Electronic Supplementary Material (ESI) for RSC Advances.

Supporting information

Microwave-assisted synthesis of novel 2, 3-disubstituted imidazo[1,2-a]pyridines via one-pot three component reactions

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1. General information.

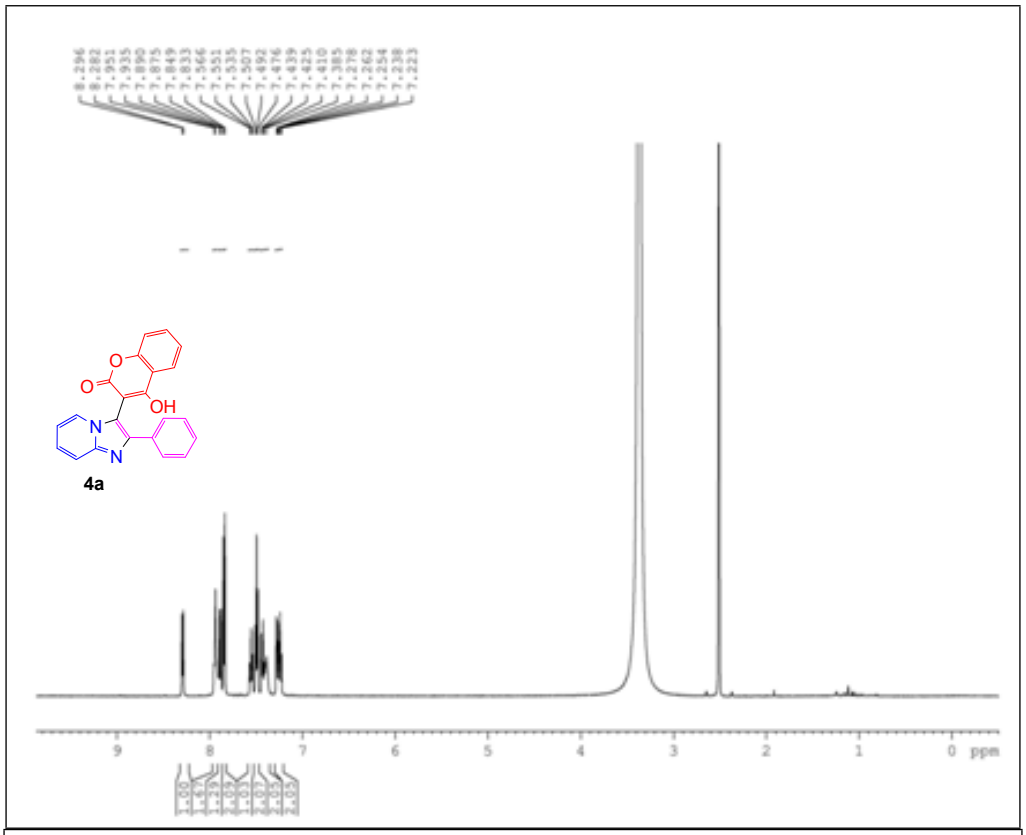
All starting materials were purchased either from Sigma Aldrich or Alfa Aesar and used without further purification. NMR spectra were recorded on 400 or 500 MHz for ^{1}H and 100 or 125 MHz for ^{13}C in D_2O or DMSO-d6, Chemical shift values were reported in δ values (ppm) downfield from tetramethylsilane. Infrared (IR) spectra were recorded on a Shimadzu IR Affinity-1, FTIR spectrometer. Elemental analyses were carried out using vario MICRO cube elemental analyzer. Microwave irradiation was carried out with Initiator 2.5 Microwave Synthesizers from Biotage, Uppsala, Sweden. Melting points were recorded by using SRS EZ-Melt automated melting point apparatus by capillary methods and uncorrected.

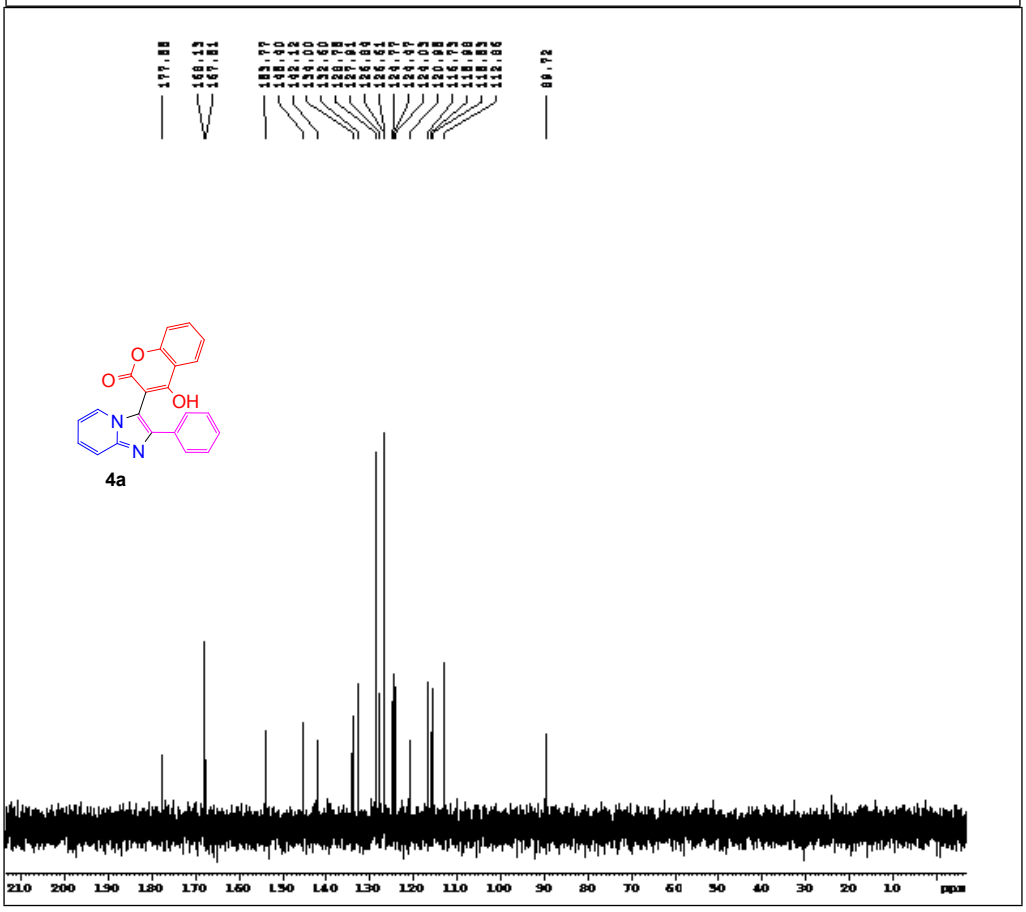
2. Typical experimental procedure for the synthesis of 1a.

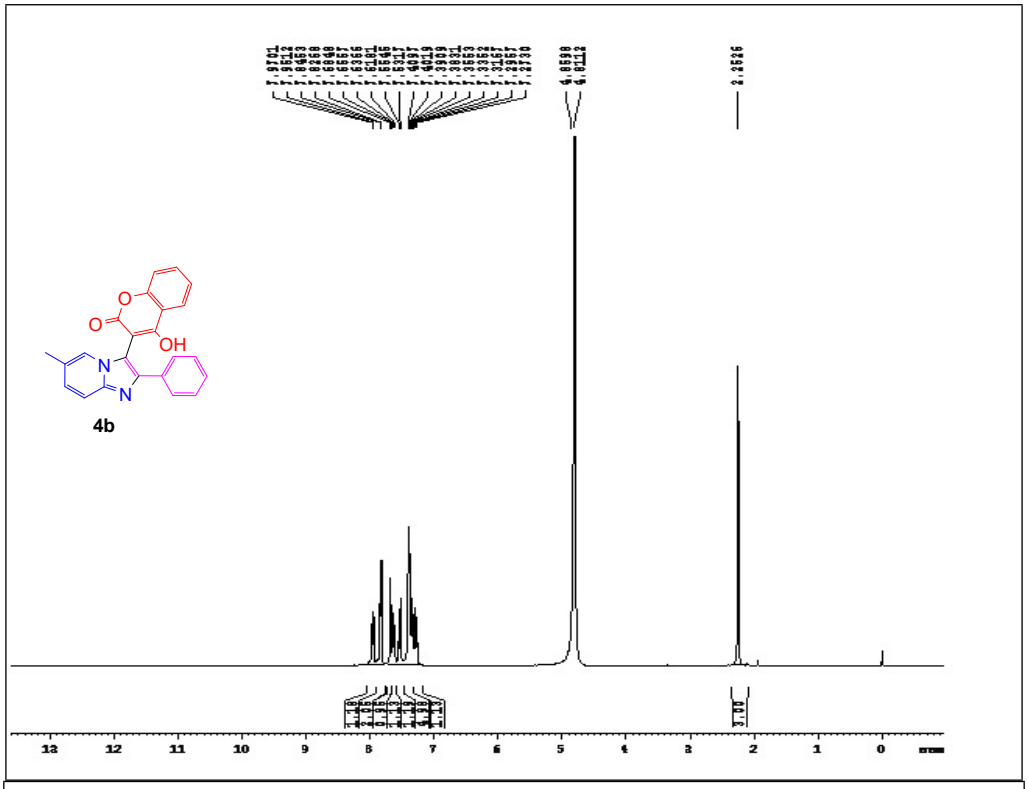
A mixture of phenylglyoxal monohydrate (**1a)** (1 mmol), 4-hydroxycoumarin (**2a)** (1 mmol), 2-aminopyridine (**3a)** (1 mmol) and iodine (0.3 mmol) in 3 mL ethanol was introduced in a 2-5 mL reaction vial, the mixture was irradiated for 15 min, keeping temperature at 130 ^{o}C (Absorption Level: High; Fixed Hold Time and 200 W). The reaction mixture was then cooled to room temperature and the solid was filtered off, and was washed with water-ethanol mixture to get the desired three component product **4a.**

Due to poor solubility of these compounds in common organic solvents, in most of the cases the resultant products were treated with 1.0 equivalent NaOH in 2 ml H_2O to prepare the corresponding sodium enolate of the products and the solvent was removed under vacuum to record NMR in D_2O.

