Supporting Information

for

Metal and Base Free Syntheses of Primary Amines via ipso Amination of Organoboronic Acids Mediated by [bis-(trifluoroacetoxy)iodo]benzene (PIFA)

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CONTENTS:

1. General information

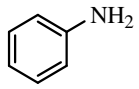
Chemicals and reagents were purchased from commercial suppliers and used without further purification. Anhydrous solvent (CH_3CN) used in the reactions was dried and freshly distilled before use. Thin layer chromatography (TLC) was performed using pre-coated plates purchased from E. Merck (silica gel 60 PF254, 0.25 mm). Column chromatography was performed using E. Merck silica gel 60 (100–200 mesh). GC-MS analyses were carried out on SHIMADZU GCMS-QP Ultra 2010 instrument. NMR spectra were recorded in $CDCl_3$, on JEOL JNM-ECS spectrometer at operating frequencies of 400 MHz (^1H) or 100 MHz (^{13}C) as indicated in the individual spectrum. Chemical shifts (δ) are given in ppm relative to residual solvent (chloroform, δ= 7.26 for ^1H and 77.16 for proton decoupled ^{13}C NMR) and coupling constants (*J*) in Hz. Multiplicity is tabulated as s for singlet, d for doublet, t for triplet, q for quartet, dd for doublet of doublet, dt for doublet of triplet and m for multiplet.

2. Syntheses and Characterizations of the Aromatic and Aliphatic Nitro Compounds

General procedure for syntheses of the amino compounds:

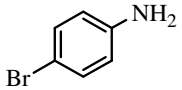
To a stirred solution of appropriate organoboronic acids (2.0 mmol, 1.0 eq.), PIFA (4.0 mmol, 2.0 eq.) and NBS (4.0 mmol, 2.0 eq.) in CH_3CN (6 mL), $MeONH_2.HCl$ (2.0 mmol, 1.0 eq.) were added and the mixture was stirred for 2-3 h. After completion of the reaction (checked by TLC), the mixture was concentrated under vacuum. The solid mass obtained, was washed with dry hexane to remove the iodobenzene and other less polar impurities. The solid was then dissolved to its optimum extent in minimum volume of water. The solution was made completely alkaline with saturated aq. NaOH solution under cold condition and the aq. solution was extracted with ethyl acetate (5×20 mL). The combined organic phase was washed with distilled water (3×7 mL) and was dried over Na_2SO_4. After evaporating the solvent, the residue was purified by column chromatography over silica gel using pentane/ether as eluent to provide the pure target product.

Characterization of compounds:



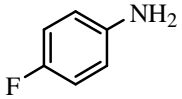
2a

Aniline (2a): 2a was obtained as a pale yellow liquid (78%, 145.1 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19-7.23 (m, 2H), 6.80-6.84 (m, 1H), 6.71-6.73 (m, 2H), 3.58 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.3, 129.3, 118.3, 115.2.



2b

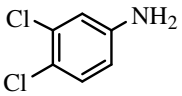
4-Bromoaniline (2b): 2b was obtained as a off-white solid (87%, 299.2 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (dd, J = 6.8 Hz, 1.8 Hz, 2H), 6.55 (dd, J = 6.4Hz, 2.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.3, 132.1, 116.8, 110.1.



2c

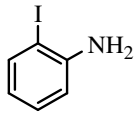
4-Fluoroaniline (2c): 2c was obtained as a light brown liquid (71%, 157.6 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.83-6.87 (m, 2H), 6.59-6.63 (m, 2H),

3.45 (s, br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.6, 155.3, 142.4, 116.2, 116.1, 115.8, 115.6.



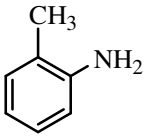
2d

3,4-Dichloroaniline (2d): 2d was obtained as a grey solid (84%, 272.2 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.16 (d, *J* = 8.7 Hz, 1H), 6.75 (d, *J* = 2.8 Hz, 1H), 6.48-6.51 (m, 1H), 3.71 (s, br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 132.7, 130.6, 121.2, 116.9, 114.9.



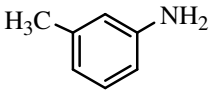
2e

2-iodoaniline (2e): 2e was obtained as a brownish solid (80%, 350.4 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (dd, *J* = 8.2 Hz, 1.4Hz, 1H), 7.13 (dt, *J* = 7.3 Hz, 1.8 Hz, 1H), 6.75 (dd, *J* = 8.2 Hz, 1.8 Hz, 1H), 6.47 (dt, *J* = 8.2 Hz, 1.8 Hz, 1H) 4.08 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.8, 139.2, 129.4, 119.6, 114.5, 84.2.



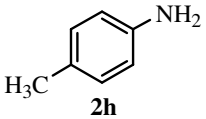
2f

2-Methylaniline (2f): 2f was obtained as a pale yellow liquid (78%, 166.9 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.07-7.10 (m, 2H), 6.76 (t, *J* = 7.4 Hz, 1H), 6.71 (d, *J* = 7.8 Hz, 1H), 3.61 (s, 3H), 3.96 (s, br, 1H), 2.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.9, 131.2, 127.0, 121.8, 118.6, 114.6, 17.4.



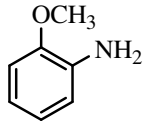
2g

3-Methylaniline (2g): 2g was obtained as a light yellow liquid (74%, 158.4 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.08 (t, *J* = 7.8 Hz, 1H), 6.62 (d, *J* = 7.8 Hz, 1H), 6.52-6.54 (m, 2H), 3.54 (s, 2H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.2, 139.1, 129.0, 119.5, 116.0, 112.3, 21.8.



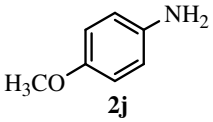
2h

4-Methylaniline (2h): 2h was obtained as a off-white semi-solid (82%, 175.5 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.97-6.99 (m, 2H), 6.61-6.64 (m, 2H), 3.39 (s, br, 2H), 2.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.9, 129.8, 127.9, 115.3, 20.5.



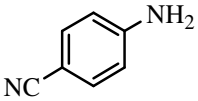
2i

2-Methoxyaniline (2i): 2i was obtained as a light yellow liquid (79%, 194.3 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.73-6.83 (m, 4H), 3.86 (s, 3H), 3.69 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.3, 136.1, 121.2, 118.2, 115.3, 110.4, 55.4.



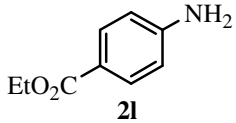
2j

4-Methoxyaniline (2j): 2j was obtained as a pale off-white solid (86%, 211.6 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.75 (dd, J = 8.6 Hz, 2.3 Hz, 2H), 6.65 (dd, J = 8.7 Hz, 2.3 Hz, 2H), 3.74 (s, 3H), 3.19 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.9, 139.8, 116.5, 114.7, 55.7.



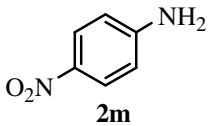
2k

4-cyanoaniline (2k): 2k was obtained as a pale yellowish solid (75%, 177.0 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 (dd, J = 8.7 Hz, 1.8 Hz, 2H), 6.64 (dd, J = 8.6 Hz, 2.3 Hz, 2H), 4.17 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.5, 133.9, 120.1, 114.5, 100.1.



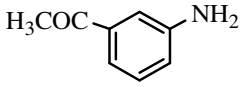
2l

Ethyl 4-aminobenzoate (2l): 2l was obtained as a off-white solid (77%, 232.6 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (dd, *J* = 8.7 Hz, 2.3 Hz, 2H), 6.63 (dd, *J* = 8.7 Hz, 2.3 Hz, 2H), 4.31 (q, 2H), 4.05 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.6, 150.7, 131.6, 120.1, 113.9, 60.1, 14.8.



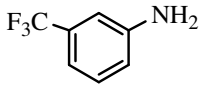
2m

4-nitroaniline (2m): 2m was obtained as a brown solid (73%, 201.5 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (d, *J* = 8.7 Hz, 2H), 6.62 (d, *J* = 9.2 Hz, 2H), 4.39 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.4, 126.3, 122.0, 113.4.



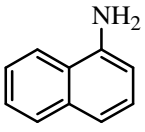
2n

3-Aminoacetophenone (2n): 2n was obtained as a brownish solid (78%, 210.6 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24-7.26 (m, 1H), 7.14-7.19 (m, 2H), 6.78-6.81 (m, 1H), 3.66 (s, br, 2H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.9, 146.8, 138.1, 129.5, 119.9, 119.0, 113.8, 26.8.



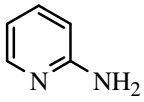
2o

3-Trifluoromethylaniline (2o): 2o was obtained as a light yellow liquid (66%, 212.5 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): 7.15 (t, *J* = 7.8 Hz, 1H), 6.90 (d, *J* = 7.8 Hz, 1H), 6.79 (m, 1H), 6.70-6.73 (m, 1H), 3.66 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.7, 131.7, 131.5, 131.1, 129,8, 128.3, 125.6, 122.9, 118.0, 115.1, 115.0, 111.4, 111.3.



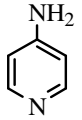
2p

1-Aminonapthaline (2p): 2p was obtained as a grey solid (82%, 234.5 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): 7.81-7.84 (m, 2H), 7.46-7.48 (m, 2H), 7.29-7.35 (m, 2H), 6.80 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.1, 134.4, 128.6, 126.4, 125.9, 124.9, 123.7, 120.8, 119.0, 109.7.



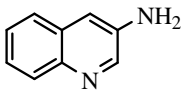
4a

2-Aminopyridine (4a): 4a was obtained as a light yellow solid (83%, 234.1 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): 8.03-8.05 (m, 1H), 7.37-7.46 (m, 1H), 6.59-6.62 (m, 1H), 6.46 (d, J = 8.2 Hz, 1H), 4.52 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.2, 148.1, 137.8, 114.0, 108.2.



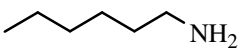
4b

4-Aminopyridine (4b): 4b was obtained as a white solid (78%, 219.9 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20 (dd, J = 6.4 Hz, 1.4 Hz, 2H), 6.51 (dd, J = 6.4 Hz, 1.4 Hz, 2H), 4.14 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.7, 150.4, 109.7.



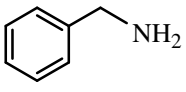
4c

3-Aminoquinoline (4c): 4c was obtained as a brownish solid (74%, 319.7mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (s, 1H), 7.94-7.96 (m, 1H), 7.55-7.58 (m, 1H), 7.39-7.44 (m, 2H), 7.21 (m, 1H), 3.65 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.1, 142.6, 139.9, 129.2, 128.9, 127.0, 125.9, 125.7, 115.1.



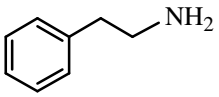
6a

Hexylamine (6a): 6a was obtained as a light yellow liquid (65%, 196.9 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.64 (t, J = 7.3 Hz, 2H), 1.36-1.41 (m, 2H), 1.21-1.31 (m, 6H), 1.16 (s, br, 2H), 0.85 (t, J = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 42.3, 39.9, 31.6, 26.4, 22.7, 14.0.



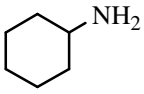
6b

Benzylamine (6b): 6b was obtained as a light yellow liquid (76%, 244.0 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.14-7.27 (m, 5H), 3.77 (s, 2H), 1.43 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.2, 128.5, 127.1, 126.8, 46.5.



6c

2-Phenylethylamine (6c): 6c was obtained as a light brown liquid (78%, 283.1 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28-7.32 (m, 2H), 7.19-7.23 (m, 3H), 2.96 (t, J = 6.9 Hz, 2H), 2.74 (t, J = 6.9 Hz, 2H), 1.24 (s, br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.8, 128.9, 128.4, 126.0, 43.8, 40.0.



