Electronic Supplementary Material (ESI) for RSC Advances.

Supporting information

L-proline catalyzed multicomponent reactions: Facile access to 2*H*-benzo[*g*]pyrazolo[3,4-*b*]quinoline-5,10(4*H*,11*H*)-dione derivatives

Shaik Karamthulla,[a] Suman Pal,[a] Tasneem Parvin,[b] and Lokman H. Choudhury*[a]

[a]Department of Chemistry, Indian Institute of Technology Patna, Patna-800013, India.
[b]Department of Chemistry, National Institute of Technology Patna, Ashok Rajpath, Patna -800005, India.

Email: lokman@iitp.ac.in

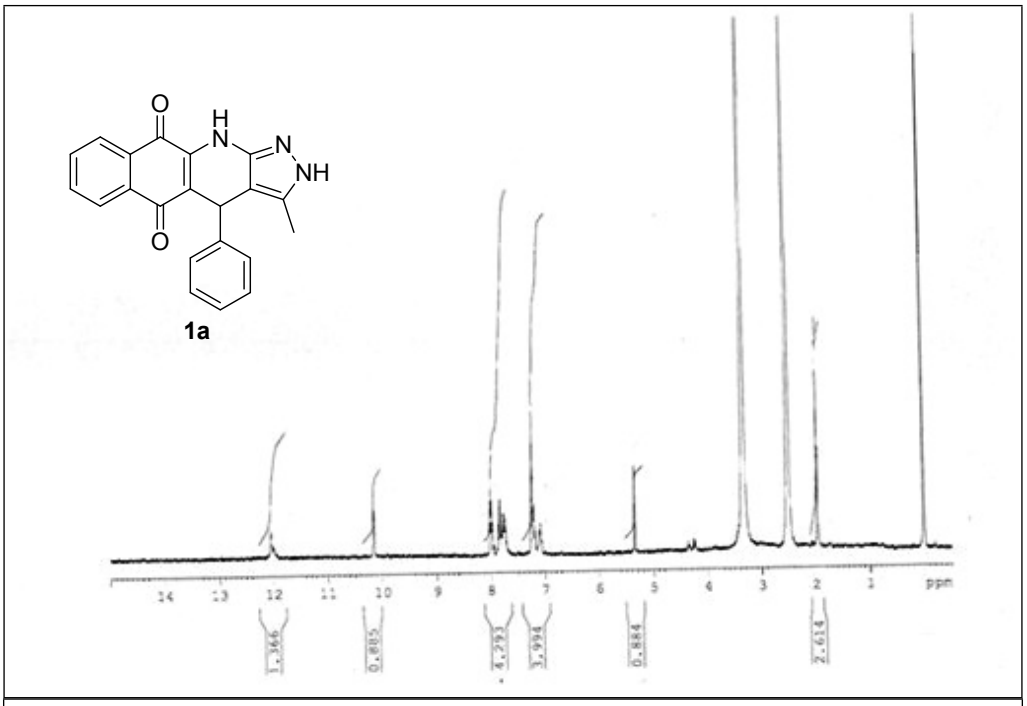
Table of contents

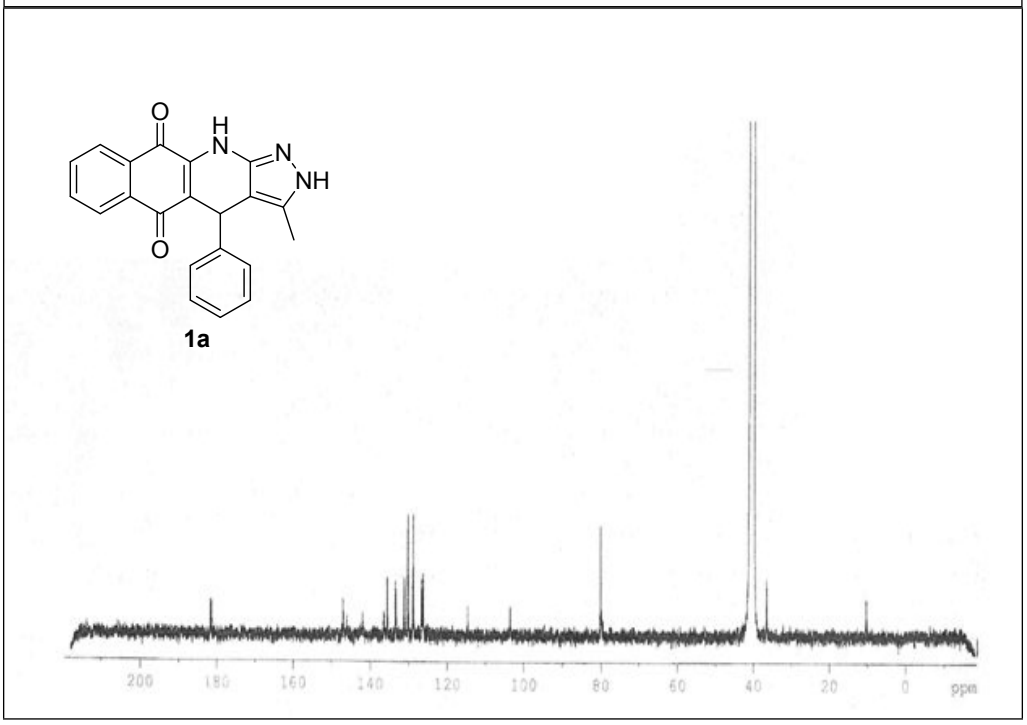
1. General information.

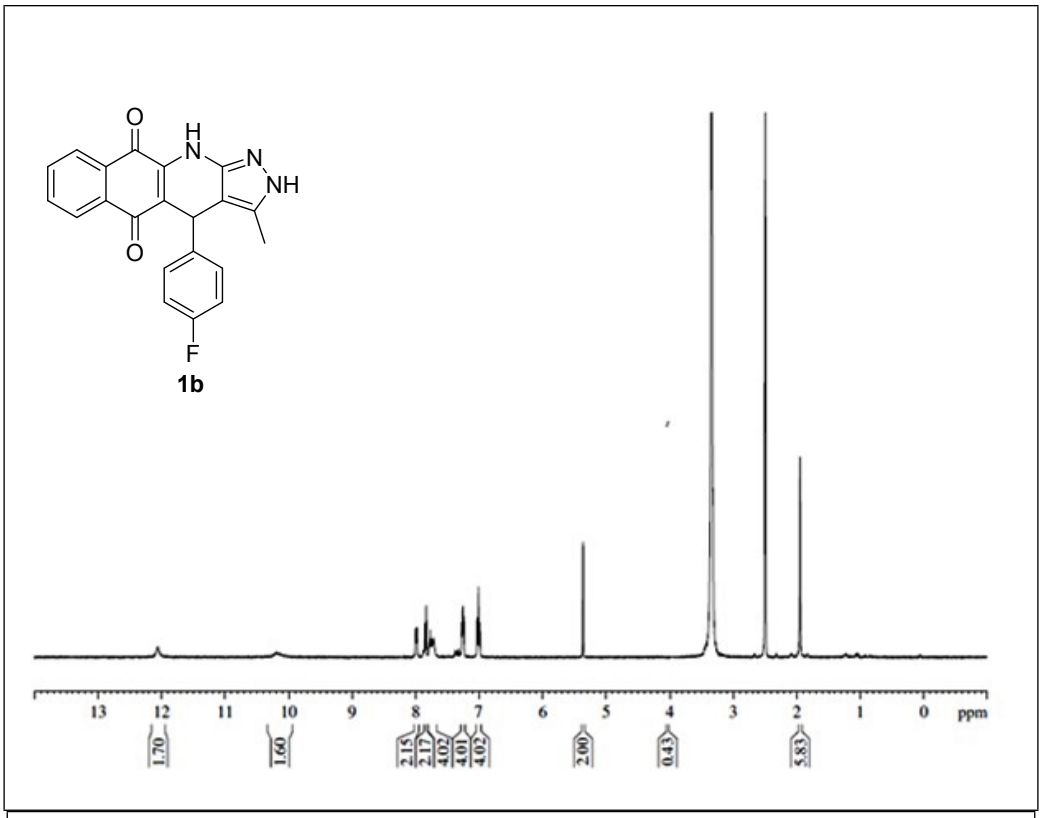
All reagents and chemicals required for the reactions were procured from commercial sources and used without further purification. IR spectra were recorded in Shimadzu FTIR spectrophotometer. ^{1}H NMR spectra and ^{13}C NMR spectra were recorded on 500, 400, 300 MHz spectrometer in DMSO-d6 using TMS as internal reference. Elemental analyses were carried out in a Perkin Elmer 2400 automatic CHN analyzer or Elementer Vario EL III. All new compounds were characterized by recording melting point without correction, ^{1}H NMR, ^{13}C NMR and elemental analysis.

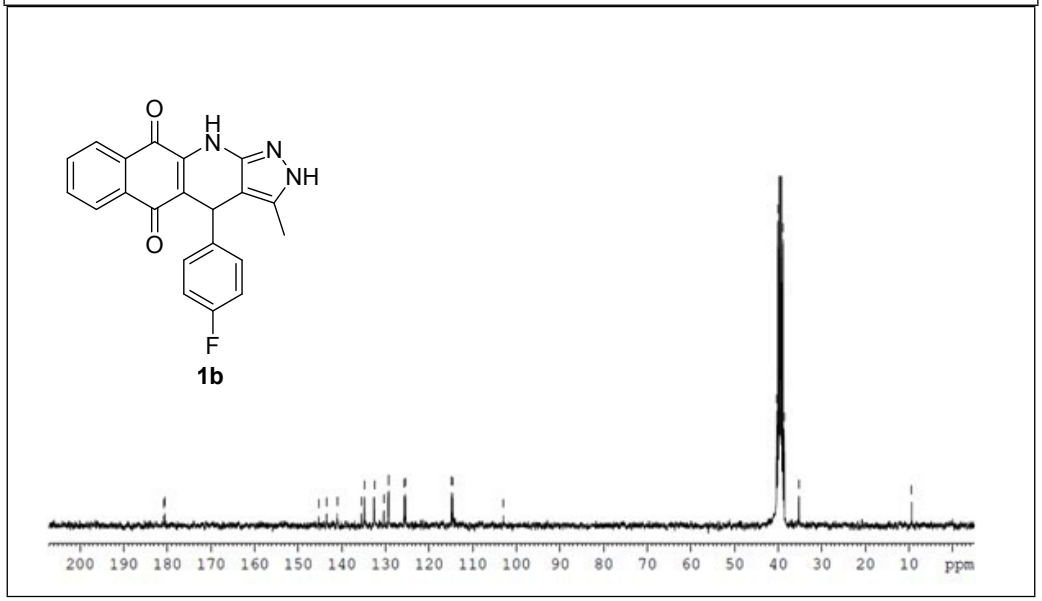
2. Typical experimental procedure for the synthesis of 1c: To a stirred mixture of 4-chlorobenzaldehyde (1 mmol) and 2-hydroxynaphthoquinone (1 mmol) in ethanol (5 ml), was added L-proline (20 mol %) and the reaction mixture was stirred at reflux temperature for 30 minutes. Then 3-amino-5-methylpyrazole (1mmol) was added to it. The resulting mixture was stirred until the reaction was completed as indicated by TLC. The resulting solid was collected by filtration and washed with ethanol to afford the product. The resulting product was pure enough for characterization.

