

Supporting Information

Nano-[Fe₃O₄@SiO₂/N-propyl-1-(thiophen-2-yl)ethanimine][ZnCl₂] as a Nano magnetite Schiff base complex and heterogeneous catalyst for the synthesis of pyrimido[4,5-b]quinolones

Soheila Esmaili,^a Ardeshir Khazaei,^{*a} Ahmad Reza Moosavi-Zare,^{*b}

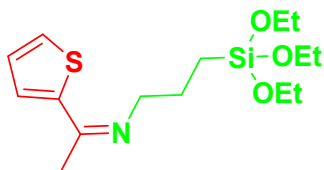
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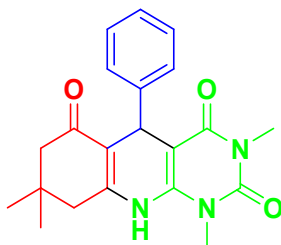
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Data of compounds



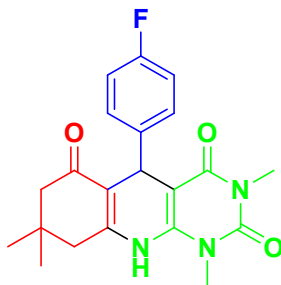
(Z)-1-(thiophen-2-yl)-N-(3-(triethoxysilyl)propyl)ethan-1-imine (1)

IR (KBr, cm^{-1}): 2925, 2885, 1663, 1620/ 1432, 1280, 1124, 705; ^1H NMR (300 MHz, DMSO- d_6): δ 0.68 (t, 2H), 1.05-1.1 (m, 4H), 2.56 (s, 9H), 3.19 (s, 3H), 3.43-3.5 (m, 6H), 7.24-7.28(m, 1H), 7.96 (d, $J=6$, 1H), 8.01 (d, $J=3$, 1H).



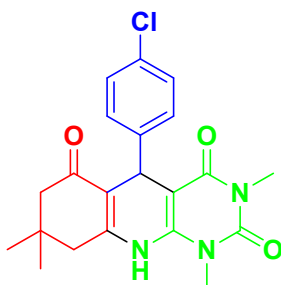
1,3,8,8-tetramethyl-5-phenyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S1)

IR (KBr, cm^{-1}): 3363, 3203, 2955, 2932, 1700, 1655, 1614, 1507, 1363, 1257; ^1H NMR (300 MHz, DMSO- d_6): δ 1.06 (s, 3H), 1.15 (s, 3H), 2.31 (s, 2H), 2.41(s, 2H), 3.12 (s, 3H), 3.38 (s, 3H), 5.5 (s, 1H), 7.12 (d, $J = 6.00$ Hz, 2H), 7.23 (d, $J = 6.00$ Hz, 2H), 7.32 (s, 1H), 12.84 (s, 1H); ^{13}C -NMR (DMSO- d_6 , 300 MHz): δ 27.5, 28.4, 29.3, 30.8, 31.6, 33.4, 44.0, 49.9, 88.3, 114.2, 125.7, 126.8, 128.4, 140.2,150.6, 155.1, 16.1, 176.5, 199.8.



5-(4-fluorophenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S2)

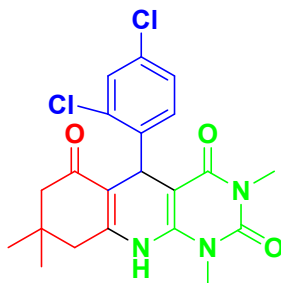
IR (KBr, cm^{-1}): 3361, 3258, 3217, 2963, 2934, 3895, 1692, 1655, 1593, 1505, 1219, 1157, 761, 507; ^1H NMR (300 MHz, DMSO- d_6): δ 1.05 (s, 3H), 1.13 (s, 3H), 2.31 (s, 2H), 2.41 (s, 2H), 3.12 (s, 3H), 3.38 (s, 3H), 5.47 (s, 1H), 7.06-7.12 (m, 3H), (7.33 s, 1H), 12.85 (s, 1H) ^{13}C -NMR (DMSO- d_6 , 300 MHz): δ 27.5, 28.5, 29.3, 30.8, 31.6, 32.9, 44.0, 49.9, 88.2, 114.2, 115.1, 128.5, 136.1, 150.6, 155.1, 159.1, 162.3, 164.1, 176.5, 199.8.



5-(4-chlorophenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S3)

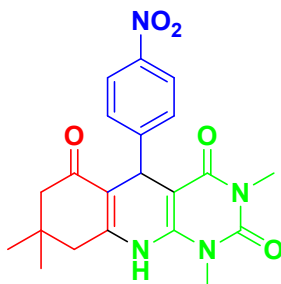
IR (KBr, cm^{-1}): 3405, 3363, 3226, 2961, 2941, 2885, 1696, 1655, 1595, 152, 1509, 1362, 1257, 1014, 925, 784, 500; ^1H NMR (300 MHz, DMSO- d_6): δ 1.05 (s, 3H), 1.13 (s, 3H), 2.29 (s, 2H), 2.39 (s, 2H), 3.12 (s, 3H), 3.37 (s, 3H), 5.39 (s, 1H), 6.63 (d, $J = 6.00$ Hz, 2H), 6.88 (d, $J = 6.00$

Hz, 2H), 7.29 (s, 1H), 12.84 (s, 1H); ¹³C-NMR (DMSO-*d*₆, 300 MHz): δ 27.5, 28.4, 29.3, 30.7, 31.5, 32.6, 44.0, 50.0, 88.6, 114.5, 115.2, 127.7, 129.9, 150.6, 154.9, 155.3, 164.0, 176.4, 199.8.



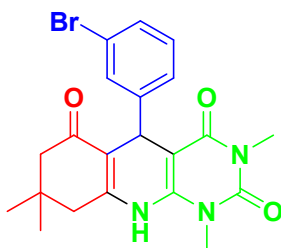
5-(2,4-dichlorophenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S4)

IR (KBr, cm⁻¹): 3280, 3221, 3093, 2957, 2931, 2875, 1704, 1661, 1643, 1494, 1379, 1211, 1149, 1046, 962, 845, 755; ¹H NMR (300 MHz, DMSO-*d*₆): δ 0.90 (s, 3H), 1.05 (s, 3H), 1.98 (d, *J* = 15.0 Hz, 1H), 2.21 (d, *J* = 15.0 Hz, 1H), 2.57 (s, 2H), 3.06 (s, 3H), 3.46 (s, 3H), 5.14 (s, 1H), 7.28-7.35 (m, 3H), 9.05 (s, 1H); ¹³C-NMR (DMSO-*d*₆, 300 MHz): δ 26.7, 28.0, 29.6, 30.7, 32.4, 34.0, 50.4, 89.6, 110.8, 126.9, 128.7, 131.3, 133.6, 134.0, 143.0, 144.6, 150.5, 150.9, 160.9, 194.8.



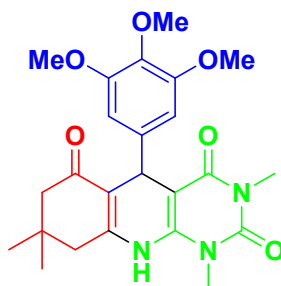
5-(4-nitrophenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S5)

IR (KBr, cm^{-1}): 3367, 3337, 3203, 2961, 2911, 1697, 1671, 1592, 1512, 1412, 1344, 1253, 1045, 748, 583; ^1H NMR (300 MHz, DMSO-d_6): δ 1.06 (s, 3H), 1.14 (s, 3H), 2.33 (s, 2H), 2.43 (2H), 3.12 (s, 3H), 3.38 (s, 3H), 5.60 (s, 1H), 7.35-7.40 (m, 2H), 8.12 (d, $J = 9$, 2H), 12.77 (s, 1H); ^{13}C -NMR ($\text{DMSO-}d_6$, 300 MHz): δ 27.7, 28.5, 29.2, 30.8, 31.6, 34.1, 43.9, 49.9, 87.6, 113.8, 123.6, 128.2, 145.9, 149.5, 150.6, 155.1, 164.1, 176.7, 199.7.



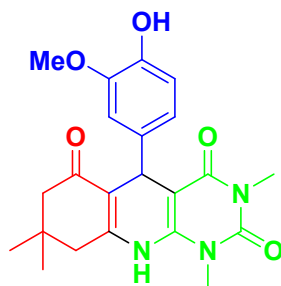
5-(3-bromophenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S6)

IR (KBr, cm^{-1}): 3410, 3163, 2961, 2933, 2872, 1697, 1593, 1507, 1355, 1303, 1244, 1204, 1145, 936, 778; ^1H NMR (300 MHz, DMSO-d_6): δ 1.06 (s, 3H), 1.13 (s, 3H), 2.31 (s, 2H), 2.42 (s, 2H), 3.13 (s, 3H), 3.37 (s, 3H), 5.51 (s, 1H), 7.13 (d, $J = 6.0$ Hz, 1H), 7.22 (t, $J = 6$ Hz, 1H), 7.27 (s, 1H), 7.32 (d, $J = 6$ Hz, 1H), 12.85 (s, 1H); ^{13}C NMR (300 MHz, DMSO-d_6): δ 27.4, 28.5, 29.3, 30.8, 31.6, 33.3, 43.9, 49.9, 87.7, 113.8, 122.0, 126.0, 128.7, 129.6, 130.5, 143.5, 150.6, 155.2, 164.1, 176.6, 199.7.



5-(3,4,5-trimethoxyphenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S7)

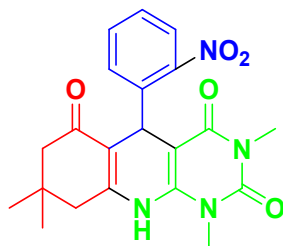
IR (KBr, cm^{-1}): 3421, 3191, 2997, 2944, 2875, 2839, 1703, 1656, 1600, 15.6, 1467, 1415, 1358, 1257, 1125, 998, 853, 786, 518, 490; ^1H NMR (300 MHz, DMSO- d_6): δ 1.07 (s, 3H), 1.16 (s, 3H), 2.31 (d, $J = 6.0$ Hz, 2H), 2.41 (d, $J = 15.0$ Hz, 2H), 3.15 (s, 3H), 3.65 (s, 3H), 3.67(s, 9H), 5.45 (s, 1H), 6.39(s, 1H), 7.30(s, 1H), 12.91 (s, 1H); ^{13}C -NMR (DMSO- d_6 , 300 MHz): δ 27.0, 28.5, 29.5, 30.8, 31.4, 33.5, 44.0, 49.9, 56.2, 60.4, 88.0, 104.4, 114.5, 135.7, 135.8, 150.6, 152.9, 155.0, 164.2, 176.7, 199.7.



