

SUPPLEMENTARY MATERIAL TO
Synthesis and biological evaluation of some new heterocyclic derivatives from substituted thiopyrimidine

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4-Methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl ethyl glycinate (2)

Yield: 70 %, colorless oil; IR (KBr, cm^{-1}): 3101, 2950, 2845, 1745, 1217, 1166; $^1\text{H-NMR}$ (400 MHz, DMSO-d_6), (δ , ppm): 8.28 (1H, s, C-H thiopyrimidine), 7.46 (2H, dd, $J=7.4, 1.5$ Hz, Ar-H), 7.29 (2H, t, $J=7.2$ Hz, Ar-H), 7.26 – 7.19 (1H, m, Ar-H), 5.50 (1H, m, N-H), 4.09 (2H, q, $J=6.0$ Hz, CH_2 ester), 3.60 (2H, d, $J=33.5$ Hz, CH_2 next to NH), 2.88 (3H, s, CH_3 thiopyrimidine), 1.31 (3H, t, $J=6.0$ Hz, CH_3 ester). $^{13}\text{C-NMR}$ (100 MHz, DMSO-d_6), (δ , ppm): 176.3, 168.6, 166.6, 160.9, 134.5, 129.4, 128.8, 127.6, 110.3, 61.2, 57.3, 24.8, 14.7.

2-((4-Methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino) aceto hydrazide (3)

Yield: 75 %, solid powder (white); M.p.: 153 – 155 $^\circ\text{C}$; IR (KBr, cm^{-1}): 3315, 2920, 2850, 1681, 1150; $^1\text{H-NMR}$ (400 MHz, DMSO-d_6), (δ , ppm): 8.17 (1H, s, NH next to NH_2), 7.41 – 7.36 (2H, m, Ar-H), 7.25 (2H, dd, $J=10.9, 4.2$ Hz, Ar-H), 7.22 – 7.18 (3H, m, Ar-H), 4.22 (1H, s, N-H) 3.80 (2H, d, $J=15.8$ Hz, NH_2), 3.56 (1H, s, CH_2 next to C=O), 2.90 (1H, s, CH_2 next to C=O), 2.68 (3H, s, CH_3). $^{13}\text{C-NMR}$ (100 MHz, DMSO-d_6), (δ , ppm): 176.3, 172.3, 166.6, 160.9, 134.5, 129.4, 128.8, 127.6, 110.3, 53.3, 24.8.

2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)-N'-(4-nitrobenzylidene)-acetohydrazide. (4)

Yield: 86 %, solid powder (yellow); M.p.: 200 – 201 $^\circ\text{C}$; IR (KBr, cm^{-1}): 3213, 2933, 2864, 1697, 1672, 1541, 1373, 1158; $^1\text{H-NMR}$ (400 MHz, DMSO-d_6), (δ , ppm): δ 9.83 (1H, s, NH next to C=O), 8.44 (1H, s, CH=N), 8.22 (1H, s, CH thiopyrimidine), 8.08 (2H, d, $J=7.5$ Hz, Ar-H), 7.78 (2H, d, $J=7.5$ Hz, Ar-H), 7.41 (2H, dd, $J=7.5, 1.5$ Hz, Ar-H), 7.30 (2H, t, $J=7.4$ Hz, Ar-H), 7.26 – 7.20 (1H, m, Ar-H), 3.75 (1H, s, CH_2), 3.61 (1H, s, CH_2), 3.49 (1H, s, NH), 2.92 (3H, s, CH_3). $^{13}\text{C-NMR}$ (100 MHz, DMSO-d_6), (δ , ppm): 176.3, 166.6, 163.9, 160.9, 148.6, 148.0, 139.9, 134.5, 129.4, 128.8, 128.0, 127.6, 124.5, 110.3, 53.6, 24.8.

N'-(2,4-dimethoxybenzylidene)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)acetohydrazide. (5)

Yield: 69 %, solid powder (pale yellow); M.p.: 108–110 $^\circ\text{C}$; IR (KBr, cm^{-1}): 3188, 2939, 2835, 1681, 1602, 1207; $^1\text{H-NMR}$ (400 MHz, DMSO-d_6), (δ , ppm): 10.41 (1H, s, NH next to C=O), 8.49 (1H, s, CH thiopyrimidine), 8.27 (1H, s, CH=N), 7.48 (1H, d, $J=7.4$ Hz, Ar-CH),

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7.44 (2H, dd, $J=7.4, 1.2$ Hz, Ar-CH), 7.29 (2H, t, $J=7.4$ Hz, Ar-CH), 7.22 (1H, m, Ar-CH), 6.61 – 6.58 (2H, m, Ar-CH), 6.00 (1H, s, NH), 3.79 (6H, s, OCH₃), 3.69 (1H, CH₂), 3.51 (1H, CH₂), 3.06 (3H, s, CH₃). ¹³C-NMR (100 MHz, DMSO-d₆), (δ , ppm): 176.3, 166.6, 163.9, 162.8, 161.0, 160.9, 149.8, 134.5, 129.7, 129.4, 128.8, 127.6, 117.5, 110.3, 107.0, 100.3, 56.8, 56.0, 53.6, 24.8.

N'-(4-(dimethyl amino) benzylidene)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)aceto hydrazide (6)

Yield: 77 %, solid powder (orang); M.p.: 229-231 °C; IR (KBr, cm⁻¹): 3201, 2923, 2836, 1675, 1634, 1150; ¹H-NMR (400 MHz, DMSO-d₆), (δ , ppm): 10.93 (1H, s, NH next to C=O), 8.54 (1H, s, CH thiopyrimidine), 8.40 (1H, s, CH=N), 7.49 (2H, dd, $J=7.4, 1.2$ Hz, Ar-CH), 7.37 (2H, d, $J=7.5$ Hz, Ar-CH), 7.30 (2H, t, $J=7.2$ Hz, Ar-CH), 7.26 – 7.20 (1H, m, Ar-CH), 6.64 (2H, d, $J=7.5$ Hz, Ar-CH), 5.73 (1H, s, NH), 3.56 (1H, s, CH₂), 3.43 (1H, s, CH₂), 2.96 (3H, s, CH₃), 2.89 (6H, s, N-CH₃). ¹³C-NMR (100 MHz, DMSO-d₆), (δ , ppm): 176.3, 166.6, 163.9, 160.9, 153.9, 148.6, 134.5, 129.4, 128.8, 128.8, 127.6, 121.0, 111.0, 110.3, 53.6, 41.9, 24.8.

2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino)-*N*-(2-(4-nitrophenyl)-4-oxothiazolidin-3-yl) acetamide (7)

Yield: 80 %, solid powder (white); M.p.: 226-228 °C; IR (KBr, cm⁻¹): 3223, 2929, 2846, 1732, 1518, 1345, 1280, 712; ¹H-NMR (400 MHz, DMSO-d₆), (δ , ppm): 9.29 (1H, s, NH), 8.45 (1H, s, CH thiopyrimidin), 8.25 (2H, d, $J=7.5$ Hz, Ar-H), 7.57 (2H, d, $J=7.7$ Hz, Ar-H), 7.39 (2H, dd, $J=7.4, 1.2$ Hz, Ar-H), 7.28 (2H, t, $J=7.2$ Hz, Ar-H), 7.24 – 7.20 (1H, m, Ar-H), 6.92 (1H, s, CH thiazolidine), 3.51 (1H, s, NH), 3.48 (2H, s, CH₂ next to C=O), 3.38 (1H, d, $J=12.4$ Hz, CH next to C=O), 3.34 (1H, d, $J=12.6$ Hz, CH next S), 3.05 (3H, s, CH₃). ¹³C-NMR (100 MHz, DMSO-d₆), (δ , ppm): 176.3, 172.3, 168.3, 166.6, 160.9, 148.6, 148.2, 134.5, 129.4, 128.8, 127.6, 125.7, 123.9, 110.3, 67.6, 52.8, 33.3, 24.8.

Dimethoxyphenyl)-4-oxothiazolidin-3-yl)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino) acetamide (8)

Yield: 69 %, solid powder (white); M.p.: 289-290 °C; IR (KBr, cm⁻¹): 3013, 2932, 2825, 1674, 1264, 1172, 1028, 720; ¹H-NMR (400 MHz, DMSO-d₆), (δ , ppm): 9.39 (1H, s, NH next to C=O), 8.47 (1H, s, CH thiopyrimidine), 7.41 (2H, dd, $J=7.4, 1.5$ Hz, Ar-H), 7.28 (2H, dd, $J=12.1, 4.6$ Hz, Ar-H), 7.26 (1H, d, $J=7.5$ Hz, Ar-H), 7.23 (1H, dd, $J=5.2, 3.8$ Hz, Ar-H), 6.63 (1H, d, $J=1.4$ Hz, Ar-H), 6.60 (1H, dd, $J=7.5, 1.4$ Hz, Ar-H), 6.47 (1H, s, C-H thiazolidine), 4.10 (1H, s, NH), 3.80 (6H, s, OCH₃), 3.50 (2H, s, CH₂ next to C=O), 3.40 (1H, br.d, $J=12.3$ Hz, CH next to C=O), 3.36 (1H, br.d, $J=12.4$ Hz, CH next to S), 3.04 (3H, s, CH₃). ¹³C-NMR (100 MHz, DMSO-d₆), (δ , ppm): 176.3, 172.3, 168.3, 166.6, 161.3, 160.9, 155.6, 134.5, 129.7, 129.4, 128.8, 127.6, 120.0, 110.3, 107.4, 100.8, 61.4, 56.8, 56.0, 52.8, 33.3, 24.8.

N-(2-(4-(dimethylamino)phenyl)-4-oxothiazolidin-3-yl)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)acetamide (9)

Yield: 75 %, solid powder (pale pink); M.p.: 247-249 °C; IR (KBr, cm⁻¹): 3372, 3221, 3062, 2953, 2848, 1715, 1290, 731; ¹H-NMR (400 MHz, DMSO-d₆), (δ , ppm): 8.45 (1H, s, C-H thiopyrimidine), 7.80 (1H, s, NH), 7.41 (2H, dd, $J=7.3, 1.5$ Hz, Ar-H), 7.25 (2H, dd, $J=10.7, 4.5$ Hz, Ar-H), 7.23 – 7.21 (1H, m, Ar-H), 7.10 (2H, d, $J=7.5$ Hz, Ar-H), 6.68 – 6.65 (3H, m, Ar-H + C-H thiazolidine), 5.12 (1H, s, NH), 3.57 (1H, s, CH₂ next to NH), 3.37 (1H, br.d, $J=12.4$ Hz, CH₂ next to C=O), 3.34 (1H, s, CH₂ next to NH), 3.33 (1H, br.d, $J=8.8$ Hz, CH₂ next to S), 3.04 (3H, s, CH₃), 2.90 (6H, s, N(CH₃)₂). ¹³C-NMR (100 MHz, DMSO-d₆), (δ ,

ppm): 176.3, 172.3, 168.3, 166.6, 160.9, 155.3, 134.6, 133.7, 129.4, 128.8, 127.6, 127.5, 111.4, 110.3, 67.6, 52.8, 41.9, 33.3, 24.8.

