Electronic Supplementary Material (ESI) for RSC Advances.

Supporting information

"On-water" synthesis of novel trisubstituted 1, 3-thiazoles via microwave-assisted catalyst-free domino reactions

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Table of contents

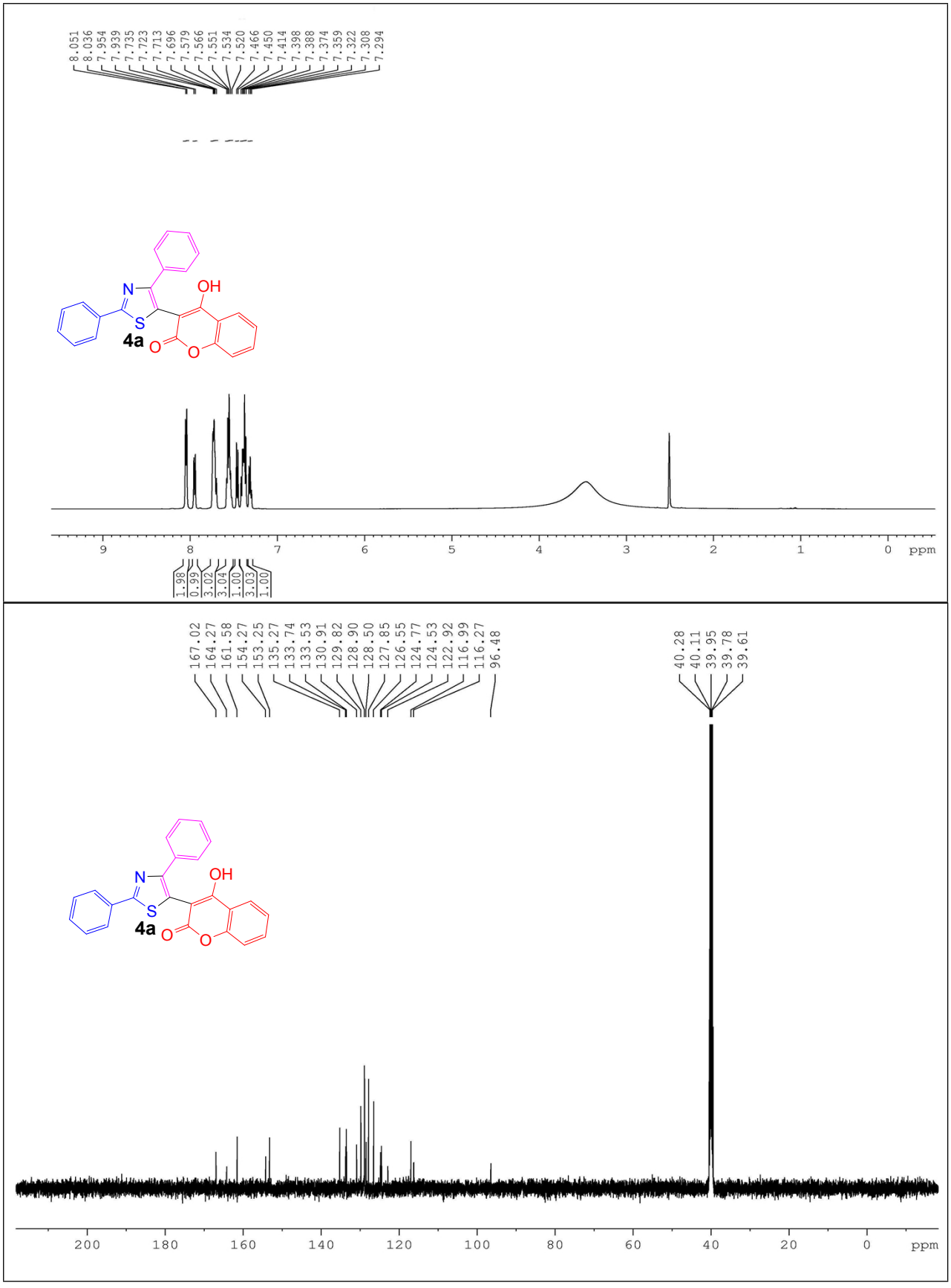
1. General information.

All starting materials were purchased from Sigma Aldrich and 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 $CDCl_3$ 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 either Elementar Vario EL III or Perkin-Elmer 2400 II elemental analyzers. Microwave irradiation was carried out with Initiator 2.5 Microwave Synthesizers from Biotage. Melting points were recorded by using SRS EZ-Melt automated melting point apparatus by capillary methods and uncorrected.

2. General procedure for the synthesis of 1, 3-thiazole analogues (4).

A mixture of arylglyoxal monohydrate **1** (1 mmol), 1, 3-dicarbonyl derivatives **2** (1 mmol), and thioamide derivatives **3** (1 mmol) in 3 mL H_2O was introduced in a 2-5 mL Initiator reaction vial, the mixture was irradiated for 15 minutes at 130 ^{o}C; The reaction mixture was then cooled to room temperature and the solid was filtered off, and was washed with 95% EtOH to yield the pure products **4**. Some of the products **4n**-**4p**, **4q, 4r** and **4t** were purified by column chromatography on a silica gel column using EtOAc–hexane mixture as the eluent.

