

Supporting Information

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One-pot multi-component process for the synthesis of 4-azaphenanthrene-3,10-dione, 1,8-dioxo-octahydroxanthene and tetrahydrobenzo[*b*]pyran derivatives catalyzed by the deep eutectic solvent choline chloride-oxalic acid

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Characterization of compounds 8c, 8d, 8g and 4e

1-(4-Chloro-phenyl)-1,4-dihydro-2H-9-oxa-4-aza-phenanthrene-3,10-dione (8c)

White powder; m.p. 218–220 °C. – FT-IR (KBr, cm^{-1}): ν = 3185, 3139, 2923, 1690, 1637, 1605, 1571, 1515, 1464, 1407, 1368, 1318, 1240, 1195, 1012, 999, 829, 769, 739, 627, 547. – ^1H NMR ($\text{DMSO-}d_6$, 500 MHz, ppm): δ = 2.58 (dd, 1H), 3.32 (d, 1H), 4.37 (d, 1H), 7.20–8.24 (m, ArH, 8H), 11.01 (s, NH, 1H). – ^{13}C NMR ($\text{DMSO-}d_6$, 125 MHz, ppm): δ = 171.6, 160.1, 152.7, 147.2, 141.7, 134.0, 133.1, 130.2, 130.0, 125.7, 124.6, 118.4, 114.5, 104.4, 37.05, 36.4.

1-(2,4-Dichloro-phenyl)-1,4-dihydro-2H-9-oxa-4-aza-phenanthrene-3,10-dione (8d)

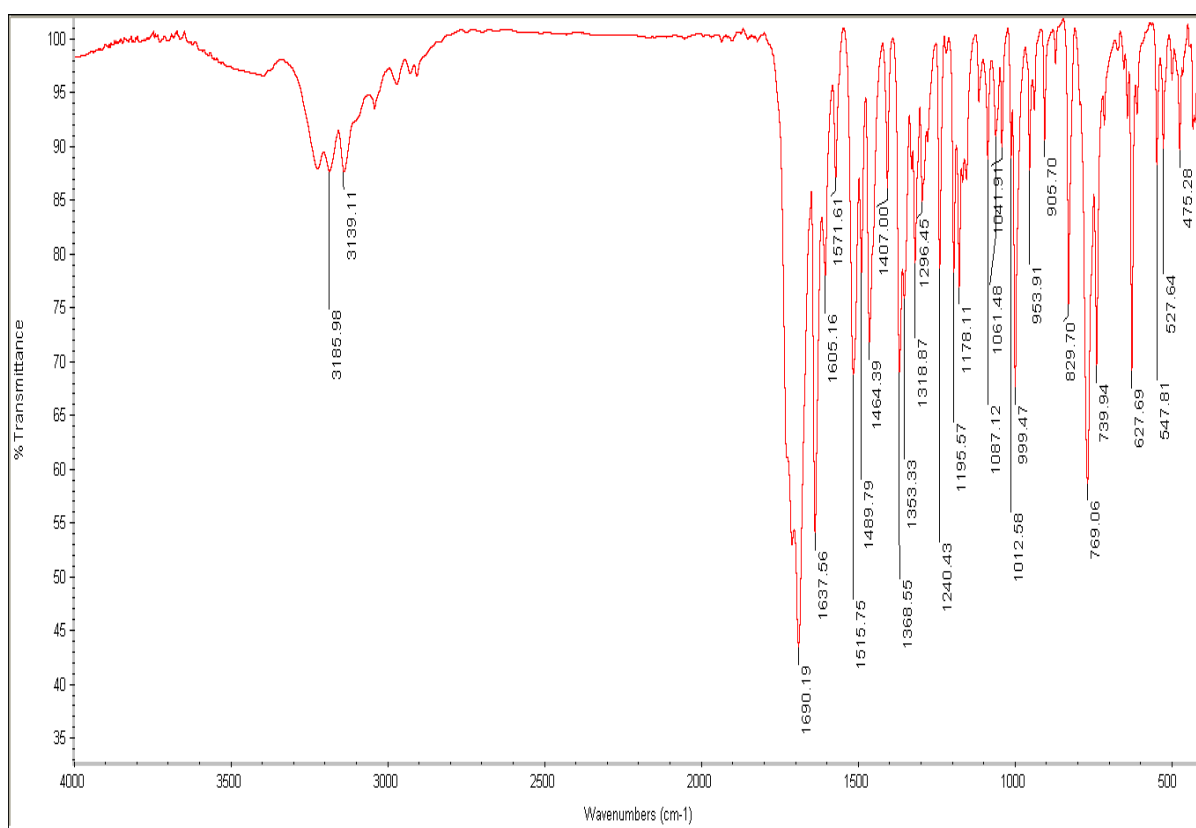
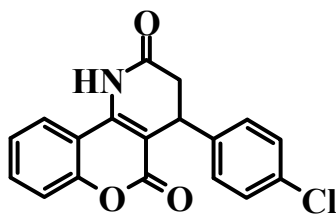
White powder; m.p. 185–187 °C. – FT-IR (KBr, cm^{-1}): ν = 3239, 3038, 2923, 1722, 1692, 1671, 1633, 1609, 1574, 1519, 1463, 1363, 1324, 1200, 1165, 1049, 821, 766, 748, 630, 598, 480. – ^1H NMR ($\text{DMSO-}d_6$, 500 MHz, ppm): δ = 2.53 (dd, 1H), 3.33 (d, 1H), 4.65 (d, 1H), 7.20–8.24 (m, ArH, 7H), 11.13 (s, NH, 1H). – ^{13}C NMR ($\text{DMSO-}d_6$, 125 MHz, ppm): δ = 170.9, 161.4, 154.3, 148.4, 138.3, 134.9, 134.2, 131.0, 130.2, 129.3, 125.8, 124.8, 118.4, 114.5, 103, 37.6, 34.4.