3. Copies of ^{1}H and ^{13}C NMR spectra of compounds



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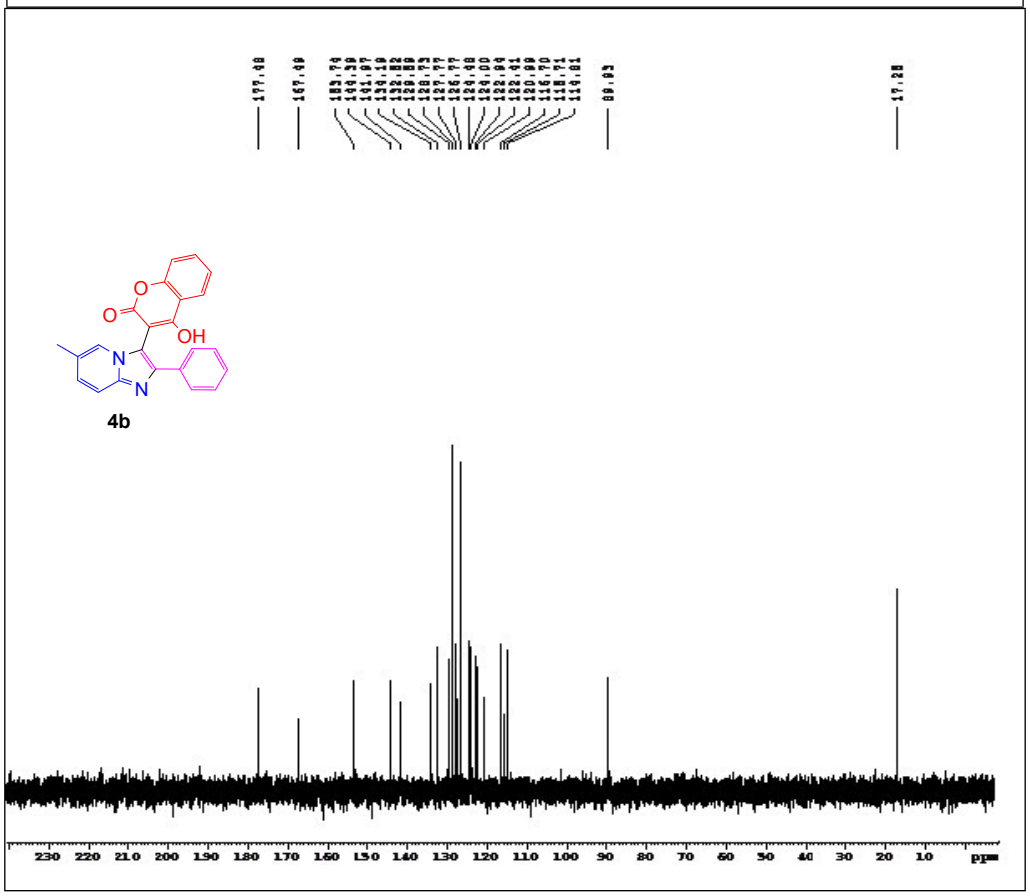
N
N
4b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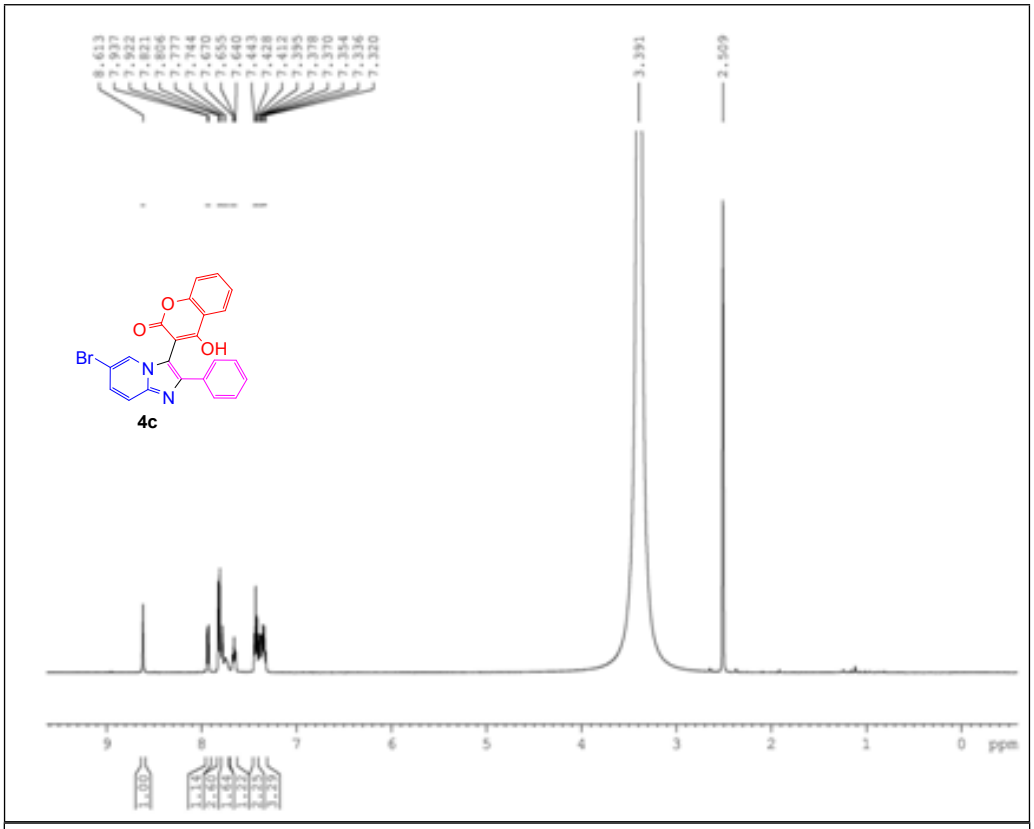
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3.391
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N
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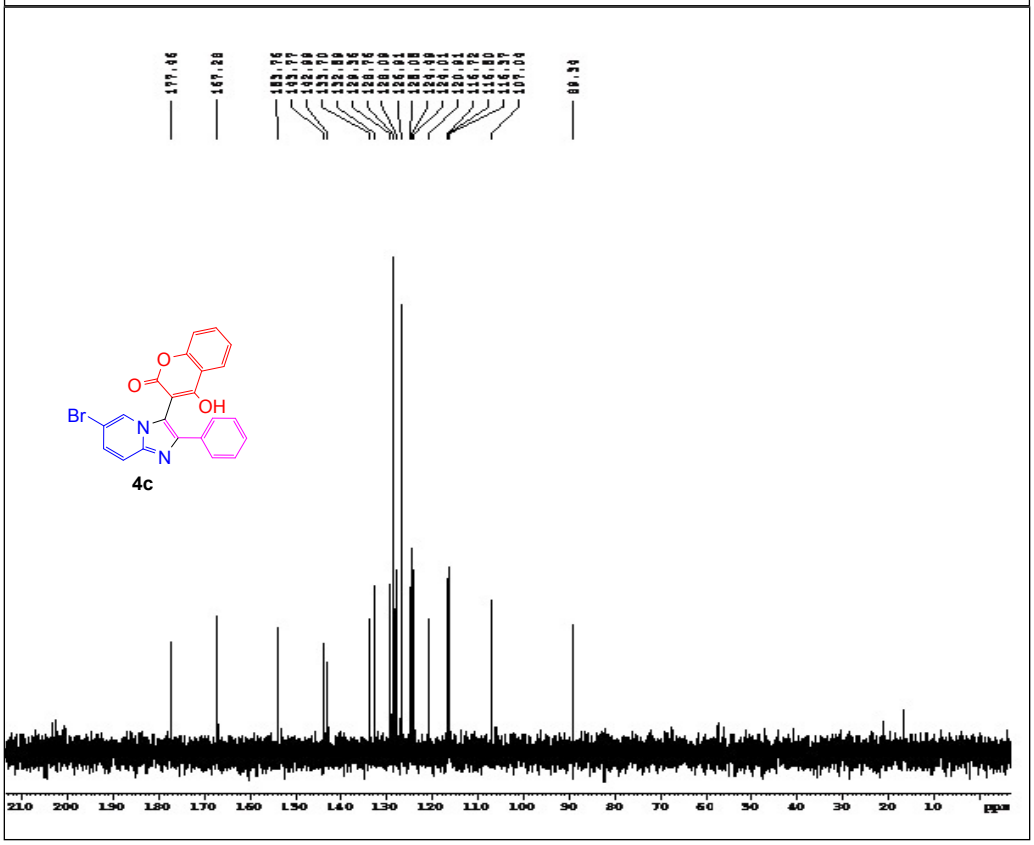
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124.01
120.91
116.72
116.80
116.37
107.04
80.34
O
O
OH
Br
N
N
4c
210
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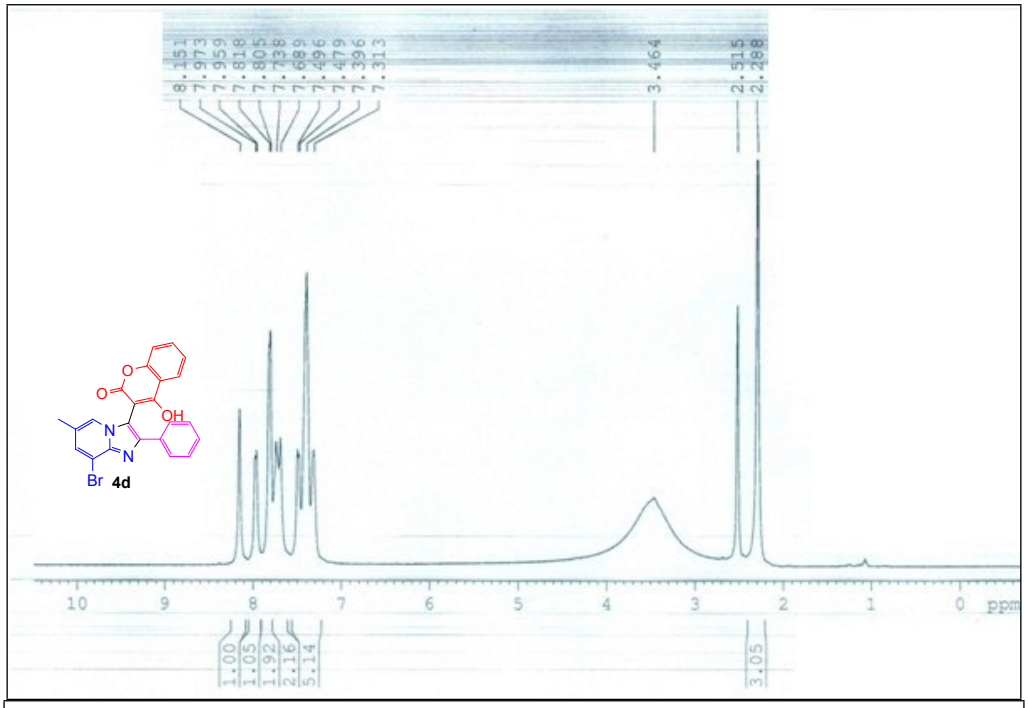
ppm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2.288
O
O
OH
N
N
Br
4d
10
9
8
7
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5
4
3
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0
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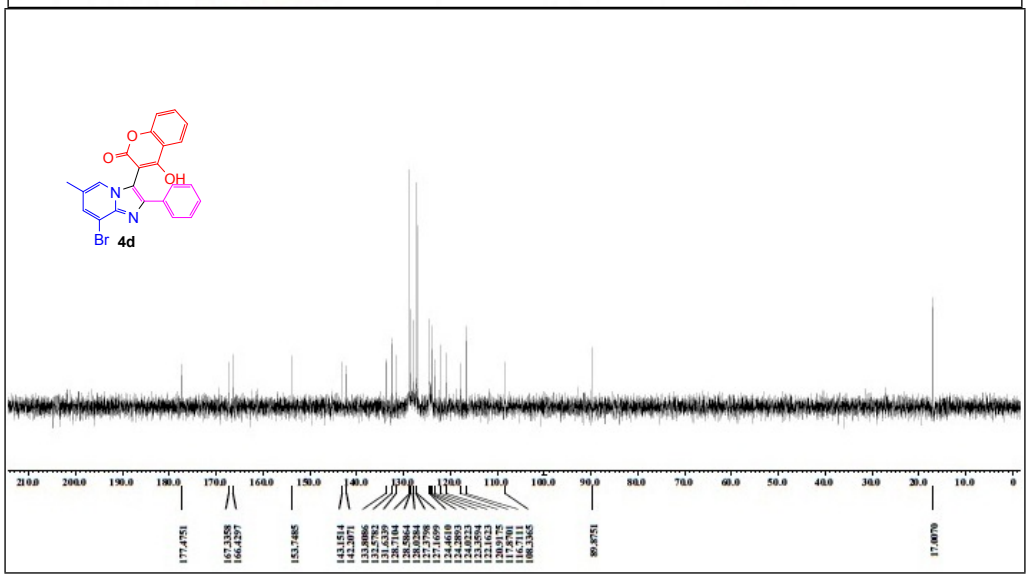
5.14
3.05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40.0
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10.0
0
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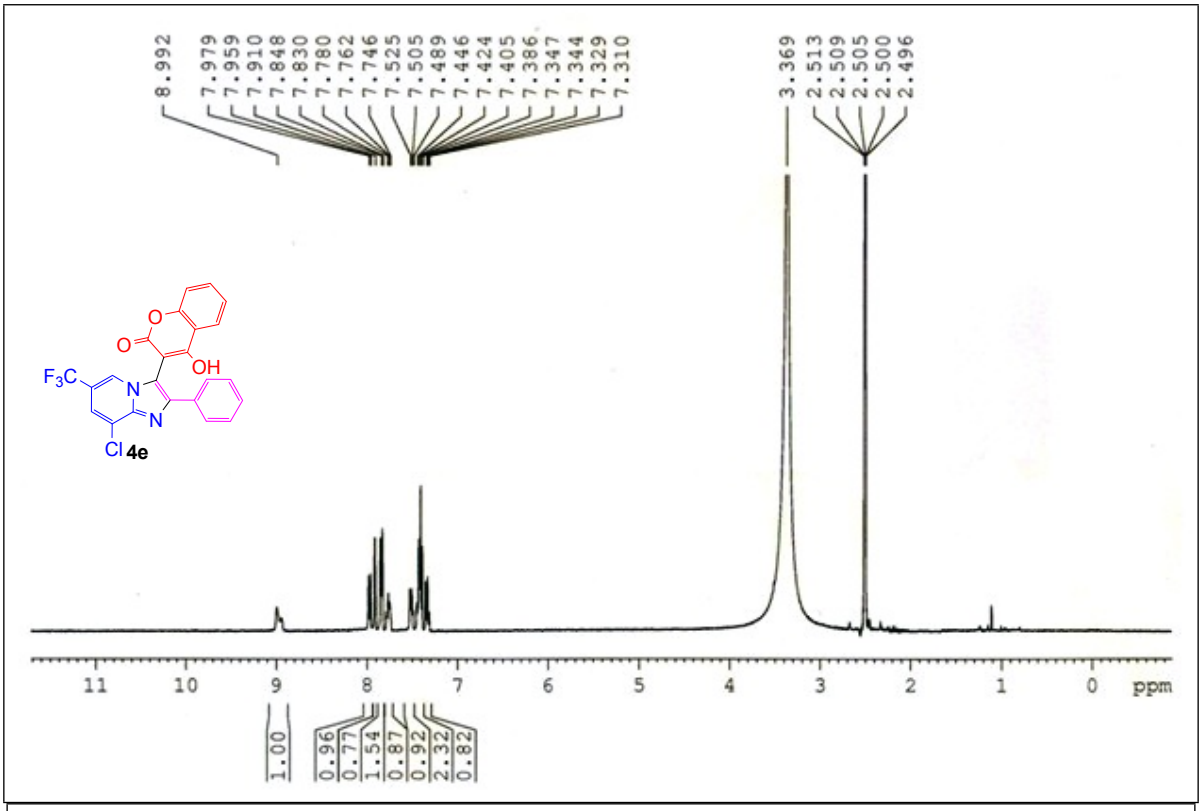
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2.509
2.505
2.500
2.496
O
O
OH
F3C
N
N
Cl
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10
9
8
7
6
5
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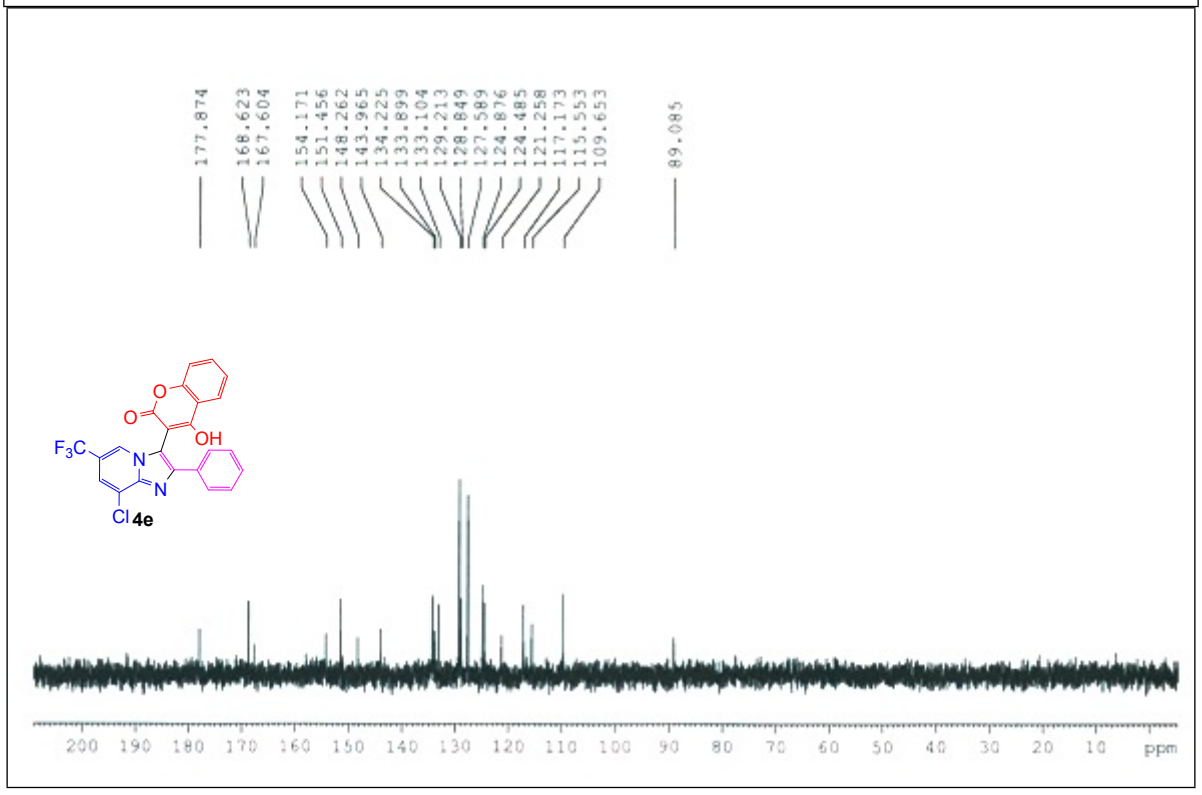
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O
OH
F3C
N
N
Cl
4e