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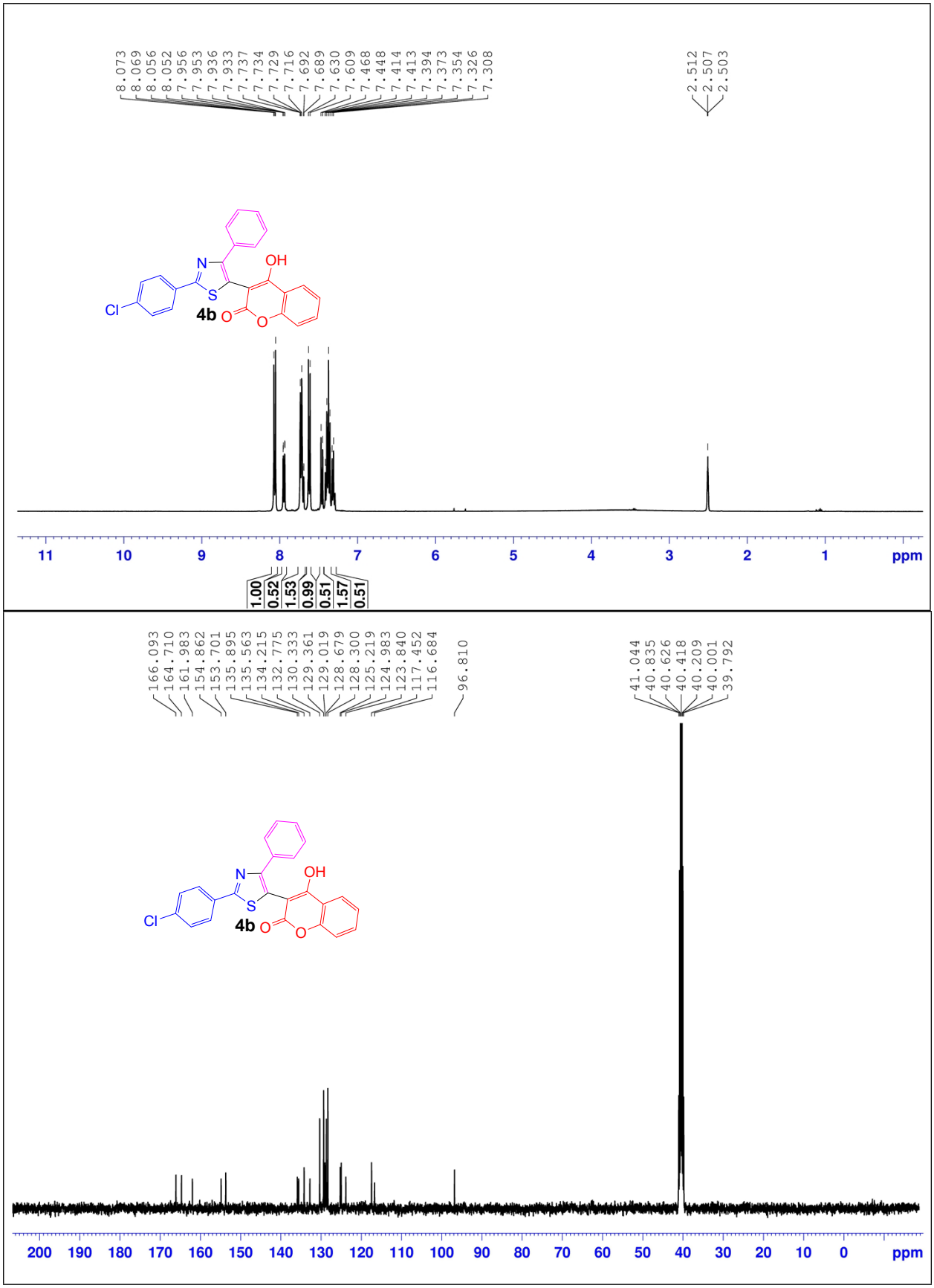
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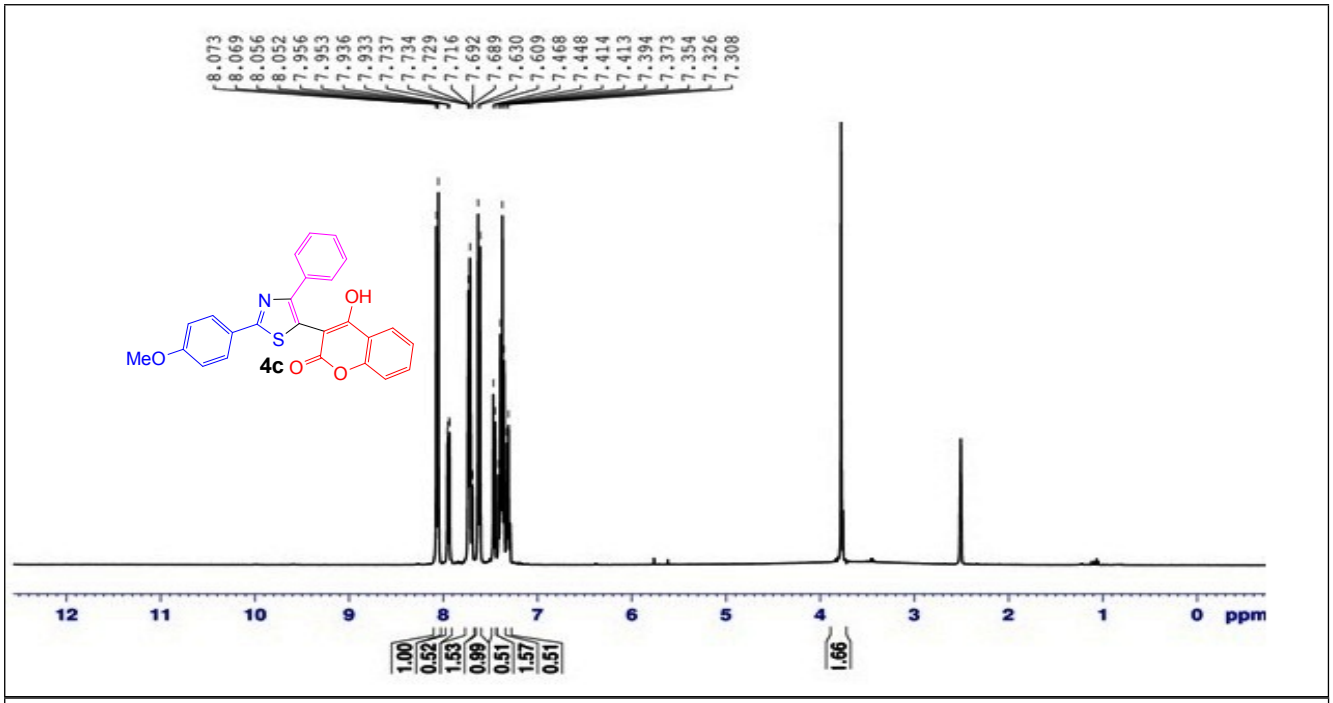
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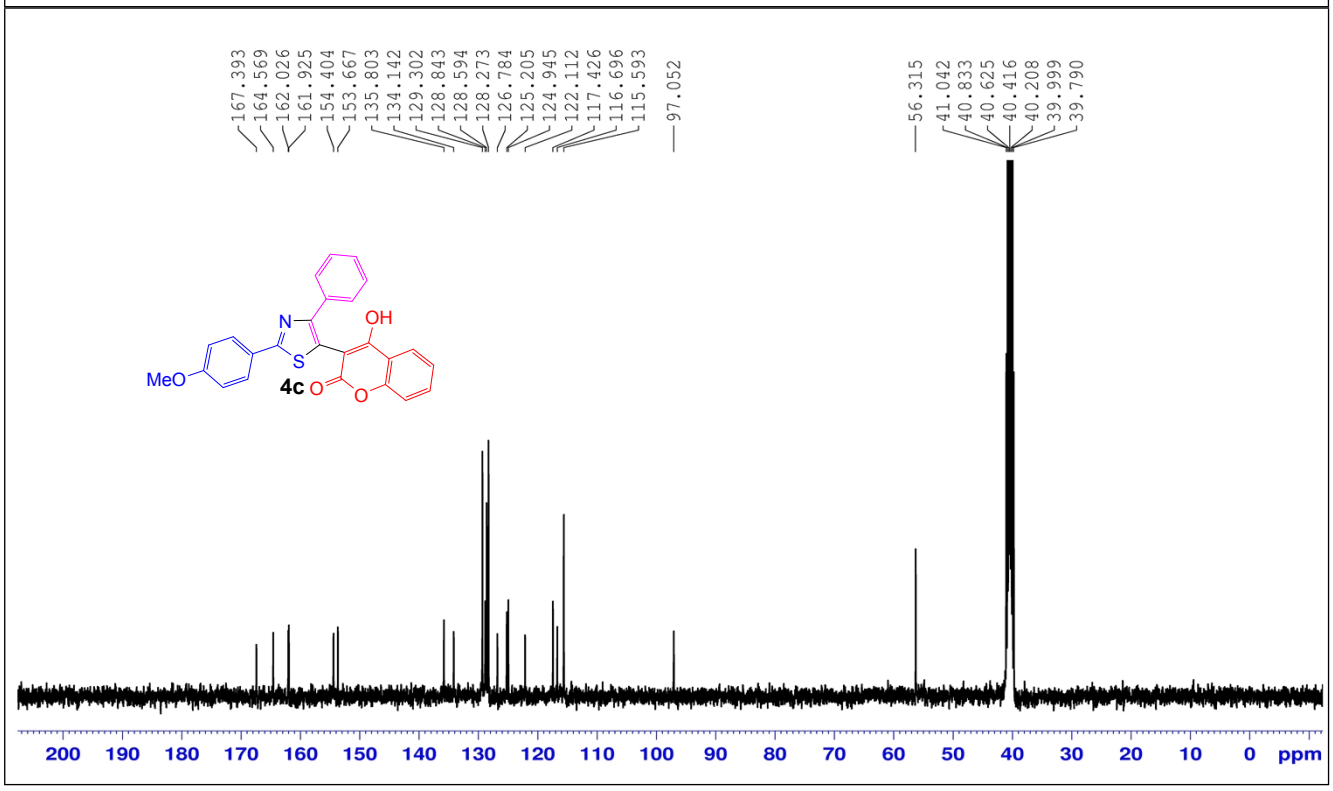
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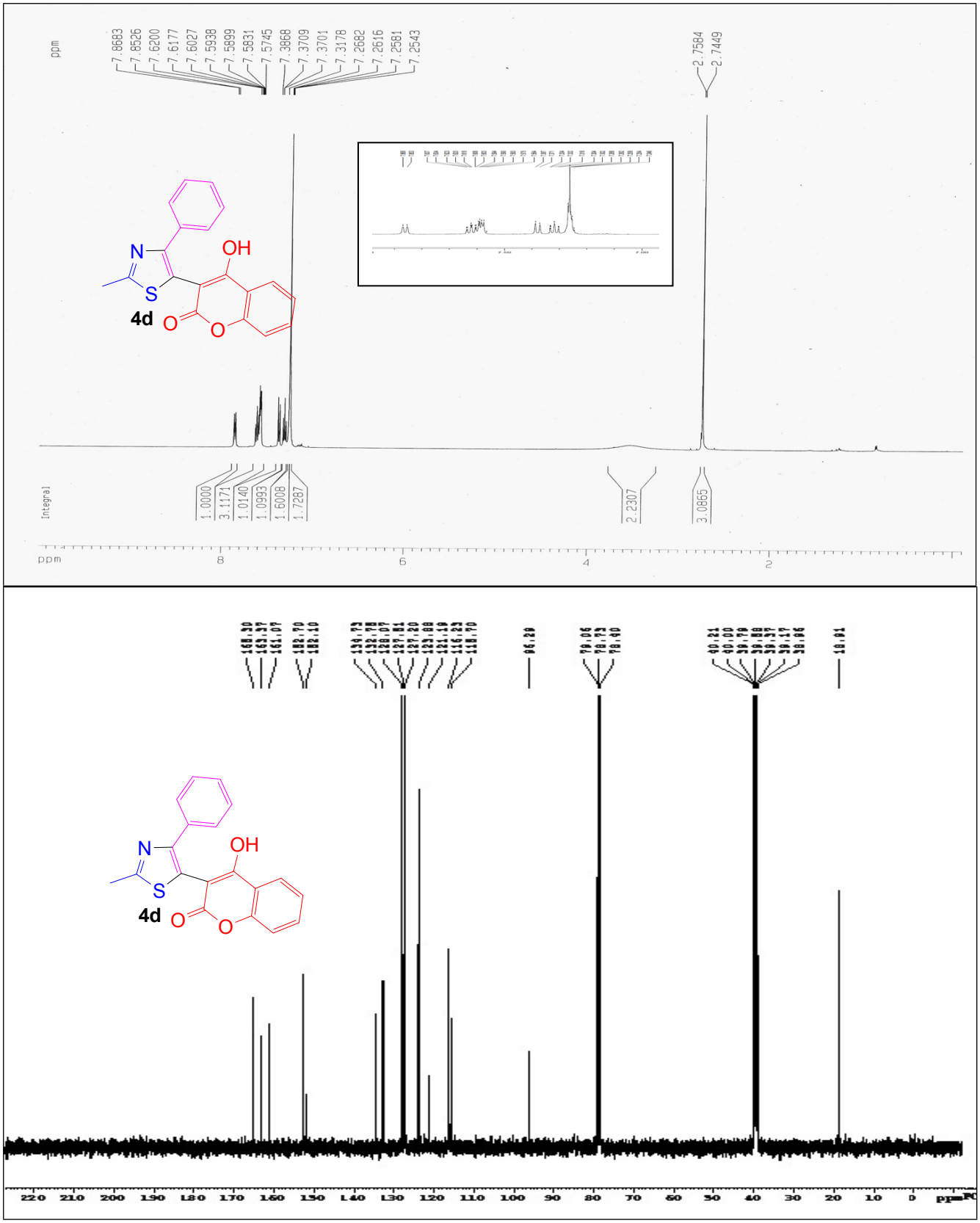
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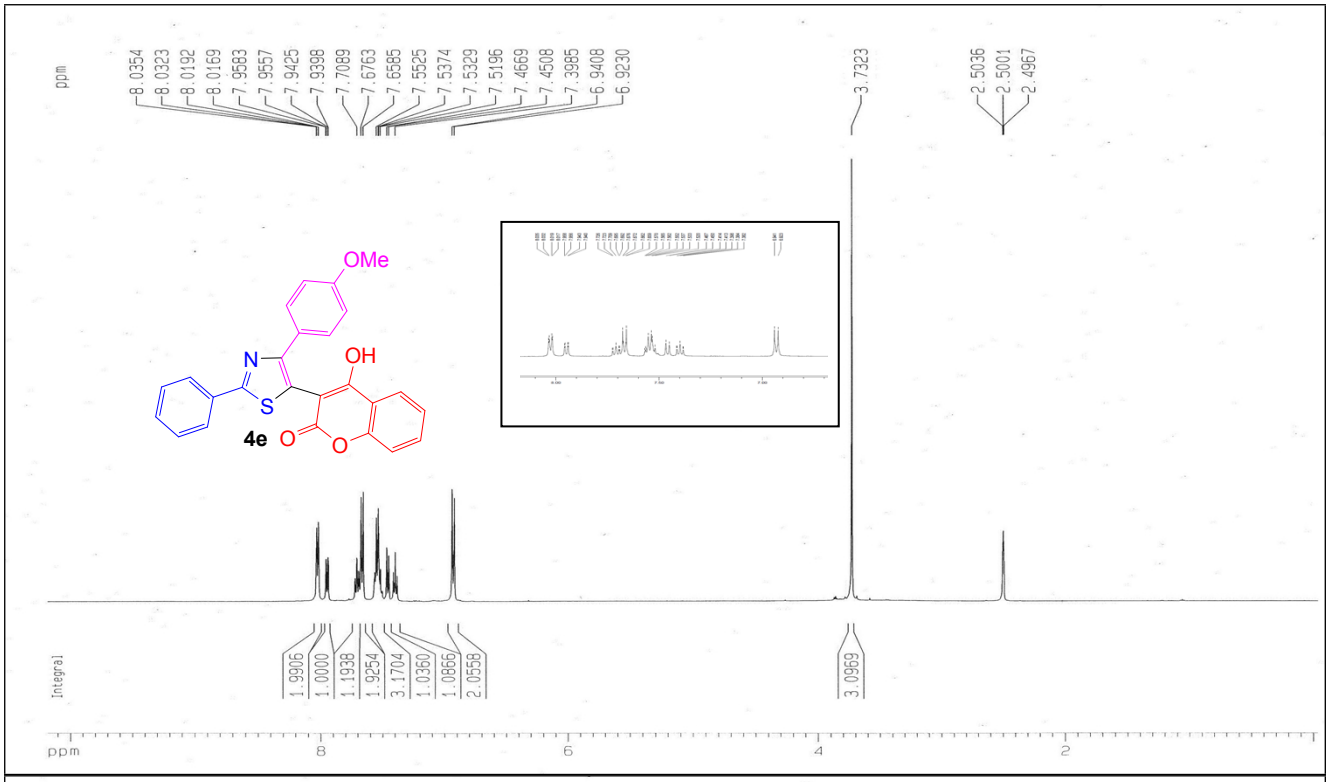
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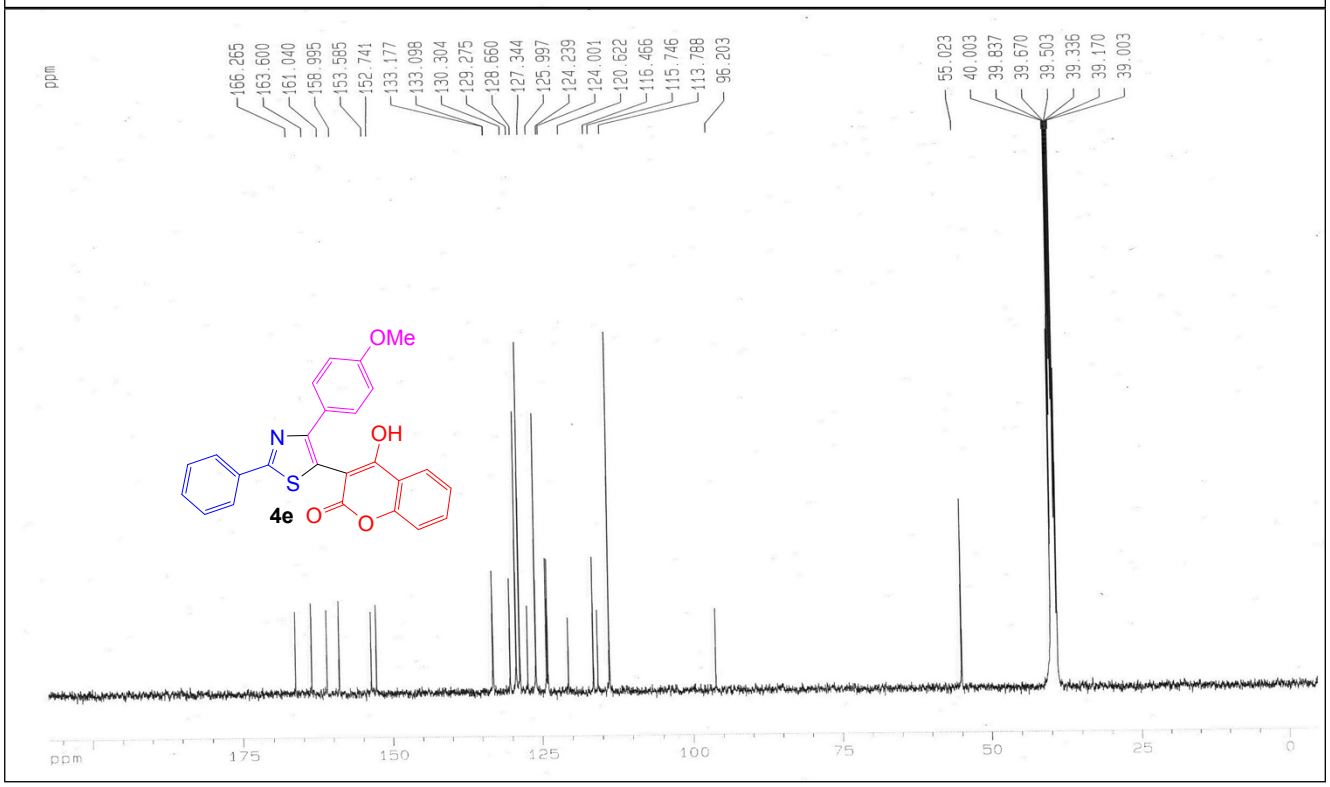
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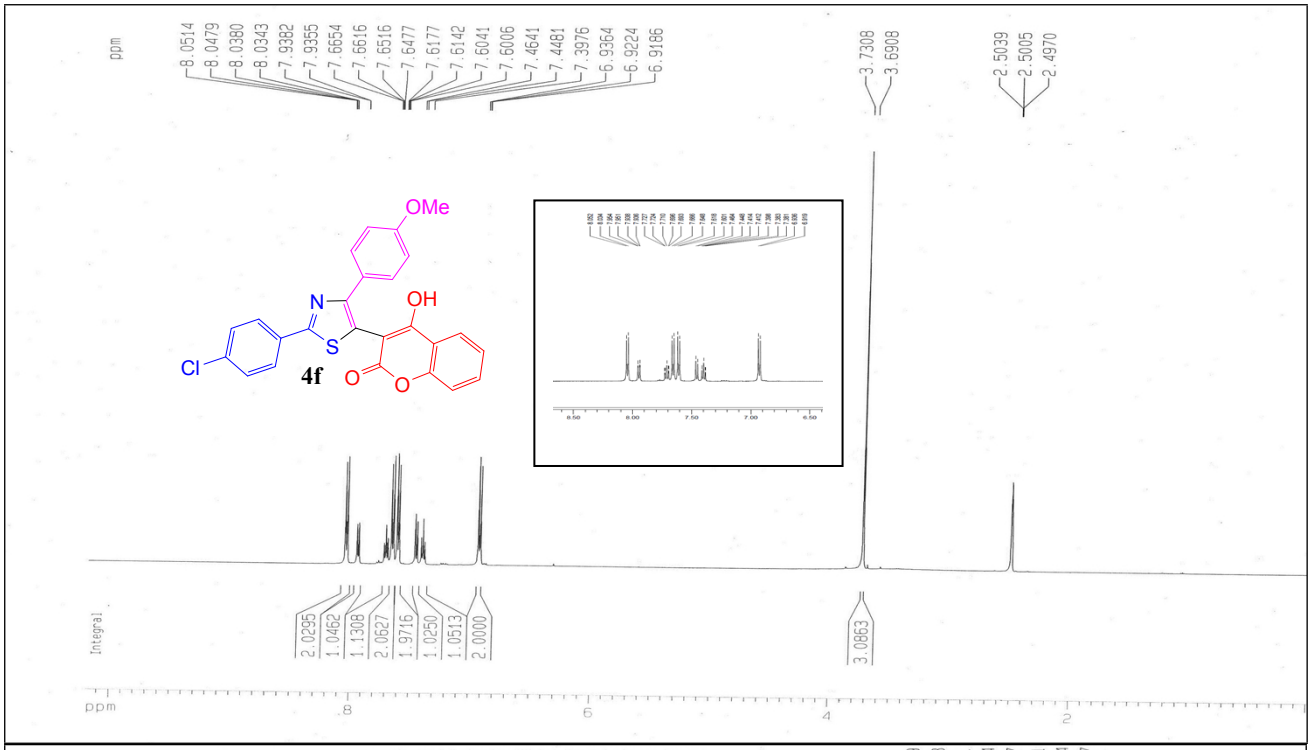
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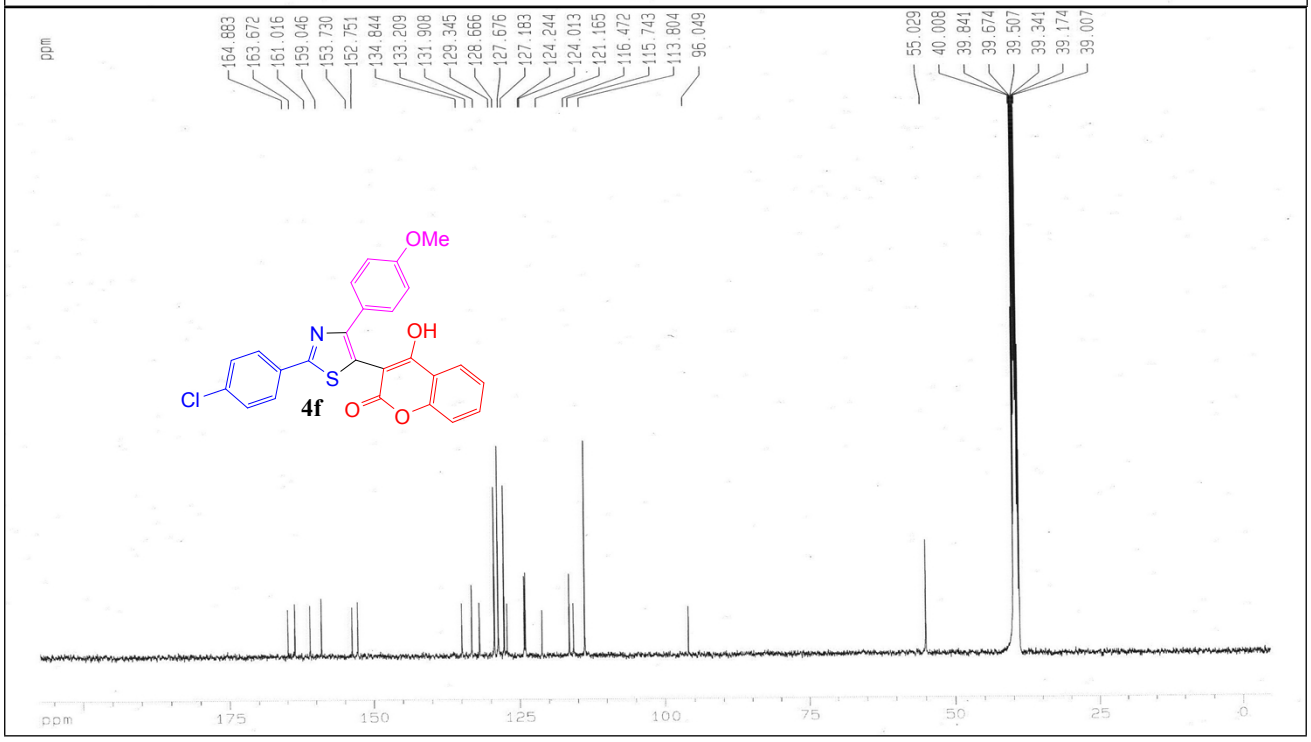
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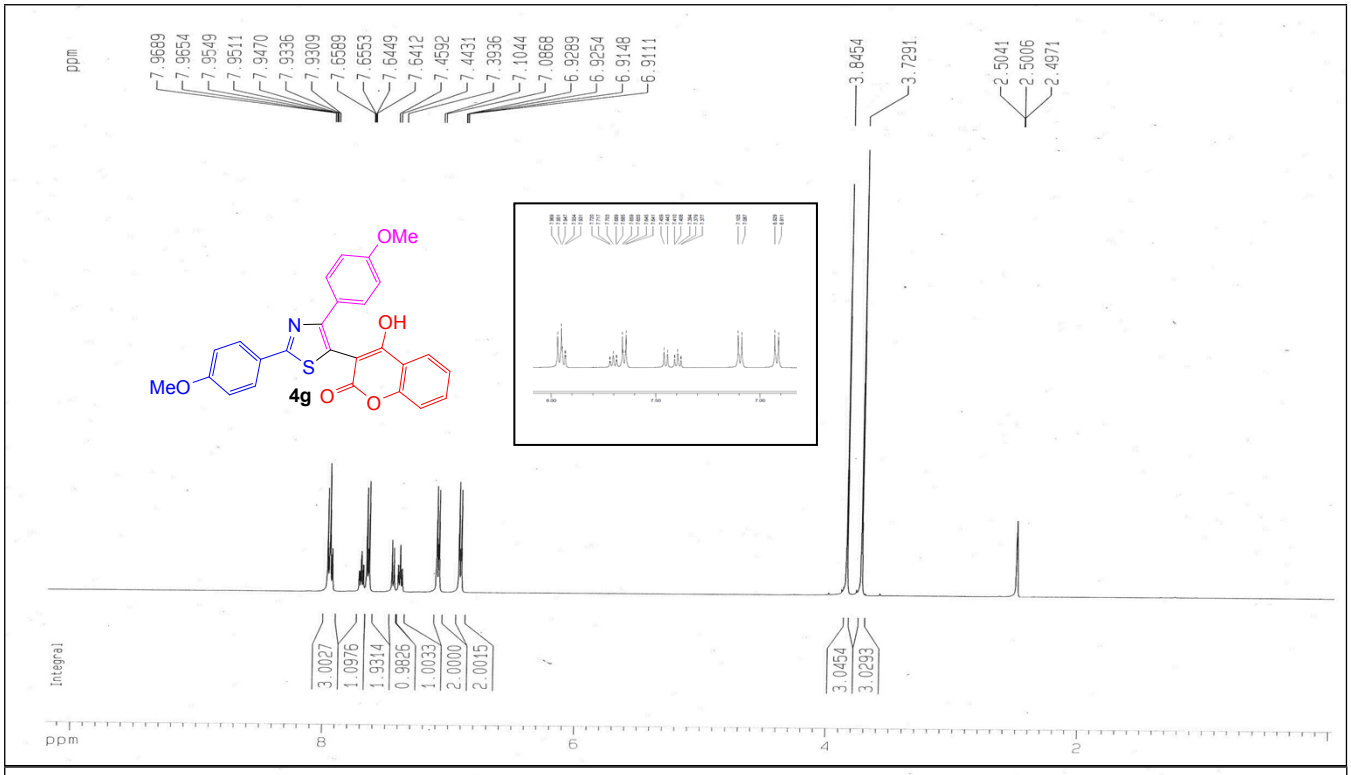
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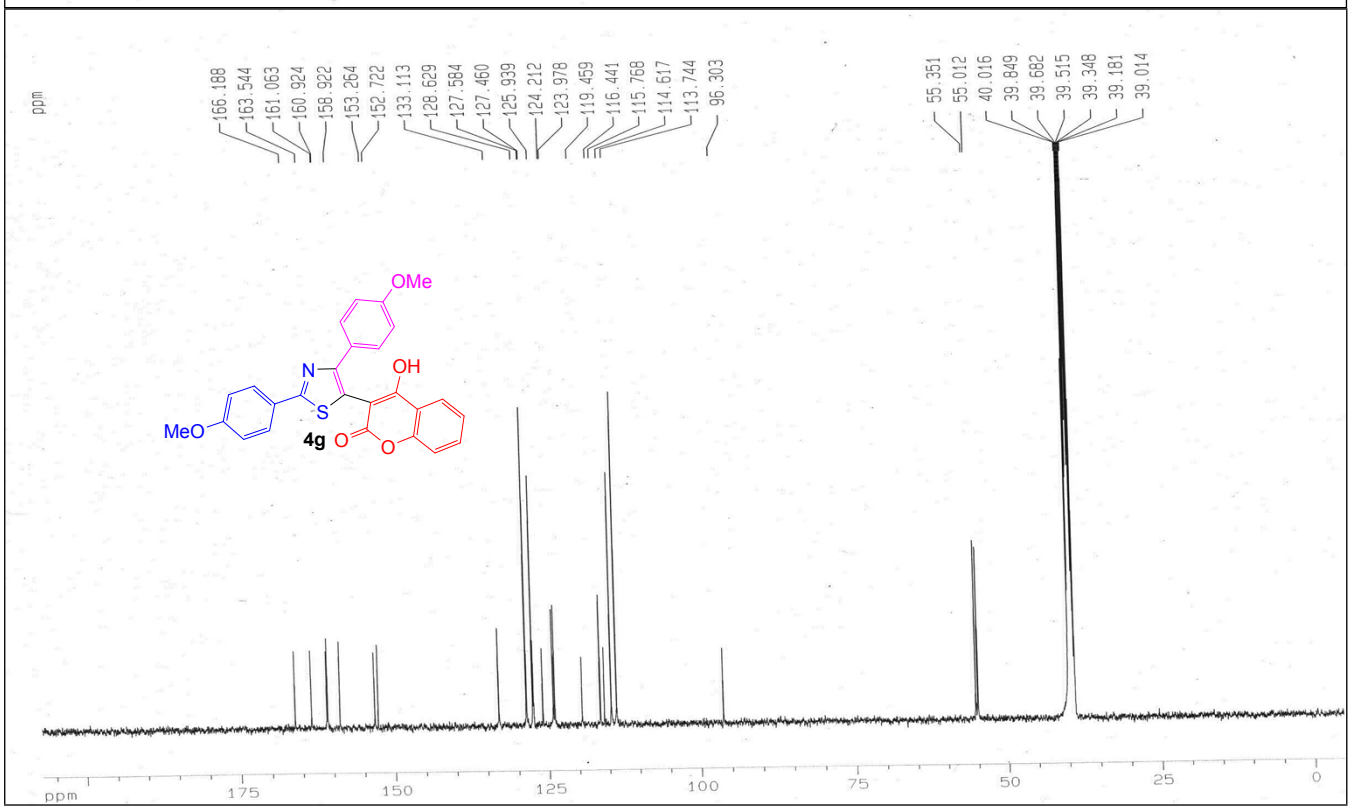
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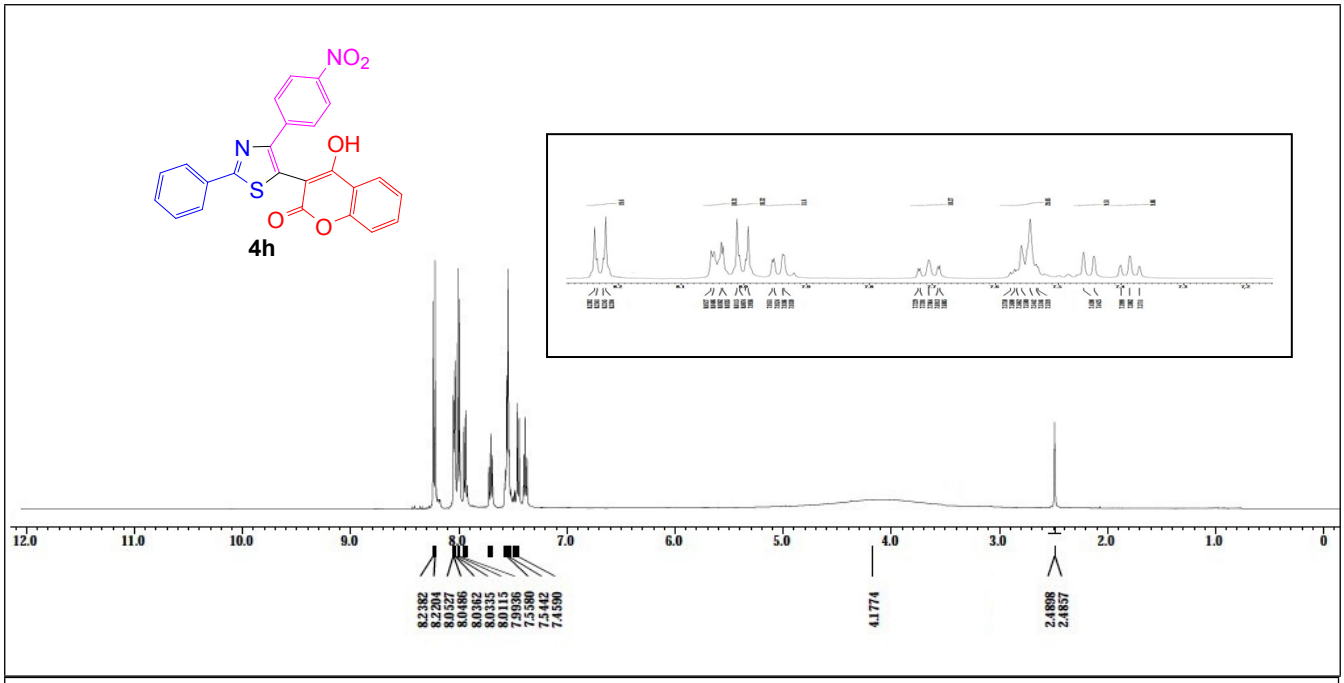
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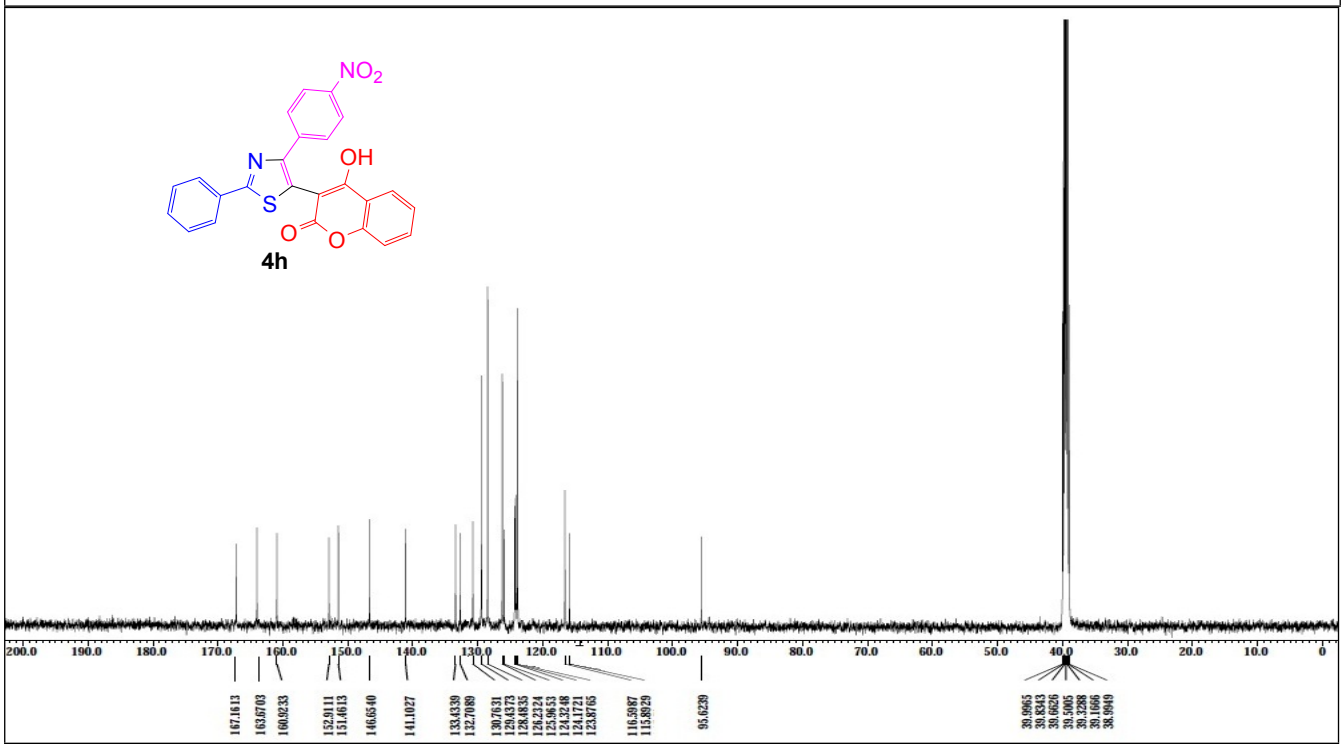
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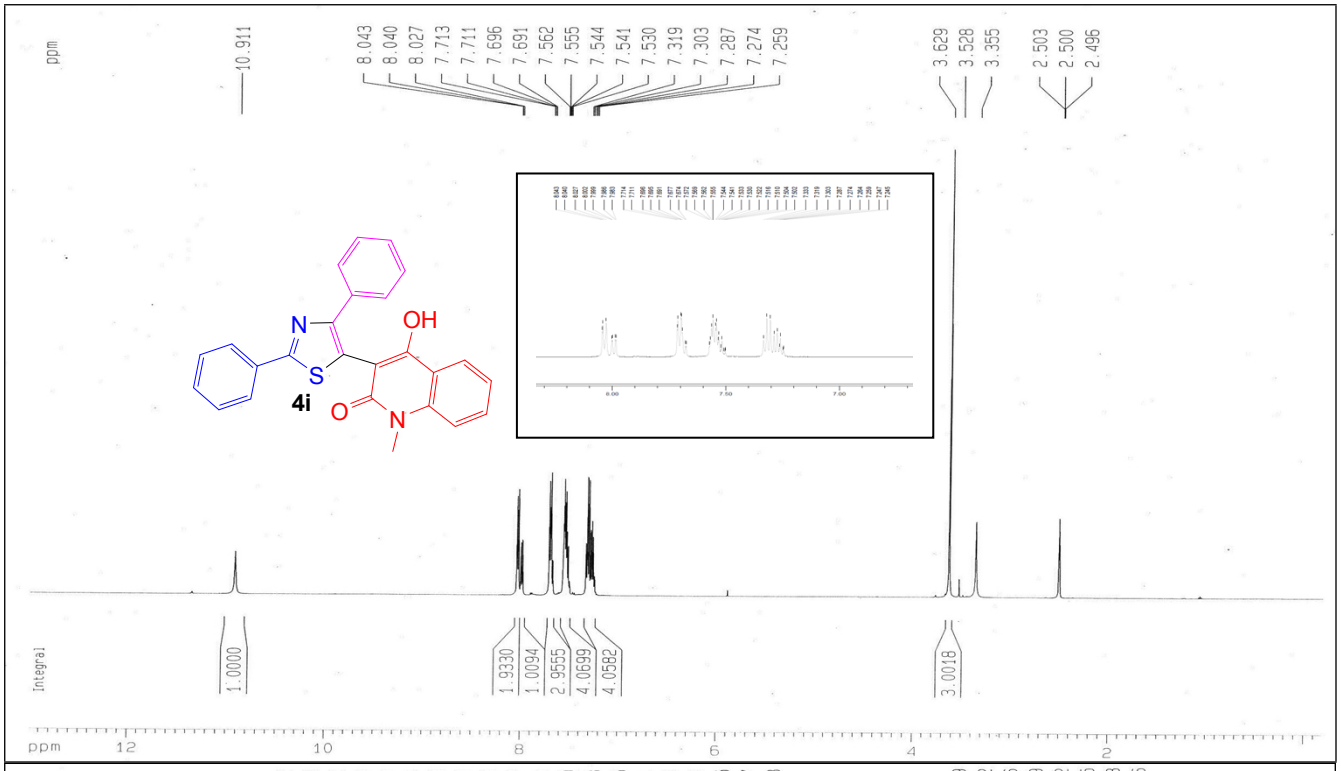
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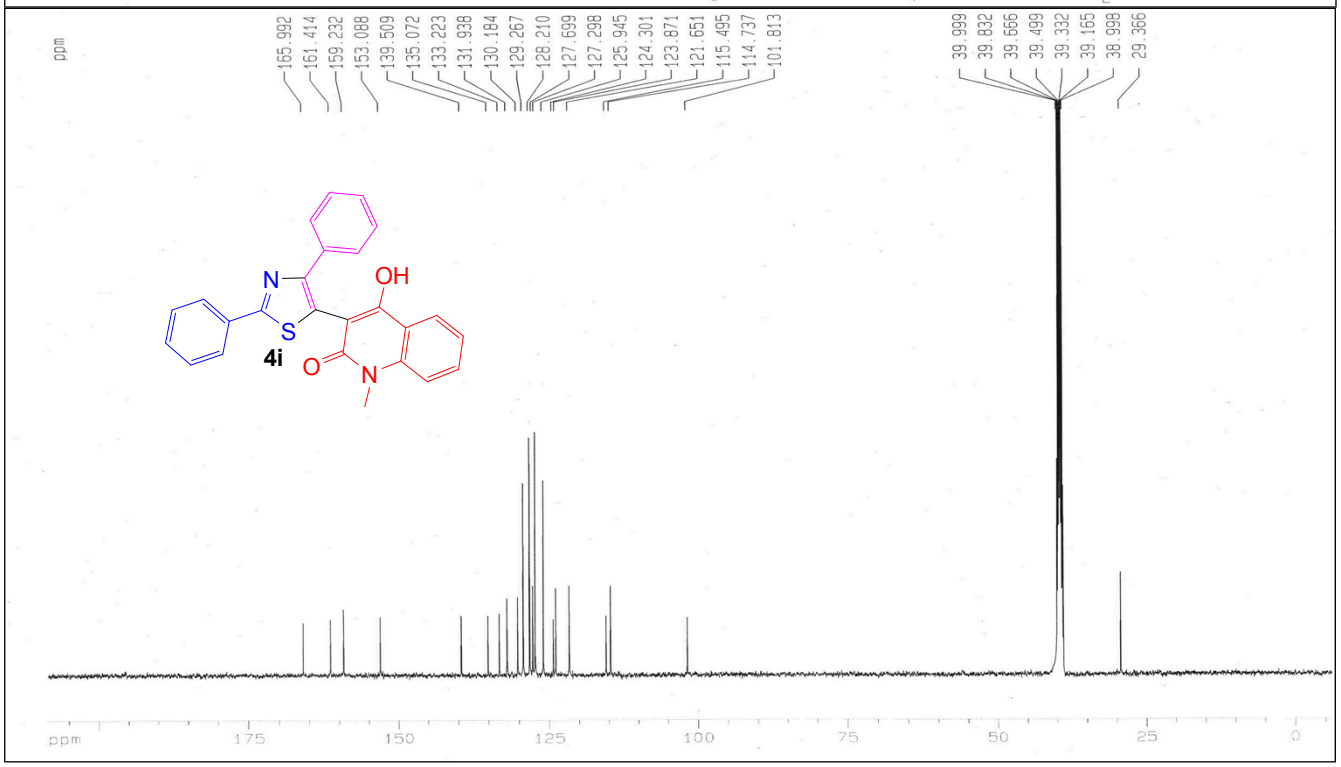
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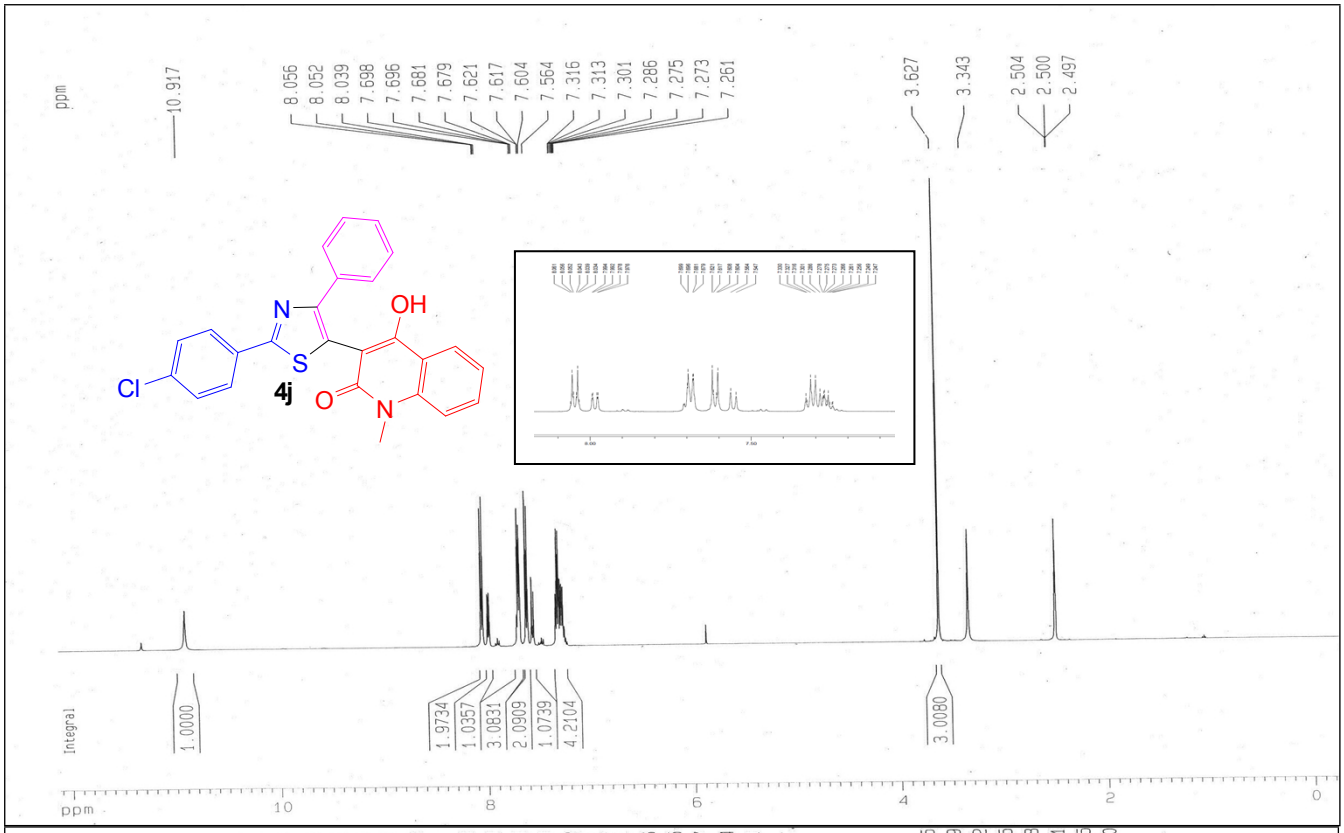
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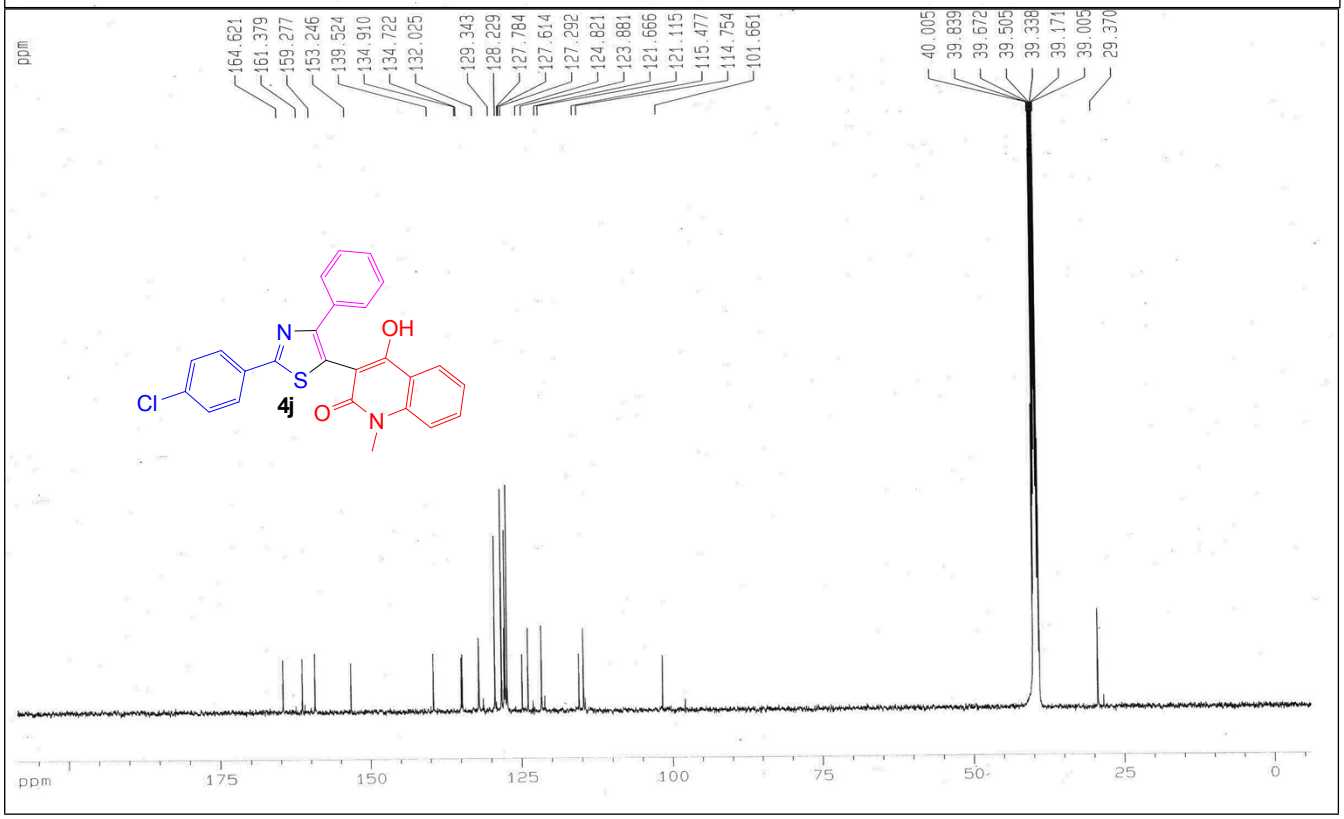
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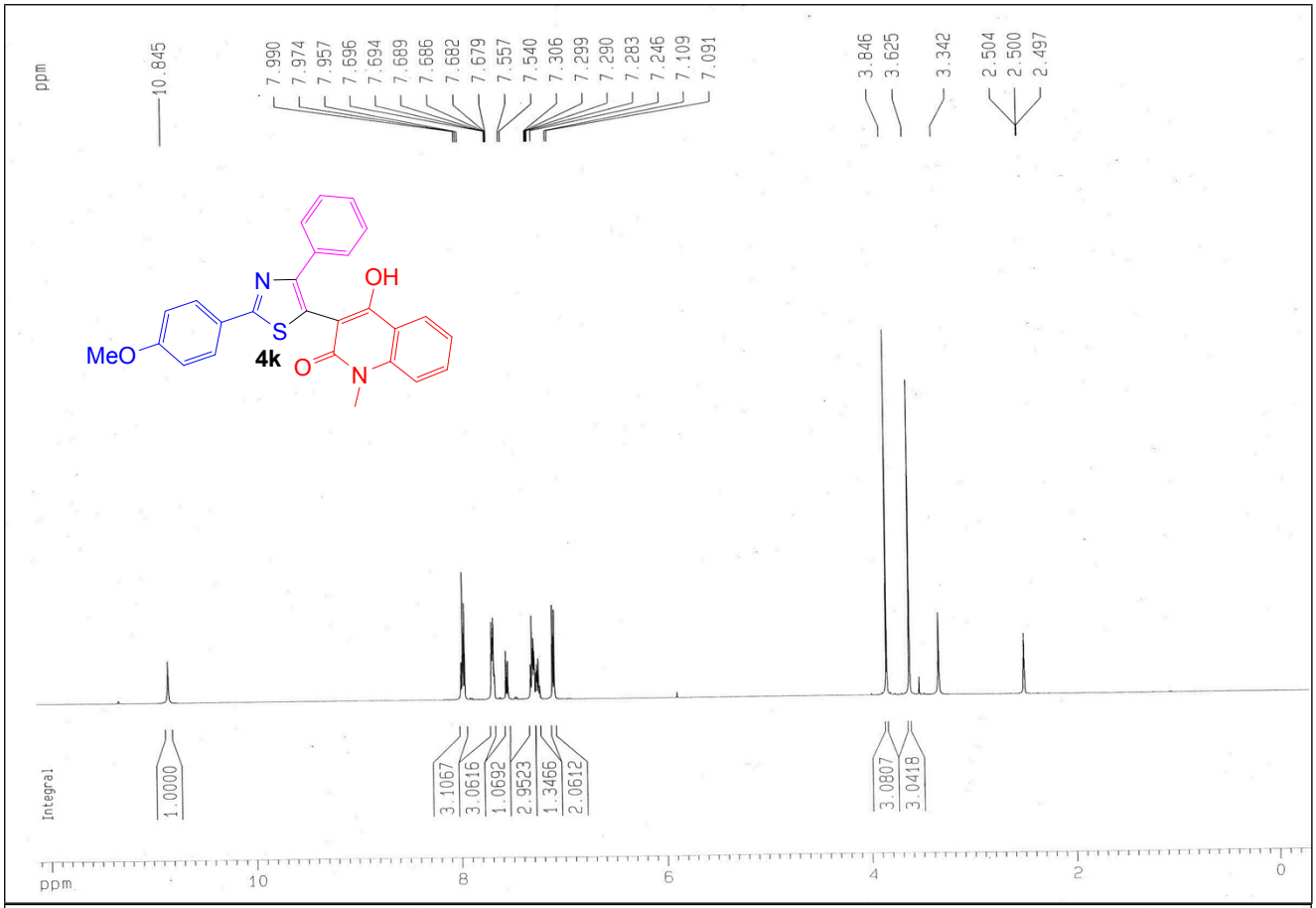
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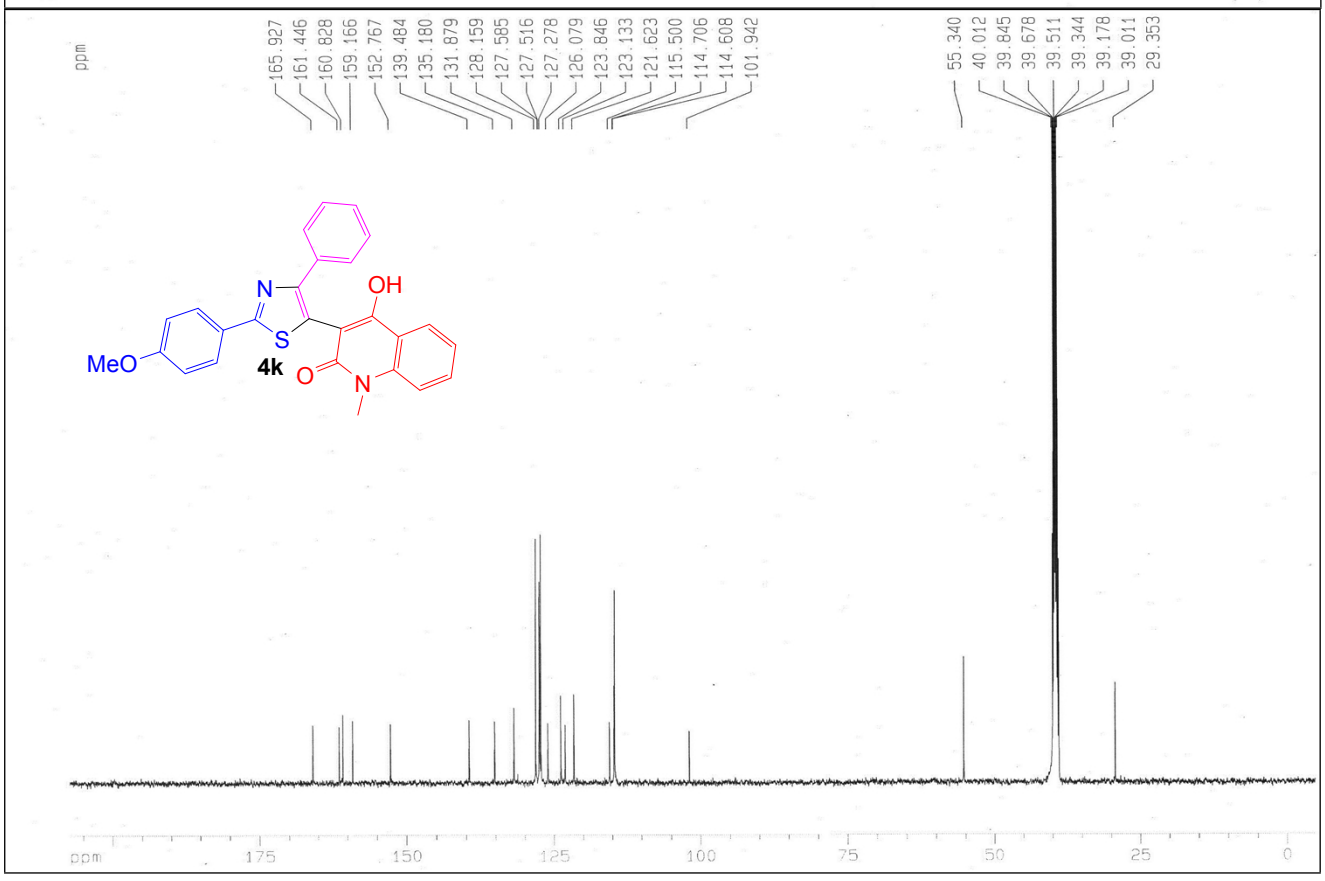
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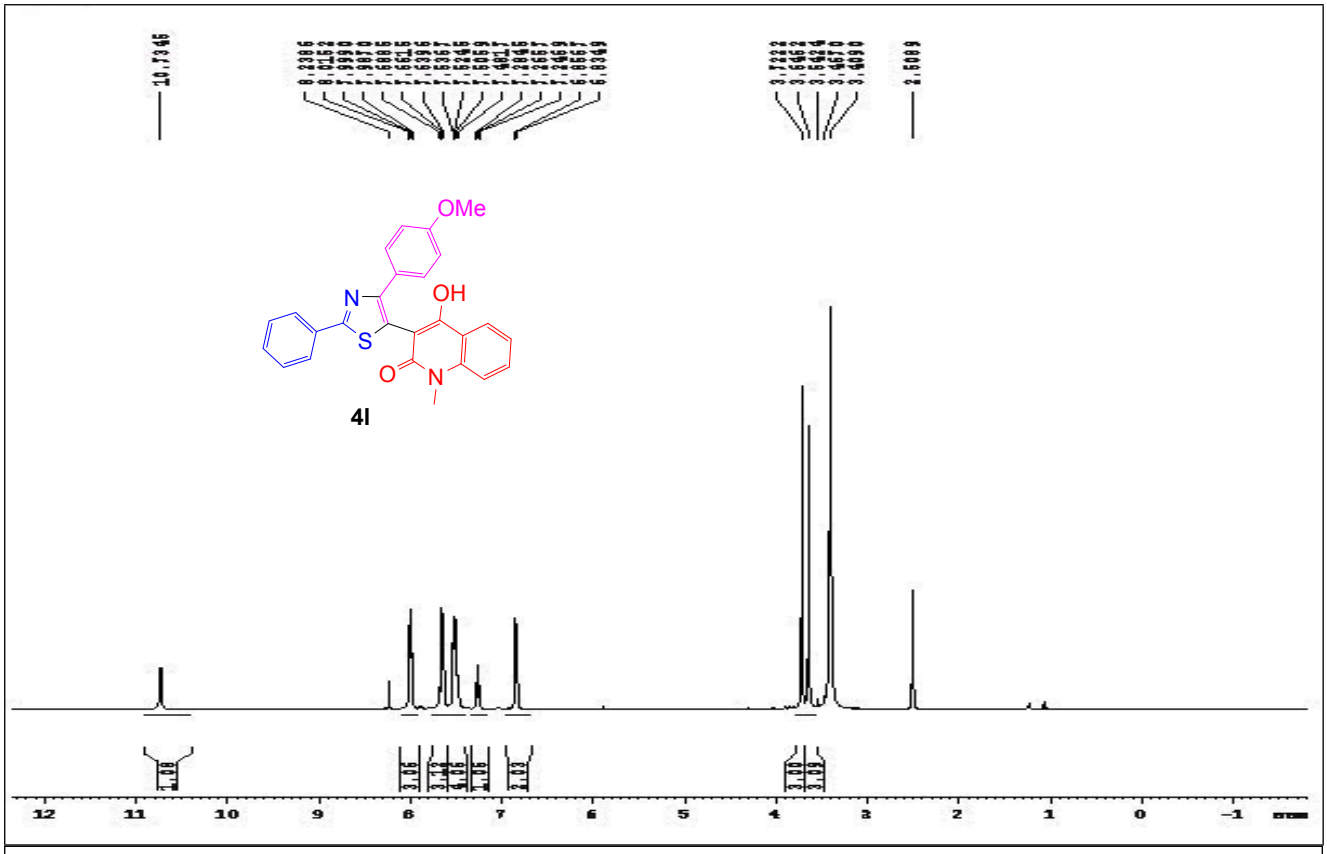

