**Electronic Supporting Information (ESI)**

# Simple and Scalable Electrosynthesis of 1*H*-1-Hydroxyquinazolin-4-ones

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## 1. General Information

If not stated otherwise, all reactions were performed under ambient conditions and chemicals in analytical grade were used as purchased without further purification. Cyclohexane and ethyl acetate were purchased in technical grade and purified by distillation at reduced pressure prior to use. Milli-Q™ water was obtained using Simplicity™ System (UV) (*Merck KGaA*, Darmstadt, Germany) for chromatography purposes. Anhydrous solvents were obtained from a solvent purification system SPS-5 (*M. Braun Incorporated*, Stratham, USA).

### Chromatography

Thin layer chromatography was performed using silica gel DC Kieselgel 60 F254 on aluminium plates (*Merck KGaA,* Darmstadt, Germany). A UV lamp (*λ* = 254 nm, NU-4 KL, *Benda*, Wiesloch, Germany). Preparative column chromatography was performed on silica gel 60 M (0.040–0.063 mm*, Macherey-Nagel GmbH & Co*, Düren, Germany) using a Sepacore™ system with a Büchi Control Unit C-620, Büchi Pump Modules C-605, a UV detector Büchi UV photometer C-635, and Büchi Fraction Collector C-660 (*Büchi- Labortechnik GmbH*, Essen, Germany). The isolation of the different quinoline derivatives was conducted with reversed phase column chromatography on Sepacore™ C18 (*Büchi-Labortechnik GmbH*, Essen, Germany) using different mixtures of water (0.1% formic acid) and acetonitrile as eluents.

### High Resolution Mass Spectrometry

Mass spectra *via* electrospray-ionization (ESI+/ESI–) mass spectrometry were recorded using an Agilent 6545 QTOF-MS (*Agilent*, Santa Clara (CA), USA). Mass-charge ratios (*m*/*z*) were obtained for the characterized compounds.

### X-ray Crystallography

The measurements of the crystal structures were carried out on a STOE IPDS-2T (*STOE & Cie GmbH*, Darmstadt, Germany) using a Mo source with graphite tube monochromator.

### Gas Chromatography (GC)

Gas chromatography was performed using a GC-2010 (*Shimadzu*, Kyoto, Japan) equipped with a flame ionization detector (FID) and a quartz capillary column HI-5MS (*Avantor VWR*, Radnor, USA) with following specification: length of 30 m, inner diameter of 0.25 mm and a stationary phase ((5%-phenyl)-dimethylsiloxane) of 0.25 µm thickness. Hydrogen was used as carrier gas with a flow rate of 45.5 cm s-1. Measurements were performed at an injector temperature of 250 °C and a detector temperature of 310 °C, starting at 50 °C and heating to 290 °C (holding for 2 min) with a temperature ramp of 15 °C/min (method: hart-18min).

### High Performance Liquid Chromatography (HPLC)

Analysis of crude reaction mixtures and purified products was performed using a modular system LC-20A *Prominence* (*Shimadzu Deutschland GmbH*, Duisburg, Germany), UV/VIS-detector SPD-20A/AV (*Shimadzu Deutschland GmbH*, Duisburg, Germany), and LCMS-2020 Single Quadrupole (*Shimadzu Deutschland GmbH*, Duisburg, Germany). Analytical separation was performed using an Eurospher II 100-5 C-18-Trennsäule (*Knauer Wissenschaftliche Geräte GmbH*, Berlin, Germany) column (length of 150 mm, diameter of 4 mm, pore size of 100 Å, particle size 5 µm). As eluents, acetonitrile and water with 5% (*v*/*v*) acetonitrile and formic acid (1 mL $L^{-1}$) were used. Given retention times were obtained at λ = 254 nm.

**Microwave Synthesizer**

Microwave reactions were performed using a Discover microwave reactor (*CEM Corporation*, Matthews (NC), USA) and 10 ml Pyrex Pressure Vessels (*CEM Corporation*, Matthews (NC), USA).

**Nuclear Magnetic Resonance (NMR) Spectroscopy**

Nuclear magnetic resonance experiments were performed using a nuclear magnetic resonance spectrometer Avance III HD300 (*Bruker*, Karlsruhe, Germany) $^{1}$H NMR (300 MHz), $^{19}$F NMR (282 MHz), Avance II 400 (*Bruker*, Karlsruhe, Germany) $^{1}$H NMR (400 MHz), $^{13}$C NMR (101 MHz), and $^{19}$F NMR (376 MHz), and Avance III 600 (*Bruker*, Karlsruhe, Germany) $^{1}$H NMR (600 MHz), $^{13}$C NMR (151 MHz). The spectra were recorded using deuterated solvents. To normalize the spectra obtained, reference was made to the existing solvent signal of non-deuterated fractions according to the data provided by *Fulmer et al.*[1]: $CDCl_3$ ($^{1}$H NMR: $\delta$ = 7.26 ppm, $^{13}$C NMR: $\delta$ = 77.2 ppm), dichloromethane-$d_2$ ($^{1}$H NMR: $\delta$ = 5.32 ppm, $^{13}$C NMR: $\delta$ =53.8 ppm), methanol-$d_4$ ($^{1}$H NMR $\delta$ = 3.31 ppm, $^{13}$C NMR: $\delta$ = 49.0 ppm) and water-$d_2$ ($^{1}$H NMR $\delta$ = 4.79 ppm) acetonitrile-$d_3$ ($^{1}$H NMR $\delta$ = 1.94 ppm, $^{13}$C NMR: $\delta$ = 118.3 ppm) and. Besides $^{1}$H, $^{13}$C and $^{19}$F NMR experiments, the 2D techniques $^{1}$H,$^{1}$H-COSY, $^{1}$H,$^{13}$C-HSQC, $^{1}$H,$^{13}$C-HMBC and $^{1}$H,$^{1}$H-NOESY were used assisting to assign the signals. The following abbreviations were used to describe the signals: s (singlet), d (doublet), t (triplet), dd (doublet of doublets), td (triplet of doublets), m (multiplet), q (quartet), hep (heptet). The spectra obtained were evaluated with MestReNova 14.2.0-26256 (*Mestrelab Research S.L.*, Spain). $^{19}$F NMR spectra were obtained with *α,α,α*-trifluorotoluene as external standard ($\delta$ = 63.9 ppm) and recorded without $^{1}$H decoupling.

**Cyclic Voltammetry (CV) Measurements**

Cyclic voltammetry was performed using a Metrohm 663 VA Stand equipped with a Autolab type III potentiostat (*Metrohm AG*, Herisau, Switzerland). WE: boron-doped diamond tip, 2 mm in diameter; CE: glassy carbon rod; RE: Ag/AgCl (*vs*. $FcH/FcH^+$ = ferrocene). Scan rate=100 $mV \cdot s^{-1}$. Electrolyte: 0.5 M sulphuric acid in a 1:1 (*v*/*v*) water–methanol mixture; *c*(substrate)=0.05 M.

**Electrochemical Set-Up**

Electrochemical reactions were carried out using a multichannel galvanostat HMP4040 (*Rohde & Schwarz*, München, Germany) with a controllable DC output of 0-32 V and 0–10 A and a maximum power of 160 W per channel was used as power source. The different cells used for screening or batch reactions are described below.

**Screening Reactions**

Teflon™ cells with a volume of 5 mL were used for the undivided set-up (figure S1, left). Divided Teflon™ screening cells were equipped with a glass frit or Nafion™ as separator material as shown (figure S1, right). Glass frits and Nafion™ N324 membrane (DuPont, Wilmington, United States) used as separator materials were pre-treated in the corresponding electrolyte prior to use. Stirring bars were used during electrolysis in each cell. The described screening systems are commercially available as IKA Screening System Package (*IKA™ Werke GmbH & Co. KG*, Staufen, Germany).

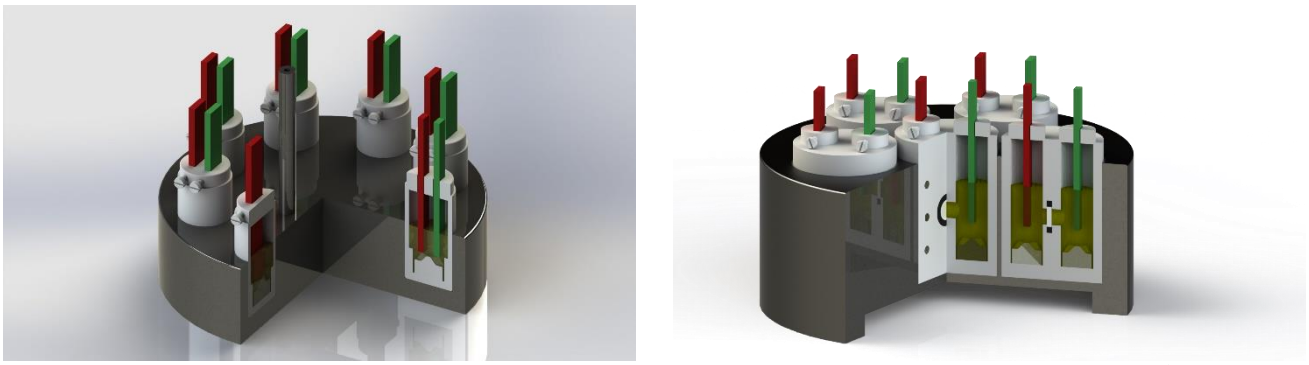


Figure S1: Undivided screening set-up (left) and divided screening set-up (right).[2]

Table S1: Electrode materials, purity and their supplier.

| Entry | Electrode Material | Purity | Supplier |
|---|---|---|---|
| 1 | Boron-doped diamond (DIACHEM®) | 15 µm boron-doped diamond layer on silicon | *CONDIAS GmbH*, Itzehoe, Germany |
| 2 | Glassy carbon | - | *HTW*, Thierhaupten, Germany |
| 3 | Isostatic graphite | - | *SGL Carbon*, Bonn, Germany |
| 4 | Lead | - | *Globus Fachmärkte GmbH & Co. KG*, Völklingen, Germany (home depot) |
| 5 | Platinum | >99% | *OEGUSSA*, Vienna, Austria |
| 6 | Leaded bronze (CuSn7Pb15) | - | *Metallwerk Langenau GMBH*, Langenau, Germany |

Scale-up experiments were performed in 50 mL, 100 mL, and 250 mL batch-type cells with a PTFE stopper and sleeve, electrodes and electrode holders used a TDK-Lambda Z+ series (*TDK-Lambda UK Limited*, Devon, United Kingdom) or a multichannel power supply HMP4040 (*Rohde & Schwarz*, München, Germany) as power sources. The batch-type cells of the scale-up experiments are commercially available as SynLectro™ Starter Kit (*Merck KGaA*, Darmstadt, Germany).[3] In the 25 mL electrolysis set-up glassy carbon BDD electrodes with dimensions of 6 cm x 2 cm were used. In the 250 mL electrolysis set-up glassy carbon and BDD electrodes with identical dimensions of 12 cm x 4 cm were used.

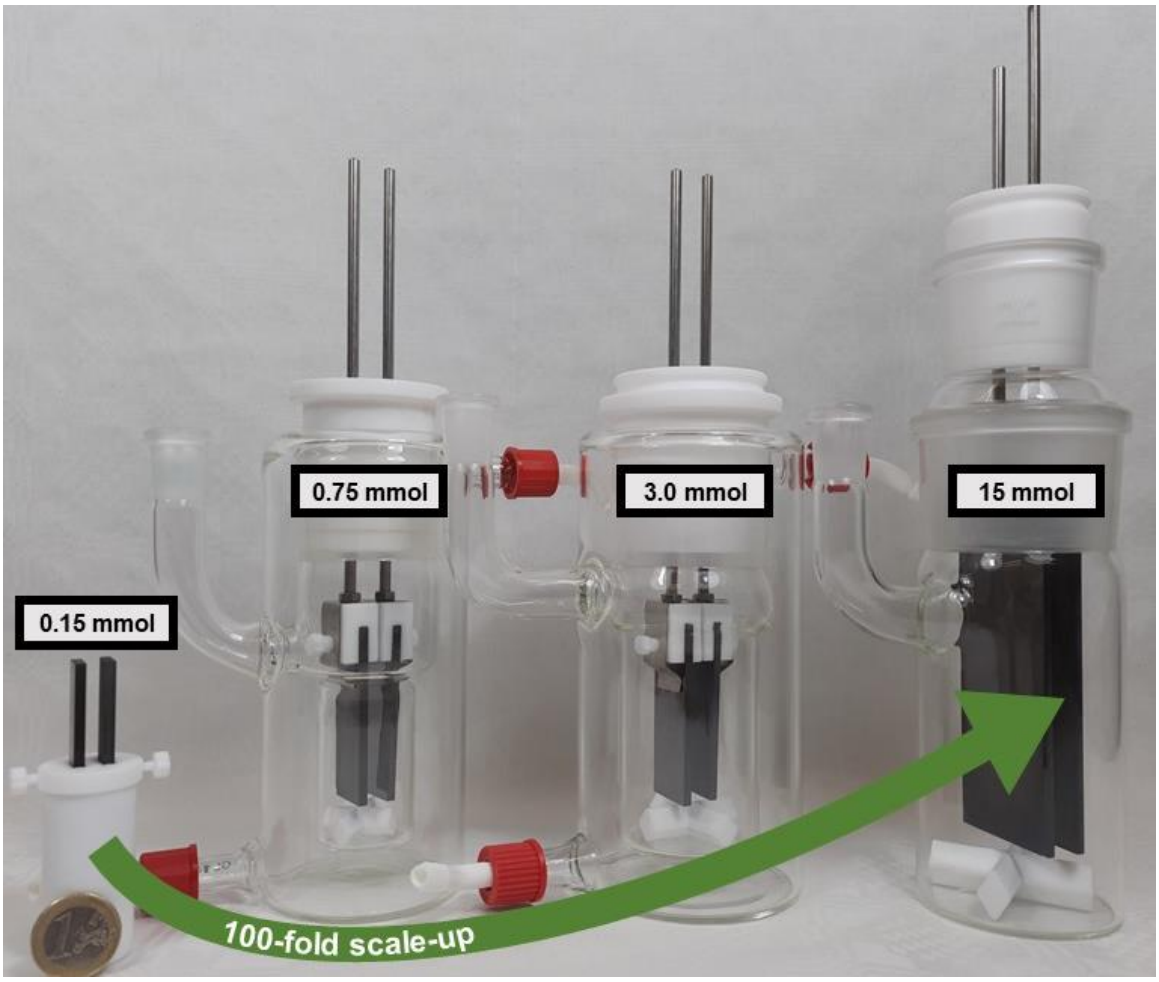
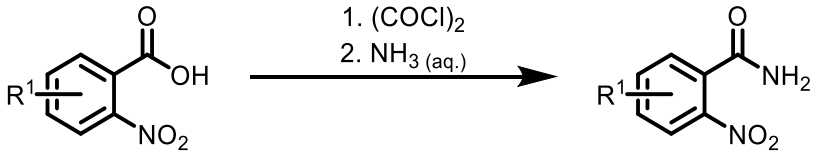



Figure S2: Different batch-type cells left to right: 5 mL Teflon™ screening cell with glassy carbon and BDD electrodes; 50 mL batch-type jacketed glass cell with glassy carbon and BDD electrodes, Teflon™ plug and electrode holders; 250 mL batch-type glass cell with glassy carbon and BDD electrodes, Teflon™ plug and electrode holders.

## 2. General Protocols

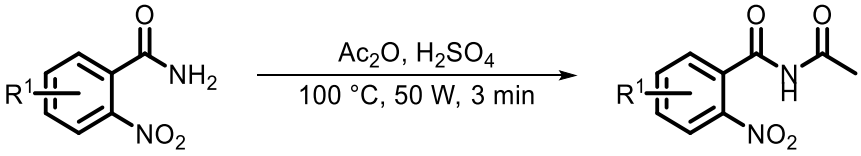
### 2.1. General Protocol for the Synthesis of Substituted 2-Nitrobenzamides (GPI)




Scheme S1: Synthesis of substituted 2-nitrobenzamides.

DMF (4 drops) was added to the 0.6 M solution of substituted 2-nitrobenzoic acid (1.0 eq.) in anhydrous THF or DCM. The reaction mixture was cooled to 0 °C and oxalyl chloride (1.1 – 2.0 eq.) was slowly added. The reaction solution was stirred at room temperature for 3 hours. After completion of the reaction the reaction mixture was added to aqueous ammonia (25% *w*/*v*, 10.0 eq) at 0 °C and stirred for 30 min at ambient temperature. The solvent was removed under reduced pressure. The precipitate formed was filtered off and washed with water. Residual water was removed by azeotropic distillation using toluene.

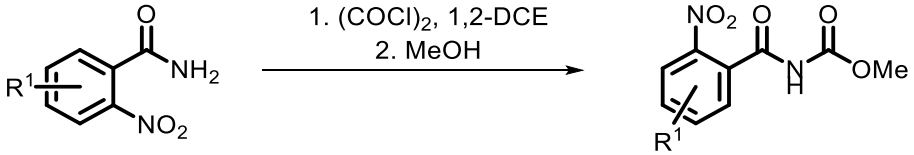
### 2.2. General Protocol for the Synthesis of Substituted *N*-Acyl-2-nitrobenzamides (GPII)




Scheme S2: Synthesis of substituted *N*-acyl-2-nitrobenzamides.

The reaction was performed in a microwave vial. Anhydride (3.0 – 10.0 eq.) was added to 2-nitrobenzamide (1.0 eq.) followed by the addition of 2 – 3 drops of 96% $H_2SO_4$. The reaction mixture was irradiated in a microwave for 3 min at 100 °C and 50 W with vigorous stirring. Then the reaction solution was diluted with 10 mL water and extracted three times with 10 mL ethyl acetate. The combined organic fractions were washed twice with 15 mL sat. aq. $NaHCO_3$, once with 15 mL brine and dried over anhydrous $Na_2SO_4$. The solvent was removed and the crude material was purified by column chromatography.

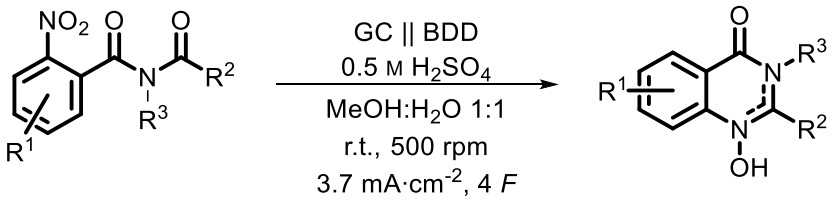
### 2.3. General Protocol for the Synthesis of Methyl (2-nitrobenzoyl)carbamates (GPIII)




Scheme S3: Electrochemical synthesis of substituted methyl (2-nitrobenzoyl)carbamates.

Oxalyl chloride (1.7 eq.) was added to a 0.4 M solution of 2-nitrobenzamide (1.0 eq.) in 1,2-dichloroethane. The solution was refluxed under argon atmosphere for 3 h. The reaction mixture was cooled to -10 °C, 4 mL of methanol:acetonitrile 1:1 (*v*:*v*) was added and stirred for 2 h. The solvent was removed and the product was precipitated by adding 10 mL of water. The solids were filtered off, washed once with water and dried.

## 2.4. General Protocol for the Electrochemical Synthesis of Substituted 1*H*-1-Hydroxyquinazolin-4-ones (GPIV)




Scheme S4: Electrochemical synthesis of substituted 1*H*-1-hydroxy-quinazolin-4-ones.

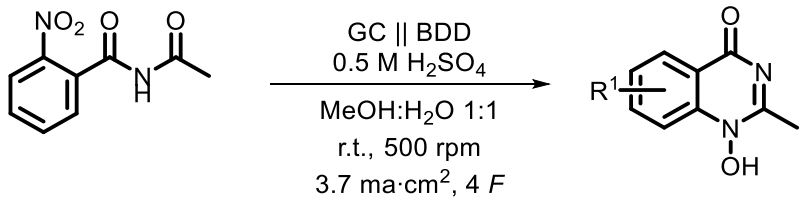
**5 mL Teflon™ screening cell:** 0.15 mmol of the corresponding substrate was dissolved in 2.5 mL methanol and diluted with 2.5 mL 1 M $H_2SO_4$ in a 5 mL Teflon™ screening cell equipped with a stirring bar. The glassy carbon anode and the BDD cathode were immersed into the solution (1.5 cm) resulting in a surface area of 1.5 $cm^2$. The electrolysis was performed under constant current conditions (current density $j$ = 3.7 $mA \cdot cm^{-2}$ for 57.9 C (4 *F*)) with a stirring speed of 500 rpm. After completion of the reaction the pH value of the reaction mixture was adjusted to pH ≈ 8 with an aqueous solution of 1 M $NaHCO_3$. $Na_2SO_4$ was precipitated by adding 20 mL of methanol. The solids were removed by vacuum filtration and washed twice with 20 mL of methanol. The pH of the filtrate was adjusted to pH ≈ 4 – 5 with glacial acetic acid and the solvent was removed. The crude product was purified by reversed phase column chromatography using water with 0.1% formic acid (*v*/*v*) and acetonitrile.

**25 mL glass cell:** 0.75 mmol of the corresponding substrate was dissolved in 12.5 mL methanol and diluted with 12.5 mL 1 M $H_2SO_4$ in a 25 mL glass cell equipped with a stirring bar. The glassy carbon anode and the BDD cathode were immersed into the solution (3 cm) resulting in a surface area of 6 $cm^2$. The electrolysis was performed under constant current conditions (current density $j$ = 3.7 $mA \cdot cm^{-2}$ for 289.5 C (4 *F*)) with a stirring velocity of 500 rpm. After completion of the reaction the pH value of the reaction mixture was adjusted to pH ≈ 8 with an aqueous solution of 1 M $NaHCO_3$. $Na_2SO_4$ was precipitated by adding 70 mL of methanol. The solids were removed by vacuum filtration and washed twice with 70 mL of methanol. The pH value of the filtrate was adjusted to pH ≈ 4 – 5 with glacial acetic acid and the solvent was removed. The crude product was purified by reversed phase column chromatography using water with 0.1% formic acid (*v*/*v*) and acetonitrile.

## 2.5. General Protocol for the Scale-Up of Electrochemical Synthesis of 6a (GPV)

The corresponding substrate was dissolved in methanol and 1 M $H_2SO_{4(aq.)}$ 1:1 (*v*:*v*) in a 100 mL or 250 mL glass cell equipped with a stirring bar. The glassy carbon anode and the BDD cathode were immersed into the solution (100 mL cell:3 cm and 25 mL cell: 7.4 cm) resulting in a surface area of 6 $cm^2$ (100 mL cell) or 29.7 $cm^2$ (250 mL cell). The electrolysis was performed under constant current conditions (current density $j$ = 3.7 $mA \cdot cm^{-2}$, 4 *F*) with a stirring speed of 500 rpm. After completion of the reaction the pH of the reaction mixture was adjusted to pH ≈ 8 with an aqueous solution of 1 M $NaHCO_3$. $Na_2SO_4$ was precipitated by adding 120 mL of methanol. The solids were removed by vacuum filtration and washed twice with 120 mL of methanol. The pH value of the filtrate was adjusted to pH ≈ 4 – 5 with glacial acetic acid and the solvent was removed. The crude product was purified by reversed phase column chromatography using water with 0.1% formic acid (*v*/*v*) and acetonitrile.

## 3. Optimisation of the Electrolytic Reaction Conditions




Scheme S5: Initial conditions of the electrochemical conversion of compound **5a** into the 1*H*-1-hydroxy-2-methylquinazolin-4-one **6a**.

The optimization experiments of the electrolytic conditions in the reductive cyclisation to the 1*H*-1-hydroxy-2-methylquinazolin-4-one **6a** were conducted using substrate **5a** as standard substrate. The electroreduction were performed according to general procedure **GPIV** determining the yield with $^1$H NMR spectroscopy using 2,2-dimethylmalonic acid as internal standard. Deviations from the standard electrolytic conditions are shown in table S1.

Table S1: Screening of electrolysis parameters by deviation from the standard conditions for the optimisation of the synthesis of 1*H*-1-hydroxy-2-methylquinazolin-4-one **6a.**

| Entry | *j* / mA·cm$^2$ | Solvent:Water 1:1 (*v*:*v*) | Supporting Electrolyte | Concentration **5a** / mol/L | Cathode | Yield[a] / % |
|---|---|---|---|---|---|---|
| 1 | - | - | - | - | - | 91 |
| 2 | 1.7 | - | - | - | - | 65 |
| 3 | 2.7 | - | - | - | - | 78 |
| 3 | 5.7 | - | - | - | - | 78 |
| 4 | - | EtOH | - | - | - | 84 |
| 5 | - | MeCN | - | - | - | 71 |
| 6 | - | DMF | - | - | - | 89 |
| 7 | - | MeOH[b] | $NH_4HCOO$ | - | - | 47 |
| 8 | - | - | 0.5 M acetate buffer[c] | - | - | 66 |
| 9 | - | - | - | 0.02 | - | 88 |
| 10 | - | - | - | 0.06 | - | 85 |
| 11 | - | - | - | 0.10 | - | 73 |
| 12 | - | - | - | - | Pt | 0% |
| 13 | - | - | - | - | GC | 90 |
| 14 | - | - | - | - | Isostatic graphite | 84 |
| 15 | - | - | - | - | Pb | 27 |
| 16 | - | - | - | - | CuSn7Pb15 | 0% |

a Yield of 6a was determined by 1H NMR spectroscopy using 2,2-dimethylmalonic acid as internal standard; b no water was used; c 0.5 M AcOH / AcONa was prepared with 90 mmol acetic acid and 10 mmol sodium acetate in 100 mL of distilled water and 100 mL methanol.

## 4. CV-Studies

Cyclic voltammetry (CV) measurements were carried out by using an Ag/AgCl in saturated EtOH as reference electrode and a glassy carbon rod as counter electrode, respectively. BDD were used as working electrodes. Prior to measurements, the solution in the cell was deaerated with an argon flow for 20–25 min. Argon was kept flowing over the electrolyte during the measurements. The CVs were measured starting from the open circuit potential (OCP) value towards cathodic potentials. Three cycles were recorded with a scan rate of 100 $mV \cdot s^{-1}$. The electrode potentials were converted to the reference redox system ferrocene/ferricenium ion ($FcH^+$/FcH). A blank measurement of the electrolyte is shown in figure S3. The CV measurement of the test substrate **5a** is depicted in figure S4. Hereby, a single broad reduction peak is observed at 0.94 V.

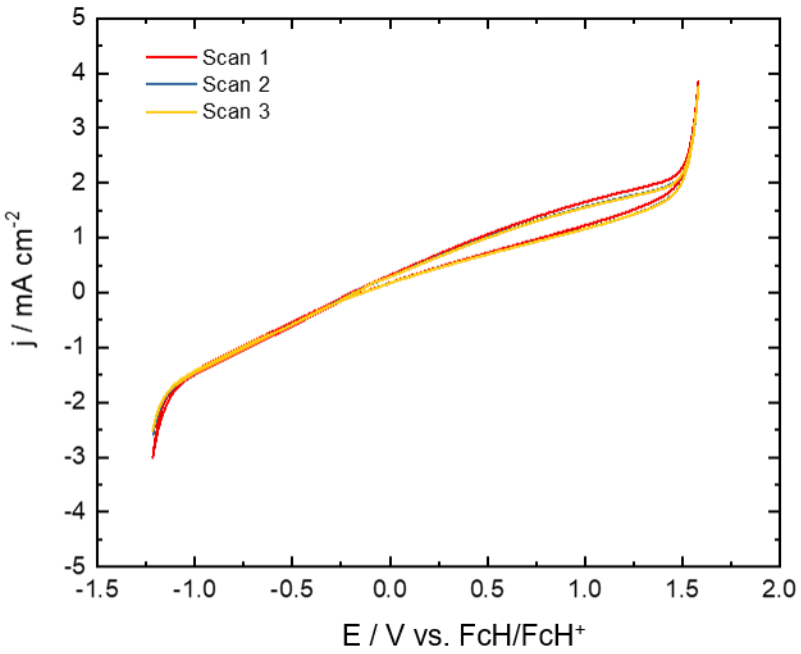



Figure S3: 0.5 M Sulphuric acid in a 1:1 (*v*:*v*) methanol–water mixture; working electrode: BDD; counter electrode: glassy carbon; reference electrode: Ag/AgCl in sat. LiCl/EtOH; Scan rate: 100 mV/s, 3 scans. Potential referenced vs. $FcH/FcH^+$ ($FcH = Fe(\eta^5\text{-}C_5H_5)_2$).

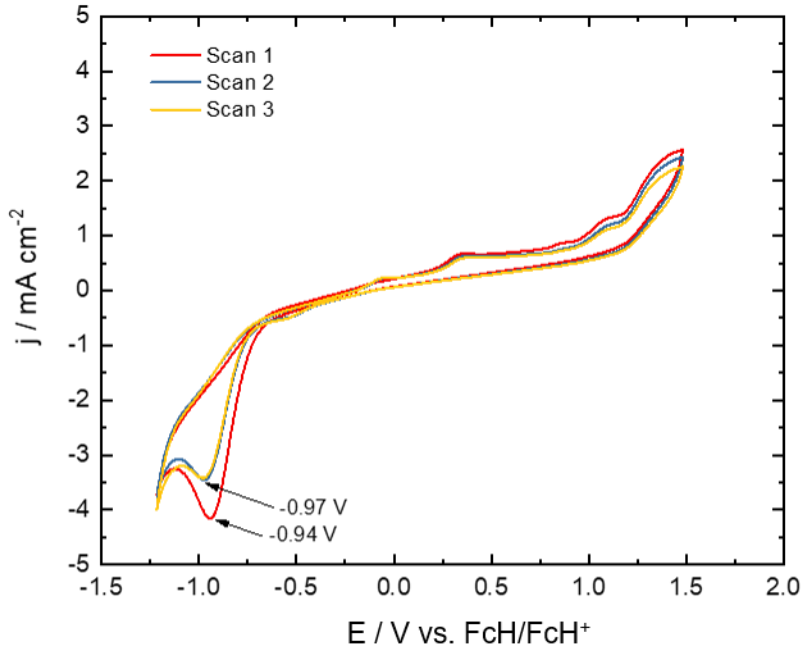
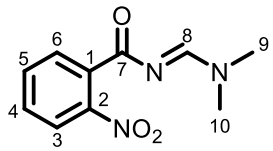



Figure S4: Test substrate **5a** *c*(substrate) = 5.0 mM + 0.5 M Sulphuric acid in a 1:1 (*v*:*v*) methanol–water mixture; working electrode: BDD; counter electrode: glassy carbon; reference electrode: Ag/AgCl in sat. LiCl/EtOH; Scan rate: 100 mV/s, 3 scans. Potential referenced vs. $FcH/FcH^+$($FcH = Fe(\eta^5\text{-}C_5H_5)_2$).

## 5. Preparation of Products and Analytical Data

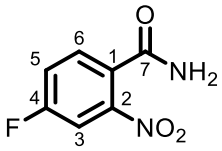
### *N*-(2-(Dimethylamino)vinyl)-2-nitrobenzamide (9)



Under argon atmosphere 2-nitrobenzamide (2.49 g, 15 mmol, 1.0 eq.) and DMF-DMA (5.36 g, 45 mmol, 3.0 eq.) were dissolved in 50 mL anhydrous toluene and refluxed for 24 h. After cooling the reaction mixture to 8 °C 20 mL of cyclohexane was added. The precipitate formed was filtered off, washed twice with cold cyclohexane and twice with cold *n*-pentane. 2.863 g (12.9 mmol, 86%) of the product was isolated as colorless amorphous solid.

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.55 (s, 1H, *H*-8), 7.92–7.86 (m, 1H, *H*-3), 7.69–7.65 (m, 1H, *H*-6), 7.60–6.54 (m, 1H, *H*-4), 7.52–7.46 (m, 1H, *H*-5), 3.15 (s, 3H, *H*-9), 3.07 (s, 1H, *H*-10).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 176.2, 160.6, 149.6, 133.5, 131.9, 130.7, 130.4, 41.7, 35.6.
**HR-MS:** *m/z* for $C_{10}H_{11}N_3O_3+H^+$, $[M+H]^+$ calculated: 222.0873; found: 222.0873.
The spectroscopic data are in accordance with those reported in the literature.[4]

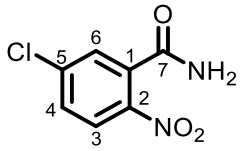
### 4-Fluoro-2-nitrobenzamide (10a)



According to general protocol I **GPI** 4-fluoro-2-nitrobenzoic acid (1.11 g, 6 mmol, 1.0 eq.) and 0.6 mL oxalyl chloride were reacted. The title compound was isolated as colourless amorphous solid (0.790 g, 4.3 mmol, 72%).

**$^1$H NMR (300 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 8.17 (s, 1H, C7-N$H_2$), 7.98 (dd, 1H, *J* = 8.6 Hz, 2.4 Hz, *H*-3), 7.77–7.62 (m, 3H, *H*-5, *H*-6, C7-N$H_2$).
**$^{13}$C NMR (75 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 166.1, 162.9, 160.4, 148.5 (d, *J* = 9.3 Hz), 131.1 (d, *J* = 8.8 Hz), 128.7 (d, *J* = 3.7 Hz), 120.1 (d, *J* = 21.4 Hz), 111.9 (d, J = 27.3 Hz).
**$^{19}$F NMR (282 MHz, DMSO-$d_6$)** $\delta$ [ppm]: -108.10 (td, *J* = 8.4 Hz, 5.6 Hz).
**HR-MS:** *m/z* for $C_7H_5FN_2O_3+H^+$, $[M+H]^+$ calculated: 185.0357; found: 185.0353.
The spectroscopic data are in accordance with those reported in the literature.[5]

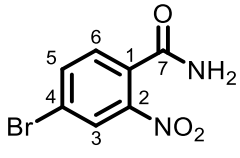
### 5-Chloro-2-nitrobenzamide (10b)



According to general protocol I **GPI** 5-chloro-2-nitrobenzoic acid (1.08 g, 5 mmol, 1.0 eq.) and 0.5 mL oxalyl chloride were reacted. The title compound was isolated as colourless amorphous solid (0.775 g, 3.9 mmol, 78%).

**$^1$H NMR (400 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 8.21 (s, 1H, C7-N$H_2$), 8.05 (d, 1H, *J* = 8.6 Hz, *H*-6), 7.82 (s, 1H, C7-N$H_2$), 7.78–7.70 (m, 2H, *H*-3, *H*-4).
**$^{13}$C NMR (101 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 165.7, 145.7, 137.9, 134.3, 130.4, 128.7, 126.1.
**HR-MS:** *m/z* for $C_7H_5{}^{35}ClN_2O_3+H^+$, $[M+H]^+$ calculated: 201.0061; found: 201.0059.

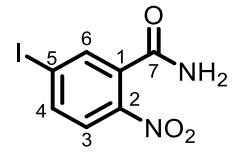
### 4-Bromo-2-nitrobenzamide (10c)



According to general protocol I **GPI** 4-bromo-2-nitrobenzoic acid (1.47 g, 6 mmol, 1.0 eq.) and 0.6 mL oxalyl chloride were reacted. The title compound was isolated as colourless amorphous solid (1.293 g, 5.3 mmol, 88%).

**$^1$H NMR (400 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 8.24 (d, 1H, *J* = 1.9 Hz, *H*-3), 8.20 (s, 1H, C7-N$H_2$), 7.99 (dd, 1H, *J* = 8.2 Hz, 1.9 Hz, *H*-5), 7.77 (s, 1H, C7-N$H_2$), 7.59 (d, *J* = 8.2 Hz, *H*-6).
**$^{13}$C NMR (101 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 166.2, 148.1, 135.9, 131.2, 130.6, 126.7, 122.8.
**HR-MS:** *m/z* for $C_7H_5{}^{79}BrN_2O_3+H^+$, $[M+H]^+$ calculated: 244.9556; found: 244.9552.

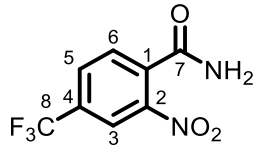
**5-Iodo-2-nitrobenzamide (10d)**



According to general protocol I **GPI** 5-iodo-2-nitrobenzoic acid (1.76 g, 6 mmol, 1.0 eq.) and 0.6 mL oxalyl chloride were reacted. The title compound was isolated as colourless amorphous solid (1.666 g, 5.7 mmol, 95%).

**$^1$H NMR (400 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 8.18 (s, 1H, C7-N*H*$_2$), 8.05 (dd, 1H, *J* = 8.5 Hz, 1.9 Hz, *H*-4), 7.97 (d, 1H, *J* = 1.9 Hz, *H*-6), 7.81–7.72 (m, 2H, C7-N*H*$_2$, *H*-3).
**$^{13}$C NMR (101 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 165.7, 146.7, 139.3, 137.1, 133.9, 125.6, 101.5.
**HR-MS:** *m/z* for $C_7H_5IN_2O_3$+$H^+$, $[M+H]^+$ calculated: 336.9327; found: 336.9326.

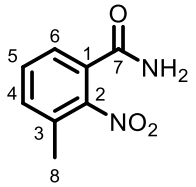
**2-Nitro-4-(trifluoromethyl)benzamide (10e)**



According to general protocol I **GPI** 2-nitro-4-(trifluoromethyl)benzoic acid (0.51 g, 2 mmol, 1.0 eq.) and 0.2 mL oxalyl chloride were reacted. The title compound was isolated as colourless amorphous solid (0.405 g, 1.7 mmol, 77%).

**$^1$H NMR (600 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 8.41 (d, 1H, *J* = 1.8 Hz, *H*-3), 8.31 (s, 1H, C7-N*H*$_2$), 8.19 (dd, 1H, *J* = 8.0 Hz, 1.0 Hz, *H*-5), 7.91 (s, 1H, C7-N*H*$_2$), 7.87 (d, 1H, *J* = 8.0 Hz, *H*-6).
**$^{13}$C NMR (151 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 166.1, 147.4, 136.1, 130.7 (q, *J* = 31.6 Hz), 130.4, 130.2 (q, *J* = 3.6 Hz), 122.8 (q, *J* = 272.8 Hz), 121.5.
**$^{19}$F NMR (282 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: -61.49.
**HR-MS:** *m/z* for $C_8H_5F_3N_2O_3$+$H^+$, $[M+H]^+$ calculated: 235.0325; found: 235.0329.

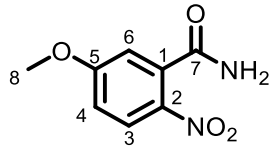
**3-Methyl-2-nitrobenzamide (10f)**



According to general protocol I **GPI** 3-methyl-2-nitrobenzoic acid (0.91 g, 5 mmol, 1.0 eq.) and 0.5 mL oxalyl chloride were reacted. The title compound was isolated as colourless amorphous solid (0.795 g, 4.4 mmol, 88%).

**$^1$H NMR (400 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 8.19 (s, 1H, C7-N*H*$_2$), 7.68 (s, 1H, C7-N*H*$_2$), 7.62–7.49 (m, 1H, *H*-4, *H*-5, *H*-6), 2.28 (s, 3H, *H*-8).
**$^{13}$C NMR (101 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 166.2, 149.2, 133.6, 130.4, 130.0, 129.5, 126.3, 16.7.
**HR-MS:** *m/z* for $C_8H_8N_2O_3$+$H^+$, $[M+H]^+$ calculated: 181.0608; found: 181.0608.
The spectroscopic data are in accordance with those reported in the literature.[6]

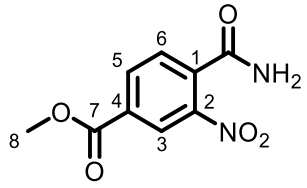
**5-Methoxy-2-nitrobenzamide (10g)**



According to general protocol I **GPI** 5-methoxy-2-nitrobenzoic acid (0.99 g, 5 mmol, 1.0 eq.) and 0.5 mL oxalyl chloride were reacted. The title compound was isolated as colourless amorphous solid (0.364 g, 1.9 mmol, 38%).

**$^1$H NMR (400 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 8.09–7.99 (m, 2H, C7-N*H*$_2$, *H*-3), 7.66 (s, 1H, C7-N*H*$_2$), 7.14 (dd, 1H, *J* = 9.1 Hz, 2.8 Hz, *H*-4), 7.05 (d, 1H, *J* = 2.8 Hz, *H*-6), 3.90 (s, 3H, *H*-8).
**$^{13}$C NMR (101 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 167.3, 162.9, 139.3, 135.9, 126.7, 114.7, 114.0, 56.4.
**HR-MS:** *m/z* for $C_8H_8N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 219.0376; found: 219.0374.

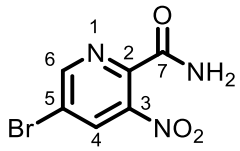
**Methyl 4-carbamoyl-3-nitrobenzoate (10h)**



According to general protocol I **GPI** 4-(methoxycarbonyl)-2-nitrobenzoic acid (0.90 g, 4 mmol, 1.0 eq.) and 0.4 mL oxalyl chloride were reacted. The title compound was isolated as colourless amorphous solid (0.759 g, 3.4 mmol, 85%).

**$^1$H NMR (300 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 8.43 (d, 1H, *J* = 1.6 Hz, *H*-3), 8.31–8.25 (m, 2H, C7-N*H*$_2$, *H*-5), 7.86 (s, 1H, C7-N*H*$_2$), 7.78 (d, 1H, *J* = 8.0 Hz, *H*-6), 3.92 (s, 3H, *H*-8).
**$^{13}$C NMR (75 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 166.5, 164.1, 147.0, 136.5, 133.7, 131.4, 129.7, 124.5, 53.0.
**HR-MS:** *m/z* for $C_9H_8N_2O_5$+H$^+$, [M+H]$^+$ calculated: 225.0506; found: 225.0506.
The spectroscopic data are in accordance with those reported in the literature.[7]

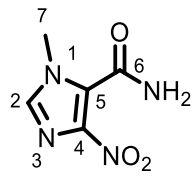
**5-Bromo-3-nitropicolinamide (10i)**



5-Bromo-3-nitropicolinonitrile (0.91 g, 4 mmol, 1.0 eq.) was dissolved in11 mL 96% sulphuric acid and heated at 70 °C for 6 h. After completion of the reaction the reaction mixture was cooled to room temperature and diluted with 50 mL cold water. The precipitates formed were filtered off and washed twice with water. Residual water was removed by azeotropic distillation with toluene. 0.922 g (3.8 mmol, 95%) of the product was isolated as colourless amorphous solid.

**$^1$H NMR (400 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 9.03 (d, 1H, *J* = 2.0 Hz, *H*-4), 8.84 (d, 1H, *J* = 2.0 Hz, *H*-6), 8.32 (s, 1H, C7-N*H*$_2$), 8.01 (s, 1H, C7-N*H*$_2$).
**$^{13}$C NMR (101 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 164.1, 152.5, 145.2, 144.0, 134.8, 121.8.
**HR-MS:** *m/z* for $C_6H_4{}^{79}BrN_3O_3$+H$^+$, [M+H]$^+$ calculated: 245.9509; found: 245.9509.

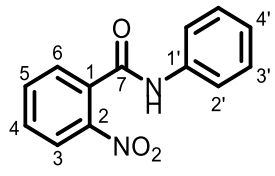
**1*H*-1-Methyl-4-nitro-imidazole-5-carboxamide (10j)**



1*H*-5-Chloro-1-methyl-4-nitro-imidazole (6.46 g, 40 mmol, 1.0 eq.) was suspended in 50 mL anhydrous ethanol. Potassium iodide (0.20 g, 1 mmol, 0.03 eq.) and potassium cyanide (5.21 g, 80 mmol, 2.0 eq.) were added and the reaction mixture was heated at reflux for 12 h. After completion 100 mL water was added. The aqueous layer was extracted three times with 100 mL dichloromethane. The organic fraction was washed twice with 100 mL water, once with 100 mL brine and dried over magnesium sulphate. The solvent was removed and the crude product was used without further purification.
The crude product was heated with 100 mL 90% sulphuric acid at 100°C. After completion the reaction mixture was cooled to room temperature and 100 mL water was added. The solution was neutralised with 1 M sodium bicarbonate. The precipitates formed were filtered off and washed twice with water and dried. 4.80 g (28mmol, 70%) of the product was isolated as colourless amorphous solid.

**$^1$H NMR (400 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 8.34 (s, 1H, C7-N*H*$_2$), 8.16 (s, 1H, C7-N*H*$_2$), 7.84 (s, 1H, *H*-2), 7.84 (s, 1H, *H*-2), 3.69 (s, 1H, *H*-7).
**$^{13}$C NMR (101 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 159.7, 143.1, 136.9, 127.7, 33.1.
**HR-MS:** *m/z* for $C_5H_6N_4O_3$+H$^+$, [M+H]$^+$ calculated: 171.0513; found: 171.0511.

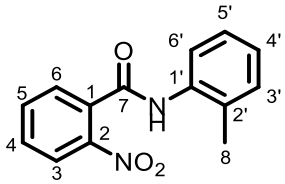
**2-Nitro-*N*-phenylbenzamide (11a)**



Aniline (5.10 g, 55 mmol, 1.8 eq.) and 6 mL pyridine was dissolved in 150 mL anhydrous THF. The reaction mixture was cooled to 0 °C and 2-nitrobenzoylchloride (5.56 g, 30 mmol, 1.0 eq.) in 50 mL anhydrous THF was added dropwise. After addition the reaction mixture was stirred for 24 h at room temperature. 30 mL of water was added and the THF was removed under reduced pressure. 50 mL of 1 M hydrochloric acid was added and the aqueous layer was extracted three times with 50 mL ethyl acetate. The combined organic fractions were washed four times with 50 mL 1 M hydrochloric acid, once with 50 mL brine and dried over magnesium sulphate. The solvent was removed and the crude product was recrystallised with ethanol. 5.447 g (22.5 mmol, 75%) of the product was obtained as colourless solid.

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.92 (s, 1H, C7-N*H*-), 8.06 (d, 1H, *J* = 8.34 Hz, *H*-3), 7.76–7.68 (m, 1H, *H*-5), 7.66–7.52 (m, 4H, *H*-4, *H*-6, *H*-2'), 7.42–7.33 (m, 2H, *H*-3'), 7.23–7.13 (m, 1H, *H*-4').
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 164.5, 146.4, 137.4, 134.1, 133.0, 130.9, 129.3, 128.7, 125.4, 124.9.
**HR-MS:** *m/z* for $C_{13}H_{10}N_2O_3+H^+$, $[M+H]^+$ calculated: 243.0764; found: 243.0764.
The spectroscopic data are in accordance with those reported in the literature.[8]

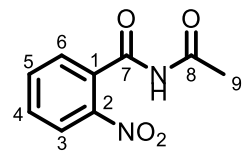
**2-Nitro-*N*-(o-tolyl)benzamide (11b)**



*o*-Toluidine (1.73 g, 15 mmol, 1.5 eq.) and 2 mL pyridine was dissolved in 50 mL anhydrous THF. The reaction mixture was cooled to 0 °C and 2-nitrobenzoylchloride (1.90 g, 10 mmol, 1.0 eq.) in 50 mL anhydrous THF was added dropwise. After addition the reaction mixture was stirred for 24 h at room temperature. 20 mL of water was added and the THF was removed under reduced pressure. 50 mL of 1 M hydrochloric acid was added and the aqueous layer was extracted three times with 50 mL ethyl acetate. The combined organic fractoions were washed four times with 50 mL 1 M hydrochloric acid, once with 50 mL brine and dried over magnesium sulphate. The solvent was removed and the crude product was recrystallised with ethanol. 1.139 g (4.4 mmol, 44%) of the product was obtained as colourless solid.

**$^1$H NMR (400 MHz, $CD_3CN$)** $\delta$ [ppm]: 8.39 (s, 1H, C7-N*H*-), 8.09 (dd, 1H, *J* = 8.2 Hz, 1.2 Hz, *H*-3), 7.81 (td, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-5), 7.75 (dd, 1H, *J* = 7.5 Hz, 1.6 Hz, *H*-6), 7.70 (td, 1H, *J* = 8.2 Hz, 1.6 Hz, *H*-4), 7.59 (d, 1H, *J* = 1.4 Hz, *H*-6'), 7.32–7.23 (m, 2H, *H*-3', *H*-4'), 7.18 (td, 1H, *J* = 7.4 Hz, 1.4 Hz, *H*-5'), 2.30 (s, 3H, *H*-8).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 164.9, 146.9, 135.4, 134.0, 132.9, 132.5, 130.9, 130.6, 129.1, 126.3, 126.3, 125.2, 124.4, 17.1.
**HR-MS:** *m/z* for $C_{14}H_{12}N_2O_3+H^+$, $[M+H]^+$ calculated: 257.0921; found: 257.0920.
The spectroscopic data are in accordance with those reported in the literature.[9]

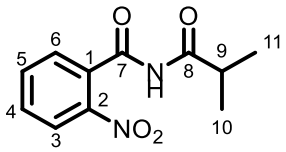
***N*-Acetyl-2-nitrobenzamide (5a)**



According to general protocol II **GPII** 2-nitrobenzamide (0.498 g, 3 mmol, 1.0 eq.) and anhydride (3 mL, 30 mmol, 10.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.587 g, 2.8 mmol, 94%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate = 1:1).

**$^1$H NMR (400 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 11.42 (s, 1H, C7-N*H*-), 8.19 (dd, 1H, *J* = 8.2 Hz, 1.2 Hz, *H*-3), 7.85 (td, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-5), 7.72 (dd, 1H, *J* = 8.2 Hz, 7.5 Hz, 1.5 Hz, *H*-4), 7.61 (dd, 1H, *J* = 7.5 Hz, 1.5 Hz, *H*-6), 2.15 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 170.6, 167.0, 145.2, 134.7, 132.8, 130.7, 128.4, 124.0, 24.5.
**HR-MS:** *m/z* for $C_9H_8N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 231.0376; found: 231.0378.
The spectroscopic data are in accordance with those reported in the literature.[10]

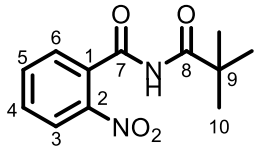
***N*-Isobutyryl-2-nitrobenzamide (5b)**



According to general protocol II **GPII** 2-nitrobenzamide (0.33 g, 2 mmol, 1.0 eq.) and anhydride (3 mL, 30 mmol, 15.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.431 g, 1.8 mmol, 90%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate = 2:1).

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 9.28 (s, 1H, C7-N*H*-), 8.22 (dd, 1H, *J* = 8.2 Hz, 1.2 Hz, *H*-3), 7.74 (td, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-5), 7.62 (ddd, 1H, *J* = 8.2 Hz, 7.5 Hz, 1.5 Hz, *H*-4), 7.43 (dd, 1H, *J* = 7.5 Hz, 1.5 Hz, *H*-6), 2.73 (hept, 1H, *J* = 6.9 Hz, *H*-9), 1.18 (s, 3H, *H*-10), 1.15 (s, 3H, *H*-11).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 176.6, 168.8, 145.3, 134.5, 132.9, 130.5, 127.7, 124.4, 36.1, 18.7.
**HR-MS:** *m/z* for $C_{11}H_{12}N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 259.0689; found: 259.0695.

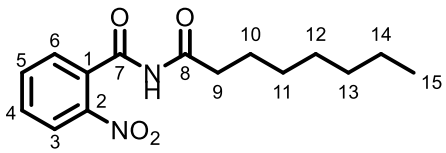
**2-Nitro-*N*-pivaloylbenzamide (5c)**



According to general protocol **GPII** 2-nitrobenzamide (0.33 g, 2 mmol, 1.0 eq.) and anhydride (4 mL, 20 mmol, 10.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.426 g, 1.7 mmol, 85%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate = 2:1).

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.87 (s, 1H, C7-N*H*-), 8.24 (dd, 1H, *J* = 8.2 Hz, 1.2 Hz, *H*-3), 7.74 (td, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-5), 7.61 (ddd, 1H, *J* = 8.2 Hz, 7.5 Hz, 1.4 Hz, *H*-4), 7.40 (dd, 1H, *J* = 7.5 Hz, 1.4 Hz, *H*-6), 1.22 (s, 9H, *H*-10).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 177.0, 169.2, 145.0, 134.5, 133.3, 130.2, 127.5, 124.2, 40.1, 26.8.
**HR-MS:** *m/z* for $C_{12}H_{14}N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 273.0846; found: 273.0847.

**2-Nitro-*N*-octanoylbenzamide (5d)**

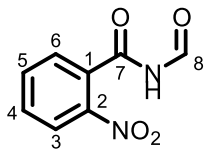


2-Nitrobenzamide (2.99 g, 18 mmol, 1.0 eq.) was suspended in nonanoic anhydride (16 mL, 54 mmol, 3.0 eq.) and 3 drops of sulphuric acid were added in a pressured vial. The reaction mixture was heated at 100 °C for 2 h. After completion of the reaction the solution was poured into 100 mL water and extracted three times with 150 mL ethyl acetate. The combined organic fractions were washed twice with 200 mL 1 M sodium bicarbonate solution, once with 200 mL brine and dried over sodium sulphate. The solvent was removed and the product was isolated as colourless amorphous solid (3.95 g, 13.5 mmol, 75%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 1:1).

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 9.14 (s, 1H, C7-N*H*-), 8.20 (dd, 1H, *J* = 8.2 Hz, 1.2 Hz, *H*-3), 7.73 (td, 1H, *J* = 7.6 Hz, 1.2 Hz, *H*-5), 7.62 (ddd, 1H, *J* = 8.2 Hz, 7.5 Hz, 1.4 Hz, *H*-4),

7.40 (dd, 1H, *J* = 7.5 Hz, 1.4 Hz, *H*-6), 2.56 (t, 2H, *J* = 7.5 Hz, *H*-9), 1.61 (p, 2H, *J* = 8.3 Hz, *H*-10), 1.37–1.21 (m, 8H, *H*-11, *H*-12, *H*-13, *H*-14), 0.92–0.82 (m, 3H, *H*-15).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 173.5, 167.8, 145.5, 134.4, 132.6, 130.7, 127.8, 124.5, 37.3, 31.7, 29.1, 29.0, 24.2, 22.7, 14.2.
**HR-MS:** *m/z* for $C_{15}H_{20}N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 315.1315; found: 315.1315.

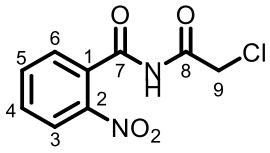
***N*-Formyl-2-nitrobenzamide (5e)**



*N*-(2-(Dimethylamino)vinyl)-2-nitrobenzamide (2.65 g, 12 mmol, 1.0 eq.) was suspended in 100 mL water. *p*-Toluenesulfonic acid (2.28 g, 12 mmol, 1.0 Eq.) in 20 mL water was added at room temperature. The resulting solution was stirred for 24 h. The precipitates formed were filtered, washed with water and residual dried by azeotropic distillation using toluene. 1.761 g (9.1 mmol, 76%) of the product were isolated as colourless amorphous solid.

**$^{1}$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 9.12 (s, 1H, *H*-8), 8.81 (s, 1H, C7-N*H*-), 8.21 (dd, 1H, *J* = 8.1 Hz, 1.3 Hz, *H*-3), 7.81 (td, 1H, *J* = 7.5 Hz, 1.3 Hz, *H*-5), 7.73 (ddd, 1H, *J* = 8.2 Hz, 7.5 Hz, 1.5 Hz, *H*-4), 7.59 (dd, 1H, *J* = 7.5 Hz, 1.4 Hz, *H*-6).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 166.4, 162.2, 146.1, 134.6, 132.2, 130.1, 128.4, 125.2.
**HR-MS:** *m/z* for $C_8H_6N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 217.0220; found: 217.0217.
The spectroscopic data are in accordance with those reported in the literature.[11]

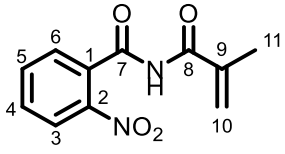
***N*-(2-Chloroacetyl)-2-nitrobenzamide (5f)**



2-Chloroacetyl chloride (0.8 mL, 4 mmol, 2.5 eq.) was added to 2-nitrobenzamide (0.66 mg, 4 mmol, 1.0 eq.). The suspension was stirred at 110 °C for 45 min. The reaction mixture was cooled down to room temperature and purified by flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1). 0.793 g (3.2 mmol, 80%) of the product was isolated as colourless amorphous solid.

**$^{1}$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 9.07 (s, 1H, C7-N*H*-), 8.24 (dd, 1H, *J* = 8.2 Hz, 1.2 Hz, *H*-3), 7.77 (td, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-5), 7.67 (ddd, 1H, *J* = 8.2 Hz, 7.5 Hz, 1.5 Hz, *H*-4), 7.46 (dd, 1H, *J* = 7.5 Hz, 1.5 Hz, *H*-6), 4.29 (s, 2H, *H*-9).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 166.8, 165.9, 145.5, 134.6, 131.7, 131.2, 127.8, 124.6, 43.4.
**HR-MS:** *m/z* for $C_9H_7{}^{35}ClN_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 264.9987; found: 264.9983.

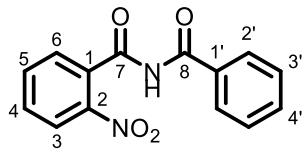
***N*-Methacryloyl-2-nitrobenzamide (5g)**



According to general protocol **GPII** 2-nitrobenzamide (0.50 g, 3 mmol, 1.0 eq.) and anhydride (1.3 mL, 9 mmol, 3.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.089 g, 0.4 mmol, 13%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate = 1:1).

**$^{1}$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 9.16 (s, 1H, C7-N*H*-), 8.26 (dd, 1H, *J* = 8.2 Hz, 1.2 Hz, *H*-3), 7.75 (td, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-5), 7.62 (ddd, 1H, *J* = 8.2 Hz, 7.5 Hz, 1.5 Hz, *H*-4), 7.42 (dd, 1H, *J* = 7.5 Hz, 1.5 Hz, *H*-6), 5.95–5.65 (m, 2H, *H*-10, *H*-10'), 1.91 (s, 3H, *H*-11).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 169.1, 165.8, 145.2, 138.7, 134.5, 133.1, 130.3, 127.5, 124.3, 18.3.
**HR-MS:** *m/z* for $C_{11}H_{10}N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 257.0533; found: 257.0535.

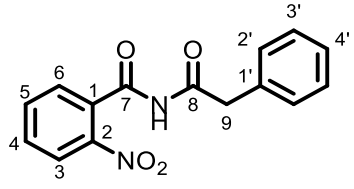
***N*-Benzoyl-2-nitrobenzamide (5h)**



According to general protocol **GPII** 2-nitrobenzamide (0.50 g, 3 mmol, 1.0 eq.) and anhydride (2.4 mL, 9 mmol, 3.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.348 g, 1.3 mmol, 43%) after flash column chromatography ($SiO_2$, dichloromethane:methanol 50:1).

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 9.43 (s, 1H, C7-N*H*-), 8.26 (dd, 1H, *J* = 8.3 Hz, 1.2 Hz, *H*-3), 7.87–7.82 (m, 2H, *H*-2'), 7.77 (td, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-5), 7.69–7.57 (m, 2H, *H*-4, *H*-6), 7.51–7.45 (m, 3H, *H*-3', *H*-4').
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 169.1, 165.0, 145.4, 134.5, 133.9, 133.1, 131.7, 130.4, 129.2, 128.1, 127.6, 124.3.
**HR-MS:** *m/z* for $C_{14}H_{10}N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 293.0533; found: 293.0535.

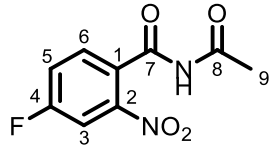
**2-Nitro-*N*-(2-phenylacetyl)benzamide (5i)**



Under argon atmosphere 2-Nitrobenzamide (0.66 g, 4 mmol, 1.0 eq.) was partially dissolved in 20 mL anhydrous THF. The reaction mixture was cooled to 0 °C, sodium hydride 60% dispersion on paraffin oil (*w*:*w*, 0.40 g, 10 mmol, 2.5 eq.) was added and the reaction was stirred at room temperature for 40 min. 2-Phenylacetyl chloride (1.2 mL, 9 mmol, 2.3 eq.) was slowly added and the reaction mixture was stirred at room temperature overnight. After completion of the reaction 20 mL saturated ammonium chloride solution was added. The aqueous layer was extracted three times with 15 mL ethyl acetate. The combined organic layers were washed once with 20 mL brine and dried over magnesium sulphate. The solvent was removed and the crude product was purified by flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1). 0.282 g (1.0 mmol, 25%) of the product was isolated as colourless amorphous solid.

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.76 (s, 1H, C7-N*H*-), 8.20 (dd, 1H, *J* = 8.3 Hz, 1.2 Hz, *H*-3), 7.71 (td, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-5), 7.61 (ddd, 1H, *J* = 8.3 Hz, 7.5 Hz, 1.2 Hz, *H*-4), 7.43–7.29 (m, 4H, *H*-6, *H*-2', *H*-4'), 7.28–7.21 (m, 2H, *H*-3'), 3.82 (s, 2H, *H*-9).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 170.6, 167.7, 145.4, 132.6, 132.5, 130.7, 129.7, 129.3, 128.0, 127.7, 124.5, 44.2.
**HR-MS:** *m/z* for $C_{15}H_{11}N_2O_4$-$H^+$, $[M-H]^-$ calculated: 283.0726; found: 283.0724.

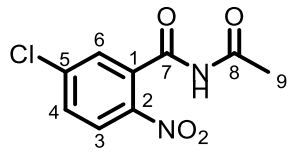
***N*-Acetyl-4-fluoro-2-nitrobenzamide (5j)**



According to general protocol **GPII** 4-fluoro-2-nitrobenzamide (0.37 g, 2 mmol, 1.0 eq.) and anhydride (1.9 mL, 20 mmol, 10.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.396 g, 1.8 mmol, 90%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1).

**$^1$H NMR (400 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 11.44 (s, 1H, C7-N*H*-), 8.12 (dd, 1H, *J* = 8.8 Hz, 2.4 Hz, *H*-3), 7.82–7.66 (m, 2H, *H*-5, *H*-6), 2.15 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 166.0, 161.7 (d, *J* = 250.8 Hz), 146.5 (d, *J* = 9.1 Hz), 130.7 (d, *J* = 8.9 Hz), 129.2, 121.7 (d, *J* = 21.8 Hz), 111.9 (d, *J* = 27.3 Hz).
**$^{19}$F NMR (282 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: -107.96 (q, *J* = 7.6 Hz).
**HR-MS:** *m/z* for $C_9H_7FN_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 249.0282; found: 249.0281.

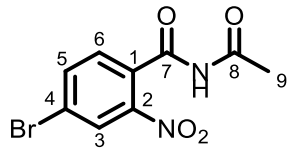
***N*-Acetyl-5-chloro-2-nitrobenzamide (5k)**



According to general protocol **GPII** 5-chloro-2-nitrobenzamide (0.30 g, 1.5 mmol, 1.0 eq.) and anhydride (1.4 mL, 15 mmol, 7.5 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.313 g, 1.3 mmol, 87%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 3:1).

**$^1$H NMR (400 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 11.51 (s, 1H, C7-N*H*-), 8.22 (d, 1H, *J* = 8.5 Hz, *H*-3), 7.88–7.70 (m, 2H, *H*-4, *H*-6), 2.14 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 170.6, 165.5, 143.6, 139.6, 134.6, 130.4, 128.1, 126.1, 24.3.
**HR-MS:** *m/z* for $C_9H_7{}^{35}ClN_2O_4$+Na$^+$, [M+Na]$^+$ calculated: 264.9987; found: 264.9985.

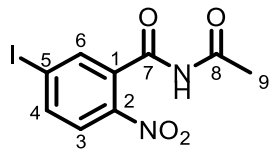
***N*-Acetyl-4-bromo-2-nitrobenzamide (5l)**



According to general protocol **GPII** 4-bromo-2-nitrobenzamide (0.49 g, 2 mmol, 1.0 eq.) and anhydride (1.9 mL, 20 mmol, 10.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.488 g, 1.7 mmol, 85%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1).

**$^1$H NMR (400 MHz, CDCl$_3$)** $\delta$ [ppm]: 9.38 (s, 1H, C7-N*H*-), 8.20 (d, 1H, *J* = 1.9 Hz, *H*-3), 7.85 (dd, 1H, *J* = 8.1 Hz, 1.9 Hz, *H*-5), 7.33 (d, 1H, *J* = 8.1 Hz, 1.9 Hz, *H*-6), 2.33 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, CDCl$_3$)** $\delta$ [ppm]: 171.0, 166.8, 146.1, 137.4, 131.0, 129.1, 127.7, 124.5, 24.9.
**HR-MS:** *m/z* for $C_9H_7{}^{79}BrN_2O_4$+Na$^+$, [M+Na]$^+$ calculated: 308.9481; found: 308.9494.

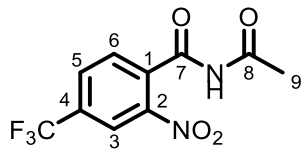
***N*-Acetyl-5-iodo-2-nitrobenzamide (5m)**



According to general protocol **GPII** 5-iodo-2-nitrobenzamide (0.58 g, 2 mmol, 1.0 eq.) and anhydride (1.9 mL, 20 mmol, 10.0 eq.) were reacted. The title compound was isolated as yellow amorphous solid (0.378 g, 1.1 mmol, 55%) after reverse phase flash column chromatography (0 → 100% acetonitrile).

**$^1$H NMR (400 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 11.45 (s, 1H, C7-N*H*-), 8.10 (dd, 1H, *J* = 8.6 Hz, 1.9 Hz, *H*-4), 8.03 (d, 1H, *J* = 1.9 Hz, *H*-6), 7.93 (d, 1H, *J* = 1.9 Hz, *H*-3), 2.14 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 170.6, 165.5, 144.6, 139.4, 136.5, 134.0, 125.5, 103.6, 24.4.
**HR-MS:** *m/z* for $C_9H_7IN_2O_4$+Na$^+$, [M+Na]$^+$ calculated: 356.9343; found: 356.9337.

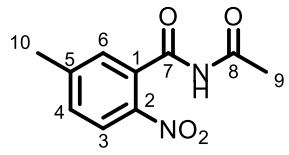
***N*-Acetyl-2-nitro-4-(trifluoromethyl)benzamide (5n)**



According to general protocol **GPII** 2-nitro-4-(trifluoromethyl)benzamide (0.25 g, 1 mmol, 1.0 eq.) and anhydride (1.0 mL, 11 mmol, 11.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.260 g, 0.9 mmol, 90%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1).

**$^1$H NMR (400 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 9.29 (s, 1H, C7-N*H*-), 8.47 (d, 1H, *J* = 1.7 Hz, *H*-3), 8.02–7.97 (m, 1H, *H*-5), 7.59 (d, 1H, *J* = 8.0 Hz, *H*-6), 2.30 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 170.4, 166.7, 145.5, 135.5, 133.3 (q, *J* = 34.6 Hz), 131.2 (q, *J* = 3.5 Hz), 128.8, 122.5 (q, *J* = 273.2 Hz), 121.9 (d, *J* = 3.8 Hz), 24.7.
**$^{19}$F NMR (282 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: -61.48.
**HR-MS:** *m/z* for $C_{10}H_7F_3N_2O_4$+Na$^+$, [M+Na]$^+$ calculated: 299.0250; found: 299.0249.