6d

Cyclohexylamine (6d): 6d was obtained as a colourless liquid (70%, 207.9 mg) following the general procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.53-2.60 (m, 1H), 1.74-1.78 (m, 2H), 1.63-1.68 (m, 2H), 1.52-1.57 (m, 1H), 1.34 (s, br, 2H), 0.94-1.26 (m,6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 50.5, 36.9, 25.7, 25.2.

References:

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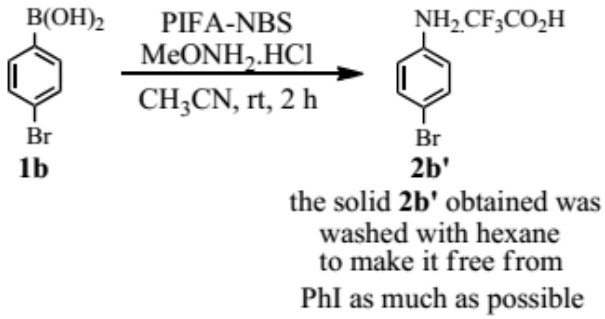
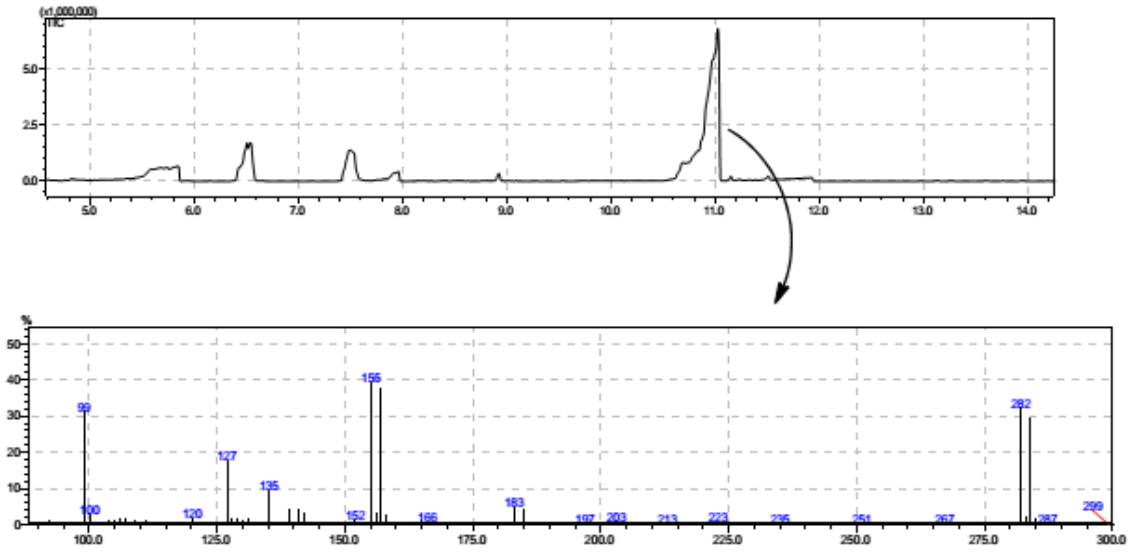
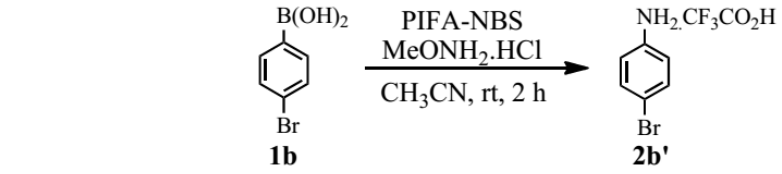
GC-MS of crude *p*-bromoanilinetrifluoroacetate salt (2b')

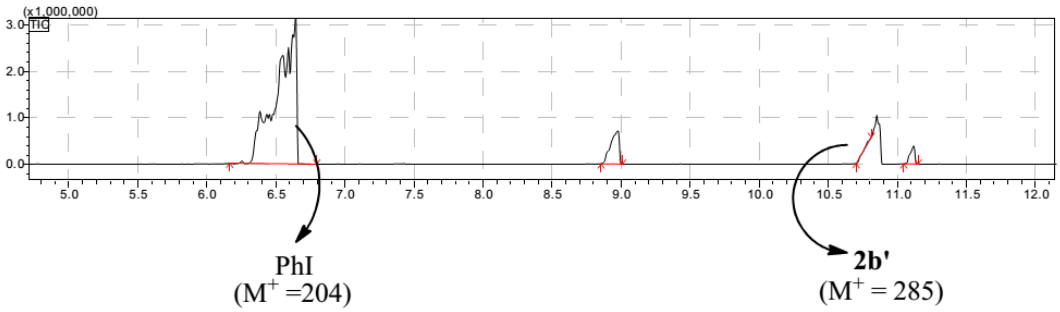


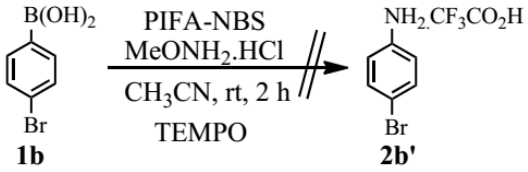


(GC-MS of crude solid **2b'** after washing it with hexane)

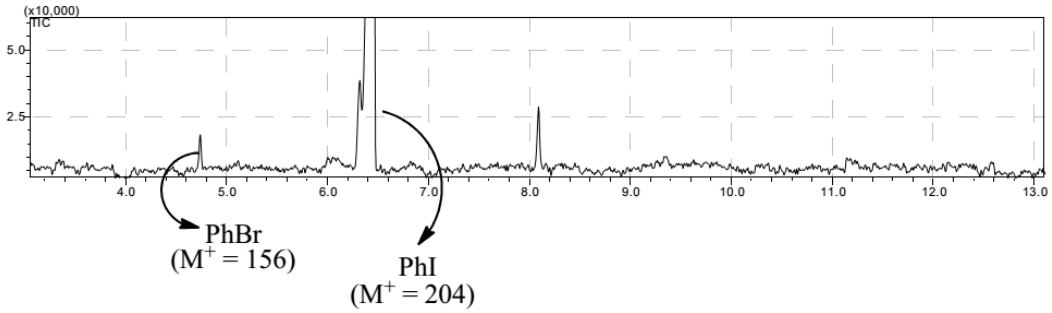
Radical scavenging experiment with TEMPO





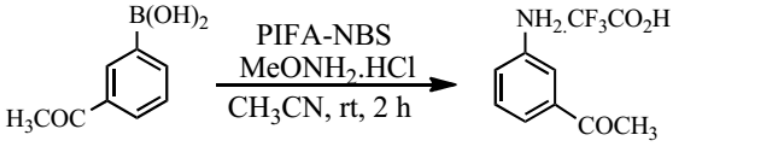


2b' was not formed
protodeboronation was observed

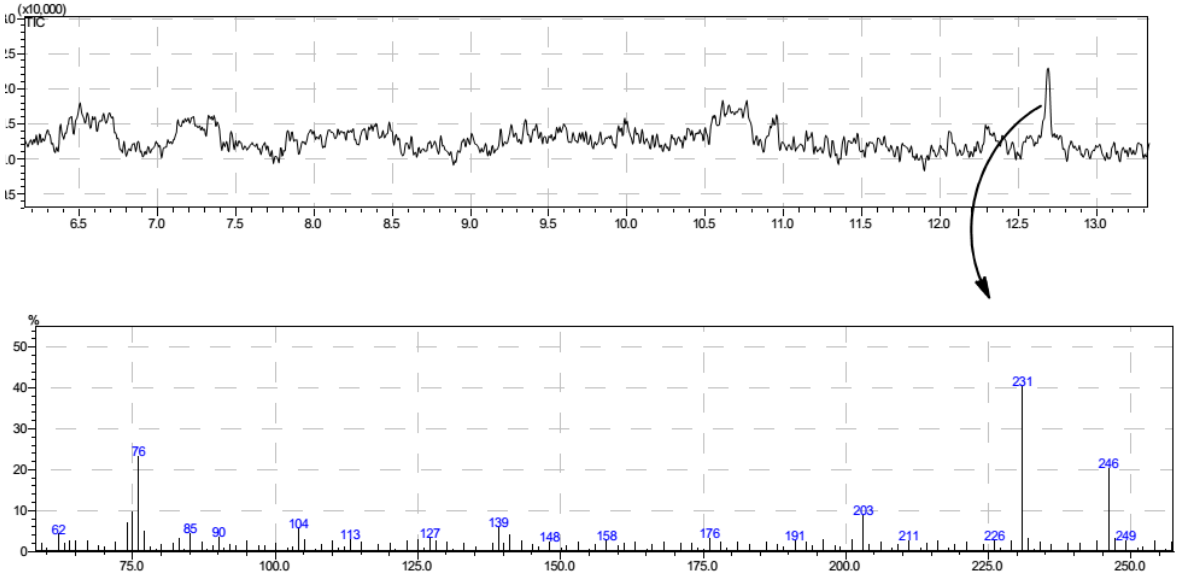


Two different reactions were performed at the same time under similar standard reaction conditions except that one of the reactions were carried out in presence of TEMPO as a radical scavanger. No aminated product was observed in the presence of TEMPO after 3 h, whereas in the other reaction, amination was found to take place as usual within 3 h.

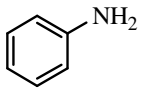
GC-MS of crude *m*-aminoacetophenonetrifluoroacetate salt (2n')



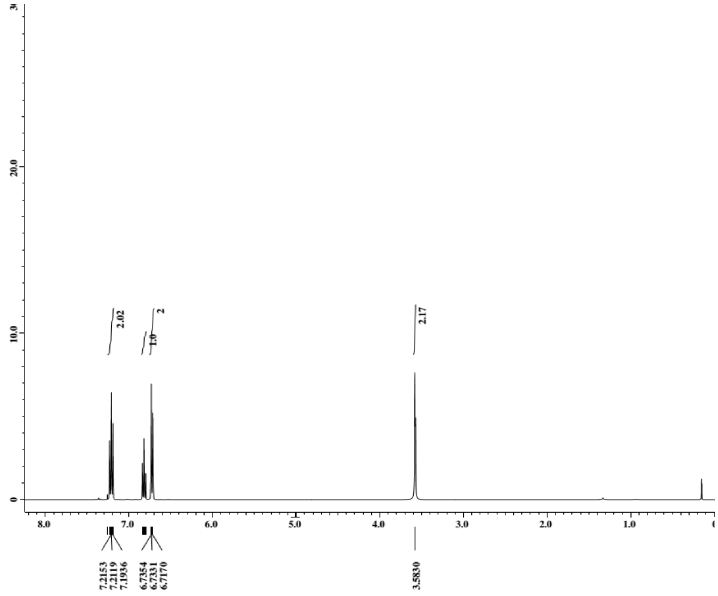
the solid product obtained was as usual washed with hexane to make it free from PhI as much as possible

