A Microwave-Assisted Multicomponent Synthesis of Substituted 3,4-Dihydroquinazolinones

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SUPPORTING INFORMATION

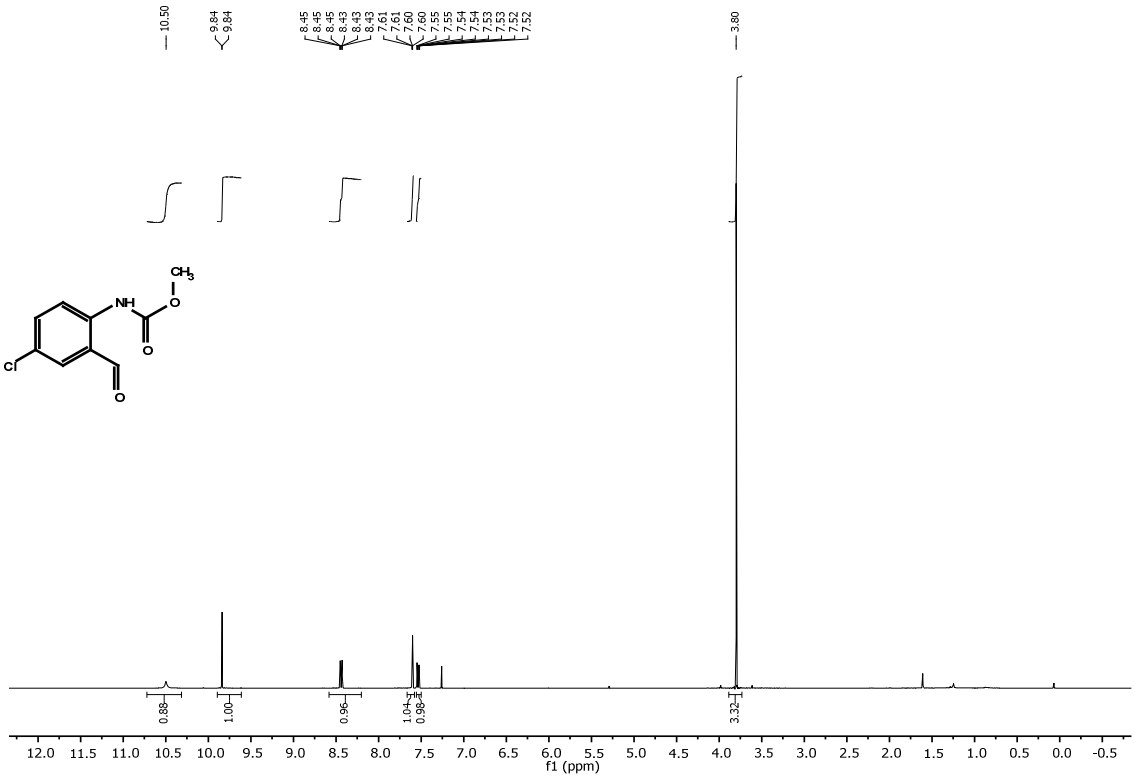
Contents

Experimental Section

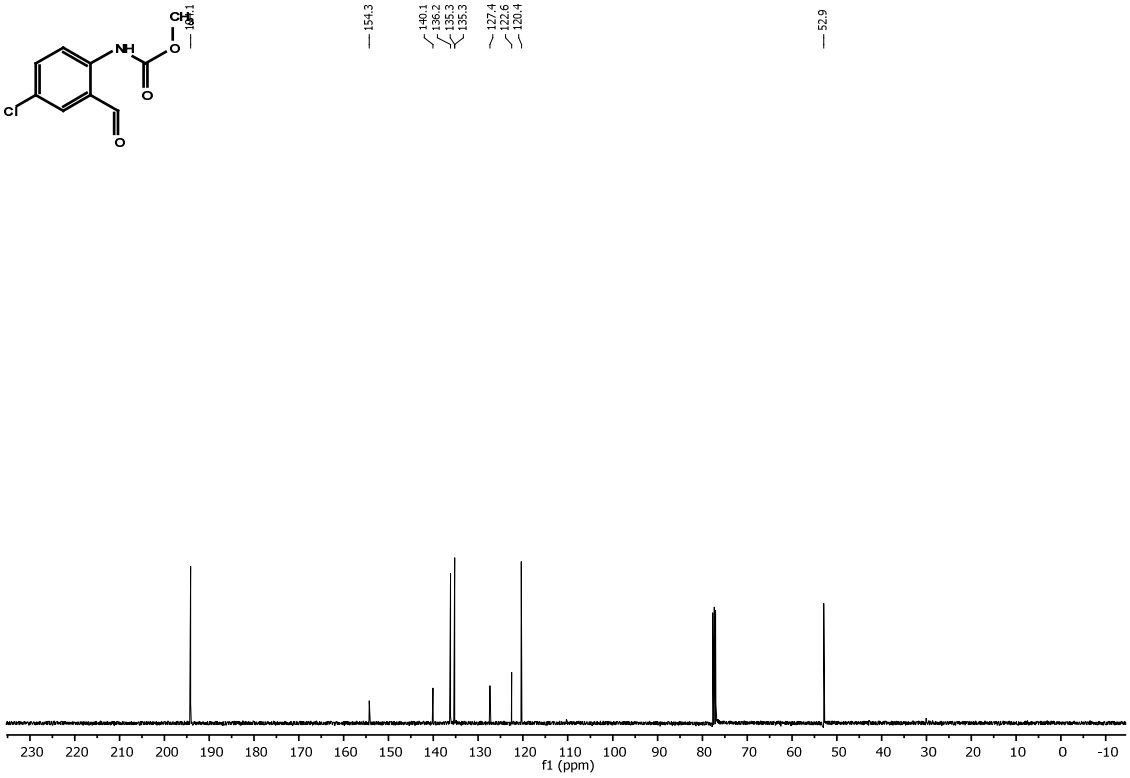
I. General Information

Analytical thin-layer chromatography was performed on silica gel 60 F-254 plates and visualized by UV light or ninhydrin stain. Flash column chromatography was performed using silica gel (60, particle size 40-63 nm). ^{1}H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra at 100 MHz. The chemical shifts for ^{1}H NMR and ^{13}C NMR were referenced to TMS via residual solvent signals (^{1}H, $CDCl_3$ at 7.26 ppm; ^{13}C, CDCl3 at 77.36 ppm; ^{1}H, DMSO-d_6 at 2.45 ppm; ^{13}C, DMSO-d_6 at 39.43 ppm, ^{1}H, CD_3OD at 3.31 ppm; ^{13}C, CD_3OD at 49.0 ppm). Microwave reactions were performed in an Initiator single mode reactor producing controlled irradiation at 2450 MHz and the temperature was monitored using the built in online IR sensor. LC/MS was performed on an instrument equipped with a CP-Sil 8 CB capillary column (50 x 3.0 mm, particle size 2.6 μm, pore size 100 Å) operating at an ionization potential of 70 eV using a CH_3CN/H_2O gradient (0.05% HCOOH). Accurate mass values were determined on a mass spectrometer equipped with an electrospray or electron impact ion source and 7-T hybrid ion trap (LTQ) or TOF detector, respectively. All reactions were performed in sealed Pyrex microwave-transparent process vials designed for 0.5–2 mL reaction volumes. Reagents were purchased at the highest commercial quality and were used without further purification. Solvents used for extraction and silica gel chromatography (EtOAc, hexanes, *n*-pentane and dichloromethane) were used without purification or removal of water.

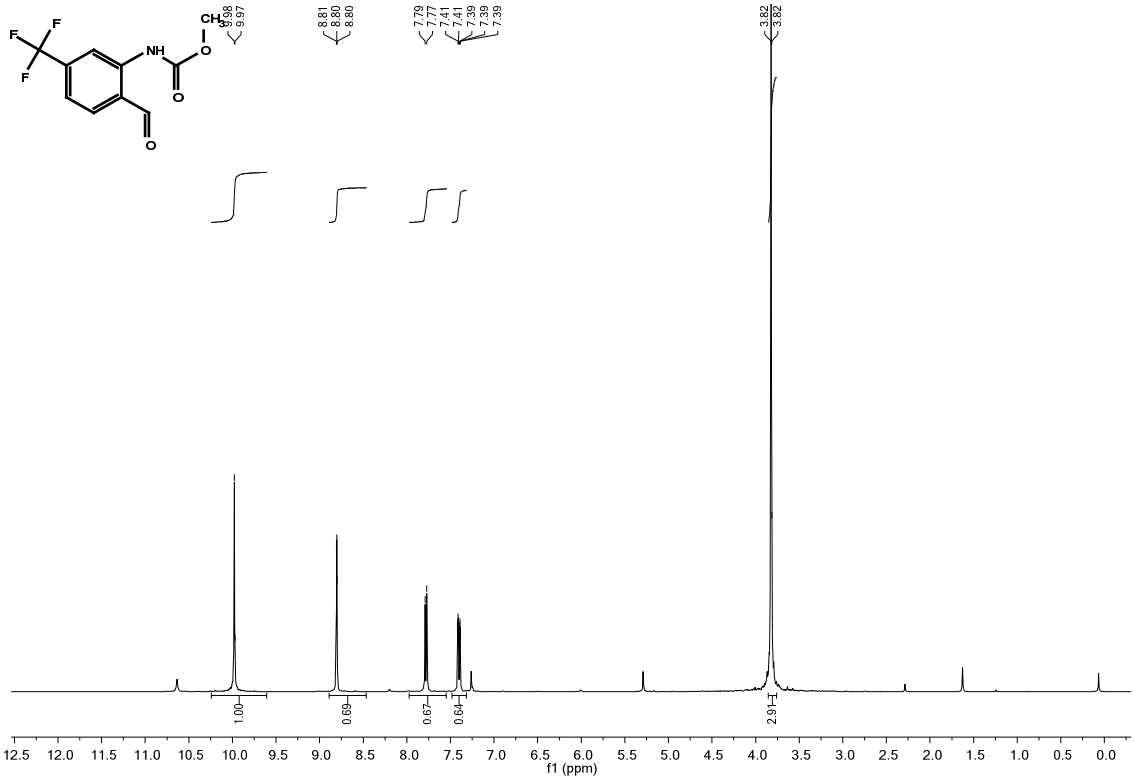
II. NMR spectra of new compounds



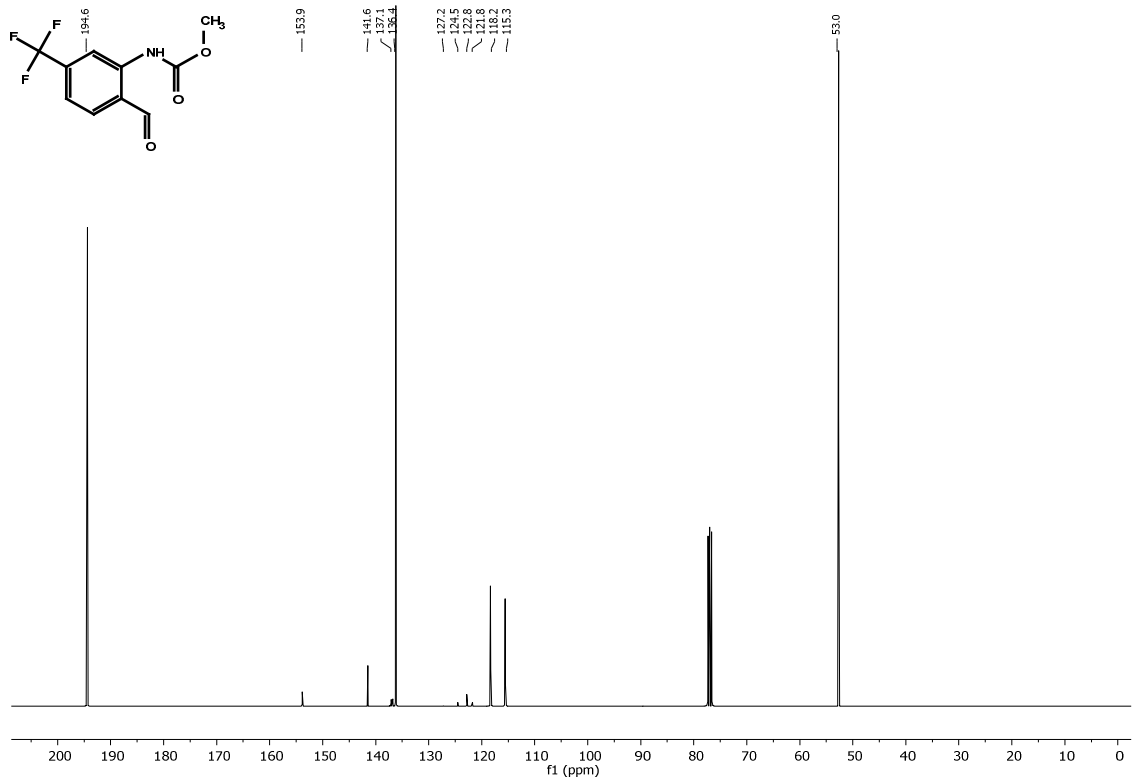
^{1}H NMR spectrum of **1b** (400 MHz, 25 °C, CD_3Cl)



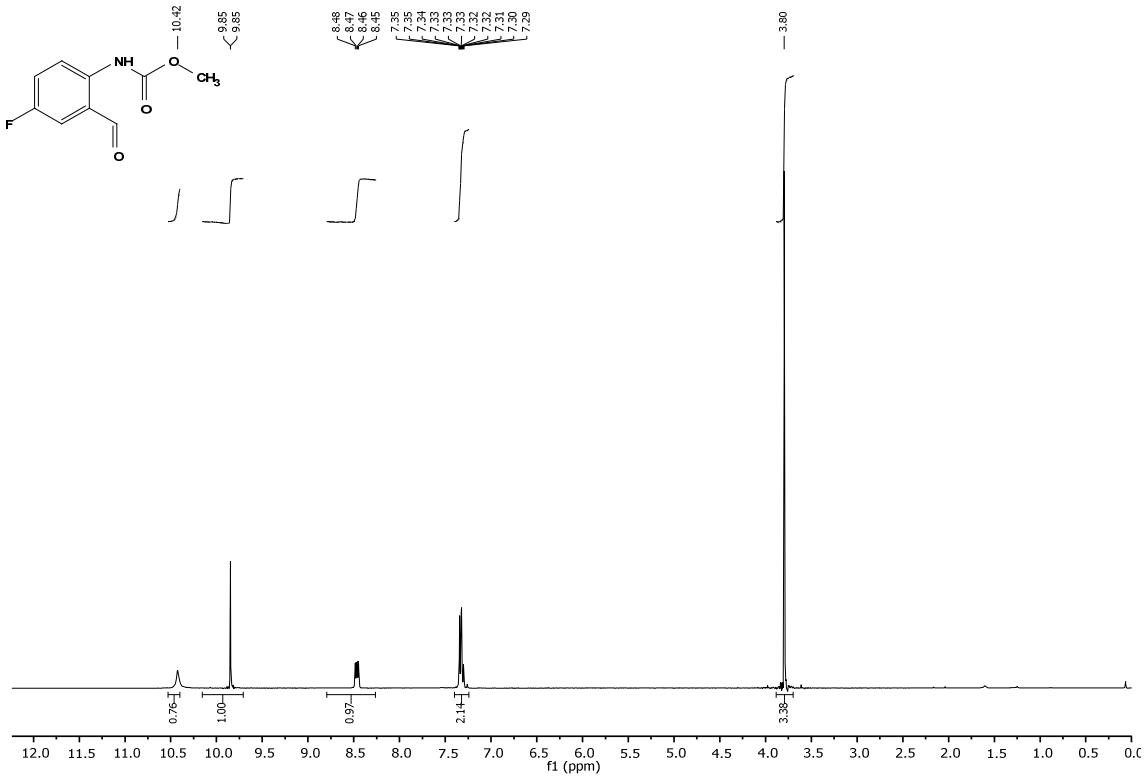
^{13}C NMR spectrum of **1b** (100 MHz, 25 °C, CD_3Cl)



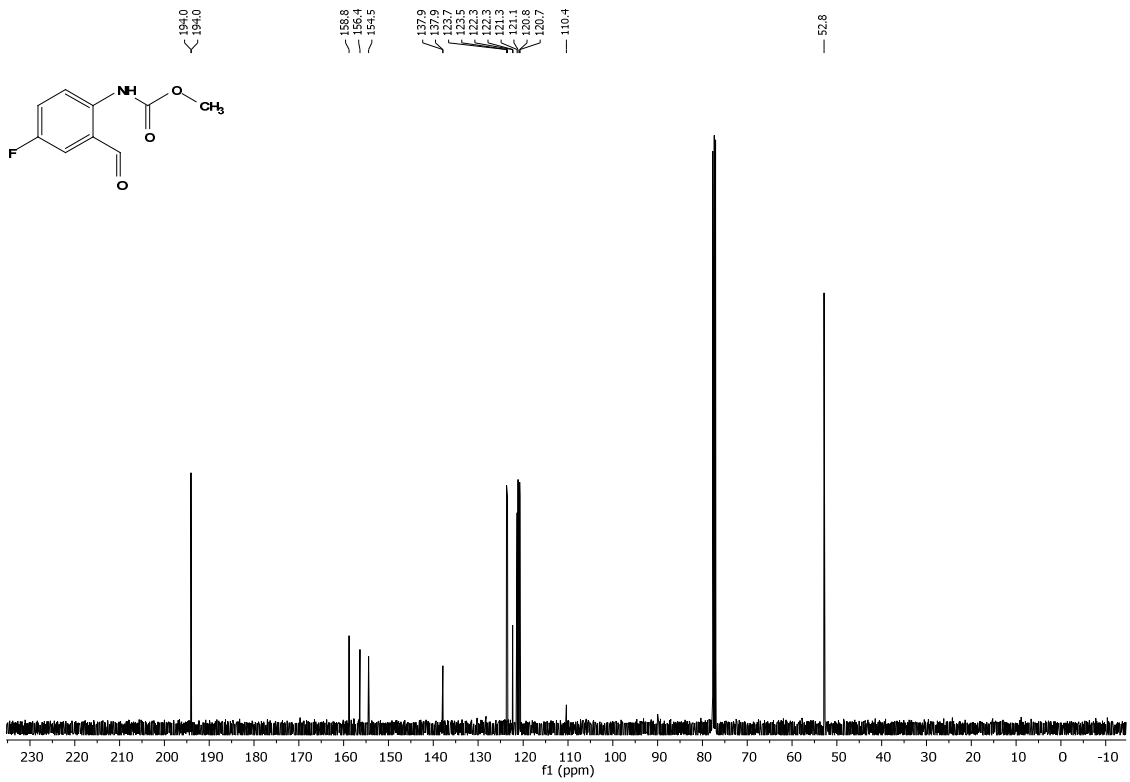
^{1}H NMR spectrum of **1c** (400 MHz, 25 °C, CD_3Cl)



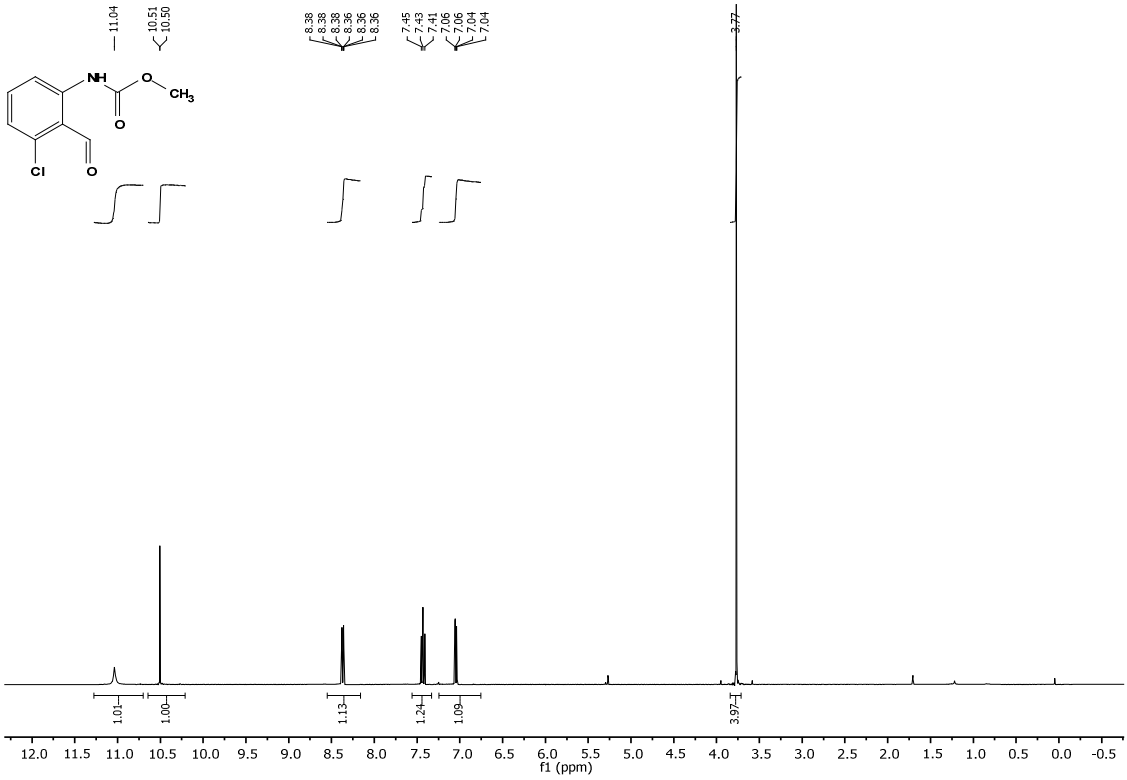
^{13}C NMR spectrum of **1c** (100 MHz, 25 °C, CD_3Cl)



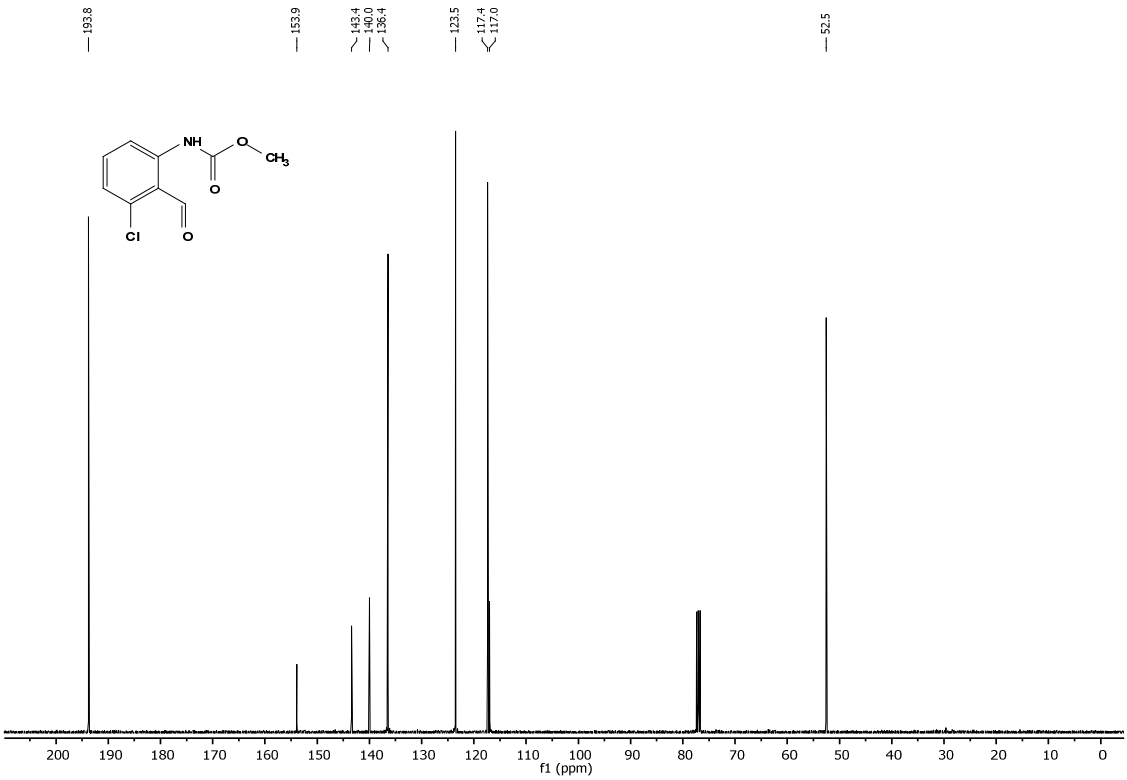
^{1}H NMR spectrum of **1d** (400 MHz, 25 °C, CD_3Cl)



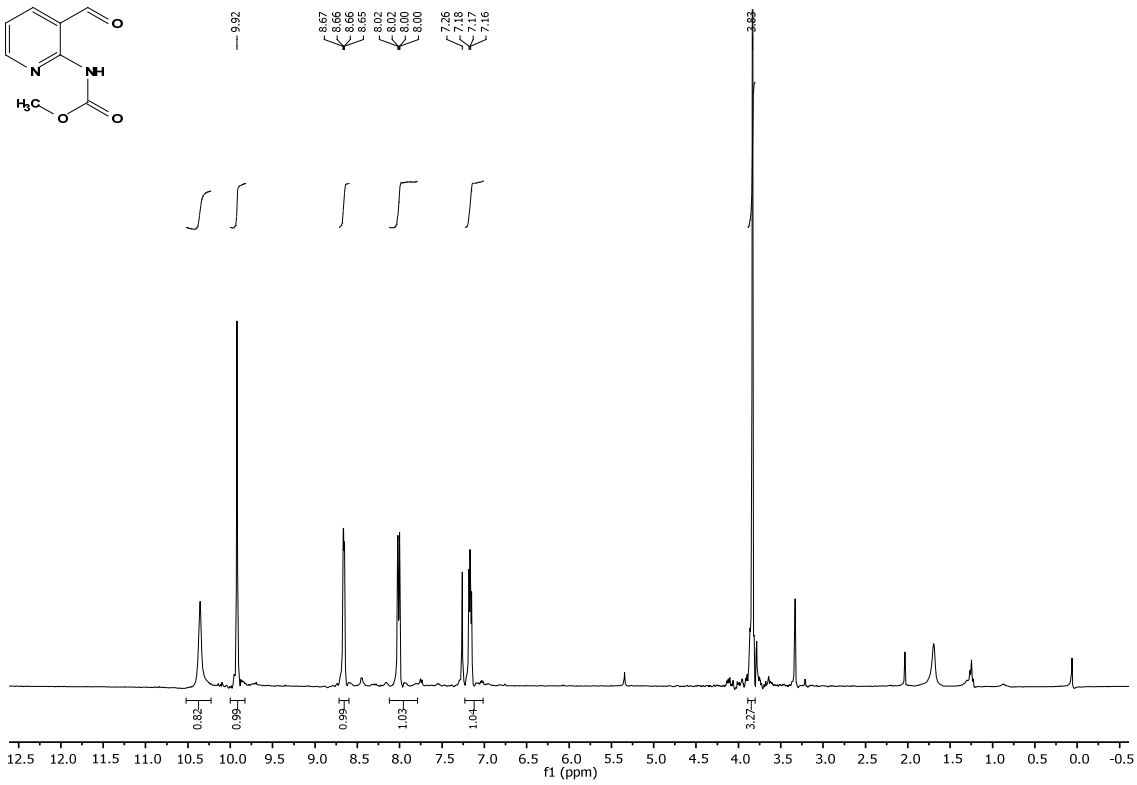
^{13}C NMR spectrum of **1d** (100 MHz, 25 °C, CD_3Cl)



^{1}H NMR spectrum of **1e** (400 MHz, 25 °C, CD_3Cl)



^{13}C NMR spectrum of **1e** (100 MHz, 25 °C, CD_3Cl)



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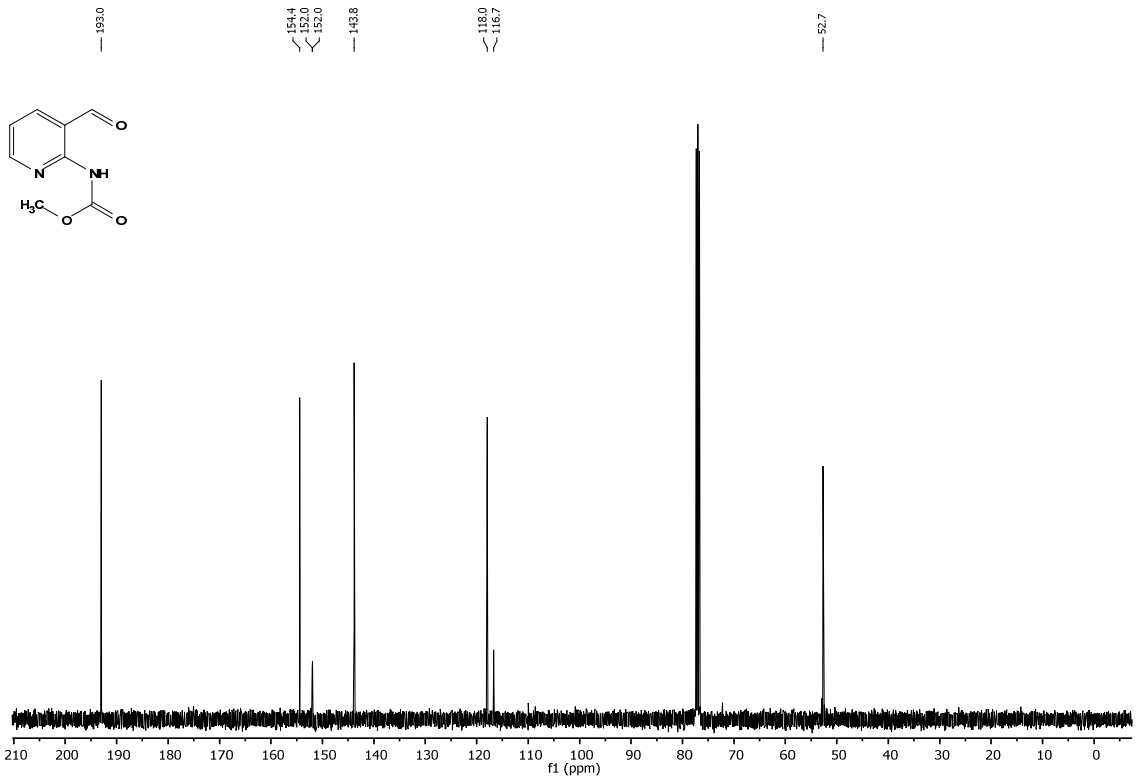
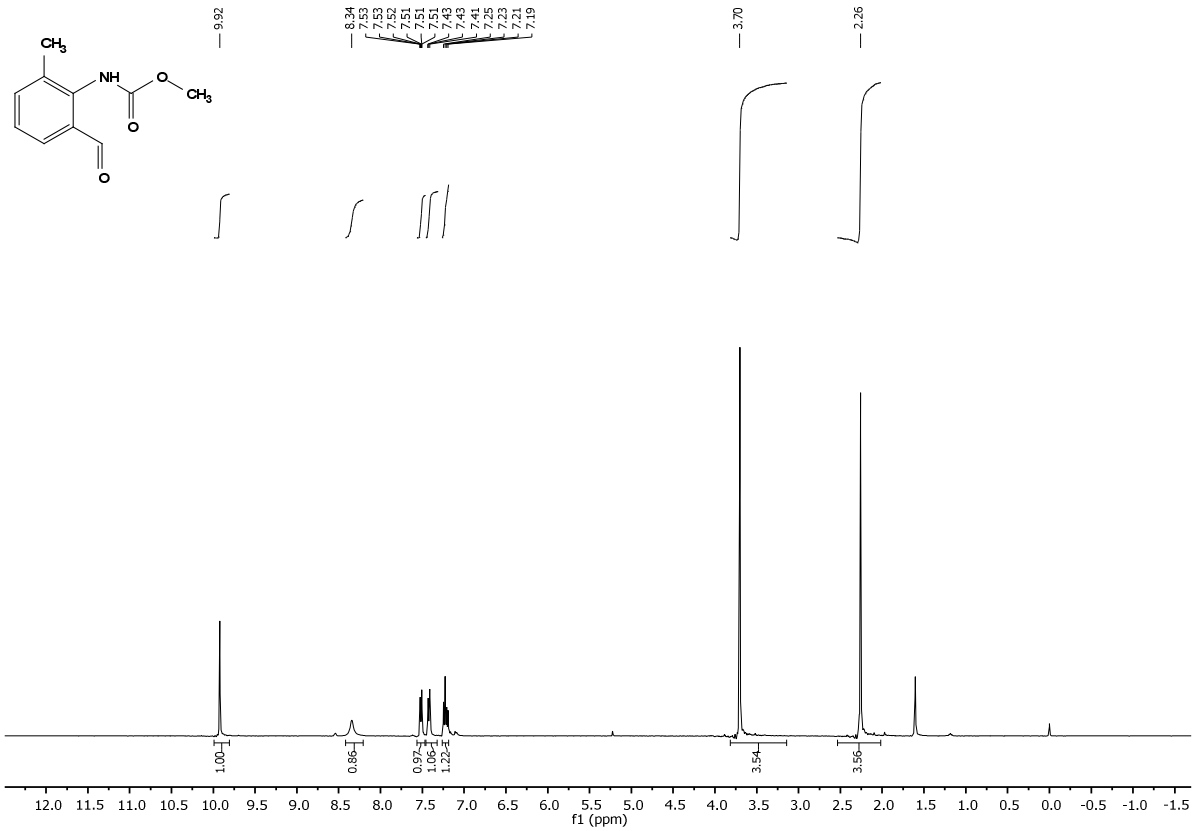
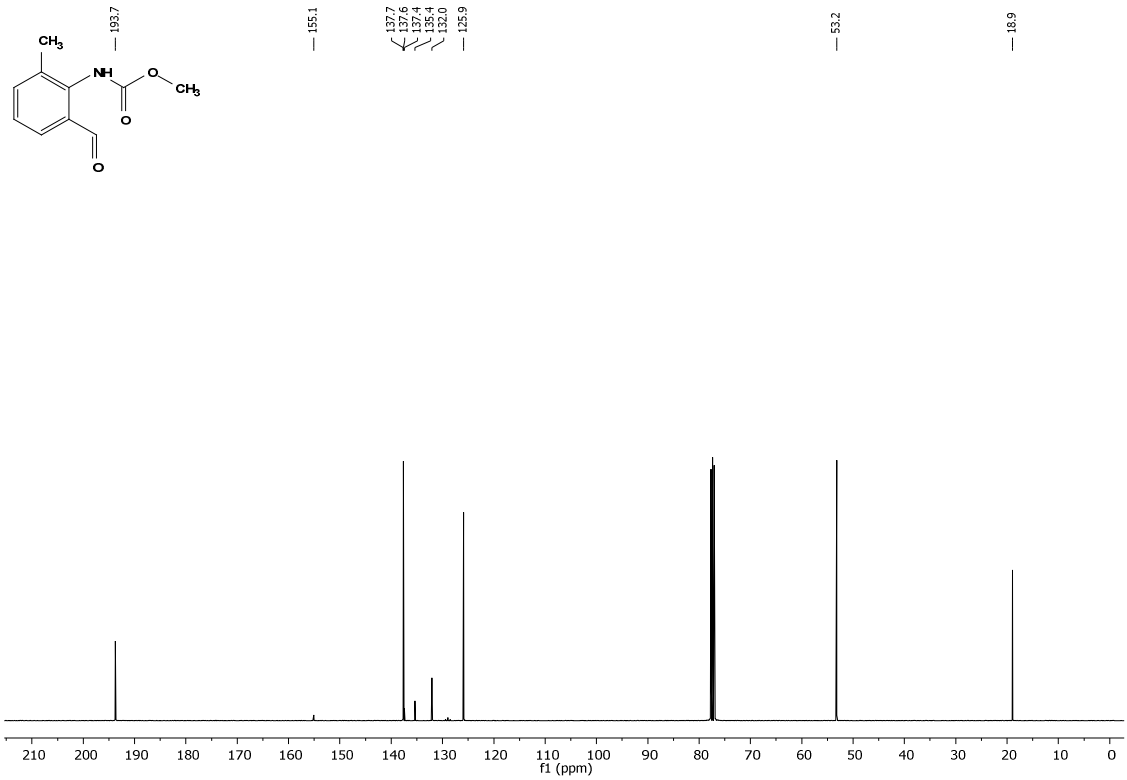


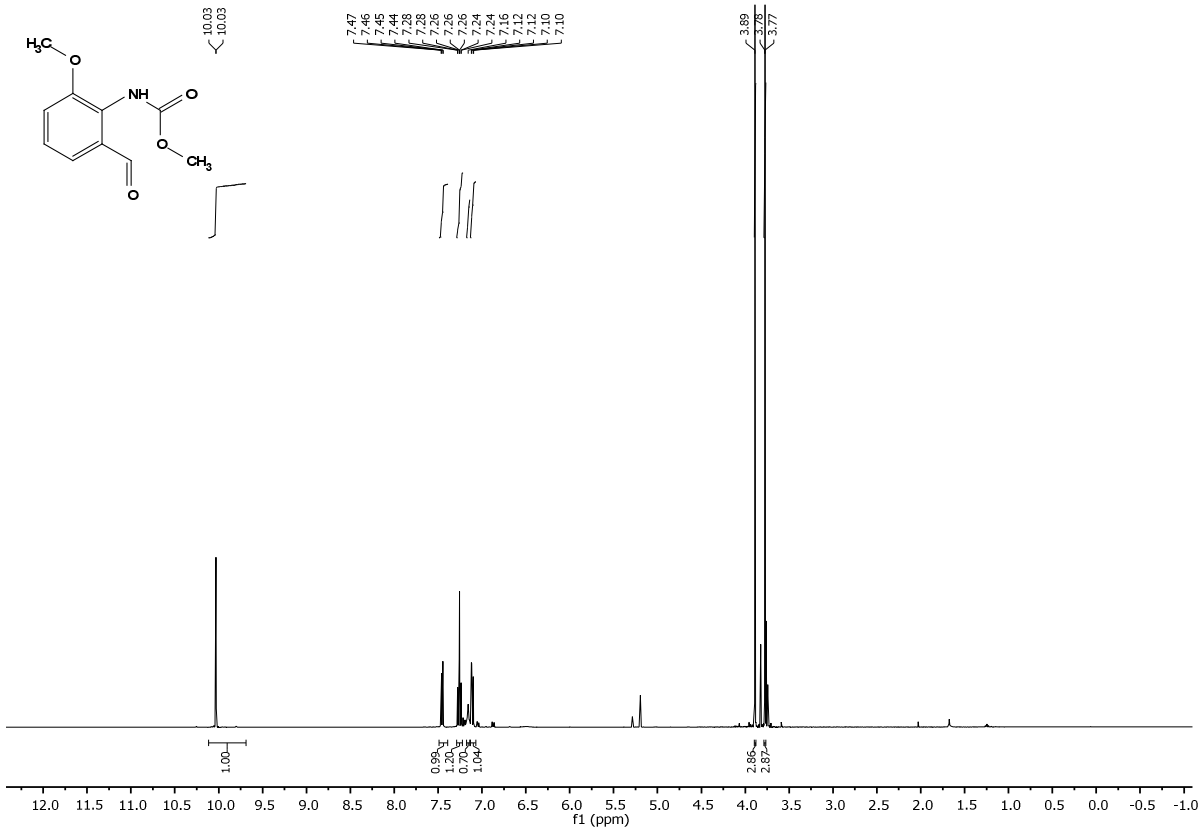
^{13}C NMR spectrum of **1f** (100 MHz, 25 °C, CD_3Cl)



