

β -Cyclodextrin catalysed synthesis of tryptanthrin in water

Atul Kumar,* Vishwa Deepak Tripathi, and Promod Kumar

Medicinal and Process Chemistry Division,
Central Drug Research Institute, CSIR, Lucknow India

Supplementary Data

Contents

1. General Consideration.....	1
2. General Procedure for synthesis of compounds 3(a-x).....	1
3. Catalyst recovery procedure.....	1
4. Analytical data.....	1
5. ¹ H and ¹³ C NMR spectra.....	6

General consideration: All the reactions were carried out at room temperature that is 28-32^oC. Unless otherwise specified, all the reagents were purchased from Sigma-Aldrich Chemical Co, Lancaster and were used directly without further any purification. NMR spectra were obtained using the Bruker DRX 300MHz spectrometer. Chemical shifts (δ) are given in ppm relative to TMS, coupling constants (J) in Hz. IR spectra were taken on VARIAN FT-IR spectrometer as KBr pellets (when solid). Elemental analysis was performed using a Perkin Elmer Autosystem XL Analyzer. Melting points were measured using a COMPLAB melting- point apparatus. Reactions were monitored by thin-layer chromatography (TLC) carried out on 0.25 mm silica gel plates visualized with UV light.

General procedure for synthesis of compounds 3(a-x) : Isatin (1.0 mmol.) and isatoic anhydride (1.0 mmol.) were combined with 10ml distilled water in a 50ml round bottom flasks equipped with a stir bar. β -cyclodextrin (20 mol%) was added. Reaction was allowed to stir at room temperature for the appropriate amount of time. After completion of reaction reaction mixture was extracted with ethyl acetate. Organic layer was dried over Na₂SO₄ and concentrated in vacuo to give crude product which was further purified by column chromatography.

Catalyst recovery procedure: After completion of reaction, reaction mixture was extracted with ethyl acetate. Aqueous layer was left overnight at temperature of 5^oC. Due to its low solubility β -CD precipitated at lower temperature. Cyclodextrin precipitated filtered off, dried and reused for next batch as such.

Analytical Data:

Indolo[2,1-*b*]quinazoline-6,12-dione¹ (3a)

yellow solid; mp >250^oC; Found C, 72.52, H, 3.19, N, 11.22 C₁₅H₈N₂O₂ requires C, 72.58, H, 3.25, N, 11.28 %; ν_{\max} (KBr): 3062, 1721, 1645, 1435, 1256, 804, 747 cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.61 (d, 1H, J = 3.4 Hz), 8.44 (d, 1H, J = 1.17 Hz), 8.06 (d, 1H, J = 8.01 Hz), 7.94-7.80 (m, 3H), 7.69 (t, 1H, J = 7.08 Hz), 7.28 (t, 1H, J = 7.44 Hz); ¹³C NMR (75 MHz CDCl₃) δ =117.5, 120.6, 125.4, 126.3, 127.1, 129.7, 130.0, 133.2, 134.6, 145.3, 146.6, 160.4, 183.8; ESIMS: m/z 249 (M+H).

2-Bromoindolo[2,1-*b*]quinazoline-6,12-dione² (3b)

Light yellow solid; mp >250^oC Found C, 54.98, H, 2.13 N, 8.48 C₁₅H₇BrN₂O₂ requires C, 55.07; H, 2.16; N, 8.56%; ν_{\max} (KBr):3124, 1724, 1647, 1440, 1255, 812, 746 cm⁻¹. ¹H NMR (300 MHz CDCl₃) δ = 8.24 (s, 1H), 8.10-8.02 (m, 2H), 7.98-7.82 (m, 2H), 7.63 (t, 1H, J = 7.2 Hz), 7.38 (t, 1H, J = 6.4 Hz); ¹³C NMR (75 MHz CDCl₃) δ = 117.8, 120.7, 123.0, 123.5, 125.7, 129.4, 129.2, 129.9, 132.3, 134.8, 136.2, 146.4, 160.8, 182.5; ESIMS: m/z 249 (M+H).

2-Nitroindolo[2,1-*b*]quinazoline-6,12-dione² (3c)

Brown solid; mp >250^oC Found C, 61.25, H, 2.26 N, 14.46 C₁₅H₇N₃O₄, requires C, 61.44; H, 2.41; N, 14.33%; ν_{\max} (KBr):3062, 1720, 1650, 1532, 1352, 1254, 747, 687cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.51 (s, 1H),

8.34 (d, 1H, $J= 5.14$ Hz), 8.14-7.98 (m, 2H), 7.94-7.77 (m, 2H), 7.36 (t, 1H, $J= 6.8$ Hz), ^{13}C NMR (75 MHz CDCl_3) $\delta= 116.5, 122.4, 123.2, 123.8, 127.2, 128.8, 129.4, 131.4, 134.2, 137.2, 139.4, 161.9, 181.5$; ESIMS: m/z 294 (M+H)

8-Methoxy-2-nitroindolo[2,1-*b*]quinazoline-6,12-dione² (3d)

Yellow solid; mp $>250^\circ\text{C}$; Found C, 59.27; H, 2.63; N, 13.16 $\text{C}_{16}\text{H}_9\text{N}_3\text{O}_5$, requires C, 59.45; H, 2.81; N, 13.00; $\nu_{\text{max}}(\text{KBr})$: 3108, 2971, 1724, 1675, 1455, 1212, cm^{-1} ; ^1H NMR (300 MHz CDCl_3) $\delta= 8.32$ (d, 1H, $J= 7.14$ Hz), 8.22 (s, 1H); 8.04-7.91 (m, 2H) 7.84 (d, 1H, $J= 6.4$ Hz), 7.67 (d, 1H, $J= 8.7$ Hz), 3.91 (s, 3H); ^{13}C NMR (75 MHz CDCl_3) $\delta= 50.3, 115.9, 117.4, 123.6, 125.2, 128.1, 128.8, 131.2, 132.8, 133.6, 140.7, 145.8, 153.2, 163.4, 193.6$; ESIMS: m/z 324 (M+H).

2-Aminoindolo[2,1-*b*]quinazoline-6,12-dione² (3e)

Brown solid; mp $>250^\circ\text{C}$ Found C, 68.40; H, 3.41; N, 15.85 $\text{C}_{15}\text{H}_9\text{N}_3\text{O}_2$ requires C, 68.44; H, 3.45; N, 15.96%. $\nu_{\text{max}}(\text{KBr})$: 3354, 3045, 1722, 1652, 1448, 1265, 804, cm^{-1} ; ^1H NMR (300 MHz CDCl_3) $\delta= 8.32$ (d, 1H, $J= 6.44$ Hz), 8.21-8.14 (m, 2H), 7.90 (s, 1H), 7.38 (t, 1H, $J= 7.4$ Hz), 7.24-7.16 (m, 2H), 6.14 (brs, 2H); ^{13}C NMR (75 MHz CDCl_3) $\delta= 113.2, 118.4, 119.7, 121.6, 123.5, 125.7, 129.8, 130.2, 133.7, 134.5, 145.2, 151.6, 160.6, 182.8$; ESIMS: m/z 264 (M+H).

