

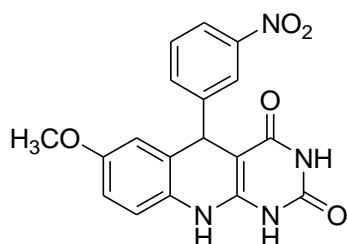
Core-shell magnetic mesoporous *N*-doped silica nanoparticles: a solid base catalyst for the preparation of some arylpyrimido[4,5-*b*]quinoline diones under green conditions

Shekofeh Neamani^a, Leila Moradi*^a, Mingxuan Sun^b

^a Department of Organic Chemistry, Faculty of Chemistry, University of Kashan, P.O. Box 8731753153, Kashan, I.R.Iran, (Email: L_moradi@kashanu.ac.ir)

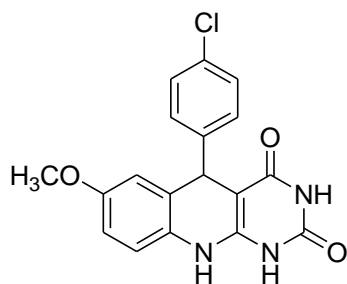
^b School of Materials Engineering, Shanghai University of Engineering Science, China

7-methoxy-5-(3-nitrophenyl)pyrimido[4,5-*b*]quinolin- 2,4 (1H, 3H, 5H, 10H) dione (4a)



White solid, mp: 234-238°C [24]. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3430, 3321, 2923, 1692, 1525, 1383, and 1248. ¹H NMR (DMSO-*d*₆, 400 MHz): δ (ppm): 10.49 (m, 3H, N-H), 7.27 (s, 1H, Ar-H), 6.90-6.92 (d, *J*= 8 Hz, 1H, Ar-H), 6.83-6.85 (d, *J*= 8 Hz, 1H, Ar-H), 3.58 (s, 3H, OCH₃).

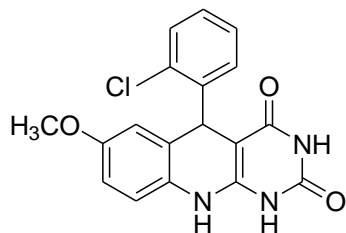
7-methoxy-5-(4-chlorophenyl)pyrimido[4,5-*b*]quinolin- 2,4 (1H, 3H, 5H, 10H) dione (4b)



Cream solid, mp: 180-184°C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3428, 3311, 2917, 1722, 1690, 1380, and 1241. ¹H NMR (DMSO-*d*₆, 400 MHz): δ (ppm): 10.33-10.54 (m, 3H, N-H), 7.44-7.46 (m, *J*= 8 Hz, 2H, Ar-H), 7.21-7.26 (m, 3H, Ar-H), 6.86-6.90 (d, *J*=12 Hz, 1H, Ar-H), 6.79-6.81 (d, *J*= 8 Hz, 1H, Ar-H), 6.10 (s, 1H), 3.57 (s, 3H, CH₃), ¹³CNMR (DMSO-*d*₆, 100 MHz): δ (ppm): 170.46, 166.124, 155.38, 153.72,

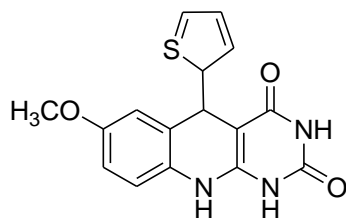
133.98, 132.48, 130.97, 130.77, 129.28, 129.19, 128.58, 126.91, 116.77, 114.82, 114.38, 113.10, 55.66, 44.19.

7-methoxy-5-(2-chlorophenyl)pyrimido[4,5-*b*] quinolin- 2,4 (1H, 3H, 5H, 10H) dione (4c)



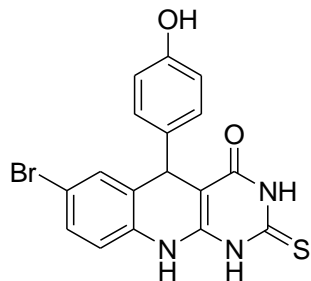
Pink solid, mp: 180-184°C. IR (KBr) (ν_{\max} / cm^{-1}): 3418, 3325, 2925, 1725, 1619, 1501, 1463 and 1237. ^1H NMR (DMSO- d_6 , 400 MHz): δ (ppm): 10.48 (s, 1H, N-H), 10.34 (s, 1H, N-H), 8.77 (s, 1H, NH), 7.33-7.35 (d, $J=8$ Hz, 1H, Ar-H), 7.12-7.20 (m, 3H, Ar-H), 6.94-6.97 (d, $J=12$ Hz, 1H, Ar-H), 6.70-6.72 (d, $J=8$ Hz, 2H, Ar-H), 5.53 (s, 1H). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ (ppm): 170.2, 160.50, 155.47, 153.62, 136.63, 134.65, 130.69, 130.30, 129.83, 129.68, 128.46, 128.14, 126.43, 117.56, 116.84, 113.81, 55.62, 52.83,

7-methoxy-5-(thiophen-2-yl)pyrimido[4,5-*b*] quinolin- 2,4 (1H, 3H, 5H, 10H) dione (4d)



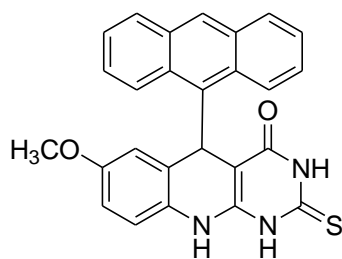
Pink solid, mp: 292-295°C. IR (KBr) (ν_{\max} / cm^{-1}): 3469, 3240, 3085, 2923, 2853, 1715, 1618, 1564, 1457 and 1233. ^1H NMR (DMSO- d_6 , 400 MHz): δ (ppm): 10.60 (s, 1H, N-H), 10.35 (s, 1H, N-H), 8.79 (s, 1H, NH), 7.17-7.18 (d, $J=4$ Hz, 1H, Ar-H), 6.96-6.98 (d, $J=8$ Hz, 1H, Ar-H), 6.89-6.90 (d, $J=4$ Hz, 1H, Ar-H), 6.77-6.82 (m, 3H, Ar-H), 5.32 (s, 1H), ^{13}C NMR (DMSO- d_6 , 100 MHz): δ (ppm): 163.127, 155.38, 155.78, 152.39, 150.61, 148.30, 129.29, 127.06, 126.54, 124.17, 123.37, 117.33, 114.70, 113.81, 84.82, 55.66, 54.52,

7-bromo-5-(4-hydroxy phenyl)-2-thioxo 2,3-dihydropyrimido[4,5-*b*] quinolin- 2,4 (1H, 3H, 5H, 10H) dione (4e)



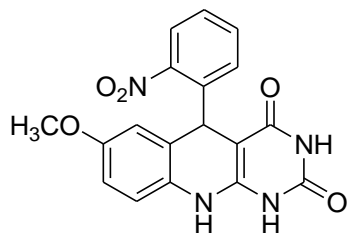
Yellow solid, mp: 330-332°C [36]. IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3443, 3217, 1689, 1653, 1517, 1443, and 1212. ^1H NMR (DMSO- d_6 , 400 MHz): δ (ppm): 12.34 (s, 1H, N-H), 11.55 (s, 1H, N-H), 10.98 (s, 1H, O-H), 8.36-8.38 (d, $J=8$ Hz, 2H, Ar-H), 8.21 (s, 1H, Ar-H), 7.73-7.76 (d, $J=8$ Hz, 1H, Ar-H), 6.90-6.92 (d, $J=8$ Hz, 2H, Ar-H), 6.53-6.55 (d, $J=8$ Hz, 1H, Ar-H), 5.83 (s, 1H).