200
190
180
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150
140
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120
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70
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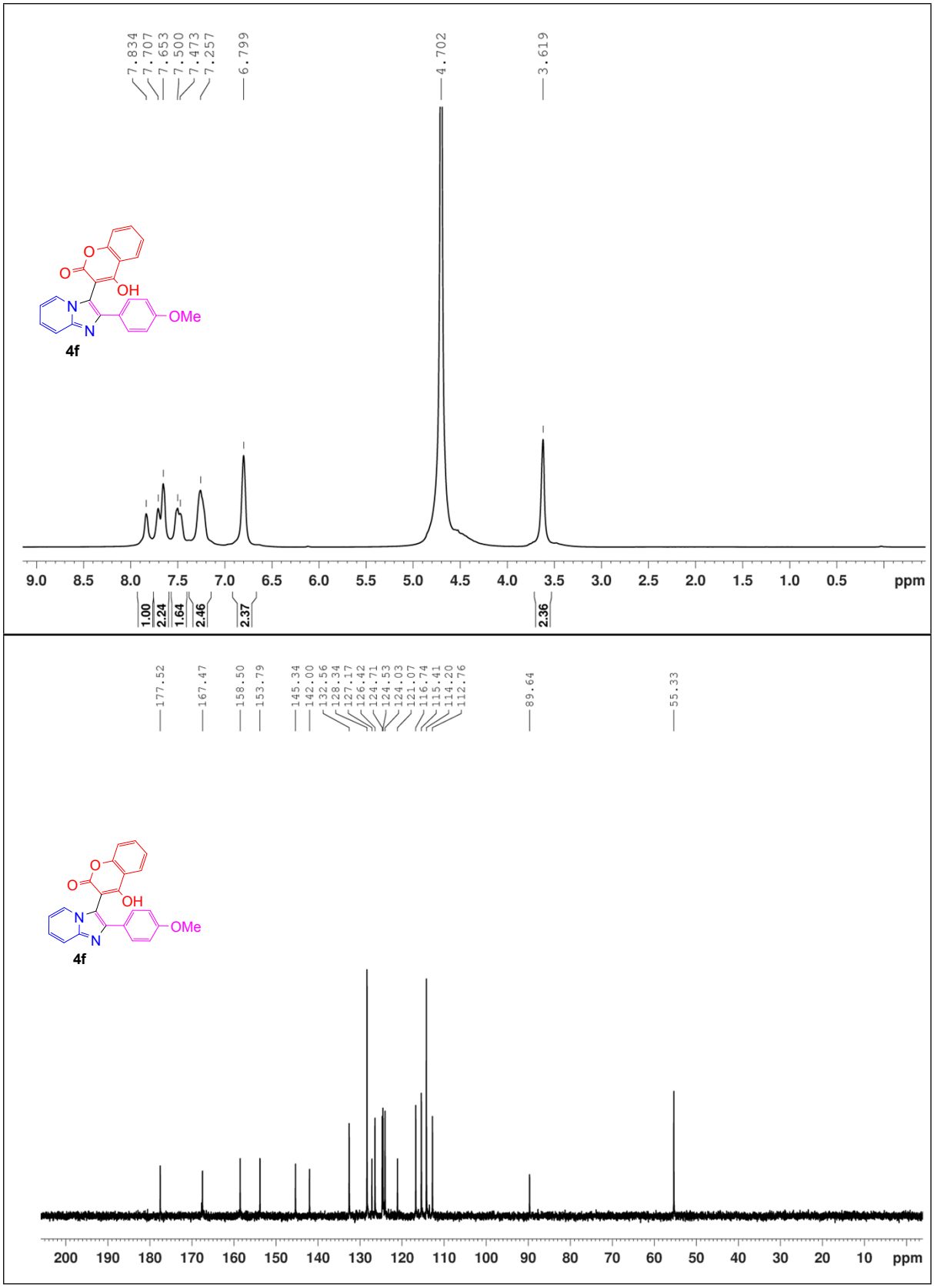
ppm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127.17
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124.71
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124.03
121.07
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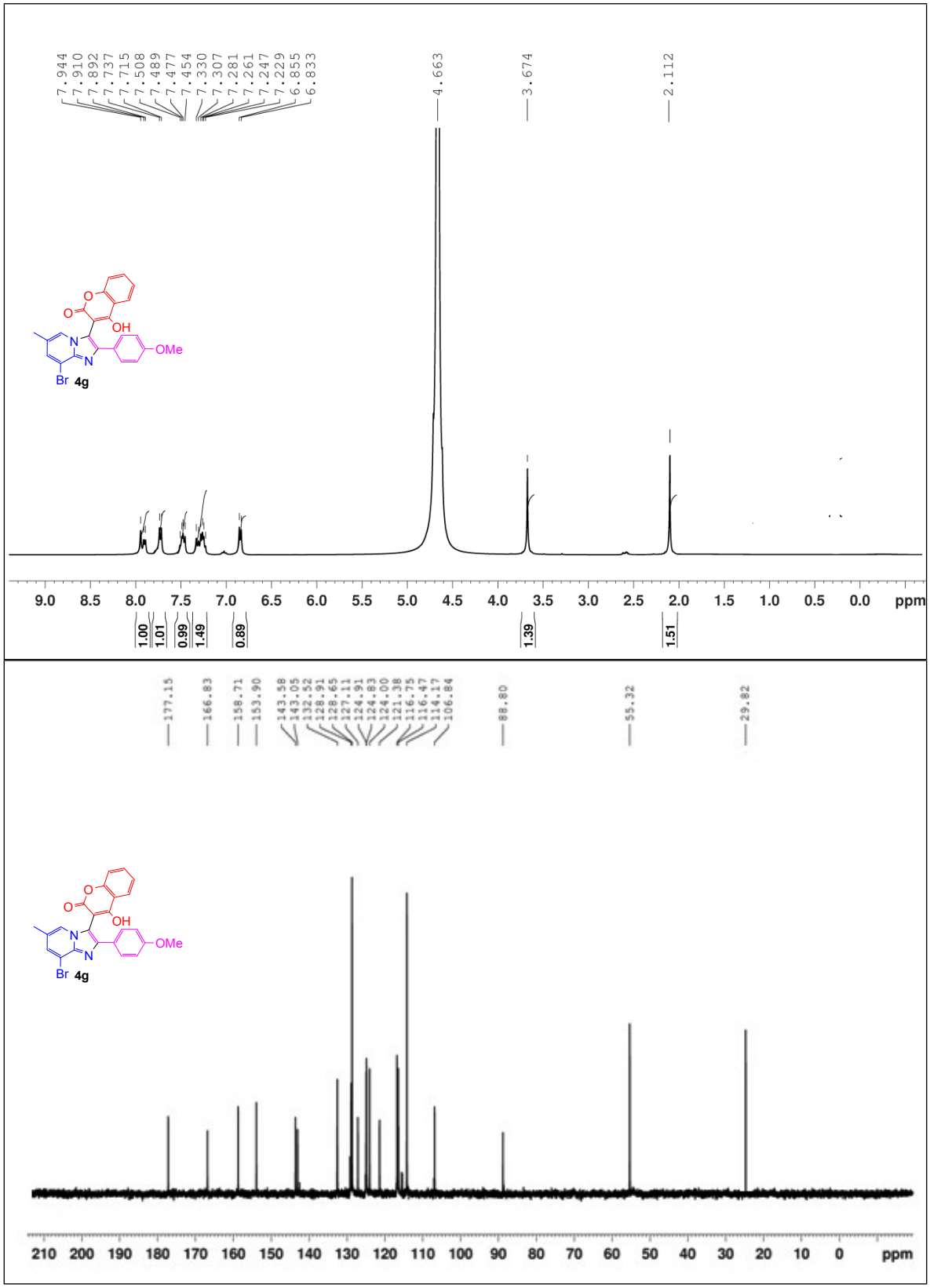
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4g
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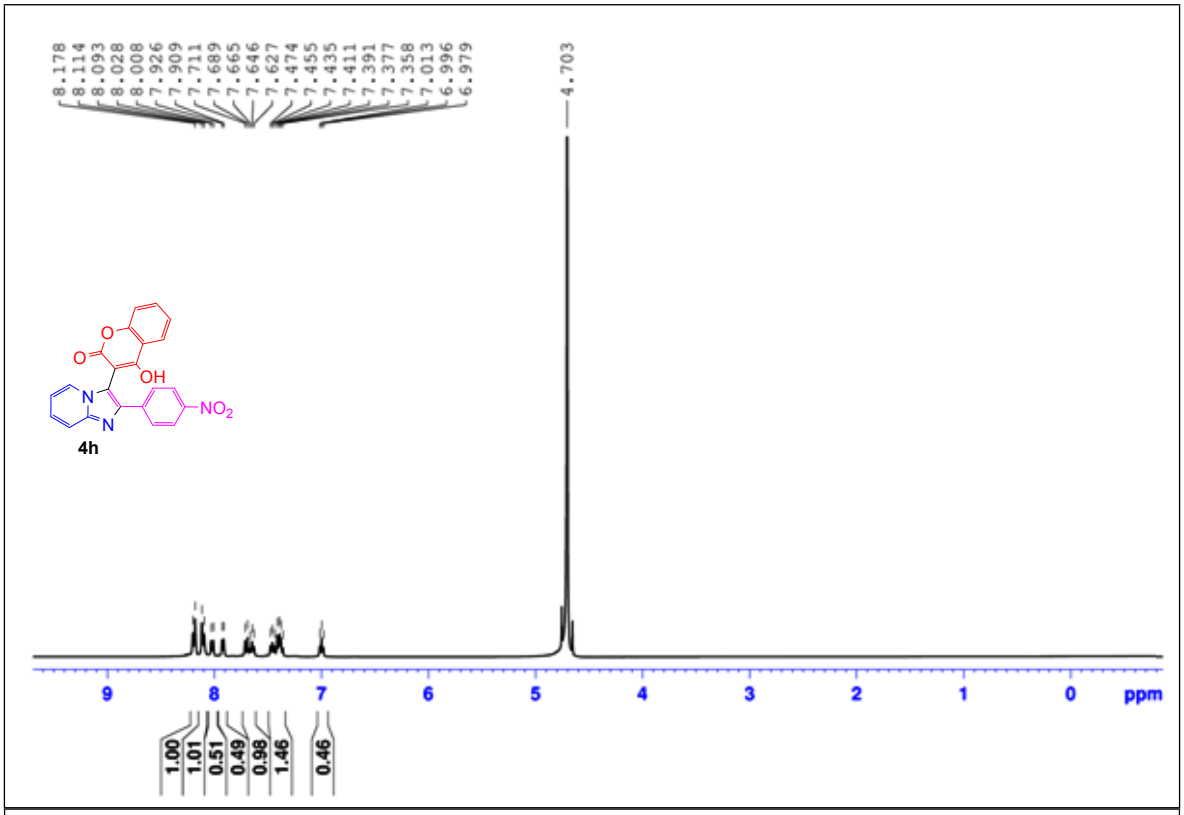
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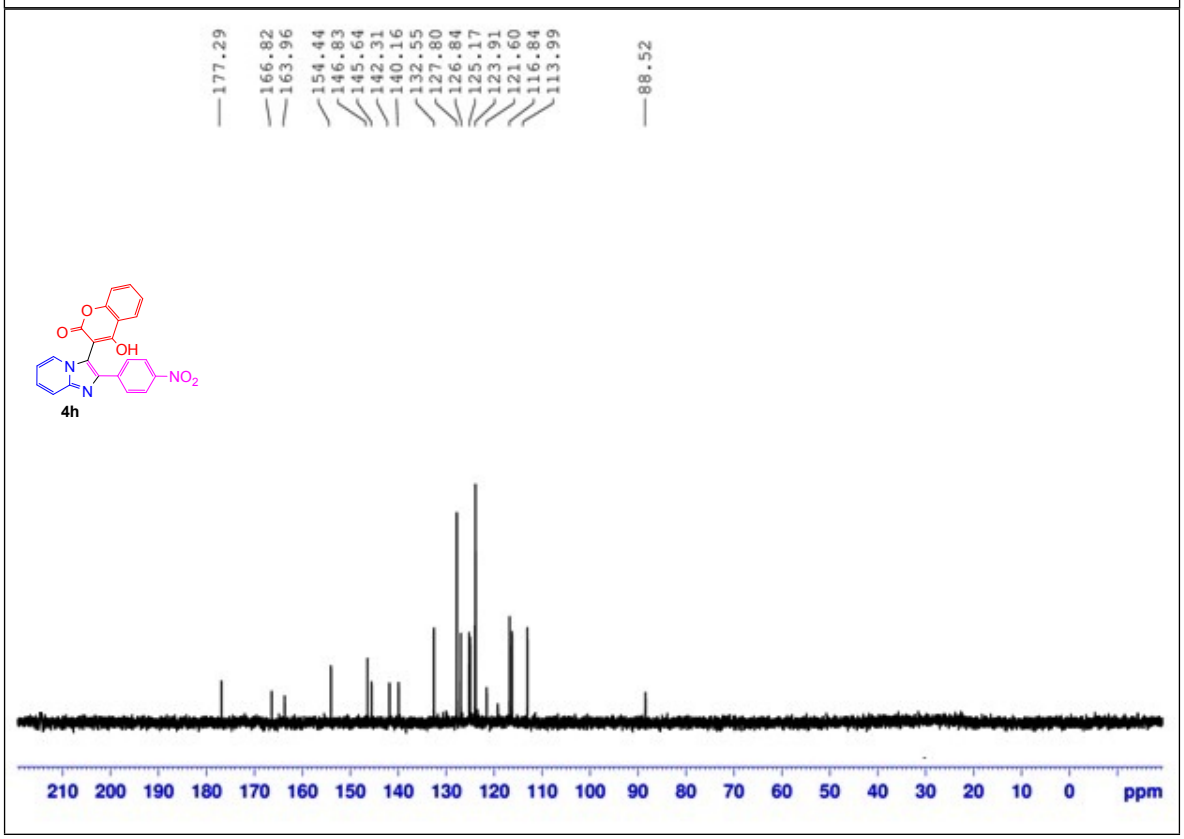
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NO2
4h
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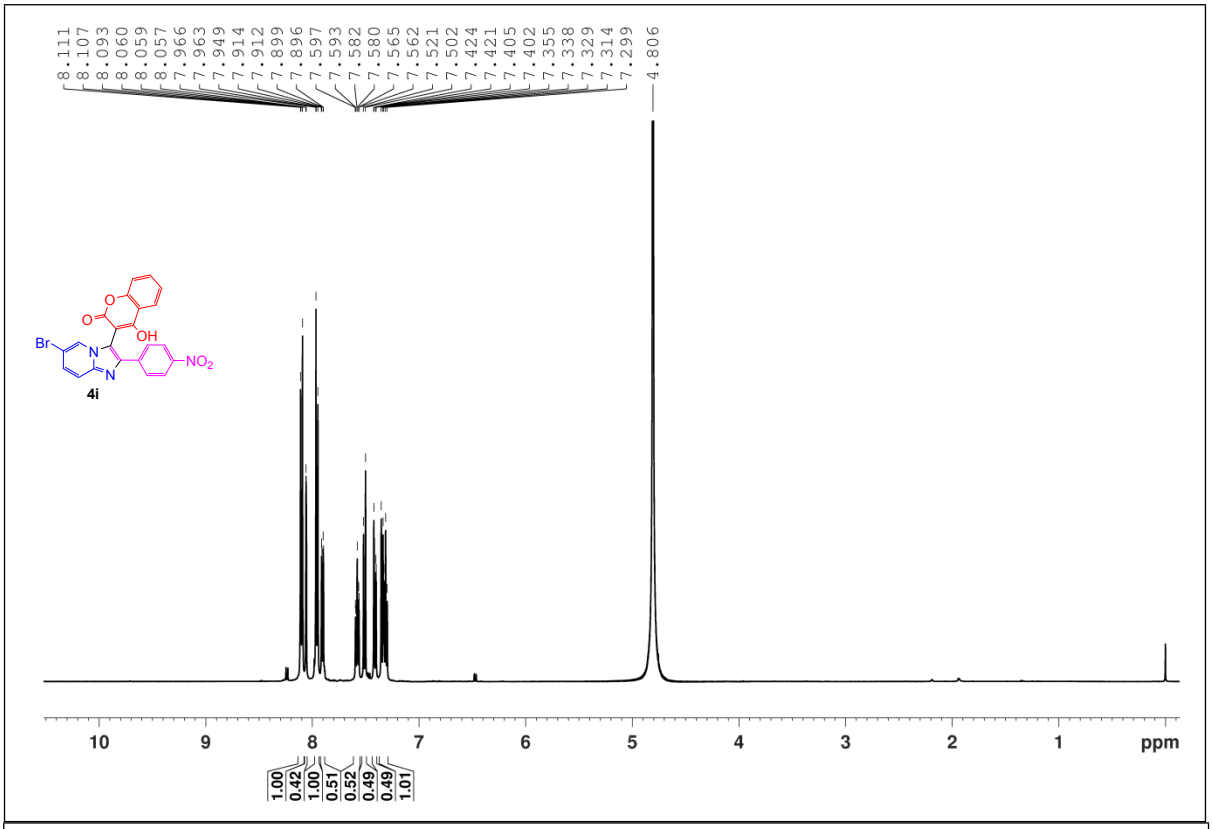
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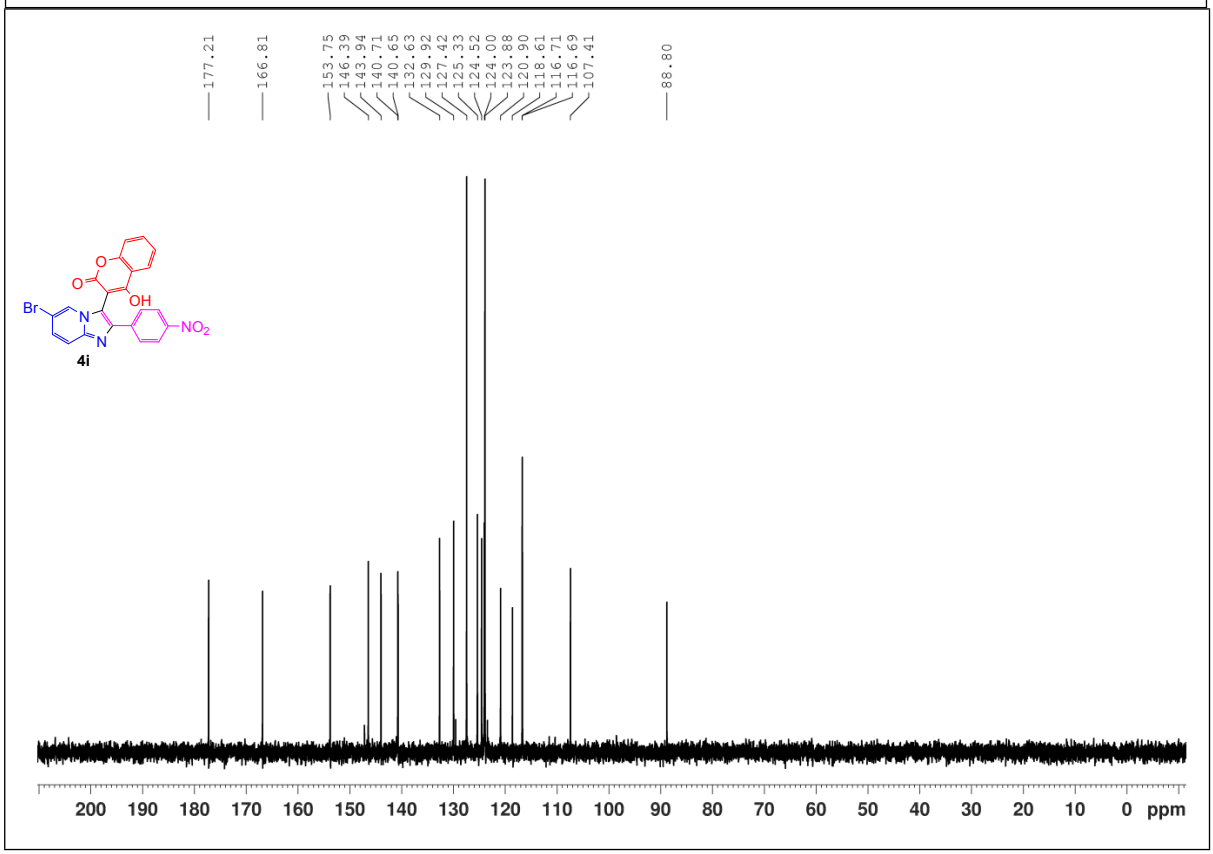
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Br
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4i
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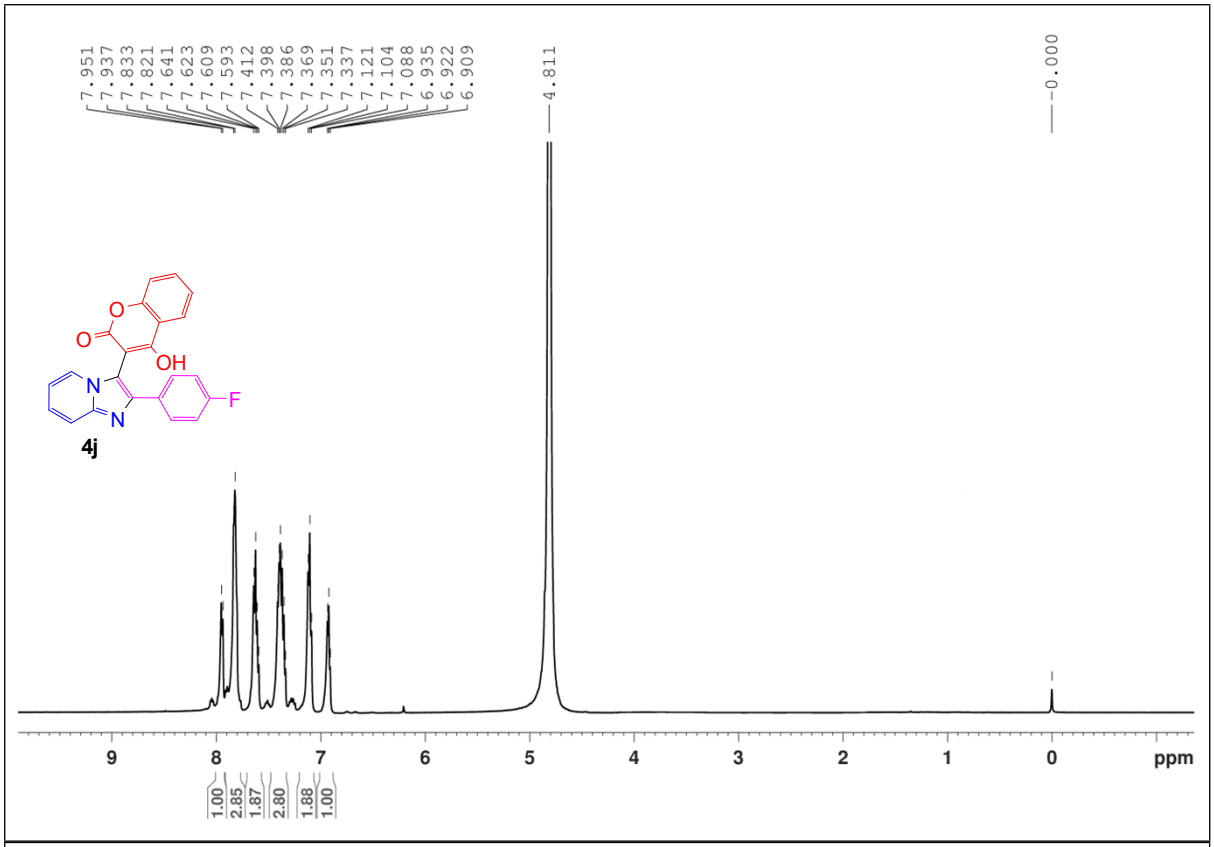
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7.351
7.337
7.121
7.104
7.088
6.935
6.922
6.909
4.811
0.000
O
O
OH
N
N
F
4j
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8
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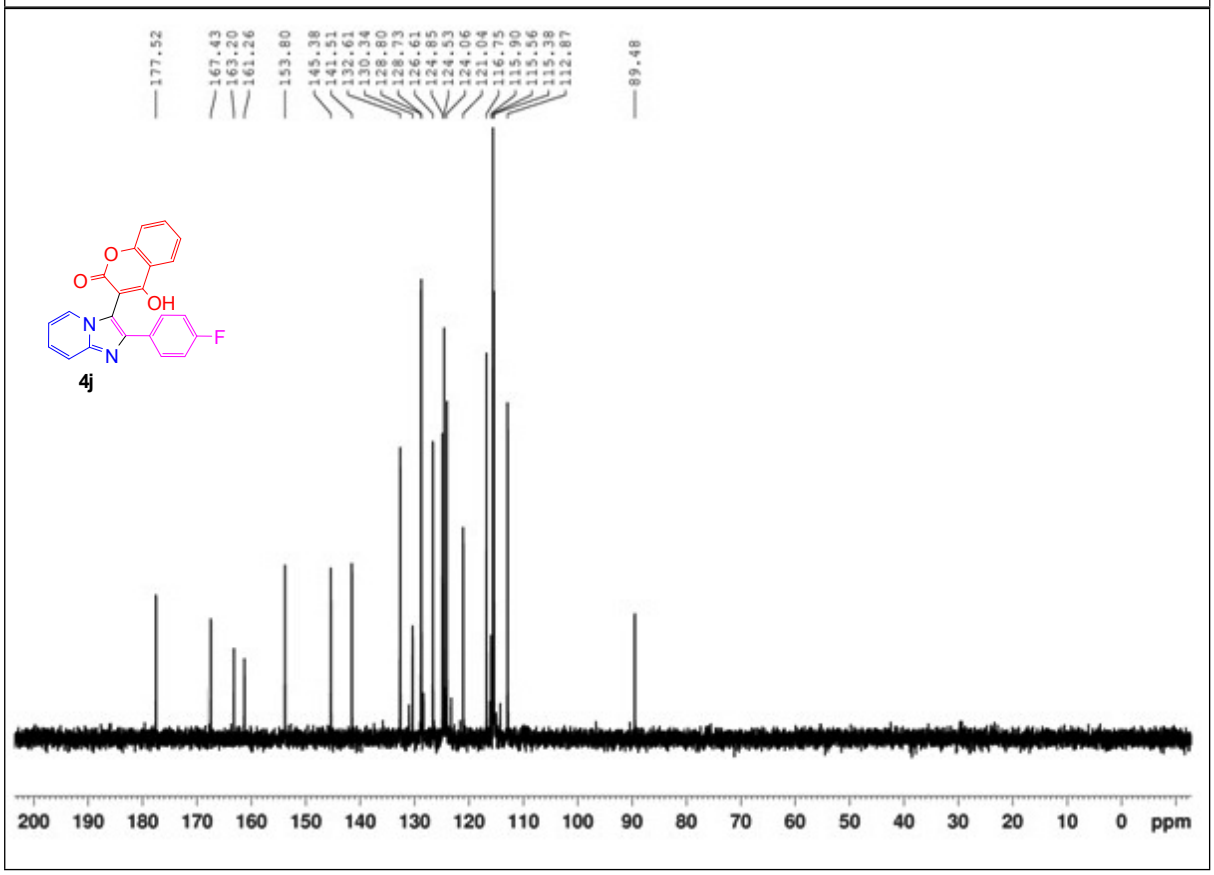