8-Bromoindolo[2,1-*b*]quinazoline-6,12-dione² (3f)

Light brown solid; mp $>250^\circ\text{C}$ Found C, 54.98; H, 2.05; N, 8.42 $\text{C}_{15}\text{H}_7\text{BrN}_2\text{O}_2$ requires C, 55.07; H, 2.16; N, 8.56%. $\nu_{\text{max}}(\text{KBr})$: 3068, 1724, 1656, 1446, 1265, cm^{-1} ; ^1H NMR (300 MHz CDCl_3) $\delta= 8.59$ -8.48 (m, 2H), 7.98 (d, 1H, $J= 7.12$ Hz), 7.84 (d, 1H, $J= 6.4$ Hz), 7.81-7.74 (m, 2H), 7.64-7.58 (m, 1H); ^{13}C NMR (75 MHz CDCl_3) $\delta= 118.3, 120.8, 122.5, 125.6, 126.9, 129.3, 132.0, 133.9, 134.4, 141.3, 143.5, 148.6, 153.7, 160.6, 189.4$; ESIMS: m/z 324 (M+H).

8-Methoxyindolo[2,1-*b*]quinazoline-6,12-dione (3g)

Yellow solid; mp $>250^\circ\text{C}$; Found C, 68.88; H, 3.43; N, 10.20 $\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_3$ requires C, 69.06; H, 3.62; N, 10.07%; ^1H NMR (300 MHz CDCl_3) $\delta= 8.03$ (1 H, d, $J= 6.24$ Hz), 7.70-7.63 (2 H, m) 7.62 (d, 1H, $J= 5.4$ Hz), 7.55 (s, 1H), 7.50-7.44 (m, 2H), 3.84 (s, 3H); ^{13}C NMR (75 MHz CDCl_3) $\delta= 54.8, 104.6, 111.4, 118.8, 126.1, 126.6, 127.1, 127.8, 132.4, 146.3, 147.5, 153.8, 160.6, 184.2$; $\nu_{\text{max}}(\text{KBr})$: 3058, 2972, 1719, 1645, 1465, 1252, cm^{-1} . ESIMS: m/z 324 (M+H)..

8-Methylindolo[2,1-*b*]quinazoline-6,12-dione² (3h)

Yellow solid; mp $>250^\circ\text{C}$; Found C, 73.20; H, 3.76; N, 10.54 $\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_2$ requires C, 73.27; H, 3.84; N, 10.68%; $\nu_{\text{max}}(\text{KBr})$: 3075, 2968, 1722, 1653, 1448, 1262, 687 cm^{-1} ; ^1H NMR (300 MHz CDCl_3) $\delta= 8.03$ -8.7.94 (m, 2H), 7.68 (s, 1H), 7.57 (d, 1H, $J= 6.21$ Hz), 7.42 (d, 1H, $J= 7.6$ Hz), 7.28-7.14 (m, 2H), 2.31 (s, 3H); ^{13}C NMR (75 MHz CDCl_3) $\delta= 21.3, 113.7, 120.8, 124.1, 125.2, 126.8, 127.4, 144.4, 144.8, 145.2, 150.7, 161.2, 183.7$; ESIMS: m/z 263 (M+H).

8-Chloroindolo[2,1-*b*]quinazoline-6,12-dione² (3i)

Yellow solid; mp >250⁰C; Found C, 63.65; H, 2.42; N, 9.86 C₁₅H₇ClN₂O₂ requires C, 63.73; H, 2.50; N, 9.91%
v_{max}(KBr):3078, 2974, 1720, 1658, 1456, 1250, cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ= 8.62 (d, 1H, *J*= 6.8 Hz), 8.44 (d, 1H, *J*= 5.8 Hz), 8.06 (d, 1H, *J*= 7.15 Hz), 7.90-7.88 (m, 2H), 7.76-7.68 (m, 2H); ¹³C NMR (75 MHz CDCl₃) δ= 116.5, 121.8, 123.5, 127.6, 127.9, 128.3, 130.0, 133.9, 134.4, 141.3, 146.5, 148.6, 152.7, 161.6, 188.4; ESIMS: m/z 263 (M+H).

8-Nitroindolo[2,1-*b*]quinazoline-6,12-dione² (3j)

Pale yellow solid; mp >250⁰C; Found C, 61.36; H, 2.32; N, 14.26 C₁₅H₇N₃O₄ requires C, 61.44; H, 2.41; N, 14.33%; v_{max}(KBr):3064, 1720, 1665, 1453, 1255, 804, 746 cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ= 8.74-8.72 (m, 2H), 8.54(s, 1H), 8.39 (d, 1H, *J*= 6.0), 7.79 (d, 2H, *J*= 3.14 Hz), 7.77-7.74 (m, 1H); ¹³C NMR (75 MHz CDCl₃) δ= 115.0, 120.0, 120.8, 123.8, 126.6, 127.3, 133.4, 145.5, 147.1, 153.9, 160.6, 186.4; ESIMS: m/z 293 (M+H).

8-Fluoroindolo[2,1-*b*]quinazoline-6,12-dione (3k)

Creamish white solid; mp >250⁰C; Found C, 67.62; H, 2.58; N, 10.46 C₁₅H₇FN₂O₂, requires C, 67.67; H, 2.65; N, 10.52; v_{max}(KBr):3075, 1719, 1656, 1459, 1253, 804, cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ= 8.48-8.21(m, 2H), 7.92-7.89(m, 1H), 7.86 (t, 1H, *J*= 6.4 Hz), 7.69-7.58(m, 2H), 7.43 (t, 1H, *J*= 5.3 Hz), 8.03 (d, 1H, *J*= 8.15 Hz), 7.81-7.94 (m, 2H), 7.79-7.71 (m, 2H); ¹³C NMR (75 MHz CDCl₃) δ= 108.6, 111.3, 118.8, 127.3, 128.6, 128.7, 129.3, 135.4, 145.8, 146.5, 162.2, 183.4; ESIMS: m/z 266 (M+H).

8-Iodoindolo[2,1-*b*]quinazoline-6,12-dione (3l)

Creamish solid; mp >250⁰C Found C, 48.07; H, 1.68; N, 7.36 C₁₅H₇IN₂O₂ requires C, 48.15; H, 1.89; N, 7.49%; v_{max}(KBr): 3068, 1724, 1656, 1446, 1265, cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ= 8.67-8.54 (m, 2H), 8.05 (d, 1H, *J*= 5.27 Hz), 7.95 (d, 1H, *J*= 6.2 Hz), 7.81-7.76 (2H, m), 7.70-7.63(m, 1H); ¹³C NMR (75 MHz CDCl₃) δ= 112.0, 124.6, 125.4, 126.4, 127.9, 128.3, 131.2, 134.4, 134.8, 147.8, 149.6, 155.8, 161.3, 183.5; ESIMS: m/z 374 (M+H).

