Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting information for

Photoredox Catalyzed Release of Carbon-based Radicals from 2-Substituted-1,3-Imidazolidines

Adrián Luguera Ruiz, Elena Mariani, Stefano Protti,* Maurizio Fagnoni*

PhotoGreen Lab, Department of Chemistry, University of Pavia, Viale Taramelli 12, 27100 Pavia, Italy

1. Experimental details

1.1. General information.

The aldehydes employed for the synthesis of imidazolines **2** were commercially available and used as received, with the only exception of adamantane-1-carbaldehyde that was prepared according to a procedure previously reported.[S1] Olefins **2a-j** (see Figure S1) were commercially available and used as received. NMR spectra were recorded on 400 and 300 (for ^{1}H) or 100 and 75 (for ^{13}C) MHz spectrometers; chemical shifts are reported in ppm downfield from TMS and the attributions were made based on ^{1}H and ^{13}C NMR. Data for ^{1}H NMR are reported as follows: chemical shift referred to TMS (δ ppm), multiplicity (s = singlet, bs = broad singlet, d = doublet, t = triplet, q = quadruplet, quint = quintuplet, sext = sextuplet, sept = septuplet, m = multiplet), coupling constant (Hz) and integration. HRMS data were acquired using a X500B QTOF System (SCIEX, Framingham, MA 01701 USA) available at the CGS of the University of Pavia, equipped with the Twin Sprayer ESI probe and coupled to an ExionLC™ system (SCIEX). The SCIEX OS software 2.1.6 was used as operating platform. For MS detection the following parameters were applied: Curtain gas 30 psi, Ion source gas 1 45 psi, Ion source gas 2 55 psi, Temperature 450 °C, Polarity negative, Ion spray voltage -4500 V, TOF mass range 50-1600 Da, declustering potential -60 V and collision energy -10 V.
GC analyses were performed using a HP SERIES 5890 II equipped with a fire ion detector (FID, temperature 350 °C). Analytes were separated using a Restek Rtx-5MS (30 m×0.25 mm×0.25 μm) capillary column with nitrogen as a carrier gas at 1 mL min^{-1}. The injector temperature was 250 °C. The GC oven temperature was held at 80 °C for 2 min, increased to 250 °C by a temperature ramp of 10 °C min^{-1}, and held for 10 min. GC/MS analyses were carried out on a Thermo Scientific DSQII single quadrupole GC/MS system (TraceDSQII mass spectrometer, Trace GC Ultra gas chromatograph, TriPlus autosampler - ThermoFisher Scientific, Waltham, MA, USA). Chromatography was performed on a Rxi-5Sil MS capillary column (30 m length×0.25 mm ID×0.25 μm film thickness, Restek, Milan, Italy) with Helium (>99.99 %) as carrier gas at a constant flow-rate of 1.0 mL min^{-1}. An injection volume of 1 μL was employed. The injector temperature was set at 250 °C and it was operated in split mode, with a split flow of 10 mL min^{-1}. The oven temperature was programmed from 80 °C (isothermal for 2 min) to 220 °C at the rate of 10 °C min^{-1}, then from 220 °C to 300 °C (isothermal for 5 min) at the rate of 4 °C min^{-1}. Mass transfer line temperature was set at 260 °C. Total GC running time was 41 min. All mass spectra were acquired with an electron ionization system (EI, Electron Impact mode) with ionization energy of 70 eV and source temperature of 250°C, with spectral acquisition in Full Scan mode, positive polarity, over a mass range of 35–650

Da with a scan rate of 940 amu s^{-1}. The chromatogram acquisition, detection of mass spectral peaks and their waveform processing were performed using Xcalibur MS Software Version 2.1 (Thermo Scientific Inc.). Assignment of chemical structures to chromatographic peaks was based on the comparison with the databases for GC-MS NIST Mass Spectral Library (NIST 08) and Wiley Registry of Mass Spectral Data (8^{th} Edition).

Cyclic Voltammetry for compound **1c** was carried out by means of a Amel model 4330 module equipped with a 20 mL standard three-electrode cell with a glassy carbon (0.49 cm^2 geometrical area) working electrode, a platinum wire as auxiliary electrode and an Ag/AgCl, 3 M NaCl reference electrode, all obtained from BASi Electrochemistry. Acetonitrile containing 0.1 M lithium perchlorate were used as solvent and supporting electrolyte, scanning the potential in the range from 0 mV to + 2500 mV, with a 5 mM compound concentration and a scan speed of 50 mV s^{-1}.

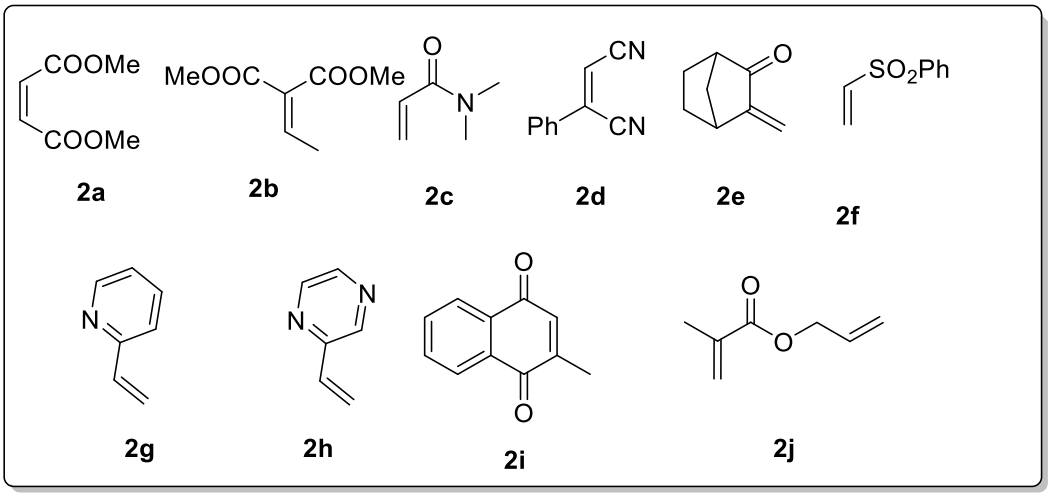


Figure S1: Electron-deficient olefins used in this work.

1.2 General Procedure for the synthesis of 2-substituted *N,N'*-dimethylimidazolidines.

The desired imidazolidines **1a-j** (Figure S1) were synthesized by adapting a procedure previously described for the synthesis of 1,3-oxazolidines.[S2] *N,N'*-dimethylethylendiamine (1 equiv) was added to a suspension of $MgSO_4$ (25 mg $mmol^{-1}$) and the corresponding aldehyde (1 equiv) in Et_2O (1.7 mL $mmol^{-1}$). The reaction mixture was refluxed and stirred overnight, and the resulting residue was diluted with DCM, filtered, and concentrated in vacuo to yield the desired imidazolidine that was employed for the photocatalytic step without any further purification.

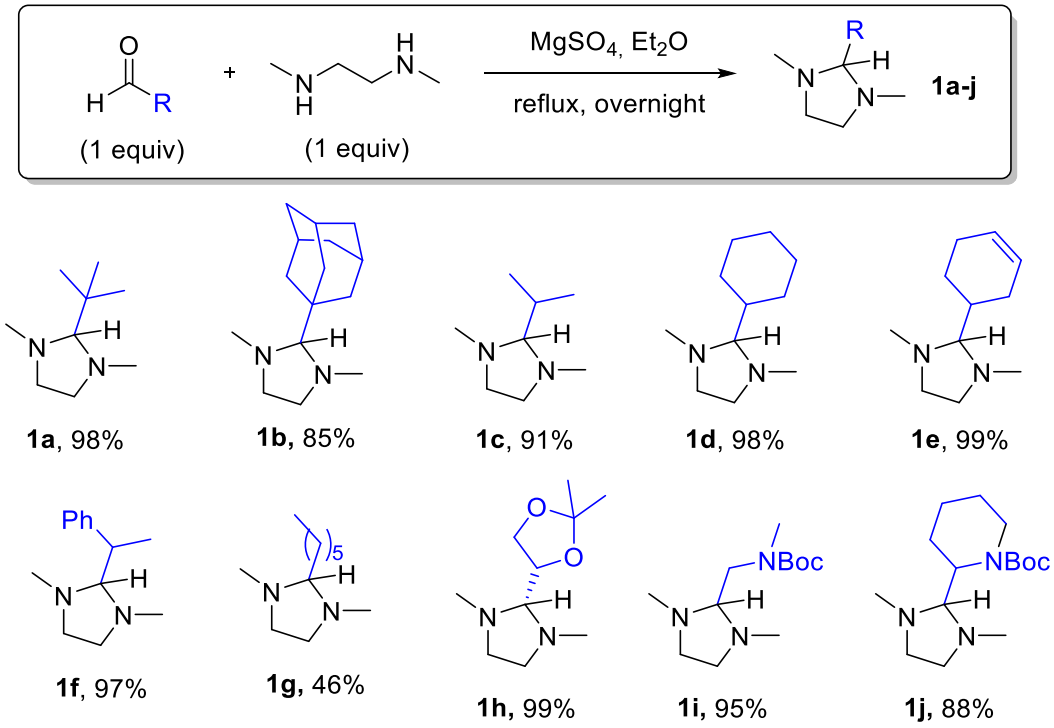


Figure S2: Synthesis of imidazolidines **1a-j.**

2-*tert*-Butyl-1,3-dimethylimidazolidine (1a). From *N,N'*-dimethylethylendiamine (1.26 mL, 11.6 mmol, 1 equiv), $MgSO_4$ (290 mg, 25 mg $mmol^{-1}$), pivalaldehyde (1.25 mL, 11.6 mmol, 1 equiv) in Et_2O (20 mL). The crude mixture was diluted in DCM, filtered and concentrated in vacuo yielding **1a** (colourless oil, 1.78 g, 98% yield).

1a. ^{1}H NMR (300 MHz, acetone-d_6) δ 2.99–2.86 (m, 2H), 2.74–2.57 (m, 3H), 2.48 (s, 6H), 0.86 (s, 9H). ^{13}C NMR (75 MHz, acetone-d_6) δ 99.0, 55.4, 51.6, 46.8, 38.3, 35.9, 26.6. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_9H_{20}N_2$ 157.1660, found 157.1699.

2-Adamantan-1-yl)-1,3-dimethylimidazolidine (1b). From *N,N'*-dimethylethylendiamine (0.65 mL, 6.0 mmol, 1 equiv), $MgSO_4$ (150 mg, 25 mg $mmol^{-1}$) adamantane-1-carbaldehyde[S1] (0.99 g, 6.0 mmol, 1 equiv) in Et_2O (10 mL). The crude mixture was diluted in DCM, filtered and concentrated in vacuo yielding **1b** (pale yellow oil, 1.20 g, 85% yield).

1b. ^{1}H NMR (300 MHz, acetone-d_6) δ 3.04–2.78 (m, 3H), 2.69–2.57 (m, 2H), 2.46 (s, 6H), 1.98–1.86 (m, 3H), 1.74–1.49 (m, 12H).). ^{13}C NMR (75 MHz, acetone-d_6) δ 99.5, 55.5, 47.1, 40.3, 39.4, 38.3, 29.5. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{15}H_{26}N_2$ 235.2130, found 235.2169.

2-*iso*-Propyl-1,3-dimethylimidazolidine (1c). From *N,N'*-dimethylethylendiamine (0.25 mL, 2.8 mmol, 1 equiv), $MgSO_4$ (70 mg, 25 mg $mmol^{-1}$), isopropylaldehyde (0.3 mL, 2.8 mmol, 1 equiv) in Et_2O (5 mL). The crude mixture was diluted in DCM, filtered and concentrated in vacuo yielding **1c** (colourless oil, 0.36 g, 91% yield).

1c. ^{1}H NMR (300 MHz, acetone-d_6) δ 2.95–2.84 (m, 2H), 2.56–2.43 (m, 3H), 2.36 (s, 6H), 1.71 (ddp, J = 10.0, 6.8, 3.1 Hz, 1H), 0.89 (d, J = 6.8 Hz, 6H). ^{13}C NMR (75 MHz, acetone-d_6) δ 95.4, 54.9, 44.0, 32.2, 18.5. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_8H_{18}N_2$ 143.1504, found 143.1543.

2-Cyclohexyl-1,3-dimethylimidazolidine (1d). From *N,N'*-dimethylethylendiamine (0.19 mL, 1.78 mmol, 1 equiv), $MgSO_4$ (45 mg, 25 mg $mmol^{-1}$), cyclohexylcarbaldehyde (0.22 mL, 1.8 mmol, 1 equiv) in Et_2O (3 mL). The crude mixture was diluted in DCM, filtered and concentrated in vacuo yielding **1d** (colourless oil, 0.3 g, 98% yield).

1d. ^{1}H NMR (300 MHz, acetone-d_6) δ 2.93–2.83 (m, 2H), 2.55–2.51 (m, 1H), 2.51–2.44 (m, 2H), 2.36 (s, 6H), 1.77–1.69 (m, 4H), 1.68–1.60 (m, 1H), 1.38 (tt, J = 11.6, 2.8 Hz, 1H), 1.27–1.07 (m, 5H). ^{13}C NMR (75 MHz, acetone-d_6) δ 95.2, 54.8, 44.3, 43.2, 28.0, 27.7. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{11}H_{22}N_2$ 183.1817, found 183.1856.

2-Cyclohexenyl-1,3-dimethylimidazolidine (1e). From *N,N'*-dimethylethylendiamine (0.2 mL, 1.8 mmol, 1 equiv), $MgSO_4$ (45 mg, 25 mg $mmol^{-1}$), 3-cyclohexene-1-carboxaldehyde (0.21 mL, 1.8 mmol, 1 equiv) in Et_2O (3 mL). The crude mixture was diluted in DCM, filtered and concentrated in vacuo yielding **1e** (colourless oil, 0.34 g, 99% yield).

1e. ^{1}H NMR (300 MHz, acetone-d_6) δ 5.73–5.53 (m, 2H), 3.01–2.81 (m, 2H), 2.65 (d, J = 3.3 Hz, 1H), 2.61–2.44 (m, 2H), 2.39 (s, 3H), 2.37 (s, 3H), 2.05–1.93 (m, 4H), 1.82–1.71 (m, 1H), 1.71–1.60 (m, 1H), 1.49–1.25 (m, 1H). ^{13}C NMR (75 MHz, acetone-d_6) δ 128.2, 127.3, 94.4, 54.8, 54.4, 44.6, 43.3, 38.7, 27.6, 26.6, 26.2. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{11}H_{20}N_2$ 181.1660, found 183.1699.

2-(1-Phenylethyl)-1,3-dimethylimidazolidine (1f). From *N,N'*-dimethylethylendiamine (0.16 mL, 1.5 mmol, 1 equiv), $MgSO_4$ (38 mg, 25 mg $mmol^{-1}$), 2-phenylpropanal (0.2 mL, 1.5 mmol, 1 equiv) in Et_2O (3 mL). The crude mixture was diluted in DCM, filtered off, and the obtained solution was concentrated in vacuo affording **1f** (pale yellow oil, 0.3 g, 97% yield).

1f. ^{1}H NMR (300 MHz, acetone-d_6) 7.42–7.19 (m, 4H), 7.19–7.08 (m, 1H), 2.99–2.84 (m, 3H), 2.84–2.72 (m, 1H), 2.55–2.39 (m, 2H), 2.36 (s, 3H), 2.06 (s, 3H), 1.29 (d, J = 6.8 Hz, 3H). δ ^{13}C NMR (75 MHz, acetone- d_6) 145.7, 129.3, 128.5, 126.6, 95.6, 54.7, 54.1, 44.5, 43.4, 42.7, 15.1. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{13}H_{20}N_2$ 205.1699, found 205.1699.

2-Hexyl-1,3-dimethylimidazolidine (1g). From *N,N'*-dimethylethylendiamine (0.24 mL, 2.2 mmol, 1 equiv), was added to a suspension of $MgSO_4$ (55 mg, 25 mg $mmol^{-1}$), heptanal (0.31 mL, 2.2 mmol, 1 equiv) in Et_2O (4 mL). The crude mixture was diluted in DCM, filtered off, and the obtained solution was concentrated in vacuo to give **1g** (colourless oil, 0.38 g, 46% yield).

1g. ^{1}H NMR (300 MHz, acetone-d_6) 2.99 (dd, J = 6.4, 2.1 Hz, 2H), 2.49 (t, J = 3.4 Hz, 1H), 2.36 (dd, J = 6.4, 2.1 Hz, 2H), 2.25 (s, 6H), 1.72–1.30 (m, 3H). ^{13}C NMR (75 MHz, acetone-d_6) 89.2, 53.8, 40.8, 32.8, 31.7, 24.1, 23.3, 14.4, 14.4. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{11}H_{24}N_2$ 185.2012, found 185.2007.

2-(2,2-Dimethyl-1,3-dioxolan-4-yl)-1,3-dimethylimidazolidine (1h). From *N,N'*-dimethylethylendiamine (0.11 mL, 1.15 mmol, 1 equiv), $MgSO_4$ (30 mg, 25 mg $mmol^{-1}$), 2,2-dimethyl-1,3-dioxolane-4-carbaldehyde (0.14 mL, 1.15 mmol, 1 equiv) in Et_2O (3 mL). The crude mixture was diluted in DCM, filtered off, and the obtained solution was concentrated in vacuo affording **1h** (yellowish oil, 0.23 g, 99% yield).

1h. ^{1}H NMR (400 MHz, chloroform- *d*) δ 4.12–4.06 (m, 1H), 4.06–3.99 (m, 1H), 3.90 (dd, J = 7.9, 6.4 Hz, 1H), 3.12–2.98 (m, 2H), 2.97 (d, J = 5.4 Hz, 1H), 2.64–2.56 (m, 2H), 2.52 (s, 3H), 2.49 (s, 3H), 1.43 (s, 3H), 1.34 (s, 3H). δ ^{13}C NMR (100 MHz, chloroform- *d*) δ 109.1, 89.6, 78.5, 66.3, 54.3, 53.8, 44.5, 43.0, 26.6, 25.1 HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{10}H_{20}N_2O_2$ 201.1598, found 201.1589.

***tert*-Butyl methyl((1,3-dimethylimidazolidin-2-yl)methyl)carbamate (1i).** From *N,N'*-dimethylethylendiamine (0.16 mL, 1.4 mmol, 1 equiv), $MgSO_4$ (36 mg, 25 mg $mmol^{-1}$), *N*-Boc-2-aminoacetaldehyde (0.24 mL, 1.4 mmol, 1 equiv) in Et_2O (3 mL). The crude mixture was diluted in DCM, filtered off, and the obtained solution was concentrated in vacuo affording **1i** (pale yellow oil, 0.33 g, 95% yield).

1i. ^{1}H NMR (400 MHz, chloroform- *d*) δ 3.23 (d, *J* = 11.3 Hz, 2H), 3.10–3.04 (m, 2H), 2.96 (s, 3H), 2.92–2.87 (m, 1H), 2.53–2.47 (m, 2H), 2.41 (s, 6H), 1.47–1.43 (m, 9H).^{13}C NMR (100 MHz, chloroform- *d*) δ 155.9, 88.0, 79.5, 77.4, 53.5, 52.8, 42.4, 36.3, 28.6. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{12}H_{25}N_3O_2$ 244.2020, found 244.2012.

***tert*-Butyl 2-(1,3-dimethylimidazolidin-2-yl)piperidine-1-carboxylate (1j).** From *N,N'*-dimethylethylendiamine (0.25 mL, 2.3 mmol, 1 equiv), $MgSO_4$ (58 mg, 25 mg mmol^{-1}), *tert*-butyl 2-formylpiperidine-1-carboxylate (0.5 g, 2.3 mmol, 1 equiv) in Et_2O (4 mL). The crude mixture was diluted in DCM, filtered off, and the obtained solution was concentrated in vacuo affording **1j** (pale yellow oil, 0.58 g, 88% yield).

1j. ^{1}H NMR (300 MHz, acetone-d_6) 3.95 (dd, *J* = 13.2, 4.9 Hz, 1H), 3.88–3.77 (m, 1H), 3.26 (d, *J* = 6.7 Hz, 1H), 3.04–2.86 (m, 3H), 2.71–2.57 (m, 2H), 2.45 (s, 3H), 2.44 (s, 3H), 1.83–1.51 (m, 3H), 1.48–1.36 (m, 12H). ^{13}C NMR (75 MHz, acetone- d_6) δ 206.1, 156.0, 89.6, 78.7, 54.5, 54.0, 45.5, 43.5, 28.7, 26.0, 24.9, 20.4. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{15}H_{29}N_3O_2$ 284.2333, found 284.2325.

2. Photoredox catalyzed preparation of compounds 3-43

General procedure. A solution of the chosen imidazolidine **1a-j** (1.5 equiv), olefin **2a-j** (1 equiv) and [Acr-Mes]$^+$(BF$_4$)$^-$ or 4CzIPN (10 mol%) in DCE (5 mL) was prepared in a Pyrex glass vessel (see Figure S3). The solution was irradiated for 24 h at 405 nm by means of a 18W EvoluChem lamp. The resulting solution was then concentrated in vacuo and the residue was purified by flash column chromatography using an Isolera apparatus (Biotage) and a SiO_2 cartridge (eluant: cyclohexane/ethyl acetate mixture except where indicated).

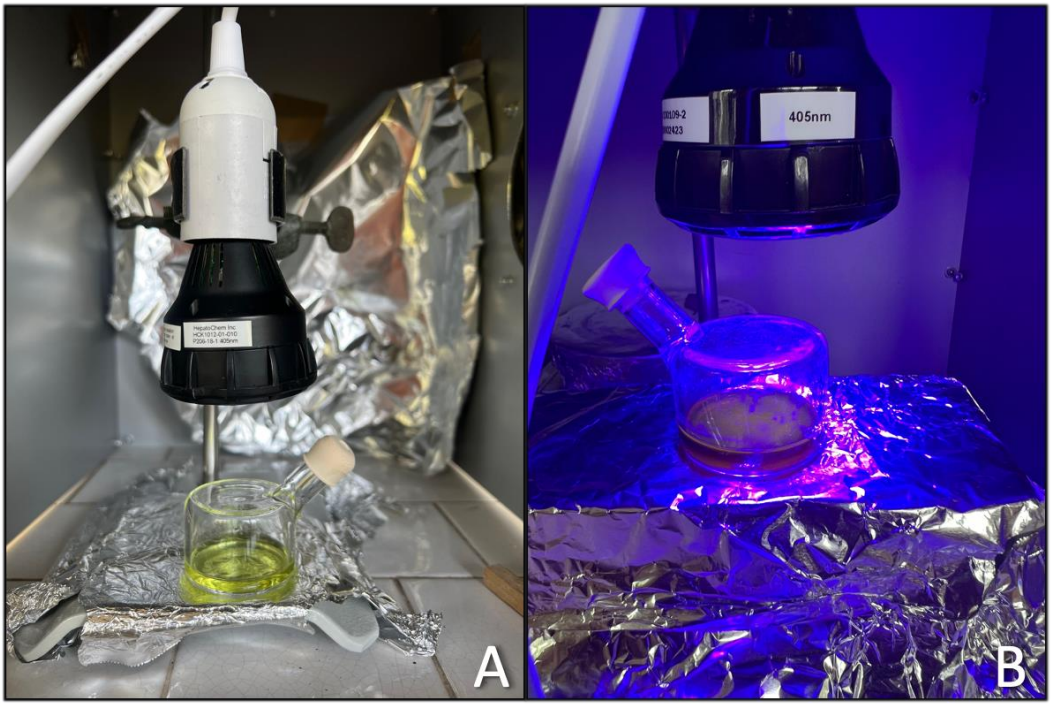


Figure S3. A) Irradiation set up before irradiation. B) Irradiation set up during irradiation.

Dimethyl 2-(*tert*-butyl)succinate (3). From **1a** (58 mg, 0.375 mmol, 1.5 equiv), dimethyl maleate (**2a,** 31 µL, 0.25 mmol, 1 equiv), [Acr-Mes]$^+$(BF$_4$)$^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 8/2) afforded **3** (colourless oil, 50.0 mg, 99% yield). Spectroscopic data are in accordance with the literature.[S3] The same compound has been obtained in 98% yield by using 4CzIPN (20 mg, 0,025 mmol, 10 mol%) instead of [Acr-Mes]$^+$(BF$_4$)$^-$ as the photocatalyst (see Table 1, entry 2 in the main text).

Dimethyl 2-(3,3-dimethylbutan-2-yl)malonate (4). From **1a** (58 mg, 0.375 mmol, 1.5 equiv) dimethyl 2-ethylidenemalonate (**2b**, 28 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%), in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 80/20) gave **4** (yellow oil, 36.6 mg, 84% yield). Spectroscopic data are in accordance with the literature.[S4]

***N,N*,4,4-Tetramethylpentanamide (5).** From **1a** (58 mg, 0.075 mmol, 1.5 equiv), *N,N*-dimethylacrylamide (**2c,** 25 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: DCM/MeOH 100/0 to 95/5) gave **5** (colourless oil, 38.9 mg, 77% yield). Spectroscopic data are in accordance with the literature.[S5]

2-(2,2-Dimethyl-1-phenylpropyl)malononitrile (6). From **1a** (58 mg, 0.375 mmol, 1.5 equiv), 2-benzylidenmalononitrile (**2d**, 38.5 mg, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 90/10) afforded **6** (white powder, 40.2 mg, 76% yield). Spectroscopic data are in accordance with the literature.[S6]

3-Neopentylbicyclo[2.2.1]heptan-2-one (7): From **1a** (58 mg, 0.375 mmol, 1.5 equiv), 3-methylenebicyclo[2.2.1]heptan-2-one (**2e**, 28 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 9/1) afforded **7** (yellowish oil, 30.5 mg, 71% yield). Spectroscopic data are in accordance with the literature.[S7]

((3,3-Dimethylbutyl)sulfonyl)benzene (8). From **1a** (58 mg, 0.375 mmol, 1.5 equiv), phenyl vinyl sulfone (**2f,** 42 mg, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: DCM/MeOH 100/0 to 95/5) gave **8** (yellowish oil, 46.6 mg, 82% yield). Spectroscopic data are in accordance with the literature.[S8]

2-(3,3-Dimethylbutyl)pyridine (9). From **1a** (58 mg, 0.375 mmol, 1.5 equiv), 2-vinylpyridine (**2g**, 26 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 80/20) gave **9** (yellowish oil, 26.7 mg, 65% yield). Spectroscopic data are in accordance with the literature.[S9]

2-(3,3-Dimethylbutyl)pyrazine (10). From **1a** (58 mg, 0.375 mmol, 1.5 equiv), 2-vinylpyrazine (**2h**, 25 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 80/20) afforded **10** (yellowish oil, 28.5 mg, 69% yield). Spectroscopic data are in accordance with the literature.[S10]

2-(*tert*-Butyl)-3-methyl-2,3-dihydronaphthalene-1,4-dione (11). From **1a** (58 mg, 0.375 mmol, 1.5 equiv), 2-methylnaphthalene-1,4-dione (**2j**, 43 mg, 0.25 mmol, 1 equiv), 4CzIPN (20 mg, 0.025 mmol, 10 mol%) in DCE (5 mL) under N_2. Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 80/20) afforded **11** (pale orange powder, 38.4 mg, 67% yield).

11. ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.06–7.97 (m, 2H), 7.74-7.71 (m, 2H), 3.40–3.44 (m, 1H), 3.03–3.05 (d, *J* = 6 Hz, 1H), 1.39–1.41 (d, *J* = 6 Hz, 3H), 1.09 (s, 9H). ^{13}C NMR (75 MHz, Chloroform- *d*) δ 199.7, 198.7, 136.6, 135.6, 133.7, 133.5, 126.3, 62.1, 46.7, 33.6, 29.6, 15.4. The compound was not stable under HRMS (EI) analysis.

Dimethyl 2-(adamantan-1-yl)succinate (12). From **1b** (88 mg, 0.375 mmol, 1.5 equiv), **2a** (31 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 90/10) afforded **12** (white powder, 67.8 mg, 98% yield). Spectroscopic data in accordance with the literature.[S3]

3-(Adamantan-1-yl)-*N*,*N*-dimethylpropanamide (13). From **1b** (88 mg, 0.375 mmol, 1.5 equiv), **2c** (26 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 90/10) gave **13** (yellowish oil, 54.6 mg, 93% yield). Spectroscopic data in accordance with the literature.[S11]

2-((Adamantan-1-yl)(phenyl)methyl)malononitrile (14). From **1b** (88 mg, 0.375 mmol, 1.5 equiv), **2d** (38 mg, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg 0.025 mmol, 10 mol%) in a DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 95/5) afforded **14** (white powder, 39.2 mg, 54% yield). Spectroscopic data in accordance with the literature.[S12]

3-((Adamantan-1-yl)methyl)bicyclo[2.2.1]heptan-2-one (15). From **1b** (88 mg, 0.375 mmol, 1.5 equiv), **2e** (26 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 90/10) furnished **15** (yellowish powder, 37.4 mg, 58% yield). Spectroscopic data in accordance with the literature.[S2]

1-(2-(Phenylsulfonyl)ethyl)adamantane (16). From **1b** (88 mg, 0.375 mmol, 1.5 equiv), **2f** (42 mg, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: DCM/MeOH 100/0 to 95/5) affording **16** (yellowish oil, 60.7 mg, 80% yield). Spectroscopic data in accordance with the literature.[S11]

2-(2-Adamantan-1-yl)ethyl)pyridine (17). From **1b** (88 mg, 0.375 mmol, 1.5 equiv), **2g** (26 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification

by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 90/10) afforded **17** (colourless oil, 52.1 mg, 86% yield). Spectroscopic data in accordance with described in literature.[S9]

2-(2-Adamantan-1-yl)ethyl)pyrazine (18). From **1b** (88 mg, 0.375 mmol, 1.5 equiv), **2h** (25 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol %,) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 9/1) gave **18** (yellowish oil, 44.3 mg, 73% yield). Spectroscopic data in accordance with described in literature.[S2]

Allyl 3-(adamantan-1-yl)-2-methylpropanoate (19). From **1b** (88 mg, 0.375 mmol, 1.5 equiv), allyl vinyl methacrylate (**2j**, 34 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 80/20) gave **19** (white powder, 55.7 mg, 85% yield). Spectroscopic data in accordance with described in literature.[S2]

Dimethyl 2-(*iso*-propyl)succinate (20). From **1c** (53 mg, 0.375 mmol, 1.5 equiv), **2a** (31 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 80/20) afforded **20** (colourless oil, 43.3 mg, 96% yield). Spectroscopic data are in accordance with the literature.[S13]

2-(2,2-Dimethyl-1-phenylpropyl)malononitrile (21). From **1c** (53 mg, 0.375 mmol, 1.5 equiv), **2d** (38.5 mg, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 90/10) furnished **21** (white powder, 16.5 mg, 33% yield). Spectroscopic data are in accordance with the literature.[S13]

3-*iso*-Butylbicyclo[2.2.1]heptan-2-one (22). From **1c** (53 mg, 0.375 mmol, 1.5 equiv), **2e** (28 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 9/1) afforded **22** (yellowish oil, 35.8 mg, 86% yield). Spectroscopic data are in accordance with the literature.[S14]

22. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.62 (d, *J* = 4.9 Hz, 2H), 2.27–2.12 (m, 1H), 2.12–2.03 (m, 1H), 1.83 (d, *J* = 8.8 Hz, 1H), 1.72–1.28 (m, 8H), 1.34–1.11 (m, 1H), 0.92 (dd, *J* = 14.2, 6.6 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 221.9, 78.6, 78.3, 78.0, 53.1, 51.8, 50.4, 43.7, 39.7, 38.4, 38.1, 36.5, 29.3, 27.6, 26.6, 24.7, 22.7, 22.5.

(*Iso*-Pentylsulfonyl)benzene (23). From **1c** (53 mg, 0.375 mmol, 1.5 equiv), **2f** (42 mg, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%)in DCE (5 mL). Purification by column chromatography (eluent: DCM/MeOH 100/0 to 95/5) afforded **23** (yellowish oil, 32.1 mg, 60% yield). Spectroscopic data are in accordance with the literature.[S15]

Dimethyl 2-cyclohexylsuccinate (24). From **1d** (68 mg, 0.375 mmol, 1.5 equiv), **2a** (31 µL, 0.25 mmol, 1 equiv), 4CzIPN (20 mg, 0,025 mmol, 10 mol%) in DCE (5 mL) under N_2. Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 80/20) gave **24** (colourless oil, 28.7 mg, 50% yield). Spectroscopic data are in accordance with the literature.[S16]

2-(2-Cyclohexyl-1-phenylpropyl)malononitrile (25). From **1d** (68 mg, 0.375 mmol, 1.5 equiv), **2d** (38.5 mg, 0.25 mmol, 1 equiv), 4CzIPN (20 mg, 0,025 mmol, 10 mol%) in DCE (5 mL) under N_2. Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 90/10) afforded **25** (white powder, 13.4 mg, 22% yield). Spectroscopic data are in accordance with the literature.[S17]

3-(Cyclohexylmethyl)bicyclo[2.2.1]heptan-2-one (26): From **1d** (58 µL, 0.375 mmol, 1.5 equiv), **2e** (28 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 90/10) gave **26** (yellowish oil, 33.6 mg, 65% yield). Spectroscopic data are in accordance with the literature.[S16]

((2-Cyclohexylethyl)sulfonyl)benzene (27). From **1d** (68 mg, 0.375 mmol, 1.5 equiv), **2f** (42 mg, 0.25 mmol, 1 equiv), 4CzIPN (20 mg, 0,025 mmol, 10 mol%) in DCE (5 mL) under N_2. Purification by column chromatography (eluent: DCM/MeOH 100/0 to 95/5) furnished **27** (yellowish oil, 46.5 mg, 74% yield). Spectroscopic data are in accordance with the literature.[S18]

2-(2-Cyclohexylethyl)pyridine (28). From **1d** (88 mg, 0.375 mmol, 1.5 equiv), **2g** (26 µL, 0.25 mmol, 1 equiv), 4CzIPN (20 mg, 0,025 mmol, 10 mol %) in DCE (5 mL) under N_2. Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 90/10) afforded **29** (colourless oil, 30.6 mg, 64% yield). Spectroscopic data in accordance with described in literature.[S19]

3-(Cyclohex-3-en-1-ylmethyl)bicyclo[2.2.1]heptan-2-one (29): From **1e** (90 mg, 0.375 mmol, 2 equiv), **2e** (28 µL, 0.25 mmol, 1 equiv), 4CzIPN (20 mg, 0,025 mmol, 10 mol%) in DCE (5 mL) under N_2. Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 90/10) gave **29** as a mixture of diastereoisomers (yellowish oil, 21.3 mg, 42% yield, dr: 1:1).

29. ^{1}H NMR (300 MHz, Chloroform- *d*, mixture of diastereoisomers) δ 5.66 (s, 2H), 2.66–2.56 (m, 2H), 2.28–1.99 (m, 5H), 1.89–1.76 (m, 2H), 1.72–1.58 (m, 7H), 1.40 (dt, *J* = 13.1, 6.2 Hz, 2H).^{13}C NMR (75 MHz, Chloroform-*d*, mixture of diastereoisomers) δ 201.4, 127.2, 127.2, 126.5, 126.3, 77.6, 76.7, 51.5, 51.4, 50.7, 38.8, 38.6, 37.3, 33.3, 32.8, 32.8, 32.0, 31.9, 31.1, 29.9, 27.9, 25.5, 25.4, 25.2, 21.5, 21.4. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{16}H_{27}NO_6$ 330.1911, found 330.1909.

Dimethyl 2-(1-phenylethyl)succinate (30). From **1g** (76 mg, 0.375 mmol, 1.5 equiv) **2a** (31 µL, 0.25 mmol, 1 equiv), 4CzIPN (20 mg, 0,025 mmol, 10 mol %) in DCE (5 mL) under N_2 Purification by

column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 8/2) afforded **30** as a mixture of diastereoisomers (yellow oil, 50.0 mg, 80% yield, dr: 1:1). Spectroscopic data in accordance with described in literature.[S20]

Dimethyl 2-(2,2-dimethyl-1,3-dioxolan-4-yl)succinate (31). From **1h** (75 mg 0.375 mmol, 1.5 equiv), **2a** (31 µL, 0.25 mmol, 1 equiv), [Acr-Mes]$^+$(BF$_4$)$^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 70/30) afforded **31** as a mixture of diastereoisomers (orange oil, 45.9 mg, 75% yield, dr: 1:1). Spectroscopic data in accordance with described in literature.[S21]

2-((2,2-Dimethyl-1,3-dioxolan-4-yl)(phenyl)methyl)malononitrile (32). From **1h** (75 mg 0.375 mmol, 1.5 equiv), **2d** (38 mg, 0.25 mmol, 1 equiv), [Acr-Mes]$^+$(BF$_4$)$^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 95/5) gave **32** as a mixture of diastereoisomers (pale yellowish powder, 50.9 mg, 81% yield, dr: 1:1). Spectroscopic data in accordance with described in literature.[S21]

3-((2,2-Dimethyl-1,3-dioxolan-4-yl)methyl)bicyclo[2.2.1]heptan-2-one (33). From **1h** (75 mg 0.375 mmol, 1.5 equiv), **2e** (29 µL, 0.25 mmol, 1 equiv), [Acr-Mes]$^+$(BF$_4$)$^-$ (10 mg, 0.025 mmol, 10 mol %,), in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 1/1) afforded **33** as a mixture of diastereoisomers (colourless oil, 47.6 mg, 86% yield, dr: 1:1). Spectroscopic data in accordance with described in literature.[S21]

2,2-Dimethyl-4-(2-(phenylsulfonyl)ethyl)-1,3-dioxolane (34). From **1h** (75 mg 0.375 mmol, 1.5 equiv), **2f** (42 mg, 0.25 mmol, 1 equiv), [Acr-Mes]$^+$(BF$_4$)$^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 40/60) furnished **34** (yellowish oil, 58.7 mg, 87% yield). Spectroscopic data are in accordance with the literature.[S22]

Dimethyl 2-(((*tert*-butoxycarbonyl)(methyl)amino)methyl)succinate (35). From **1i** (91 mg, 0.375 mmol, 1.5 equiv), **2a** (31 µL, 0.25 mmol, 1 equiv), [Acr-Mes]$^+$(BF$_4$)$^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 9/1) afforded **35** (yellow oil, 55.2 mg, 77% yield). Spectroscopic data in accordance with the literature.[S23]

***tert*-Butyl (3,3-dicyano-2-phenylpropyl)(methyl)carbamate (36).** From **1i** (91 mg, 0.375 mmol, 1.5 equiv), **2d** (38 mg 0.25 mmol, 1 equiv), [Acr-Mes]$^+$(BF$_4$)$^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 70/30) gave **36** (yellow oil, 43.4 mg, 58% yield). Spectroscopic data in accordance with the literature.[S23]

***tert*-Butyl methyl(2-(3-oxobicyclo[2.2.1]heptan-2-yl)ethyl)carbamate (37).** From **1i** (91 mg, 0.375 mmol, 1.5 equiv), **2e** (28 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 1/1) afforded **37** (yellow oil, 64,9 mg, 86% yield). Spectroscopic data in accordance with the literature.[S2]

***tert*-Butyl methyl(3-(pyrazin-2-yl)propyl)carbamate (38).** From **1i** (91 mg, 0.375 mmol, 1.5 equiv), **2h** (25 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 20/80) afforded **38** (yellow oil, 33.2 mg, 54% yield). Spectroscopic data in accordance with the literature. [S2]

Dimethyl 2-(1-(tert-butoxycarbonyl)piperidin-2-yl)succinate (39). From **1j** (106 mg, 0.5 mmol, 1.5 equiv), **2a** (31 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 80/20) gave **39** as a mixture of diastereoisomers (yellow oil, 54.0 mg, 63% yield, dr: 1:1).

39. ^{1}H NMR (300 MHz, Chloroform-*d* mixture of diastereoisomers) δ 4.36 (s, 1H), 3.99 (s, 1H), 3.71 (s, 3H), 3.65 (s, 3H), 3.36 (td, *J* = 10.8, 4.4 Hz, 1H), 2.69 (td, *J* = 18.0, 11.6 Hz, 2H), 2.42 (dd, *J* = 16.6, 4.4 Hz, 1H), 1.66–1.60 (m, 2H), 1.60–1.50 (m, 4H), 1.47 (s, 9H). ^{13}C NMR (75 MHz, Chloroform-*d* mixture of diastereoisomers) δ 174.3, 172.5, 154.9, 80.2, 52.2, 52.0, 41.7, 38.6, 34.3, 29.8, 28.6, 27.1, 25.3, 19.2. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{16}H_{27}NO_6$ 330.1911, found 330.1909.

Dimethyl 2-(1-(1-(*tert*-butoxycarbonyl)piperidin-2-yl)ethyl)malonate (40). From **1j** (106 mg, 0.5 mmol, 1.5 equiv), **2b** (28 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%), in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 1/1) afforded **40** as a mixture of diastereoisomers (pale yellow oil, 68.6 mg, 80% yield, dr: 1:1).

40. ^{1}H NMR (400 MHz, Chloroform-*d*, mixture of diastereoisomers) δ 4.16 (s, 1H), 4.05 (s, 1H), 3.77–3.74 (m, 6H), 3.74–3.71 (m, 6H), 3.66–3.61 (m, 1H), 3.56 (t, *J* = 7.4 Hz, 2H), 3.48 (d, *J* = 4.2 Hz, 1H), 3.25 (d, *J* = 8.1 Hz, 1H), 2.72 (tdd, *J* = 11.1, 9.0, 5.5 Hz, 2H), 2.34 (t, *J* = 7.5 Hz, 1H), 2.31–2.18 (m, 2H), 1.83 (p, *J* = 6.1 Hz, 1H), 1.72–1.55 (m, 12H), 1.48–1.41 (m, 18H), 1.00–0.94 (m, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*, mixture of diastereoisomers) δ 169.3, 169.2, 168.6, 163.0, 155.1, 83.9, 77.3, 77.0, 76.7, 57.5, 52.5, 52.3, 52.2, 40.4, 33.9, 33.4, 31.9, 31.8, 29.4, 29.4, 29.3, 29.1, 28.4, 28.3, 28.1, 26.1, 25.3, 24.8, 24.1, 19.0, 16.9, 14.1, 12.8. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{17}H_{29}NO_6$ 344.2068, found 344.2075.

***tert*-Butyl 2-(2,2-dicyano-1-phenylethyl)piperidine-1-carboxylate (41).** From **1j** (106 mg, 0.5 mmol, 1.5 equiv), **2d** (38 mg 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%)

in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 1/1) gave **41** as a mixture of diastereoisomers (yellow powder, 55.6 mg, 61% yield, dr: 1:1). Spectroscopic data in accordance with the literature.[S24]

***tert*-Butyl 2-(((*1S,4R*)-3-oxobicyclo[2.2.1]heptan-2-yl)methyl)piperidine-1-carboxylate (42).** From **1j** (106 mg, 0.5 mmol, 1.5 equiv), **2e** (28 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol%) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 1/1) afforded **42** as a mixture of diastereoisomers (pale yellow oil, 38.7 mg, 50% yield, dr: 1:1)

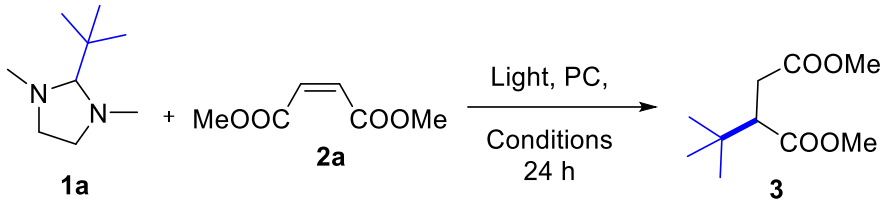
42. ^{1}H NMR (300 MHz, Chloroform-*d* mixture of diastereoisomers) δ 4.25 (s, 1H), 3.95 (s, 1H), 2.86–2.73 (m, 2H), 2.61 (d, *J* = 5.0 Hz, 1H), 2.33 (ddd, *J* = 15.1, 12.1, 3.3 Hz, 1H), 1.90–1.75 (m, 2H), 1.70–1.63 (m, 4H), 1.63–1.59 (m, 3H), 1.56–1.51 (m, 2H), 1.48–1.43 (m, 12H), 1.40–1.30 (m, 2H), 1.07 (ddd, *J* = 15.0, 11.5, 3.8 Hz, 1H).^{13}C NMR (75 MHz, Chloroform-*d* mixture of diastereoisomers) δ 155.2, 155.0, 79.4, 79.4, 77.6, 76.7, 51.9, 51.2, 50.7, 50.4, 38.6, 38.3, 37.4, 37.2, 29.8, 28.7, 28.6, 28.5, 27.7, 26.2, 25.8, 25.7, 25.7, 25.6, 21.5, 21.4, 19.2, 19.0. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{18}H_{29}NO_3$ 308.2220, found 308.2221.

***tert*-Butyl 2-(2-(pyridin-2-yl)ethyl)piperidine-1-carboxylate (43).** From **1j** (106 mg, 0.5 mmol, 1.5 equiv), **2g** (25 µL, 0.25 mmol, 1 equiv), $[Acr\text{-}Mes]^+(BF_4)^-$ (10 mg, 0.025 mmol, 10 mol %) in DCE (5 mL). Purification by column chromatography (eluent: Cyclohexane/AcOEt 100/0 to 80/20) furnished **43** as a mixture of diastereoisomers (pale yellow oil, 45.8 mg, 62% yield, dr: 1:1).

43. ^{1}H NMR (400 MHz, Chloroform-*d* mixture of diastereoisomers) δ 8.52 (d, *J* = 3.0 Hz, 1H), 7.58 (td, *J* = 7.7, 1.9 Hz, 1H), 7.16 (d, *J* = 7.8 Hz, 1H), 7.10 (ddd, *J* = 7.5, 5.0, 1.2 Hz, 1H), 4.32 (s, 1H), 3.98 (s, 1H), 2.88–2.63 (m, 3H), 2.16 (dtd, *J* = 13.8, 10.2, 5.8 Hz, 1H), 1.85 (ddt, *J* = 13.7, 10.5, 5.9 Hz, 1H), 1.66–1.55 (m, 6H), 1.44 (s, 9H).^{13}C NMR (100 MHz, Chloroform-*d* mixture of diastereoisomers) δ 162.0, 155.3, 149.3, 136.6, 123.1, 121.2, 79.3, 77.5, 77.2, 76.8, 35.4, 30.1, 28.9, 28.6, 28.2, 25.8, 19.2. HRMS (EI) m/z: $[M+H]^+$ calculated for $C_{17}H_{26}N_2O_2$ 291.2067, found 291.2065.

3. Optimization of the reaction conditions

Table S1. Optimization of the photoredox catalysed synthesis of **3** in batch



Entry	Conditions	3 (% Yield)
1	**2a** (0.05 M), **1a** (1.5 equiv), $Ph_3Pyrylium^+$ BF_4^- (10 mol%), air, DCE, 405 nm	-
2	**2a** (0.05 M), **1a** (1.5 equiv), $Ir(ppy)_3$ (5 mol%), N_2, DCE, 405 nm	-
3	**2a** (0.05 M), **1a** (1.5 equiv), 4CzIPN (10 mol%), N_2, DCE, 405 nm,	98
4	**2a (0.05 M), 1a (1.5 equiv), Acr-Mes$^+$ BF_4^- (10 mol %), air, DCE, 405 nm**	**99**
5	**2a** (0.05 M), **1a** (1.5 equiv), Acr-Mes$^+$ BF_4^- (10 mol%), N_2, DCE, 405 nm	90
6	**2a** (0.05 M), **1a** (1.5 equiv), 3CzClIPN (10 mol%), N_2, CPME, 405 nm,	51
7	**2a** (0.05 M), **1a** (1.5 equiv), 3CzClIPN (10 mol%), N_2, DMC, 405 nm,	22
8	**2a** (0.05 M), **1a** (1.5 equiv), Acr-Mes$^+$ BF_4^- (10 mol%), air, MeOH, 405 nm	-
9	**2a** (0.05 M), **1a** (1.5 equiv), Acr-Mes$^+$ BF_4^- (10 mol%), air, MeCN, 405 nm	-
10	**2a** (0.05 M), **1a** (1.5 equiv), Acr-Mes$^+$ BF_4^- (10 mol%), air, DCM, 405 nm	84
11	**2a** (0.05 M), **1a** (1.25 equiv), Acr-Mes$^+$ BF_4^- (10 mol%), air, DCE, 405 nm	53
12	**2a** (0.05 M), **1a** (1.5 equiv), Acr-Mes$^+$ BF_4^- (5 mol%), air, DCE, 405 nm	71
13	**2a** (0.05 M), **1a** (1.5 equiv), Acr-Mes$^+$ BF_4^- (10 mol%), air, DCE, **no light**	-
14	**2a** (0.05 M), **1a** (1.5 equiv), Acr-Mes$^+$ BF_4^- (10 mol%), air, DCE, **natural sunlight**, 2 days, 6 hours/day	32
15	**2a** (0.05 M), **1a** (1.5 equiv), **TEMPO (1.0 equiv.)**, Acr-Mes$^+$ BF_4^- (10 mol%), air, DCE, 405 nm	50

CPME = Cyclopentyl methyl ether

General procedure for the synthesis of compound 3 under flow conditions: A solution of the imidazolidine **1a** (1.5 equiv,), olefin **2a** (1 equiv) and [Acr-Mes]$^{+}$(BF_4)$^{-}$ (10 mol %) in DCM (2.5 mL) was prepared and charged into a coiled tubing reservoir (PTFE, 1 mm internal diameter, see Figures S4, S5). The reaction mixture was then flown through the coiled reactor (PTFE, 1 mm internal diameter) by a syringe pump set at the corresponding flow rate upon irradiation with a 405 nm (EvoluChem, 18W) or 390 nm (Kessil PR-160L, 40W) LED lamp. Fan cooling was applied to maintain room temperature. The resulting solution was collected and concentrated in vacuo. The residue was purified by flash column chromatography using an Isolera apparatus (Biotage) and a SiO_2 cartridge (eluant: cyclohexane/ethyl acetate mixture).

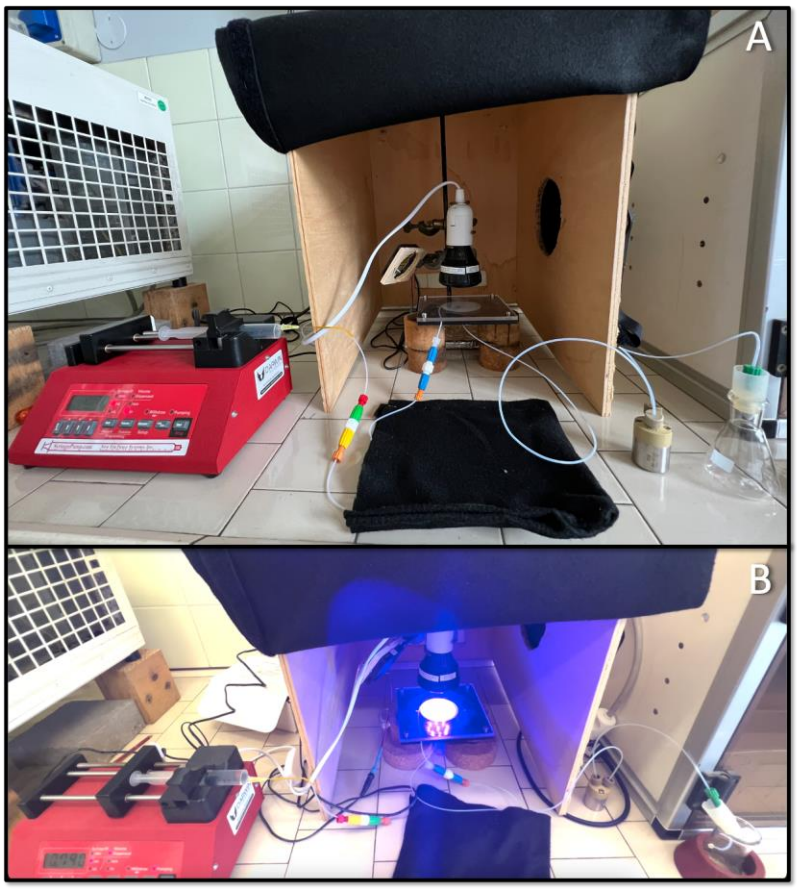


Figure S4. A) Flow system set up before irradiation. B) Flow system set up during irradiation**.**

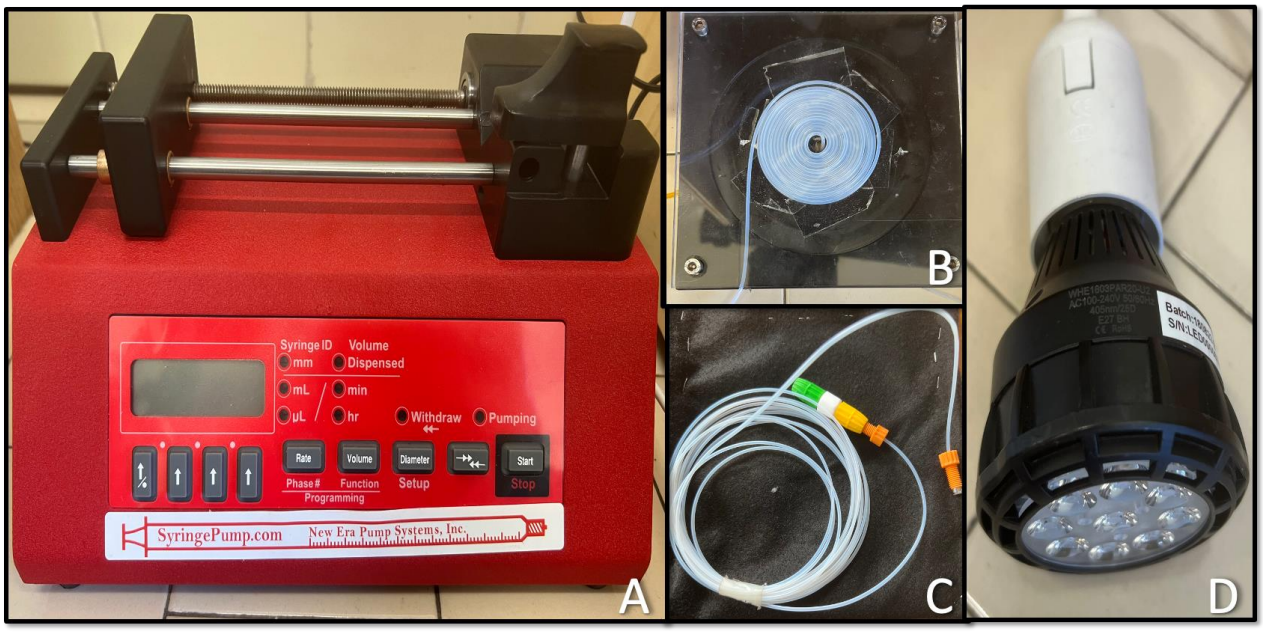
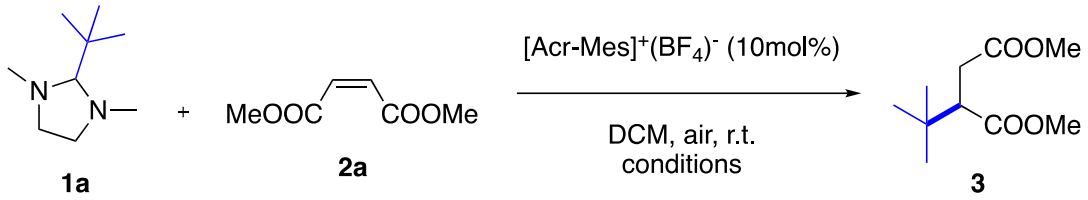


Figure S5. Description of hand-made flow system. A) Syringe pump. B) Coiled reactor. C) Coiled solution reservoir. D) 405 nm Evoluchem lamp (18W).

Table S2. Optimization of the photoredox catalyzed synthesis of **3** under flow conditions.



