Supporting Information

Nickel(II)– hydrazineylpyridine catalyzed regioselective synthesis of *α*-benzyl substituted *β*-hydroxy ketones *via* Fenton free radical reaction

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Table of Contents

[1.1] General Experimental Details:

All reactions were conducted at different temperature using the respective solvents. Distilled water was used during the work-up procedures, and all solvents were dried and stored over activated molecular sieves (4 Å).

Analytical tools: All the industrial reagents used were purchased from Sigma-Aldrich, Avra, Aldrich, TCI and Otto-chemie chemical. Thin layer chromatography (TLC) functioned on a pre-coated silica gel 60 F254 plate. Visualization over TLC was accomplished by UV light (254 nm), and $KMnO_4$ strain. The melting point is determined via the Optimelt melting apparatus. Column chromatography was done on silica gel of 200-300 mesh using respectable eluents. The UV-visible spectra were obtained using a Shimadzu Europe UV-2600 Scientific UV-visible spectrophotometer. Infrared spectra were recorded using KBr pellets with a PerkinElmer FT-IR spectrometer. A Bruker-D AVANCE (III)-AV-500 (500 MHz) Spectrometer recorded ^{1}H and ^{13}C NMR spectroscopic data, and the chemical shifts were measured in parts per million (*ppm*) referenced to the appropriate solvent peak [$CDCl_3$ and DMSO-d_6]. Chemical shifts (δ) exist in ppm and coupling constants (J) are in Hertz (*Hz*). The subsequent abbreviations are used to explain the multiplicities in NMR spectra: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. FT-IR spectra were recorded by Perkin–Elmer FT–IR Spectrometer.

Chemicals: Commercially available chemicals from Sigma-Aldrich, Avra, Aldrich, TCI and Otto-chemie were purchased and used without further purification.

[1.2] General Procedures

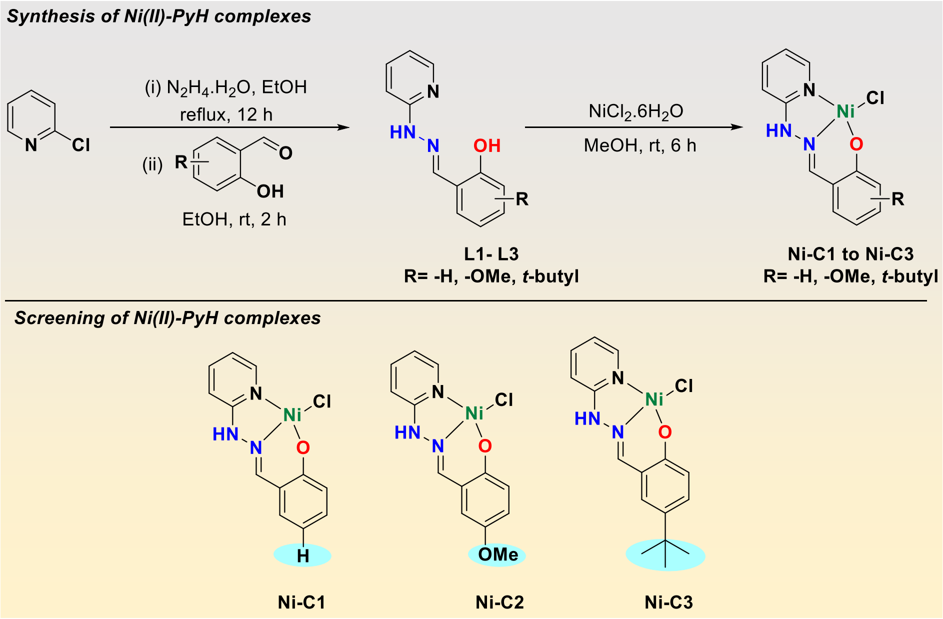
General Procedure for precursor 2-hydrazineylpyridine and ligands synthesis

2-hydrazineylpyridine synthesized from the 2-chloropyridine (1 mmol, 0.113 g) precursor in the presence of hydrazine hydrate (5 mmol, 0.250 g) in ethanol at reflux condition for 12 h. After TLC (Thin layer chromatography) monitoring, the mixture is dried via vacuum and extracted via column chromatography with mobile phase EtOAc: Hexane (1:4).

2-hydrazineylpyridine: 87.2 mg, 80% yield, white solid, melting point = 46 ^oC - 47 ^oC, R_f = 0.21 [EtOAc: Hexane(1:4)]. **FT**-**IR** spectra show a sharp band at 1032 cm^{-1} due to v(N–

N) stretch. **^{1}H NMR (500 MHz, $CDCl_3$):** δ 8.09 (dd, J = 3.2, 1.9 Hz, 1H), 7.51 – 7.41 (m, 1H), 6.68 – 6.62 (m, 2H), 6.05 – 6.01 (m, 1H), 3.51 (s, 2H). **^{13}C NMR (126 MHz, $CDCl_3$):** δ 161.30, 147.71, 137.62, 114.54, 107.12.

Scheme S1. Synthesis of ligand and nickel metal complex



The Ligands (**L_1-L_3**), were synthesized by a condensation reaction between equimolar 2-hydrazino pyridine (1 mmol, 0.109 g) and salicylaldehyde (1 mmol, 0.122 g) at room temperature in ethanol. After 2 h, the precipitate formed was collected by vacuum filtration. All ligands were crystallised in solvent MeOH and DCM at a 3:1 ratio, separated on cooling, and collected by filtration. Various characterization of ligands such as IR, ^{1}H and ^{13}C NMR, and CHNS studies were performed.

General Procedure for the synthesis of Nickel complexes

Procedure A: In a 50 mL round bottom flask, **L_1** (1 mmol, 0.213 g) and $NiCl_2.6H_2O$ (1 mmol, 0.237 g) were added, followed by methanol at room temperature for 6 h. The bright yellow precipitate formed was filtered, washed with methanol and dried. **Ni-C1** was synthesised with 92 % yield, i.e., 0.294 g, which was further characterized using spectroscopic techniques such as UV-Vis, IR, XPS, and TGA. Similarly, **Ni-C2** and **Ni-C3** were synthesised and characterized.

Procedure B: In a 50 mL round-bottom flask, **L_1** (1 mmol, 0.213 g), and $NiCl_2.6H_2O$ (1 mmol, 0.237 g) were added, followed by dropwise addition of TEA (1 equiv.) in methanol at room temperature for 6 h. The bright yellow precipitate formed was filtered, washed with methanol and dried. **Ni-C1** synthesised with 95 % yield i.e., 0.304 g.

General Procedure for the synthesis of γ-hydroxy ketone

In a 50 mL round-bottom flask, add **Ni-C3** catalyst (0.5 mol%), *30% aq.* H_2O_2 (5 mmol) in 5 mL of toluene and stirred at room temperature for 6 h. Afterwards, add (*E*)-chalcone/ *2*'-hydroxychalcone / *2'*-aminochalcone (1 mmol) and benzyl or aliphatic alcohol (1 mmol) to the reaction mixture and stir for a further 6 h at 100 ^{o}C. TLC monitoring, the toluene was evaporated and then reaction mixture diluted using distilled water. The reaction mixture was separated with EtOAc three times and dried over Na_2SO_4. The column chromatography was performed on silica gel (100-200 mesh), eluted with hexane as a mobile phase. All the products observed are oily in nature, which was further characterized spectroscopically.

[1.3] Catalyst Characterization

UV-Visible and Infrared studies

The UV-visible data were recorded in ethanol solvent. UV-visible studies of all the ligands show similar two bands around 341 and 307 nm in the UV region. Depending on their extinction coefficient, these bands must be of n−π* and π−π* transitions (**Figure S1**). Titrating the ligand solution with its equimolar nickel salt solution shows the rise of two new bands at 404 and 258 nm, respectively, resembling the reaction mixture's spectra. Reduction in the intensity of the 341 nm peak of ligand solution confirms the ligand metal interaction. A small hump due to a peak at 404 nm is represented visually by its colour change while adding salt solution dropwise. Concentrations, 1 nM of nickel salt and ligand solutions in ethanol are used. No peak is observed for a visible reason except the 404 nm peak. The peak at 341 nm due to ligand n−π* vanishes, and a peak at 404 nm come into view, showing chelation of metal ion with the ligand (LMCT), i.e., the ligand to metal charge transfer. Infrared spectra of complexes showed a band in the range of 1600-1625 cm^{-1} resulting to the azomethine (C=N) stretching frequency and the two strong bands near 365 cm^{-1} and 770 cm^{-1} were observed in all complexes which were attributed to ligand and metal bond Ni-Cl and Ni-O bonding. In context to the ligand, the azomethine (C=N) stretching frequency decreases for ~20 cm^{-1} (**Figure S2**). All the ligands and metal complexes show similar trends in IR absorption (**Figure S2-S4**).

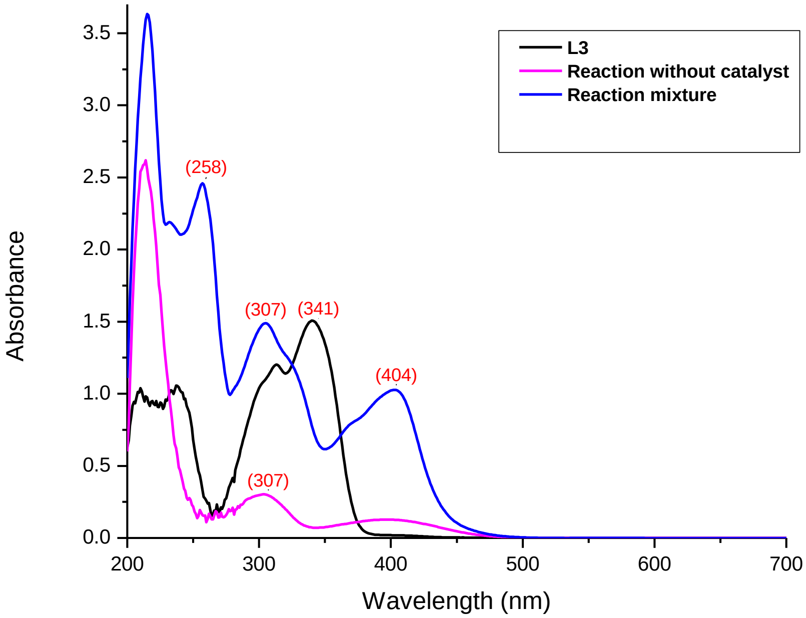


Figure S1. UV-Vis spectra of **L3** (1 nM) in EtOH with Ni^{+2} salt (1 nM) in reaction mixture and reaction without catalyst.

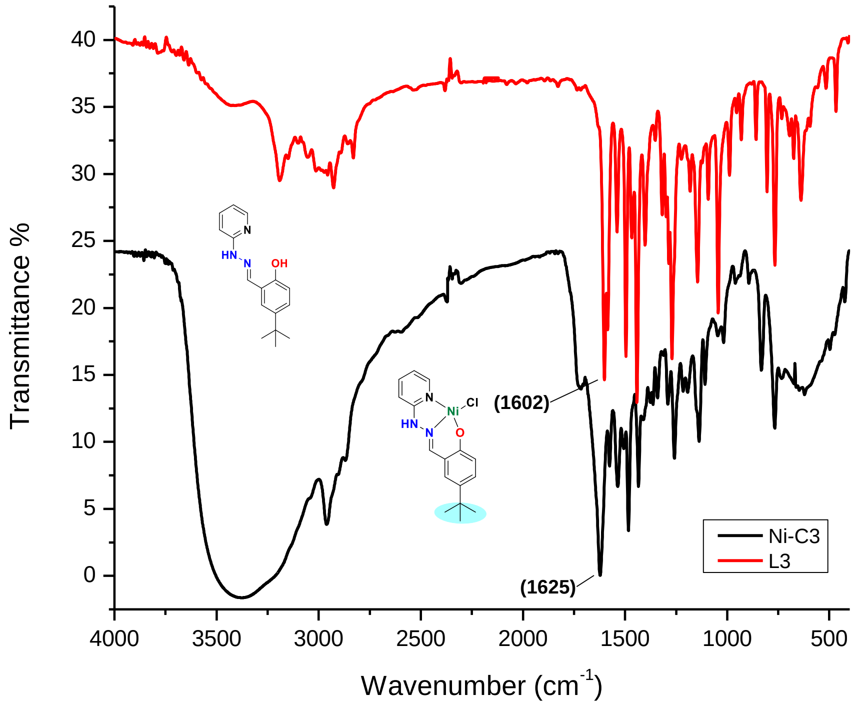


Figure S2. Infrared spectra for **L3** and **Ni-C3**

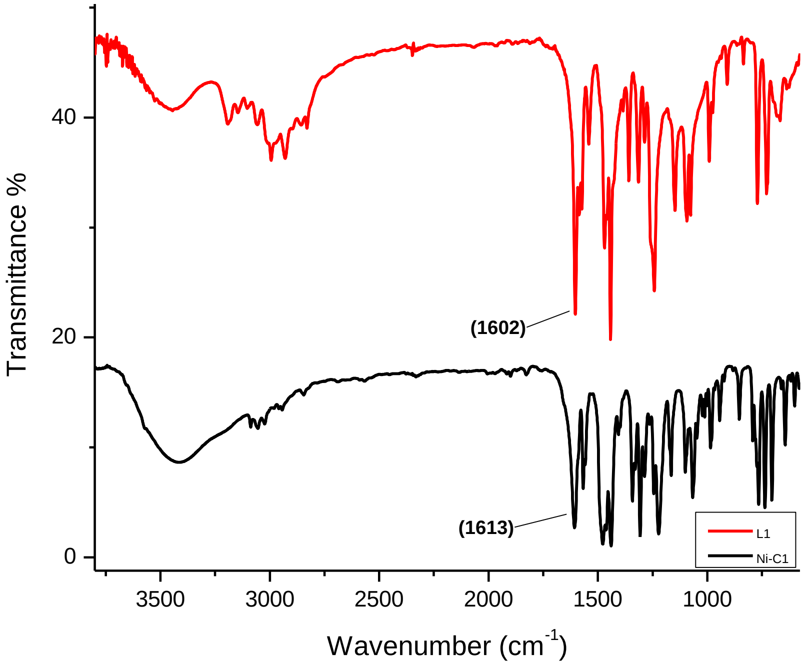


Figure S3. Infrared spectra for **L1** and **Ni-C1**

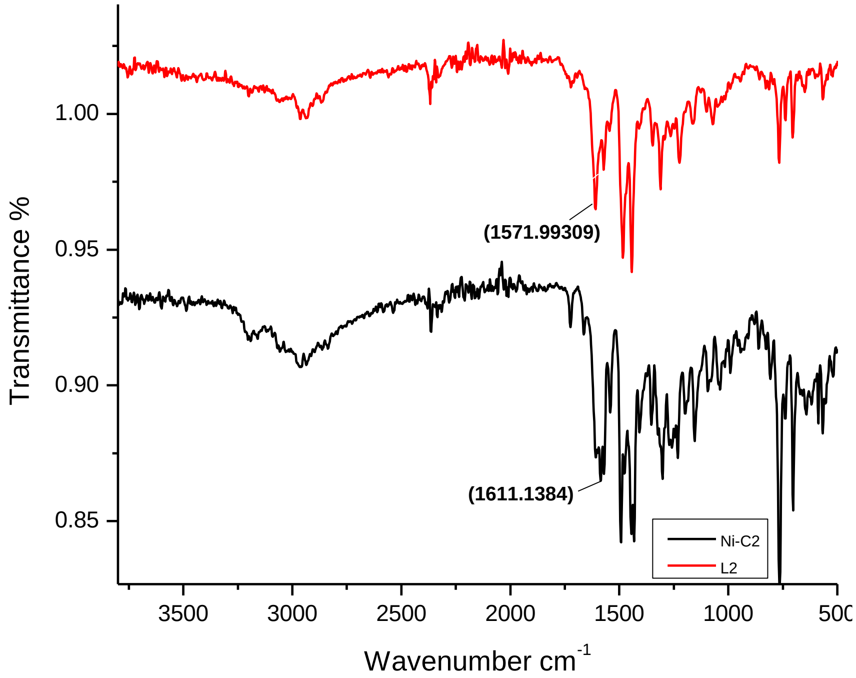
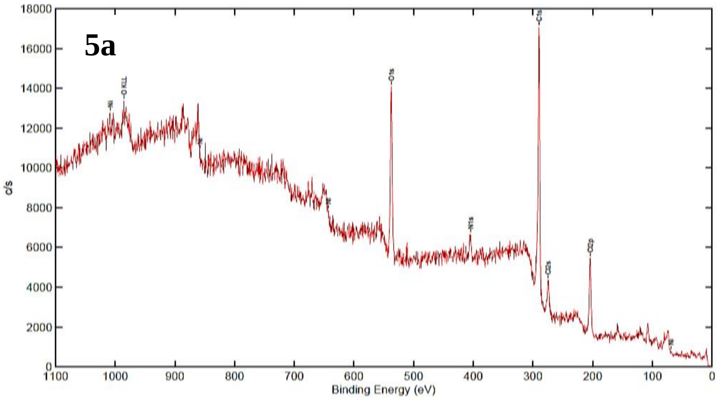
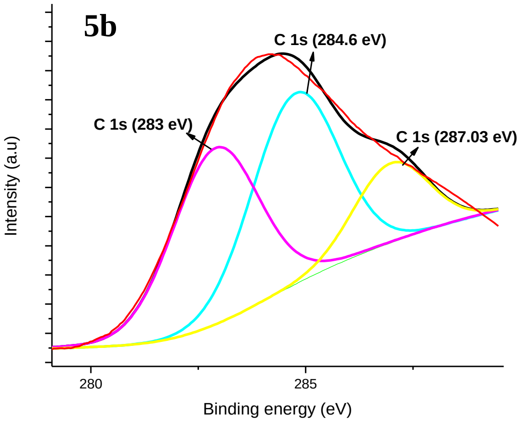


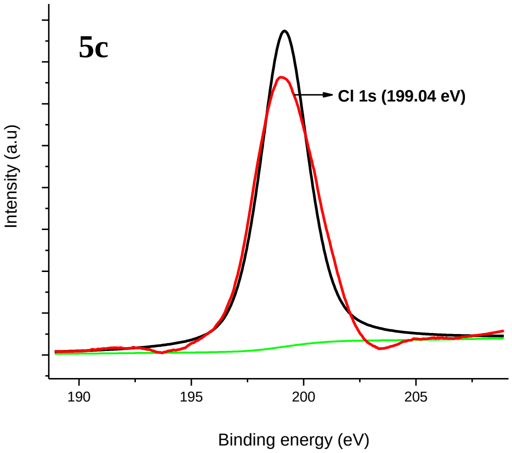
Figure S4. Infrared spectra for **L2** and **Ni-C2**

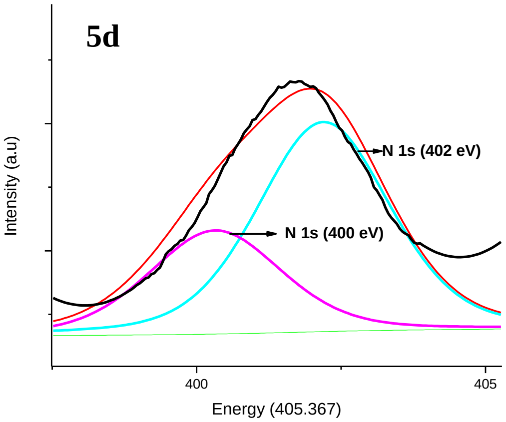
XPS studies

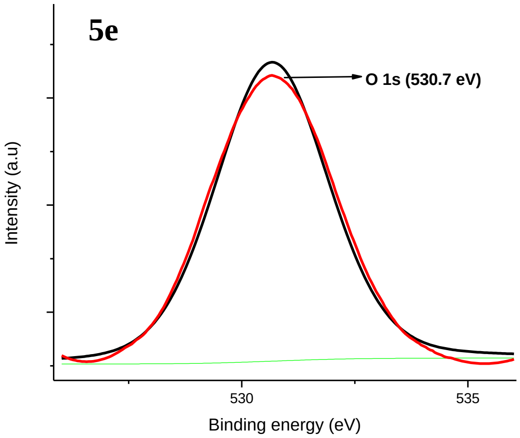
The XPS experiment allows us to measure the kinetic energy of the photoelectrons extracted or emitted from the sample or solid, within the energy range that allows the X-ray source. To confirm the structure and metal oxidation state in **Ni-C3**, XPS characterization is done with the PHI-5000 VersaProbe III ULVAC-PHI instrument. The survey confirmed C, Cl, Ni, O and N atoms in the framework (**Figure S5a**), the C 1s high-resolution XPS spectrum of Ni-C3 showed three peaks at 287.03, 284.6, and 283 eV corresponding to the number of chemical states and therefore the number of peaks C1, C2 and C3 respectively (**Figure S5b inset**). High-resolution XPS spectrum for Cl 1s (**Figure S5c inset**) showed two peaks at 199.04 eV for the corresponding Cl; the two distinct peaks of N1, and N2 for N1s are visible at 400 and 402 eV, respectively (**Figure S5d inset**).; O 1s atom shows binding energy at 530.7 eV (**Figure S5e inset**). The catalyst Ni-C3 was measured, with a sharp peak centred at 861.4 eV for Ni 2p3/2 (**Figure S5f inset**). This can be assigned as our catalyst's Ni (II) oxidation state. The Ni 2p1/2 state shows a peak at 878 eV along with a satellite peak at 885 eV.


5a
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
c/s
1100
1000
900
800
700
600
500
400
300
200
100
0
Binding Energy (eV)
Ni
O KLL
Ni
Ni
O1s
N1s
C1s
Cl2s
Cl2p
Ni


5b
C 1s (284.6 eV)
C 1s (283 eV)
C 1s (287.03 eV)
Intensity (a.u)
280
285
Binding energy (eV)


5c
Cl 1s (199.04 eV)
Intensity (a.u)
190
195
200
205
Binding energy (eV)


5d
N 1s (402 eV)
N 1s (400 eV)
Intensity (a.u)
400
405
Energy (405.367)


5e
O 1s (530.7 eV)
Intensity (a.u)
530
535
Binding energy (eV)

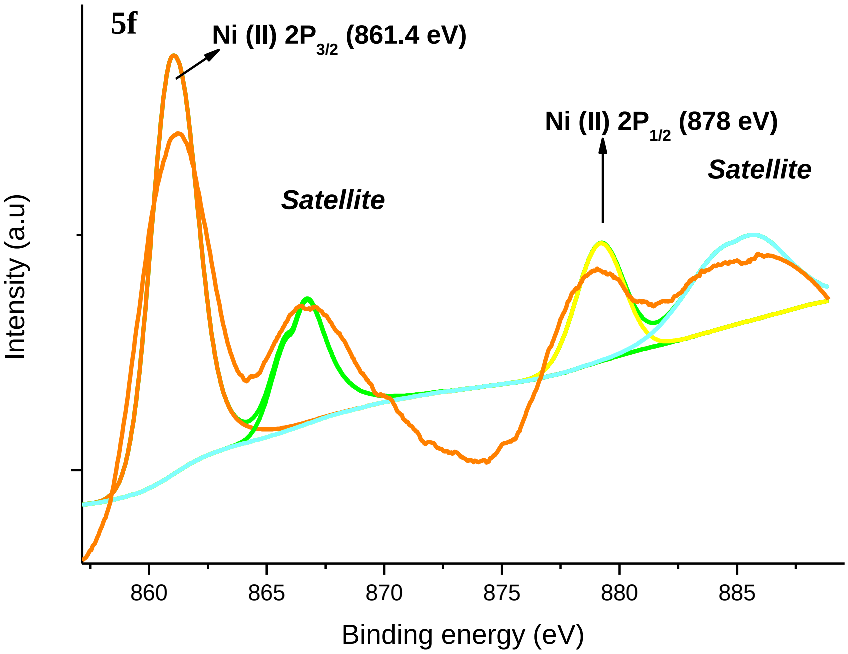


Figure S5. X-ray photoelectron spectra of Ni-C3.

Recyclability of catalyst and TGA studies

For the recyclability of the **Ni-C3** catalyst from the reaction mixture of catalyst (5 mol%), we separated using the vacuum filtration technique. First, toluene (5 mL) was evaporated using a vacuum evaporator and then washed on Whatman qualitative filter paper with hot hexane and ether. The hexane and ether mixture contains unreacted reactants and products. The undissolved **Ni-C3** catalyst remains on filter paper and reused in the reaction after drying the solvents. The **Ni-C3** catalyst remains efficient up to 4 cycles, but the catalyst lossed in work-up (i.e., 100% to less than 4 % remains) (**Figure S6**).

In TGA studies, the **Ni-C3** complex was tested under an air atmosphere with a heating rate of 10 °C per minute. It remained stable up to 290 °C. The complex started decomposing at 90°C, with 95% of the decomposition attributed to solvent and H_2O evaporation, as shown in the graph. The maximum mass loss occurred exothermically in two steps between 290 °C and 475 °C. The **Ni-C3** complex displayed thermal stability up to a maximum of 290 °C, with 87.5% remaining. Subsequently, at 770 °C, the remaining **Ni-C3** complex is expected to decrease to 15%, leading to the formation of its metal oxide NiO_2 (**Figure S6**).

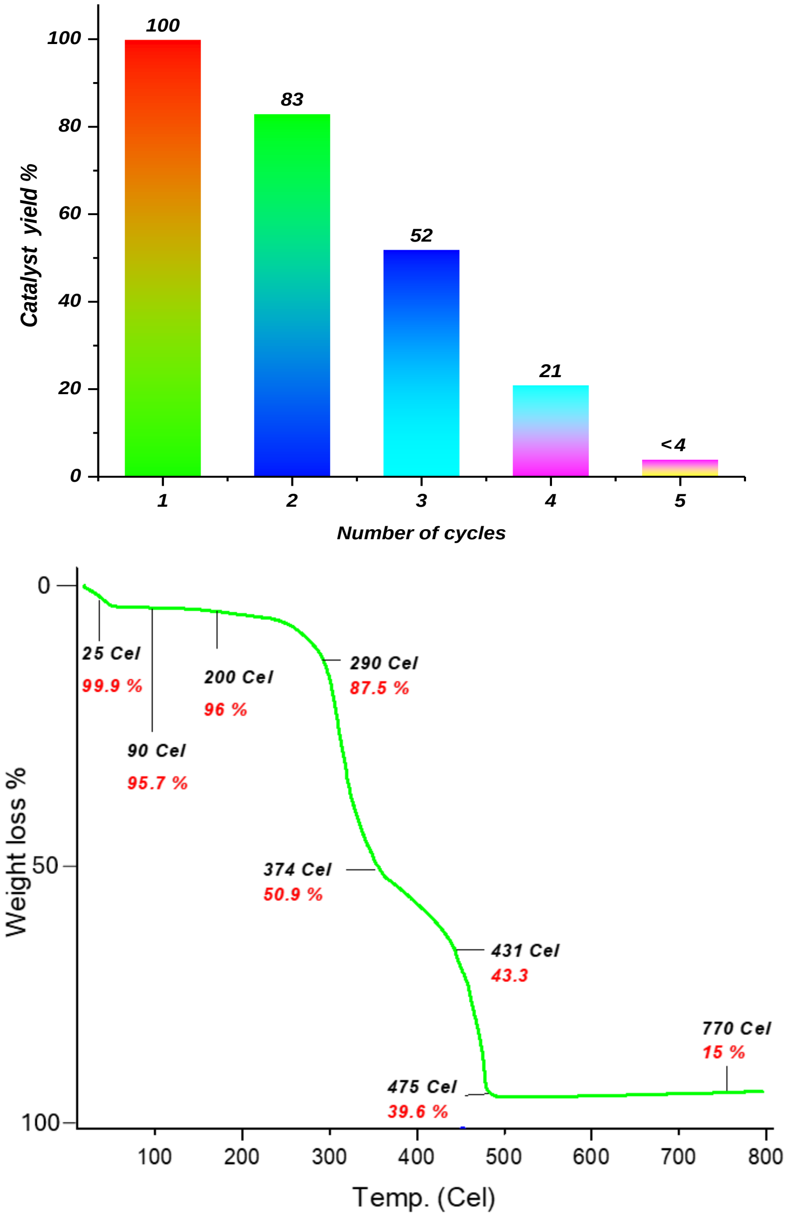


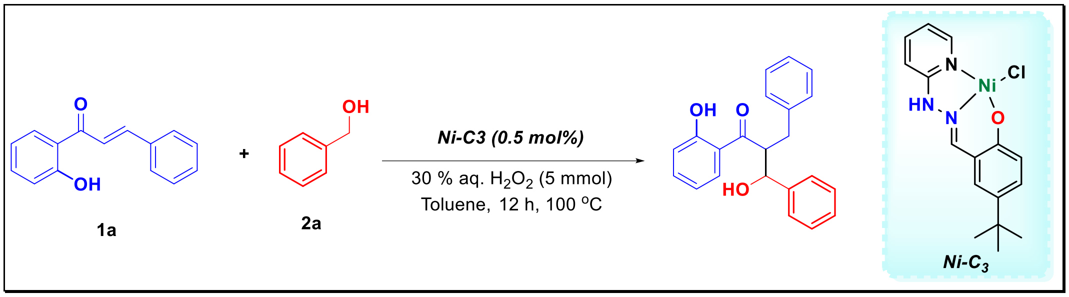
Figure S6. Graphical representation of catalytic re-usage and TGA graph for Ni-C3.

EPR Studies

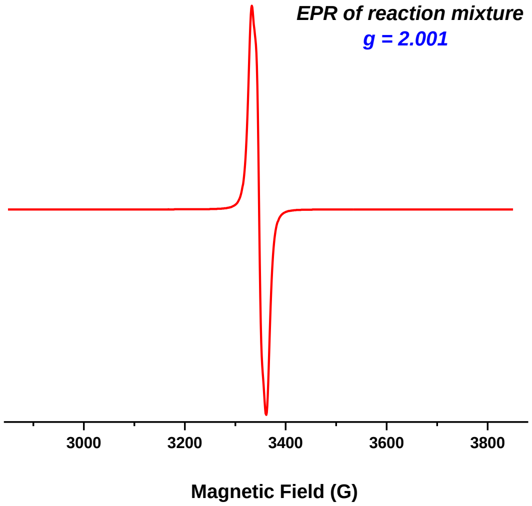
The presence of the radical species was then confirmed using EPR analysis for unfinished reactions. Interestingly, EPR signals were observed with values of **g** = 2.001 for the standard reaction mixture. When the reaction proceeds without substrates **1a** and

2a, the **g** value is 2.004, providing insight into forming carbon-based organic radicals (**Figure S7**). In the reaction containing substrates without the **Ni-C3** catalyst, no EPR signal was observed. However, adding **Ni-C3** under standard reaction conditions detected an EPR active signal. Based on controlled EPR studies, it is evident that the presence of the catalyst is essential for the radical process.

Table S1. Controlled experiment using EPR analysis



Sl. No.	Experiment	EPR activity	g-value
1.	EPR of the reaction mixture (*in-situ* generated radical)	***Active***	2.001
2.	Reaction mixture without H_2O_2	*Inactive*	-
3.	Reaction mixture without **1a and 2a**	***Active***	2.004



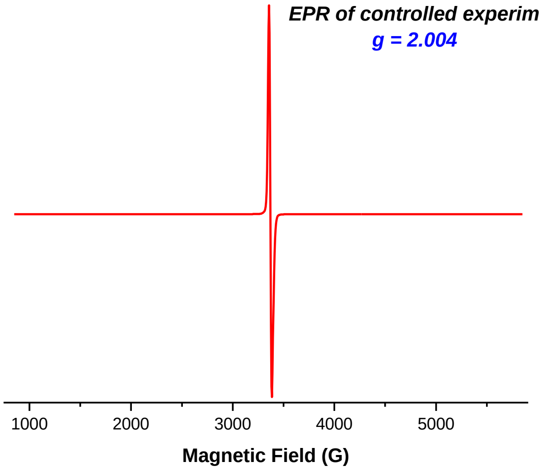
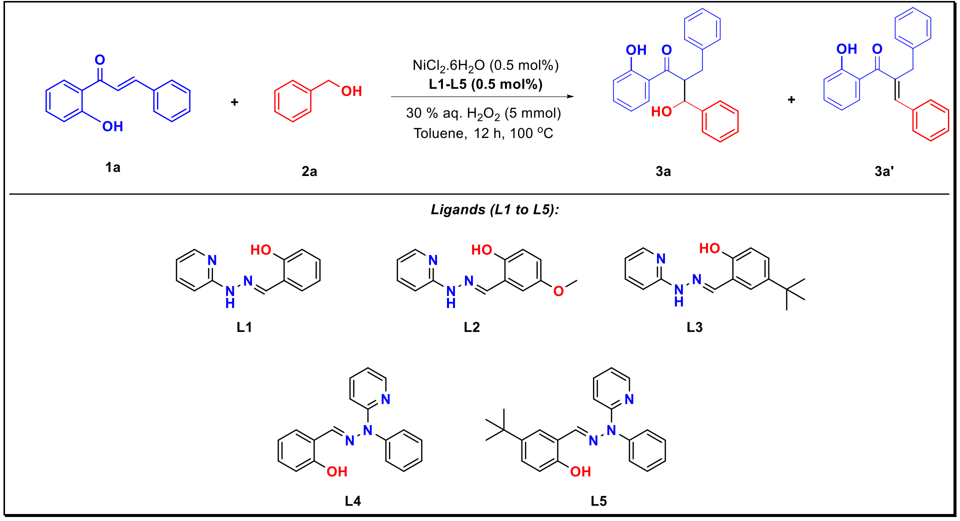


Figure S7. (a) EPR spectra of the reaction mixture containing standard reactants in toluene. (b) EPR spectra of the reaction containing **Ni-C3** catalyst and *30 % aq.* H_2O_2 without substrate chalcone (**1a**) and alcohol (**2a**).

[1.4] Optimization studies:

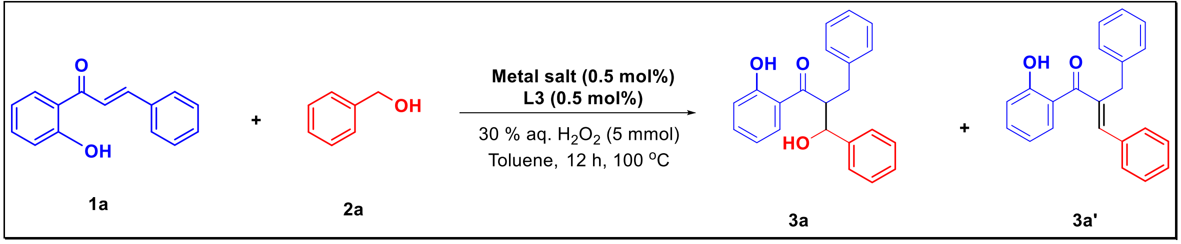
Table S2. Screening of ligands with $NiCl_2.6H_2O^a$



Entry	Metal salt	Yield 3a (%)	3a' (%)
1.	(E)-2-((2-(pyridin-2-yl)hydrazineylidene)methyl)phenol **L1**	31%	-
2.	(E)-4-methoxy-2-((2-(pyridin-2-yl)hydrazineylidene)methyl)phenol **L2**	trace	-
3.	(E)-4-(tert-butyl)-2-((2-(pyridin-2-yl)hydrazineylidene)methyl)phenol **L3**	52%	-
4.	(E)-2-((2-phenyl-2-(pyridin-2-yl)hydrazineylidene)methyl)phenol **L4**	-	-
5.	(E)-4-(tert-butyl)-2-((2-phenyl-2-(pyridin-2-yl)hydrazineylidene)methyl)phenol **L5**	<5%	-

[a] ***Reaction condition:*** *2'*-hydroxychalcone **1a** (1 mmol), benzyl alcohol **2a** (1 mmol), **$NiCl_2.6H_2O$** (0.5 mol%), **L** (0.5 mol%), **30% aq. H_2O_2** (5 mmol), toluene (5 mL), 100 °C oil bath, 12 h reaction time. Yield was determined by column chromatography.

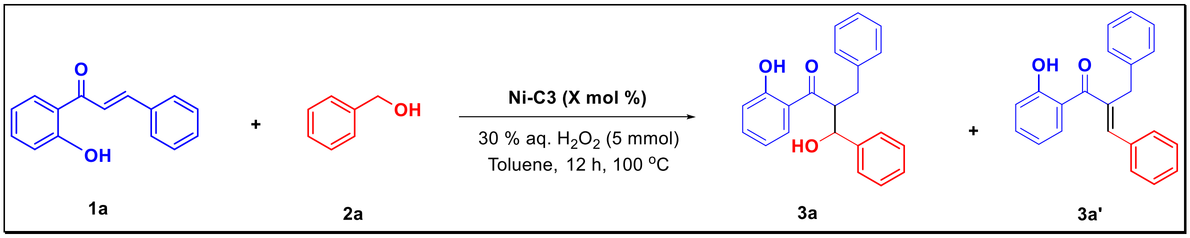
Table S3: Screening of metal salts with L3[a]



Entry	Metal salt	Yield 3a (%)	3a' (%)
1.	$FeCl_3.6H_2O$	trace	-
2.	$Cu(CO_2CH_3)_2$	-	-
3.	CuCl	-	-
4.	$Zn(CO_2CH_3)_2$	-	-
5.	$MnCl_2.4H_2O$	trace	-

[a]***Reaction condition:*** 2'-hydroxychalcone **1a** (1 mmol), benzyl alcohol **2a** (1 mmol), **metal salt** (0.5 mol%), **L3** (0.5 mol%), **30% aq. H_2O_2** (5 mmol), toluene (5 mL), 100 °C oil bath, 12 h reaction time. Yield was determined by column chromatography.

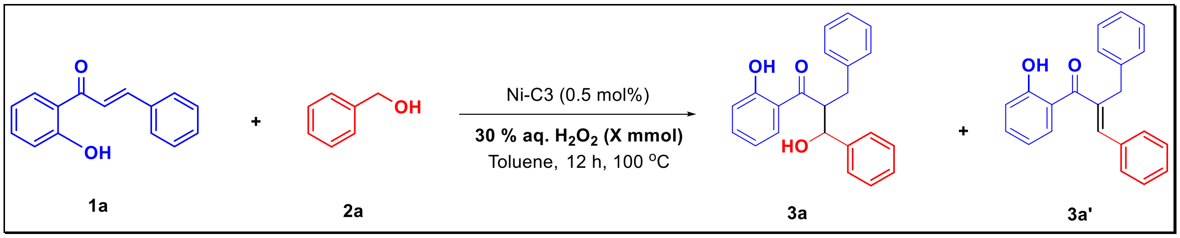
Table S4: Screening of catalyst loading[a]



Entry	Catalyst loading (mol%)	Yield 3a (%)	3a' (%)
1.	No catalyst	-	-
2.	0.025	-	-
3.	0.05	trace	-
4.	0.1	<25	-
5.	0.5	83	<8
6.	0.75	47	trace

[a]***Reaction condition:*** 2'-hydroxychalcone **1a** (1 mmol), benzyl alcohol **2a** (1 mmol), **Ni-C3 (X mol%)**, **30% aq. H_2O_2** (5 mmol), toluene (5 mL), 100 °C oil bath, 12 h reaction time. Yield was determined by column chromatography.

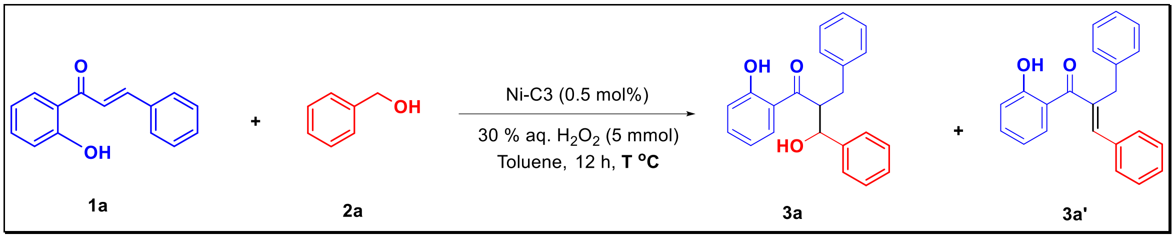
Table S5: Screening of H_2O_2 equivalent[a]



Entry	30 % aq. H_2O_2 (mmol)	Yield 3a (%)	3a' (%)
1.	0.5	-	-
2.	1	-	-
3.	3	-	-
4.	5	83	trace
5.	7	35	-

^{a}***Reaction condition:*** *2'*-hydroxychalcone **1a** (1 mmol), benzyl alcohol **2a** (1 mmol), **Ni-C3** (0.5 mol%), **30% aq. H_2O_2** (X mmol), toluene (5 mL), 100 °C oil bath, 12 h reaction time. Yield was determined by column chromatography.

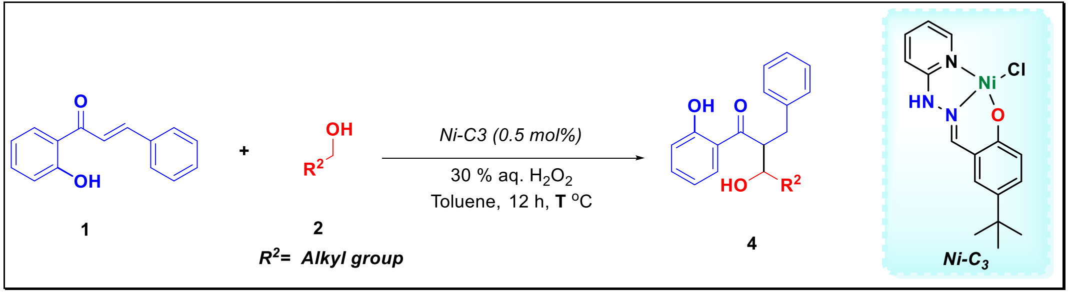
Table S6: Screening of reaction temperature^a



Entry	Reaction temperature	Yield 3a (%)	3a' (%)
1.	**80**	**--**	**--**
2.	90	32	--
3.	**100**	**83**	Trace
4.	110	26	15

^{a}***Reaction condition:*** *2'*-hydroxychalcone **1a** (0.5 mmol), benzyl alcohol **2a** (0.5 mmol), **Ni-C3** (0.25 mol%), **30% aq. H_2O_2** (2.5 mmol), toluene (2.5 mL), **T °C** oil bath, 12 h reaction time. Yield was determined by column chromatography.

Table S7: Screening of temperature in nickel catalyzed aldol reaction of α, β-unsaturated ketones and aliphatic alcohols^a



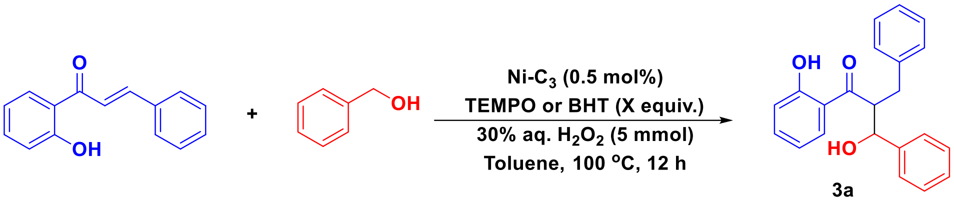
Entry	Aliphatic alcohol (substrate)	B.Pt. (°C)	4(%) @ B.Pt.	4 (%) @100 °C
1.	Methanol	60	25	28
2.	Ethanol	80	51	56
3.	Propan-2-ol	80	54	60
4.	Butan-2-ol	100	67	67
5.	Pentan-2-ol	140	62	71

^{a}***Reaction condition:*** *2'*-hydroxychalcone **1** (0.5 mmol), aliphatic alcohol **2** (0.5 mmol), **Ni-C3** (0.25 mol%), **30% aq. H_2O_2** (2.5 mmol), toluene (2.5 mL), **T °C** oil bath, 12 h reaction time. Yield was determined by column chromatography.igure S6

[1.5] Radical quenching experiment:

To trap in-situ generated radicals, the reaction mixtures with 1 equiv. of BHT or TEMPO with benzyl alcohol in standard reaction conditions were analyzed instantly after the completion of the reaction in a high-resolution mass spectrometer (**Figure S8**). In the case of BHT, HRMS spectra have shown mass (m/z) = 327.1360 which corresponds to the calculated mass value [M+H$^+$] (m/z) = 327.1360 of BHT adduct of benzyl alcohol

Scheme S2: Radical quenching and radical trapping experiment



	TEMPO	*BHT*
	3a	**3a**
0.5 equiv.	25%	8%
1.0 equiv.	0%	0%

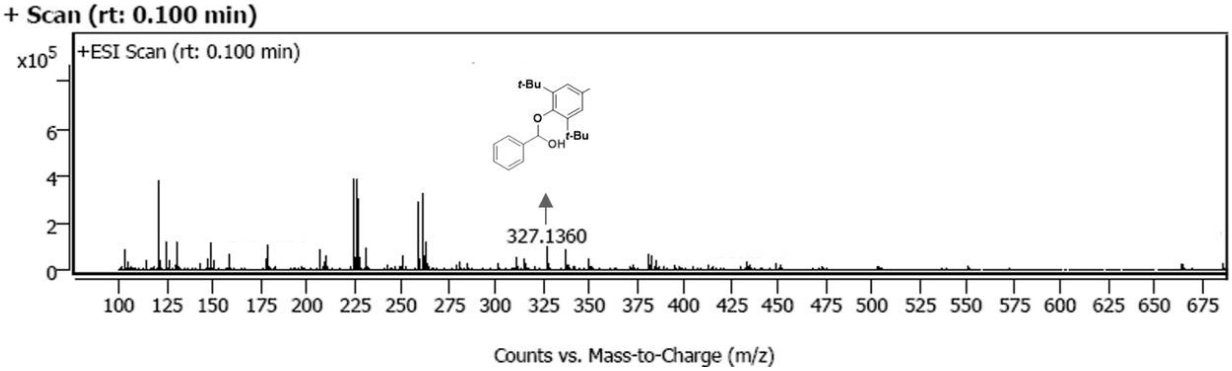
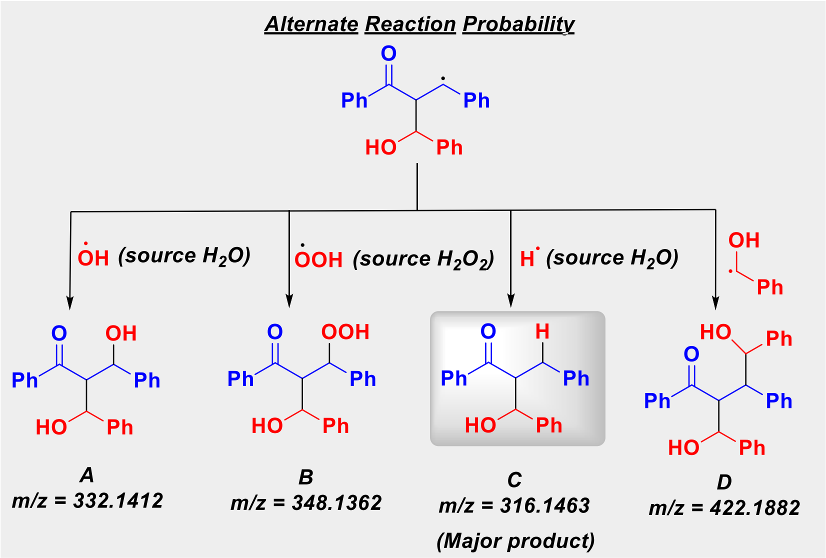


Figure S8. BHT adduct formed with benzyl alcohol substrate in reaction after 12 h of reaction completion.

[1.6] HRMS data, GC-MS spectra of product in the reaction mixture

Scheme S3: Mechanistic validation: HRMS data and GC-MS spectra



The HRMS data for the reaction between substrate 1 and 2 in the presence of 0.5 mol % of **Ni-C3** and 5 mmol of *aq. 30%* H_2O_2 were studied. After the reaction is completed, the solvent is evaporated, and the catalyst is recovered by washing the mixture with hot hexane. The synthesised organic product gets dissolved in hexane, which is later dried and proceeded further for HRMS study. In the HRMS spectra, we observed peaks corresponding to potential products. The peak at an *m/z* value of 316.1463 display the major product formed in the reaction. While, the possible other products represented accordingly with their *m/z* values. It validates the trace amount of side product formed by the radical formed in the system, which produced by the quenching of the phenyl radical formed (**Figure S9**).

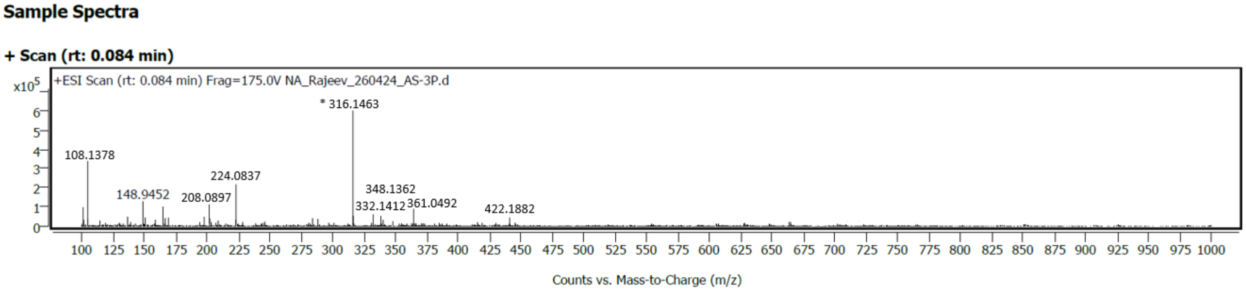
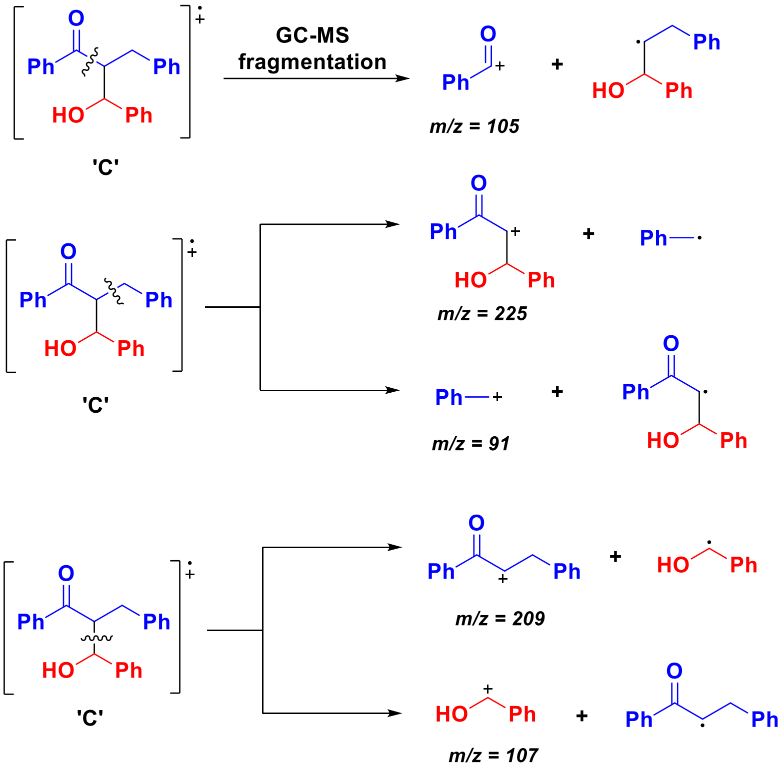


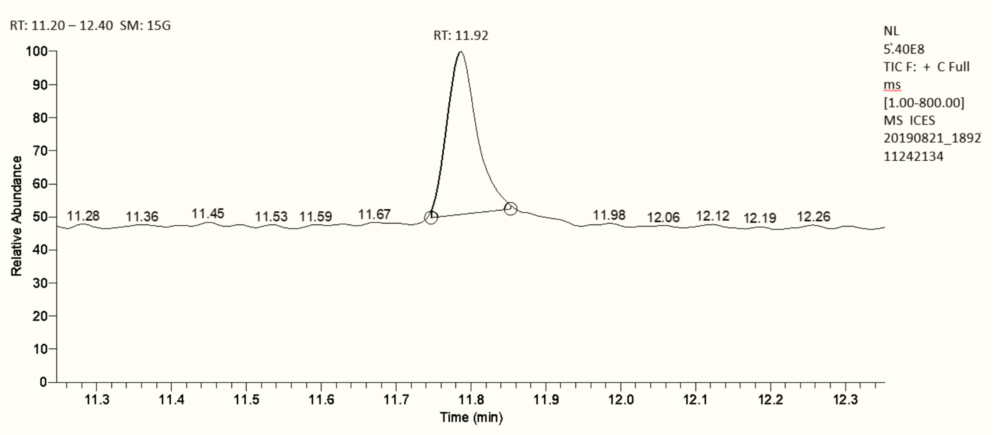
Figure S9. HRMS spectra of the reaction mixture.

GC-MS of Product 'C':

The fragmentation pattern of the mass spectrum of the product '**C**' is represented here. The molecular ions for product '**C**' are energetically unstable, and some of them will break up into smaller pieces. The simplest scenario occurs when a molecular ion divides into two parts - one of which becomes another positive ion, and the other becomes an uncharged free radical.



Scheme S4.The fragmentation pattern observed while proceeding with the GC-MS of the extracted product '**C**'. With a retention time of 11.92 minutes, we observed different base peaks at *m/z* values of 105, 225, 91, 209, and 107, respectively. Other peaks with *m/z* values 1 or 2 less often result from the loss of one or more hydrogen atoms during the fragmentation process.



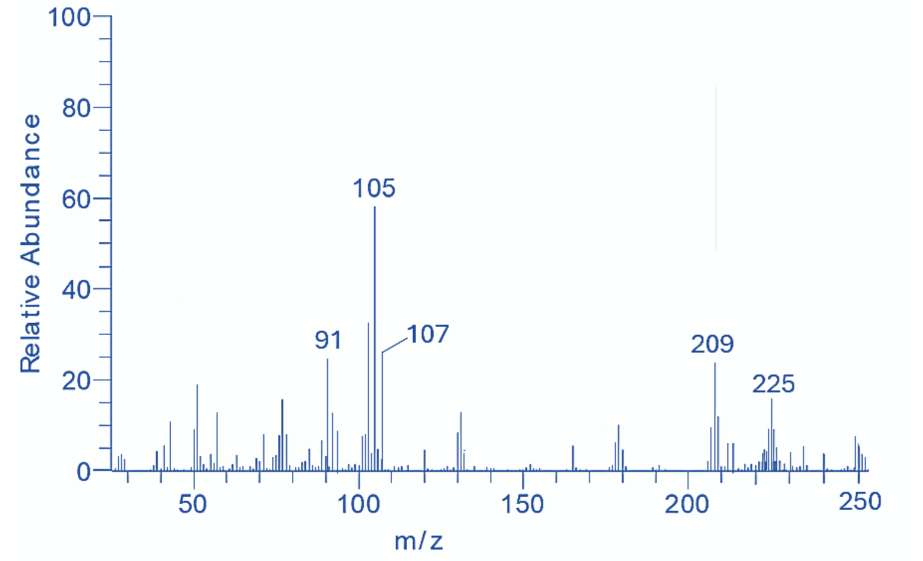
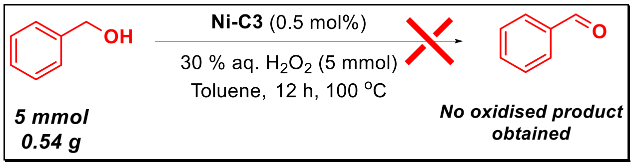


Figure S10. GC-MS spectra for the synthesised product ‘**C**’.

