Graphite-Catalysed Synthesis of phenolic Schiff's Bases: Rapid, Efficient and Environmentally benign Synthetic Approach

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Abstract

Herein we report a rapid and straightforward approach for the synthesis of phenolic Schiff bases employing graphite, a versatile form of carbon, as an effective heterogeneous catalyst. The synthesis involved the reaction of differently substituted aromatic aldehydes with o-amino phenol in ethanol solvent under reflux conditions, utilizing a catalytic amount of readily available graphite powder.

This environmentally benign procedure offers several notable advantages including high product yields, the accessibility of the catalyst, simplified workup processes, short reaction times and advantages of recycling. Exploring the use of graphite as a catalyst in organic synthesis reveals its potential as an efficient and sustainable alternative. The method's simplicity and efficiency make it a promising pathway for the preparation of phenolic Schiff bases, showcasing the versatility and applicability of graphite in catalytic processes.

Keywords: Schiff's base, o-amino phenol, graphite, heterogenous catalyst, green synthesis.

Introduction

The concept of green chemistry[2,13] and its role in advancing cleaner and more environmentally friendly chemical processes has become a significant solution in the realm of synthetic organic chemistry. A range of approaches and strategies have been devised to achieve this goal. Currently, there has been a noteworthy focus on environmentally friendly synthetic techniques and one of these approaches involves the utilization of sustainable catalysts that includes natural catalyst[3], solid-supported reagents[26,28], phase transfer catalysts[16], metal-bases nanoparticles[1,20] and so on.

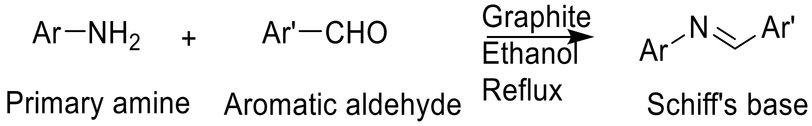
Recently, carbonaceous materials like activated carbon, carbon nanotubes, graphene and graphene oxide have widespread application, both as catalysts themselves and as catalyst supports in various chemical synthesis processes[15,18]. Among these materials, graphite, which represents a versatile form and allotrope of carbon, has attracted substantial interest as a promising alternative for metal free heterogeneous catalyst, often overlooked in its role as a catalyst. Graphite exhibits great potential due to its unique structural and chemical properties as it has layered structure that comprises of hexagonally arranged carbon atoms, thus providing an extensive surface area with numerous active sites for catalysis.

There are few reports in literature using unmodified graphite as catalyst in various organic transformations such as benzoylation of aromatic compounds[12], cleavage of ethers[12], oxidation[29], reduction of nitrobenzene by hydrazine[14], Friedel-Crafts alkylation[24], quinoxalines synthesis[10] and Michael addition[22].

In the present study, we have shown that graphite can be employed as an effective and green heterogenous catalyst towards the synthesis of Schiff's bases (Scheme 1). Schiff's bases are important class of organic compounds possessing diverse applications in many fields including biological[9,11], inorganic[6,21,27] and analytical chemistry.[4,5,25] These compounds are often synthesised by condensation of primary amines with either an aldehyde or a ketone in presence of acid catalyst.

However, this conventional approach involves rigorous reaction conditions and laborious workup procedures. Therefore, due to the continued interest in this area of research, there is a demand for the development of a more convenient, efficient and simple method for synthesizing Schiff bases.

In literature, there are quite a few reports that have showcased the use of heterogenous catalysts to improvise the drawbacks for this transformation. Some of the catalyst are: $Cu(NO_3)_2$[17], neutral Al_2O_3[30], P_2O_5-Al_2O_3[30], CaO[7], SiO_2[8] and montmorillonite[19].



Scheme 1: Preparation of Schiff Base

https://doi.org/10.25303/295rjce1280144

Material and Methods

General Preparation of Schiff Bases 3a-i: A 50mL round bottom flask was charged with 2-aminophenol **1** (1 mmol), aromatic aldehyde 2a-i (1 mmol) and commercially available graphite powder (10mg) in ethanol (5mL). The mixture was kept for refluxing for appropriate time as mentioned in table 1. The reaction was continuously monitored on TLC (20% ethyl acetate- petroleum ether mixture) to check for formation of product. After completion of the reaction, the reaction mixture was filtered while hot to remove the catalyst and then it was concentrated. The solid product obtained was purified by recrystallisation using ethanol as solvent. The structures of all the products were established based on literature melting points and spectral analysis (^{1}HNMR and ^{13}CNMR).[25]

General Preparation of Schiff Bases 5: A 50mL round bottom flask was charged with o-Phenylenediamine (1 mmol), salicylaldehyde (2 mmol) and commercially available graphite powder (10mg) in ethanol (5mL). The mixture was kept for refluxing for about 30 minutes. The reaction was continuously monitored on TLC (20% ethyl acetate- petroleum ether mixture) to check for formation of product. After completion of the reaction, the reaction mixture was filtered while hot to remove the catalyst and concentrated. The solid product 5 obtained was purified by recrystallisation using ethanol as solvent. The structures of all the products were established based on literature melting points and spectral analysis (^{1}HNMR and ^{13}CNMR).

2-(Benzylideneamino)phenol Compound 3a: Brown solid, melting point 110-112°C (112-114°C), H^{1}NMR (CDCl$_3$): δ (ppm)= 13.0 (s, 1H), 8.73 (s, 1H), 8.01-7.96 (m, 2H,), 7.56-7.52 (m, 3H), 7.51-7.35 (m, 2H), 7.24-7.22 (m, 1H), 6.97-6.93 (m, 1H), C^{13}NMR (CDCl$_3$): δ(ppm) = 157.1, 152.4, 147.0, 135.8, 135.5, 129.0, 128.9, 128.85, 120.14, 115.9, 115.05.

2-(2-Nitrobenzylideneamino) phenol Compound 3b: Yellow solid, melting point 138-140°C (reported=140-142°C). H^{1}NMR (CDCl$_3$): δ (ppm)= 8.73 (s, 1H), 7.96-7.94 (m, 2H,), 7.56-7.54 (m, 2H), 7.35-7.33 (m, 1H), 7.26-7.22 (m, 1H), 7.07-7.04 (m, 1H), 6.97-6.93 (m, 1H), C$_{13}$NMR (CDCl$_3$): δ(ppm) = 154.5,151.4,136.7, 134.17, 133.3,128.92, 128.27,128.24, 119.19,114.84,114.18.

2-(2-hydroxybenzylideneamino) phenol Compound 3c: Orange solid, melting point 162°C (reported=159-160°C). H^{1}NMR (CDCl$_3$): δ (ppm)= 13.77 (s, 1H), 9.67 (s, 1H), 8.92 (s,1H), 7.57 (m, 2H,), 7.36-7.31 (m, 2H), 7.10-6.86 (m, 4H), C^{13}NMR (CDCl$_3$): δ(ppm)= 162.02,160.78, 151.41, 135.63, 133.11, 128.35, 120, 119.19, 119.07, 117.12, 116.98, 116.87.

2-(4-N, N-dimethylaminobenzylideneamino) phenol Compound 3d: Maroon solid, melting point 110°C (reported=112°C). H^{1}NMR (CDCl$_3$): δ (ppm)= 9.79 (s, 1H), 8.58 (s, 1H), 7.83-7.82 (m, 2H,), 7.30-7.28 (m, 1H), 7.17-7.14 (m, 1H), 7.05-7.03 (m, 1H), 6.94-6.91 (m, 1H), 6.77-6.76 (m,1H), 3.09 (s, 6H) C^{13}NMR (CDCl$_3$): δ(ppm) = 159.22,154.7, 153.93, 138.70, 132.60, 129.4, 126.03, 121.9, 117.67, 116.4, 113.6, 42.16.

2-(4-Methylbenzylideneamino)phenol Compound 3e: Brown solid, melting point 110°C (111-113°C)H^{1}NMR (CDCl$_3$): δ (ppm)= 9.92 (s, 1H), 8.60 (s, 1H), 7.77-7.76 (m, 2H,), 7.25-7.24 (m, 3H), 7.18-7.14 (m, 1H), 7.01-6.86 (m, 3H), 2.39 (s,3H) C^{13}NMR (CDCl$_3$): δ(ppm) = 157.18, 152.31, 142.34, 135.76, 135.76, 133.37, 126.6, 128.99, 128.7, 120.13, 115.93, 114.97, 21.72.

2-(4-Methoxybenzylideneamino)phenol Compound 3f: Light brown solid, melting point 98-100°C H^{1}NMR (CDCl$_3$): δ (ppm)= 9.92 (s, 1H), 8.65 (s, 1H), 7.91-7.89 (m, 2H,), 7.31-7.29 (m, 1H), 7.22-7.19 (m, 1H), 7.06-7.02 (m, 3H), 6.95-6.92 (m,1H), 3.91(s, 3H), C^{13}NMR (CDCl$_3$): δ(ppm) = 162.56, 156.65, 152.12, 135.95, 130.61, 128.84, 128.34, 120.09, 115.8, 114.82, 114.34, 55.5.

2-(4-Fluorobenzylideneamino)phenol Compound 3g: Light brown solid, melting point 82°C, H^{1}NMR (CDCl$_3$): δ (ppm)= .8.68 (s, 1H), 7.96-7.93 (m, 2H,), 7.92(m,1H), 7.39-7.30 (m, 3H), 7.25-7.04 (m, 1H), 6.96-6.92 (m,2H), C^{13}NMR (CDCl$_3$): δ(ppm) = 165.89,163.88, 155.69, 152.29, 135.36, 132.25, 132.22, 130.85, 130.78, 129.02, 120.17, 116.21, 116.04, 115.85, 115.10.

2-(4-chlorobenzylideneamino) phenol Compound 3h: Brown solid, melting point 108-110°C (reported=109-111°C). H1-NMR (CDCl3): δ (ppm)= 9.19 (s, 1H), 8.29-8.27 (m, 1H,), 8.09-8.09(m,1H), 7.79-7.75 (m, 1H), 7.68-7.64 (m, 1H), 7.39-7.37 (m, 1H), 7.30-7.28 (m, 1H), 7.17-6.94 (m,2H), C13NMR (CDCl3): δ(ppm) = 160.18, 152.0,149.4, 134.75, 133.4, 131.43, 130.5, 130.25, 129.55, 124.71, 120.39, 116.42, 115.49.

2-(4-nitroobenzylideneamino) phenol Compound 3i: Brown solid, melting point 160°C (reported=164-166°C). H^{1}NMR (CDCl$_3$): δ (ppm)= 9.65 (s, 1H), 8.93 (s, 1H,), 8.03-7.82 (m, 2H), 7.53-7.38 (m, 2H), 7.13-6.81 (m, 4H), 7C^{13}NMR (CDCl$_3$): δ(ppm) = 157.81, 152.94, 142.97, 136.39, 134.0, 130.29, 129.52, 129.32, 125.00, 120.76, 116.56, 115.60.

Bis(salicylaldehyde)phenylenediimine Compound 5: Orange solid, melting point 162°C (reported=160°C). H1-NMR (CDCl$_3$): δ (ppm)= 13.057 (s, 2H), 8.630 (s, 2H), 7.387-7.37 (m, 6H,), 7.35-7.33 (m, 2H), 7.23-7.06 (m, 2H), 6.93-6.07 (m, 2H), C13NMR (CDCl3): δ(ppm) = 117.54, 118.97, 119.22, 119.71, 127.70, 132.34, 133.36, 142.54, 161.34, 163.71.

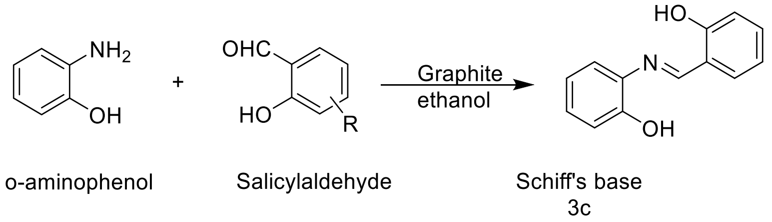
Results and Discussion

To initiate with our work towards synthesis of Schiff's bases, we chose salicylaldehyde (1mmol) and o-amino phenol (1mmol) as the model substrate in ethanol as solvent and

30mg of commercially available graphite powder as catalyst at room temperature under stirring condition (Scheme 2). The reaction was continuously monitored on thin layer chromatography (TLC) using petroleum ether-ethyl acetate mixture (8:2 ratio respectively). After approximately three hours of continuous stirring, the reaction had reached completion which was confirmed on TLC.

