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Green Synthesis of 2,3-Dihydroquninazolin-4(1H)-one Derivative by using Polyaniline Supported Zinc Oxide Nanocomposite

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Abstract: A facile synthesis of substituted 2,3-Dihydroquinazolin-4(1H)-ones analogs are by the reaction of various substituted aldehyde, anthranilamide under polyaniline supported zinc oxide heterogeneous catalyst and the formation of product good to excellent yield. We have recovered and reused polyaniline-supported zinc oxide catalyst. The heterogeneous catalyst was characterized by UV, FT-IR, 1H NMR, SEM, and XRD techniques.

Keywords: heterogeneous catalyst; zinc oxide nanoparticle; 2,3-dihydroquninazolin-4(1H)-ones; anthranilamide.

1. Introduction

Quinazolinone is the most popular heterocyclic nucleus in a large organic molecule that has diverse biological activities, substituted 2,3-dihydroquinazoline-4(1H)-ones derivatives, our wide variety of biological, pharmaceutical, and medicinal activities such as antifungal, anti-inflammatory, antimicrobial, anti-convulsant, analgesic, antibacterial, anticancer and anti-diuretic [1]. The multicomponent reaction has emerged as an attractive strategy for synthetic chemists because the product can be obtained in a single step. Moreover, synthesizing a novel 2,3-dihydroquinazolinone method that decreases pollution in the chemical industry has established an important consideration because of increasing environmental concern [2-3]. Driven by the current global challenges to partake in a pollution-free environment, our interest has turned towards energy-consuming chemistry and catalysis [4]. Various protocols for the synthesis of quinazolinone have been discovered in the past. Some common method include condensation of 2-aminobenzamides and substituted benzoyl chloride or their equivalent in ionic liquid [5]. These heterocyclic compounds contribute to physiological procedures as indicators or courier molecules, and many pharmaceuticals based on these heterocycles have been described in the past few decades [6]. In arrears to an extensive range of applications, several methods for the preparation of 2,3-dihydroquinazolinones. The desirable path is to catalyze the condensation of anthranilamide with aldehyde directly for the goal product. Nevertheless, most of these methods are connected with several boundaries, including difficult responses, vast excess of oxidants, non-renewable solvents, harsh reaction conditions, lengthy reaction times, and low reaction yield. In current years, the emphasis has been on the growth

of cheap and suitable methods for the synthesis of 2,3-dihydroquinazoline-4(1H)-one derivatives via acceptable approaches [7]. Numerous methods have been reported for the synthesis of 2,3-dihydroquinazolin-4(1H) ones, which embrace the multicomponent reaction of isatoic anhydride, ammonium chloride, and aldehyde using indium chloride, condensation of 2-aminobenzamide with aldehyde using zirconiumchloride (IV) in ethanol, and palladium-catalyzed cyclocarbonylation of o-iodoanilines. Some of the methodologies produce good results [8]. Numerous procedures have been projected to obtain quinazoline analogs using catalysts like zirconyl chloride, ammonium bromide, heteropoly acid, gallium (III) triflate, titanium oxide nanoparticles starch solution, and cyclodextrin sulphonic acid. The greatest of these practices suffer from long reaction times, high temperatures, the use of costly catalysts, and dull work procedures [9-10]. The frequency of cyclocondensation depended upon temperature. Thus, the reaction temperature is a significant factor. The yield of the reaction was resolute at various temperatures and found to grow with increasing temperature mostly to 75 °C [11-13]. Reported methodologies for this synthesis include various catalysts such as Bi(0), $HClO_4/SiO_2$, $CeCl_3.7H_2O$, DABCO, TMSCl, $Ga(ClO_4)_3$, and transition metals like Mn, Ru, Pd, and Cu. Solid phase synthesis and microwave-assisted [14-16]. To illustrate this concept, we did well in using polyaniline-supported zinc oxide as a catalyst in organic transformation, succeeding our interest in its use in medicinal chemistry [17-20]. Thus, the present technique characterizes substantial developments over the previously reported method from a green and supportable point of view [21-28].