8-Bromo-2,3-dimethoxyindolo[2,1-*b*]quinazoline-6,12-dione (3m)

Yellow solid; mp >250⁰C; Found C, 52.48; H, 2.64; N, 7.31 C₁₇H₁₁BrN₂O₄ requires C, 52.74; H, 2.86; N, 7.24%; v_{max}(KBr): 3074, 2875, 1728, 1646, 1446, 1245 cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ= 8.54 (s, 1H), 8.41 (s, 1H), 8.20(d, 1 H, *J*= 6.4 Hz); 7.89-7.68(m, 2H); 3.98(s, 6H); ¹³C NMR (75 MHz CDCl₃) δ= 48.04, 108.2, 110.5, 116.6, 118.4, 121.32, 123.4, 135.7, 137.2, 139.8, 143.0, 146.6, 162.9, 163.9, 188.3; ESIMS: m/z 388 (M+H).

8-Bromo-2-nitroindolo[2,1-*b*]quinazoline-6,12-dione (3n)

Pale yellow solid; mp >250⁰C ; Found C, 48.20; H, 1.44; N, 11.41 C₁₅H₆BrN₃O₄ requires C, 48.41; H, 1.63; N, 11.29%; v_{max}(KBr): 3064, 1720, 1665, 1453, 1255, 804, 746 cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ= 8.38 (d, 1H, *J*= 8.1 Hz), 8.21(s, 1H), 8.09 (s, 1H), 8.03-7.88 (m, 2H), 7.79 (d, 1H, *J*= 7.14 Hz); ¹³C NMR (75 MHz CDCl₃) δ=

119.4, 121.2, 121.9, 124.8, 126.5, 128.3, 133.3, 138.5, 142.0, 145.7, 153.2, 161.1, 164.8, 194.02; ESIMS: m/z 373 (M+H).

2-Amino-8-bromoindolo[2,1-*b*]quinazoline-6,12-dione (3o)

Yellow solid; mp >250°C; Found C, 52.34; H, 2.15; N, 12.38 C₁₅H₈BrN₃O₂ requires C, 52.66; H, 2.36; N, 12.28; ν_{\max} (KBr): 3078, 2984, 1724, 1656, 1444, 1250, cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.58 (d, 1H, *J*= 6.9 Hz), 8.42 (d, 1H, *J*= 5.8 Hz), 8.02 (s, 1H); 7.98 (d, 1H, *J*= 7.15 Hz); 7.89-7.77 (m, 2H); 5.51 (brs, 2H); ¹³C NMR (75 MHz CDCl₃) δ = 118.9, 119.2, 120.4, 121.2, 121.8, 122.6, 124.9, 137.4, 139.2, 140.5, 149.6, 164.3, 168.0, 189.1; ESIMS: m/z 343 (M+H).

2-Bromo-8-nitroindolo[2,1-*b*]quinazoline-6,12-dione (3p)

Yellow solid; mp >250°C ; Found C, 48.28; H, 1.51; N, 11.34 C₁₅H₆BrN₃O₄ requires C, 48.41; H, 1.63; N, 11.29%. ν_{\max} (KBr): 3082, 2862, 1724, 1658, 1442, 1248, cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.46 (d, 1H, *J*= 7.4 Hz), 8.31(s, 1H); 8.13-7.92(m, 2H), 7.82(d, 1H, *J*= 6.13 Hz), 7.68 (d, 1H, *J*= 7.16 Hz); ¹³C NMR (75 MHz CDCl₃) δ = 118.6, 121.4, 122.3, 122.9, 124.03, 128.1, 134.9, 138.5, 143.6, 145.8, 152.2, 160.3, 165.9, 191.4; ESIMS: m/z 373 (M+H),

2,3-Dimethoxy-8-nitroindolo[2,1-*b*]quinazoline-6,12-dione (3q)

Yellow solid; mp >250°C; Found C, 57.58; H, 2.98; N, 11.96 C₁₇H₁₁N₃O₆ requires C, 57.80; H, 3.14; N, 11.89%; ν_{\max} (KBr): 3064, 2878, 1721, 1686, 1456, 1205 cm⁻¹. ¹H NMR (300 MHz CDCl₃) δ = 8.25 (s, 1H), 8.14 (s, 1H), 8.04(d, 1H, *J*= 6.4 Hz); 7.88-7.80 (m, 2H); 3.94 (s, 6H); ¹³C NMR (75 MHz CDCl₃) δ = 51.04, 106.2, 112.5, 114.06, 117.4, 120.32, 124.4, 132.7, 134.2, 138.8, 140.0, 143.6, 160.9, 165.3, 192.03; ESIMS: m/z 354 (M+H).

2-Chloro-8-nitroindolo [2,1-*b*]quinazoline-6,12-dione (3r)

Yellow solid; mp >250°C ; Found C, 54.79; H, 1.81; N, 12.94 C₁₅H₆ClN₃O₄ requires C, 54.98; H, 1.85; N, 12.82. ν_{\max} (KBr): 3052, 2872, 1721, 1648, 1462, 1208, cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.52 (d, 1H, *J*= 6.9 Hz), 8.40 (s, 1H); 8.04-7.88 (m, 2H), 7.76 (d, 1H, *J*= 6.13 Hz), 7.52 (d, 1H, *J*= 7.16 Hz); ¹³C NMR (75 MHz CDCl₃) δ = 117.3, 122.6, 123.8, 124.2, 124.9, 126.3, 132.9, 138.4, 139.6, 144.4, 152.5, 162.6, 164.8, 192.2; ESIMS: m/z 328 (M+H).

8-Chloro-2-nitroindolo[2,1-*b*]quinazoline-6,12-dione (3s)

Yellow solid; mp >250°C ; Found C, 54.81; H, 1.68; N, 12.89 C₁₅H₆ClN₃O₄ requires C, 54.98; H, 1.85; N, 12.82; ν_{\max} (KBr): 3058, 2965, 1724, 1656, 1452, 1216, cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.51 (s, 1H); 8.30 (d, 1H, *J*= 8.1 Hz), 8.12 (d, 1H, *J*= 7.3 Hz), 7.81-7.68 (m., 3H); ¹³C NMR (75 MHz CDCl₃) δ = 118.6, 121.2, 123.7, 124.3, 125.1, 131.2.6, 131.7, 138.8, 140.9, 143.8, 153.5, 161.7, 165.2, 193.02 ; ESIMS: m/z 328 (M+H).

8-Chloro-2,3-dimethoxyindolo[2,1-*b*]quinazoline-6,12-dione (3t)

Yellow solid; mp >250°C; Found C, 59.36; H, 3.08; N, 8.29 C₁₇H₁₁ClN₂O₄ requires C, 59.57; H, 3.23; N, 8.17; ν_{\max} (KBr): 3054, 2862, 1725, 1656, 1441, 1225 cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.41 (s, 1H), 8.29 (s, 1H), 8.14 (d, 1H, *J*= 8.3 Hz); 7.91-7.79 (m, 2H); 3.86 (s, 6H); ¹³C NMR (75 MHz CDCl₃) δ = 51.604, 111.4, 113.2, 115.1, 117.3, 121.0, 121.7, 133.9, 138.2, 141.3, 143.2, 148.1, 162.0, 164.8, 193.3; ESIMS: m/z 388 (M+H).

