Supporting Information

Palladium-Catalyzed Aerobic Oxidative Dehydrogenation of Cyclohexenes to Substituted Arene Derivatives

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General Considerations

All commercially available organic compounds and solvents were used as received from Sigma Aldrich and TCI America unless otherwise specified. $Pd(OAc)_2$ was purchased from Sigma Aldrich (# 520764) and used without any further purification. $Pd(TFA)_2$ was purchased from Strem (# 46-0280) and used without any further purification after its purity was confirmed by NMR spectroscopy. Chlorobenzene used was purchased from Sigma Aldrich and used as received. ^{1}H and ^{13}C NMR spectra were recorded on Bruker 300, 400 and 500 MHz spectrometers and chemical shifts are given in parts per million (ppm) relative to standard tetramethylsilane (0.00 ppm for ^{1}H NMR) or residual solvent peaks for ^{13}C NMR. High resolution mass spectra were obtained using a Water Autospec by the mass spectrometry facility at the University of Wisconsin. Chromatography was performed using an automated Isco Combiflash Rf® system with reusable high performance silica gel 60 (Silicycle) and eluted with hexane/ethyl acetate. Melting points were recorded on a Melt-Temp® apparatus.

General procedure for dehydrogenation reaction optimization and experimental data shown in Table 1 – Orbital mixing (Procedure A)

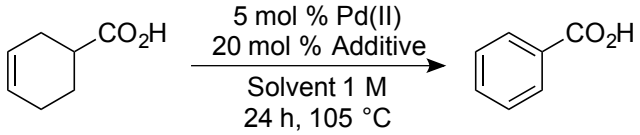
To a test tube were added sequentially Pd^{II} salt (0.0125 mmol), additive (0.05 mmol) and the 3-cyclohexene-1-carboxylic acid substrate (31.5 mg, 0.25 mmol), followed by solvent (0.25 ml). The test tube was placed in a custom parallel shaker reactor, and the headspace was filled with O_2 slightly above 1 atm and vented 10 times, followed by a 5 minute purge of the headspace with O_2. Upon sealing the reactor, the reactor was subjected to orbital shaking (40 rpm) at 105 °C (or other specified temperature) for 24 h, after which the shaking was stopped and the reactor allowed to cool to room temperature. The O_2 pressure was released and then to the contents of each test tube were added a known quantity of external standard (1,1,2,2-tetrachloroethane or 1,2-dibromomethane) in $CDCl_3$ and an aliquot was taken from this mixture, filtered through a short pad of celite and analyzed by ^{1}H NMR spectroscopy.

General procedure for preparative-scale reactions using an O_2 balloon – Magnetic stirring (Procedure B)

A 20 x 150 mm culture tube was equipped with a stir bar, oven dried and capped with a septum. Meanwhile, on a weighing paper were weighed $MgSO_4$ (100 mg), AMS (62.1 mg, 0.2 mmol), $Pd(TFA)_2$ (16.6 mg, 0.05 mmol) and substrate (1 mmol), if it is a solid. The solid mixture was added to the dry test tube, followed by addition of the substrate if it is a liquid and chlorobenzene (1.0 ml). The test tube was capped with a septum immediately. The reaction tube was purged with oxygen for 10 min using a 20 ga. needle inserted into the solvent. The solution was stirred while purging. An O_2 balloon was then attached to the sealed test tube, this time with the needle above the solvent level, and the mixture was heated to 110 °C using an oil bath. Because some of the solids do not fully dissolve and the temperature is close to the boiling point of chlorobenzene, stirring was adjusted to ensure that solids are not dispersed on the walls of the tube. After 24 h, the mixture was diluted with DCM or hexanes and placed directly on a column which was run using an automated Combiflash using Hexane/EtOAc gradients from 0% EtOAc to 100% EtOAc.

This procedure was successfully applied on a 10 mmol scale for **1a**, using a 250 ml round bottom flask and a football shaped stir bar.

Table S1. Optimization of Reaction Conditions using Procedure A (Orbital Mixing)



1a **2a**

Entry	Pd(II) (5 mol %)	Solvent (1 M)	Additive (20 mol %)	Yield (Conv)[a]
1	$PdCl_2$	Mesitylene	-	0 (21)
2	$Pd(OAc)_2$	Mesitylene	-	30 (82)
3	$Pd(TFA)_2$	Mesitylene	-	32 (98)
4	$Pd(TFA)_2$	DMSO	-	4 (30)
5	$Pd(TFA)_2$	DMF	-	26 (87)
6	$Pd(TFA)_2$	BuOAc	-	29 (49)
7	$Pd(TFA)_2$	NMP	-	32 (100)
8	$Pd(TFA)_2$	diglyme	-	62 (100)
9	$Pd(TFA)_2$	Propylene carbonate	-	27 (100)
10	$Pd(TFA)_2$	$EtCO_2H$	-	18 (100)
11	**$Pd(TFA)_2$**	**PhCl**	**-**	**71 (93)**
12	$Pd(TFA)_2$	DMAc	-	25 (98)
13	$Pd(TFA)_2$	PhCl	$Cu(OAc)_2$	2 (98)
14	$Pd(TFA)_2$	PhCl	$Cu(TFA)_2$	5 (100)
15	$Pd(TFA)_2$	PhCl	$Cu(OTf)_2$	15 (96)
16	$Pd(TFA)_2$	PhCl	AgOAc	1 (56)
17	$Pd(TFA)_2$	PhCl	AgTFA	3 (27)
18	$Pd(TFA)_2$	PhCl	NaOAc	36 (100)
19[b]	$Pd(TFA)_2$	PhCl	*p*TsOH	17 (79)
20	$Pd(TFA)_2$	PhCl	DMSO	24 (40)
21	$Pd(TFA)_2$	PhCl	$EtCO_2H$	83 (100)
22	$Pd(TFA)_2$	PhCl	TfOH	23 (96)
23	**$Pd(TFA)_2$**	**PhCl**	**AMS**	**99 (100)**
24	$Pd(TFA)_2$	PhCl	BQ	36 (64)
25	$Pd(TFA)_2$	PhCl	Anthraquinone	92 (100)
26	$Pd(TFA)_2$	PhCl	$PhSO_3Na$	90 (100)
27	$Pd(TFA)_2$	$PhCF_3$	AMS	89 (100)
28	$Pd(TFA)_2$	PhCN	AMS	11 (n.d.)

29	$Pd(TFA)_2$	1,1,2,2-tetrachloroethane	AMS	71 (89)
30	$Pd(TFA)_2$	Decane	AMS	69 (100)
31	$Pd(TFA)_2$	tAmylOH	AMS	62 (100)
32	$Pd(TFA)_2$	Diglyme	AMS	56 (100)
33	$Pd(TFA)_2$	BuOAc	AMS	60 (100)
34	-	PhCl	AMS	0 (0)
35[c]	$Pd(TFA)_2$	PhCl	AMS	69 (81)
36[d]	$Pd(TFA)_2$	PhCl	AMS	85 (100)
37[e]	$Pd(TFA)_2$	PhCl	AMS	87 (96)

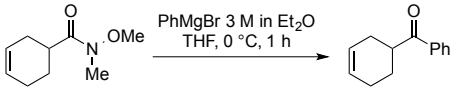
[a]Reactions performed on 0.25 mmol scale; [**1a**] = 1 M. [1]H NMR yields with 1,1,2,2-tetrachloroethane or dibromomethane as standard. [b]40 mol % *p*TsOH. [c]1 mol % $Pd(TFA)_2$, 20 mol % AMS. [d]1 mol % $Pd(TFA)_2$, 4 mol % AMS. [e]Reaction performed at 80 °C.

Synthesis and Characterization of Substrate Precursors

Substrates **1a**, **1b**, **1c**, **1d**, **1e**, **1f**, **1g**, **1h** and **1r** are commercially available. Substrates **1p**[1] and **1q**[2] were prepared according to literature reports. Substrates that are not prepared via procedures reported in the literature are presented below.

Synthesis of Ketones: Procedure C

To an oven-dried round bottom flask was added THF (50 ml) and the Weinreb amide shown below (5.0 mmol), which had been prepared according to procedure D (see below). The reaction was cooled to 0 °C, at which point the solution of Grignard reagent (15.0 mmol) was added dropwise via syringe. The reaction mixture was allowed to stir for the indicated time for each substrate at 0 °C, after which it was diluted with DCM and a solution of saturated ammonium chloride (50 ml) was added. The organic layer was washed again with water and then dried using sodium sulfate and concentrated *in vacuo*. The mixture was purified using column chromatography to afford the desired product.

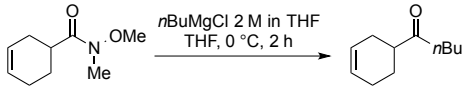


72%

(3-cyclohexen-1-yl)phenylmethanone (1i)

PhMgBr (3 M in Et_2O, 5 ml) was used according to Procedure C, and the reaction stirred for 1 h. The product **1i** was obtained as a colorless liquid (672.6 mg, 3.6 mmol, 72% yield).

^{1}H NMR (300 MHz, Chloroform-d) δ 8.08-7.89 (m, 2H), 7.64-7.34 (m, 3H), 5.87-5.65 (m, 2H), 3.63-3.42 (m, 1H), 2.45-2.29 (m, 1H), 2.29-2.07 (m, 3H), 2.07-1.90 (m, 1H), 1.84-1.58 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 203.45, 136.30, 132.90, 128.57, 126.61, 125.78, 41.53, 27.89, 25.74, 24.91; **HRMS (ESI)** Calcd. for $C_{13}H_{15}O$ ($[M+H]^+$): 187.1118, found: 187.1117.



76%

Butyl 3-cyclohexenyl ketone (1j)

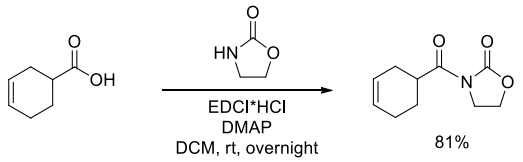
*n*BuMgBr (2 M in THF, 7.5 ml) was used according to Procedure C, and the reaction stirred for 2 h. The product **1j** was obtained as a colorless liquid (629.0 mg, 3.8 mmol, 76% yield).

^{1}H NMR (300 MHz, Chloroform-d) δ 5.75-5.60 (s, 2H), 2.60 (m, 1H), 2.47 (td, J = 7.3, 3.3 Hz, 3H), 2.22-2.04 (m, 5H), 1.99-1.86 (m, 1H), 1.65-1.48 (m, 5H), 1.39-1.24 (m, 4H), 0.91 (t, J = 7.3 Hz, 5H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 213.88, 126.61, 125.46, 77.05, 76.74, 46.55, 40.48, 26.92, 25.87, 24.75, 22.45, 13.91; **HRMS (ESI)** Calcd. for $C_{11}H_{19}O$ ($[M+H]^+$): 167.1431, found: 167.1435.

Synthesis of Amides: Procedure D

Procedures were performed on a scale of 5-15 mmol of 3-cyclohexene-1-carboxylic acid. To an oven dried round bottom flask were added DMAP (1 eq.) and DCM (0.1 M) under N_2. The 3-cyclohexene-1-carboxylic acid (1 eq.) was added via syringe and then EDCI (1.2 eq.) was added in one portion. The reaction mixture was stirred for 15 minutes, after which the corresponding amine (1.1 eq.) was added dropwise via syringe. The reaction stirred overnight at room temperature. Aqueous HCl (1N, 50 ml) was added to the reaction mixture and the organic layer was washed again with 1N HCl. The organic layer was dried using sodium sulfate and then

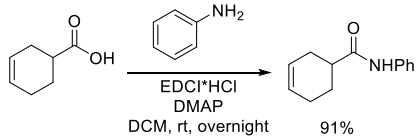
concentrated *in vacuo*. The crude mixture was pass through a silica plug, affording the desired product.



3-(3-Cyclohexen-1-yl carbonyl)-1,3-oxazolidin-2-one (1k)

Using Procedure D on 7.5 mmol scale with 1,3-oxazolidin-2-one as the amine partner, the product **1k** was obtained as viscous, colorless oil (1.19 g, 6.1 mmol, 81% yield).

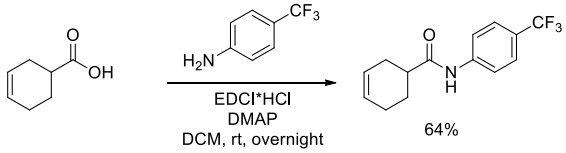
^{1}H NMR (400 MHz, Chloroform-d) δ 5.84-5.59 (m, 2H), 4.42 (t, *J* = 8.1 Hz, 2H), 4.12-3.90 (m, 2H), 3.88-3.63 (m, 1H), 2.48-2.03 (m, 4H), 2.01-1.91 (m, 1H), 1.74-1.52 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 176.52, 153.19, 126.63, 125.07, 61.96, 42.83, 38.19, 27.17, 25.59, 24.69; **HRMS (ESI)** Calcd. for $C_{10}H_{14}NO_3$ ($[M+H]^+$): 196.0973, found: 196.0969.



***N*-phenylcyclohex-3-enecarboxamide (1l)**

Using Procedure D on 5 mmol scale with aniline as the amine partner, the product **1l** was obtained as a white solid (915.8 mg, 4.55 mmol, 91% yield.)

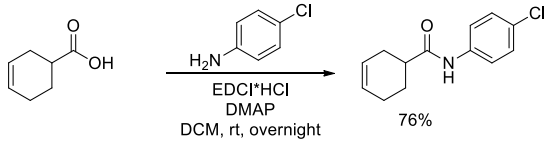
M.P. 119-120 °C; **^{1}H NMR** (300 MHz, Chloroform-d) δ 7.73-7.43 (m, 3H), 7.37-7.21 (m, 2H), 7.16-7.01 (m, 1H), 5.72 (d, *J* = 2.4 Hz, 2H), 2.45-2.25 (m, 1H), 2.24-2.07 (m, 2H), 2.06-1.94 (m, 1H), 1.90-1.72 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 174.39, 138.11, 128.99, 126.89, 125.28, 124.21, 119.95, 42.28, 28.11, 25.79, 24.58; **HRMS (ESI)** Calcd. for $C_{13}H_{16}NO$ ($[M+H]^+$): 202.1227, found: 202.1228.



***N*-(4-(trifluoromethyl)phenyl)cyclohex-3-enecarboxamide (1m)**

Using Procedure D on 5 mmol scale with 4-(trifluoromethyl)aniline as the amine partner, the product **1m** was obtained as an off white solid (860.9 mg, 3.2 mmol, 64% yield).

M.P. 158-159 °C; **^{1}H NMR** (300 MHz, Chloroform-d) δ 7.73-7.39 (m, 5H), 5.84-5.62 (m, 2H), 2.65-2.46 (m, 1H), 2.47-2.29 (m, 2H), 2.29-2.09 (m, 2H), 2.09-1.95 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 174.59, 141.06, 126.96, 126.25 (q, *J* = 3.9 Hz), 125.82, 125.43, 125.00, 122.73, 119.42, 42.35, 27.95, 25.72, 24.46; **HRMS (ESI)** Calcd. for $C_{14}H_{15}F_3NO$ ($[M+H]^+$): 270.1102, found: 270.1093.

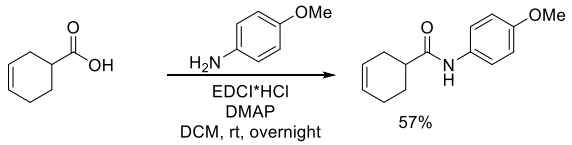


***N*-(4-chlorophenyl)cyclohex-3-enecarboxamide (1n)**

Using Procedure D on 5 mmol scale with 4-chloroaniline as the amine partner, the product **1n** was obtained as an off white solid (895.0 mg, 3.8 mmol, 76% yield).

M.P. 167-168 °C; **^{1}H NMR** (300 MHz, Chloroform-d) δ 7.57-7.38 (m, 3H), 7.30-7.22 (m, 1H), 5.81-5.65 (m, 2H), 2.51 (dddd, *J* = 10.9, 9.2, 5.7, 2.9 Hz, 1H), 2.43-2.26 (m, 2H), 2.26-2.07 (m,

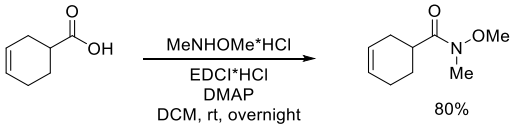
2H), 2.00 (dddd, *J* = 17.5, 6.2, 4.4, 2.7 Hz, 1H), 1.80 (dddd, *J* = 12.9, 11.1, 9.5, 6.5 Hz, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 174.13, 136.55, 129.15, 129.00, 126.96, 125.11, 121.04, 42.30, 28.00, 25.75, 24.51; **HRMS (ESI)** Calcd. for $C_{13}H_{15}ClNO$ ($[M+H]^+$): 236.0837, found: 236.0839.



***N*-(4-methoxyphenyl)cyclohex-3-enecarboxamide (1o)**

Using Procedure D on 10 mmol scale with 4-methoxyaniline as the amine partner, the product **1o** was obtained as an off white solid (1.3 g, 5.6 mmol, 56% yield).

M.P. 136-138 °C; **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.46-7.40 (m, 2H), 7.15 (s, 1H), 6.89-6.83 (m, 2H), 5.78-5.69 (m, 2H), 3.79 (s, 3H), 2.54-2.44 (m, 1H), 2.44-1.96 (m, 5H), 1.88-1.75 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 174.03, 156.54, 131.28, 127.13, 125.48, 121.84, 114.34, 55.71, 42.39, 28.31, 26.02, 24.80; **HRMS (ESI)** Calcd. for $C_{14}H_{18}NO_2$ ($[M+H]^+$): 232.1333, found: 232.1327.



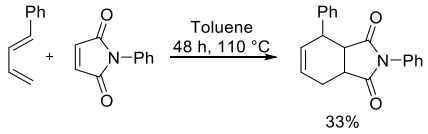
***N*-methoxy-*N*-methylcyclohex-3-enecarboxamide**

Using a modification of Procedure D (2.0 eq. instead of 1.0 eq. of DMAP) on 15 mmol scale with *N*,*O*-dimethylhydroxylamine hydrochloride as the amine partner, the product was obtained as a colorless liquid (2.02 g, 11.9 mmol, 80% yield).

^{1}H NMR (300 MHz, Chloroform-d) δ 5.79-5.63 (m, 2H), 3.70 (s, 3H), 3.20 (s, 3H), 2.92 (m, 1H), 2.39-2.20 (m, 1H), 2.19-2.04 (m, 3H), 1.93-1.80 (m, 1H), 1.80-1.60 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 177.20, 126.44, 125.81, 61.57, 36.05, 32.32, 27.67, 25.34, 24.89; **HRMS (ESI)** Calcd. for $C_9H_{15}NO_2$ ($[M]^+$): 169.1098, found: 169.1091.

Synthesis of substituted tetrahydrophthalimides

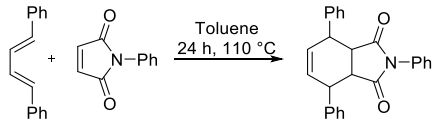
The following procedures were not optimized.



2,4-diphenyl-3a,4,7,7a-tetrahydro-1H-isoindole-1,3(2H)-dione (3a)

In a pressure tube, 1-phenyl-1,3-butadiene (779 mg, 4.5 mmol) and N-phenylmaleimide (755 mg, 5.8 mmol) were added in toluene (2.5 ml). The tube was capped and placed in an oil bath at 110 °C. The reaction was stirred for 48 h. The reaction crude was taken directly to column chromatography (Hexane:Ethyl Acetate). The product was obtained as a white solid in 33% yield.

M.P. 98-99 °C; **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.33 – 7.25 (m, 6H), 7.20 (dd, *J* = 7.6, 1.8 Hz, 2H), 6.59 (dd, *J* = 7.3, 2.1 Hz, 2H), 6.25 – 6.09 (m, 2H), 3.97 (t, *J* = 6.2 Hz, 1H), 3.47 (dd, *J* = 9.0, 7.3 Hz, 1H), 3.37 (td, *J* = 9.6, 3.0 Hz, 1H), 3.06 – 2.93 (m, 1H), 2.59 (ddd, *J* = 18.1, 10.2, 3.6 Hz, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 178.81, 176.74, 138.46, 131.71, 129.54, 129.23, 129.04, 128.74, 128.57, 127.69, 127.18, 126.47, 44.84, 41.04, 38.15, 21.86; **HRMS (ESI)** Calcd. for $C_{20}H_{18}NO_2$ ($[M+H]^+$): 304.1333, found: 304.1345.



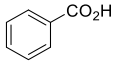
80%

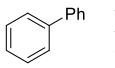
2,4,7-triphenyl-3a,4,7,7a-tetrahydro-1H-isoindole-1,3(2H)-dione (3b)

In a 100 ml Schlenk flask, 1,4-diphenyl-1,3-butadiene (1.24 g, 6.0 mmol) and N-phenylmaleimide (865.8 mg, 5 mmol) were added in toluene (5 ml). The reaction mixture was heated at 110 °C for 24 h. Upon cooling, a white solid crashed out. The toluene was decanted and the solid was washed 4 times with diethyl ether. The solid was dried to provide the desired product as a white solid in 80% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.45 – 7.24 (m, 13H), 7.06 – 6.94 (m, 2H), 6.60 – 6.48 (m, 2H), 3.93 (d, *J* = 4.9 Hz, 2H), 3.66 (dd, *J* = 4.9, 2.2 Hz, 2H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 175.08, 139.34, 131.81, 131.58, 129.14, 129.06, 128.60, 128.45, 127.41, 126.47, 46.74, 41.92; **HRMS (ESI)** Calcd. for $C_{26}H_{22}NO_2$ ($[M+H]^+$): 380.1650, found: 380.1646.

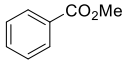
Spectral Data for Dehydrogenation Products

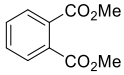
 **Benzoic Acid (2a)**[3] Yield: 97%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 12.67 (s, 1H), 8.21-8.07 (m, 2H), 7.71-7.54 (m, 1H), 7.52-7.46 (m, 2H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 172.46, 133.86, 130.25, 129.34, 128.52.

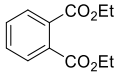
 **Biphenyl (2b)**[4] Yield: 75%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.67-7.53 (m, 2H), 7.52-7.39 (m, 2H), 7.39-7.29 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 141.27, 128.78, 127.28, 127.20.

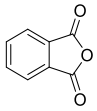
 **1,2,3,4-tetrahydro-1,4-methanonaphthalene (2c)**[5] Yield: 80%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.21 – 7.10 (m, 2H), 7.10 – 7.00 (m, 2H), 3.34 (s, 2H), 1.97 – 1.81 (m, 2H), 1.79 – 1.69 (m, 1H), 1.56 – 1.46 (m, 1H), 1.23 – 1.10 (m, 2H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 148.41, 125.61, 120.64, 49.54, 43.89, 27.23.

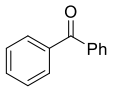
 **Naphthalene (2d)**[6] Yield: 78%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.89 – 7.80 (m, 4H), 7.52 – 7.43 (m, 4H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 133.48, 127.92, 125.86.

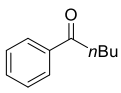
 **Methyl Benzoate (2e)**[7] Yield: 90%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 8.12 – 7.96 (m, 2H), 7.64 – 7.51 (m, 1H), 7.51 – 7.36 (m, 2H), 3.91 (s, 3H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 167.23, 133.05, 130.30, 129.71, 128.50, 52.23.

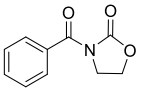
 **Phthalic acid dimethyl ester (2f)**[8] Yield: 95%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.81 – 7.65 (m, 2H), 7.63 – 7.45 (m, 2H), 3.90 (s, 6H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 168.06, 131.92, 131.13, 128.88, 52.67.

 **Phthalic acid diethyl ester (2g)**[9] Yield: 89%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.78 – 7.67 (m, 2H), 7.57 – 7.47 (m, 2H), 4.37 (q, *J* = 7.2 Hz, 4H), 1.37 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 167.78, 132.39, 131.10, 128.99, 61.77, 14.27.

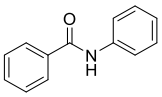
 **Phthalic Anhydride (2h)**[10] Yield: 85%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 8.10 – 7.99 (m, 2H), 7.99 – 7.85 (m, 2H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 162.77, 136.04, 131.36, 125.73.

 **Benzophenone (2i)**[11] Yield: 93%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.89 – 7.72 (m, 4H), 7.69 – 7.55 (m, 2H), 7.55 – 7.40 (m, 4H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 196.79, 137.61, 132.44, 130.08, 128.30.

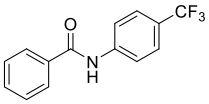
 **n-Butyl phenyl ketone (2j)**[12] Yield: 62%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 8.05 – 7.86 (m, 2H), 7.55 (h, 1H), 7.51 – 7.41 (m, 2H), 2.97 (t, *J* = 7.4 Hz, 2H), 1.73 (p, 2H), 1.47 – 1.34 (h, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 200.82, 137.29, 133.06, 128.74, 128.25, 38.55, 26.70, 22.70, 14.16.



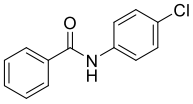
3-Benzoyloxazolidin-2-one (2k)[13] Yield: 58%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.74 – 7.62 (m, 2H), 7.62 – 7.52 (m, 1H), 7.52 – 7.39 (m, 2H), 4.50 (dd, *J* = 8.3, 7.3 Hz, 2H), 4.19 (dd, *J* = 8.3, 7.3 Hz, 2H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 169.83, 153.21, 132.59, 132.46, 129.12, 127.91, 62.26, 43.74.



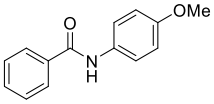
***N*-Phenylbenzamide (2l)**[14] Yield: 97%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.92 – 7.81 (m, 3H), 7.69 – 7.61 (m, 2H), 7.58 – 7.52 (m, 1H), 7.48 (dd, *J* = 8.2, 6.6 Hz, 2H), 7.37 (dd, *J* = 8.5, 7.4 Hz, 2H), 7.15 (t, *J* = 7.4 Hz, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 165.75, 137.93, 135.03, 131.87, 129.13, 128.82, 127.03, 124.60, 120.21.



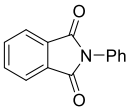
***N*-(4-(trifluoromethyl)phenyl)benzamide (2m)**[15] Yield: 86%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.97 – 7.84 (m, 3H), 7.79 (d, *J* = 8.5 Hz, 2H), 7.64 (d, *J* = 8.6 Hz, 2H), 7.62 – 7.56 (m, 1H), 7.55 – 7.49 (m, 2H); **^{13}C NMR** (101 MHz, Acetone-d6) δ 165.75, 142.85, 134.78, 131.72, 128.37, 127.45, 125.77 (q, *J* = 3.9 Hz),124.59 (q, *J* = 269.9 Hz), 124.55 (q, *J* = 32.4 Hz), 119.80.



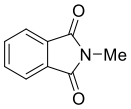
***N*-(4-Chlorophenyl)benzamide (2n)**[14] Yield: 88%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.89 – 7.84 (m, 2H), 7.80 (s, 1H), 7.63 – 7.58 (m, 2H), 7.56 (d, *J* = 7.4 Hz, 1H), 7.50 (dd, *J* = 8.2, 6.6 Hz, 2H), 7.38 – 7.30 (m, 2H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 165.66, 136.50, 134.65, 132.09, 129.58, 129.16, 128.90, 127.01, 121.40.



***N*-(4-Methoxyphenyl)benzamide (2o)**[14] Yield: 93%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.87 (d, *J* = 6.8 Hz, 2H), 7.70 (s, 1H), 7.59 – 7.43 (m, 5H), 6.92 (d, *J* = 9.0 Hz, 2H), 3.82 (s, 3H); **^{13}C NMR** (126 MHz, Chloroform-d) δ 165.58, 156.61, 135.02, 131.71, 130.96, 128.76, 126.95, 122.05, 114.23, 55.51.

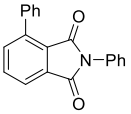


***N*-phenyl phthalimide (2p)**[16] Yield: 99%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.97 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.51 (d, *J* = 7.4 Hz, 2H), 7.48 – 7.38 (m, 3H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 167.31, 134.42, 131.79, 129.15, 128.14, 126.60, 126.40, 123.79.



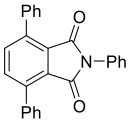
***N*-Methyl phthalimide (2q)**[17] Yield: 94%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.71 (dd, *J* = 5.4, 3.0 Hz, 2H), 3.19 (s, 3H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 168.71, 134.09, 132.46, 123.40, 24.17.

Benzonitrile (2r)[18] Yield: 62%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.69 – 7.64 (m, 2H), 7.61 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.48 (dd, *J* = 8.3, 7.1 Hz, 2H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 132.97, 132.36, 129.32, 119.05, 112.67.



2,4-diphenyl-isoindole-1,3-dione (4a) Yield: 79%. **M.P.** 163-165 °C; **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.96 (d, *J* = 7.3 Hz, 1H), 7.81 (t, *J* = 7.5 Hz, 1H), 7.72 (d, *J* = 7.7 Hz, 1H), 7.67 – 7.55 (m, 2H), 7.54 – 7.29 (m, 8H). **^{13}C NMR** (101 MHz, Chloroform-d) δ 167.14, 167.05, 141.76, 136.67, 136.25, 134.38, 133.11, 131.84,

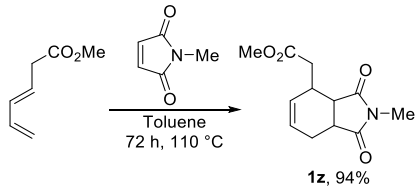
129.65, 129.19, 128.98, 128.31, 128.2, 127.27, 126.87, 122.85; **HRMS (ESI)** Calcd. for $C_{20}H_{14}NO_2$ ($[M+H]^+$): 300.1024, found: 300.1020.



2,4,7-triphenyl-isoindole-1,3-dione (4b)[19] Yield: 84%. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.74 (s, 2H), 7.66 – 7.58 (m, 4H), 7.53 – 7.44 (m, 6H), 7.43 – 7.32 (m, 5H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 166.63, 140.66, 136.60, 136.42, 131.78, 129.70, 129.04, 128.90, 128.28, 128.21, 128.12, 126.98; **HRMS (ESI)** Calcd. for $C_{26}H_{17}NO_2$ ($[M+H]^+$): 376.1337, found: 376.1133.

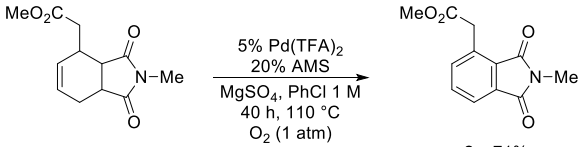
Synthesis of the Precursor to a TRPA1 Modulator

The isomerization of methyl sorbate to methyl (*E*)-3,5-hexadienoate (**7**) was conducted according to literature conditions.[20]



1z, 94%

Methyl 2-(2-methyl-1,3-dioxo-2,3,3a,4,7,7a-hexahydro-1H-isoindol-4-yl)acetate (3d)
In a pressure tube, methyl (*E*)-3,5-hexadienoate (**4**) (630.8 mg, 5.0 mmol) and *N*-methylmaleimide (422.2 mg, 3.8 mmol) were added in toluene (2 ml). The tube was heated at 110 °C for 72 hours. The toluene was removed on the rotatory evaporator and the crude mixture was subjected to column chromatography (Hexane, then Hexane:Ethyl acetate from 80:20 to 0:100). The product was obtained as viscous, yellow oil (847.1 mg, 3.6 mmol, 94% yield).
^{1}H NMR (400 MHz, Chloroform-d) δ 5.91 (ddt, *J* = 9.8, 7.1, 3.0 Hz, 1H), 5.69 (dt, *J* = 9.3, 3.3 Hz, 1H), 3.73 (s, 3H), 3.32 (dd, *J* = 8.9, 5.9 Hz, 1H), 3.21 – 3.05 (m, 2H), 2.91 (s, 3H), 2.86 – 2.69 (m, 3H), 2.26 – 2.13 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 179.95, 178.30, 173.16, 132.44, 128.32, 51.94, 42.35, 40.32, 35.57, 32.03, 24.94, 24.35; **HRMS (ESI)** Calcd. for $C_{12}H_{12}NO_4$ ($[M+H]^+$): 238.1074, found: 238.1077.



1z **2z**, 71% (average of two runs)

Methyl-(2-Methyl-1,3-dioxo-2,3-dihydro-1H-isoindol-4-yl)acetate (4d)
The dehydrogenation reaction was conducted on 1.0 mmol scale in a test tube with an O_2 balloon (Procedure B). Yield was based on an average of two reactions (164.4mg, 69% and 170.0 mg, 73% yield, respectively). Product was obtained as an off white solid.
M.P. 86-87 °C; **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.78 (d, *J* = 7.6 Hz, 1H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.54 (d, *J* = 7.7 Hz, 1H), 4.17 (s, 2H), 3.74 (s, 3H), 3.16 (s, 3H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 170.97, 168.94, 168.30, 136.30, 133.93, 133.07, 132.86, 129.76, 122.44, 52.45, 36.25, 23.99; **HRMS (ESI)** Calcd. for $C_{12}H_{12}NO_4$ ($[M+H]^+$): 234.0761, found: 234.0766.

