Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Visible-Light Mediated Tryptanthrin Derivatives Synthesis from Isatin and Isatoic Anhydride under Transition Metal-Free Conditions

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1. General information

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents used in reactions were p.A. grade and dried only if indicated. Solvents for chromatography were technical grade and distilled prior to use. Analytical thin-layer chromatography (TLC) was performed on Merck silica gel aluminium plates with F-254 indicator, visualized by irradiation with UV light. Column chromatography was performed using silica gel Merck 60 (particle size 0.063 – 0.2 mm). Solvent mixtures are understood as volume/volume.

^{1}H-NMR, ^{13}C-NMR were recorded on a Variance VNMR 400 or Bruker AV-600 spectrometer in $CDCl_3$ or DMSO-d_6. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated s (singlet), d (doublet), t (triplet), m (multiplet), dd (doublet of doublets), dt (doublet of triplets), td (triplet of doublets); coupling constants (J) are in Hertz (Hz). Mass spectra (MS-EI, 70 eV) were conducted on Firma Finnigan SHIMADZU LCMS-2020. IR spectra were recorded on a Perkin Elmer Spectrum 100 FTIR (KBr disc) and are reported in terms of frequency of absorption (cm^{-1}).

2. General procedure for the Synthesis of Tryptanthrin derivatives from Isatin

An oven-dried vial equipped with a stirrer bar was charged with Rose Bengal (0.01 mmol, 0.05 equiv.), K_2CO_3 (0.2 mmol, 1.0 equiv.), Isatin **1** (0.20 mmol) and DMF (2 mL) as solvent. The solution was stirred for 24-36h irradiated by 23 W Fluorescent bulb under the room temperature. After the Isatin **1** was completely consumed (monitored by TLC), the crude mixture was directly purified by flash column chromatography on silica gel (EtOAc/petroleum ether 1:3) to give the desired product **2a**-**2e**.

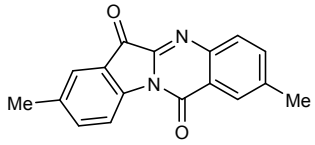
3. General procedure for the Synthesis of Tryptanthrin derivatives from Isatin and Isatoic anhydride

An oven-dried vial equipped with a stirrer bar was charged with Rose Bengal (0.005 mmol, 0.05 equiv.), K_2CO_3 (0.1 mmol, 1.0 equiv.), Isatin **1** (0.10 mmol),

Isatoic anhydride **3** (0.24 mmol) and DMF (2 mL) as solvent. The solution was stirred for 12-24h irradiated by 23 W Fluorescent bulb under the room temperature. After the Isatin **1** was completely consumed (monitored by TLC), the crude mixture was directly purified by flash column chromatography on silica gel (EtOAc/petroleum ether 1:3) to give the desired product **2a**-**2c**, **2e**-**2o**.

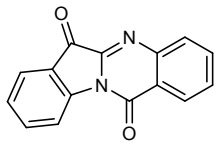
4. characterizations of Products

2,8-dimethyindolo(2,1-*b*)quinazoline-6,12-dione[1] (2a):



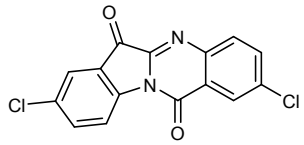
Yellow soild; m.p. 249.8–251.6 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.46 (d, *J* = 8.2 Hz, 1H), 8.20 (s, 1H), 7.89 (d, *J* = 8.2 Hz, 1H), 7.68 (s, 1H), 7.63 (dd, *J* = 8.2, 1.9 Hz, 1H), 7.56 (dd, *J* = 8.2, 1.0 Hz, 1H), 2.54 (s, 3H), 2.44 (s, 3H); IR (neat): *v* =1674, 1599, 1476, 1327, 1205, 1122, 1038, 844 cm^{-1}; MS-EI: m/z(%): 277.05 (M+H$^+$, 100%), 299.05 (M+Na$^+$, 18%).

Indolo(2,1-*b*)quinazoline-6,12-dione[1] (2b):



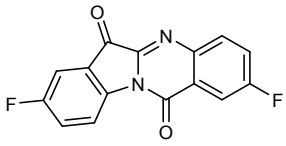
Yellow soild; m.p. 267.4–269.2 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.63 (d, *J* = 8.1 Hz, 1H), 8.44 (d, *J* = 8.0 Hz, 1H), 8.03 (d, *J* = 8.1 Hz, 1H), 7.92 (d, *J* = 7.6 Hz, 1H), 7.85 (t, *J* = 7.7 Hz, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 1H); IR (neat): *v* =1726, 1679, 1590, 1459, 1308, 1189, 1105, 758, 688 cm^{-1}; MS-EI: m/z(%): 249.05 (M+H$^+$, 100%), 270.00 (M+Na$^+$, 31%).

2,8-dichloroindolo(2,1-*b*)quinazoline-6,12-dione[1] (2c):



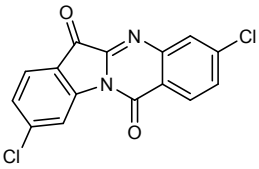
Yellow soild; m.p. 288.4–290.2 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.58 (d, *J* = 8.5 Hz, 1H), 8.40 (d, *J* = 2.2 Hz, 1H), 7.97 (d, *J* = 8.6 Hz, 1H), 7.88 (d, *J* = 1.9 Hz, 1H), 7.80 (dd, *J* = 8.6, 2.3 Hz, 1H), 7.75 (dd, *J* = 8.6, 2.1 Hz, 1H); IR (neat): *v* = 3070, 1736, 1671, 1588, 1456, 1324, 1185, 844, 763, 639 cm^{-1}; MS-EI: m/z(%): 316.90 (M+H$^+$, 57%), 338.65 (M+Na$^+$, 100%).

2,8-difluoroindolo(2,1-*b*)quinazoline-6,12-dione[1] (2d):



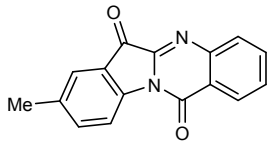
Yellow soild; m.p. 292.3–294.8 °C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 8.49 (dd, *J* = 8.7, 4.1 Hz, 1H), 8.05 (dt, *J* = 8.4, 4.2 Hz, 2H), 7.86 (dd, *J* = 8.8, 2.8 Hz, 1H), 7.84 – 7.79 (m, 1H), 7.74 (td, *J* = 9.0, 2.8 Hz, 1H); IR (neat): *v* =1733, 1672, 1608, 1475, 1350, 1289, 1210, 849, 756, 678 cm^{-1}; MS-EI: m/z(%): 285.00 (M+H$^+$, 100%), 307.02 (M+Na$^+$, 74%).

3,9-dichloroindolo(2,1-*b*)quinazoline-6,12-dione (2e):



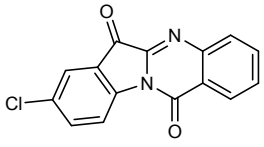
Yellow soild; m.p. 298.2–300.2 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.67 (s, 1H), 8.37 (d, *J* = 8.5 Hz, 1H), 8.01 (d, *J* = 1.7 Hz, 1H), 7.85 (d, *J* = 8.2 Hz, 1H), 7.64 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.42 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz): 181.0, 157.3, 147.6, 146.7, 145.1, 144.8, 141.7, 130.9, 130.1, 129.0, 127.9, 126.3, 121.8, 120.1, 118.5; IR (neat): *v* = 3084, 1690, 1587, 1423, 1346, 1290, 1193, 1109, 1045, 928, 768, 688, 622 cm^{-1}; MS-EI: m/z(%): 316.90 (M+H$^+$, 100%), 338.85 (M+Na$^+$, 89%)

8-methylindolo(2,1-*b*)quinazoline-6,12-dione[1] (2f):



Yellow soild; m.p. 282.4–283.9 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.47 (d, *J* = 8.2 Hz, 1H), 8.42 (d, *J* = 7.9 Hz, 1H), 8.01 (d, *J* = 8.1 Hz, 1H), 7.83 (t, *J* = 7.5 Hz, 1H), 7.69 (s, 1H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.57 (d, *J* = 8.3 Hz, 1H), 2.45 (s, 3H); IR (neat): *v* =3086, 2983, 1687, 1597, 1478, 1326, 1205, 1134, 1040, 890, 831, 776, 690 cm^{-1}; MS-EI: m/z(%): 263.05 (M+H$^+$, 100%), 285.00 (M+Na$^+$, 23%).

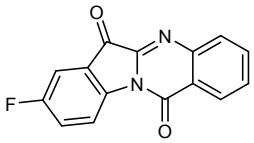
8-chloroindolo(2,1-*b*)quinazoline-6,12-dione[1] (2g):



Yellow soild; m.p. 294.4–295.2 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.60 (d, *J* = 8.6 Hz, 1H), 8.44 (dd, *J* = 7.9, 1.1 Hz, 1H), 8.03 (dd, *J* = 8.1, 0.6 Hz, 1H), 7.88 (dd, *J* = 6.1, 1.1 Hz, 1H), 7.86 (dd, *J* = 7.6, 0.9 Hz, 1H), 7.74 (dd, *J* = 8.6, 2.2 Hz, 1H), 7.72–7.67 (m, 1H); IR

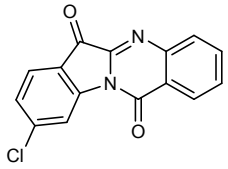
(neat): v = 3069, 1672, 1592, 1452, 1325, 1185, 1115, 1043, 872, 774, 685 cm^{-1}; MS-EI: m/z(%): 282.95 ($M+H^+$, 100%), 304.93 ($M+Na^+$, 72%).