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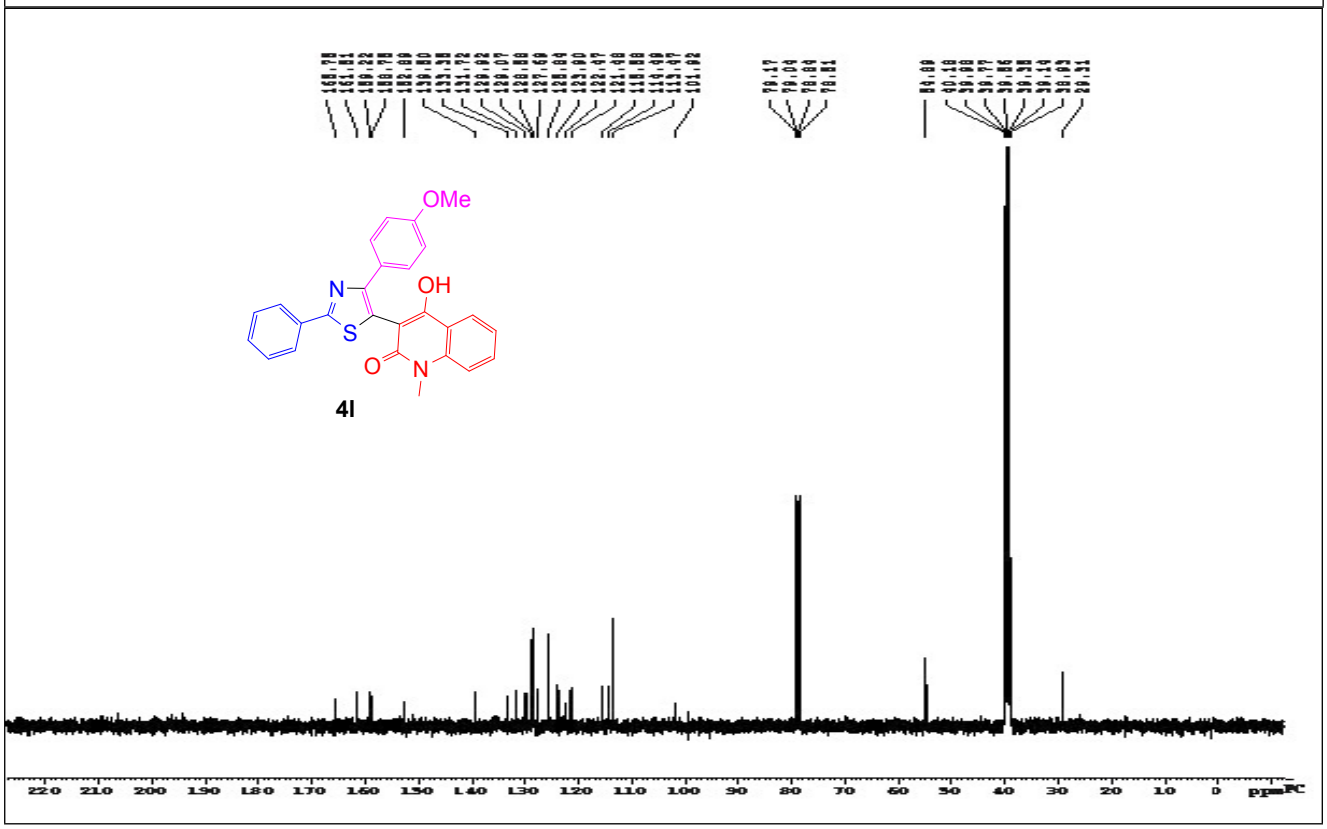

