

Solvent free synthesis of dihydropyrimidinone derivatives catalyzed by NiCoFe₂O₄ nanoparticles under thermal condition

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¹H NMR & FT-IR spectra of the synthesis compound

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Spectral data of products:

Ethoxycarbonyl-4-(4-phenyl)-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4a) White solid; Yield: 95%, MF: C₁₄H₁₆N₂O₃ MW: 260.29 mp: 202-204°C, mp (Lit. [27]: 201-203°C); IR (KBr) $\nu(\text{cm}^{-1})$: 3243, 3114, 2977, 1723, 1711, 1646, 1421, 1222, 1091. ¹H NMR (400 MHz, DMSO) δ (ppm): 1.08 (t, J= 13.2 Hz, 3H), 2.24 (s, 3H), 3.99 (q, 2H, CH₂), 5.14 (s, 1H), 7.28-7.30 (m, 5H, ArH), 7.73 (1H, NH), 9.19 (s, 1H, NH).

Ethoxycarbonyl-4-[(4-methyl) phenyl]-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4b) Yellow solid, Yield: 94%, MF: C₁₅H₁₈N₂O₃ MW: 274.13 mp: 211-213 °C, mp (Lit. [28]: 215-216°C); IR (KBr) $\nu(\text{cm}^{-1})$: 3260, 2371, 1736, 1485, 1219, 1079. ¹H NMR (400 MHz, DMSO) δ (ppm): 1.08 (t, J= 7.2 Hz, 3H, CH₃), 2.22 (s, 3H, CH₃), 2.24 (s, 3H, CH₃), 3.95 (q, 2H, CH₂), 5.08 (s, 1H, CH), 7.10 (q, 4H, ArH), 7.68 (s, 1H, NH), 9.15 (s, 1H, NH).

Ethoxycarbonyl-4-[(2,4-dichloro)phenyl]-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4c) White solid, Yield: 95% , MF: C₁₄H₁₅Cl₂N₂O₃ MW: 329.18 mp: 243-244°C, mp (Lit. [28] 251-252°C); IR (KBr) $\nu(\text{cm}^{-1})$: 3358, 3221, 2974, 1645, 1558, 1465. ¹H NMR (400 MHz, DMSO) δ (ppm): 0.98 (t, J= 6.8 Hz, 3H, CH₃), 2.28 (s, 3H, CH₃), 3.89 (q, 2H, CH₂), 5.58 (s, 1H, CH), 7.29 (d, J= 8.4 Hz, 1H, Ar-H), 7.41 (d, J= 8.4 Hz, 1H, Ar-H), 7.54 (s, 1H, NH), 7.74 (s, 1H, Ar-H), 9.31 (1H, NH).

Ethoxycarbonyl-4-[(4-methoxy) phenyl]-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4d) White solid, Yield: 94%, MF: C₁₅H₁₈N₂O₄ MW: 290.31 mp: 202-204 °C, mp (Lit. [29]: 203-205°C); IR (KBr) $\nu(\text{cm}^{-1})$: 3242, 3111, 2954, 1705, 1648, 1512, 1222. ¹H NMR (400 MHz, DMSO) δ (ppm): 1.01 (t, J= 13.2 Hz, 3H, CH₃), 2.22 (s, 3H, CH₃), 2.49 (s, 3H, CH₃), 3.66 (q,

2H, CH₂), 3.97 (s, 3H, OCH₃), 5.07 (s, 1H), 7.12 (d, J= 8.4 Hz, 2H, Ar-H) 7.14 (d, J = 9.6 Hz, 2H), 7.67 (1H, NH), 9.15 (1H, NH).

Ethoxycarbonyl-4-(3-nitrophenyl)-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4e) White solid, Yield: 97%, MF: C₁₄H₁₅N₃O₅ MW: 305.10 mp: 209-210 °C, mp (Lit. [27]: 205-207 °C); IR (KBr) $\nu(\text{cm}^{-1})$: 3350, 3229, 2977, 1712, 1644, 1534, 1455. ¹H NMR (400 MHz, DMSO) δ (ppm): 1.08 (t, J= 7.0 Hz, 3H, CH₃), 2.26 (s, 3H, CH₃), 3.99 (q, 2H, CH₂), 5.30 (s, 1H, CH), 7.64 (d, J= 8.0 Hz, 2H, Ar-H), 8.08 (s, H, NH), 8.11 (d, J= 8.0 Hz, 2H, Ar-H), 9.37 (s, 1H, NH).

Ethoxycarbonyl-4-(4-nitrophenyl)-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4f) White solid, Yield: 98%, MF: C₁₄H₁₅N₃O₅ MW: 305.10 mp °C: 218-219, mp (Lit. [27]: 223-225⁰C); IR (KBr) $\nu(\text{cm}^{-1})$: 3234, 3118, 2981, 1702, 1728, 1643, 1520, 1462. ¹H NMR (400 MHz, DMSO) δ (ppm): 1.081 (t, J= 14.0 Hz, 3H, CH₃), 2.25 (s, 3H, CH₃), 3.98 (q, 2H, CH₂), 5.26 (s, 1H, CH), 7.50 (d, J= 8.0 Hz, 2H, Ar-H), 7.89 (s, H, NH), 8.19 (d, J= 6.0 Hz, 2H, Ar-H), 9.35 (s, 1H, NH).

Ethoxycarbonyl-4-[(4-hydroxy)phenyl]-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4g) White solid, Yield: 93%, MF: C₁₄H₁₆N₂O₄ MW: 276.28 mp: 237-238 °C, mp (Lit. [30]: 236-238°C); IR (KBr) $\nu(\text{cm}^{-1})$: 3285, 2981, 1706, 1513, 1458, 1230. ¹H NMR (400 MHz, DMSO) δ (ppm): 1.07 (t, J= 12.0 Hz, 3H, CH₃), 2.21 (s, 3H, CH₃), 3.97 (q, 2H, CH₂), 5.02 (s, 1H, CH), 6.66 (d, 2H, J= 8.0 Hz, ArH), 7.00 (d, 2H, J= 6.0 Hz, ArH), 7.62 (s, 1H, NH), 9.09 (s, 1H, NH), 10.24 (s, 1H, OH).

Ethoxycarbonyl-4-[(4-chloro)phenyl]-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4h) White solid, Yield: 96%, MF: C₁₄H₁₅ClN₂O₃ MW: 294.73 mp: 214-216°C, mp (Lit. [29]: 210-212°C); IR (KBr) $\nu(\text{cm}^{-1})$: 3259, 2981, 1743, 1487, 1232. ¹H NMR (400 MHz, DMSO) δ (ppm):

1.07 (t, J= 14.0 Hz, CH₃), 2.24 (s, 3H, CH₃), 3.97 (q, 2H, CH₂), 5.13 (s, 1H, CH), 7.22 (d, J = 8.0 Hz, 2H, Ar-H), 7.38 (s, J = 8.4 Hz, 2H, NH), 7.76 (s, 1H, NH), 9.24 (s, 1H, NH).

Ethoxycarbonyl-4-[(4-flouro) phenyl]-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4i)

Yellow solid, Yield: 96%, MF: C₁₄H₁₅FN₂O₃ MW: 278.28 mp: 156-157°C, mp (Lit. [29]: 152-153°C); IR(KBr) ν (cm⁻¹): 3348, 323, 2975, 1455, 1223, 1099. ¹H NMR (400 MHz, DMSO) δ (ppm): 1.07 (t, J= 13.6 Hz, 3H, CH₃), 2.23 (s, 3H, CH₃), 3.95 (q, 2H, CH₂), 5.13 (s, 1H, CH), 7.13 (d, J= 8.4 Hz 4H), 7.24 (J= 7.2 Hz d), 7.73 (s, 1H, NH), 9.21 (1H, NH).