Recently a variety of synthetic methods have been reported to obtain 2,3-dihydroquinazoline-4(1H)one by using condensation of an aldehyde with anthranilamide in the presence of a different catalyst such as palladium catalyst [29], Para-aminobenzoic acid grafted on the silica-coated magnetic nanoparticle (Fe_3O_4@SiO_2@Pr-PABA) [30], lewis acid catalyst cross-linked poly (4-vinylpyridine)supported BF3 [31], heterogenized guanidino-nickel catalyst (hercynite@SiO_2-L-Arginine-Ni.) [32], Lewis acid catalyst $BF_3.OEt_2$ [33], SBA-15-@n-Pr-THAM-ZrO [34], MCM-41-SP-DMG-Cu(II) [35], base promoted metal free [36], magnetic catalyst [37], montmorillonite-K10 [38], zirconium-based metal-organic framework or reduced graphene oxide Nanocatalyst [39], and Cu(II) complex decorated hybrid nanomaterial has been used [40].

2. Materials and Methods

2.1. Synthesis of zinc oxide nanoparticle.

In this experiment, we have synthesized zinc oxide nanoparticles by using the co-precipitation method; in this procedure, 0.5 M aqueous zinc nitrate ($Zn(NO_3)_2.6H_2O$) solution was kept under magnetic stirring for 30 min. and then the addition of 0.5 M sodium hydroxide (NaOH) solution and continue the stirring. Then form the precipitate and filter the solution to dry it at room temperature.

2.2. Synthesis of PANI-ZnO nanocomposite.

In a round bottom flask, 2.5 gm of aniline hydrochloride was added to prepare PANI-ZnO nanocomposite in 100 ml double distilled water, which was agitated for 15 min. The homogenous mixture is maintained in an ice bath for 2 hr. This formed solution is given the name A. The formed ZnO nanocomposite (2 wt %) was dispersed in the above-mentioned solution A. 5 gm of APS was dissolved in 25 ml of double distilled water and kept in an ice

bath for 2 hr. This obtained solution was named given as B. We added solution of B to the solution dropwise, and the obtained solution (A+B) was stirred for 3 hr. After adding APS, this reaction combination turned green compared to bare PANI, indicating that the ZnO nanocomposite increases the formation of PANI/ZnO. This solution was kept stable for 24 hr. The dark green precipitate of emeraldine salt was separated by filtration. The formed dark green compound was used for further characterization.

2.3. General procedure for the synthesis of 2,3-dihydroquinazoline-4(1H)ones.

In a round bottom flask 2-aminobenamide **(1)** (1mmol) and aromatic aldehyde **(2a-m)** (1mmol) were added in ethanol and also added 2 (mol%) polyaniline supported zinc oxide heterogeneous catalysts. The mixture was heated up to 2 hr. at 70 °C. The improvement of the reaction was checked on the thin layer chromatography plate. After the reaction was accomplished, the mixture was cooled down by a room temperature polyaniline-supported zinc oxide catalyst, which was separated by a filtration process. The resulting solid crud products **(3a-m)** were filtered and recrystallized from pure ethanol.

2.3.1. 2-(4-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (3a).

MP: 182; UV:210; IR (KBr): 3287, 1645, 1615, 1513,1298, 1195,1069 cm^{-1}. ^{1}H NMR (500 MHz, DMSO) δ 9.03 (s, 1H), 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.41 – 7.34 (m, 2H), 7.37 – 7.33 (m, 1H), 7.30 (td, J = 8.1, 1.6 Hz, 1H), 6.97 (dd, J = 8.2, 1.5 Hz, 1H), 6.78 – 6.72 (m, 2H), 6.15 (dd, J = 6.6, 5.8 Hz, 1H), 5.88 (d, J = 6.2 Hz, 1H), 5.80 (d, J = 6.0 Hz, 1H). ^{13}C NMR (125 MHz, DMSO) δ 164.34, 158.08, 145.62, 134.51, 133.06, 129.25, 127.93, 117.56, 116.09, 115.54, 115.12, 67.71. MS: 240; Anal. Calculation for $C_{14}H_{12}N_2O_2$: Elemental Analysis: C, 69.99; H, 5,03; N,11.66.

2.3.2. 2-(4-hydroxy-3-methoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (3b).