1-(4-Nitro-phenyl)-1,4-dihydro-2H-9-oxa-4-aza-phenanthrene-3,10-dione (8g)

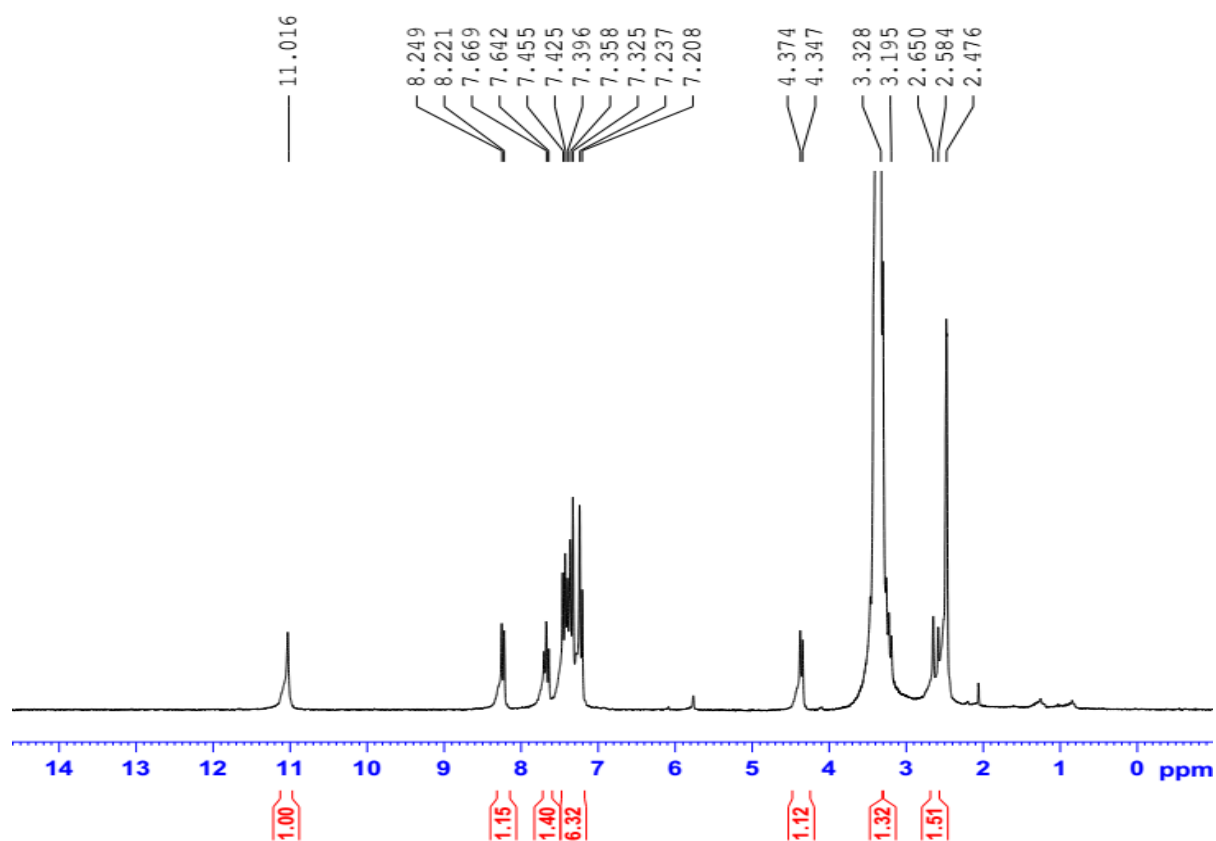
White powder; m.p. 201–203 °C. – FT-IR (KBr, cm^{-1}): ν = 3265, 3078, 1723, 1690, 1684, 1625, 1511, 1459, 1411, 1348, 1277, 1231, 1196, 1155, 1112, 1070, 1004, 946, 906, 858, 758, 730, 632, 518. – ^1H NMR ($\text{DMSO-}d_6$, 500 MHz, ppm): δ = 2.89 (dd, 1H), 3.33 (d, 1H), 4.52 (d, 1H), 7.20–8.27 (m, ArH, 8H), 11.10 (s, NH, 1H). – ^{13}C NMR ($\text{DMSO-}d_6$, 125 MHz, ppm): δ = 171.3, 161.6, 154.3, 150.6, 148.1, 147.6, 134.1, 129.6, 125.7, 125.4, 124.7, 118.4, 114.5, 103.6, 38.9, 36.9.

2-Amino-3-cyano-4-(4-methylphenyl)-7,7-dimethyl-5-oxo-4*H*-5,6,7,8-tetrahydrobenzo[*b*]pyran (4e)