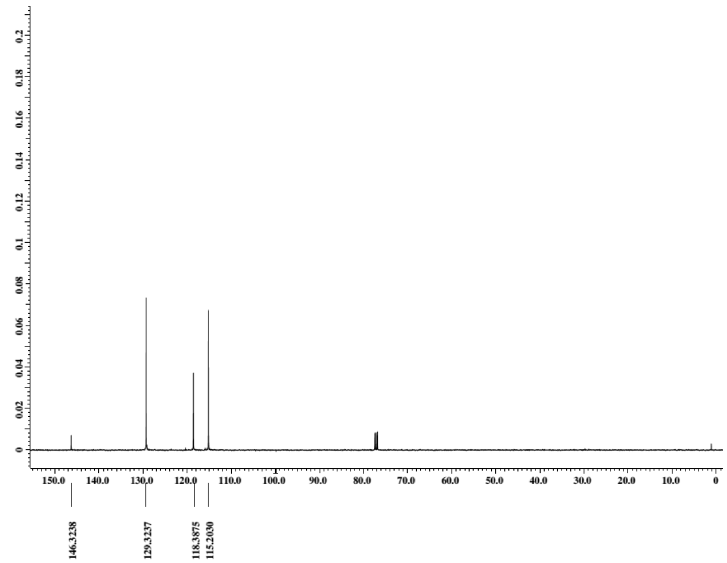


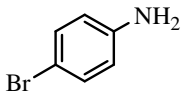
(GC-MS of crude solid **2n'** after washing it with hexane)



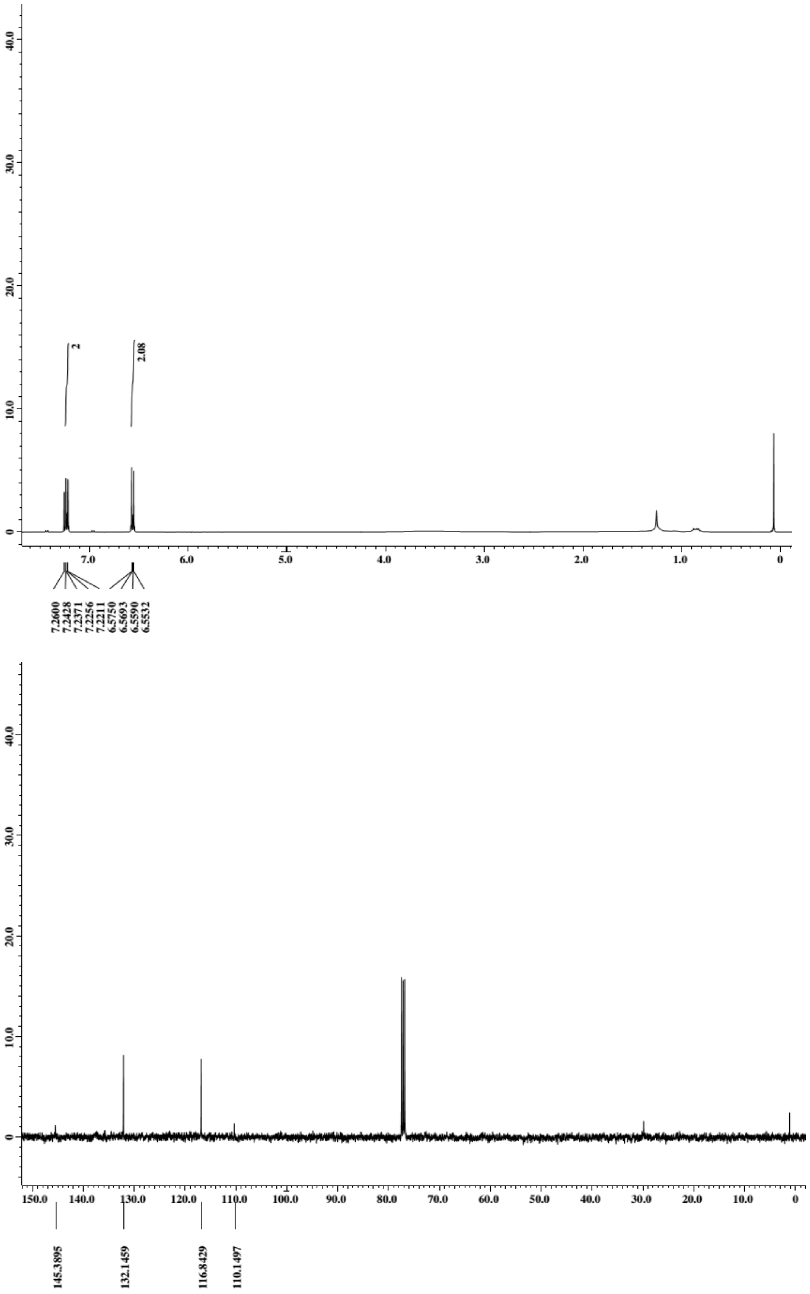
2a

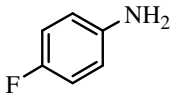

2.02
2
1.0
2.17
7.2153
7.2119
7.1936
6.7354
6.7331
6.7170
3.5830


146.3238
129.3237
118.3875
115.2030

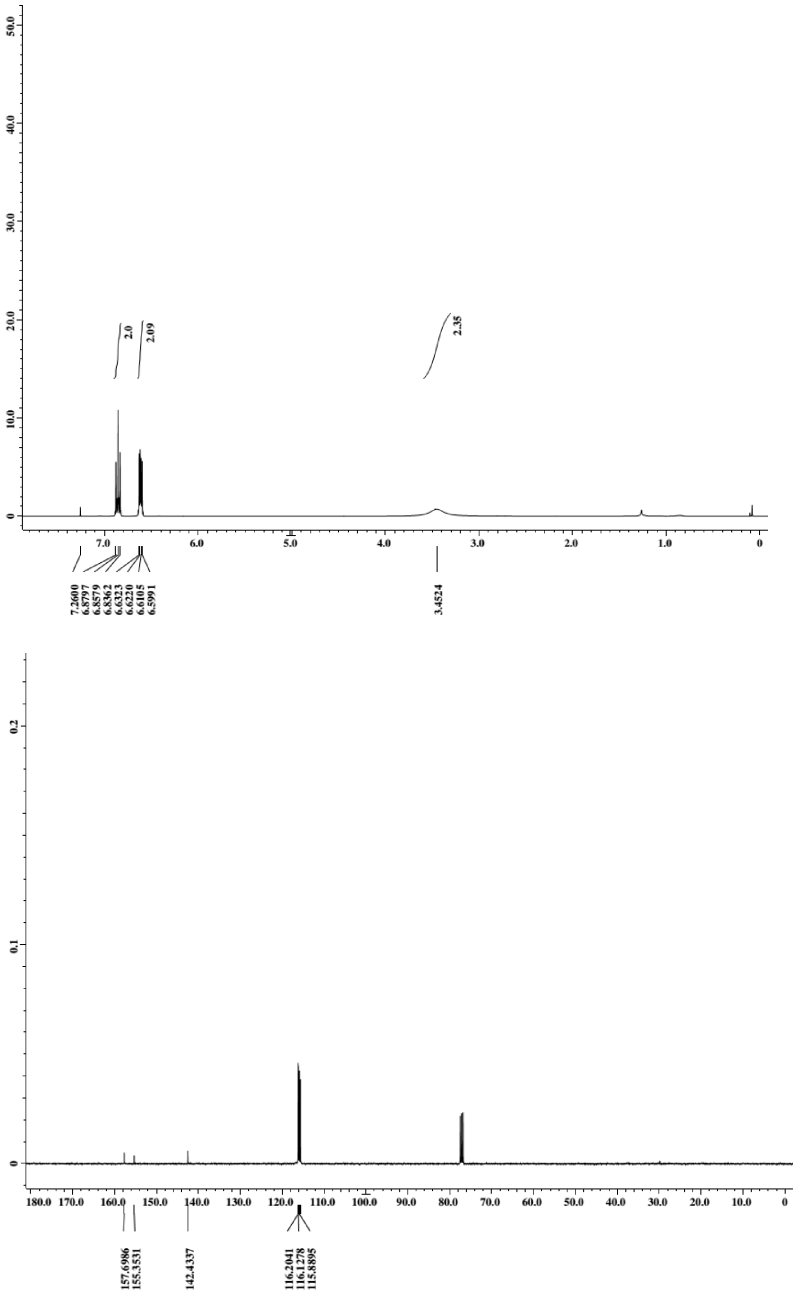

NH2
Br

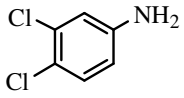
2b


2
2.08
7.2600
7.2428
7.2371
7.2256
7.2211
6.5750
6.5693
6.5590
6.5532
145.3095
132.1459
116.8429
110.1497

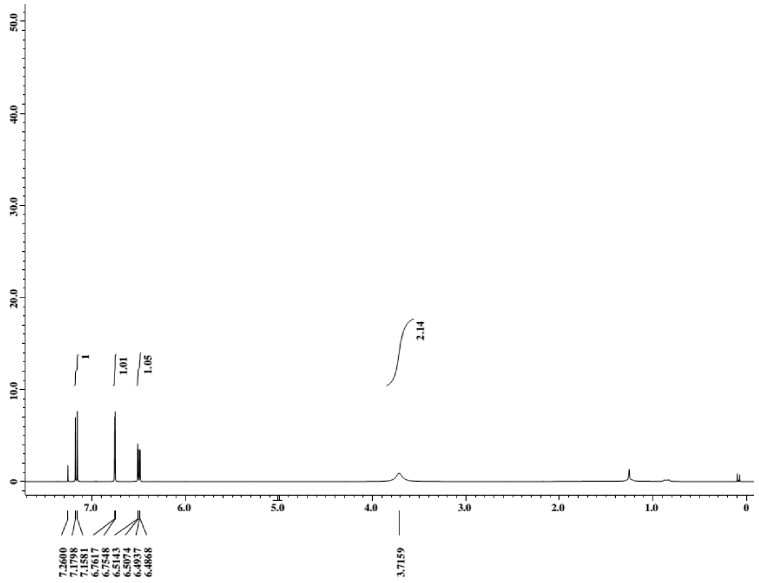

NH2
F

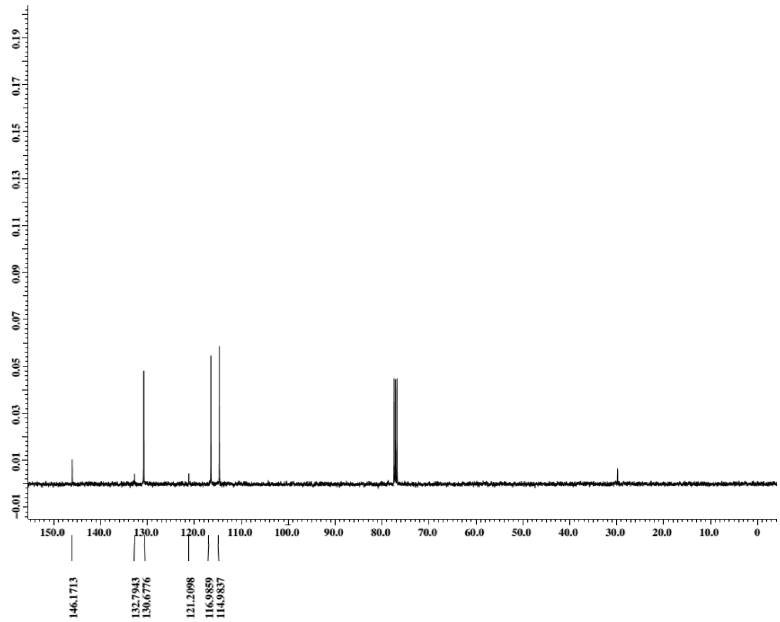
2c

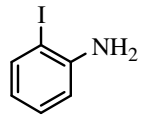

2.0
2.09
2.35
7.2600
6.8797
6.8579
6.8362
6.6323
6.6220
6.6105
6.5991
3.4524
157.6986
155.3531
142.4337
116.2041
116.1278
115.8895