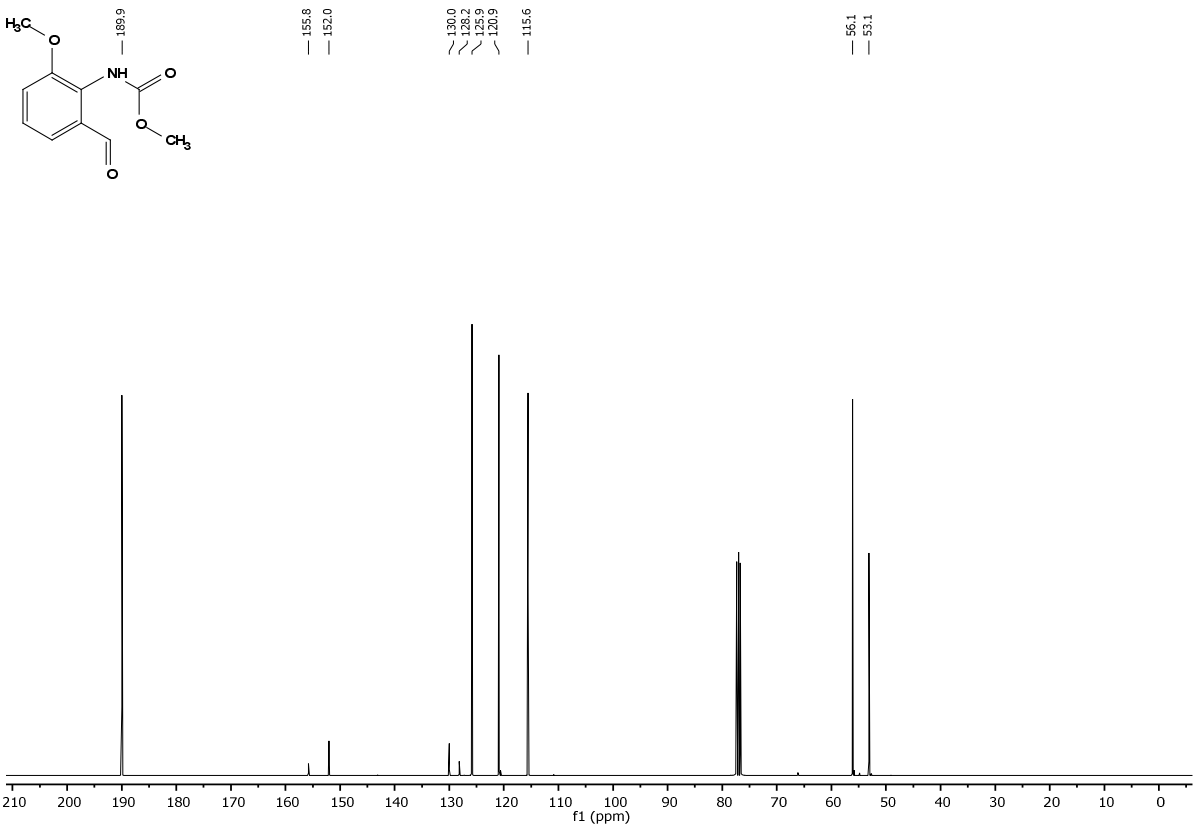
^{1}H NMR spectrum of **1g** (400 MHz, 25 °C, CD_3Cl)



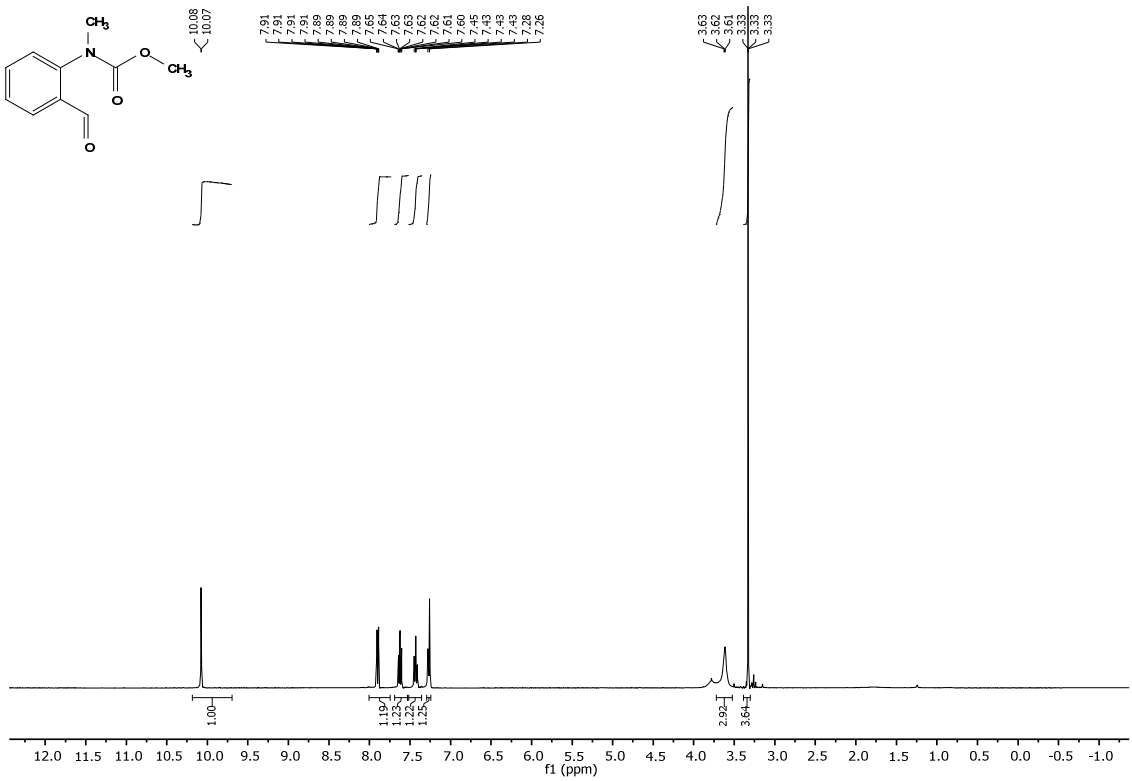
^{13}C NMR spectrum of **1g** (100 MHz, 25 °C, CD_3Cl)



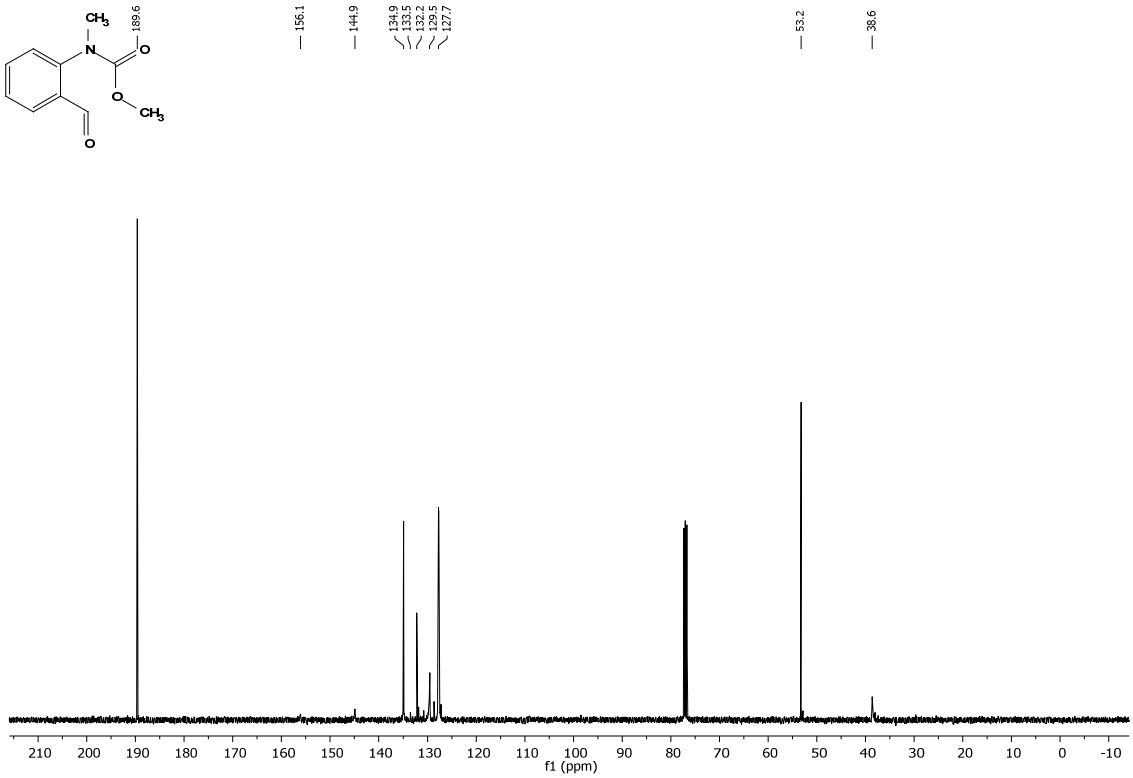
^{1}H NMR spectrum of **1h** (400 MHz, 25 °C, CD_3Cl)



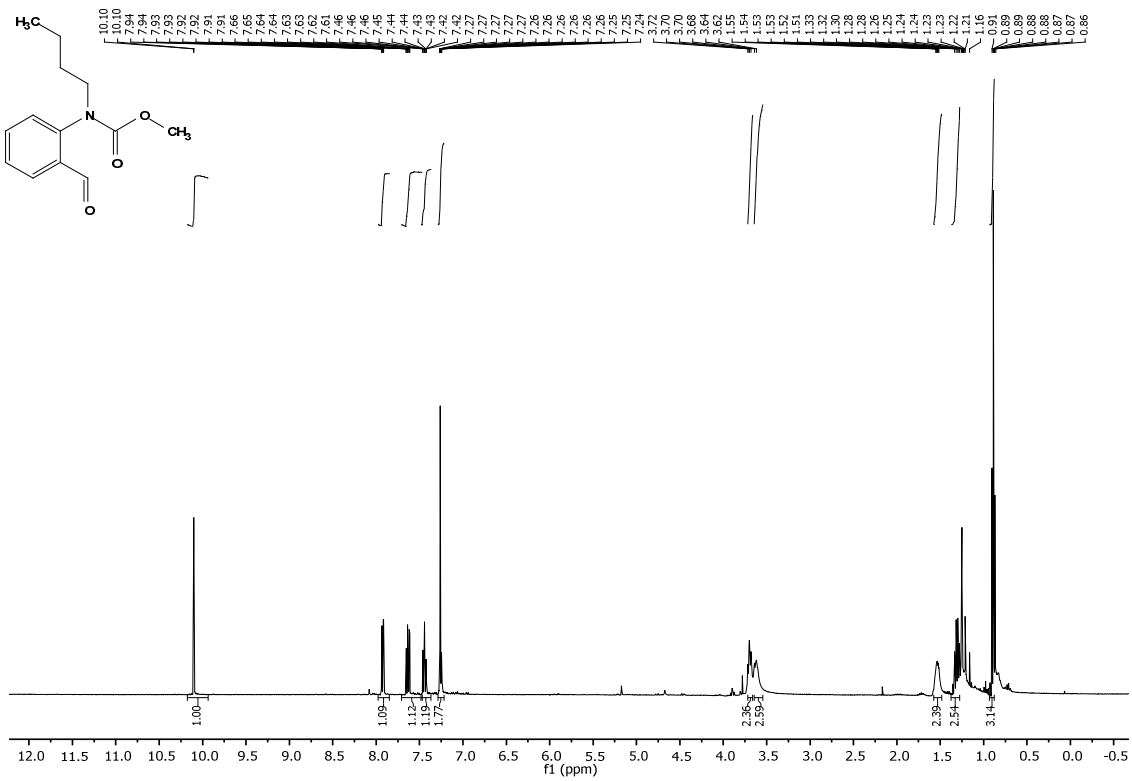
^{13}C NMR spectrum of **1h** (100 MHz, 25 °C, CD_3Cl)



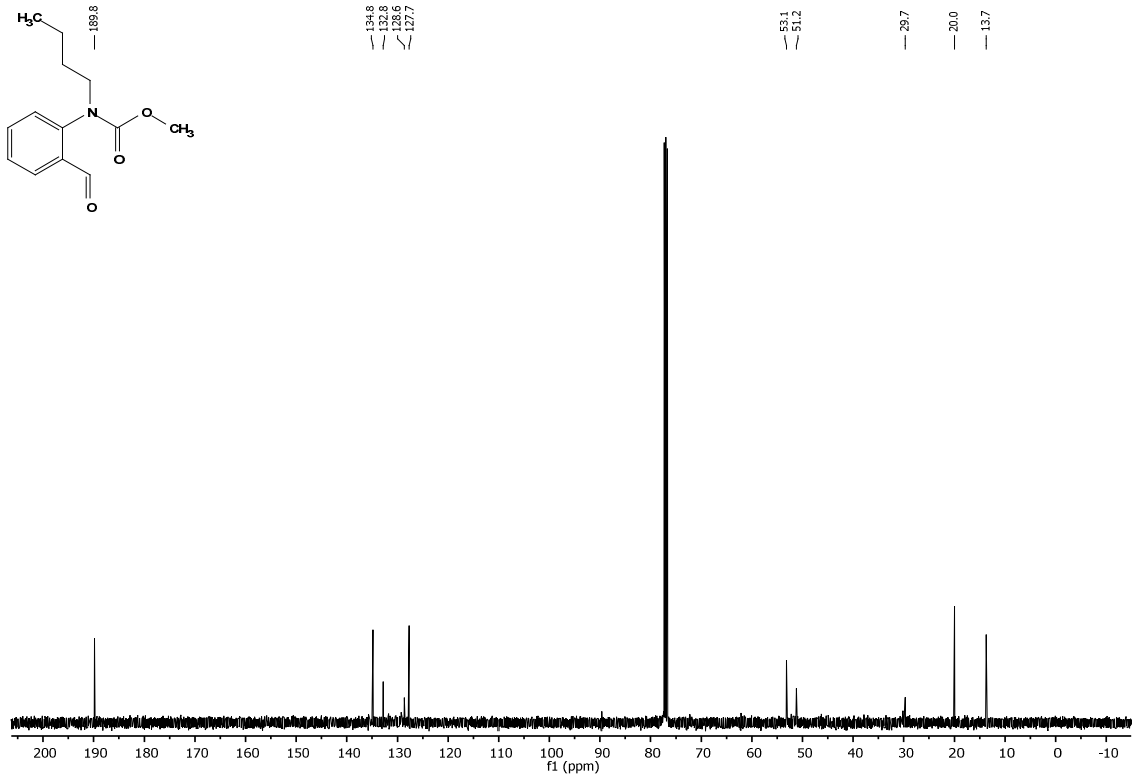
^{1}H NMR spectrum of **1i** (400 MHz, 25 °C, CD_3Cl)



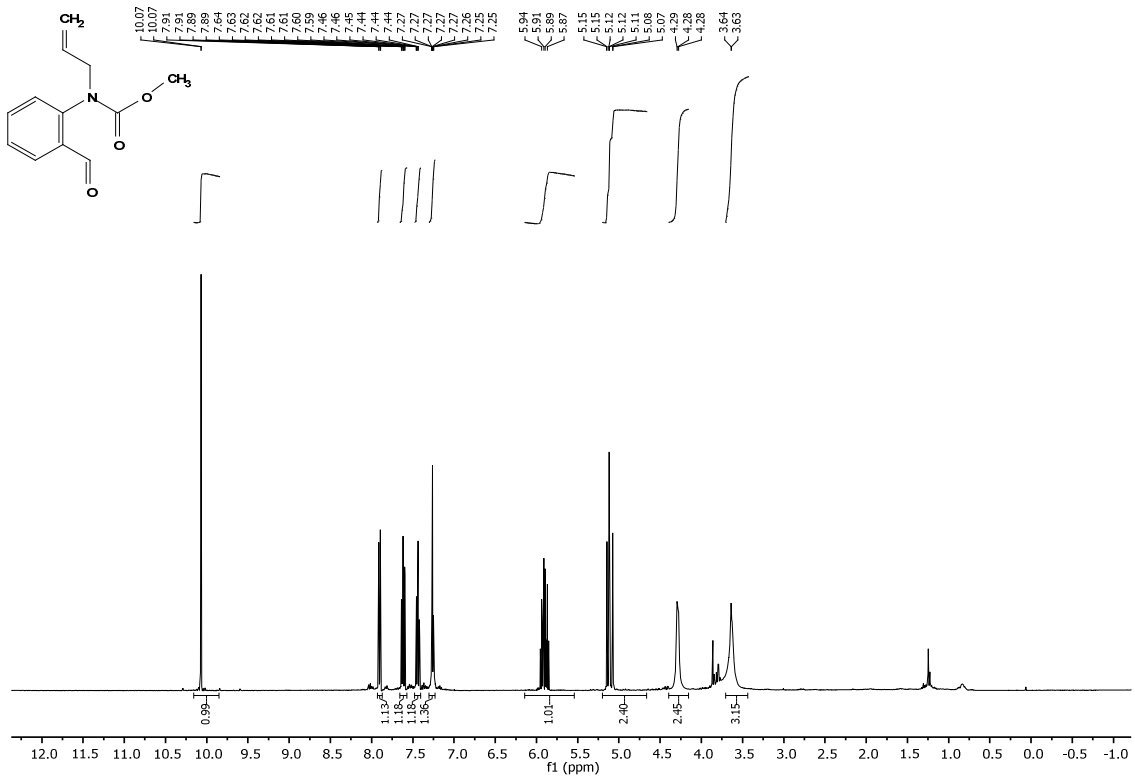
^{13}C NMR spectrum of **1i** (100 MHz, 25 °C, CD_3Cl)



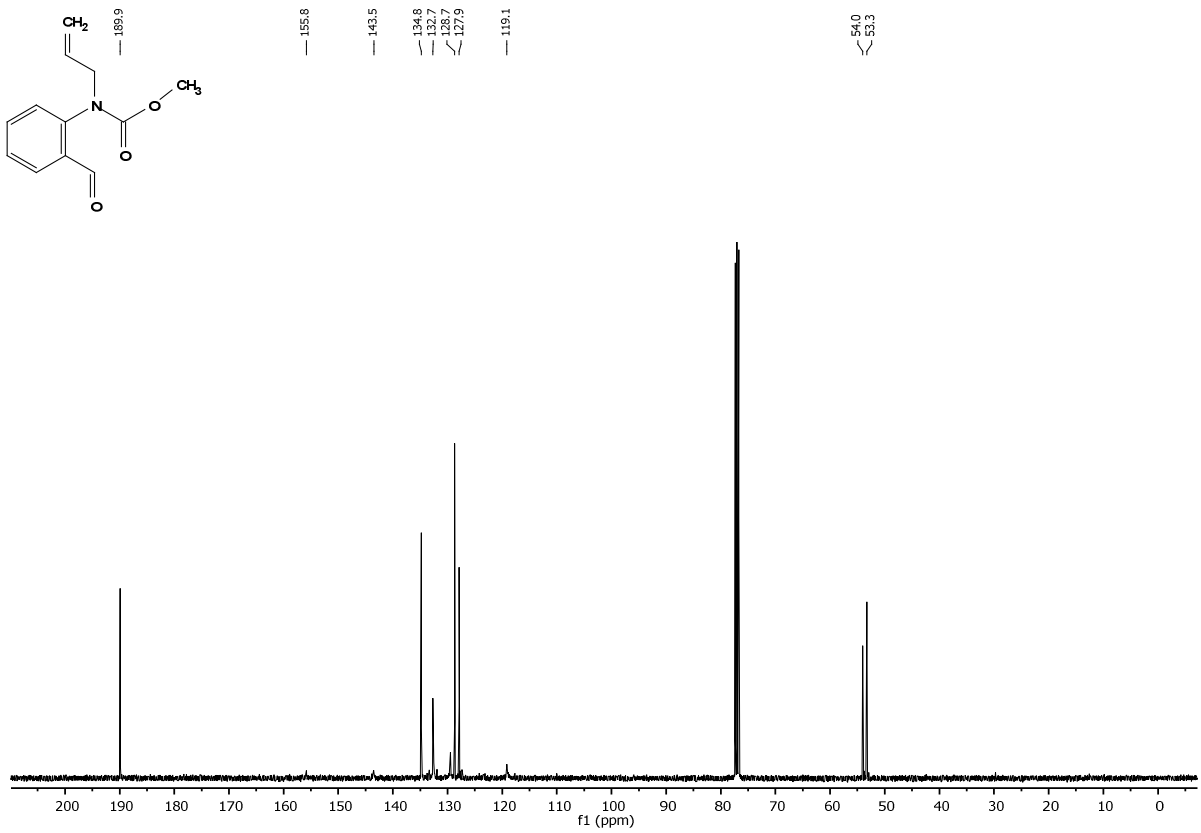
^{1}H NMR spectrum of **1j** (400 MHz, 25 °C, CD_3Cl)



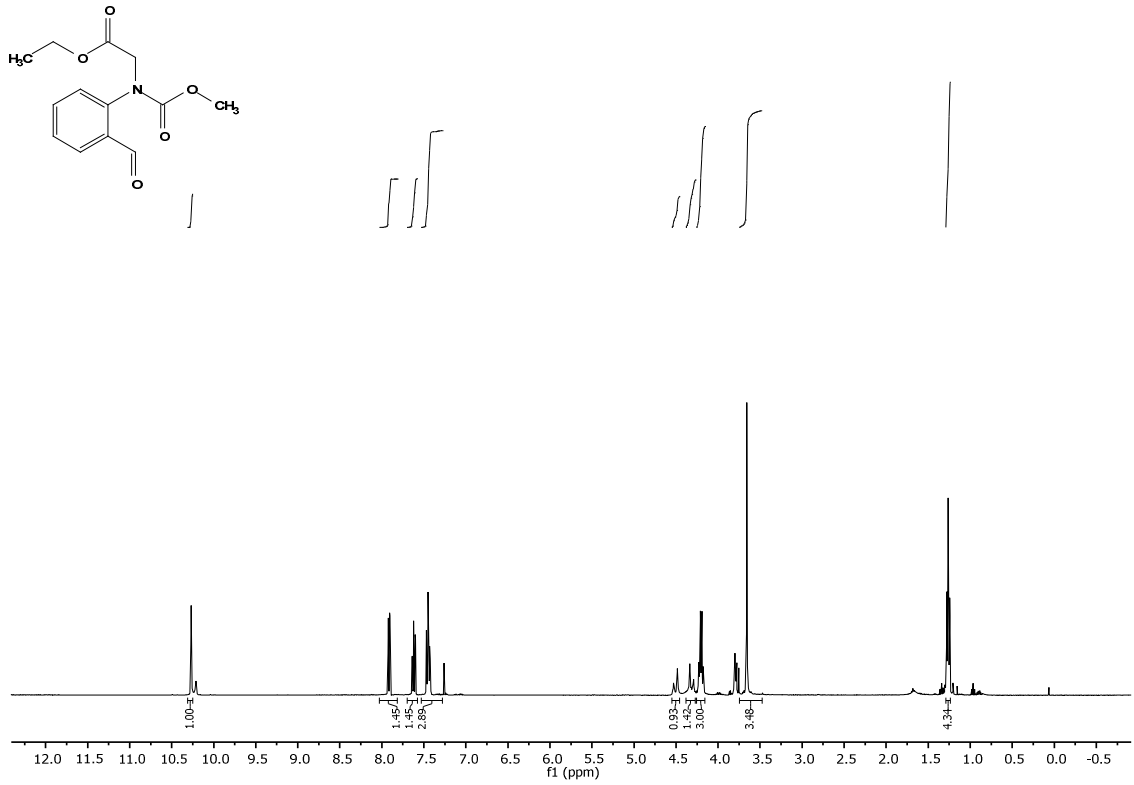
^{13}C NMR spectrum of **1j** (100 MHz, 25 °C, CD_3Cl)



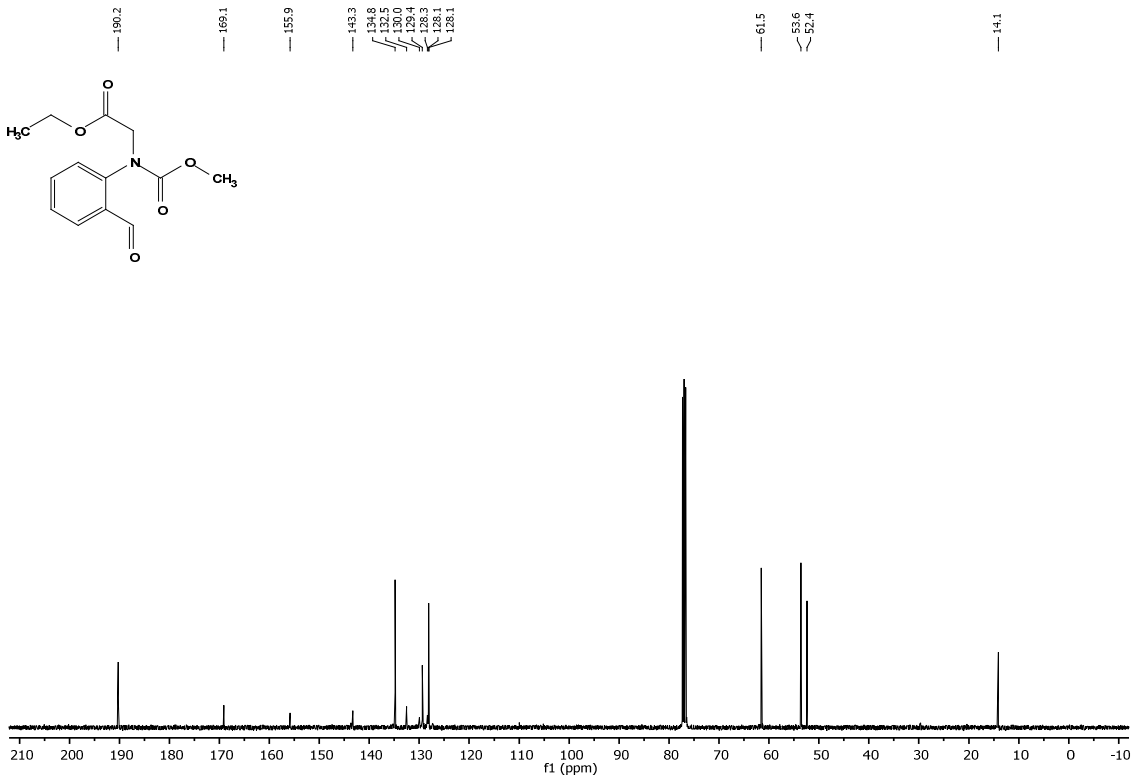
^{1}H NMR spectrum of **1k** (400 MHz, 25 °C, CD_3Cl)



^{13}C NMR spectrum of **1k** (100 MHz, 25 °C, CD_3Cl)



^{1}H NMR spectrum of **1l** (400 MHz, 25 °C, CD_3Cl)



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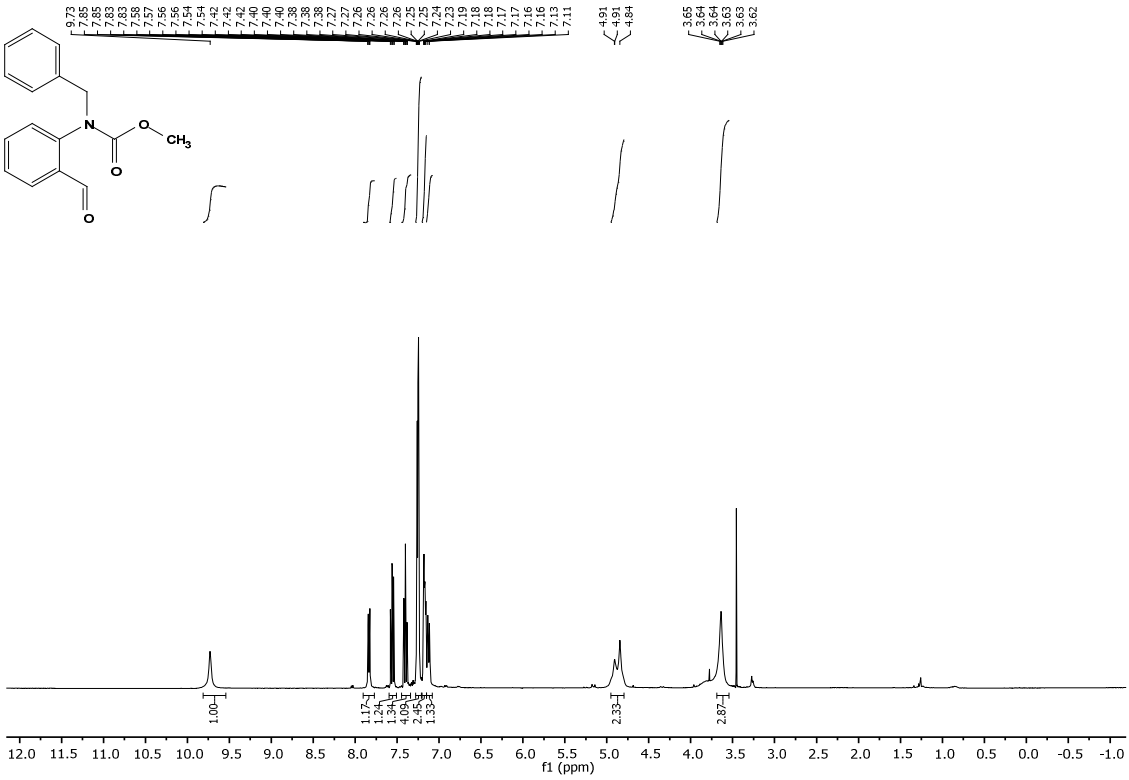
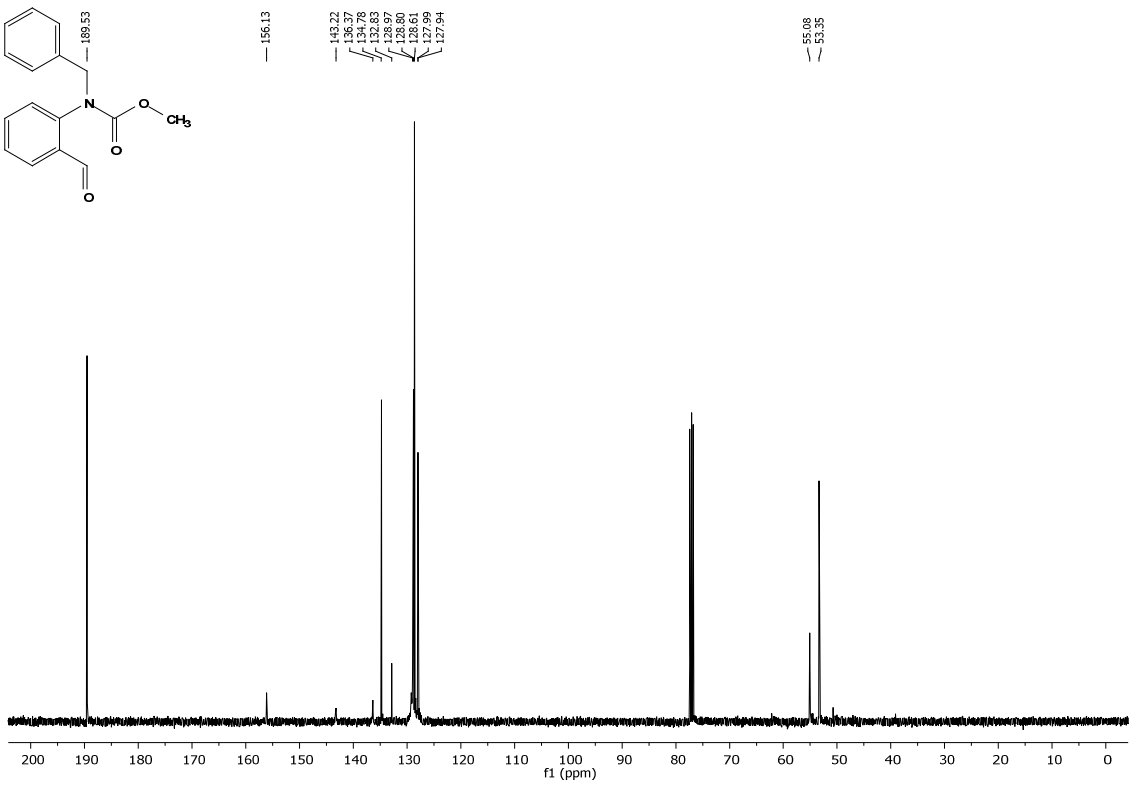
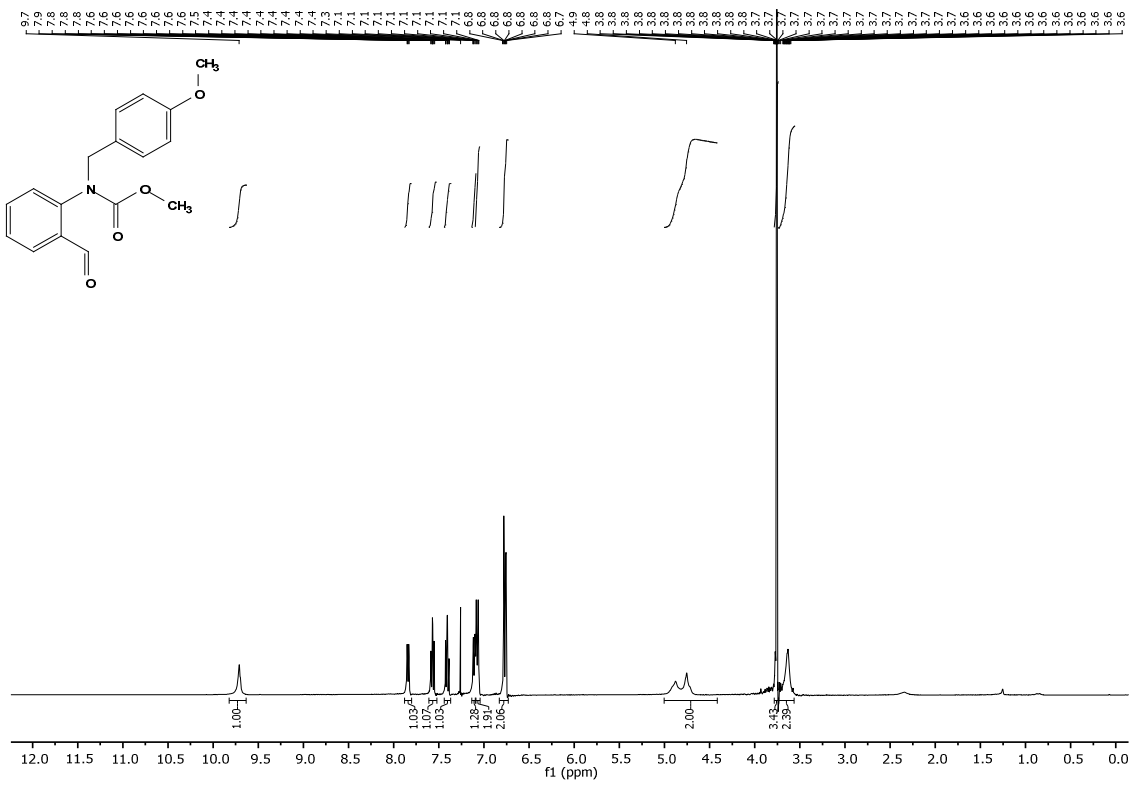


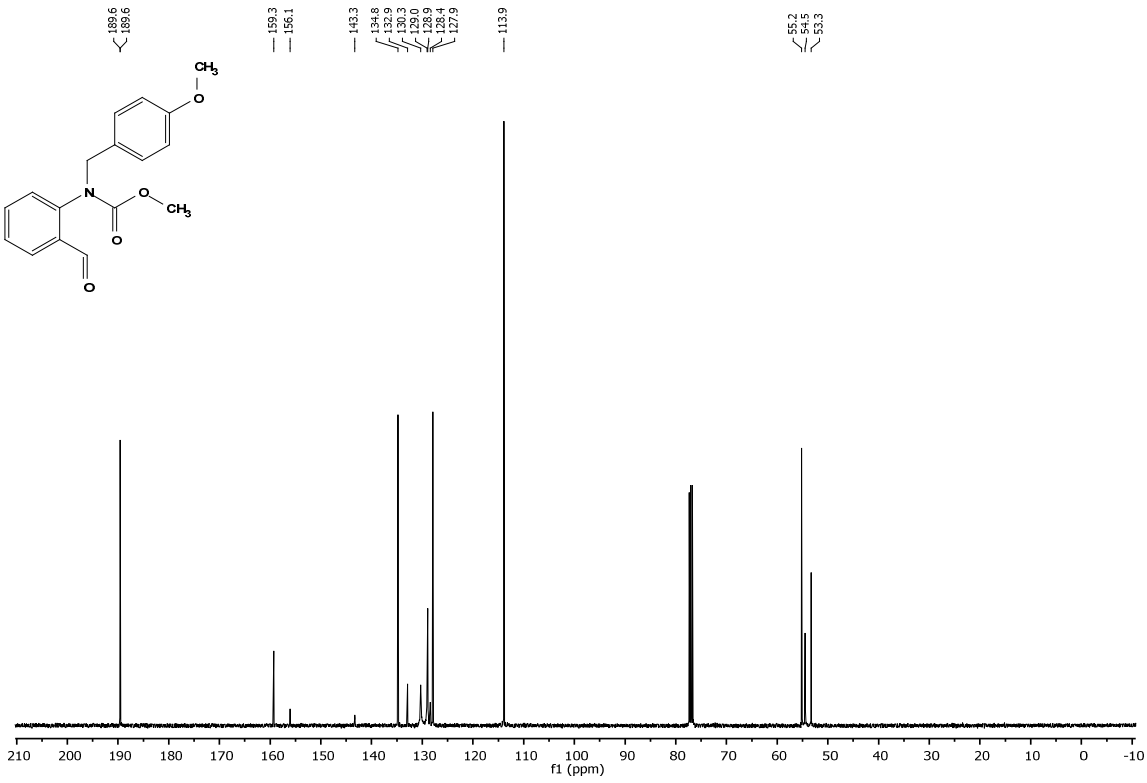
^{1}H NMR spectrum of **1m** (400 MHz, 25 °C, CD_3Cl)