5-(anthracene-10-yl)-7-methoxy-2-thioxo-2,3-dihydropyrimido[4,5-*b*] quinolin-4(1H, 5H, 10H) one (4f)



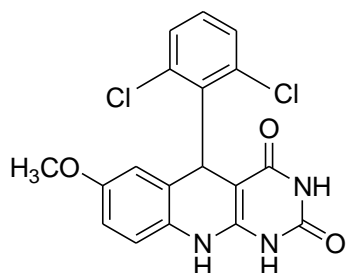
Pink solid, mp: 375-380°C [24]. IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3426, 3129, 2920, 1673, 1539, 1443, and 1314. ^1H NMR (DMSO- d_6 , 400 MHz): δ (ppm): 12.59 (s, 1H, N-H), 12.19 (s, 1H, N-H), 9.0 (s, 1H, Ar-H), 8.66 (s, 1H, Ar-H), 8.11-8.13 (d, $J=8$ Hz, 3H, Ar-H), 7.96-7.98 (d, $J=8$ Hz, 3H, Ar-H), 7.47-7.55 (m, 6H, Ar-H), 3.34 (s, 3H, CH_3).

7-methoxy-5-(2-nitrophenyl)pyrimido[4,5-*b*] quinolin-2,4(1H, 3H, 5H, 10H) dione (4g)



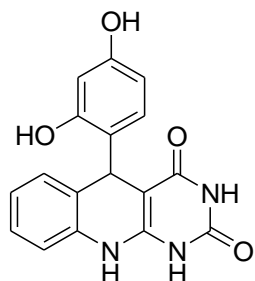
white solid, mp: 187-190°C [24]. IR (KBr) (ν_{\max} / cm^{-1}): 3517, 3318, 2999, 1655, 1618, 1519 and 1346. ^1H NMR (DMSO- d_6 , 400 MHz): δ (ppm): 10.46 (s, 1H, N-H), 10.40 (s, 1H, N-H), 8.87 (s, 1H, NH), 7.72-7.74 (d, $J=8$ Hz), 7.50-7.54 (t, $J=8$ Hz, 1H, Ar-H), 7.30-7.34 (t, $J=8$ Hz, 1H, Ar-H), 7.22-7.24 (d, $J=8$ Hz), 2H, Ar-H), 7.05-7.07 (d, 1H, Ar-H), 6.78-6.80 (d, $J=8$ Hz, 1H, Ar-H), 5.64 (s, 1H), 3.61 (s, 3H, CH_3).

7-methoxy-5-(2,6-dichlorophenyl)pyrimido[4,5-*b*]quinolin-2,4 (1H, 3H, 5H, 10H) dione (4h)



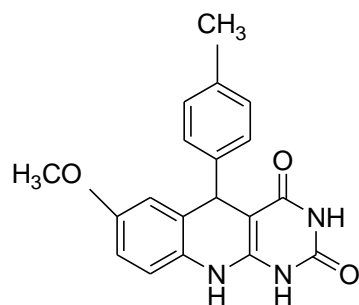
Pink solid, mp: 181-183°C [24]. IR (KBr) (ν_{\max} / cm^{-1}): 3347, 3073, 3000, 2930, 1695, 1643, 1561, 1463, and 1236. ^1H NMR (DMSO- d_6 , 400 MHz): δ (ppm): 10.37 (s, 1H, N-H), 10.25 (s, 1H, N-H), 8.83 (s, 1H, NH), 7.45-7.47 (d, $J=8$ Hz, 1H, Ar-H), 7.20-7.25 (m, 2H, Ar-H), 7.16-7.18 (d, 1H, $J=8$ Hz, 1H, Ar-H), 6.88-6.90 (d, $J=8$ Hz, 1H, Ar-H), 6.71-6.73 (d, $J=8$ Hz, 1H, Ar-H), 6.27 (s, 1H, Ar-H), 6.05 (s, 1H).

5-(2,4-dihydroxy phenyl)pyrimido[4,5-*b*]quinolin-2,4 (1H, 3H, 5H, 10H) dione (4i)

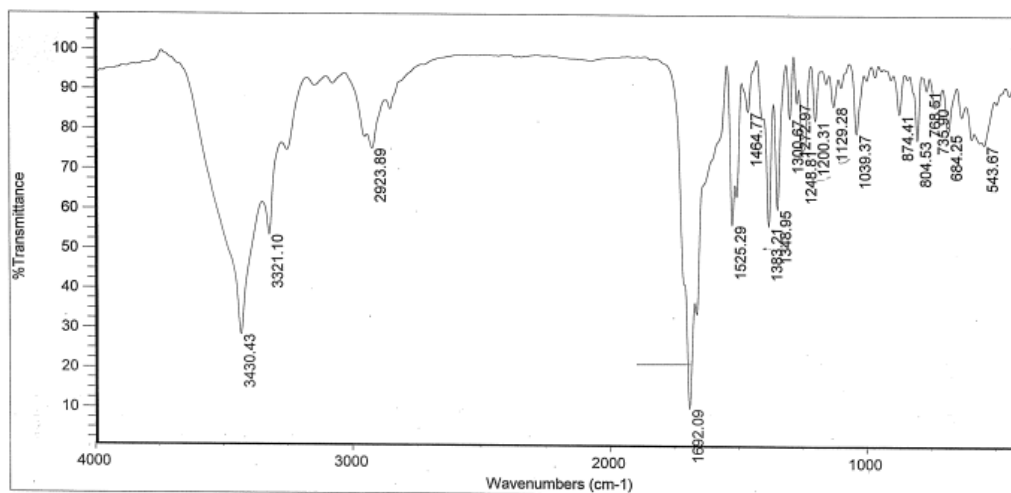


Yellow solid, mp: 200-204°C [37]. IR (KBr) (ν_{\max} / cm^{-1}): 3384, 3169, 3002, 2923, 1726, 1646, 1507, 1445, and 1241. ^1H NMR (DMSO- d_6 , 400 MHz): δ (ppm): 10.91 (s, 1H, N-H), 9.70 (s, 1H, N-H), 6.38-6.98 (m, 8H, Ar-H), 4.88 (s, 1H, O-H), 4.63 (s, 1H),