Cl
NH2
Cl

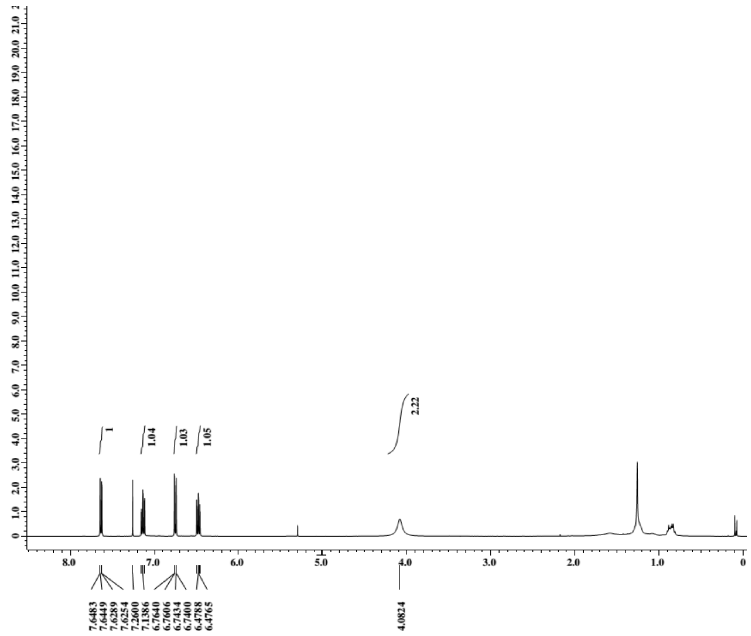
2d

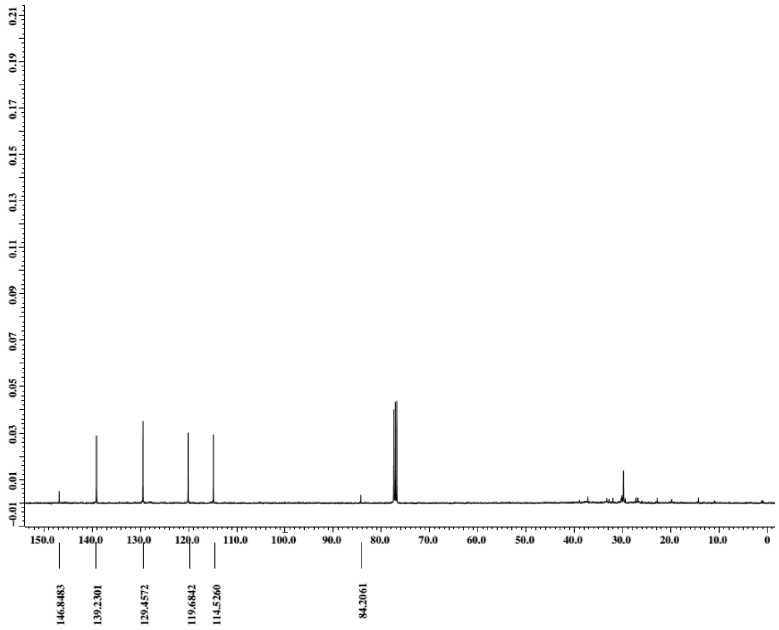

1
1.01
1.05
2.14
7.2600
7.1706
7.1581
6.7617
6.7548
6.5143
6.5074
6.4937
6.4868
3.7159


146.1713
132.7943
130.6776
121.2098
116.9859
114.9837


I
NH2

2e


1
1.04
1.03
1.05
2.22
7.6483
7.6254
7.2600
7.1356
6.7640
6.7606
6.7434
6.4788
6.4765
4.0824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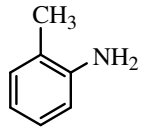
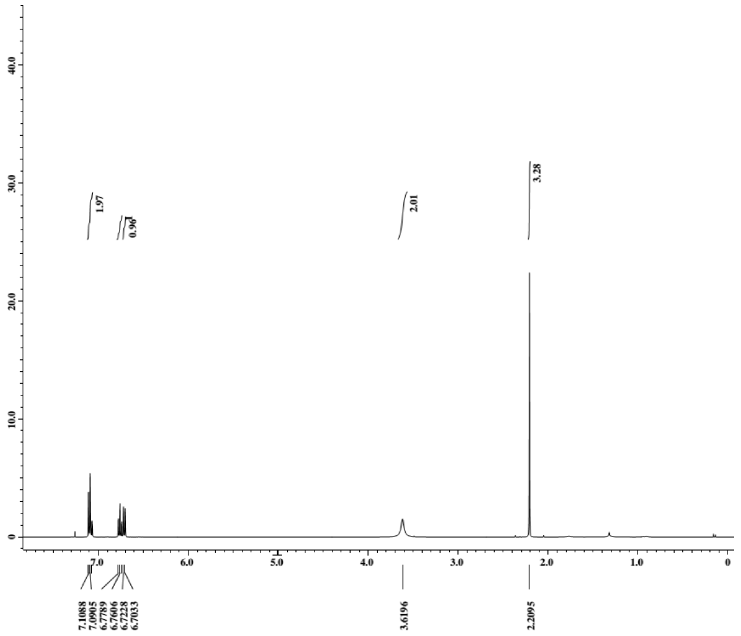
139.2301
129.4572
119.6842
114.5260
84.2061


CH3
NH2

2f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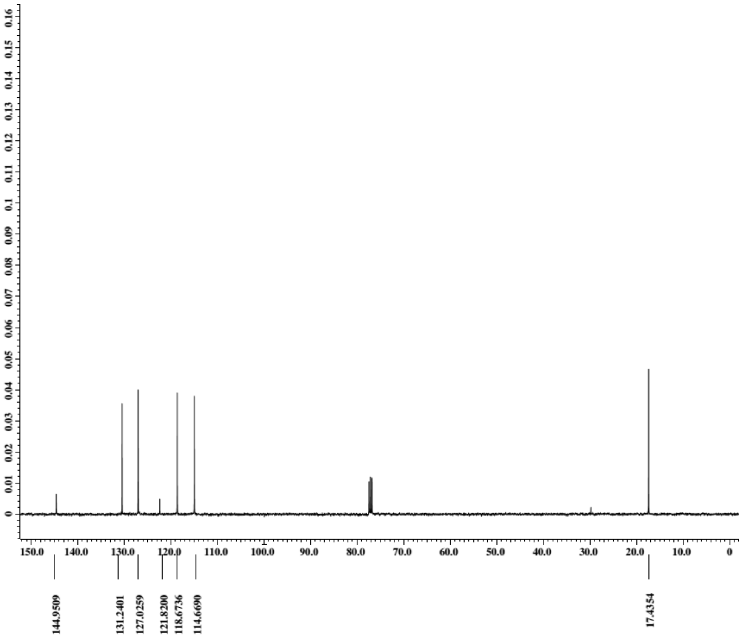
0.90
2.01
3.28
7.1088
7.0905
6.7789
6.7606
6.7233
6.7033
3.6196
2.2095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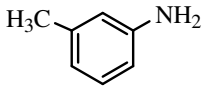
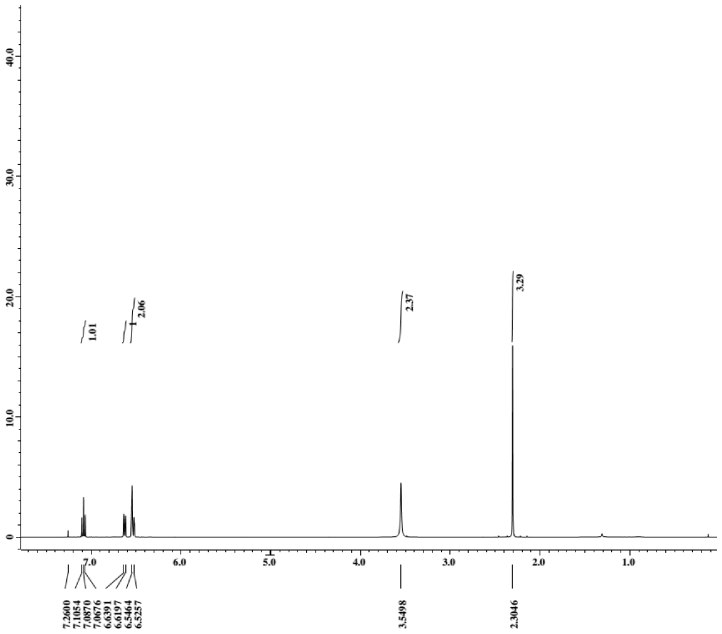
127.0259
121.8200
118.6736
114.6690
17.4354


H3C
NH2

2g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2.06
2.37
3.29
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7.0876
7.0076
6.6491
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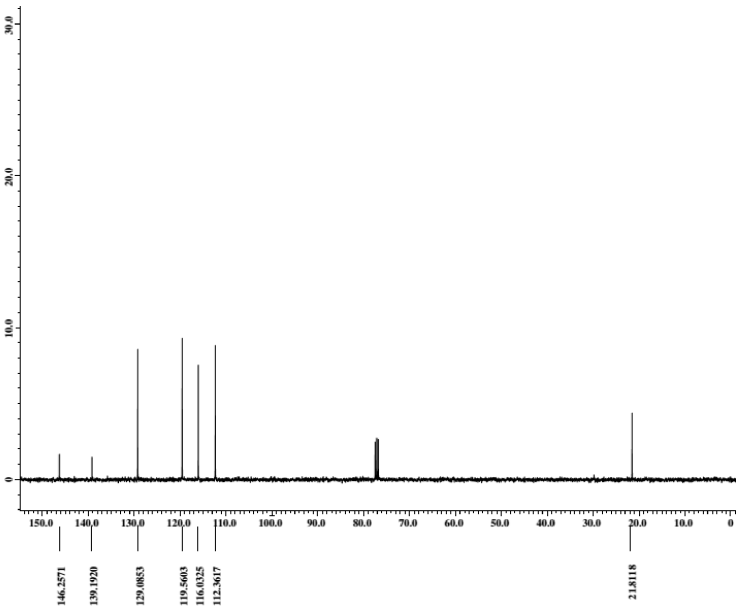
6.5464
6.5257
3.5498
2.3046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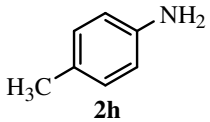
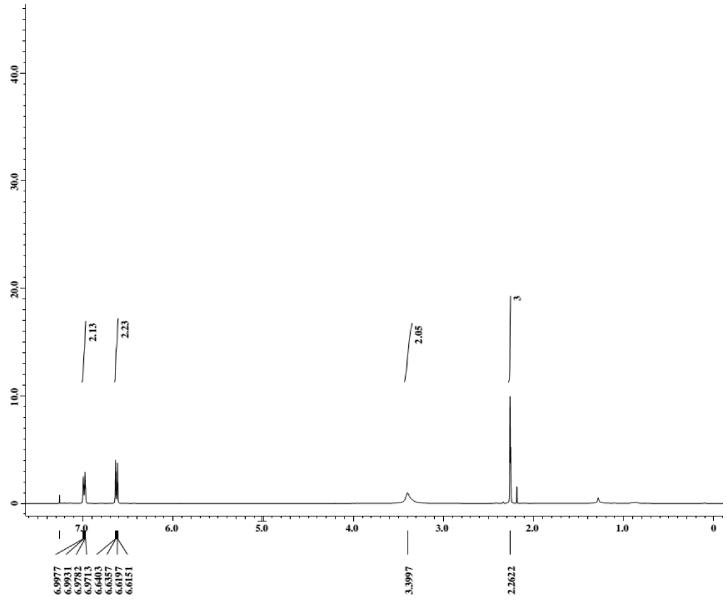
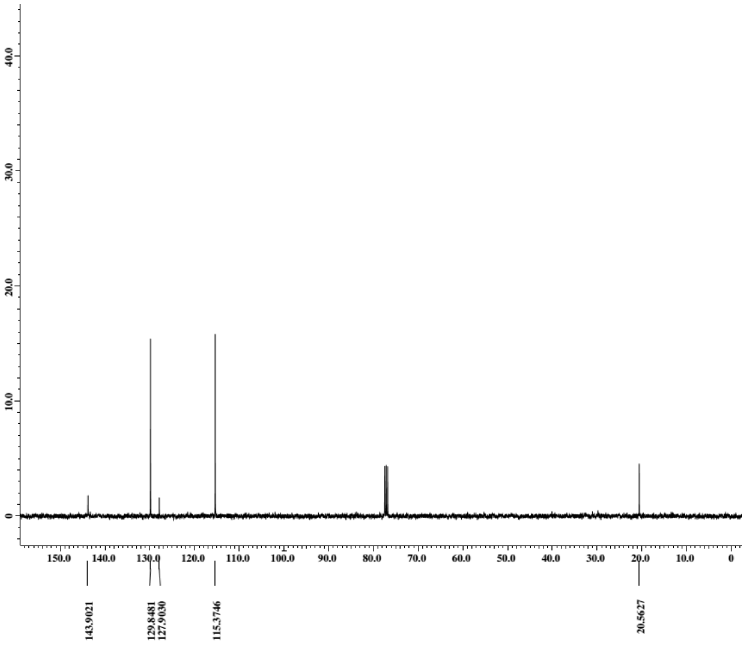
112.3617
21.8118


