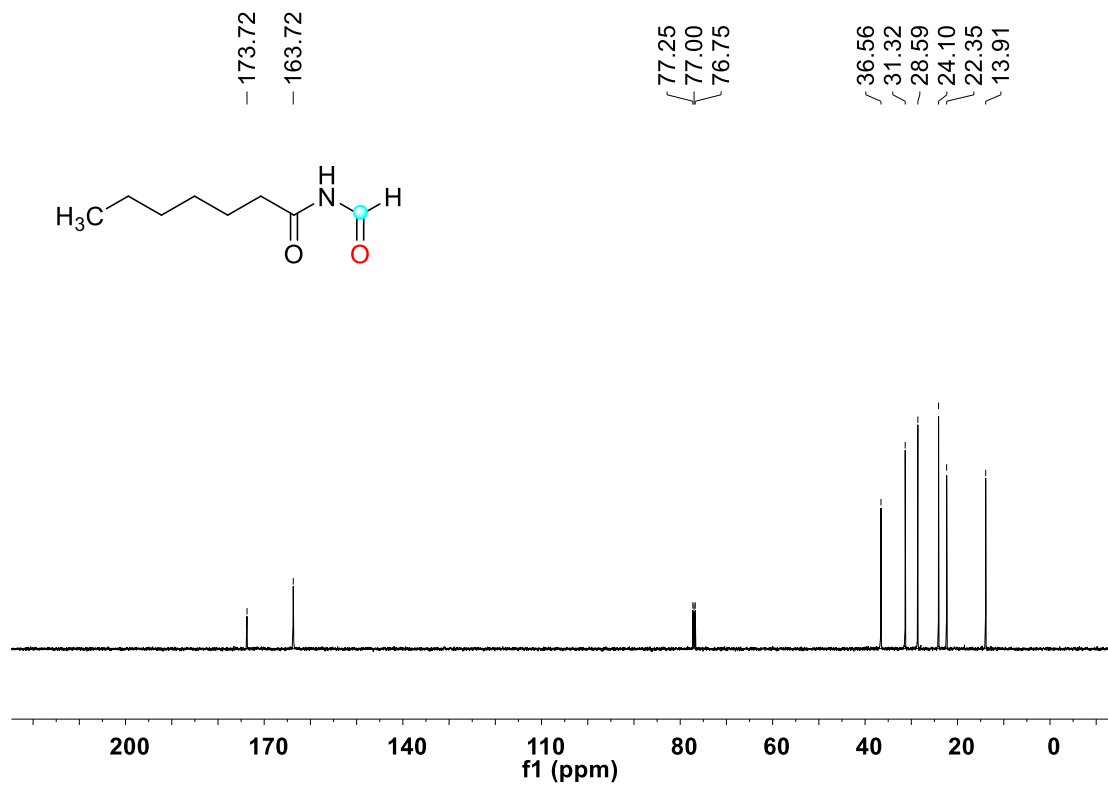
¹⁹F NMR (377 MHz, CDCl₃) spectra for compound **2v**



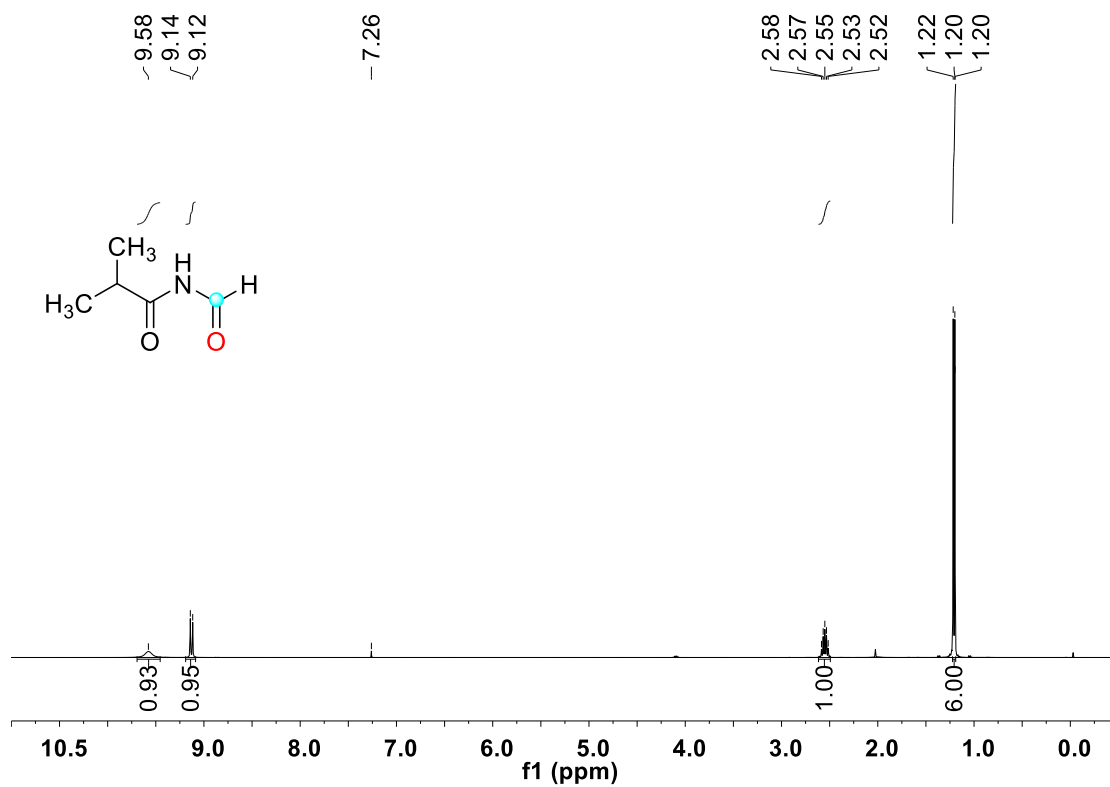
¹H NMR (400 MHz, CDCl₃) spectra for compound 2x



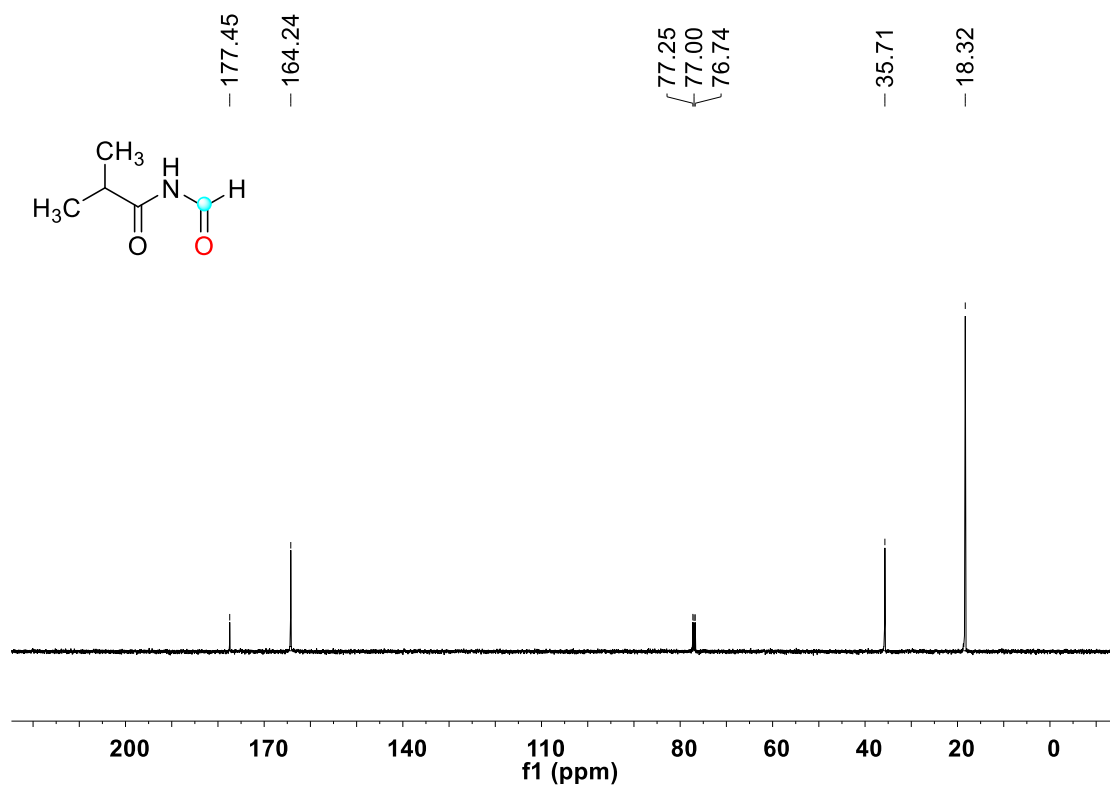
¹³C NMR (126 MHz, CDCl₃) spectra for compound 2x



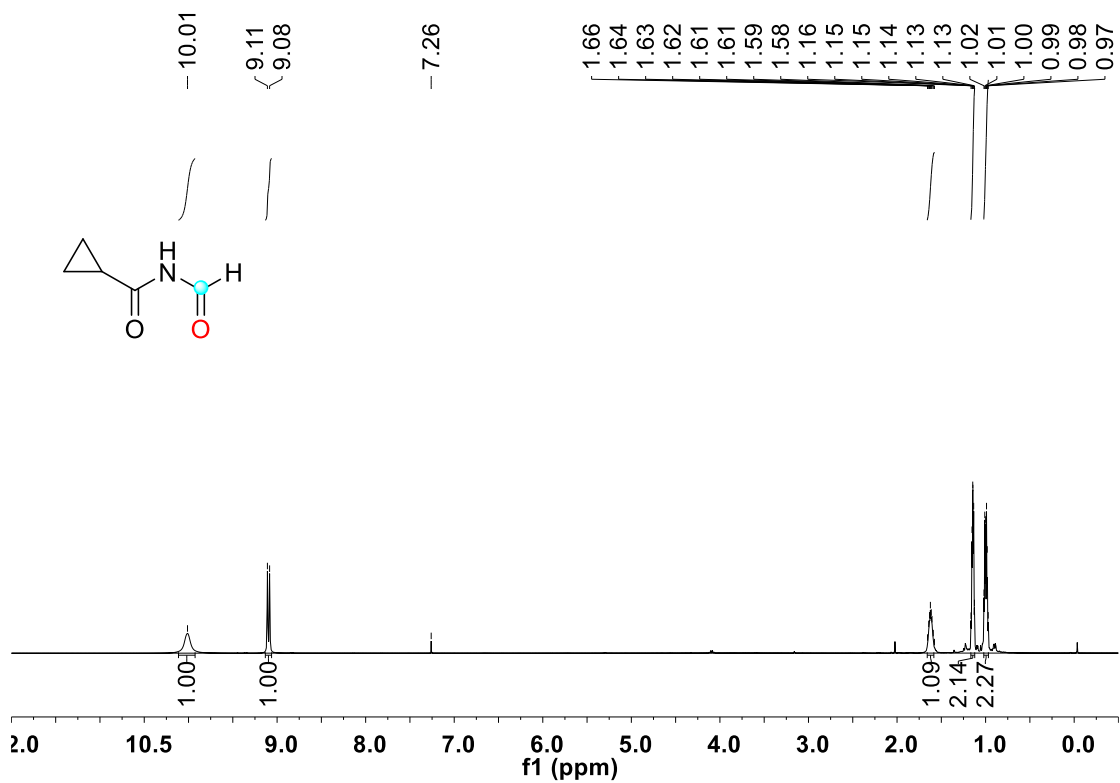
¹H NMR (400 MHz, CDCl₃) spectra for compound **2y**



