Incorporation of Keggin-Based $H_3PW_7Mo_5O_{40}$ into Bentonite: Synthesis, Characterization and Catalytic Application

Dipak S Aher, Kiran R Khillare, Prof. Sunil G Shankarwar*

Department of Chemistry, Dr. Babasaheb Ambedkar Marathwada University, Aurangabad 431004, M.S., India; E-mail: shankarwar_chem@yahoo.com

---

Supporting Information

Content

S1. EDS analysis graph:

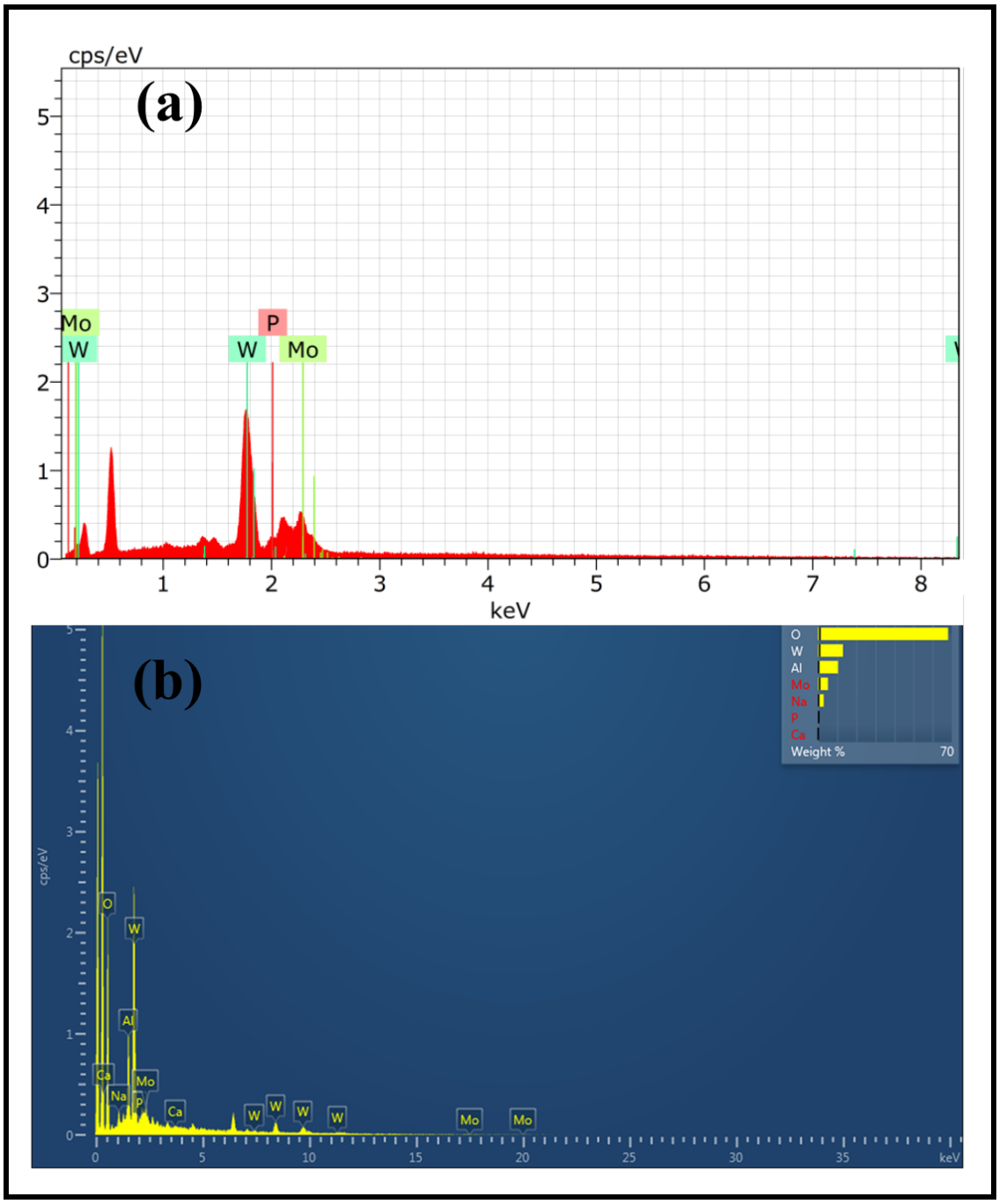


Figure 1. EDS of (a) bulk PW_7Mo_5 (b) 20% PW_7Mo_5/Bentonite catalyst.

S2. Elemental mapping images:

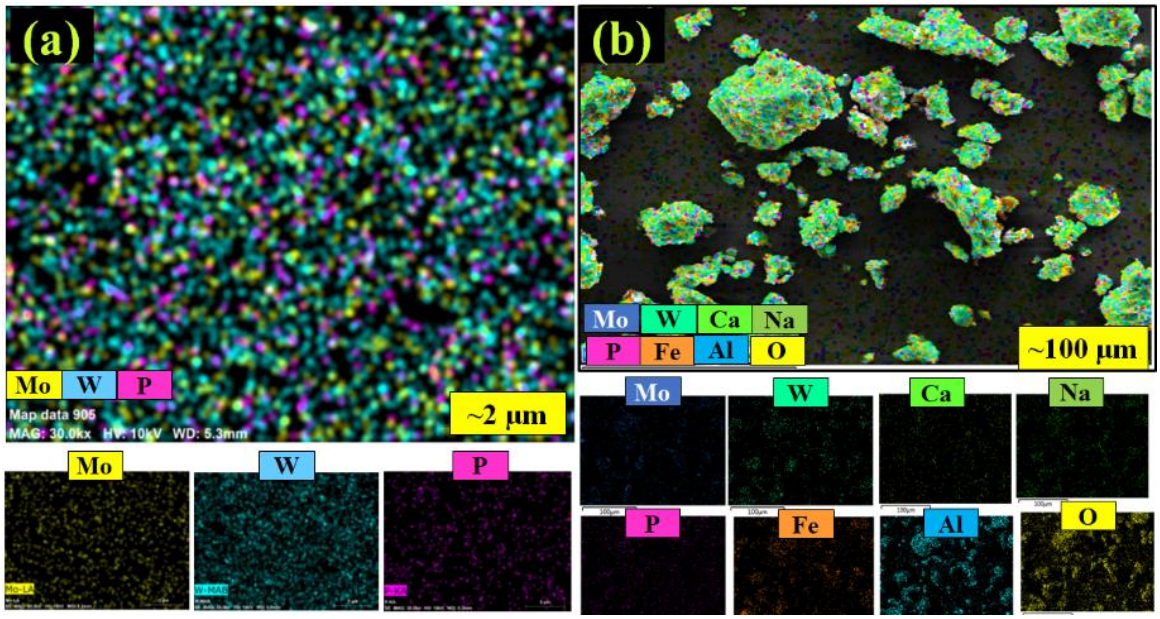


Figure 2. Elemental mapping images of (a) bulk PW_7Mo_5 (b) 20% PW_7Mo_5/Bentonite catalyst.

S3.Recovered 20% PW_7Mo_5/Bentonite catalyst.

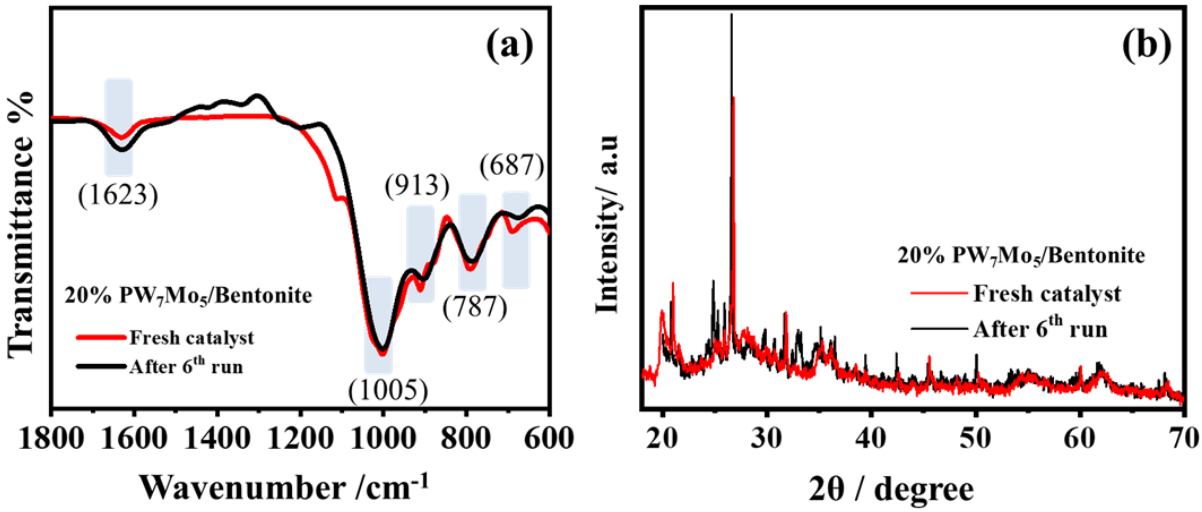


Figure 3. FT-IR (a) and XRD (b) analysis of recovered 20% PW_7Mo_5/Bentonite catalyst.

S4. Validation of Green metrics for all synthesized compounds (3a-n & 4a-n)

Calculation of green metrics:

Materials used for green metrics calculation:

Reactant 1: Benzaldehyde 0.31gm (Mol. Wt. = 106.12), 4-Chlorobenzaldehyde 0.42 gm (Mol. Wt = 140.56), 4-Nitrobenzaldehyde 0.45 gm (Mol. Wt. = 151.12), 4-Bromobenzaldehyde 0.55 gm (Mol. Wt. = 185.02), 4-Flurobenzaldehyde 0.37 gm (Mol. Wt. = 124.11), 3-Nitrobenzaldehyde 0.45 gm (Mol. Wt. = 151.12), 4-Anisaldehyde 0.40 gm (Mol. Wt. = 136.15), 4-Methylbenzaldehyde 0.36 (Mol. Wt. = 120.14), 3-Chloro-4-Flurobenzaldehyde 0.47 gm (Mol. Wt. = 158.56), 3-Bromobenzaldehyde 0.55 gm (Mol. Wt. = 185.02), 4-Hydroxybenzaldehyde 0.36 gm (Mol. Wt. = 122.12), 3-Chlorobenzaldehyde 0.42 gm (Mol. Wt. = 140.56), Salicylaldehyde 0.36 gm (Mol. Wt. = 122.12), Furfural 0.28 gm (Mol. Wt. = 96.09). 2-Chlorobenzaldehyde 0.42 gm (Mol. Wt. = 140.56). **Reactant 2:** 5, 5-dimethyl-1, 3-cyclohexanedione 0.84 gm (Mol. Wt. = 140.18). **Reactant 3:** Ammonium acetate 0.23 gm (Mol. Wt. = 77.08).

Analysis of green metrics:

The following listed formulae were used for calculating E-factor, Atom economy (AE), Reaction mass efficiency (RME), Effective mass yield (EMY), and Optimum efficiency (OE). The Calculated data for compounds 3(a-n) and 4(a-n) are presented in Table 1.

Calculation of green chemistry metrics for one representative entry, viz. 3a

E-factor = Total mass waste (g) / Mass of product

= Total input (g) – Total output (g) / Total output (g)

e.g. For the product **3a;** E-factor = (0.31 gm + 0.84 gm) – 0.959 / 0.959 = 0.19

Atom Economy (AE) (%) = (Molecular wt. of product)/ (Total molecular wt. of reactants) x 100

e.g. For the product **3a;** AE = (350.46) / (106.12 + (2 x 140.18)) x 100 = 90.67 %

##RME (%) = (Mass of isolated product)/ (Total mass of reactant) x 100

e.g. For the product **3a;** RME = (0.959) / (0.31 gm + 0.84 gm) x 100 = 83.39

##EMY (%) = (Mass of Product)/ (Mass of non-benign reagents) x 100

e.g. For the product **3a;** EMY = (0.959) / (0.31 gm + 0.84 gm) x 100 = 83.39 %

##OE (%) = RME/ AE x 100

e.g. For the product **3a;** OE = (83.39) / (90.67) x 100 = 91.97 %

Table 1. Green metrics calculation:

Entry	Product	E-factor	Atom Economy (AE)	Reaction Mass Efficiency (RME) %	Effective Mass Yield (EMY) %	Optimum Efficiency (OE) %
1.	3a	0.19	90.67	83.39	83.39	91.97
2.	3b	0.19	91.26	83.88	83.88	91.91
3.	3c	0.21	91.65	82.48	82.48	89.96
4.	3d	0.20	92.25	83.02	83.02	89.99
5.	3e	0.29	91.02	77.43	77.43	85.06
6.	3f	0.22	91.58	81.47	81.47	88.96
7.	3g	0.21	91.28	82.09	82.09	89.93
8.	3h	0.29	90.93	77.25	77.25	84.95
9.	3i	0.36	91.79	73.35	73.35	79.91
10.	3j	0.19	92.25	83.88	83.88	90.92
11.	3k	0.29	91.05	77.33	77.33	84.93
12.	3l	0.21	91.44	82.22	82.22	89.91
13.	3m	0.23	91.05	80.91	80.91	88.86
14.	3n	0.29	90.43	77.41	77.41	86.60
15.	4a	0.44	75.38	69.34	69.34	91.98
16.	4b	0.41	77.09	67.49	67.49	87.54
17.	4c	0.51	77.56	65.92	65.92	84.99
18.	4d	0.39	78.96	71.85	71.85	90.99
19.	4e	0.45	77.56	69.03	69.03	89.20
20.	4f	0.44	76.88	69.19	69.19	89.99
21.	4g	0.52	77.09	65.52	65.52	84.99
22.	4h	0.58	78.96	63.17	63.17	80.00
23.	4i	0.64	76.20	60.95	60.95	79.98
24.	4j	0.47	76.30	67.90	67.90	89.10
25.	4k	0.50	76.11	66.50	66.50	87.37
26.	4l	0.56	77.88	63.86	63.86	81.99
27.	4m	0.66	76.20	60.20	60.20	79.00
28.	4n	0.64	77.09	60.89	60.89	78.98

Note: Since the catalyst was recovered during the reaction, therefore it's used amount is not included in the calculations.

S5. Spectral data of the compounds:

3,3,6,6-tetramethyl-9-phenyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3a).

IR (ATR, υ cm^{-1}): 685, 835, 1035, 1242, 1364, 1581, 1664, 2958. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H (ppm) = 1.09 (s, 6H 2x CH_3), 1.23 (s, 6H 2x CH_3), 2.37 (m, 8H 4x CH_2), 5.54 (s, 1H CH), 7.09 (d, J = 8.2 Hz, 2H Ar-H), 7.22 (t, 1H Ar-H), 7.26 (t, J = 8.4 Hz, 2H Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$) δ_C (ppm) = 27.5, 29.8, 31.5, 32.8, 46.5, 47.1, 115.7, 125.9, 126.8, 128.3, 138.1, 189.6, 190.6.

9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3b).

IR (ATR, υ cm^{-1}): 649, 863, 1011, 1091, 1155, 1365, 1576, 1663, 2955. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H (ppm) = 1.15 (s, 6H 2x CH_3), 1.27 (s, 6H 2x CH_3), 2.57 - 2.27 (m, 8H 4x CH_2), 5.53 (s, 1H CH), 7.07 (d, J= 8.5 Hz, Ar-H), 7.25 (d, J= 8.3 Hz, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$) δ_C (ppm) = 27.4, 29.6, 31.4, 32.4, 46.4, 47.1, 76.7, 77.1, 77.4, 115.3, 128.2, 128.4, 131.6, 136.7, 189.5, 190.6.

3,3,6,6-tetramethyl-9-(-4-nitrophenyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3c).

IR (ATR, υ cm^{-1}): 677, 843, 1114, 1238, 1342, 1506, 1577, 3200. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H (ppm) = 1.12 (s, 6H 2x CH_3), 1.24 (s, 6H 2x CH_3), 2.55 - 2.30 (m, 8H 4x CH_2), 5.57 (s, 1H CH), 7.40 (d, J= 8.4 Hz, 2H Ar-H), 8.10 (d, J= 8.7 Hz, 2H Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$) δ_C (ppm) =27.5, 29.5, 31.5, 33.3, 46.4, 47.0, 76.7, 77.0, 77.3, 114.9, 123.5, 127.6, 146.2, 189.6, 190.9.

3,3,6,6-tetramethyl-9-(-3-nitrophenyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3f).

IR (ATR, υ cm^{-1}): 652, 801, 1042, 1148, 1351, 1580, 1661, 2954. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H (ppm) = 1.12 (s, 6 H 2x CH_3), 1.27 (s, 6H 2x CH_3), 2.48 - 2.36 (m, 8H 4x CH_2), 5.54 (s, 1 H CH), 7.49 -7.35 (m, 2H Ar-H), 8.00 (dt, 1H Ar-H), δ 8.07-8.02 (s, 1H Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$) δ_C (ppm) =27.4, 28.8, 31.5, 32.9, 46.1, 47.1, 114.9, 121.1, 122.3, 129.2, 133.0, 140.7, 148.5, 189.7, 191.2.

9-(3-bromophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3j).

IR (ATR, υ cm^{-1}): 664, 785, 1033, 1244, 1366, 1581, 2952. ^{1}H NMR (400 MHz , $CDCl_3$) δ_H (ppm) = 1.05 (s, 6H 2x CH_3), 1.18 (s, 6H 2x CH_3), 2.53 - 2.25 (m, 8H 4x CH_2), 5.49 (s, 1H CH), 7.07 (t, *J*= 8.0 1H Ar-H), 7.14 (t, *J* = 8.0 Hz, 1H Ar-H), 7.23 (d, *J* = 7.6 Hz, 1H Ar-H), 7.33 (s, 1H Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$) δ_C (ppm) = 27.4, 29.7, 31.5, 32.7, 47.1, 115.1, 122.6, 125.5, 129.0, 130.2, 140.8, 189.5, 190.7, 207.1.

9-(3-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3l).