2-Chloro-8-fluoroindolo[2,1-*b*]quinazoline-6,12-dione (3u)

Yellow solid; mp >250°C ; Found C, 59.79; H, 1.88; N, 9.48 C₁₅H₆ClFN₂O₂, requires C, 59.92; H, 2.01; N, 9.32; ν_{\max} (KBr): 3061, 2945, 1728, 1652, 1450, 1218, cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.63 (d, 1H, *J*= 7.1 Hz), 8.51 (s, 1H); 8.38 (d, 1H, *J*= 6.7 Hz), 8.28-8.14 (m, 2H), 8.04 (d, 1H, *J*= 7.4 Hz) ; ¹³C NMR (75 MHz CDCl₃) δ = 116.8, 119.2, 122.4, 122.8, 124.6; 128.4, 136.7, 138.2, 143.9, 147.6, 156.9, 163.4, 166.8, 196.2; ESIMS: m/z 301 (M+H),

8-Fluoro-2-nitroindolo[2,1-*b*]quinazoline-6,12-dione (3v)

Yellow solid; mp >250°C ; Found C, 57.71; H, 1.80; N, 13.64 . C₁₅H₆FN₃O₄, requires C, 57.89; H, 1.94; N, 13.50; ν_{\max} (KBr): 3108, 2975, 1724, 1636, 1440, 12039 cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.46 (s, 1H); 8.34 (d, 1H, *J*= 7.6 Hz), 8.14 (d, 1H, *J*= 8.4 Hz), 7.98-7.85 (m, 2H), 7.74 (d, 1H, *J*= 7.9 Hz); ¹³C NMR (75 MHz CDCl₃) δ = 119.4, 120.8, 123.9, 124.3, 126.1, 126.6, 132.7, 137.4, 141.9, 144.2, 154.5, 163.1, 165.8, 195.0 ; ESIMS: m/z 311 (M+H).

2-Chloro-8-methoxyindolo[2,1-*b*]quinazoline-6,12-dione (3w)

Yellow solid; mp >250°C; Found C, 61.59; H, 2.98; N, 8.81 C₁₆H₉ClN₂O₃, requires C, 61.45; H, 2.90; N, 8.96; ν_{\max} (KBr): 3218, 2872, 1721, 1665, 1445, 1232, cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.28 (d, 1H, *J*= 6.24 Hz), 8.02 (s, 1H); 7.91-7.84 (m, 2H) 7.68 (d, 1H, *J*= 5.4 Hz), 7.52 (d, 1H, *J*= 6.8 Hz), 3.68 (s, 3H); ¹³C NMR (75 MHz CDCl₃) δ = 53.4, 116.3, 118.1, 124.8, 126.2, 127.1, 128.8, 131.6, 142.7, 147.8, 153.2, 162.1, 190.2; ESIMS: m/z 313 (M+H),

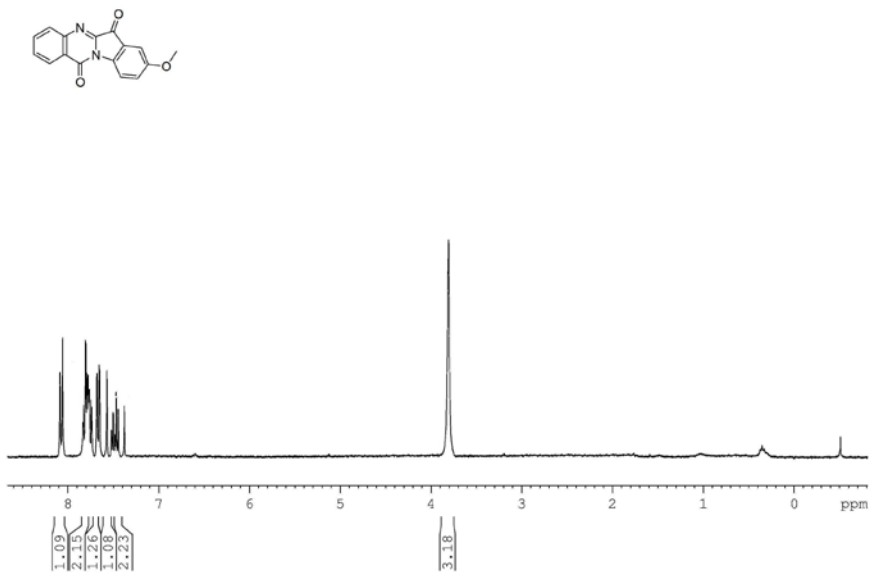
8-Methoxy-2-nitroindolo[2,1-*b*]quinazoline-6,12-dione (3x)

Yellow solid; mp >250°C; Found C, 59.27; H, 2.63; N, 13.16 C₁₆H₉N₃O₅, requires C, 59.45; H, 2.81; N, 13.00; ν_{\max} (KBr): 3108, 2971, 1724, 1675, 1455, 1212, cm⁻¹; ¹H NMR (300 MHz CDCl₃) δ = 8.32 (d, 1H, *J*= 7.14 Hz), 8.22 (s, 1H); 8.04-7.91 (m, 2H) 7.84 (d, 1H, *J*= 6.4 Hz), 7.67 (d, 1H, *J*= 8.7 Hz), 3.91 (s, 3H); ¹³C NMR (75 MHz CDCl₃) δ = 50.3, 115.9, 117.4, 123.6, 125.2, 128.1, 128.8, 131.2, 132.8, 133.6, 140.7, 145.8, 153.2, 163.4, 193.6; ESIMS: m/z 324 (M+H).

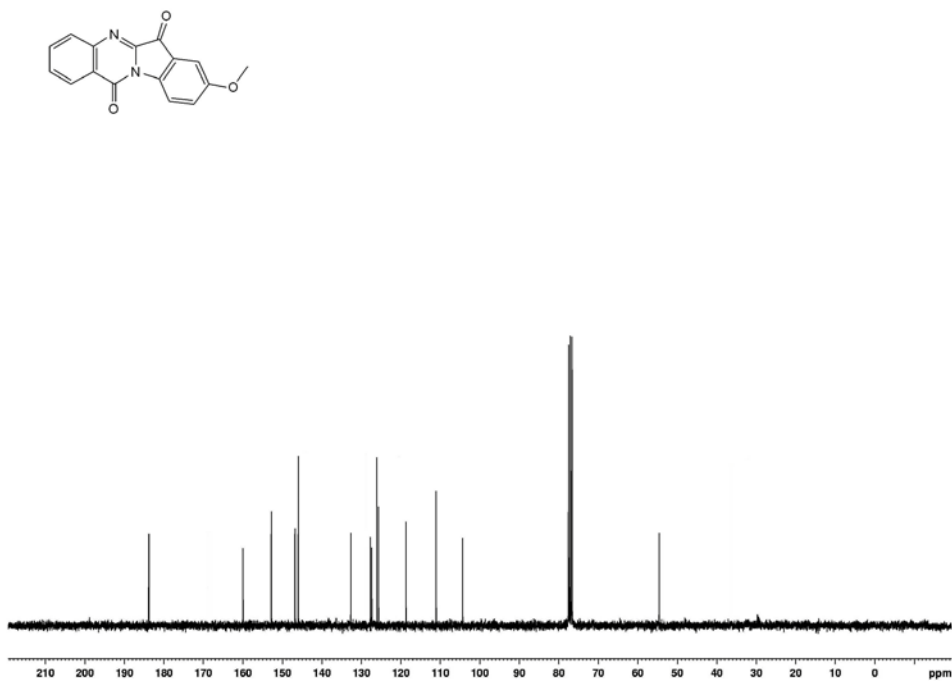
Reference:

1. B. Batanero, and F. Barba, *Tetrahedron Lett.* 2006, **47**, 8201-8203.
2. V. M. Sharma, P. Prasanna, K. V. Adi Seshu, B. Renuka, C. V. Laxman Rao, G. Sunil Kumar, C. Prasad Narasimhulu, P. Aravind Babu, R. C. Puranik, D. Subramanyam, A. Venkateswarlu, S. Rajagopal, K. B. Sunil Kumar, C. S. Rao, N. V. S. Rao Mamidi, D. S. Deevi, R. Ajaykumarb and R. Rajagopalanb, *Bioorg. Med. Chem. Lett.*, 2002, **12**, 2303-2307
3. S.-T. Yu, J.-W. Chern, T.-M. Chen, Y.-F. Chiu, H.-T. Chen and Y.-H. Chen, *Acta Pharmacologica Sinica*, 2010, **31**, 259-264.

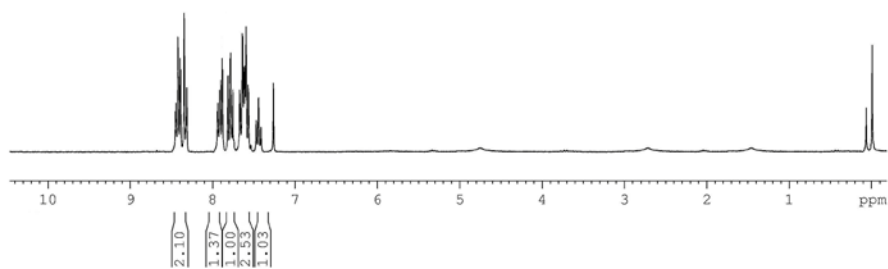
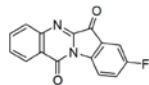
3g)



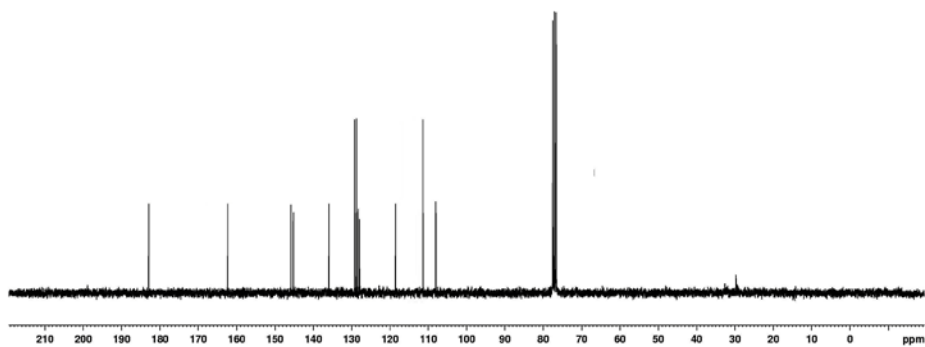
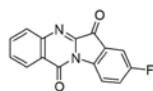
3g)



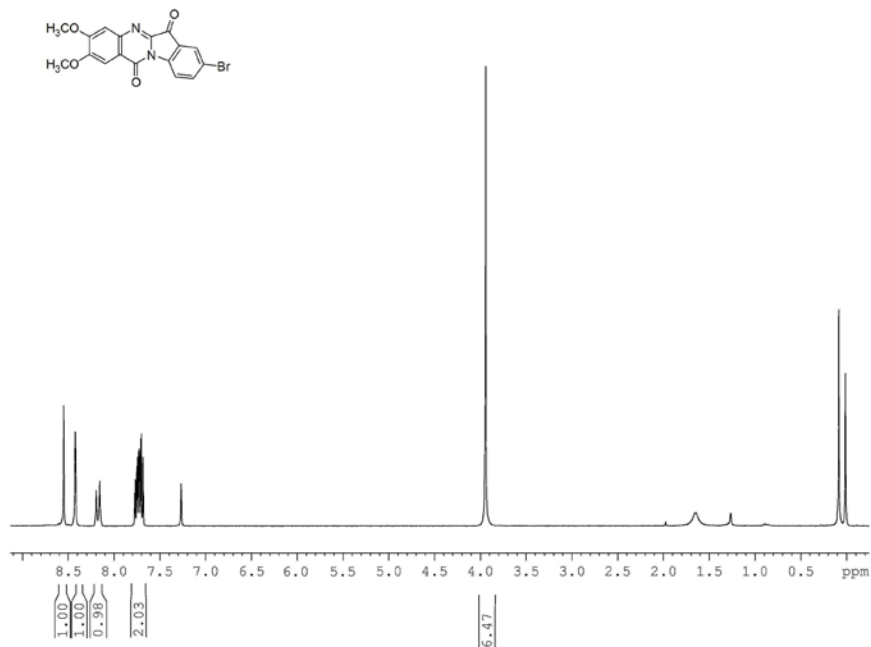
3k)



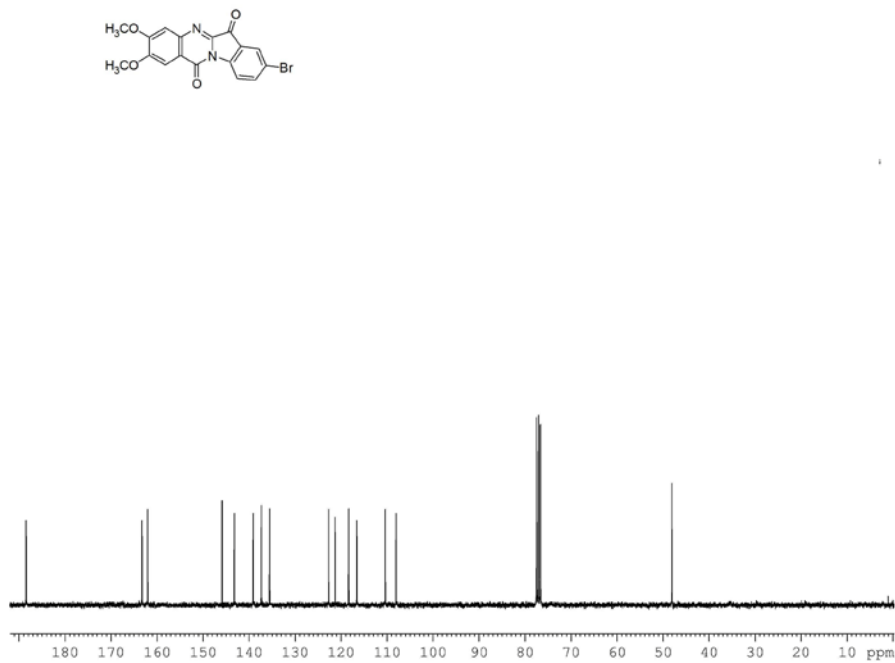
3k)