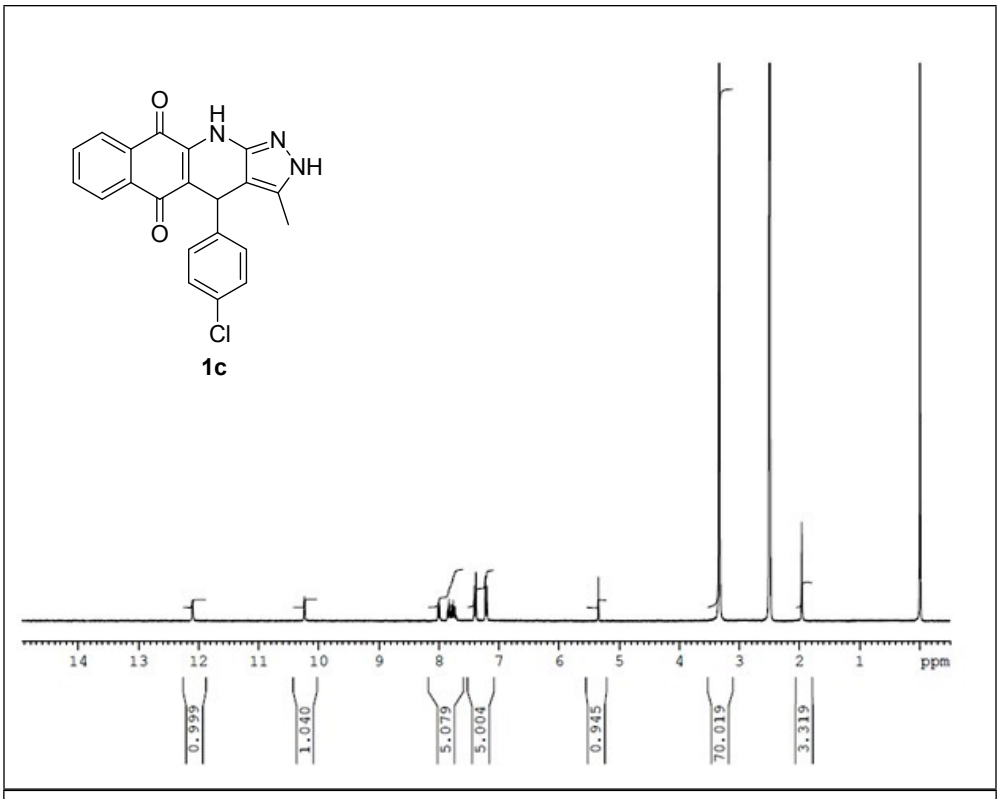
3. Copies of ^{1}H and ^{13}C NMR spectra of compounds

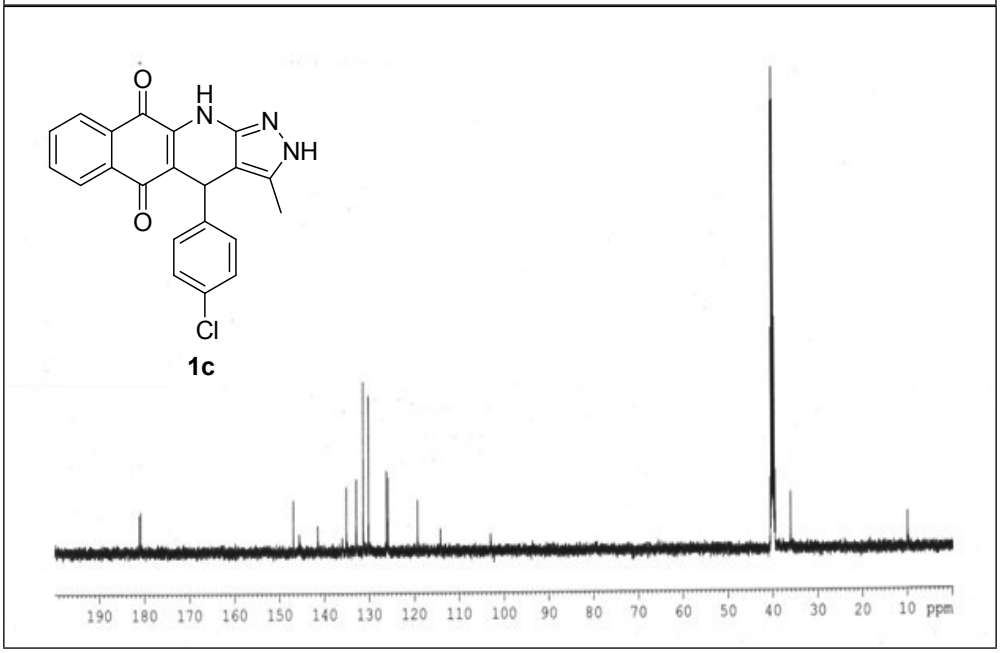


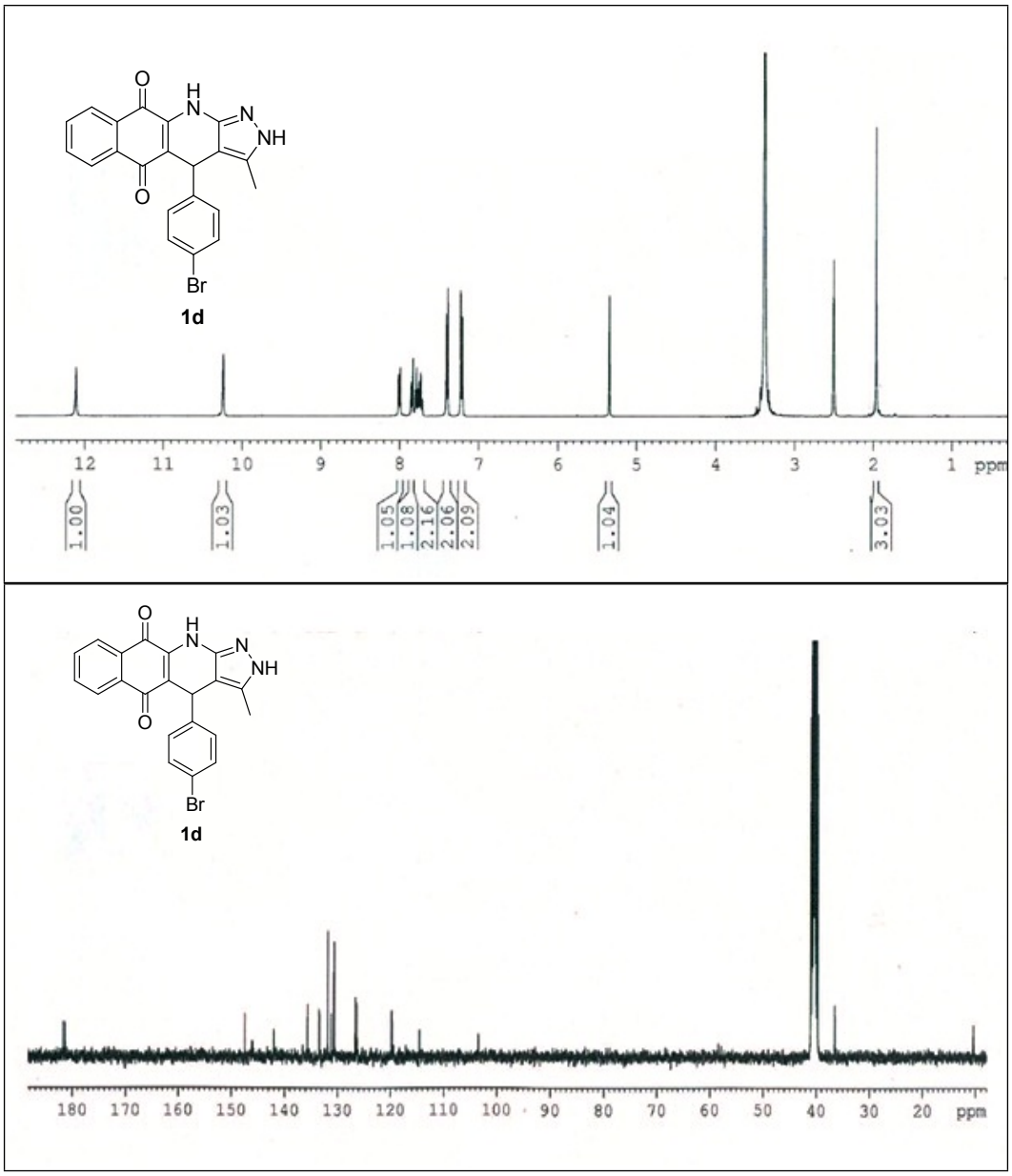


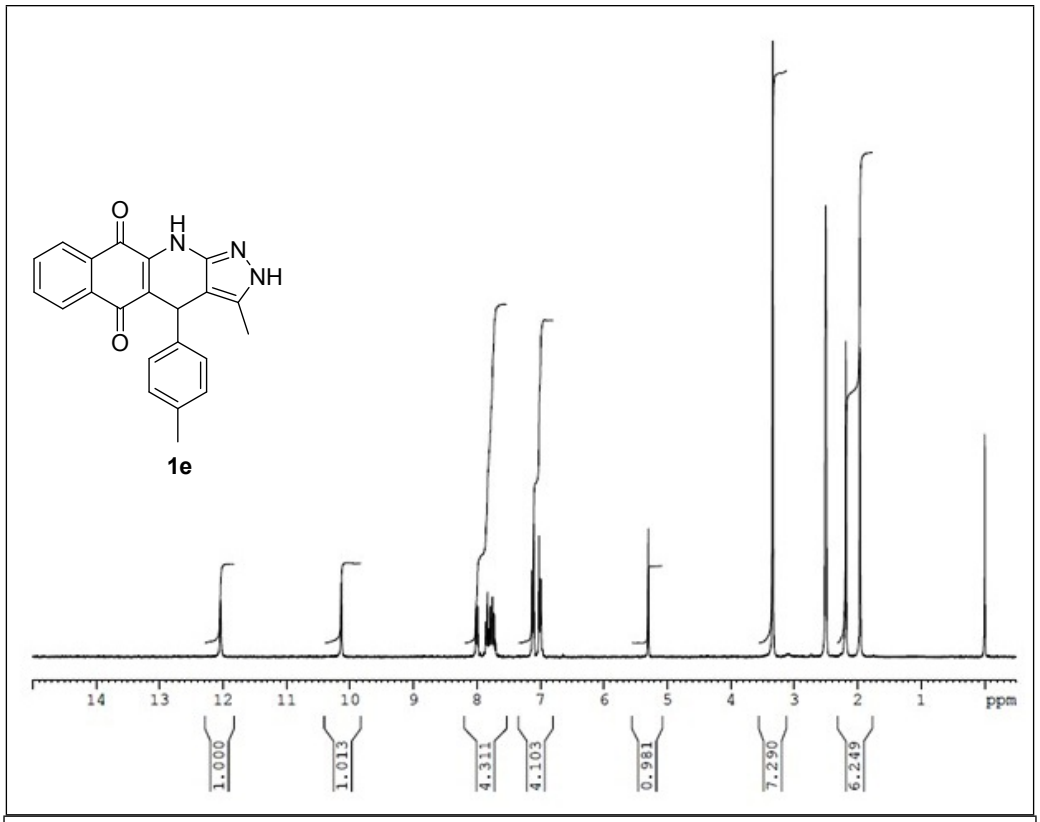

O
H
N
N
NH
O
F
1b
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.70 1.60 2.15 2.17 4.02 4.01 4.02 2.00 0.43 5.83