5-(4-hydroxy-3-methoxyphenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S12)

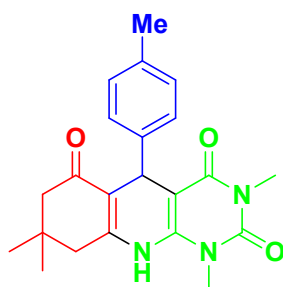
IR (KBr, cm^{-1}): 3426, 3302, 3225, 3080, 2961, 1703, 1661, 1644, 1615, 1379, 1359, 1208, 1126, 1034; ^1H NMR (300 MHz, DMSO- d_6): δ 0.94 (s, 3H), 1.06 (s, 3H), 2.06 (d, $J = 15.0$ Hz, 1H), 2.24 (d, $J = 18.0$ Hz, 1H), 2.58 (d, $J = 9.0$ Hz, 2H), 3.13 (s, 3H), 3.46 (s, 3H), 3.71(s, 3H), 4.81

(s, 1H), 6.59-6.6(s, 2H), 6.81(s, 1H), 8.67(s, 1H), 8.99(s, 1H); ^{13}C -NMR (DMSO- d_6 , 300 MHz): δ 26.8, 28.1, 29.7, 30.6, 32.5, 33.4, 39.9, 50.5, 55.9, 91.0, 112.4, 112.7, 115.3, 120.17, 138.1, 143.9, 145.2, 147.2, 149.7, 151.0, 161.2, 195.1.



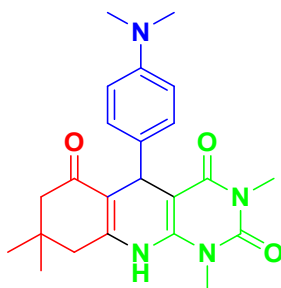
1,3,8,8-tetramethyl-5-(2-nitrophenyl)-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S8)

IR (KBr, cm^{-1}): 3472, 3271, 3216, 3096, 2964, 2951, 2875, 1698, 1667, 1648, 1531, 1498, 1379, 1353, 1244, 1214, 1149, 751, 534.



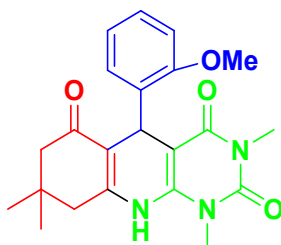
1,3,8,8-tetramethyl-5-(p-tolyl)-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S9)

IR (KBr, cm^{-1}): 3334, 3180, 2964, 2885, 1700, 1667, 1607, 1586, 1503, 1370, 1258, 1145, 863, 795, 507.



5-(4-(dimethylamino)phenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S10)

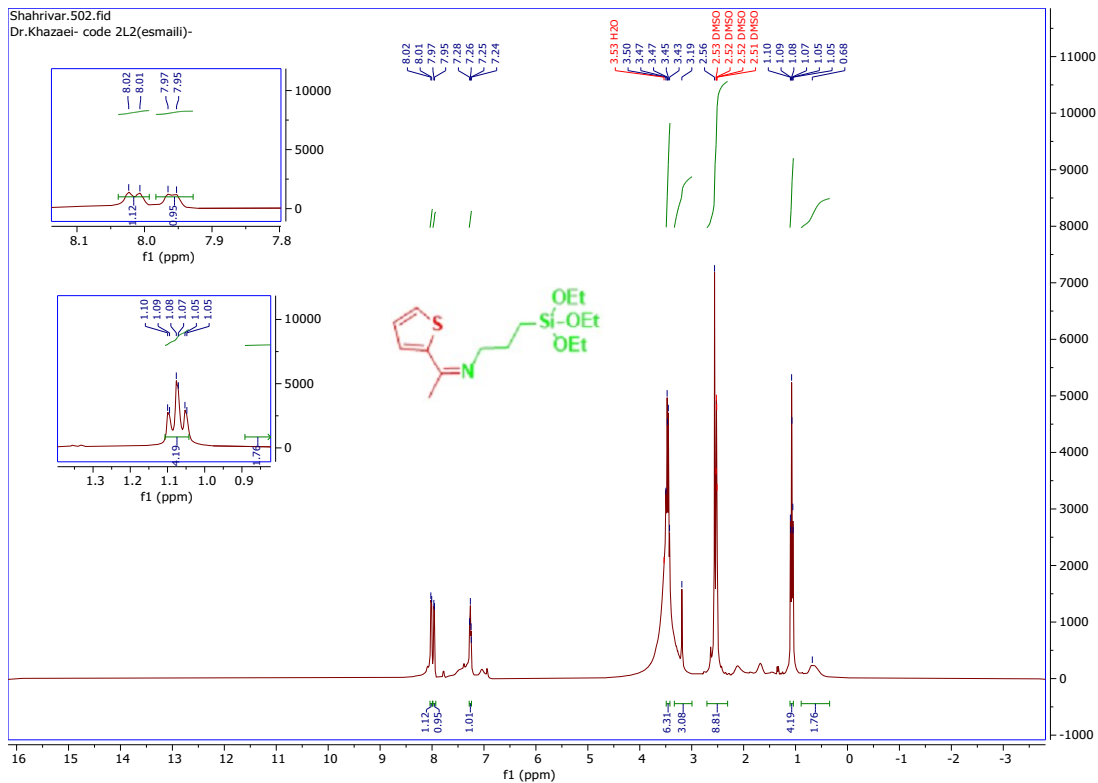
IR (KBr, cm^{-1}): 3396, 3283, 3227, 3096, 2957, 2885, 2806, 1701, 1662, 1643, 1610, 1500, 1444, 1379, 1357, 1242, 1208, 1149, 753.



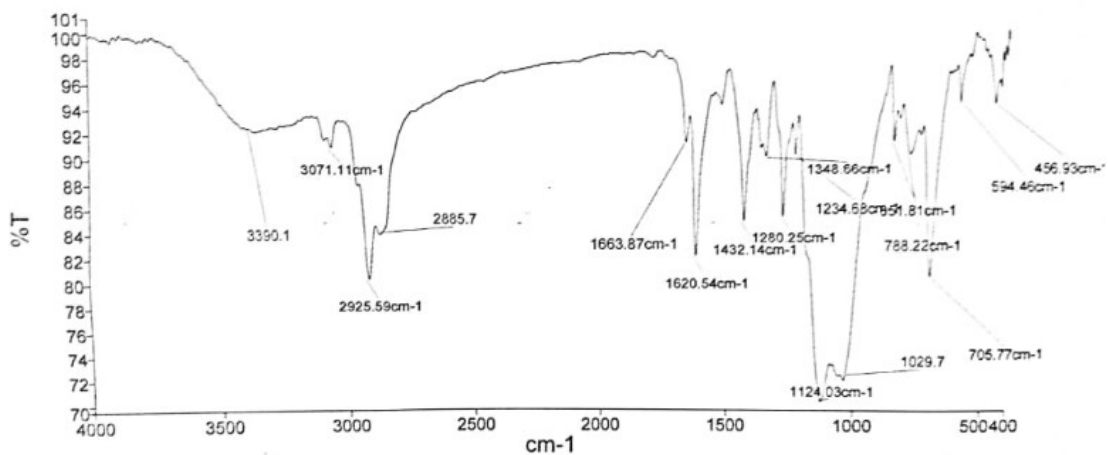
5-(2-methoxyphenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S11)

IR (KBr, cm^{-1}): 3436, 3279, 3212, 3076, 2953, 2834, 1698, 1655, 1637, 1497, 1380, 1289, 1212, 1117, 1051, 964, 859, 505

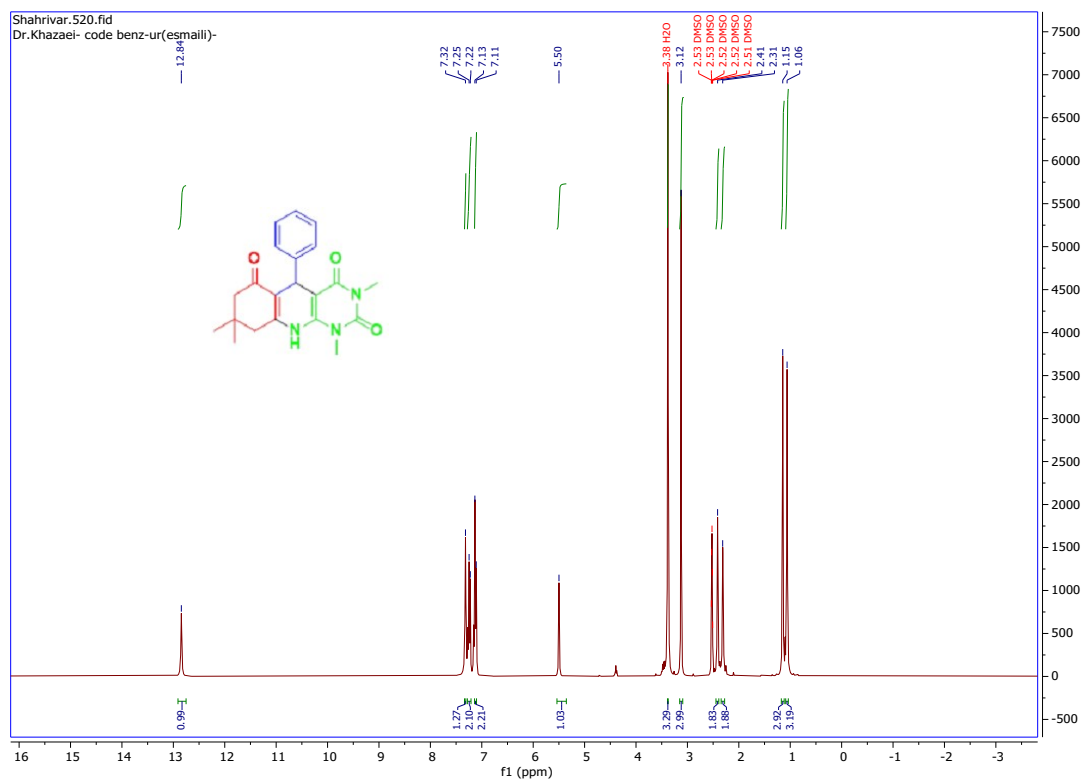
(Z)-1-(thiophen-2-yl)-N-(3-(triethoxysilyl)propyl)ethan-1-imine (1)