1-(4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)-3-phenylthiourea (10)

Yield: 88 %, solid powder (pale green); M.p.: 157-159 °C; IR (KBr, cm⁻¹): 3172, 2920, 2837, 1560, 1485, 1372; ¹H-NMR (400 MHz, DMSO-d₆), (δ, ppm): 9.86 (1H, s, NH), 8.39 (1H, s, C-H thiopyrimidine), 7.52 (2H, dd, *J* = 7.4, 1.2 Hz Ar-H), 7.32 (2H, t, *J* = 7.4 Hz, Ar-H), 7.29 – 7.24 (4H, m, Ar-H), 7.22 – 7.17 (1H, m, Ar-H), 7.13 – 7.08 (2H, m, Ar-H), 2.90 (3H, s, CH₃). ¹³C-NMR (100 MHz, DMSO-d₆), (δ, ppm): 180.5, 176.2, 166.6, 162.9, 139.2, 134.5, 129.4, 129.0, 128.8, 127.6, 124.5, 121.5, 113.2, 24.8.

5-(4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)-1-phenyl-6-thioxopiperidine-2,4-dione(11)

Yield: 65 %, solid powder (pale brown); M.p.: 87-89 °C; IR (KBr, cm⁻¹): 3020, 2978, 2840, 1734, 1647, 1550-1392, 1502; ¹H-NMR (400 MHz, DMSO-d₆), (δ, ppm): 8.68 (1H, s, thiopyrimidine), 7.49 (2H, br.dd, *J* = 7.5, 1.4 Hz, Ar-H), 7.45 (2H, br.dd, *J* = 7.4, 1.3 Hz, Ar-H), 7.37 (2H, t, *J* = 7.5 Hz, Ar-H), 7.29 (2H, t, *J* = 7.5 Hz, Ar-H), 7.23 – 7.19 (1H, m, Ar-H), 7.15 – 7.10 (1H, m, Ar-H), 3.03 (3H, s, CH₃), 2.89 (2H, s, CH₂ Aliph. ring). ¹³C-NMR (100 MHz, DMSO-d₆), (δ, ppm): 181.5, 169.2, 164.9, 164.5, 164.4, 161.5, 137.4, 134.6, 130.5, 129.9, 129.7, 129.4, 129.2, 127.8, 110.5, 43.6, 24.8.

N-(4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) acet amide (12)

Yield: 85 %, solid powder (brown); M.p.: 184-186 °C; IR (KBr, cm⁻¹): 3207, 2983, 2841, 1735, 1595-1404; ¹H-NMR (400 MHz, DMSO-d₆), (δ, ppm): 9.05 (1H, s, NH), 8.26 (1H, s, CH thiopyrimidine), 7.36 (2H, dd, *J* = 7.3, 1.3 Hz, Ar-H), 7.23 (2H, t, *J* = 7.2 Hz, Ar-H), 7.20 – 7.15 (1H, m, Ar-H), 2.88 (3H, s, CH₃ thiopyrimidine), 2.01 (3H, s, CH₃ next to C=O). ¹³C-NMR (100 MHz, DMSO-d₆), (δ, ppm): 176.2, 166.6, 166.5, 134.5, 129.3, 128.8, 127.7, 113.2, 24.8, 20.5.

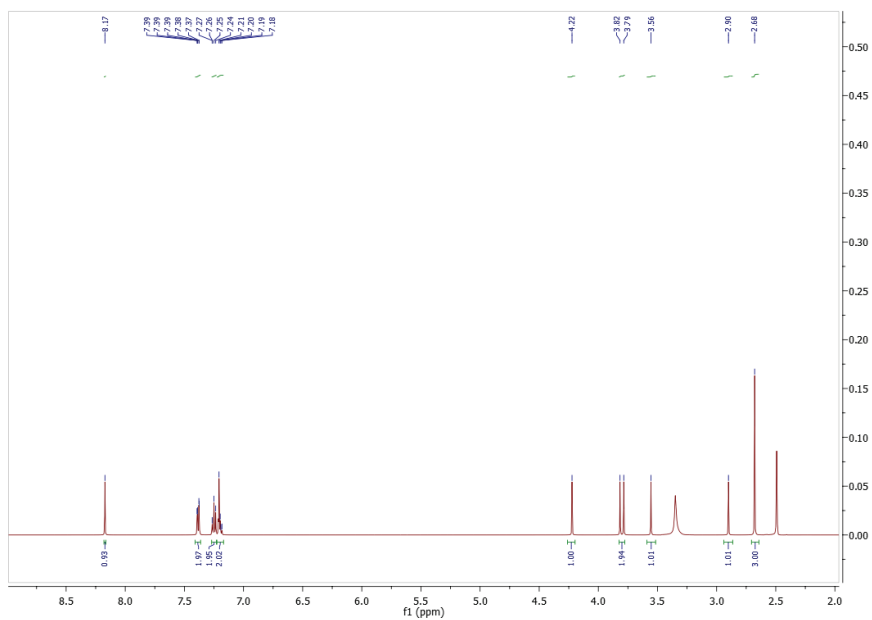
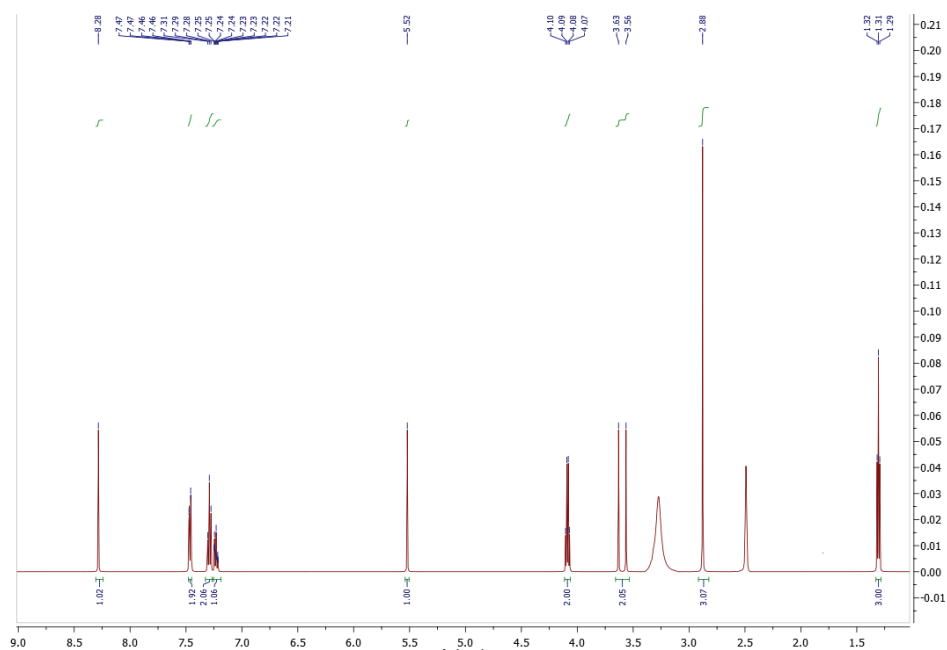
N-(4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)-3-(4-nitrophenyl) acrylamide (13)

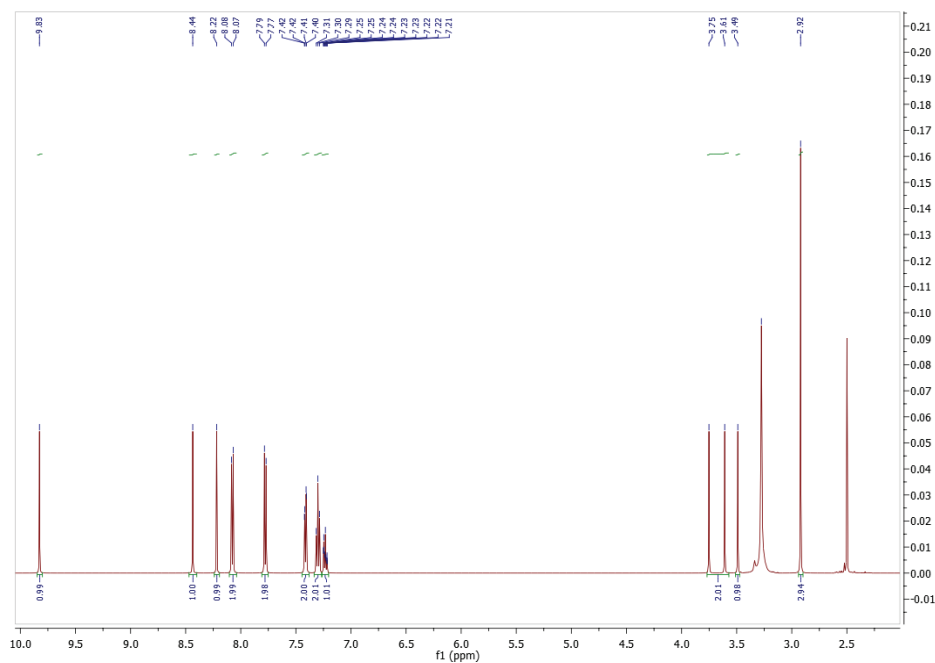
Yield: 70 %, solid powder (Pale brown); M.p.: 178-180 °C; IR (KBr, cm⁻¹): 3116, 2935, 2866, 1703, 1628; ¹H-NMR (400 MHz, DMSO-d₆), (δ, ppm): 9.55 (1H, s, NH), 8.41 (1H, s, C-H thiopyrimidine), 8.17 (2H, d, *J* = 7.5 Hz, Ar-H), 7.79 (1H, d, *J* = 15.2 Hz, β-CH), 7.55 (2H, d, *J* = 7.7 Hz, Ar-H), 7.42 (2H, dd, *J* = 7.4, 1.5 Hz, Ar-H), 7.26 (2H, t, *J* = 7.3 Hz, Ar-H), 7.22 – 7.17 (1H, m, Ar-H), 7.03 (1H, d, *J* = 15.2 Hz, α-CH), 3.06 (3H, s, CH₃). ¹³C-NMR (100 MHz, DMSO-d₆), (δ, ppm): 176.2, 166.6, 166.1, 162.9, 147.8, 146.7, 142.4, 134.5, 129.4, 129.1, 128.8, 127.6, 124.5, 122.2, 113.2, 24.8.