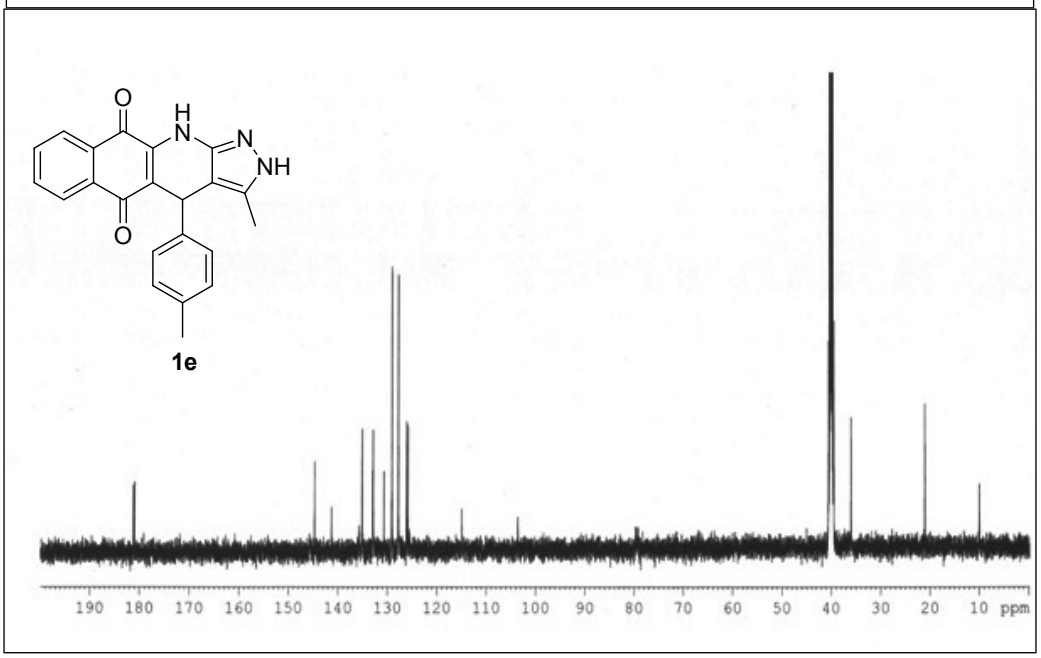

O
H
N
N
NH
O
F
1b
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

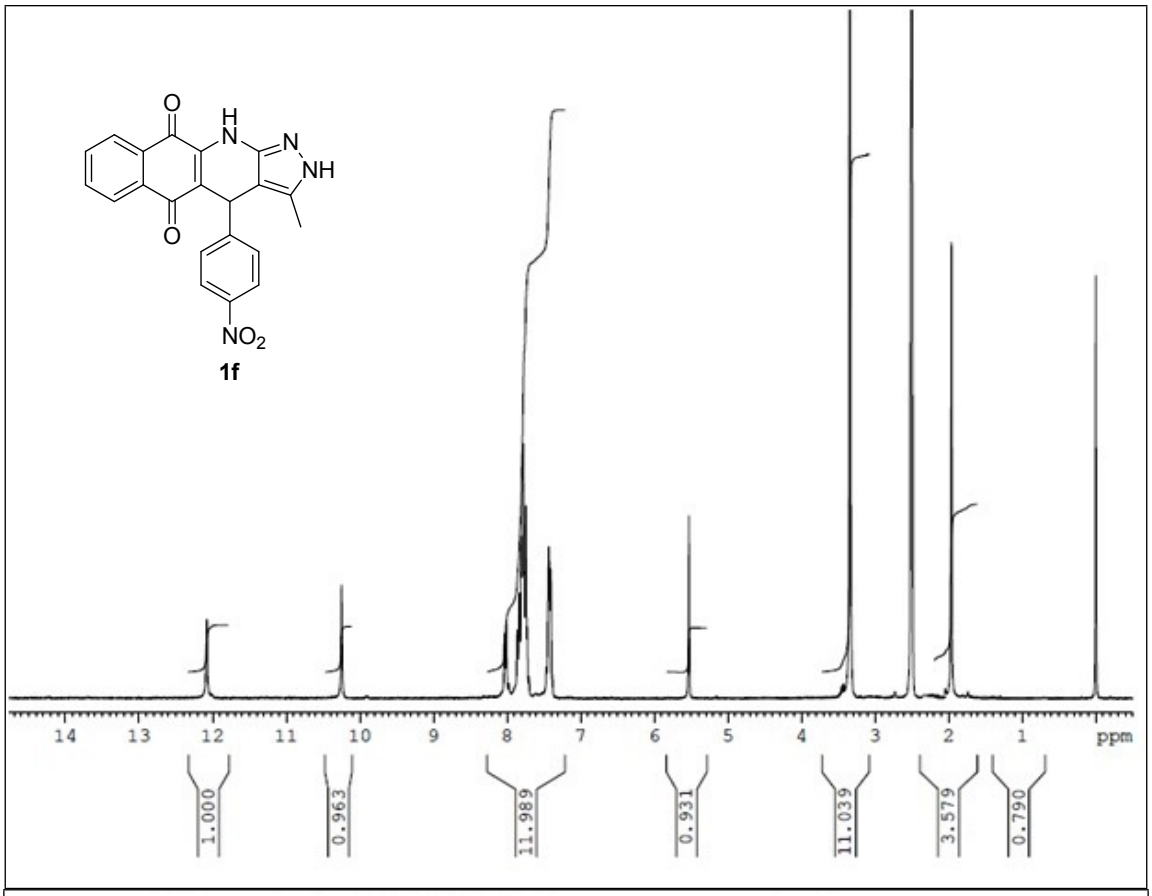

O
H
N
N
NH
O
Cl
1c
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.999
1.040
5.079
5.004
0.945
70.019
3.319


O
H
N
N
NH
O
Cl
1c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

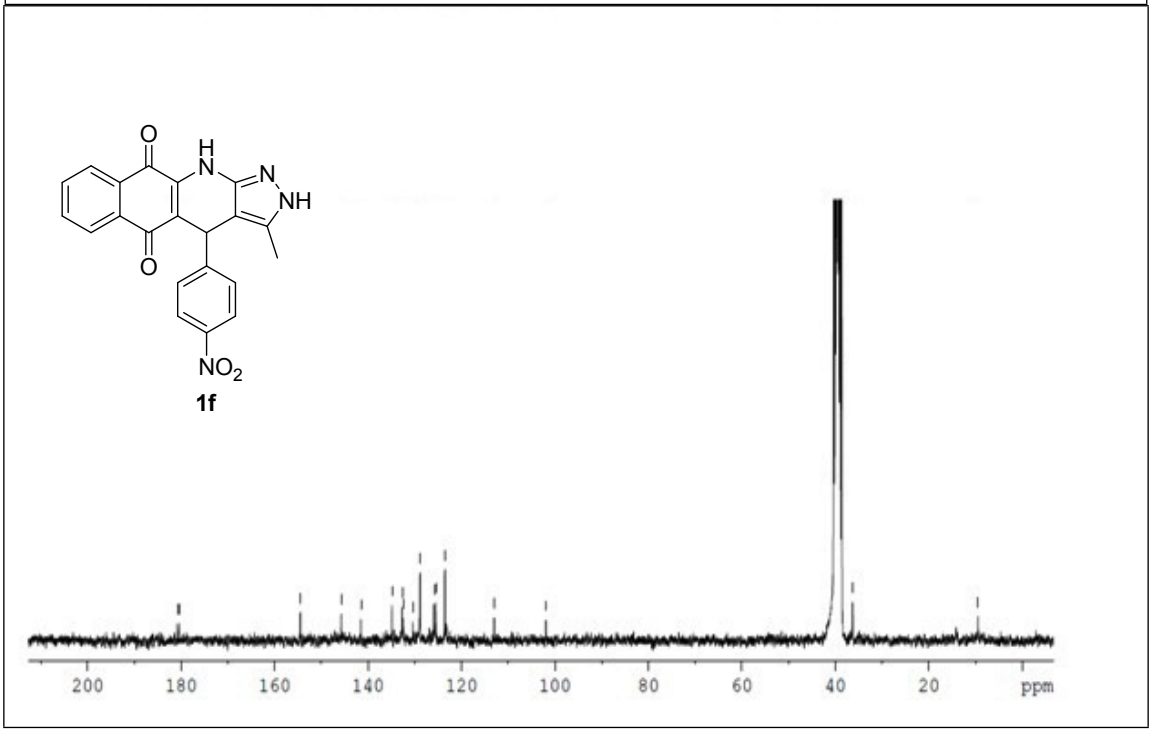

O
H
N
N
NH
O
Br
1d
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.03
1.05
1.08
2.16
2.06
2.09
1.04
3.03
O
H
N
N
NH
O
Br
1d
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


O
H
N
N
NH
O
1e
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.000
1.013
4.311
4.103
0.981
7.290
6.249