8-fluoroindolo(2,1-*b*)quinazoline-6,12-dione[4] (2h):



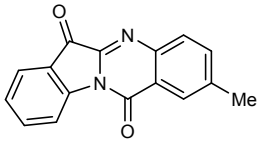
Yellow soild; m.p. 289.0–291.0 ^oC; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.64 (dd, J = 8.8, 4.1 Hz, 1H), 8.43 (dd, J = 7.9, 1.1 Hz, 1H), 8.03 (dd, J = 8.1, 0.6 Hz, 1H), 7.89–7.82 (m, 1H), 7.71–7.66 (m, 1H), 7.58 (dd, J = 6.5, 2.7 Hz, 1H), 7.48 (td, J = 8.7, 2.7 Hz, 1H); IR (neat): v = 3082, 1689, 1598, 1478, 1348, 895, 832, 772, 683 cm^{-1}; MS-EI: m/z(%): 267.00 ($M+H^+$, 100%), 289.00 ($M+Na^+$, 53%)

9-chloroindolo(2,1-*b*)quinazoline-6,12-dione[2,3] (2i):



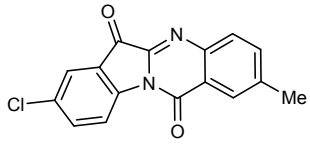
Yellow soild; m.p. 237.2–239.0 ^oC; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.69 (d, J = 1.4 Hz, 1H), 8.45 (d, J = 7.9 Hz, 1H), 8.04 (d, J = 8.0 Hz, 1H), 7.90–7.87 (m, 1H), 7.86–7.83 (m, 1H), 7.70 (t, J = 7.6 Hz, 1H), 7.41 (dd, J = 8.1, 1.7 Hz, 1H); IR (neat): v = 1674, 1587, 1425, 1340, 1340, 1198, 1115, 1043, 877, 773, 685 cm^{-1}; MS-EI: m/z(%): 282.95 ($M+H^+$, 100%), 304.93 ($M+Na^+$, 69%).

2-methyindolo(2,1-*b*)quinazoline-6,12-dione[1] (2j):



Yellow soild; m.p. 251.4–253.2 ^oC; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.62 (d, J = 8.1 Hz, 1H), 8.22 (s, 1H), 7.91 (dd, J = 7.7, 4.3 Hz, 2H), 7.77 (t, J = 7.7 Hz, 1H), 7.65 (d, J = 8.1 Hz, 1H), 7.41 (t, J = 7.5 Hz, 1H), 2.55 (s, 3H); IR (neat): v =1982, 2879, 1724, 1682, 1594, 1482, 1313, 1204, 846,763 cm^{-1}; MS-EI: m/z(%): 263.05 ($M+H^+$, 100%), 285.05 ($M+Na^+$, 28%)

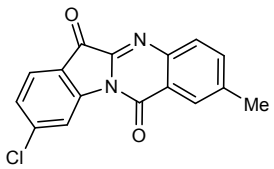
8-chloro-2-methylindolo(2,1-*b*)quinazoline-6,12-dione[1] (2k):



Yellow soild; m.p. 281.6–282.4 ^oC; ^1H NMR ($CDCl_3$, 400 MHz): δ 8.57 (d, J = 8.6 Hz, 1H), 8.19 (s, 1H), 7.90 (d, J =

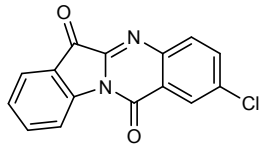
8.2 Hz, 1H), 7.84 (s, 1H), 7.71 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.65 (d, *J* = 8.2 Hz, 1H), 2.55 (s, 3H); IR (neat): *v* =3049, 1730, 1675, 1593, 14671, 1339, 1301, 1182, 1119, 1037, 841, 784, 692, 632 cm^{-1}; MS-EI: m/z(%): 296.95 ($M+H^+$, 100%), 318.95 ($M+Na^+$, 43%).

9-chloro-2-methylindolo(2,1-*b*)quinazoline-6,12-dione[3] (2l):



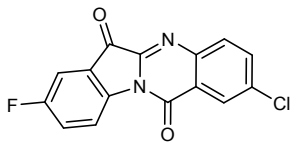
Yellow soild; m.p. 222.0–223.4 ^oC; ^1H NMR ($CDCl_3$, 400 MHz): δ 8.69 (s, 1H), 8.22 (s, 1H), 7.92 (d, *J* = 8.2 Hz, 1H), 7.84 (d, *J* = 8.1 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 1H), 2.57 (s, 3H); IR (neat): ν = 3072, 1724, 1679, 1588, 1463, 1424, 1346, 1285, 1088, 926, 876, 842, 785, 612 cm^{-1}; MS-EI: m/z(%): 297.00 ($M+H^+$, 100%), 219.00 ($M+Na^+$, 33%).

2-chloroindolo(2,1-*b*)quinazoline-6,12-dione (2m):



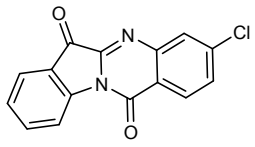
Yellow soild; m.p. 310.2–312.0 ^oC; ^1H NMR ($CDCl_3$, 400 MHz): δ 8.62 (d, *J* = 8.0 Hz, 1H), 8.40 (d, *J* = 2.3 Hz, 1H), 7.97 (d, *J* = 8.6 Hz, 1H), 7.92 (d, *J* = 7.7 Hz, 1H), 7.80 (t, *J* = 8.0 Hz, 2H), 7.45 (t, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz):182.2, 156.9, 146.0, 145.1, 144.5, 138.3, 136.6, 135.5, 132.0, 127.5, 127.1, 125.5, 124.9, 121.9, 118.0; IR (neat): ν = 3098, 1730, 1684, 1592, 1464, 1313, 1203, 1083, 901, 845, 758, 648 cm^{-1}; MS-EI: m/z(%): 282.95 ($M+H^+$, 100%), 304.20 ($M+Na^+$, 54%).

2-Chloro-8-fluoroindolo[2,1-b]quinazoline-6,12-dione[4] (2n):



Yellow soild; m.p. 279.4–281.2 ^oC; ^1H NMR ($CDCl_3$, 400 MHz): δ 8.63 (dd, *J* = 8.8, 4.0 Hz, 1H), 8.40 (d, *J* = 2.3 Hz, 1H), 7.97 (d, *J* = 8.6 Hz, 1H), 7.80 (dd, *J* = 8.6, 2.4 Hz, 1H), 7.59 (dd, *J* = 6.4, 2.6 Hz, 1H), 7.50 (td, *J* = 8.6, 2.7 Hz, 1H); IR (neat): *v* = 3061, 2945, 1728, 1652, 1450, 1218, 876, 825, 764, 695 cm^{-1}; MS-EI: m/z(%): 301.00 ($M+H^+$, 100%), 322.95 ($M+Na^+$, 37%)

3-chloroindolo(2,1-*b*)quinazoline-6,12-dione[3] (2o):