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NMR Spectra

for

Palladium-Catalyzed Aerobic Oxidative Dehydrogenation of Cyclohexenes to Substituted Arene Derivatives

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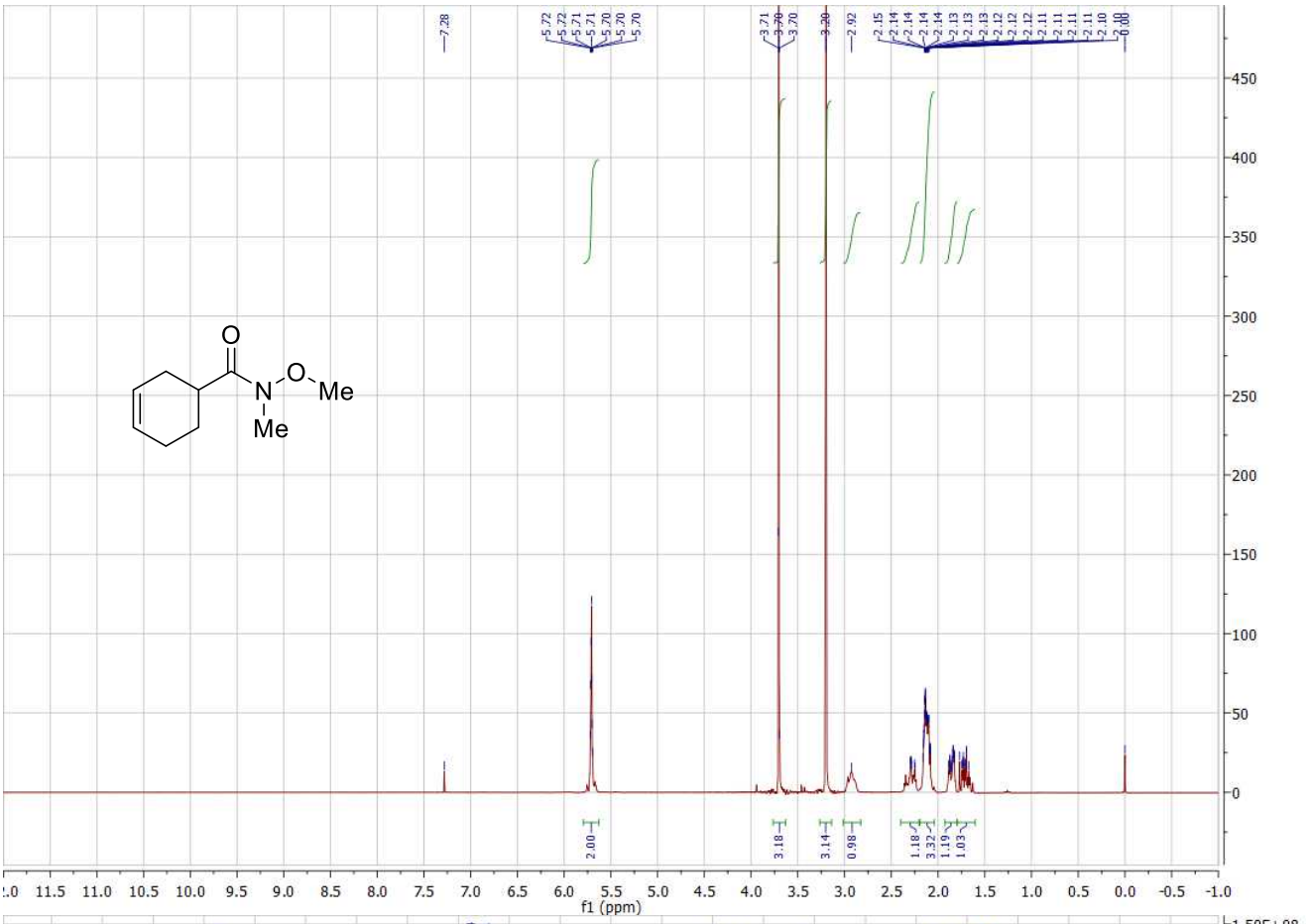
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5.70
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3.70
3.70
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2.14
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2.11
2.11
2.11
2.11
2.10
0.00
O
N
O
Me
Me
2.00
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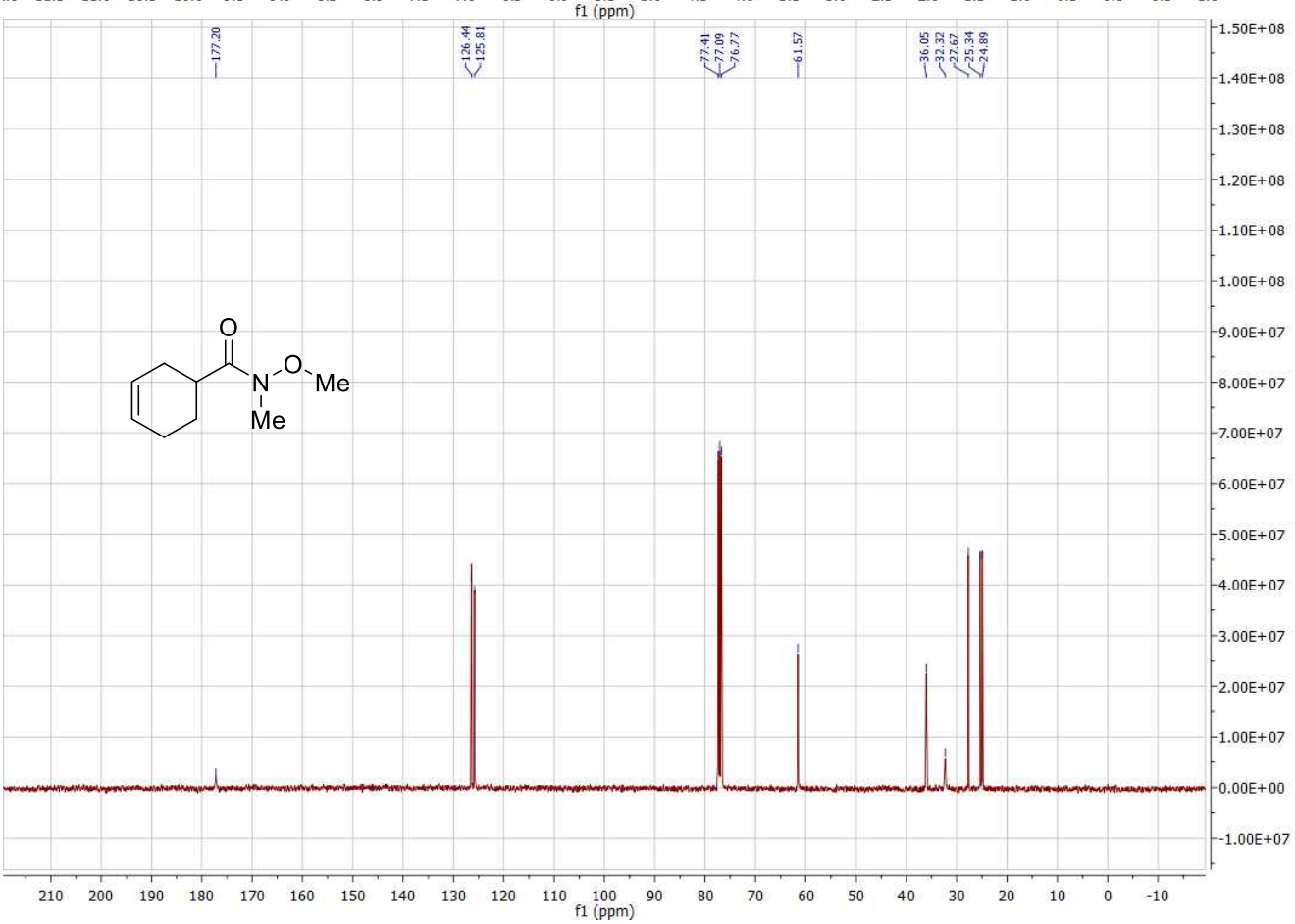
1.18
3.32
1.19
1.03
f1 (ppm)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N
O
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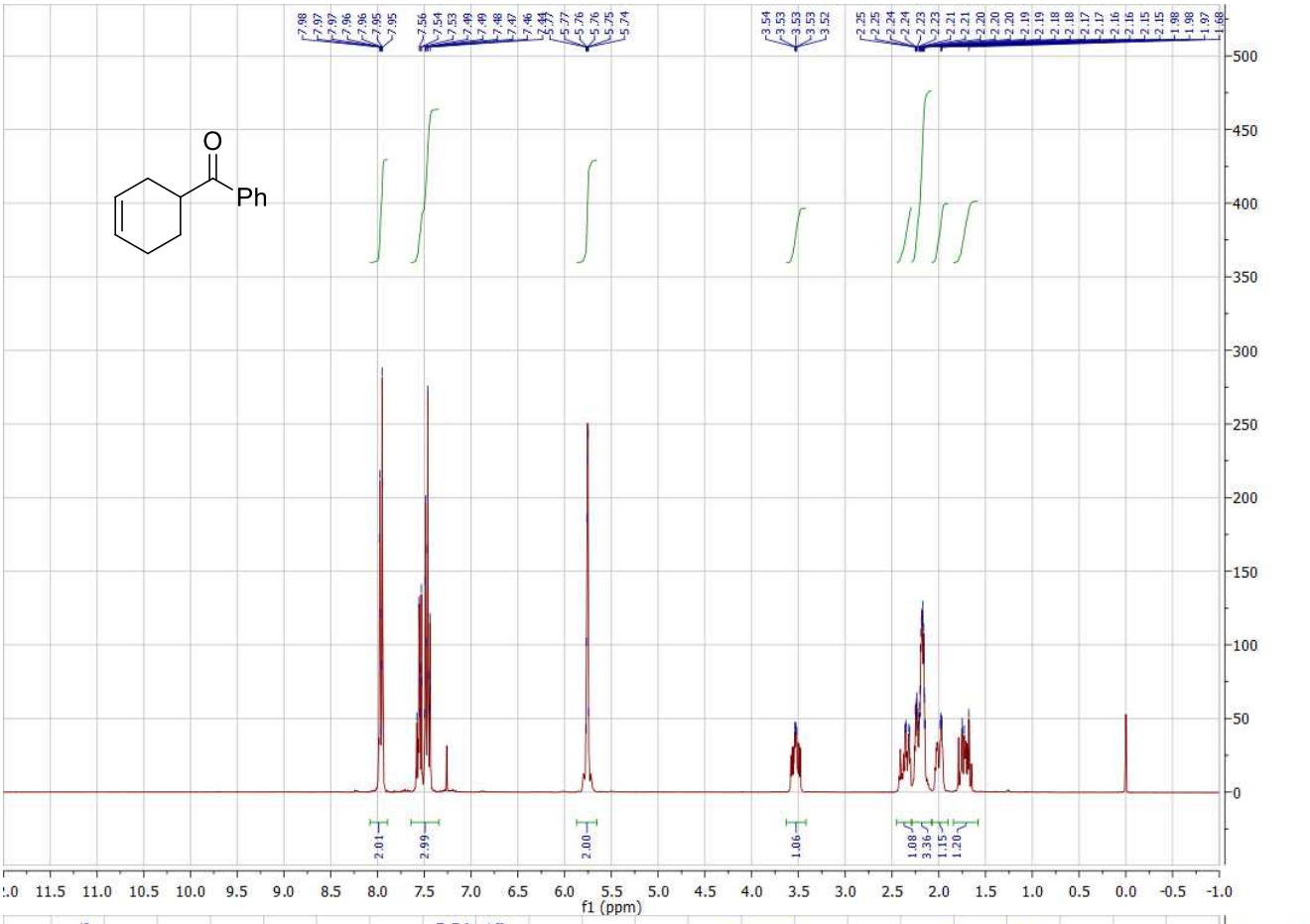
Me
f1 (ppm)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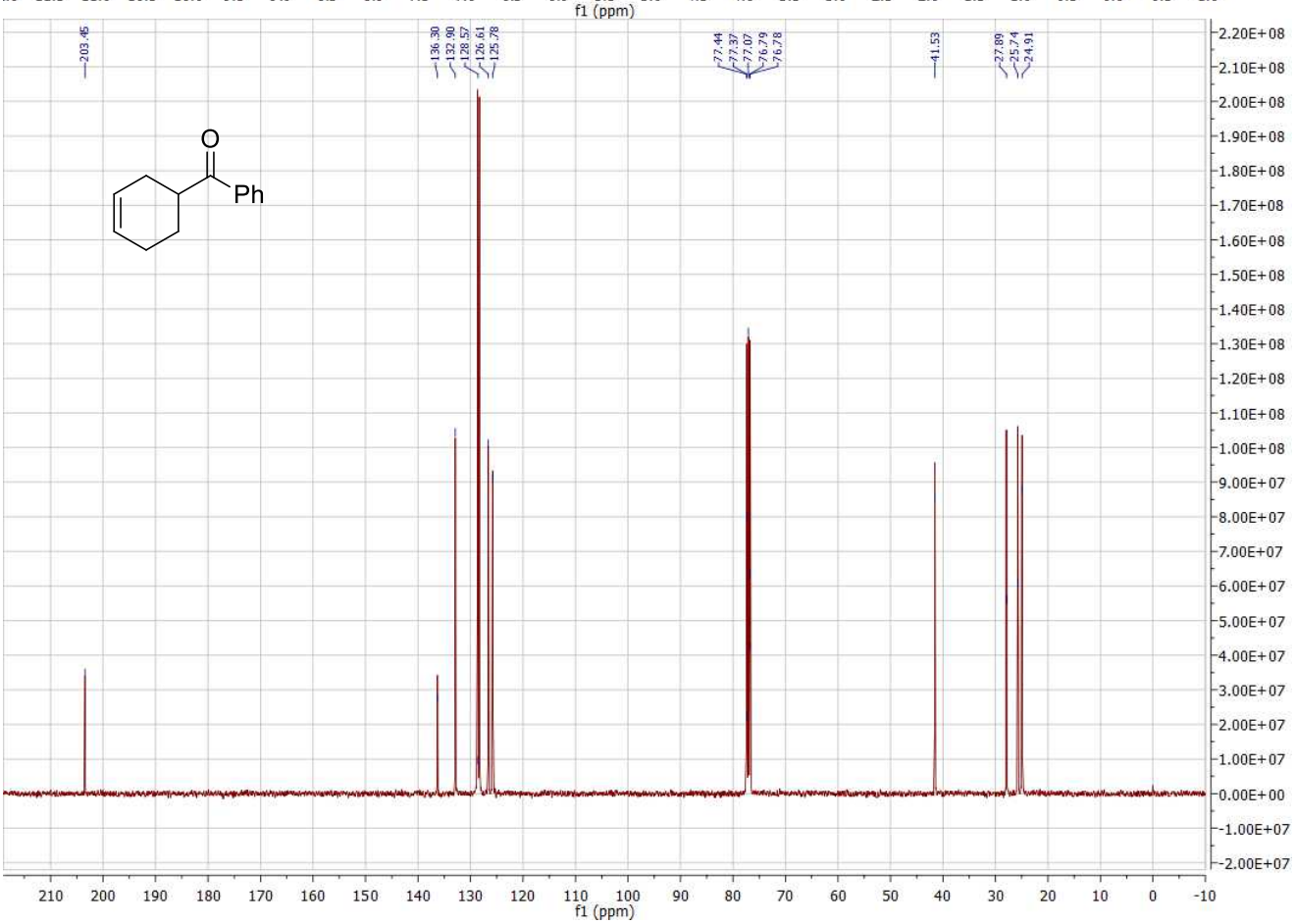
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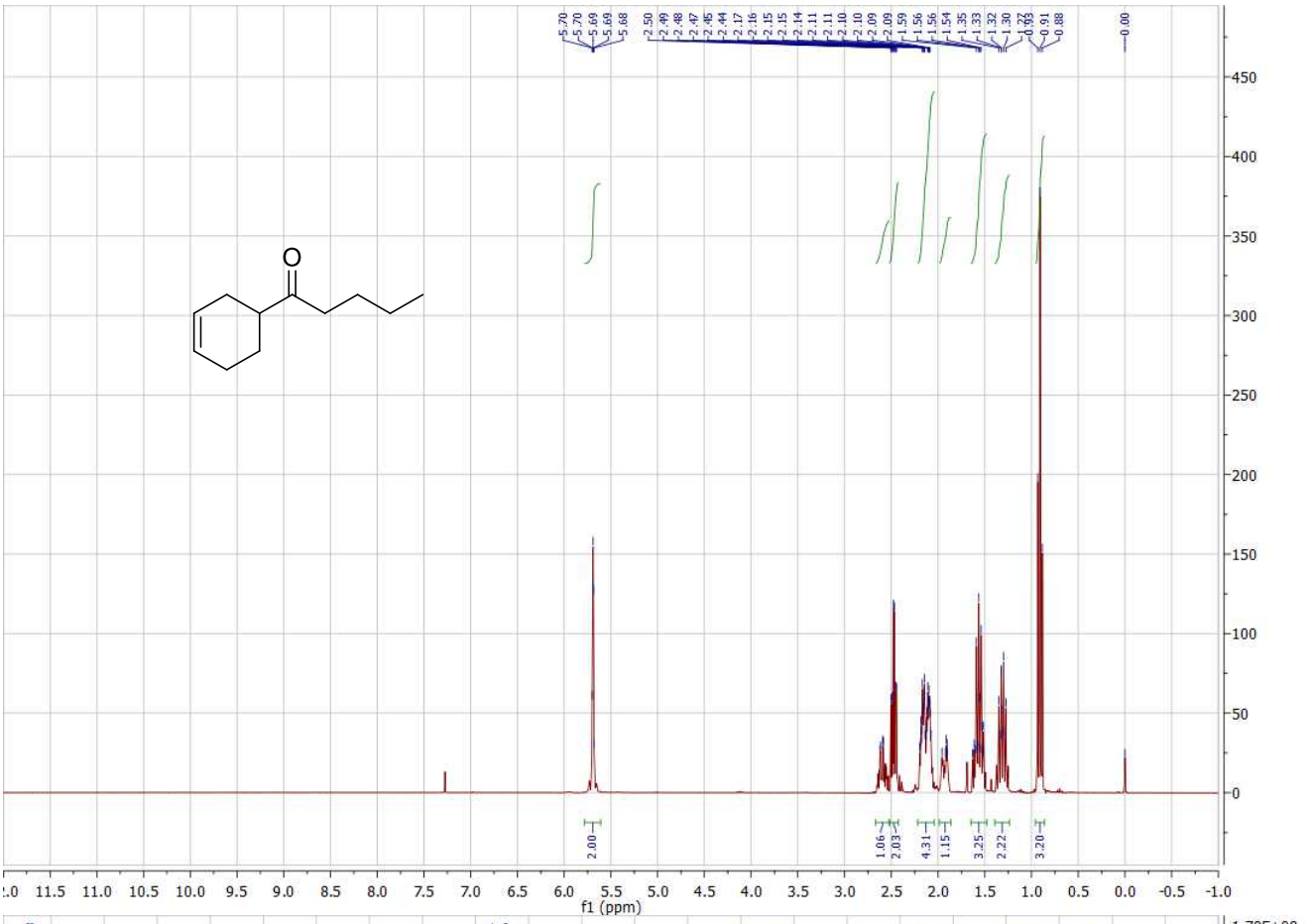
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2.11
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0.91
0.88
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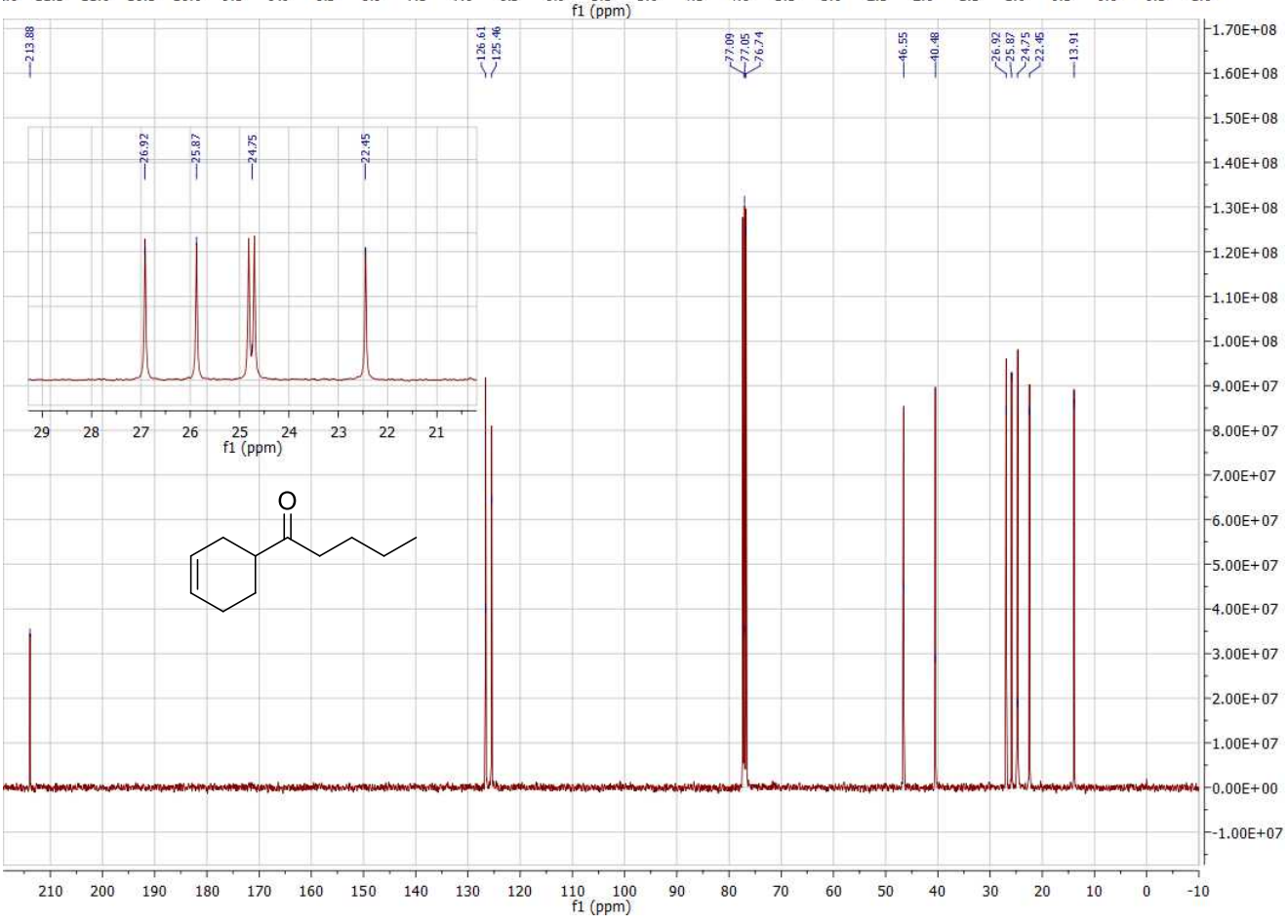
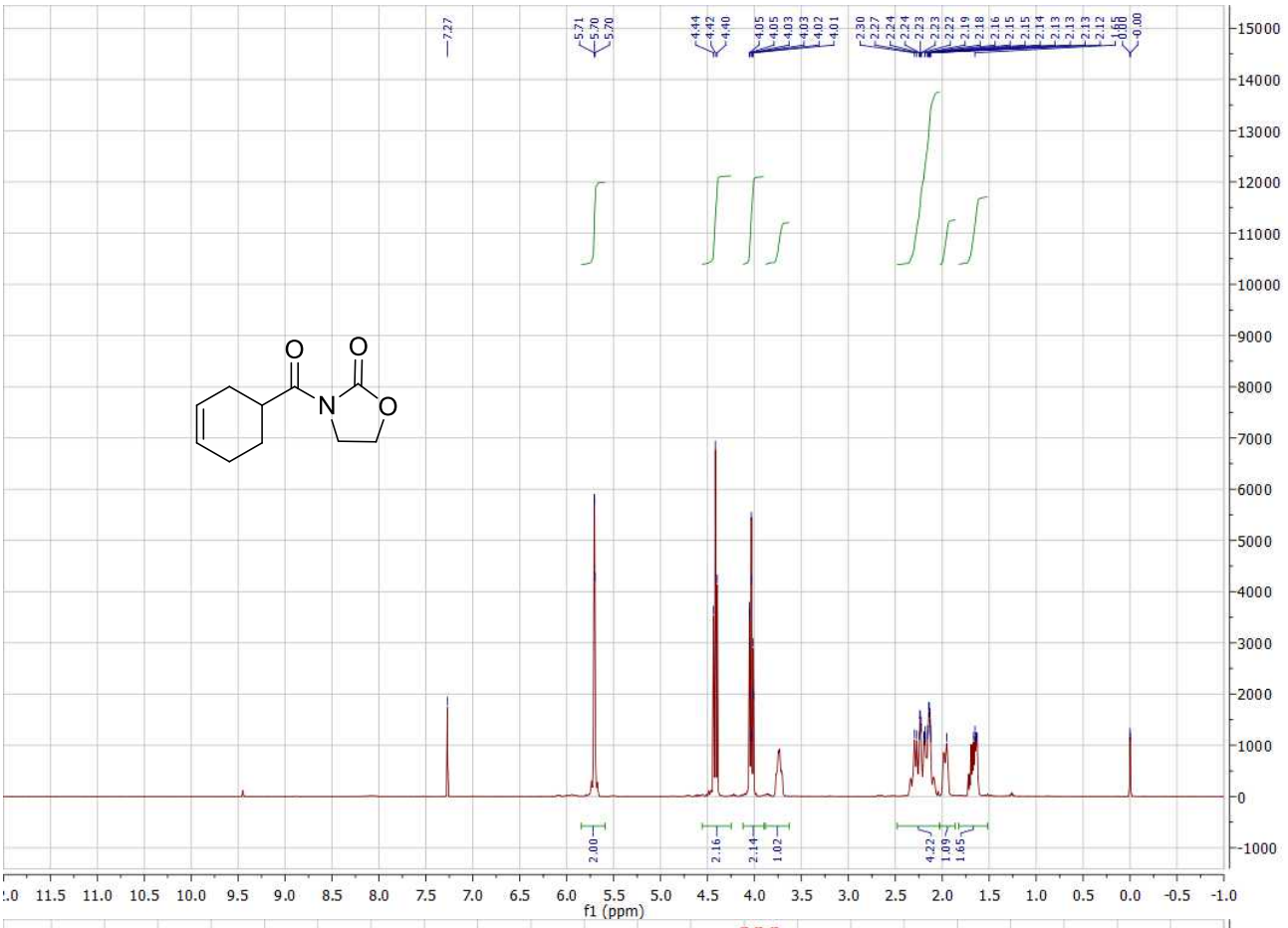
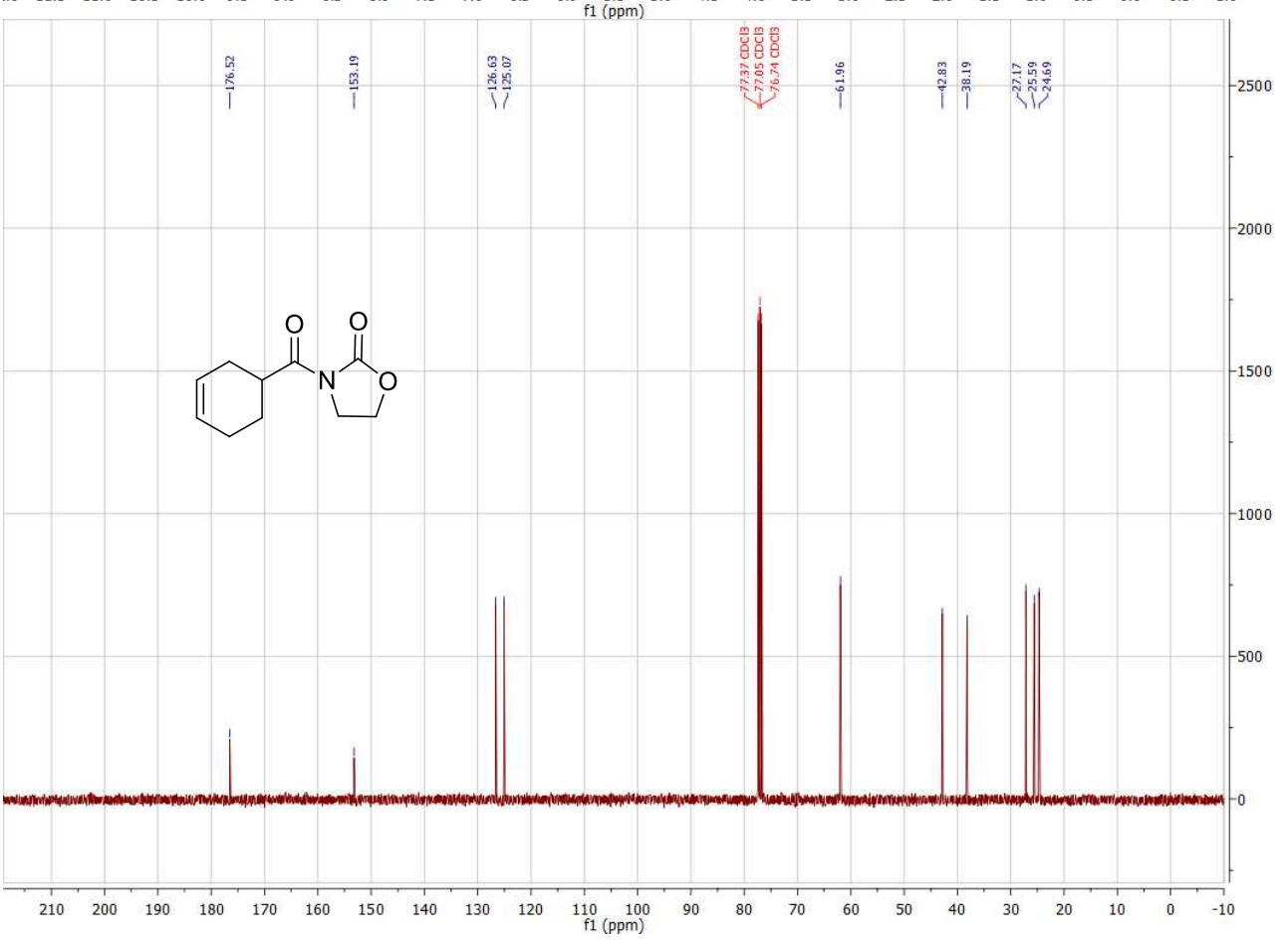
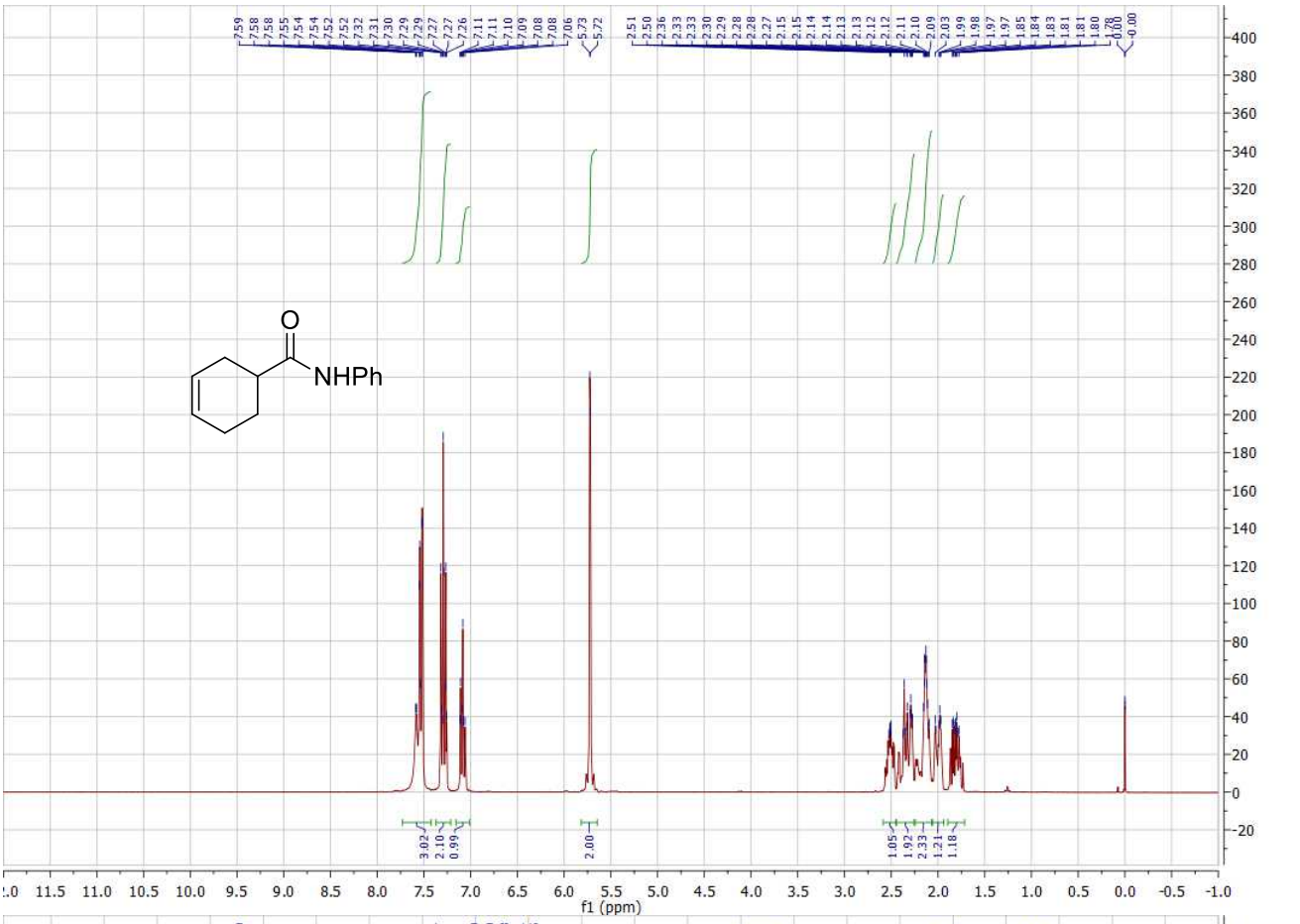
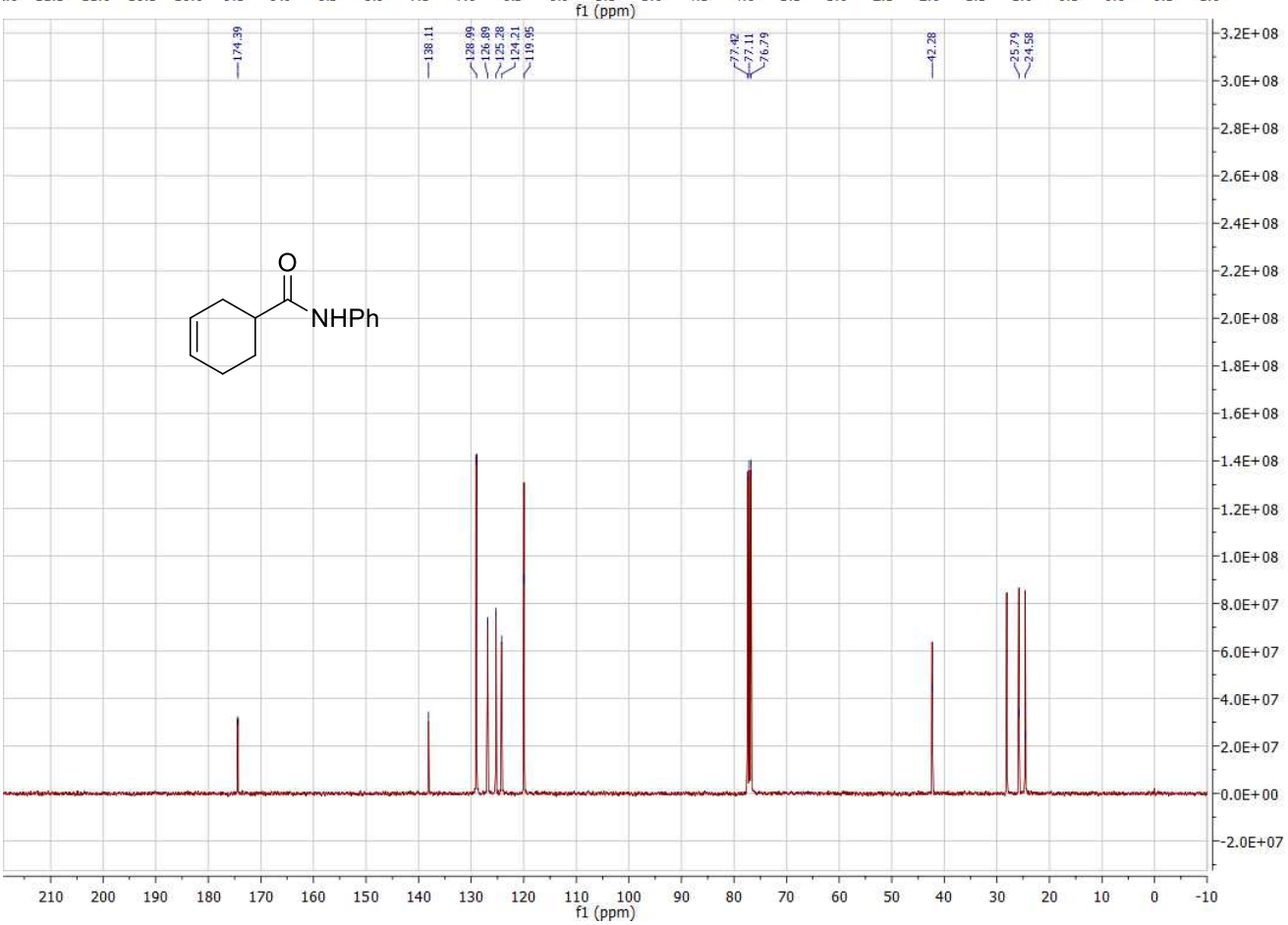
2.03
4.31
1.15
3.25
2.22
3.20
f1 (ppm)