MP: 185; UV: 220; IR (KBr): 3380, 3230, 3072, 1640, 1625, 1586, 1565, 1516, 1482 cm^{-1}. ^{1}H NMR (500 MHz, DMSO) δ 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.60 (s, 1H), 7.37 (td, J = 7.9, 1.4 Hz, 1H), 7.30 (td, J = 8.1, 1.6 Hz, 1H), 7.17 – 7.12 (m, 2H), 7.00 – 6.94 (m, 1H), 6.83 (d, J = 8.4 Hz, 1H), 6.56 (d, J = 6.2 Hz, 1H), 6.20 – 6.13 (m, 1H), 5.62 (s, 1H), 3.85 (s, 3H). ^{13}C NMR (125 MHz, DMSO) δ 164.35, 148.78, 147.10, 145.59, 134.51, 132.95, 127.93, 122.04, 117.56, 116.21, 115.04, 114.95, 111.43, 68.12, 56.22. MS: 270; Anal. calc. for $C_{15}H_{14}N_2O_3$: Elemental Analysis: C,66.66; H,5.22; N,10.36.

2.3.3. 2-(4-bromophenyl)-2,3-dihydroquinazolin-4(1H)-one (3c).

MP: 196; UV: 225; IR (KBr): 3304, 3185, 3050, 2925, 1655, 1603 , 1530 , 1460, 1340 cm^{-1}. ^{1}H NMR (500 MHz, DMSO) δ 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.51 – 7.43 (m, 4H), 7.37 (td, J = 8.0, 1.4 Hz, 1H), 7.30 (td, J = 8.1, 1.7 Hz, 1H), 6.97 (dd, J = 8.2, 1.5 Hz, 1H), 6.18 (t, J = 6.2 Hz, 1H), 5.86 (d, J = 6.4 Hz, 1H), 5.80 (d, J = 6.0 Hz, 1H). ^{13}C NMR (125 MHz, DMSO) δ 164.34, 145.62, 139.78, 134.51, 131.42, 129.54, 127.93, 122.26, 117.56, 116.09, 115.12, 67.79. MS: 302; Anal. calc. for $C_{14}H_{11}BrN_2O$: Elemental Analysis: C,55.47; H,3.66; Br, 26.36; N, 9.24.

2.3.4. 2-(3,4-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (3d).

MP: 181; UV: 204; IR (KBr): 3352, 3330, 3120, 3080, 1667, 1607, 1511, 1362 cm^{-1}. ^{1}H NMR (500 MHz, DMSO) δ 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.37 (td, J = 7.9, 1.4 Hz, 1H), 7.30 (td, J = 8.1, 1.6 Hz, 1H), 7.21 – 7.15 (m, 1H), 7.01 (dd, J = 1.8, 0.7 Hz, 1H), 6.97 (dd, J = 8.2, 1.5 Hz, 1H), 6.85 (d, J = 7.9 Hz, 1H), 6.56 (d, J = 6.2 Hz, 1H), 6.20 – 6.13 (m, 1H), 5.62 (d, J = 6.0 Hz, 1H), 3.84 (s, 6H). ^{13}C NMR (125 MHz, DMSO) δ 164.35, 150.46, 149.64, 145.59, 134.77, 134.51, 127.93, 121.83, 117.56, 116.21, 115.04, 112.25, 111.60, 67.87, 55.92, 55.90. MS: 284; Anal. calc. for $C_{16}H_{16}N_2O_3$: Elemental Analysis: C,67.59; H, 5.67; N, 9.85.

2.3.5. 2-(3-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (3e).

MP: 207; UV; 205 IR (KBr): 3352, 3330, 3120, 3080, 1667, 1607, 1511, 1362 cm^{-1}. ^{1}H NMR (500 MHz, DMSO) δ 8.40 (s, 1H), 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.41 – 7.34 (m, 2H), 7.34 – 7.26 (m, 2H), 6.97 (dd, J = 8.2, 1.5 Hz, 1H), 6.91 – 6.86 (m, 1H), 6.77 – 6.71 (m, 1H), 6.56 (d, J = 6.2 Hz, 1H), 6.01 (dd, J = 6.5, 5.7 Hz, 1H), 5.57 (d, J = 6.2 Hz, 1H). ^{13}C NMR (125 MHz, DMSO) δ 164.35, 158.18, 145.60, 143.13, 134.51, 130.04, 127.93, 119.45, 117.56, 116.21, 116.01, 115.62, 115.04, 67.97. MS: 240; Anal. calc. for $C_{14}H_{12}N_2O_2$: Elemental Analysis: C,69.99; H,5.03; N,11.66.

2.3.7. 2-(4-fluorophenyl)-2,3-dihydroquinazolin-4(1H)-one (3f).