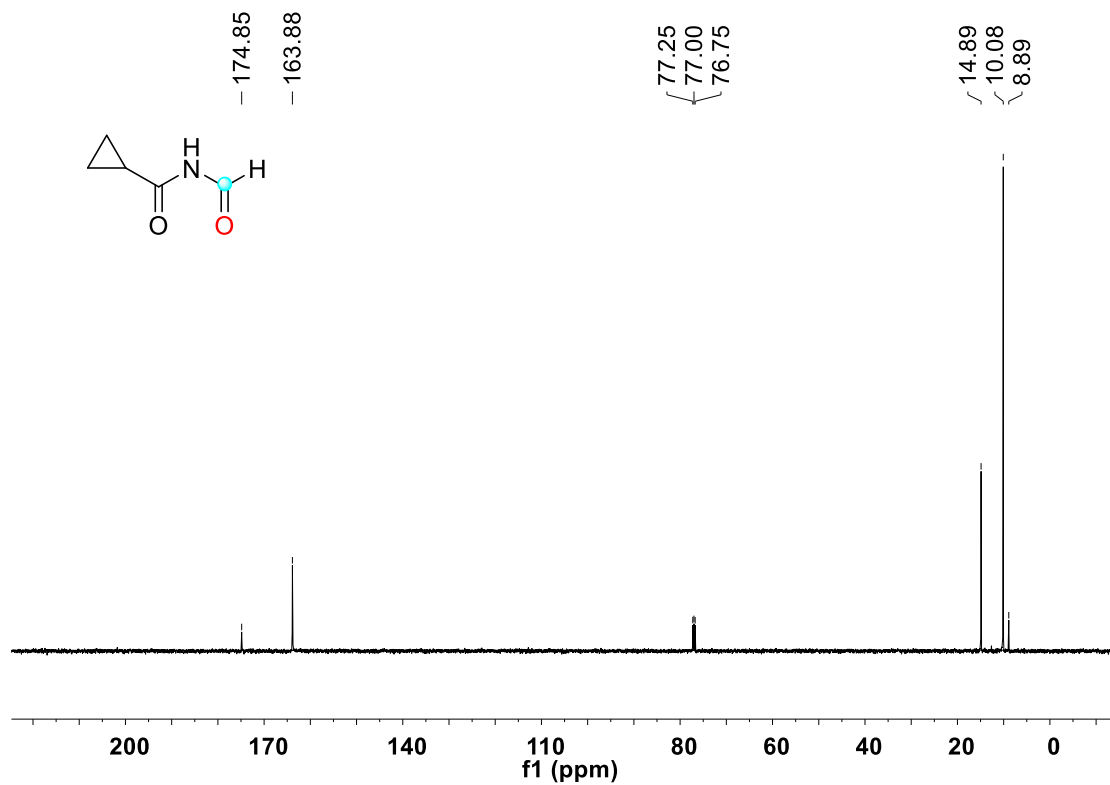
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2y**



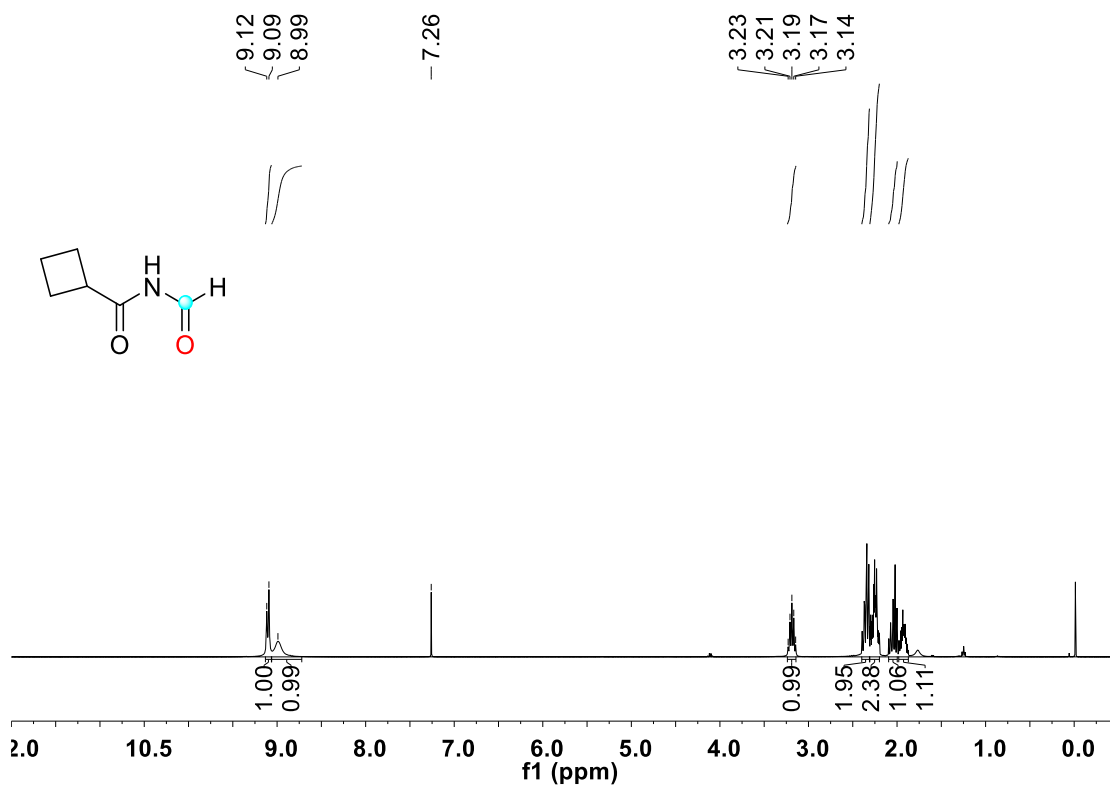
¹H NMR (400 MHz, CDCl₃) spectra for compound **2z**



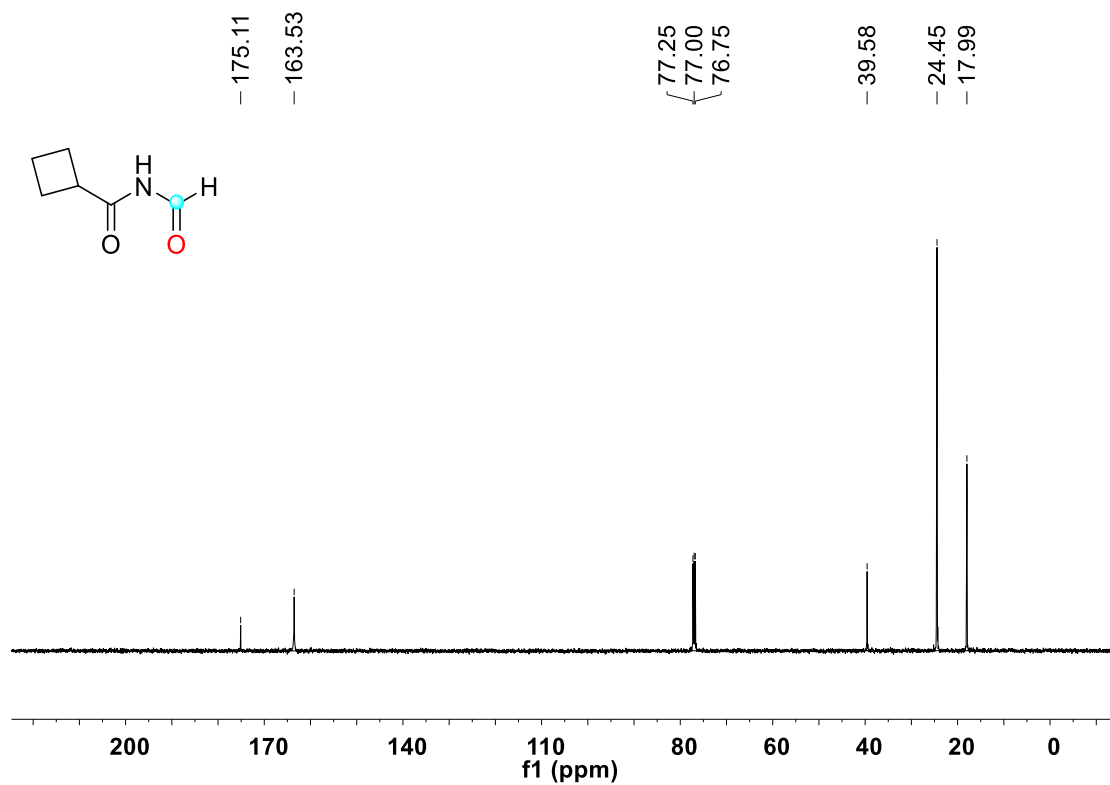
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2z**



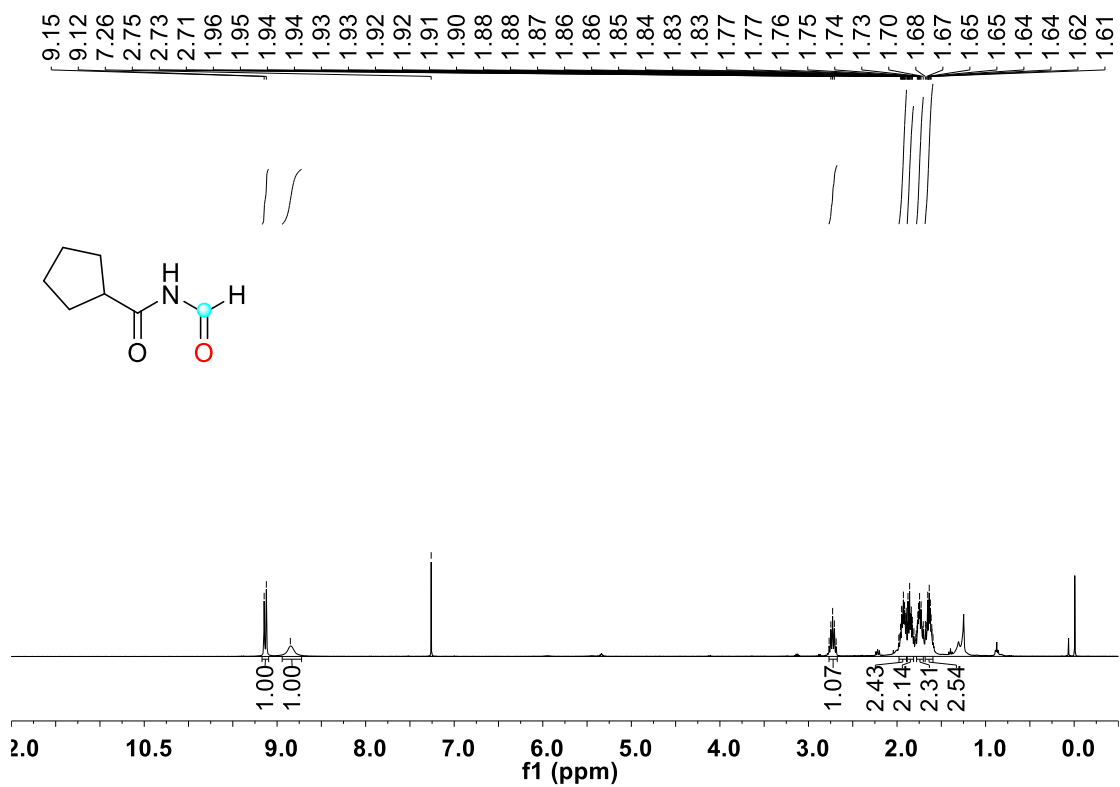
¹H NMR (400 MHz, CDCl₃) spectra for compound **2aa**