IR (ATR, υ cm^{-1}): 666, 787, 869, 1034, 1244, 1367, 1581, 1666, 2953. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H (ppm) = 1.10 (s, 6 H, 2x CH_3), 1.23 (s, 6H, 2x CH_3), 2.45-2.34 (m, 8H 4x CH_2) 5.49 (s, 1H CH), 6.97 (d, J=7.6 Hz, 1H Ar-H), 7.07 (t, 8Hz 1H Ar-H), 7.23 - 7.12 (m, 2H Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$) δ_C (ppm) = 27.4, 29.6, 31.5, 32.7, 46.4, 47.0, 115.1, 125.0, 126.1, 127.2, 129.4, 134.2, 140.5, 189.5, 190.7.

3,3,6,6-tetramethyl-9-phenyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4a).

IR (ATR, υ cm^{-1}): 661, 833, 1035, 1154, 1237, 1363, 1582, 1630, 2956, 3286. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H (ppm) = 1.09 (s, 6H, 2x CH_3); 1.24 (s, 6H, 2x CH_3), 2.51 - 2.33 (m, 8H 4x CH_2), 5.55 (s, 1H CH), 7.11 (d, J = 8.2 Hz, 2H Ar-H), 7.19 (d, J = 6.9 Hz, 2H Ar-H), 7.26 (s, 1H Ar-H), 7.27 (s, 1H -NH). ^{13}C NMR (100 MHz, $CDCl_3$) δ_C (ppm) = 27.5, 29.8, 31.5, 32.8, 47.1, 115.7, 125.9, 126.8, 128.1, 138.1, 189.5, 190.6.

9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4b).

IR (ATR, υ cm^{-1}): 766, 836, 1141, 1215, 1487, 1603, 1645, 2955, 3047, 3351. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H (ppm) = 0.93 (s, 6H, 2x CH_3), 1.05 (s, 6H, 2x CH_3), 2.34 - 2.13 (m, 8H 4x CH_2), 5.02 (s, 1H CH), 7.14 (d, J = 8.2 Hz, 2 H), 7.28 - 7.24 (m, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ_C (ppm) = 27.1, 29.6, 31.5, 32.7, 33.4, 40.8, 50.7, 128.3, 128.4, 129.5, 131.7, 196.2.

3,3,6,6-tetramethyl-9-(4-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4c).

IR (ATR, υ cm^{-1}): 693, 826, 1134, 1251, 1336, 1443, 1515, 1639, 2914, 3376. ^{1}H NMR (400 MHz, DMSO-d_6) δ_H (ppm) = 0.81 (s, 6H 2x CH_3), 0.97 (s, 6H 2x CH_3), 1.94 (d, *J* = 6.5 Hz, 2H CH_2), 2.15 (d, *J* = 6.5 Hz, 2H CH_2), 2.31 (m, 4H 2x CH_2), 4.87 (s, 1H CH), 7.49 - 7.34 (m, 2H Ar-H), 8.13 - 7.92 (m, 2 H Ar-H), 9.44 (s, 1H -NH). ^{13}C NMR (100 MHz, DMSO-d_6) δ_C (ppm) =27.0, 29.5, 32.7, 34.4, 50.6, 110.9, 123.5, 129.4, 146.0, 150.5, 155.1, 194.9.

3,3,6,6-tetramethyl-9-(3-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4e).

IR (ATR, υ cm^{-1}): 685, 893, 1135, 1215, 1350, 1478, 1642, 2950, 3178. ^{1}H NMR (400 MHz, DMSO-d_6) δ_H (ppm) = 0.86 (s, 6H 2x CH_3), 1.02 (s, 6H 2x CH_3), 2.02 (br. s., 2H CH_2), 2.19 (br. s., 2H CH_2), 2.39 (br. s., 4H 2x CH_2), 4.93 (br. s., 1H CH), 7.50 (br. s., 1H Ar-H), 7.63 (br. s., 1H Ar-H), 7.97 (br. s., 2H Ar-H), 9.48 (br. s., 1H -NH). ^{13}C NMR (100 MHz, DMSO-d_6) δ_C (ppm) = 26.8, 29.5, 32.6, 34.0, 50.5, 111.0, 121.2, 122.5, 129.7, 134.9, 147.8, 149.7, 150.5, 194.9.

9-(3-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4g).

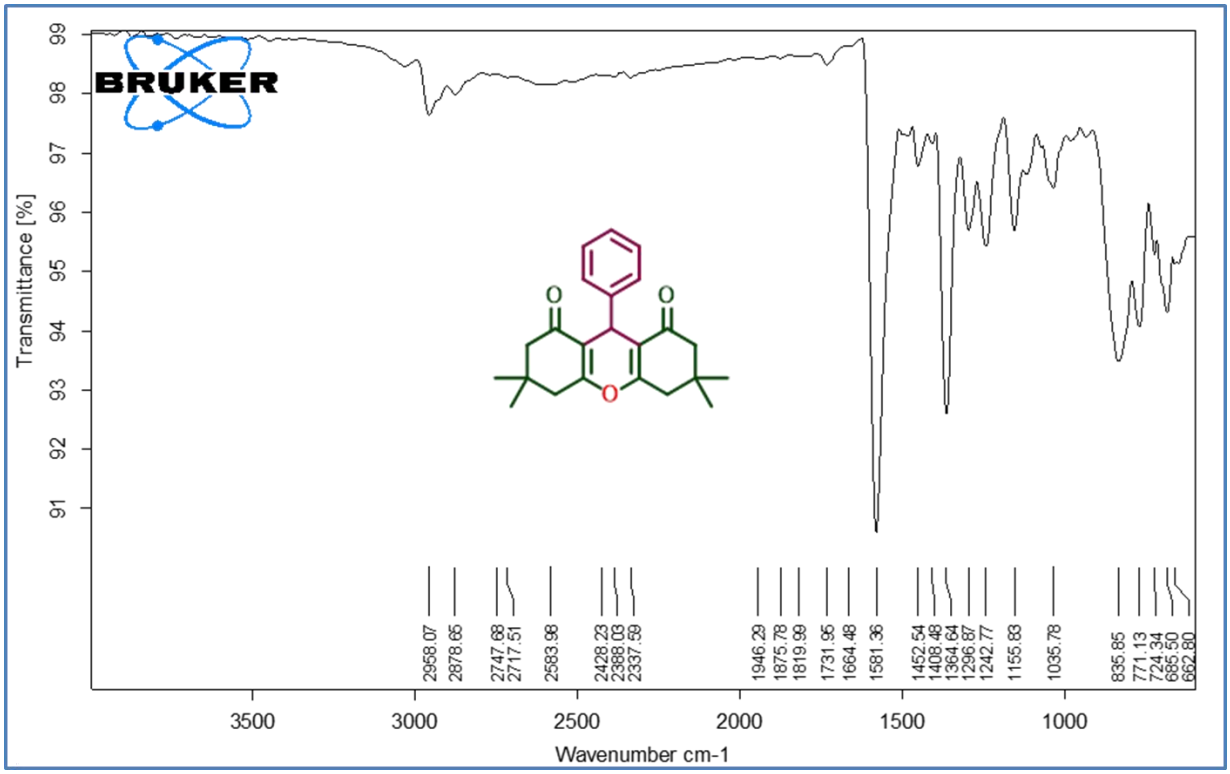
IR (ATR, υ cm^{-1}): 686, 797, 1136, 1213, 1354, 1592, 1636, 2947, 3058, 3171. ^{1}H NMR (400 MHz, DMSO-d_6) δ_H (ppm) = 0.87 (s, 6H 2x CH_3), 1.01 (s, 6H 2x CH_3), 2.01 (d, *J* = 15.3 Hz, 2H CH_2), 2.18 (d, *J* = 15.4 Hz, 2H CH_2), 2.44 - 2.27 (m, 4H 2x CH_2), 4.80 (br. s., 1H CH), 7.29 -6.99 (m, 4H Ar-H), 9.37 (s, 1H -NH). ^{13}C NMR (100 MHz, DMSO-d_6) δ_C (ppm) = 26.9, 29.5, 32.6, 33.5, 50.6, 111.3, 126.0, 126.7, 128.1, 130.0, 132.7, 149.9, 150.2, 194.9.

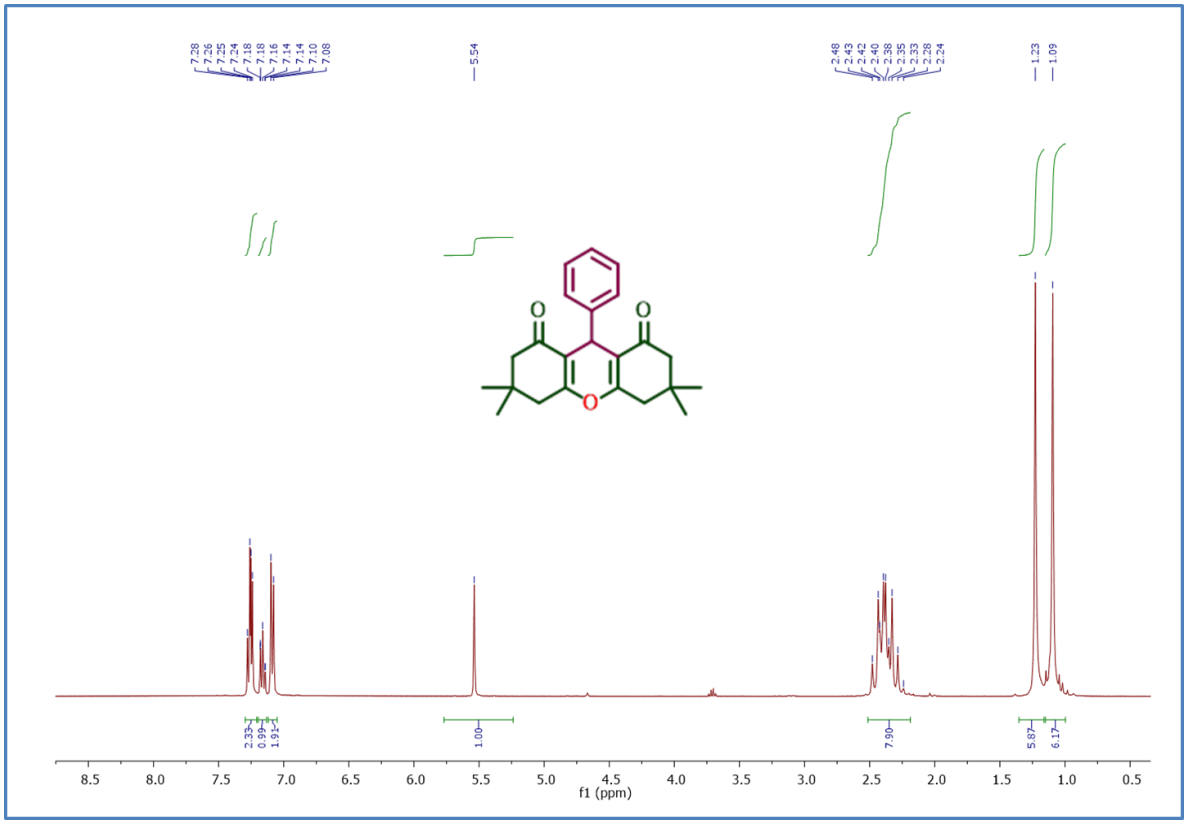
9-(3-bromophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4h).

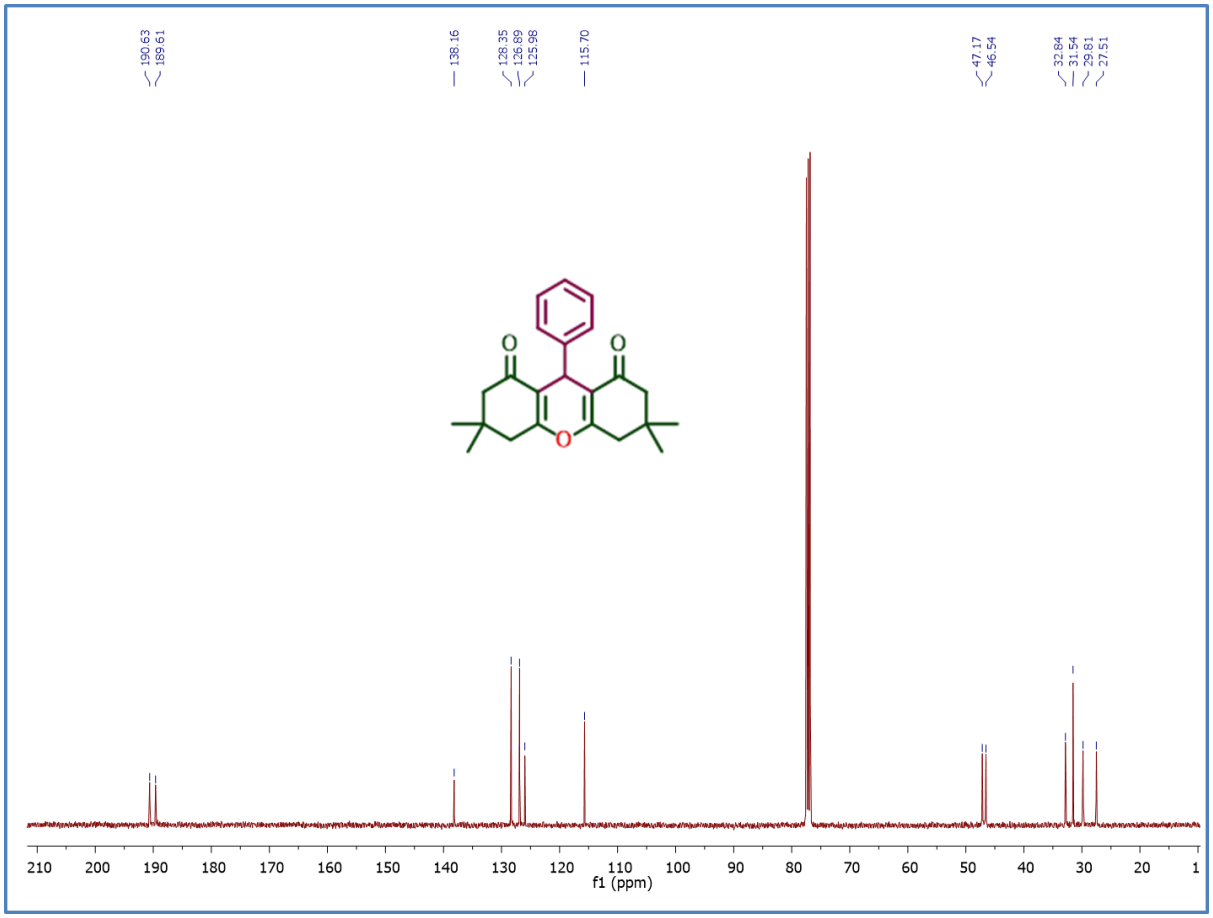
IR (ATR, υ cm^{-1}): 684, 765, 882, 1009, 1136, 1480, 1602, 1636, 2951, 3054, 3263. ^{1}H NMR (400 MHz, DMSO-d_6) δ_H (ppm) = 0.82 (s, 6H 2x CH_3), 0.96 (s, 6H 2x CH_3), 1.97 (br. s., 2H CH_2), 2.12 (br. s., 2H CH_2), 2.40-2.31 (m 4H 2x CH_2), 4.73 (br. s., 1H CH), 7.25-7.09 (m, 4H Ar-H), 9.34 (s, 1H -NH). ^{13}C NMR (100 MHz, DMSO-d_6) δ_C (ppm) =26.9, 29.6, 32.7, 33.6, 50.6, 111.4, 121.5, 127.1, 128.9, 130.5, 150.2, 194.9.

S6. FT-IR, ^{1}H & ^{13}C NMR spectra of the compounds:

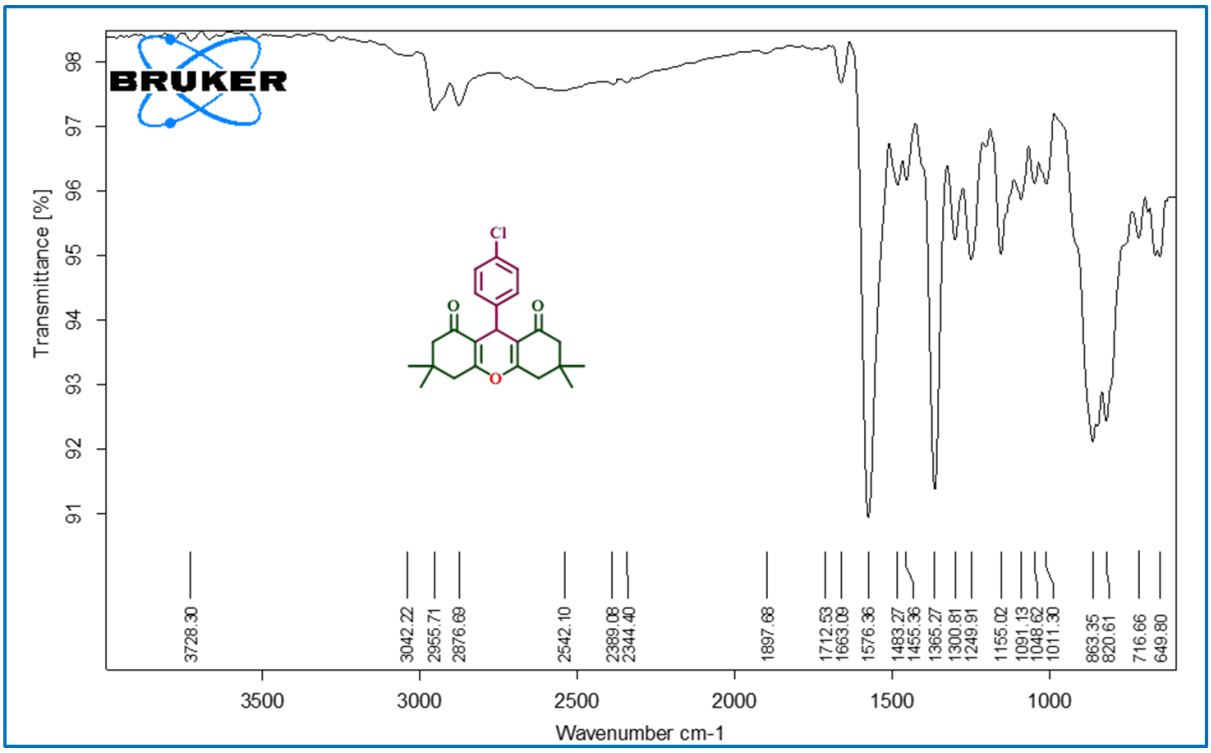
3,3,6,6-tetramethyl-9-phenyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3a).