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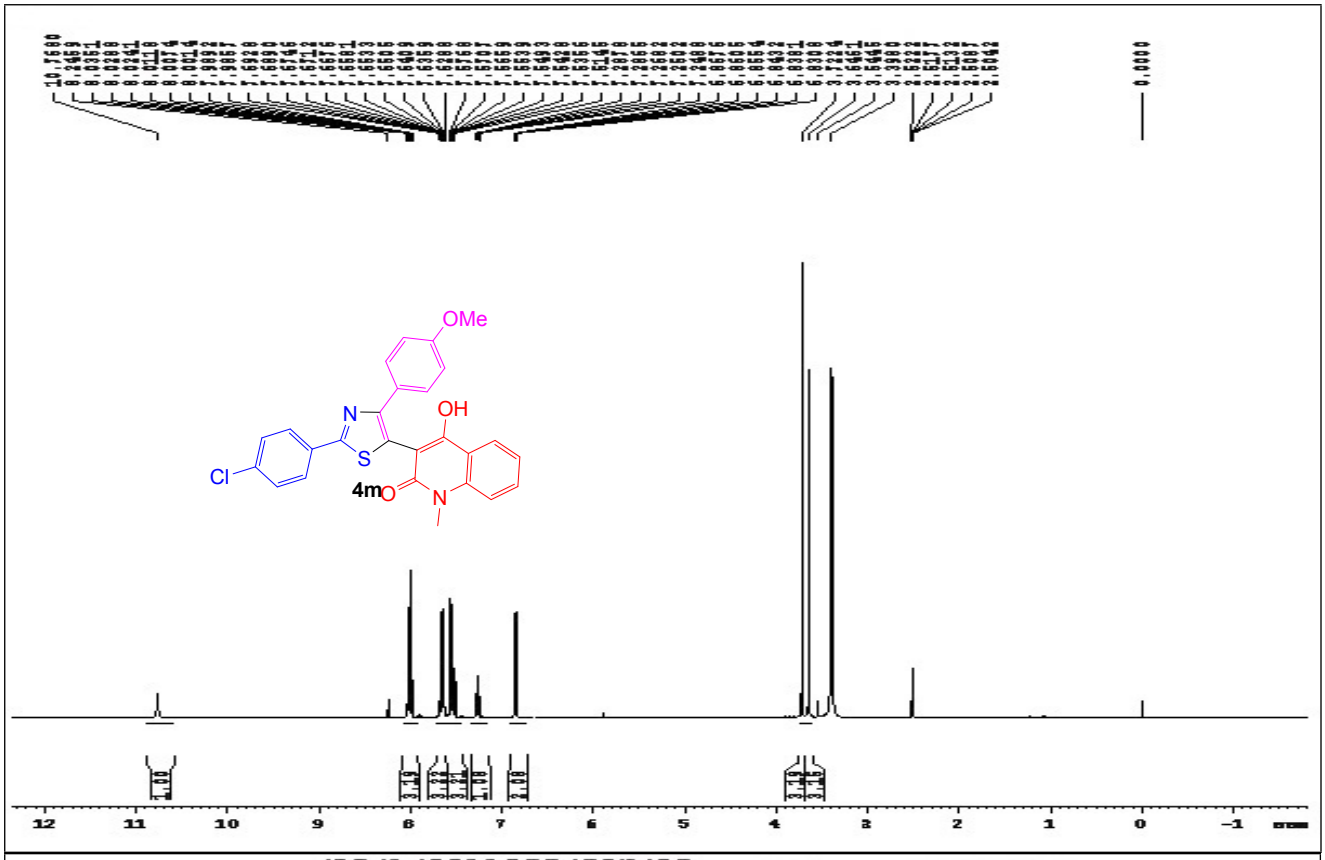
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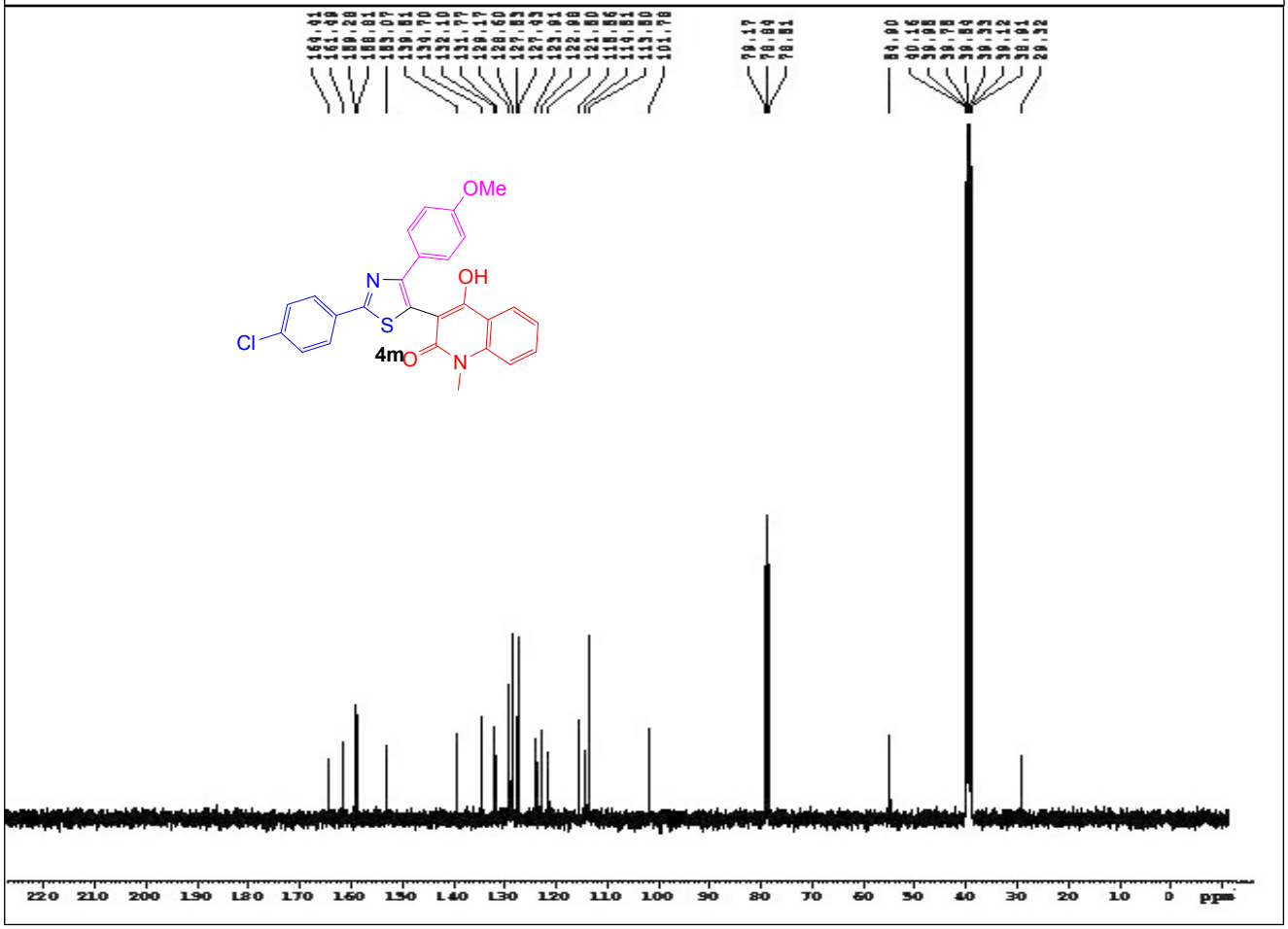
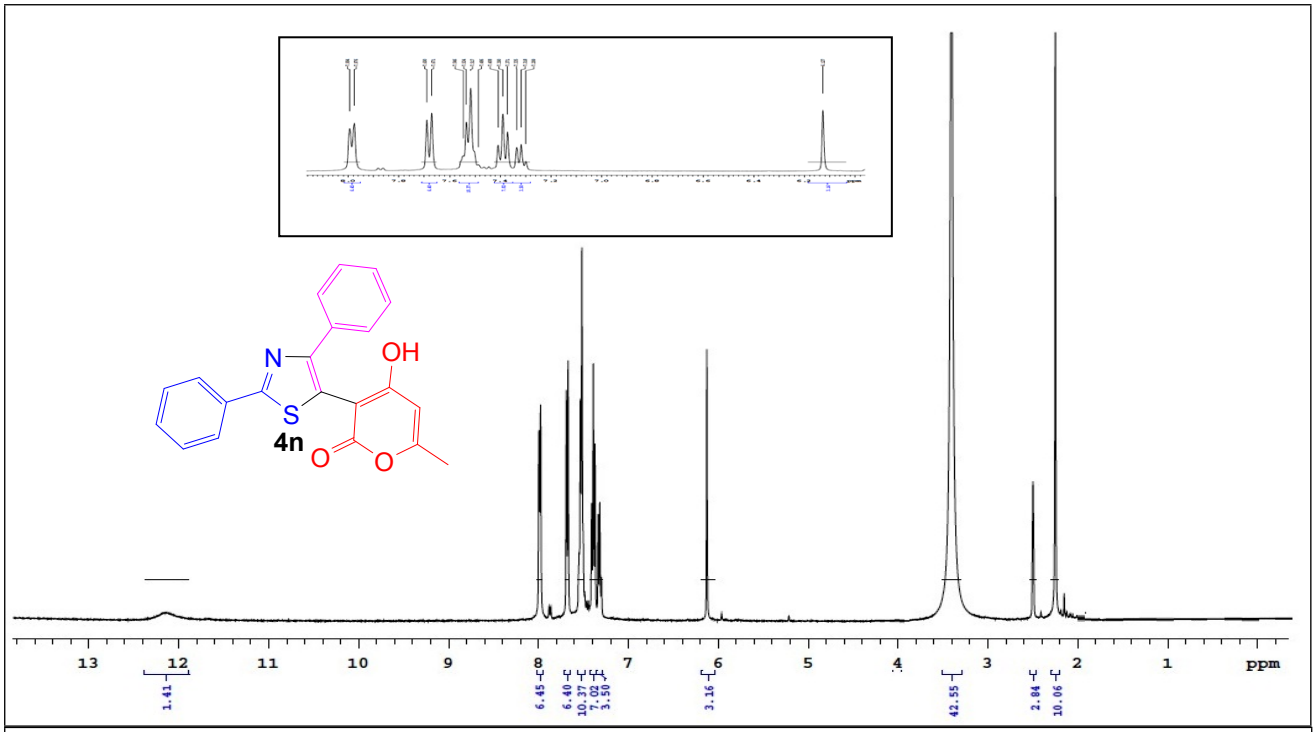
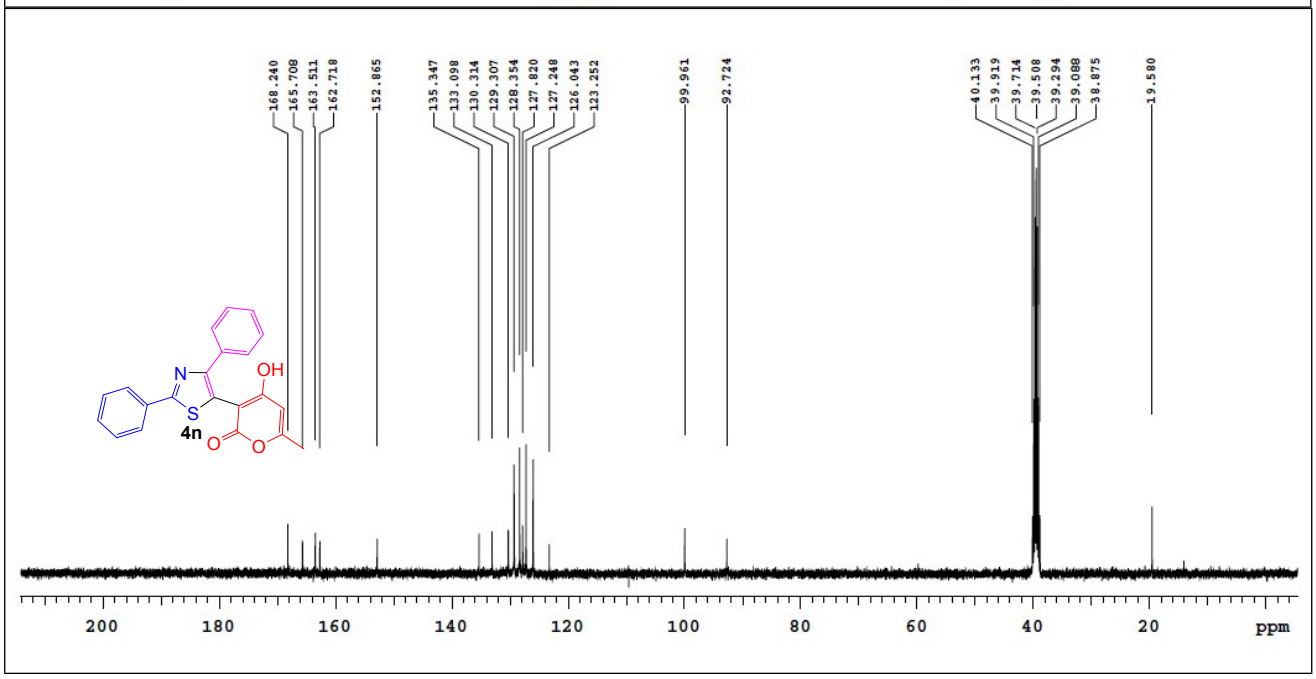
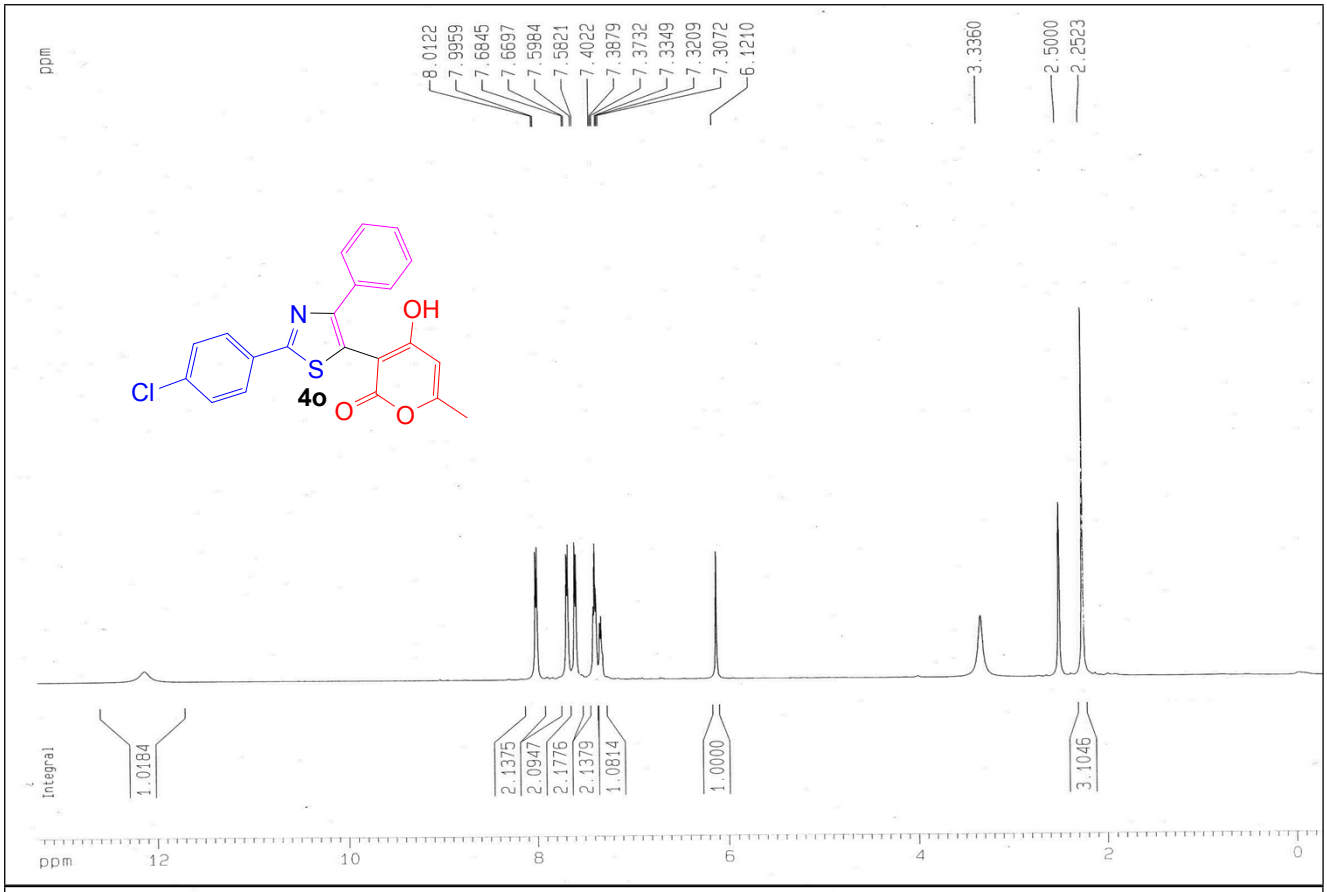
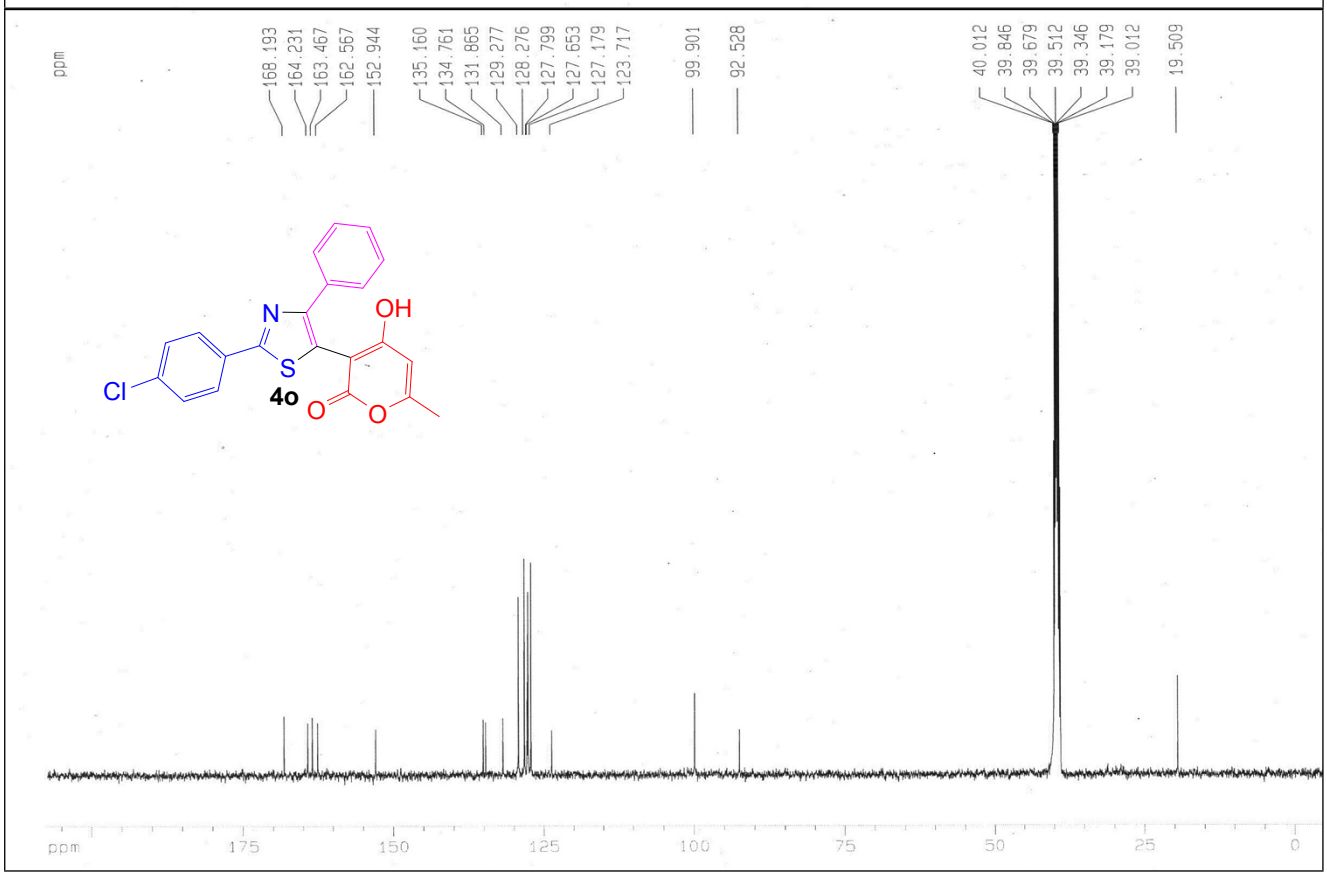
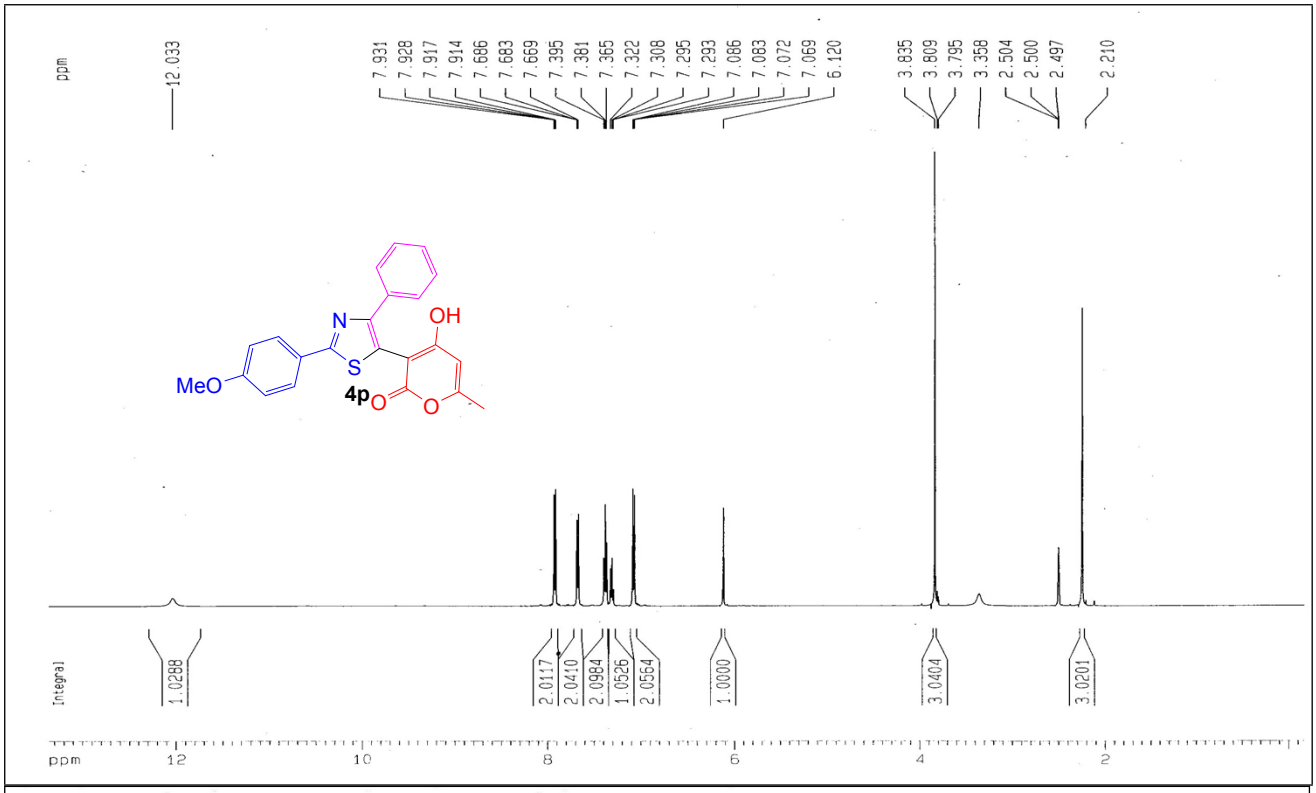