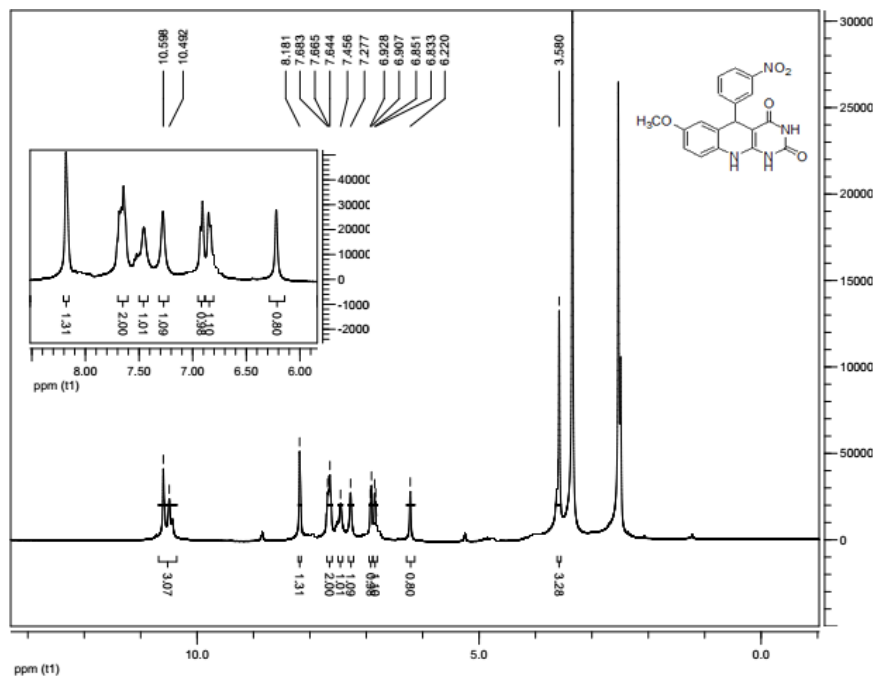
5-(4-methyl phenyl)pyrimido[4,5-*b*]quinolin-2,4 (1H, 3H, 5H, 10H) dione (4j)



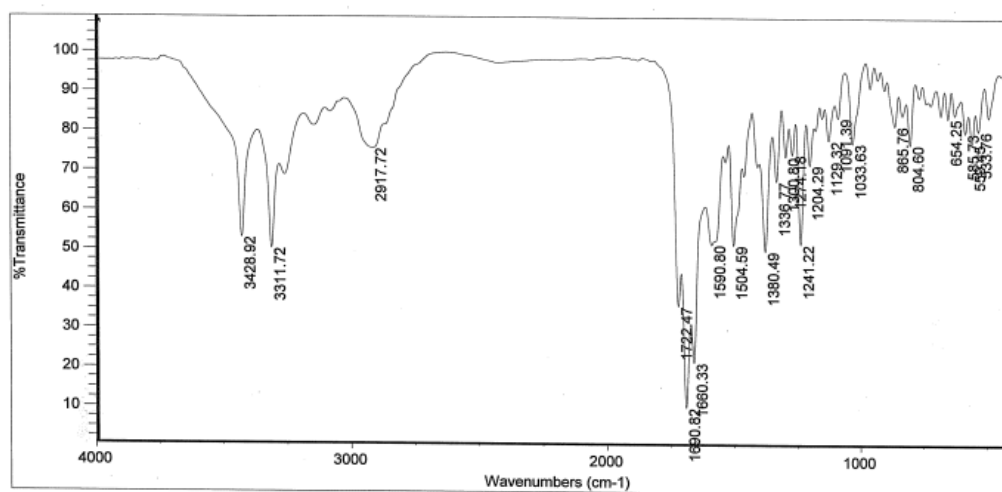
White solid, mp: 264-268°C [36]. IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3273, 3193, 1721, 1668, 1534, 1409, 1288 and 1185. ^1H NMR (DMSO- d_6 , 400 MHz): δ (ppm): 11.24 (s, 1H, N-H), 11.11 (s, 1H, N-H), 10.79 (s, 1H, NH), 8.30-8.33 (m, 3H, Ar-H), 8.19 (s, 1H, Ar-H), 6.85-6.87 (d, $J = 8$ Hz, 3H, Ar-H), 5.81 (s, 1H), 2.63 (s, 3H, CH_3).