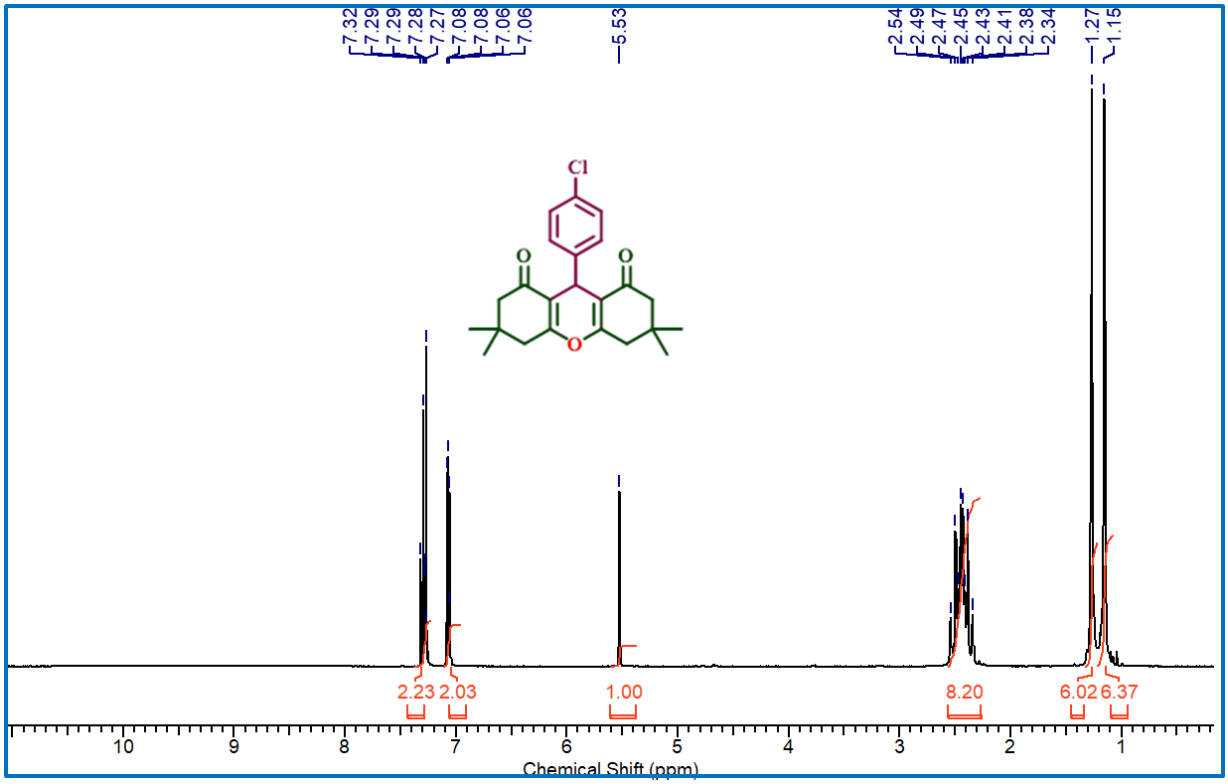


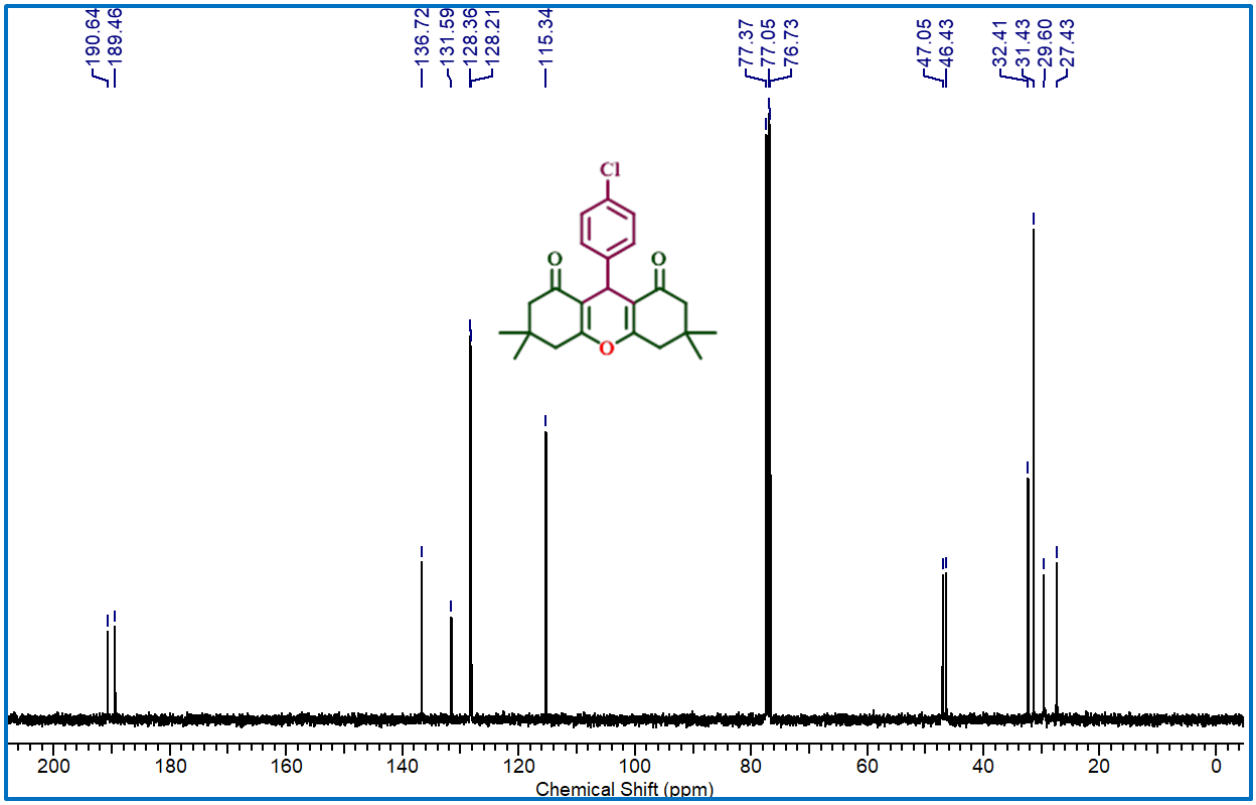




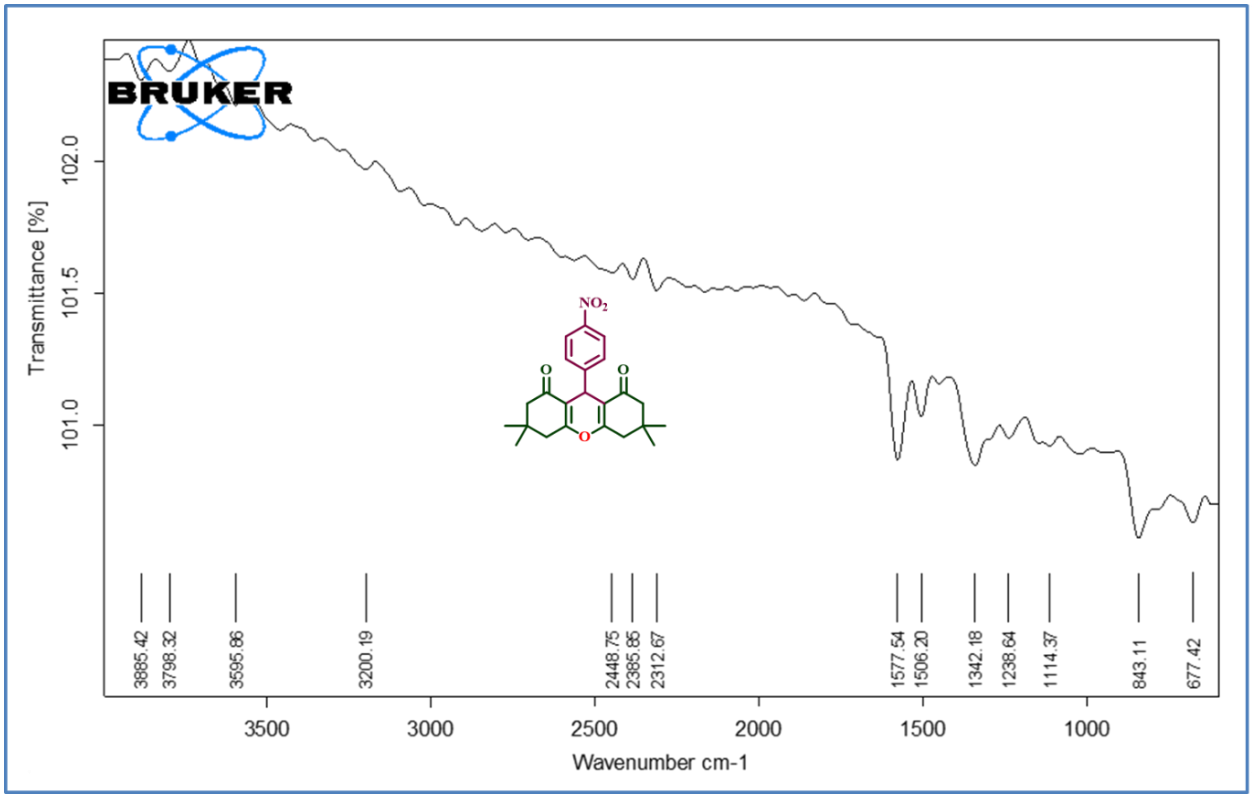
9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3b).

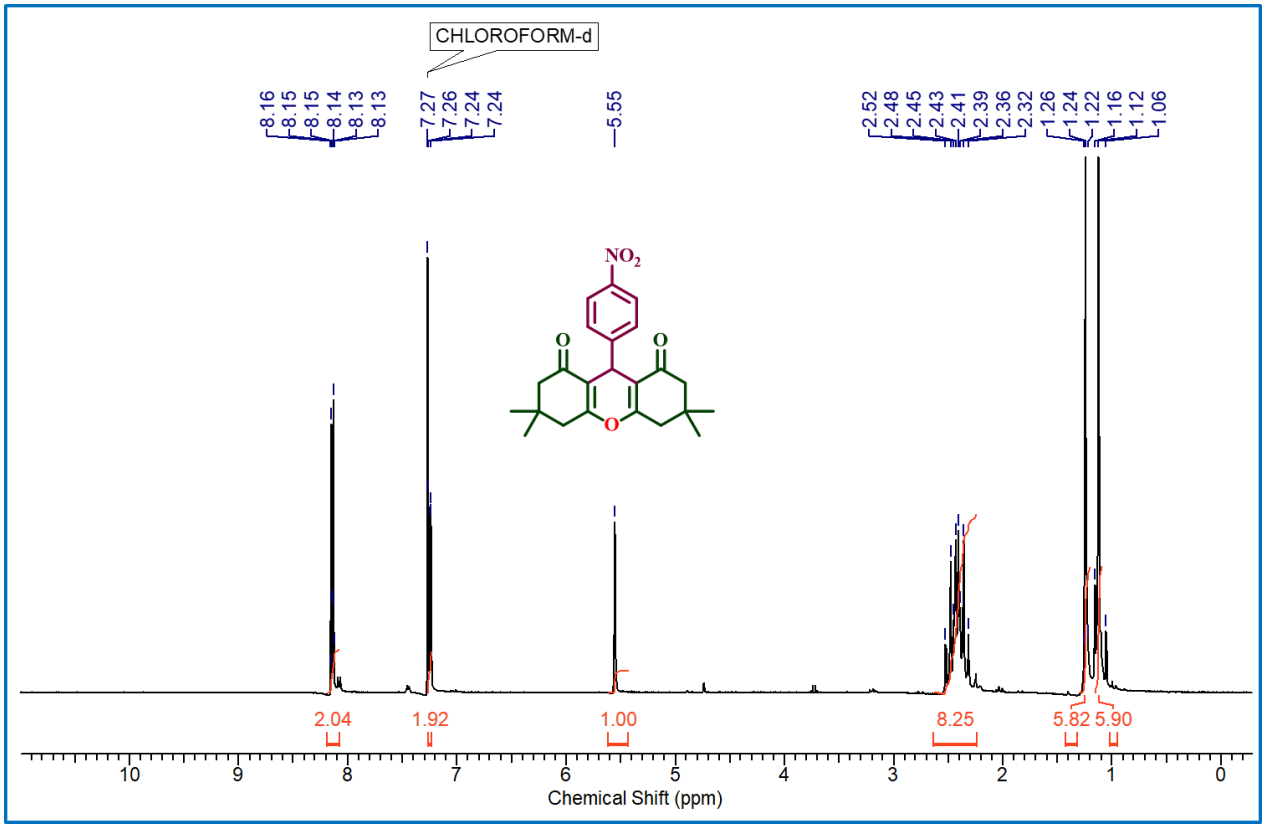


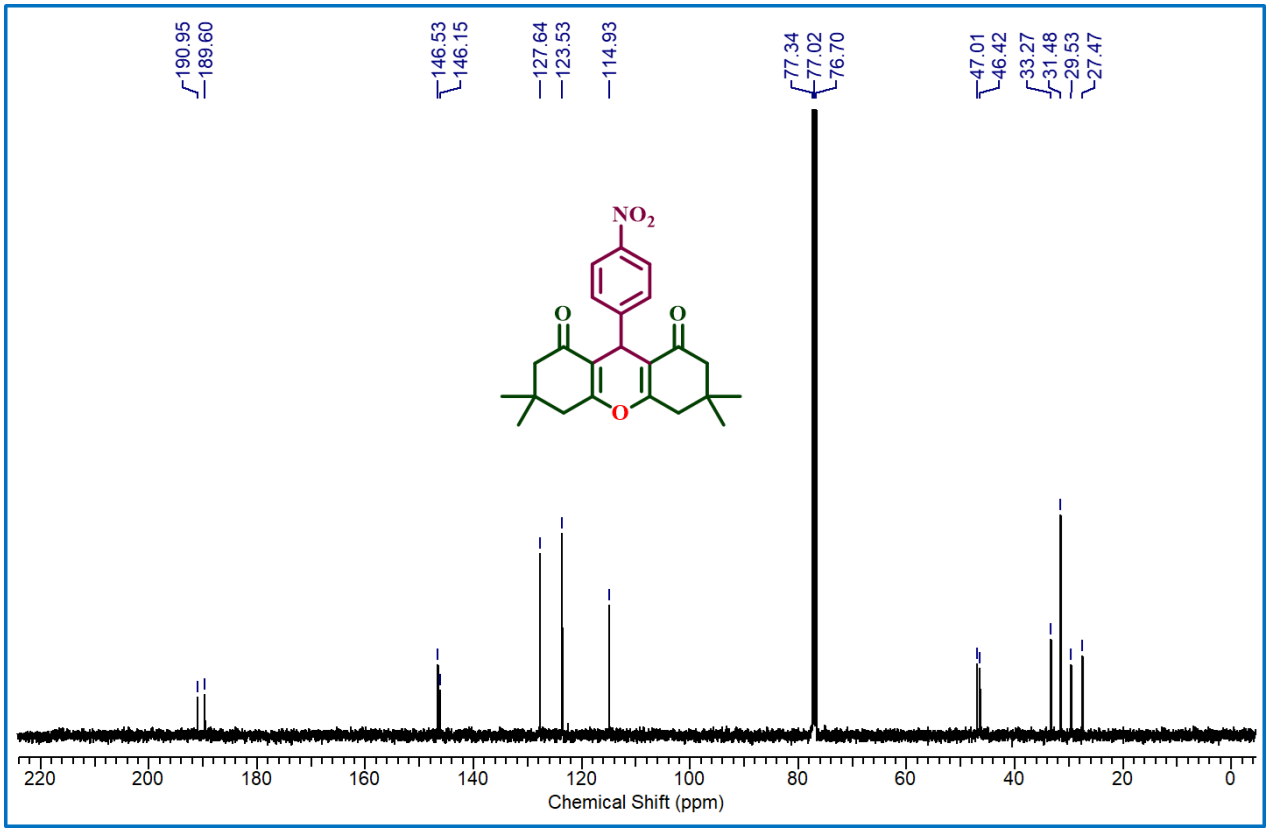

7.32
7.29
7.29
7.28
7.27
7.08
7.08
7.06
7.06
5.53
2.54
2.49
2.47
2.45
2.43
2.41
2.38
2.34
1.27
1.15
Cl
O
O
O
2.23
2.03
1.00
8.20
6.02
6.37
10
9
8
7
6
5
4
3
2
1
Chemical Shift (ppm)


190.64
189.46
136.72
131.59
128.36
128.21
115.34
77.37
77.05
76.73
47.05
46.43
32.41
31.43
29.60
27.43
Cl
O
O
O
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