4-methyl-1-((6-(4-nitrophenyl)-2-thioxo-1,2-dihydropyrimidin-4-yl) amino)-6-phenylpyrimidine-2(1H)-thione (14)

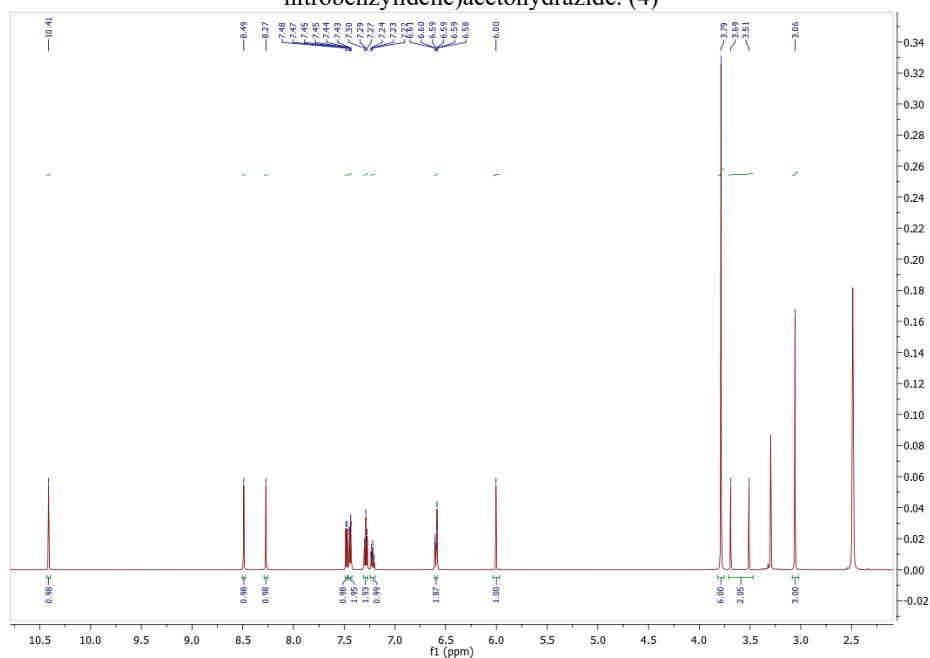
Yield: 81 %, solid powder (pale green); M.p.: 81-82 °C; IR (KBr, cm⁻¹): 3428, 2930, 1632, 1593; ¹H-NMR (400 MHz, DMSO-d₆), (δ, ppm): 9.02 (1H, s, C-H heterocyclic ring), 8.88 (1H, s, C-H thiopyrimidine), 8.21 (2H, d, *J* = 7.5 Hz, Ar-H), 7.73 (2H, d, *J* = 7.5 Hz, Ar-H), 7.49 (2H, dd, *J* = 7.4, 1.3 Hz, Ar-H), 7.24 (2H, t, *J* = 7.4 Hz, Ar-H), 7.20 – 7.12 (1H, m, Ar-H), 2.96 (3H, s, CH₃). ¹³C-NMR (100 MHz, DMSO-d₆), (δ, ppm): 176.2, 175.8, 166.6, 163.8, 162.9, 148.8, 146.8, 142.3, 134.5, 129.4, 128.8, 127.8, 127.6, 124.1, 113.2, 98.4, 24.8.

Note: The signal at 2.5 ppm in all spectra is related to the deuterated solvent (DMSO-d₆), and the signal around 3.3 ppm is due to the moisture in (DMSO-d₆).

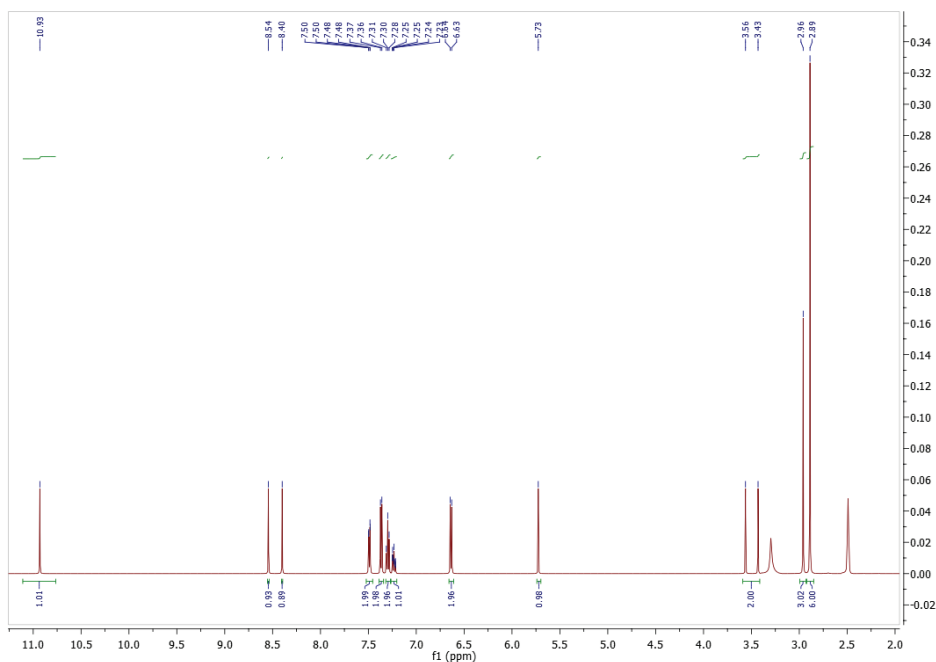




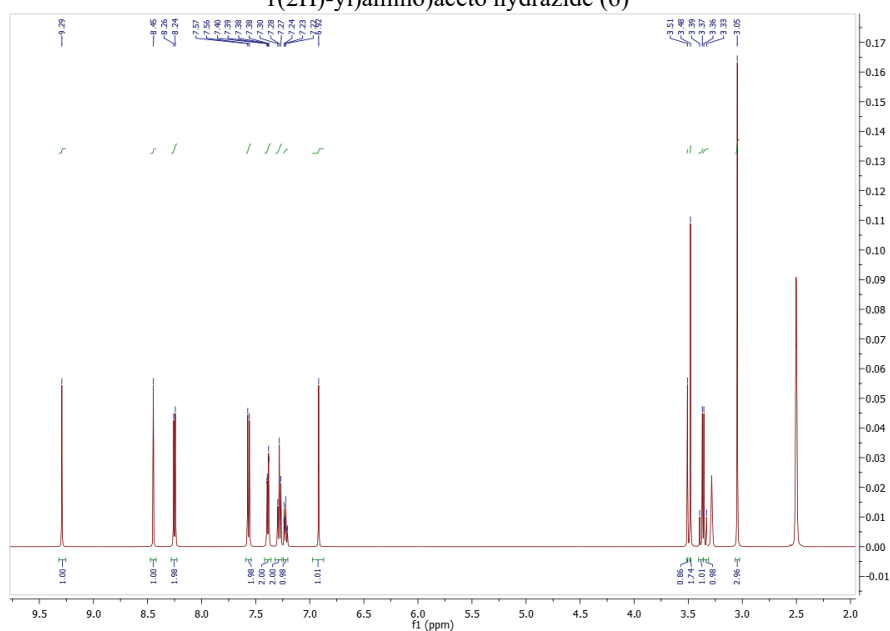
¹H-NMR for 2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)-N'-(4-nitrobenzylidene)acetohydrazide. (4)



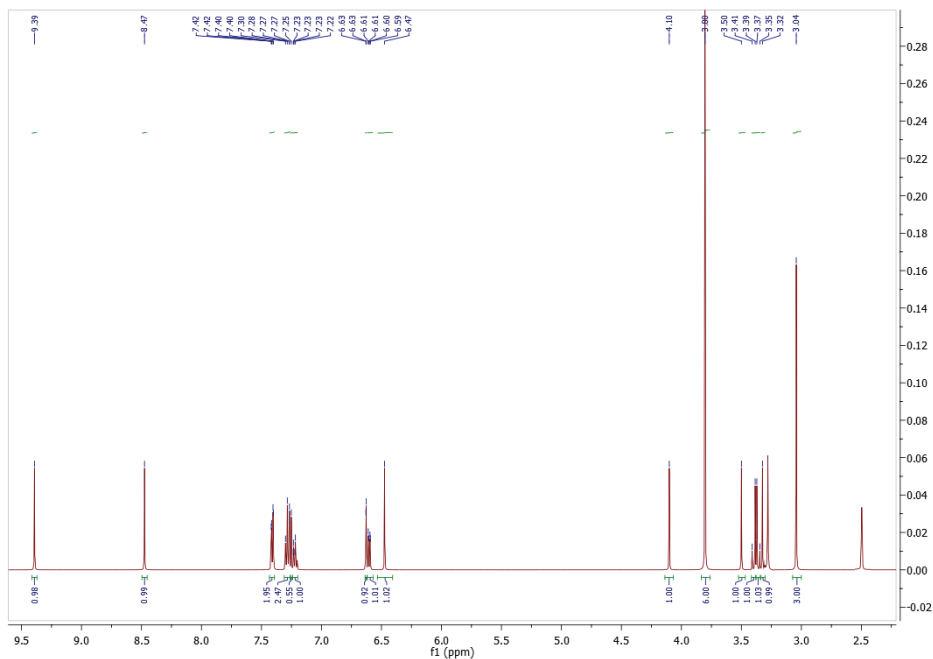
¹H-NMR for N'-(2,4-dimethoxybenzylidene)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)acetohydrazide.(5)



