

Supplementary Materials

Hexamethylenetetramine-based ionic liquid/MIL-101(Cr) metal-organic framework composite: A novel and versatile tool for the preparation of pyrido[2,3-d:5,6-d']dipyrimidines

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5-(5-bromo-2-hydroxyphenyl)-7,9-dimethyl-8-thioxo-5,8,9,10-tetrahydropyrido[2,3-d:6,5-d']dipyrimidine-2,4,6(1H,3H,7H)-trione (4o): White solid; mp: >300 °C. ¹H NMR (250 MHz, DMSO-*d*₆) δ: 3.01 (s, 3H, CH₃), 3.66 (s, 3H, CH₃), 4.22 (s, 1H, CH), 6.96- 6.99 (d, 1H, J=7.4 Hz, ArH), 7.07 (s, 1H, ArH), 7.30-7.33 (d, 1H, J=7.8 Hz, ArH), 9.27 (s, 1H, OH), 10.40 (s, 1H, NH), 10.64 (s, 1H, NH), 11.58 (s, 1H, NH) ; ¹³C NMR (62.9 MHz, DMSO-*d*₆) δ: 27.49, 31.76, 50.39, 82.52, 97.14, 114.97, 118.79, 123.66, 129.29, 131.61, 135.34, 146.33, 150.33, 151.57, 152.85, 157.33, 162.33. FT-IR (KBr): 3365, 3172, 3051, 1705, 1674, 1591 cm⁻¹; Anal. Calcd. For C₁₇H₁₄BrN₅O₄S: C 50.80, H 3.62, Br 7.50, N 17.77, O 13.53, S 6.78. Found C 50.76, H 3.61, Br 7.53, N 17.74, O 13.46, S 6.64.; MS (EI) (m/z): 462.99 (M⁺).

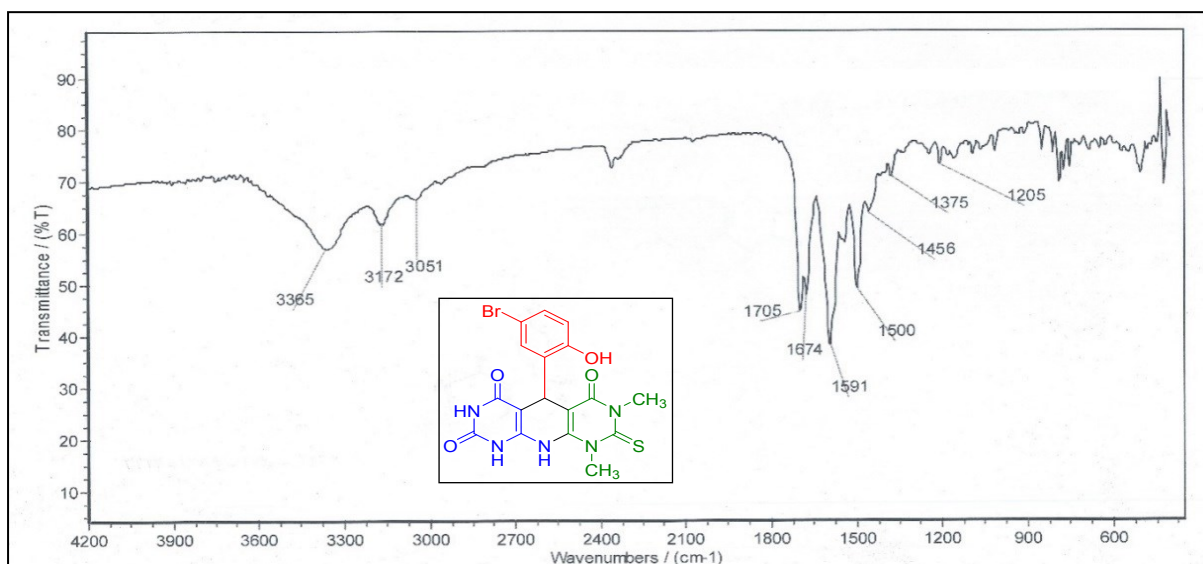


Figure S1: FT-IR spectra of 4o.