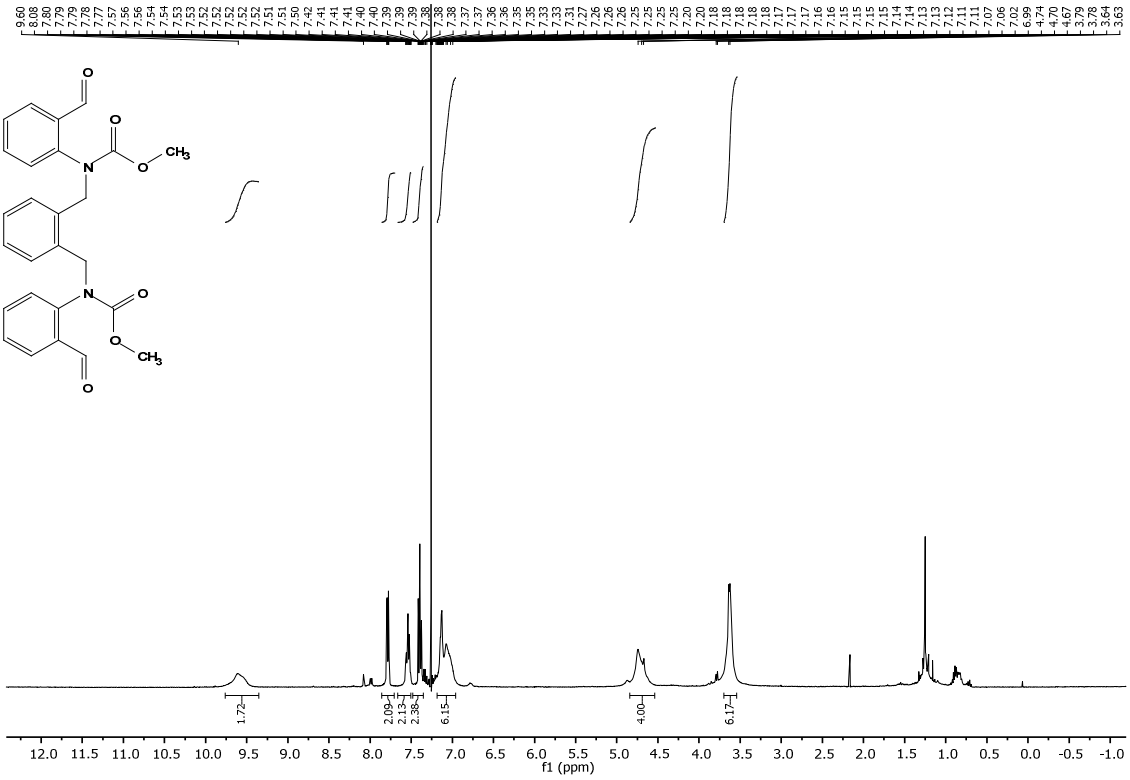
^{13}C NMR spectrum of **1m** (100 MHz, 25 °C, CD_3Cl)



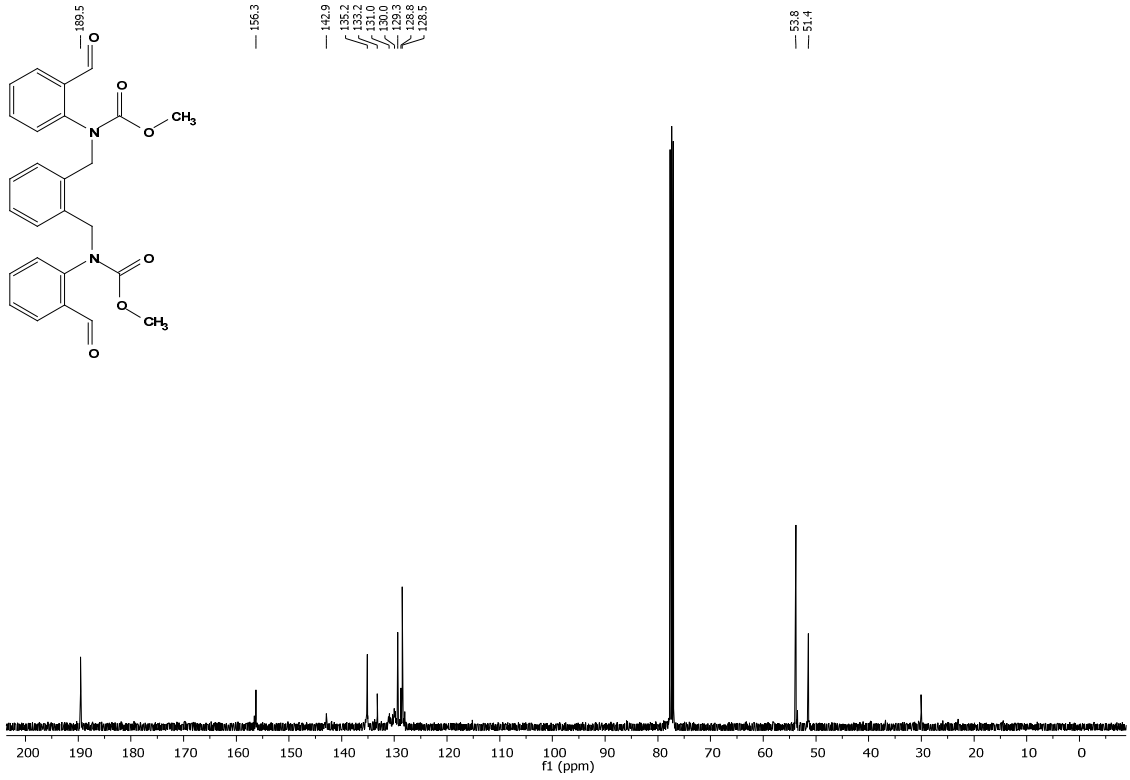
^{1}H NMR spectrum of **1n** (400 MHz, 25 °C, CD_3Cl)



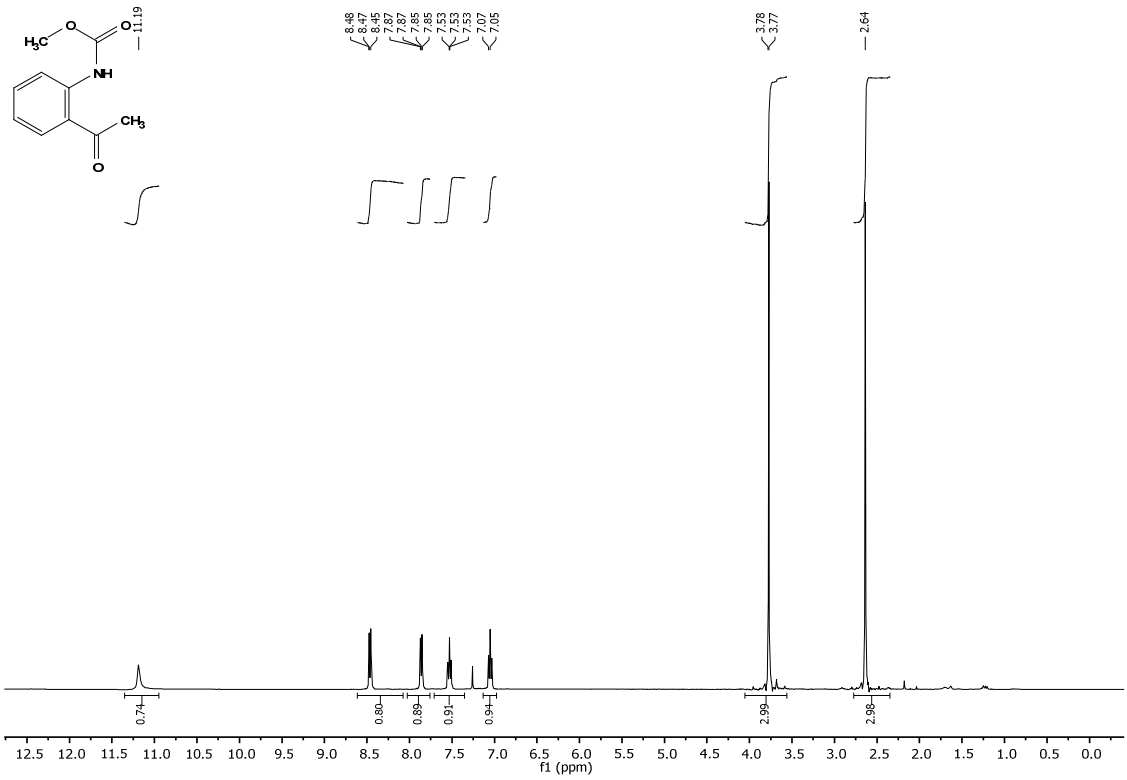
^{13}C NMR spectrum of **1n** (100 MHz, 25 °C, CD_3Cl)



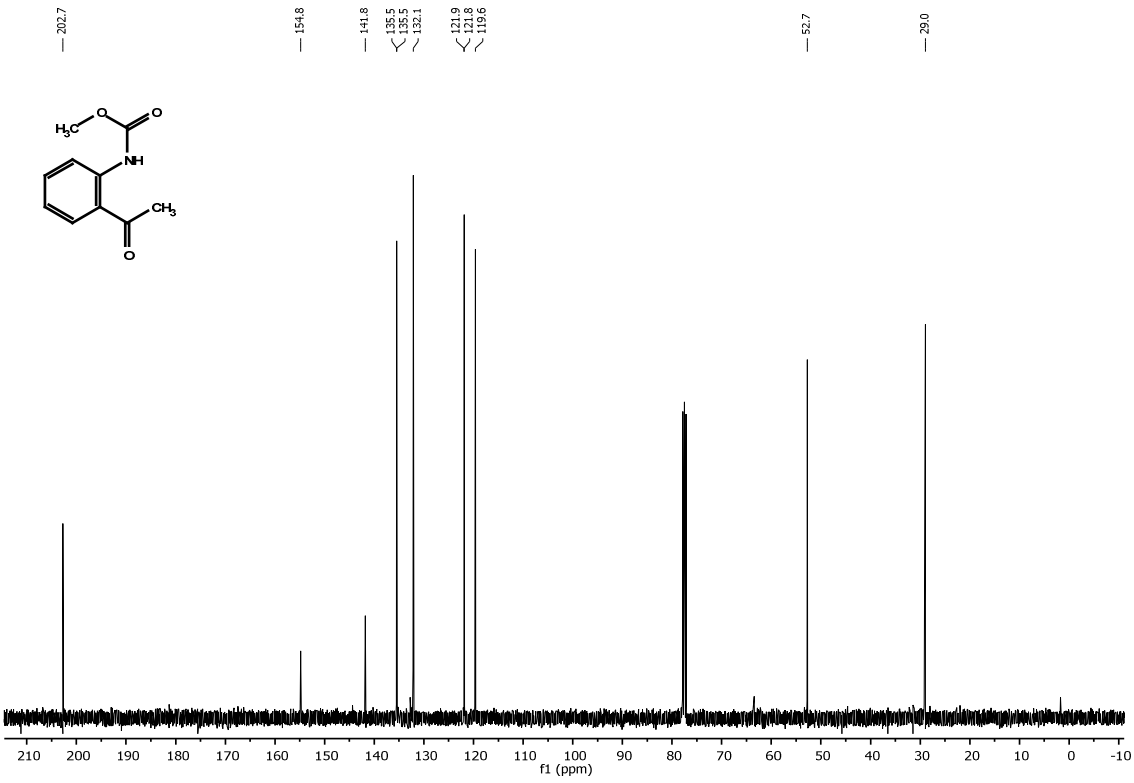
^{1}H NMR spectrum of **1o** (400 MHz, 25 °C, CD_3Cl)



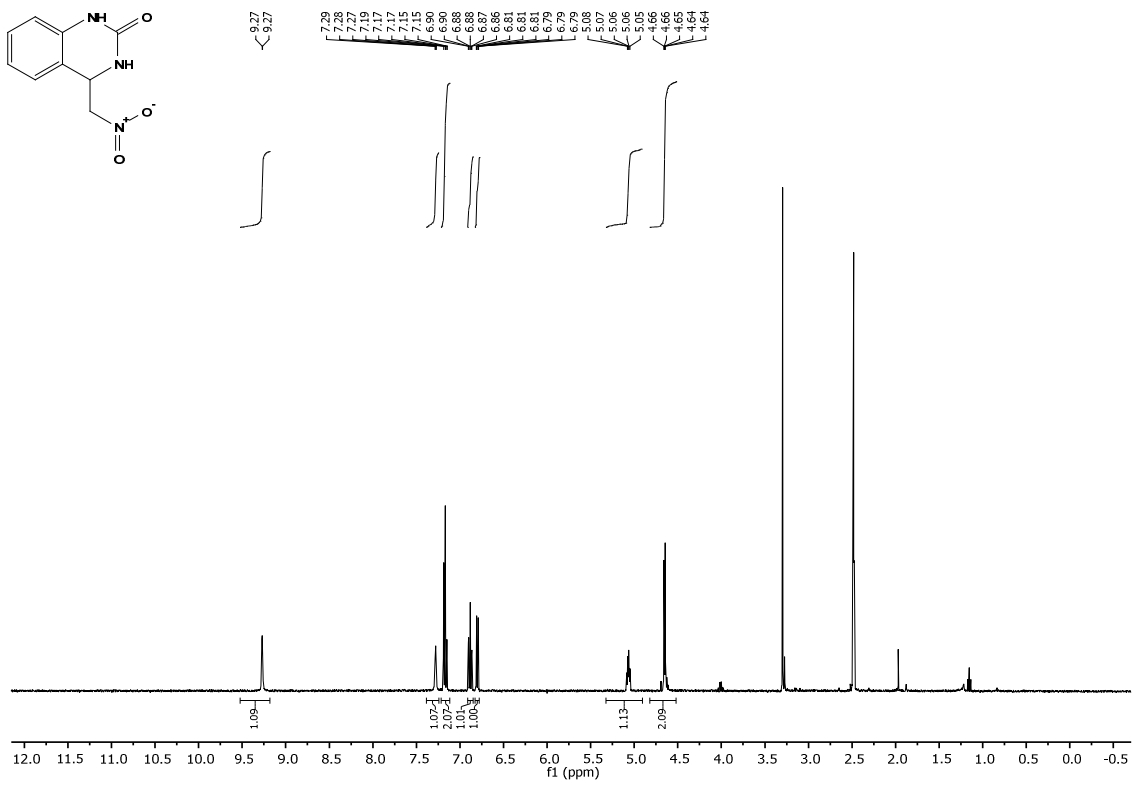
^{13}C NMR spectrum of **1o** (100 MHz, 25 °C, CD_3Cl)



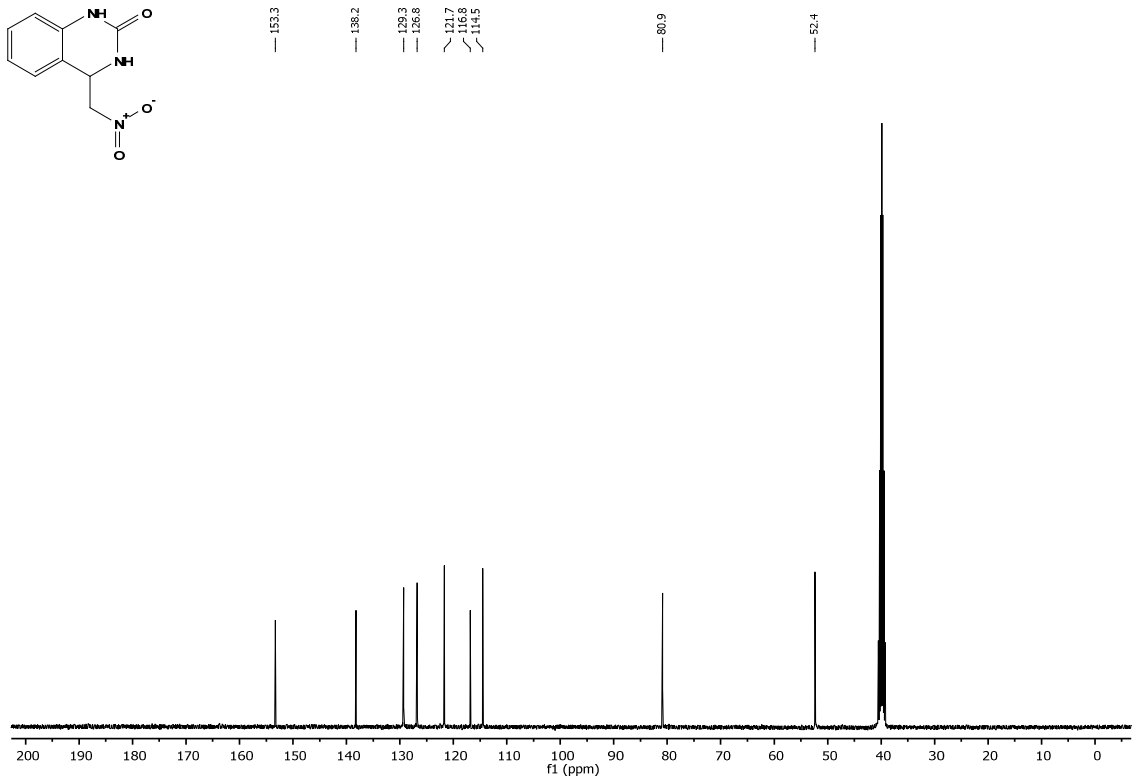
^{1}H NMR spectrum of **1p** (400 MHz, 25 °C, CD_3Cl)



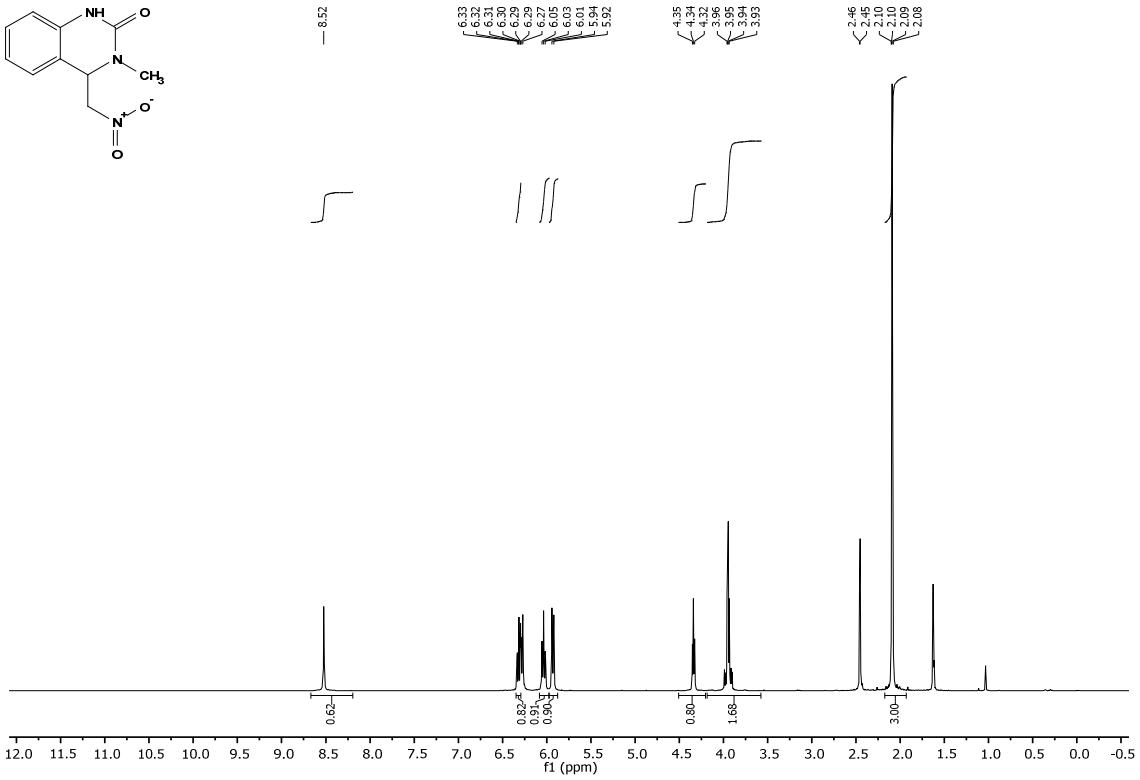
^{13}C NMR spectrum of **1p** (100 MHz, 25 °C, CD_3Cl)



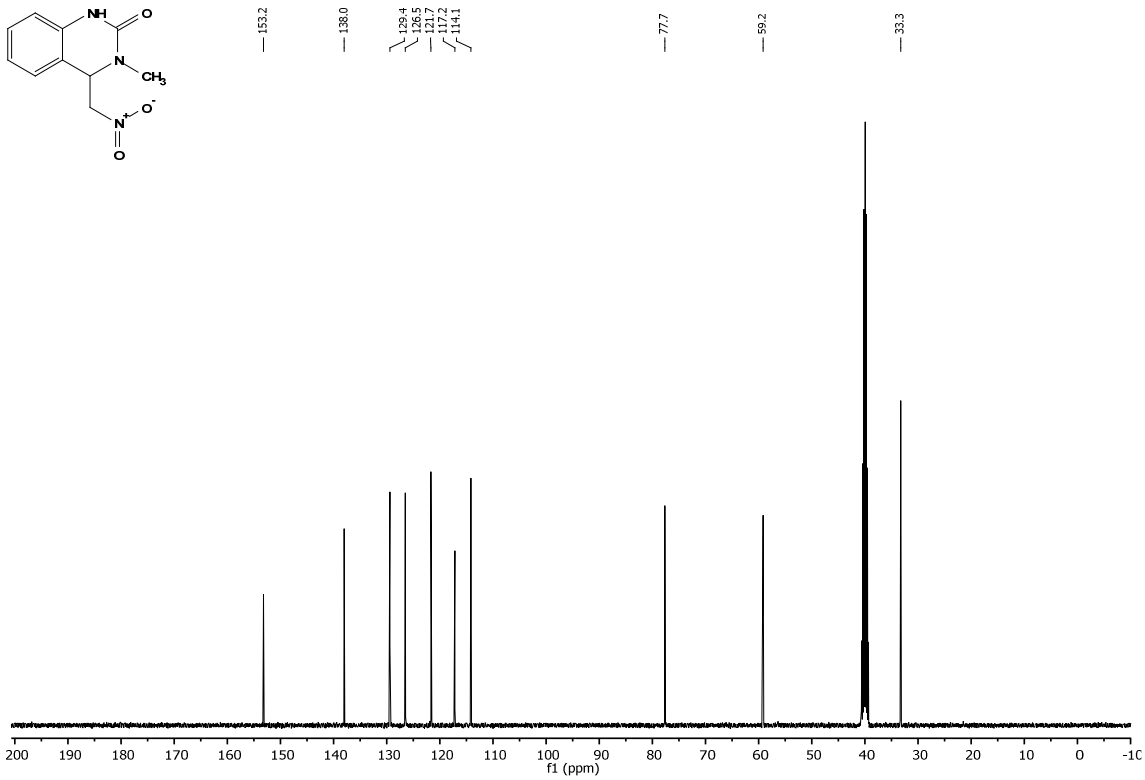
^{1}H NMR spectrum of **2a** (400 MHz, 25 °C, DMSO-d_6)



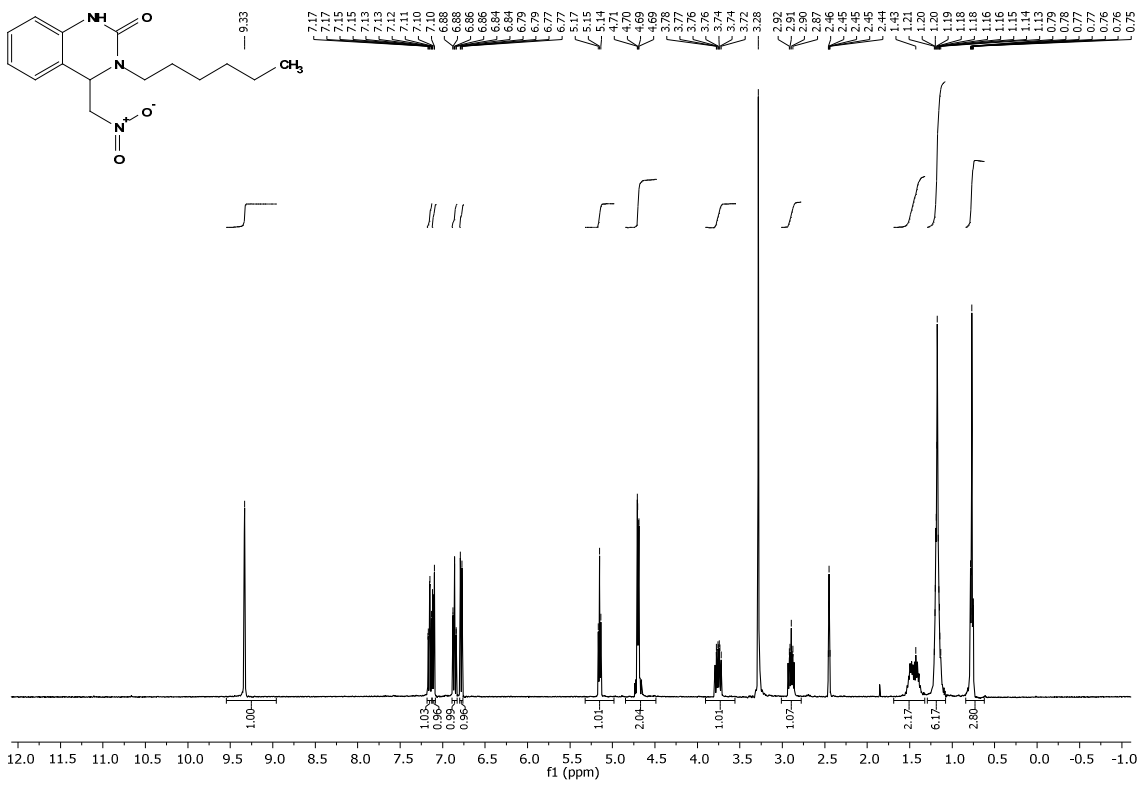
^{13}C NMR spectrum of **2a** (100 MHz, 25 °C, DMSO-d_6)



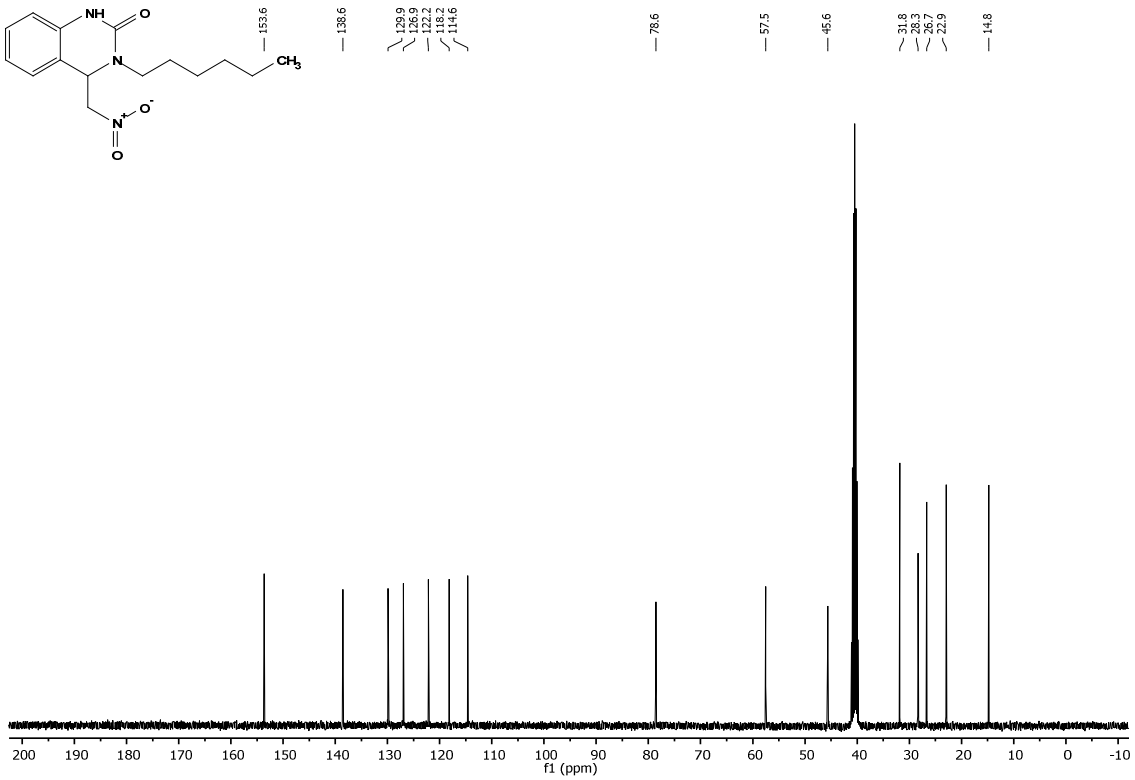
^{1}H NMR spectrum of **2b** (400 MHz, 25 °C, DMSO-d_6)



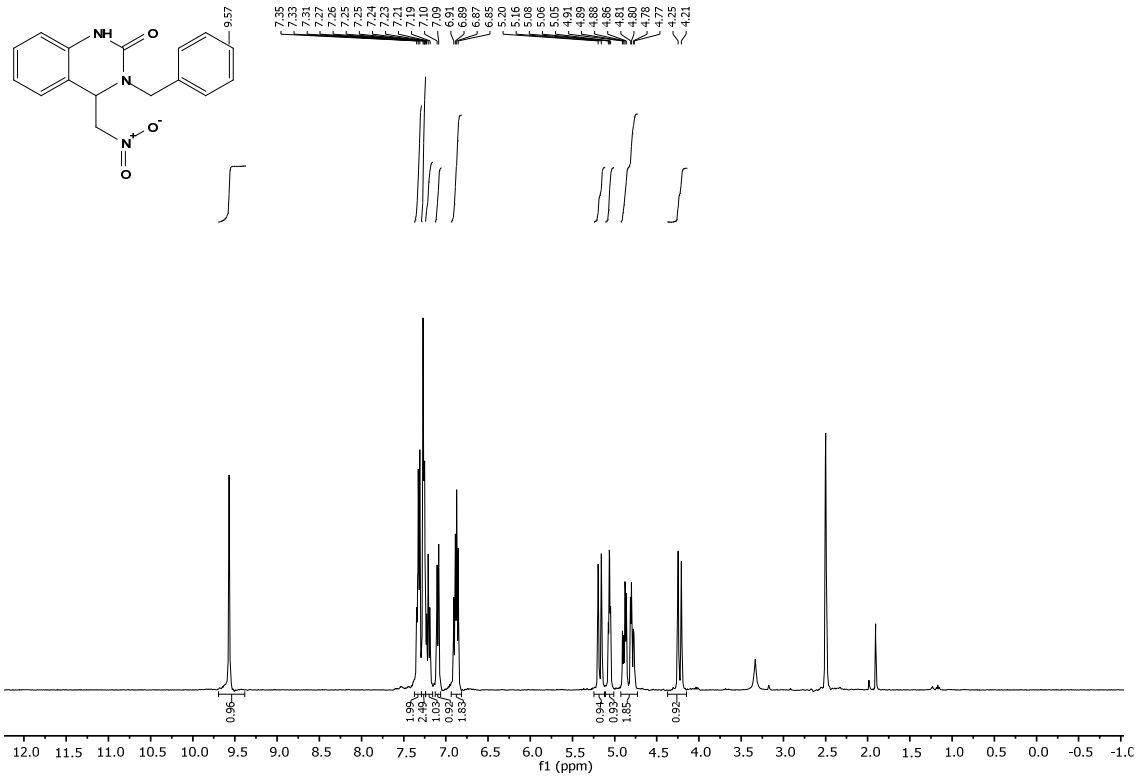
^{13}C NMR spectrum of **2b** (100 MHz, 25 °C, DMSO-d_6)



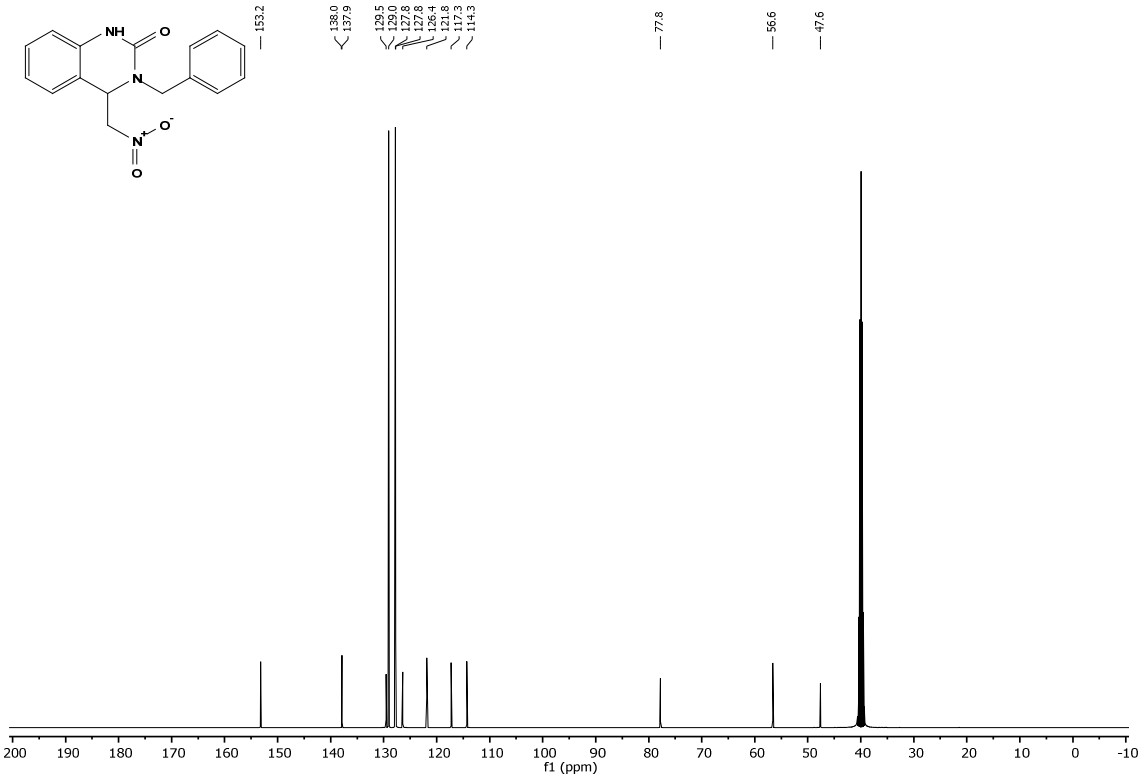
^{1}H NMR spectrum of **2c** (400 MHz, 25 °C, DMSO-d_6)



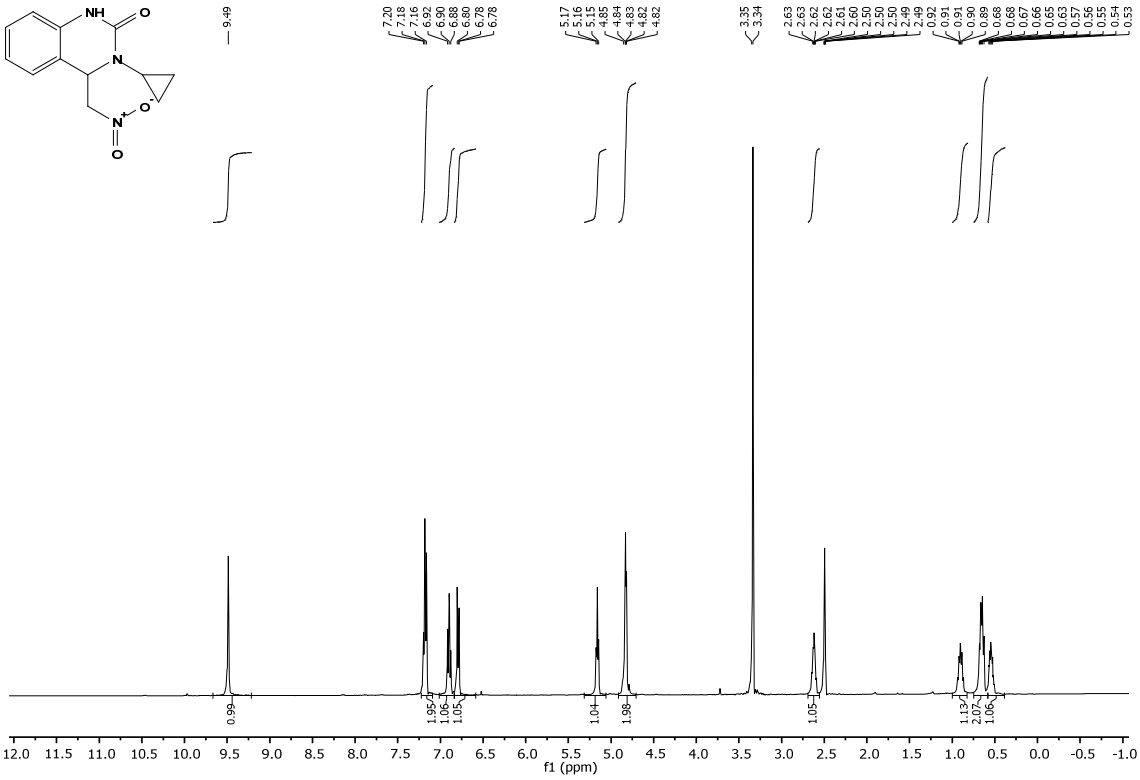
^{13}C NMR spectrum of **2c** (100 MHz, 25 °C, DMSO-d_6)