Ethoxycarbonyl-4-[(4-phenyl)-6-methyl-3,4-dihydropyrimidine-2(1H)-thione (4j) White

solid, Yield: 92%, MF: C₁₄H₁₆N₂O₃, MW: 276.35 mp: 202-204°C: mp (Lit. [29]: 201-203°C; IR (KBr) ν (cm⁻¹): 3184, 3026, 2925, 1691, 1569, 1457. ¹H NMR (400 MHz, DMSO) δ (ppm): 1.07 (t, J= 6.0 Hz, 3H), 2.36 (s, 3H), 3.97 (q, 2H, CH₂), 5.16 (s, 1H), 7.20 (m, 5H, ArH), 9.19 (s, 1H, NH) 10.33 (s, 1H, NH).

Ethoxycarbonyl-4-[(4-hydroxy-phenyl)-6-methyl-3,4-dihydropyrimidine-2(1H)-thione (4k)

White solid, Yield: 91%, MW: C₁₄H₁₆N₂O₃S, MW: 292.34 mp: 198-199°C, mp (Lit. [30]: 202-203°C); IR (KBr) ν (cm⁻¹): 3499, 3187, 3016, 1687, 1577, 1455. ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 1.07 (t, J= 6.8 Hz, 3H, CH₃), 2.24 (s, 3H, CH₃), 3.97 (q, 2H, CH₂), 5.05 (s, 1H, CH), 6.68 (d, 2H, J= 8.0 Hz, ArH), 6.70, (d, 2H, J= 8.0 Hz, ArH) ,7.06 (s, 1H, NH), 9.53 (s, 1H, NH), 10.22 (1H, OH).

Ethoxycarbonyl-4-[(2,4-dichloro-phenyl)-6-methyl-3,4-dihydropyrimidine-2(1H)-thione (4l)

White solid, Yield: 93%, MF: C₁₄H₁₄Cl₂N₂O₂S, MW: 345.24 mp: 249-251°C, mp (Lit. [31]: 241-243°C) IR(KBr) ν (cm⁻¹): 3188, 2980, 1706, 1649, 1565, 1466, 1232. ¹H NMR (400 MHz, DMSO) δ (ppm): 1.00 (t, J= 11.6 Hz, 3H, CH₃), 2.28 (s, 3H, CH₃), 3.90 (q, 2H, CH₂), 5.59 (s,

1H, CH), 7.28 (d, J= 8.0 Hz, 1H, Ar-H), 7.41 (d, J= 8.4 Hz, 1H, Ar-H), 7.74 (s, 1H, Ar-H), 9.62 (1H, NH), 10.43 (1H, NH).

Ethoxycarbonyl-4-[(2-chloro)phenyl]-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4m)

White solid, Yield: 92%, MF: C₁₄H₁₅ClN₂O₃ MW: 294.73 mp: 256-258°C, mp (Lit. [32]: 252-253°C); IR (KBr) $\nu(\text{cm}^{-1})$: 3352, 3112, 2975, 1696, 1450, 1227. ¹H NMR (400 MHz, DMSO) δ (ppm): 0.97 (t, J= 14.0 Hz, 3H, CH₃), 2.28 (s, 3H, CH₃), 3.88 (q, 2H, CH₂), 5.61 (s, 1H, CH), 7.24-7.38 (m, 3H, Ar-H), 7.39 (d, J= 7.6 Hz), 7.70 (s, 1H, NH), 9.27 (s, 1H, NH).

Ethoxycarbonyl-4-[(2-nitro)phenyl]-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4n) yellow

solid, Yield: 94%, MF: C₁₅H₁₈N₃O₅ MW: 305.29, mp: 213-214°C: mp (Lit. [33]: 208-210°C); IR (KBr) $\nu(\text{cm}^{-1})$: 3364, 3236, 3125, 2980, 1703, 1642, 1524, 1449, 1224. ¹H NMR (400 MHz DMSO) δ (ppm): 0.90 (t, J= 7.0 Hz, 3H, CH₃), 2.27 (s, 3H, CH₃), 3.84 (q, 2H, CH₂), 5.80 (s, 1H, CH), 7.48-7.56 (m, 3H, Ar-H), 7.87 (d, J= 8.0 Hz) 8.21 (s, 1H, NH), 9.40 (s, 1H, NH).

Ethoxycarbonyl-4-(4-phenyl)-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4a)

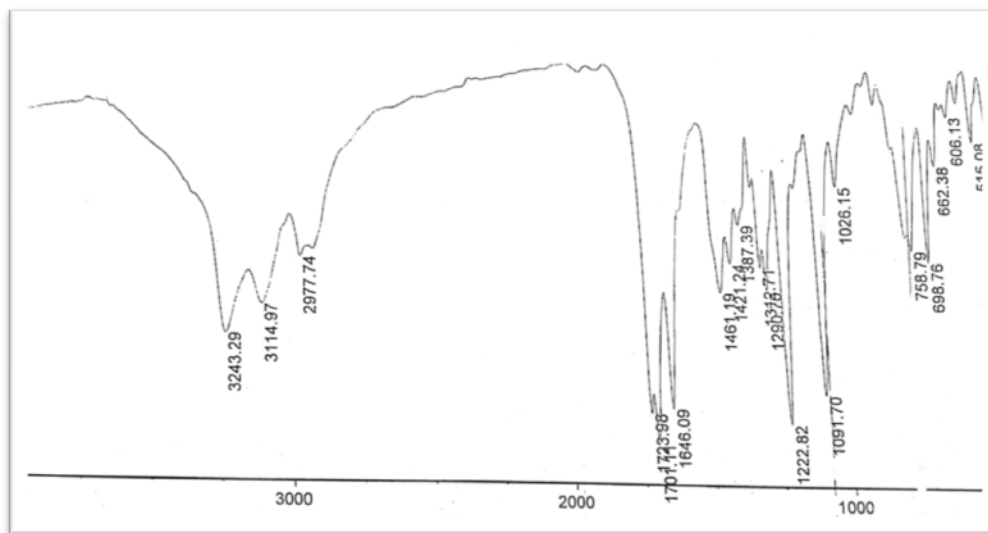


Figure S1. FT-IR spectrum of **4a**

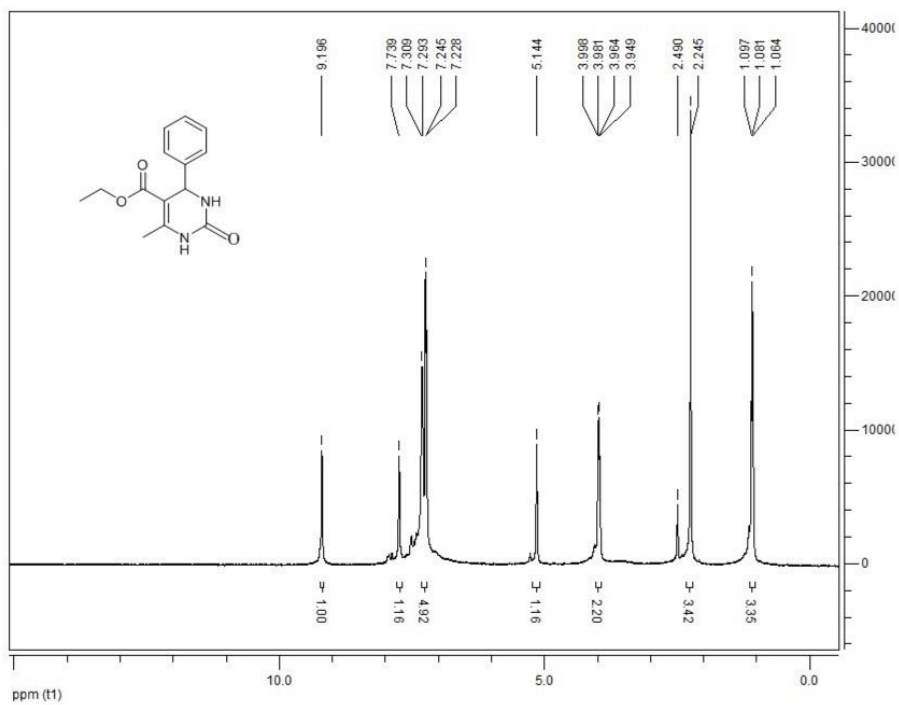


Figure S2. ¹H NMR spectrum of **4a**

Ethoxycarbonyl-4-[(4-methyl)phenyl]-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4b)