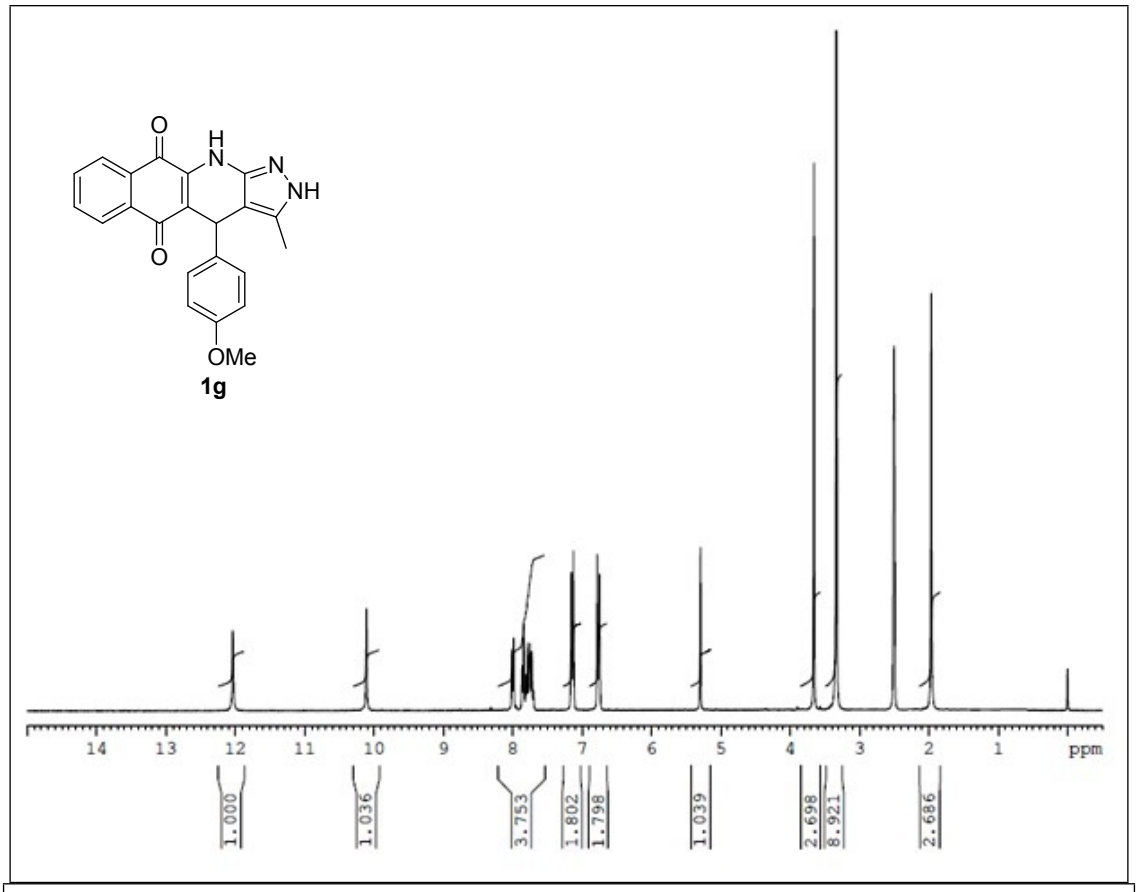

O
H
N
N
NH
O
1e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

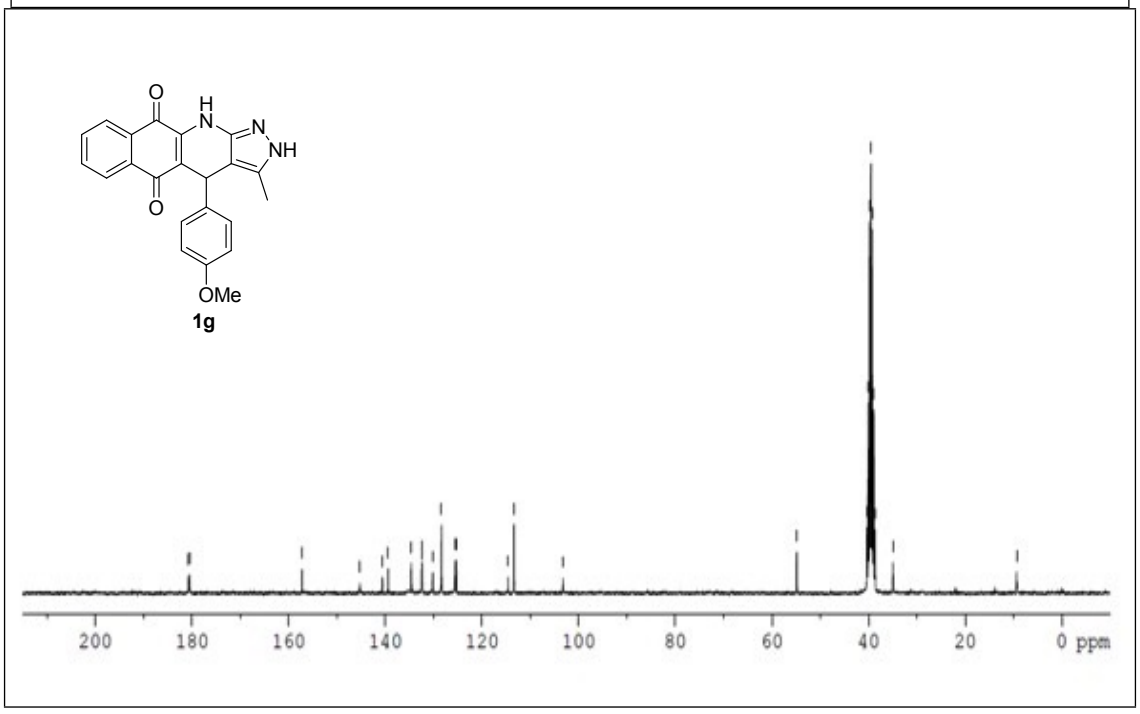

O
H
N
N
NH
O
NO2
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.000
0.963
11.989
0.931
11.039
3.579
0.790