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124.53
124.06
121.04
116.75
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115.56
115.38
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OH
N
N
F
4j
200
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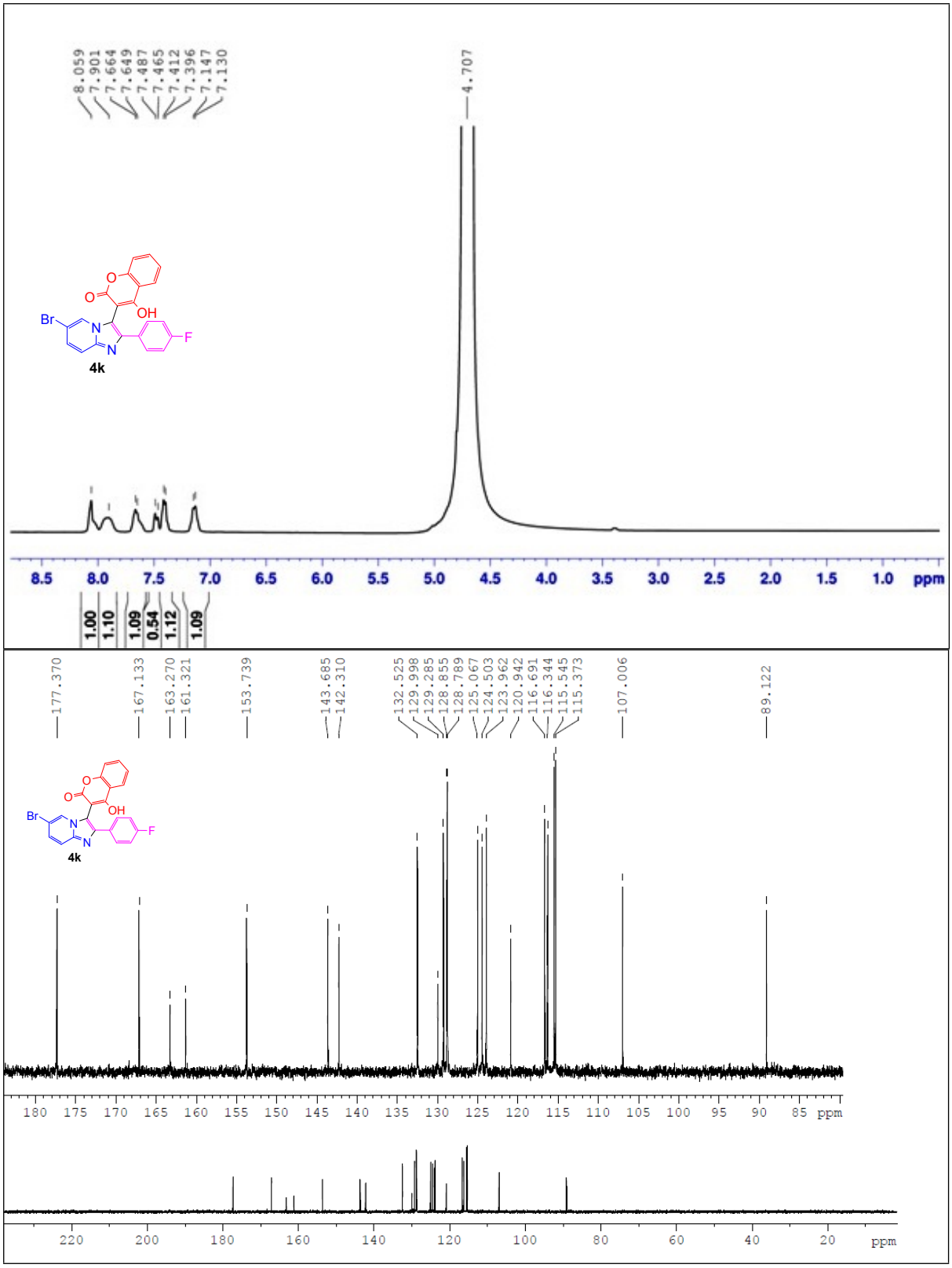
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OH
N
F
4k
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1.10
1.09
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1.09
177.370
167.133
163.270
161.321
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143.685
142.310
132.525
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129.285
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128.789
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123.962
120.942
116.691
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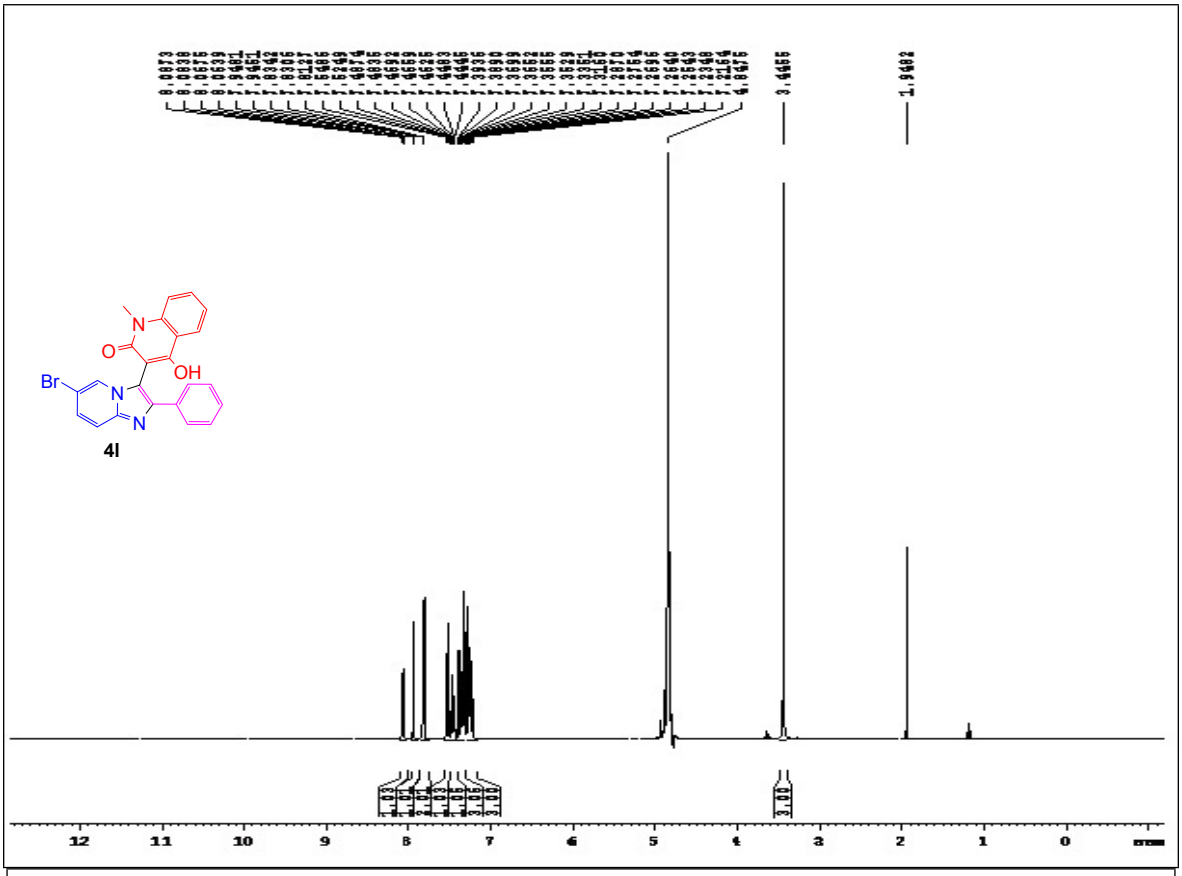
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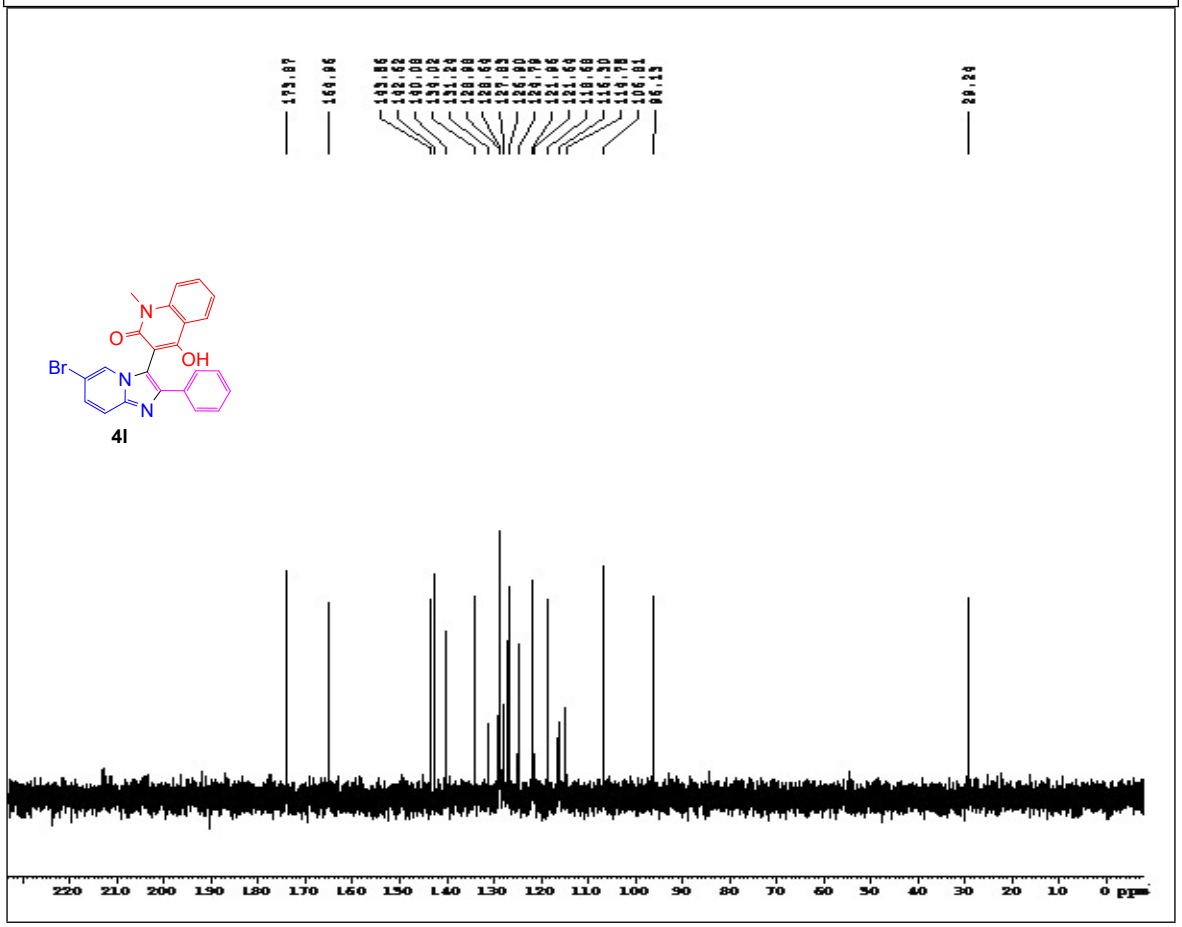
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Br
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4l
220
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150
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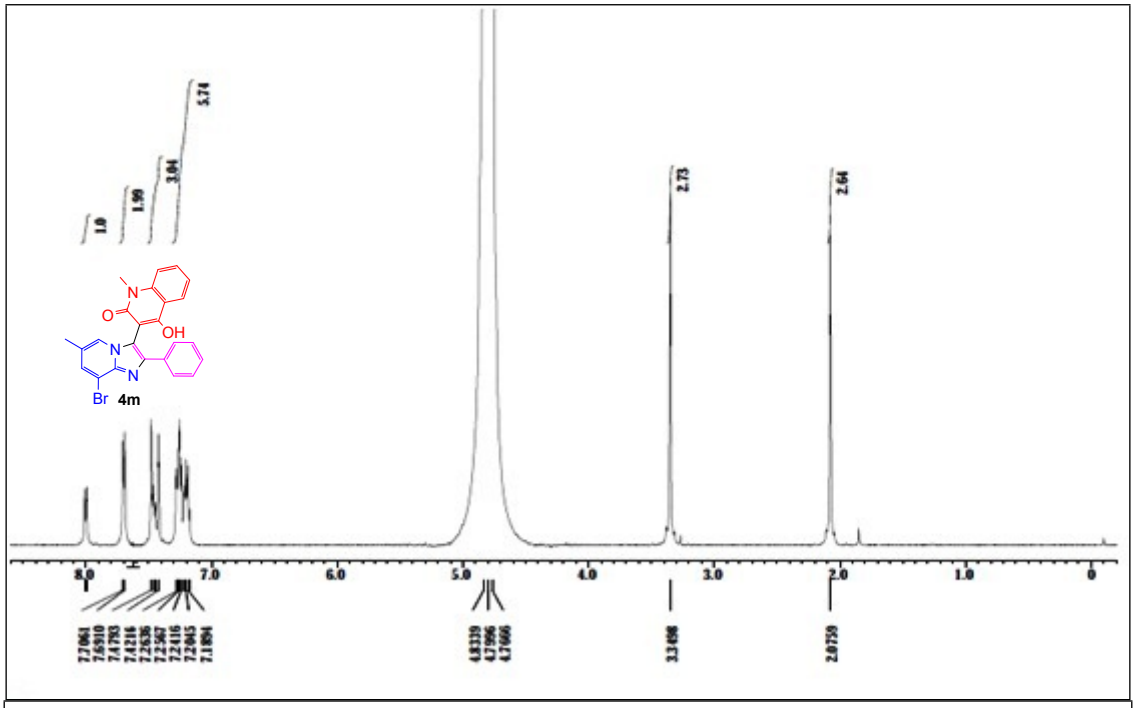
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5.74
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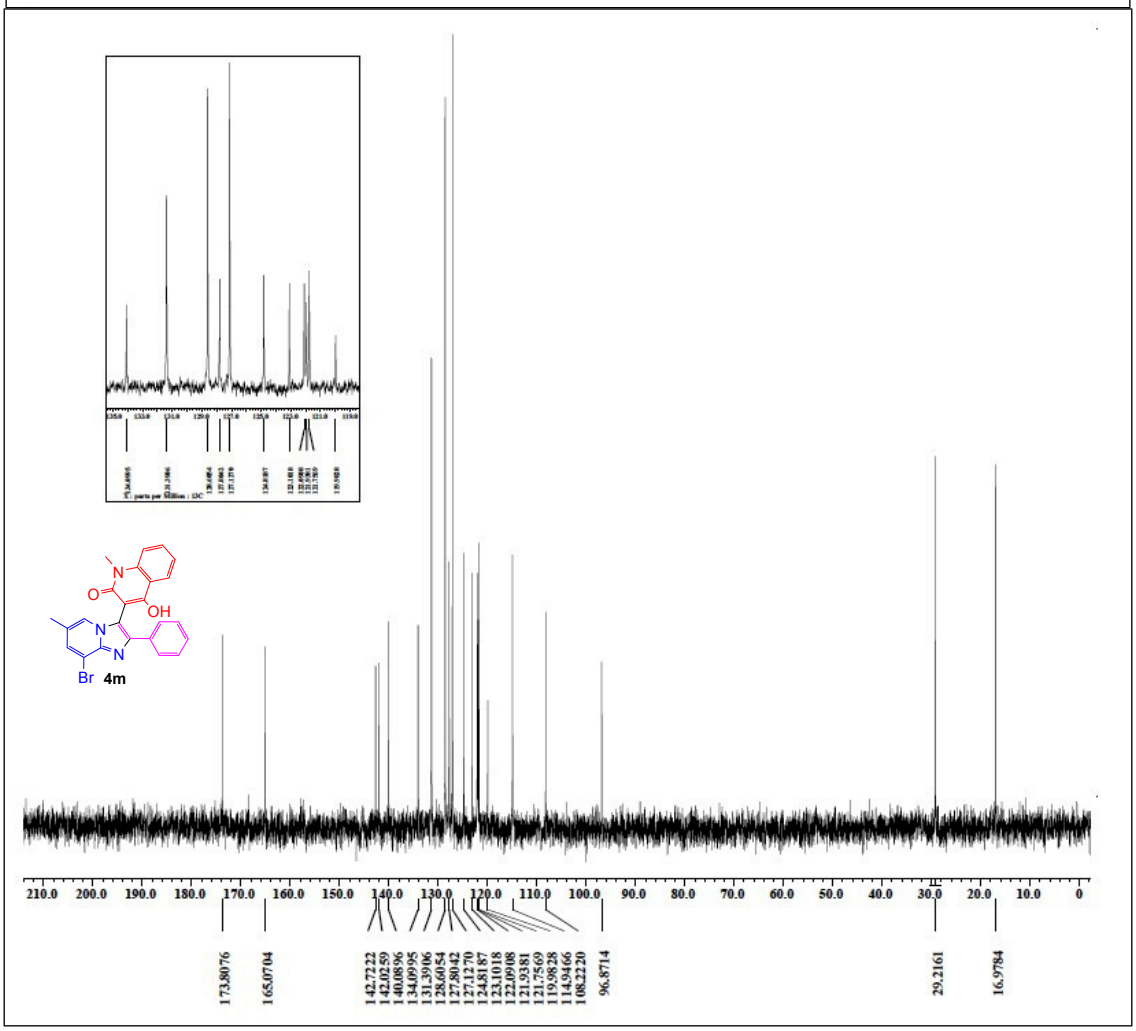
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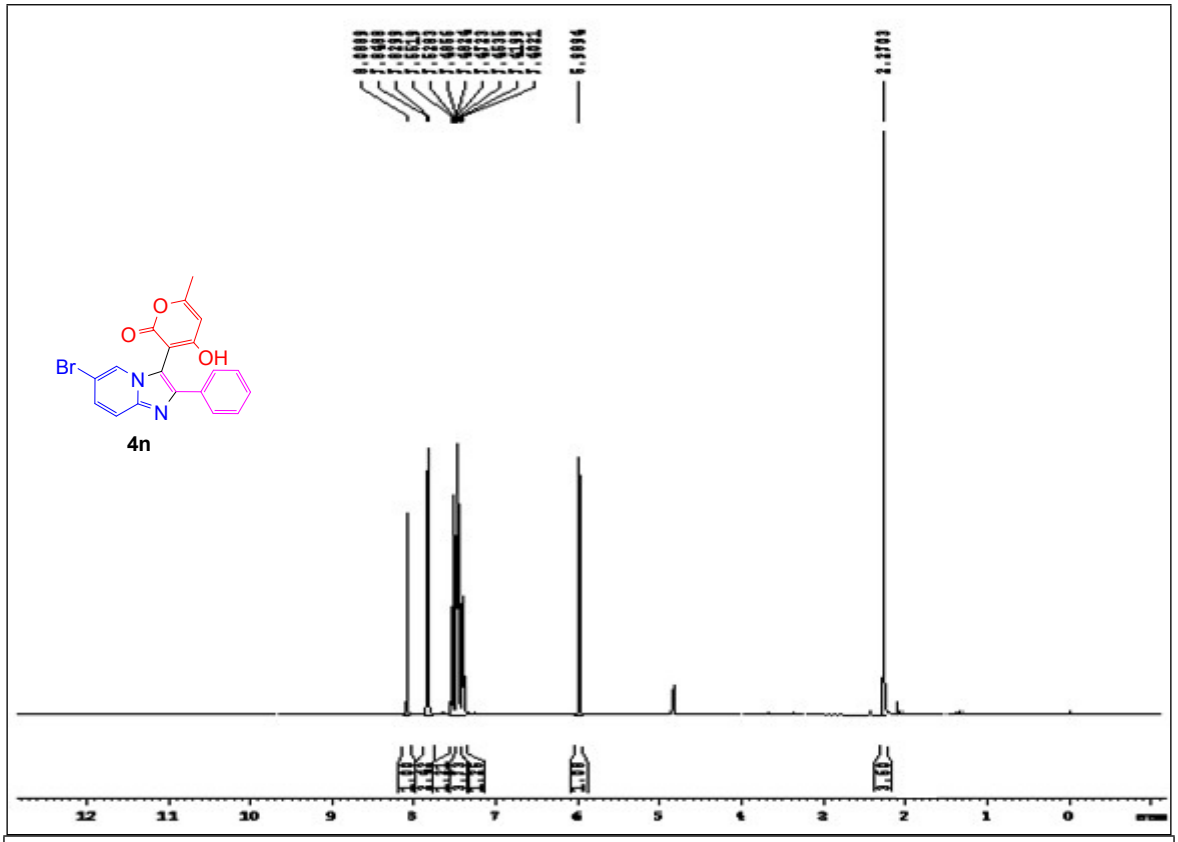