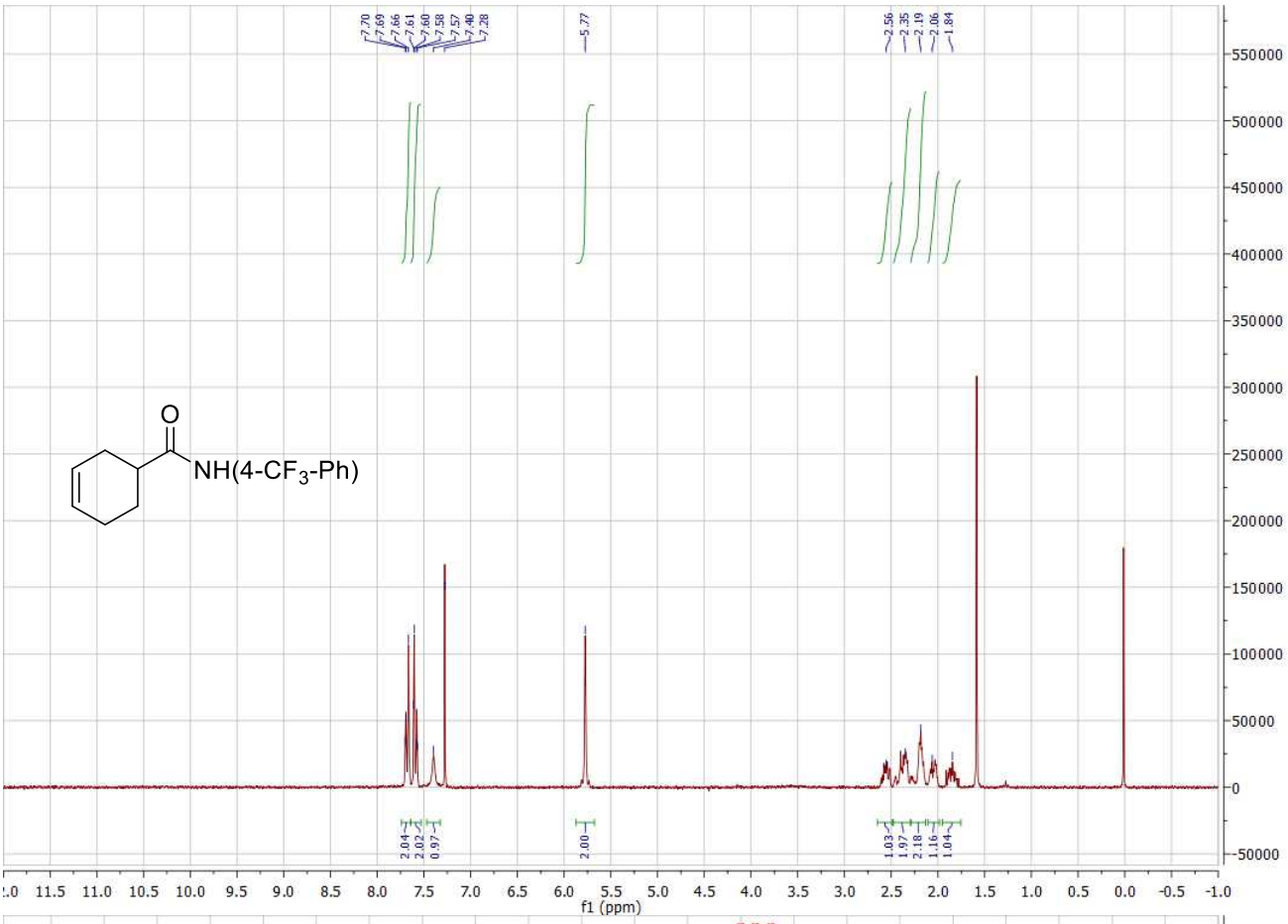

213.88
126.61
125.46
77.09
77.05
76.74
46.55
40.48
26.92
25.87
24.75
22.45
13.91
f1 (ppm)

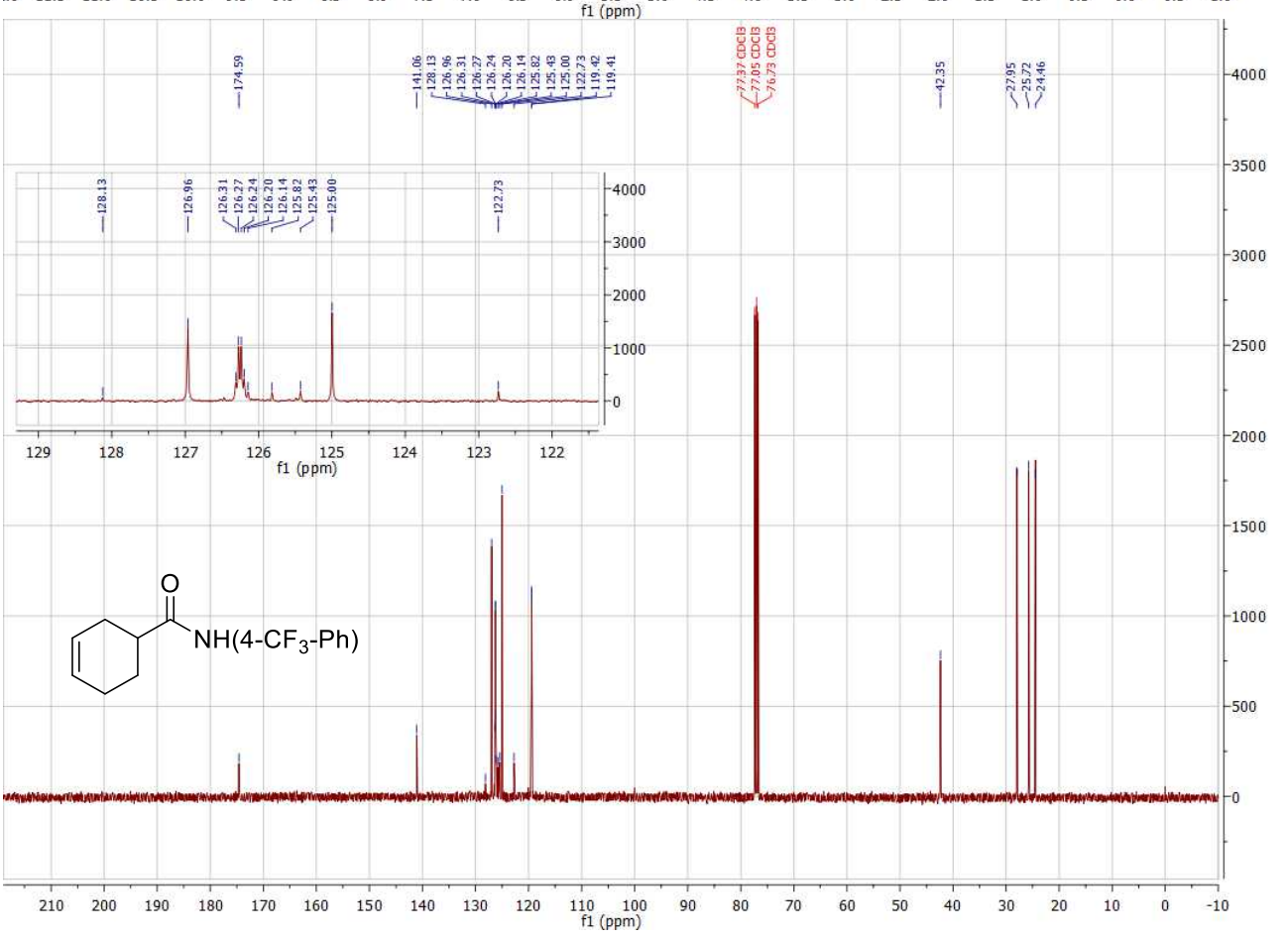

7.27
5.71
5.70
5.70
4.44
4.42
4.40
4.05
4.05
4.03
4.03
4.02
4.01
2.30
2.27
2.24
2.24
2.23
2.23
2.22
2.19
2.18
2.16
2.15
2.15
2.14
2.13
2.13
2.12
1.65
0.00
-0.00
2.00
2.16
2.14
1.02
4.22
1.09
1.65
f1 (ppm)

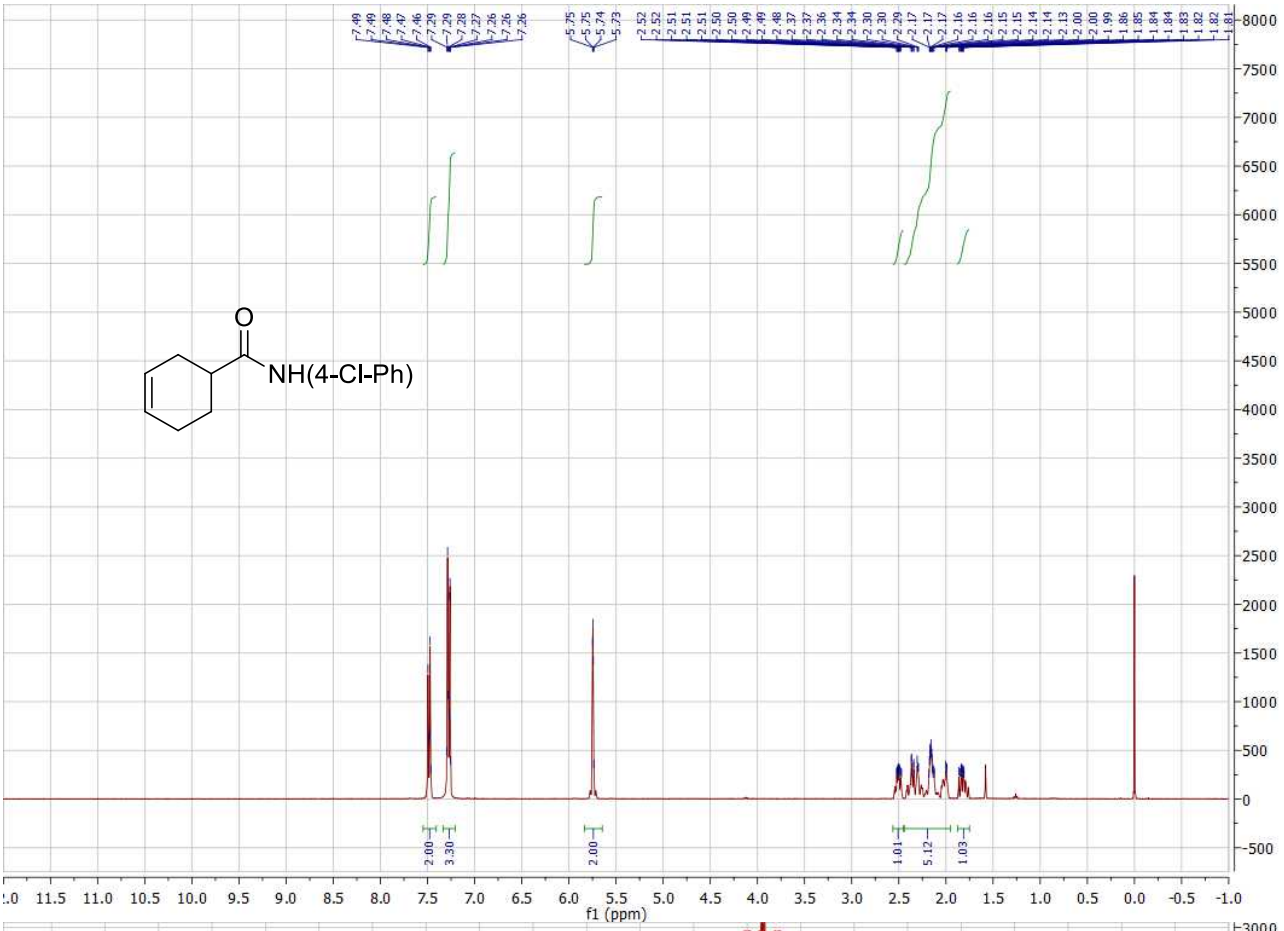

176.52
153.19
126.63
125.07
77.37 CDCl3
77.05 CDCl3
76.74 CDCl3
61.96
42.83
38.19
27.17
25.59
24.69
f1 (ppm)

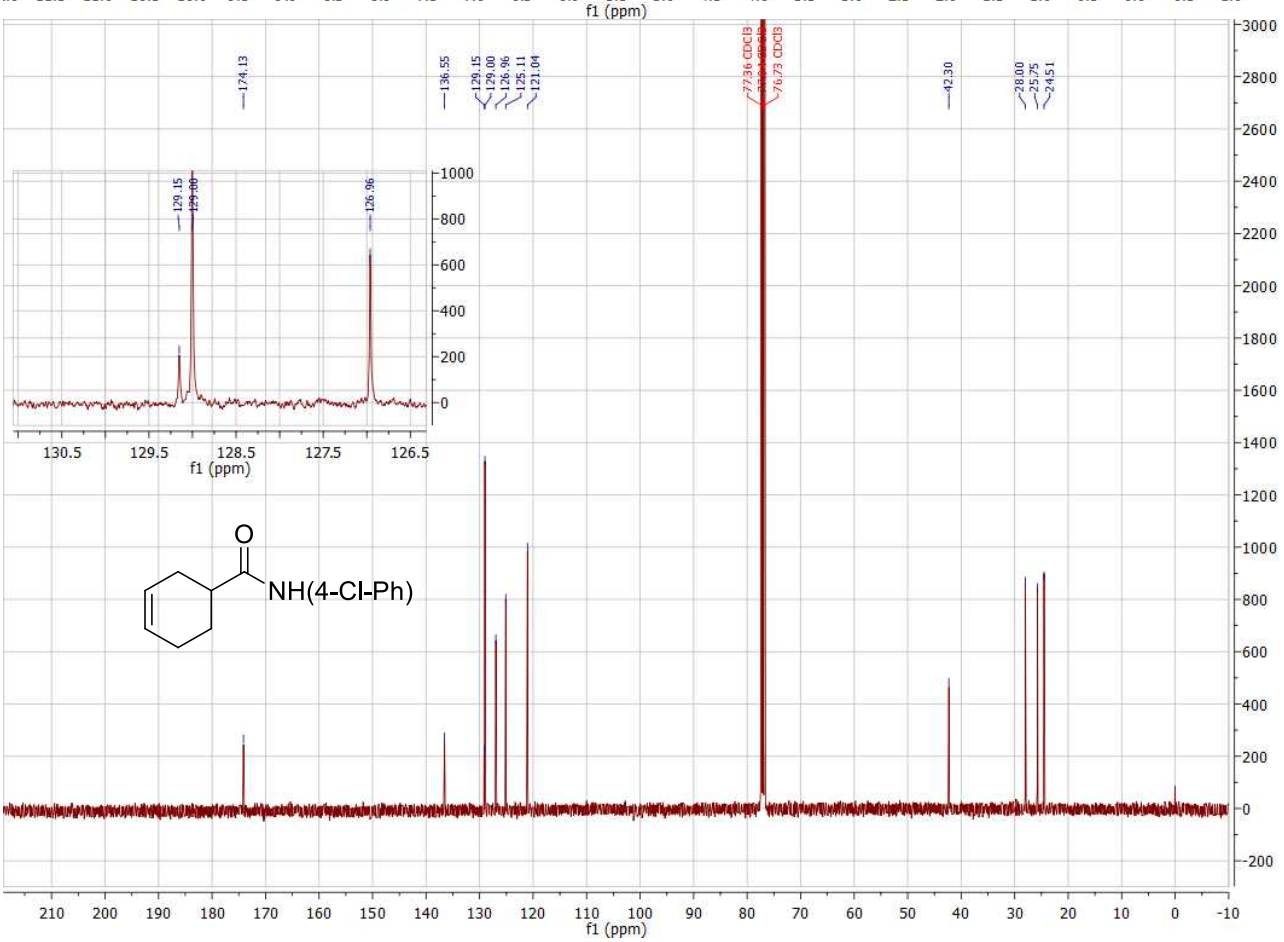

O
NHPh
f1 (ppm)

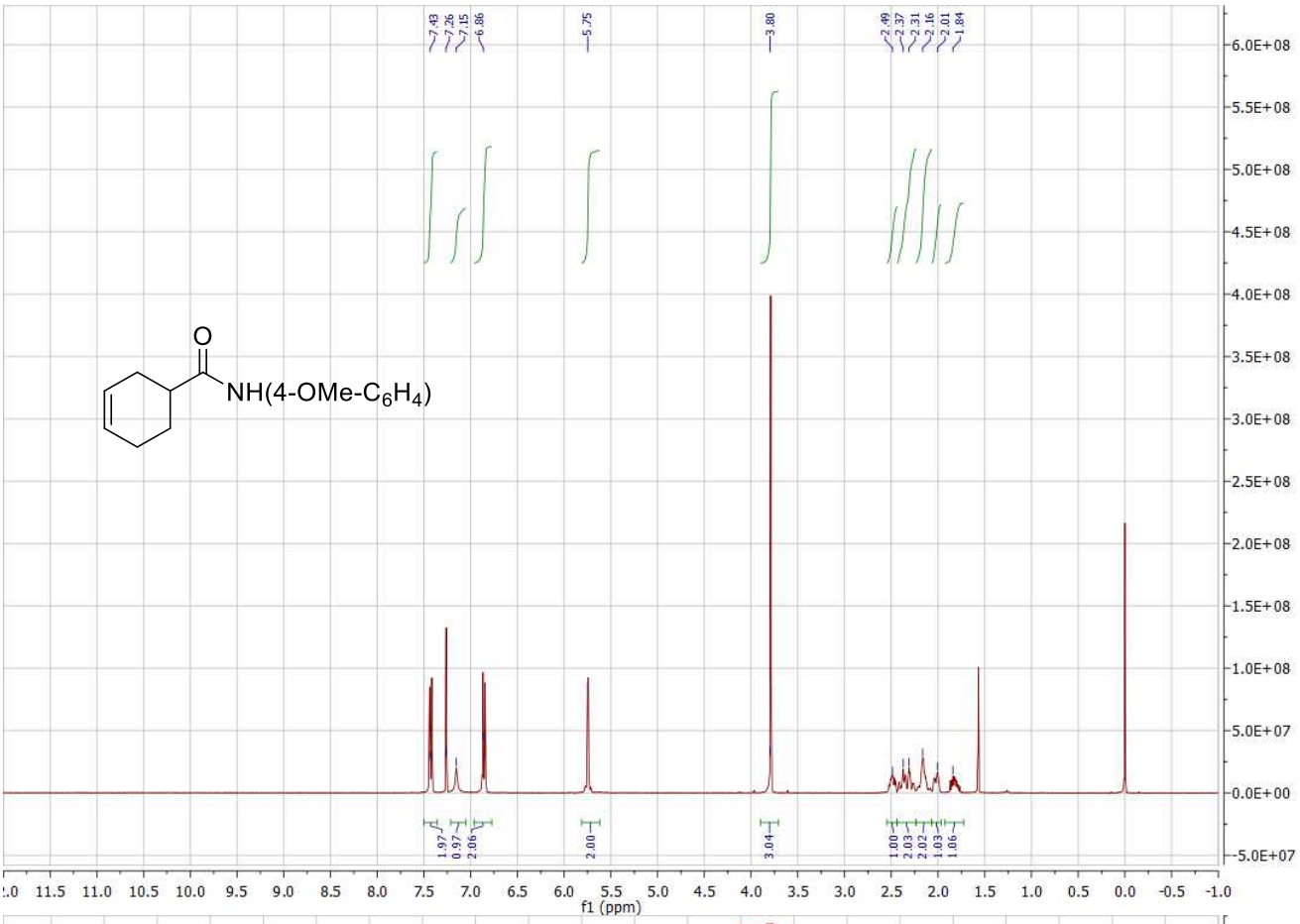

O
NHPh
f1 (ppm)

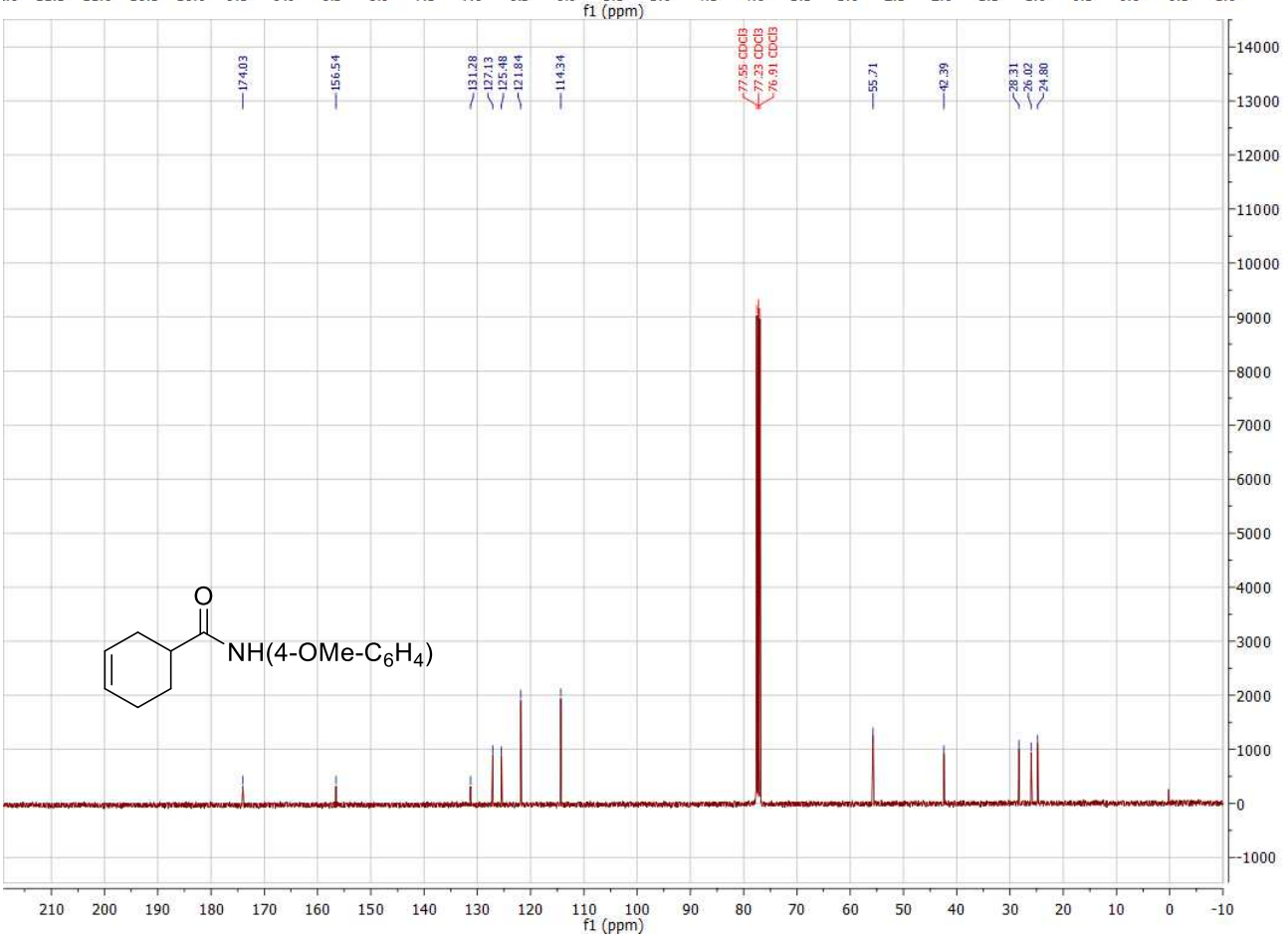

7.70
7.69
7.66
7.61
7.60
7.58
7.57
7.40
7.28
5.77
2.56
2.35
2.19
2.06
1.84
O
NH(4-CF3-Ph)
2.04
2.02
0.97
2.00
1.03
1.97
2.18
1.16
1.04
f1 (ppm)

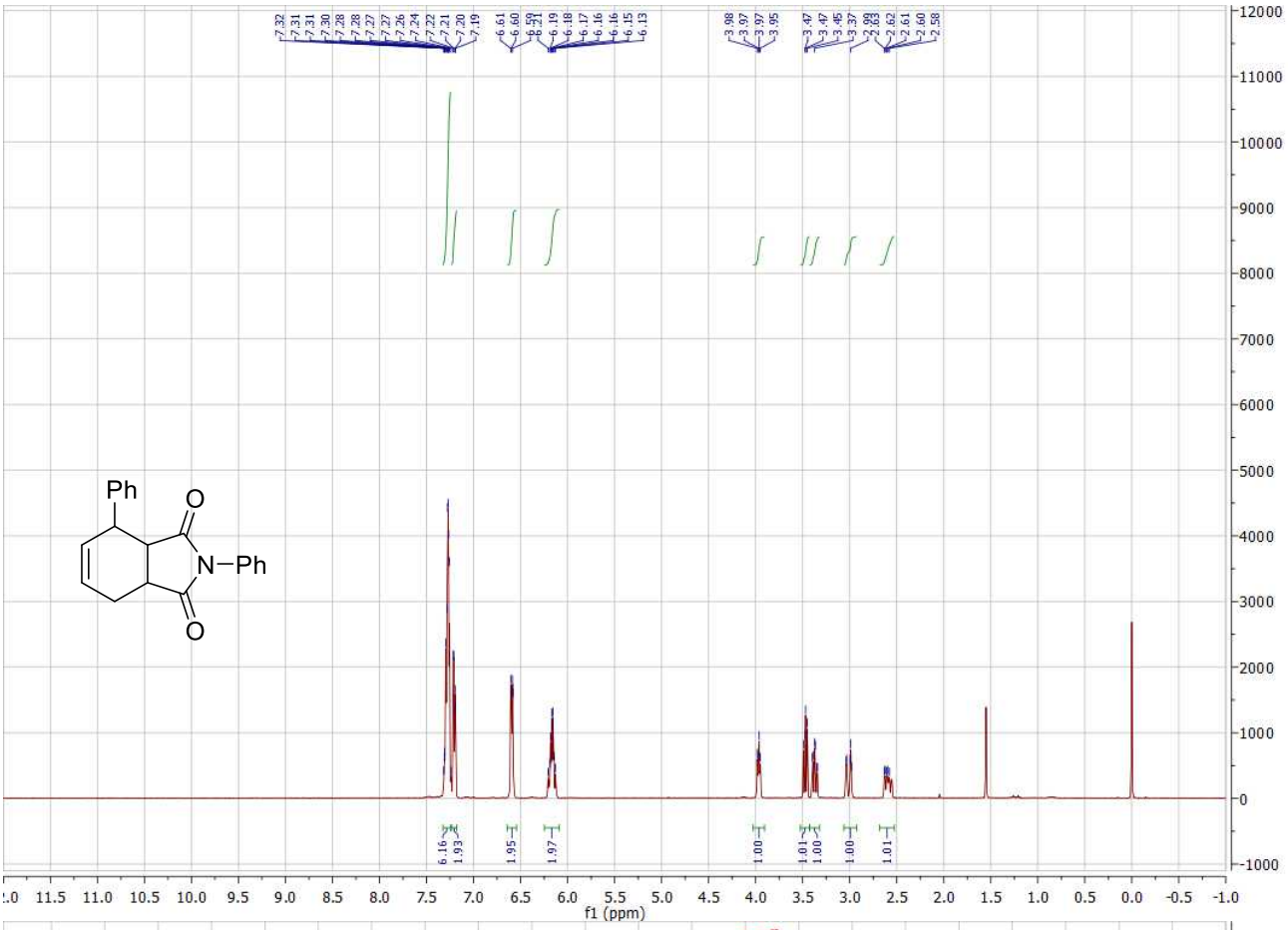

174.59
141.06
128.13
126.96
126.31
126.27
126.24
126.20
126.14
125.82
125.43
125.00
122.73
119.42
119.41
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
42.35
27.95
25.72
24.46
O
NH(4-CF3-Ph)
f1 (ppm)

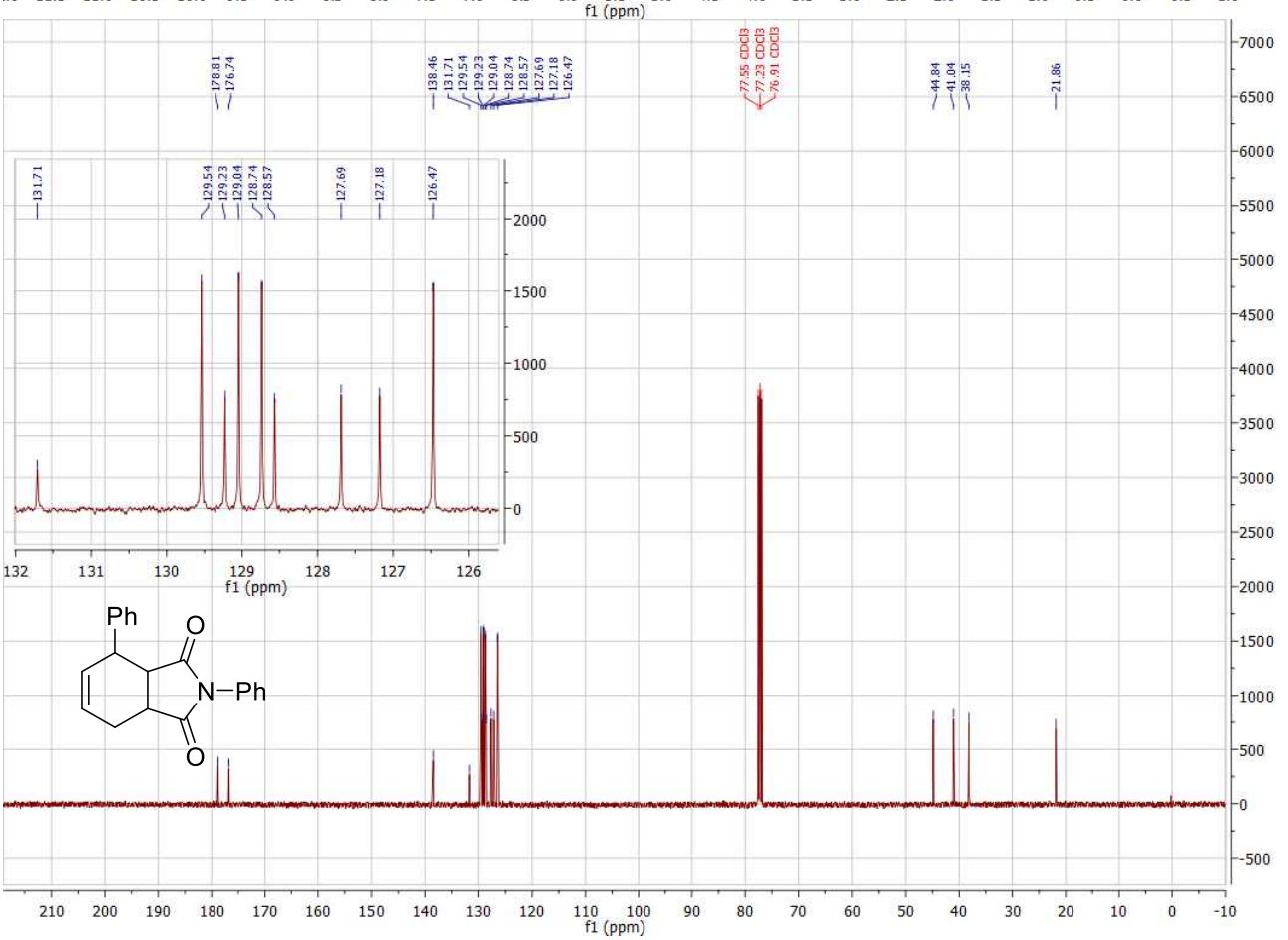

O
NH(4-Cl-Ph)
f1 (ppm)

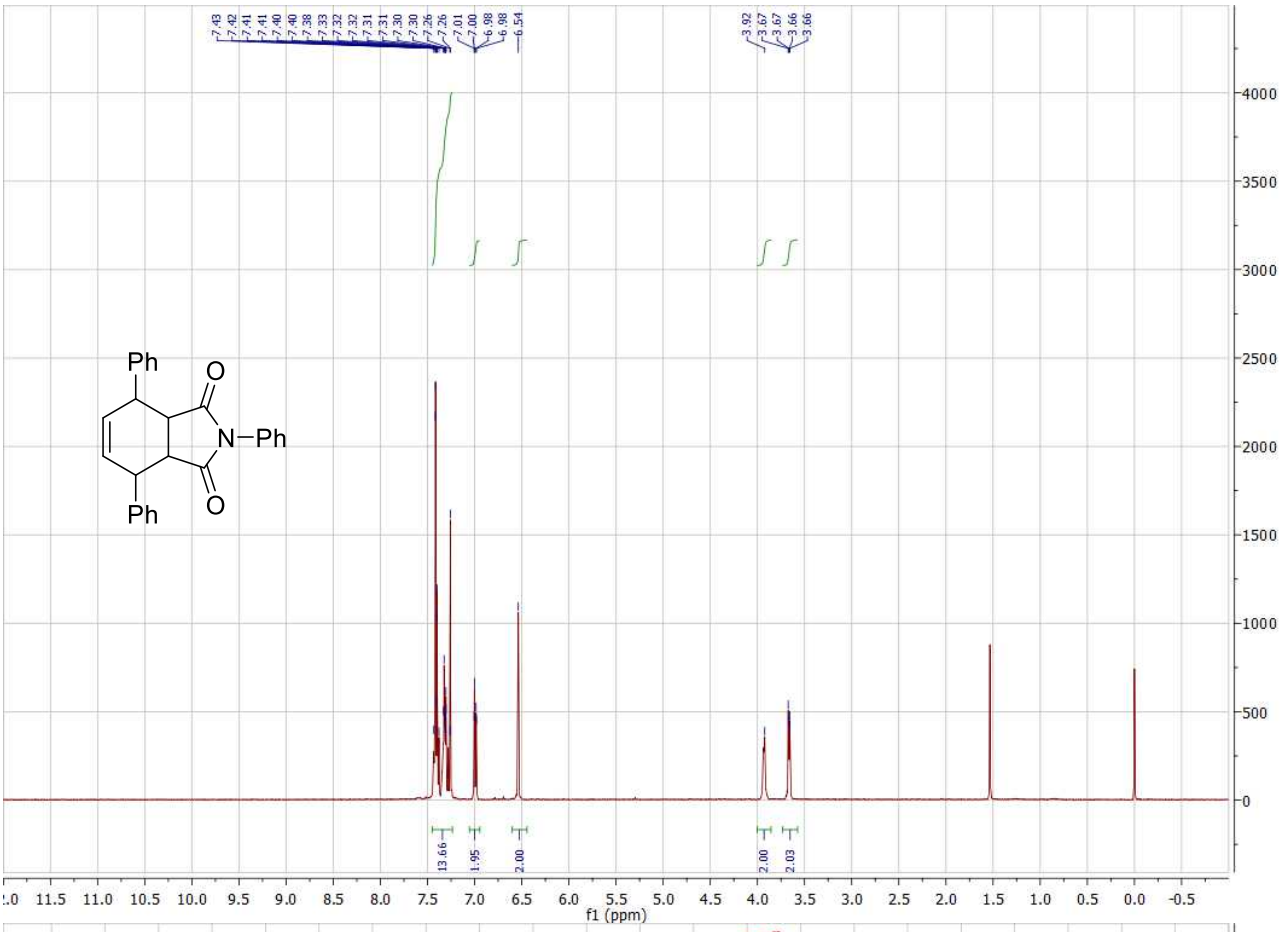

174.13
136.55
129.15
129.00
126.96
125.11
121.04
77.36 CDCl3
76.73 CDCl3
42.30
28.00
25.75
24.51
O
NH(4-Cl-Ph)
f1 (ppm)