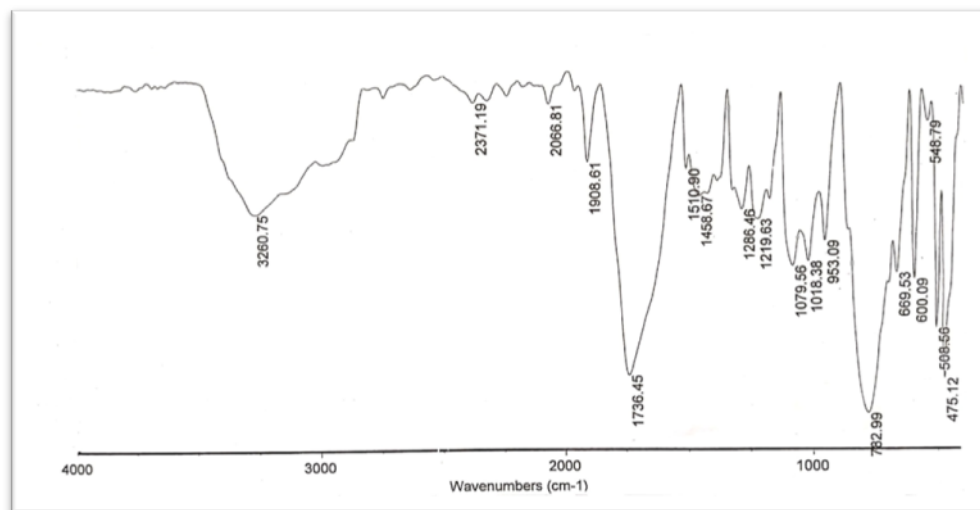


Figure S3. FT-IR spectrum of **4b**

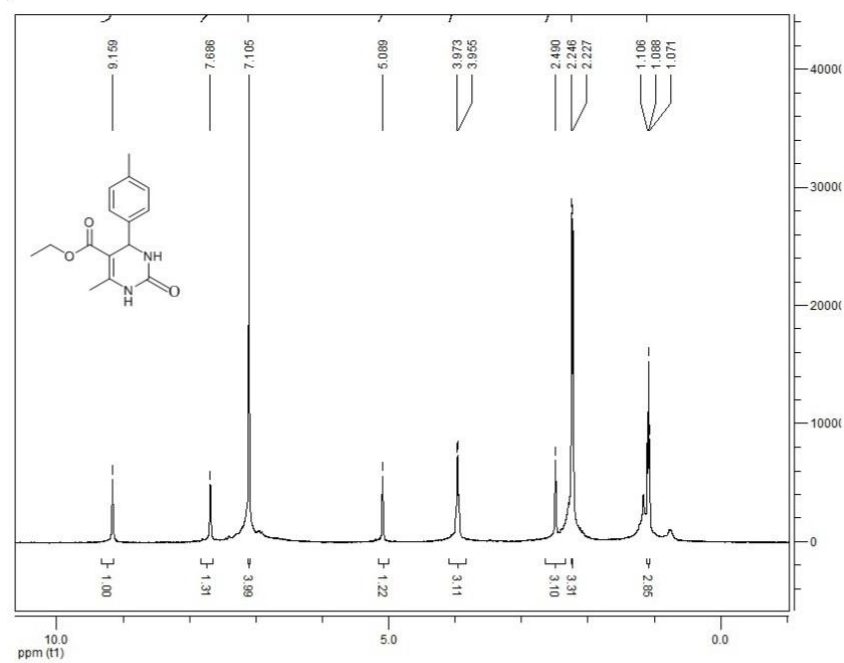


Figure S4. ¹H NMR spectrum of **4b**

Ethoxycarbonyl-4-[(2,4-dichloro)phenyl]-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4c)

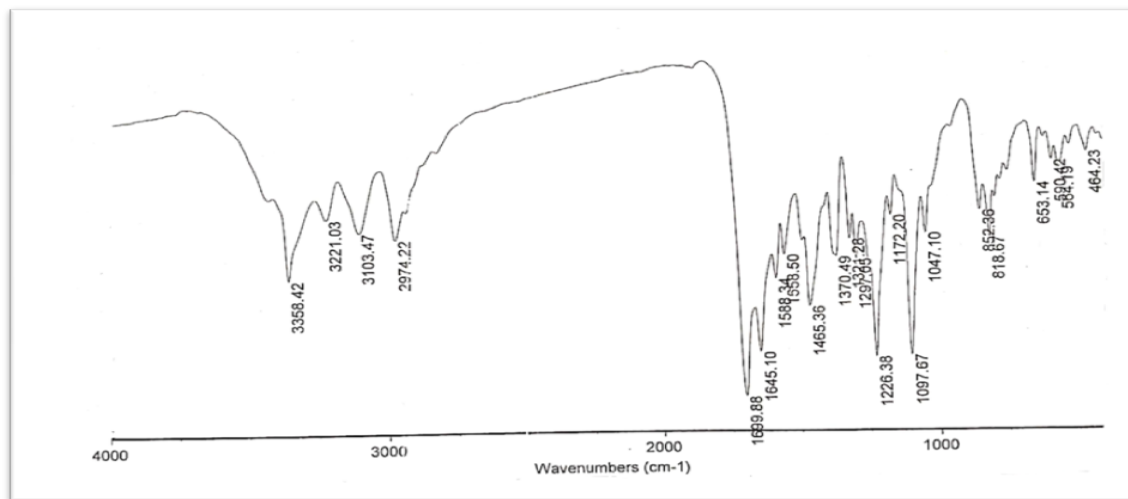


Figure S5. FT-IR spectrum of **4c**

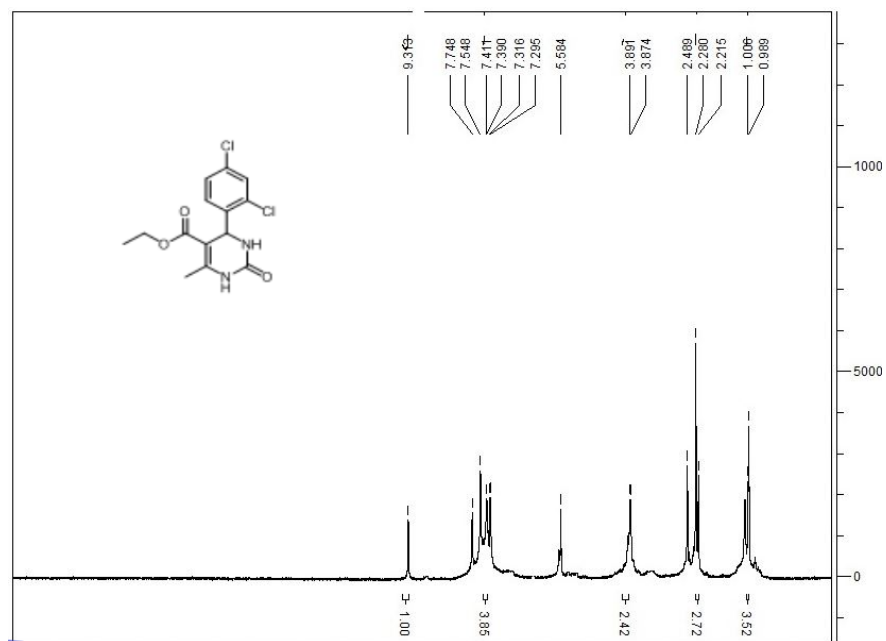


Figure S6. ^1H NMR spectrum of **4c**

Ethoxycarbonyl-4-[(4-methoxy)phenyl]-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4d)