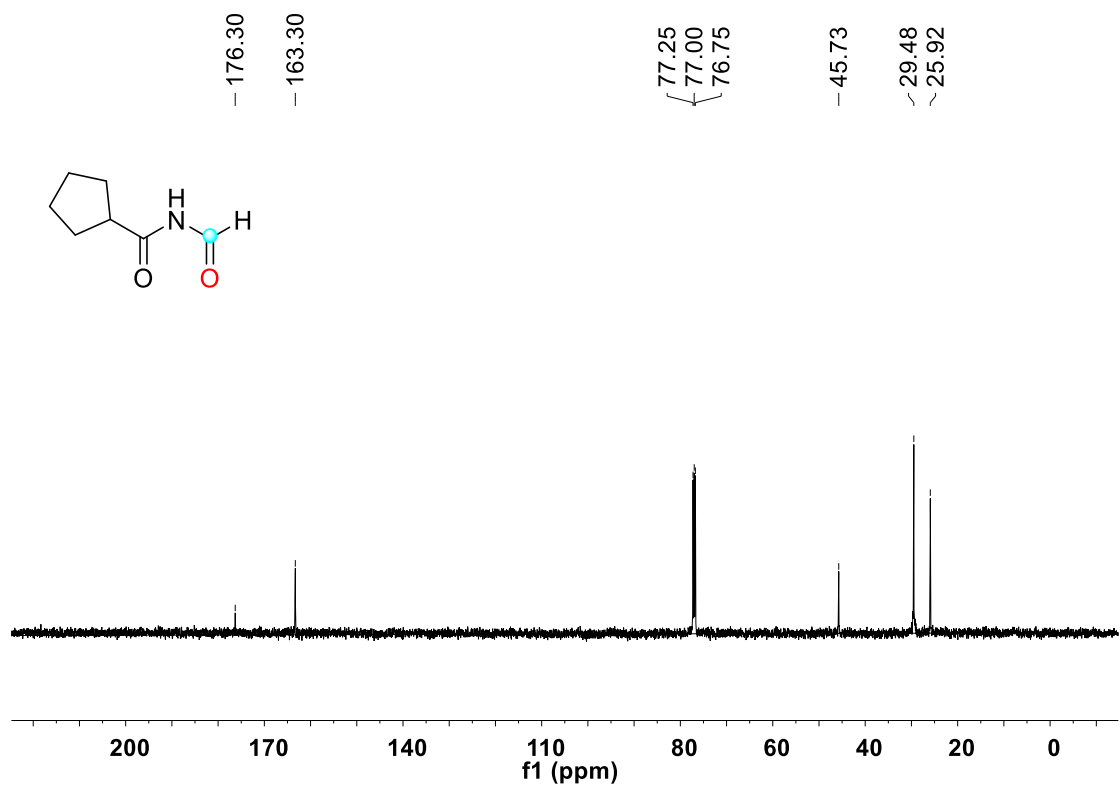
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2aa**



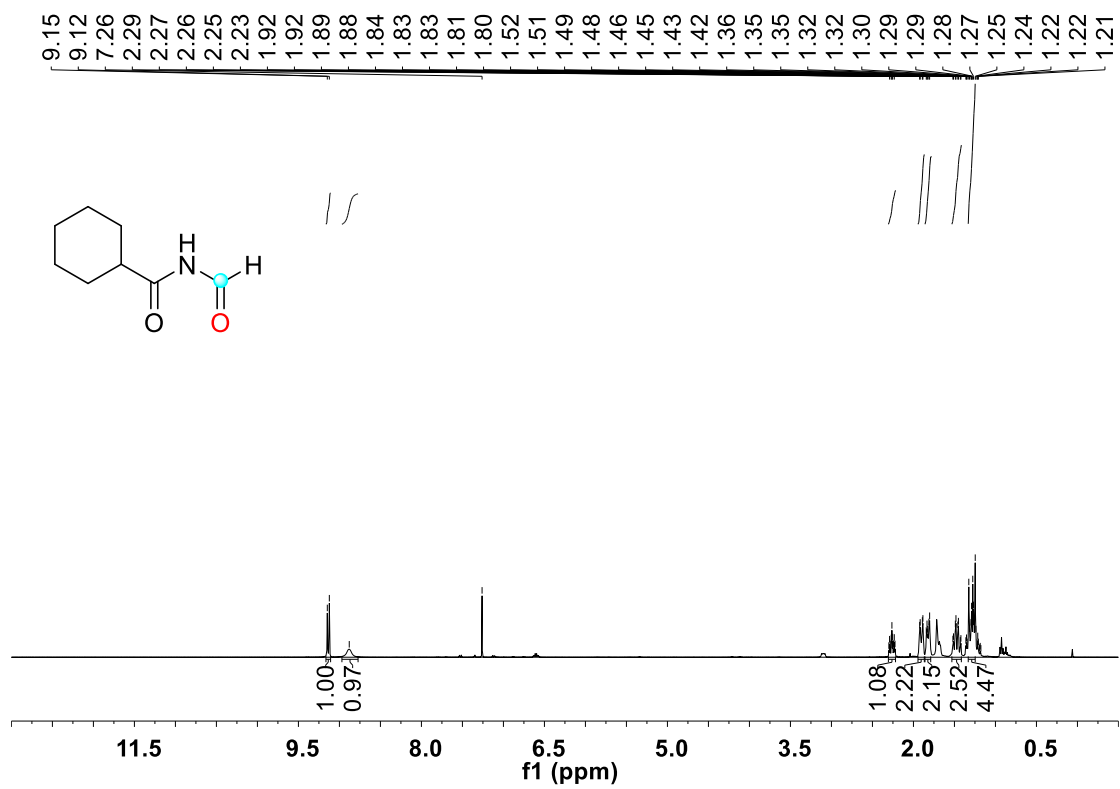
¹H NMR (400 MHz, CDCl₃) spectra for compound **2ab**



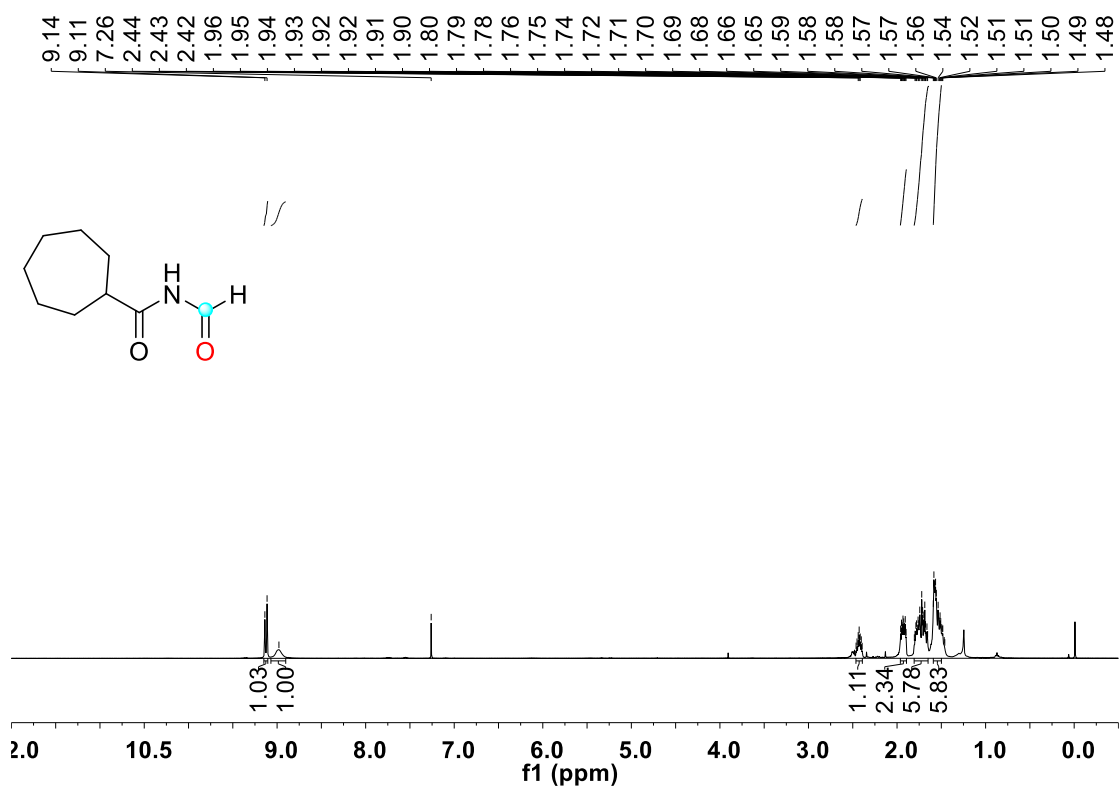
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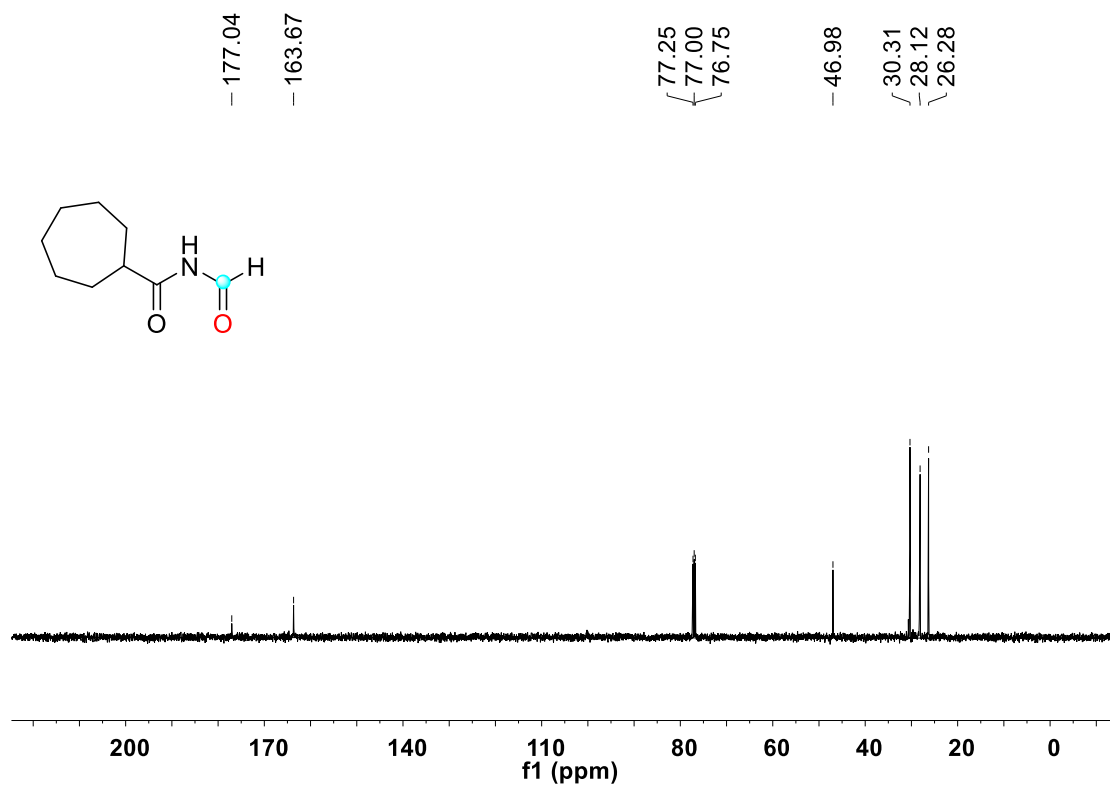
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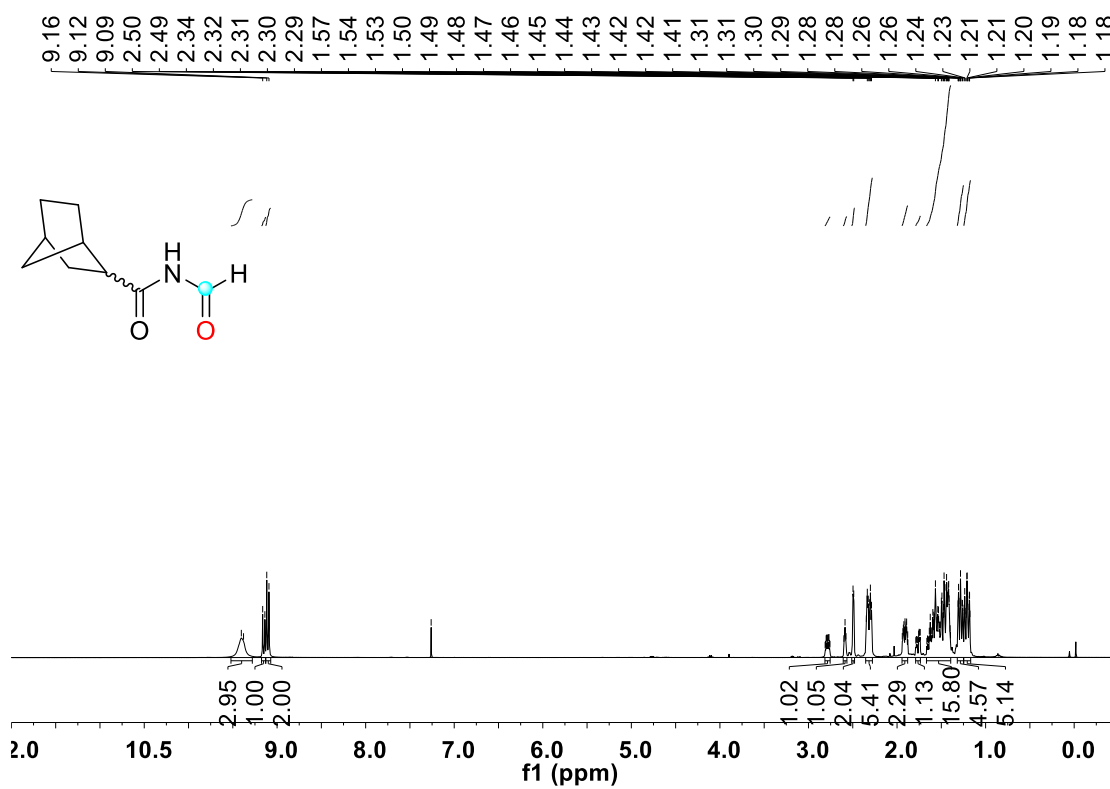
¹H NMR (400 MHz, CDCl₃) spectra for compound **2ad**