Next, we carried out the same reaction under refluxing condition and interestingly we observed that the reaction was complete in one hour. This observation indicated the temperature is a crucial factor for the above synthetic transformation. After finalizing, the next task was to find the optimum catalyst loading for effective conversion. So, we carried out the reaction using different amounts of catalyst load and revealed that a catalyst concentration of 15 mg was sufficient for sustaining the reaction and achieving the highest isolated yield (93% yield, entry 4 in table 1). Once the catalyst loading and temperature conditions were optimised, we further carried out the reaction (scheme 3) using different solvents. The solvents employed for the reaction were water, ethanol, ethanol-water mixture (1:1v/v), methanol, methanol-water mixture (1:1 v/v). Interestingly, when absolute ethanol was employed as the solvent, the desired product was obtained with an exceptional yield (entries 1-9 as shown in table 2).

From the above findings, it can be concluded that the optimum reaction conditions required for the synthesis of Schiff's base are condensation of salicylaldehyde (1mmol) and o-amino phenol (1mmol) as the model substrate in ethanol as solvent under refluxing conditions in the presence of 15 mg of graphite as catalyst.



Scheme 2: Schiff base reaction of salicylaldehyde and phenol

Table 1
Effect of catalyst loading on scheme 2.

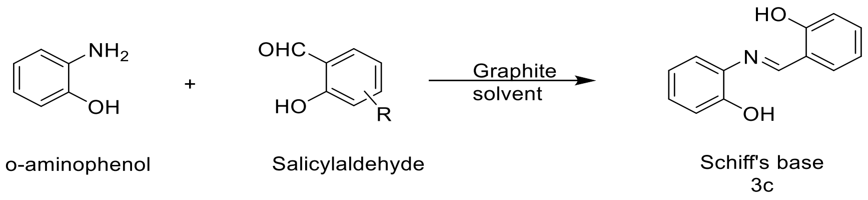
Entry	Catalyst in mg [a]	% yield [b]
1	0	35
2	5	65
3	10	68
4	15	93
5	20	85
6	30	65

[a] Reaction conditions: o-amino phenol 1 (1.0 mmol), salicylaldehyde 2**c** (1.0 mmol), graphite (15mg), ethanol (3 mL) at reflux conditions; [b] isolated yield.

Table 2
Screening of different solvents for the synthesis of Schiff's base (3c)

Entry	Solvents [a]	% yield [b] 3c
1	Water	50
2	Methanol+water (1:1 v/v)	70
3	Methanol	85
4	Ethanol+water (1:1 v/v)	77
5	Ethanol	93
6	Neat	65

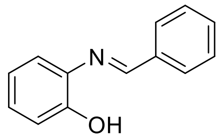
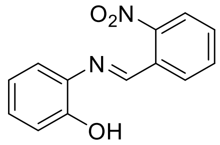
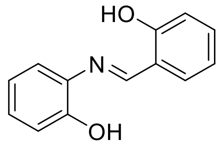
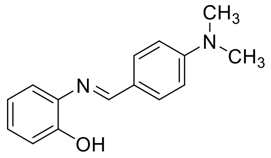
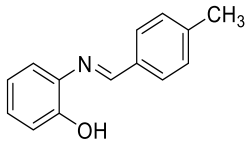
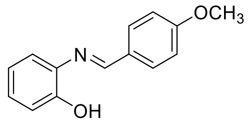
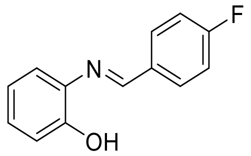
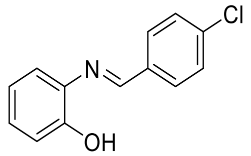
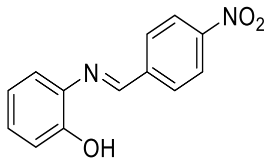
[a] Reaction conditions: o-amino phenol 1 (1.0 mmol), salicylaldehyde 2**c** (1.0 mmol), graphite (15mg), solvent (3 mL) at reflux conditions; [b] isolated yield.



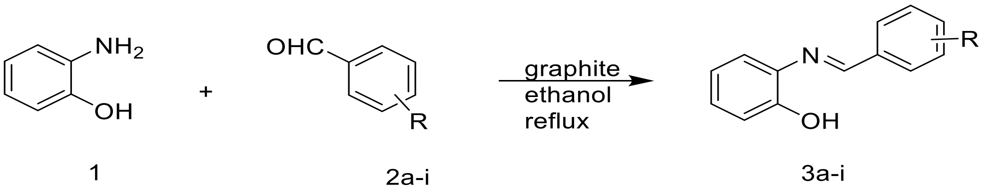
Scheme 3: Schiff base reaction of salicylaldehyde and phenol

To evaluate the effectiveness of the optimized parameters, various differently substituted aromatic aldehydes 2a-i were condensed with o-aminophenol 1 (scheme 4) to give the corresponding phenolic Schiff's bases 3a-i (entries 1-9, Table 3) in excellent yields. Though the role of graphite has not been clearly understood, a plausible mechanism for product, Schiff's base, is proposed as presented in scheme 5.

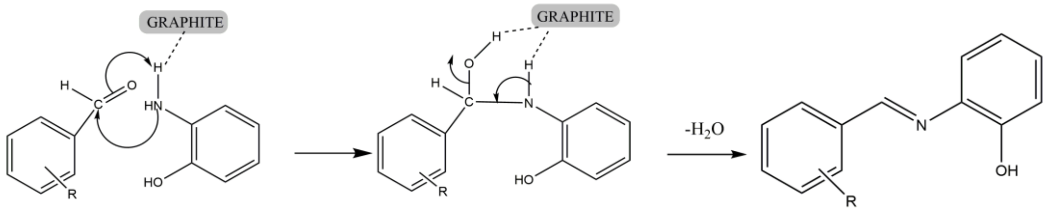
Table 3
Syntheses of phenolic Schiff's bases via condensation of aromatic aldehydes and o-aminophenol in presence of graphite catalyst.

Entry	Aromatic Aldehydes 2a-g R	Product Schiff's base. 3a-g	Reflux time [a] in minutes	% yield [b]
a	H		30	85
b	2-NO_2		10	94
c	2-OH		30	93
d	4-$N(CH_3)_2$		10	89
e	4-CH_3		20	86
f	4-OCH_3		20	87
g	4-F		15	82
h	4-Cl		30	88
i	4-NO_2		15	90

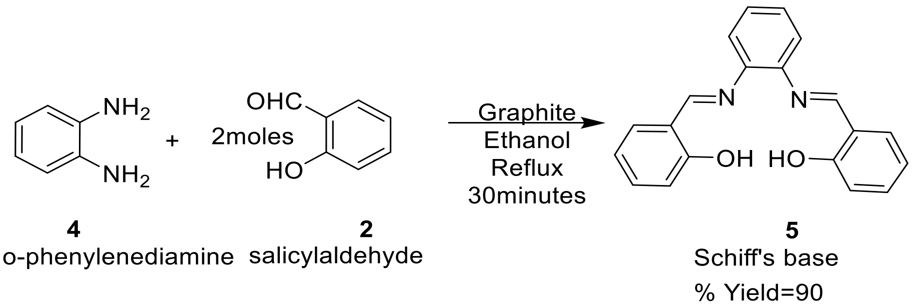
[a] Reaction conditions: o-amino phenol 1 (1.0 mmol), salicylaldehyde 2**c** (1.0 mmol), graphite (15mg), ethanol (3 mL) at reflux conditions; [b] isolated yield.



Scheme 4: Schiff base reaction of benzaldehyde and phenol



Scheme 5: Plausible mechanism of Schiff base reaction



Scheme 6: Schiff base reaction of salicylaldehyde and phenol

Table 4
Catalyst Recyclability

Entry	Run[a]	% yield [b] 3c
1	0	93
2	1	90
3	2	88
4	3	85
5	4	85

[a] Reaction conditions: o-amino phenol 1 (1.0 mmol), salicylaldehyde 2c (1.0 mmol), graphite (15mg), ethanol (3 mL) at reflux conditions; [b] isolated yield.

Furthermore, we applied the optimized conditions to the condensation reaction between salicylaldehyde 2c and o-phenylenediamine 4 in a 2:1 mole ratio (Scheme 6). This reaction was carried out under reflux conditions in ethanol, with the addition of a graphite catalyst. Remarkably, the desired Schiff's base product 5 was successfully obtained with an excellent yield of 90% within just 30 minutes of refluxing.

The catalyst could be easily separated by simple filtration, washed with ethyl acetate or acetone and recovered by drying it in oven at 100°C, maintaining its active state. We investigated the catalyst's reusability for the reaction between salicylaldehyde (2c) and o-aminophenol (1) in a fresh reaction cycle and found that it consistently provided satisfactory yields for up to 4 consecutive cycles, as outlined in the table 4.

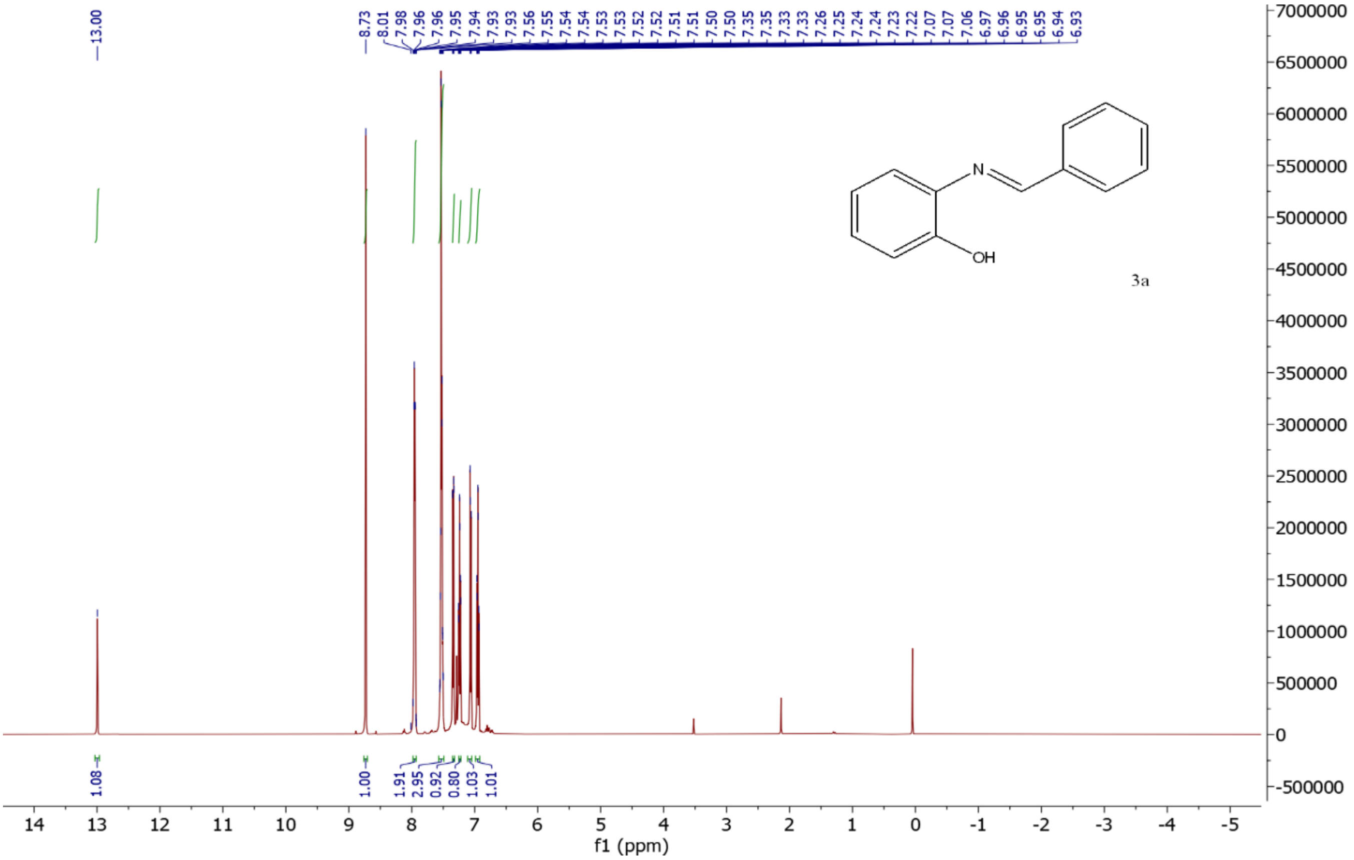
Conclusion

In conclusion, we have reported a rapid, simple and green method for synthesizing phenolic Schiff bases by employing graphite, a highly versatile form of carbon, as an efficient heterogeneous catalyst. This environmentally friendly approach offers several distinct advantages over the conventional methods including high yields of the desired products, easy access to the catalyst, simplified workup procedures and short reaction times.