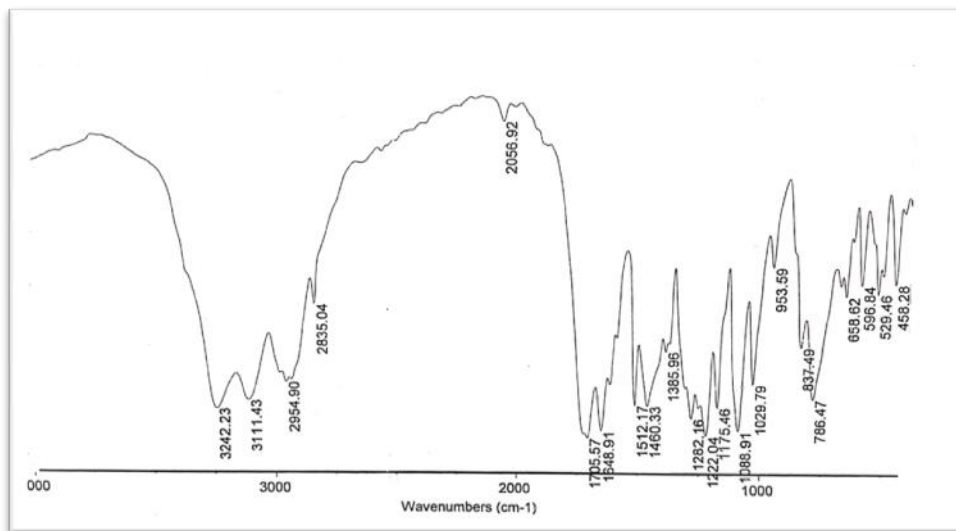


Figure S7. FT-IR spectrum of **4d**

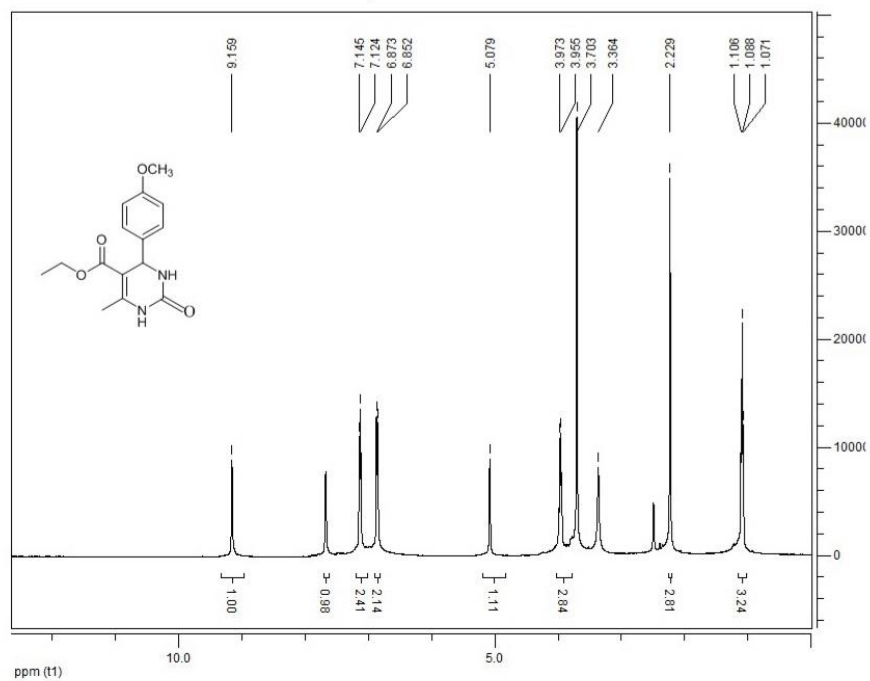


Figure S8. ^1H NMR spectrum of **4d**

Ethoxycarbonyl-4-(3-nitrophenyl)-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4e)

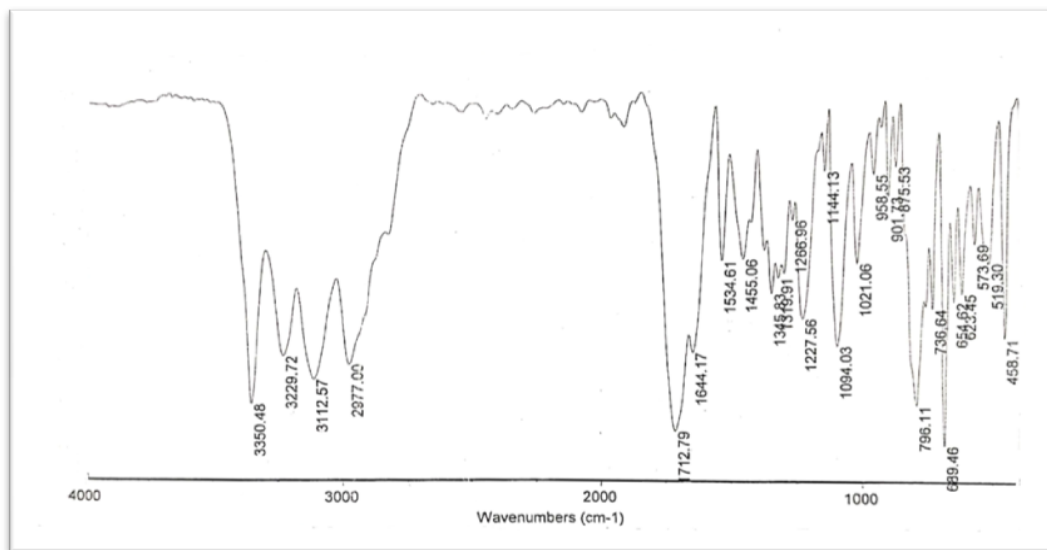


Figure S9. FT-IR spectrum of 4e

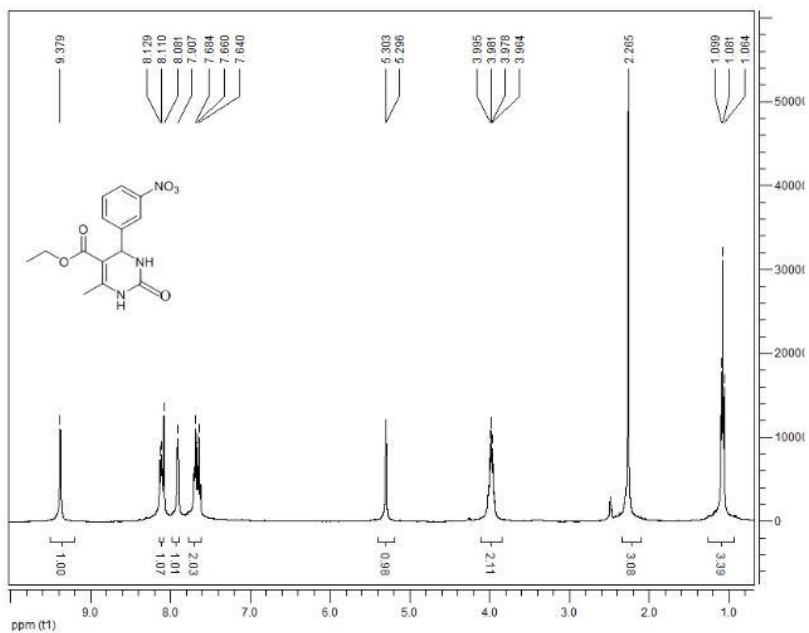


Figure S10. ¹H NMR spectrum of 4e

Ethoxycarbonyl-4-(4-nitrophenyl)-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4f)