NH2
H3C

2h


2.13
2.23
2.05
3
6.9977
6.9931
6.9782
6.9713
6.6493
6.6357
6.6197
6.6151
3.3997
2.2622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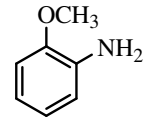
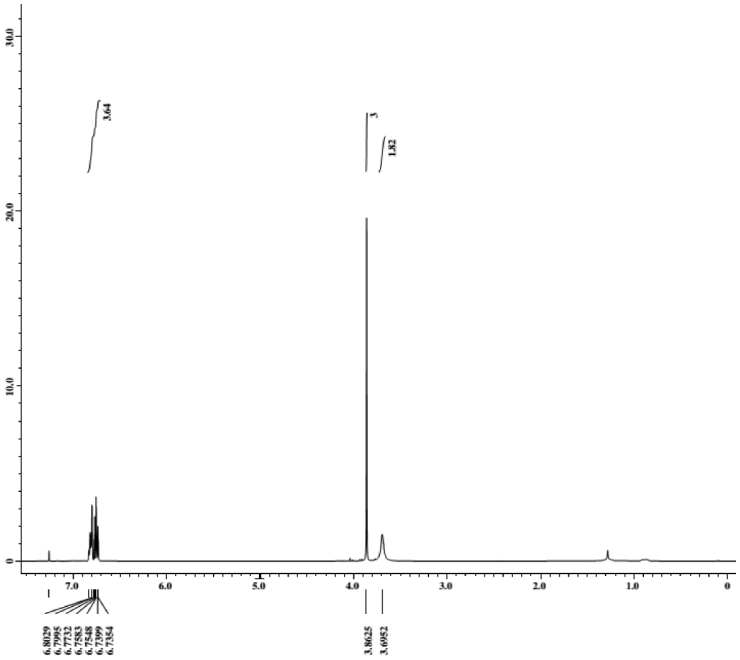
129.8481
127.9030
115.3746
20.5627


OCH3
NH2

2i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3
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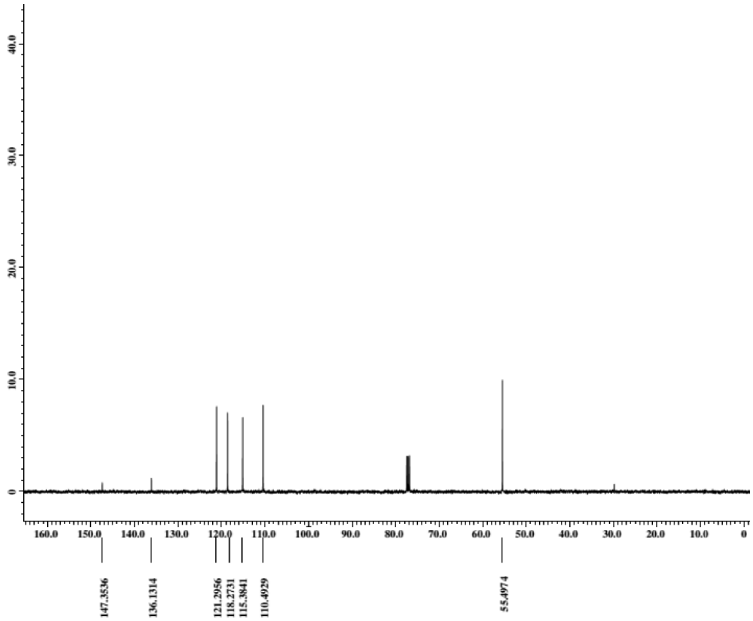
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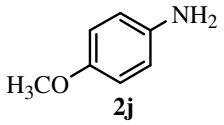
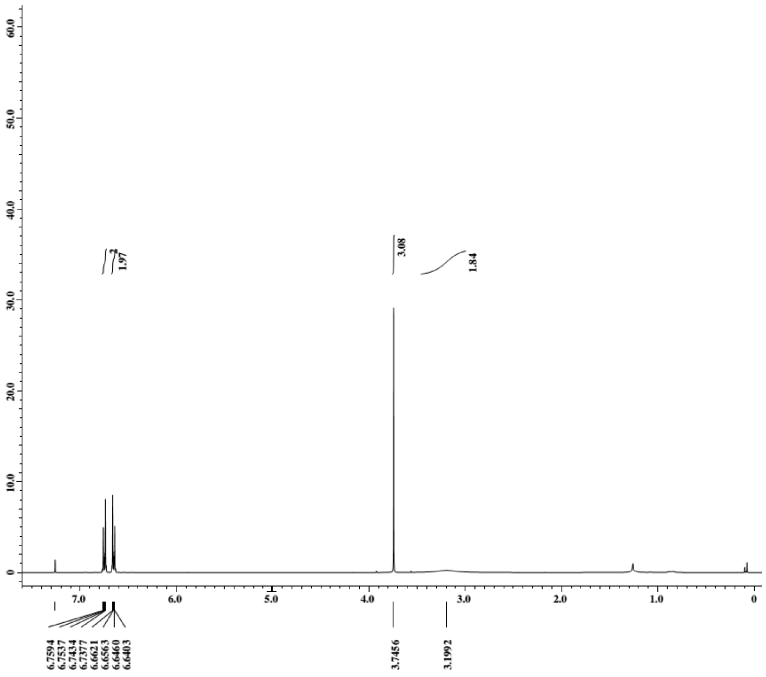

NH2
H3CO

2j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6.6460
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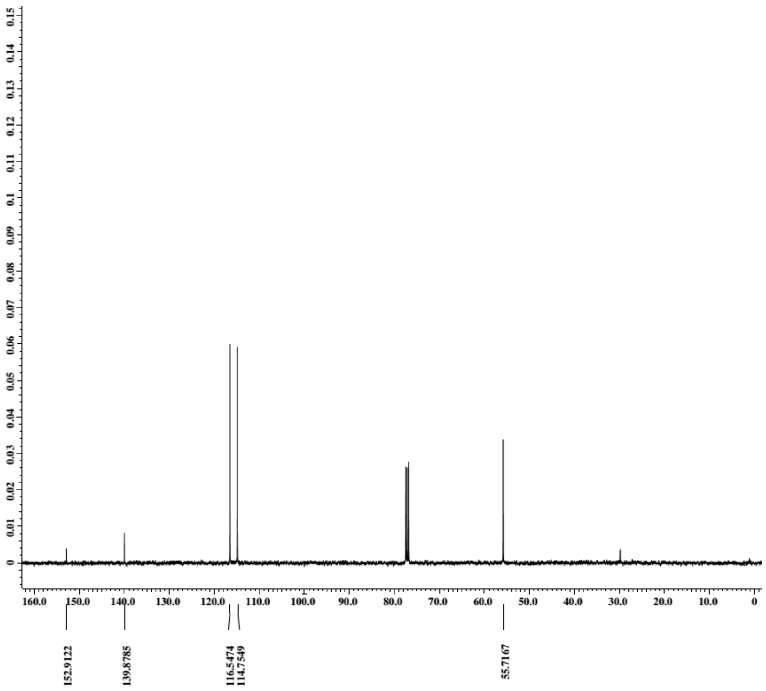
3.7456
3.1992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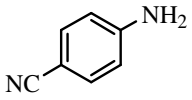
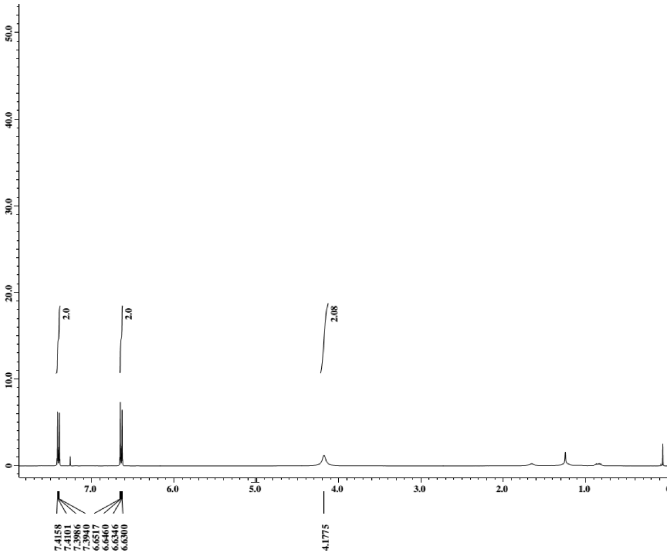
114.7549
55.7167