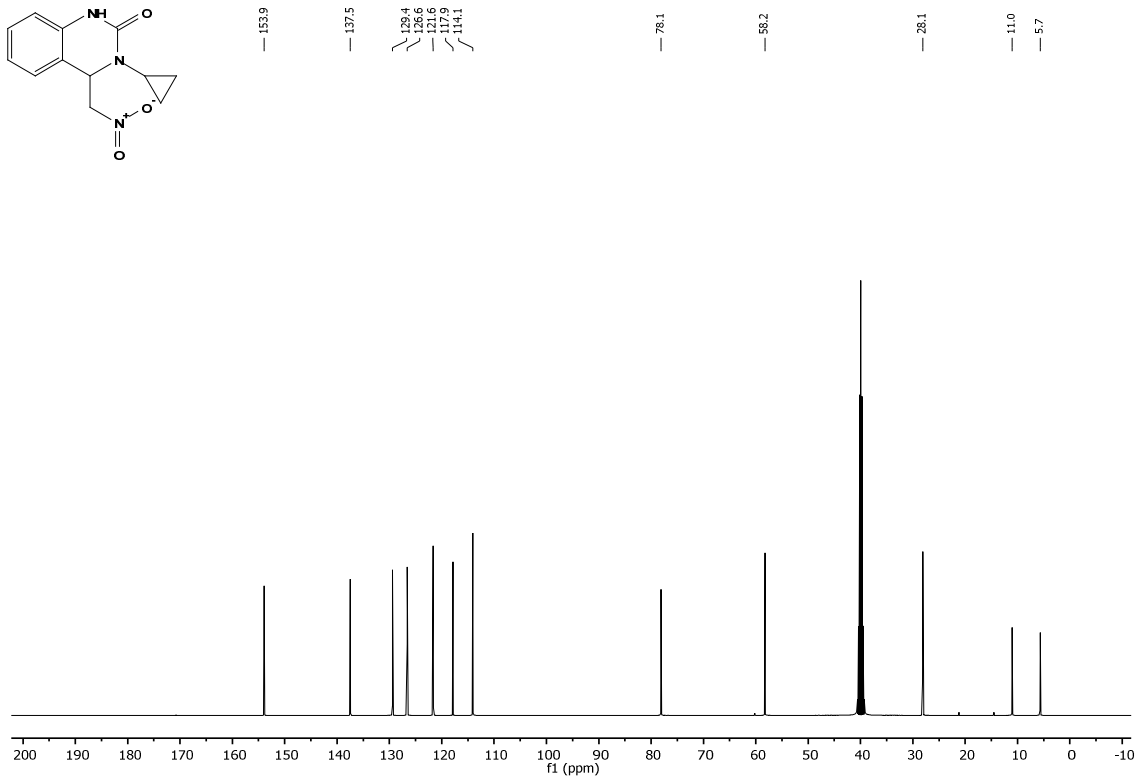
^{1}H NMR spectrum of **2d** (400 MHz, 25 °C, DMSO-d_6)



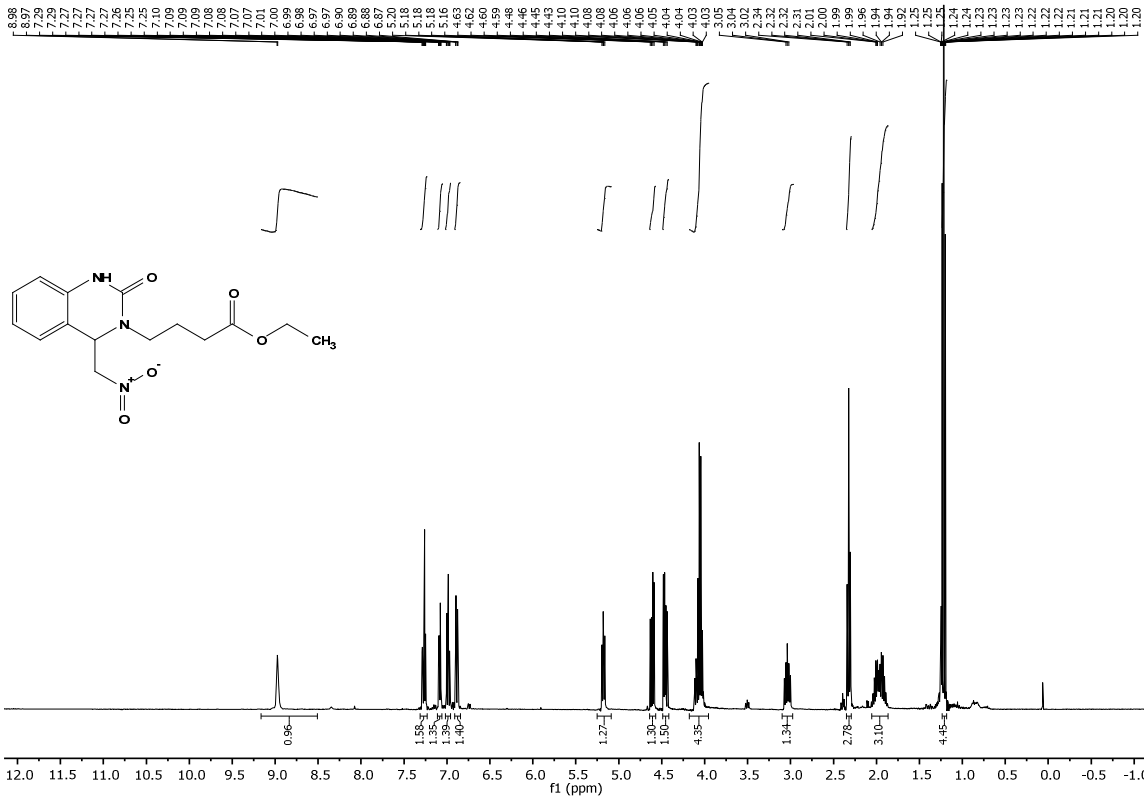
^{13}C NMR spectrum of **2d** (100 MHz, 25 °C, DMSO-d_6)



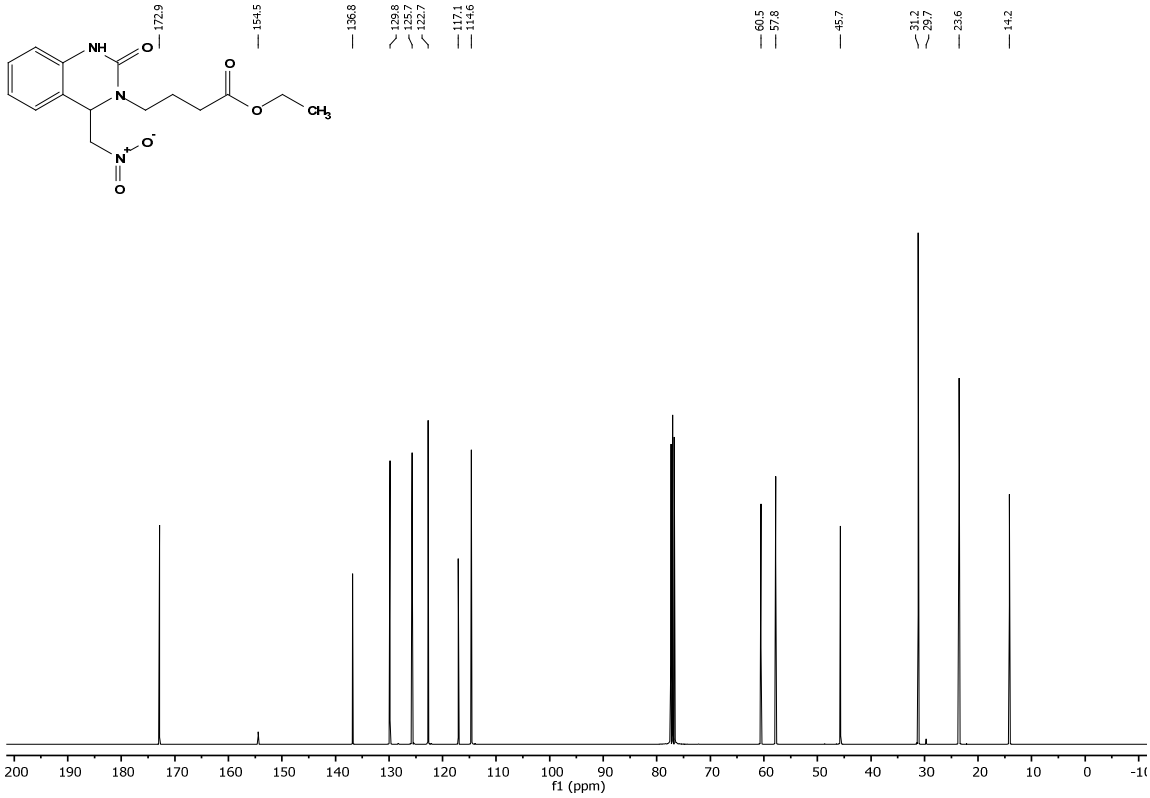
^{1}H NMR spectrum of **2e** (400 MHz, 25 °C, DMSO-d_6)



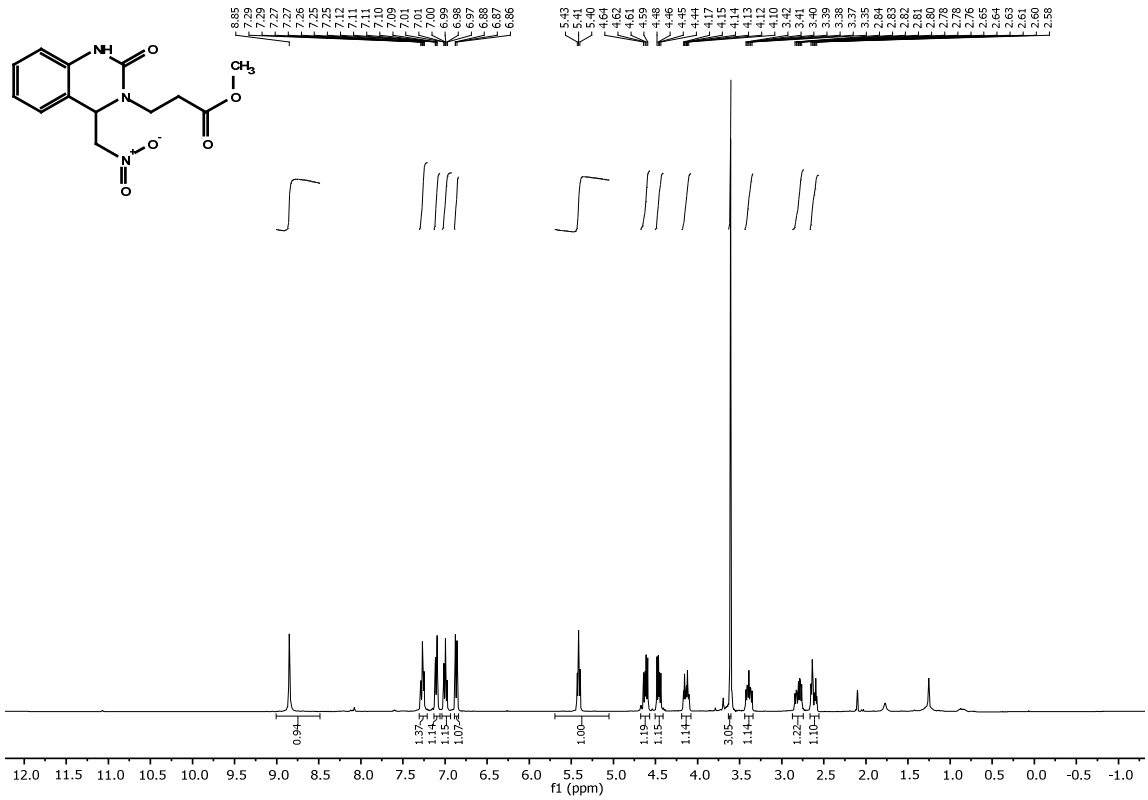
^{13}C NMR spectrum of **2e** (100 MHz, 25 °C, DMSO-d_6)



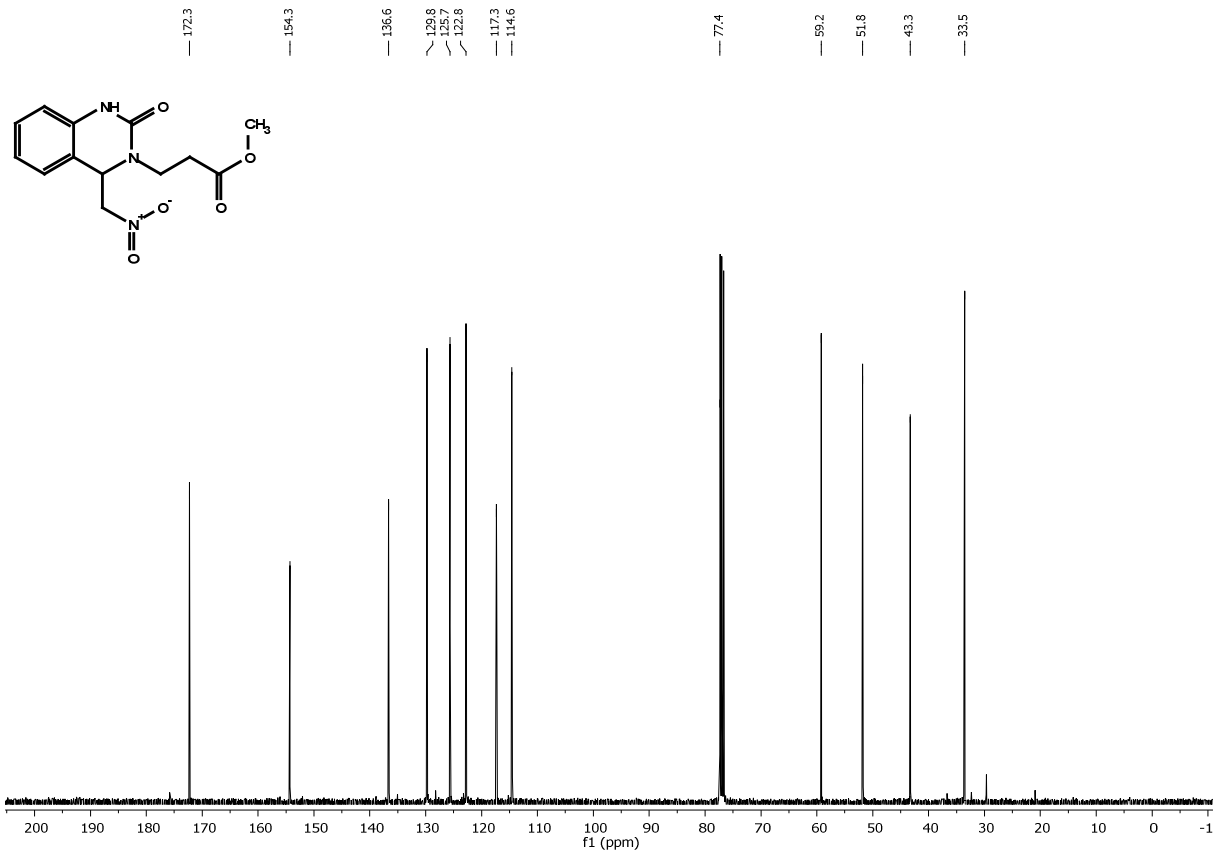
^{1}H NMR spectrum of **2f** (400 MHz, 25 °C, CD_3Cl)



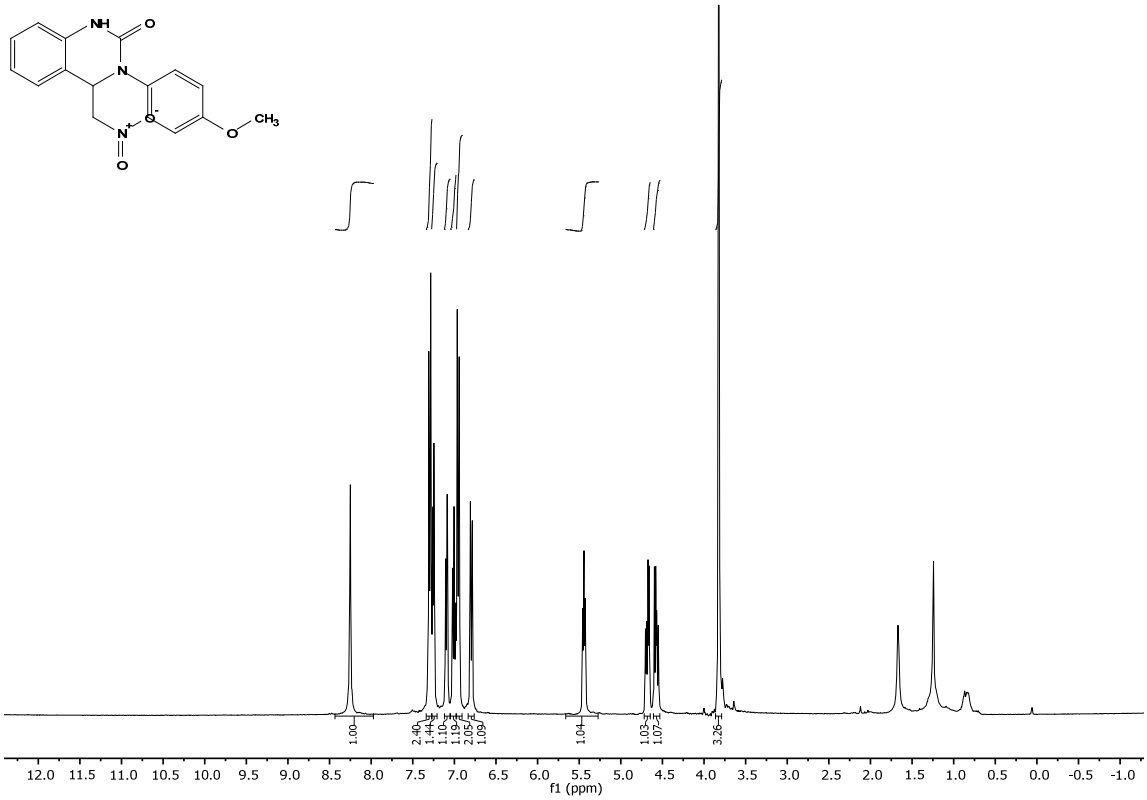
^{13}C NMR spectrum of **2f** (100 MHz, 25 °C, CD_3Cl)



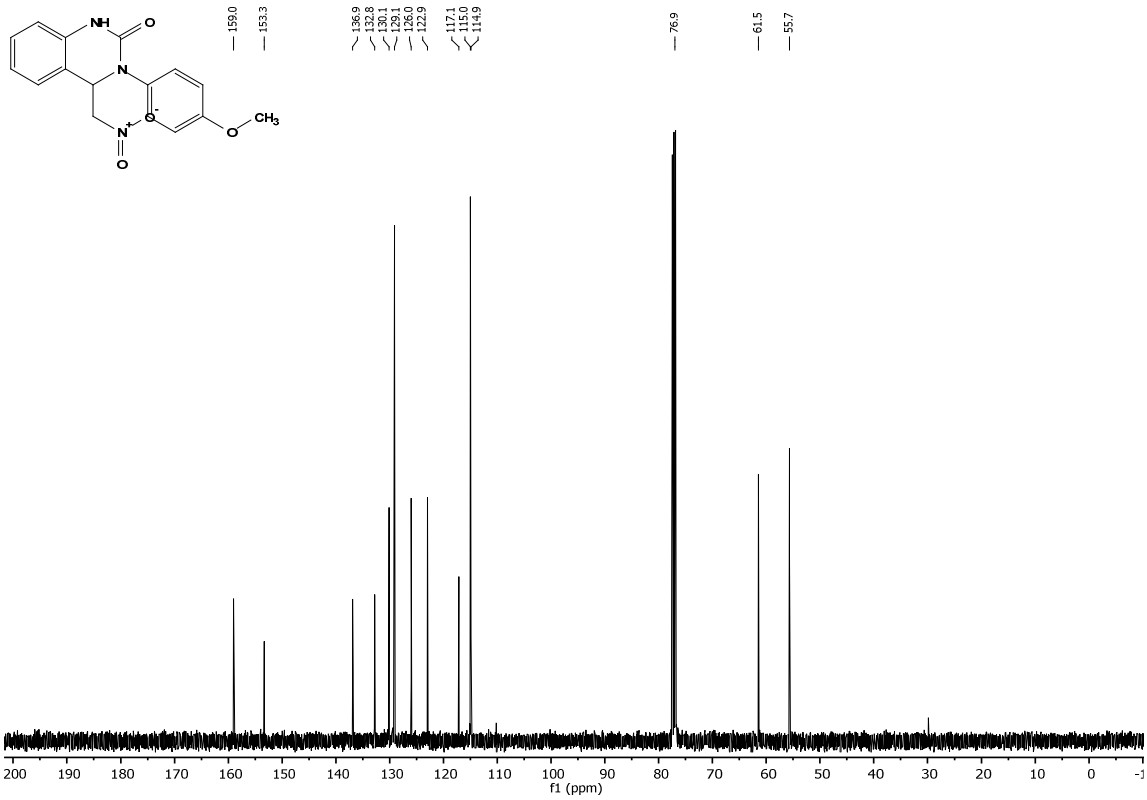
^{1}H NMR spectrum of **2g** (400 MHz, 25 °C, CD_3Cl)



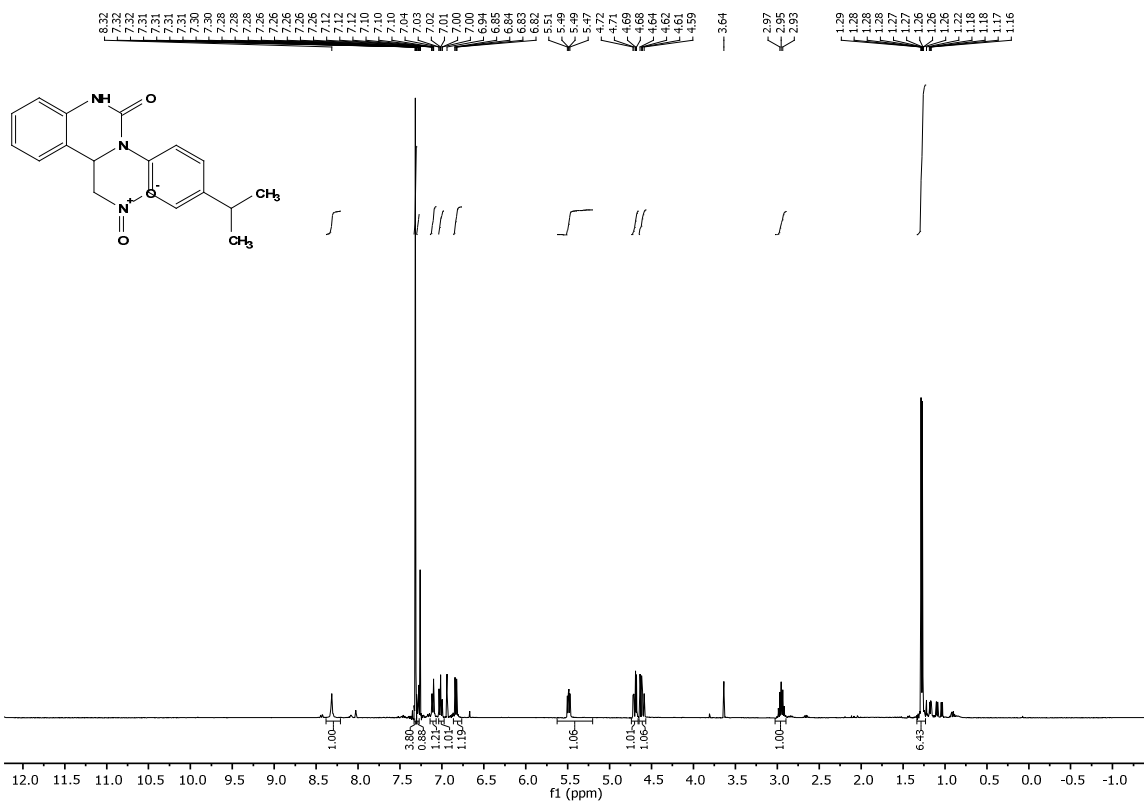
^{13}C NMR spectrum of **2g** (100 MHz, 25 °C, CD_3Cl)



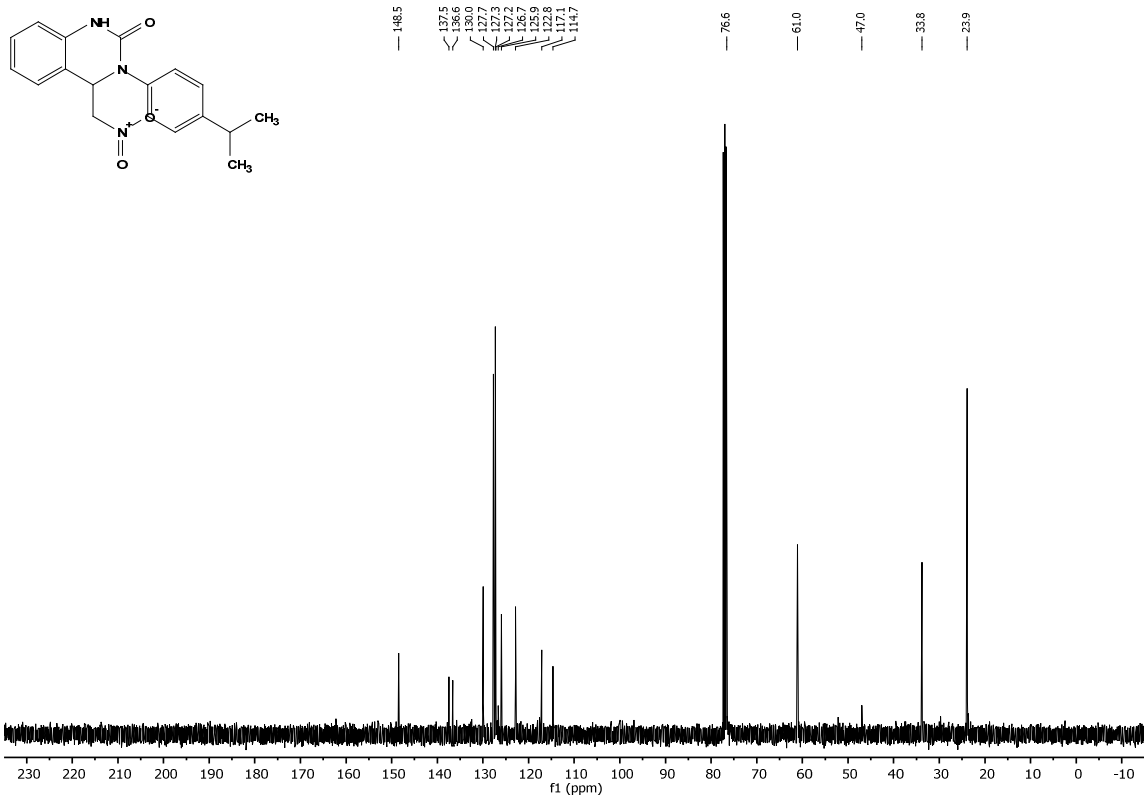
^{1}H NMR spectrum of **2h** (400 MHz, 25 °C, CD_3Cl)



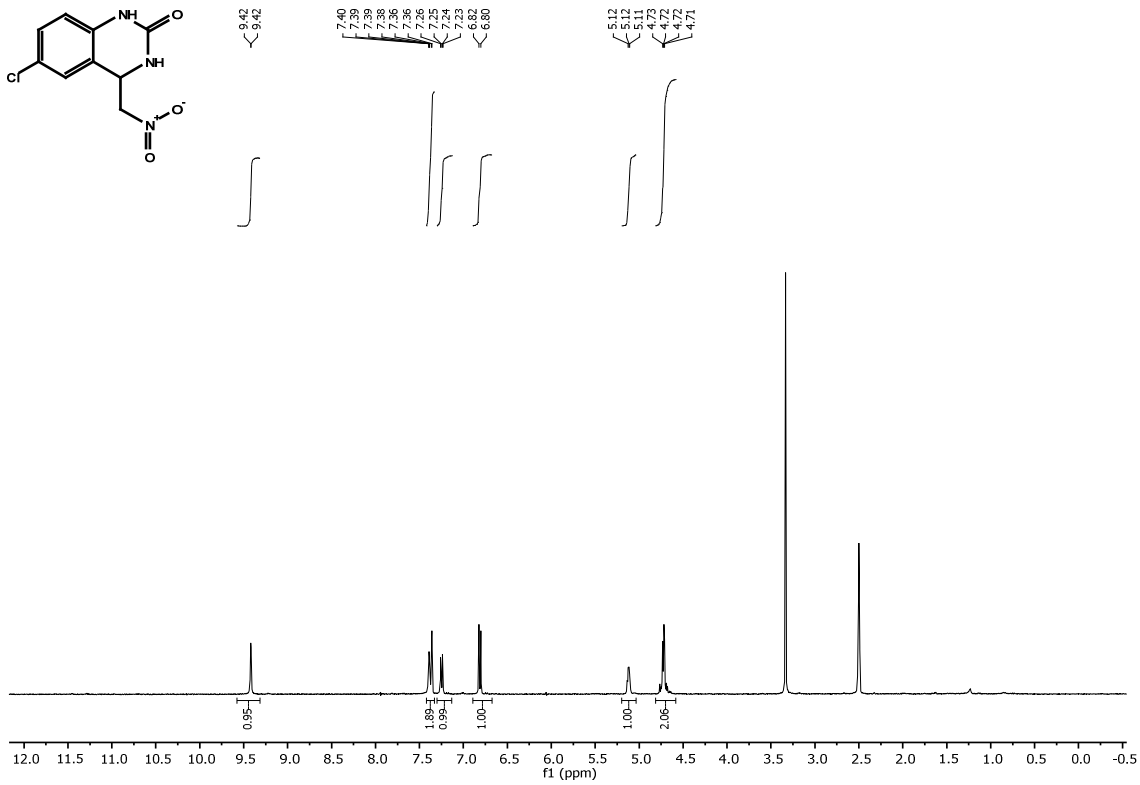
^{13}C NMR spectrum of **2h** (100 MHz, 25 °C, CD_3Cl)



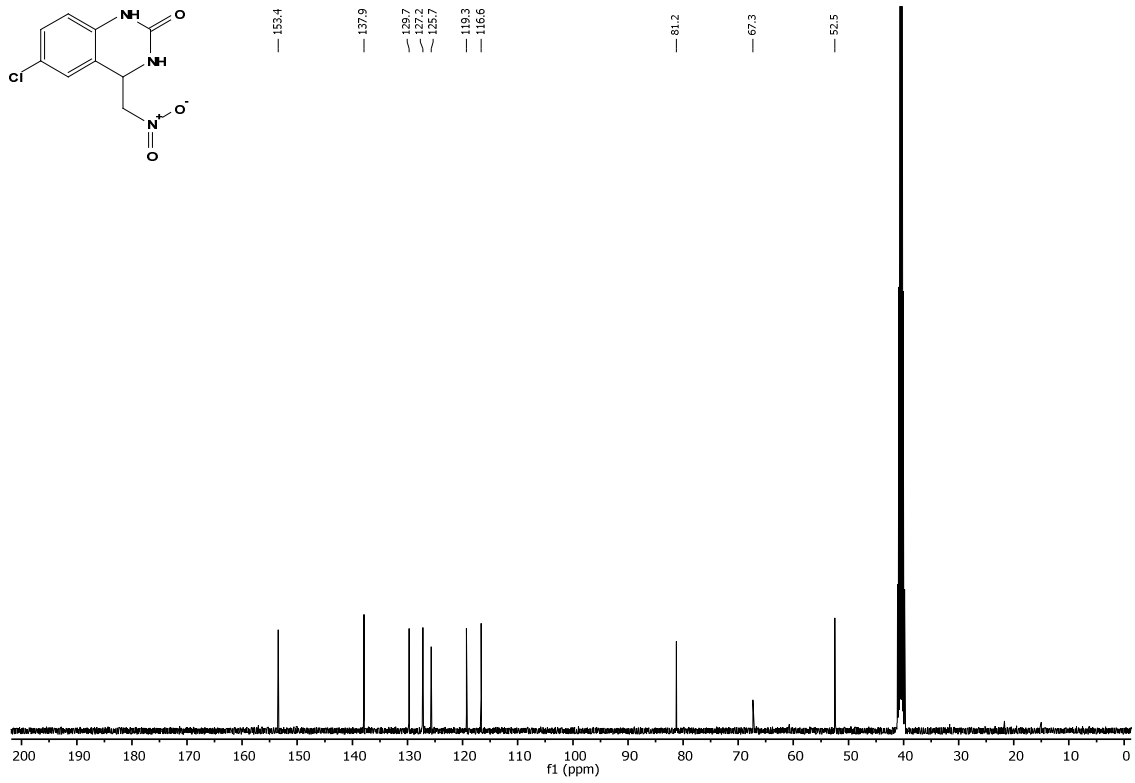
^{1}H NMR spectrum of **2i** (400 MHz, 25 °C, CD_3Cl)



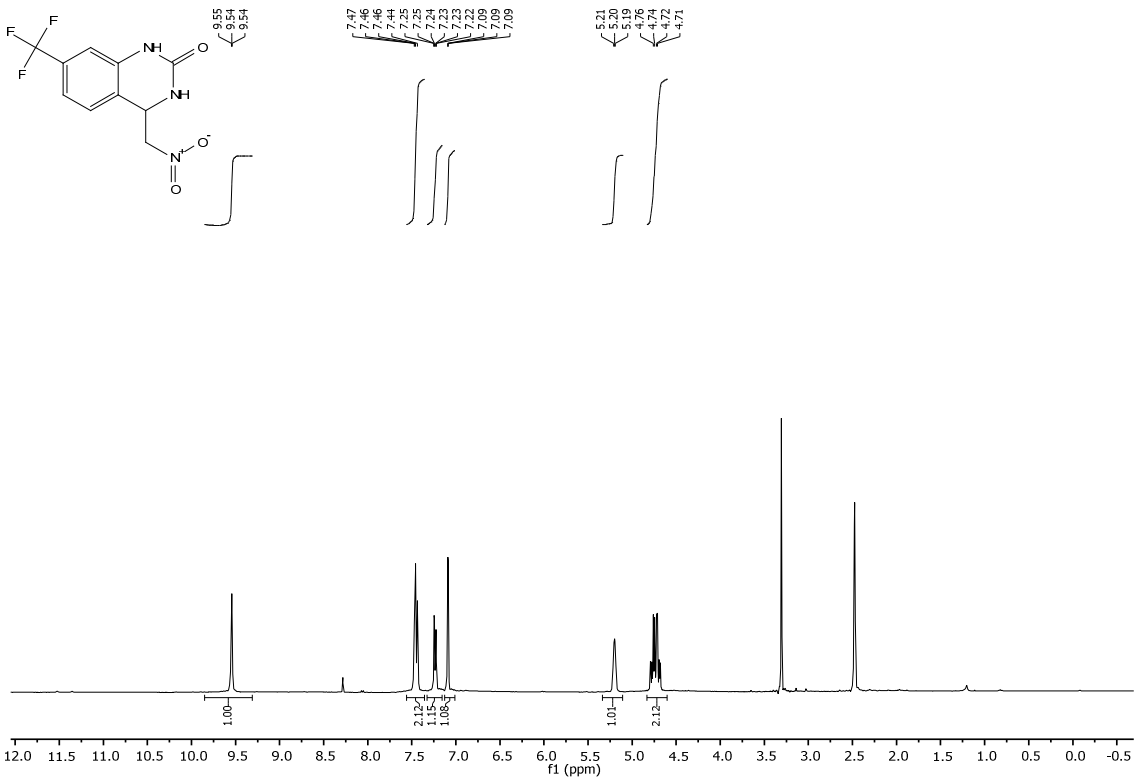
^{13}C NMR spectrum of **2i** (100 MHz, 25 °C, CD_3Cl)



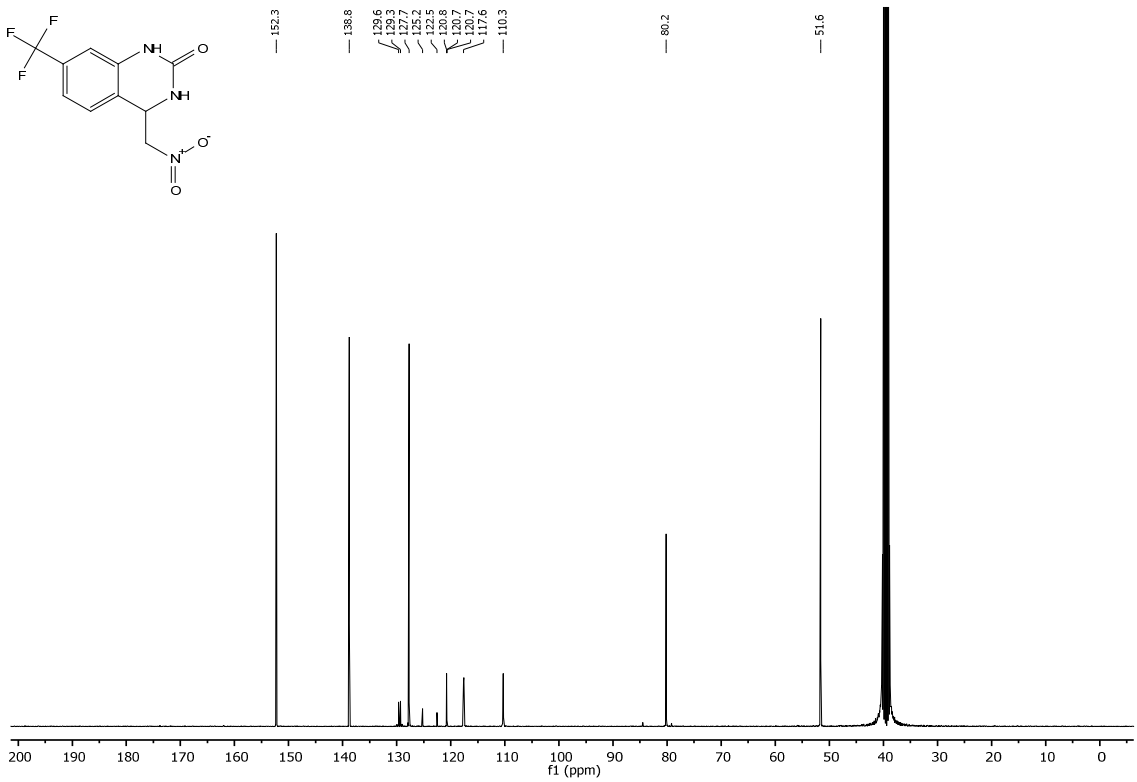
^{1}H NMR spectrum of **3a** (400 MHz, 25 °C, DMSO-d_6)