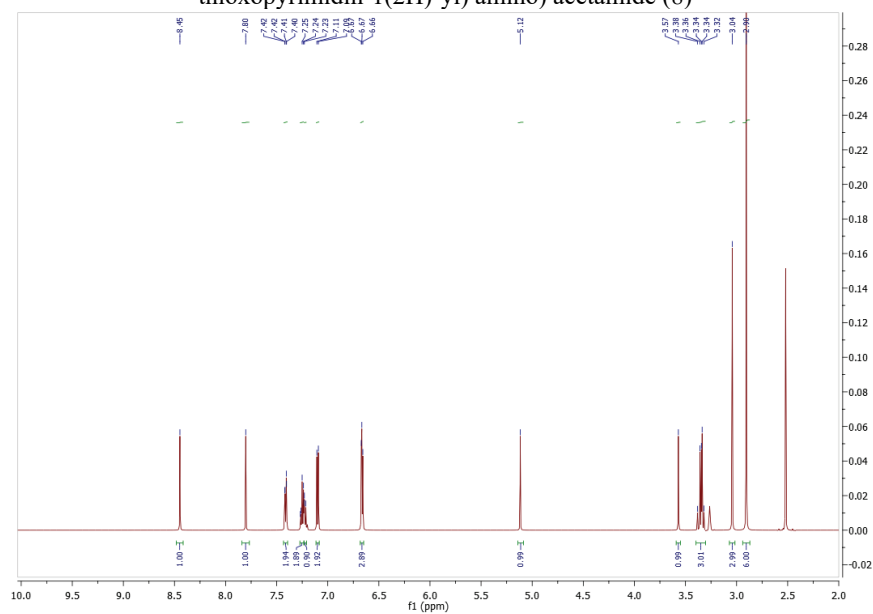
$^1\text{H-NMR}$ for N' -(4-(dimethyl amino) benzylidene)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)aceto hydrazide (6)



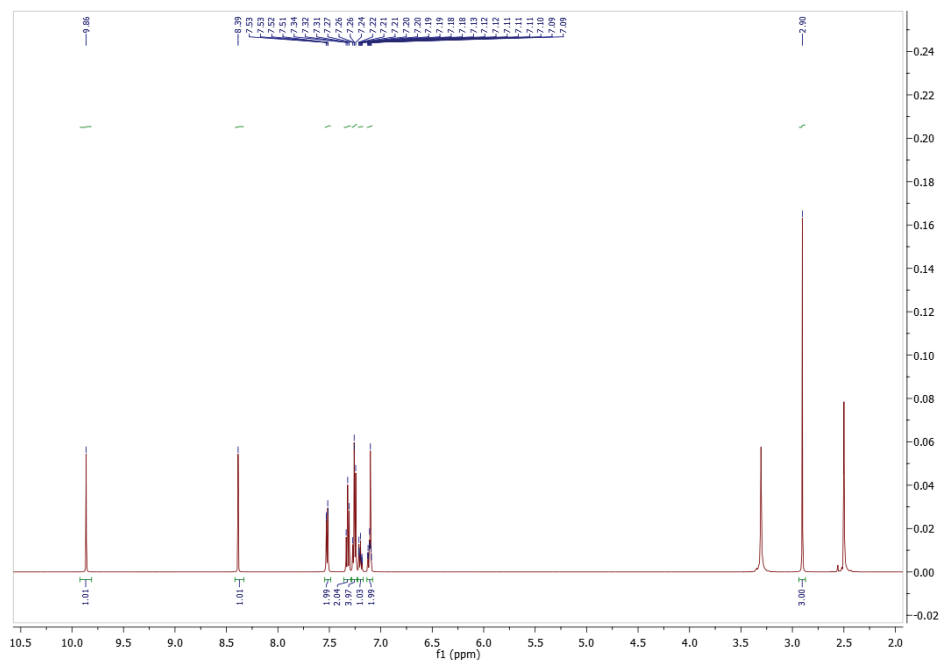
$^1\text{H-NMR}$ for 2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino)- N -(2-(4-nitrophenyl)-4-oxothiazolidin-3-yl) acetamide (7)



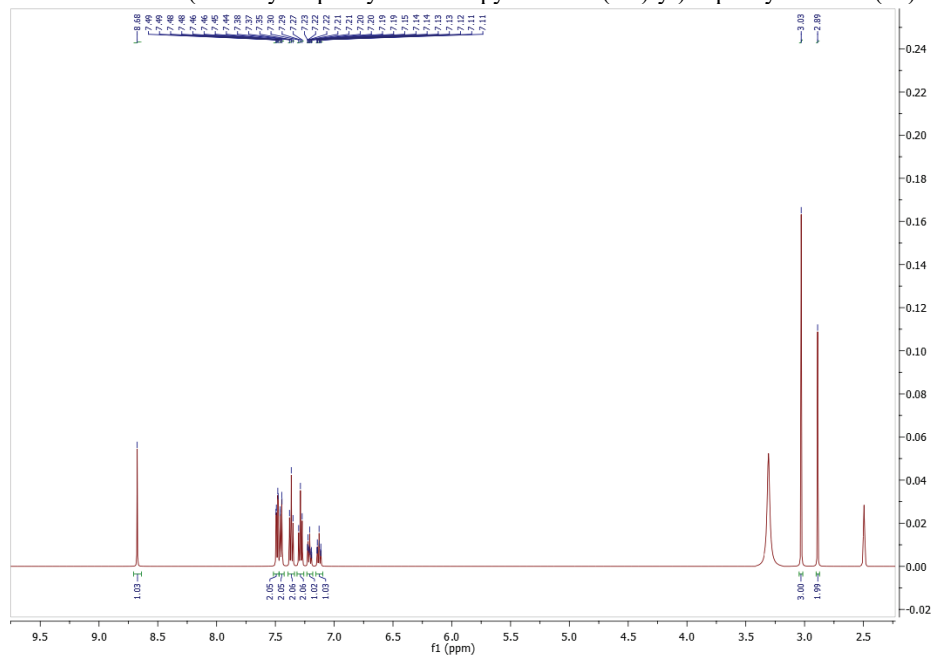
¹H-NMR for dimethoxyphenyl)-4-oxothiazolidin-3-yl)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino) acetamide (8)



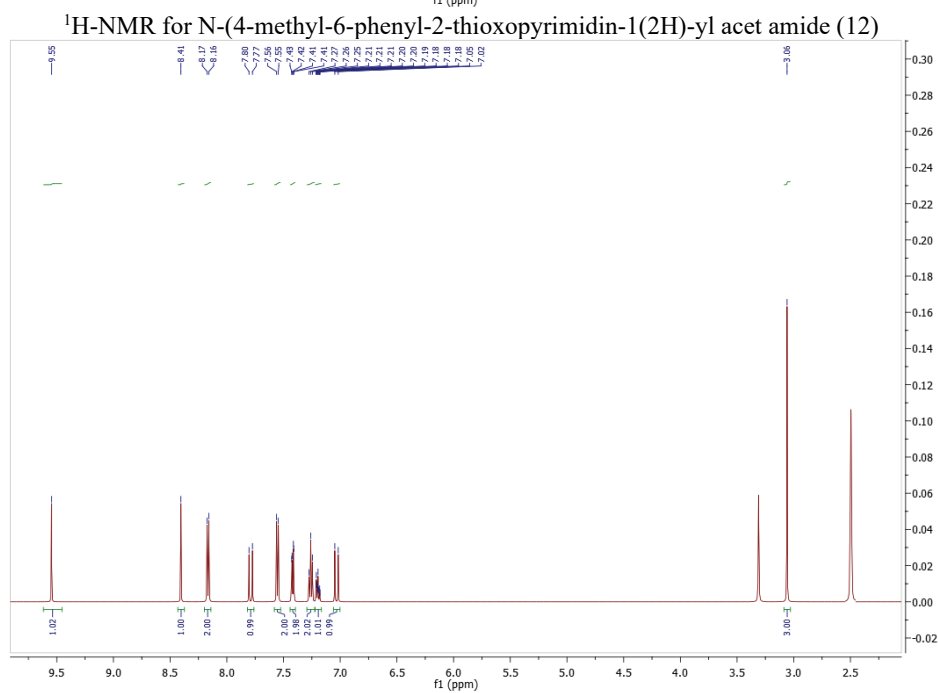
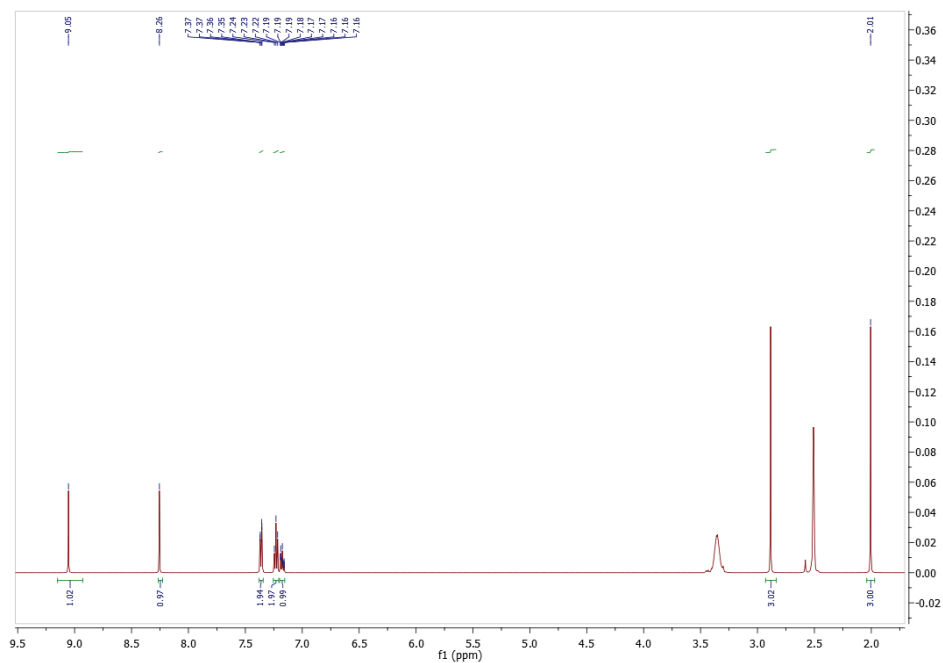
¹H-NMR for N-(2-(4-(dimethylamino)phenyl)-4-oxothiazolidin-3-yl)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)acetamide (9)