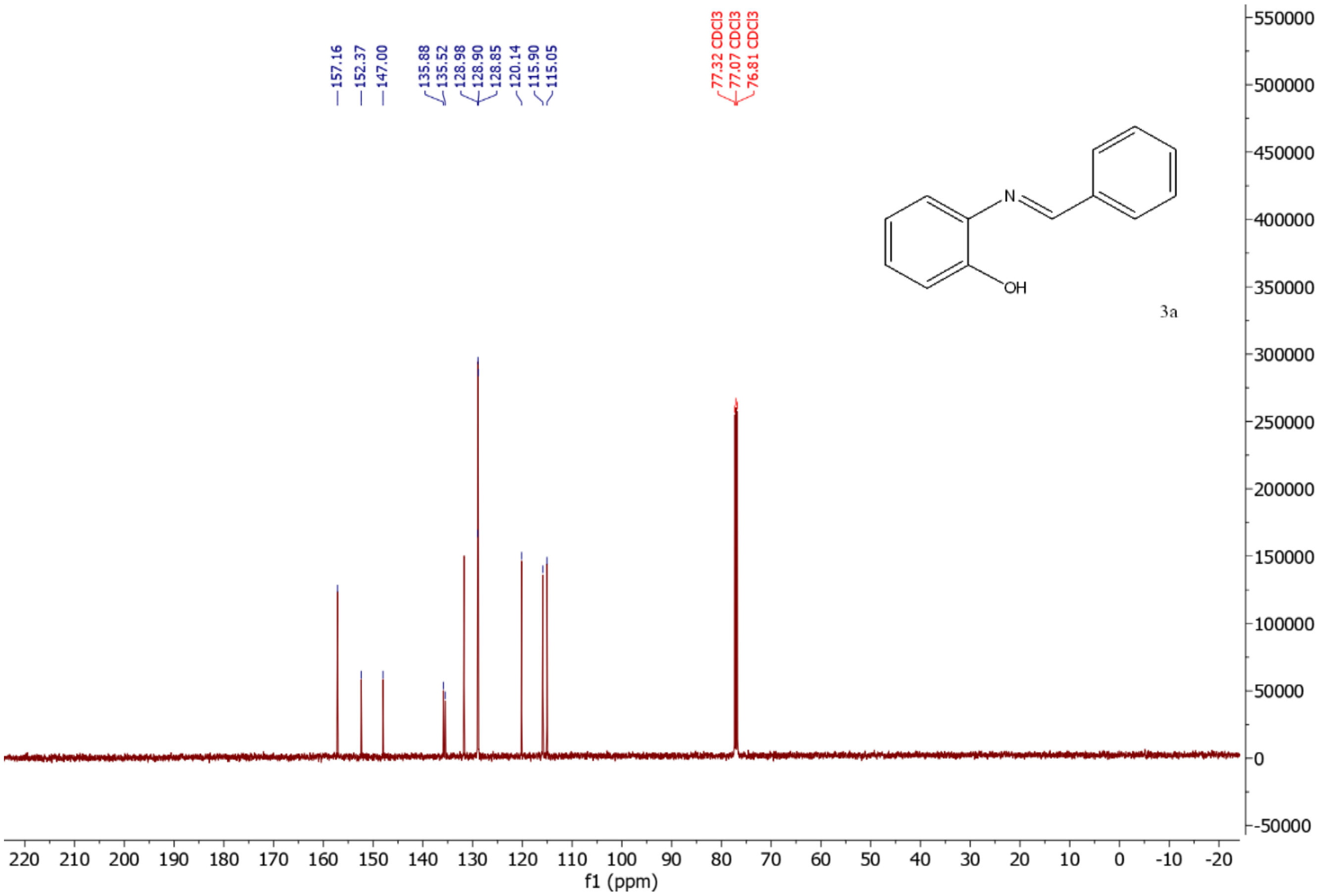
Also, the catalyst could be easily separated and successfully reused for upto four cycles quite effectively. The method's simplicity and efficiency make it a promising route for the synthesis of phenolic Schiff bases, demonstrating the versatility and applicability of graphite in catalytic processes.