OMe
OH
N
S
Cl
4m
O
N

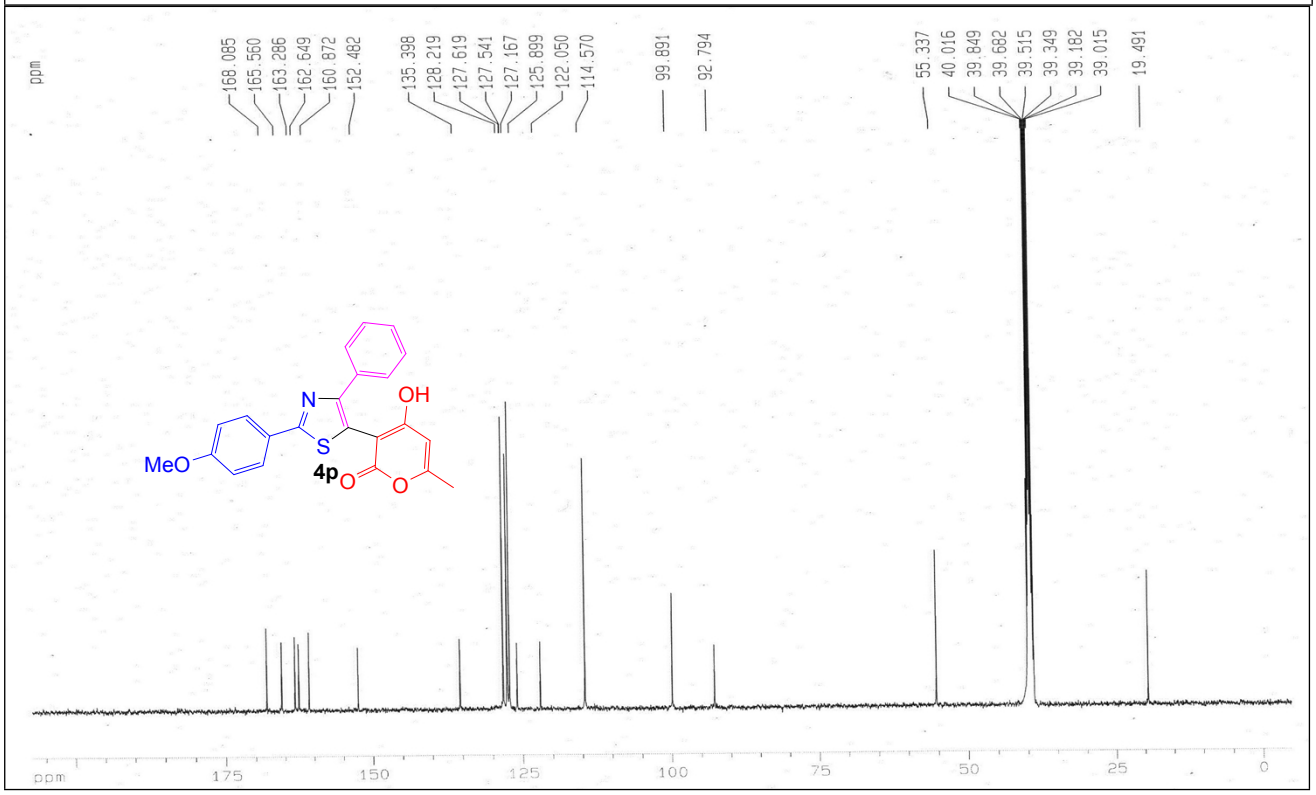

N
S
OH
O
O
4n
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.41
6.45
6.40
10.37
12.02
3.50
3.16
42.55
2.84
10.06