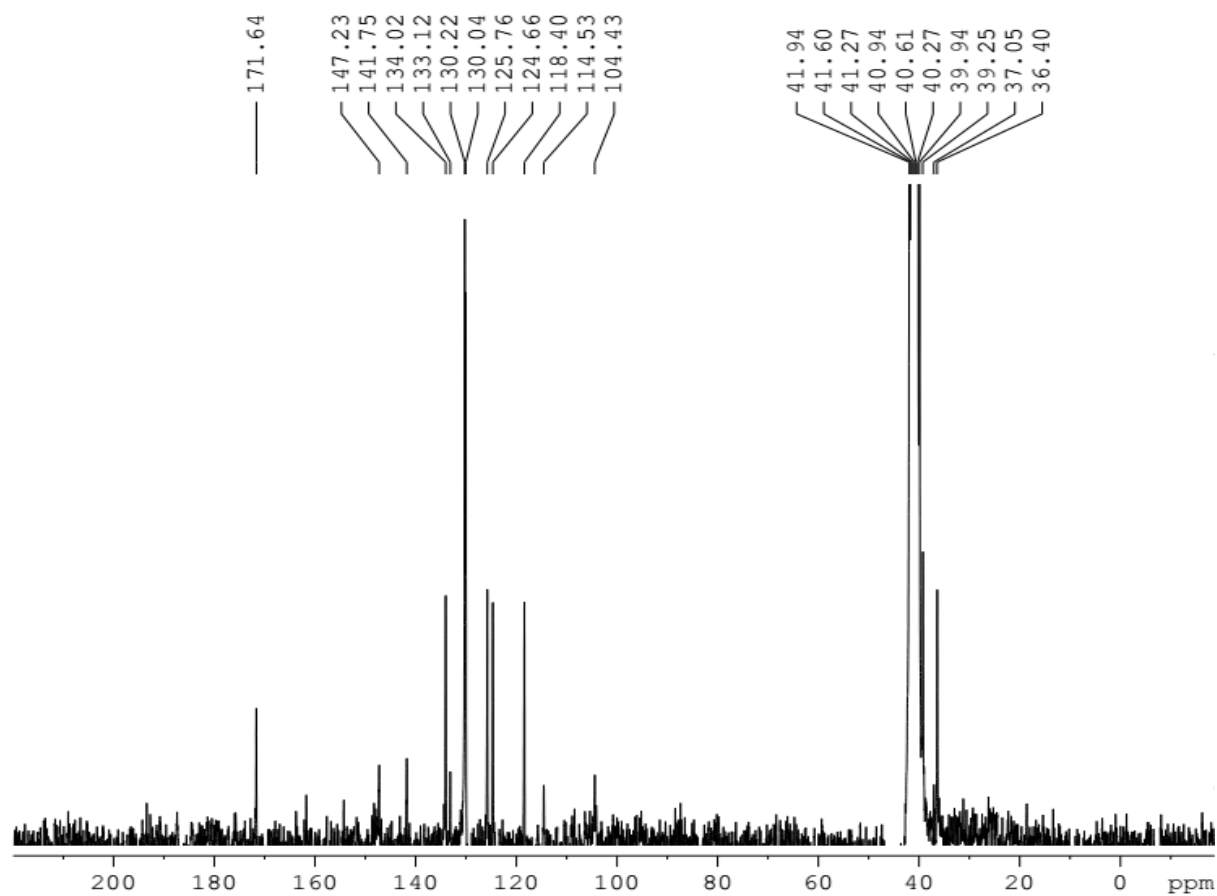
White powder; m.p. 218–220 °C. – FT-IR (KBr, cm^{-1}): ν = 3425, 3321, 2995, 2187, 1672, 1638, 1598, 1484, 1365. – ^1H NMR ($\text{DMSO-}d_6$, 500 MHz, ppm): δ = 0.89 (s, 3H), 0.96 (s, 3H), 1.89 (d, 1H), 2.03 (d, 1H), 2.12 (s, 3H), 2.33 (s, 2H), 4.13 (s, 1H), 5.8 (s, NH, 2H), 6.90–9.96 (m, ArH, 4H). – ^{13}C NMR ($\text{DMSO-}d_6$, 125 MHz, ppm): δ = 196.0, 162.7, 158.8, 142.2, 136.0, 129.3, 127.5, 120.1, 113.2, 58.8, 50.4, 39.8, 35.5, 32.2, 28.8, 27.1, 21.0.



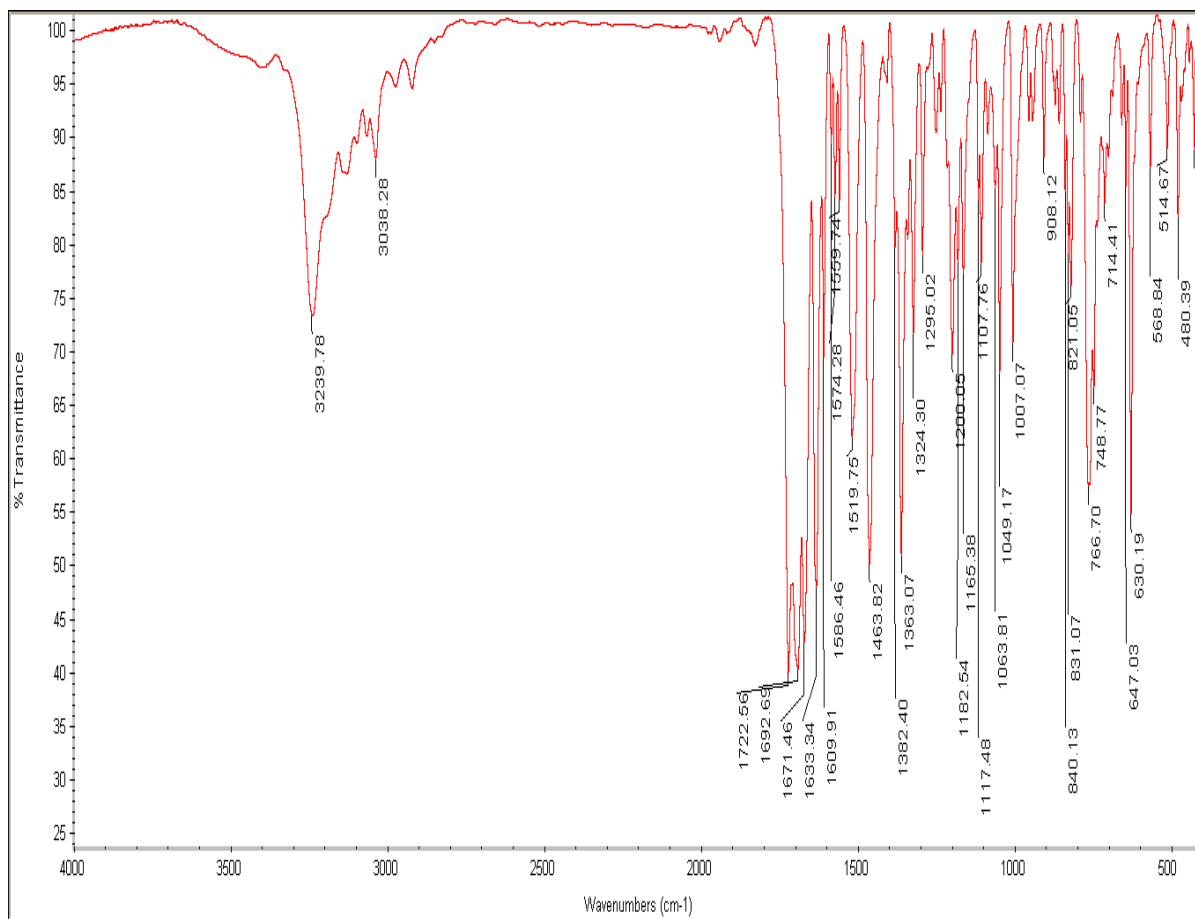
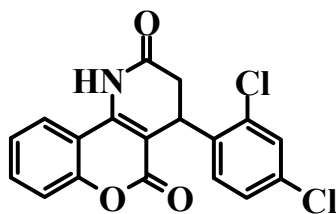
FT-IR: 1-(4-Chloro-phenyl)-1,4-dihydro-2*H*-9-oxa-4-aza-phenanthrene-3,10-dione (8c)