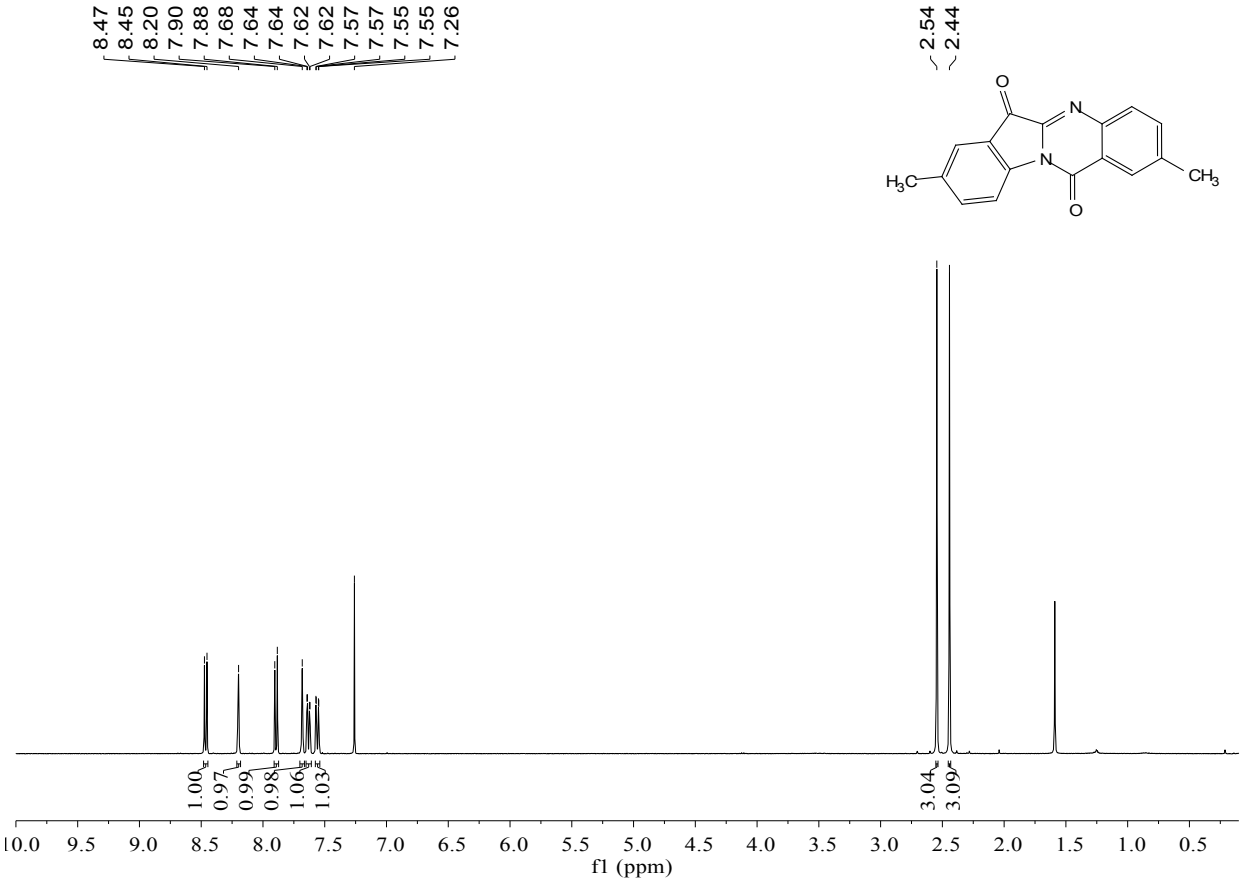


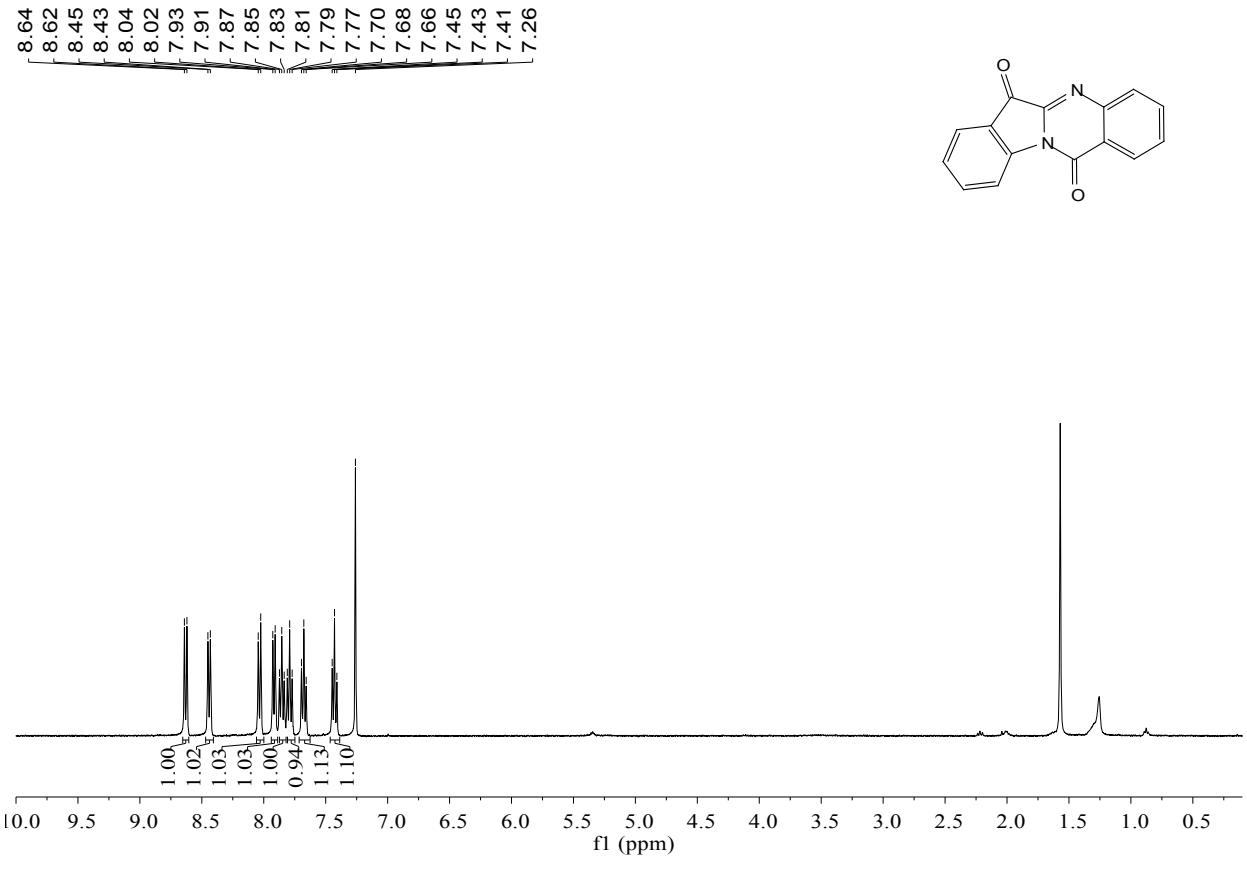
Yellow soild; m.p. 248.6–250.4 ^{o}C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 8.46 (d, *J* = 7.8 Hz, 1H), 8.31 (d, *J* = 8.4 Hz, 1H), 8.05 (s, 1H), 7.88 (t, *J* = 8.3 Hz, 2H), 7.78 (d, *J* = 8.2 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 1H); ^{13}C NMR (DMSO-d_6, 100 MHz): 183.1, 158.0, 148.6, 147.0, 146.8, 140.6, 138.7, 130.8, 129.9, 129.7, 123.1, 123.0, 117.9; IR (neat): *v* =3093, 1696, 1588, 1461, 1310, 1194, 1106, 932, 784, 685, 616 cm^{-1}; MS-EI: m/z(%): 283.00 (M+H^{+}, 100%), 304.90 (M+Na^{+}, 29%).

References

1. C. Wang, L. Zhang, A. Ren, P. Lu, Y. Wang, *Org.Lett*., 2013, **15**, 2982-2985.
2. J.-M. Hwang, T. Oh, T. Kaneko, A. M. Upton, S. G. Franzblau, Z. Ma, S.-N. Cho, P. Kim, *J. Nat. Prod*., 2013, **76,** 354-367.
3. S. Guo, J. Zhai, X. Fan, *Org. Biomol. Chem*., 2017, **15**, 1521-1529.
4. A. Kumar, V. D. Tripathi, P. Kumar, *Green Chem*., 2011, **13**, 51-54.

5. ^{1}H NMR and ^{13}C NMR spectra of products






8.59
8.57
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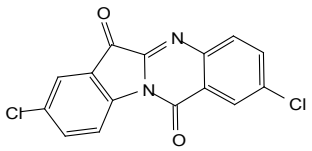
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Cl
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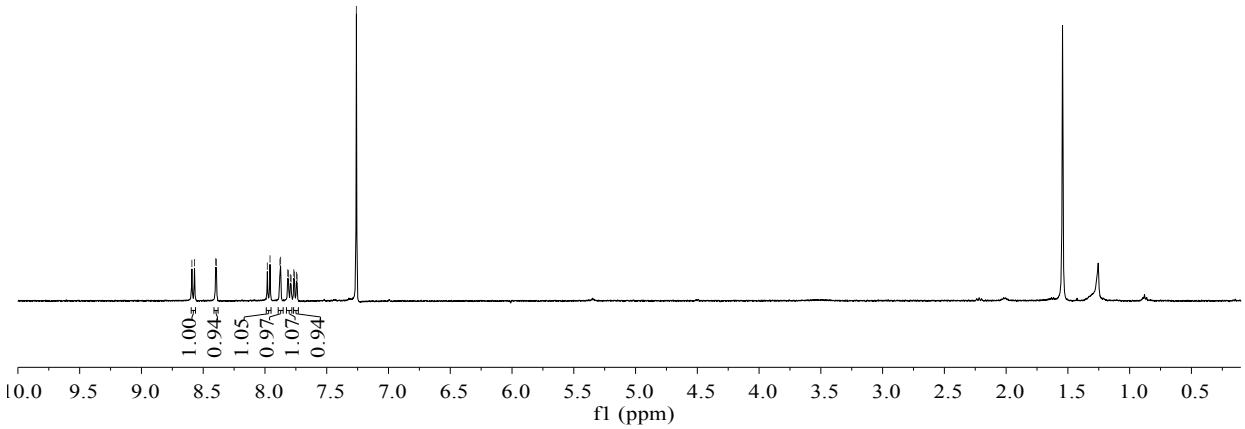
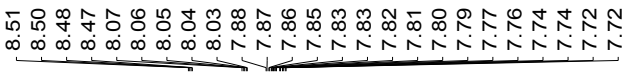

1.00
0.94
1.05
0.97
1.07
0.94
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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8.03
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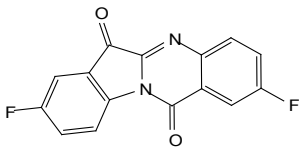
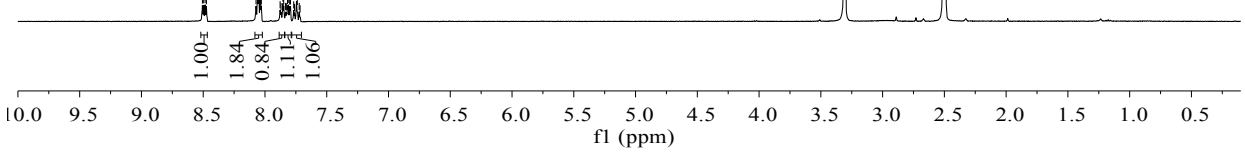
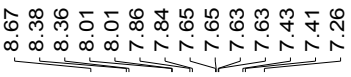
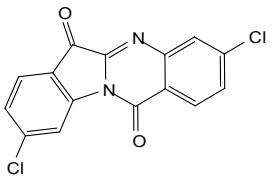
7.83
7.82
7.81
7.80
7.79
7.77
7.76
7.74
7.74
7.72
7.72