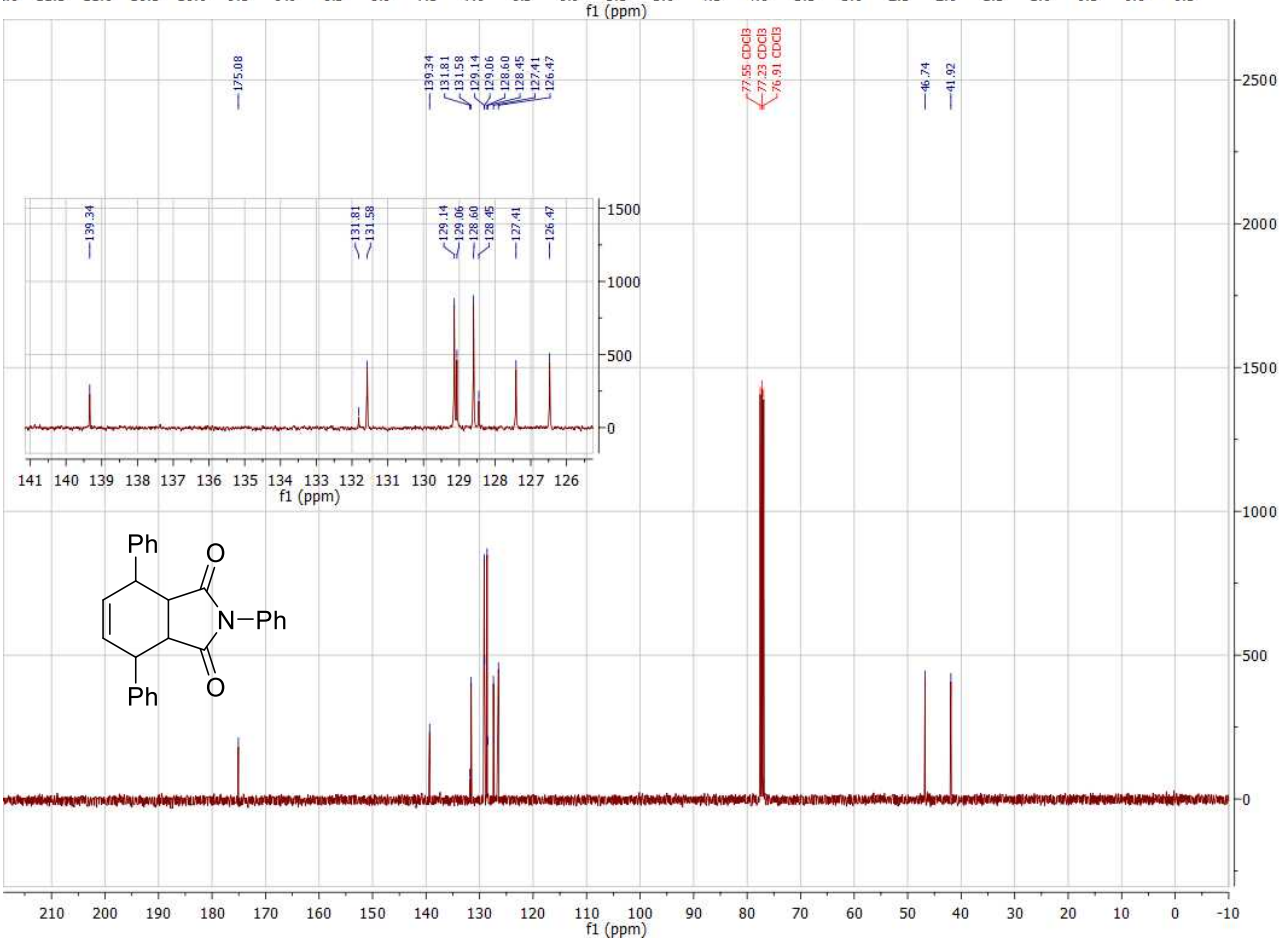

7.43
7.26
7.15
6.86
5.75
3.80
2.49
2.37
2.31
2.16
2.01
1.84
O
NH(4-OMe-C6H4)
1.97
0.97
2.06
2.00
3.04
1.00
2.03
2.02
1.03
1.06
f1 (ppm)

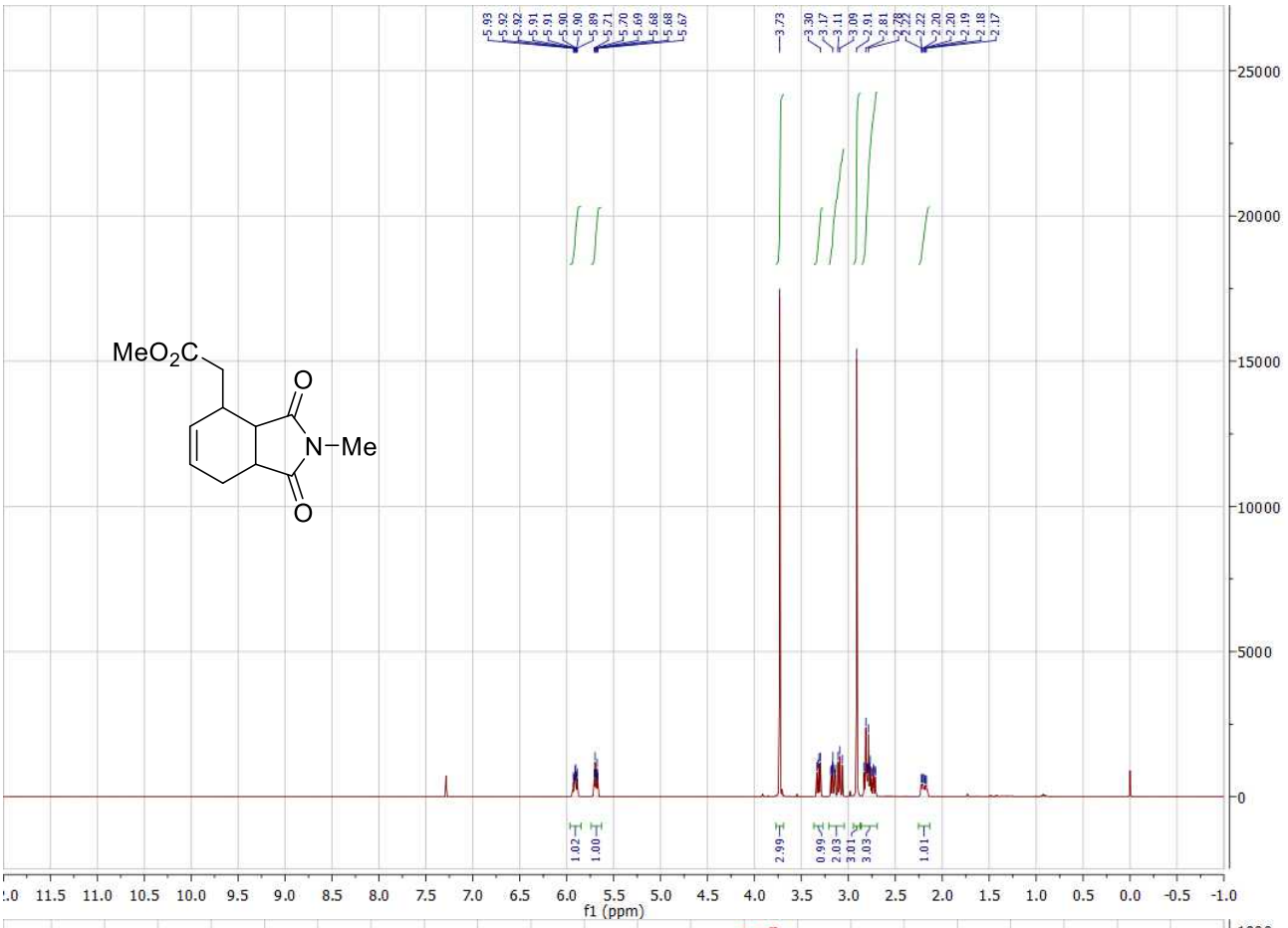

174.03
156.54
131.28
127.13
125.48
121.84
114.34
77.55 CDCl3
77.23 CDCl3
76.91 CDCl3
55.71
42.39
28.31
26.02
24.80
O
NH(4-OMe-C6H4)
f1 (ppm)

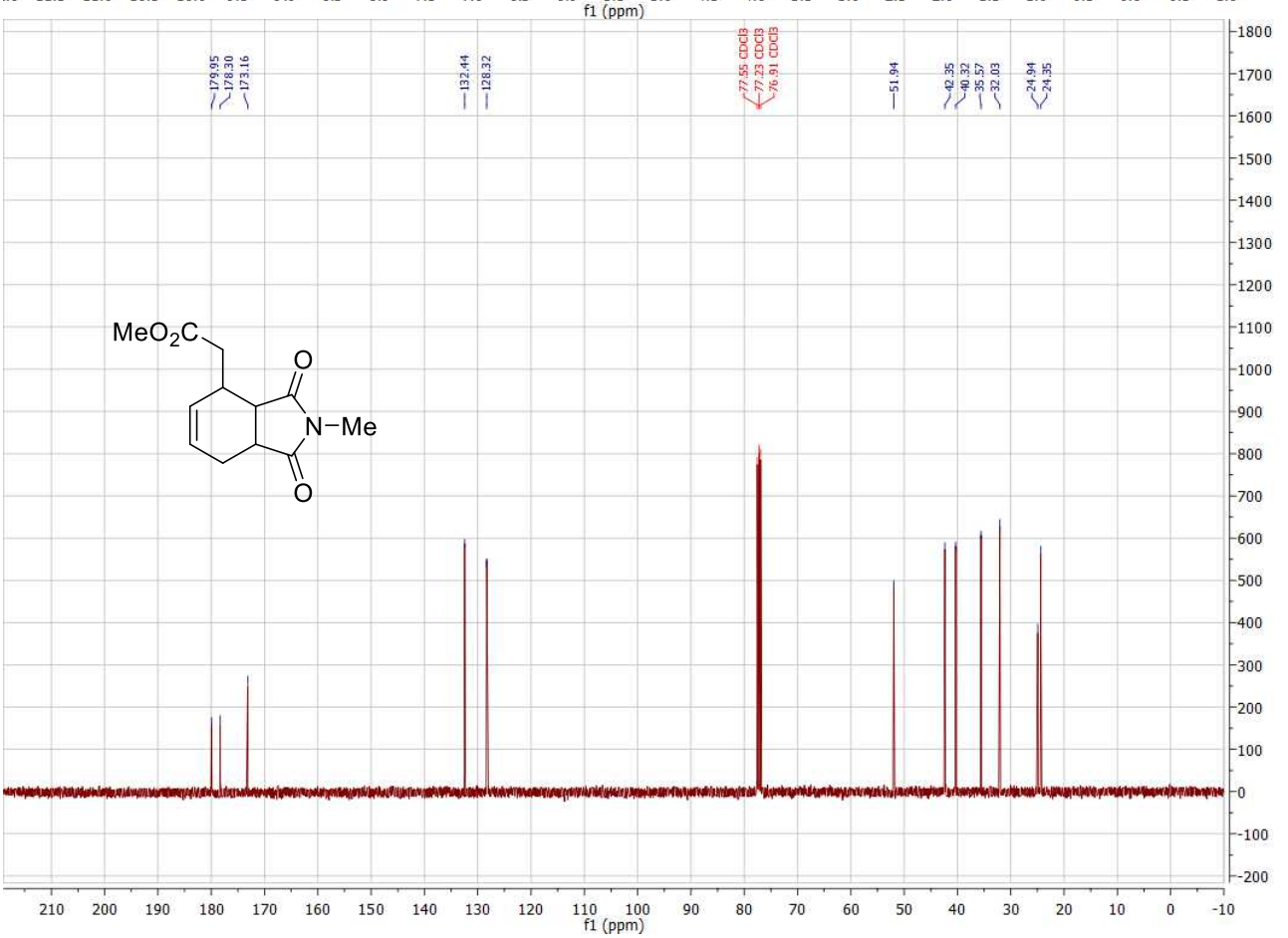

Ph
O
N–Ph
O
f1 (ppm)

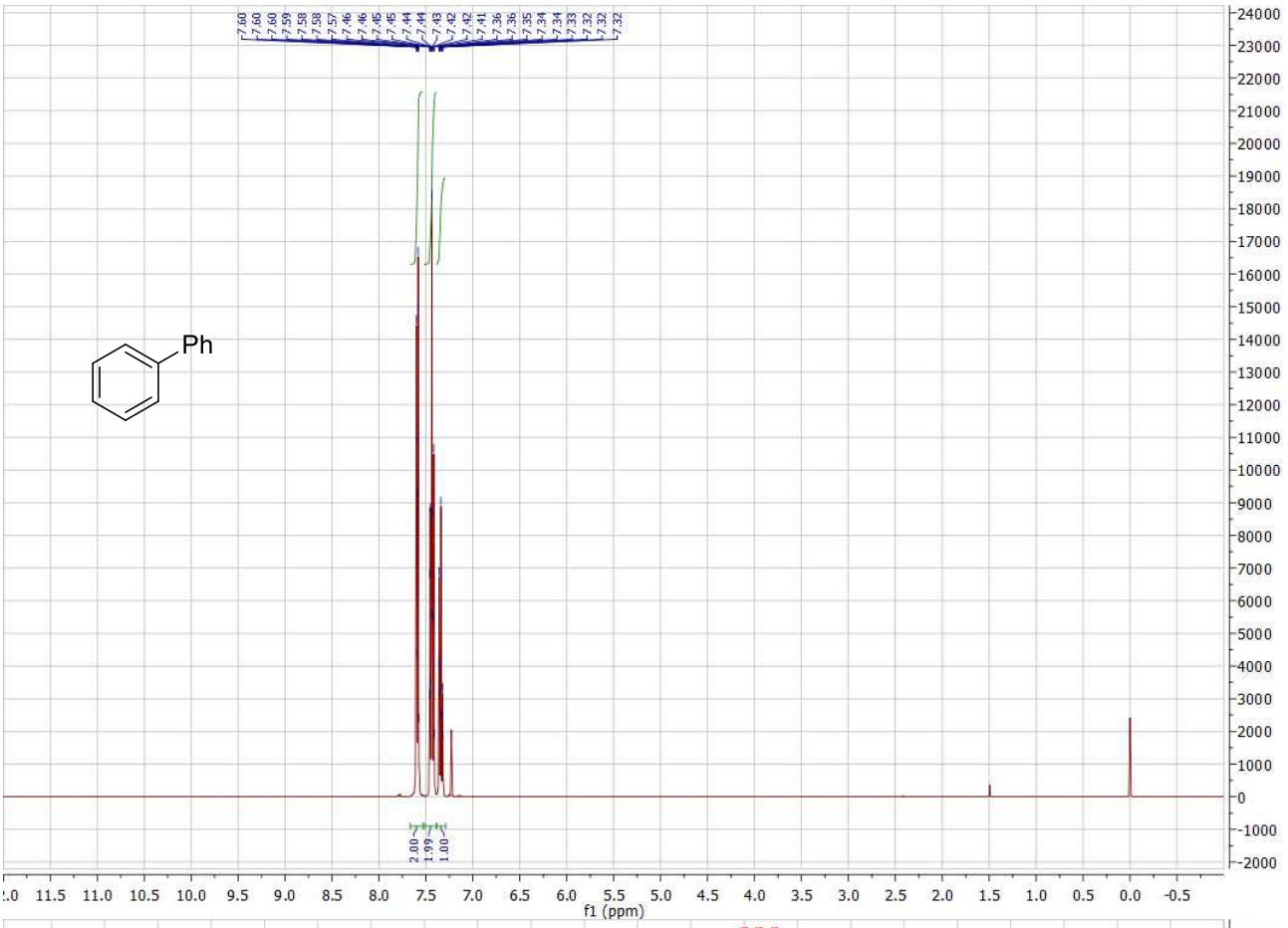

Ph
O
N–Ph
O
f1 (ppm)

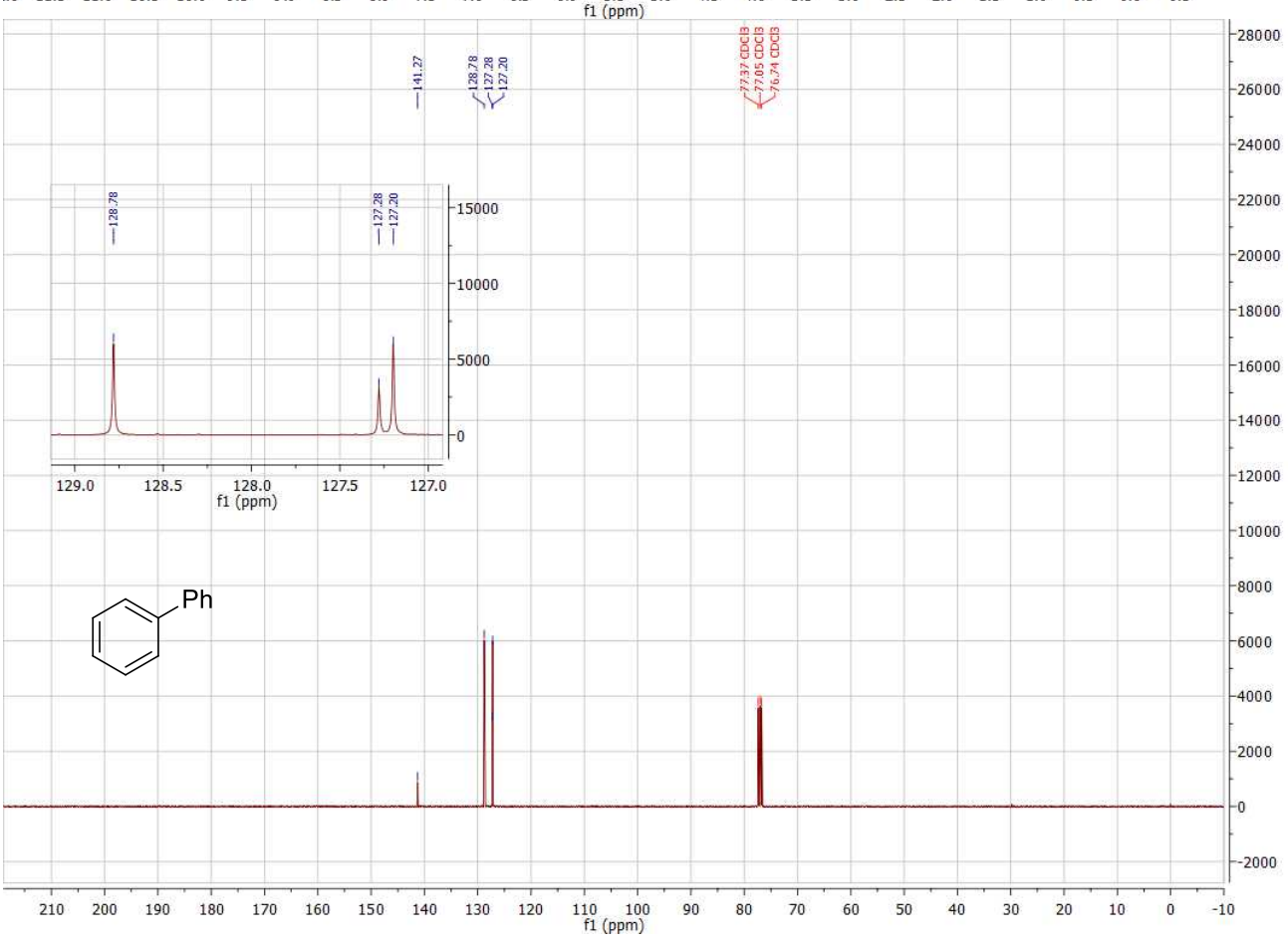

7.43
7.42
7.41
7.41
7.40
7.40
7.38
7.33
7.32
7.32
7.31
7.31
7.30
7.30
7.26
7.26
7.01
7.00
6.98
6.98
6.54
3.92
3.67
3.67
3.66
3.66
Ph
O
N–Ph
O
Ph
13.66
1.95
2.00
2.00
2.03
f1 (ppm)

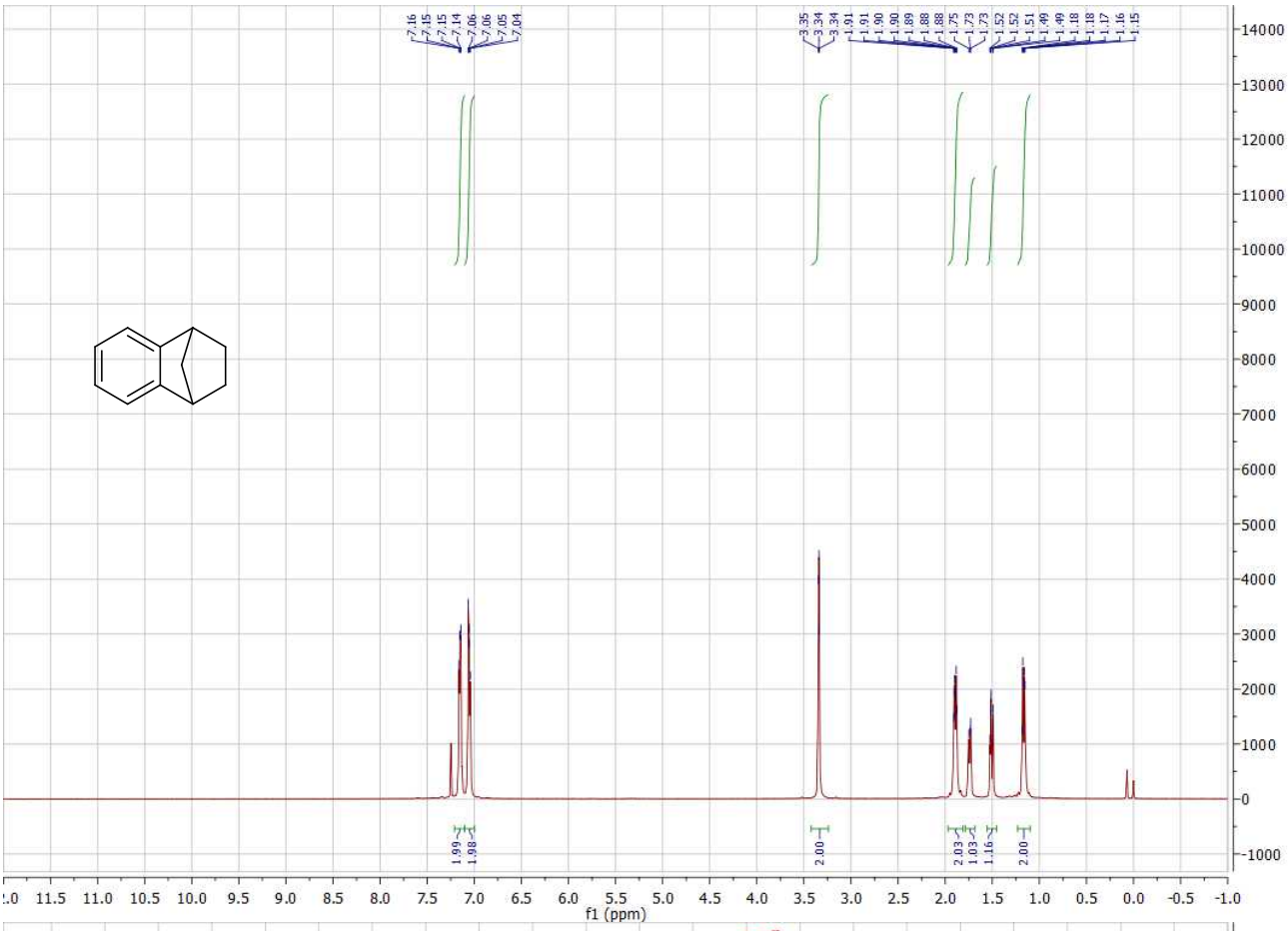

175.08
139.34
131.81
131.58
129.14
129.06
128.60
128.45
127.41
126.47
77.55 CDCl3
77.23 CDCl3
76.91 CDCl3
46.74
41.92
f1 (ppm)
Ph
O
N–Ph
O
Ph
f1 (ppm)


MeO2C
O
N−Me
O
f1 (ppm)


MeO2C
O
N−Me
O
f1 (ppm)


Ph
f1 (ppm)


141.27
128.78
127.28
127.20
77.37 CDCl3
77.05 CDCl3
76.74 CDCl3
Ph
f1 (ppm)


7.16
7.15
7.15
7.14
7.06
7.06
7.05
7.04
3.35
3.34
3.34
1.91
1.91
1.90
1.90
1.89
1.88
1.88
1.75
1.73
1.73
1.52
1.52
1.51
1.49
1.49
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1.17
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1.99
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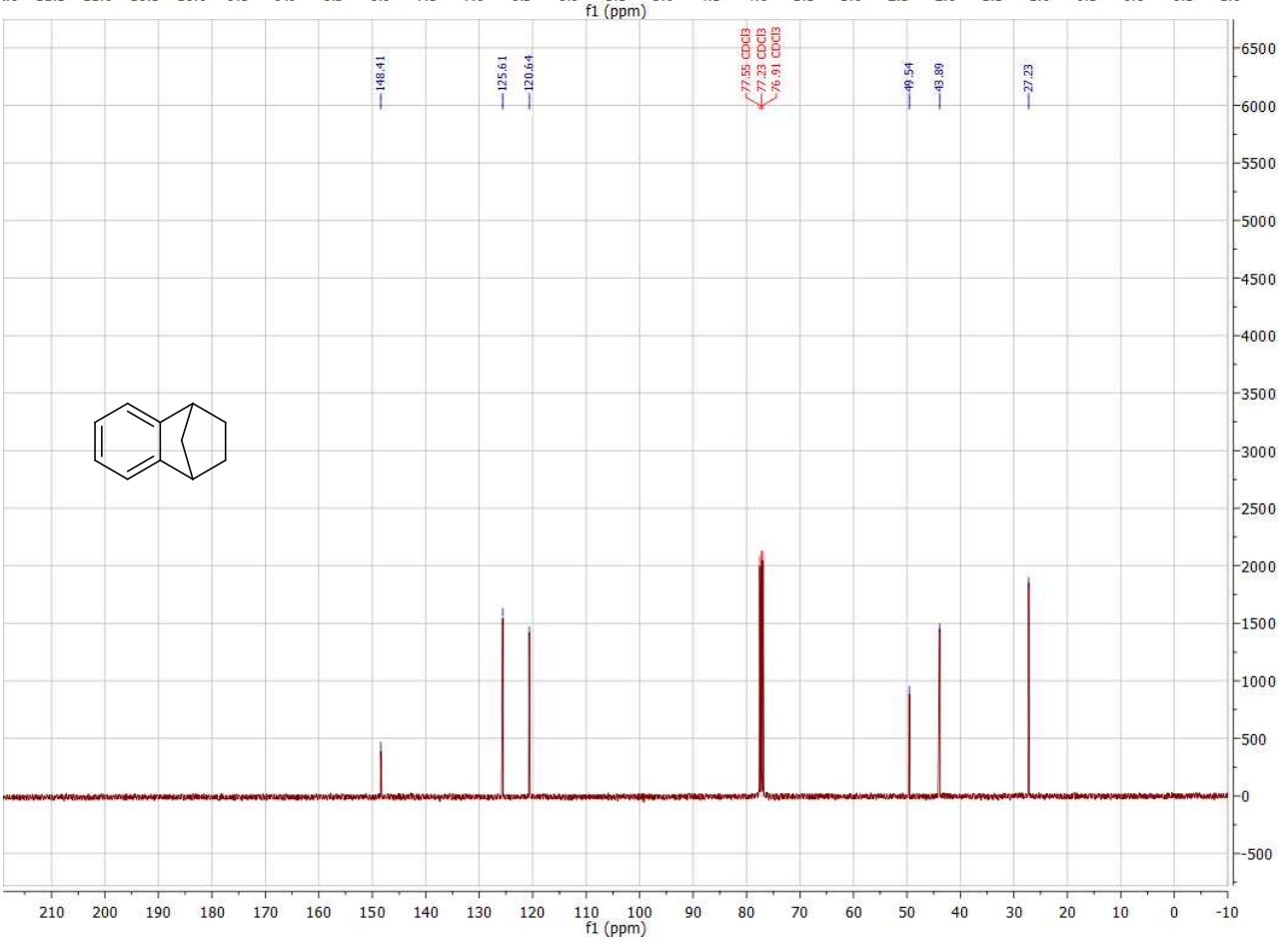
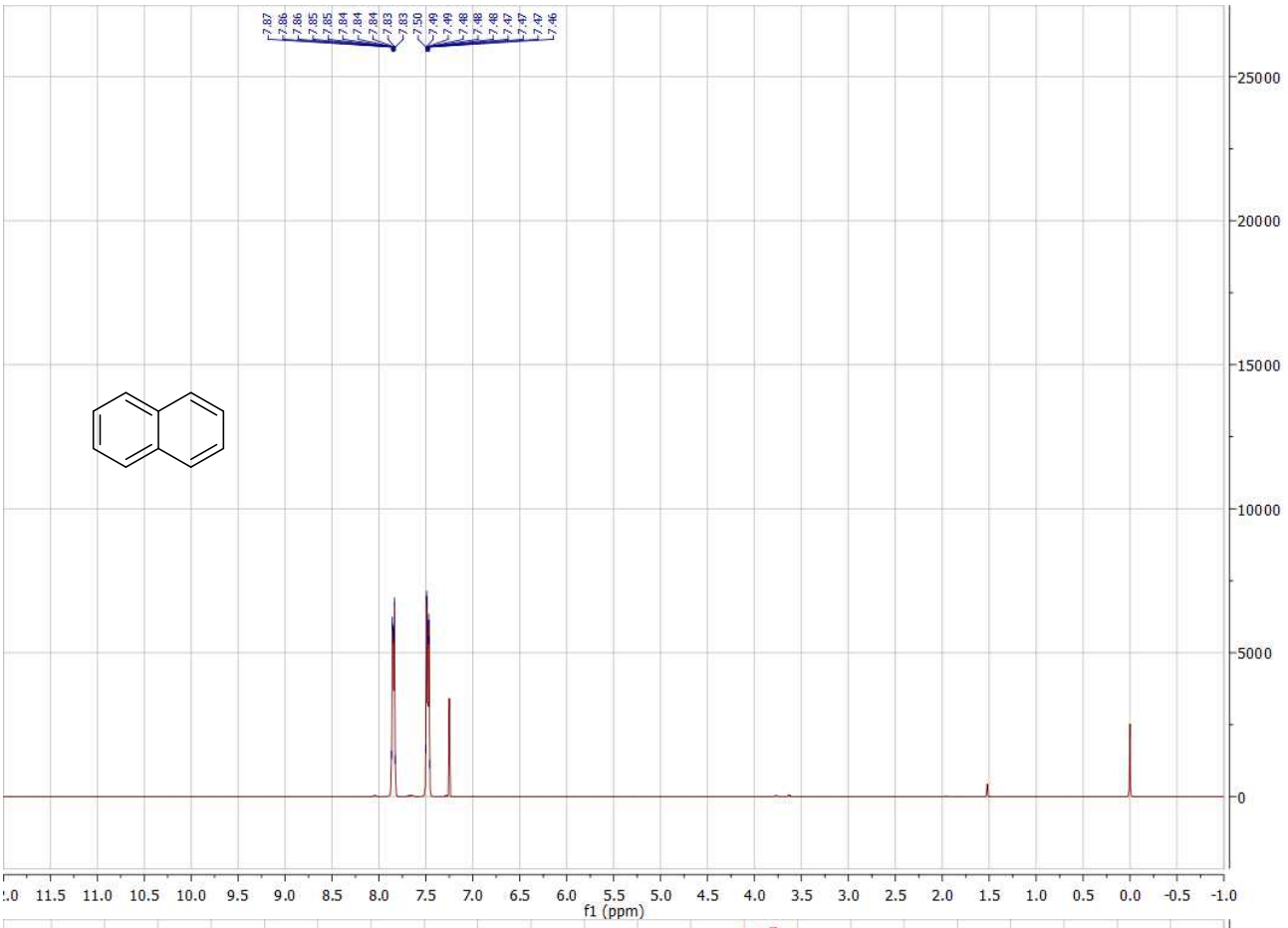
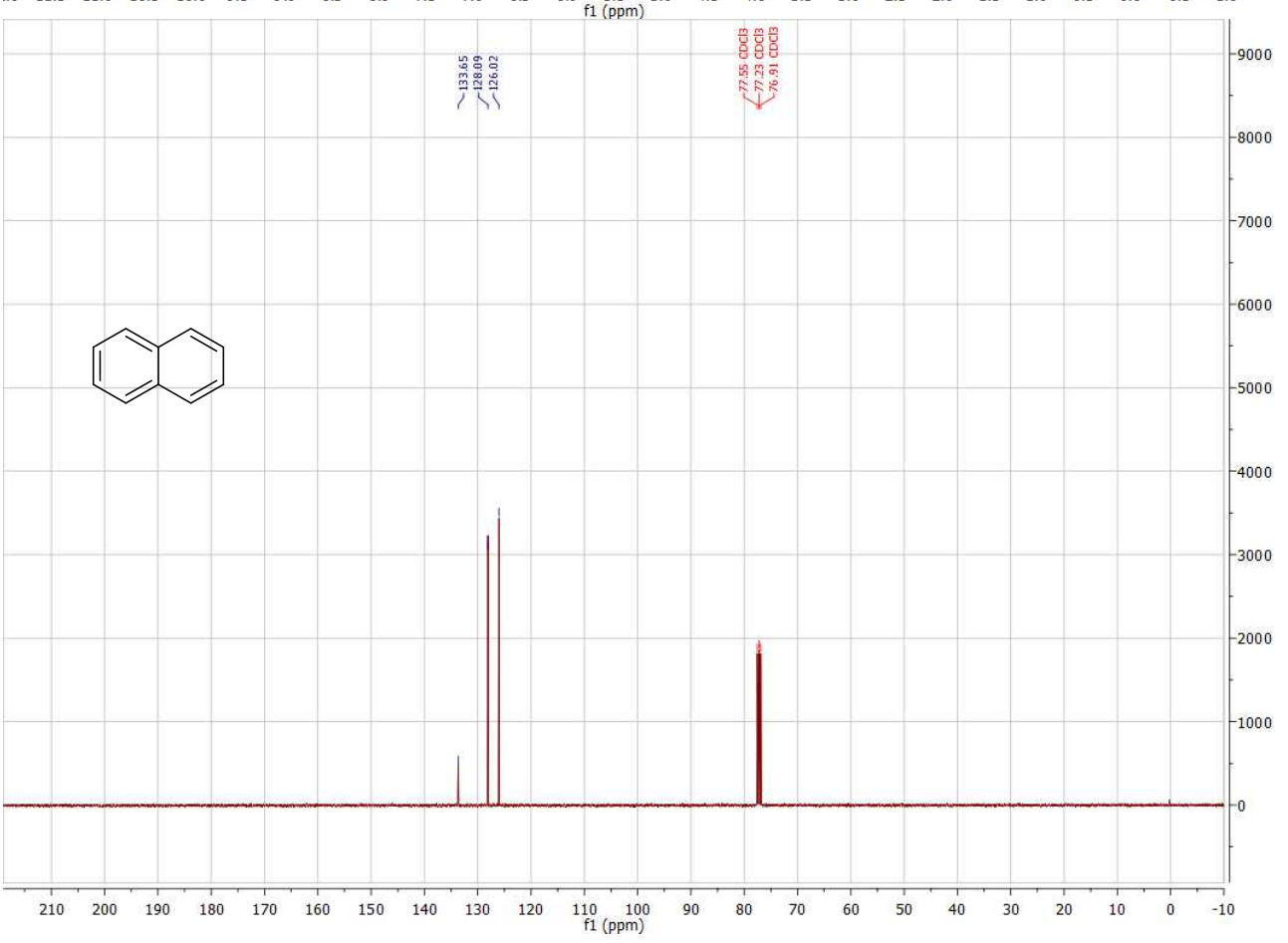
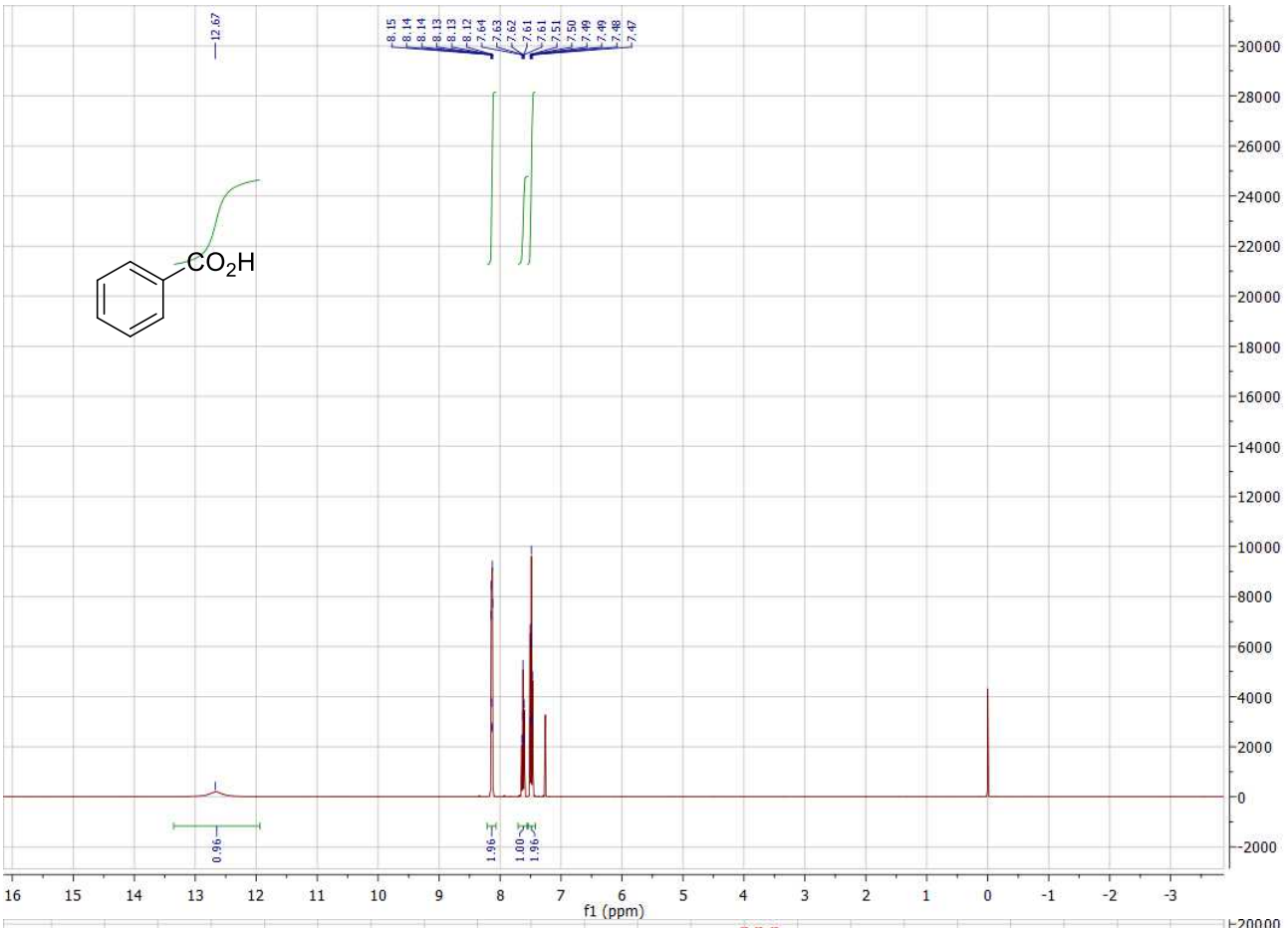
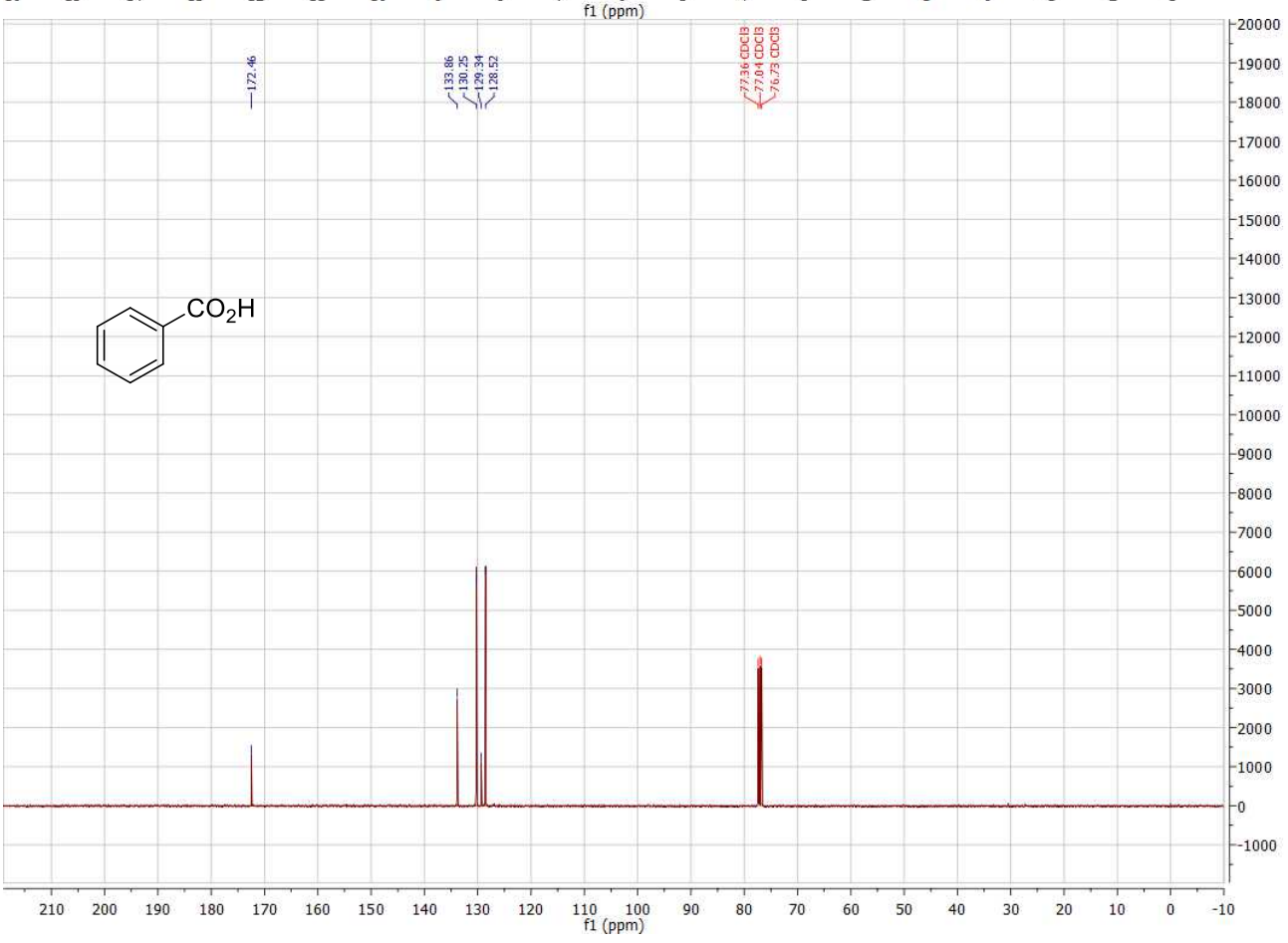
2.00
2.03
1.03
1.16
2.00
f1 (ppm)