[1.7] NMR of reaction containing benzyl alcohol substrate



To ensure the reaction mechanism, whether alcohol is oxidizing to the aldehyde and or the chalcone hydrogenated to the saturated ketone by the nickel catalyst. A reaction containing 5 mmol of benzyl alcohol is performed which ensure that oxidation of benzyl alcohol to aldehyde doesn’t take place confirmed by ^{1}HNMR (**Figure S11**). Similarly, using chalcone and catalyst failed to give saturated ketones after ^{1}HNMR analysis.

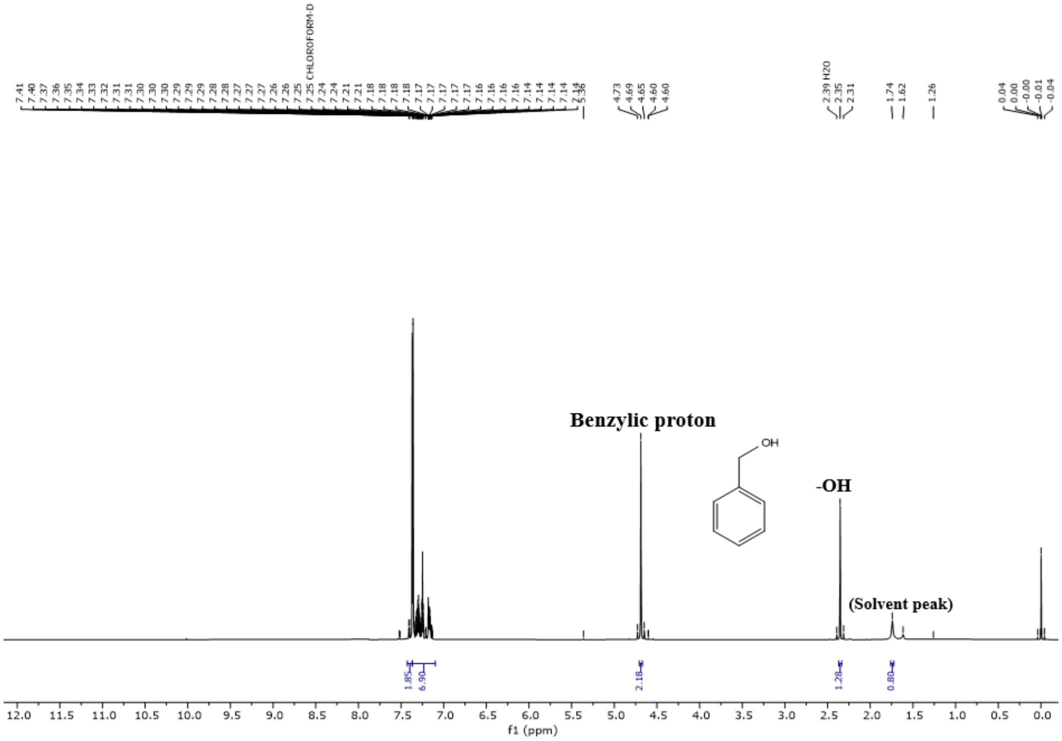
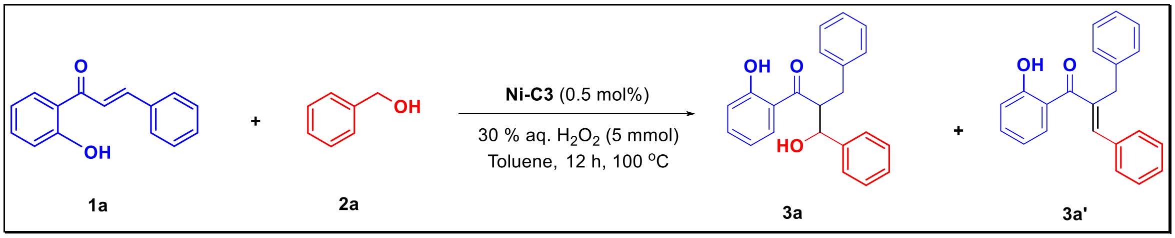


Figure S11. ^{1}HNMR analysis of reaction mixture.

[1.8] IR spectra of compound 3a and 3a’



IR data for the **3a** and **3a’** products synthesised explain the formation of α-alkyl alcohol substituted product. Stretching frequency due to the carbonyl group in **3a** is 1743 cm^{-1}, which is decreased by the condensed product formation, i.e., **3a’** to 1638 cm^{-1}, which validates the formation of a conjugated enone system (**Figure S12**).

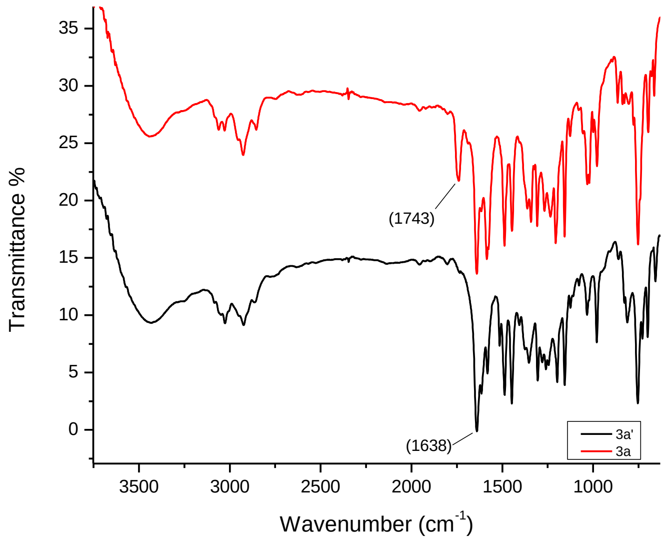
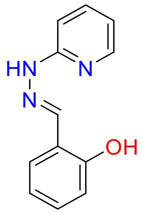


Figure S12. IR spectra for the product **3a** and **3a**’.

[1.9] Analytical data:

(E)-2-((2-(pyridin-2-yl)hydrazineylidene)methyl)phenol (L$_1$):

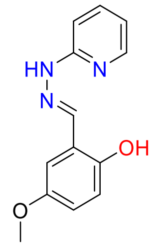


85% yield (0.181 g), white solid, **mp** = 114 ºC -115 ºC, **Rf** = 0.58 EtOAc:Hexane (1:9). **IR** spectra of the ligands show a sharp band at 1601 cm^{-1} due to ν(C=N) stretch, and 1090 cm^{-1} due to ν(C–O)stretch.

^{1}H NMR (500 MHz, CDCl$_3$) δ 11.79 (s, 1H), 8.73 (s, 1H), 8.25 – 8.23 (dd, *J* = 4.3, 1.7 Hz, 1H), 8.08 (s, 1H), 7.66 (td, *J* = 6.9, 1.7 Hz, 1H), 7.48 (dd, *J* = 7.4, 1.3 Hz, 1H), 7.31 (dd, *J* = 6.9, 1.5 Hz, 1H), 7.66 (ddd, *J* = 8.5, 7.5, 1.2 Hz, 1H), 6.89 – 6.83 (m, 3H).

^{13}C NMR (126 MHz, CDCl$_3$) δ 161.88, 152.83, 147.45, 142.95, 134.69, 134.45, 124.24, 123.73, 121.17, 120.37, 110.93. **Calcd (%)** for C$_{12}$H$_{11}$N$_3$O: C 67.59, H 5.20, N 19.71; Found: C 67.80, H 5.99, N 19.15.

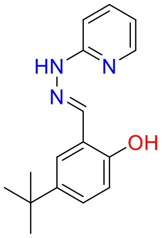
a. 4-methoxy-2-((2-(pyridin-2-yl)hydrazineylidene)methyl)phenol (L$_2$):



78% yield (0.189 g), white solid, **mp** = 142 ºC -143 ºC **Rf** = 0.65 EtOAc:Hexane (1:9). **IR** spectra of the ligands show a sharp band at 1630 cm^{-1} due to v(C=N) stretch, 1098 cm^{-1} due to v(C–O)stretch.

^{1}H NMR (500 MHz, CDCl$_3$) δ 10.31 (s, 1H), 8.16 (ddd, *J* = 5.0, 1.9, 0.9 Hz, 1H), 7.88 (s, 1H), 7.64 (ddd, *J* = 8.8, 7.2, 1.9 Hz, 1H), 7.07 (dd, *J* = 8.4, 1.0 Hz, 1H), 6.92 (d, *J* = 8.9 Hz, 1H), 6.87 – 6.79 (m, 3H), 6.69 (d, *J* = 3.0 Hz, 1H), 3.77 (s, 3H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 155.84, 152.75, 151.40, 147.56, 142.62, 138.85, 118.35, 117.40, 116.82, 116.43, 114.11, 106.84, 55.99. **Calcd (%)** for $C_{13}H_{13}N_3O_2$: C 64.19, H 5.39, N 17.27; Found: C 64.86, H 5.90, N 17.05.

b. 4-*tert*-butyl-2-((2-(pyridin-2-yl)hydrazineylidene)methyl)phenol (L$_3$):

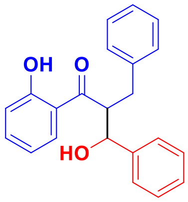


88% yield (0.237 g), white solid, **mp** = 148 ºC -149 ºC, **Rf** = 0.62 EtOAc:Hexane (1:9). **IR** spectra of the ligands show a sharp band at 1602 cm^{-1} due to v(C=N) stretch, 1088 cm^{-1} due to v(C–O)stretch.

^{1}H NMR (500 MHz, CDCl$_3$ δ 10.57 (s, 1H), 9.20 (s, 1H), 8.18 (ddd, *J* = 5.1, 1.9, 0.9 Hz, 1H), 7.95 (s, 1H), 7.64 (ddd, *J* = 8.4, 7.2, 1.8 Hz, 1H), 7.29 (dd, *J* = 8.6, 2.4 Hz, 1H), 7.15 (d, *J* = 2.4 Hz, 1H), 7.07 (dd, *J* = 8.4, 1.0 Hz, 1H), 6.94 (d, *J* = 8.6 Hz, 1H), 6.83 (ddd, *J* = 7.3, 4.9, 1.0 Hz, 1H), 1.30 (s, 9H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 155.91, 155.01, 147.69, 143.54, 142.40, 138.80, 127.91, 126.52, 117.59, 116.32, 106.80, 34.12, 31.55. **Calcd (%)** for $C_{16}H_{19}N_3O$: C 71.35, H 7.11, N 15.60; Found: C 71.85, H 7.93, N 15.23.

Analytical data for Scheme 3:

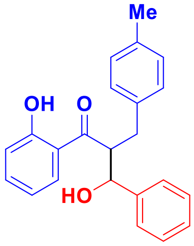
2-benzyl-3-hydroxy-1-(2-hydroxyphenyl)-3-phenylpropan-1-one (3a):



Yellow oil, fruity odour, yield 83 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.89 (s, 1H), 7.76 (dd, J = 8.0, 1.5 Hz, 1H), 7.46 (td, J = 8.2, 1.6 Hz, 1H), 7.39 – 7.27 (m, 9H), 7.27 – 7.21 (m, 1H), 6.98 (dd, J = 8.3, 1.4 Hz, 1H), 6.93 (td, J = 8.0, 1.3 Hz, 1H), 5.18 (ddt, J = 8.3, 5.4, 1.0 Hz, 1H), 3.75 – 3.66 (m, 1H), 3.58 (dd, J = 16.5, 8.2 Hz, 1H), 3.49 (dd, J = 16.4, 7.8 Hz, 1H), 3.10 (d, J = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 205.52, 162.60, 145.60, 140.88, 136.57, 136.48, 129.98, 128.84, 128.76, 128.72, 128.55, 128.40, 126.47, 120.24, 119.41, 119.07, 119.01, 118.77, 118.68, 66.44, 40.15, 30.11. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{22}H_{20}O_3]$ 332.1412; Found 332.1463.

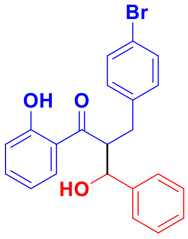
3-hydroxy-1-(2-hydroxyphenyl)-2-(4-methylbenzyl)-3-phenylpropan-1-one (3b):



Yellow oil, fruity odour, yield 90 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.89 (s, 1H), 7.76 (dd, J = 8.1, 1.5 Hz, 1H), 7.46 (td, J = 8.2, 1.6 Hz, 1H), 7.36 (ddd, J = 7.7, 2.5, 1.0 Hz, 2H), 7.34 – 7.30 (m, 2H), 7.30 – 7.27 (m, 1H), 7.19 – 7.13 (m, 2H), 7.12 – 7.07 (m, 2H), 6.98 (dd, J = 8.3, 1.4 Hz, 1H), 6.93 (td, J = 8.0, 1.3 Hz, 1H), 5.16 (ddt, J = 8.5, 5.4, 1.1 Hz, 1H), 3.77 – 3.68 (m, 1H), 3.58 (dd, J = 16.4, 8.1 Hz, 1H), 3.47 (dd, J = 16.5, 7.9 Hz, 1H), 3.10 (d, J = 5.4 Hz, 1H), 2.33 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 206.81, 166.80, 148.57, 143.56, 140.83, 140.81, 135.25, 134.66, 133.43, 133.05, 132.75, 132.23, 126.86, 125.51, 121.33, 81.98, 53.31, 46.67, 26.08. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{23}H_{22}O_3]$ 346.1569; Found 346.1571.

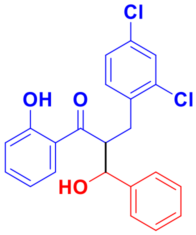
2-(4-bromobenzyl)-3-hydroxy-1-(2-hydroxyphenyl)-3-phenylpropan-1-one (3c):



Yellow oil, fruity odour, yield 78 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.84 (s, 1H), 7.72 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.42 (td, *J* = 8.2, 1.6 Hz, 1H), 7.38 – 7.33 (m, 2H), 7.33 – 7.24 (m, 6H), 7.24 (t, *J* = 1.1 Hz, 1H), 6.94 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.89 (td, *J* = 8.0, 1.3 Hz, 1H), 5.12 (ddt, *J* = 8.5, 5.4, 1.1 Hz, 1H), 3.73 – 3.64 (m, 1H), 3.54 (dd, *J* = 16.4, 8.1 Hz, 1H), 3.45 (dd, *J* = 16.5, 7.9 Hz, 1H), 3.06 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 205.68, 162.63, 140.91, 137.81, 136.50, 136.47, 135.97, 130.02, 129.49, 129.46, 128.78, 128.62, 128.58, 128.45, 126.49, 119.43, 119.09, 118.69, 40.33, 30.11, 29.92, 21.20. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{22}H_{19}BrO_3]$ 410.0518; Found 410.0523.

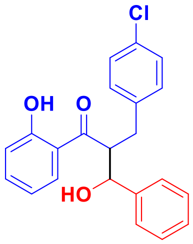
2-(2,4-dichlorobenzyl)-3-hydroxy-1-(2-hydroxyphenyl)-3-phenylpropan-1-one (3d):



Yellow oil, fruity odour, yield 63 %

^{1}H NMR (500 MHz $CDCl_3$) δ 12.31 (s, 1H), 7.72 (dt, *J* = 8.1, 1.9 Hz, 1H), 7.45 (td, *J* = 8.7, 1.6 Hz, 1H), 7.38 (d, *J* = 2.1 Hz, 1H), 7.36 - 7.22 (m, 6H), 7.18 – 7.15 (t, J = 1.1 Hz ,1H), 6.97 (dd, *J* = 8.4, 1.2 Hz, 1H), 6.86 (td, *J* = 8.2, 1.2 Hz, 1H), 5.16 (dd, J = 8.5, 1.1 Hz, 1H), 3.31 - 3.13 (dd, *J* = 8.2, 6.9 Hz, 2H), 3.06 (dd, *J* = 8.4, 7.0 Hz, 2H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 196.50, 156.79, 138.07, 131.65, 130.82, 129.78, 128.57, 126.40, 125.24, 125.11, 123.42, 123.04, 122.59, 122.22, 116.84, 115.50, 111.32, 71.66, 38.43, 36.79. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{22}H_{18}Cl_2O_3]$ 400.0633; Found 346.0640.

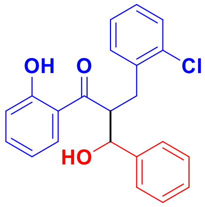
2-(4-chlorobenzyl)-3-hydroxy-1-(2-hydroxyphenyl)-3-phenylpropan-1-one (3e):



Yellow oil, fruity odour, yield 72 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.84 (s, 1H), 7.72 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.42 (td, *J* = 8.2, 1.6 Hz, 1H), 7.38 – 7.33 (m, 2H), 7.33 – 7.24 (m, 6H), 7.23 (t, *J* = 1.1 Hz, 1H), 6.94 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.89 (td, *J* = 8.0, 1.3 Hz, 1H), 5.12 (ddt, *J* = 8.5, 5.4, 1.1 Hz, 1H), 3.73 – 3.64 (m, 1H), 3.54 (dd, *J* = 16.4, 8.1 Hz, 1H), 3.45 (dd, *J* = 16.5, 7.9 Hz, 1H), 3.05 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 207.80, 167.80, 149.56, 145.14, 141.82, 140.93, 136.24, 135.32, 135.03, 134.43, 134.04, 133.22, 127.86, 126.50, 122.32, 53.98, 47.65. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{22}H_{19}ClO_3]$ 366.1023; Found 366.1051.

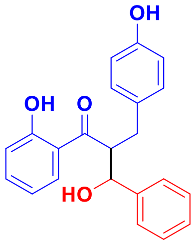
2-(2-chlorobenzyl)-3-hydroxy-1-(2-hydroxyphenyl)-3-phenylpropan-1-one (3f):



Yellow oil, fruity odour, yield 70 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.85 (s, 1H), 7.74 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.48 (td, *J* = 8.2, 1.6 Hz, 1H), 7.43 – 7.39 (m, 1H), 7.39 – 7.25 (m, 9H), 6.95 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.90 (td, *J* = 8.0, 1.3 Hz, 1H), 5.12 (ddt, *J* = 8.8, 5.4, 1.1 Hz, 1H), 3.75 – 3.62 (m, 2H), 3.56 (dd, *J* = 16.3, 7.9 Hz, 1H), 2.83 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 197.73, 158.02, 139.30, 133.77, 132.05, 130.45, 126.47, 126.19, 124.65, 124.56, 124.27, 123.96, 123.45, 118.07, 116.73, 112.55, 72.90, 40.01, 38.04. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{22}H_{19}ClO_3]$ 366.1023; Found 366.1051.

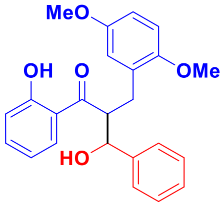
3-hydroxy-2-(4-hydroxybenzyl)-1-(2-hydroxyphenyl)-3-phenylpropan-1-one (3g):



Yellow oil, fruity odour, yield 83 %

^{1}H NMR (500 MHz $CDCl_3$) δ 12.82 (s, 1H), 7.98 – 7.89 (m, 2H), 7.74 – 7.44 (m, 3H), 7.44 – 7.41 (m, 2H), 7.35 – 7.18 (m, 2 H), 7.06 – 6.91 (m, 2H), 6.87 (ddd, J = 8.3, 7.1, 1.2 Hz, 1H), 3.33 (dd, J = 8.5, 7.0 Hz, 2H), 3.07 (dd, J = 8.5, 7.0 Hz, 2H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 201.80, 161.79, 156.24, 143.56, 135.82, 134.98, 130.24, 129.26, 128.42, 128.04, 127.22, 121.85, 120.50, 116.32, 115.55, 48.26, 41.68. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for $[C_{22}H_{20}O_4]$ 348.1362; Found 348.1564.

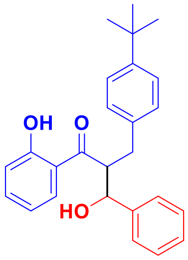
2-(2,5-dimethoxybenzyl)-3-hydroxy-1-(2-hydroxyphenyl)-3-phenylpropan-1-one (3h):



Yellow oil, fruity odour, yield 80 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.86 (s, 1H), 7.74 (dd, J = 8.1, 1.5 Hz, 1H), 7.44 (td, J = 8.2, 1.6 Hz, 1H), 7.36 – 7.30 (m, 2H), 7.30 – 7.24 (m, 3H), 6.96 (dd, J = 8.3, 1.4 Hz, 1H), 6.91 (td, J = 8.0, 1.3 Hz, 1H), 6.85 – 6.79 (m, 2H), 6.74 (dd, J = 8.5, 2.2 Hz, 1H), 5.21 (ddt, J = 8.5, 5.1, 1.0 Hz, 1H), 3.79 (s, 3H), 3.77 (s, 3H), 3.74 – 3.61 (m, 2H), 3.57 (dd, J = 16.1, 7.8 Hz, 1H), 2.87 (d, J = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 197.85, 158.16, 151.43, 147.85, 139.45, 132.18, 127.30, 126.60, 124.80, 124.40, 123.59, 118.33, 116.86, 112.68, 111.45, 110.37, 109.51, 73.00, 51.98, 37.92, 37.88. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for $[C_{24}H_{24}O_5]$ 392.1624; Found 392.1627.

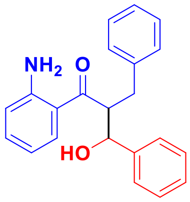
2-(4-(tert-butyl)benzyl)-3-hydroxy-1-(2-hydroxyphenyl)-3-phenylpropan-1-one (3i):



Yellow oil, fruity odour, yield 95 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.89 (s, 1H), 7.78 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.46 - 7.43 (td, *J* = 8.2, 1.6 Hz, 1H), 7.35 (ddd, *J* = 7.6, 2.5, 1.0 Hz, 2H), 7.34 – 7.29 (m, 3H), 7.29 – 7.22 (m, 2H), 7.18 (dd, *J* = 7.7, 1.0 Hz, 2H), 6.93 (td, *J* = 8.0, 1.3 Hz, 1H), 5.16 (ddt, *J* = 8.5, 5.4, 1.1 Hz, 1H), 3.77 – 3.68 (m, 1H), 3.58 (dd, *J* = 16.4, 8.1 Hz, 1H), 3.48 (dd, *J* = 16.5, 7.9 Hz, 1H), 3.11 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 201.80, 161.79, 149.81, 143.56, 139.81, 135.82, 130.24, 128.42, 128.37, 128.04, 127.22, 125.56, 121.85, 120.50, 116.32, 76.97, 48.35, 41.67, 34.72, 31.17. **HRMS** (ESI-TOF) m/z: [M]$^{+}$ Calcd for [$C_{26}H_{28}O_3$] 388.2038; Found 388.2050.

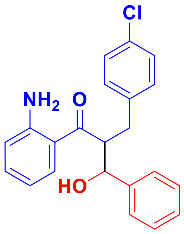
1-(2-aminophenyl)-2-benzyl-3-hydroxy-3-phenylpropan-1-one (3j):



Yellow oil, fruity odour, yield 74 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.72 (dd, *J* = 7.7, 1.6 Hz, 2H), 7.39 – 7.20 (m, 22H), 6.76 – 6.69 (m, 3H), 6.69 (d, *J* = 1.3 Hz, 1H), 6.15 (s, 4H), 5.18 (ddt, *J* = 8.3, 5.4, 1.0 Hz, 2H), 3.75 – 3.67 (m, 2H), 3.66 – 3.57 (m, 2H), 3.51 (dd, *J* = 16.6, 7.8 Hz, 2H), 3.10 (d, *J* = 5.4 Hz, 2H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 198.32, 146.29, 139.18, 136.04, 130.45, 126.12, 124.66, 124.04, 123.66, 123.30, 122.84, 122.32, 117.23, 113.55, 112.67, 72.60, 43.86, 36.95. **HRMS** (ESI-TOF) m/z: [M]$^{+}$ Calcd for [$C_{22}H_{21}NO_2$] 331.1572; Found 331.1601.

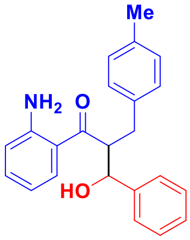
1-(2-aminophenyl)-2-(4-chlorobenzyl)-3-hydroxy-3-phenylpropan-1-one (3k):



Yellow oil, fruity odour, yield 61 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.71 (ddd, *J* = 11.2, 8.2, 1.5 Hz, 2H), 7.34 – 7.14 (m, 9H), 6.67 – 6.61 (m, 3H), 6.27 (s, 1H), 3.31 – 3.19 (m, 3H), 3.02 (ddd, *J* = 15.5, 8.5, 6.9 Hz, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 201.66, 201.18, 150.47, 141.60, 140.05, 134.48, 134.39, 131.87, 131.14, 131.04, 129.90, 128.66, 128.61, 128.52, 126.17, 117.91, 117.82, 117.51, 117.48, 115.92, 41.08, 40.73, 30.67, 29.90. **HRMS** (ESI-TOF) m/z: [M]$^{+}$ Calcd for [$C_{22}H_{21}ClNO_2$] 365.1183; Found 365.1187.

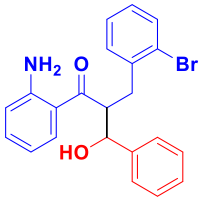
1-(2-aminophenyl)-3-hydroxy-2-(4-methylbenzyl)-3-phenylpropan-1-one (3l):



Yellow oil, fruity odour, yield 79 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.72 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.39 – 7.28 (m, 5H), 7.25 (td, *J* = 7.7, 1.5 Hz, 1H), 7.19 – 7.13 (m, 2H), 7.12 – 7.07 (m, 2H), 6.76 – 6.69 (m, 1H), 6.15 (s, 2H), 5.18 (ddt, *J* = 8.3, 5.4, 1.0 Hz, 1H), 3.74 – 3.65 (m, 1H), 3.62 (dd, *J* = 16.5, 8.1 Hz, 1H), 3.51 (dd, *J* = 16.6, 7.8 Hz, 1H), 3.10 (d, *J* = 5.4 Hz, 1H), 2.33 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 197.62, 145.59, 138.48, 133.41, 130.73, 129.75, 125.42, 124.57, 123.34, 122.96, 122.72, 122.14, 116.53, 112.85, 111.97, 71.89, 43.16, 36.25, 15.99. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{23}H_{23}NO_2]$ 345.1729; Found 345.1738.

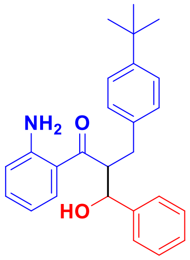
1-(2-aminophenyl)-2-(2-bromobenzyl)-3-hydroxy-3-phenylpropan-1-one (3m):



Yellow oil, fruity odour, yield 67 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.75 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.50 (ddd, *J* = 8.3, 5.7, 2.7 Hz, 1H), 7.45 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.42 – 7.24 (m, 9H), 6.76 (td, *J* = 7.6, 1.2 Hz, 1H), 6.72 (dd, *J* = 7.8, 1.3 Hz, 1H), 6.18 (s, 2H), 5.29 (ddt, *J* = 8.5, 5.4, 1.0 Hz, 1H), 3.82 (qd, *J* = 8.2, 1.0 Hz, 1H), 3.69 (dd, *J* = 16.2, 8.2 Hz, 1H), 3.60 (dd, *J* = 16.2, 7.9 Hz, 1H), 2.88 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 202.45, 150.67, 143.06, 139.22, 134.83, 132.87, 130.50, 130.18, 129.26 (d, *J* = 7.2 Hz), 128.42, 128.04, 127.22, 124.80, 121.61, 117.93, 117.05, 76.59, 45.99, 41.18. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{22}H_{20}BrNO_2]$ 409.677; Found 409.0681.

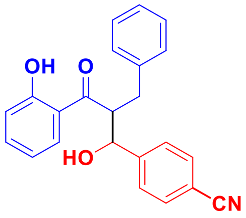
1-(2-aminophenyl)-2-(4-(tert-butyl)benzyl)-3-hydroxy-3-phenylpropan-1-one (3n):



Yellow oil, fruity odour, yield 84 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.72 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.36 (ddd, *J* = 5.1, 2.7, 0.9 Hz, 1H), 7.35 – 7.28 (m, 4H), 7.28 – 7.21 (m, 3H), 7.21 – 7.16 (m, 2H), 6.76 – 6.69 (m, 1H), 6.15 (s, 2H), 5.18 (ddt, *J* = 8.3, 5.4, 1.0 Hz, 1H), 3.75 – 3.67 (m, 1H), 3.66 – 3.57 (m, 1H), 3.51 (dd, *J* = 16.6, 7.8 Hz, 1H), 3.10 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 196.63, 156.62, 144.63, 138.38, 134.64, 130.65, 125.07, 123.25, 123.19, 122.86, 122.05, 120.39, 116.68, 115.33, 111.15, 71.80, 43.18, 36.49, 29.54, 26.00. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{26}H_{29}NO_2]$ 387.2198; Found 387.2203.

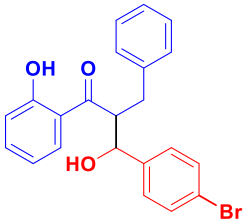
4-(2-benzyl-1-hydroxy-3-(2-hydroxyphenyl)-3-oxopropyl)benzonitrile (3o):



Yellow oil, fruity odour, yield 42 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.89 (s, 1H), 7.76 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.65 – 7.59 (m, 2H), 7.46 (td, *J* = 8.2, 1.6 Hz, 1H), 7.38 – 7.32 (m, 2H), 7.30 (d, *J* = 4.8 Hz, 4H), 7.27 – 7.20 (m, 1H), 6.98 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.93 (td, *J* = 8.0, 1.3 Hz, 1H), 5.21 (ddt, *J* = 8.5, 5.4, 1.0 Hz, 1H), 3.75 – 3.66 (m, 1H), 3.58 (dd, *J* = 16.5, 8.2 Hz, 1H), 3.49 (dd, *J* = 16.4, 7.8 Hz, 1H), 3.05 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 206.51, 166.51, 149.39, 145.21, 140.54, 137.23, 134.95, 133.75, 132.66, 132.40, 131.42, 126.57, 125.21, 122.82, 121.03, 115.95, 82.34, 53.13, 46.38. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for [$C_{23}H_{19}NO_3$] 357.1365; Found 357.1373.

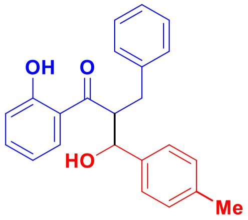
2-benzyl-3-(4-bromophenyl)-3-hydroxy-1-(2-hydroxyphenyl)propan-1-one (3p):



Yellow oil, fruity odour, yield 55 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.89 (s, 1H), 7.76 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.52 – 7.47 (m, 2H), 7.46 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.33 – 7.26 (m, 6H), 7.26 – 7.20 (m, 1H), 6.98 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.93 (td, *J* = 8.0, 1.3 Hz, 1H), 5.21 (ddt, *J* = 8.5, 5.4, 1.0 Hz, 1H), 3.75 – 3.66 (m, 1H), 3.58 (dd, *J* = 16.5, 8.2 Hz, 1H), 3.49 (dd, *J* = 16.4, 7.8 Hz, 1H), 3.10 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 195.74, 155.74, 135.40, 134.44, 129.77, 125.55, 124.19, 122.99, 122.61, 121.63, 120.65, 115.80, 115.36, 114.45, 110.27, 70.99, 42.36, 35.61. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for [$C_{22}H_{19}BrO_3$] 410.0518; Found 410.0523.

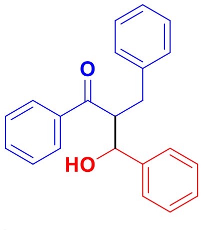
2-benzyl-3-hydroxy-1-(2-hydroxyphenyl)-3-(p-tolyl)propan-1-one (3q):



Yellow oil, fruity odour, yield 87 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.89 (s, 1H), 7.76 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.46 (td, *J* = 8.2, 1.6 Hz, 1H), 7.32 – 7.24 (m, 5H), 7.24 – 7.20 (m, 3H), 7.20 – 7.14 (m, 2H), 6.98 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.93 (td, *J* = 8.0, 1.3 Hz, 1H), 5.22 (ddt, *J* = 8.5, 5.4, 1.0 Hz, 1H), 3.75 – 3.66 (m, 1H), 3.58 (dd, *J* = 16.5, 8.2 Hz, 1H), 3.49 (dd, *J* = 16.4, 7.8 Hz, 1H), 3.10 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 197.72, 157.72, 136.42, 135.99, 131.75, 126.17, 125.41, 124.97, 123.83, 123.61, 122.63, 117.78, 116.43, 112.25, 73.00, 44.34, 37.60, 17.00. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for [$C_{23}H_{22}O_3$] 346.1569; Found 346.1574.

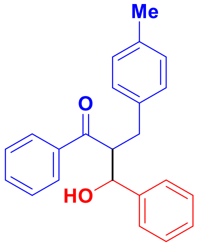
2-benzyl-3-hydroxy-1,3-diphenylpropan-1-one (3r):



Yellow oil, fruity odour, yield 85 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.88 – 7.82 (m, 2H), 7.51 – 7.45 (m, 1H), 7.45 – 7.38 (m, 2H), 7.31 – 7.19 (m, 9H), 7.19 – 7.12 (m, 1H), 5.10 (ddt, *J* = 8.5, 5.4, 1.0 Hz, 1H), 3.67 – 3.58 (m, 2H), 3.55 – 3.47 (m, 1H), 3.02 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 194.35, 138.73, 135.48, 132.82, 128.52, 124.43, 124.20, 123.64, 123.59, 123.20, 122.86, 122.38, 121.87, 72.15, 43.25, 35.64. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{22}H_{20}O_2]$ 316.1462; Found 316.1468.

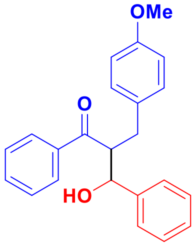
3-hydroxy-2-(4-methylbenzyl)-1,3-diphenylpropan-1-one (3s):



Yellow oil, fruity odour, yield 92 %

^{1}H NMR (500 MHz CDCl$_3$) δ 7.96 – 7.90 (m, 2H), 7.59 – 7.52 (m, 1H), 7.52 – 7.46 (m, 2H), 7.39 – 7.27 (m, 5H), 7.19 – 7.13 (m, 2H), 7.12 – 7.07 (m, 2H), 5.18 (ddt, *J* = 8.3, 5.1, 1.0 Hz, 1H), 3.79 – 3.68 (m, 2H), 3.54 – 3.45 (m, 1H), 3.10 (d, *J* = 5.4 Hz, 1H), 2.33 (s, 3H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 204.08, 148.46, 143.35, 142.55, 140.70, 138.25, 134.55, 134.16, 133.37, 133.32, 132.93, 132.72, 132.11, 81.88, 53.03, 45.37, 25.96. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{23}H_{22}O_2]$ 330.1620; Found 330.1682.

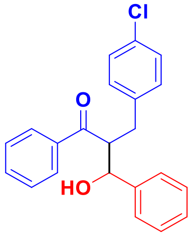
3-hydroxy-2-(4-methoxybenzyl)-1,3-diphenylpropan-1-one (3t):



Yellow oil, fruity odour, yield 95 %

^{1}H NMR (500 MHz CDCl$_3$) δ 8.01 – 7.95 (m, 2H), 7.64 – 7.57 (m, 1H), 7.57 – 7.51 (m, 2H), 7.44 – 7.31 (m, 5H), 7.24 – 7.19 (m, 2H), 6.93 – 6.87 (m, 2H), 5.20 (ddt, *J* = 8.5, 5.4, 1.0 Hz, 1H), 3.84 (s, 3H), 3.79 – 3.70 (m, 2H), 3.68 – 3.60 (m, 1H), 3.15 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 195.23, 154.76, 139.62, 133.71, 131.84, 129.40, 125.31, 124.72, 124.53, 124.48, 124.09, 123.27, 110.28, 73.01, 51.40, 44.40, 36.52. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{23}H_{22}O_3]$ 346.1569; Found 346.1574.

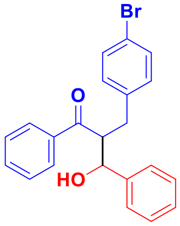
2-(4-chlorobenzyl)-3-hydroxy-1,3-diphenylpropan-1-one (3u):



Yellow oil, fruity odour, yield 78 %

^{1}H NMR (500 MHz CDCl$_3$) δ 7.90 – 7.84 (m, 2H), 7.53 – 7.46 (m, 1H), 7.46 – 7.40 (m, 2H), 7.30 (td, *J* = 2.4, 1.0 Hz, 1H), 7.29 – 7.25 (m, 3H), 7.25 – 7.15 (m, 6H), 5.11 (ddt, *J* = 8.5, 5.4, 1.0 Hz, 1H), 3.68 – 3.57 (m, 2H), 3.57 – 3.49 (m, 1H), 3.04 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 205.18, 149.56, 145.09, 143.66, 140.92, 139.35, 135.42, 135.26, 135.02, 134.48, 134.42, 134.04, 133.22, 82.94, 54.07, 46.47. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{22}H_{19}ClO_2]$ 350.1074; Found 350.1083.

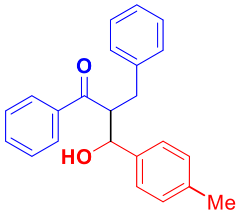
2-(4-bromobenzyl)-3-hydroxy-1,3-diphenylpropan-1-one (3v):



Yellow oil, fruity odour, yield 71 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.91 – 7.85 (m, 2H), 7.54 – 7.41 (m, 3H), 7.35 (s, 1H), 7.35 – 7.29 (m, 3H), 7.28 – 7.22 (m, 5H), 5.12 (ddt, *J* = 8.5, 5.4, 1.0 Hz, 1H), 3.69 – 3.58 (m, 2H), 3.58 – 3.50 (m, 1H), 3.05 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 199.18, 143.56, 139.77, 137.66, 133.35, 131.79, 129.26, 129.14, 128.48, 128.42, 128.04, 127.22, 121.85, 77.30, 48.28, 40.46. . **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{22}H_{19}BrO_2]$ 394.0568; Found 394.0593.

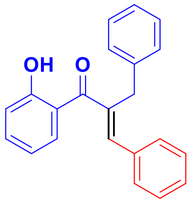
2-benzyl-3-hydroxy-1-phenyl-3-(p-tolyl)propan-1-one (3w):



Yellow oil, fruity odour, yield 89 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.91 – 7.85 (m, 2H), 7.54 – 7.48 (m, 1H), 7.48 – 7.41 (m, 2H), 7.25 (s, 4H), 7.18 (ddd, *J* = 9.4, 3.5, 1.7 Hz, 2H), 7.17 – 7.08 (m, 3H), 5.18 (ddt, *J* = 8.5, 5.3, 1.1 Hz, 1H), 3.69 – 3.60 (m, 2H), 3.58 – 3.50 (m, 1H), 3.05 (d, *J* = 5.4 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 193.18, 134.32, 134.05, 131.66, 129.82, 127.35, 123.48, 123.26, 123.04, 122.48, 121.91, 121.70, 120.71, 71.09, 42.08, 34.47, 15.07. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{23}H_{22}O_2]$ 330.1620; Found 330.1682.

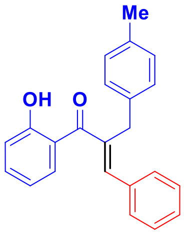
(E)-2-benzyl-1-(2-hydroxyphenyl)-3-phenylprop-2-en-1-one (3a'):



Yellow oil, fruity odour, yield 63 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.96 (s, 1H), 7.84 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.70 (t, *J* = 1.1 Hz, 1H), 7.53 (dd, *J* = 7.8, 2.4 Hz, 2H), 7.47 – 7.36 (m, 3H), 7.35 – 7.28 (m, 1H), 7.25 – 7.13 (m, 5H), 6.94 (dd, *J* = 8.4, 1.4 Hz, 1H), 6.90 (td, *J* = 8.0, 1.3 Hz, 1H), 3.87 (q, *J* = 1.0 Hz, 2H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 199.38, 161.38, 142.06, 139.30, 136.32, 135.40, 134.97, 130.80, 129.70, 129.38, 129.26, 129.16, 128.63, 127.54, 122.34, 118.39, 116.22, 35.52. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{22}H_{18}O_2]$ 314.1320; Found 314.5668.

(E)-1-(2-hydroxyphenyl)-2-(4-methylbenzyl)-3-phenylprop-2-en-1-one (3b'):

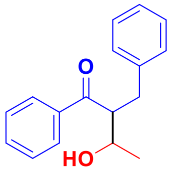


Pale yellow oil, fruity odour, yield 71 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.88 (s, 1H), 7.75 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.61 (t, *J* = 1.0 Hz, 1H), 7.45 (dd, *J* = 7.8, 2.4 Hz, 2H), 7.38 – 7.28 (m, 3H), 7.27 – 7.20 (m, 1H), 6.99 – 6.94 (m, 2H), 6.89 – 6.83 (m, 3H), 6.83 – 6.78 (m, 1H), 3.83 (q, *J* = 1.1 Hz, 2H), 2.20 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 199.38, 161.38, 142.06, 139.30, 135.94, 135.40, 134.97, 134.38, 130.80, 129.70, 129.44, 129.26, 129.20, 128.63, 122.34, 118.39, 116.22, 35.38, 21.04. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{23}H_{20}O_2]$ 328.1463; Found 328.8379.

Analytical data for Scheme 4:

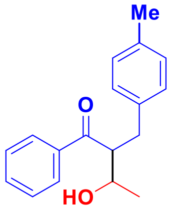
2-benzyl-3-hydroxy-1-phenylbutan-1-one (4a):



Colourless oil, fruity odour, yield 65 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.90 – 7.84 (m, 2H), 7.53 – 7.46 (m, 1H), 7.46 – 7.40 (m, 2H), 7.25 – 7.12 (m, 5H), 4.03 (pd, *J* = 6.5, 5.1 Hz, 1H), 3.62 (dd, *J* = 16.6, 8.1 Hz, 1H), 3.56 – 3.48 (m, 1H), 3.22 – 3.13 (m, 1H), 2.84 (d, *J* = 5.1 Hz, 1H), 1.20 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 199.37, 140.52, 137.67, 133.35, 129.26, 128.99, 128.48, 127.40, 126.70, 69.80, 49.44, 40.35, 21.12. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{17}H_{18}O_2]$ 254.1307; Found 254.1318.

3-hydroxy-2-(4-methylbenzyl)-1-phenylbutan-1-one (4b):

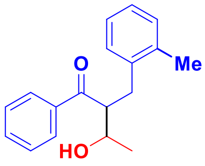


Pale yellow oil, fruity odour, yield 67 %

^{1}H NMR (500 MHz $CDCl_3$) δ 8.08 – 8.02 (m, 2H), 7.71 – 7.64 (m, 1H), 7.64 – 7.58 (m, 2H), 7.26 – 7.19 (m, 4H), 4.27 – 4.17 (m, 1H), 3.80 (dd, *J* = 16.6, 8.1 Hz, 1H), 3.74 – 3.65 (m, 1H), 3.43 – 3.34 (m, 1H), 3.02 (d, *J* = 5.1 Hz, 1H), 1.38 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 197.37, 136.91, 135.67, 133.81, 131.35, 127.66, 127.26, 126.48, 125.45, 67.81, 47.37, 38.35, 19.12, 19.07.

HRMS (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{18}H_{20}O_2]$ 268.1463; Found 268.1472.

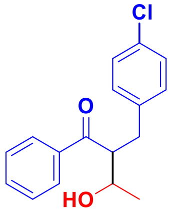
3-hydroxy-2-(2-methylbenzyl)-1-phenylbutan-1-one (4c):



Pale yellow oil, fruity odour, yield 73 %

^{1}H NMR (500 MHz $CDCl_3$) δ 8.07 – 8.01 (m, 2H), 7.71 – 7.64 (m, 1H), 7.64 – 7.57 (m, 2H), 7.39 (td, *J* = 7.9, 1.3 Hz, 1H), 7.29 – 7.19 (m, 3H), 4.21 – 4.11 (m, 1H), 3.78 (dd, *J* = 16.5, 7.9 Hz, 1H), 3.72 – 3.64 (m, 1H), 3.51 (tdd, *J* = 8.0, 7.0, 1.1 Hz, 1H), 3.16 (d, *J* = 5.1 Hz, 1H), 1.37 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 199.33, 141.62, 137.67, 137.24, 133.35, 129.82, 129.26, 128.48, 127.08, 126.92, 125.81, 69.70, 47.13, 40.36, 21.19, 19.58. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{18}H_{20}O_2]$ 268.1463; Found 268.1472.

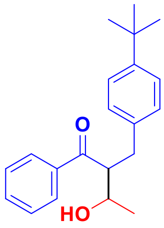
2-(4-chlorobenzyl)-3-hydroxy-1-phenylbutan-1-one (4d):



Pale yellow oil, fruity odour, yield 52 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.96 – 7.90 (m, 2H), 7.59 – 7.53 (m, 1H), 7.53 – 7.46 (m, 2H), 7.28 – 7.22 (m, 2H), 7.22 – 7.17 (m, 2H), 4.15 – 4.05 (m, 1H), 3.68 (dd, *J* = 16.6, 8.1 Hz, 1H), 3.62 – 3.54 (m, 1H), 3.31 – 3.23 (m, 1H), 2.90 (d, *J* = 5.1 Hz, 1H), 1.26 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 201.65, 150.47, 141.60, 138.49, 135.65, 134.38, 131.14, 129.28, 128.38, 126.17, 117.94, 117.48, 115.90, 41.08, 30.69, 30.28, 21.10. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{17}H_{17}ClO_2]$ 288.0917; Found 288.0925.

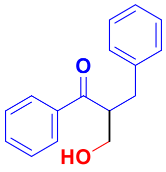
2-(4-(tert-butyl)benzyl)-3-hydroxy-1-phenylbutan-1-one (4e):



Pale yellow oil, fruity odour, yield 69 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.96 – 7.90 (m, 2H), 7.59 – 7.53 (m, 1H), 7.53 – 7.46 (m, 2H), 7.24 – 7.19 (m, 2H), 7.14 – 7.08 (m, 2H), 4.15 – 4.05 (m, 1H), 3.68 (dd, *J* = 16.6, 8.1 Hz, 1H), 3.62 – 3.54 (m, 1H), 3.27 (tdt, *J* = 8.2, 7.1, 1.0 Hz, 1H), 2.90 (d, *J* = 5.1 Hz, 1H), 1.33 (s, 9H), 1.26 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 199.37, 149.83, 140.19, 137.67, 133.35, 129.26, 128.48, 128.07, 125.56, 69.81, 49.41, 40.35, 34.72, 31.17, 21.12. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{21}H_{26}O_2]$ 310.1933; Found 310.1952.

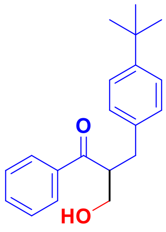
2-benzyl-3-hydroxy-1-phenylpropan-1-one (4f):



Pale yellow oil, fruity odour, yield 40 %

^{1}H NMR (500 MHz CDCl$_3$) δ 7.96 – 7.90 (m, 2H), 7.59 – 7.53 (m, 1H), 7.53 – 7.46 (m, 2H), 7.32 – 7.18 (m, 5H), 3.97 – 3.83 (m, 2H), 3.64 (dd, *J* = 16.7, 7.9 Hz, 1H), 3.58 – 3.49 (m, 1H), 3.31 – 3.22 (m, 1H), 2.68 (t, *J* = 5.7 Hz, 1H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 198.55, 141.01, 136.26, 132.35, 127.46, 127.43, 127.14, 126.27, 125.77, 65.88, 46.20, 40.81. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for [$C_{16}H_{16}O_2$] 240.1150; Found 240.1182.

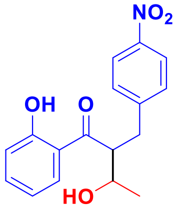
2-(4-(tert-butyl)benzyl)-3-hydroxy-1-phenylpropan-1-one (4g):



Pale yellow oil, fruity odour, yield 68 %

^{1}H NMR (500 MHz CDCl$_3$) δ 7.94 – 7.88 (m, 2H), 7.58 – 7.52 (m, 1H), 7.52 – 7.44 (m, 2H), 7.25 – 7.19 (m, 2H), 7.13 – 7.07 (m, 2H), 3.95 – 3.82 (m, 2H), 3.63 (dd, *J* = 16.7, 7.9 Hz, 1H), 3.56 – 3.48 (m, 1H), 3.29 (ttt, *J* = 7.8, 5.5, 1.0 Hz, 1H), 2.67 (t, *J* = 5.7 Hz, 1H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 196.55, 146.54, 138.22, 134.25, 130.34, 125.45, 125.42, 124.48, 122.65, 63.87, 44.18, 38.80, 31.71, 28.16. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for [$C_{20}H_{24}O_2$] 296.1776; Found 296.1795.

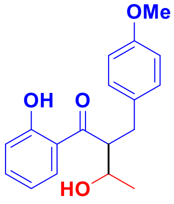
3-hydroxy-1-(2-hydroxyphenyl)-2-(4-nitrobenzyl)butan-1-one (4h):



Pale yellow oil, fruity odour, yield 40 %

^{1}H NMR (500 MHz CDCl$_3$) δ 7.93 – 7.87 (m, 2H), 7.77 – 7.71 (m, 2H), 7.41 – 7.34 (m, 1H), 7.34 – 7.27 (m, 2H), 7.27 – 7.21 (m, 2H), 3.71 (ddt, *J* = 17.8, 12.2, 6.0 Hz, 2H), 3.45 (dd, *J* = 16.8, 7.8 Hz, 1H), 3.39 – 3.30 (m, 1H), 3.21 – 3.11 (m, 1H), 2.50 (d, *J* = 11.5 Hz, 1H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 201.78, 161.79, 150.65, 146.24, 135.82, 130.24, 128.18, 123.96, 121.77, 120.53, 116.32, 69.74, 49.21, 41.55, 21.09. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for [$C_{17}H_{17}NO_5$] 315.1107; Found 315.1112.

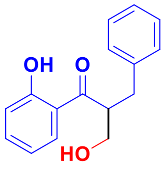
3-hydroxy-1-(2-hydroxyphenyl)-2-(4-methoxybenzyl)butan-1-one (4i):



Pale yellow oil, fruity odour, yield 60 %

^{1}H NMR (500 MHz CDCl$_3$) δ 11.89 (s, 1H), 7.76 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.46 (td, *J* = 8.2, 1.6 Hz, 1H), 7.18 – 7.12 (m, 2H), 6.98 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.93 (td, *J* = 8.0, 1.3 Hz, 1H), 6.87 – 6.81 (m, 2H), 4.15 – 4.05 (m, 1H), 3.80 (s, 3H), 3.65 (dd, *J* = 16.5, 8.2 Hz, 1H), 3.60 – 3.51 (m, 1H), 3.32 – 3.23 (m, 1H), 2.90 (d, *J* = 5.1 Hz, 1H), 1.26 (s, 3H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 201.78, 161.79, 158.68, 135.84, 135.82, 130.24, 128.28, 121.77, 120.53, 116.32, 114.19, 69.75, 55.35, 49.44, 41.54, 21.11. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{18}H_{20}O_4]$ 300.1363; Found 300.1369.

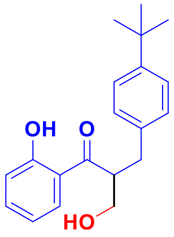
2-benzyl-3-hydroxy-1-(2-hydroxyphenyl)propan-1-one (4j):



Pale yellow oil, fruity odour, yield 28 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.87 (s, 1H), 7.74 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.45 (td, *J* = 8.2, 1.6 Hz, 1H), 7.30 – 7.16 (m, 5H), 6.97 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.92 (td, *J* = 8.0, 1.3 Hz, 1H), 3.95 – 3.82 (m, 2H), 3.60 (dd, *J* = 16.5, 7.9 Hz, 1H), 3.55 – 3.46 (m, 1H), 3.34 – 3.25 (m, 1H), 2.67 (d, *J* = 11.5 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 203.45, 161.79, 142.07, 135.82, 130.24, 128.14, 127.24, 126.77, 121.89, 120.53, 116.32, 66.88, 47.14, 43.18. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{16}H_{16}O_3]$ 256.1099; Found 256.1108.

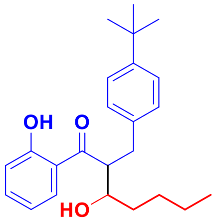
2-(4-(tert-butyl)benzyl)-3-hydroxy-1-(2-hydroxyphenyl)propan-1-one (4k):



Pale yellow oil, fruity odour, yield 34 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.78 (s, 1H), 7.66 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.36 (td, *J* = 8.2, 1.6 Hz, 1H), 7.16 – 7.10 (m, 2H), 7.04 – 6.98 (m, 2H), 6.88 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.83 (td, *J* = 8.0, 1.3 Hz, 1H), 3.86 – 3.73 (m, 2H), 3.51 (dd, *J* = 16.6, 7.8 Hz, 1H), 3.41 (dd, *J* = 16.6, 8.1 Hz, 1H), 3.30 – 3.21 (m, 1H), 2.58 (t, *J* = 5.7 Hz, 1H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 198.00, 156.35, 144.10, 135.80, 130.38, 124.79, 122.03, 120.22, 116.45, 115.08, 110.88, 61.43, 41.74, 37.73, 29.27, 25.72. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{20}H_{24}O_3]$ 312.1725; Found 312.1729.

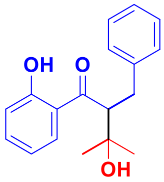
2-(4-(tert-butyl)benzyl)-3-hydroxy-1-(2-hydroxyphenyl)heptan-1-one (4l):



Pale yellow oil, fruity odour, yield 71 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.90 (s, 1H), 7.78 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.48 (td, *J* = 8.2, 1.6 Hz, 1H), 7.26 – 7.21 (m, 2H), 7.16 – 7.10 (m, 2H), 7.00 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.95 (td, *J* = 8.0, 1.3 Hz, 1H), 4.05 – 3.96 (m, 1H), 3.65 (dd, *J* = 16.5, 7.7 Hz, 1H), 3.55 (dd, *J* = 16.5, 7.9 Hz, 1H), 3.35 (qt, *J* = 7.8, 1.0 Hz, 1H), 2.87 (d, *J* = 5.4 Hz, 1H), 1.56 (dddt, *J* = 13.4, 9.0, 6.3, 4.4 Hz, 2H), 1.48 – 1.32 (m, 13H), 0.96 – 0.87 (m, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 200.72, 160.79, 148.80, 139.62, 134.82, 129.24, 127.18, 124.56, 120.69, 119.53, 115.32, 72.39, 46.75, 40.81, 33.72, 33.71, 30.17, 26.94, 21.74, 13.07. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{24}H_{32}O_3]$ 368.2351; Found 368.2368.

