Supporting Information for

Ozone-Mediated Amine Oxidation: A Solvent Free, Flow-Chemistry Approach

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General Experimental Details:

Unless otherwise indicated, reagents were obtained from Sigma Aldrich, Fisher Scientific or Combi-Blocks and used as received. Silicycle F60 40-63 µm silica gel was used for amine suspension and column chromatography. The silica gel was stored at room temperature and dried in a 140 °C oven for ~24 hours prior to use in the packed bed reactor. Analytical thin layer chromatography (TLC) was conducted using aluminum-backed EMD Millipore Silica Gel 60. Visualization of developed plates was performed under UV light (254 nm) and/or using $KMnO_4$ stains.

Instrumentation and Flow Reactor Details

^{1}H NMR and ^{13}C NMR were recorded on a Bruker AVANCE 400 MHz spectrometer and referenced to residual solvent signals. Data for ^{1}H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant (Hz), integration. Yields for optimization were determined by NMR or GC analysis of the crude reaction mixture using 1,3,5-trimethoxybenzene as an internal standard. FTIR spectra were collected on a Varian 640-IR spectrometer equipped with an attenuated total reflectance accessory (ATR, Pike MIRacle) in the 4000-400 cm^{-1} range with 64 scans per sample. Unless otherwise indicated, continuous flow experiments were performed using 1/16" O.D., 1.0 mm I.D. PFA. PEEK fittings were used for all PFA tubing. PEEK fittings and parts were purchased from Upchurch Scientific. Vapourtec V3 pumps with blue tubing and built-in Tee joints were used for pumping and reagent selection.

General Procedure A:

To a clean round-bottom flask was added the corresponding amine (0.3 mmol), followed by pentane (20 mL). 3 g of silica gel (stored in a 140 °C oven overnight) was added to form a slurry (0.1 mmol of substrate per gram of silica). The solvent was slowly evaporated under reduced pressure (no lower than 800 mbar to avoid silica bumping as it dries) to yield silica gel loaded with starting material. 1 g of this impregnated silica gel was added to an omnifit column measuring 150 mm long with an I.D of 10 mm (Part number# 006EZ-10-15-AF). The silica plug measured around 3 cm long inside the column. This assembly was connected to a Vapourtec E-series (easy-Medchem) reactor and cooled down to -60 °C using the with Vapourtec Cooling Module (#50-1314) under a constant flow of oxygen through the reaction vessel (10 psi). Once the reactor was cooled, the in-line ozone generator (Oxidation Technologies, VMUS-DG1) was turned on. This was operated at maximum power with an approximate flow rate of 0.12 L/min of oxygen. This corresponds to an approximate production of 1.75 g/h or 0.6 mmol of O_3 per minute. Ozone exit tubing from the reaction led to a solution of sodium metabisulfite in water to quench any remaining excess.

After 15 minutes, the ozone generation was turned off while allowing oxygen to continue to flow. After the remaining ozone is purged from the silica gel (~10 seconds; loss of blue color), the reactor is allowed to warm to room temperature. Silica gel was harvested from the reactor and the column was cleaned with acetone before repeating above procedure again. Two combined 1 g reactions were directly dry-loaded to a column of silica gel for purification following a pentane wash to remove excess grease.

General Procedure B:

Identical to General Procedure A, but reactor is cooled to -20 °C and ozone is applied for 60 mins.

Reaction Optimization:

We began our exploration into this topic by using dodecylamine as a model substrate using the reactor setup illustrated in Figure S1 below. With this, various ozonation times and temperatures were screened to determine optimal conditions (Table S1). It was previously known that these types of oxidations have taken place at -78 °C, but our system allowed us greater control over specific temperatures than a simple dry ice bath. It was found that -60 °C gave the best balance between selectivity while still enabling the desired reaction to proceed quickly. The next variable to be optimized was time, and 15 minutes proved to be the point at which yield was highest, even though some starting material was still recovered. If the reaction is left to continue for longer, degradation pathways will begin to outcompete product formation.

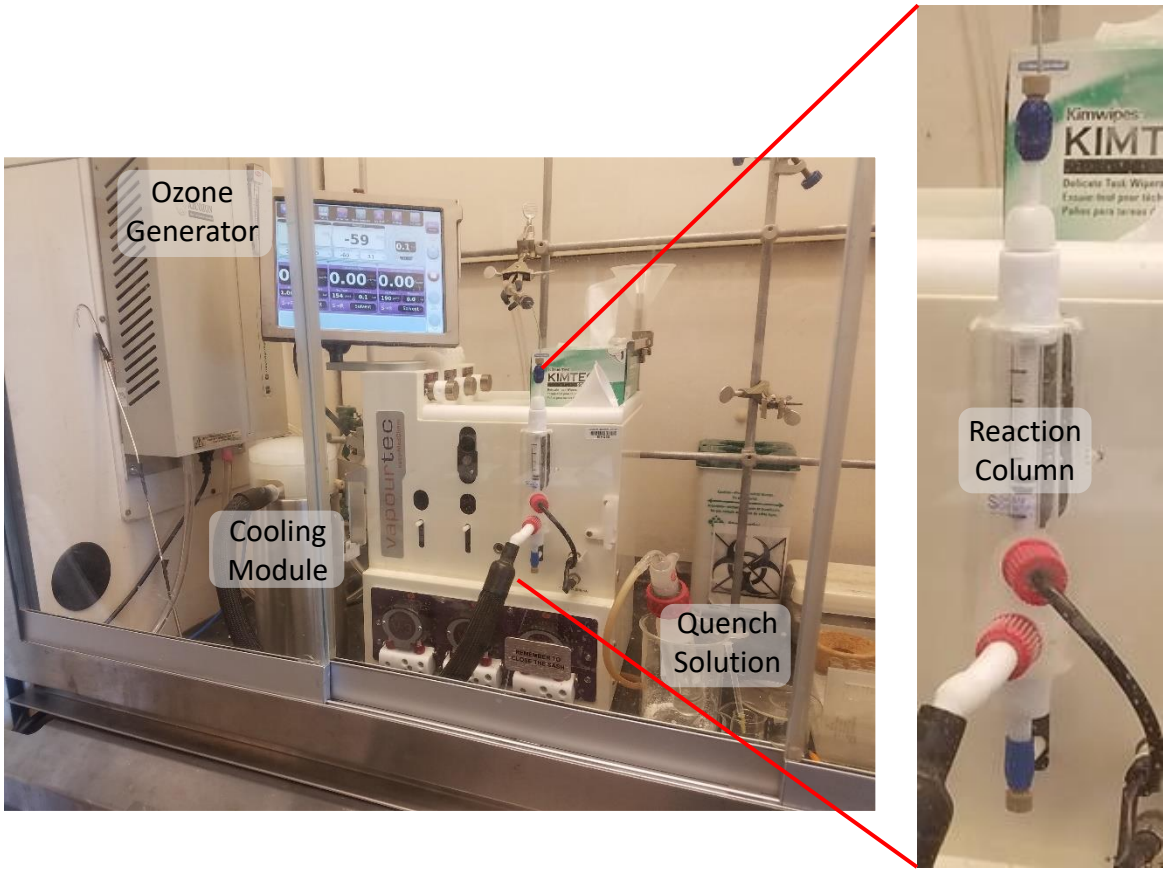
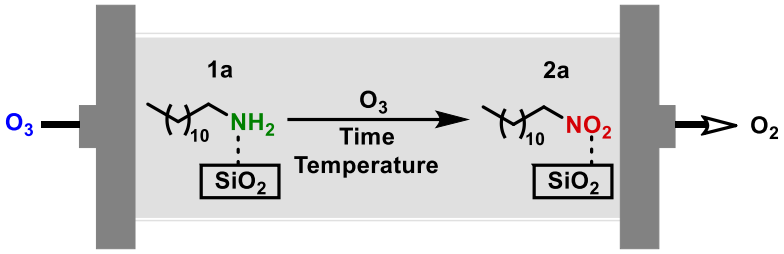


Figure S1: Photograph of the complete reactor setup

Table S1: Optimization of Ozonation Time and Temperature[a]

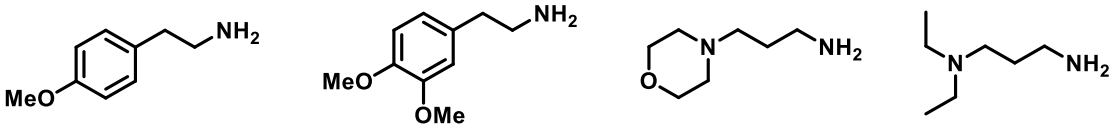


Entry	Temperature (°C)	Time (min)	Yield[b] (%)	Amine Recovery[b]
1	-40	10 s	11	47
2	-40	15	51	25
3	-40	30	42	0
4	-60	5	50	32
5	**-60**	**15**	**62**	**25**
6	-60	30	49	13
7	-70	20	29	n/a

[a]Reactions were run on a 0.1 mmol scale. [b]Yields were obtained via crude NMR spectroscopy using 1,3,5 trimethoxybenzene as an internal standard.

Failed Scope Examples:

Notably, two classes of attempted substrates did not yield any product: Electron rich aromatic rings, and substrates containing multiple amines. Our hypothesis for the former is that the increased electron density of the aromatic ring make it more prone to reaction with the ozone. The latter class of substrates failing is most likely due to multiple equivalents of ozone being employed in our method. Each nitrogen can undergo this reaction independently of one another, and so multiple sites of oxidation could increase chances of molecule degradation. As such, no product, starting material or discernable side product were observed with either substrate.



1 **0%** **2** **0%** **3** **0%** **4** **0%**

Figure S2: Failed Scope Examples

Flow Automation Optimization:

The reactor cycling set-up described in manuscript Scheme 4 is outlined in more detail in **Figure S3** below. Figure S3A illustrates the flow path (in red) for normal reactor operation for ozonation. Oxygen flow from a gas tank flows through the ozone generator to the reactor inlet. Passing through the reactor, it exits and flows to a solution of aqueous sodium metabisulfite. **Figure S3B** illustrates the flow paths for loading the amine solution onto the column (red path), and flushing material off the column (blue to red path). **Table S2** illustrates reproducibility in triplicate (Entry 1 a-c). Alternate loading conditions were tested using pentane as the solvent (Entry 2), which resulted in a 0% yield due to a lack of even dispersion of the amine along the silica length. If the column is not allowed to fully dry before reaction is started, a drastically reduced yield is also observed (Entry 3). Drying the column using either air pressure or heat alone give reasonable yields (Entry 4 and 5).

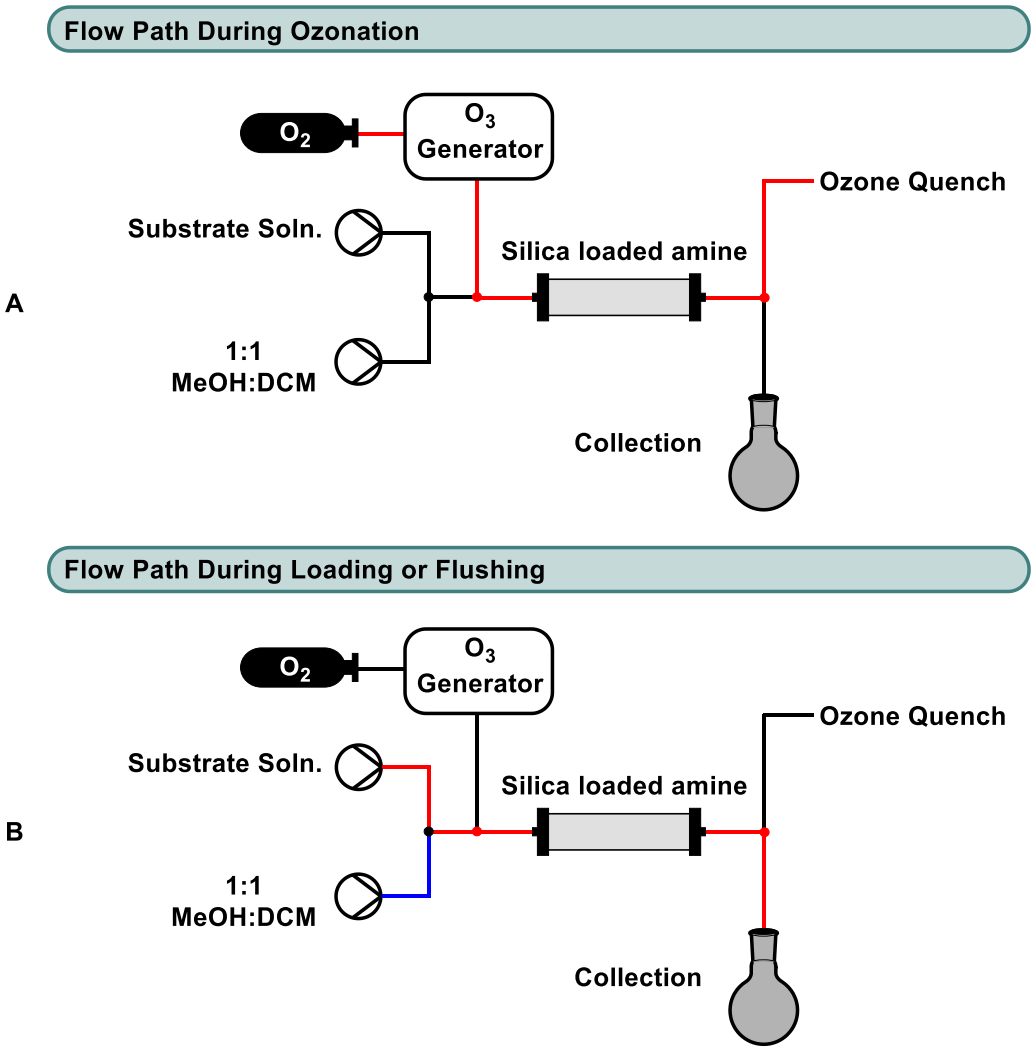
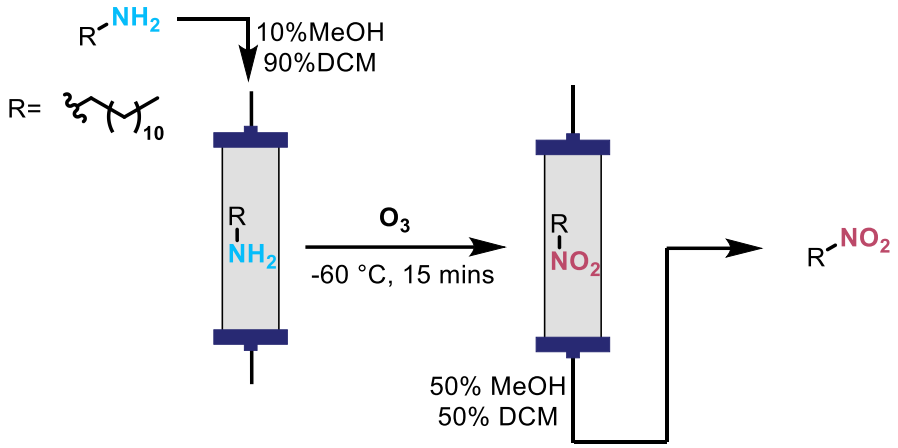


Figure S3: Schematic of Complete Flow Reactor Set Up

Table S2: Optimization of Automation Process[a]



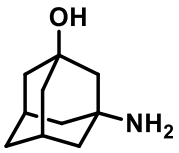
Entry	Deviation from Standard Conditions	Yield (%)[b]
1a	None – first reproduction	60[c]
1b	None – second reproduction	59[c]
1c	None – third reproduction	62[c]
2	Loaded amine in pentane	0
3	Column not fully dried	20
4	Column dried using heat only	55
5	Column dried using air pressure only	57

[a] Reactions performed on a 0.1 mmol scale using dodecylamine.

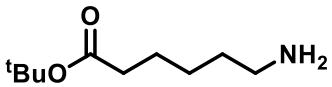
[b] Yields obtained via crude NMR spectroscopy using 1,3,5 trimethoxybenzene as an internal standard.

[c] Yield 1a, 1b, and 1c are performed using identical conditions in triplicate.

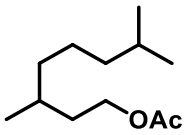
Starting Material Synthesis:



3-Aminoadamantol (1m): Synthesized according to literature precedent.[1] Adamantanamine (1 g, 5.33 mmol) was added to a pot of premixed H_2SO_4 (10.3 mL) and nitric acid (1 mL) at 0 °C and yielded the title product as a white solid (0.6987 g , 78%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 2.28-2.19 (m, 4H), 1.68-1.59 (m, 4H), 1.56 (s, 2H), 1.52-1.48 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 69.8, 53.9, 50.4, 44.9, 44.1, 34.8, 31.1.

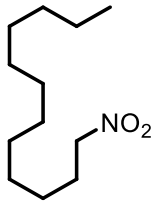


6-aminohexanoic acid tert-butyl ester (1h): Synthesized according to literature precedent.[2] 6-aminohexanoic acid (0.6560 g, 5.0 mmol) was mixed with thionyl chloride (1.63 mL, 22.5 mmol) and *t*-BuOH (2.7 mL) to yield the title compound as an off white solid (0.4064 g, 44%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 2.63 (t, J= 7.0 Hz, 2H), 2.19 (s, 2H), 2.13 (t, J= 7.4 Hz, 2H), 1.51 (quin, J= 7.4 Hz, 2H), 1.42 (quin, J= 7.4 Hz, 2H), 1.35 (s, 9H), 1.31-1.22 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 79.4, 41.6, 35.3, 32.7, 28.0, 26.2, 24.7.