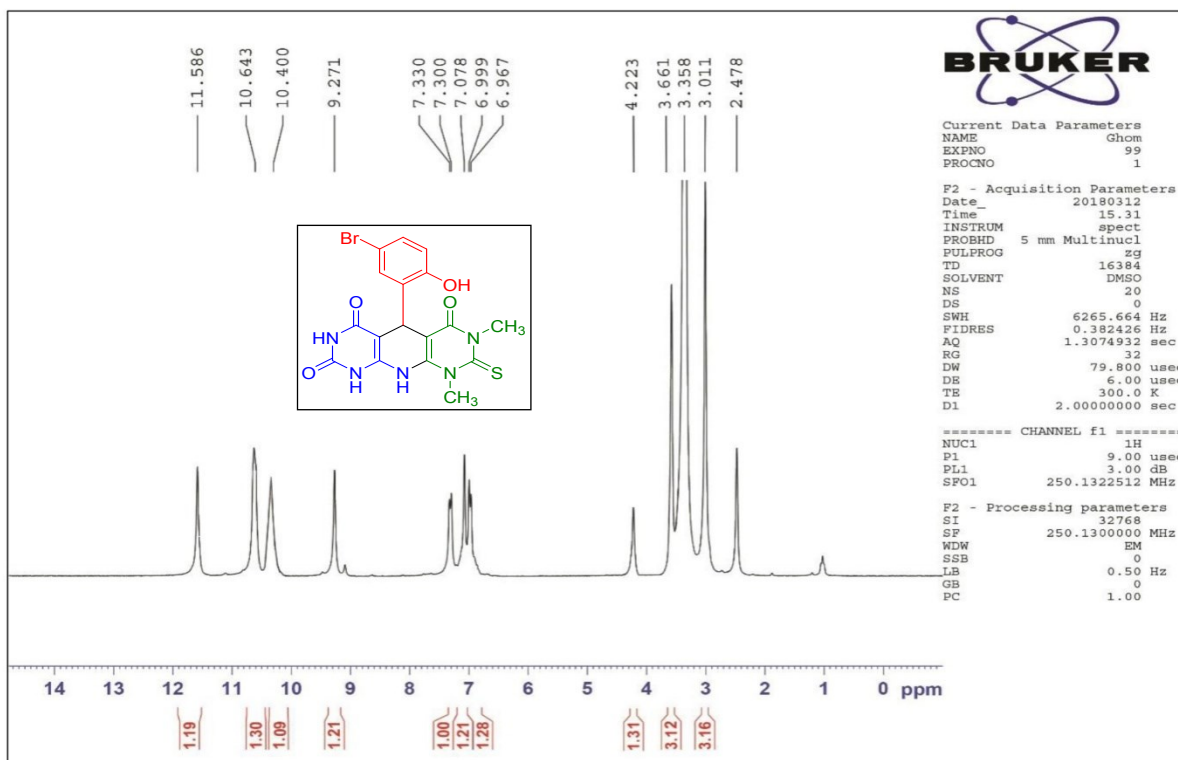


Figure S2: ¹H-NMR spectra of 4o.