NH2
NC

2k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2.0
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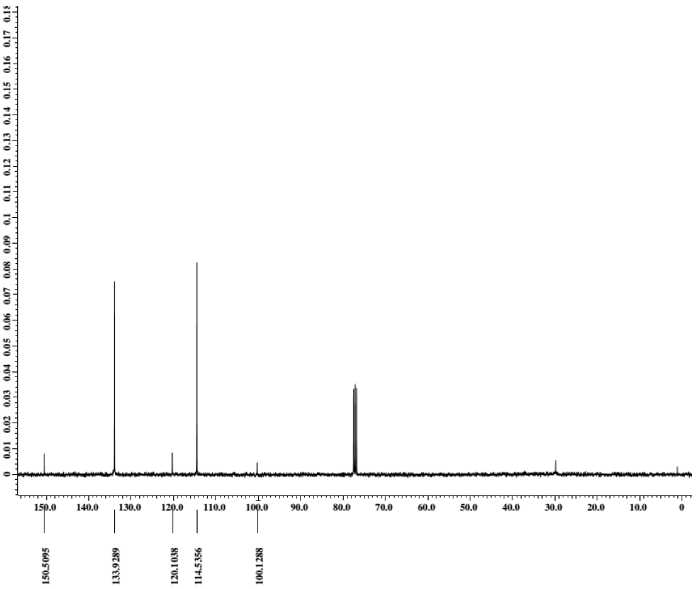
6.6360
4.1775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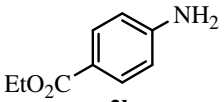
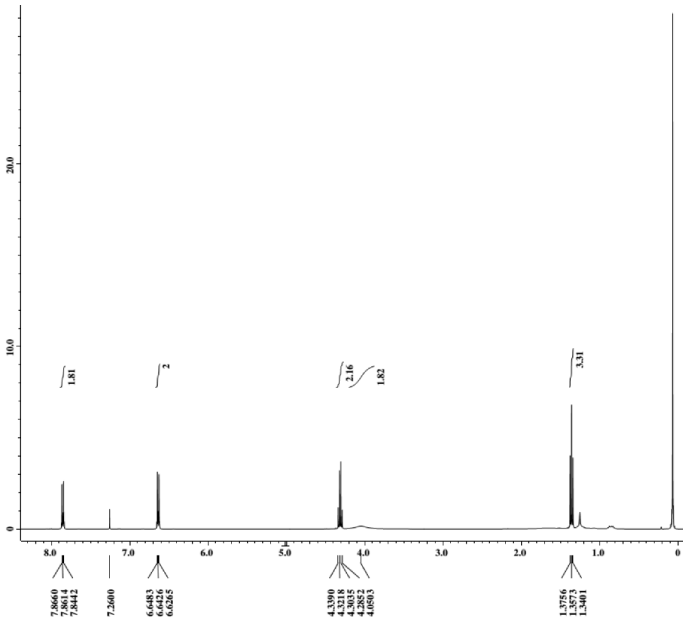
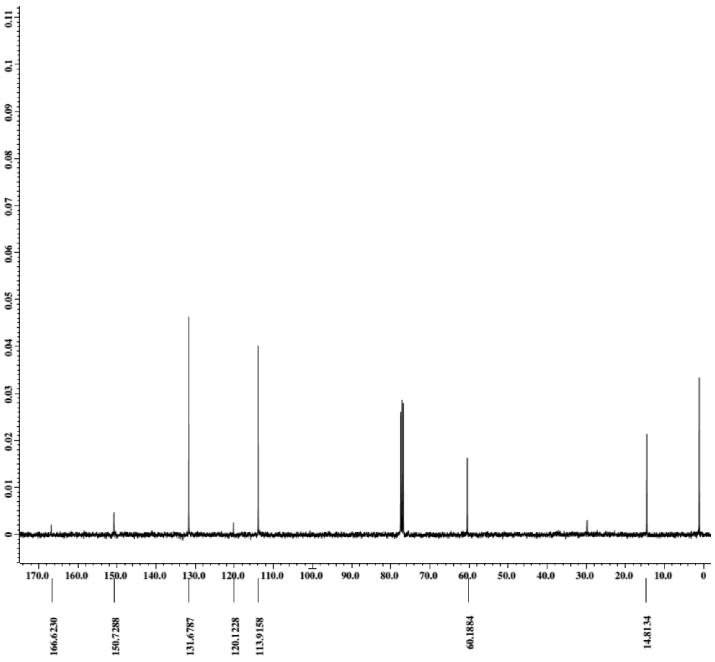
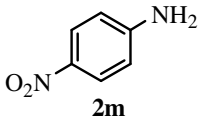
100.1288



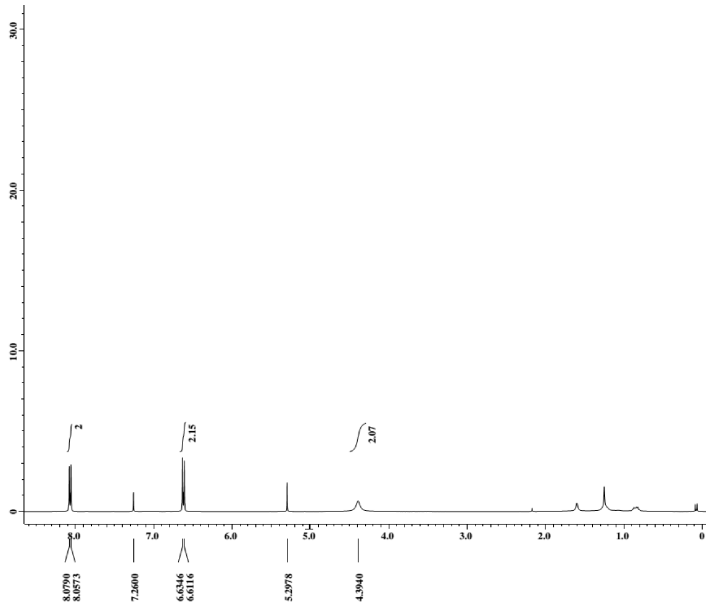
2l






NH2
O2N

2m


2
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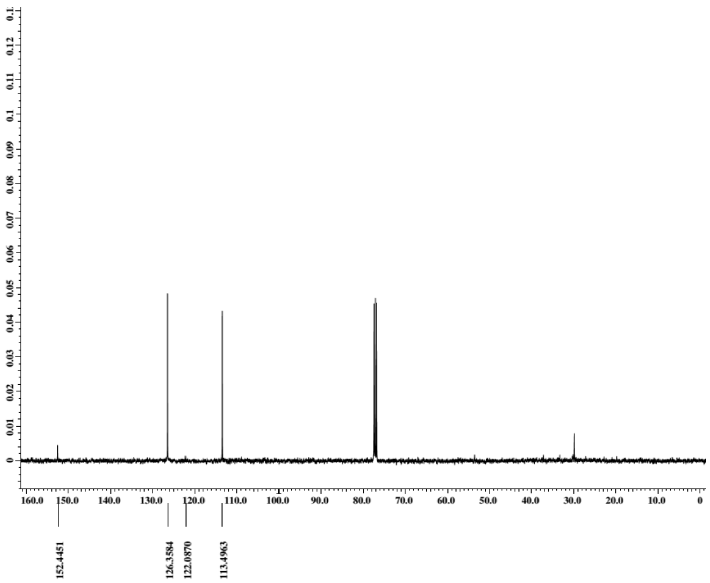
2.07
8.0790
8.0573
7.2600
6.6346
6.6116
5.2978
4.3940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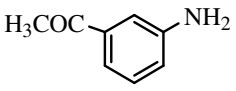
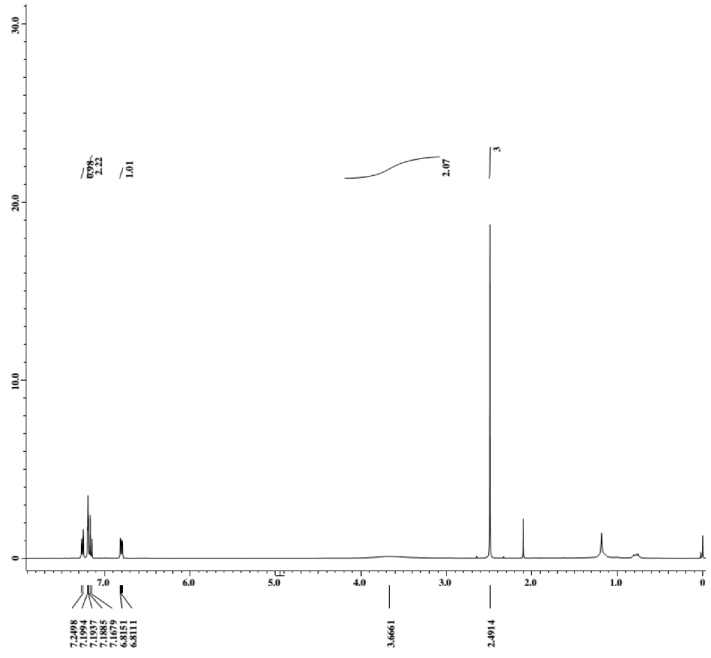

H3COC
NH2

2n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7.1679
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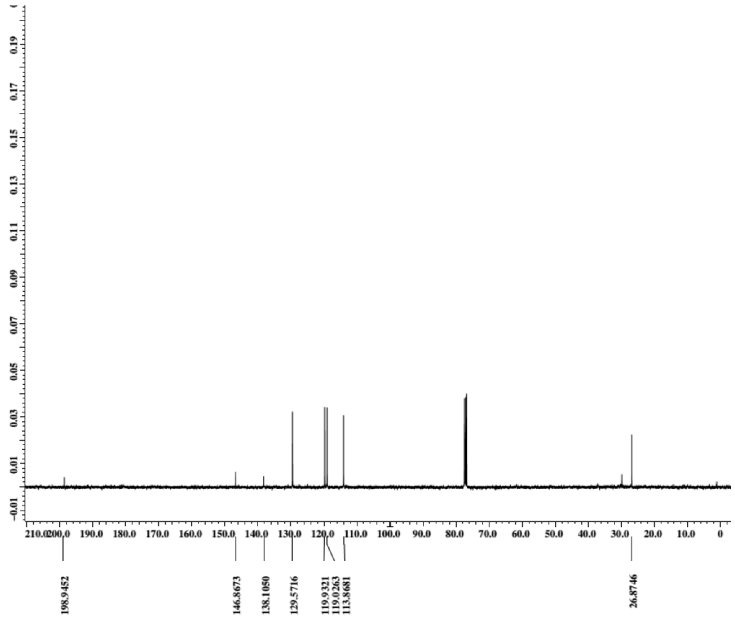
3.6661
2.4914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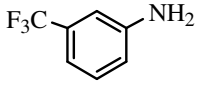
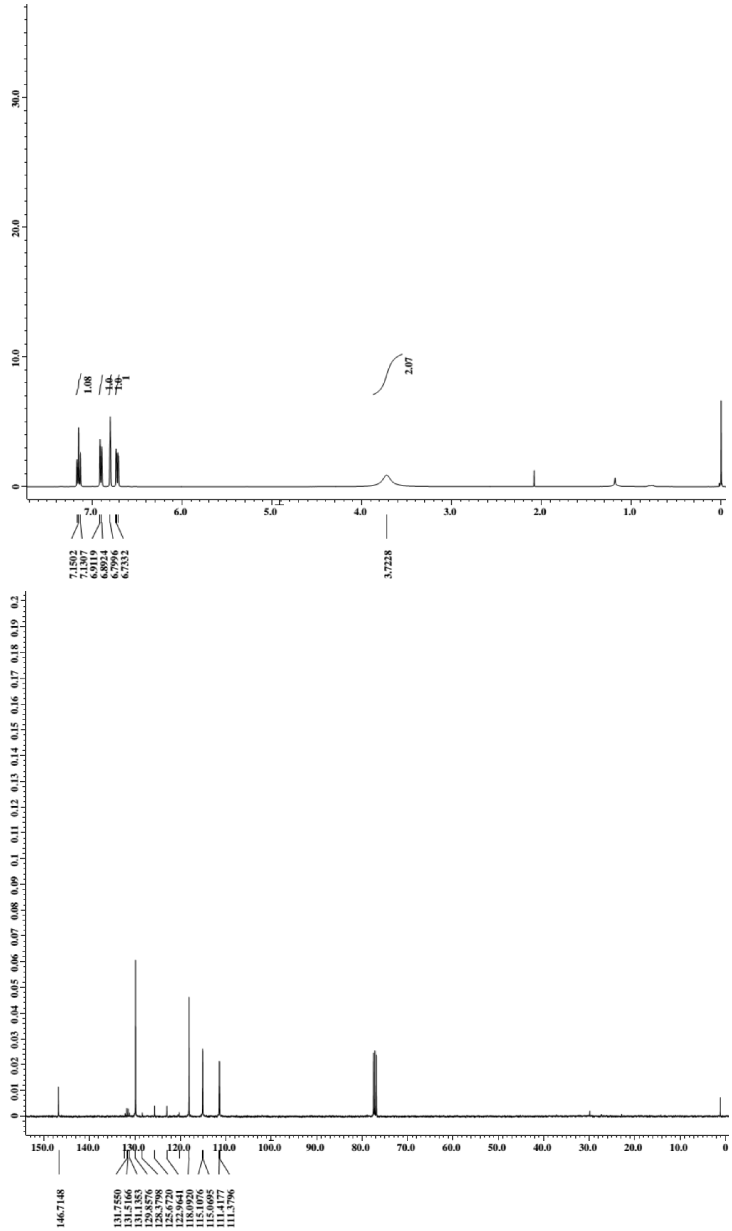
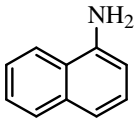
26.8746