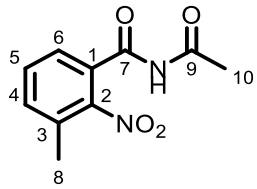
**_N_-Acetyl-5-methyl-2-nitrobenzamide (5o)**



According to general protocol **GPII** 5-methyl-2-nitrobenzamide (1.00 g, 6 mmol, 1.0 eq.) and anhydride (6 mL, 56 mmol, 9.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (1.07 g, 4.8 mmol, 80%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1).

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 9.11 (s, 1H, C7-N*H*-), 8.07 (d, 1H, *J* = 8.4 Hz, *H*-3), 7.39 (ddd, 1H, *J* = 8.4 Hz, 1.9 Hz, 0.8 Hz, *H*-4), 7.24 (d, 1H, *J* = 1.9 Hz, *H*-6), 2.47 (s, 3H, *H*-10), 2.36 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 171.1, 167.5, 146.2, 143.3, 132.3, 131.4, 128.3, 124.7, 25.0, 21.6.
**HR-MS:** *m/z* for $C_{10}H_{10}N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 245.0533; found: 245.0532.

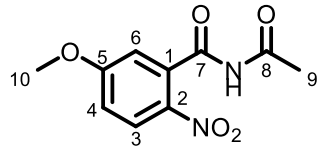
**_N_-Acetyl-3-methyl-2-nitrobenzamide (5p)**



According to general protocol **GPII** 3-methyl-2-nitrobenzamide (0.13 g, 0.7 mmol, 1.0 eq.) and anhydride (0.7 mL, 7 mmol, 10.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.140 g, 0.63 mmol, 90%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1).

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.98 (s, 1H, C7-N*H*-), 7.57–7.43 (m, 3H, *H*-4, *H*-5, *H*-6), 2.49 (s, 3H, *H*-10), 2.44 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 172.8, 164.8, 149.2, 135.3, 132.6, 131.0, 129.1, 125.6, 25.5, 18.2.
**HR-MS:** *m/z* for $C_{10}H_{10}N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 245.0533; found: 245.0534.

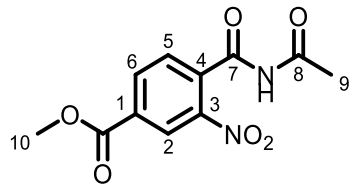
**_N_-Acetyl-5-methoxy-2-nitrobenzamide (5q)**



According to general protocol **GPII** 5-methoxy-2-nitrobenzamide (0.25 g, 1.3 mmol, 1.0 eq.) and anhydride (1.2 mL, 13 mmol, 13.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.299 g, 1.3 mmol, quant.) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1).

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 9.09 (s, 1H, C7-N*H*-), 8.17 (d, 1H, *J* = 9.2 Hz, *H*-3), 7.01 (dd, 1H, *J* = 9.2 Hz, 2.7 Hz, *H*-4), 6.86 (d, 1H, *J* = 2.7 Hz, *H*-6), 3.91 (s, 3H, *H*-10), 2.36 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 171.1, 167.2, 164.3, 138.3, 134.7, 127.2, 115.3, 112.9, 56.4, 25.0.
**HR-MS:** *m/z* for $C_{10}H_{10}N_2O_5$+$Na^+$, $[M+Na]^+$ calculated: 261.0482; found: 261.0478.

**Methyl 4-(acetylcarbamoyl)-3-nitrobenzoate (5r)**

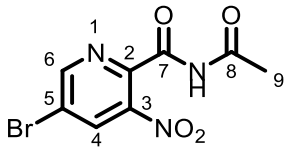


According to general protocol **GPII** methyl 4-carbamoyl-3-nitrobenzoate (0.67 g, 3 mmol, 1.0 eq.) and anhydride (2.8 mL, 30 mmol, 10 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.651 g, 2.5 mmol, 83%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1).

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 9.43 (s, 1H, C7-N*H*-), 8.79 (d, 1H, *J* = 1.5 Hz, *H*-2), 8.36 (dd, 1H, *J* = 7.9 Hz, 1.5 Hz, *H*-6), 7.52 (d, 1H, *J* = 7.9 Hz, *H*-5), 3.99 (s, 3H, *H*-10), 2.31 (s, 3H, *H*-9).

**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 170.8, 167.2, 164.4, 145.5, 135.8, 135.2, 132.8, 128.1, 125.6, 53.2, 24.8.
**HR-MS:** *m/z* for $C_{11}H_{10}N_2O_6$+$Na^+$, $[M+Na]^+$ calculated: 289.0431; found: 289.0428.

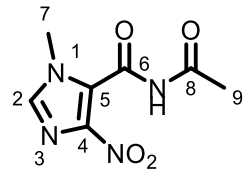
**2-(Acetylcarbamoyl)-5-bromo-3-nitropyridine (5s)**



According to general protocol **GPII** 5-bromo-3-nitropicolinamide (0.41 g, 2 mmol, 1.0 eq.) and anhydride (1.6 mL, 17 mmol, 8.5 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.374 g, 1.3 mmol, 65%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1).

**$^1$H NMR (600 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 11.73 (s, 1H, C7-N*H*-), 9.11 (d, 1H, *J* = 2.0 Hz, *H*-4), 8.98 (d, 1H, *J* = 2.0 Hz, *H*-6), 2.12 (s, 3H, *H*-9).
**$^{13}$C NMR (151 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 170.9, 165.3, 154.9, 147.7, 141.5, 135.3, 120.4, 23.9.
**HR-MS:** *m/z* for $C_8H_6{}^{79}BrN_3O_4$-$H^+$, $[M-H]^-$ calculated: 284.9469; found: 284.9468.

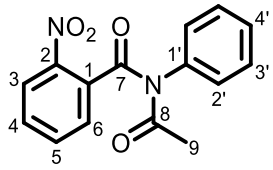
**1*H*-*N*-Acetyl-1-methyl-4-nitro-1*H*-imidazole-5-carboxamide (5t)**



According to general protocol **GPII** 1*H*-1-methyl-4-nitro-imidazole-5-carboxamide (2.99 g, 18 mmol, 1.0 eq.) and anhydride (16 mL, 54 mmol, 3.0 eq.) were reacted. The title compound was isolated as colourless amorphous solid (2.864 g, 13.5 mmol, 75%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 2:1).

**$^1$H NMR (400 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 11.67 (s, C6-N*H*-), 7.91 (s, 1H, *H*-2), 3.70 (s, 3H, *H*-7), 2.22 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 170.8, 158.7, 143.7, 137.7, 126.1, 33.3, 24.8.
**HR-MS:** *m/z* for $C_8H_8N_4O_4$-$H^+$, $[M-H]^-$ calculated: 213.0618; found: 213.0616.

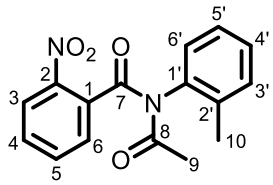
***N*-Acetyl-2-nitro-*N*-phenylbenzamide (5u)**



2-Nitro-*N*-phenylbenzamide (2.37 g, 10 mmol, 1.0 eq.) was dissolved in 50 mL anhydrous THF. 20 mL 1 M lithium bis(trimethylsilyl)amide (20 mmol, 2.0 eq.) was added at 0 °C and the reaction mixture was stirred for 15 min. Acetic anhydride (5 mL, 53 mmol, 5.3 eq.) was added dropwise, the reaction mixture was slowly allowed to warm to room temperature and stirred for additional 24 h. After completion the reaction mixture was poured into 50 mL water. The THF was removed and the aqueous layer was extracted three times with 50 mL ethyl acetate. The combined organic fractions were washed once with 50 mL brine, dried over magnesium sulphate and the solvent was removed. The title compound was isolated as beige amorphous solid (1.705 g, 6.0 mmol, 60%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 3:1).

**$^1$H NMR (400 MHz, $CD_3CN$)** $\delta$ [ppm]: 8.15 (dd, 1H, *J* = 8.2 Hz, 1.2 Hz, *H*-3), 7.76 (td, 1H, *J* = 7.6 Hz, 1.2 Hz, *H*-5), 7.62–7.52 (m, 2H, *H*-4, *H*-6), 7.51–7.36 (m, 5H, *H*-2', *H*-3', *H*-4'), 2.10 (s, 3H, *H*-9)
**$^{13}$C NMR (101 MHz, $CD_3CN$)** $\delta$ [ppm]: 173.2, 169.5, 145.5, 139.4, 135.6, 135.5, 130.8, 130.6, 130.1, 130.1, 128.5, 125.0, 26.3.
**HR-MS:** *m/z* for $C_{15}H_{12}N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 307.0689; found: 307.0683.

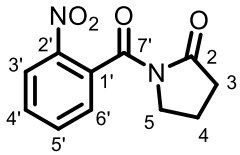
**_N_-Acetyl-2-nitro-_N_-(o-tolyl)benzamide (5v)**



2-Nitro-*N*-(o-tolyl)benzamide (0.48 g, 2 mmol, 1.0 eq.) was dissolved in 50 mL anhydrous THF. 5 mL 1 M lithium bis(trimethylsilyl)amide (4 mmol, 2.0 eq.) was added at 0 °C and the reaction mixture was stirred for 15 min. Acetic anhydride (1 mL, 6 mmol, 3.0 eq.) was added dropwise, the reaction mixture was slowly allowed to warm to room temperature and stirred for additional 24 h. After completion the reaction mixture was poured into 50 mL water. The THF was removed and the aqueous layer was extracted three times with 50 mL ethyl acetate. The combined organic fractions were washed once with 50 mL brine, dried over magnesium sulphate and the solvent was removed. The title compound was isolated as beige amorphous solid (0.258 g, 0.87 mmol, 44%) after flash column chromatography ($SiO_2$, cyclohexane:ethyl acetate 5:1).

**$^1$H NMR (400 MHz, $CD_3CN$)** $\delta$ [ppm]: 8.23 (dd, 1H, *J* = 8.3 Hz, 1.2 Hz, *H*-3), 7.80 (td, 1H, *J* = 7.6 Hz, 1.2 Hz, *H*-5), 7.64 (ddd, 1H, *J* = 8.3 Hz, 7.5 Hz, 1.4 Hz, *H*-4), 7.52 (dd, 1H, *J* = 7.6 Hz, 1.4 Hz, *H*-6), 7.44–7.33 (m, 4H, *H*-3', *H*-4', *H*-5', *H*-6'), 2.16 (s, 3H, *H*-9), 1.90 (s, 3H, *H*-10).
**$^{13}$C NMR (101 MHz, $CD_3CN$)** $\delta$ [ppm]: 173.0, 169.1, 138.3, 137.9, 136.0, 135.7, 132.4, 130.7, 130.6, 129.9, 128.5, 127.7, 125.1, 25.6, 17.7.
**HR-MS:** *m/z* for $C_{16}H_{14}N_2O_4$+$Na^+$, $[M+Na]^+$ calculated: 299.1026; found: 299.1022.
The spectroscopic data are in accordance with those reported in the literature.[12]

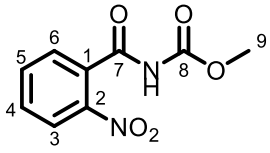
**1-(2-Nitrobenzoyl)pyrrolidin-2-one (5w)**



Under argon atmosphere pyrrolidin-2-one (0.643 g, 7.6 mmol, 1.5 eq.) was dissolved in 30 mL pyridine. 2-Nitrobenzoyl chloride (0.928, 5.0 mmol, 1.0 eq.) was added dropwise and the reaction mixture was heated at 60 °C for 24 h. 50 mL of water and 50 mL ethyl acetate was added and the organic layer was fractionated. The aqueous layer was extracted twice with 50 mL ethyl acetate. The combined organic fractions were washed four times with 50 mL 1 M hydrochloric, once with 50 mL brine and dried over magnesium sulphate. The solvent was removed and the crude product was further purified by flash column chromatography ($SiO_2$, cyclohexane:ethylacetate, 0 → 10% ethyl acetate). 0.268 g (1.1 mmol, 22%) of the product was isolated as colourless amorphous solid.

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.22 (dd, 1H, *J* = 8.3 Hz, 1.2 Hz, *H*-3'), 7.72 (td, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-5'), 7.60 (td, 1H, *J* = 8.3 Hz, 1.5 Hz, *H*-4'), 7.36 (dd, 1H, *J* = 7.5 Hz, 1.5 Hz, *H*-6'), 4.05 (t, 1H, *J* = 7.0 Hz, *H*-5), 2.54 (t, 1H, *J* = 8.0 Hz, *H*-3), 2.15 (p, 1H, *J* = 8.0 Hz, *H*-4).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 174.9, 166.6, 145.3, 134.5, 133.2, 130.2, 128.0, 124.2, 45.5, 32.9, 17.5.
**HR-MS:** *m/z* for $C_{11}H_{10}N_2O_4$+$H^+$, $[M+H]^+$ calculated: 235.0713; found: 235.0712.
The spectroscopic data are in accordance with those reported in the literature.[13]

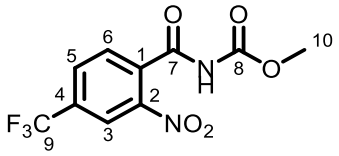
**Methyl (2-nitrobenzoyl)carbamate (5x)**



According to general protocol **GPIII** 2-nitrobenzamide (6.00 g, 36.1 mmol, 1.0 eq.) and oxalyl chloride (5.3 mL, 61 mmol, 1.7 eq.) were reacted. The title compound was isolated as colourless amorphous solid (7.48 g, 33.4 mmol, 92%) by filtration.

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.37 (s, 1H, C7-N*H*-), 8.25 (dd, 1H, *J* = 8.3 Hz, 1.3 Hz, *H*-3), 7.75 (td, 1H, *J* = 7.5 Hz, 1.3 Hz, *H*-5), 7.63 (td, 1H, *J* = 8.4 Hz, 1.5 Hz, *H*-4), 7.45 (dd, 1H, *J* = 7.5 Hz, 1.5 Hz, *H*-6), 3.68 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 168.4, 152.0, 145.3, 134.5, 132.1, 130.6, 127.9, 124.3, 53.5.
**HR-MS:** *m/z* for $C_9H_8N_2O_5$+$Na^+$, $[M+Na]^+$ calculated: 247.0325; found: 247.0326.
The spectroscopic data are in accordance with those reported in the literature.[14]

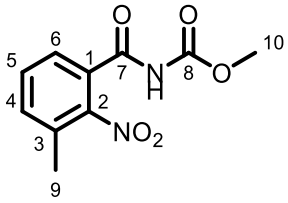
**Methyl (2-nitro-4-(trifluoromethyl)benzoyl)carbamate (5y)**



According to general protocol **GPIII** 2-nitro-4-(trifluoromethyl)benzamide (0.39 g, 1.7 mmol, 1.0 eq.) and oxalyl chloride (0.3 mL, 2.9 mmol, 1.7 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.441 g, 1.51 mmol, 89%) by filtration.

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.55–8.42 (m, 1H, C7-N*H*-), 8.01 (dd, 1H, *J* = 8.0 Hz, 1.0 Hz, *H*-5), 7.60 (d, 1H, *J* = 8.0 Hz, *H*-6), 3.69 (s, 3H, *H*-10).zz
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 167.4, 152.1, 145.3, 135.3, 133.1 (q, *J* = 34.7 Hz), 131.2 (q, *J* = 3.5 Hz), 128.8, 122.6 (q, *J* = 273.0 Hz), 121.7 (q, *J* = 3.9 Hz), 53.7.
**$^{19}$F NMR (282 MHz, $CDCl_3$)** $\delta$ [ppm]: -63.05.
**HR-MS:** *m/z* for $C_9H_8N_2O_5$+$Na^+$, $[M+Na]^+$ calculated: 247.0325; found: 247.0326.
The spectroscopic data are in accordance with those reported in the literature.[14]

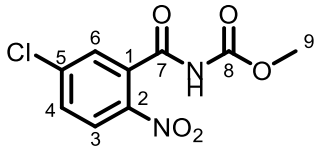
**Methyl (3-methyl-2-nitrobenzoyl)carbamate (5z)**



According to general protocol **GPIII** 3-methyl-2-nitrobenzamide (0.36 g, 2 mmol, 1.0 eq.) and oxalyl chloride (0.3 mL, 3 mmol, 1.7 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.444 g, 1.9 mmol, 95%) by filtration.

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.25 (s, 1H, C7-N*H*-), 7.53–7.40 (m, 2H, *H*-4, *H*-5), 7.38–7.32 (m, 1H, *H*-6), 3.76 (s, 3H, *H*-10), 2.51 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 166.4, 151.8, 147.8, 134.6, 133.1, 131.6, 130.9, 125.8, 53.6, 19.4.
**HR-MS:** *m/z* for $C_{10}H_{10}N_2O_5$+$Na^+$, $[M+Na]^+$ calculated: 261.0482; found: 261.0477.
The spectroscopic data are in accordance with those reported in the literature.[14]

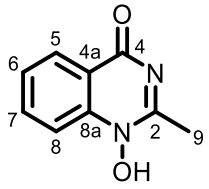
**Methyl (5-chloro-2-nitrobenzoyl)carbamate (5aa)**



According to general protocol **GPIII** 5-chloro-2-nitrobenzamide (0.40 g, 2 mmol, 1.0 eq.) and oxalyl chloride (0.3 mL, 3 mmol, 1.7 eq.) were reacted. The title compound was isolated as colourless amorphous solid (0.383 g, 1.5 mmol, 75%) by filtration.

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.26 (s, 1H, C7-N*H*-), 8.20 (d, 1H, *J* = 8.8 Hz, *H*-3), 7.58 (dd, 1H, *J* = 8.8 Hz, 2.2 Hz, *H*-4), 7.41 (d, 1H, *J* = 2.2 Hz, *H*-3), 3.70 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 166.8, 151.9, 143.6, 141.4, 133.5, 130.6, 128.0, 125.7, 53.6.
**HR-MS:** *m/z* for $C_9H_7{}^{35}ClN_2O_5$+$Na^+$, $[M+Na]^+$ calculated: 280.9936; found: 280.9934.

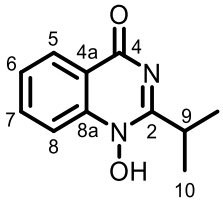
**1*H*-1-Hydroxy-2-methylquinazolin-4-one (6a)**



The title compound was prepared according to **GPIV** using 156 mg (0.75 mmol) of *N*-acetyl-2-nitrobenzamide. 120 mg (0.682 mmol, 91%) of the product were isolated as beige solid by flash column chromatography (silica gel, dichloromethane:methanol 6:1 + 0.5% acetic acid).

**$^1$H NMR (400 MHz, D$_2$O+NaOD)** $\delta$ [ppm]: 7.94–7.84 (m, 2H, *H*-6, *H*-8), 7.77–7.67 (m, 1H, *H*-7), 7.42–7.35 (m, 1H, *H*-5), 2.48 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, D$_2$O+NaOD)** $\delta$ [ppm]: 168.5, 156.4, 141.7, 134.1, 126.3, 126.0, 119.5, 116.1, 23.2.
**HR-MS:** *m/z* for $C_9H_8N_2O_2+H^+$, $[M+H]^+$ calculated: 177.0659; found: 177.0655.

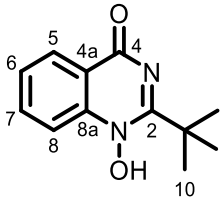
**1*H*-1-Hydroxy-2-isopropylquinazolin-4-one (6b)**



The title compound was prepared according to **GPIV** using 177 mg (0.75 mmol) of *N*-isobutyryl-2-nitrobenzamide. 138 mg (0.676 mmol, 90%) of the product were isolated as beige solid by flash column chromatography (silica gel, dichloromethane:methanol 20:1 + 0.5% acetic acid).

**$^1$H NMR (400 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 12.04 (s, 1H, N-O*H*), 8.05–7.98 (m, 1H, *H*-8), 7.84 (ddd, 1H, *J* = 8.5 Hz, 7.0 Hz, 1.5 Hz, *H*-6), 7.77 (d, 1H, *J* = 8.5 Hz, *H*-5), 7.50 (ddd, 1H, *J* = 8.1 Hz, 7.0 Hz, 1.2 Hz, *H*-7), 3.52 (hept, 1H, *J* = 6.8 Hz, *H*-9), 1.23 (d, 6H, *J* = 6.8 Hz, *H*-10).
**$^{13}$C NMR (101 MHz, DMSO-*d*$_6$)** $\delta$ [ppm]: 166.5, 165.2, 140.6, 134.0, 127.0, 126.0, 119.4, 114.5, 29.3, 19.9.
**HR-MS:** *m/z* for $C_{11}H_{12}N_2O_2+H^+$, $[M+H]^+$ calculated: 205.0972; found: 205.0966.

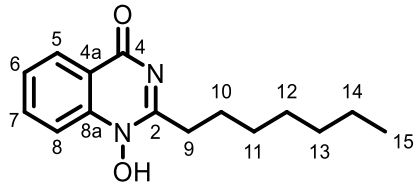
**1*H*-2-(*tert*-Butyl)-1-hydroxyquinazolin-4-one (6c)**



The title compound was prepared according to **GPIV** using 188 mg (0.75 mmol) of 2-nitro-*N*-pivaloylbenzamide. 128 mg (0.587 mmol, 78%) of the product were isolated as beige solid by flash column chromatography (silica gel, dichloromethane:methanol 20:1 + 0.5% acetic acid).

**$^1$H NMR (400 MHz, D$_2$O)** $\delta$ [ppm]: 8.10–8.03 (m, 1H, *H*-8), 7.98 (dd, 1H, *J* = 8.1 Hz, 1.5 Hz, *H*-5), 7.80 (ddd, 1H, *J* = 8.7 Hz, 7.1 Hz, 1.5 Hz, *H*-7), 7.48 (ddd, 1H, *J* = 8.1 Hz, 7.1 Hz, 1.1 Hz, *H*-6), 1.46 (s, 9H, *H*-10).
**$^{13}$C NMR (101 MHz, D$_2$O)** $\delta$ [ppm]: 168.9, 163.8, 143.7, 134.0, 126.4, 125.8, 119.2, 116.7, 38.1, 26.8.
**HR-MS:** *m/z* for $C_{12}H_{14}N_2O_2+H^+$, $[M+H]^+$ calculated: 219.1125; found: 219.1128.

**1*H*-2-Heptyl-1-hydroxyquinazolin-4-one (6d)**

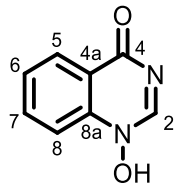


The title compound was prepared according to **GPIV** using 220 mg (0.75 mmol) of 2-nitro-*N*-octanoylbenzamide. 103 mg (0.396 mmol, 52%) of the product were isolated as beige solid by flash column

chromatography (reversed phase silica gel, water:acetonitrile 15% → 35% acetonitrile).

**$^1$H NMR (400 MHz, $CD_3OD$)** $\delta$ [ppm]: 8.23 (dd, 1H, *J* = 7.9 Hz, 1.5 Hz, *H*-8), 8.15 (d, 1H, *J* = 8.4 Hz, *H*-5), 7.97 (ddd, 1H, *J* = 8.4 Hz, 7.2 Hz, 1.5 Hz, *H*-6), 7.67 (ddd, 1H, *J* = 7.9 Hz, 7.2 Hz, 1.1 Hz, *H*-6), 3.05–2.93 (m, 2H, *H*-9), 1.91–1.79 (m, 2H, *H*-10), 1.54–1.25 (m, 8H, *H*-11, *H*-12, *H*-13, *H*-14, *H*-15), 0.96–0.86 (m, 3H, *H*-15).
**$^{13}$C NMR (101 MHz, $CD_3OD$)** $\delta$ [ppm]: 156.2, 148.4, 143.1, 136.5, 129.2, 128.3, 121.6, 117.1, 114.7, 32.8, 31.8, 30.5, 30.0, 27.1, 23.7, 14.4.
**HR-MS:** *m/z* for $C_{15}H_{20}N_2O_2+H^+$, $[M+H]^+$ calculated: 261.1598; found: 261.1590.

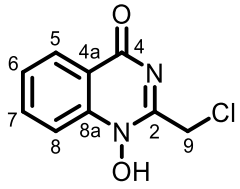
### 1*H*-1-Hydroxyquinazolin-4-one (6e)




The title compound was prepared according to **GPIV** using 145 mg (0.75 mmol) of 2-nitro-*N*-octanoylbenzamide. 128 mg (0.488 mmol, 65%) of the product were isolated as beige solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 10% acetonitrile) as formic acid adduct.

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 8.53 (s, 1H, *H*-2)8.03 (dd, 1H, *J* = 8.1 Hz, 1.4 Hz, *H*-8), 7.95 (dd, 1H, *J* = 8.5 Hz, 1.2 Hz, *H*-5), 7.88 (ddd, 1H, *J* = 8.5 Hz, 7.1 Hz, 1.4 Hz, *H*-6), 7.67 (ddd, 1H, *J* = 8.1 Hz, 7.1 Hz, 1.2 Hz, *H*-6).
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 169.6, 145.9, 141.0, 134.4, 128.8, 126.4, 119.9, 116.2.
**HR-MS:** *m/z* for $C_8H_6N_2O_2+H^+$, $[M+H]^+$ calculated: 163.0502; found: 163.0501.

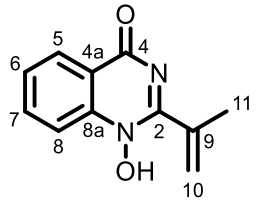
### 1*H*-2-(Chloromethyl)-1-hydroxyquinazolin-4-one (6f)




The title compound was prepared according to **GPIV** using 184 mg (0.75 mmol) of *N*-(2-chloroacetyl)-2-nitrobenzamide. 38 mg (0.181 mmol, 24%) of the product were isolated as beige solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 7.99 (d, 1H, *J* = 8.5 Hz, *H*-8), 7.90 (dd, 1H, *J* = 8.1 Hz, 1.4 Hz, *H*-5), 7.81–7.54 (m, 1H, *H*-6), 7.50–7.44 (m, 1H, *H*-7), 4.72 (s, 2H, *H*-9).
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 168.5, 152.9, 141.8, 134.3, 127.4, 126.1, 120.2, 116.6, 40.7.
**HR-MS:** *m/z* for $C_9H_7{}^{35}ClN_2O_2-H^+$, $[M-H]^-$ calculated: 209.0123; found: 209.0124.

### 1*H*-1-Hydroxy-2-(prop-1-en-2-yl)quinazolin-4-one (6g)

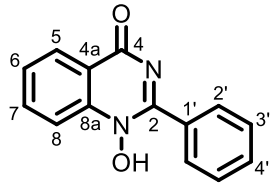



The title compound was prepared according to **GPIV** using 175 mg (0.75 mmol) of *N*-methacryloyl-2-nitrobenzamide. 102 mg (0.505 mmol, 67%) of the product were isolated as beige solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 8.24–8.18 (m, 1H, *H*-8), 8.12–8.07 (m, 1H, *H*-5), 7.98–7.92 (m, 1H, *H*-6), 7.66 (ddd, 1H, *J* = 8.1 Hz, 7.2 Hz, 1.4 Hz, *H*-7), 5.69–5.63 (m, 2H, *H*-10), 2.26 (s, 3H, *H*-11).
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 166.7, 155.9, 143.1, 137.5, 136.4, 129.3, 128.3, 123.9, 121.7, 117.7, 20.2.

**HR-MS:** *m/z* for $C_{11}H_{10}N_2O_2+H^+$, $[M+H]^+$ calculated: 203.0815; found: 203.0809.

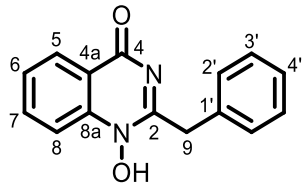
### 1*H*-1-Hydroxy-2-phenylquinazolin-4-one (6h)




The title compound was prepared according to **GPIV** using 149 mg (0.55 mmol due to the low solubility of the starting material) of *N*-benzoyl-2-nitrobenzamide. 103 mg (0.433 mmol, 79%) of the product were isolated as beige solid by flash column chromatography (silica gel, dichloromethane:methanol 20:1 + 0.5% acetic acid).

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 7.88-7.78 (m, 2H, *H*-8, H-5), 7.65–7.55 (m, 3H, *H*-6H, *H*-2'), 7.41–7.26 (m, 3H, *H*-3', *H*-4'), 7.29 (ddd, *J* = 8.1 Hz, 7.2 Hz, 1.1 Hz, *H*-7).
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 168.4, 154.3, 142.0, 134.1, 133.2, 130.1, 128.8, 128.2, 126.9, 125.9, 119.8, 116.7.
**HR-MS:** *m/z* for $C_{14}H_{10}N_2O_2+H^+$, $[M+H]^+$ calculated: 239.0815; found: 239.0811.

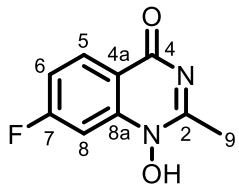
### 1*H*-2-Benzyl-1-hydroxyquinazolin-4-one (6i)




The title compound was prepared according to **GPIV** using 213 mg (0.75 mmol) of *N*-(2-phenylacetyl)-2-nitrobenzamide. 154 mg (0.611 mmol, 81%) of the product were isolated as beige solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 7.99–7.93 (m, 2H, *H*-5, *H*-8), 7.75–7.68 (m, 1H, *H*-6), 7.44–7.37 (m, 1H, *H*-7), 7.32–7.26 (m, 4H, *H*-2', *H*-3'), 7.26–7.18 (m, 1H, *H*-4'), 4.24 (s, 2H, *H*-9).
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 168.7, 157.3, 142.0, 136.2, 134.1, 128.7, 128.6, 126.8, 126.7, 126.1, 119.7, 116.6.
**HR-MS:** *m/z* for $C_{15}H_{12}N_2O_2+H^+$, $[M+H]^+$ calculated: 253.0970; found: 253.0972.

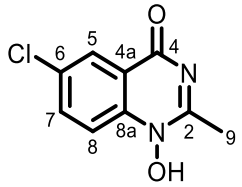
### 1*H*-7-Fluoro-1-hydroxy-2-methylquinazolin-4-one (6j)




The title compound was prepared according to **GPIV** using 170 mg (0.75 mmol) of *N*-acetyl-4-fluoro-2-nitrobenzamide. 107 mg (0.552 mmol, 74%) of the product were isolated as light yellow solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 7.62 (dd, 1H, *J* = 9.0 Hz, 5.9 Hz, *H*-6), 7.34 (dd, 1H, *J* = 10.3 Hz, 2.5 Hz, *H*-8), 6.85 (td, 1H, *J* = 8.7 Hz, 2.5 Hz, *H*-5), 2.36 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 167.4, 166.8, 164.3, 157.4, 143.1 (d, *J* = 12.3 Hz), 129.1 (d, *J* = 10.6 Hz), 116.0, 101.9 (d, *J* = 27.3 Hz), 18.9.
**$^{19}$F NMR (282 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: -102.96.
**HR-MS:** *m/z* for $C_9H_7FN_2O_2+H^+$, $[M+H]^+$ calculated: 195.0564; found: 195.0563.

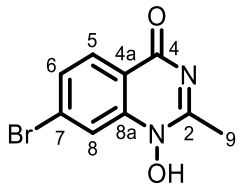
**1*H*-6-Chloro-1-hydroxy-2-methylquinazolin-4-one (6k)**



The title compound was prepared according to **GPIV** using 181 mg (0.75 mmol) of *N*-acetyl-5-chloro-2-nitrobenzamide. 121 mg (0.576 mmol, 77%) of the product were isolated as beige solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 7.80 (d, 1H, *J* = 9.0 Hz, *H*-8), 7.66 (d, 1H, *J* = 2.3 Hz, *H*-5), 7.54 (dd, 1H, *J* = 9.0 Hz, 2.3 Hz, *H*-7), 2.47 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 167.1, 156.7, 140.3, 134.0, 131.3, 125.0, 120.4, 118.3, 18.9.
**HR-MS:** *m/z* for $C_9H_7{}^{35}ClN_2O_2+H^+$, $[M+H]^+$ calculated: 211.0269; found: 211.0266.

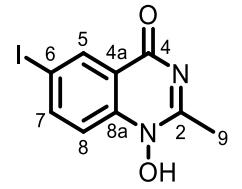
**1*H*-7-Bromo-1-hydroxy-2-methylquinazolin-4-one (6l)**



The title compound was prepared according to **GPIV** using 214 mg (0.75 mmol) of *N*-acetyl-4-bromo-2-nitrobenzamide. 158 mg (0.620 mmol, 83%) of the product were isolated as beige solid by flash column chromatography (reverse phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 8.05 (d, 1H, *J* = 1.9 Hz, *H*-8), 7.72 (d, 1H, *J* = 8.6 Hz, *H*-6), 7.44 (dd, 1H, *J* = 8.6 Hz, 1.9 Hz, *H*-6), 2.53 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 168.0, 157.5, 142.2, 129.4, 128.3, 127.9, 118.9, 118.3, 19.3.
**HR-MS:** *m/z* for $C_9H_7{}^{79}BrN_2O_2+H^+$, $[M+H]^+$ calculated: 254.9764; found: 254.9757.

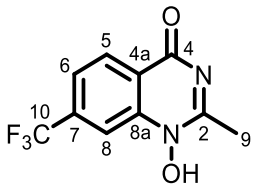
**1*H*-1-Hydroxy-6-iodo-2-methylquinazolin-4-one (6m)**



The title compound was prepared according to **GPIV** using 250 mg (0.75 mmol) of *N*-acetyl-5-iodo-2-nitrobenzamide. 113 mg (0.374 mmol, 50%) of the product were isolated as colourless solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 8.02 (d, 1H, *J* = 2.0 Hz, *H*-5), 7.84 (dd, 1H, *J* = 8.9 Hz, 2.0 Hz, *H*-7), 7.61 (d, 1H, *J* = 8.9 Hz, *H*-8), 2.51 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 166.8, 156.9, 142.3, 141.1, 134.7, 121.0, 118.2, 90.2, 19.5.
**HR-MS:** *m/z* for $C_9H_7IN_2O_2+H^+$, $[M+H]^+$ calculated: 302.9625; found: 302.9623.

**1*H*-1-Hydroxy-2-methyl-7-(trifluoromethyl)quinazolin-4-one (6n)**

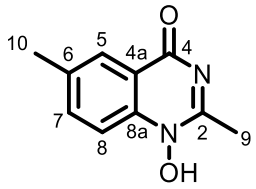


The title compound was prepared according to **GPIV** using 207 mg (0.75 mmol) of *N*-acetyl-2-nitro-4-(trifluoromethyl)benzamide. 169 mg (0.693 mmol, 92%) of the product were isolated as beige solid by flash column chromatography (silica gel, dichloromethane:methanol 20:1 + 0.5% acetic acid).

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 8.08 (d, 1H, *J* = 8.2 Hz, *H*-5), 7.77 (s, 1H, *H*-8), 7.62 (dd, 1H, *J* = 8.2 Hz, 1.7 Hz, *H*-6), 2.43 (s, 3H, *H*-9).

**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 165.0, 159.4, 140.9, 132.7 (q, *J* = 32.5 Hz), 128.7, 123.3 (q, *J* = 272.6 Hz), 121.8, 121.1, 111.8, 20.1.
**$^{19}$F NMR (282 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: -64.06.
**HR-MS:** *m/z* for $C_{10}H_7F_3N_2O_2$+$H^+$, $[M+H]^+$ calculated: 245.0532; found: 245.0533.

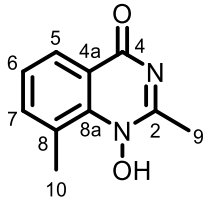
### 1*H*-1-Hydroxy-2,6-dimethylquinazolin-4-one (6o)




The title compound was prepared according to **GPIV** using 167 mg (0.75 mmol) of *N*-acetyl-5-methyl-2-nitrobenzamide. 128 mg (0.673 mmol, 89%) of the product were isolated as colourless solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 10% → 90% acetonitrile) as formate adduct.

**$^{1}$H NMR (400 MHz, $D_2O$)** $\delta$ [ppm]: 7.80 (d, 1H, *J* = 8.8 Hz, *H*-8), 7.63 (d, 1H, *J* = 2.0 Hz, *H*-5), 7.53 (dd, 1H, *J* = 8.8 Hz, 2.0 Hz, *H*-7), 2.52 (s, 3H, *H*-10), 2.34 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $D_2O$)** $\delta$ [ppm]: 168.1, 155.2, 139.8, 136.9, 135.4, 125.1, 119.4, 116.1, 20.3, 19.4.
**HR-MS:** *m/z* for $C_{10}H_{10}N_2O_2$+$H^+$, $[M+H]^+$ calculated:191.0815; found: 191.0814.

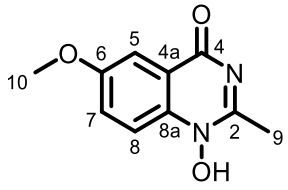
### 1*H*-1-Hydroxy-2,8-dimethylquinazolin-4-one (6p)




The title compound was prepared according to **GPIV** using 153 mg (0.69 mmol due to the low solubility of the starting material) of *N*-acetyl-2-methyl-6-nitrobenzamide. 96 mg (0.505 mmol, 73%) of the product were isolated as colourless solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^{1}$H NMR (400 MHz, $D_2O$)** $\delta$ [ppm]: 7.86 (dd, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-5), 7.50 (dd, 1H, *J* = 7.5 Hz, 1.2 Hz, *H*-7), 7.31 (t, 1H, *J* = 7.5 Hz, *H*-6), 2.85 (s, 3H, *H*-10), 2.50 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $D_2O$)** $\delta$ [ppm]: 168.7, 157.5, 140.9, 137.9, 129.4, 126.3, 124.5, 121.0, 23.0, 19.9
**HR-MS:** *m/z* for $C_{10}H_{10}N_2O_2$+$H^+$, $[M+H]^+$ calculated:191.0815; found: 191.0814.

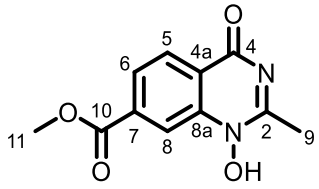
### 1*H*-1-Hydroxy-6-methoxy-2-methylquinazolin-4-one (6q)




The title compound was prepared according **GPIV** using 175 mg (0.75 mmol) of *N*-acetyl-5-methoxy-2-nitrobenzamide. 119 mg (0.578 mmol, 77%) of the product were isolated as colourless solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^{1}$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 7.80 (d, 1H, *J* = 9.3 Hz, *H*-8), 7.21 (dd, 1H, *J* = 9.3 Hz, 2.8 *H*-7), 7.14 (d, 1H, *J* = 2.8 Hz, *H*-5), 3.73 (s, 3H, *H*-10), 2.41 (s, 3H, *H*-9).Hz
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 167.7, 157.0, 154.0, 136.7, 123.5, 120.6, 118.2, 105.7, 55.5, 18.7.
**HR-MS:** *m/z* for $C_{10}H_{10}N_2O_3$+$H^+$, $[M+H]^+$ calculated: 207.0764; found: 207.0762.

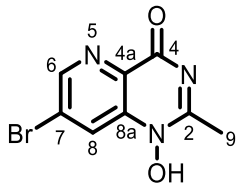
**Methyl 1-hydroxy-2-methyl-4-oxo-1,4-dihydroquinazoline-7-carboxylate (6r)**



The title compound was prepared according **GPIV** using 200 mg (0.75 mmol) of methyl 4-(acetylcarbamoyl)-3-nitrobenzoate. 150 mg (0.641 mmol, 85%) of the product were isolated as light yellow solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 8.44 (d, 1H, *J* = 1.6 Hz, *H*-8), 8.06 (d, 1H, *J* = 8.3 Hz *H*-5), 7.89 (dd, 1H, *J* = 8.3 Hz, 1.6 Hz, *H*-7), 3.28 (s, 3H, *H*-11), 2.53 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $D_2O$+NaOD)** $\delta$ [ppm]: 174.0, 168.4, 157.2, 142.0, 141.9, 126.5, 126.2, 120.9, 116.7, 48.8.3, 19.2.
**HR-MS:** *m/z* for $C_{11}H_{10}N_2O_4+H^+$, $[M+H]^+$ calculated: 235.0713; found: 235.0708.

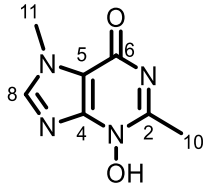
**1*H*-7-Bromo-1-hydroxy-2-methylpyrido[3,2-*d*]pyrimidin-4-one (6s)**



The title compound was prepared according to **GPIV** using 215 mg (0.75 mmol) of *N*-acetyl-6-bromo-3-nitropicolinamide. 157 mg (0.616 mmol, 82%) of the product were isolated as light yellow solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, $D_2O$)** $\delta$ [ppm]: 8.61 (d, 1H, *J* = 2.1 Hz, *H*-8), 8.53 (dd, 1H, *J* = 2.1 Hz, 2.0 Hz, *H*-6), 2.50 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $D_2O$)** $\delta$ [ppm]: 167.1, 158.5, 149.6, 139.2, 132.8, 127.9, 125.0, 19.1.
**HR-MS:** *m/z* for $C_8H_6{}^{79}BrN_3O_2+H^+$, $[M+H]^+$ calculated: 255.9716; found: 255.9707.

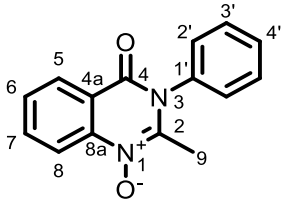
**6*H*-3-Hydroxy-2,7-dimethyl-3,7-dihydropurin-6-one (6t)**



The title compound was prepared according to **GPIV** using 159 mg (0.75 mmol) of 1*H*-*N*-Acetyl-1-methyl-4-nitro-imidazole-5-carboxamide. 196 mg (0.430 mmol, 57%) of the product were isolated as beige solid by flash column chromatography (silica gel, dichloromethane:methanol 50% → 100% methanol) as a acetate adduct.

**$^1$H NMR (400 MHz, $D_2O$)** $\delta$ [ppm]: 7.97 (s, 1H, *H*-8), 4.00 (s, 3H, *H*-11), 2.50 (s, 3H, *H*-10).
**$^{13}$C NMR (101 MHz, $D_2O$)** $\delta$ [ppm]: 158.2, 150.9, 146.3, 142.2, 113.9, 30.9, 15.7.
**HR-MS:** *m/z* for $C_7H_8N_4O_2+H^+$, $[M+H]^+$ calculated: 181.0720; found: 181.0714.

**2-Methyl-4-oxo-3-phenyl-3,4-dihydroquinazoline 1-oxide (7a)**

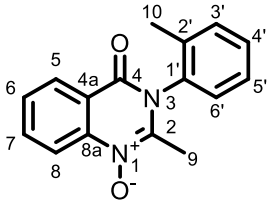


The title compound was prepared according to **GPIV** using 213 mg (0.75 mmol) of *N*-acetyl-2-nitro-*N*-phenylbenzamide. 81 mg (0.321 mmol, 43%) of the product were isolated as light yellow oil by flash column chromatography (reversed phase silica gel, water:acetonitrile 10% → 90% acetonitrile).

**$^1$H NMR (400 MHz, $CD_3CN$)** $\delta$ [ppm]: 8.43–8.36 (m, 1H, *H*-8), 8.24–8.17 (m, 1H, *H*-5), 8.01–7.92 (m, 1H, *H*-6), 7.52–7.74 (m, 4H, *H*-7, *H*-4', *H*-3'), 7.45–7.37 (m, 2H, *H*-2'), 2.38 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $CD_3CN$)** $\delta$ [ppm]: 164.5, 158.9, 150.5, 143.2, 138.0, 136.6, 131.0, 130.8, 130.0, 129.3, 128.2, 119.8, 17.7.

**HR-MS:** *m/z* for $C_{15}H_{12}N_2O_2+H^+$, $[M+H]^+$ calculated: 253.0972; found: 253.0966.

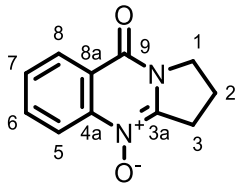
**2-Methyl-4-oxo-3-(o-tolyl)-3,4-dihydroquinazoline 1-oxide (7b)**



The title compound was prepared according to **GPIV** using 217 mg (0.75 mmol) of *N*-acetyl-2-nitro-*N*-(o-tolyl)benzamide. 172 mg (0.646 mmol, 86%) of the product were isolated as light yellow oil by flash column chromatography (reversed phase silica gel, water:acetonitrile 10% → 90% acetonitrile).

**$^1$H NMR (400 MHz, $CD_3OD$)** $\delta$ [ppm]: 8.48–8.41 (m, 1H, *H*-8), 8.32–8.23 (m, 1H, *H*-5), 8.11–8.03 (m, 1H, *H*-6), 7.80–7.73 (m, 1H, *H*-5), 7.52–7.47 (m, 2H, *H*-6', *H*-4'), 7.46–7.39 (m, 1H, *H*-5'), 7.37–7.31 (m, 1H, *H*-3'), 3.35 (s, 3H, *H*-10), 2.14 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, $CD_3OD$)** $\delta$ [ppm]: 164.7, 158.9, 151.9, 143.2, 137.3, 137.1, 136.9, 132.8, 131.5, 130.7, 129.4, 129.0, 128.8, 121.3, 119.8, 17.3.
**HR-MS:** *m/z* for $C_{16}H_{14}N_2O_2+H^+$, $[M+H]^+$ calculated: 267.1128; found: 267.1122.

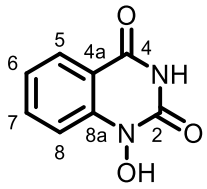
**9-Oxo-1,2,3,9-tetrahydropyrrolo[2,1-*b*]quinazoline 4-oxide (7c)**



The title compound was prepared according to **GPIV** using 161 mg (0.75 mmol) of 1-(2-Nitrobenzoyl)pyrrolidin-2-one. 109 mg (0.540 mmol, 72%) of the product were isolated as beige solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 5% → 10% acetonitrile).

**$^1$H NMR (400 MHz, $CDCl_3$)** $\delta$ [ppm]: 8.46 (dd, 1H, *J* = 7.3 Hz, 1.1 Hz, *H*-5), 7.63 (dd, 1H,, *J* = 8.0 Hz, 1.5 Hz, *H*-8), 7.95–7.87 (m, 1H, *H*-7), 7.68–7.59 (m, 1H, *H*-6), 4.39–4,27 (m, 2H, *H*-1), 3.59 (t, 2H, *H*-3), 2.48–2.32 (m, 2H, *H*-2).
**$^{13}$C NMR (101 MHz, $CDCl_3$)** $\delta$ [ppm]: 156.6, 150.1, 143.3, 135.3, 128.9, 127.3, 120.7, 118.8, 49.2, 29.6, 19.4.
**HR-MS:** *m/z* for $C_{11}H_{10}N_2O_2+H^+$, $[M+H]^+$ calculated: 203.0815; found: 203.0814.

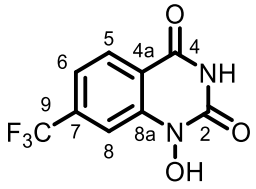
**1*H*,3*H*-1-Hydroxyquinazoline-2,4-dione (8a)**



The title compound was prepared according to **GPIV** using 168 mg (0.75 mmol) of methyl (2-nitrobenzoyl)carbamate. 103 mg (0.579 mmol, 77%) of the product were isolated as colourless solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 11.63 (s, 1H, *N*-H), 7.94 (dd, 1H, *J* = 7.9 Hz, 1.5 Hz, *H*-8), 7.80–7.72 (m, 1H, *H*-6), 7.52–7.43 (m, 1H, *J* = 8.4 Hz, 1.0 Hz, *H*-5), 7.21–7.20 (m, 1H, *H*-7), 4.92 (s, 1H, *O*-H).
**$^{13}$C NMR (101 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 161.3, 148.2, 141.6, 135.5, 127.3, 122.7, 114.1, 112.5.
**HR-MS:** *m/z* for $C_8H_6N_2O_3+H^+$, $[M+H]^+$ calculated: 179.0451; found: 179.0451.
The spectroscopic data are in accordance with those reported in the literature.[15]

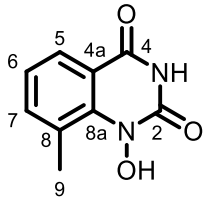
**1*H*,3*H*-1-Hydroxy-7-(trifluoromethyl)quinazoline-2,4-dione (8b)**



The title compound was prepared according to **GPIV** using 44 mg (0.15 mmol) of methyl (2-nitro-4-(trifluoromethyl)benzoyl)carbamate. 22 mg (0.0894 mmol, 59%) of the product were isolated as colourless solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 12.08–11.42 (m, 2H, *N*-H, *O*-H), 8.13 (d, 1H, *J* = 8.2 Hz, *H*-8), 7.67 (d, 1H, *J* = 1.7 Hz, *H*-5), 7.57 (dd, 1H, *J* = 8.2 Hz, 1.7 Hz, *H*-7), 2.52 (s, 3H, *H*-10), 2.34 (s, 3H, *H*-9).
**$^{13}$C NMR (101 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 160.5, 148.1, 142.0, 134.6 (q, *J* = 37.3 Hz), 129.0, 124.8, 122.1, 118.9 (q, *J* = 140.3 Hz), 117.2, 109.2 (q, *J* = 4.1 Hz).
**$^{19}$F NMR (282 MHz, DMSO-$d_6$** $\delta$ [ppm]: -62.02.
**HR-MS:** *m/z* for $C_9H_5F_3N_2O_3$-$H^+$, $[M-H]^-$ calculated: 245.0180; found: 245.0179.

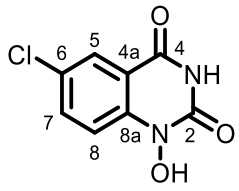
**1*H*,3*H*-1-Hydroxy-8-methylquinazoline-2,4-dione (8c)**



The title compound was prepared according to **GPIV** using 179 mg (0.75 mmol) of methyl (3-methyl-2-nitrobenzoyl)carbamate. 95 mg (0.495 mmol, 66%) of the product were isolated as colourless solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 11.67 (s, 1H, *N*-H), 10.86 (s, 1H, *O*-H), 7.83 (dd, 1H, *J* = 7.8 Hz, 1.7 Hz, *H*-5), 7.51 (dd, 1H, *J* = 7.3 Hz, 1.7 Hz, *H*-7), 7.13 (dd, 1H, *J* = 7.8 Hz, 7.3 Hz, *H*-6),2.62 (s, 3H, *H*-9),
**$^{13}$C NMR (101 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 161.4, 149.4, 140.2, 139.2, 125.8, 124.3, 122.9, 115.4, 22.6.
**HR-MS:** *m/z* for $C_9H_8N_2O_3$+$H^+$, $[M+H]^+$ calculated: 193.0608; found: 193.0607.

**1*H*,3*H*-6-Chloro-1-hydroxyquinazoline-2,4-dione (8d)**

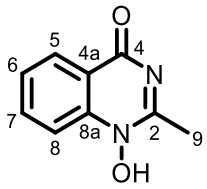


The title compound was prepared according to **GPIV** using 194 mg (0.75 mmol) of methyl (5-chloro-2-nitrobenzoyl)carbamate. 76 mg (0.358 mmol, 48%) of the product were isolated as colourless solid by flash column chromatography (reversed phase silica gel, water:acetonitrile 0% → 100% acetonitrile).

**$^1$H NMR (400 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 11.78 (s, 1H, *N*-H), 11.21 (s, 1H, *O*-H), 7.82 (d, 1H, *J* = 8.8 Hz, *H*-8), 7.78 (dd, 1H, *J* = 8.8 Hz, 1.7 Hz, *H*-7), 7.48 (d, 1H, *J* = 2.5 Hz, 1.7 Hz, *H*-7).
**$^{13}$C NMR (101 MHz, DMSO-$d_6$)** $\delta$ [ppm]: 160.3, 148.0, 140.5, 135.2, 126.8, 126.2, 115.5, 114.8.
**HR-MS:** *m/z* for $C_8H_5{}^{35}ClN_2O_3$-$H^+$, $[M-H]^-$ calculated: 210.9916; found: 210.9914.

## 6. Crystallographic Data

### 1*H*-1-Hydroxy-2-methylquinazolin-4-one (6a)




Crystallization was carried out by dissolving the compound in acetonitrile. Slow evaporating resulted in crystal formation.

| | | |
|---|---|---|
| CCDC Number | | |
| Empirical formular | $C_9H_8N_2O_2$ | |
| Moiety formular | $C_9H_8N_2O_2$ | |
| Formular weight | 176.17 $g \cdot mol^{-1}$ | |
| Temperature | 120(2) K | |
| Wavelength, radiation type | 0.71073 Å, MoKα | |
| Diffractometer | STOE IPDS 2T | |
| Crystal system | Monoclinic | |
| Space group name, number | P $2_1/n$, (14) | |
| Unit cell dimensions | a = 9.1643(7) Å | α = 90° |
| | b = 9.1814(7) Å | β = 115.381(6)° |
| | c = 10.4783(9) Å | γ = 90° |
| Volume | 796.56(12) $Å^3$ | |
| Number of reflections | 3502 | |
| And range used for lattice parameters | 3.09° <=θ<= 28.33° | |
| Z | 4 | |
| Density (calculated) | 1.469 $mg/m^3$ | |
| Absorption coefficient | 0.107 $mm^{-1}$ | |
| Absorption correction | None | |
| F(000) | 368 | |
| Crystal size, colour and form | 0.120·0.120·0.120 $mm^3$, colourless block | |
| Theta range for data collection | 3.091 to 27.845°. | |
| Index ranges | -12<=h<=11, -12<=k<=12, -12<=l<=13 | |
| Number of reflections: | | |
| collected | 4012 | |
| independent | 1883 [R(int) = 0.0409] | |
| observed [I>2sigma(I)] | 1370 | |
| Completeness to theta = 25.2° | 99.9% | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 1883 / 0 / 148 | |
| Goodness-of-fit on $F^2$ | 1.123 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0645, wR2 = 0.1532 | |
| R indices (all data) | R1 = 0.0961, wR2 = 0.1769 | |
| Largest diff. peak and hole | 0.382 and -0.388 $eÅ^{-3}$ | |

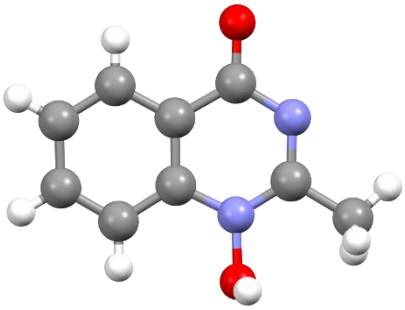


Figure S5: Molecular structure of compound 6a.

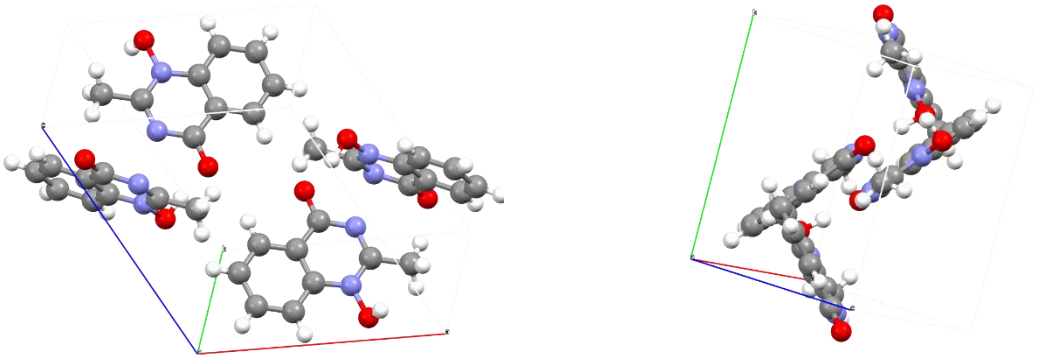
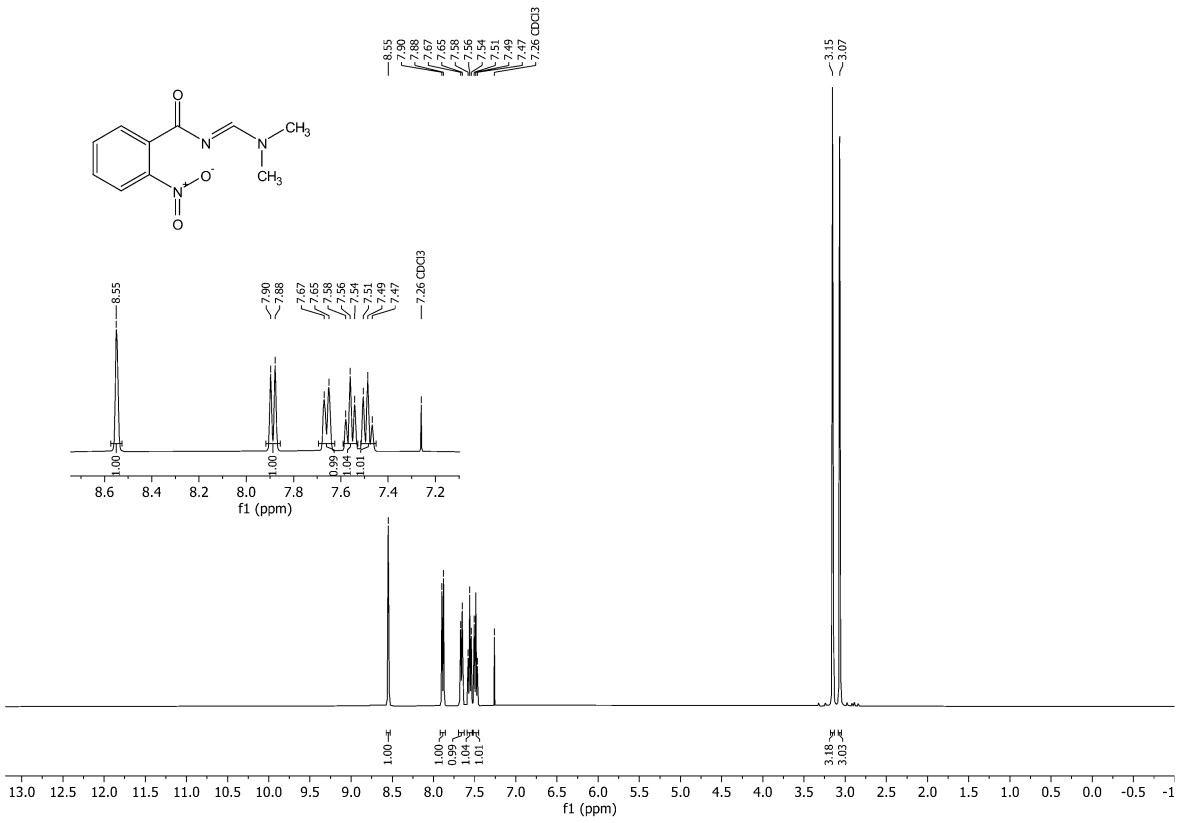
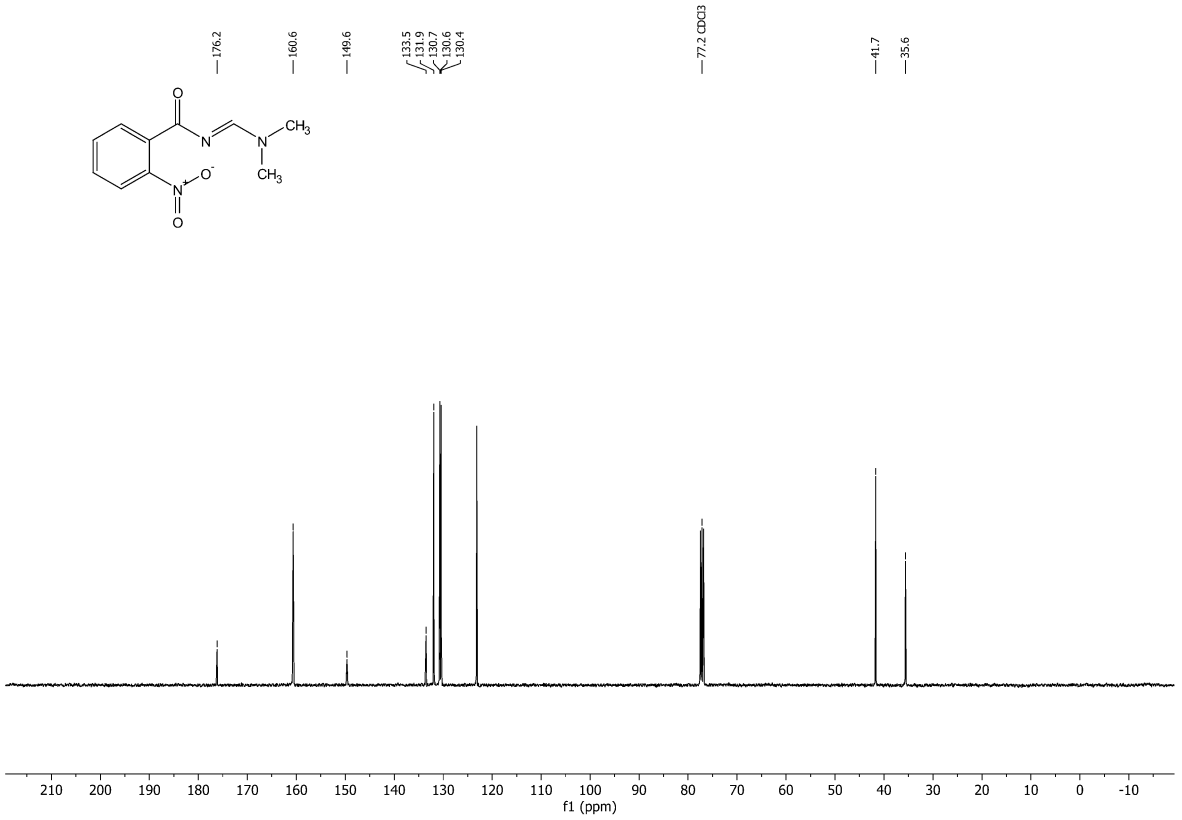


Figure Sb: Packing of compound 6a.

