Green chemistry approach to the synthesis of 2-aryl/heteroaryl substituted 2,3-dihydroquinazolin-4(1*H*)-ones using lemon juice under concentrated solar radiations as a renewable source

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1. Experimental

1.1. General Remarks

All chemical reagents in high purity were purchased (*Make: Sigma-Aldrich, Merck, SD fine chemicals and Avra synthesis*) and used without any further purification. The FT-IR, ^{1}H NMR and ^{13}C NMR spectral analyses were performed on a Bruker using $CDCl_3$ and DMSO-d6 as solvents. The FT-IR spectroscopic analysis was recorded on Bruker with KBr pellets. Melting points were determined in open capillaries and uncorrected. All the glass apparatus were dried prior to use.

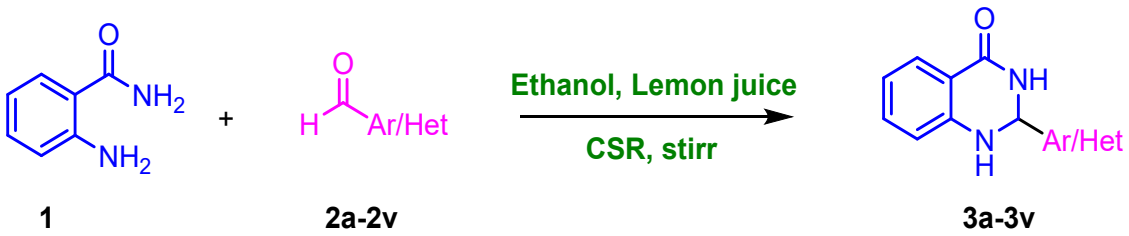
1.2. Preparation of lemon juice

Fresh lemon we taken and washed it thoroughly with water and cut by using a knife and then pieces where pressed manually. Then, the juice was filtered to remove solid material and to get clear juice which was used as a catalyst (**Figure S1**).

Figure S1: Preparation of lemon juice

1.3. Synthesis of 2-aryl/heteroaryl substituted quinozolin-4(1*H*)-ones

A mixture of anthranilamide (**1**, 0.01 mol) and aryl/heteroaryl aldehyde (**2**, 0.01 mol) and lemon juice (0.3 mL) were mixed in a 50 mL conical flask (fitted with condenser) containing ethyl alcohol as a solvent. This mixture was exposed concentrated solar radiation for an appropriate time (monitored by TLC) (**Figure S2**). The mixture is then cooled to room temperature and to this ethanol was added. The reaction mixture was then filtered to get pure products (**Scheme 1**). The structures of all products were confirmed by using spectroscopic data such as FT-IR, ^{1}H NMR and ^{13}C NMR spectral data.



Scheme 1 Synthesis of 2-aryl/heteroaryl substituted quinozolin-4(1*H*)-ones

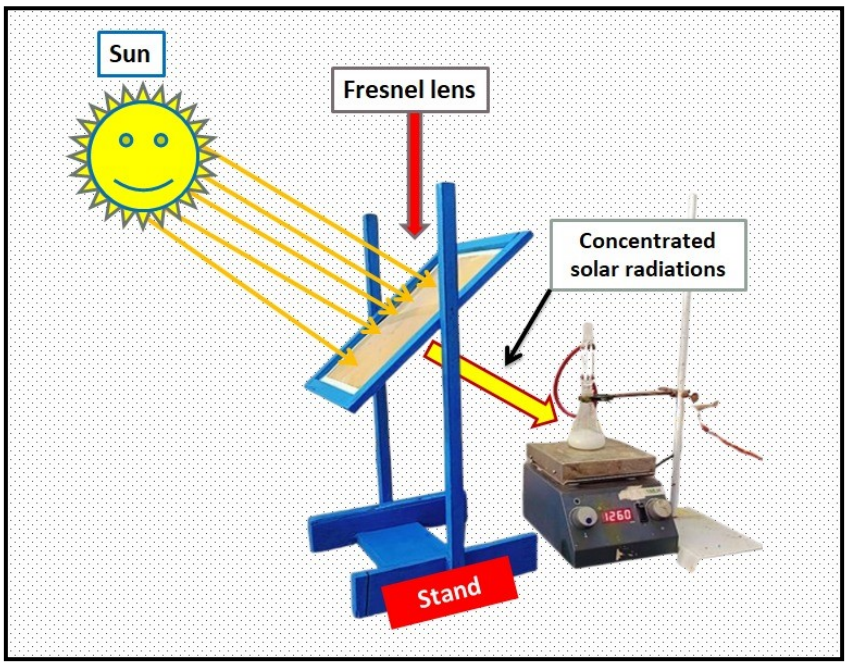
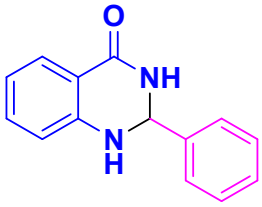


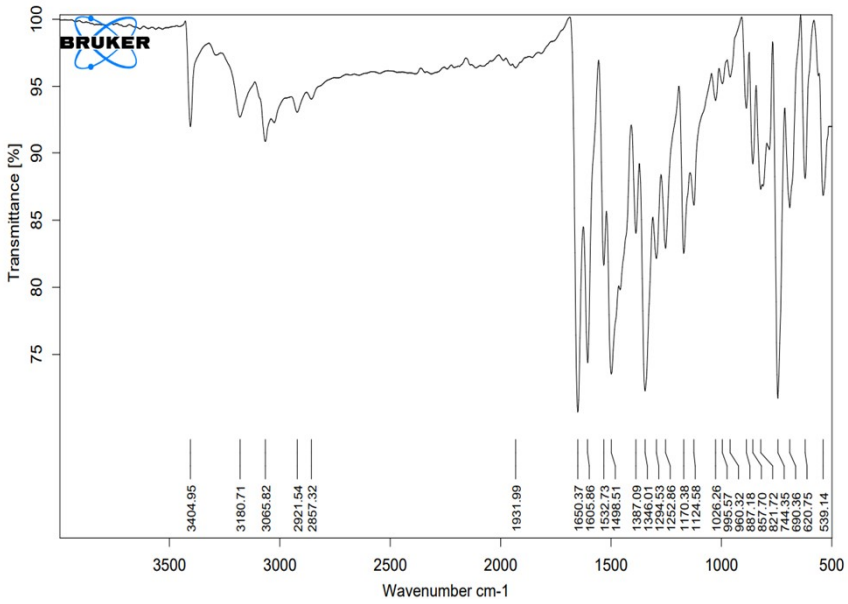
Figure S2 Diagram showing CSR for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-one