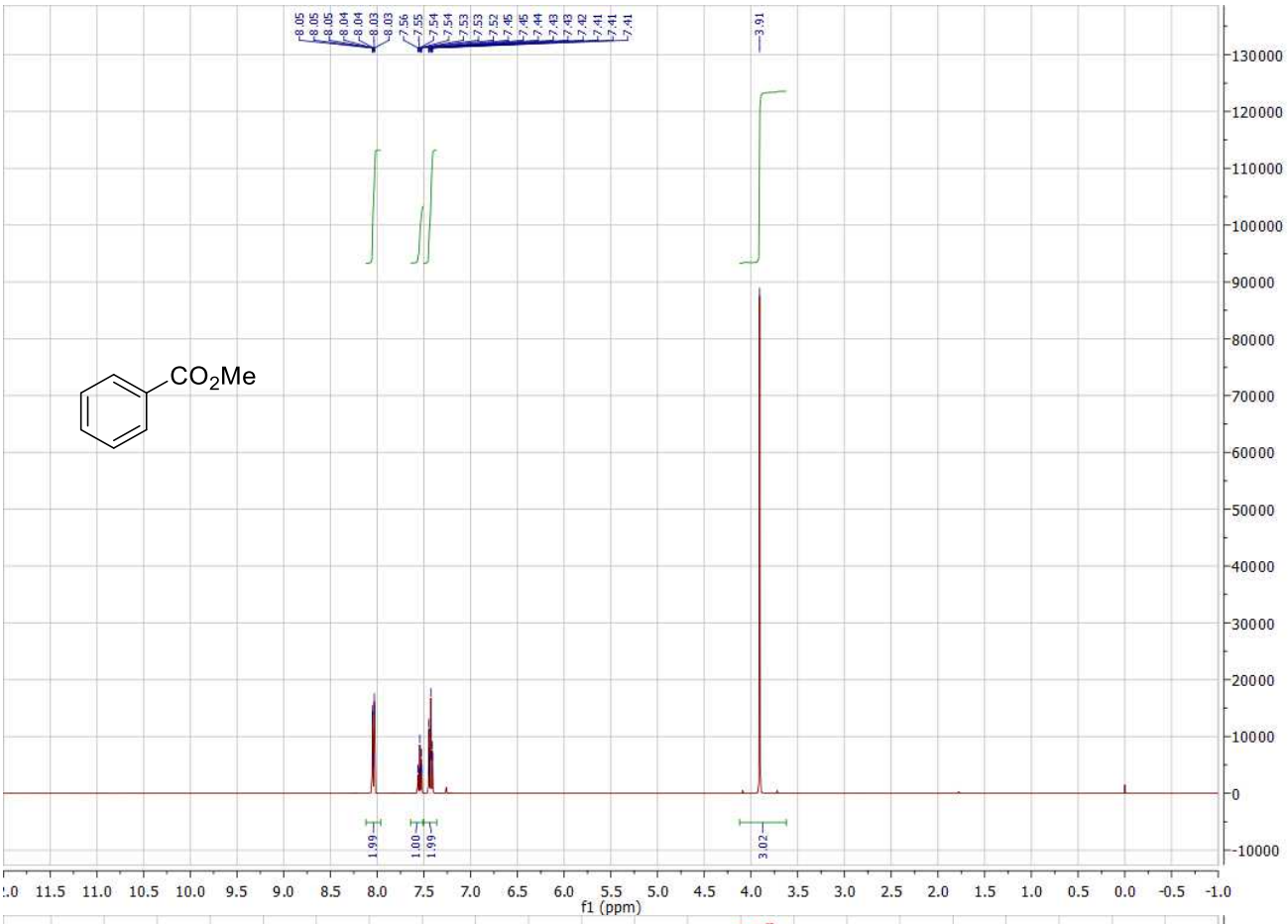

148.41
125.61
120.64
77.55 CDCl3
77.23 CDCl3
76.91 CDCl3
49.54
43.89
27.23
f1 (ppm)

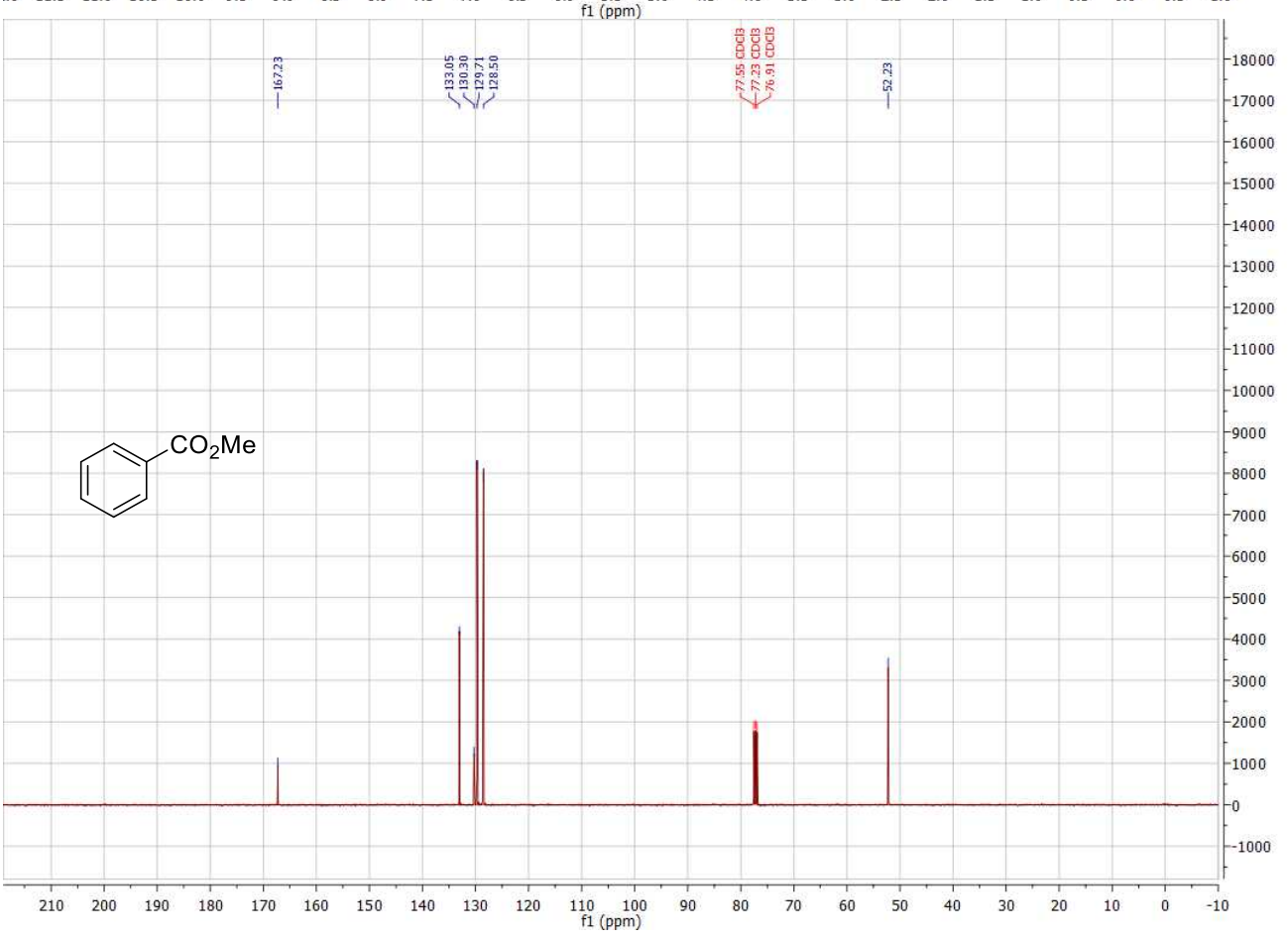

7.87
7.86
7.86
7.85
7.85
7.84
7.84
7.84
7.84
7.83
7.83
7.50
7.49
7.49
7.48
7.48
7.48
7.47
7.47
7.47
7.46
25000
20000
15000
10000
5000
0
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

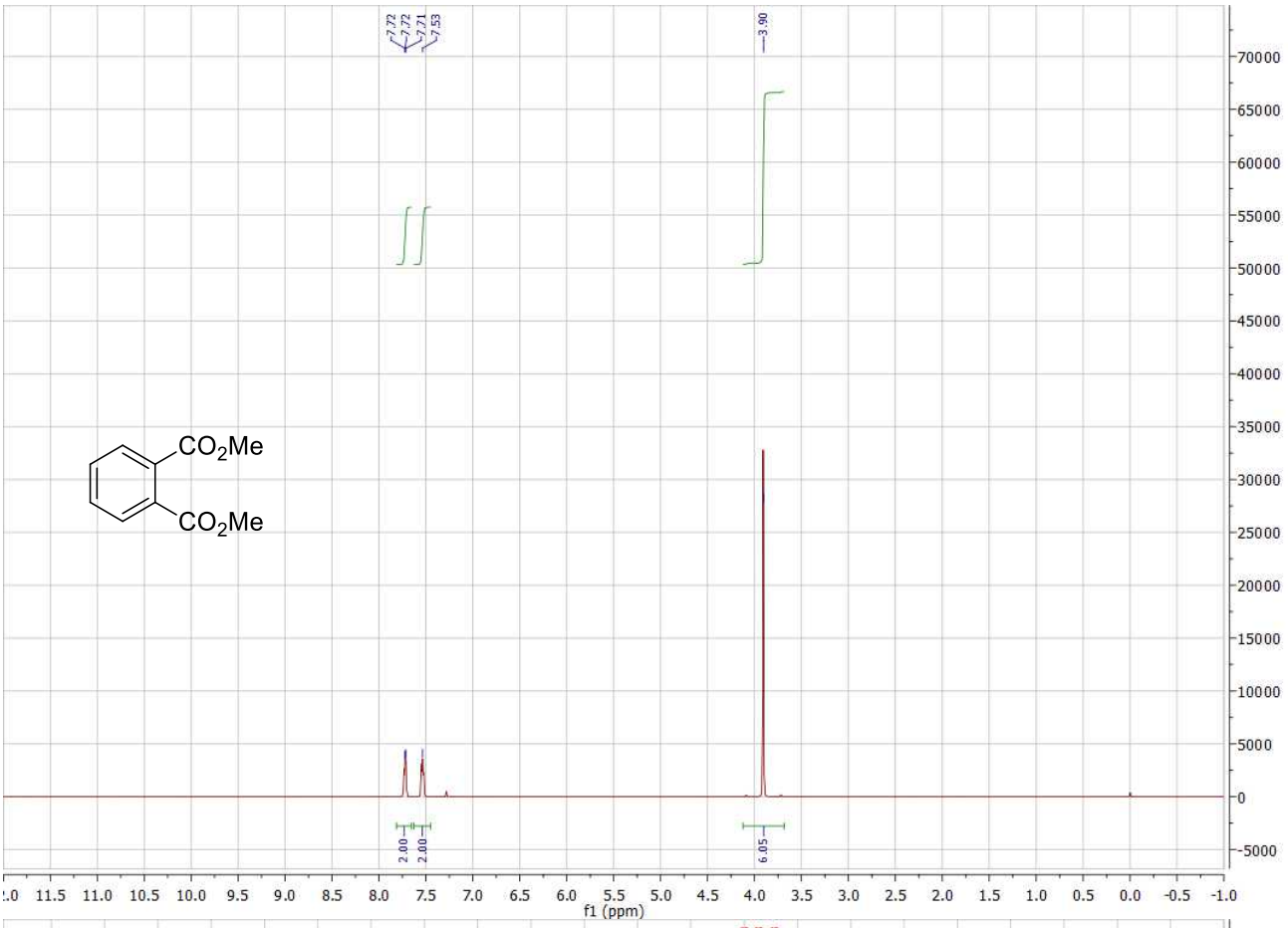

133.65
128.09
126.02
77.55 CDCl3
77.23 CDCl3
76.91 CDCl3
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

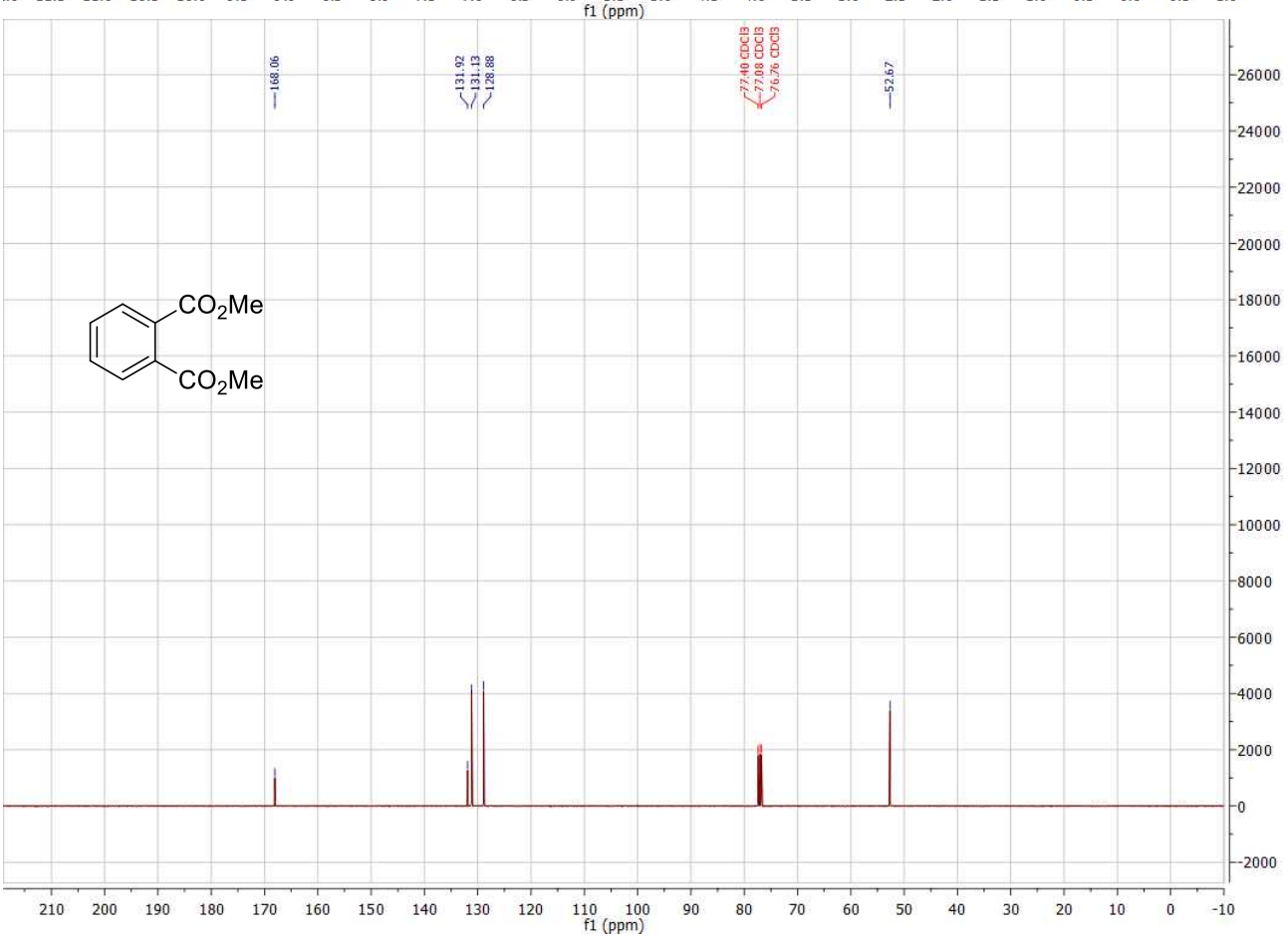

12.67
8.15
8.14
8.14
8.13
8.13
8.12
7.64
7.63
7.62
7.61
7.61
7.51
7.50
7.49
7.49
7.48
7.47
CO2H
0.96
1.96
1.00
1.96
f1 (ppm)

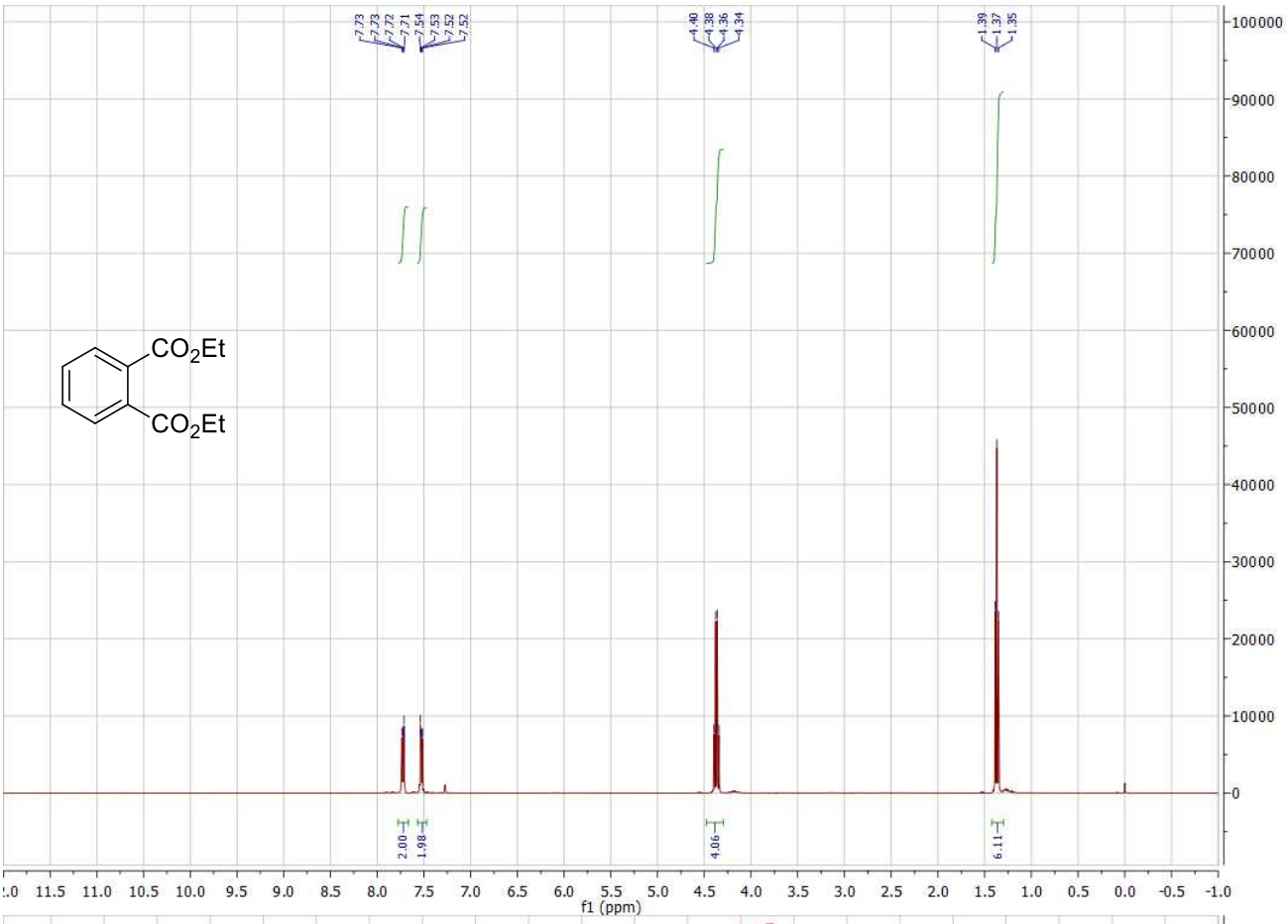

172.46
133.86
130.25
129.34
128.52
77.36 CDCl3
77.04 CDCl3
76.73 CDCl3
CO2H
f1 (ppm)

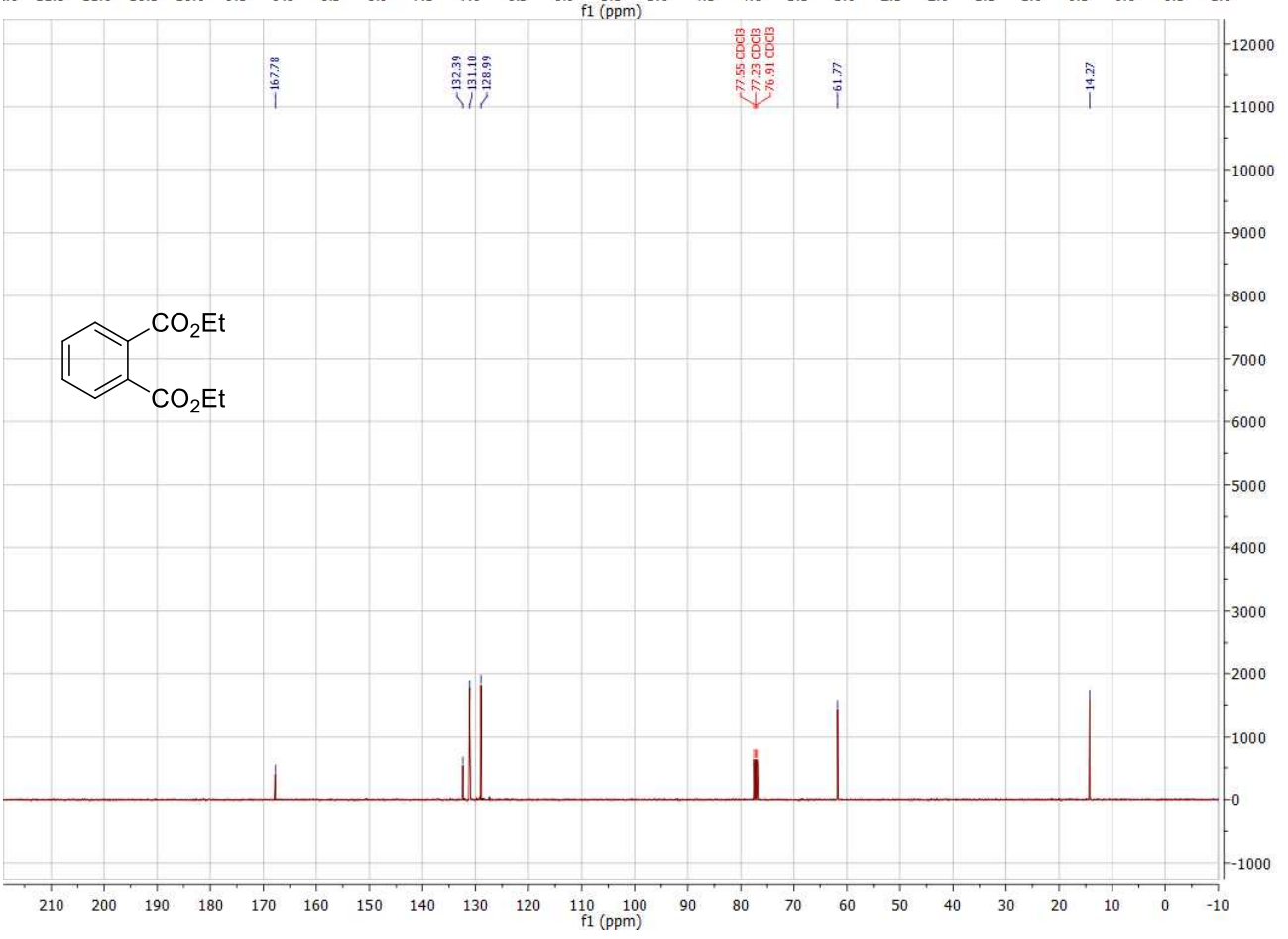

8.05
8.05
8.05
8.04
8.04
8.03
8.03
7.56
7.55
7.54
7.54
7.53
7.53
7.52
7.45
7.44
7.43
7.43
7.42
7.41
7.41
7.41
3.91
CO2Me
1.99
1.00
1.99
3.02
f1 (ppm)

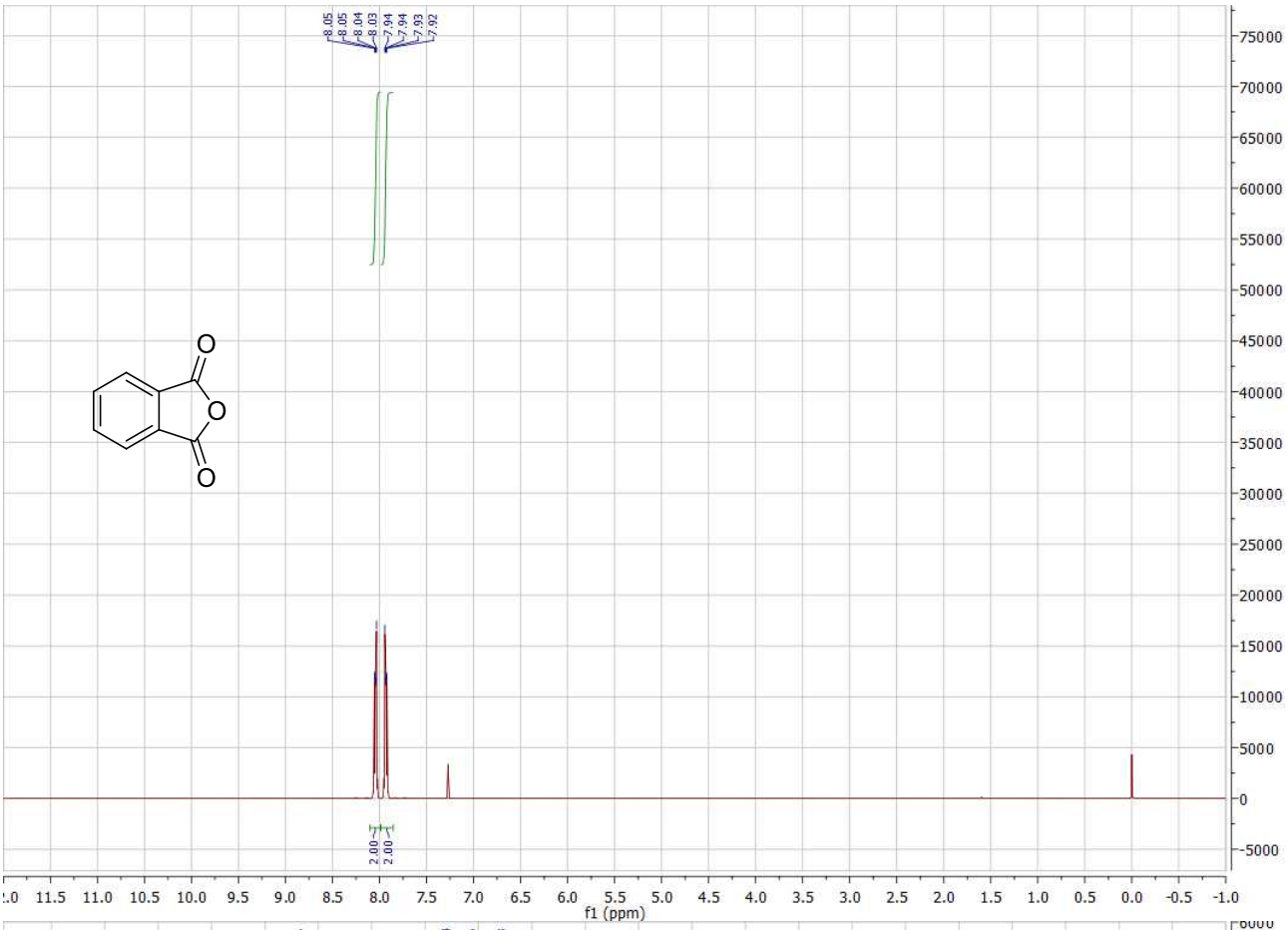

167.23
133.05
130.30
129.71
128.50
77.55 CDCl3
77.23 CDCl3
76.91 CDCl3
52.23
CO2Me
f1 (ppm)