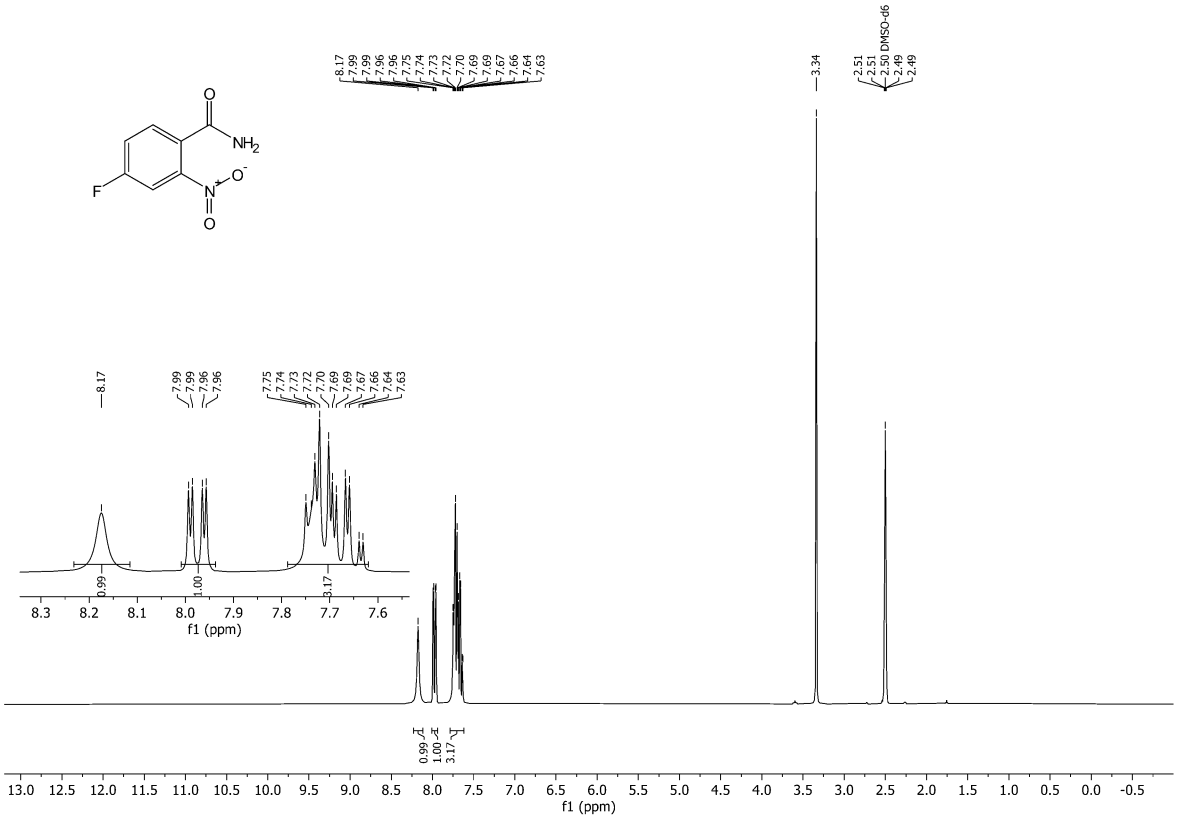
## 7. NMR Spectra

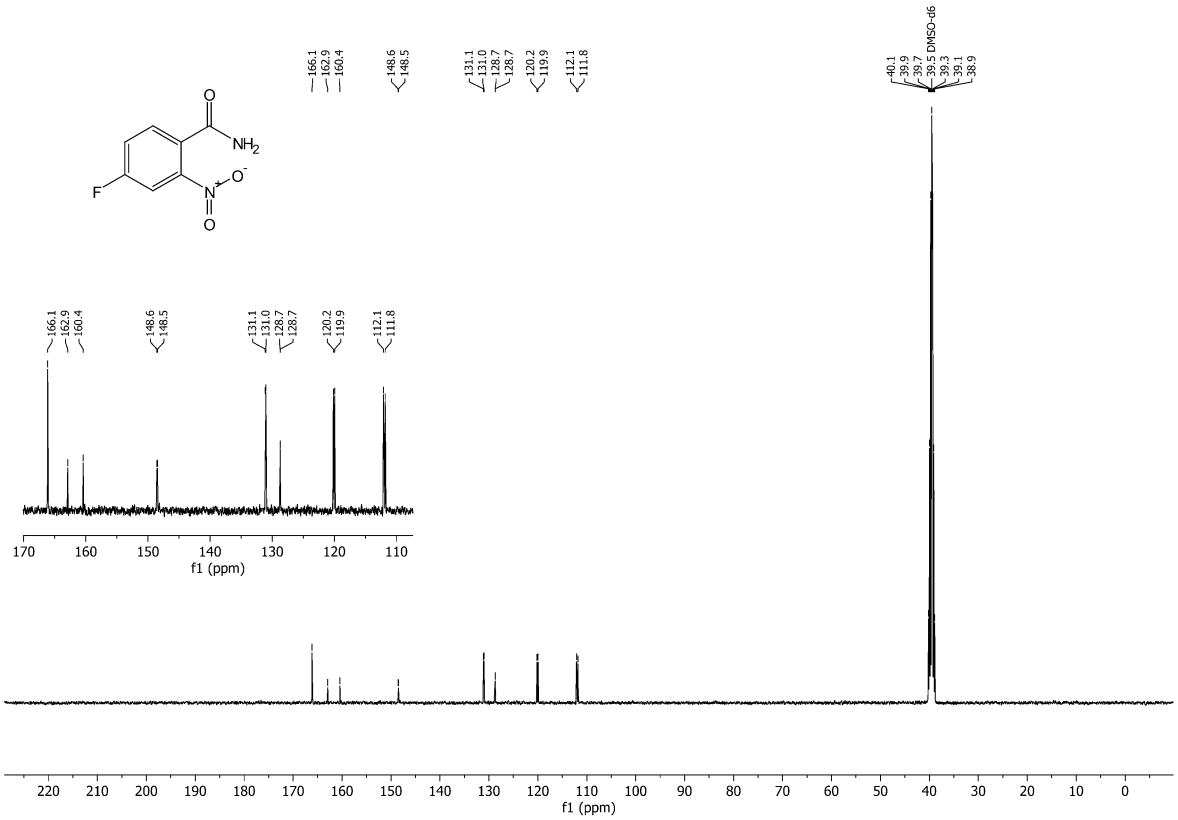
### *N*-(2-(Dimethylamino)vinyl)-2-nitrobenzamide (9)

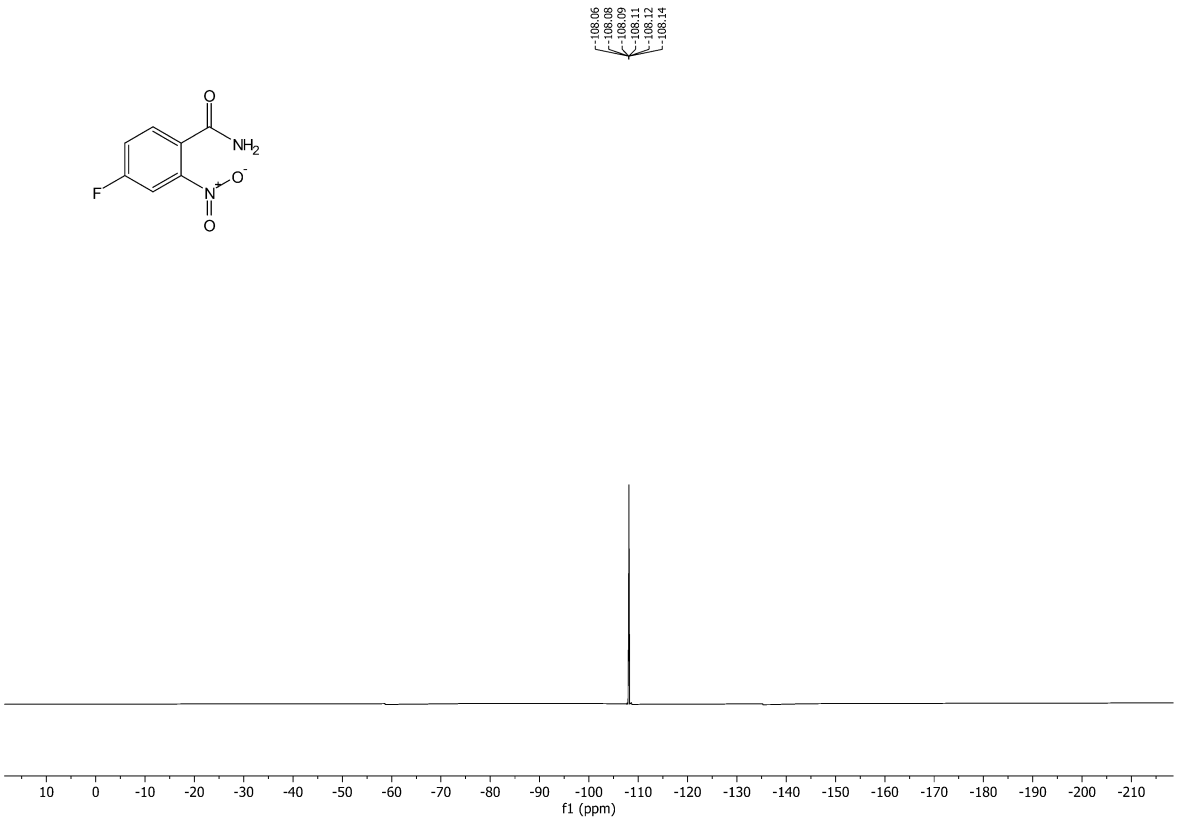


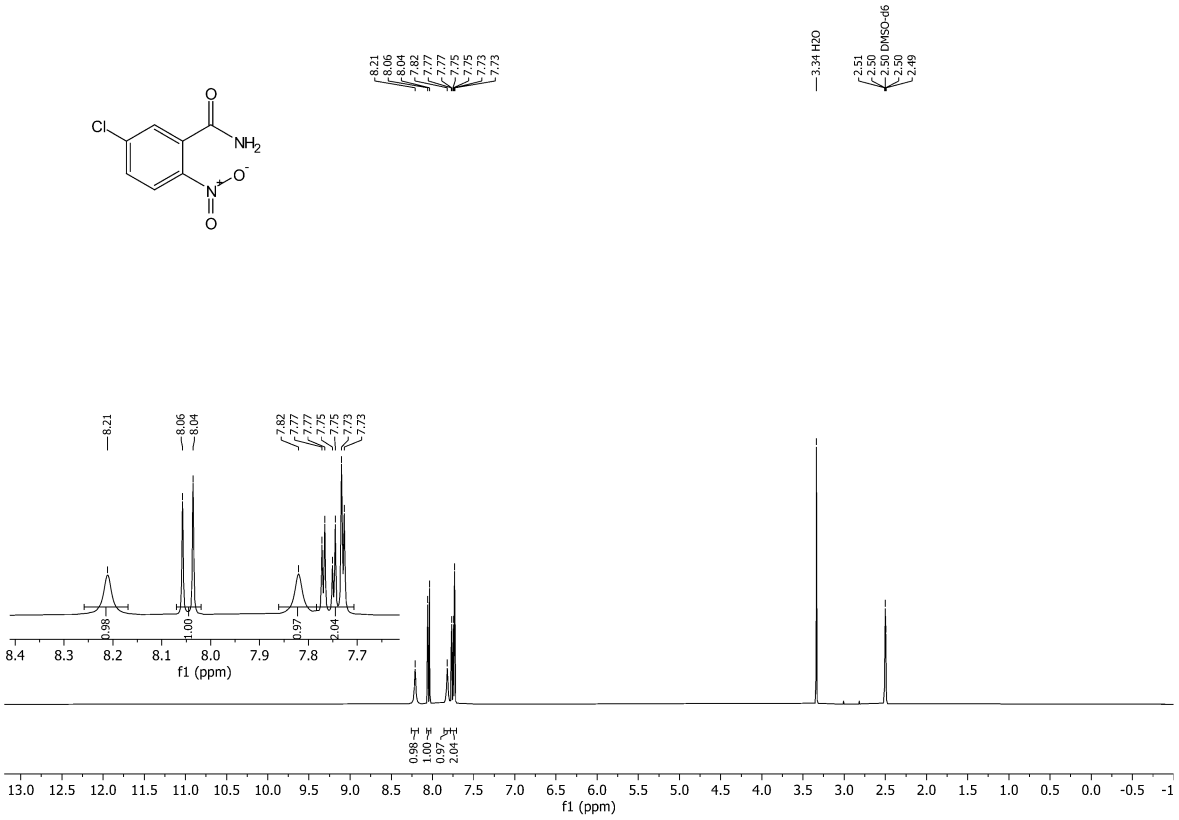
## 4-Fluoro-2-nitrobenzamide (10a)

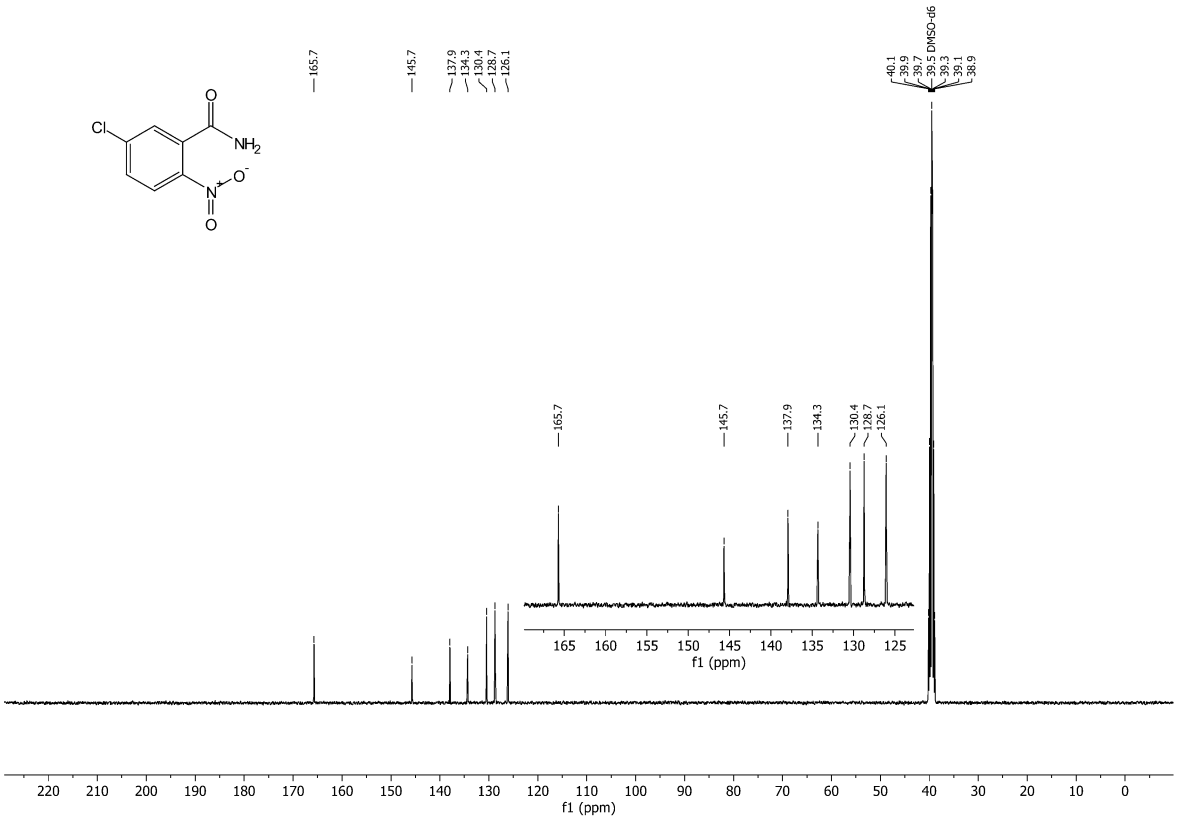


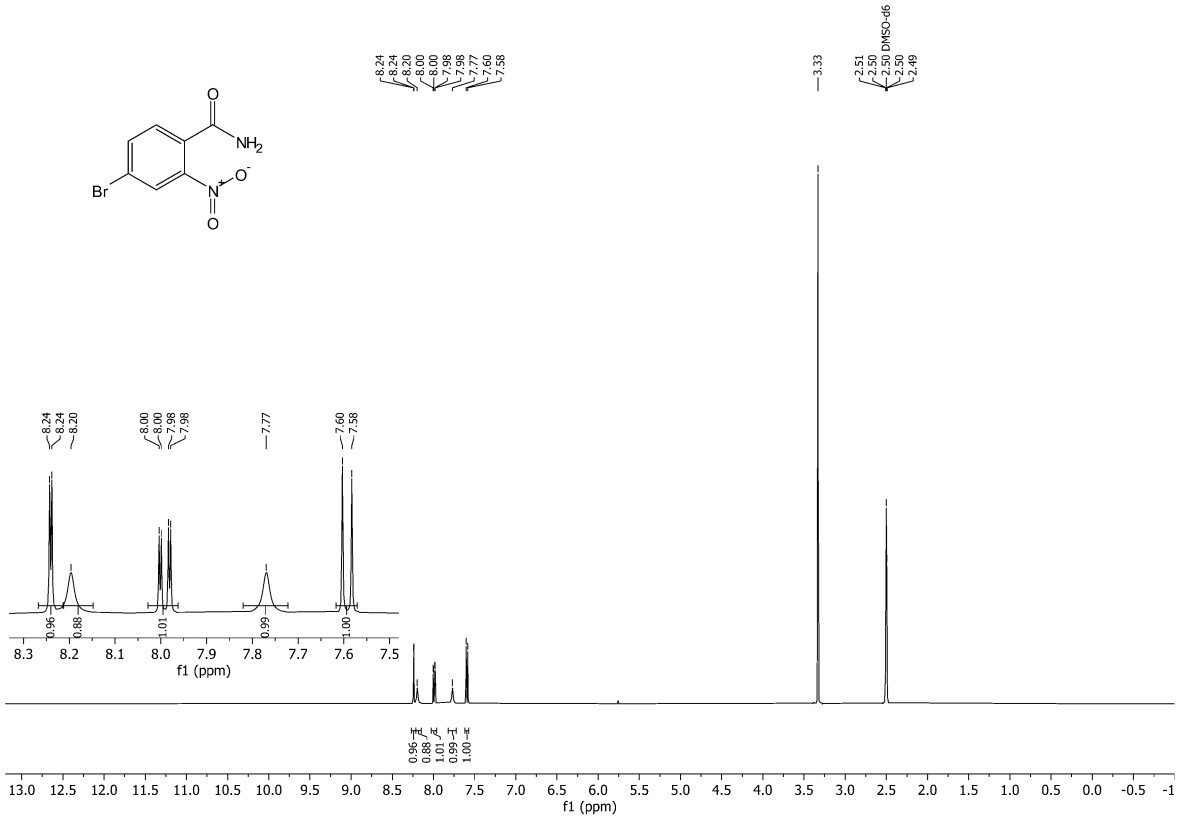

-108.06
-108.08
-108.09
-108.11
-108.12
-108.14
O
NH2
O-
N+
O
F
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

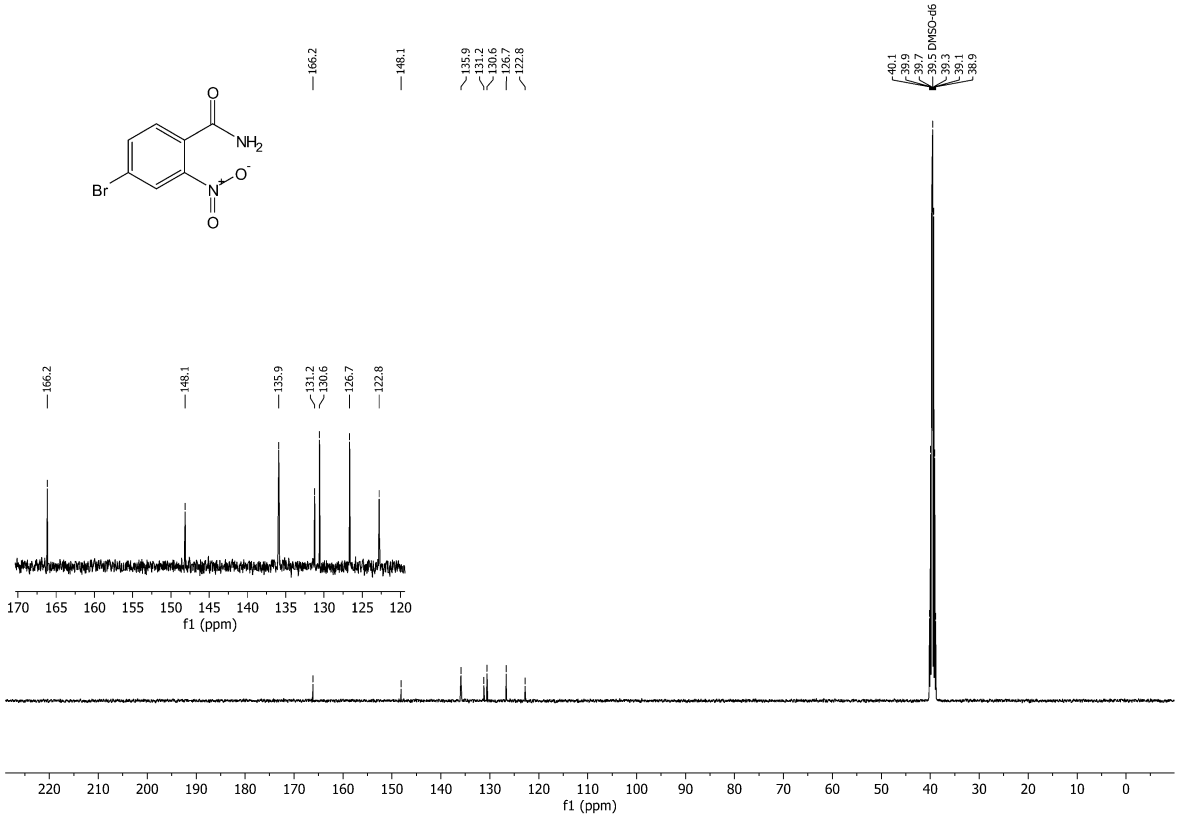
## 5-Chloro-2-nitrobenzamide (10b)



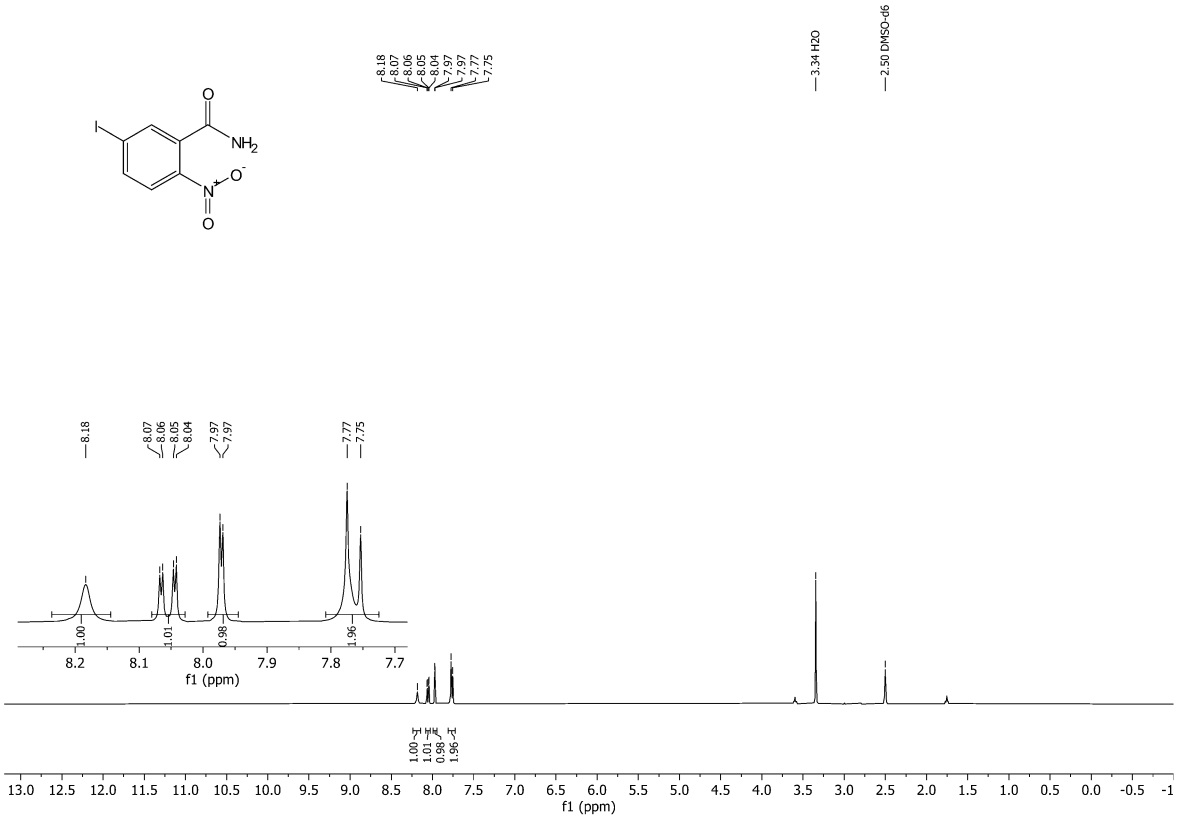



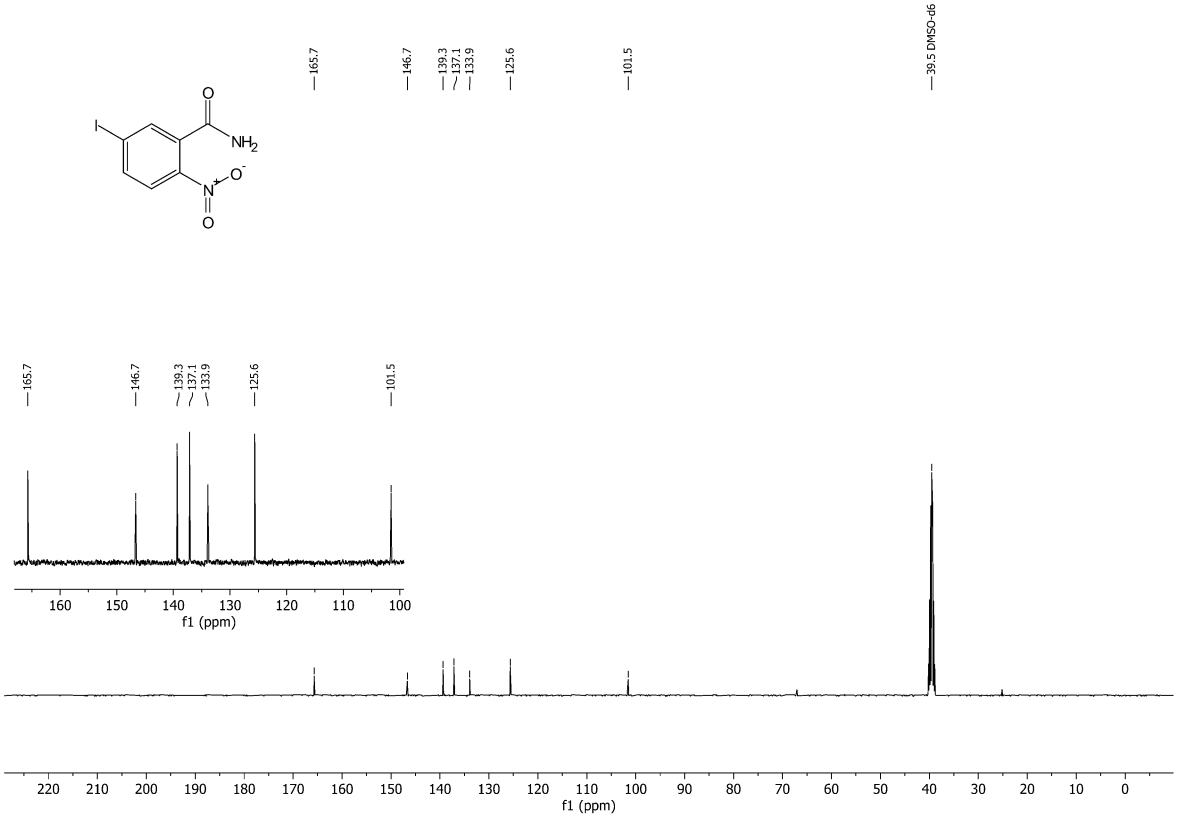
## 4-Bromo-2-nitrobenzamide (10c)



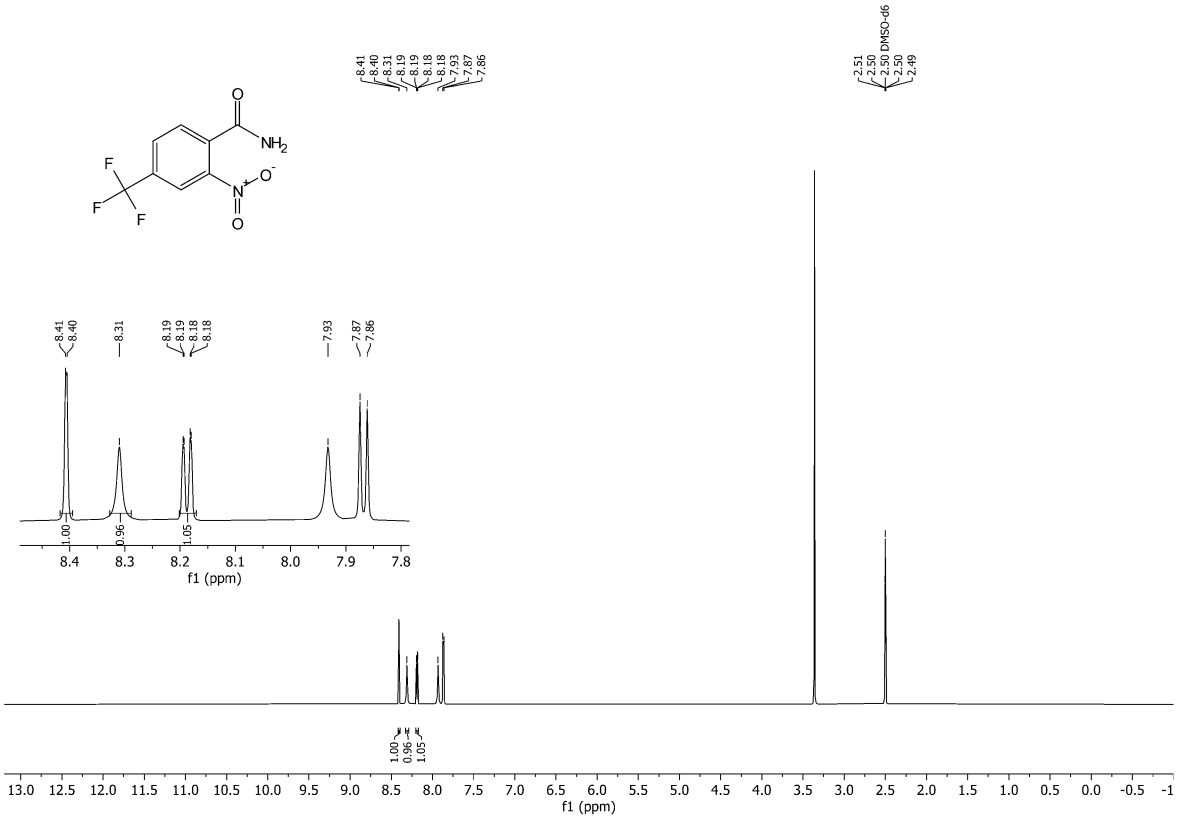



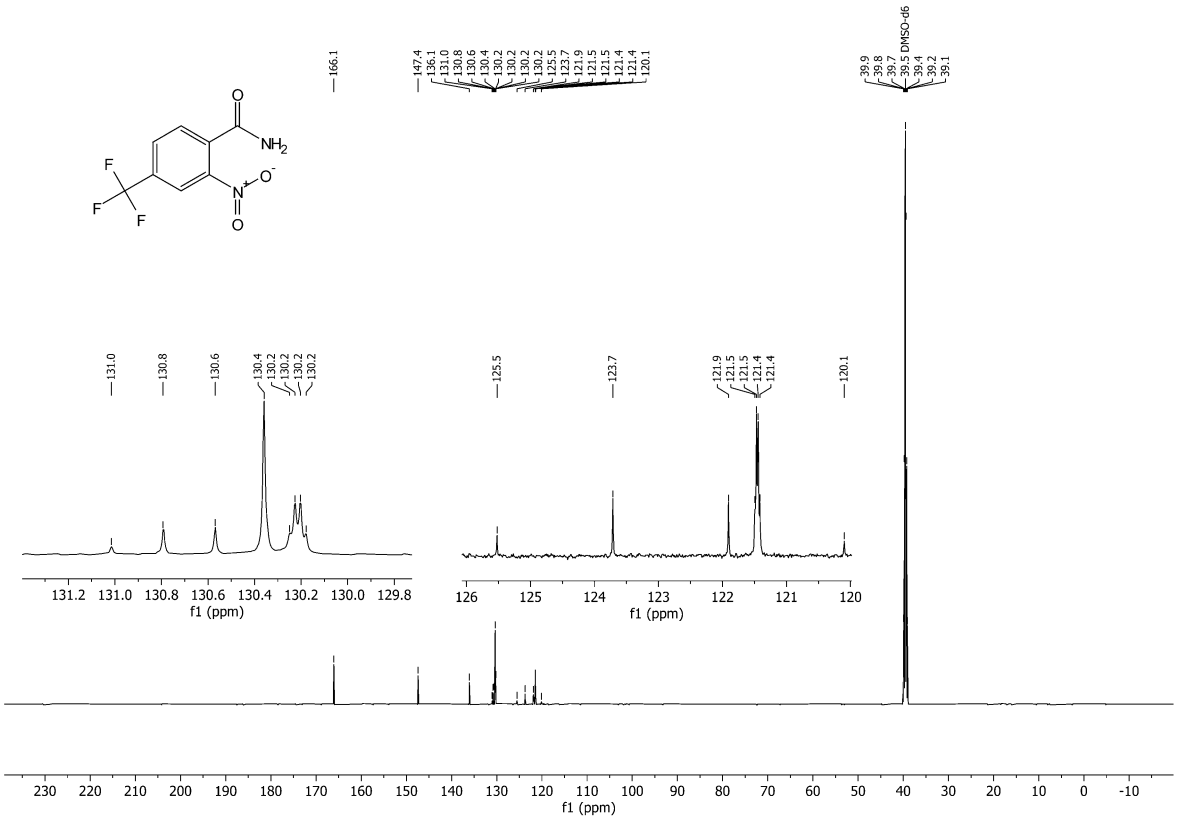
## 5-Iodo-2-nitrobenzamide (10d)

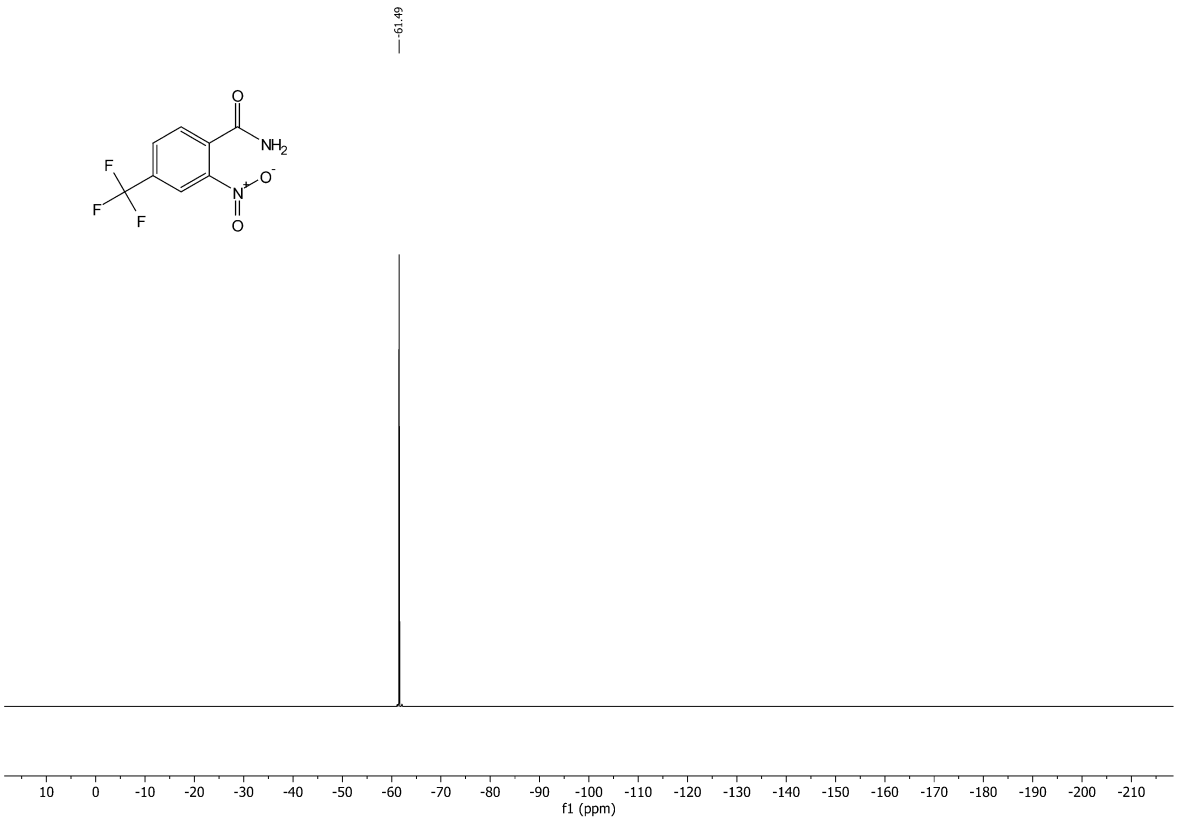


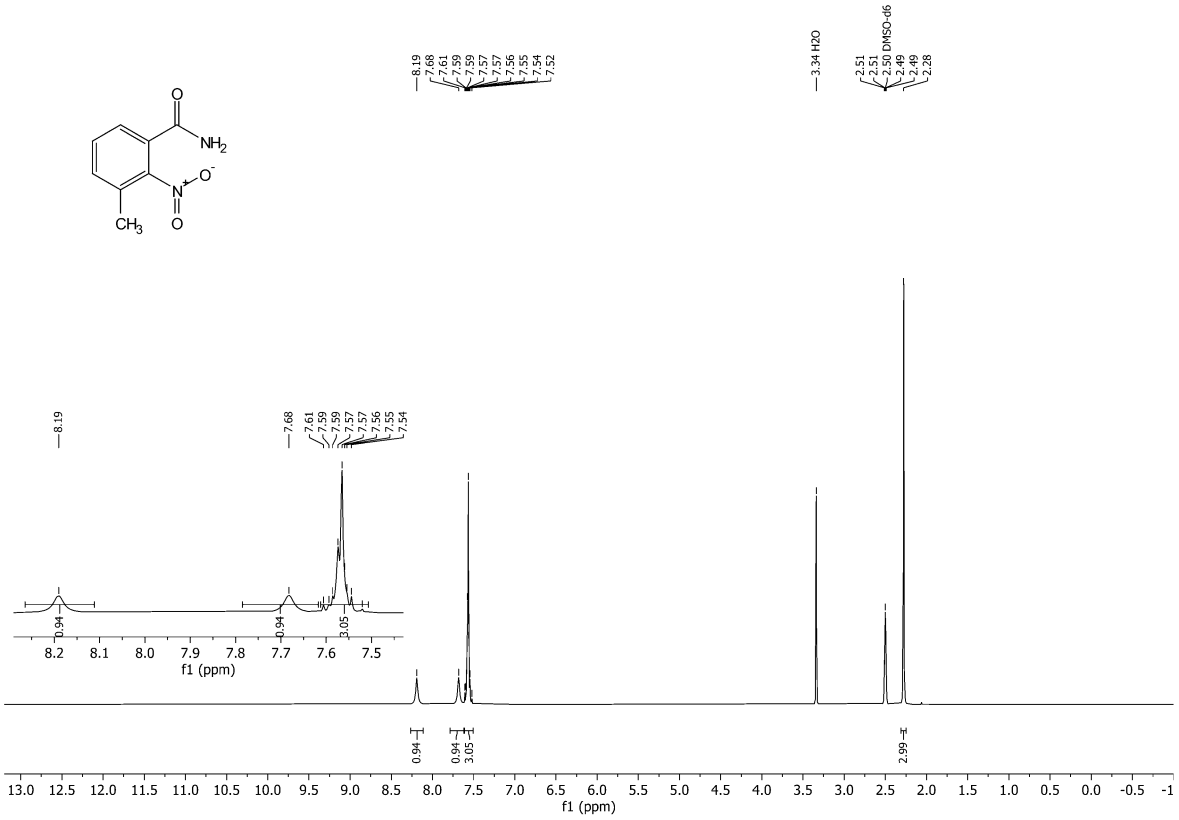
## 2-Nitro-4-(trifluoromethyl)benzamide (10e)

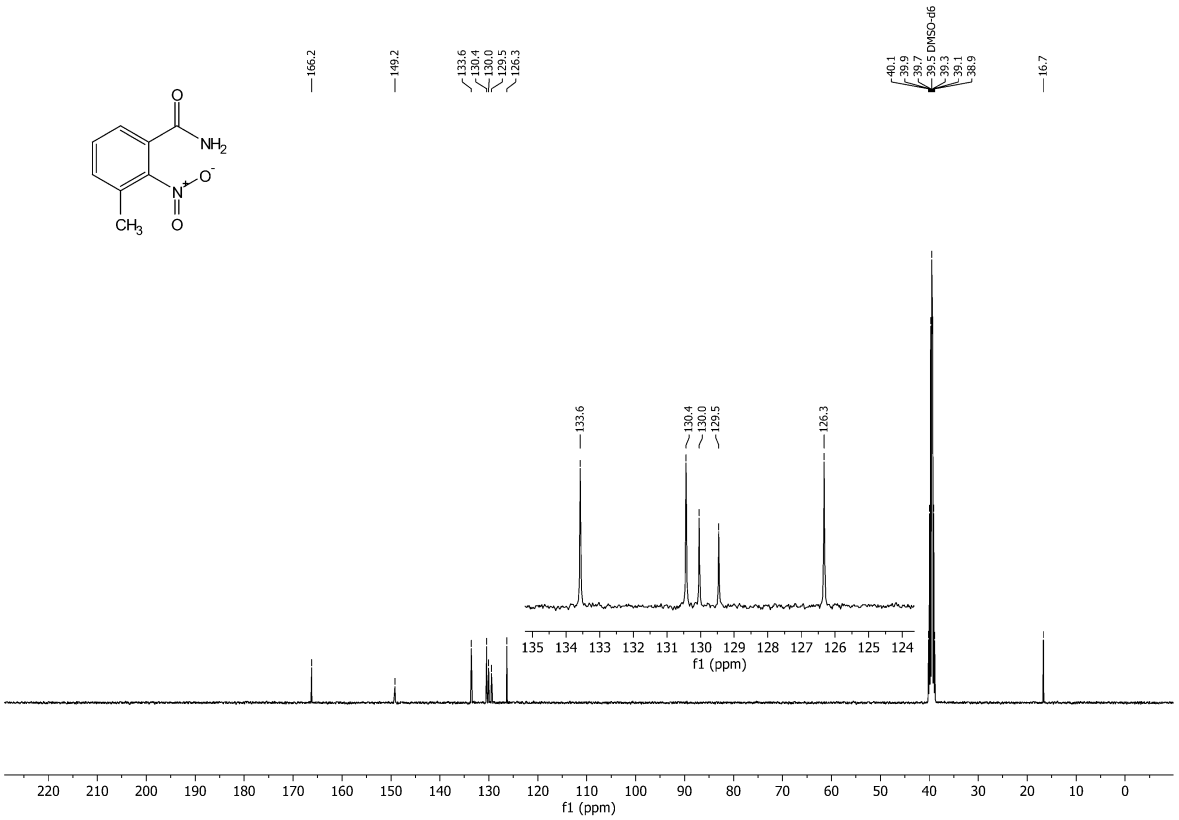


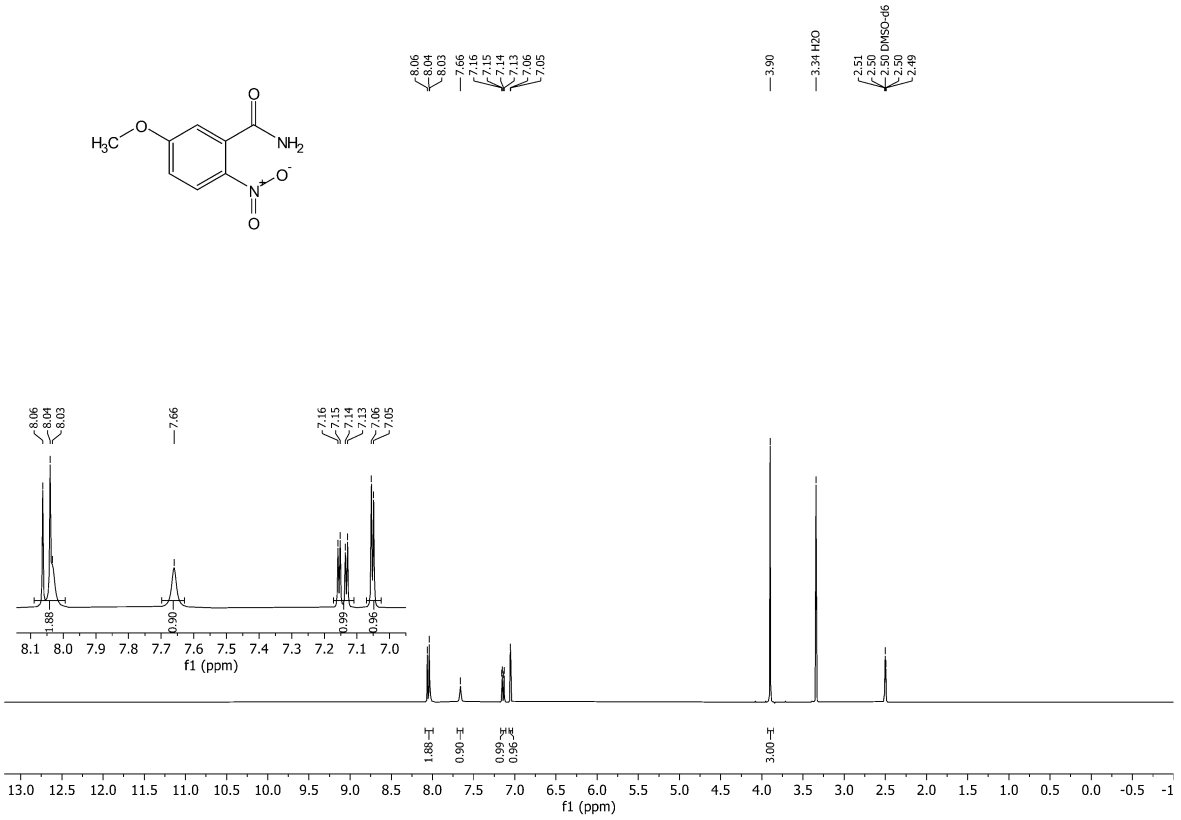

-61.49
O
NH2
F
F
F
N+
O-
O
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

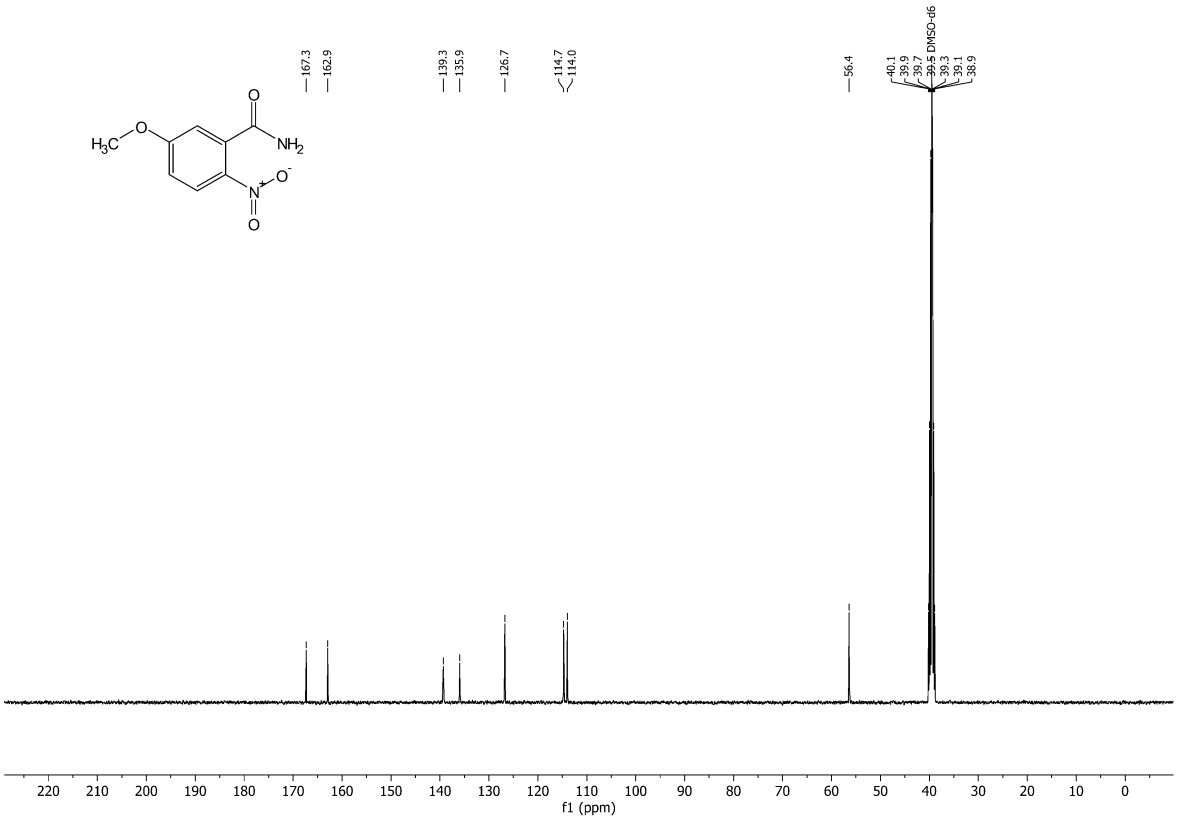
## 3-Methyl-2-nitrobenzamide (10f)



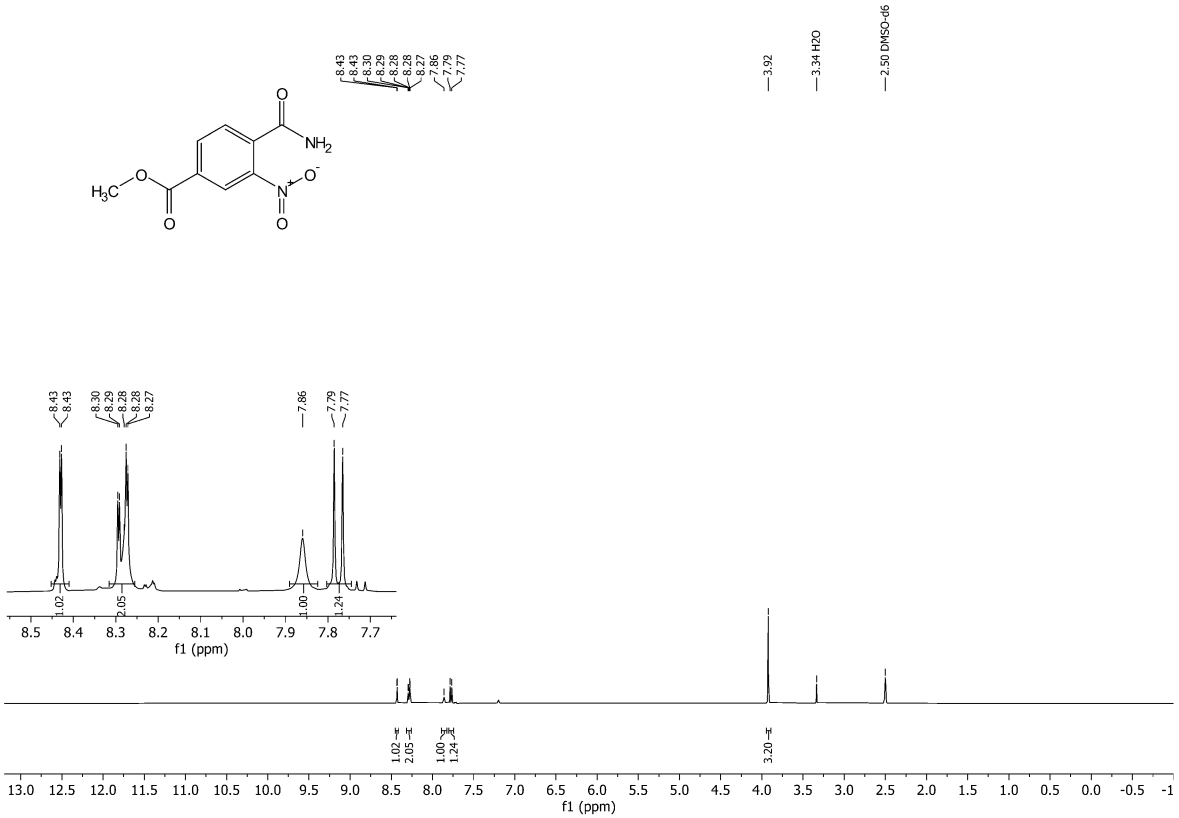



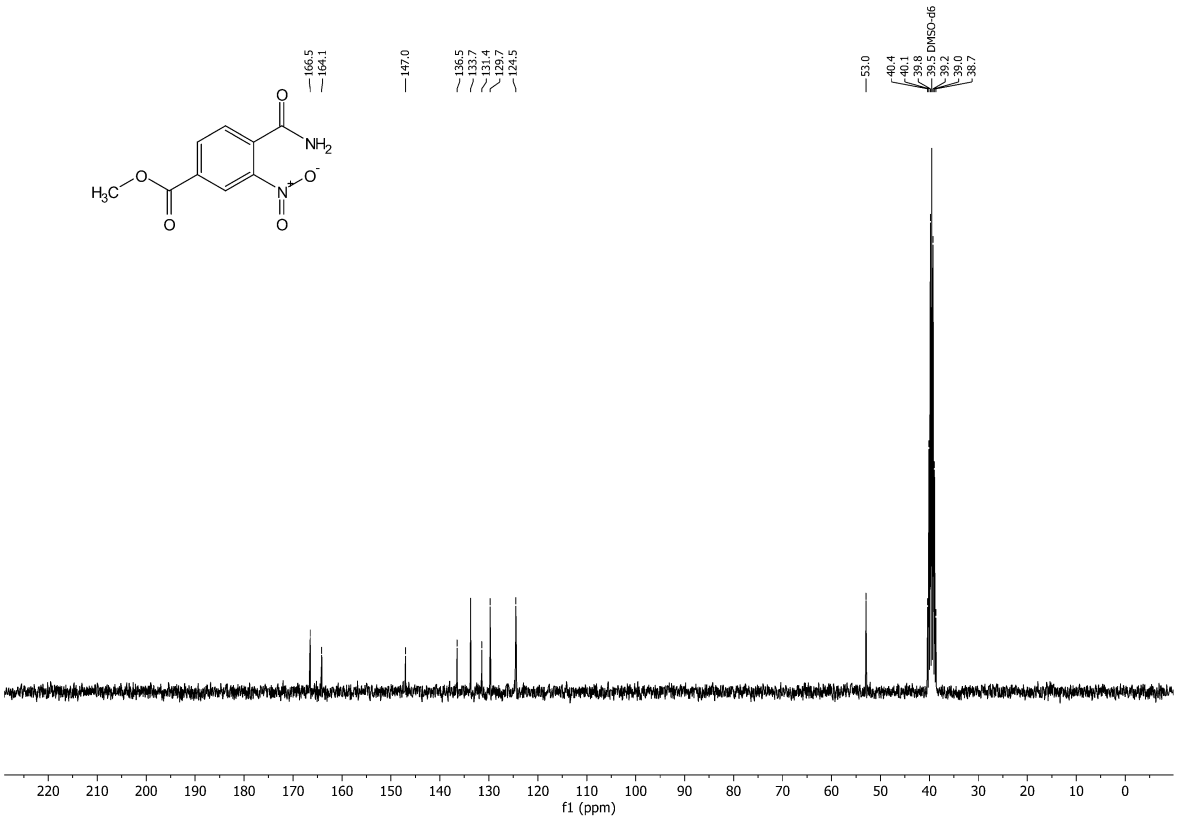
## 5-Methoxy-2-nitrobenzamide (10g)



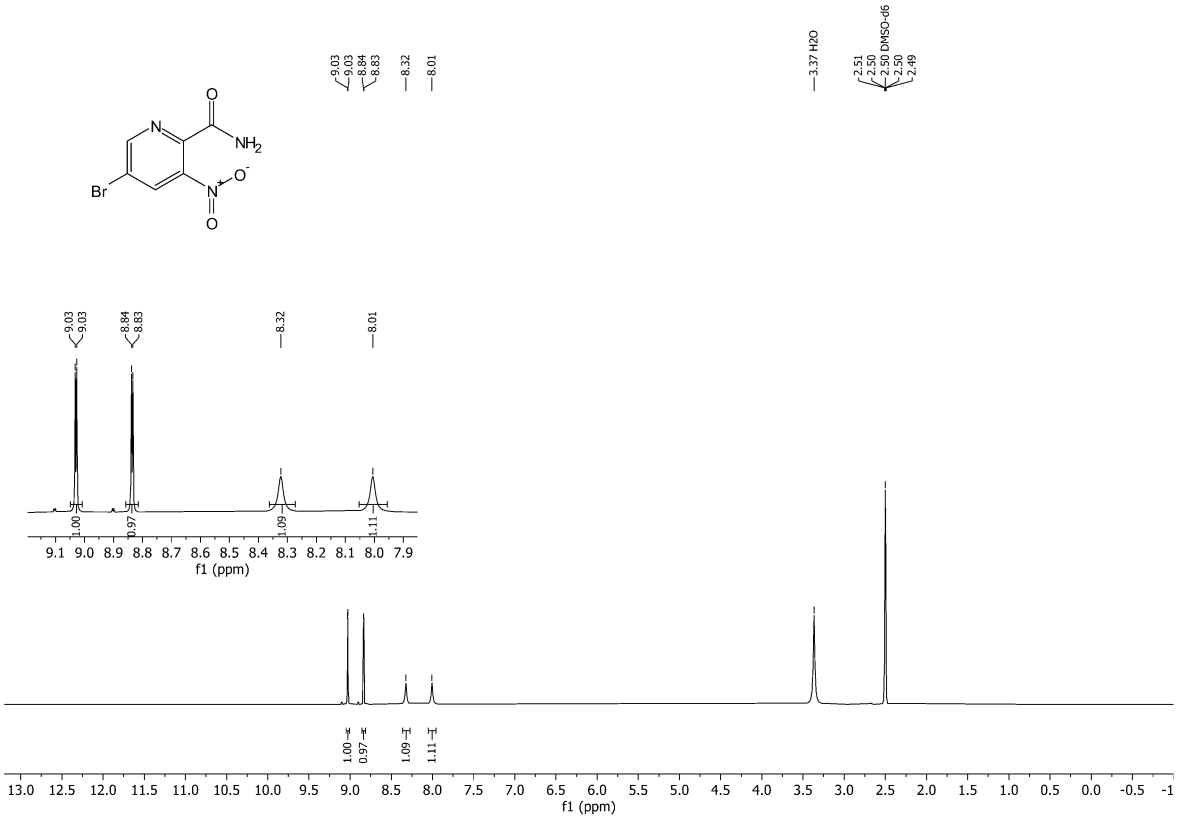



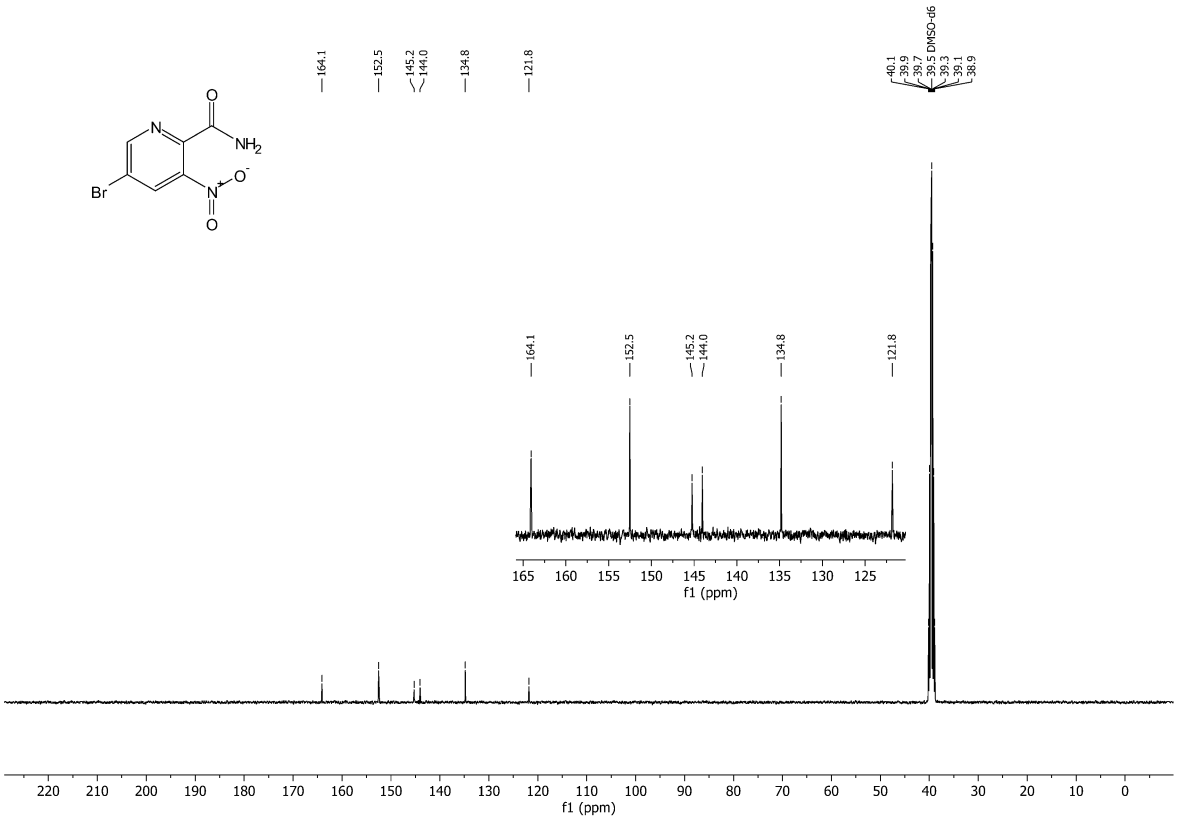
## Methyl 4-carbamoyl-3-nitrobenzoate (10h)



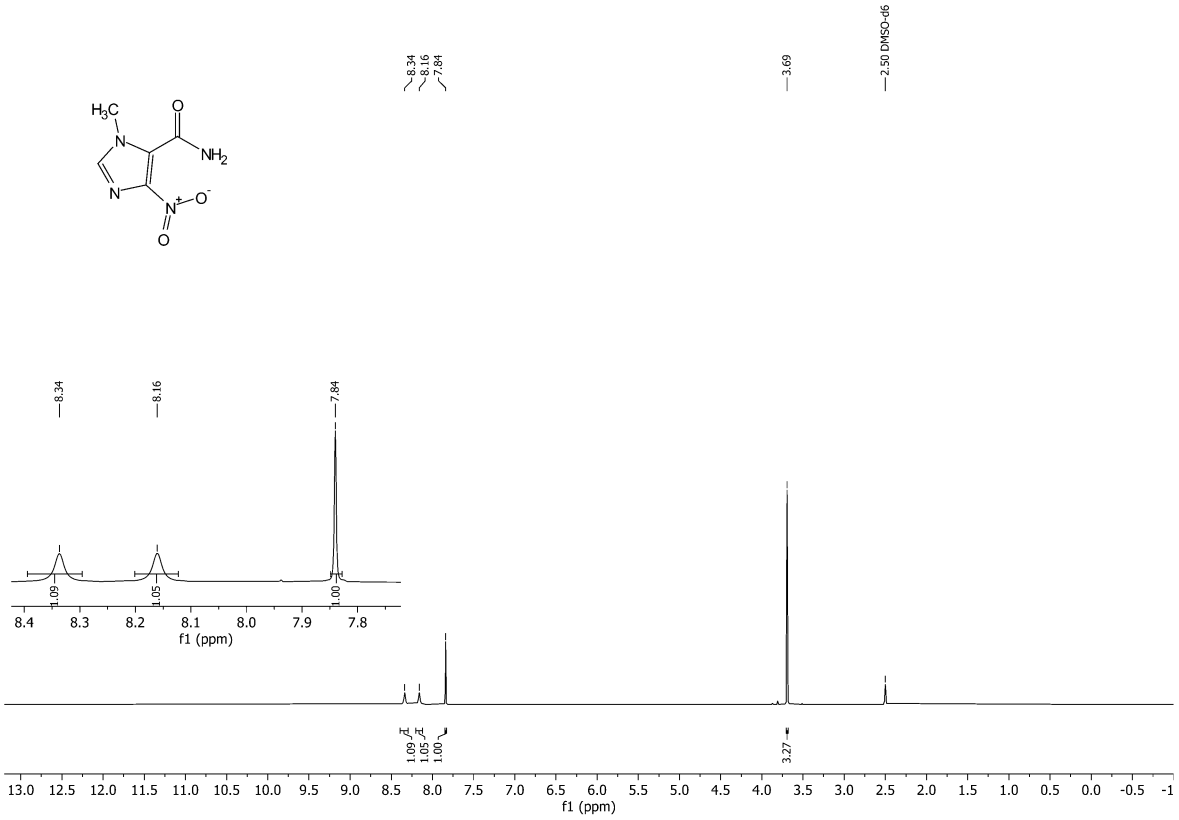



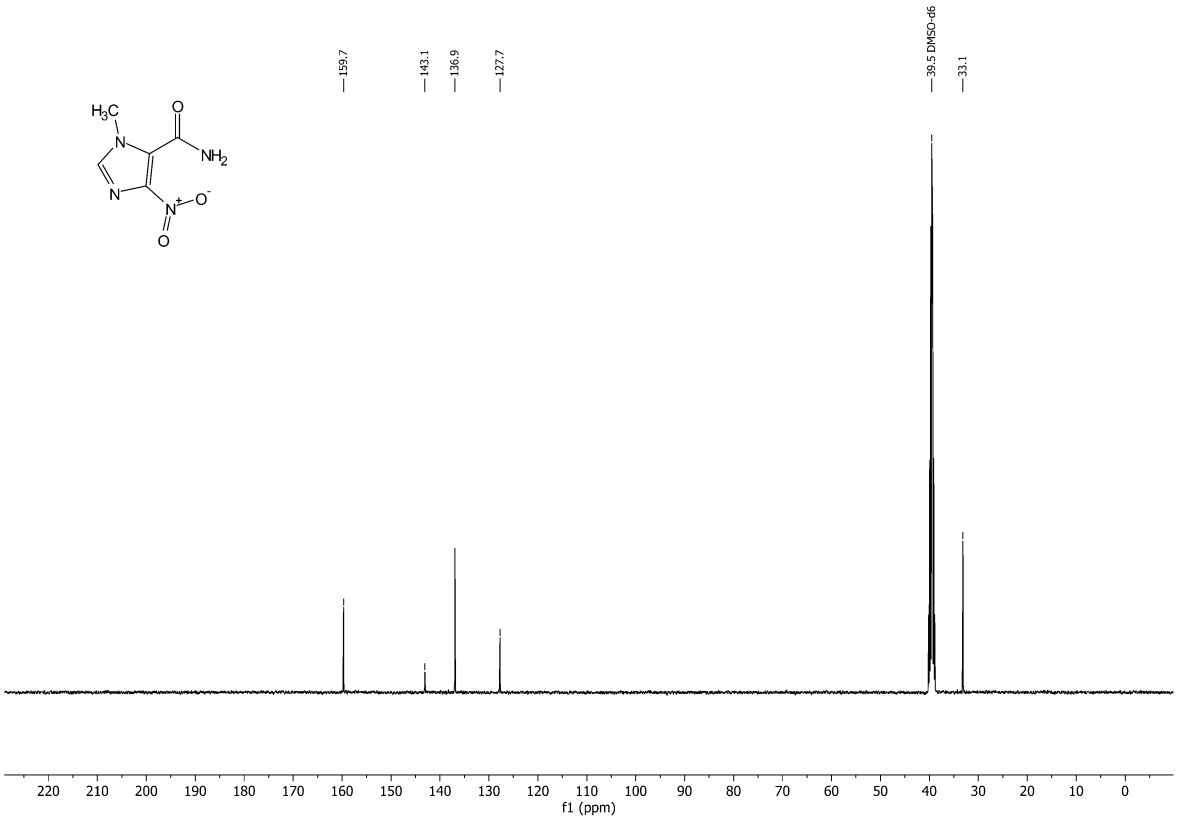
## 5-Bromo-3-nitropicolinamide (10i)