2. Representative Spectral results

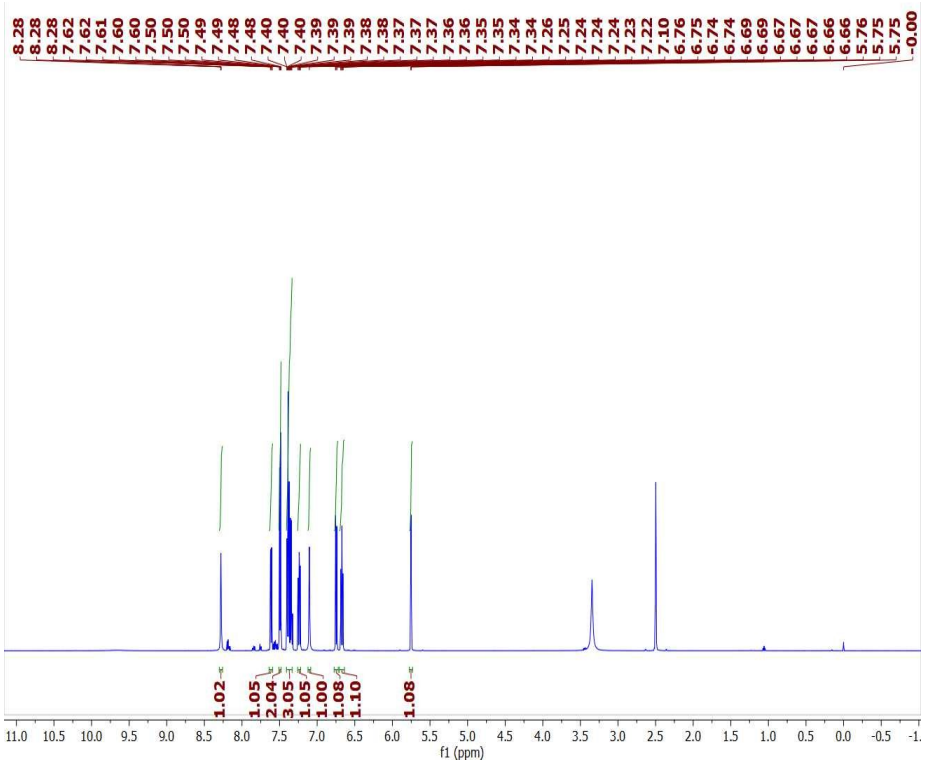
2.1.1. 2-phenyl-2,3-dihydroquinazolin-4(1H)-one



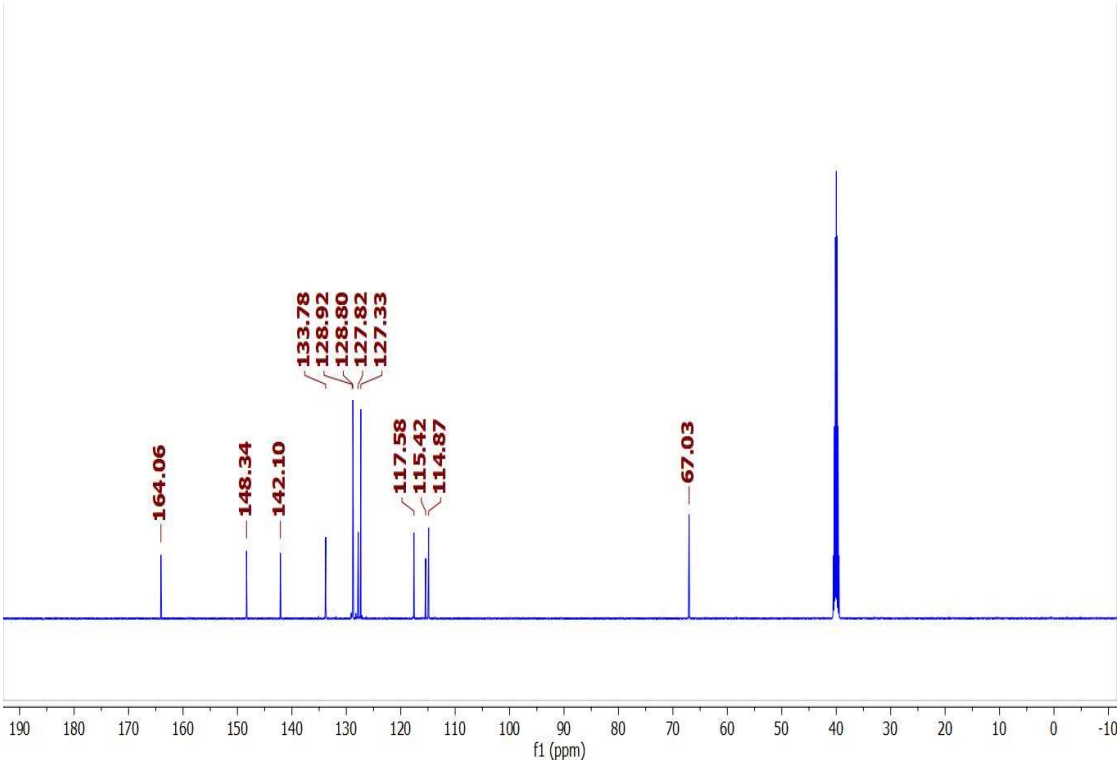
FT-IR (cm^{-1}): 3298.88, 3168.51, 3058.15, 1944.90, 1652.02, 1608.01, 1505.01, 1477.18, 1440.25, 1383.18, 1325.59, 1299.44, 1245.78, 1155.34, 1026.57, 914.83, 857.45, 806.22, 743.81, 693.15, 662.45, 597.67, 564.45.



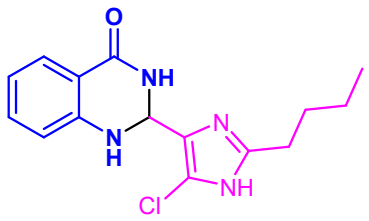
^{1}H NMR (500 MHz, DMSO-d6) δ: 8.28 (t, *J* = 1.7 Hz, 1H), 7.61 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.52 – 7.46 (m, 2H), 7.41 – 7.33 (m, 3H), 7.27 – 7.20 (m, 1H), 7.10 (s, 1H), 6.75 (dd, *J* = 8.2, 1.0 Hz, 1H), 6.71 – 6.64 (m, 1H), 5.75 (t, *J* = 1.7 Hz, 1H)



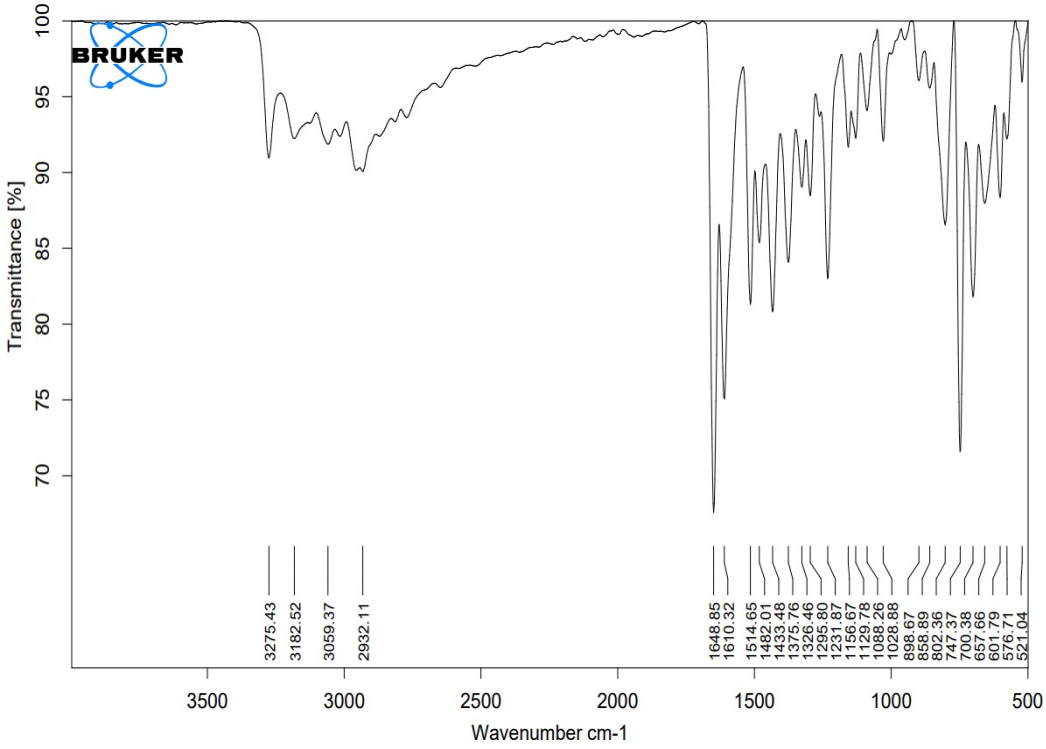
^{13}C NMR (126 MHz, DMSO-d6) δ: 164.06, 148.34, 142.10, 133.78, 128.92, 128.80, 127.82, 127.33, 117.58, 115.42, 114.87, 67.03