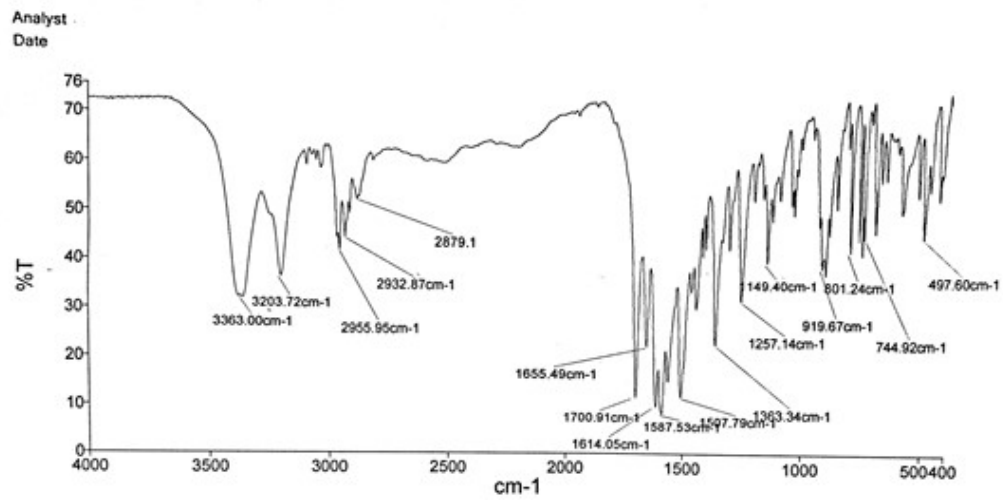
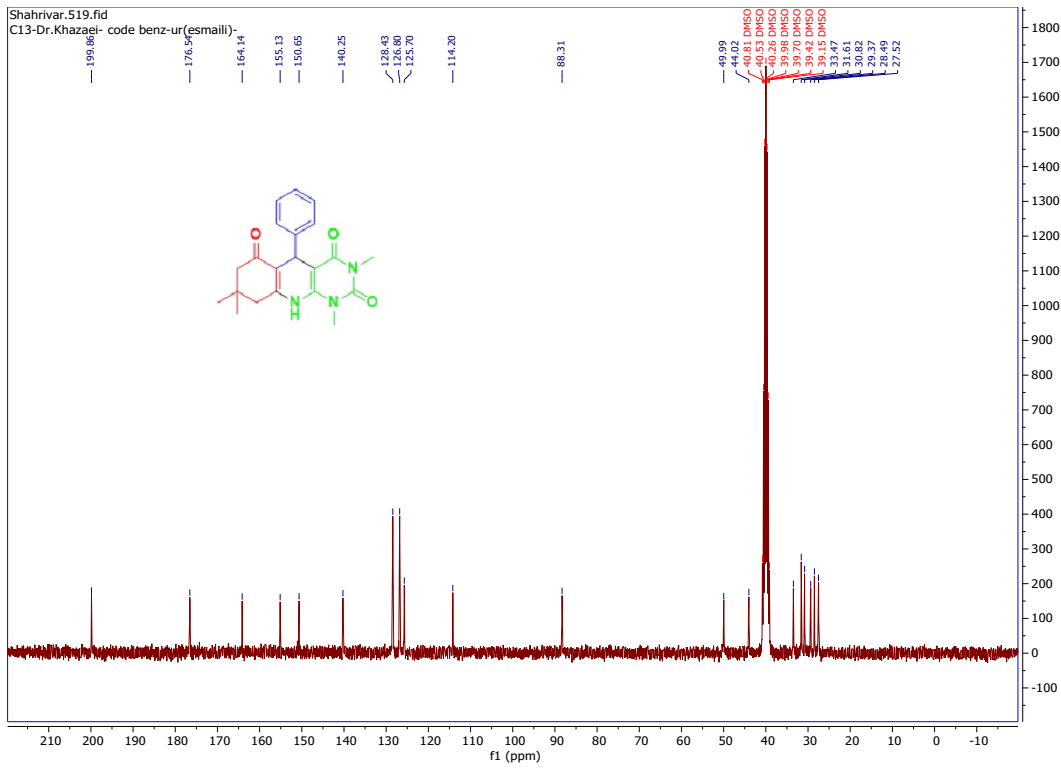


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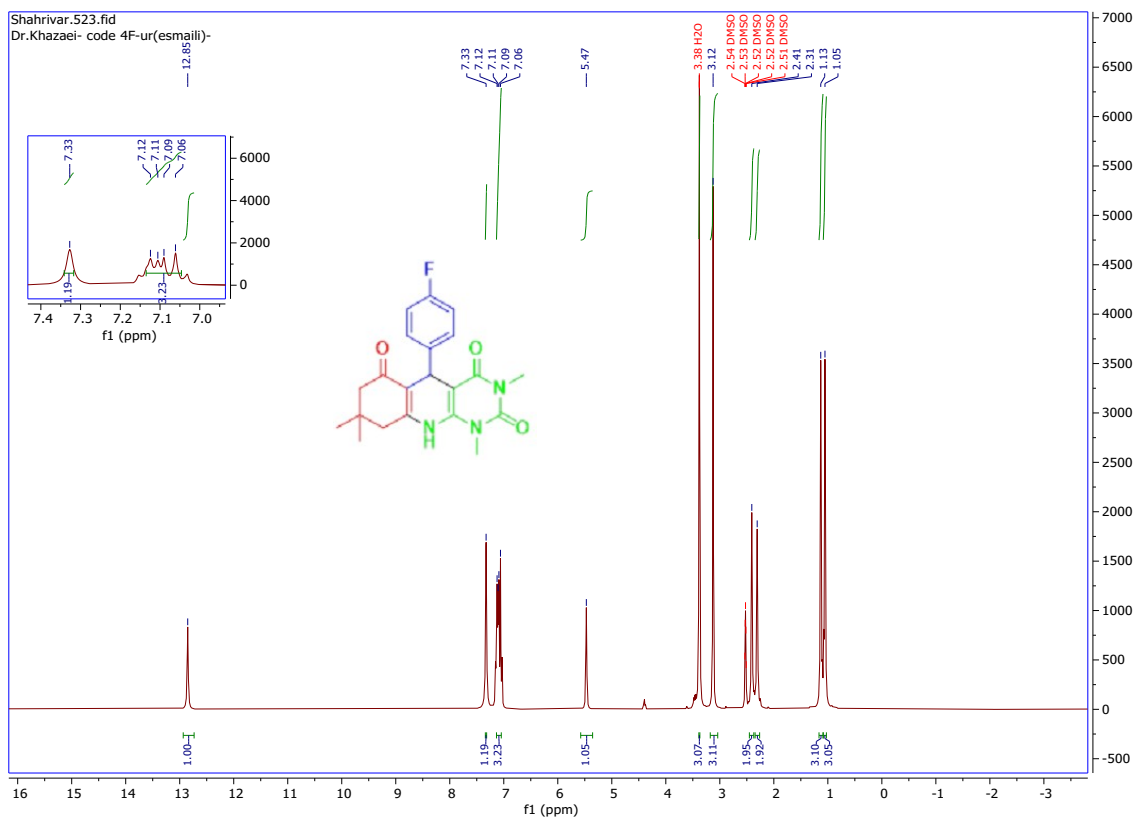


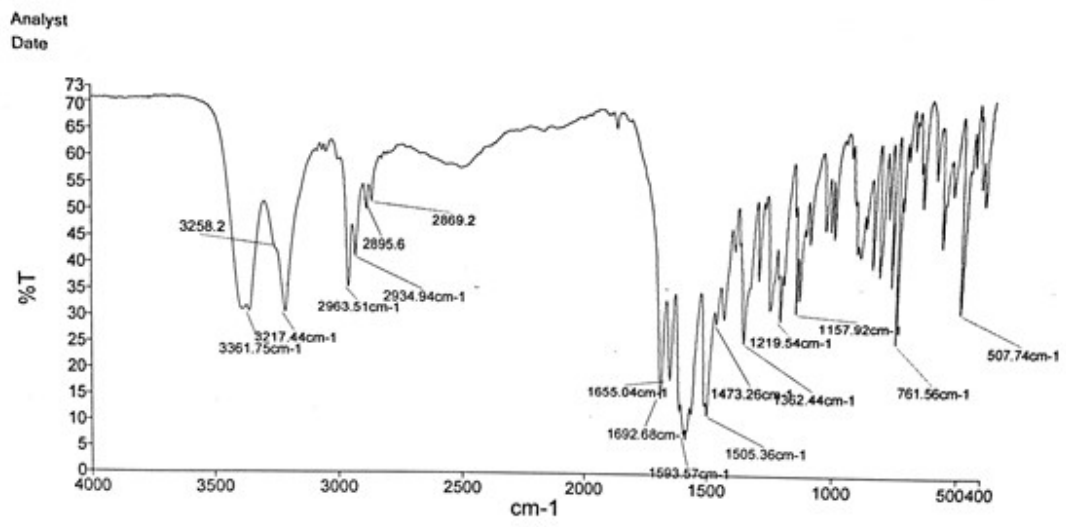
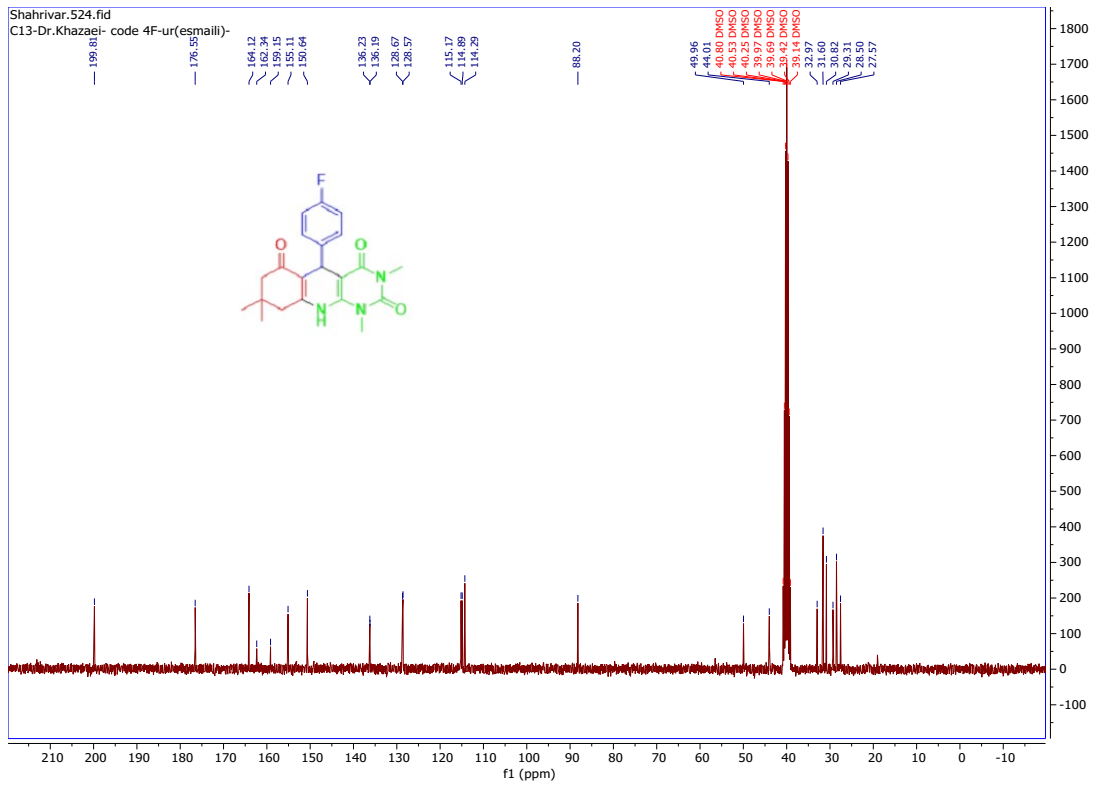
1,3,8,8-tetramethyl-5-phenyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S1)