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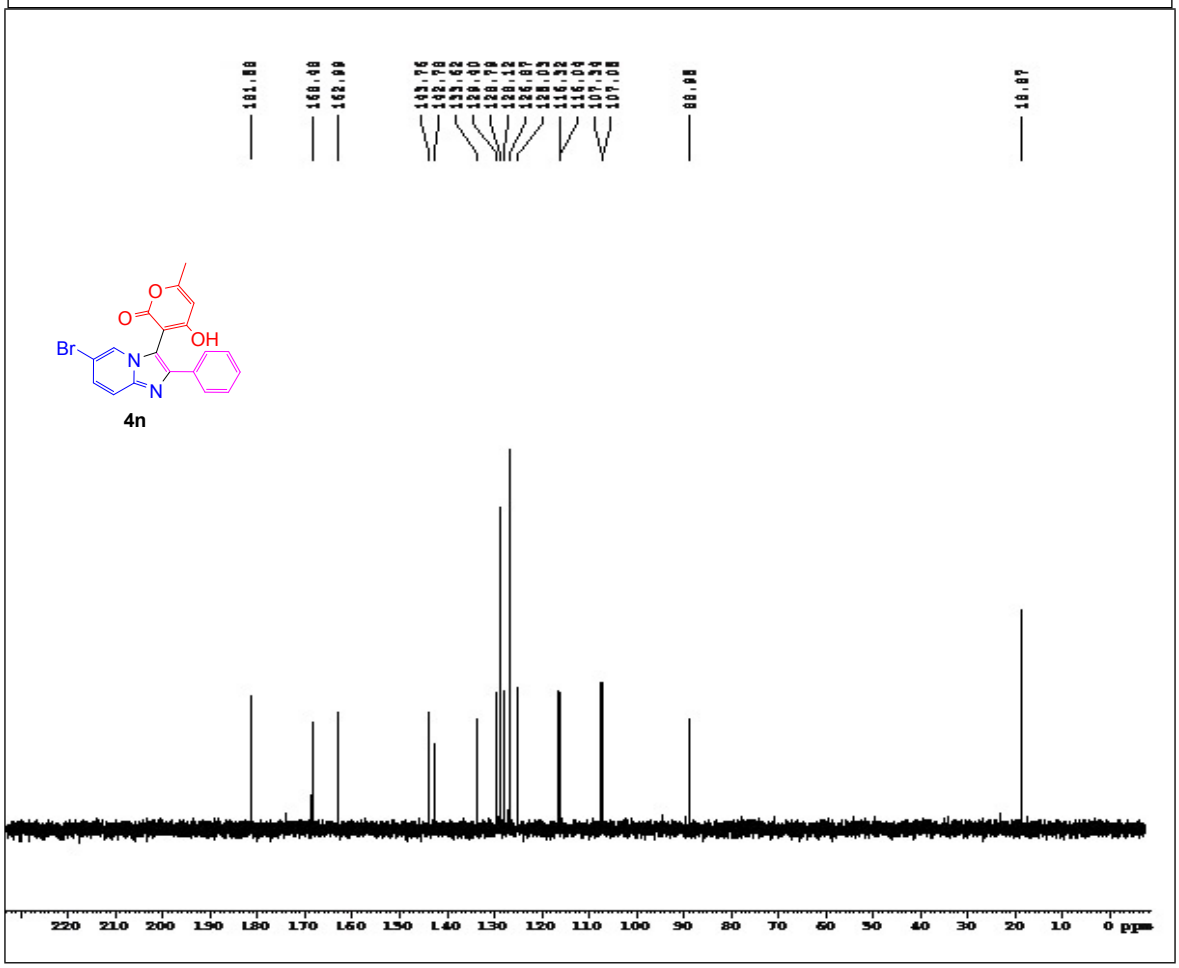