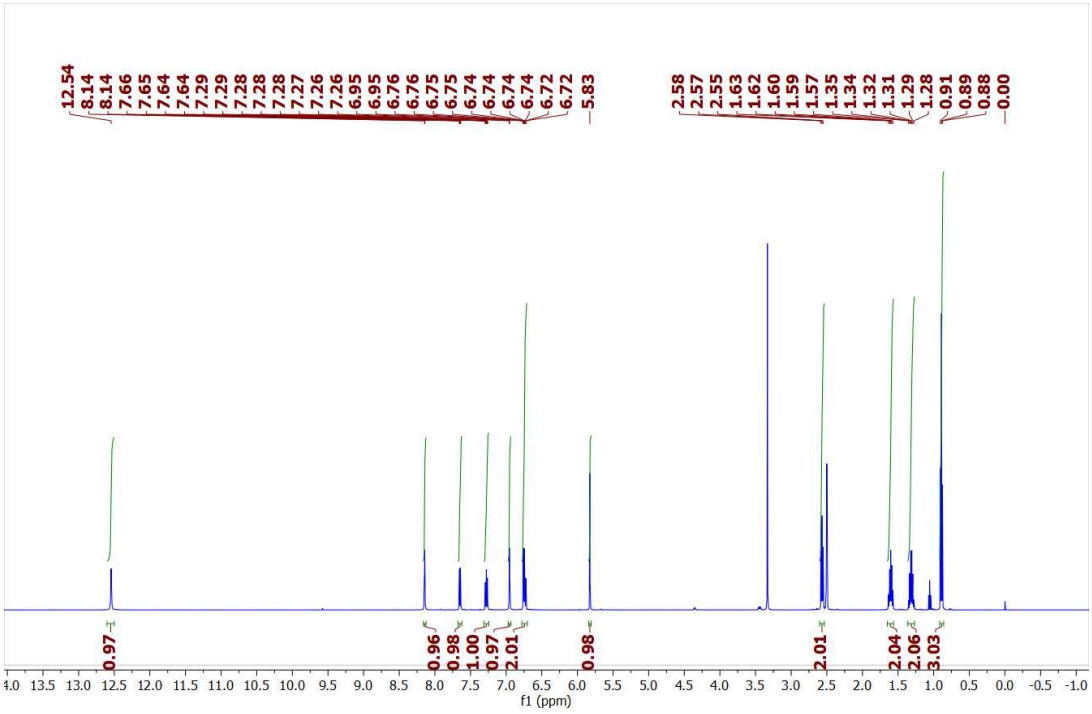
2.1.2. *2-2-(2-butyl-5-chloro-1H-imidazol-4-yl)-2,3-dihydroquinazolin-4(1H)-one*



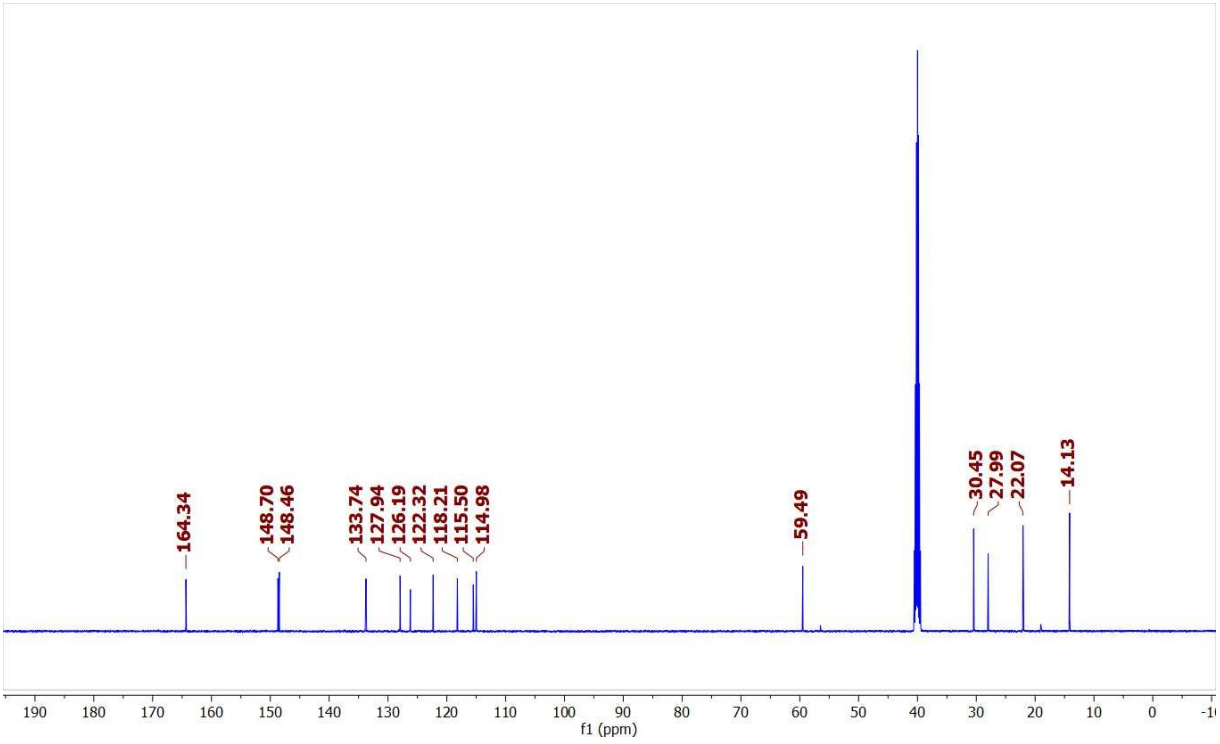
FT-IR (cm^{-1}): 3275.43, 3182.52, 3059.37, 2932.11, 1648.85, 1610.32, 1514.65, 1482.01, 1433.48, 1375.76, 1326.46, 1295.80, 1231.87, 1156.67, 1129.78, 1088.26, 1028.88, 898.67, 858.89, 802.36, 747.37, 700.38, 657.66, 601.79, 576.71.



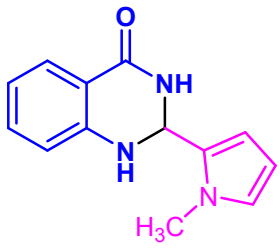
^{1}H NMR (500 MHz, DMSO-d6) δ: δ 12.54 (s, 1H), 8.14 (d, J = 1.7 Hz, 1H), 7.65 (dd, J = 7.7, 1.6 Hz, 1H), 7.28 (ddd, J = 8.1, 7.2, 1.6 Hz, 1H), 6.95 (d, J = 1.6 Hz, 1H), 6.78 – 6.70 (m, 2H), 5.83 (s, 1H), 2.57 (t, J = 7.5 Hz, 2H), 1.60 (quin, J = 7.5 Hz, 2H), 1.31 (sext, J = 7.5 Hz, 2H), 0.89 (t, J = 7.5 Hz, 3H)