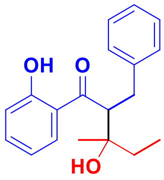
2-benzyl-3-hydroxy-1-(2-hydroxyphenyl)-3-methylbutan-1-one (4m):



Pale yellow oil, fruity odour, yield 60 %

^{1}H NMR (500 MHz $CDCl_3$) δ 11.89 (s, 1H), 7.76 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.46 (td, *J* = 8.2, 1.6 Hz, 1H), 7.30 – 7.24 (m, 3H), 7.24 – 7.15 (m, 2H), 6.98 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.93 (td, *J* = 8.0, 1.3 Hz, 1H), 3.52 (dd, *J* = 16.6, 8.5 Hz, 1H), 3.48 – 3.39 (m, 1H), 3.15 (tt, *J* = 8.7, 1.0 Hz, 1H), 2.83 (s, 1H), 1.31 (s, 3H), 1.26 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 203.38, 161.79, 139.69, 135.82, 130.24, 128.74, 128.29, 127.25, 121.38, 120.50, 116.32, 73.65, 55.18, 41.21, 28.09. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{18}H_{20}O_3]$ 284.1412; Found 284.1431.

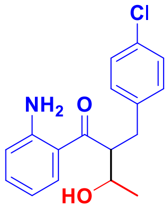
2-benzyl-3-hydroxy-1-(2-hydroxyphenyl)-3-methylpentan-1-one (4n):



Pale yellow oil, fruity odour, yield 67 %

^{1}H NMR (500 MHz $CDCl_3$) δ11.89 (s, 1H), 7.76 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.46 (td, *J* = 8.2, 1.6 Hz, 1H), 7.25 (dtd, *J* = 5.4, 4.3, 1.0 Hz, 3H), 7.21 – 7.16 (m, 2H), 6.98 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.93 (td, *J* = 8.0, 1.3 Hz, 1H), 3.56 (dd, *J* = 16.7, 7.9 Hz, 1H), 3.49 – 3.40 (m, 1H), 3.19 (tt, *J* = 8.0, 1.0 Hz, 1H), 2.83 (s, 1H), 1.58 (q, *J* = 8.0 Hz, 2H), 0.94 (t, *J* = 8.0 Hz, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 198.50, 156.96, 135.17, 130.99, 125.41, 123.92, 123.36, 122.42, 116.58, 115.67, 111.49, 71.23, 48.92, 36.93, 28.51, 20.06, 3.38. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{19}H_{22}O_3]$ 298.1569; Found 298.1583.

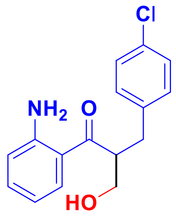
1-(2-aminophenyl)-2-(4-chlorobenzyl)-3-hydroxybutan-1-one (4o):



Pale yellow oil, fruity odour, yield 35 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.74 (dd, *J* = 7.7, 1.6 Hz, 2H), 7.31 – 7.24 (m, 6H), 7.24 – 7.19 (m, 4H), 6.79 – 6.72 (m, 3H), 6.71 (d, *J* = 1.3 Hz, 1H), 6.17 (s, 4H), 4.17 – 4.08 (m, 2H), 3.66 (dd, *J* = 16.4, 8.1 Hz, 2H), 3.60 – 3.51 (m, 2H), 3.35 – 3.26 (m, 2H), 2.93 (d, *J* = 5.1 Hz, 2H), 1.29 (d, *J* = 6.6 Hz, 6H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 202.58, 150.67, 139.46, 134.91, 134.83, 130.50, 129.03, 128.93, 121.28, 117.93, 117.15, 69.75, 49.18, 41.10, 21.05. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{17}H_{18}ClNO_2]$ 303.1026; Found 303.1037.

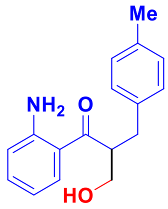
1-(2-aminophenyl)-2-(4-chlorobenzyl)-3-hydroxypropan-1-one (4p):



Pale yellow oil, fruity odour, yield 33 %

^{1}H NMR (500 MHz CDCl3) δ 7.69 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.25 – 7.19 (m, 3H), 7.18 – 7.12 (m, 2H), 6.74 – 6.67 (m, 2H), 6.66 (d, *J* = 1.3 Hz, 1H), 6.12 (s, 2H), 3.94 – 3.81 (m, 2H), 3.52 (dd, *J* = 16.6, 7.8 Hz, 1H), 3.45 – 3.29 (m, 2H), 2.66 (t, *J* = 5.7 Hz, 1H). **^{13}C NMR** (126 MHz, CDCl3) δ 202.62, 150.67, 140.47, 134.83, 133.24, 130.50, 129.09, 128.99, 121.28, 117.93, 117.15, 66.88, 46.85, 42.72. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for [$C_{16}H_{16}O_3$] 256.1099; Found 256.1108. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for [$C_{16}H_{16}ClNO_2$] 289.0870; Found 289.0879.

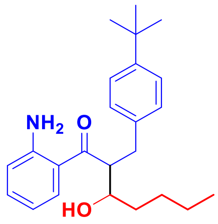
1-(2-aminophenyl)-3-hydroxy-2-(4-methylbenzyl)propan-1-one (4q):



Pale yellow oil, fruity odour, yield 24 %

^{1}H NMR (500 MHz CDCl3) δ 7.71 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.23 (td, *J* = 7.7, 1.5 Hz, 1H), 7.15 – 7.04 (m, 4H), 6.75 – 6.68 (m, 2H), 6.13 (s, 2H), 3.96 – 3.82 (m, 2H), 3.53 (dd, *J* = 16.6, 7.8 Hz, 1H), 3.46 – 3.30 (m, 2H), 2.67 (t, *J* = 5.7 Hz, 1H), 2.32 (s, 3H). **^{13}C NMR** (126 MHz, CDCl3) δ 202.62, 150.67, 139.39, 135.55, 134.83, 130.50, 129.73, 127.49, 121.28, 117.93, 117.15, 66.88, 47.16, 42.72, 21.07. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for [$C_{17}H_{19}NO_2$] 269.1416; Found 269.1424.

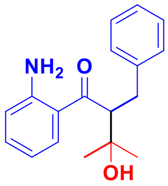
1-(2-aminophenyl)-2-(4-(tert-butyl)benzyl)-3-hydroxyheptan-1-one (4r):



Pale yellow oil, fruity odour, yield 59 %

^{1}H NMR (500 MHz CDCl3) δ 7.74 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.30 – 7.23 (m, 2H), 7.23 (s, 1H), 7.16 – 7.10 (m, 2H), 6.78 – 6.71 (m, 1H), 6.16 (s, 2H), 4.01 (dtd, *J* = 7.8, 6.6, 5.6 Hz, 1H), 3.58 (dd, *J* = 16.5, 7.7 Hz, 1H), 3.47 (dd, *J* = 16.5, 7.9 Hz, 1H), 3.36 (qt, *J* = 7.6, 1.0 Hz, 1H), 2.87 (d, *J* = 5.4 Hz, 1H), 1.56 (dddt, *J* = 13.4, 9.0, 6.3, 4.4 Hz, 2H), 1.48 – 1.28 (m, 13H), 0.96 – 0.88 (m, 3H). **^{13}C NMR** (126 MHz, CDCl3) δ 202.65, 150.67, 149.80, 140.60, 134.83, 130.50, 128.17, 125.56, 121.24, 117.93, 117.15, 73.38, 47.53, 41.48, 34.74, 34.72, 31.17, 27.94, 22.74, 14.07. **HRMS** (ESI-TOF) m/z: [M]$^+$ Calcd for [$C_{24}H_{33}NO_2$] 367.2511; Found 367.2537.

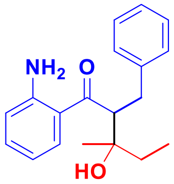
1-(2-aminophenyl)-2-benzyl-3-hydroxy-3-methylbutan-1-one (4s):



Pale yellow oil, fruity odour, yield 53 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.71 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.29 – 7.14 (m, 6H), 6.75 – 6.66 (m, 2H), 6.14 (s, 2H), 3.57 – 3.49 (m, 1H), 3.44 (t, *J* = 8.3 Hz, 1H), 3.14 (tt, *J* = 8.6, 1.0 Hz, 1H), 2.82 (s, 1H), 1.27 (d, *J* = 24.9 Hz, 6H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 202.04, 150.67, 139.69, 134.83, 130.50, 128.74, 128.28, 127.25, 121.17, 117.93, 117.05, 73.64, 55.02, 40.98, 28.02. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{18}H_{21}NO_2]$ 283.1572; Found 283.1593.

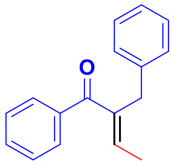
1-(2-aminophenyl)-2-benzyl-3-hydroxy-3-methylpentan-1-one (4t):



Pale yellow oil, fruity odour, yield 58 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.72 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.30 – 7.17 (m, 6H), 6.76 – 6.67 (m, 2H), 6.15 (s, 2H), 3.55 (dd, *J* = 16.7, 7.9 Hz, 1H), 3.47 – 3.39 (m, 1H), 3.19 (tt, *J* = 7.9, 1.0 Hz, 1H), 2.83 (s, 1H), 1.58 (t, *J* = 7.9 Hz, 2H), 1.27 (s, 3H), 0.94 (t, *J* = 8.0 Hz, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 198.01, 146.67, 135.99, 130.83, 126.50, 124.75, 124.14, 123.25, 117.15, 113.93, 113.05, 72.06, 49.63, 37.60, 29.50, 20.88, 4.21. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{19}H_{23}NO_2]$ 297.1729; Found 297.1732.

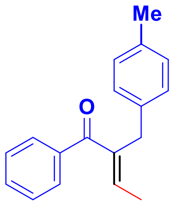
(E)-2-benzyl-1-phenylbut-2-en-1-one (4a'):



Colourless oil, fruity odour, yield 46 %

^{1}H NMR (500 MHz $CDCl_3$) δ 7.90 (dq, *J* = 8.2, 1.7 Hz, 2H), 7.56 – 7.49 (m, 1H), 7.46 – 7.39 (m, 2H), 7.30 – 7.18 (m, 5H), 6.74 (qt, *J* = 5.6, 1.0 Hz, 1H), 3.67 (hept, *J* = 1.0 Hz, 2H), 1.84 – 1.80 (m, 3H).**^{13}C NMR** (126 MHz, $CDCl_3$) δ 195.65, 138.53, 137.45, 137.19, 136.81, 132.72, 129.36, 129.34, 129.16, 127.77, 127.54, 35.28, 15.05. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{17}H_{16}O]$ 236.1210; Found 236.9585.

(E)-2-(4-methylbenzyl)-1-phenylbut-2-en-1-one (4b'):

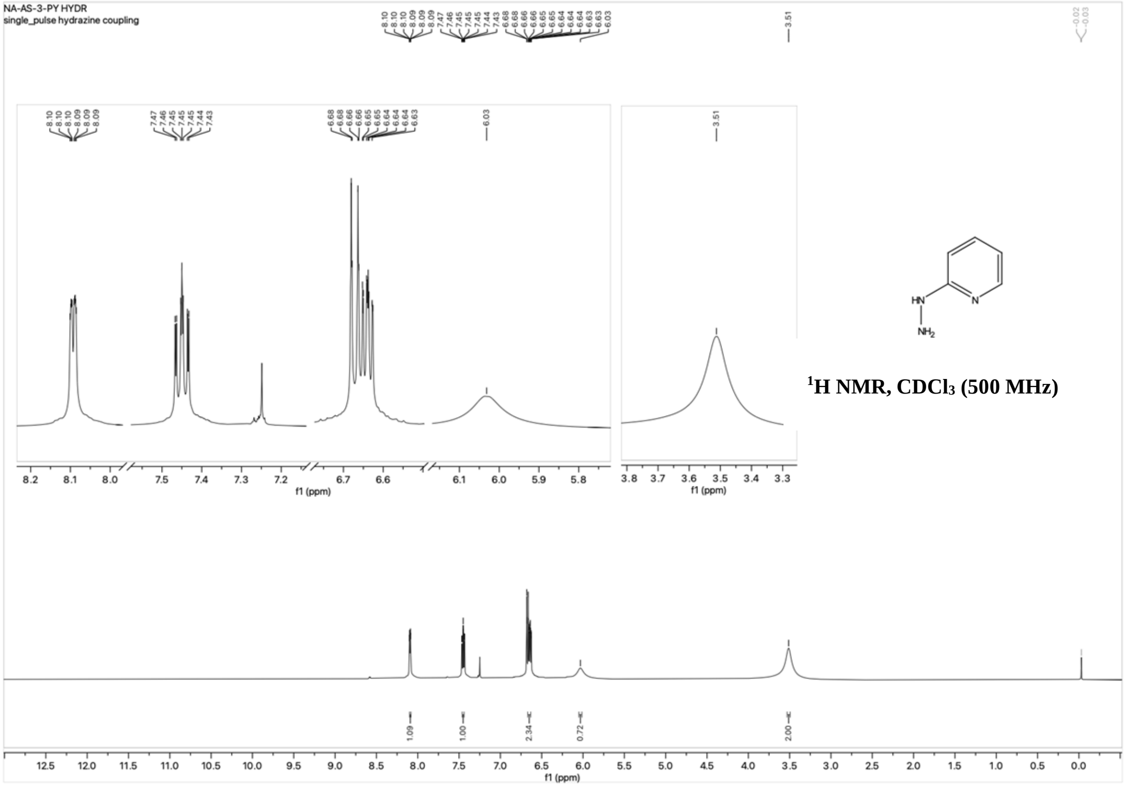


Colourless oil, fruity odour, yield 52 %

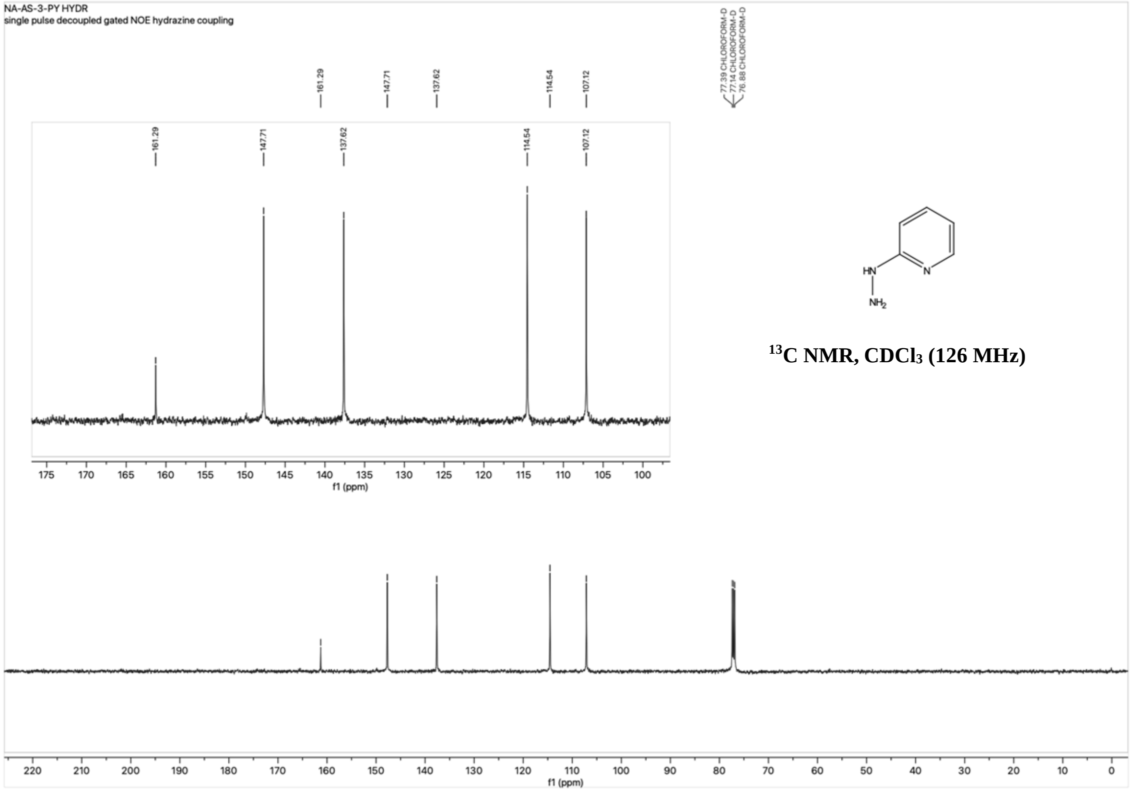
^{1}H NMR (500 MHz $CDCl_3$) δ 7.71 (dq, *J* = 8.1, 1.6 Hz, 2H), 7.38 – 7.31 (m, 1H), 7.28 – 7.21 (m, 2H), 6.92 (dd, *J* = 7.4, 1.2 Hz, 2H), 6.80 (dt, *J* = 7.9, 1.0 Hz, 2H), 6.59 – 6.52 (m, 1H), 3.51 (hept, *J* = 0.9 Hz, 2H), 2.15 (s, 3H), 1.64 (dt, *J* = 5.9, 1.0 Hz, 3H). **^{13}C NMR** (126 MHz,

$CDCl_3$) δ 195.65, 138.53, 137.45, 136.81, 135.94, 134.71, 132.72, 129.44, 129.36, 129.20, 127.77, 35.19, 21.04, 15.05. **HRMS** (ESI-TOF) m/z: $[M]^+$ Calcd for $[C_{17}H_{16}O]$ 250.1358; Found 250.9590.

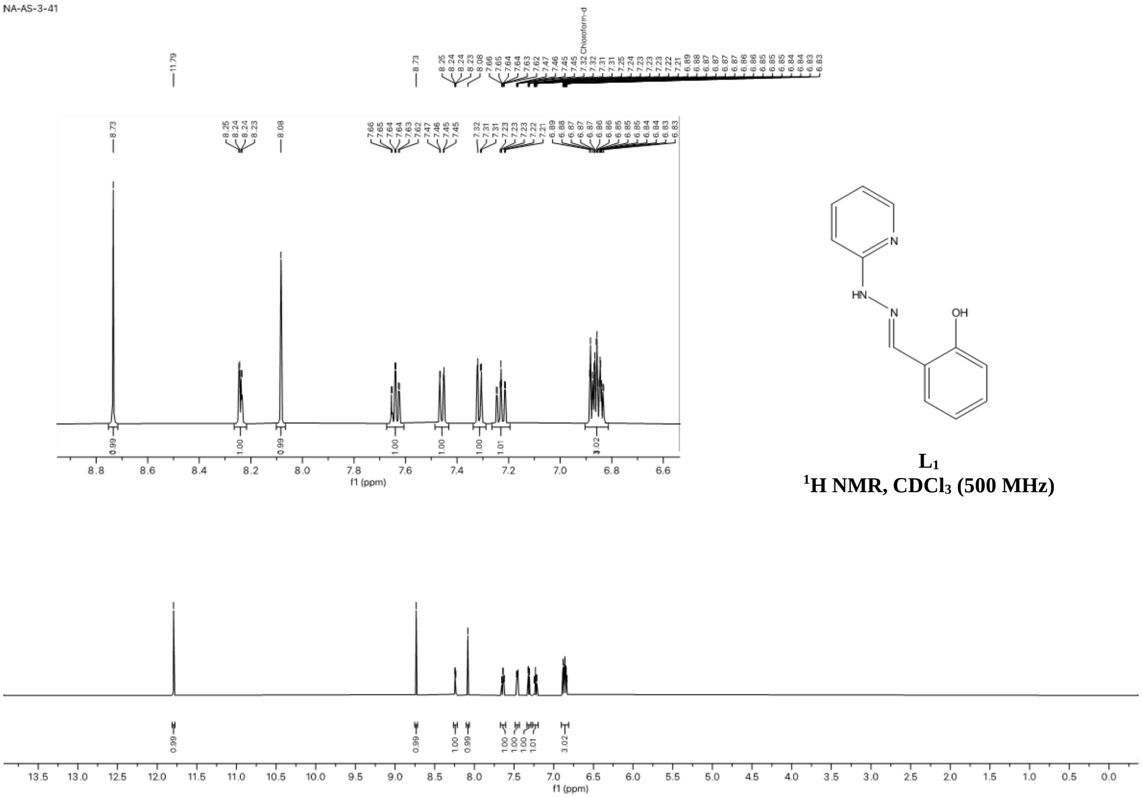
[2.0] NMR copies:



^1H NMR, $CDCl_3$ (500 MHz)



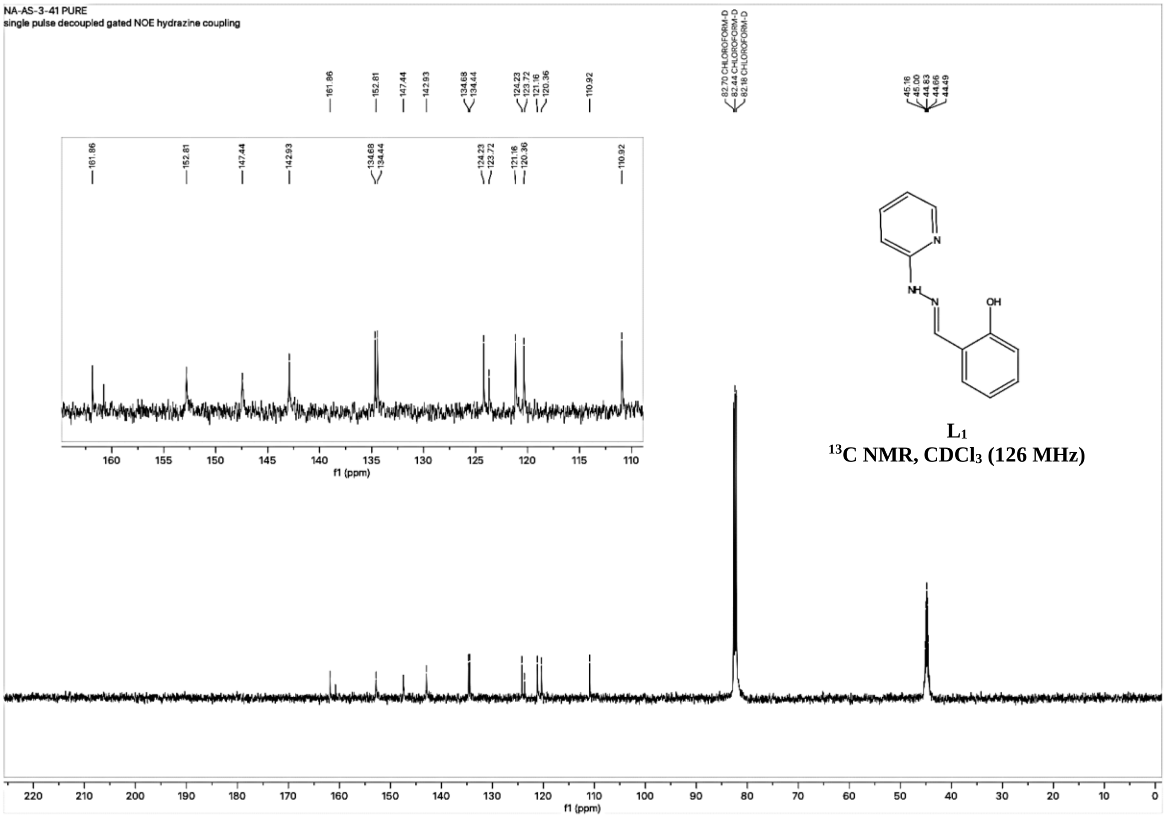
^{13}C NMR, CDCl$_3$ (126 MHz)



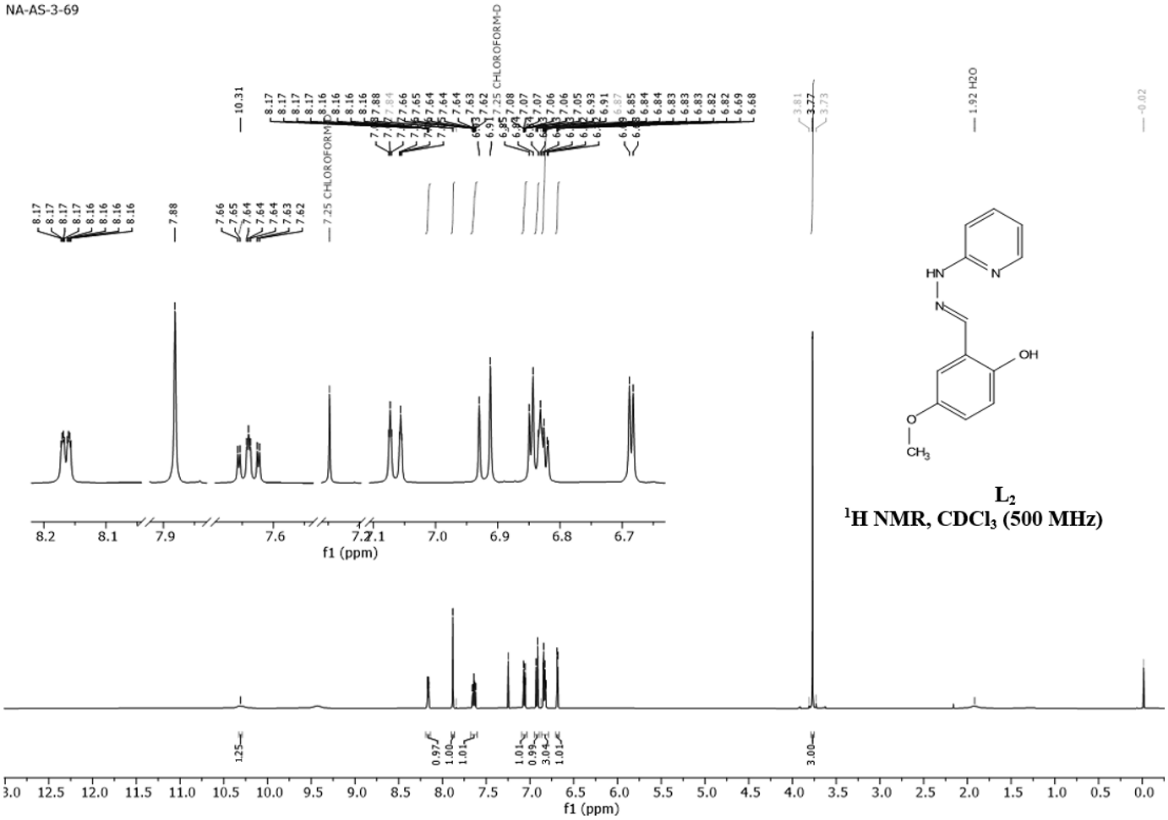
L$_1$

^{1}H NMR, CDCl$_3$ (500 MHz)

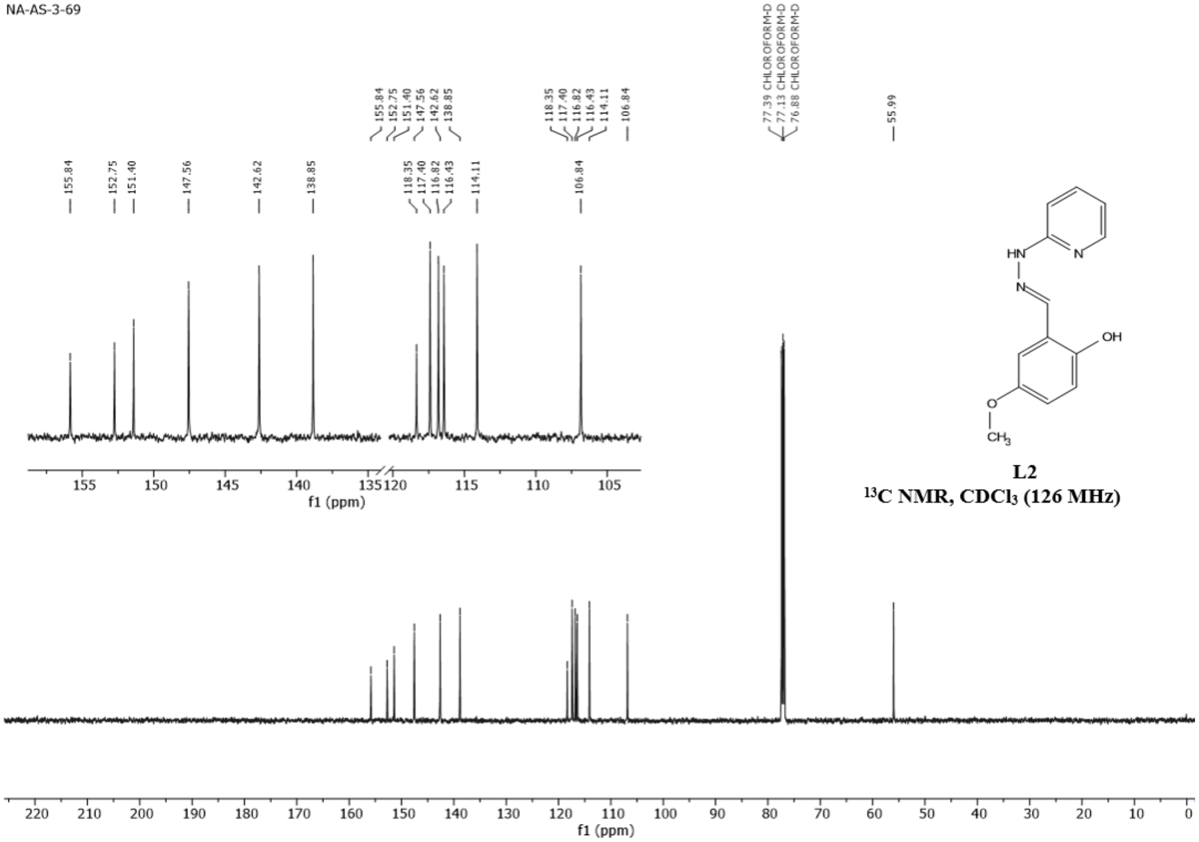
.



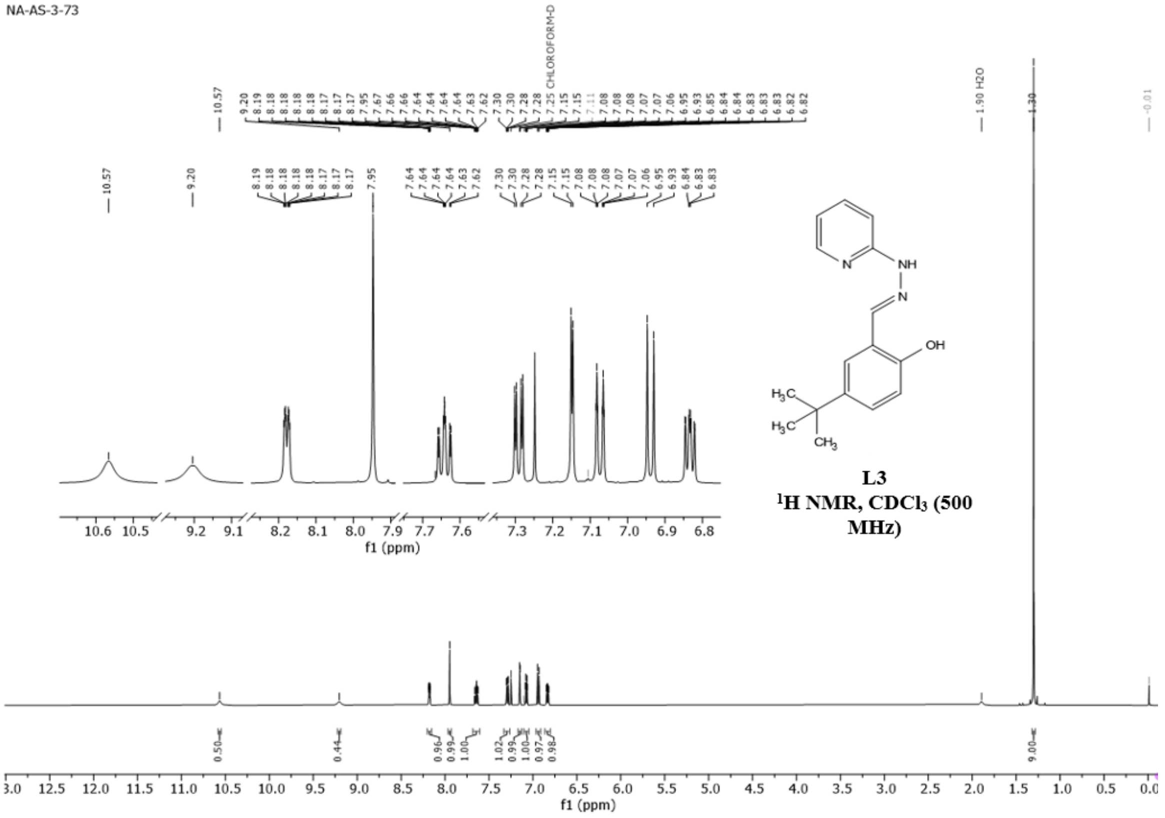
L_1
^{13}C NMR, $CDCl_3$ (126 MHz)



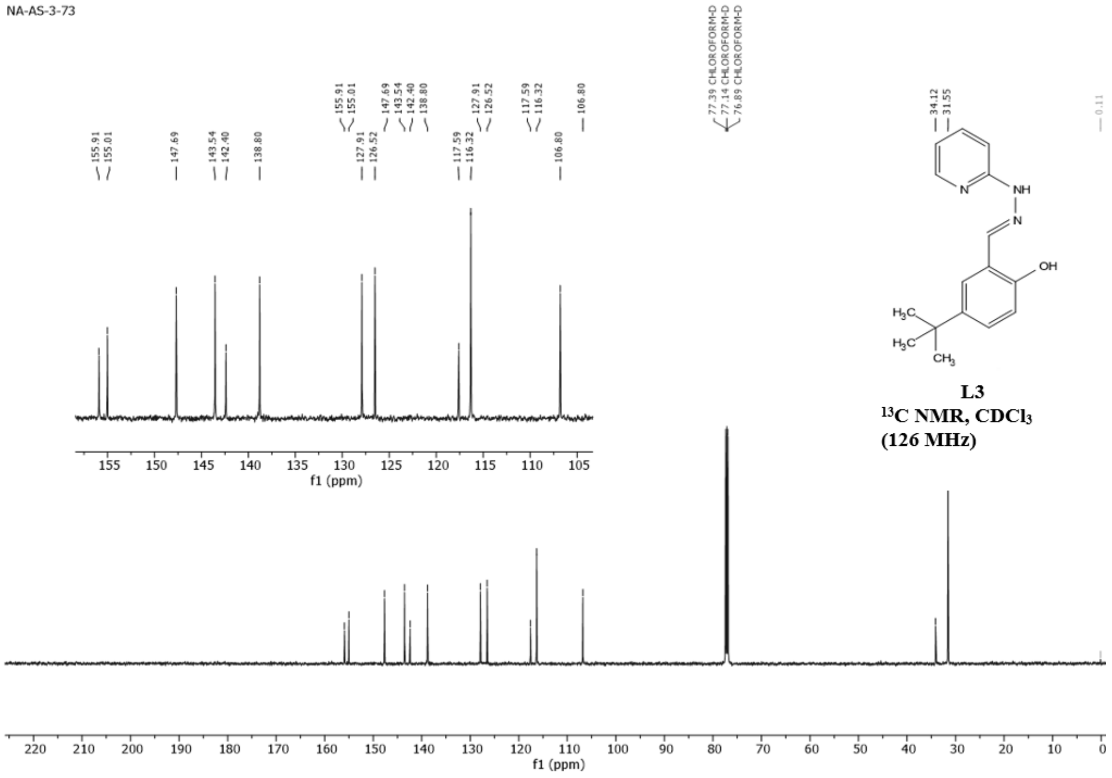
L_2
^{1}H NMR, $CDCl_3$ (500 MHz)



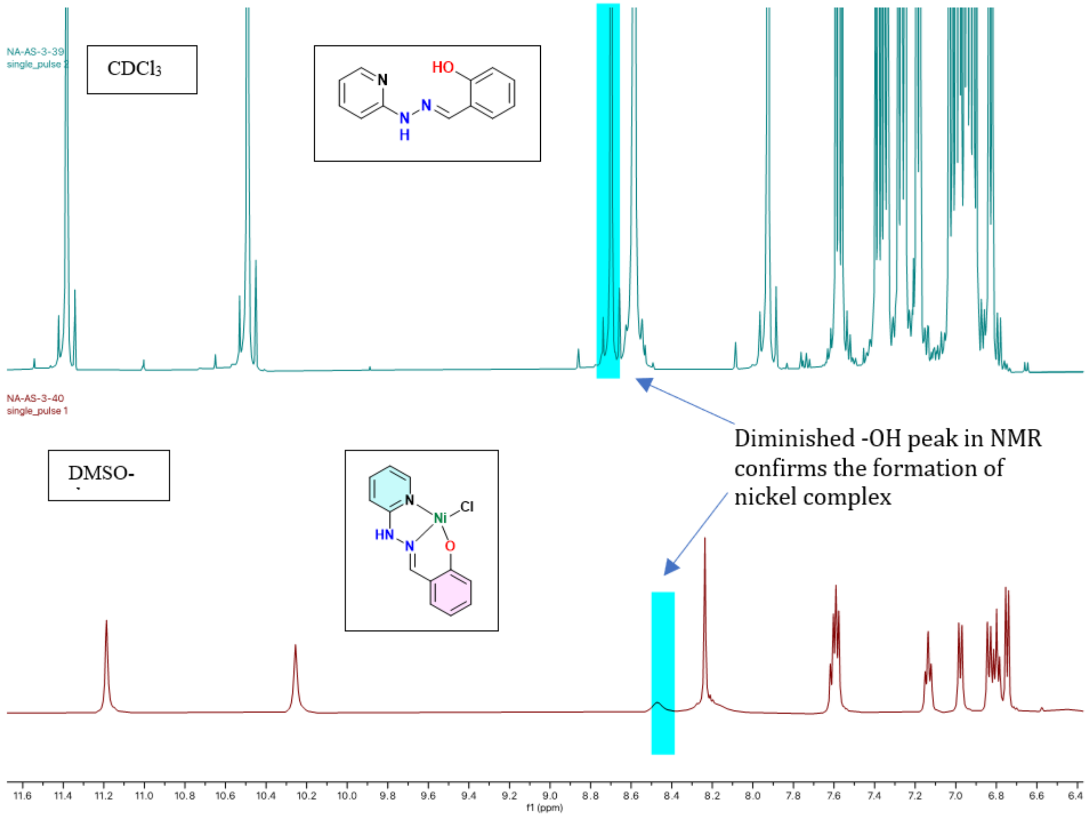
L2
^{13}C NMR, CDCl$_3$ (126 MHz)



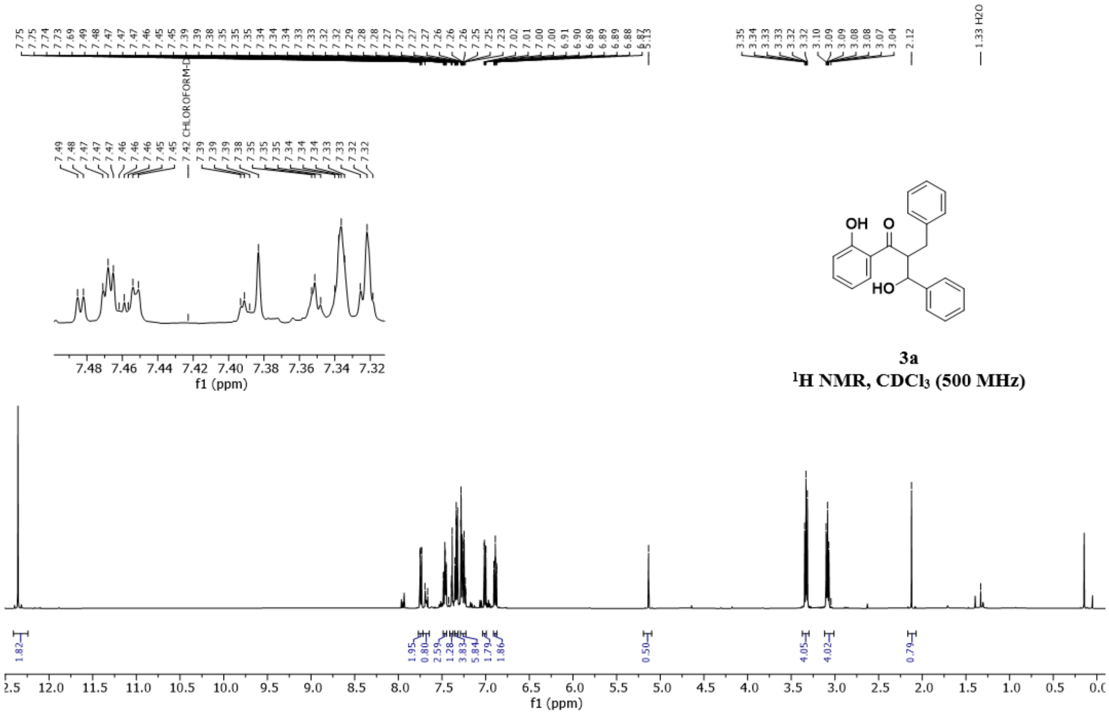
L3
^{1}H NMR, CDCl$_3$ (500 MHz)



L3
^{13}C **NMR,** $CDCl_3$
(126 MHz)

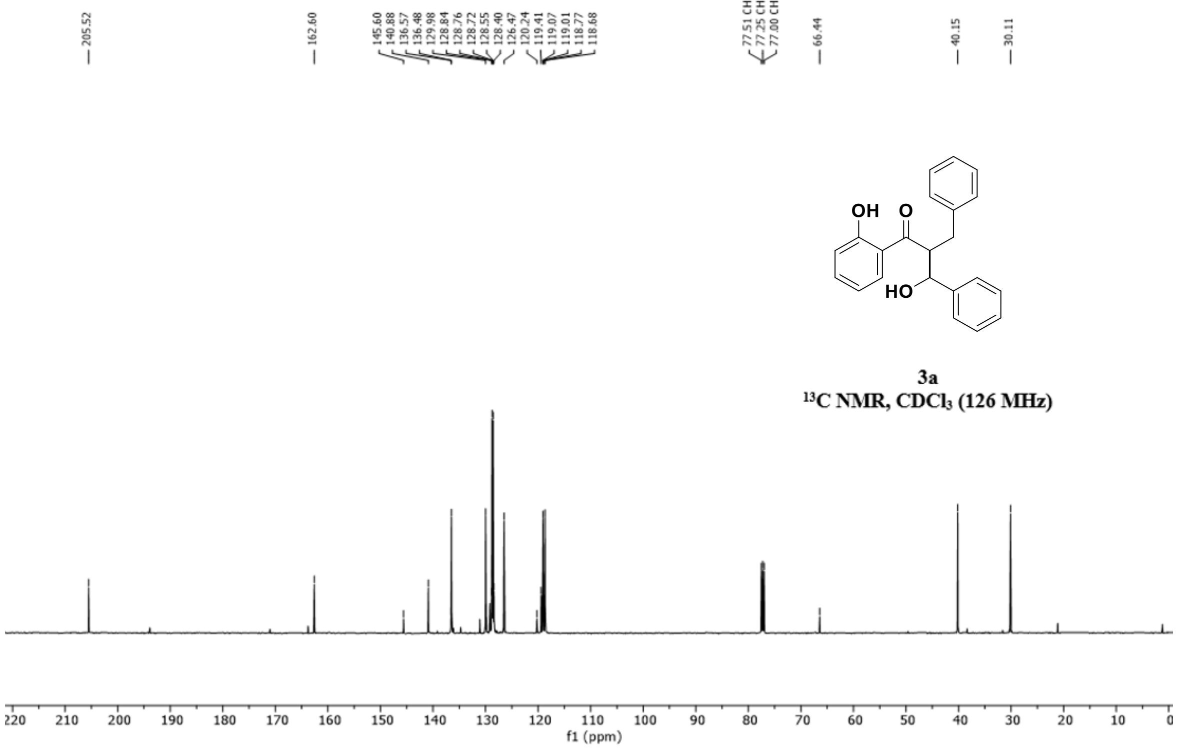


Diminished -OH peak in NMR confirms the formation of nickel complex



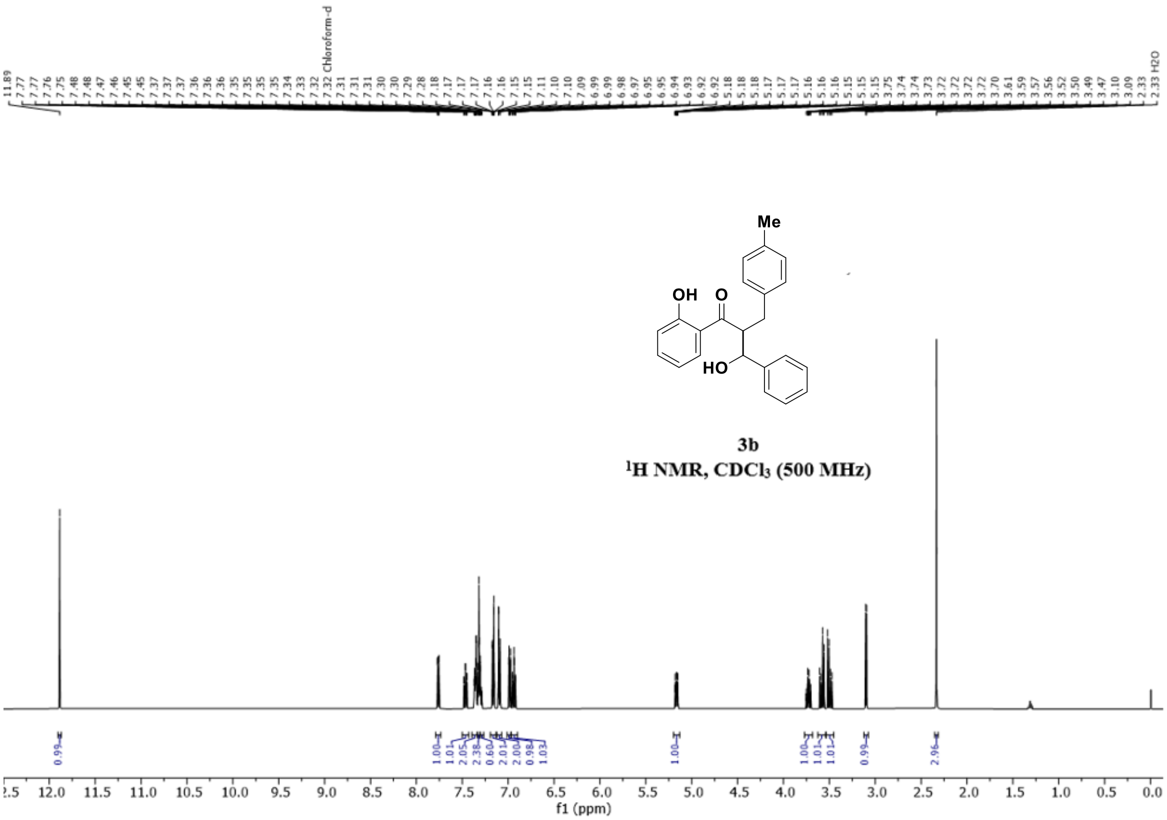
3a

^{1}H NMR, CDCl$_3$ (500 MHz)



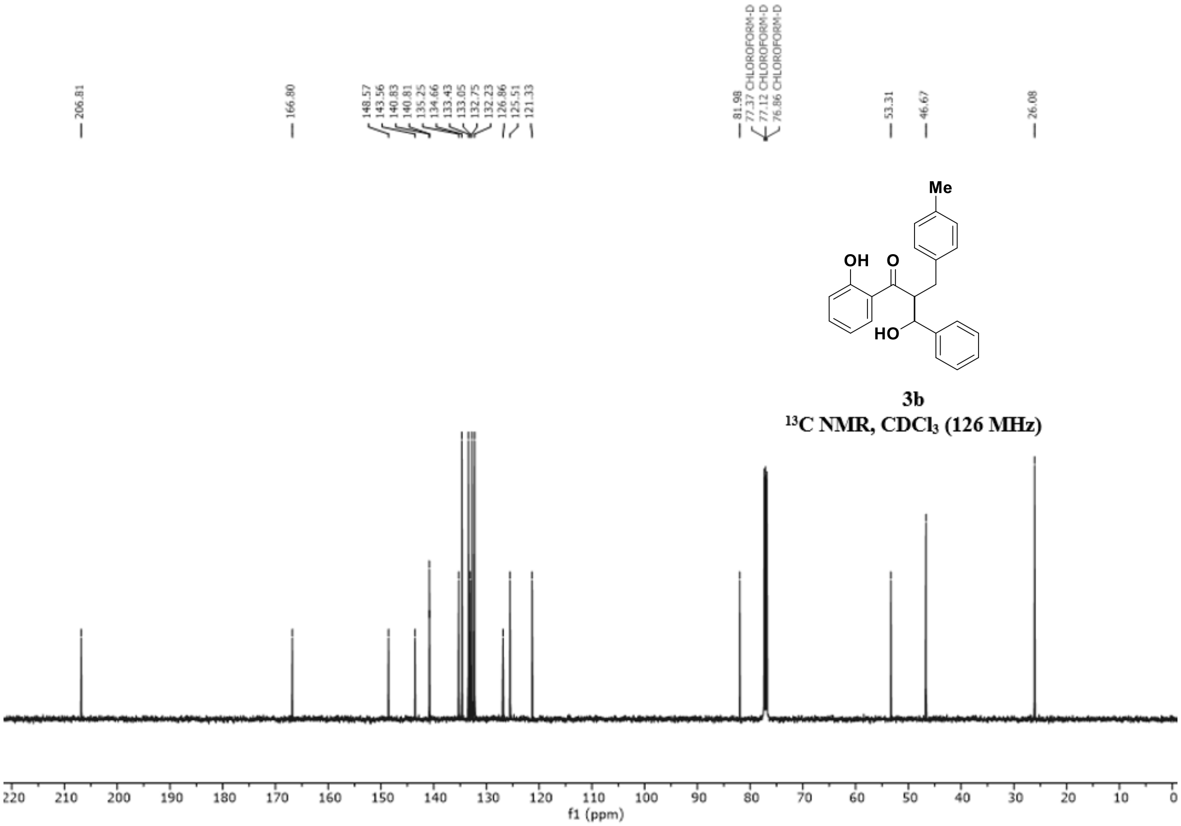
3a

^{13}C NMR, CDCl$_3$ (126 MHz)



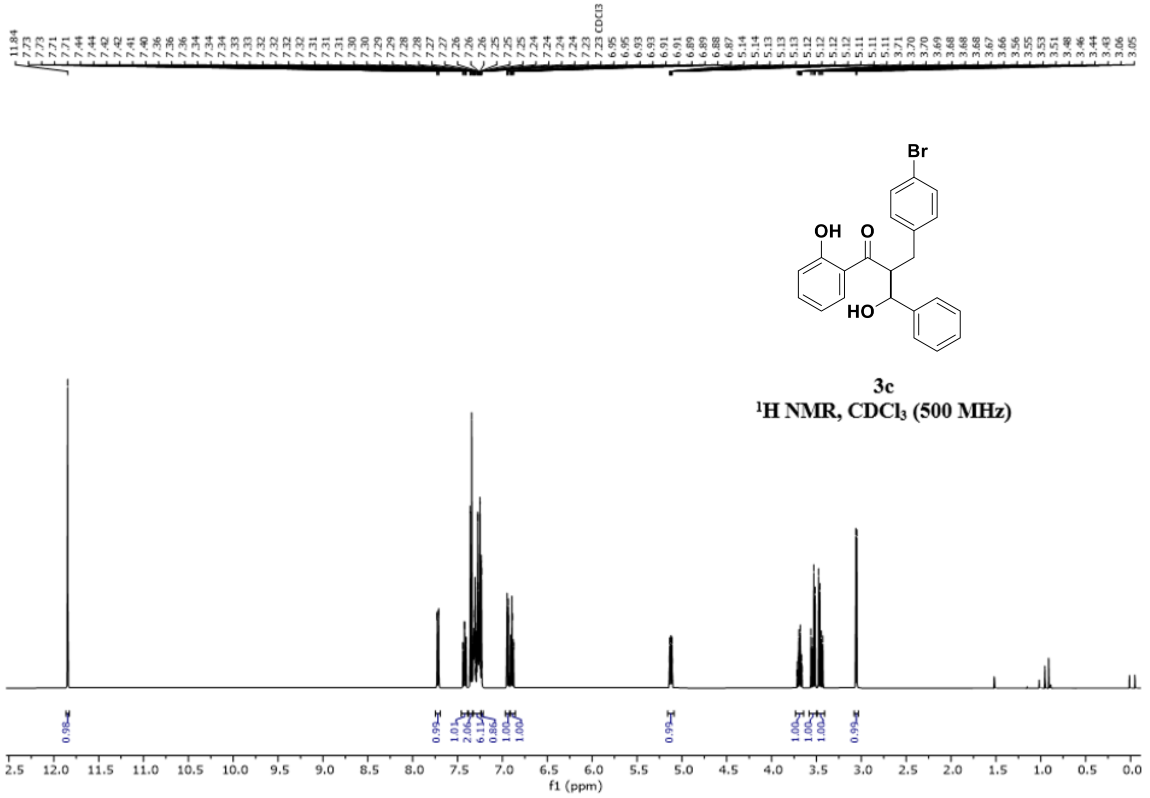
3b

^{1}H NMR, CDCl$_3$ (500 MHz)