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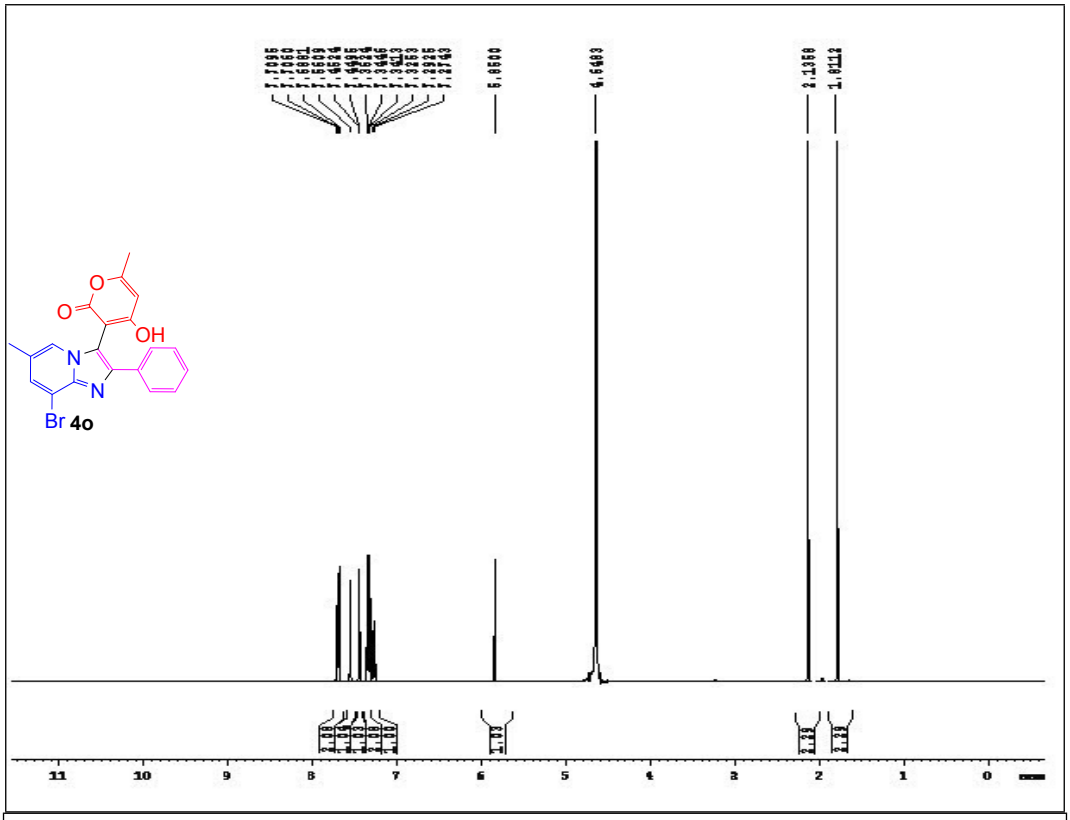
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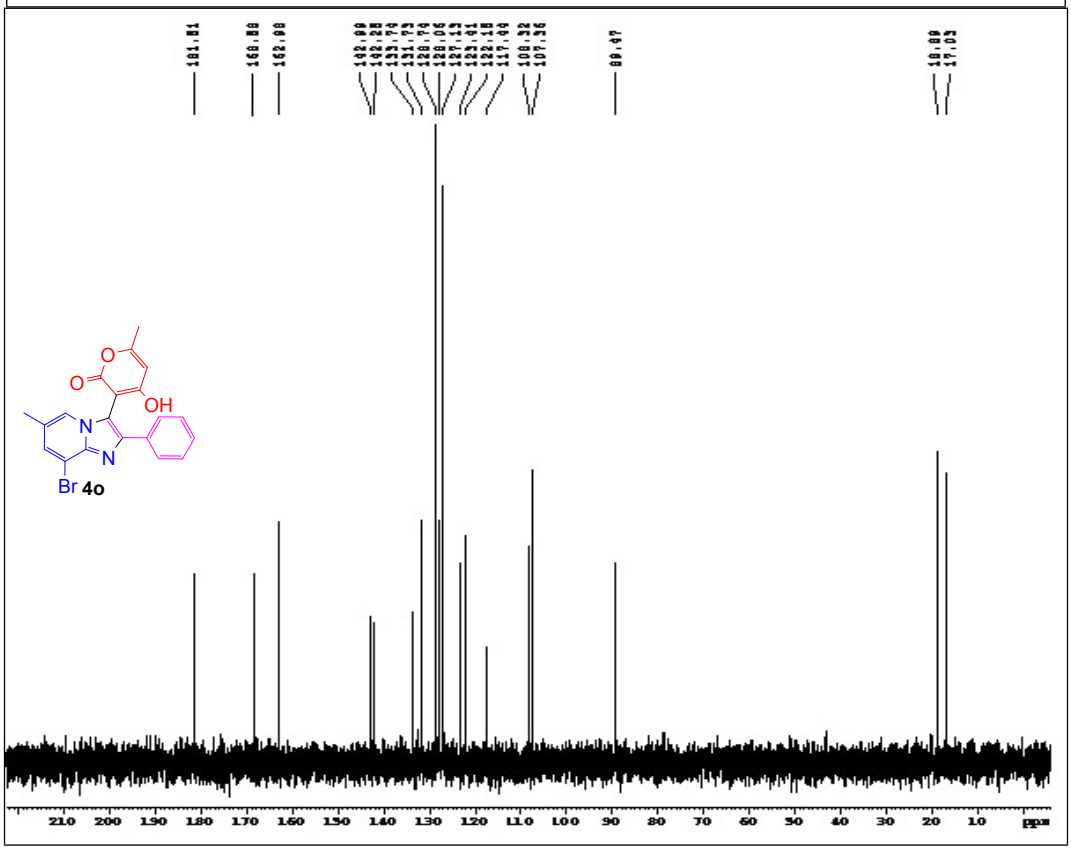
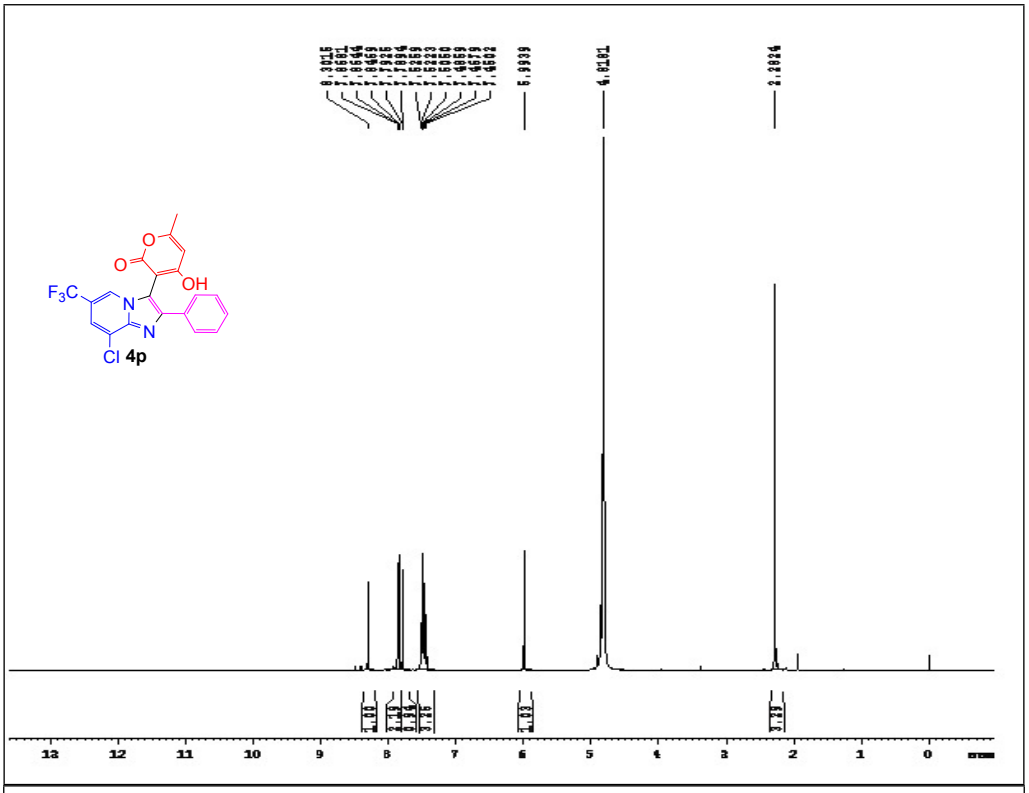
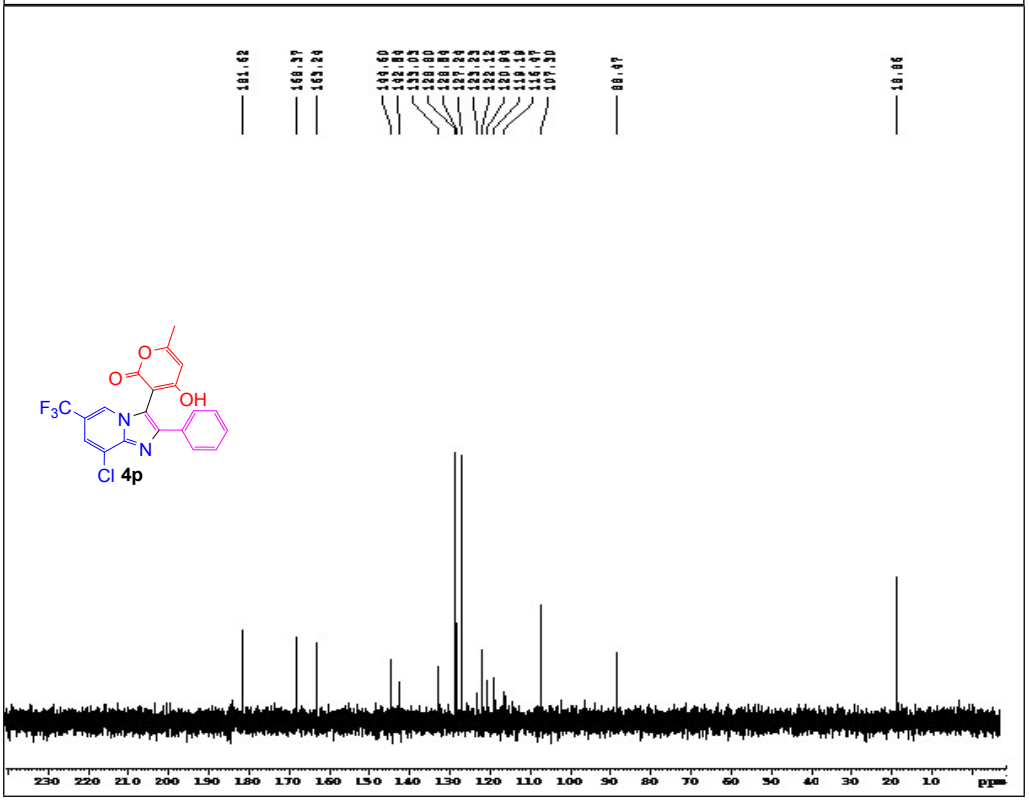
OH
N
N
Br
4o