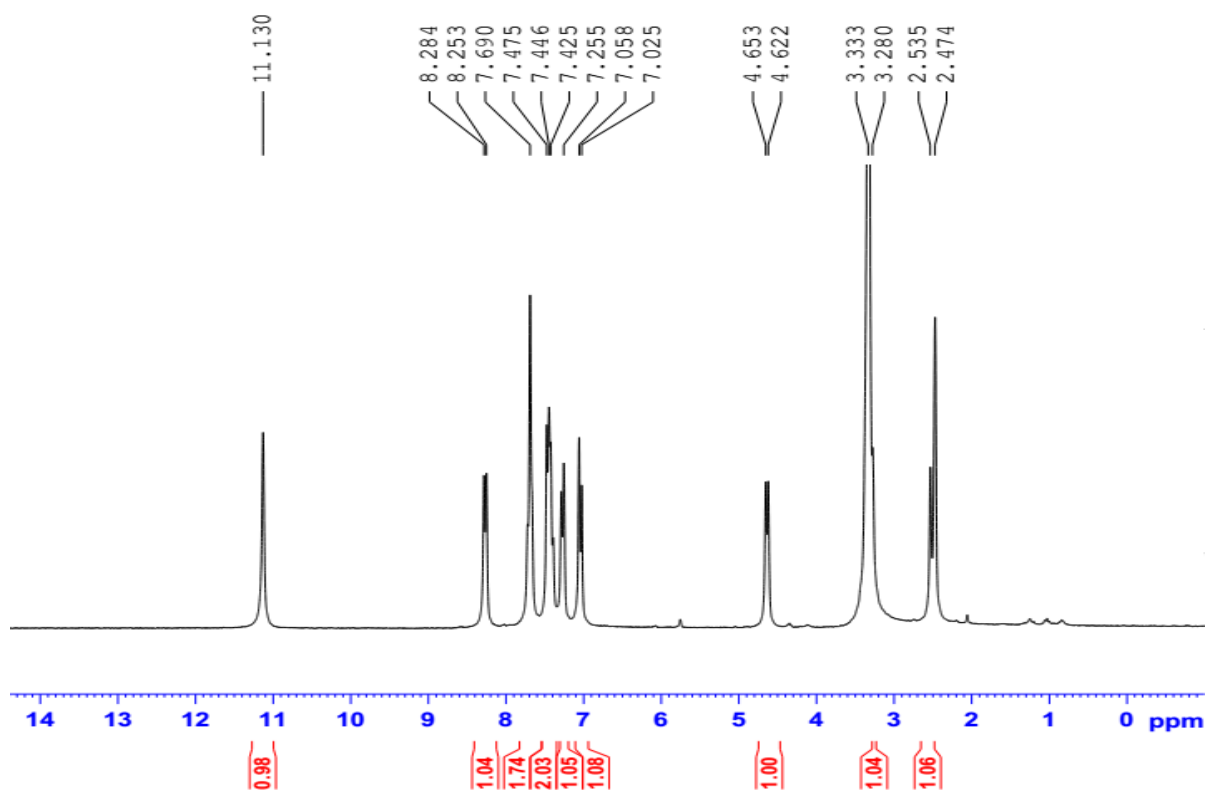
¹H NMR: 1-(4-Chloro-phenyl)-1,4-dihydro-2H-9-oxa-4-aza-phenanthrene-3,10-dione (8c)