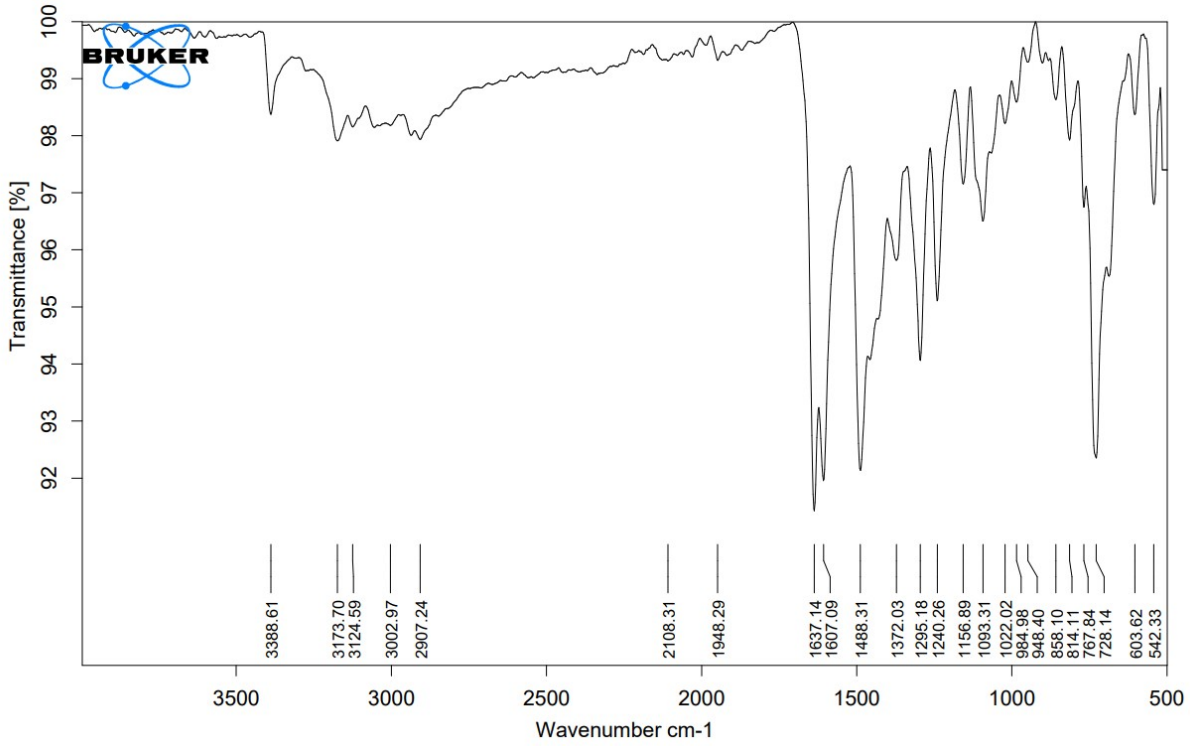
^{13}C NMR (126 MHz, DMSO-d6) δ: 164.34, 148.70, 148.46, 133.74, 127.94, 126.19, 122.32, 118.21, 115.50, 114.98, 59.49, 30.45, 27.99, 22.07, 14.13



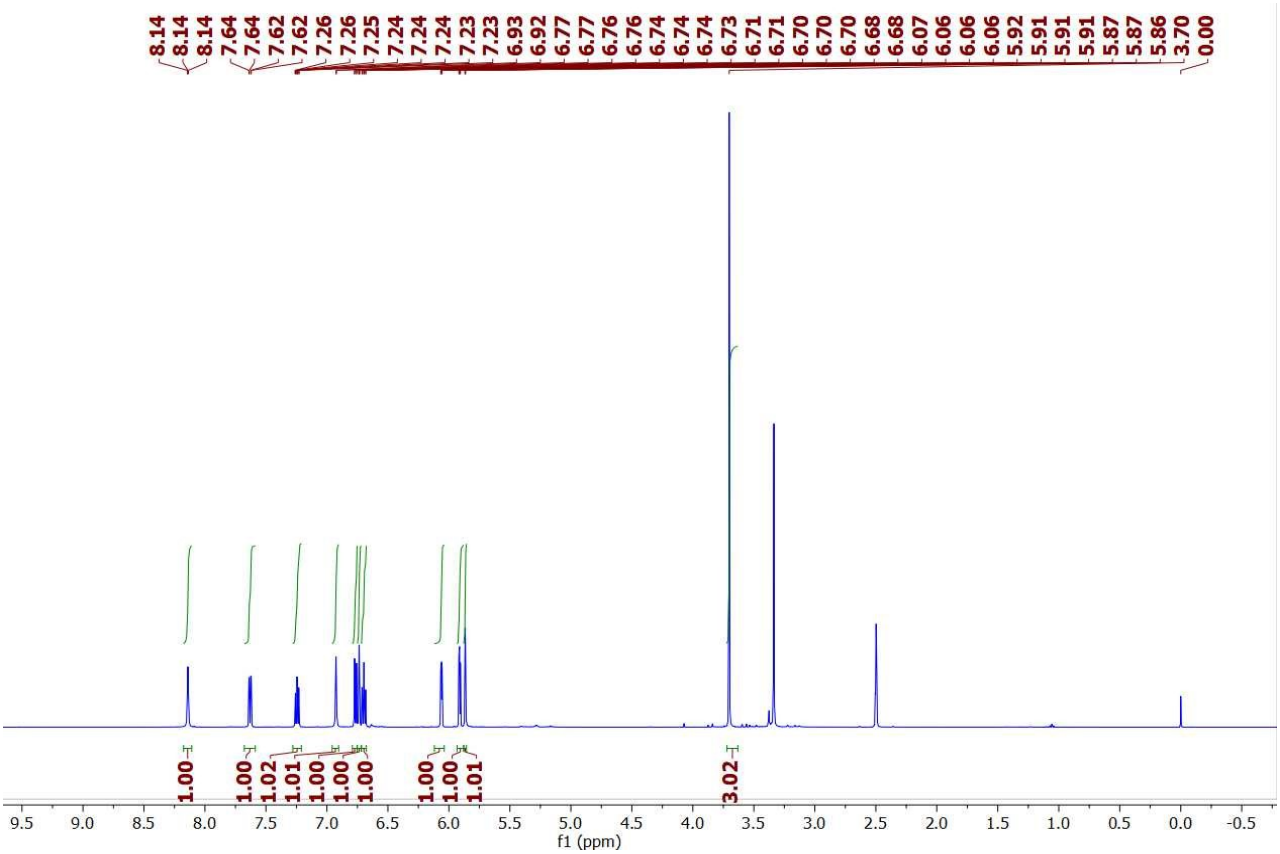
2.1.3. 2-(1-methyl-1H-pyrrol-2-yl)-2,3-dihydroquinazolin-4(1H)-one