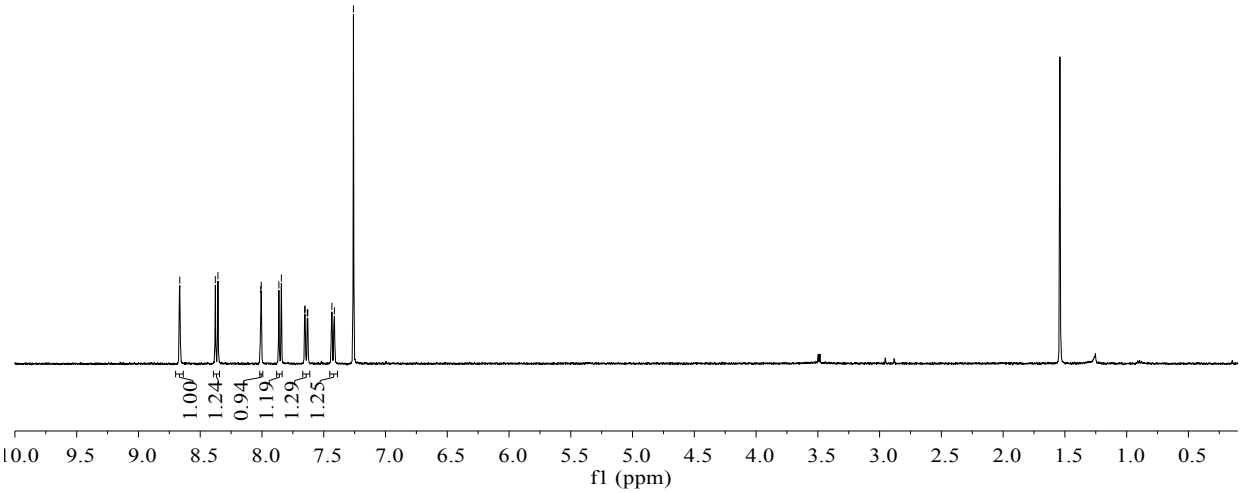

3.31
2.50

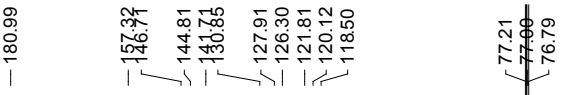

O
N
N
F
F
O

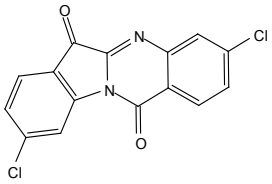

1.00
1.84
0.84
1.11
1.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

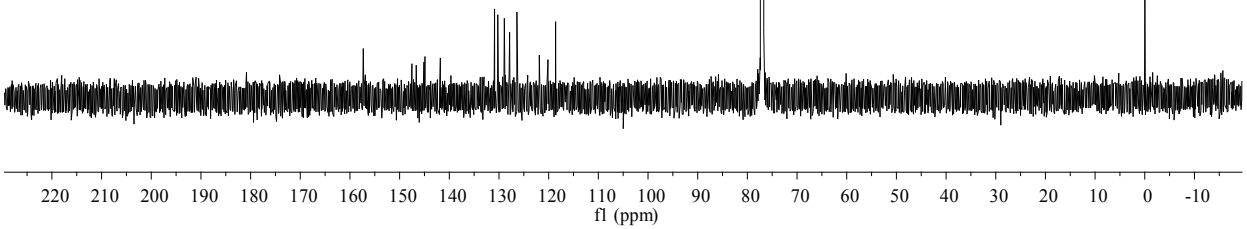

8.67
8.38
8.36
8.01
8.01
7.86
7.84
7.65
7.65
7.63
7.63
7.43
7.41
7.26

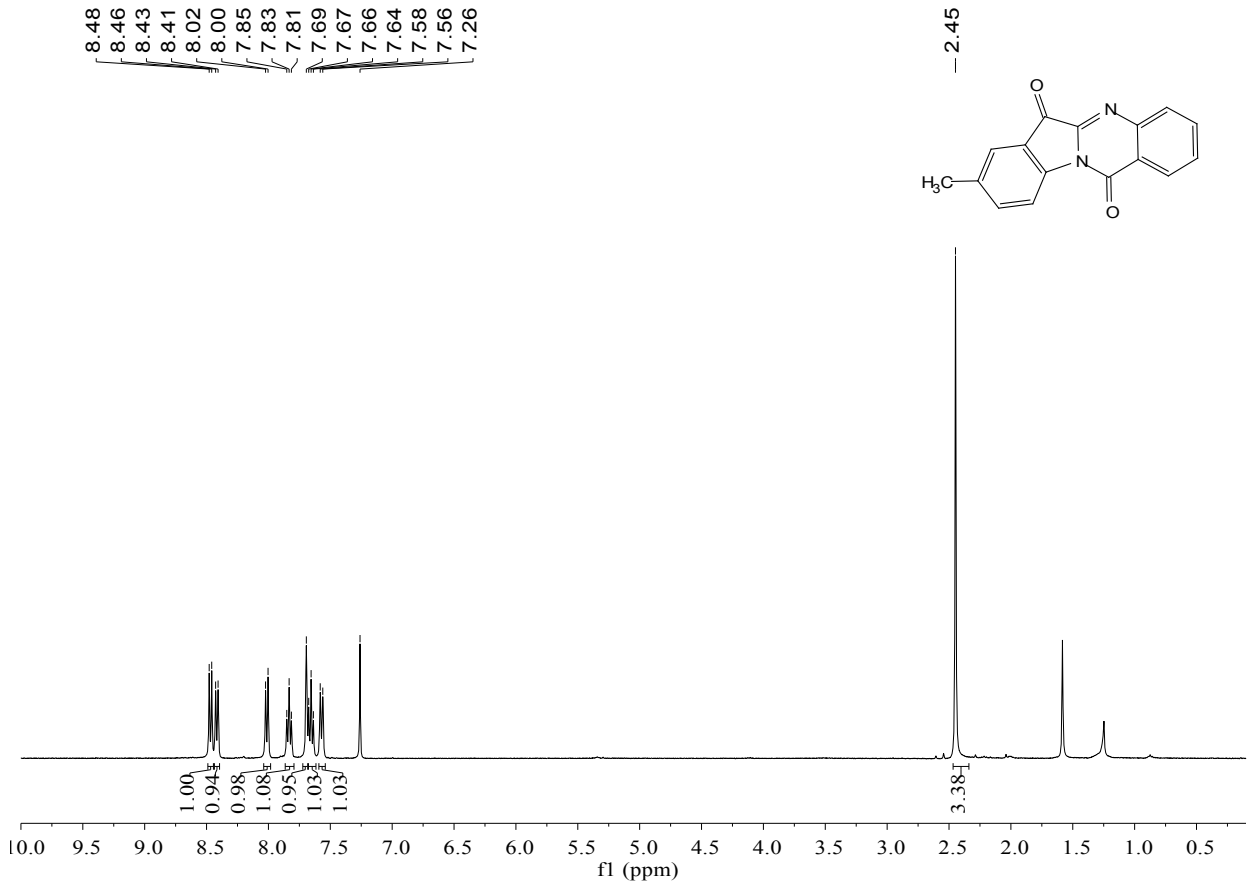

O
N
Cl
N
O
Cl

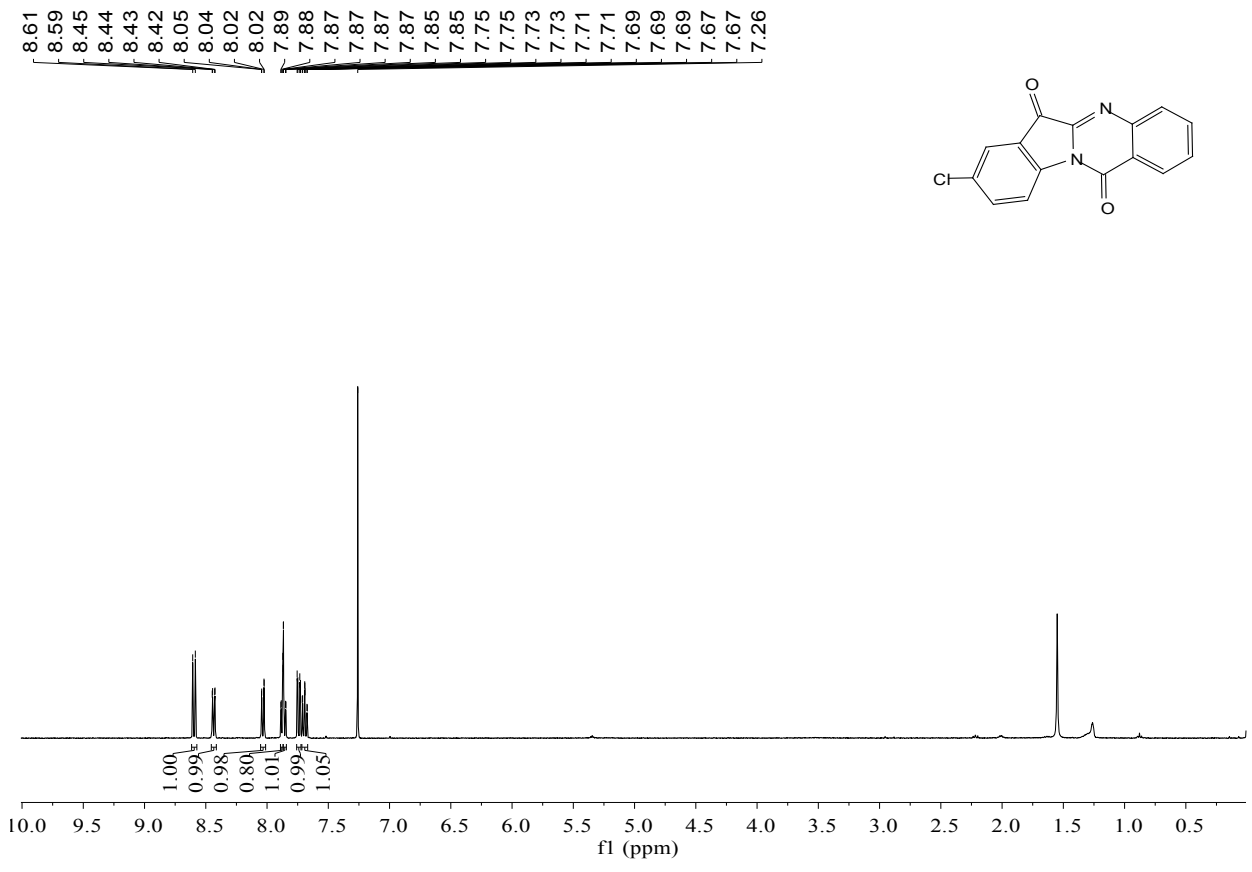

1.00
1.24
0.94
1.19
1.29
1.25
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

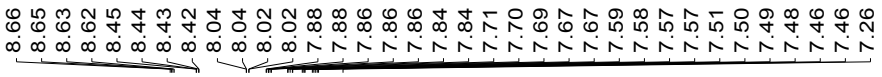

180.99
157.32
146.71
144.81
141.71
130.85
127.91
126.30
121.81
120.12
118.50
77.21
77.00
76.79

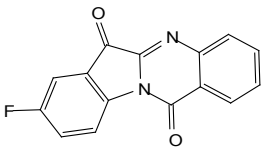

O
N
Cl
N
O
Cl

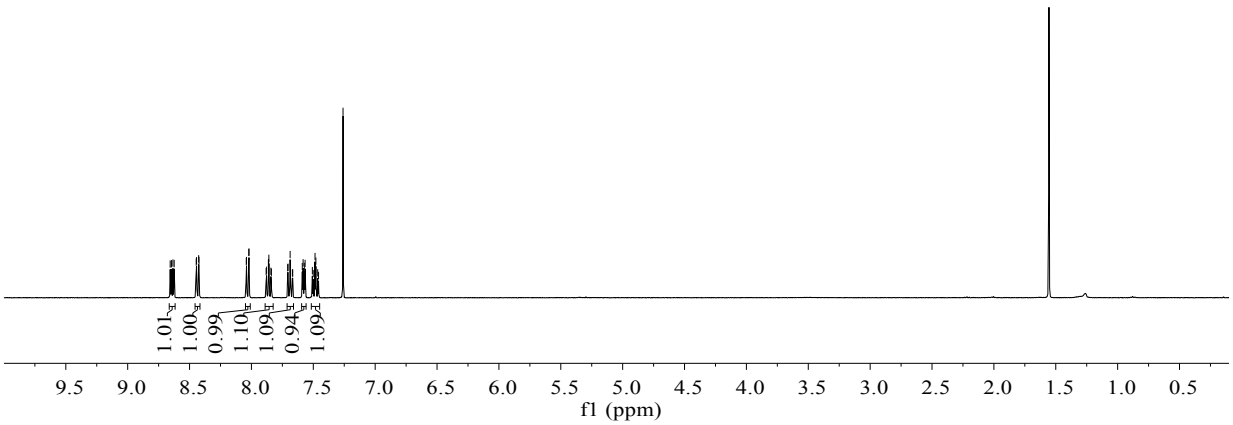