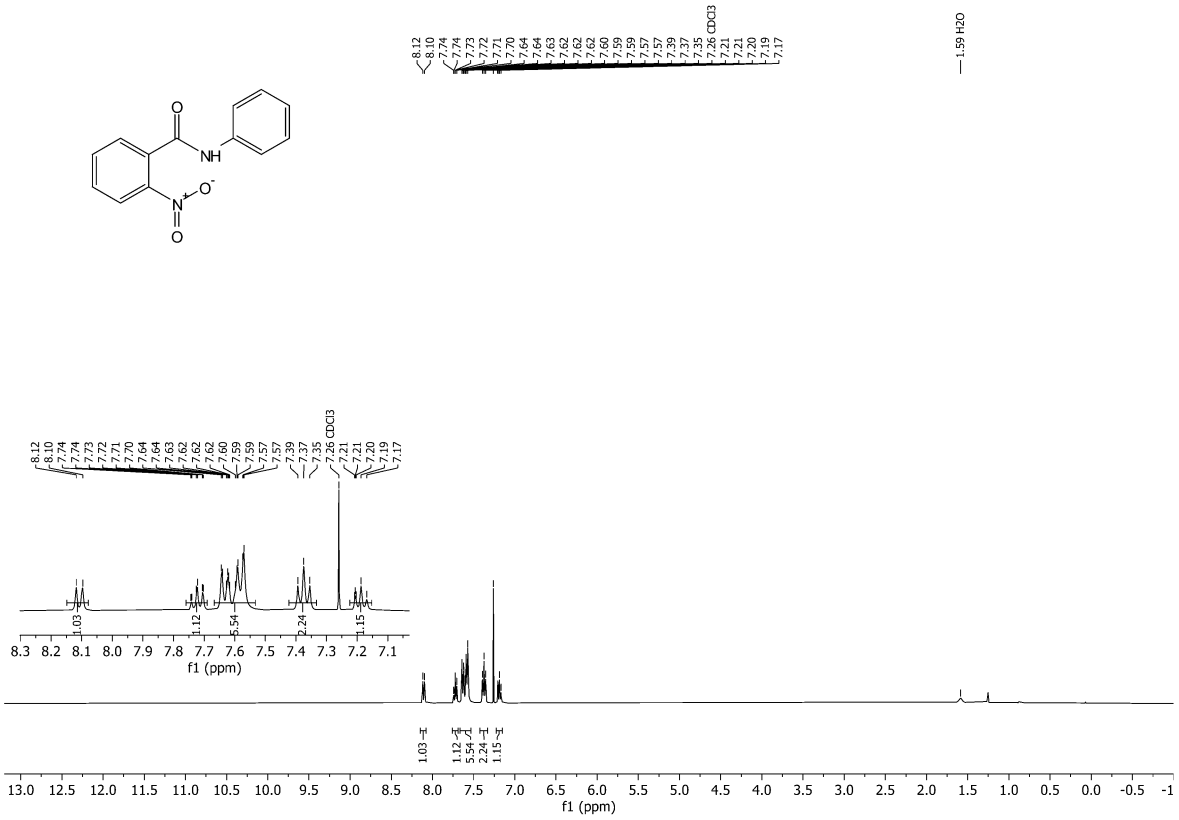



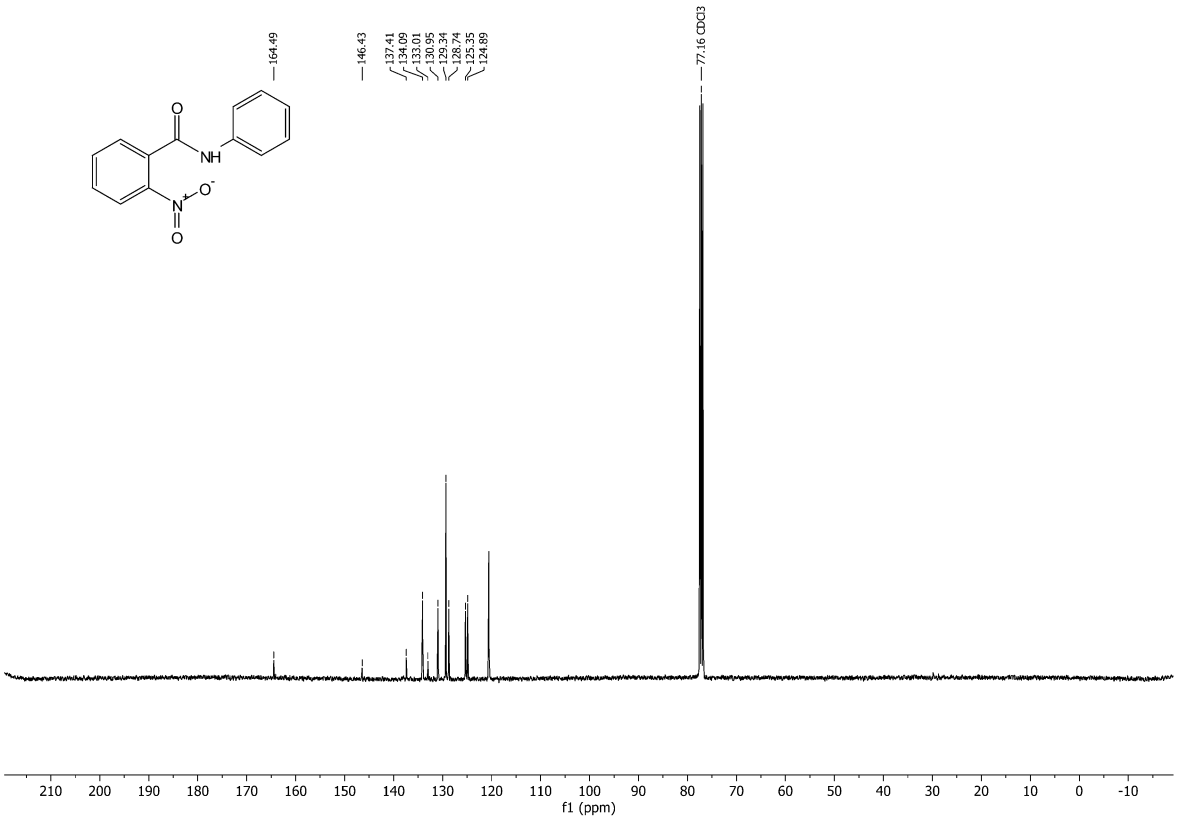
## 1*H*-1-Methyl-4-nitro-imidazole-5-carboxamide (10j)



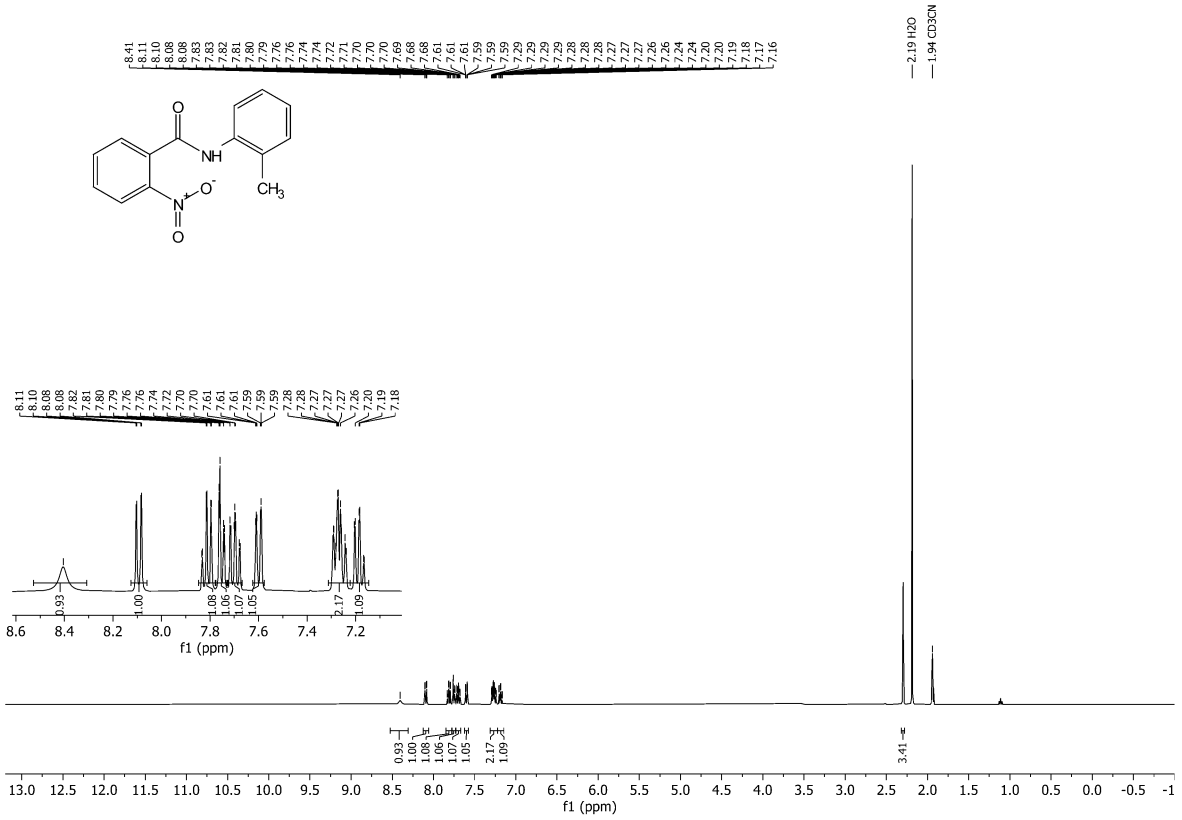



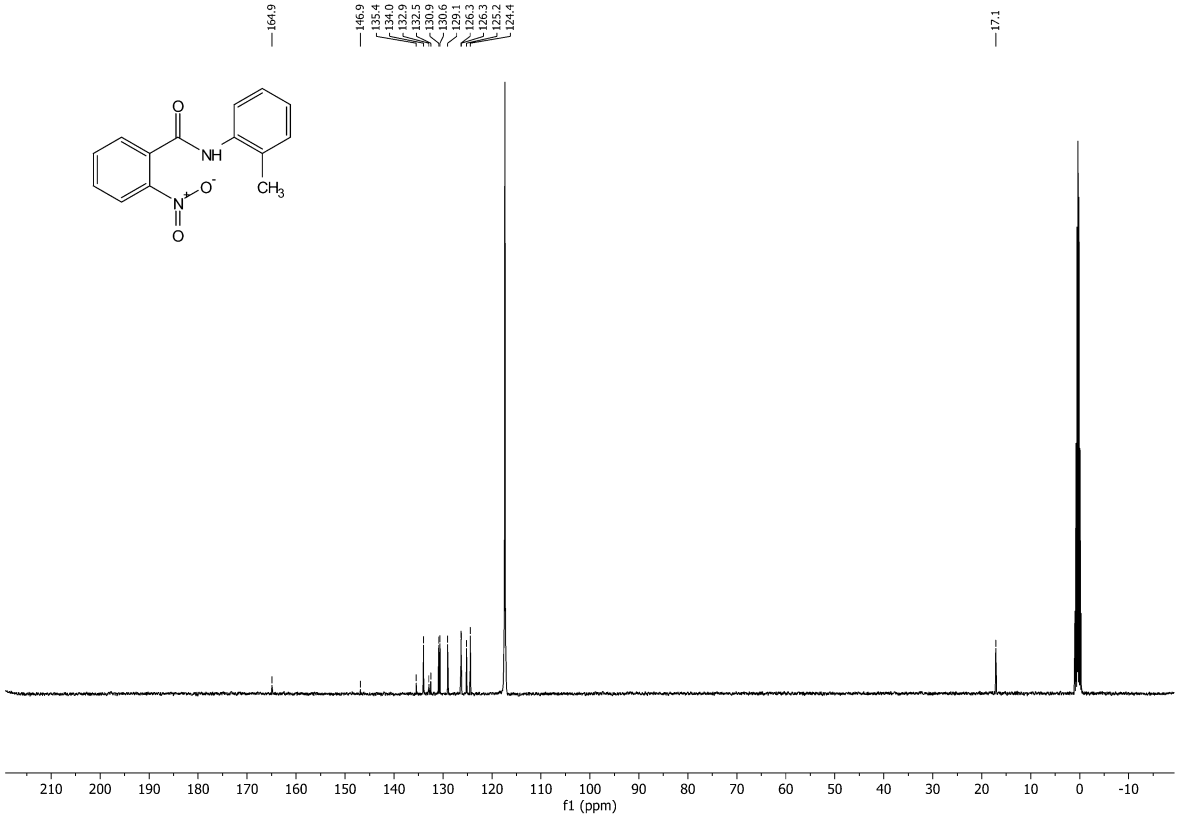
## 2-Nitro-*N*-phenylbenzamide (11a)



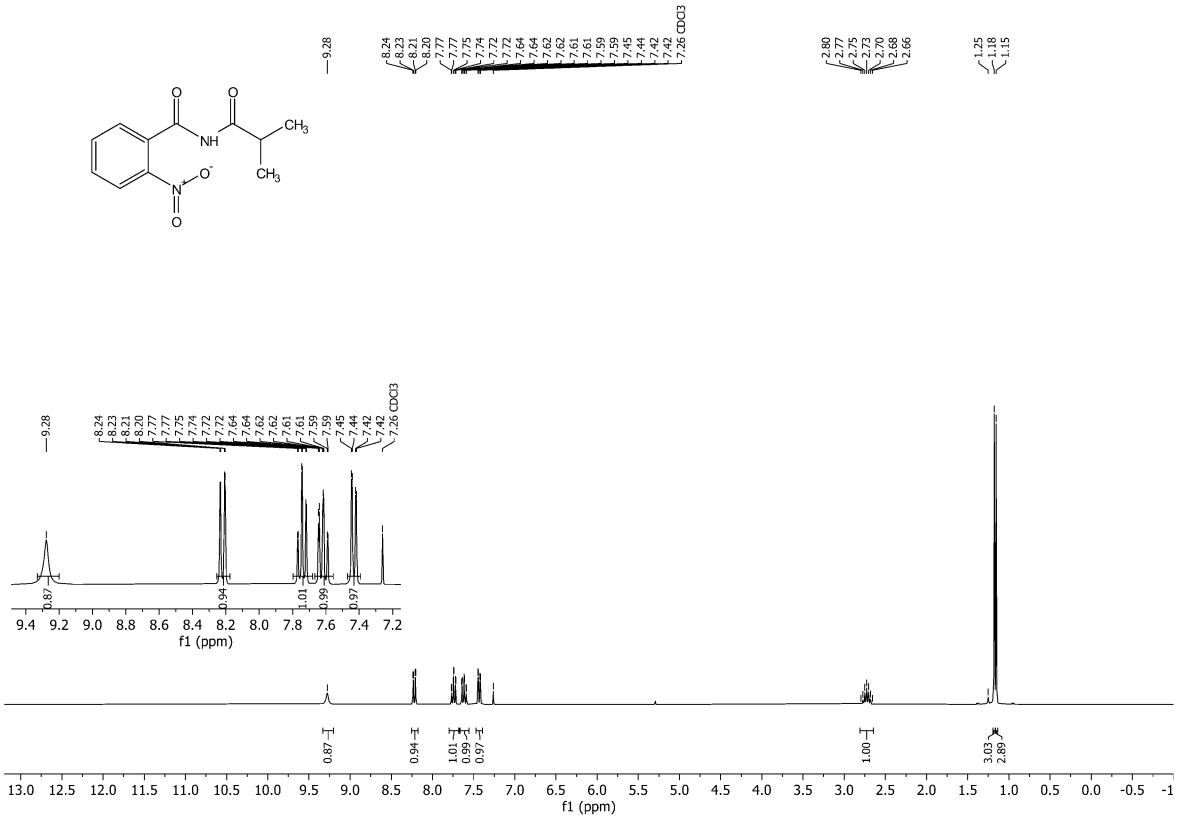
## 2-Nitro-*N*-(o-tolyl)benzamide (11b)

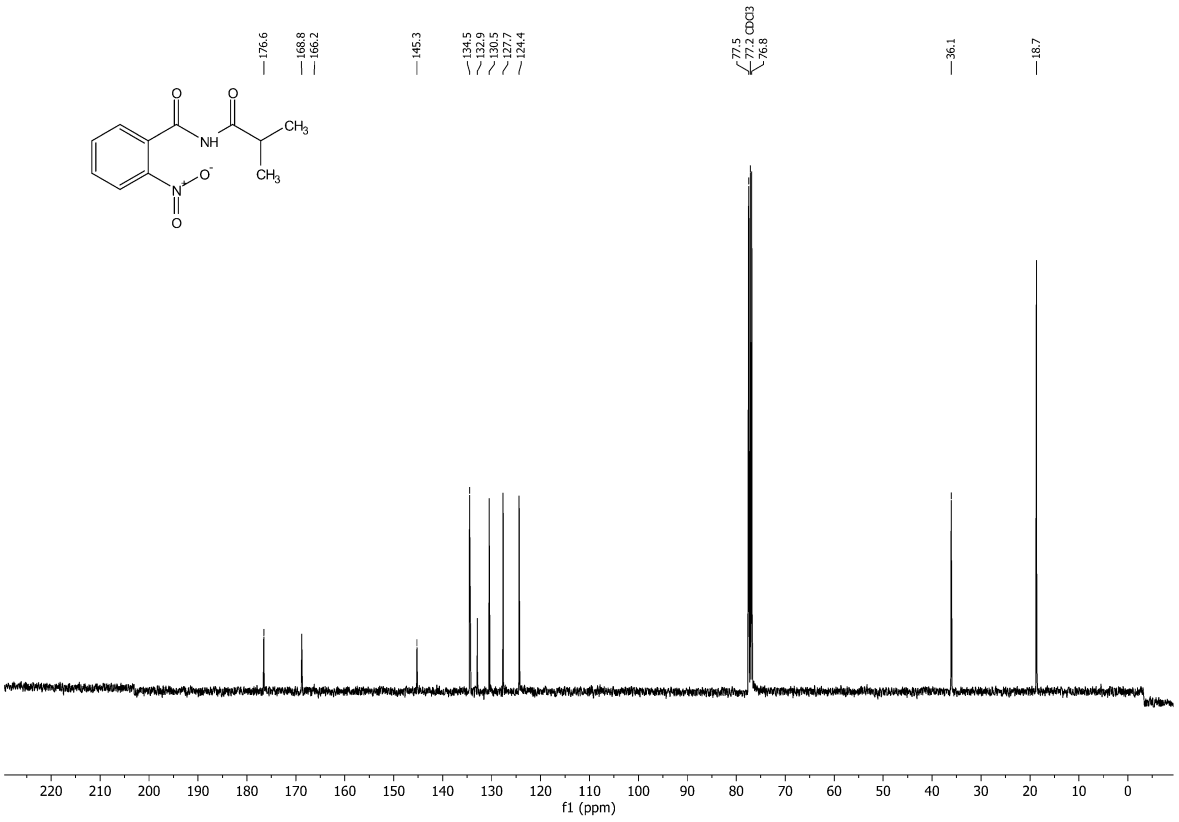


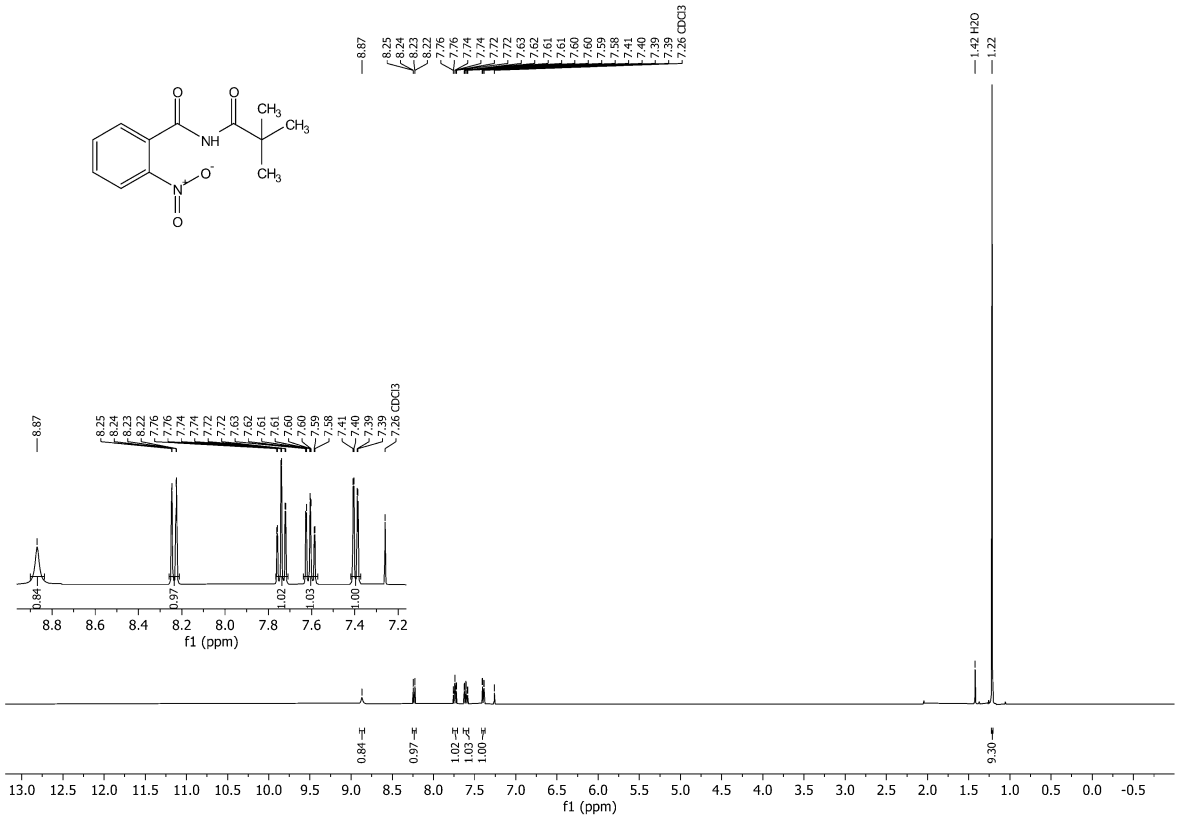
## *N*-Acetyl-2-nitrobenzamide (5a)

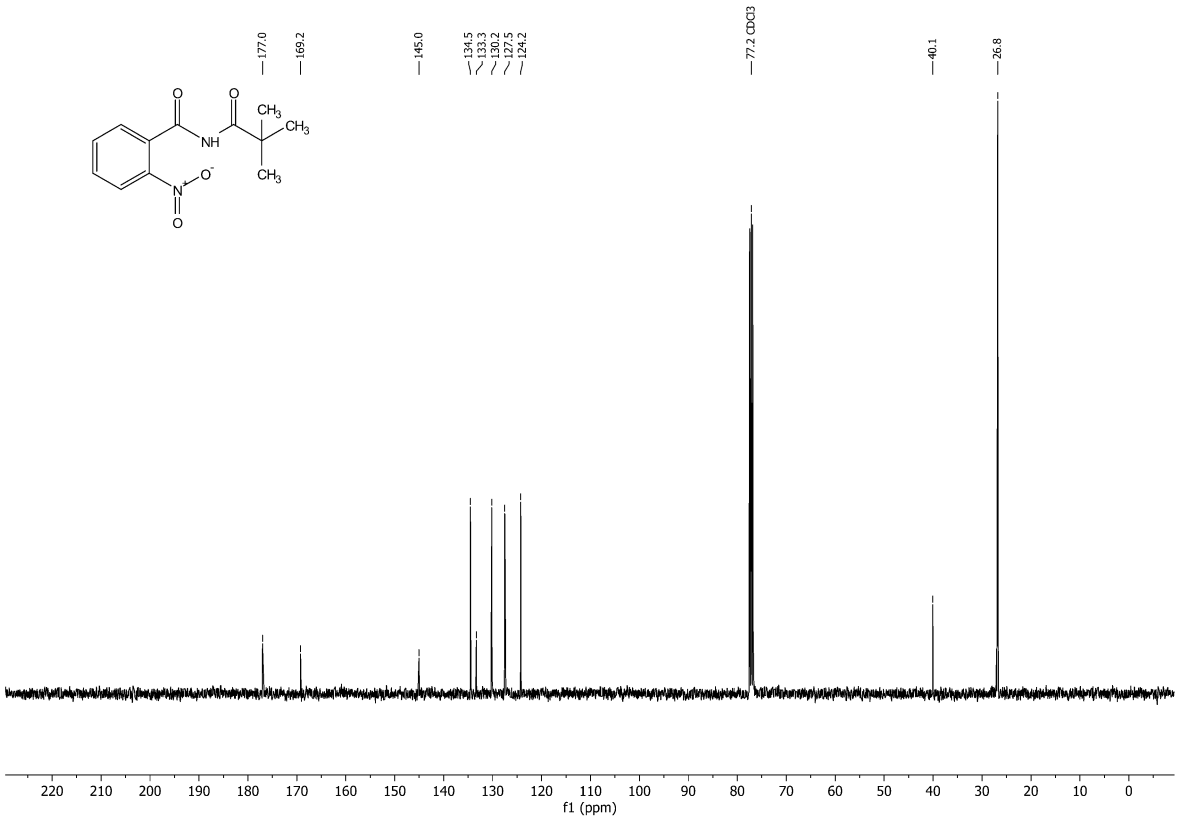
## *N*-Isobutyryl-2-nitrobenzamide (5b)



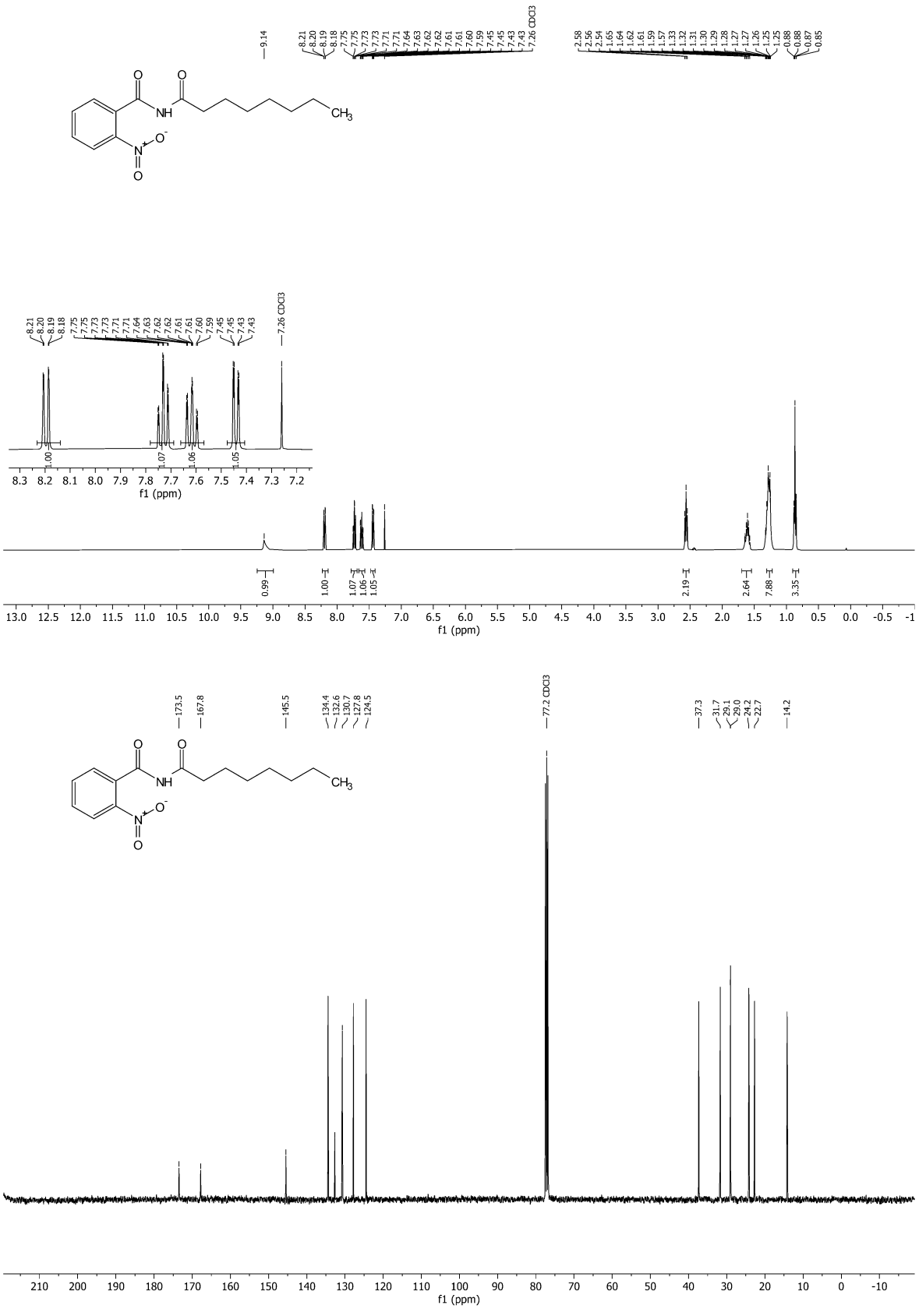



## 2-Nitro-*N*-pivaloylbenzamide (5c)

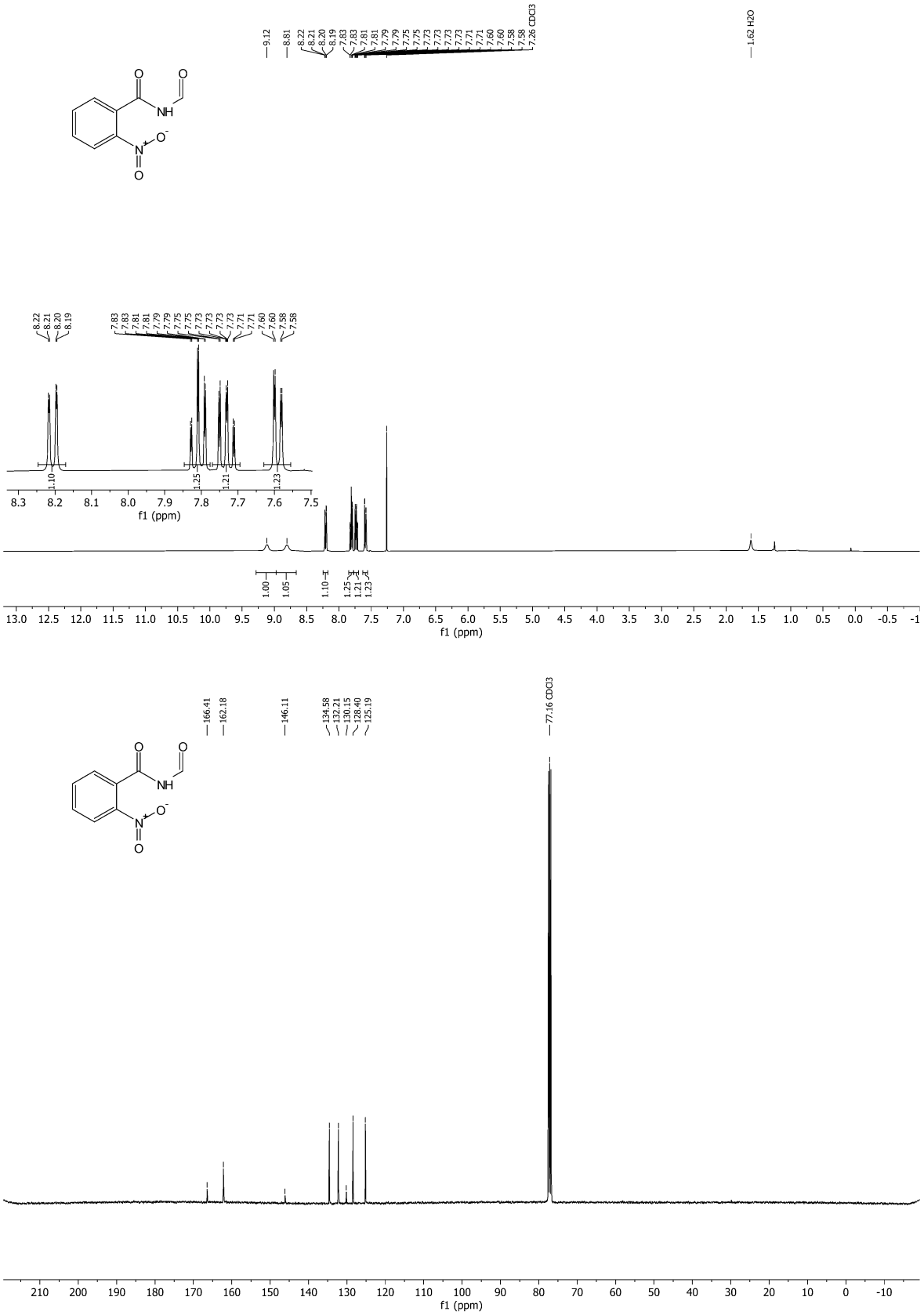


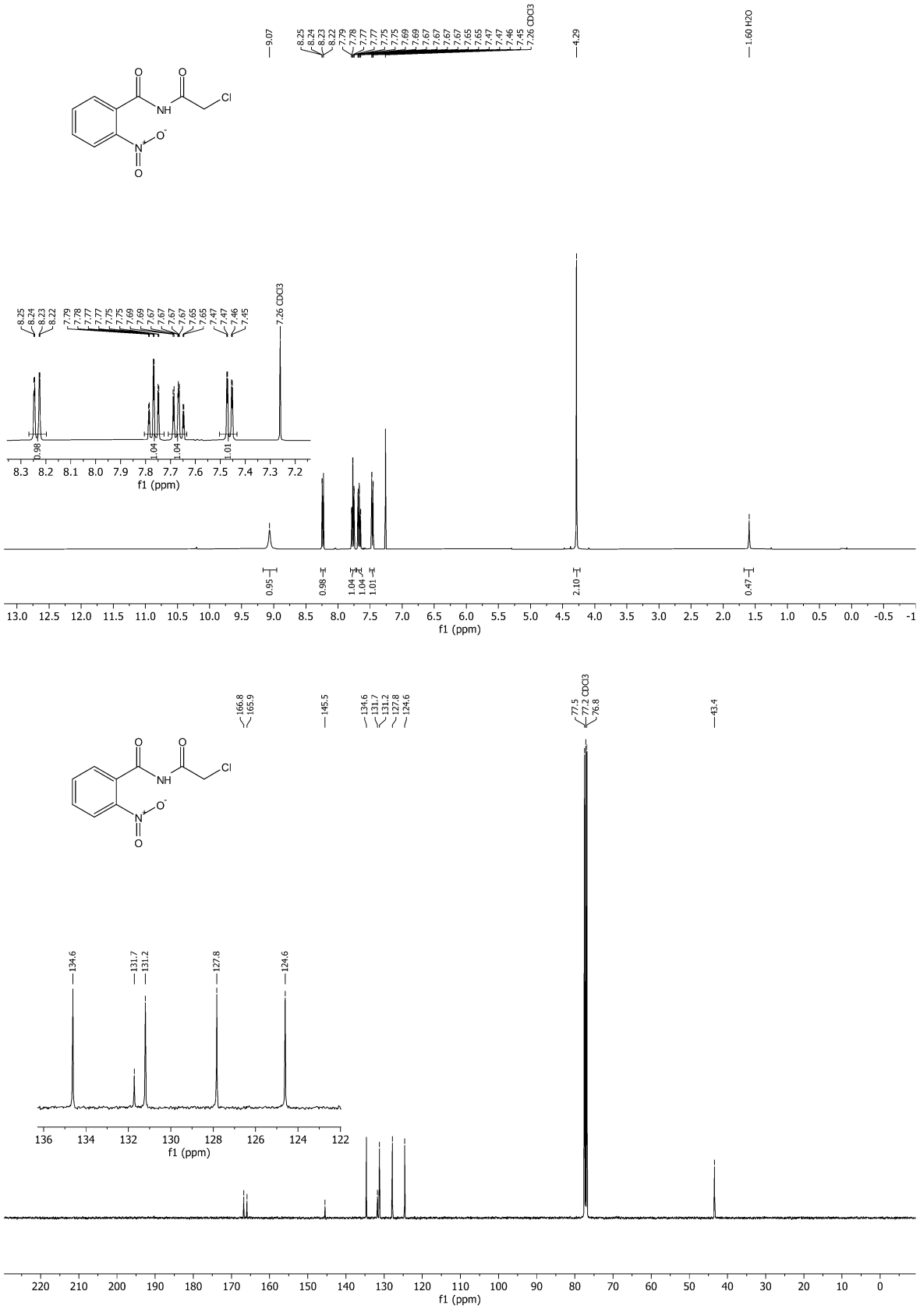
## 2-Nitro-*N*-octanoylbenzamide (5d)



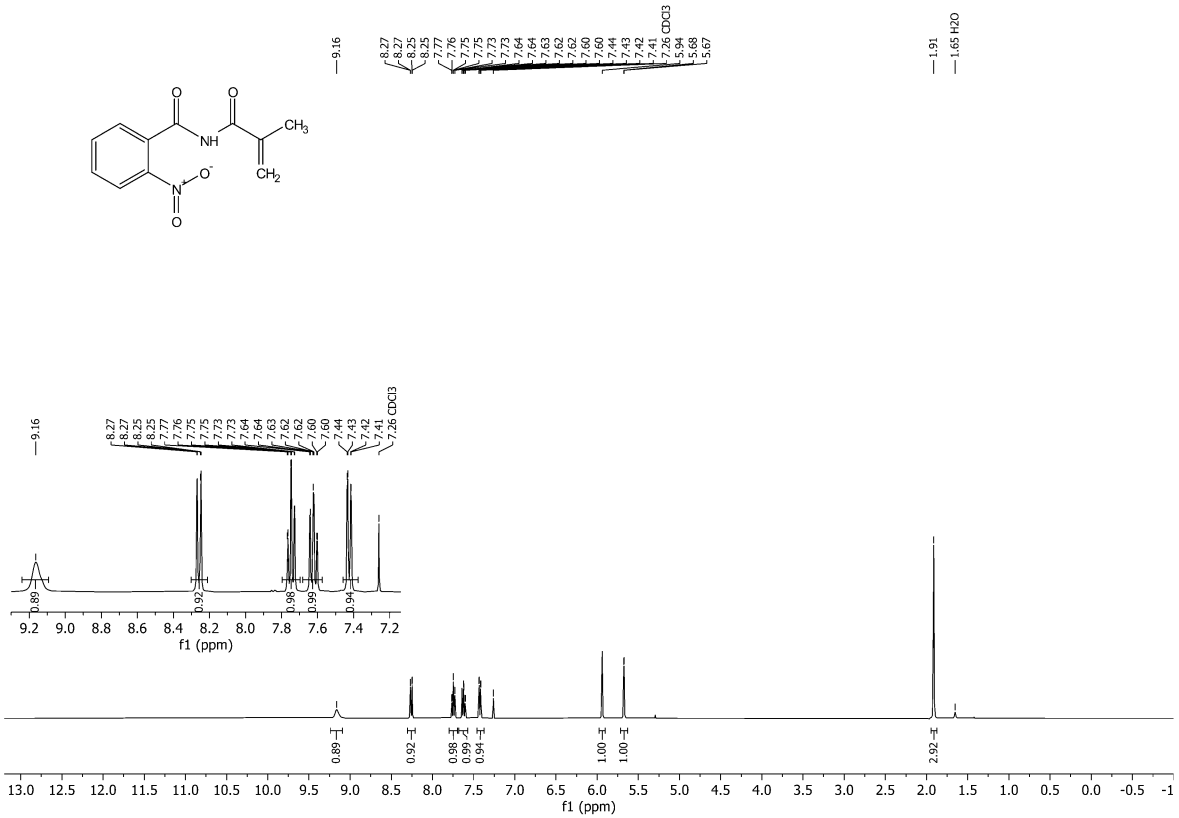
## *N*-Formyl-2-nitrobenzamide (5e)

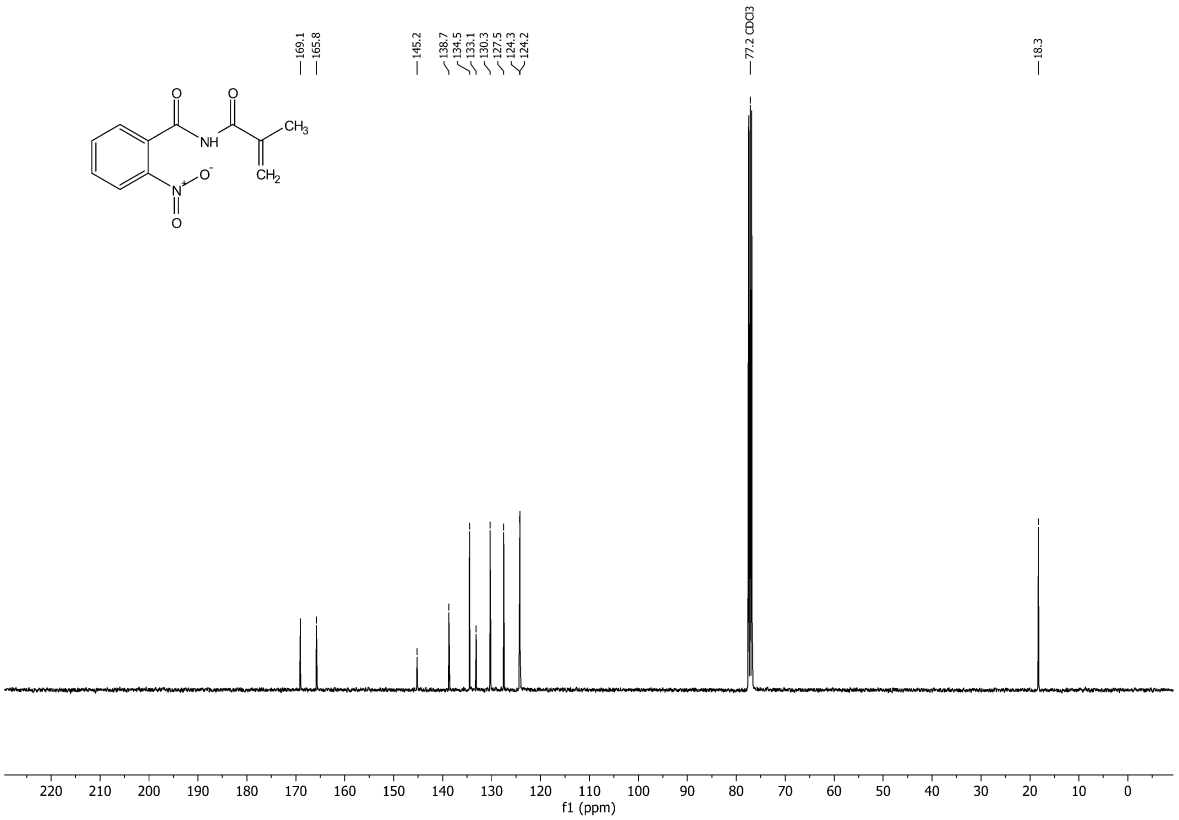


## *N*-(2-Chloroacetyl)-2-nitrobenzamide (5f)

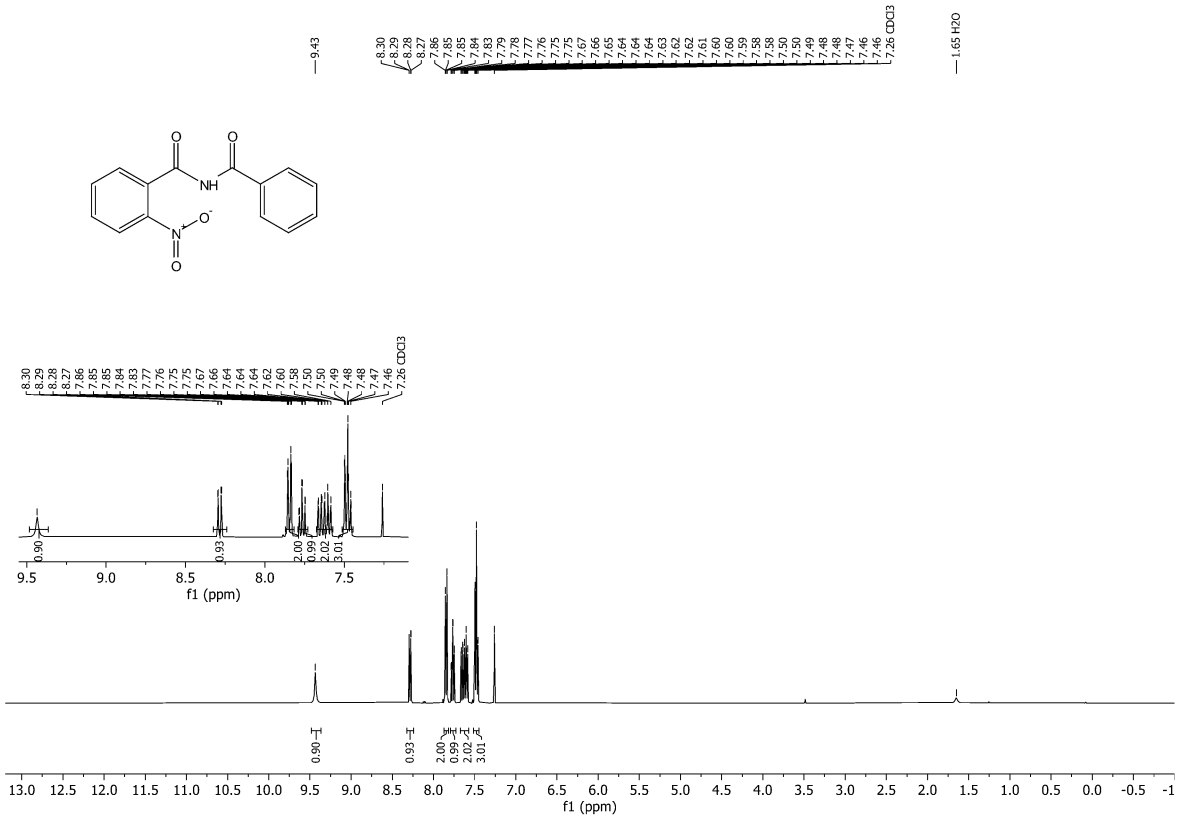


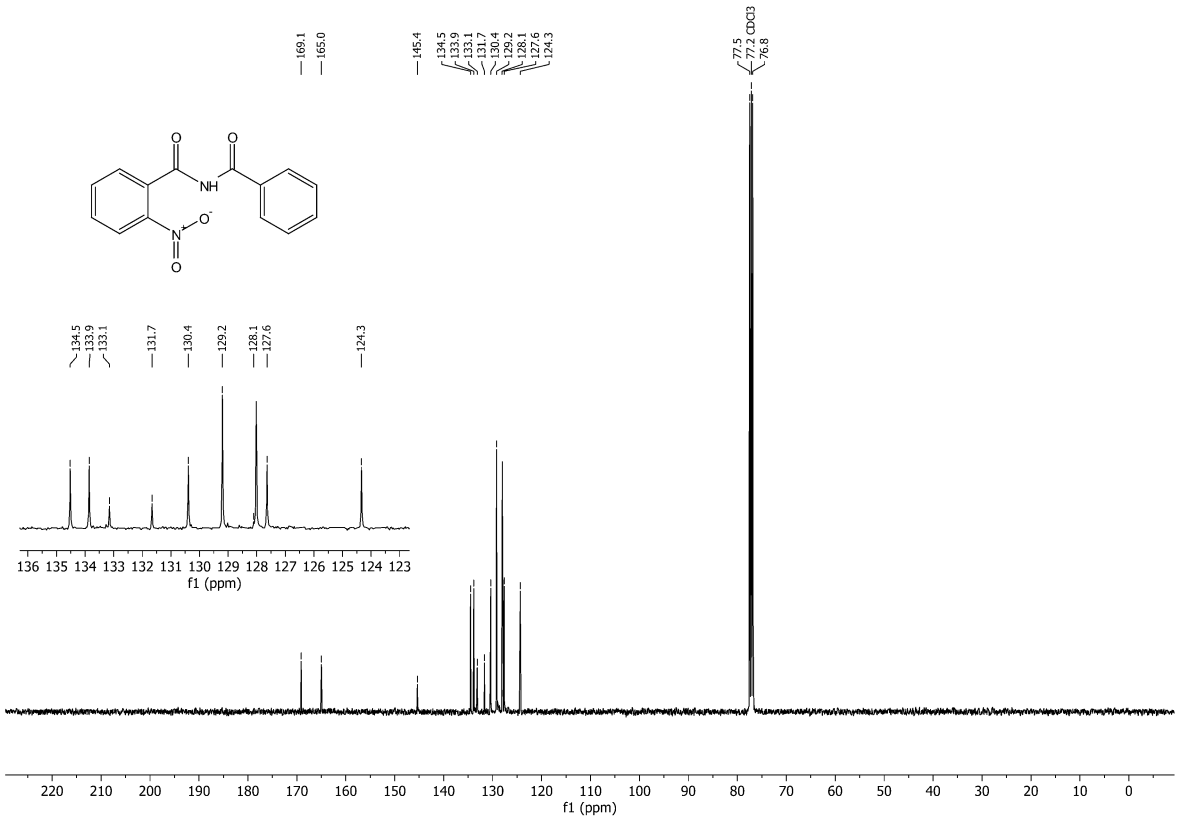
## *N*-Methacryloyl-2-nitrobenzamide (5g)



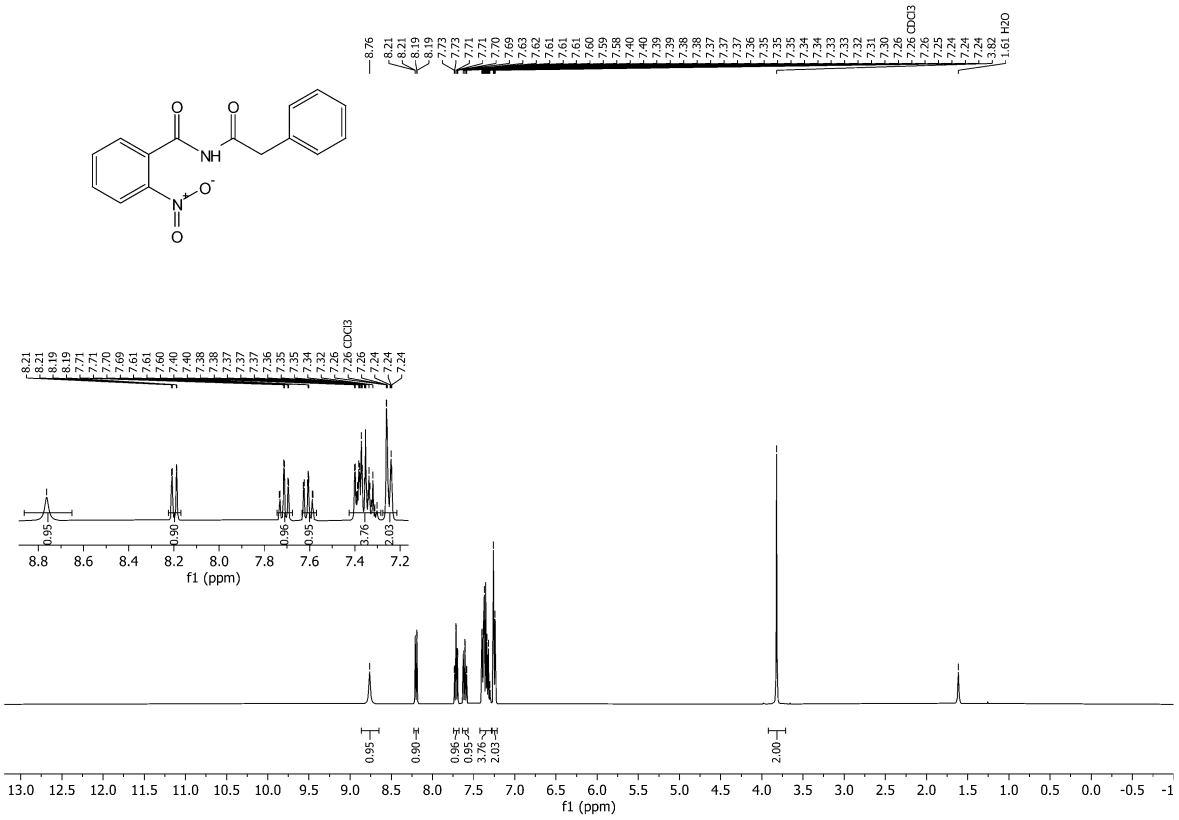



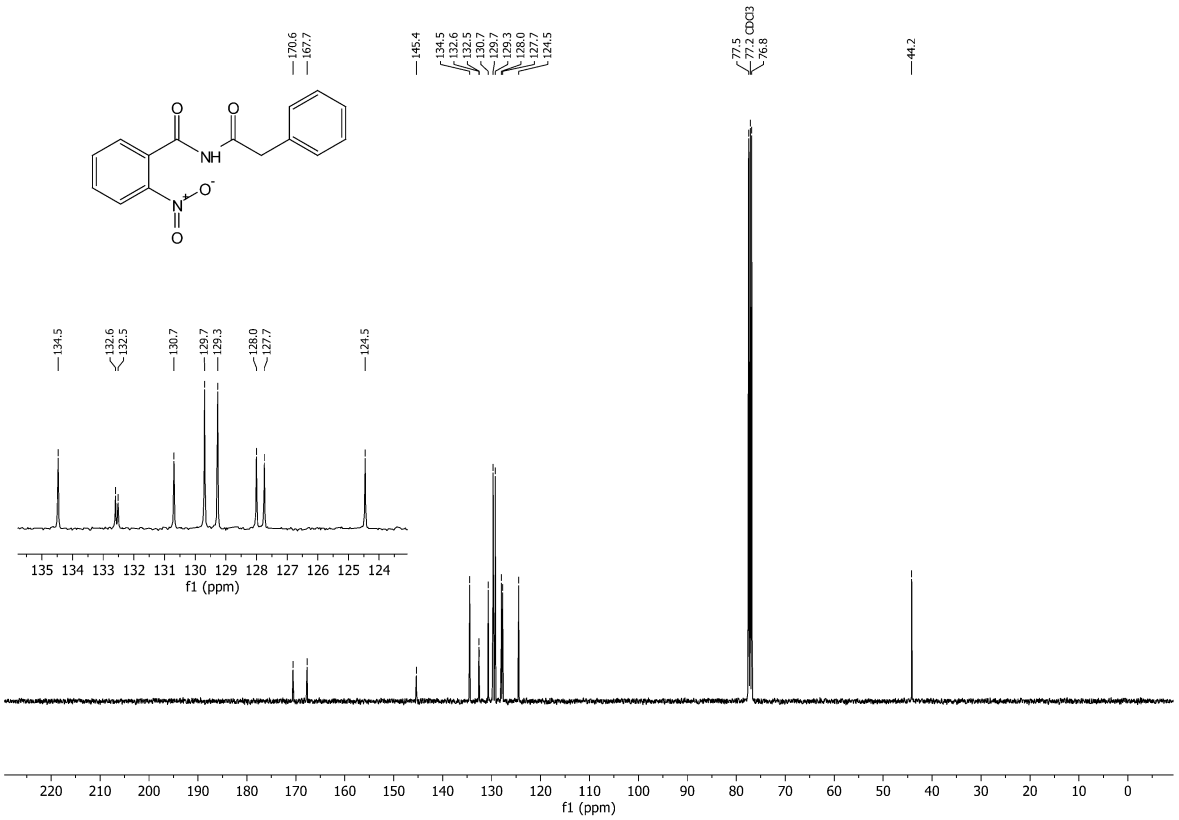
## *N*-Benzoyl-2-nitrobenzamide (5h)



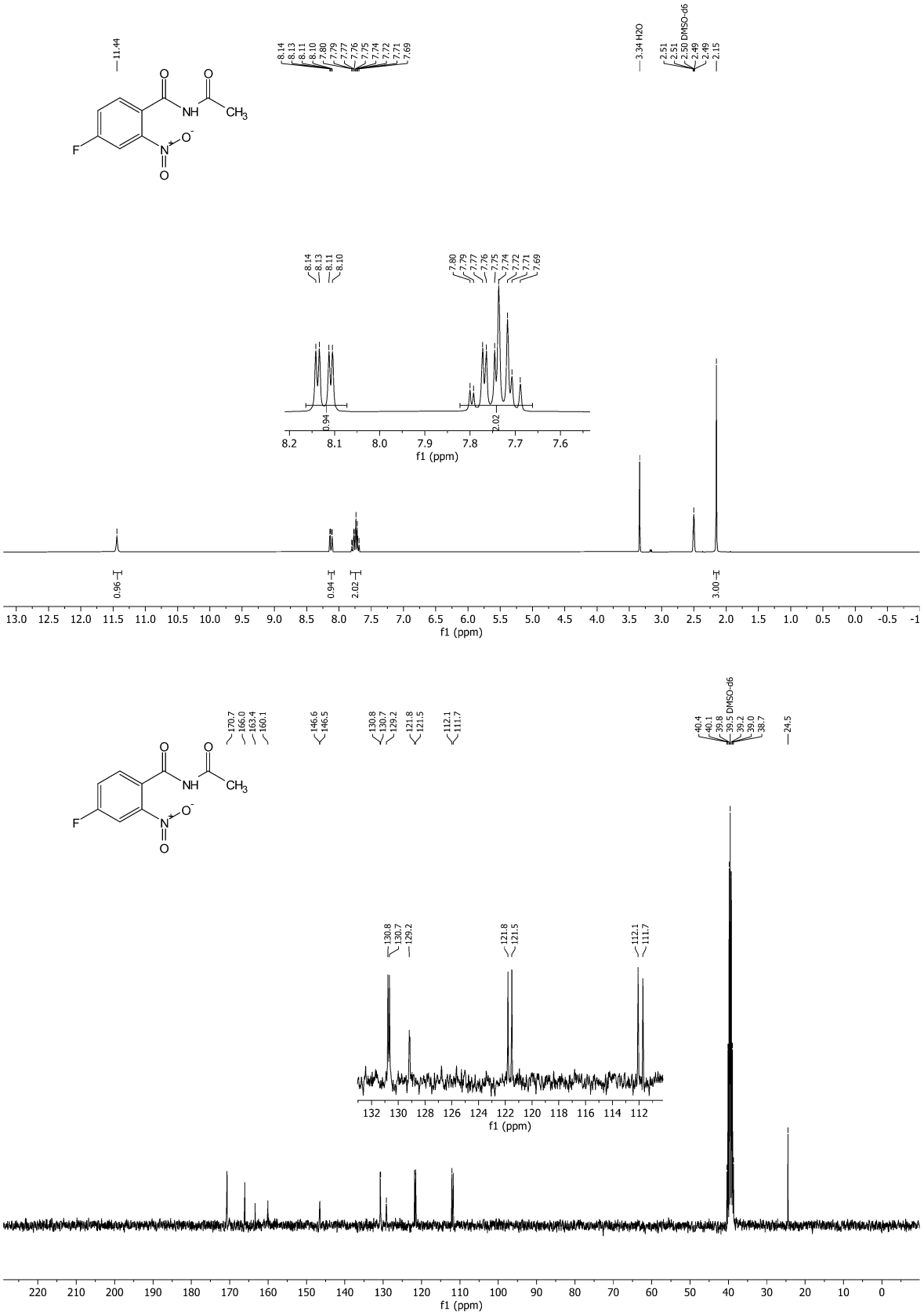



## 2-Nitro-*N*-(2-phenylacetyl)benzamide (5i)



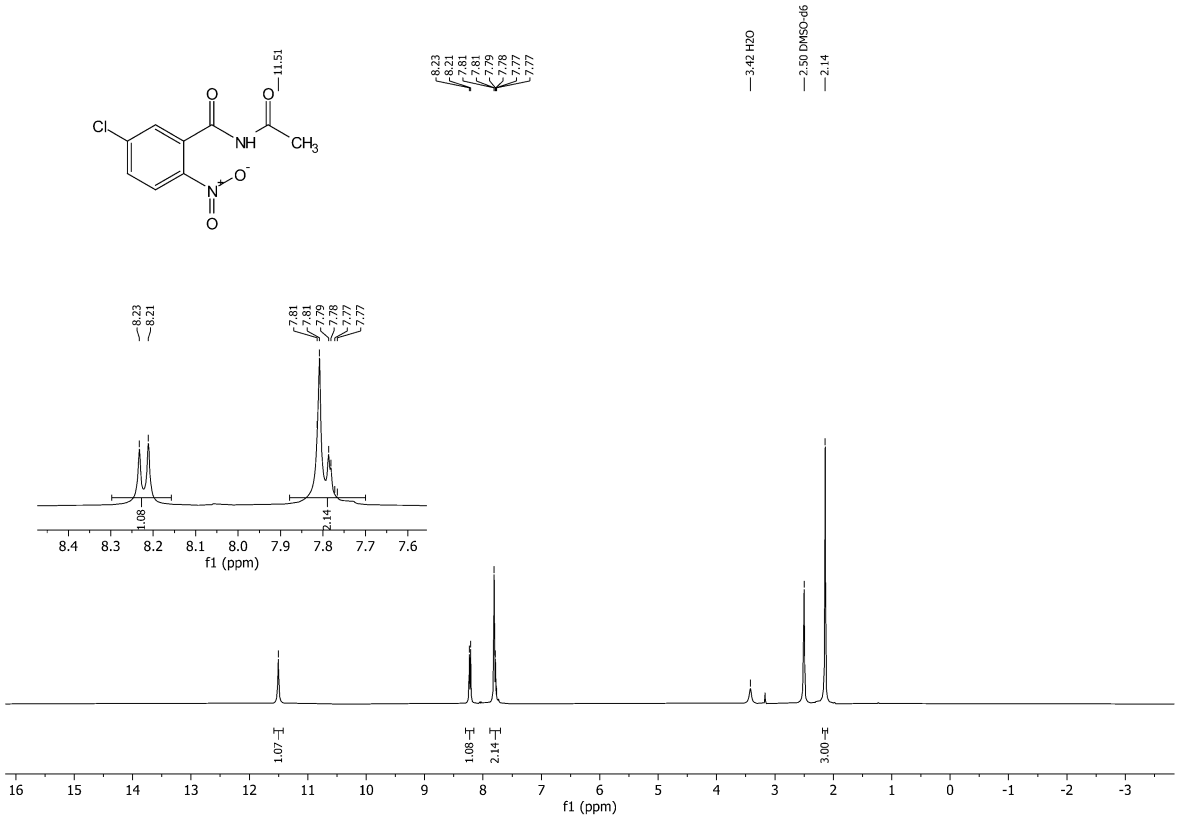



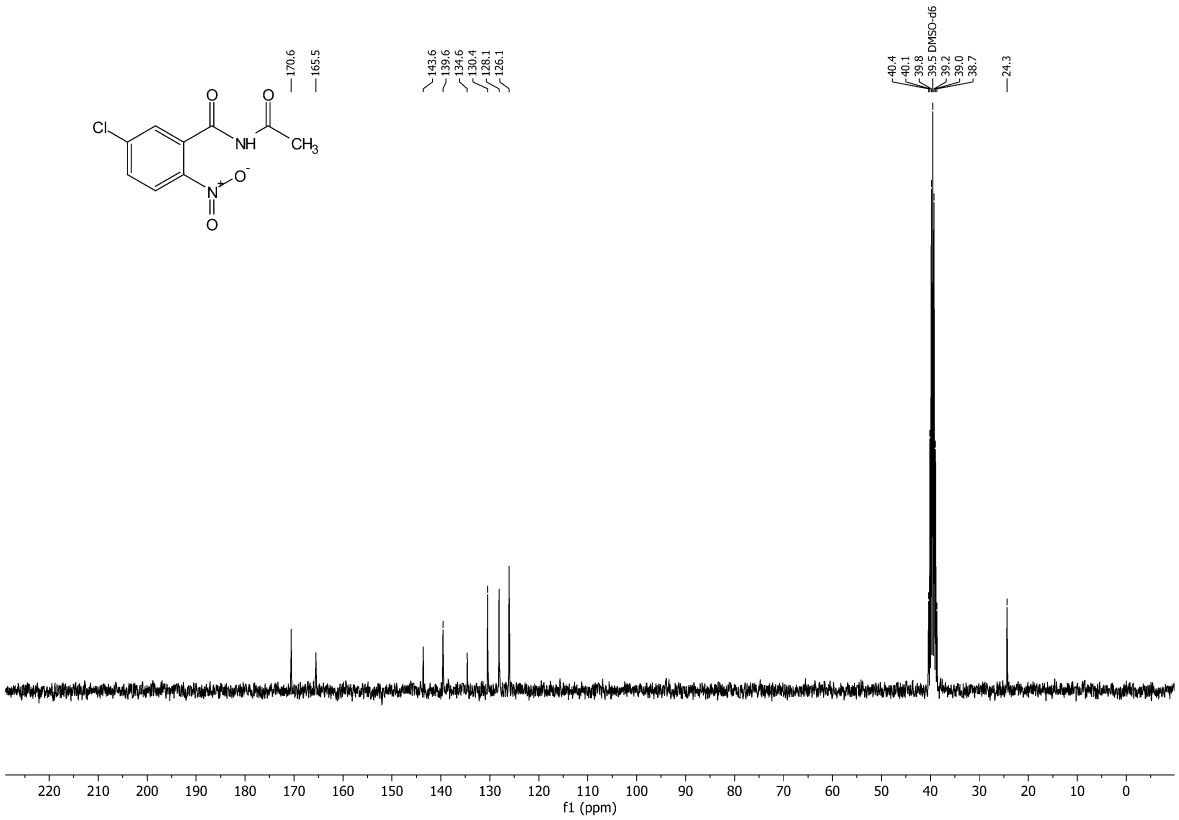
## *N*-Acetyl-4-fluoro-2-nitrobenzamide (5j)



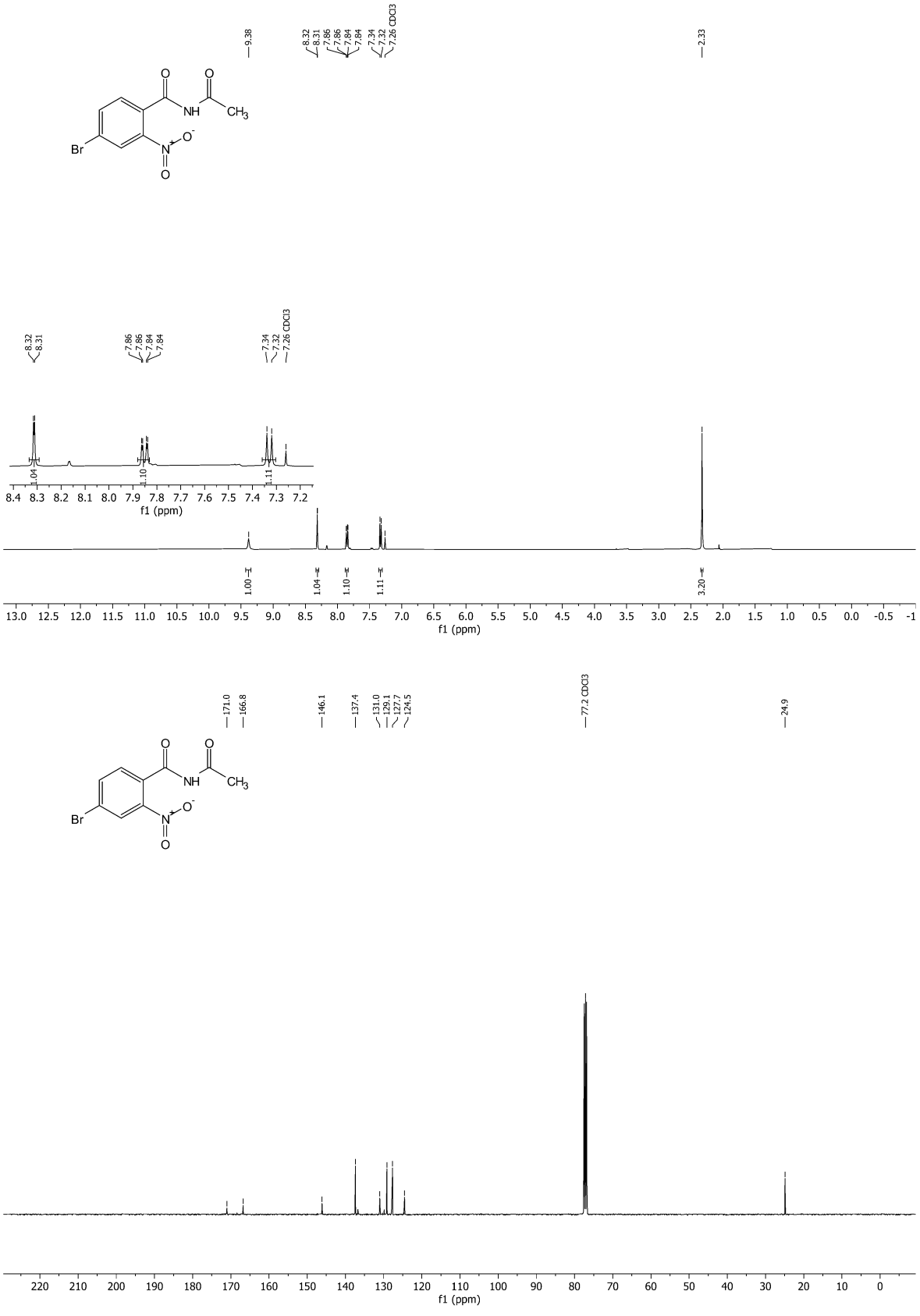

107.92
107.95
107.97
108.00
O
O
NH
CH3
F
N+
O-
O
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