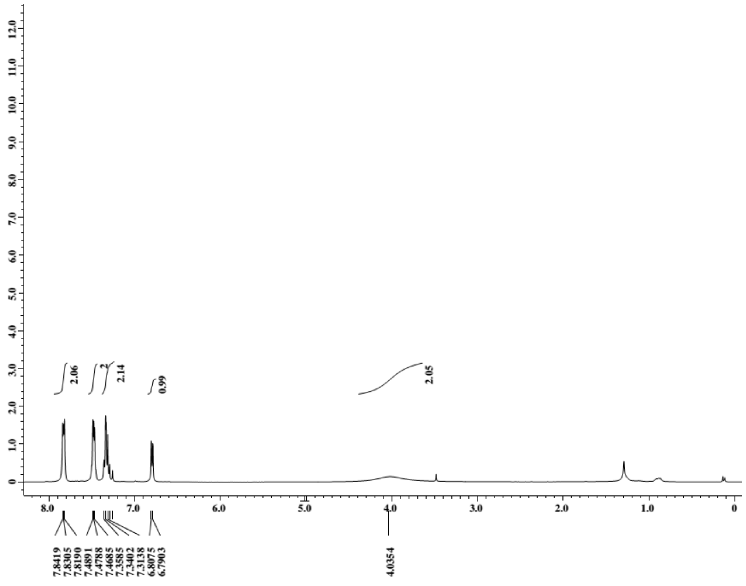


2o




NH2

2p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2.14
0.99
2.05
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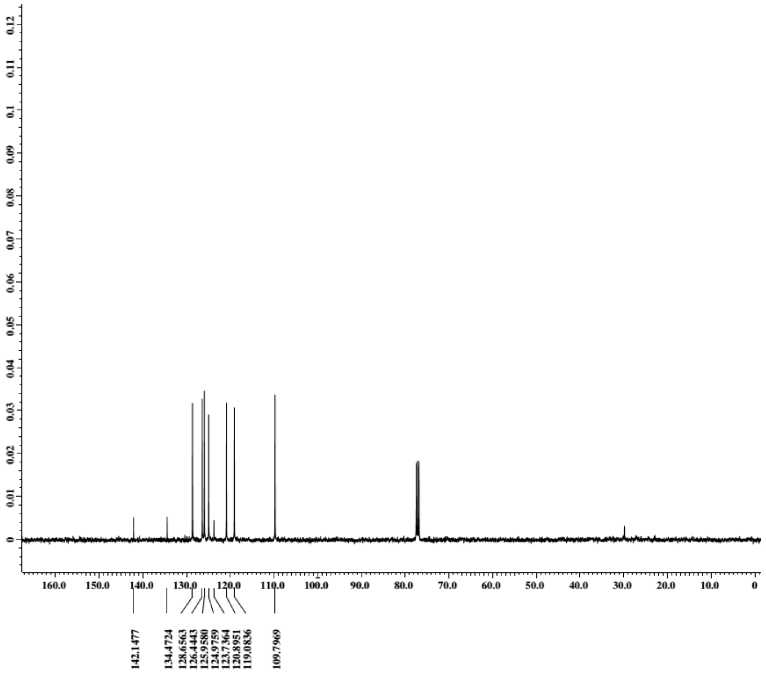
7.3138
6.6955
6.7903
4.0354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124.4580
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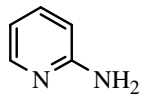
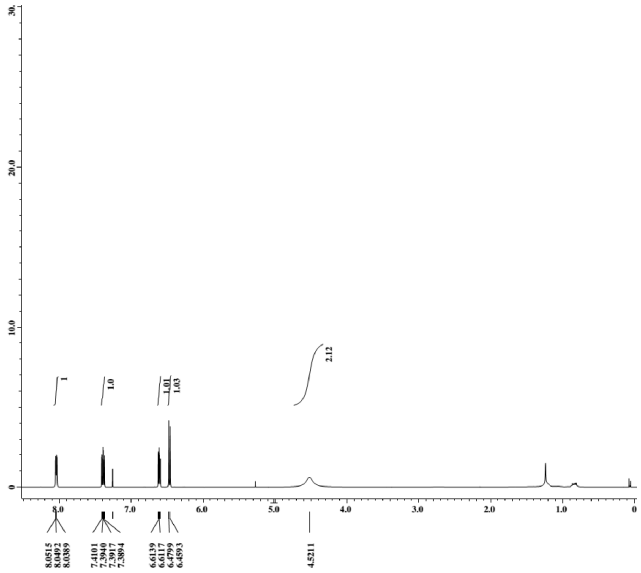
109.7969


N
NH2

4a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1.01
1.03
2.12
8.0515
8.0492
8.0389
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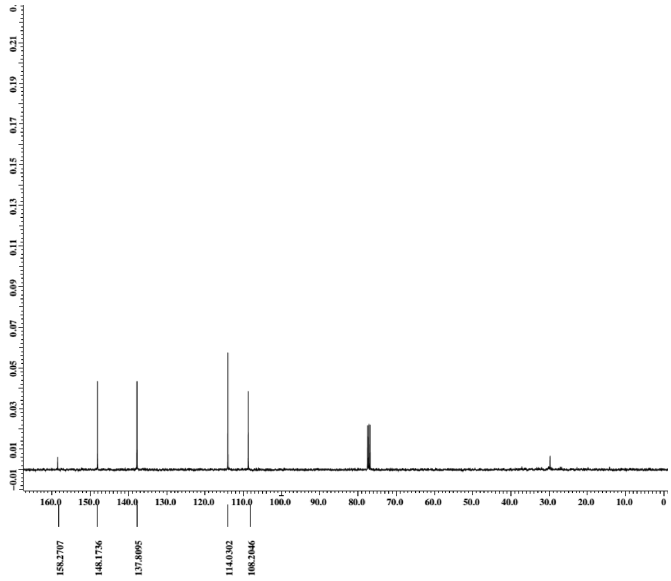
7.3980
7.3917
7.3884
6.6139
6.6117
6.4799
6.4593
4.5211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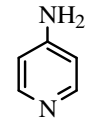

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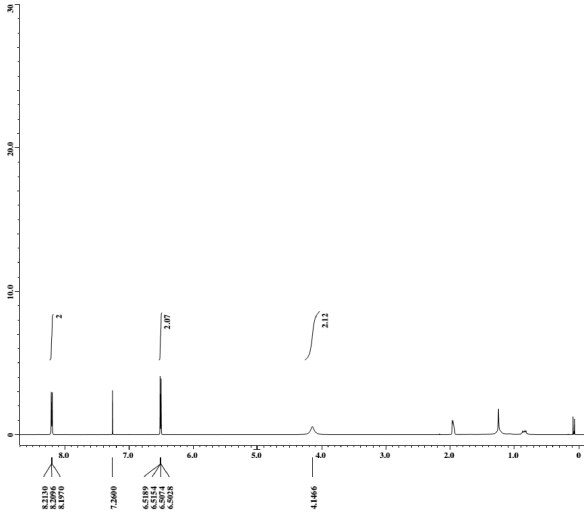
2
2.07
2.12
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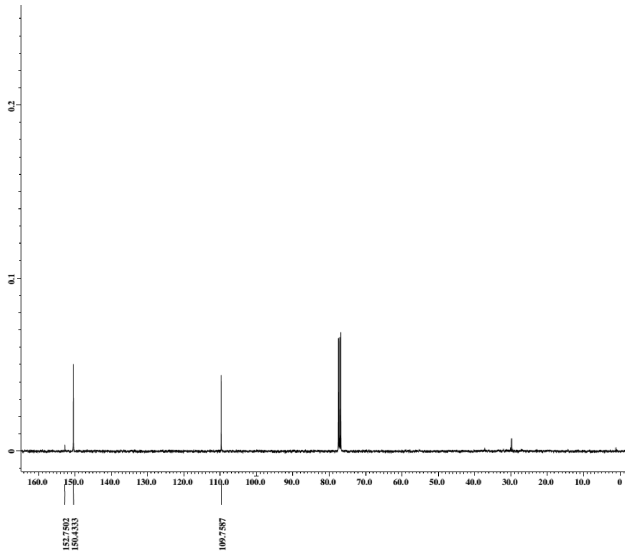
8.2004
8.1970
7.2600
6.5189
6.5154
6.5074
6.5028
4.1466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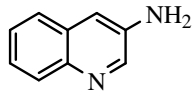
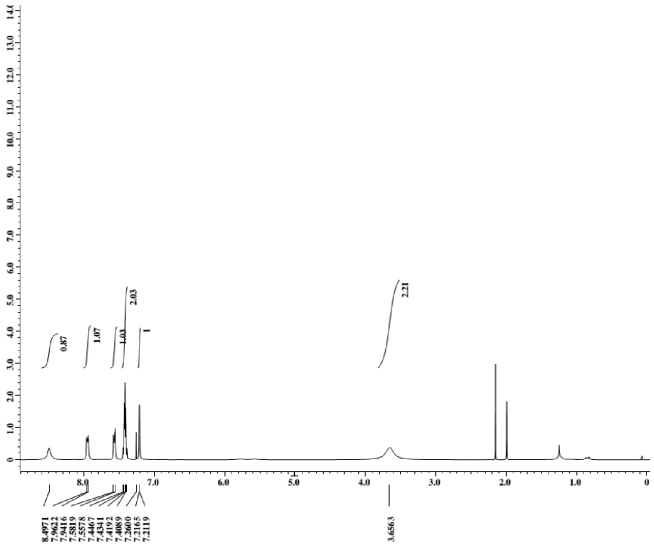
150.4333
109.7857


NH2
N

4c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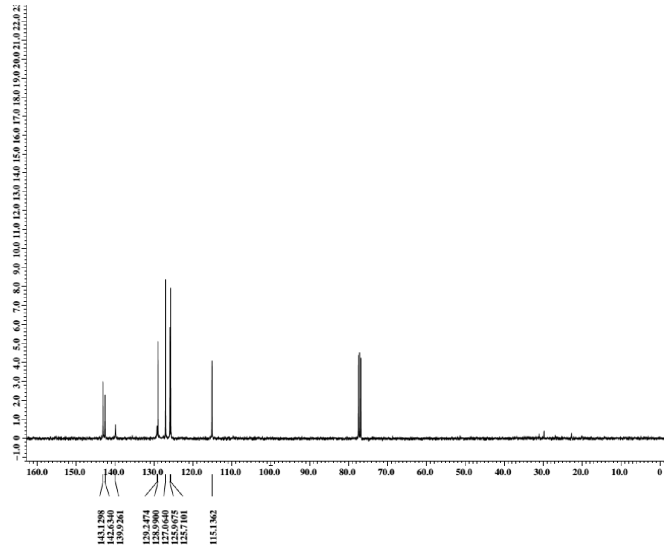
7.4069
7.2600
7.2165
7.2119
3.6563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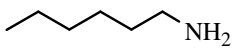
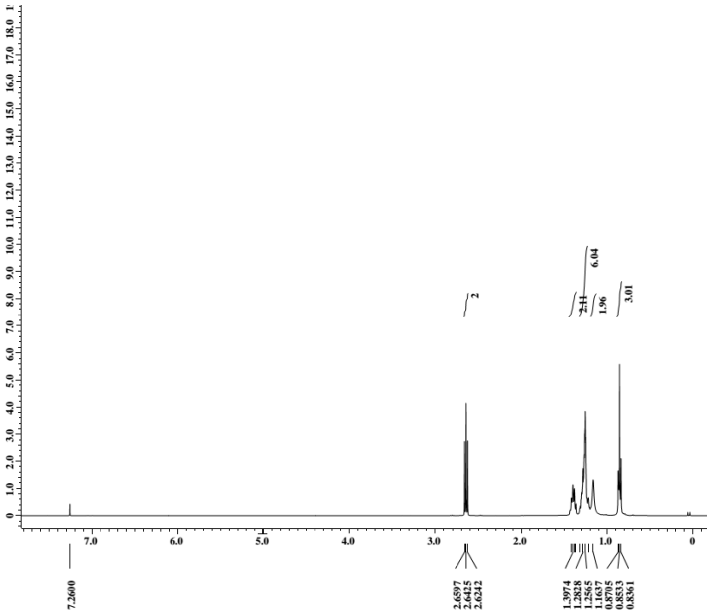