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.48
8.46
8.43
8.41
8.02
8.00
7.85
7.83
7.81
7.69
7.67
7.66
7.64
7.58
7.56
7.26
2.45
O
N
N
O
H3C
1.00
0.94
0.98
1.08
0.95
1.03
1.03
3.38
10.0 9.5 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
f1 (ppm)

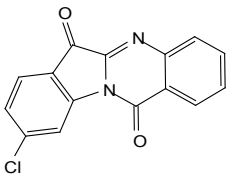

8.61
8.59
8.45
8.44
8.43
8.42
8.05
8.04
8.02
8.02
7.89
7.88
7.87
7.87
7.87
7.87
7.85
7.85
7.75
7.75
7.73
7.73
7.71
7.71
7.69
7.69
7.69
7.67
7.67
7.26
O
N
N
O
Cl
1.00
0.99
0.98
0.80
1.01
0.99
1.05
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

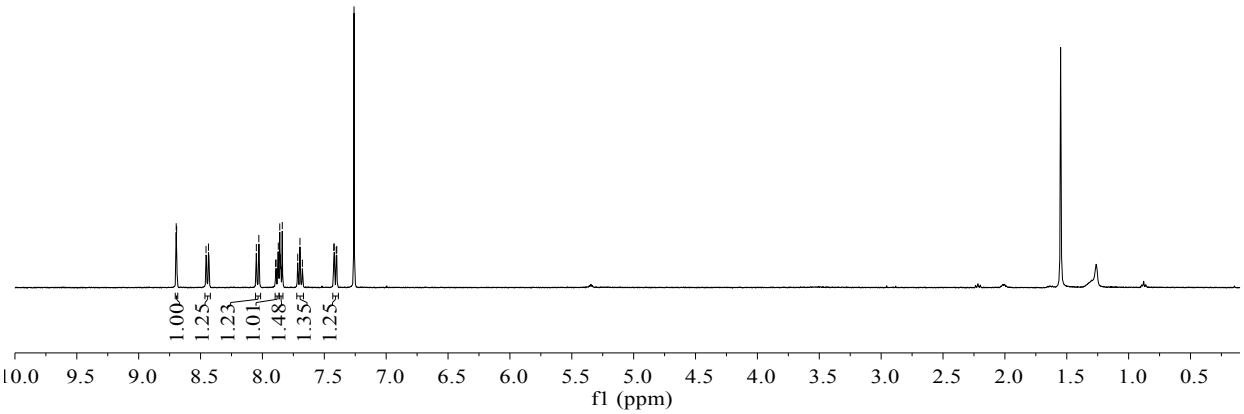

8.66
8.65
8.63
8.62
8.45
8.44
8.43
8.42
8.04
8.04
8.02
8.02
7.88
7.88
7.86
7.86
7.86
7.84
7.84
7.71
7.70
7.69
7.67
7.67
7.59
7.58
7.57
7.57
7.51
7.50
7.49
7.48
7.46
7.46
7.26