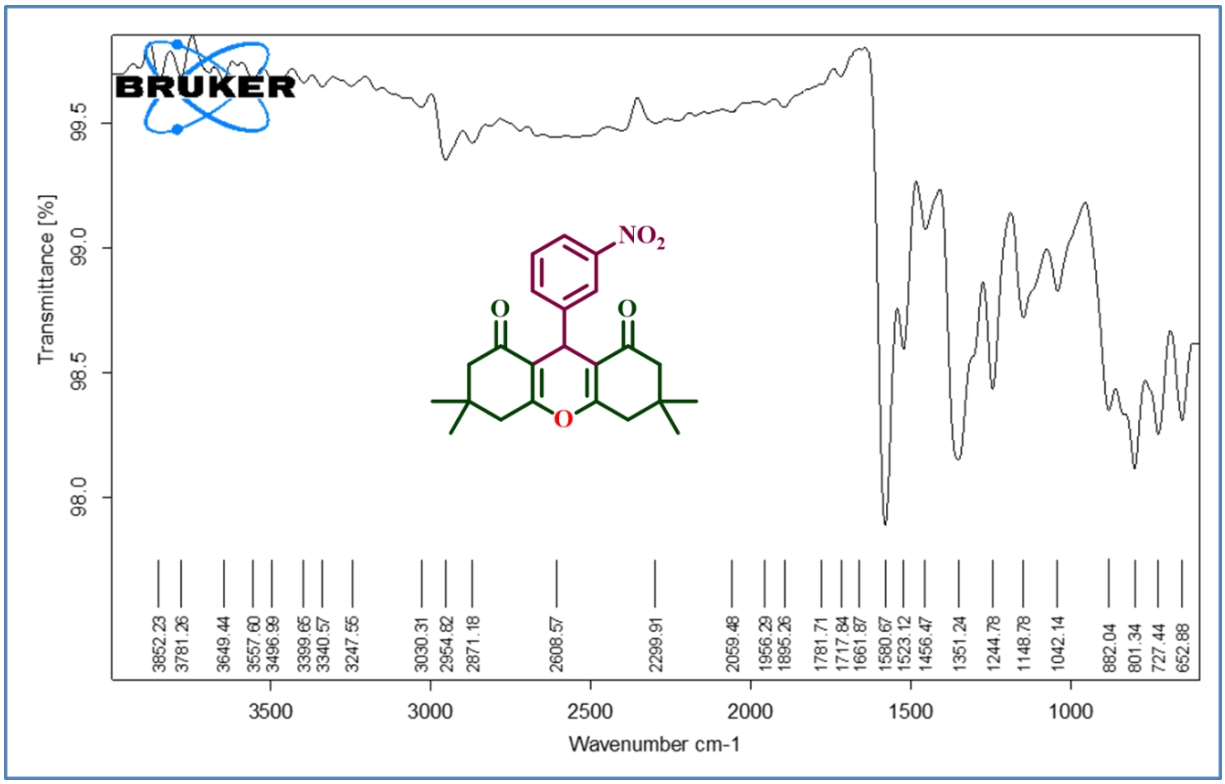
3,3,6,6-tetramethyl-9-(-4-nitrophenyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3c)

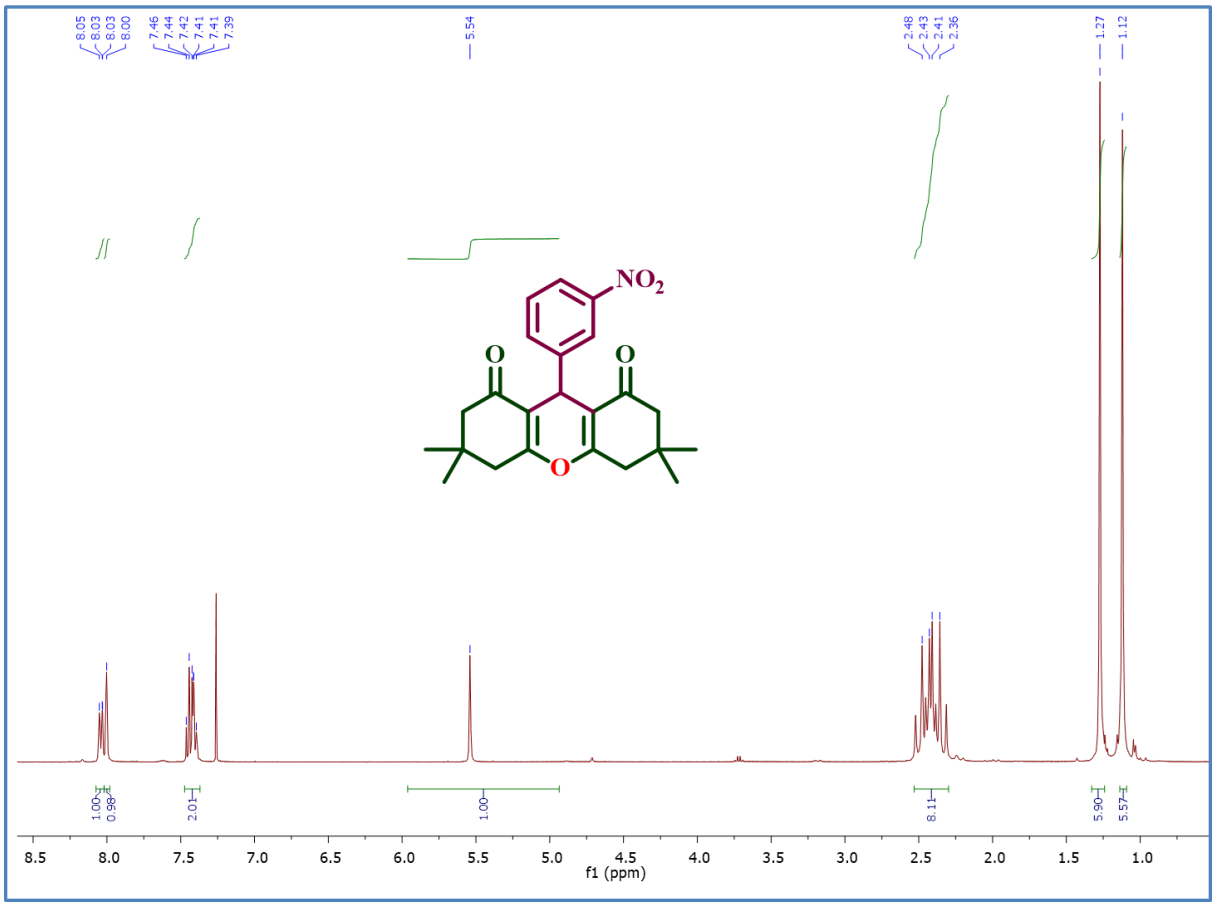


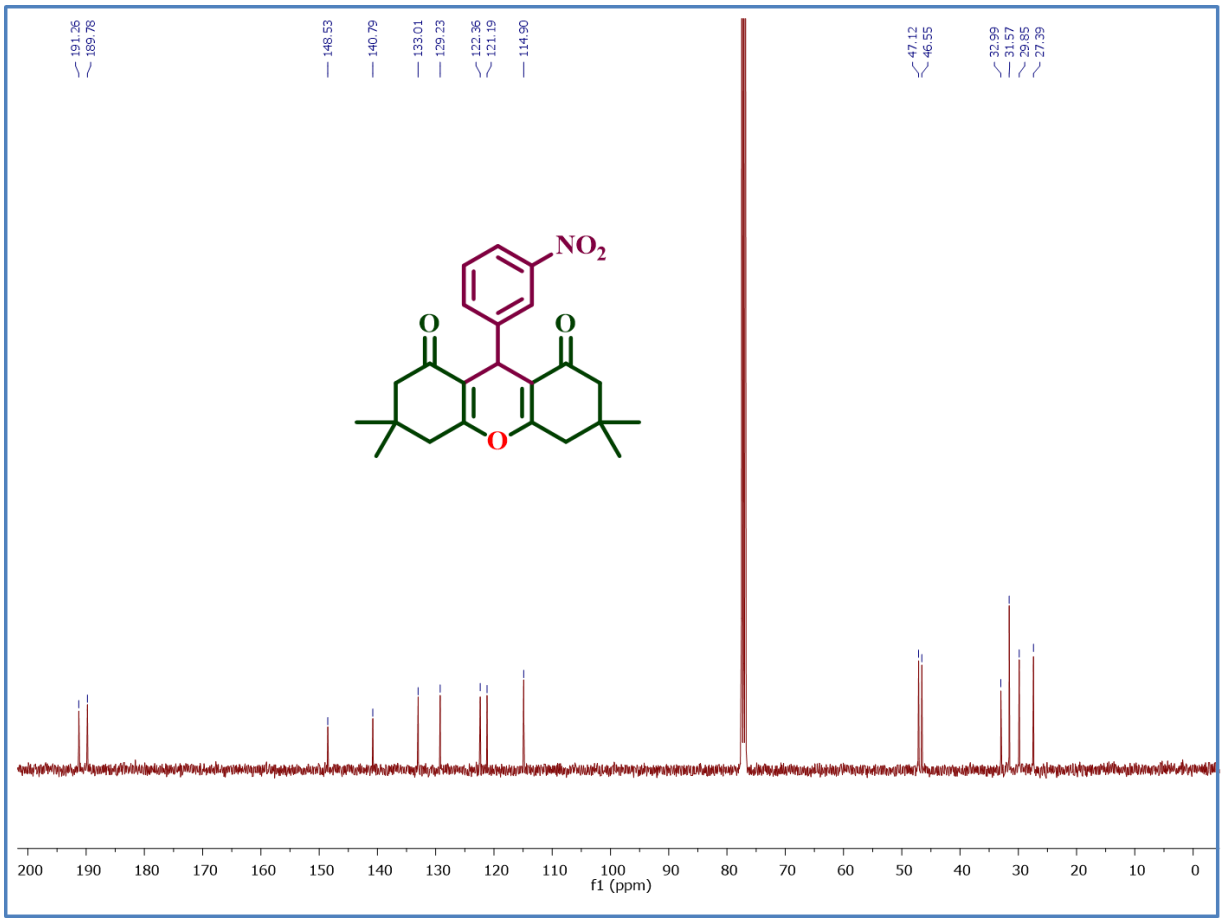




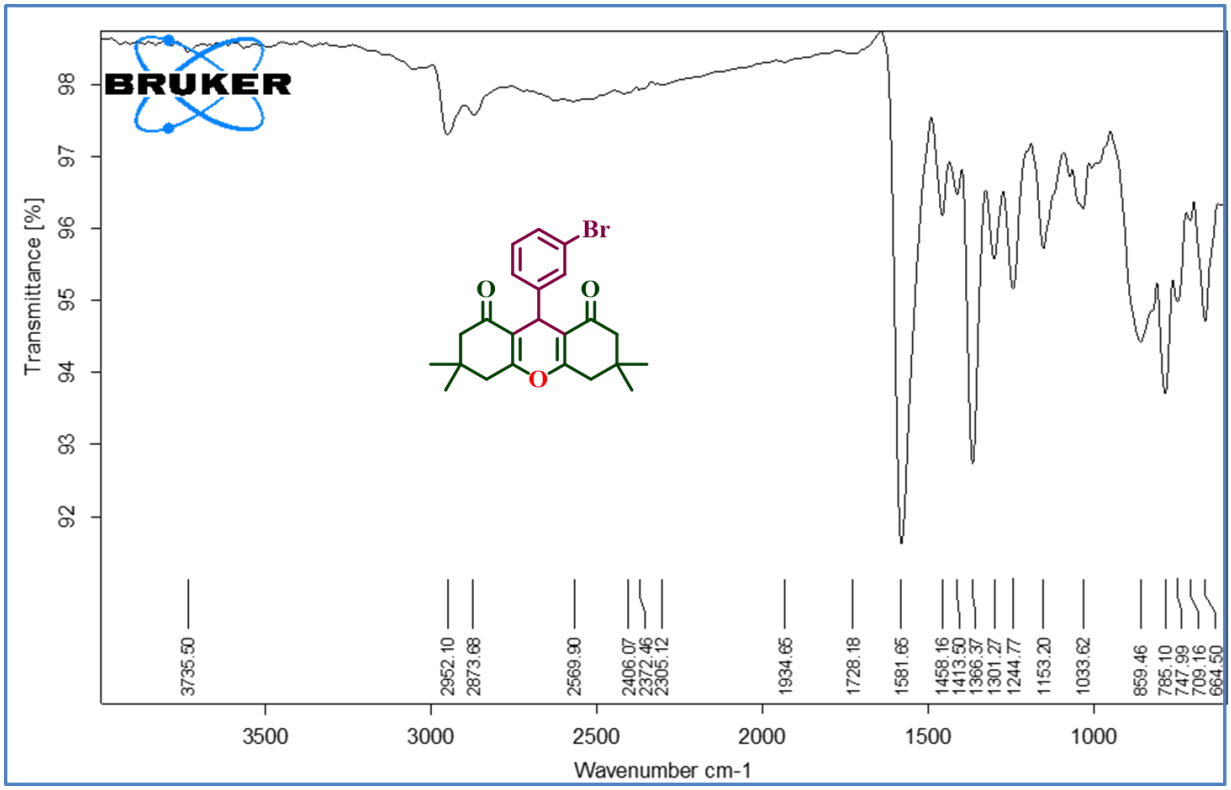
3,3,6,6-tetramethyl-9-(-3-nitrophenyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3f)

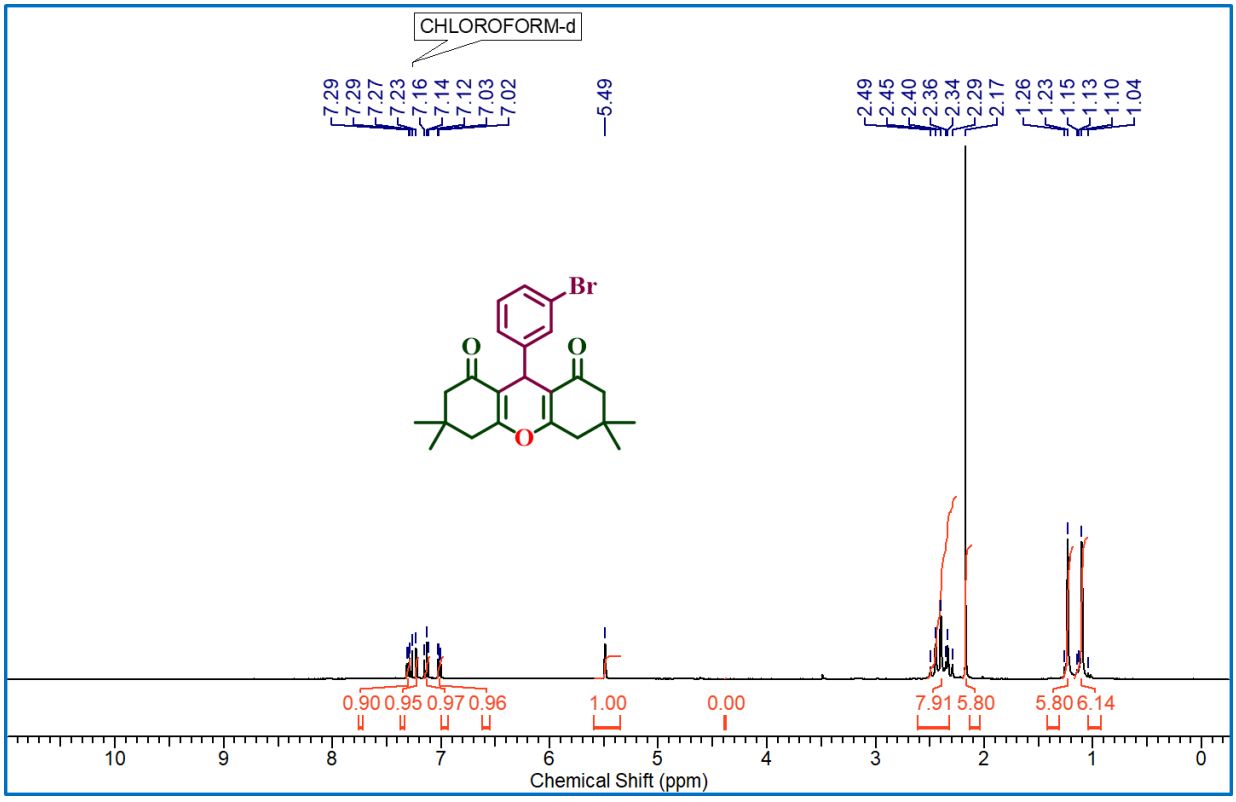


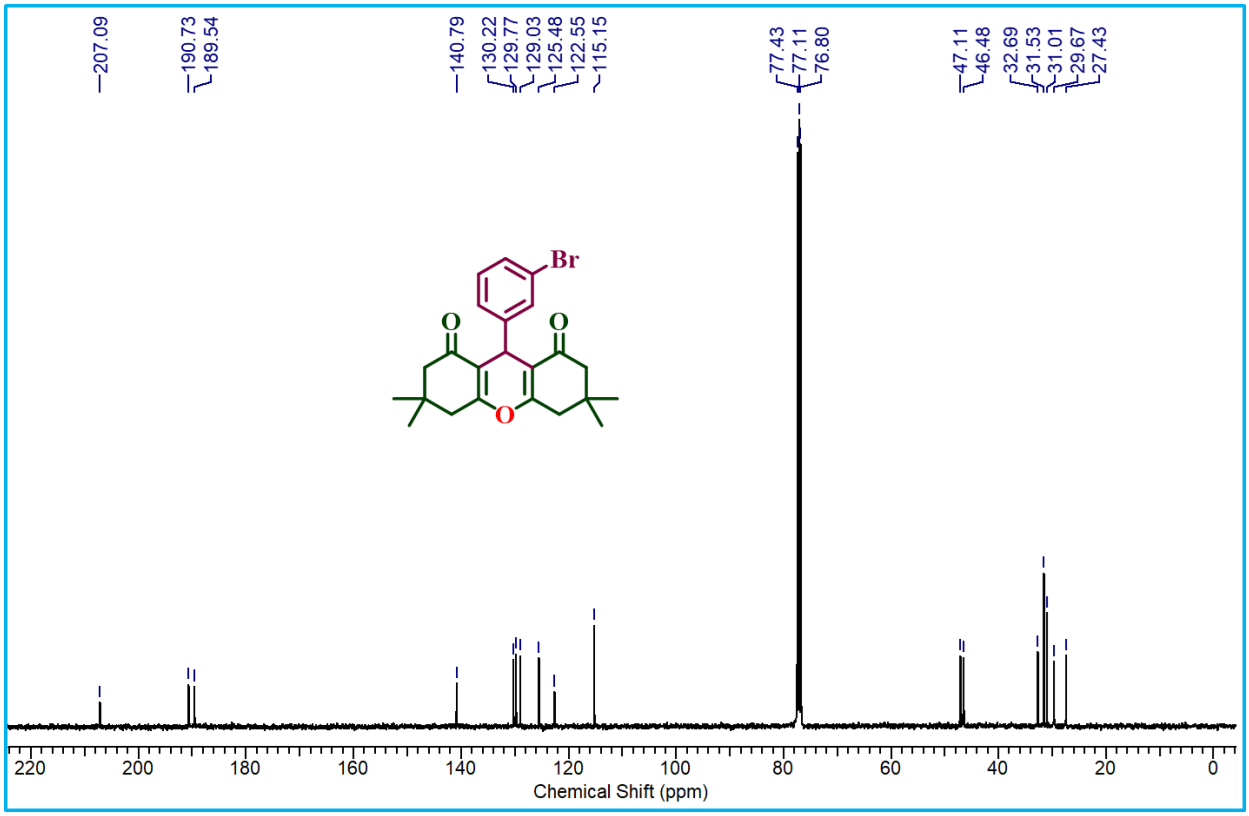

8.05
8.03
8.03
8.00
7.46
7.44
7.42
7.41
7.41
7.39
5.54
2.48
2.43
2.41
2.36
1.27
1.12
NO2
O
O
O
1.00
0.98
2.01
1.00
8.11
5.90
5.57
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)