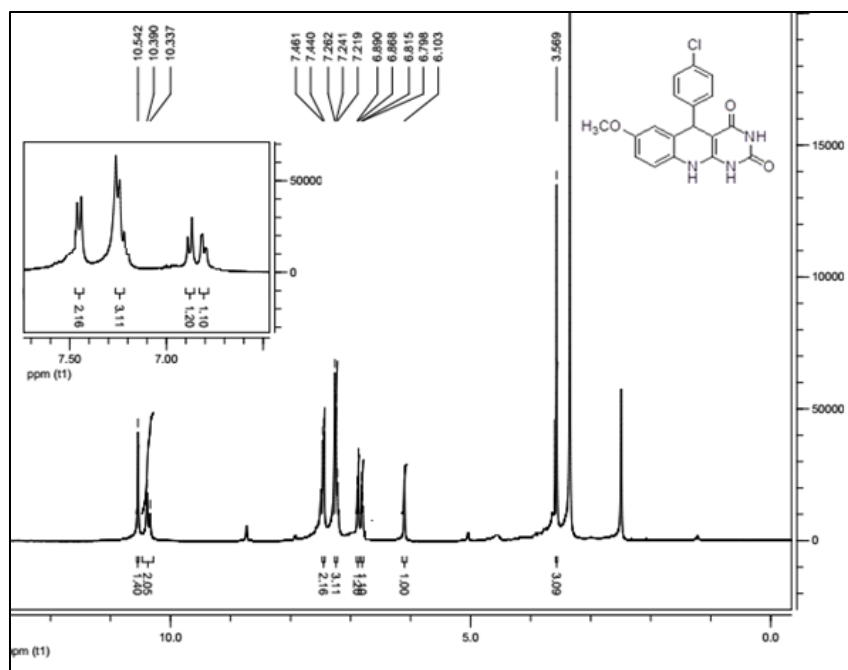
FTIR of 4a



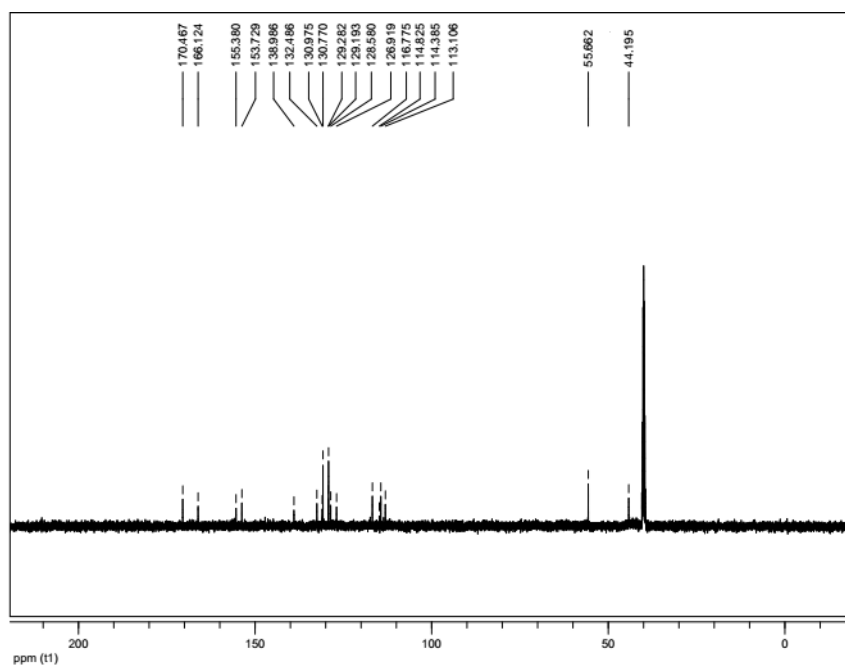
¹H NMR of 4a



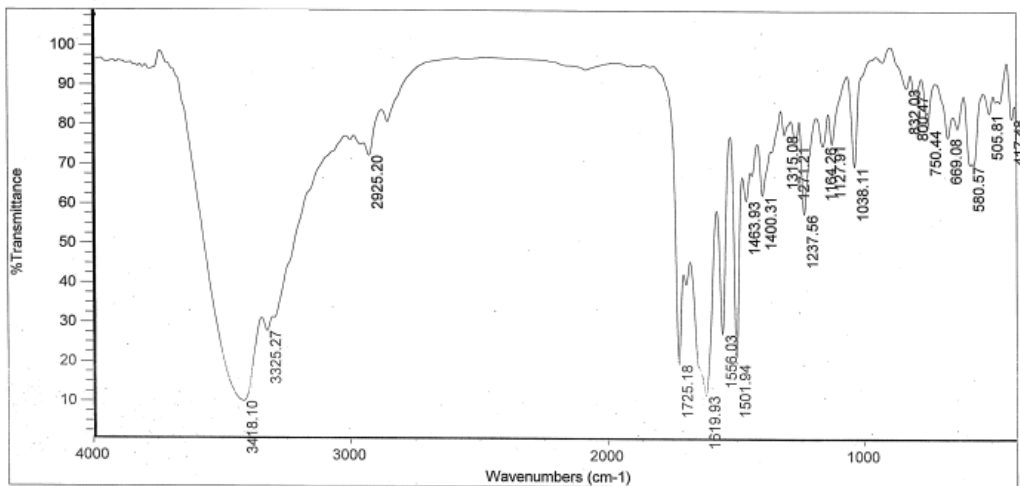
FTIR of 4b



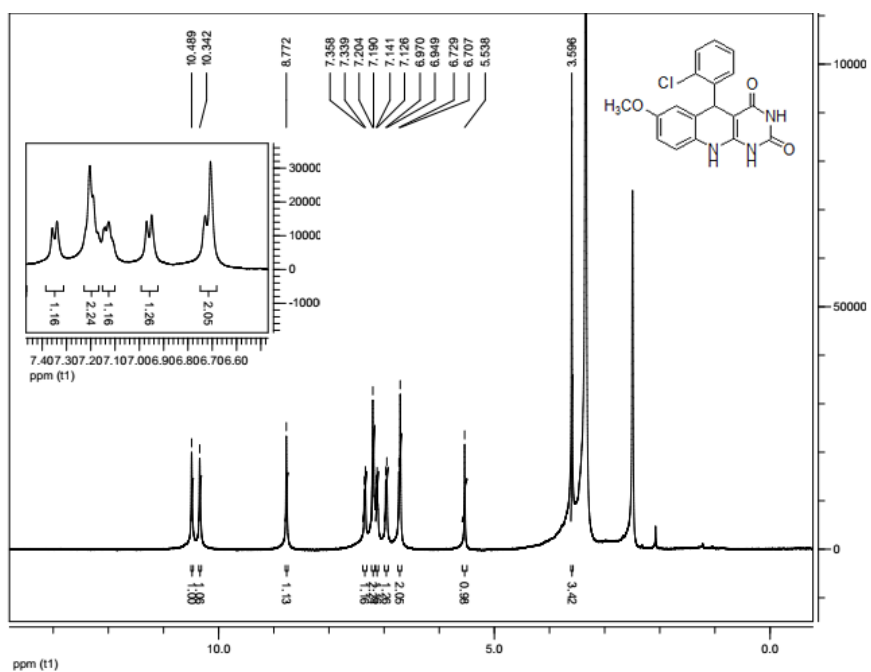
¹H NMR of 4b