O
O
OH
N
N
Br
4o


F3C
OH
Cl 4p


F3C
OH
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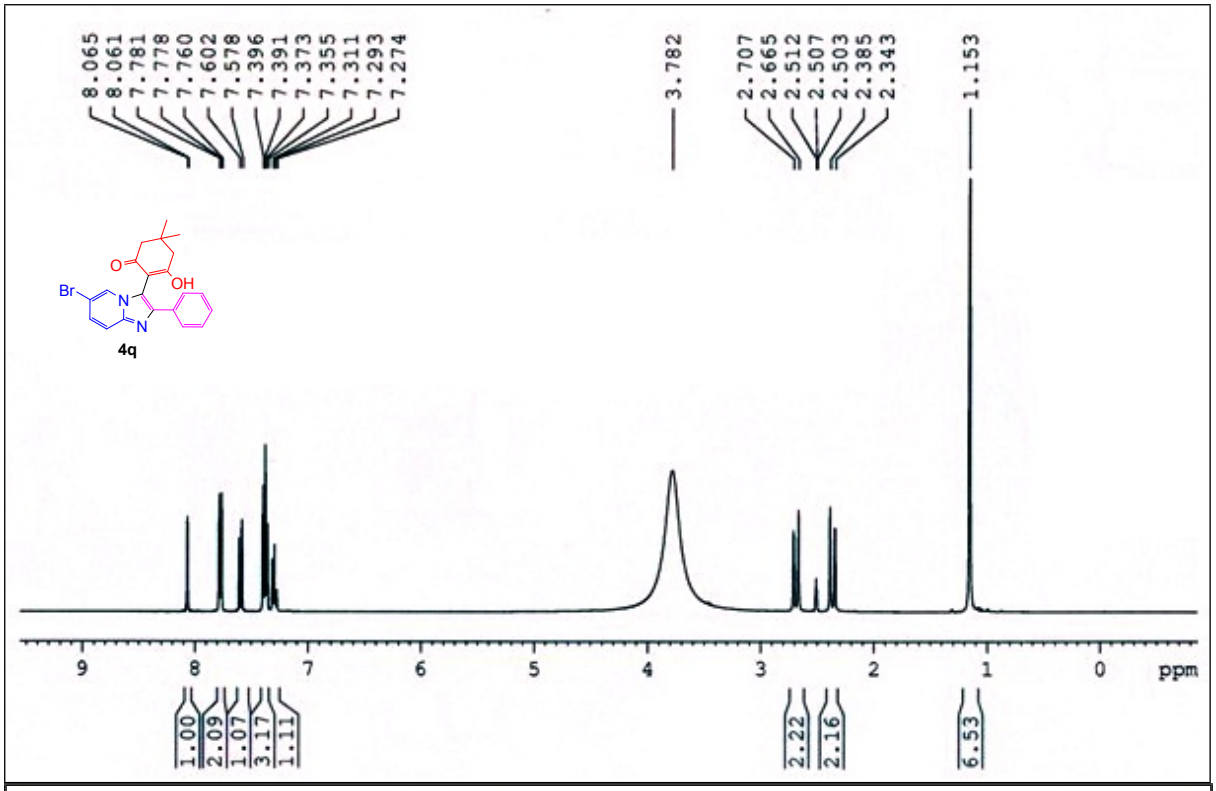
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8.061
7.781
7.778
7.760
7.602
7.578
7.396
7.391
7.373
7.355
7.311
7.293
7.274
3.782
2.707
2.665
2.512
2.507
2.503
2.385
2.343
1.153
Br
N
N
O
OH
4q
9
8
7
6
5
4
3
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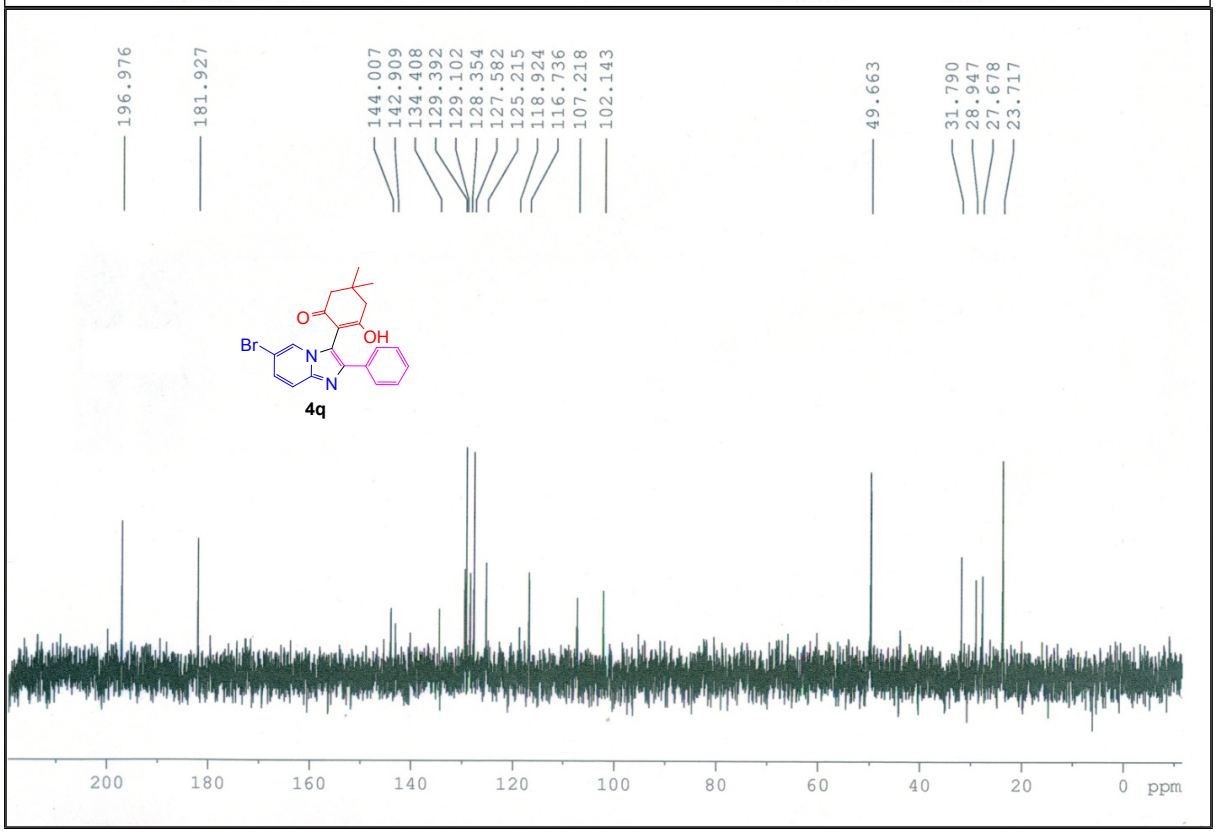
2.09
1.07
3.17
1.11
2.22
2.16
6.53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125.215
118.924
116.736
107.218
102.143
49.663
31.790
28.947
27.678
23.717
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N
O
OH
4q
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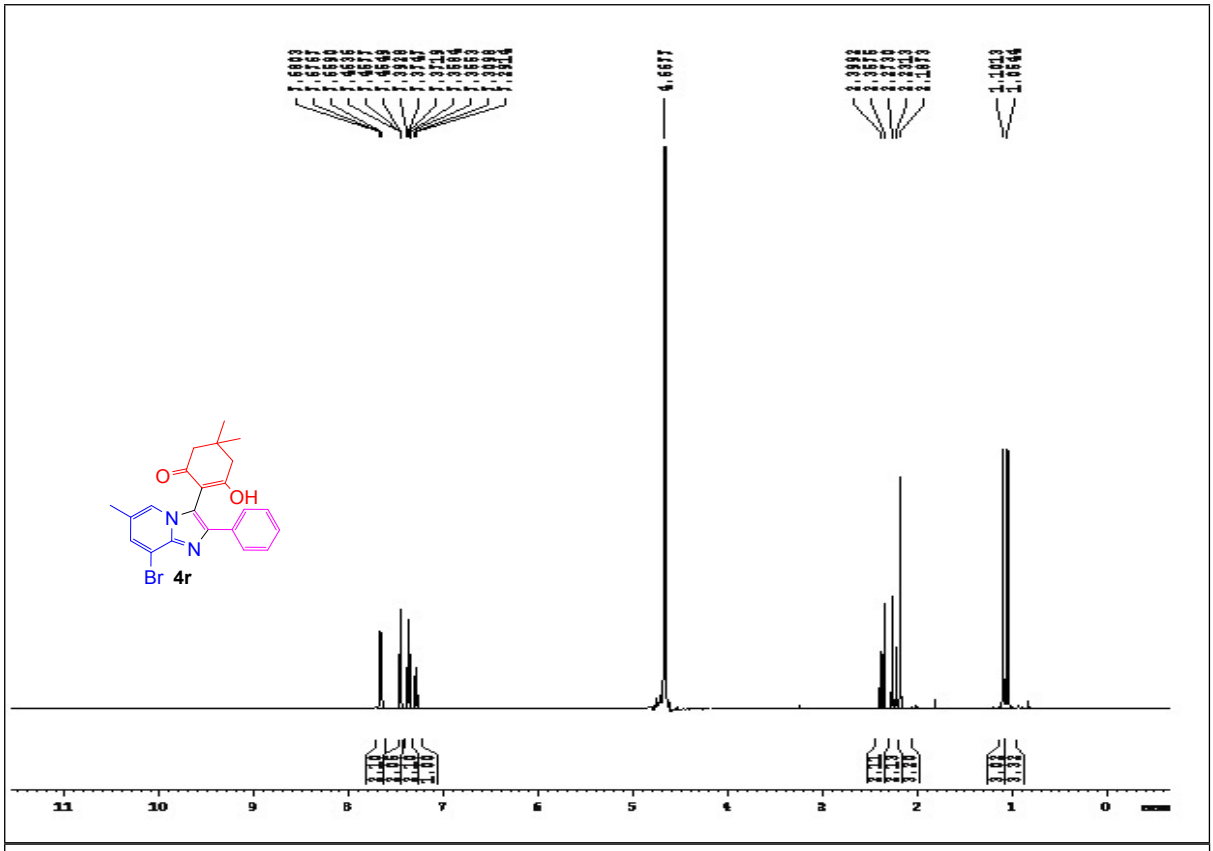
180
160
140
120
100
80
60
40
20
0
ppm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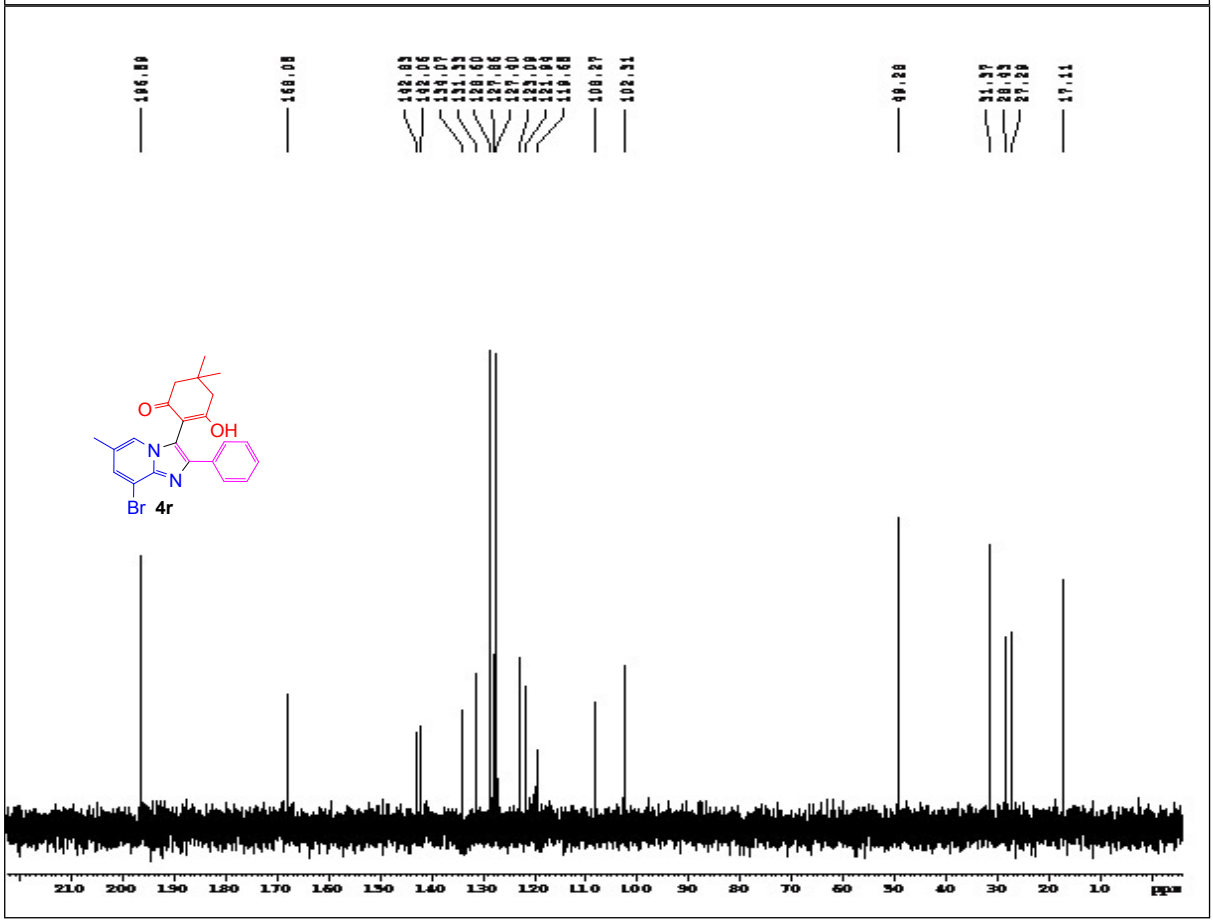
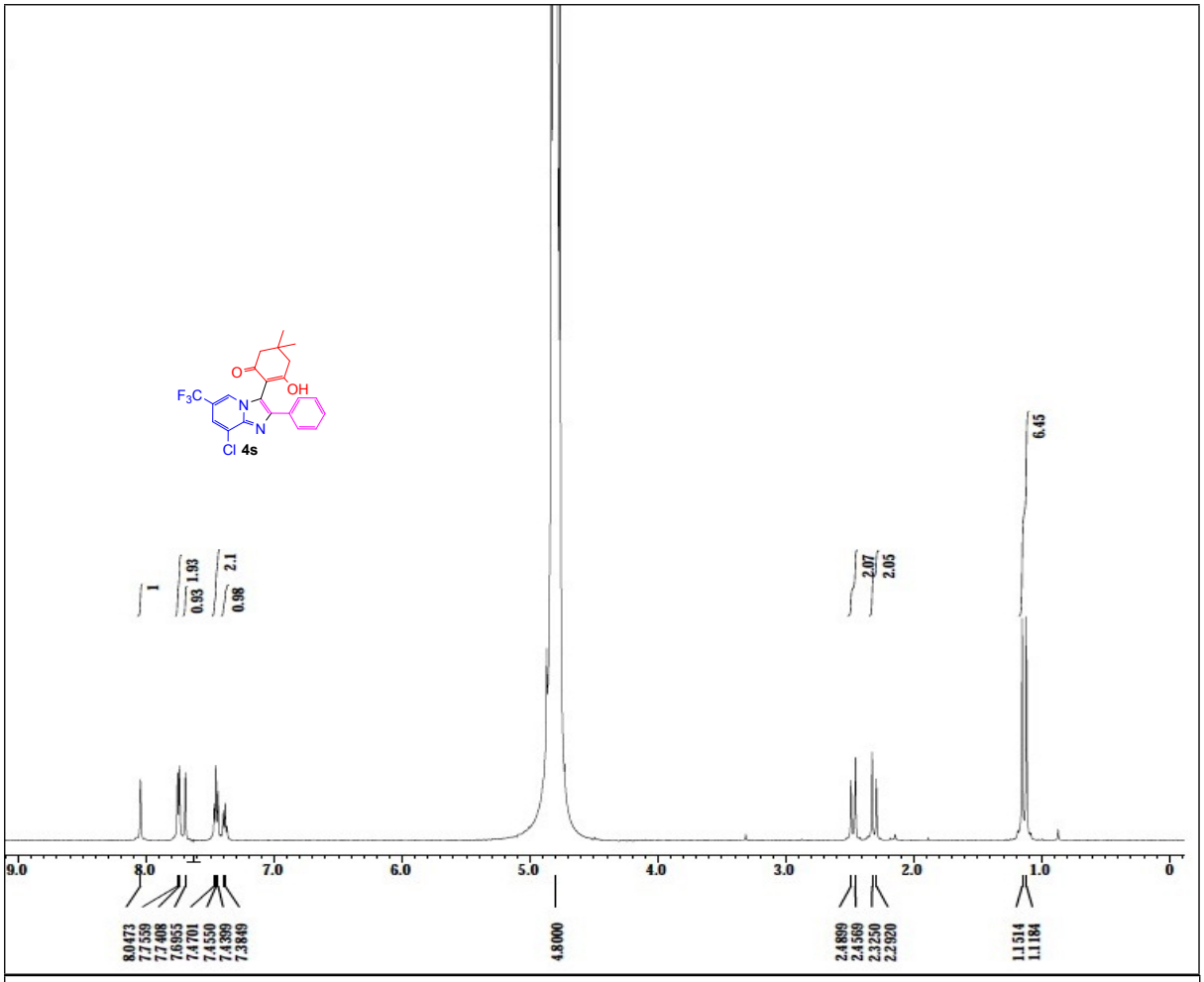
OH
N
N
Br 4r