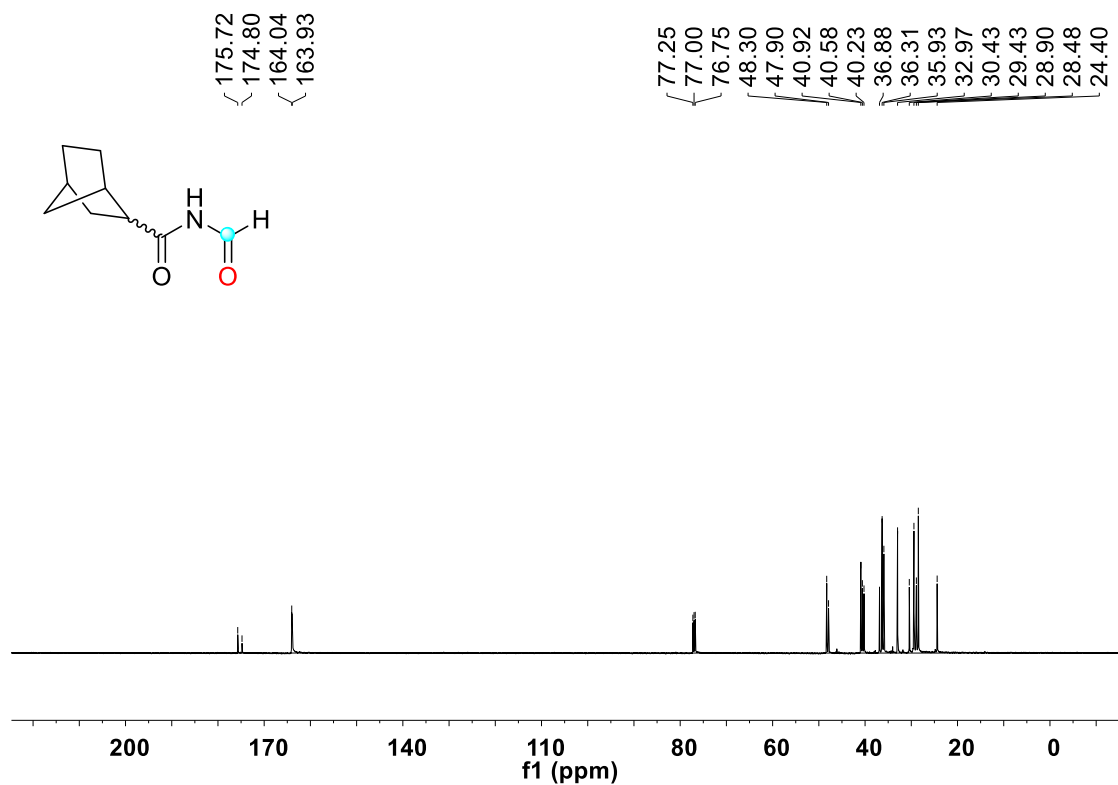
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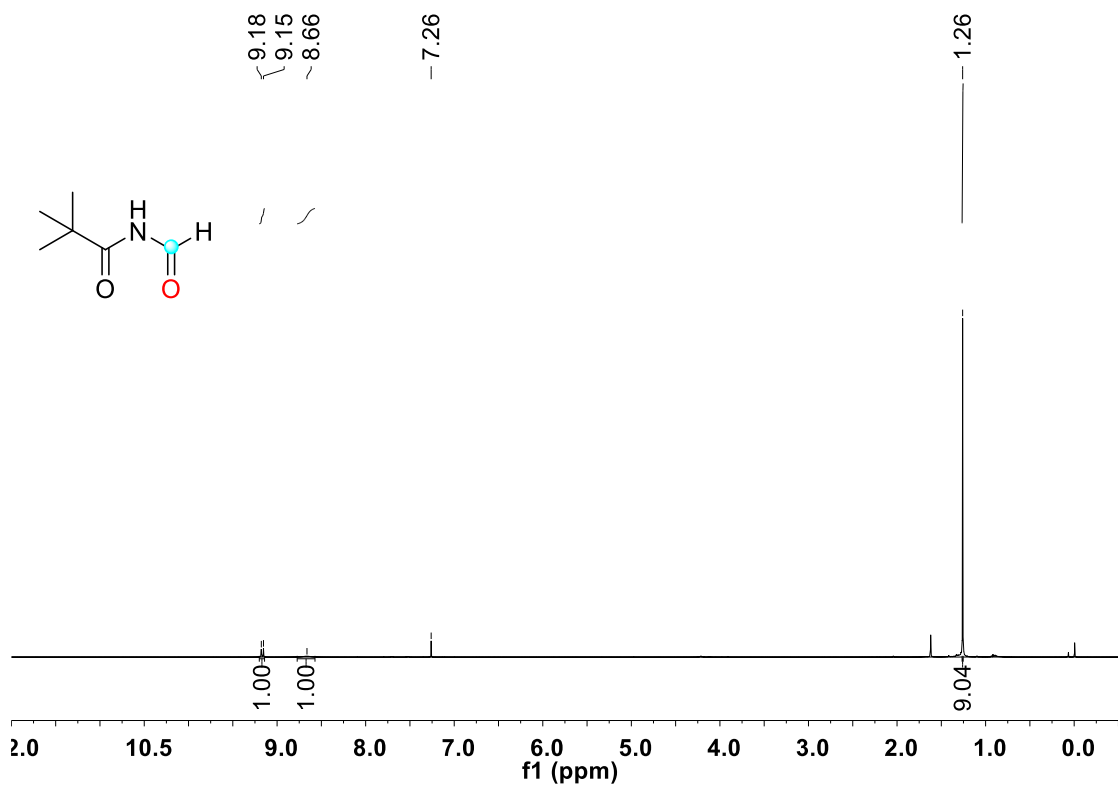
¹H NMR (400 MHz, CDCl₃) spectra for compound **2ae**