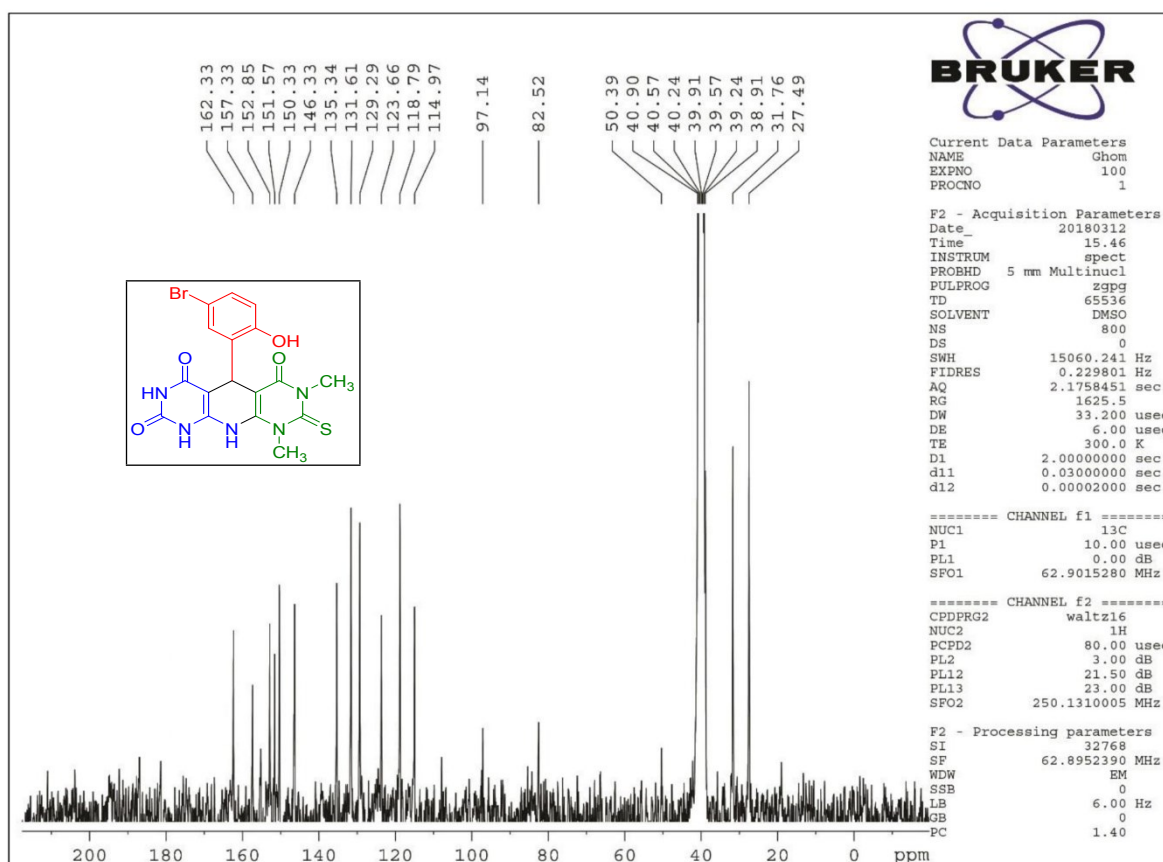


Figure S3: ¹³C-NMR spectra of 4o.

5-(2-hydroxy-5-nitrophenyl)-1,3-dimethyl-8-thioxo-5,8,9,10-tetrahydropyrido[2,3-d:6,5-d']dipyrimidine-2,4,6(1H,3H,7H)-trione (4p): White solid; mp: >300 °C. ¹H NMR (250 MHz, DMSO-*d*₆) δ: 3.09 (s, 3H, CH₃), 3.22 (s, 3H, CH₃), 4.13 (s, 1H, CH), 6.73 (d, 1H, J=7.4 Hz, ArH), 7.11(d, 1H, J=7.7 Hz, ArH), 7.19 (d, 1H, J=7.8 Hz, ArH), 9.12 (s, 1H, OH); 9.18 (s, 1H, NH), 10.28 (s, 1H, NH), 11.12 (s, 1H, NH); ¹³C NMR (62.9 MHz, DMSO-*d*₆) δ: 27.64, 33.24, 50.53, 82.52, 97.68, 116.51, 121.32, 123.53, 126.94, 128.65, 135.82, 146.86, 150.74, 151.57, 152.43, 157.24, 162.45. FT-IR (KBr): 3456, 3444, 3107, 2951, 1693, 1668, 1597cm⁻¹; Anal. Calcd. For C₁₇H₁₄N₆O₆S: C 47.44, H 3.28, N 19.53, O 22.30, S 7.45. Found C 47.42, H 3.31, N 19.51, O 22.32, S 7.42.; MS (EI) (m/z): 430.07 (M⁺).