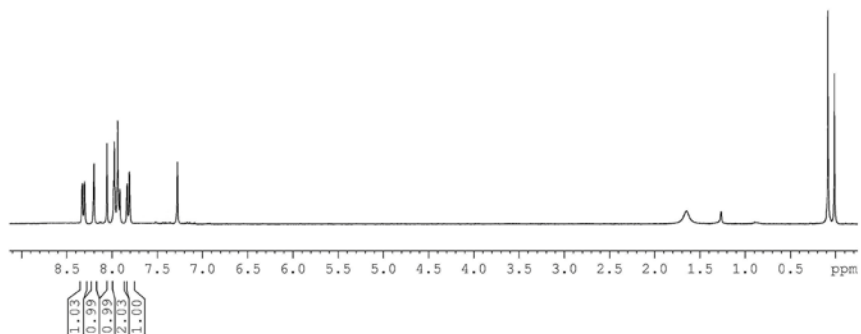
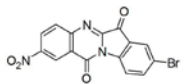
3m)



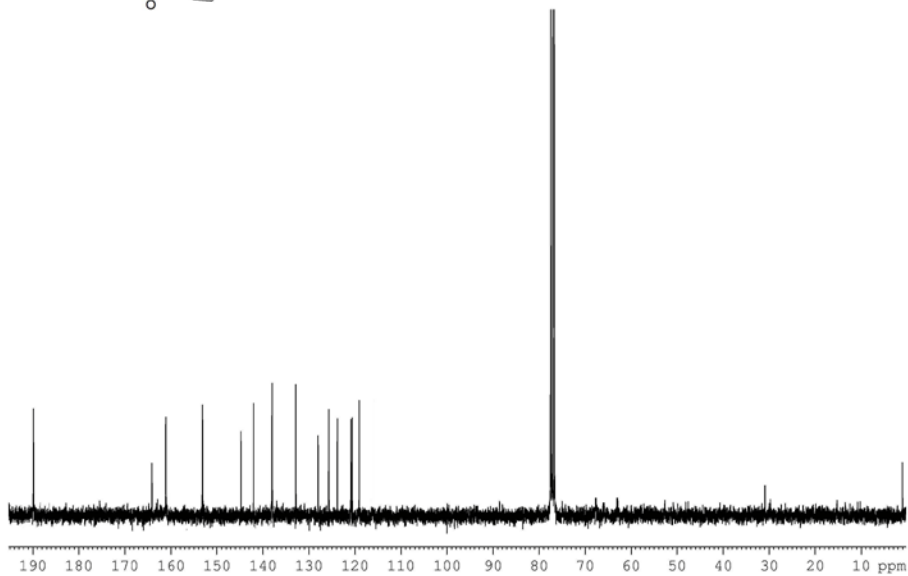
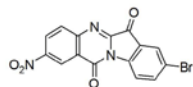
3m)



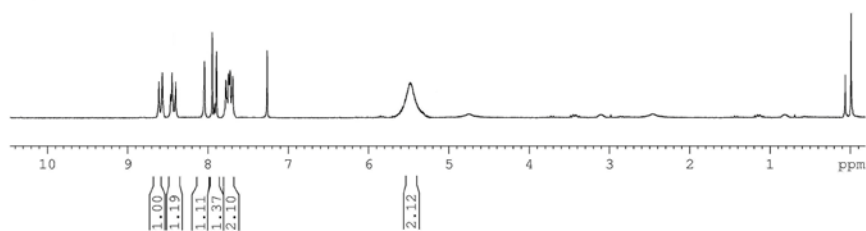
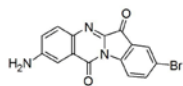
3n)



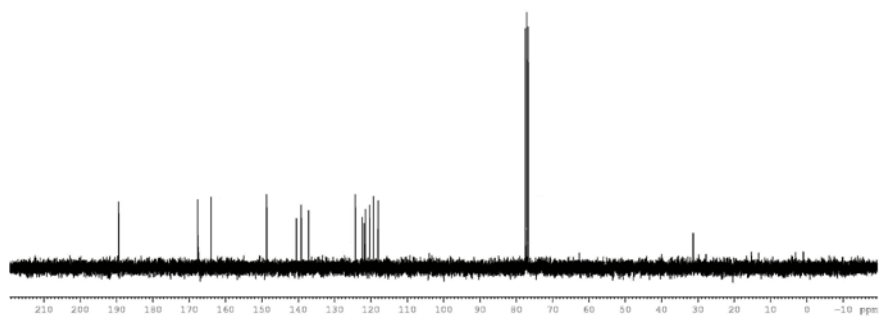
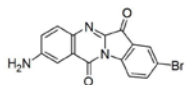
3n)



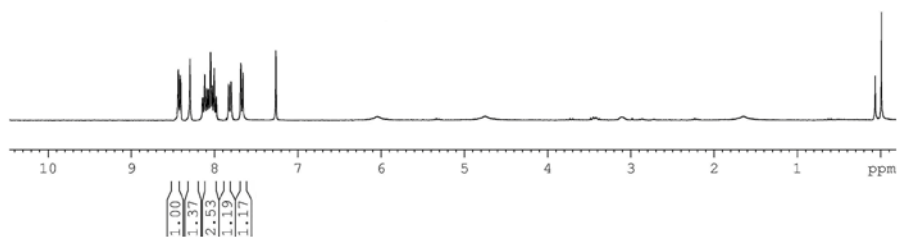
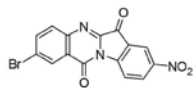
3o)



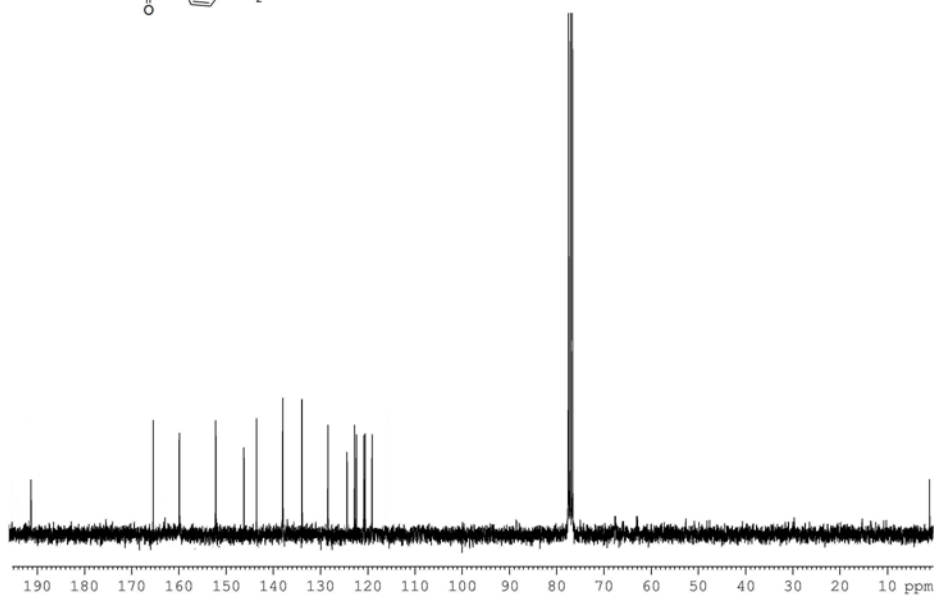
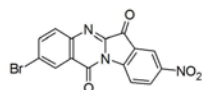
3o)