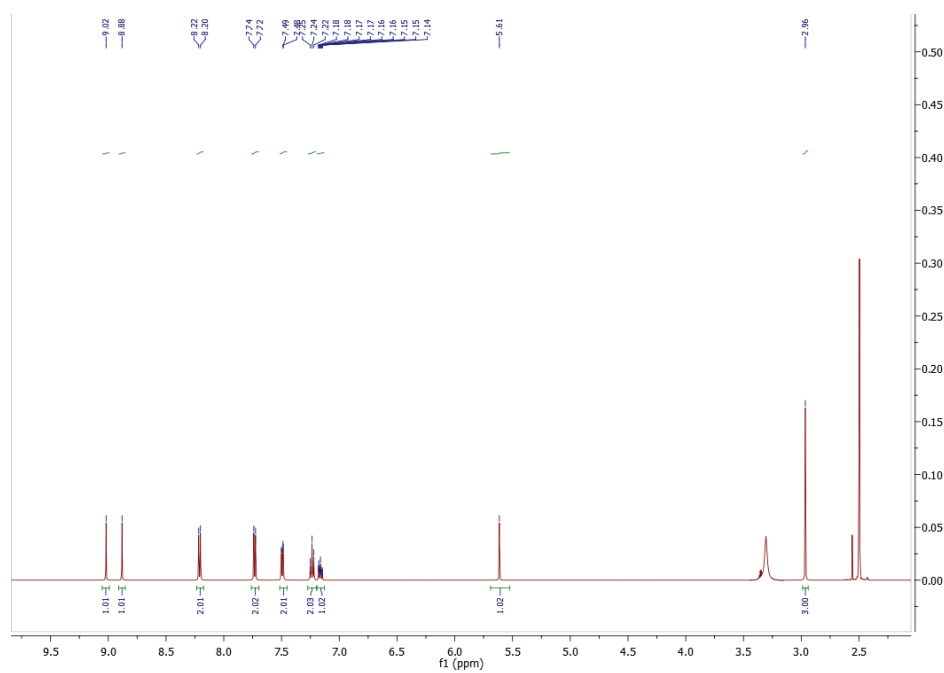


$^1\text{H-NMR}$ for 1-(4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)-3-phenylthiourea (10)

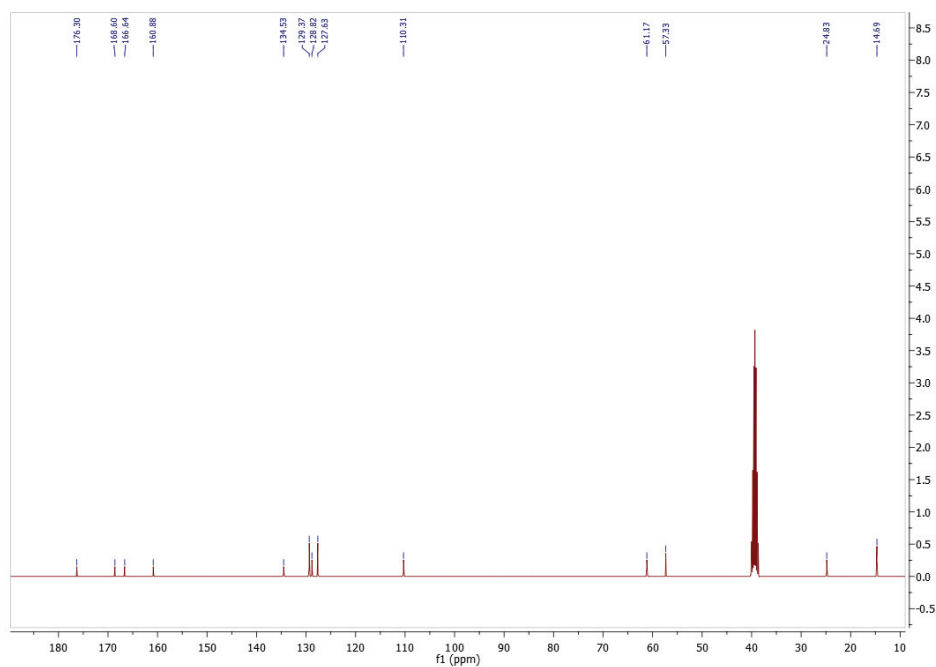
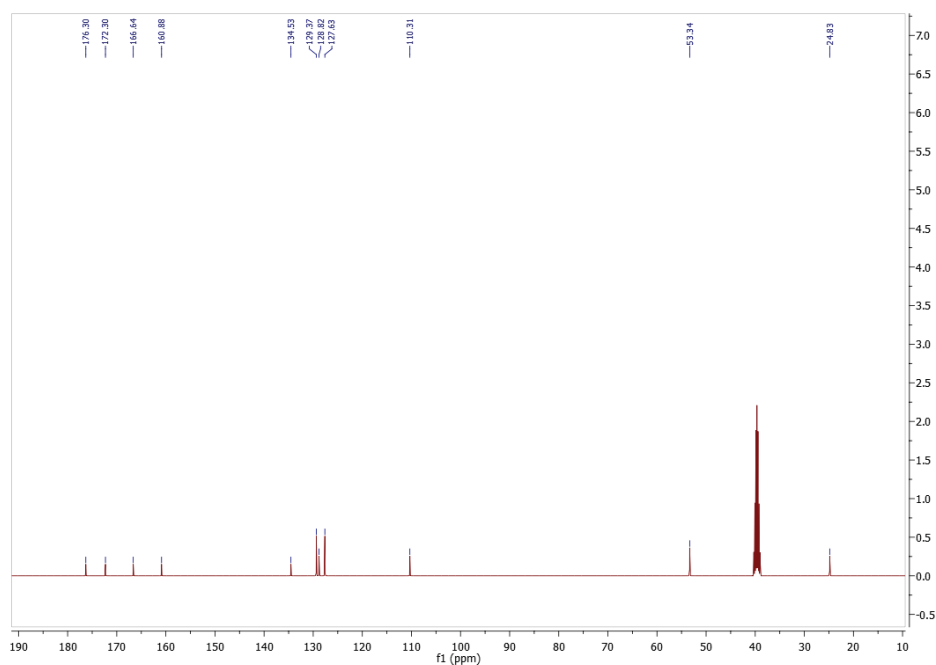


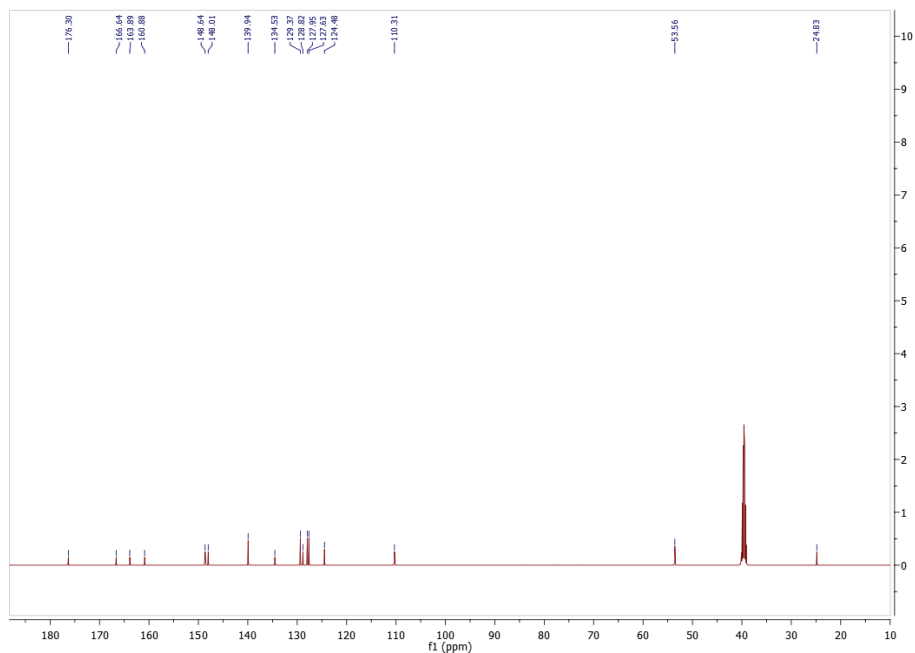
$^1\text{H-NMR}$ for 5-(4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)-1-phenyl-6-thioxopiperidine-2,4-dione (11)



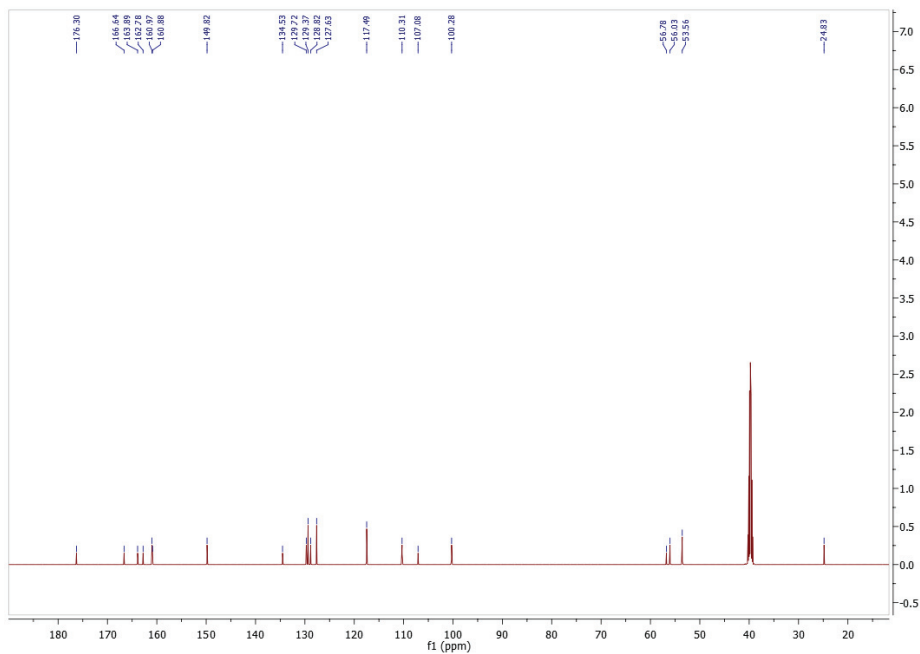


$^1\text{H-NMR}$ for 4-methyl-1-((6-(4-nitrophenyl)-2-thioxo-1,2-dihydropyrimidin-4-yl) amino)-6-phenylpyrimidine-2(1H)-thione (14)