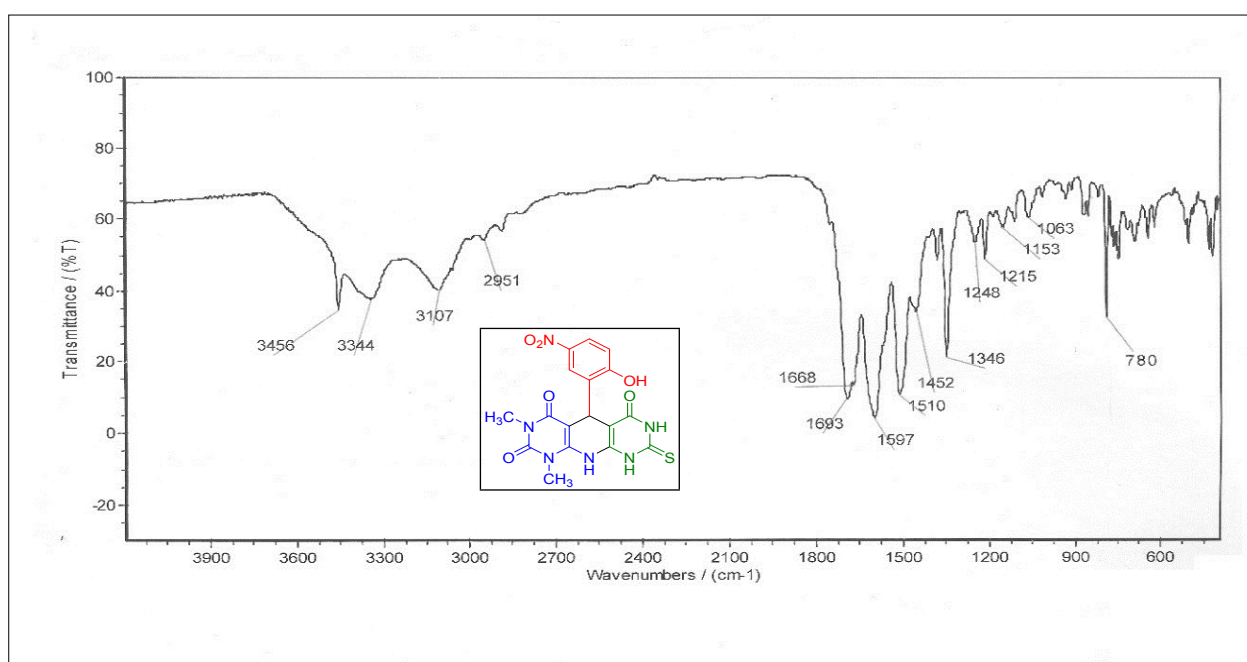


Figure S4: FT-IR spectra of 4p.