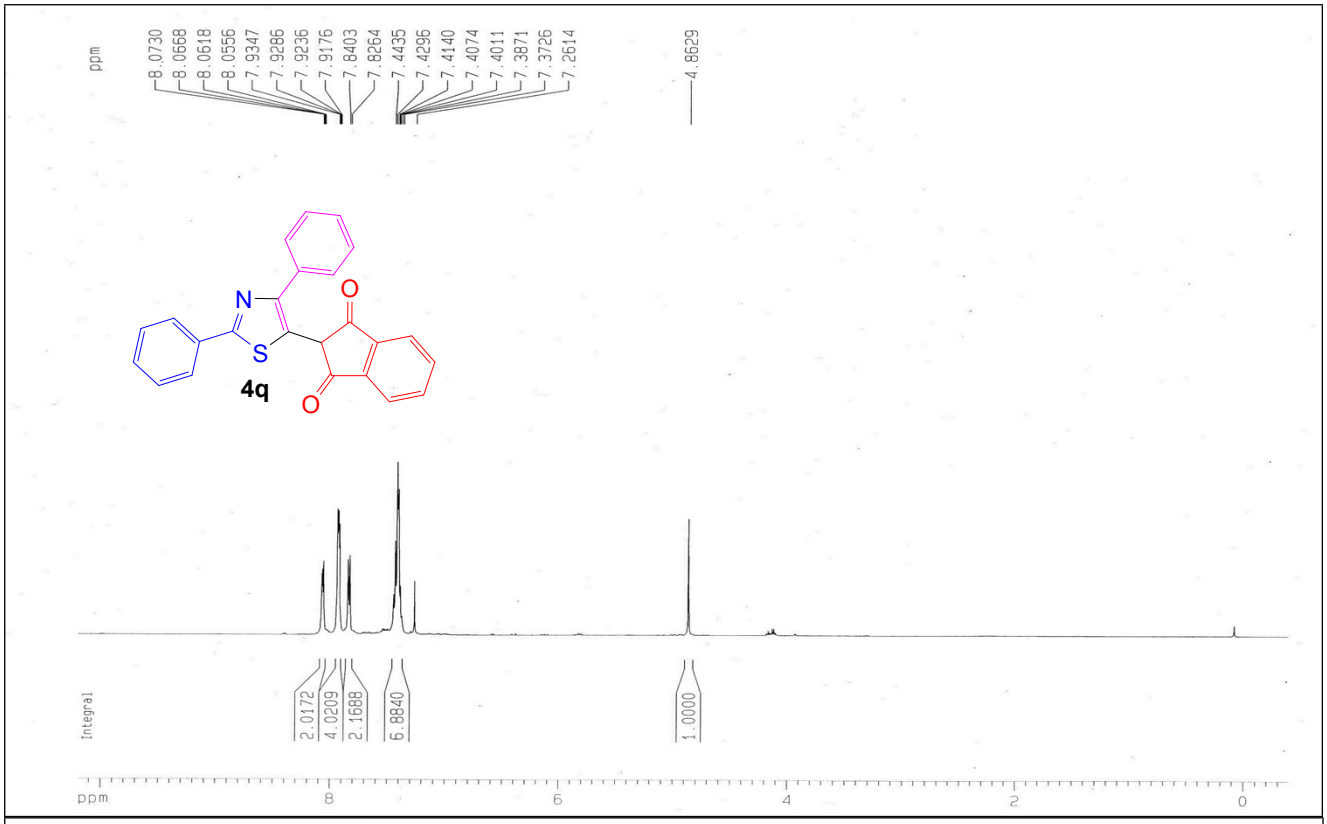

168.240
165.700
163.511
162.718
152.865
135.347
133.098
130.314
129.307
128.354
127.820
127.248
126.043
123.252
99.961
92.724
40.133
39.919
39.714
39.508
39.294
39.088
38.875
19.580
N
S
OH
O
O
4n
200
180
160
140
120
100
80
60
40
20
ppm

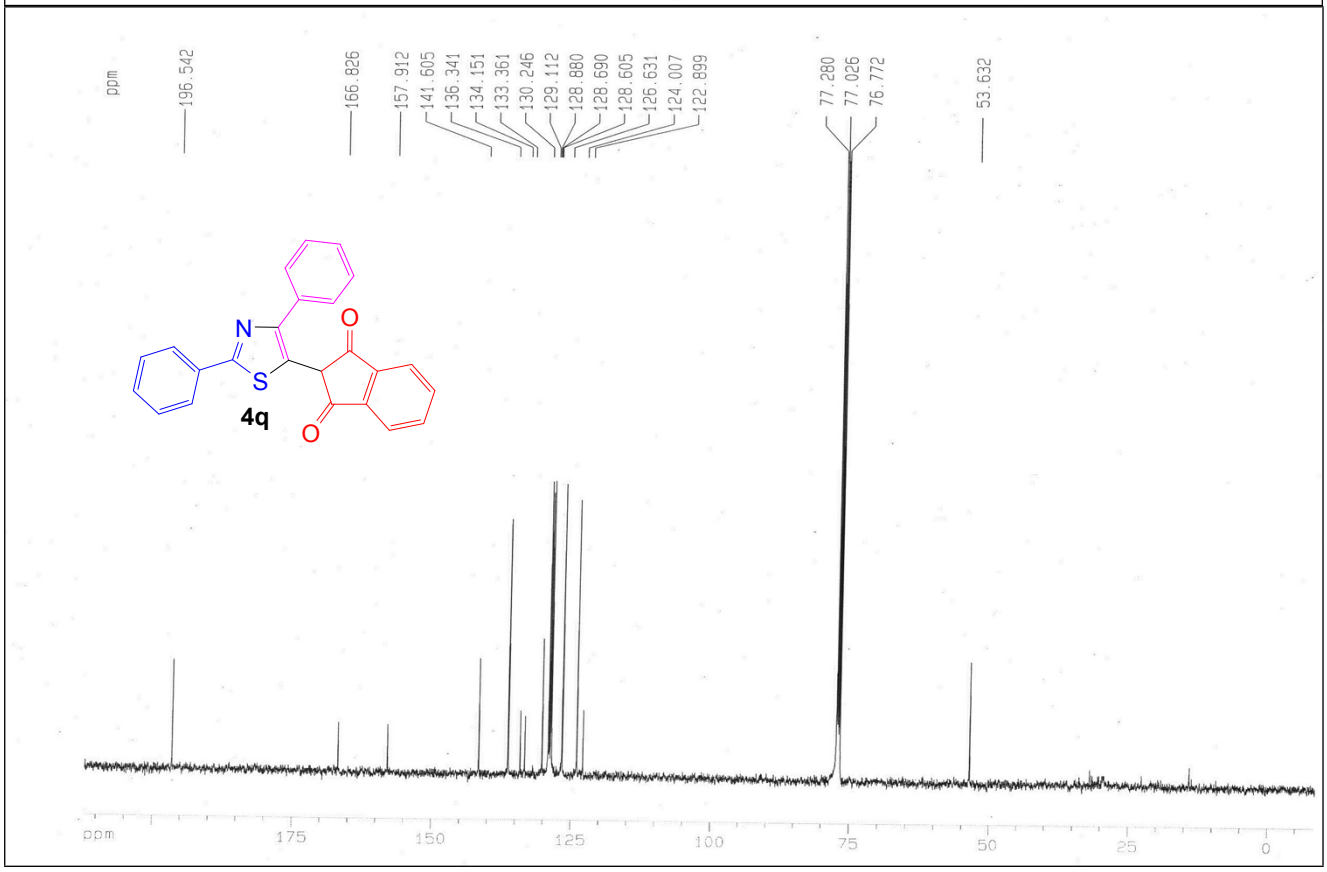

ppm
8.0122
7.9959
7.6845
7.6697
7.5984
7.5821
7.4022
7.3879
7.3732
7.3349
7.3209
7.3072
6.1210
3.3360
2.5000
2.5523
OH
N
S
Cl
4o
O
O
Integral
1.0184
2.1375
2.0947
2.1776
2.1379
1.0814
1.0000
3.1046
ppm
12
10
8
6
4
2
0

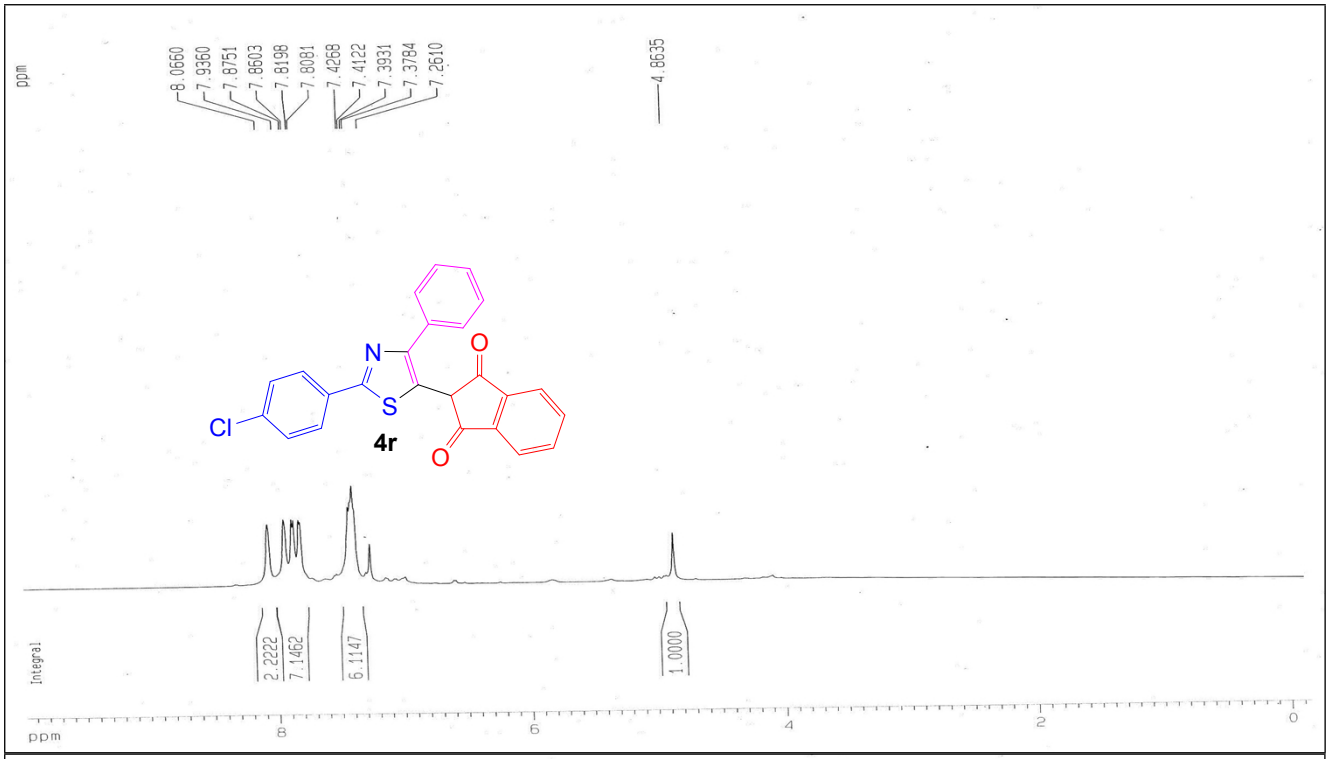

ppm
168.193
164.231
163.467
162.567
152.944
135.160
134.761
131.865
129.277
128.276
127.799
127.653
127.179
123.717
99.901
92.528
40.012
39.846
39.679
39.512
39.346
39.179
39.012
19.509
OH
N
S
Cl
4o
O
O
ppm
175
150
125
100
75
50
25
0

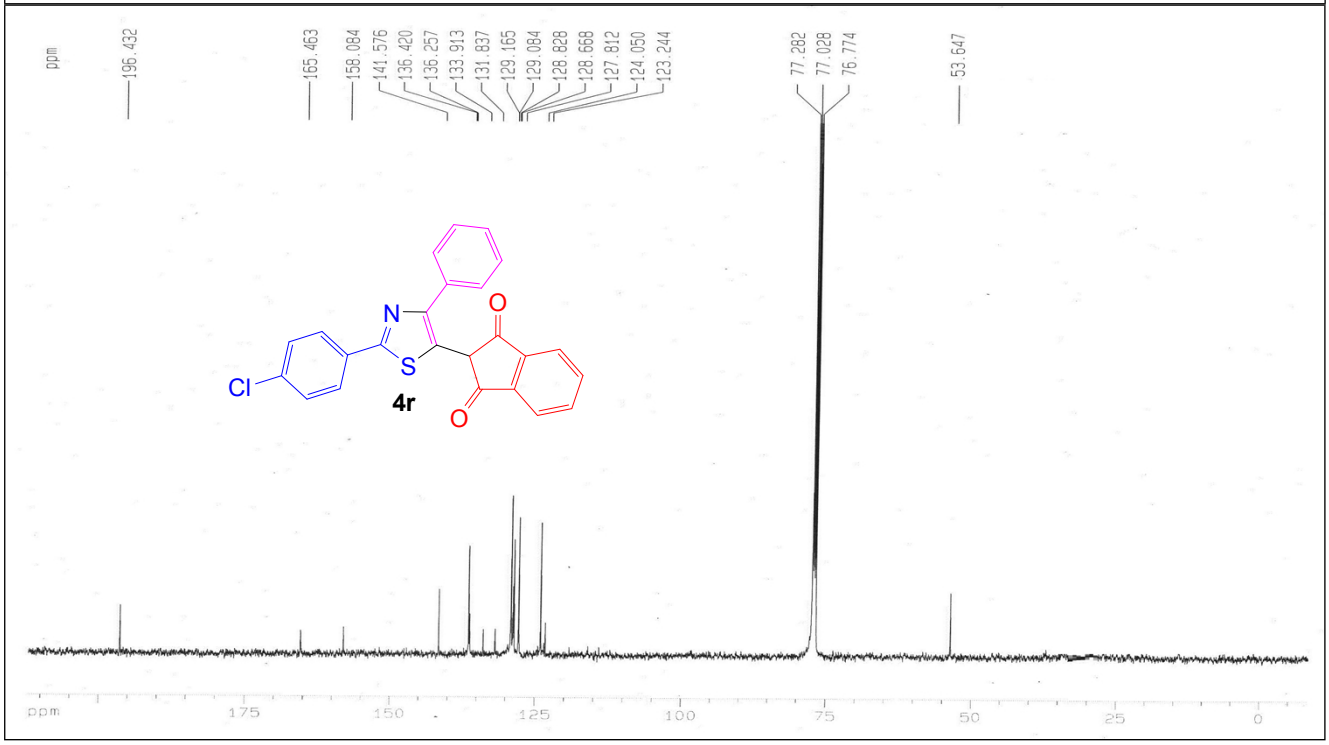

ppm
12.033
7.931
7.928
7.917
7.914
7.686
7.683
7.669
7.395
7.381
7.365
7.322
7.308
7.295
7.293
7.086
7.083
7.072
7.069
6.120
3.835
3.809
3.795
3.358
2.504
2.500
2.497
2.210
N
S
OH
MeO
4p
O
O
Integral
1.0288
2.0117
2.0410
2.0984
1.0526
2.0564
1.0000
3.0404
3.0201
ppm
12
10
8
6
4
2

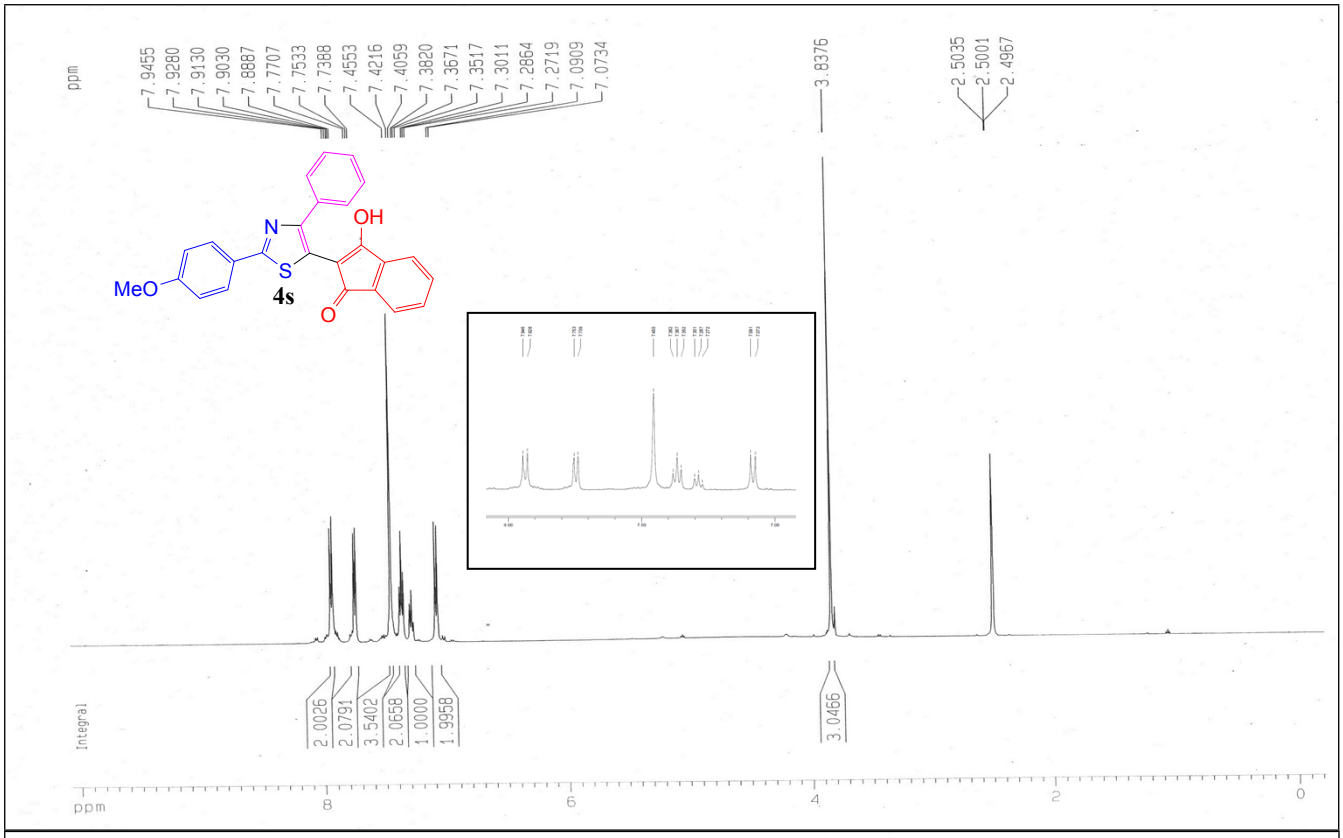

ppm
168.085
165.560
163.286
162.649
160.872
152.482
135.398
128.219
127.619
127.541
127.167
125.899
122.050
114.570
99.891
92.794
55.337
40.016
39.849
39.682
39.515
39.349
39.182
39.015
19.491
N
S
OH
MeO
4p
O
O
ppm
175
150
125
100
75
50
25
0