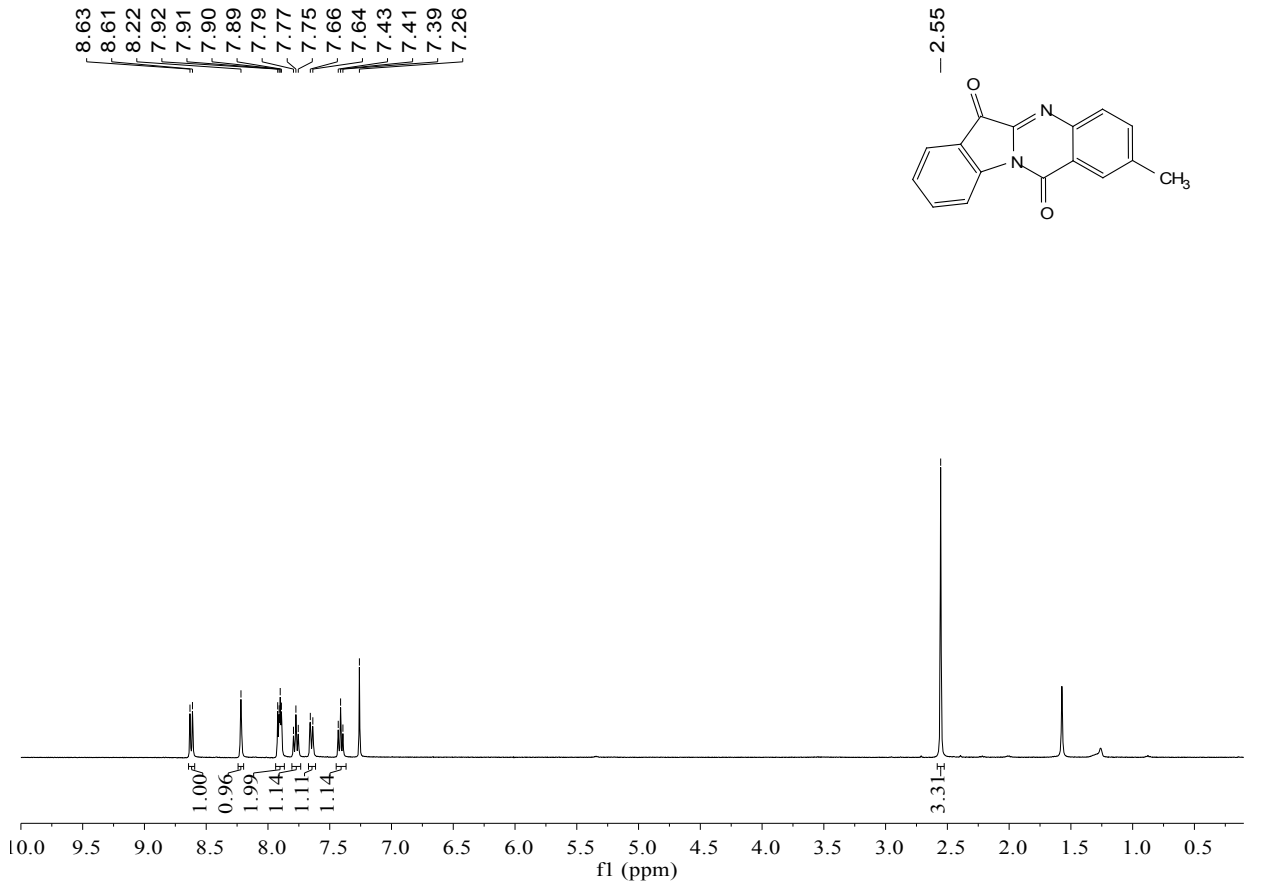

O
N
N
F
O

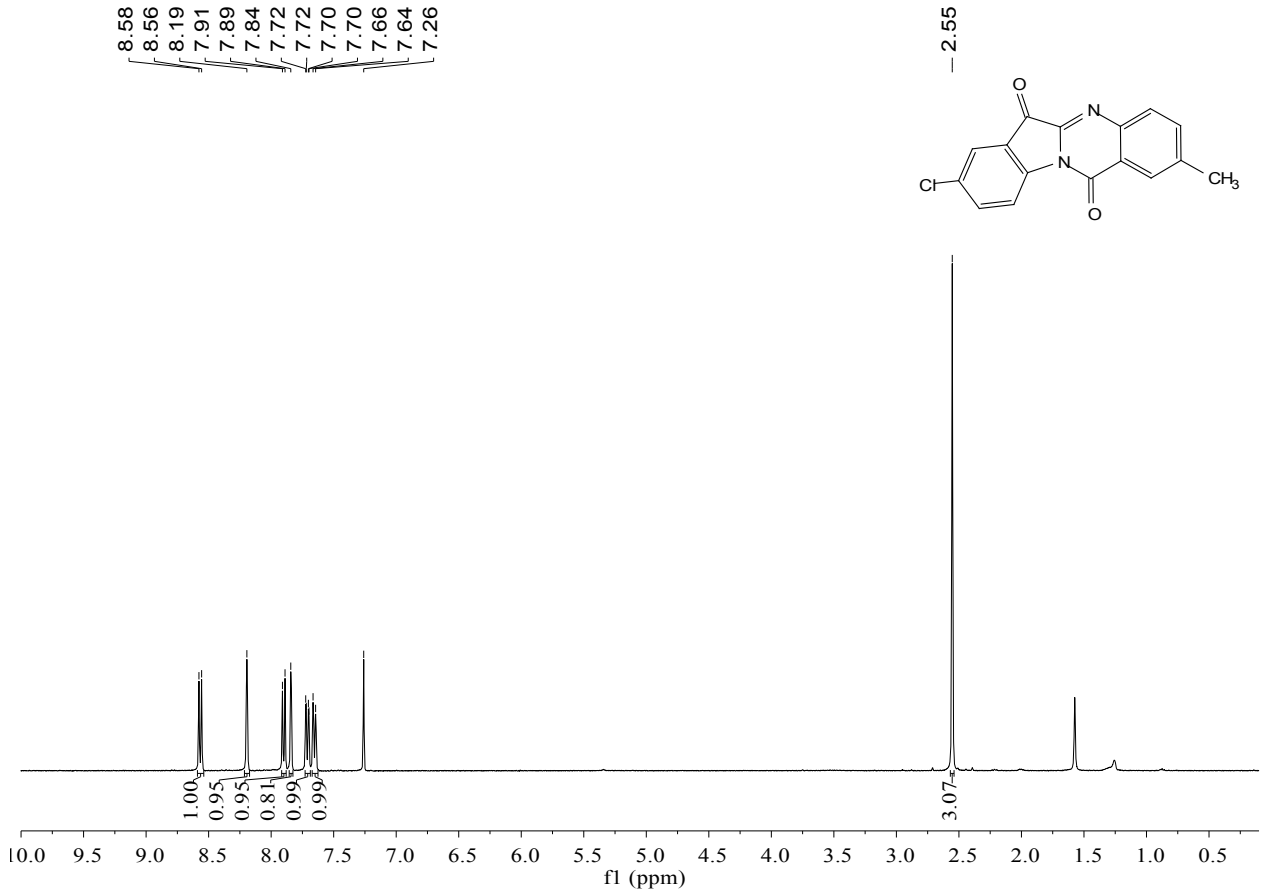

1.01
1.00
0.99
1.10
1.09
0.94
1.09
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

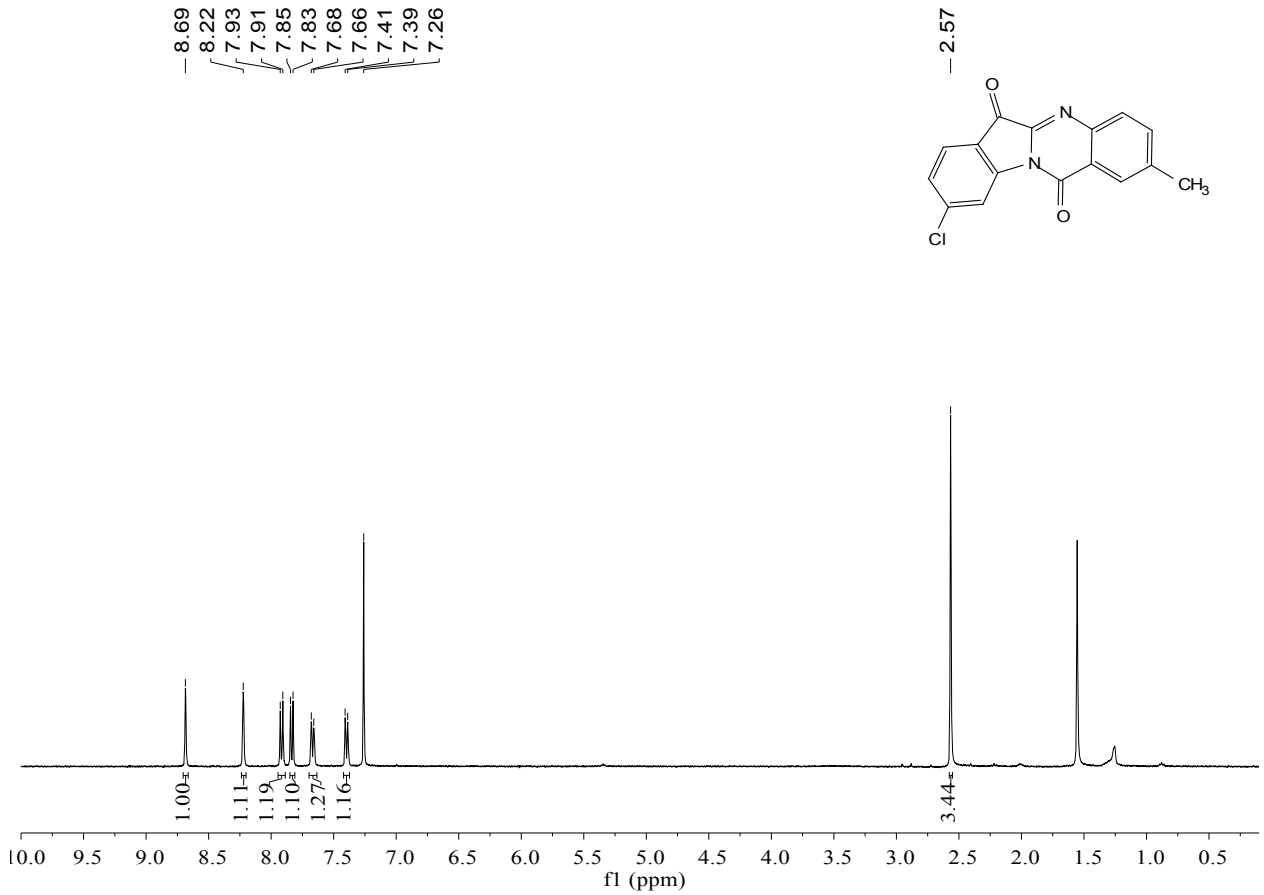

8.70
8.69
8.46
8.44
8.05
8.03
7.89
7.89
7.87
7.86
7.86
7.84
7.71
7.70
7.68
7.42
7.42
7.40
7.40
7.26