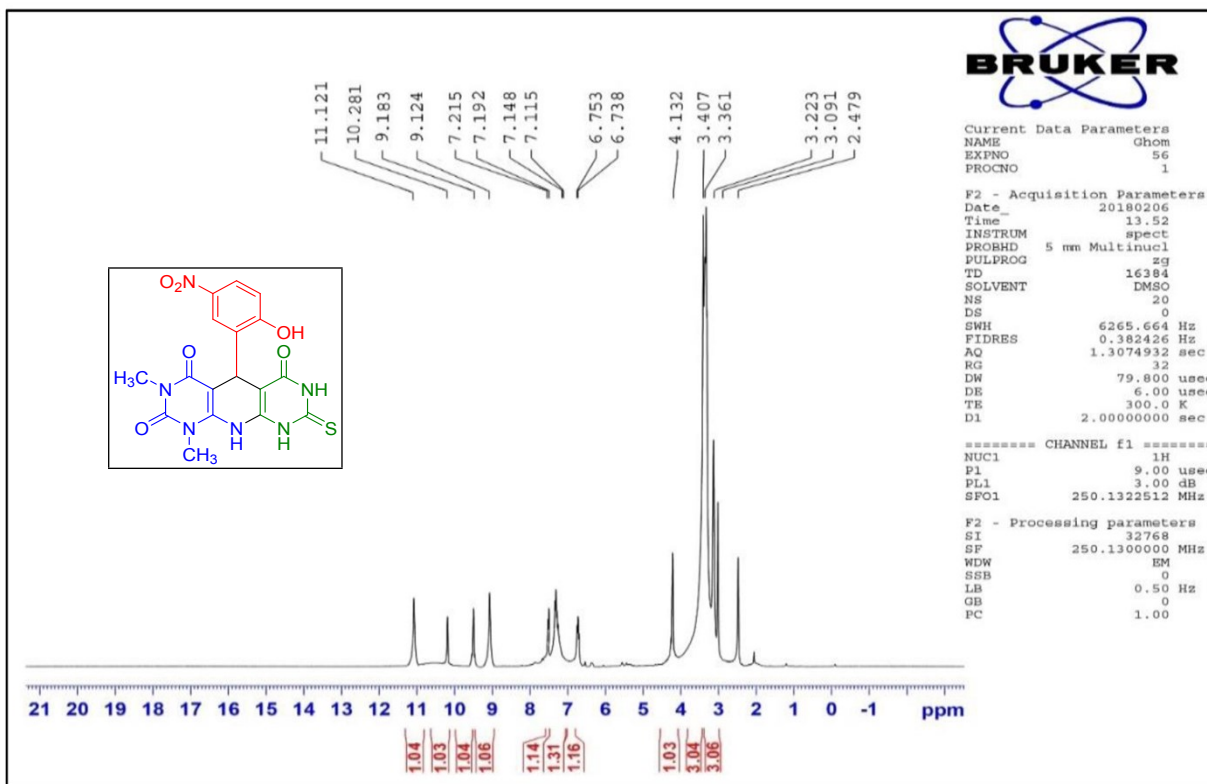


Figure S5: ^1H -NMR spectra of 4p.

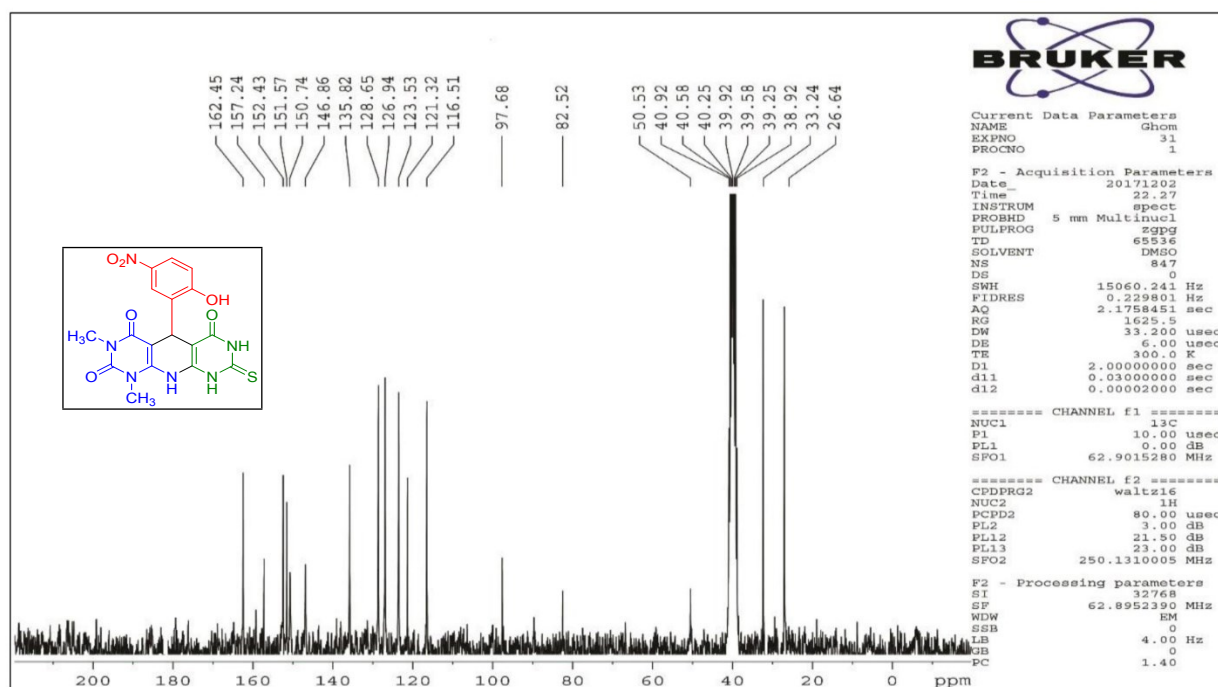


Figure S6: ^{13}C -NMR spectra of 4p.