Entry	Conditions	3 (% Yield)
1	**2a** (0.1 M), **1a** (1.5 equiv), 390 nm (50% Power), flow rate: 1.2 mL/min, 1.5 h	65
2	**2a** (0.1 M), **1a** (1.5 equiv), 405 nm (100% Power), flow rate: 1.6 mL/min, 1 h	43
3	**2a (0.1 M), 1a (1.5 equiv), 405 nm (100% Power), flow rate: 0.8 mL/min, 2 h**	**99**
4	**2a** (0.1 M), **1a** (1.5 equiv), 390 nm (100% Power), flow rate: 0.8 mL/min, 2 h	96
5	**2a** (0.1 M), **1a** (1.5 equiv), 405 nm (100% Power), flow rate: 1.6 mL/min, 1 h	44
6	**2a** (0.16 M), **1a** (1.5 equiv), 405 nm (100% Power), flow rate: 0.8 mL/min, 2 h	93
7	**2a** (0.4 M), **1a** (1.5 equiv), 405 nm (100% Power), flow rate: 0.8 mL/min, 2 h	88
8	**2a (0.4 M), 1a (1.5 equiv), 405 nm (100% Power), flow rate: 0.8 mL/min, 2 h**	**92**

Synthesis of 3 under flow conditions. A solution of the imidazolidine **1a** (236 mg, 1.5 mmol, 1.5 equiv), dimethyl maleate (**2a**, 0.13 mL, 1 mmol, 1 equiv) and $[Acr\text{-}Mes]^+(BF_4)^-$ (40 mg, 0.1 mmol, 10 mol%) in DCM (2.5 mL) was prepared and charged into a coiled tubing reservoir in dark. The reaction mixture was then flown through the coiled reactor by a syringe pump setting the flow rate at 0.8 mL/h upon irradiation with the corresponding 405 nm (EvoluChem, 18W) LED lamp. Fan cooling was applied to maintain room temperature. The resulting solution was collected and concentrated in vacuo. The residue was purified by flash column chromatography using an Isolera apparatus (Biotage) and a SiO_2 cartridge (eluant: Cyclohexane/AcOEt 100/0 to 8/2) affording **3** (colourless oil, 187.3 mg, 92% yield). Spectroscopic data are in accordance with the literature.[S3]

4. Mechanistic studies

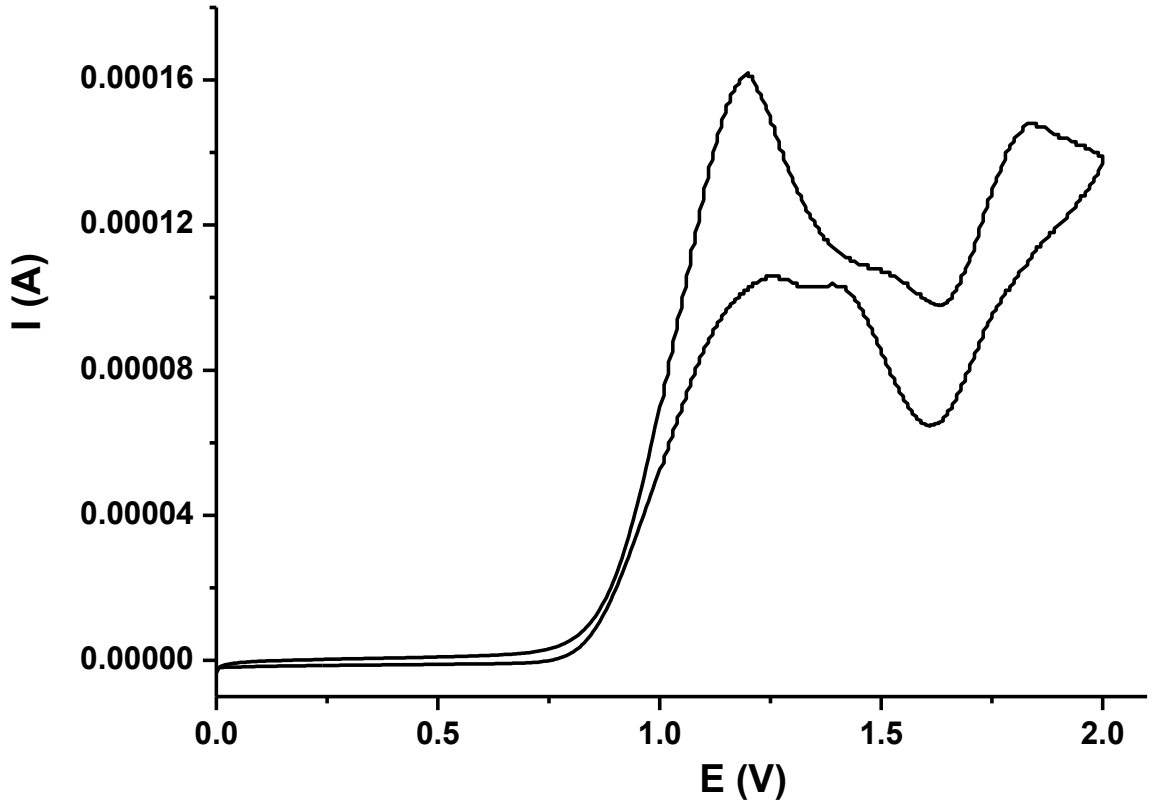
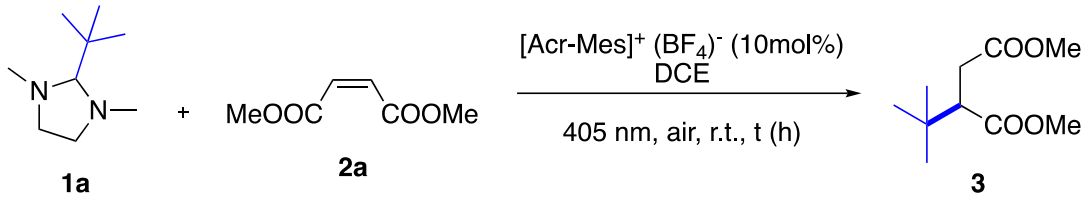


Figure S6. Cyclic voltammetry of **1c.** E_{ox} = 1.21 V vs Ag/AgCl corresponding to E_{ox} = 1.16 V vs SCE.

Table S3. On-Off experiment for the photoredox catalyzed synthesis of **3**.



Entry	Time (h)	Irradiation	2a (% Yield)
1	0.0	OFF	0
2	0.5	ON	38
3	1.0	OFF	49
4	1.5	ON	68
5	2.0	OFF	68
6	2.5	ON	73
7	3.0	OFF	73
8	4.0	ON	99
9	5.0	OFF	99

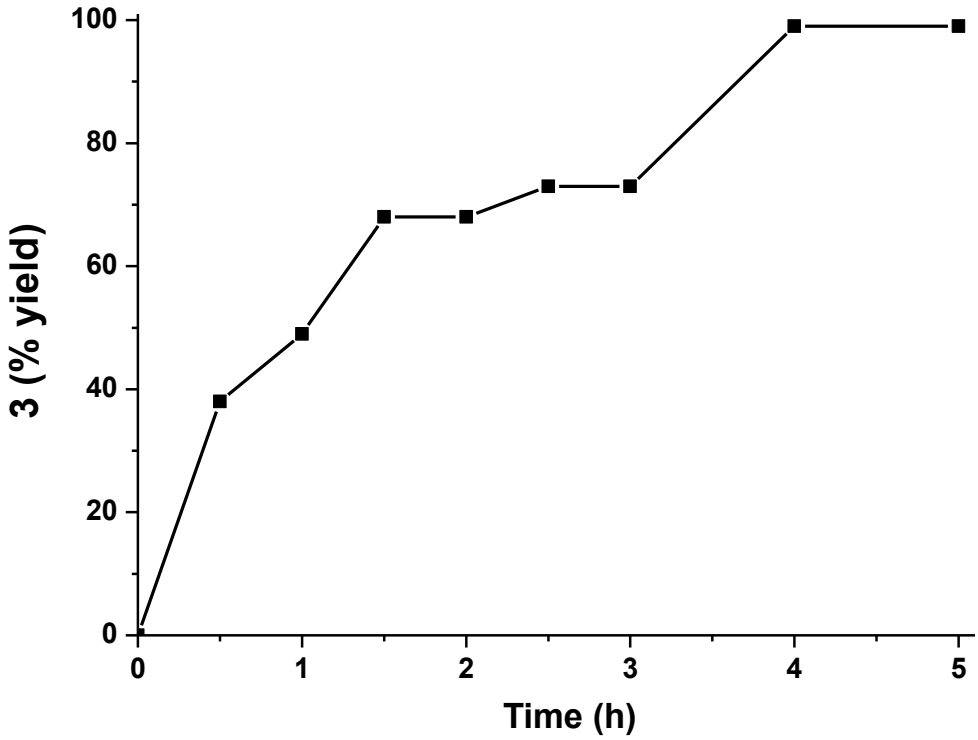
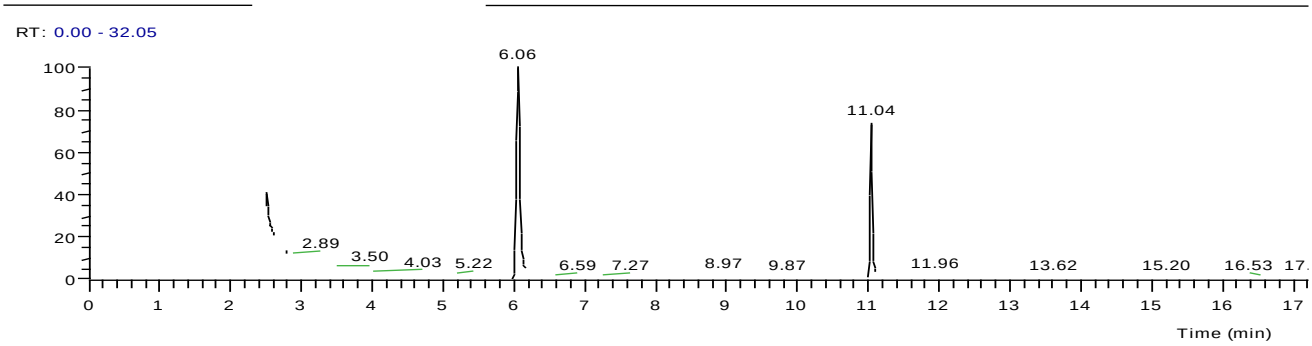
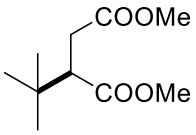


Figure S7. On-Off experiment for the photoredox catalysed synthesis of **3**.

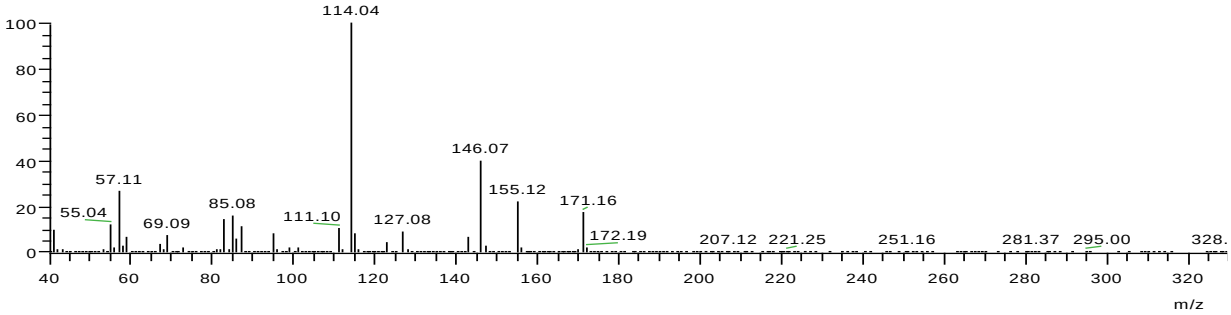
Experiments in deuterated media.





3

retention time = 11.04 min



The GC-MS fragmentation pattern observed for compound **3** is in accordance with the deuterium-free derivative as previously described,[S2] so no deuterium incorporation in compound **3** occurred during the irradiation.

5. References

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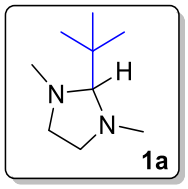
6. Copy of ^{1}H and ^{13}C NMR spectra.



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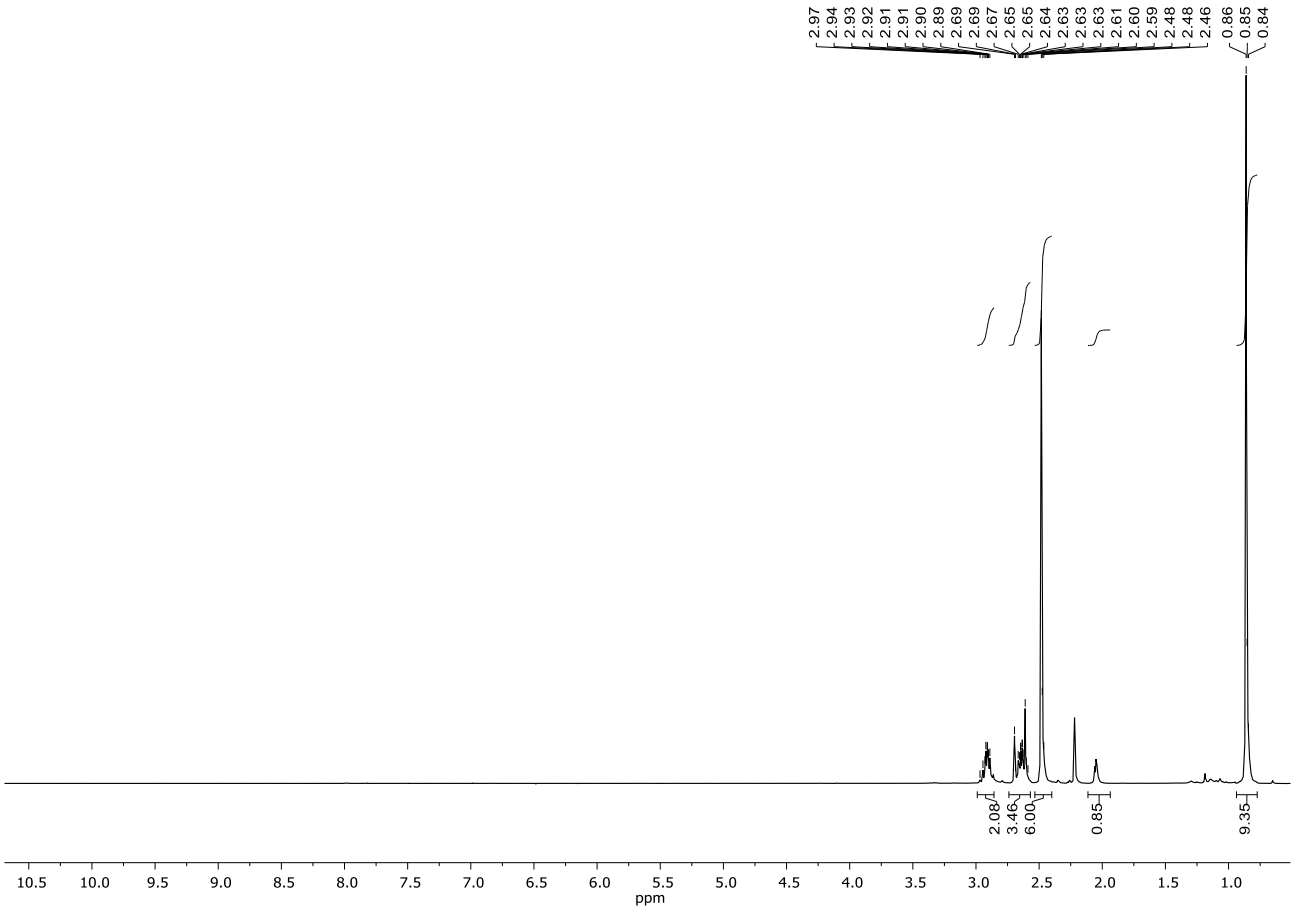

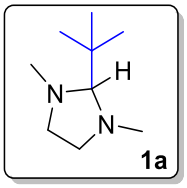

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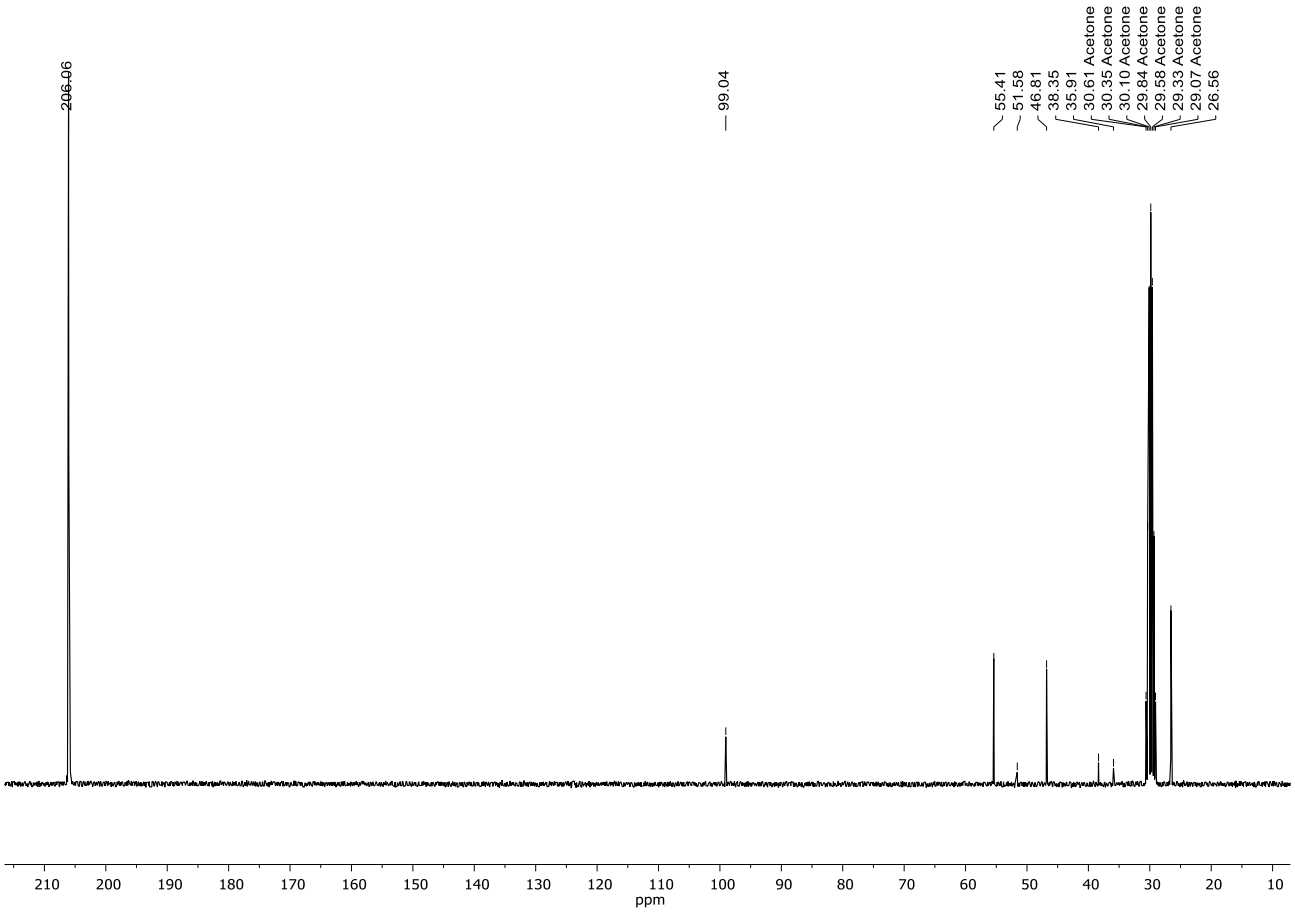

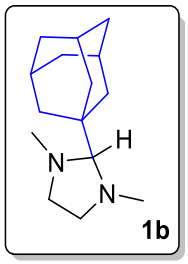

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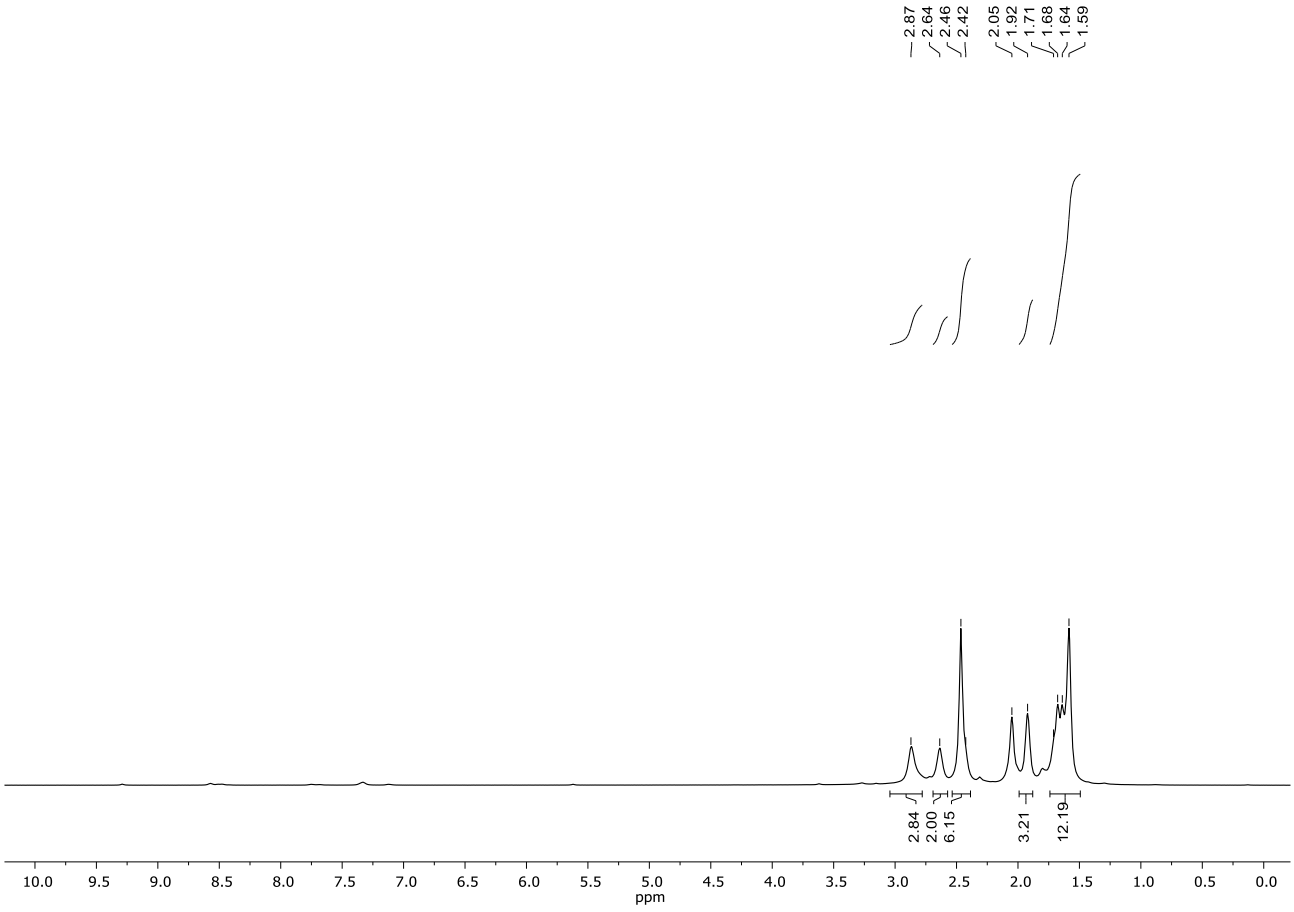

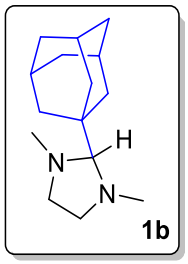

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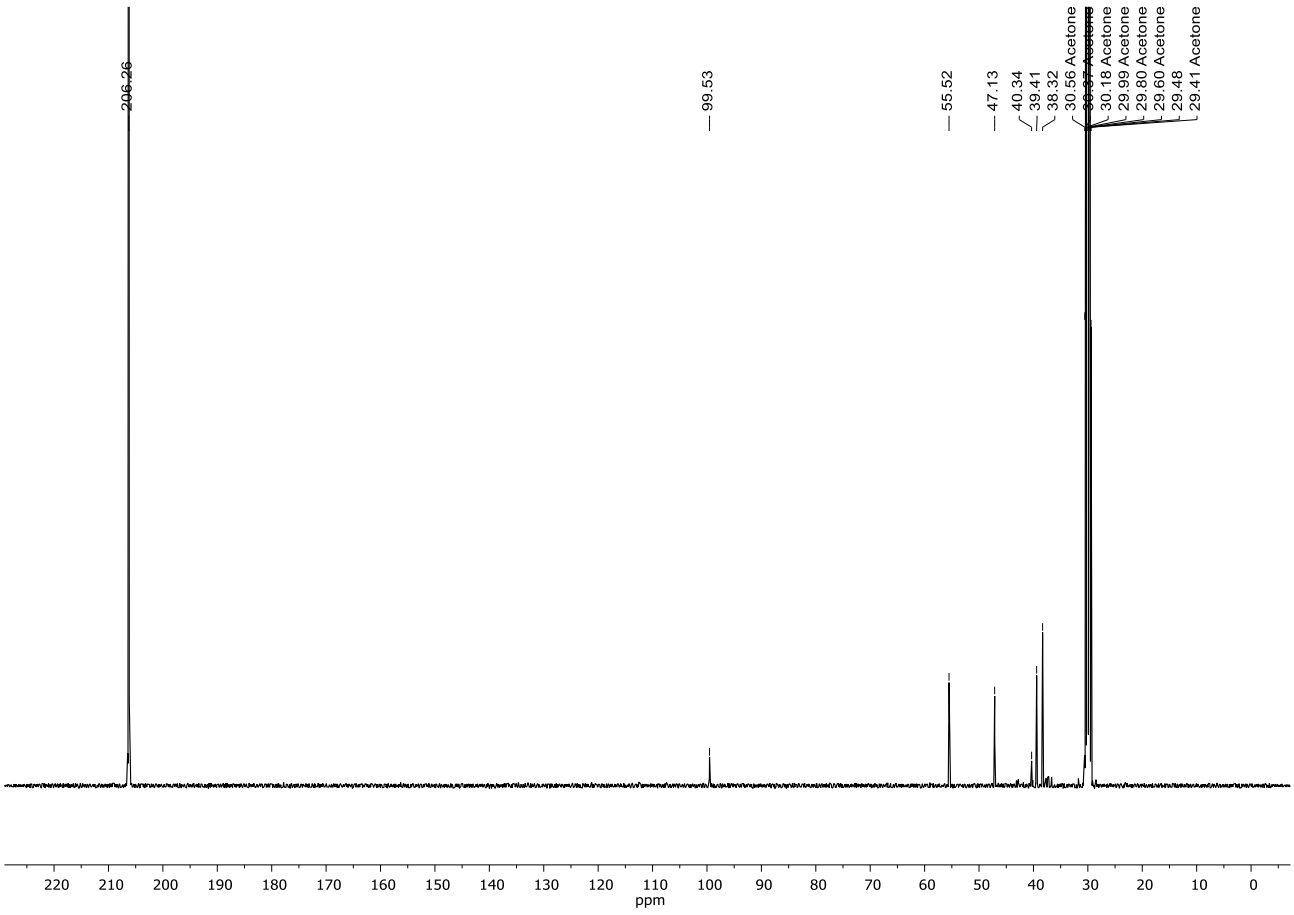

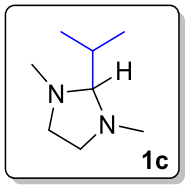

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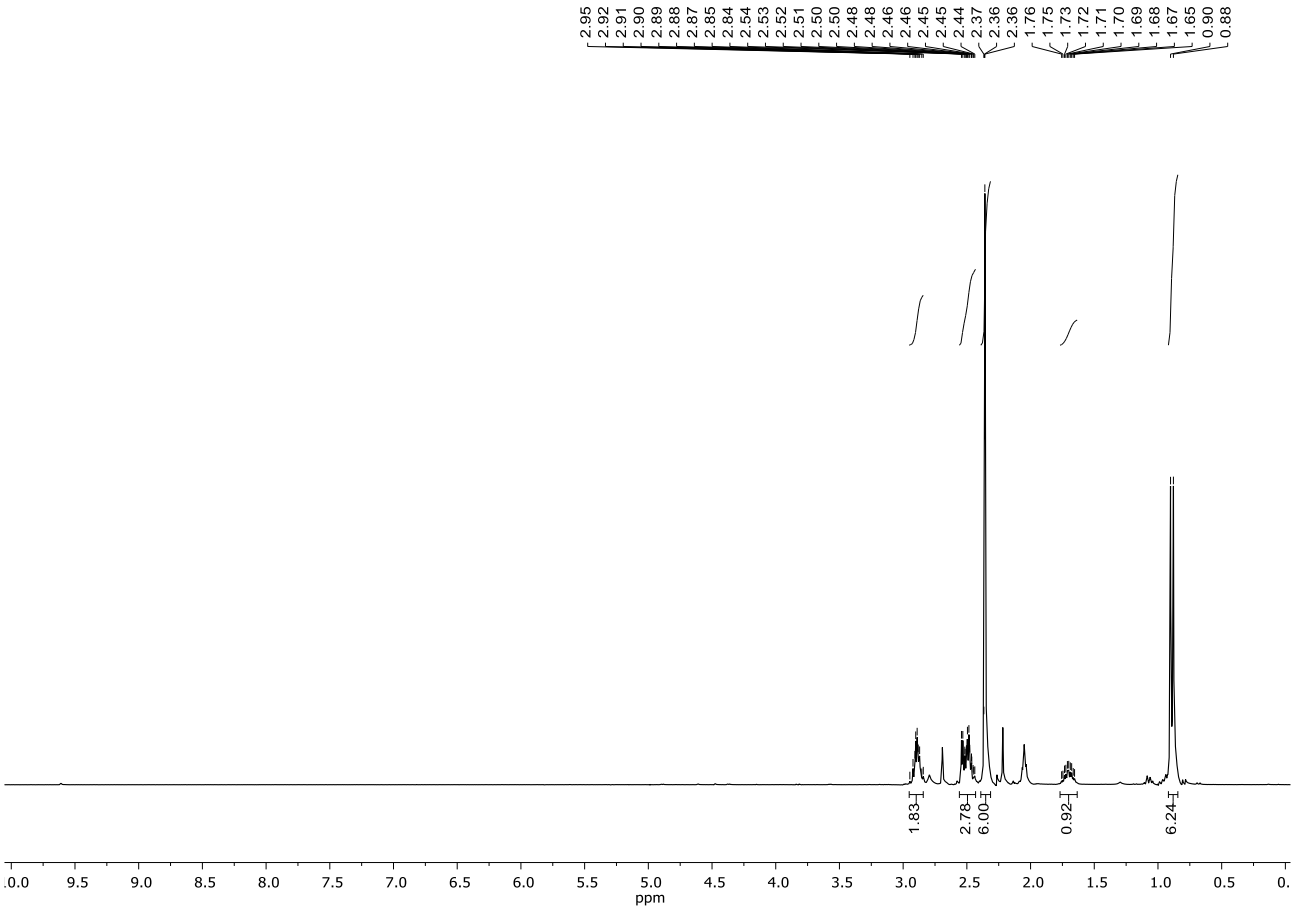

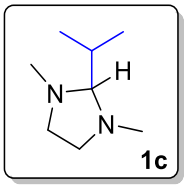

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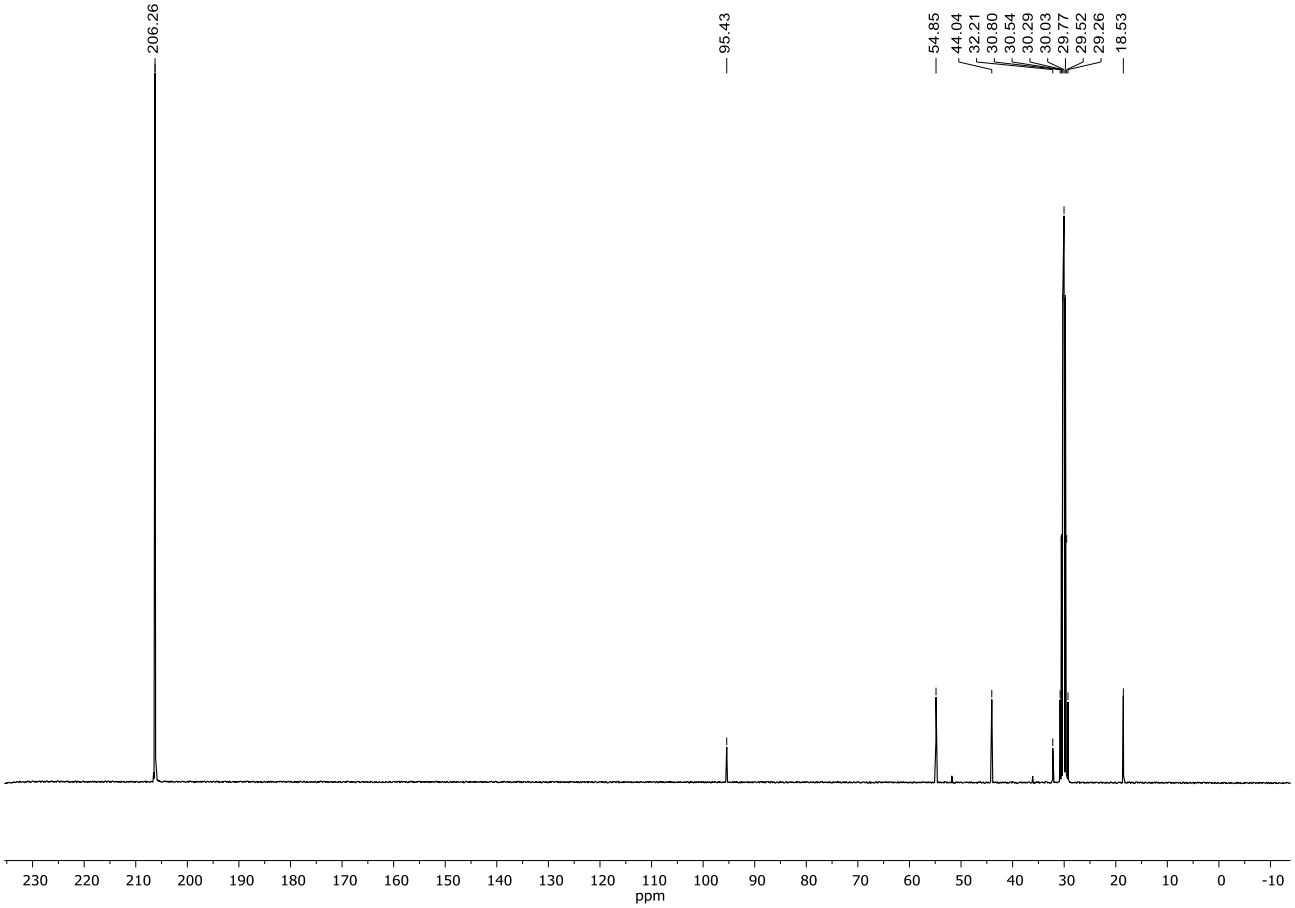

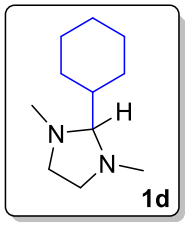

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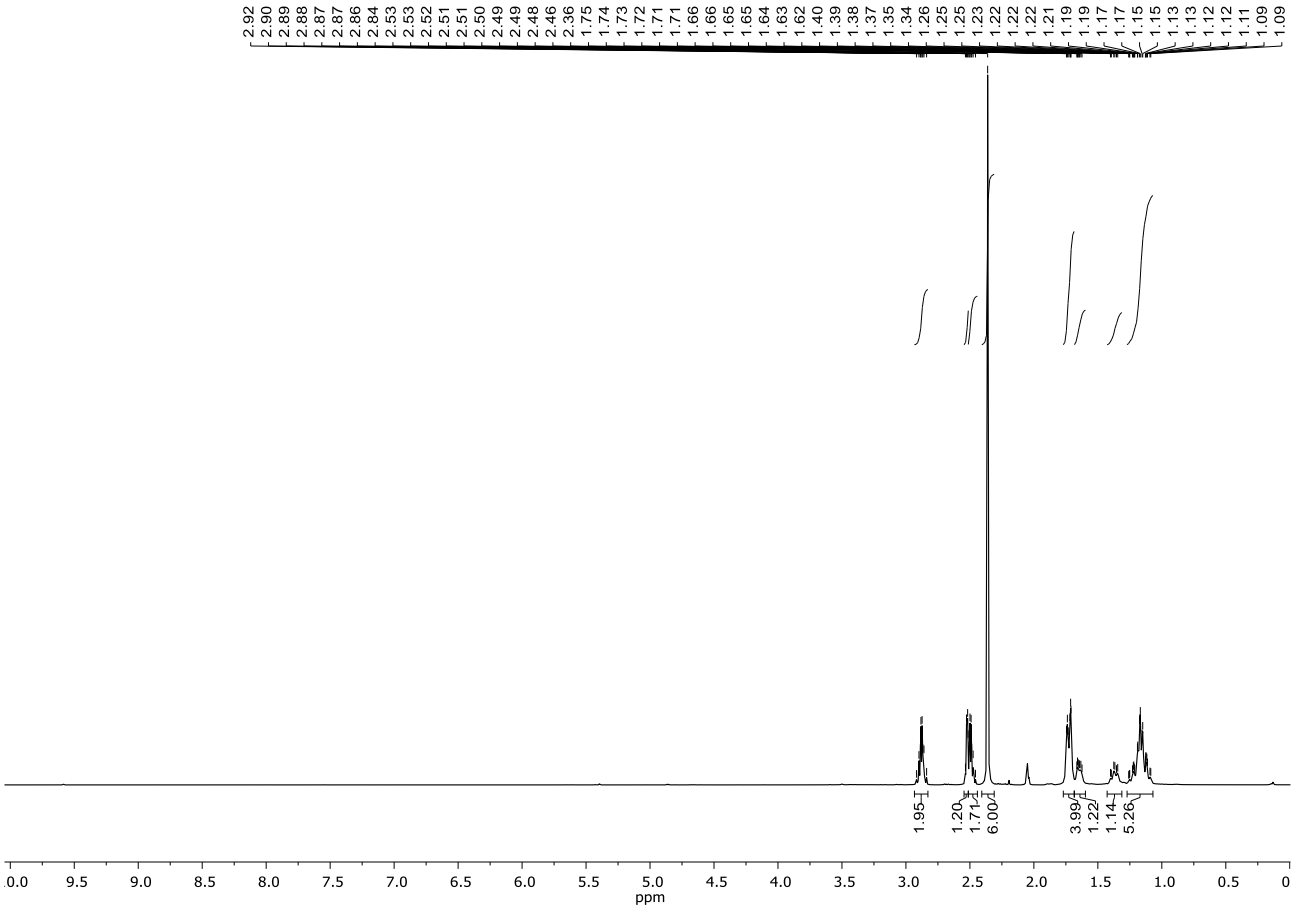

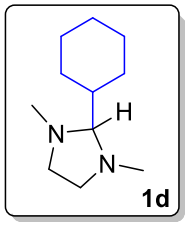

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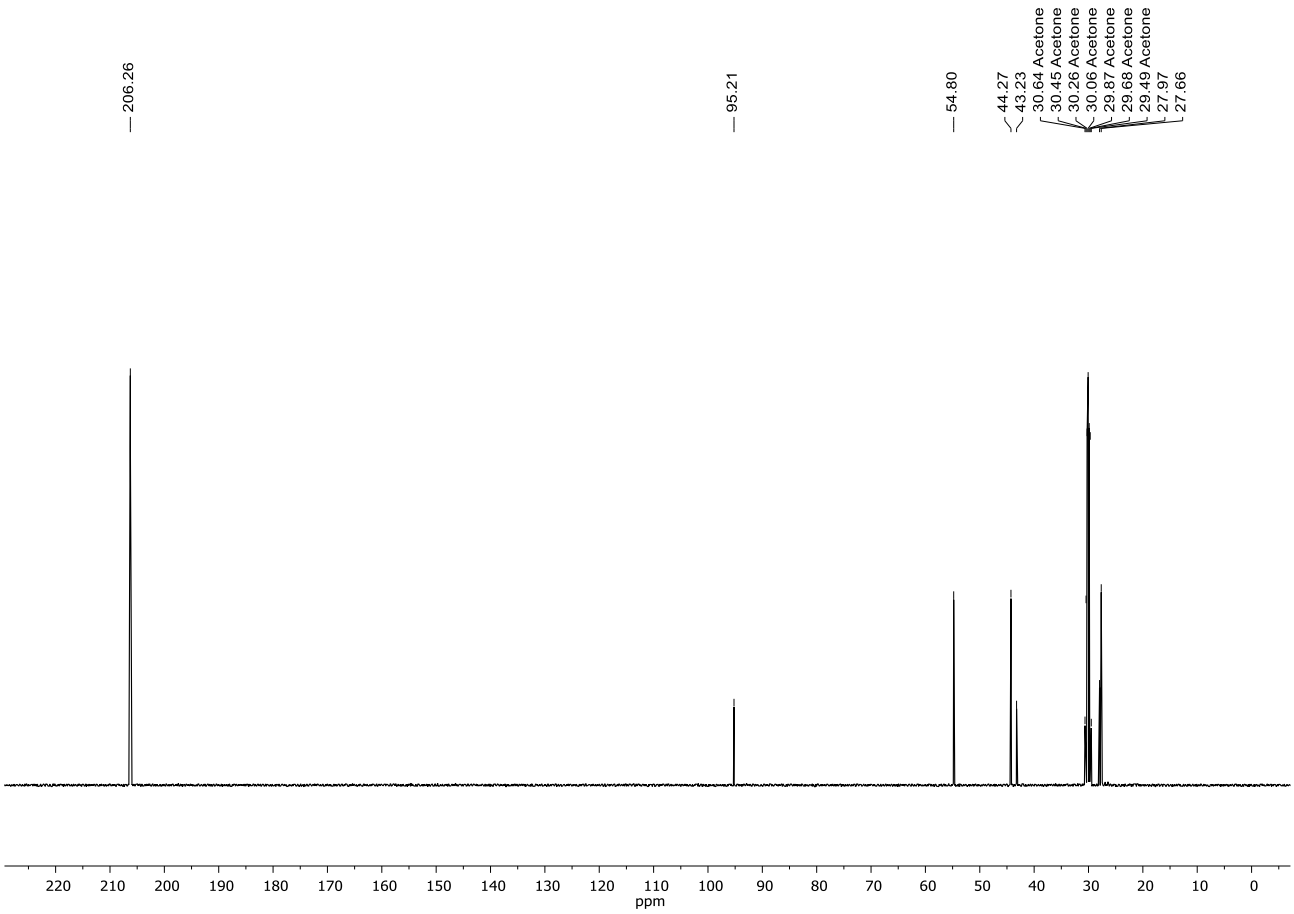

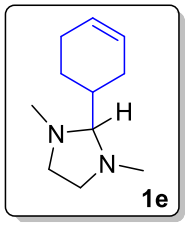

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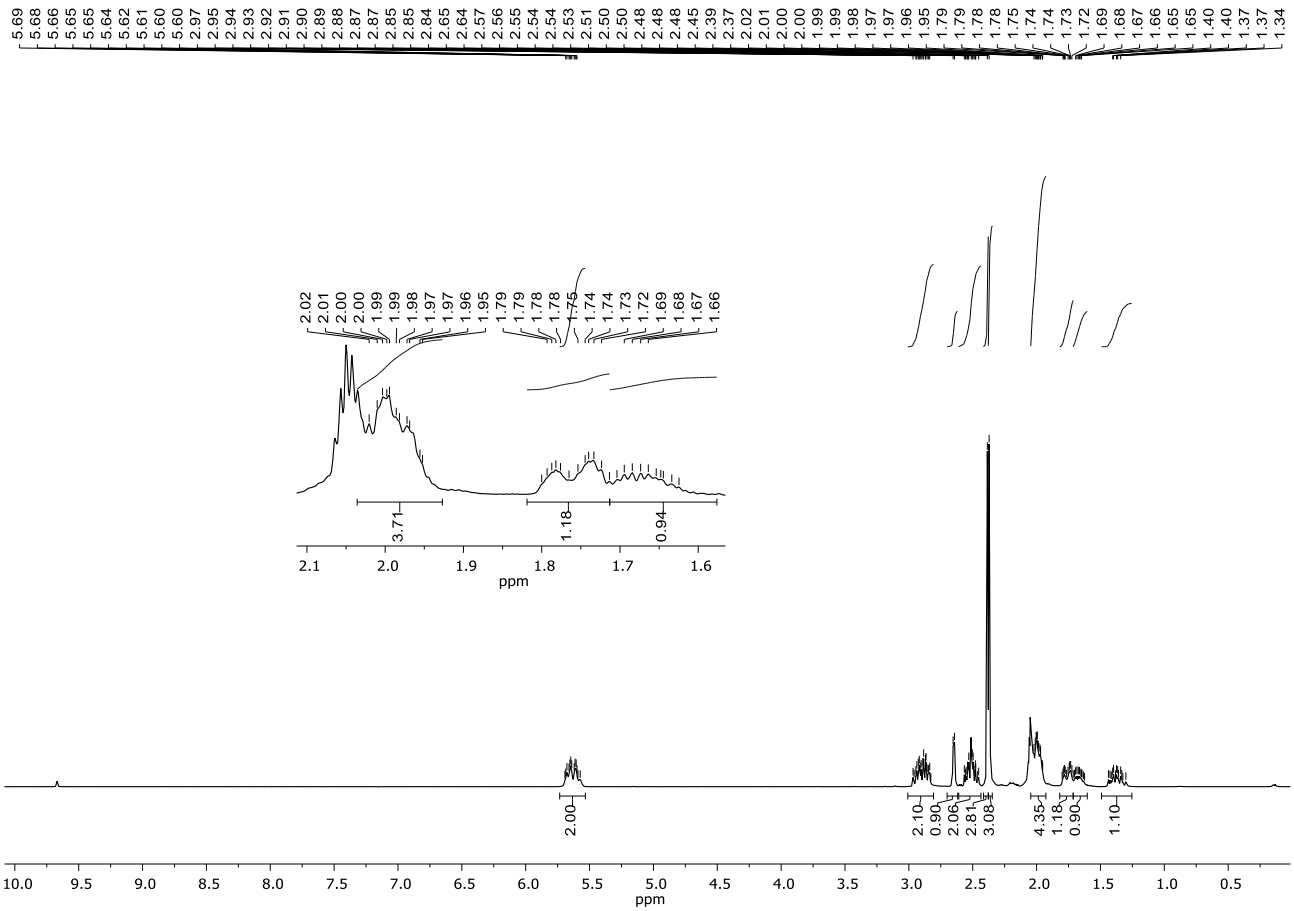

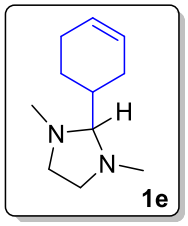

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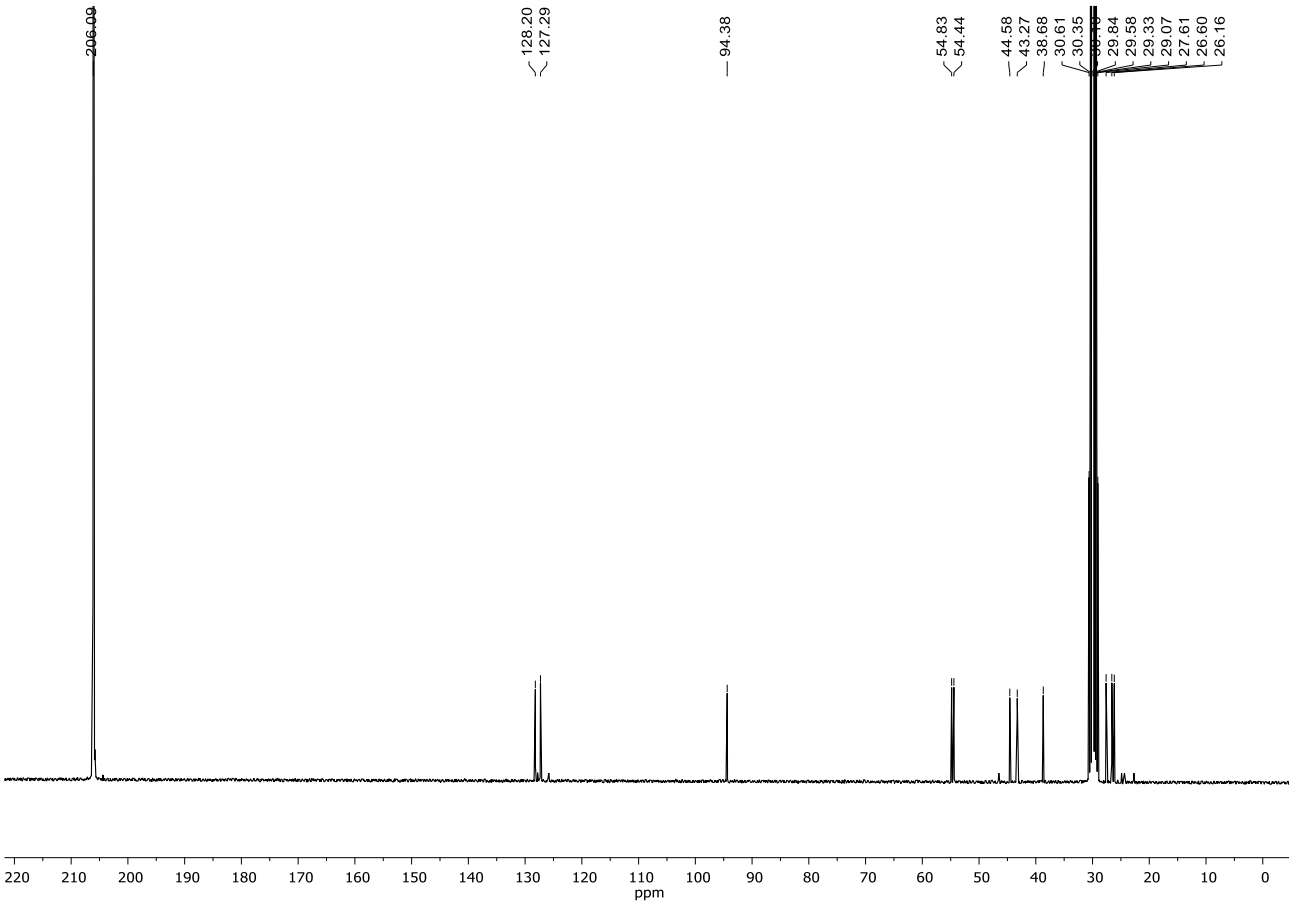

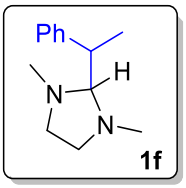

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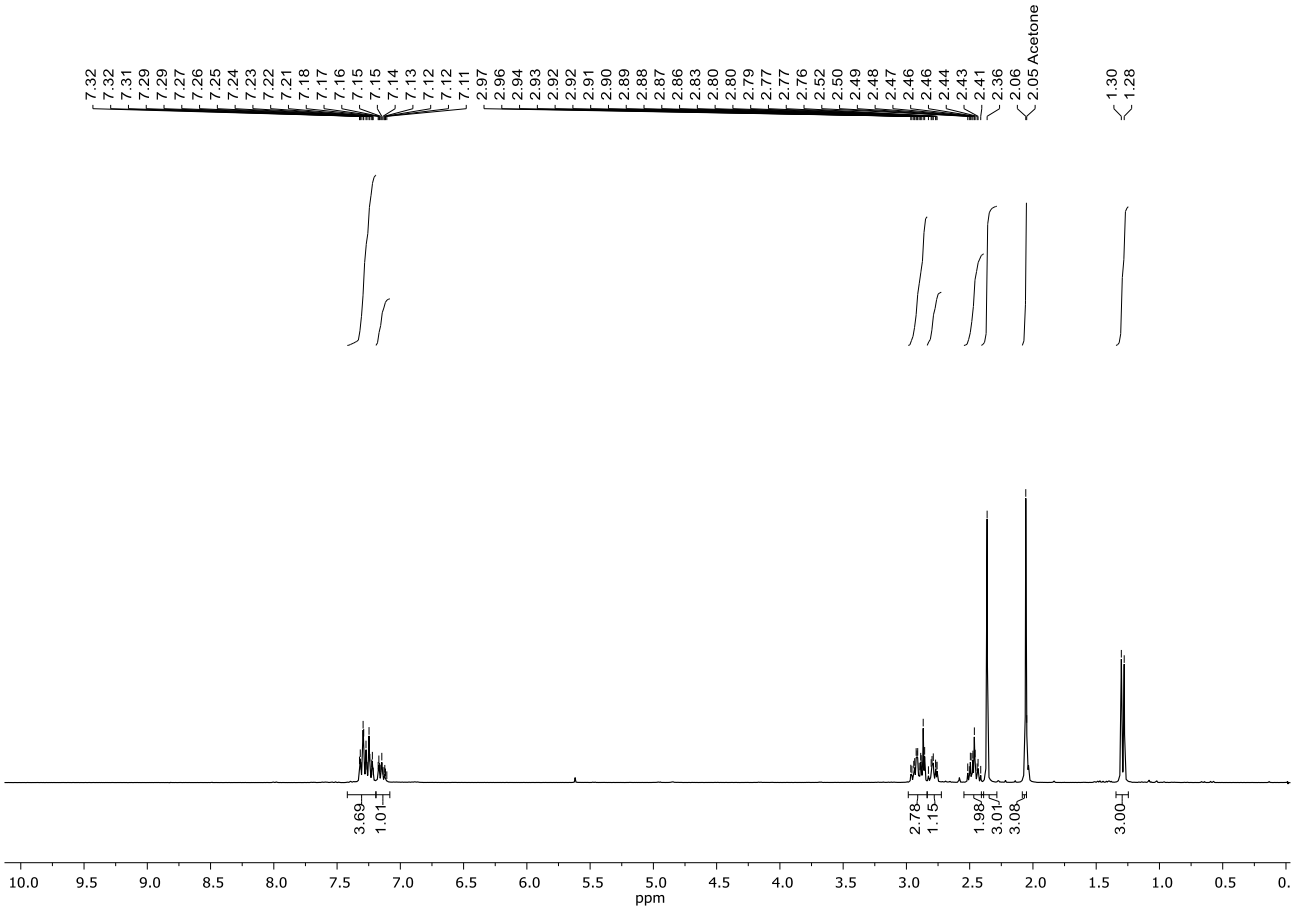

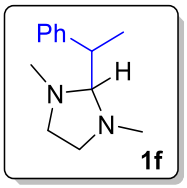

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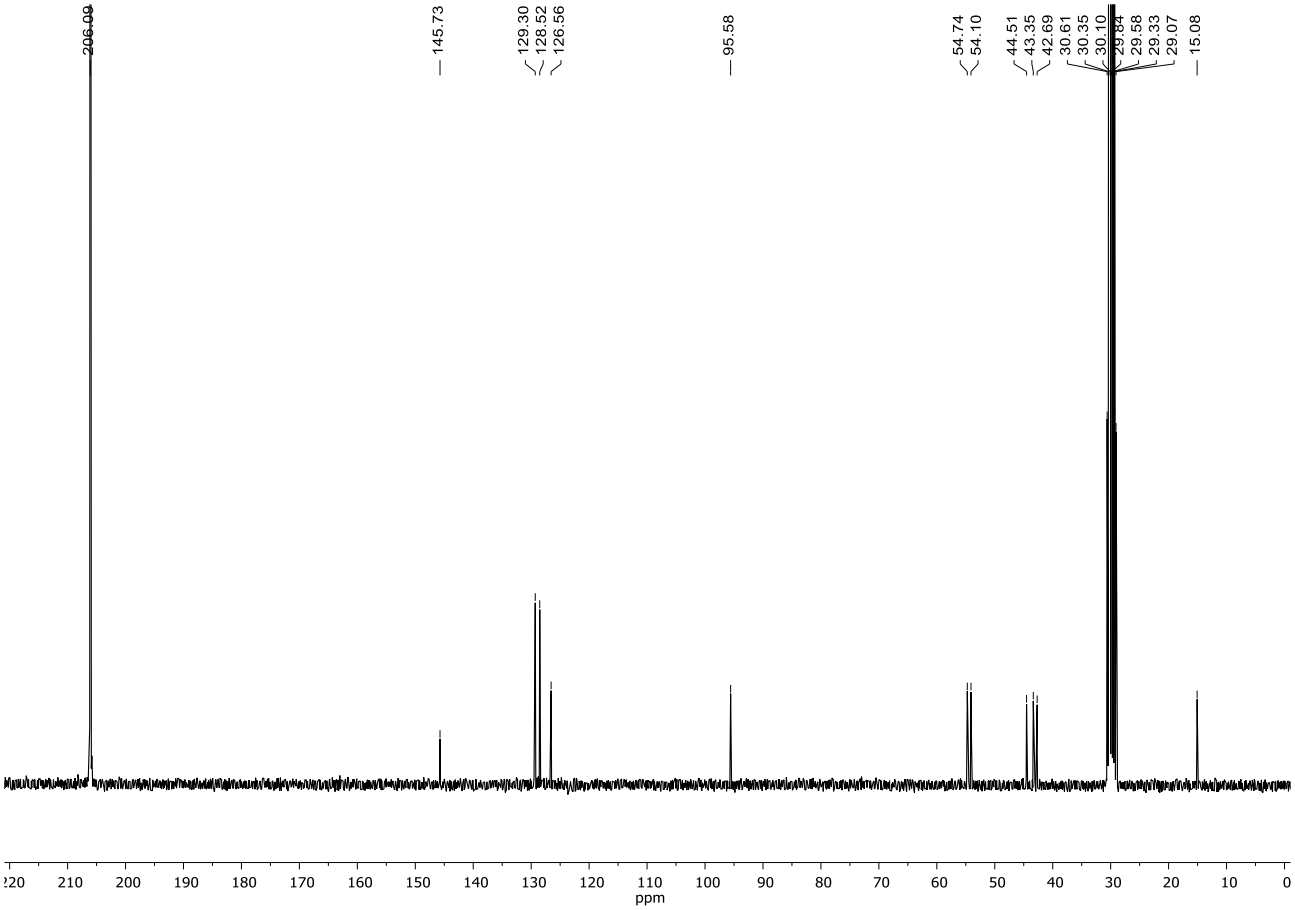