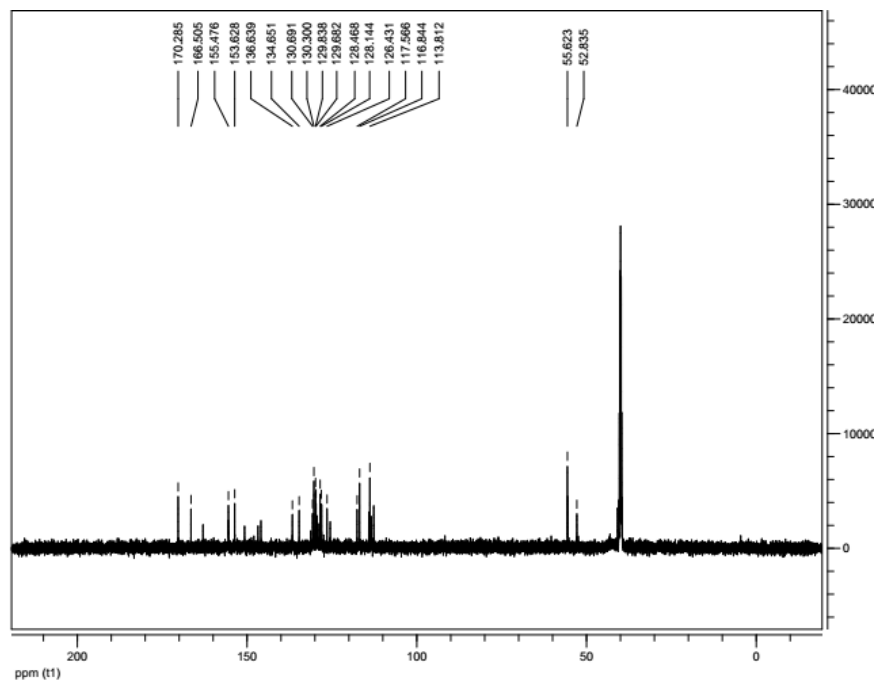
¹³C NMR of 4b



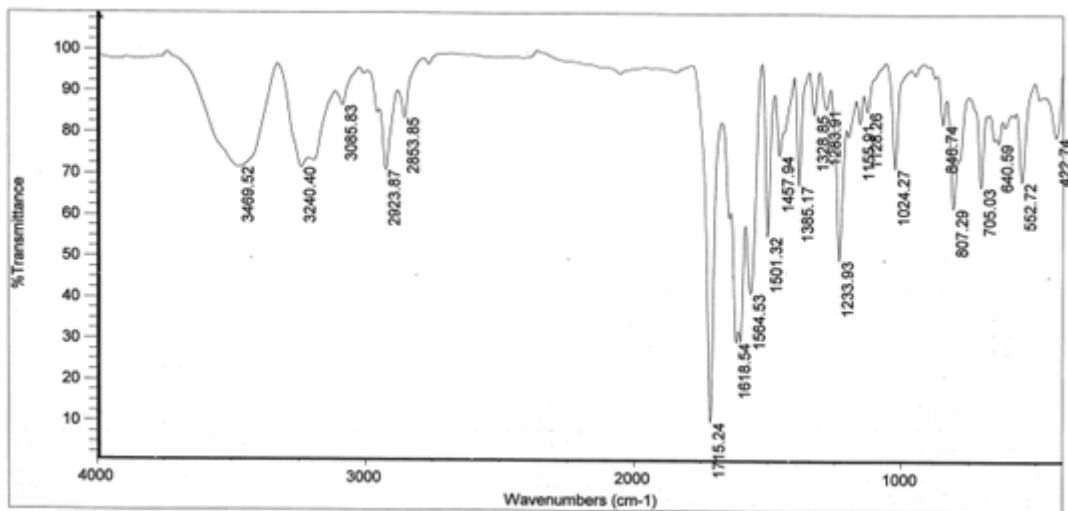
FTIR of 4c



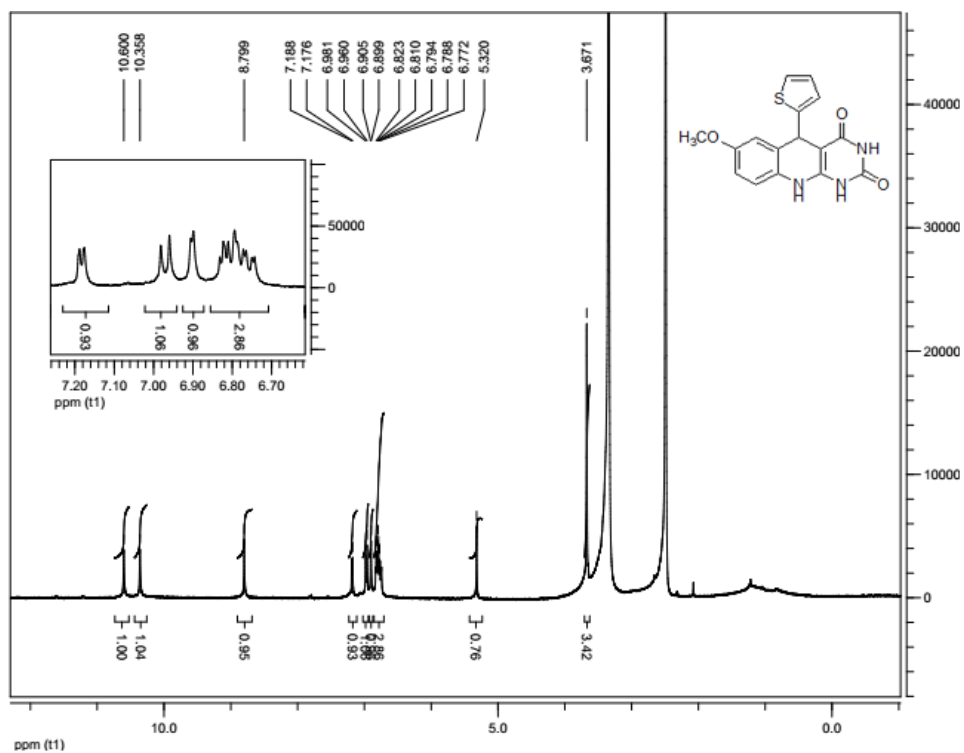
¹H NMR of 4c



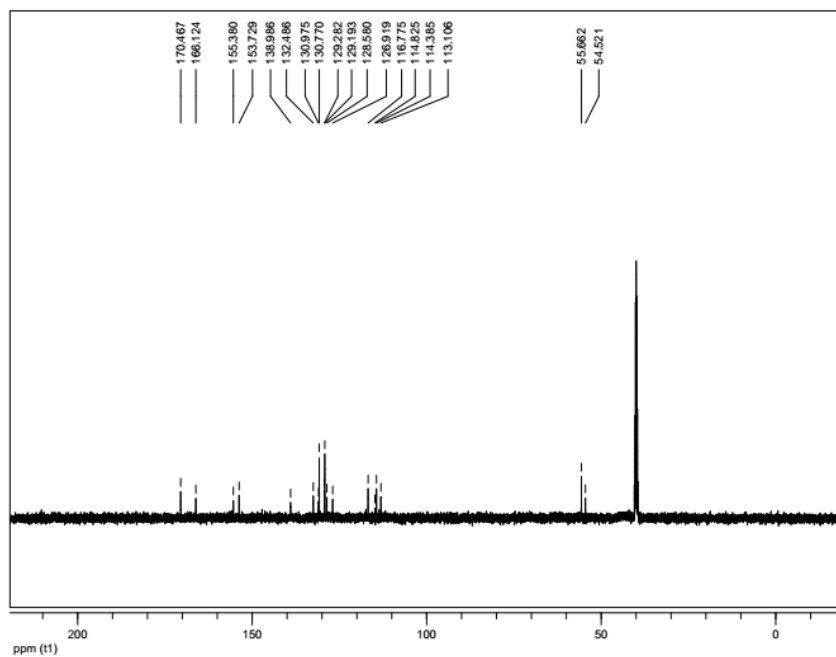
^{13}C NMR of 4c



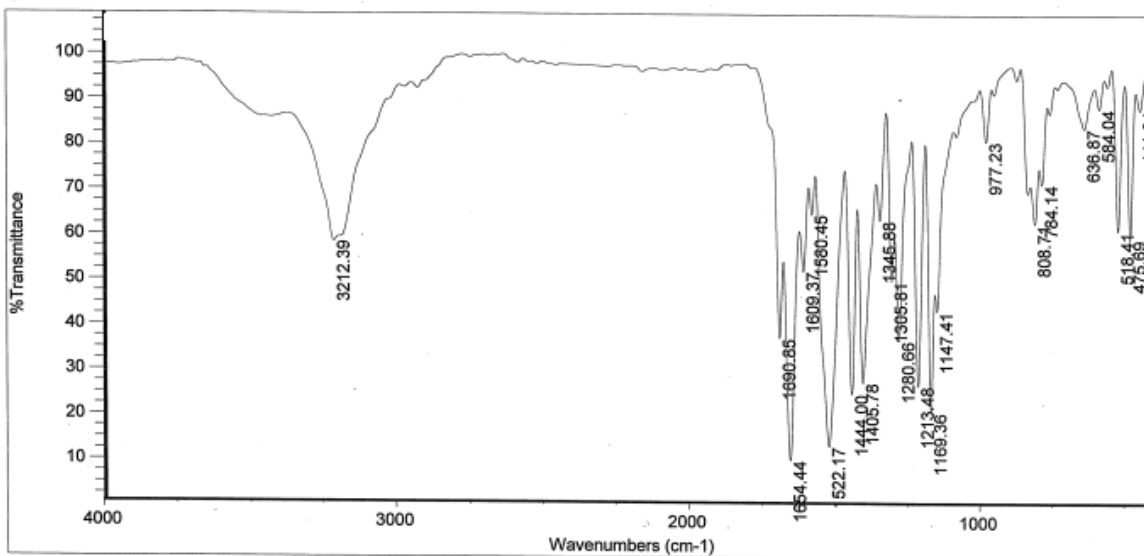