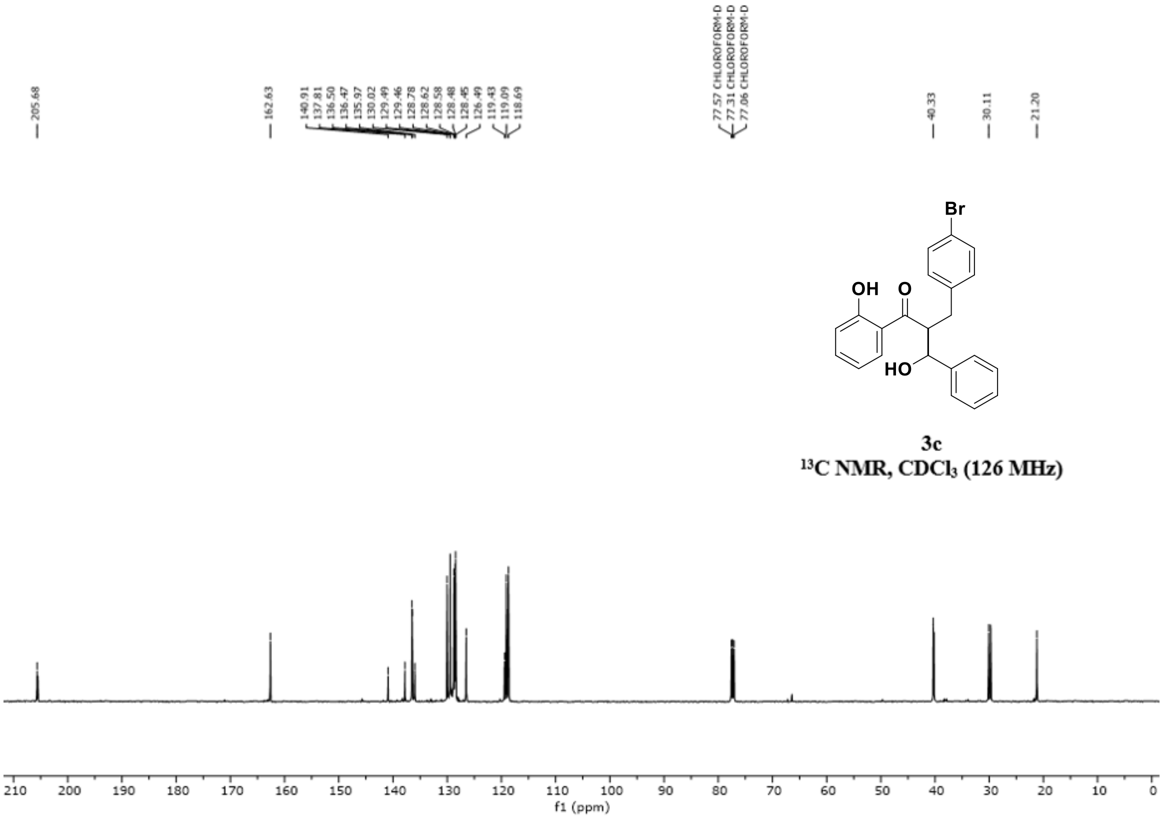
3b

^{13}C NMR, CDCl$_3$ (126 MHz)



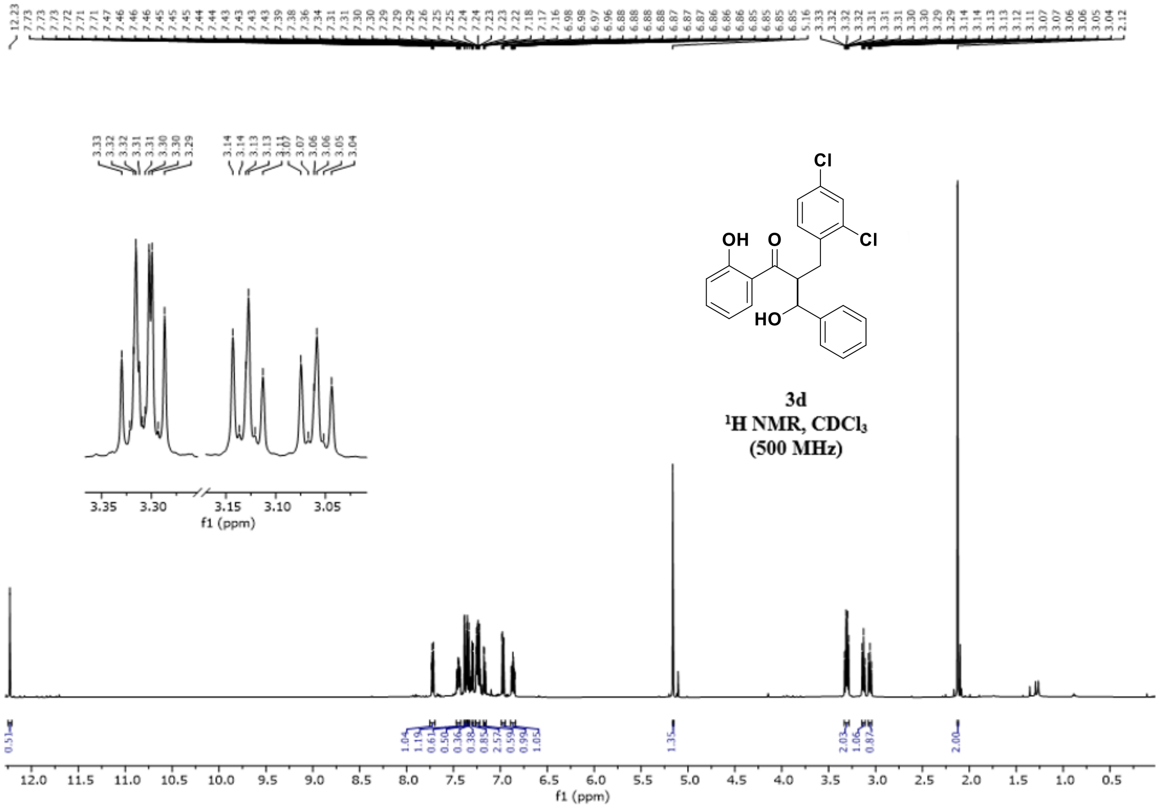
3c

^{1}H NMR, $CDCl_3$ (500 MHz)

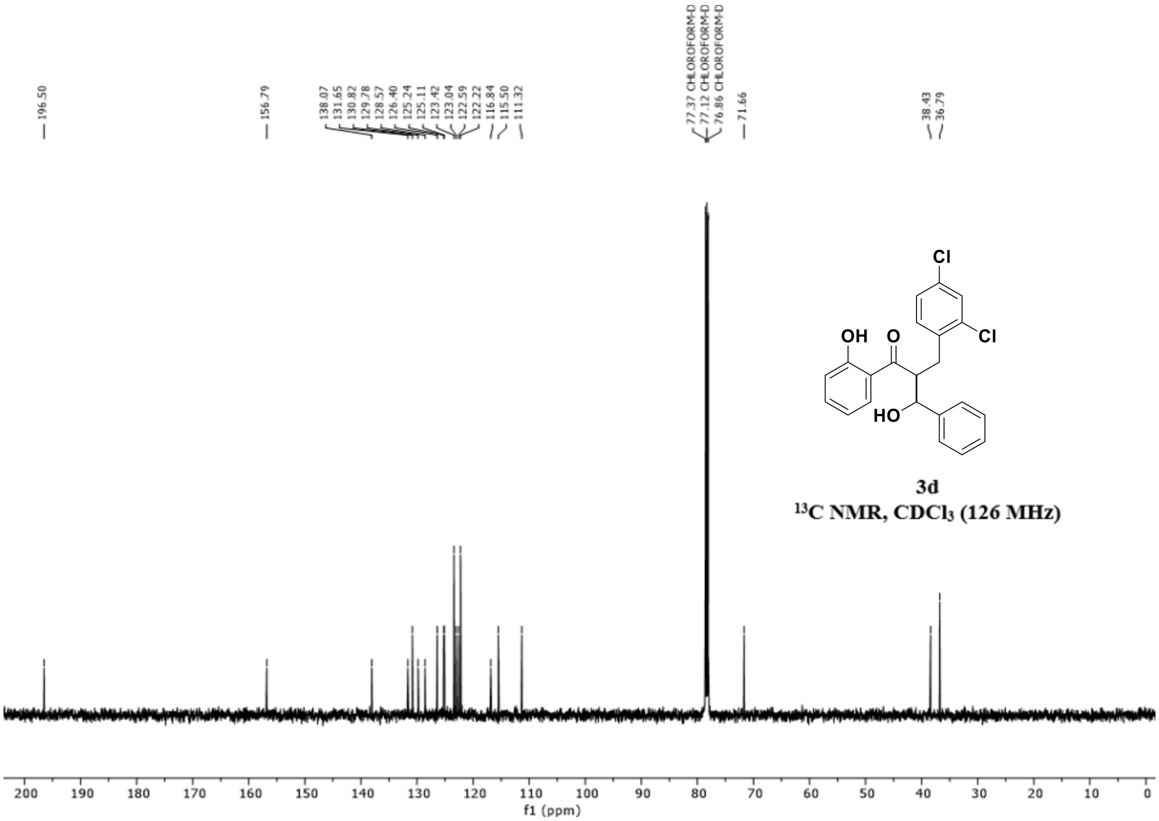


3c

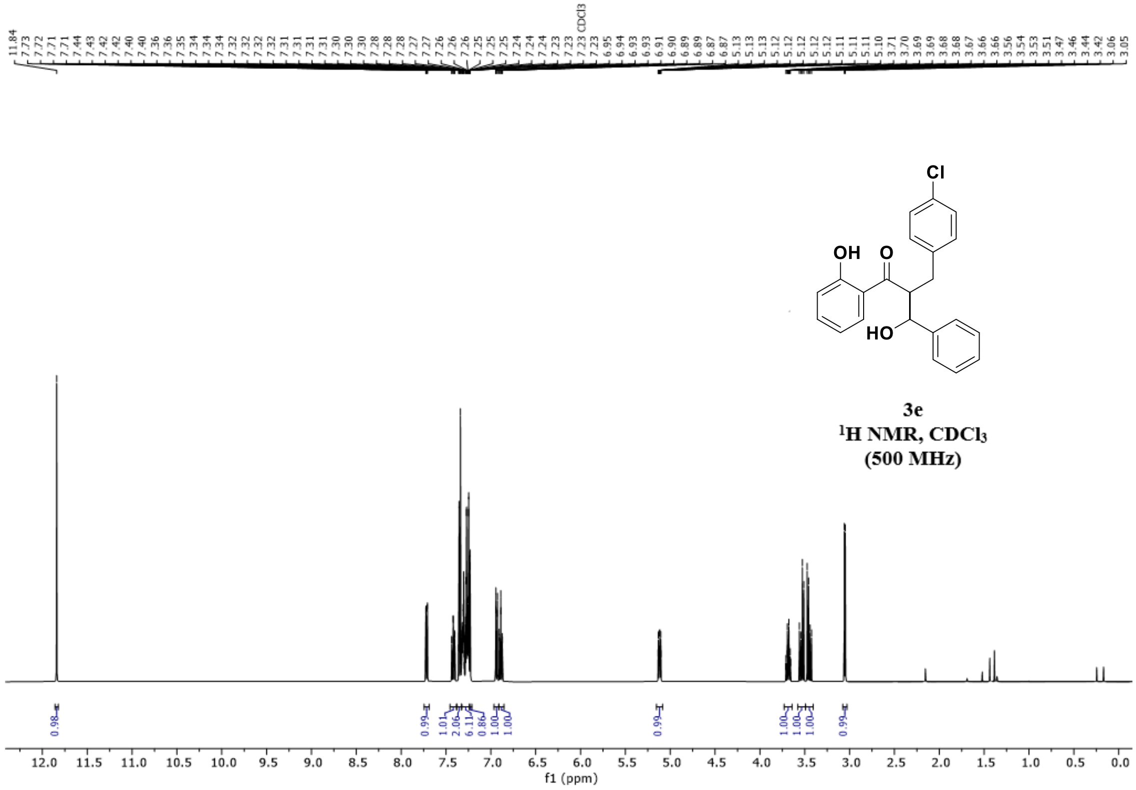
^{13}C NMR, $CDCl_3$ (126 MHz)



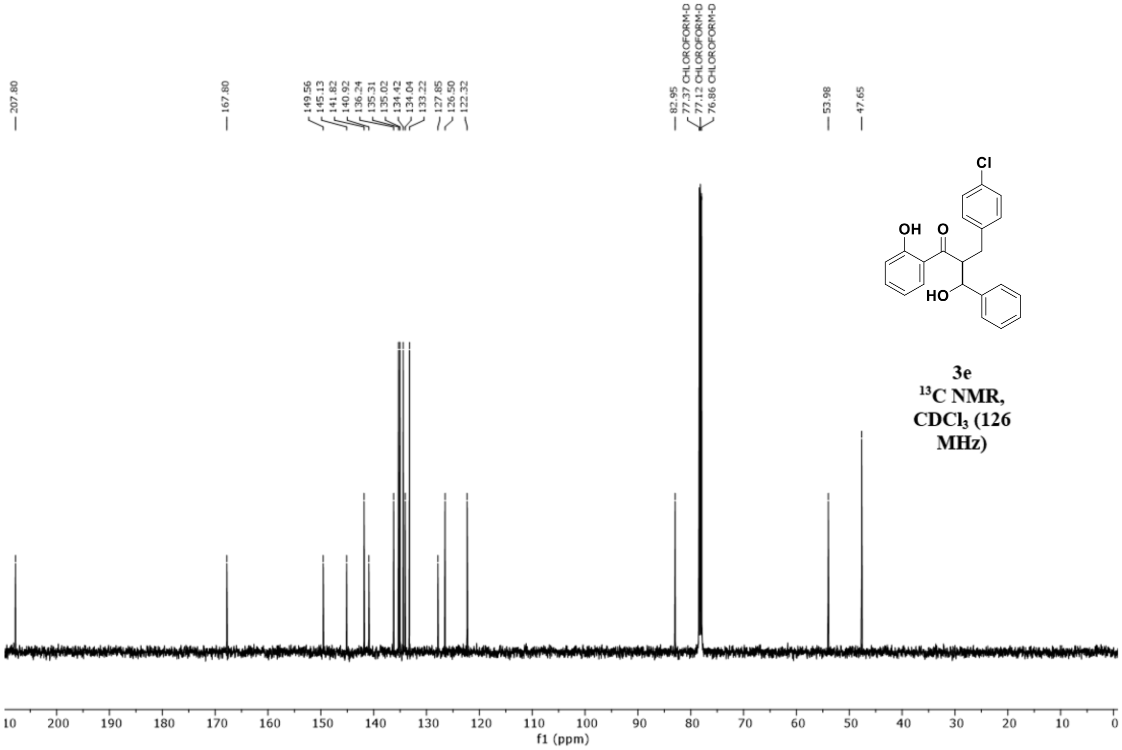
3d
^{1}H NMR, CDCl$_3$
(500 MHz)



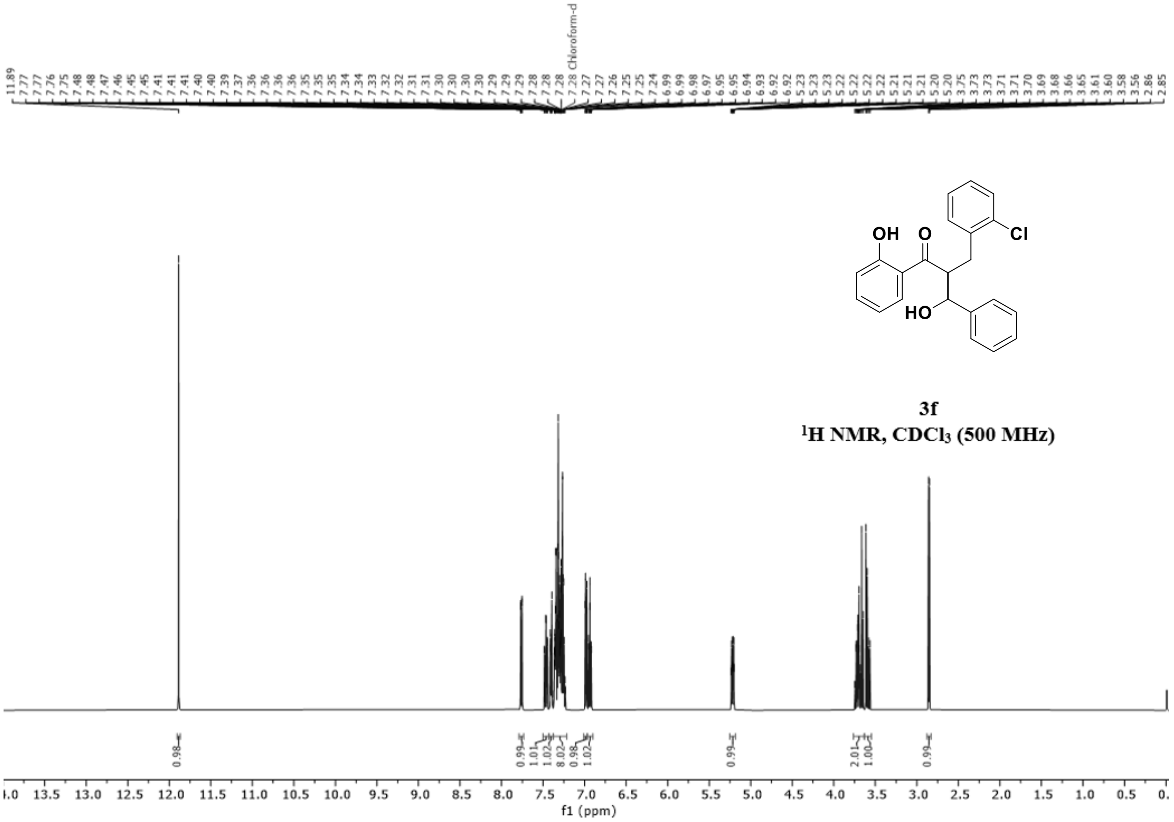
3d
^{13}C NMR, CDCl$_3$ (126 MHz)



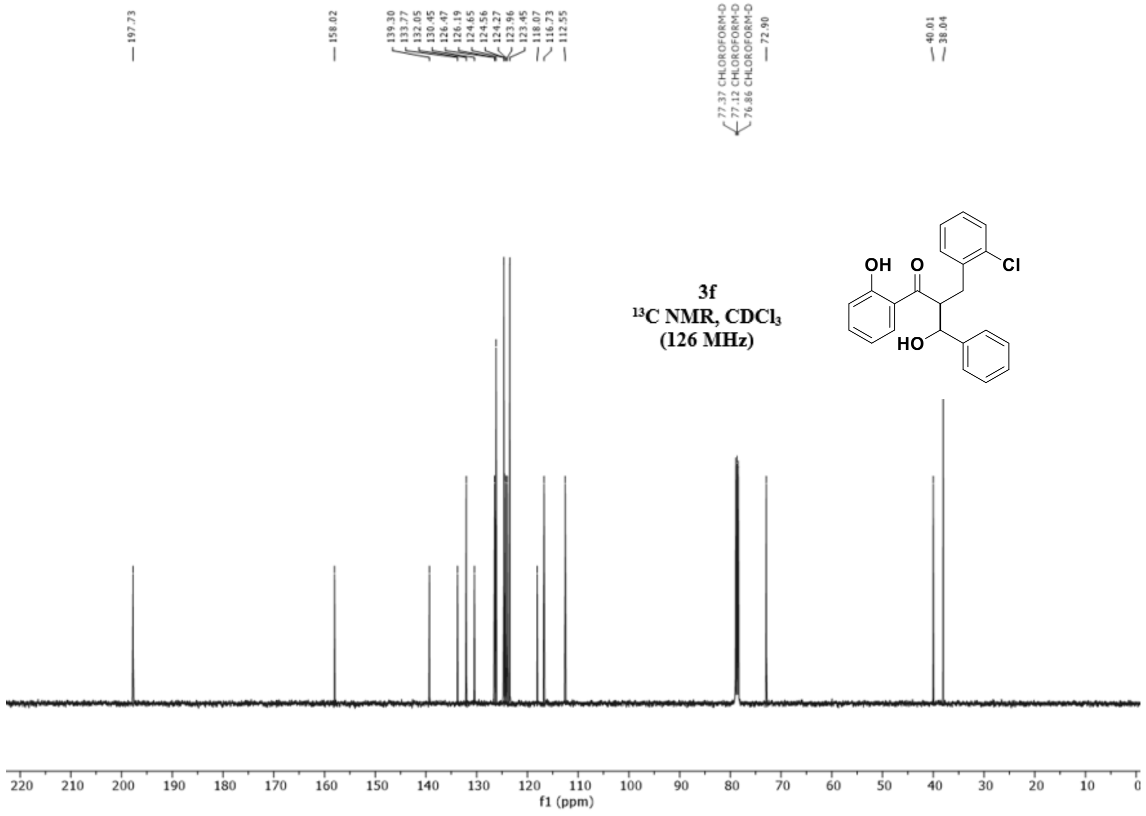
3e
^{1}H NMR, CDCl$_3$
(500 MHz)



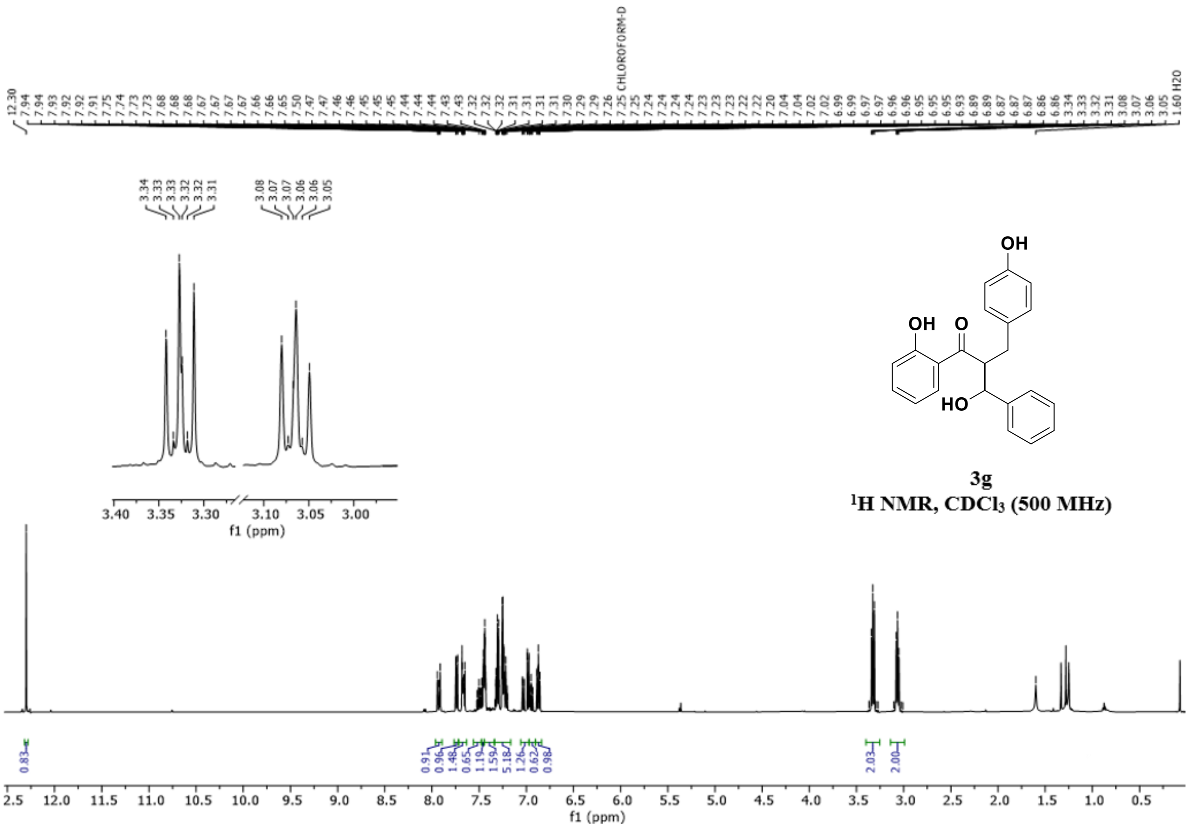
3e
^{13}C NMR,
CDCl$_3$ (126
MHz)



3f
^{1}H NMR, CDCl$_3$ (500 MHz)

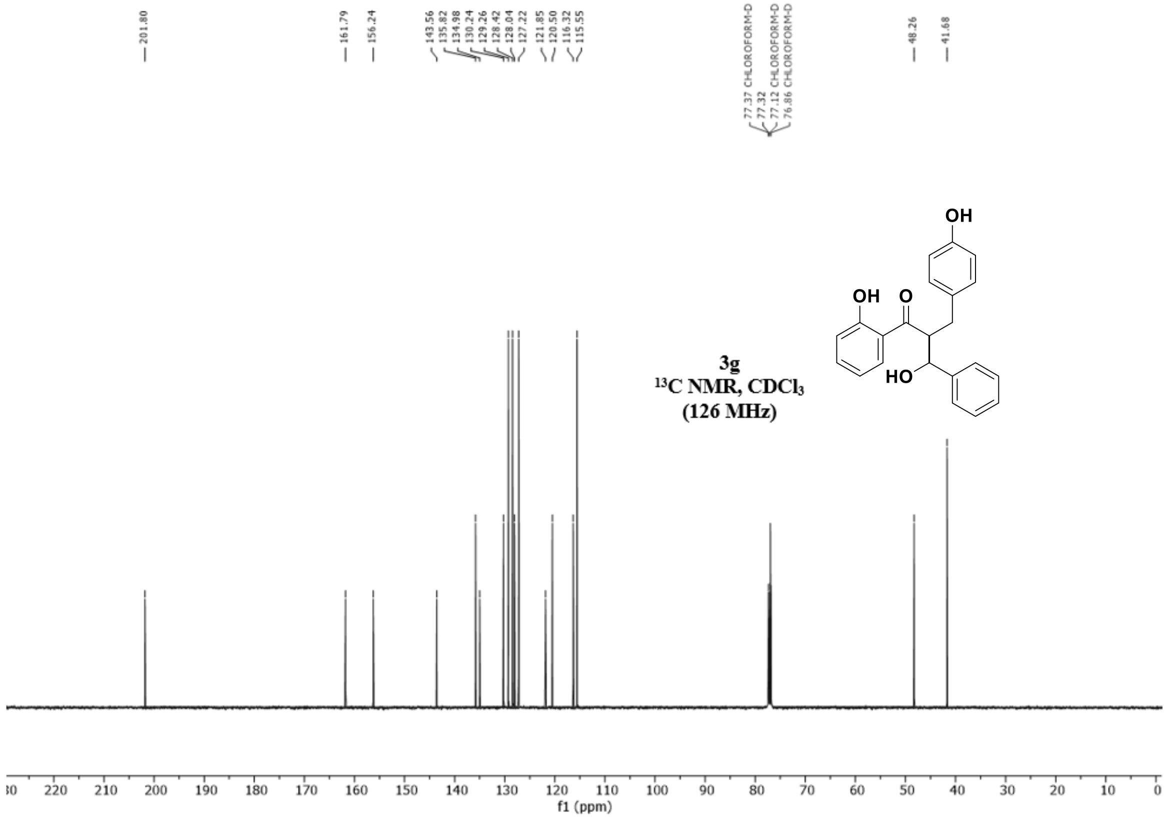


3f
^{13}C NMR, CDCl$_3$
(126 MHz)



3g

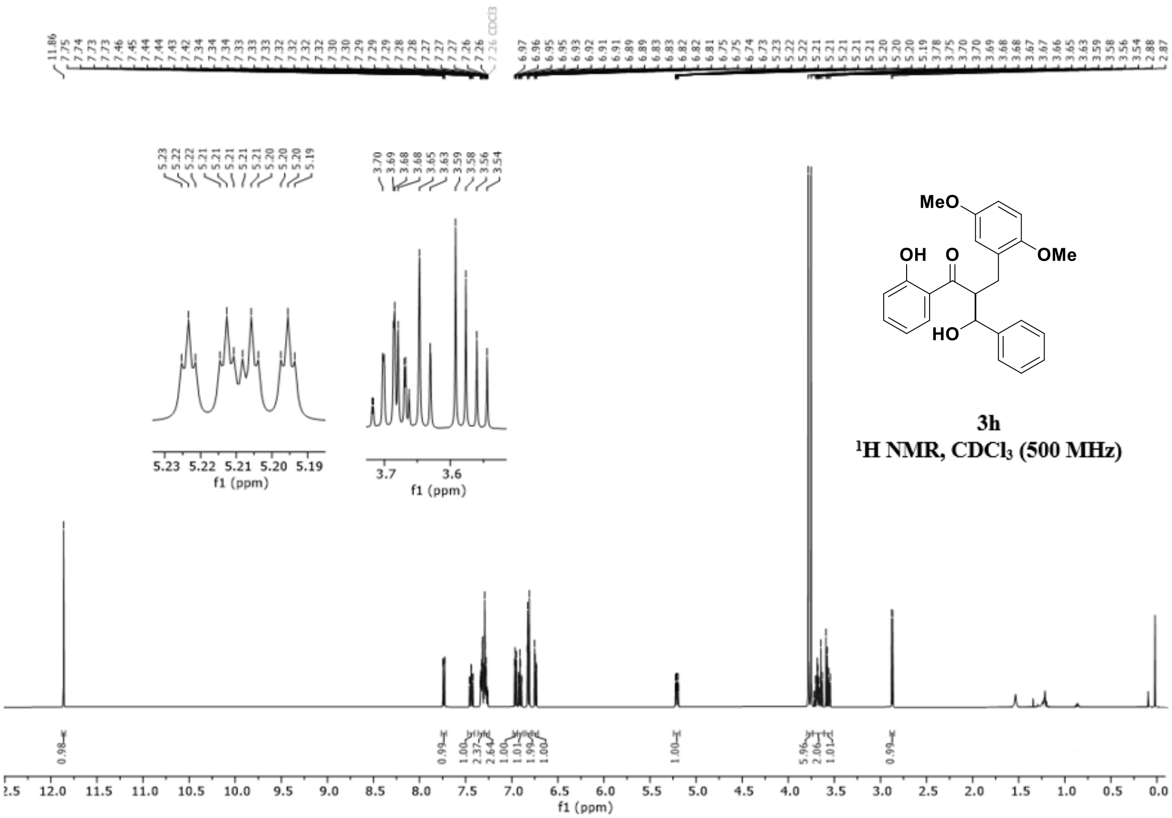
^{1}H NMR, CDCl$_3$ (500 MHz)



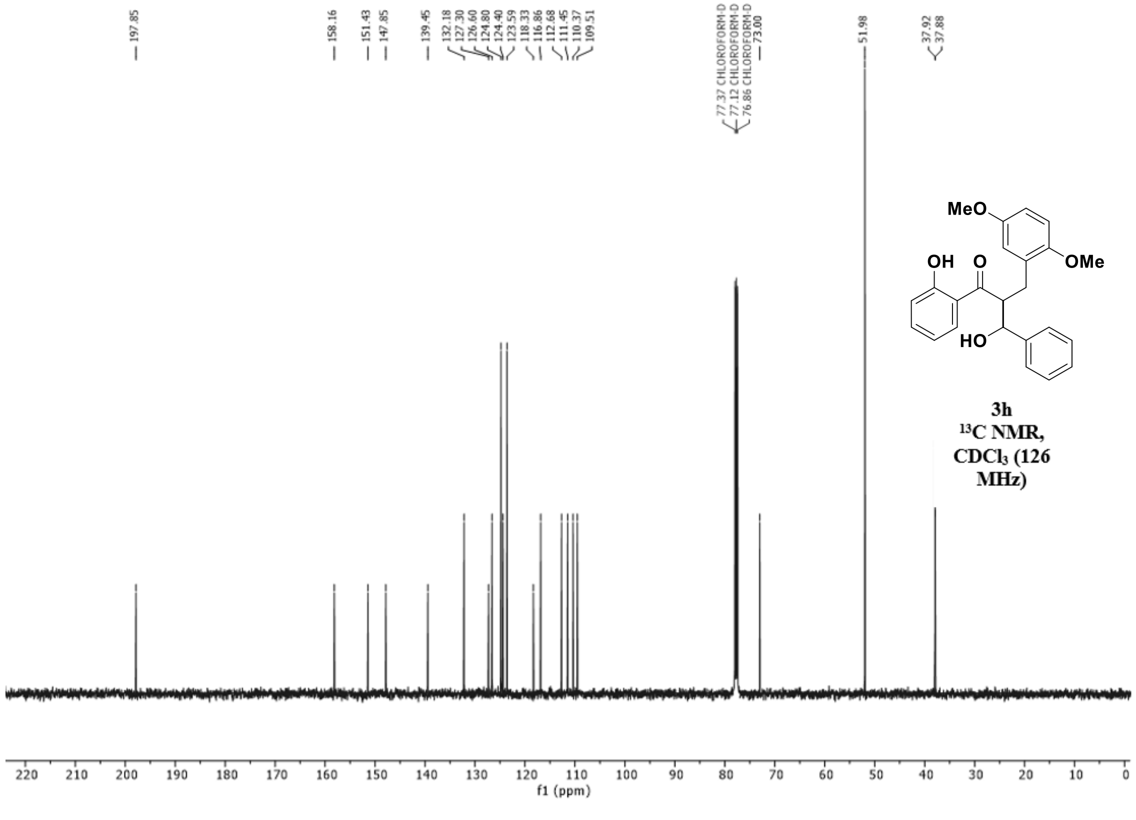
3g

^{13}C NMR, CDCl$_3$

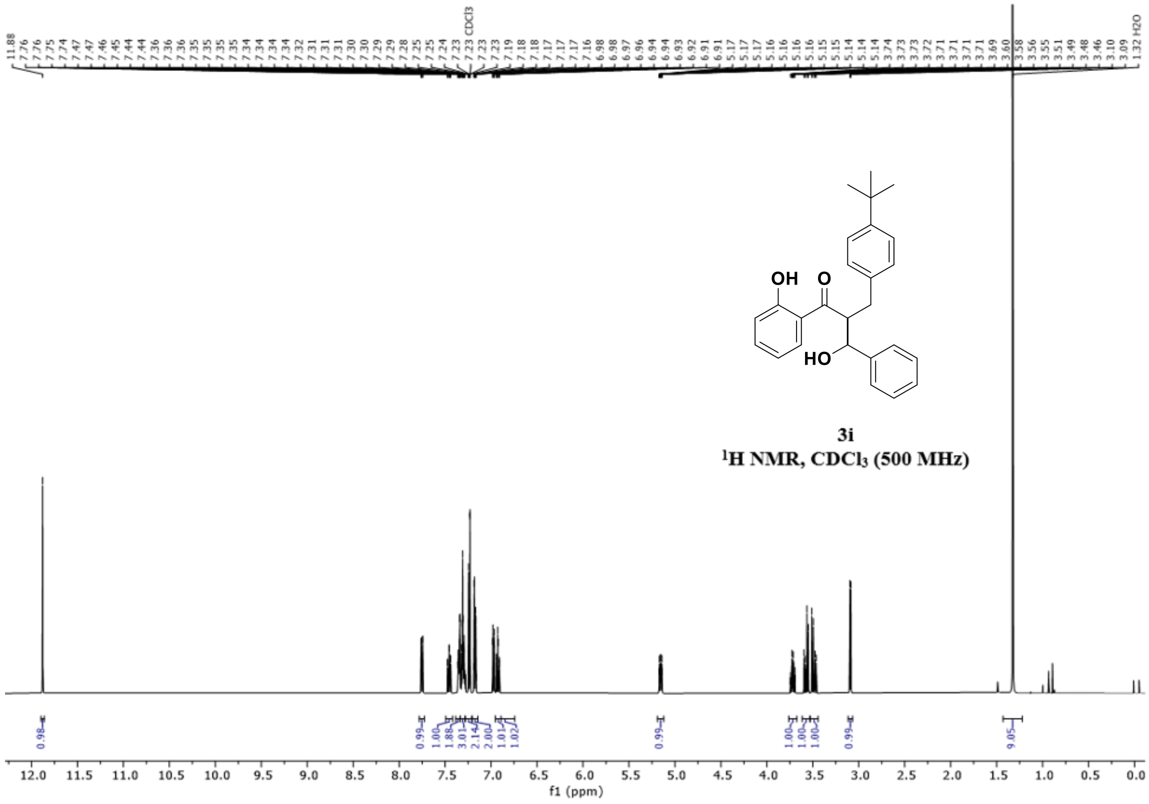
(126 MHz)



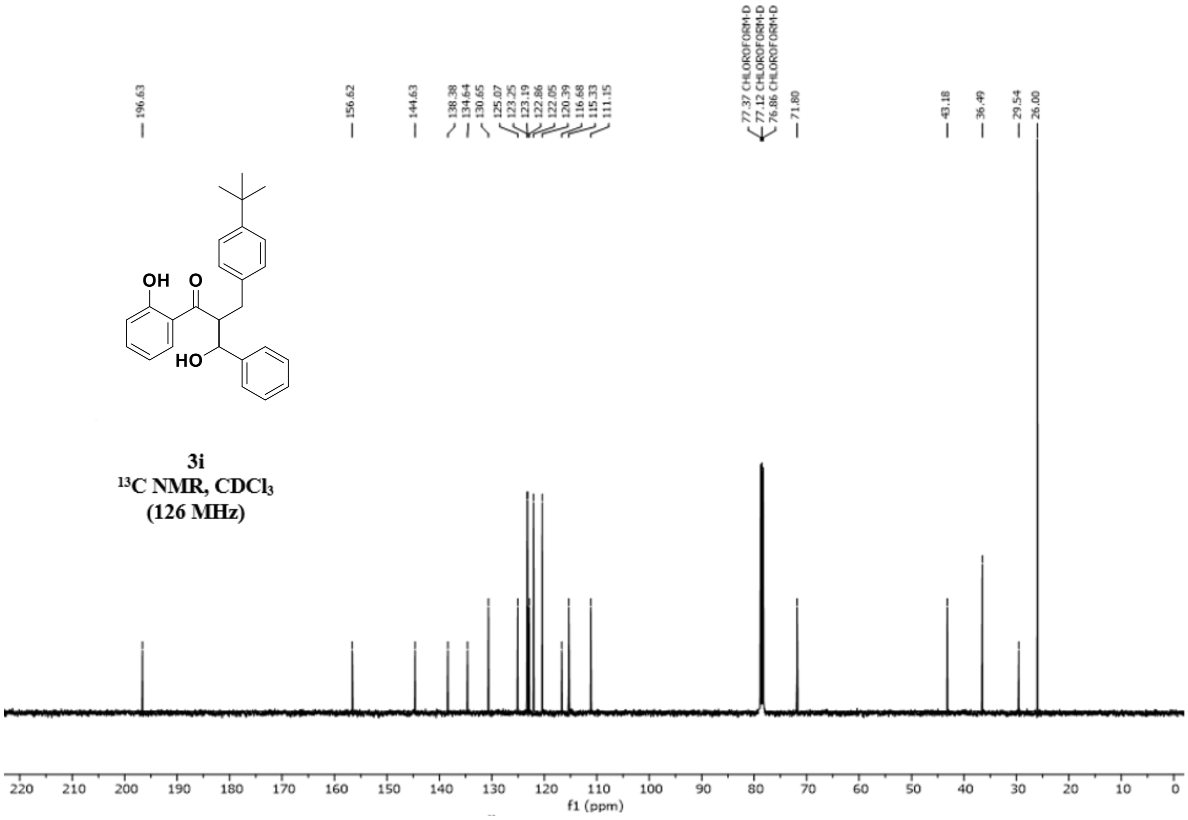
3h
^{1}H NMR, CDCl$_3$ (500 MHz)



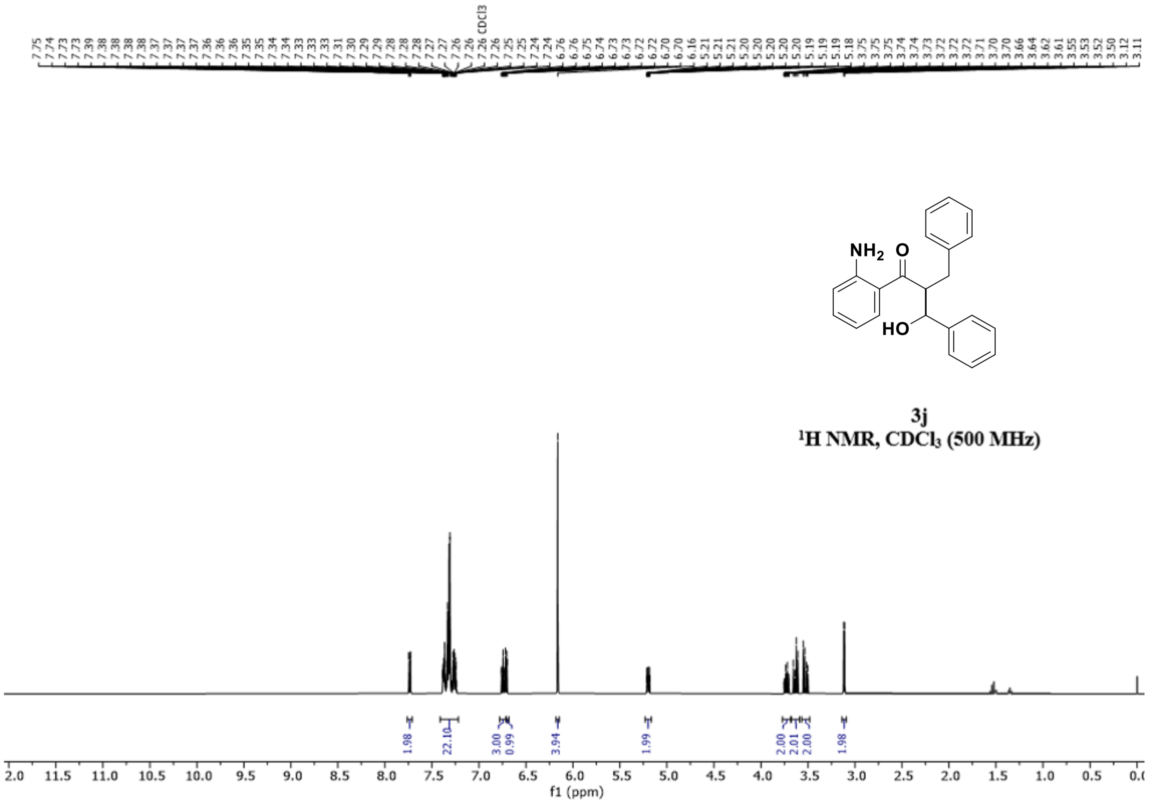
3h
^{13}C NMR,
CDCl$_3$ (126
MHz)



3i
^{1}H NMR, CDCl$_3$ (500 MHz)

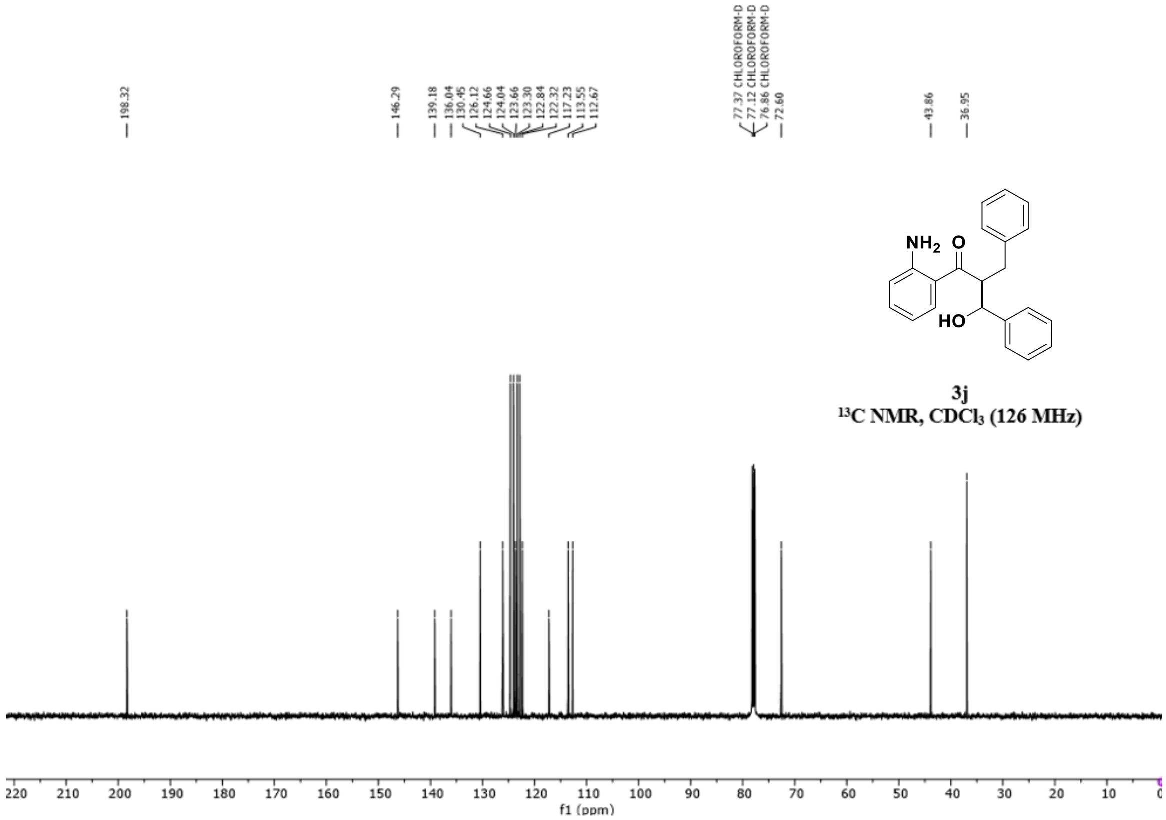


3i
^{13}C NMR, CDCl$_3$
(126 MHz)



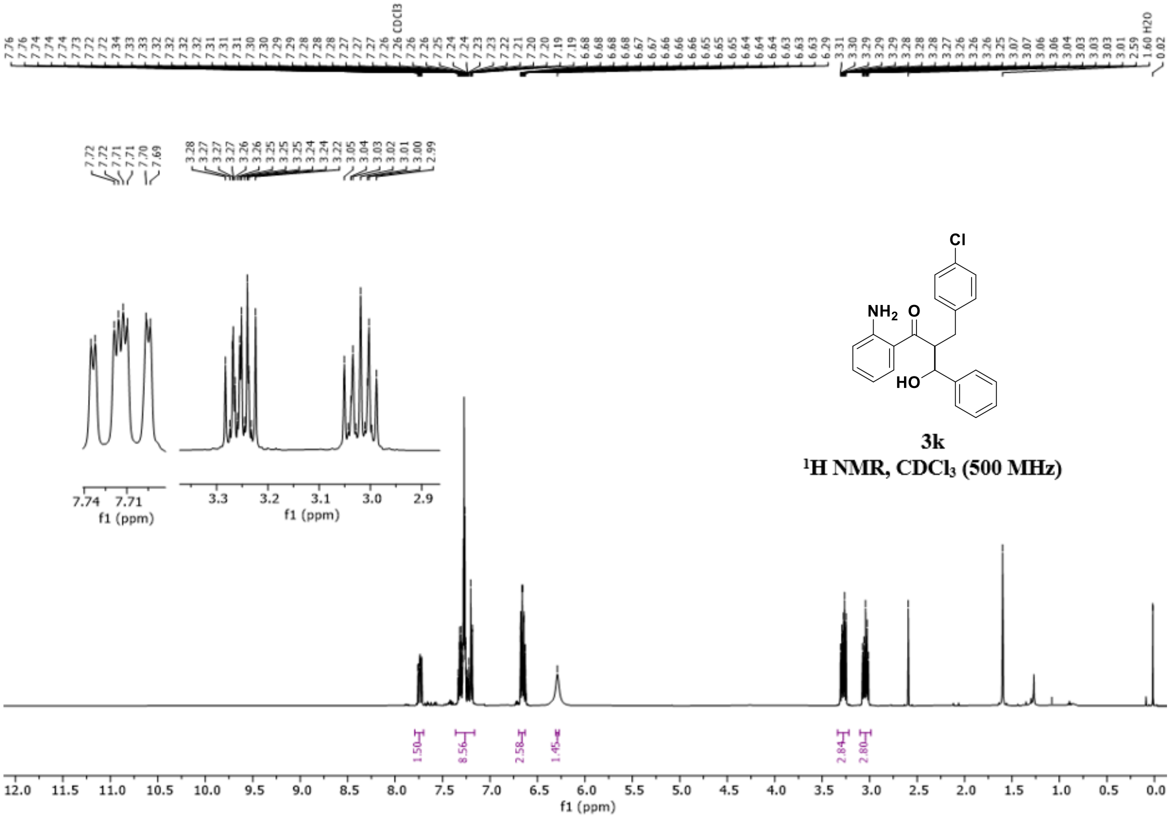
3j

^{1}H NMR, $CDCl_3$ (500 MHz)



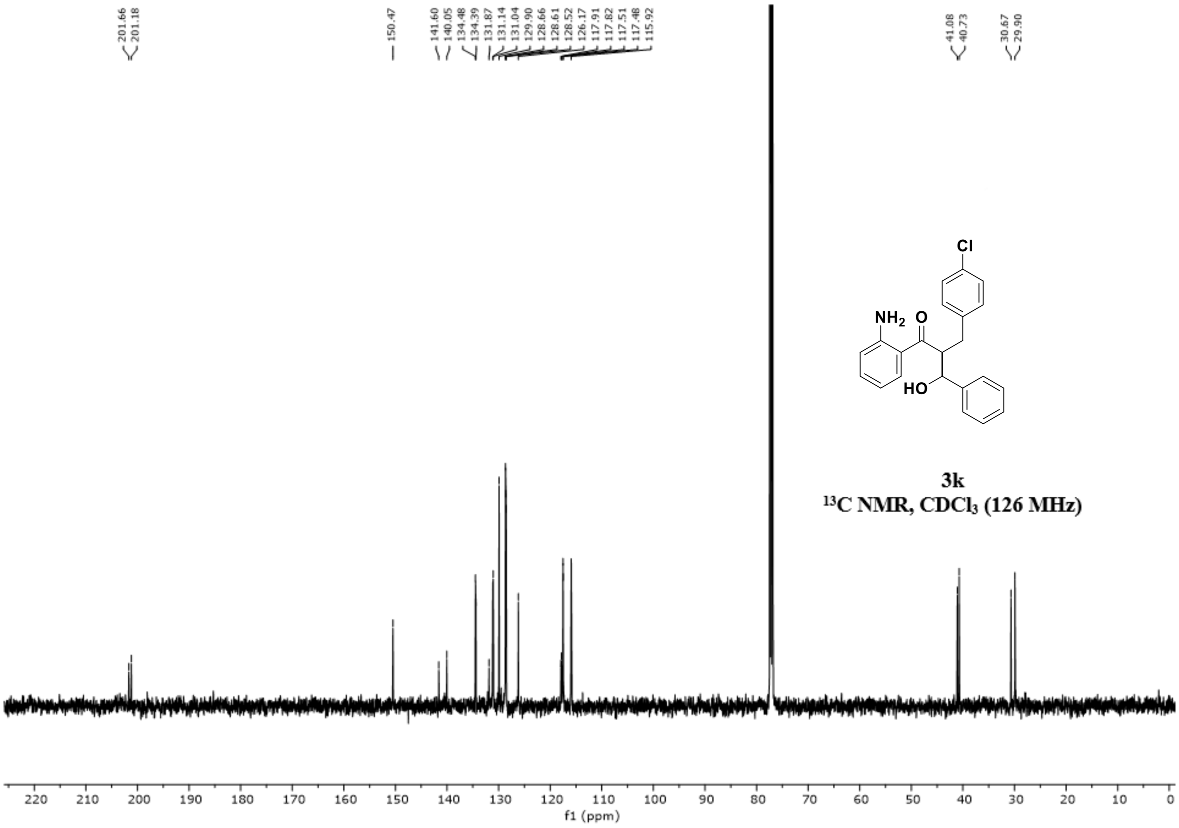
3j

^{13}C NMR, $CDCl_3$ (126 MHz)



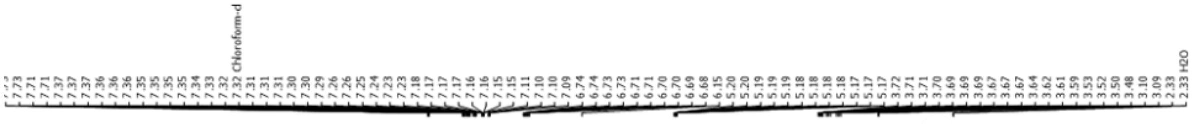
3k

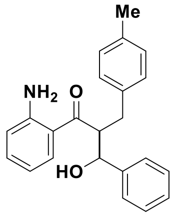
^{1}H NMR, $CDCl_3$ (500 MHz)



3k

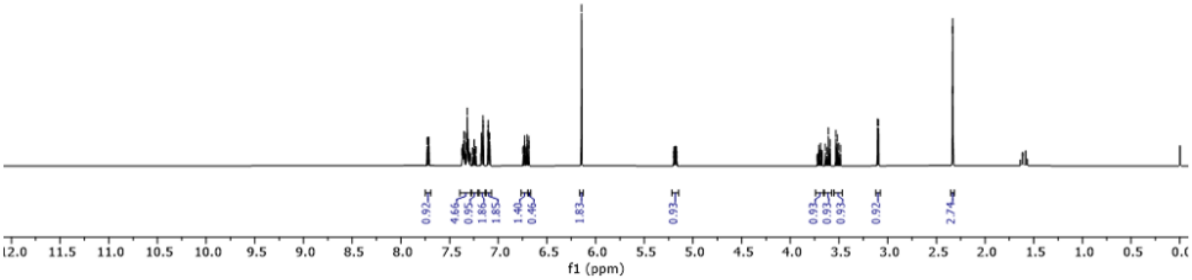
^{13}C NMR, $CDCl_3$ (126 MHz)

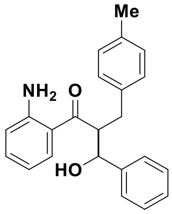




3l

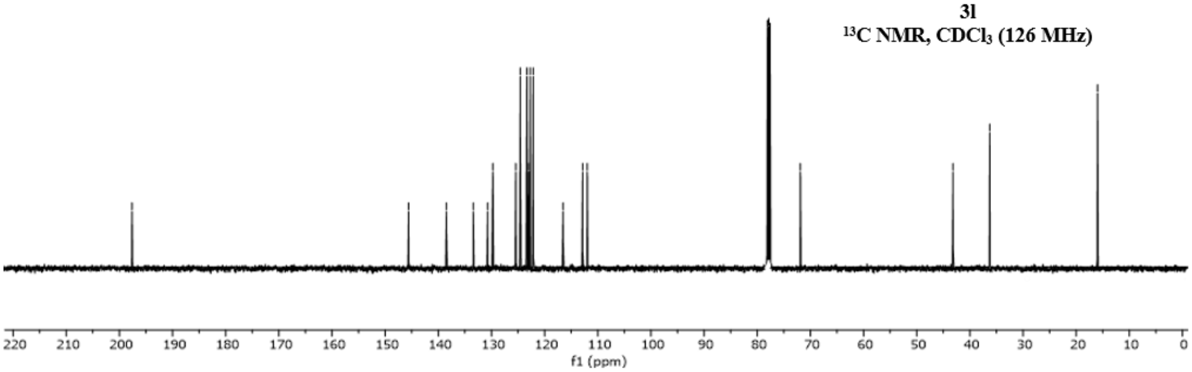
^{1}H NMR, CDCl$_3$ (500 MHz)