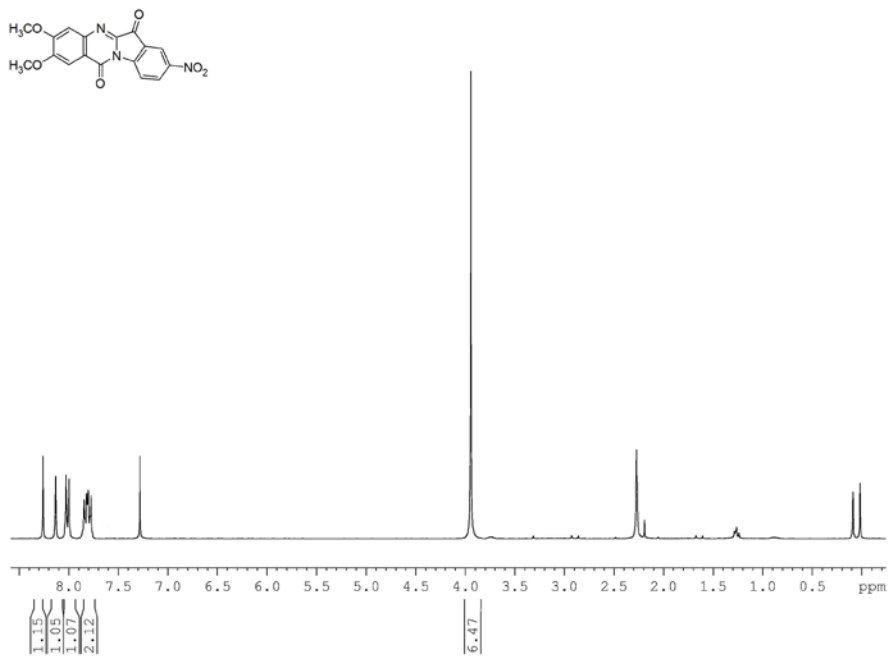
3p)



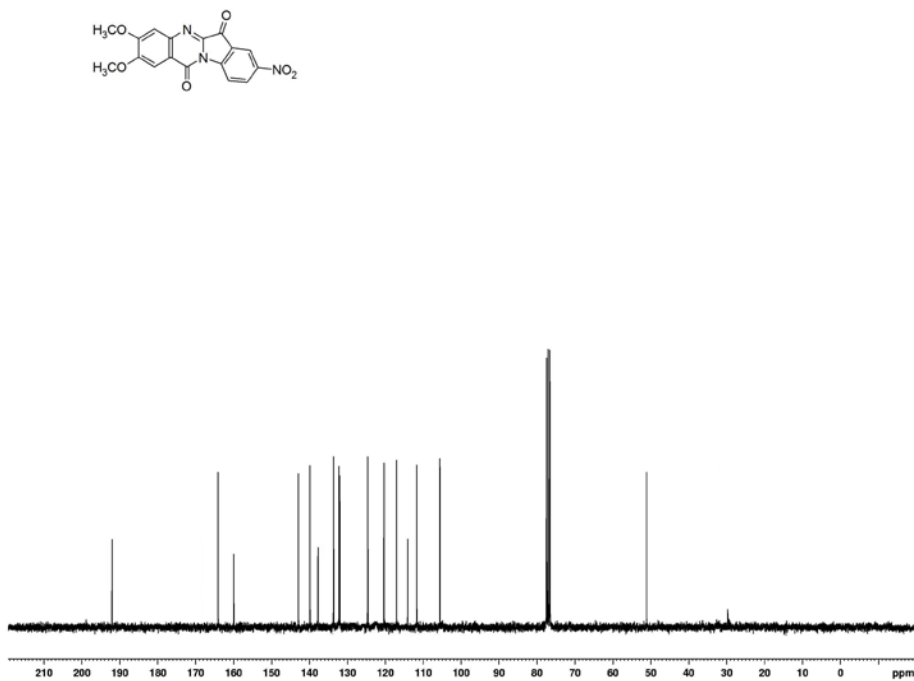
3p)



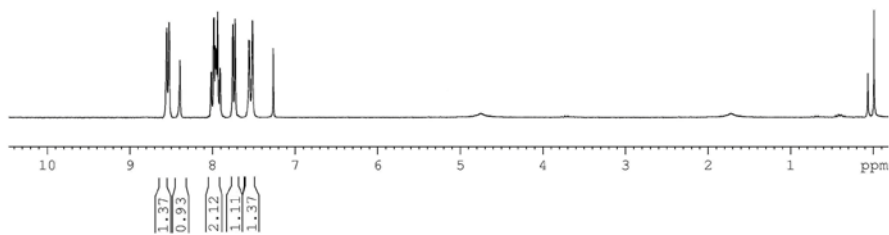
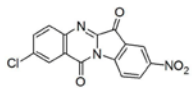
3q)



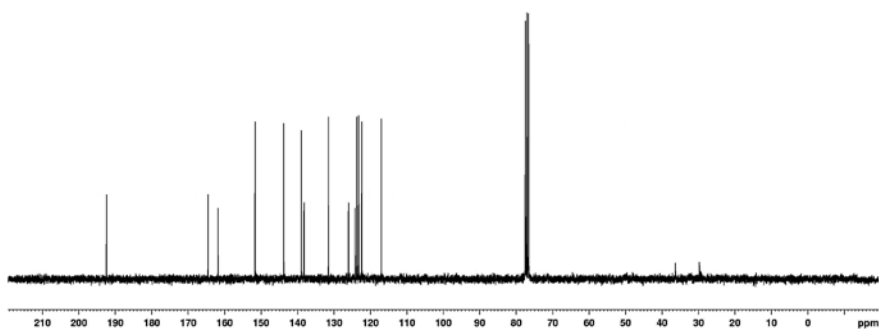
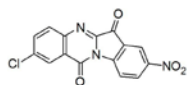
3q)