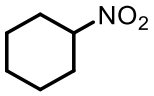


3,7-dimethyloctyl acetate (5a): Synthesized according to literature precedent.[3] 3,7-Dimethyloctanol (0.96 mL, 5.0 mmol) and acetyl chloride (0.43 mL, 6.0 mmol) were mixed to yield the title compound as a colourless oil (0.7712 g, 77%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 4.15-4.03 (m, 2H), 2.04 (s, 3H), 1.70-1.60 (m, 1H), 1.57-1.48 (m, 2H), 1.47-1.37 (m, 1H), 1.35-1.08 (m, 6H), 0.89 (d, J=6.5 Hz, 3H), 0.86 (d, J=6.6 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 63.1, 39.1, 37.1, 35.5, 29.8, 27.9, 24.6, 22.7, 22.6, 21.0, 19.5.

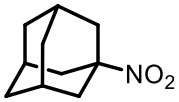
Characterization Data for Products:



1-Nitrododecane (2a): The title compound was synthesized according to general procedure A using dodecylamine (0.0370 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (5% EtOAc/Hexanes, R_f: 0.43). The title compound was isolated as a yellow oil (0.013 g, 60%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 4.38 (t, *J* = 7.1 Hz, 2H), 2.00 (quin, *J* = 7.1 Hz, 2H), 1.42-1.20 (m, 18H), 0.88 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 75.7, 31.9, 29.7, 29.6, 29.4, 29.3, 29.2, 28.8, 27.4, 26.2, 22.7, 14.1. Spectral data was consistent with literature reports.[4]

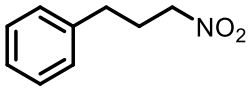


Nitrocyclohexane (2b): The title compound was synthesized according to general procedure A using cyclohexylamine (0.0298 g, 0.3 mmol) and 3 g of dry silica gel. After reaction, the product was isolated using flash chromatography (10% EtAOc/Hexanes, R_f: 0.44). The title compound was isolated as a yellow oil (0.0216 g, 56%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 4.36 (tt, *J*= 10.7, 4.0 Hz, 1H), 2.26-2.19 (m, 2H), 1.91-1.82 (m, 4H), 1.69-1.62 (m, 1H), 1.39-1.23 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 84.6, 30.9, 24.7, 24.1. Spectral data was consistent with literature reports.[5]

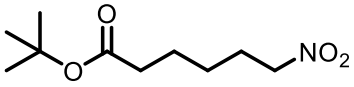


Nitroadamantane (2c): The title compound was synthesized according to general procedure A using adamantylamine (0.0306 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (5% EtOAc/Hexanes, R_f: 0.44). The title compound was isolated as a yellowish

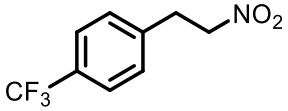
solid (0.0290 g, 80%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 2.23 (s, 3H), 2.21 (s, 6H), 1.77-1.66 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 84.7, 40.8, 35.5, 29.7. Spectral data was consistent with literature reports.[6]



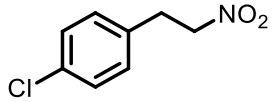
(3-Nitropropyl)benzene (2d): The title compound was synthesized according to general procedure A using 3-phenylpropylamine (0.0270 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (5% EtOAc/Hexanes, R_f: 0.26). The title compound was isolated as a pale-yellow oil (0.0283 g, 85%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.34-7.16 (m, 5H), 4.37 (t, *J*= 6.9 Hz, 2H), 2.73 (t, *J*= 7.4 Hz), 2.37-2.30 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.5, 128.7, 128.4, 126.6, 74.7, 32.3, 28.8. Spectral data was consistent with literature reports.[7]



6-Nitrohexanoic Acid *Tert*-Butyl Ester (2e): The title compound was synthesized according to general procedure A using *tert*-butyl-6-aminohexanoate (0.0375 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (10% EtOAc/Hexanes, R_f: 0.21). The title compound was isolated as a white solid (0.0294 g, 70%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 4.38 (t, *J*= 7.0 Hz, 2H), 2.23 (t, *J*= 7.3 Hz, 2H), 2.06-1.99 (m, 2H), 1.67-1.58 (m, 2H), 1.44 (s, 9H), 1.42-1.39 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 80.4, 75.4, 35.0, 28.1, 27.1, 25.7, 24.2. IR: 1722.306, 1554.190, 1264.298, 1151.132, 730.028. HRMS (ESI+): [M+] m/z Calc'd for $C_{10}H_{19}NO_4Na$: 240.1212; Found: 240.1202.

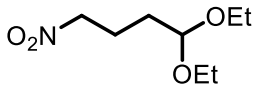


1-(2-Nitroethyl)-4-(Trifluoromethyl)benzene (2f): The title compound was synthesized according to general procedure A using 2-(4-trifluoromethylphenyl)ethylamine (0.0378 g, 0.2 mmol) using 2 g of dry silica gel. After reaction the product was isolated using flash chromatography (10% EtOAc/Hexanes, R_f: 0.33). The title compound was isolated as a pale-yellow oil (0.0325 g, 74%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.60 (d, *J*= 7.9 Hz, 2H), 7.34 (d, *J*= 7.9 Hz, 2H), 4.64 (t, *J*= 7.2 Hz, 2H), 3.38 (t, *J*= 7.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.7, 129.0, 125.9 (quart), 122.6, 75.6, 33.0. Spectral data was consistent with literature reports.[8]

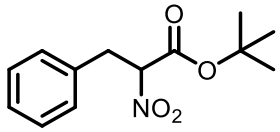


1-(2-Nitroethyl)-4-Chlorobenzene (2g): The title compound was synthesized according to general procedure A using 2-(4-Chlorophenyl)ethylamine (0.0311 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (5% EtOAc/Hex, R_f: 0.15). The title compound was isolated as a colorless oil (0.0271 g, 73%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.32-7.28 (m, 2H),

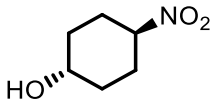
7.16-7.13 (m, 2H), 4.60 (t, *J*= 7.13 Hz, 2H), 3.29 (t, *J*= 7.13 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.1, 133.4, 129.9, 129.2, 76.0, 32.7. Spectral data was consistent with literature reports.[9]



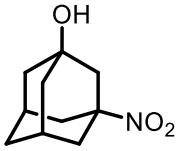
1,1-Diethoxy-4-Nitrobutane (2h): The title compound was synthesized according to general procedure A using 1,1-Diethoxy-4-Aminobutane (0.0322 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (25% EtOAc/Hexanes, R_f: 0.47). The title compound was isolated as a yellow oil (0.0352g, 92%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 4.50 (t, *J*= 5.3 Hz, 1H), 4.42 (t, *J*= 7.0 Hz, 2H), 3.68-3.60 (dq, *J*= 9.4, 7.1 Hz, 2H), 3.51-3.44 (dq, *J*= 9.4, 7.1 Hz, 2H), 2.13-2.06 (m, 2H), 1.73-1.68 (m, 2H), 1.20 (t, *J*= 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 102.0, 75.4, 61.7, 30.3, 22.6, 15.3. Spectral data was consistent with literature reports.[10]



***Tert*-Butyl Nitrophenylpropanoate (2i):** The title compound was synthesized according to general procedure A using phenylalanine *tert*-butyl ester (0.0443 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated used flash chromatography (5% EtOAc/Hexanes, R_f: 0.35). The title compound was isolated as a white solid (0.0333 g, 66%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.33-7.20 (m, 5H), 5.25 (dd, *J*= 9.3, 5.9 Hz), 3.55-3.40 (m, 2H), 1.46 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9, 134.3, 128.9, 128.8, 127.7, 89.9, 84.8, 36.2, 27.7. IR: 2982.828, 1742.389, 1560.800, 1455.847, 1370.294, 1148.553. HRMS (ESI-H): $[M-H]^-$ m/z Calc'd for $C_{13}H_{16}NO_4$: 250.1079; Found: 250.1075.

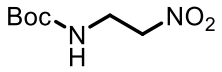


Trans-4-Nitrocyclohexanol (2j): The title compound was synthesized according to general procedure A using trans-4-aminocyclohexanol (0.0230 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (50% EtOAc/Hexanes, R_f: 0.31). The title compound was isolated as white needle-like crystals (0.0167 g, 58% yield). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 4.39 (tt, *J*=10.8, 4.1 Hz, 1H), 3.76 (tt, *J*=9.9, 4.1 Hz, 1H), 2.35-2.29 (m, 2H), 2.11-2.05 (m, 2H), 2.02-1.91 (m. 2H), 1.47-1.38 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 83.3, 68.3, 32.3, 28.2. Spectral data was consistent with literature reports.[11]

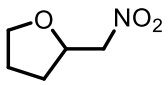


1-Nitro-3-Adamantol (2k): The title compound was synthesized according to general procedure A using 3-Aminoadamantol (0.0335 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (30% EtOAc/Hexanes, R_f: 0.20). The title compound was isolated as colorless needle-like crystals (0.0227 g, 58%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 2.46-2.41 (m, 2H), 2.19 (s, 2H), 2.15-2.11

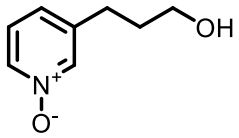
(m, 4H), 1.73 (d, *J*= 3.1 Hz, 4H), 1.61-1.58 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 86.0, 69.7, 48.0, 43.5, 39.7, 34.1, 30.8. Spectral data was consistent with literature reports.[12]



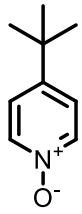
***Tert*-Butyl (2-Nitroethyl)carbamate (2l):** The title compound was synthesized according to general procedure A using *N*-Boc-Ethylenediamine (0.0320 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (20% EtOAc/Hex, R_f: 0.26). The title compound was isolated as a colorless oil (0.0193 g, 51%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 5.03 (s, 1H), 4.51 (t, *J*= 5.4 Hz, 2H), 3.69 (quart, *J*= 5.4 Hz), 1.44 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.6, 80.3, 74.2, 37.8, 28.3. IR: 3449.654, 2979.477, 2925.307, 1703.860, 1555.827, 1366.561. Spectral data was consistent with literature reports.[13]



2-(Nitromethyl)tetrahydrofuran (2m): The title compound was synthesized according to general procedure A using Tetrahydrofurfylamine (0.0202 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (20% EtOAc/Hex, R_f: 0.29). The title compound was isolated as a colourless oil (0.0174 g, 66%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 4.60-4.52 (m, 1H), 4.47-4.37 (m, 2H), 3.94-3.80 (m, 2H), 2.20-2.10 (m, 1H), 2.00-1.92 (m, 2H), 1.72-1.62 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 78.9, 75.2, 68.7, 29.0, 25.4. Spectral data was consistent with literature reports.[14]

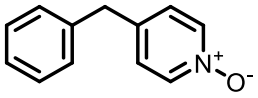


3-Pyridinylpropanol *N*-oxide (4a): The title compound was synthesized according to general procedure A using 3-pyridinylpropanol (0.0274 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (20% MeOH/EtOAc, R_f: 0.18). The title compound was isolated as a colorless oil (0.0198 g, 65%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.15 (s, 1H), 8.09-8.05 (m, 1H), 7.23-7.17 (m, 2H), 3.63 (t, *J*= 6.1 Hz, 2H) 2.70 (t, *J*= 7.7 Hz, 2H), 1.88-1.81 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.4, 139.1, 136.8, 127.5, 125.7, 60.8, 32.9, 28.9. IR: 3350.784, 2929.856, 2855.369, 1744.059, 1157.842. HRMS (ESI-TOF) m/z: $[M+Na]^+$ Calc'd for $C_8H_{11}NO_2Na$ 176.0687; Found: 176.0679.

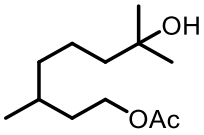


4-*Tert*-Butylpyridine *N*-oxide (4b): The title compound was synthesized according to general procedure A using 4-tertbutylpyridine (0.0270 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (10% MeOH/EtOAc, R_f: 0.16). The title compound was isolated as a colorless oil (0.3070, 92%). ^{1}H NMR: (400 MHz, $CDCl_3$) 8.11 (d, J= 7.1 Hz, 2H), 7.23 (d, J= 7.1 Hz, 2H), 1.28

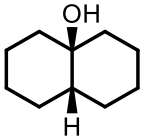
(s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 138.5, 123.1, 34.5, 30.5. Spectral data was consistent with literature reports.[15]



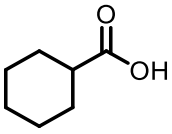
4-Benzylpyridine *N*-oxide (4c): The title compound was synthesized according to general procedure A using 4-benzylpyridine (0.0338 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (10% MeOH/EtoAc, R_f: 0.09). The title compound was isolated as a yellow oil (0.0316, 85%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.14-8.10 (m, 2H), 7.35-7.30 (m, 2H), 7.29-7.23 (m, 1H), 7.17-7.12 (m, 2H), 7.08-7.04 (m, 2H), 3.95 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.0, 139.0, 138.0, 129.0, 129.0, 128.9, 127.1, 126.3, 40.3. Spectral data was consistent with literature reports.[16]



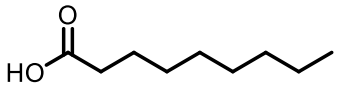
7-Hydroxy-3,7-Dimethyloctyl Acetate (6a): The title compound was synthesized according to general procedure B using 3,7-dimethyloctyl acetate (0.0401 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (10% EtOAc/Hex, R_f: 0.31). The title compound was isolated as a colourless oil (0.0199 g, 46%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 4.12-4.01 (m, 2H), 2.02 (s, 3H), 1.69-1.24(m, 10H), 1.19 (s, 6H), 1.17-1.09 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.23, 71.0, 63.0, 44.1, 37.4, 35.5, 29.8, 29.3, 29.2, 21.6, 21.0, 19.5. Spectral data was consistent with literature reports.[6]



Cis-9-Decalol (6b): The title compound was synthesized according to general procedure B using cis-decalin (0.0276 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (10% EtOAc/Hex, R_f: 0.27). The title compound was isolated as a colourless oil (0.0118 g, 39%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 1.80-1.19 (m, 18H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 71.8, 42.8, 29.7, 28.0. Spectral data was consistent with literature reports.[17]

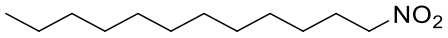


Cyclohexane Carboxylic Acid (8a): The title compound was synthesized according to general procedure B using Cyclohexylbenzene (0.0320 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (20% EtOAc/Hex, 1% AcOH, R_f: 0.32). The title compound was isolated as a colourless oil (0.0122 g, 48%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 11.1 (br, 1H), 2.33 (tt, *J*= 3.6, 11.2 Hz, 1H), 1.97-1.90 (m, 2H), 1.80-1.73 (m, 2H), 1.68-1.60 (m, 1H), 1.51-1.40 (m, 2H), 1.35-1.20 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.6, 42.8, 28.8, 25.7, 25.3. Spectral data was consistent with literature reports.[18]