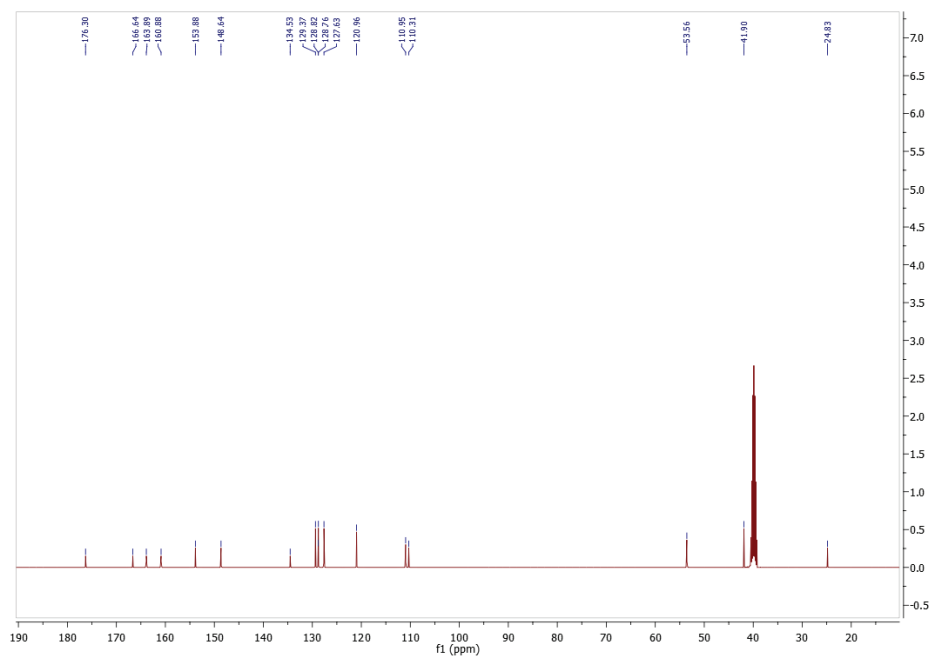
 $^{13}\text{C-NMR}$ for 4-Methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl ethyl glycinat (2) $^{13}\text{C-NMR}$ for 2-((4-Methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino) aceto hydrazide (3)



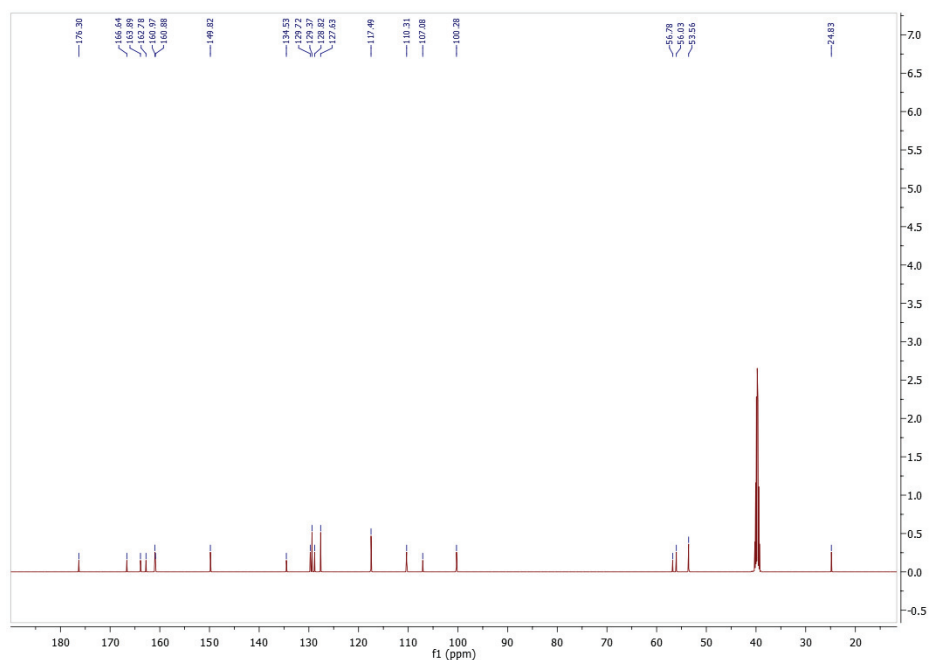
$^{13}\text{C-NMR}$ for 2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)-N'-(4-nitrobenzylidene)acetohydrazide. (4)



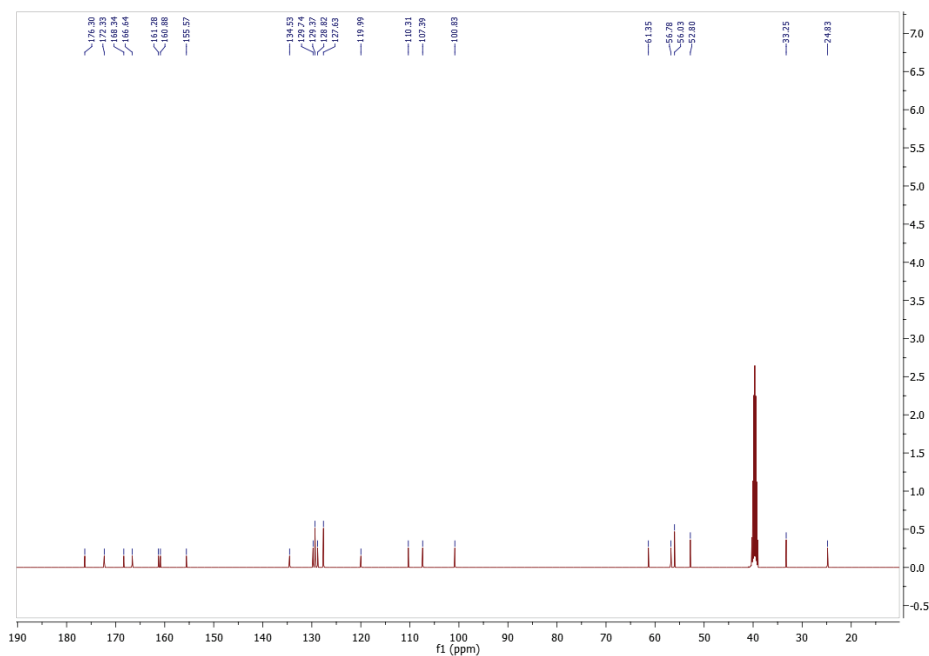
$^{13}\text{C-NMR}$ for N'-(2,4-dimethoxybenzylidene)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)acetohydrazide. (5)



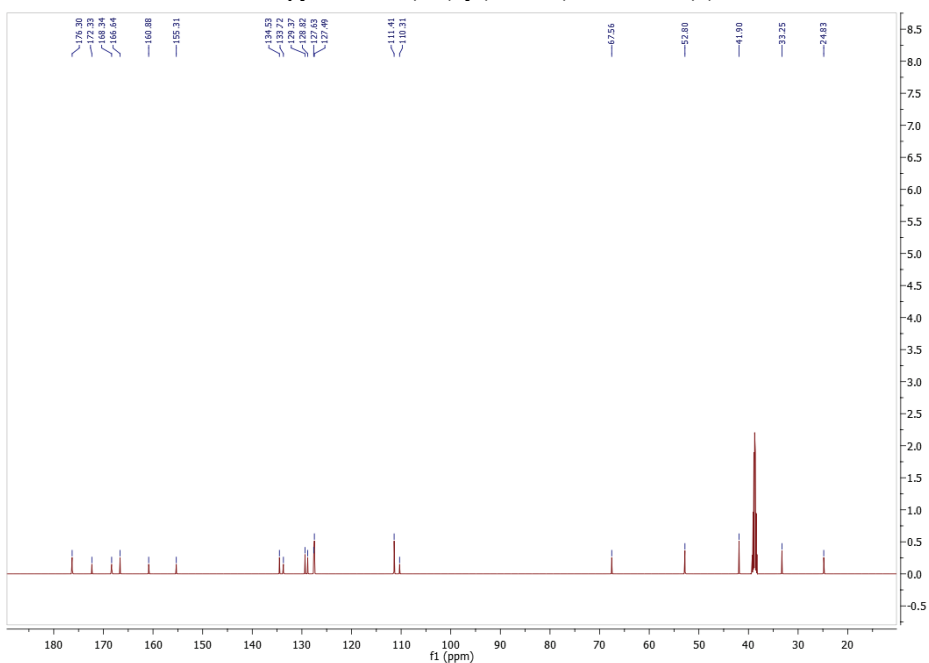
^{13}C -NMR for N' -(4-(dimethyl amino) benzylidene)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)aceto hydrazide (6)



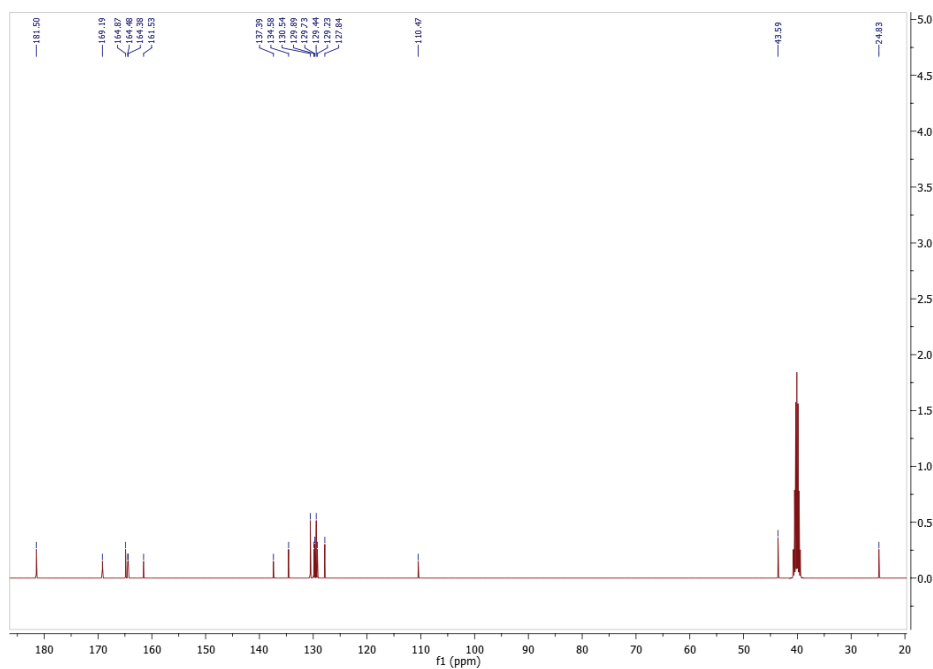
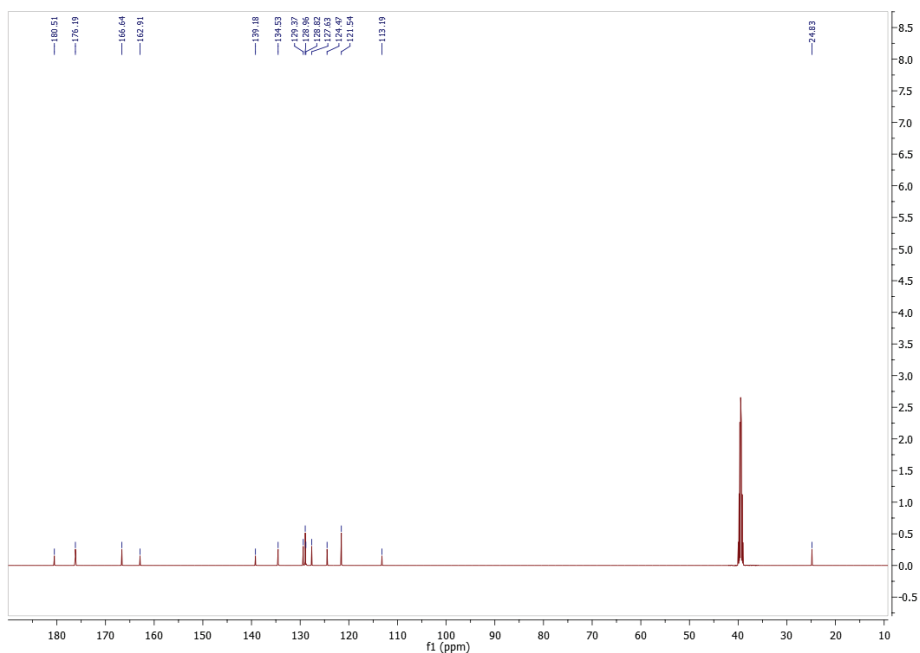
^{13}C -NMR for 2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino)- N -(2-(4-nitrophenyl)-4-oxothiazolidin-3-yl) acetamide (7)