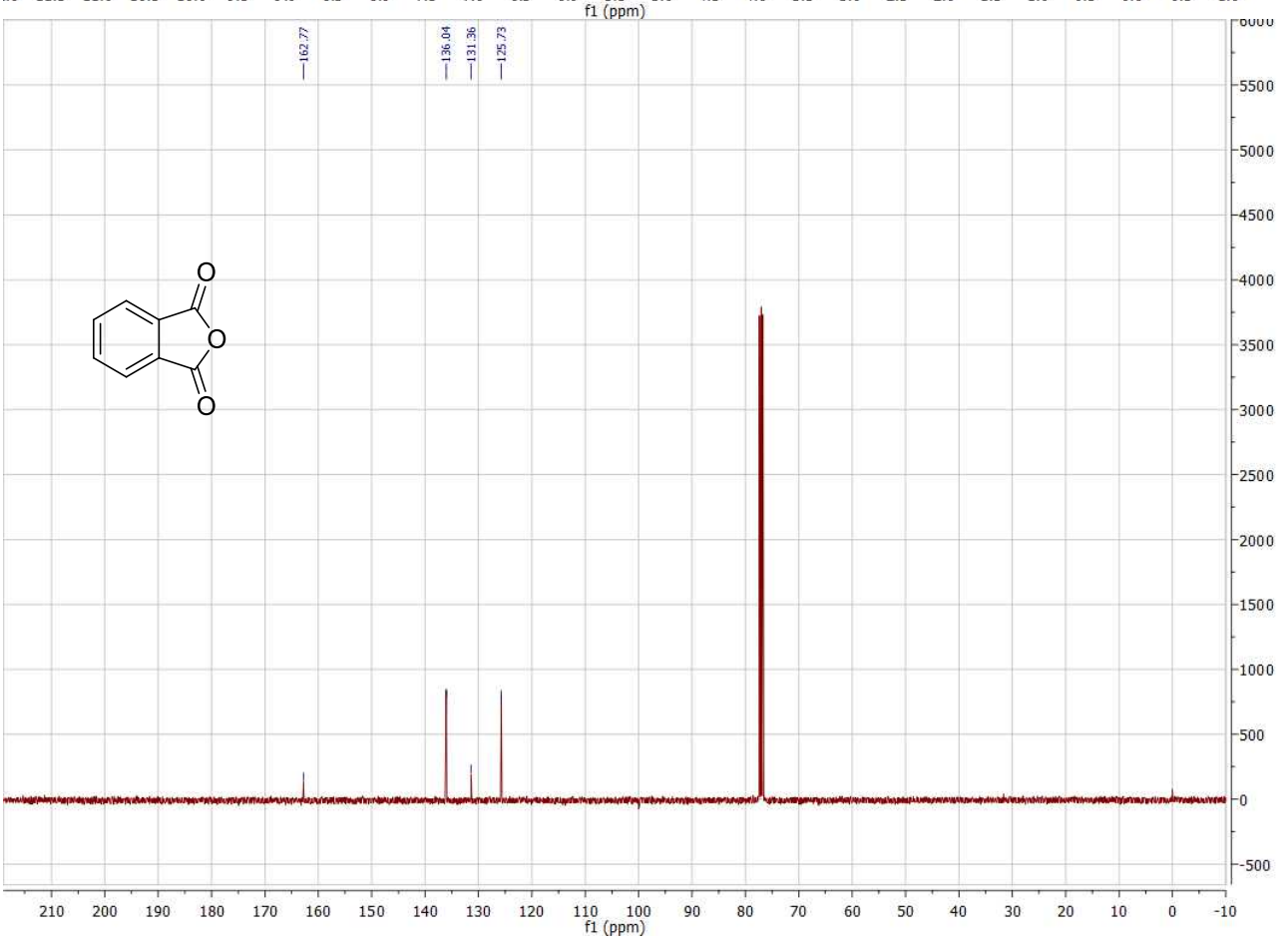

7.72
7.72
7.71
7.53
3.90
CO2Me
CO2Me
2.00
2.00
6.05
f1 (ppm)

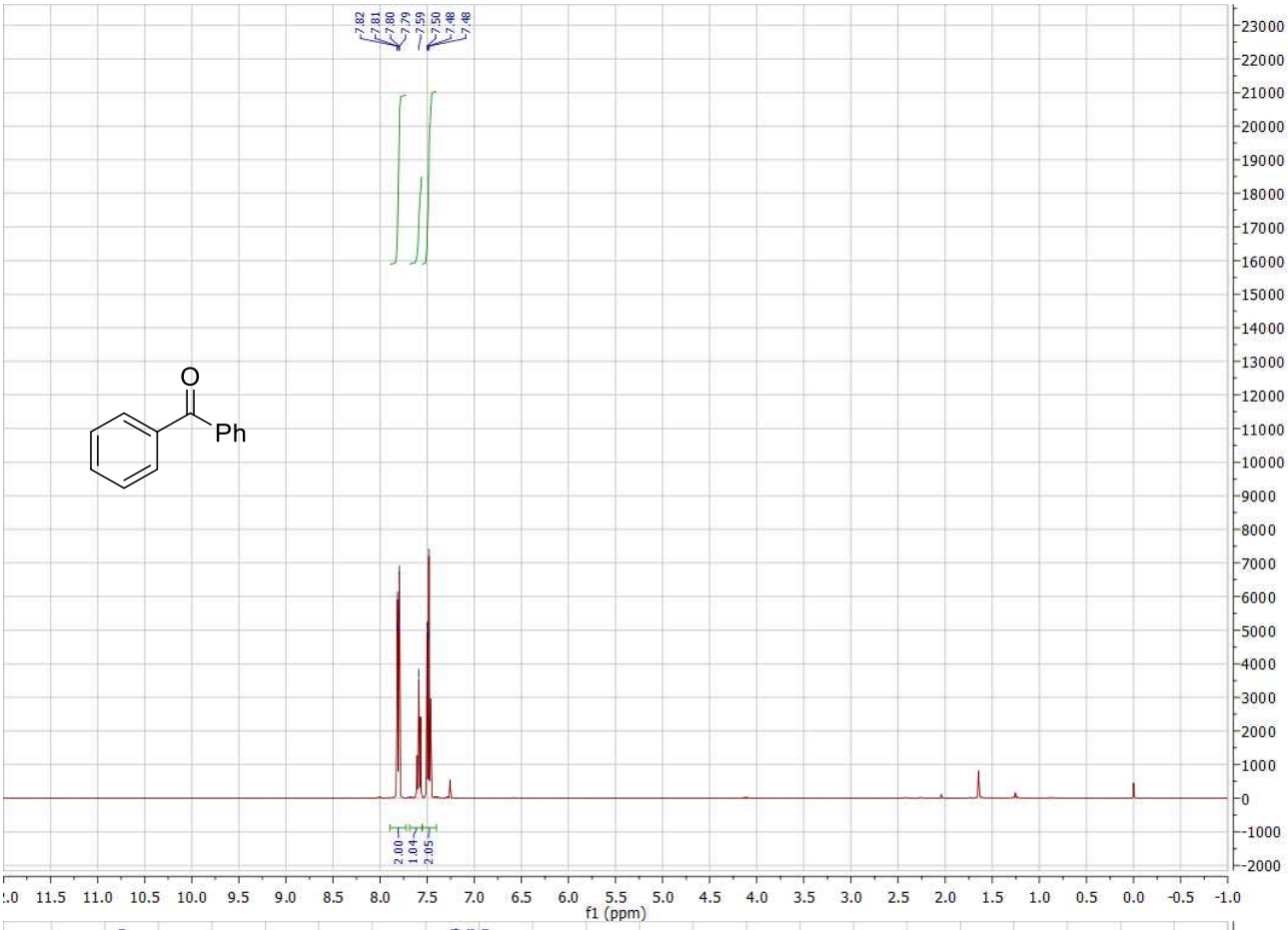

168.06
131.92
131.13
128.88
77.40 CDCl3
77.08 CDCl3
76.76 CDCl3
52.67
CO2Me
CO2Me
f1 (ppm)

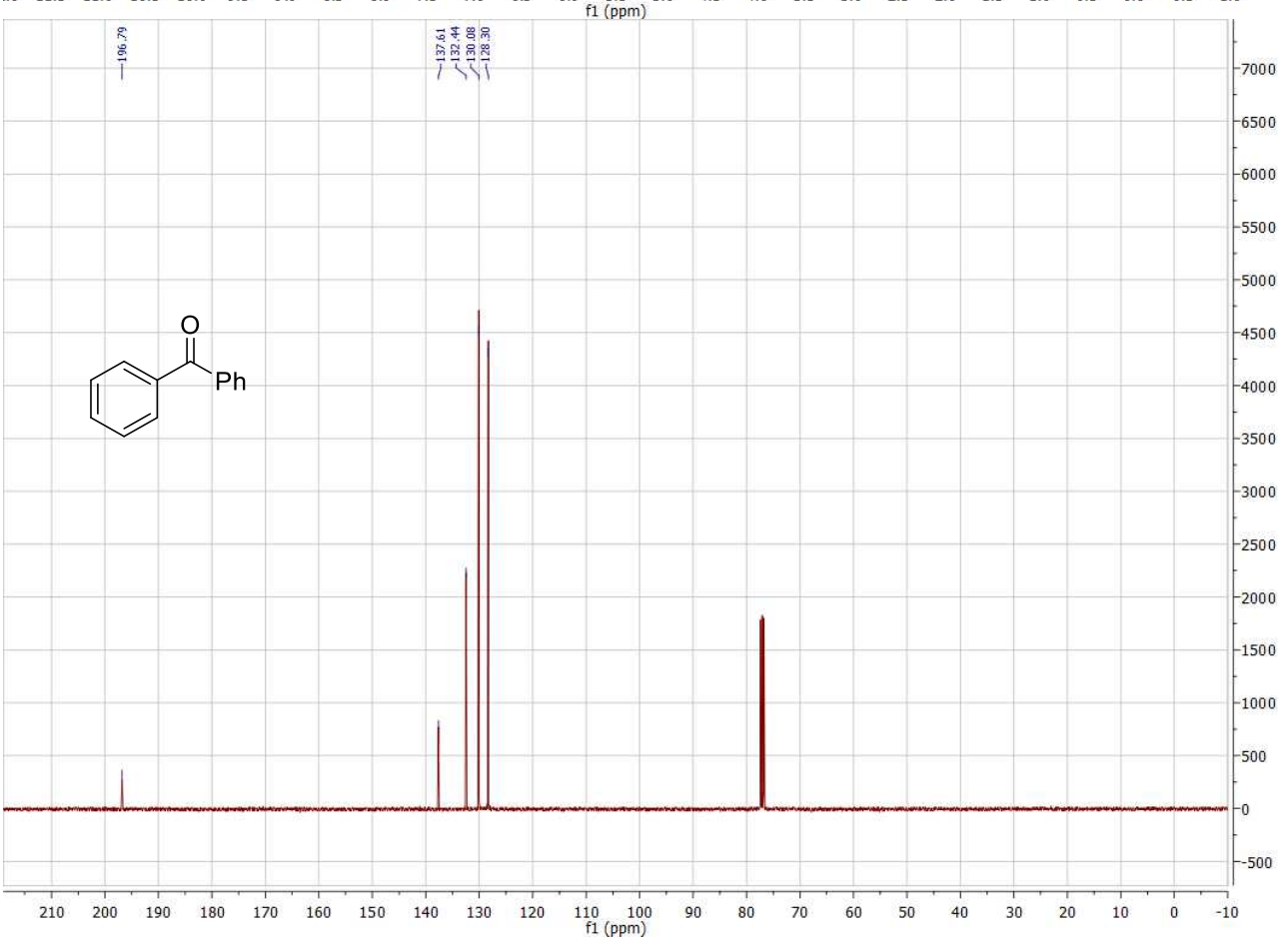

7.73
7.73
7.72
7.71
7.54
7.53
7.52
7.52
4.40
4.38
4.36
4.34
1.39
1.37
1.35
CO2Et
CO2Et
2.00
1.98
4.06
6.11
f1 (ppm)

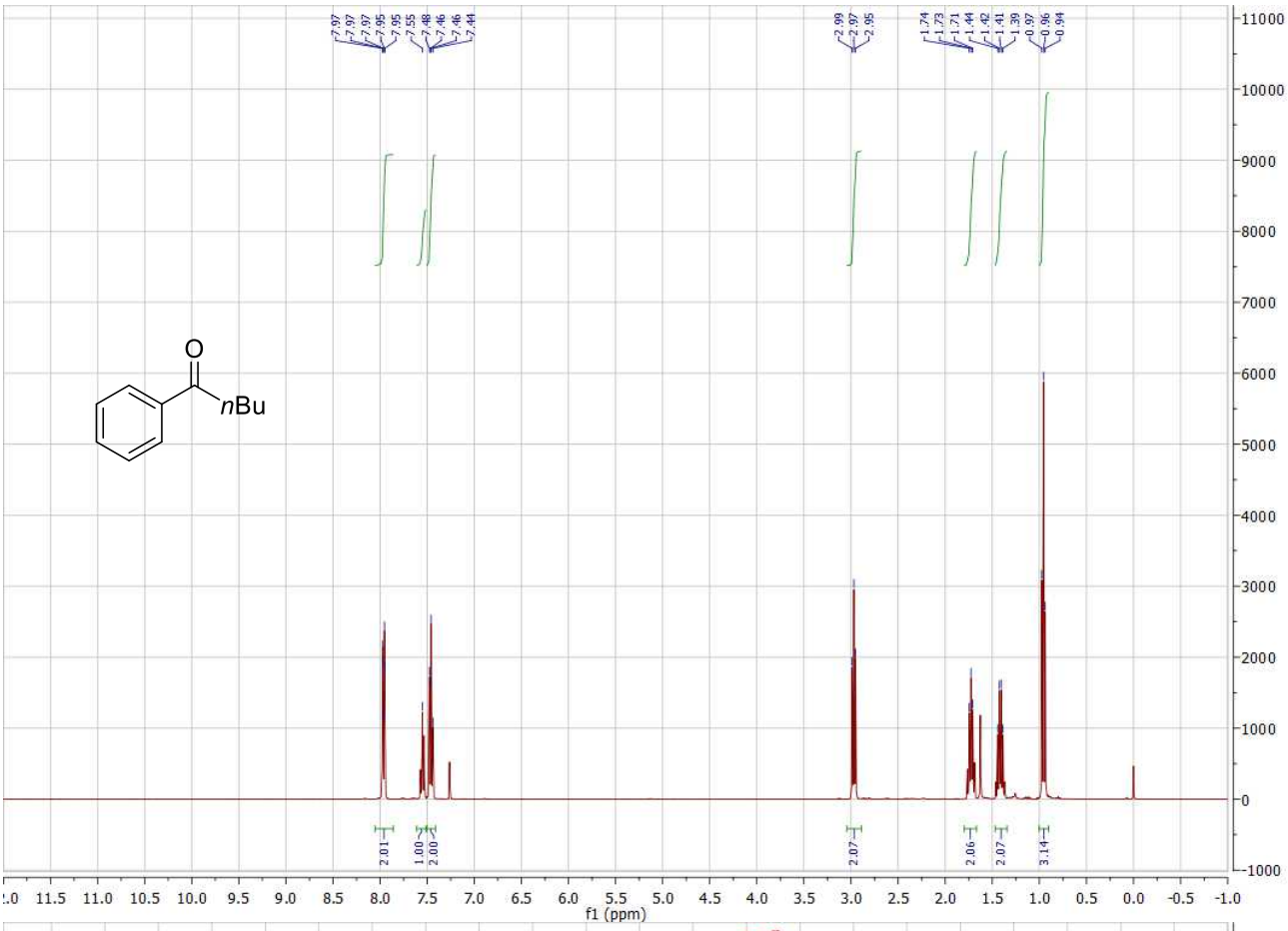

167.78
132.39
131.10
128.99
77.55 CDCl3
77.23 CDCl3
76.91 CDCl3
61.77
14.27
CO2Et
CO2Et
f1 (ppm)

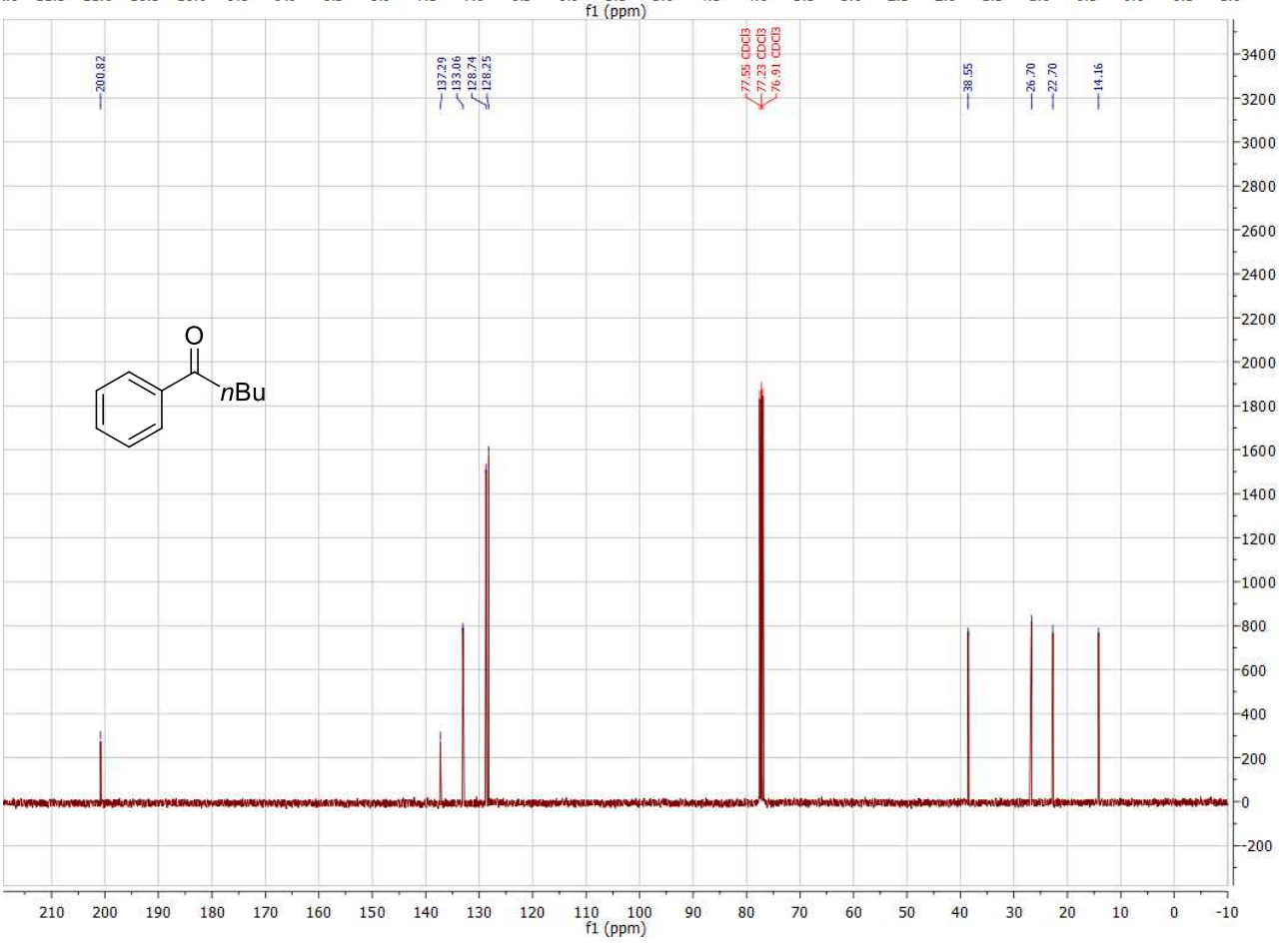

8.05
8.05
8.04
8.03
7.94
7.94
7.93
7.92
2.00
2.00
f1 (ppm)

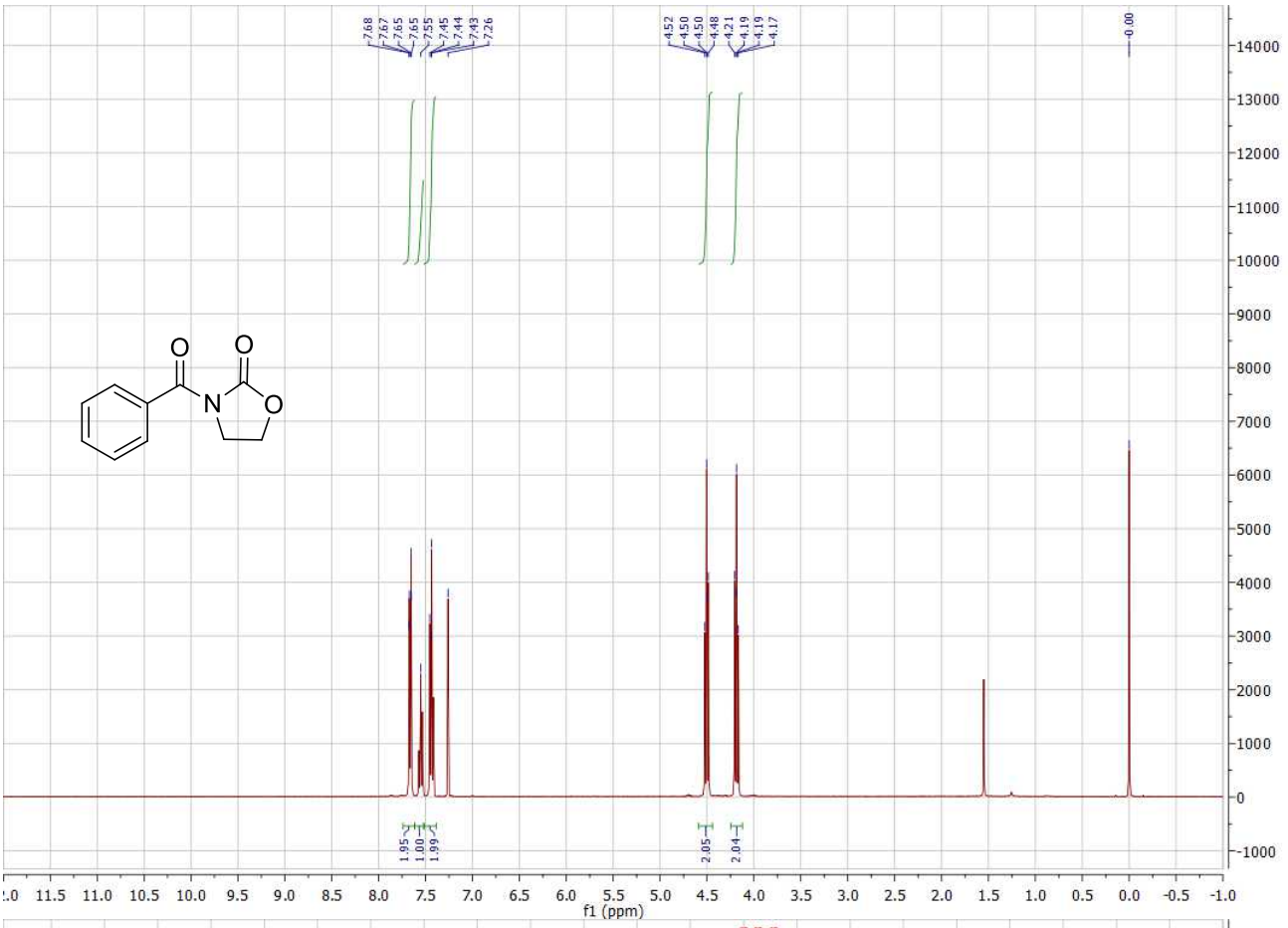

162.77
136.04
131.36
125.73
f1 (ppm)

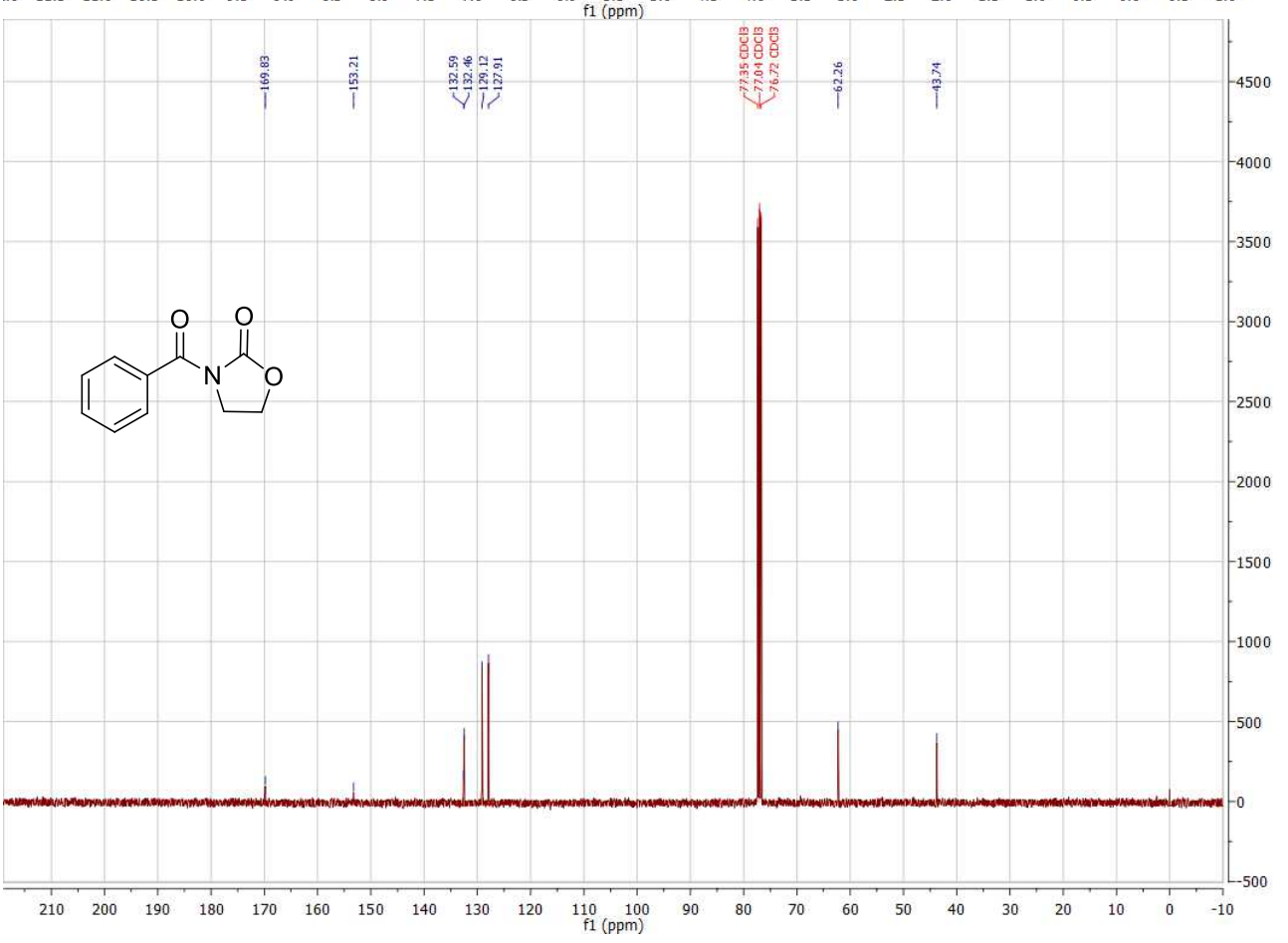

7.82
7.81
7.80
7.79
7.59
7.50
7.48
7.46
O
Ph
2.00
1.04
2.05
f1 (ppm)

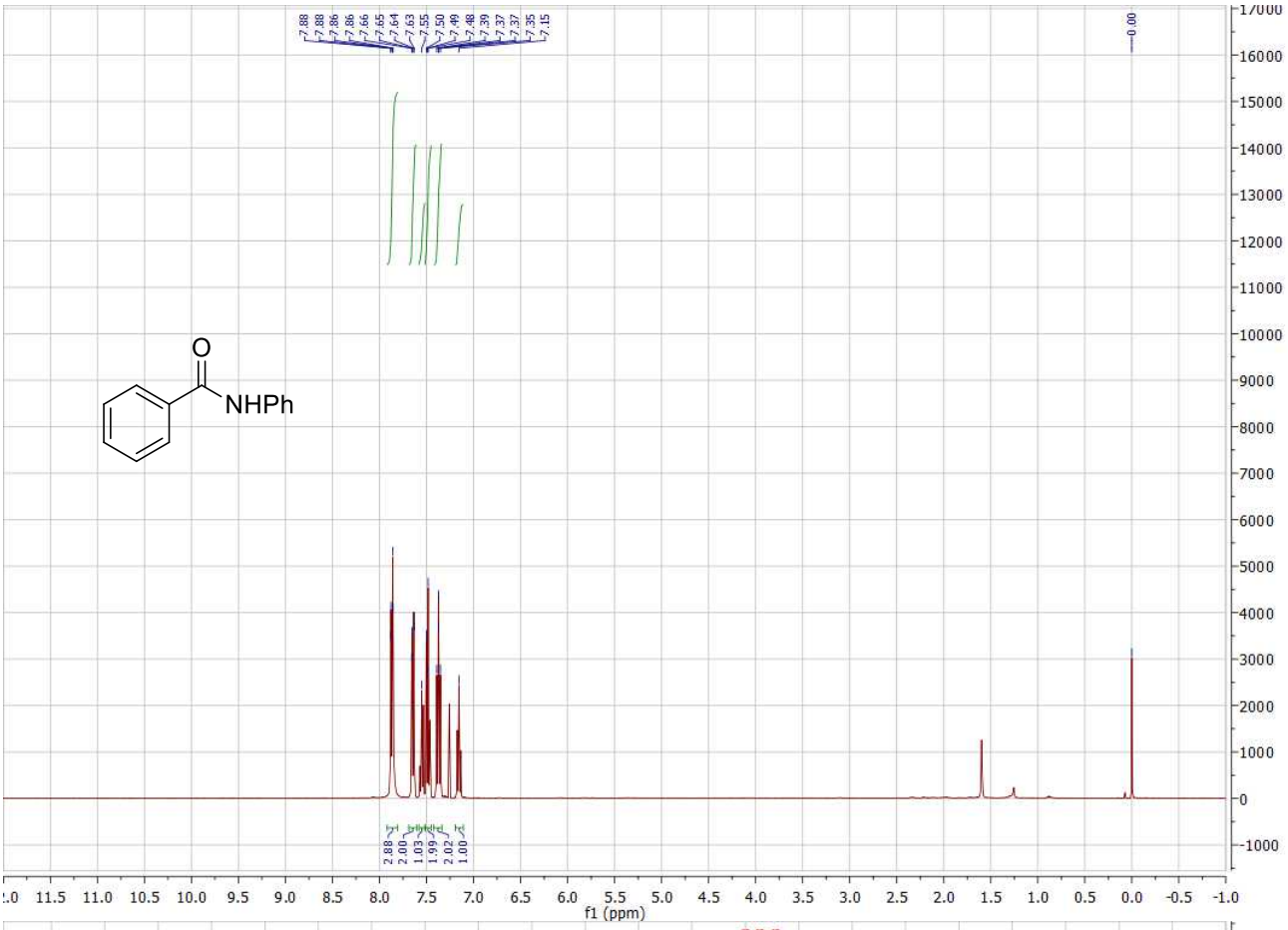

196.79
137.61
132.44
130.08
128.30
O
Ph
f1 (ppm)

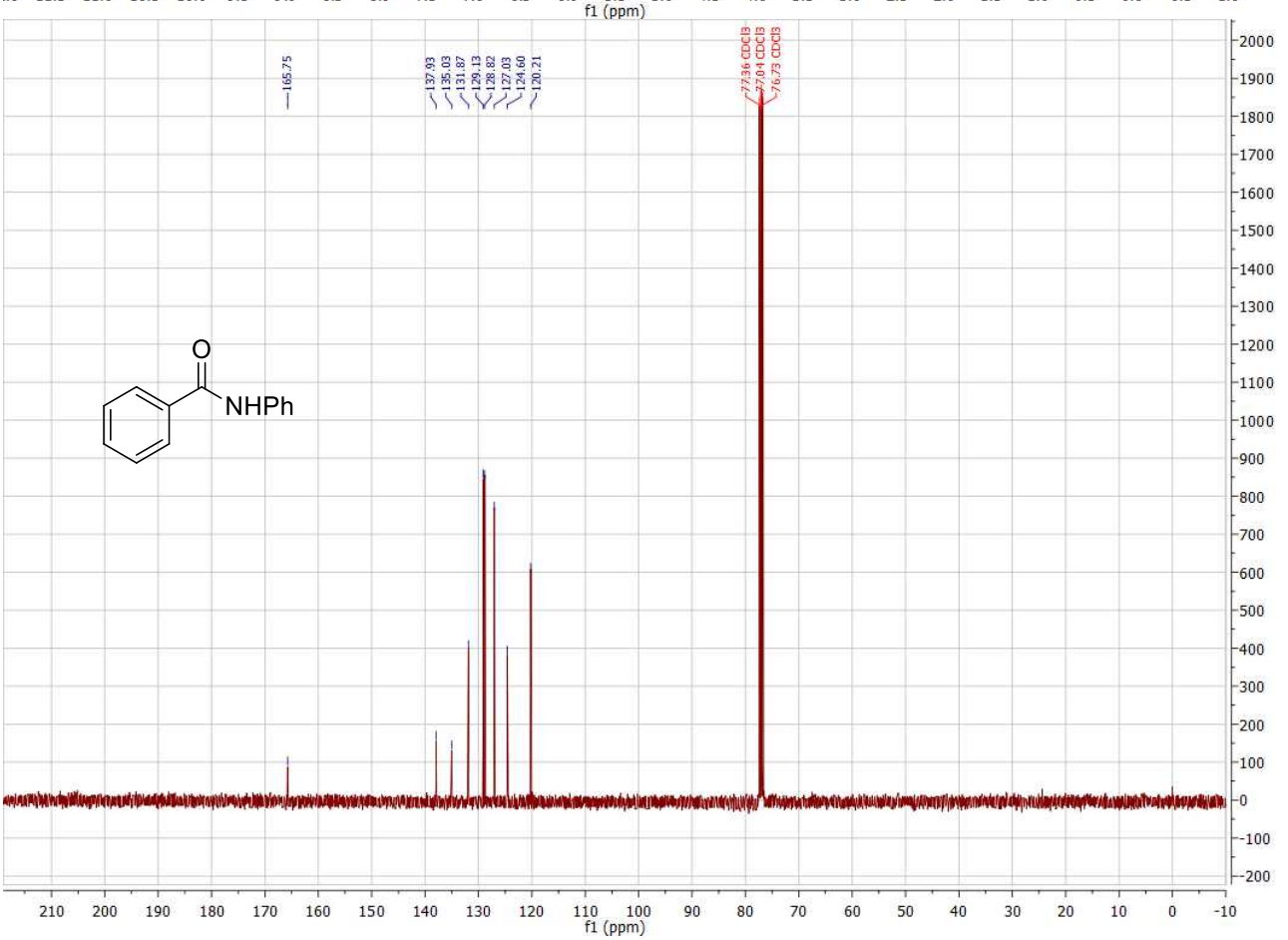

7.97
7.97
7.97
7.95
7.95
7.55
7.48
7.46
7.46
7.44
2.99
2.97
2.95
1.74
1.73
1.71
1.44
1.42
1.41
1.39
0.97
0.96
0.94
O
nBu
2.01
1.00
2.00
2.07
2.06
2.07
3.14
f1 (ppm)