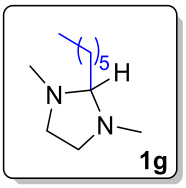

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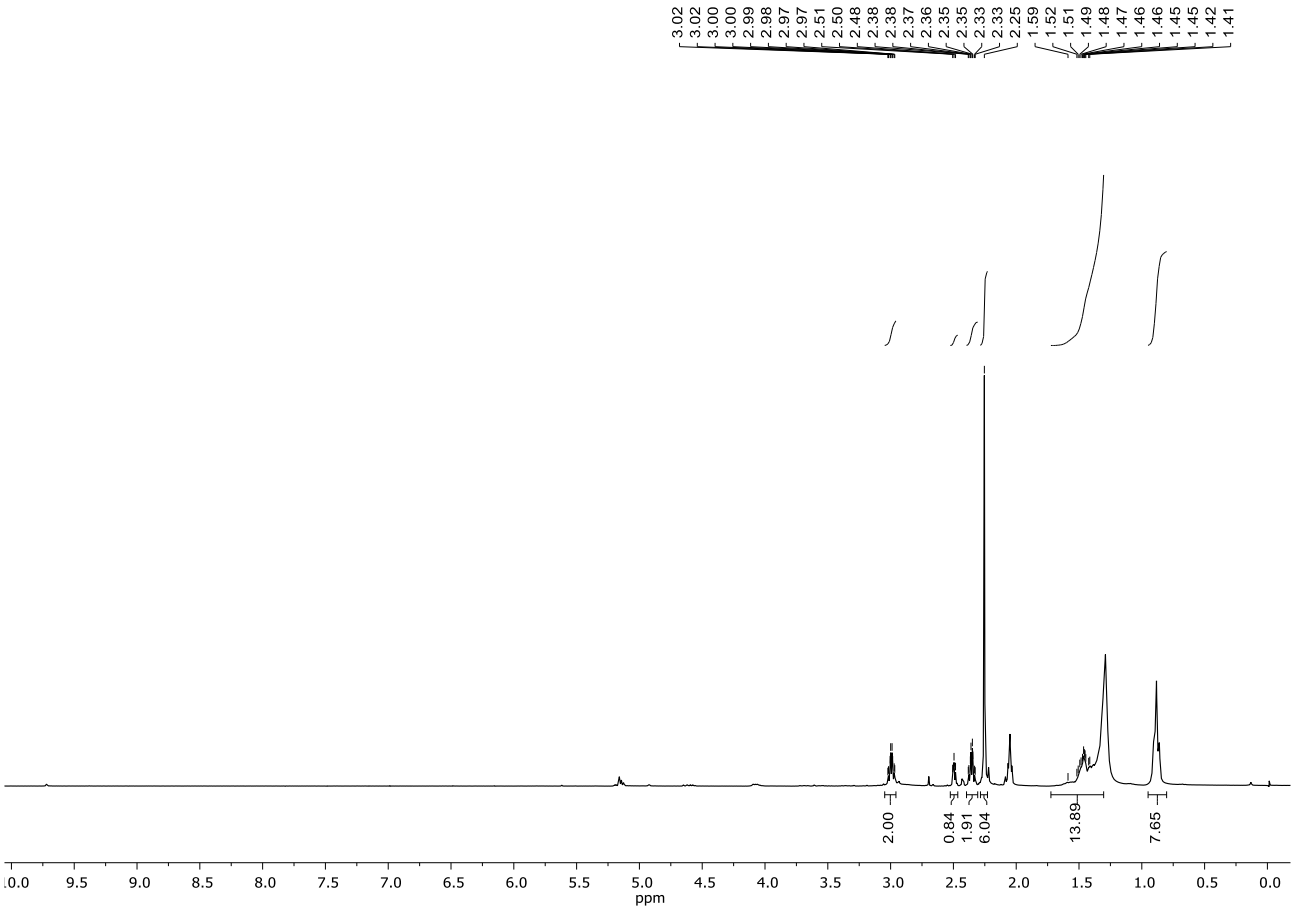

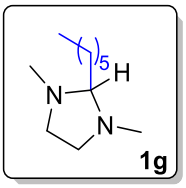

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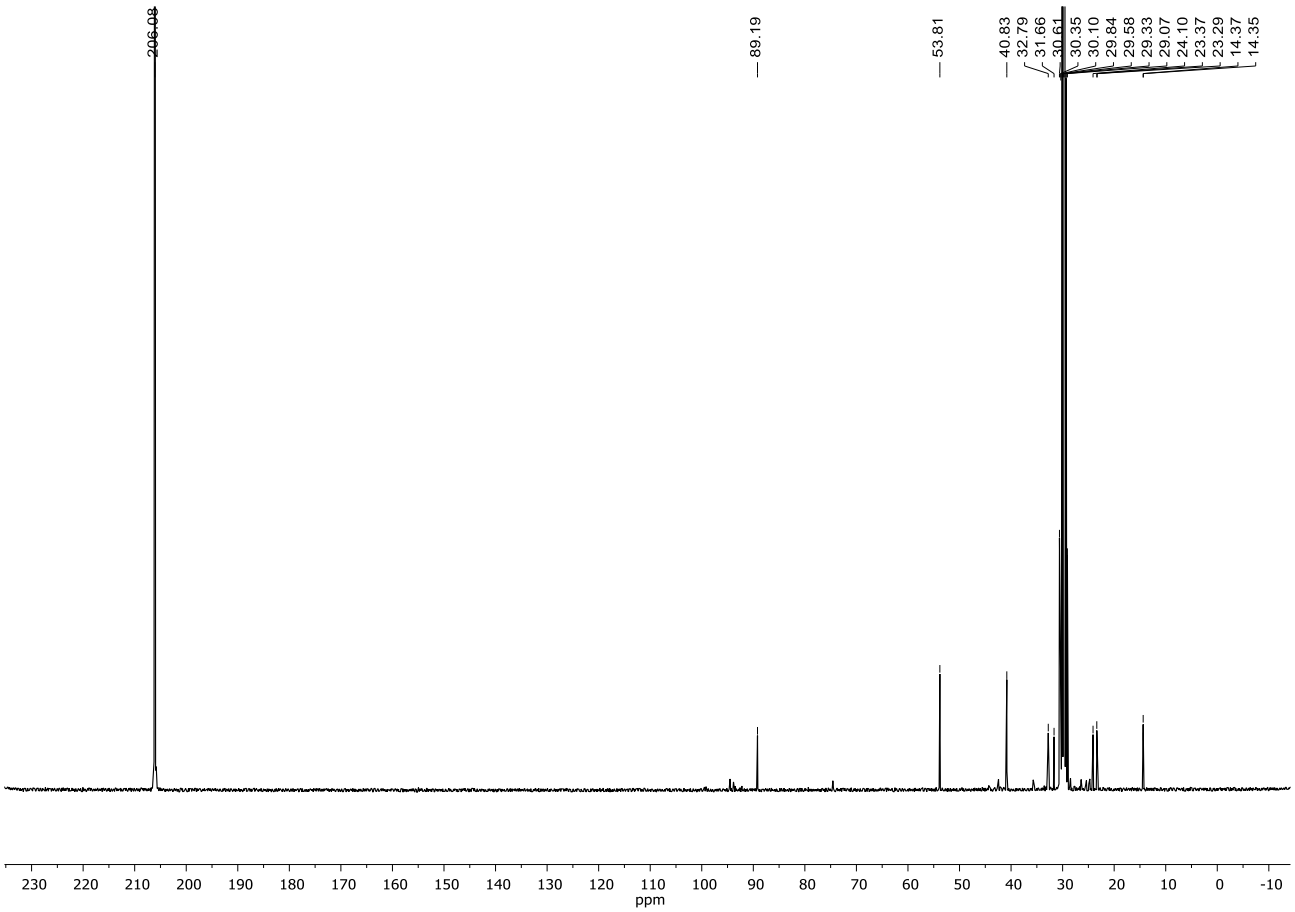

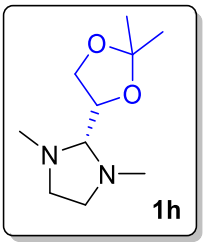

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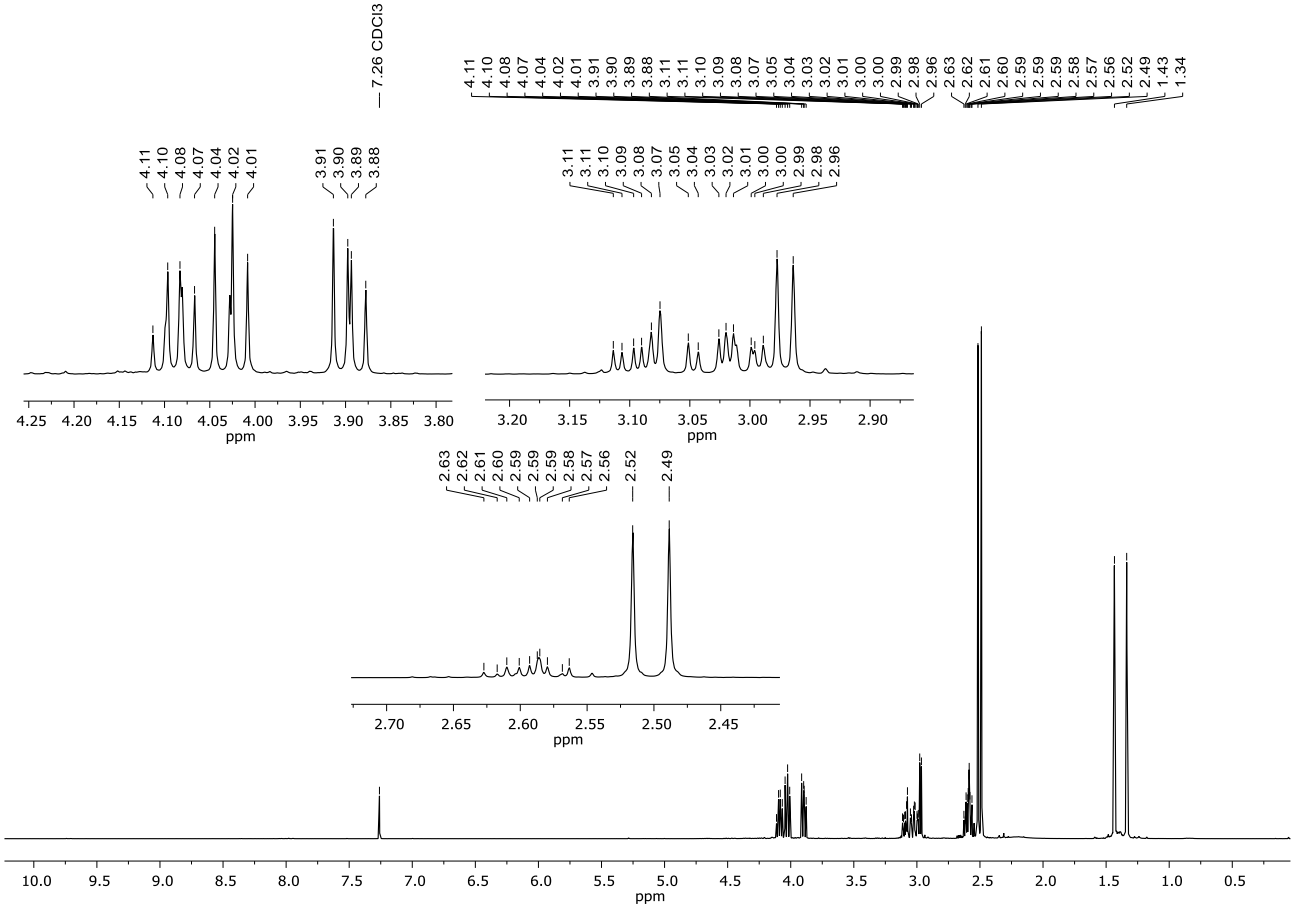

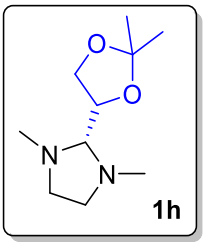

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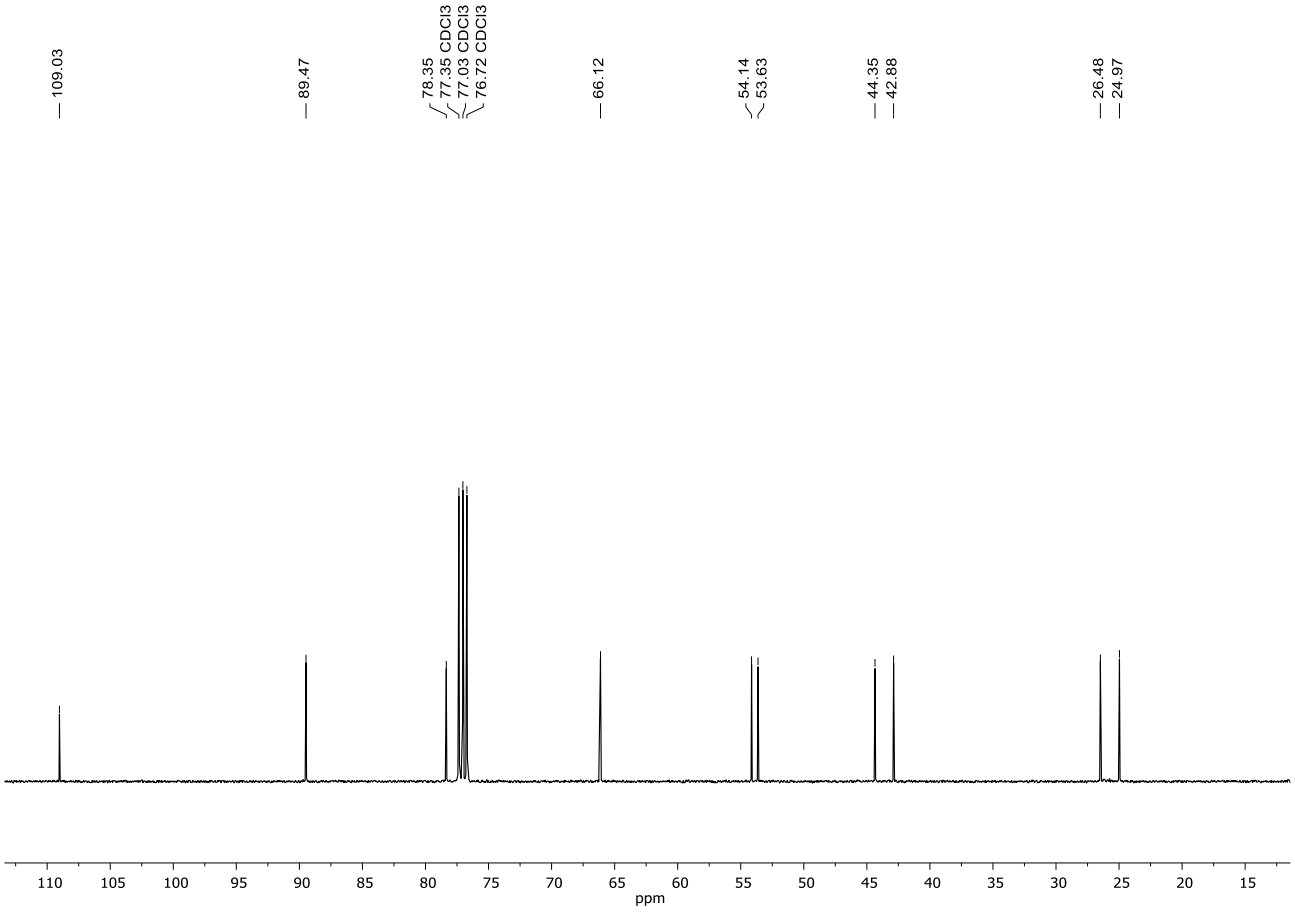
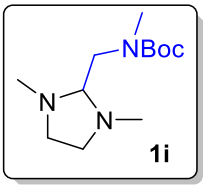
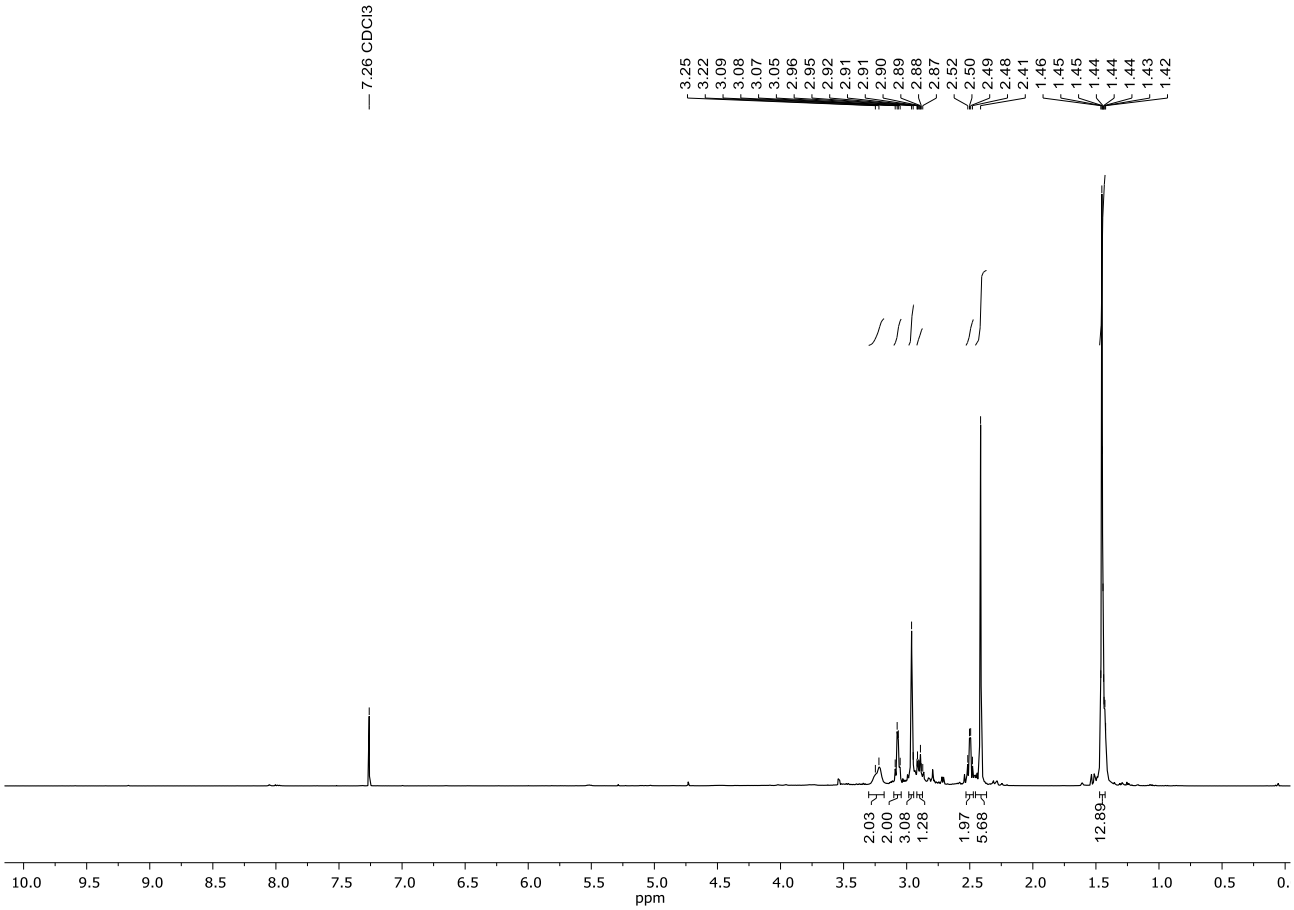
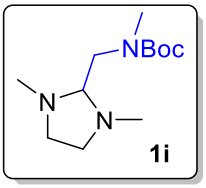


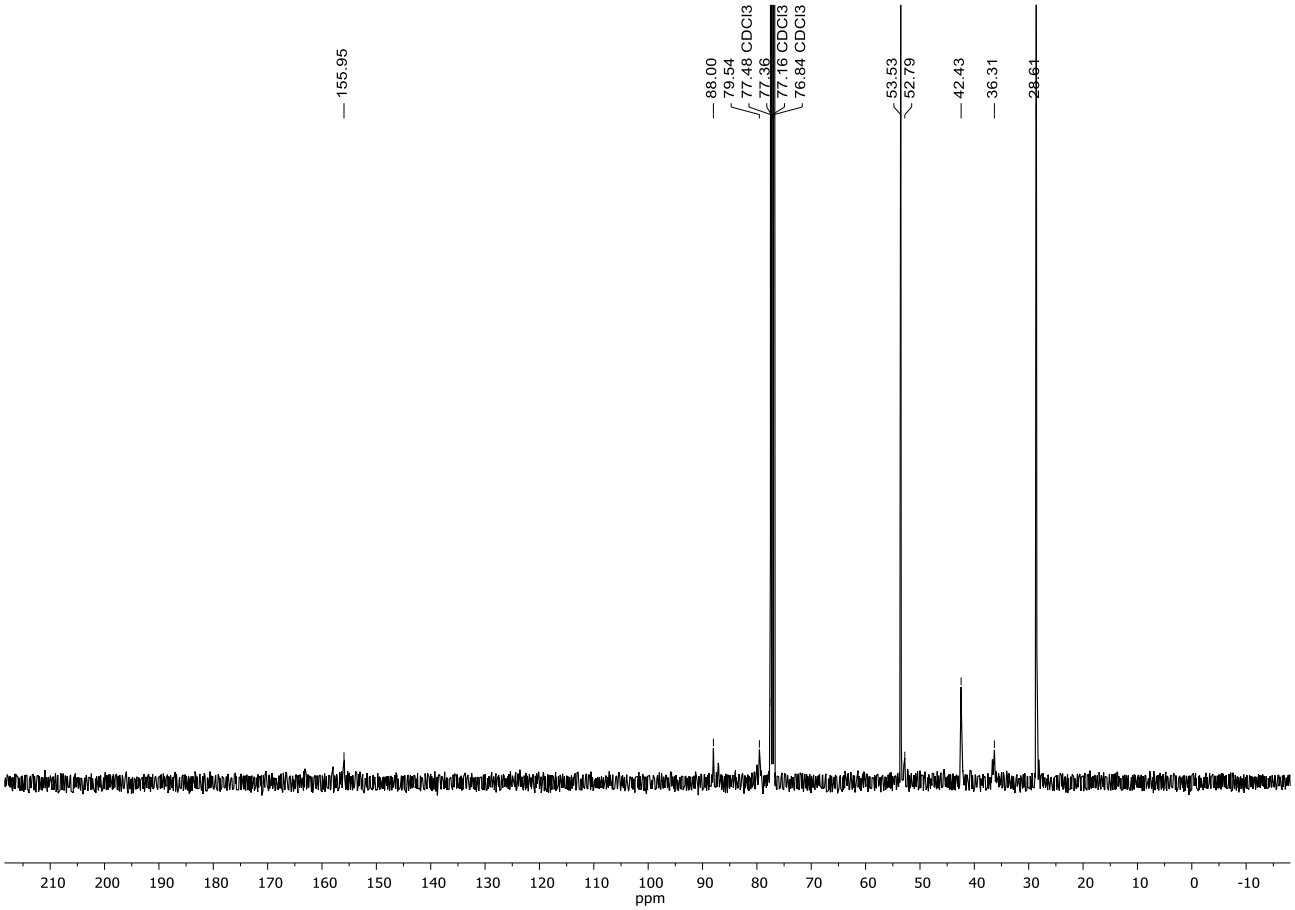


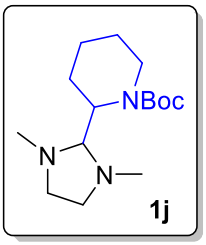
^{1}H-NMR (400 MHz, chloroform-*d*)



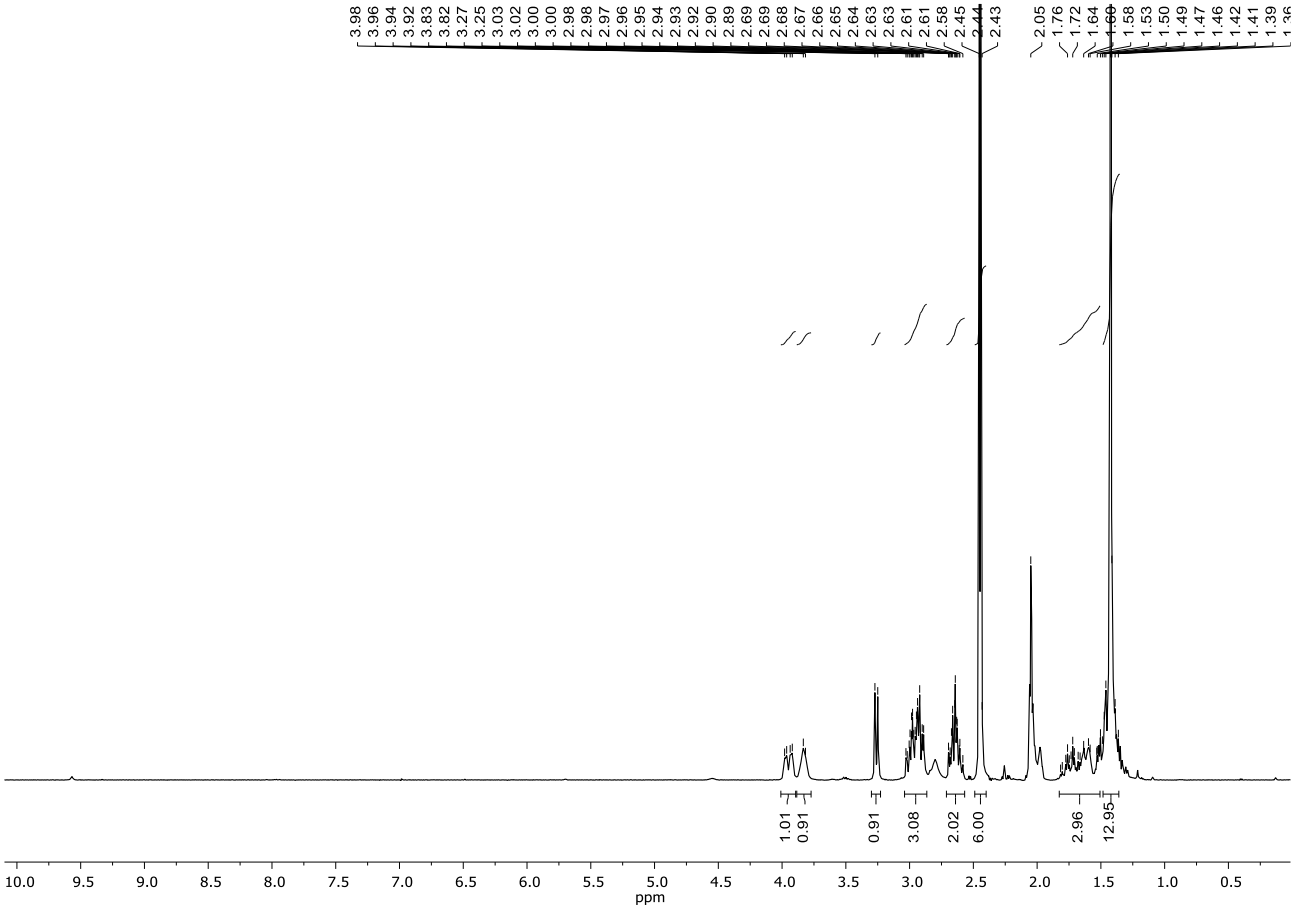


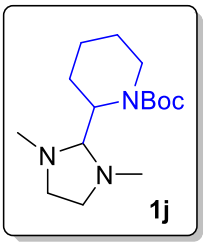
^{13}C-NMR (100 MHz, chloroform-*d*)



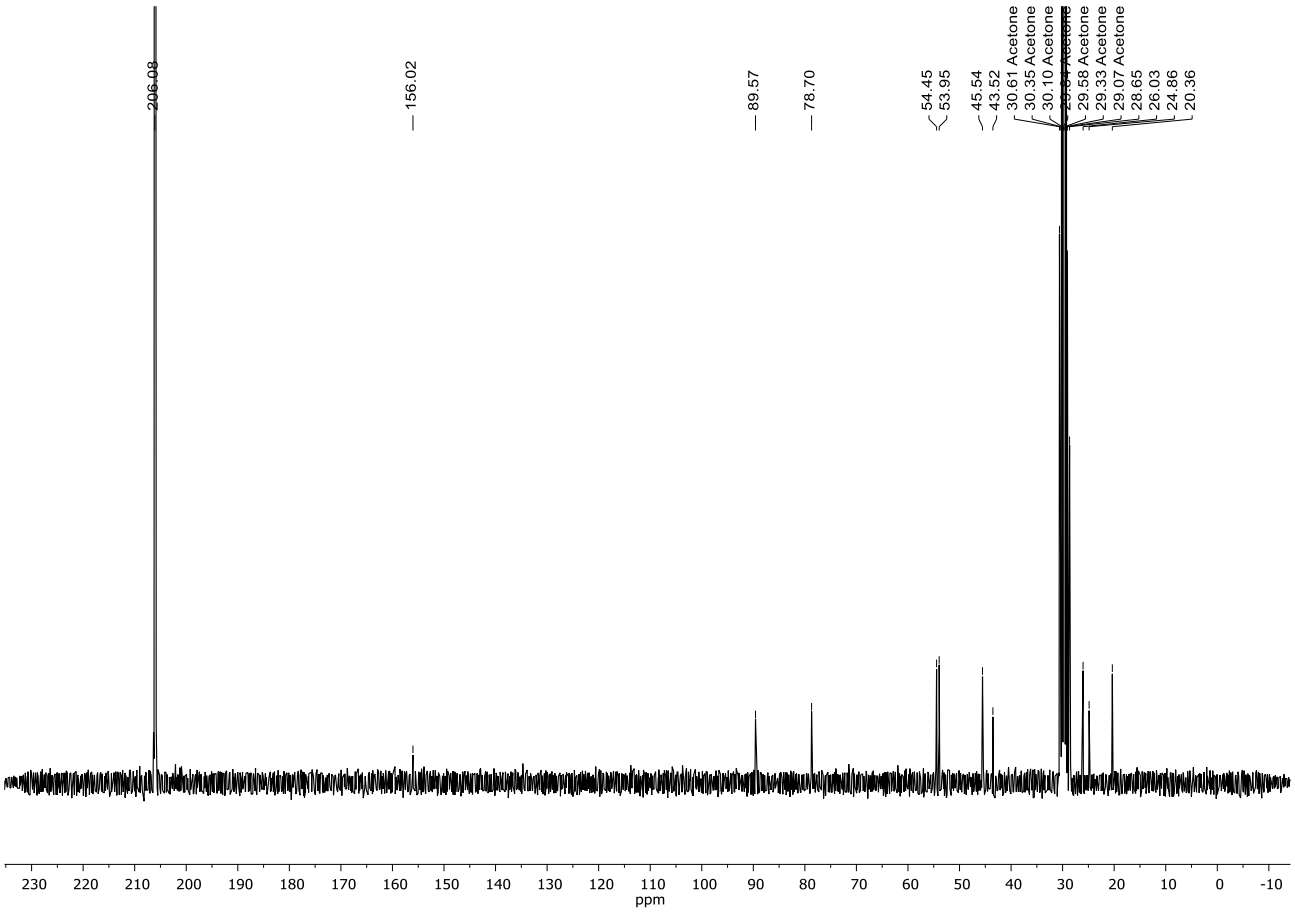


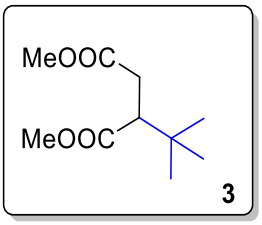
^{1}H-NMR (300 MHz, acetone-d_6)



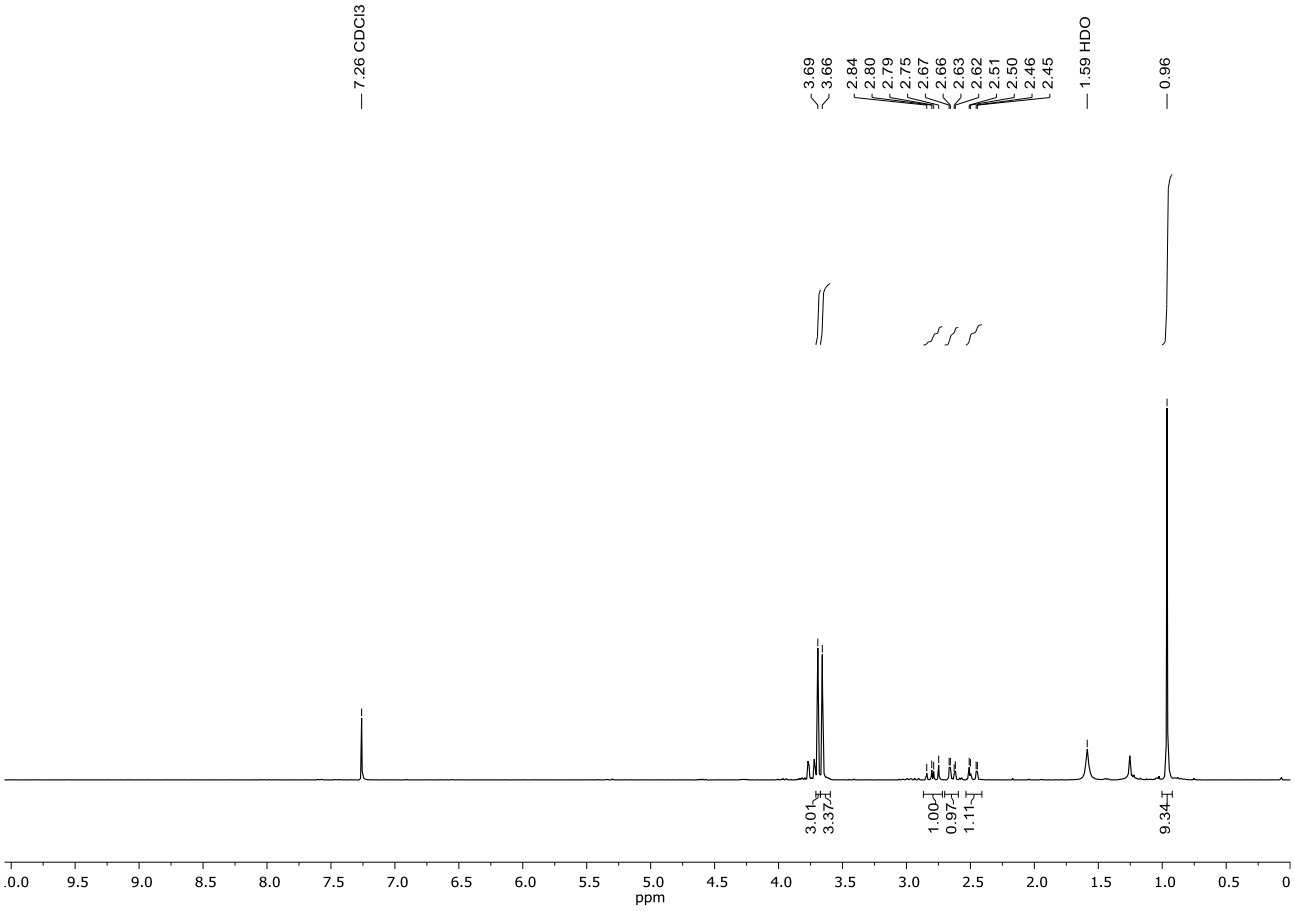


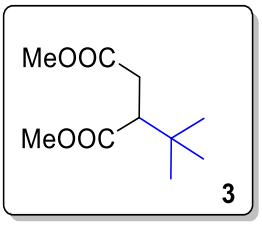
^{13}C-NMR (75 MHz, acetone-d_6)



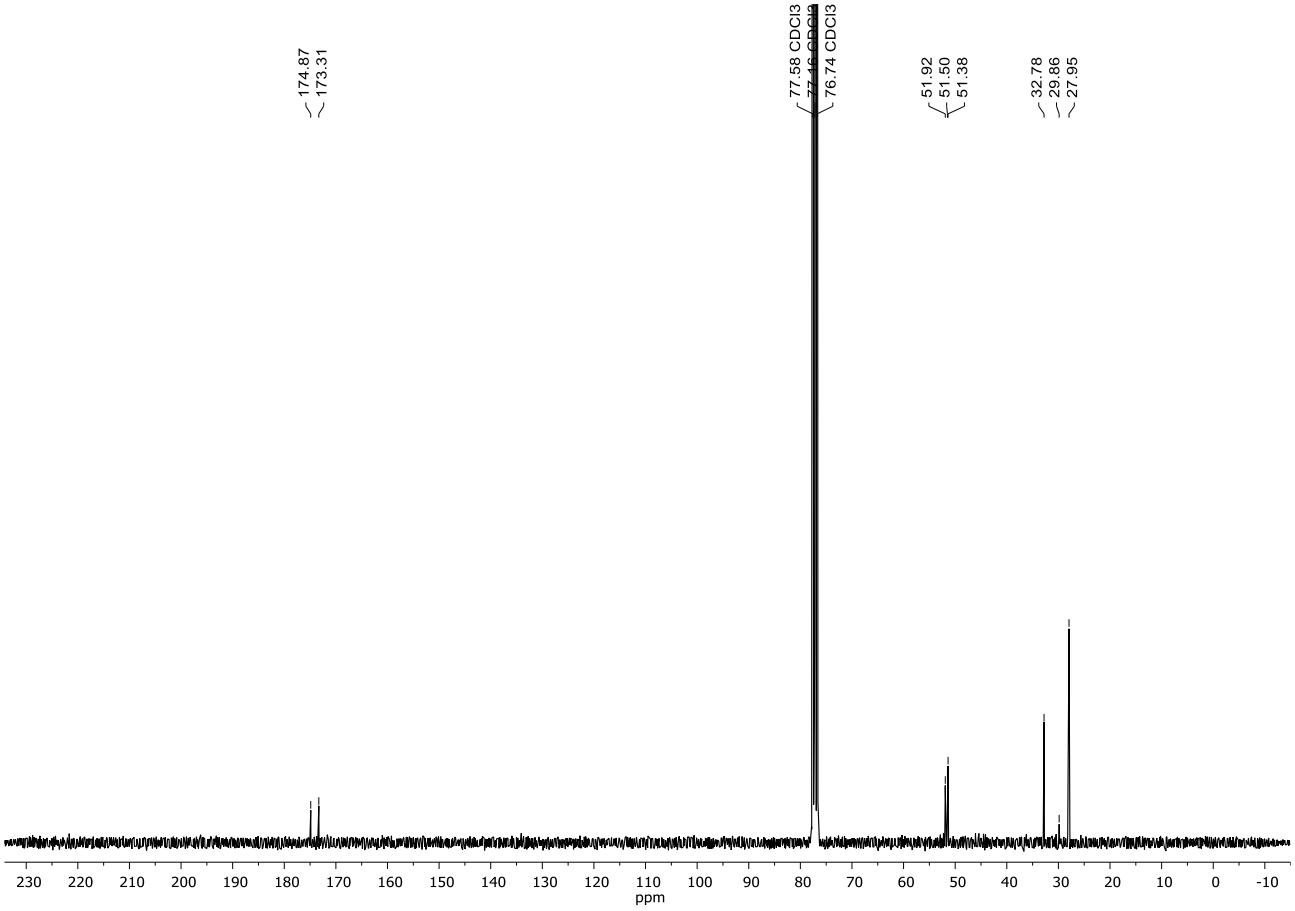


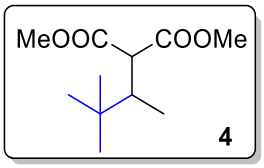
^{1}H-NMR (300 MHz, chloroform-*d*)



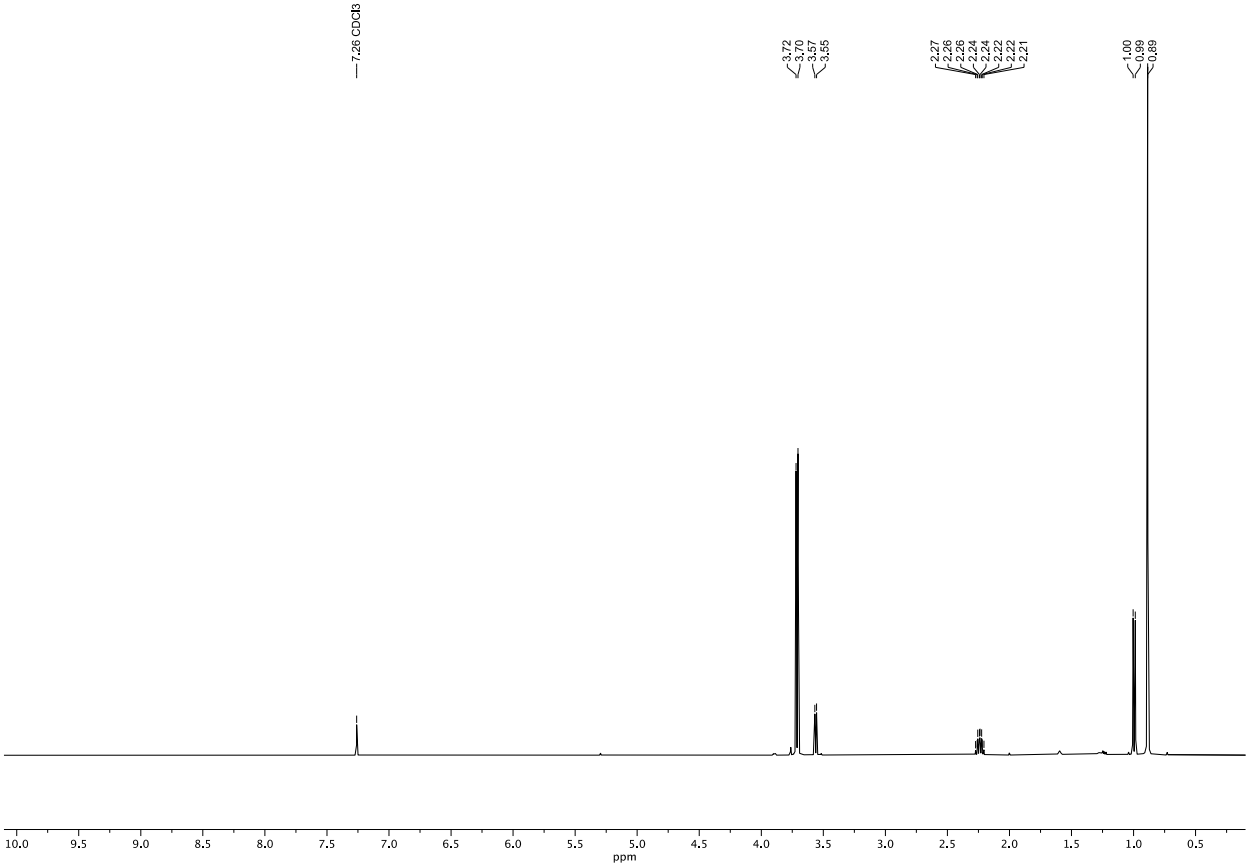


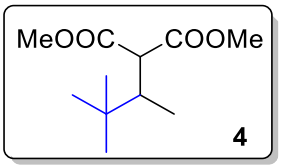
^{13}C-NMR (75 MHz, chloroform-*d*)



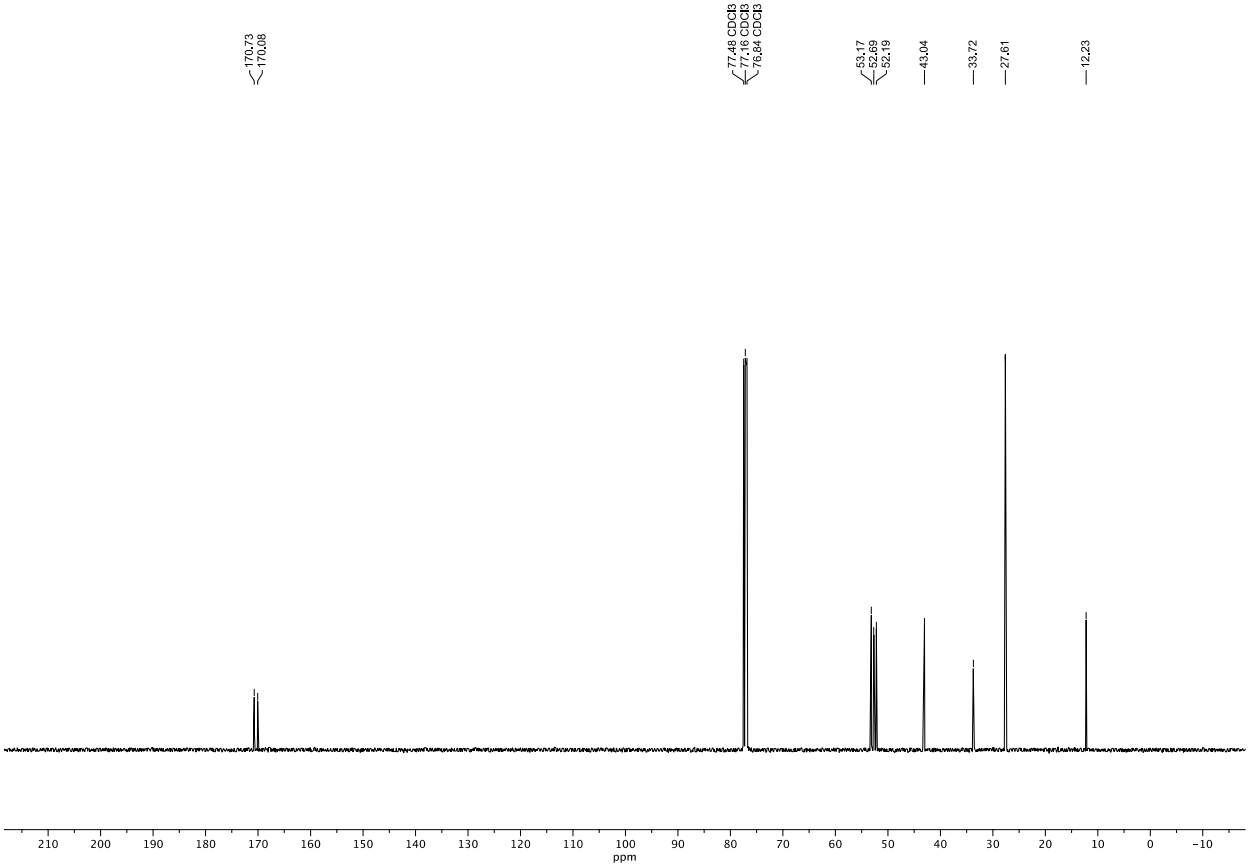


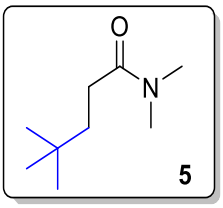
^{1}H-NMR (400 MHz, chloroform-*d*)



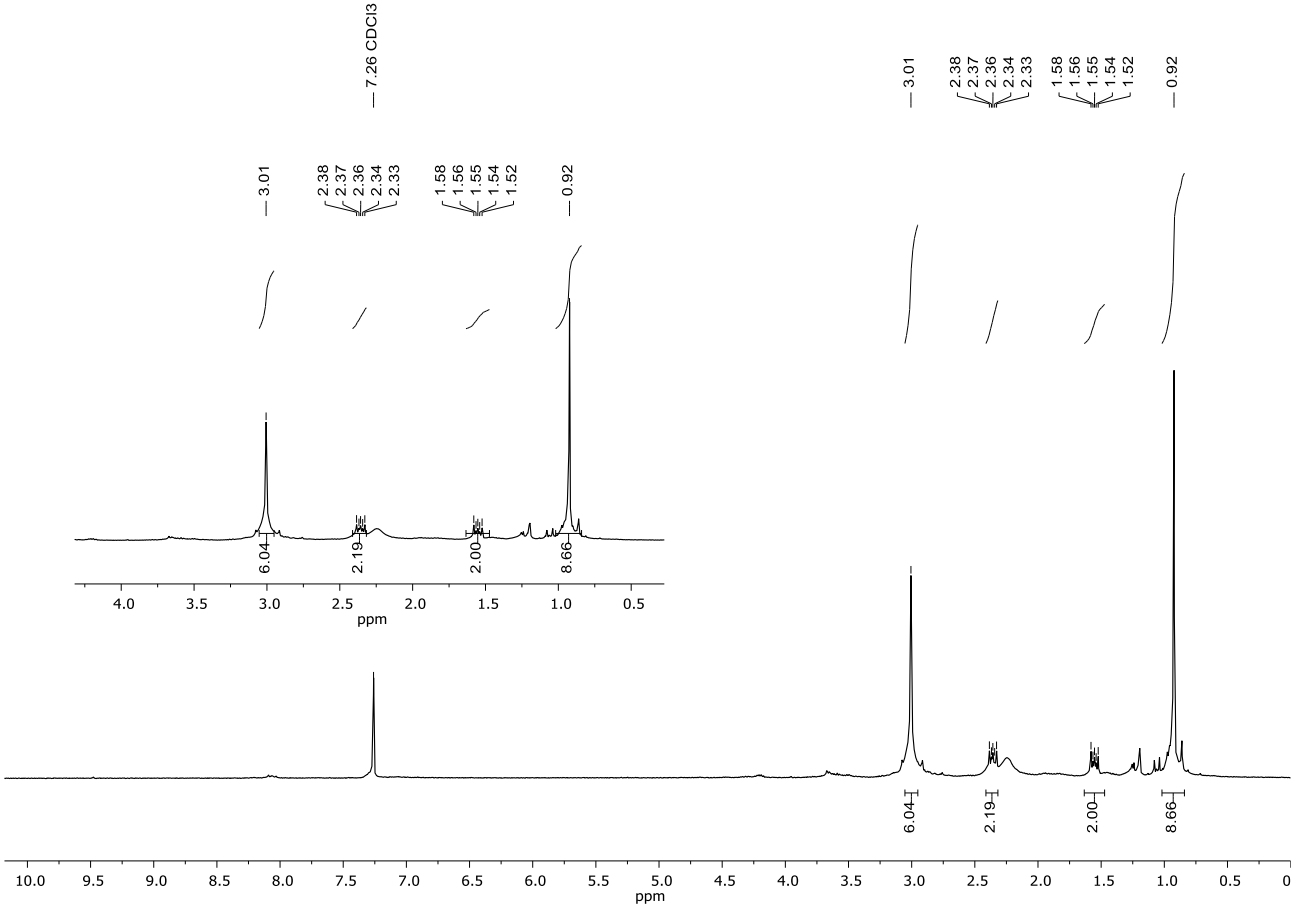


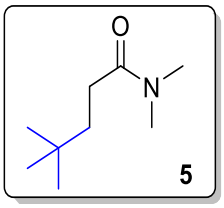
^{13}C-NMR (100 MHz, chloroform-*d*)



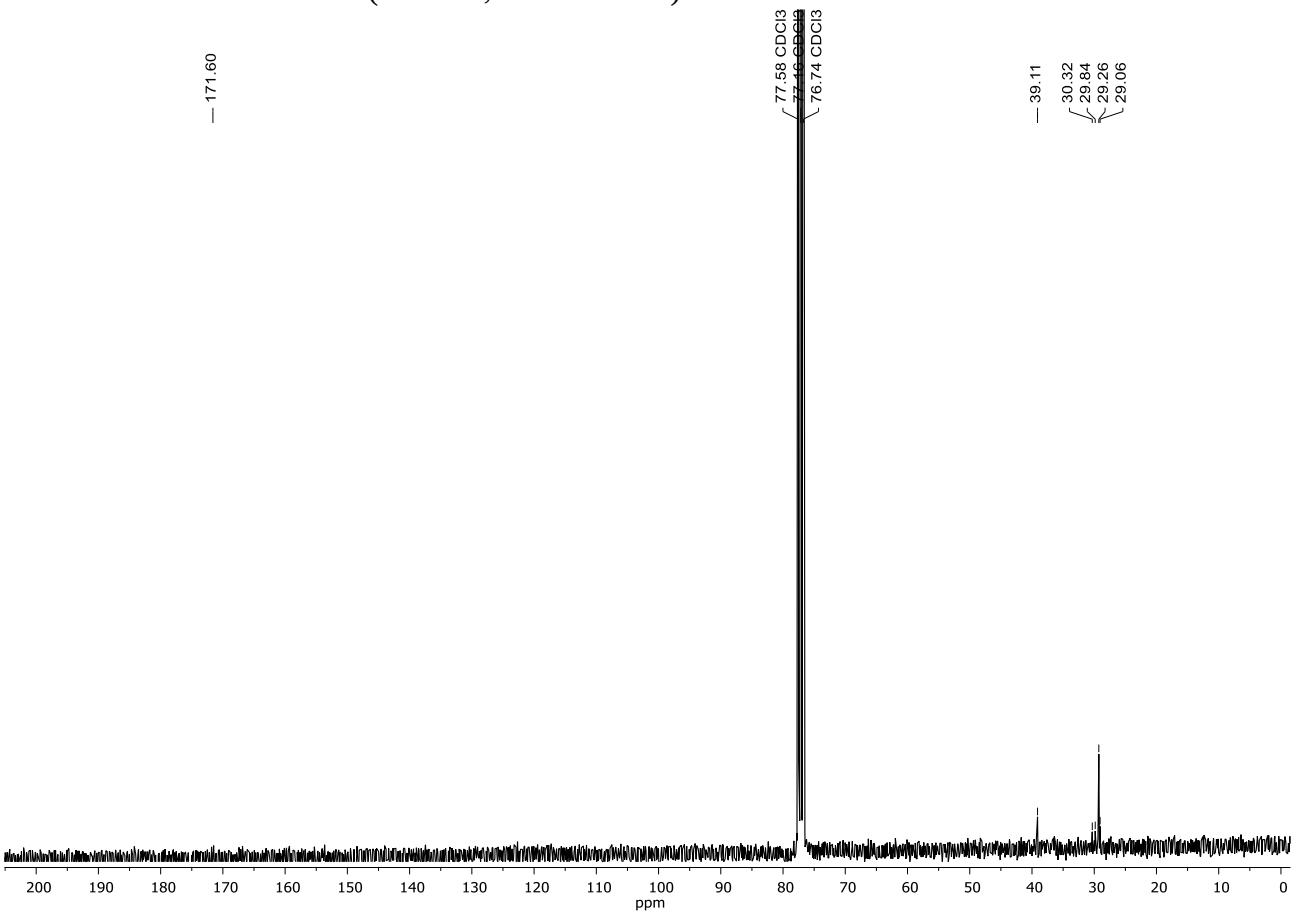


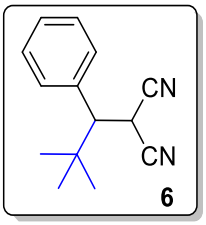
^{1}H-NMR (300 MHz, chloroform-*d*)



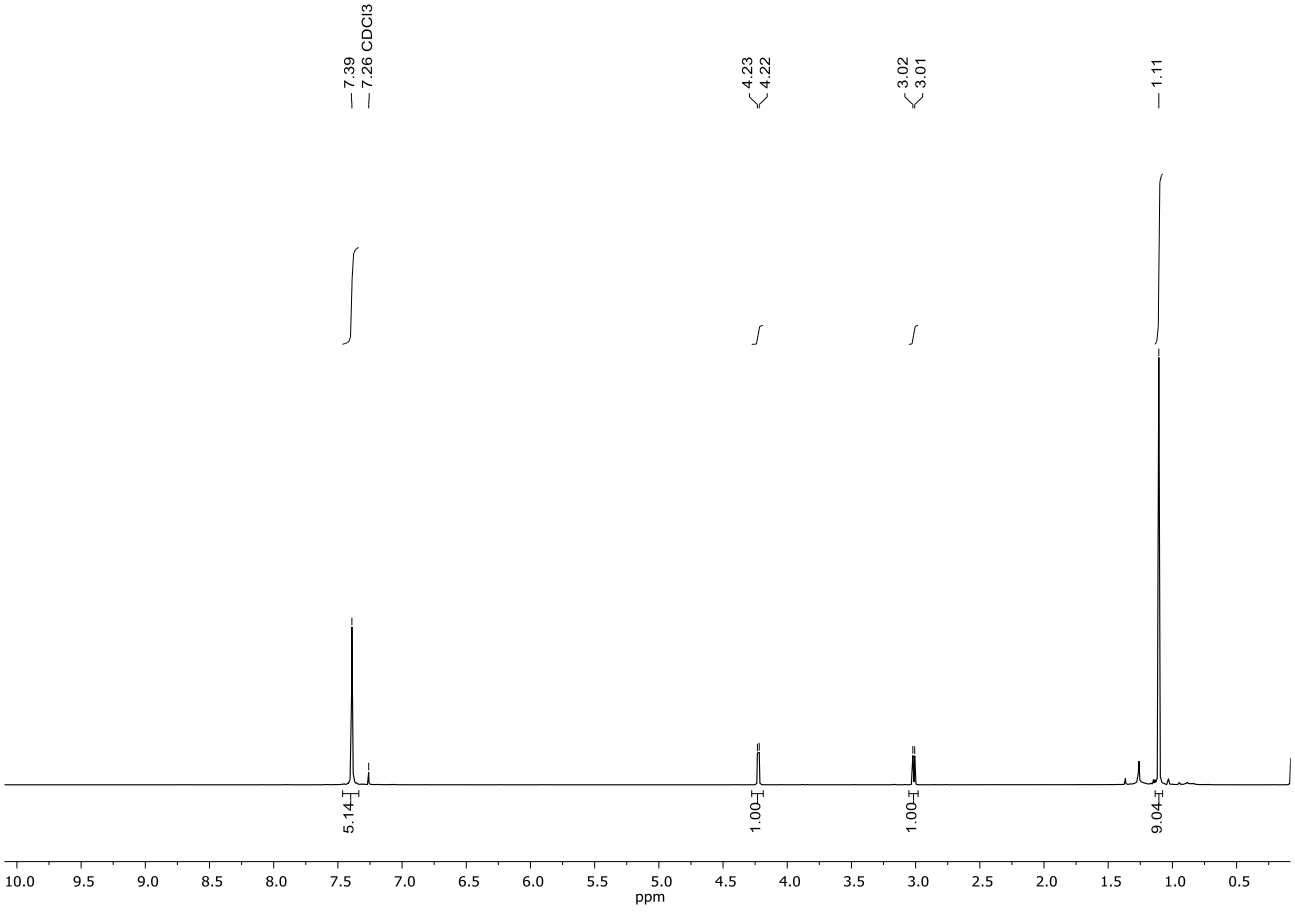


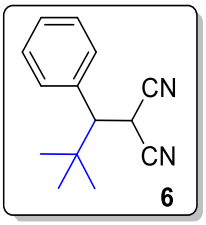
^{13}C-NMR (75 MHz, chloroform-*d*)