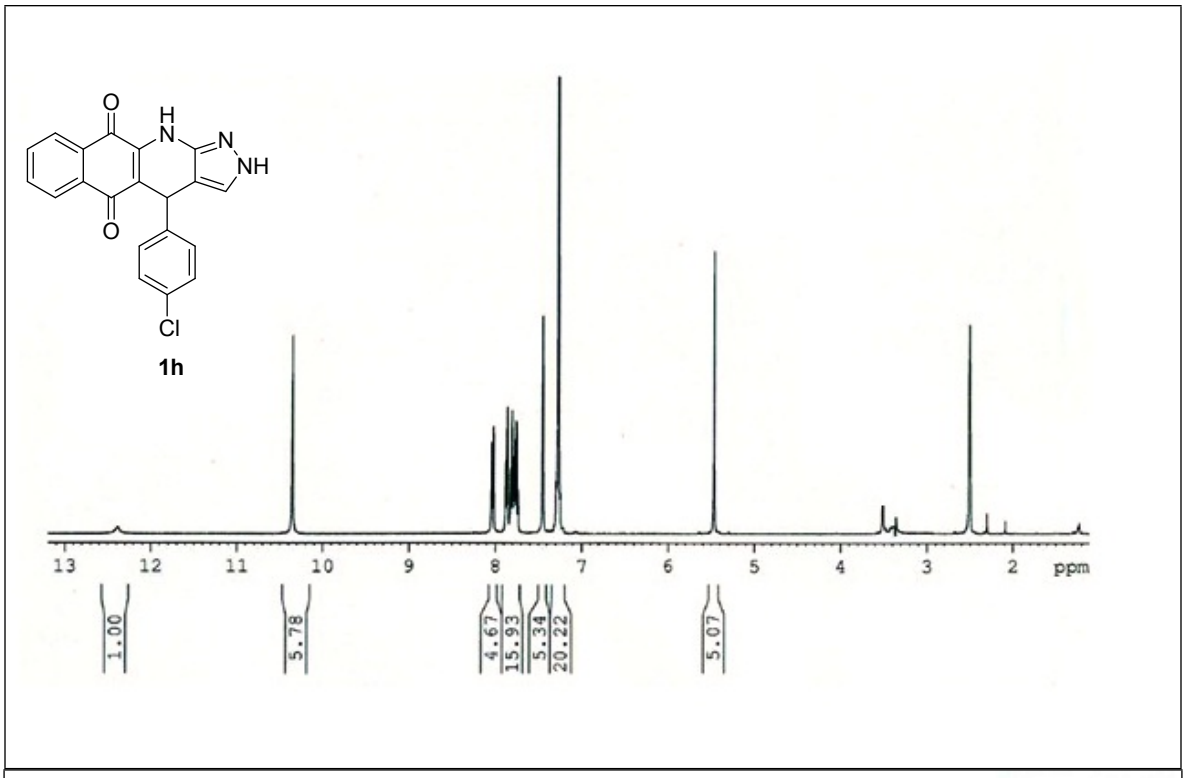
1f

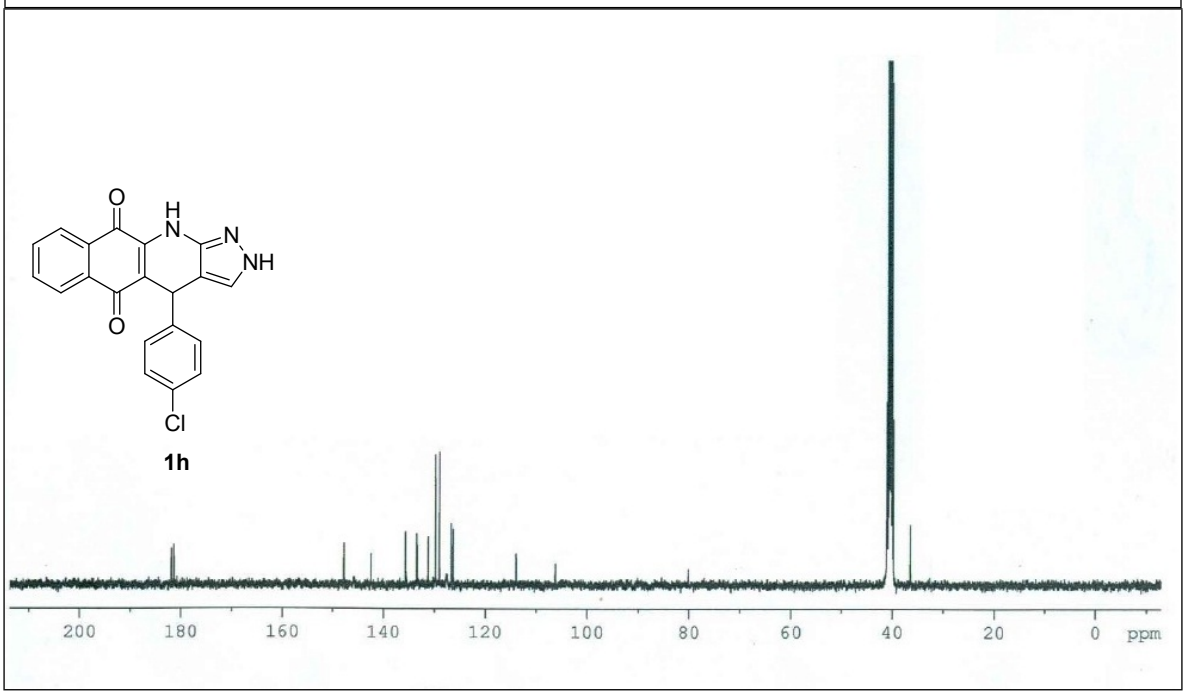

O
H
N
N
NH
O
NO2
200
180
160
140
120
100
80
60
40
20
ppm

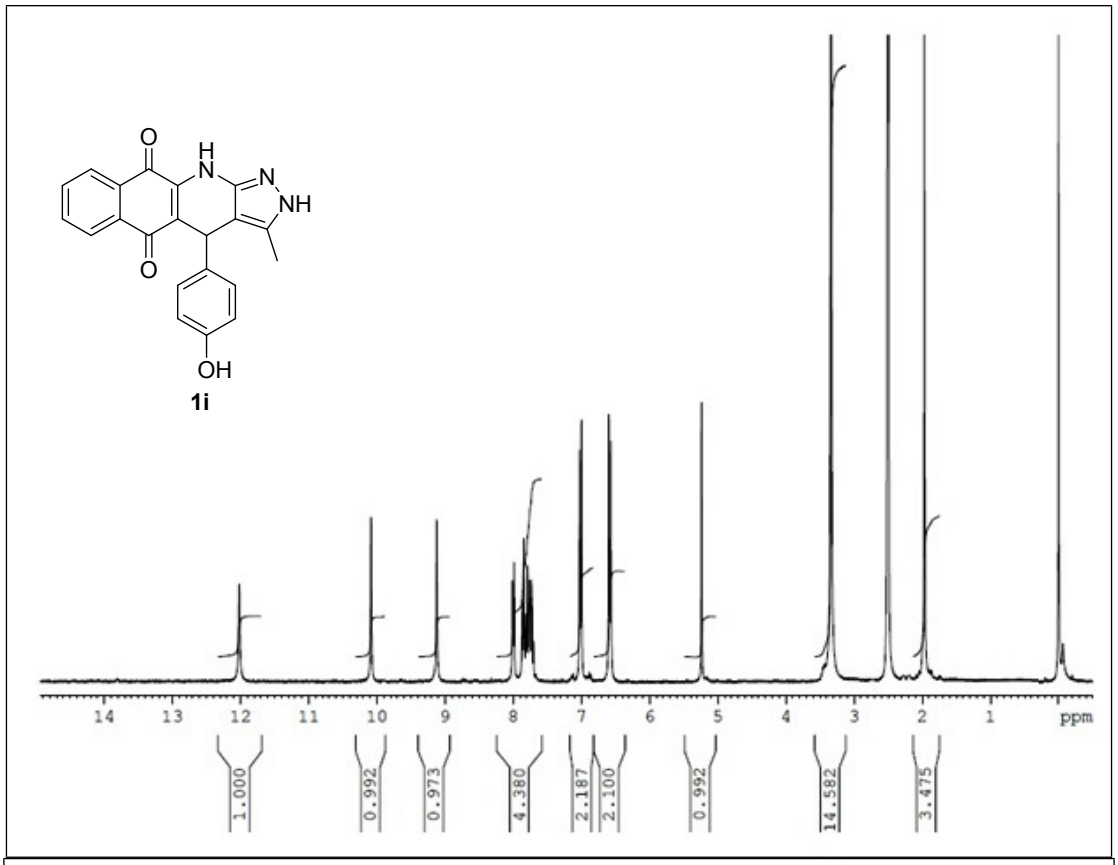
1f

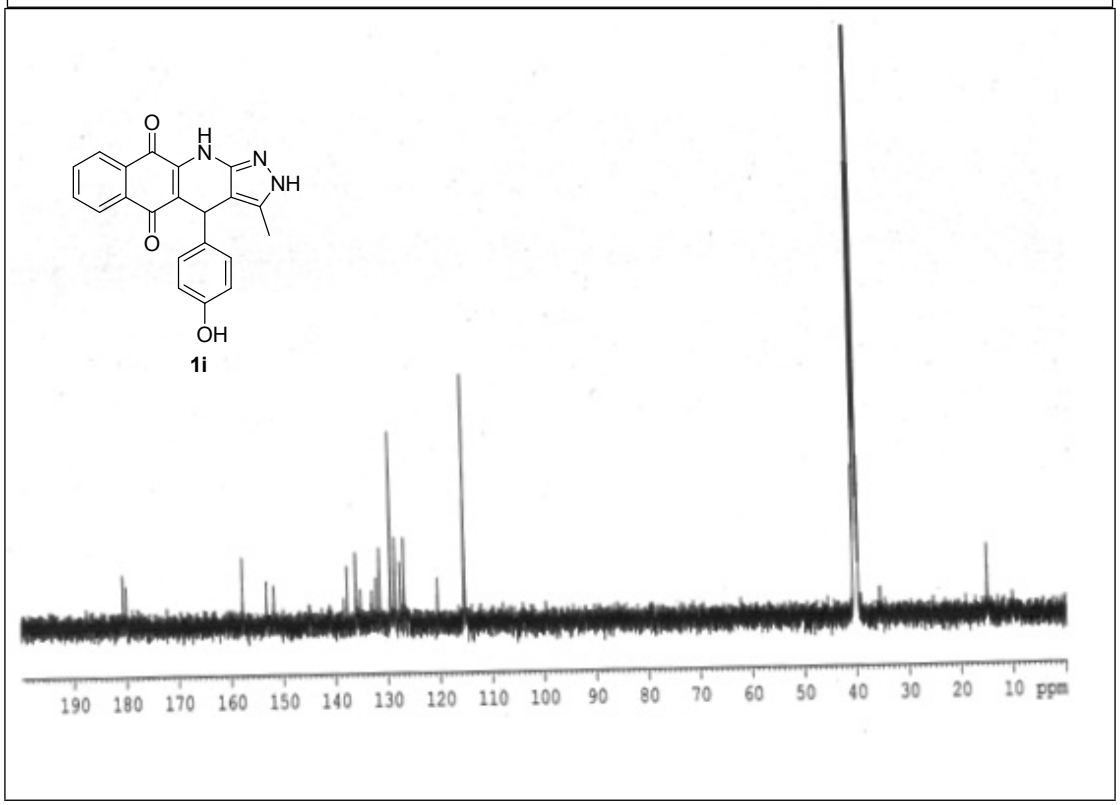

O
H
N
N
NH
O
OMe
1g
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.000
1.036
3.753
1.802
1.798
1.039
2.698
8.921
2.686

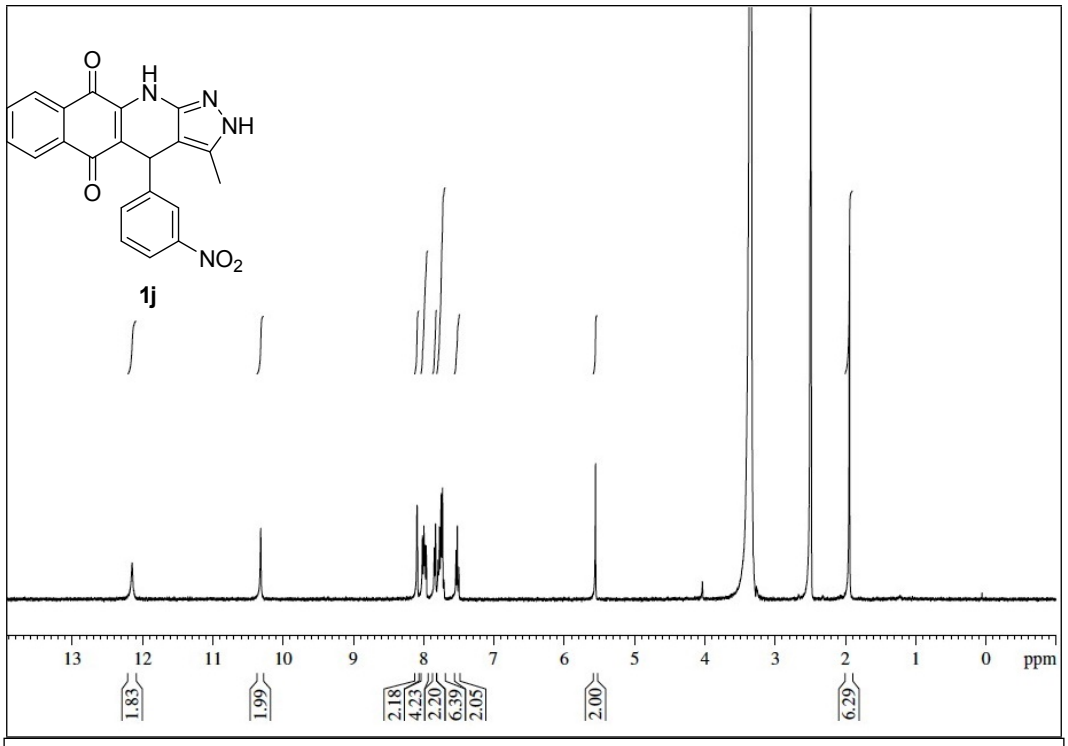

O
H
N
N
NH
O
OMe
1g
200
180
160
140
120
100
80
60
40
20
0 ppm

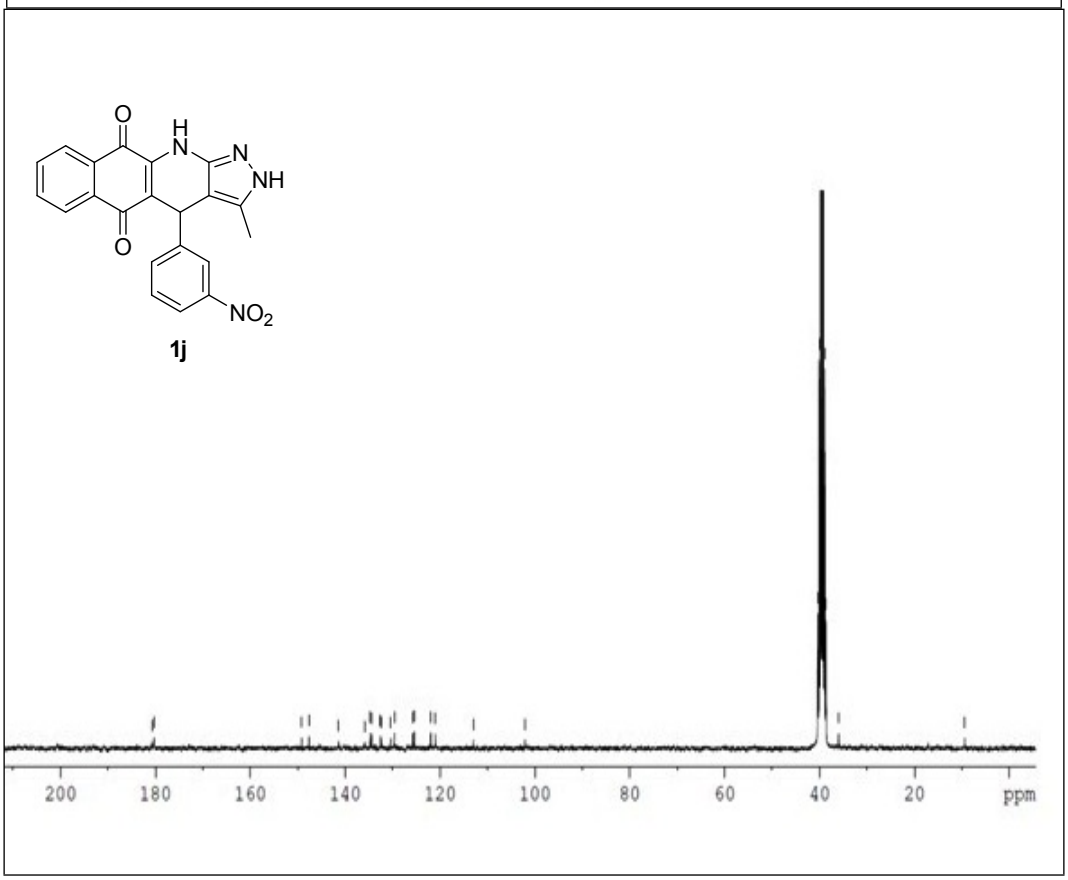

O
H
N
N
NH
O
Cl
1h
13
12
11
10
9
8
7
6
5
4
3
2
ppm
1.00
5.78
4.67
15.93
5.34
20.22
5.07