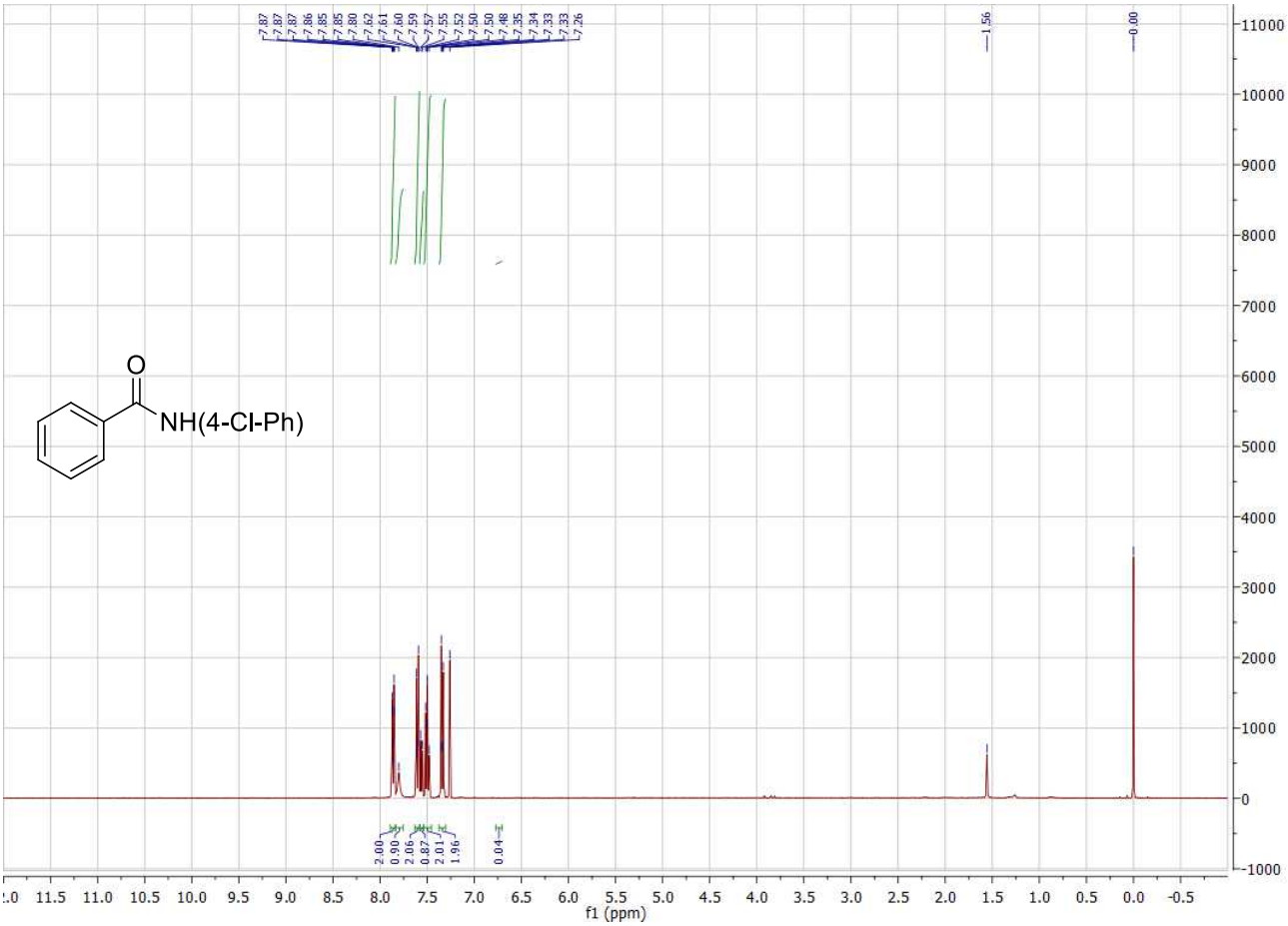

200.82
137.29
133.06
128.74
128.25
77.55 CDCl3
77.23 CDCl3
76.91 CDCl3
38.55
26.70
22.70
14.16
O
nBu
f1 (ppm)

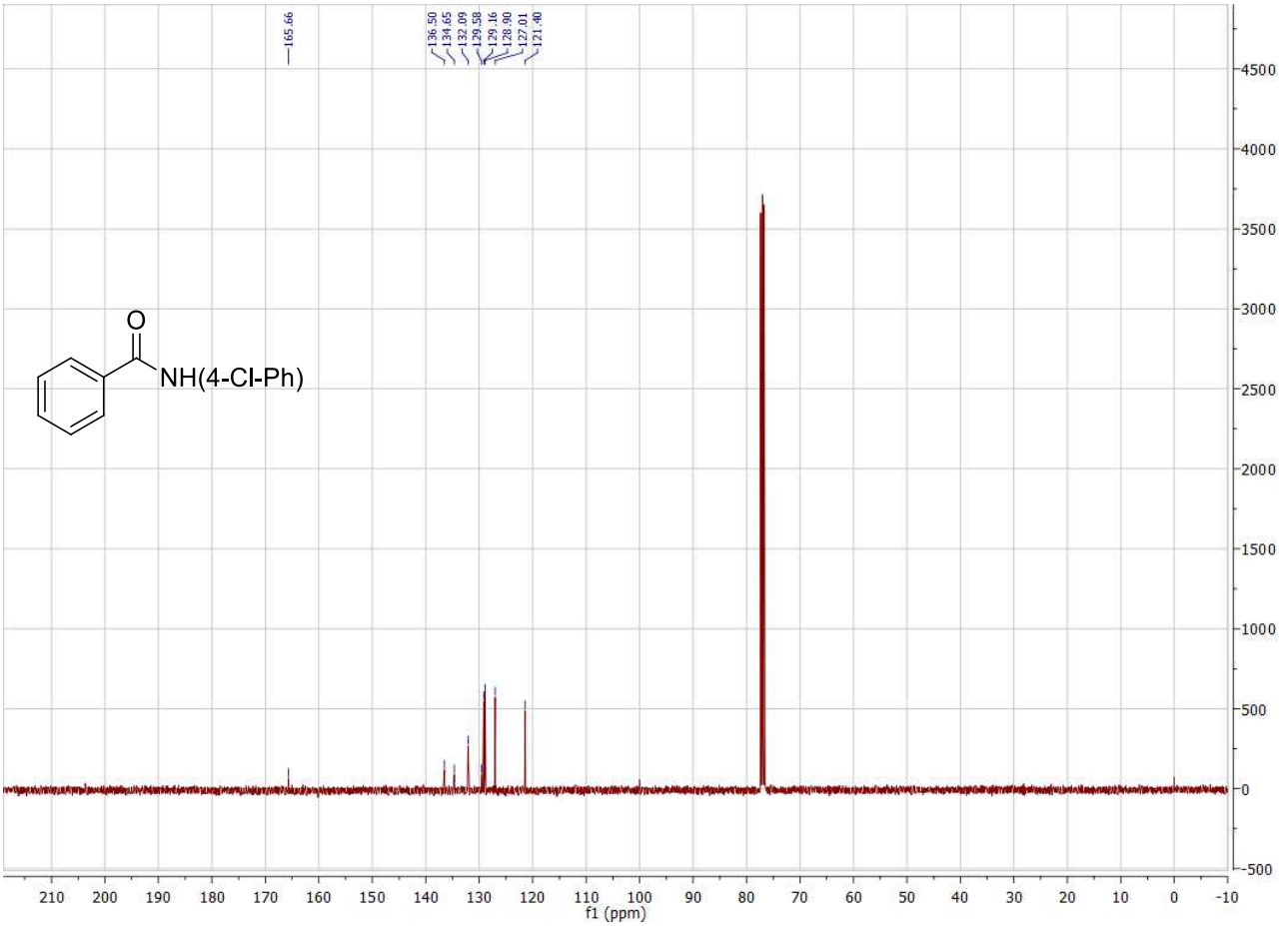

7.68
7.67
7.65
7.65
7.55
7.45
7.44
7.43
7.26
4.52
4.50
4.50
4.50
4.48
4.21
4.19
4.19
4.17
0.00
1.95
1.00
1.99
2.05
2.04
f1 (ppm)

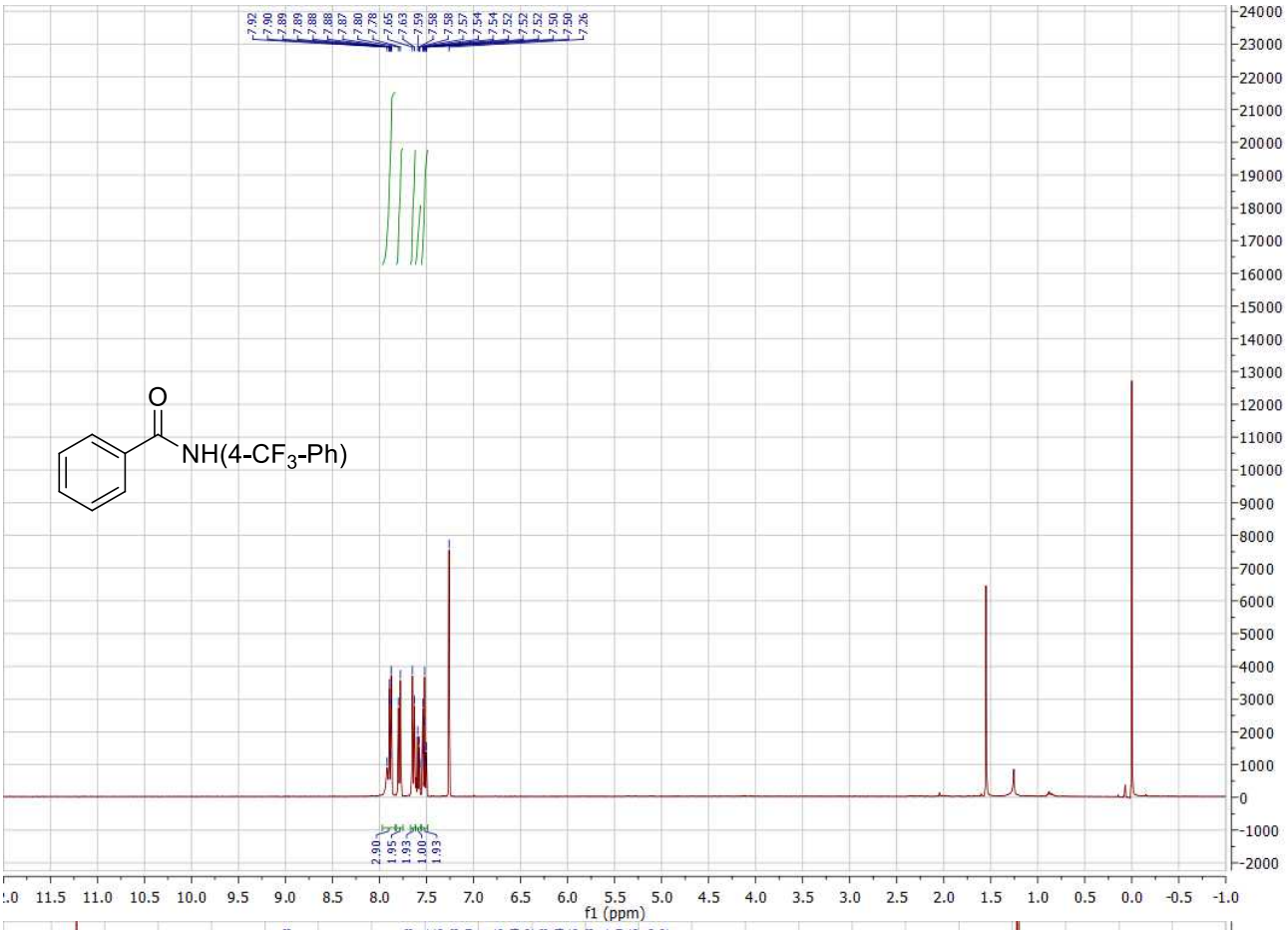

169.83
153.21
132.59
132.46
129.12
127.91
77.35 CDCl3
77.04 CDCl3
76.72 CDCl3
62.26
43.74
f1 (ppm)

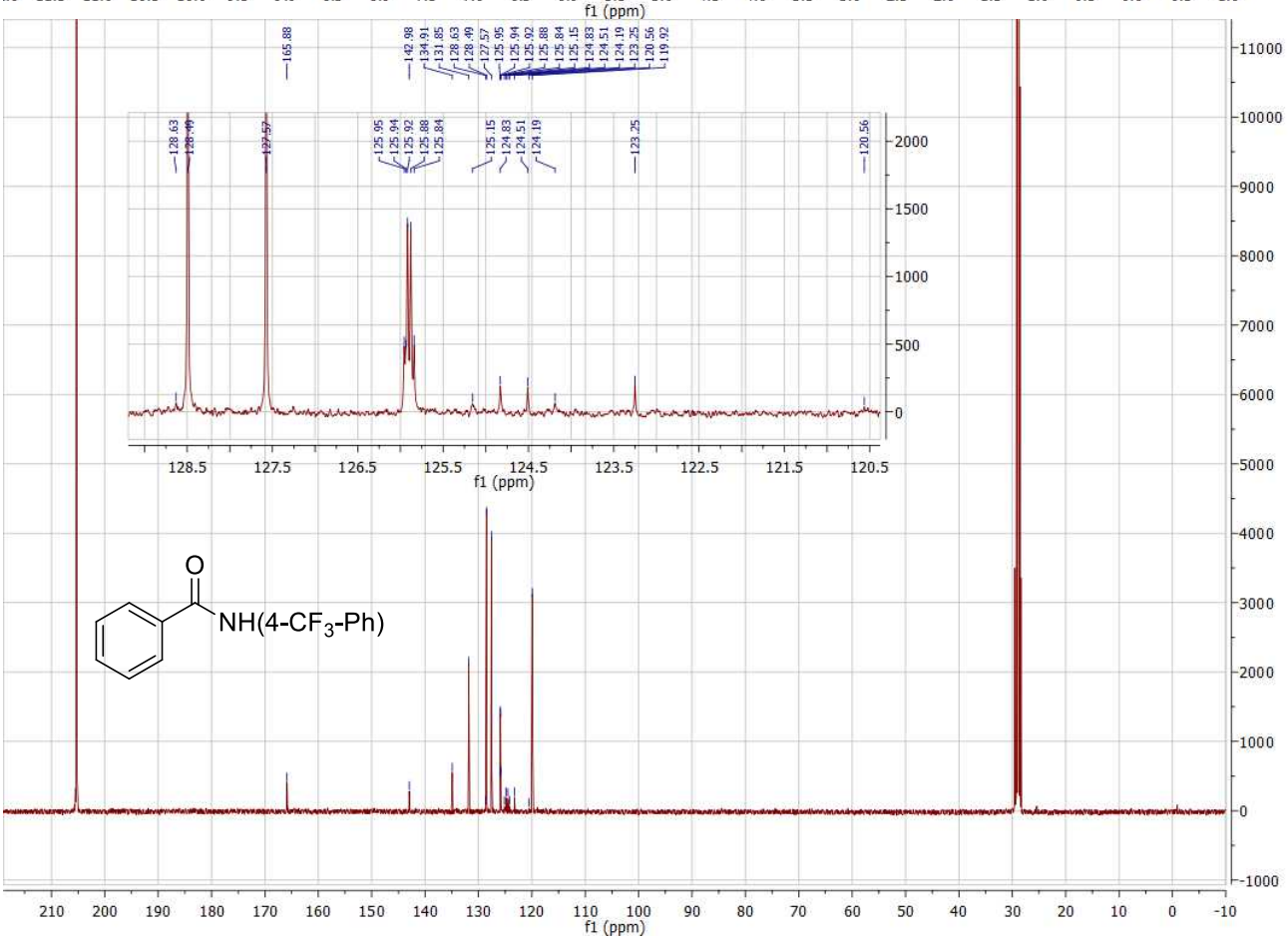

7.88
7.88
7.86
7.86
7.66
7.65
7.64
7.63
7.55
7.50
7.49
7.48
7.39
7.37
7.37
7.35
7.15
0.00
O
NHPh
2.88
2.00
1.03
1.99
2.02
1.00
f1 (ppm)

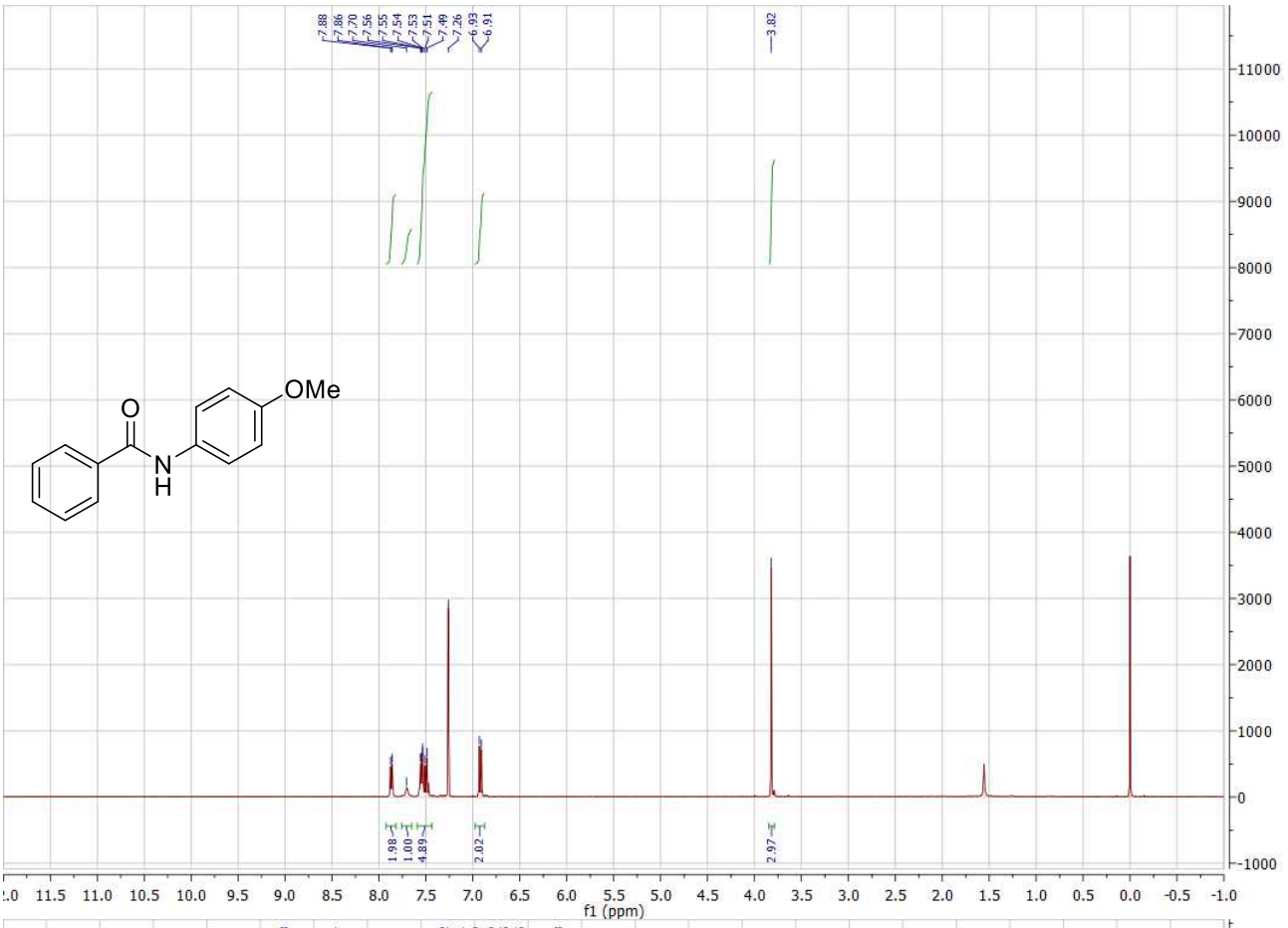

165.75
137.93
135.03
131.87
129.13
128.82
127.03
124.60
120.21
77.36 CDCl3
77.04 CDCl3
76.73 CDCl3
O
NHPh
f1 (ppm)

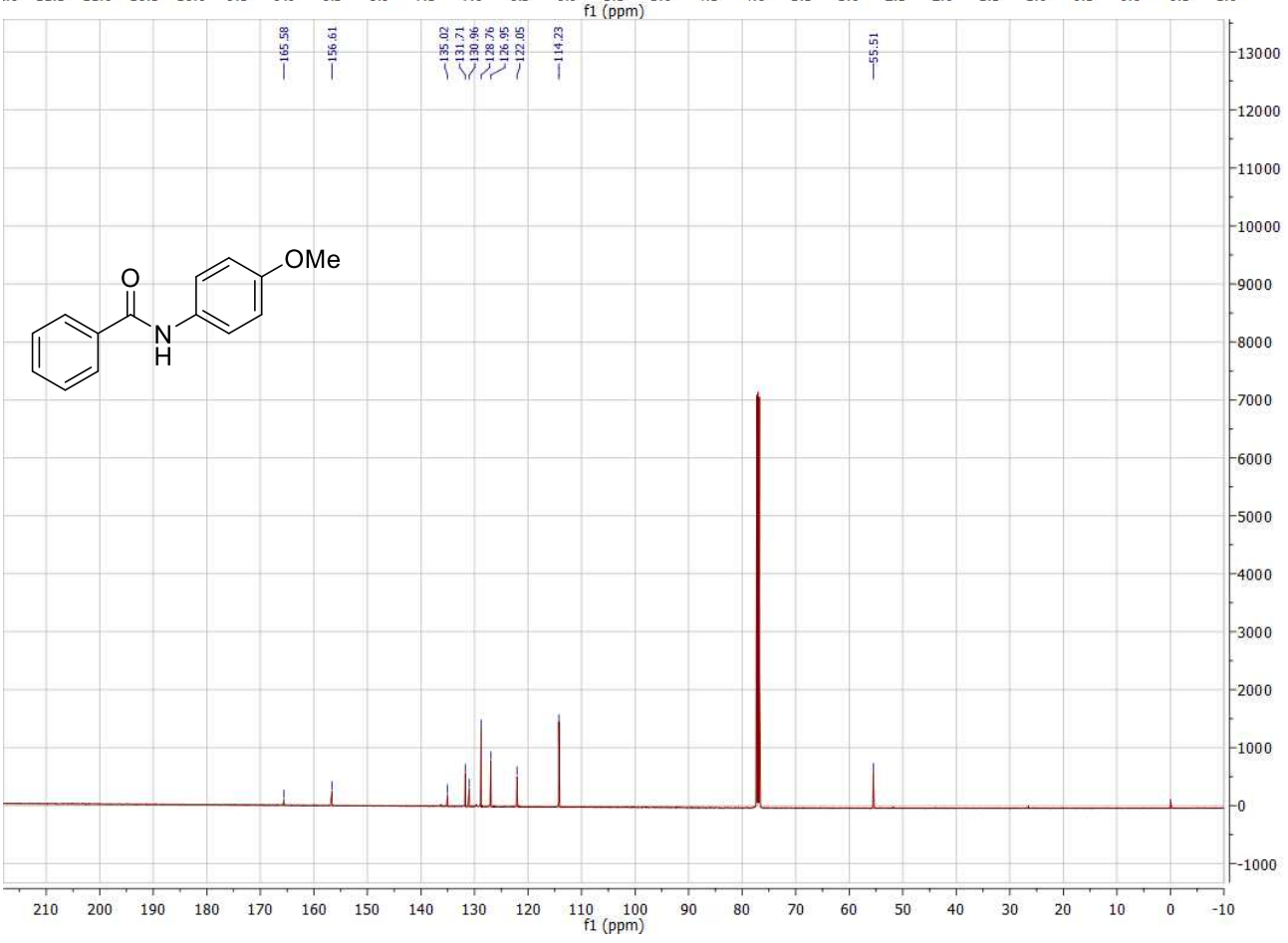

NH(4-Cl-Ph)
f1 (ppm)

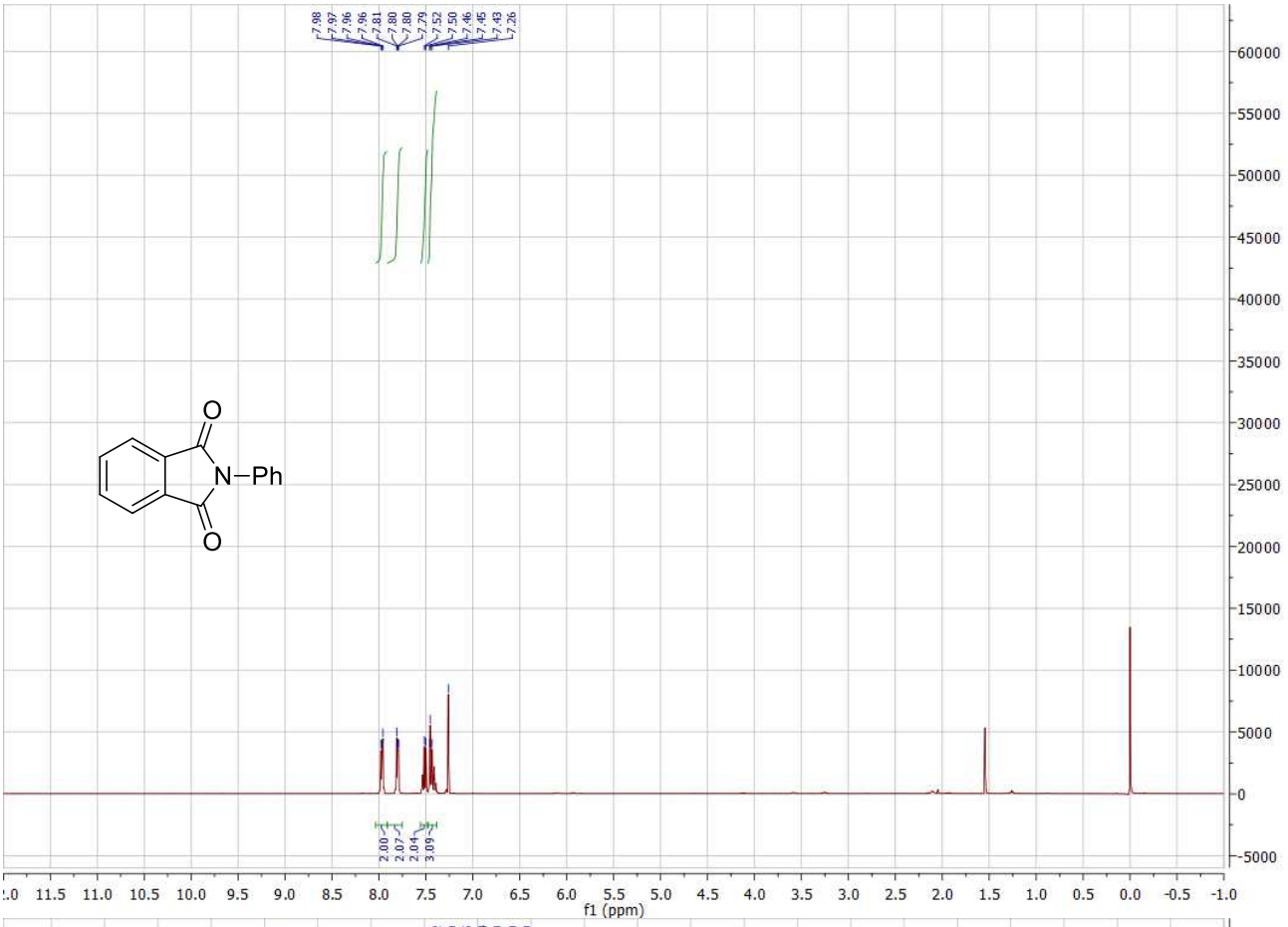

NH(4-Cl-Ph)
f1 (ppm)

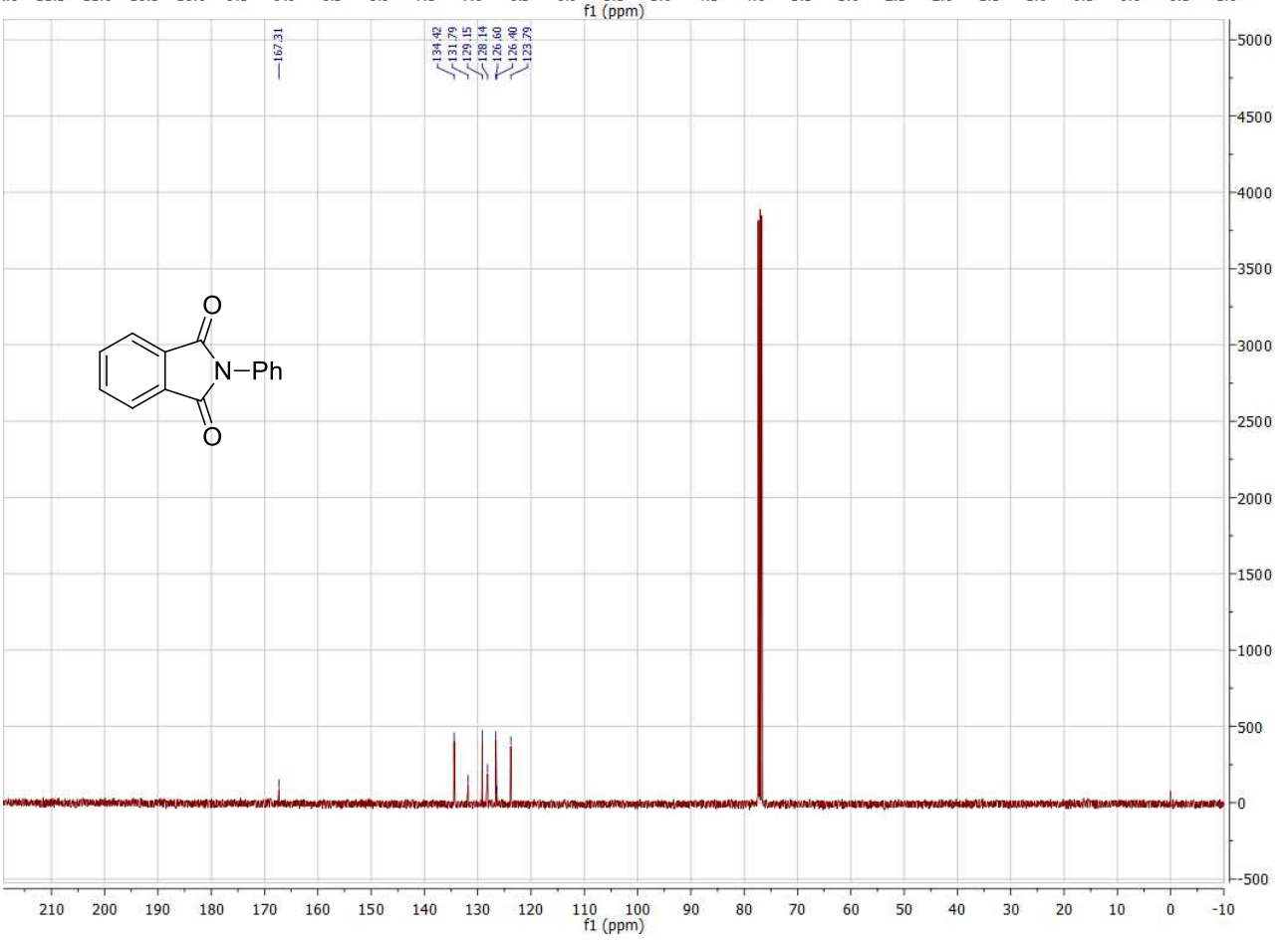

NH(4-CF3-Ph)
f1 (ppm)

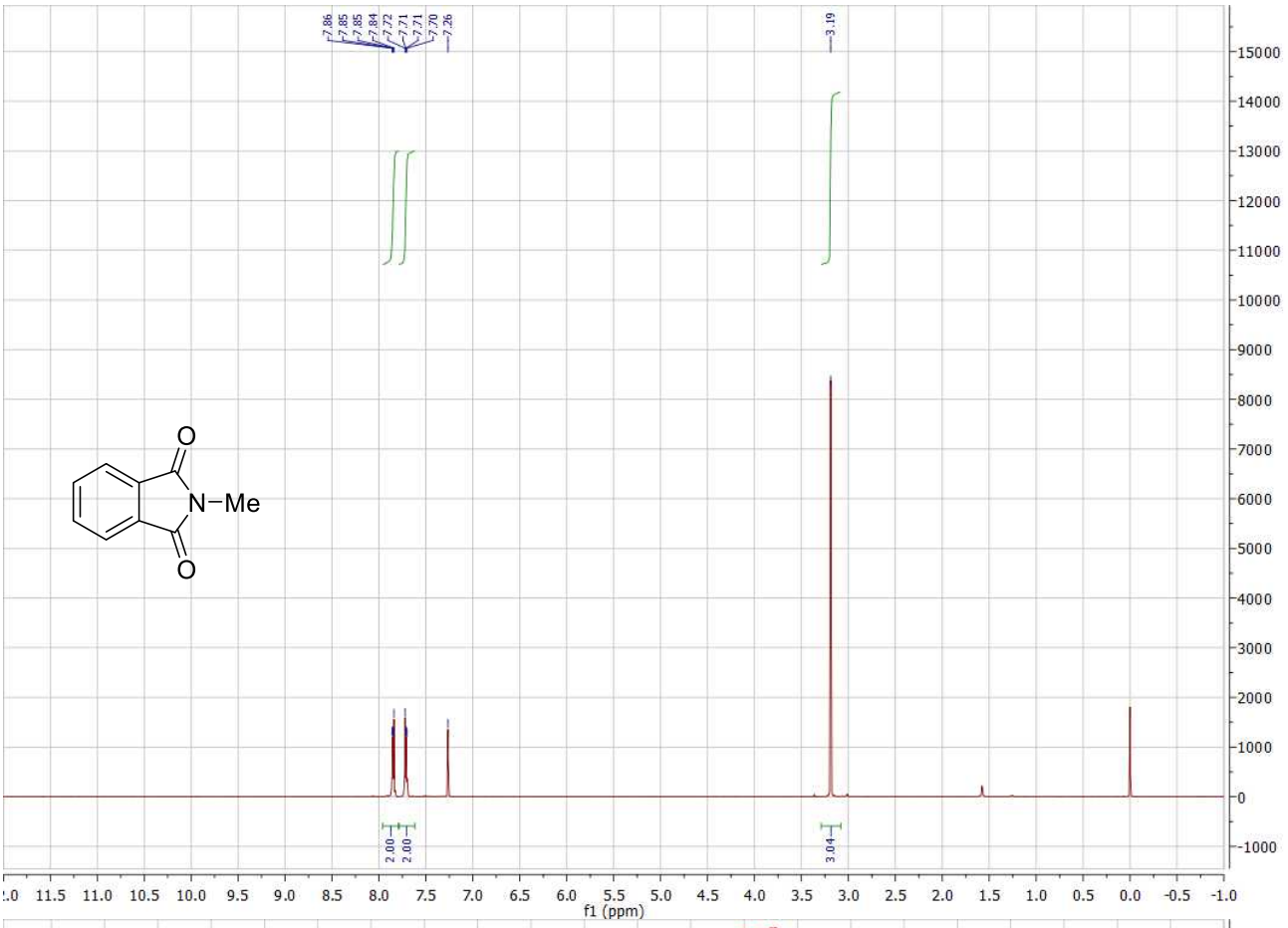

NH(4-CF3-Ph)
f1 (ppm)