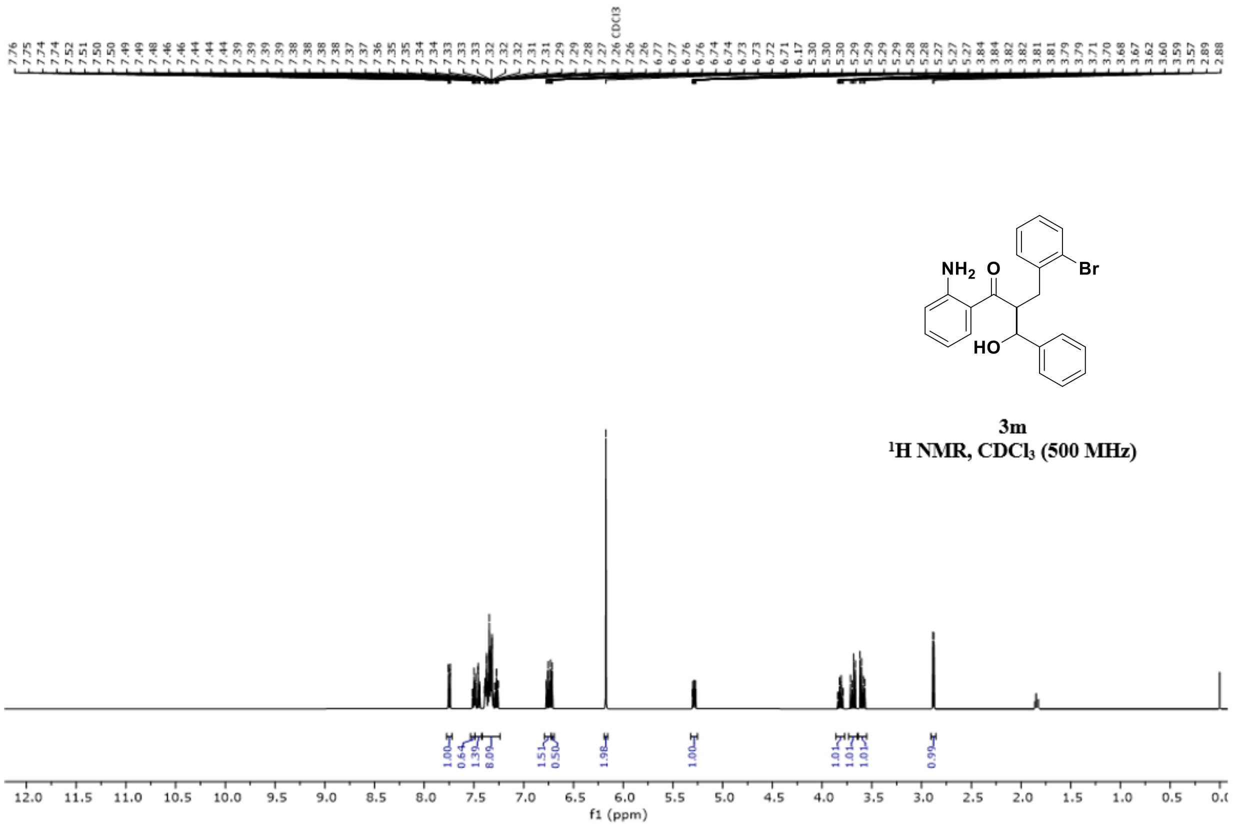




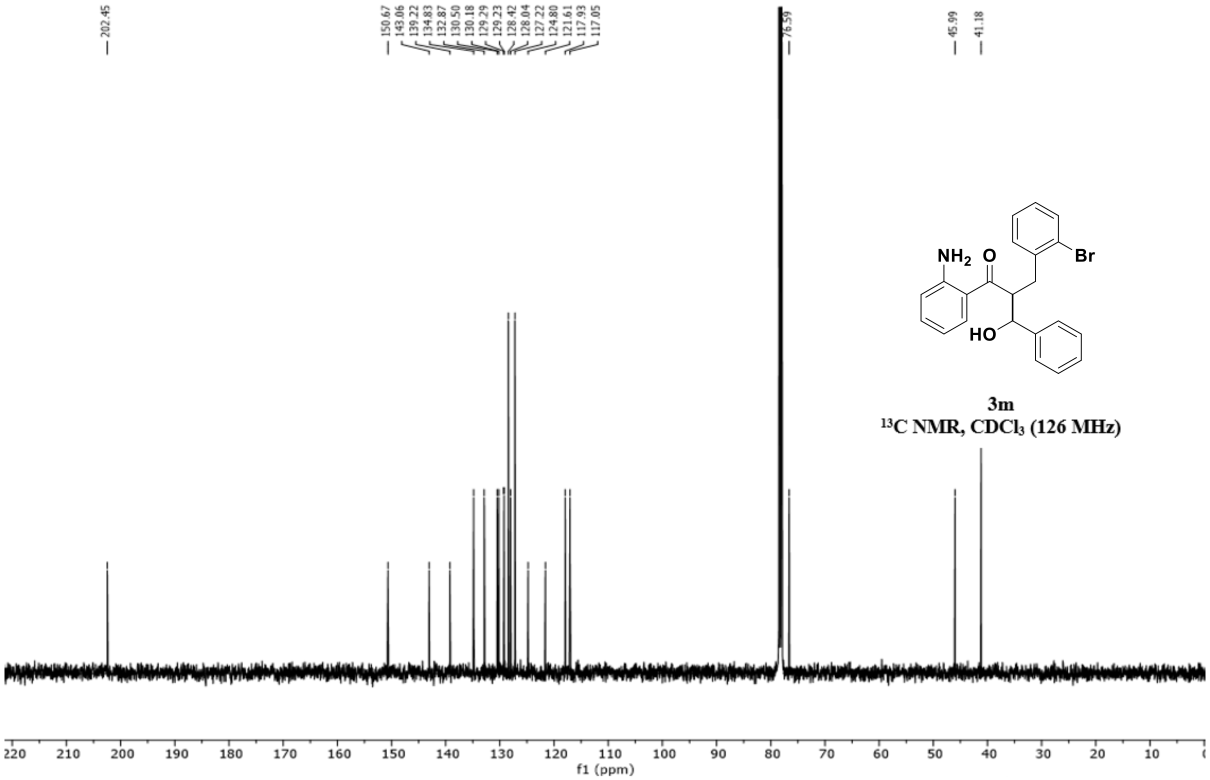
3l

^{13}C NMR, CDCl$_3$ (126 MHz)

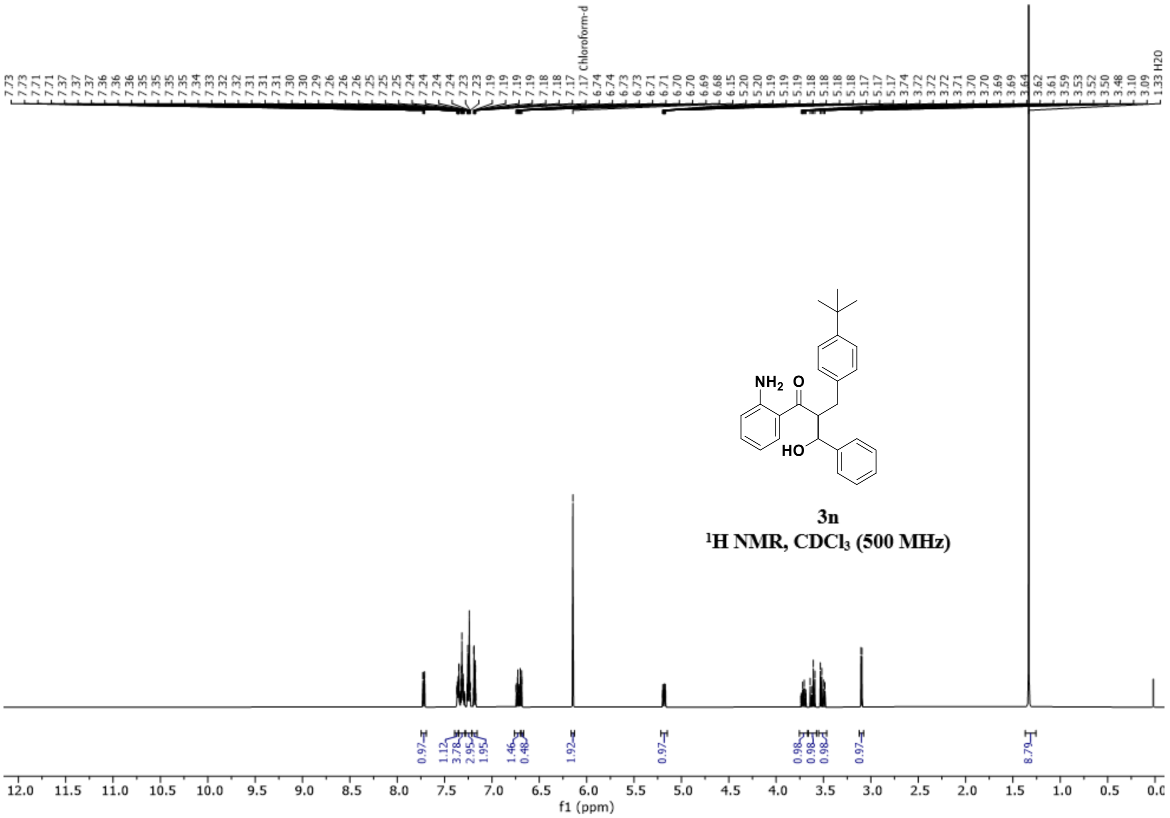


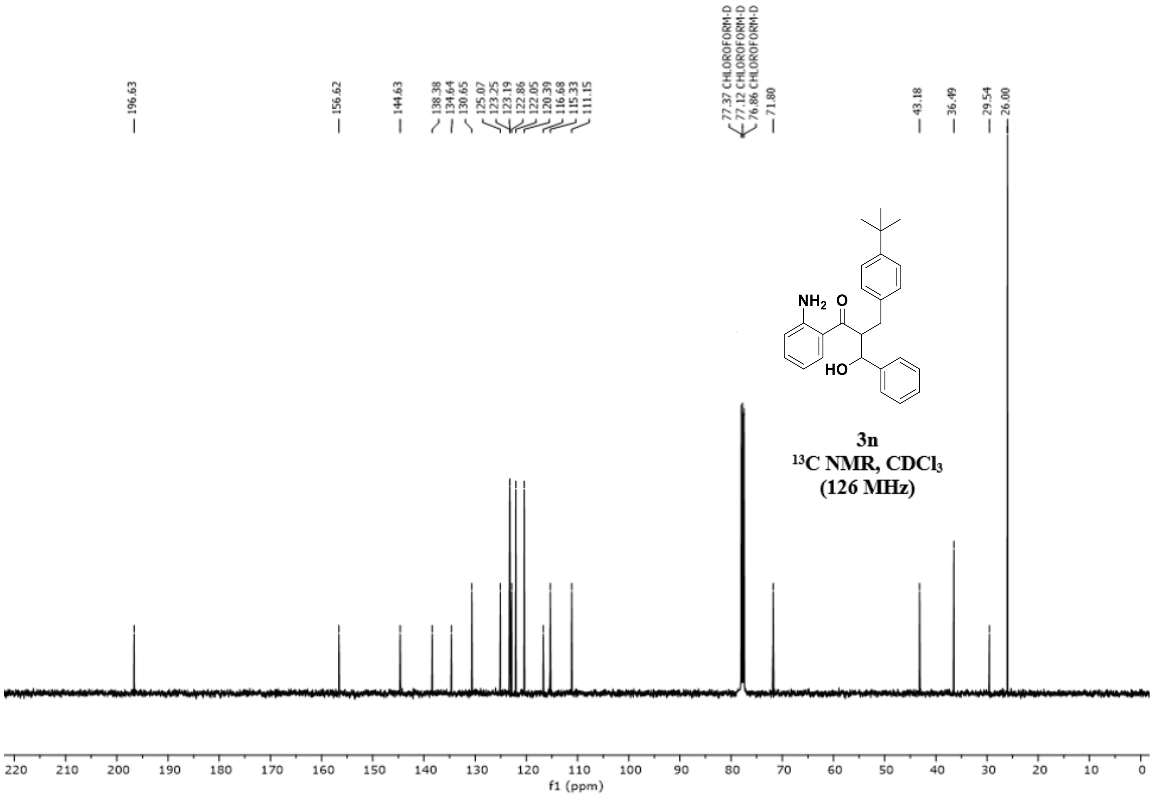


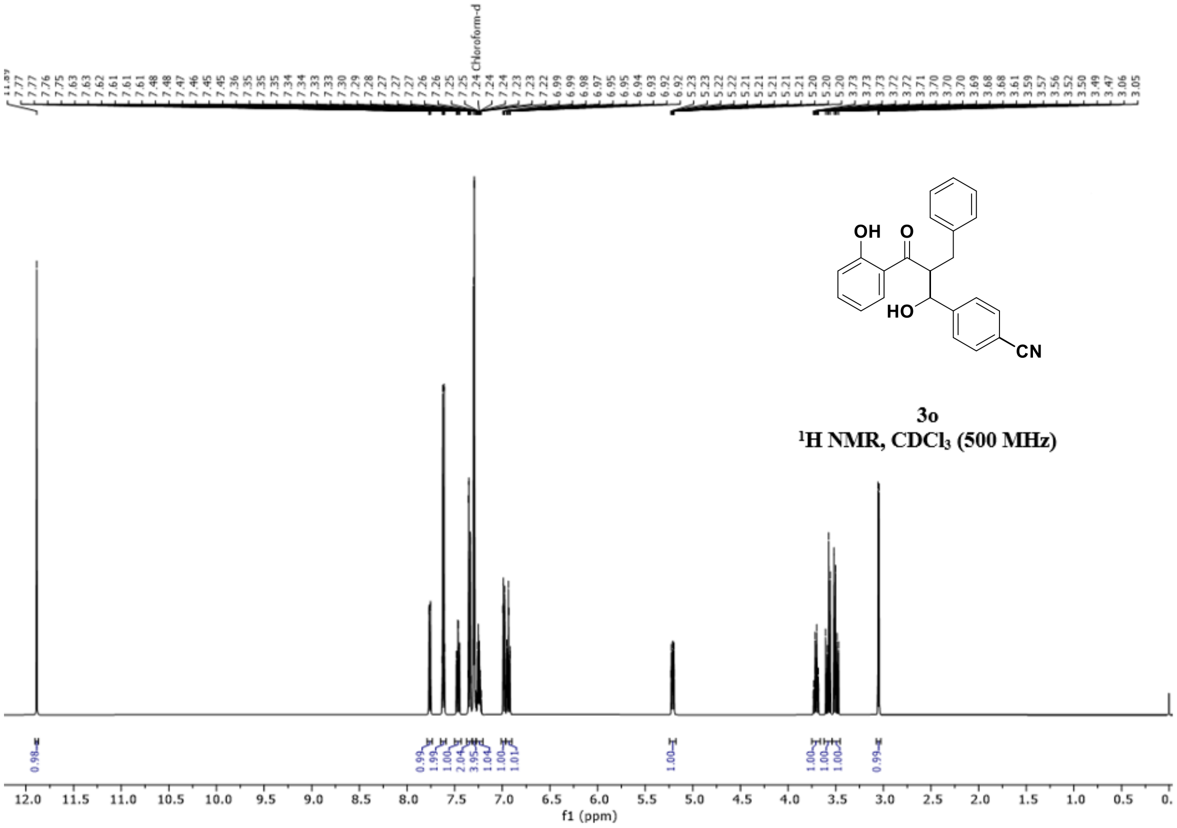
3m
^{1}H NMR, CDCl$_3$ (500 MHz)



3m
^{13}C NMR, CDCl$_3$ (126 MHz)

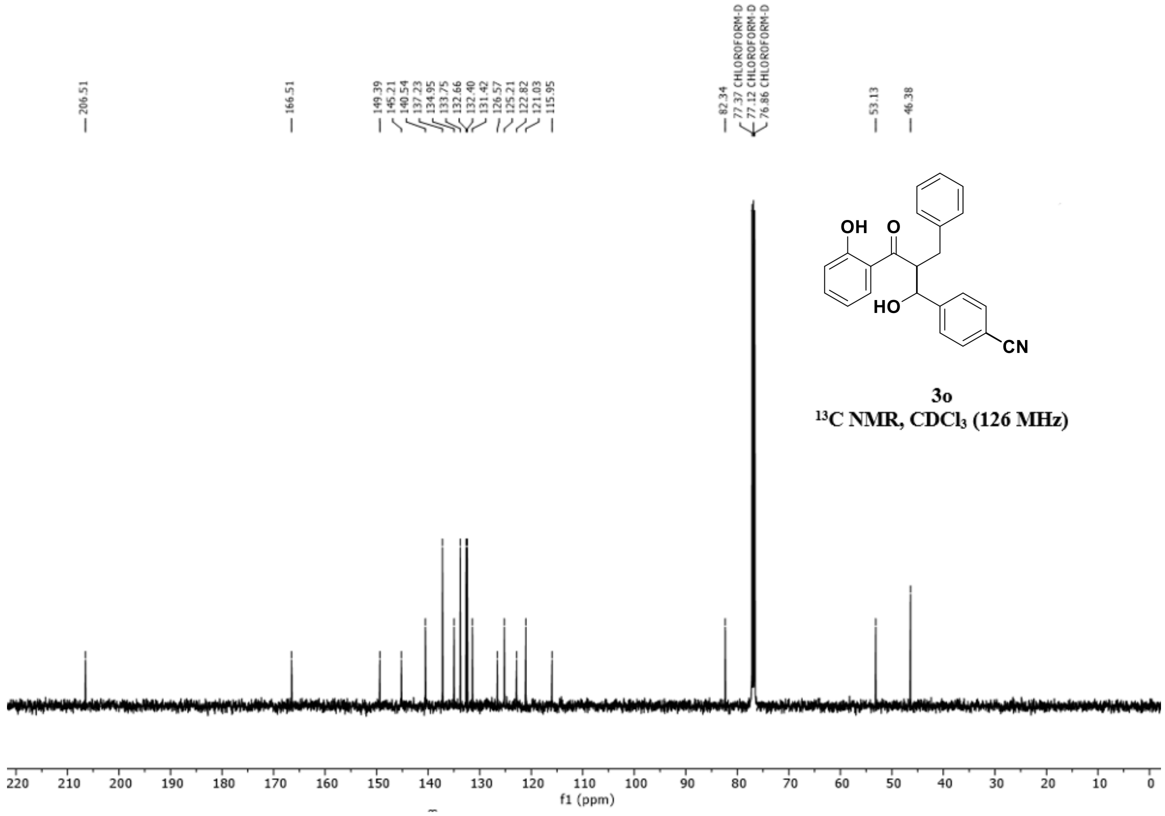

3n
^1H NMR, $CDCl_3$ (500 MHz)


3n
^{13}C NMR, $CDCl_3$
(126 MHz)



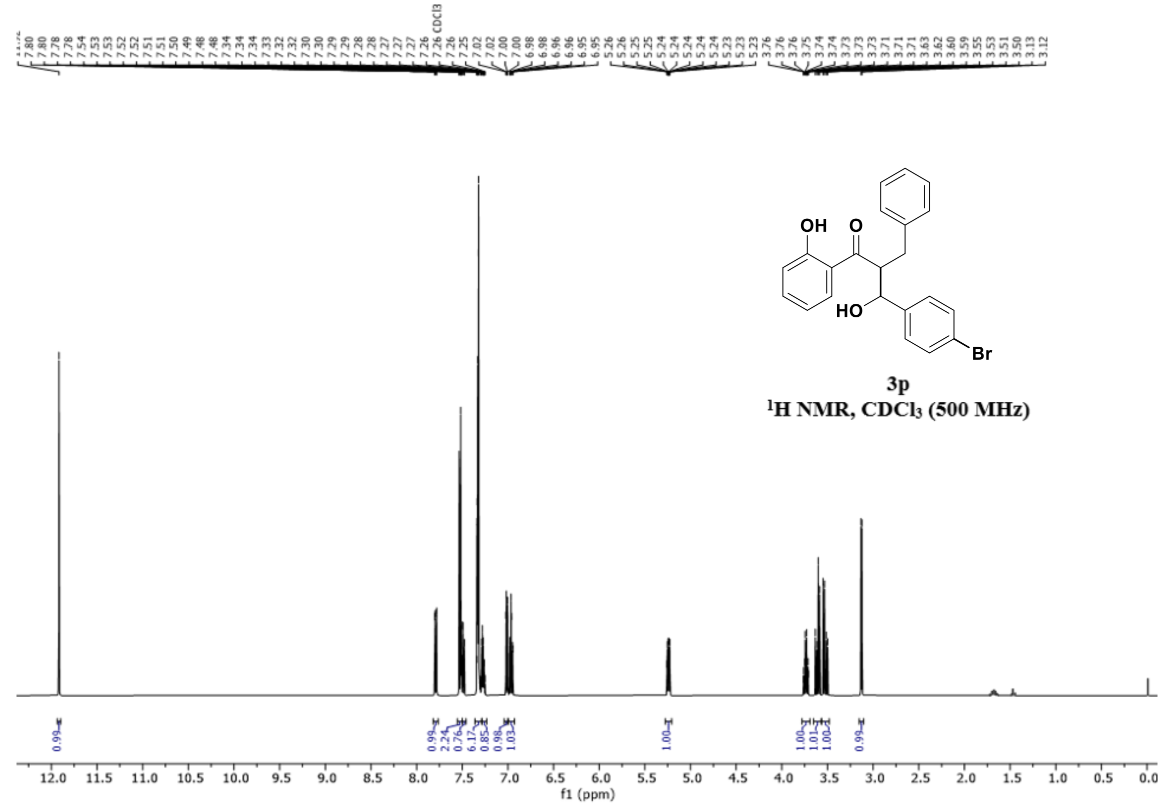
3o

^{1}H NMR, CDCl$_3$ (500 MHz)

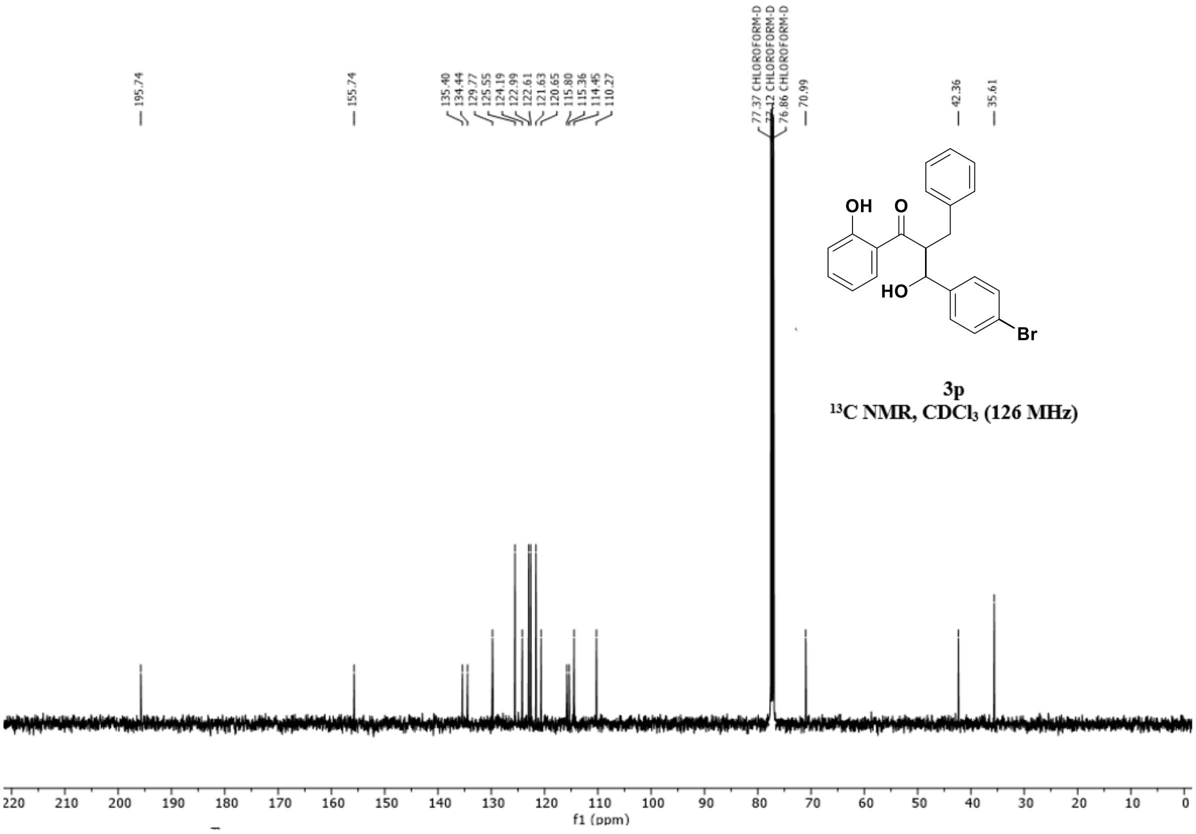


3o

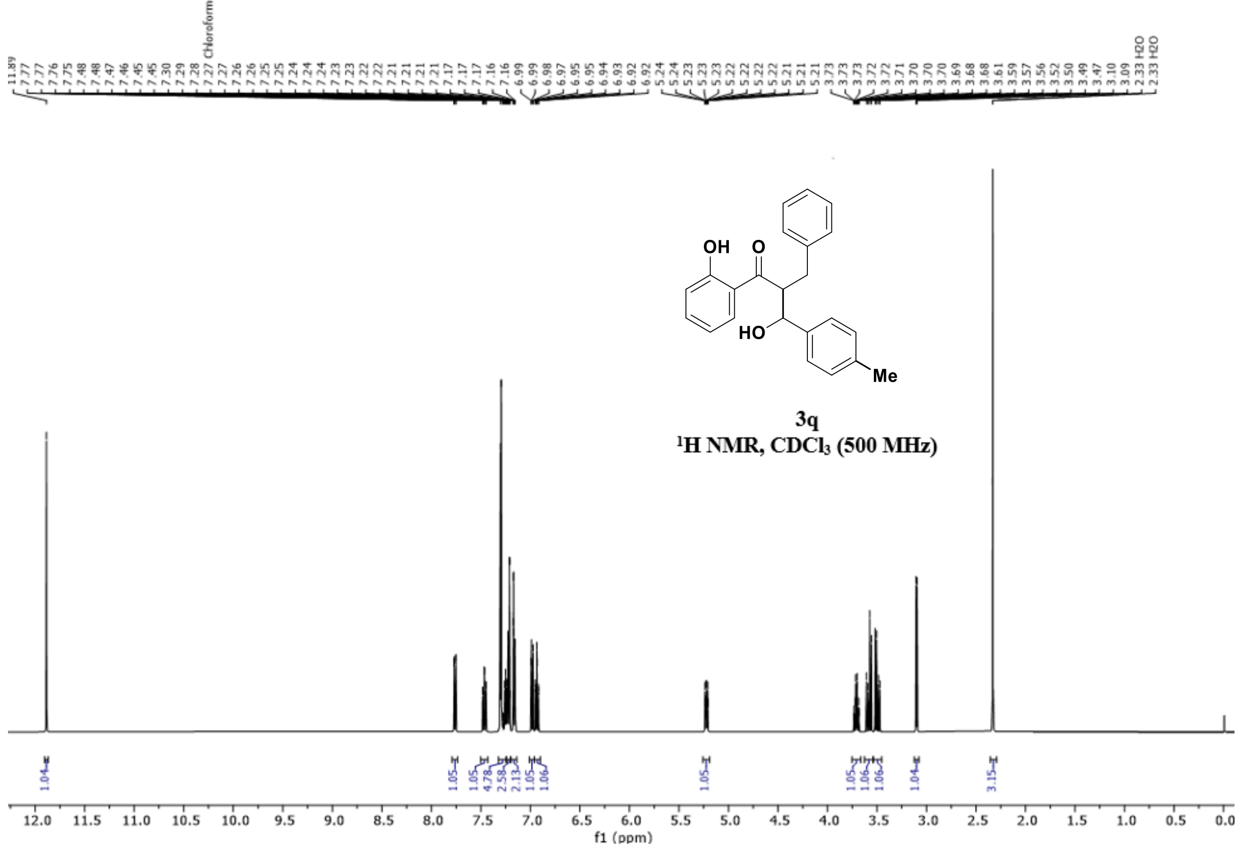
^{13}C NMR, CDCl$_3$ (126 MHz)



3p
^{1}H NMR, CDCl$_3$ (500 MHz)

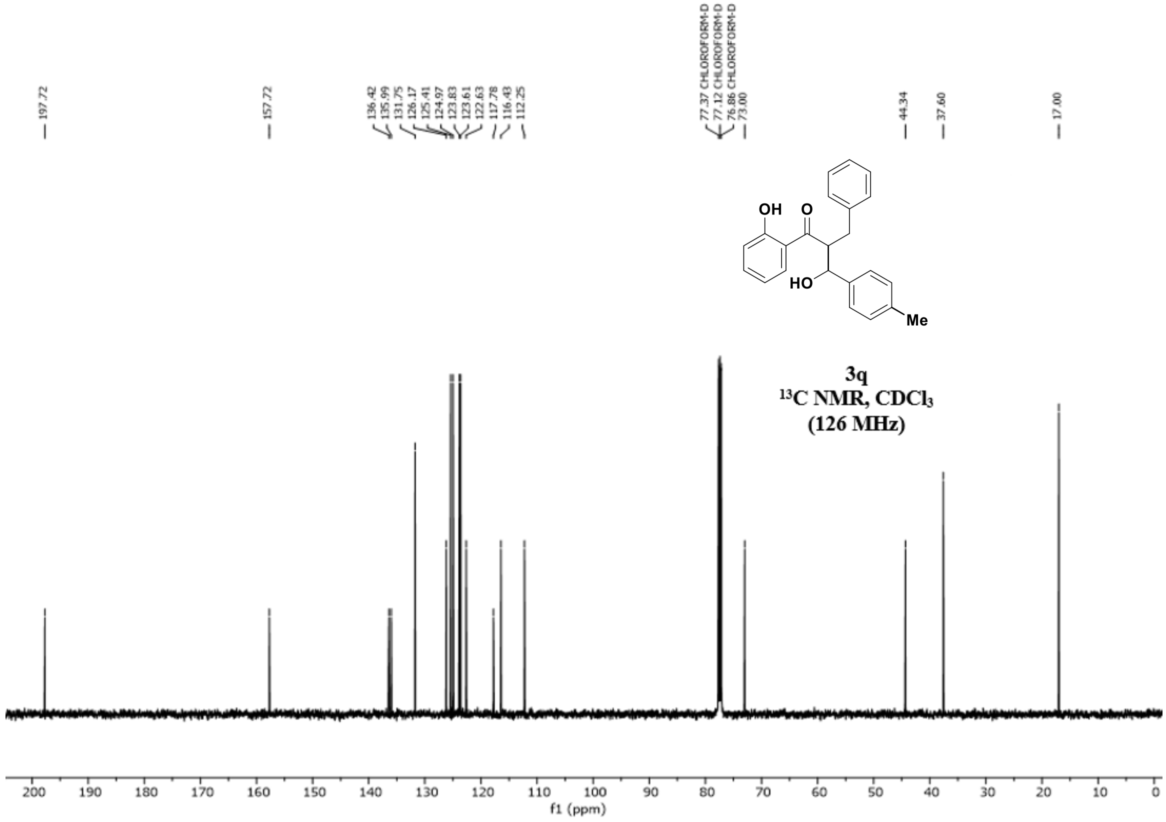


3p
^{13}C NMR, CDCl$_3$ (126 MHz)



3q

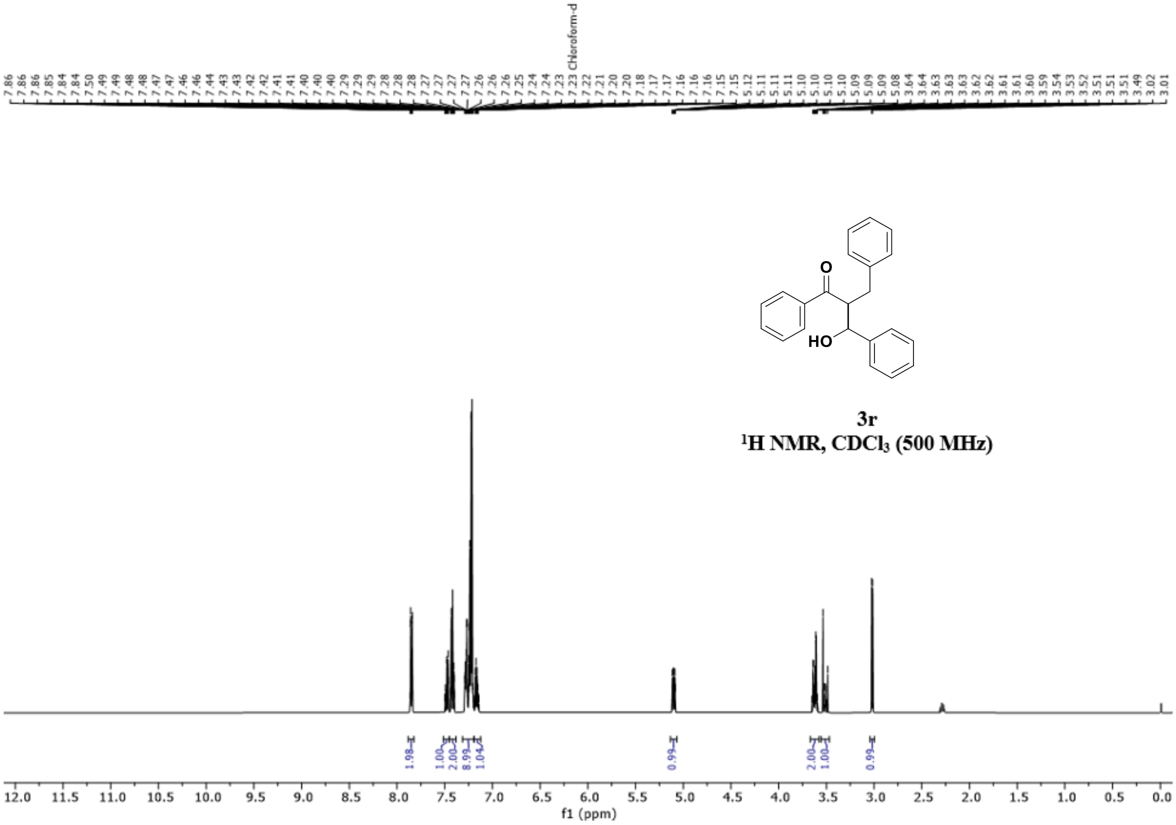
^{1}H NMR, CDCl$_3$ (500 MHz)



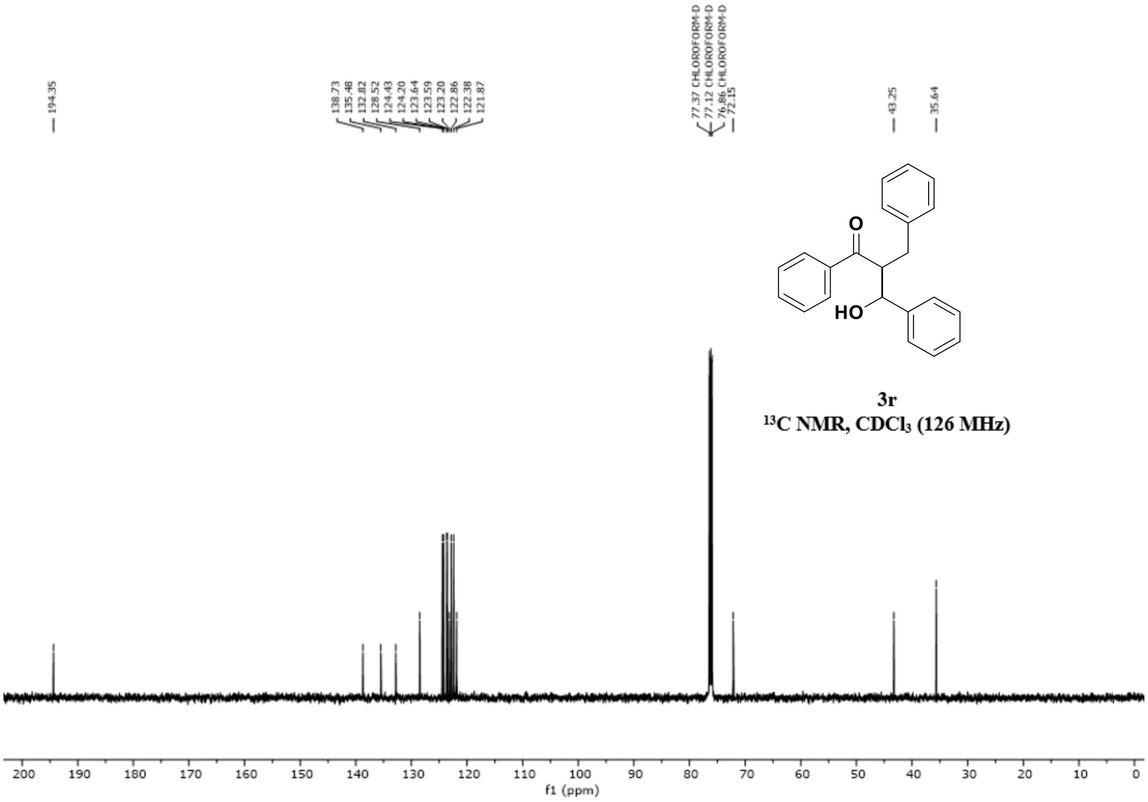
3q

^{13}C NMR, CDCl$_3$

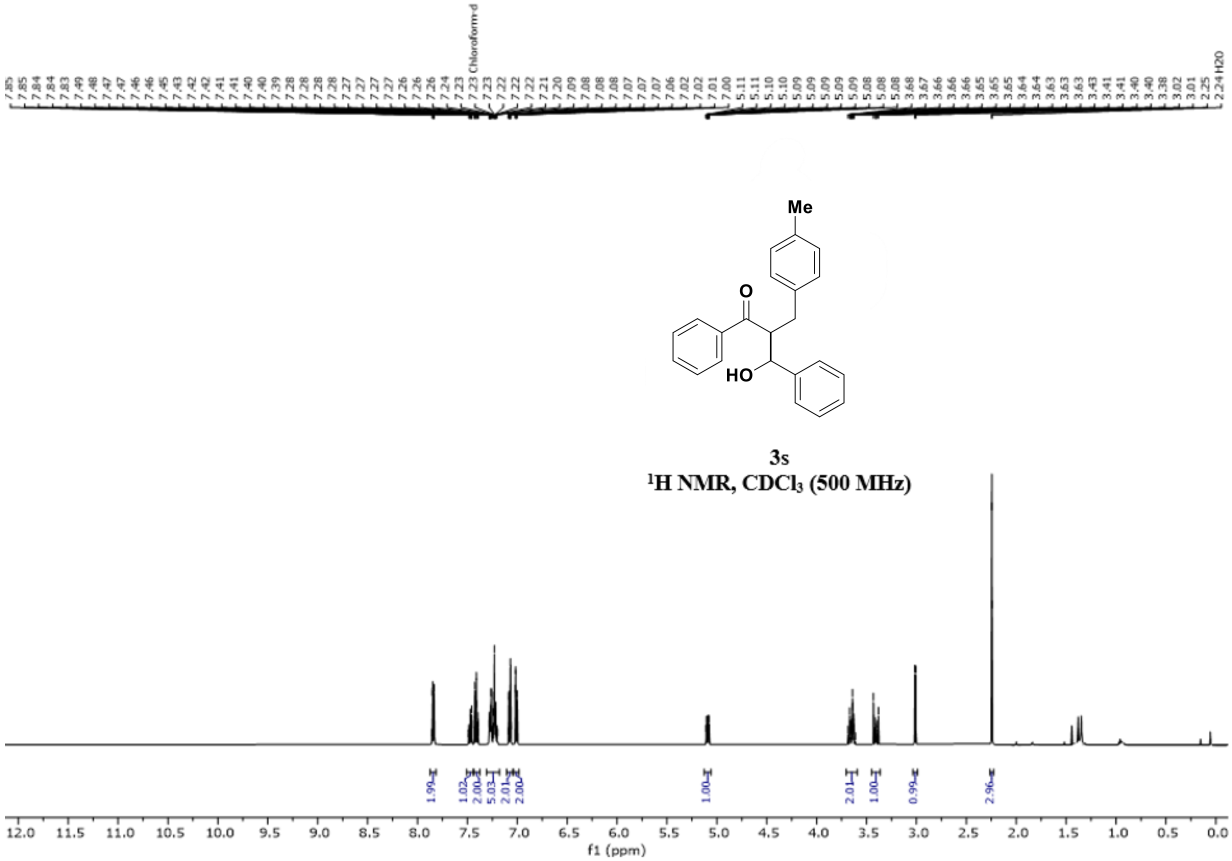
(126 MHz)



3r
^{1}H NMR, CDCl$_3$ (500 MHz)

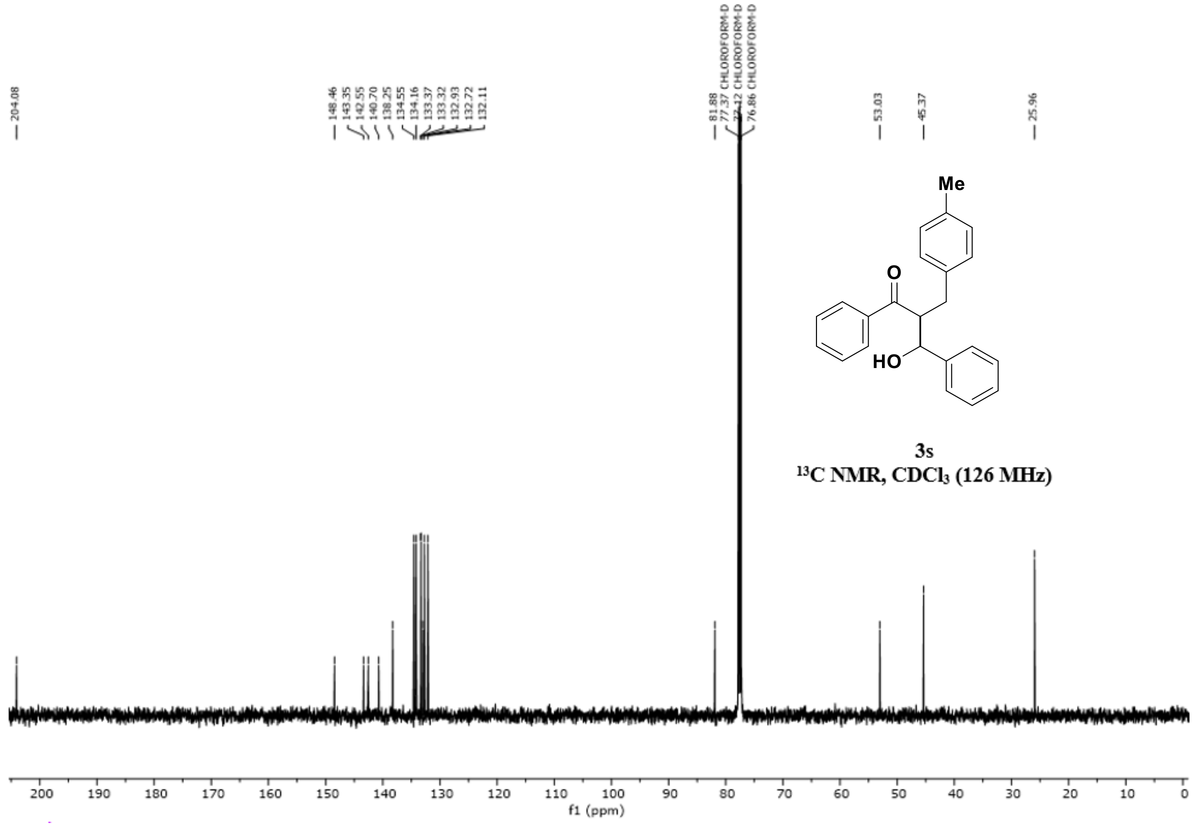


3r
^{13}C NMR, CDCl$_3$ (126 MHz)



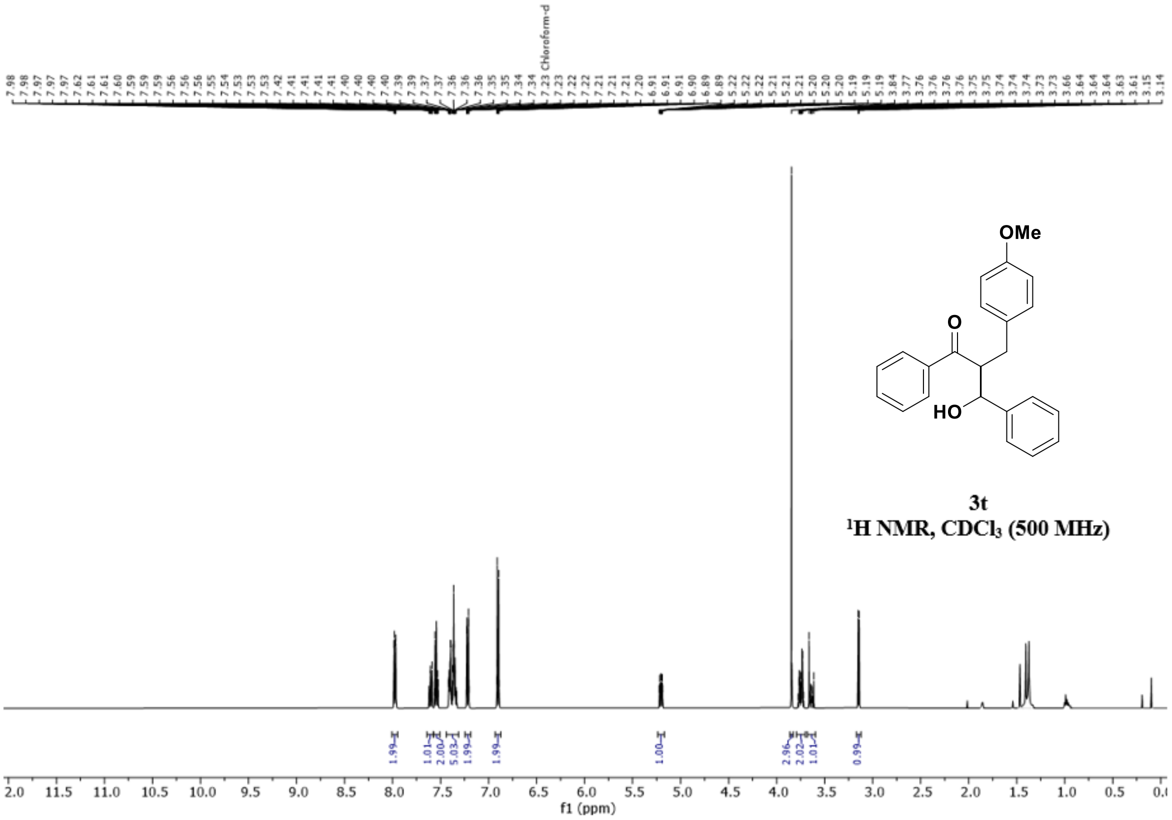
3s

^{1}H NMR, CDCl$_3$ (500 MHz)



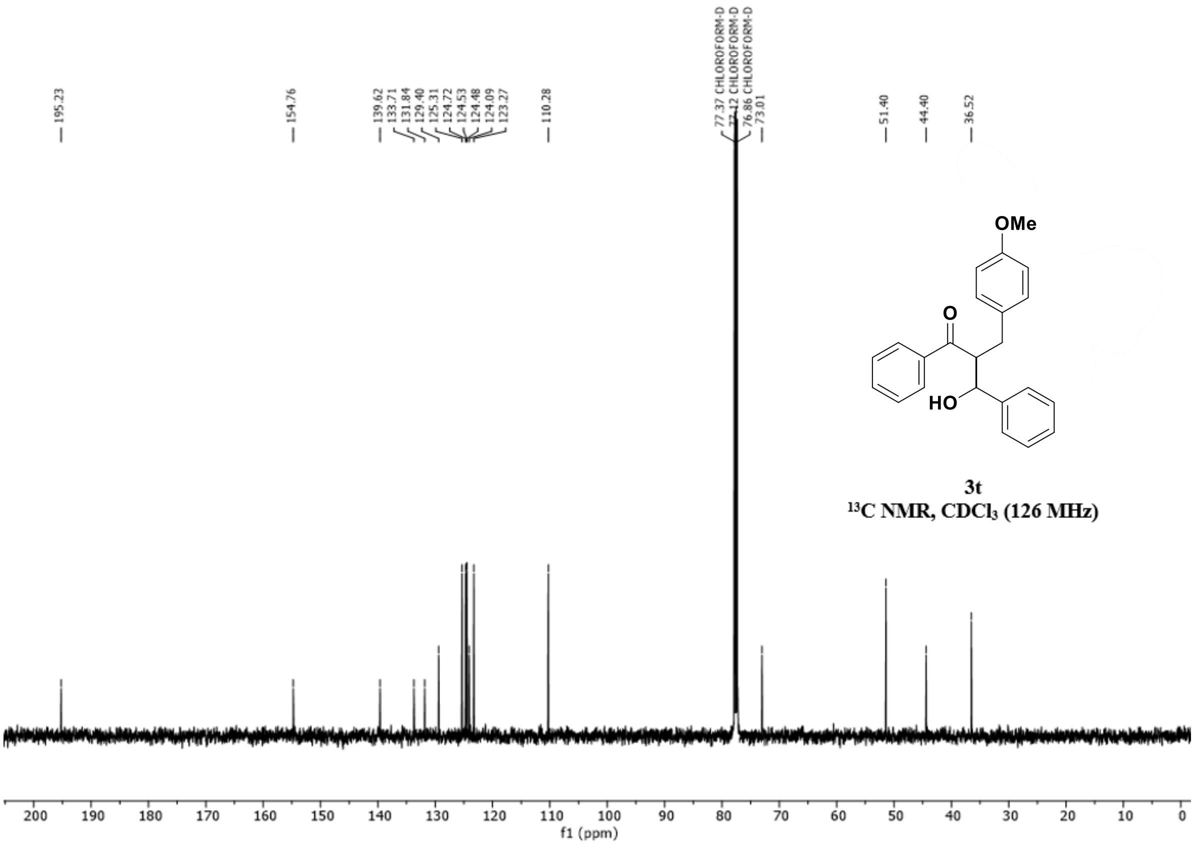
3s

^{13}C NMR, CDCl$_3$ (126 MHz)



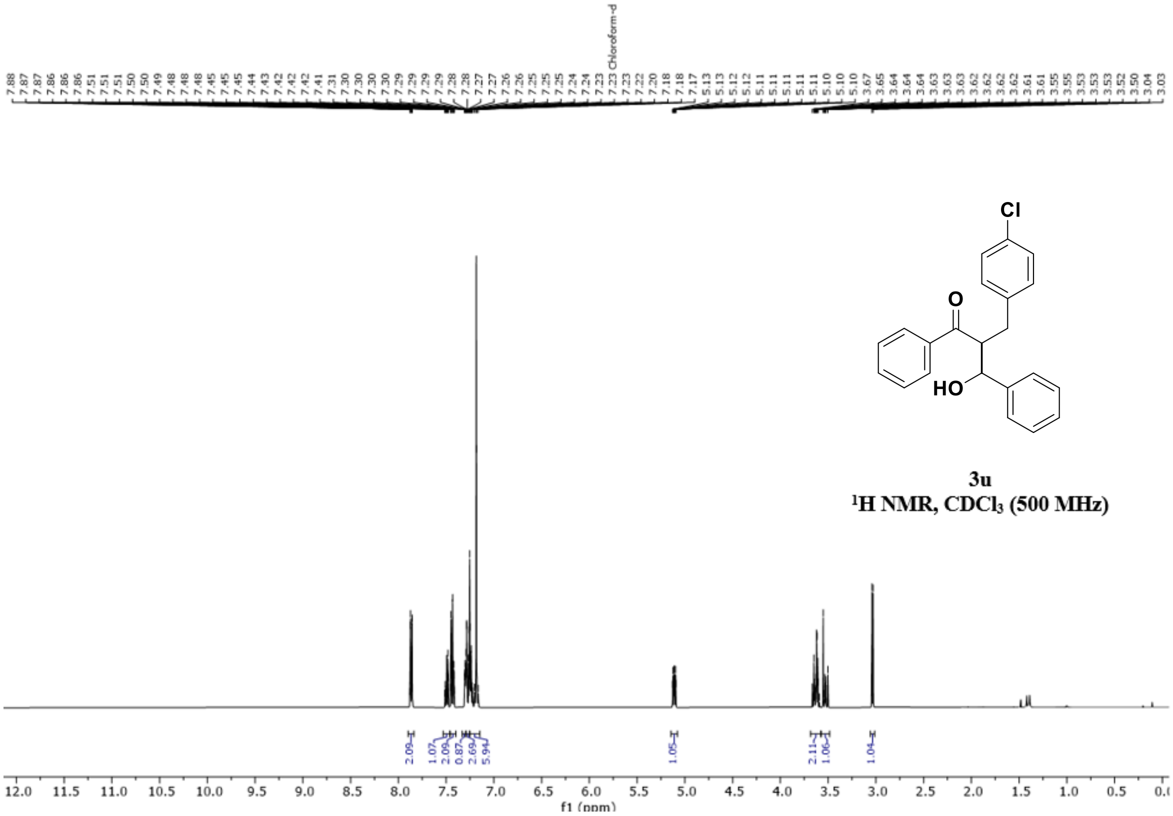
3t

^{1}H NMR, CDCl$_3$ (500 MHz)



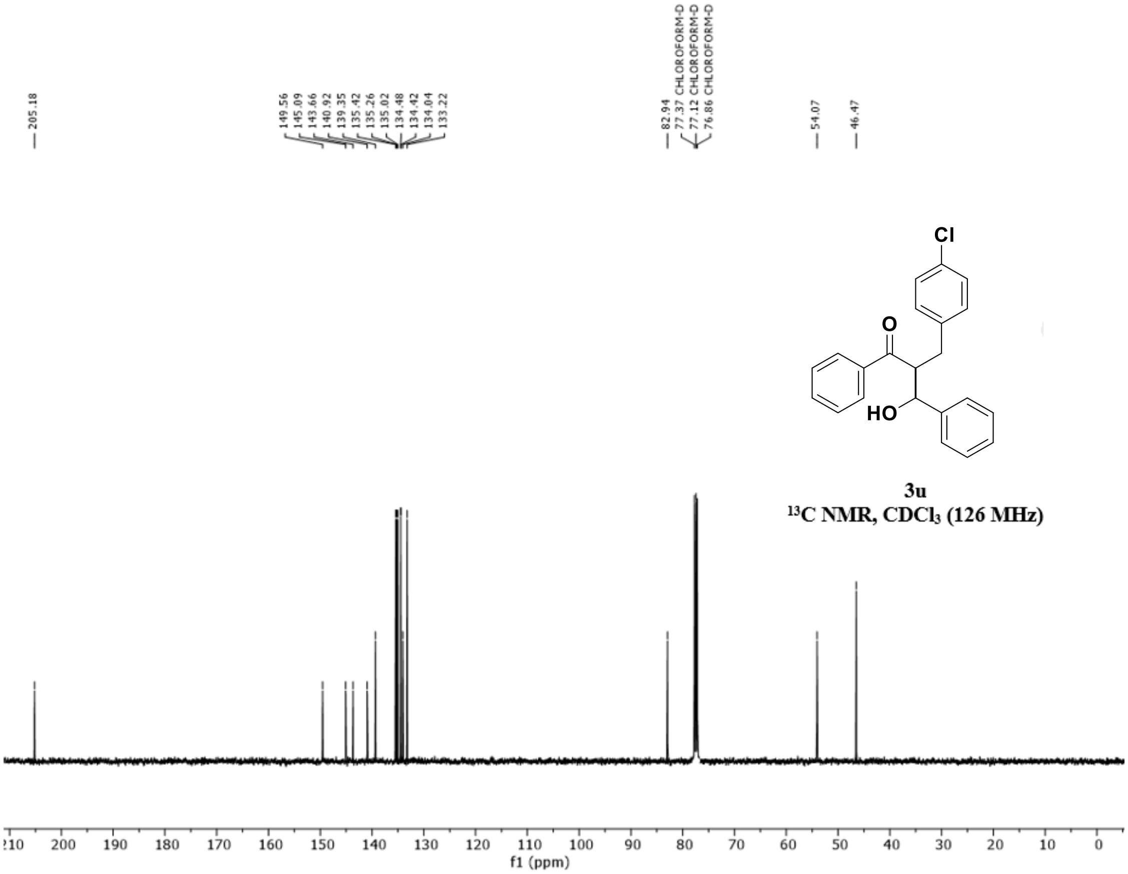
3t

^{13}C NMR, CDCl$_3$ (126 MHz)