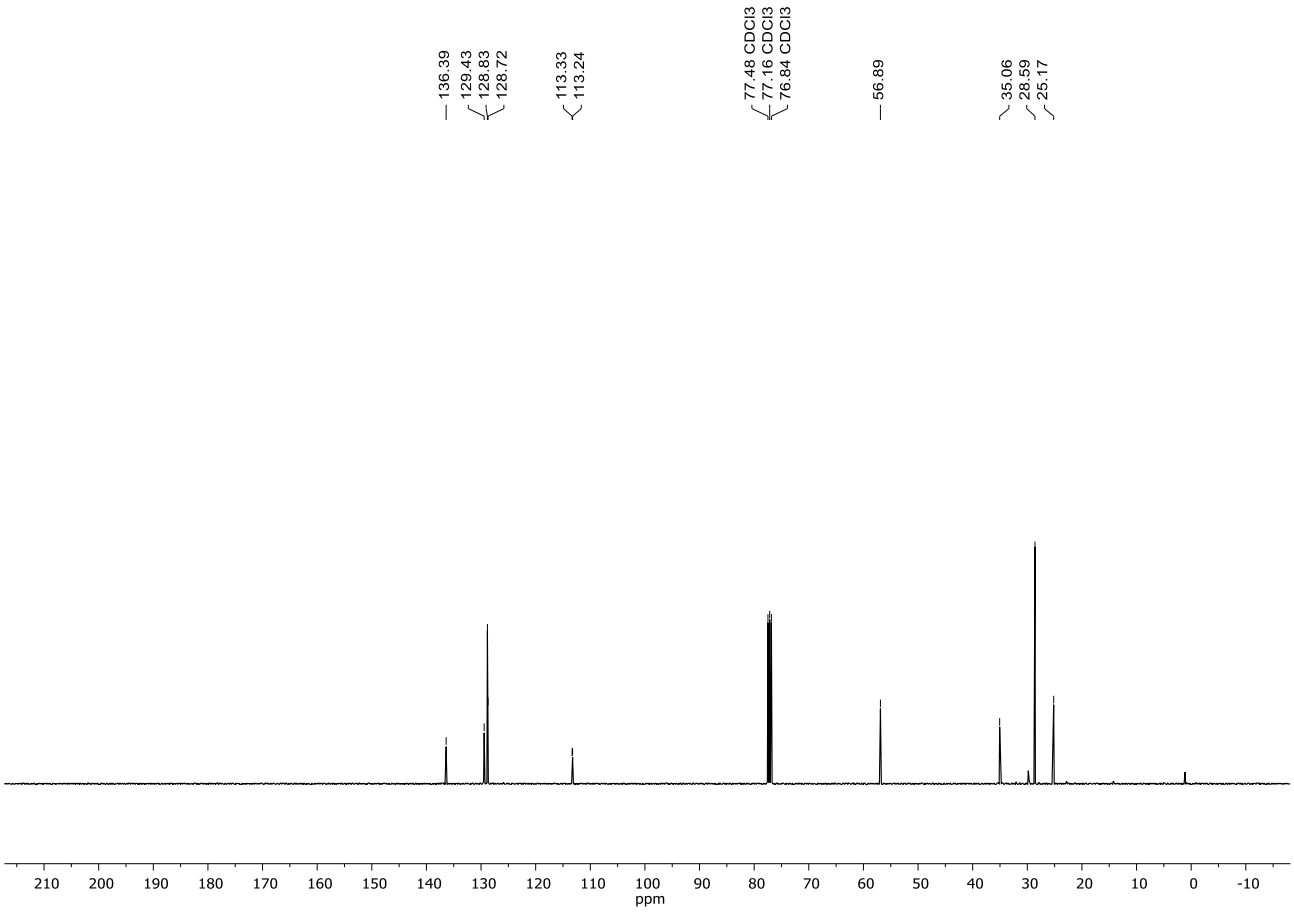


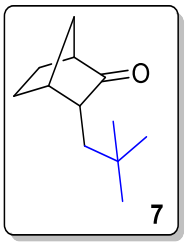
^{1}H-NMR (400 MHz, chloroform-*d*)



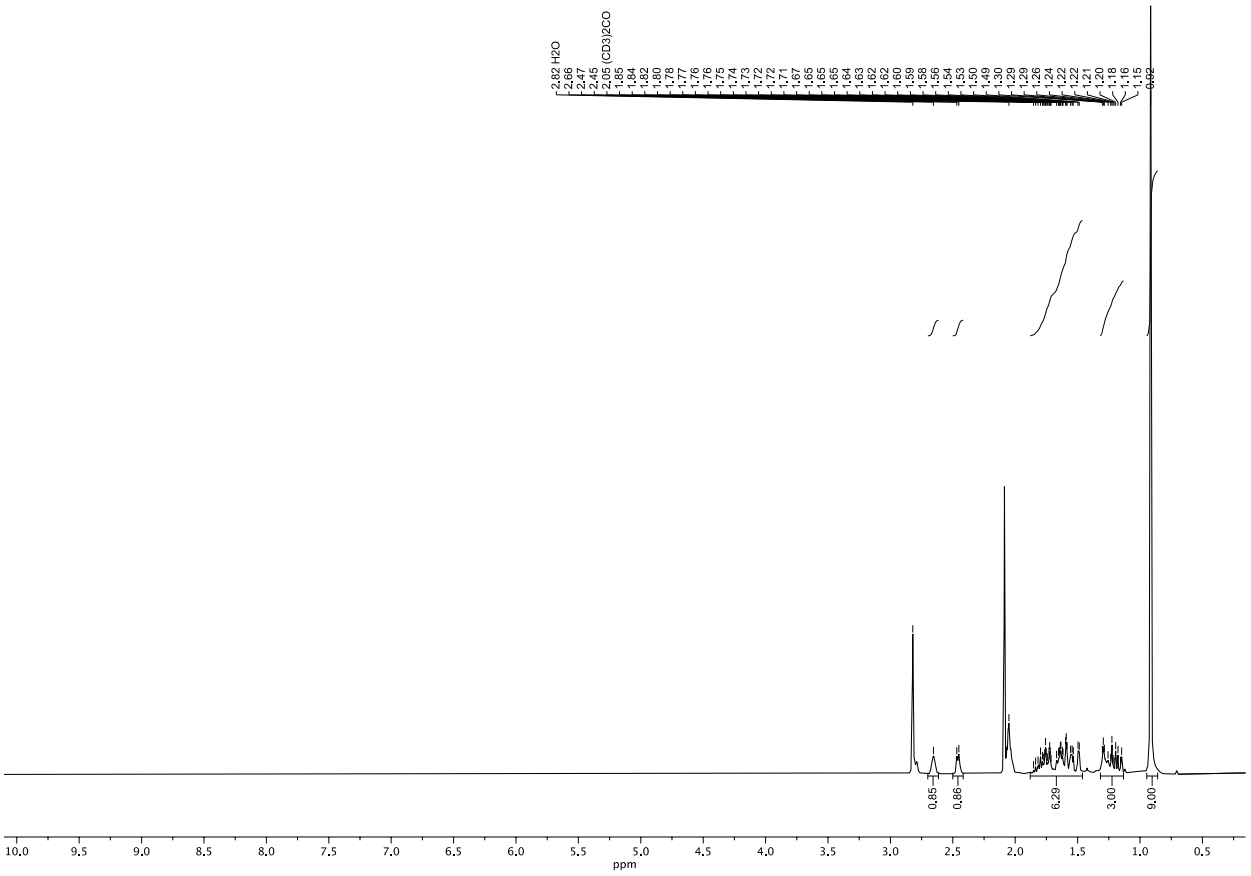


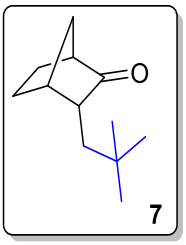
^{13}C-NMR (100 MHz, chloroform-*d*)



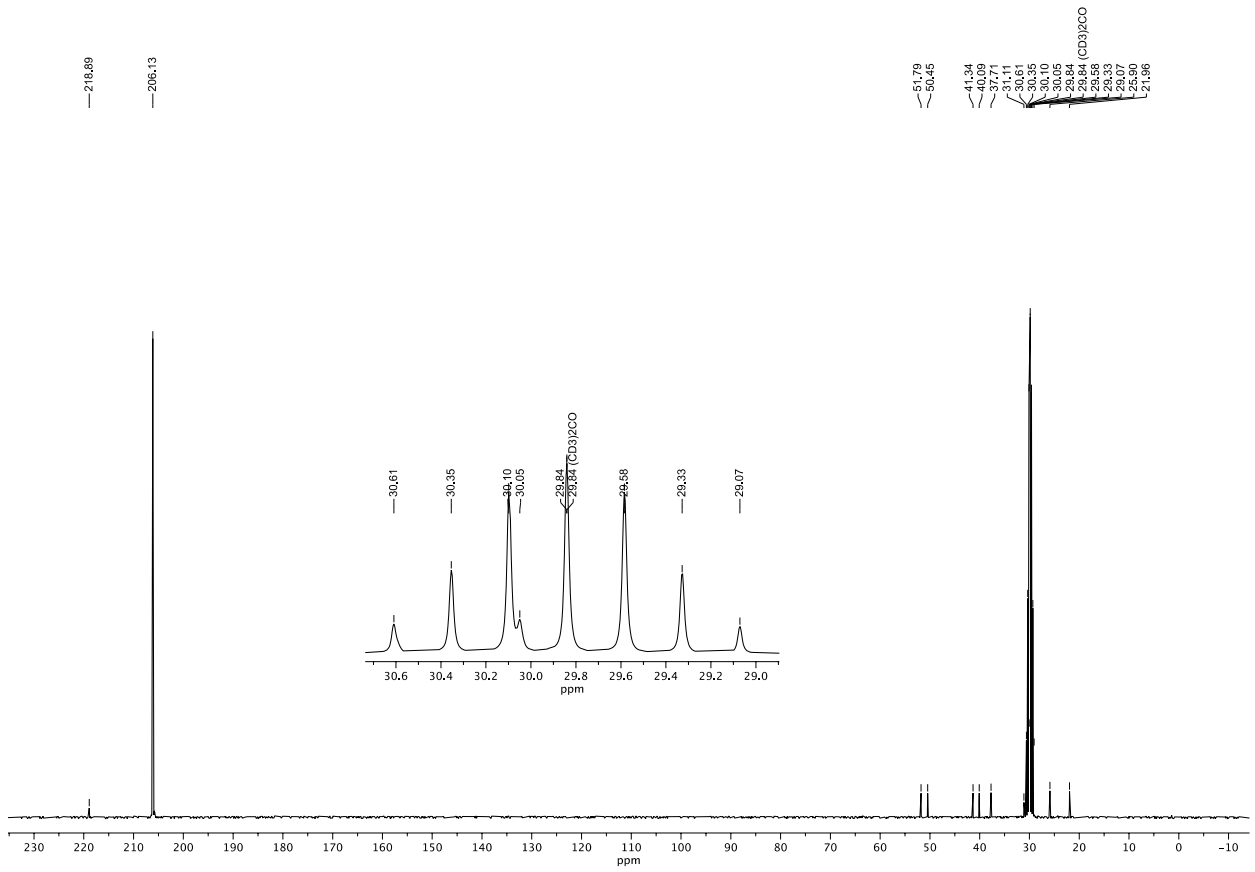


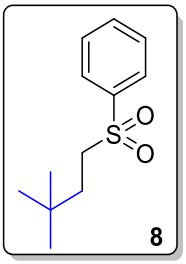
^{1}H-NMR (300 MHz, acetone-d_6)



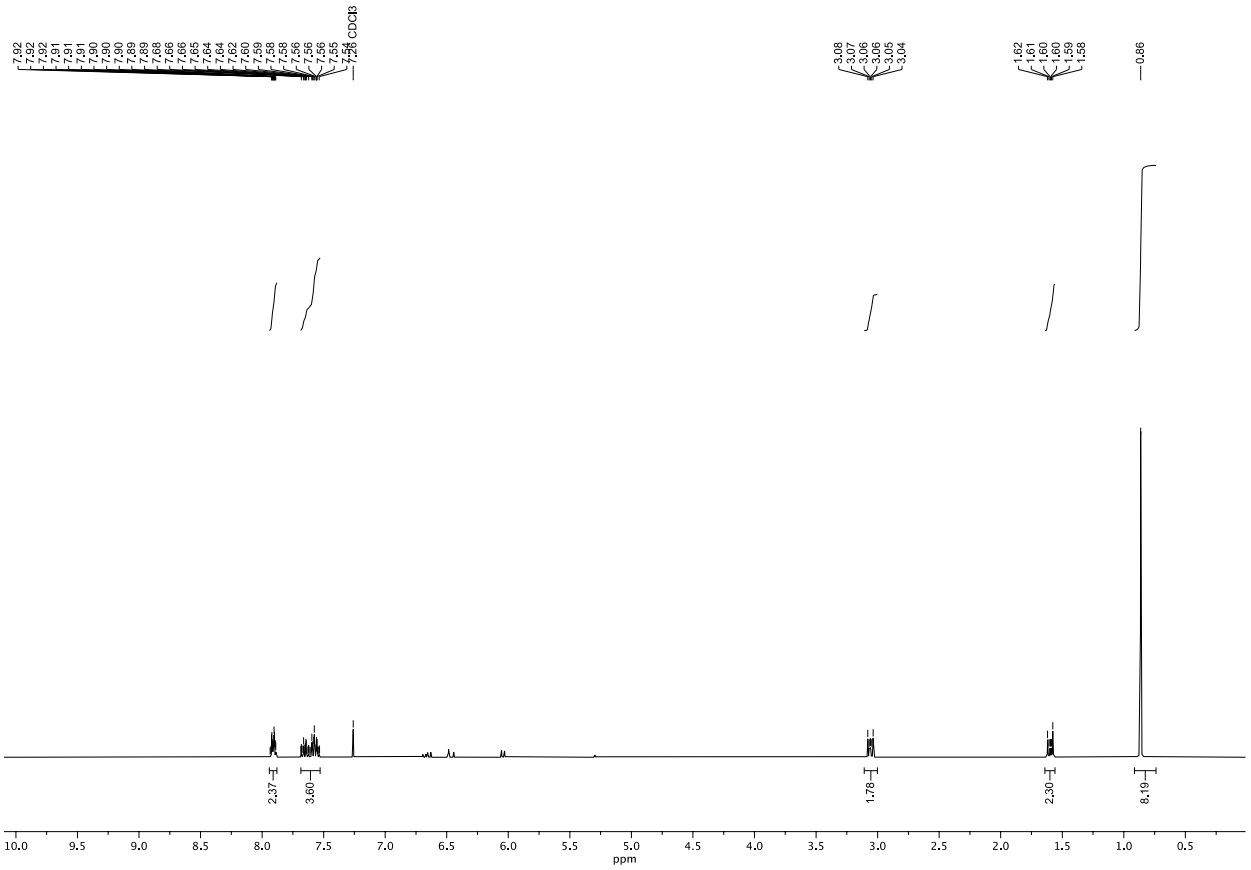


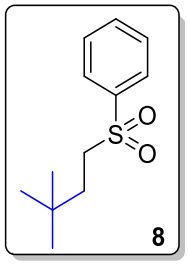
^{13}C-NMR (75 MHz, acetone-d_6)



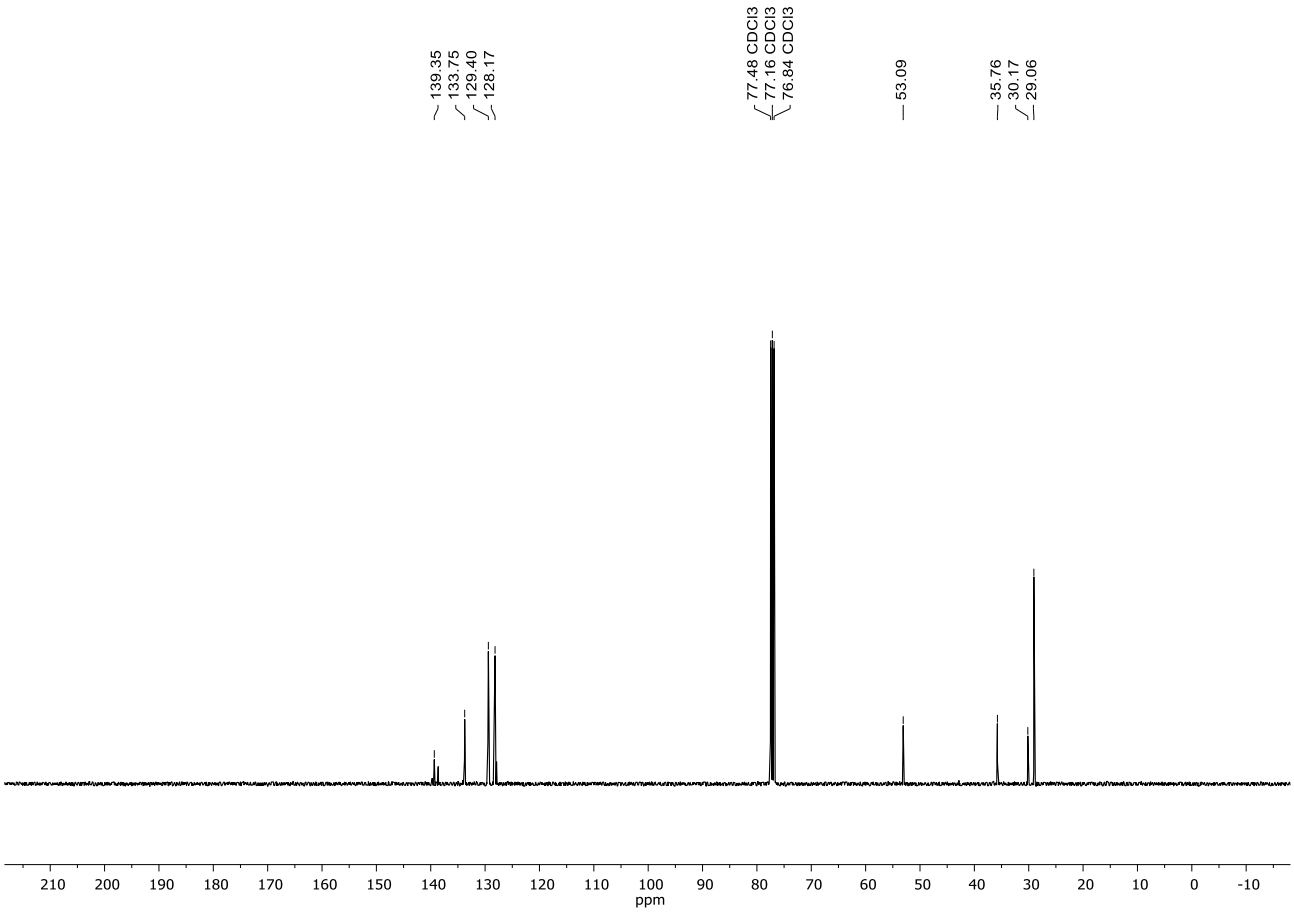


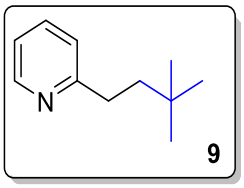
^{1}H-NMR (400 MHz, chloroform-*d*)



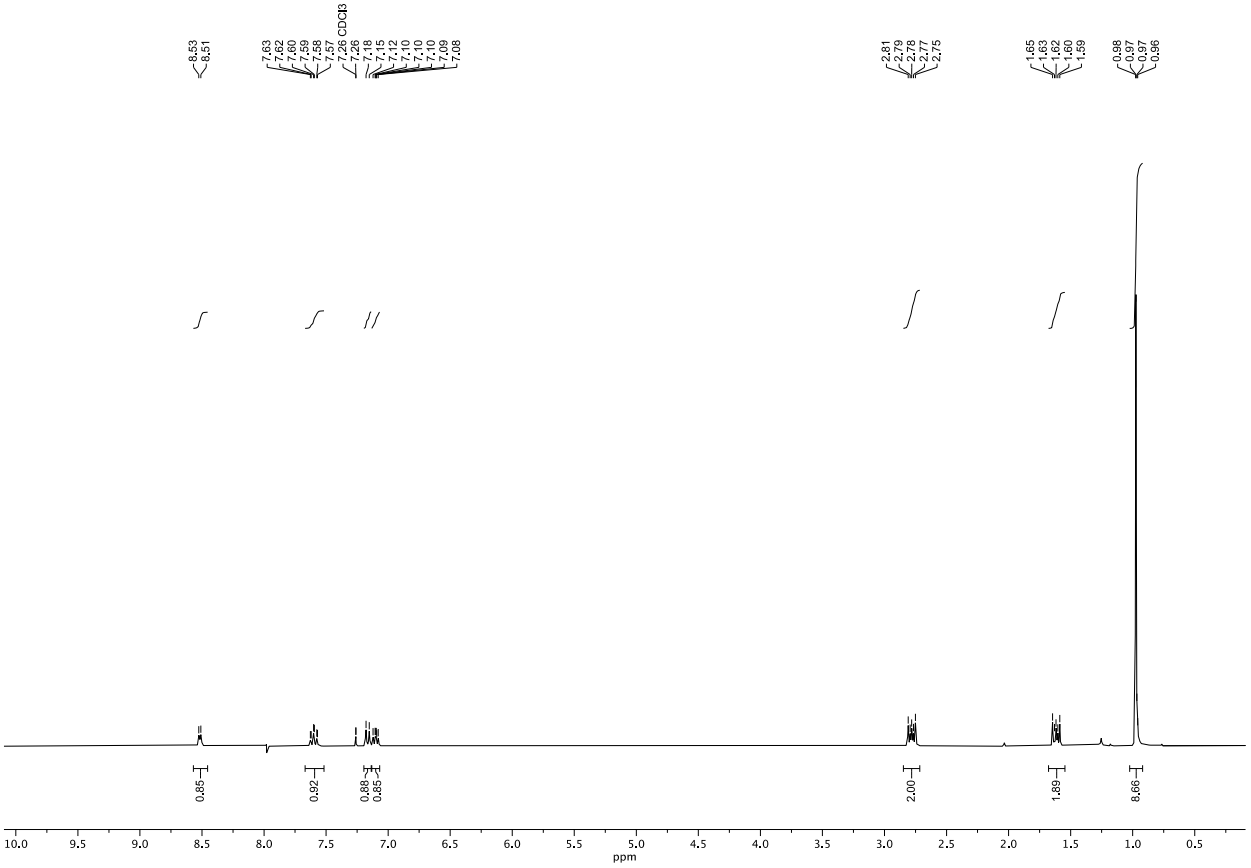


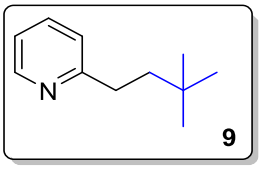
^{13}C-NMR (100 MHz, chloroform-*d*)



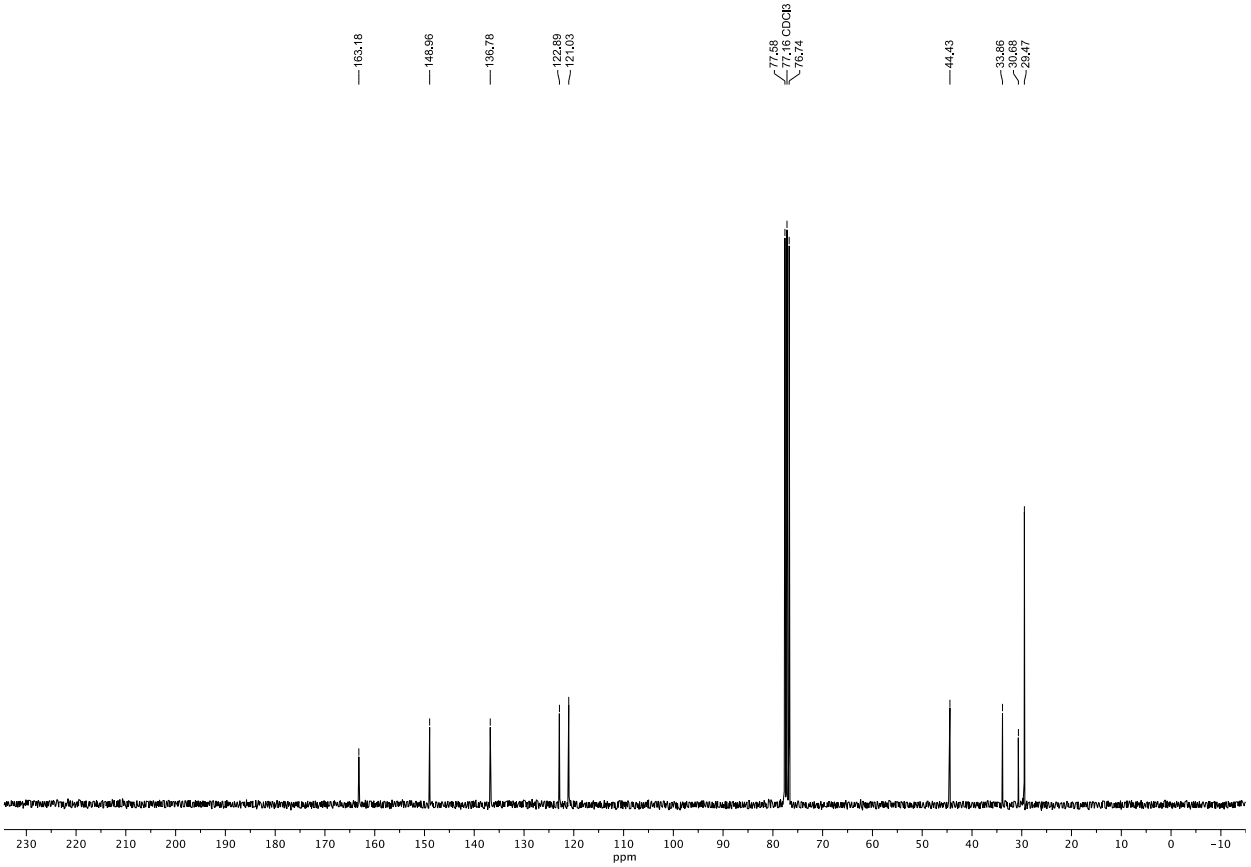


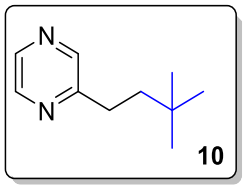
^{1}H-NMR (300 MHz, chloroform-*d*)



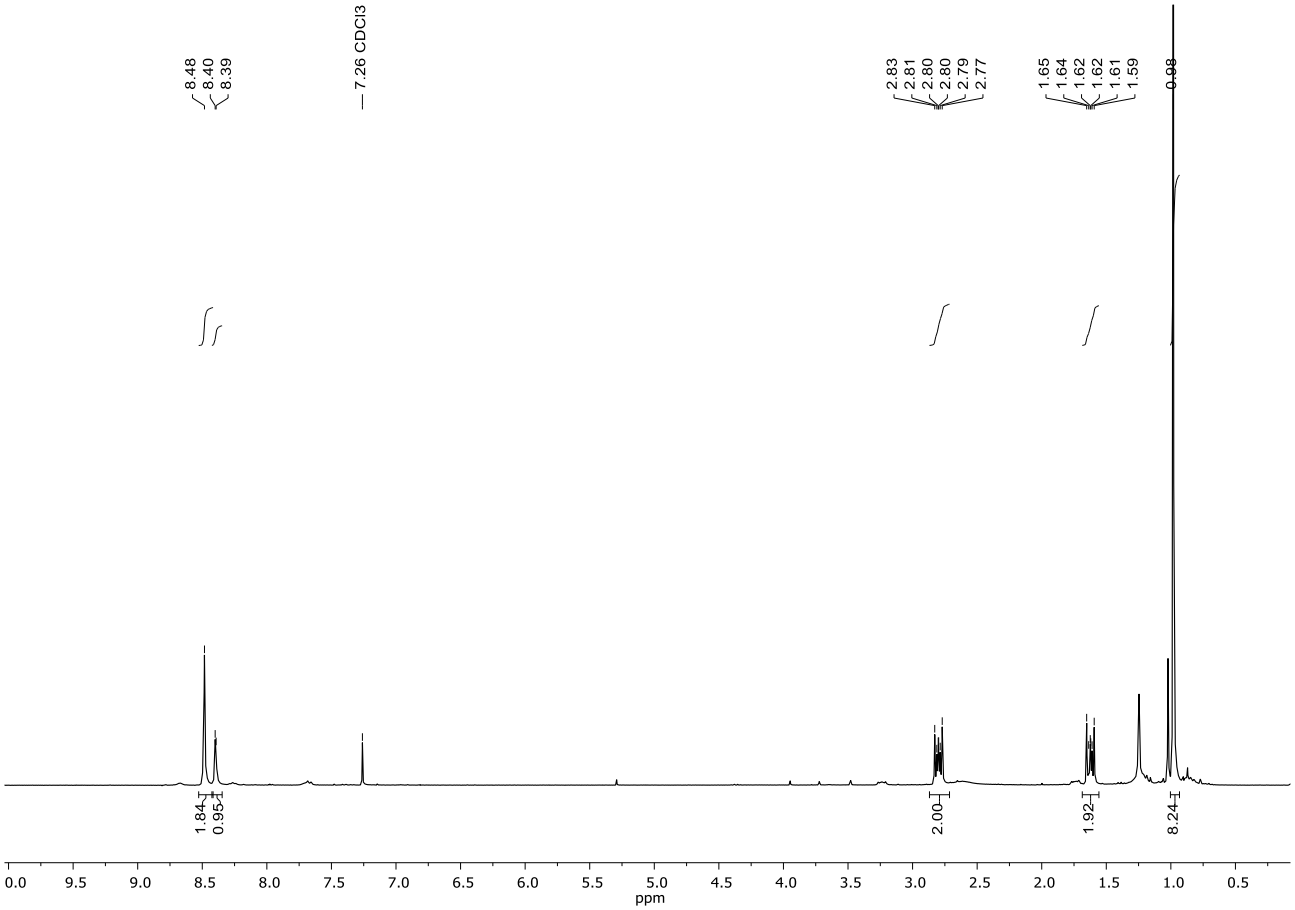


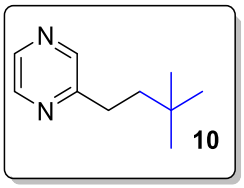
^{13}C-NMR (75 MHz, chloroform-*d*)



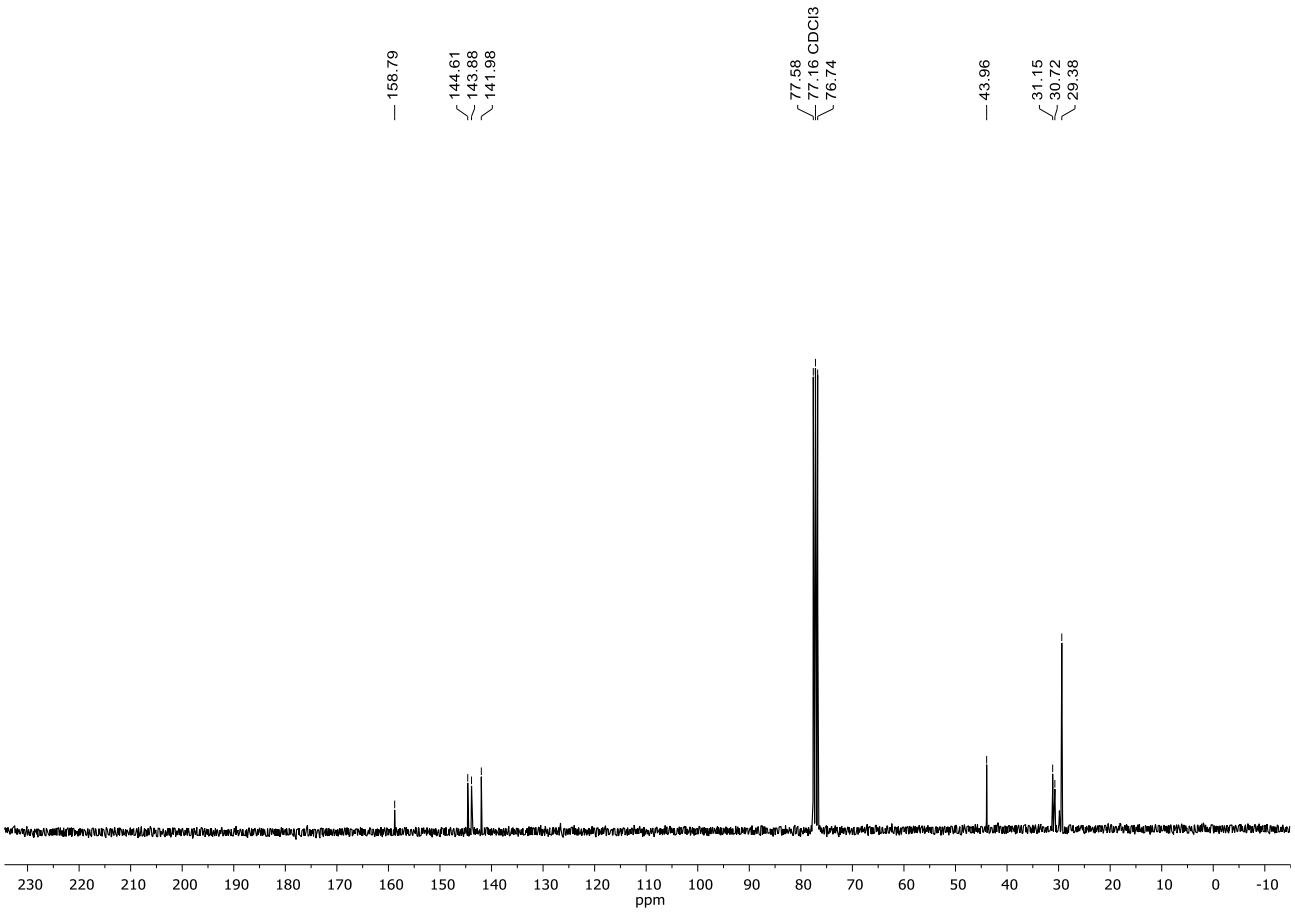


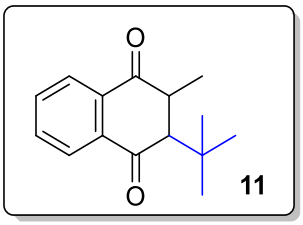
^{1}H-NMR (300 MHz, chloroform-*d*)



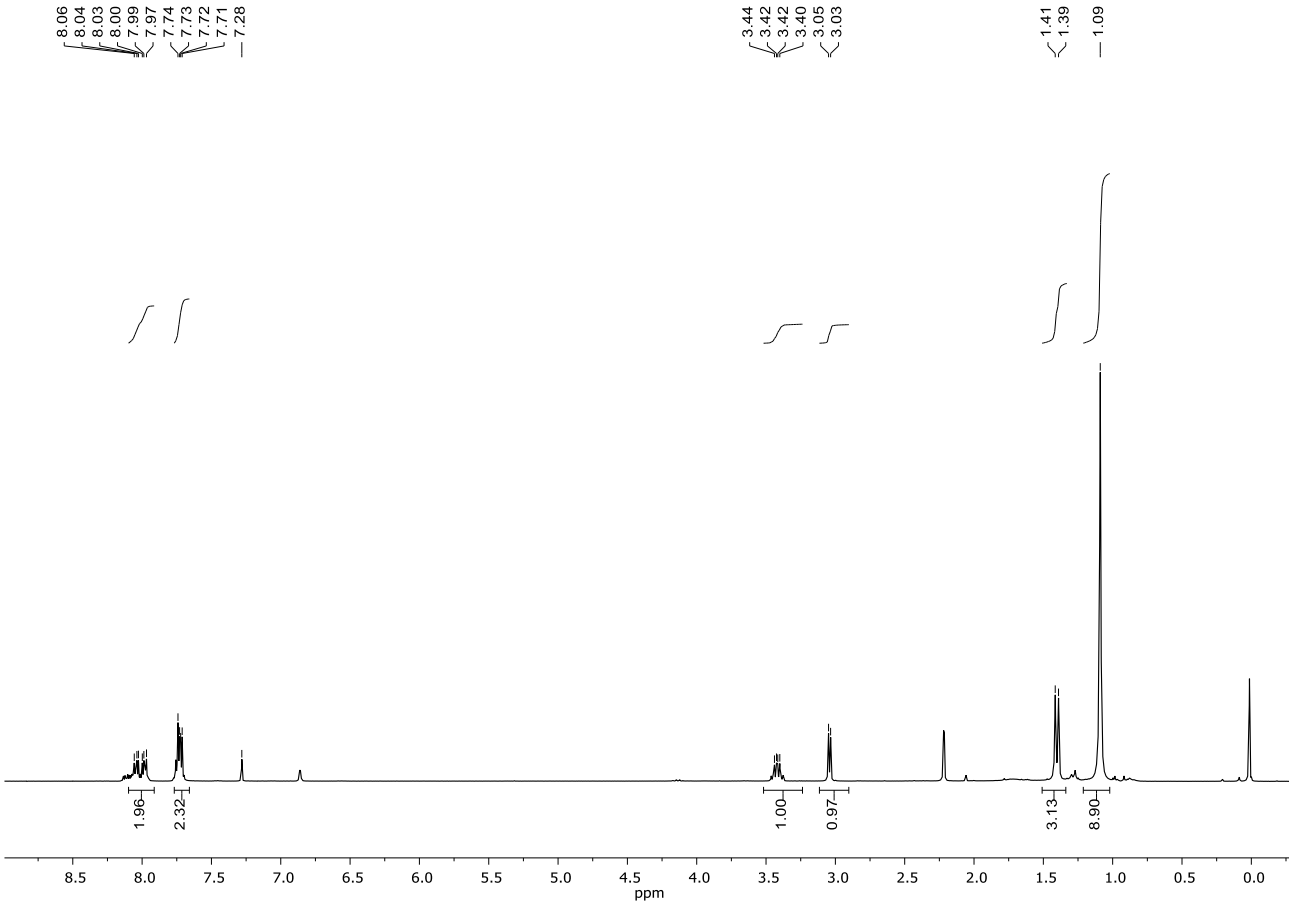


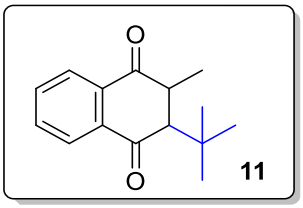
^{13}C-NMR (75 MHz, chloroform-*d*)



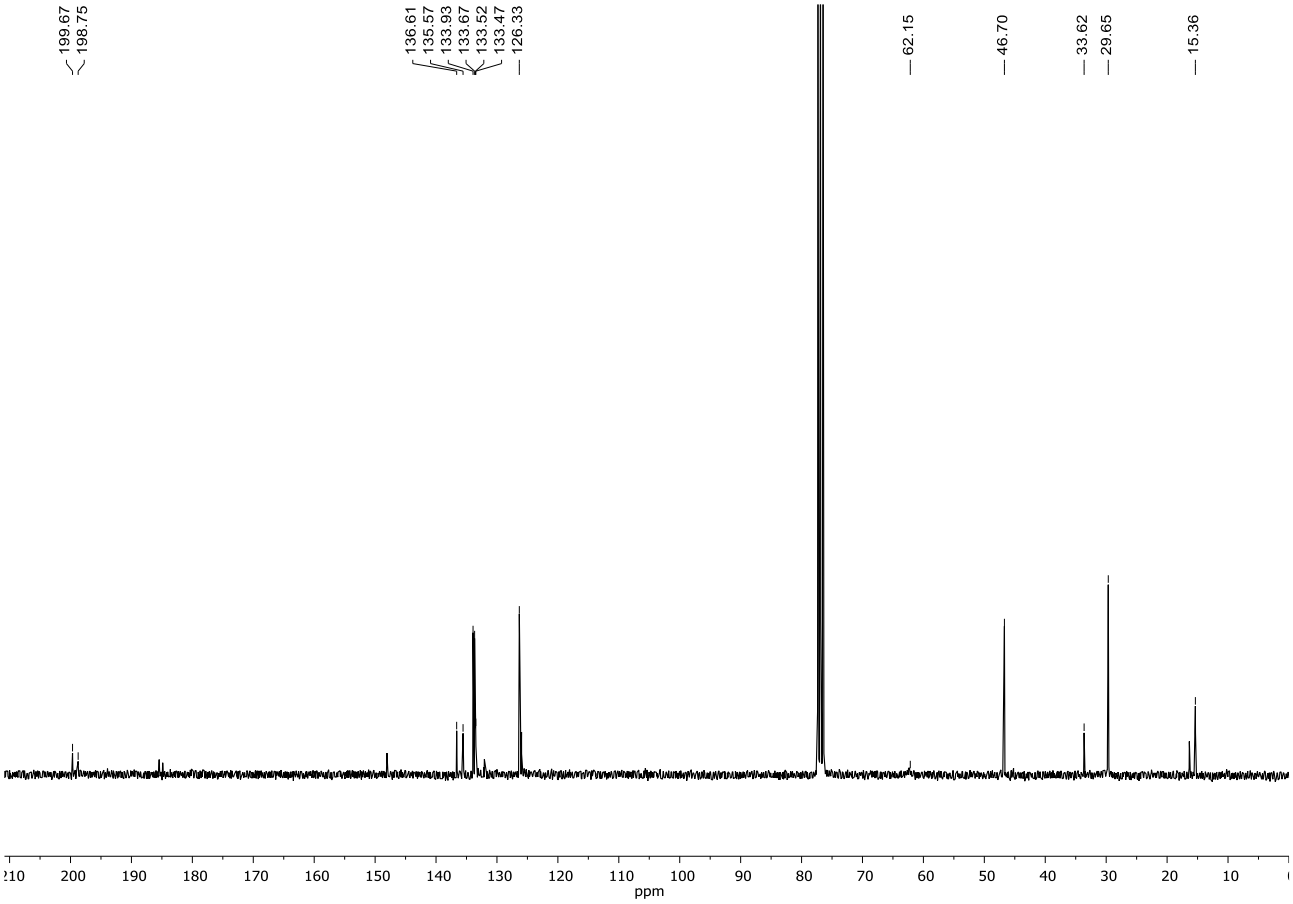


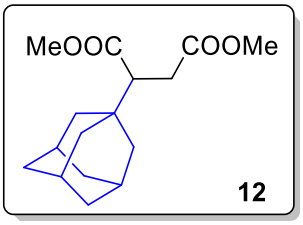
^{1}H-NMR (300 MHz, chloroform-*d*)



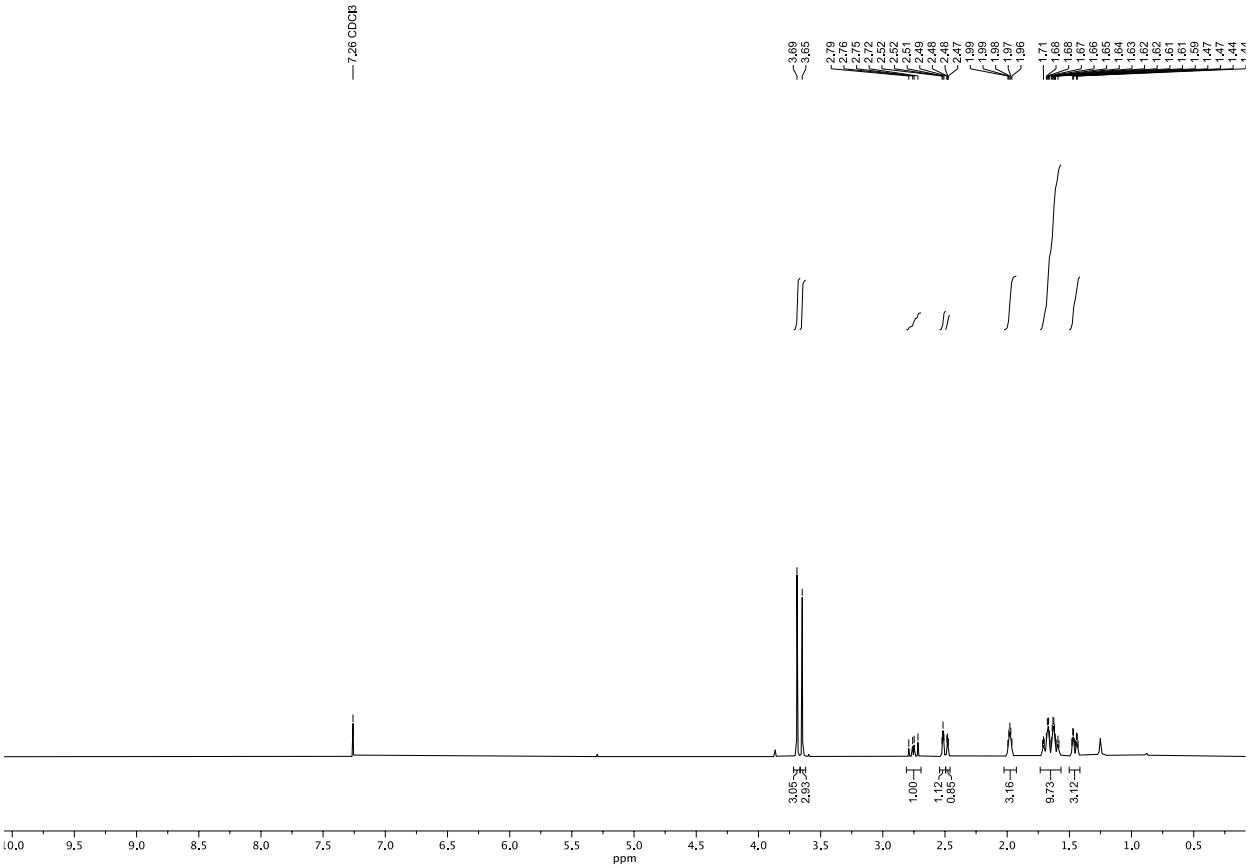


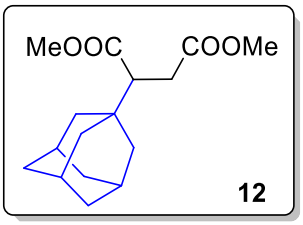
^{13}C-NMR (75 MHz, chloroform-*d*)



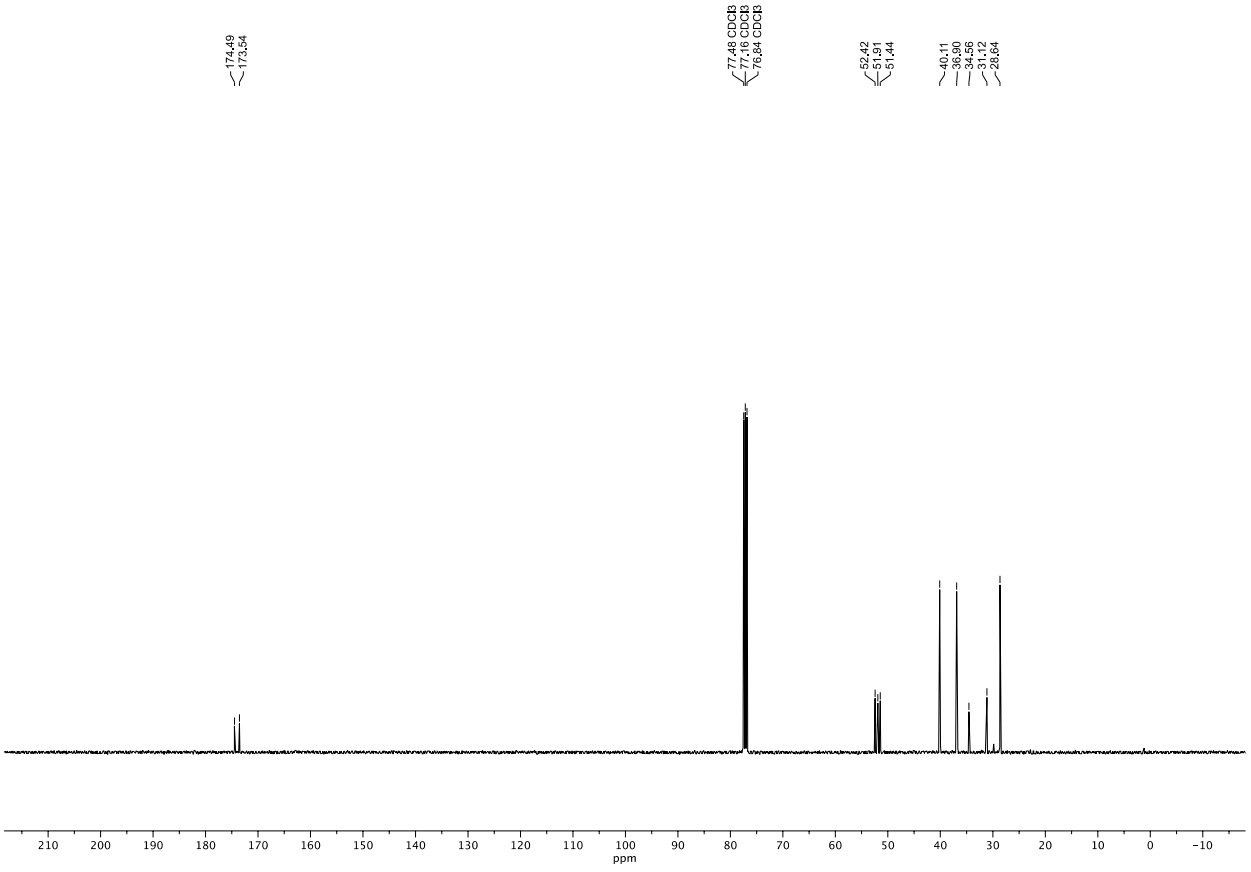


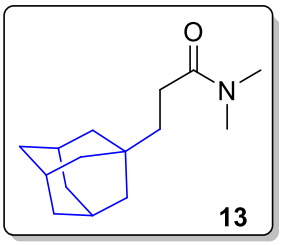
^{1}H-NMR (400 MHz, chloroform-*d*)



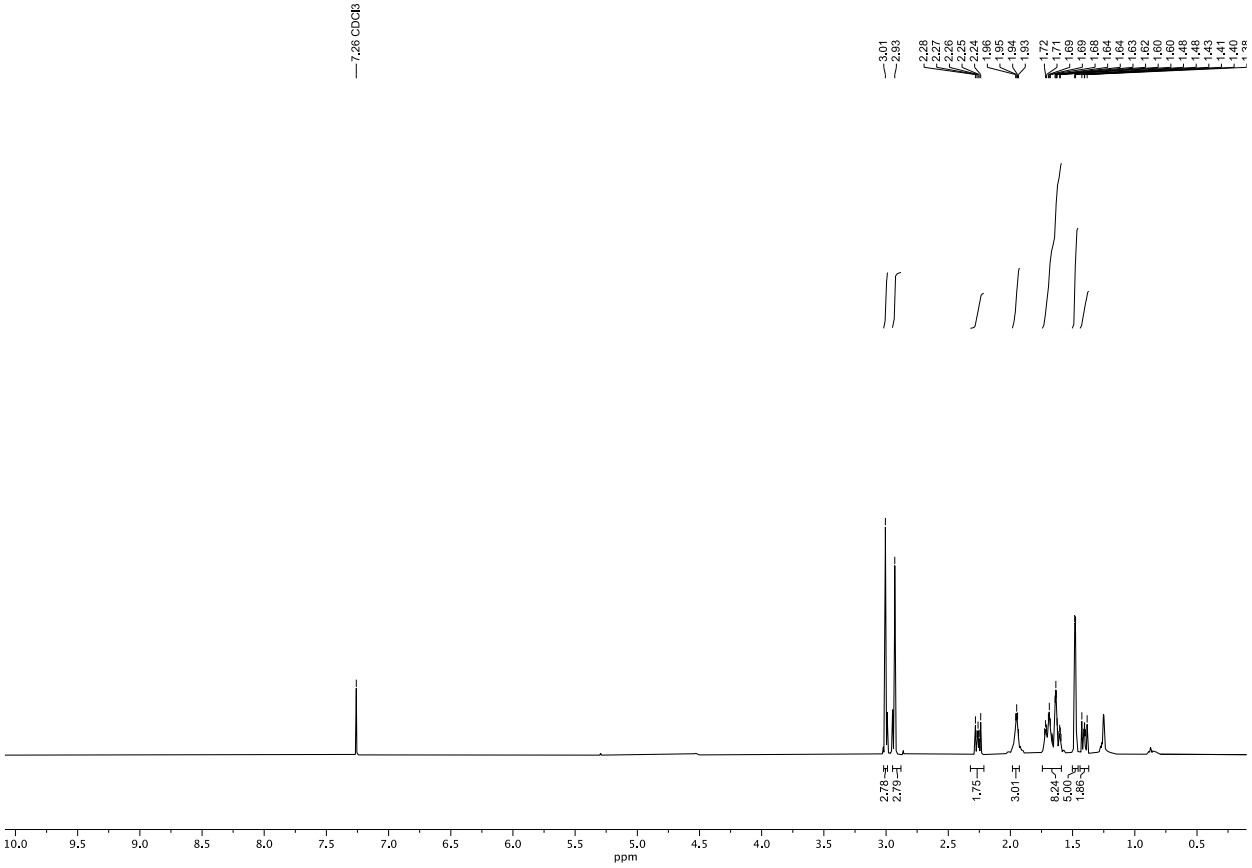


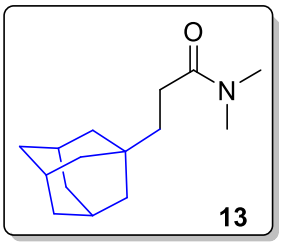
^{13}C-NMR (100 MHz, chloroform-*d*)



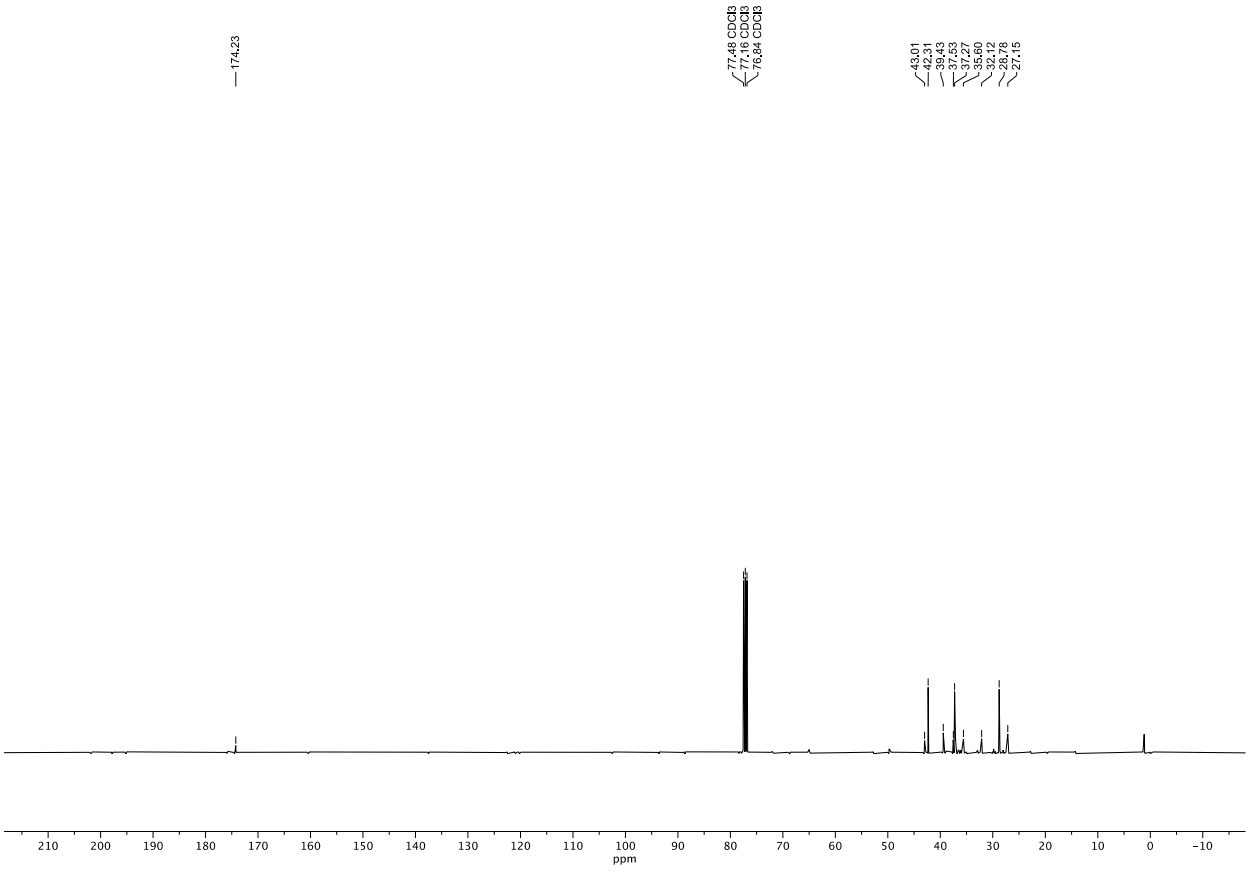


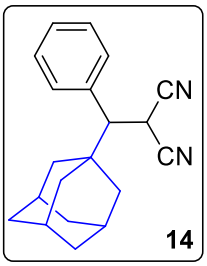
^{1}H-NMR (400 MHz, chloroform-*d*)