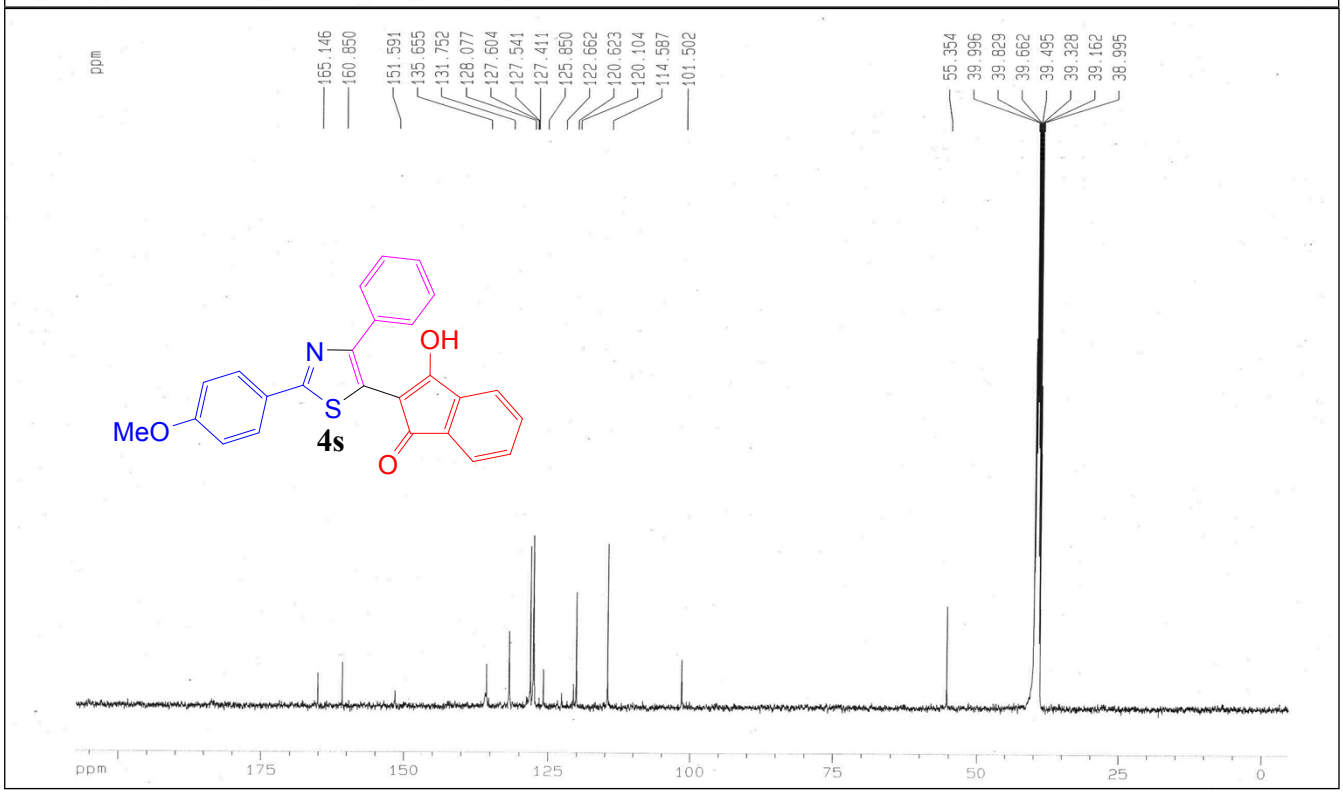

ppm
8.0730
8.0668
8.0618
8.0556
7.9347
7.9286
7.9236
7.9176
7.8403
7.8264
7.4435
7.4296
7.4140
7.4074
7.4011
7.3871
7.3726
7.2614
4.8629
N
S
O
O
4q
Integral
2.0172
4.0209
2.1688
6.8840
1.0000
ppm
8
6
4
2
0

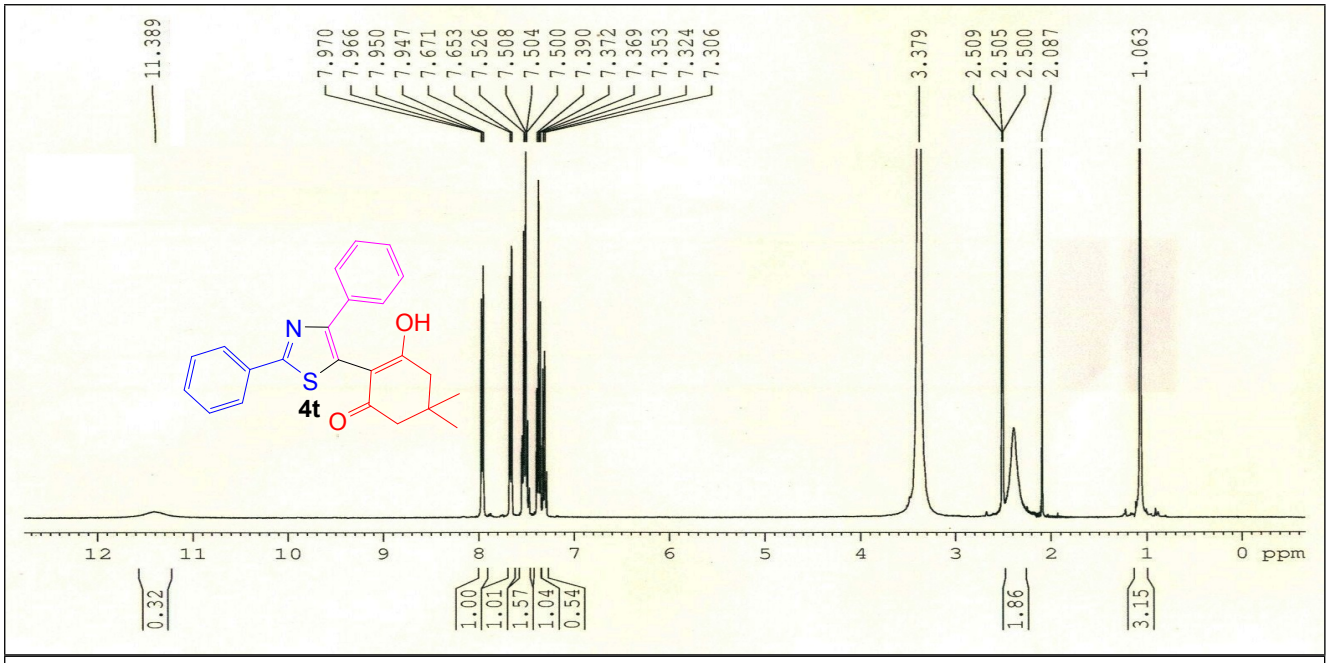

ppm
196.542
166.826
157.912
141.605
136.341
134.151
133.361
130.246
129.112
128.880
128.690
128.605
126.631
124.007
122.899
77.280
77.026
76.772
53.632
N
S
O
O
4q
ppm
175
150
125
100
75
50
25
0

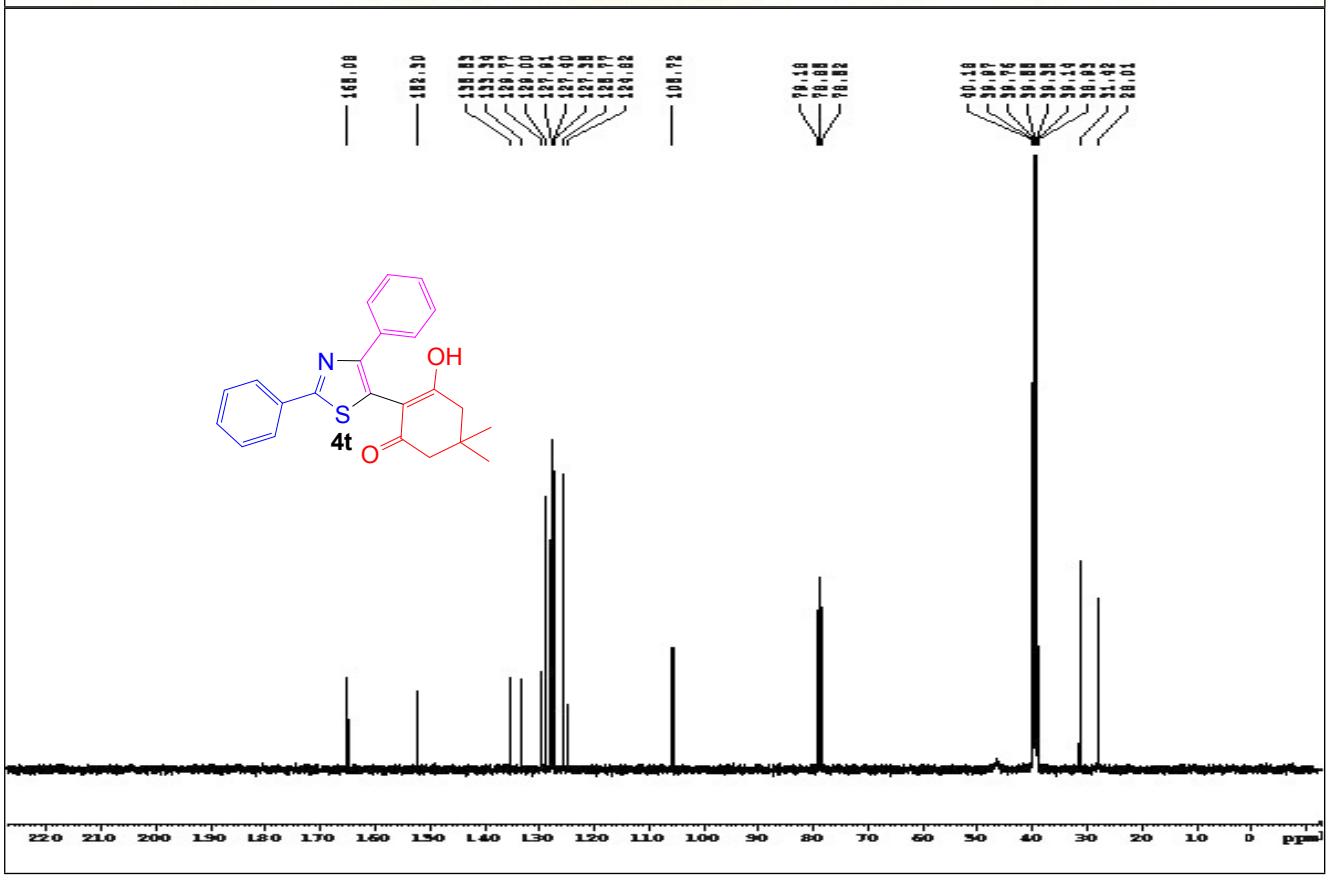

ppm
8.0660
7.9360
7.8751
7.8603
7.8198
7.8081
7.4268
7.4122
7.3931
7.3784
7.2610
4.8635
N
O
S
Cl
O
4r
Integral
2.2222
7.1462
6.1147
1.0000
ppm
8
6
4
2
0

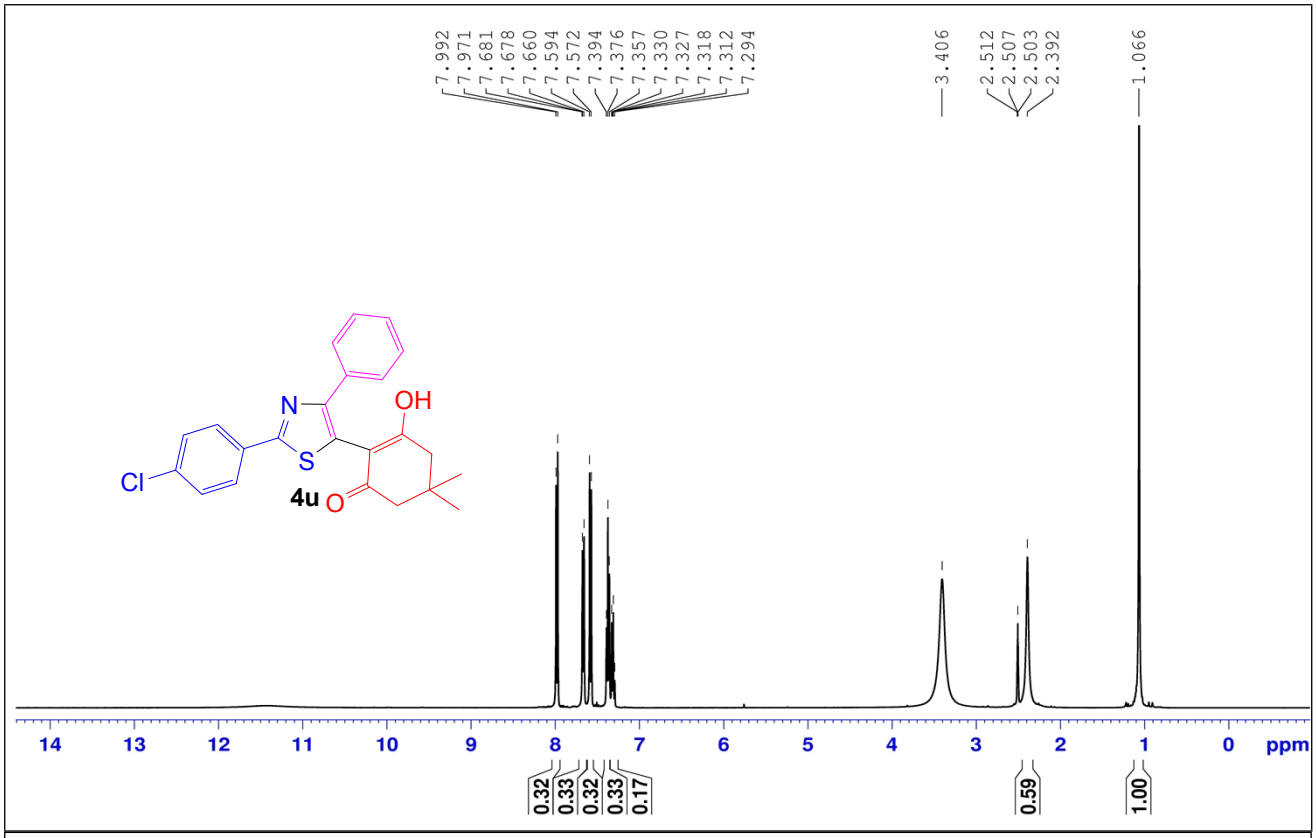

ppm
196.432
165.463
158.084
141.576
136.420
136.257
133.913
131.837
129.165
129.084
128.828
128.668
127.812
124.050
123.244
77.282
77.028
76.774
53.647
N
O
S
Cl
O
4r
ppm
175
150
125
100
75
50
25
0

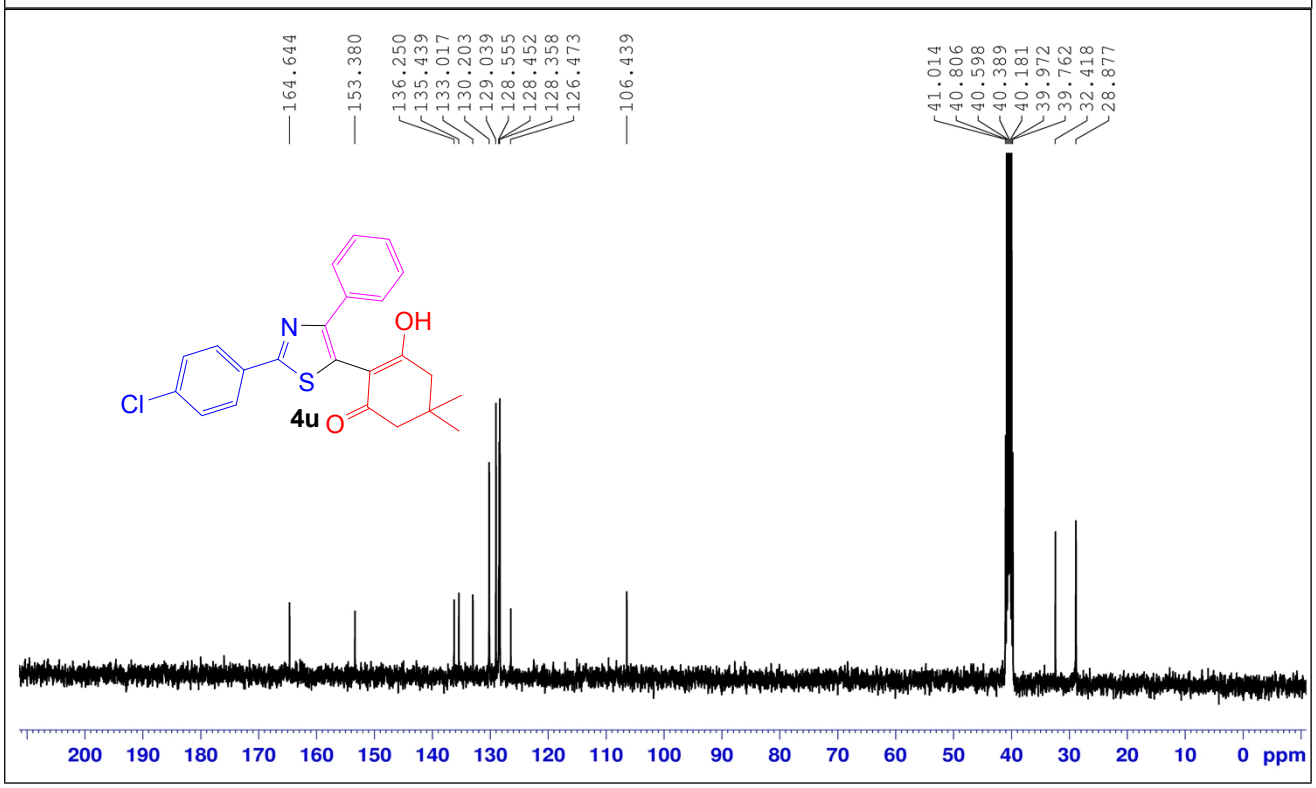

ppm
7.9455
7.9280
7.9130
7.9030
7.8887
7.7707
7.7533
7.7388
7.4553
7.4216
7.4059
7.3820
7.3671
7.3517
7.3011
7.2864
7.2719
7.0909
7.0734
3.8376
2.5035
2.5001
2.4967
OH
N
S
MeO
O
4s
Integral
2.0026
2.0791
3.5402
2.0658
1.0000
1.9958
3.0466
ppm
8
6
4
2
0