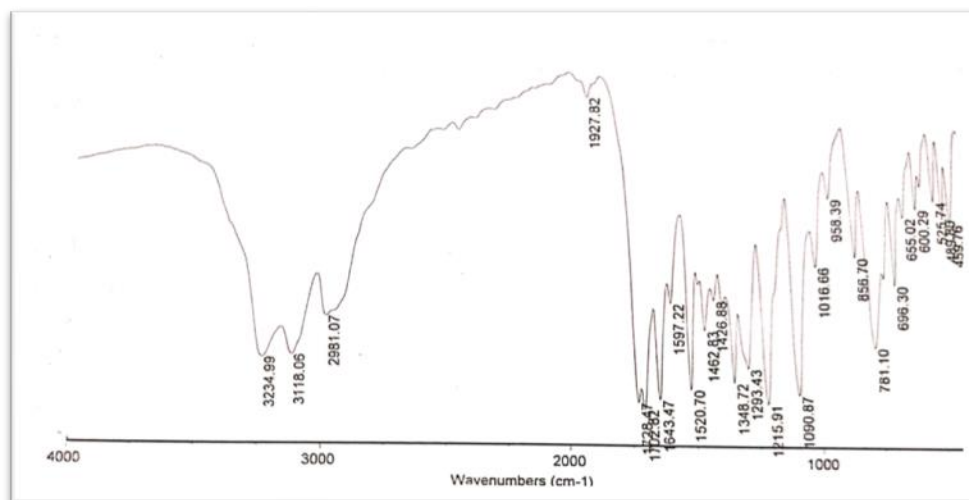


Figure S11. FT-IR spectrum of **4f**

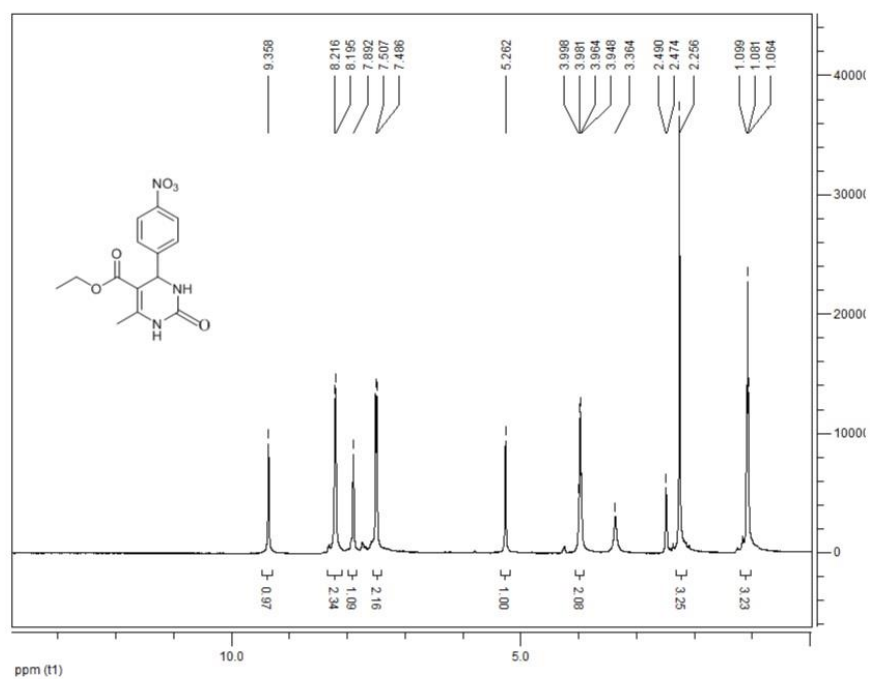


Figure S12. ¹H NMR spectrum of **4f**

Ethoxycarbonyl-4-[(4-hydroxy)phenyl]-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4g)

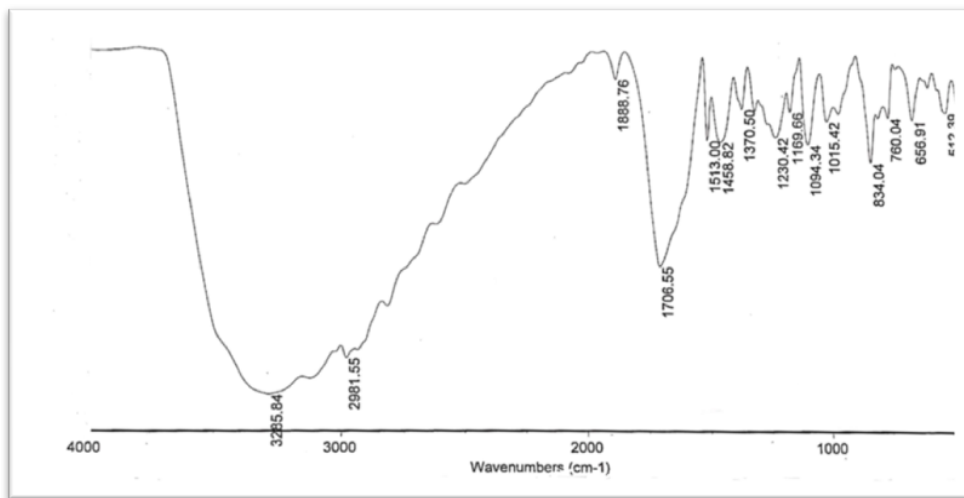


Figure 13. FT-IR spectrum of **4g**

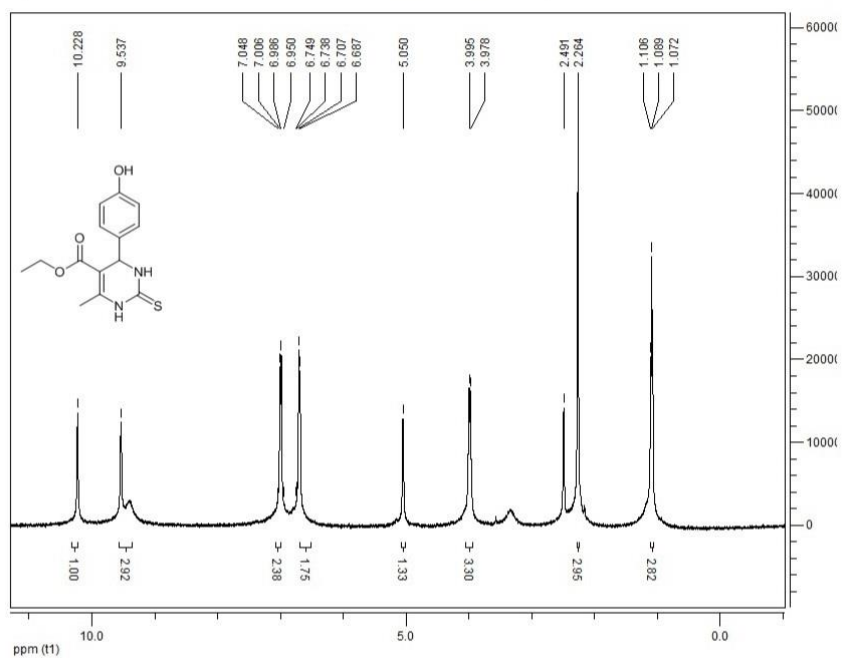


Figure S14. ¹H NMR spectrum of **4g**

Ethoxycarbonyl-4-[(4-chloro)phenyl]-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4h)