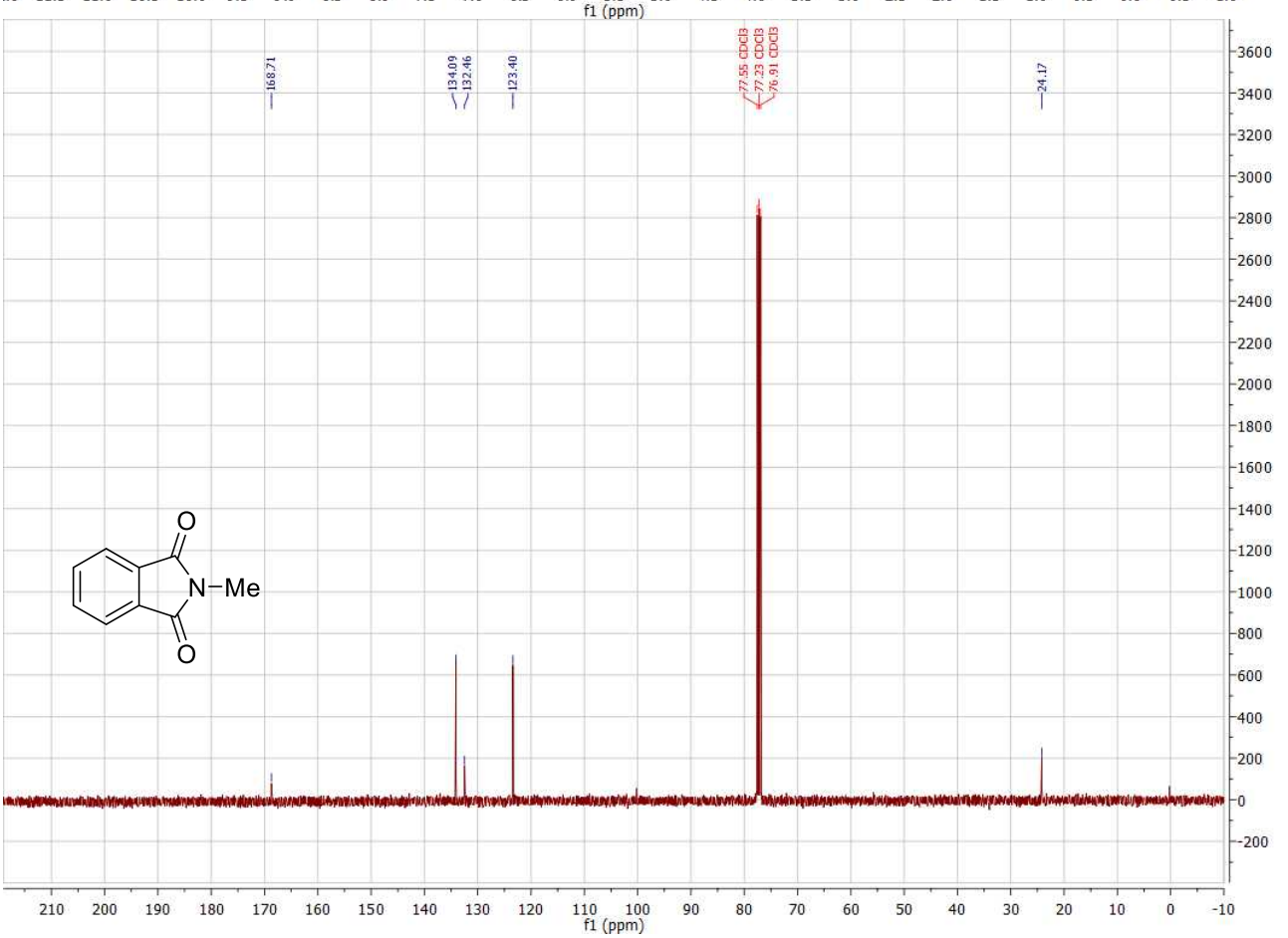

7.88
7.86
7.70
7.56
7.55
7.54
7.53
7.51
7.49
7.26
6.93
6.91
3.82
OMe
O
N
H
1.98
1.00
4.89
2.02
2.97
f1 (ppm)

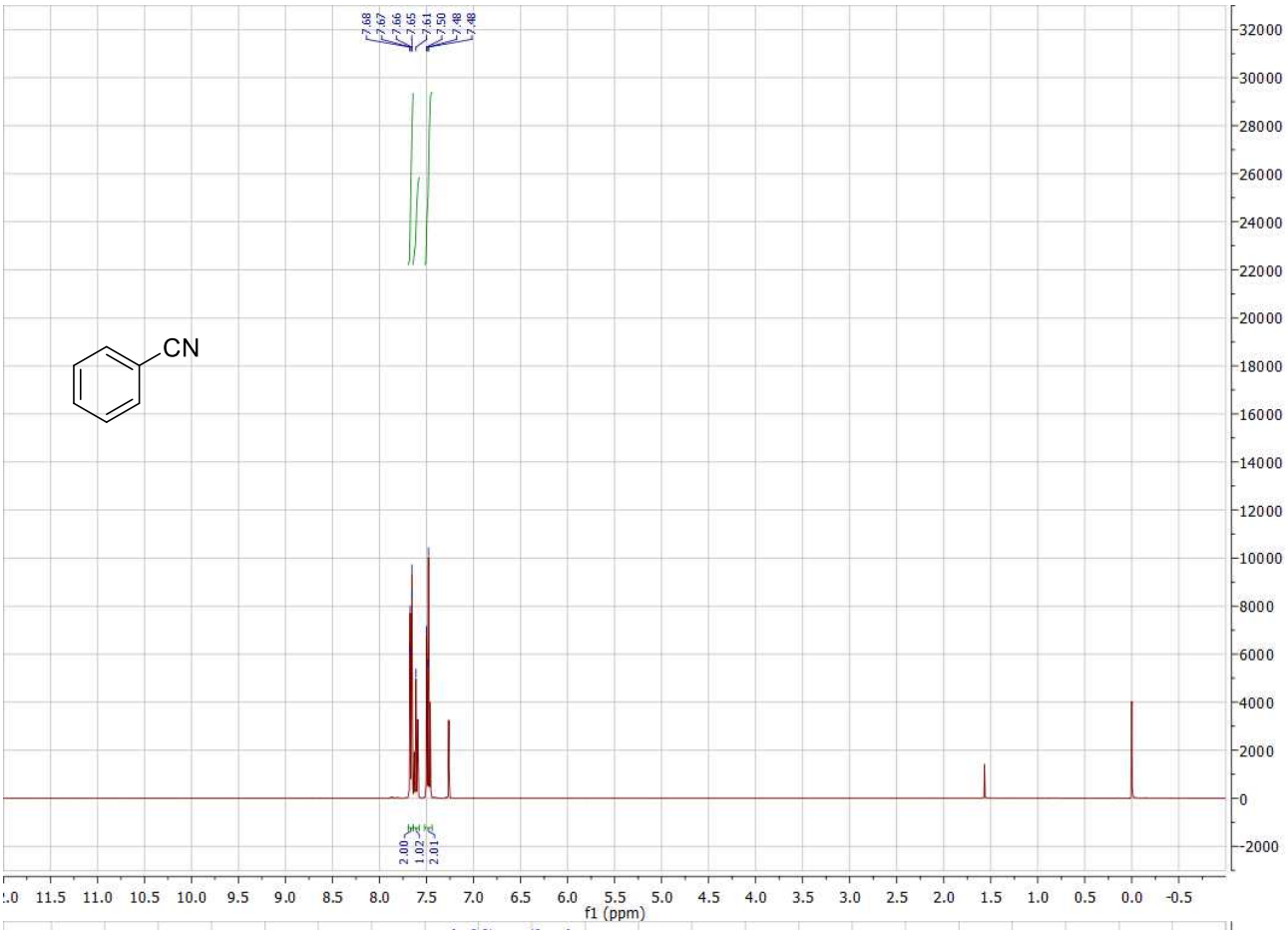

165.58
156.61
135.02
131.71
130.96
128.76
126.95
122.05
114.23
55.51
OMe
O
N
H
f1 (ppm)

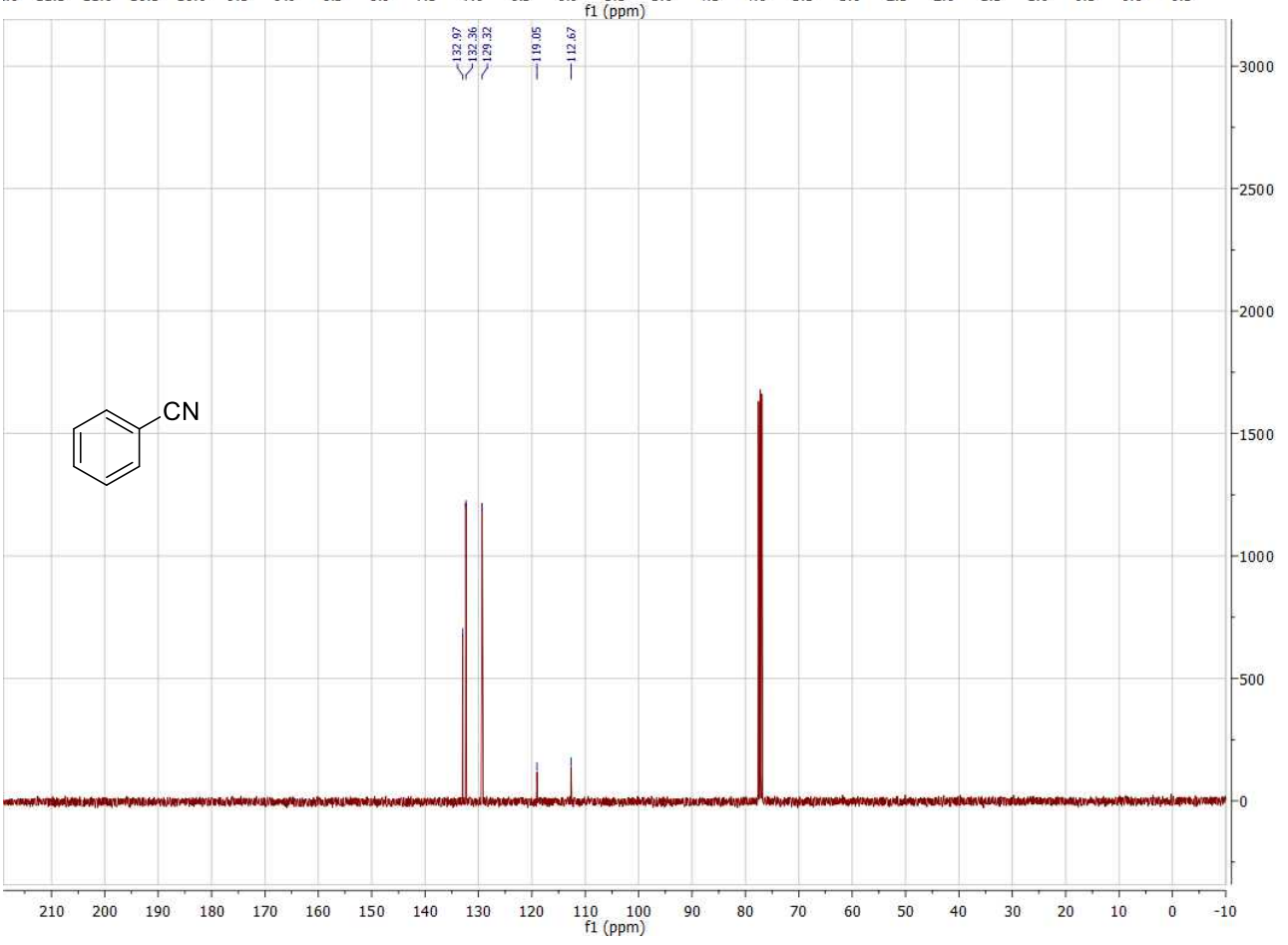

7.98
7.97
7.96
7.96
7.81
7.80
7.80
7.79
7.52
7.50
7.46
7.45
7.43
7.26
N–Ph
2.00
2.07
2.04
3.09
f1 (ppm)

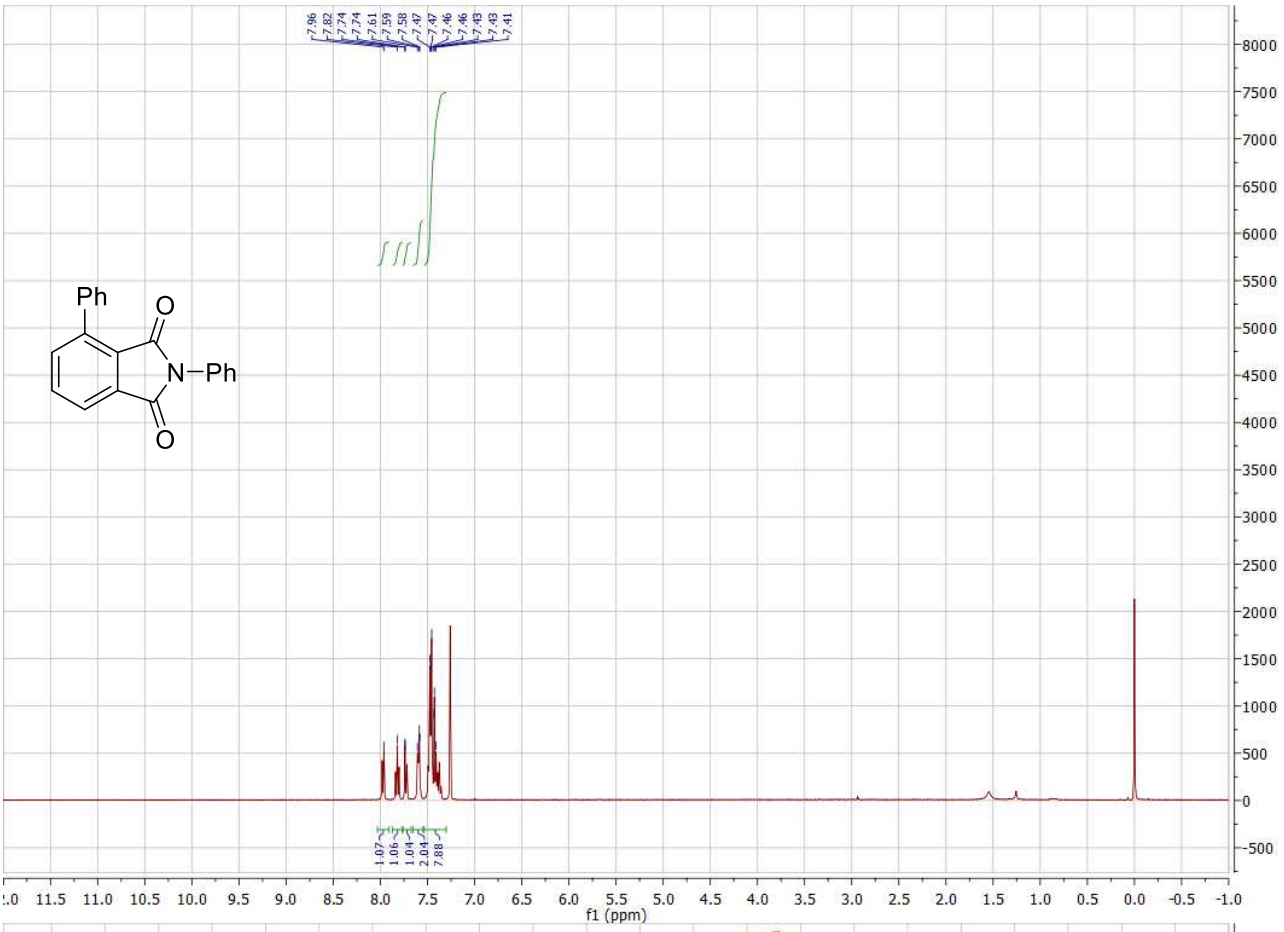

167.31
134.42
131.79
129.15
128.14
126.60
126.40
123.79
N–Ph
f1 (ppm)

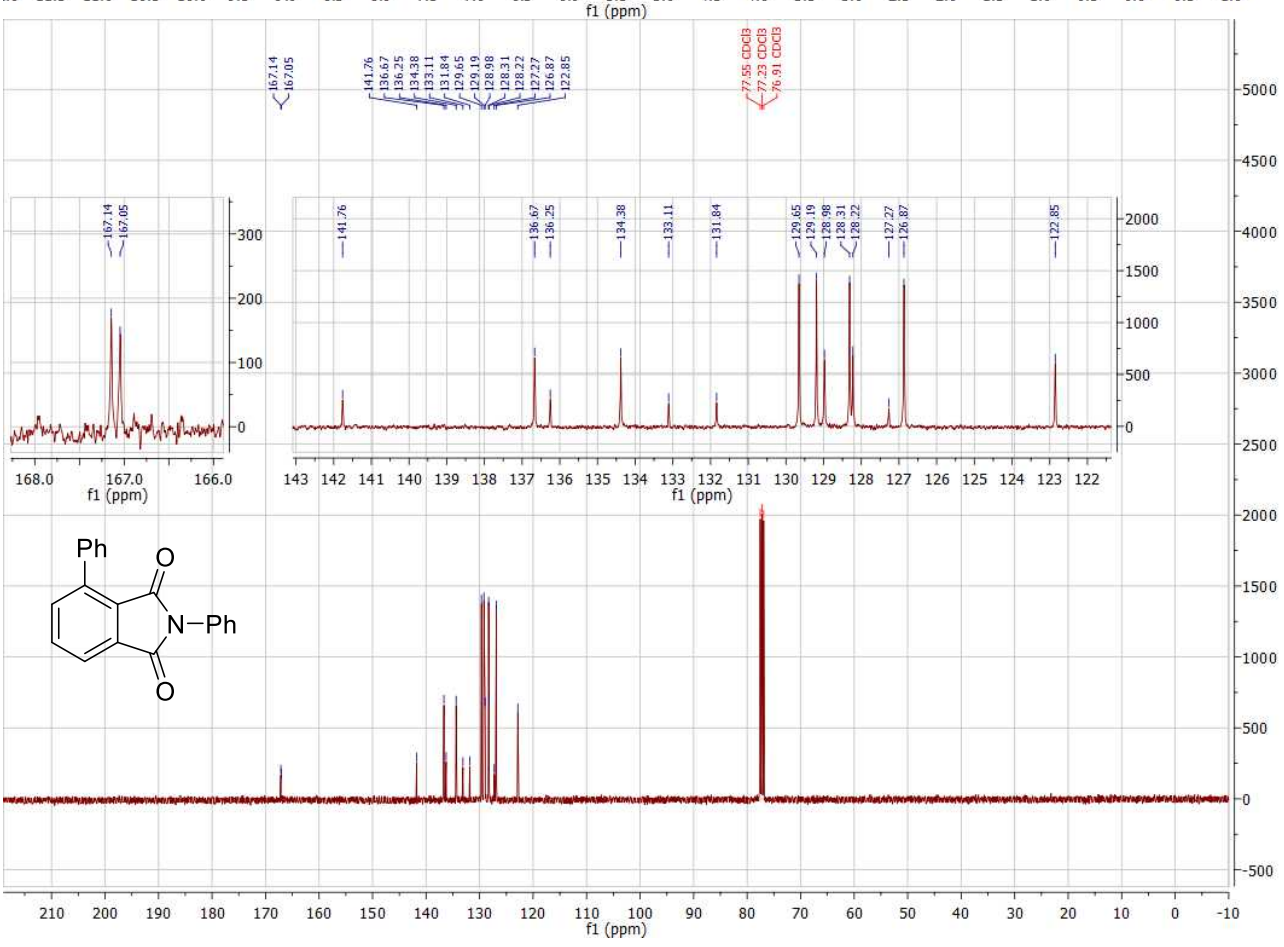

7.86
7.85
7.85
7.84
7.72
7.71
7.71
7.70
7.26
3.19
O
N−Me
O
2.00
2.00
3.04
f1 (ppm)

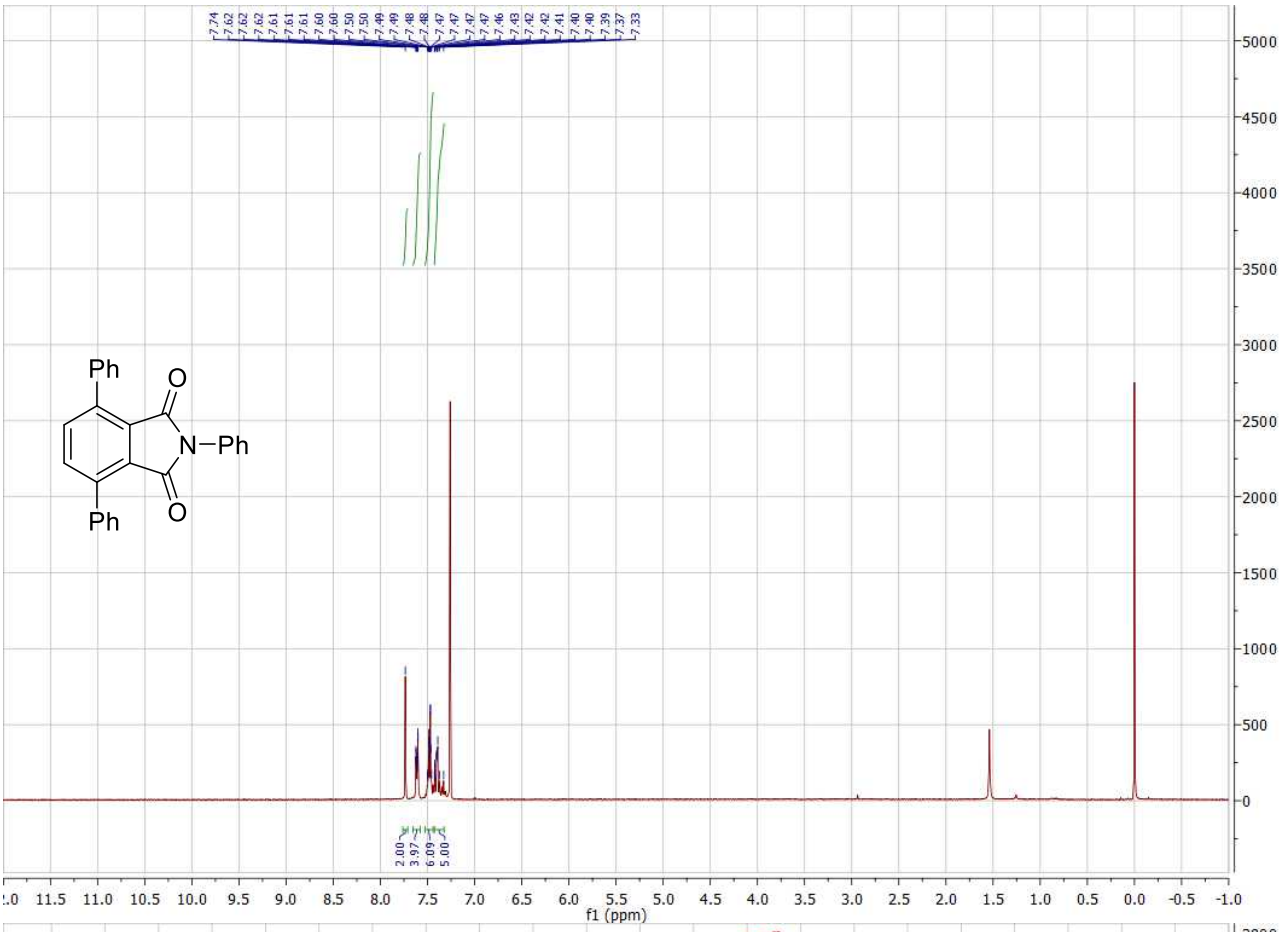

168.71
134.09
132.46
123.40
77.55 CDCl3
77.23 CDCl3
76.91 CDCl3
24.17
O
N−Me
O
f1 (ppm)

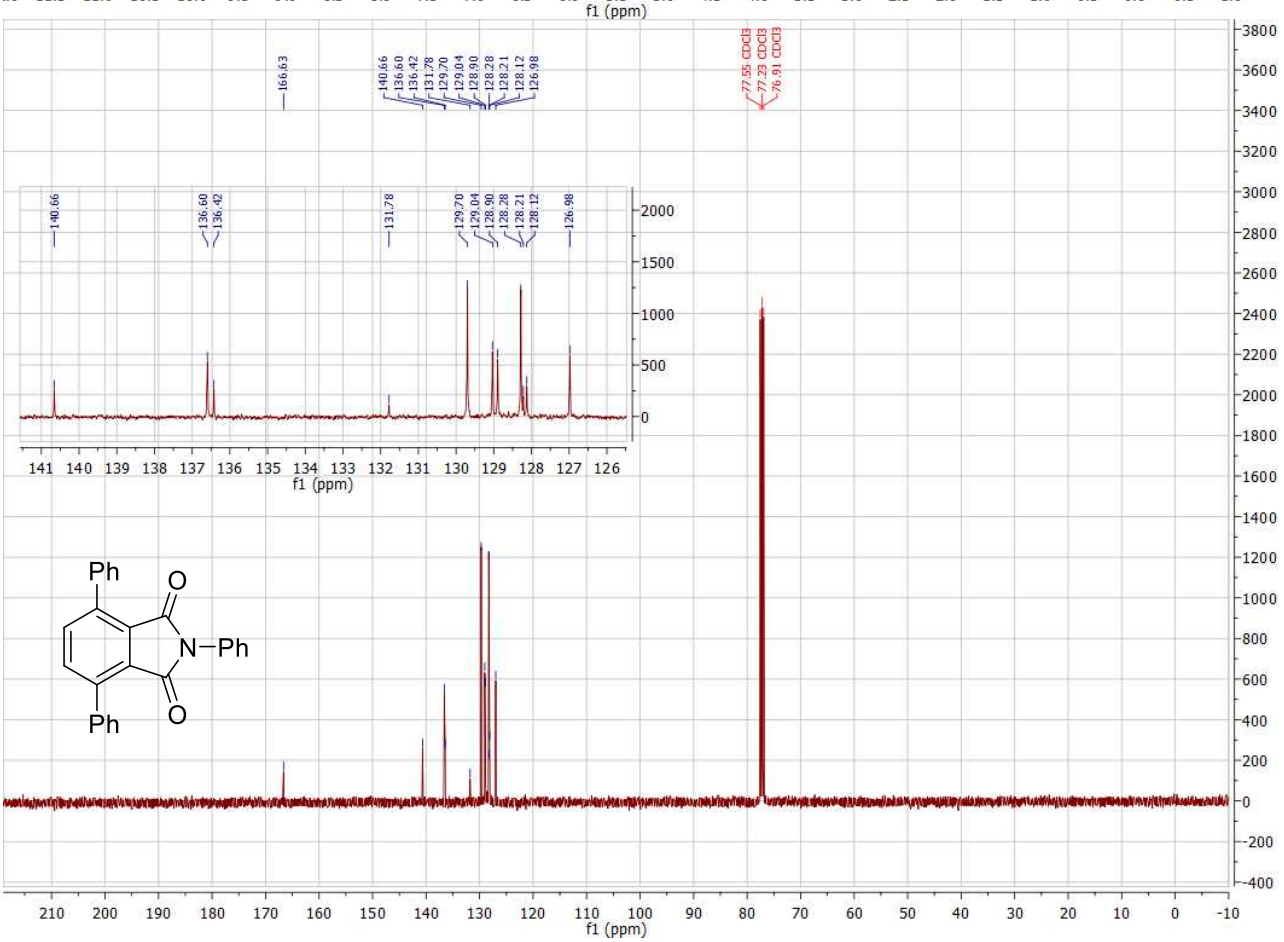

7.68
7.67
7.66
7.65
7.61
7.50
7.48
7.48
CN
2.00
1.02
2.01
f1 (ppm)

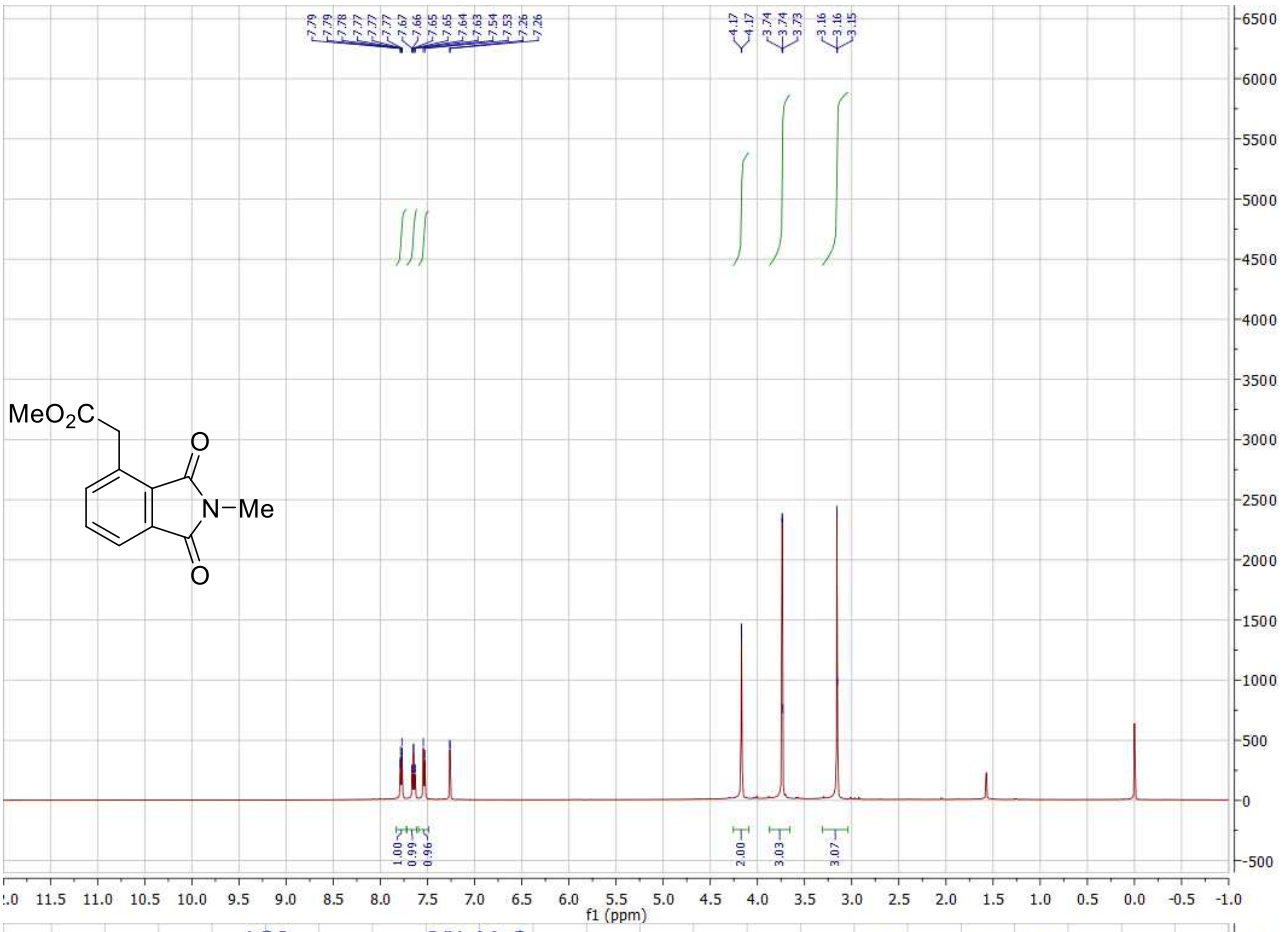

132.97
132.36
129.32
119.05
112.67
CN
f1 (ppm)

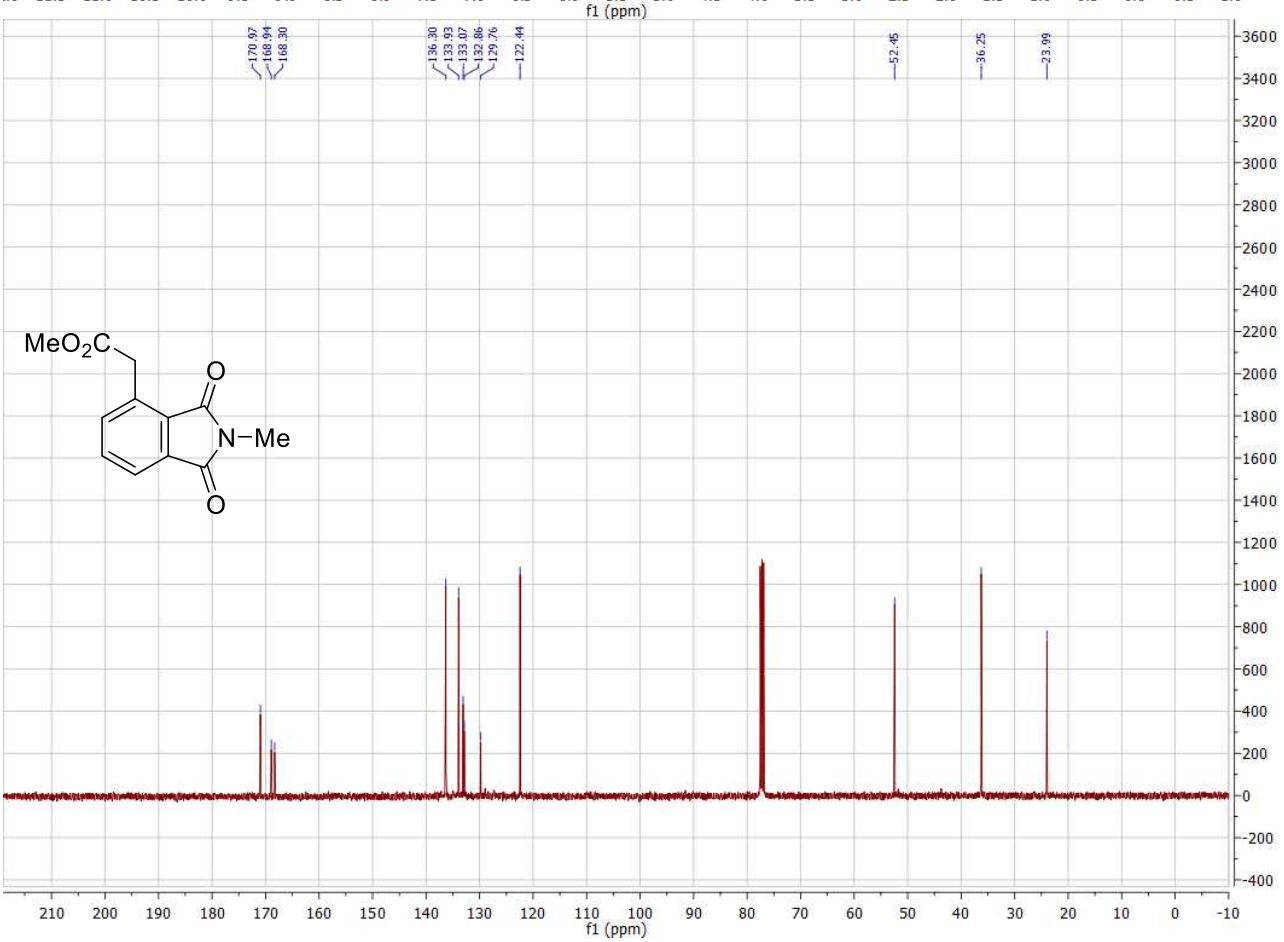

Ph
O
N–Ph
O
f1 (ppm)


Ph
O
N–Ph
O
f1 (ppm)


Ph
O
N−Ph
O
Ph
f1 (ppm)


Ph
O
N−Ph
O
Ph
f1 (ppm)


MeO2C
O
N−Me
O
f1 (ppm)


MeO2C
O
N−Me
O
f1 (ppm)