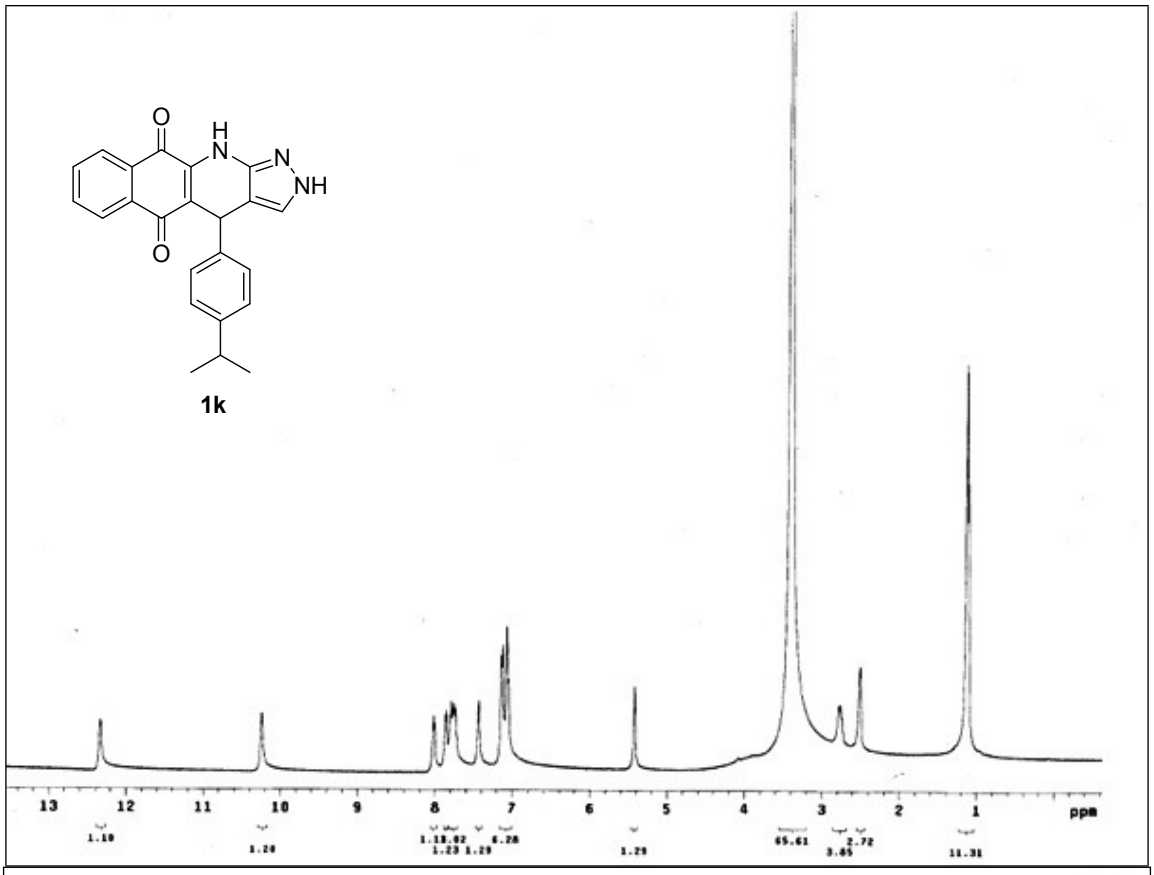

O
H
N
N
NH
O
Cl
1h
200
180
160
140
120
100
80
60
40
20
0
ppm

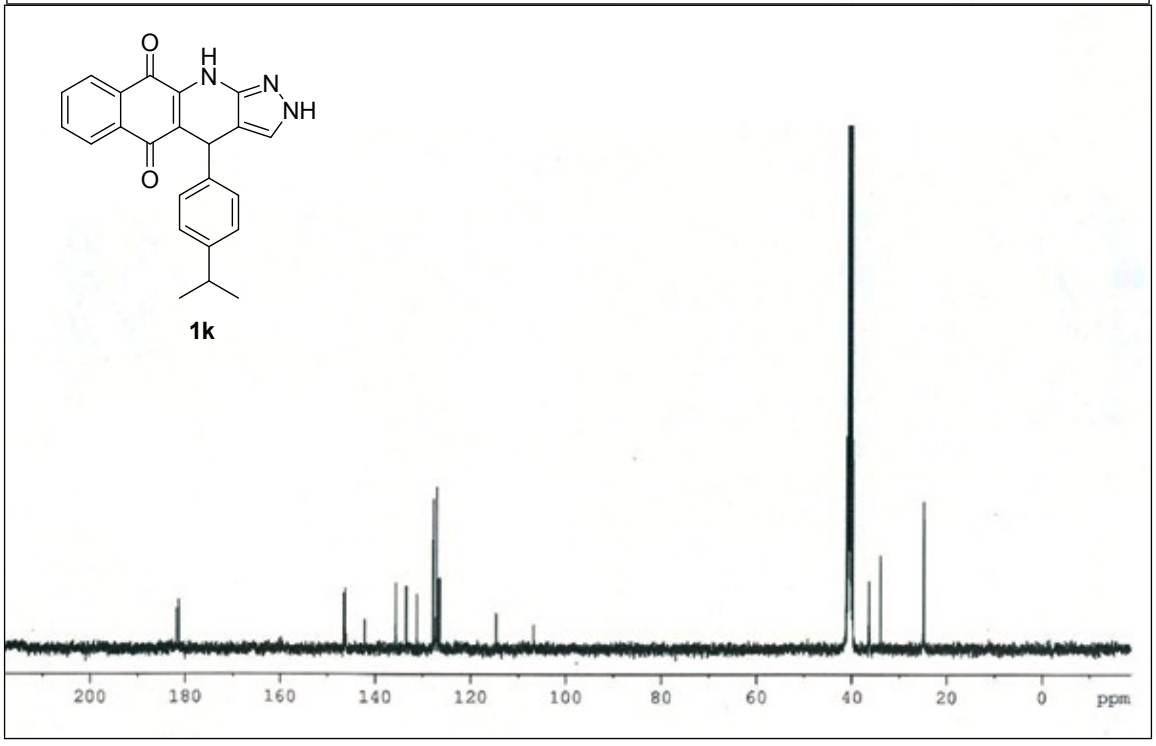

O
H
N
N
NH
O
OH
1i
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.000
0.992
0.973
4.380
2.187
2.100
0.992
14.582
3.475

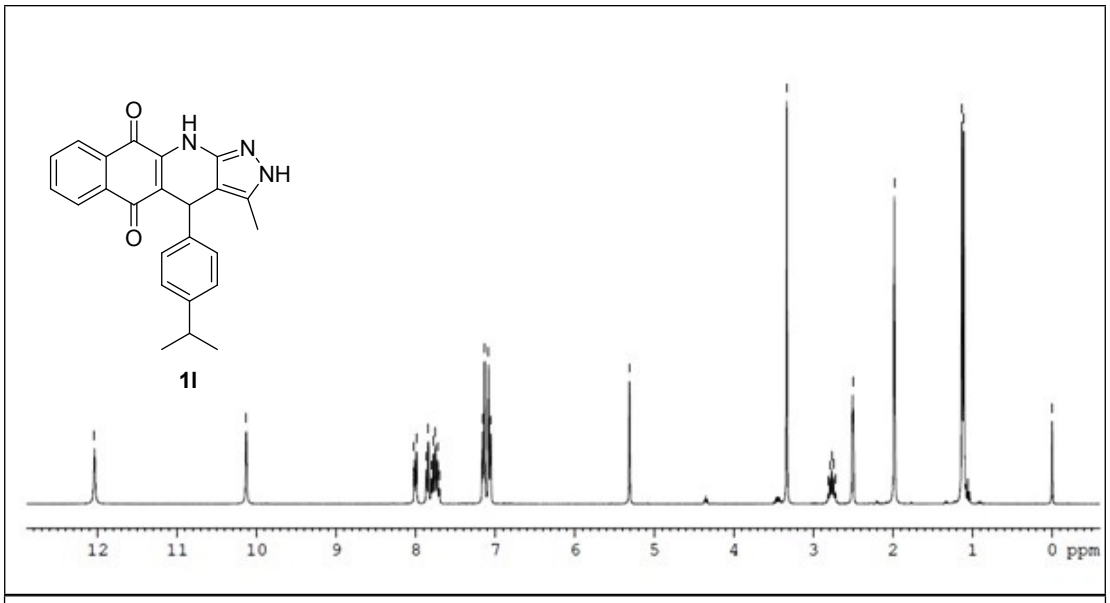

O
H
N
N
NH
O
OH
1i
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


O
H
N
N
NH
O
NO2
1j
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.83
1.99
2.18
4.23
2.20
6.39
2.05
2.00
6.29