191.26
189.78
148.53
140.79
133.01
129.23
122.36
121.19
114.90
47.12
46.55
32.99
31.57
29.85
27.39
NO2
O
O
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

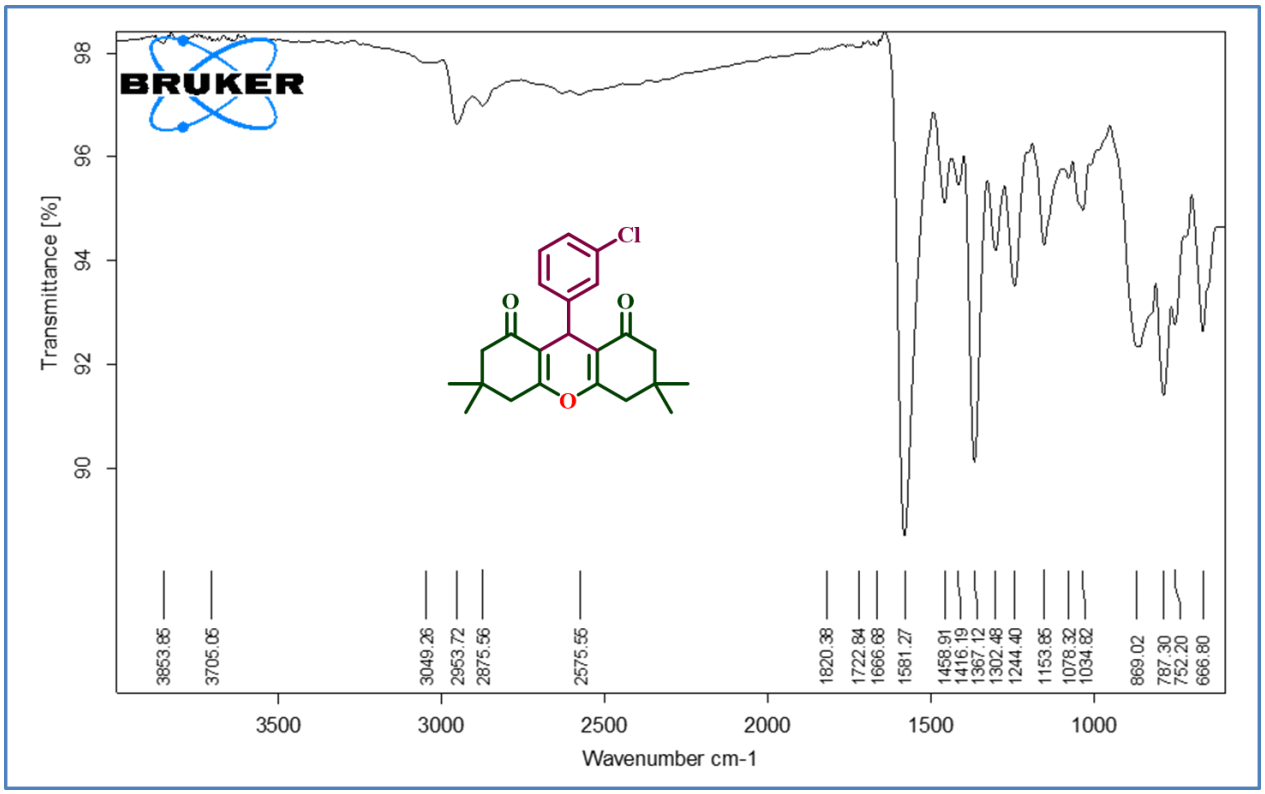
9-(3-bromophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3j).

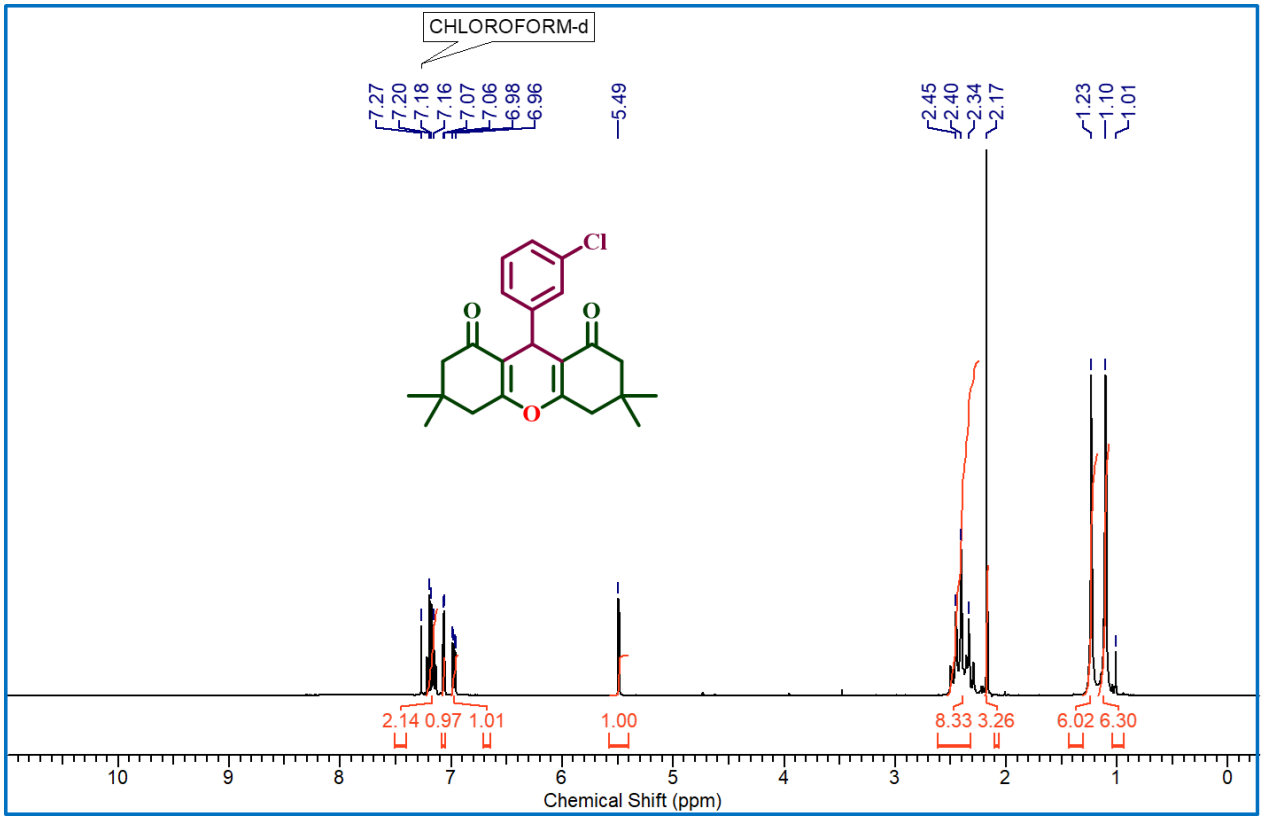


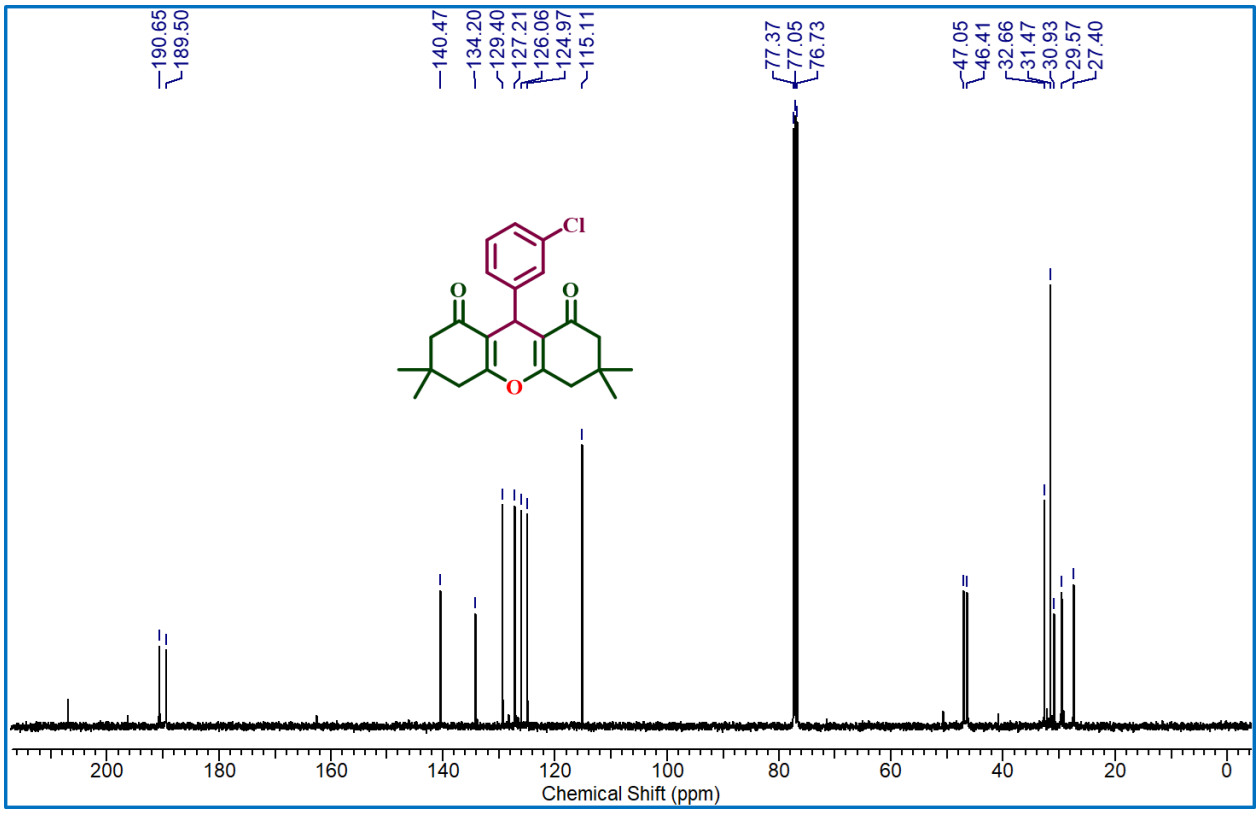




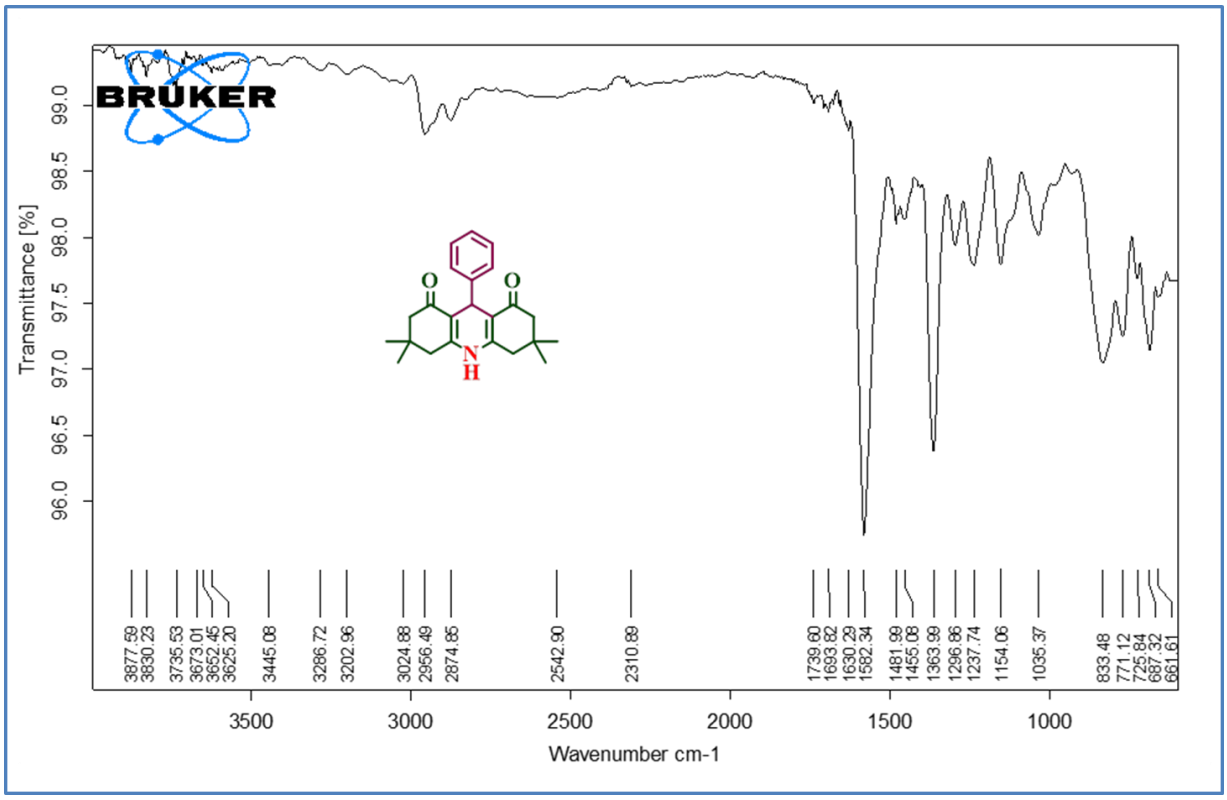
9-(3-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (3l).