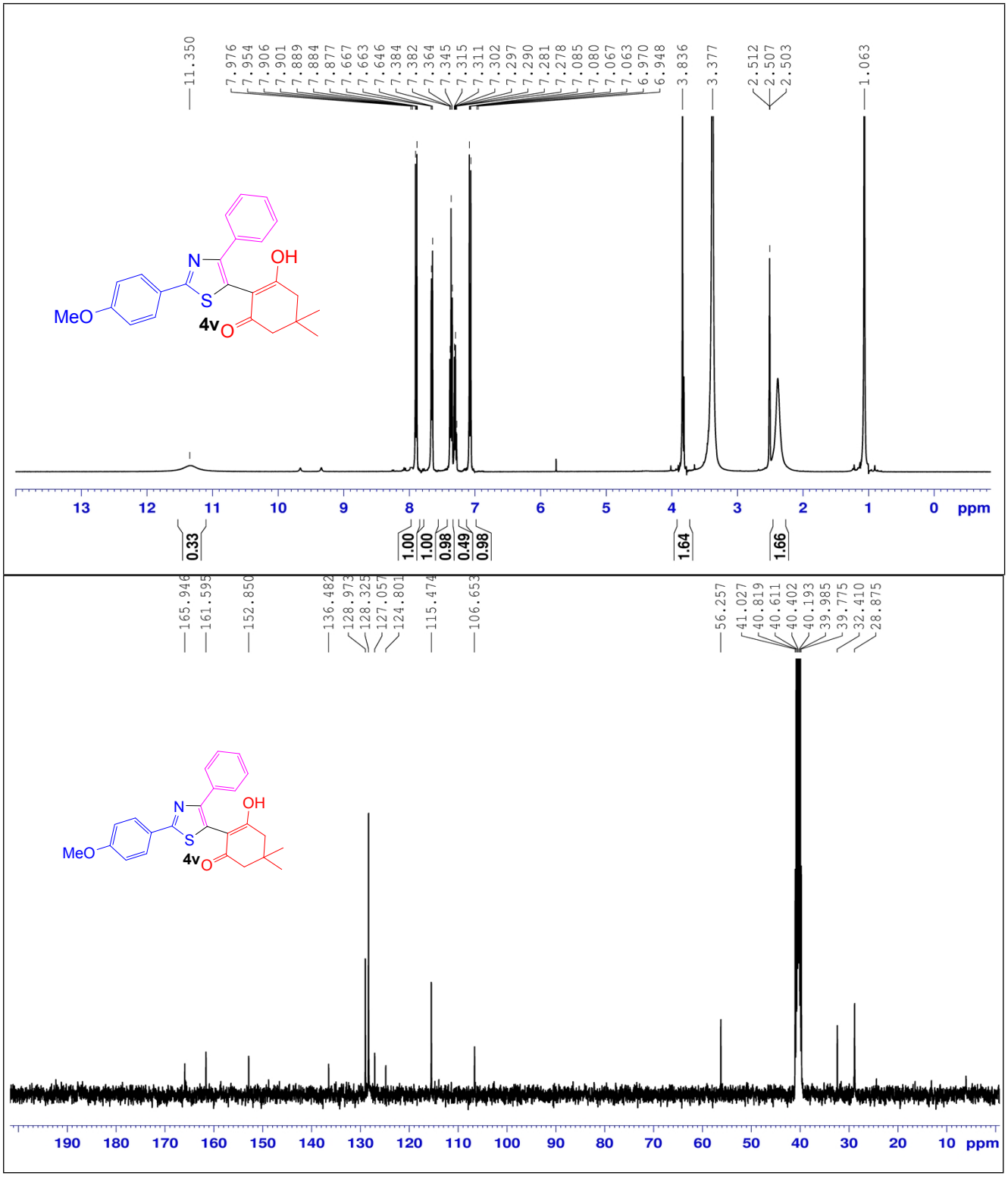

ppm
165.146
160.850
151.591
135.655
131.752
128.077
127.604
127.541
127.411
125.850
122.662
120.623
120.104
114.587
101.502
55.354
39.996
39.829
39.662
39.495
39.328
39.162
38.995
OH
N
S
MeO
O
4s
ppm
175
150
125
100
75
50
25
0

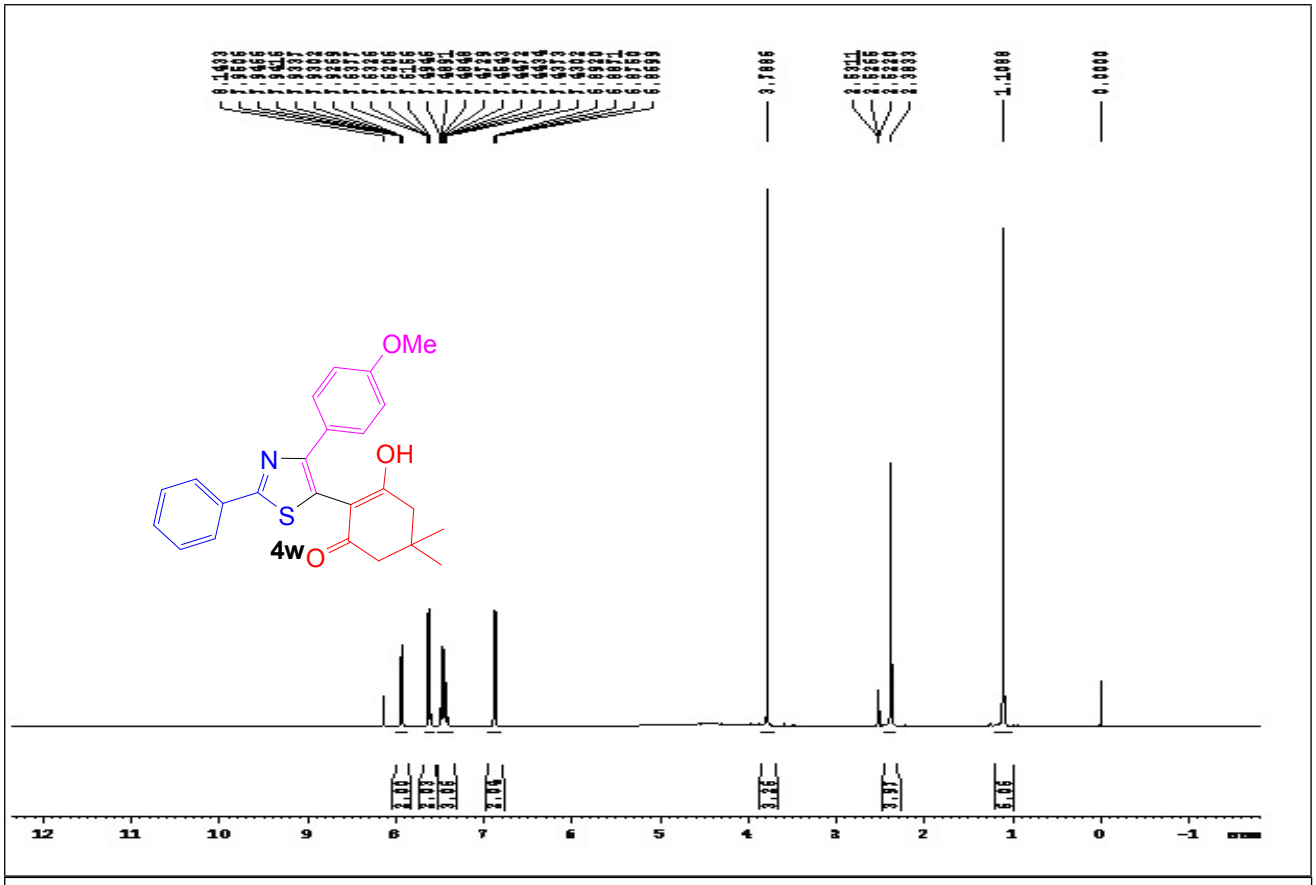

11.389
7.970
7.966
7.950
7.947
7.671
7.653
7.526
7.508
7.504
7.500
7.390
7.372
7.369
7.353
7.324
7.306
3.379
2.509
2.505
2.500
2.087
1.063
N
S
OH
O
4t
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.32
1.00
1.01
1.57
1.04
0.54
1.86
3.15

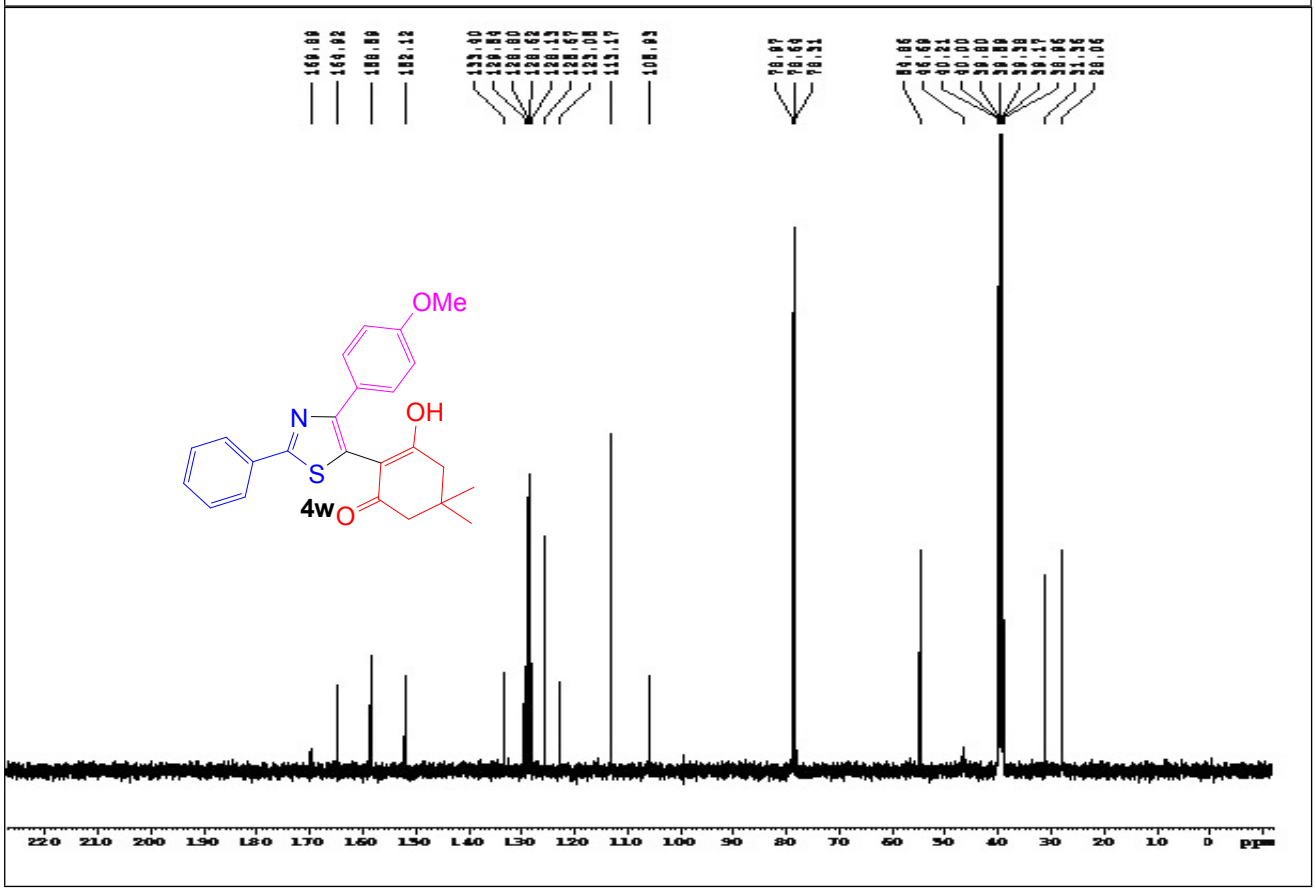

N
S
OH
O
4t
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

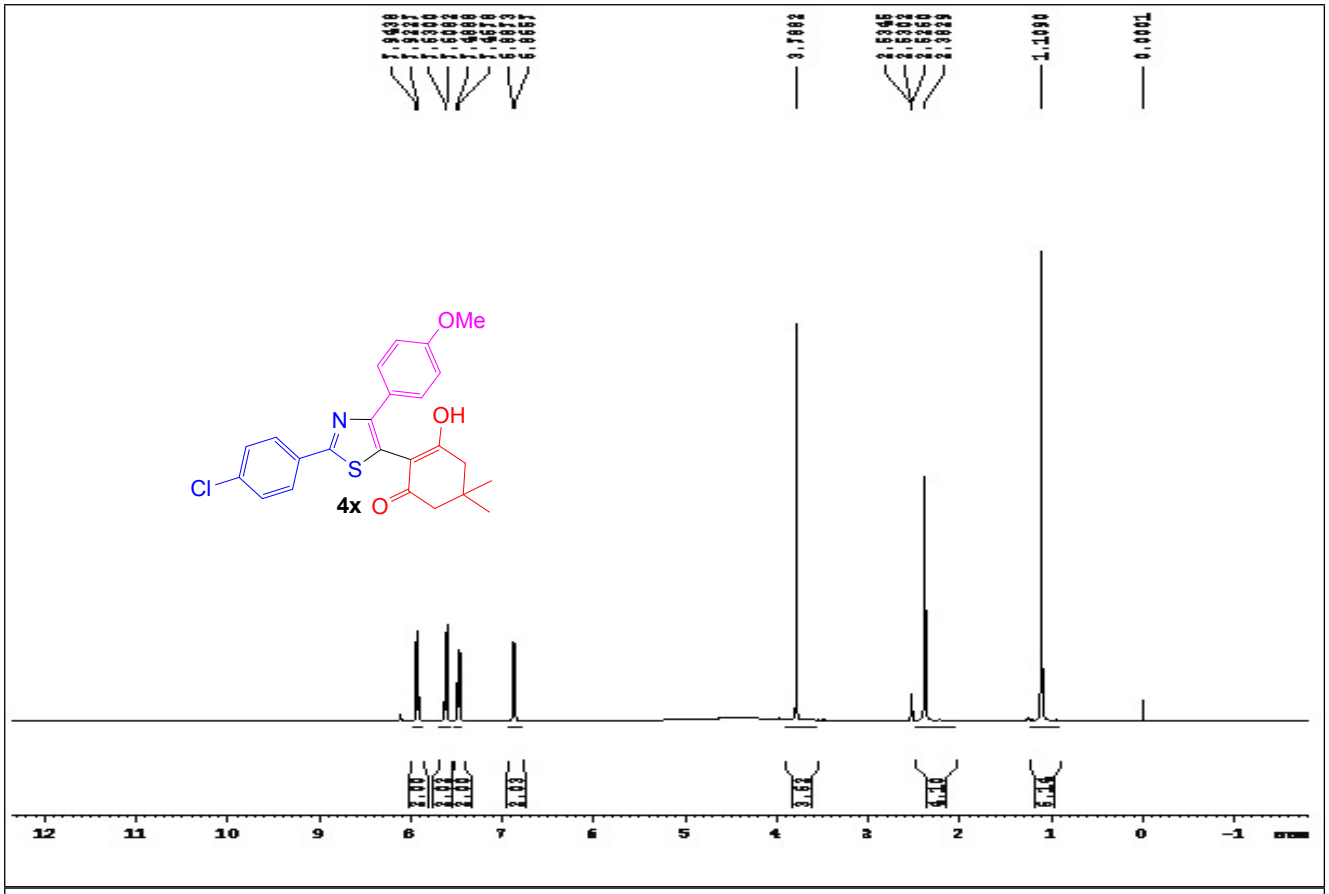

7.992
7.971
7.681
7.678
7.660
7.594
7.572
7.394
7.376
7.357
7.330
7.327
7.318
7.312
7.294
3.406
2.512
2.507
2.503
2.392
1.066
N
OH
S
Cl
4u
O
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.32
0.33
0.32
0.33
0.17
0.59
1.00

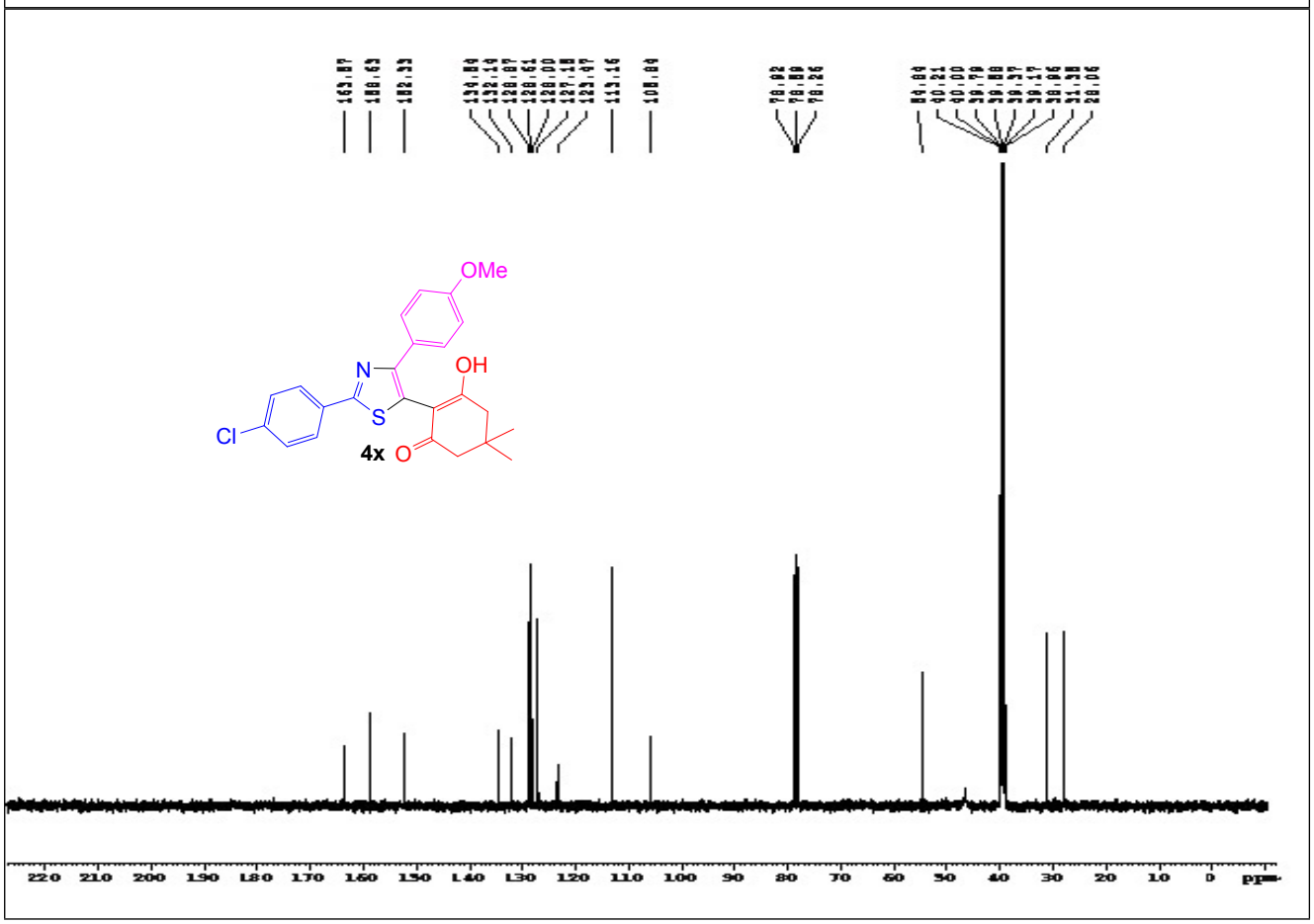

164.644
153.380
136.250
135.439
133.017
130.203
129.039
128.555
128.452
128.358
126.473
106.439
41.014
40.806
40.598
40.389
40.181
39.972
39.762
32.418
28.877
N
OH
S
Cl
4u
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


11.350
7.976
7.954
7.906
7.901
7.889
7.884
7.877
7.667
7.663
7.646
7.384
7.382
7.364
7.345
7.315
7.311
7.302
7.297
7.290
7.281
7.278
7.085
7.080
7.067
7.063
6.970
6.948
3.836
3.377
2.512
2.507
2.503
1.063
N
OH
S
MeO
4v
O
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.33
1.00
1.00
0.98
0.49
0.98
1.64
1.66
165.946
161.595
152.850
136.482
128.973
128.325
127.057
124.801
115.474
106.653
56.257
41.027
40.819
40.611
40.402
40.193
39.985
39.775
32.410
28.875
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


OMe
N
OH
S
4w
O


OMe
N
OH
S
4w
O


OMe
N
OH
S
Cl
4x O


OMe
N
OH
S
Cl
4x O