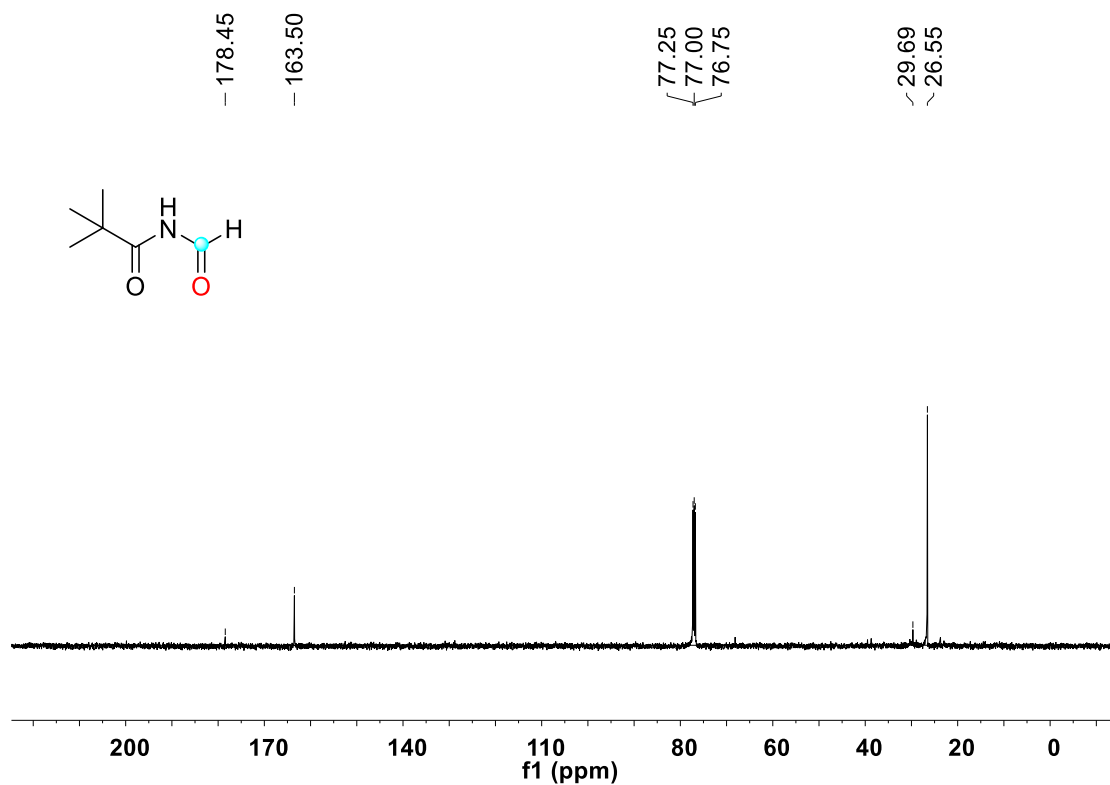
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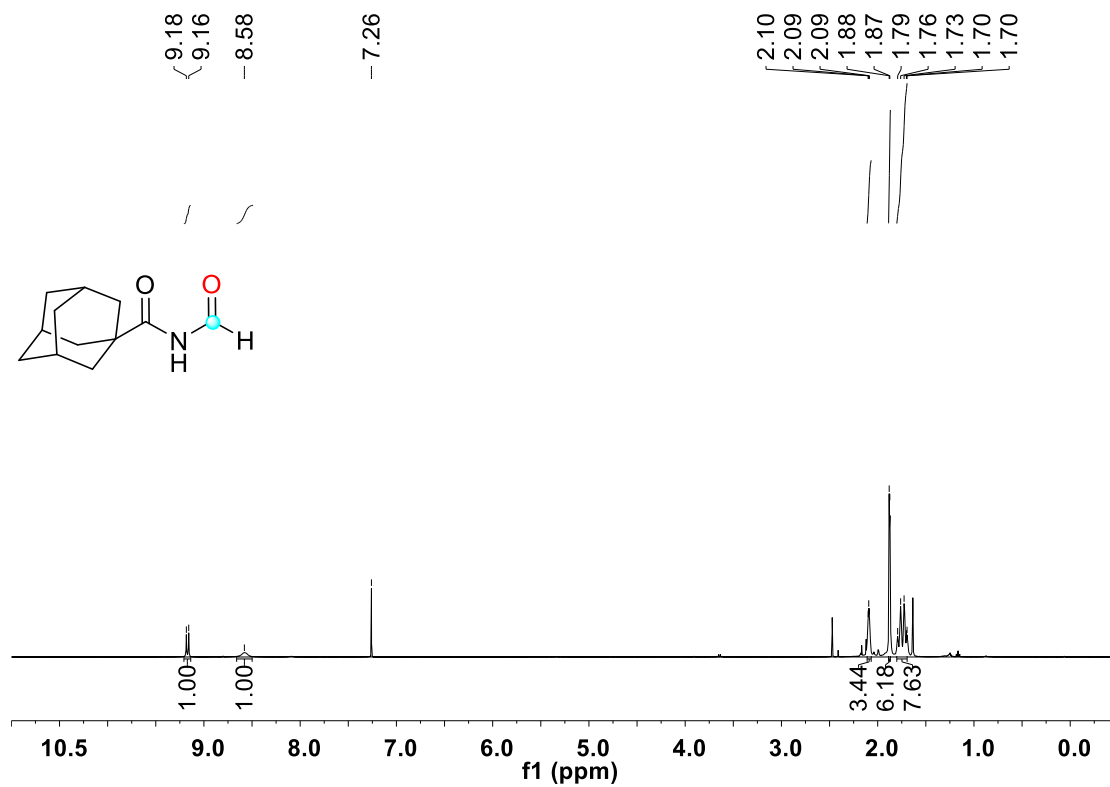
¹H NMR (400 MHz, CDCl₃) spectra for compound **2af**



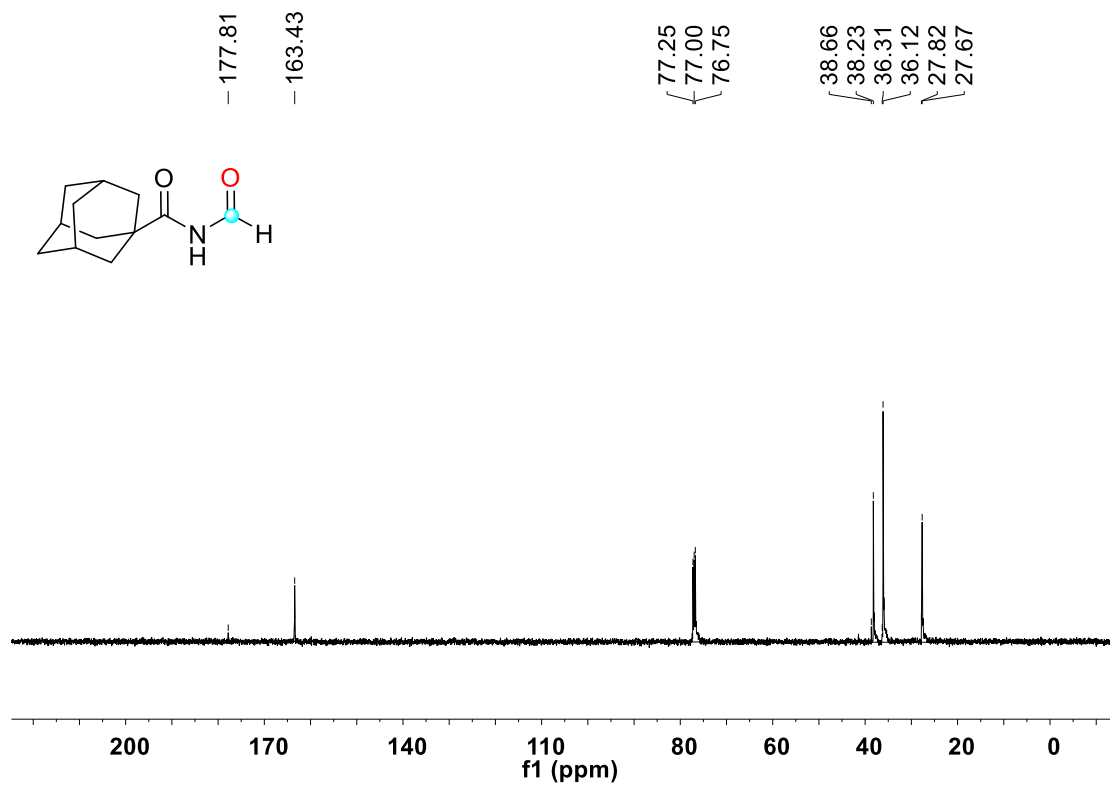
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2af**



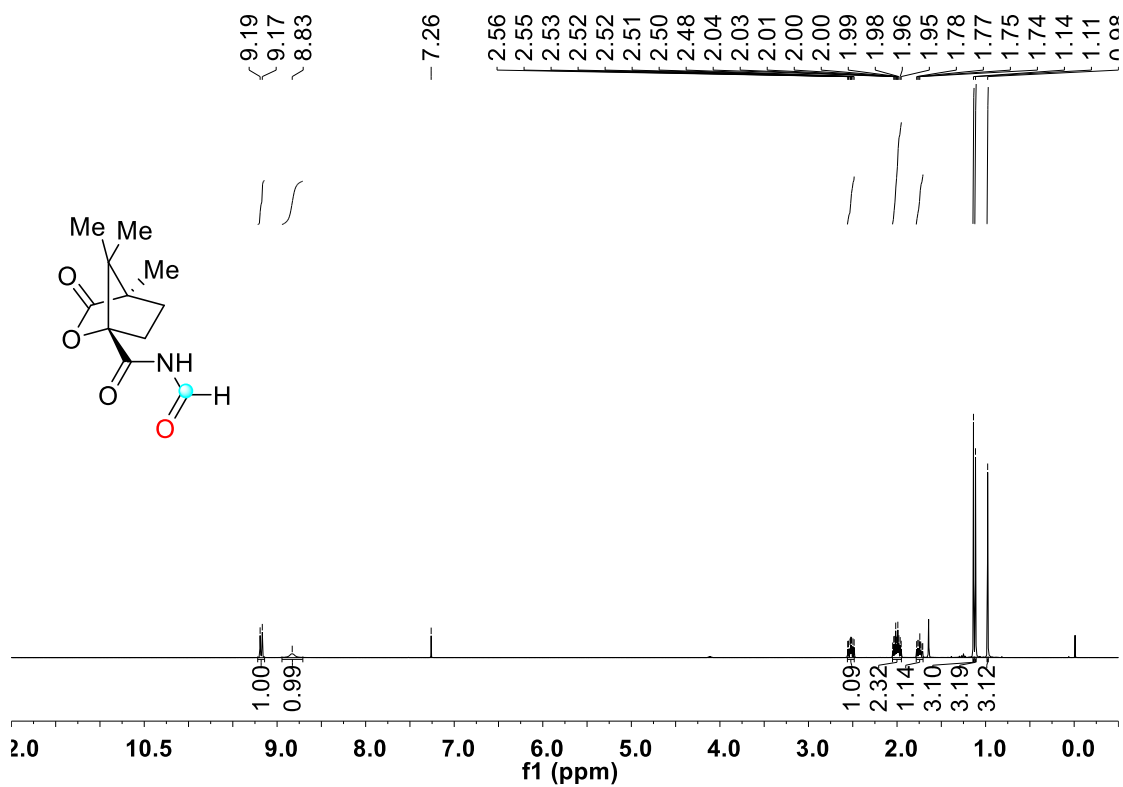
¹H NMR (400 MHz, CDCl₃) spectra for compound **2ag**