Compound 3a

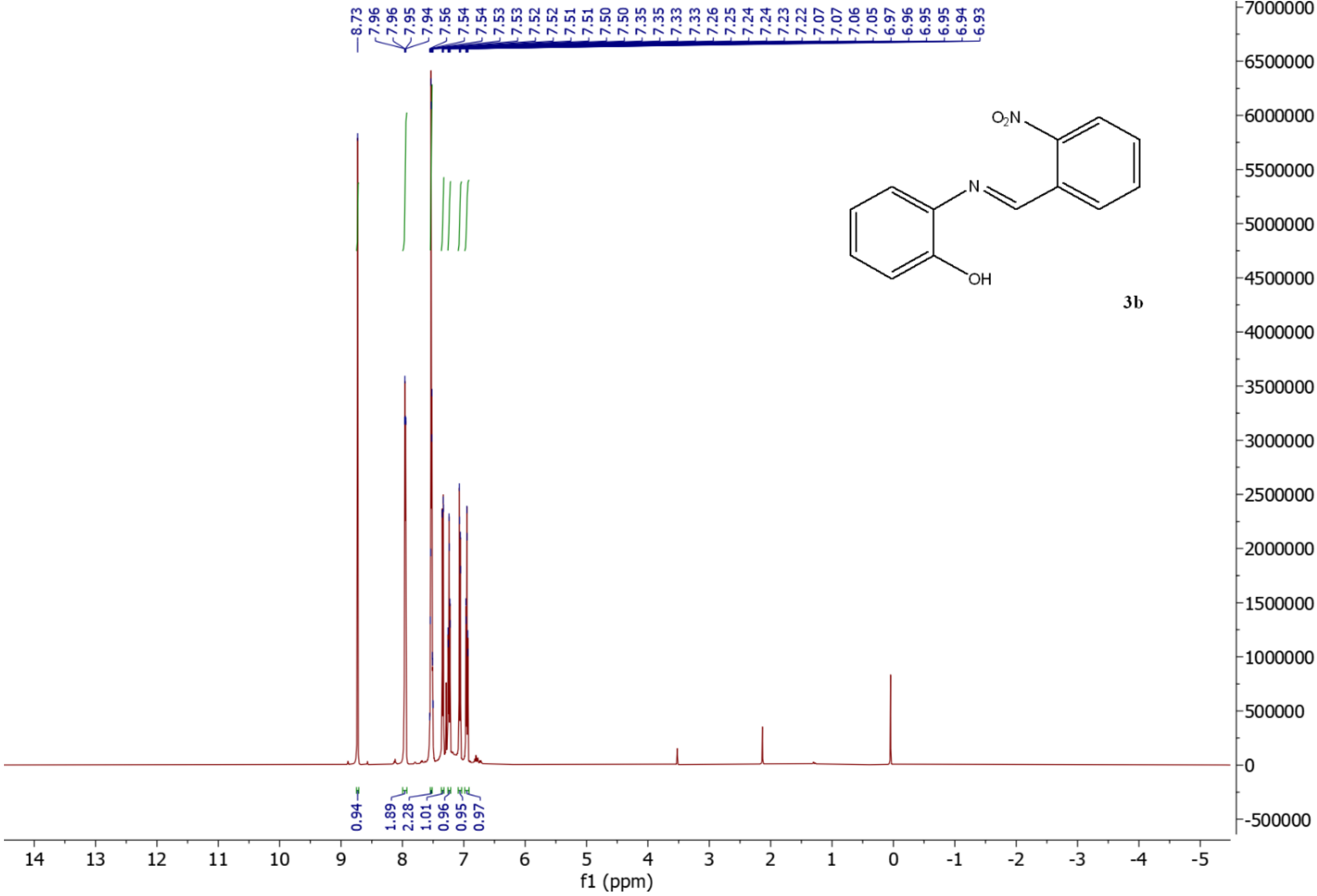
1HNMR



^{13}CNMR



Compound 3b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6.97
6.96
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6.95
6.94
6.93
O2N
N
OH
3b
0.94
1.89
2.28
1.01
0.96
0.95
0.97
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)
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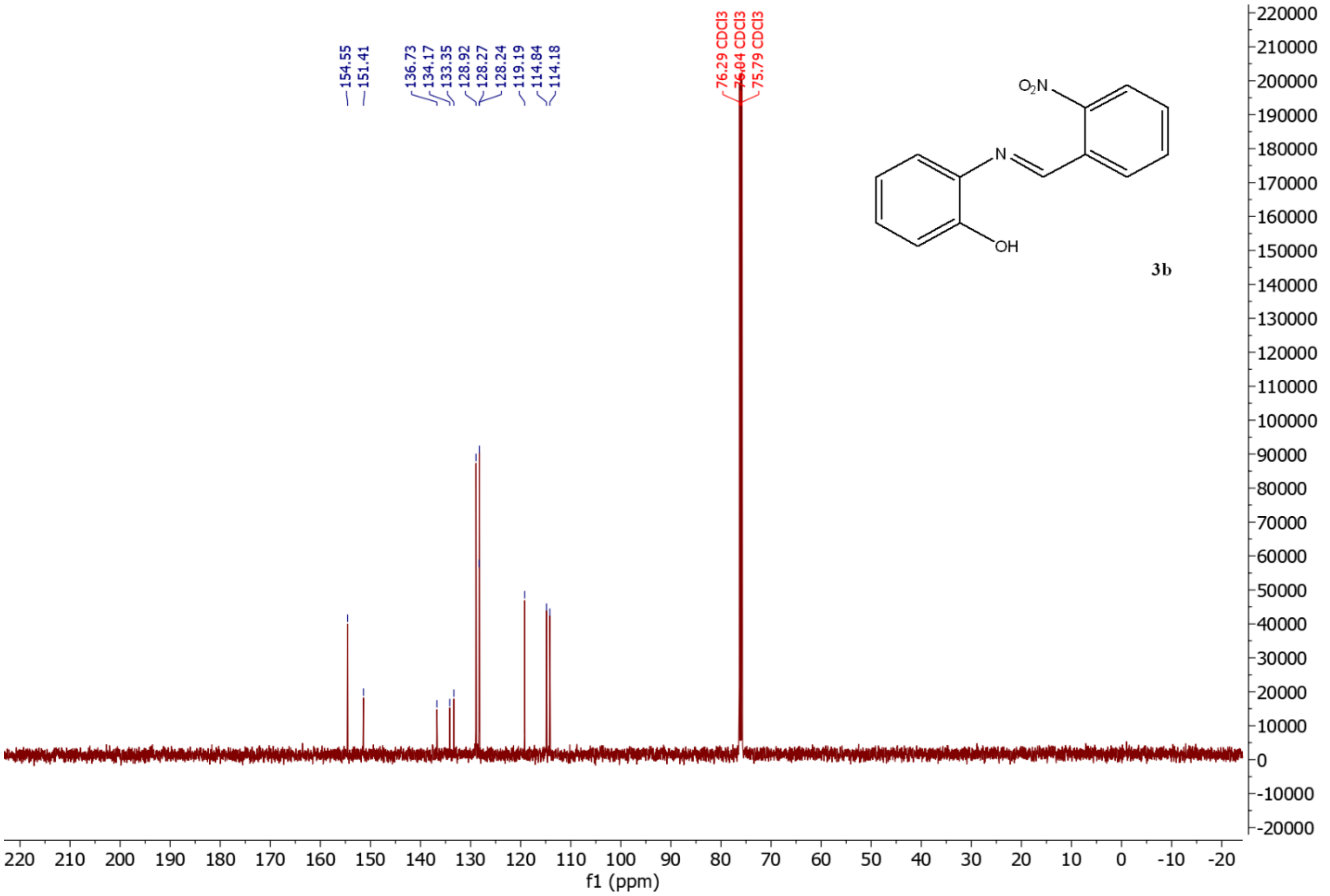
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75.79 CDCl3
O2N
N
OH
3b
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)
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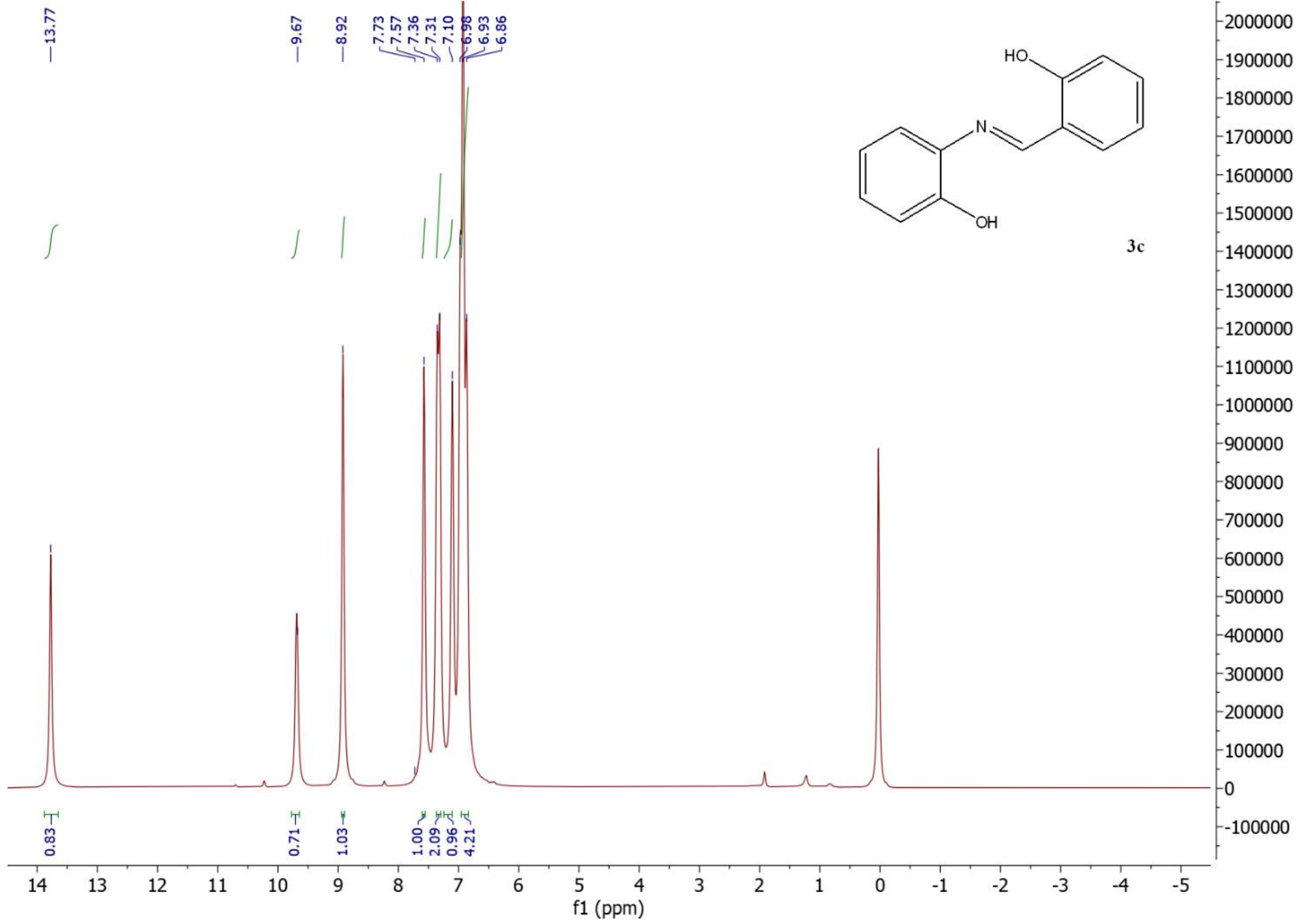
Compound 3c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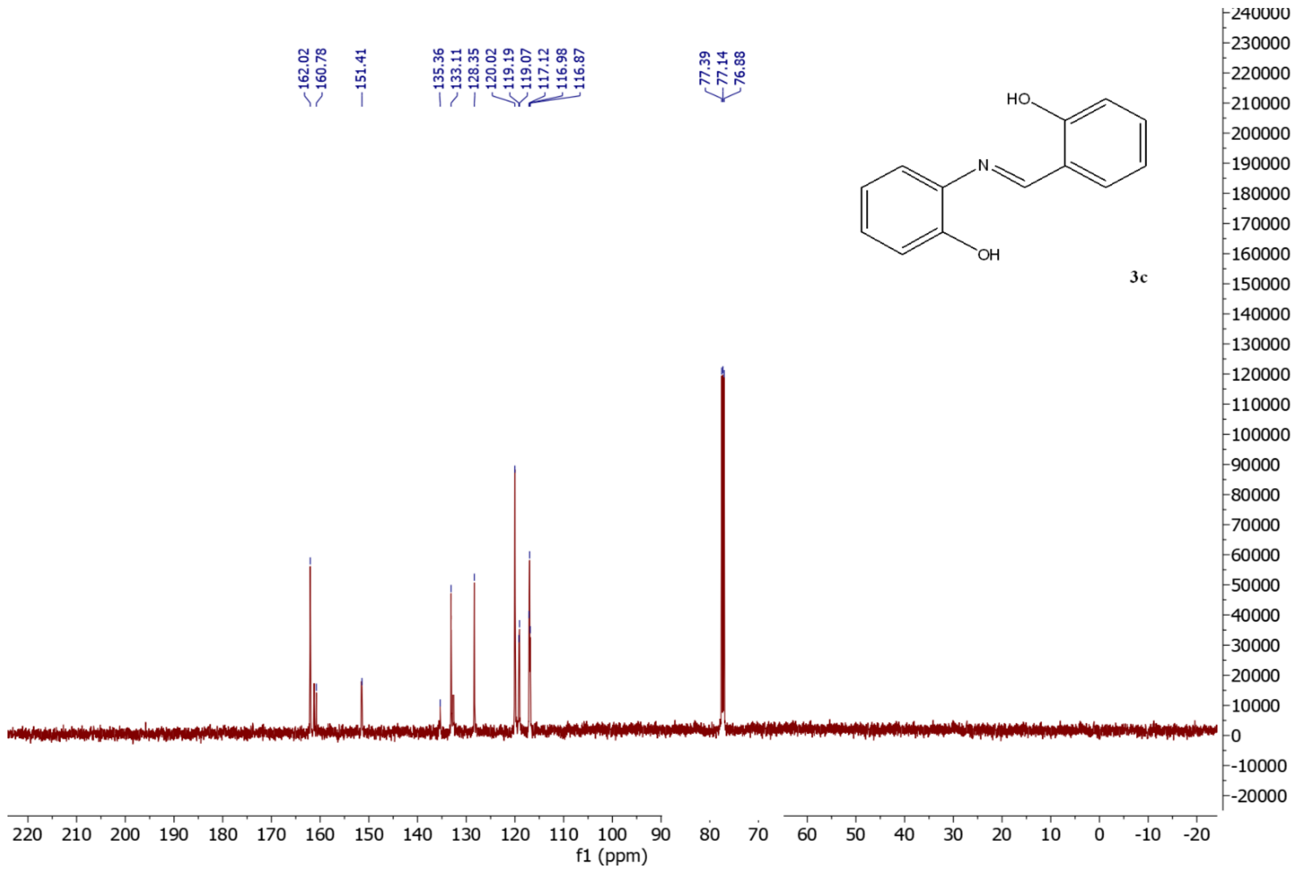
f1 (ppm)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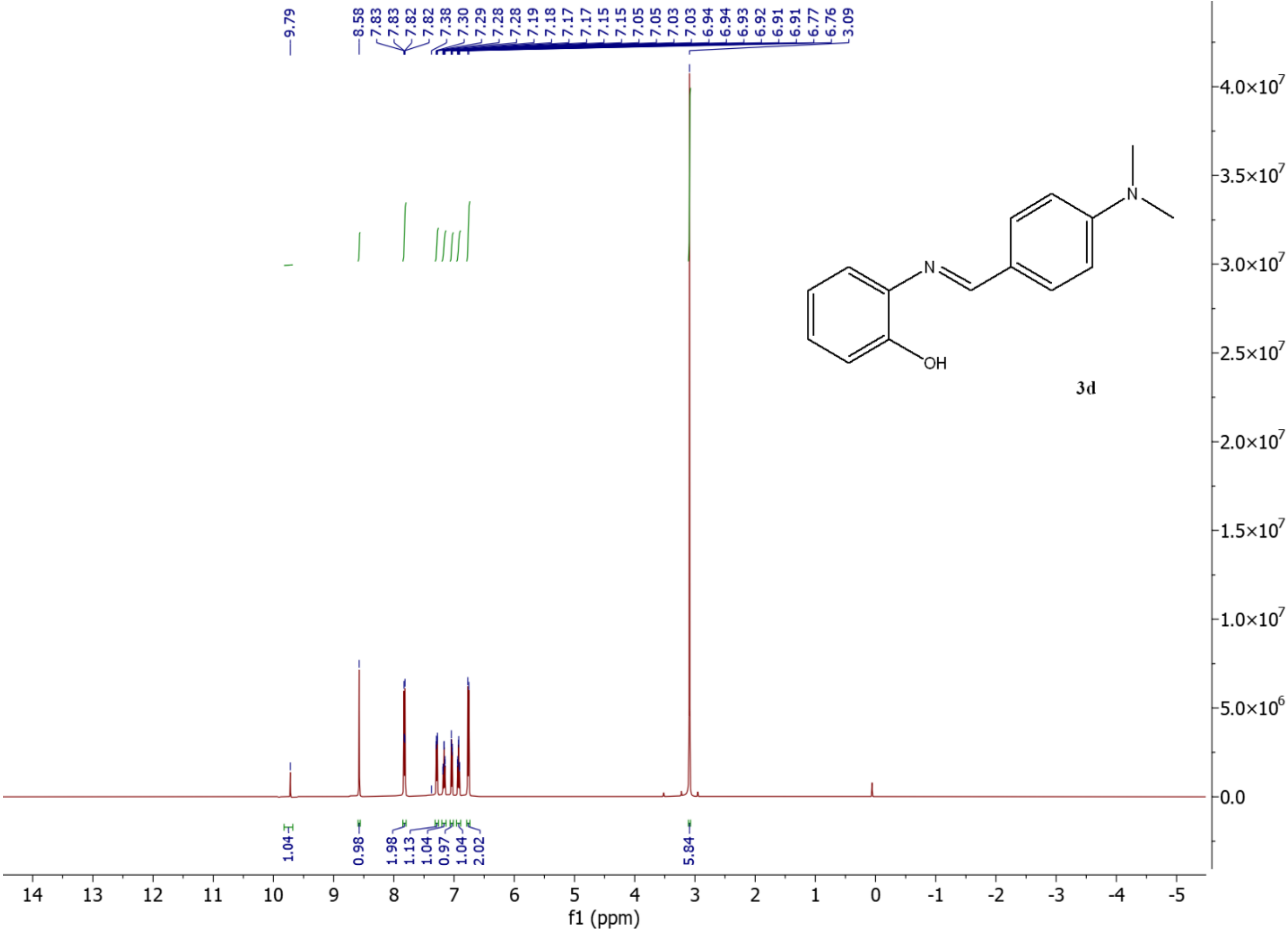
HO
N
OH
3c
f1 (ppm)