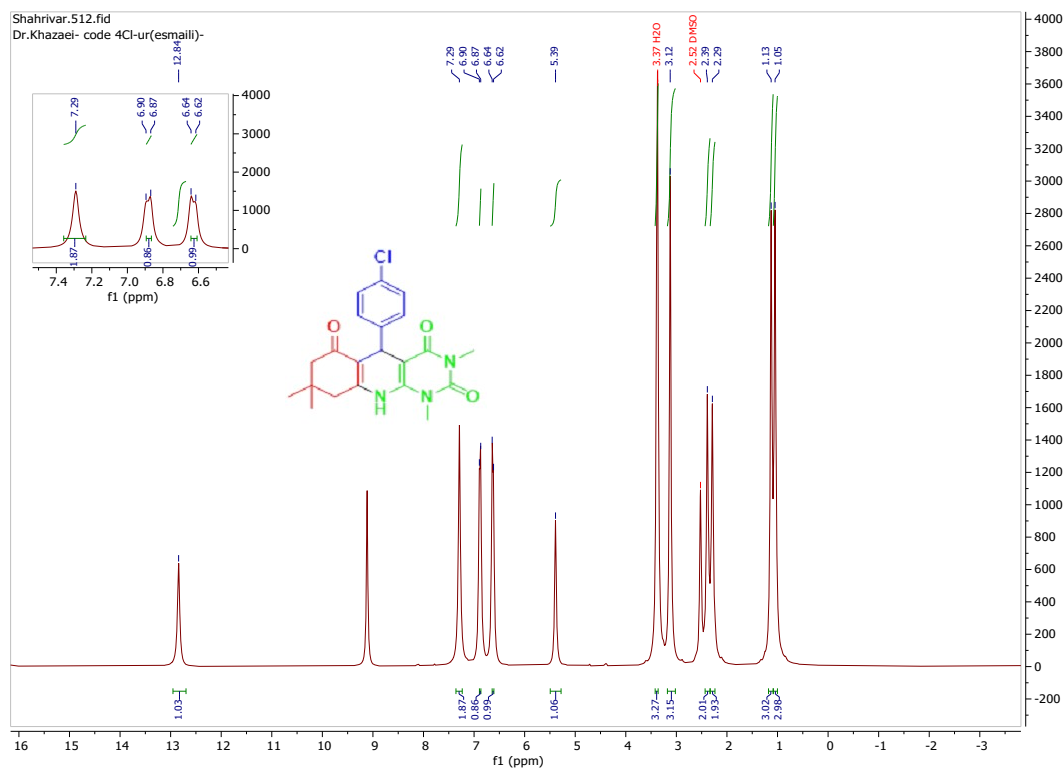


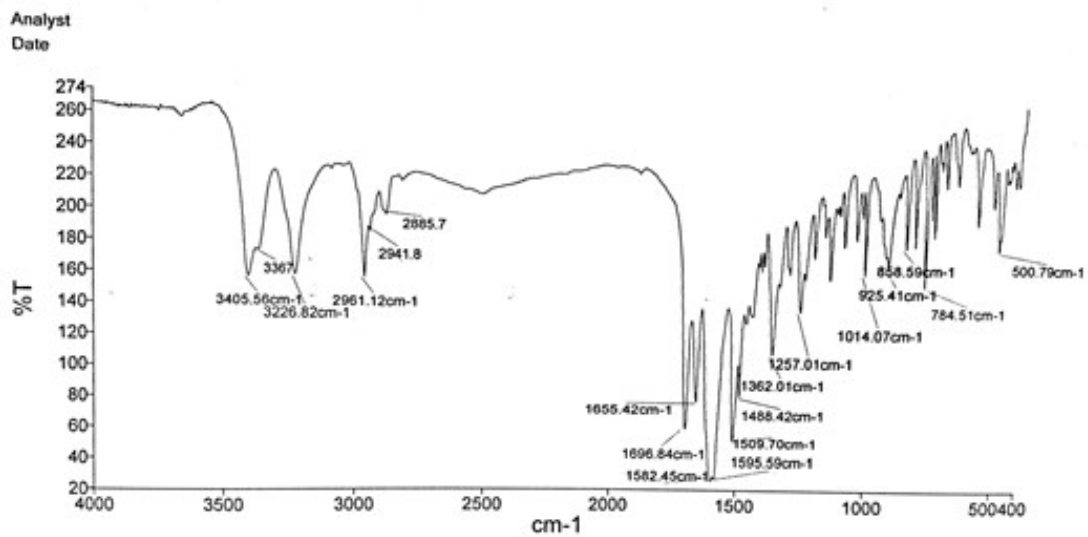
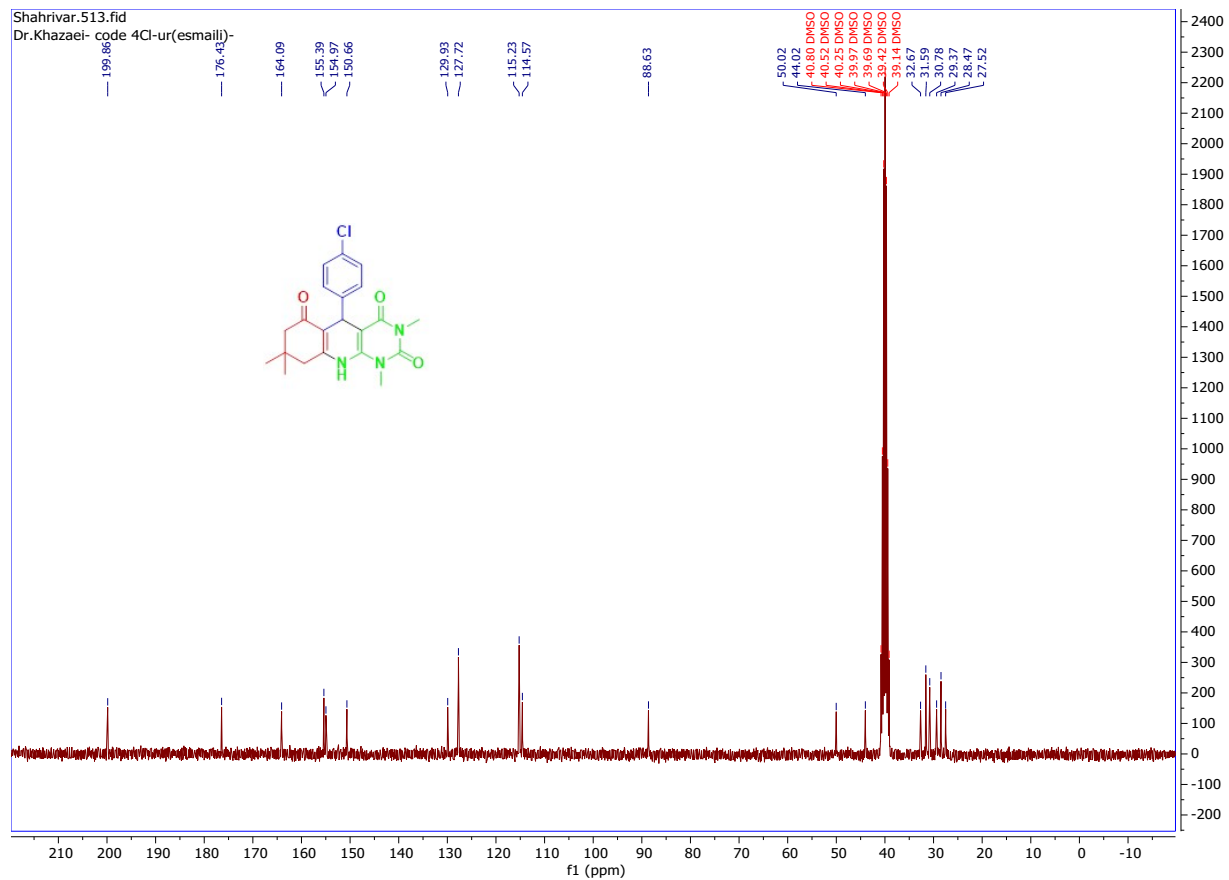
5-(4-fluorophenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S2)