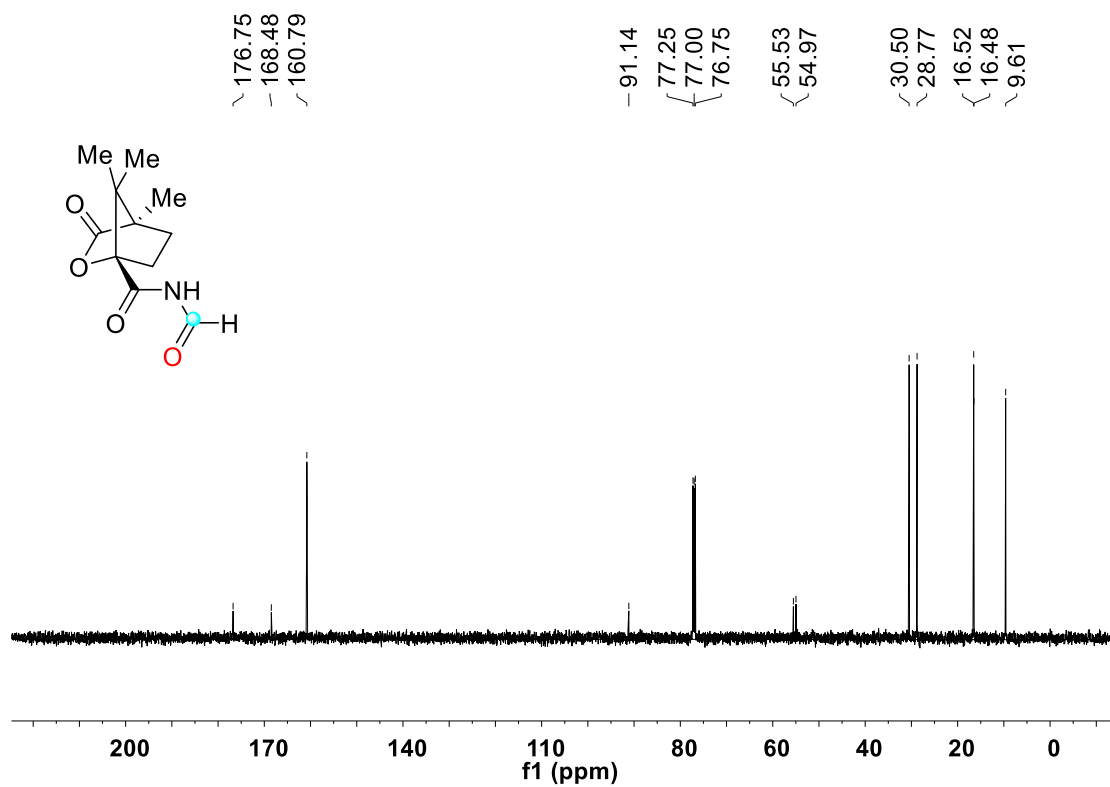
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2ag**



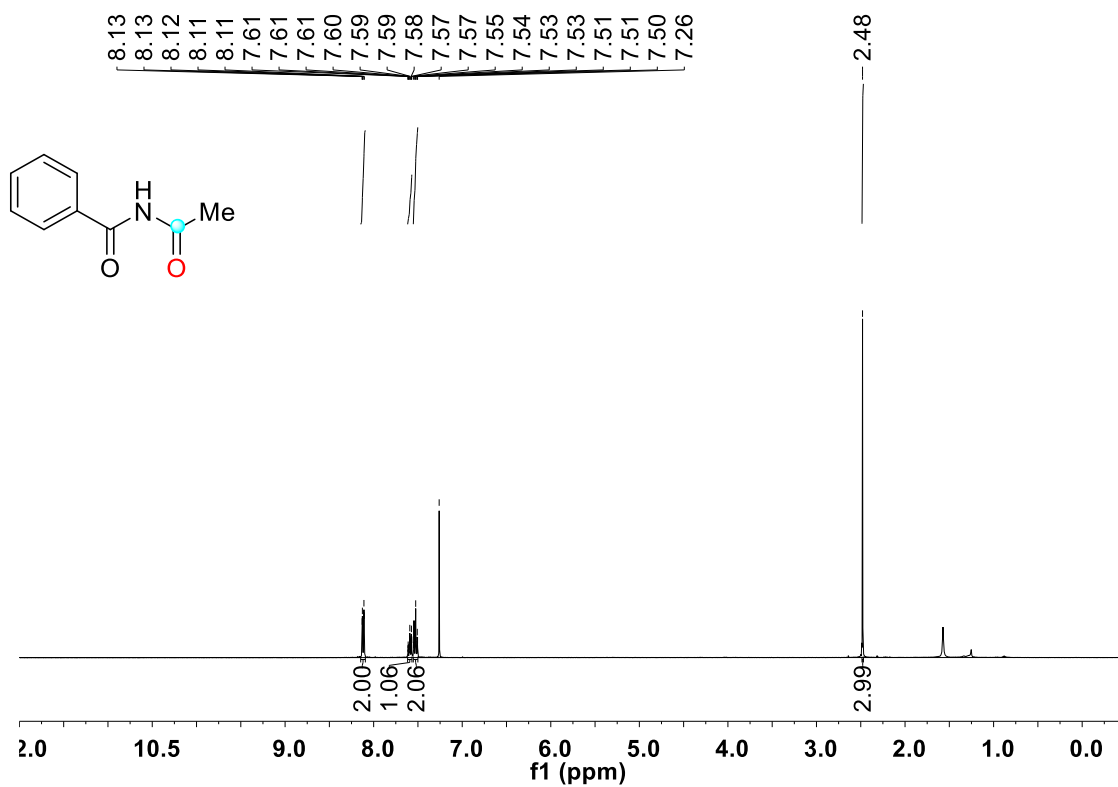
¹H NMR (400 MHz, CDCl₃) spectra for compound **2ah**



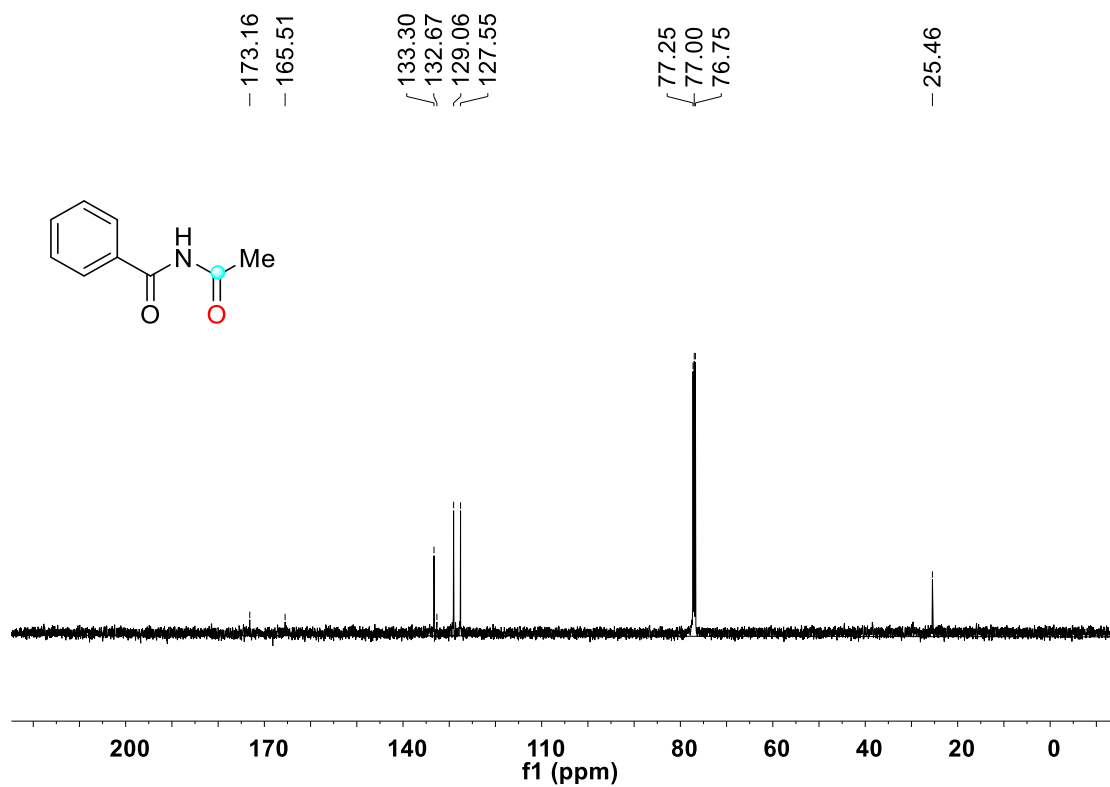
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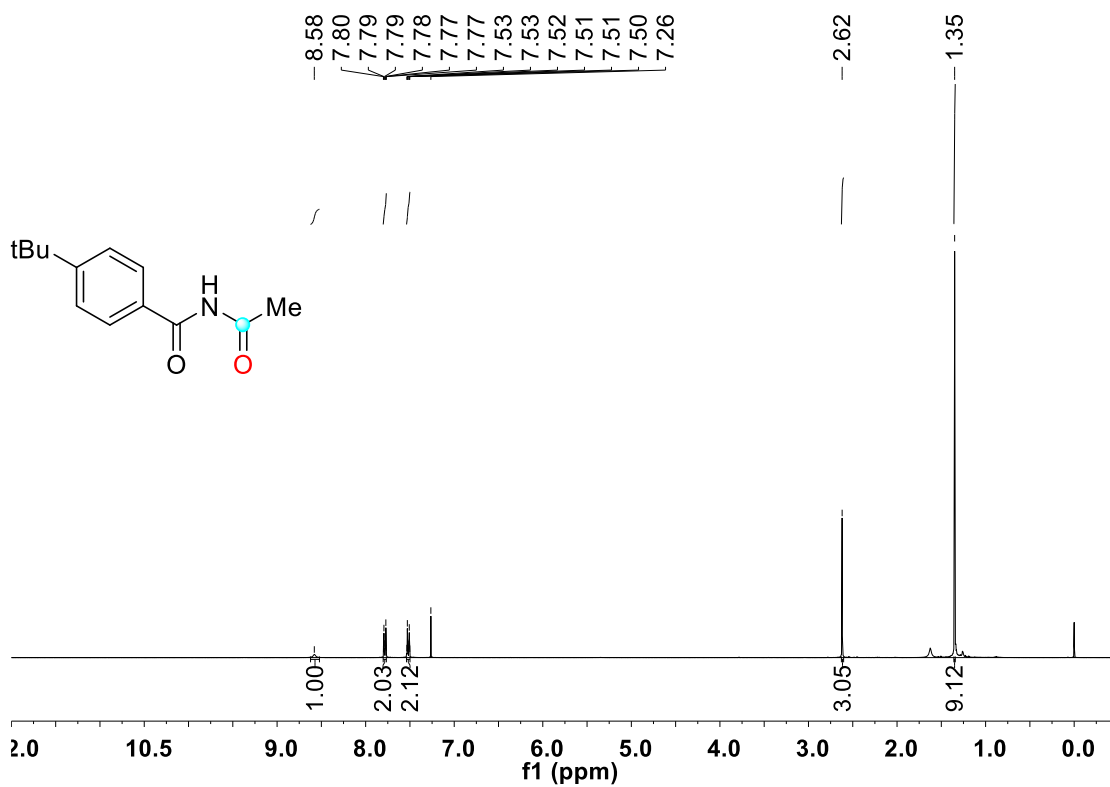
¹H NMR (400 MHz, CDCl₃) spectra for compound **2ai**