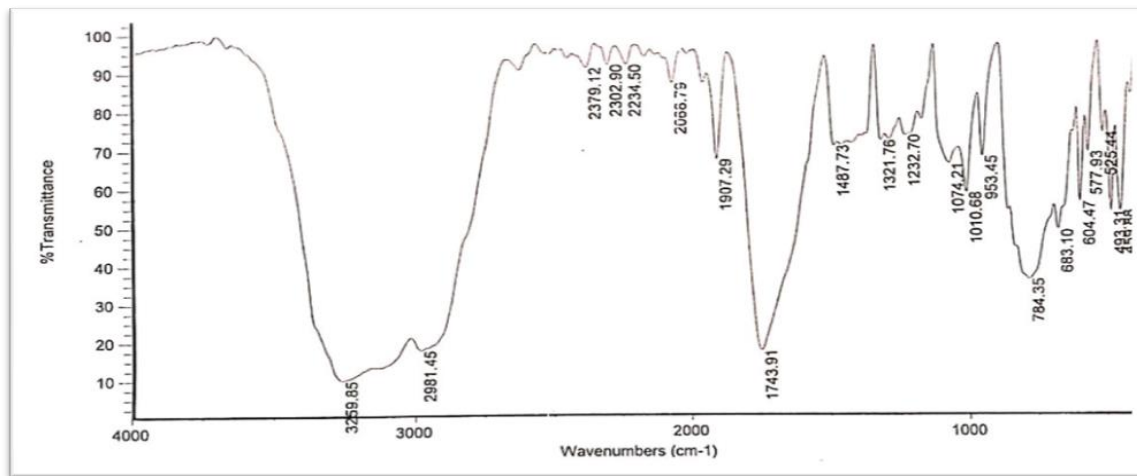


Figure S5. FT-IR spectrum of 4h

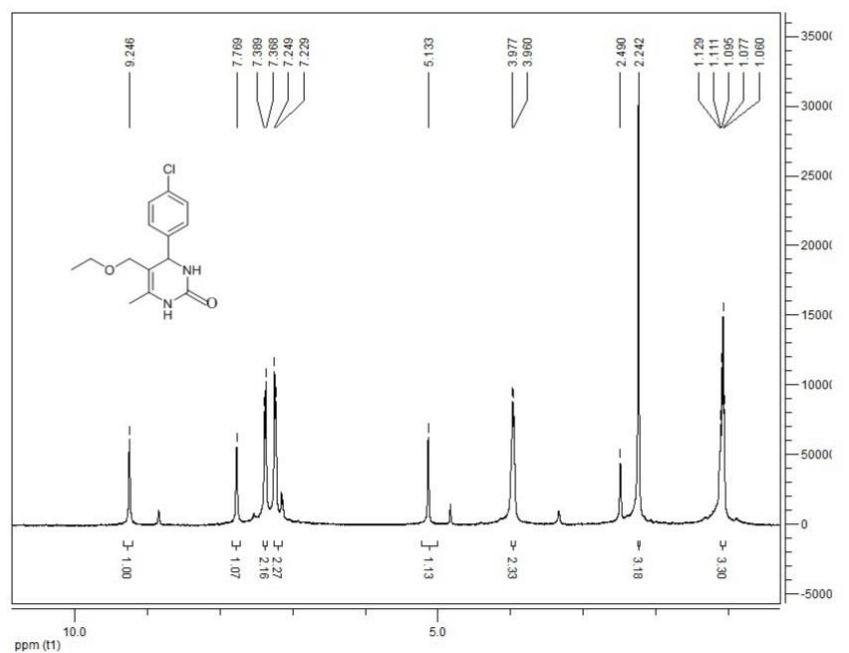


Figure S16. ¹H NMR spectrum of 4h

Ethoxycarbonyl-4-[(2-chloro)phenyl]-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4i)

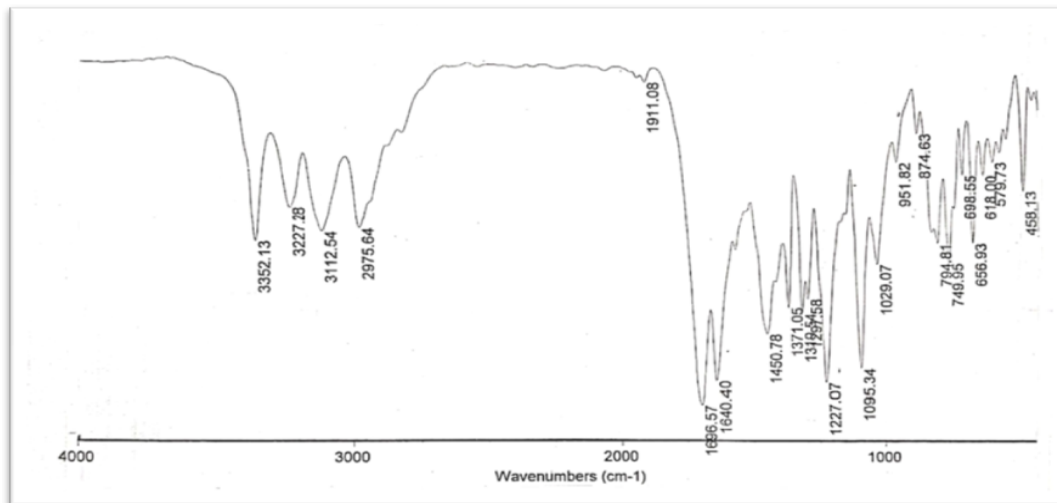


Figure S17. FT-IR spectrum of **4i**

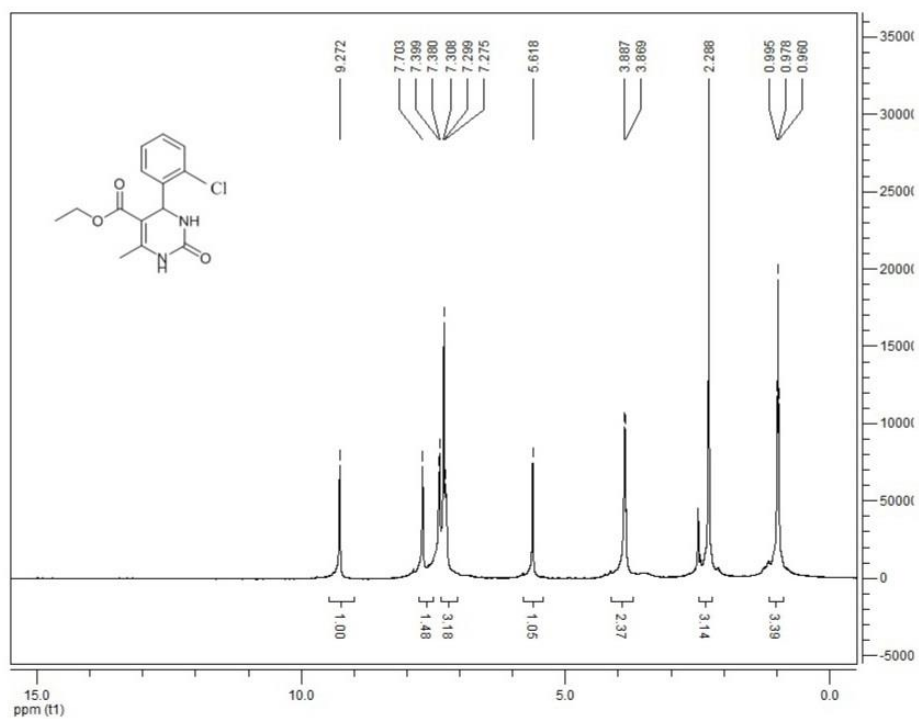


Figure S18. ¹H NMR spectrum of **4i**

Ethoxycarbonyl-4-[(4-fluoro)phenyl]-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4j)