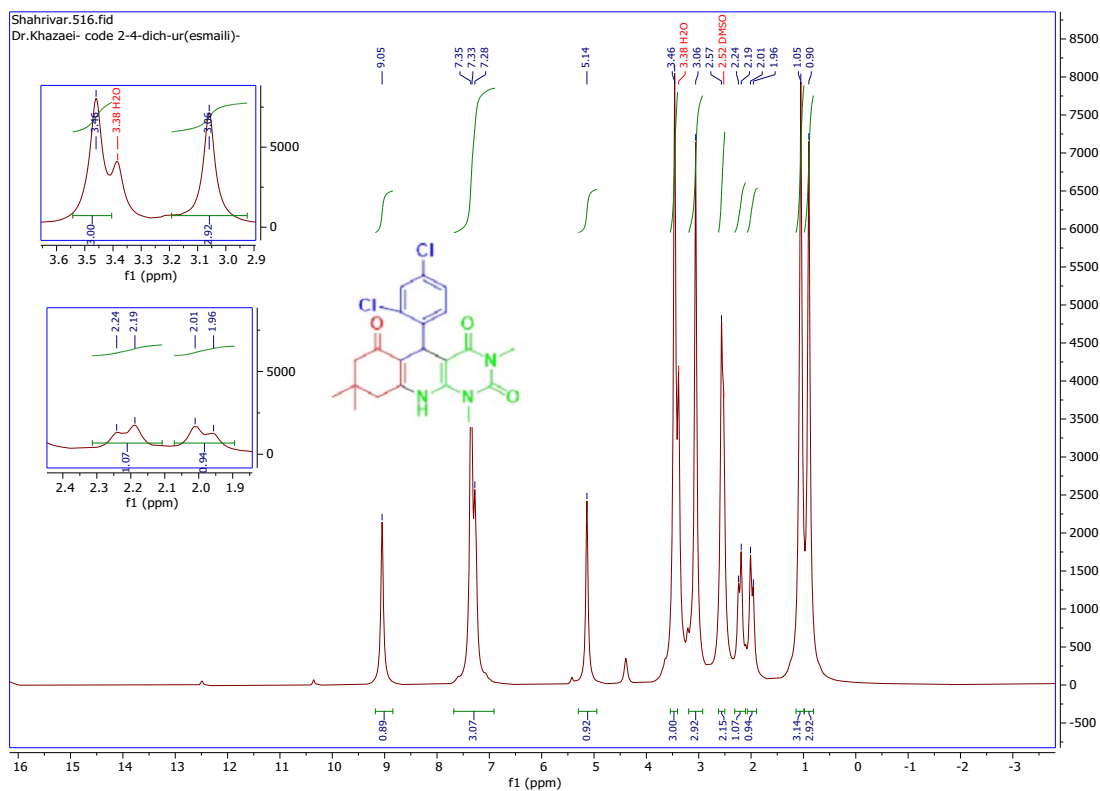


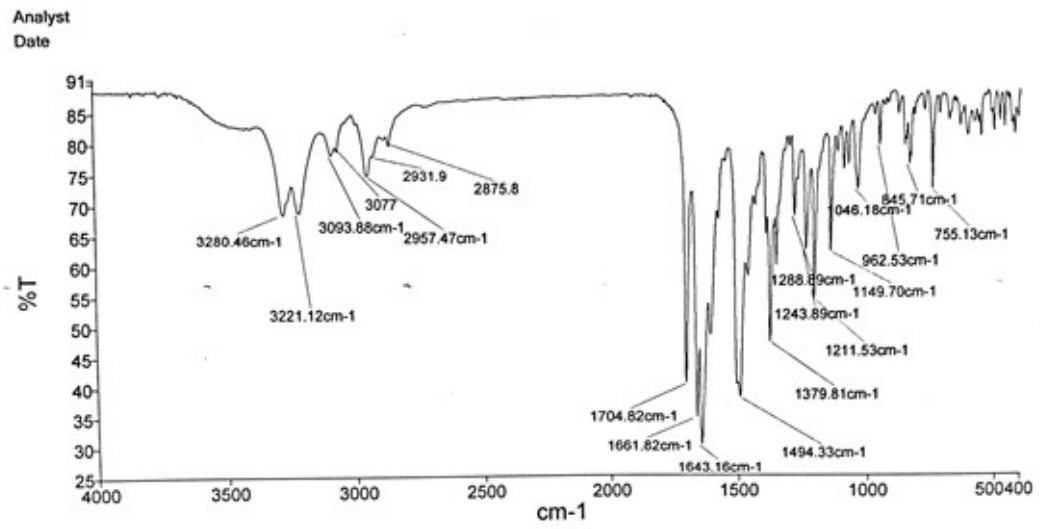
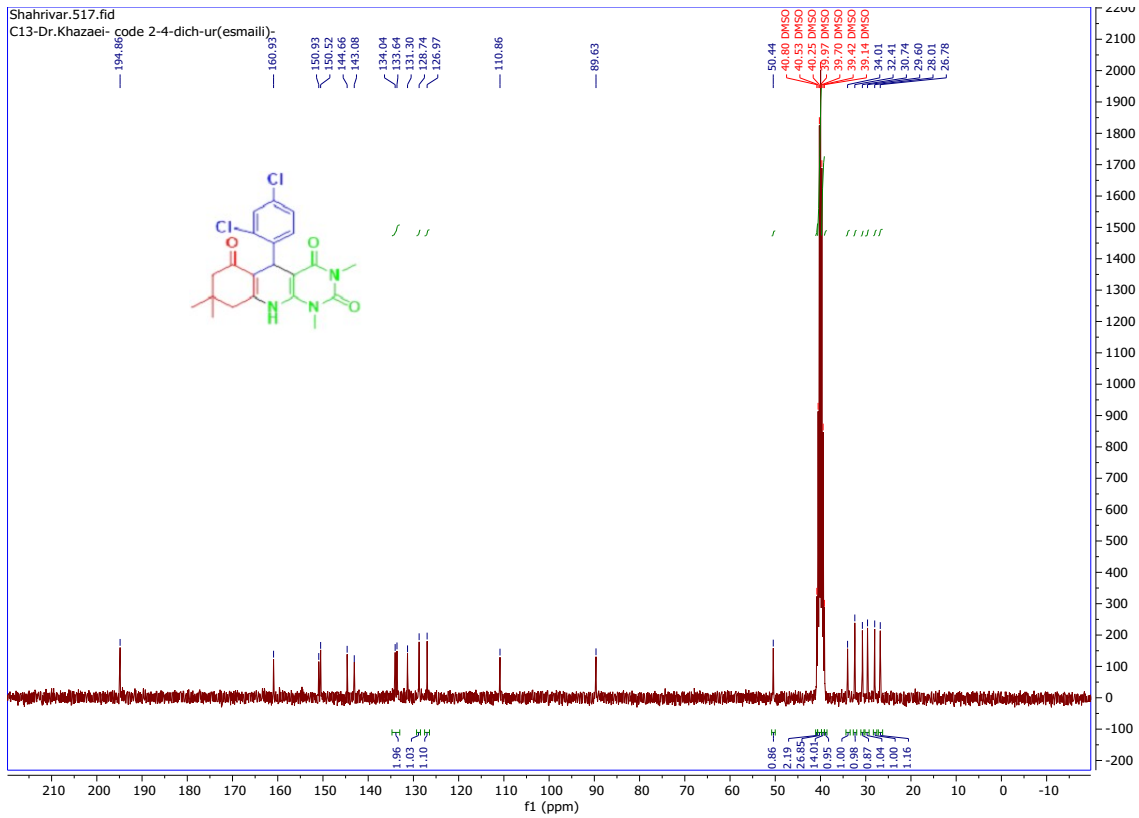
5-(4-chlorophenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S3)