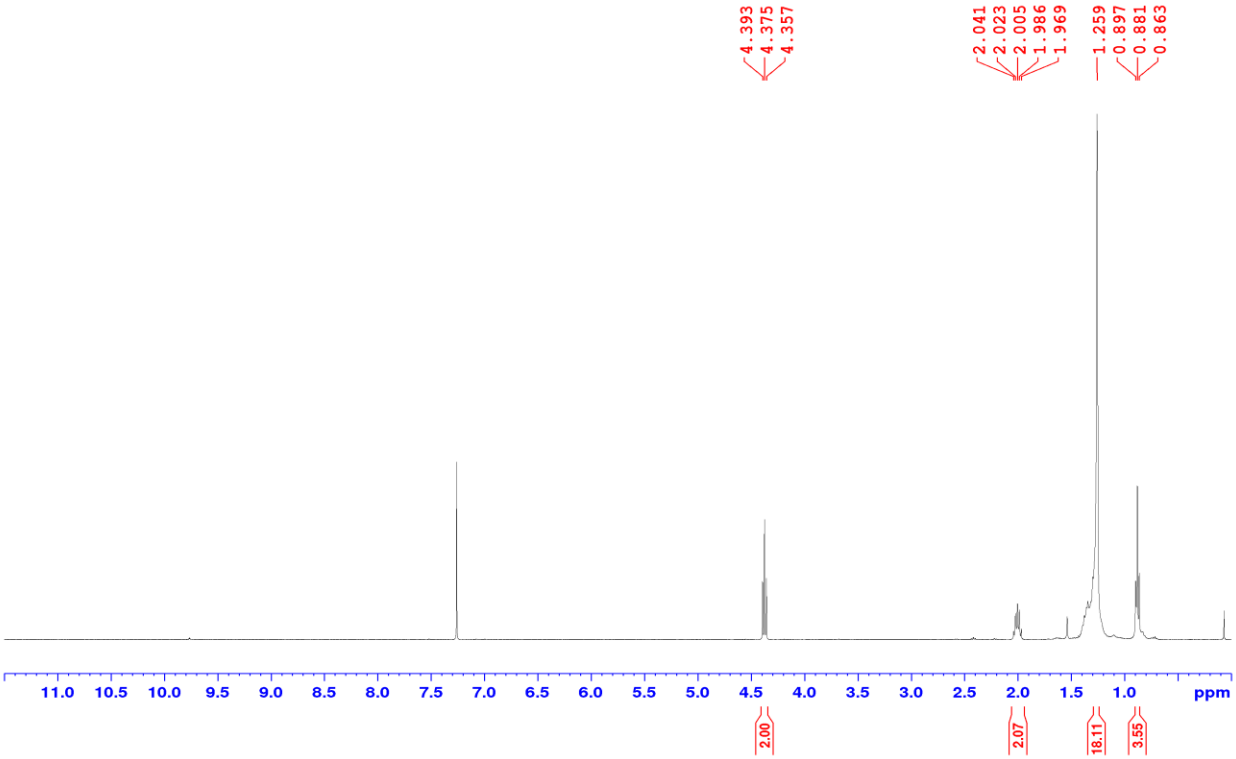


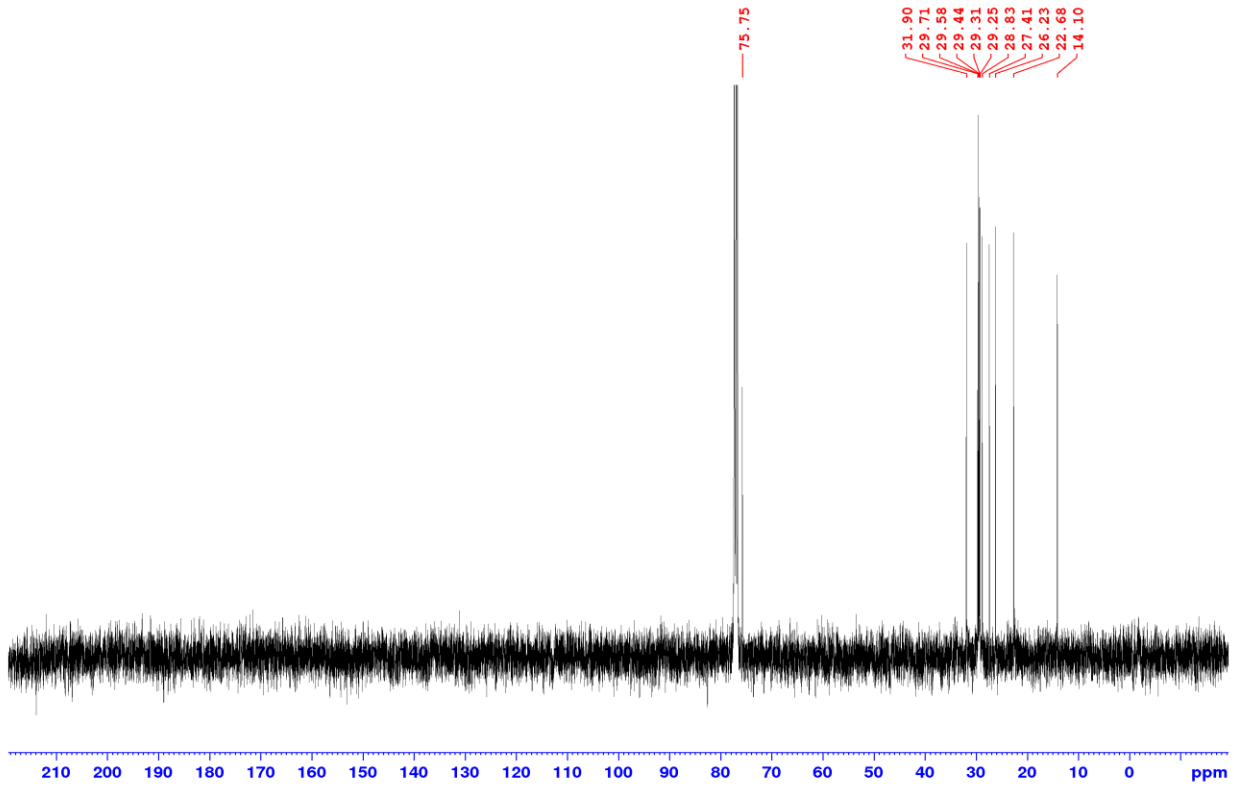
Nonanoic Acid (8b): The title compound was synthesized according to general procedure B using Octylbenzene (0.0381 g, 0.2 mmol) and 2 g of dry silica gel. After reaction, the product was isolated using flash chromatography (10% EtOAc/Hex, 1% AcOH, R_f: 0.27). The title compound was isolated as a colourless oil (0.0132 g, 46%). ^{1}H NMR: (400 MHz, $CDCl_3$) δ 2.35 (t, *J*= 7.5 Hz, 2H), 1.63 (quin, *J* = 7.5 Hz, 2H), 1.35-1.22 (m, 8H), 0.88 (t, *J*= 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.5, 34.0, 31.8, 29.2, 29.1, 24.7, 22.6, 14.1. Spectral data was consistent with literature reports.[19]

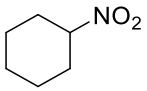
NMR Spectra:



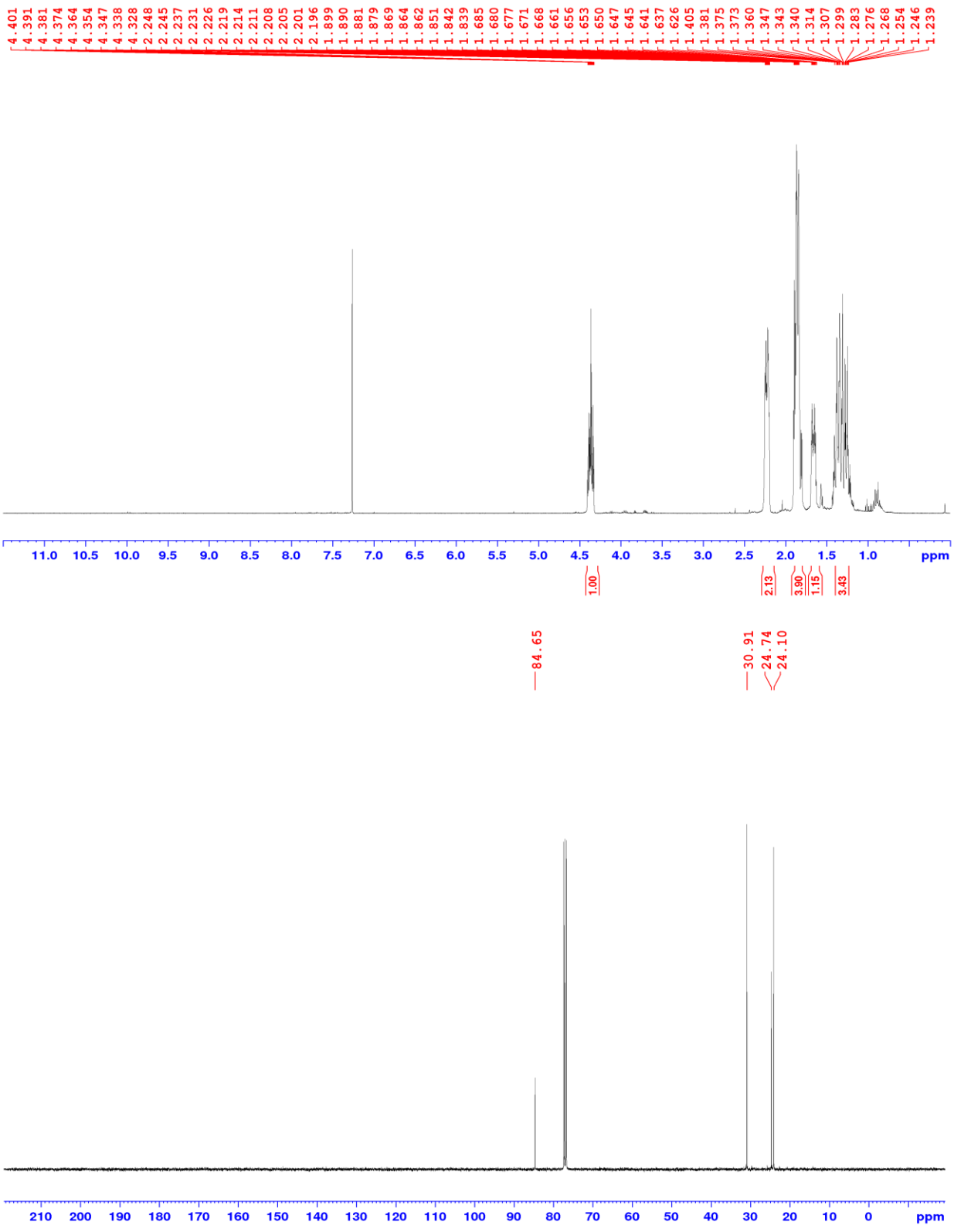
2a -Nitrododecane

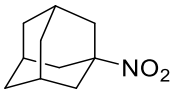




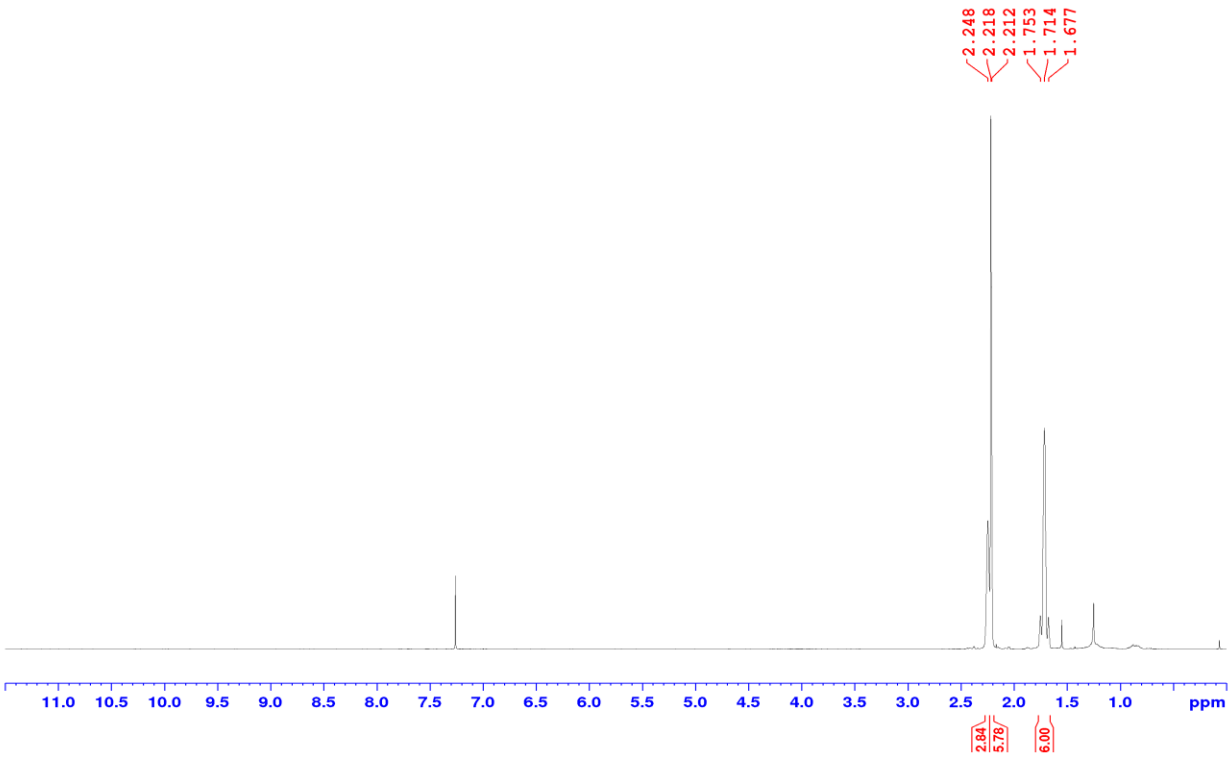


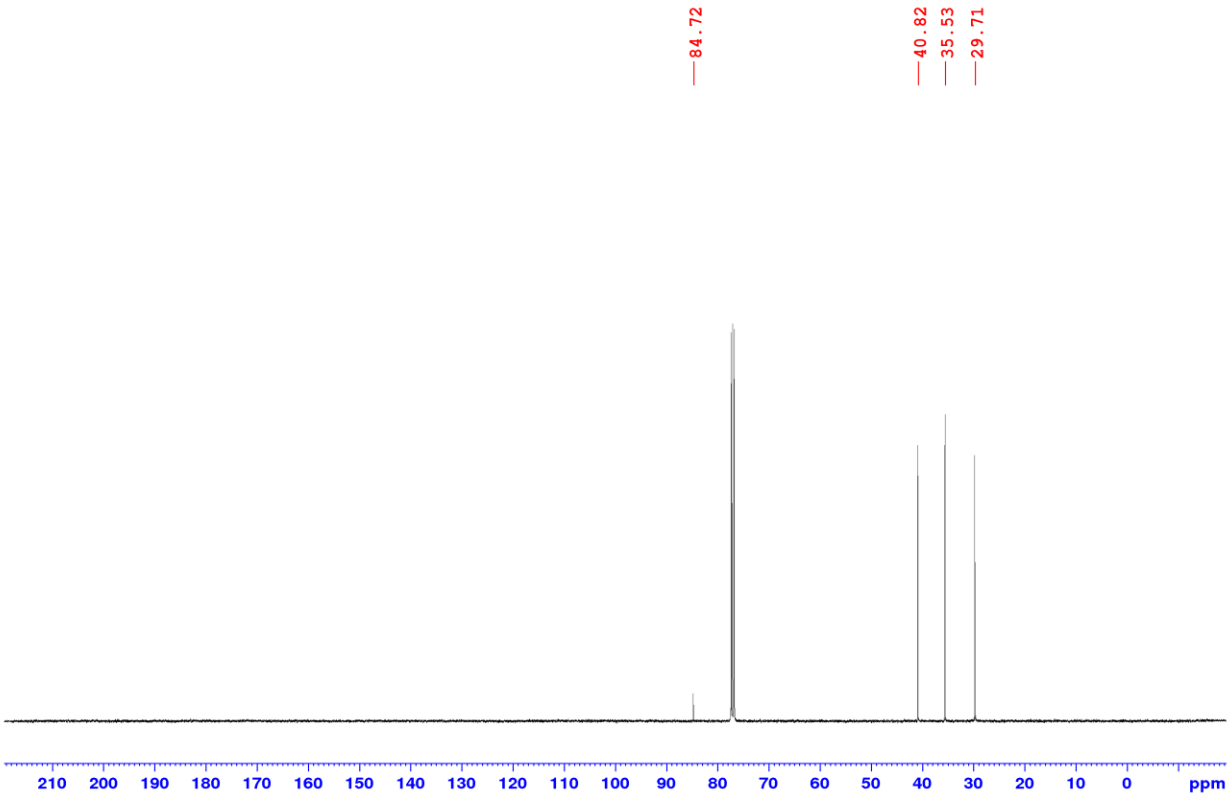
2b - Nitrocyclohexane

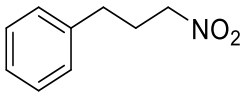




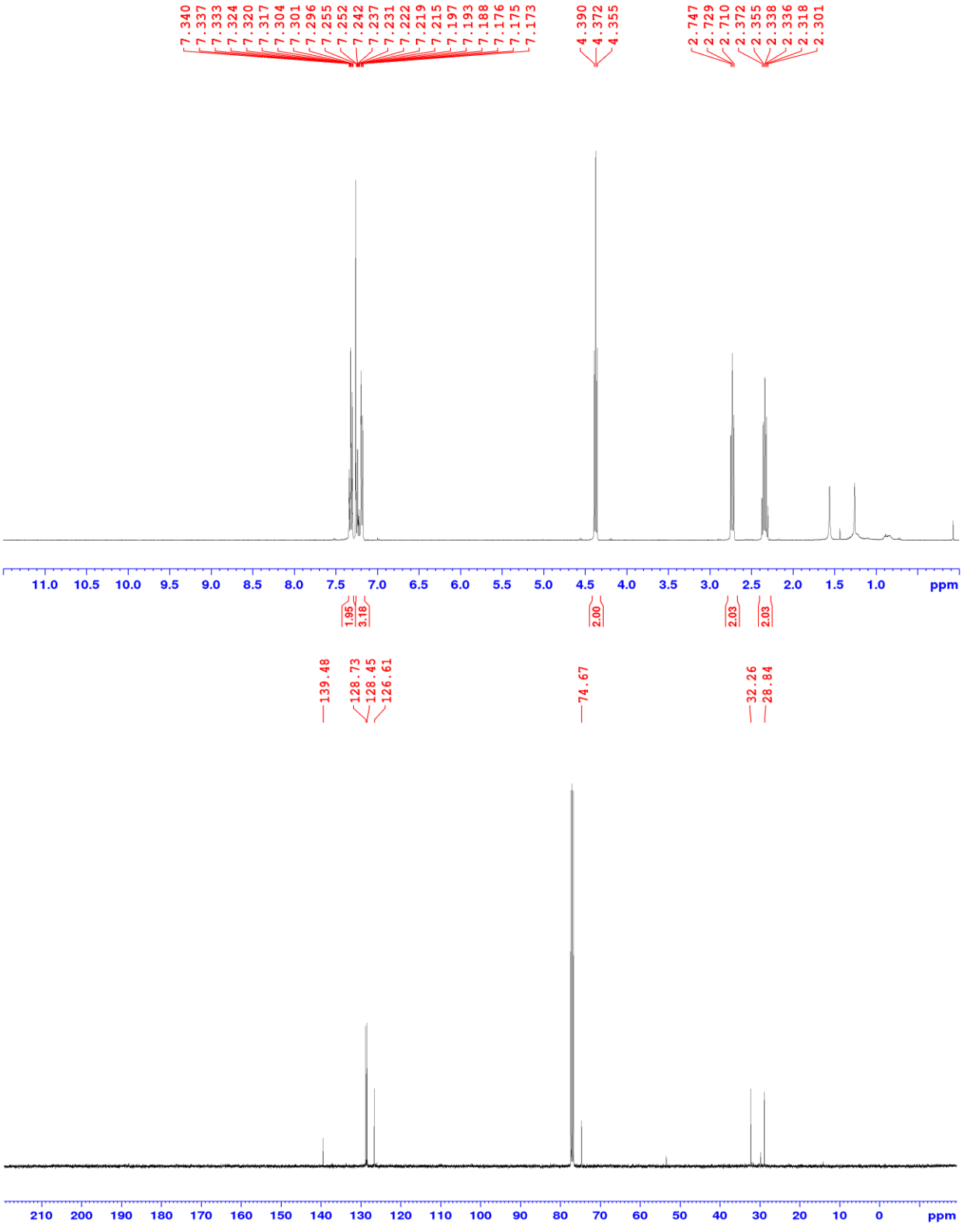
2c - Nitroadamantane

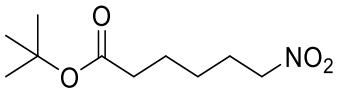




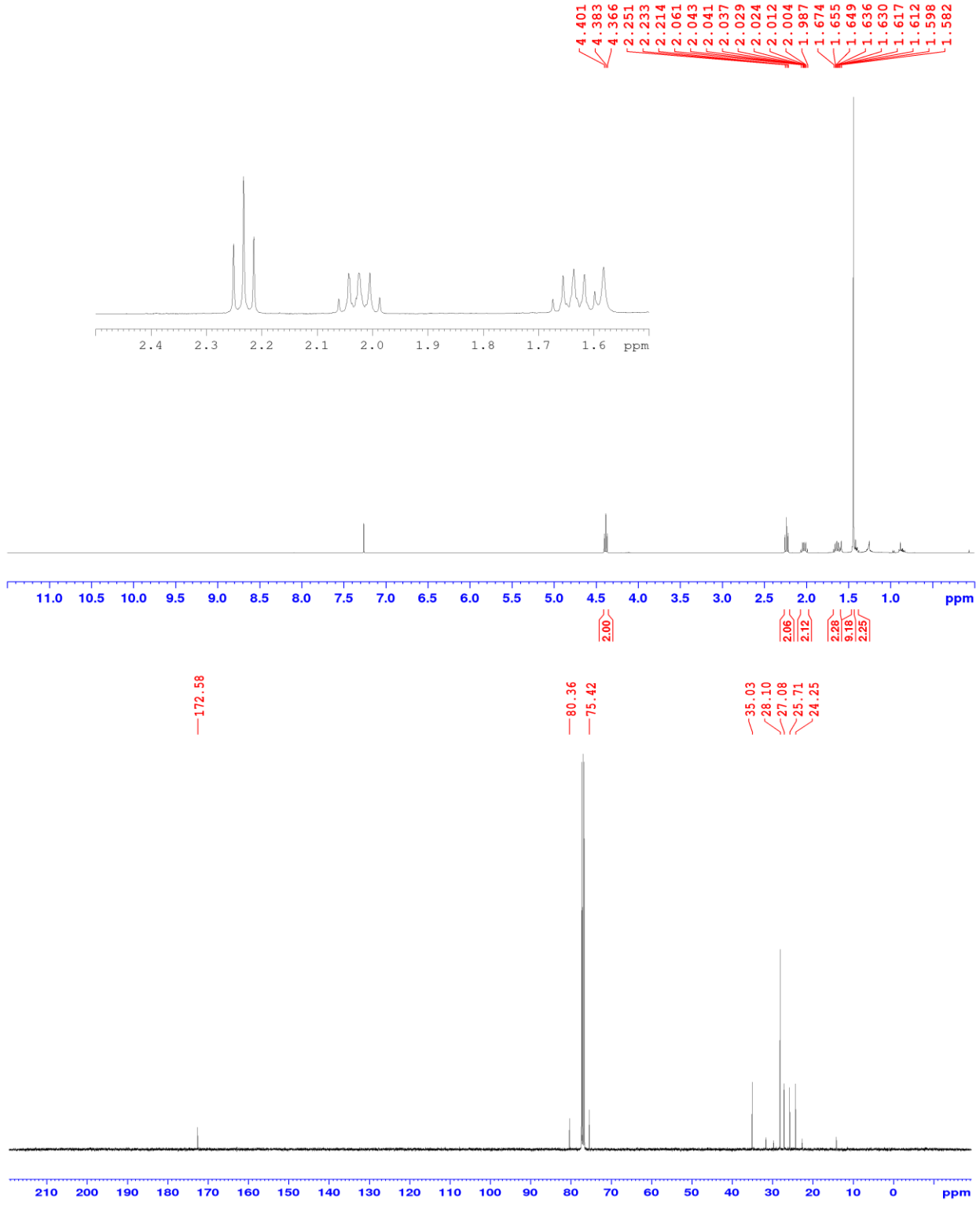


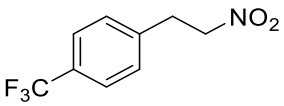
2d - (3-Nitropropyl)benzene



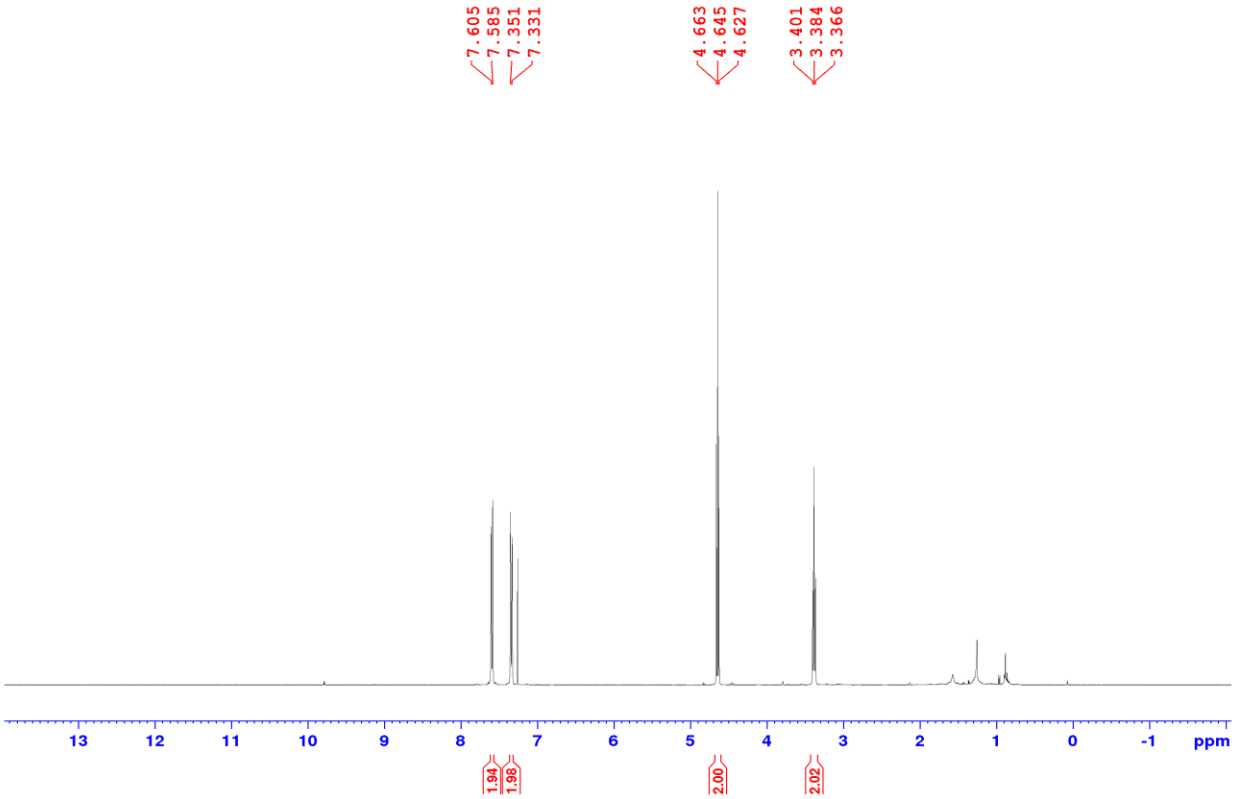


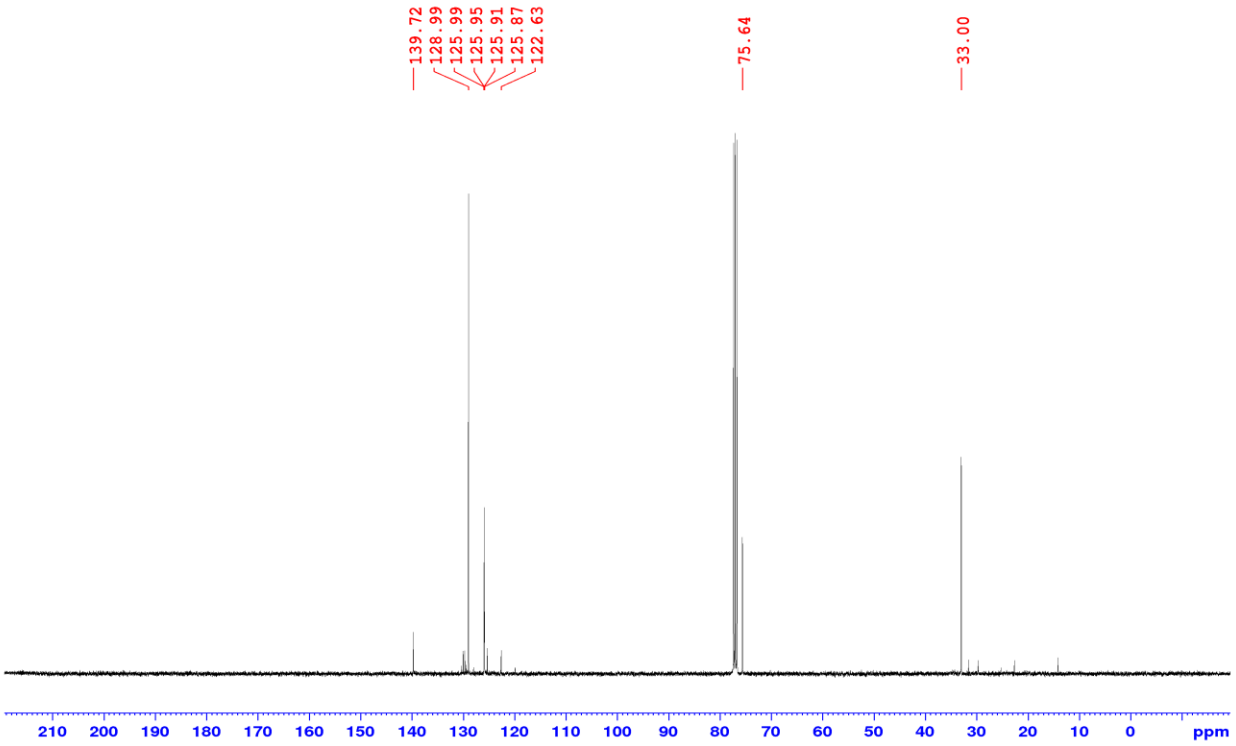
2e - 6-Nitrohexanoic Acid *Tert*-Butyl Ester

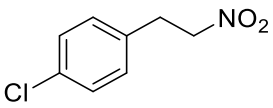




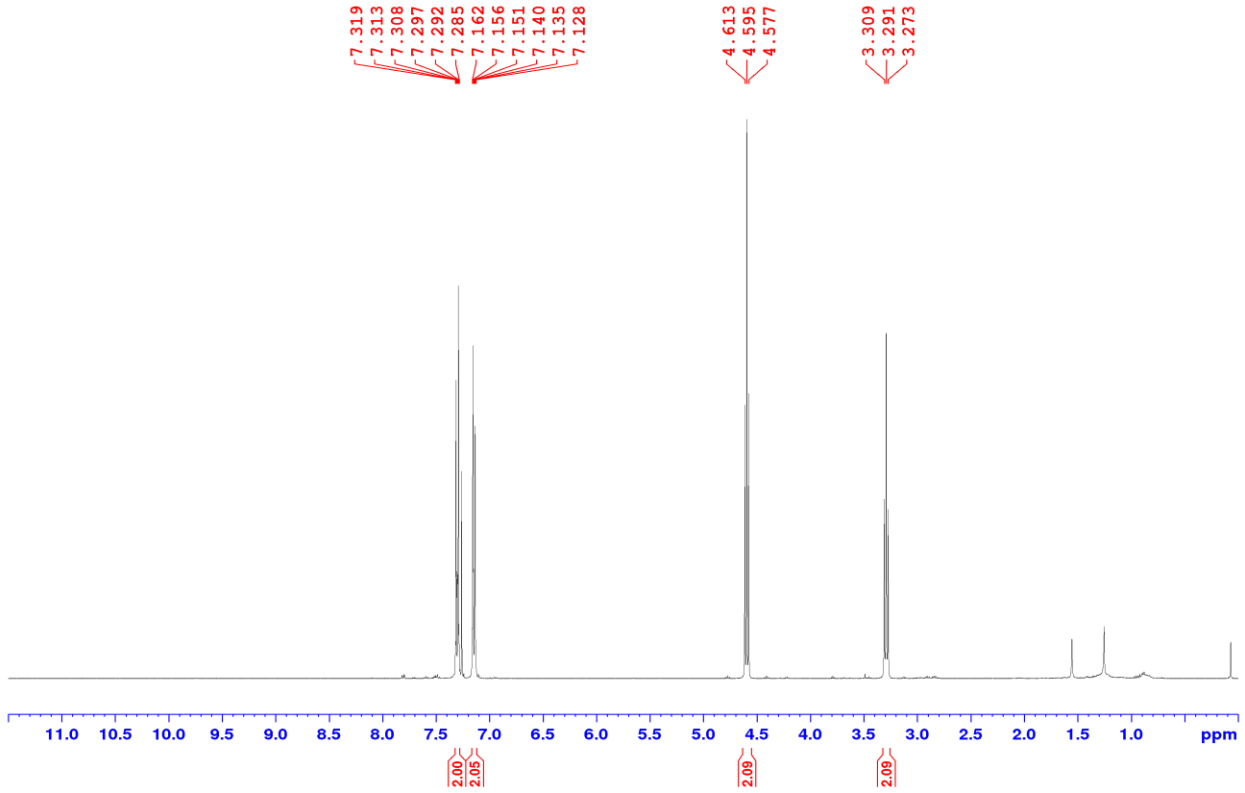
2f - 1-(2-Nitroethyl)-4-(Trifluoromethyl)benzene