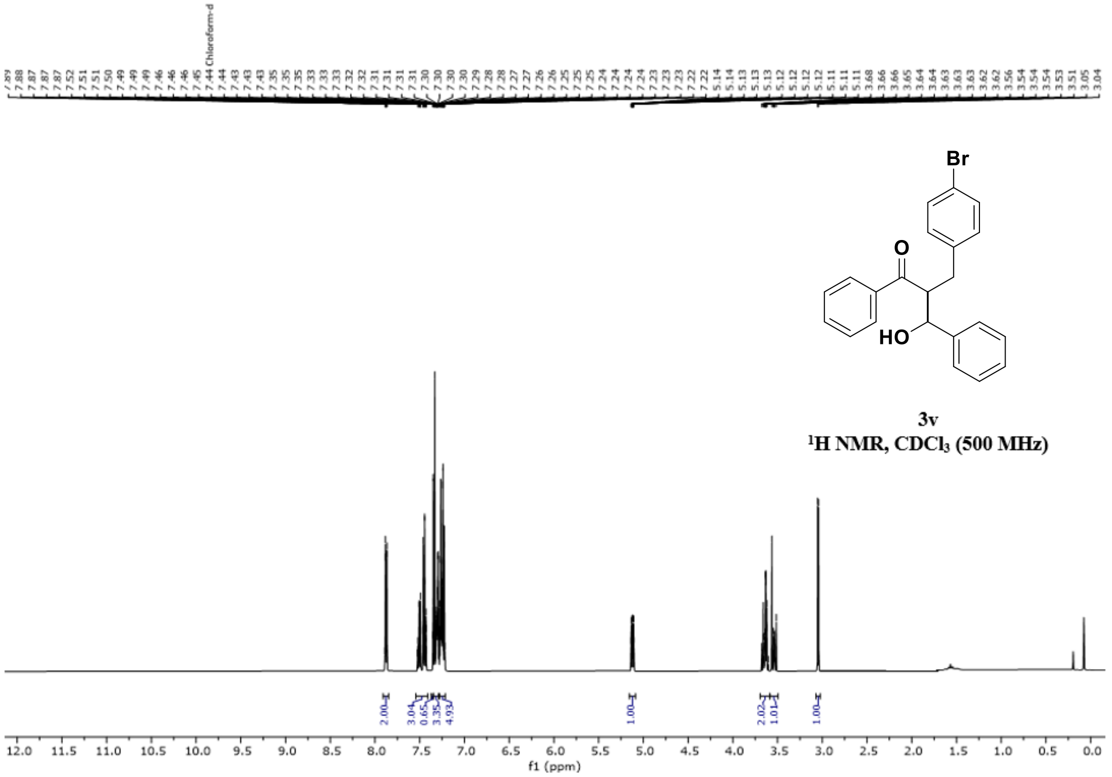
3u

^{1}H NMR, $CDCl_3$ (500 MHz)



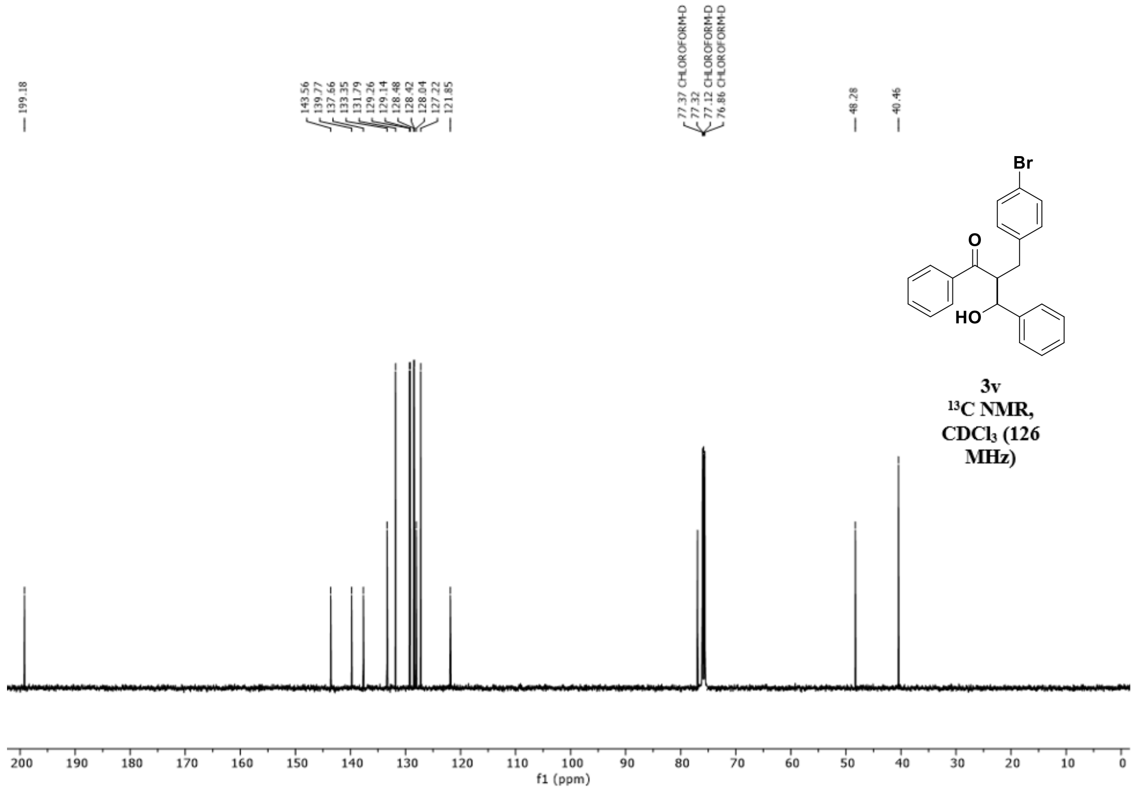
3u

^{13}C NMR, $CDCl_3$ (126 MHz)



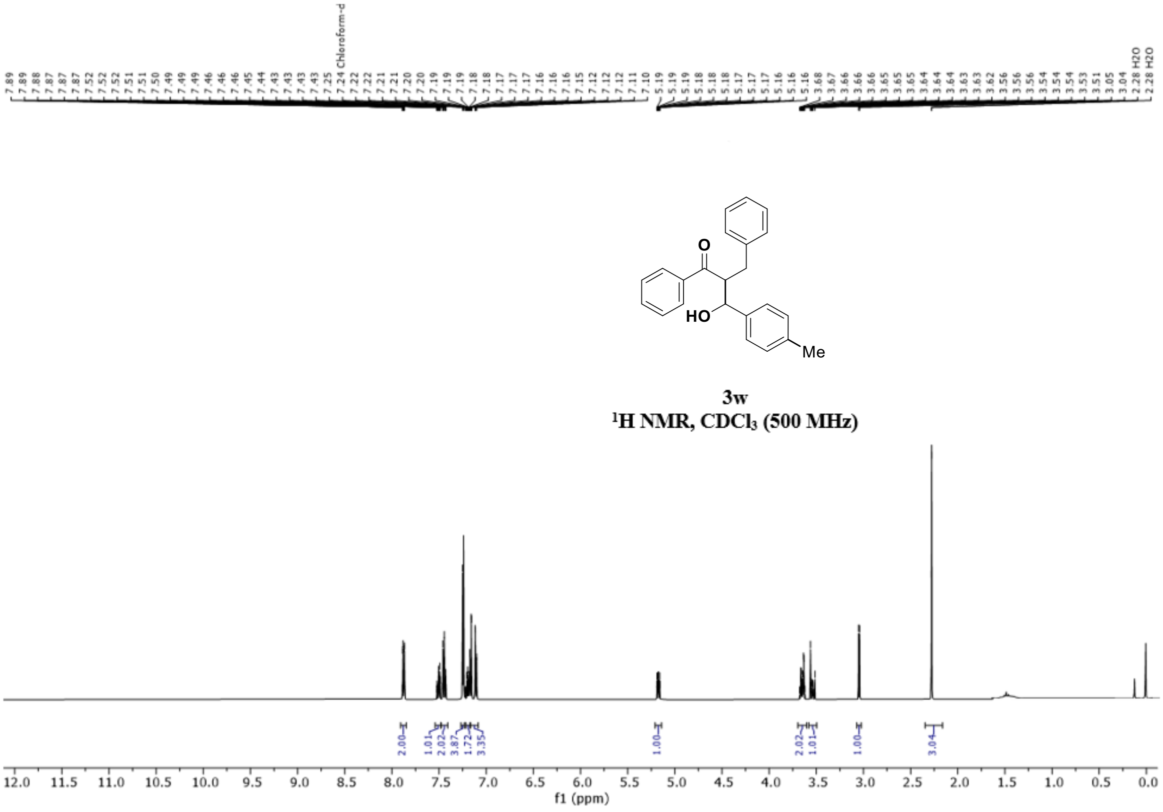
3v

^{1}H NMR, CDCl$_3$ (500 MHz)



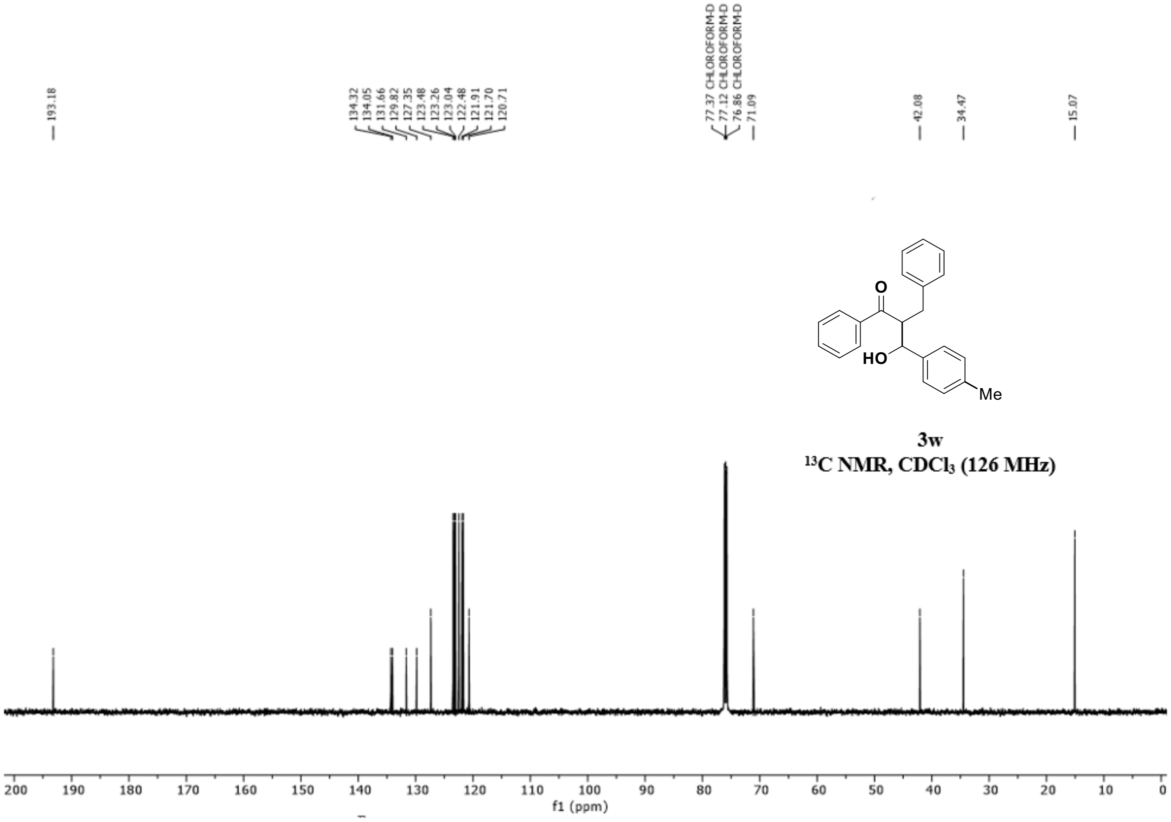
3v

^{13}C NMR, CDCl$_3$ (126 MHz)



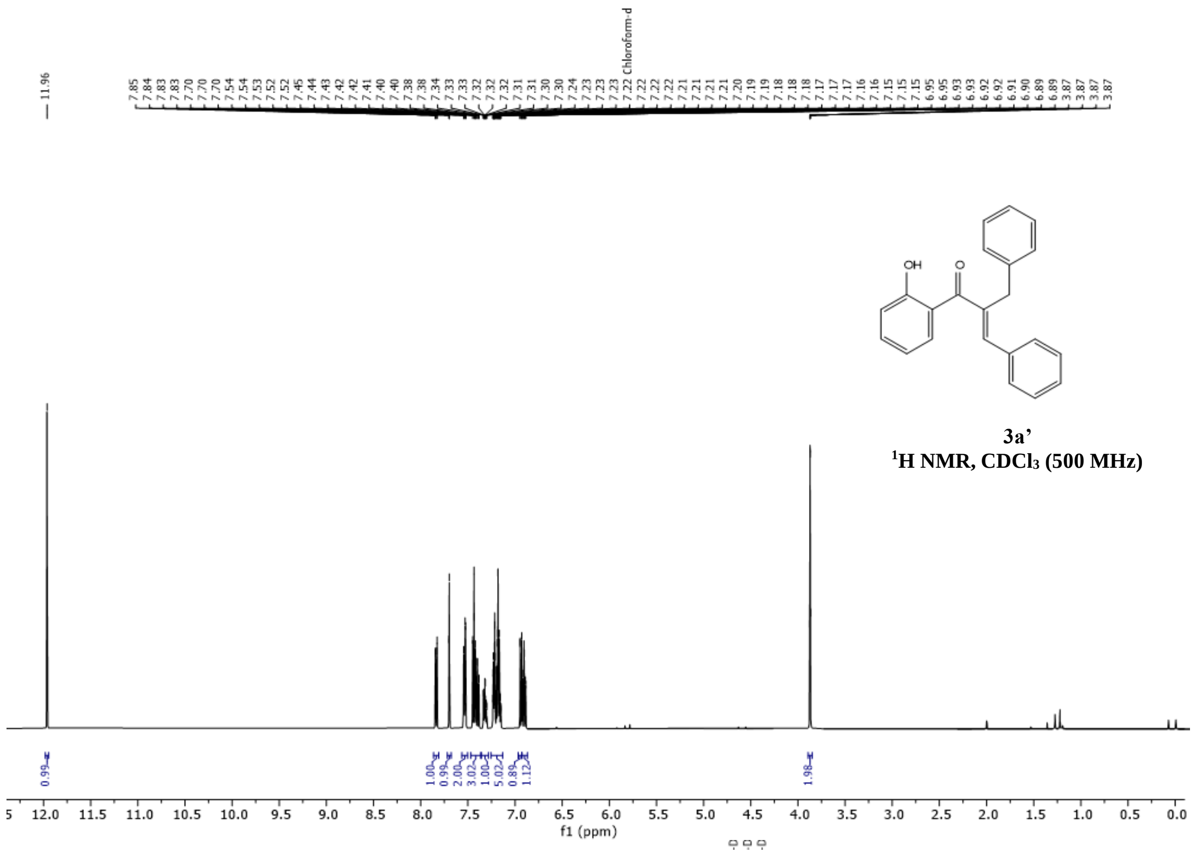
3w

^{1}H NMR, CDCl$_3$ (500 MHz)



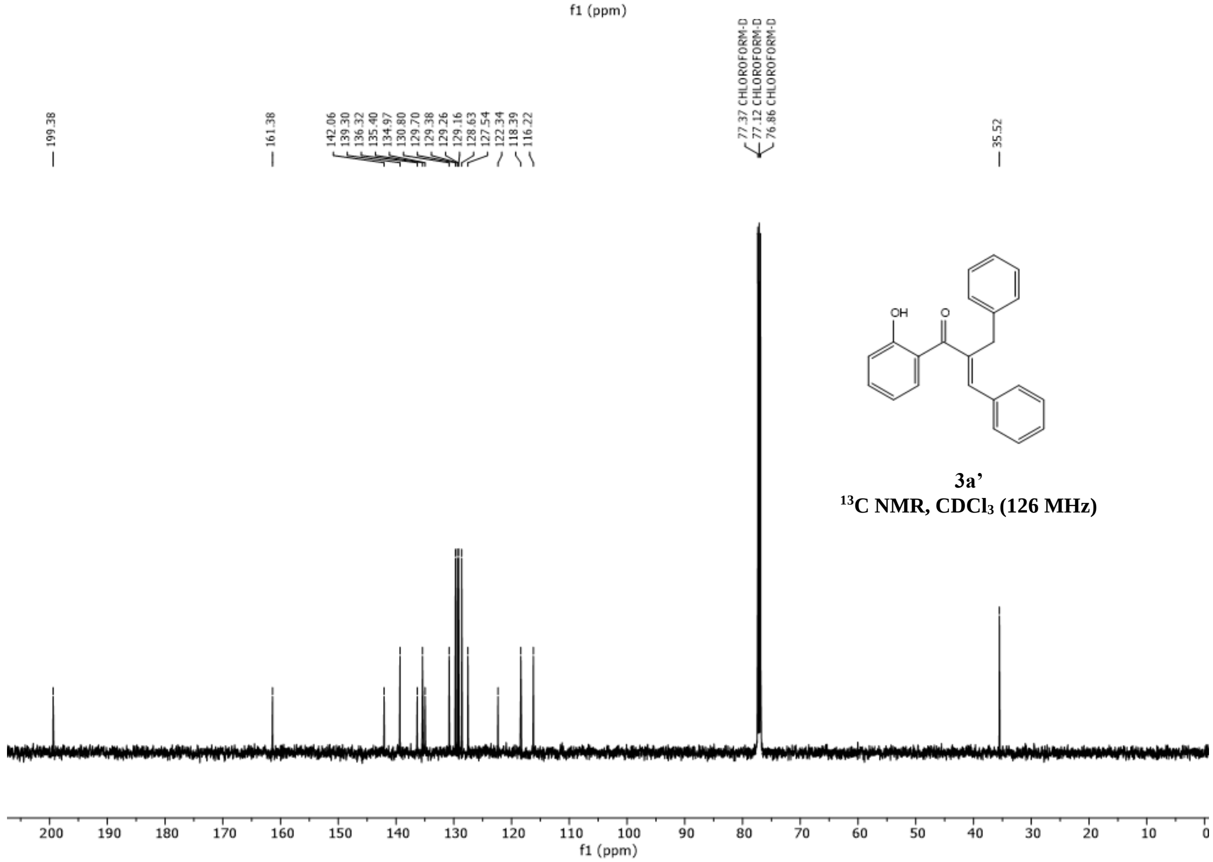
3w

^{13}C NMR, CDCl$_3$ (126 MHz)



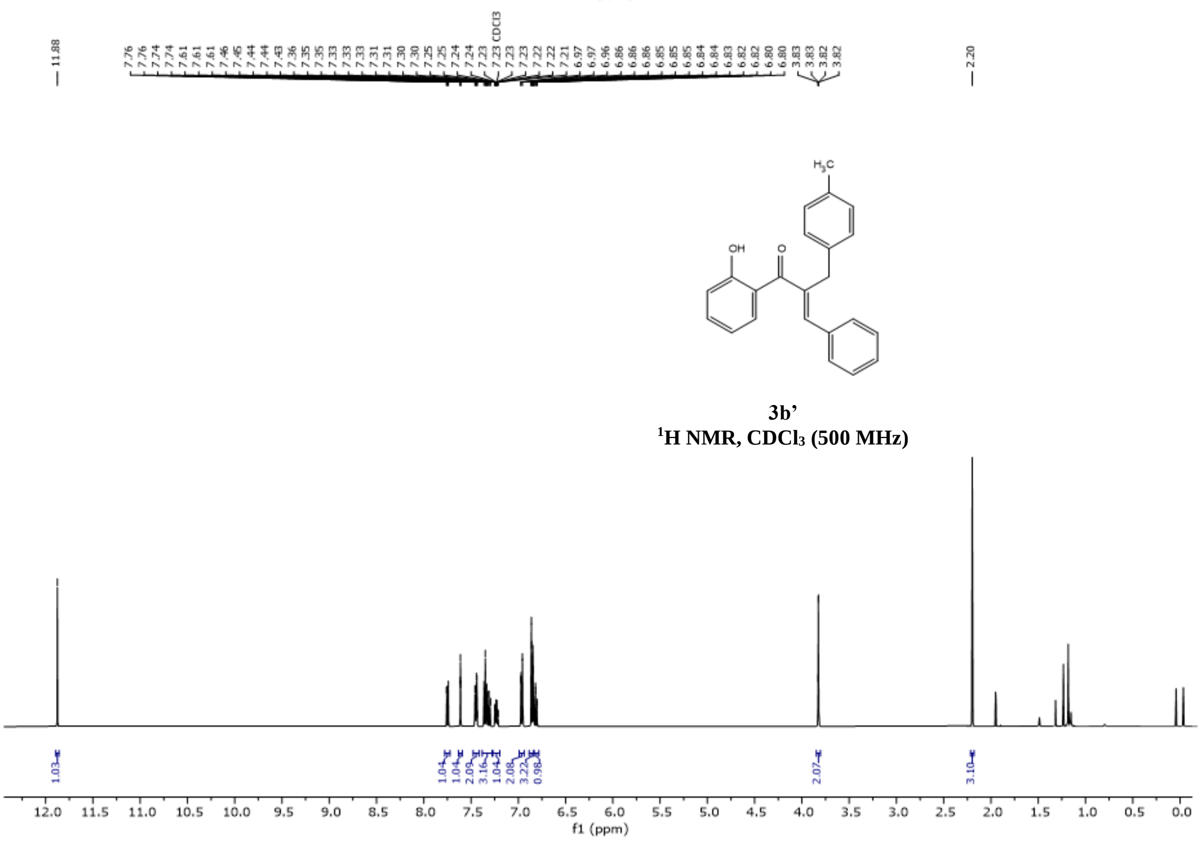
3a'

^{1}H NMR, CDCl$_3$ (500 MHz)



3a'

^{13}C NMR, CDCl$_3$ (126 MHz)



3b'

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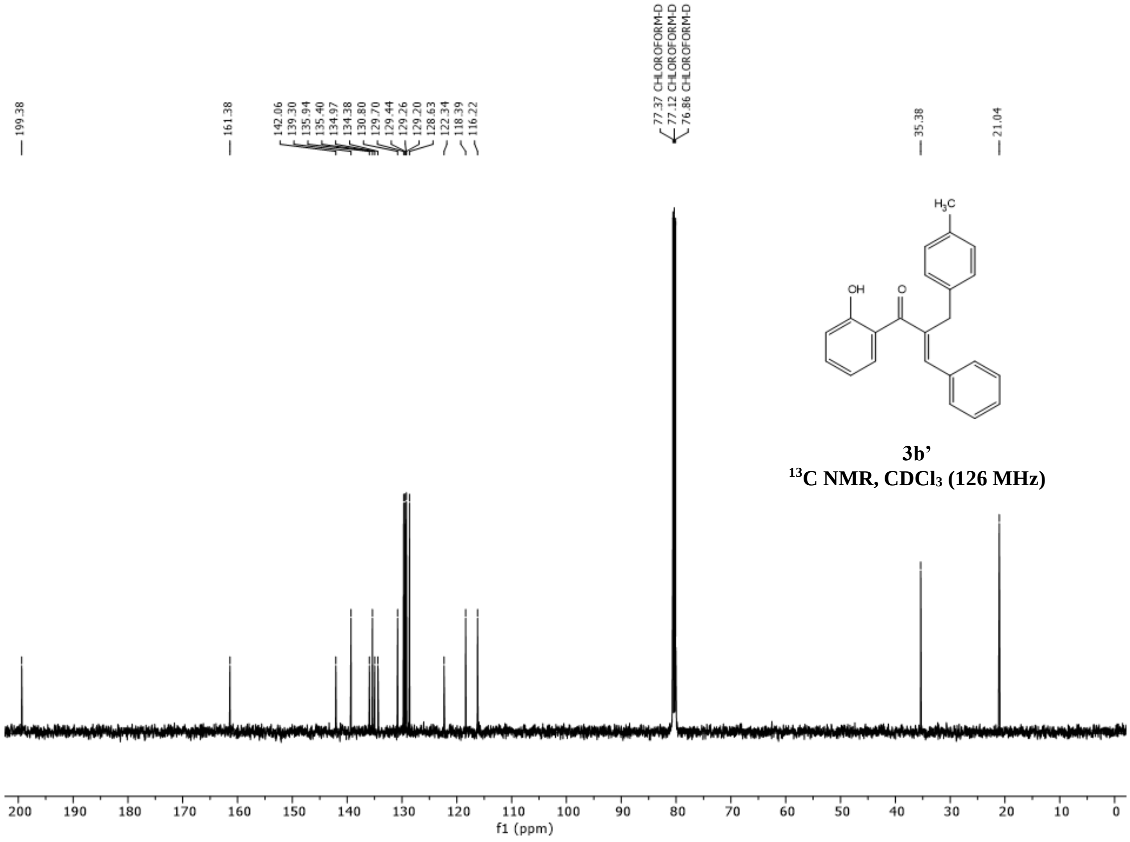
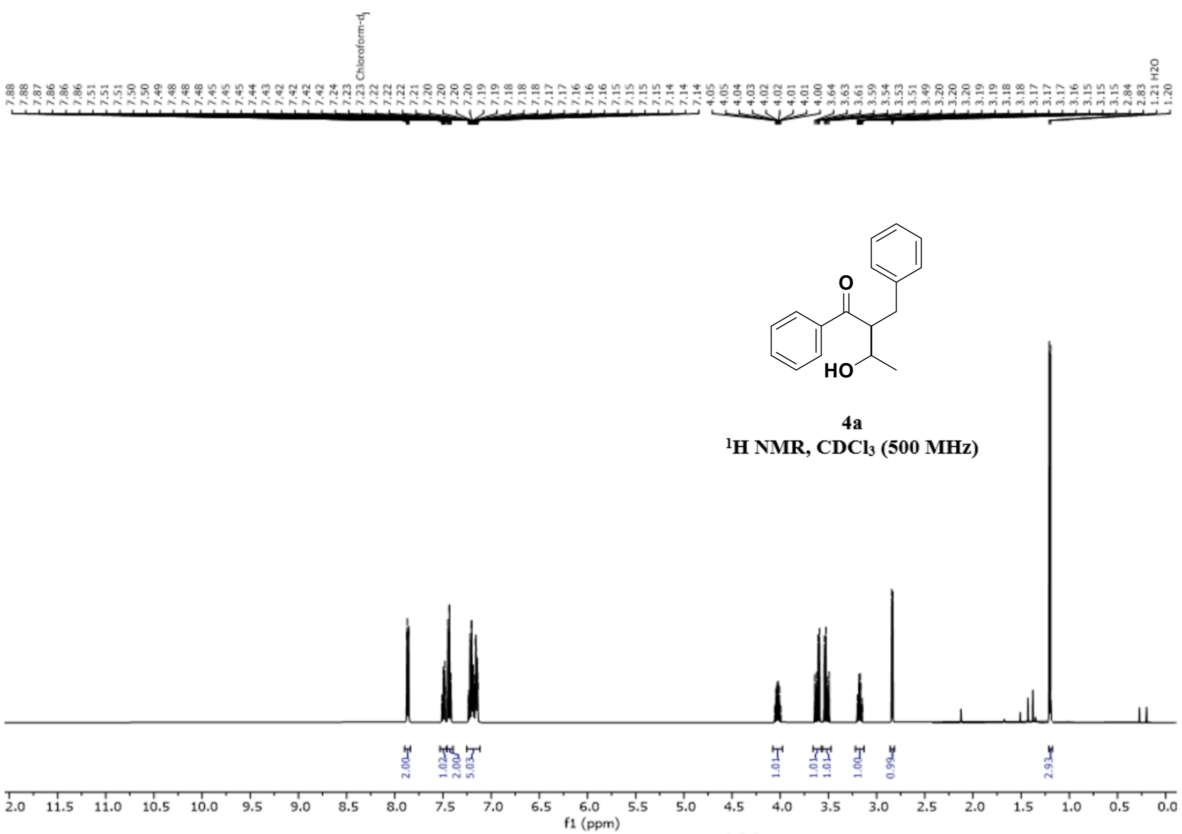


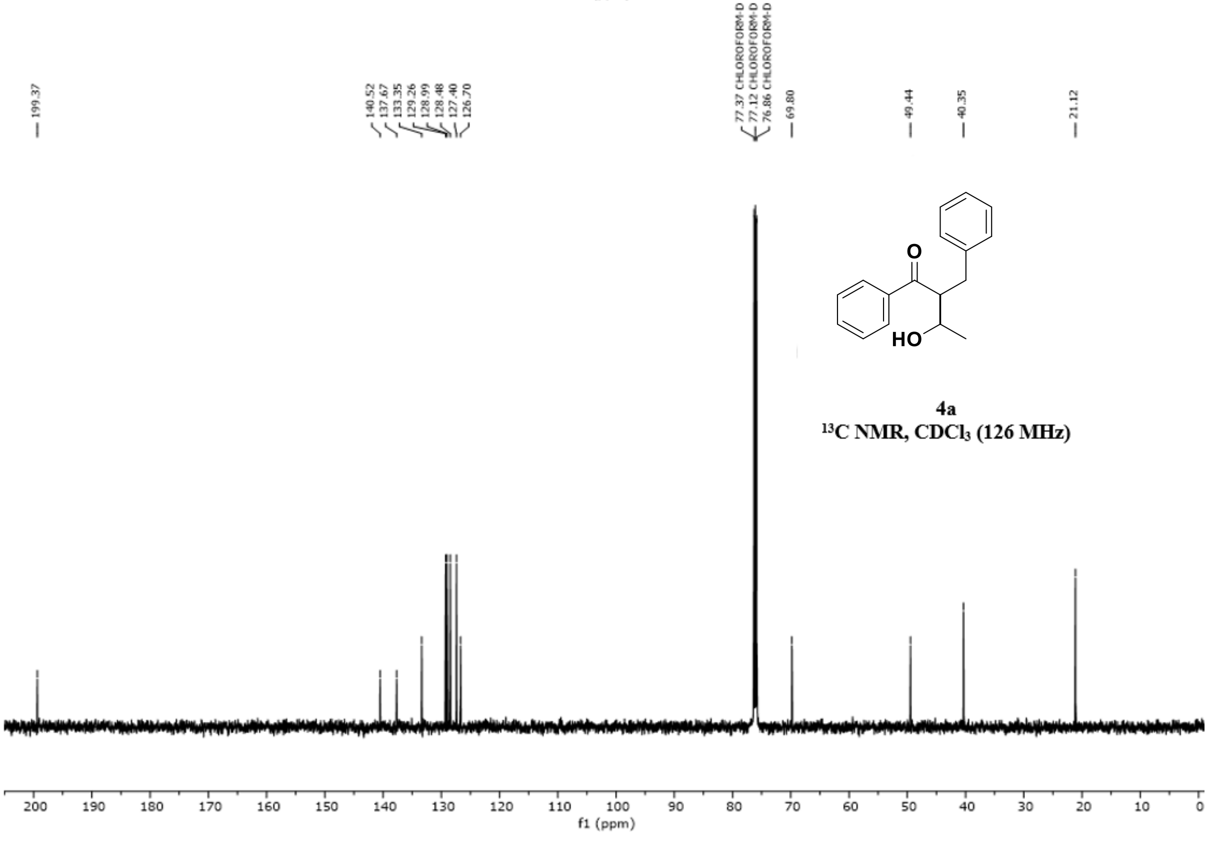
3b'

^{13}C NMR, $CDCl_3$ (126 MHz)



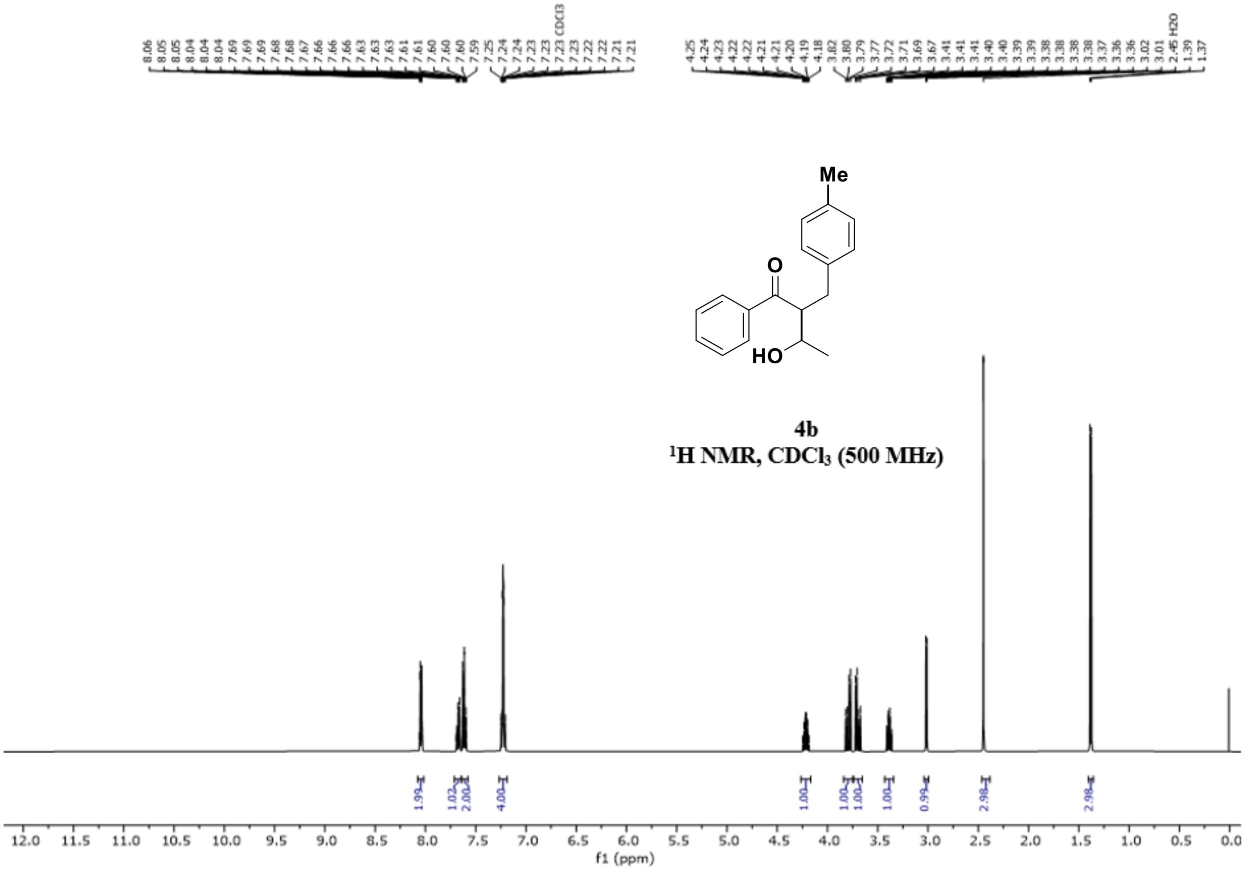
4a

^{1}H NMR, CDCl$_3$ (500 MHz)

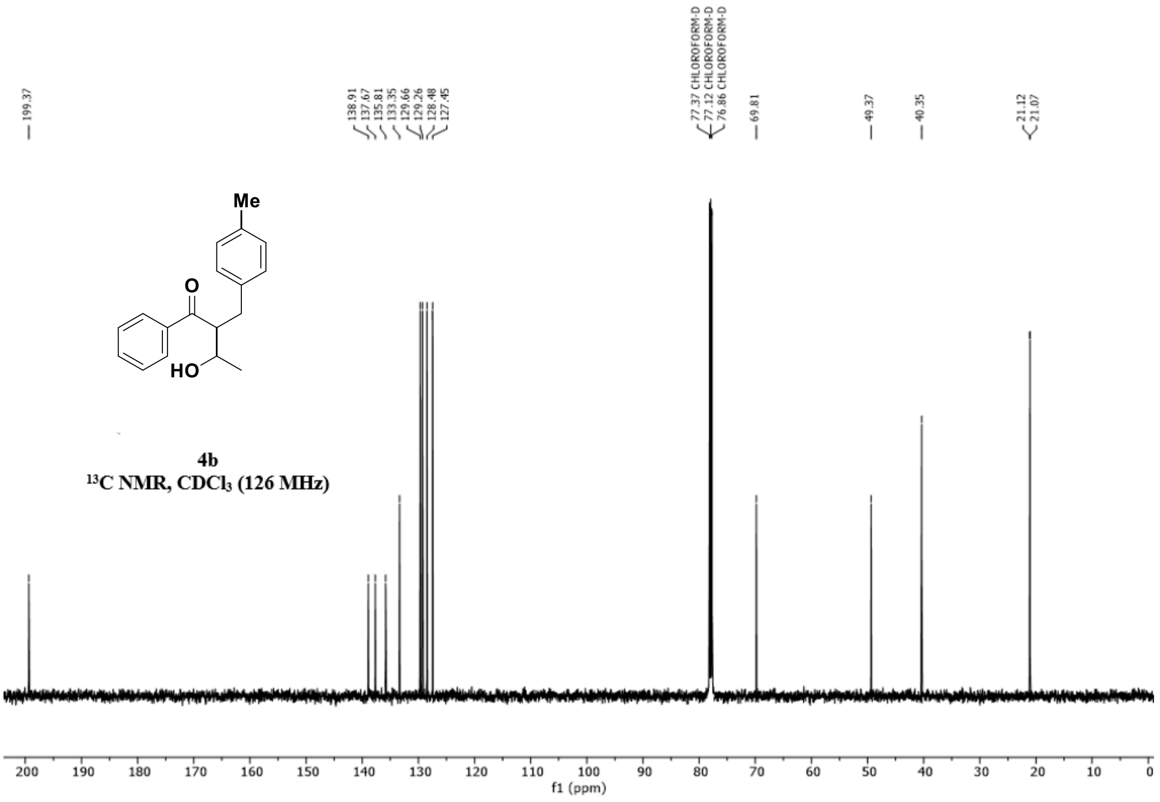


4a

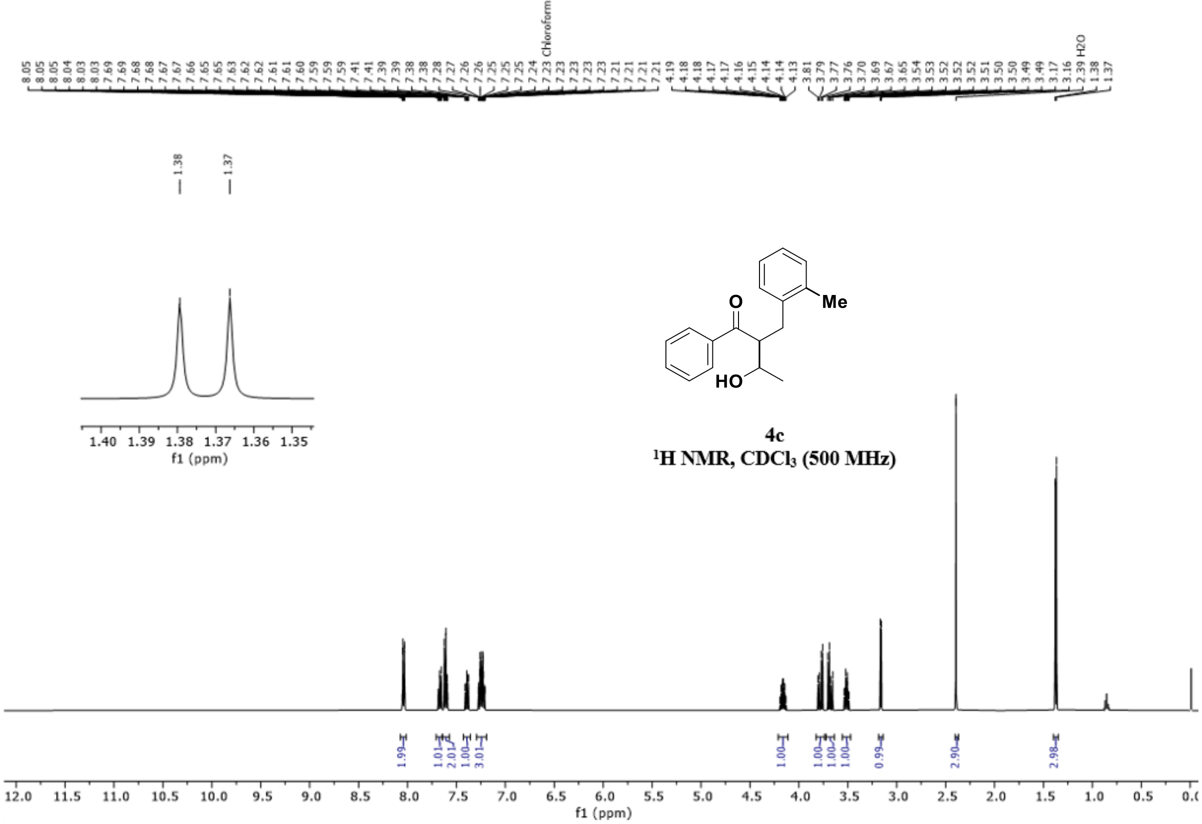
^{13}C NMR, CDCl$_3$ (126 MHz)



4b
^{1}H NMR, CDCl$_3$ (500 MHz)

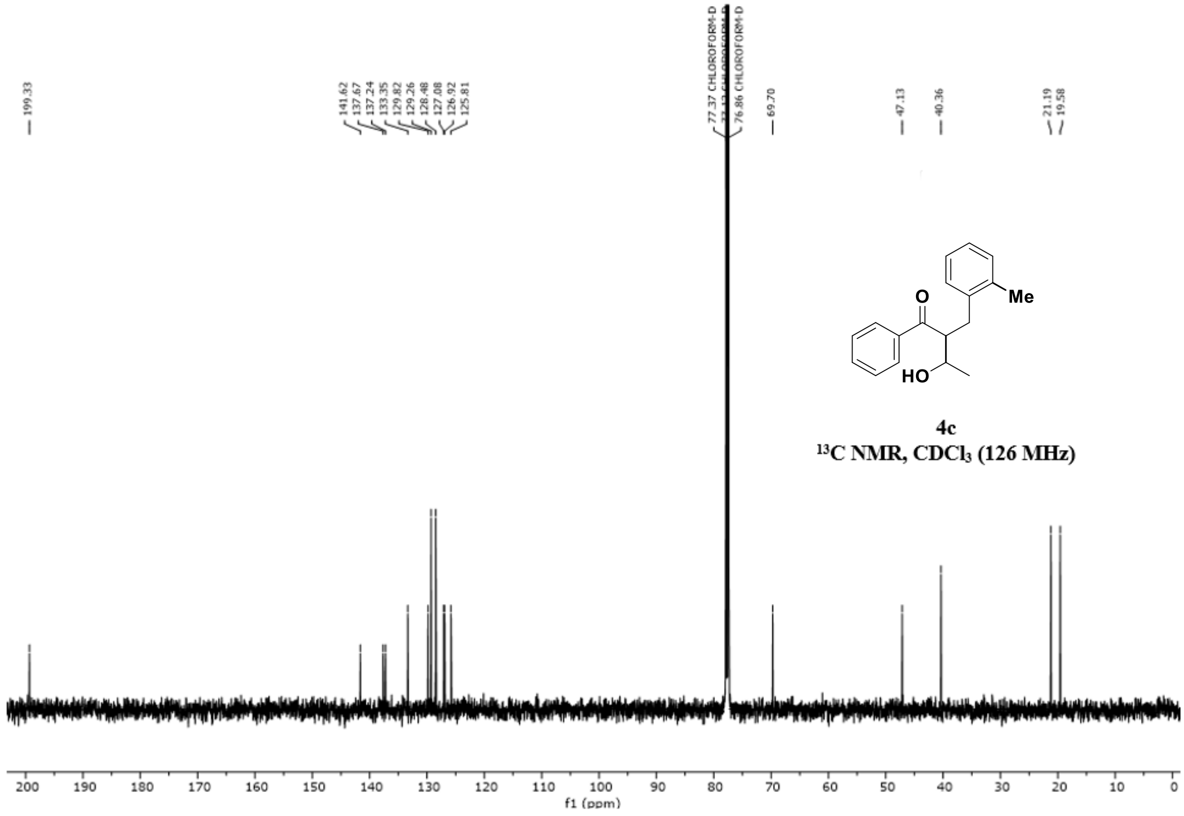


4b
^{13}C NMR, CDCl$_3$ (126 MHz)



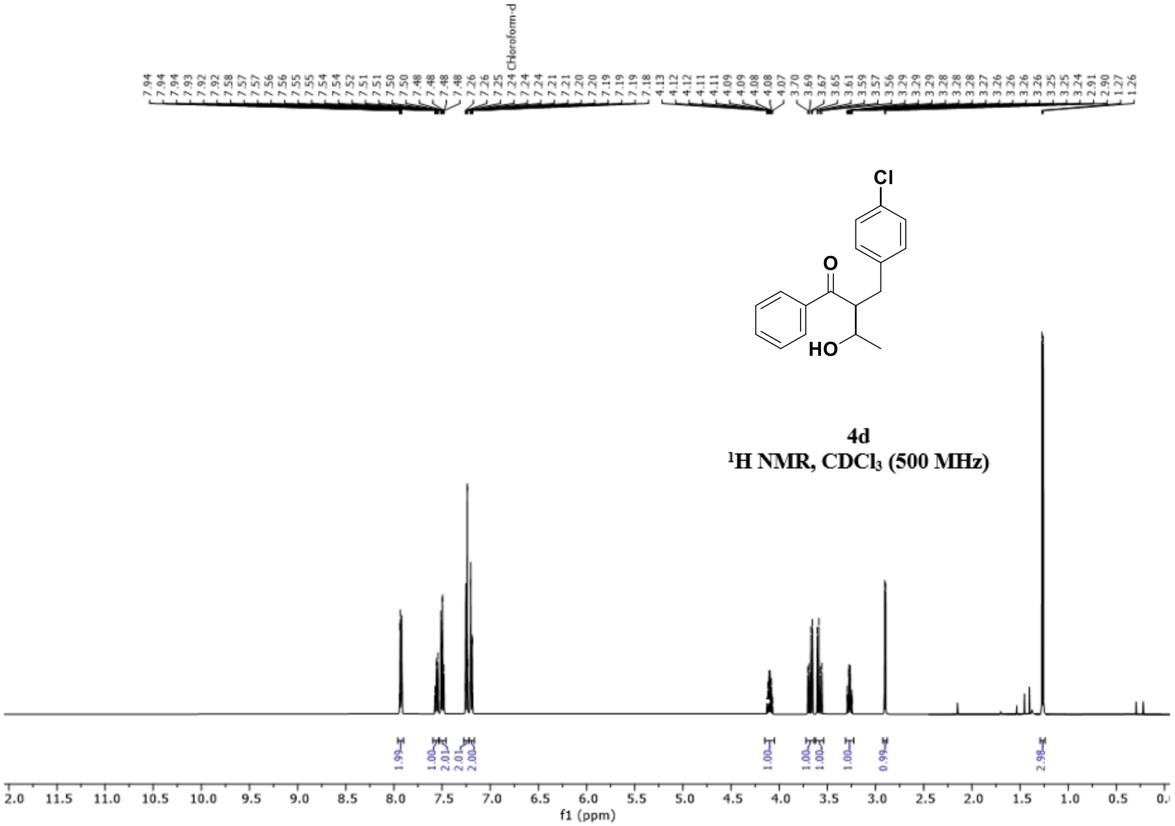
4c

^{1}H NMR, CDCl$_3$ (500 MHz)



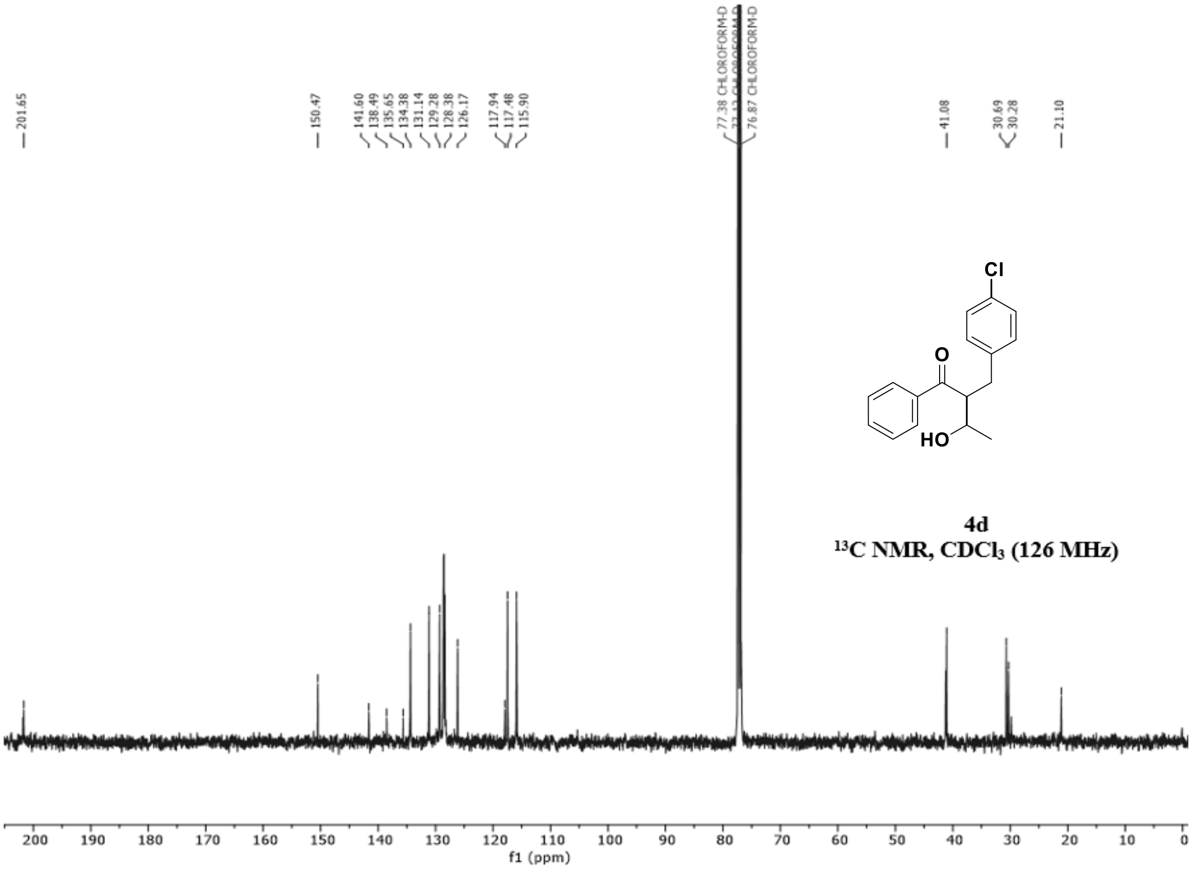
4c

^{13}C NMR, CDCl$_3$ (126 MHz)



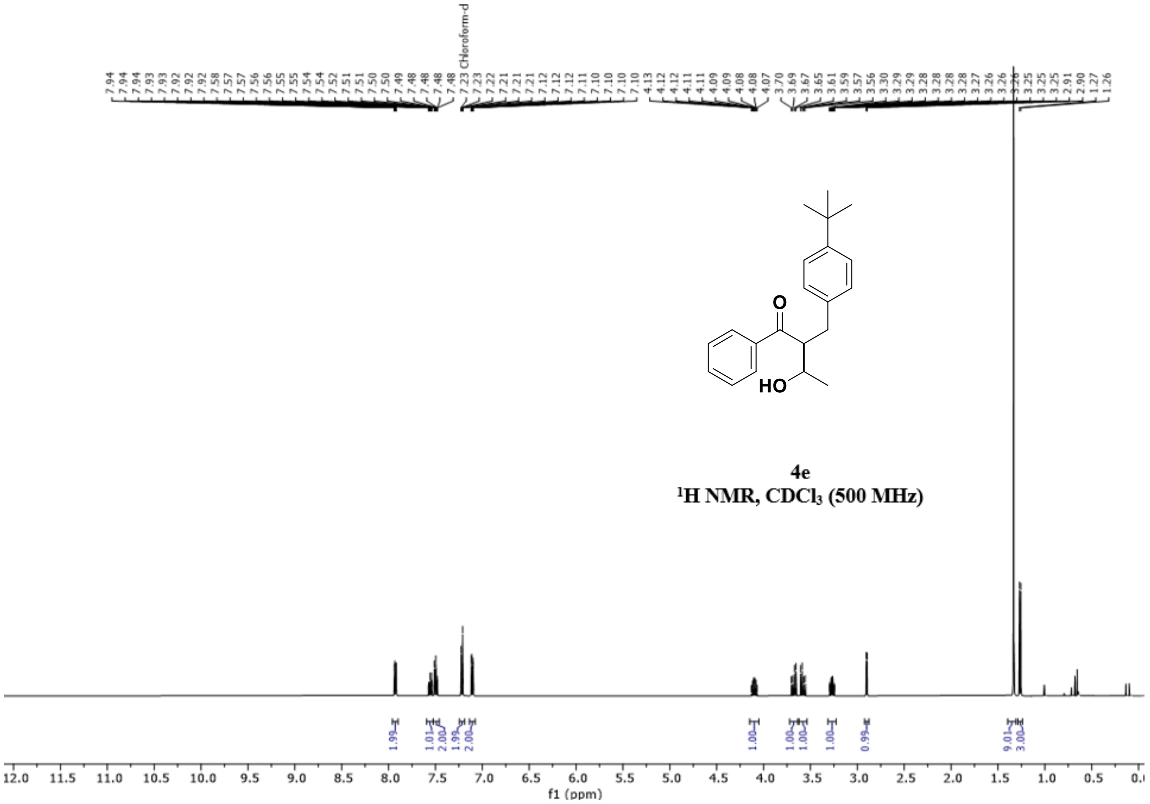
4d

^{1}H NMR, $CDCl_3$ (500 MHz)