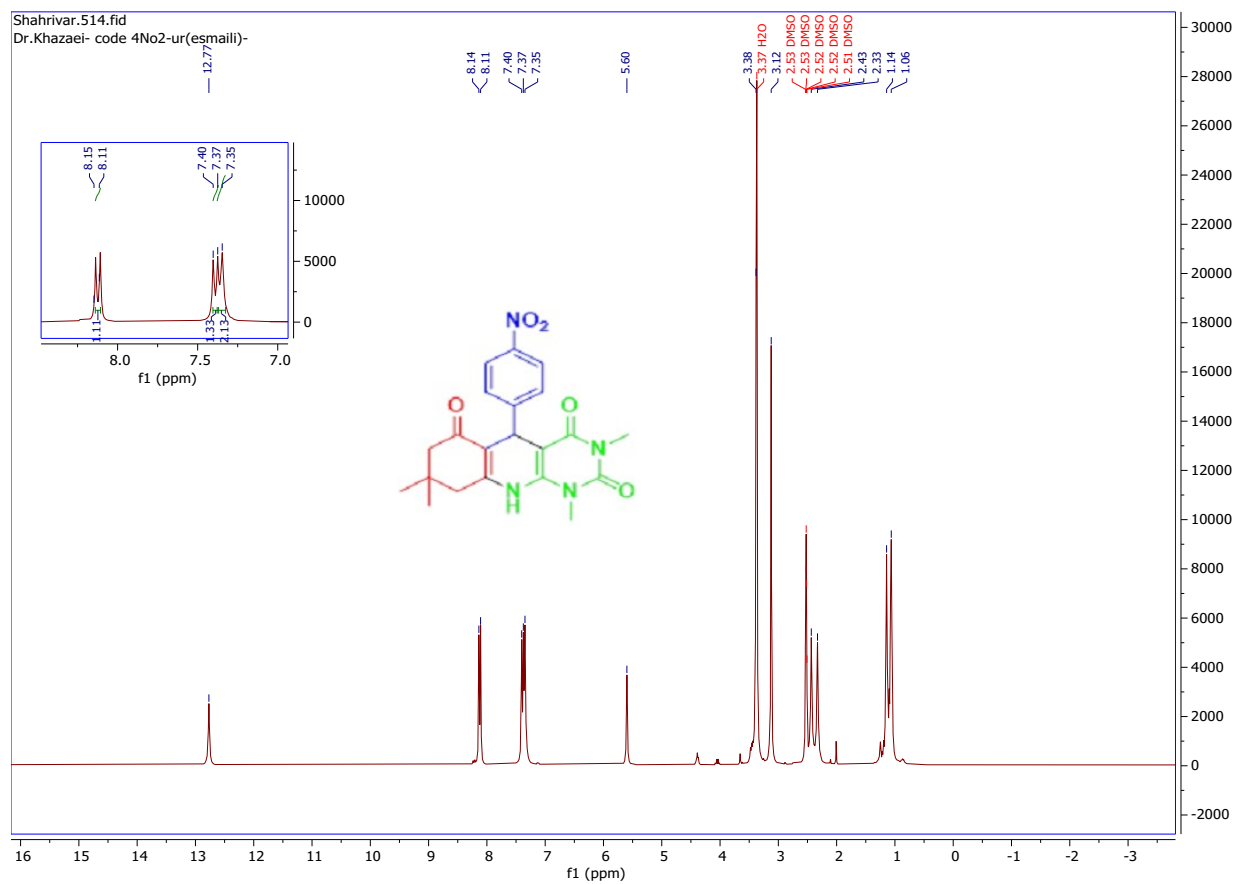


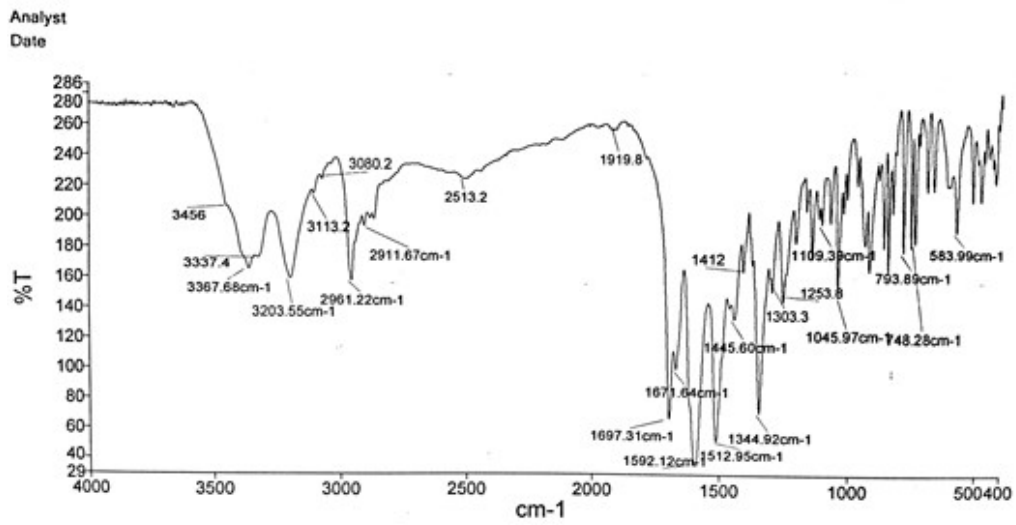
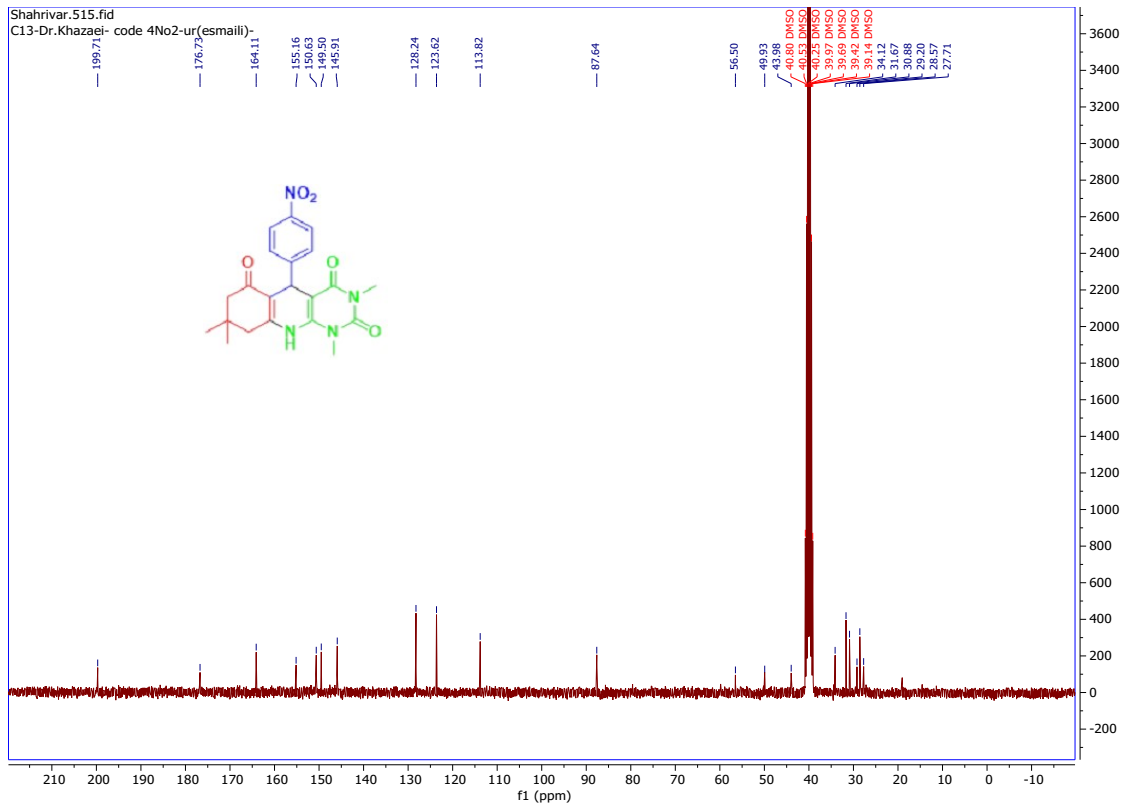
5-(2,4-dichlorophenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S4)



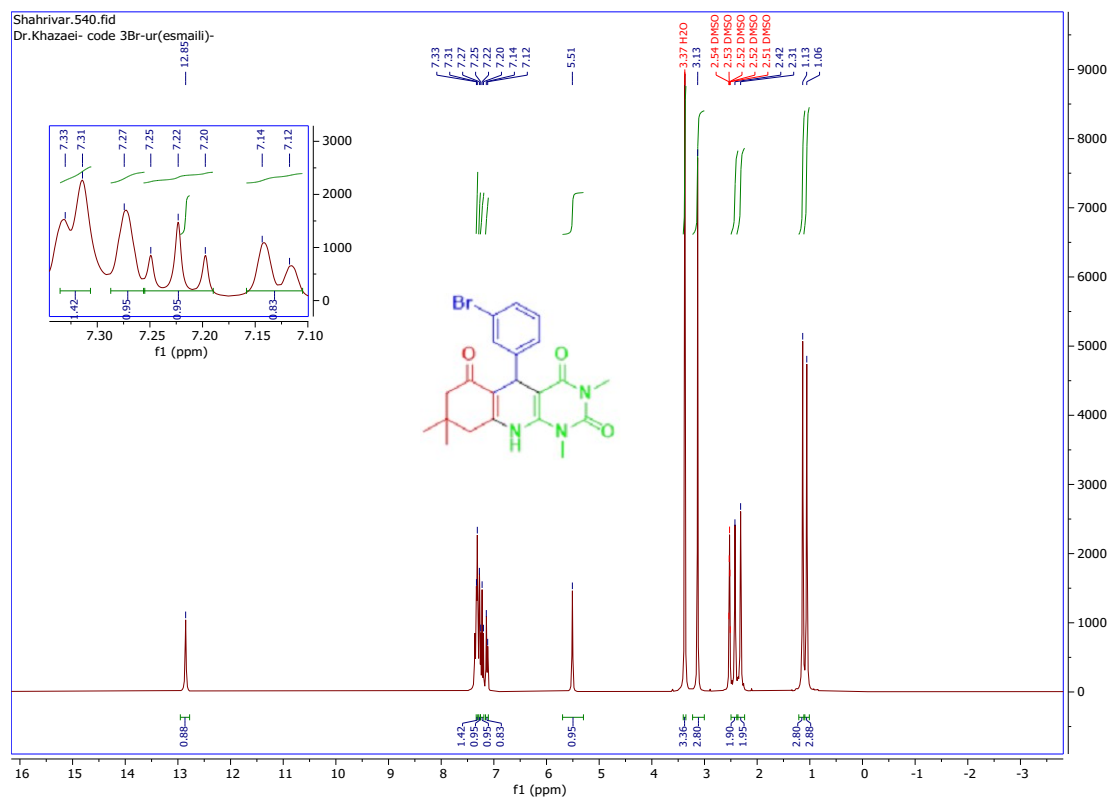


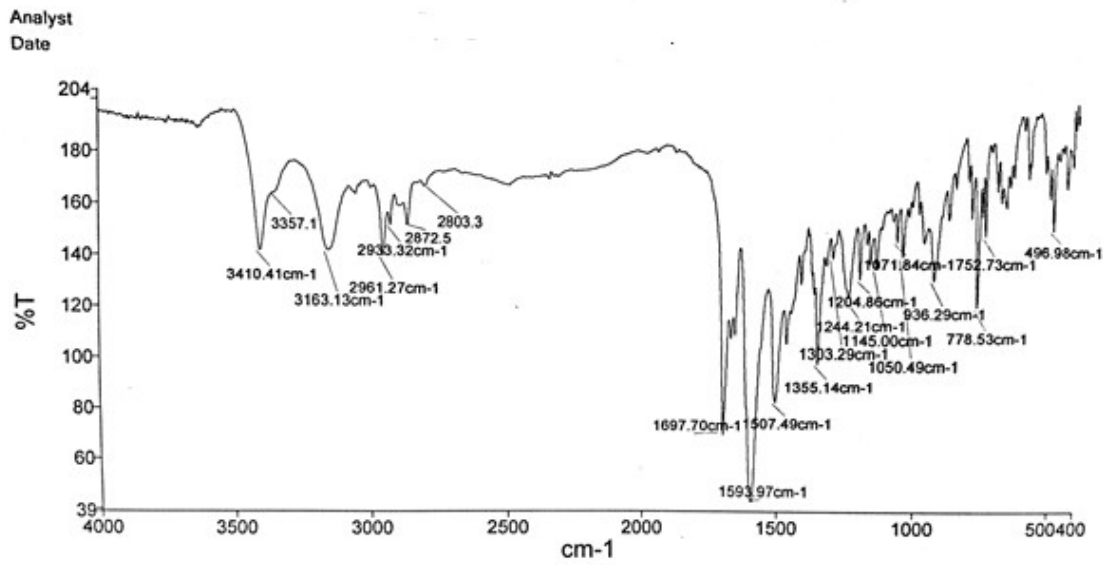
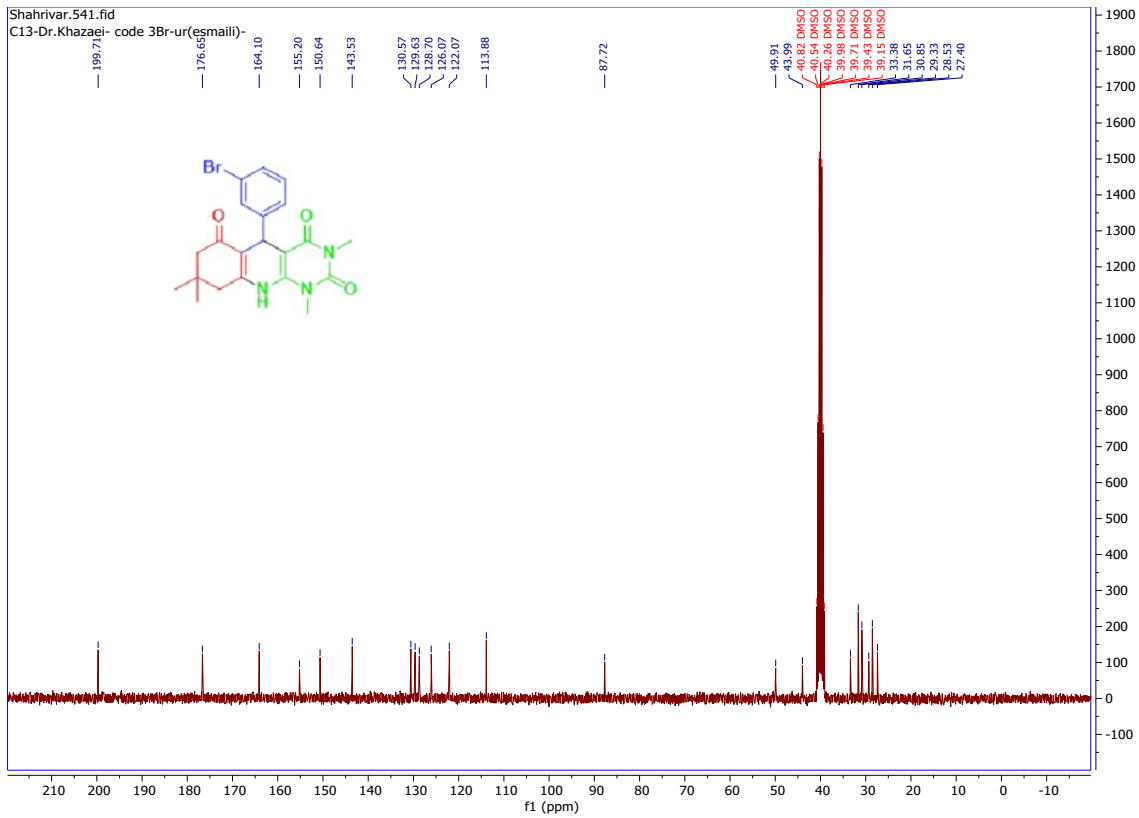
5-(4-nitrophenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trion (S5)