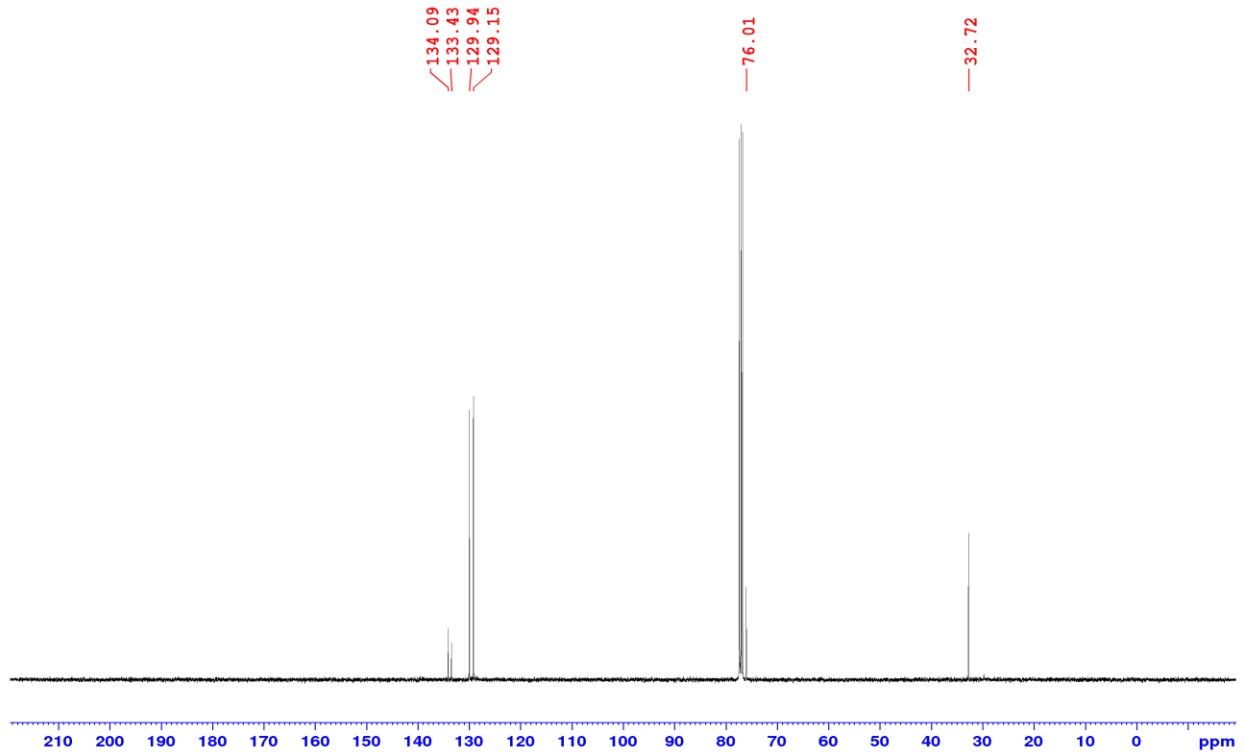


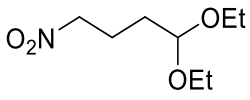




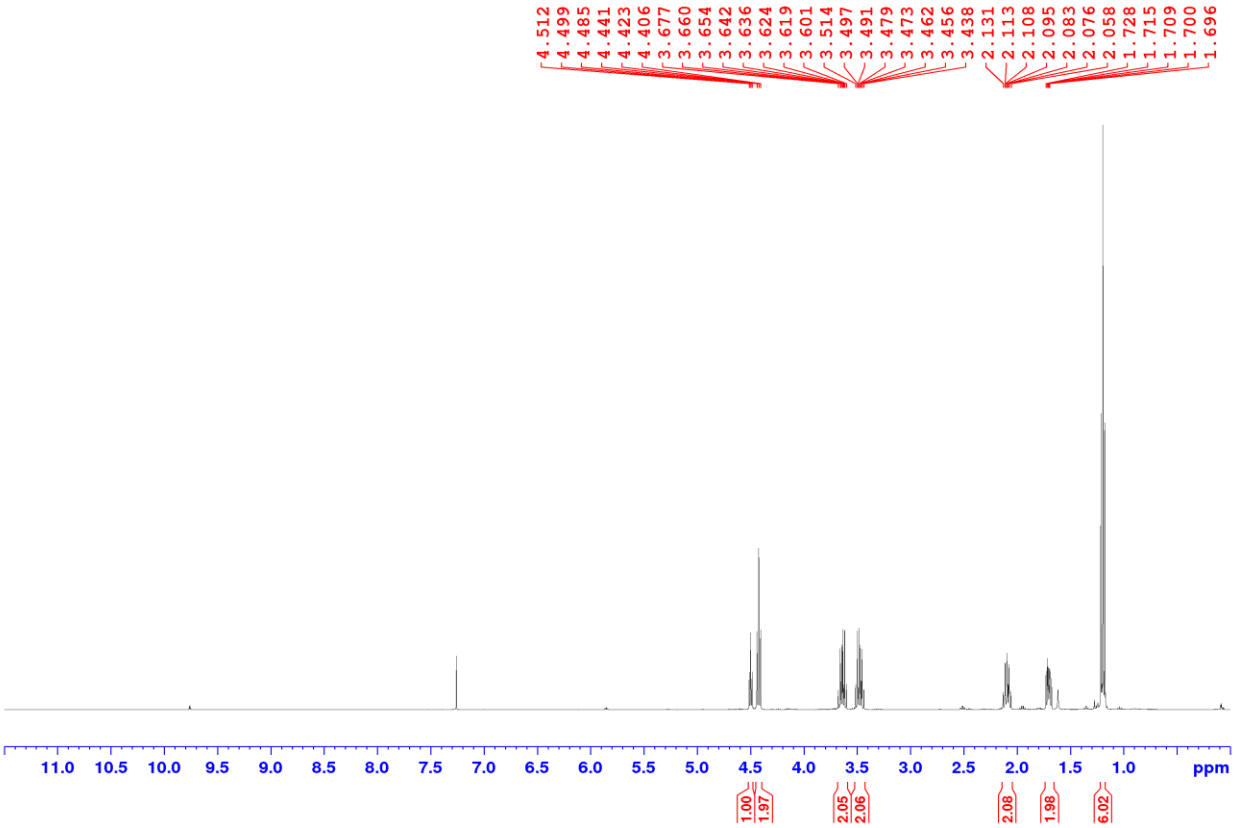
2g - 1-(2-Nitroethyl)-4-Chlorobenzene

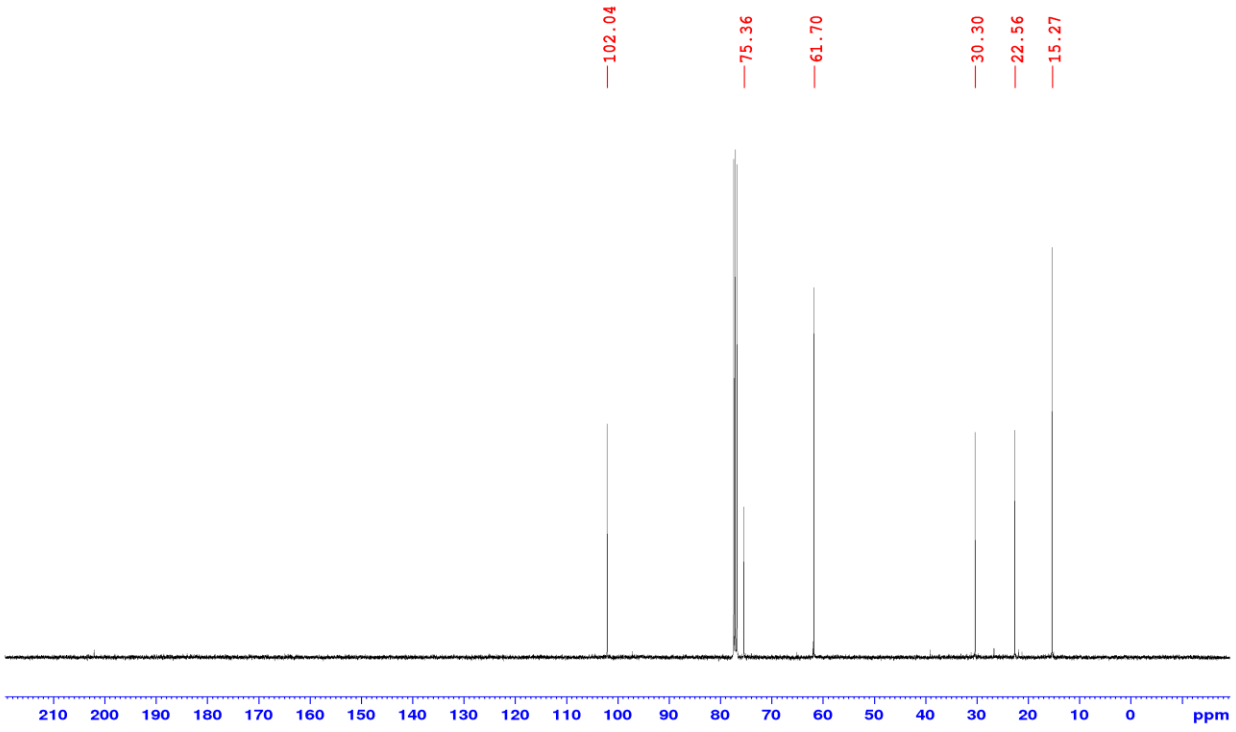


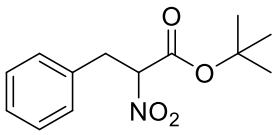




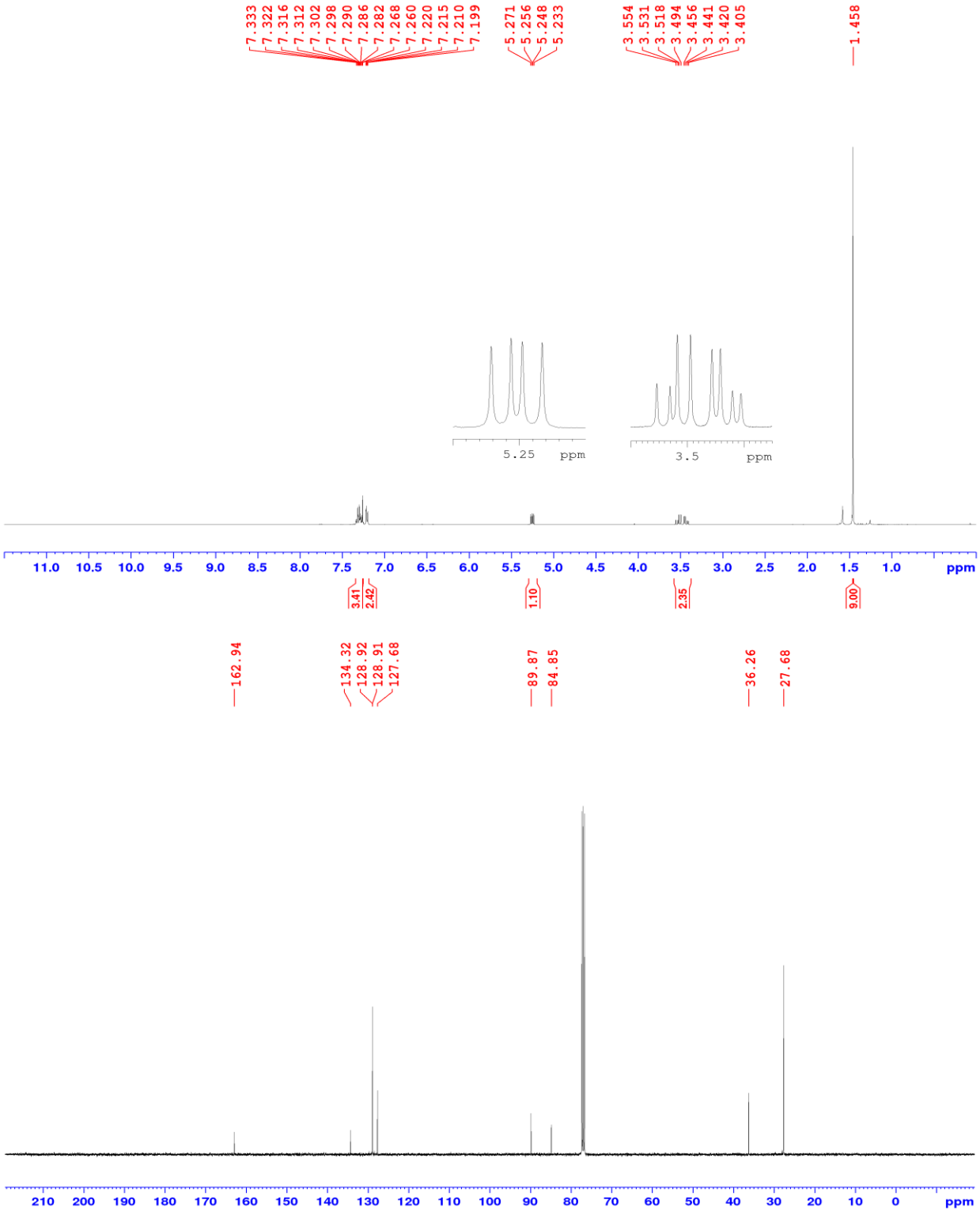
2h - 1,1-Diethoxy-4-Nitrobutane

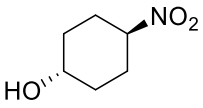




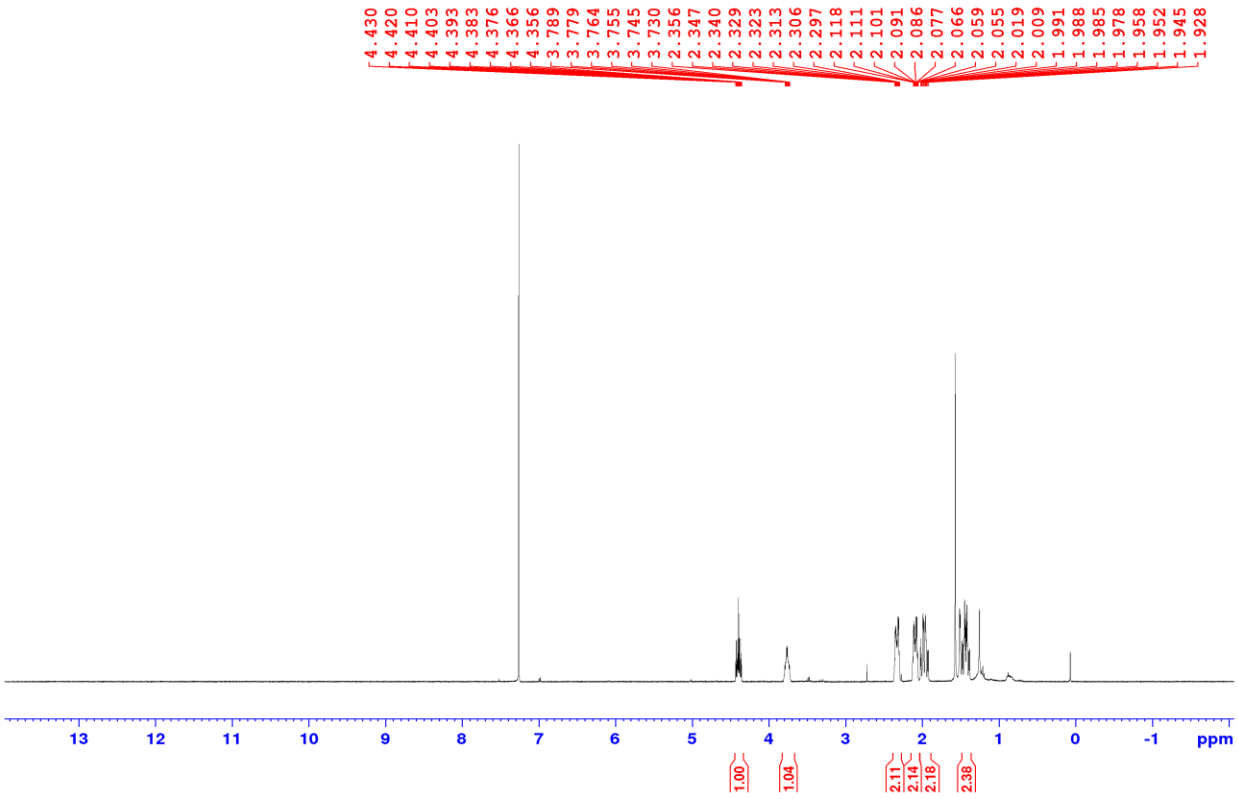


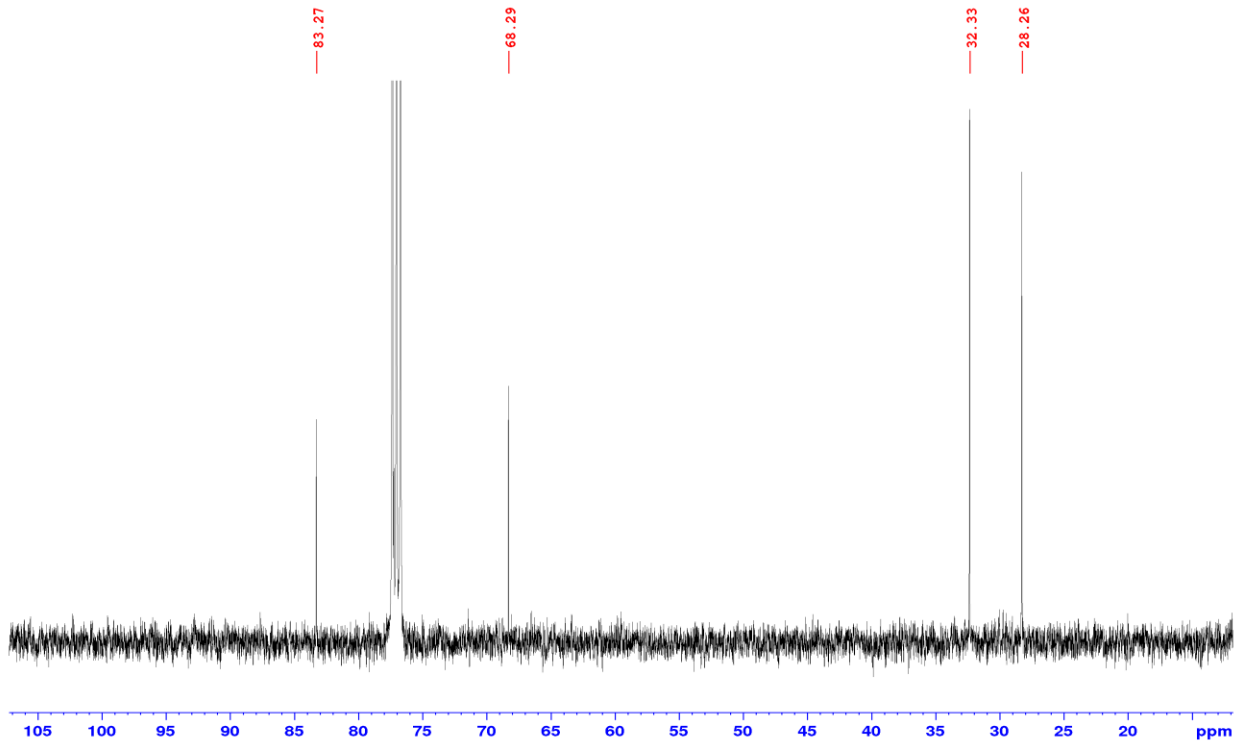