## *N*-Acetyl-5-chloro-2-nitrobenzamide (5k)

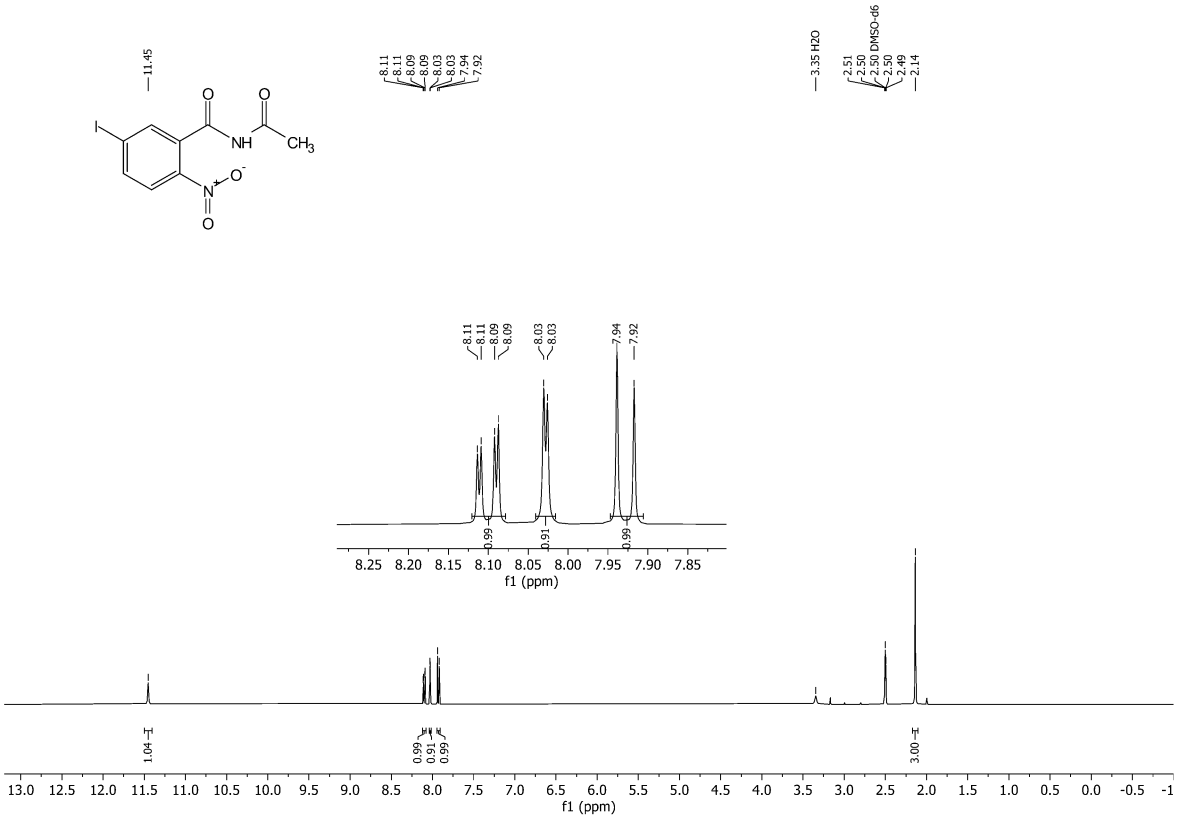


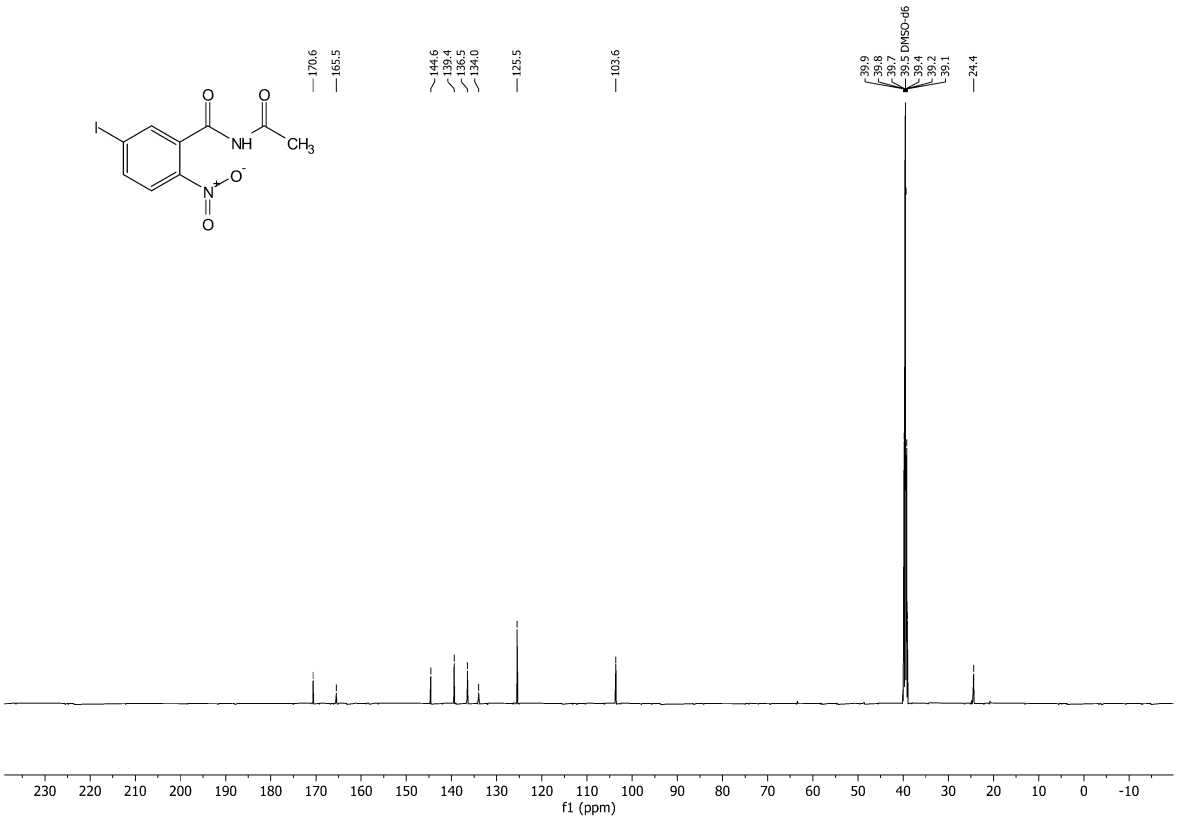



## *N*-Acetyl-4-bromo-2-nitrobenzamide (5l)

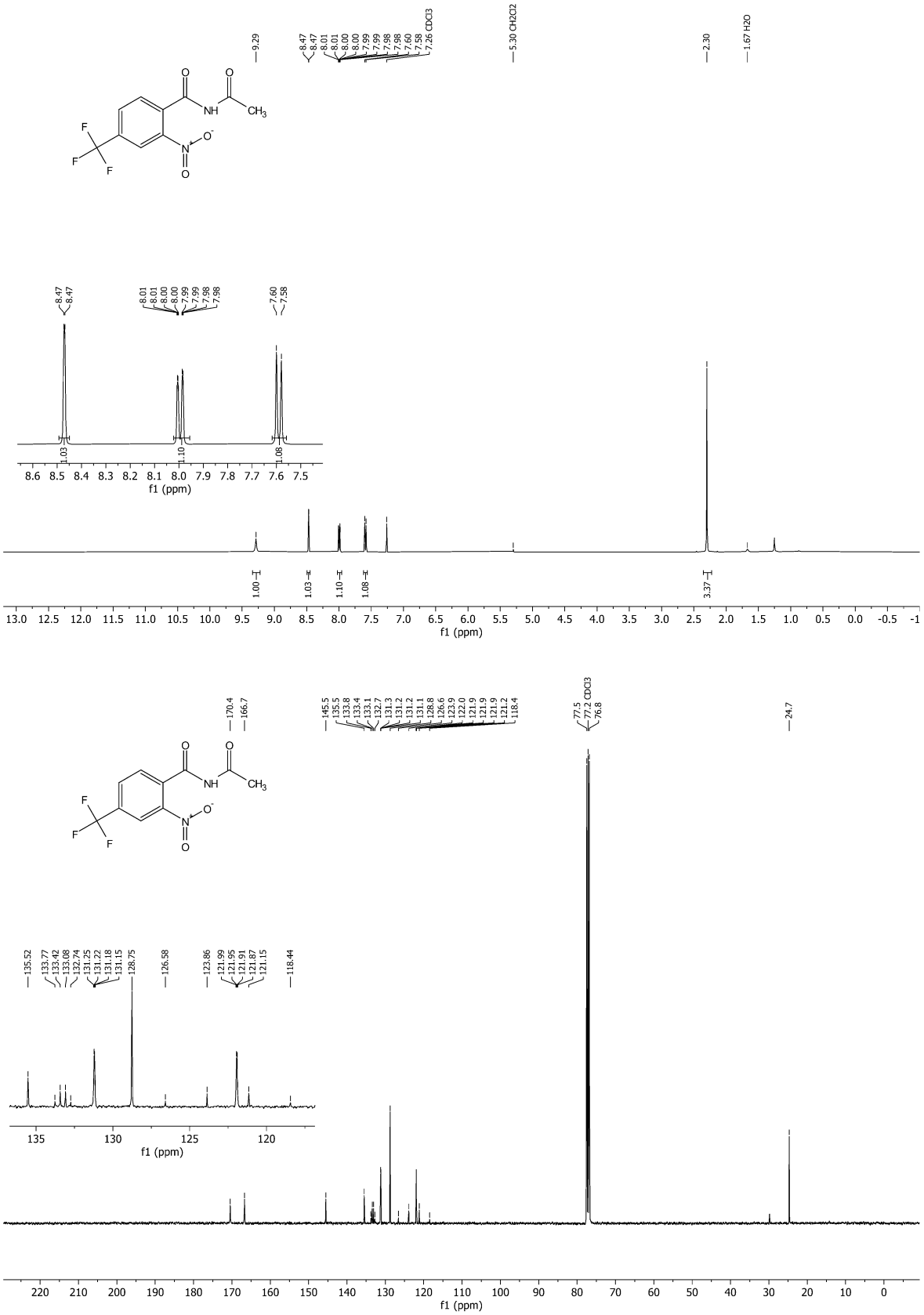


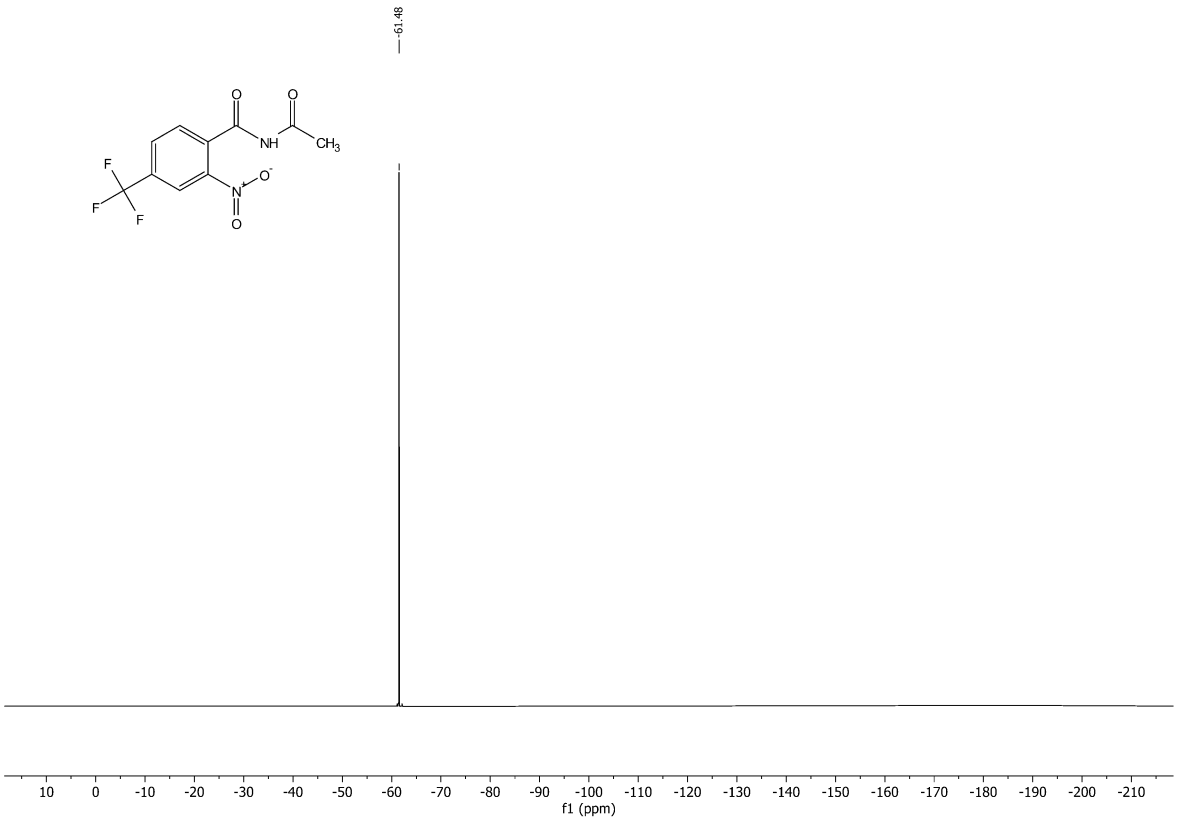
## *N*-Acetyl-5-iodo-2-nitrobenzamide (5m)



## *N*-Acetyl-2-nitro-4-(trifluoromethyl)benzamide (5n)




-61.48
O
O
NH
CH3
F
F
F
N+
O-
O
10
0
-10
-20
-30
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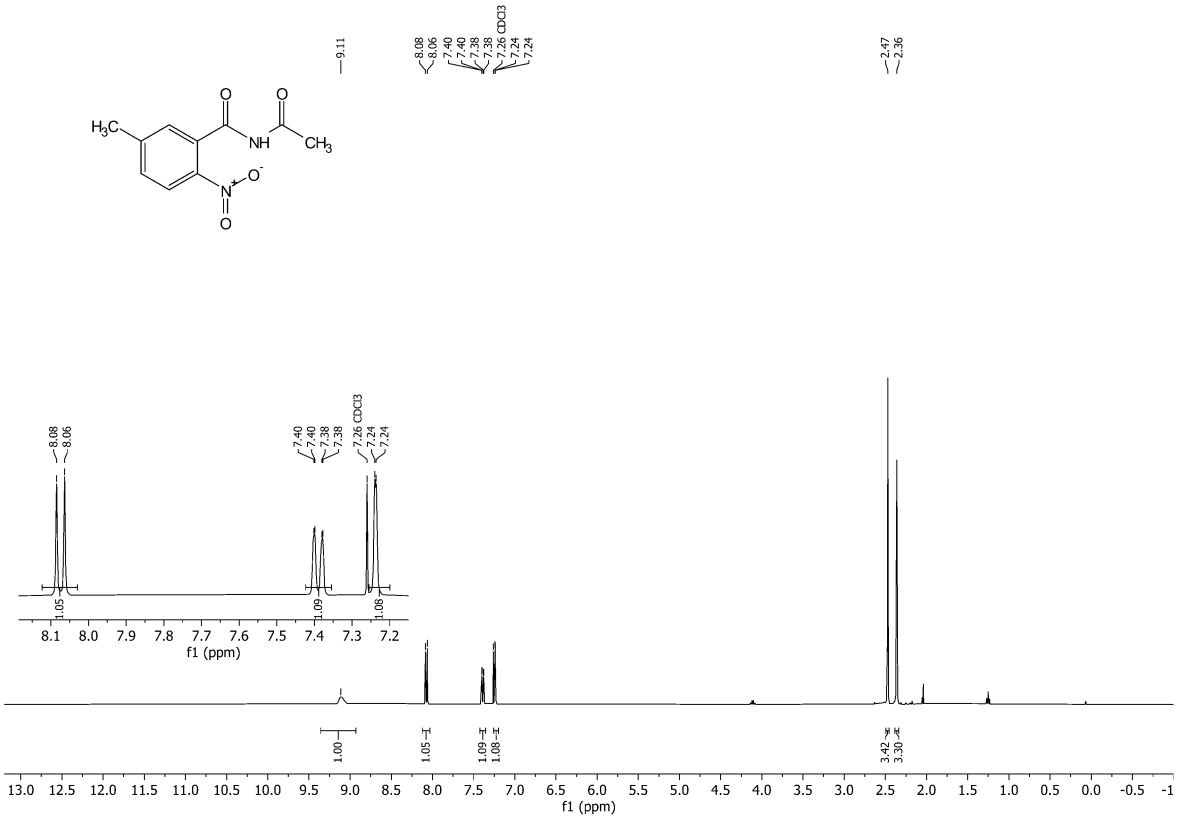
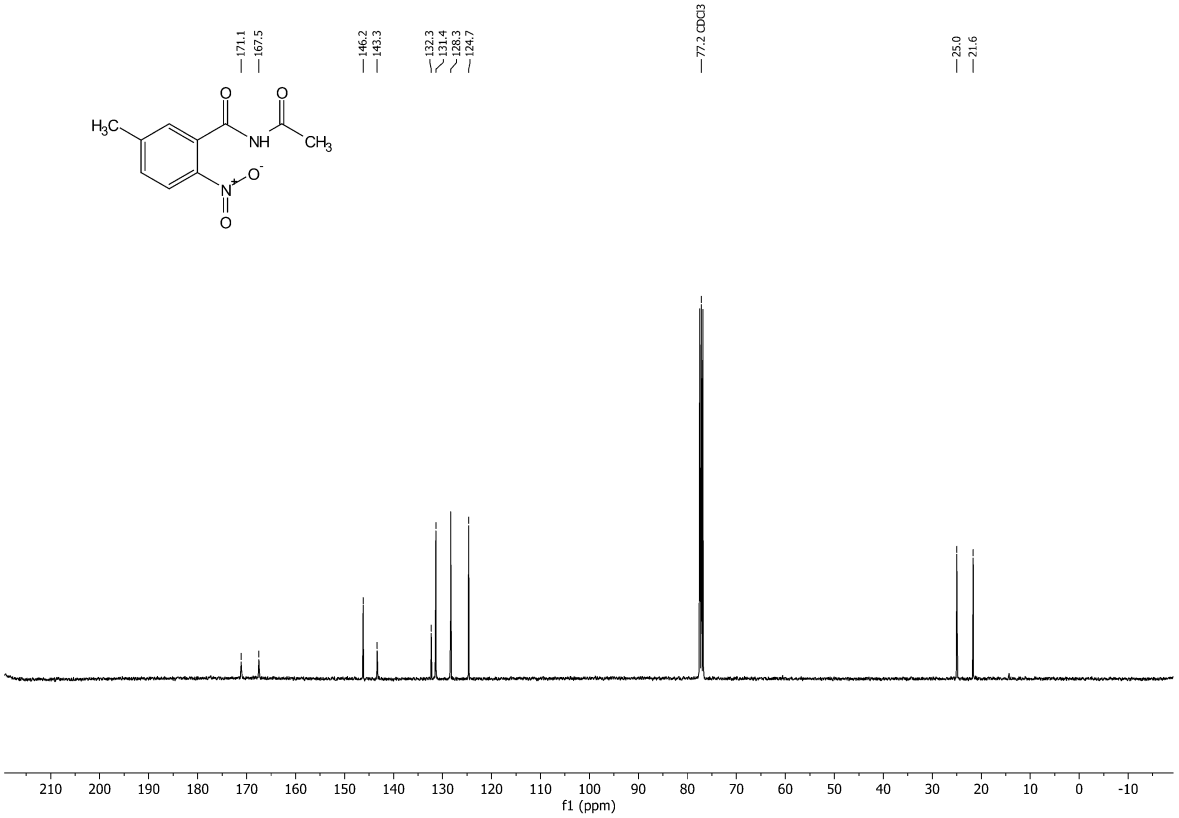
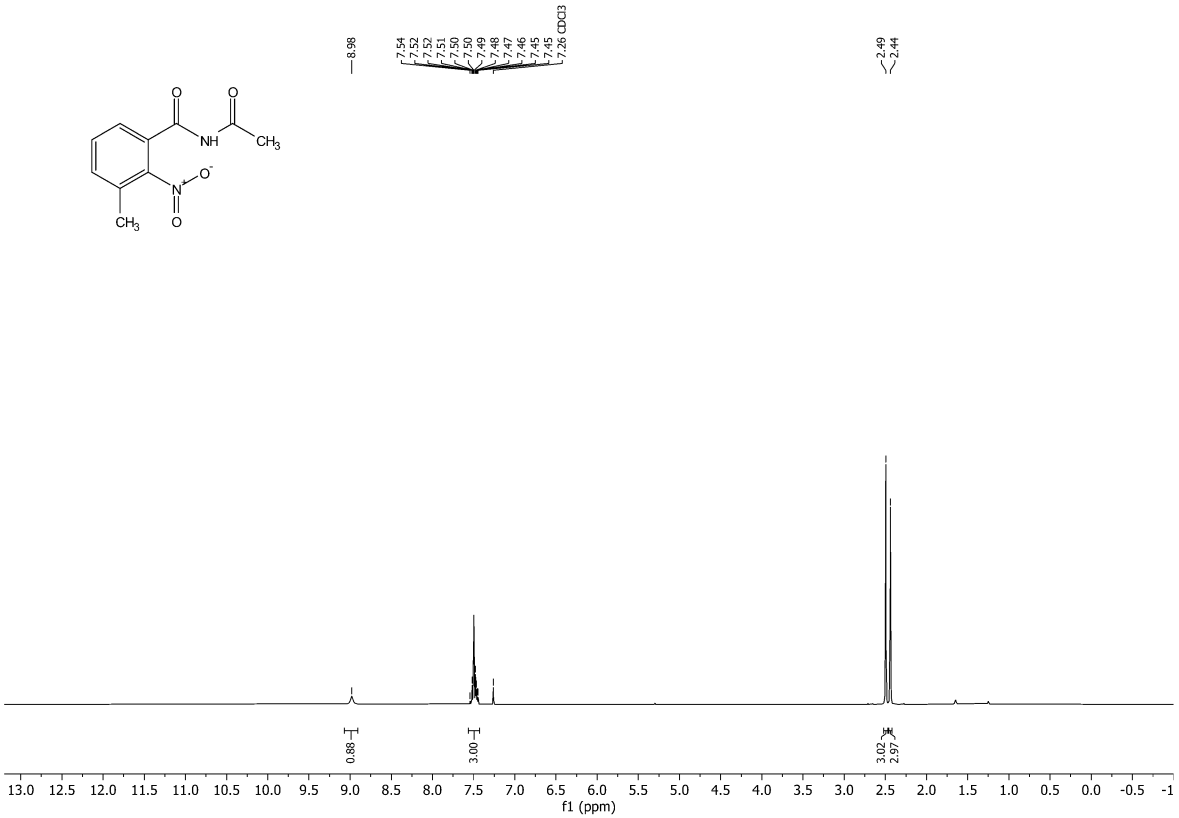
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

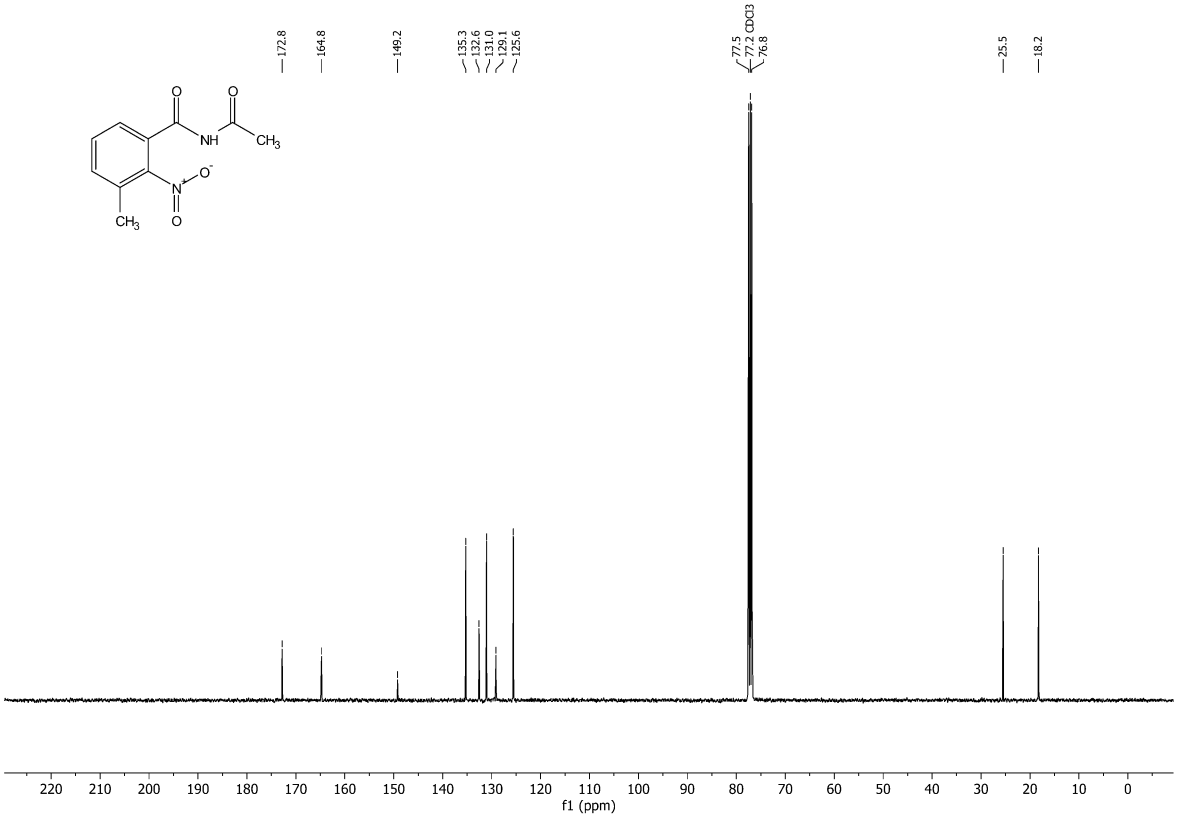
## *N*-Acetyl-5-methyl-2-nitrobenzamide (5o)



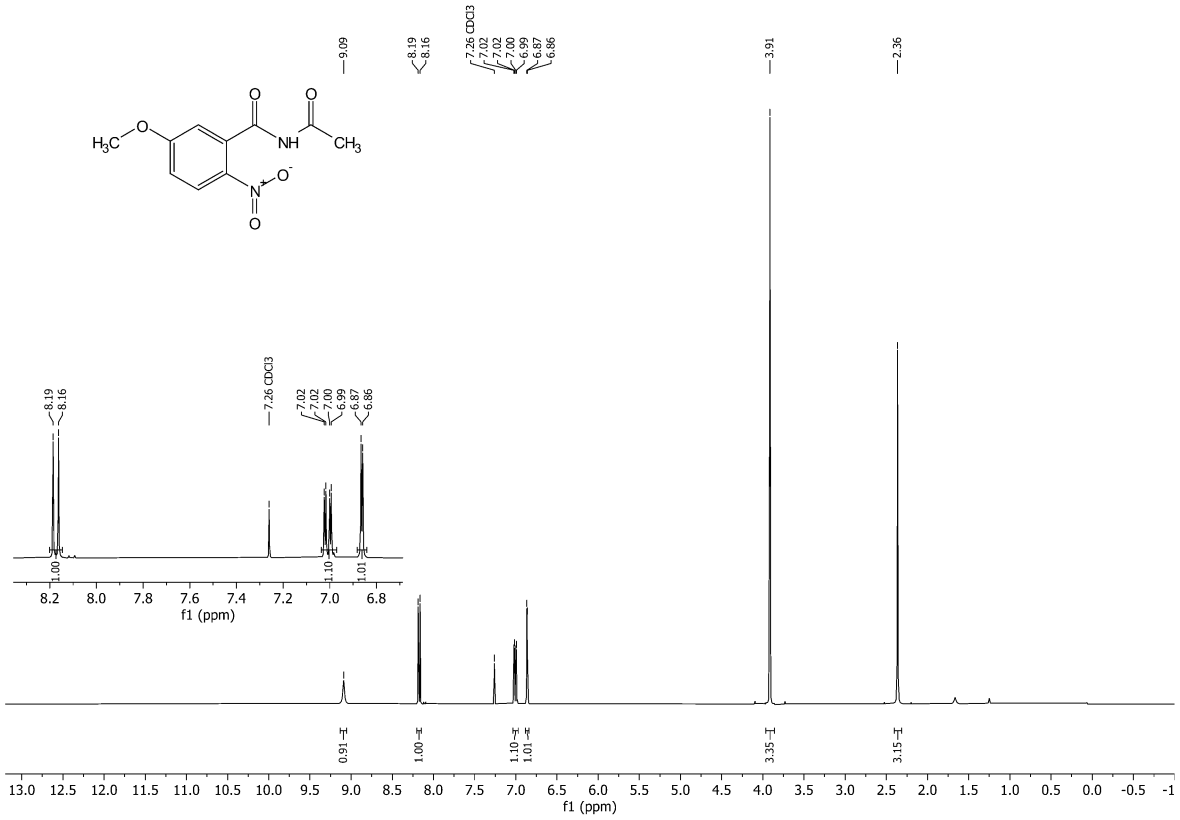



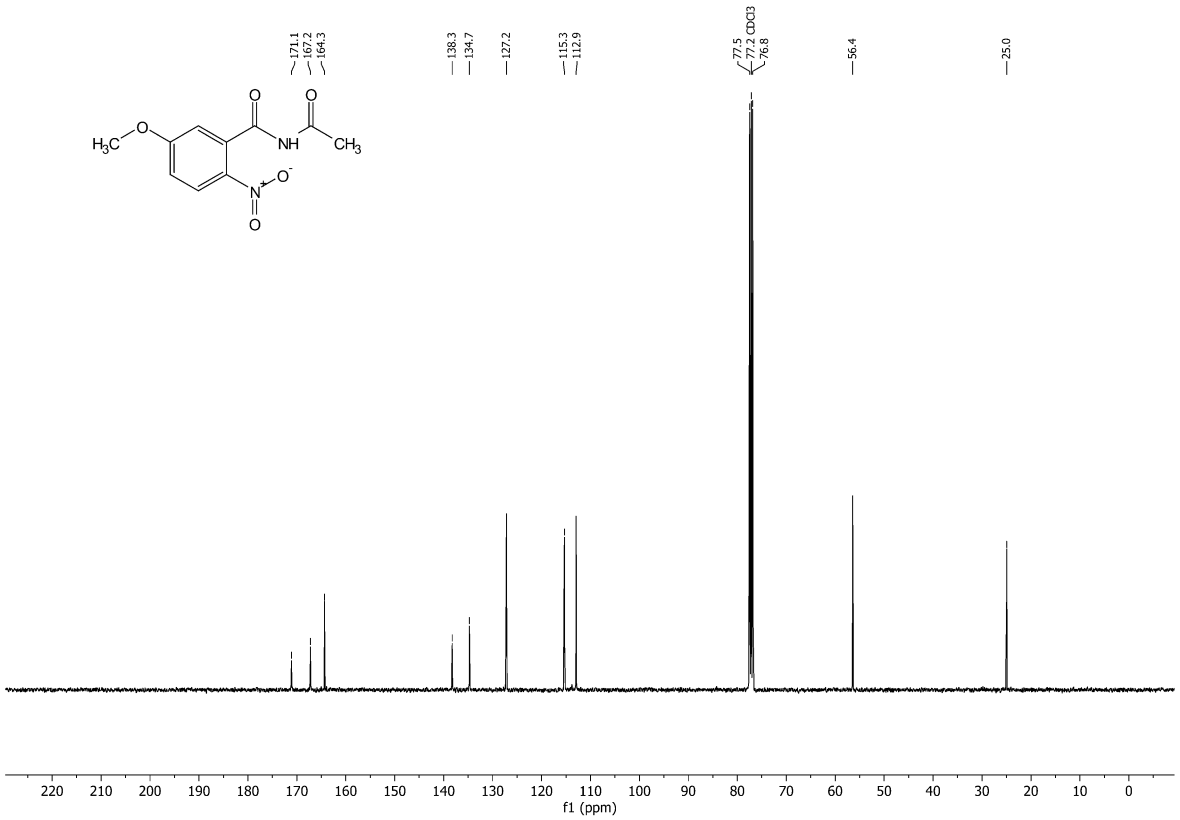
## *N*-Acetyl-3-methyl-2-nitrobenzamide (5p)



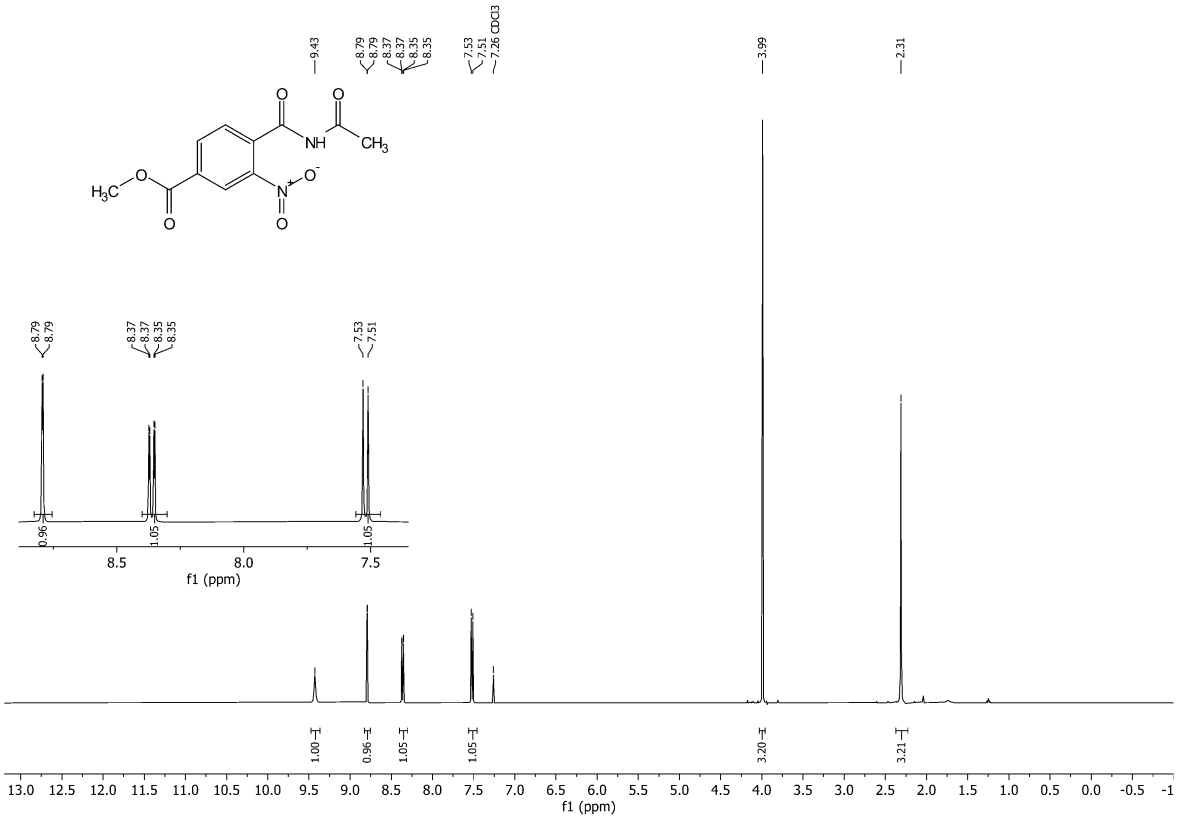



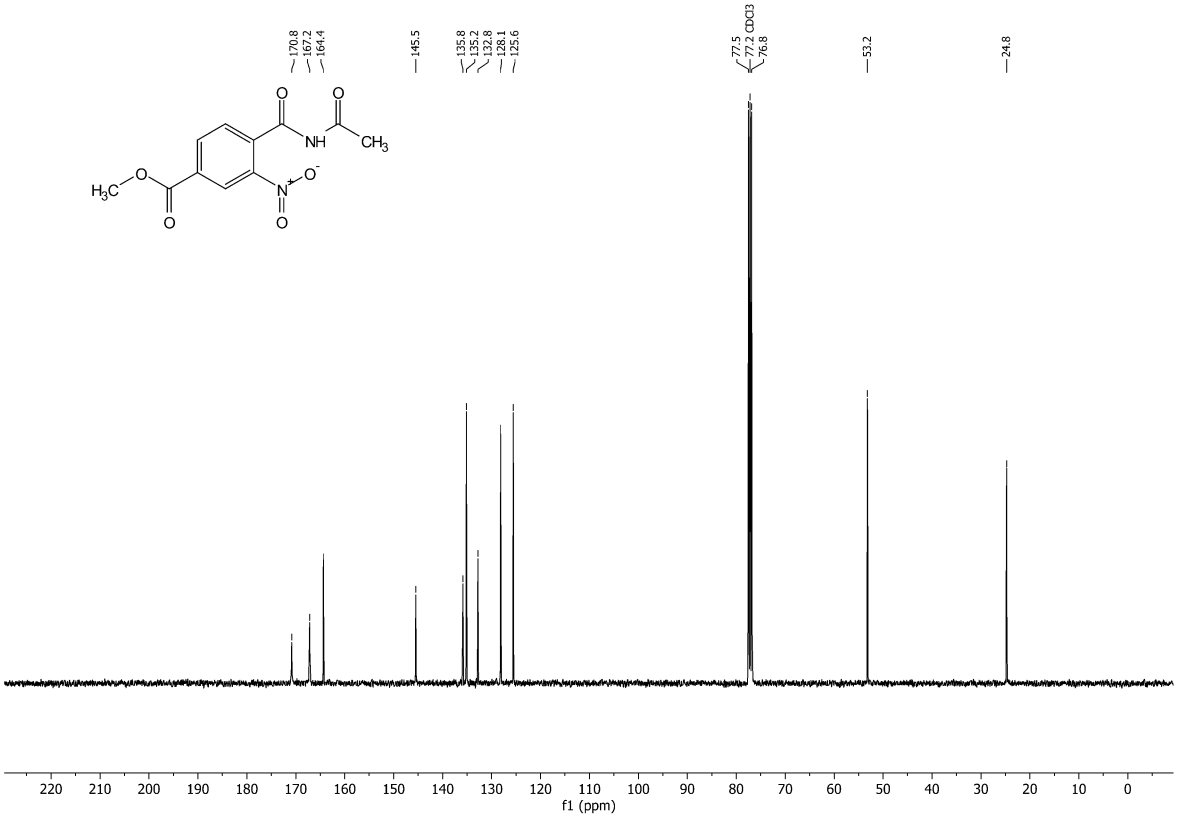
## *N*-Acetyl-5-methoxy-2-nitrobenzamide (5q)



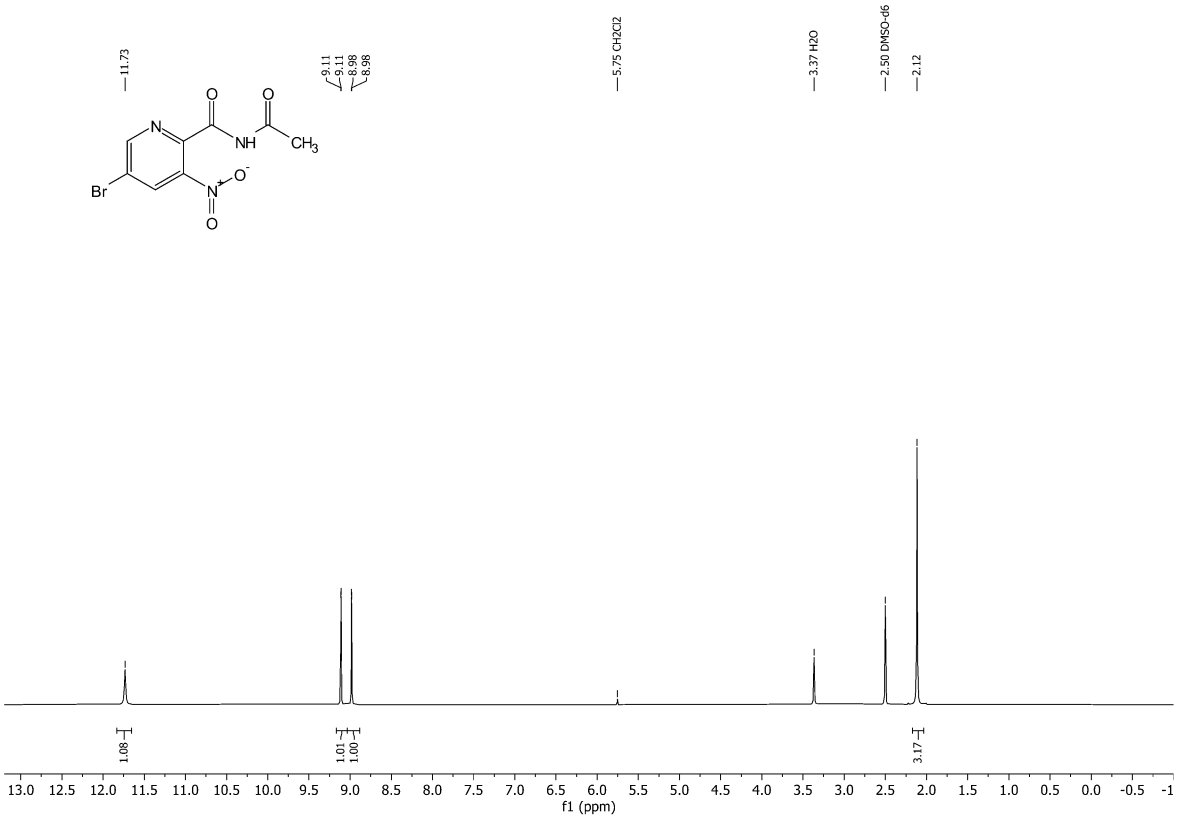



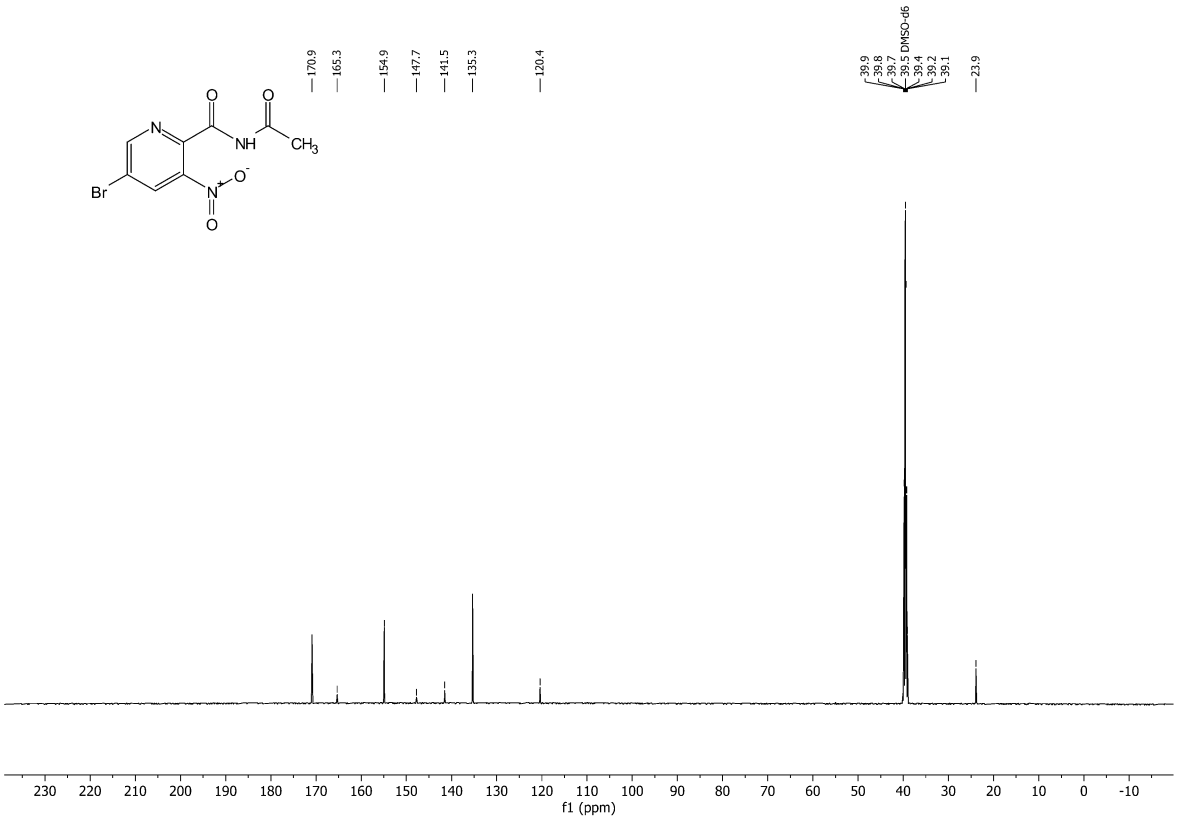
## Methyl 4-(acetylcarbamoyl)-3-nitrobenzoate (5r)



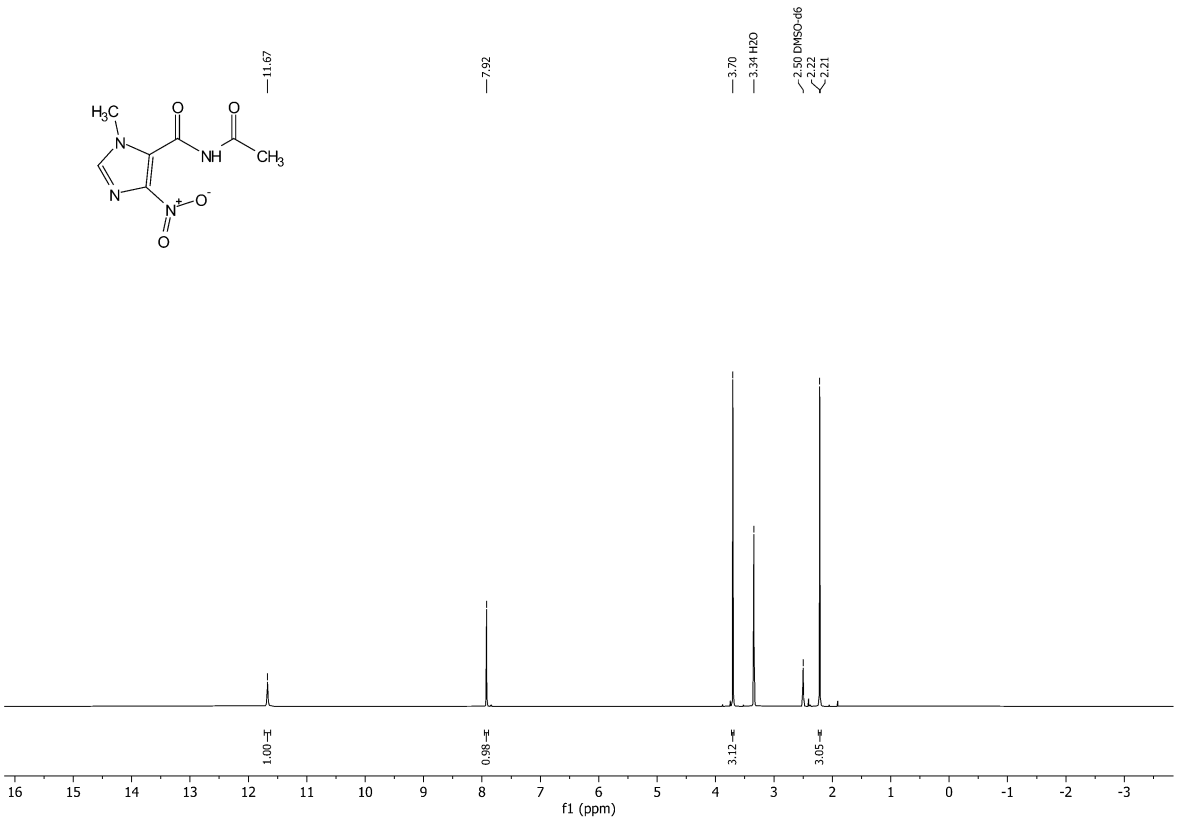



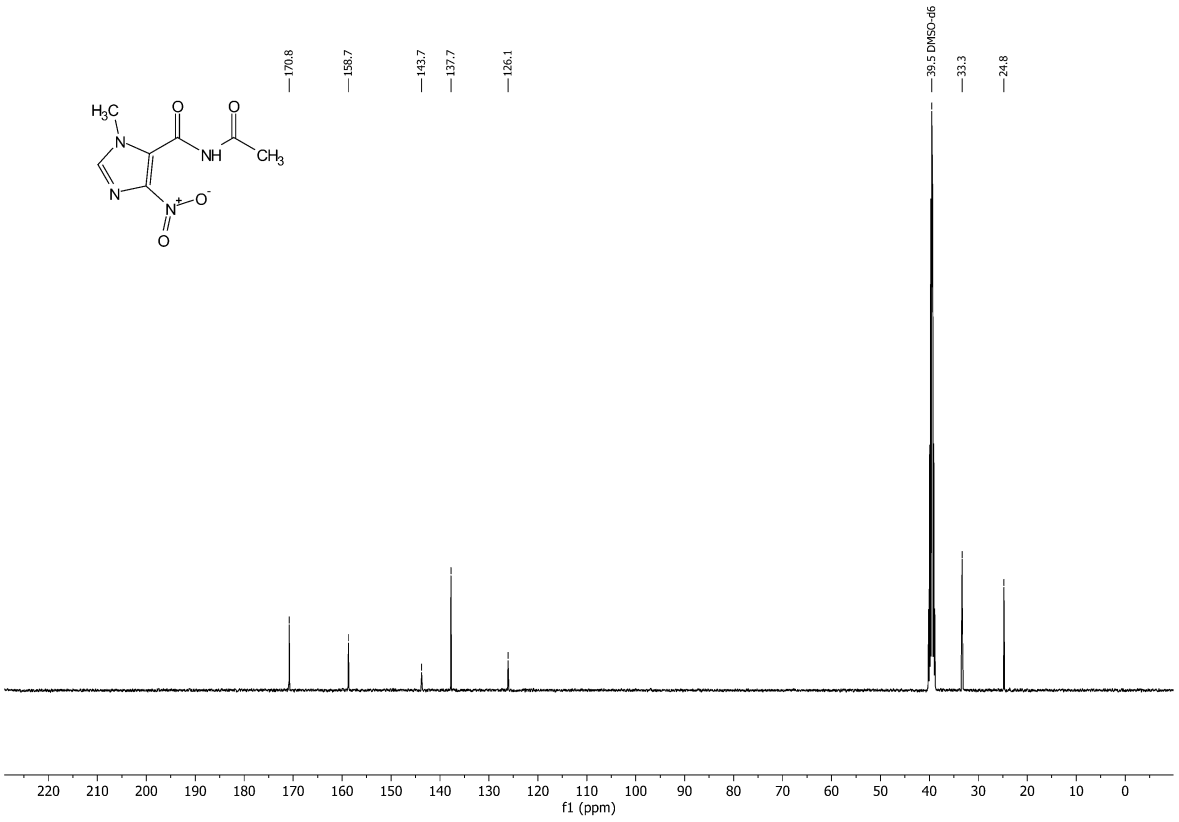
## 2-(Acetylcarbamoyl)-5-bromo-3-nitropyridine (5s)



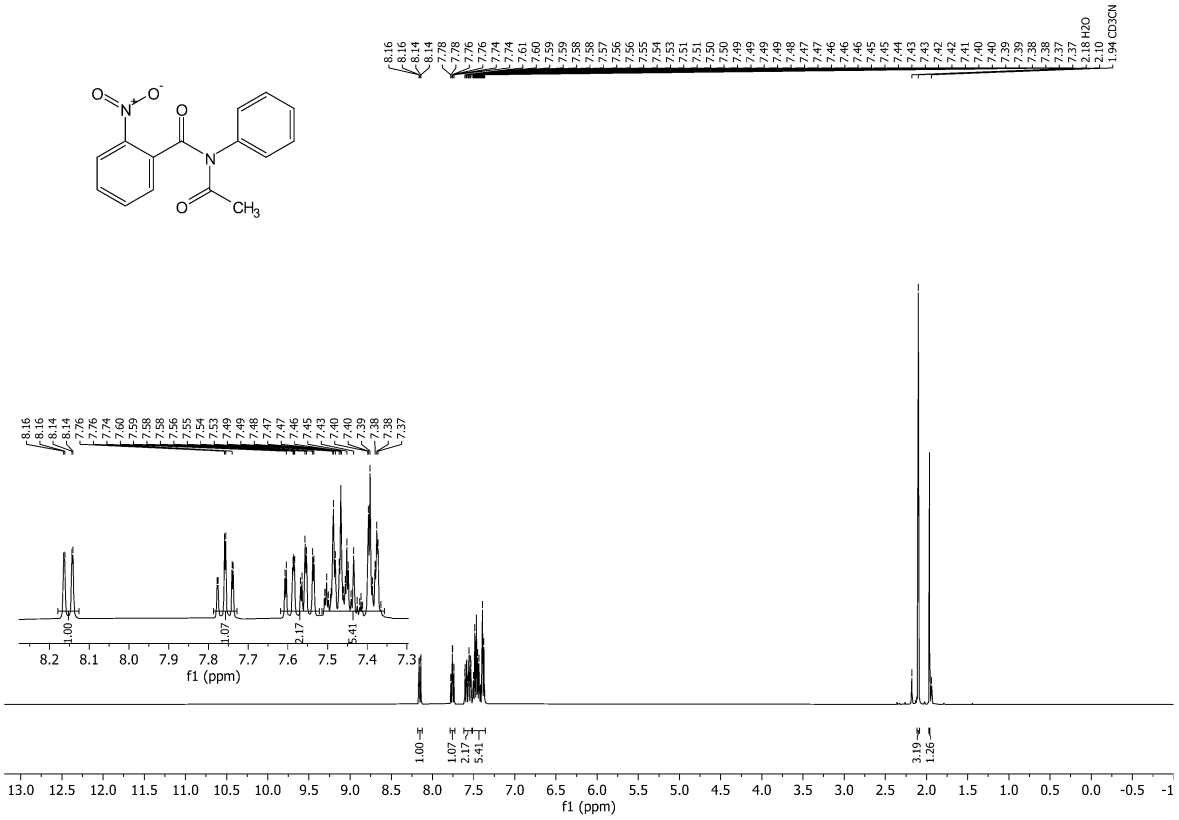



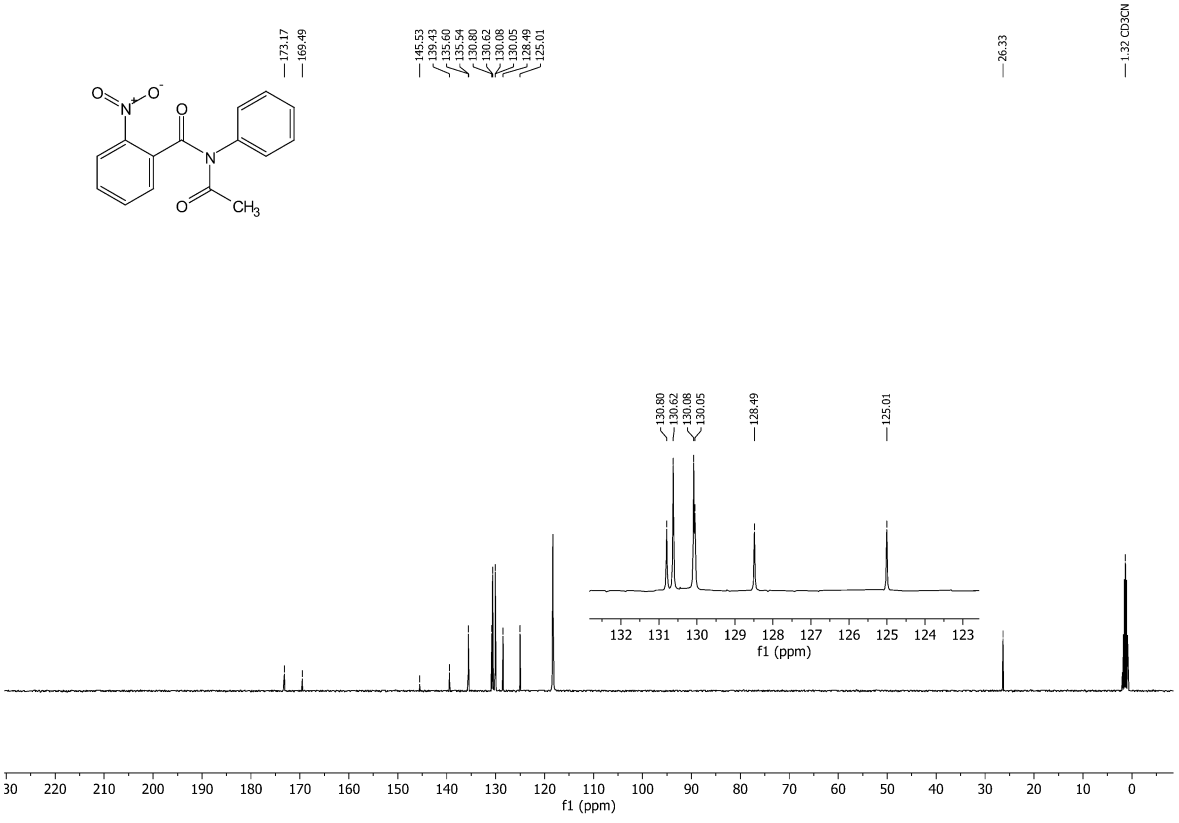
**1*H*-*N*-Acetyl-1-methyl-4-nitro-1*H*-imidazole-5-carboxamide (5t)**



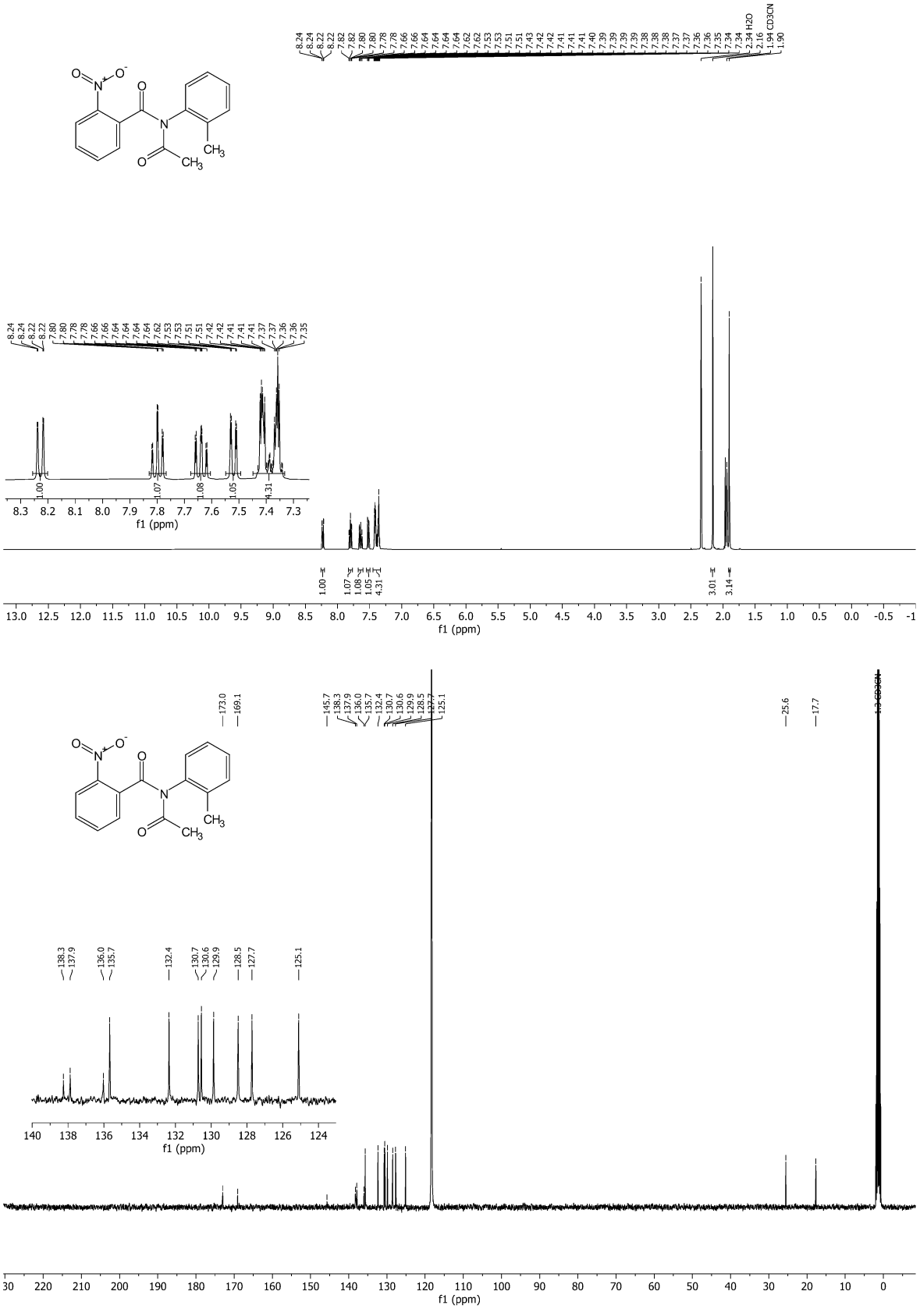



## *N*-Acetyl-2-nitro-*N*-phenylbenzamide (5u)

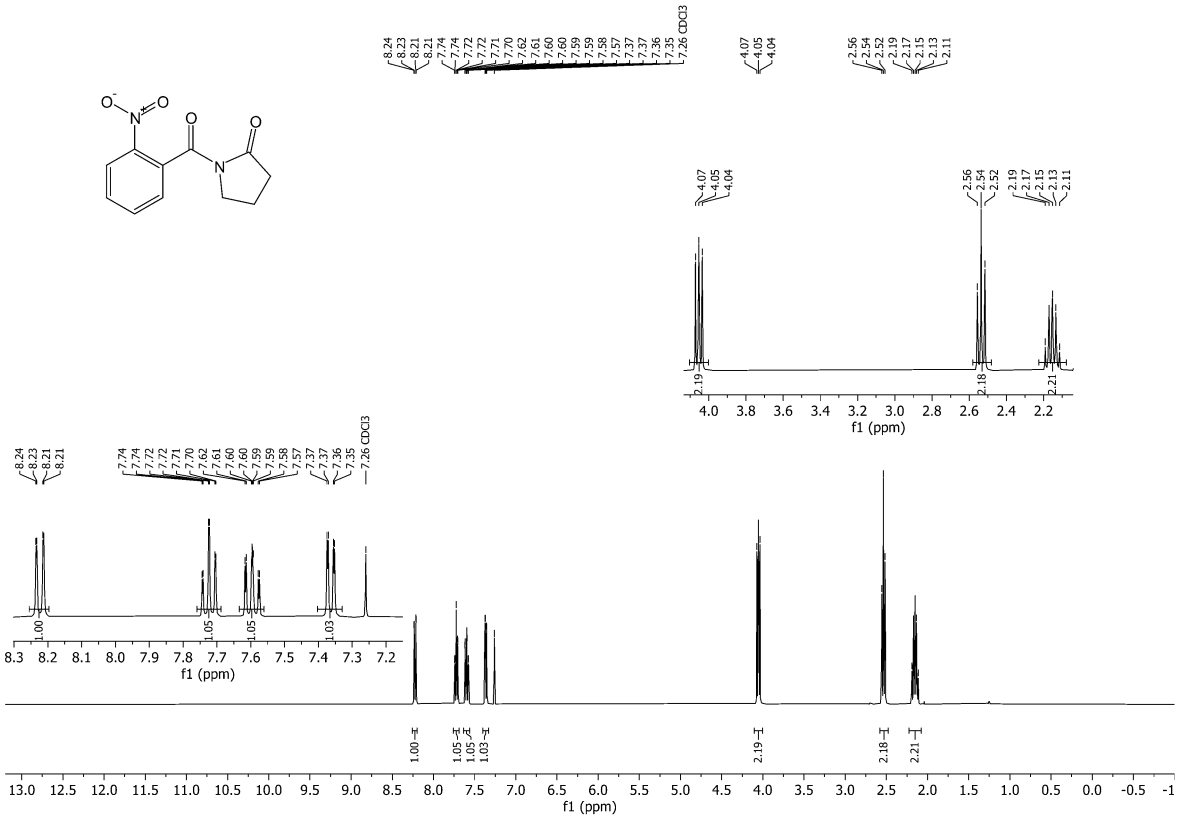


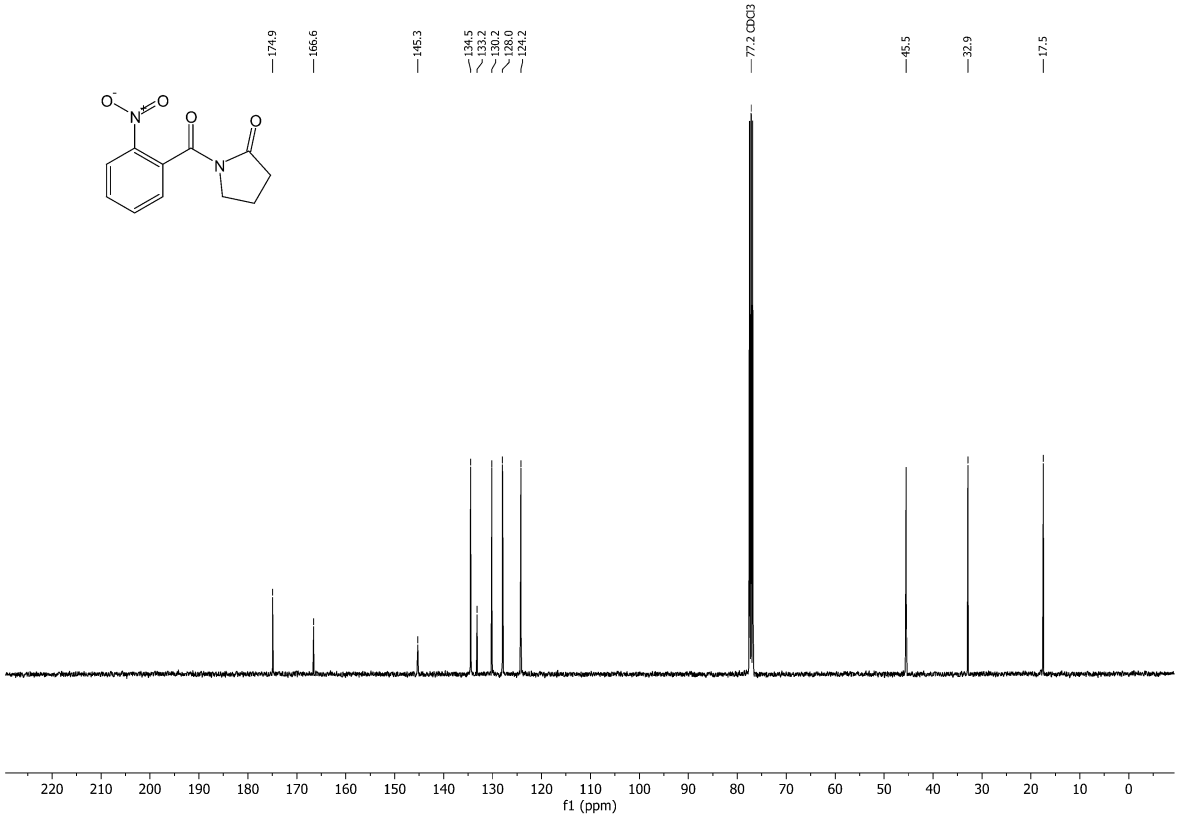



## *N*-Acetyl-2-nitro-*N*-(o-tolyl)benzamide (5v)

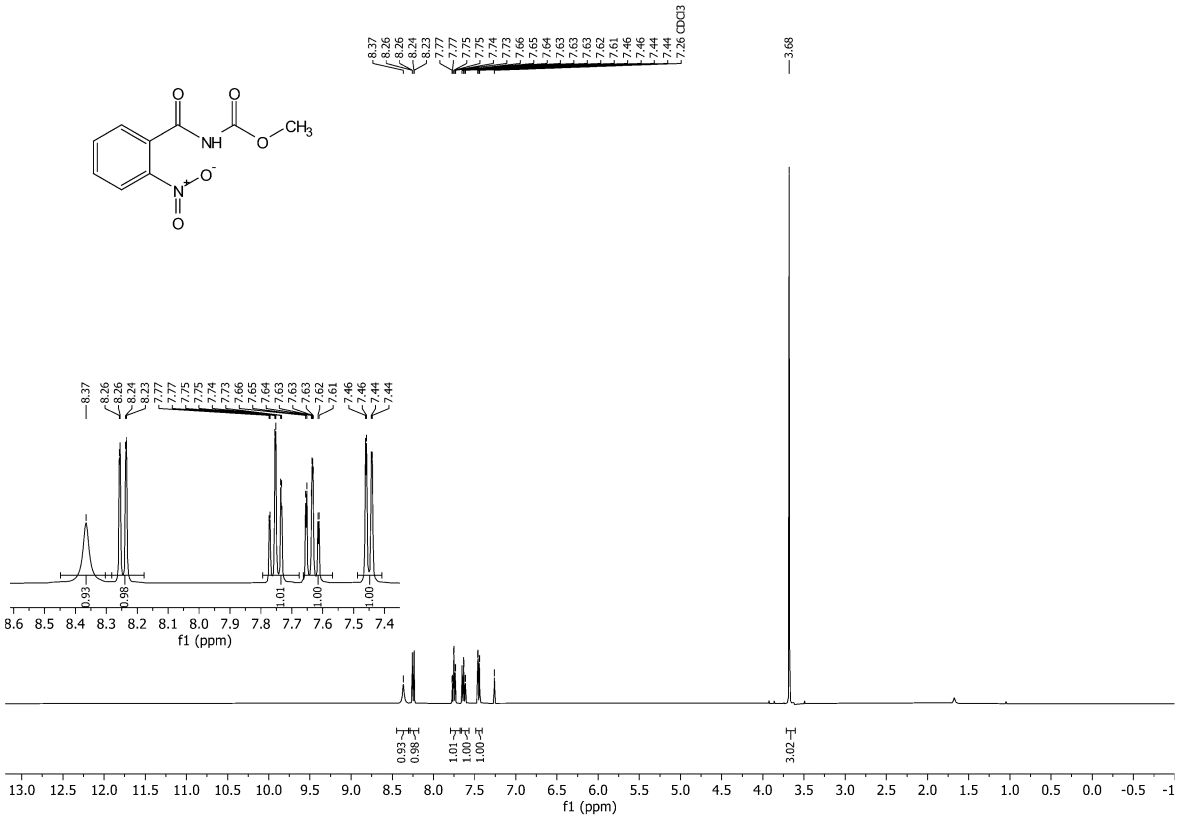


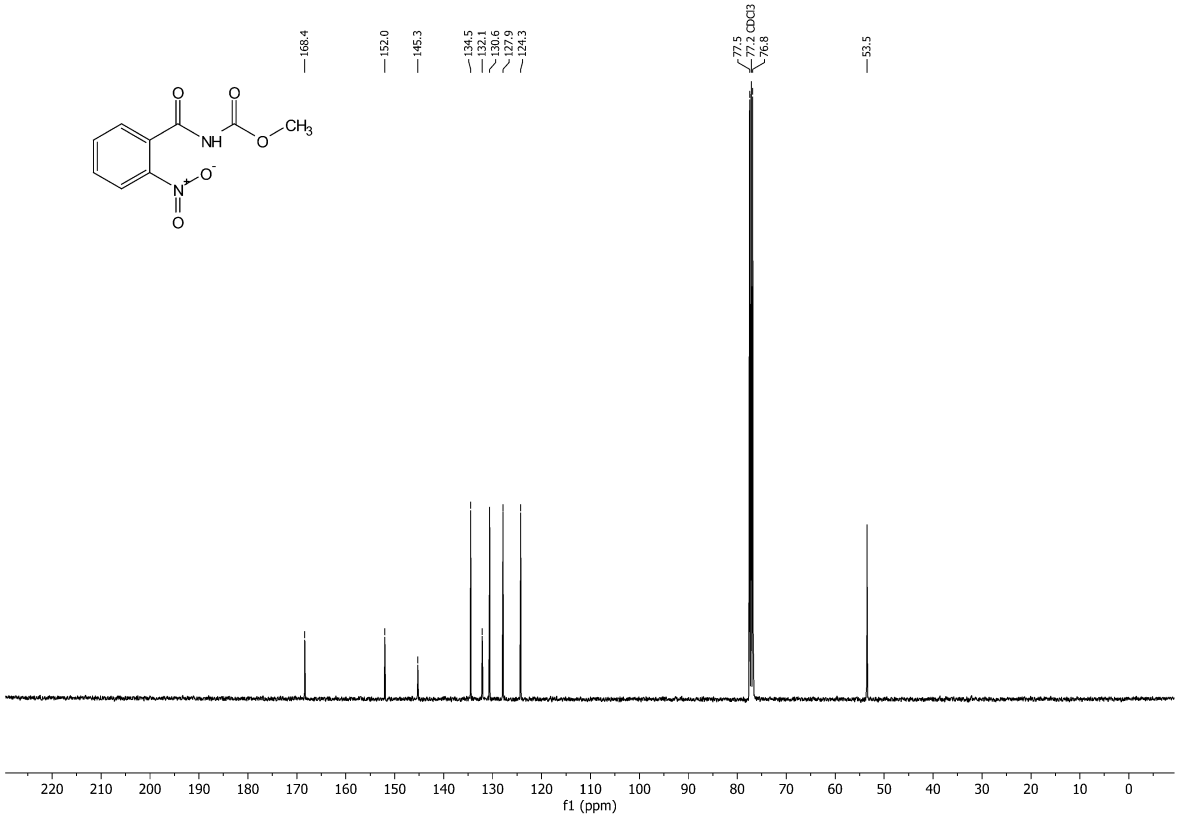
## 1-(2-Nitrobenzoyl)pyrrolidin-2-one (5w)