Compound 3d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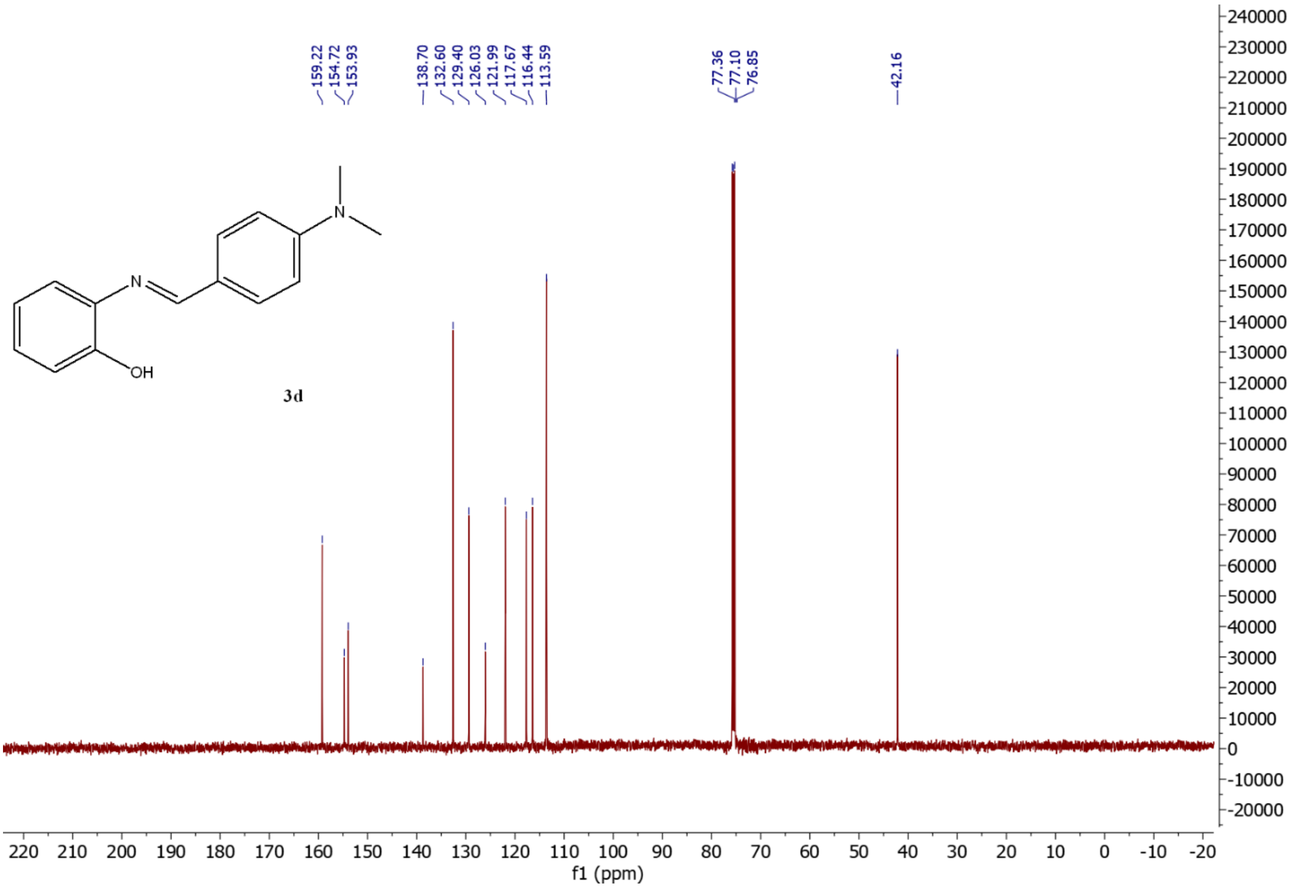
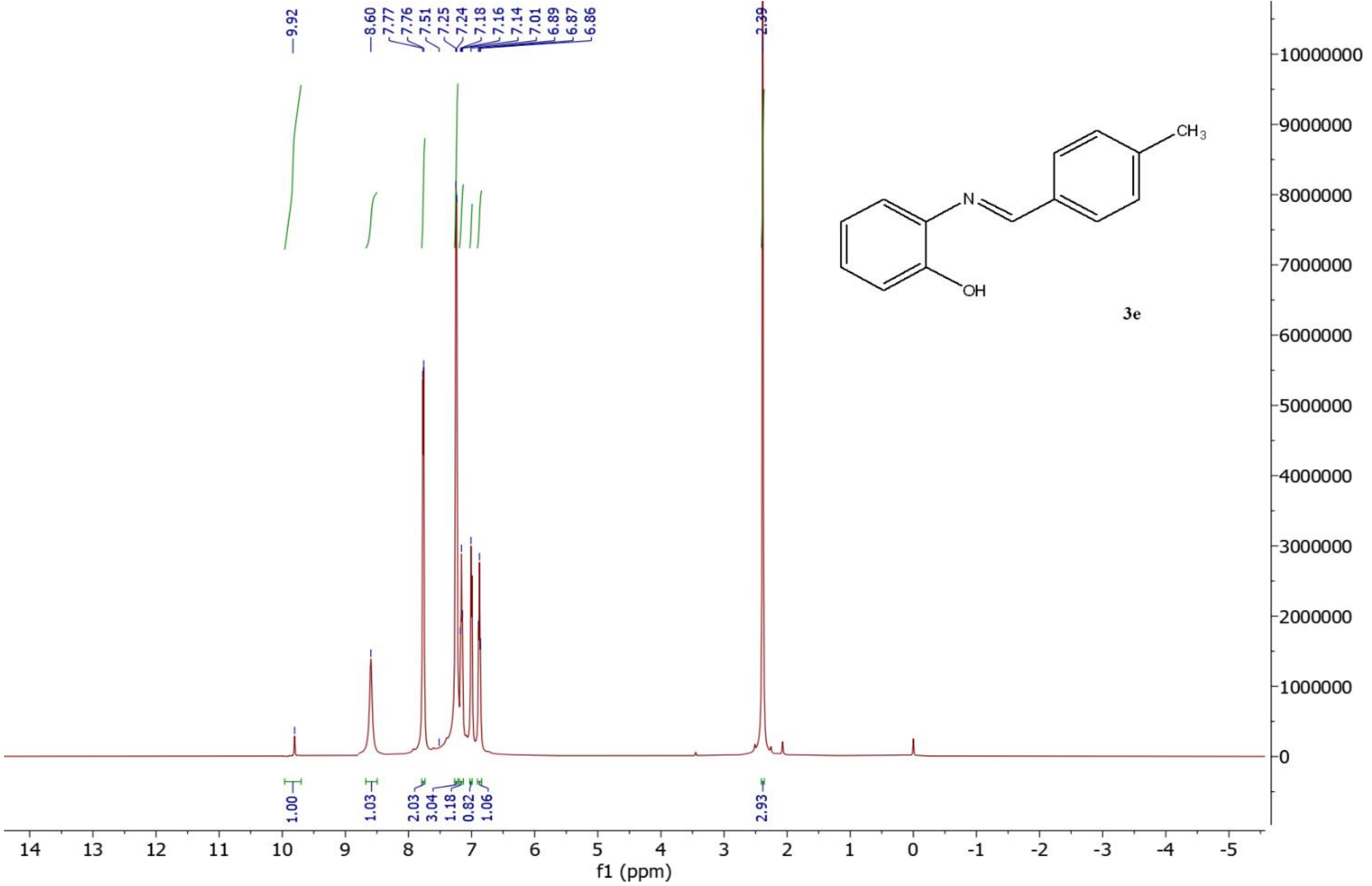
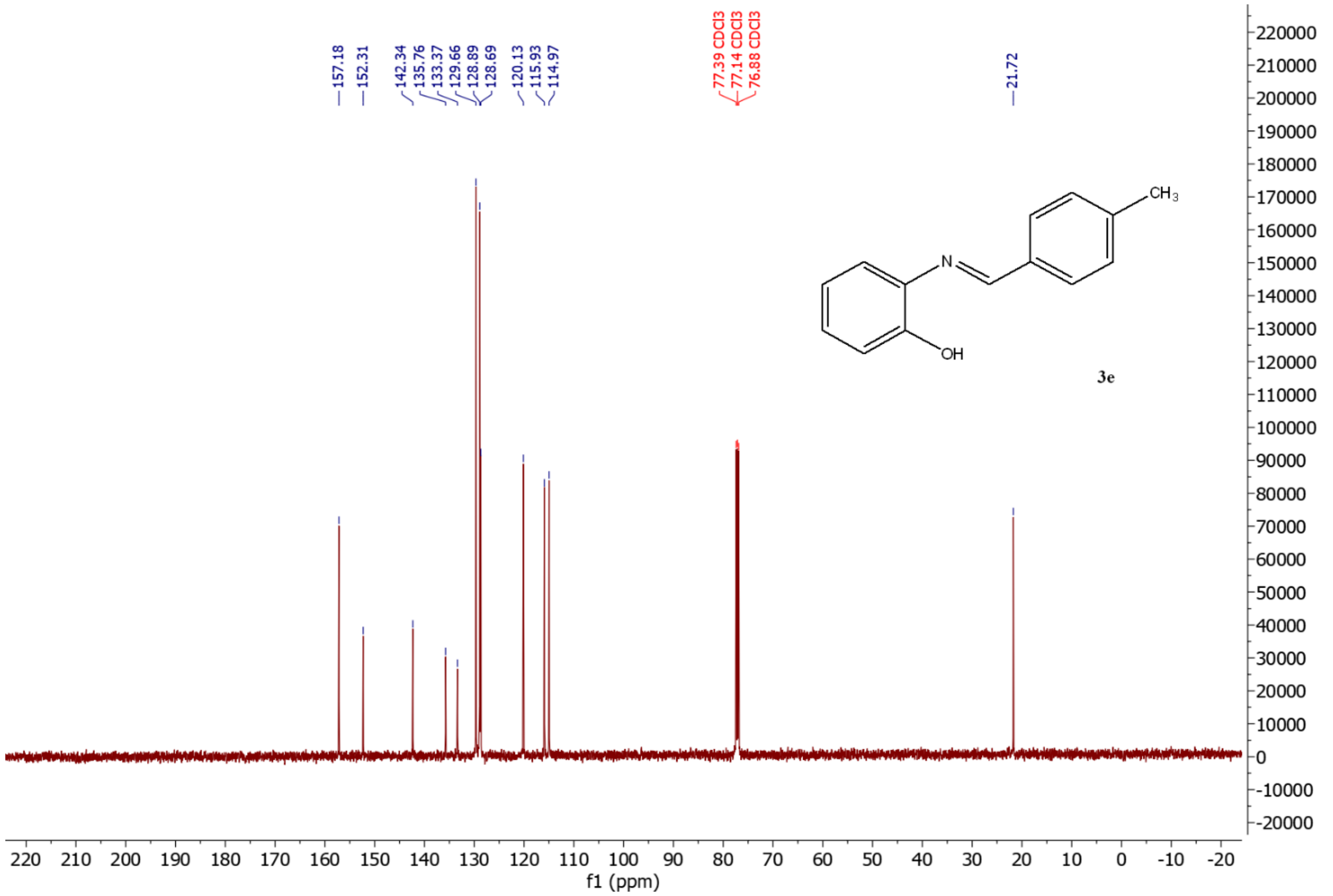
6.94
6.93
6.92
6.91
6.91
6.77
6.76
3.09
N
N
OH
3d
1.04
0.98
1.98
1.13
1.04
0.97
1.04
2.02
5.84
4.0×10^7
3.5×10^7
3.0×10^7
2.5×10^7
2.0×10^7
1.5×10^7
1.0×10^7
5.0×10^6
0.0
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
-5
f1 (ppm)

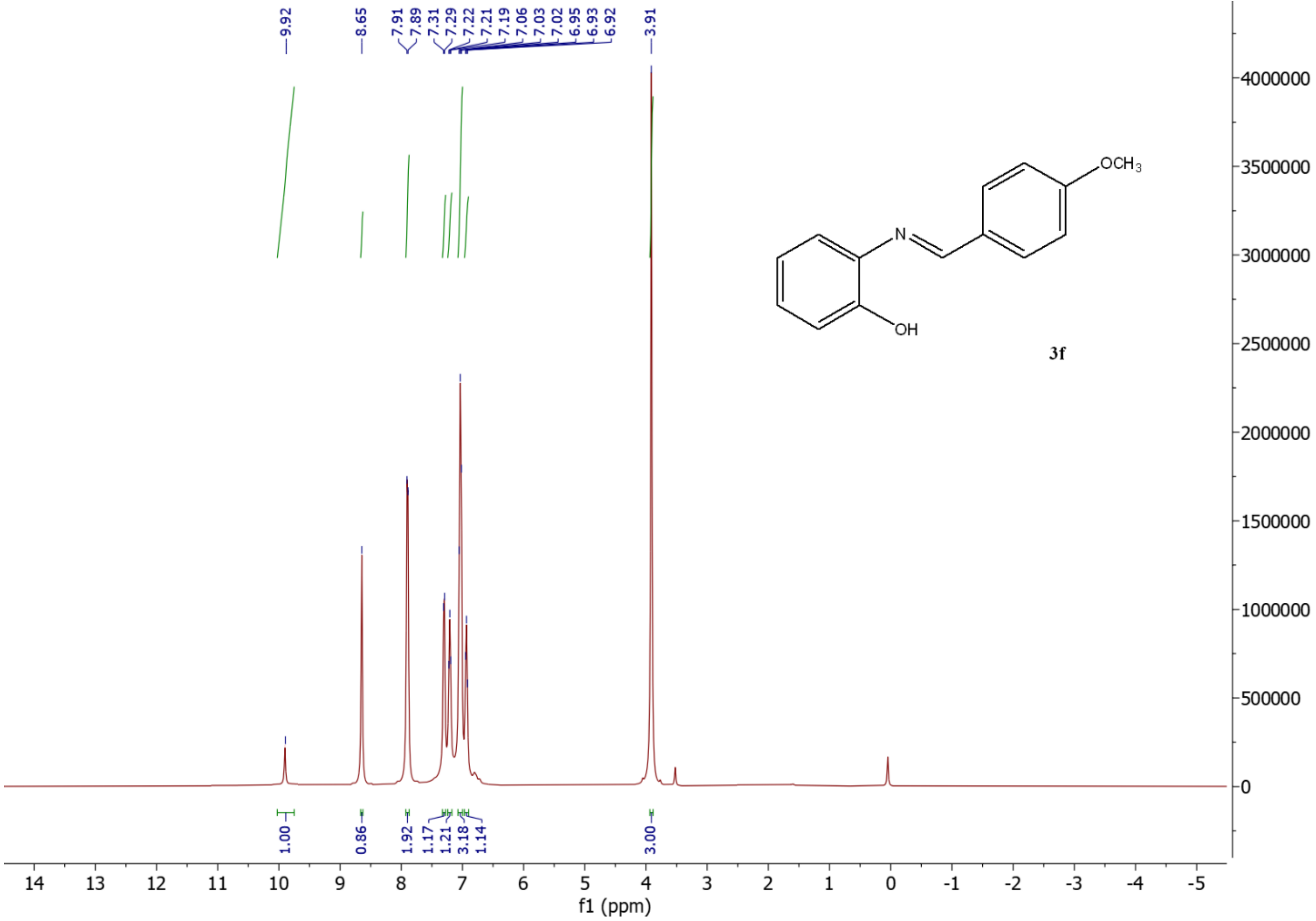

159.22
154.72
153.93
138.70
132.60
129.40
126.03
121.99
117.67
116.44
113.59
77.36
77.10
76.85
42.16
N
N
OH
3d
240000
230000
220000
210000
200000
190000
180000
170000
160000
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