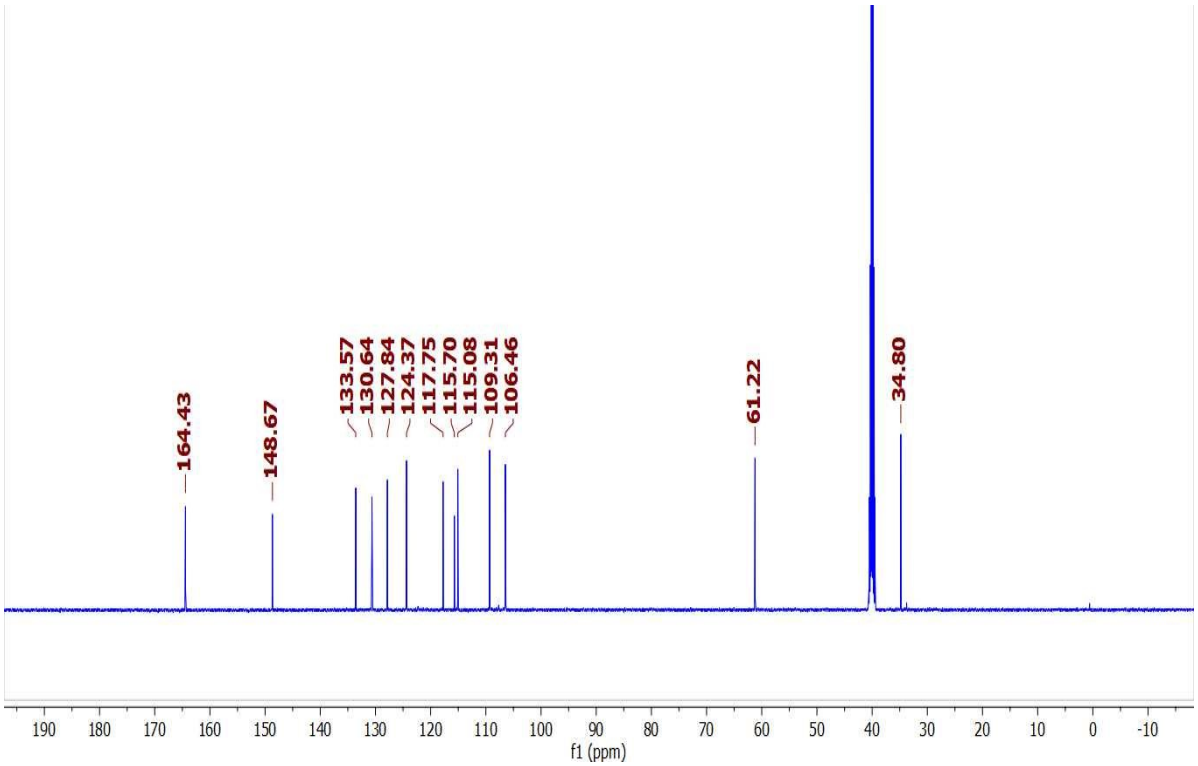
FT-IR (cm^{-1}): 3388.61, 3173.70, 3124.59, 3002.97, 2907.24, 2108.31, 1948.29, 1637.14, 1607.09, 1488.31, 1372.03, 1295.18, 1240.26, 1156.89, 1093.31, 1022.02, 984.98, 948.40, 858.10, 814.11, 767.84, 728.14, 603.62, 542.33.



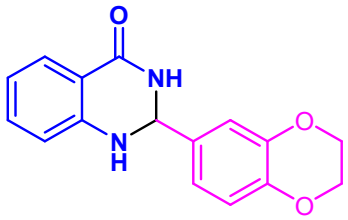
^{1}H NMR (500 MHz, DMSO-d6) δ: 8.14 (t, J = 1.7 Hz, 1H), 7.63 (dd, J = 7.8, 1.6 Hz, 1H), 7.24 (ddd, J = 8.1, 7.2, 1.6 Hz, 1H), 6.92 (d, J = 1.8 Hz, 1H), 6.77 (dd, J = 8.2, 1.1 Hz, 1H), 6.74 (dd, J = 2.7, 1.9 Hz, 1H), 6.70 (ddd, J = 8.0, 7.2, 1.1 Hz, 1H), 6.06 (dd, J = 3.6, 1.9 Hz, 1H), 5.91 (dd, J = 3.6, 2.7 Hz, 1H), 5.87 (t, J = 1.5 Hz, 1H), 3.70 (s, 3H)



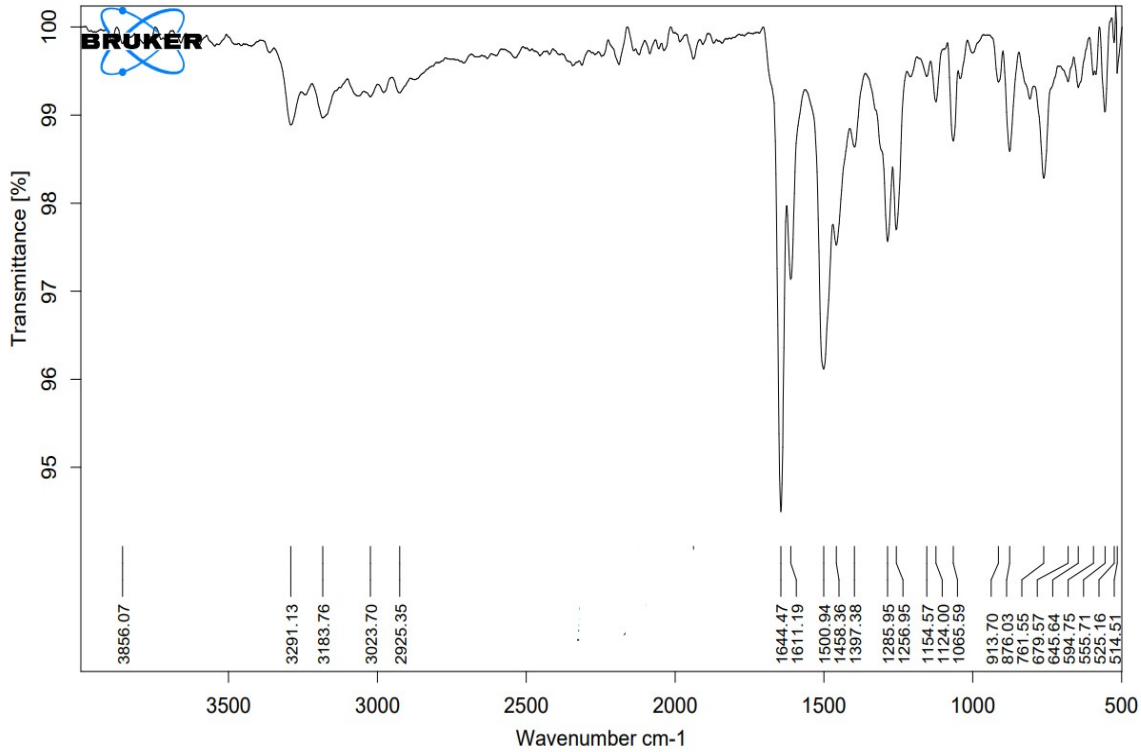
^{13}C NMR (126 MHz, DMSO-d6) δ: 164.43, 148.67, 133.57, 130.64, 127.84, 124.37, 117.75, 115.70, 115.08, 109.31, 106.46, 61.22, 34.80