2i - *Tert*-Butyl Nitrophenylpropanoate

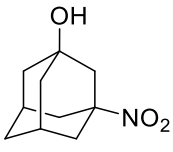




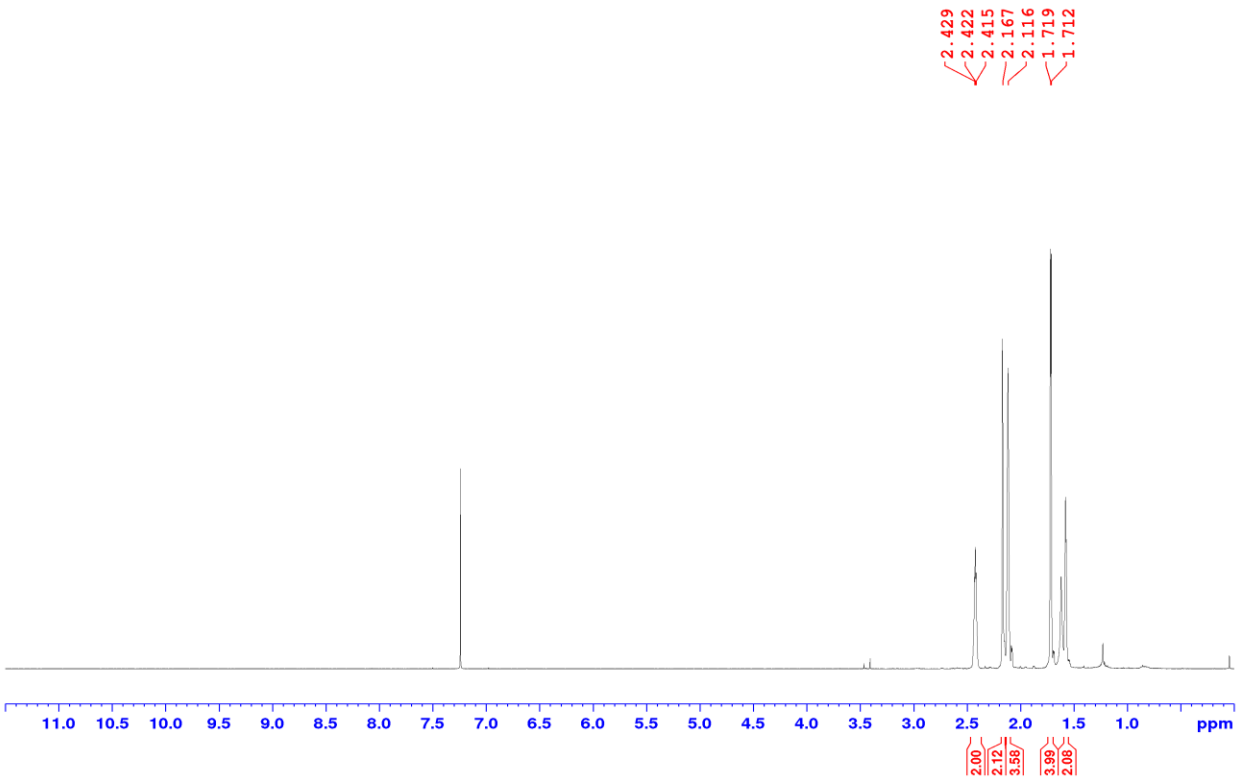
2j - Trans-4-Nitrocyclohexanol

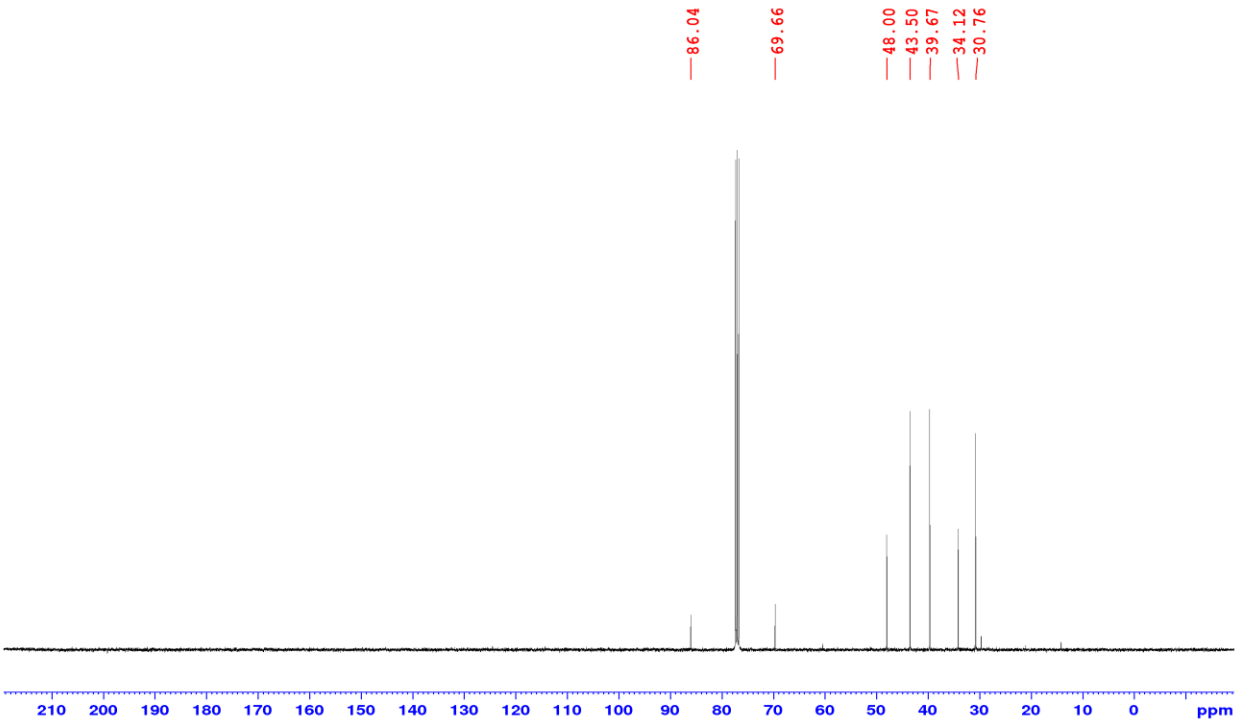


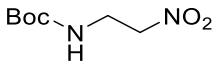




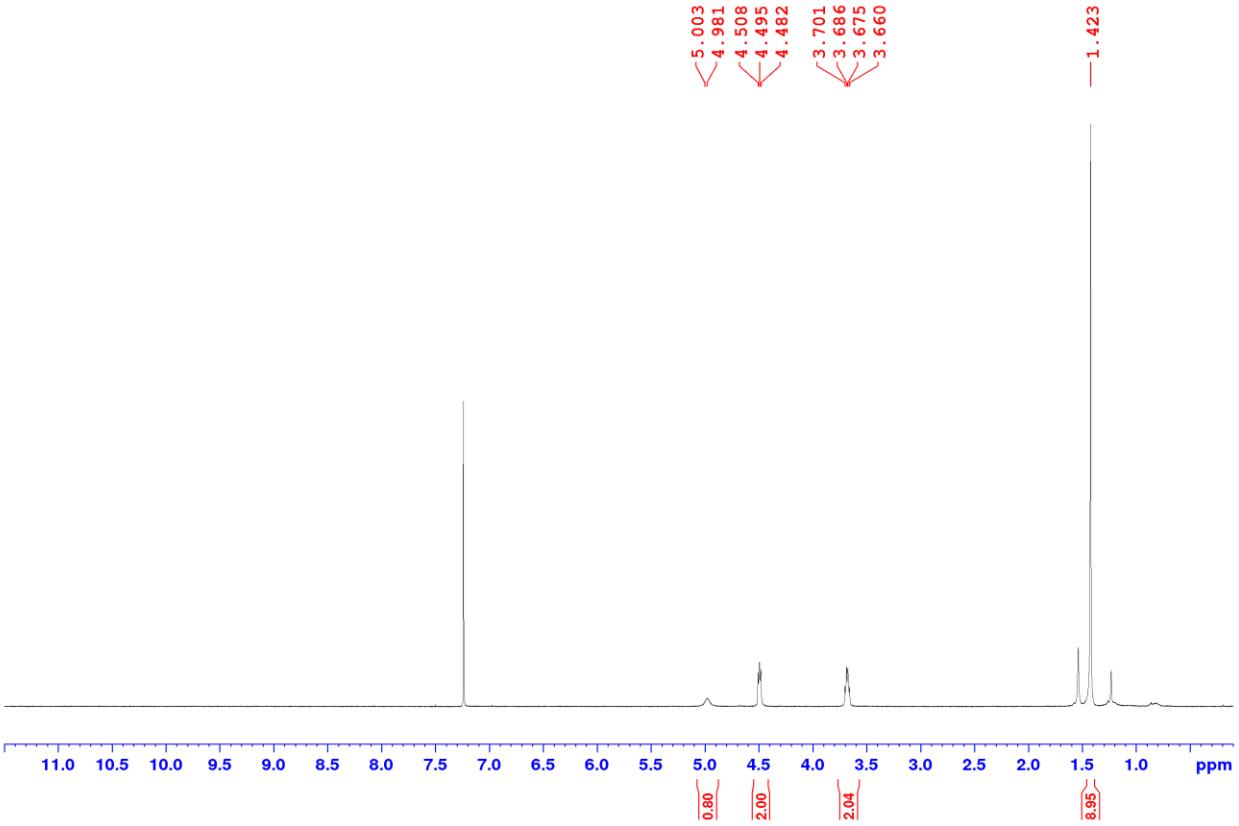
2k - 1-Nitro-3-Adamantol

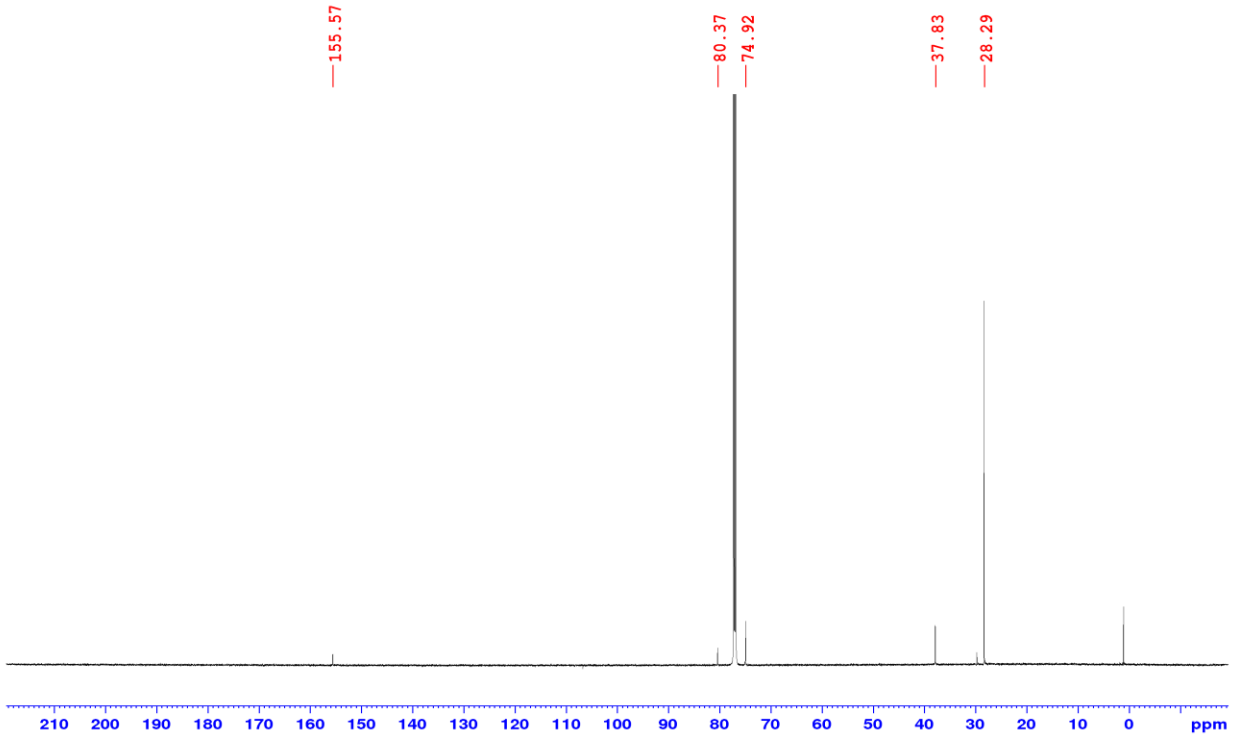


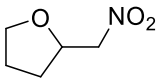




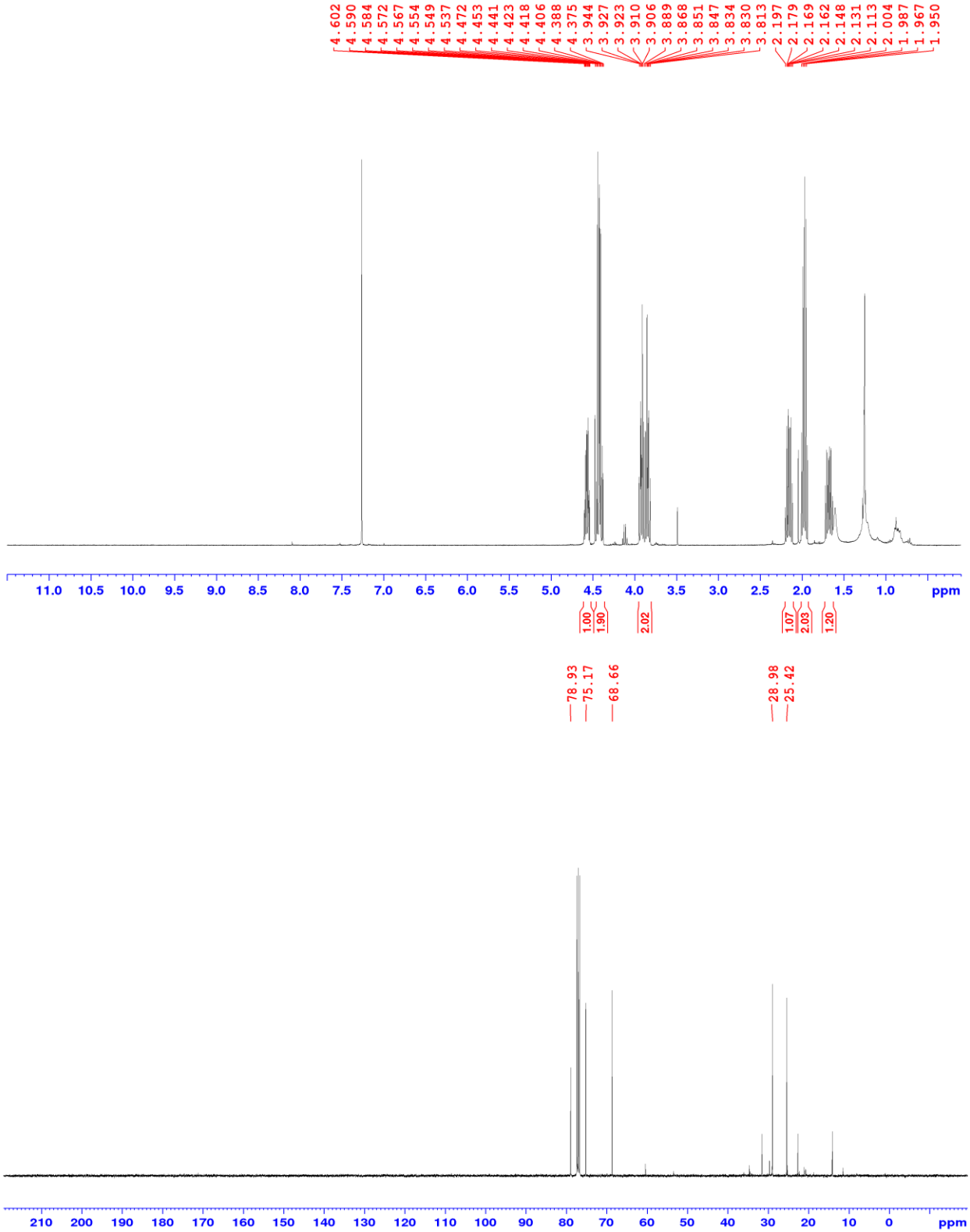
2l - *Tert*-Butyl (2-Nitroethyl)carbamate