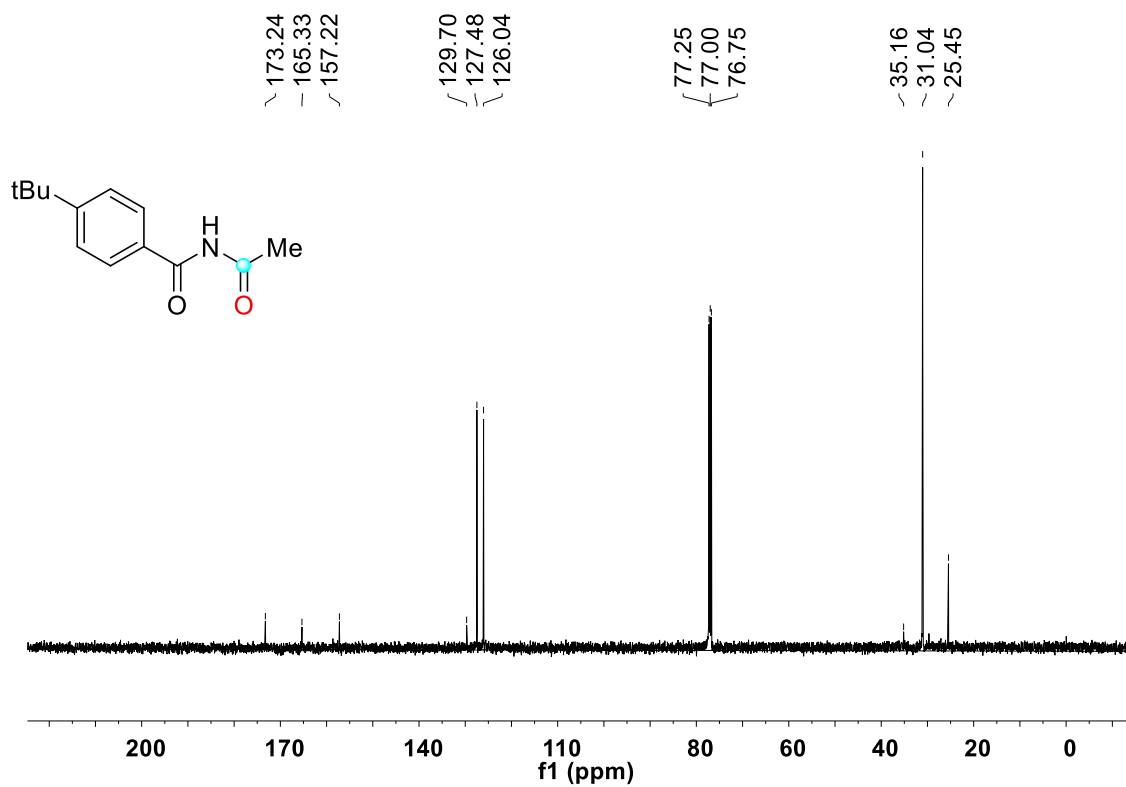
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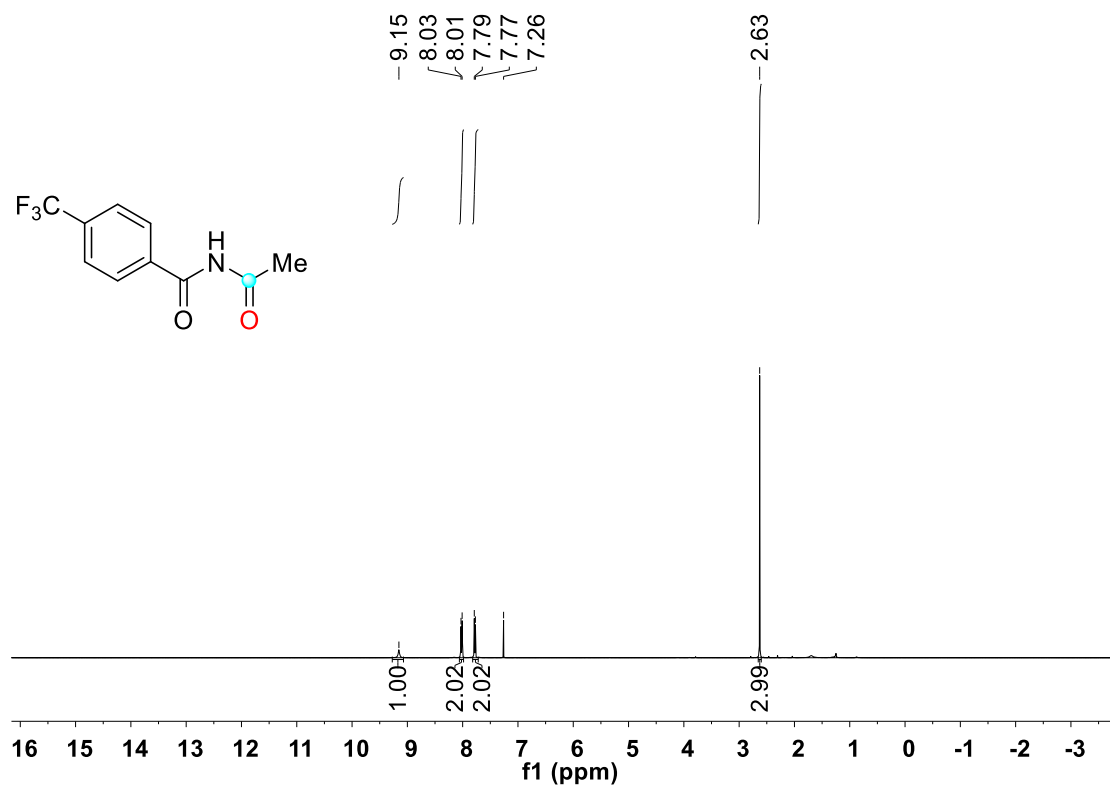
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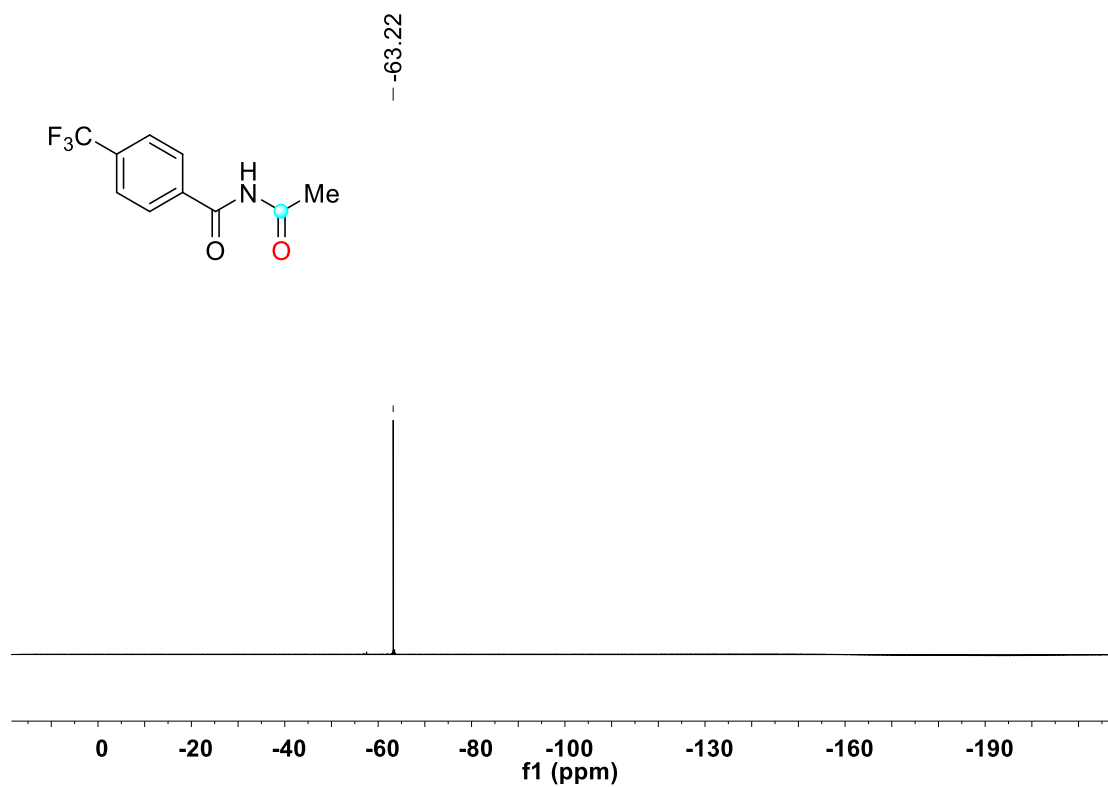
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2aj**



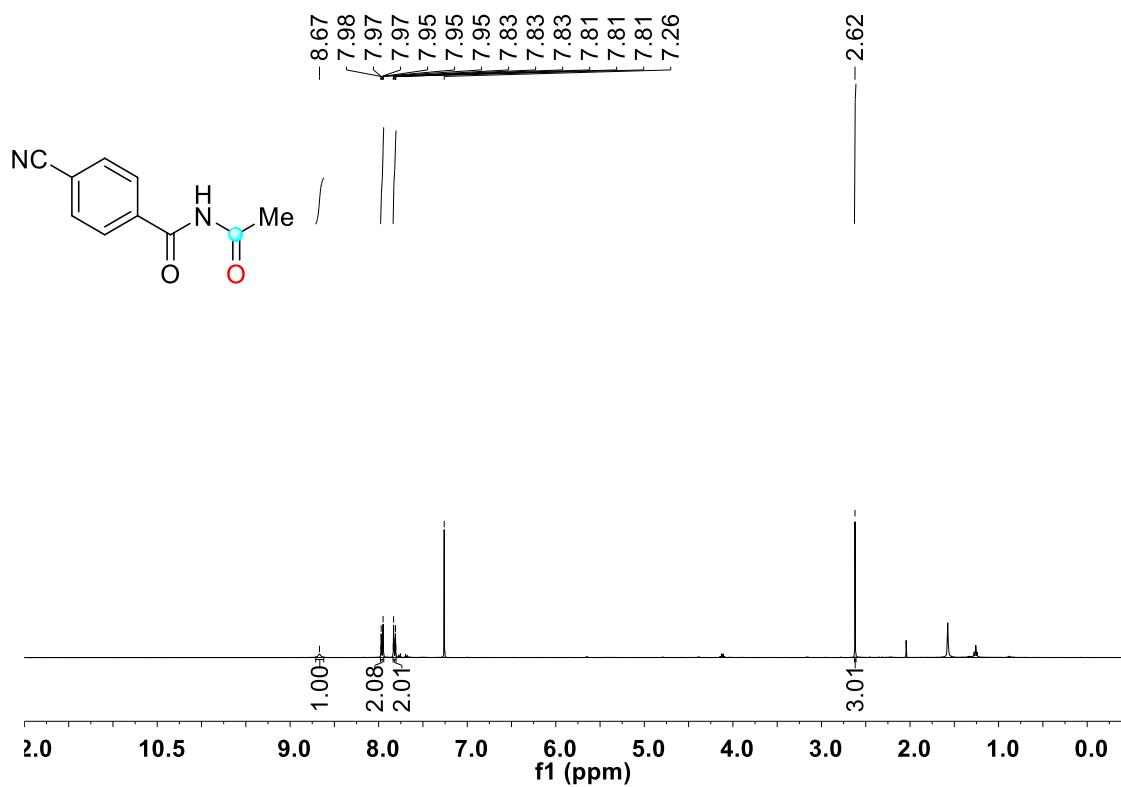
¹H NMR (400 MHz, CDCl₃) spectra for compound **2ak**