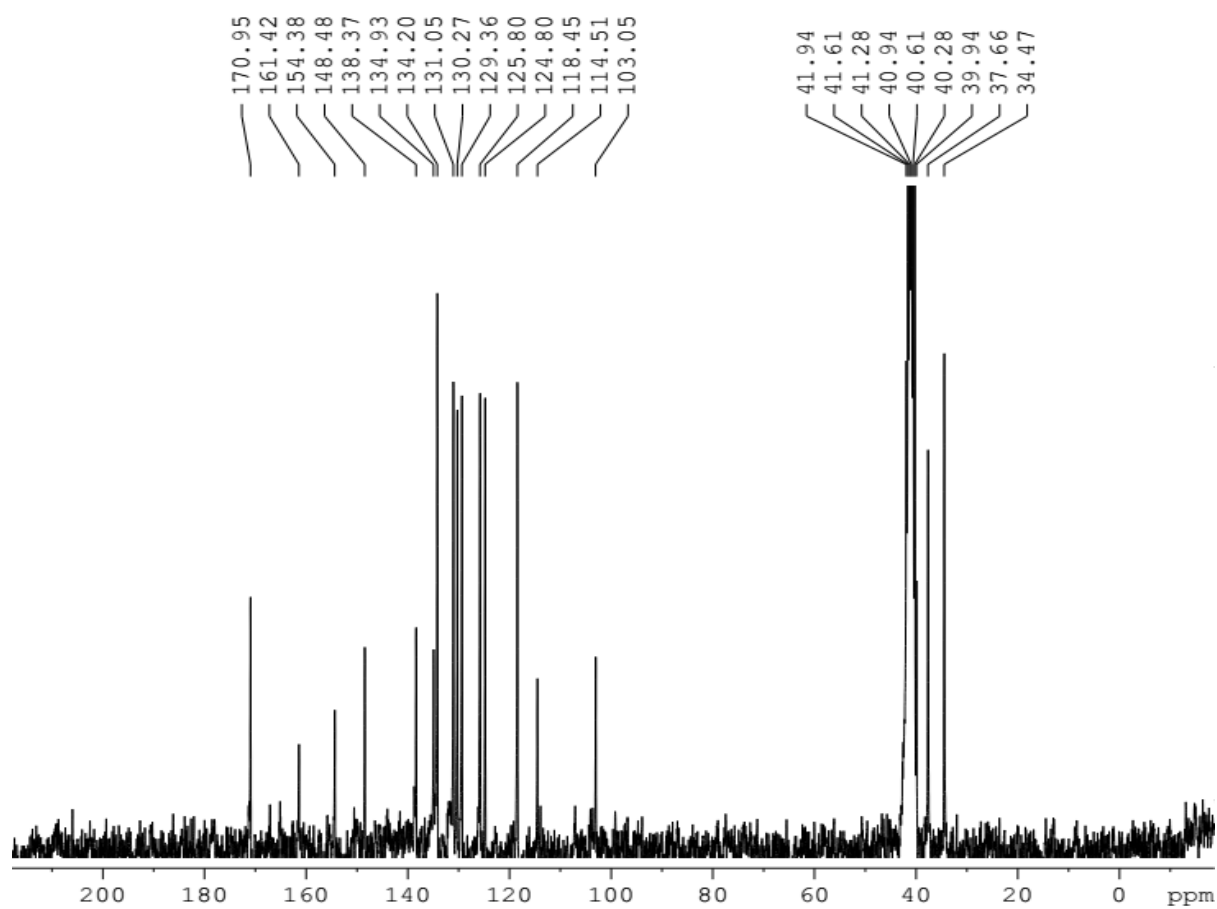
^{13}C NMR: 1-(4-Chloro-phenyl)-1,4-dihydro-2*H*-9-oxa-4-aza-phenanthrene-3,10-dione (8c)



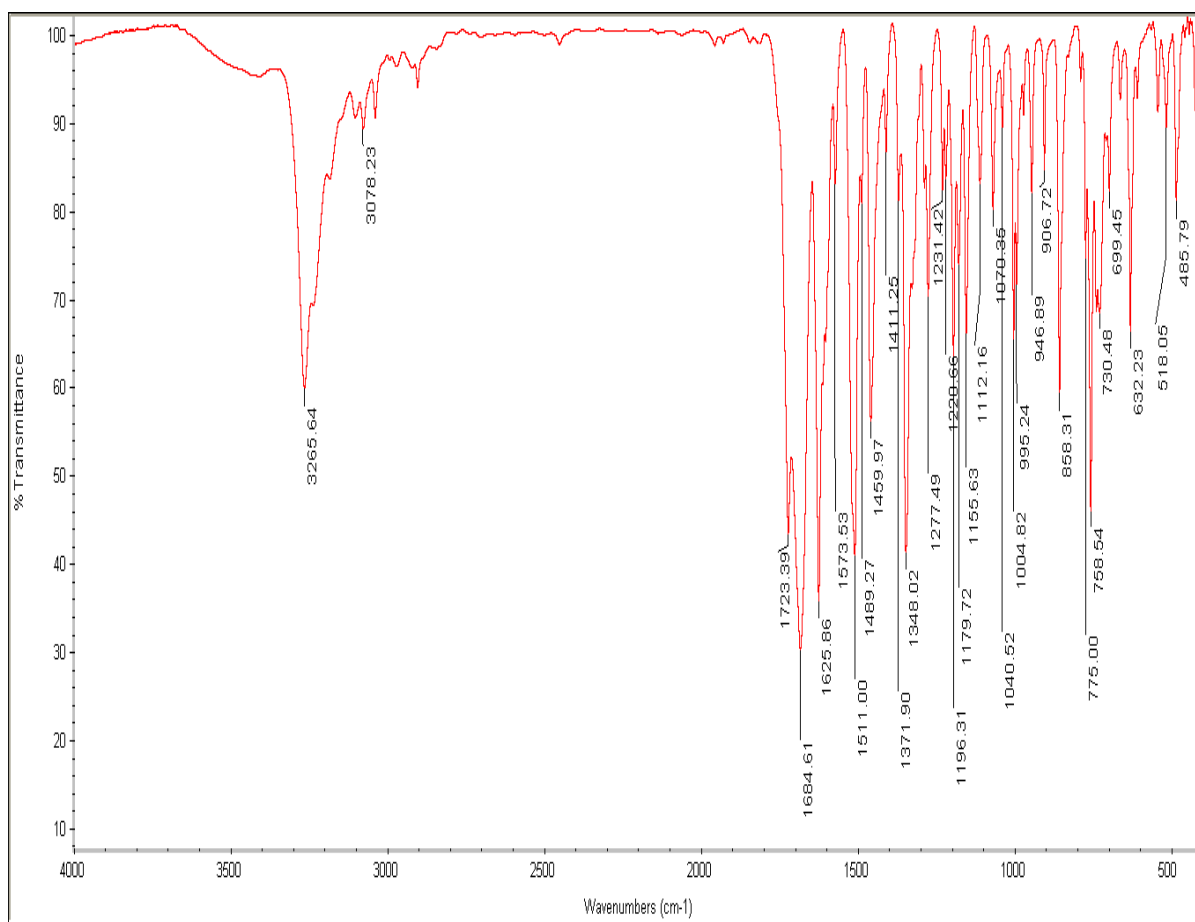
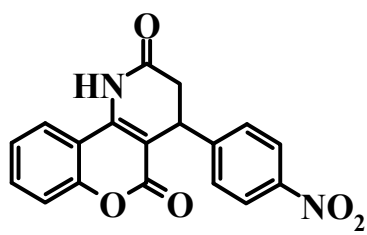
FT-IR: 1-(2,4-Dichloro-phenyl)-1,4-dihydro-2H-9-oxa-4-aza-phenanthrene-3,10-dione (8d)