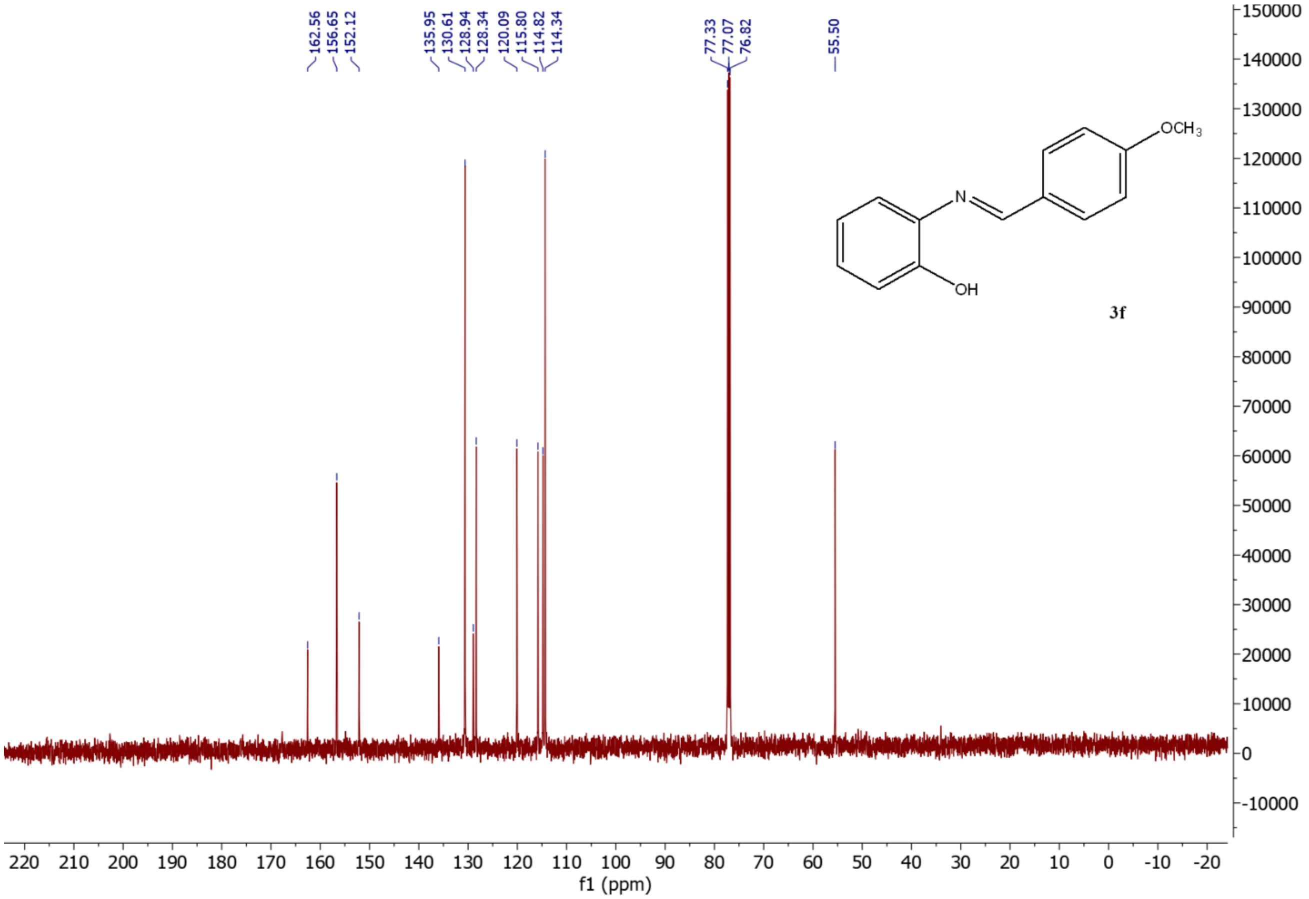
Compound 3e


9.92
8.60
7.77
7.76
7.51
7.25
7.24
7.18
7.16
7.14
7.01
6.89
6.87
6.86
2.39
1.00
1.03
2.03
3.04
1.18
0.82
1.06
2.93
N
OH
CH3
3e
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)

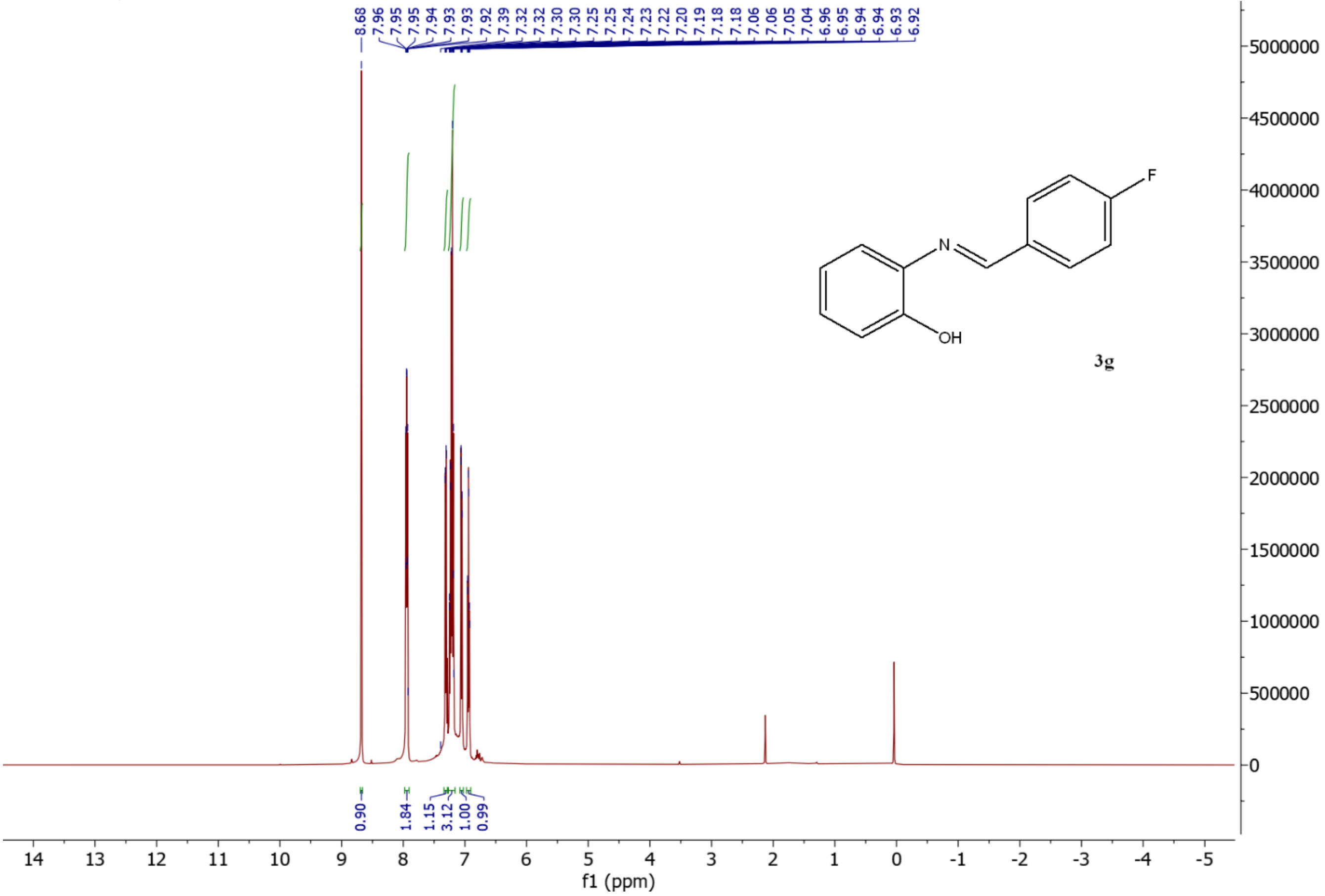

157.18
152.31
142.34
135.76
133.37
129.66
128.89
128.69
120.13
115.93
114.97
77.39 CDCl3
77.14 CDCl3
76.88 CDCl3
21.72
N
OH
CH3
3e
220000
210000
200000
190000
180000
170000
160000
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

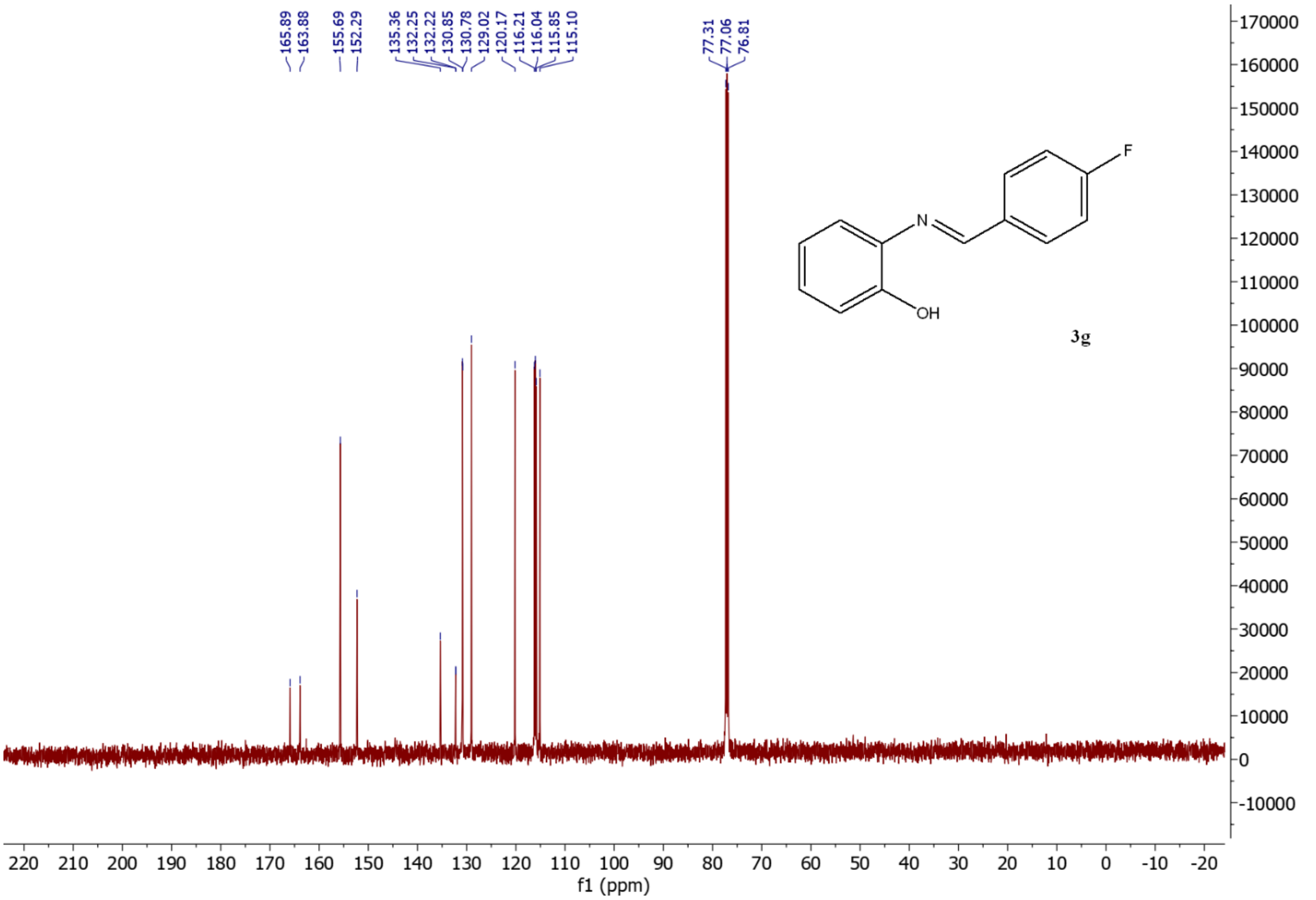
Compound 3f


9.92
8.65
7.91
7.89
7.31
7.29
7.22
7.21
7.19
7.06
7.03
7.02
6.95
6.93
6.92
3.91
OCH3
N
OH
3f
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
1.00
0.86
1.92
1.17
1.21
3.18
1.14
3.00
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
-5
f1 (ppm)