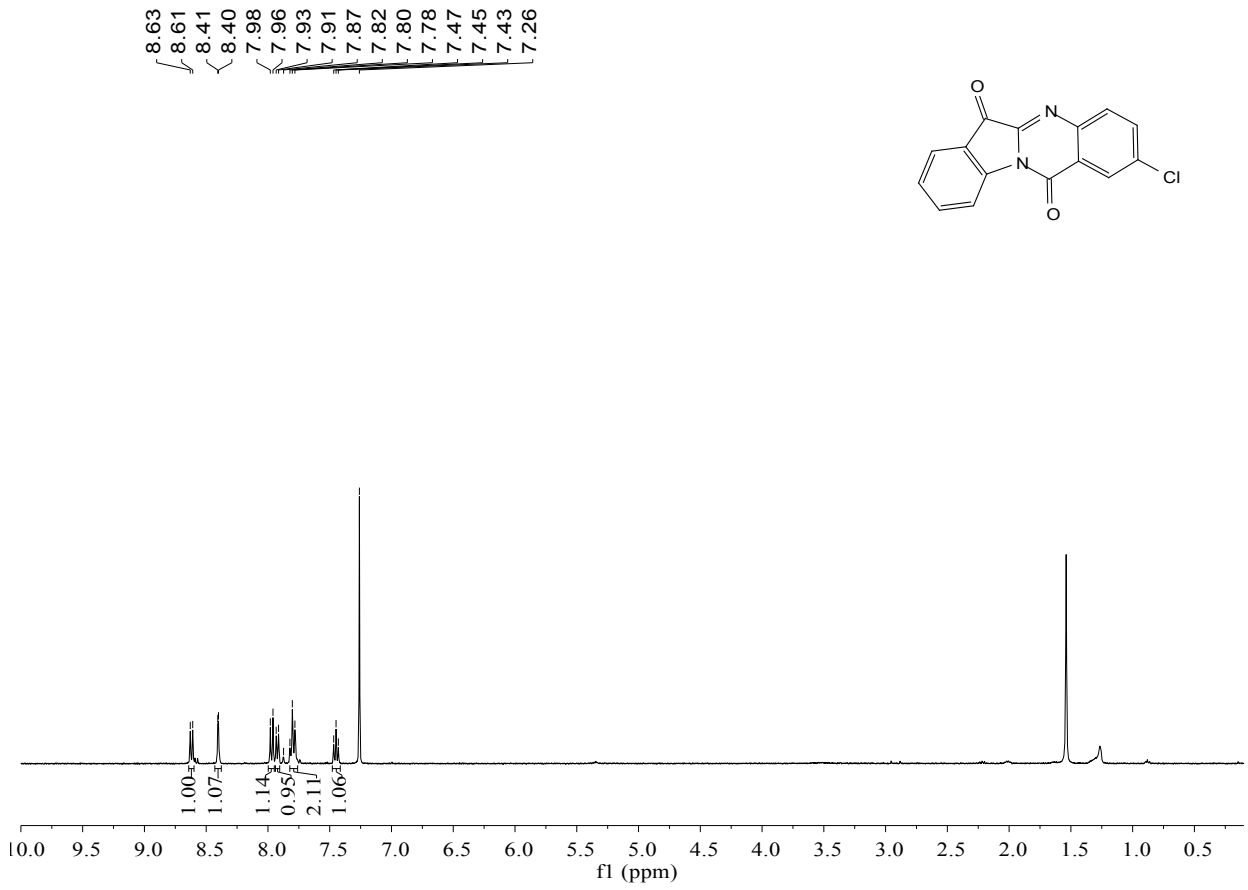

O
N
N
O
Cl

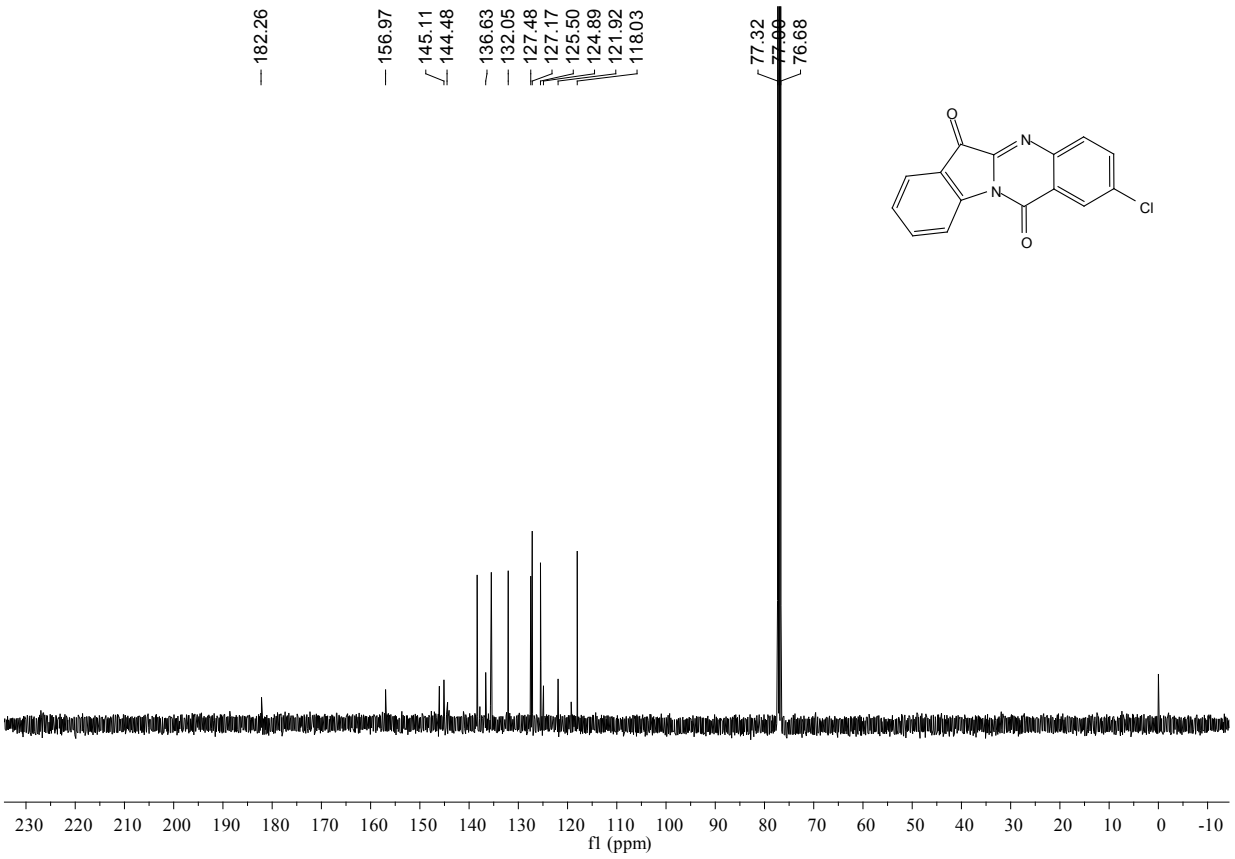

1.00
1.25
1.23
1.01
1.48
1.35
1.25
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

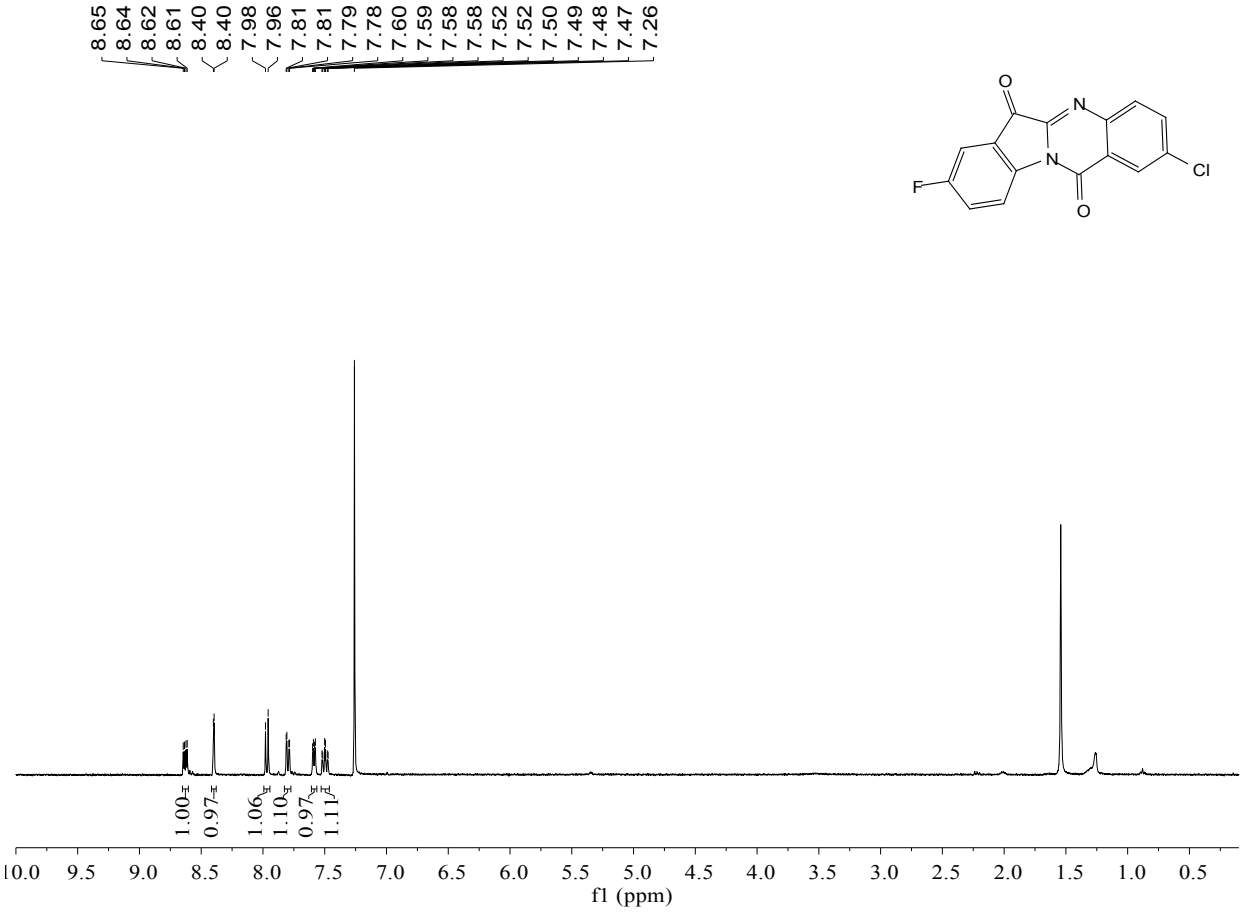

8.63
8.61
8.22
7.92
7.91
7.90
7.89
7.79
7.77
7.75
7.66
7.64
7.43
7.41
7.39
7.26
2.55
O
N
N
O
CH3
1.00
0.96
1.99
1.14
1.11
1.14
3.31
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