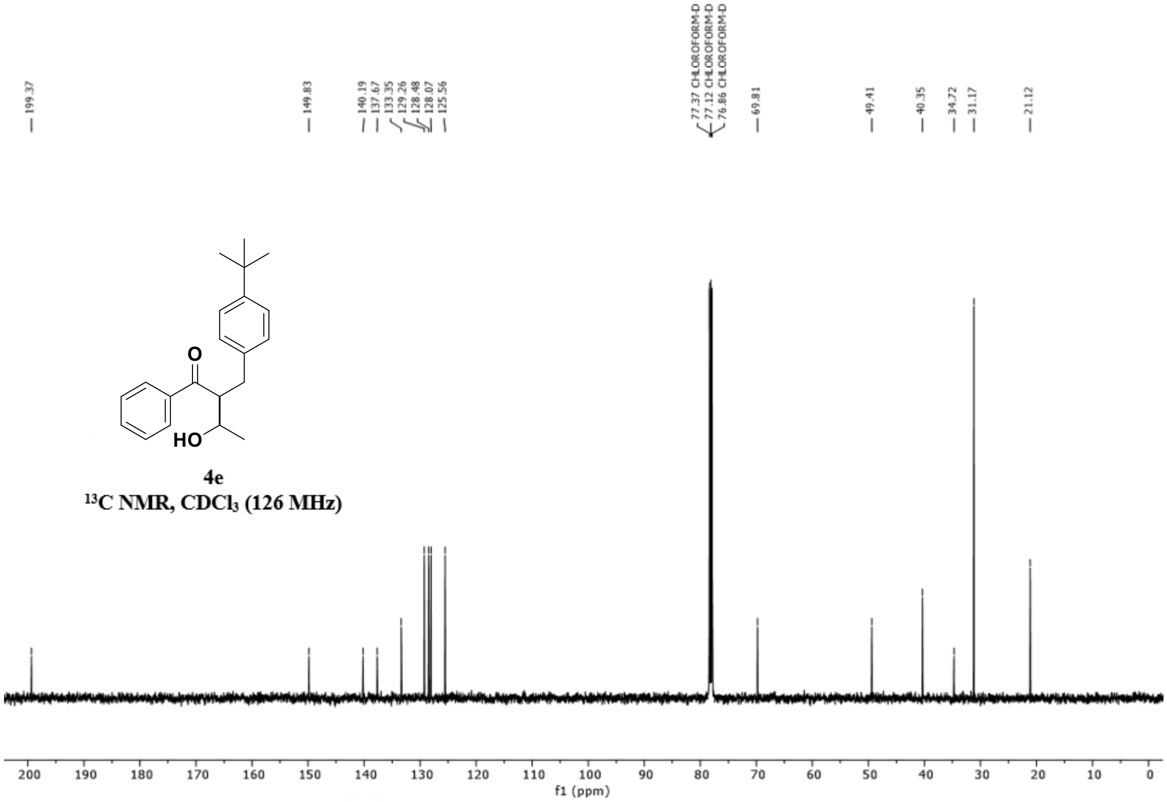


4d

^{13}C NMR, $CDCl_3$ (126 MHz)



4e
^{1}H NMR, CDCl$_3$ (500 MHz)



4e
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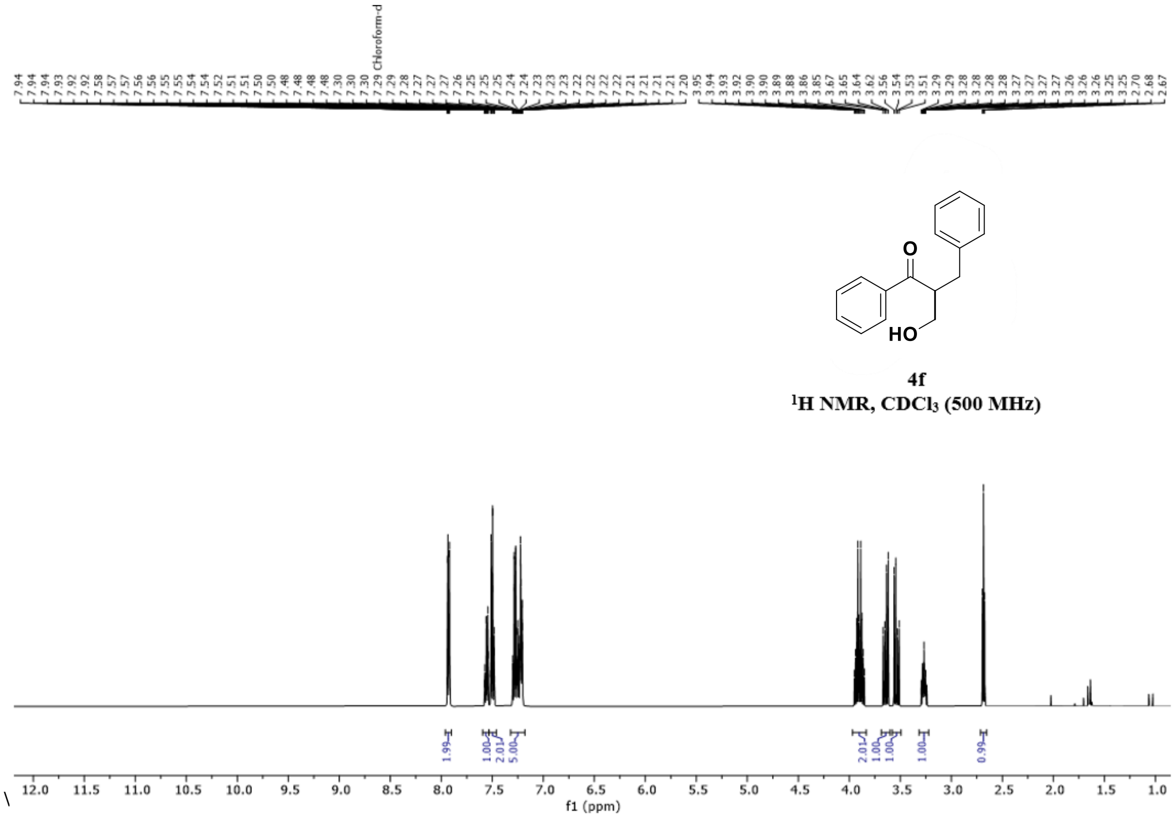
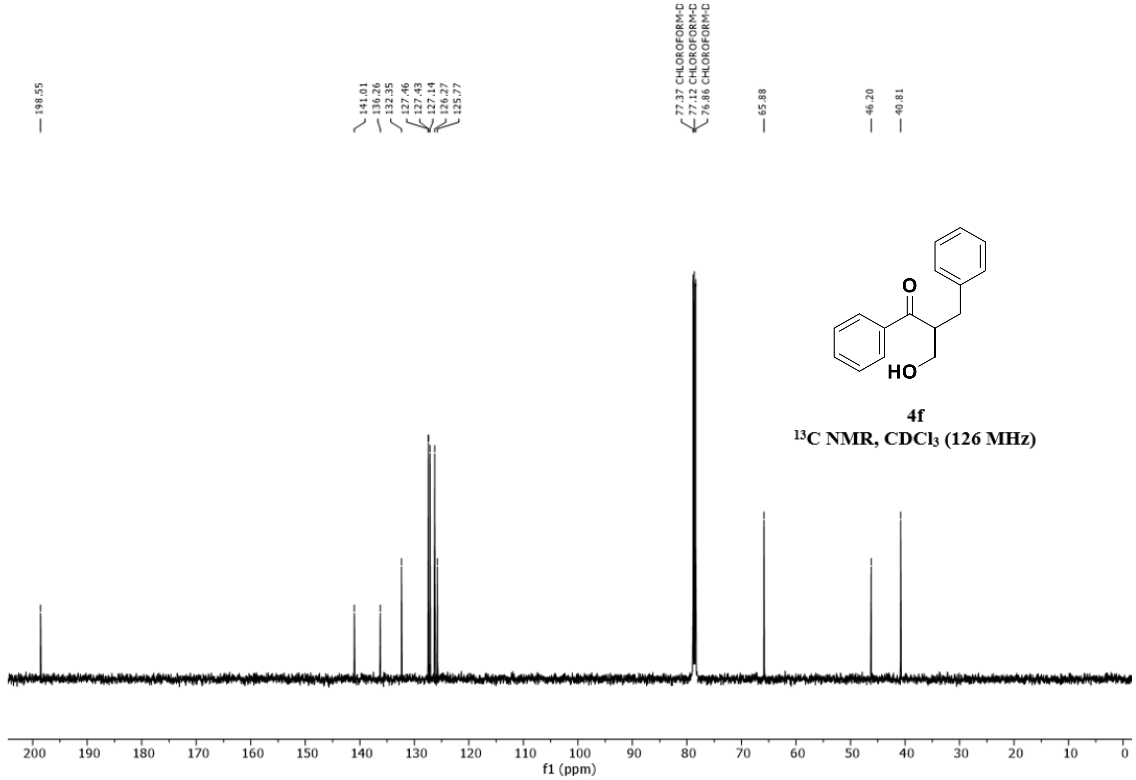
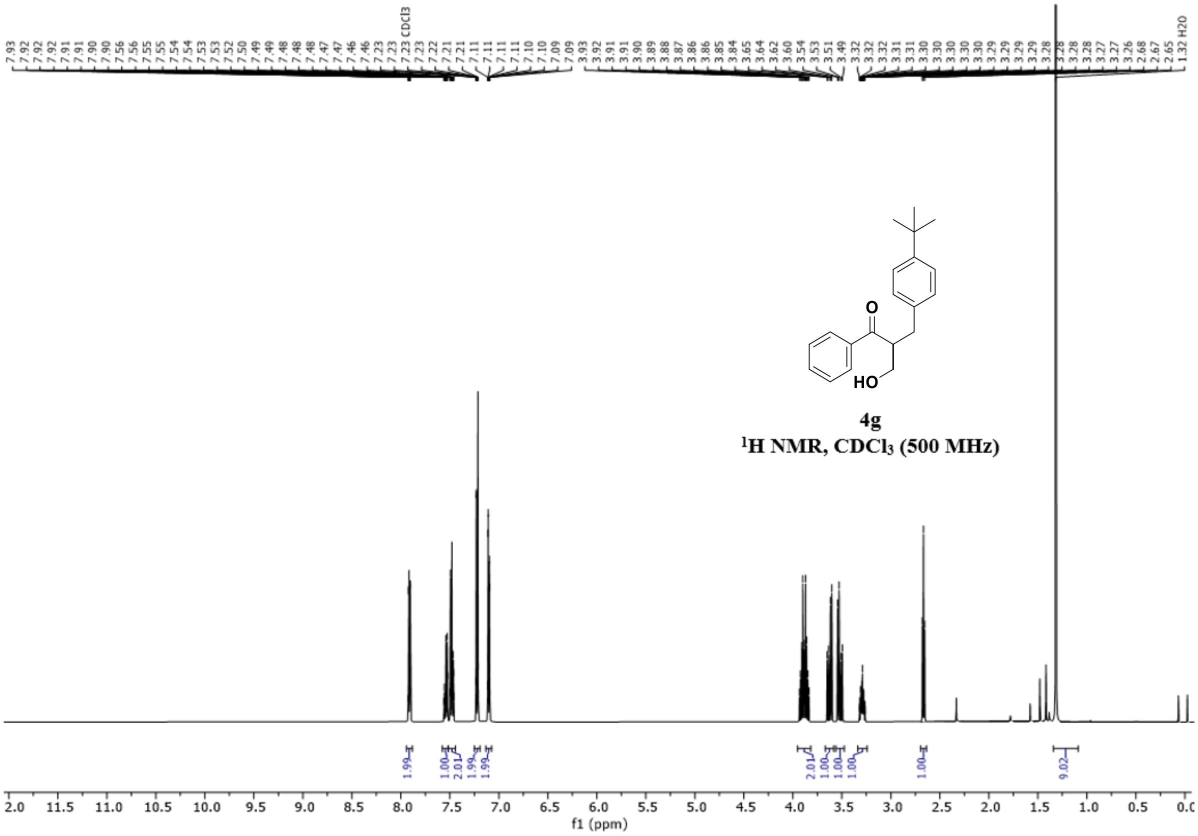


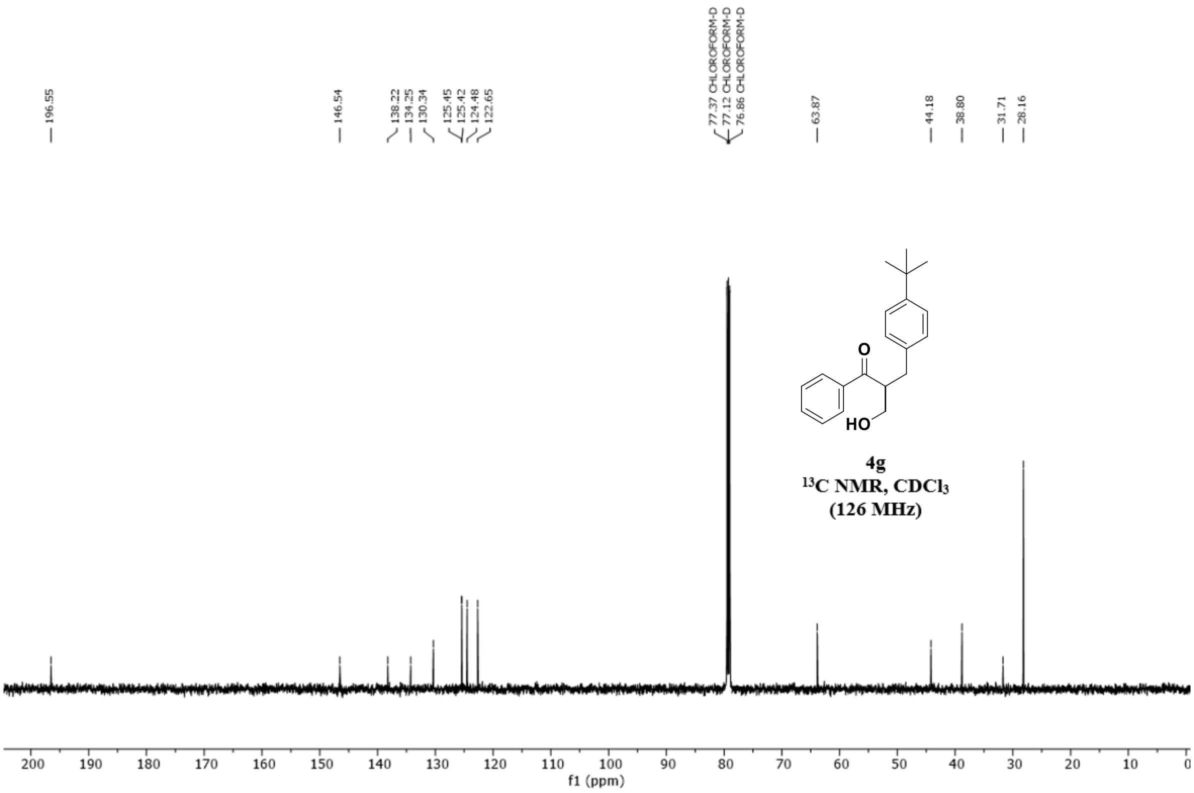
4f
^{1}H NMR, CDCl$_3$ (500 MHz)



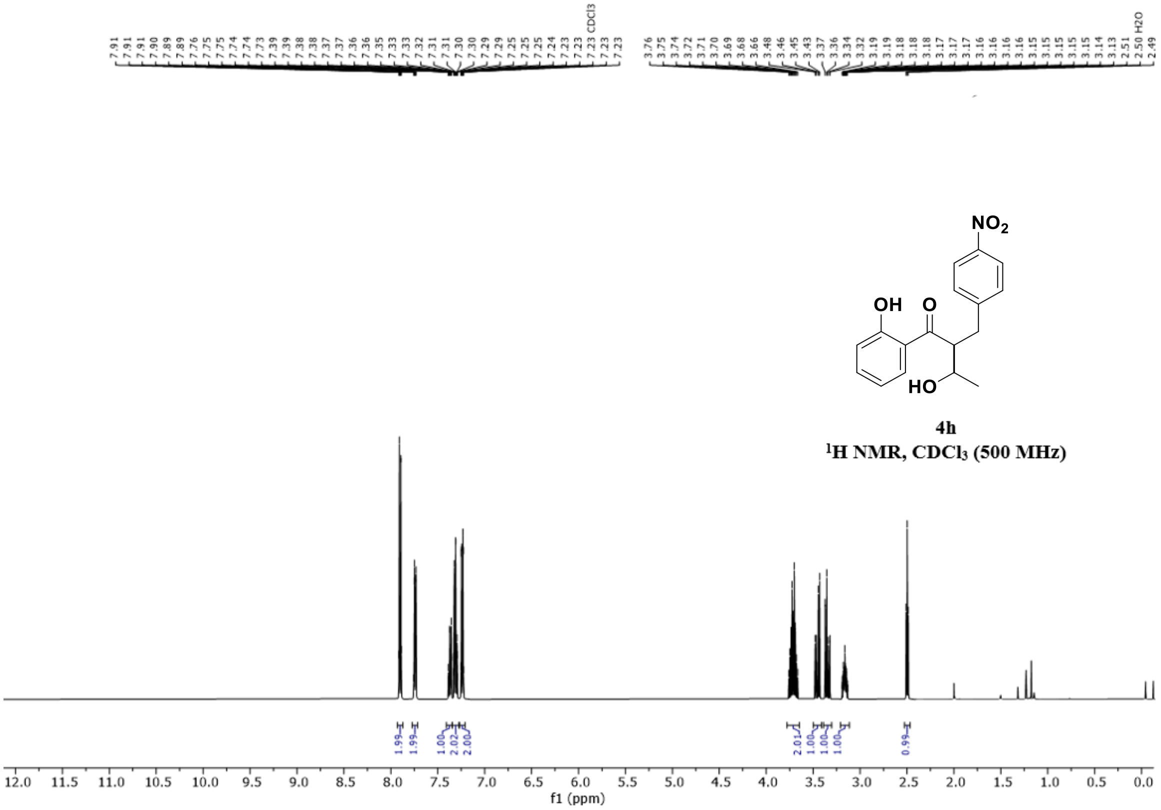
4f
^{13}C NMR, CDCl$_3$ (126 MHz)



4g
^{1}H NMR, CDCl$_3$ (500 MHz)

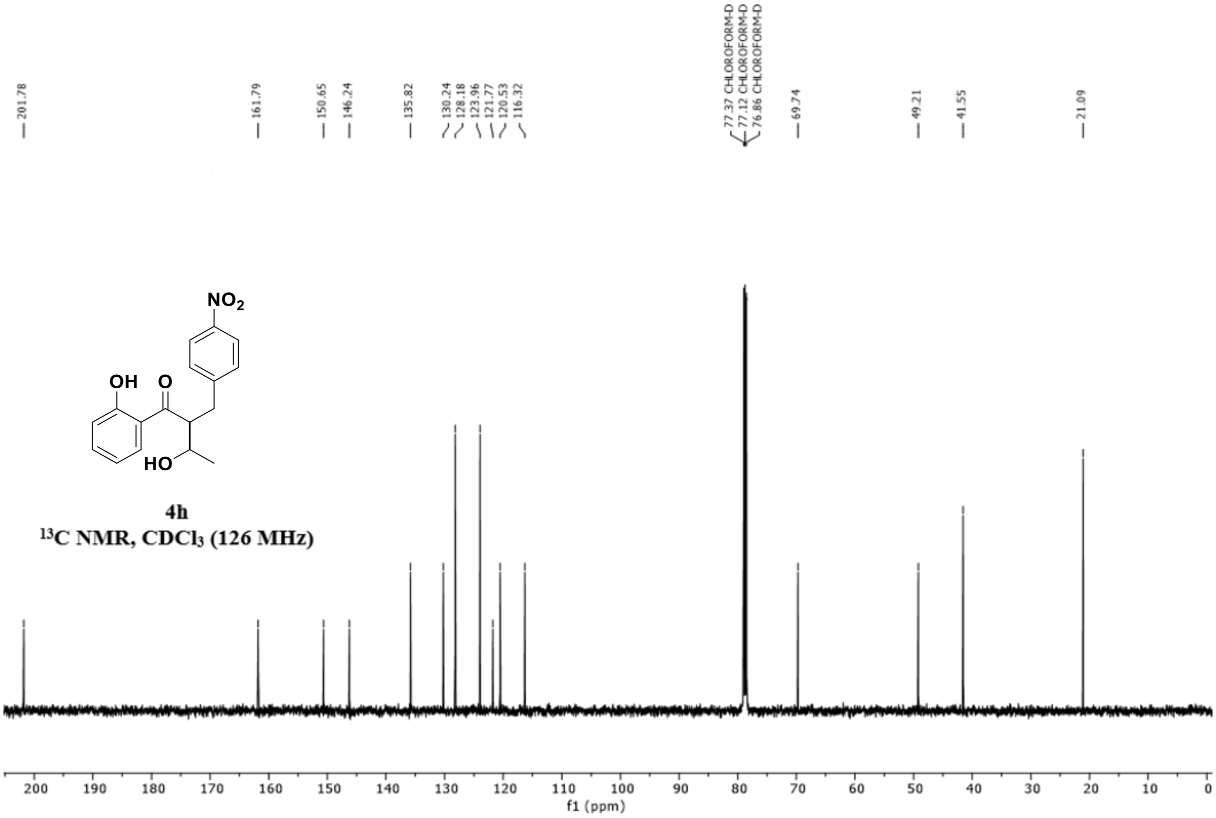


4g
^{13}C NMR, CDCl$_3$
(126 MHz)



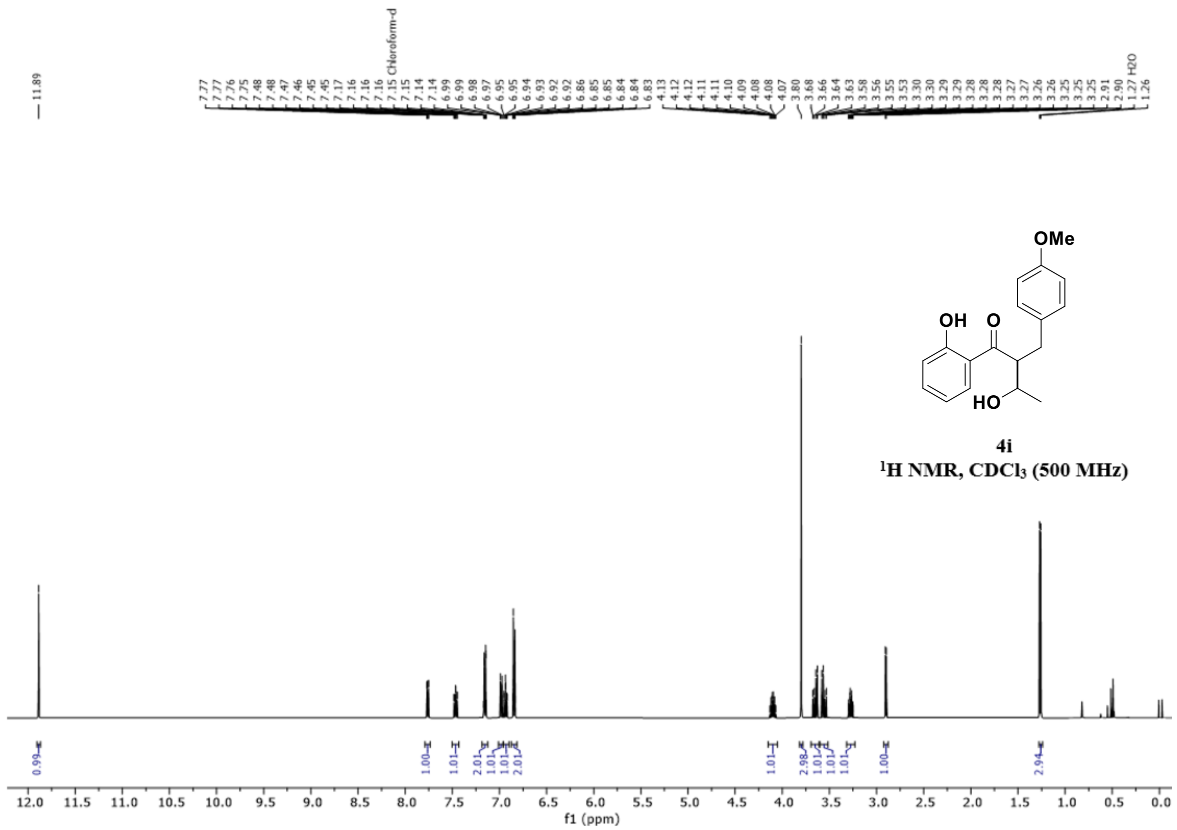
4h

^{1}H NMR, CDCl$_3$ (500 MHz)



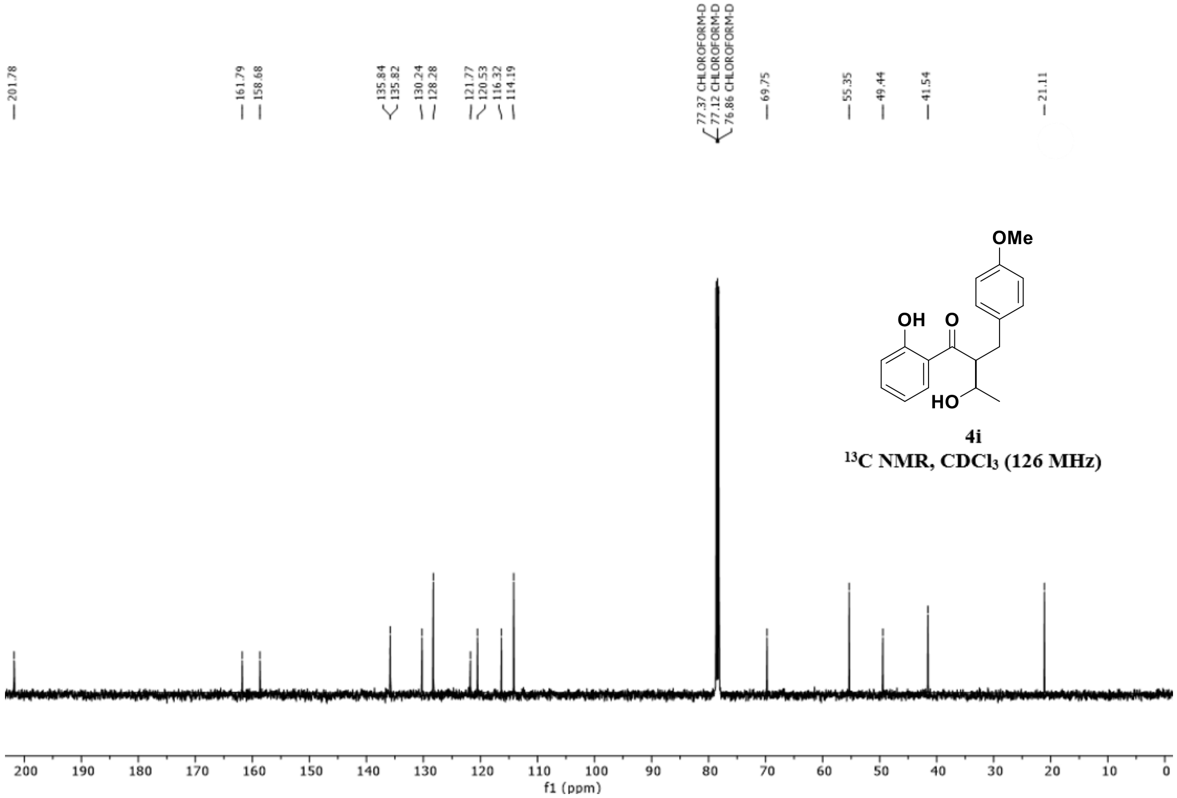
4h

^{13}C NMR, CDCl$_3$ (126 MHz)



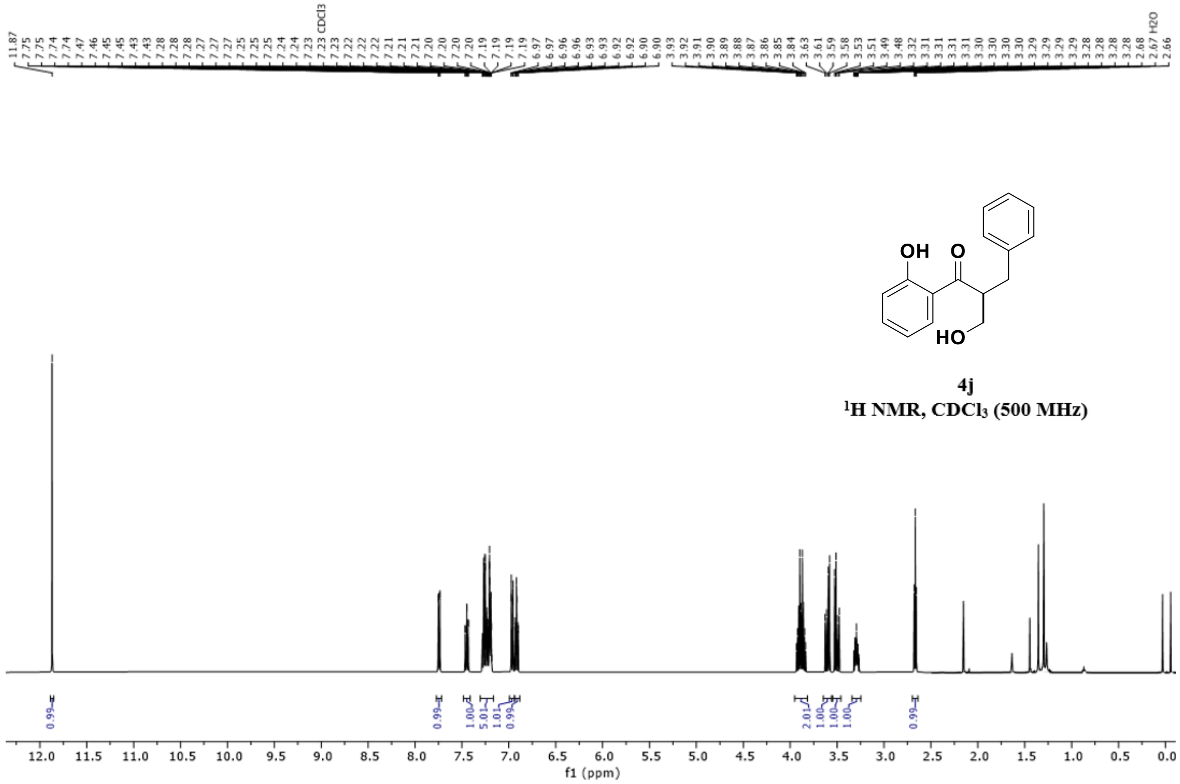
4i

^{1}H NMR, CDCl$_3$ (500 MHz)



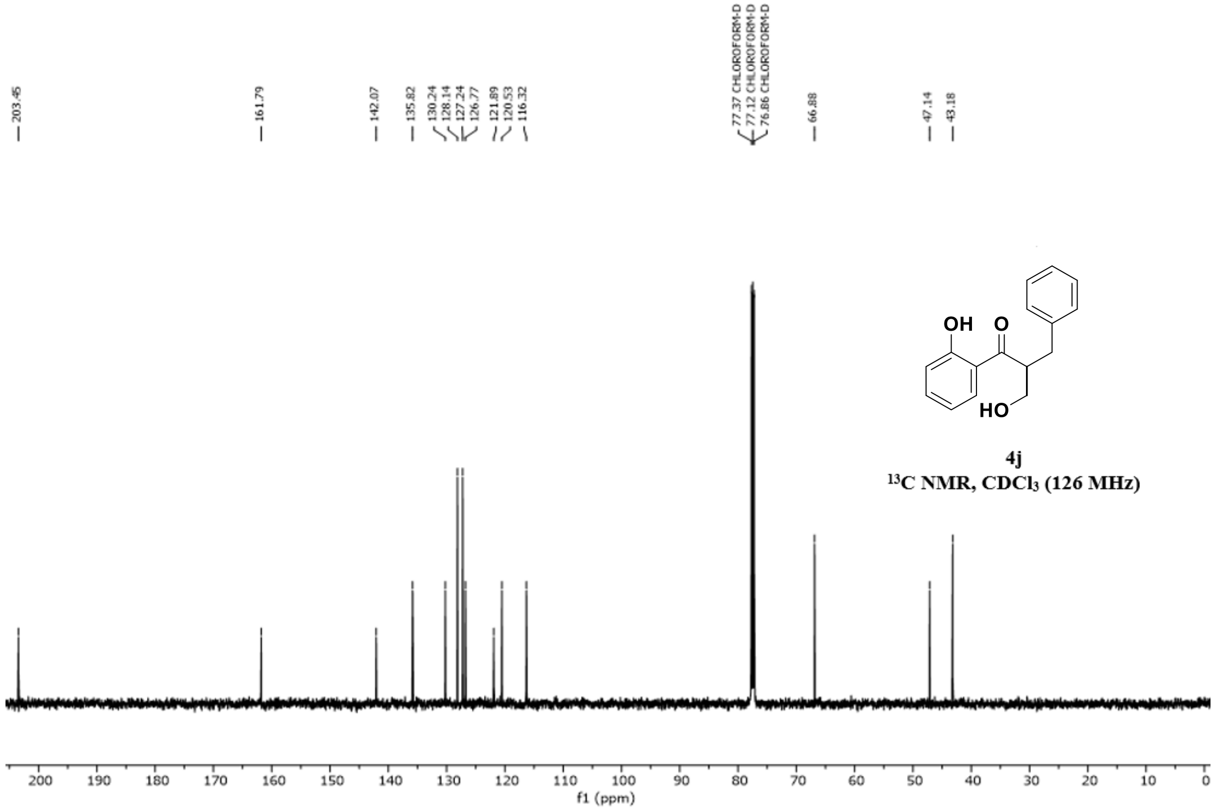
4i

^{13}C NMR, CDCl$_3$ (126 MHz)



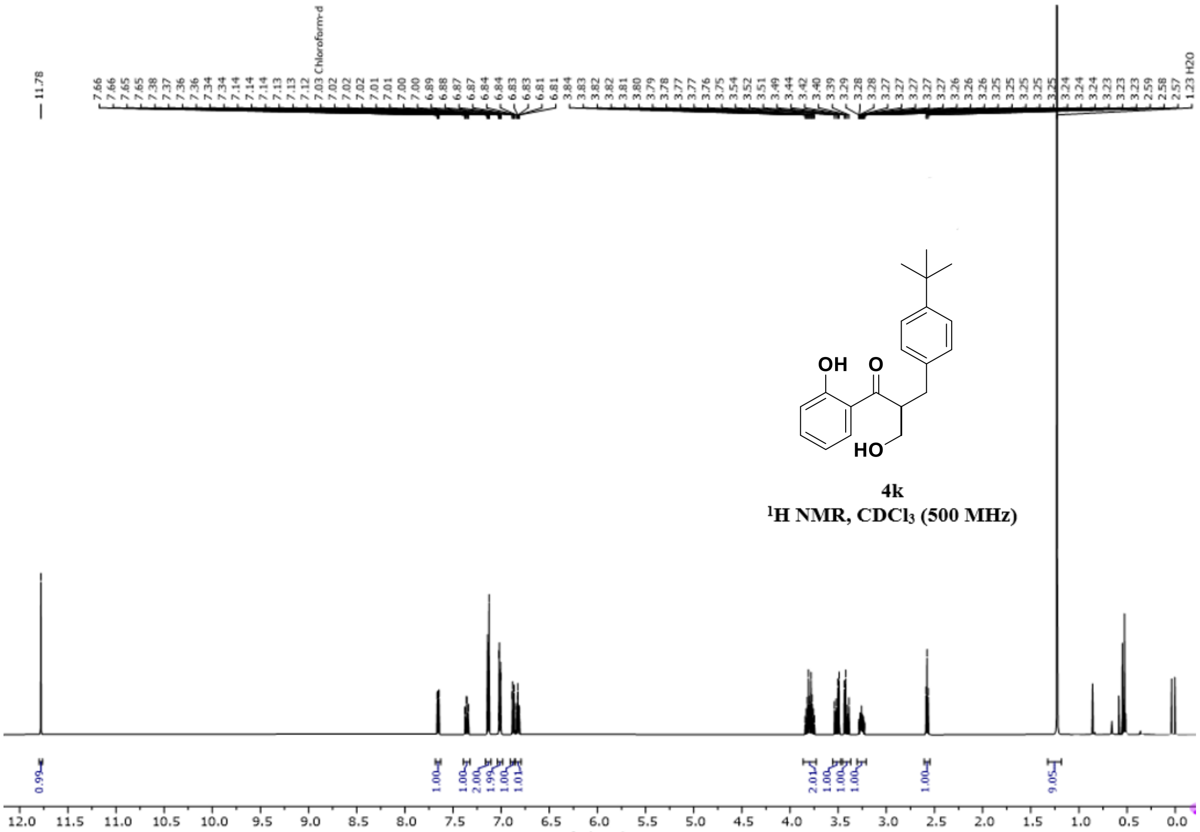
4j

^{1}H NMR, CDCl$_3$ (500 MHz)



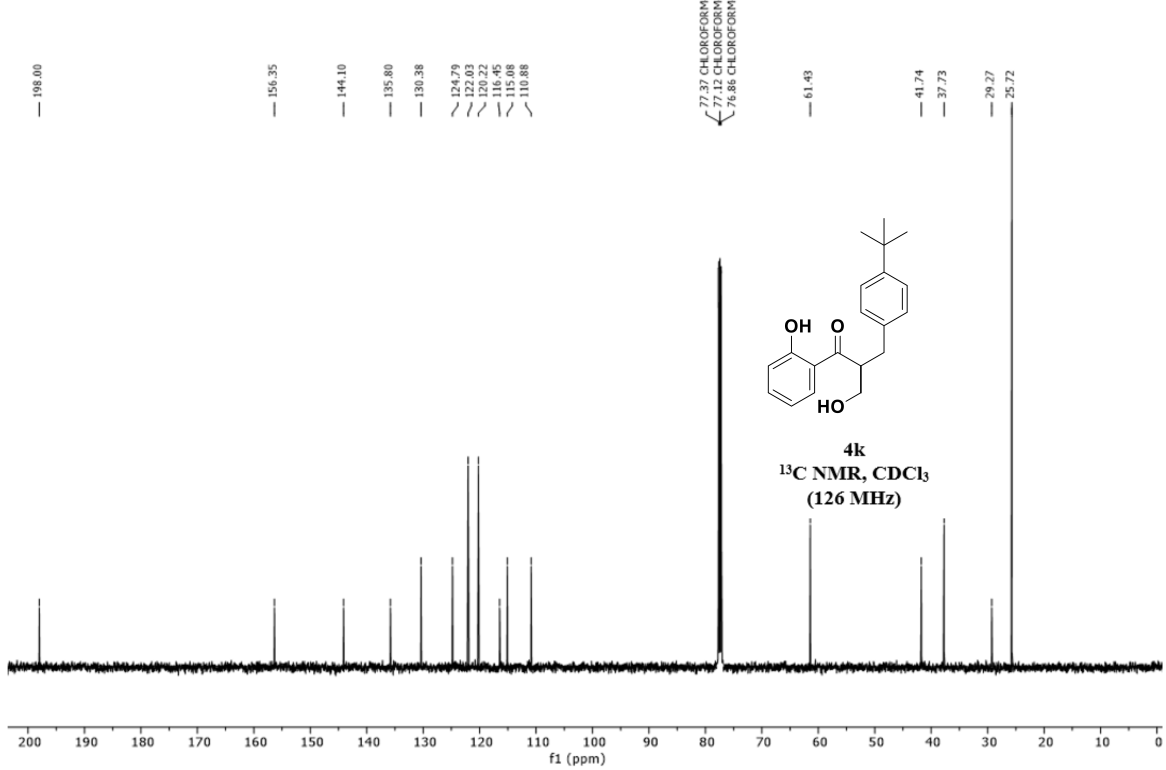
4j

^{13}C NMR, CDCl$_3$ (126 MHz)



4k

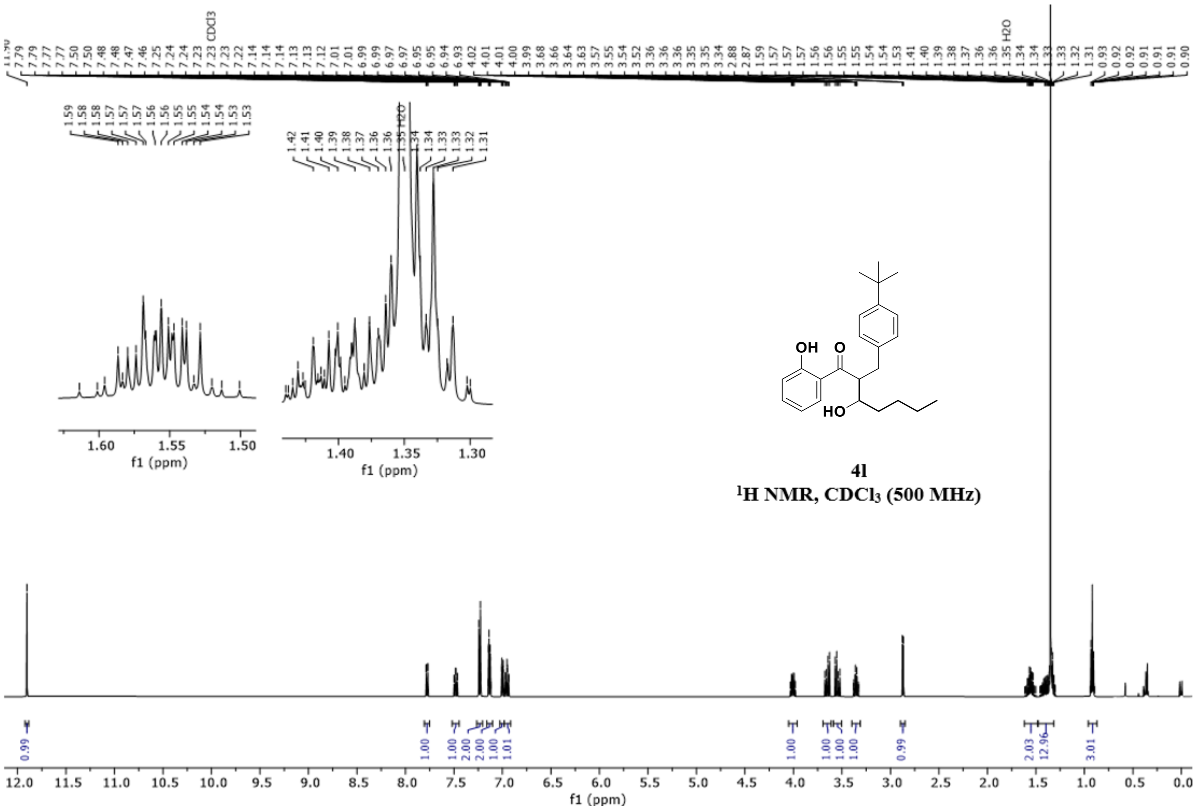
^{1}H NMR, CDCl$_3$ (500 MHz)



4k

^{13}C NMR, CDCl$_3$

(126 MHz)



4l

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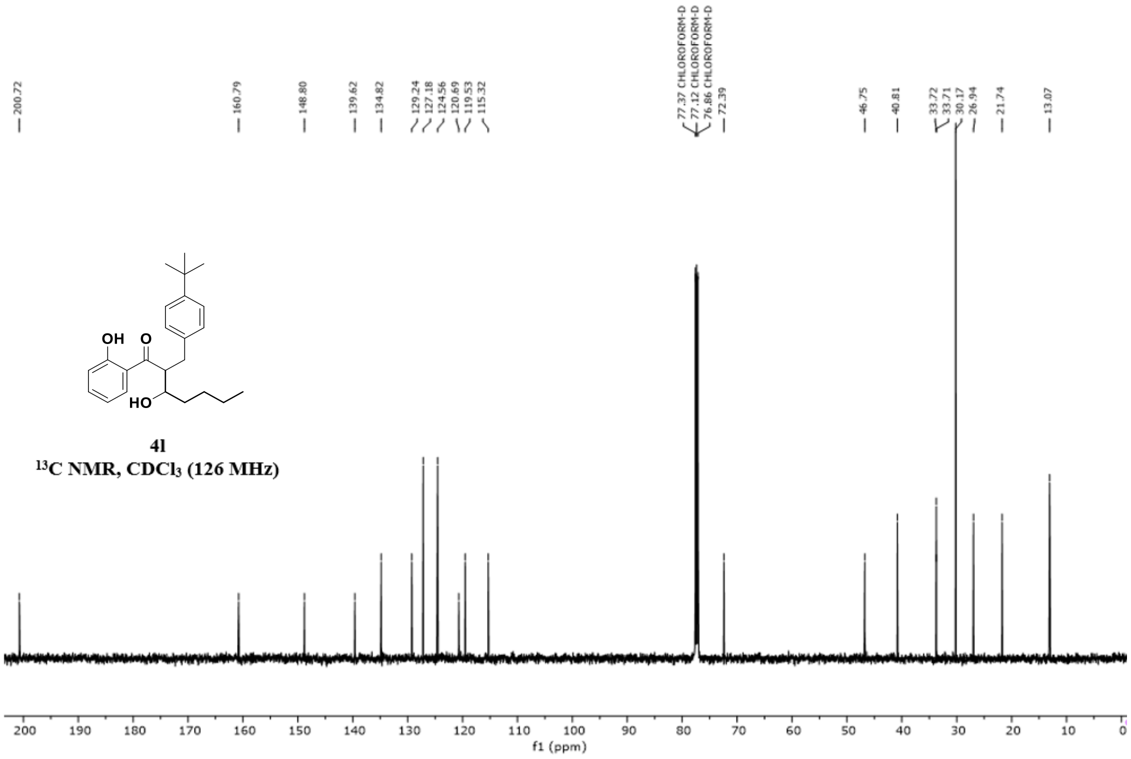

4l

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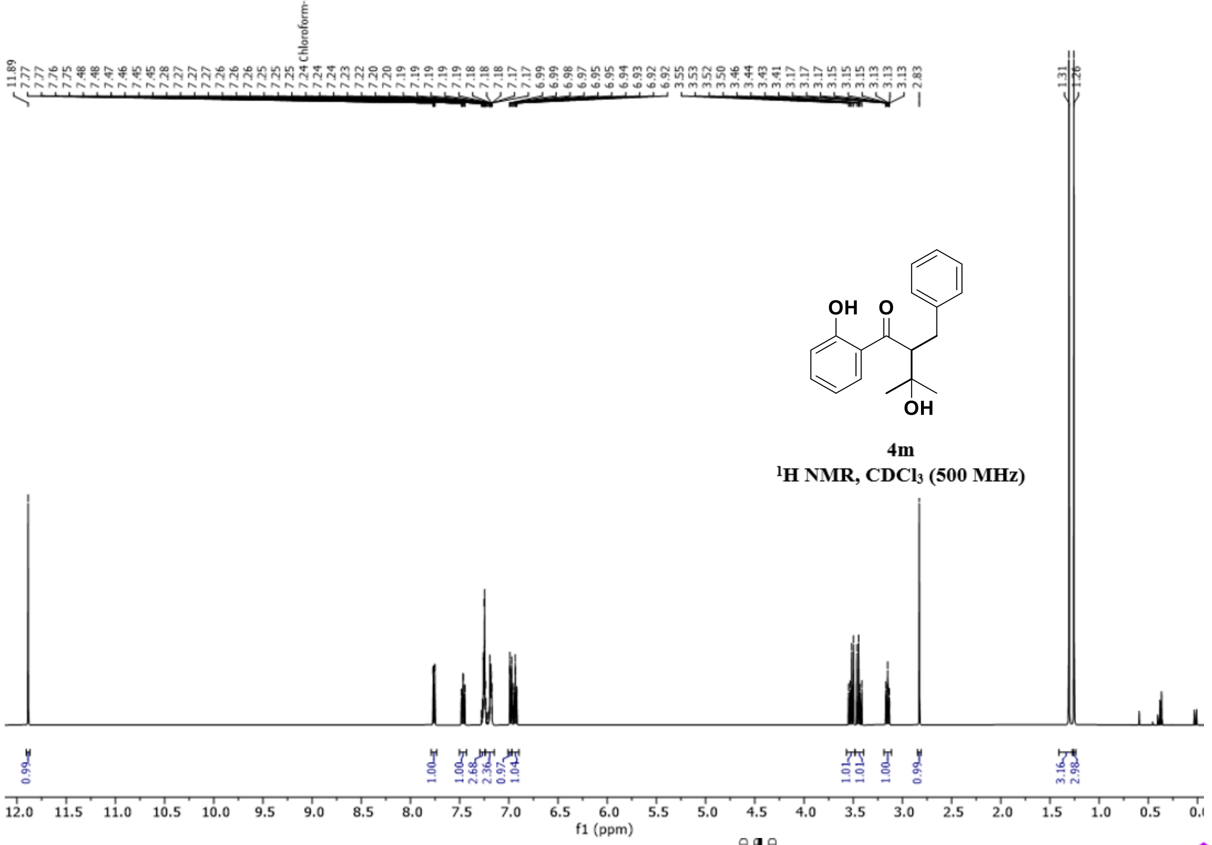

4m

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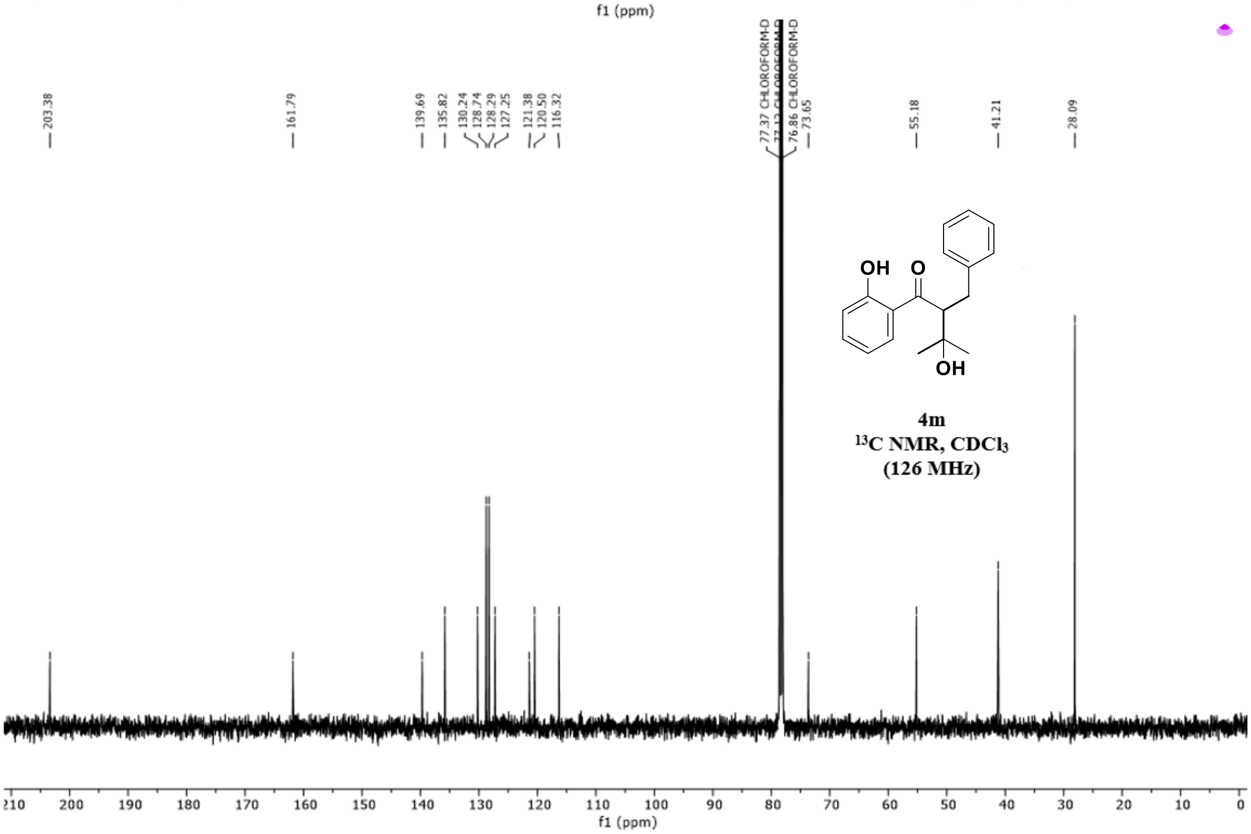
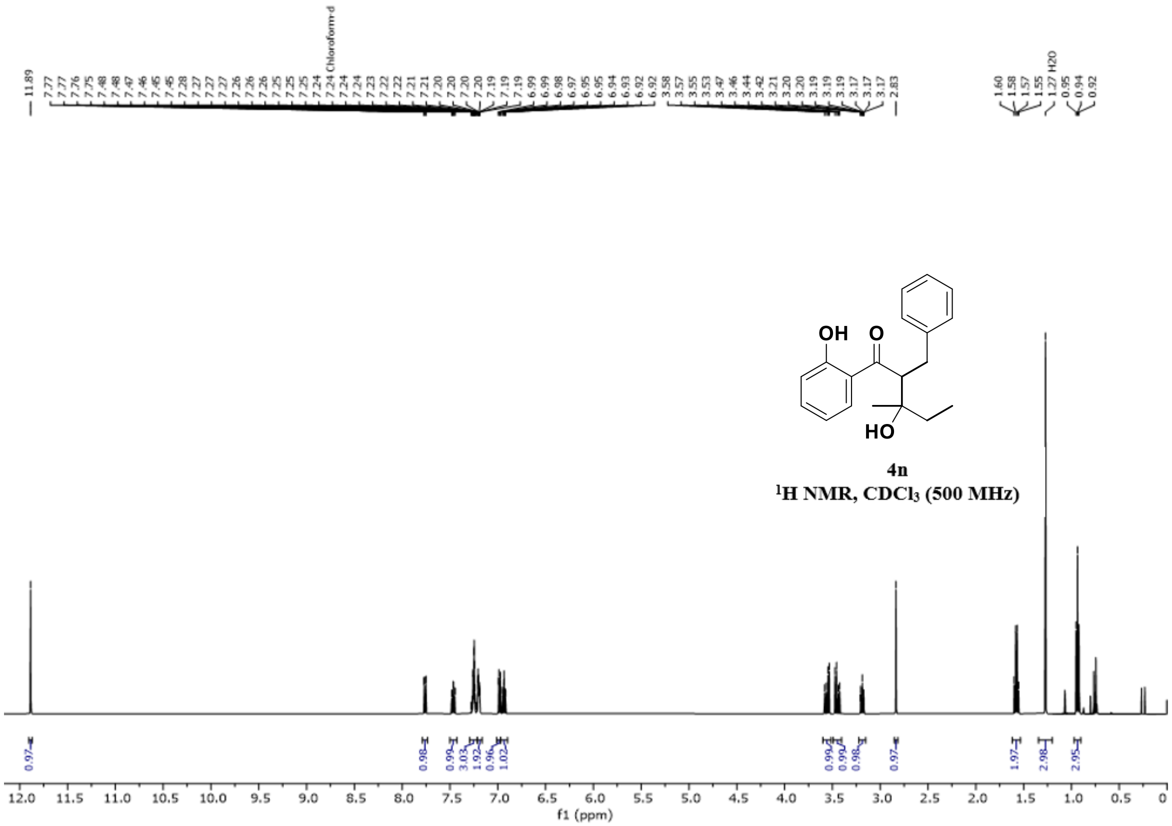