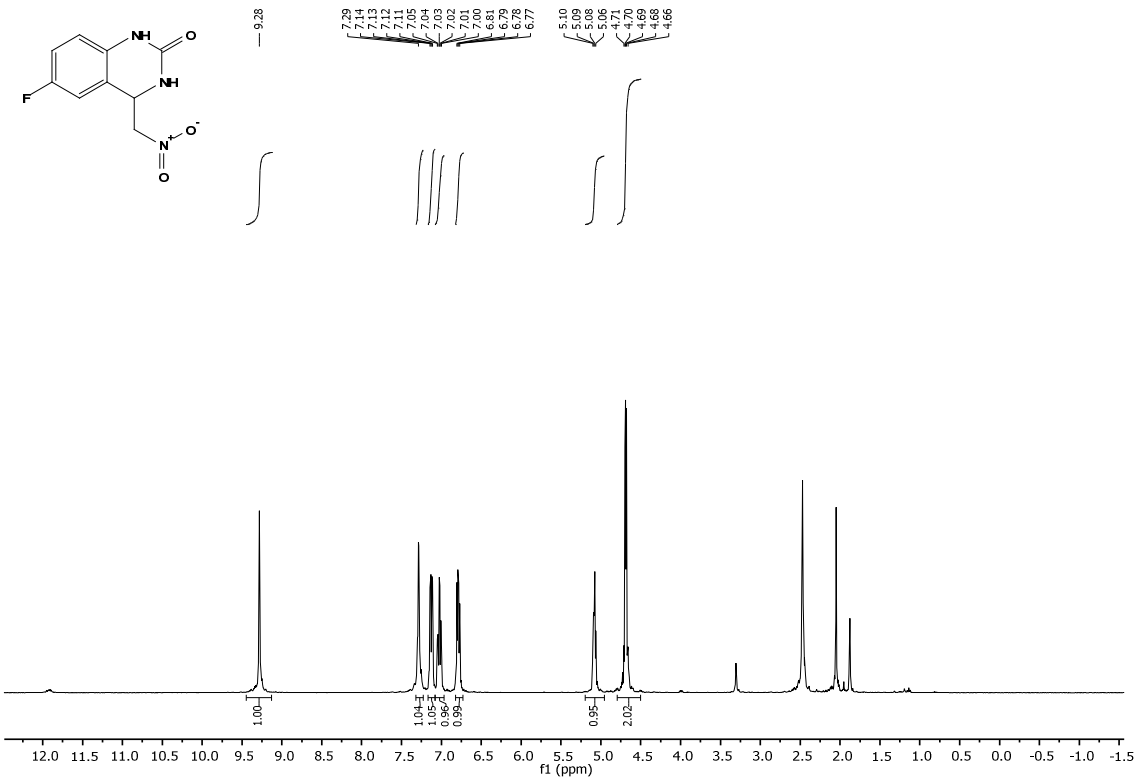
^{13}C NMR spectrum of **3a** (100 MHz, 25 °C, DMSO-d_6)



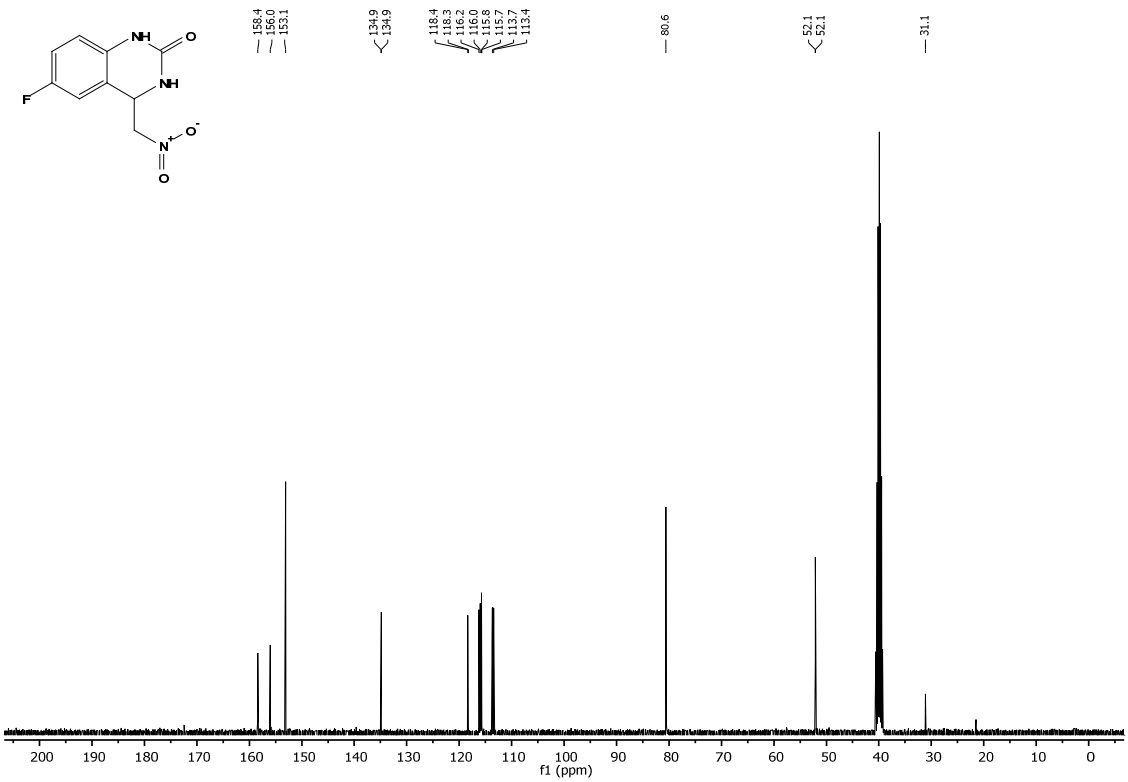
^{1}H NMR spectrum of **3b** (400 MHz, 25 °C, DMSO-d_6)



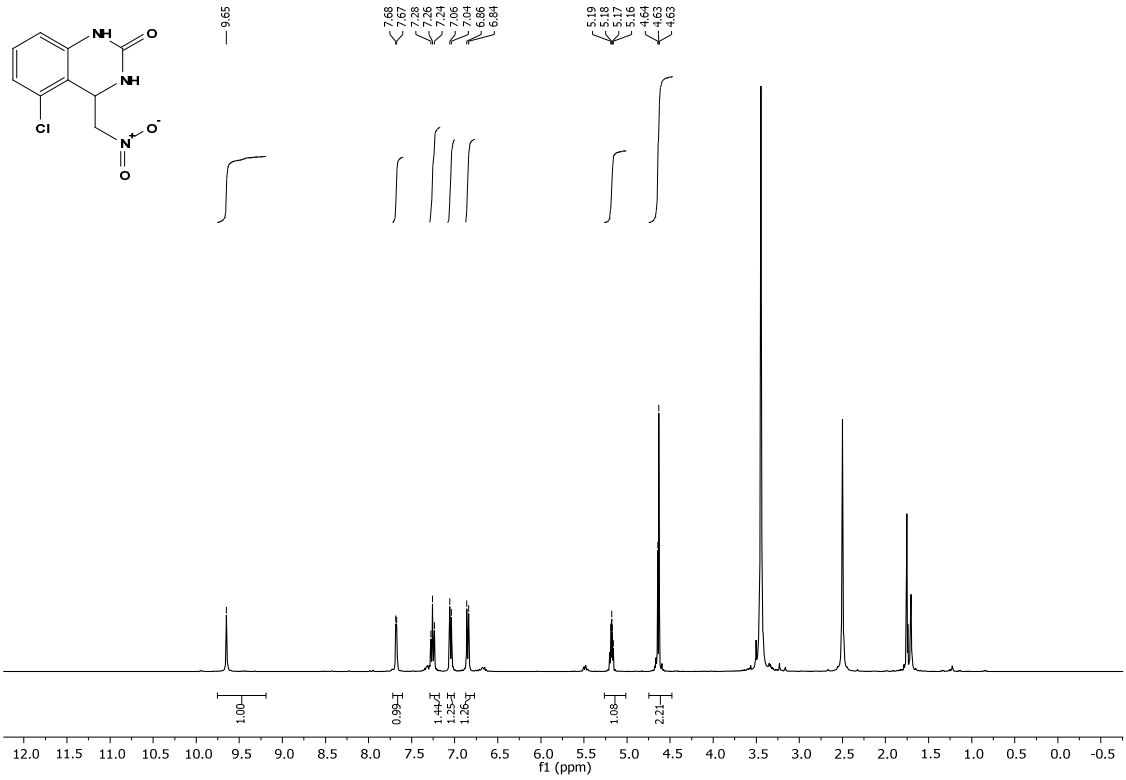
^{13}C NMR spectrum of **3b** (100 MHz, 25 °C, DMSO-d_6)



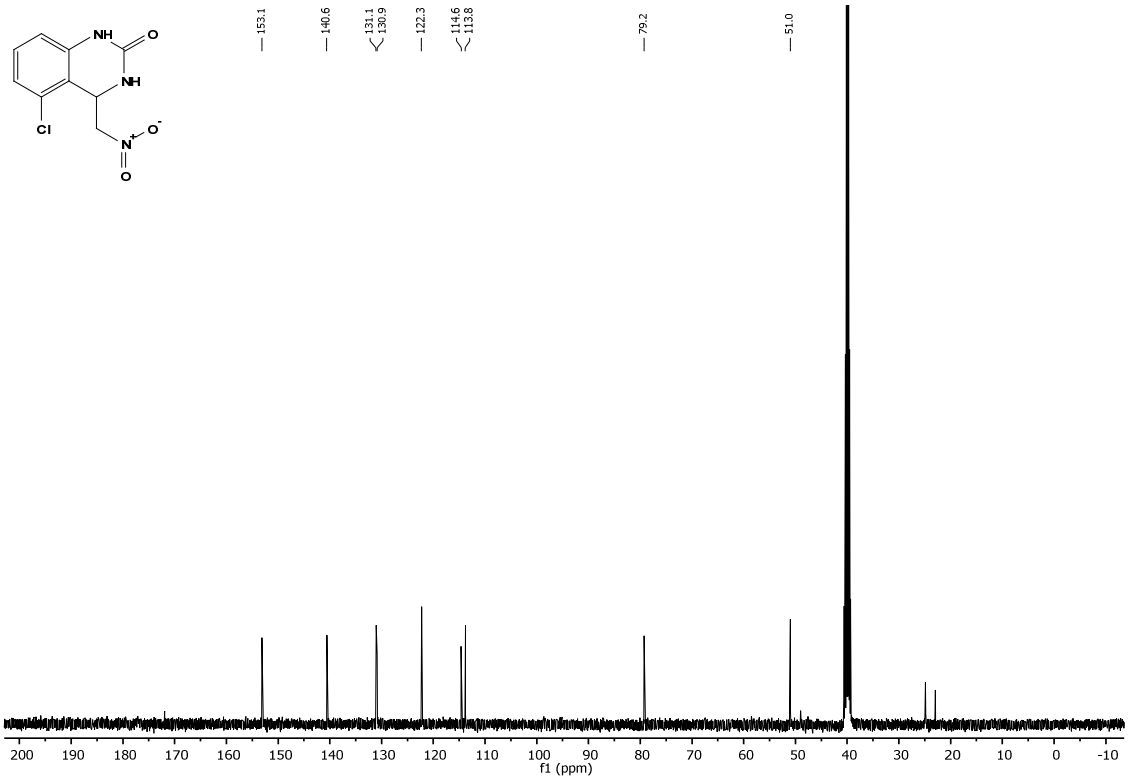
^{1}H NMR spectrum of **3c** (400 MHz, 25 °C, DMSO-d_6)



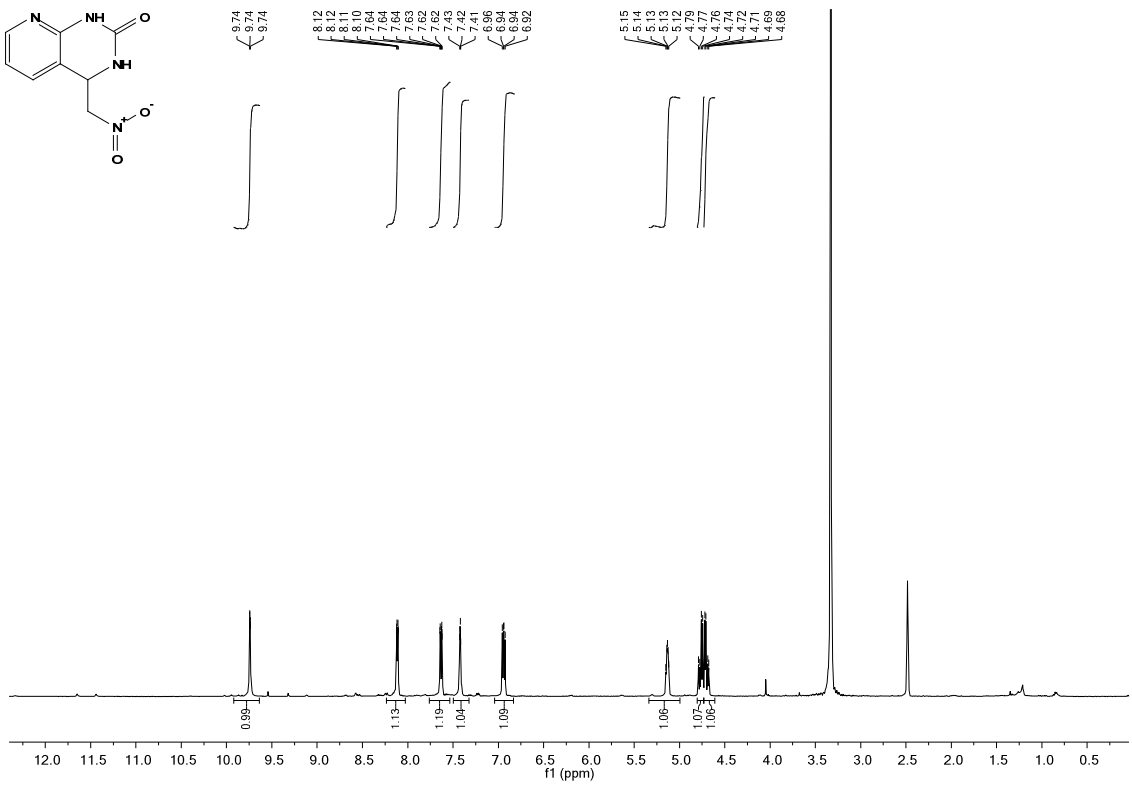
^{13}C NMR spectrum of **3c** (100 MHz, 25 °C, DMSO-d_6)



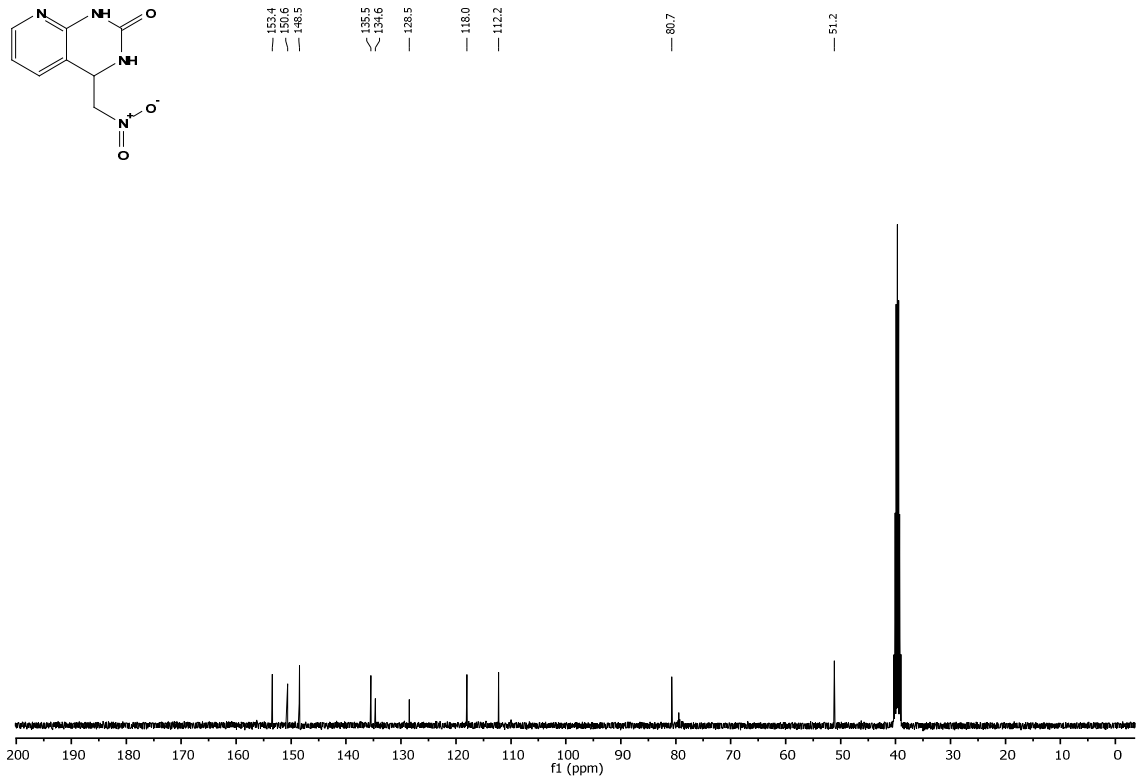
^{1}H NMR spectrum of **3d** (400 MHz, 25 °C, DMSO-d_6)



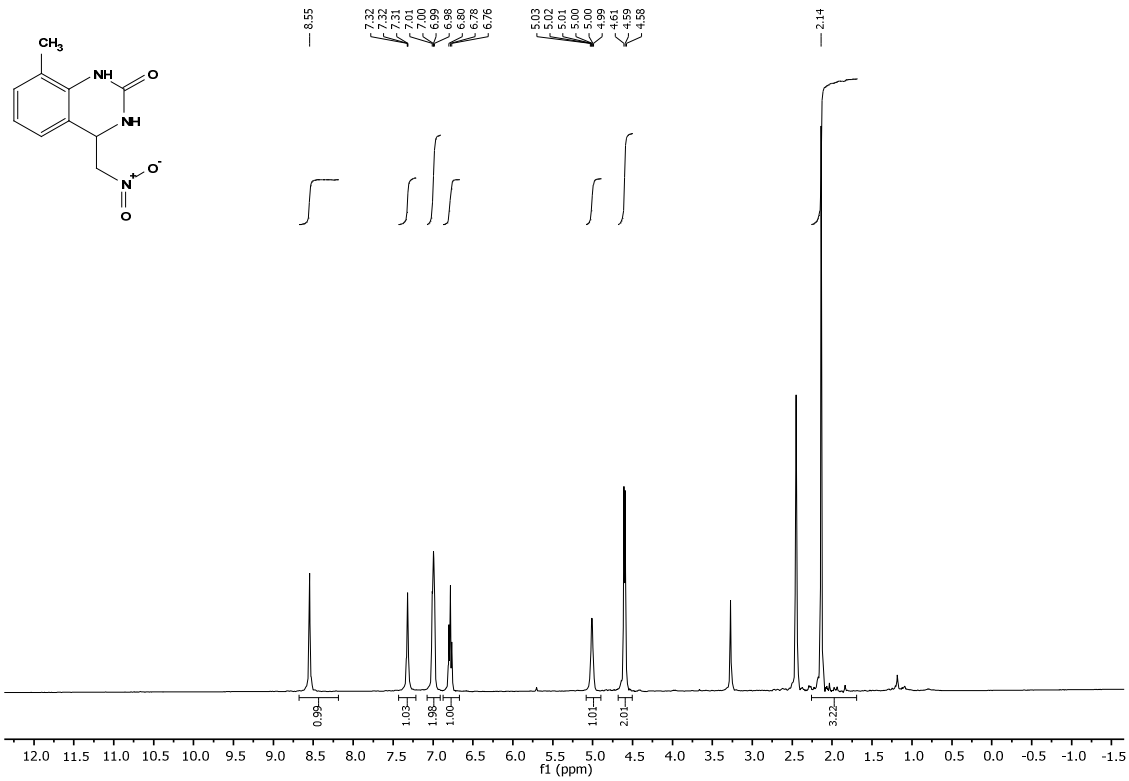
^{13}C NMR spectrum of **3d** (100 MHz, 25 °C, DMSO-d_6)



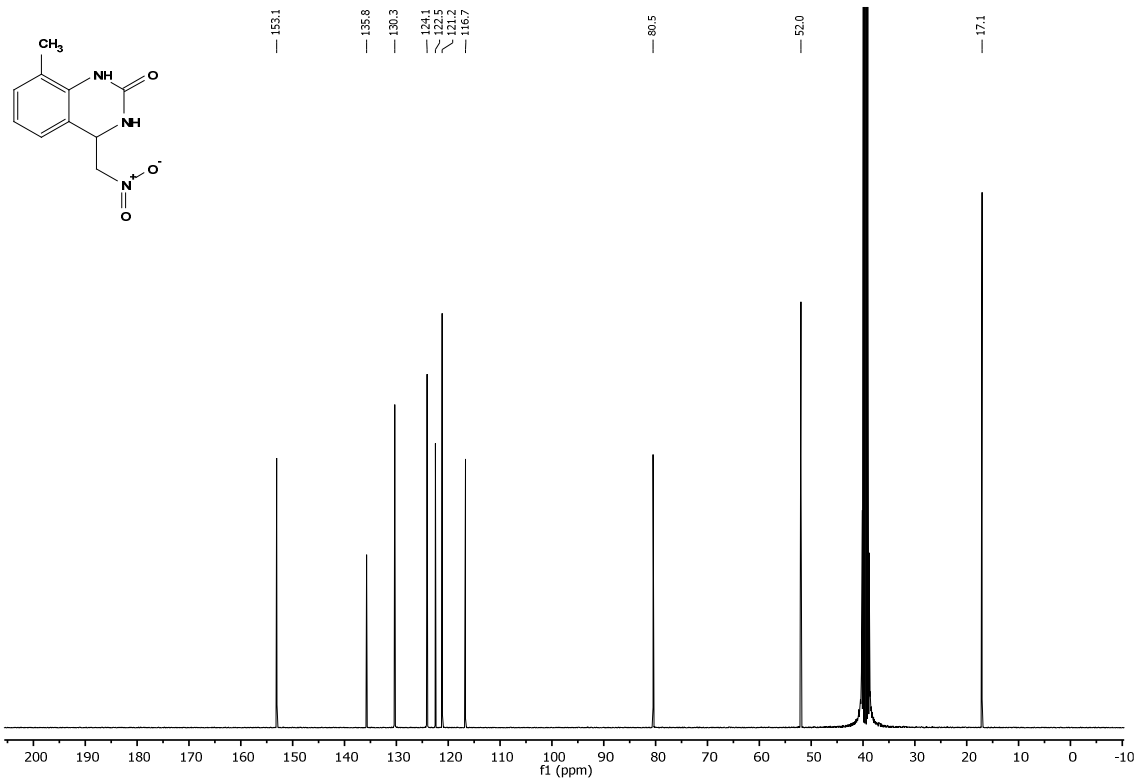
^{1}H NMR spectrum of **3e** (400 MHz, 25 °C, DMSO-d_6)



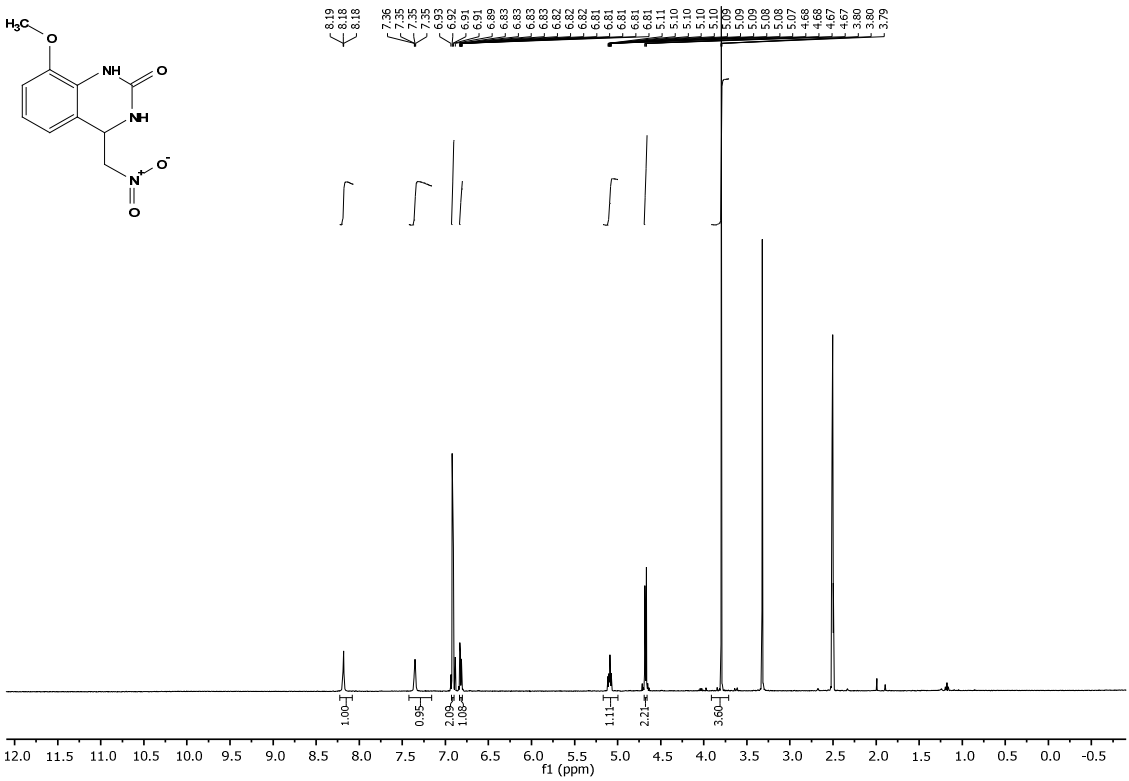
^{13}C NMR spectrum of **3e** (100 MHz, 25 °C, DMSO-d_6)



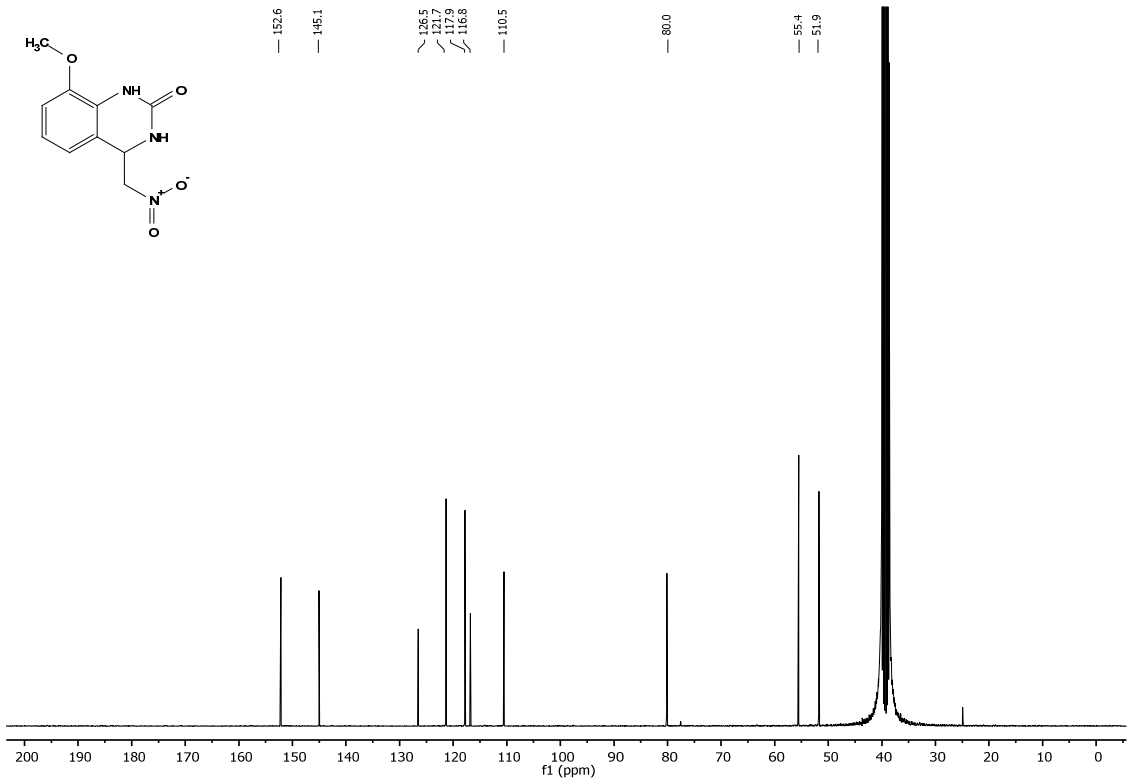
^{1}H NMR spectrum of **3f** (400 MHz, 25 °C, DMSO-d_6)



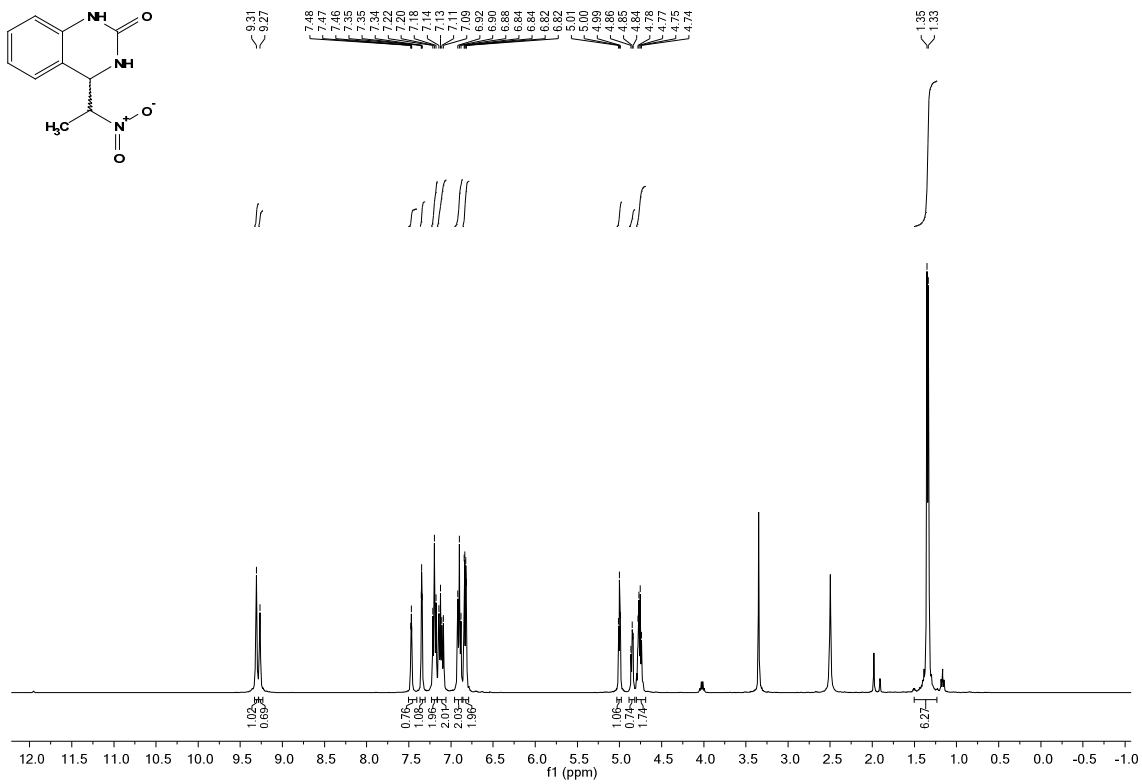
^{13}C NMR spectrum of **3f** (100 MHz, 25 °C, DMSO-d_6)



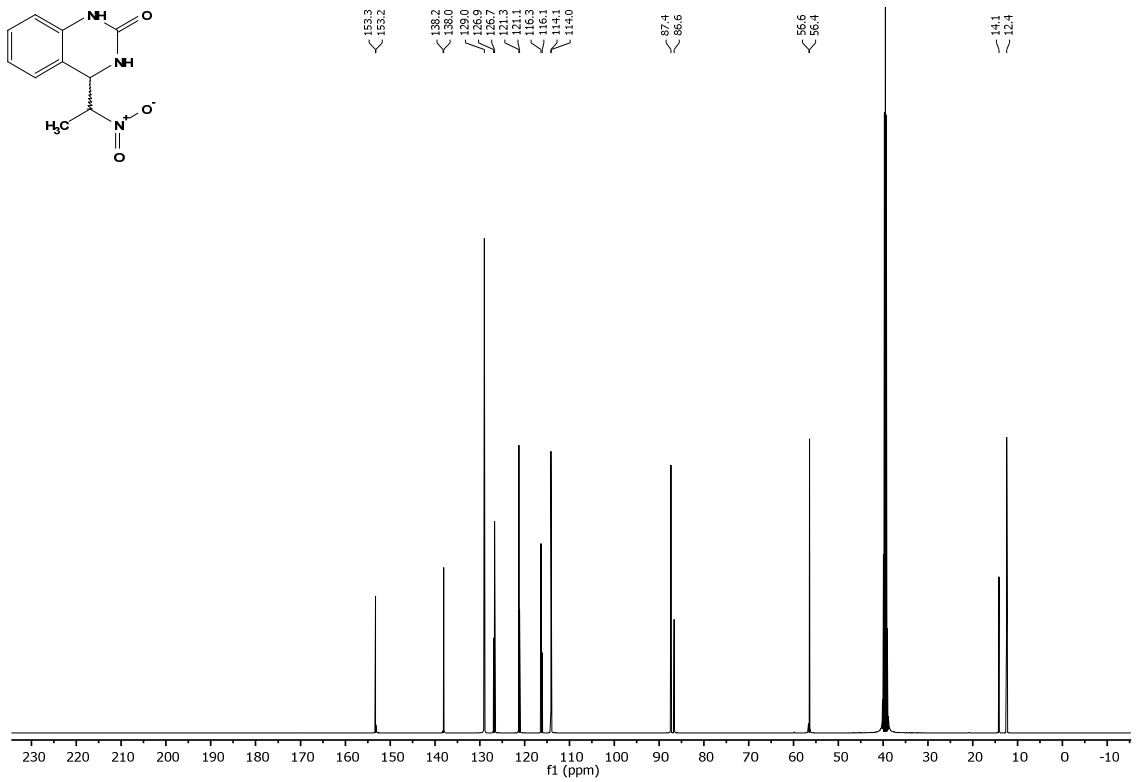
^{1}H NMR spectrum of **3g** (400 MHz, 25 °C, DMSO-d_6)



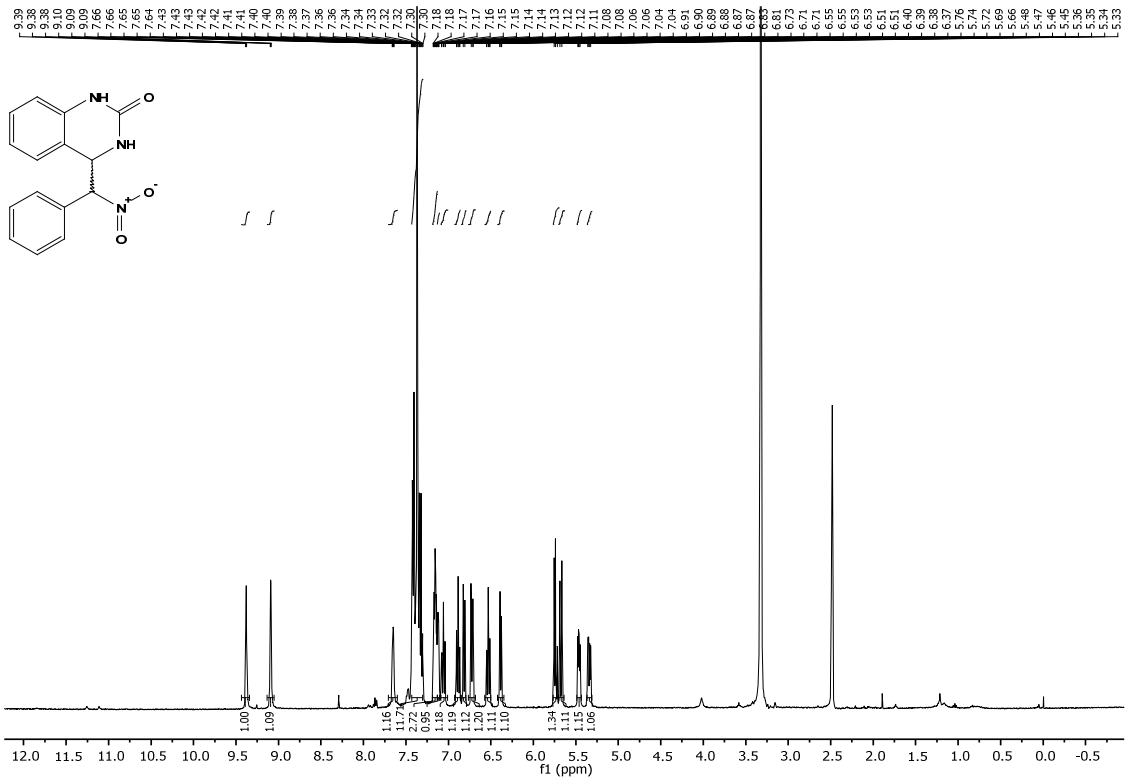
^{13}C NMR spectrum of **3g** (100 MHz, 25 °C, DMSO-d_6)