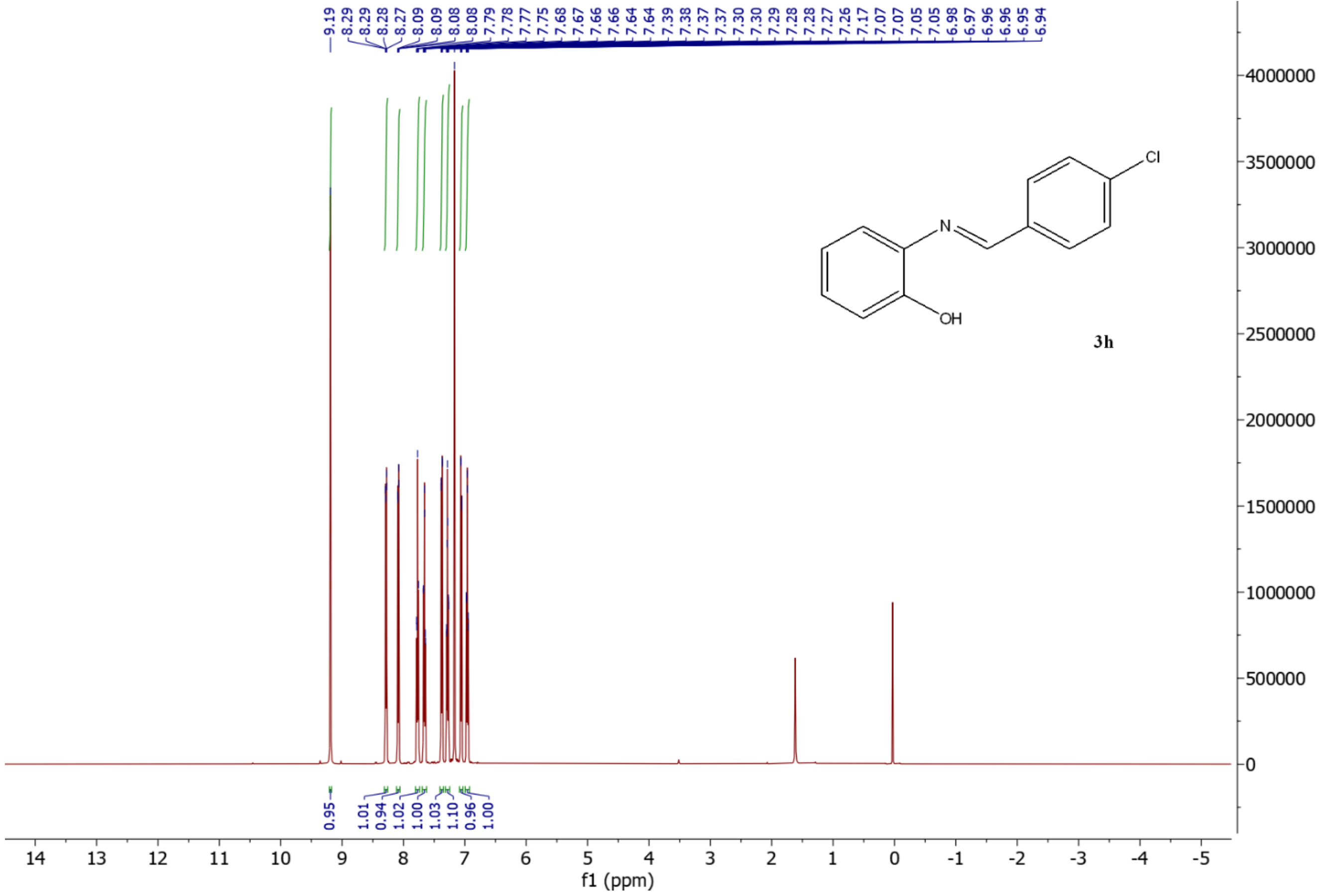

162.56
156.65
152.12
135.95
130.61
128.94
128.34
120.09
115.80
114.82
114.34
77.33
77.07
76.82
55.50
OCH3
N
OH
3f
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

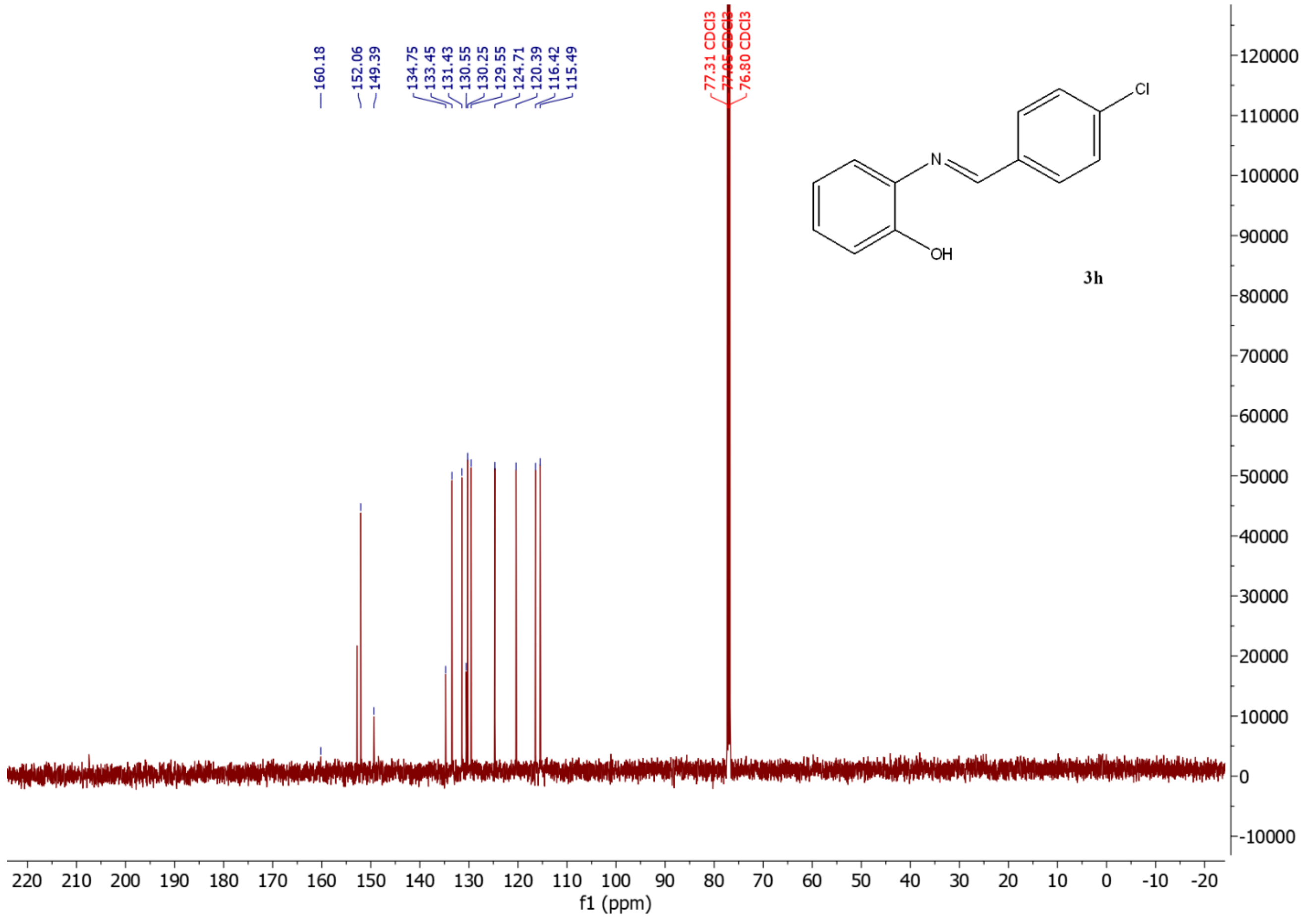
Compound 3g


8.68
7.96
7.95
7.95
7.94
7.93
7.93
7.92
7.39
7.32
7.32
7.30
7.30
7.25
7.25
7.24
7.23
7.22
7.20
7.19
7.18
7.18
7.06
7.06
7.05
7.04
6.96
6.95
6.94
6.94
6.93
6.92
F
N
OH
3g
0.90
1.84
1.15
3.12
1.00
0.99
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)

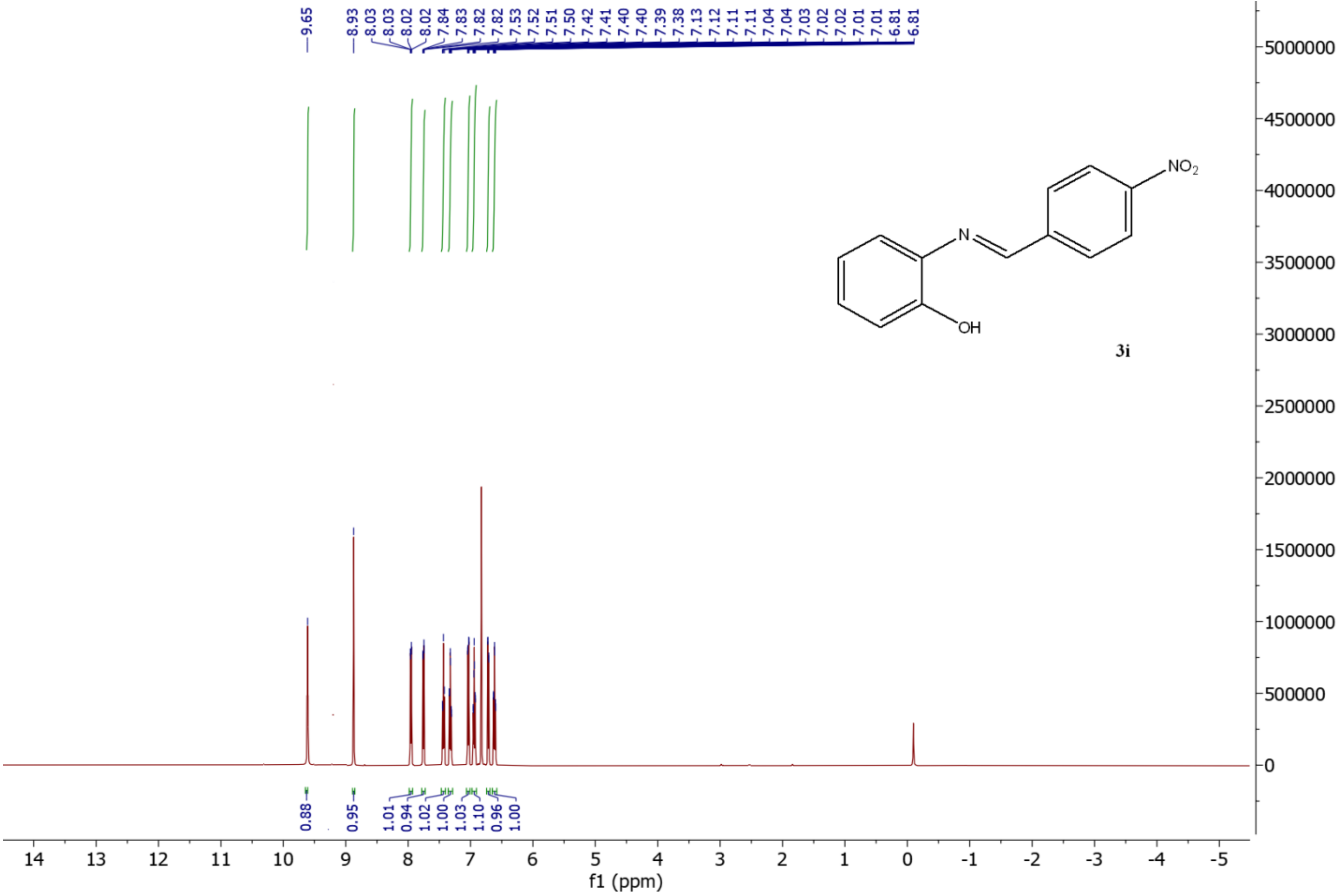

165.89
163.88
155.69
152.29
135.36
132.25
132.22
130.85
130.78
129.02
120.17
116.21
116.04
115.85
115.10
77.31
77.06
76.81
F
N
OH
3g
170000
160000
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