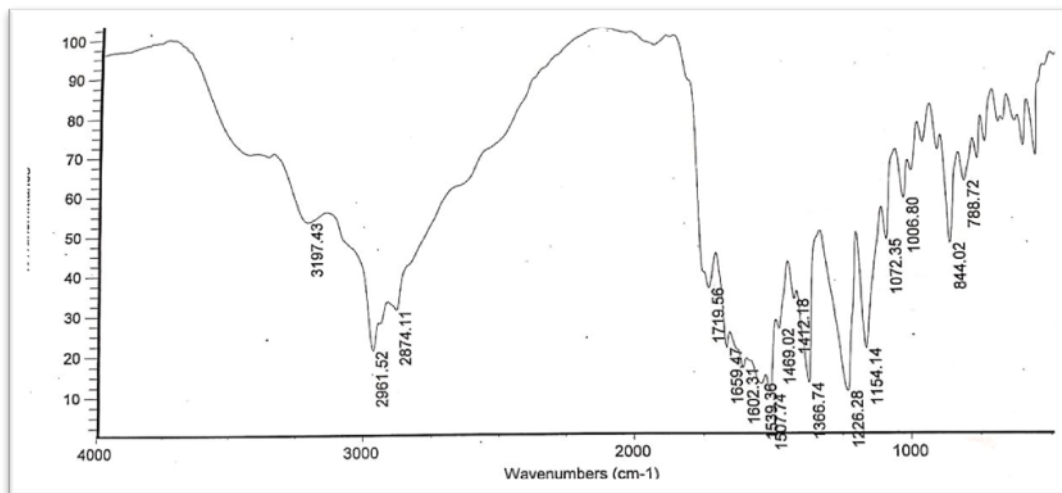


Figure S19. FT-IR spectrum of **4j**

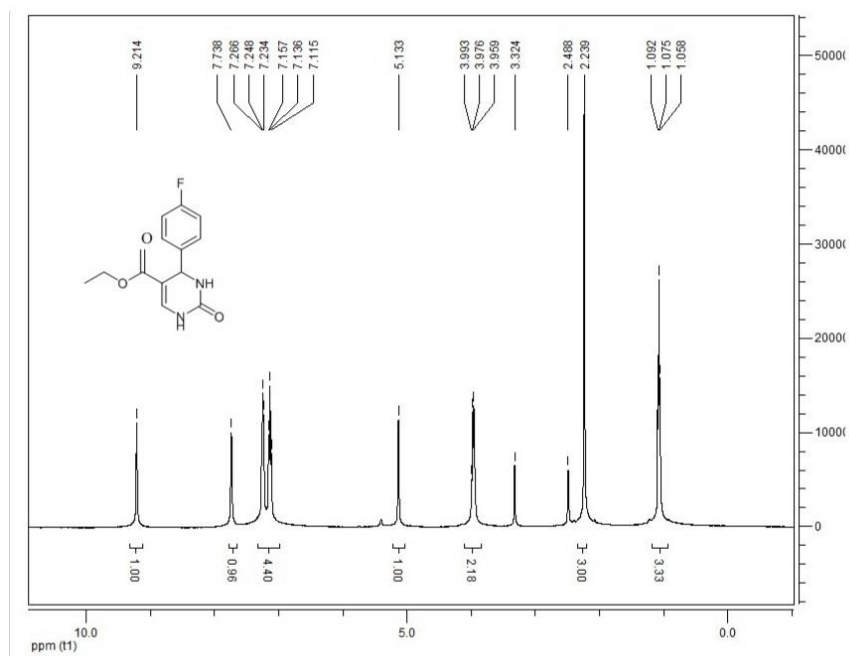


Figure S20. ¹H NMR spectrum of **4j**

Ethoxycarbonyl-4-[(2-Nitro)phenyl]-6-methyl-3,4-dihydropyrimidin-2(1H)-one (4k)

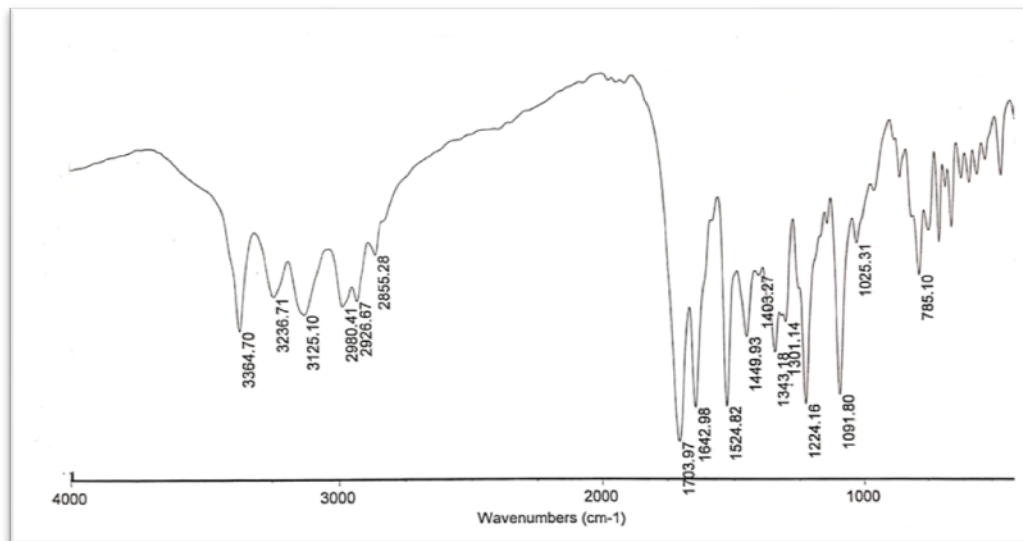


Figure S21. FT-IR spectrum of **4k**

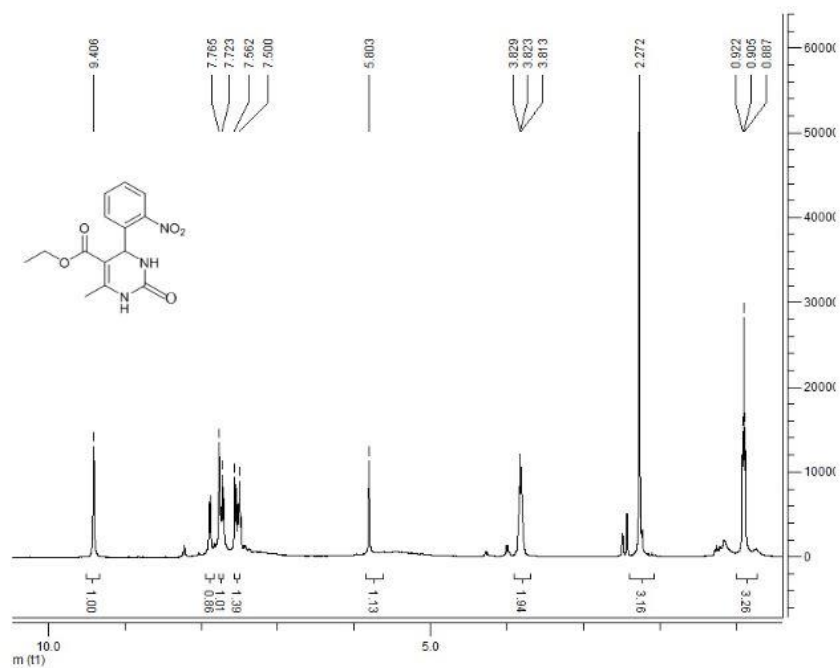


Figure S22. ¹H NMR spectrum of **4k**

Ethoxycarbonyl-4-[(4-phenyl)-6-methyl-3,4-dihydropyrimidin-2(1H)-thione
(4I)