MP: 193; UV: 206; IR (KBr): 3300, 3173, 3056, 2924, 1655 , 1608 , 1506 , 1472, 1335. ^{1}H NMR (500 MHz, DMSO) δ 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.53 – 7.46 (m, 2H), 7.37 (td, J = 8.0, 1.4 Hz, 1H), 7.30 (td, J = 8.1, 1.6 Hz, 1H), 7.08 – 7.00 (m, 2H), 6.97 (dd, J = 8.2, 1.5 Hz, 1H), 6.18 (dd, J = 6.5, 5.7 Hz, 1H), 5.86 (d, J = 6.2 Hz, 1H), 5.80 (d, J = 6.0 Hz, 1H). ^{13}C NMR (125 MHz, DMSO) δ 164.34, 163.51, 161.54, 145.62, 137.20, 137.17, 134.51, 129.83, 129.76, 127.93, 117.56, 116.09, 115.80, 115.62, 115.12, 67.35. MS: 242; Anal. Calc. for $C_{14}H_{11}FN_2O$: Elemental Analysis: C,69.41; H,4.58; F,7.84; N,11.56.

2.3.7. 2-(4-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one (3g).

MP: 213; UV: 207; IR (KBr): 3276, 3172, 3030, 2920, 1645, 1606, 1518, 1459, 1343. ^{1}H NMR (500 MHz, DMSO) δ 8.15 – 8.09 (m, 2H), 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.77 – 7.70 (m, 2H), 7.37 (td, J = 8.0, 1.4 Hz, 1H), 7.30 (td, J = 8.1, 1.6 Hz, 1H), 6.97 (dd, J = 8.2, 1.5 Hz, 1H), 6.15 (dd, J = 6.6, 5.9 Hz, 1H), 5.90 (d, J = 6.2 Hz, 1H), 5.80 (d, J = 6.0 Hz, 1H). ^{13}C NMR (125 MHz, DMSO) δ 164.34, 146.81, 145.62, 144.79, 134.51, 128.50, 127.93, 123.72, 117.56, 116.09, 115.12, 67.46. MS: 269; Anal. calc. for: $C_{14}H_{11}N_3O_3$: Elemental Analysis: C,62.45;H,4.12;N,15.61.

2.3.8. 2-(3-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one (3h).

MP: 211; UV: 209; IR (KBr): 3290, 3188, 3070, 2920, 1650 1604, 1522, 1459, 1340. ^{1}H NMR (500 MHz, DMSO) δ 8.37 (td, J = 2.1, 0.7 Hz, 1H), 8.09 (dt, J = 7.7, 2.1 Hz, 1H), 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.70 – 7.64 (m, 1H), 7.47 (dd, J = 8.4, 7.7 Hz, 1H), 7.37 (td, J = 8.0, 1.4 Hz, 1H), 7.30 (td, J = 8.1, 1.6 Hz, 1H), 6.97 (dd, J = 8.2, 1.5 Hz, 1H), 6.61 (d, J = 6.2 Hz, 1H), 6.34 – 6.28 (m, 1H), 5.96 (d, J = 6.0 Hz, 1H). ^{13}C NMR (125 MHz, DMSO) δ 164.36, 149.18, 145.37, 140.33, 134.51, 133.68, 129.95, 127.93, 123.47, 123.02, 117.56, 116.21,

115.08, 67.02. MS: 269; Anal. Calc. for: $C_{14}H_{11}N_3O_3$: Elemental Analysis: C,62.45; H,4.12; N,15.61.

2.3.9. 2-(3,4,5-trimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (3i).

MP: 185; UV: 211; IR (KBr): 3352, 3330, 3120, 3080, 1667, 1607, 1511, 1362 cm^{-1}. ^1H NMR (500 MHz, DMSO) δ 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.37 (td, J = 7.9, 1.3 Hz, 1H), 7.30 (td, J = 8.1, 1.6 Hz, 1H), 7.11 (d, J = 6.2 Hz, 1H), 6.97 (dd, J = 8.2, 1.5 Hz, 1H), 6.80 (d, J = 0.7 Hz, 2H), 6.36 (d, J = 6.0 Hz, 1H), 6.14 (dd, J = 6.5, 5.8 Hz, 1H), 3.86 (s, 9H). ^{13}C NMR (125 MHz, DMSO) δ 164.50, 153.36, 145.29, 138.59, 134.59, 134.51, 127.93, 117.56, 116.21, 115.08, 105.15, 68.73, 61.04, 56.17. MS: 314.; Anal.Calc. for: $C_{12}H_{18}N_2O_4$: Elemental Analysis: C,64.96;H,5.77;N,8.91.