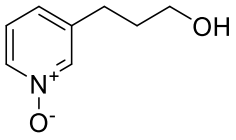




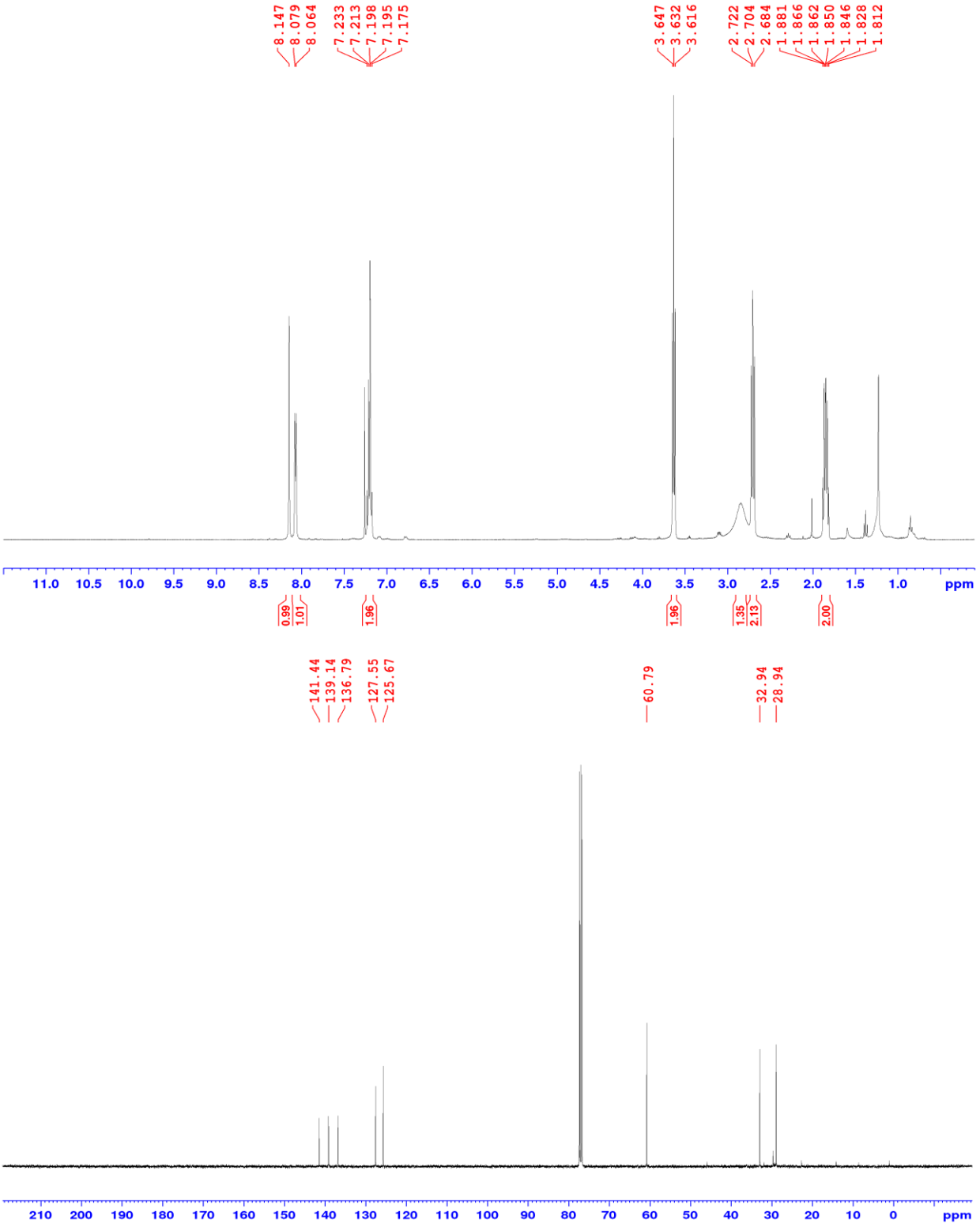


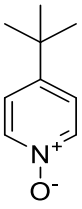
2m - 2-(Nitromethyl)tetrahydrofuran



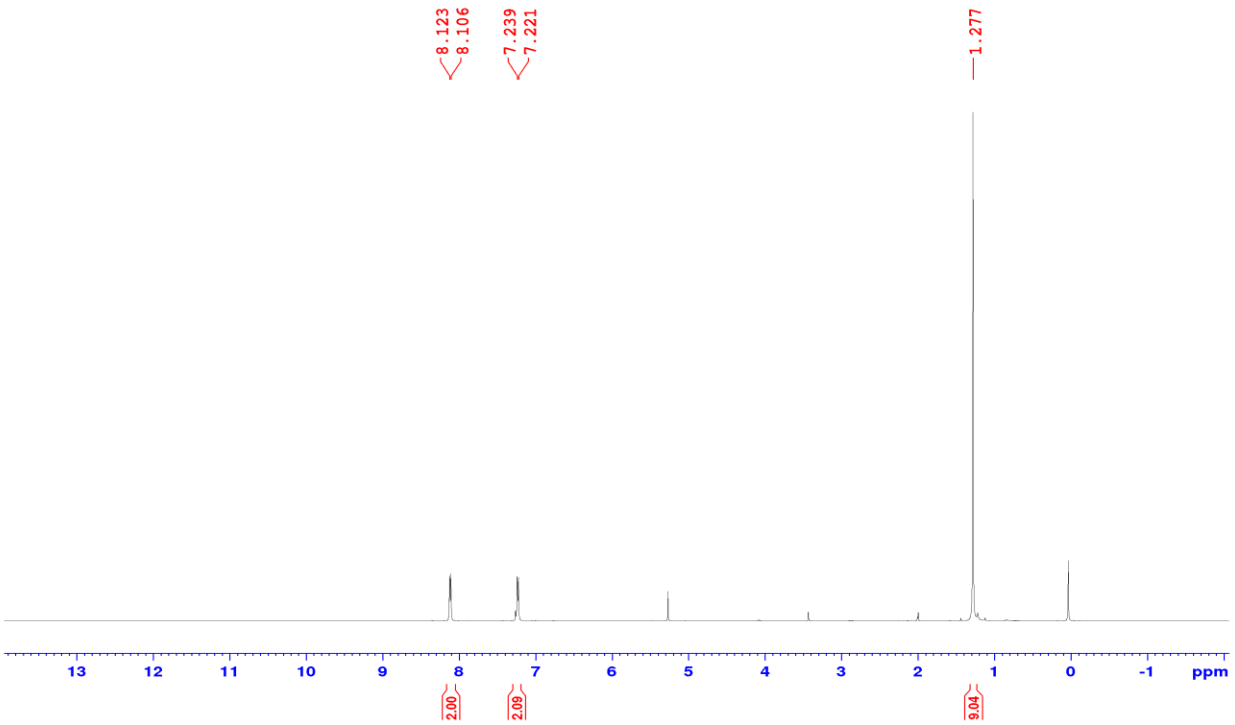


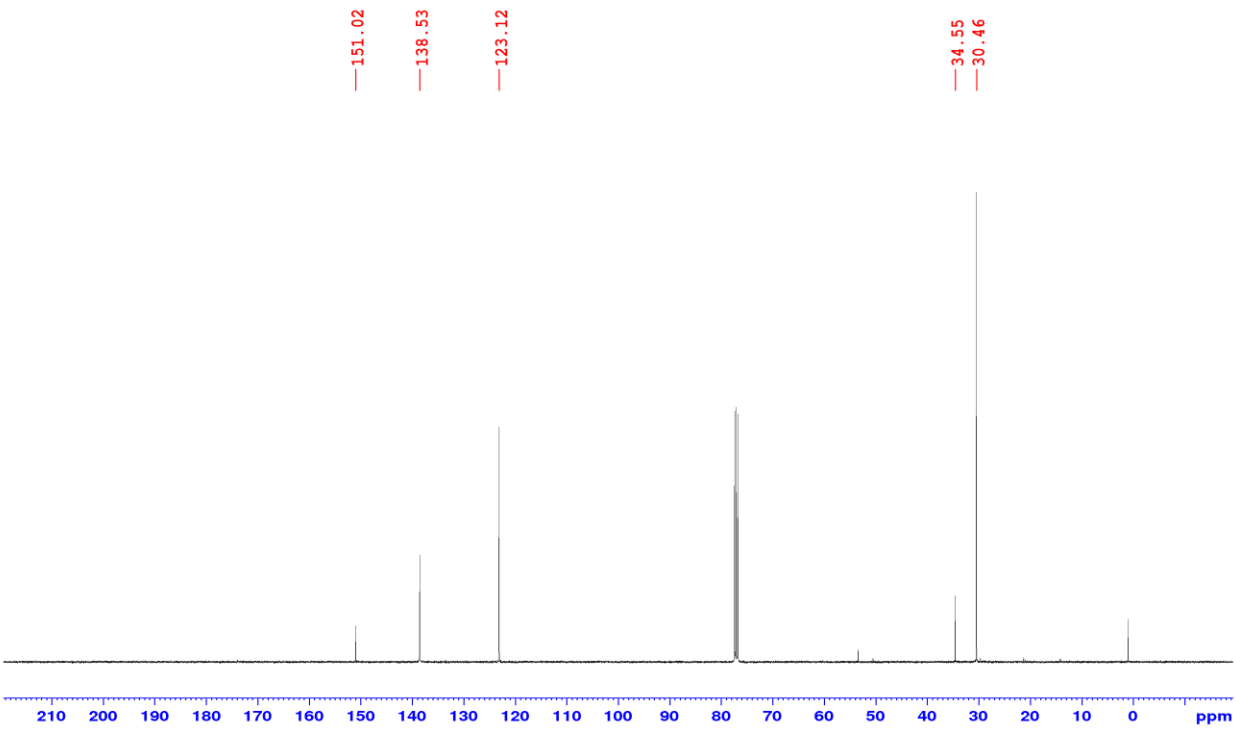
4a - 3-Pyridinylpropanol *N*-oxide

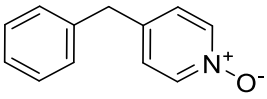




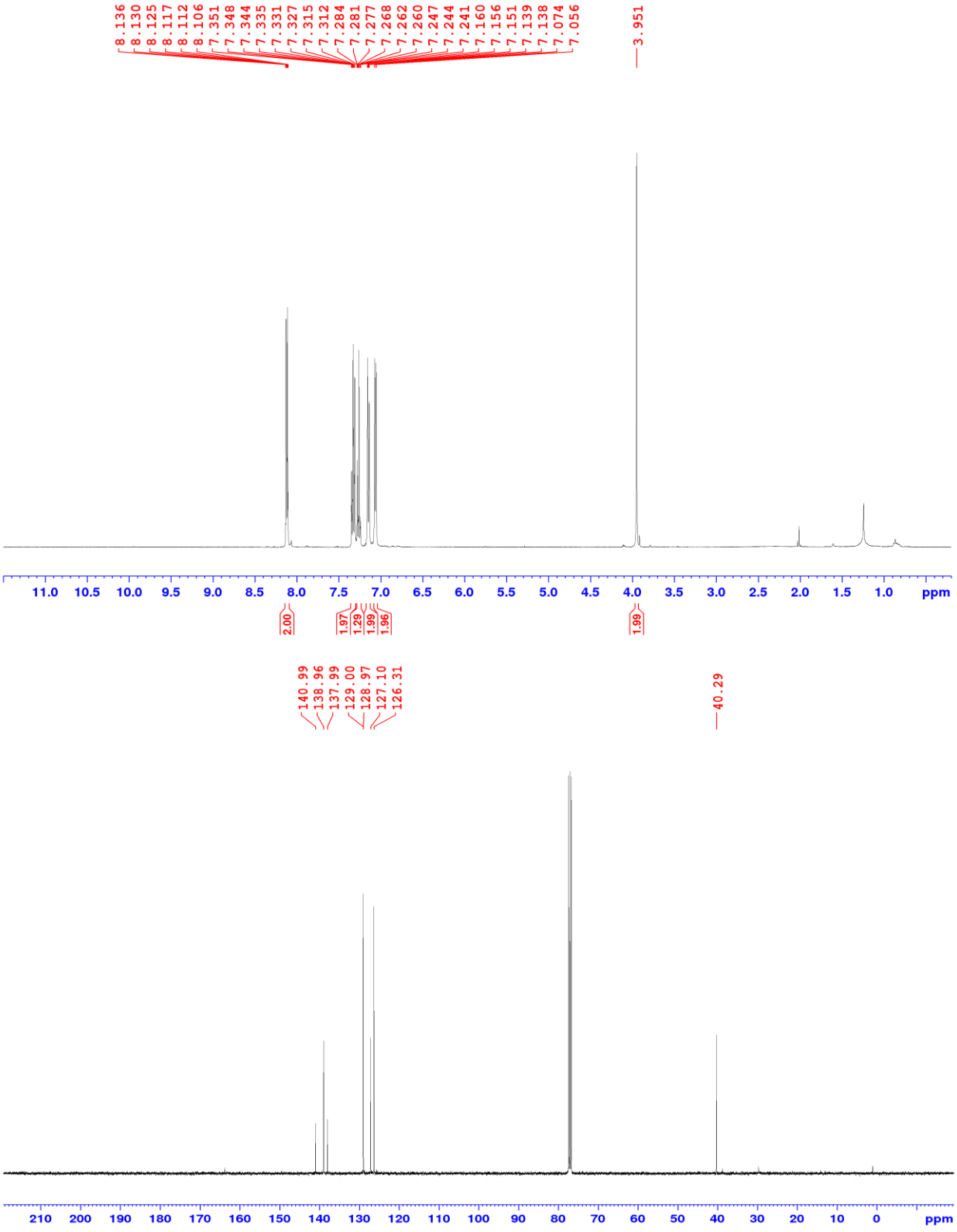
4b - 4-*Tert*-Butylpyridine *N*-oxide

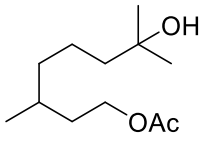




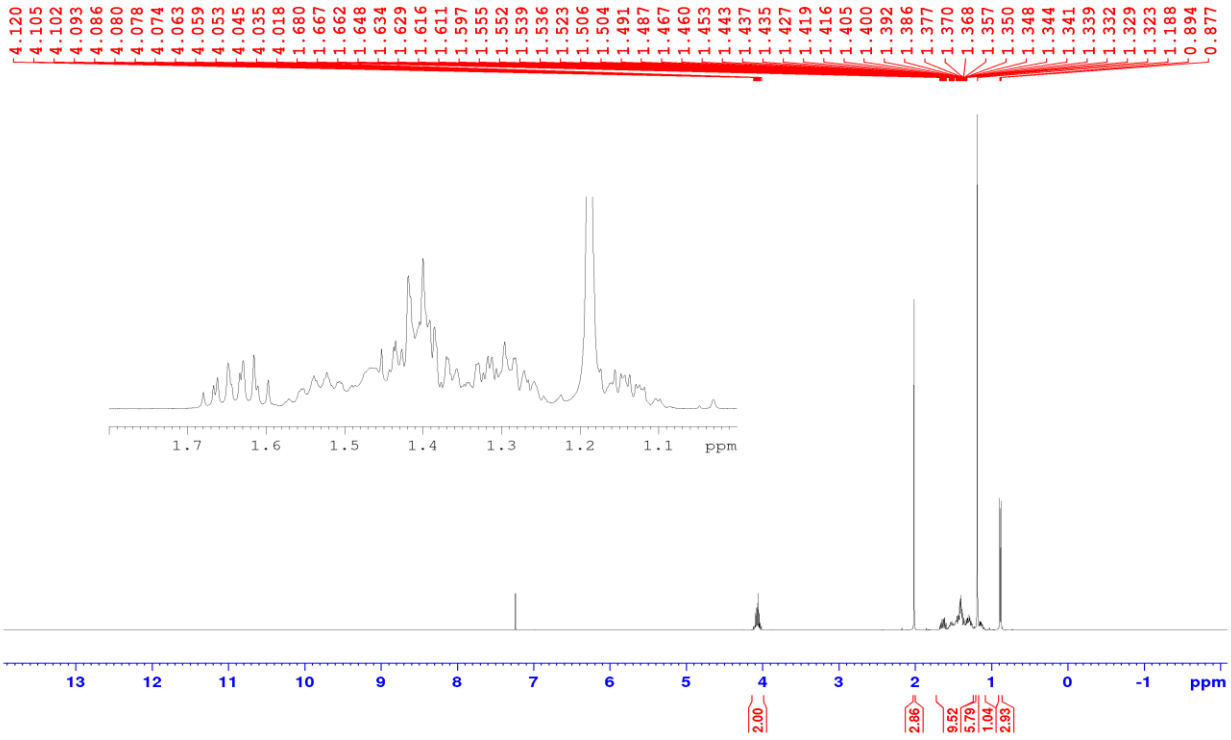


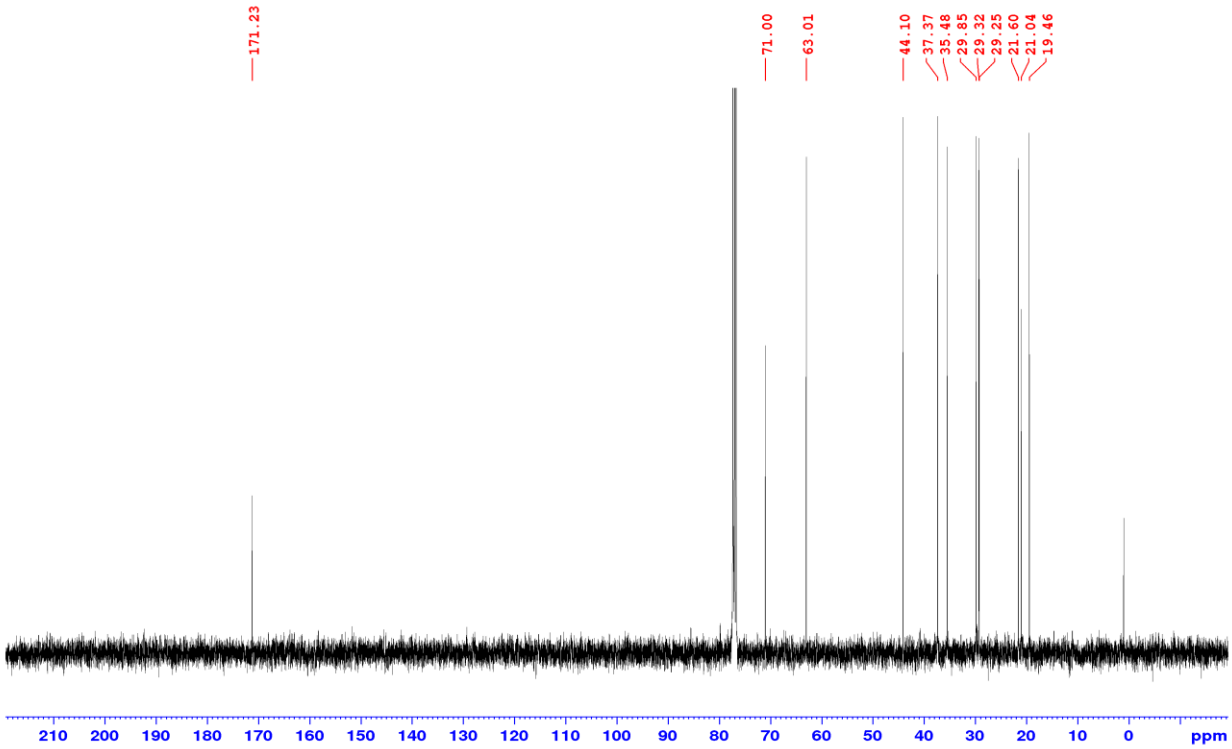