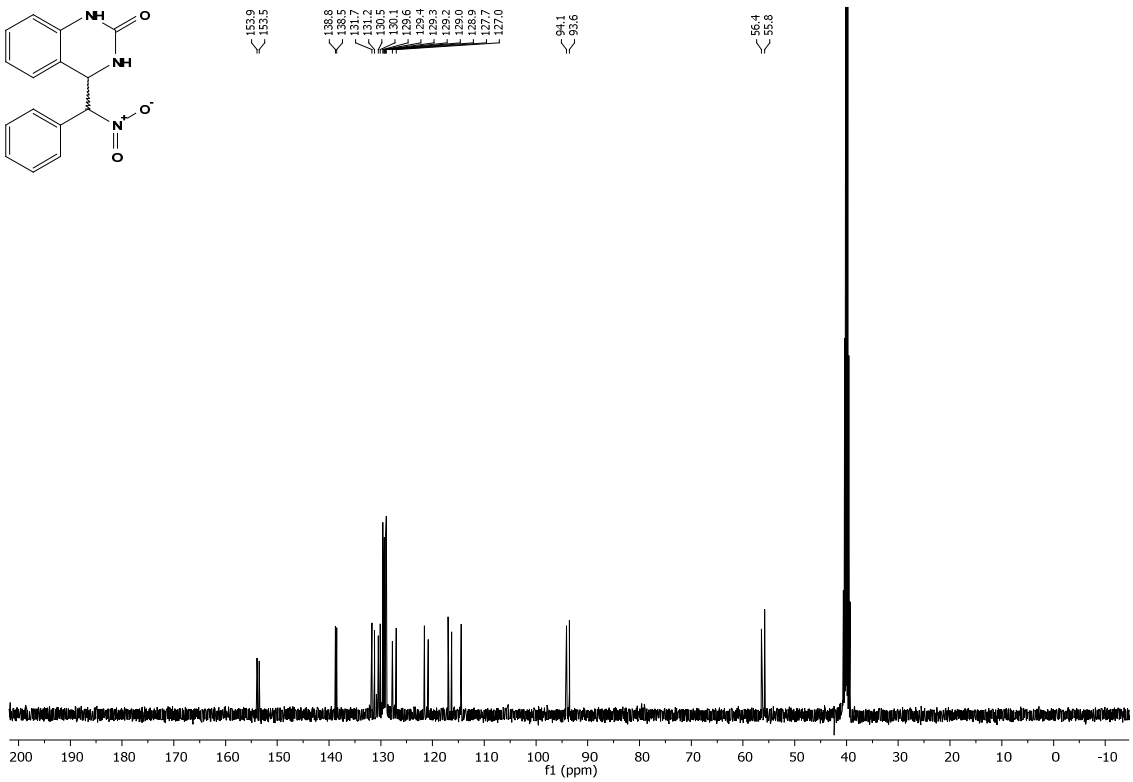
^{1}H NMR spectrum of **4a** (400 MHz, 25 °C, DMSO-d_6), 0.7:1 mixture of diastereomers



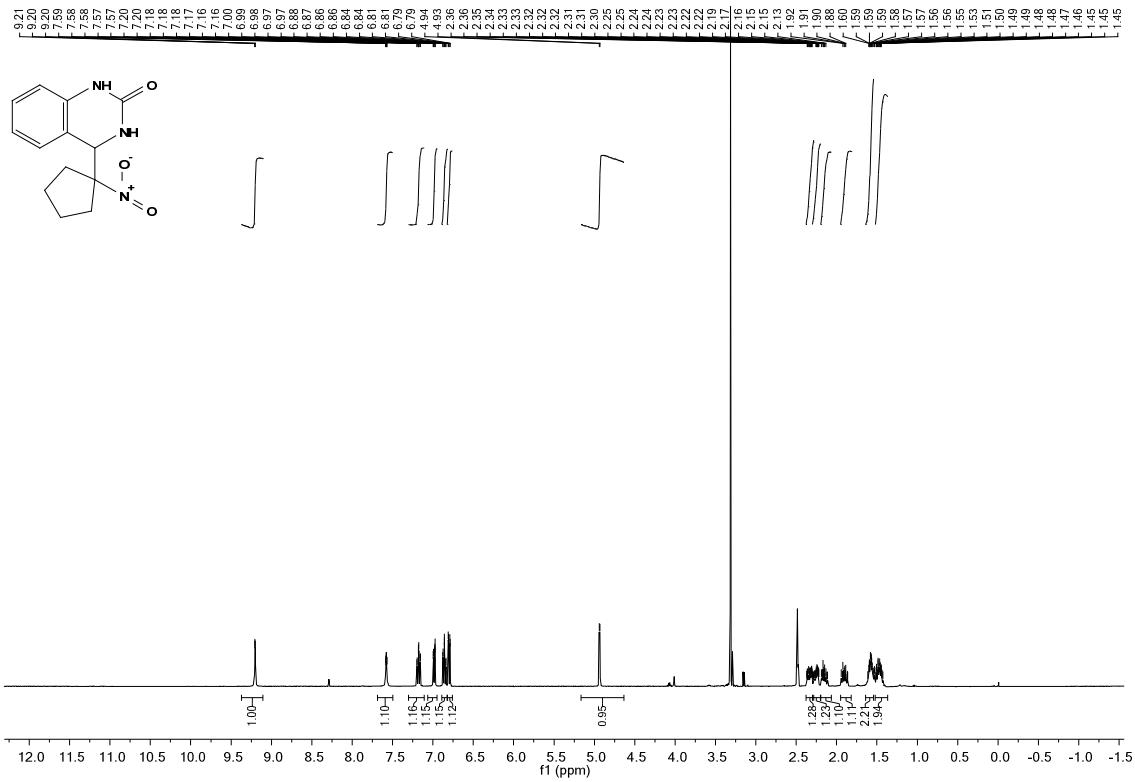
^{13}C NMR spectrum of **4a** (100 MHz, 25 °C, DMSO-d_6), 0.7:1 mixture of diastereomers



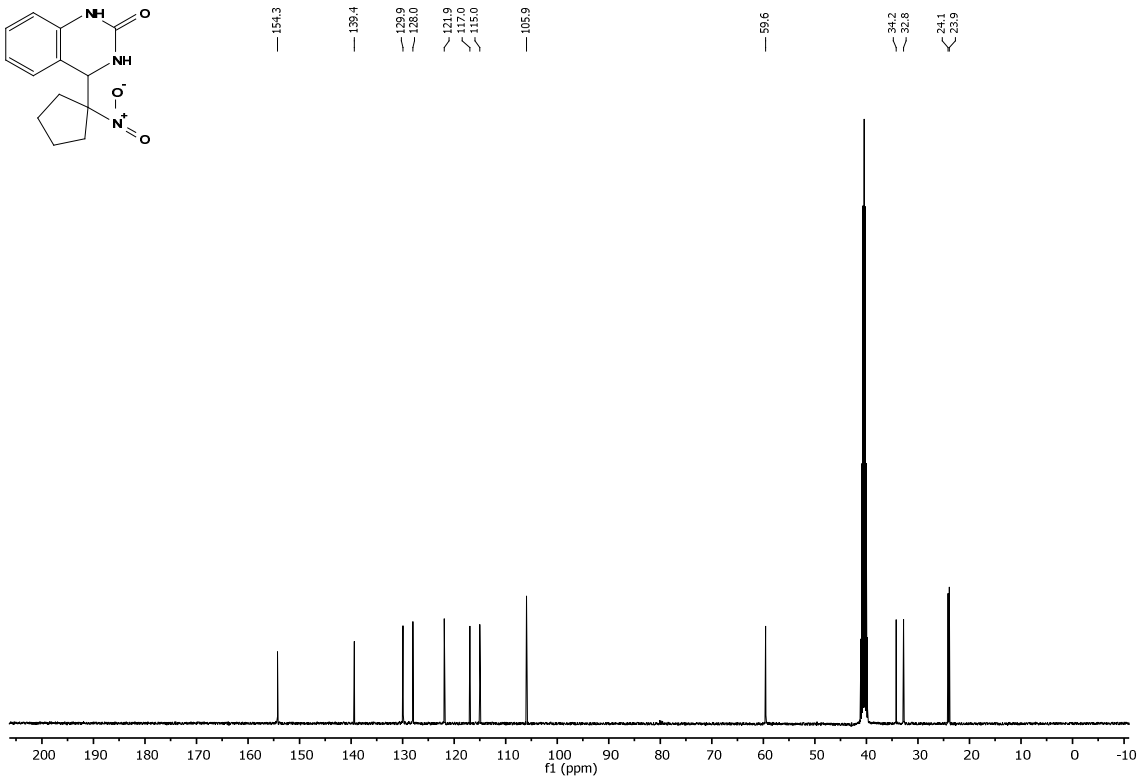
^{1}H NMR spectrum of **4b** (400 MHz, 25 °C, DMSO-d_6), 1:1 mixture of diastereomers



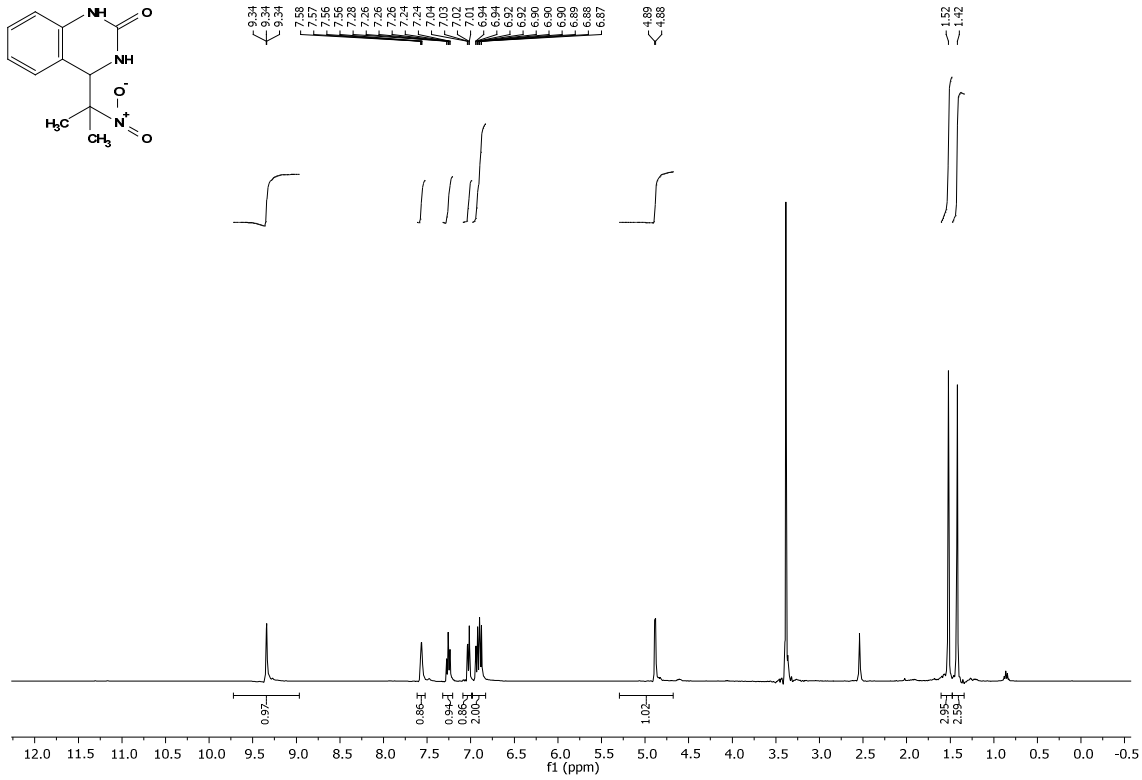
^{13}C NMR spectrum of **4b** (100 MHz, 25 °C, DMSO-d_6), 1:1 mixture of diastereomers



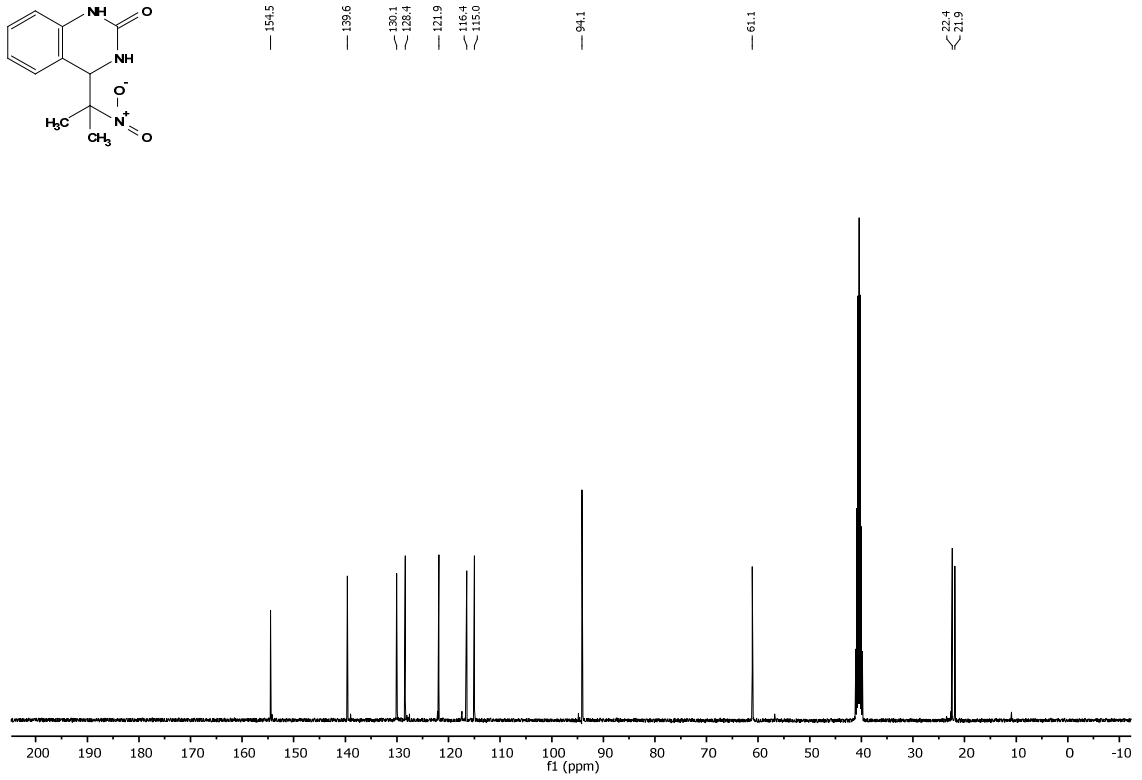
^{1}H NMR spectrum of **4c** (400 MHz, 25 °C, DMSO-d_6)



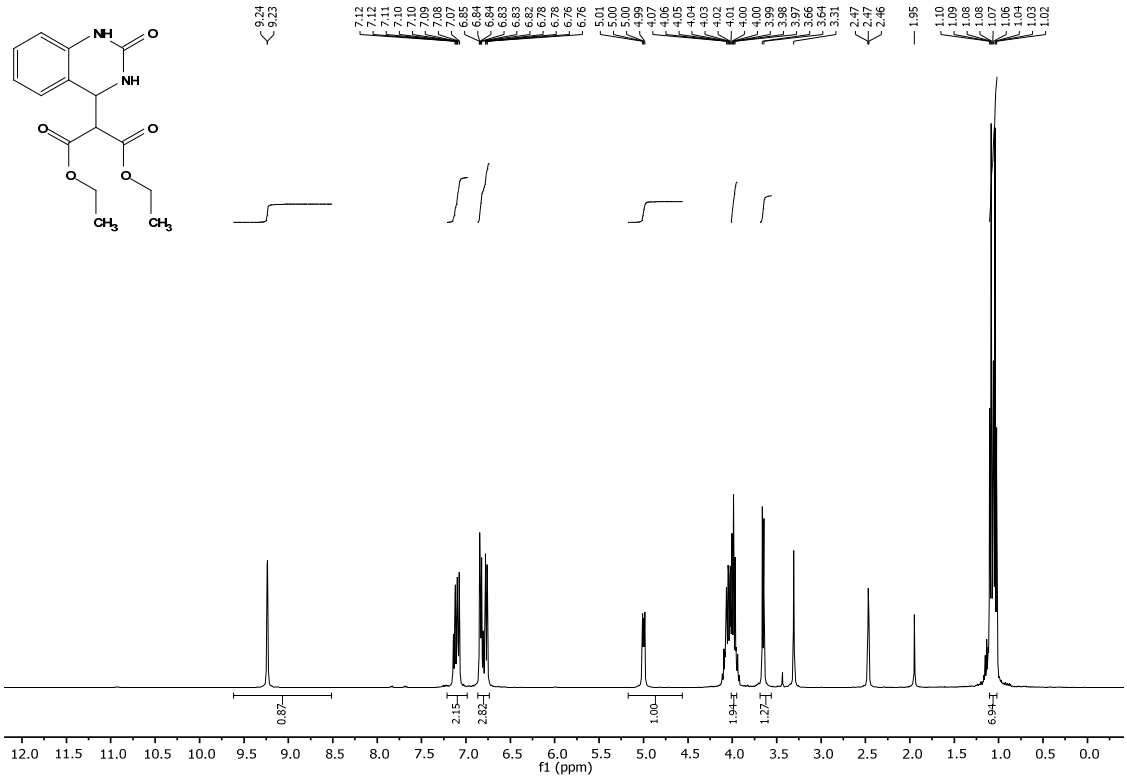
^{13}C NMR spectrum of **4c** (100 MHz, 25 °C, DMSO-d_6)



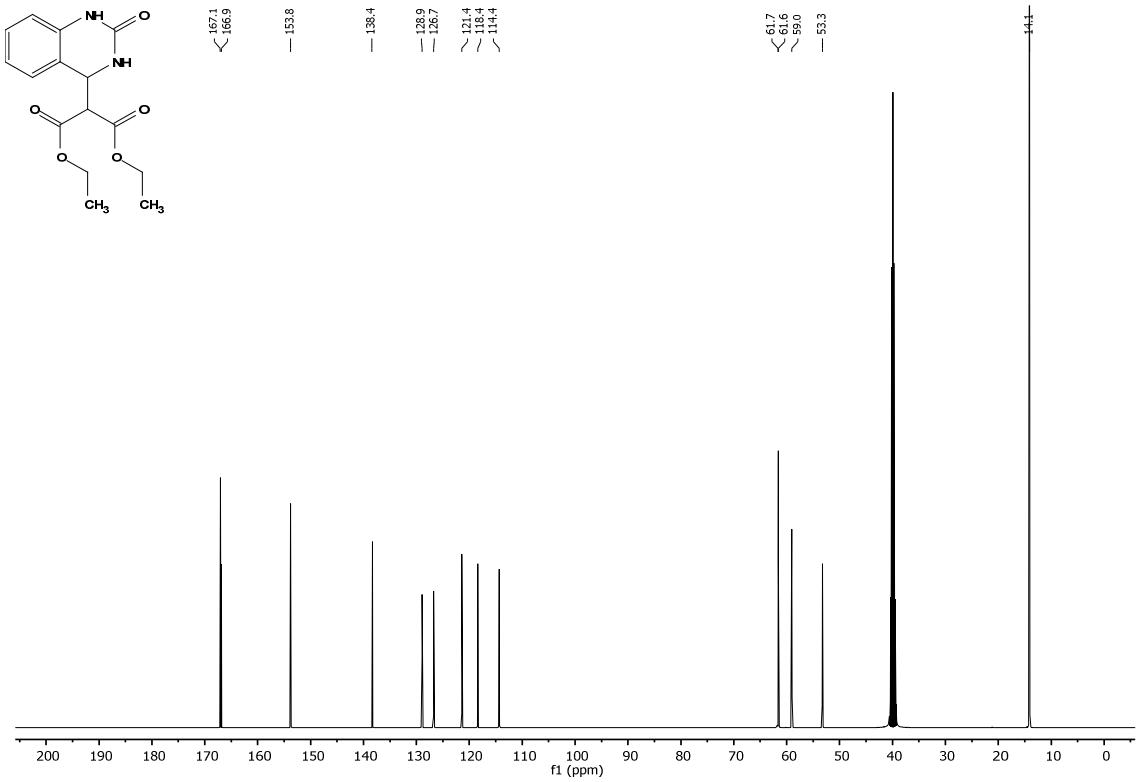
^{1}H NMR spectrum of **4d** (400 MHz, 25 °C, DMSO-d_6)



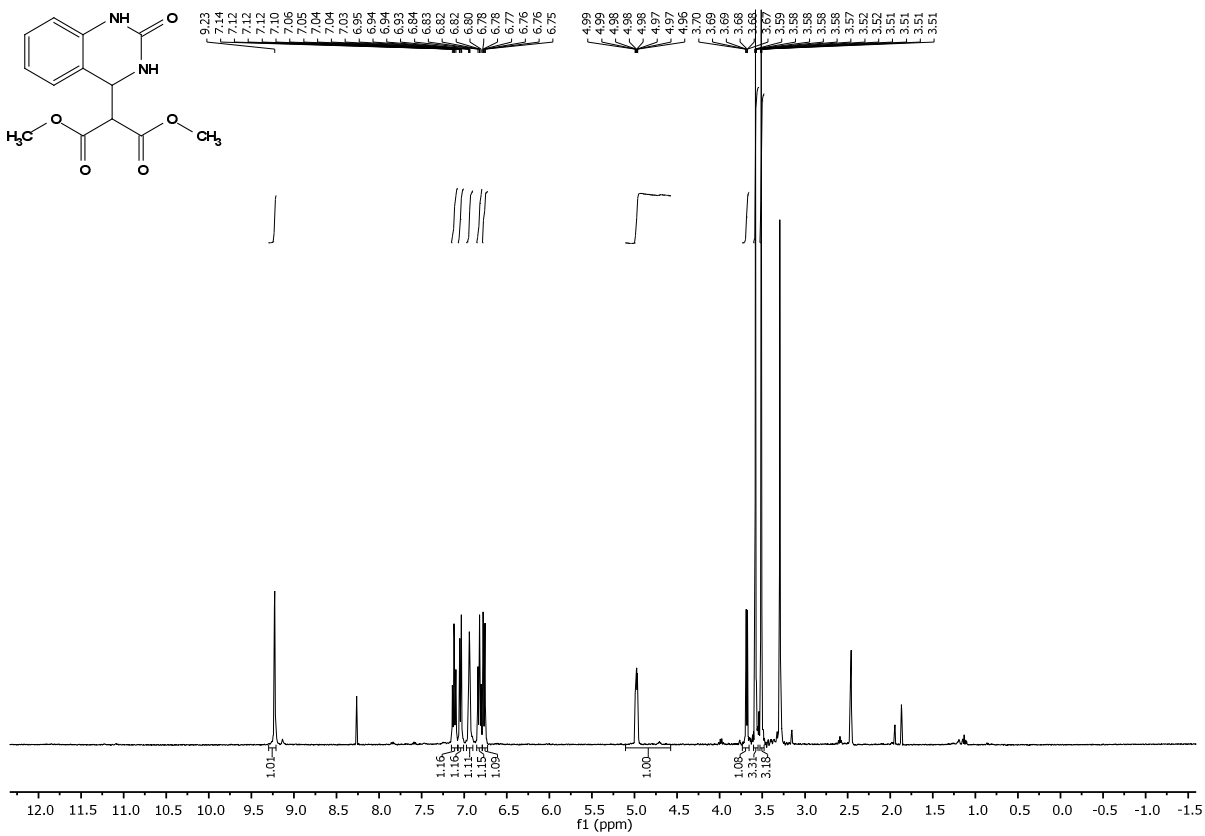
^{13}C NMR spectrum of **4d** (100 MHz, 25 °C, DMSO-d_6)



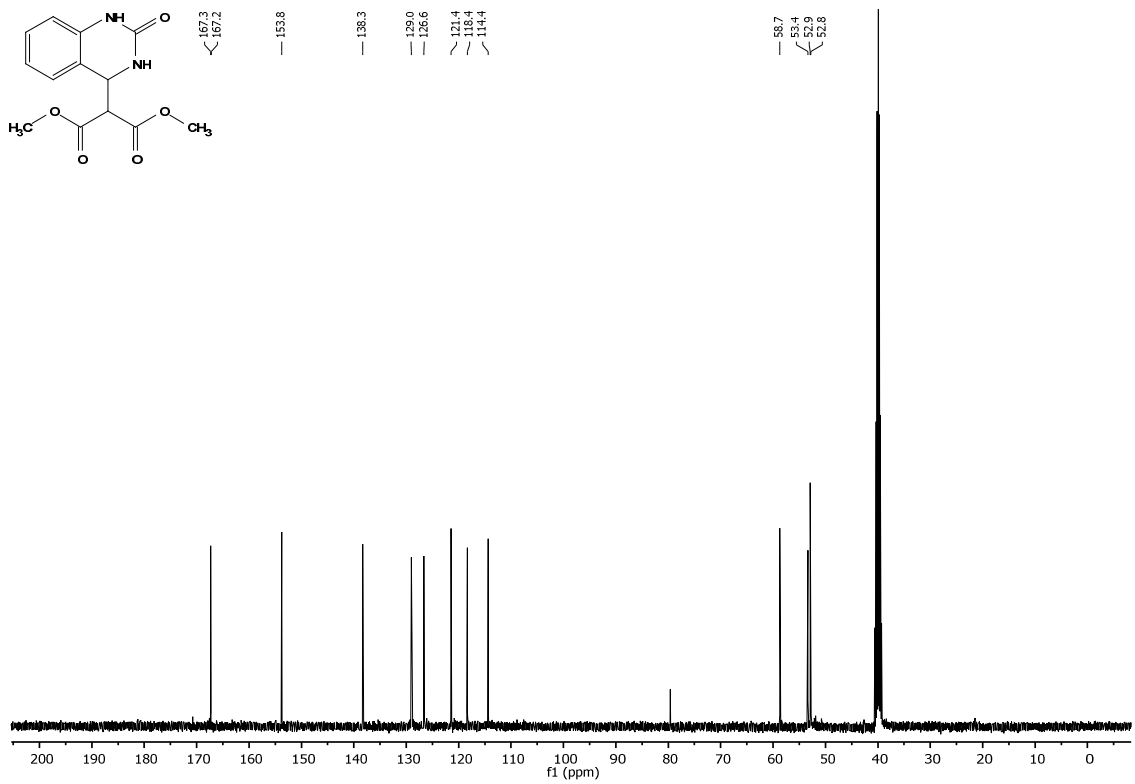
^{1}H NMR spectrum of **4e** (400 MHz, 25 °C, DMSO-d_6)



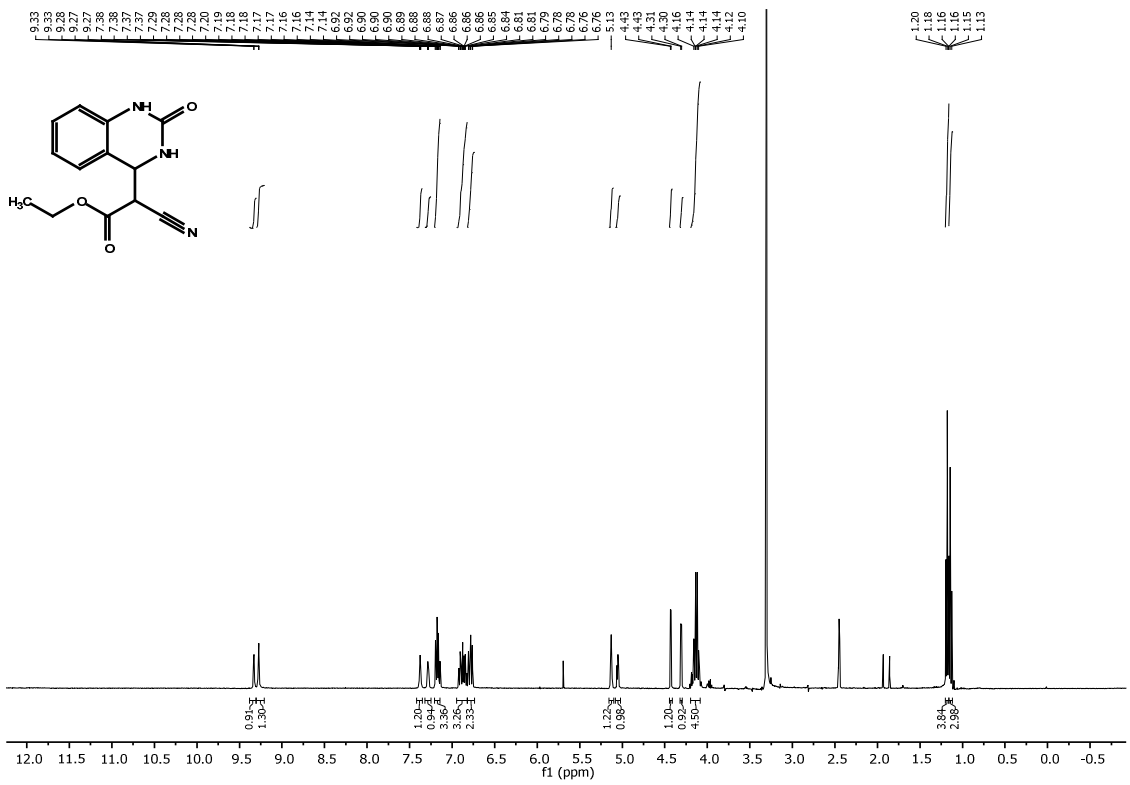
^{13}C NMR spectrum of **4e** (100 MHz, 25 °C, DMSO-d_6)



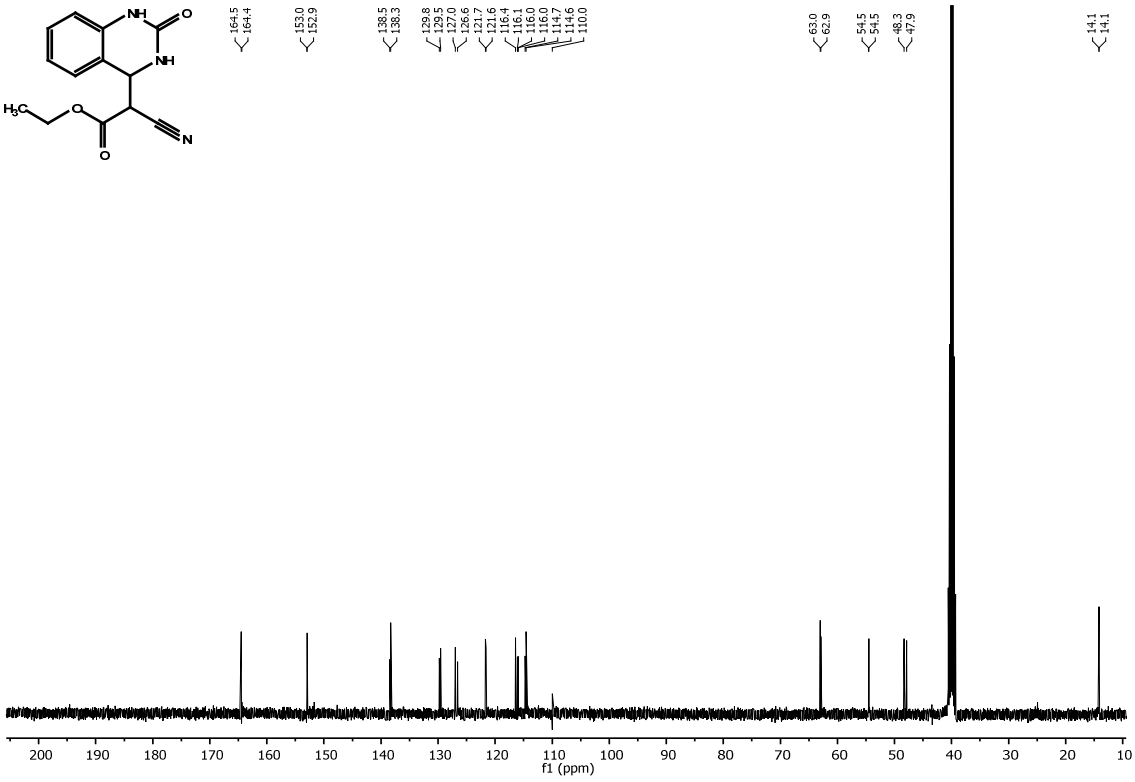
^{1}H NMR spectrum of **4f** (400 MHz, 25 °C, DMSO-d_6)



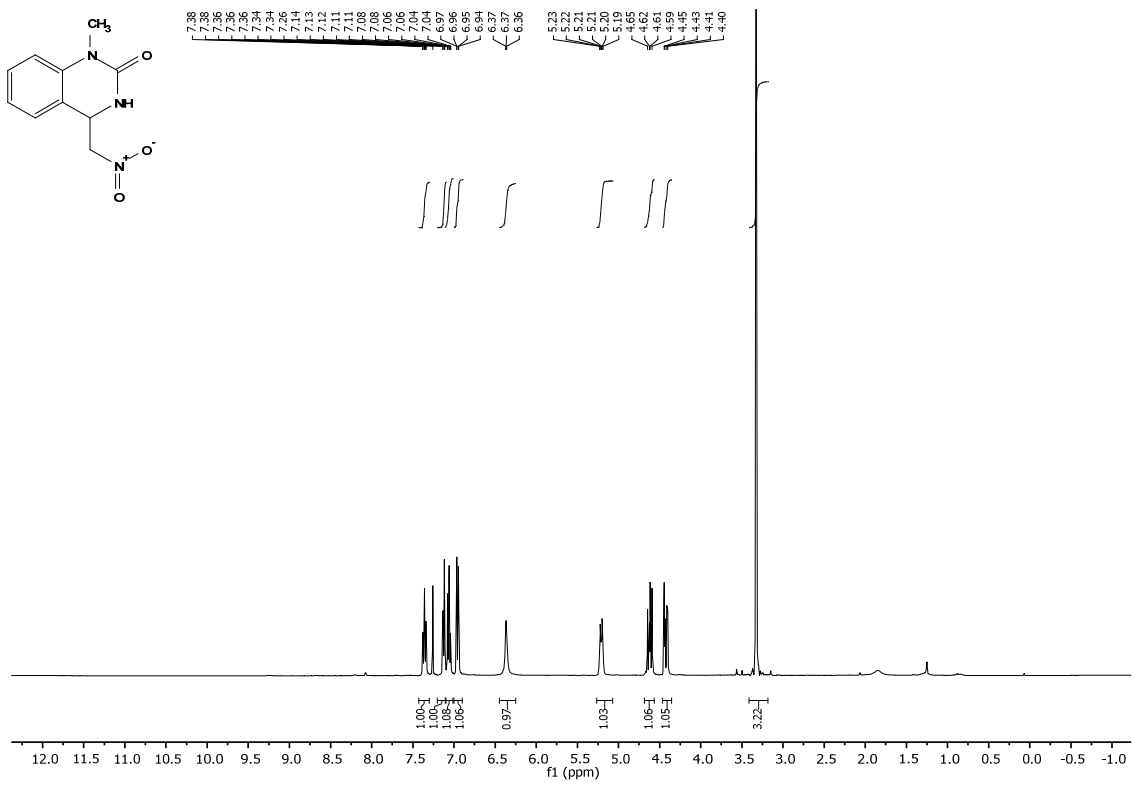
^{13}C NMR spectrum of **4f** (100 MHz, 25 °C, DMSO-d_6)



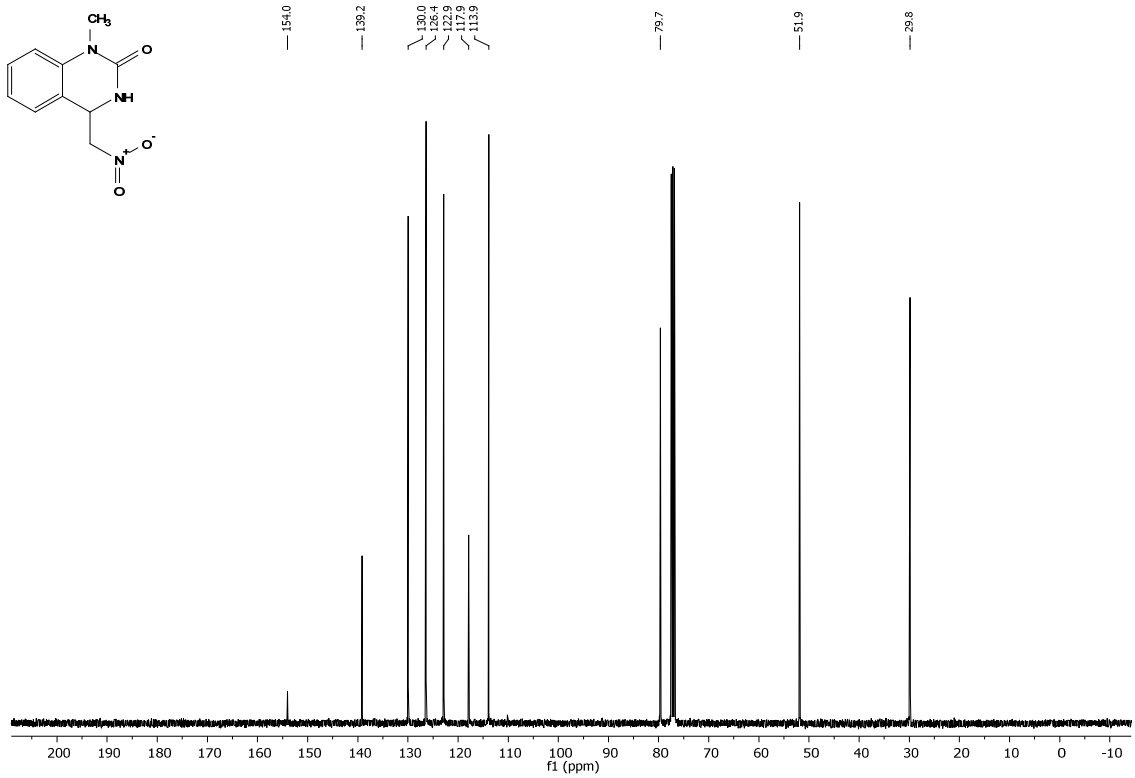
^{1}H NMR spectrum of **4g** (400 MHz, 25 °C, DMSO-d_6), 0.8:1 mixture of diastereomers