2.3.10. 2-(p-tolyl)-2,3-dihydroquinazolin-4(1H)-one (3j).

MP: 226; UV:212; IR(KBr): ν_{max} 3310, 3194, 3060, 1654, 1607, 1508, 1484 cm^{-1}. ^1H NMR (500 MHz, DMSO) δ 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.44 – 7.38 (m, 2H), 7.37 (td, J = 8.0, 1.4 Hz, 1H), 7.30 (td, J = 8.1, 1.7 Hz, 1H), 7.16 – 7.10 (m, 2H), 6.97 (dd, J = 8.2, 1.5 Hz, 1H), 6.17 (dd, J = 6.6, 5.8 Hz, 1H), 5.86 (d, J = 6.4 Hz, 1H), 5.80 (d, J = 6.0 Hz, 1H), 2.38 (s, 3H). ^{13}C NMR (125 MHz, DMSO) δ 164.34, 145.62, 138.46, 138.13, 134.51, 129.40, 127.93, 127.76, 117.56, 116.09, 115.12, 67.76, 21.06. MS:238.; Anal. Calc. for: $C_{15}H_{14}N_2O$: Elemental Analysis: C,75.61; H,5.92; N,11.76.

2.3.11. 2-(4-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (3k).

MP: 207; UV: 211; IR (KBr): 3339, 3170, 3065, 1660, 1620, 1588, 1563, 1485 cm^{-1}. ^1H NMR (500 MHz, DMSO) δ 7.97 (dd, J = 8.2, 1.6 Hz, 1H), 7.52 – 7.46 (m, 2H), 7.37 (td, J = 8.0, 1.4 Hz, 1H), 7.30 (td, J = 8.1, 1.6 Hz, 1H), 7.27 – 7.22 (m, 2H), 6.97 (dd, J = 8.2, 1.5 Hz, 1H), 6.17 (dd, J = 6.6, 5.8 Hz, 1H), 5.86 (d, J = 6.4 Hz, 1H), 5.80 (d, J = 6.0 Hz, 1H). ^{13}C NMR (125 MHz, DMSO) δ 164.34, 145.62, 138.77, 135.20, 134.51, 129.62, 128.74, 127.93, 117.56, 116.09, 115.12, 66.87. MS: 258; Anal. Calc. for: $C_{14}H_{11}ClN_2O$: Elemental Analysis: C, 65.00; H,4.29; Cl,13.70; N,10.83.

2.3.12. (E)-2-styryl-2,3-dihydroquinazolin-4(1H)-one (3l).

MP: 195; UV: 212; IR (KBr): 3296 (NH), 3191 (NH), 3071 (CH) (NH-CH-NH), 2925 (C=C-H), 1658 (C=O), 1610 (CONH). ^1H NMR (500 MHz, DMSO) δ 7.93 (dd, J = 8.1, 1.6 Hz, 1H), 7.41 – 7.32 (m, 5H), 7.35 – 7.23 (m, 2H), 6.96 (dd, J = 8.2, 1.5 Hz, 1H), 6.50 – 6.42 (m, 1H), 6.38 (d, J = 6.2 Hz, 1H), 5.93 (dd, J = 16.3, 3.7 Hz, 1H), 5.71 (d, J = 6.2 Hz, 1H), 5.63 (tdd, J = 6.2, 3.7, 1.7 Hz, 1H). ^{13}C NMR (125 MHz, DMSO) δ 164.89, 145.53, 137.97, 134.68, 130.12, 128.97, 128.83, 127.86, 127.15, 118.51, 117.45, 115.46, 115.24, 65.65. MS:250.; Anal. Calc. for: $C_{16}H_{14}N_2O$: Elemental Analysis: C,76.78; H,5.64; N,11.19.

2.3.13. 2-phenyl-2,3-dihydroquinazolin-4(1H)-one (3m).