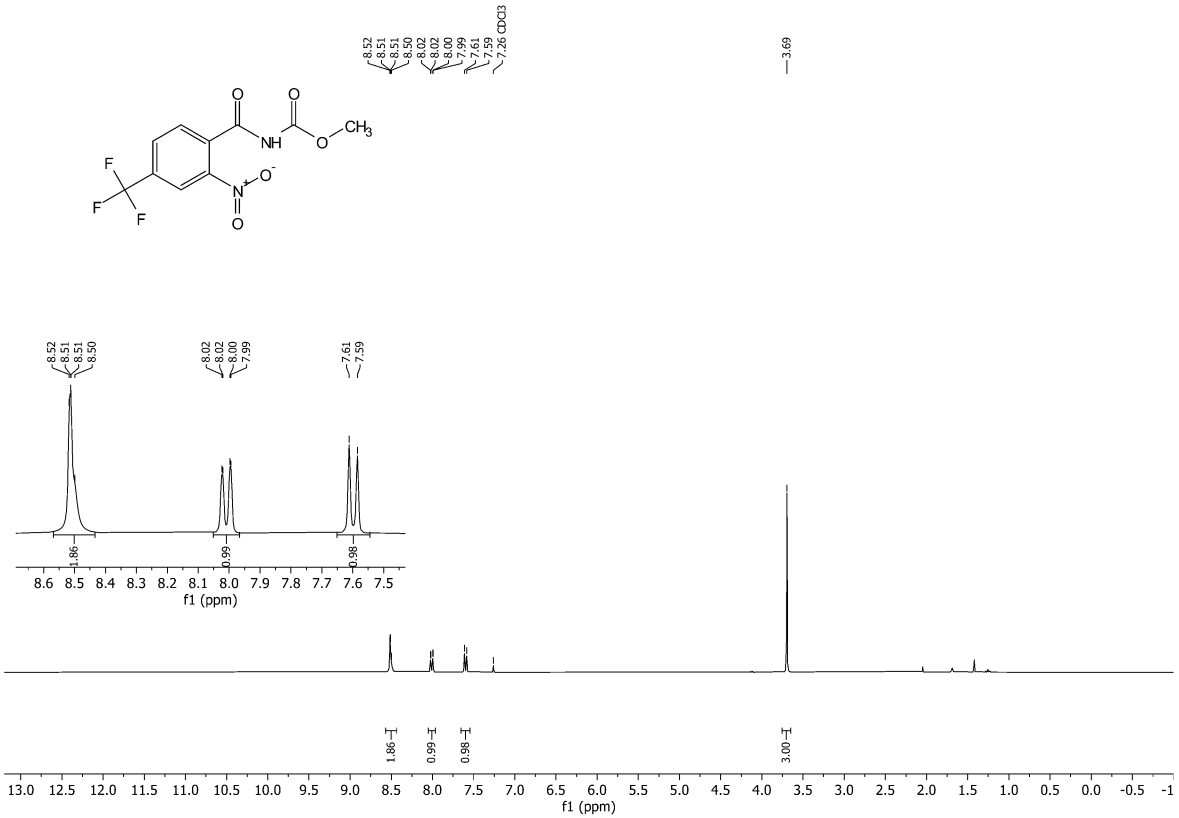



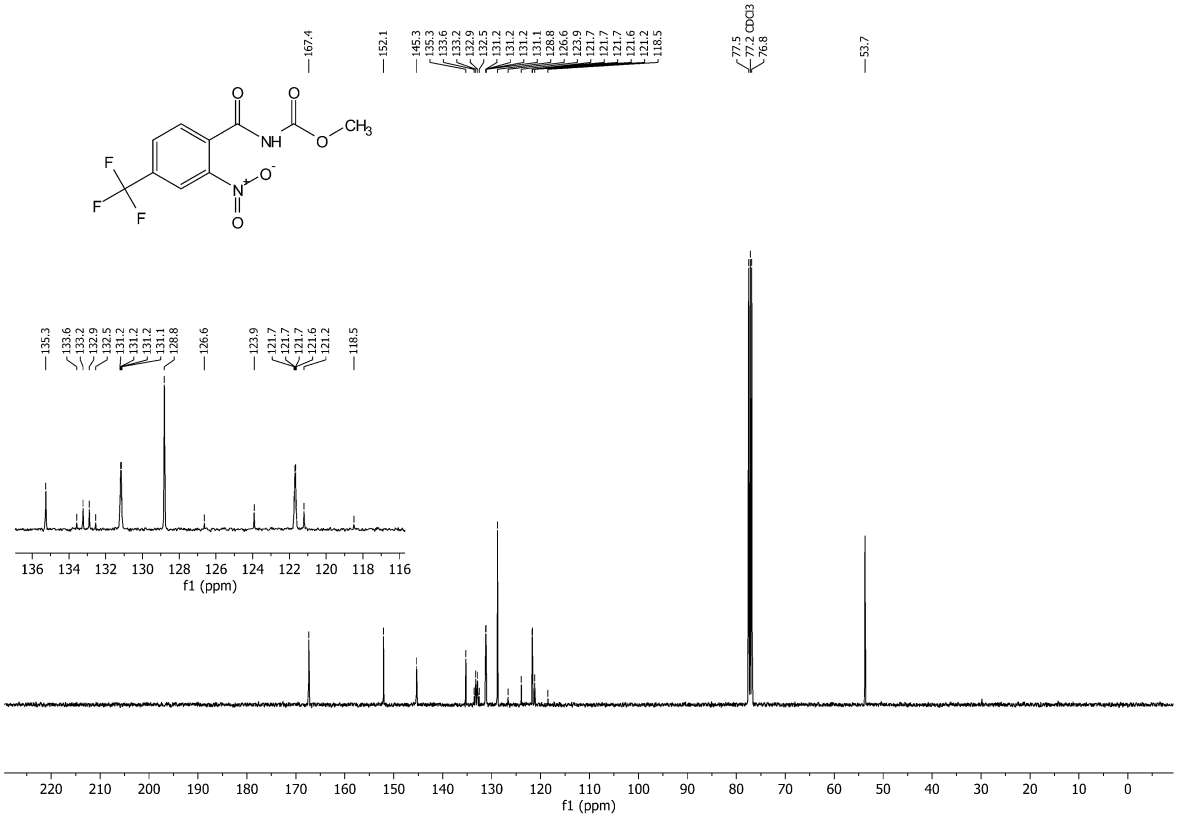
## Methyl (2-nitrobenzoyl)carbamate (5x)

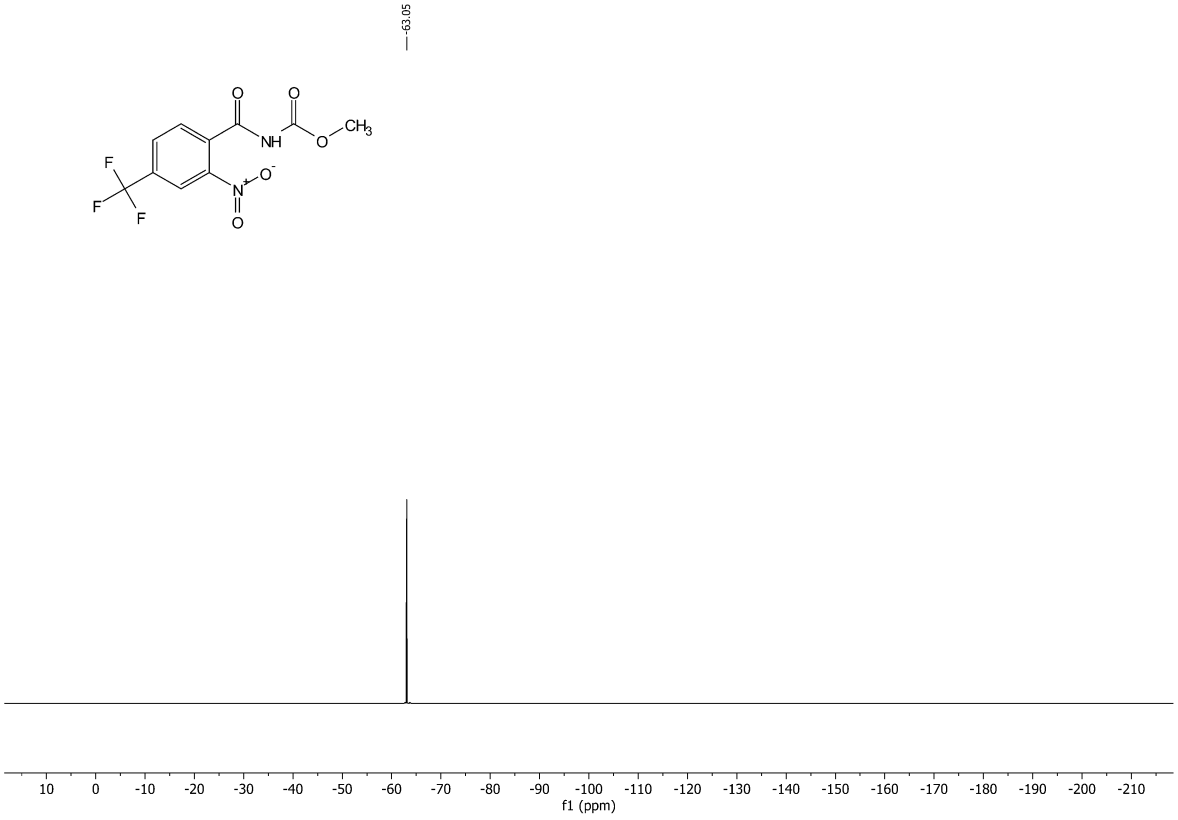


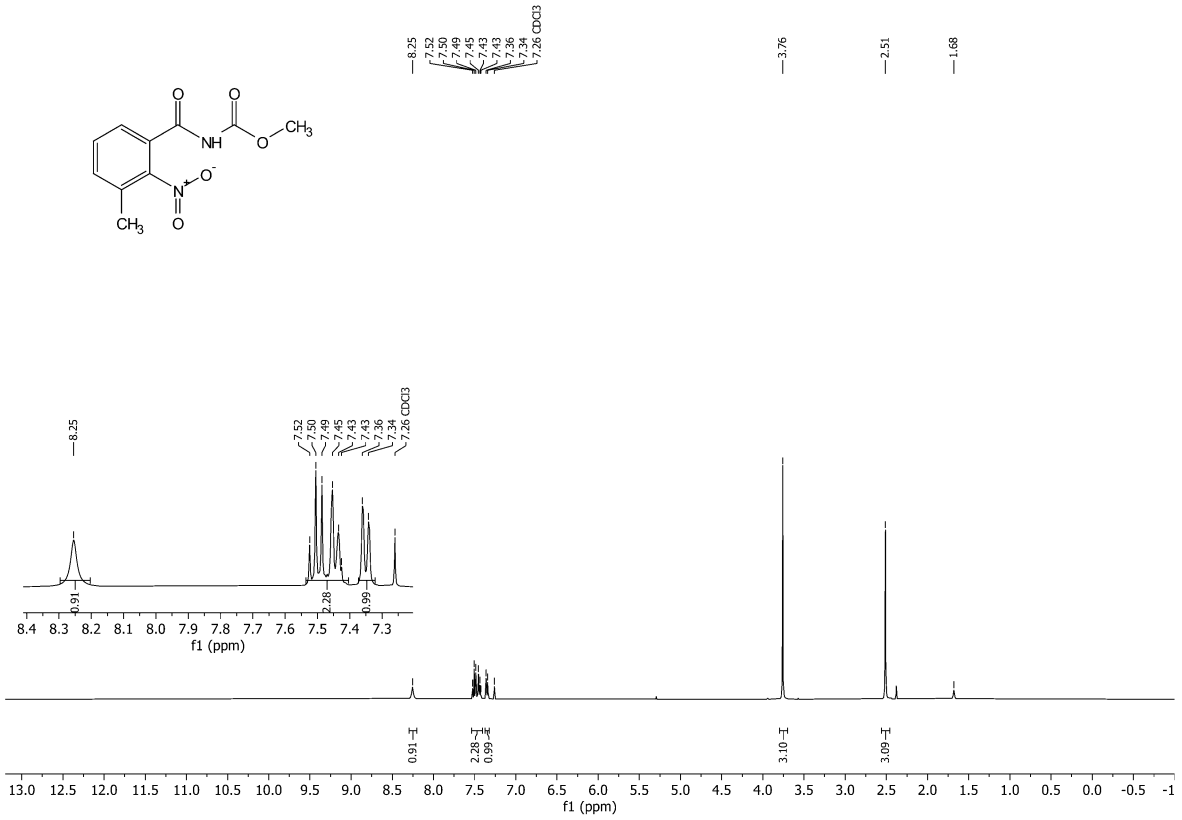
## Methyl (2-nitro-4-(trifluoromethyl)benzoyl)carbamate (5y)

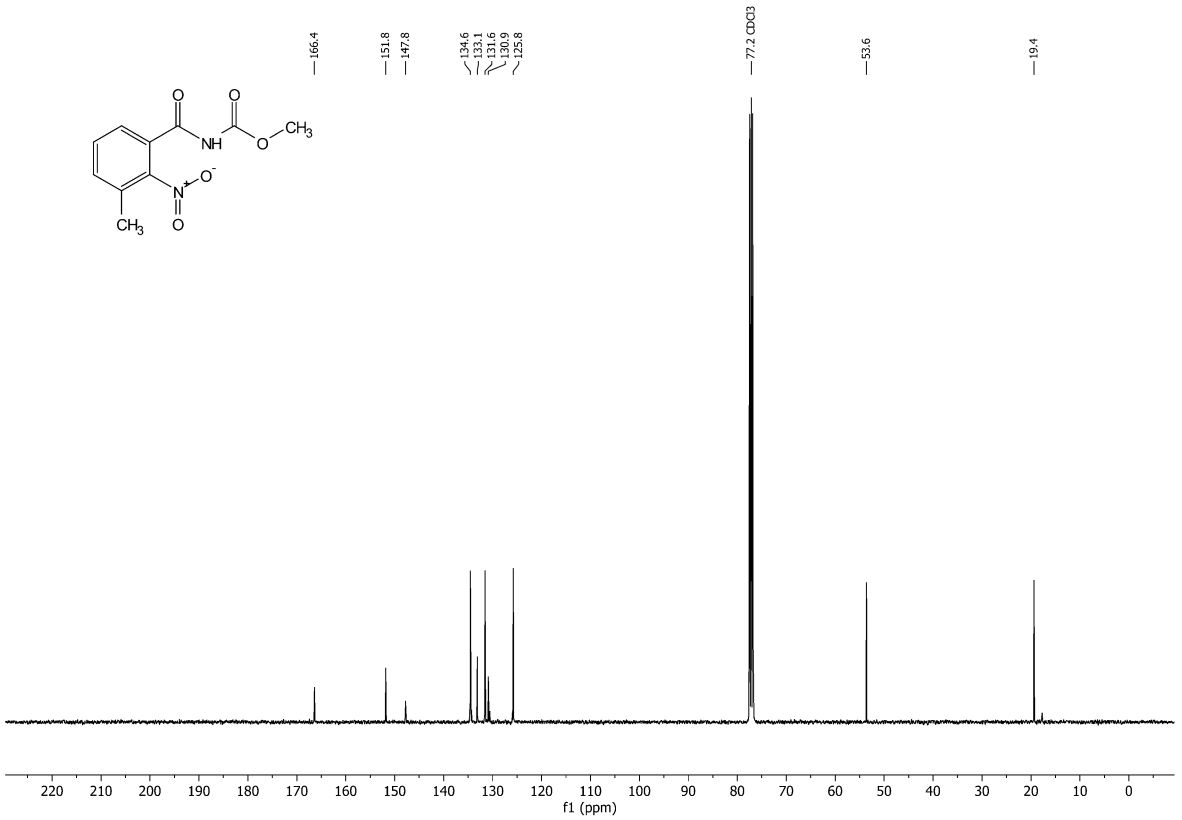


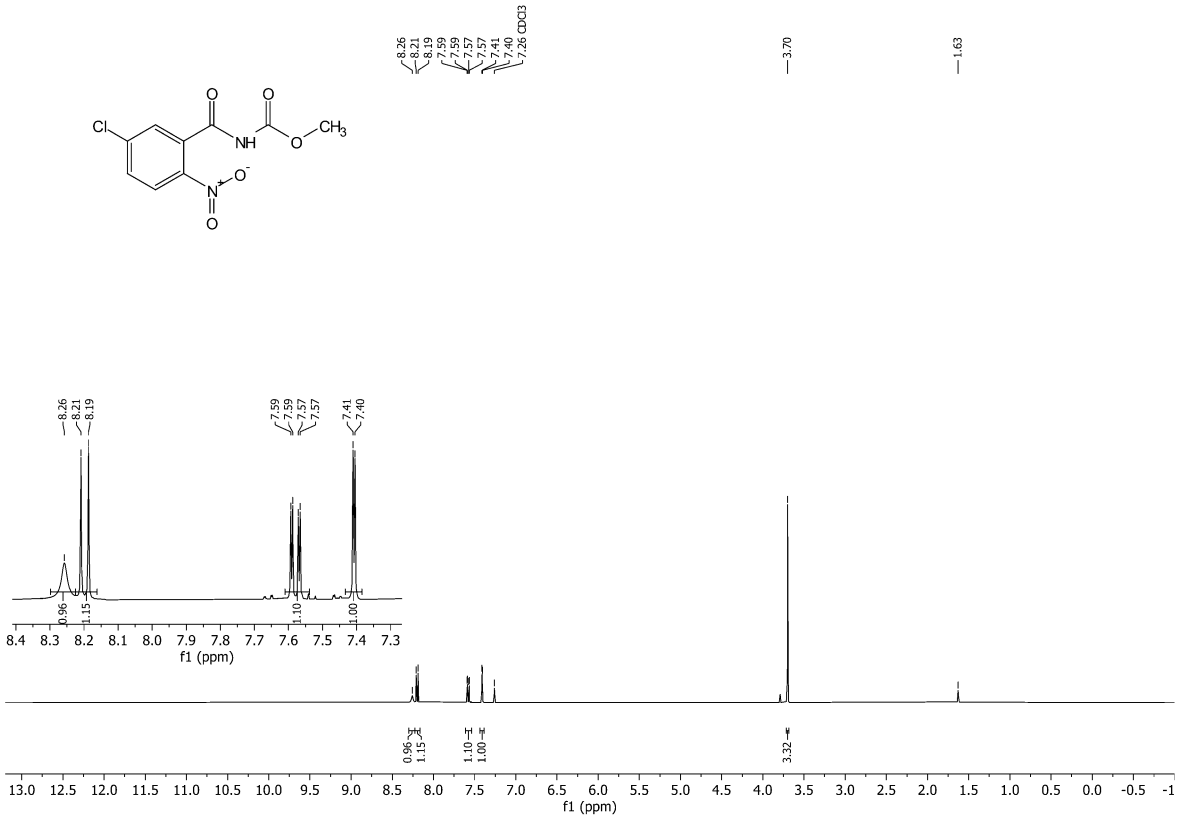

-63.05
O
O
NH
O
CH3
F
F
F
N+
O-
O
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

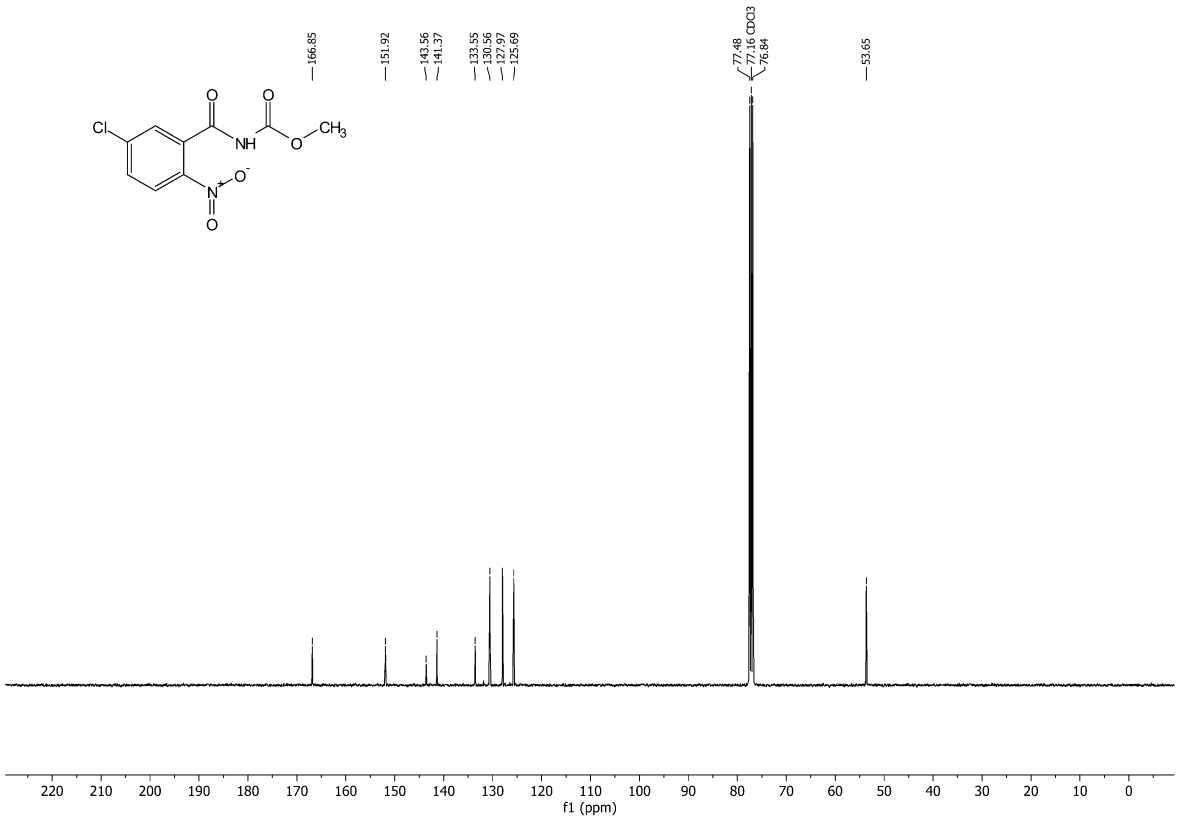
## Methyl (3-methyl-2-nitrobenzoyl)carbamate (5z)



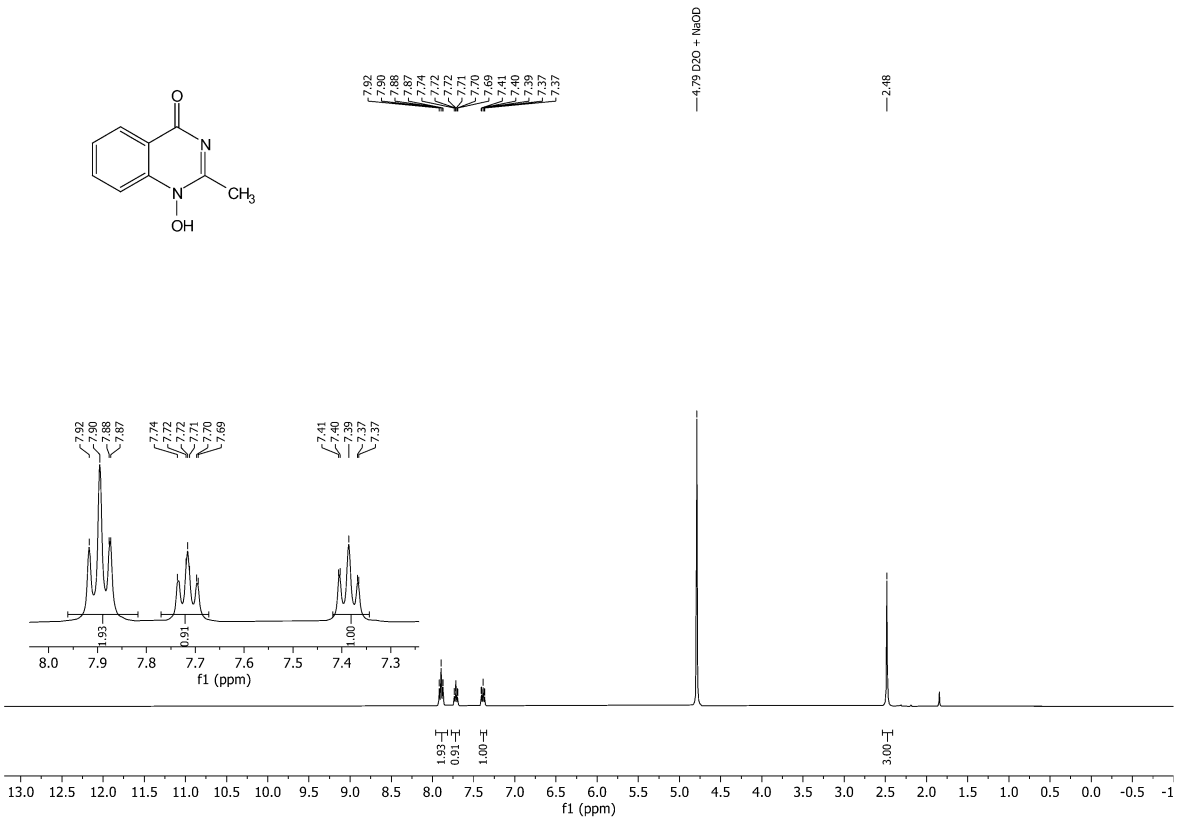



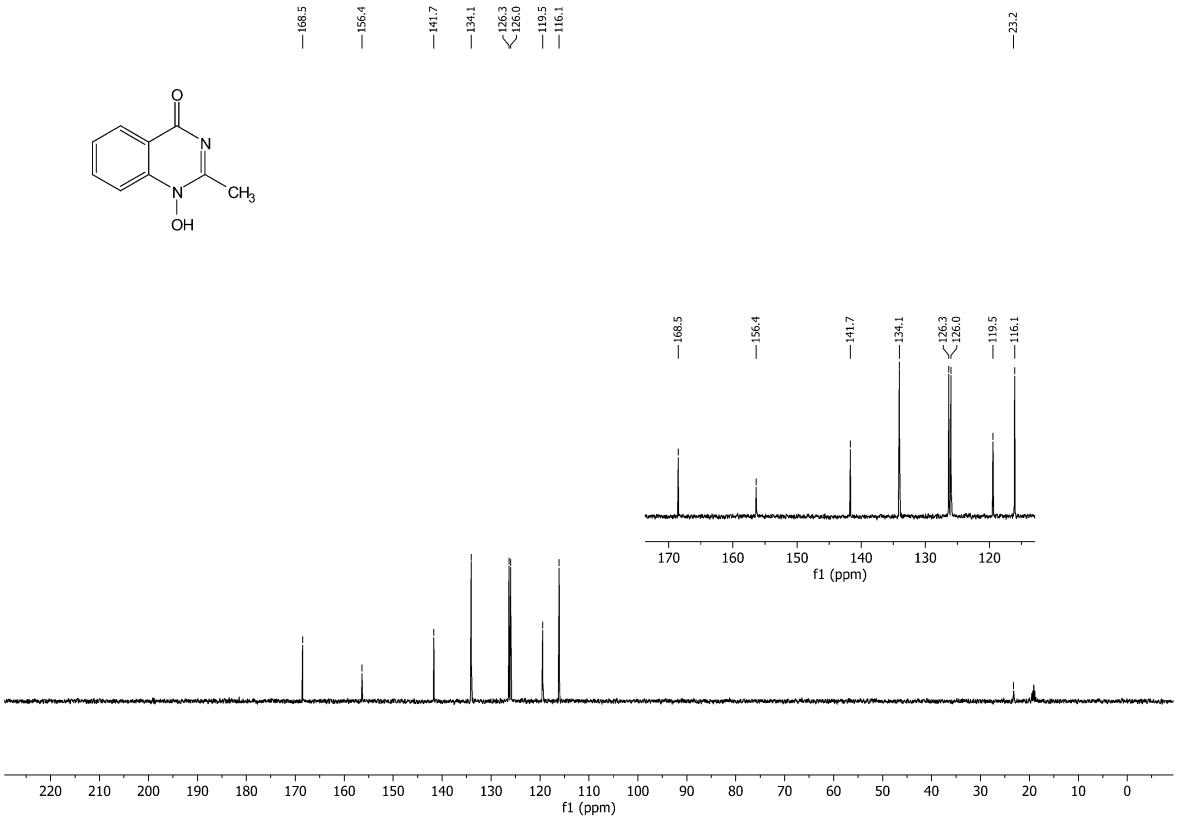
## Methyl (5-chloro-2-nitrobenzoyl)carbamate (5aa)



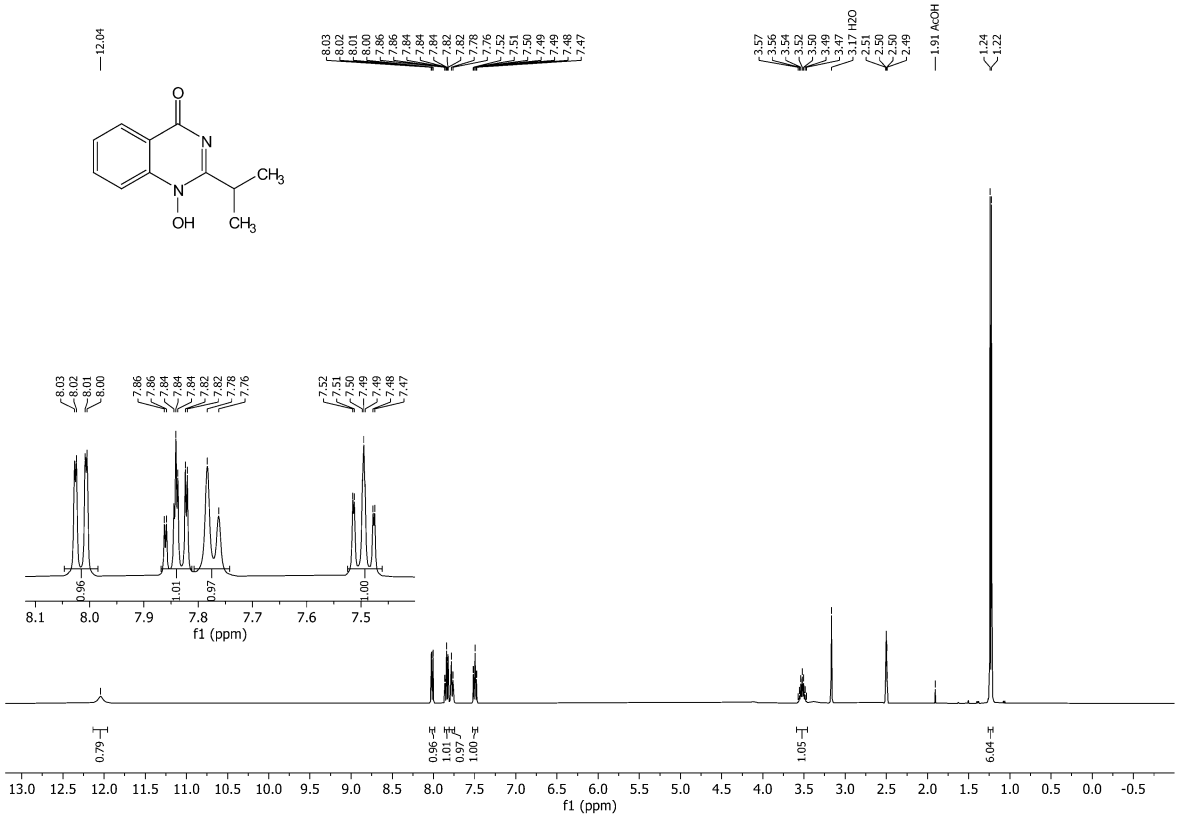



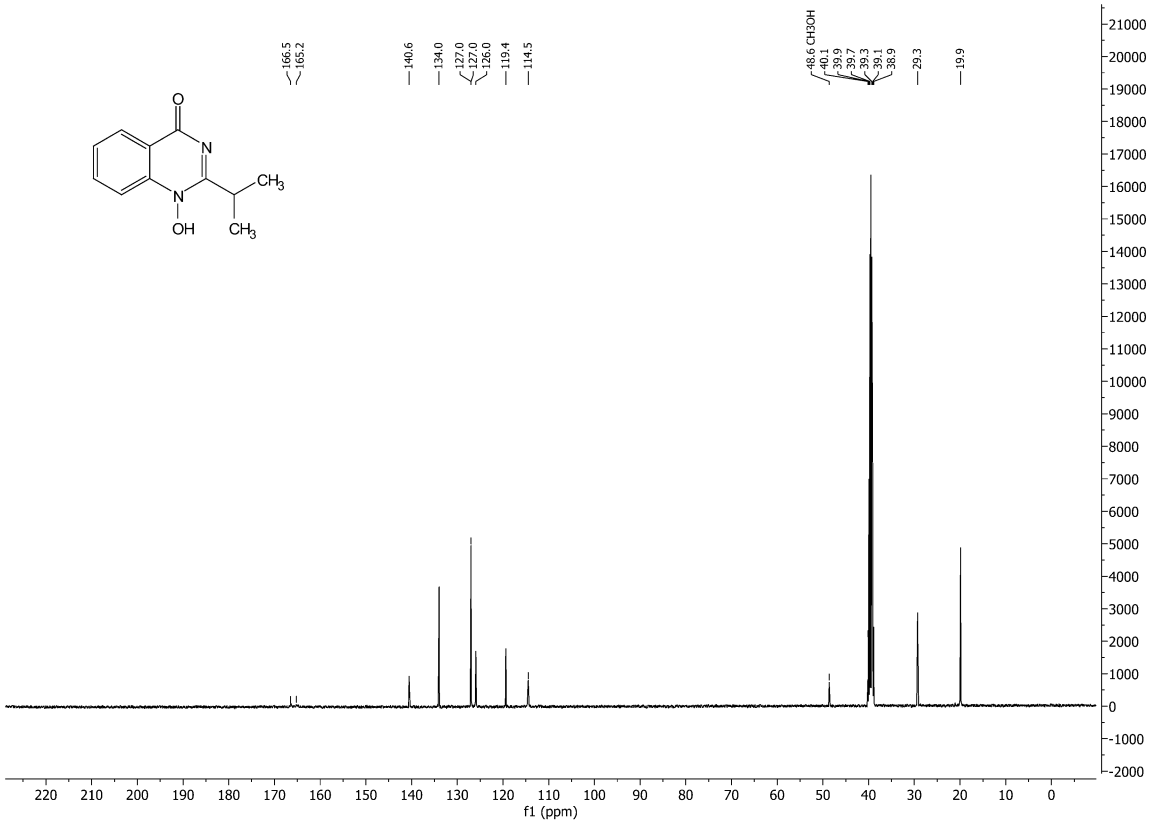
## 1*H*-1-Hydroxy-2-methylquinazolin-4-one (6a)



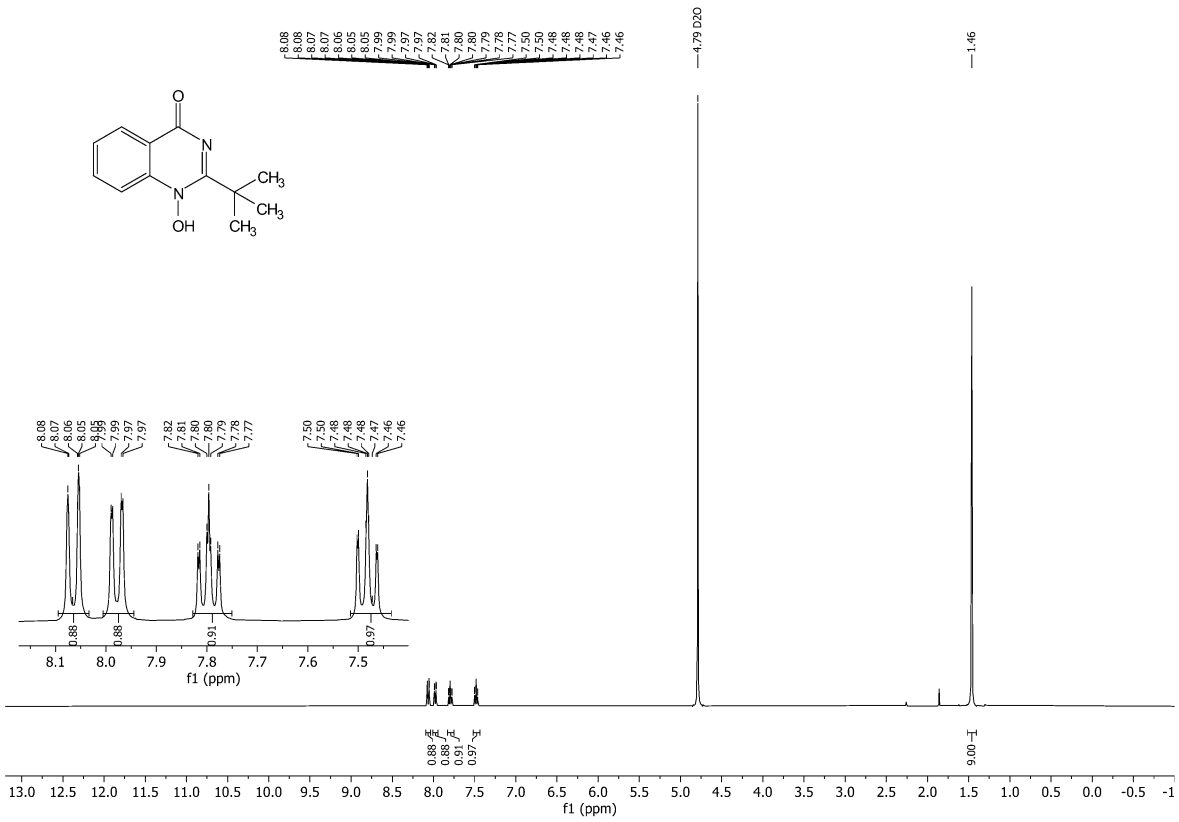



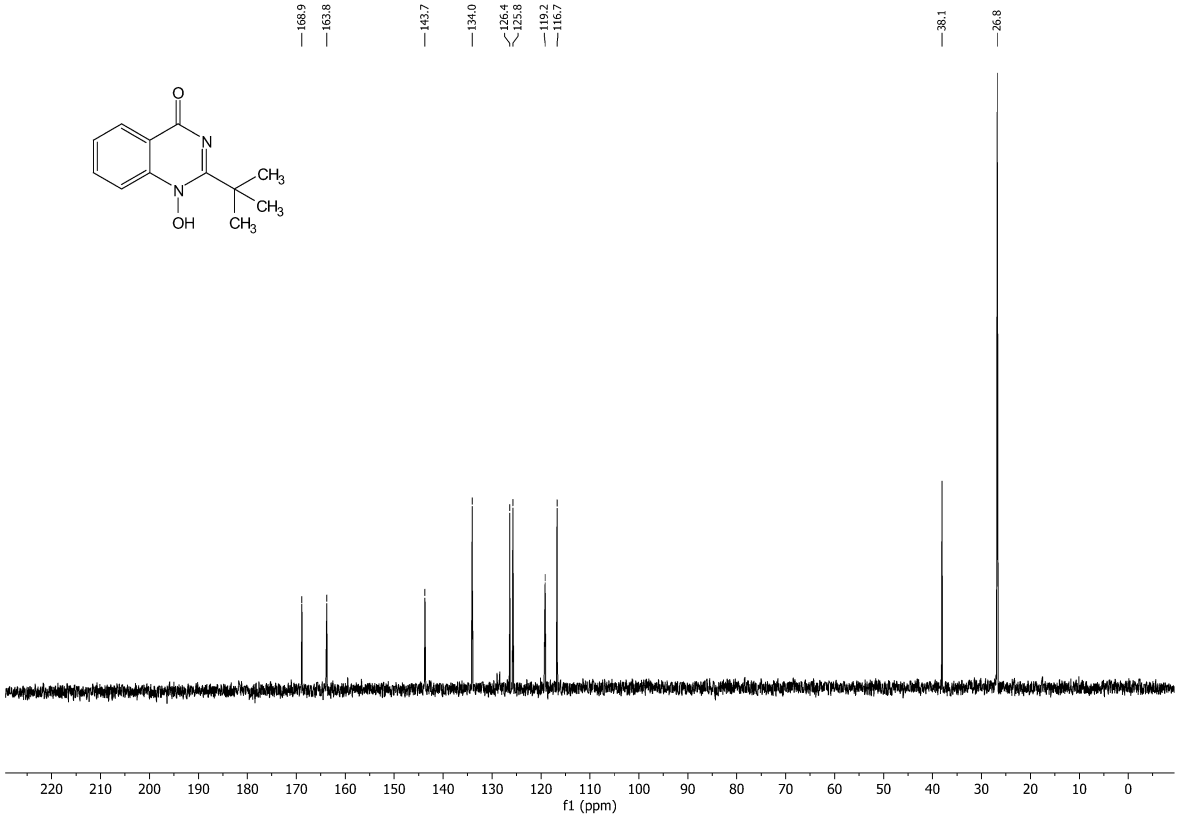
## 1*H*-1-Hydroxy-2-isopropylquinazolin-4-one (6b)



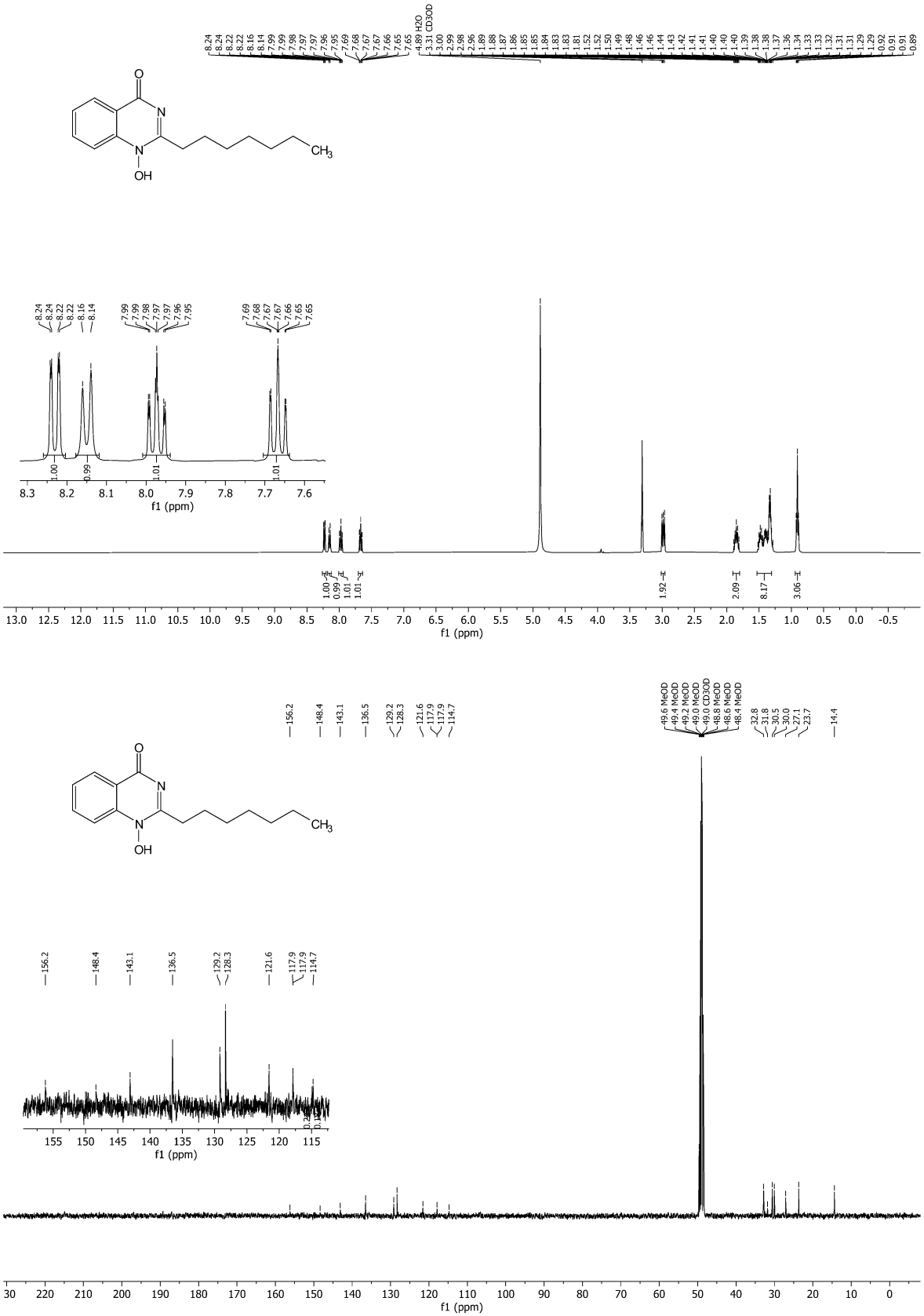



## 1*H*-2-(*tert*-Butyl)-1-hydroxyquinazolin-4-one (6c)

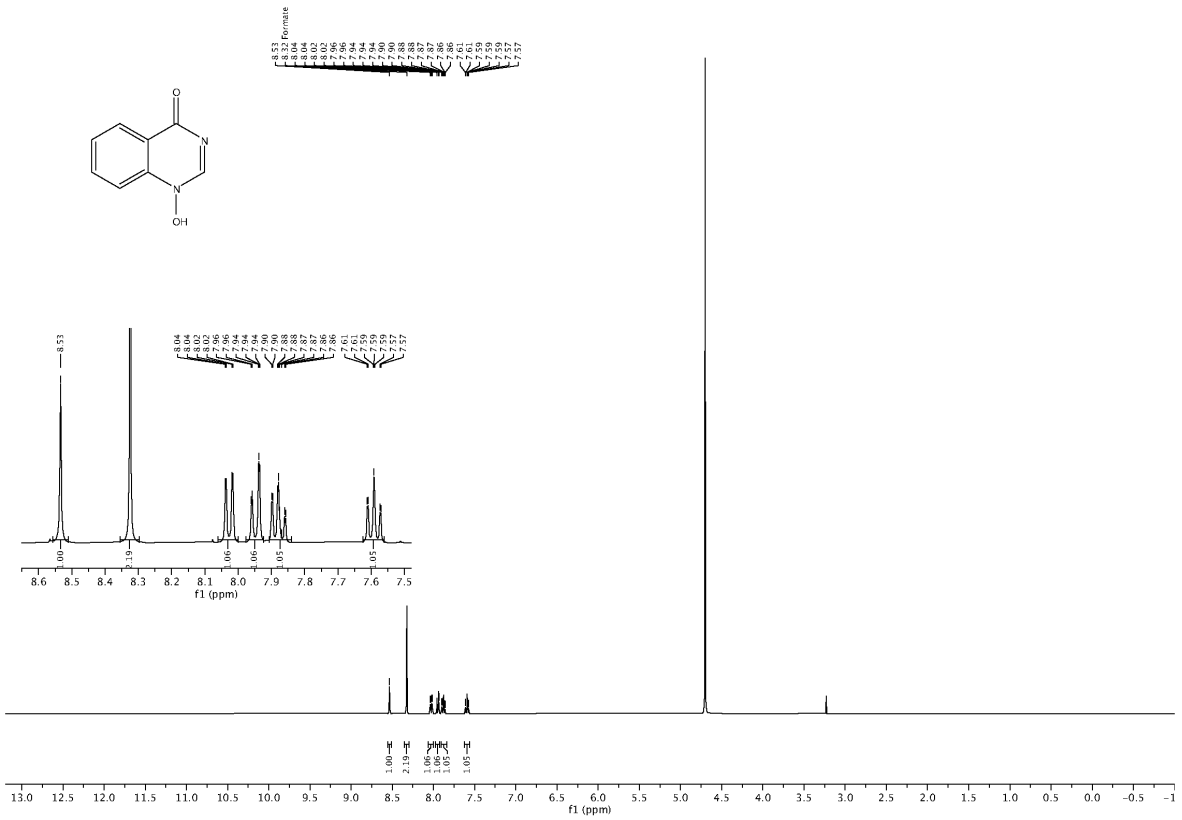


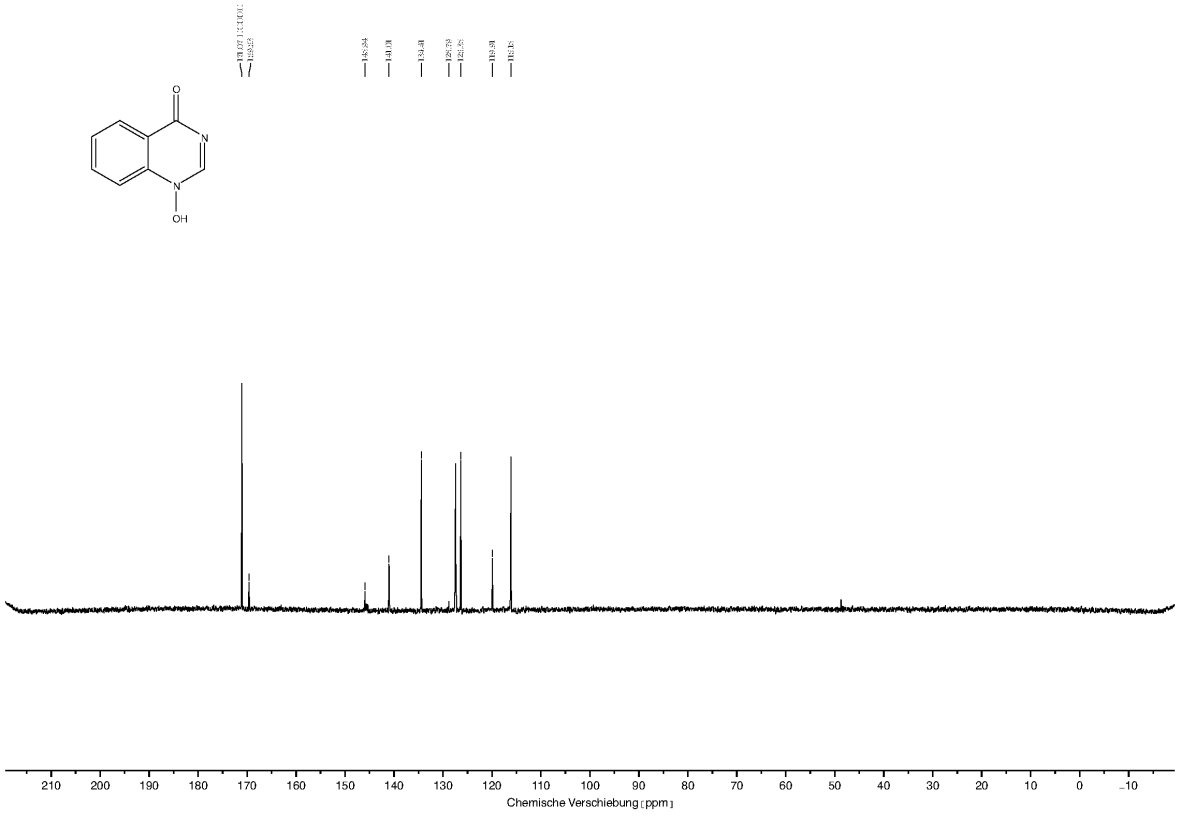



## 1*H*-2-Heptyl-1-hydroxyquinazolin-4-one (6d)

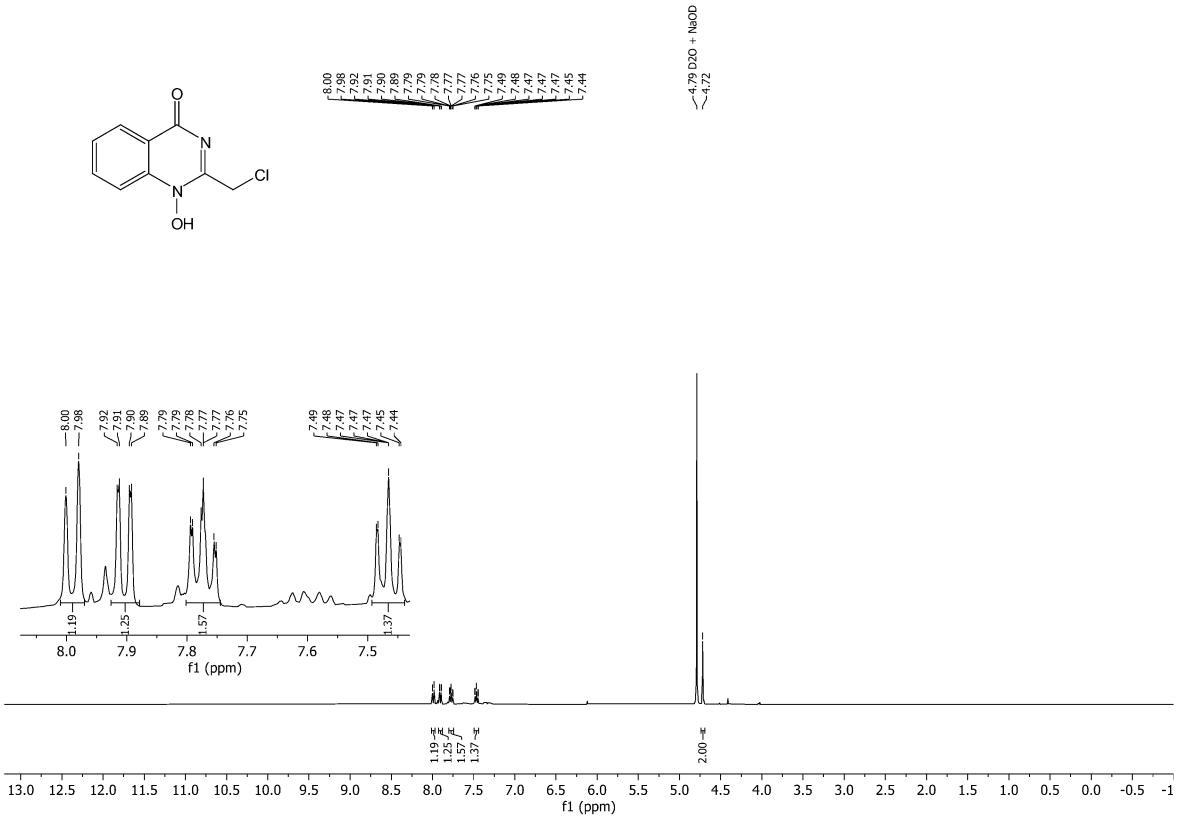


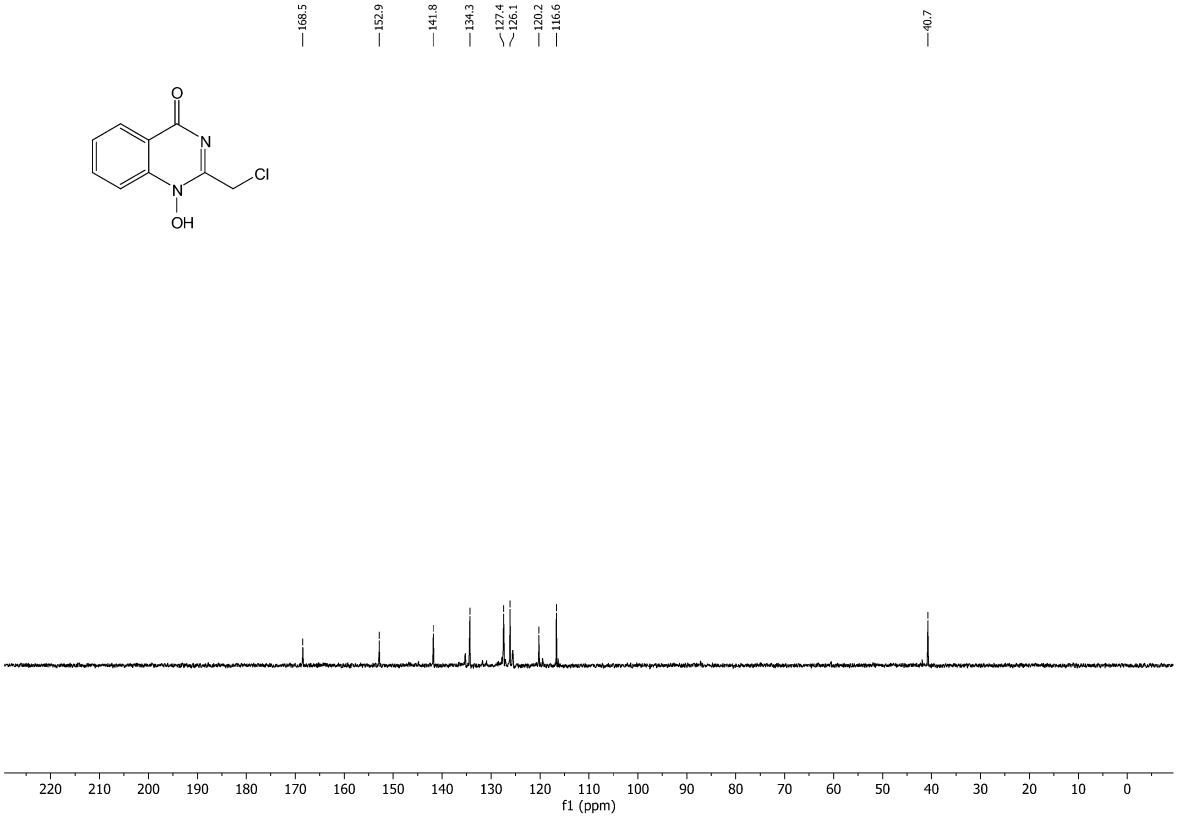
## 1*H*-1-Hydroxyquinazolin-4-one (6e)