FTIR of 4d



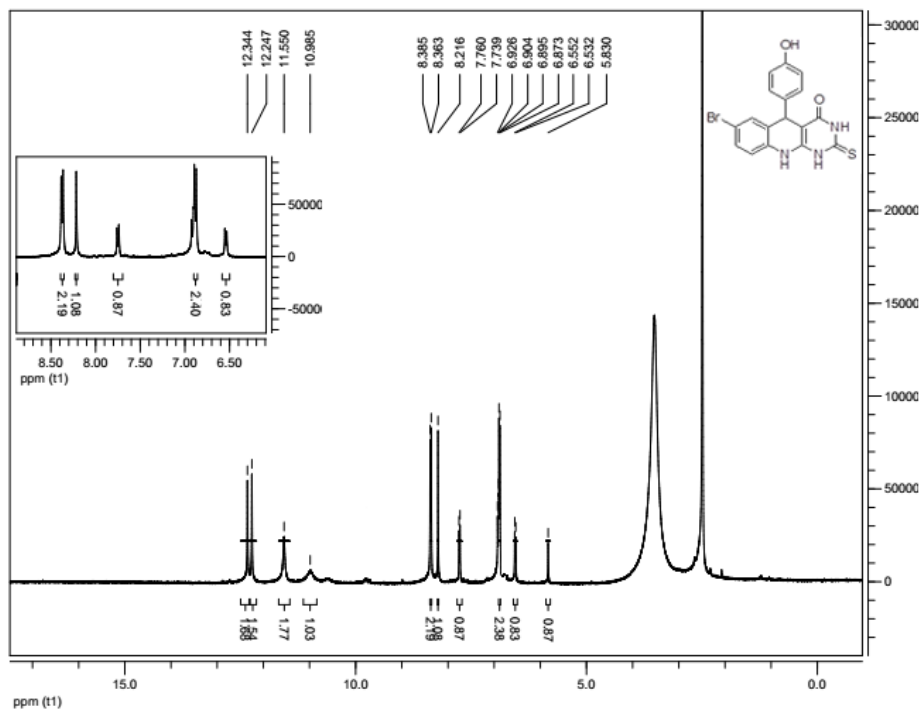
$^1\text{H NMR}$ of 4d



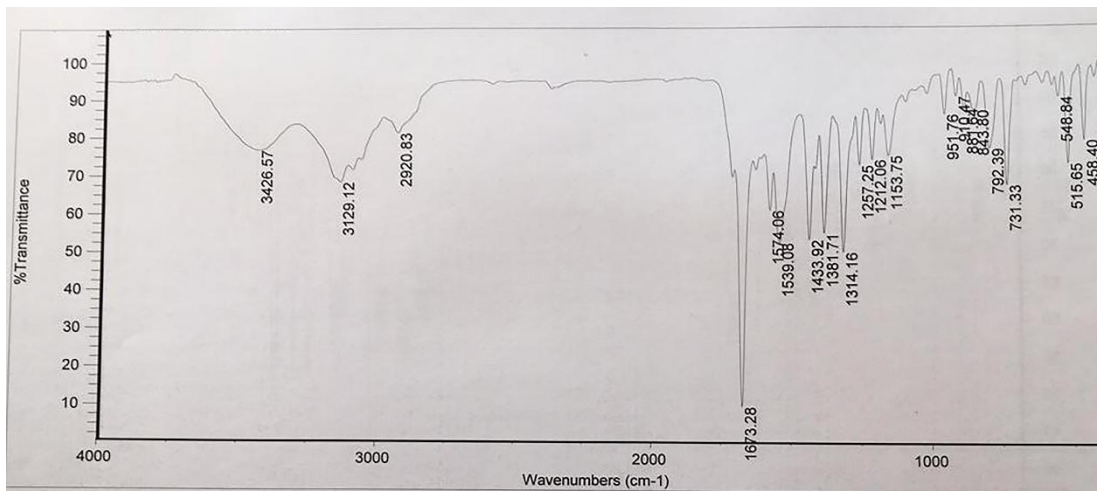
$^{13}\text{C NMR}$ of 4d



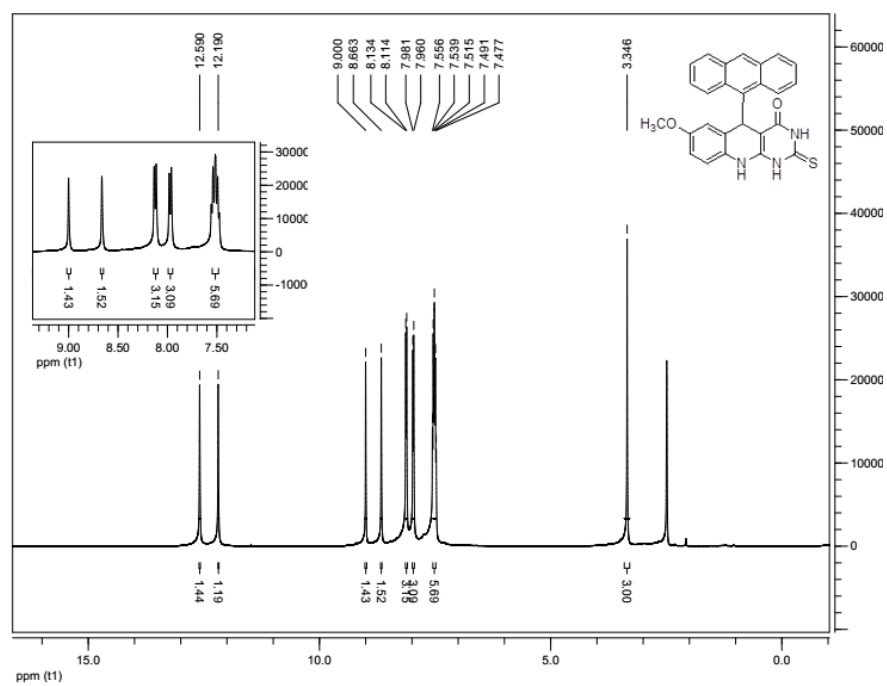
FTIR of 4e