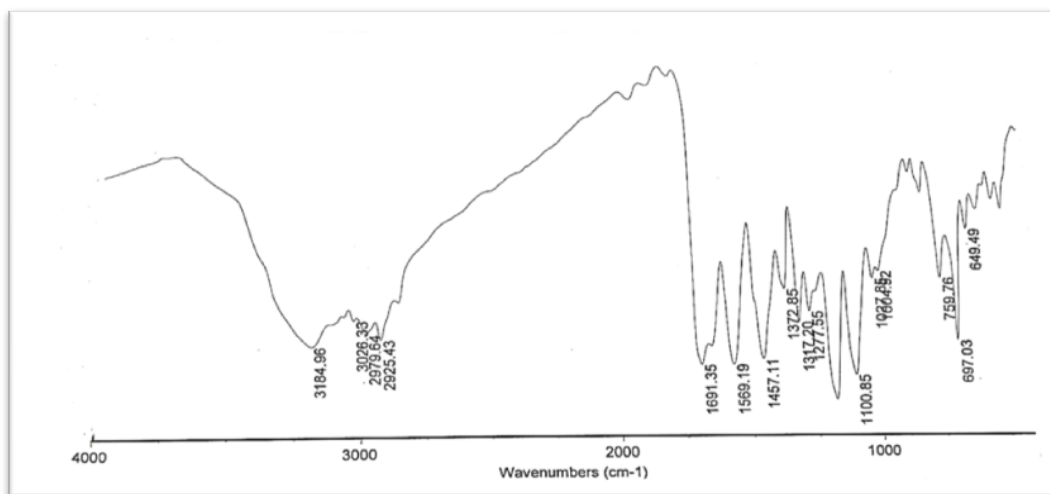


Figure S23. FT-IR spectrum of **4I**

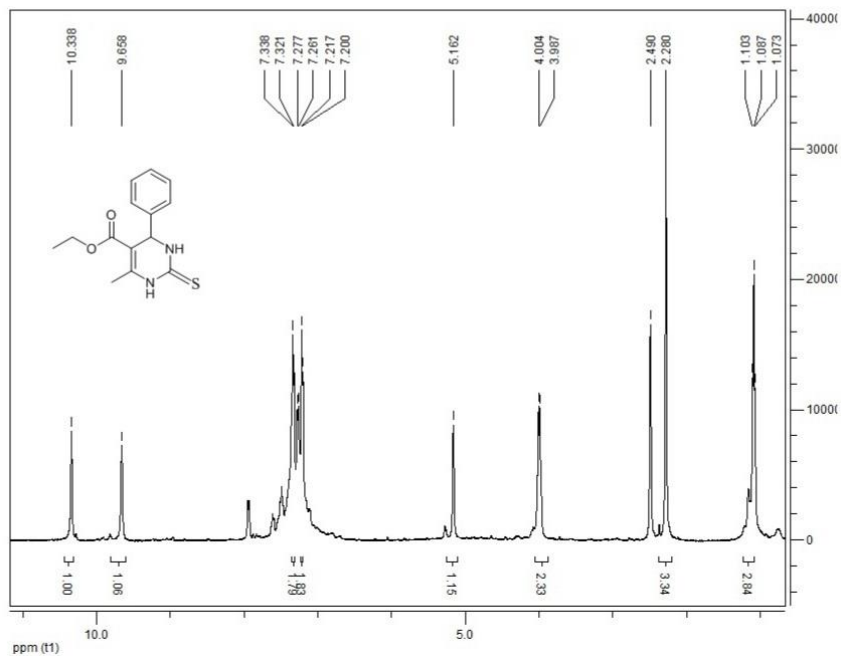


Figure S24. ¹H NMR spectrum of **4I**

Ethoxycarbonyl-4-[(4-hydroxy)phenyl]-6-methyl-3,4-dihydropyrimidin-2(1H)-thione (4m)

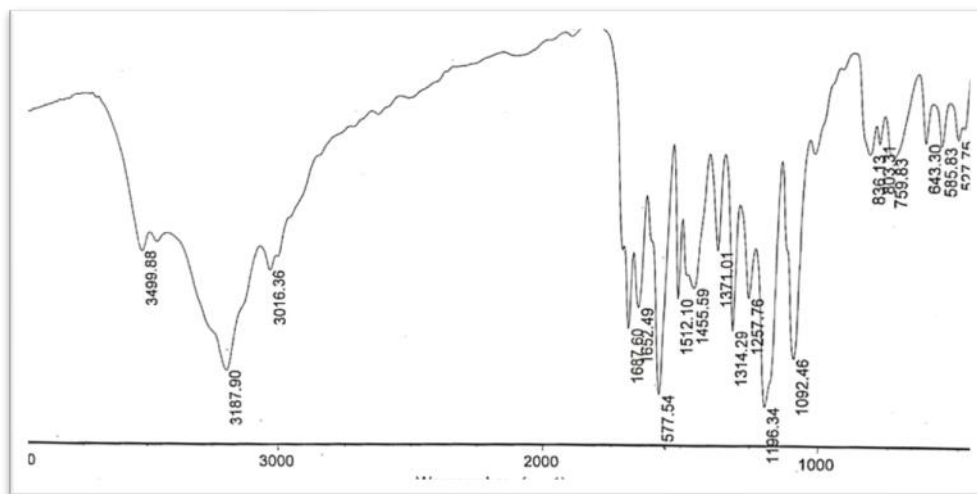


Figure S25. FT-IR spectrum of **4m**

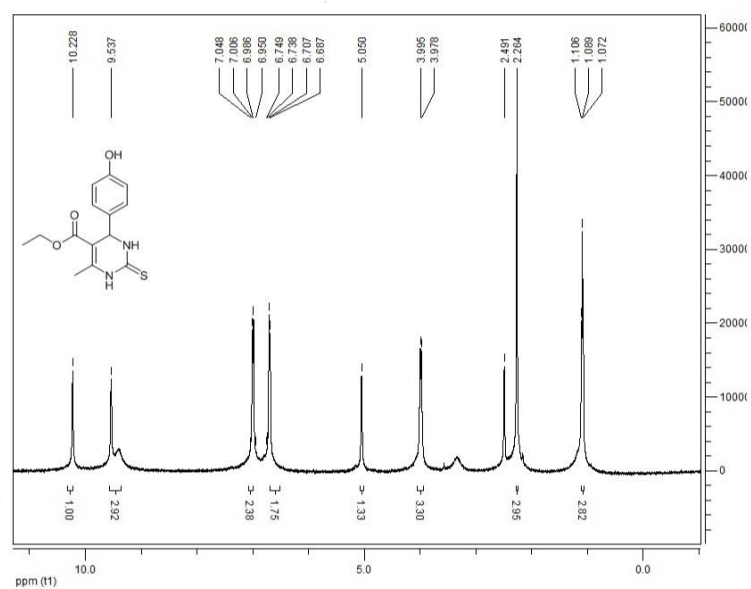


Figure S26. ¹H NMR spectrum of **4m**

Ethoxycarbonyl-4-[(2,4-dichloro-phenyl]-6-methyl-3,4-dihydropyrimidin-2(1H)-thione (4n)

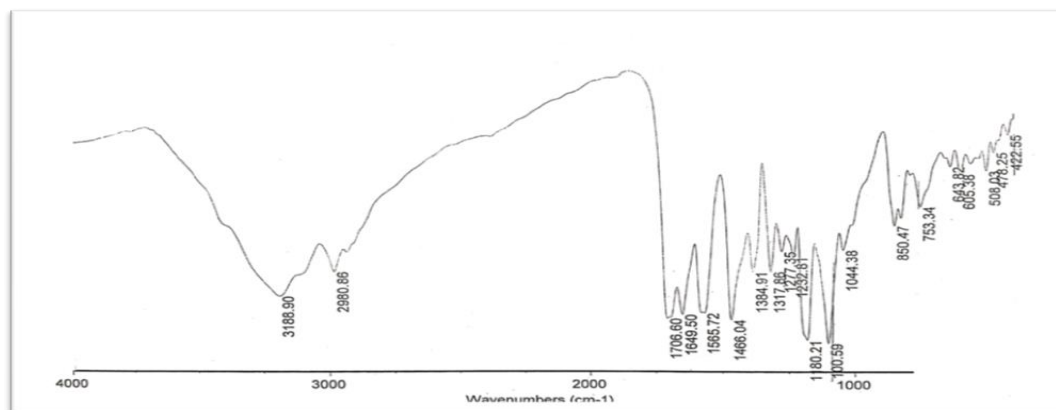


Figure S27. FT-IR spectrum of **4n**

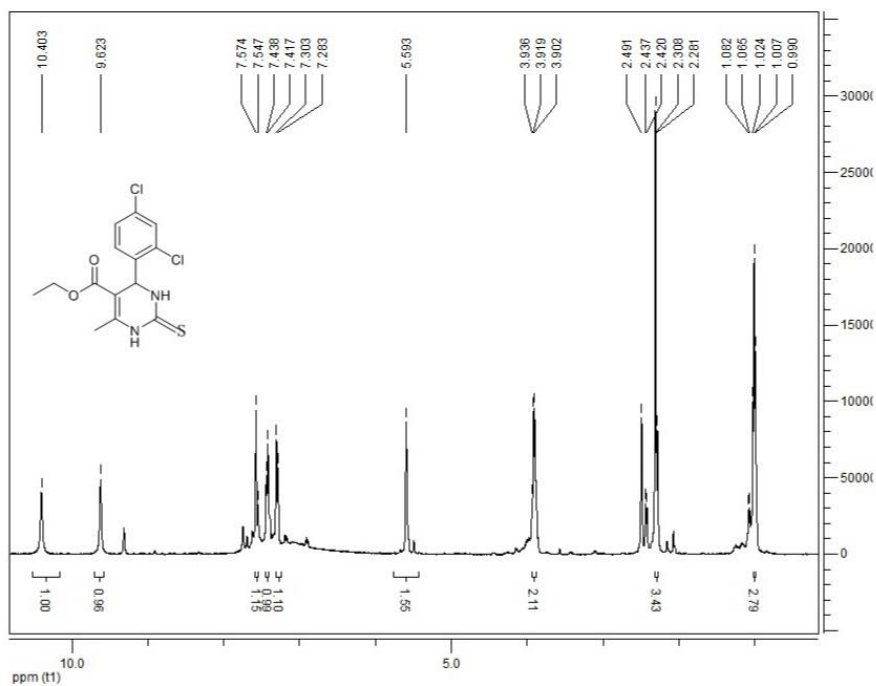


Figure S28. ¹H NMR spectrum of **4n**