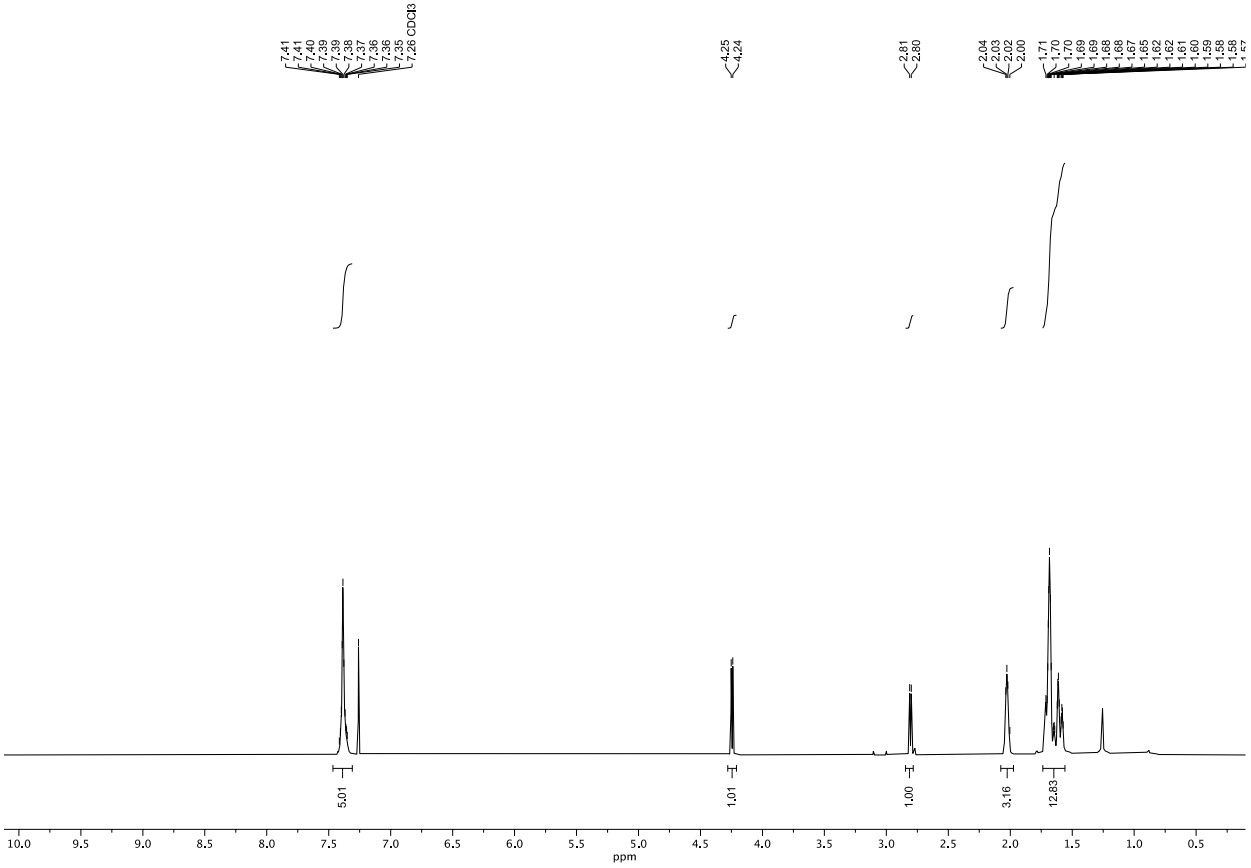


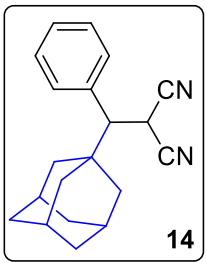
^{13}C-NMR (100 MHz, chloroform-*d*)



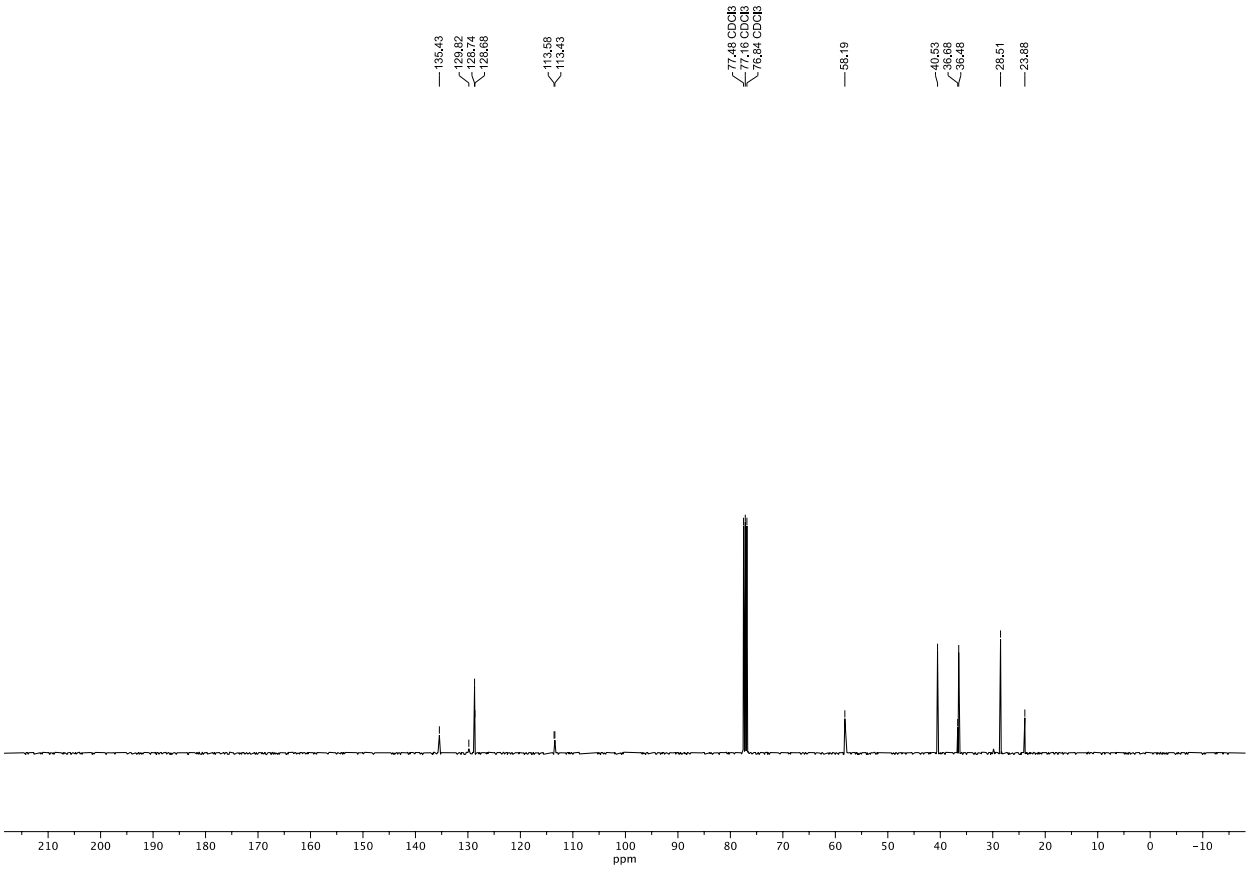


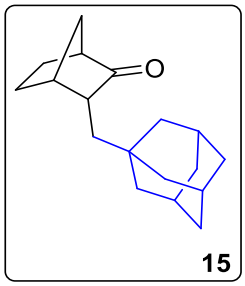
^{1}H-NMR (400 MHz, chloroform-*d*)



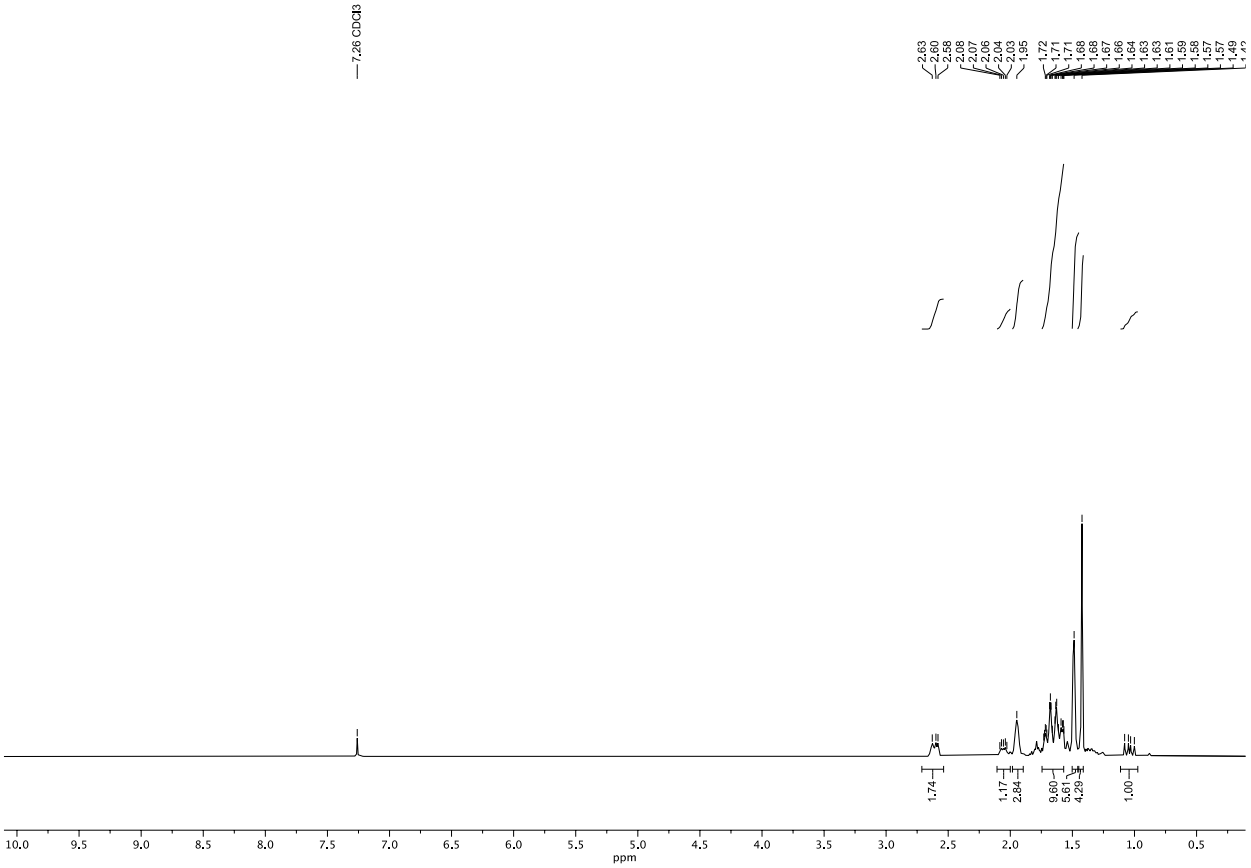


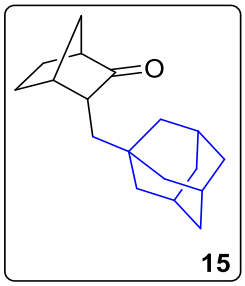
^{13}C-NMR (100 MHz, chloroform-*d*)



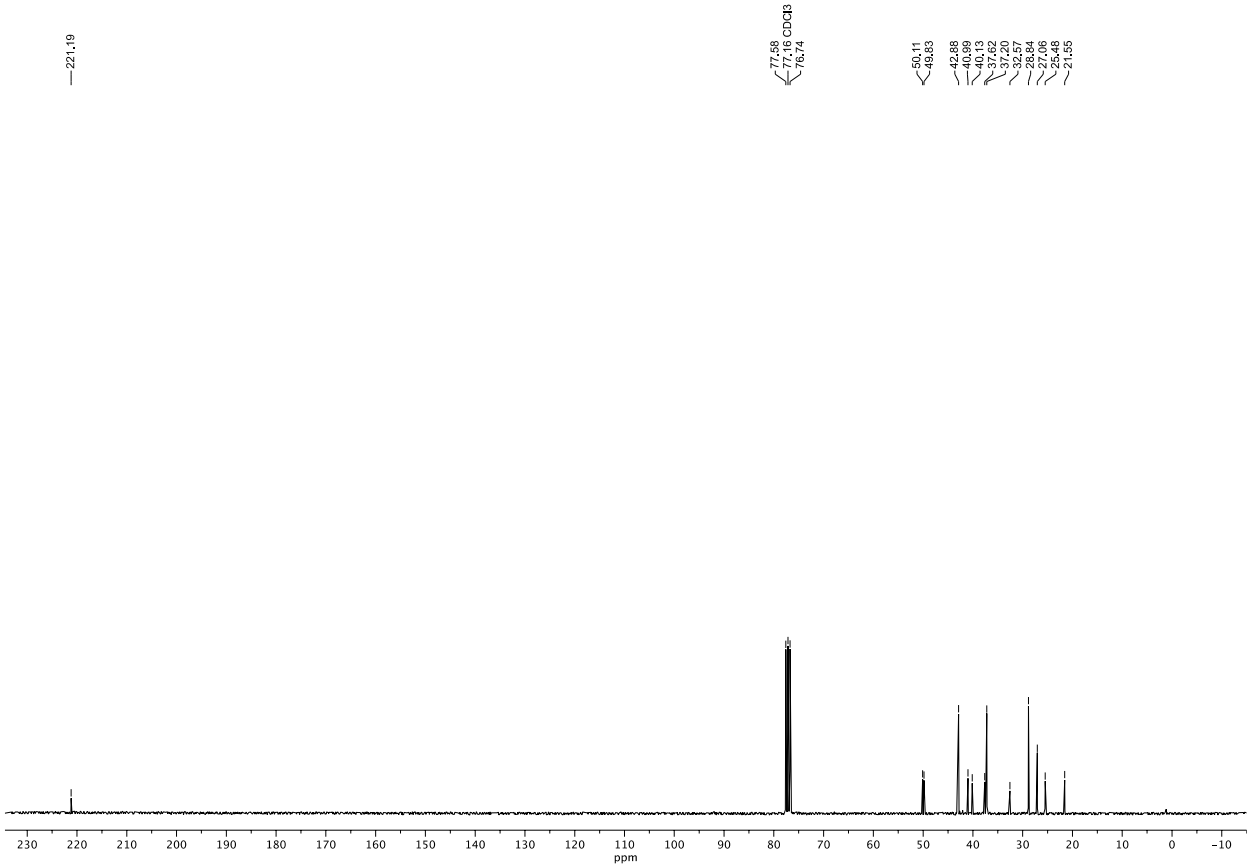


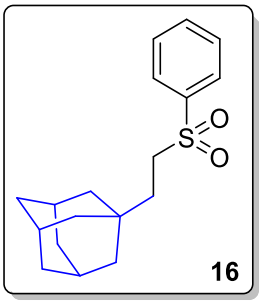
^{1}H-NMR (300 MHz, chloroform-*d*)



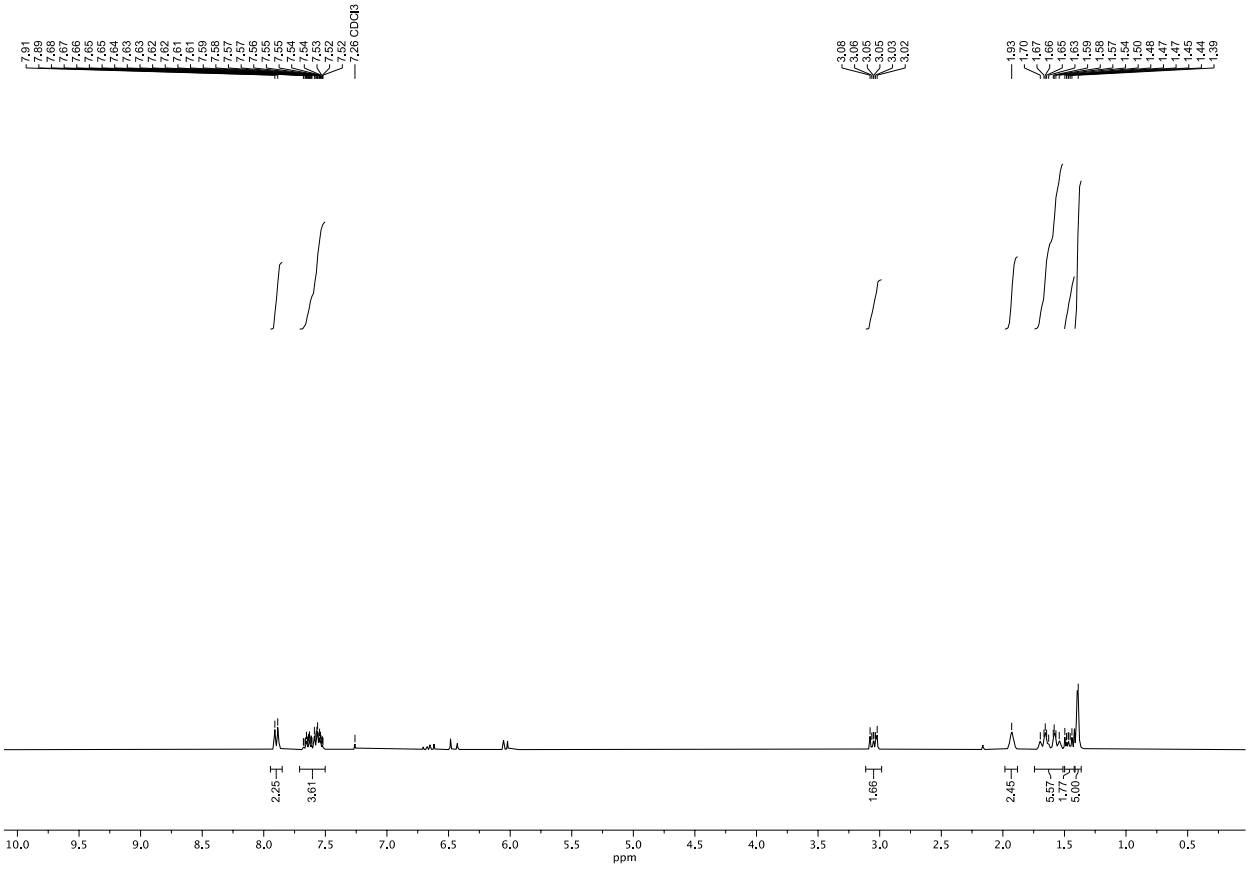


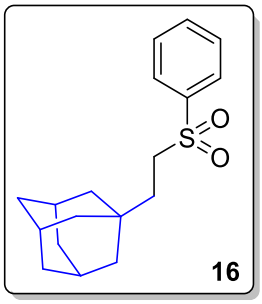
^{13}C-NMR (75 MHz, chloroform-*d*)



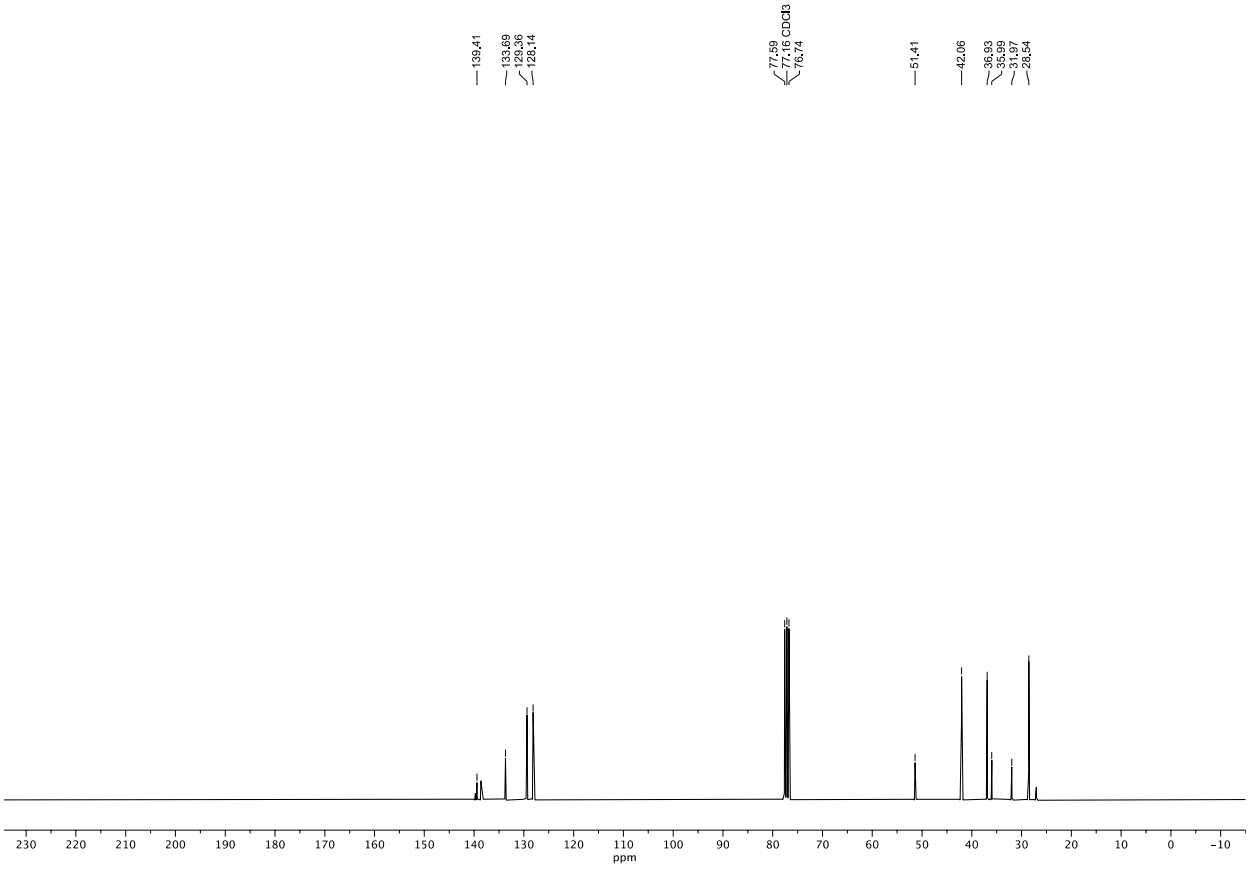


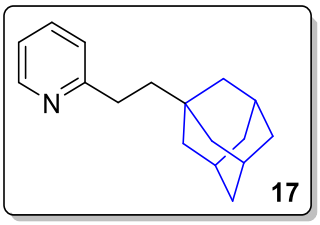
^{1}H-NMR (300 MHz, chloroform-*d*)



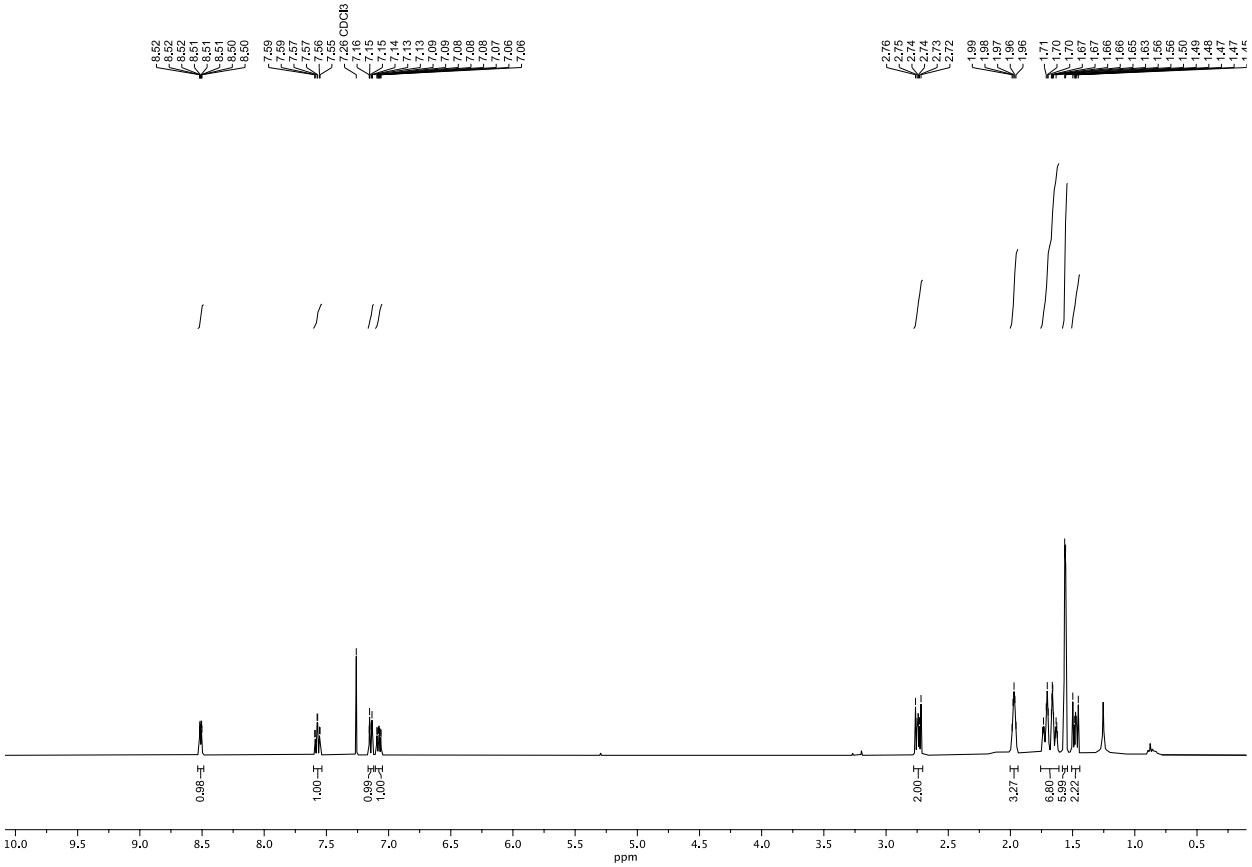


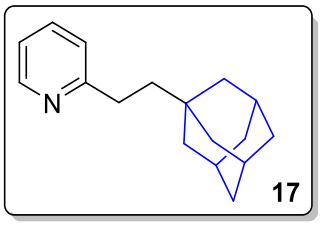
^{13}C-NMR (75 MHz, chloroform-*d*)



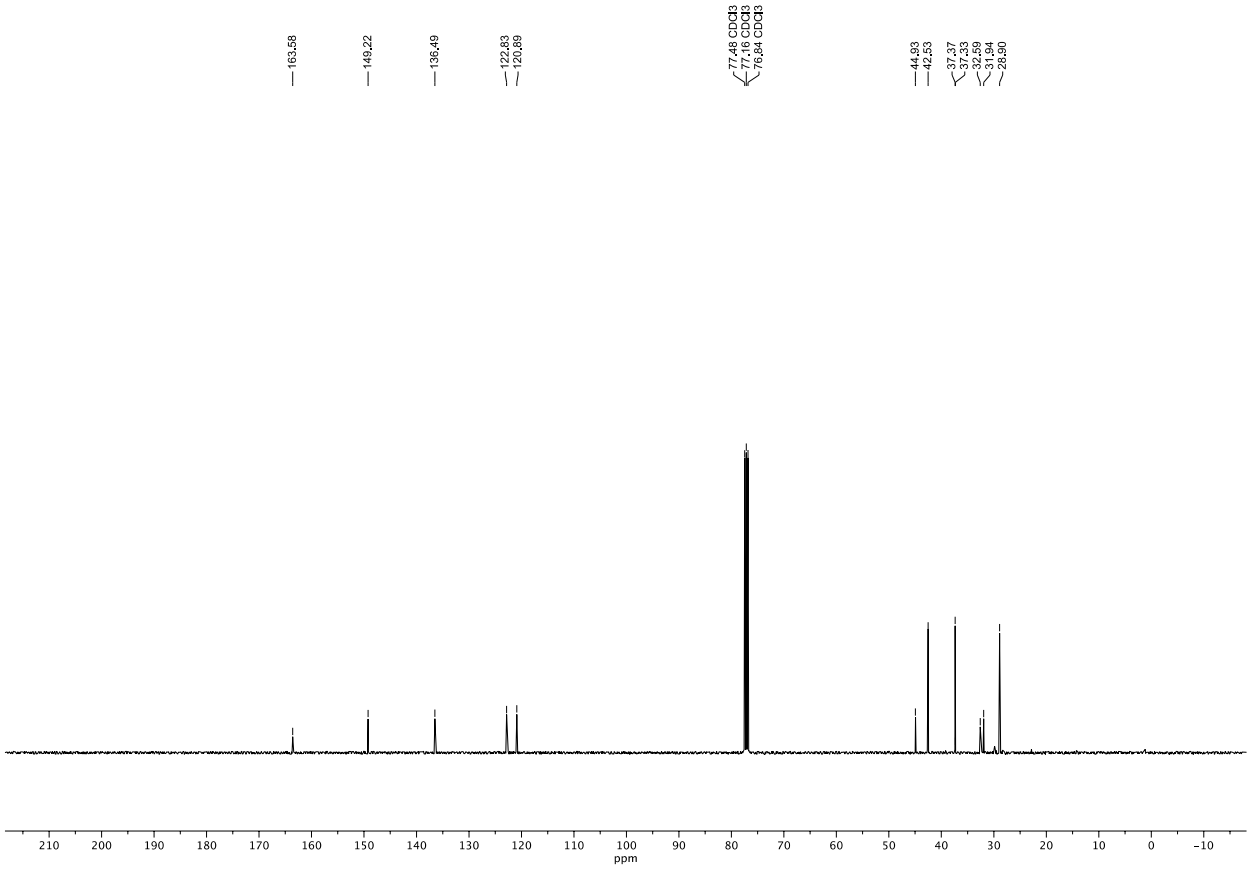


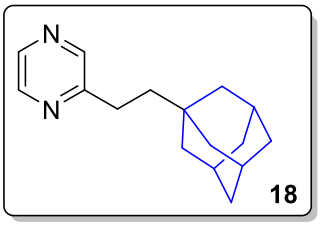
^{1}H-NMR (400 MHz, chloroform-*d*)



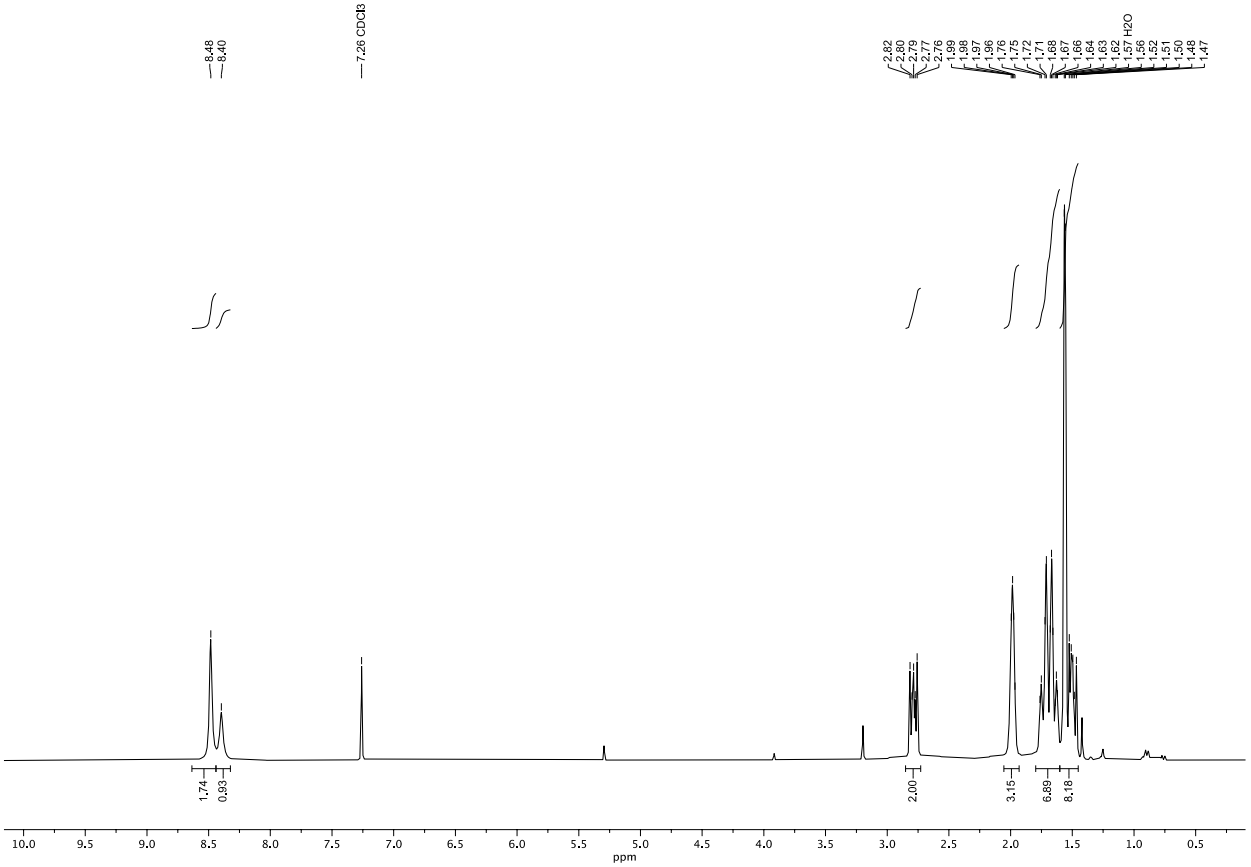


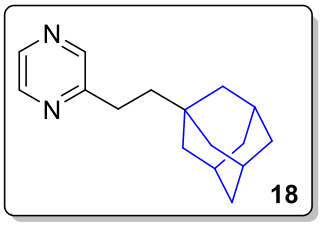
^{13}C-NMR (100 MHz, chloroform-*d*)



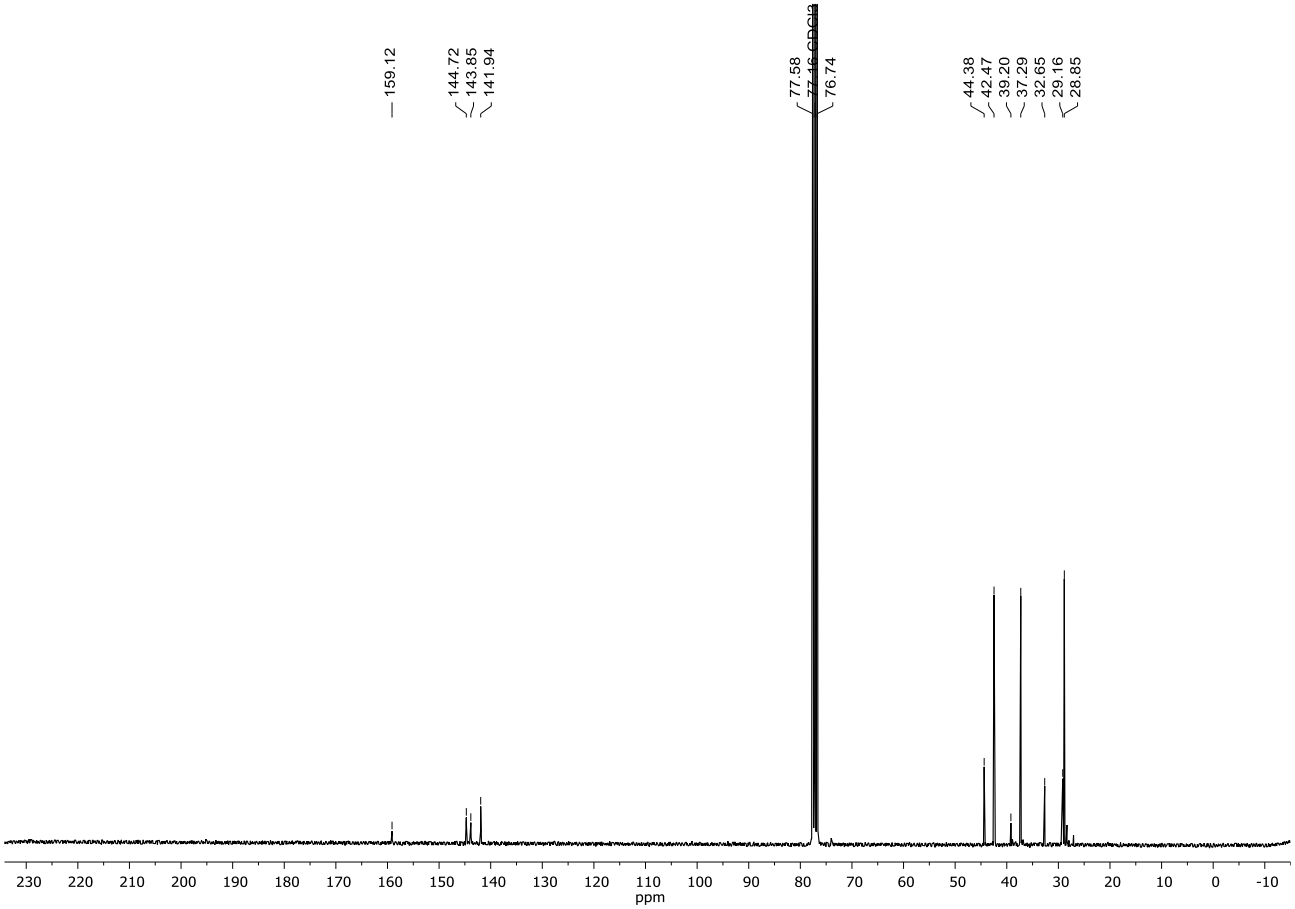


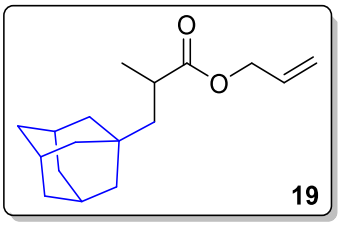
^{1}H-NMR (300 MHz, chloroform-*d*)



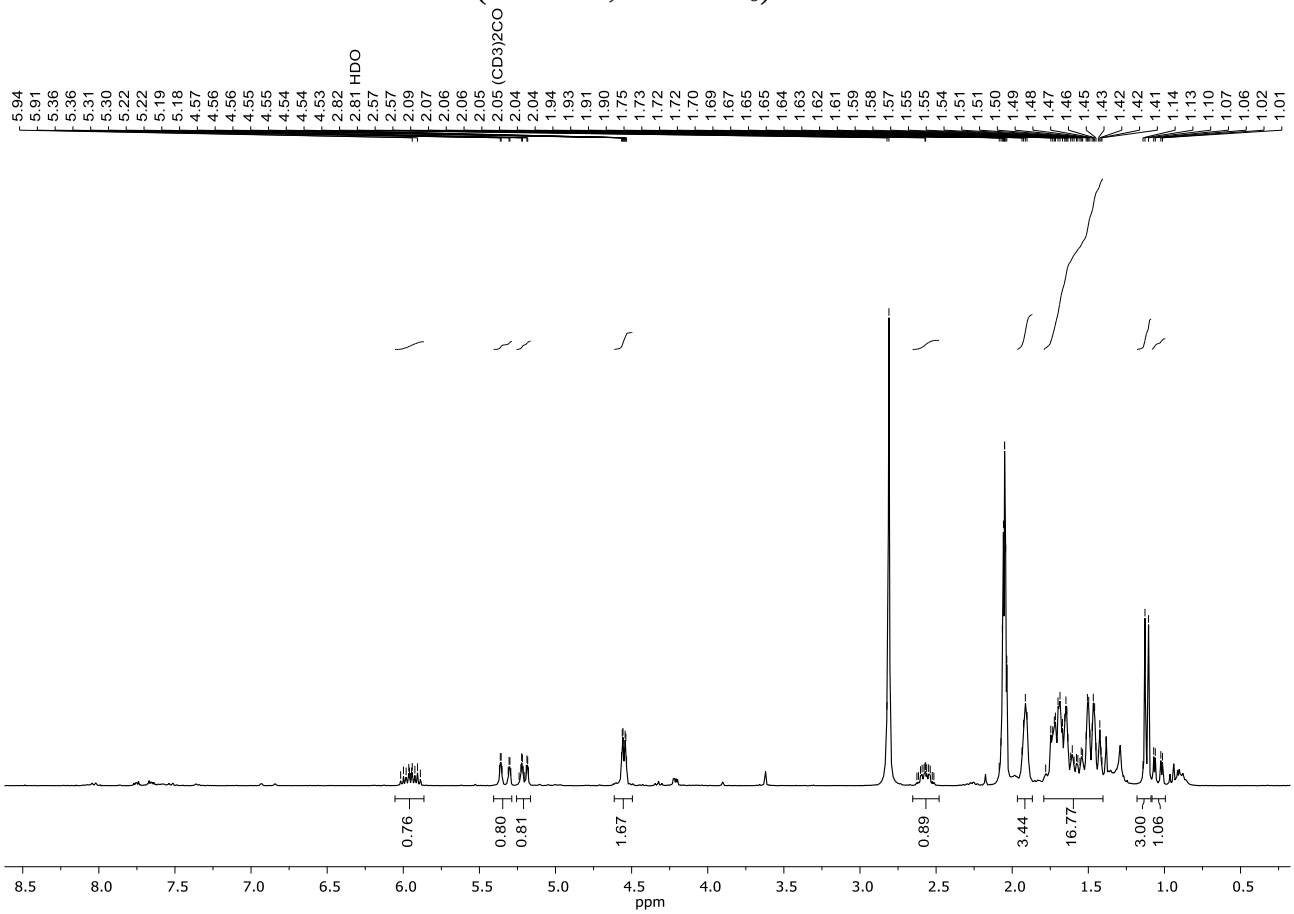


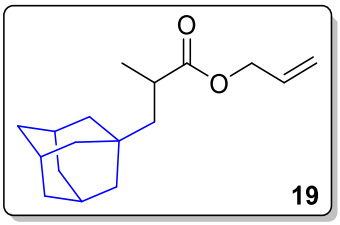
^{13}C-NMR (75 MHz, chloroform-*d*)



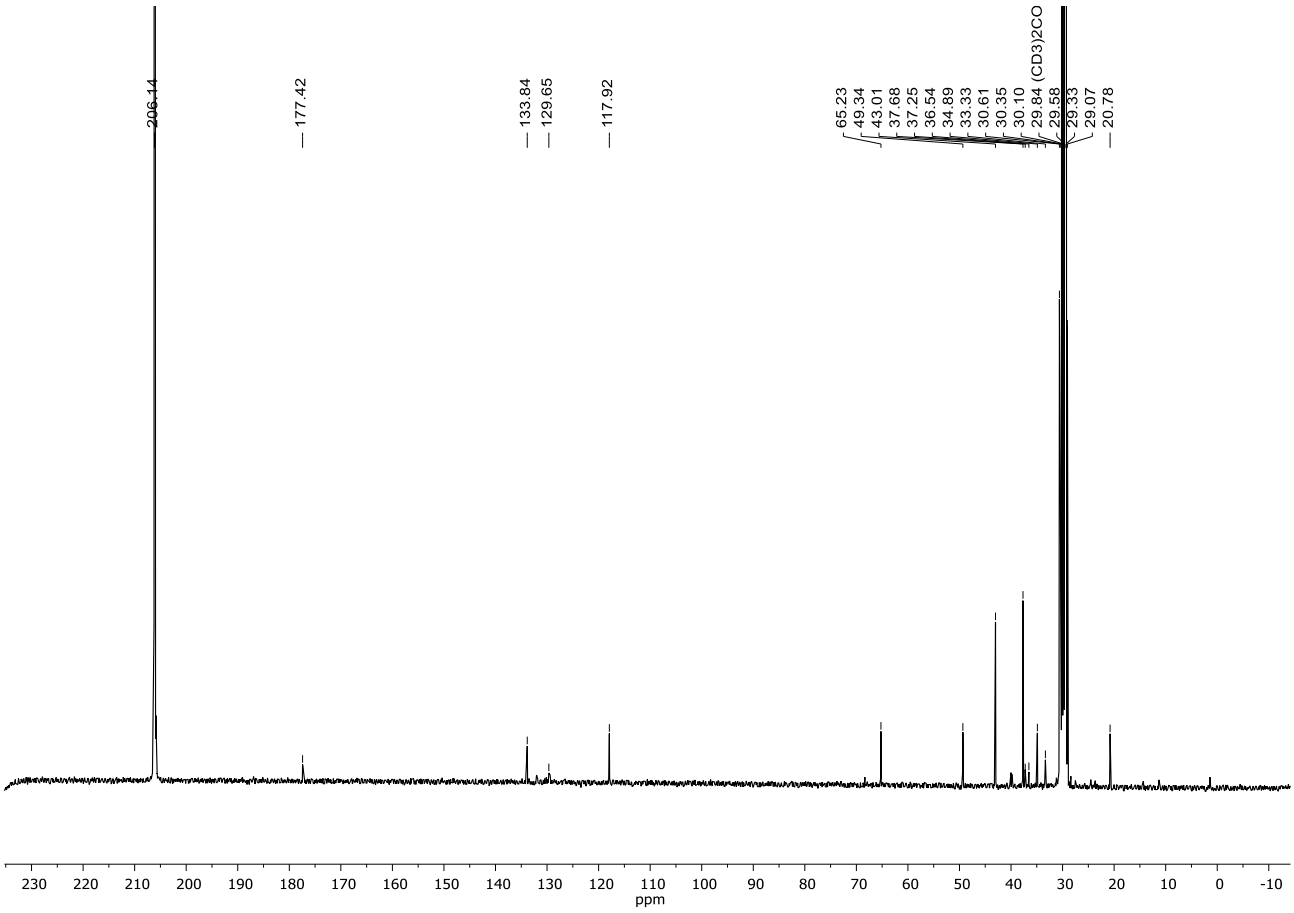


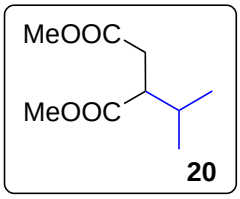
^{1}H-NMR (400 MHz, acetone-d_6)



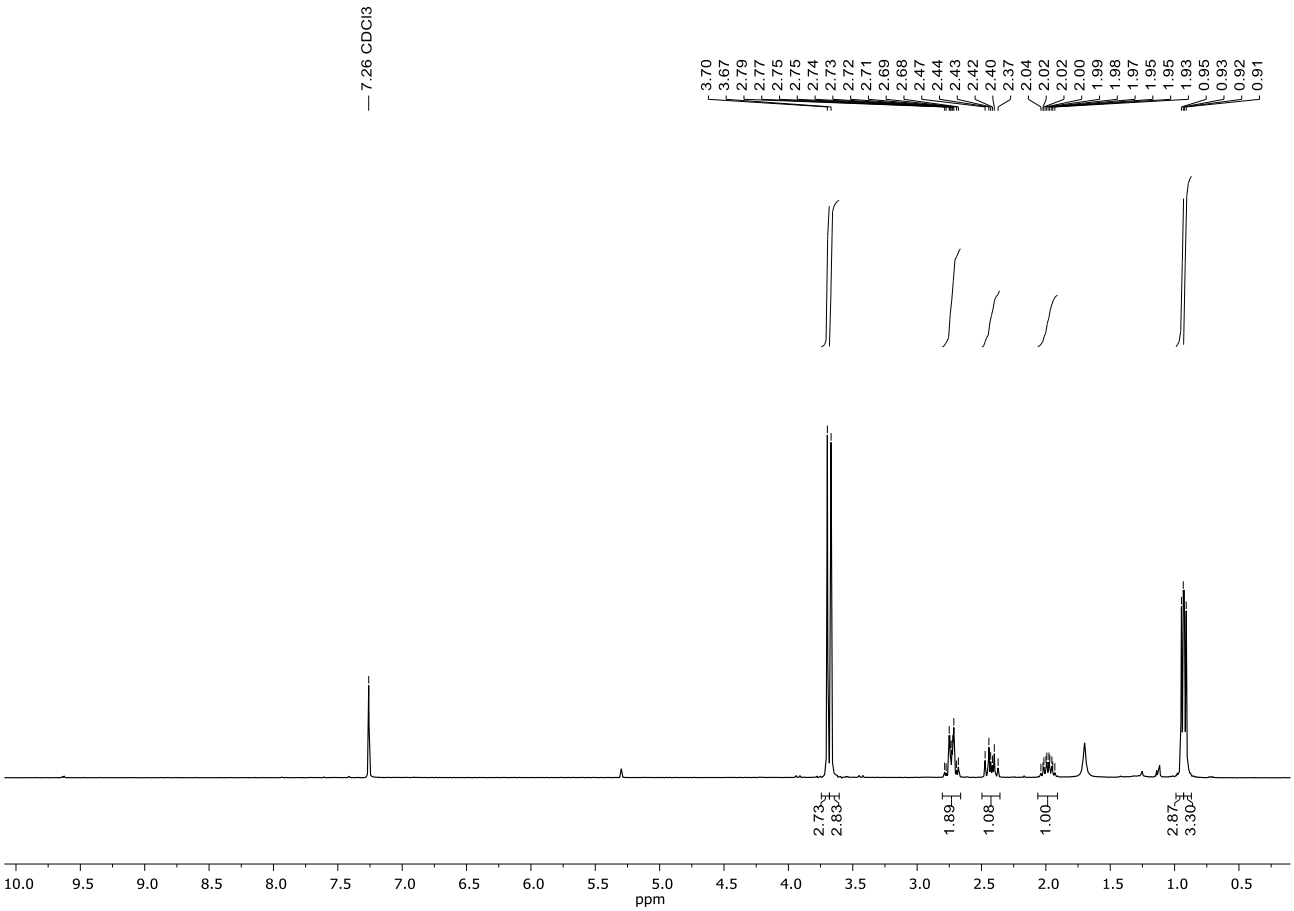


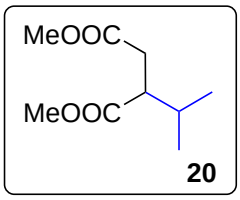
^{13}C-NMR (75 MHz, acetone-d_6)



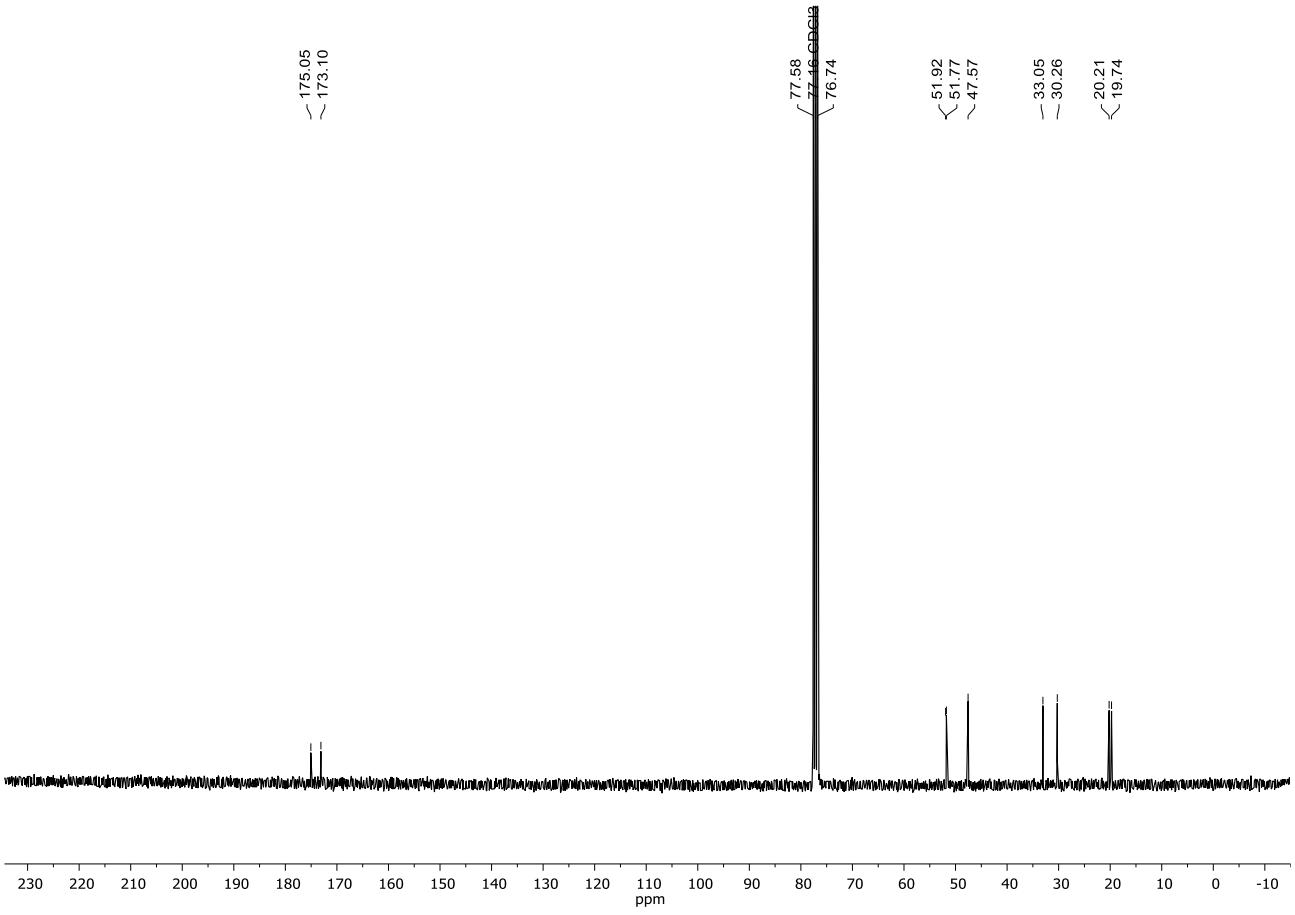


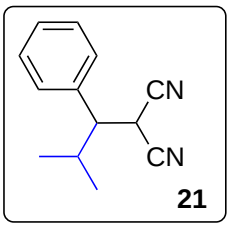
^{1}H-NMR (300 MHz, chloroform-*d*)



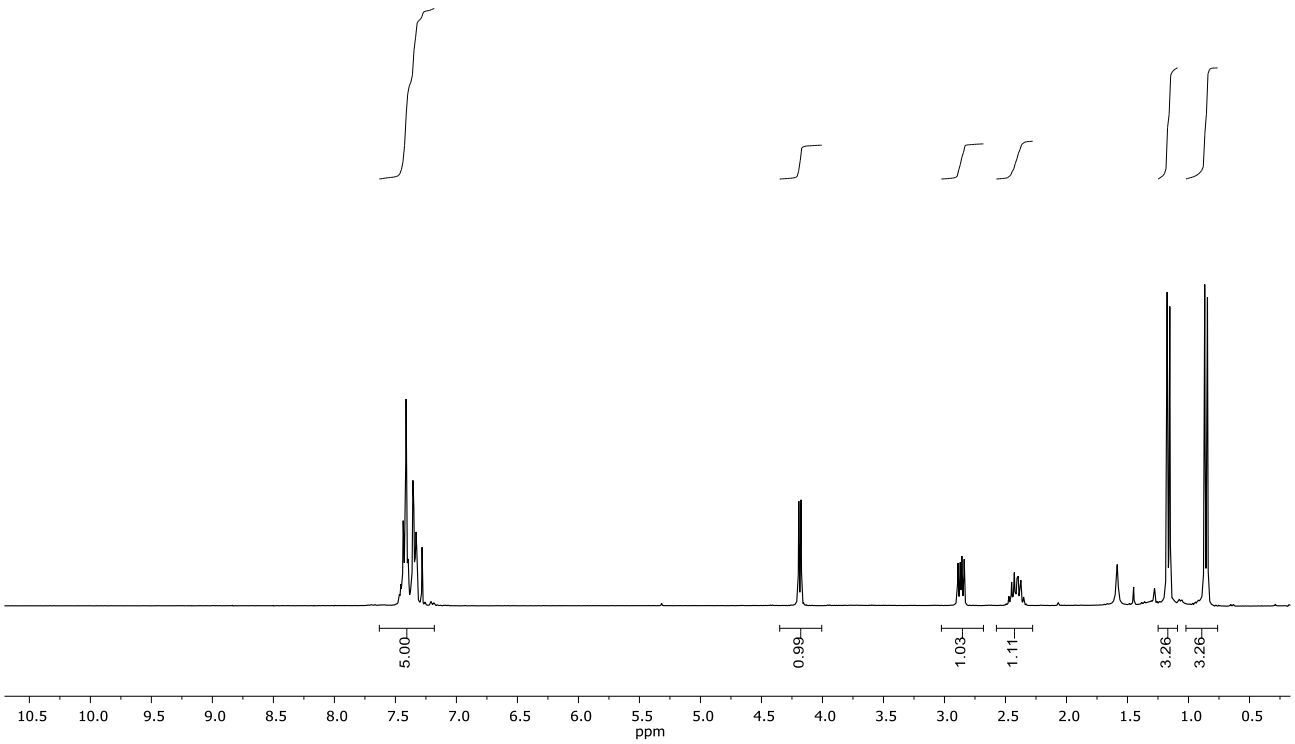


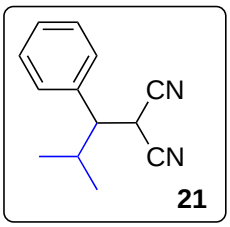
^{13}C-NMR (75 MHz, chloroform-*d*)