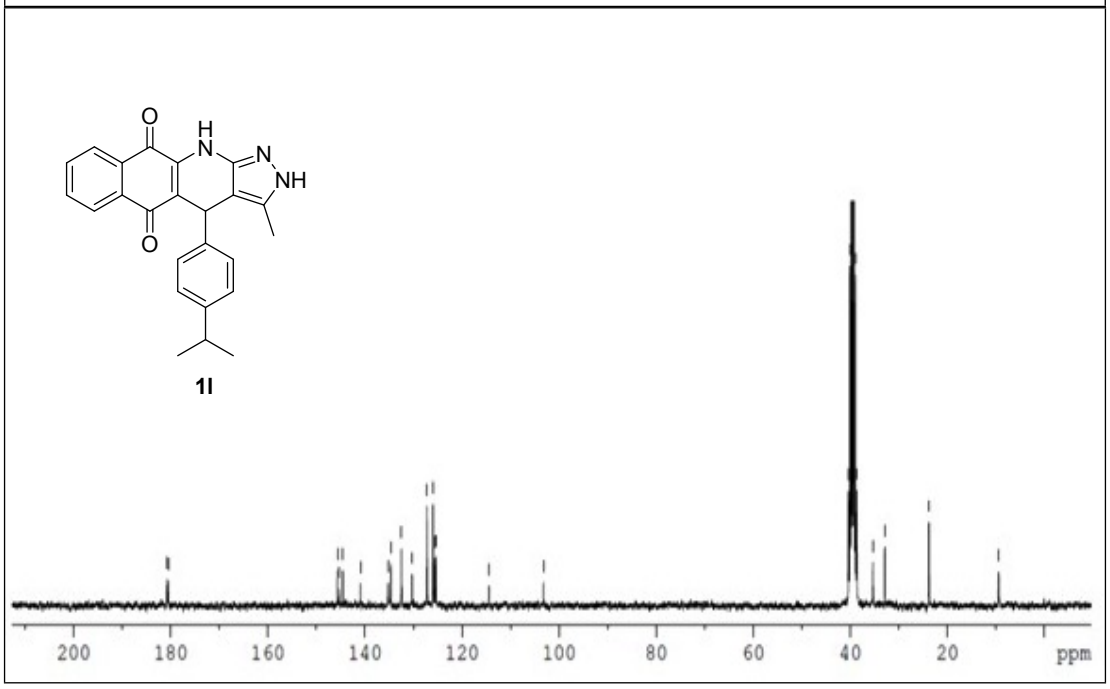

O
H
N
N
NH
O
NO2
1j
200
180
160
140
120
100
80
60
40
20
ppm


O
H
N
N
NH
O
1k
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm

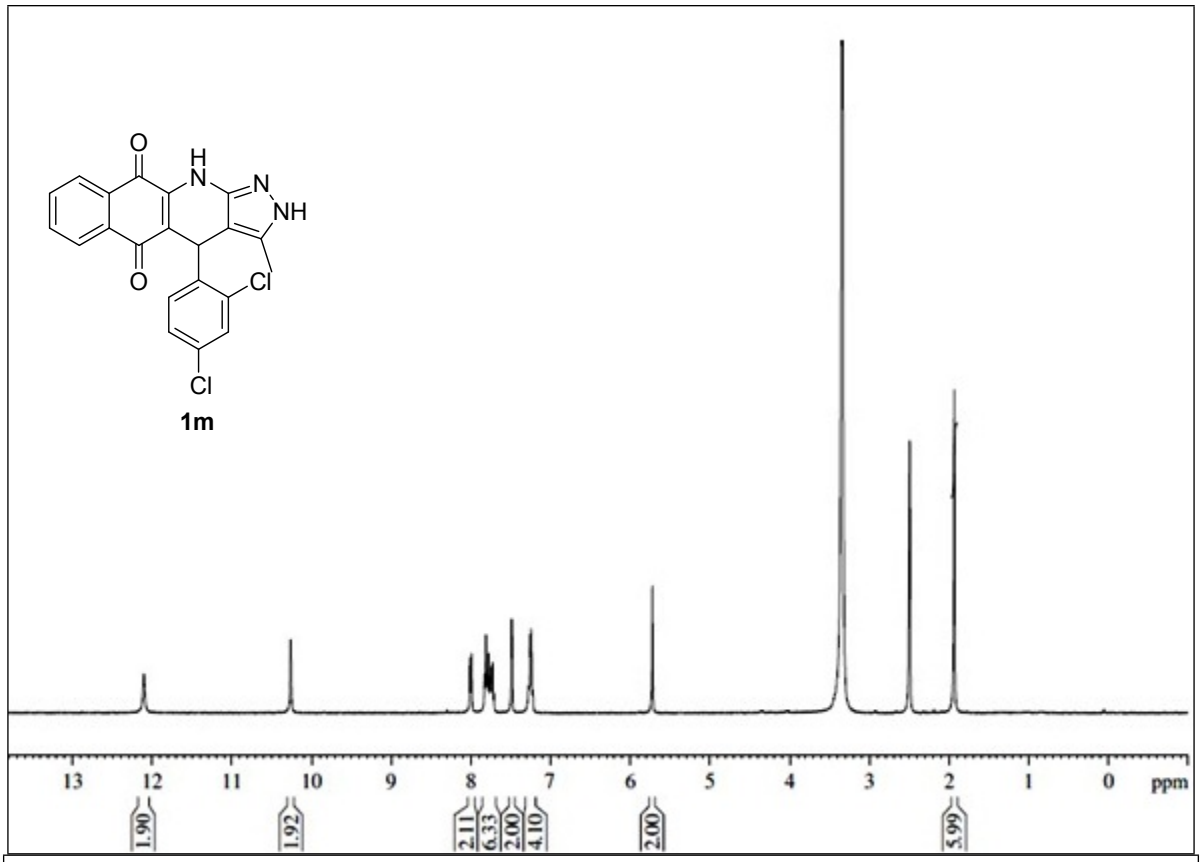

O
H
N
N
NH
O
1k
200
180
160
140
120
100
80
60
40
20
0
ppm


O
H
N
N
NH
O
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm

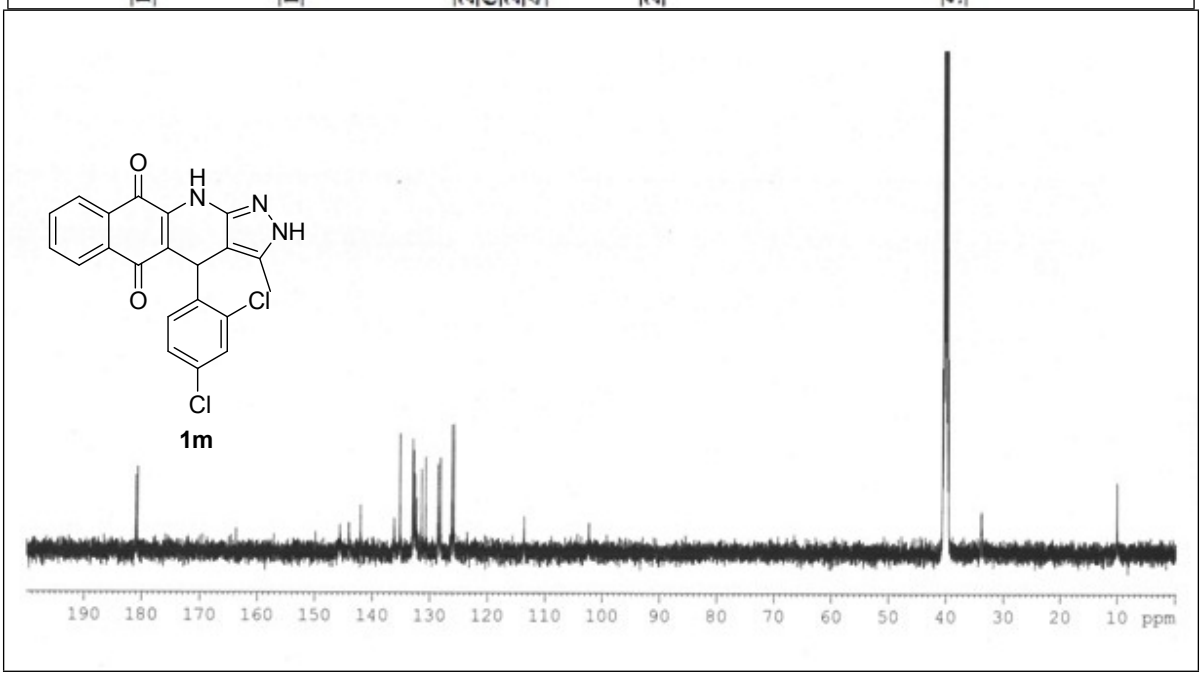
1l


O
H
N
N
NH
O
200
180
160
140
120
100
80
60
40
20
ppm

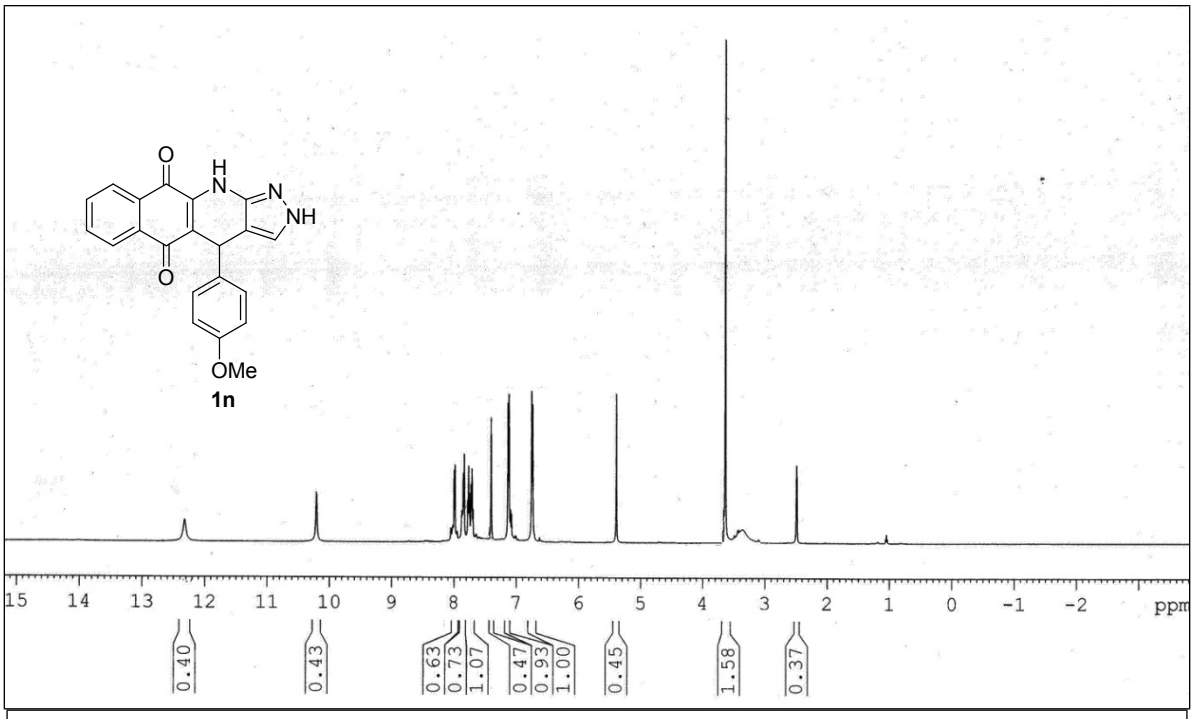
1l

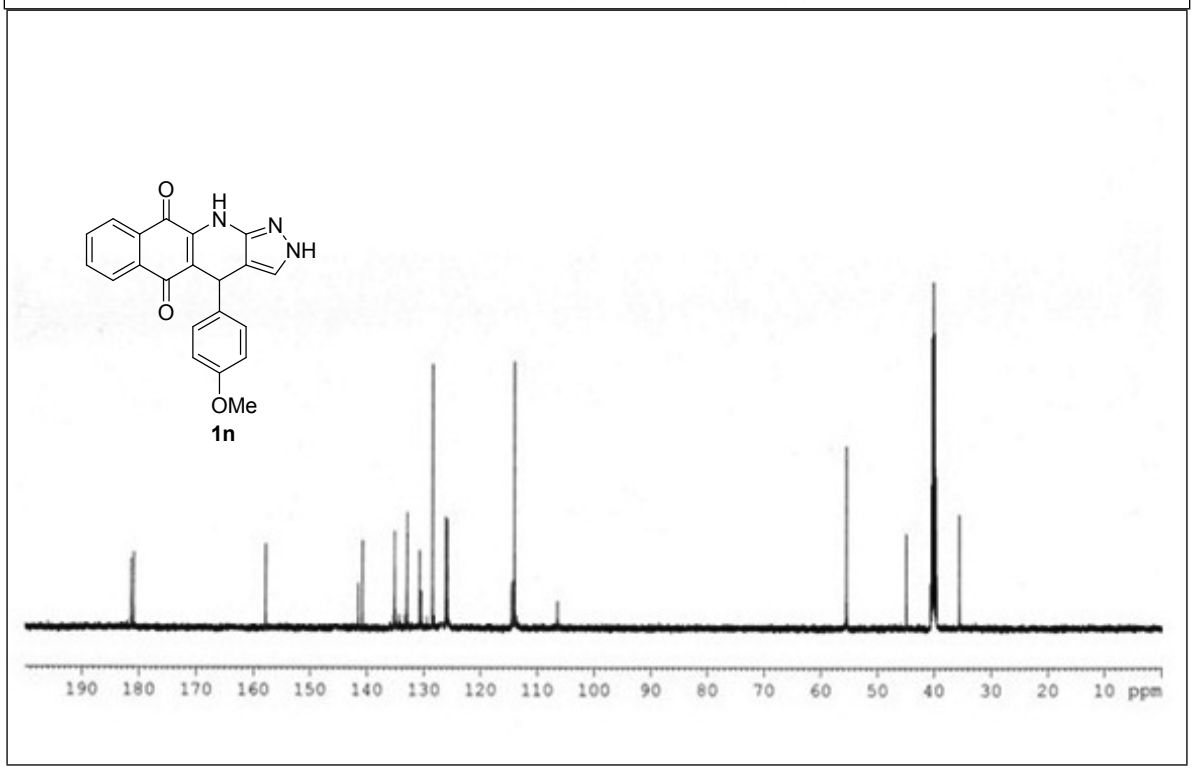

O
H
N
N
NH
O
Cl
Cl
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.90
1.92
2.11
6.33
2.00
4.10
2.00
5.99