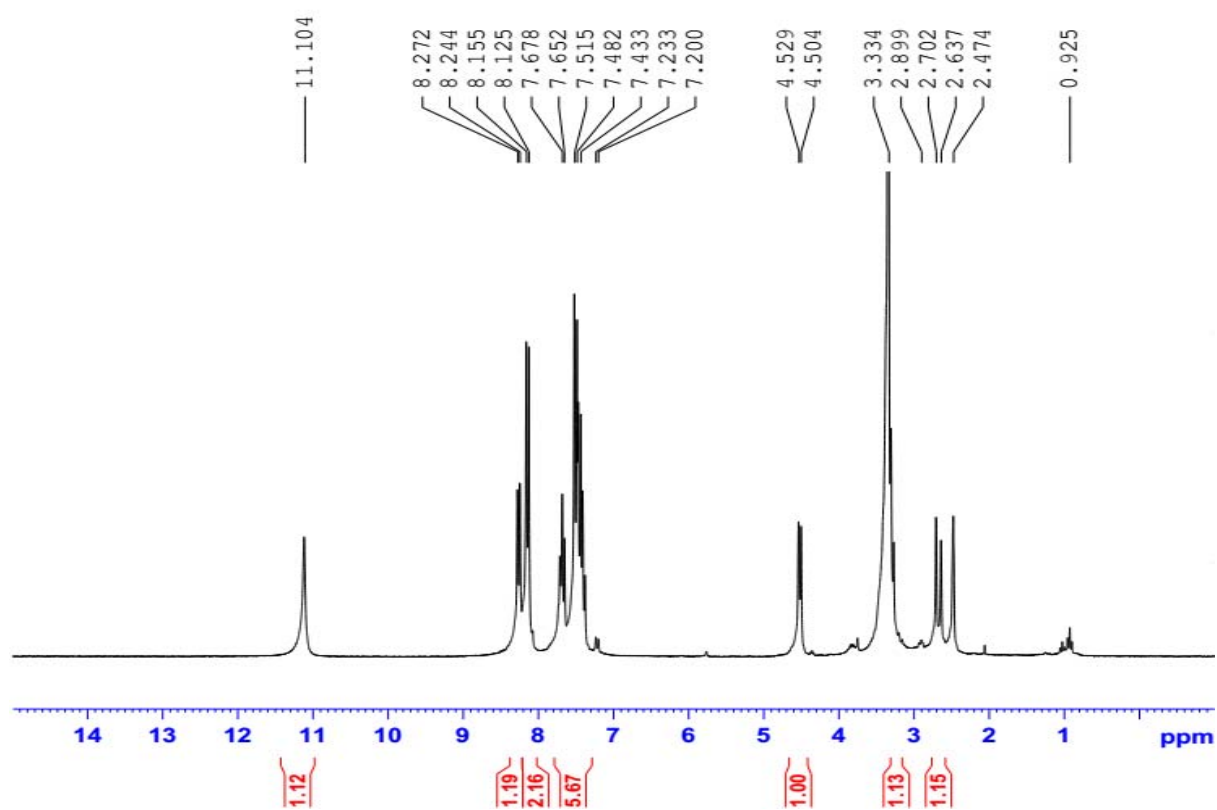
¹H NMR: 1-(2,4-Dichloro-phenyl)-1,4-dihydro-2*H*-9-oxa-4-aza-phenanthrene-3,10-dione (8d)



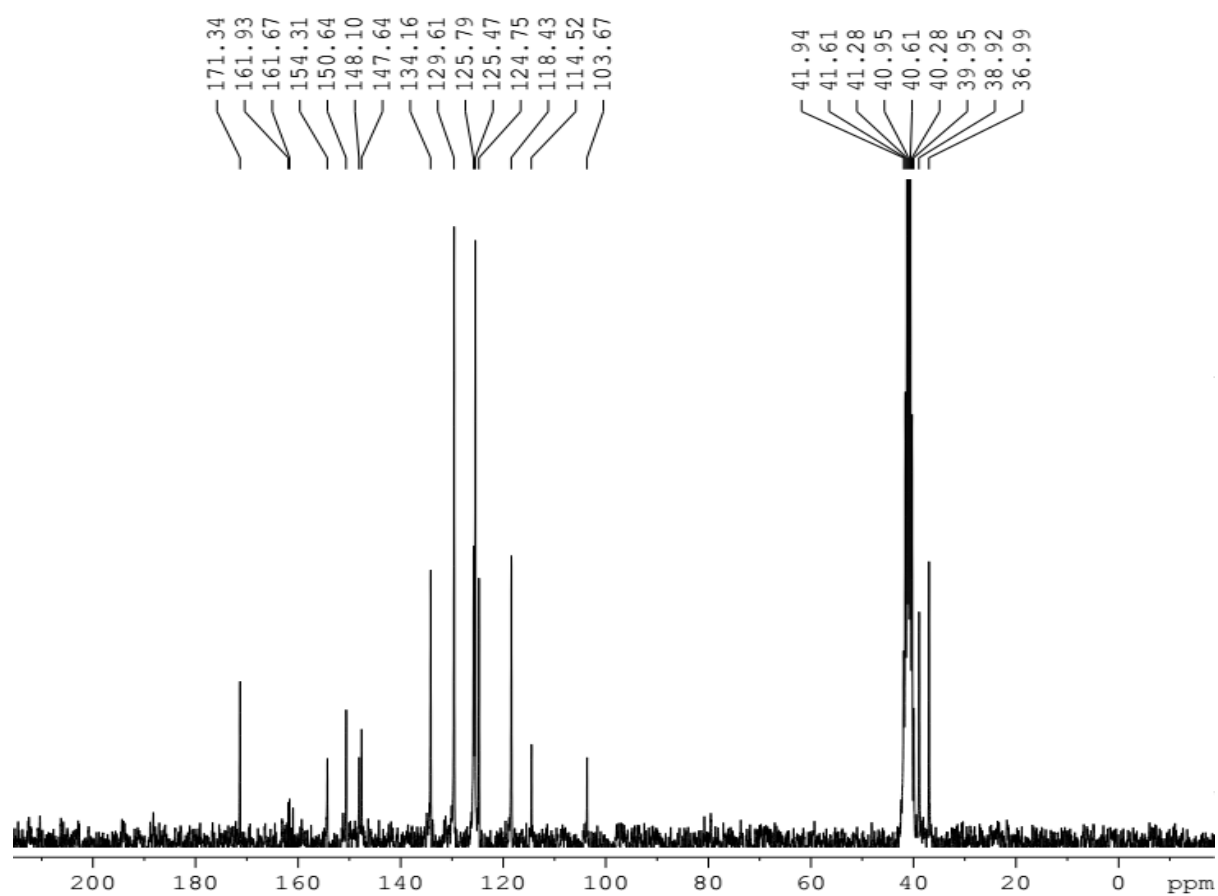
¹³C NMR: 1-(2,4-Dichloro-phenyl)-1,4-dihydro-2H-9-oxa-4-aza-phenanthrene-3,10-dione (8d)