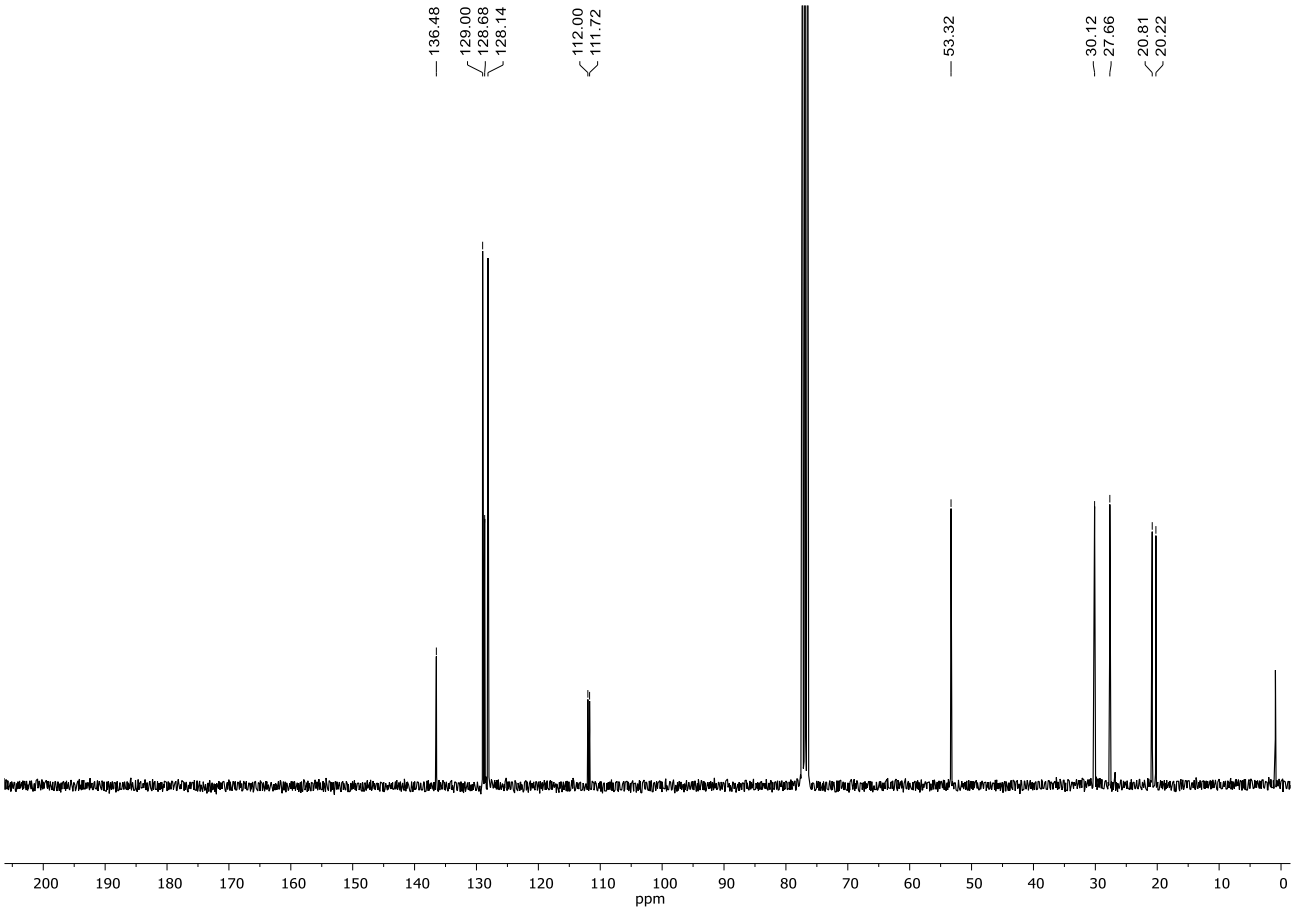


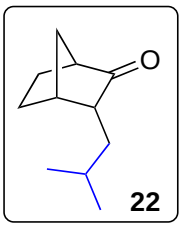
^{1}H-NMR (300 MHz, chloroform-*d*)



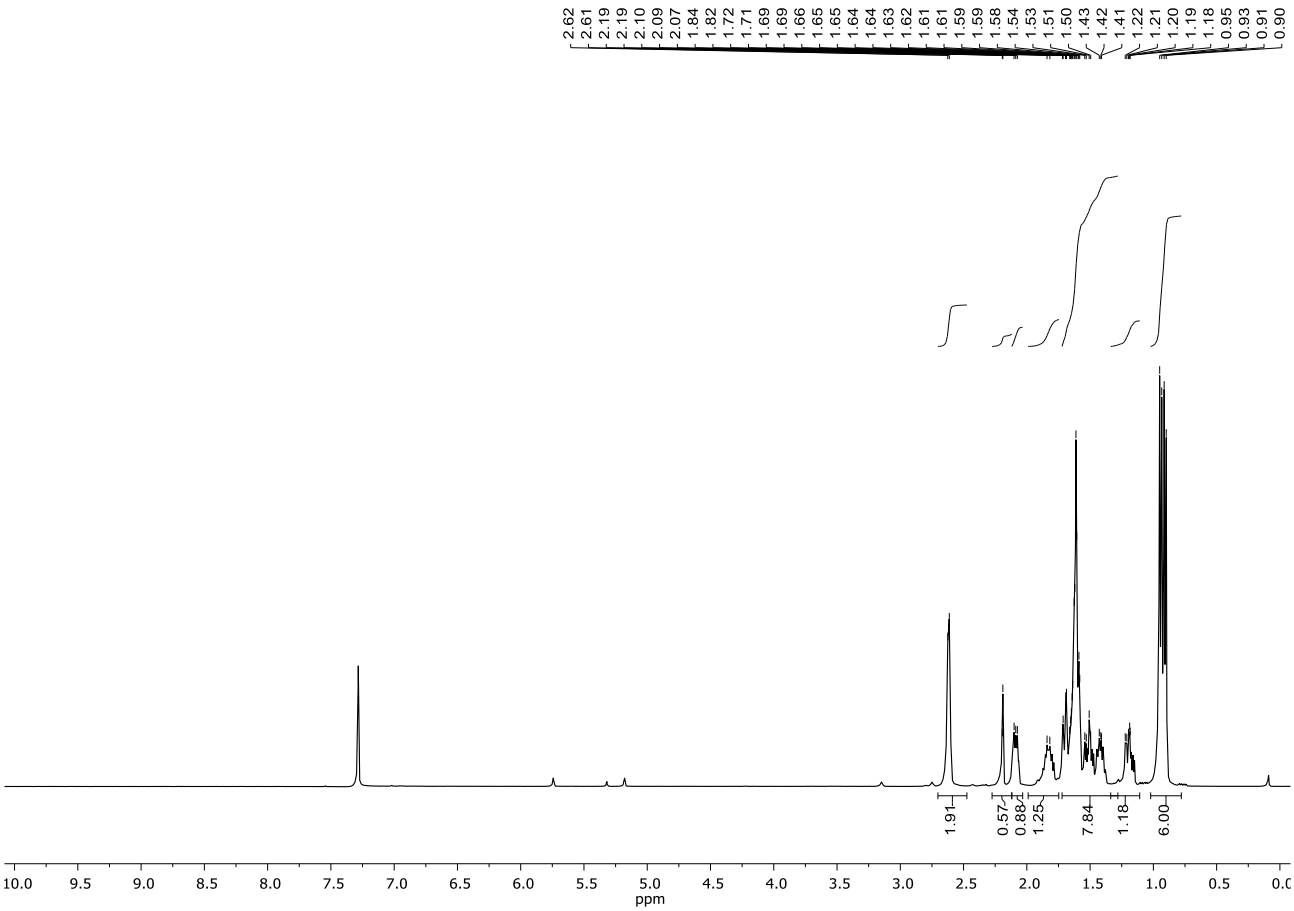


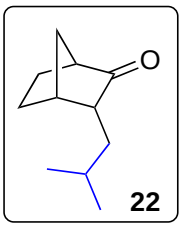
^{13}C-NMR (75MHz, chloroform-*d*)



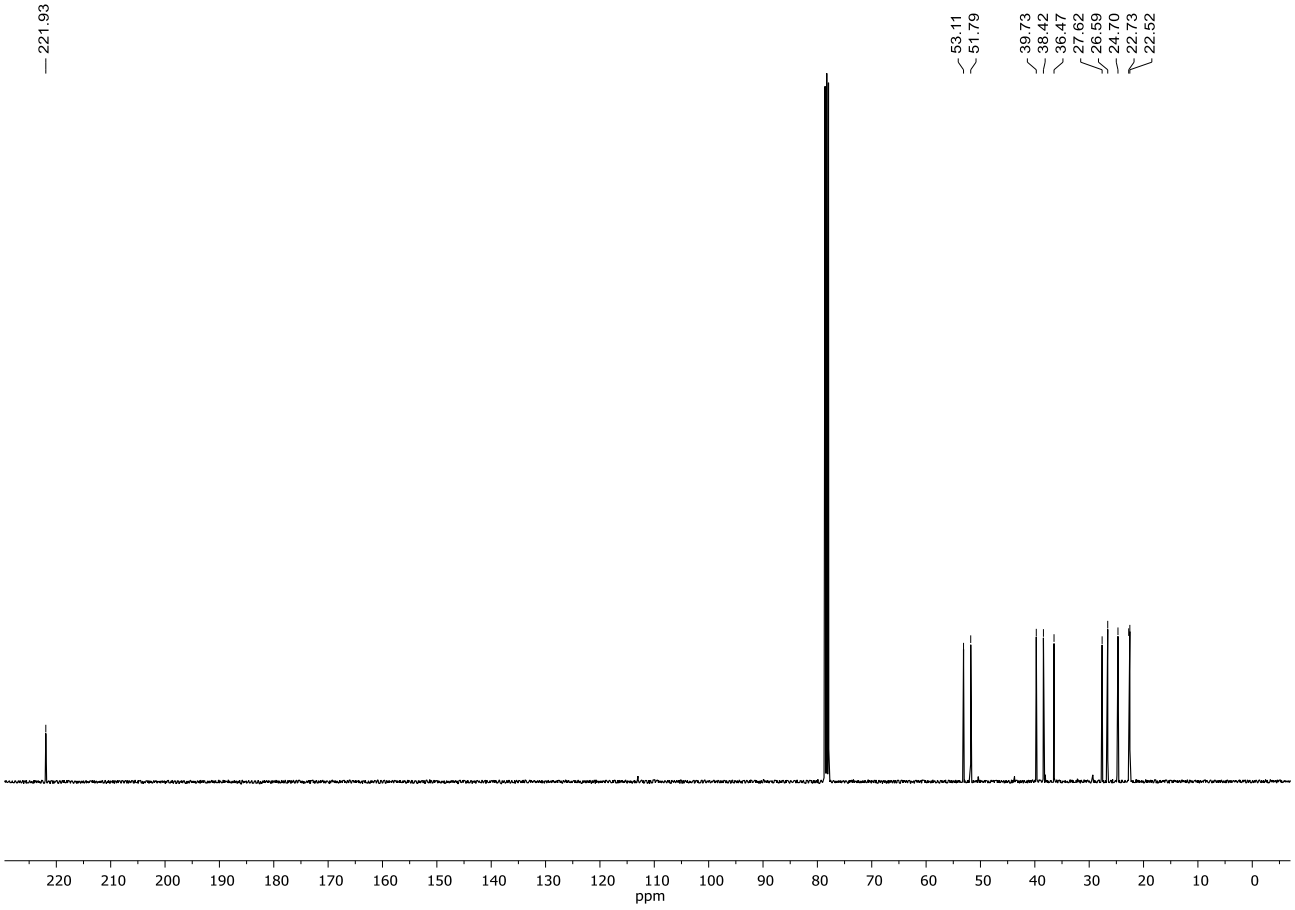


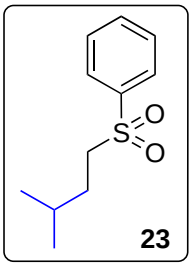
^{1}H-NMR (300 MHz, chloroform-*d*)



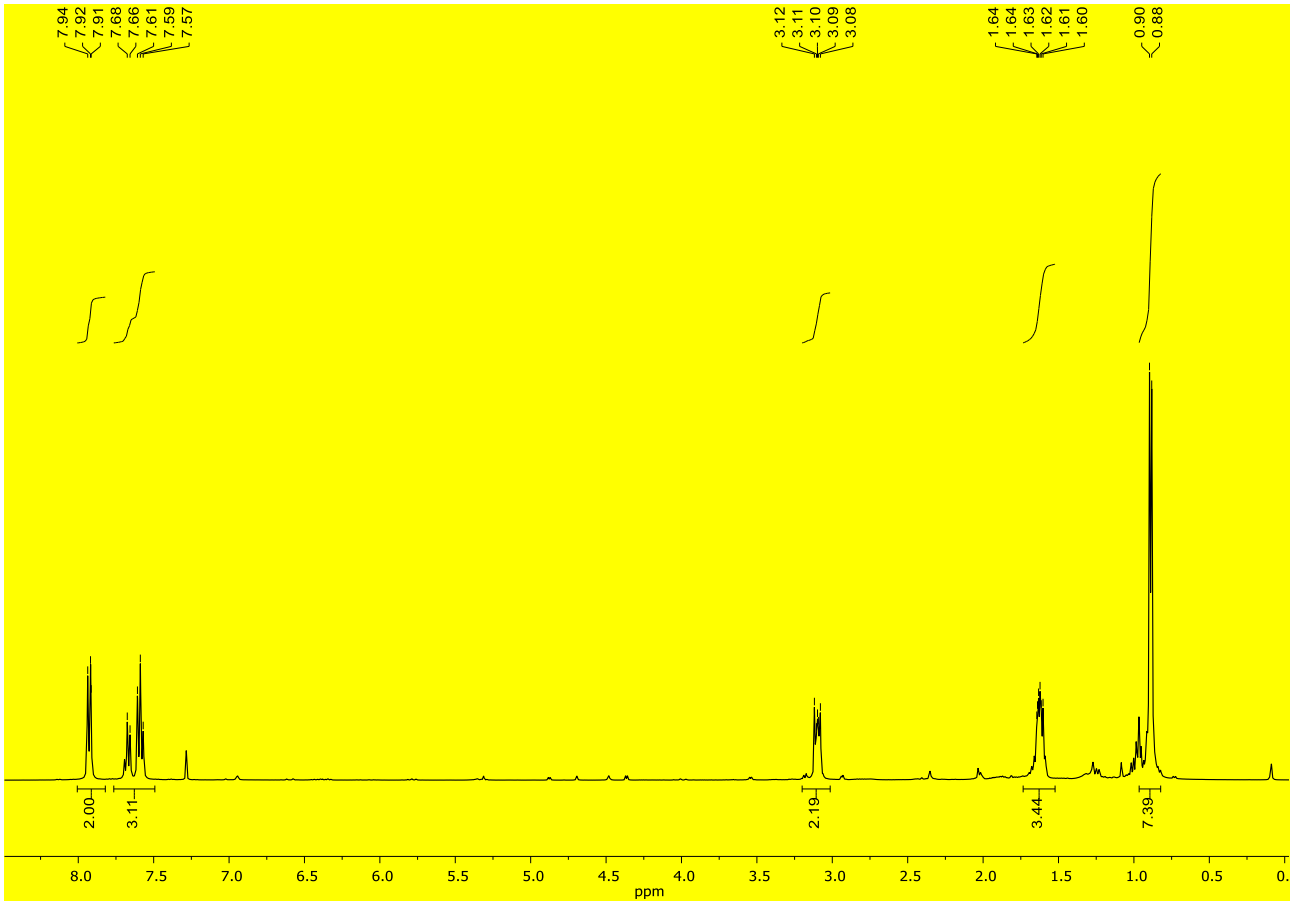


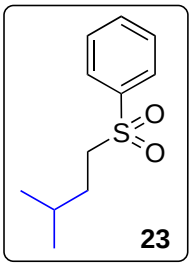
^{13}C-NMR (75 MHz, chloroform-*d*)



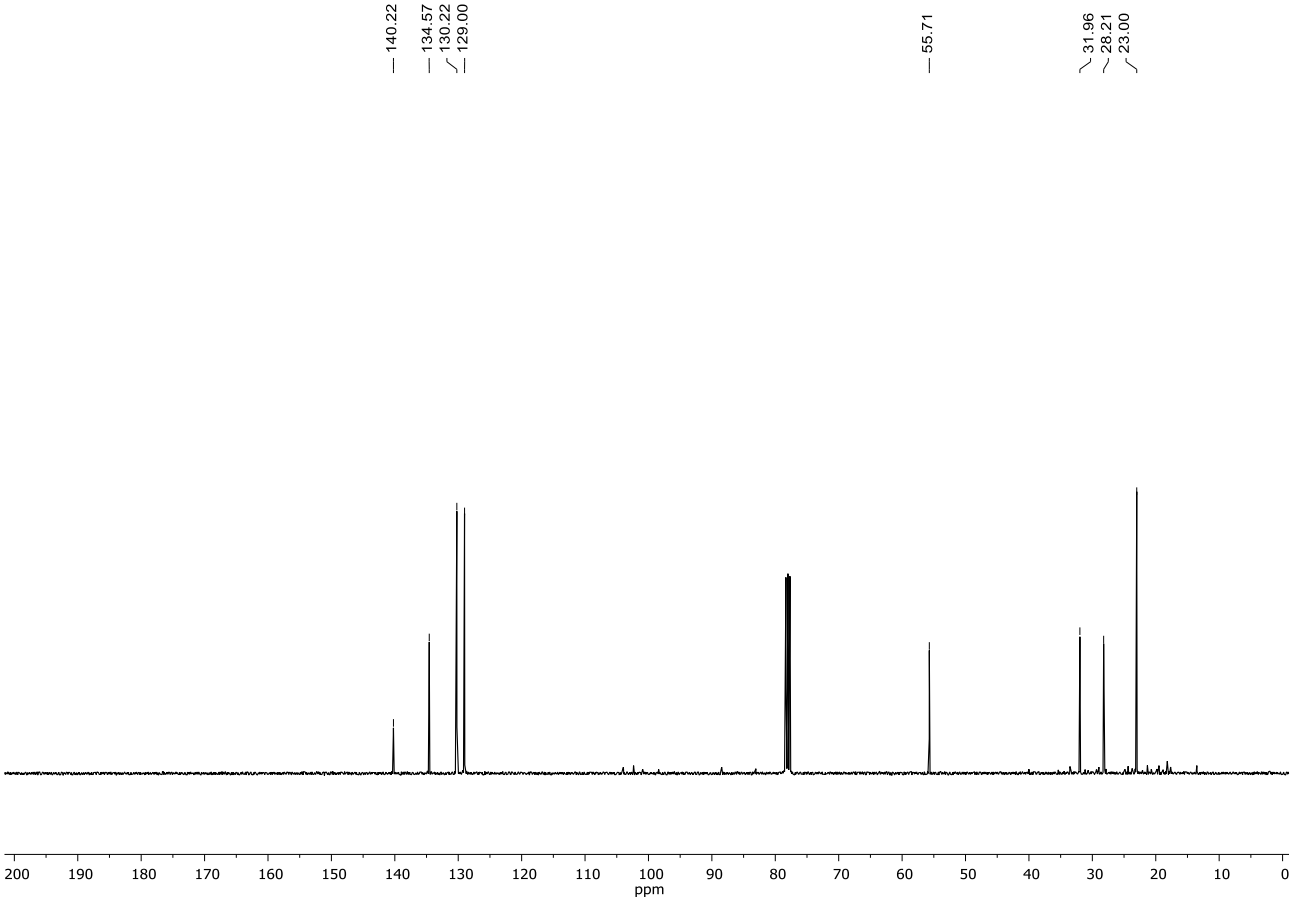


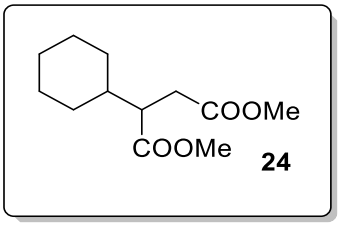
^{1}H-NMR (300 MHz, chloroform-*d*)



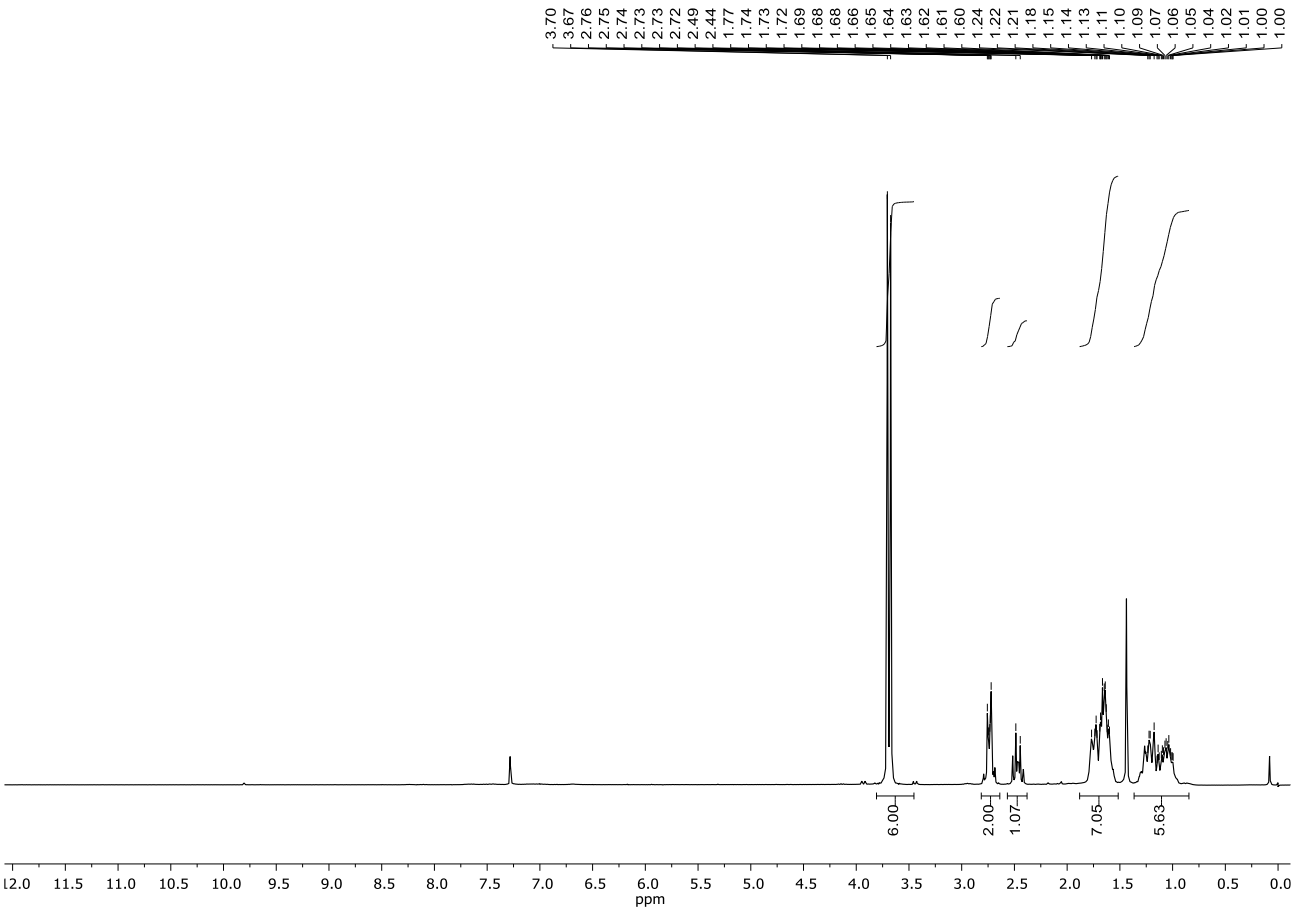


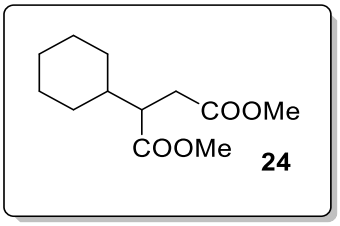
^{13}C-NMR (75 MHz, chloroform-*d*)



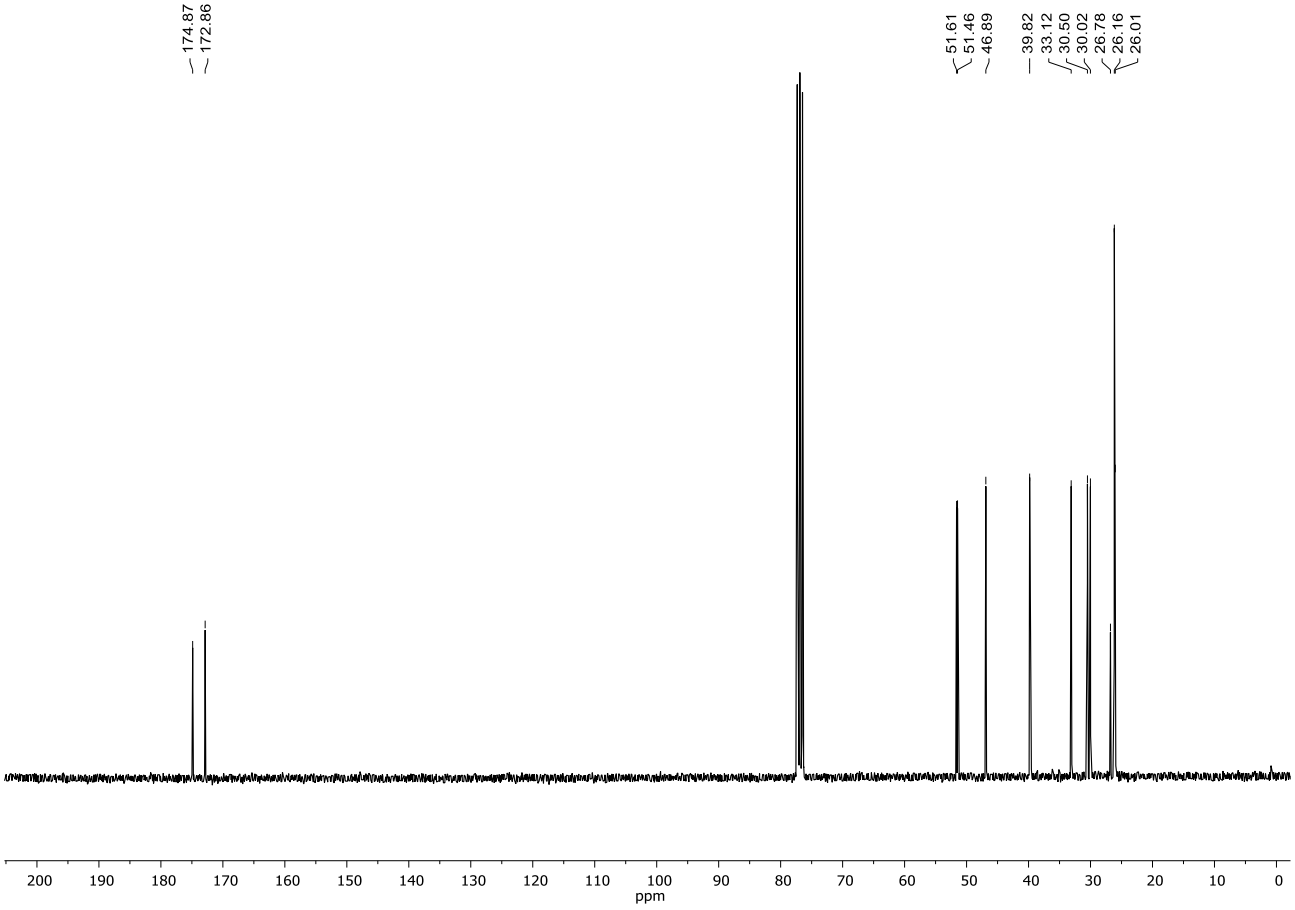


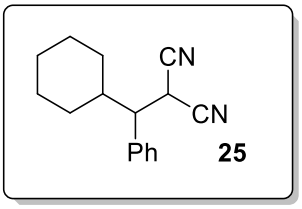
^{1}H-NMR (300 MHz, chloroform-*d*)



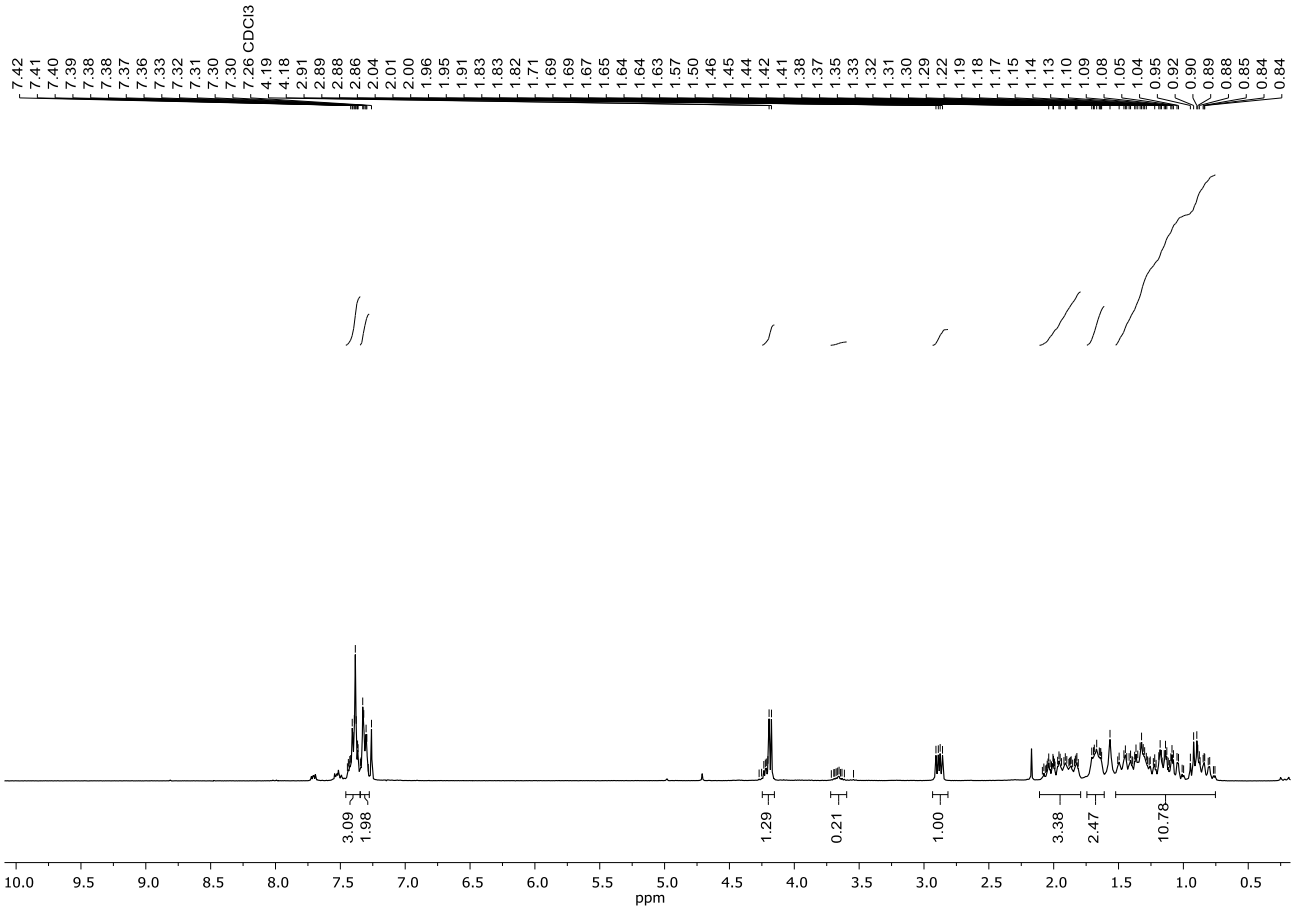


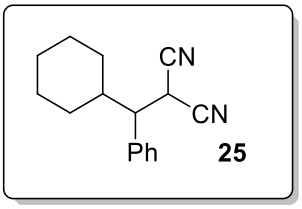
^{13}C-NMR (75 MHz, chloroform-*d*)



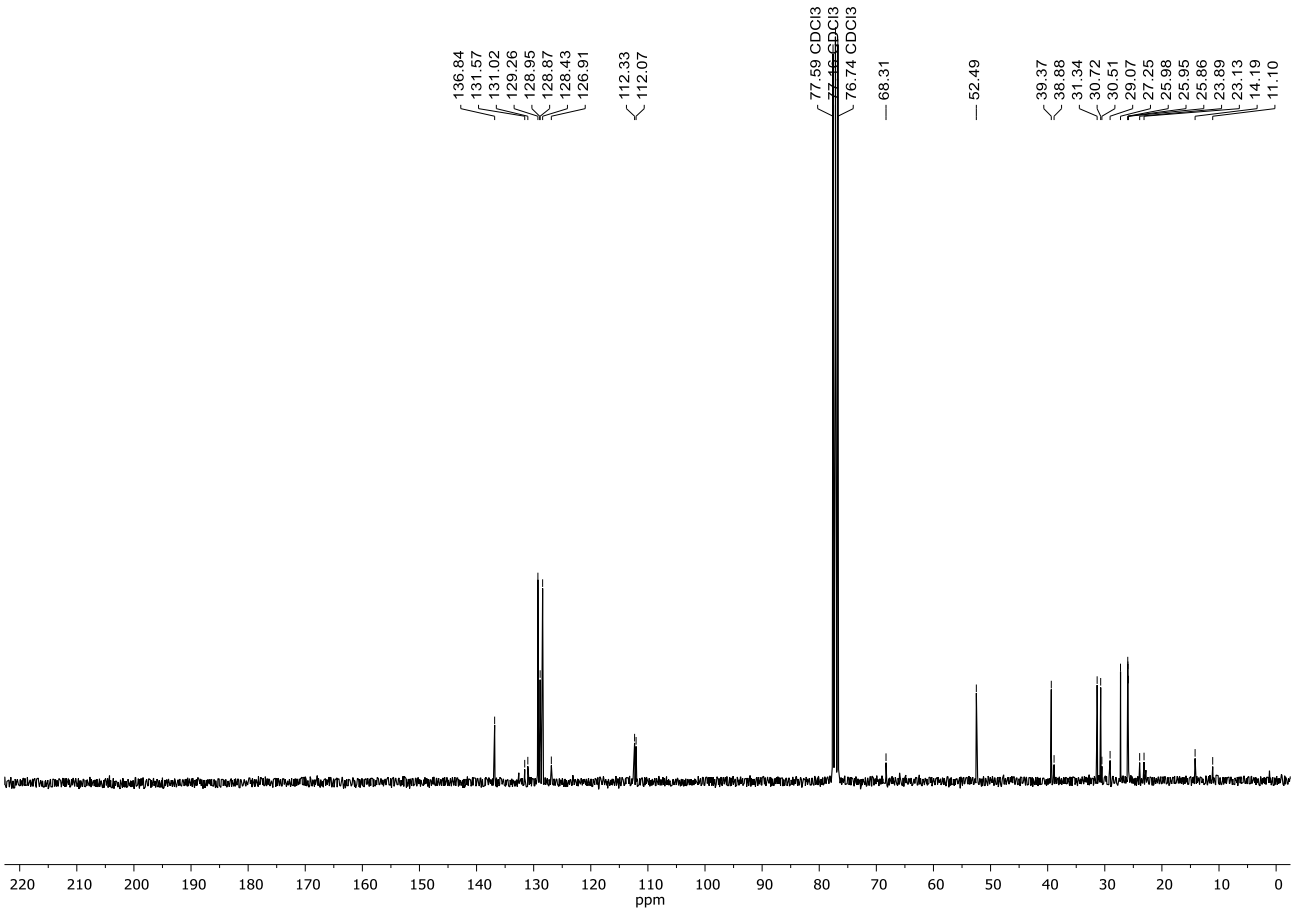


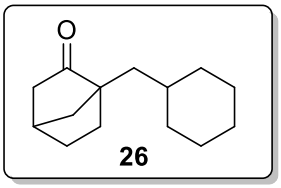
^{1}H-NMR (300 MHz, chloroform-*d*)



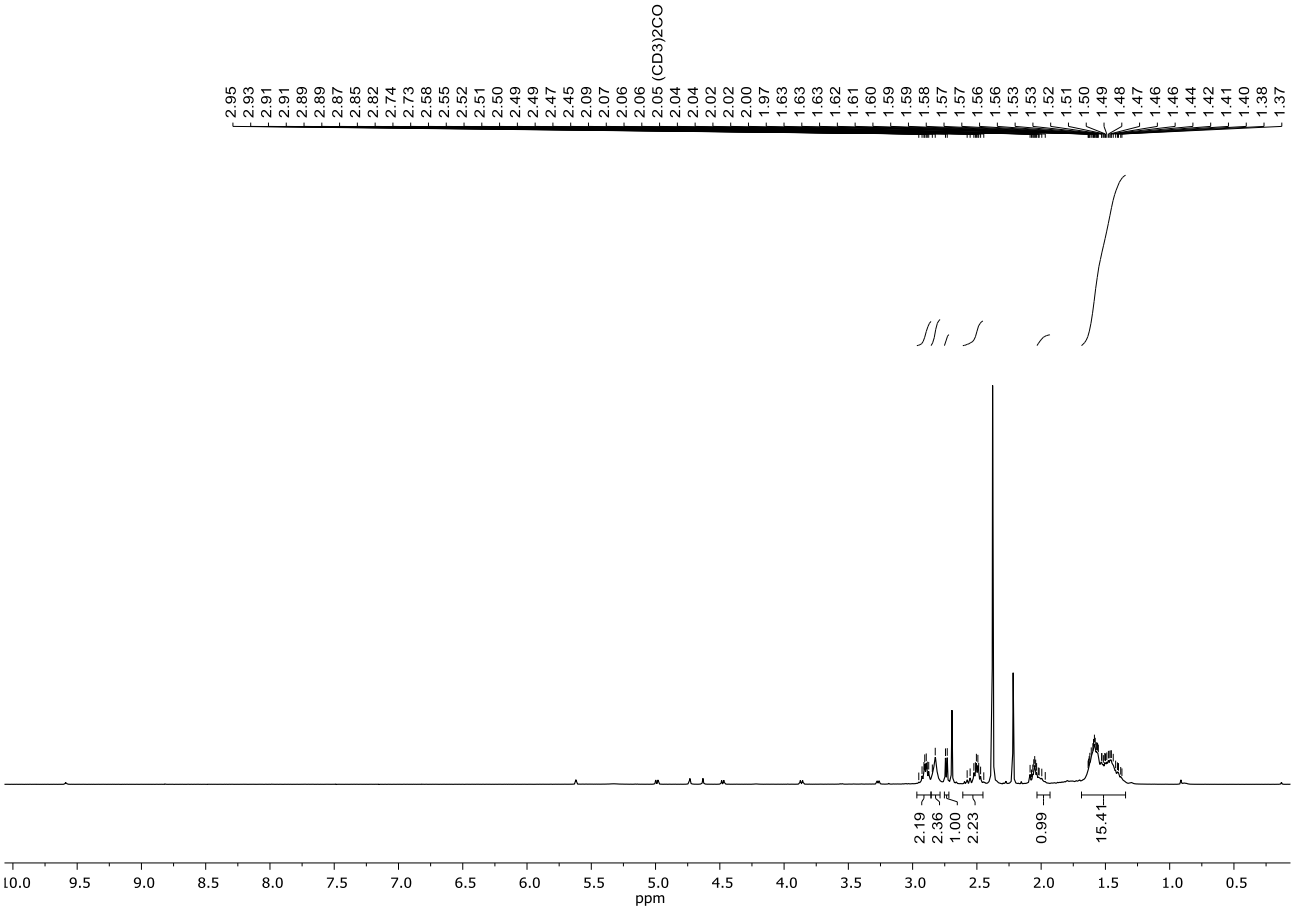


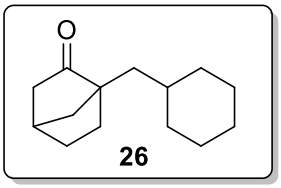
^{13}C-NMR (75 MHz, chloroform-*d*)



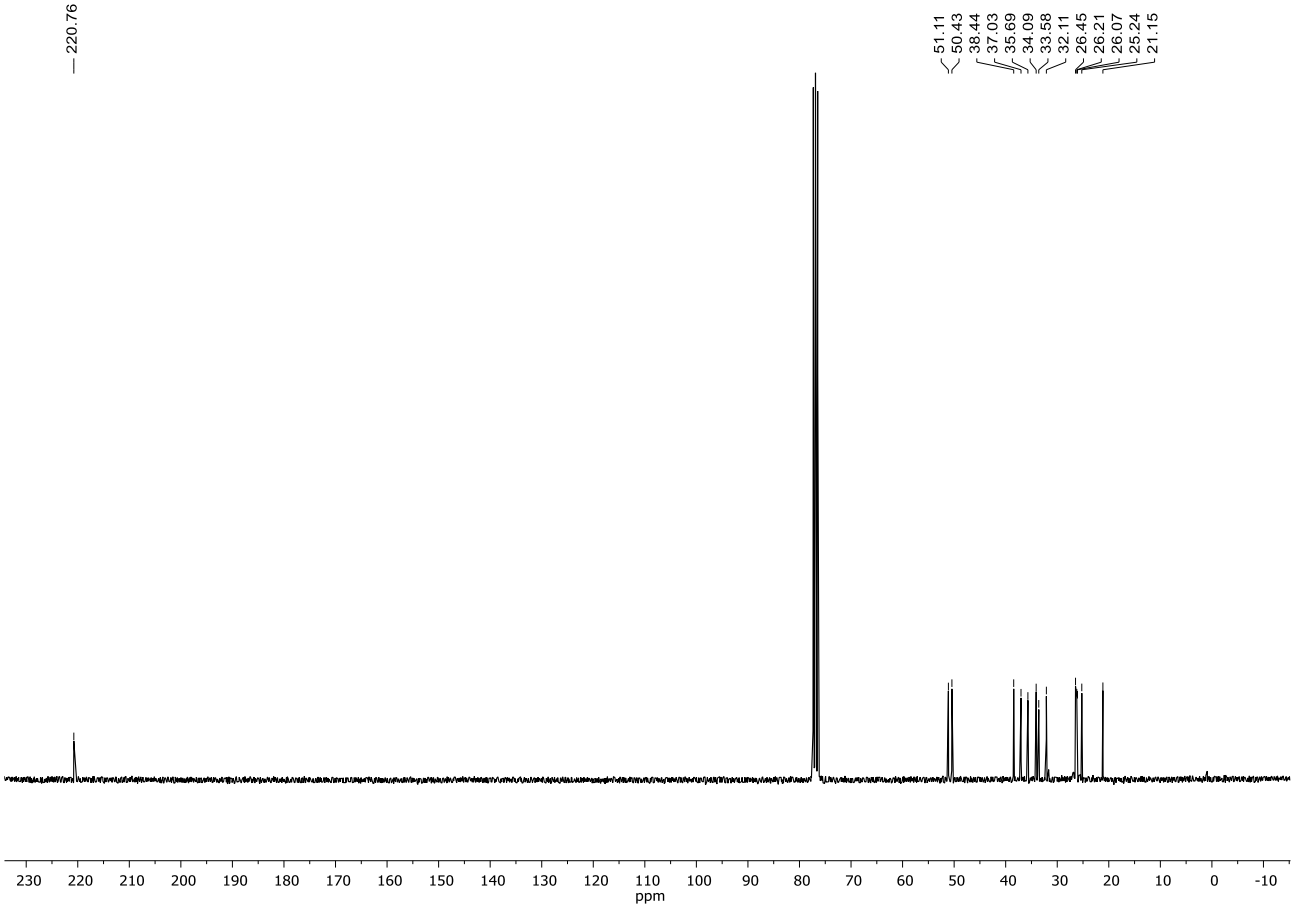


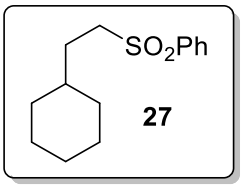
^{1}H-NMR (300 MHz, acetone-d_6)



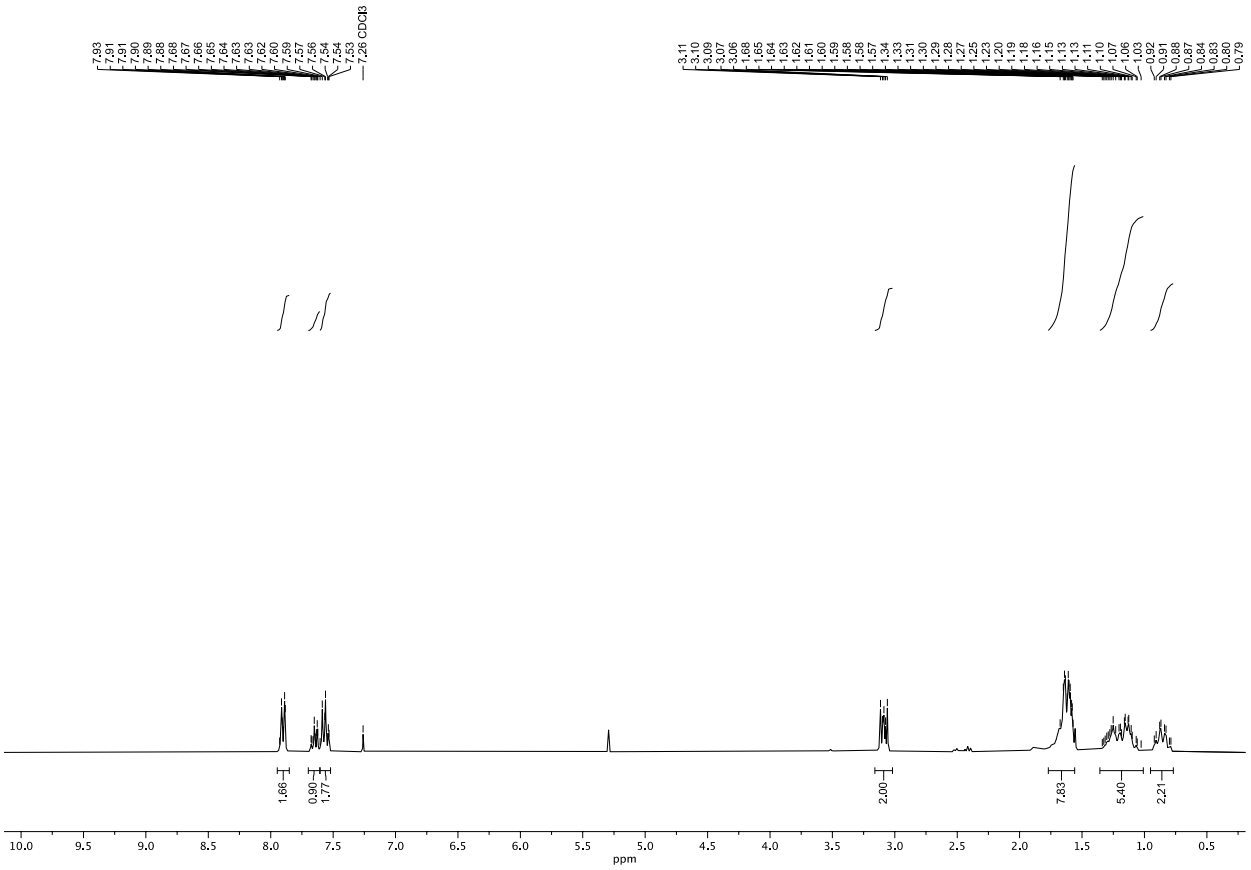


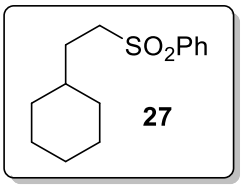
^{13}C-NMR (75MHz, acetone-d_6)



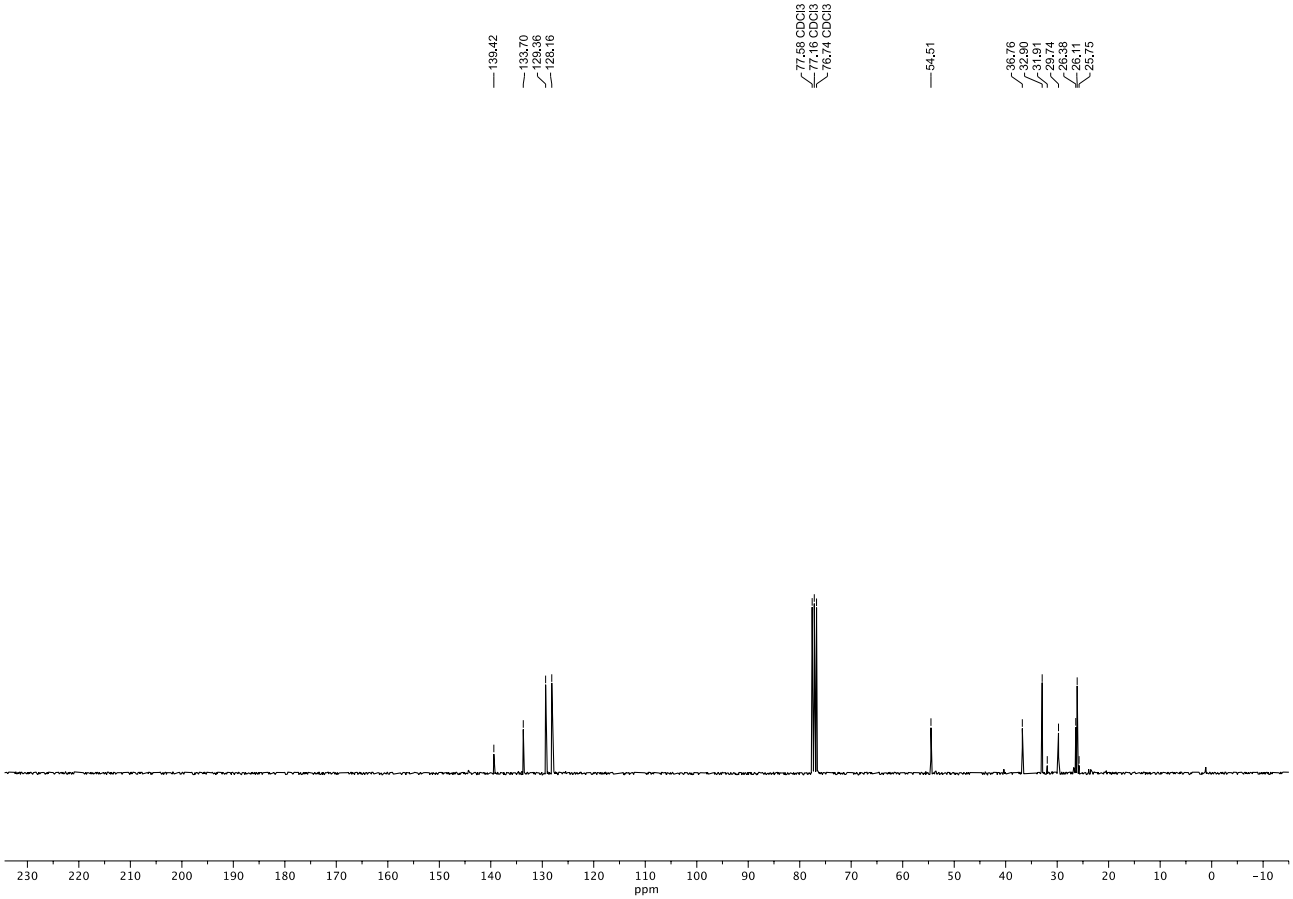


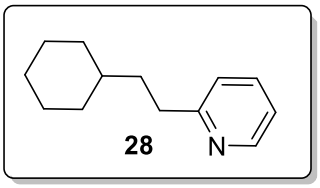
^{1}H-NMR (300 MHz, chloroform-*d*)



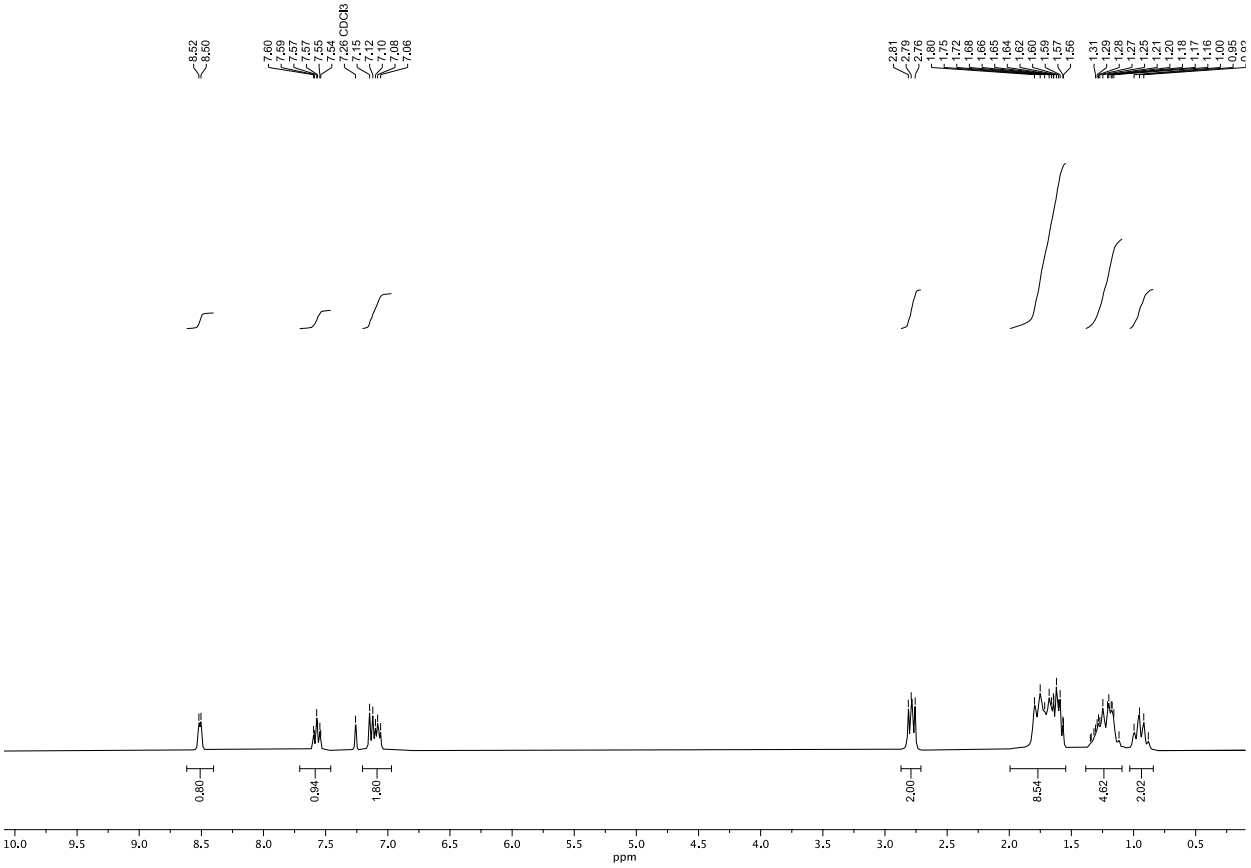


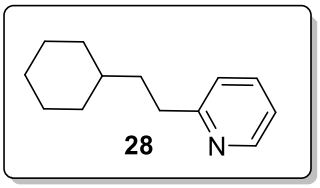
^{13}C-NMR (75 MHz, chloroform-*d*)