NH2

6a


2
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6.04
1.96
3.01
7.2600
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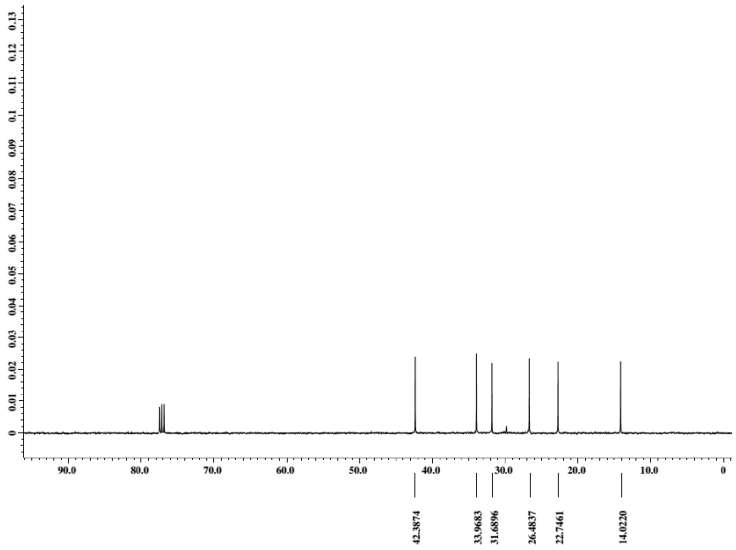
2.6242
1.3974
1.2828
1.2565
1.1457
0.8795
0.8533
0.8361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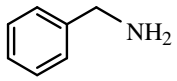
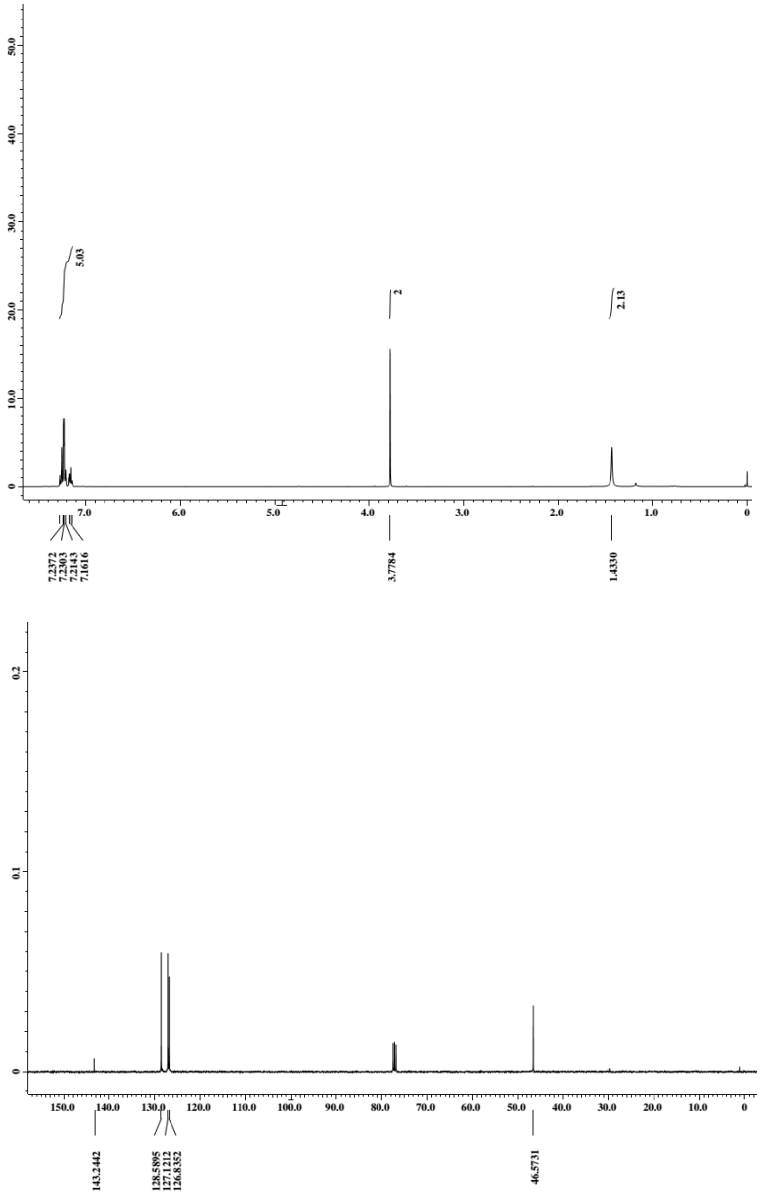
31.6896
26.4837
22.7461
14.0220


NH2

6b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2
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1.4330
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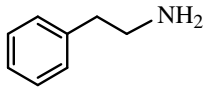
127.1212
126.8352
46.5731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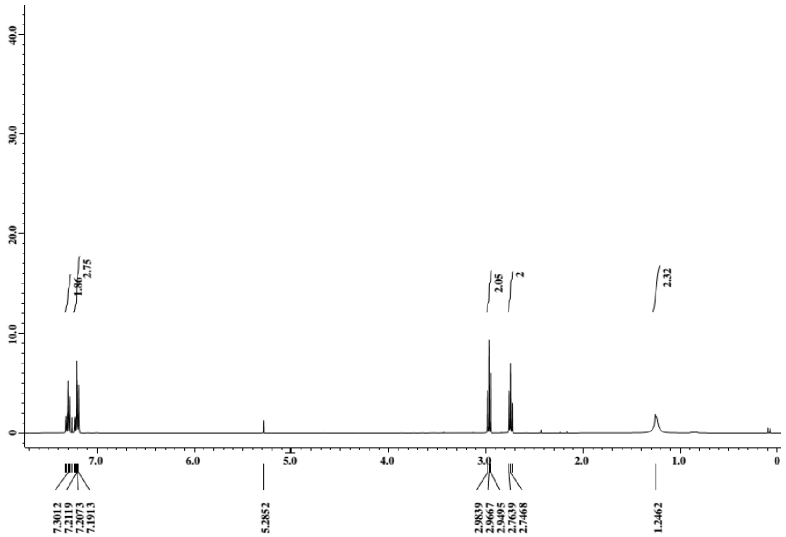
6c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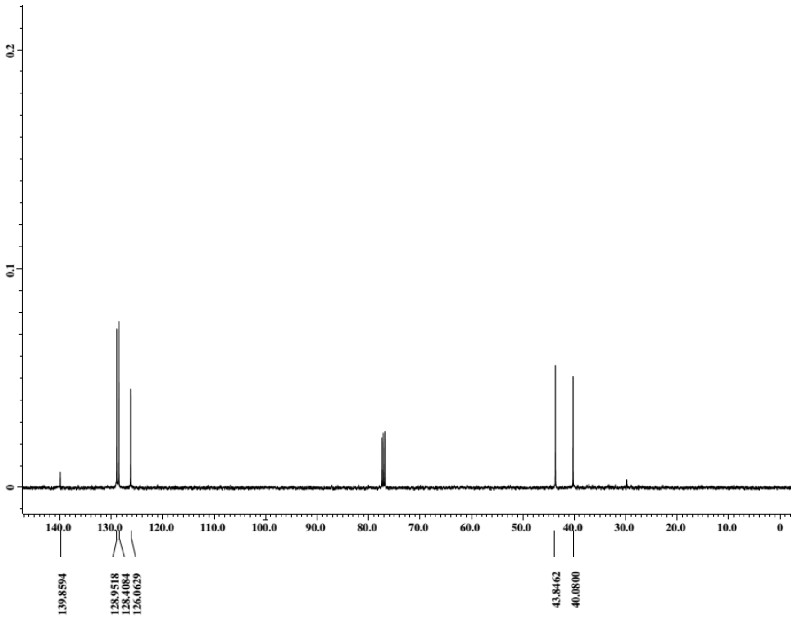
30.0
20.0
10.0
0
1.86
2.75
2.05
2
2.32
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.3012
7.2710
7.2073
7.1913
5.2852
2.9839
2.9667
2.8485
2.7639
2.7468
1.2462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0.1
0
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130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
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20.0
10.0
0
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128.4084
126.0029
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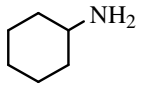
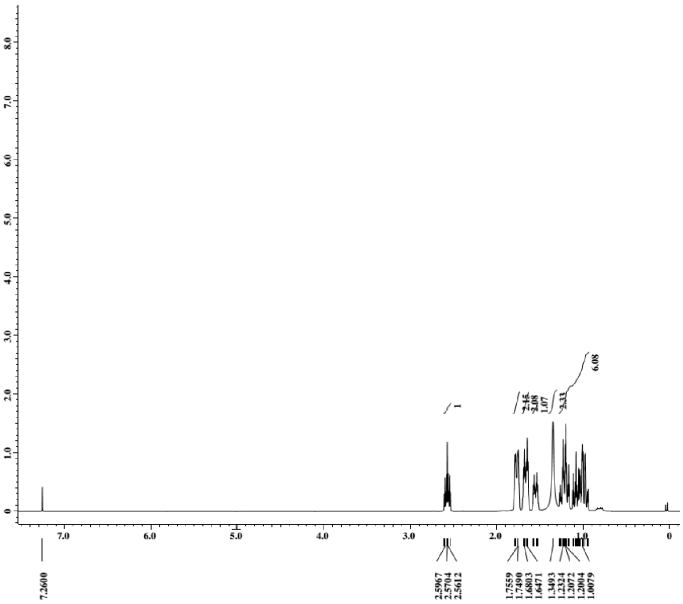
40.0800


NH2

6d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1.07
3.33
6.08
7.2600
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1.7490
1.6683
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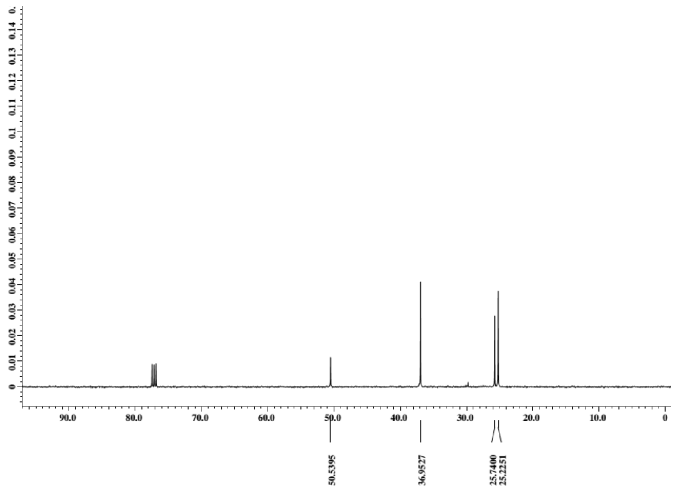
1.3493
1.2324
1.2192
1.2004
1.0079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25.2251