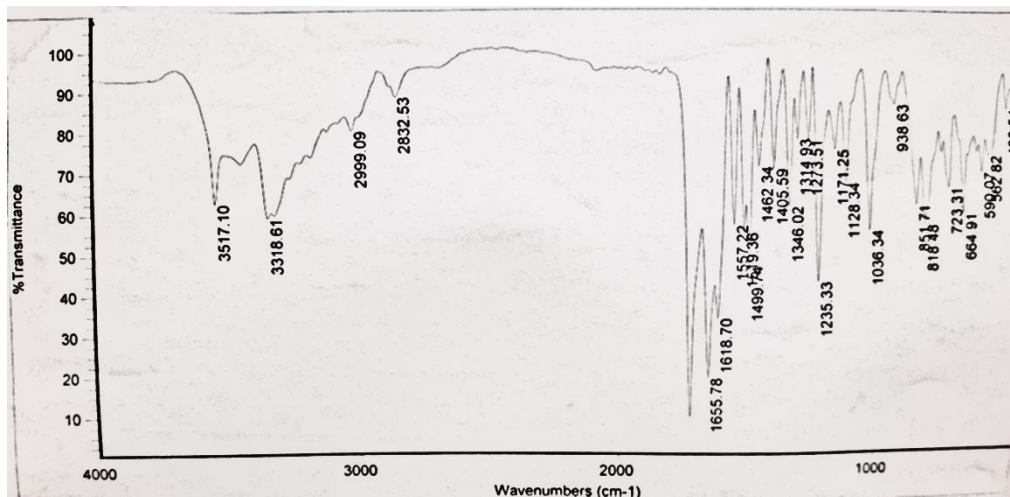
¹H NMR of 4e



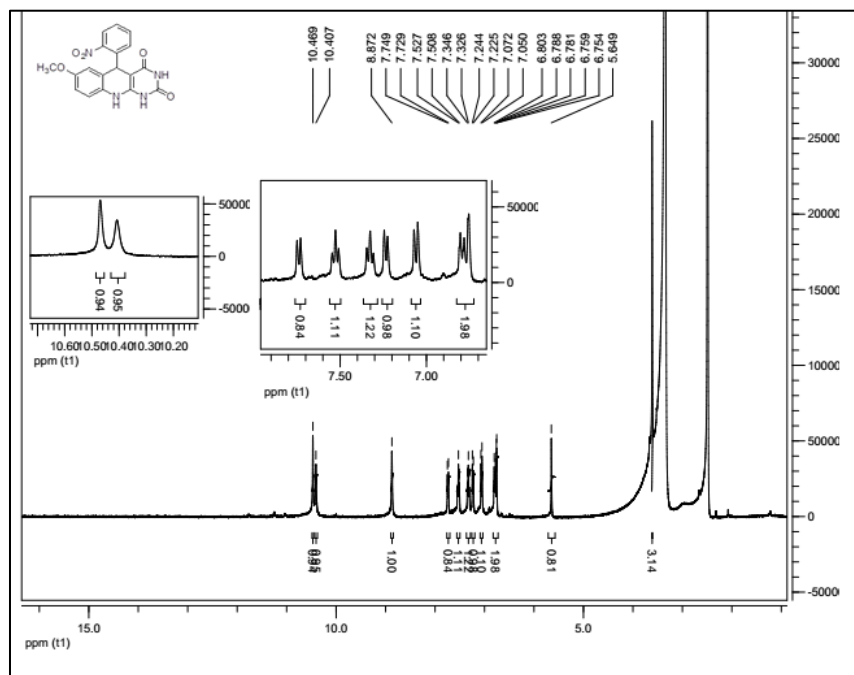
FTIR of 4f



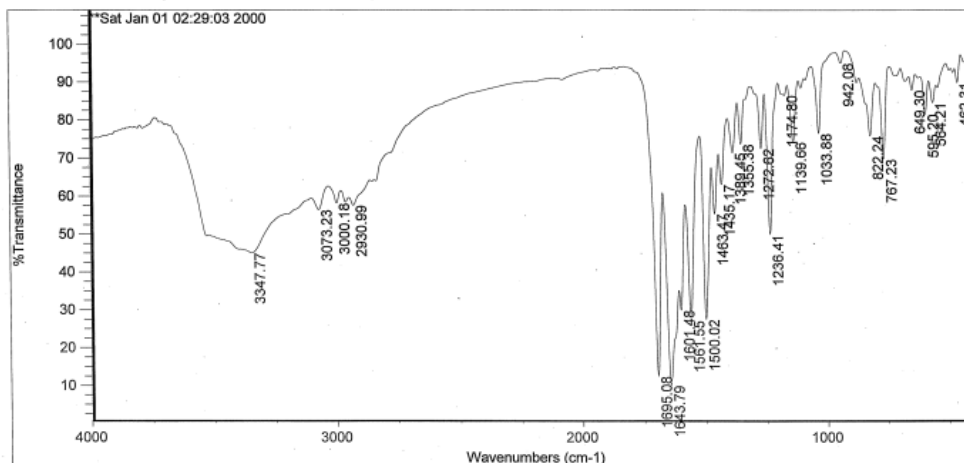
¹H NMR of 4f



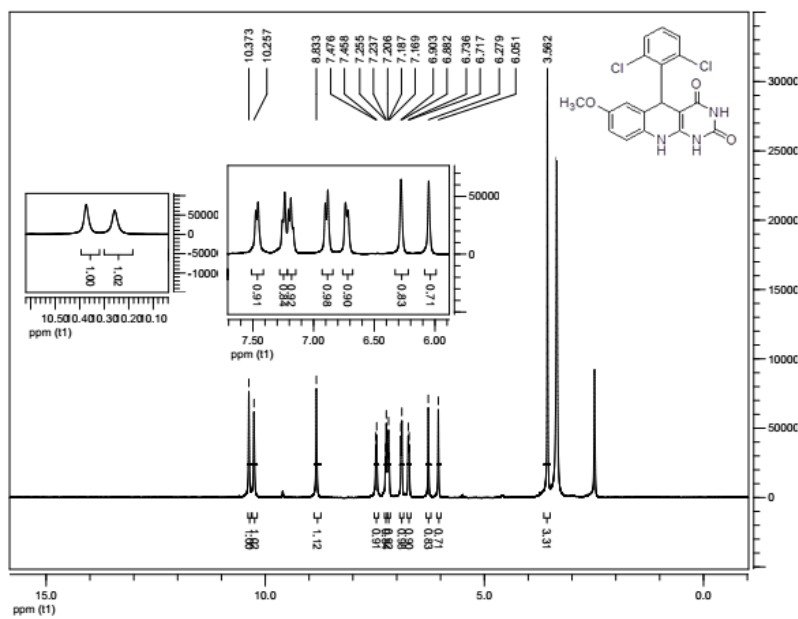
FTIR of 4g



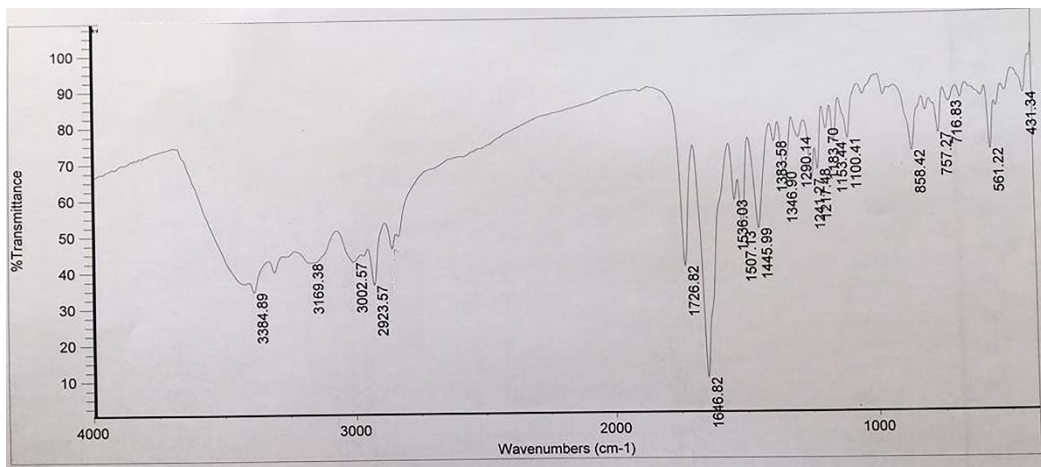