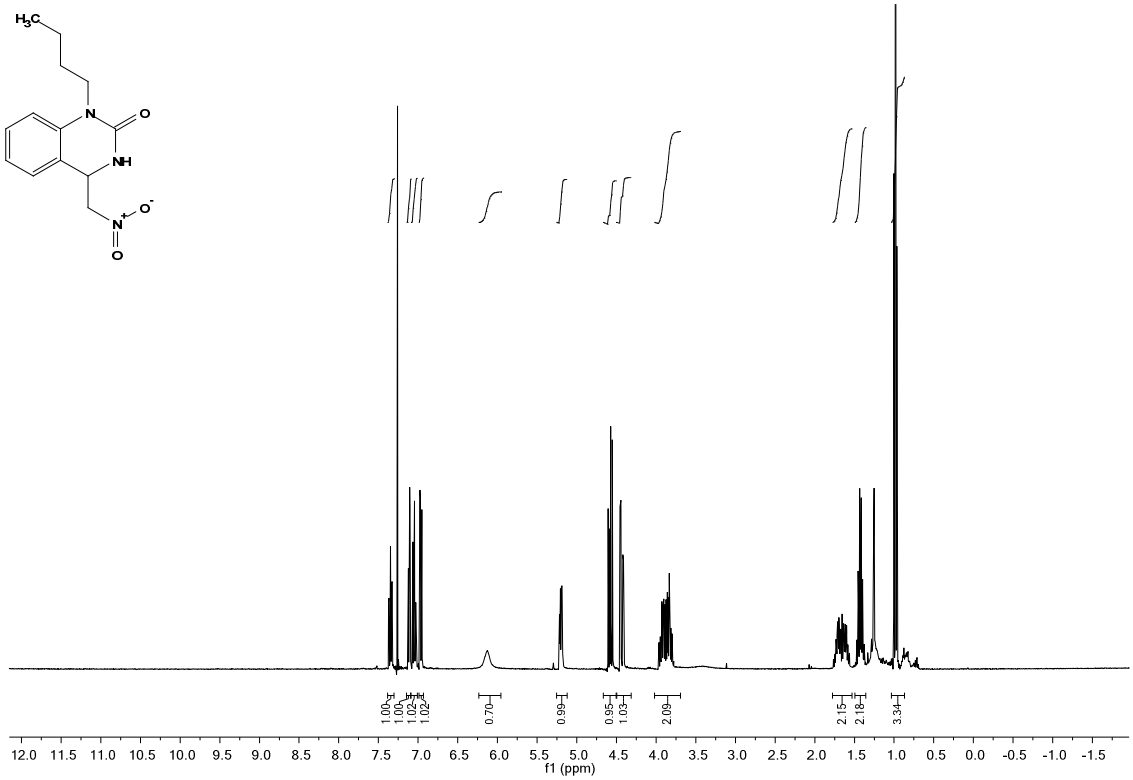
^{13}C NMR spectrum of **4g** (100 MHz, 25 °C, DMSO-d_6), 0.8:1 mixture of diastereomers



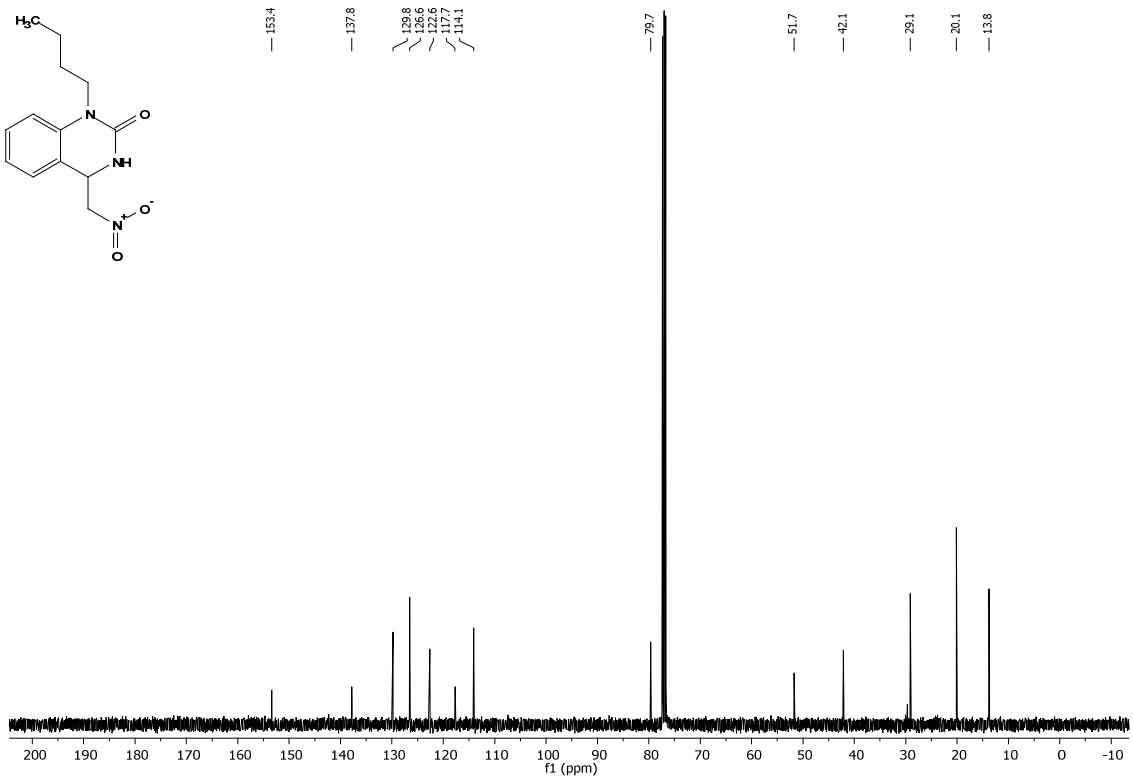
^{1}H NMR spectrum of **5a** (400 MHz, 25 °C, $CDCl_3$)



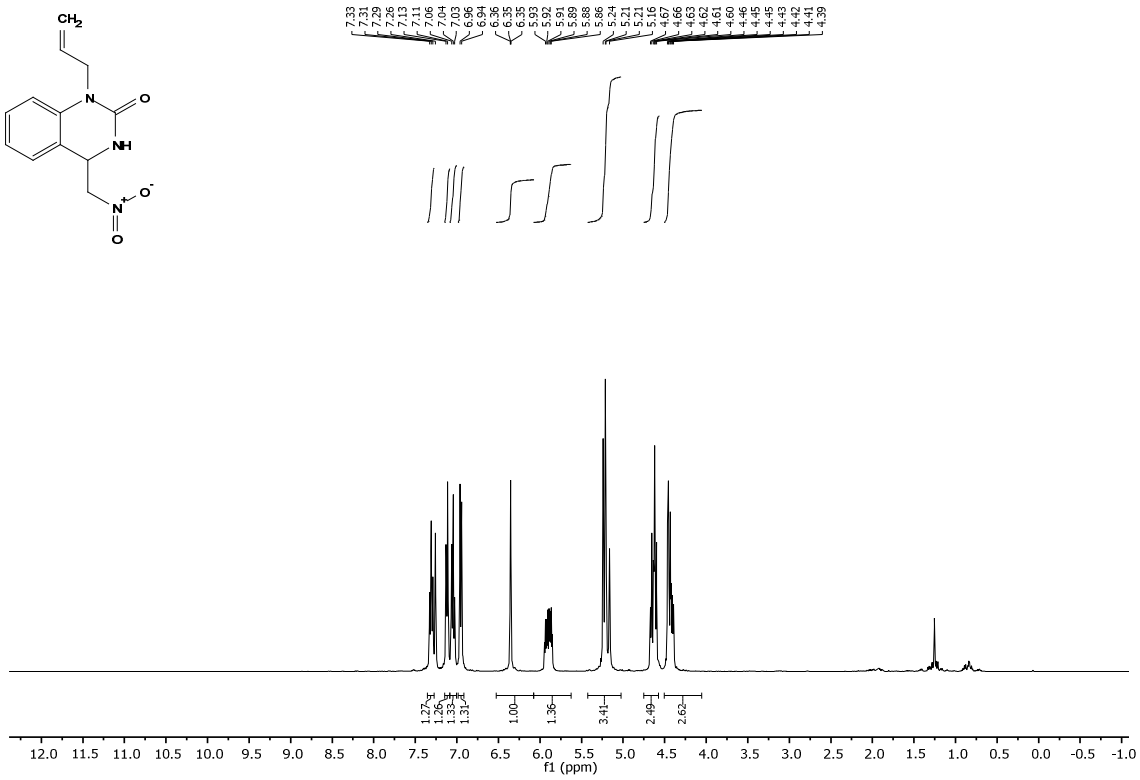
^{13}C NMR spectrum of **5a** (100 MHz, 25 °C, $CDCl_3$)



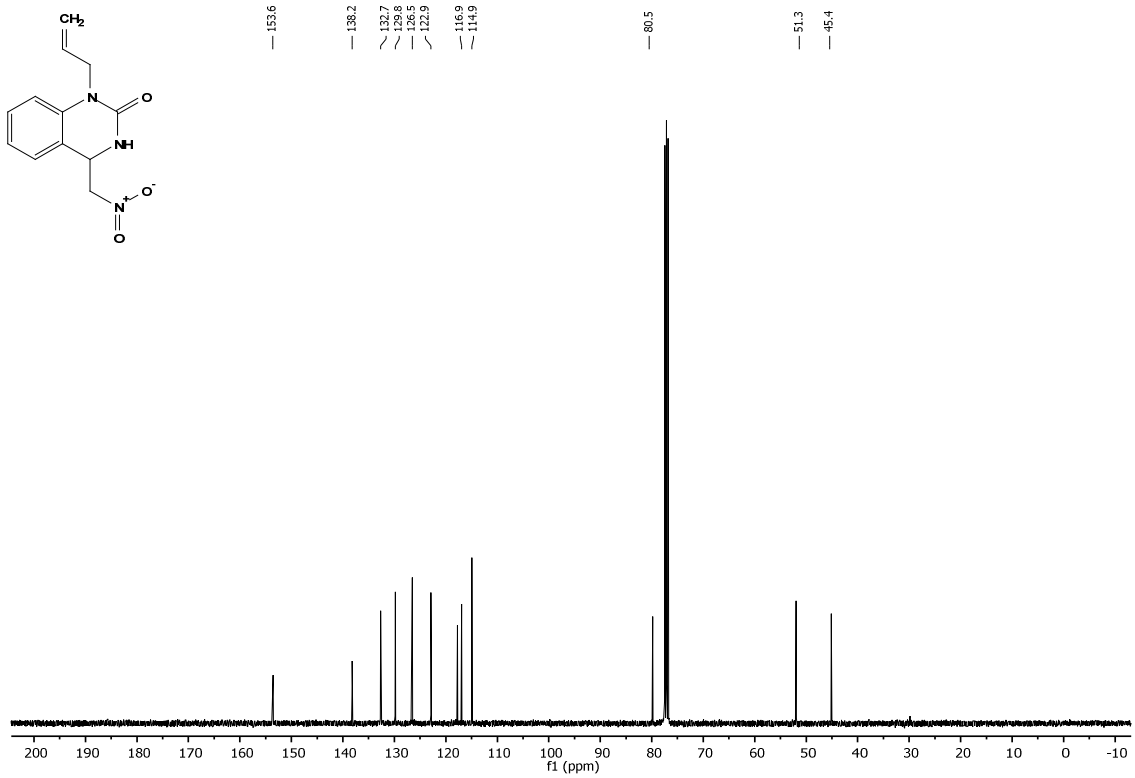
^{1}H NMR spectrum of **5b** (400 MHz, 25 °C, $CDCl_3$)



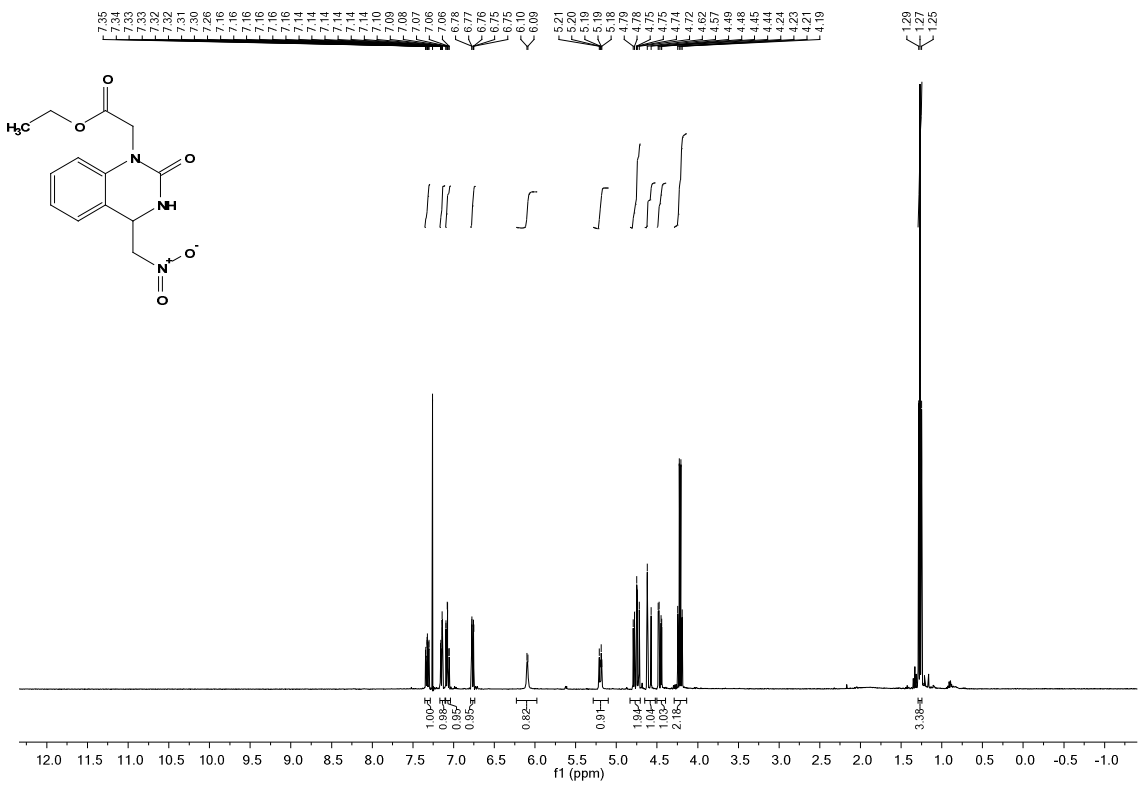
^{13}C NMR spectrum of **5b** (100 MHz, 25 °C, $CDCl_3$)



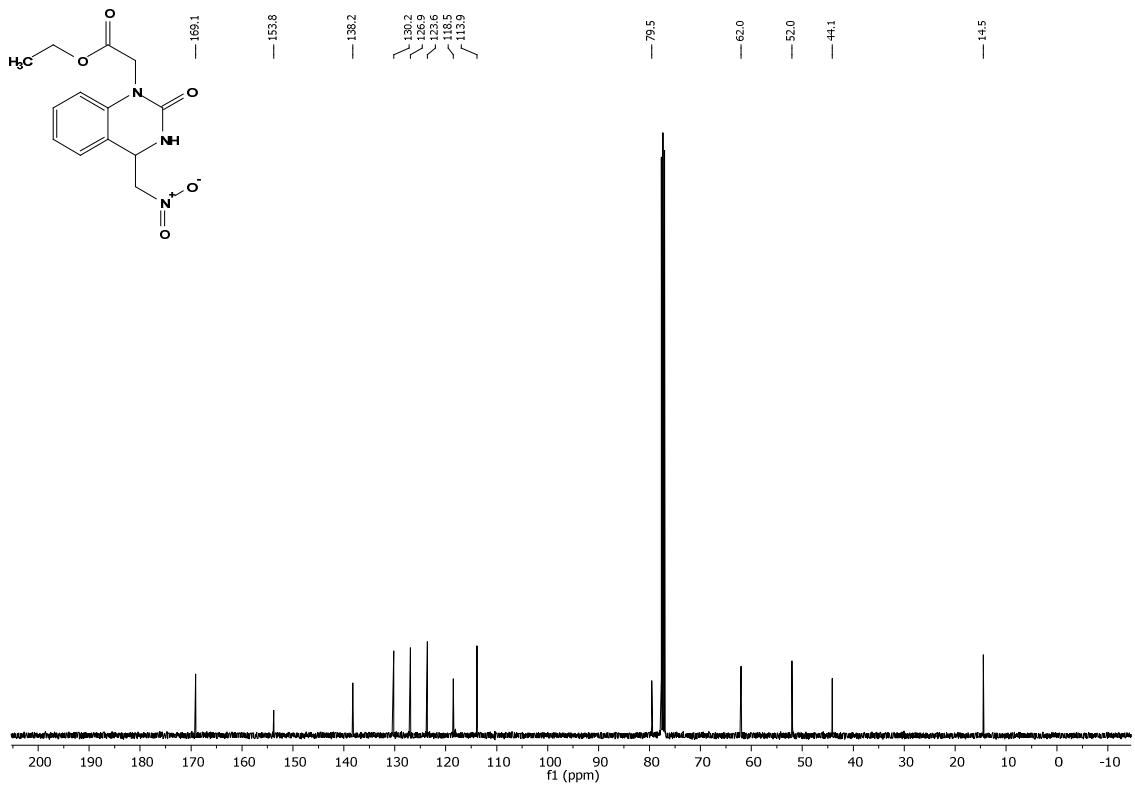
^{1}H NMR spectrum of **5c** (400 MHz, 25 °C, $CDCl_3$)



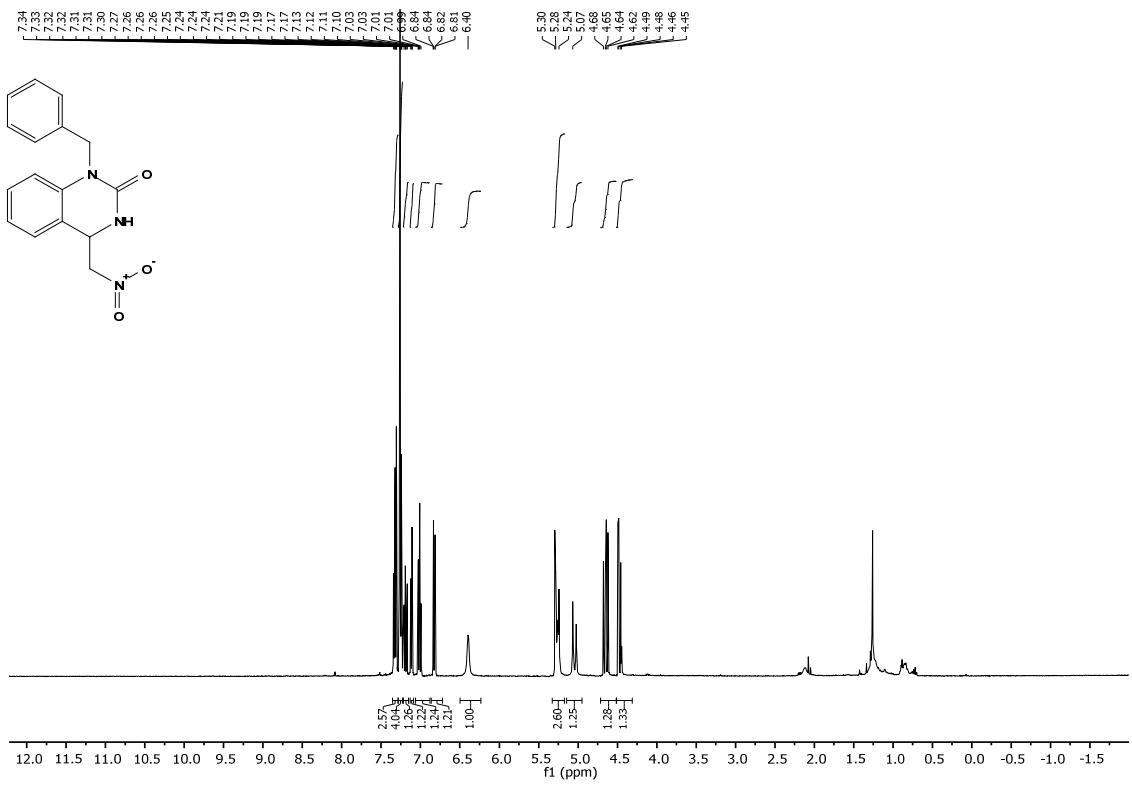
^{13}C NMR spectrum of **5c** (100 MHz, 25 °C, $CDCl_3$)



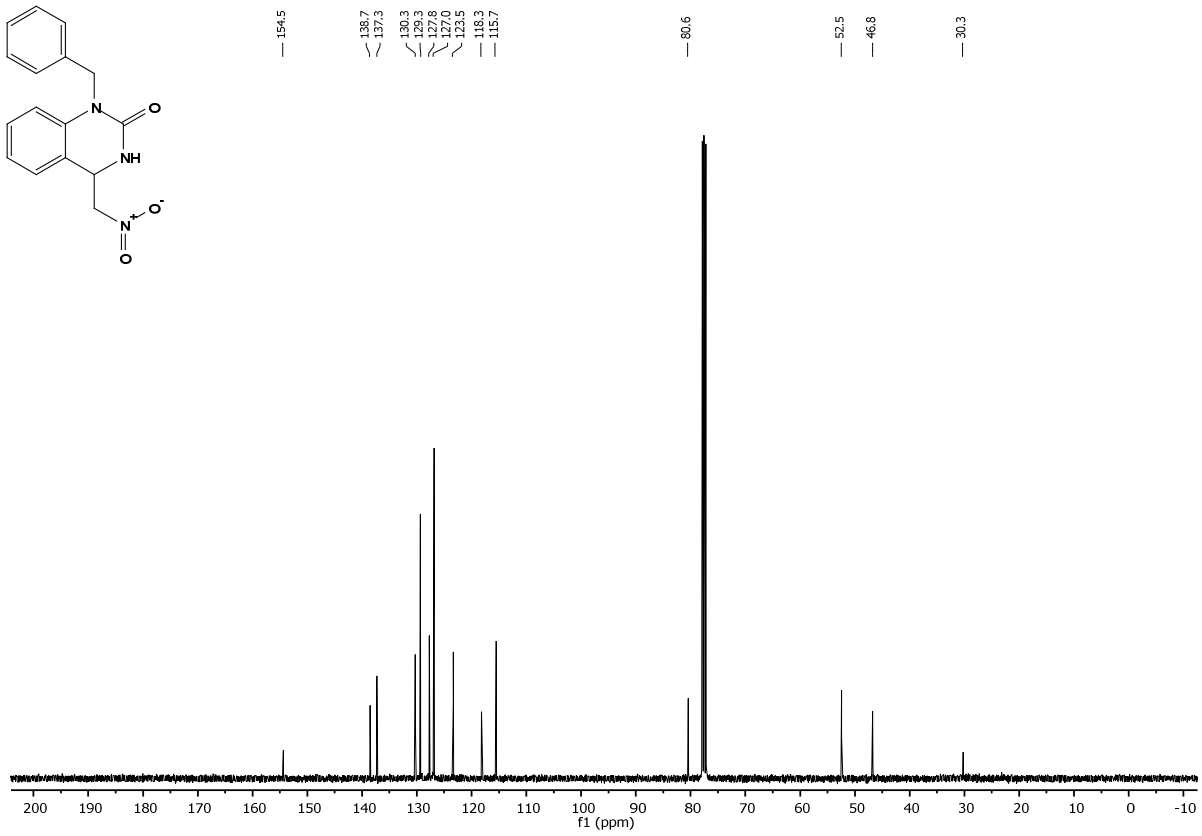
^{1}H NMR spectrum of **5d** (400 MHz, 25 °C, $CDCl_3$)



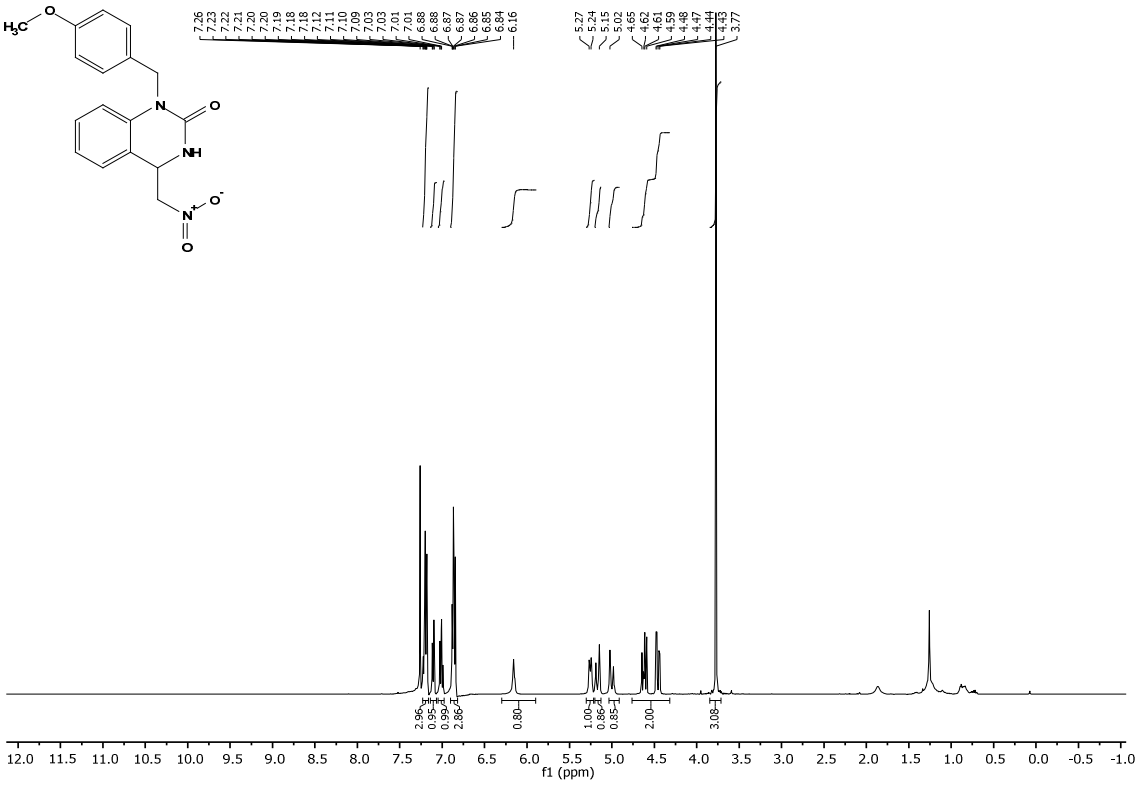
^{13}C NMR spectrum of **5d** (100 MHz, 25 °C, $CDCl_3$)



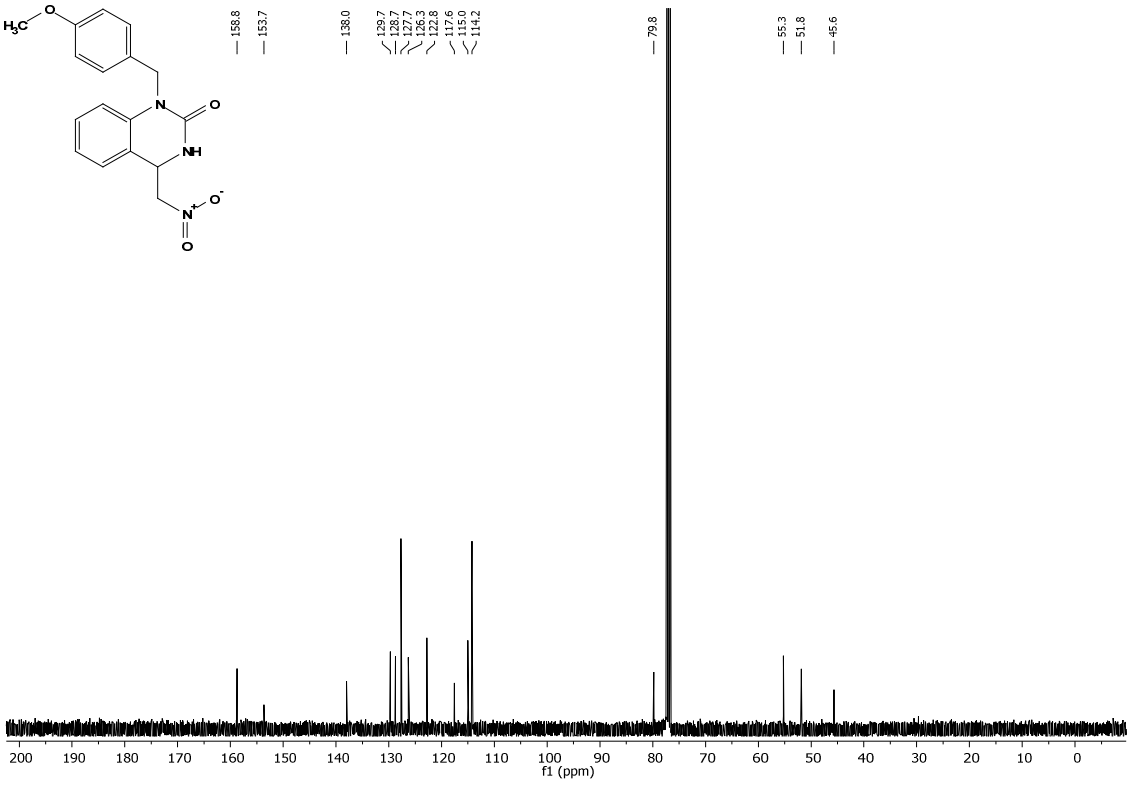
^{1}H NMR spectrum of **5e** (400 MHz, 25 °C, $CDCl_3$)



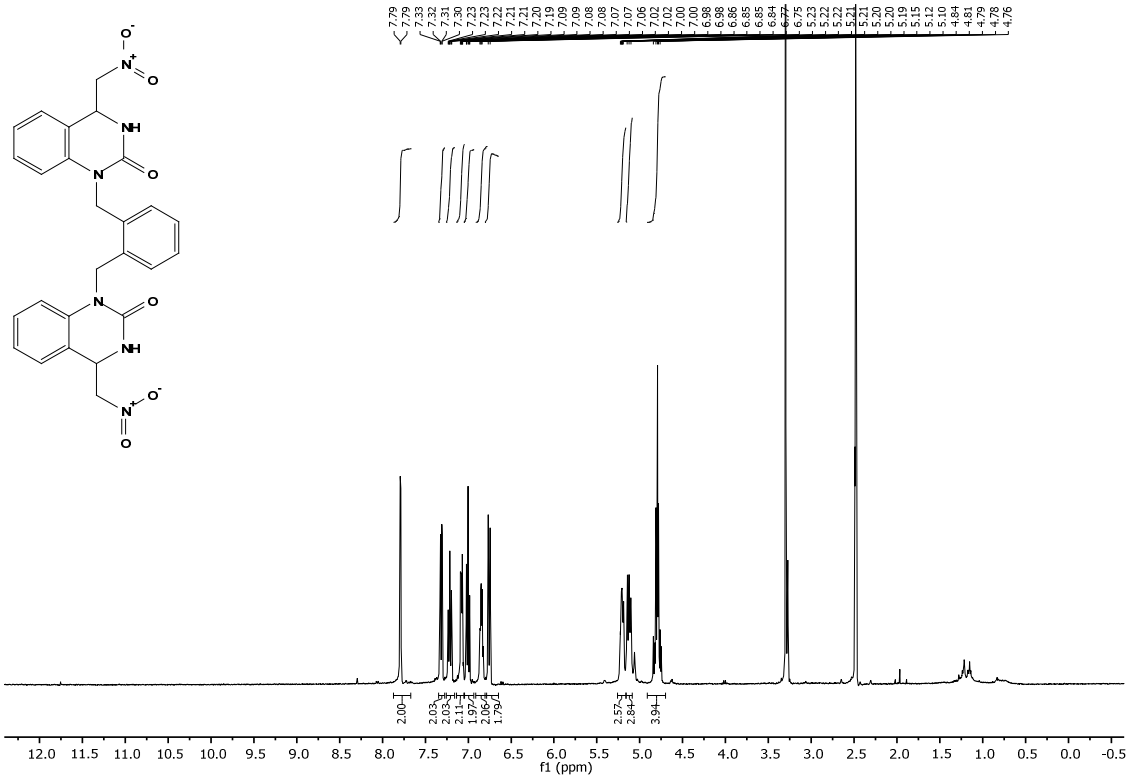
^{13}C NMR spectrum of **5e** (100 MHz, 25 °C, $CDCl_3$)



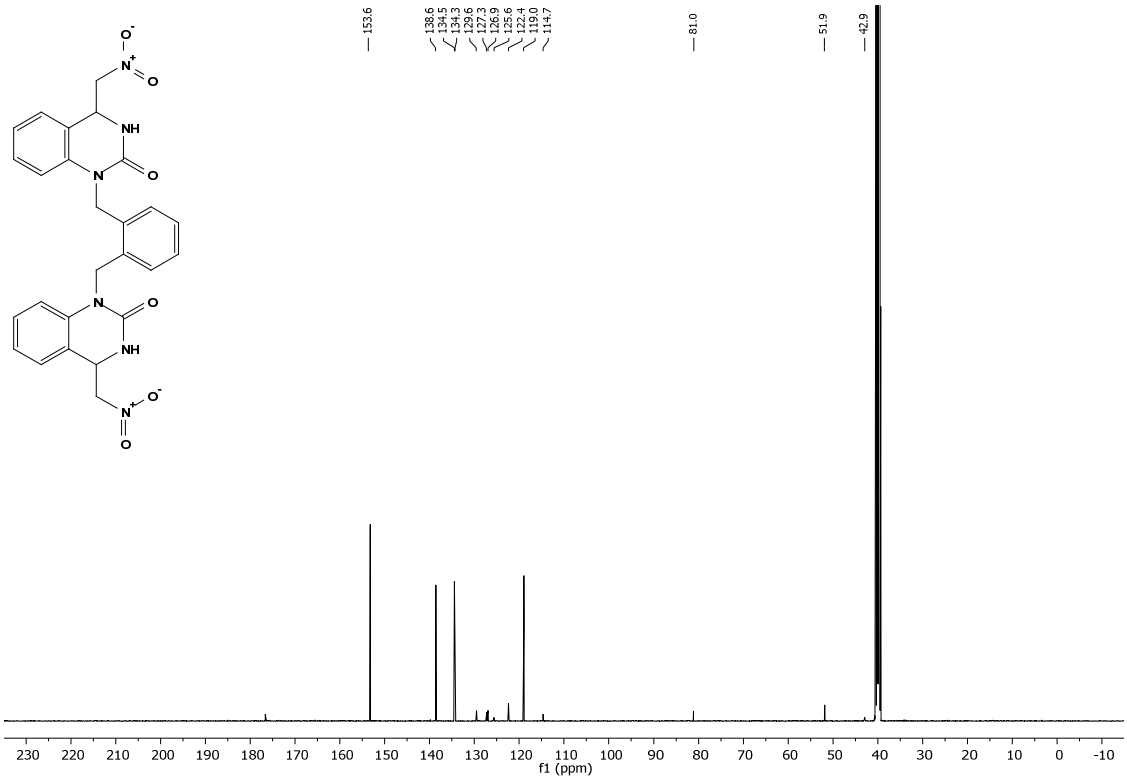
^{1}H NMR spectrum of **5f** (400 MHz, 25 °C, $CDCl_3$)



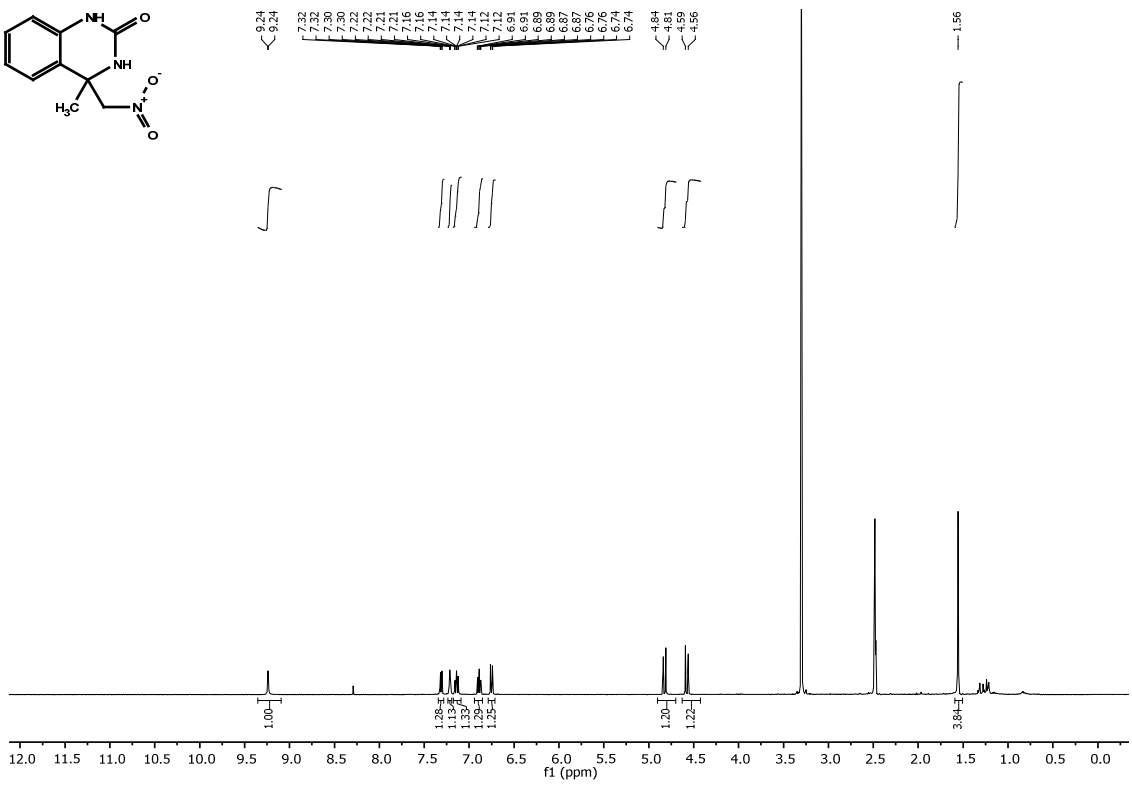
^{13}C NMR spectrum of **5f** (100 MHz, 25 °C, $CDCl_3$)