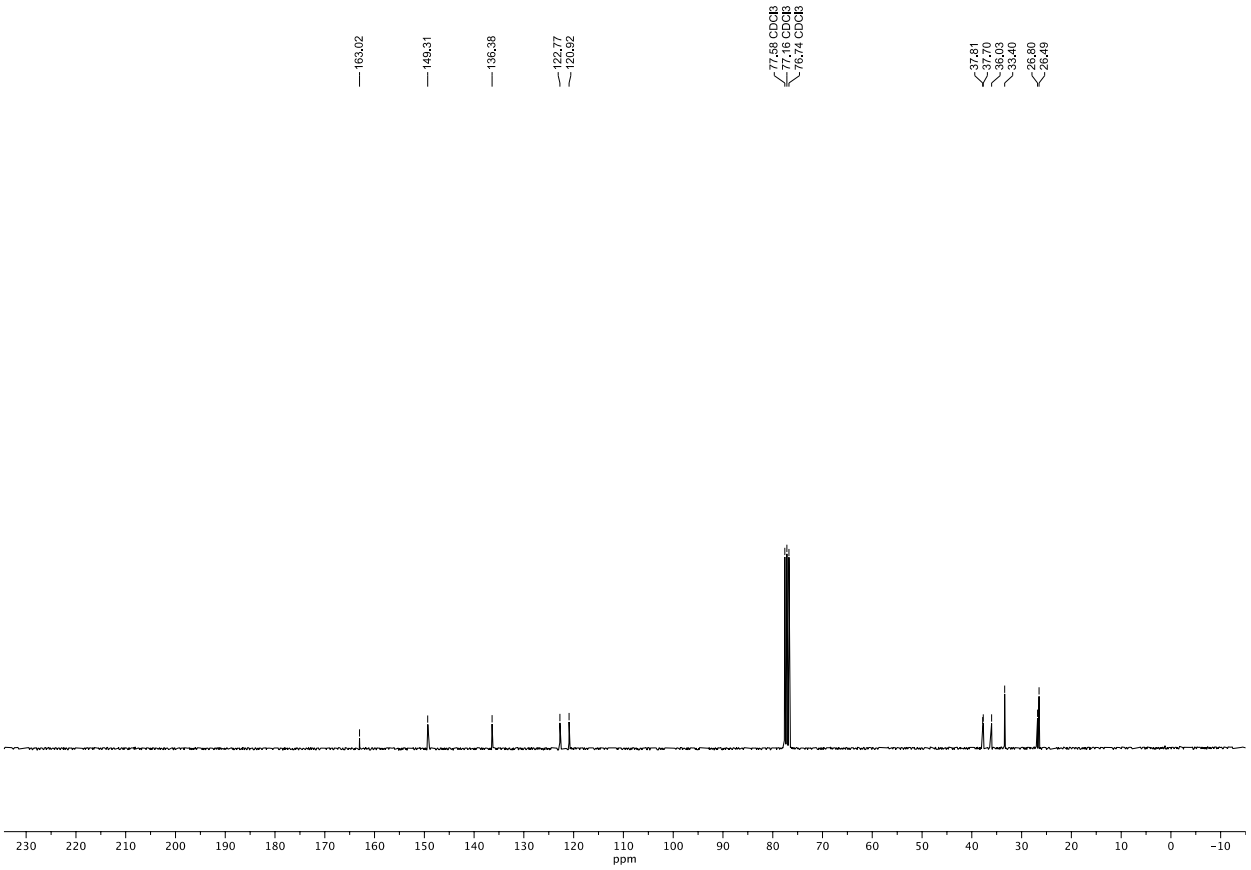


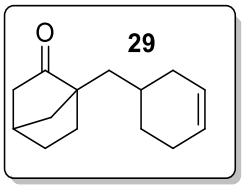
^{1}H-NMR (300 MHz, chloroform-*d*)



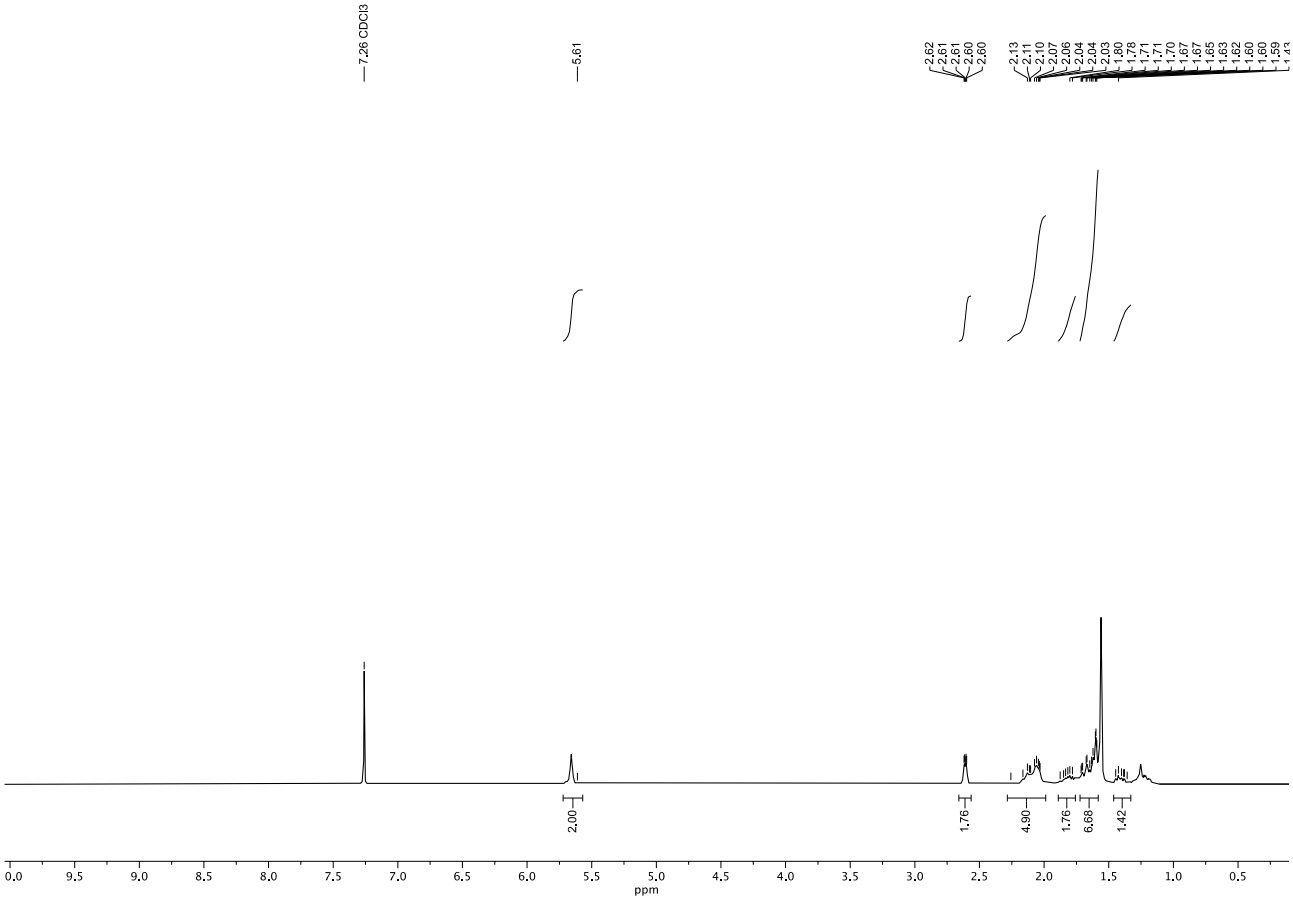


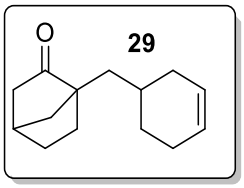
^{13}C-NMR (75 MHz, chloroform-*d*)



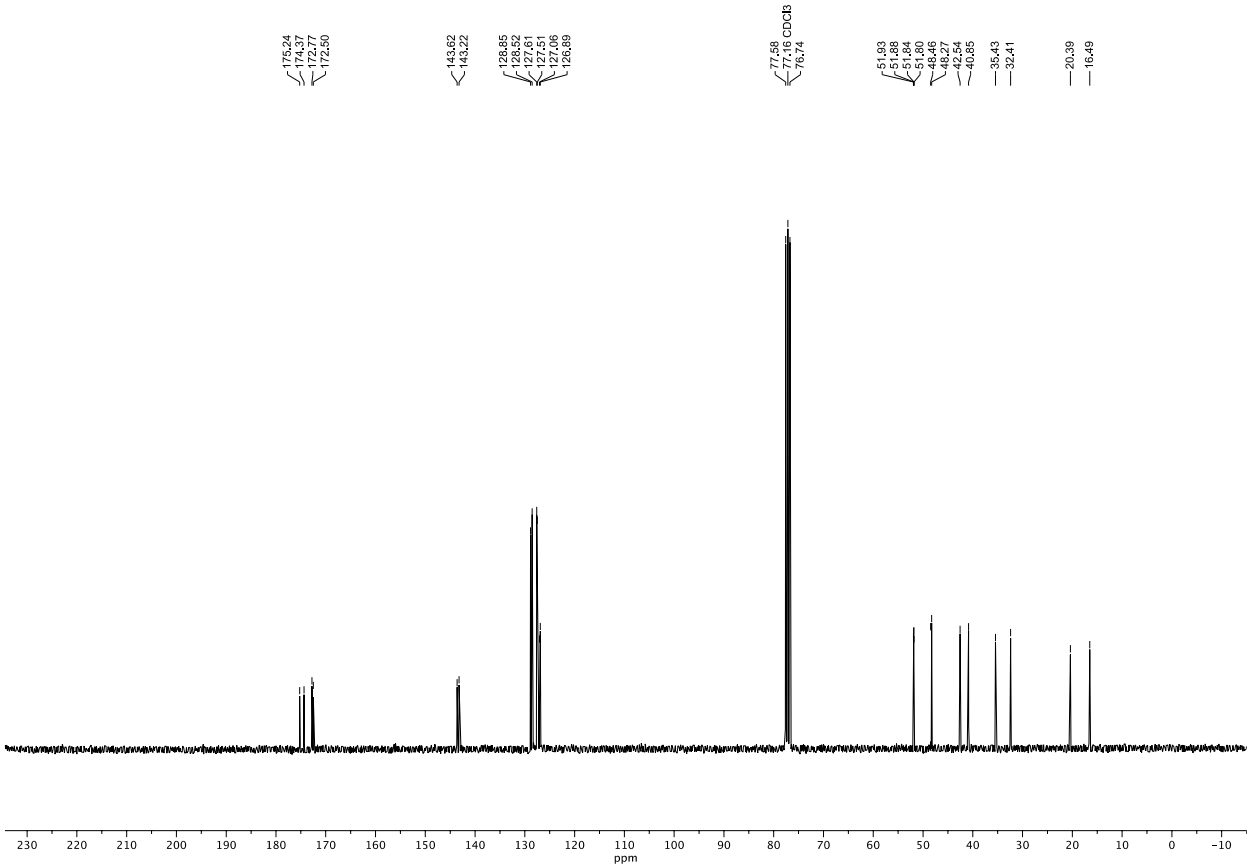


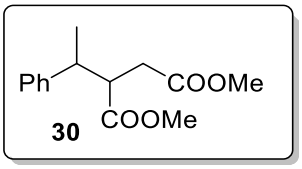
^{1}H-NMR (300 MHz, chloroform-*d*, mixture of two diastereoisomers)



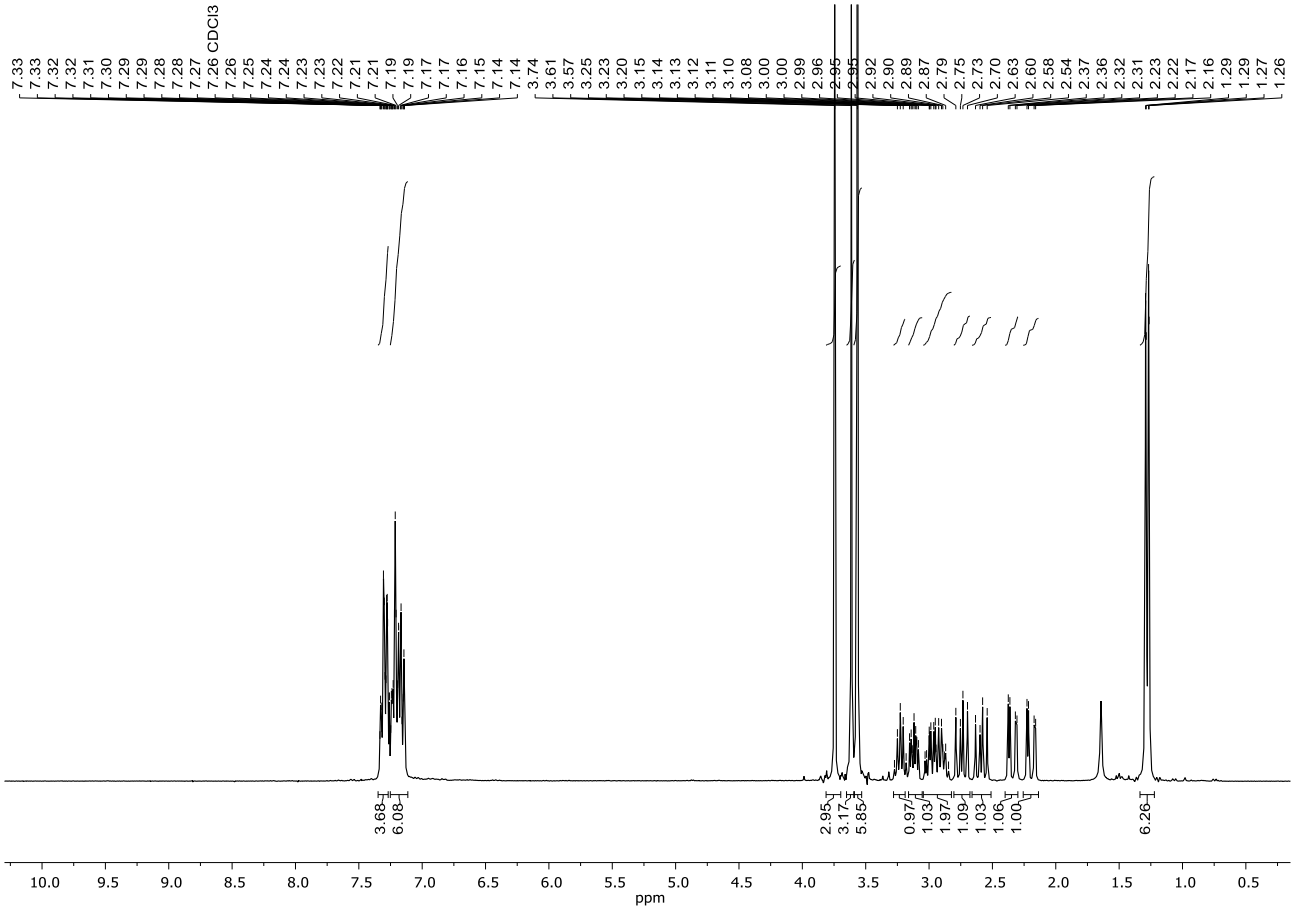


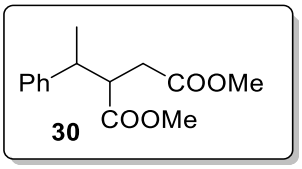
^{13}C-NMR (75 MHz, chloroform-*d*, mixture of two diastereoisomers)



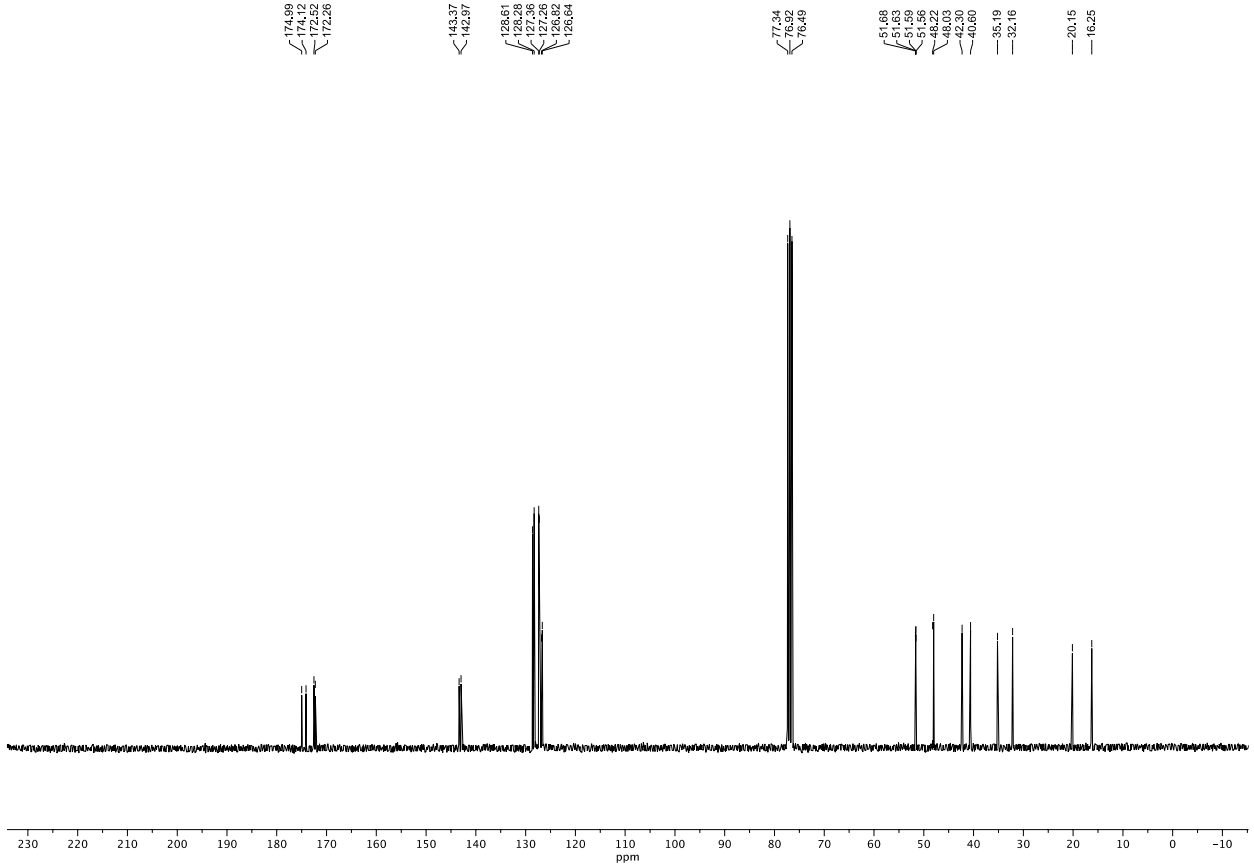


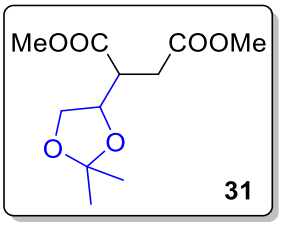
^{1}H-NMR (300 MHz, chloroform-*d*, mixture of two diastereoisomers)



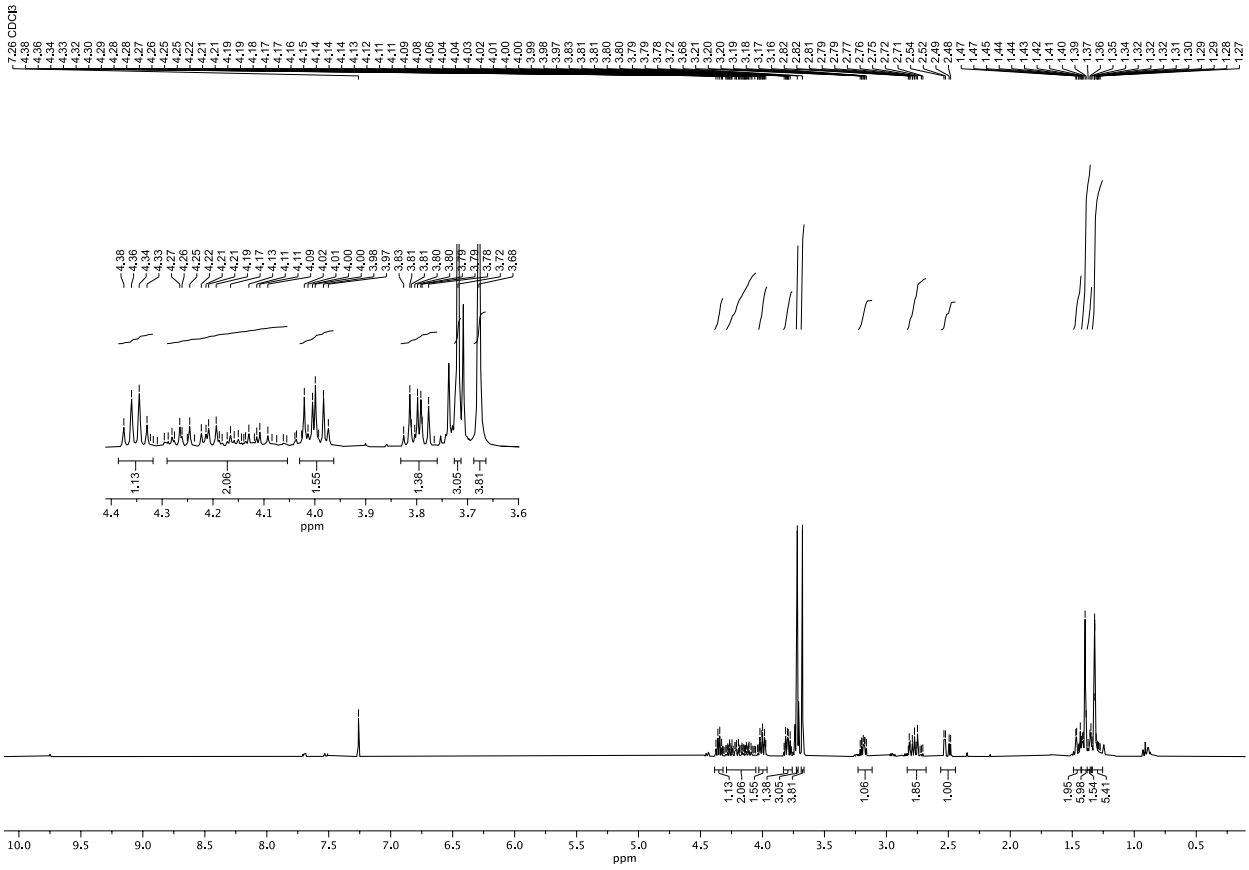


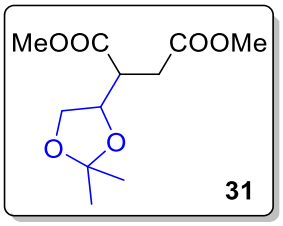
^{13}C-NMR (75 MHz, chloroform-*d*, mixture of two diastereoisomers)



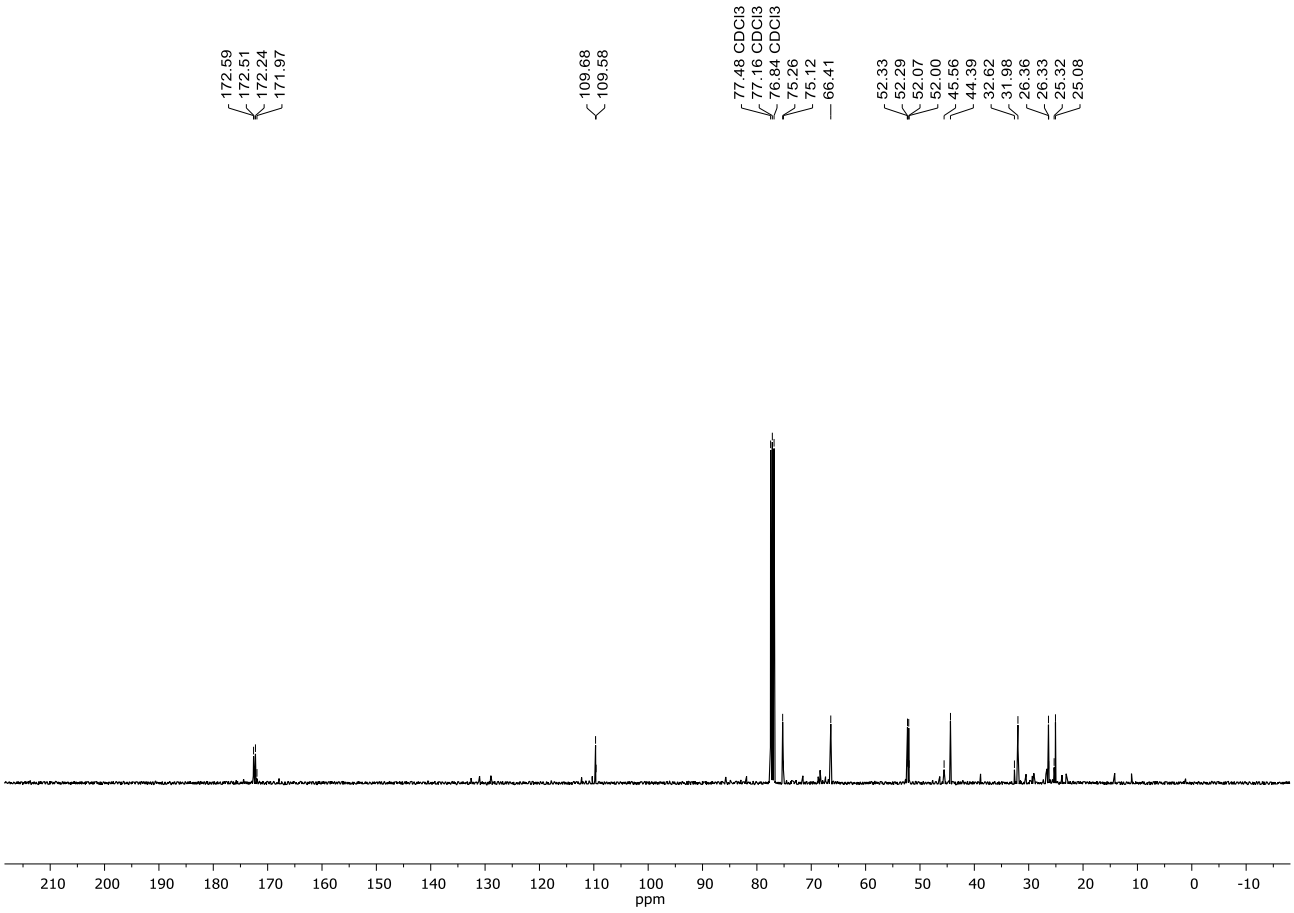


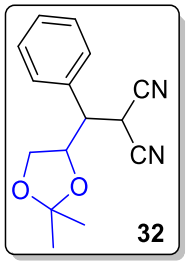
^{1}H-NMR (400 MHz, chloroform-*d*, mixture of two diasteroisomers)



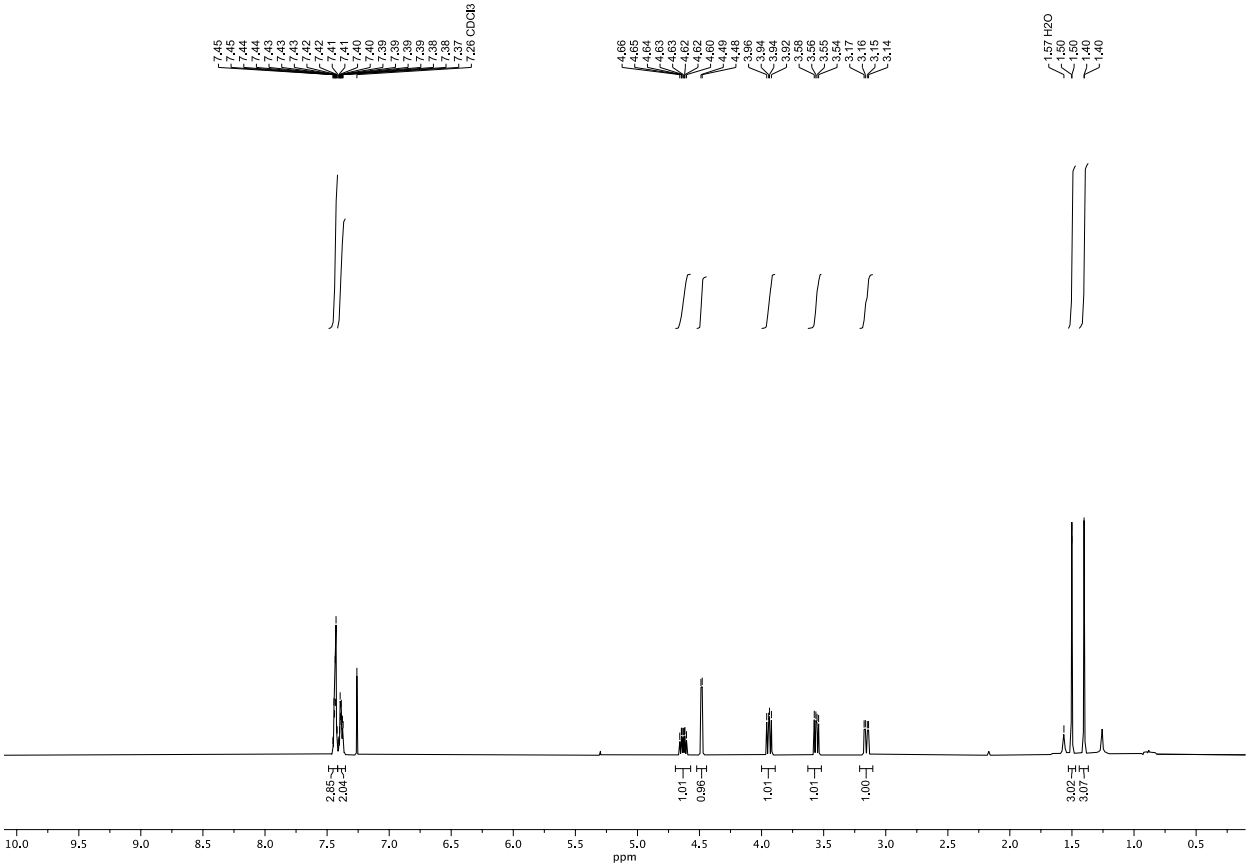


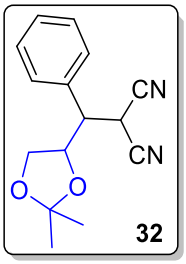
^{13}C-NMR (100 MHz, chloroform-*d*, mixture of two diasteroisomers)



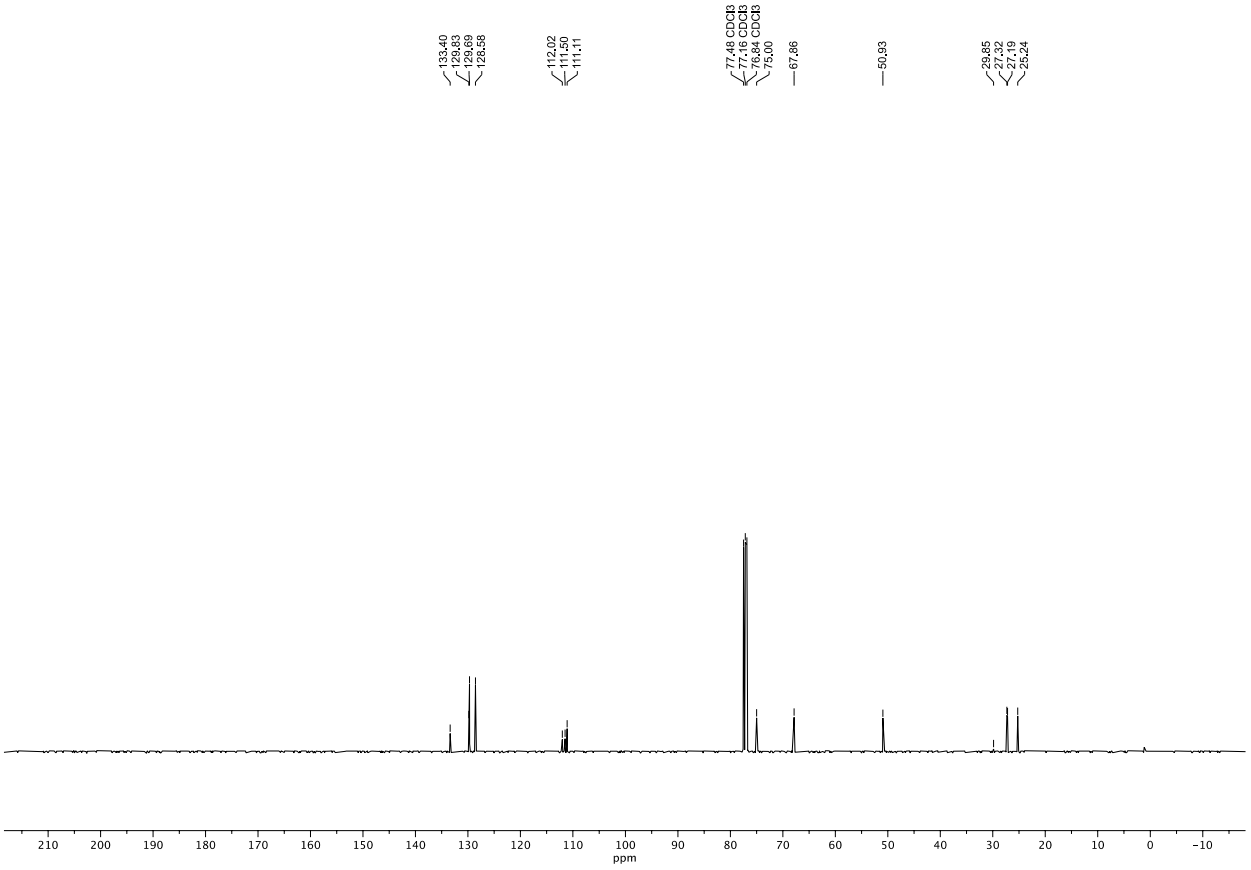


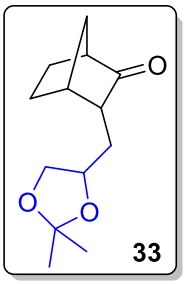
^{1}H-NMR (400 MHz, chloroform-*d*, mixture of two diasteroisomers)



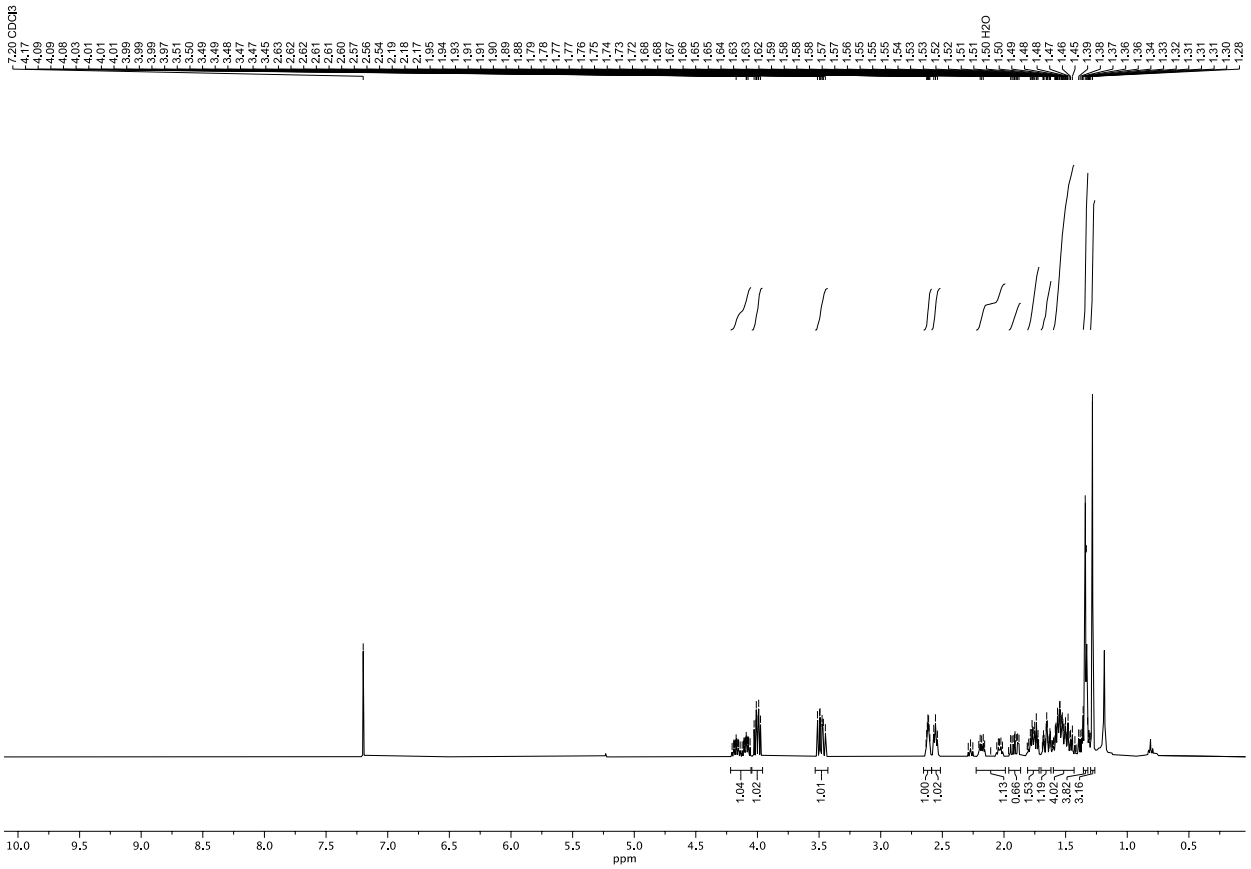


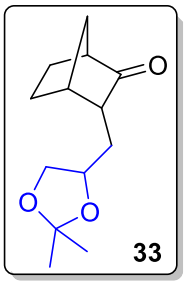
^{13}C-NMR (100 MHz, chloroform-*d*, mixture of two diasteroisomers)



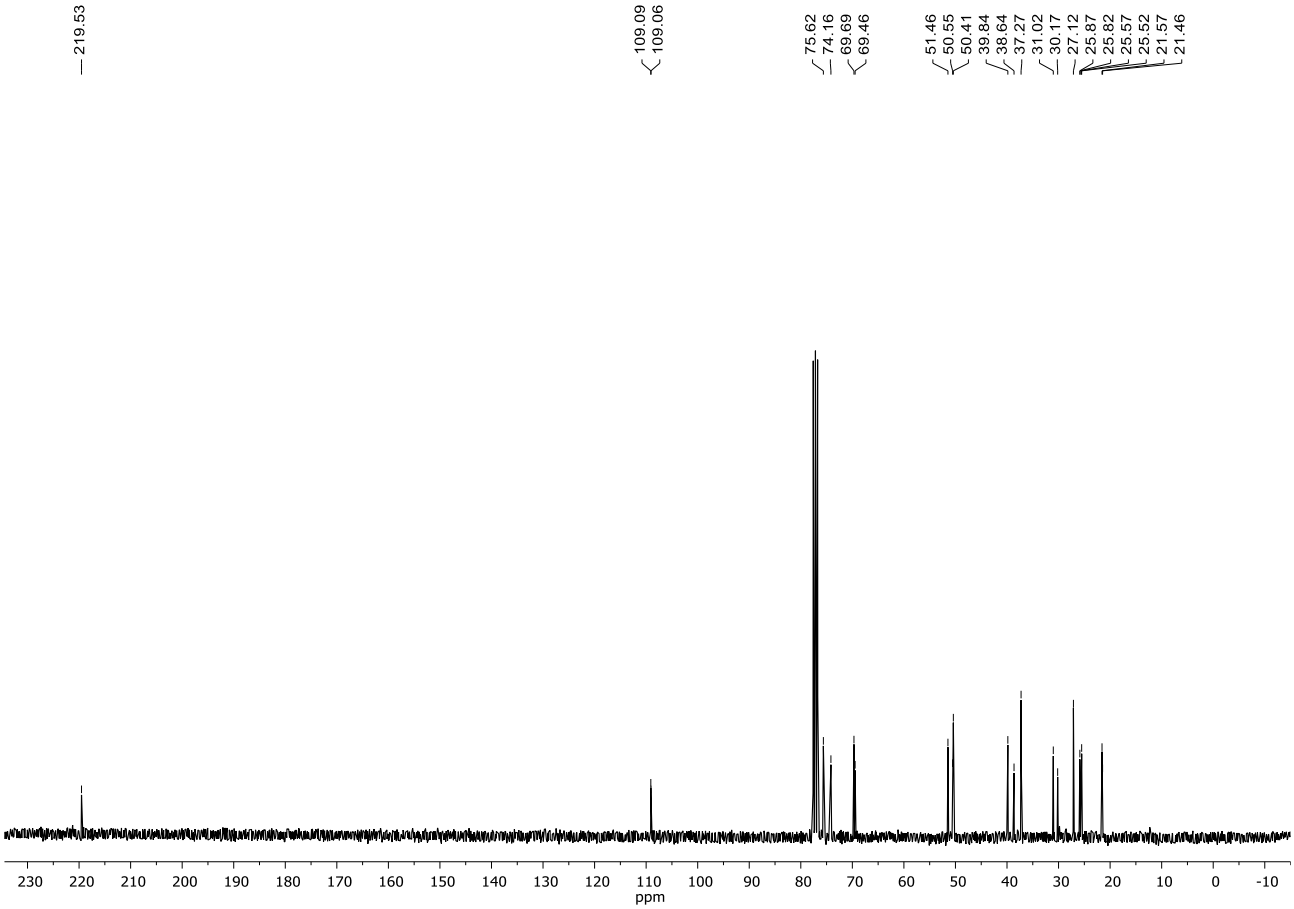


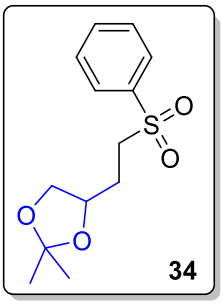
^{1}H-NMR (400 MHz, chloroform-*d*, mixture of two diasteroisomers)



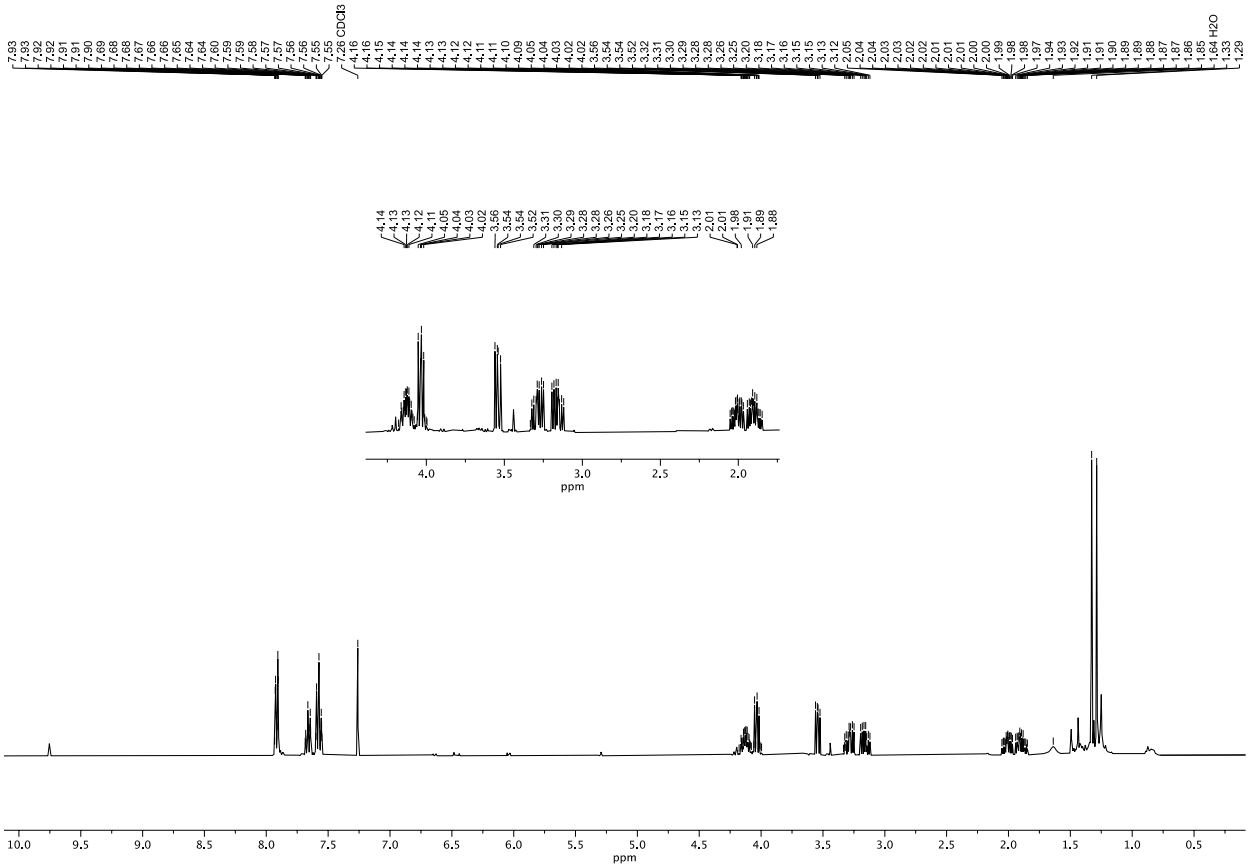


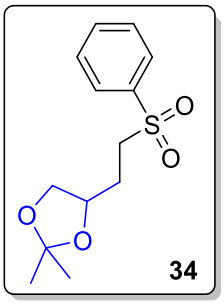
^{13}C-NMR (100 MHz, chloroform-*d*, mixture of two diasteroisomers)



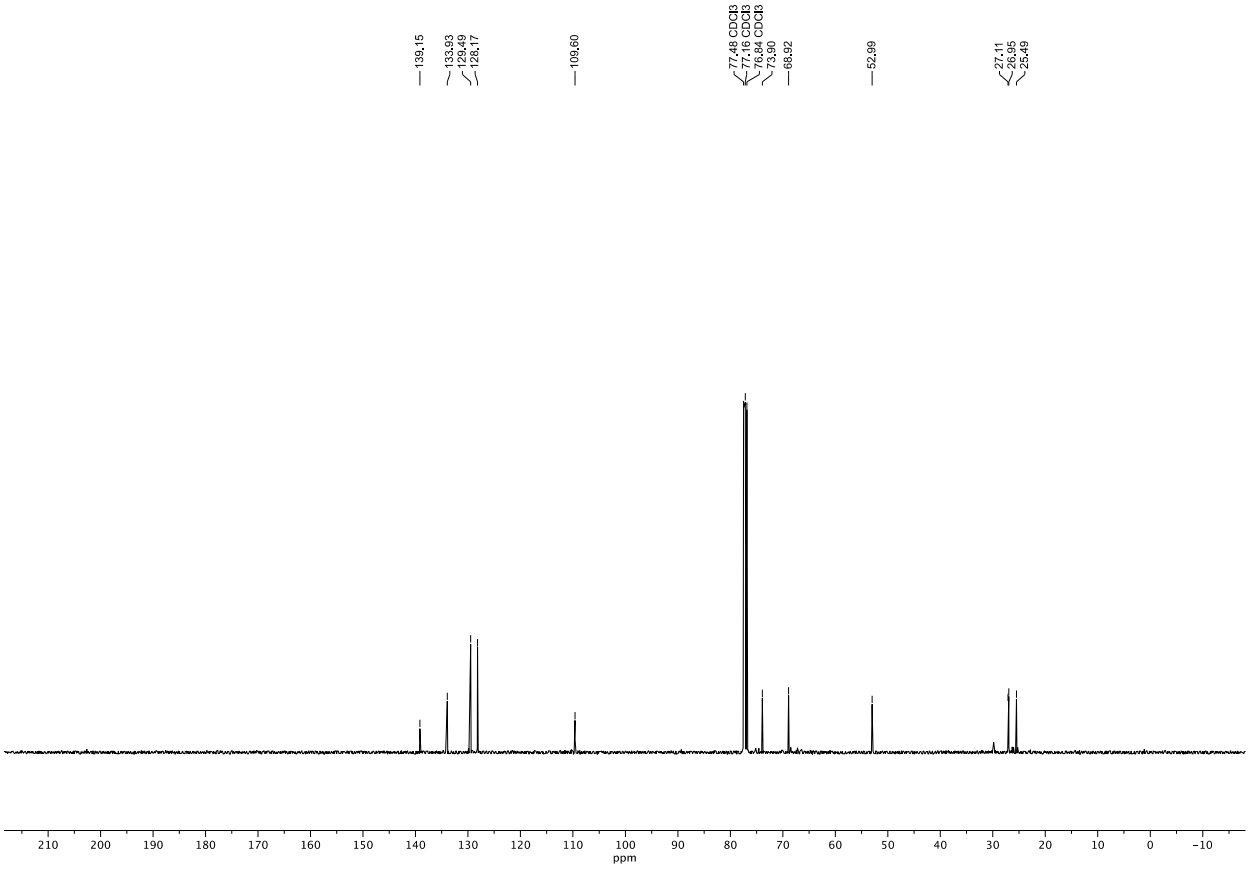


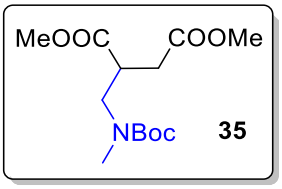
^{1}H-NMR (400 MHz, chloroform-*d*)



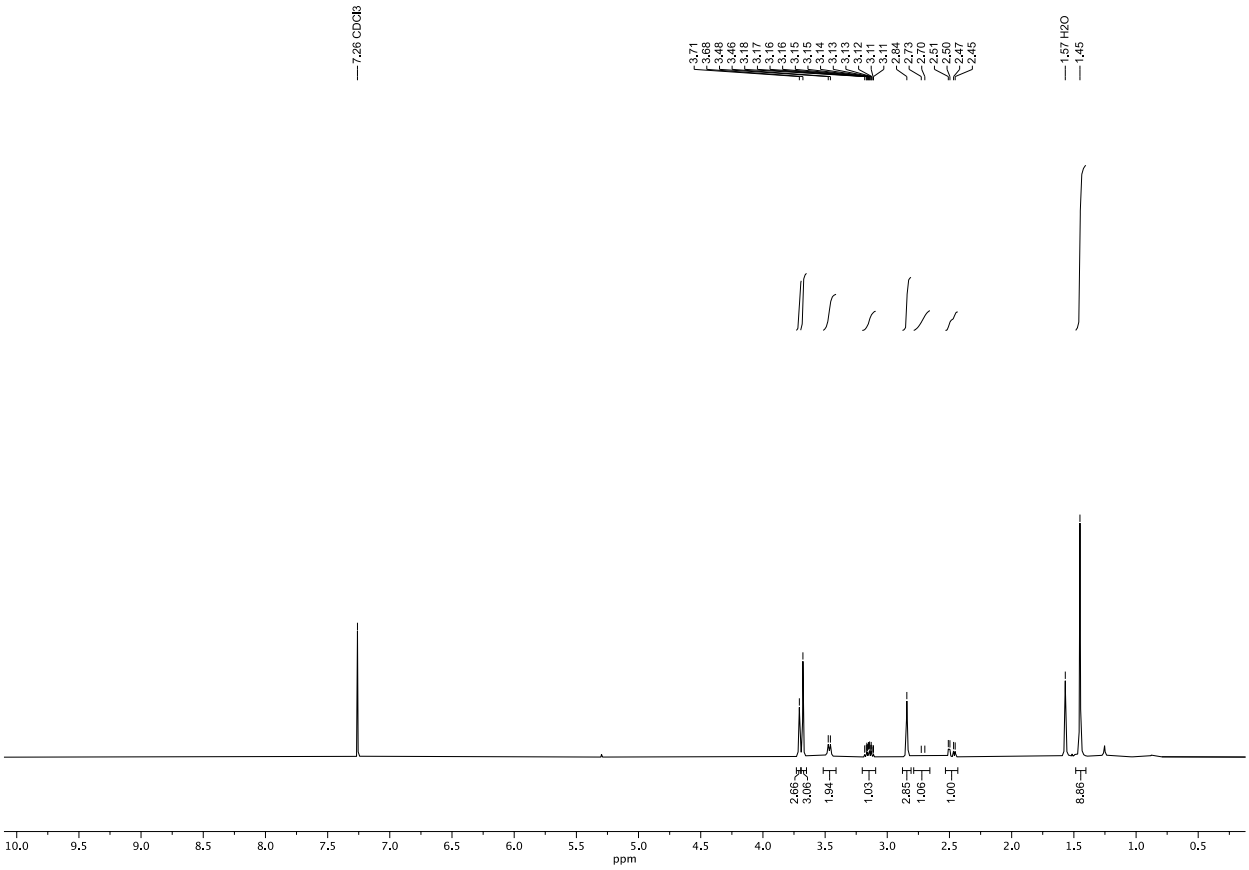


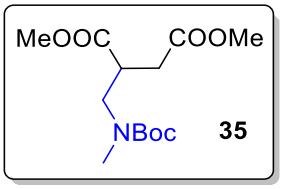
^{13}C-NMR (100 MHz, chloroform-*d*)



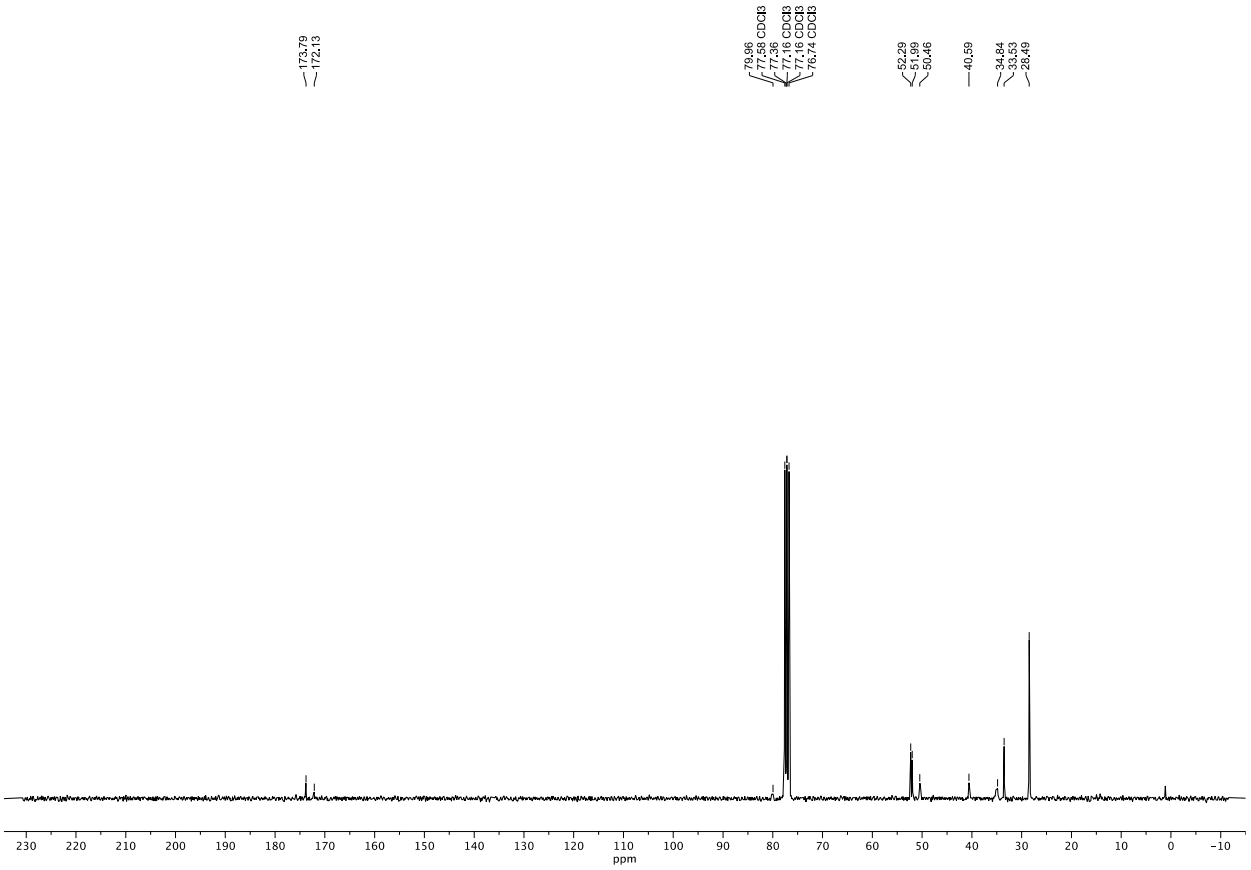


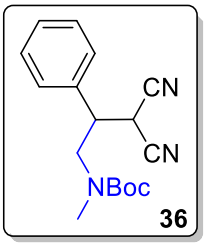
^{1}H-NMR (400 MHz, chloroform-*d*)