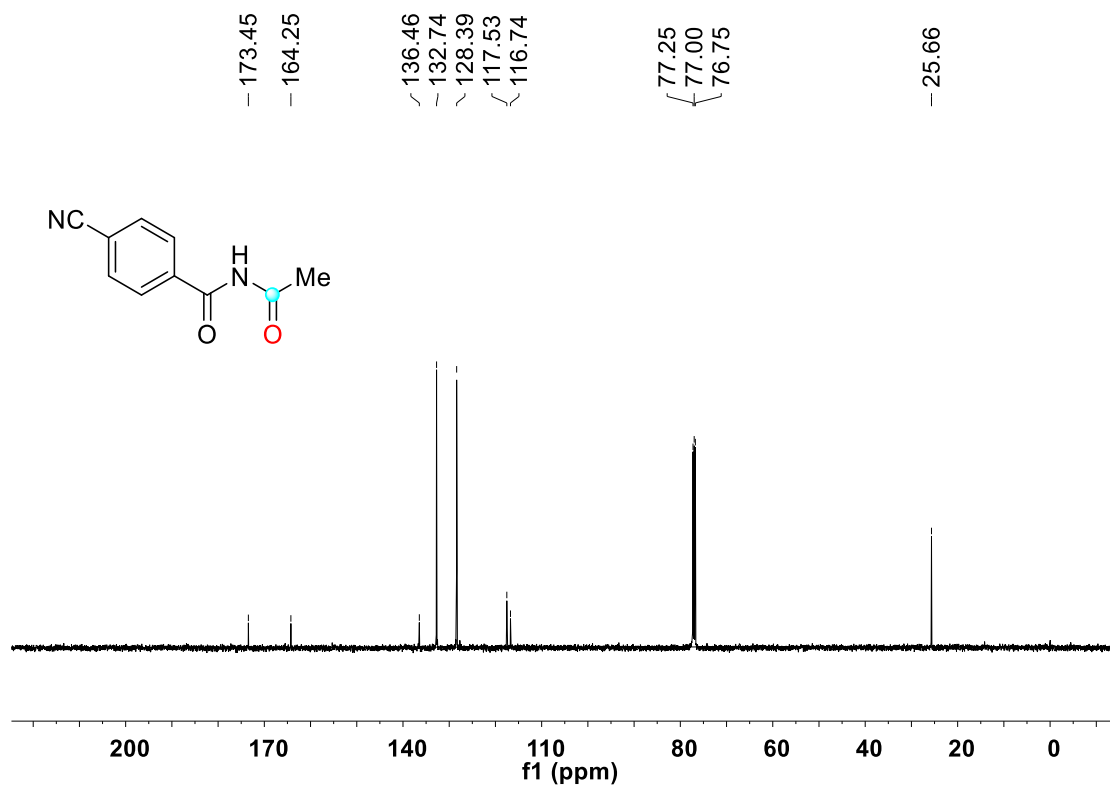
^{19}F NMR (377 MHz, CDCl_3) spectra for compound **2ak**



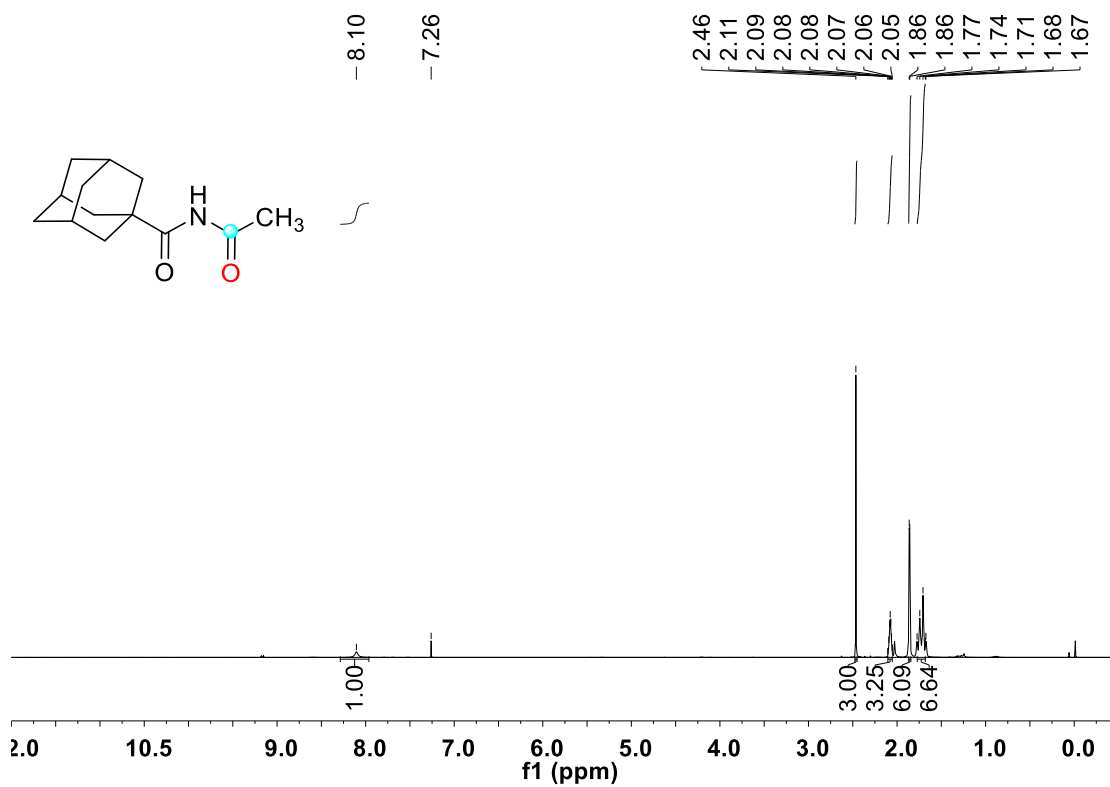
^1H NMR (400 MHz, CDCl_3) spectra for compound **2al**



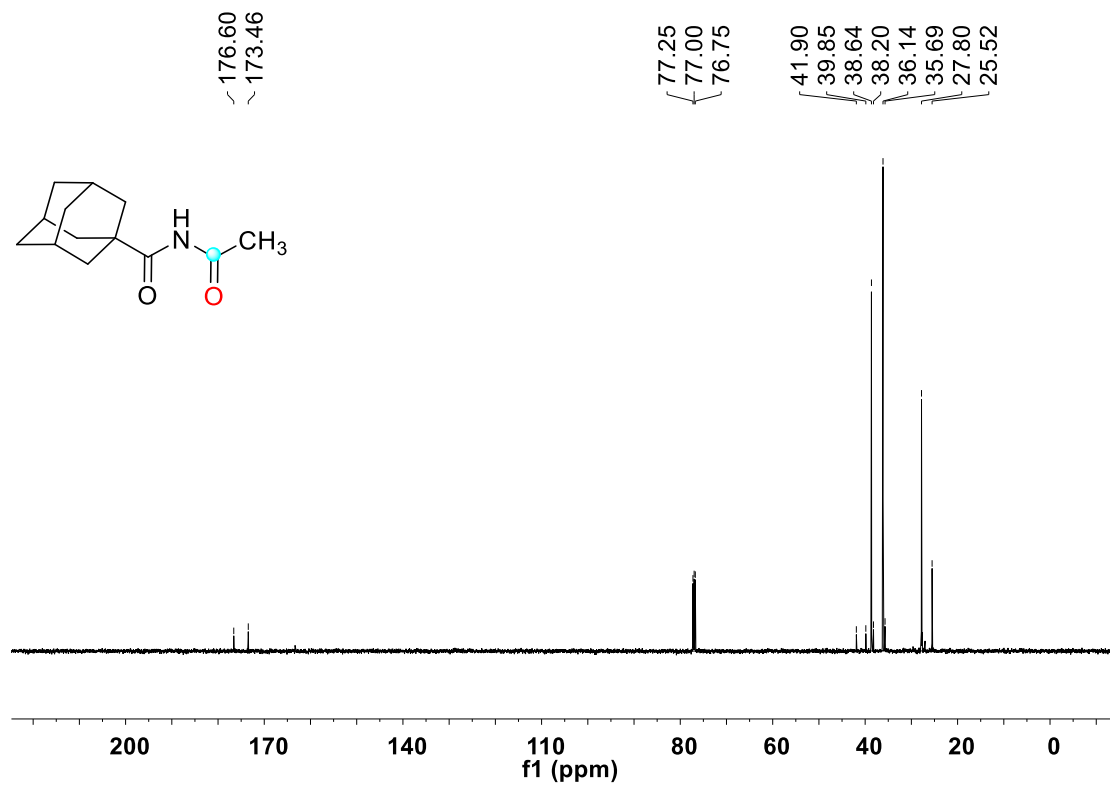
¹³C NMR (126 MHz, CDCl₃) spectra for compound **2al**



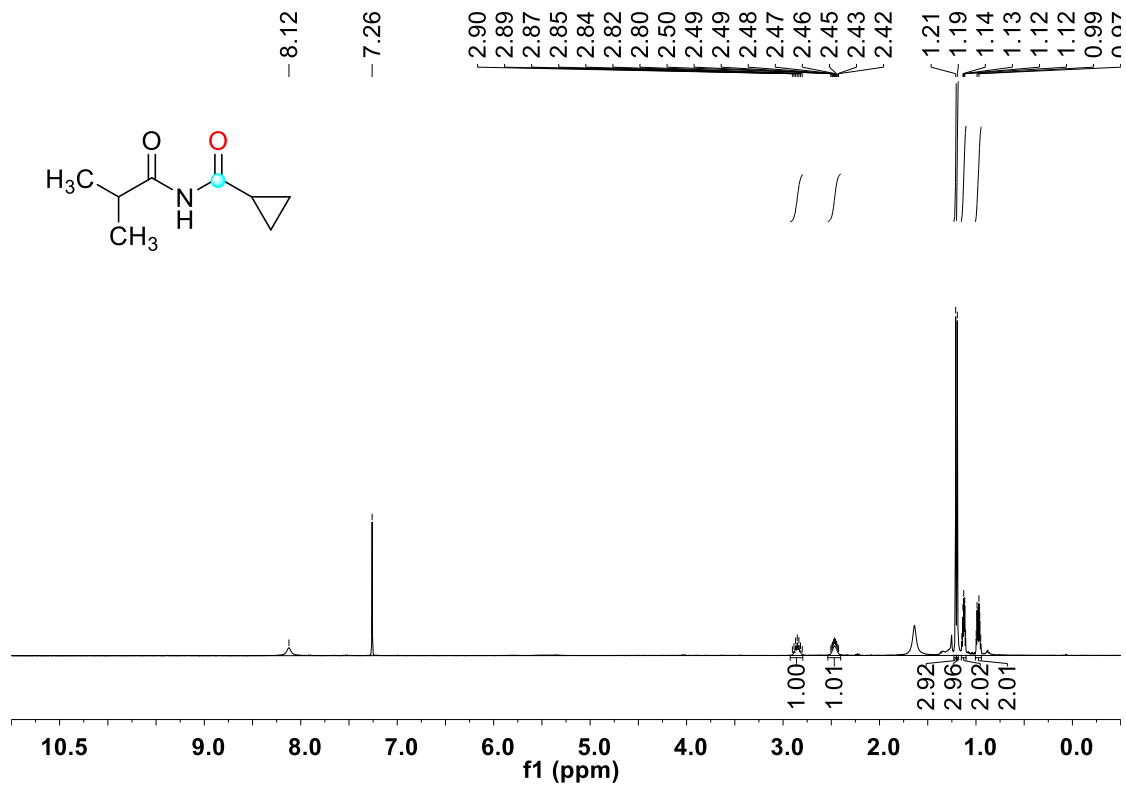
¹H NMR (400 MHz, CDCl₃) spectra for compound **2am**



¹³C NMR (126 MHz, CDCl₃) spectra for compound **2am**



¹H NMR (400 MHz, CDCl₃) spectra for compound **2an**



^{13}C NMR (126 MHz, CDCl_3) spectra for compound **2an**