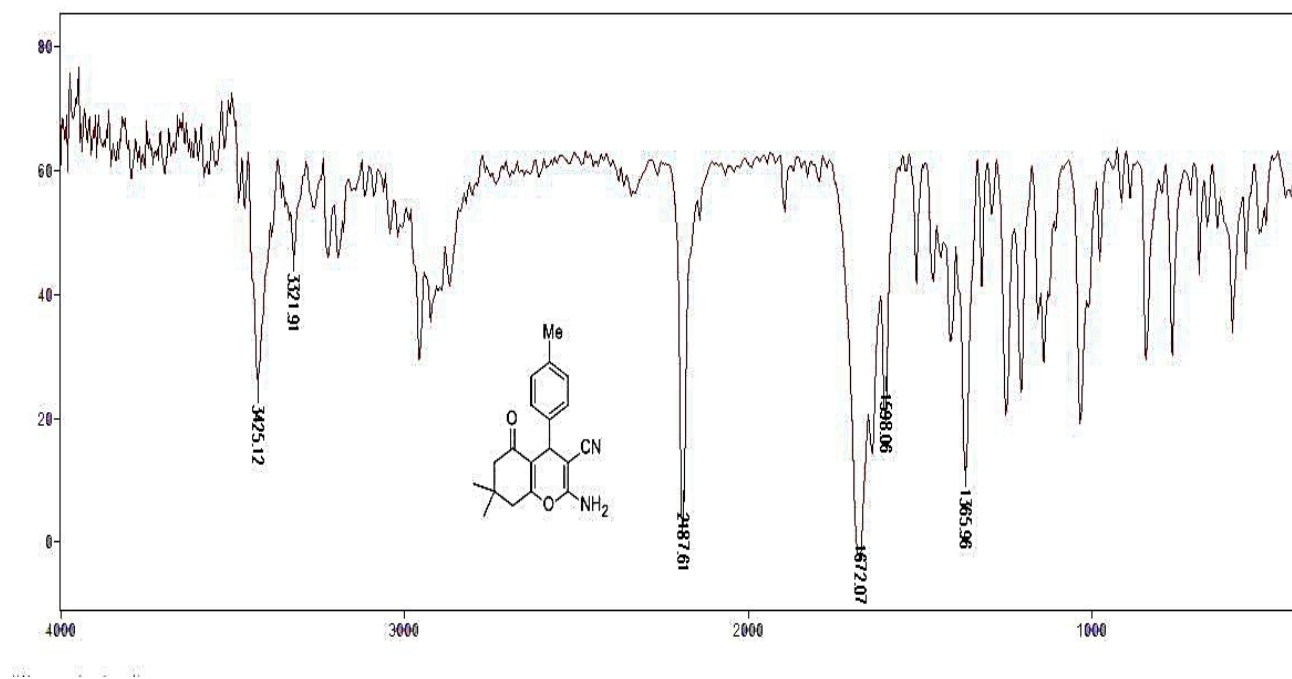
FT-IR: 1-(4-Nitro-phenyl)-1,4-dihydro-2H-9-oxa-4-aza-phenanthrene-3,10-dione (8g)