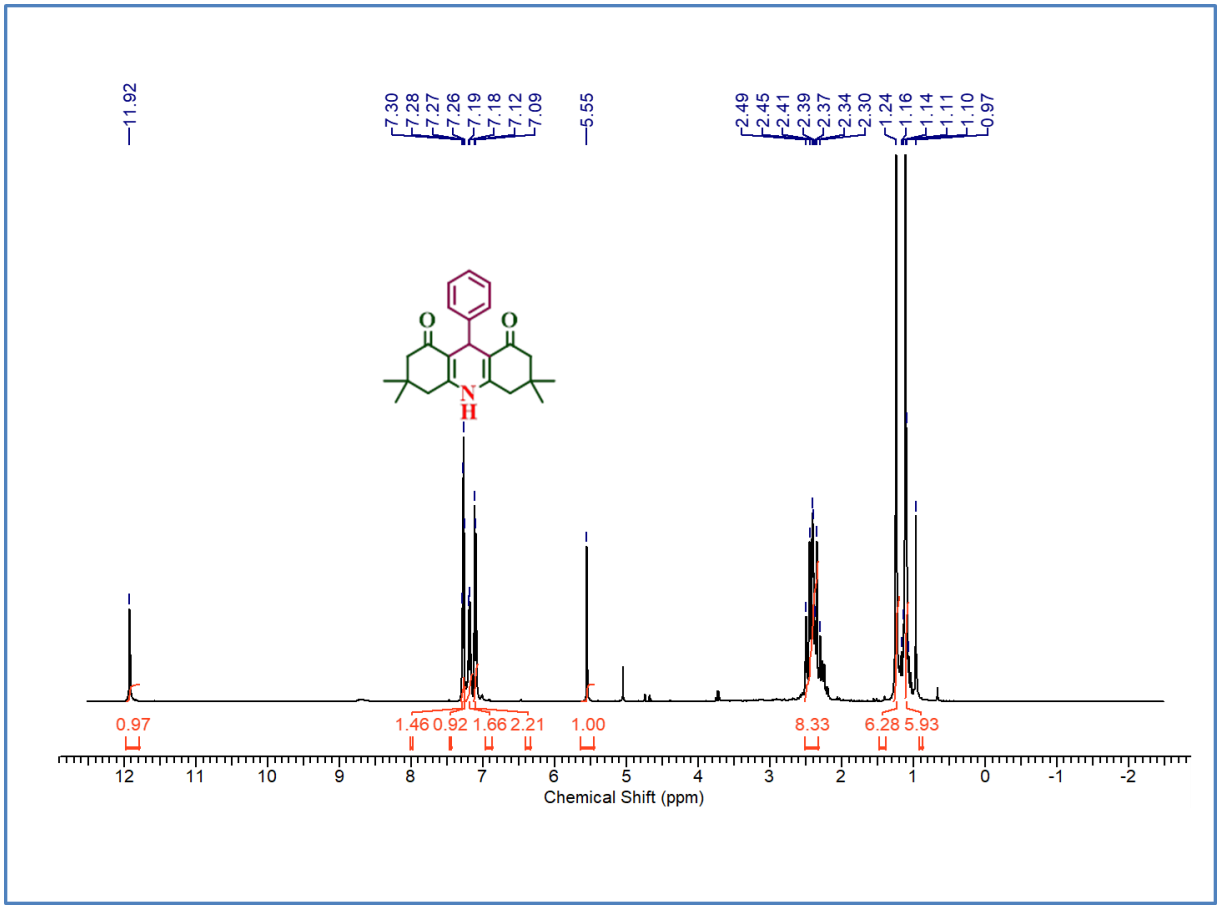


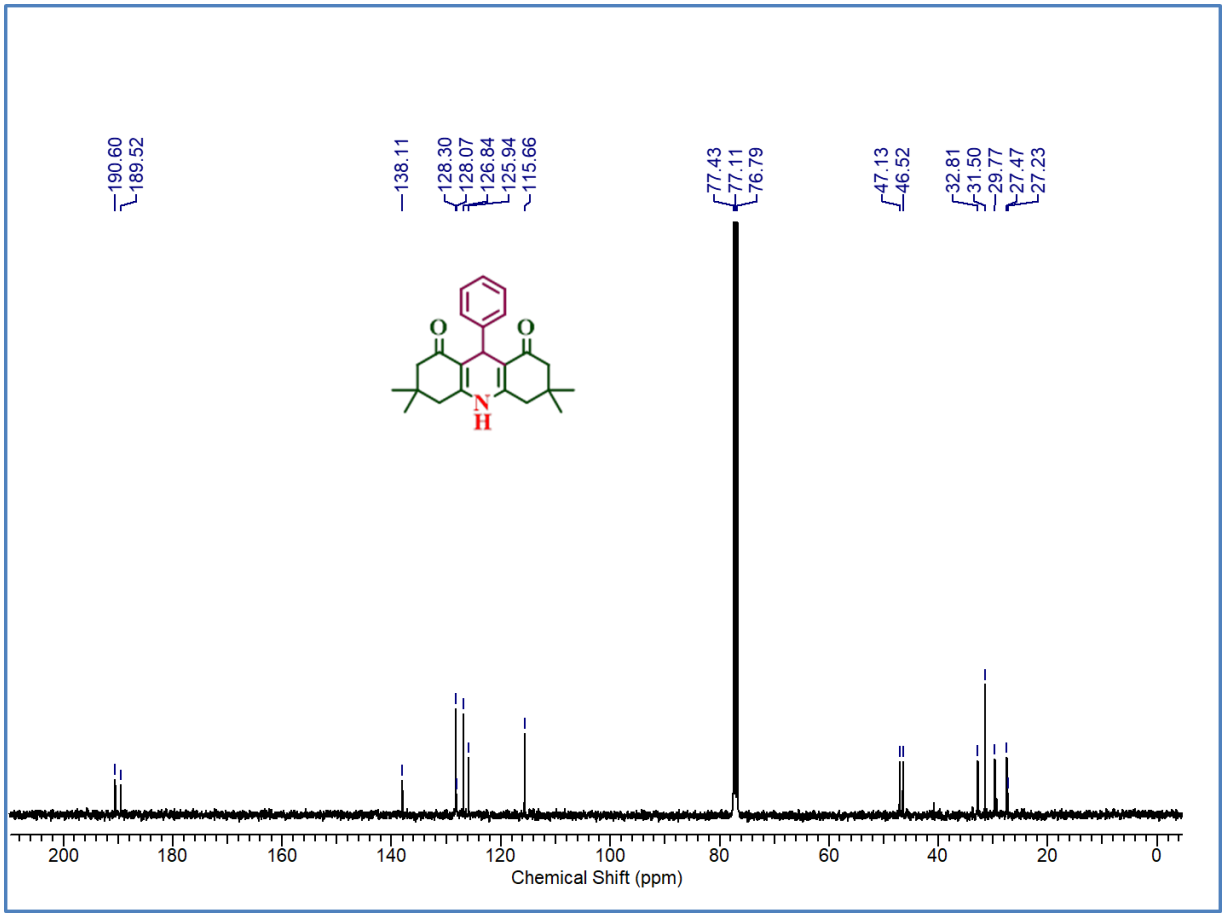

CHLOROFORM-d
7.27
7.20
7.18
7.16
7.07
7.06
6.98
6.96
5.49
2.45
2.40
2.34
2.17
1.23
1.10
1.01
Cl
O
O
O
2.14
0.97
1.01
1.00
8.33
3.26
6.02
6.30
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


190.65
189.50
140.47
134.20
129.40
127.21
126.06
124.97
115.11
77.37
77.05
76.73
47.05
46.41
32.66
31.47
30.93
29.57
27.40
Cl
O
O
O
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

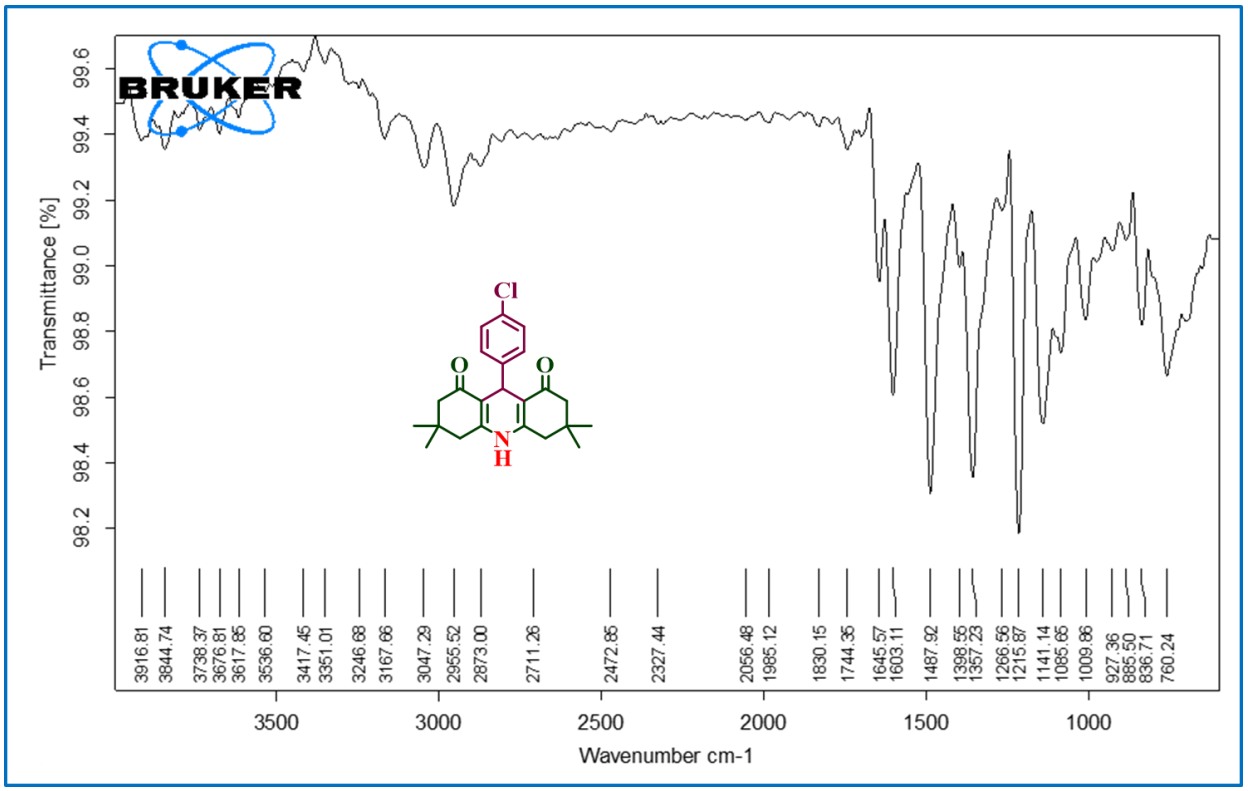
3,3,6,6-tetramethyl-9-phenyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4a).

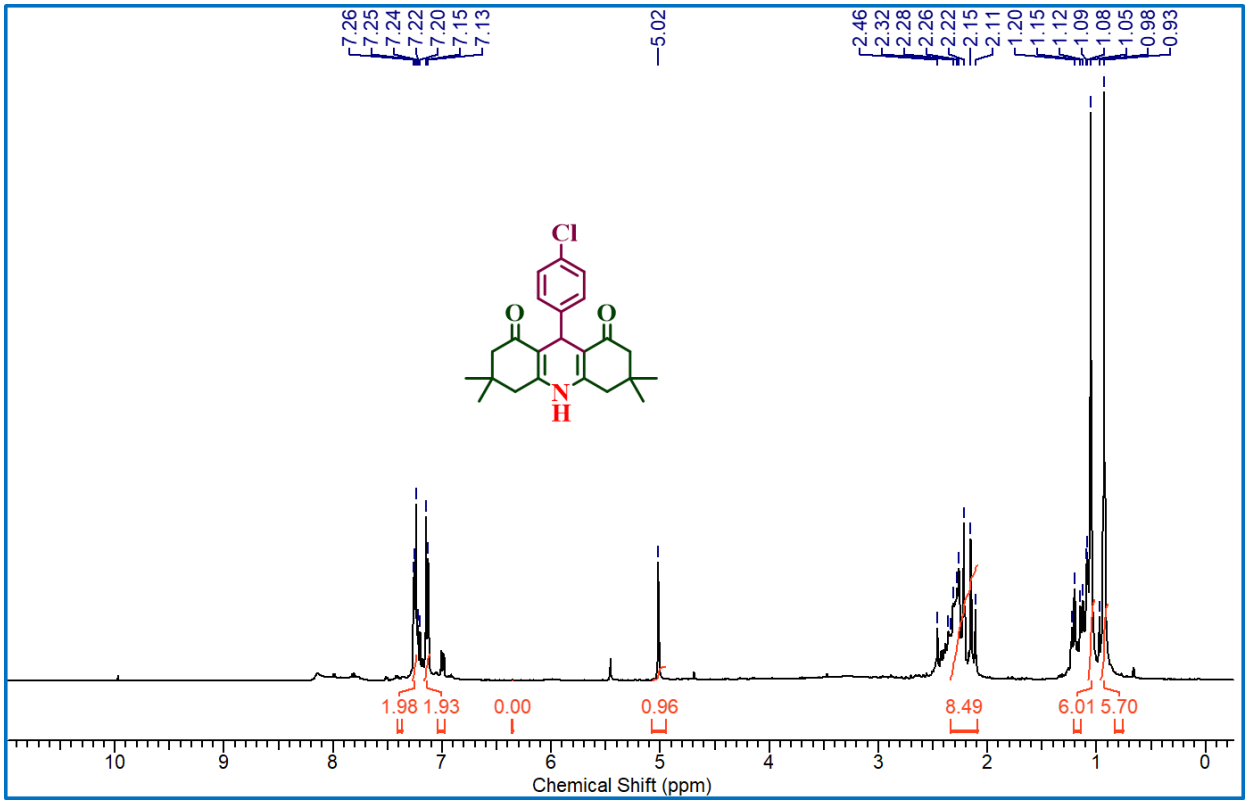


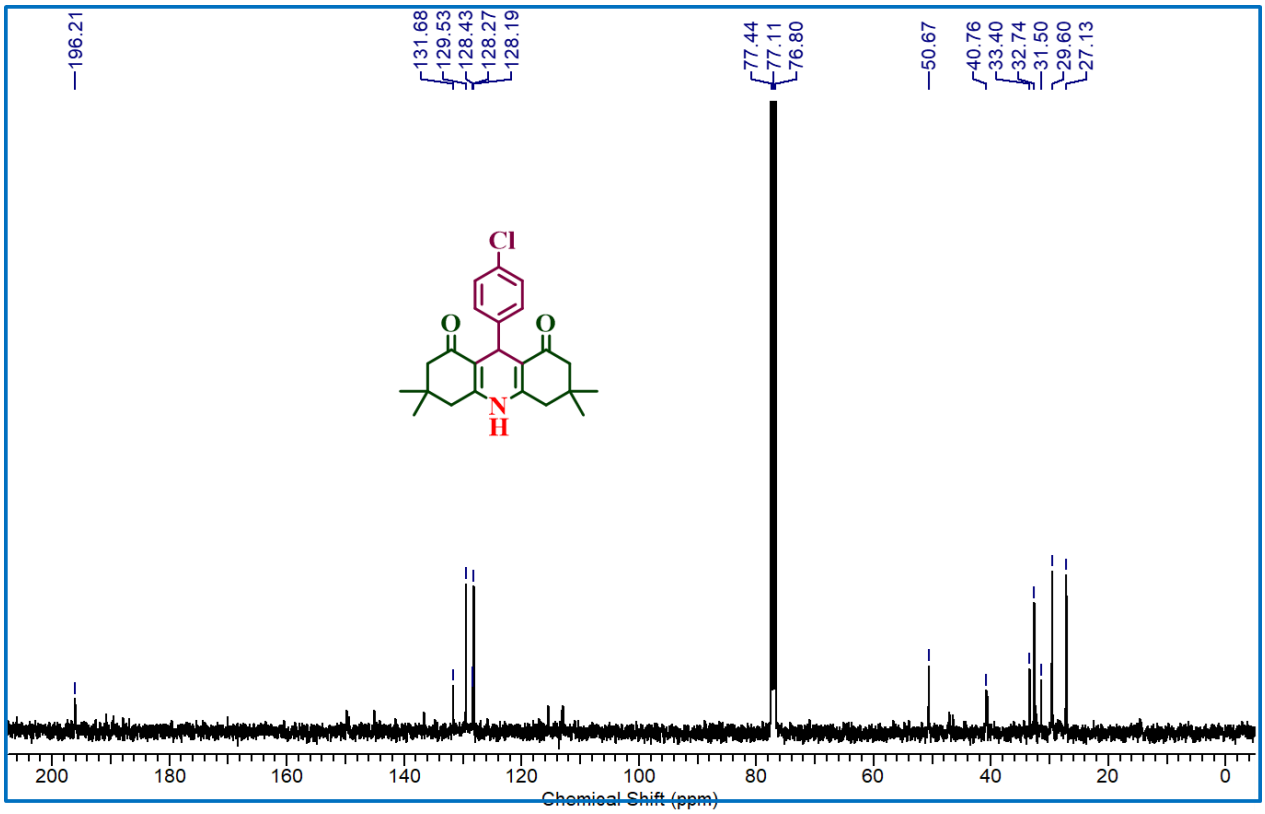




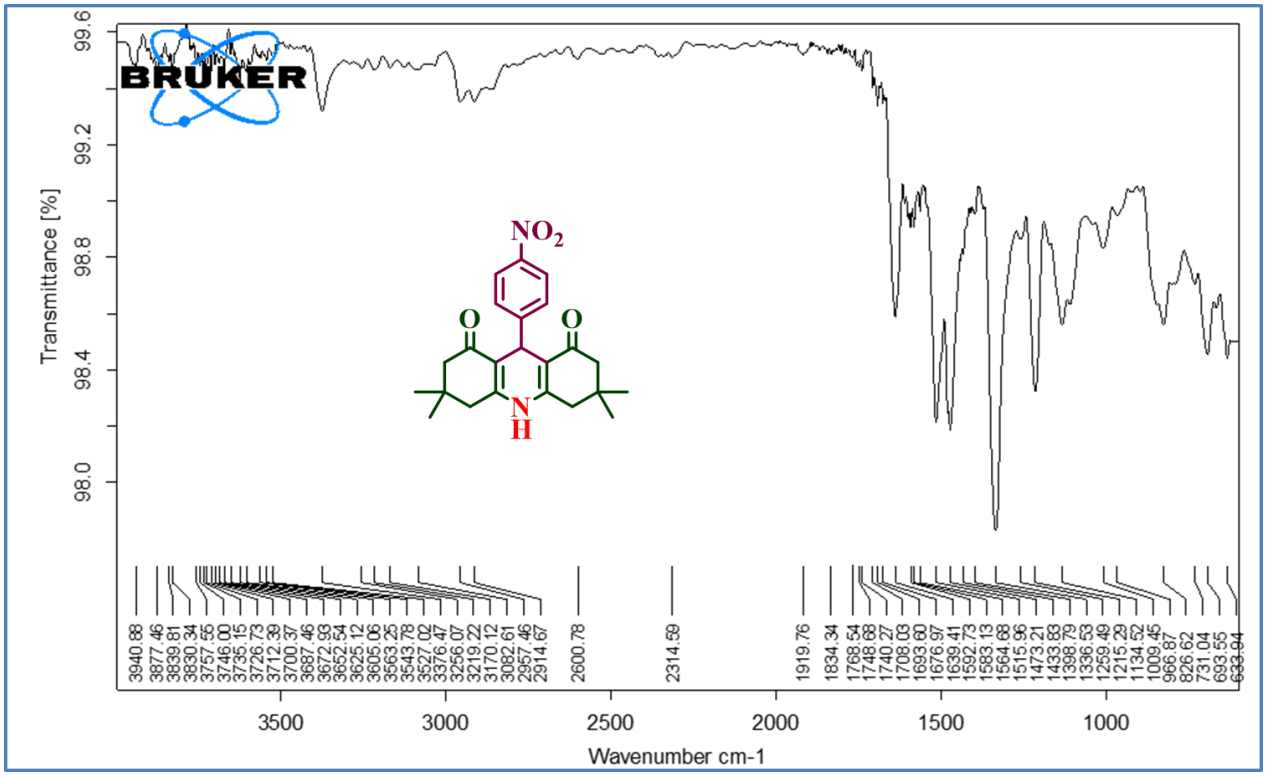
9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4b).