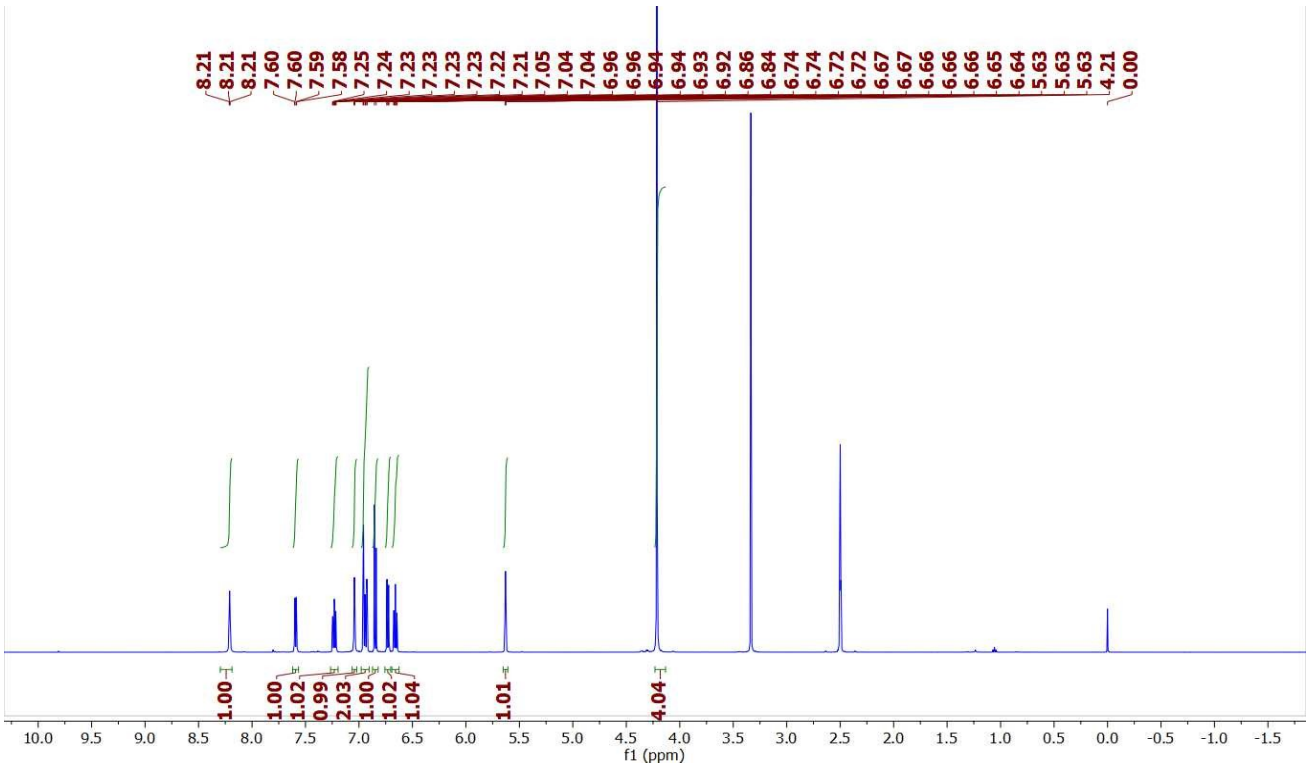
2.1.4. 2-(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)-2,3-dihydroquinazolin-4(1H)-one



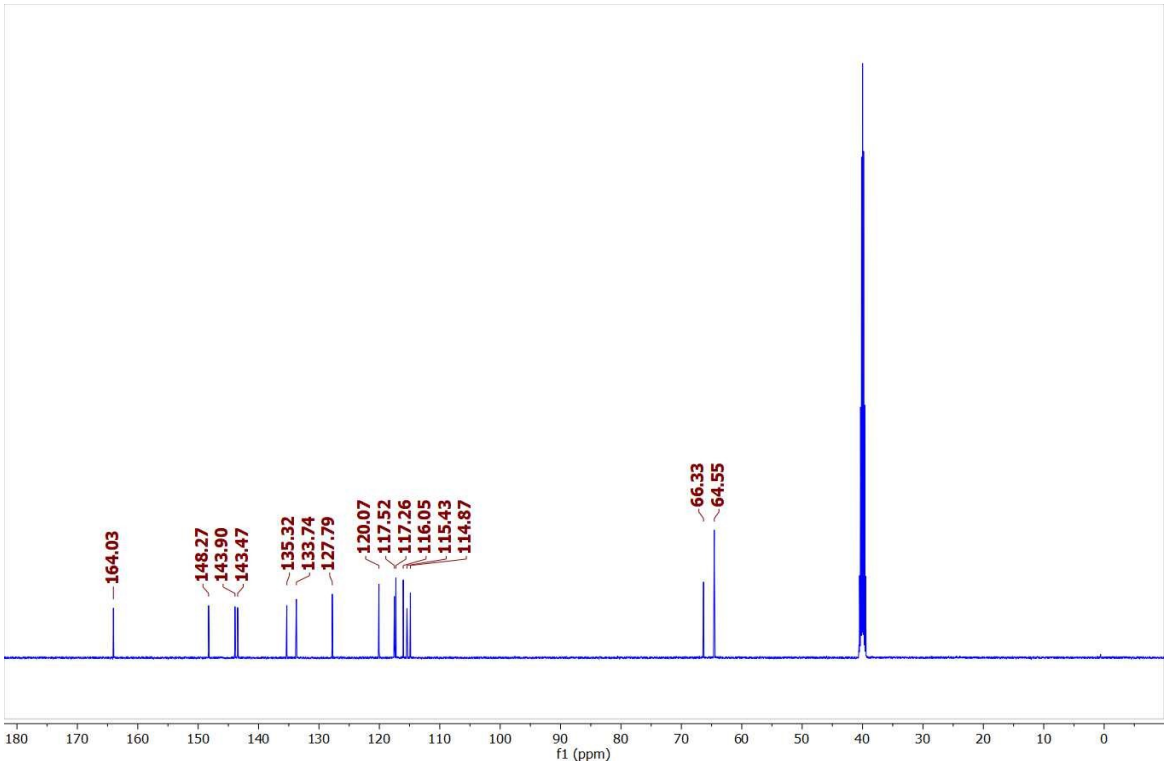
FT-IR (cm^{-1}): 3291.13, 3183.76, 3023.70, 2925.35, 1644.47, 1611.19, 1500.94, 1458.36, 1397.38, 1285.95, 1256.95, 1154.57, 1124.00, 1065.59, 913.70, 876.03, 761.55, 679.57, 645.64, 594.75, 555.71, 525.16



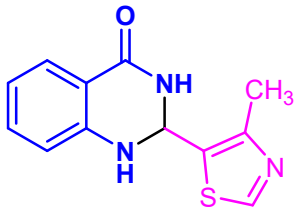
^{1}H NMR (500 MHz, DMSO-d6) δ 8.30 – 8.19 (m, 1H), 7.59 (dd, J = 7.8, 1.6 Hz, 1H), 7.23 (ddd, J = 8.1, 7.2, 1.7 Hz, 1H), 7.04 (d, J = 1.8 Hz, 1H), 6.98 – 6.90 (m, 2H), 6.85 (d, J = 8.3 Hz, 1H), 6.73 (dd, J = 8.2, 1.1 Hz, 1H), 6.69 – 6.63 (m, 1H), 5.63 (t, J = 2.0 Hz, 1H), 4.21 (s, 4H)



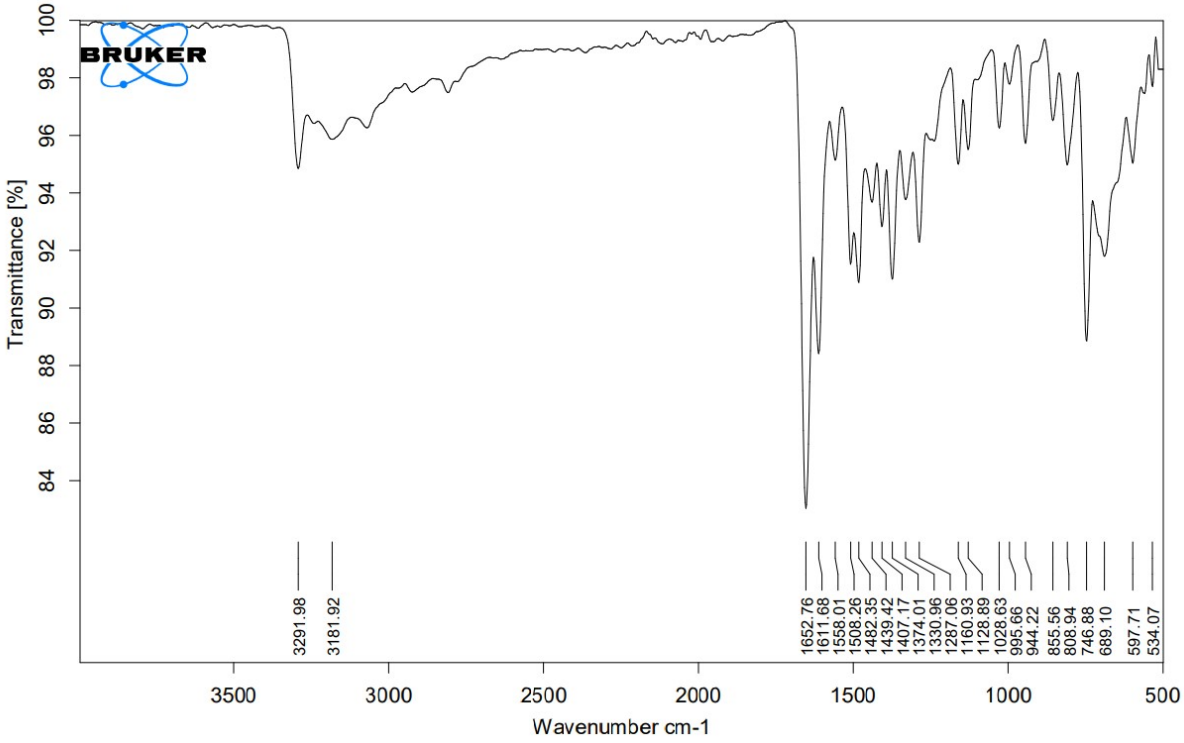
^{13}C NMR (126 MHz, DMSO-d6) δ 164.03, 148.27, 143.90, 143.47, 135.32, 133.74, 127.79, 120.07, 117.52, 117.26, 116.05, 115.43, 114.87, 66.33, 64.55