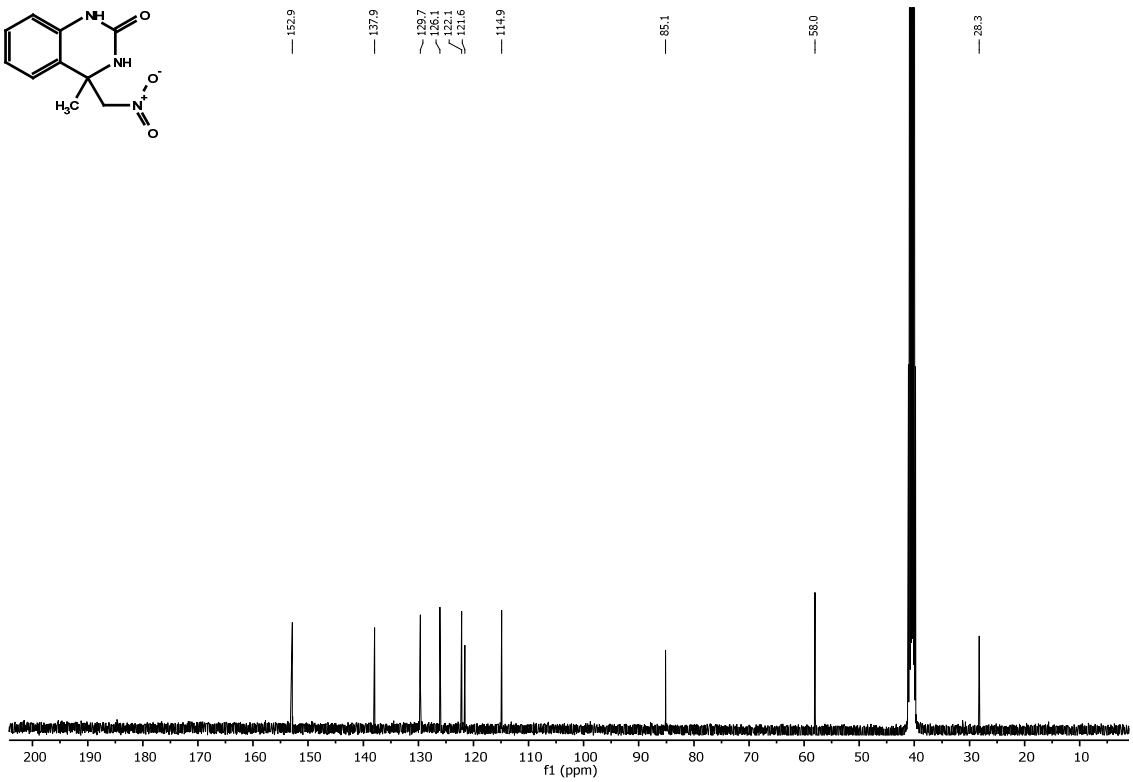
^{1}H NMR spectrum of **5g** (400 MHz, 25 °C, $CDCl_3$)



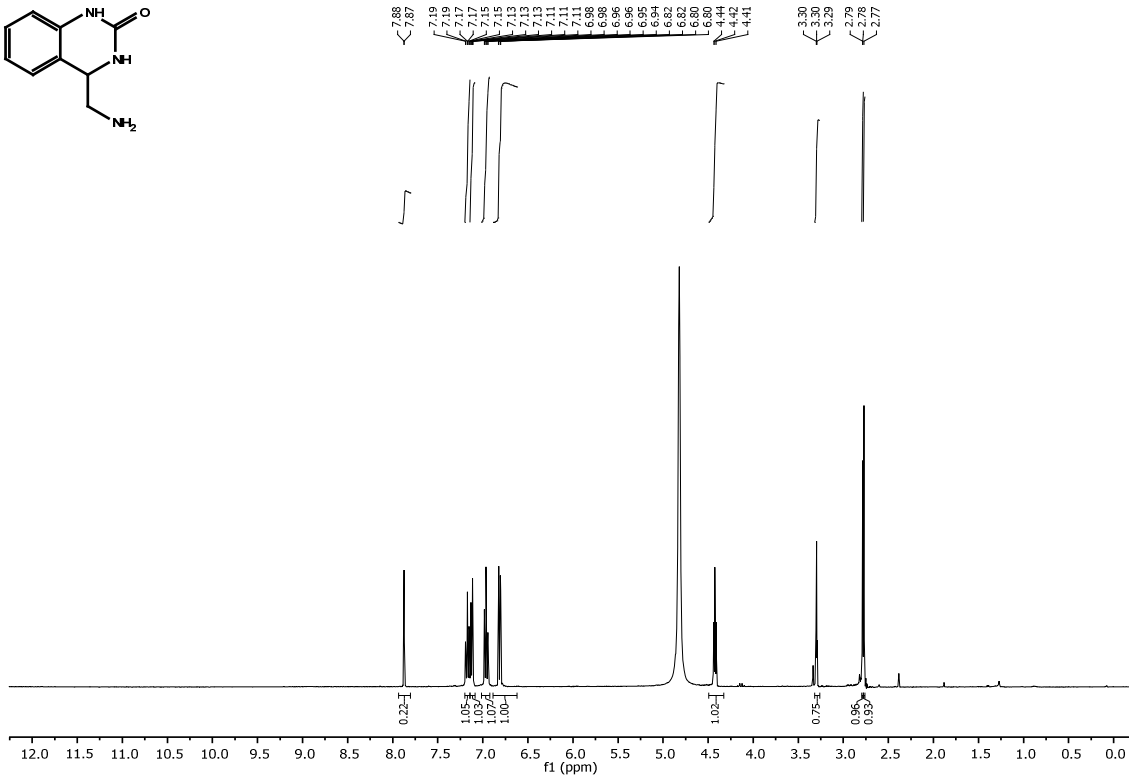
^{13}C NMR spectrum of **5g** (100 MHz, 25 °C, $CDCl_3$)



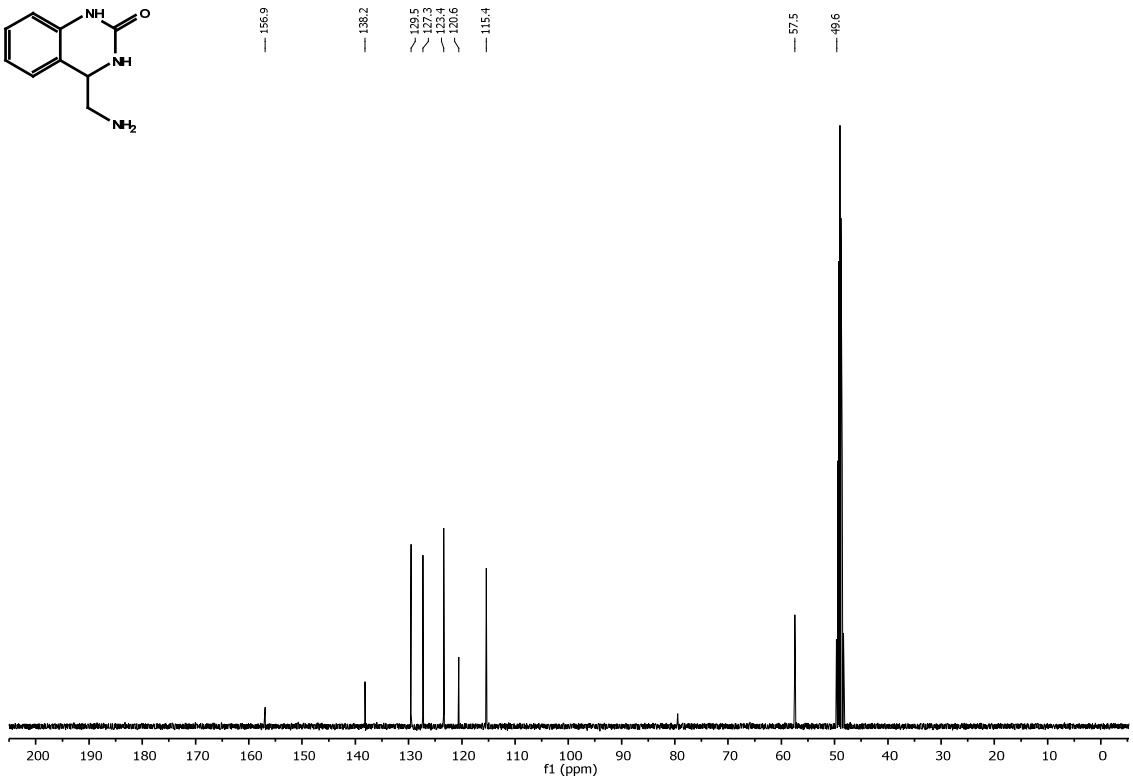
^{1}H NMR spectrum of **6** (400 MHz, 25 °C, $CDCl_3$)



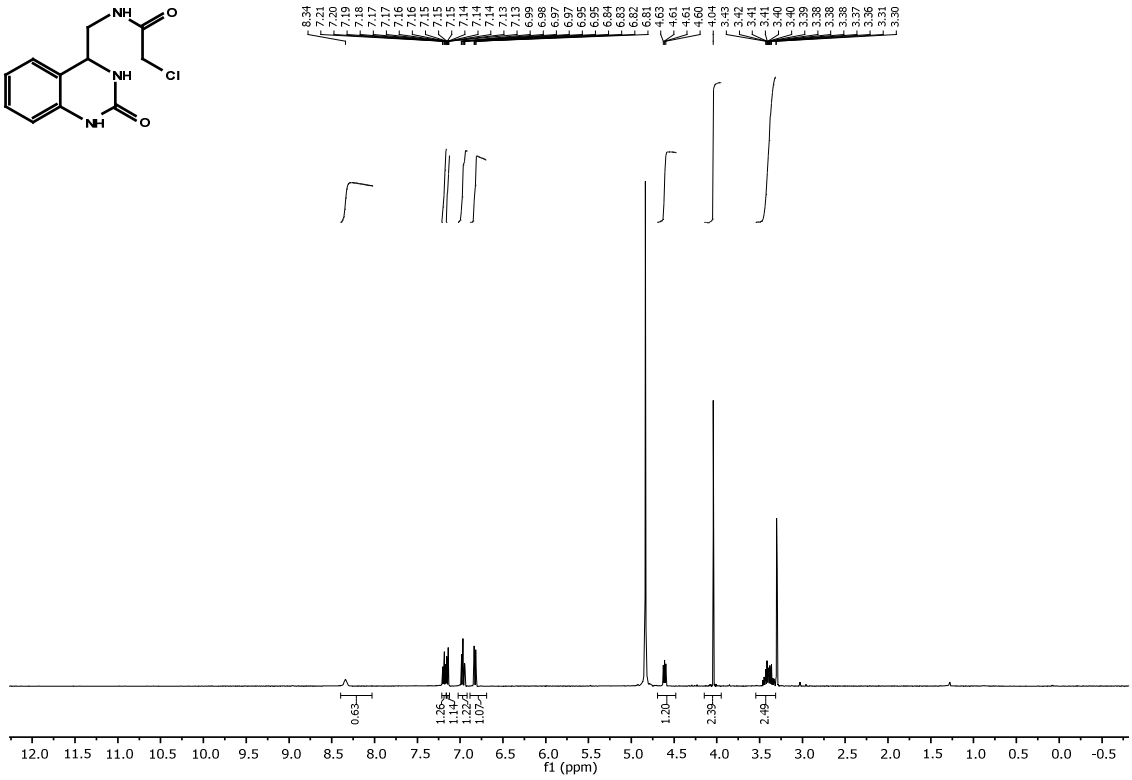
^{13}C NMR spectrum of **6** (100 MHz, 25 °C, CD_3OD)



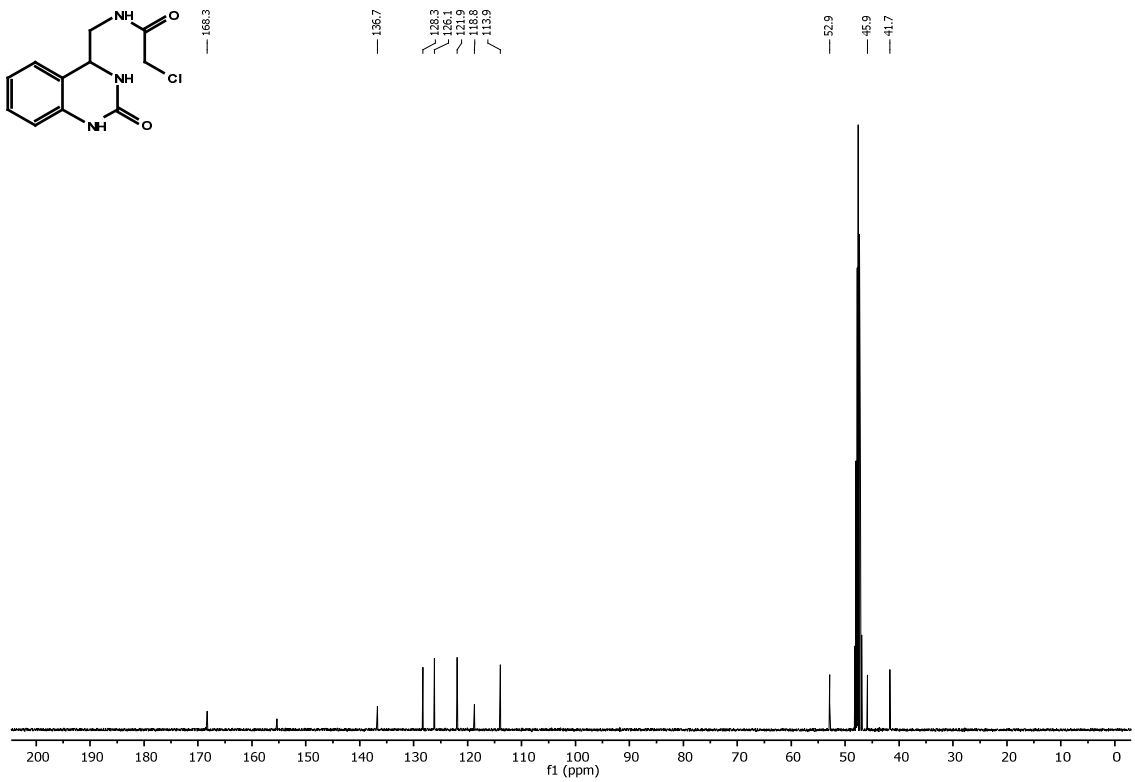
^{1}H NMR spectrum of **7** (400 MHz, 25 °C, CD_3OD)



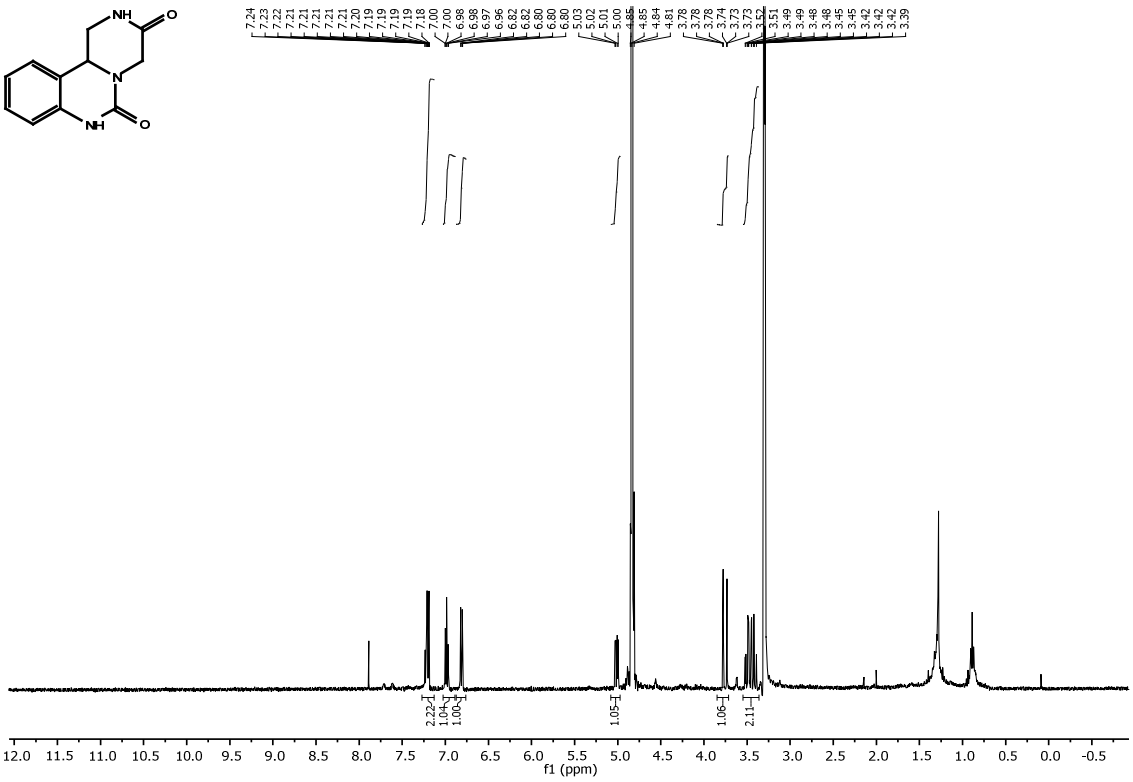
^{13}C NMR spectrum of **7** (100 MHz, 25 °C, CD_3OD)



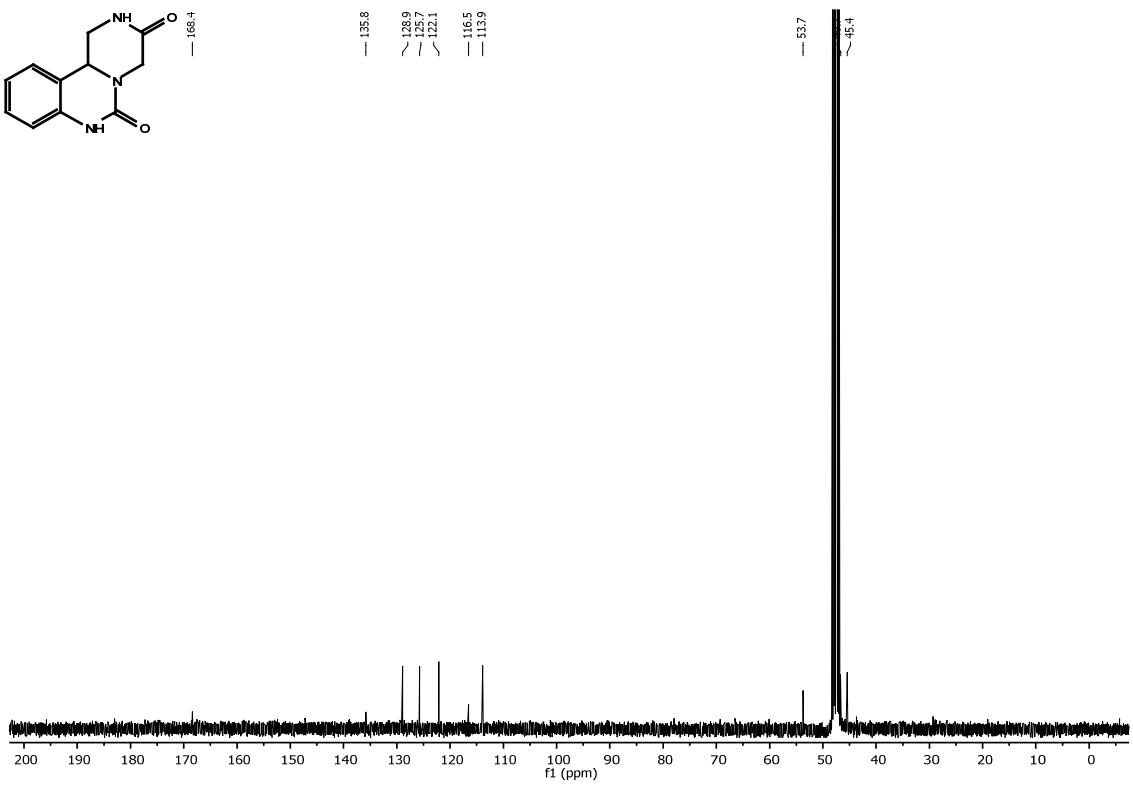
^{1}H NMR spectrum of **8** (400 MHz, 25 °C, CD_3OD)



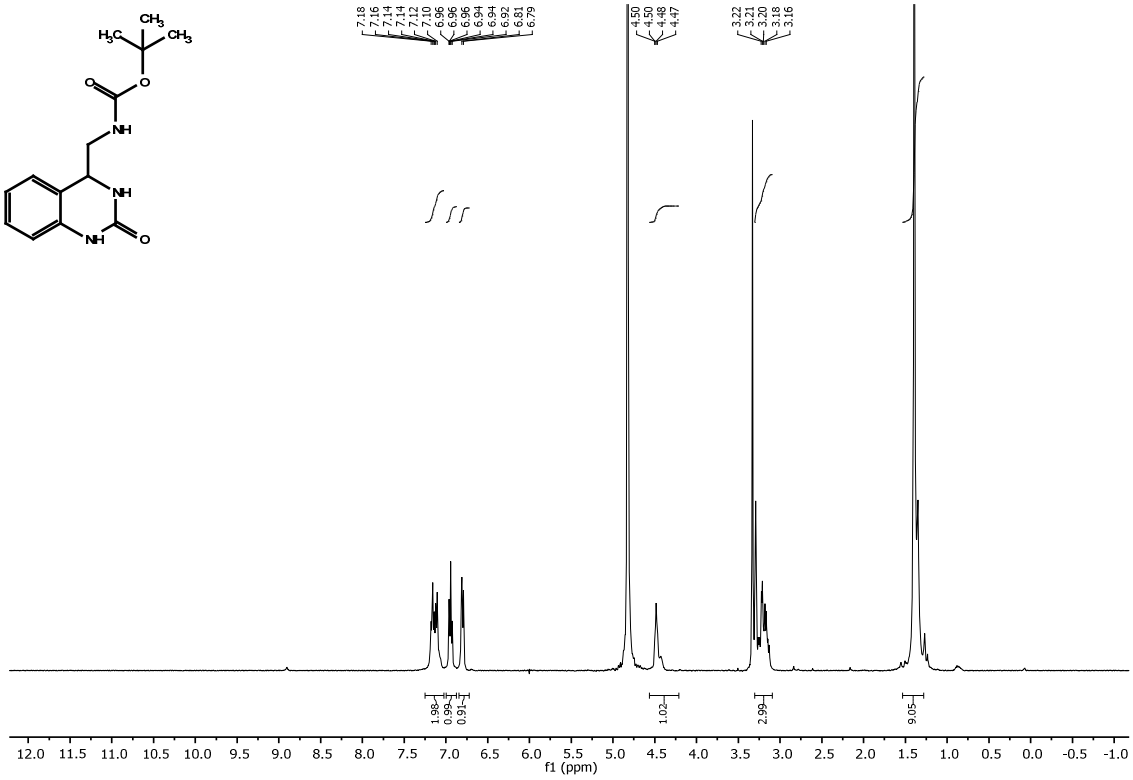
^{13}C NMR spectrum of **8** (100 MHz, 25 °C, CD_3OD)



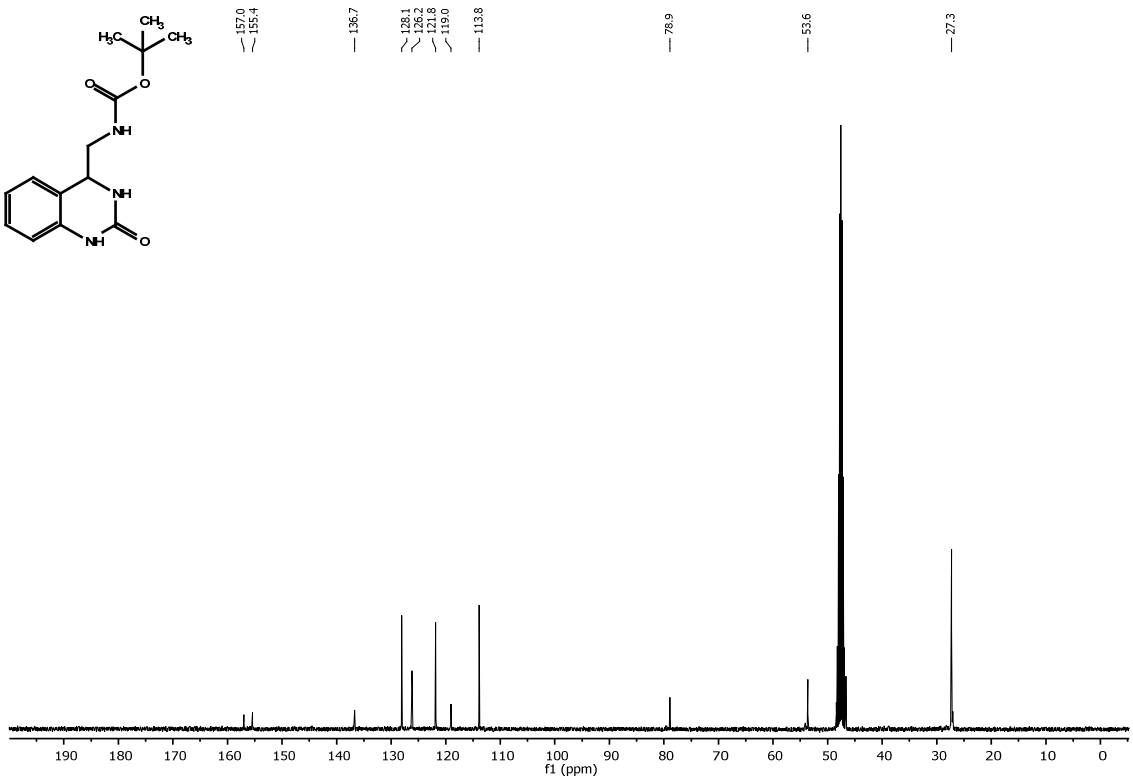
^{1}H NMR spectrum of **9** (400 MHz, 25 °C, CD_3OD)



^{13}C NMR spectrum of **9** (100 MHz, 25 °C, CD_3OD)

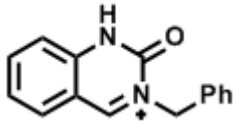


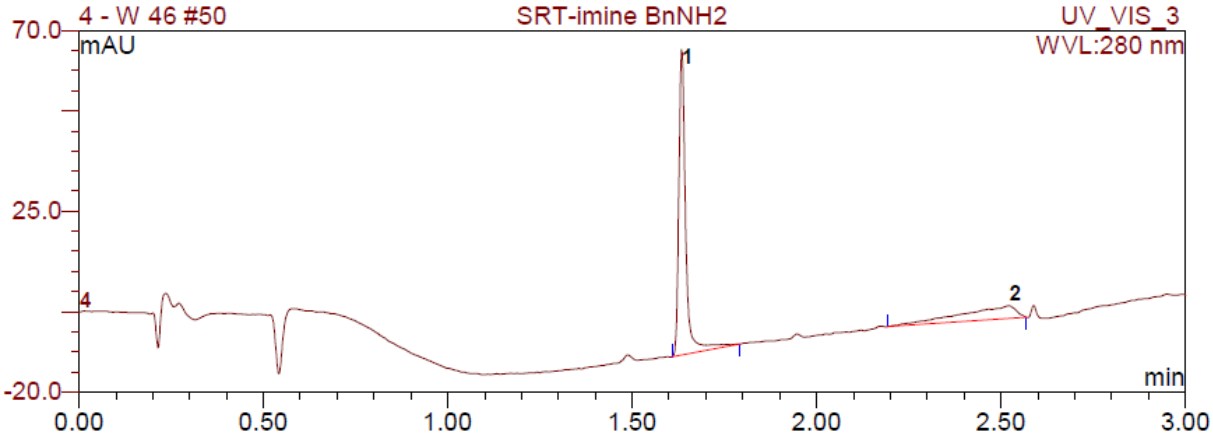
^{1}H NMR spectrum of **10** (400 MHz, 25 °C, CD_3OD)

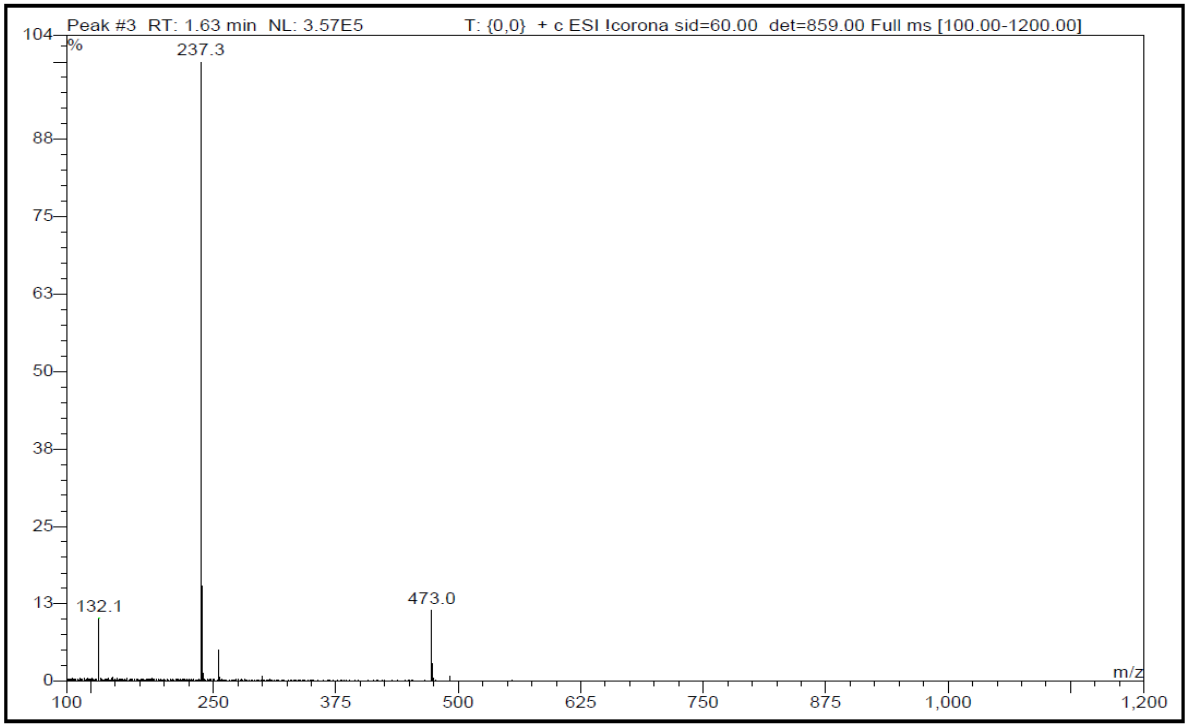


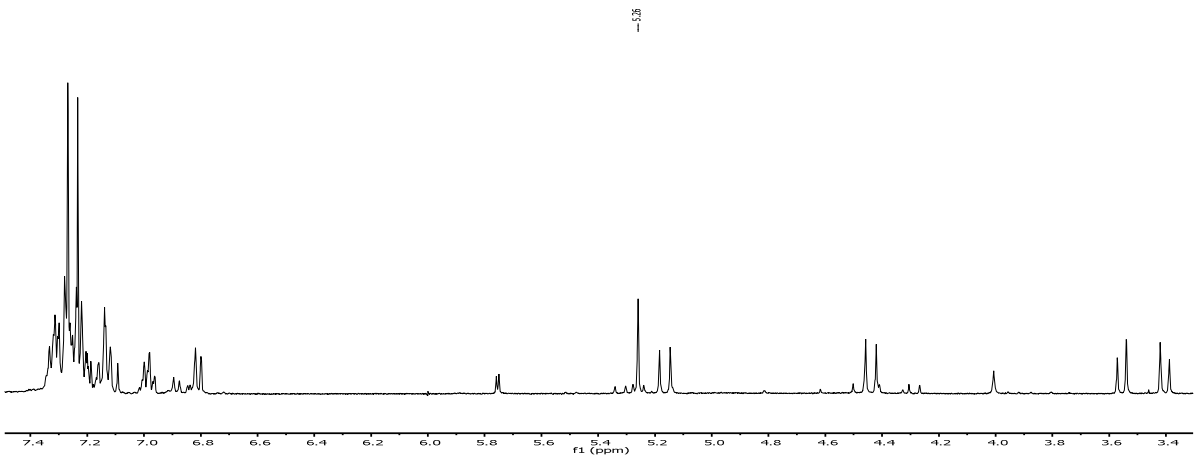
^{13}C NMR spectrum of **10** (100 MHz, 25 °C, CD_3OD)

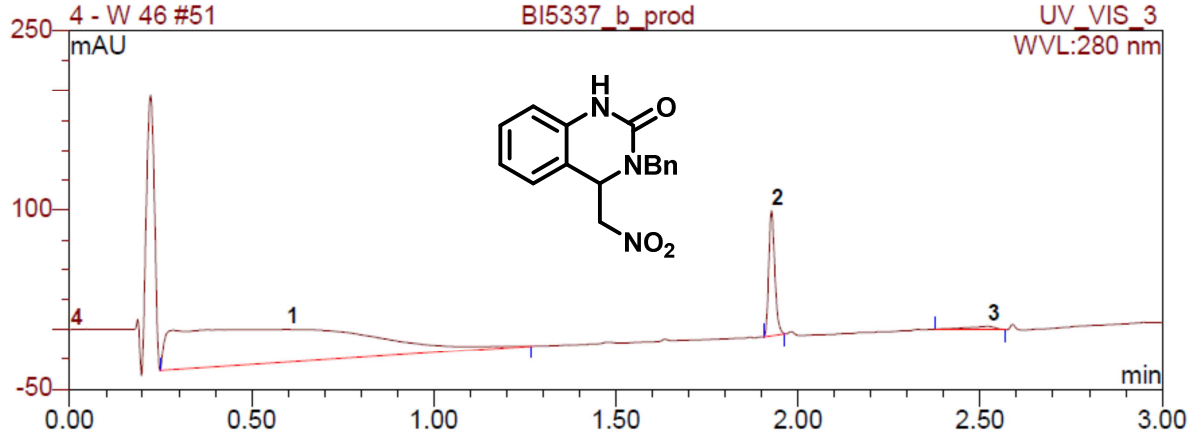
III. Detection of iminium ion *via* LC/UV/MS and NMR and subsequent cyclization to give compound 2d







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min
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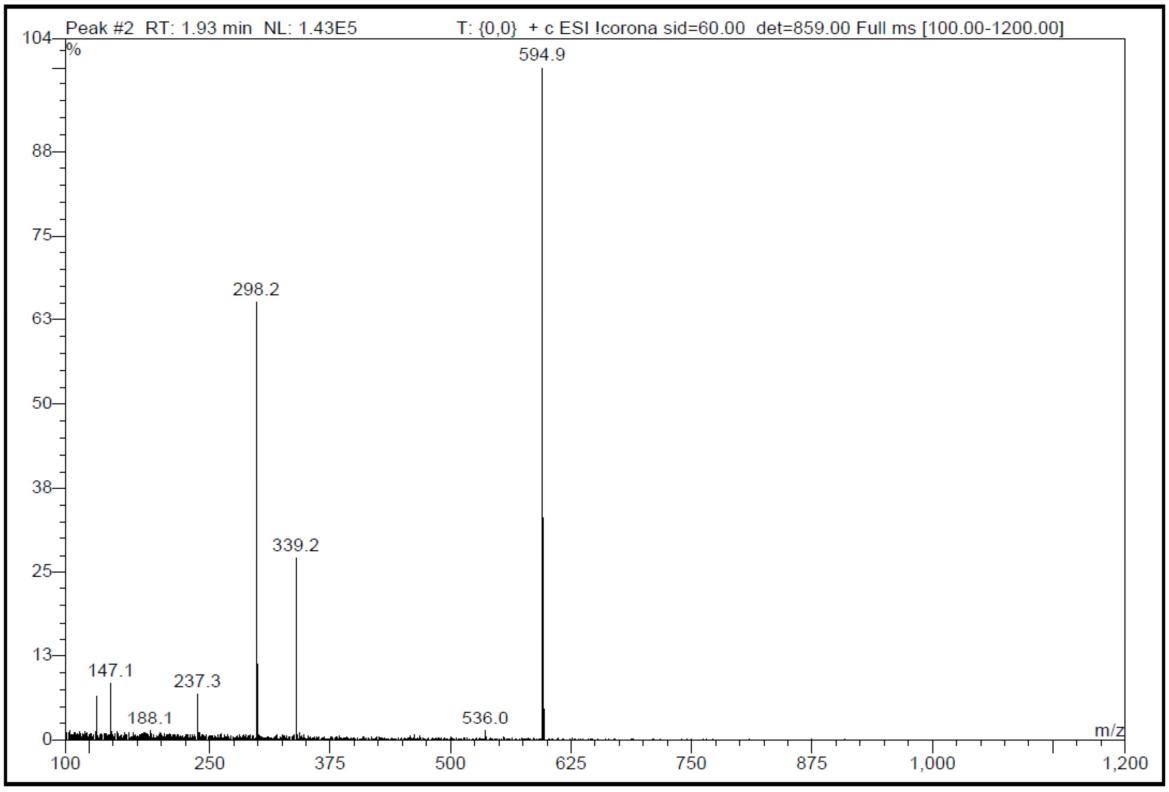
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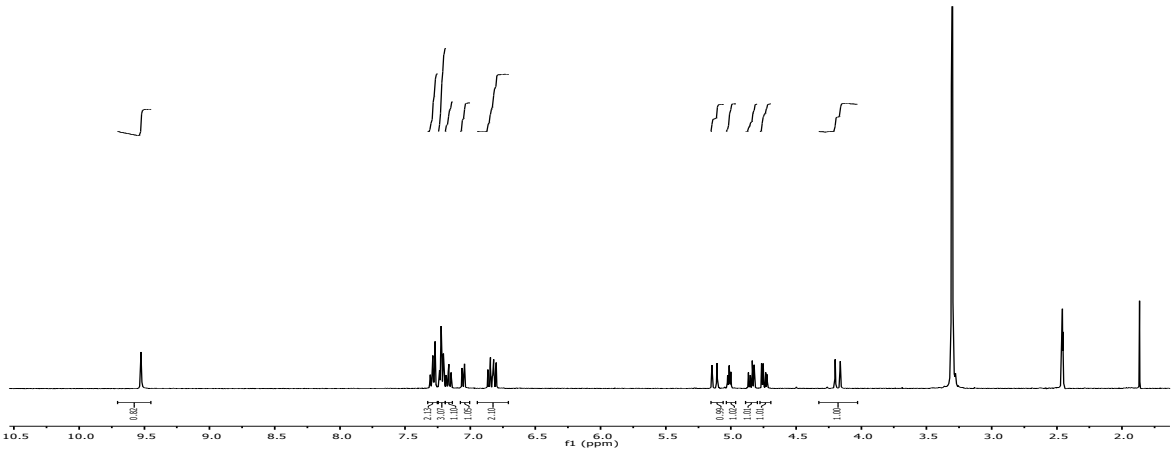
298.2
339.2
147.1
237.3
188.1
536.0


f1 (ppm)