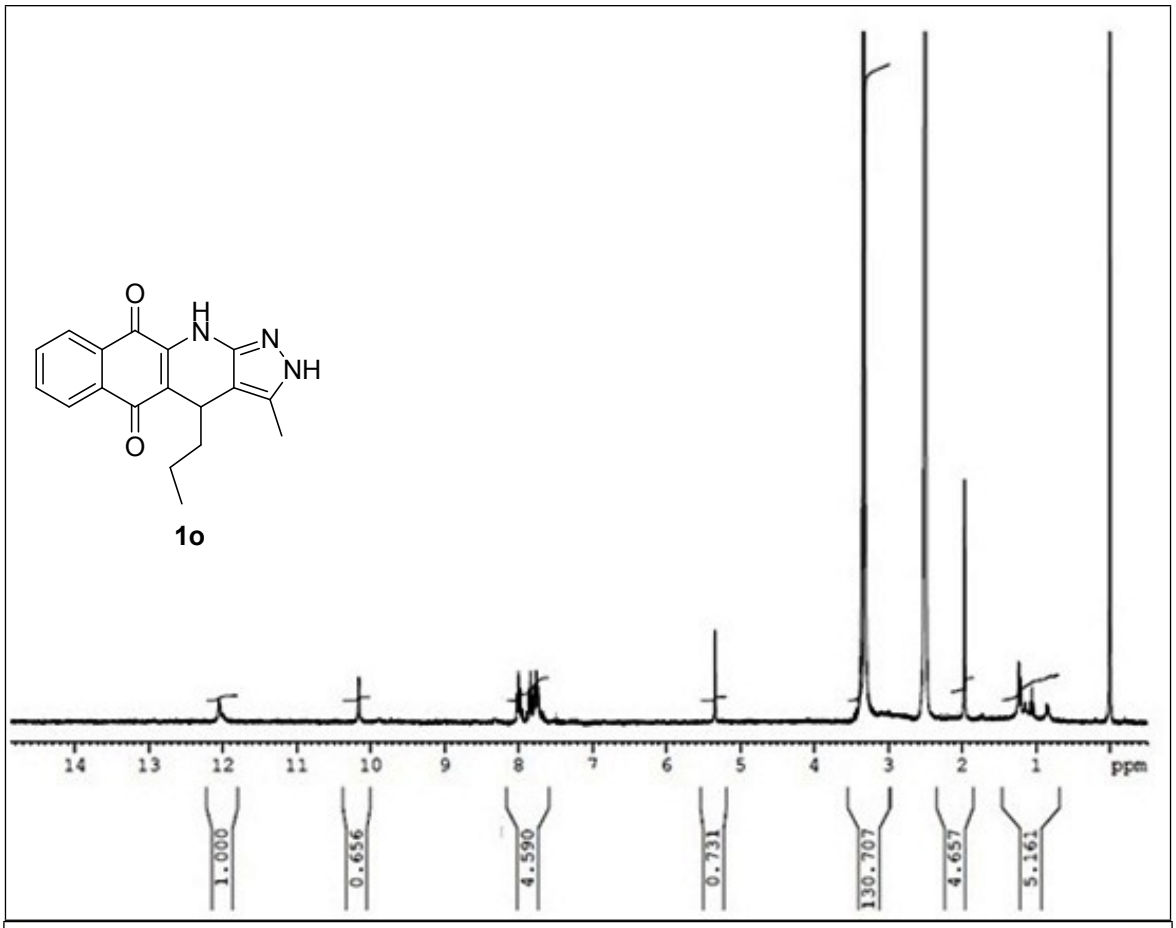
1m

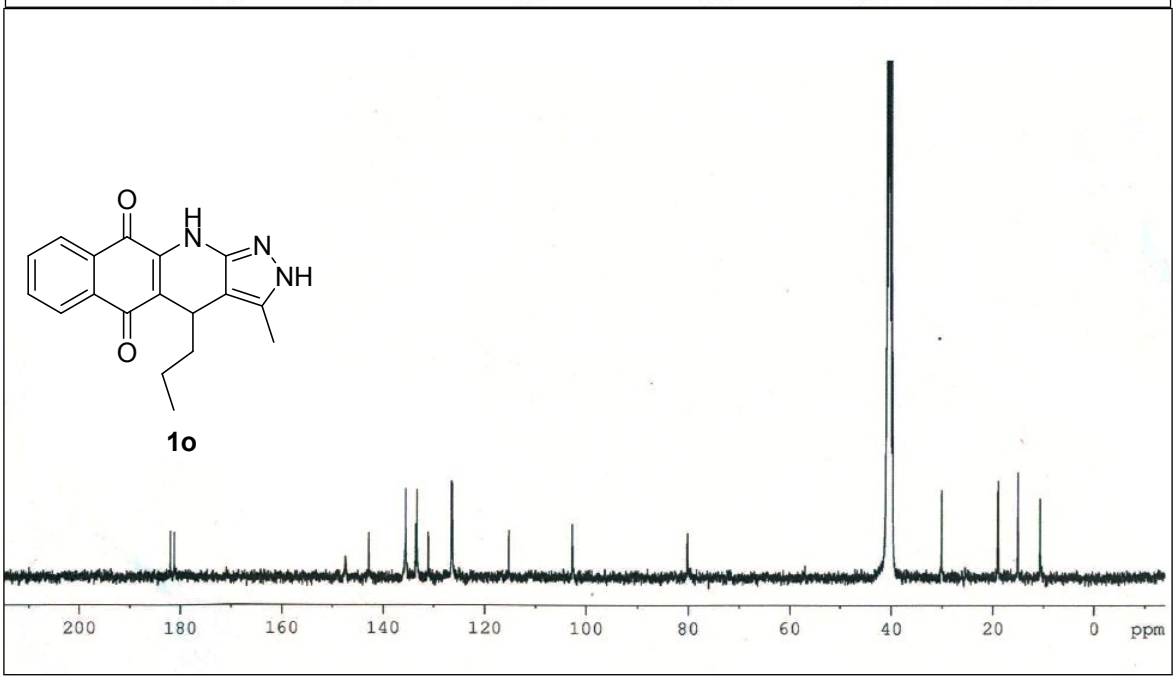

O
H
N
N
NH
O
Cl
Cl
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

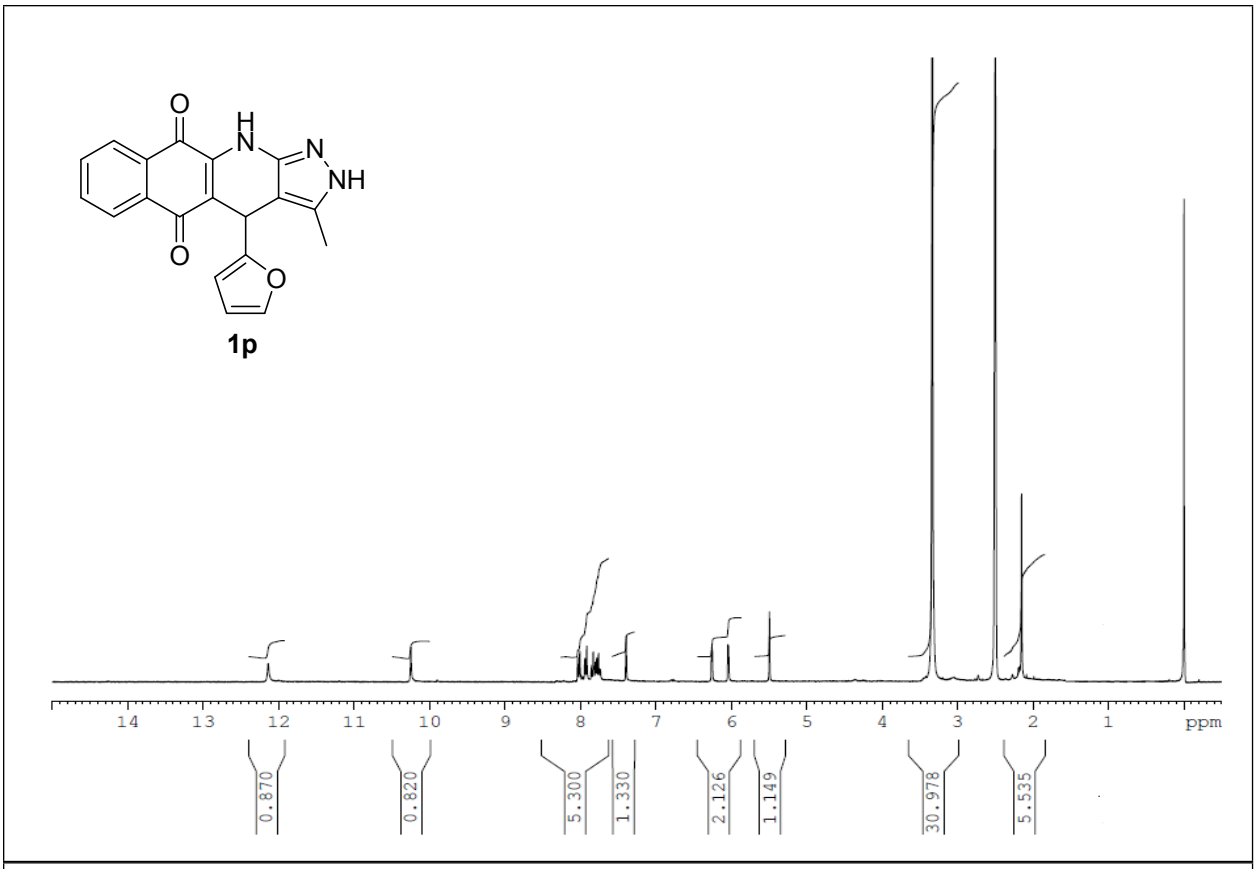
1m


O
H
N
N
NH
O
OMe
1n
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 ppm
0.40
0.43
0.63
0.73
1.07
0.47
0.93
1.00
0.45
1.58
0.37

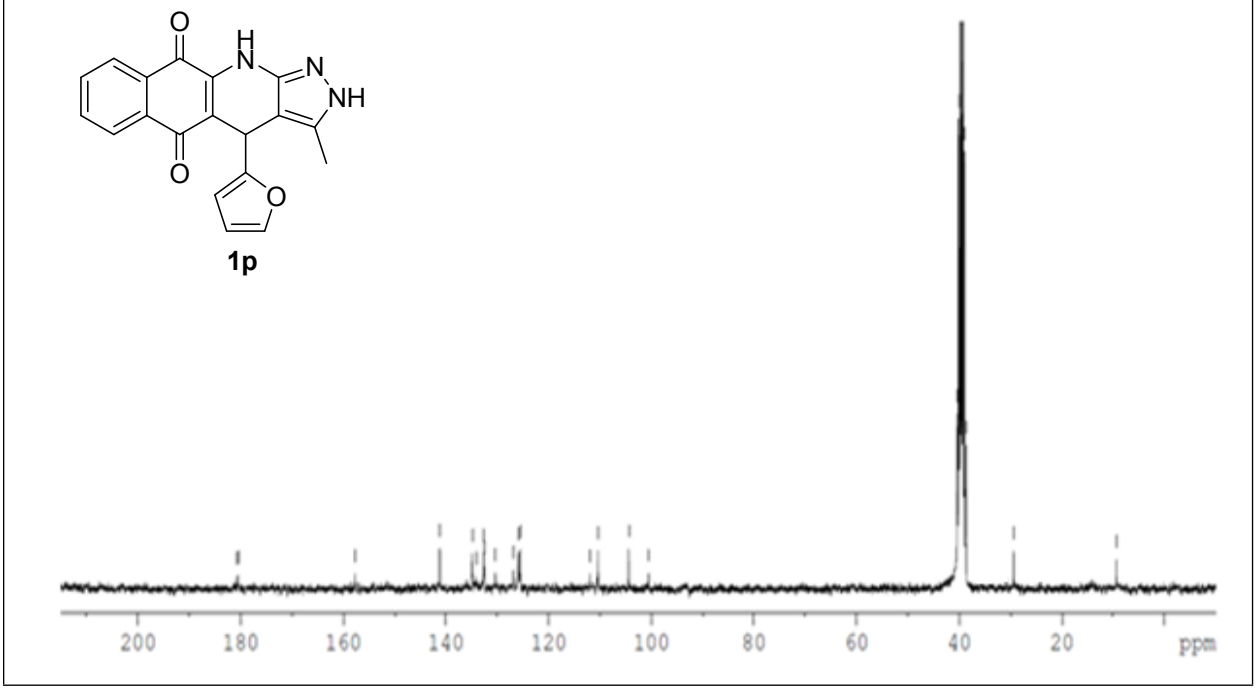

O
H
N
N
NH
O
OMe
1n
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


O
H
N
N
NH
O
1o
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.000
0.656
4.590
0.731
130.707
4.657
5.161


O
H
N
N
NH
O
1o
200
180
160
140
120
100
80
60
40
20
0
ppm


O
H
N
N
NH
O
O
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.870
0.820
5.300
1.330
2.126
1.149
30.978
5.535

1p


O
H
N
N
NH
O
O
200
180
160
140
120
100
80
60
40
20
ppm

1p