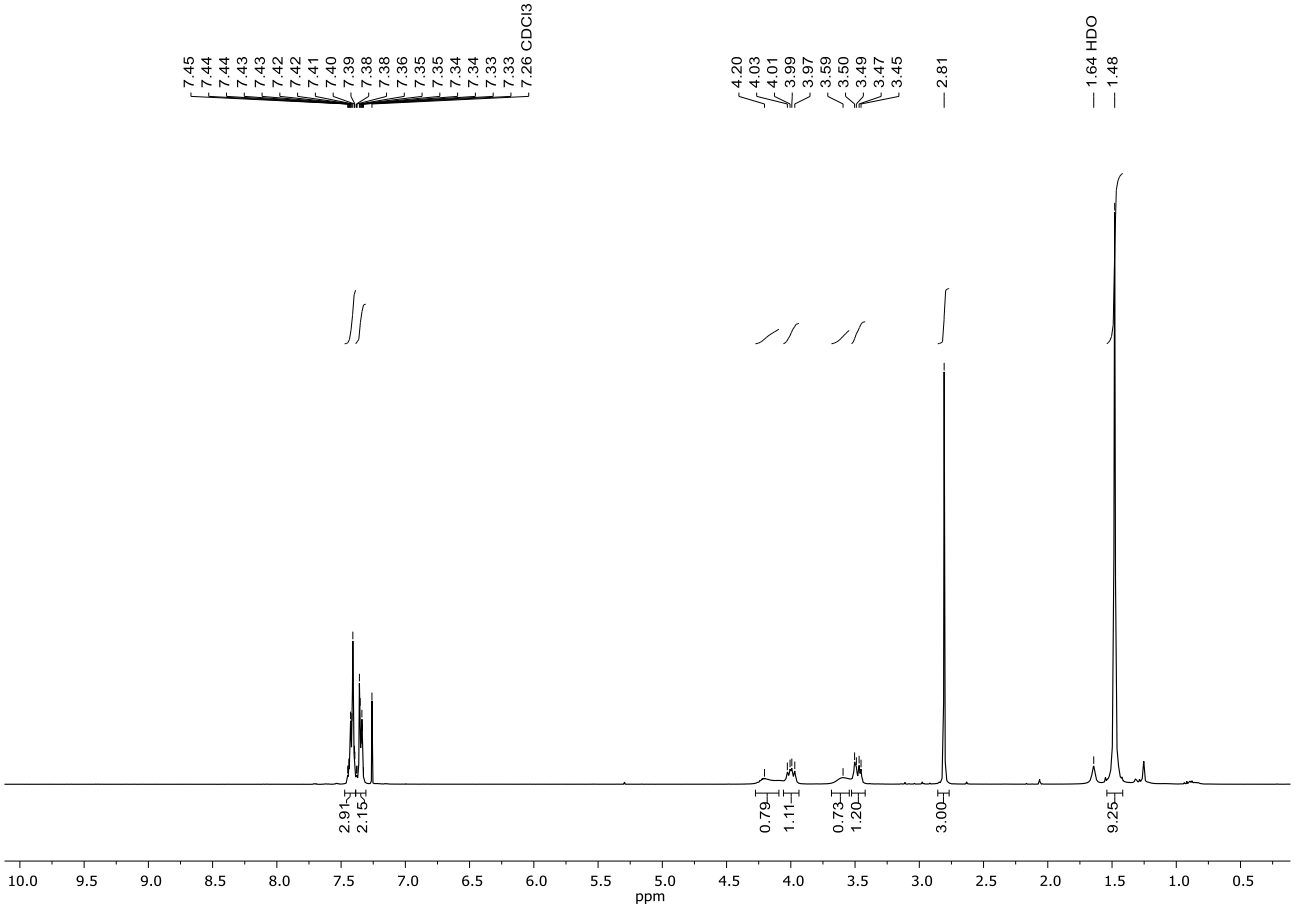


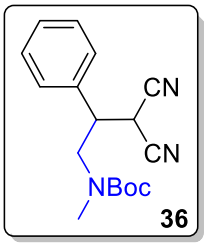
^{13}C-NMR (100 MHz, chloroform-*d*)



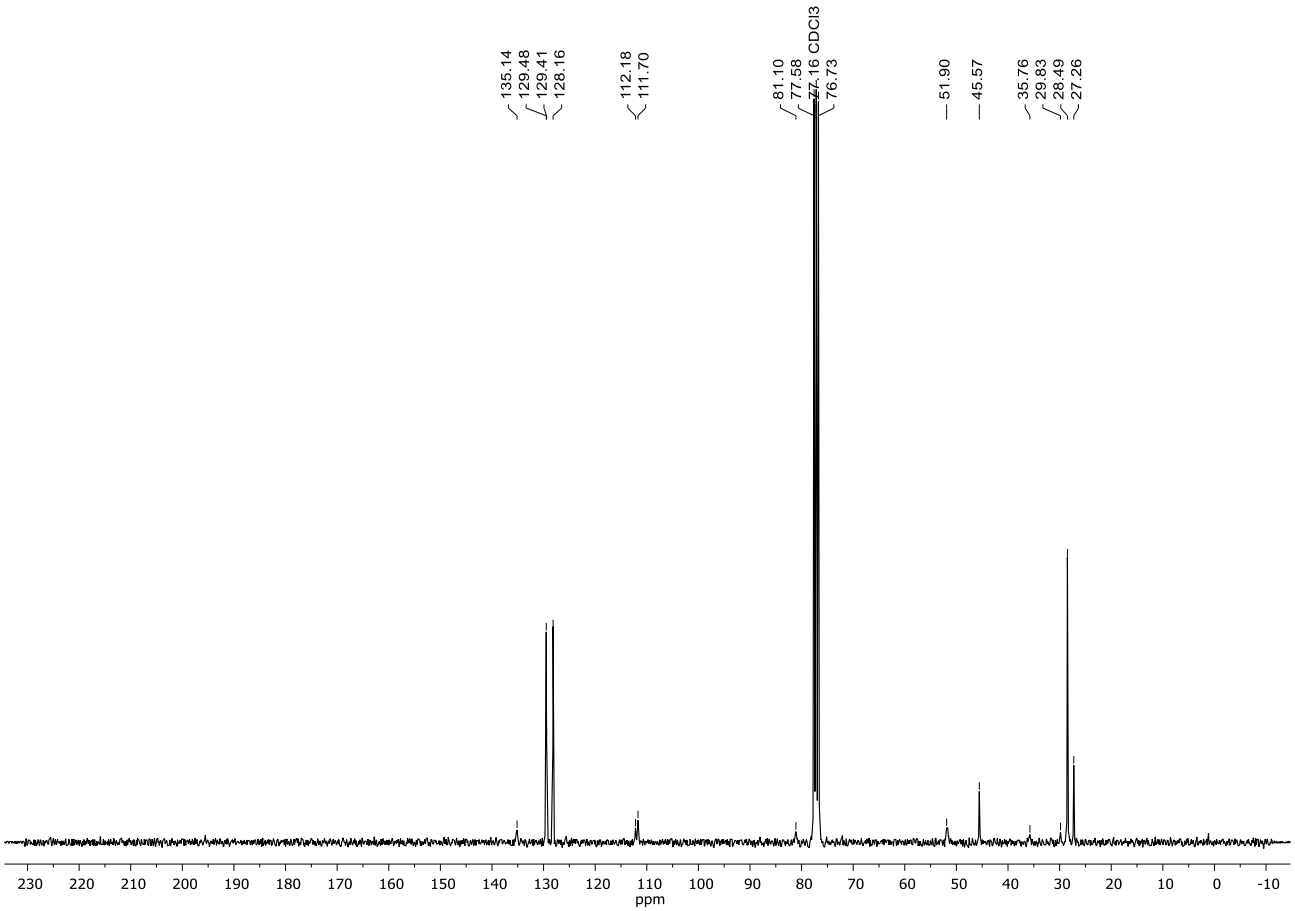


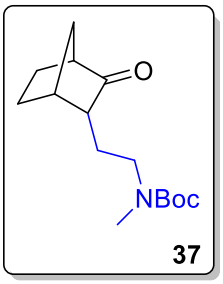
^{1}H-NMR (400 MHz, chloroform-*d*)



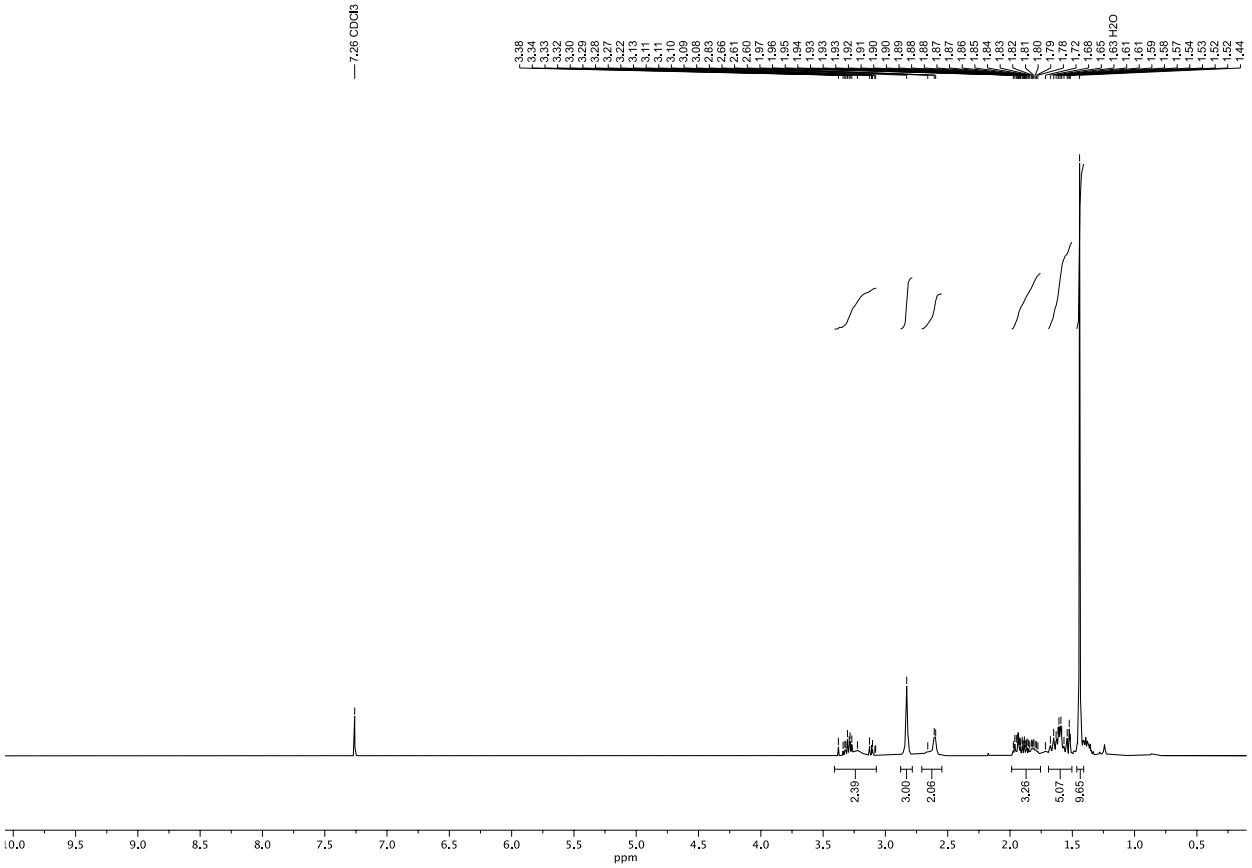


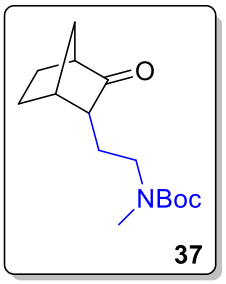
^{13}C-NMR (100 MHz, chloroform-*d*)



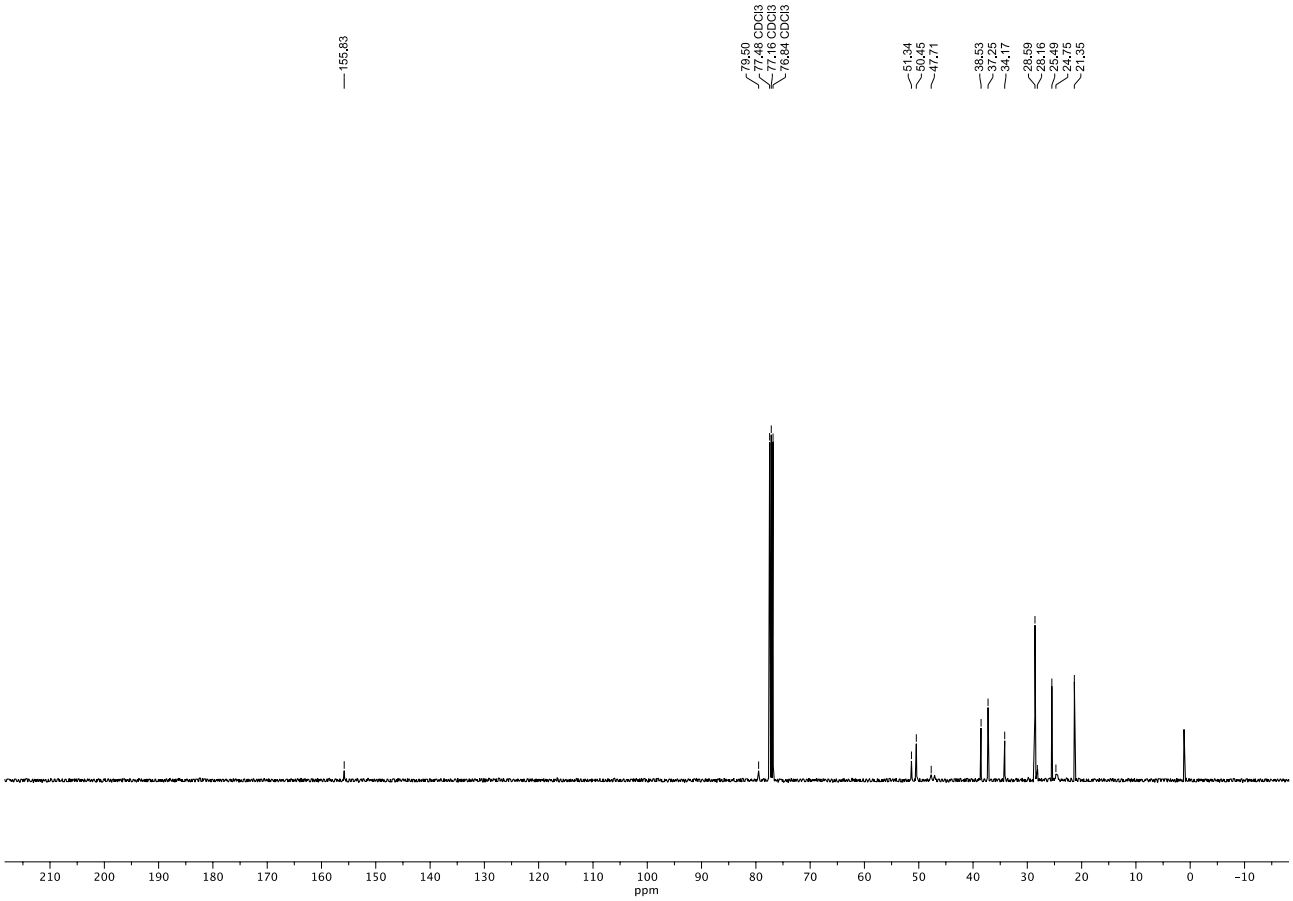


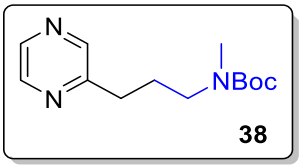
^{1}H-NMR (400 MHz, chloroform-*d*)



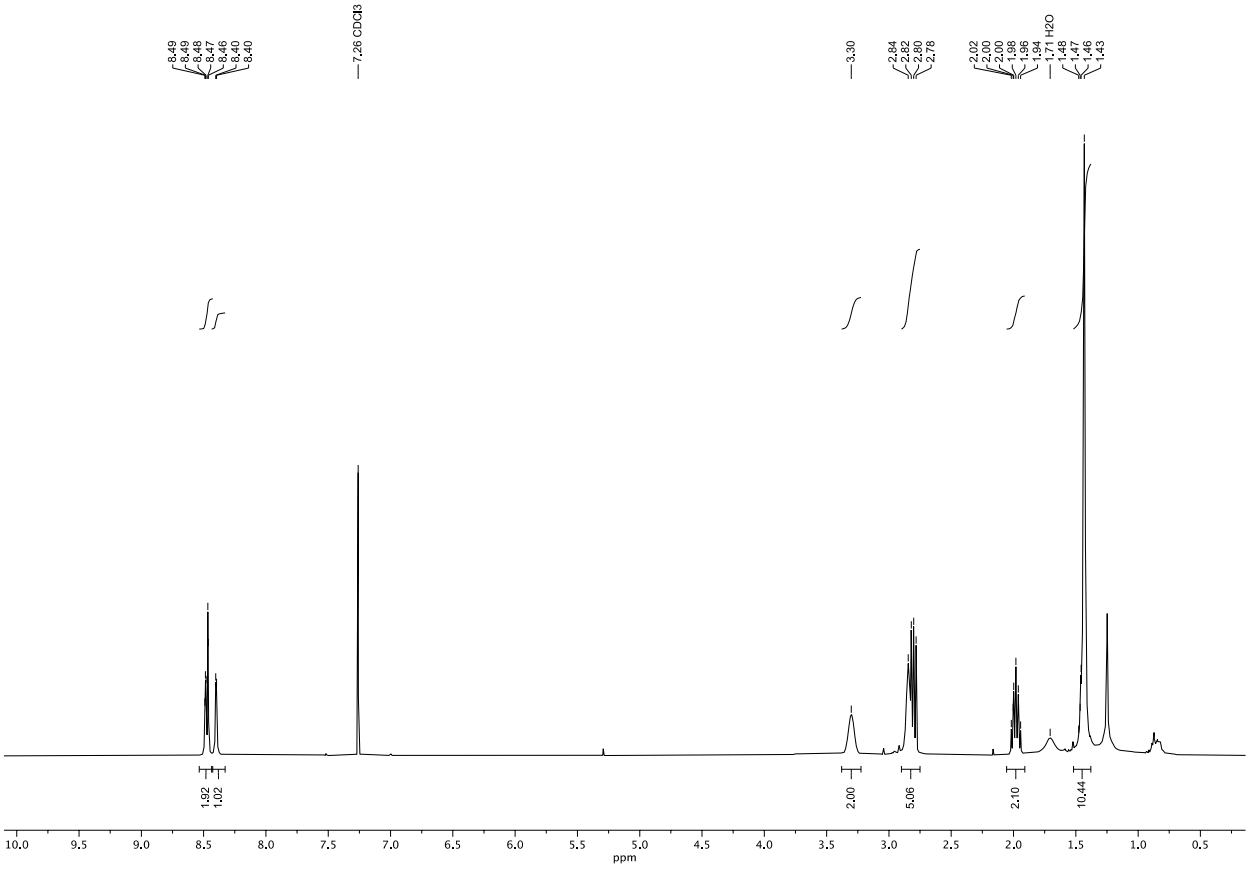


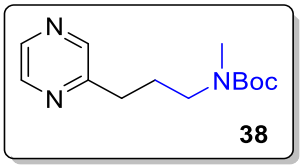
^{13}C-NMR (100 MHz, chloroform-*d*)



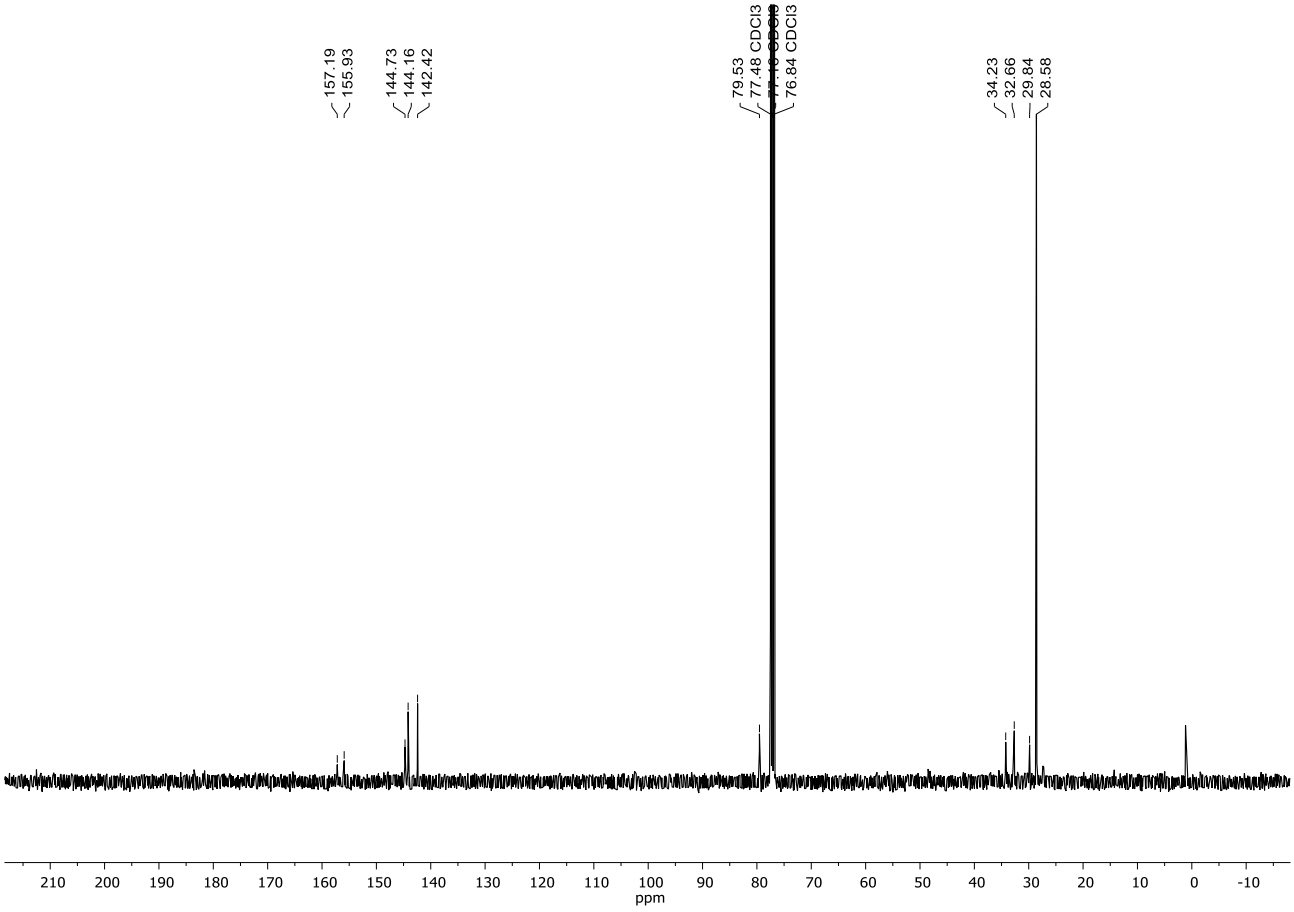


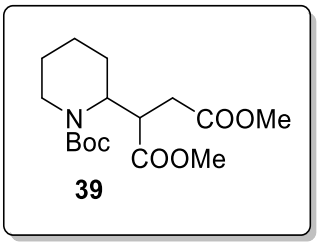
^{1}H-NMR (400 MHz, chloroform-*d*)





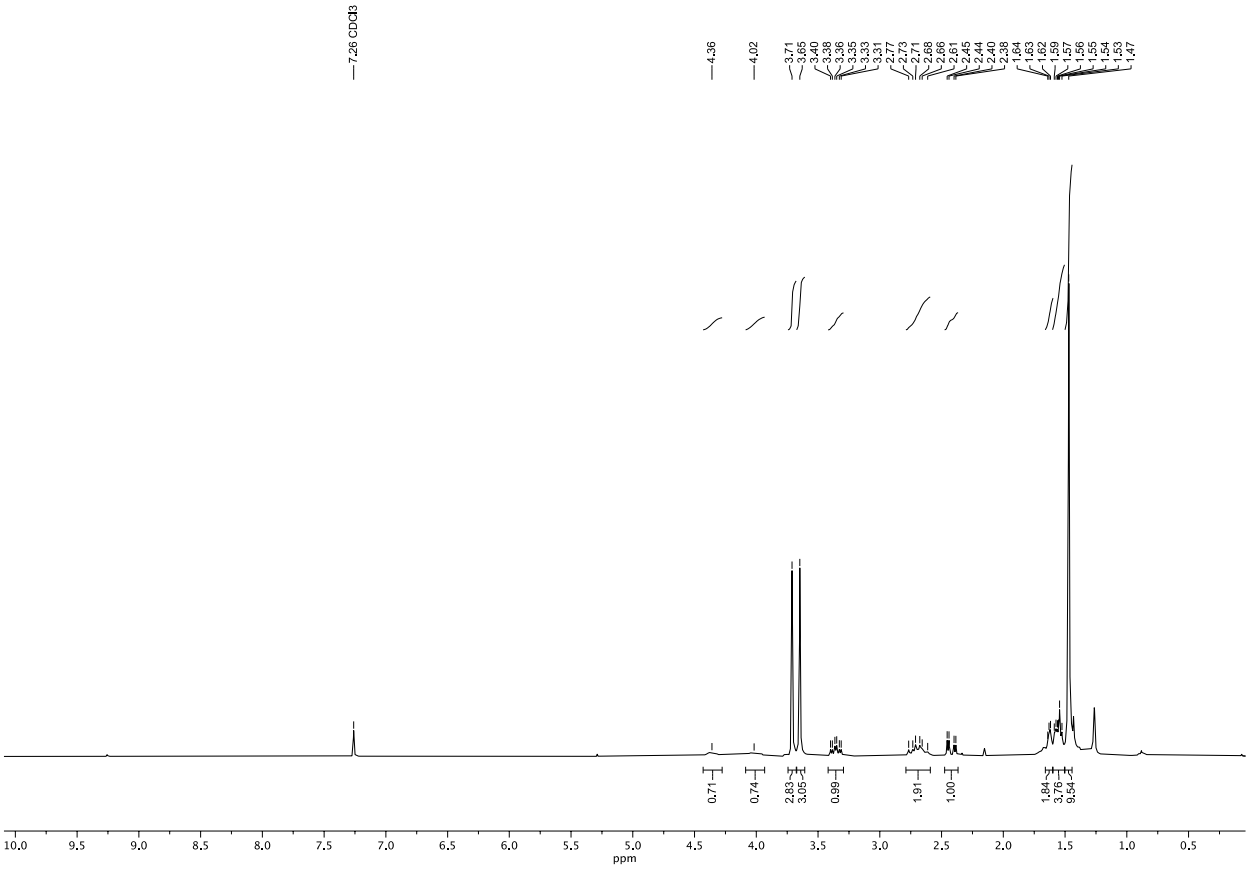
^{13}C-NMR (100 MHz, chloroform-*d*)

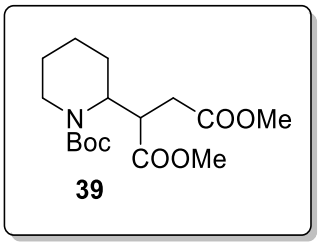




39

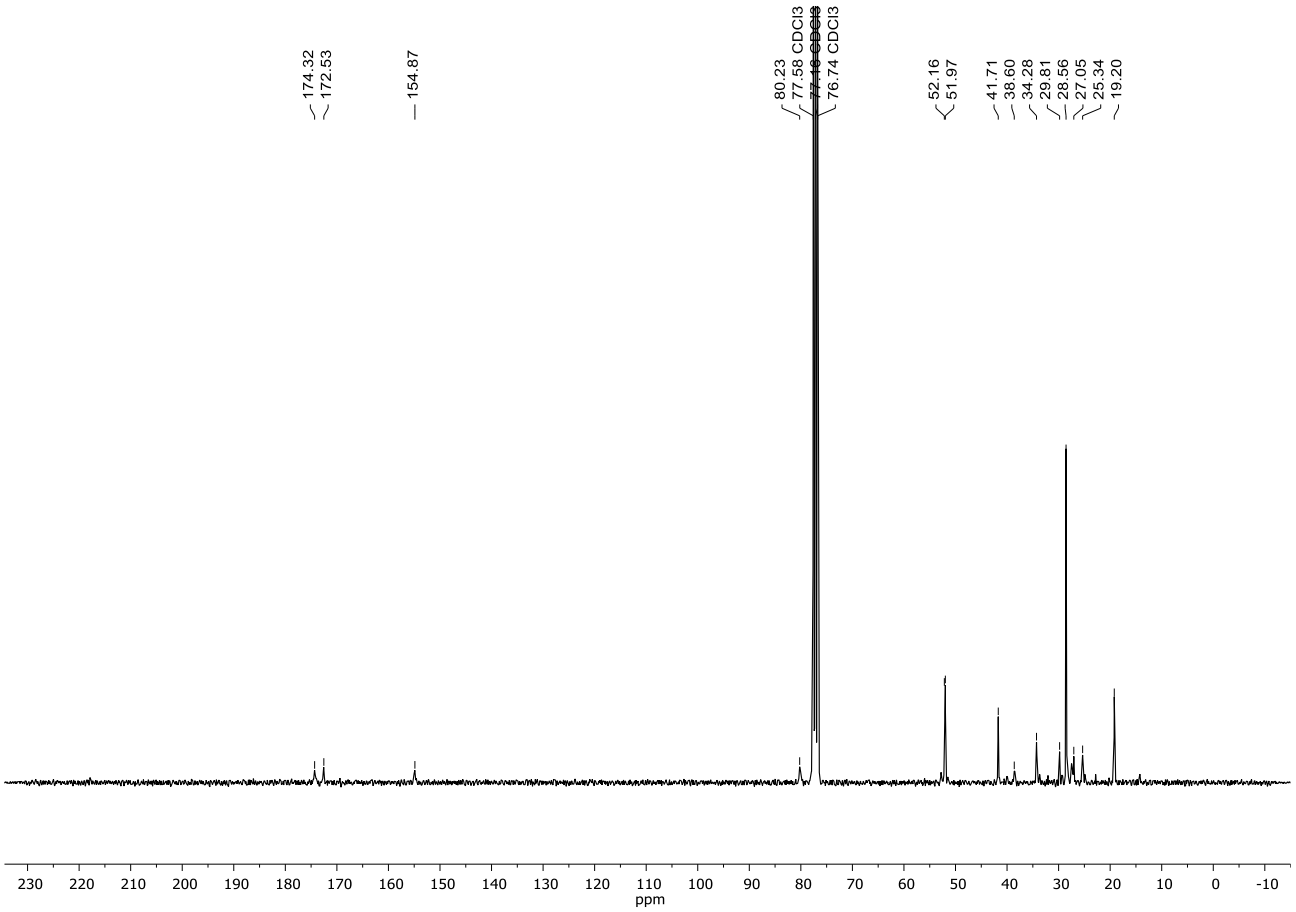
^{1}H-NMR (300 MHz, chloroform-*d*, mixture of two diasteroisomers)

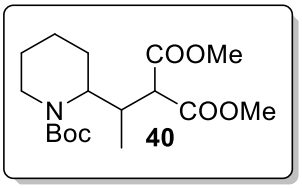




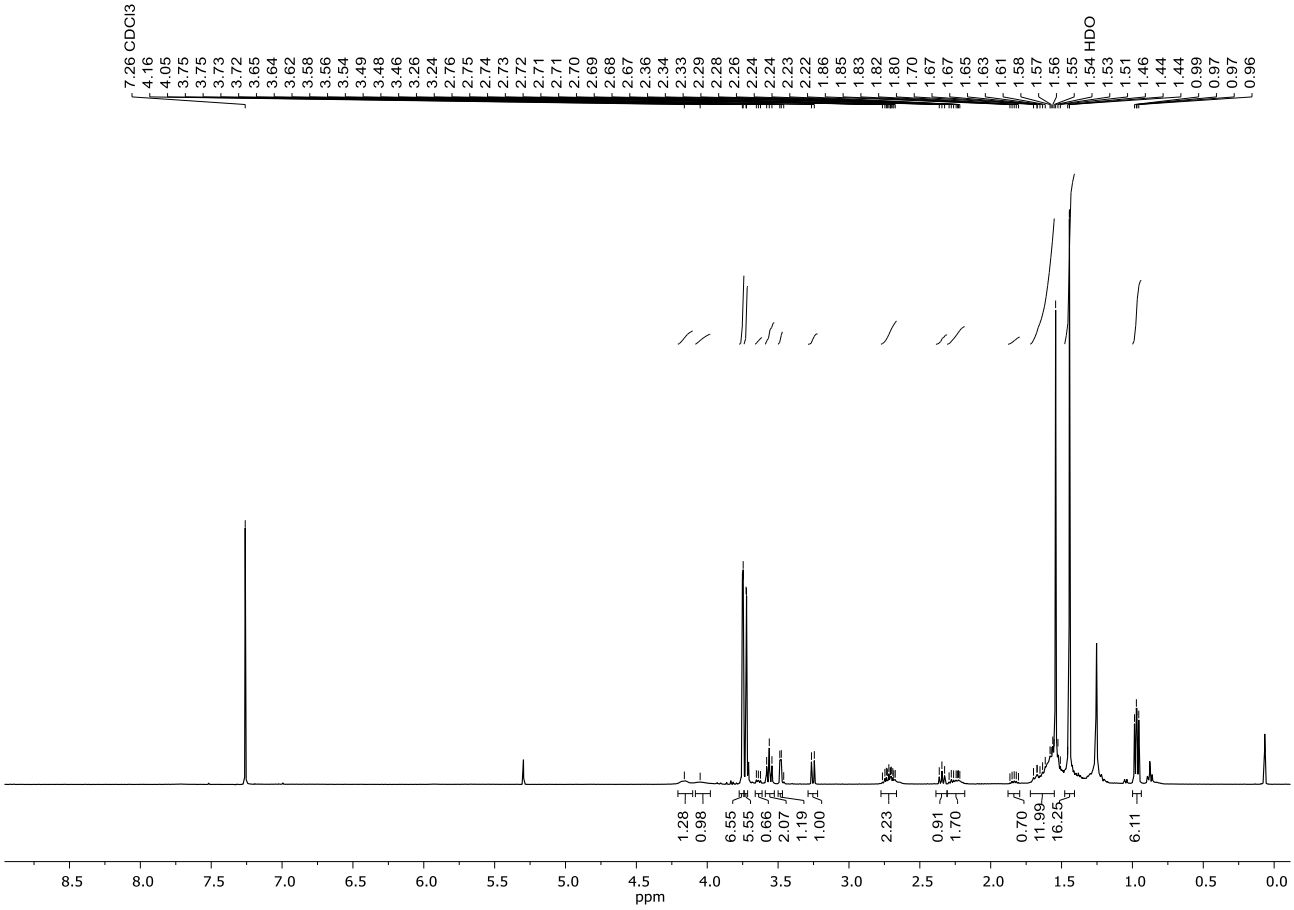
39

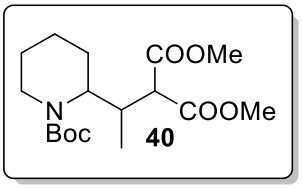
^{13}C-NMR (75 MHz, chloroform-*d*, mixture of two diasteroisomers)



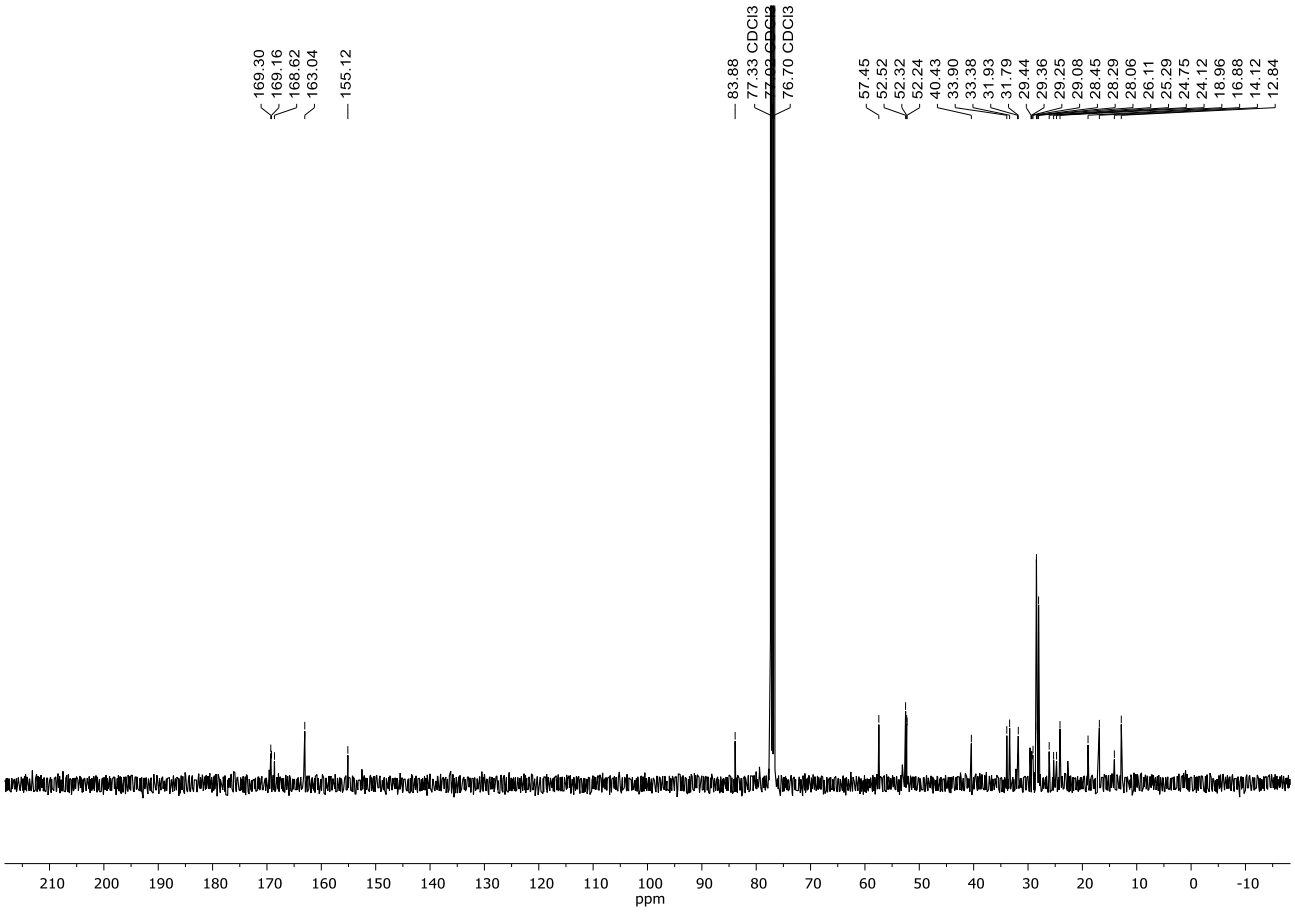


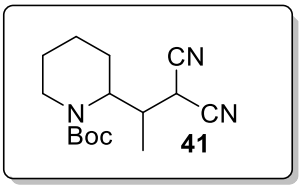
^{1}H-NMR (400 MHz, chloroform-*d*, mixture of two diasteroisomers)



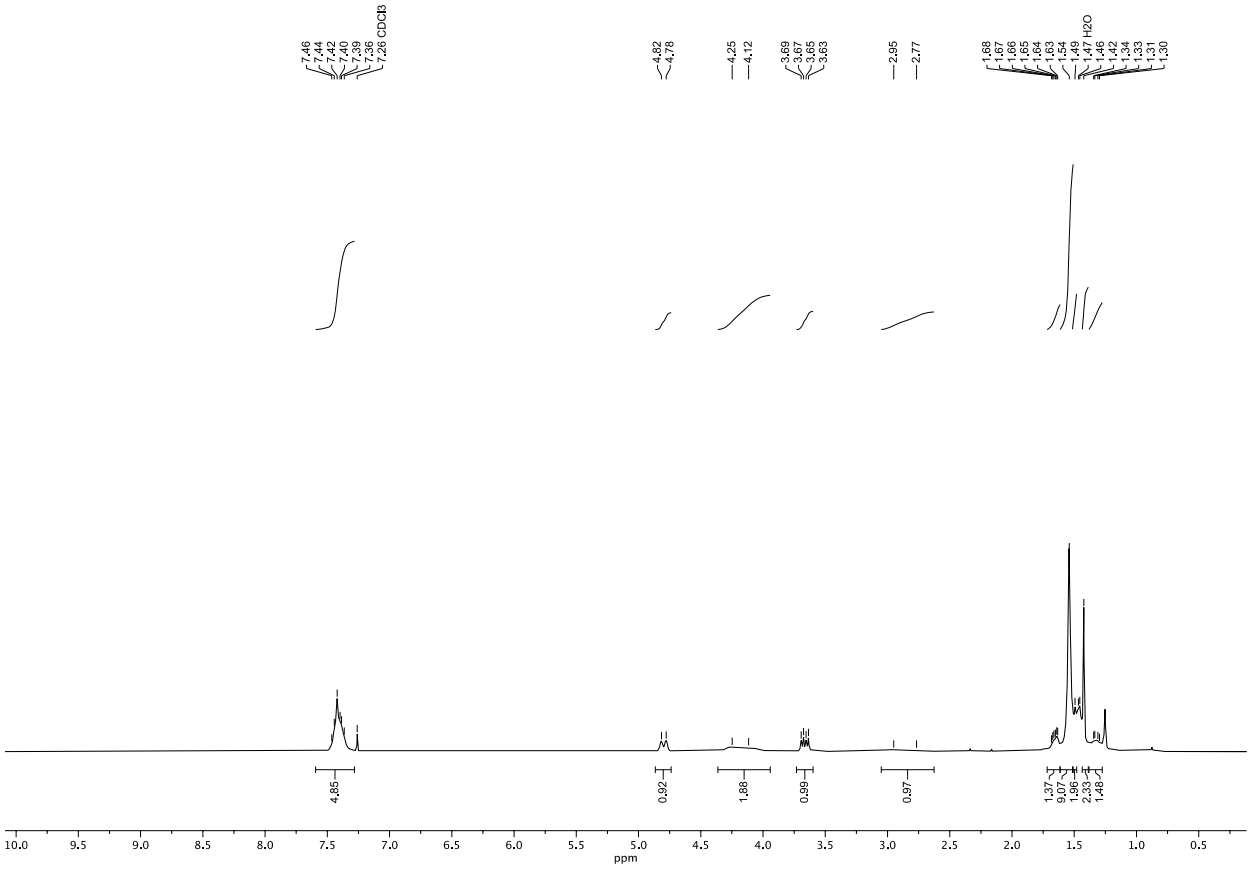


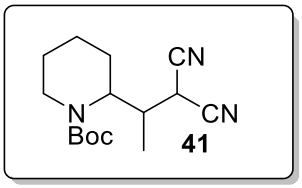
^{13}C-NMR (100 MHz, chloroform-*d*, mixture of two diasteroisomers)



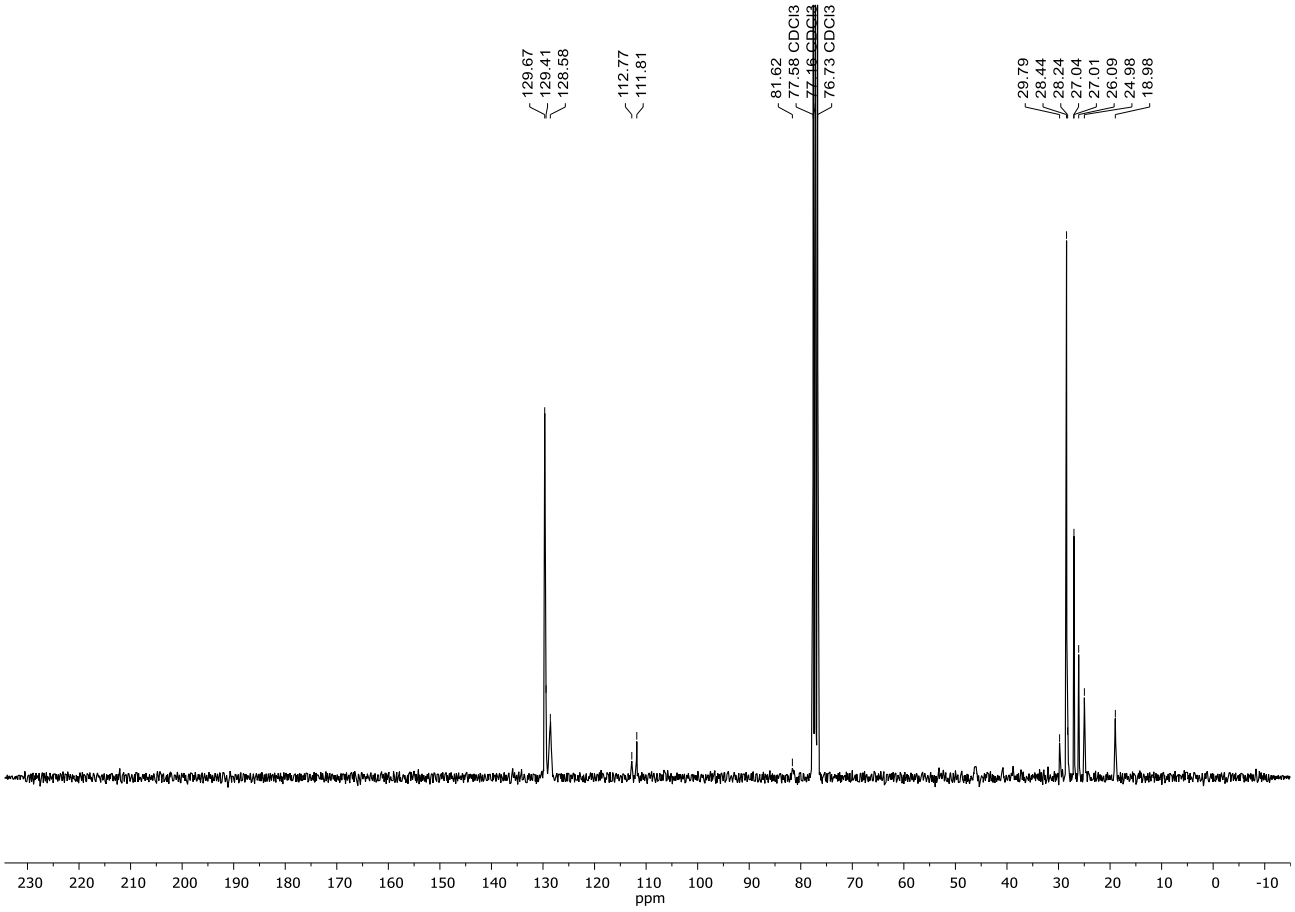


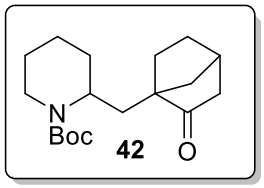
^{1}H-NMR (300 MHz, chloroform-*d*, mixture of two diasteroisomers)



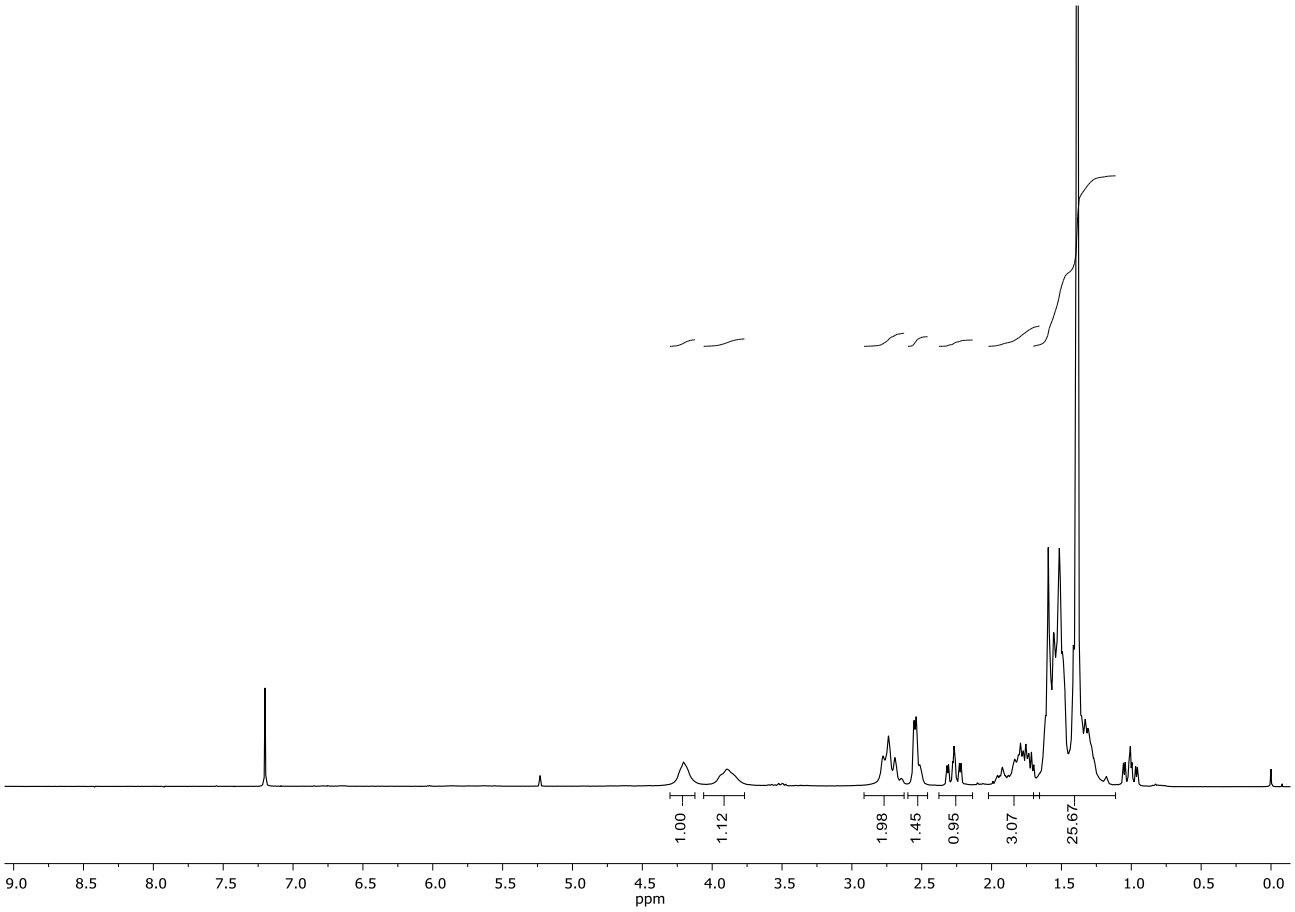


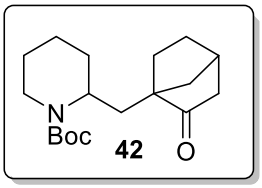
^{13}C-NMR (75 MHz, chloroform-*d*, mixture of two diasteroisomers)



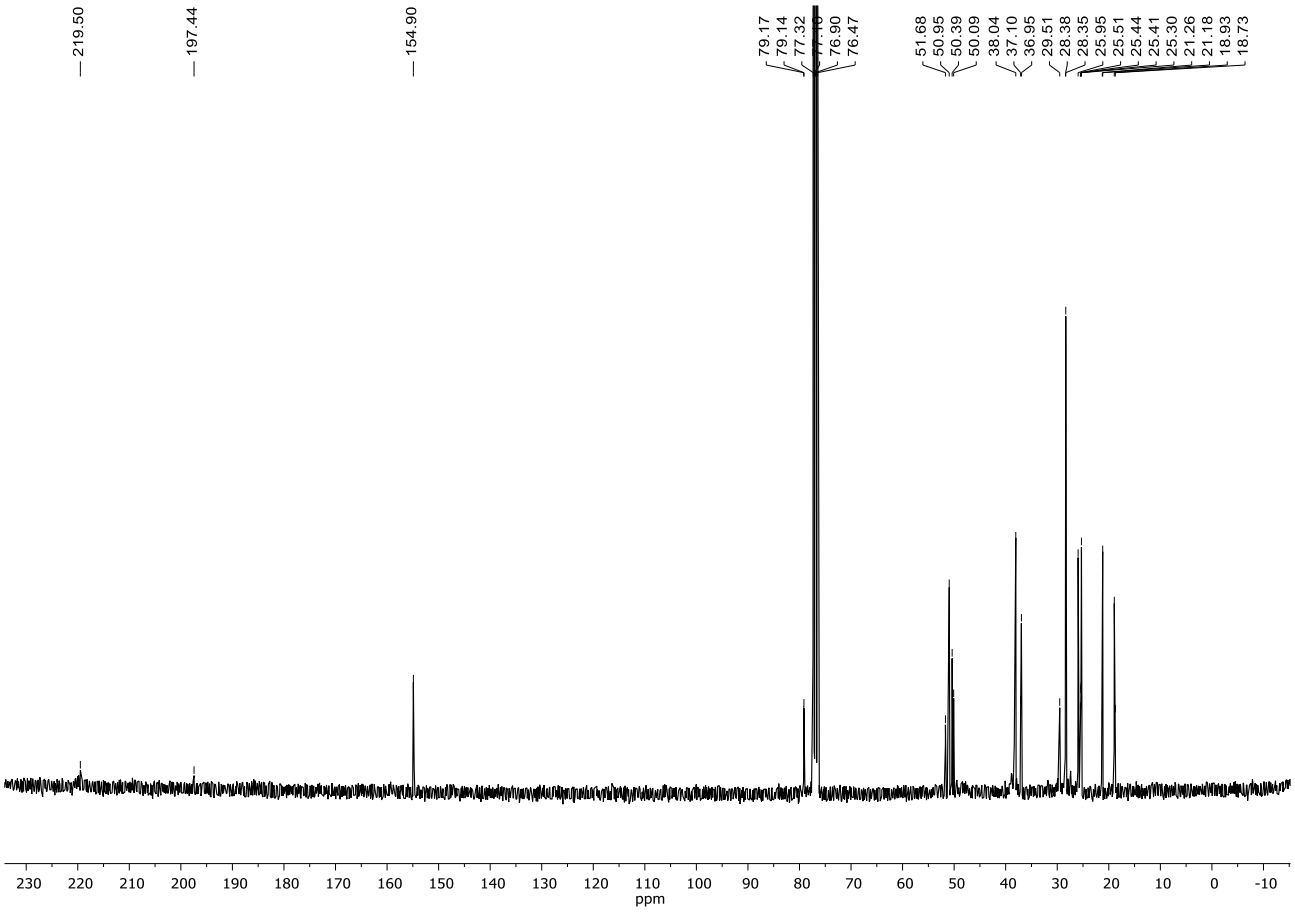


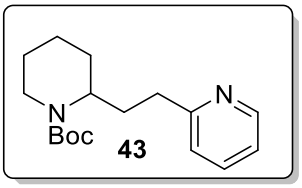
^{1}H-NMR (300 MHz, chloroform-*d*, mixture of two diasteroisomers)



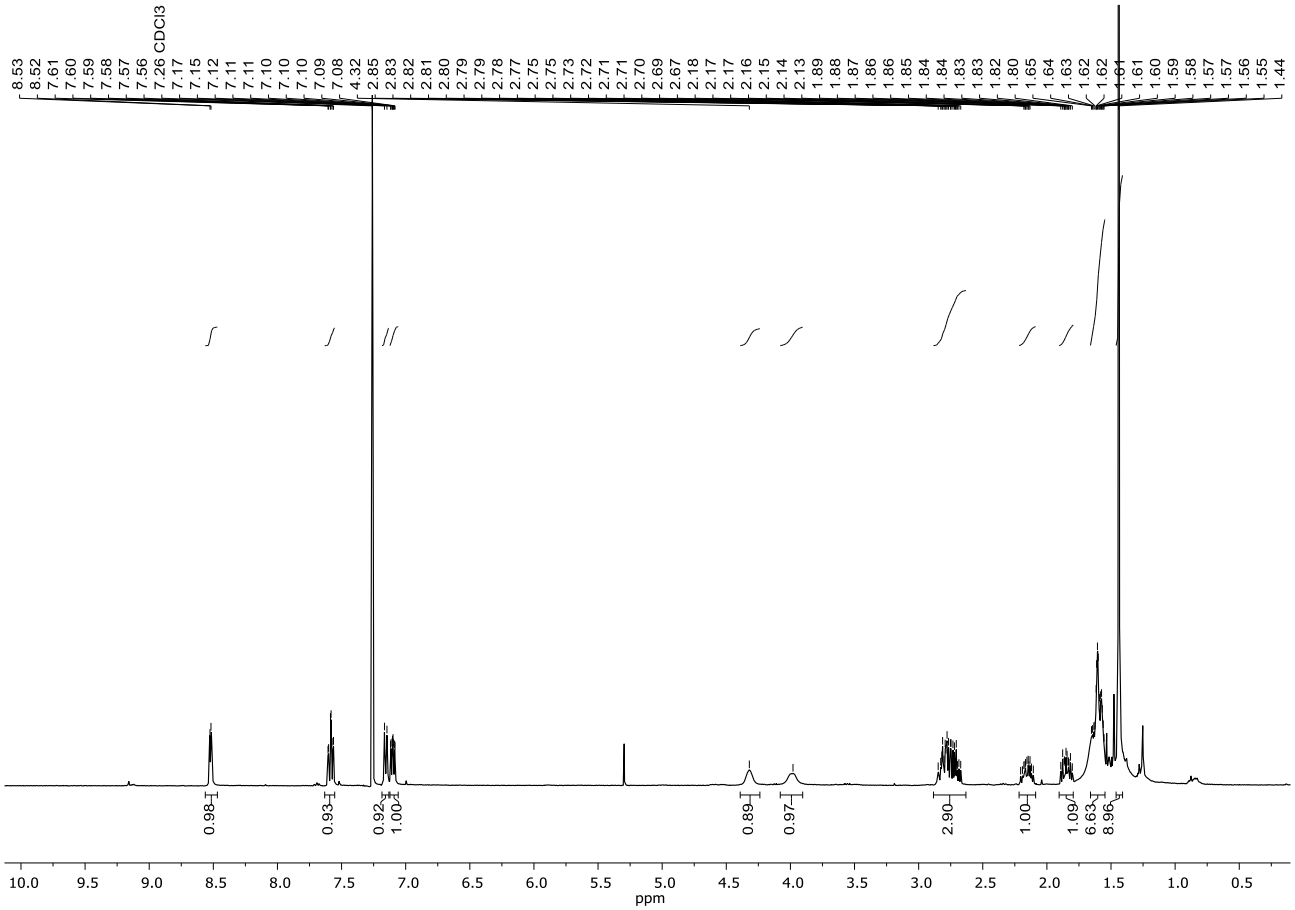


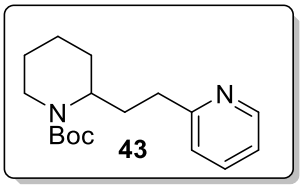
^{13}C-NMR (75 MHz, chloroform-*d*, mixture of two diasteroisomers)





^{1}H-NMR (400 MHz, chloroform-*d*)





^{13}C-NMR (100 MHz, chloroform-*d*)