4m

^{13}C NMR, $CDCl_3$

(126 MHz)



4n

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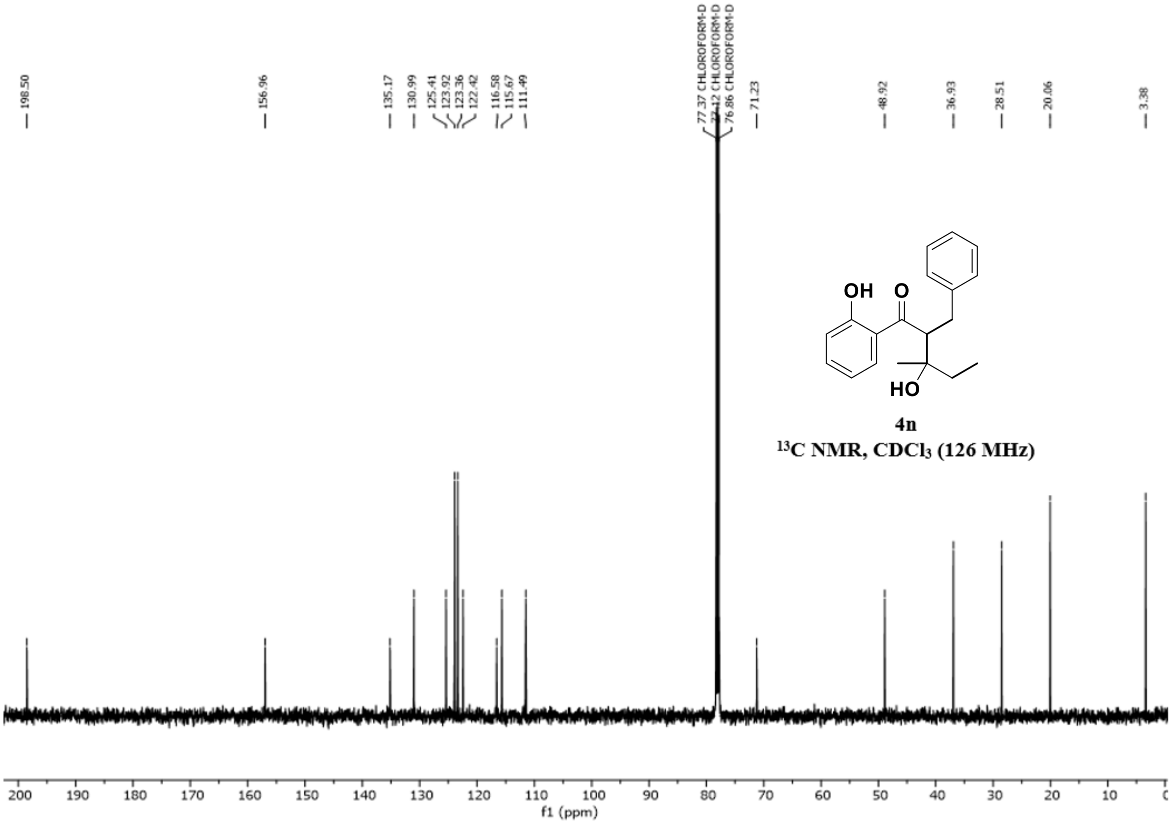
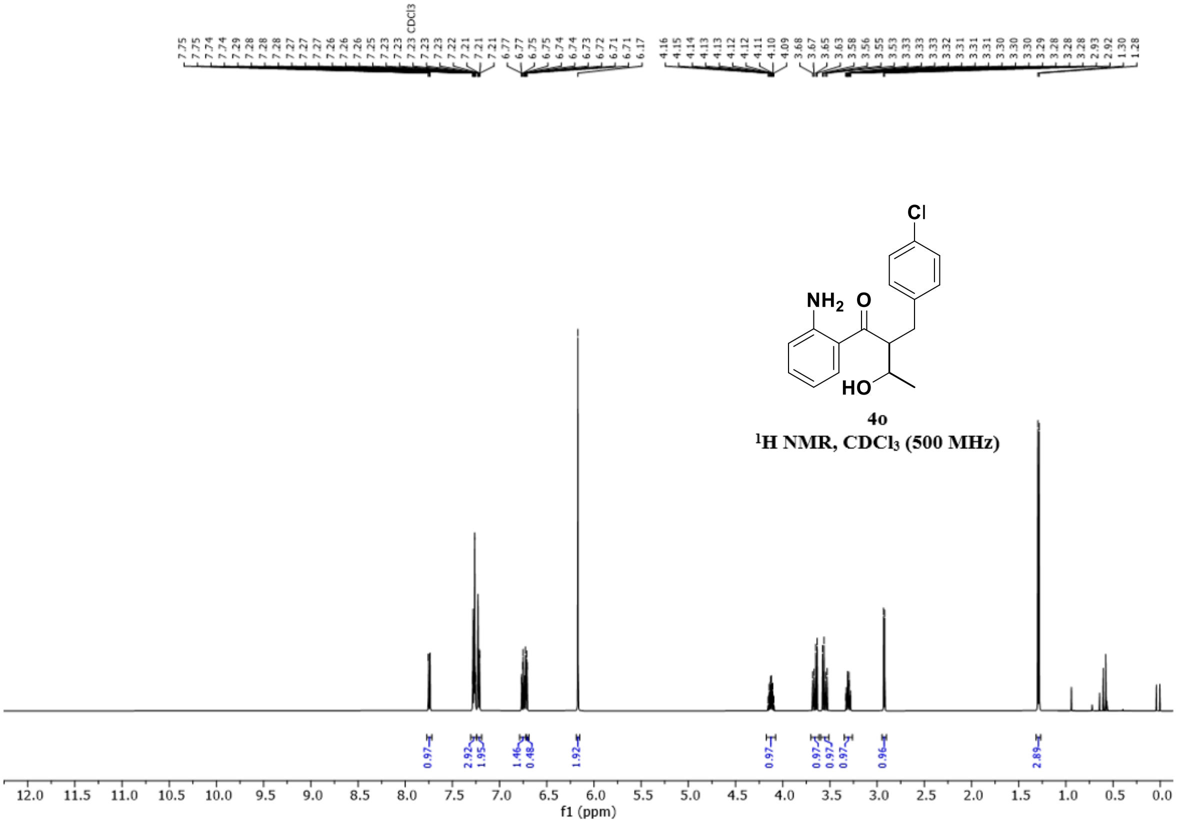


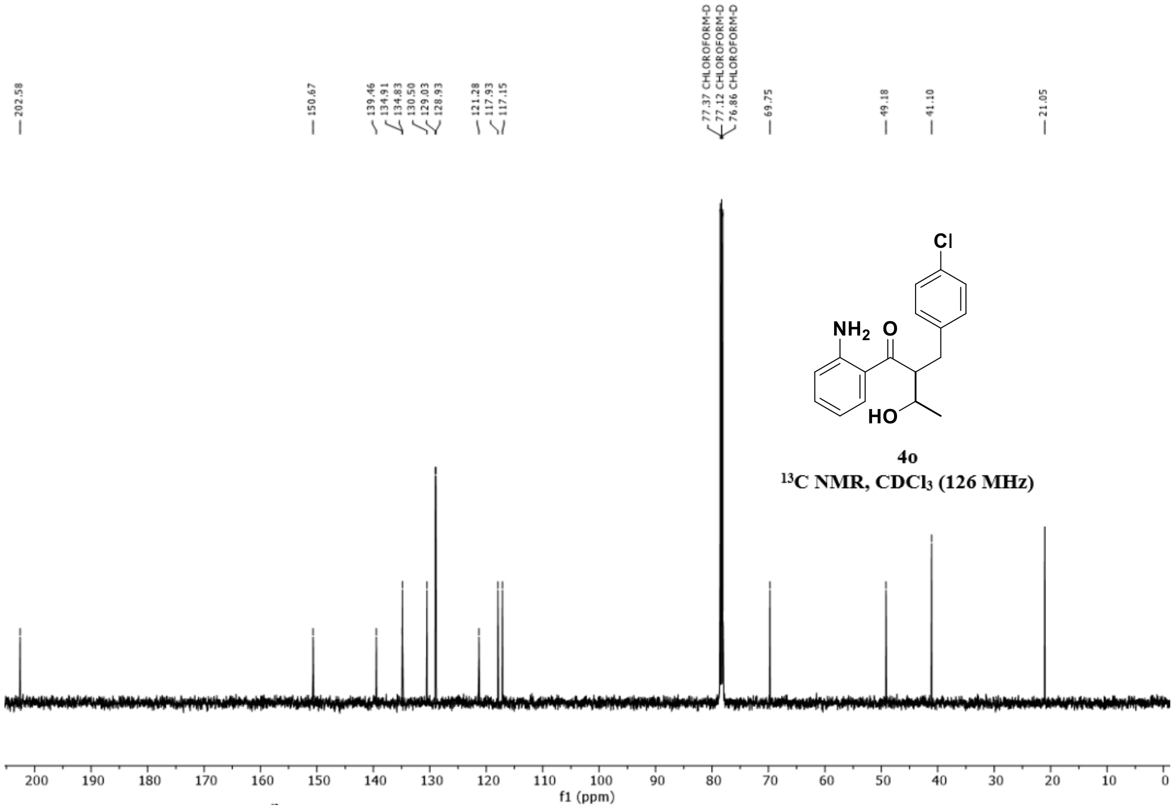
4n

^{13}C NMR, $CDCl_3$ (126 MHz)



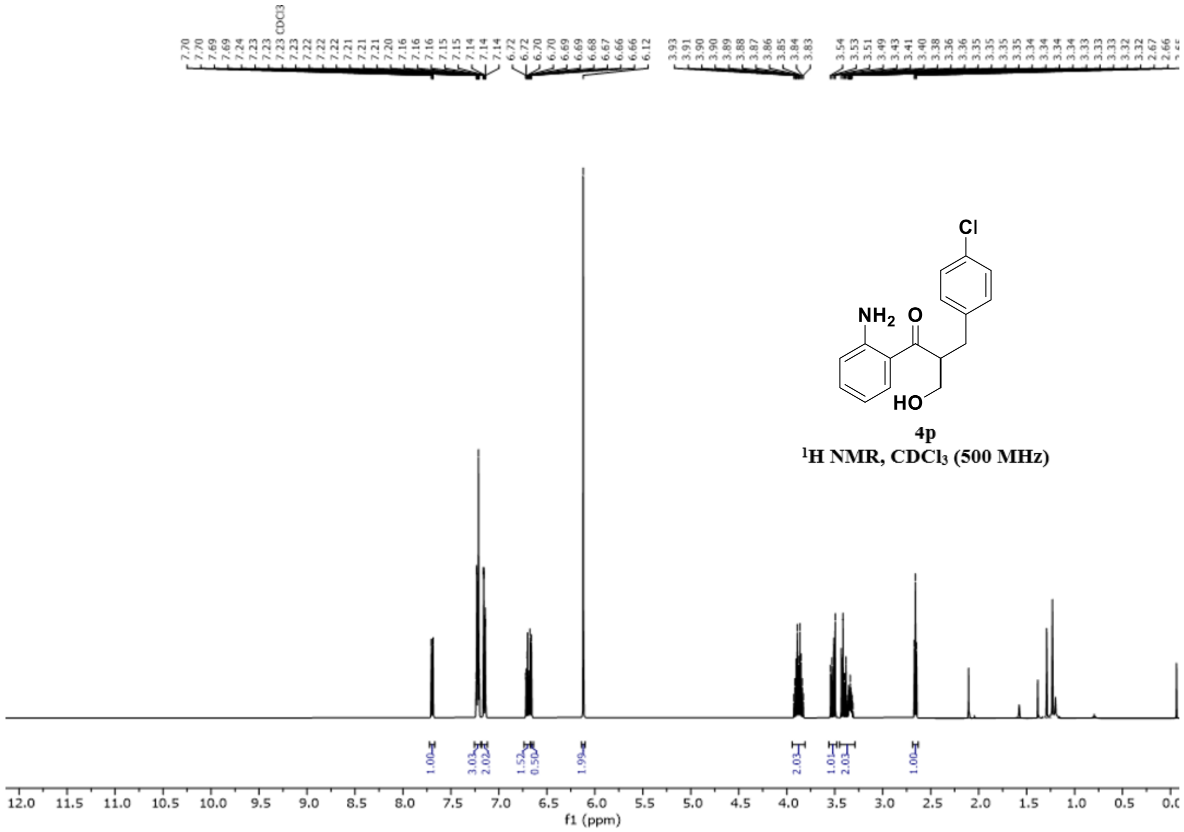
4o

^{1}H NMR, CDCl$_3$ (500 MHz)



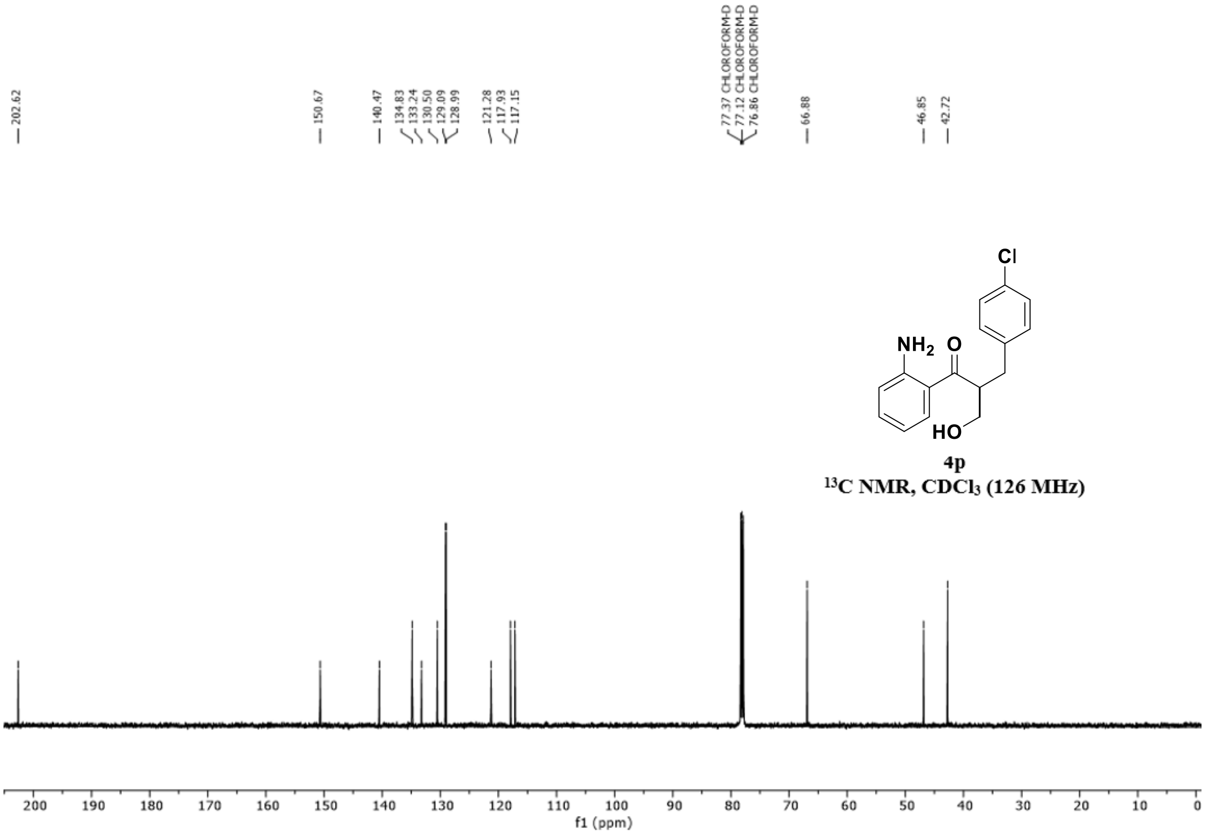
4o

^{13}C NMR, CDCl$_3$ (126 MHz)



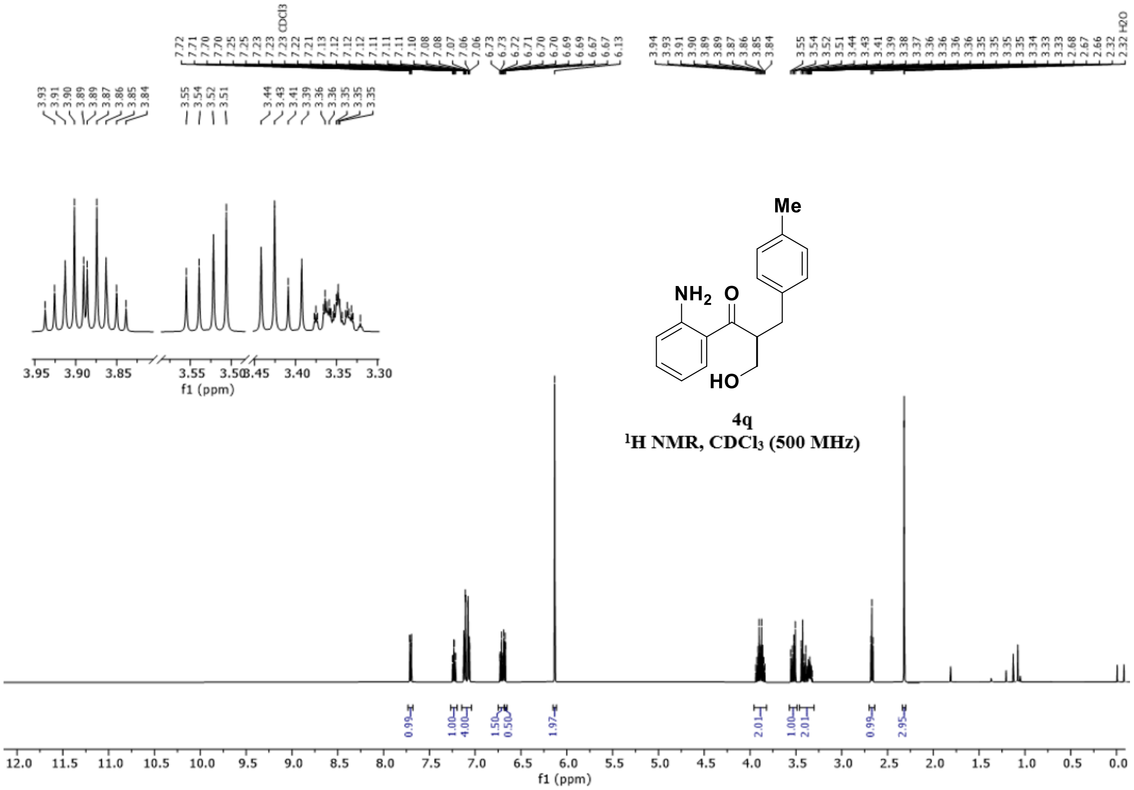
4p

^{1}H NMR, CDCl$_3$ (500 MHz)



4p

^{13}C NMR, CDCl$_3$ (126 MHz)



4q

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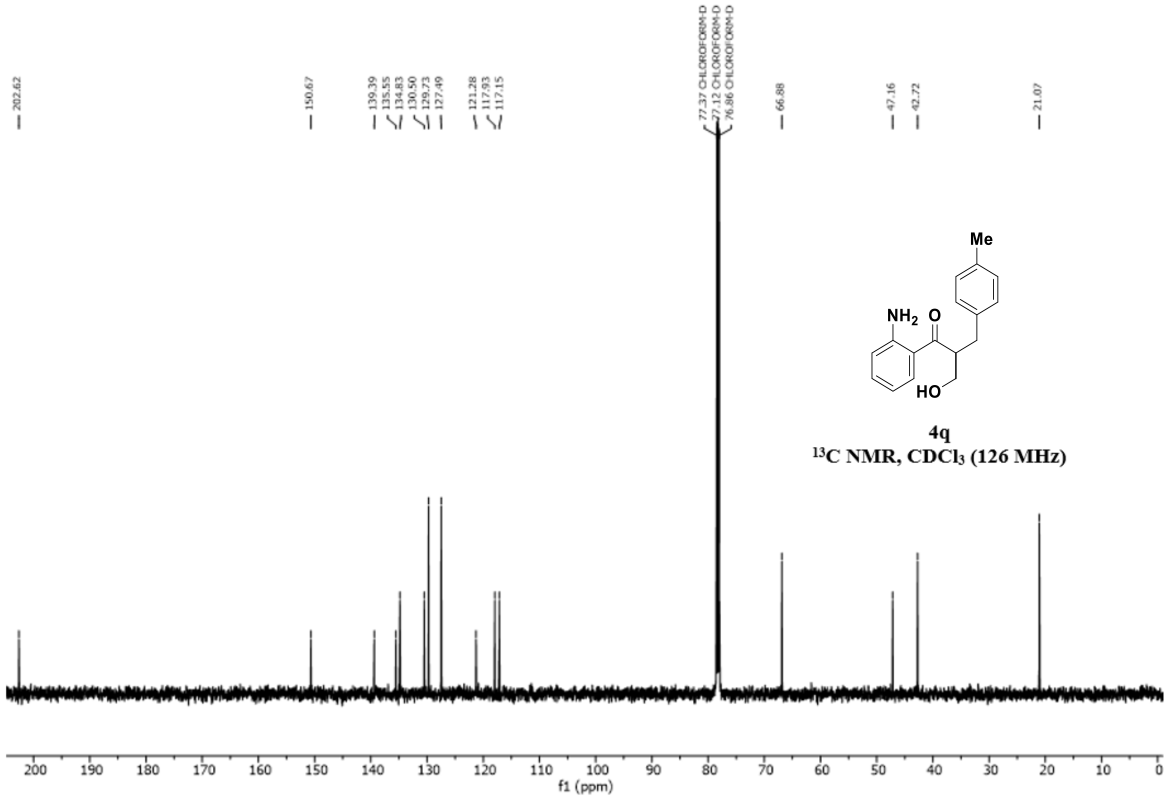
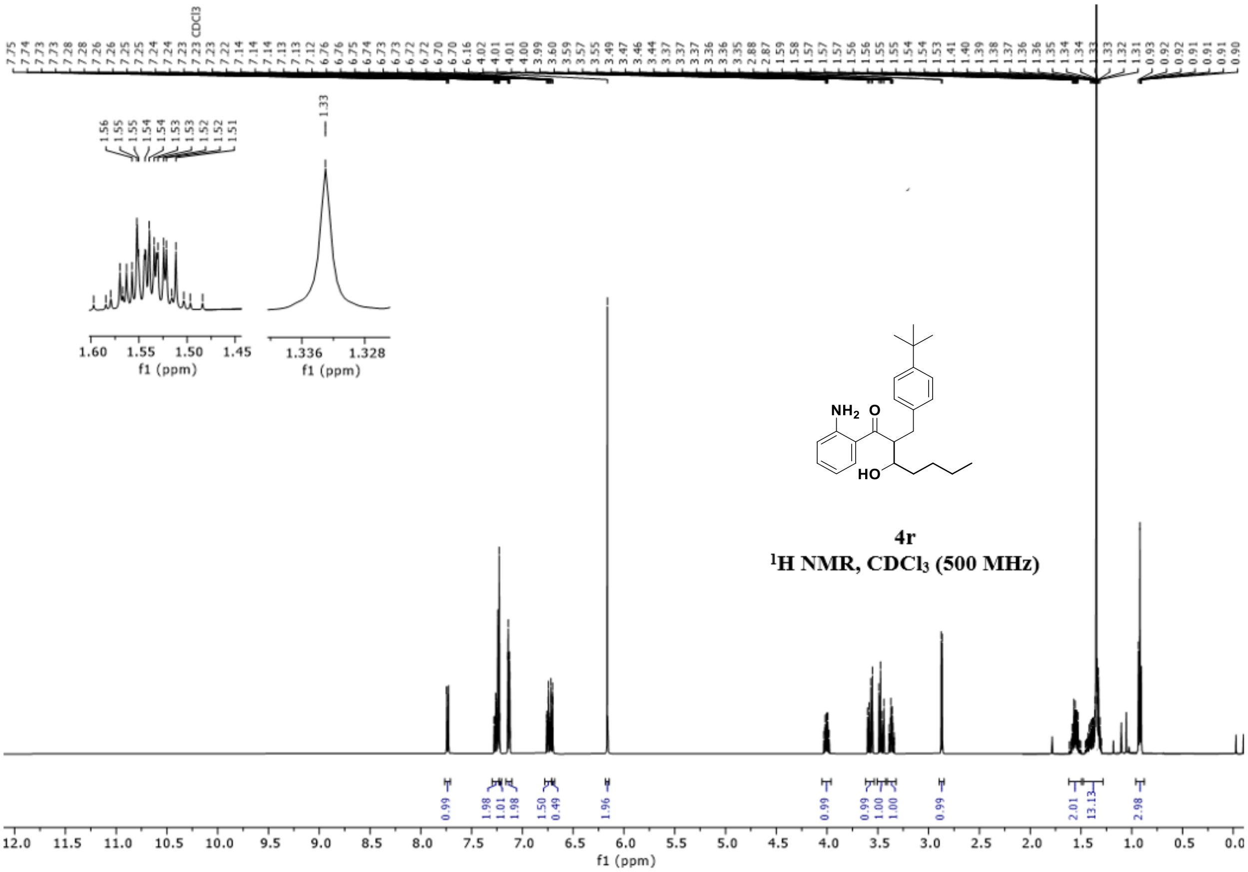


4q

^{13}C NMR, $CDCl_3$ (126 MHz)



4r

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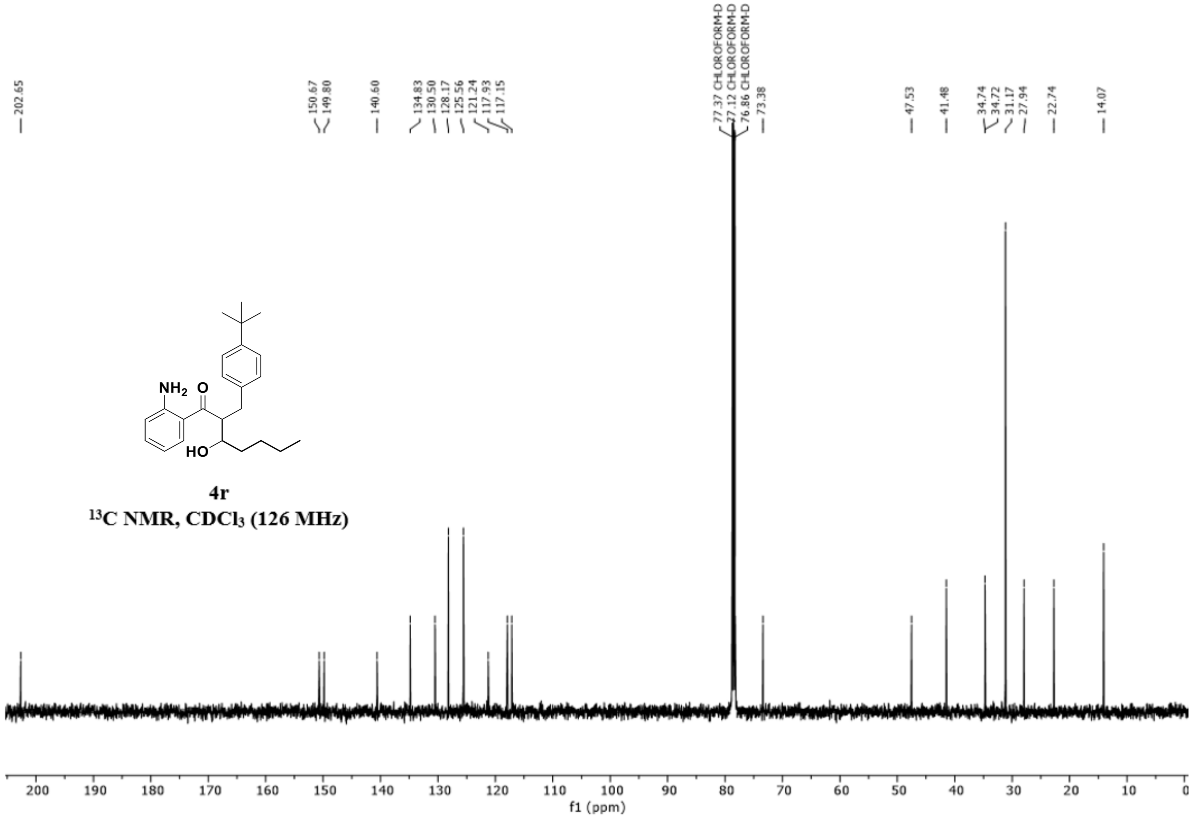
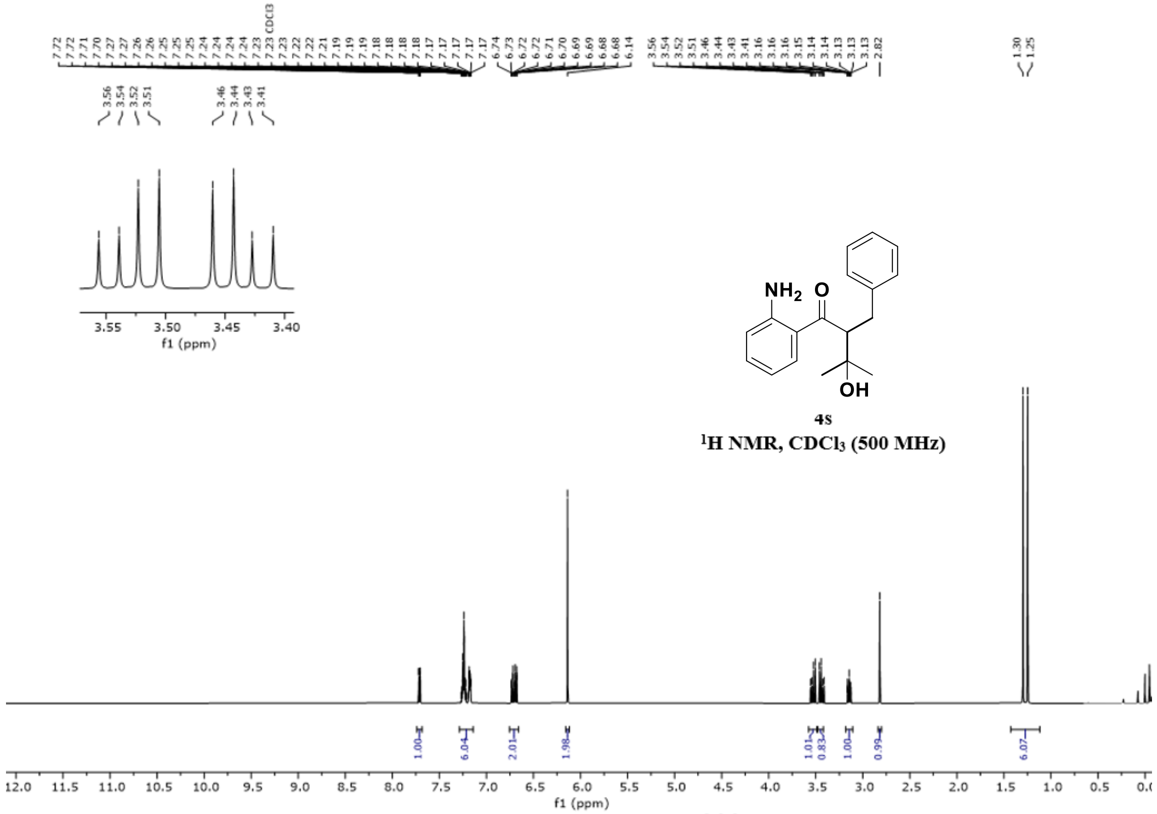


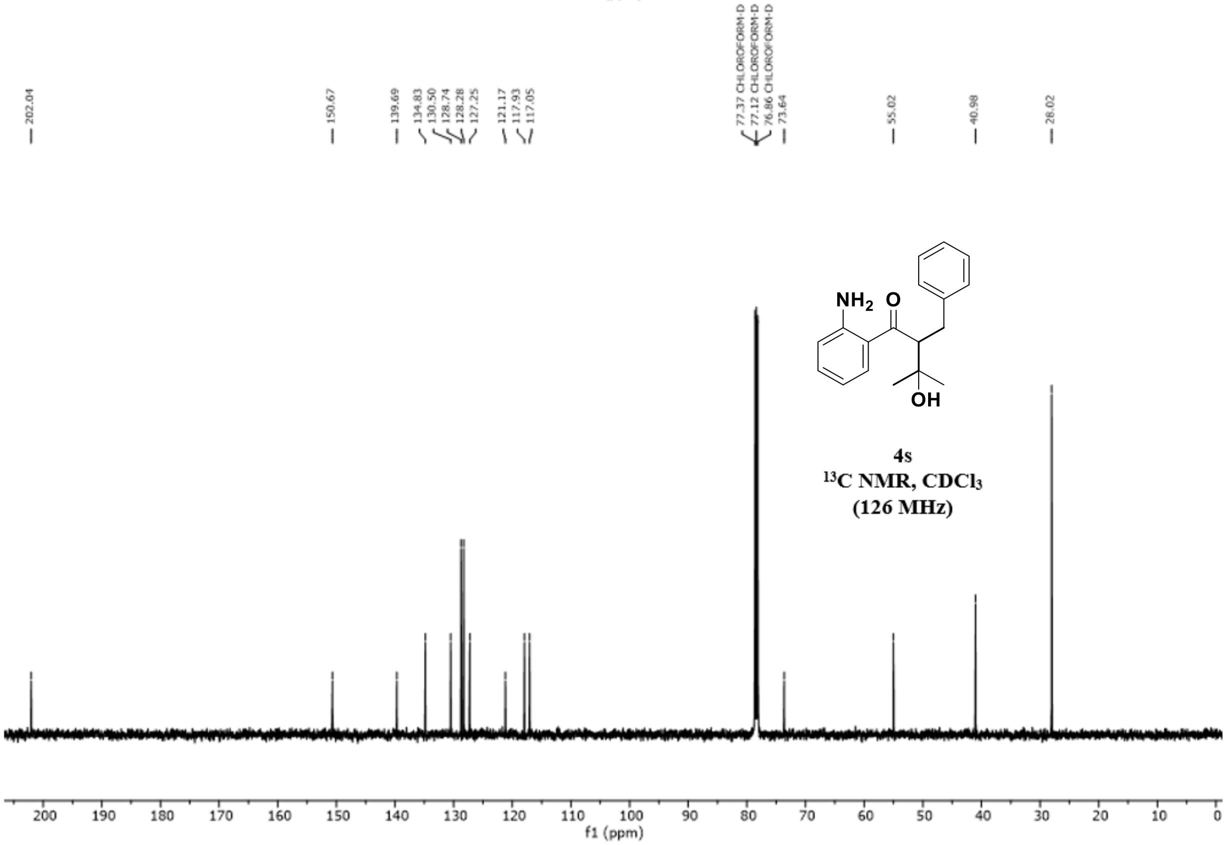
4r

^{13}C NMR, CDCl$_3$ (126 MHz)



4s

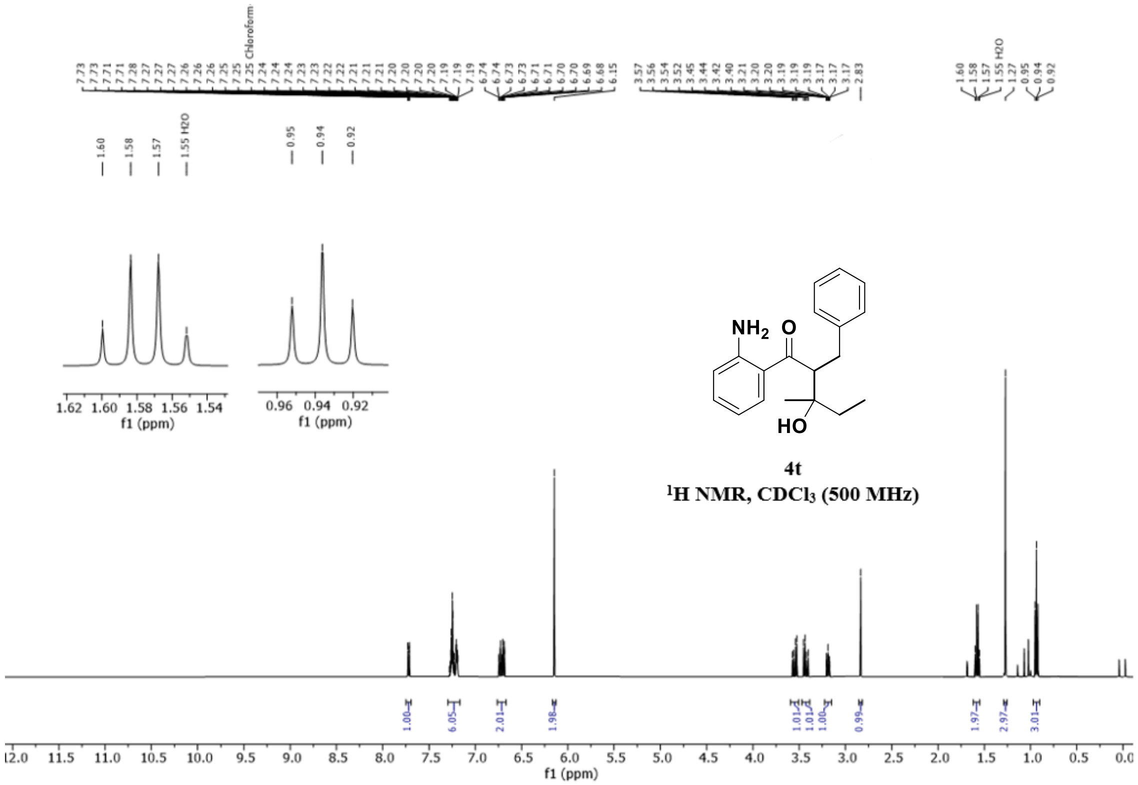
^{1}H NMR, CDCl$_3$ (500 MHz)



4s

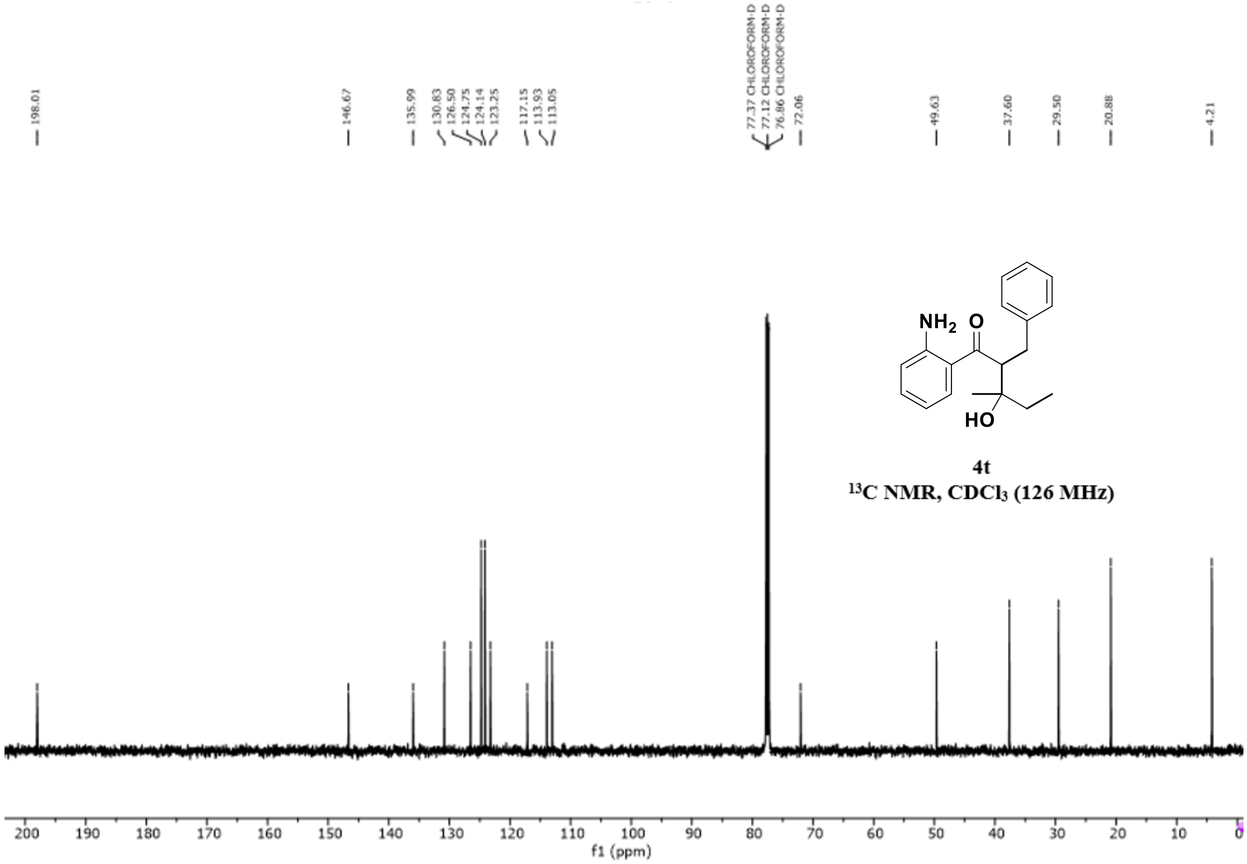
^{13}C NMR, CDCl$_3$

(126 MHz)



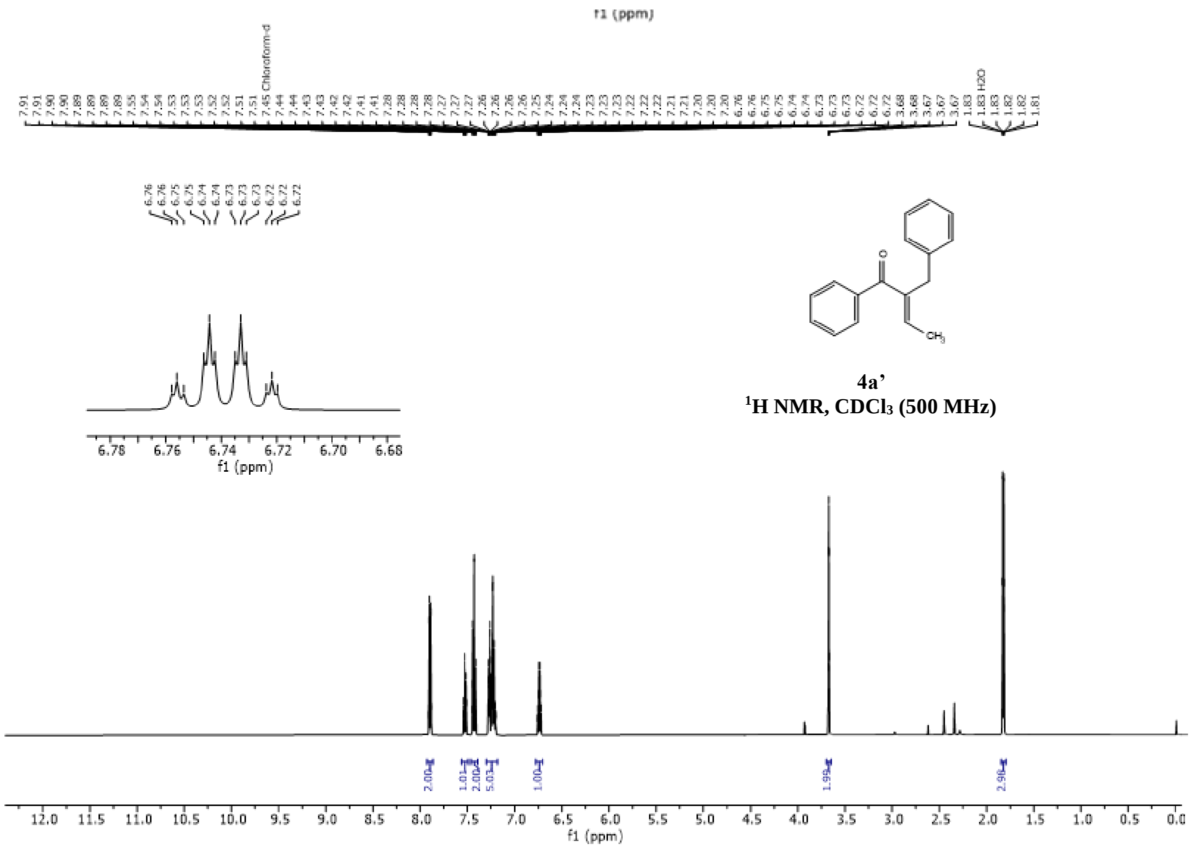
4t

^{1}H NMR, $CDCl_3$ (500 MHz)

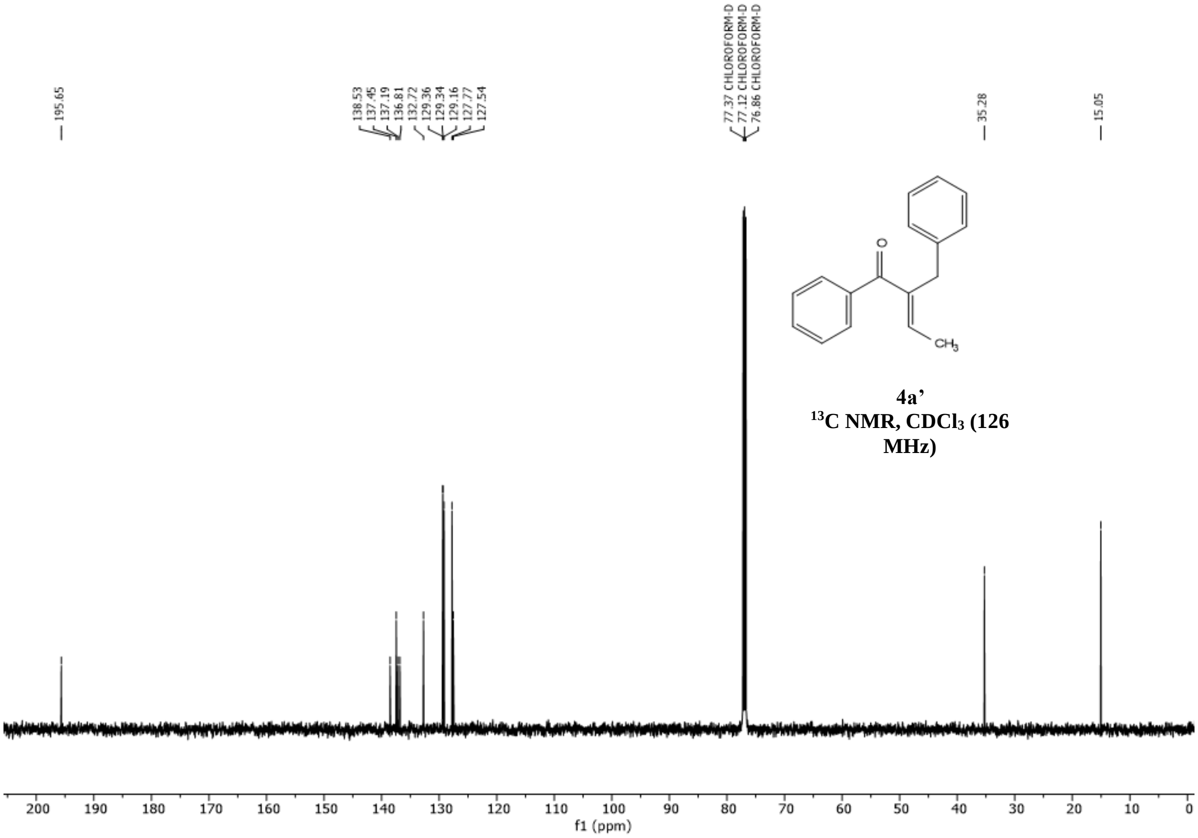


4t

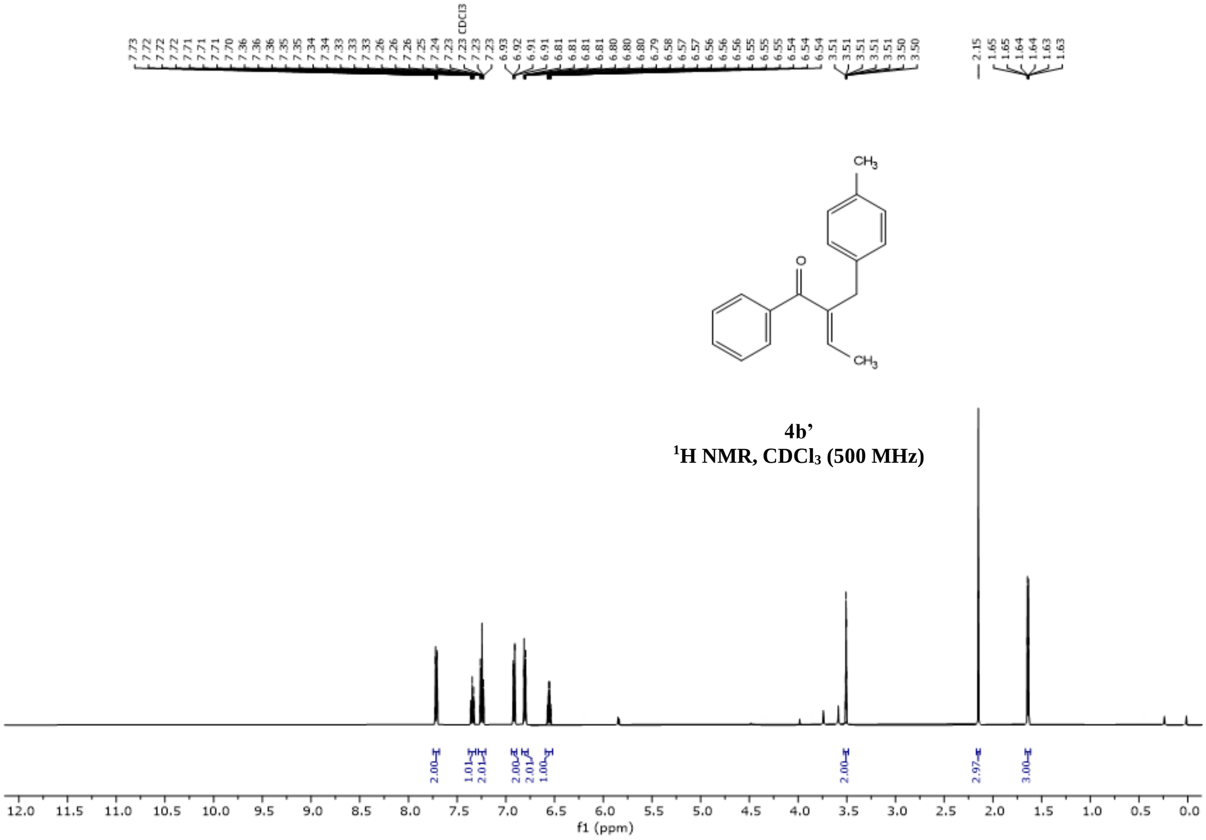
^{13}C NMR, $CDCl_3$ (126 MHz)



4a’
^{1}H NMR, $CDCl_3$ (500 MHz)



4a’
^{13}C NMR, $CDCl_3$ (126 MHz)



4b’
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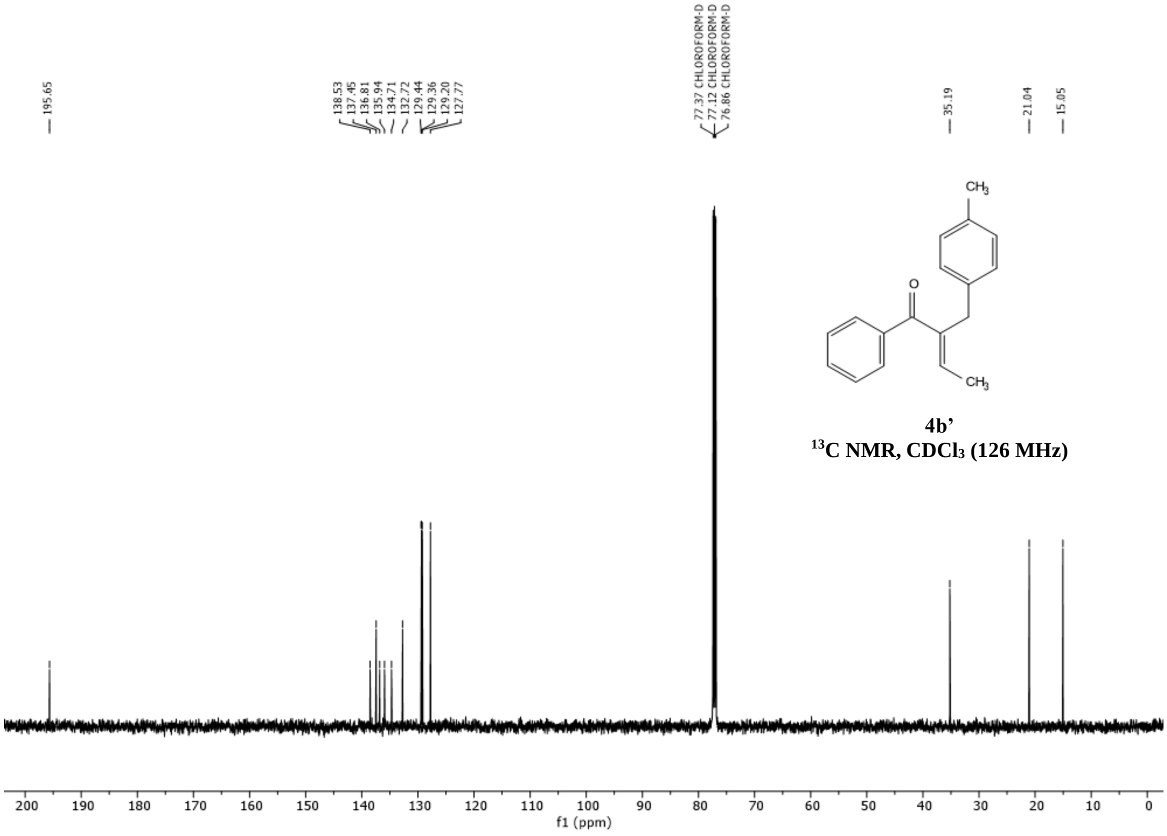

4b’
^{13}C NMR, CDCl$_3$ (126 MHz)