O
OH
N
N
Br 4r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OH
Cl 4s
6.45
2.07
2.05
1
1.93
0.93
2.1
0.98
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.0473
7.7559
7.7408
7.6955
7.4701
7.4550
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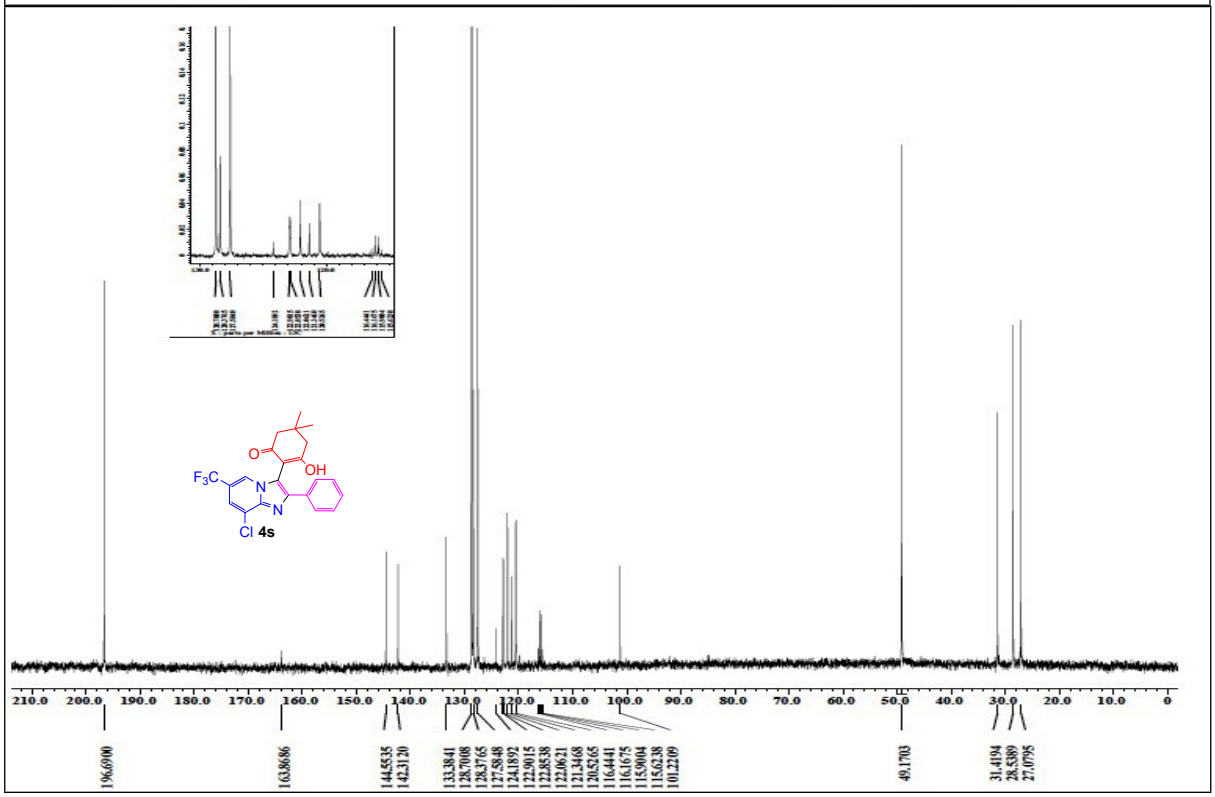
7.3849
4.8000
2.4899
2.4569
2.3250
2.2920
1.1514
1.1184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OH
Cl 4s
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200.0
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180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
196.6900
163.8686
144.5535
142.3120
133.3841
128.7008
128.3765
127.5848
124.1892
122.9015
122.8538
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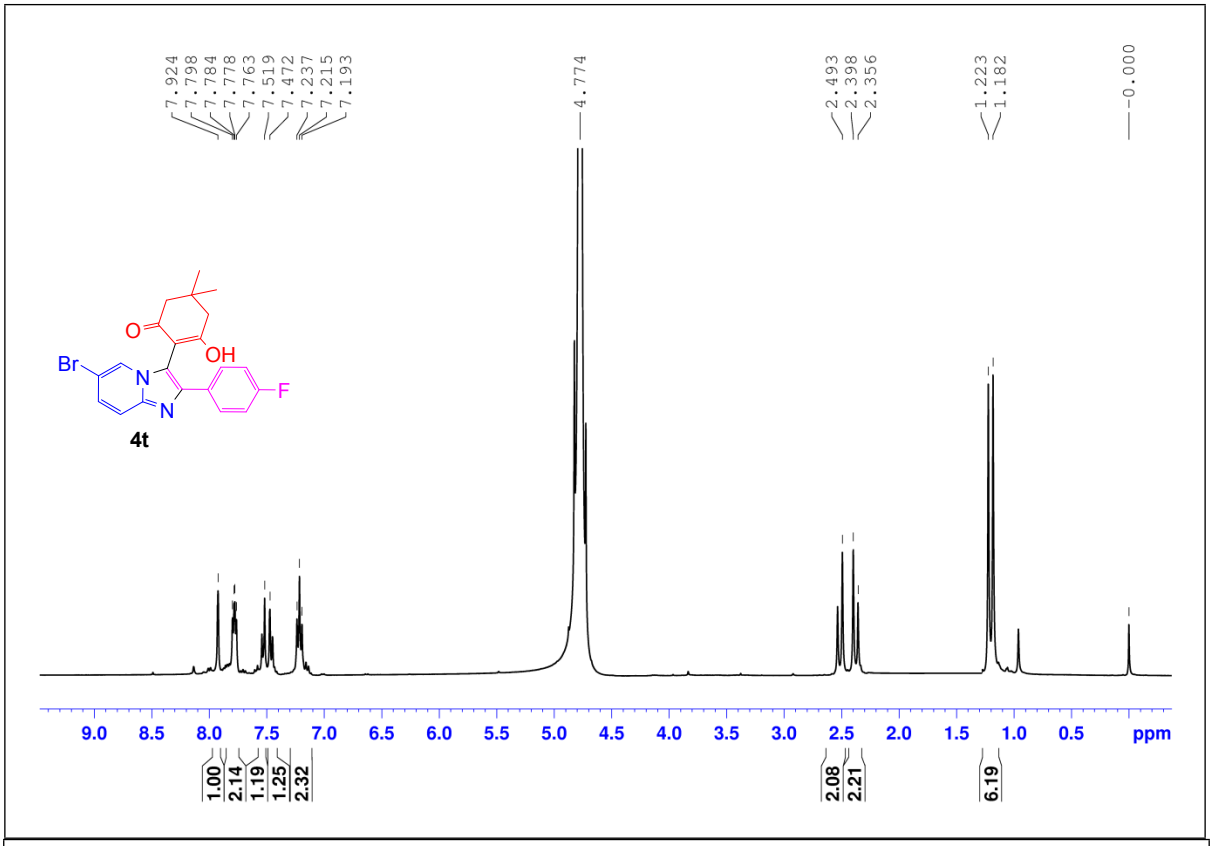
27.0795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7.519
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7.237
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7.193
4.774
2.493
2.398
2.356
1.223
1.182
0.000
O
OH
Br
N
N
F
4t
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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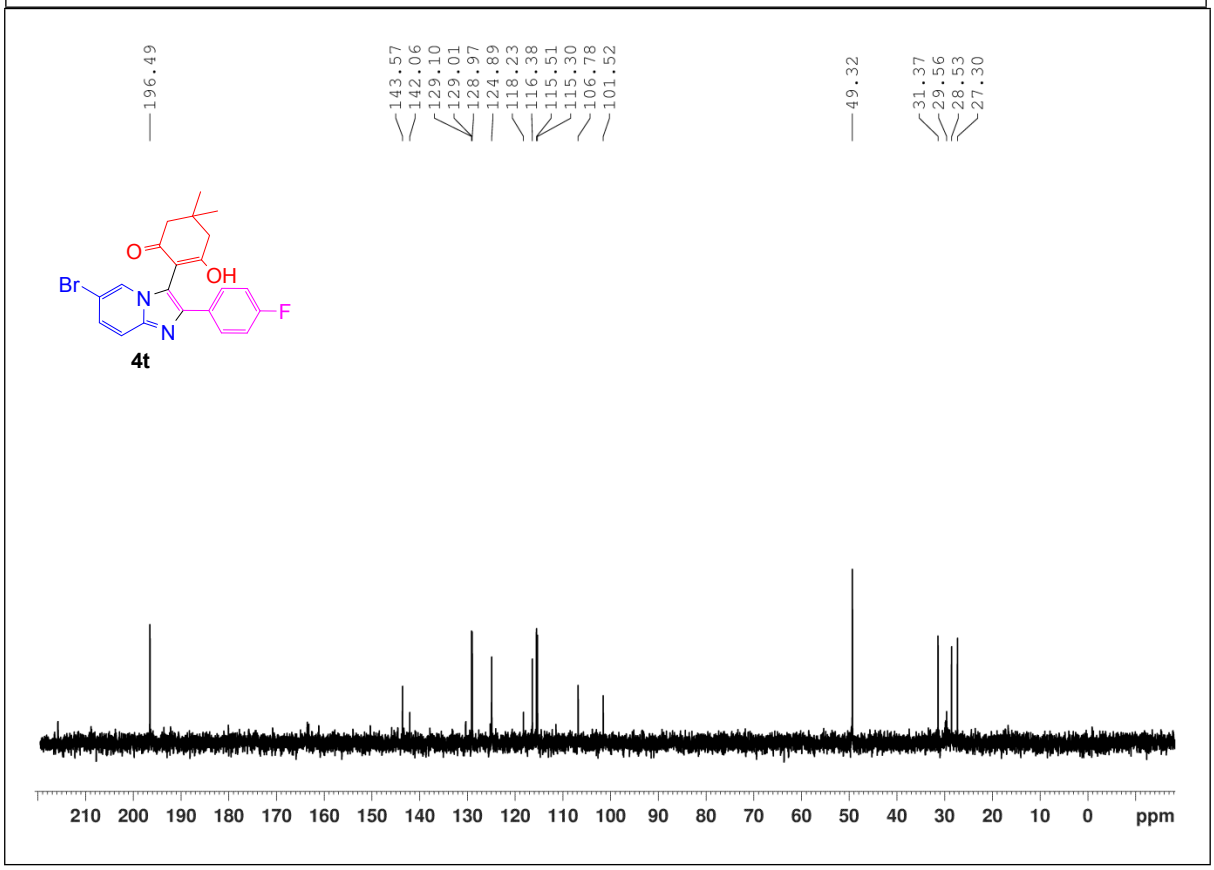
1.19
1.25
2.32
2.08
2.21
6.19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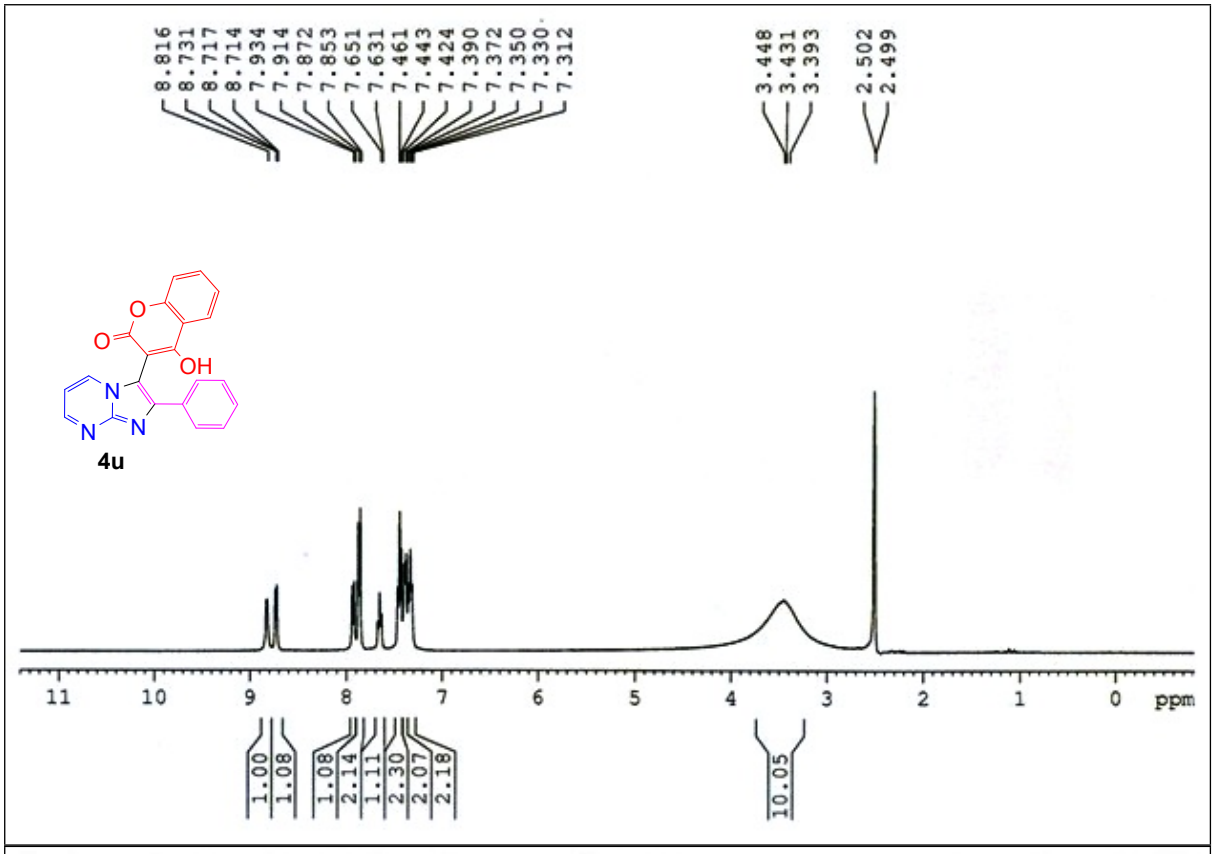
143.57
142.06
129.10
129.01
128.97
124.89
118.23
116.38
115.51
115.30
106.78
101.52
49.32
31.37
29.56
28.53
27.30
O
OH
Br
N
N
F
4t
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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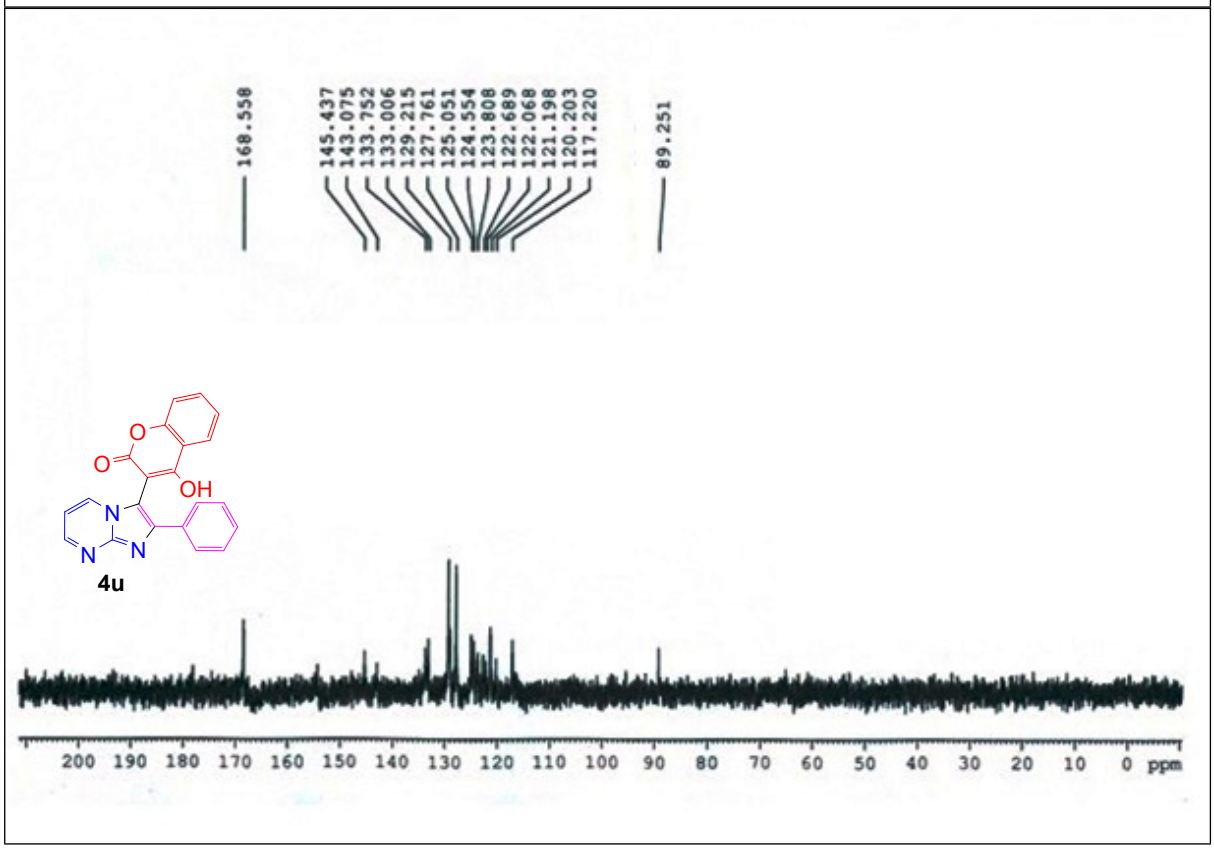
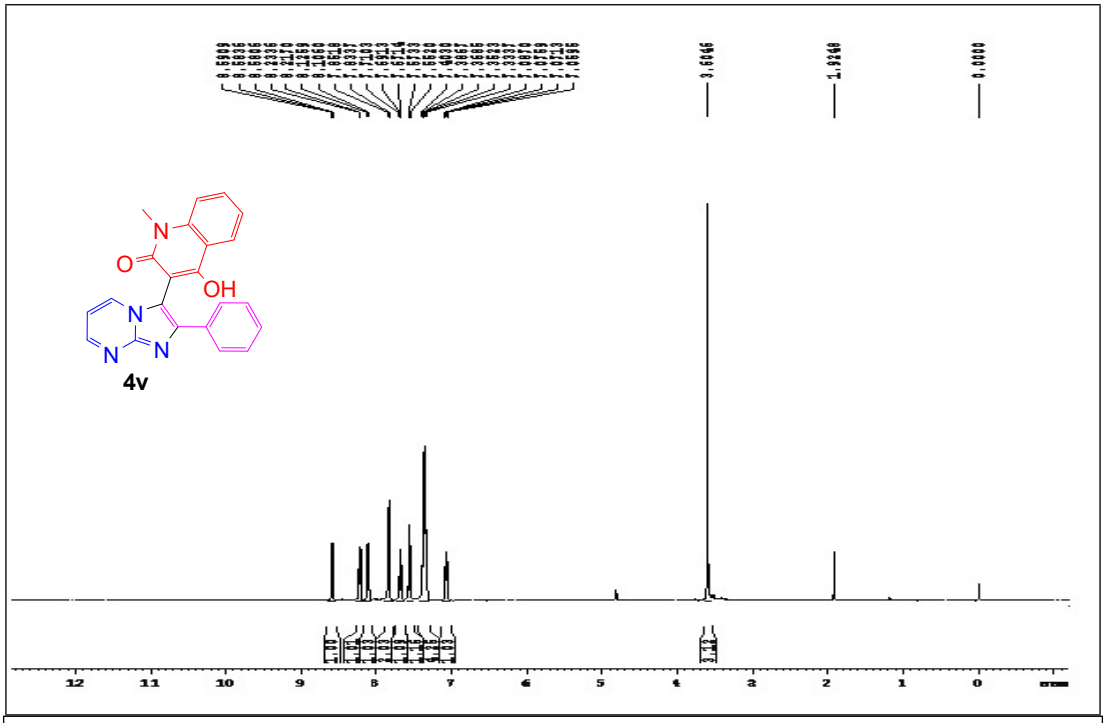
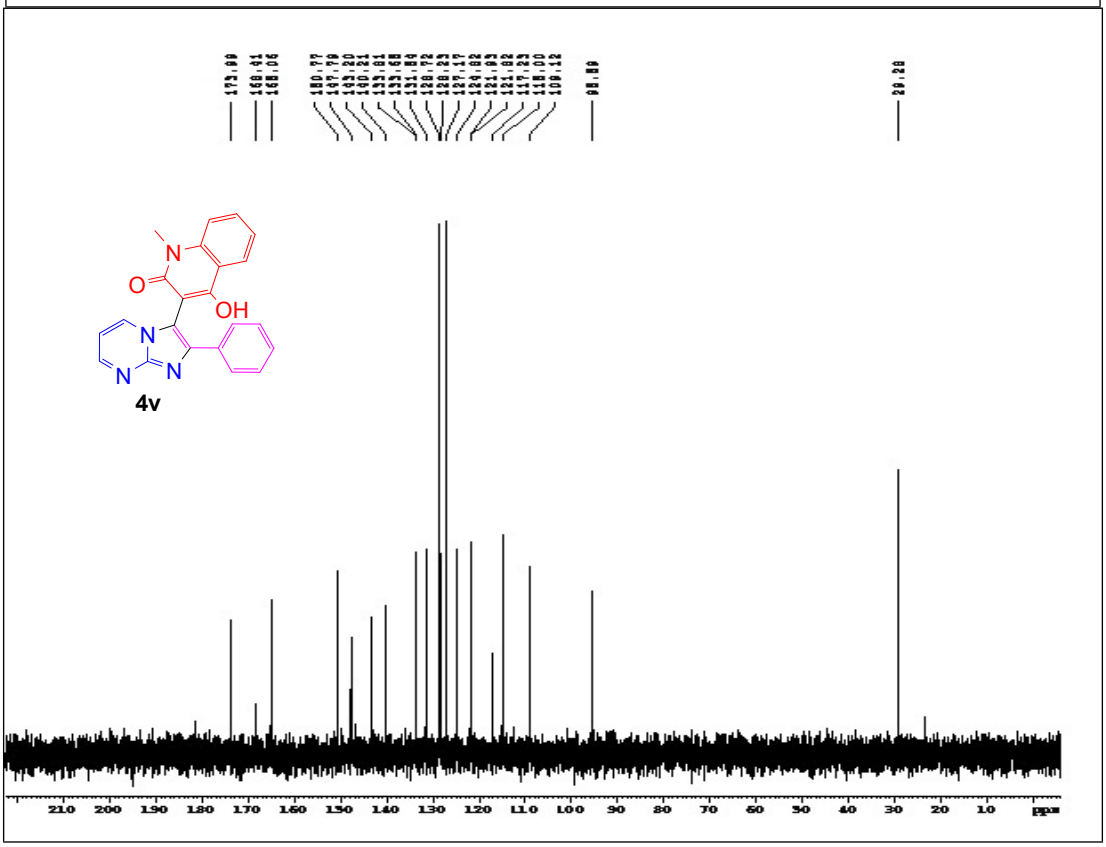
8.731
8.717
8.714
7.934
7.914
7.872
7.853
7.651
7.631
7.461
7.443
7.424
7.390
7.372
7.350
7.330
7.312
3.448
3.431
3.393
2.502
2.499
O
O
OH
N
N
N
4u
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.08
1.08
2.14
1.11
2.30
2.07
2.18
10.05


168.558
145.437
143.075
133.752
133.006
129.215
127.761
125.051
124.554
123.808
122.689
122.068
121.198
120.203
117.220
89.251
O
O
OH
N
N
N
4u
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


4v


4v