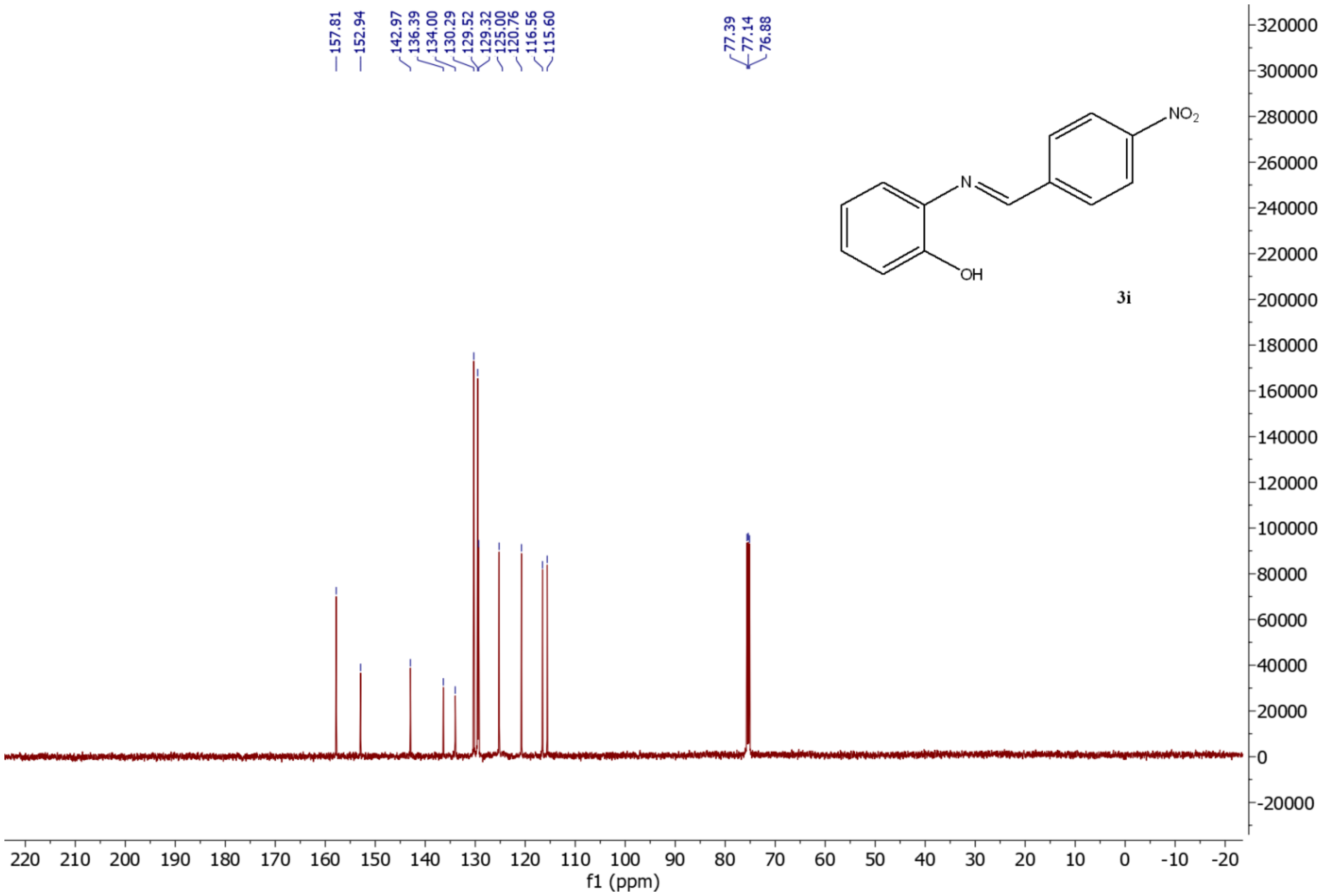
Compound 3h


9.19
8.29
8.29
8.28
8.27
8.09
8.09
8.08
8.08
7.79
7.78
7.77
7.75
7.68
7.67
7.66
7.66
7.64
7.64
7.39
7.38
7.37
7.37
7.30
7.30
7.29
7.28
7.28
7.27
7.26
7.17
7.07
7.07
7.05
7.05
6.98
6.97
6.96
6.96
6.95
6.94
N
Cl
OH
3h
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
0.95
1.01
0.94
1.02
1.00
1.03
1.10
0.96
1.00
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
-5
f1 (ppm)

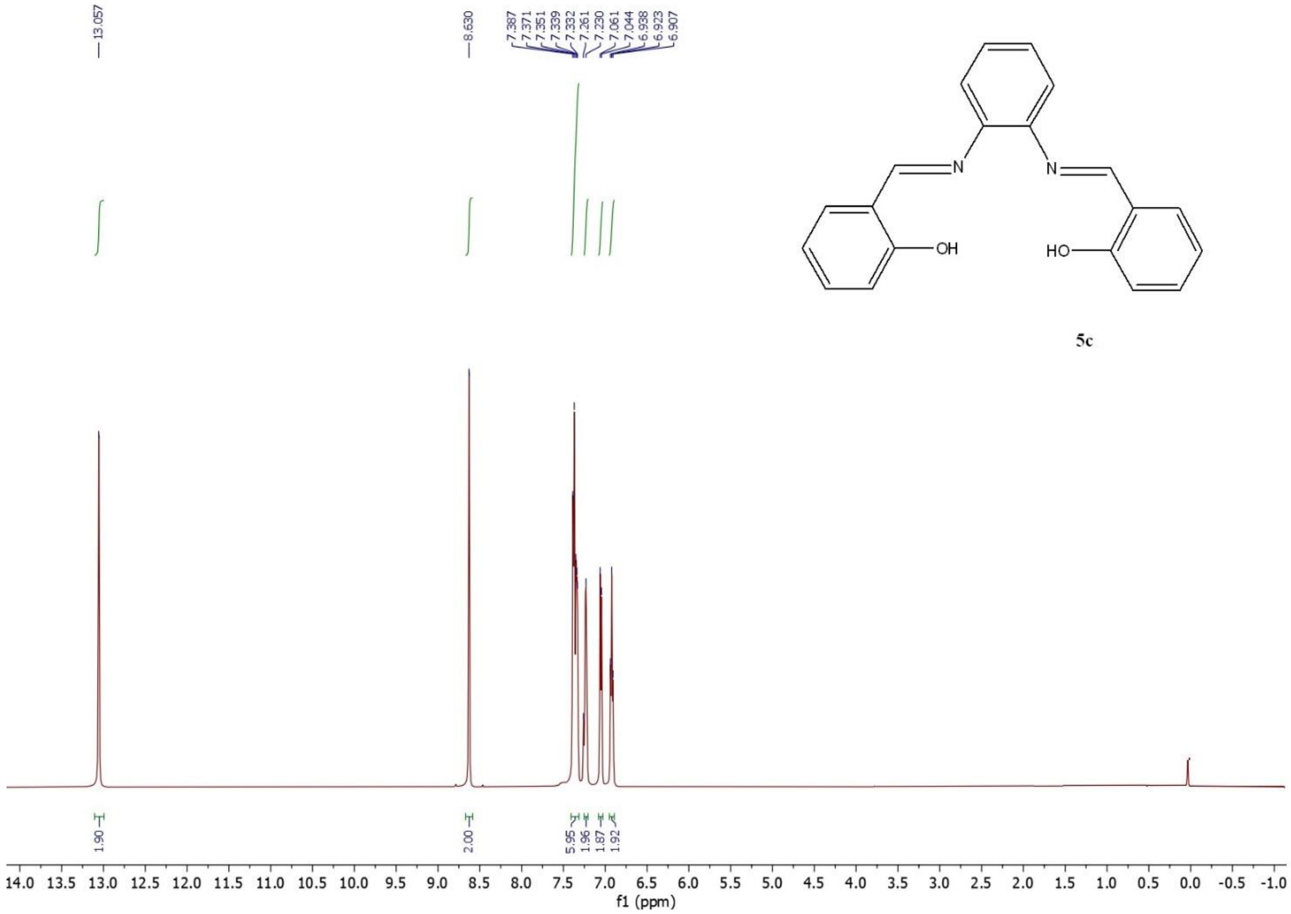

160.18
152.06
149.39
134.75
133.45
131.43
130.55
130.25
129.55
124.71
120.39
116.42
115.49
77.31 CDCl3
77.05 CDCl3
76.80 CDCl3
N
Cl
OH
3h
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

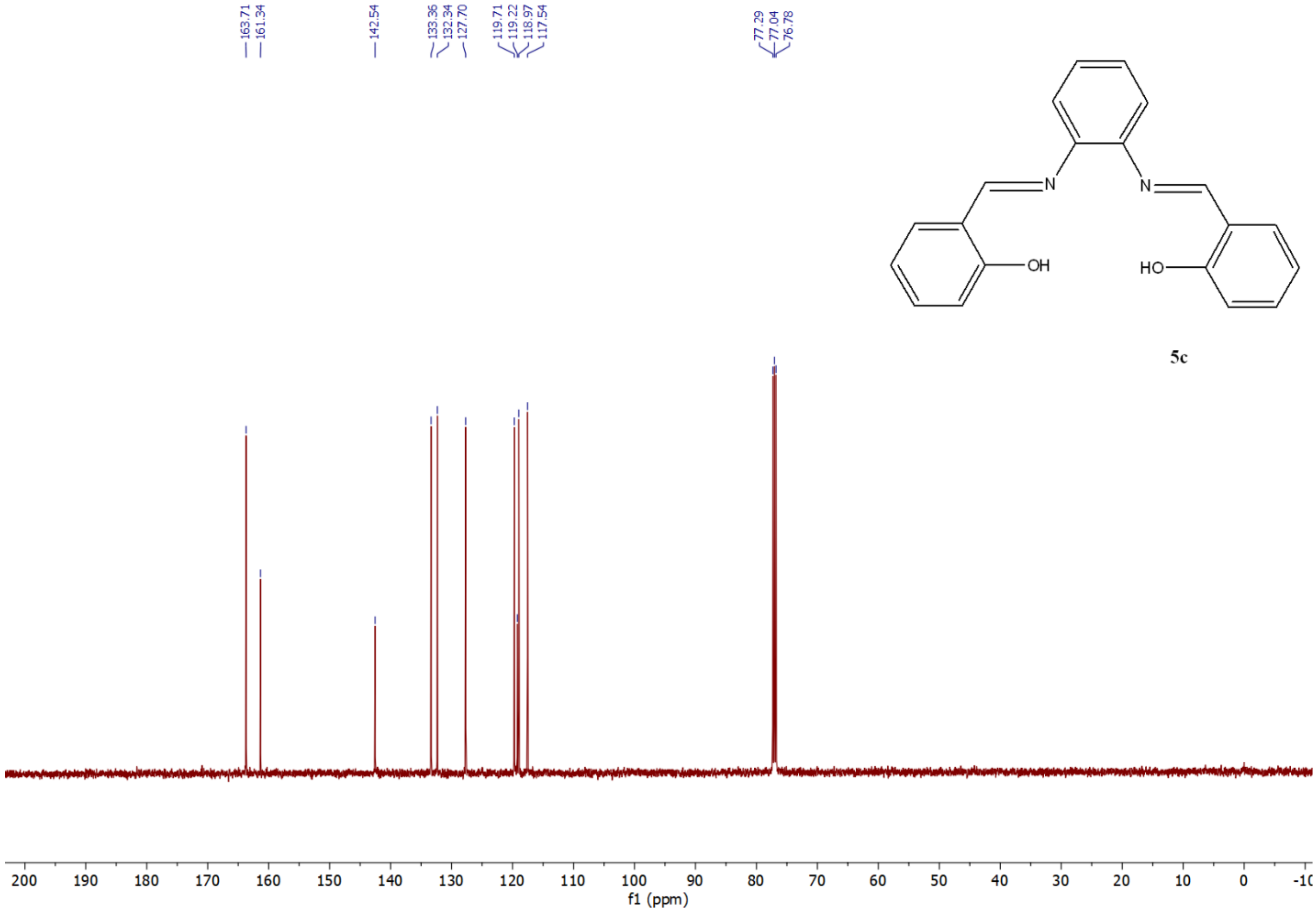
Compound 3i


9.65
8.93
8.03
8.03
8.02
8.02
7.84
7.83
7.82
7.82
7.53
7.52
7.51
7.50
7.42
7.41
7.40
7.40
7.39
7.38
7.13
7.12
7.11
7.11
7.04
7.04
7.03
7.02
7.02
7.01
7.01
6.81
6.81
0.88
0.95
1.01
0.94
1.02
1.00
1.03
1.10
0.96
1.00
NO2
N
OH
3i
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
-5
f1 (ppm)


157.81
152.94
142.97
136.39
134.00
130.29
129.52
129.32
125.00
120.76
116.56
115.60
77.39
77.14
76.88
NO2
N
OH
3i
320000
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

Compound 5c


13.057
8.630
7.387
7.371
7.351
7.339
7.332
7.261
7.230
7.061
7.044
6.938
6.923
6.907
N
N
OH
HO
5c
1.90
2.00
5.95
1.96
1.87
1.92
14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


163.71
161.34
142.54
133.36
132.34
127.70
119.71
119.22
118.97
117.54
77.29
77.04
76.78
N
N
OH
HO
5c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

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(Received 22nd September 2024, accepted 28th October 2024)