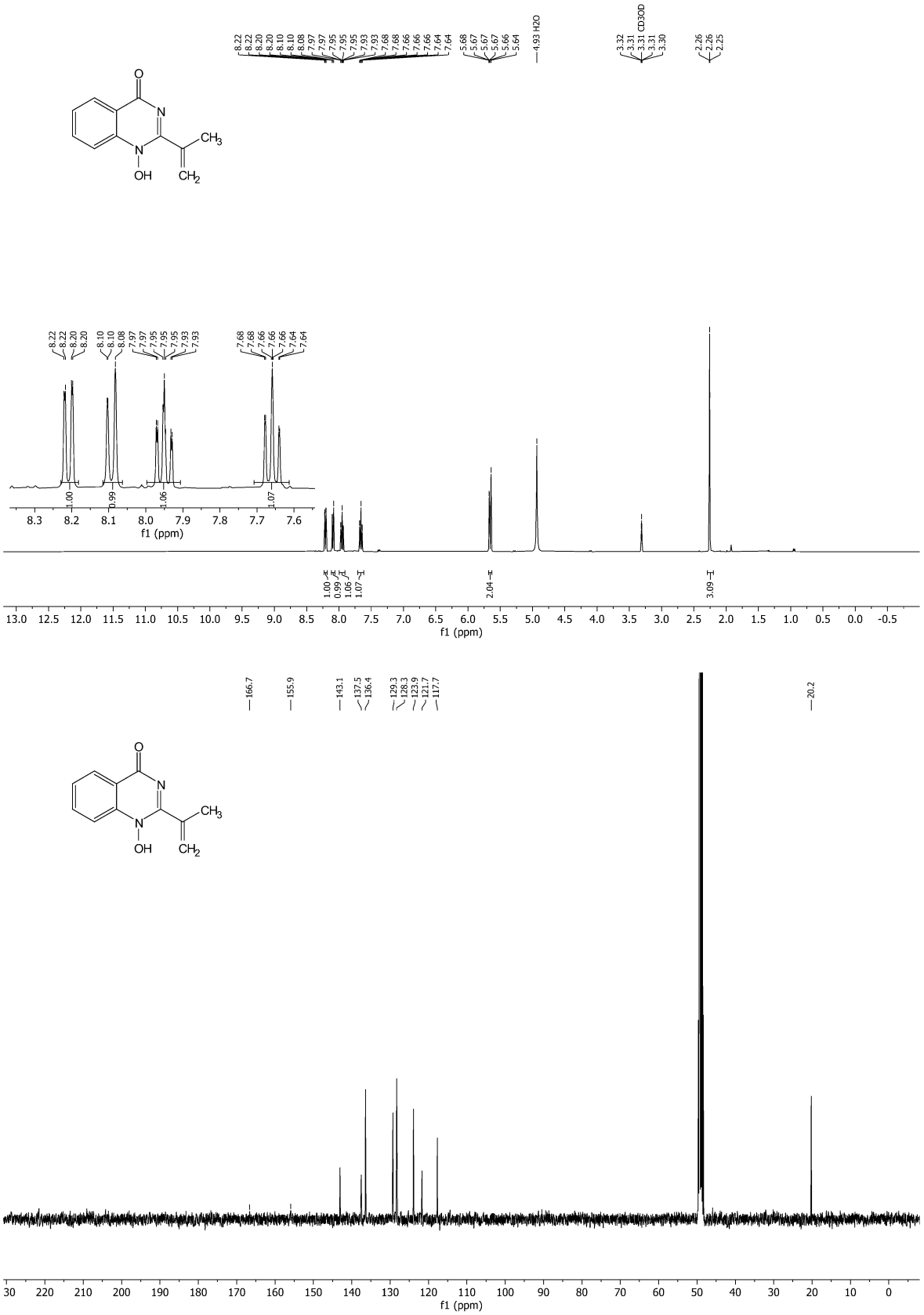



## 1*H*-2-(Chloromethyl)-1-hydroxyquinazolin-4-one (6f)

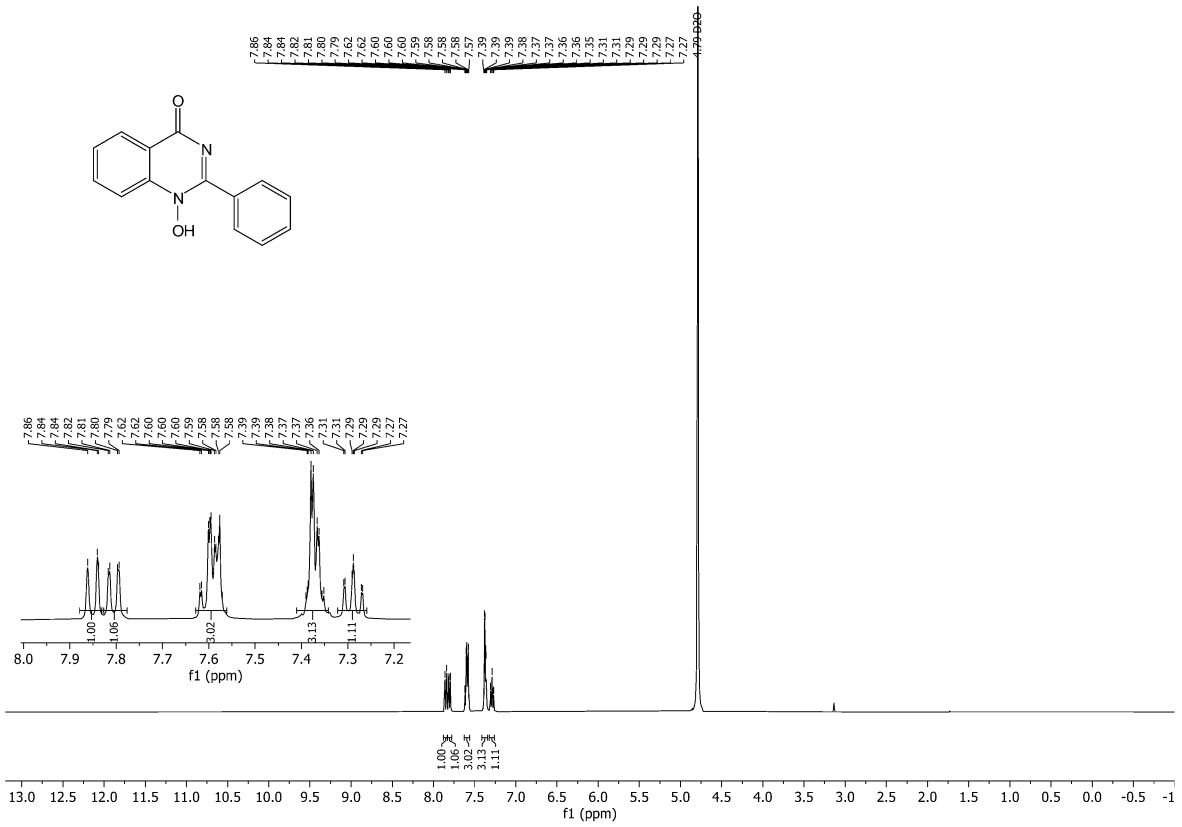


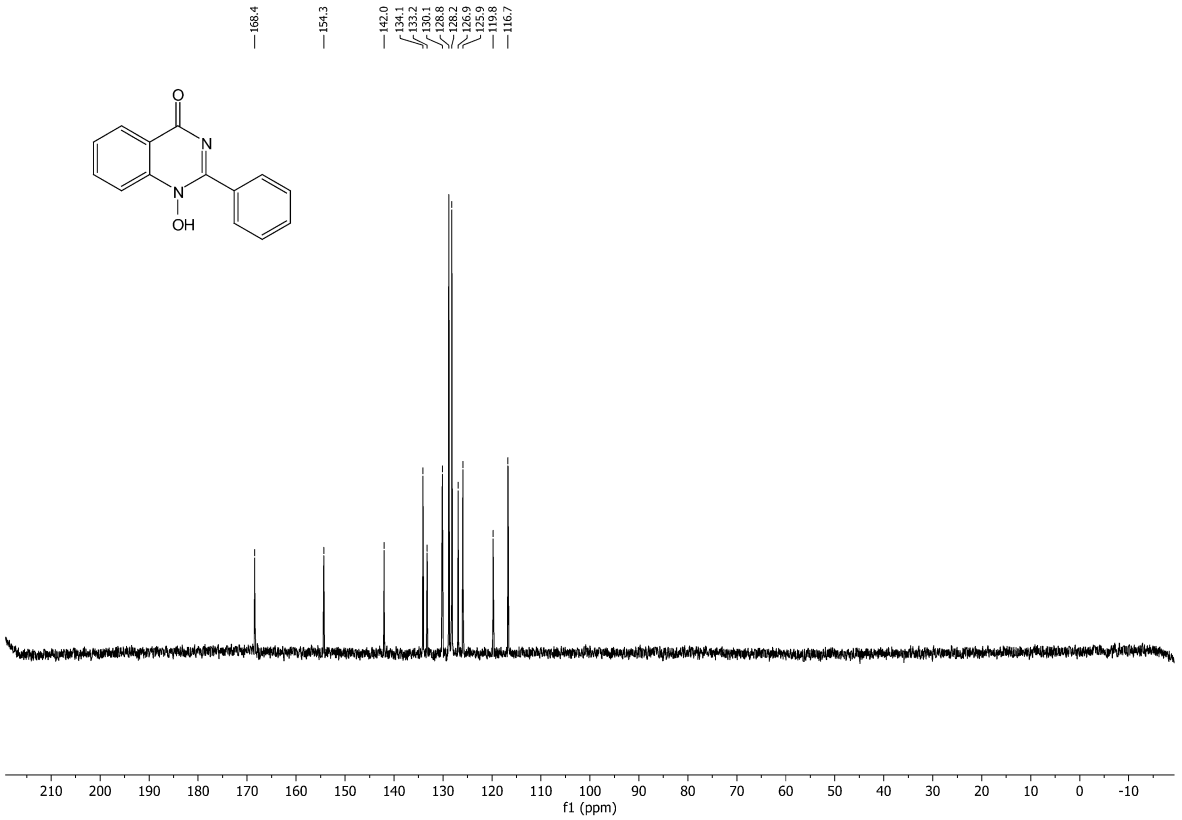



**1*H*-1-Hydroxy-2-(prop-1-en-2-yl)quinazolin-4-one (6g)**

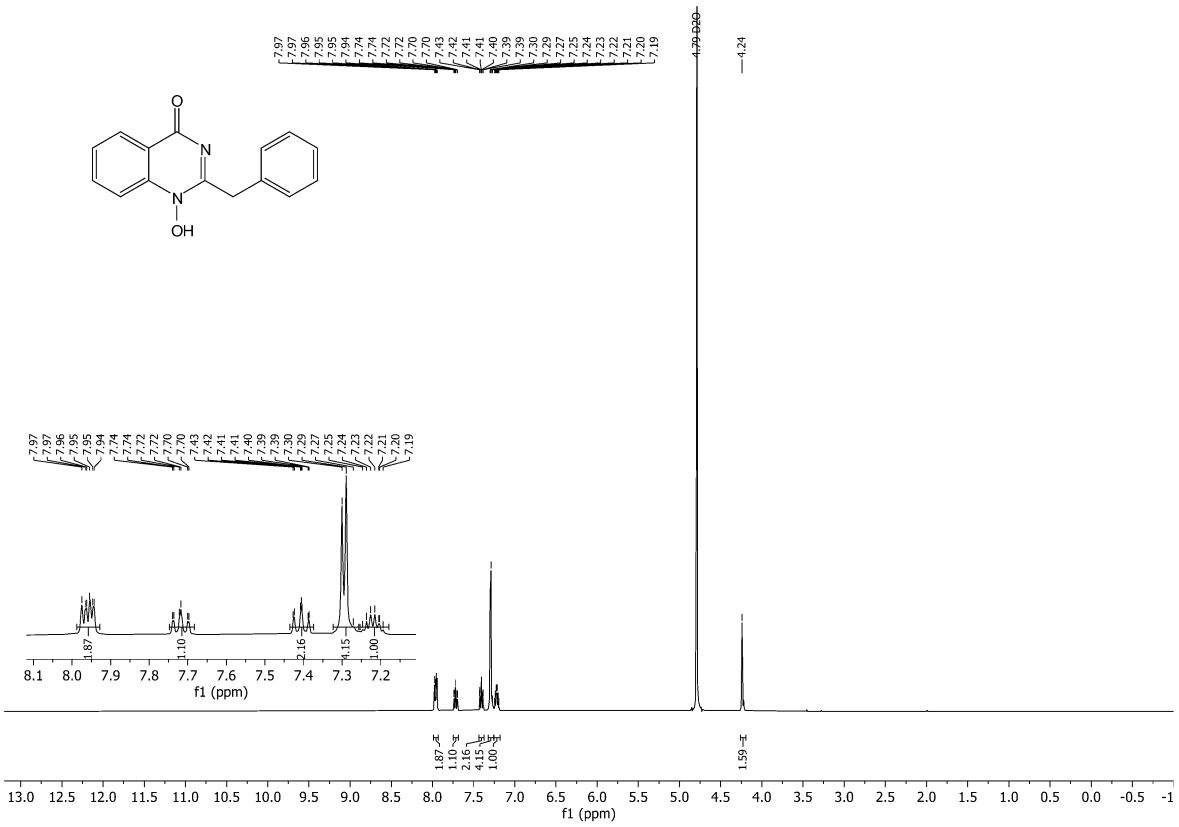


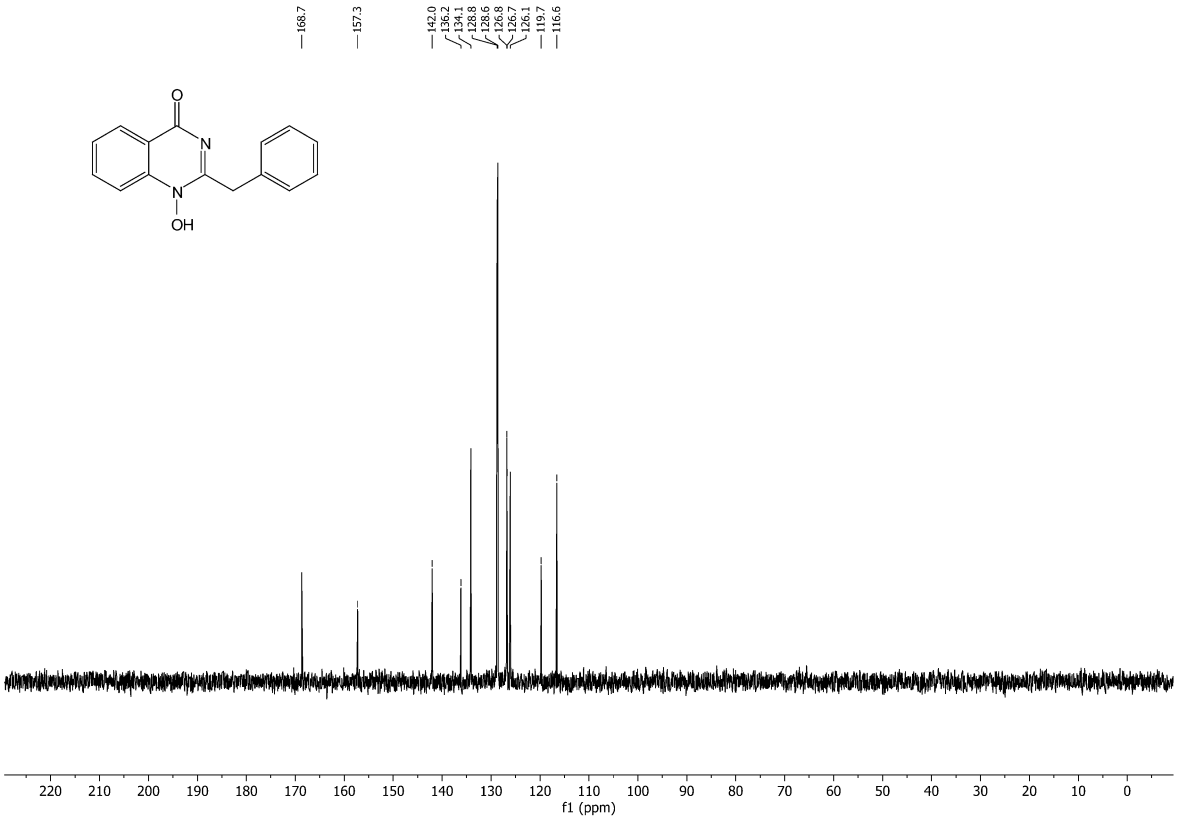
## 1*H*-1-Hydroxy-2-phenylquinazolin-4-one (6h)



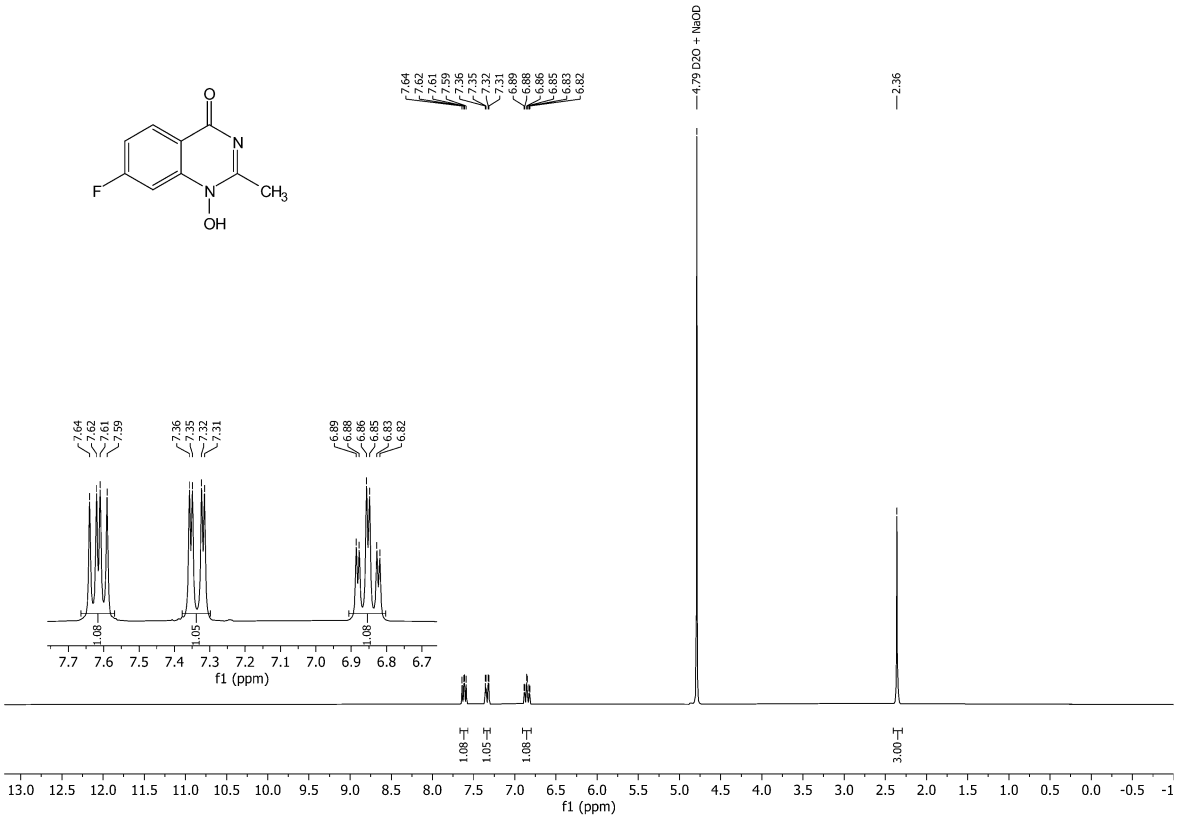



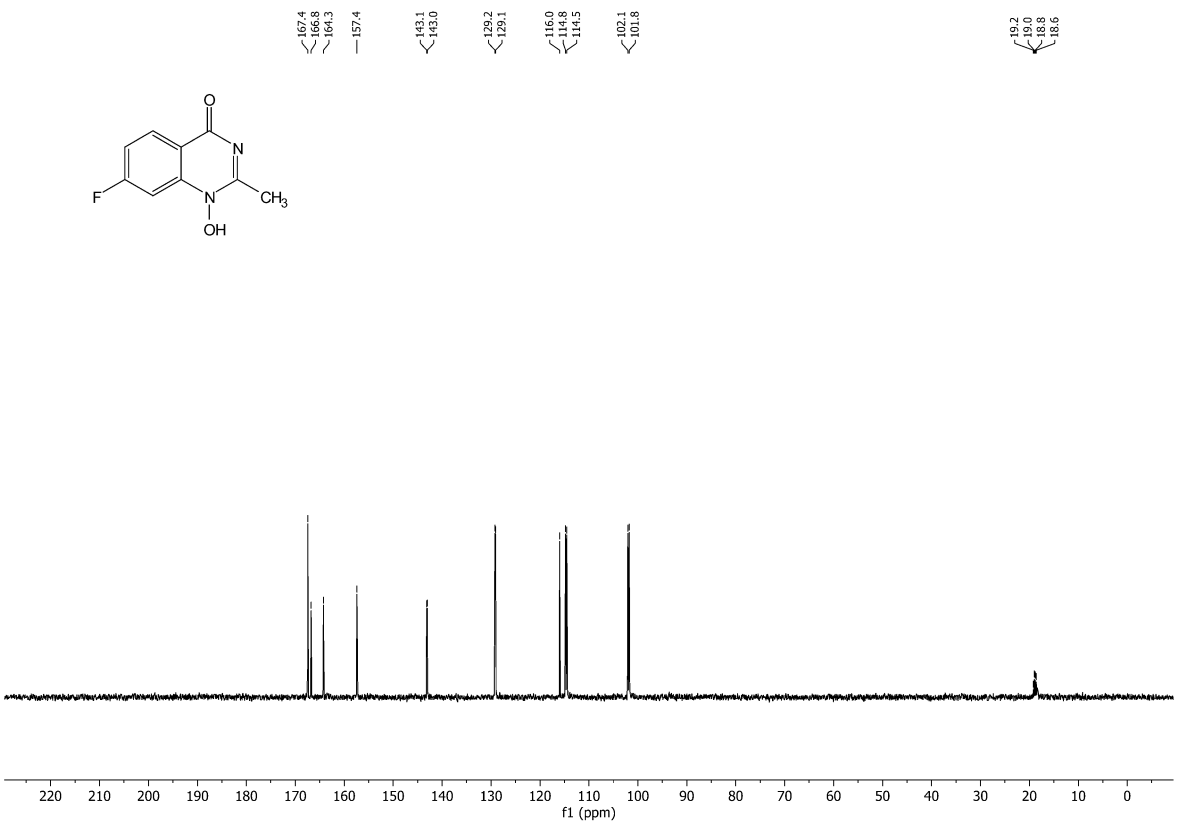
## 1*H*-2-Benzyl-1-hydroxyquinazolin-4-one (6i)



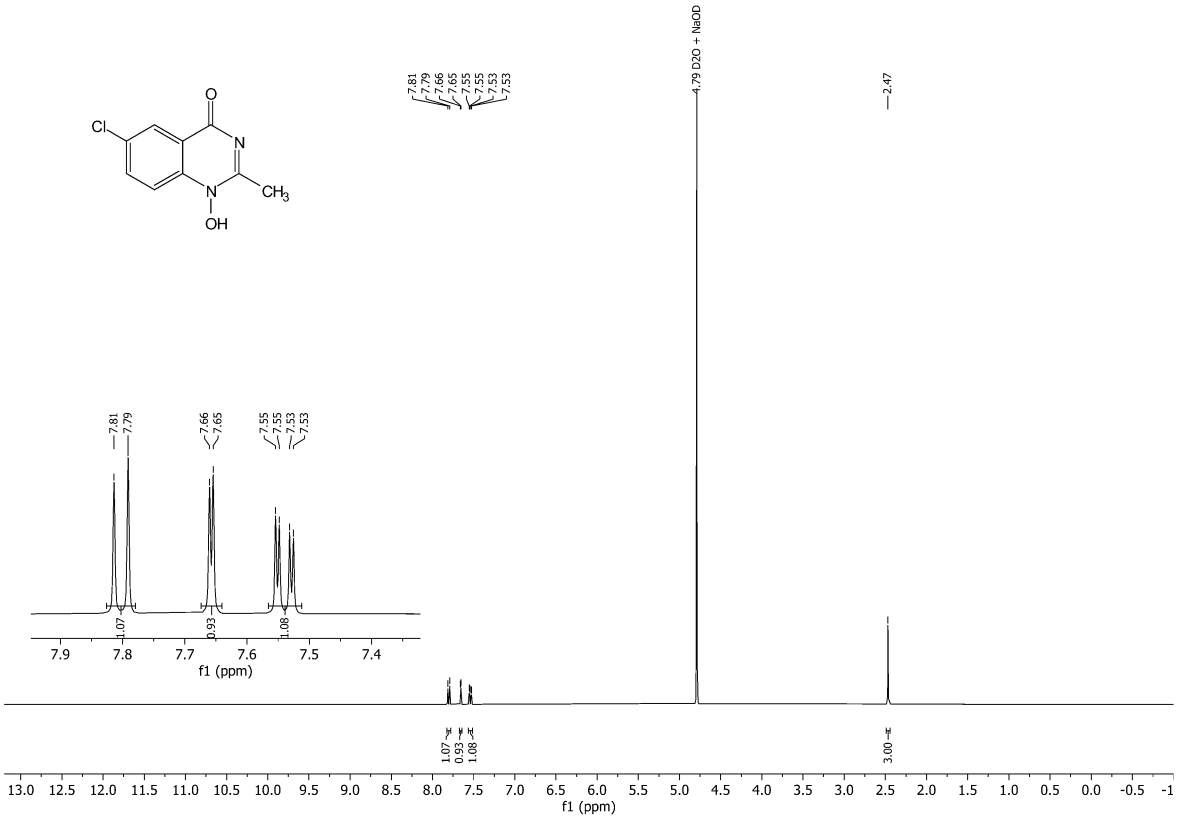
## 1*H*-7-Fluoro-1-hydroxy-2-methylquinazolin-4-one (6j)

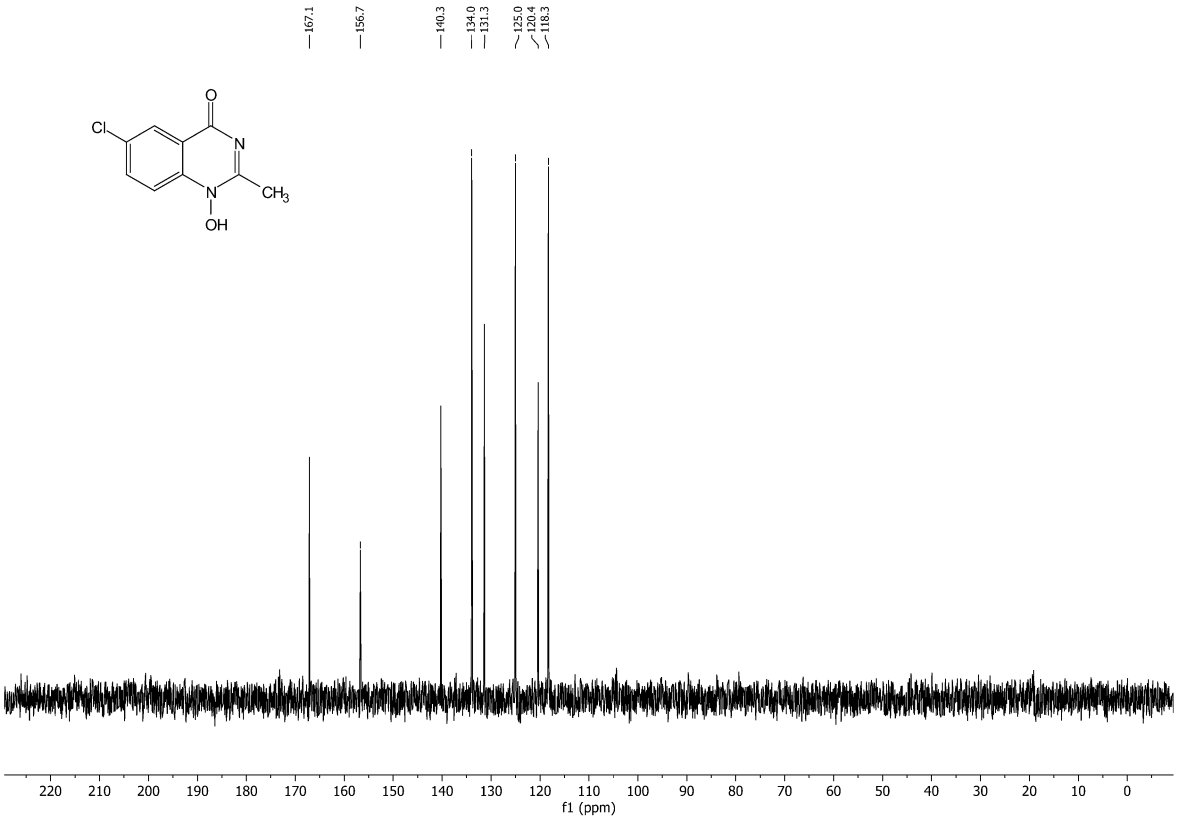


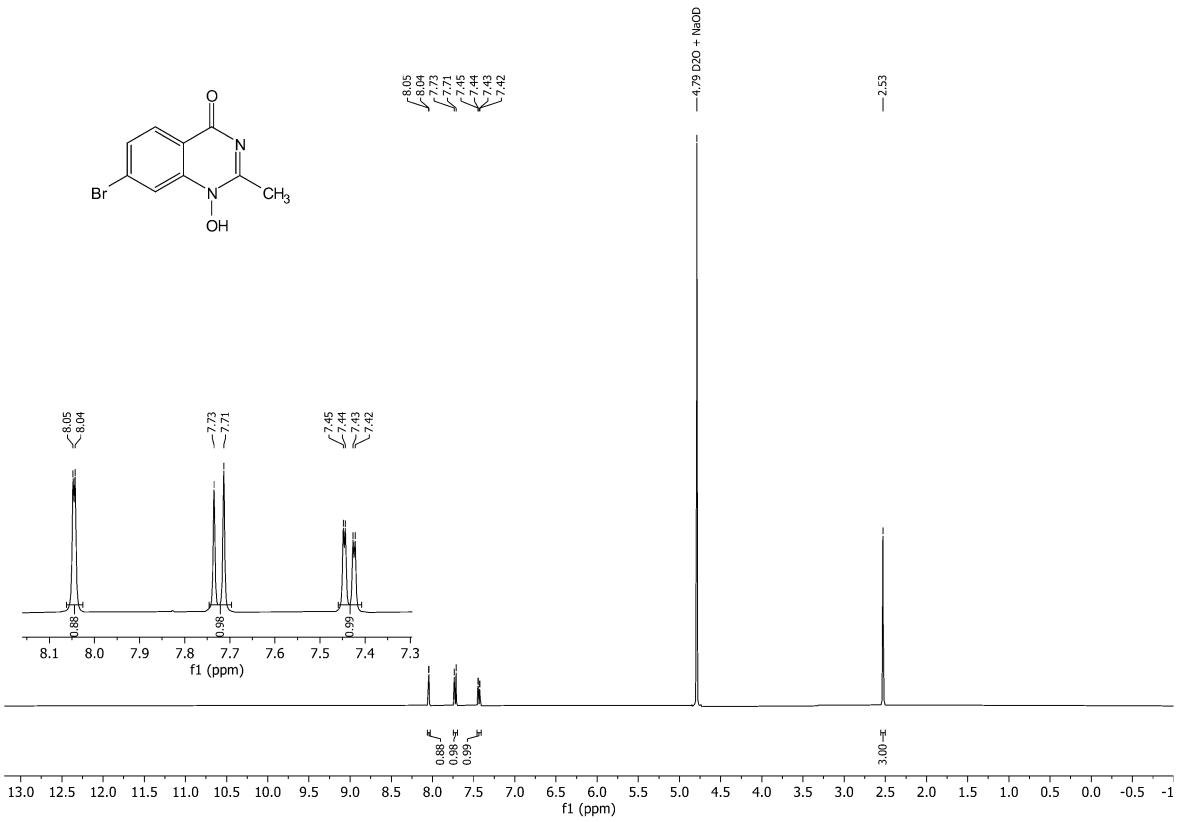

-102.92
-102.94
-102.95
-102.96
-102.97
-102.98
-102.99
-103.01
O
N
F
N
CH3
OH
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

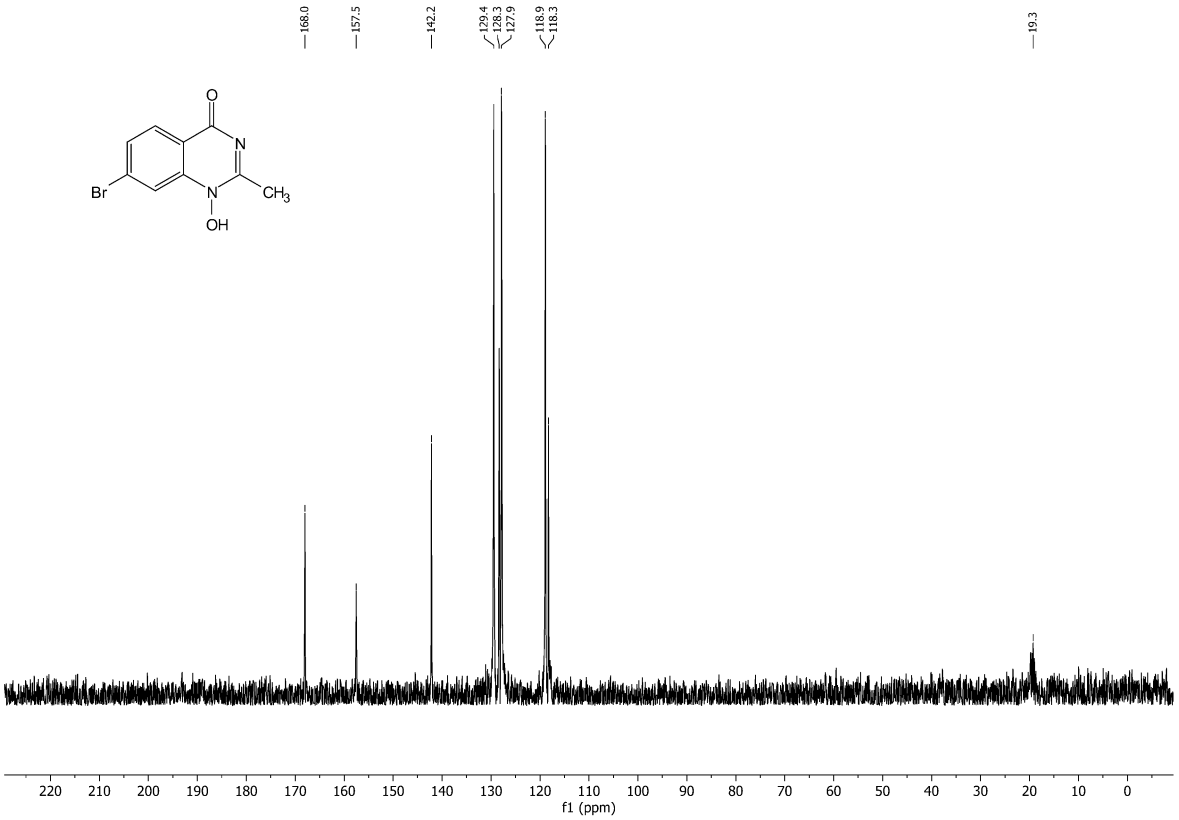
## 1*H*-6-Chloro-1-hydroxy-2-methylquinazolin-4-one (6k)



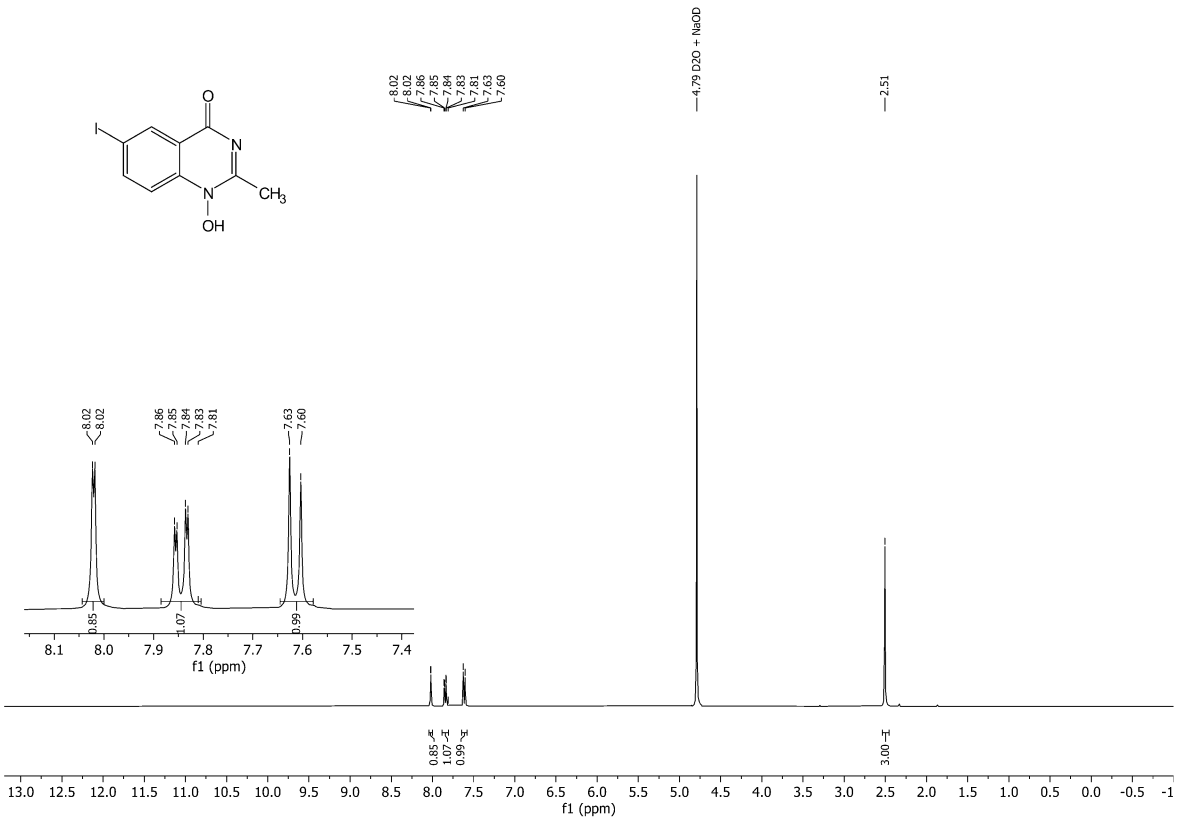



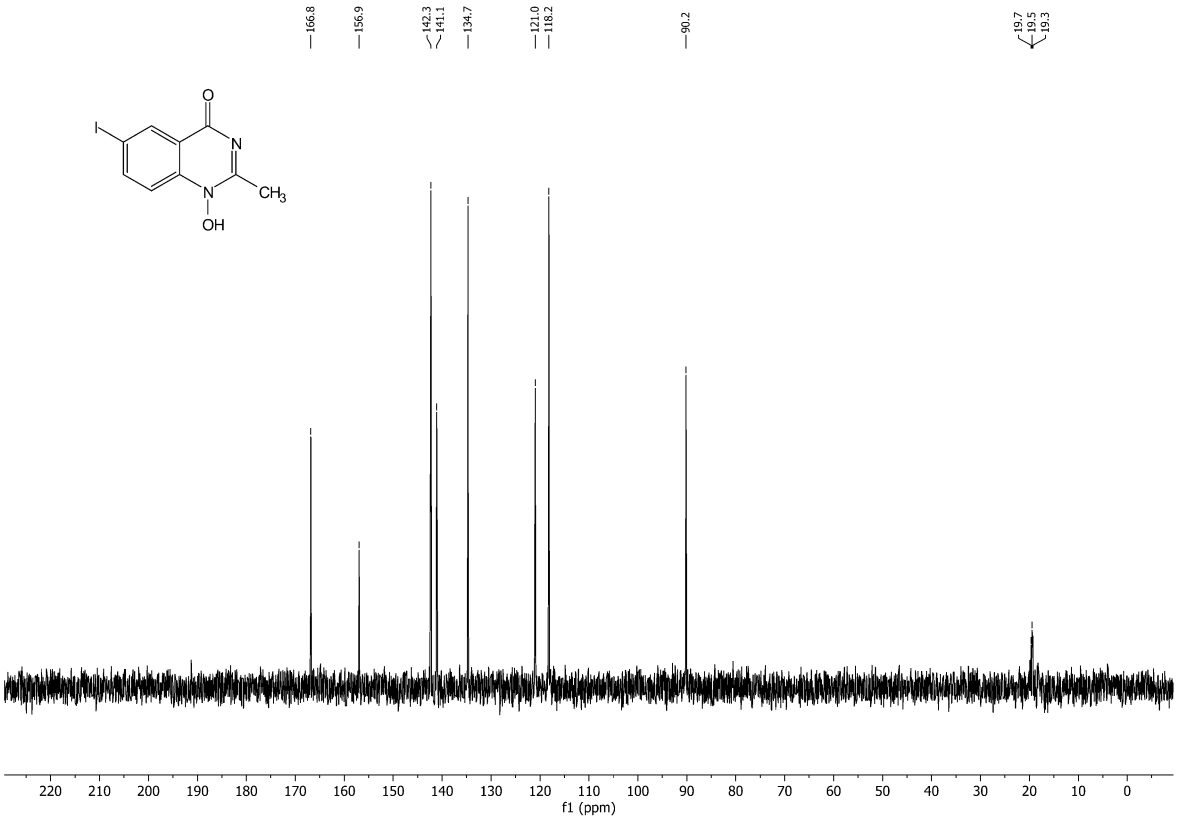
**1*H*-7-Bromo-1-hydroxy-2-methylquinazolin-4-one (6l)**



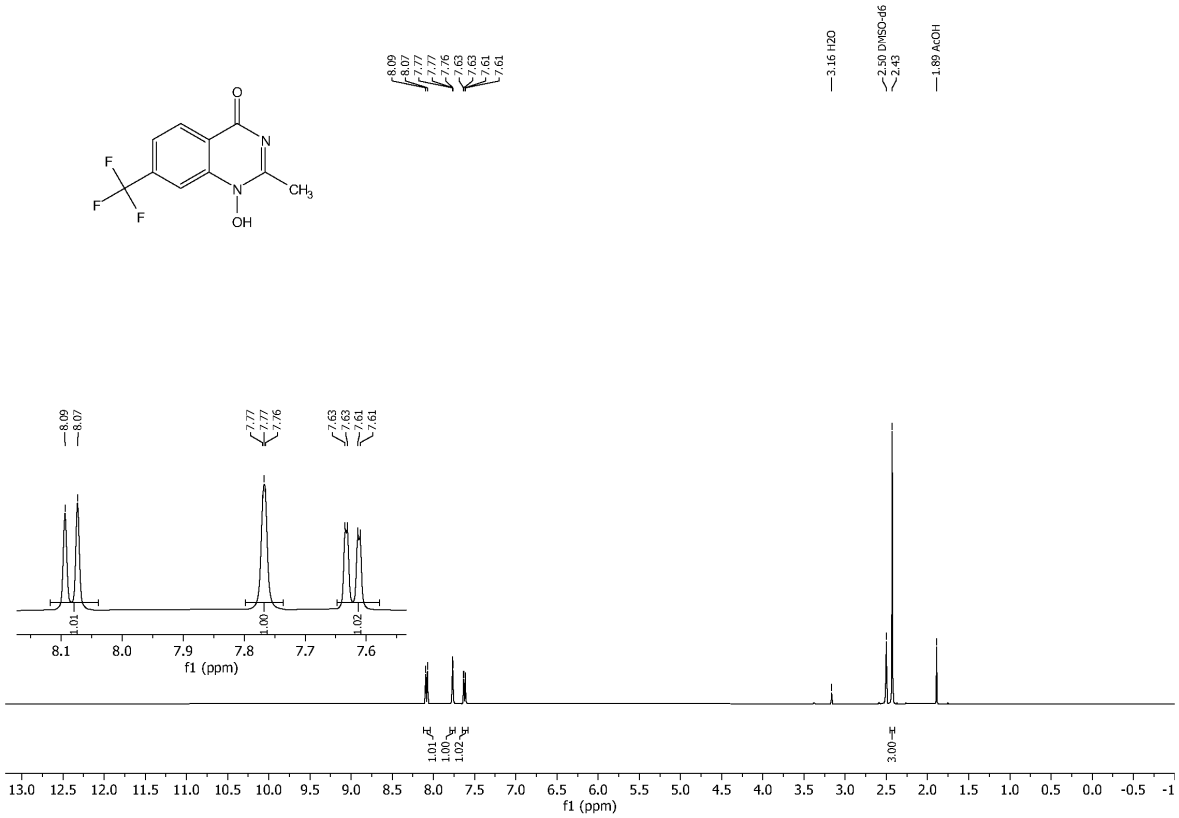



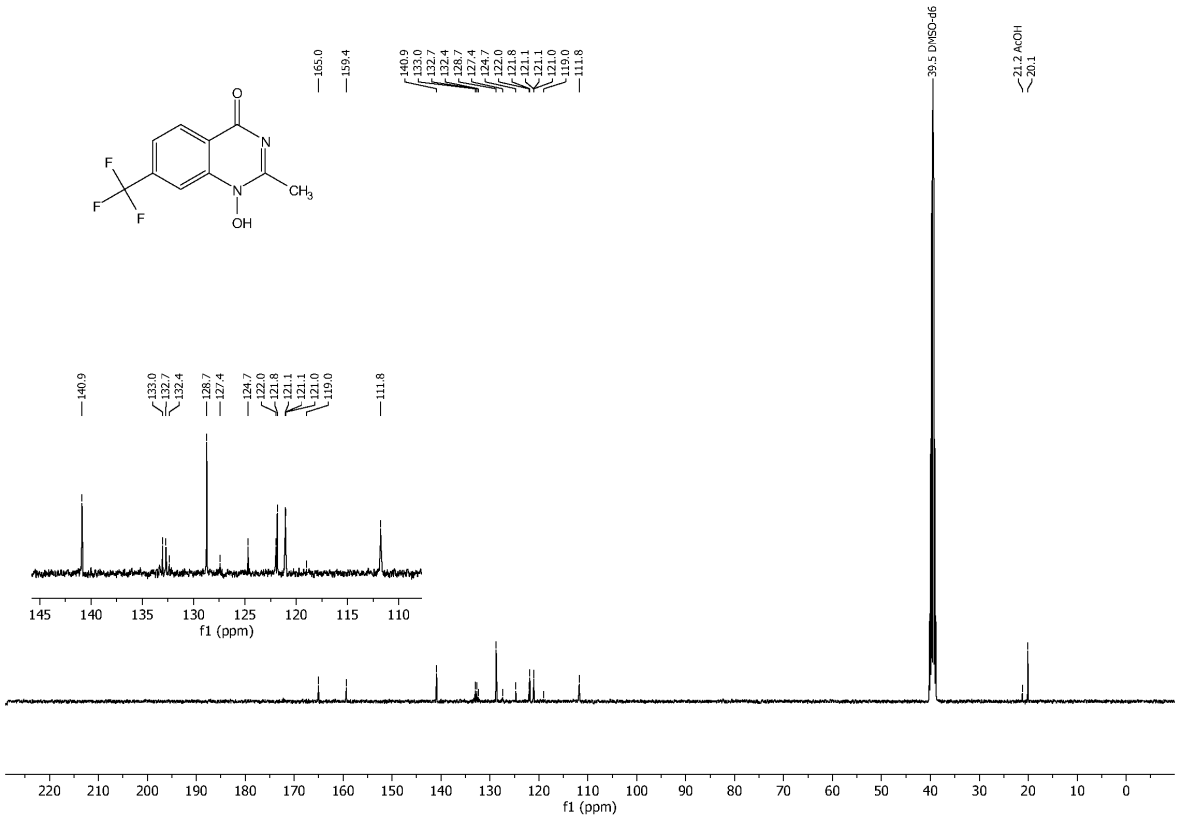
## 1*H*-1-Hydroxy-6-iodo-2-methylquinazolin-4-one (6m)

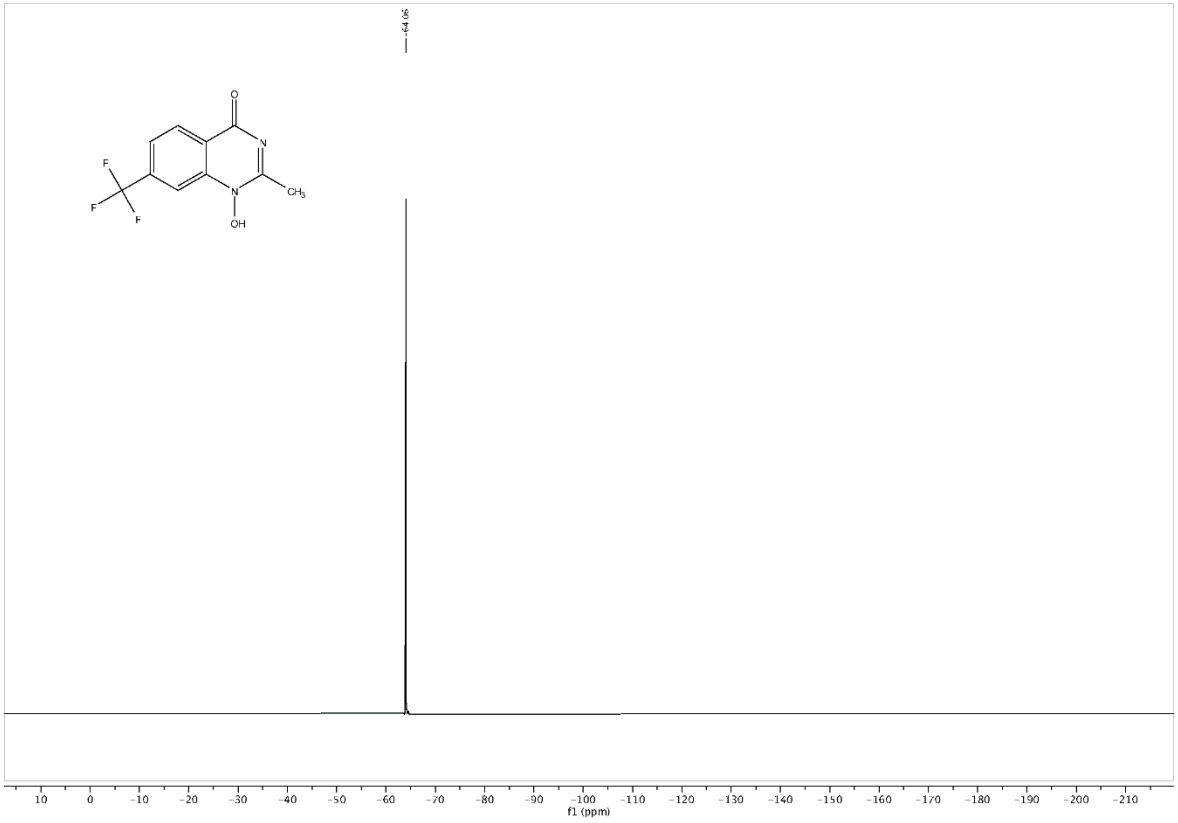


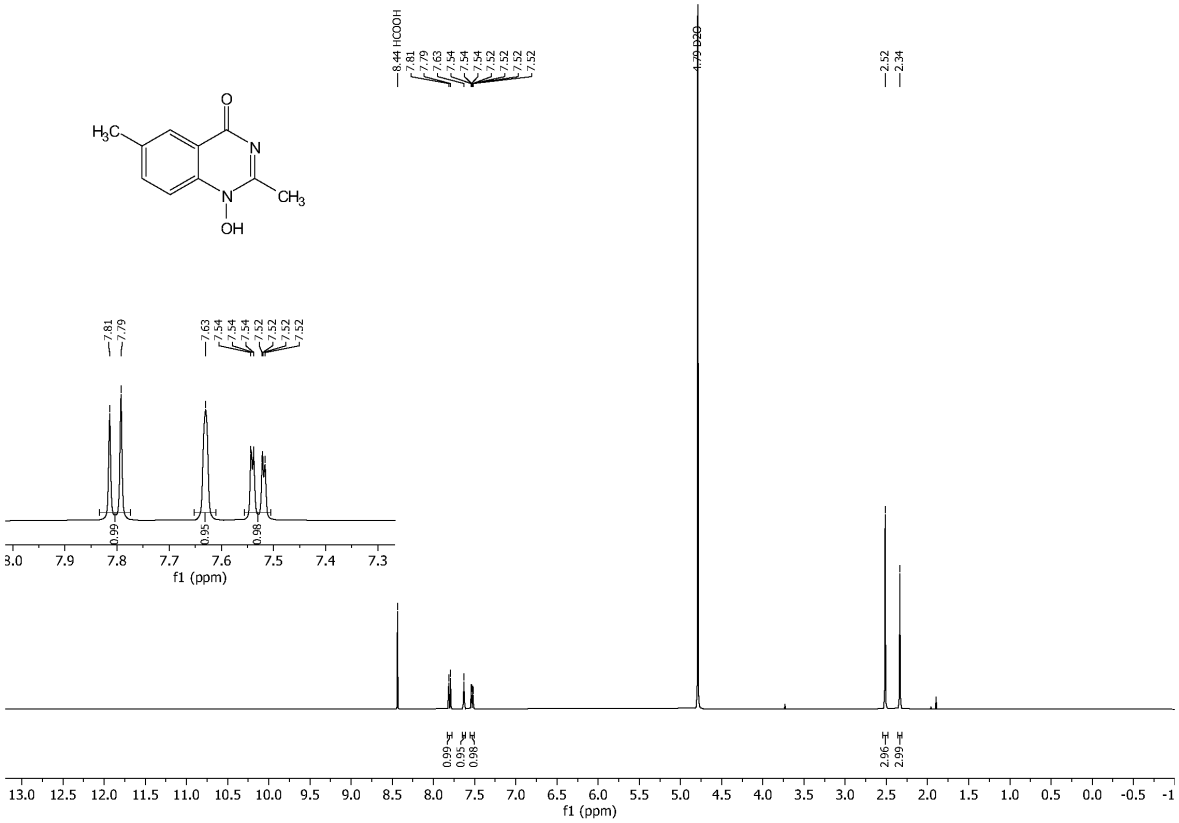
**1*H*-1-Hydroxy-2-methyl-7-(trifluoromethyl)quinazolin-4-one (6n)**

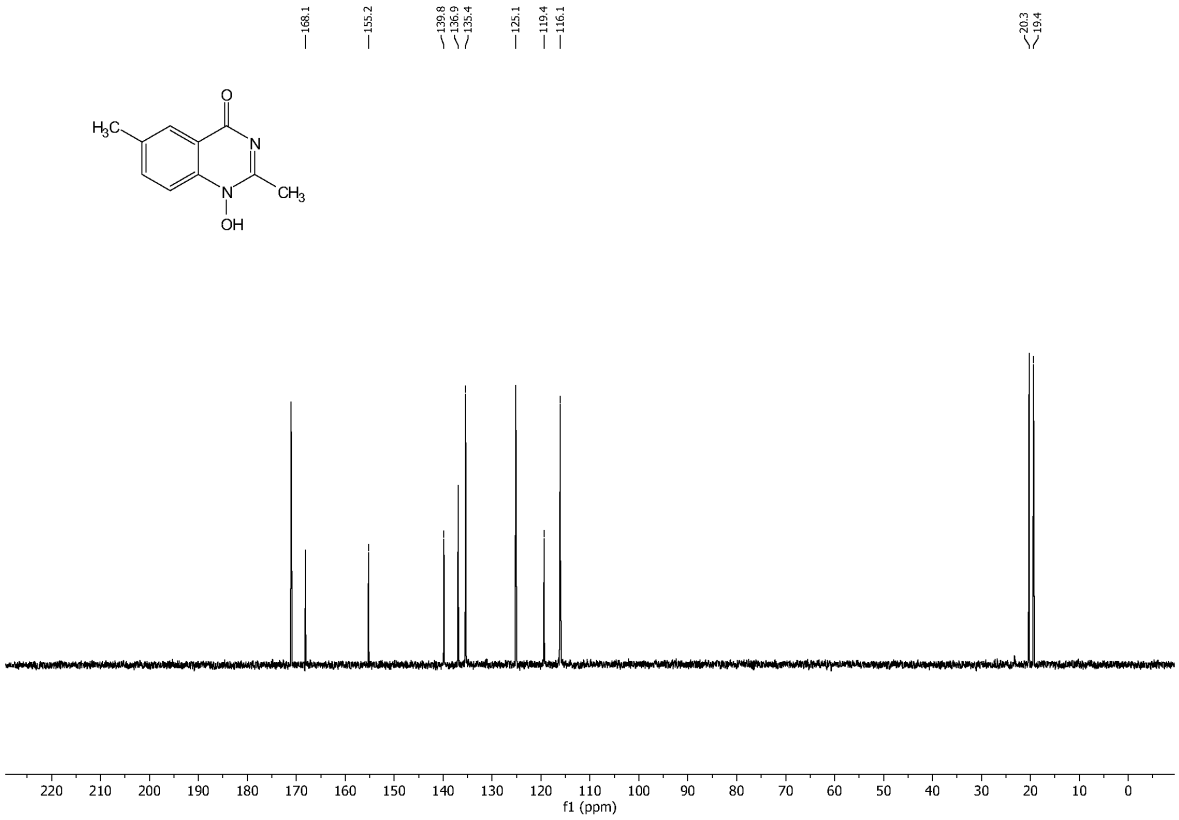


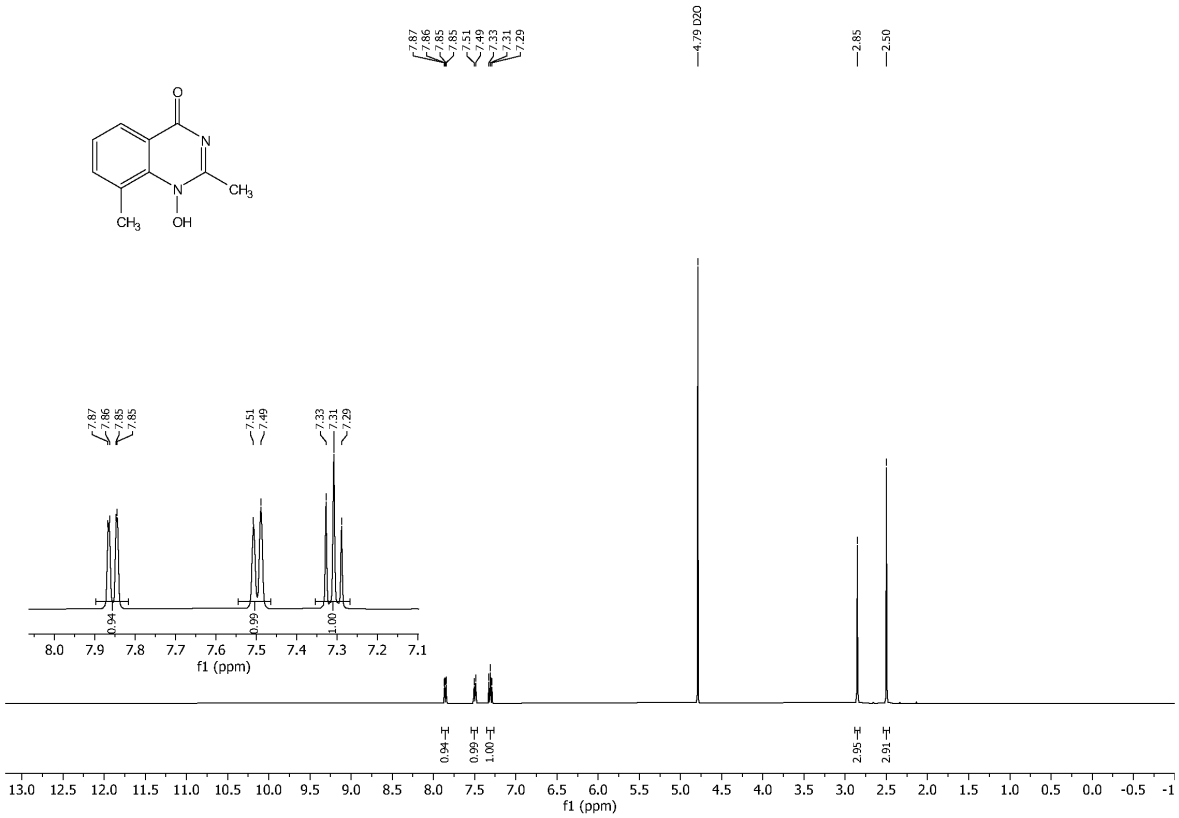

-64.06
O
N
F
F
F
N
CH3
OH
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

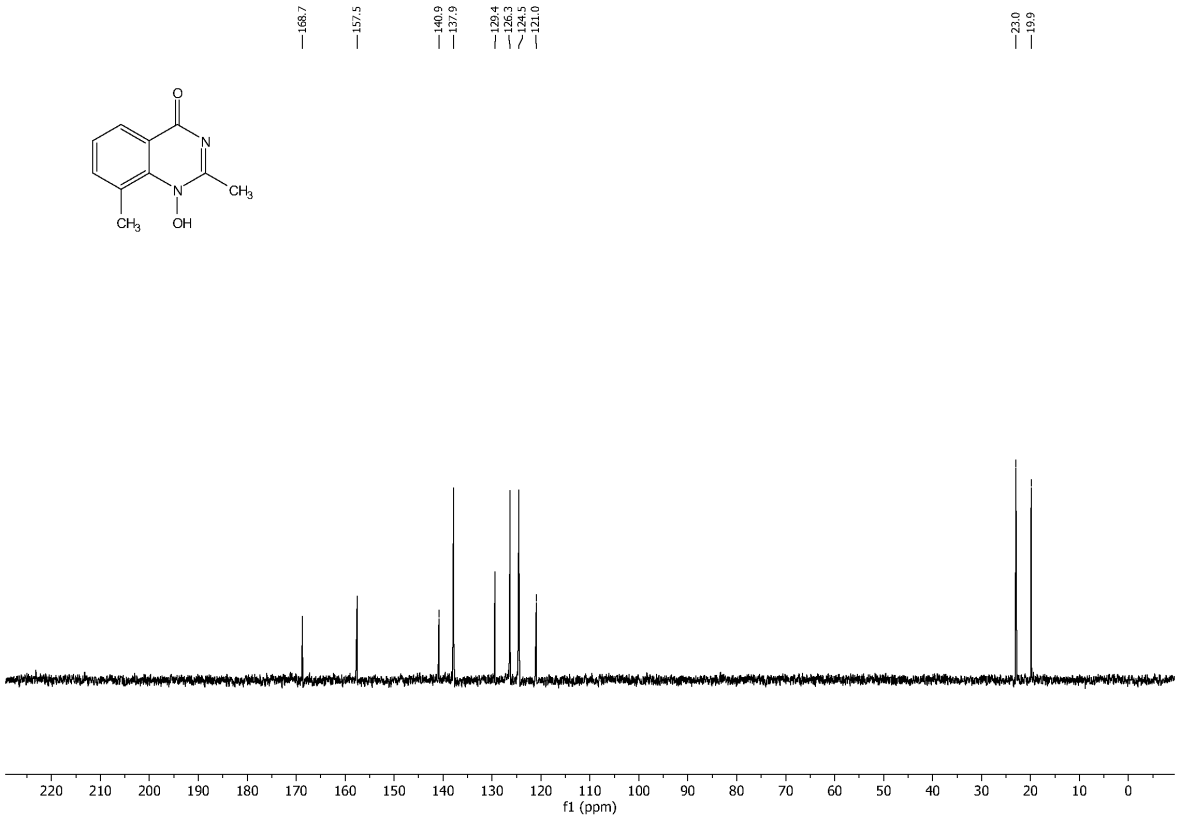
## 1*H*-1-Hydroxy-2,6-dimethylquinazolin-4-one (6o)



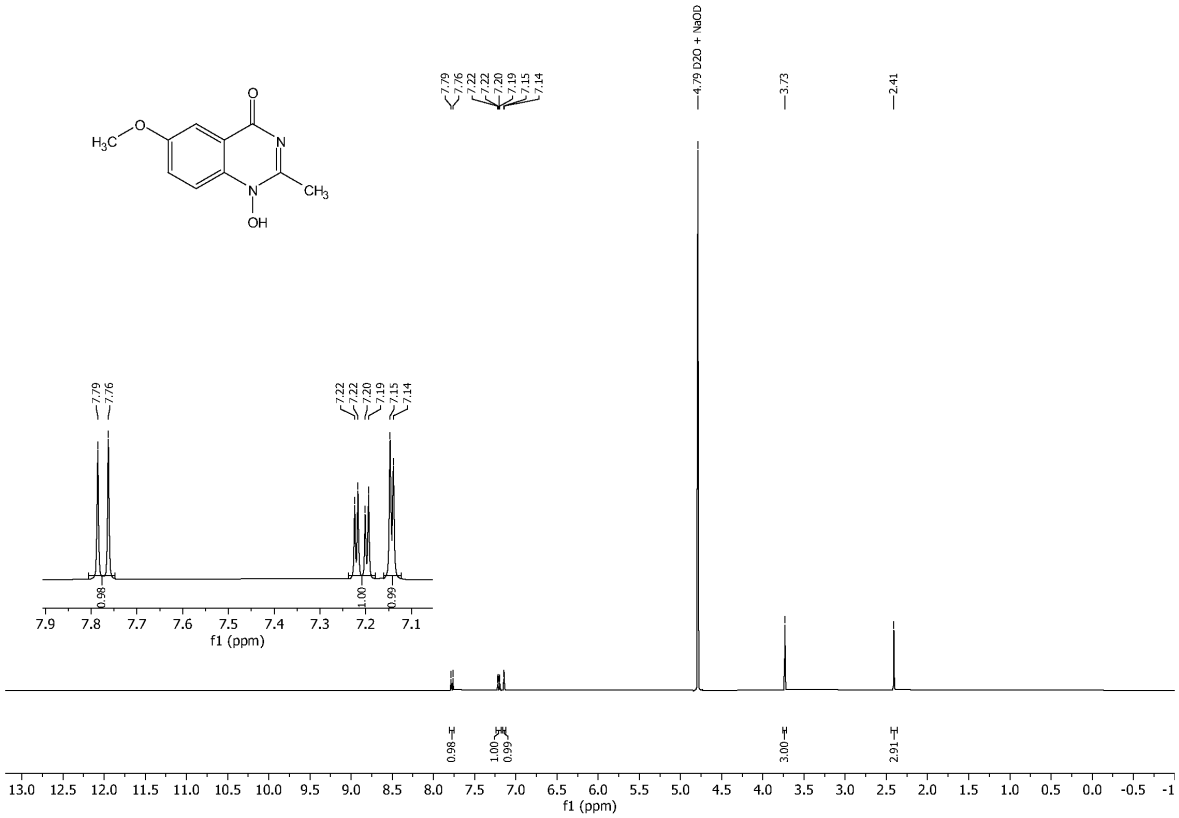



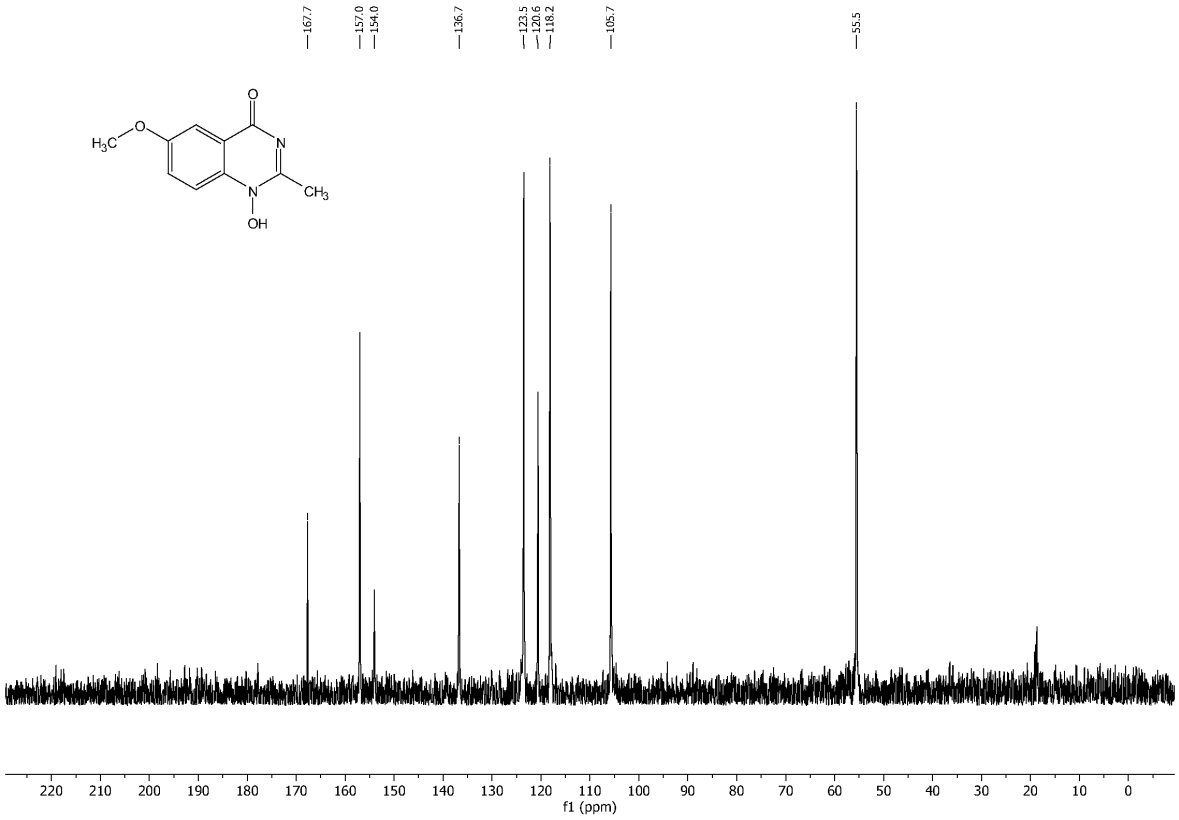
## 1*H*-1-Hydroxy-2,8-dimethylquinazolin-4-one (6p)