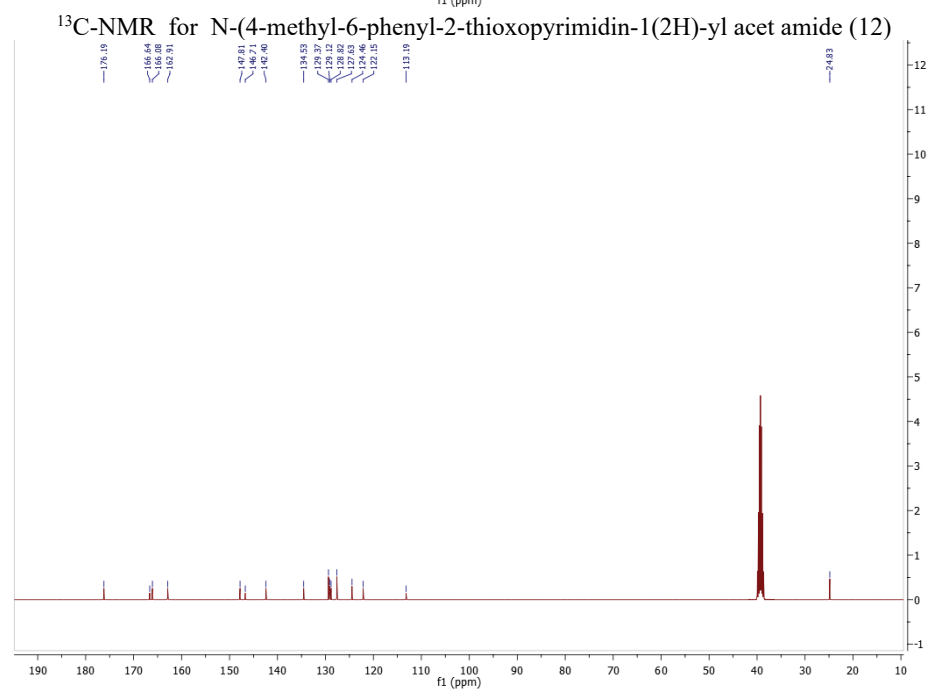
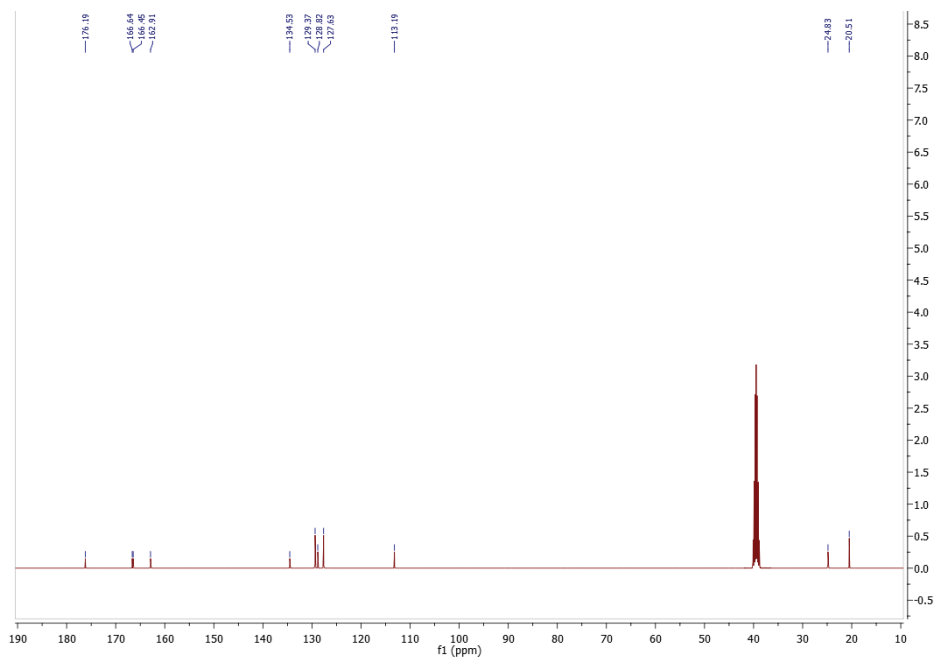


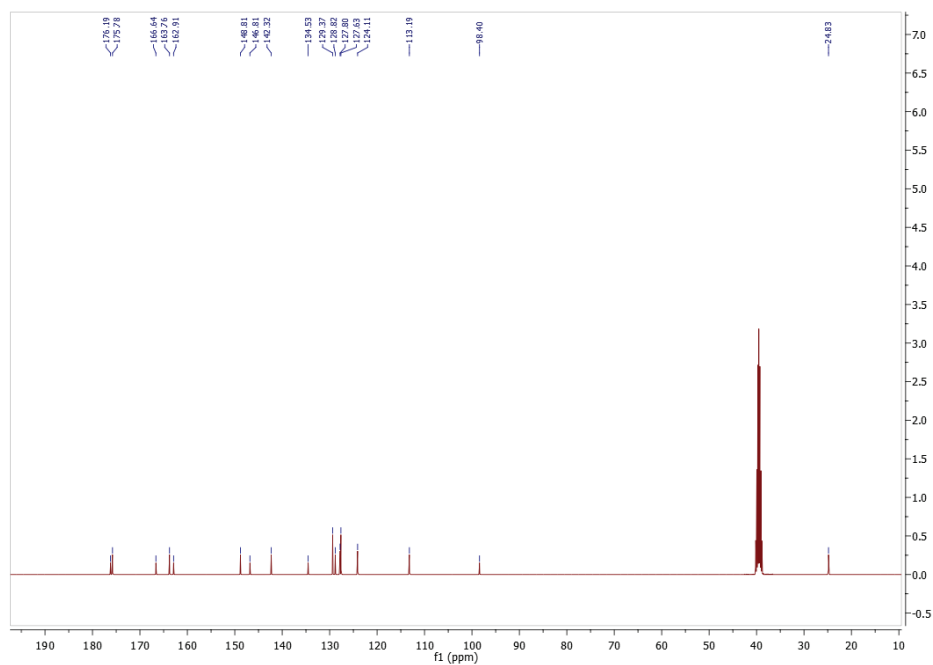
¹³C-NMR for dimethoxyphenyl)-4-oxothiazolidin-3-yl)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino) acetamide (8)



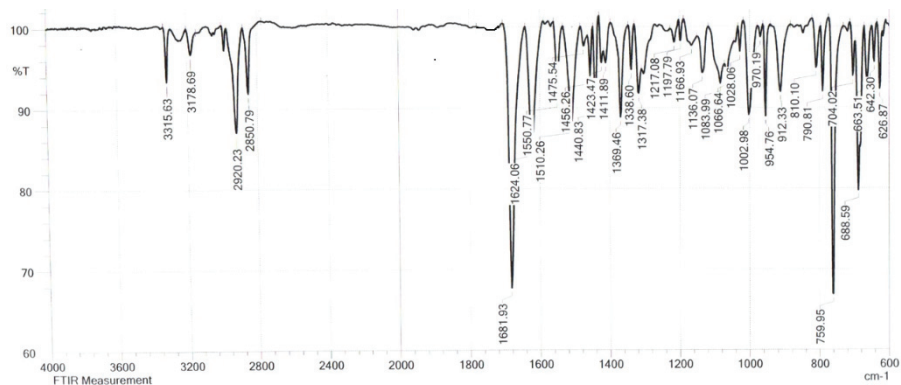
¹³C-NMR for N-(2-(4-(dimethylamino)phenyl)-4-oxothiazolidin-3-yl)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)acetamide (9)



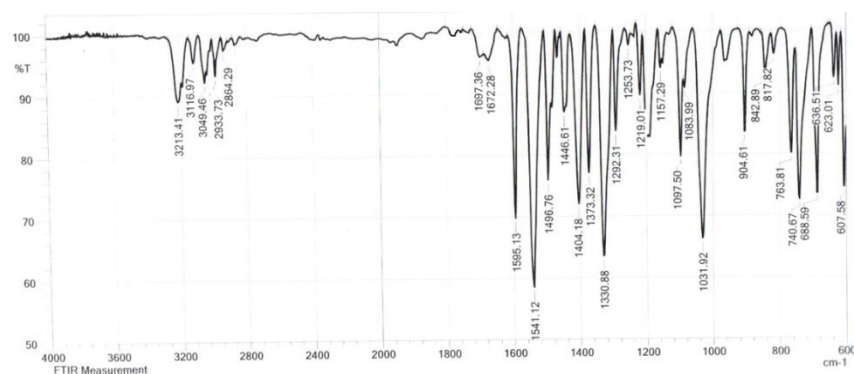




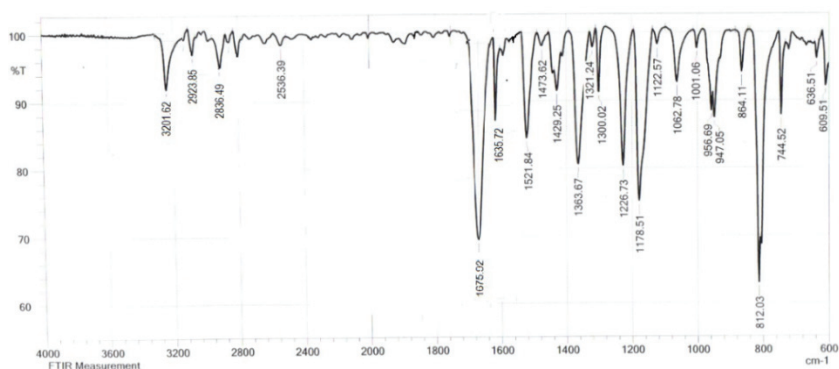
^{13}C -NMR for 4-methyl-1-((6-(4-nitrophenyl)-2-thioxo-1,2-dihydropyrimidin-4-yl) amino)-6-phenylpyrimidine-2(1H)-thione (14)