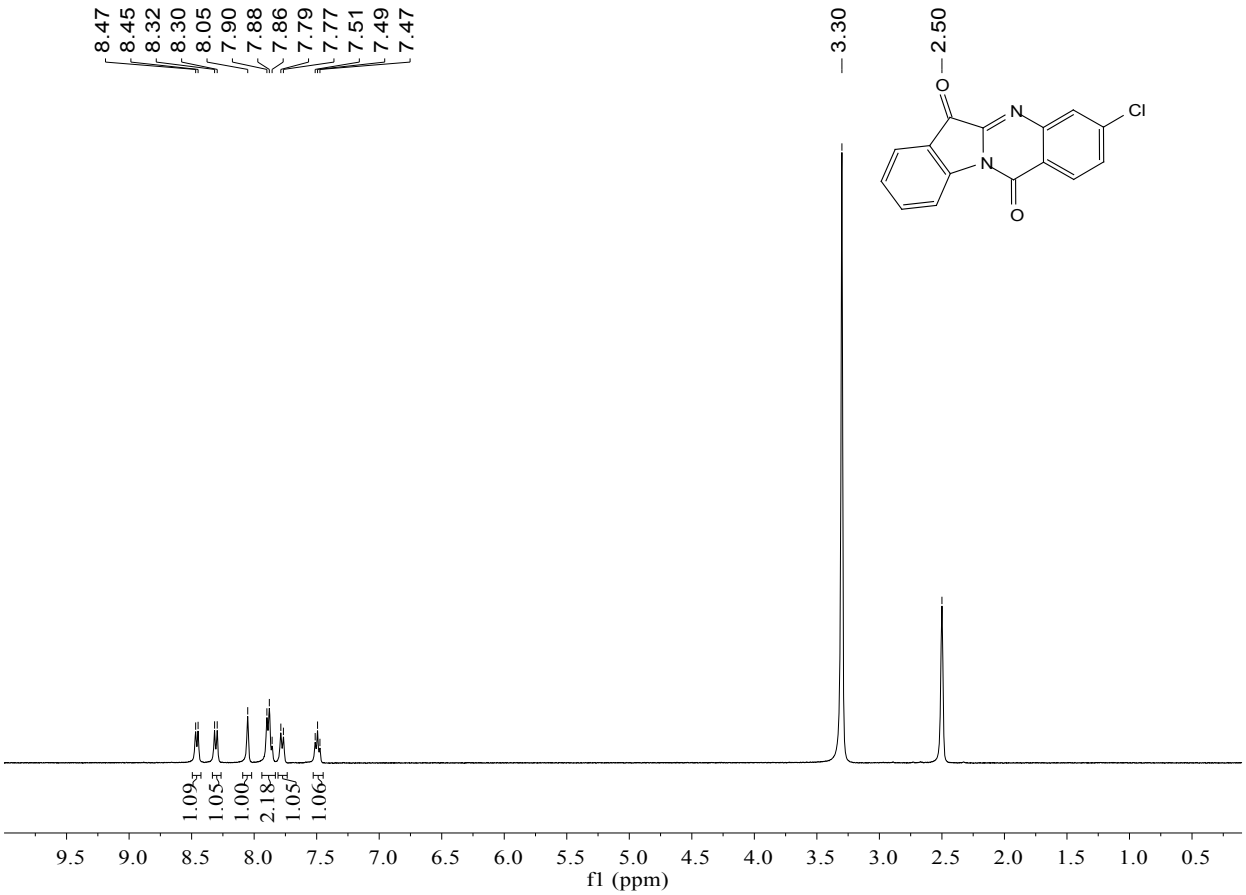

8.58
8.56
8.19
7.91
7.89
7.84
7.72
7.72
7.70
7.70
7.66
7.64
7.26
2.55
O
N
N
O
Cl
CH3
1.00
0.95
0.95
0.81
0.99
0.99
3.07
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

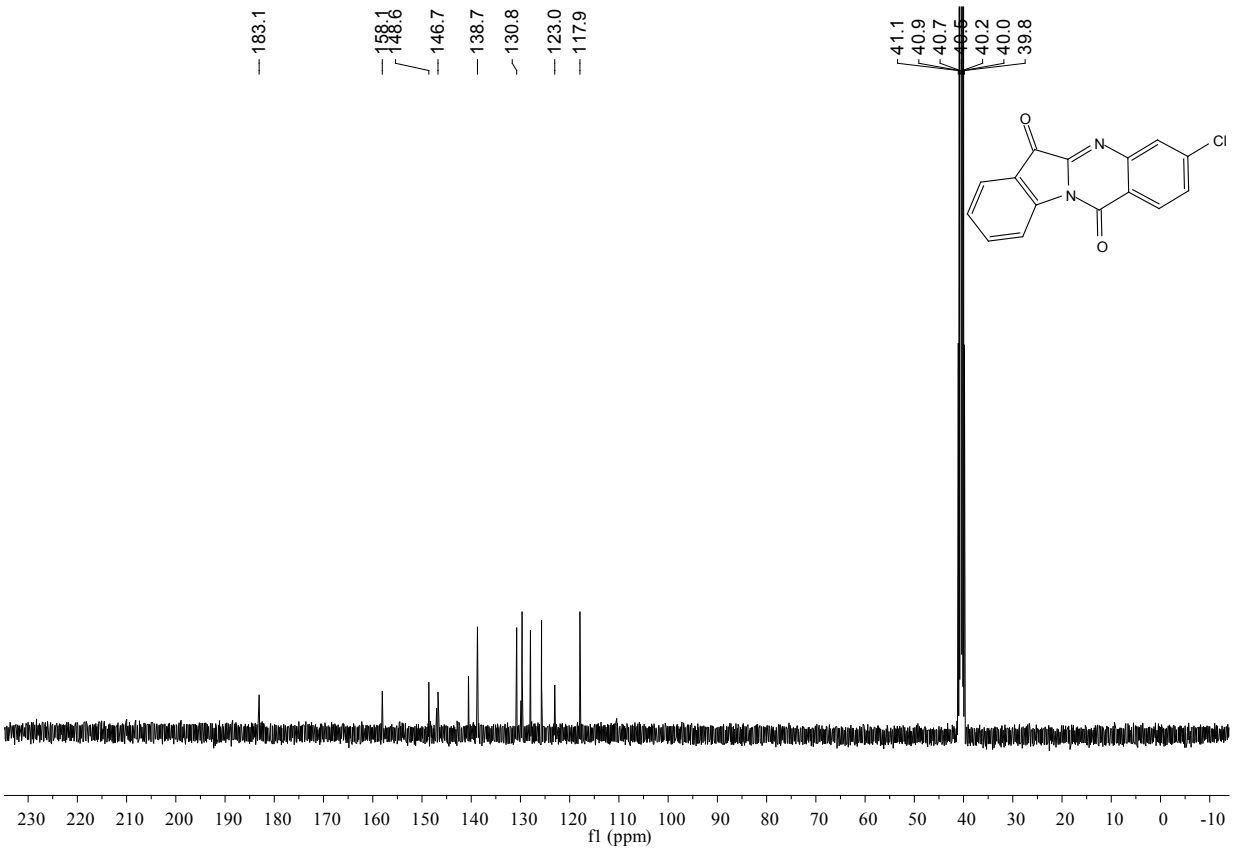

8.69
8.22
7.93
7.91
7.85
7.83
7.68
7.66
7.41
7.39
7.26
2.57
O
N
N
O
CH3
Cl
1.00
1.11
1.19
1.10
1.27
1.16
3.44
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


8.63
8.61
8.41
8.40
7.98
7.96
7.93
7.91
7.87
7.82
7.80
7.78
7.47
7.45
7.43
7.26
O
N
N
O
Cl
1.00
1.07
1.14
0.95
2.11
1.06
10.0 9.5 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
f1 (ppm)


182.26
156.97
145.11
144.48
136.63
132.05
127.48
127.17
125.50
124.89
121.92
118.03
77.32
77.00
76.68
O
N
N
Cl
O
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.65
8.64
8.62
8.61
8.40
8.40
7.98
7.96
7.81
7.81
7.79
7.78
7.60
7.59
7.58
7.58
7.52
7.52
7.50
7.49
7.48
7.47
7.26
O
N
N
Cl
F
O
1.00
0.97
1.06
1.10
0.97
1.11
10.0 9.5 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
f1 (ppm)


8.47
8.45
8.32
8.30
8.05
7.90
7.88
7.86
7.79
7.77
7.51
7.49
7.47
3.30
2.50
O
N
Cl
N
O
1.09
1.05
1.00
2.18
1.05
1.06
9.5 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
f1 (ppm)


183.1
158.1
148.6
146.7
138.7
130.8
123.0
117.9
41.1
40.9
40.7
40.5
40.2
40.0
39.8
O
N
Cl
N
O
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)