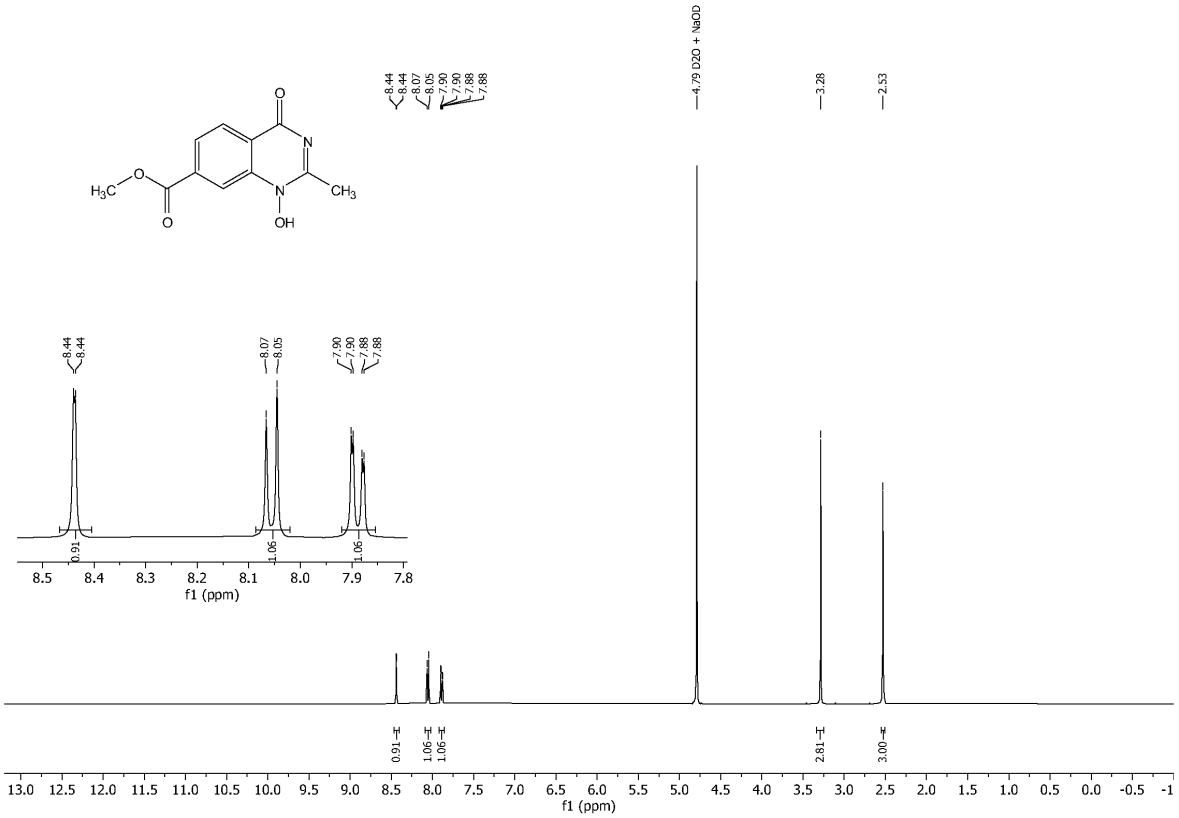



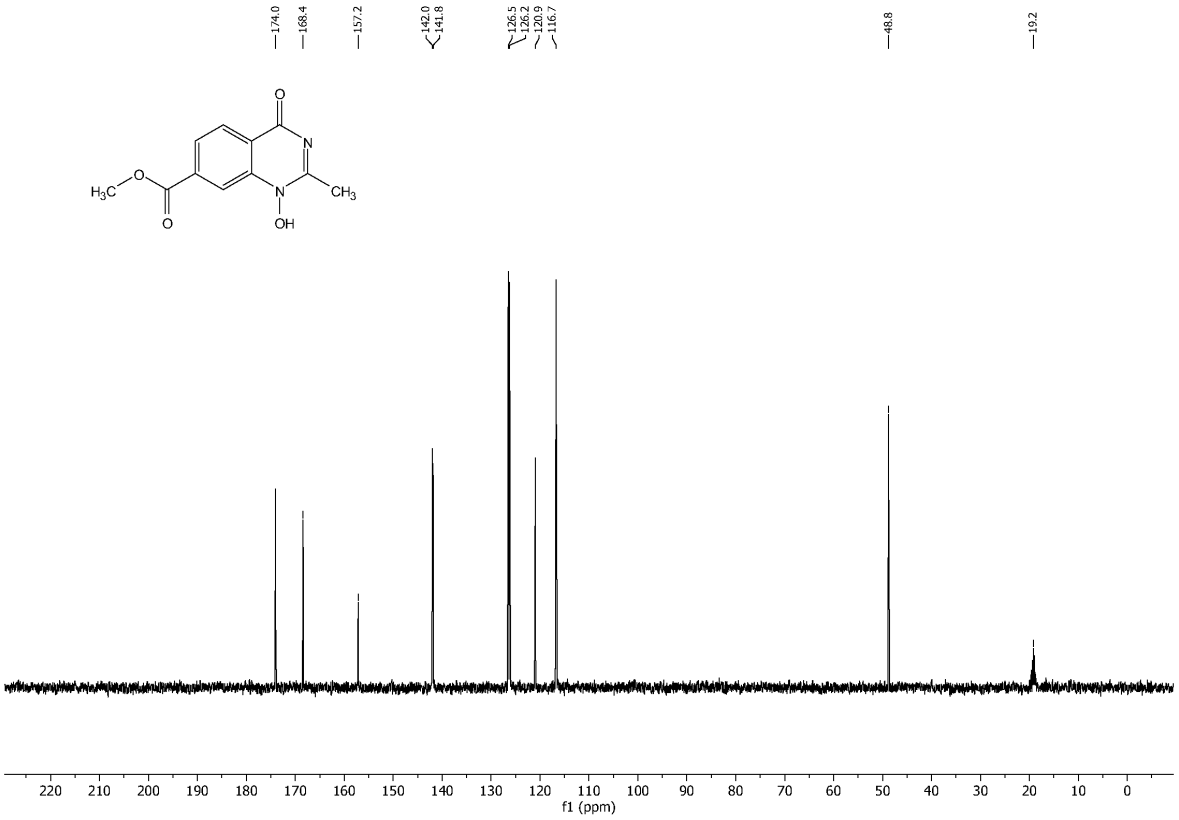
**1*H*-1-Hydroxy-6-methoxy-2-methylquinazolin-4-one (6q)**



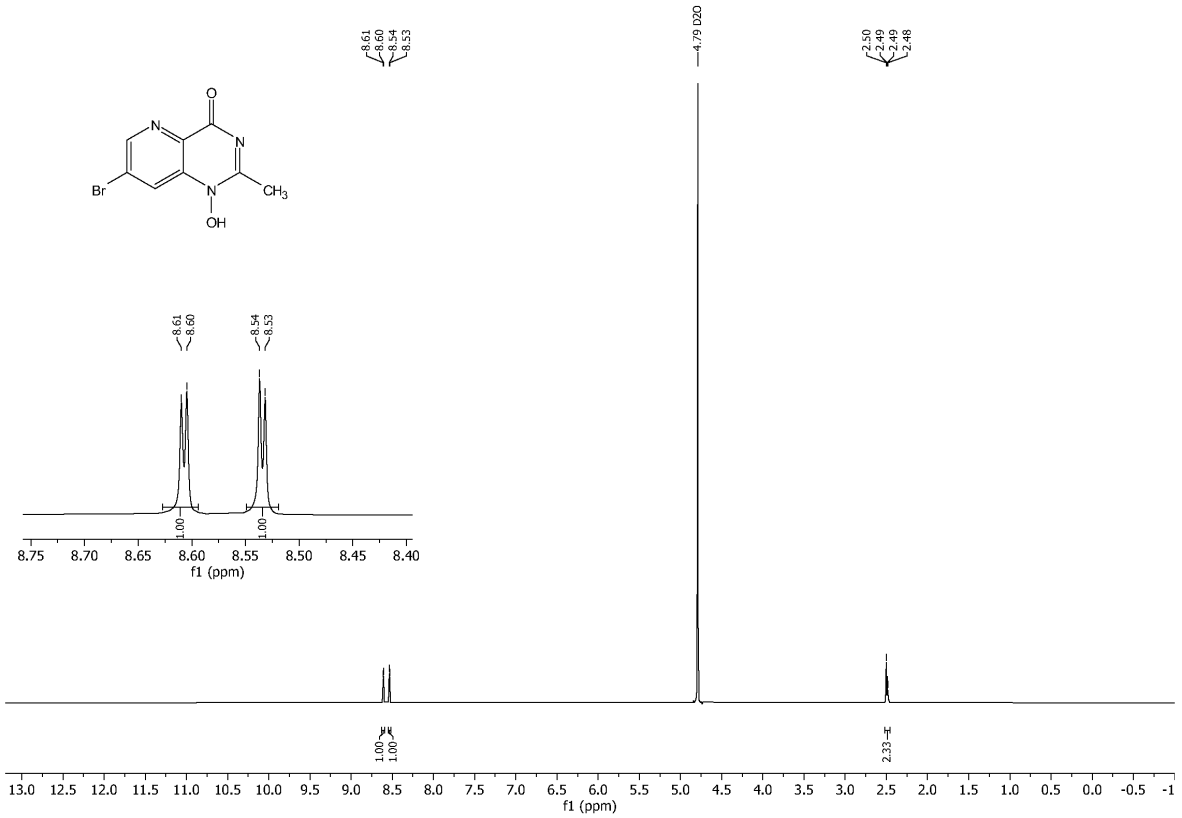



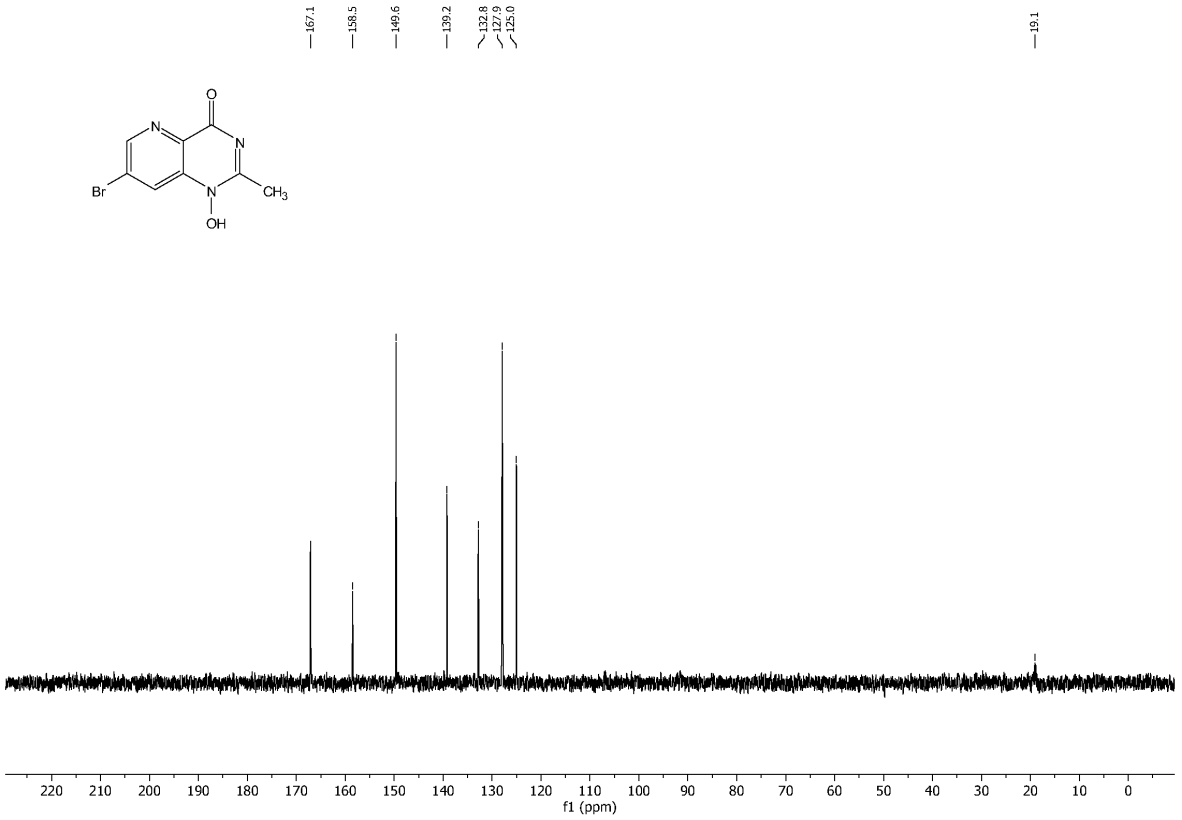
**Methyl 1-hydroxy-2-methyl-4-oxo-1,4-dihydroquinazoline-7-carboxylate (6r)**



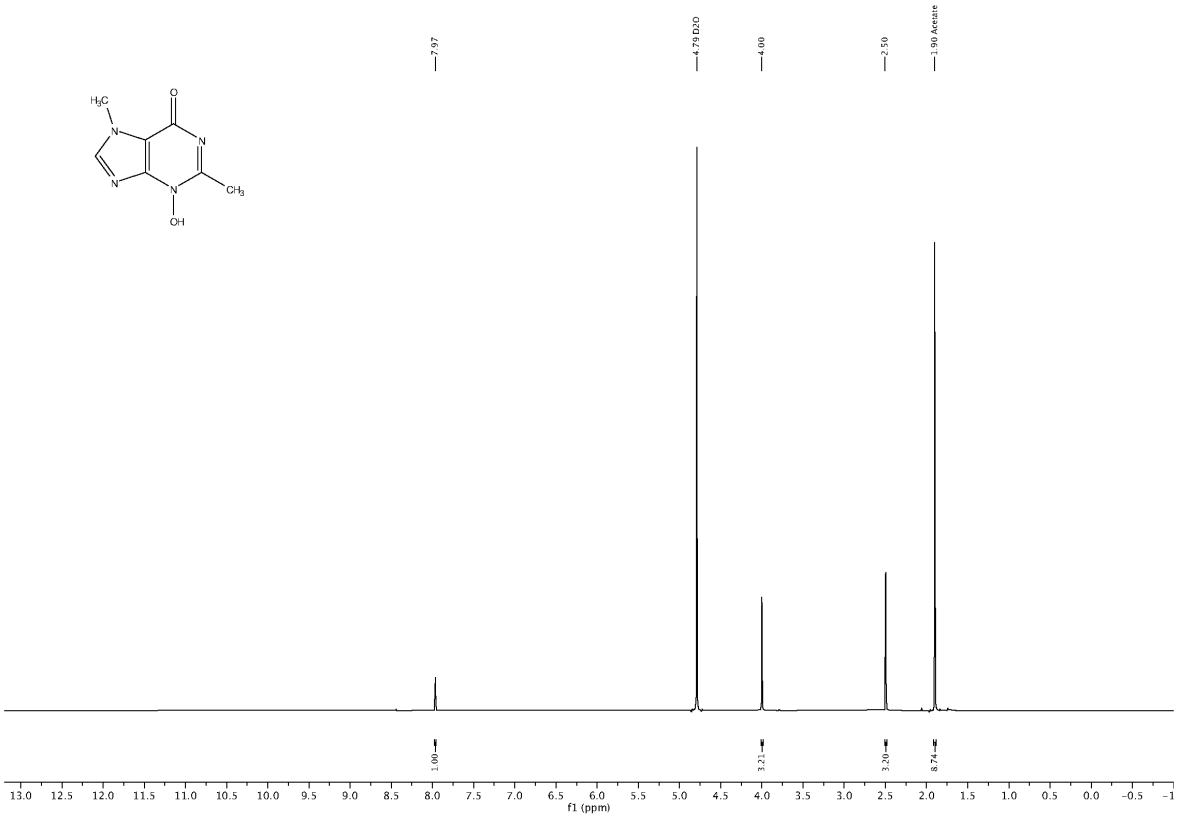



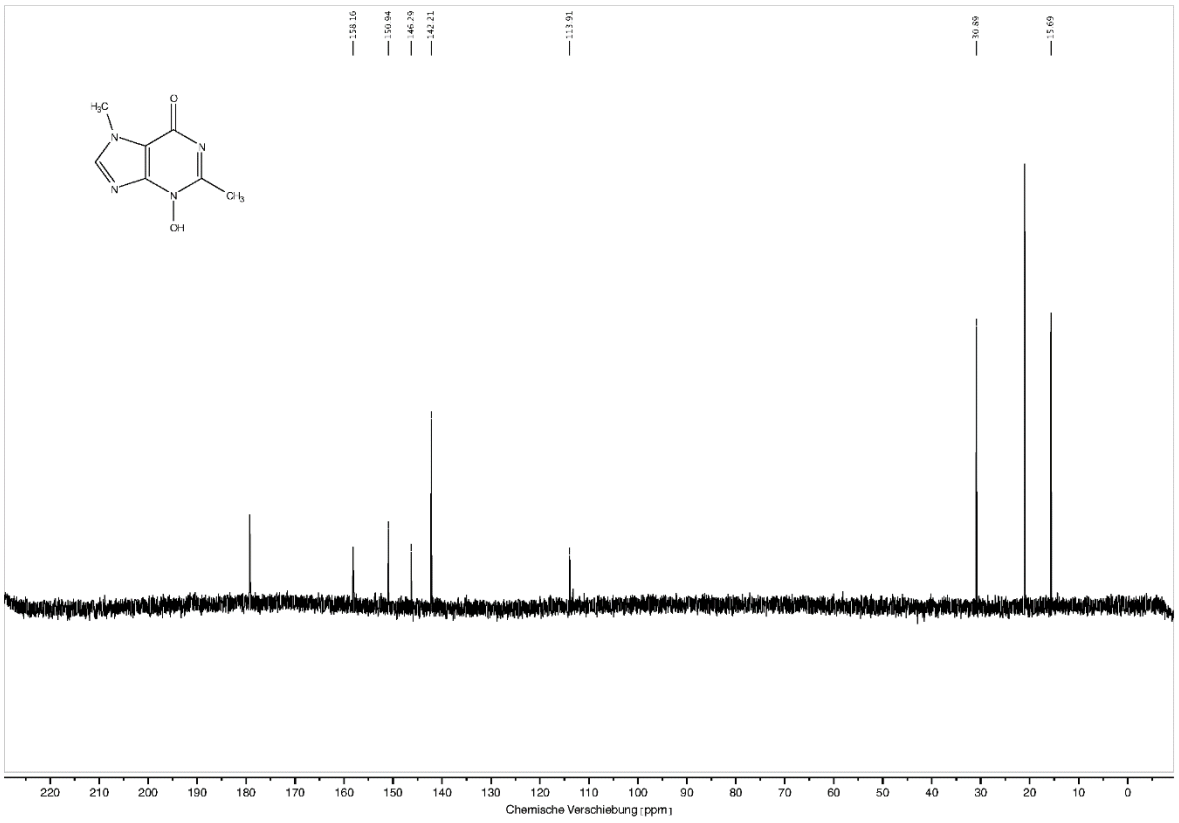
**1*H*-7-Bromo-1-hydroxy-2-methylpyrido[3,2-*d*]pyrimidin-4-one (6s)**



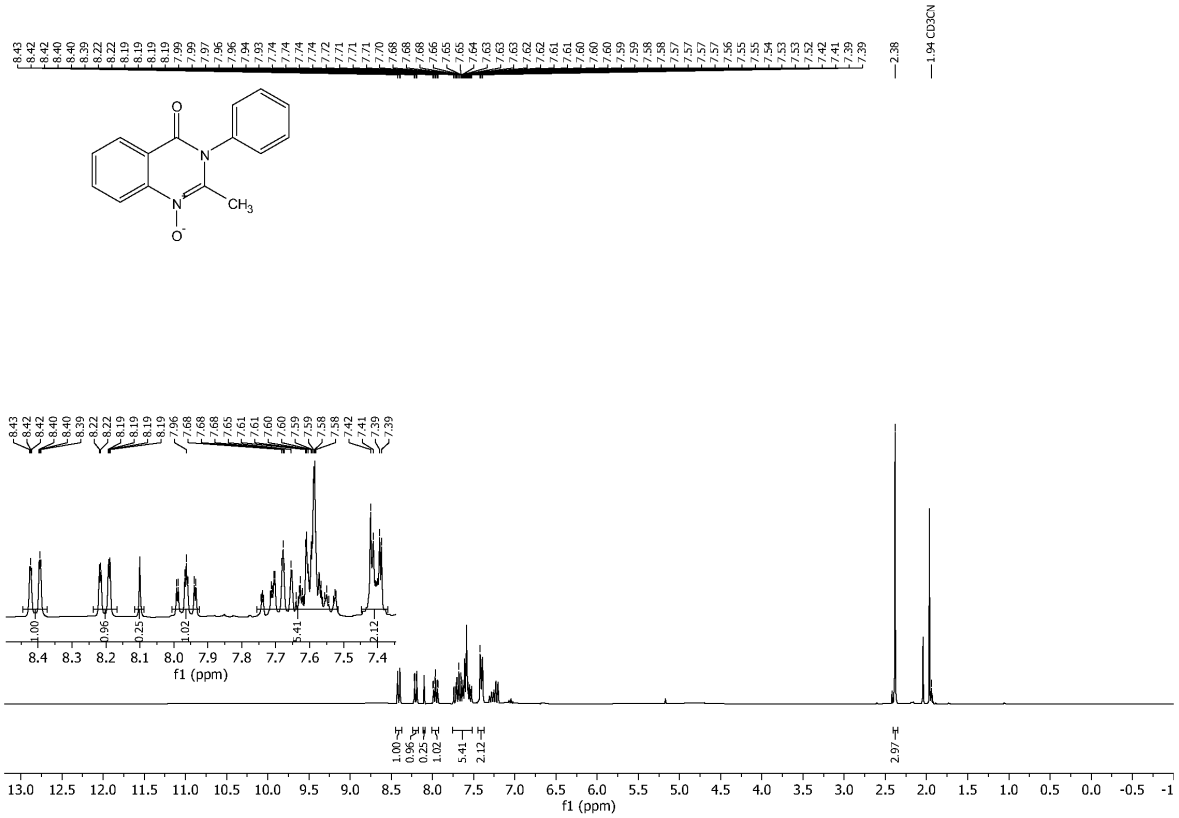



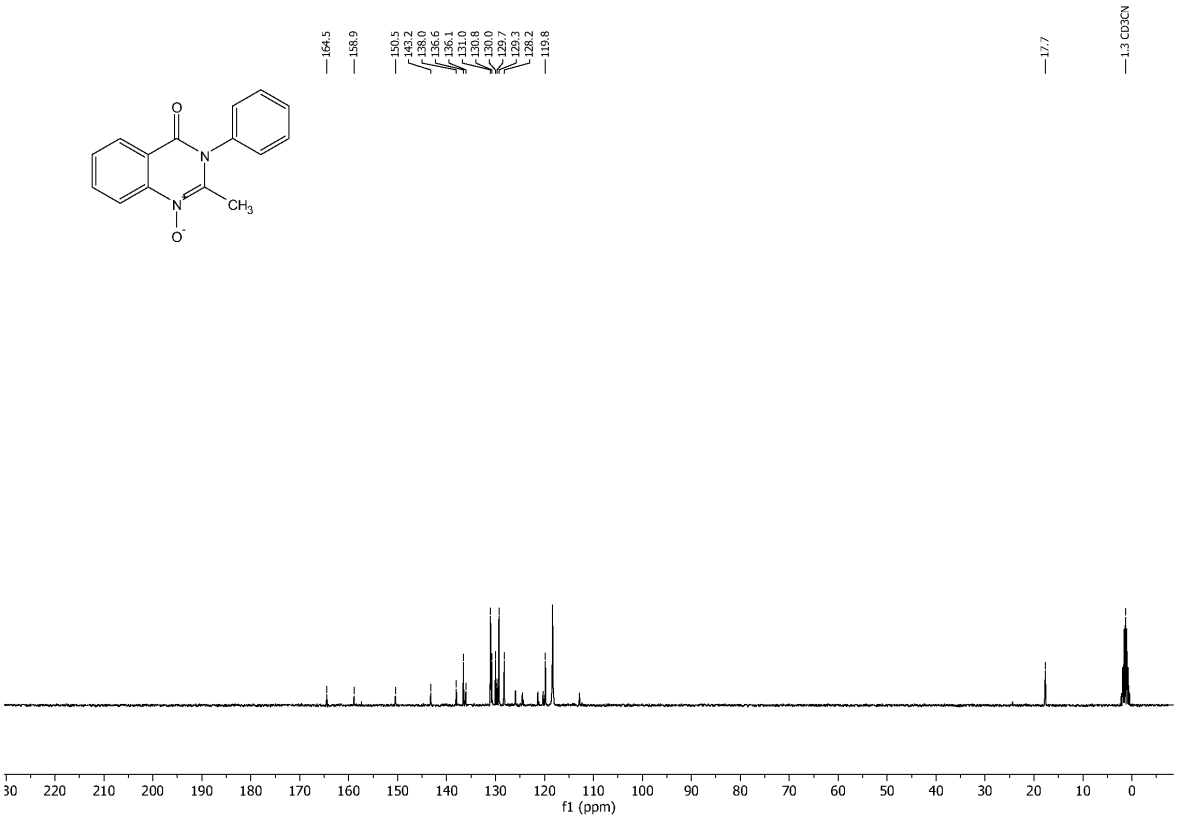
**6*H*-3-Hydroxy-2,7-dimethyl-3,7-dihydropurin-6-one (6t)**



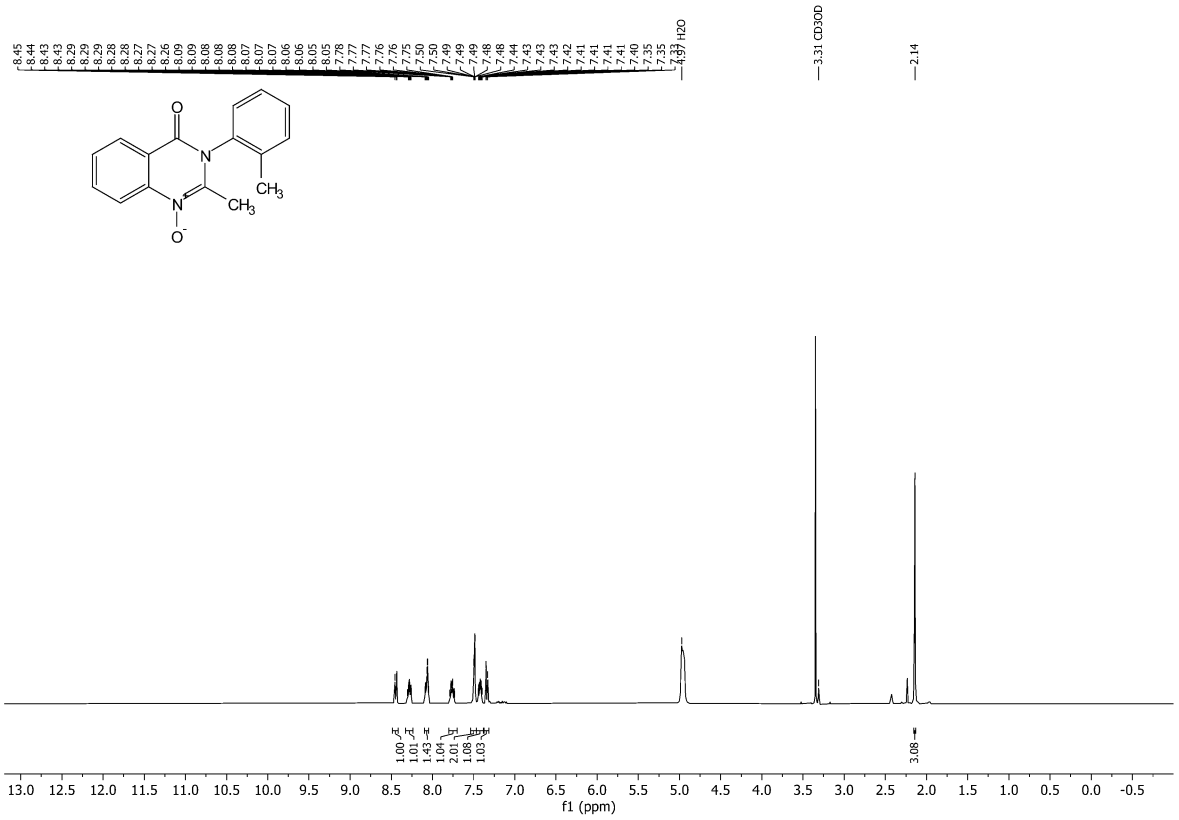



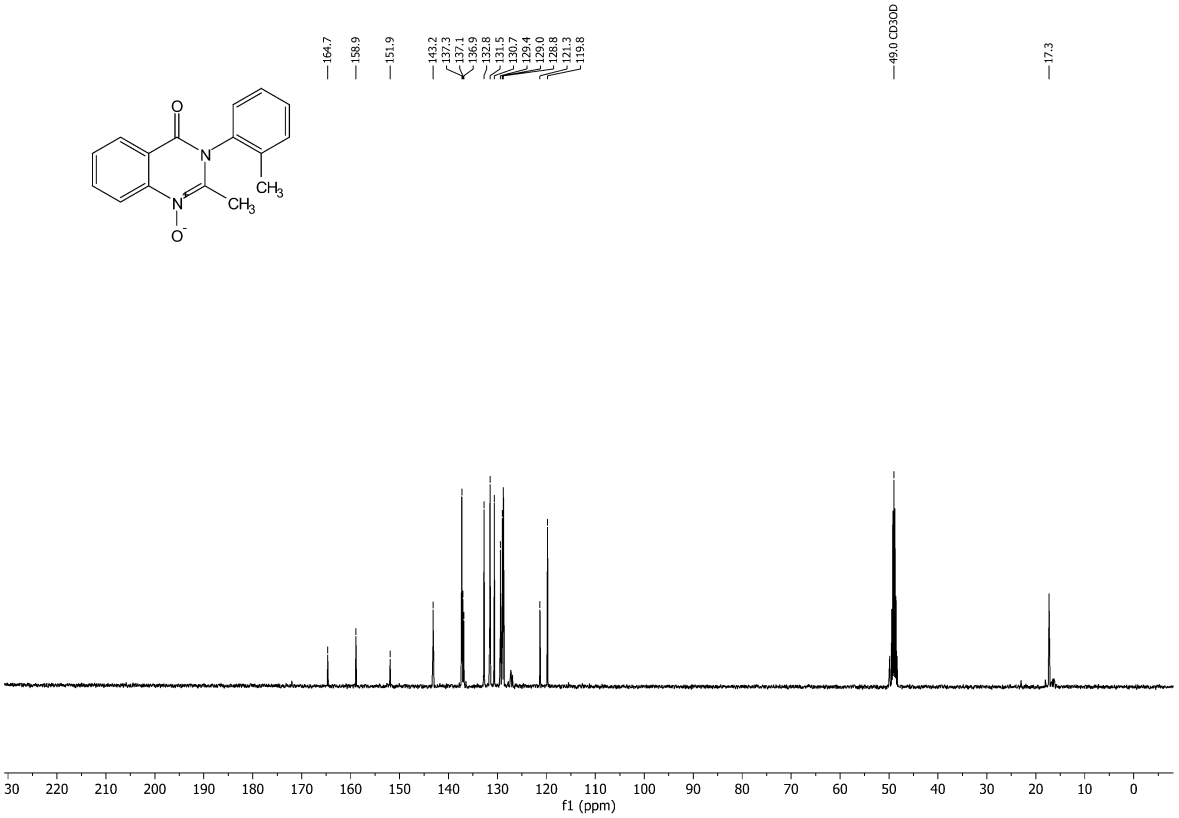
## 2-Methyl-4-oxo-3-phenyl-3,4-dihydroquinazoline 1-oxide (7a)



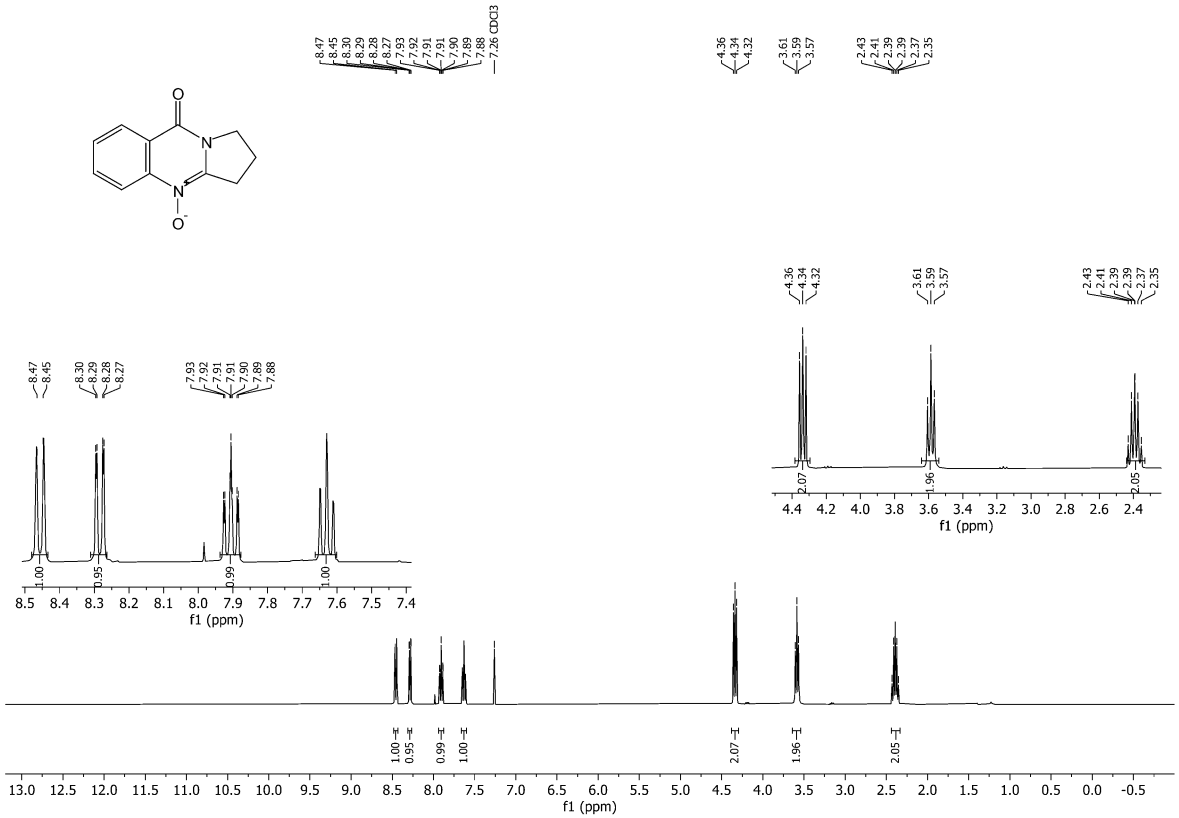


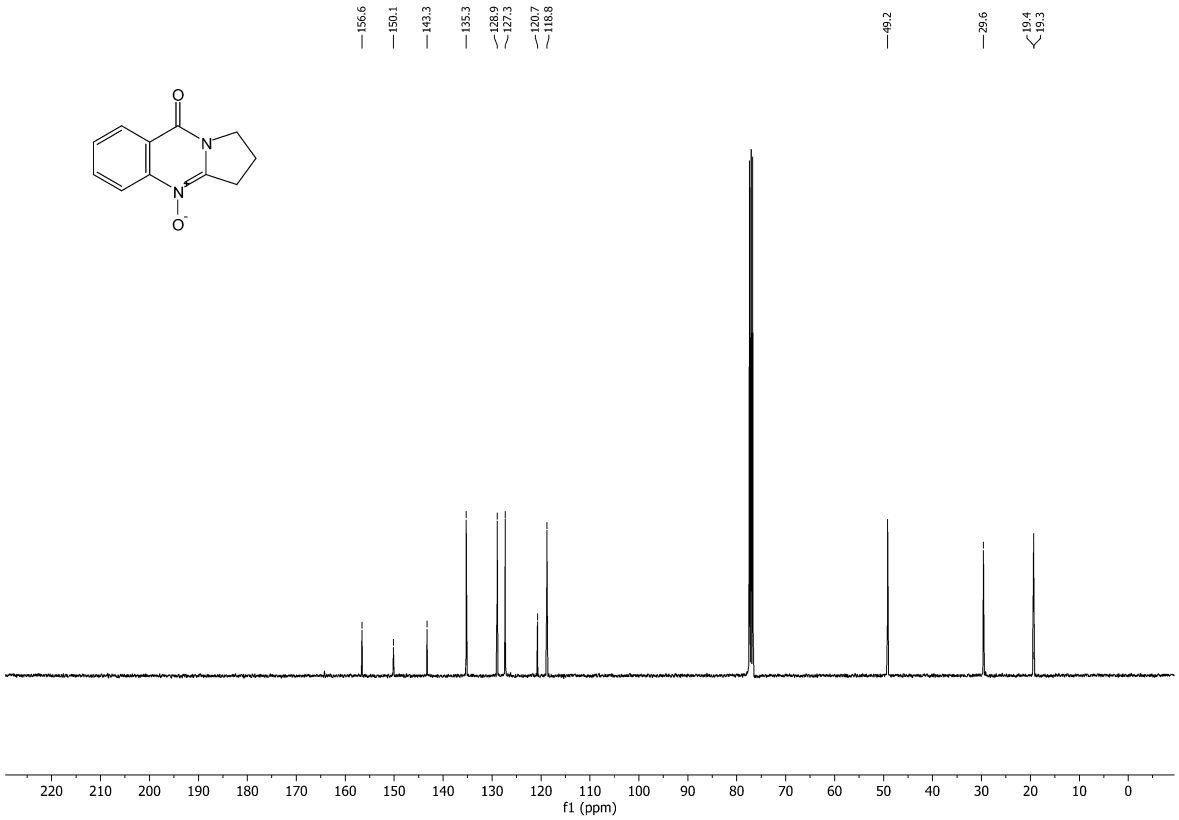
## 2-Methyl-4-oxo-3-(o-tolyl)-3,4-dihydroquinazoline 1-oxide (7b)



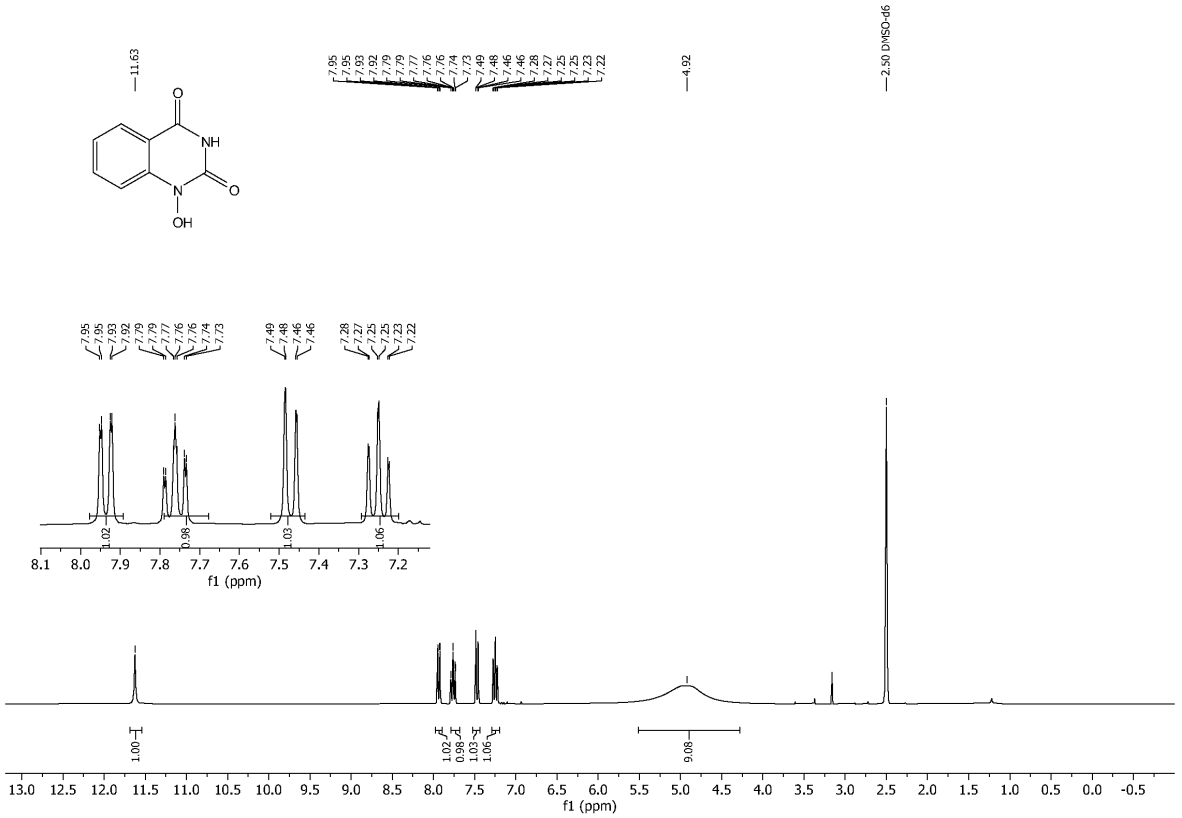



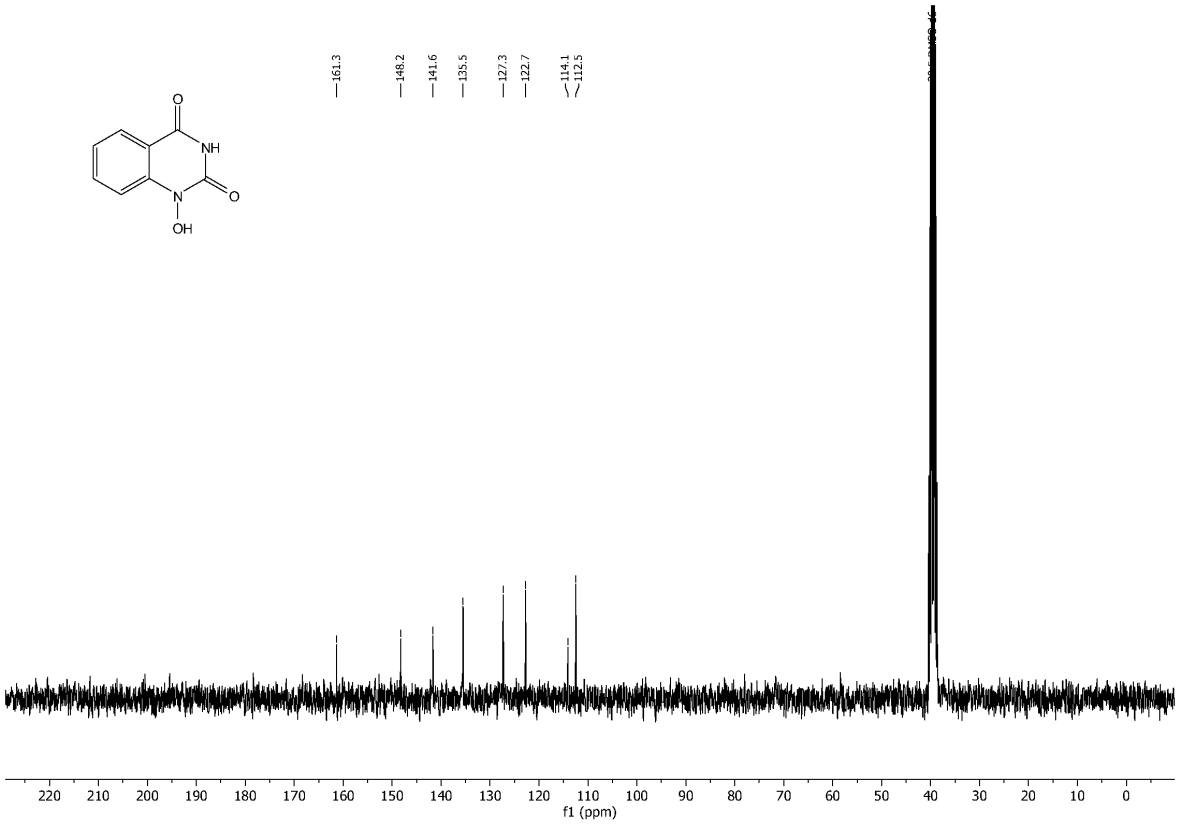
## 9-Oxo-1,2,3,9-tetrahydropyrrolo[2,1-*b*]quinazoline 4-oxide (7c)



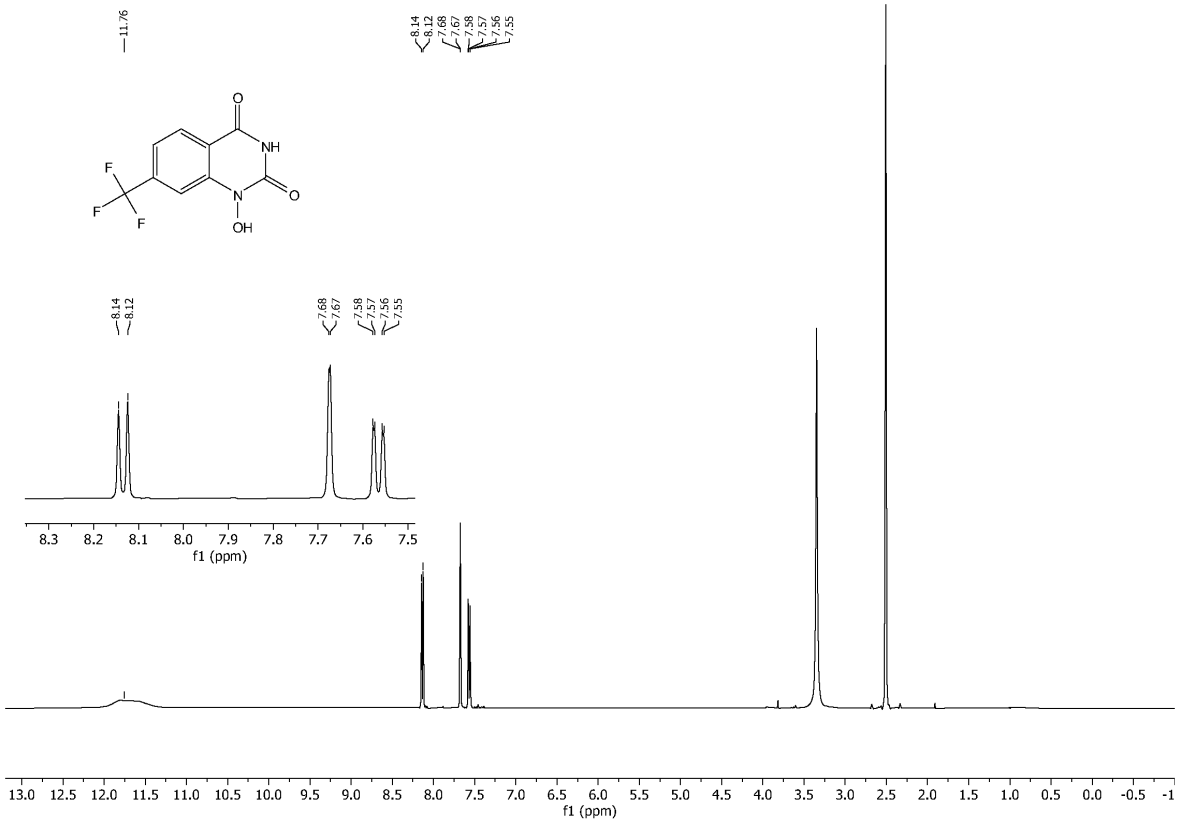



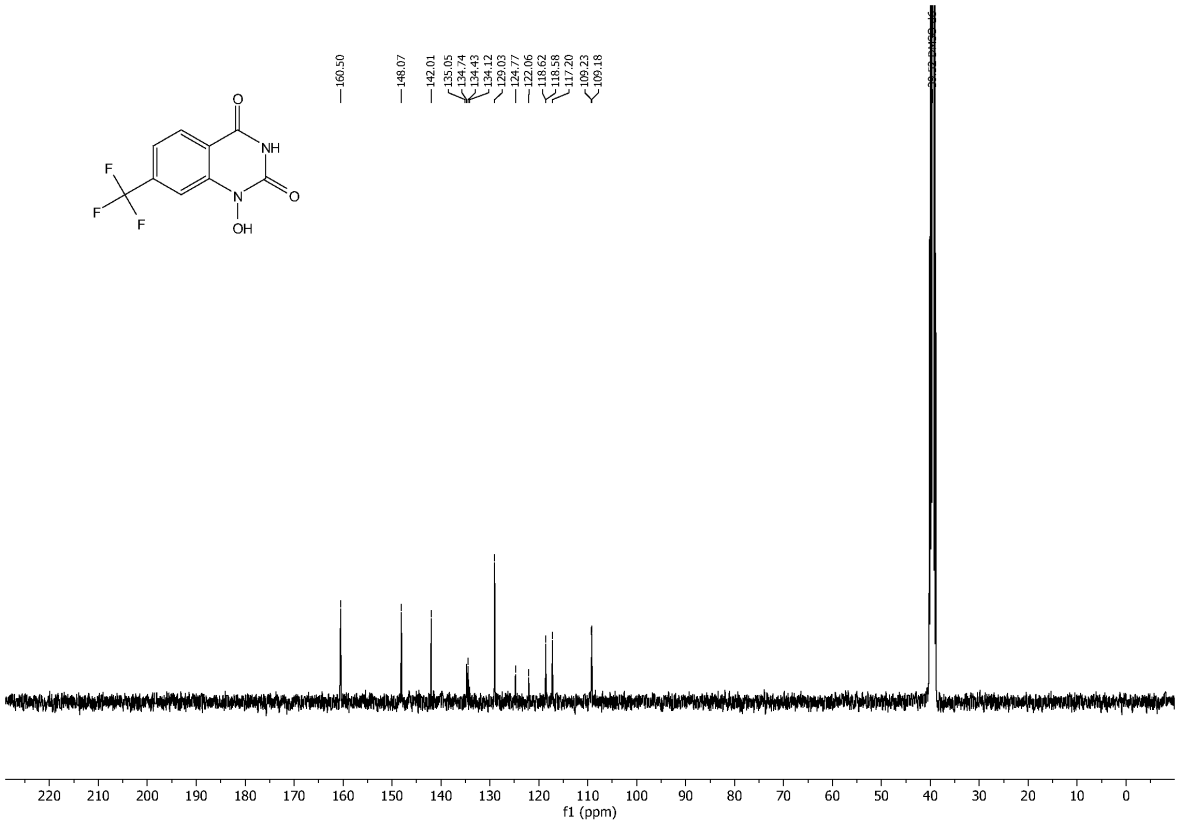
## 1*H*,3*H*-1-Hydroxyquinazoline-2,4-dione (8a)

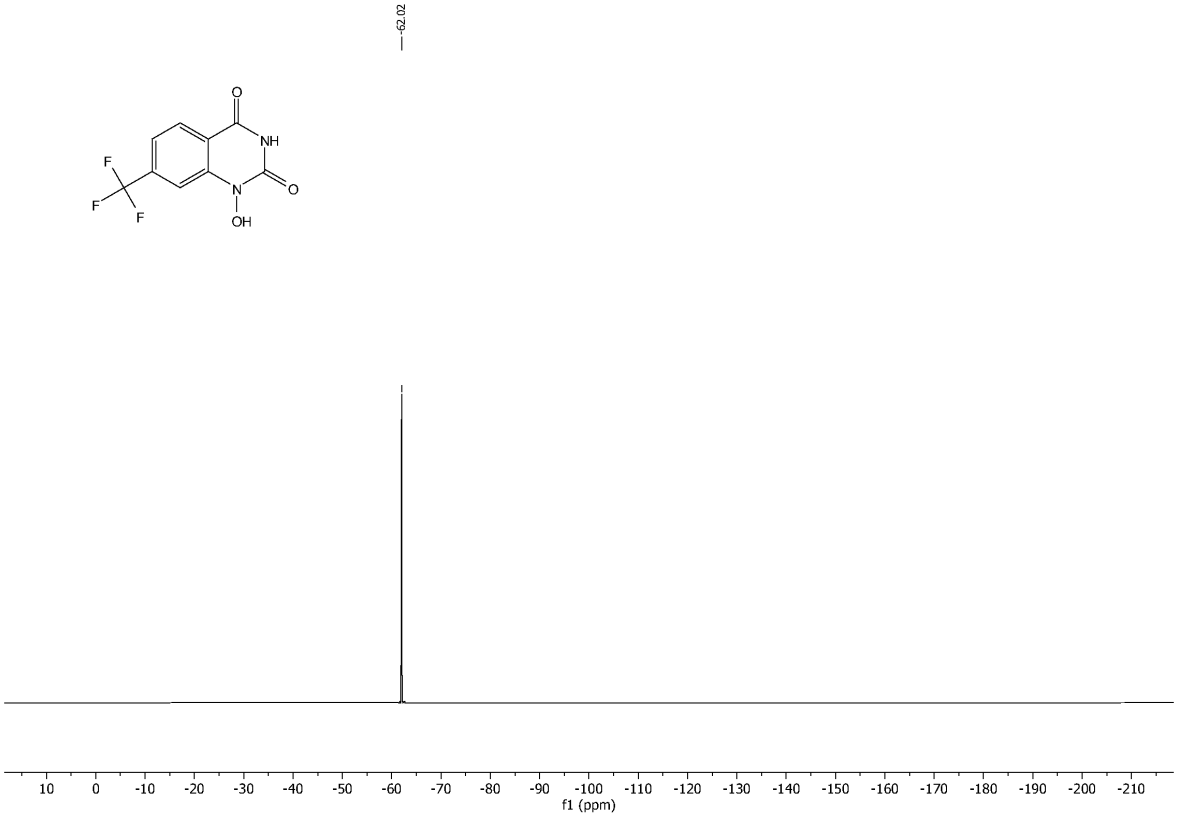


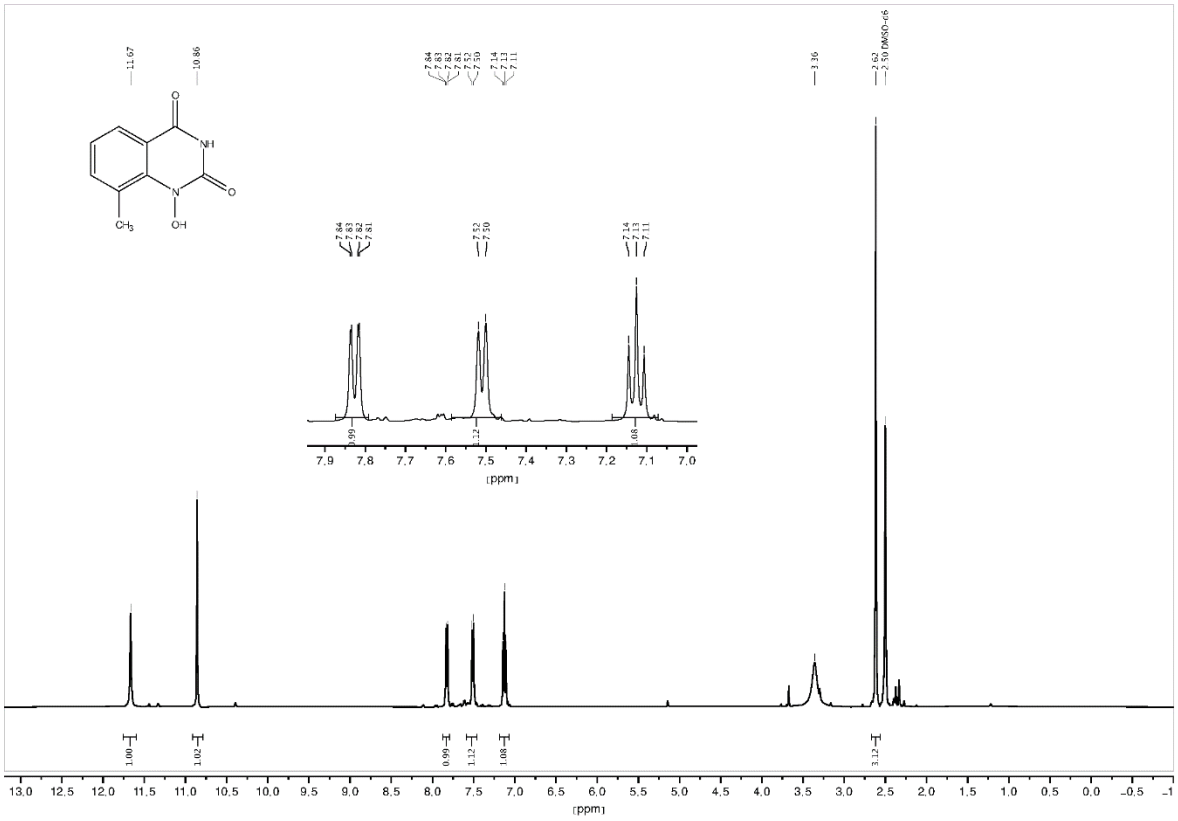
**1*H*,3*H*-1-Hydroxy-7-(trifluoromethyl)quinazoline-2,4-dione (8b)**

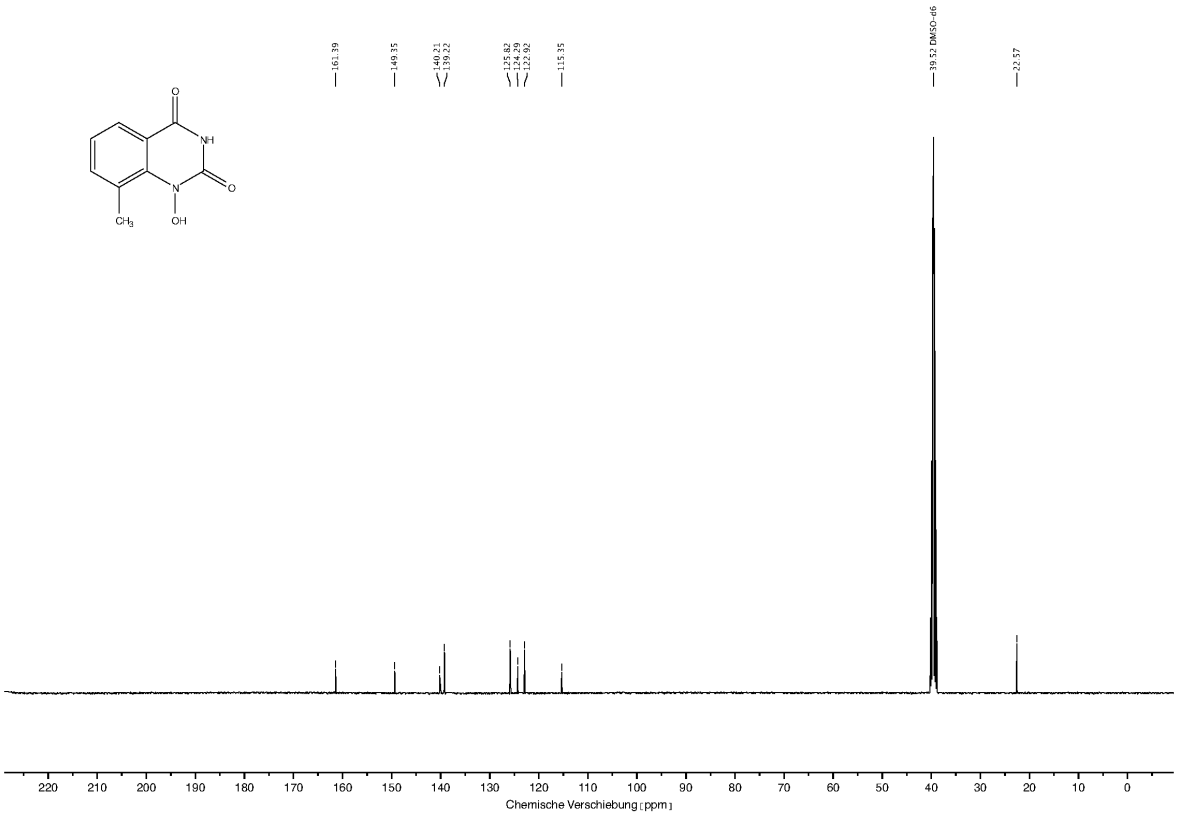


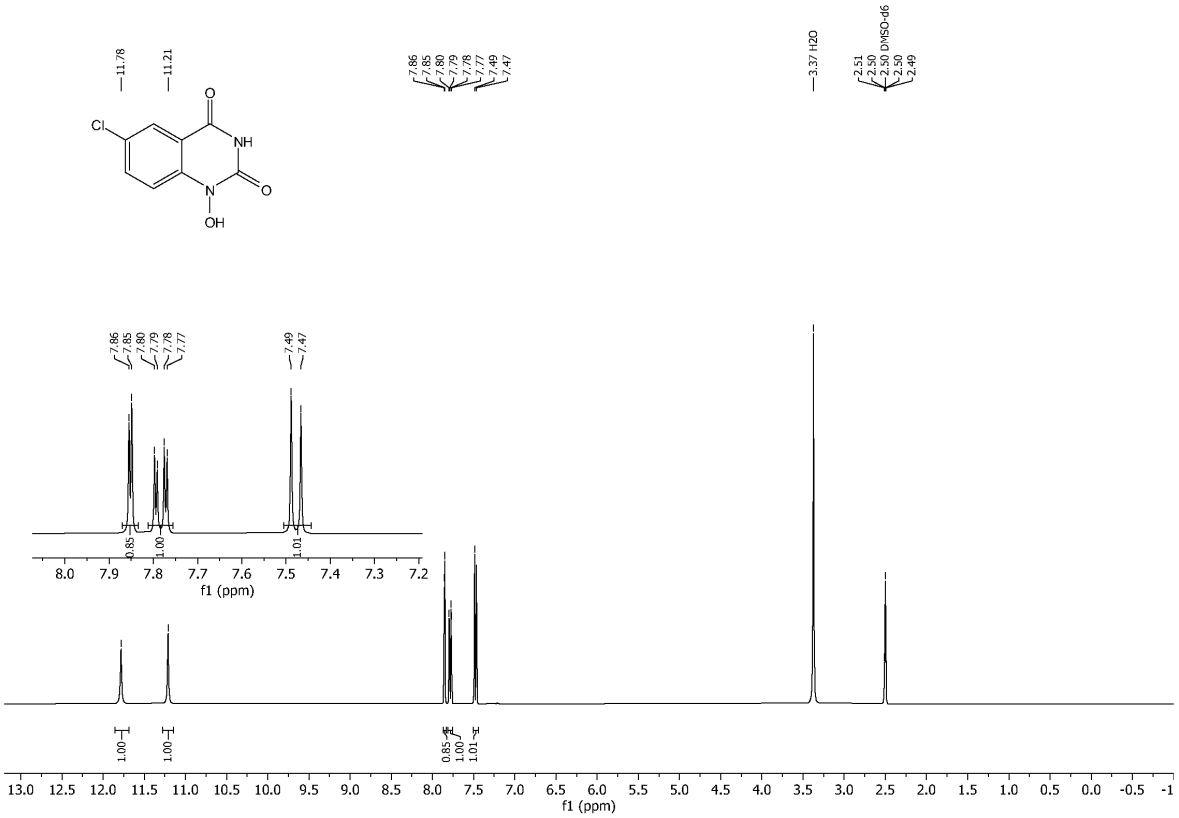

-62.02
O
NH
O
N
OH
F
F
F
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

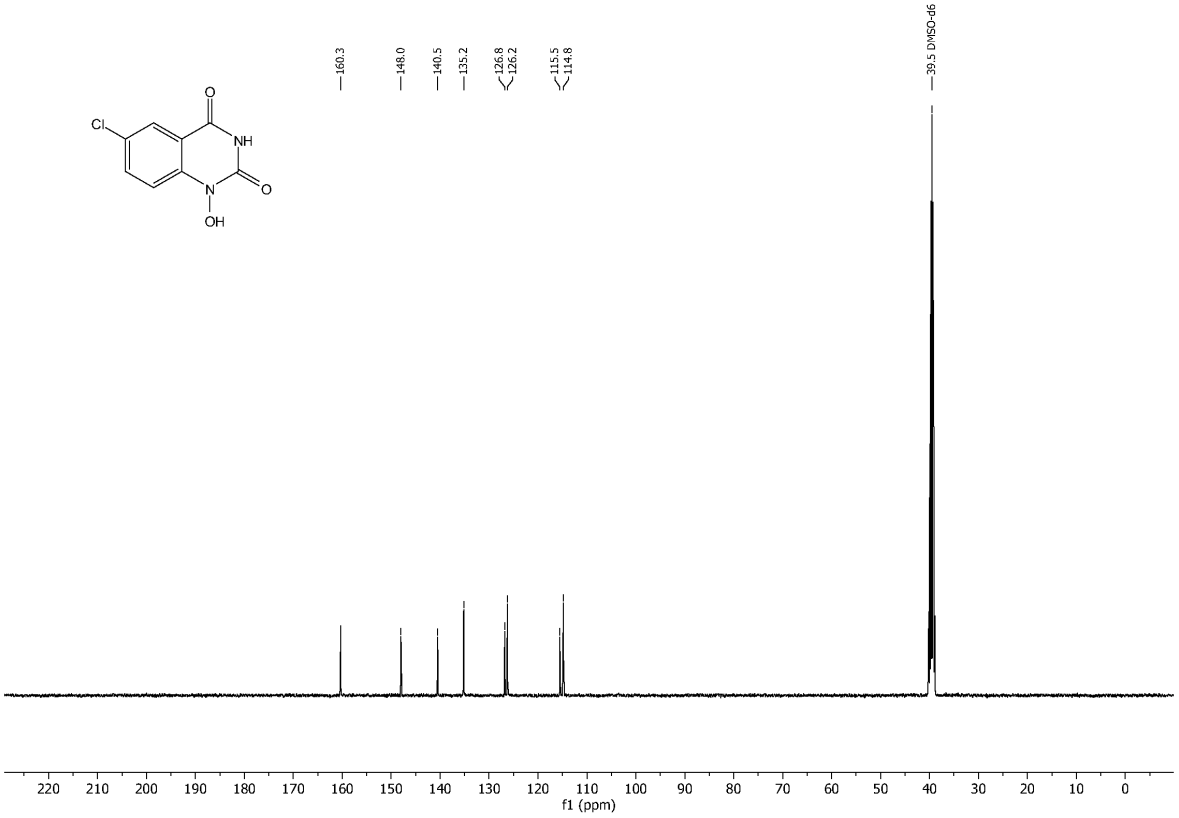
**1*H*,3*H*-1-Hydroxy-8-methylquinazoline-2,4-dione (8c)**



**1*H*,3*H*-6-Chloro-1-hydroxyquinazoline-2,4-dione (8d)**



## 8. References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