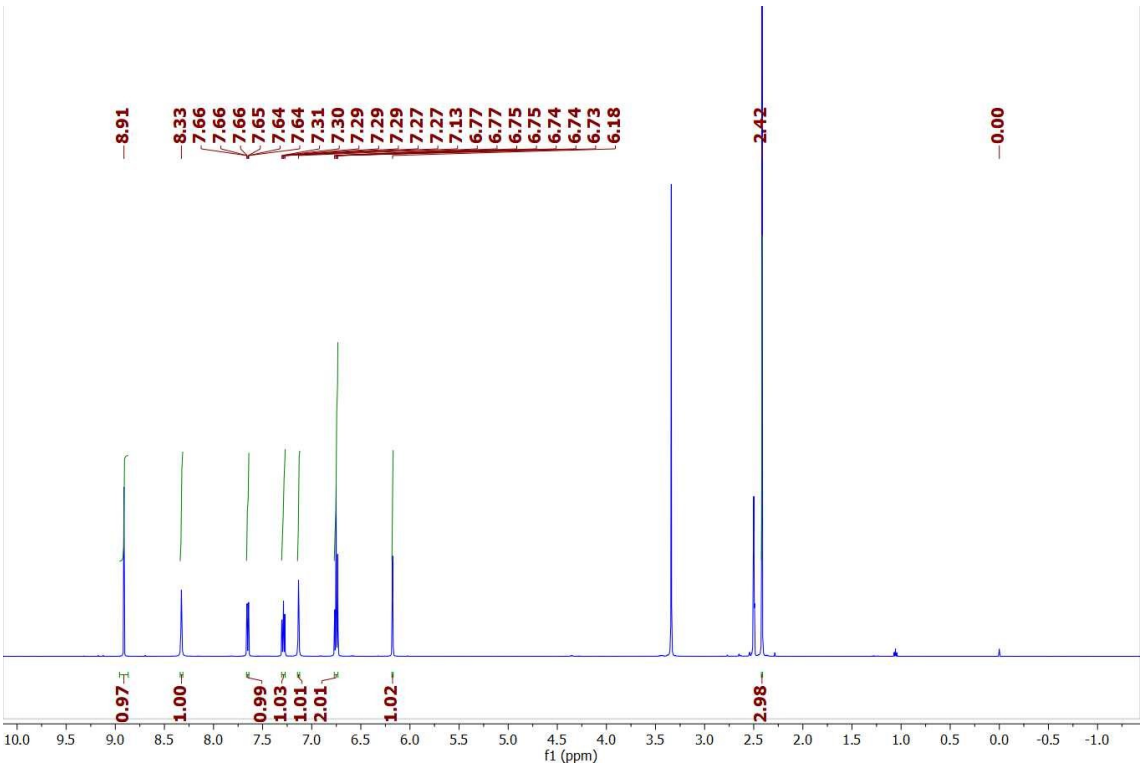
2.1.5. 2-(4-methylthiazol-5-yl)-2,3-dihydroquinazolin-4(1H)-one



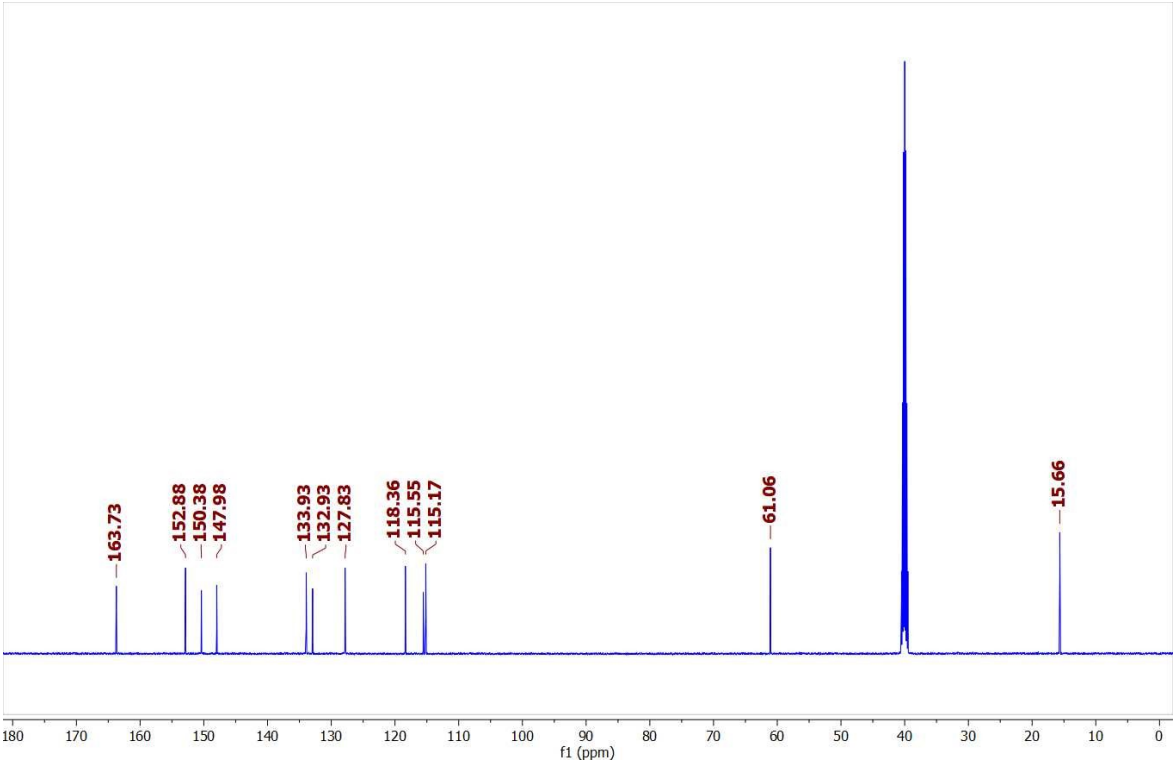
FT-IR (cm^{-1}): 3291.98, 3181.92, 1652.76, 1611.68, 1558.01, 1508.26, 1482.35, 1439.42, 1407.17, 1374.01, 1330.96, 1287.06, 1160.93, 1128.89, 1028.63, 995.66, 944.22, 855.56, 808.94, 746.88, 689.10, 597.71, 534.07