¹H NMR of 4g



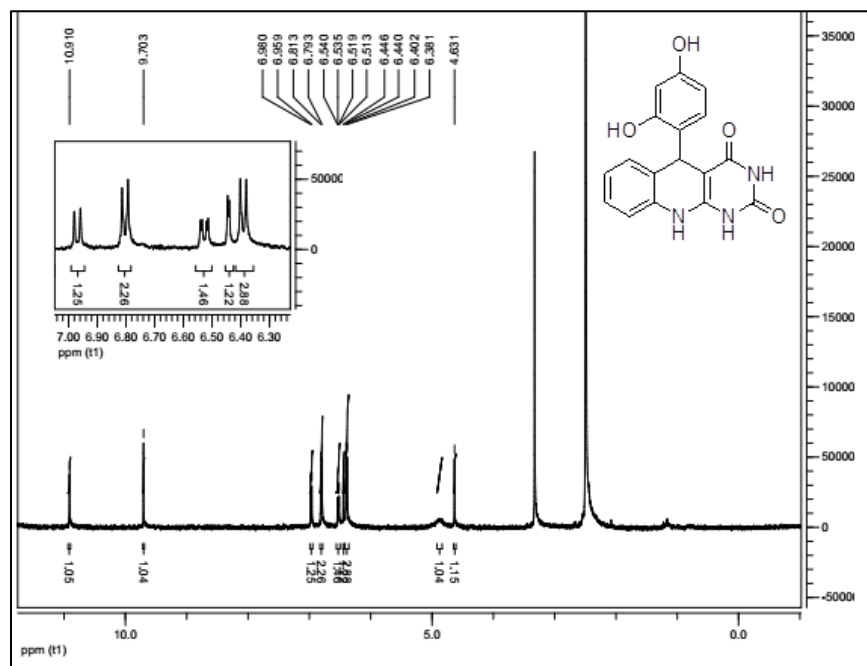
FTIR of 4h



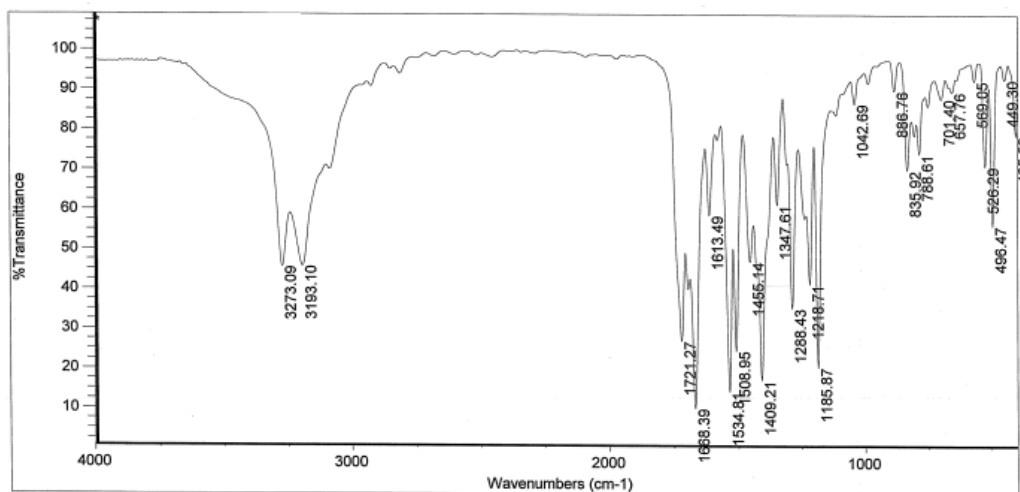
¹H NMR of 4h



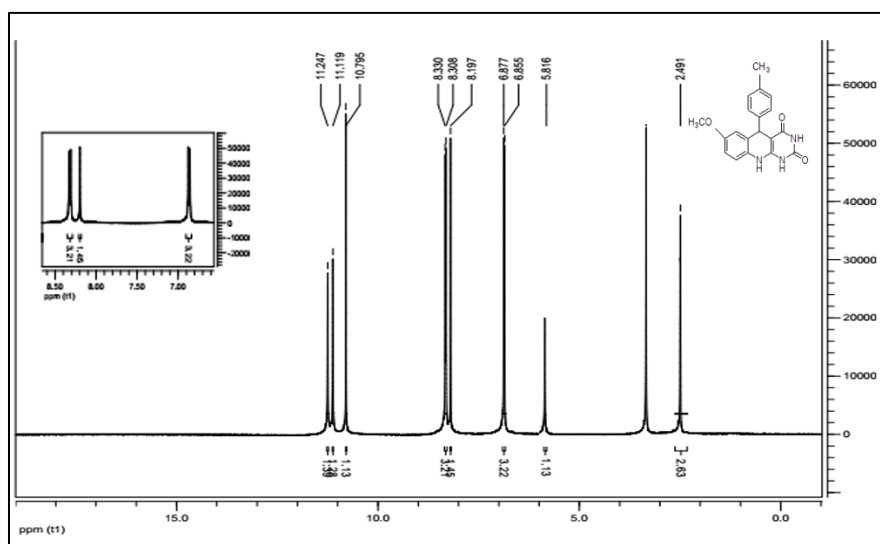
FTIR of 4i



¹H NMR of 4i



FTIR of 4j



¹H NMR of 4j