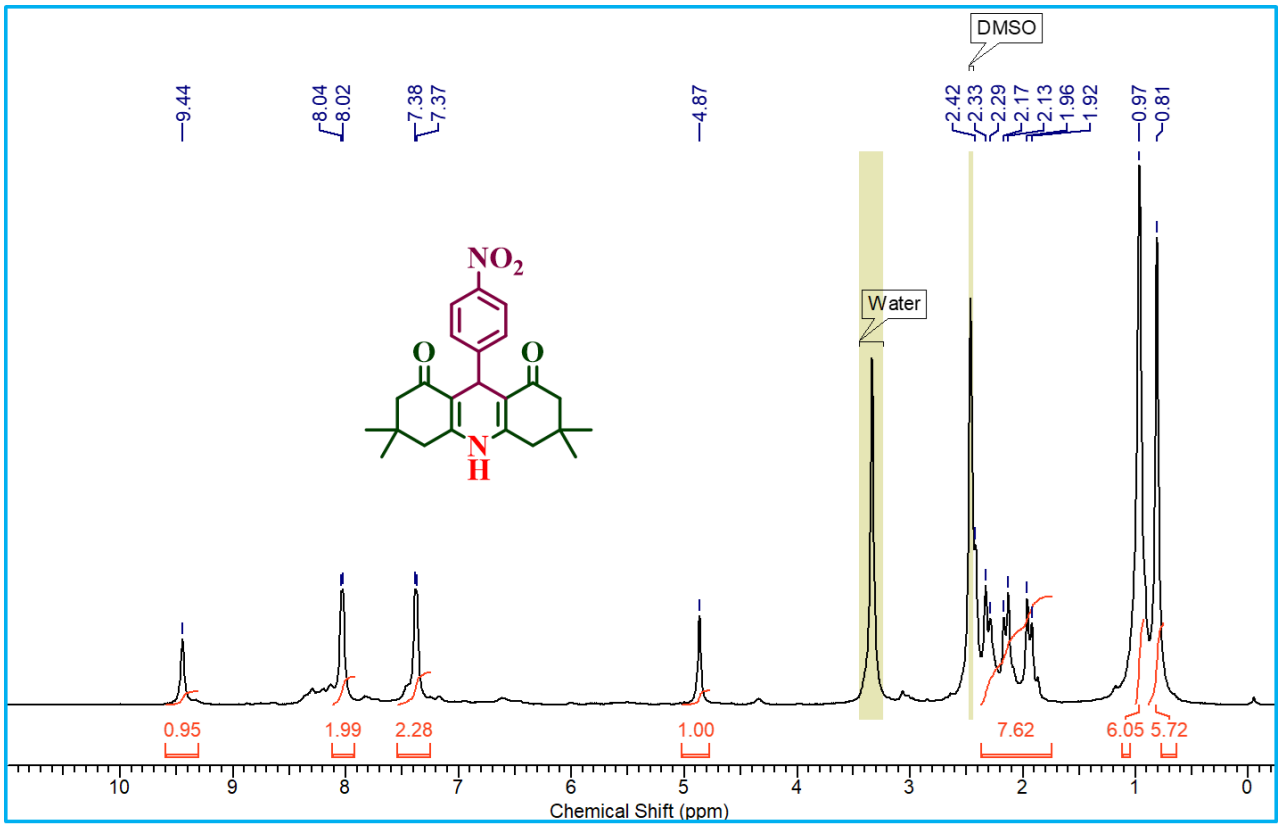


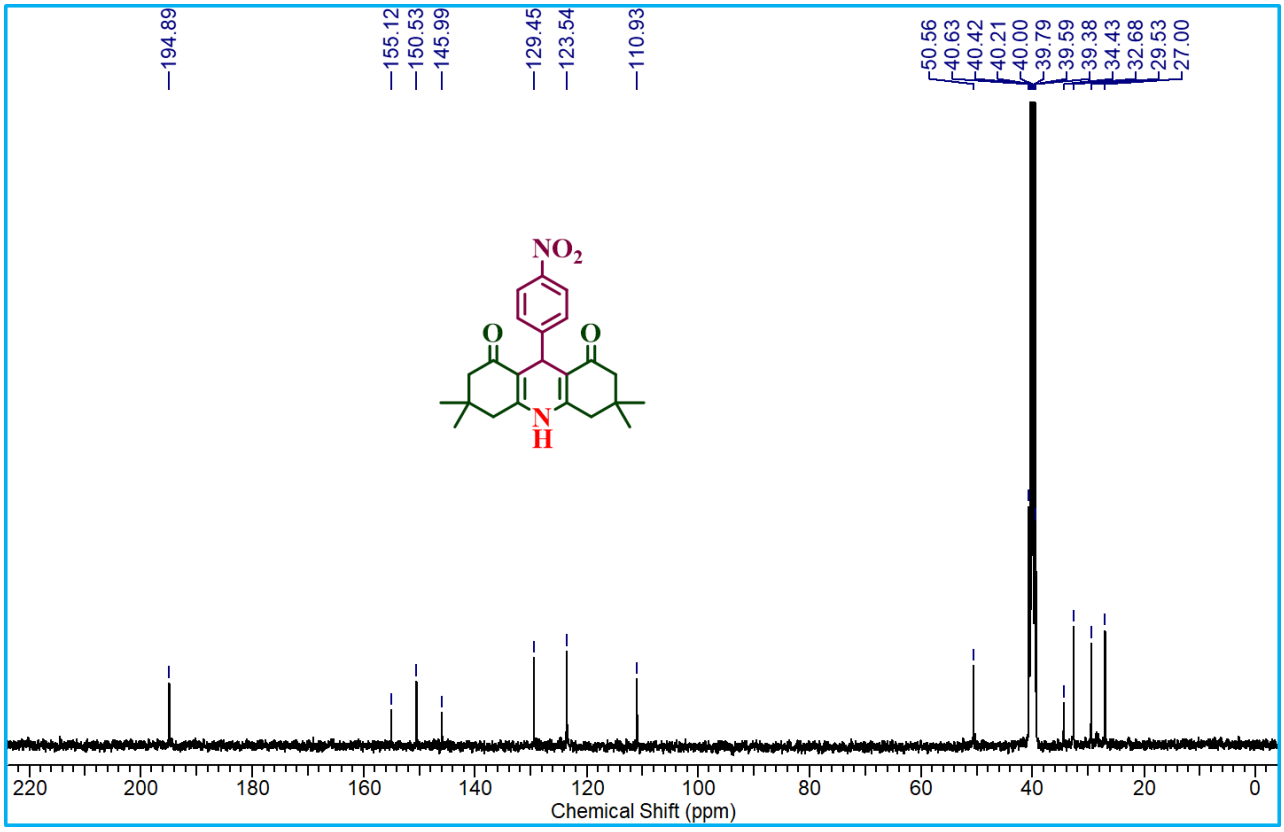

7.26
7.25
7.24
7.22
7.20
7.15
7.13
5.02
2.46
2.32
2.28
2.26
2.22
2.15
2.11
1.20
1.15
1.12
1.09
1.08
1.05
0.98
0.93
Cl
O
O
N
H
1.98
1.93
0.00
0.96
8.49
6.01
5.70
Chemical Shift (ppm)


196.21
131.68
129.53
128.43
128.27
128.19
77.44
77.11
76.80
50.67
40.76
33.40
32.74
31.50
29.60
27.13
Cl
O
O
N
H
Chemical Shift (ppm)

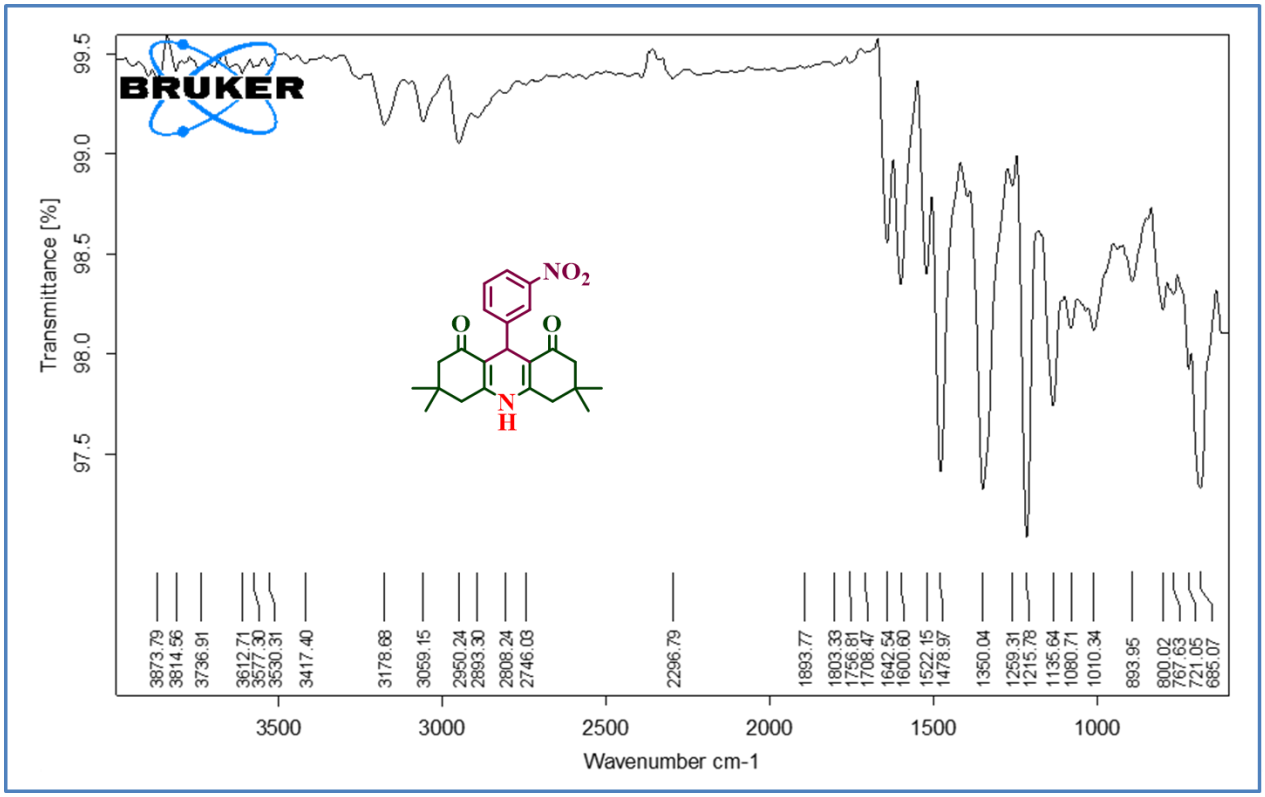
3,3,6,6-tetramethyl-9-(4-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4c).

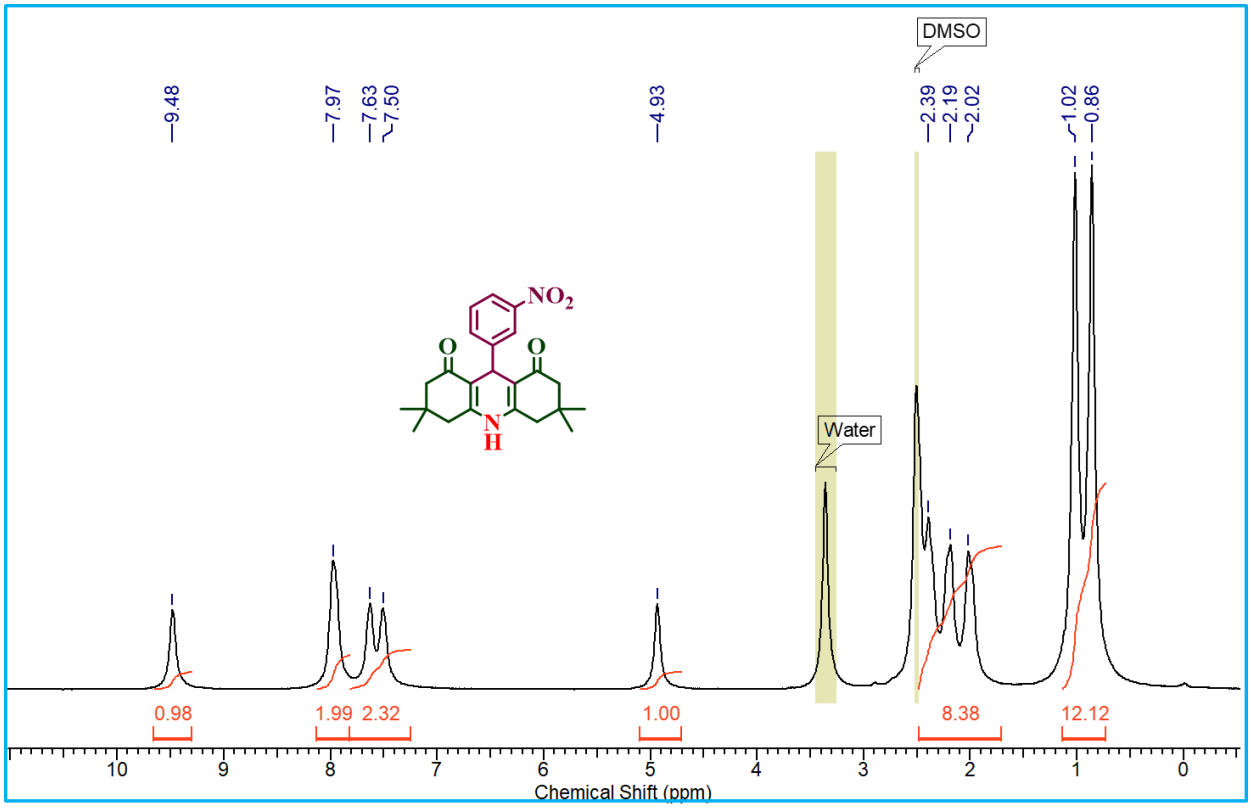


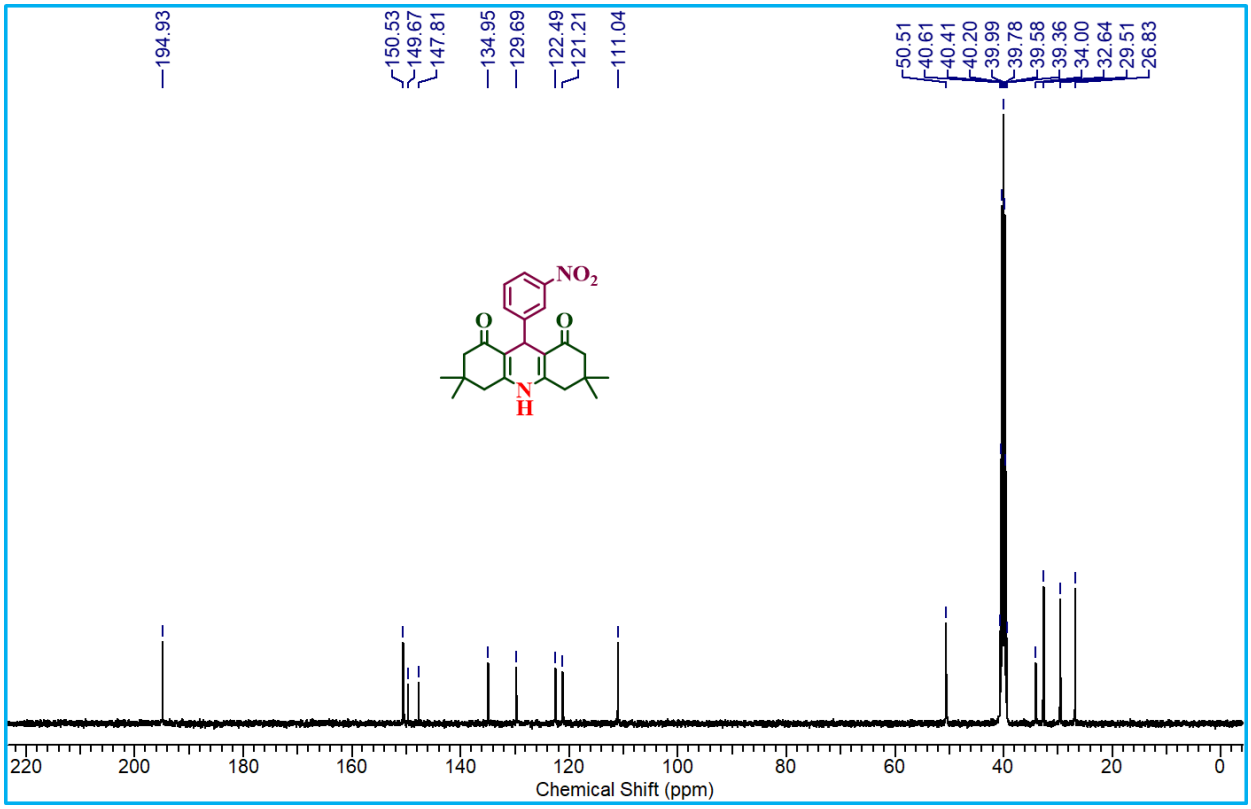




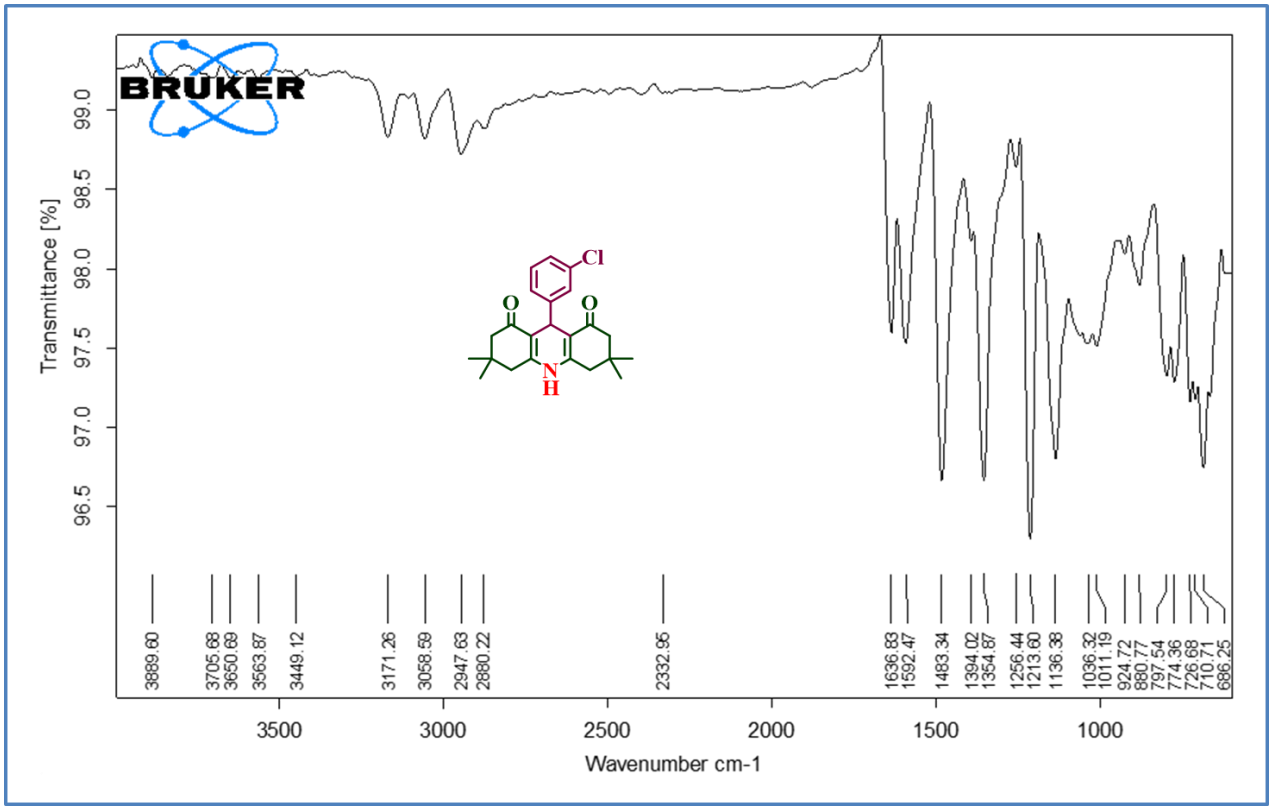
3,3,6,6-tetramethyl-9-(3-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4e).