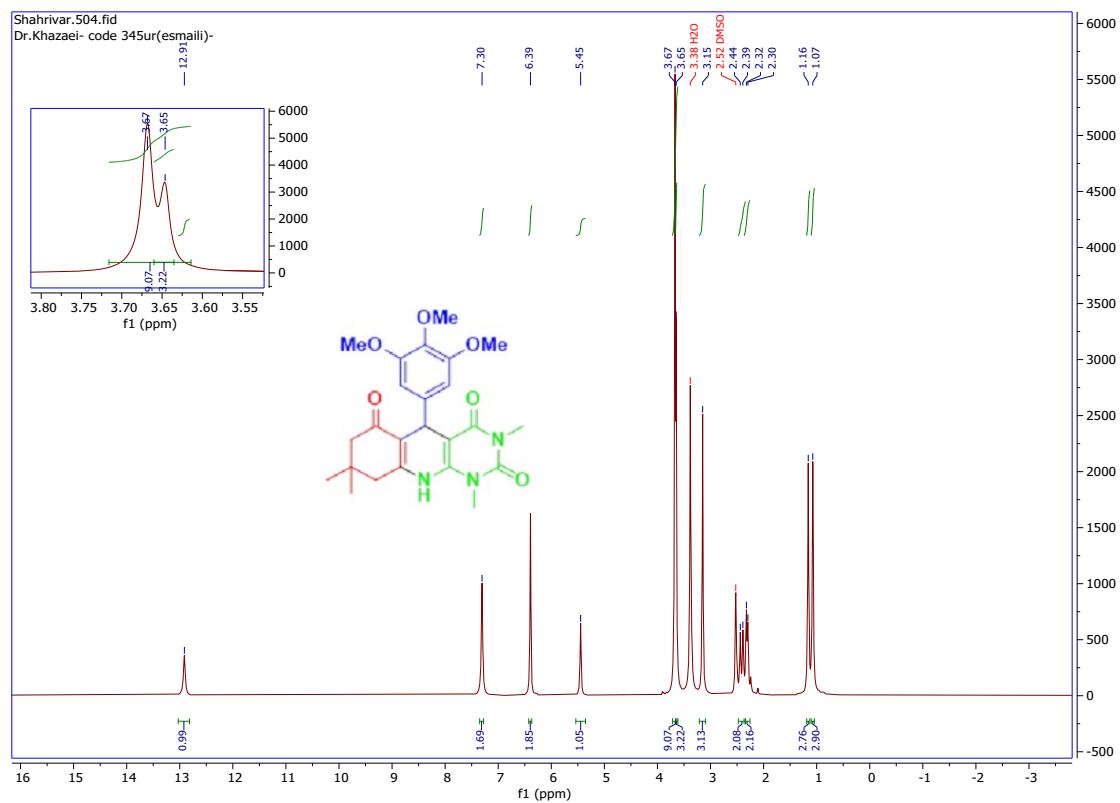


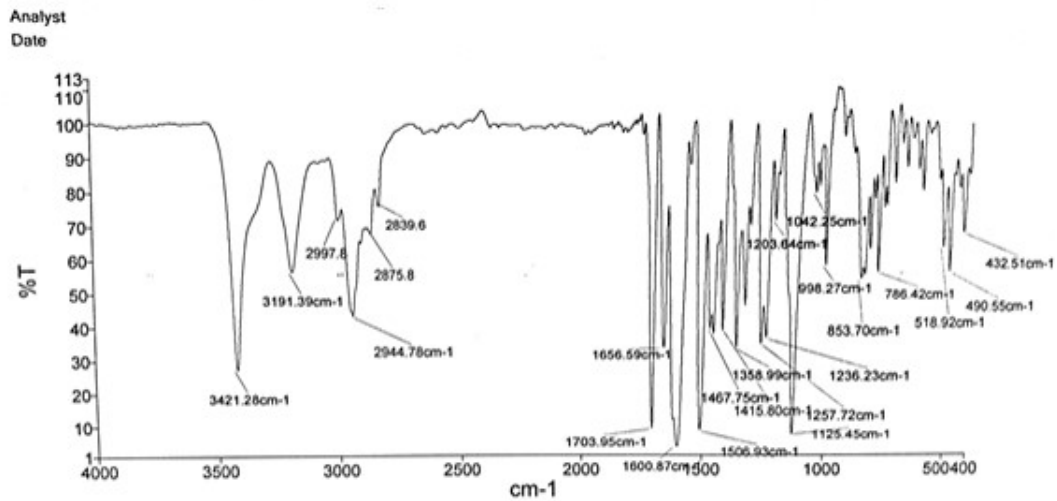
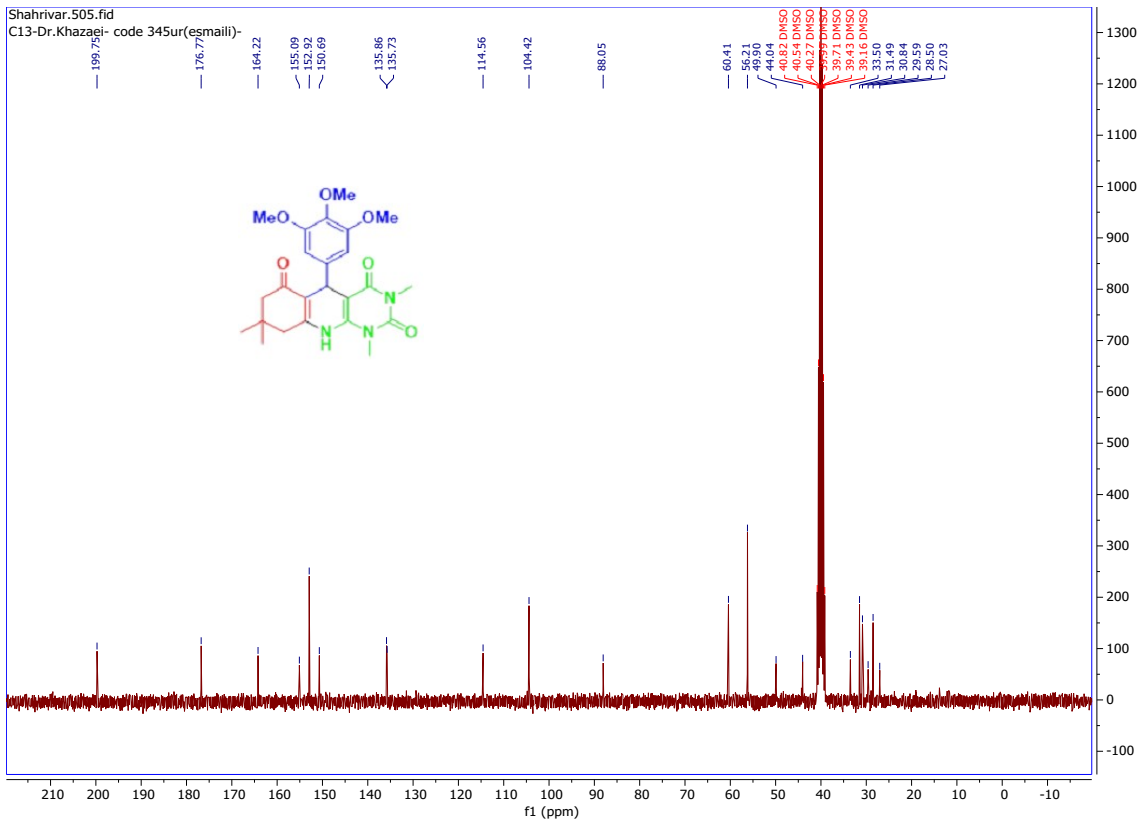
5-(3-bromophenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S6)