4c - 4-Benzylpyridine *N*-oxide

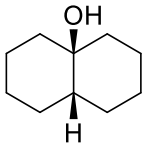




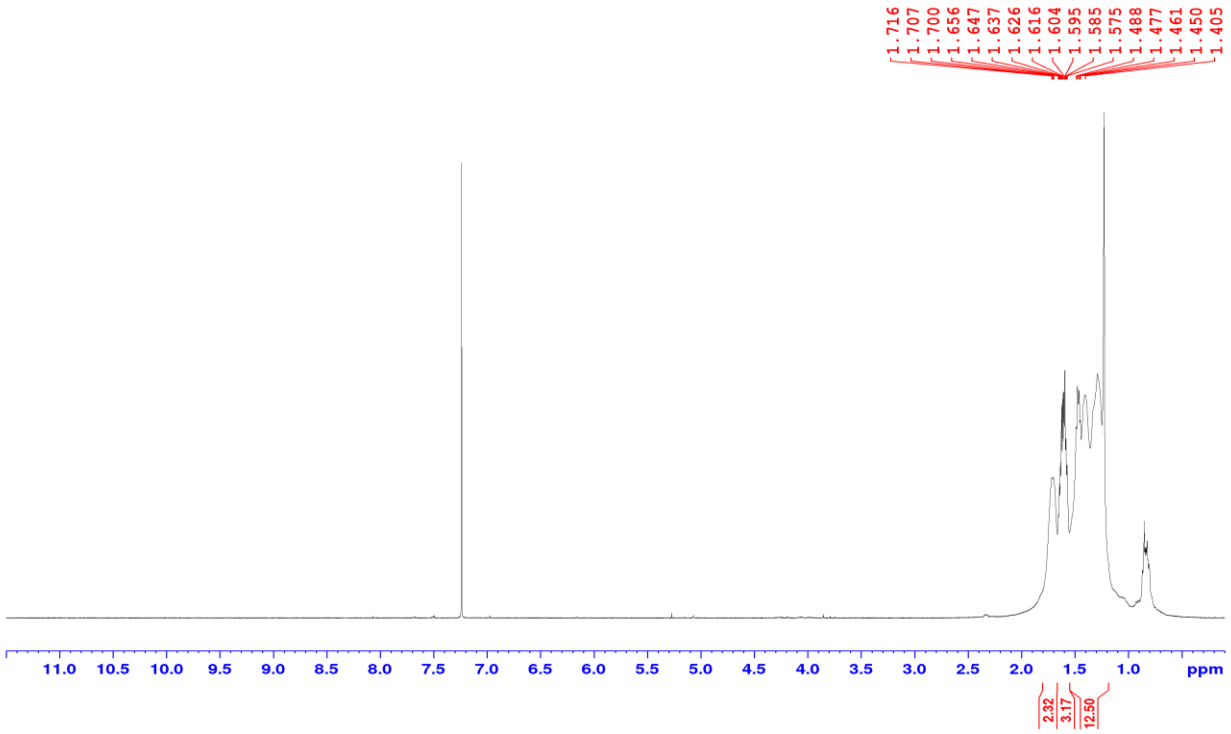
6a - 7-Hydroxy-3,7-Dimethyloctyl Acetate

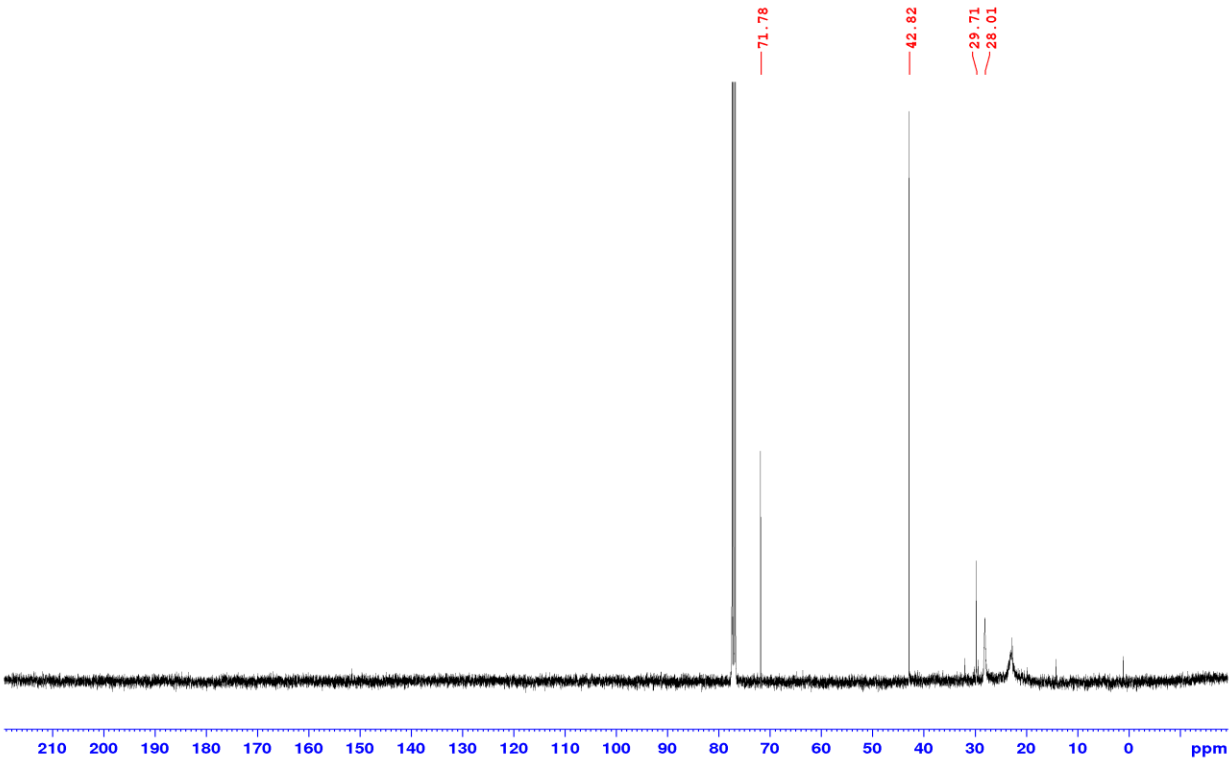


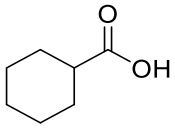




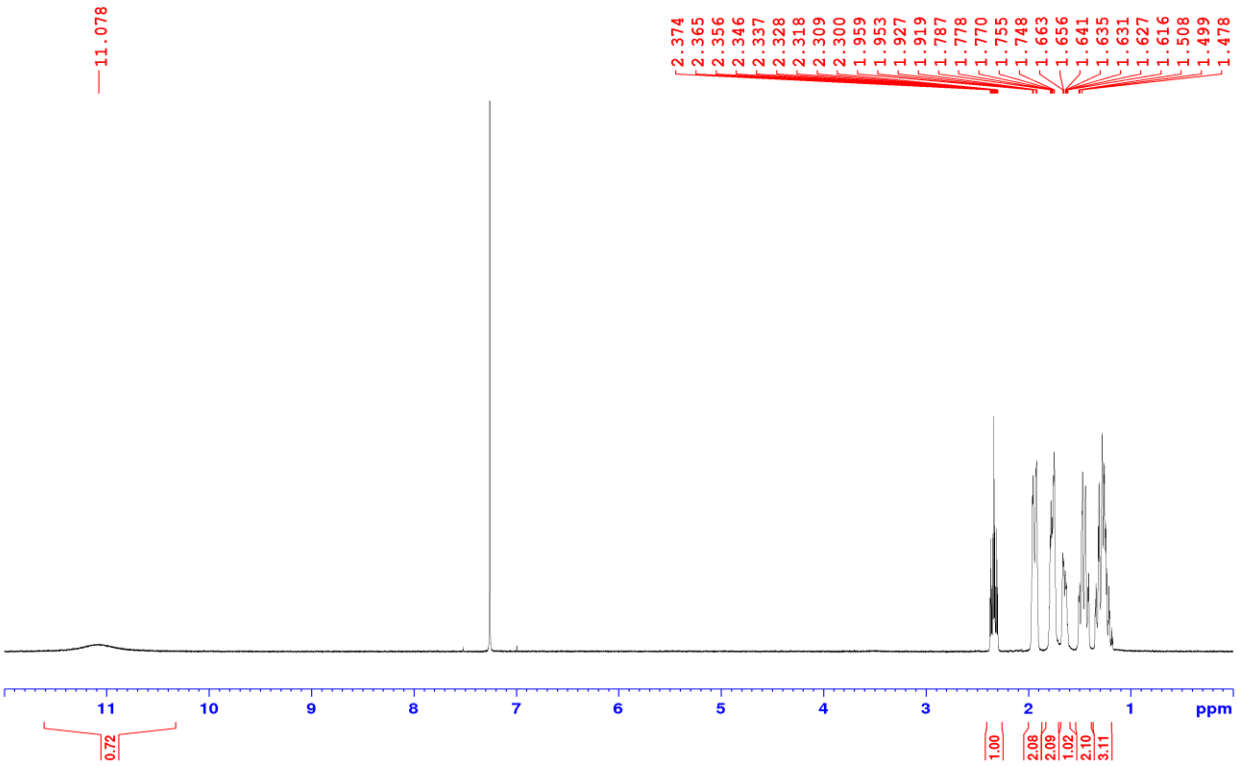
6b - Cis-9-Decalol

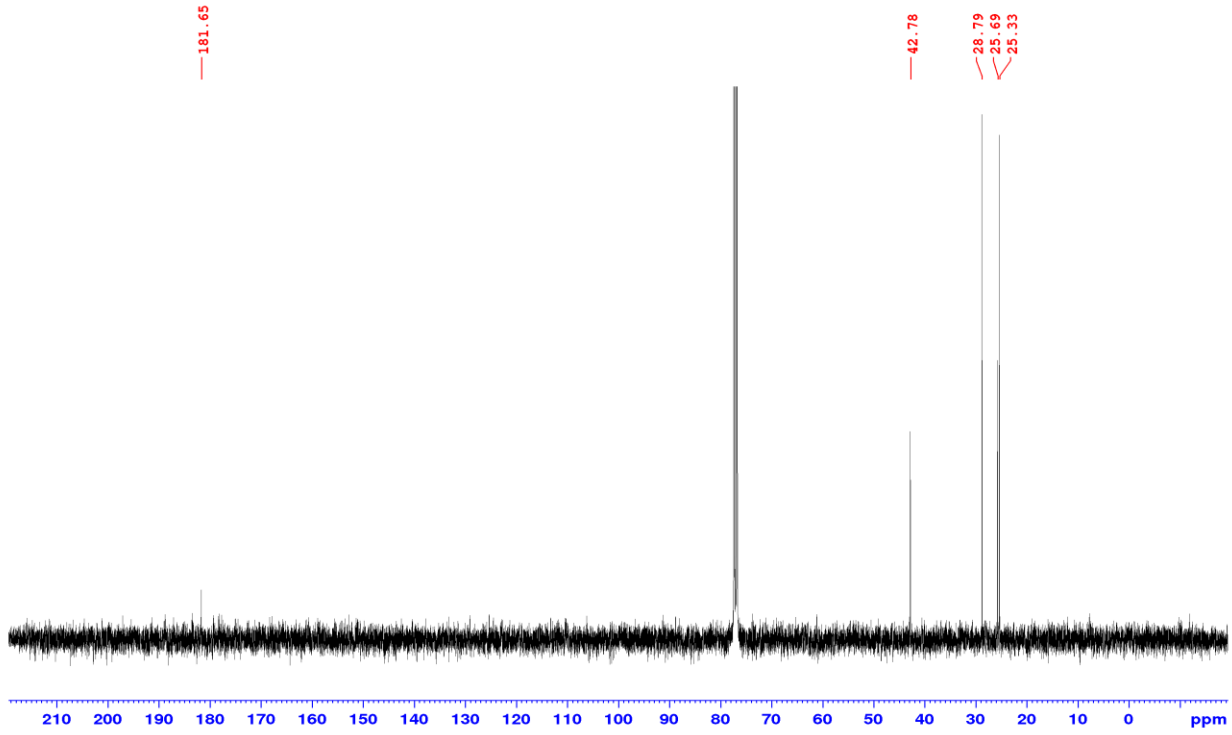


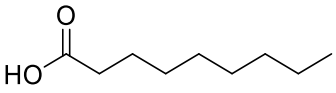




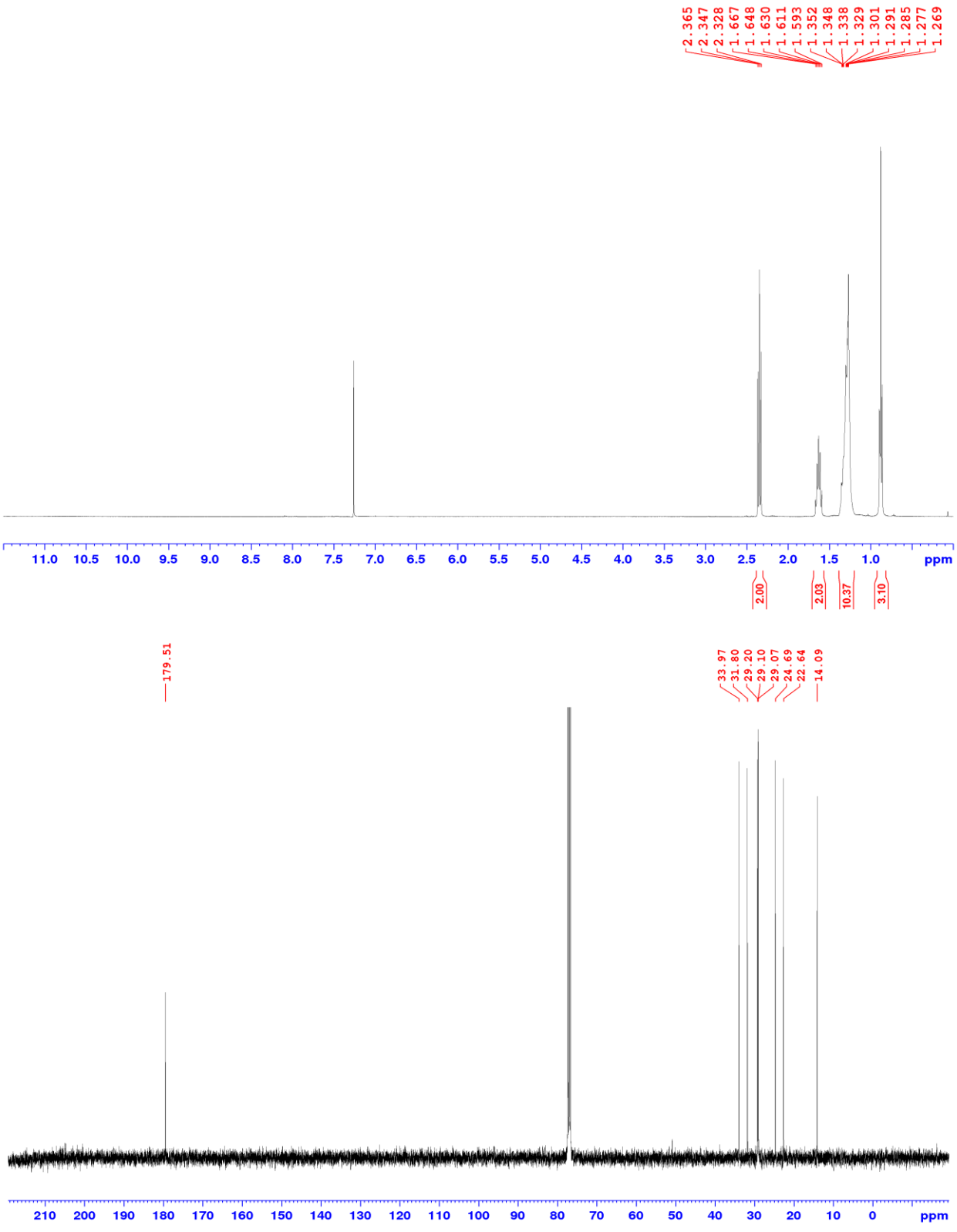
8a - Cyclohexane Carboxylic Acid







8b - Nonanoic Acid



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