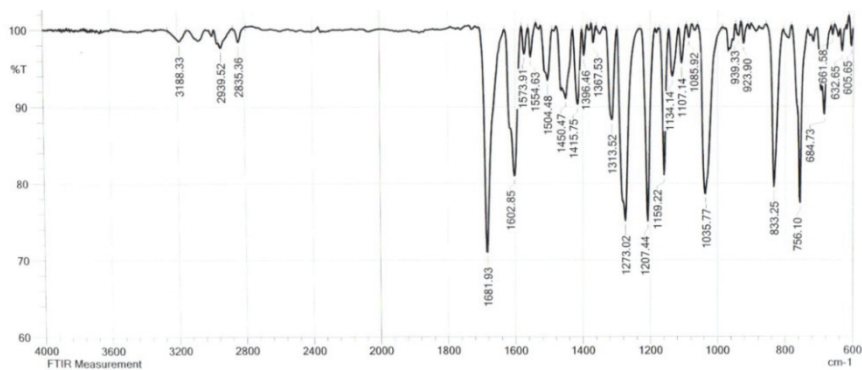
IR spectrum for 2-((4-Methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino) aceto hydrazide (3)



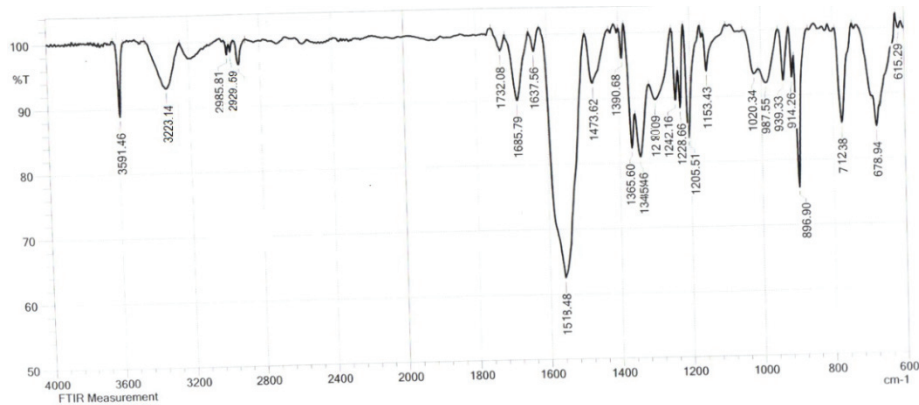
IR spectrum for 2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)-N'-(4-nitrobenzylidene)acetohydrazide. (4)



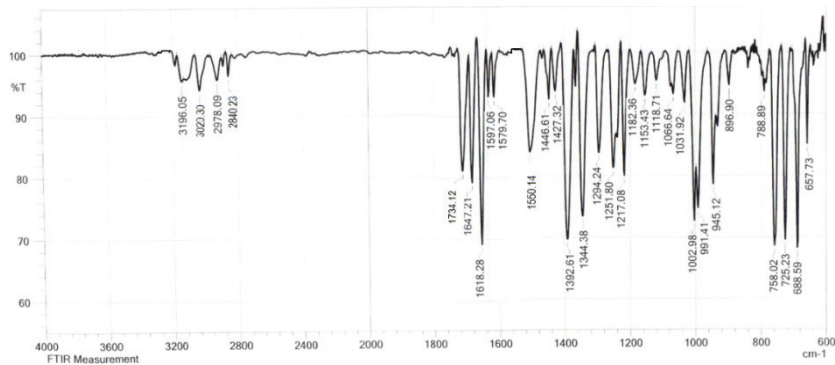
IR spectrum for N'-(2,4-dimethoxybenzylidene)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)acetohydrazide.(5)



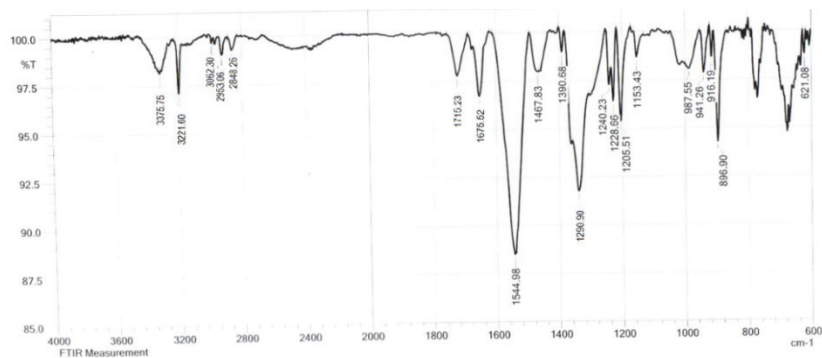
IR spectrum for N'-(4-(dimethyl amino) benzylidene)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)amino)aceto hydrazide (6)



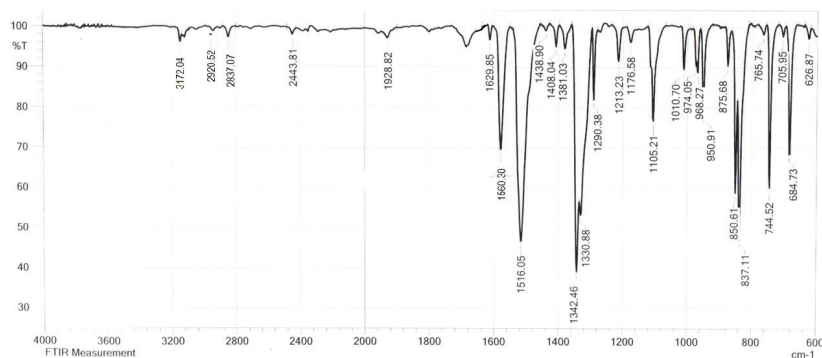
IR spectrum for 2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino)-N-(2-(4-nitrophenyl)-4-oxothiazolidin-3-yl) acetamide (7)



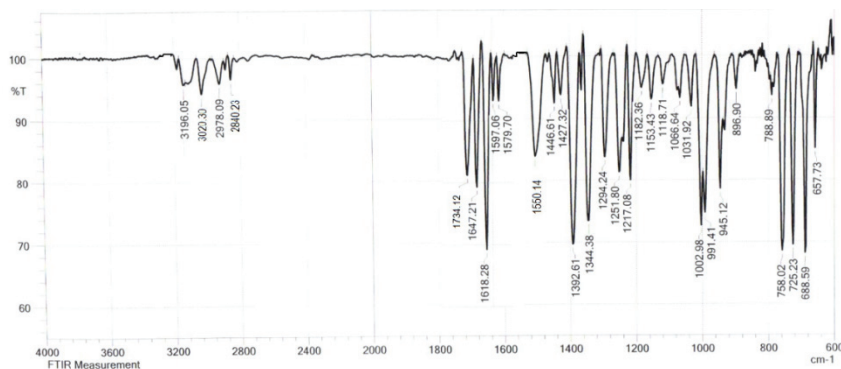
IR spectrum for dimethoxyphenyl)-4-oxothiazolidin-3-yl)-2-((4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) amino) acetamide (8)



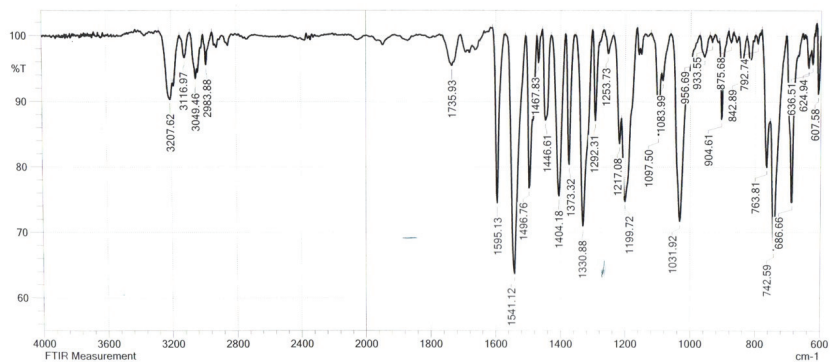
IR spectrum for N-(2-(4-(dimethylamino)phenyl)-4-oxothiazolidin-3-yl)-2-((4-methyl-6-phenyl-2-thioxypyrimidin-1(2H)-yl)amino)acetamide (9)



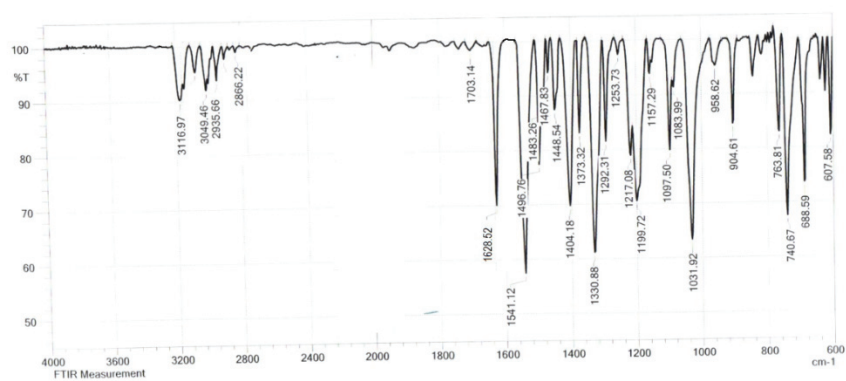
IR spectrum for 1-(4-methyl-6-phenyl-2-thioxypyrimidin-1(2H)-yl)-3-phenylthiourea (10)



IR spectrum for 5-(4-methyl-6-phenyl-2-thioxypyrimidin-1(2H)-yl)-1-phenyl-6-thioxopiperidine-2,4-dione(11)



IR spectrum for N-(4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl) acet amide (12)



IR spectrum for N-(4-methyl-6-phenyl-2-thioxopyrimidin-1(2H)-yl)-3-(4-nitrophenyl) acrylamide (13)