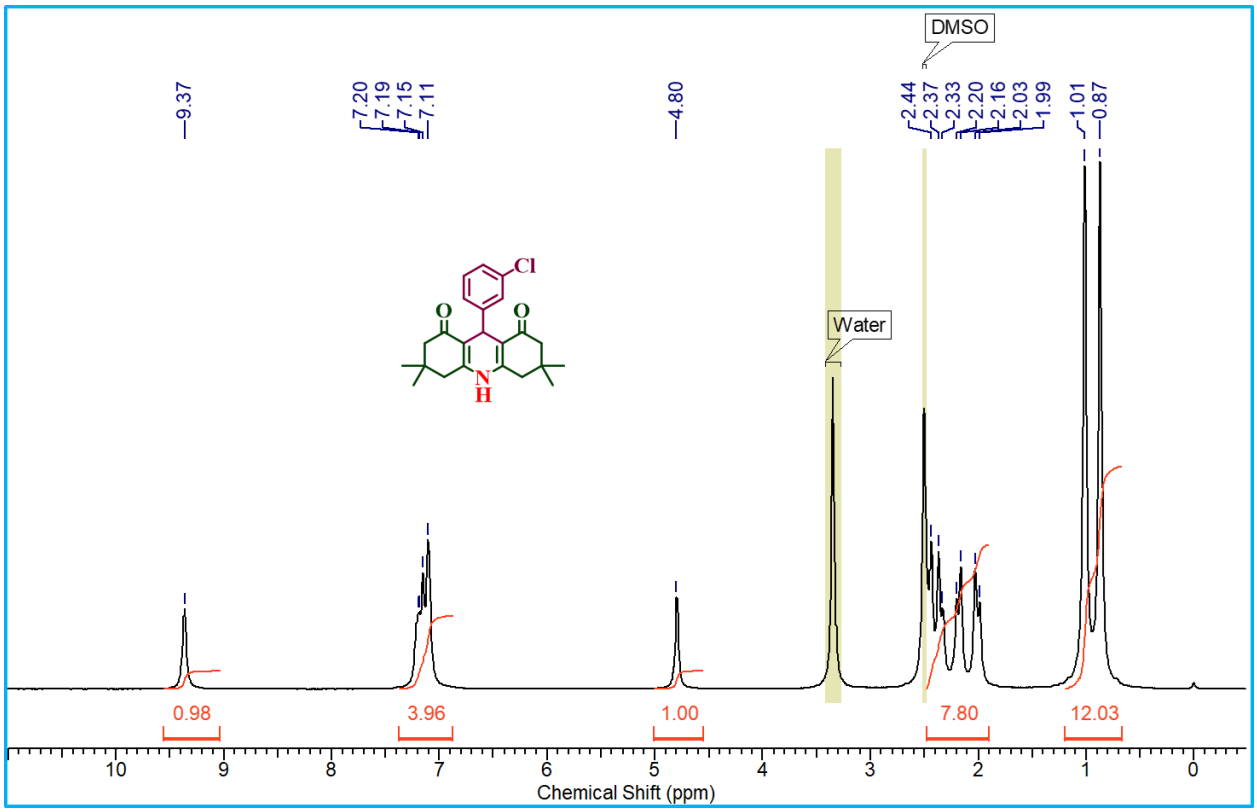


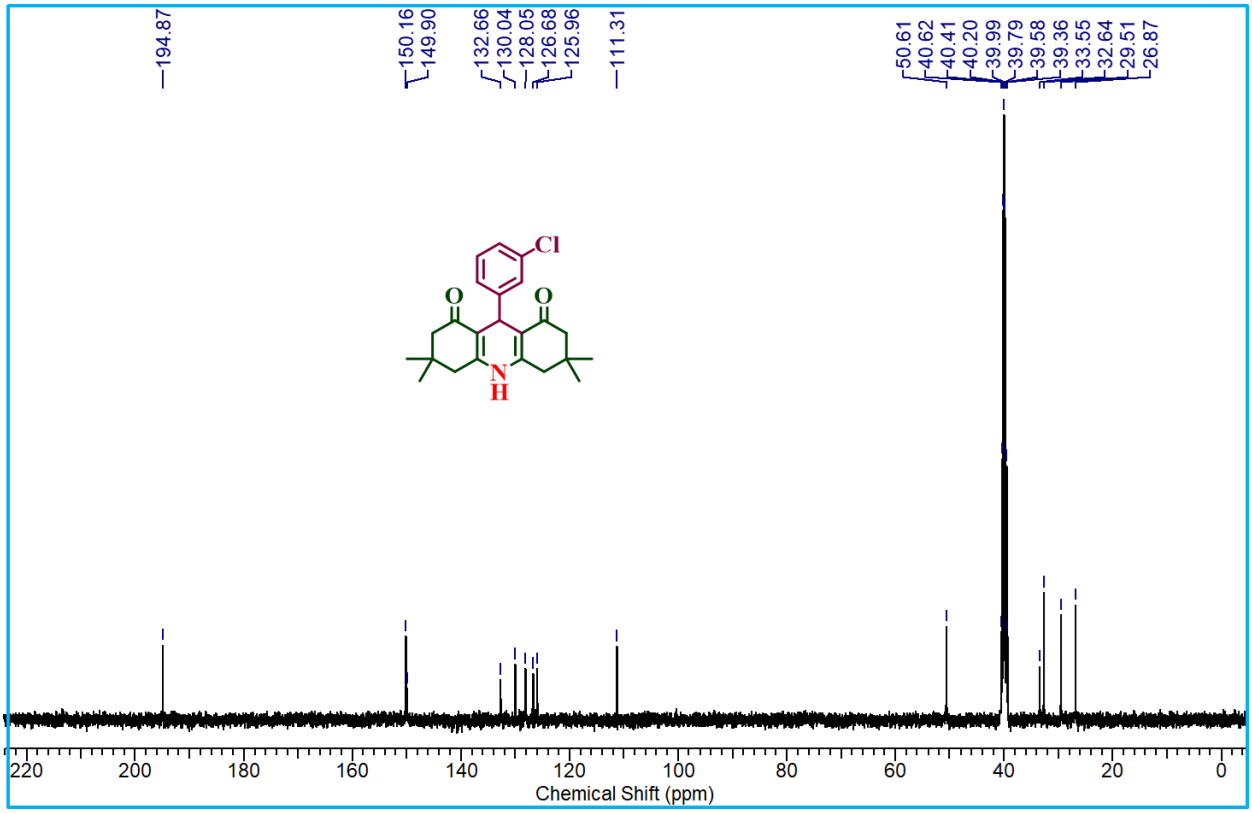

DMSO
9.48
7.97
7.63
7.50
4.93
2.39
2.19
2.02
1.02
0.86
Water
0.98
1.99
2.32
1.00
8.38
12.12
Chemical Shift (ppm)


194.93
150.53
149.67
147.81
134.95
129.69
122.49
121.21
111.04
50.51
40.61
40.41
40.20
39.99
39.78
39.58
39.36
34.00
32.64
29.51
26.83
Chemical Shift (ppm)

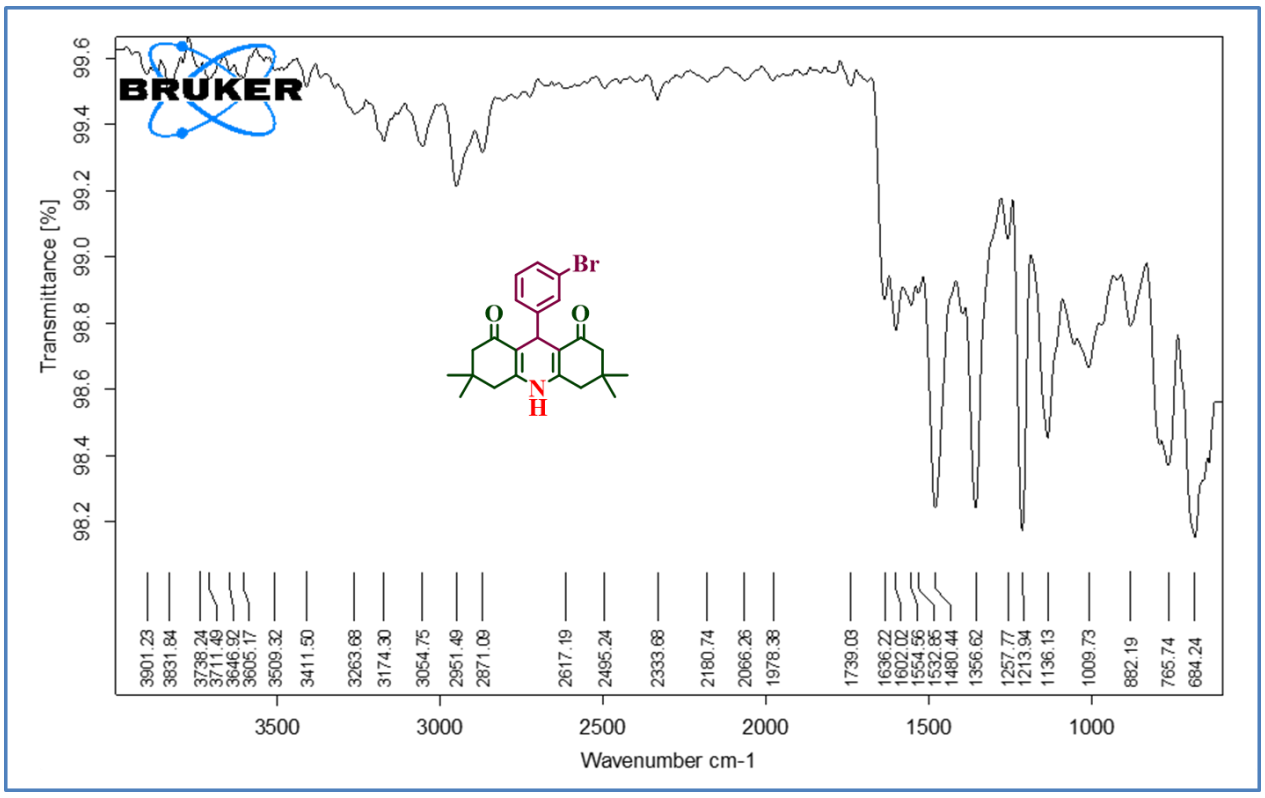
9-(3-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4g).

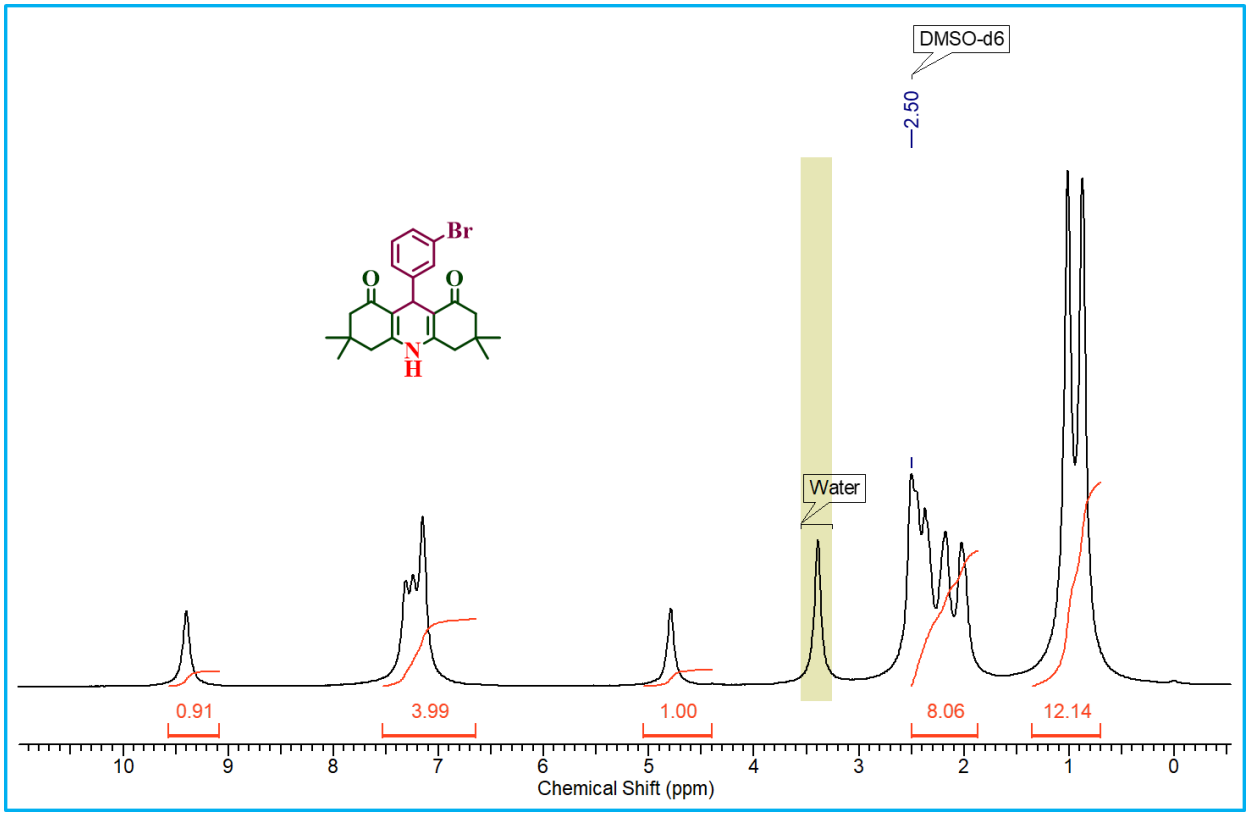


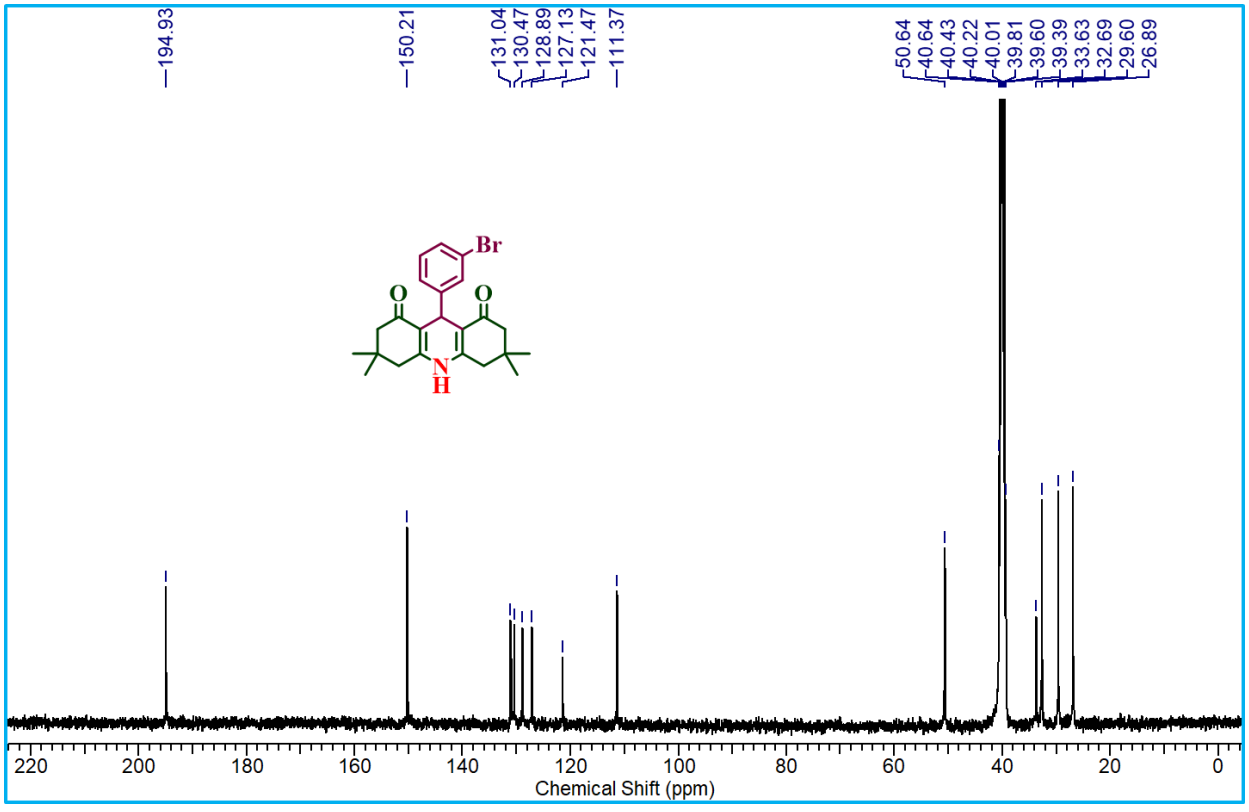




9-(3-bromophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4h)




DMSO-d6
2.50
Br
O
O
N
H
Water
0.91
3.99
1.00
8.06
12.14
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


194.93
150.21
131.04
130.47
128.89
127.13
121.47
111.37
50.64
40.64
40.43
40.22
40.01
39.81
39.60
39.39
33.63
32.69
29.60
26.89
Br
O
O
N
H
220
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)