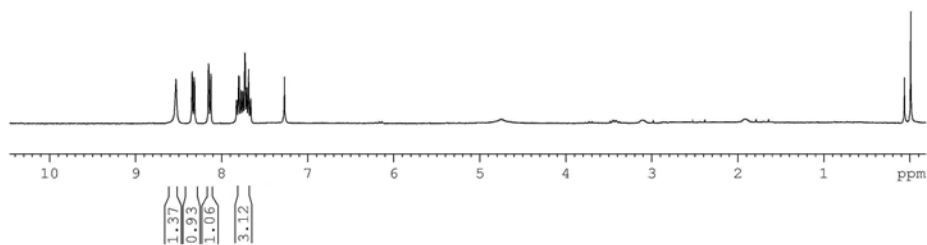
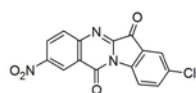
3r)



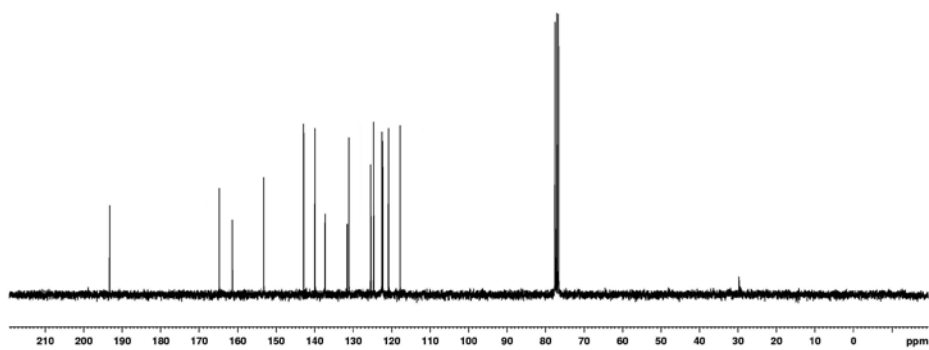
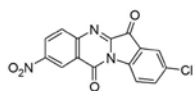
3r)



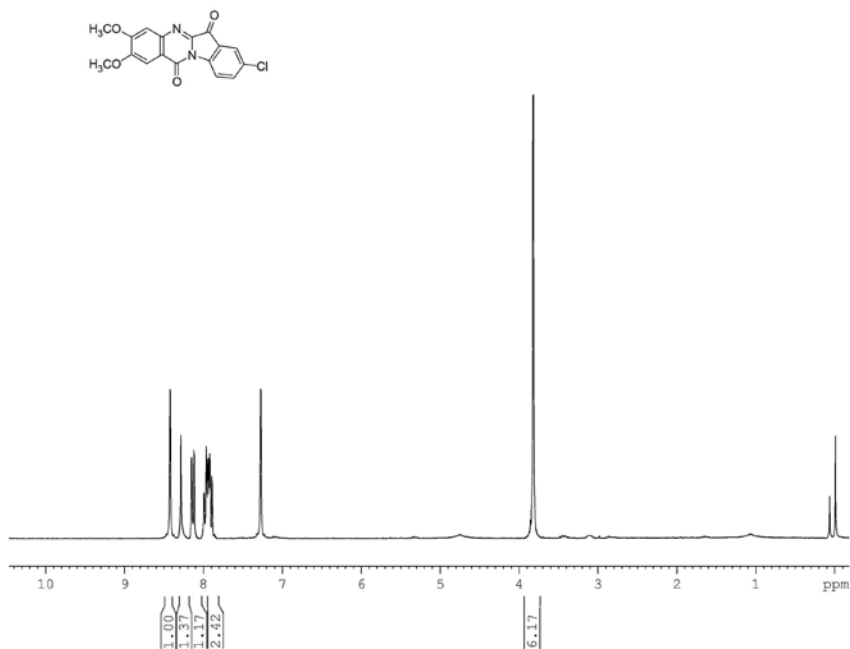
3s)



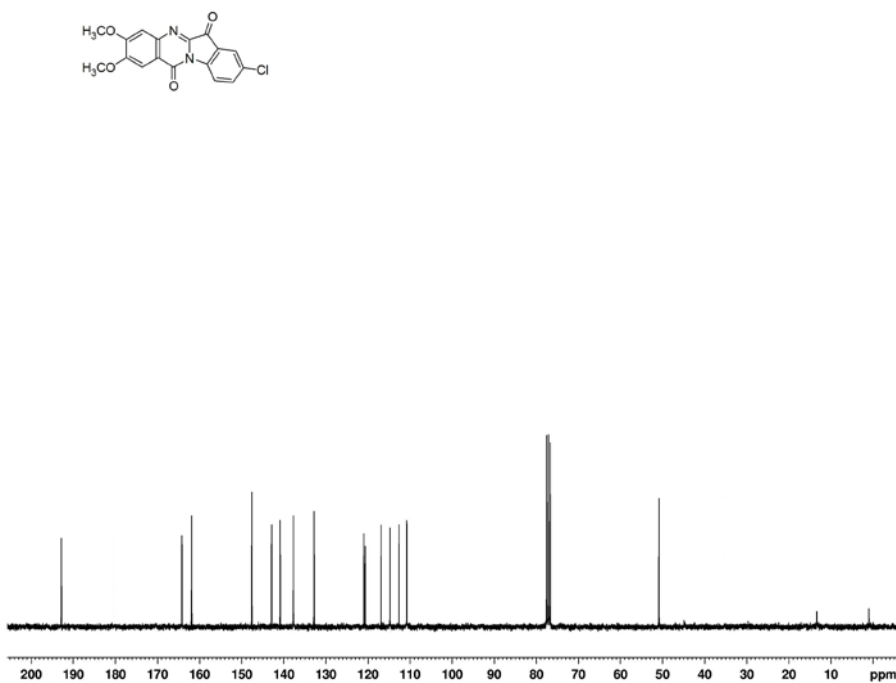
3s)



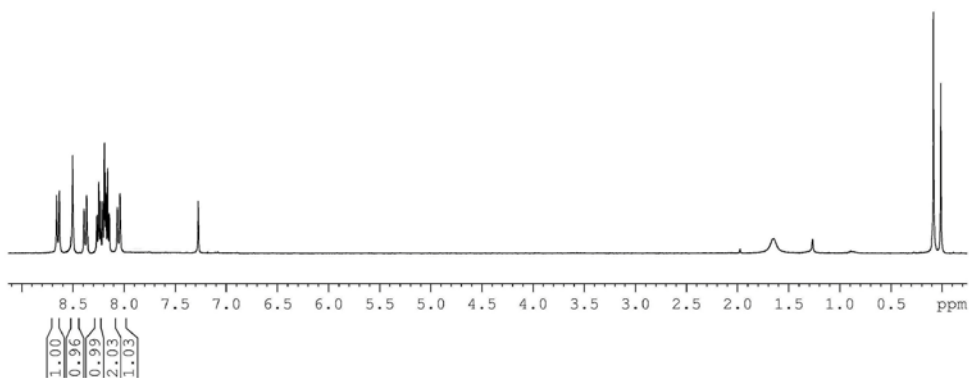
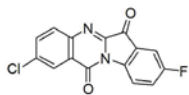
3t)



3t)



3u)



3u)