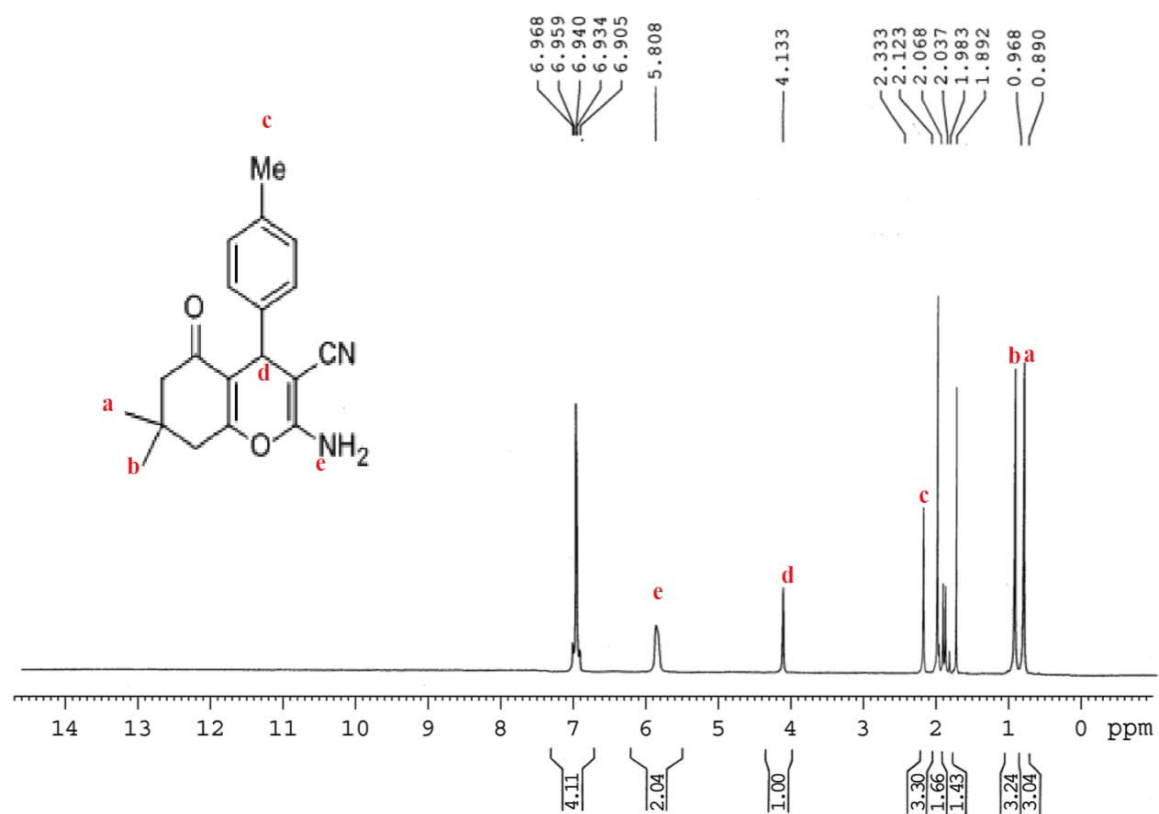
¹H NMR: 1-(4-Nitro-phenyl)-1,4-dihydro-2H-9-oxa-4-aza-phenanthrene-3,10-dione (8g)



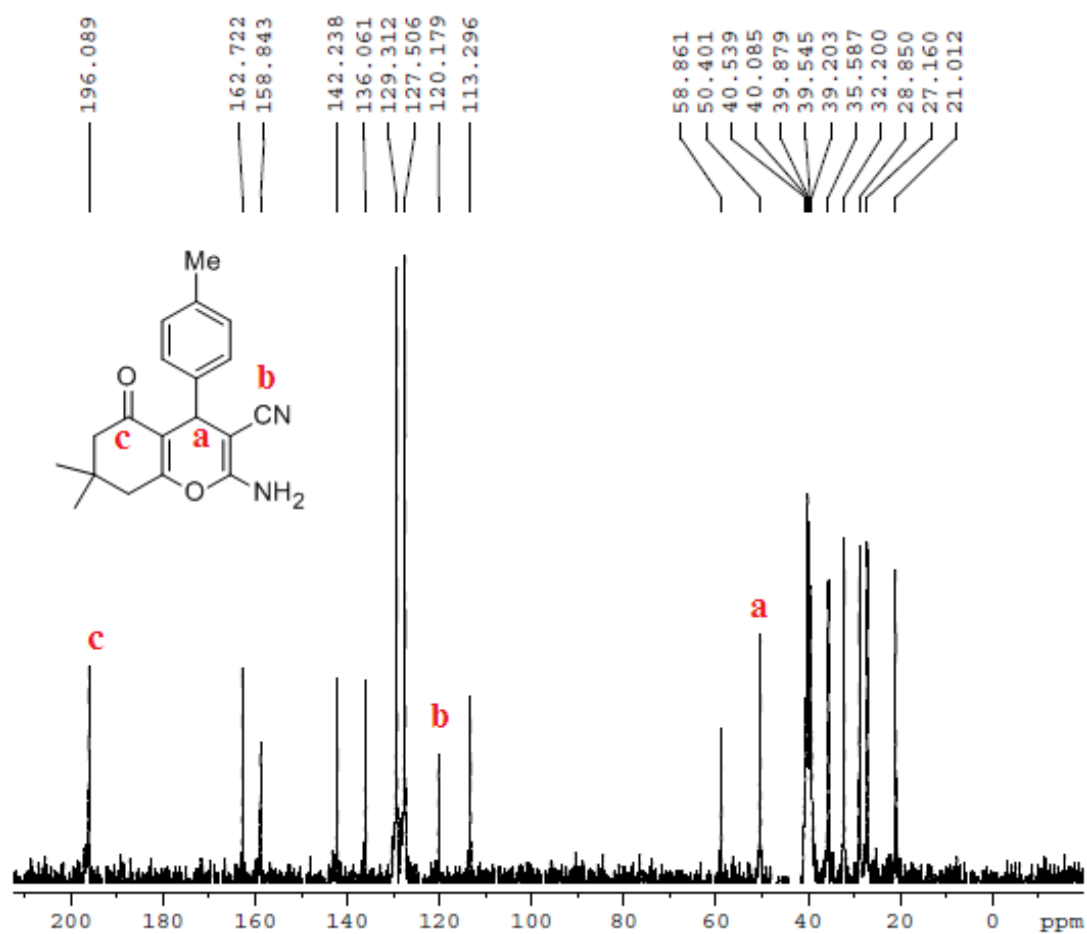
^{13}C NMR: 1-(4-Nitro-phenyl)-1,4-dihydro-2*H*-9-oxa-4-aza-phenanthrene-3,10-dione (8g)



FT-IR: 2-Amino-3-cyano-4-(4-methylphenyl)-7,7-dimethyl-5-oxo-4H-5,6,7,8-tetrahydrobenzo[b] pyran (4e)



¹H NMR: 2-Amino-3-cyano-4-(4-methylphenyl)-7,7-dimethyl-5-oxo-4H-5,6,7,8-tetrahydrobenzo[b] pyran (4e)



^{13}C NMR: 2-Amino-3-cyano-4-(4-methylphenyl)-7,7-dimethyl-5-oxo-4H-5,6,7,8-tetrahydrobenzo[b]pyran (4e)