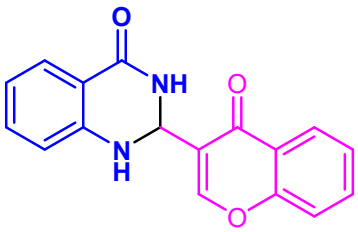
^{1}H NMR (500 MHz, DMSO-d6) δ 8.91 (s, 1H), 8.33 (s, 1H), 7.68 – 7.62 (m, 1H), 7.32 – 7.25 (m, 1H), 7.13 (s, 1H), 6.79 – 6.72 (m, 2H), 6.18 (s, 1H), 2.49 (s, 3H)



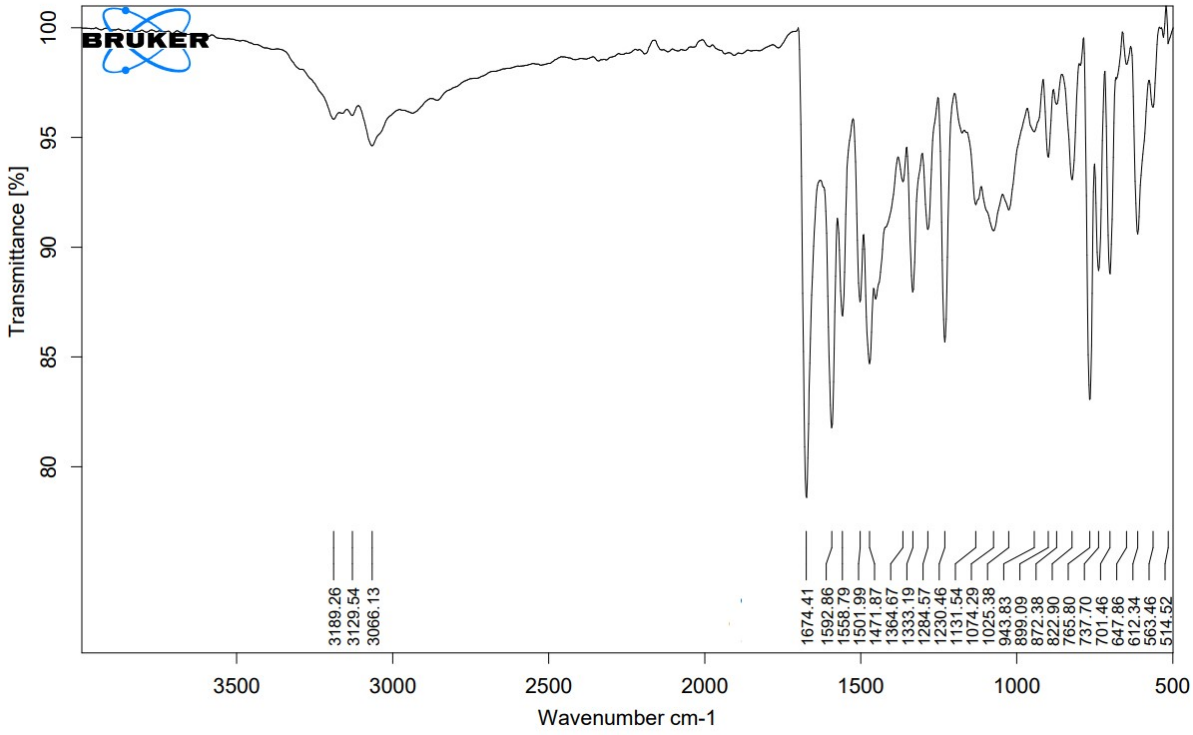
^{13}C NMR (126 MHz, DMSO-d6) δ 163.73, 152.88, 150.38, 147.98, 133.93, 132.93, 127.83, 118.36, 115.55, 115.17, 61.06, 15.66



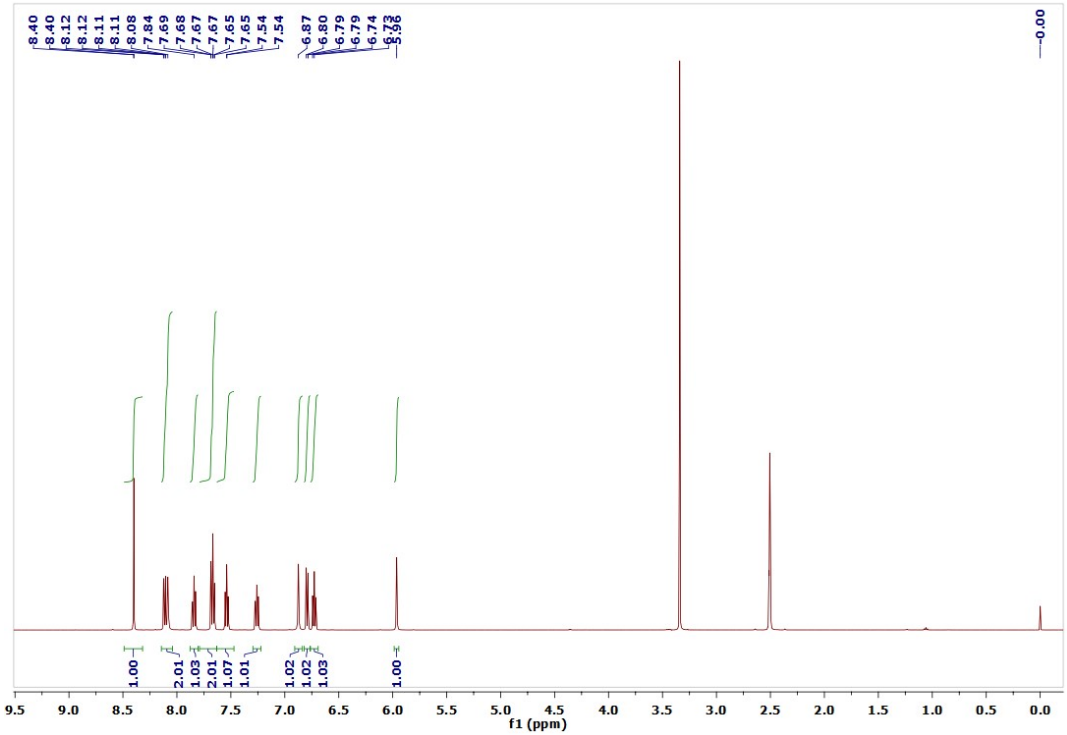
2.1.6. 2-(4-oxo-4H-chromen-3-yl)-2,3-dihydroquinazolin-4(1H)-one



3189.26, 3129.54, 3066.13, 1674.41, 1592.86, 1558.79, 1501.99, 1471.87, 1364.67, 1333.19, 1284.57, 1230.46, 1131.54, 1074.29, 1025.38, 943.83, 899.09, 872.38, 822.90, 765.80, 737.70, 701.46, 647.86, 612.34, 563.46.



^{1}H NMR (500 MHz, DMSO-d6) δ 8.40 (d, *J* = 0.6 Hz, 1H), 8.13 – 8.06 (m, 2H), 7.84 (ddd, *J* = 8.7, 7.2, 1.7 Hz, 1H), 7.67 (td, *J* = 8.2, 1.0 Hz, 2H), 7.54 (ddd, *J* = 8.1, 7.2, 1.0 Hz, 1H), 7.26 (ddd, *J* = 8.2, 7.2, 1.6 Hz, 1H), 6.87 (s, 1H), 6.81 – 6.78 (m, 1H), 6.74 – 6.68 (m, 1H), 5.96 (s, 1H).



^{13}C NMR (126 MHz, DMSO-d6) δ 175.94, 164.10, 156.18, 155.78, 148.14, 135.10, 133.84, 127.92, 126.33, 125.54, 123.72, 123.43, 119.02, 118.09, 115.34, 115.29, 60.01.