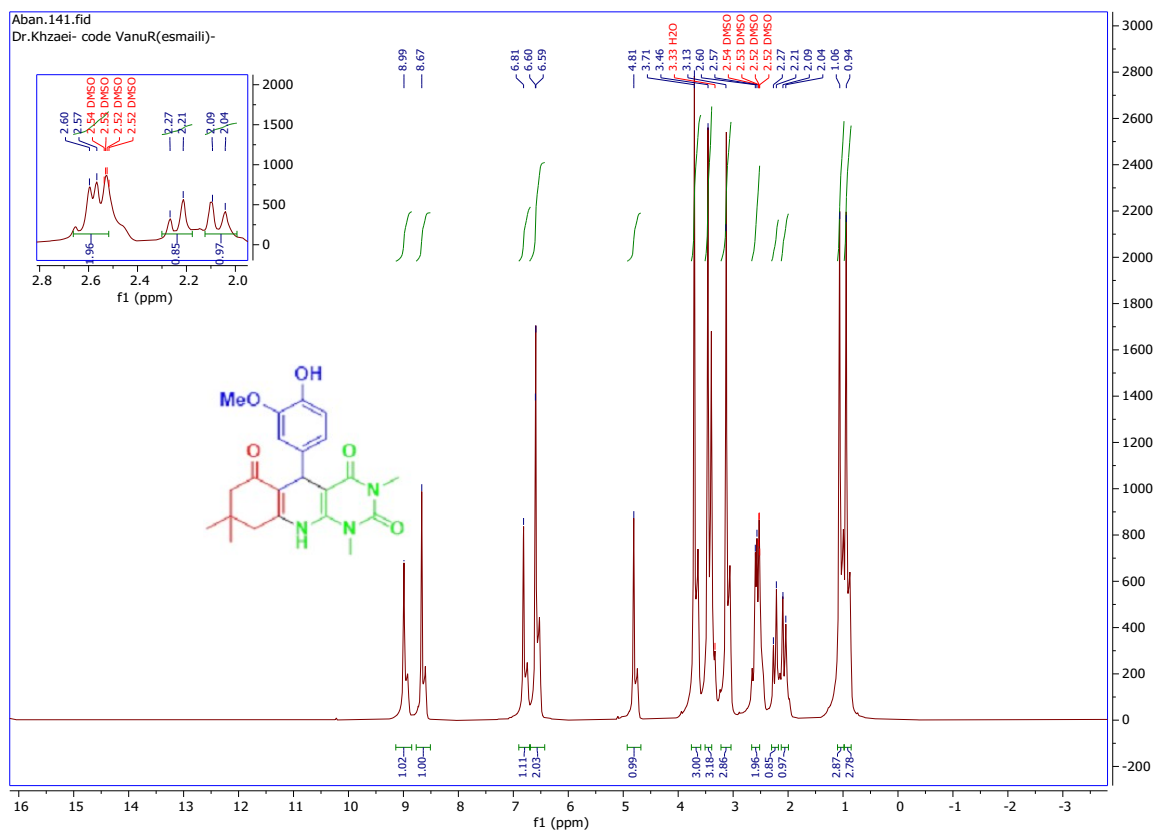


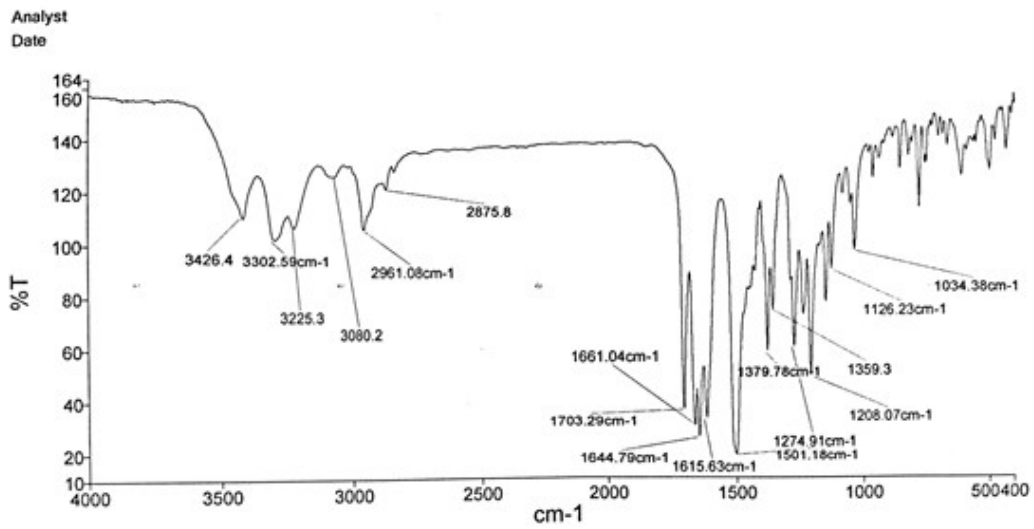
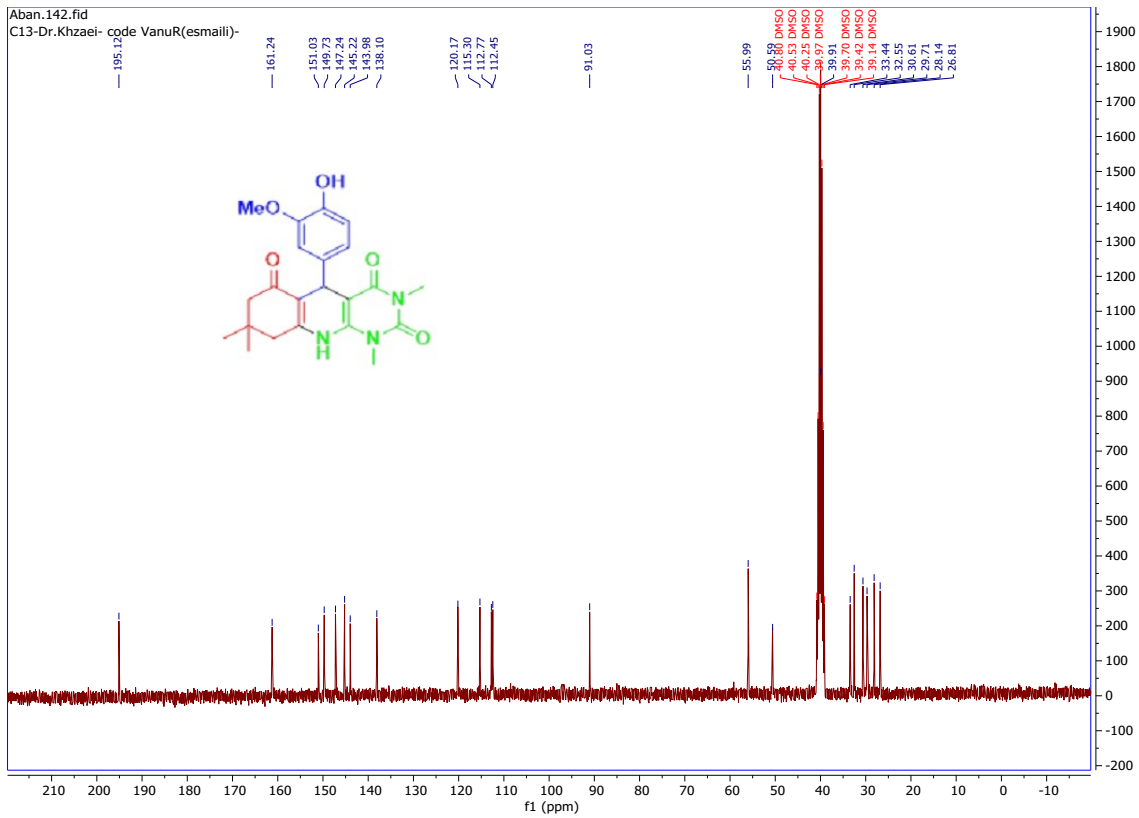
5-(3,4,5-trimethoxyphenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S7)



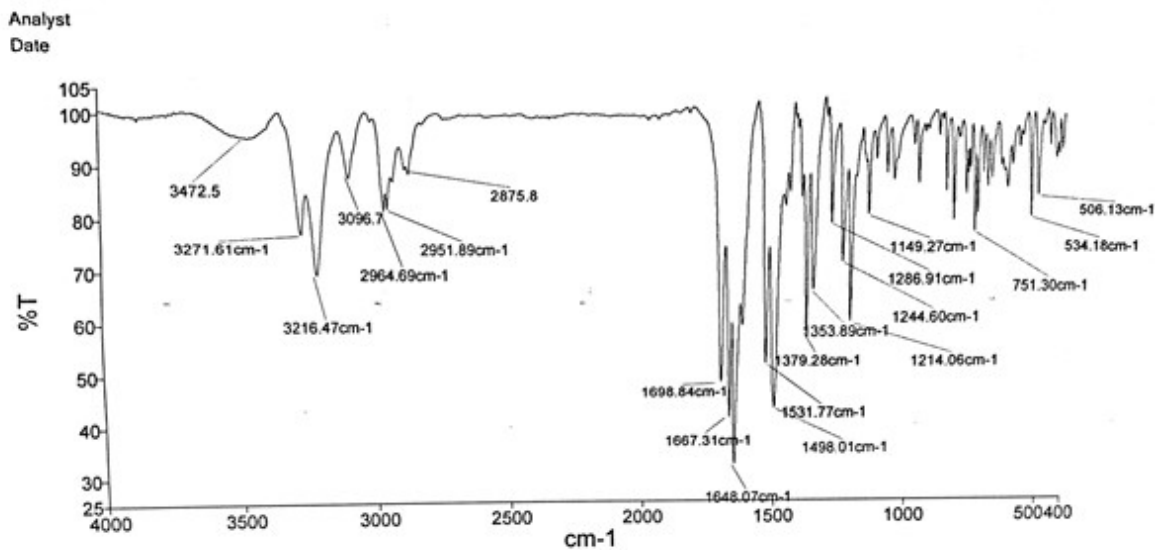


5-(4-hydroxy-3-methoxyphenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S12)

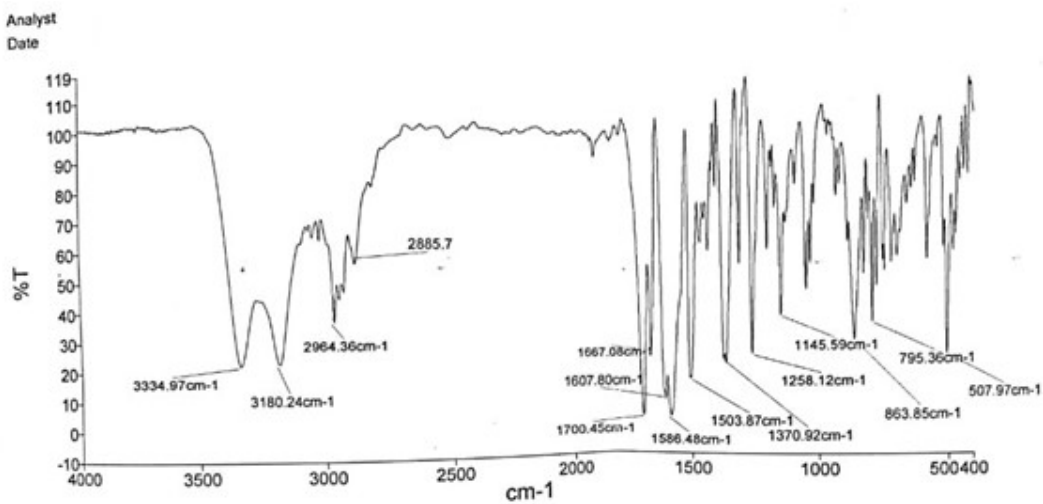




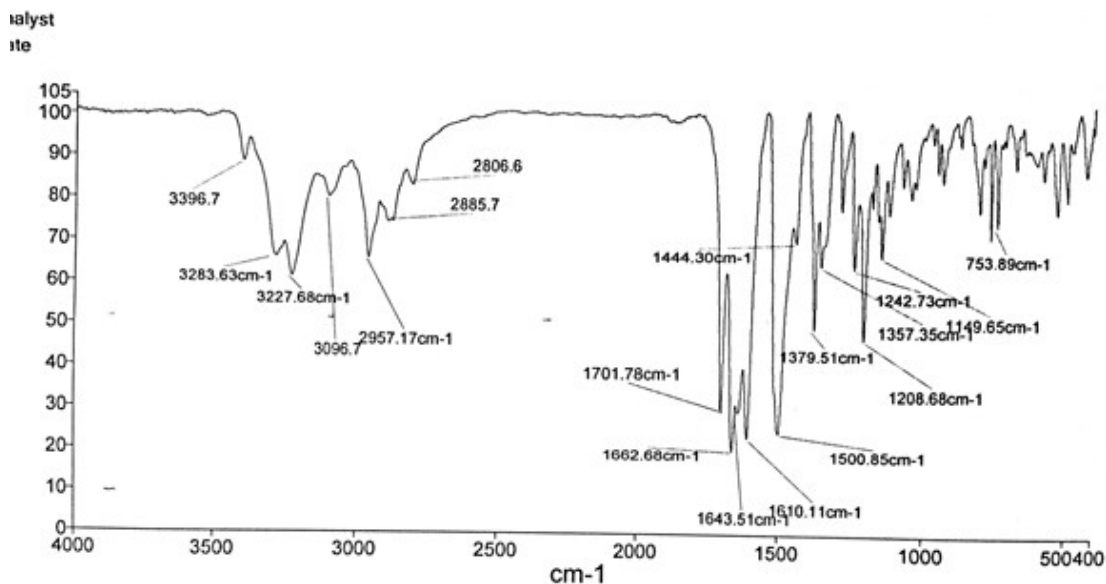
**1,3,8,8-tetramethyl-5-(2-nitrophenyl)-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-
2,4,6(1H,3H,7H)-trione (S8)**



**1,3,8,8-tetramethyl-5-(p-tolyl)-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-
2,4,6(1H,3H,7H)-trione (S9)**



5-(4-(dimethylamino)phenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S10)



5-(2-methoxyphenyl)-1,3,8,8-tetramethyl-5,8,9,10-tetrahydropyrimido[4,5-b]quinoline-2,4,6(1H,3H,7H)-trione (S11)