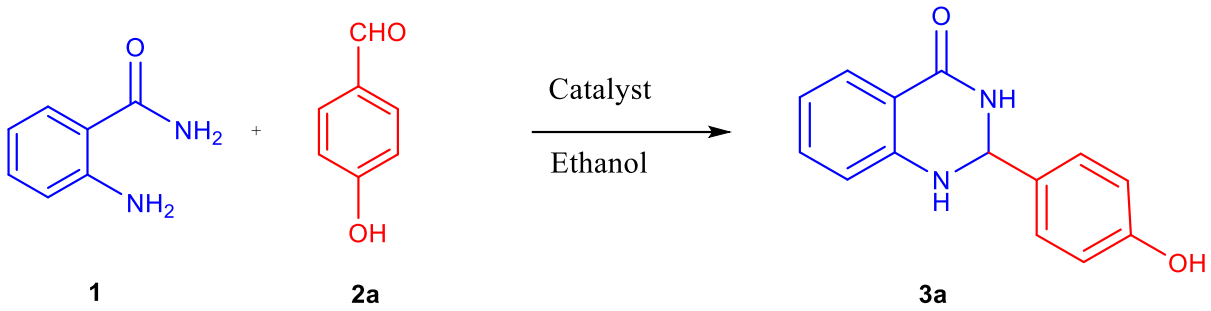
MP: 216.; UV:211; IR (KBr): 3294, 3188, 3068, 1653, 1603 cm^{-1}. ^1H NMR (500 MHz, DMSO-d6): δ 7.93 (d, 1H) 7.54−7.23 (m, 6H), 6.90 (t,1H), 6.65 (d, 1H), 5.88 (s, 1H), 5.80 (s, 1H), 4.38 (s, 1H). ^{13}C NMR (125 MHz, DMSO-d6): δ 169.2, 152.4, 145.4, 138.2, 133.4, 133.1,

132.2, 132.0, 122.3, 119.4, 119.2, 72.3.; MS: 224; Anal. Calc. for: $C_{14}H_{12}N_2O$: Elemental Analysis: C,74.98; H,5.39; N,12.49.

3. Results and Discussion

3.1. Effect of solvent and catalyst.

The reaction between anthranilamide (1a) and 4-hydroxybenzaldehyde (2a) was chosen as the model system to govern the optimal condition for the synthesis of 2-(4-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (3a) (Scheme 1, Table 1). The reaction was carried out over polyaniline-supported zinc oxide to find out the best catalyst for this transformation. Among the catalysts examined, polyaniline-supported zinc oxide exhibited higher catalytic activity and gave the corresponding product.



Scheme 1. Synthesis of 2-(4-hydroxyphenyl)-2,3-dihydroquinazoline-4(1H)one.

Reaction condition (3a): Anthranilamide (1) (1mmol), hydroxybenzaldehyde (2a) (1mmol), ethanol, catalyst, reflux 2-5 hr.

We have performed various catalysts and ethanol as a solvent. In this scheme, we perform anthranilamide (1a) (1mmol), anthranilamide (2) (1mmol), various catalysts, and ethanol as a solvent at reflux temperature. We have the optimization of the reaction condition for scheme 1, as a model reaction. Also, we have checked the altered catalyst, solvent time, and yield results of these studies and summarised them in Table 1.

Table 1. Screening of various catalysts for synthesis of compound (3a).

Entry	Catalyst	Medium	Time (min)	Yield (%)
1	None	Ethanol	-	-
2	Oxalic Acid	Ethanol	45	70
3	Molecular Iodine	Ethanol	40	65
4	TBAB	Ethanol	50	55
5	DBSA	Ethanol	180	45
6	**PANI ZnO**	**Ethanol**	**30**	**90**

Reaction condition (3a): Anthranilamide (1) (1mmol), 4-hydroxybenzaldehyde (2a) (1mmol), ethanol, catalyst, reflux, 30-180 min.

Using the optimised amount of the catalyst, the model reaction was investigated in different solvents water, ethanol, water-ethanol (1:1) mixture, acetonitrile, toluene, DCM, and methanol to select the suitable reaction condition. The result indicated that the optimum yield of the product was obtained with ethanol at a shorter reaction time. It was also observed that the yield of the product increases as temperature increases.

Table 2. Screening of the solvent for the synthesis of compound (3a)[a].

Entry	PANI ZnO (mol %)	Solvent	Temperature	Time (h)	Yield[b]
1	0	water	RT	9	37
2	1	DMF	RT	7	40
3	2	Methanol	RT	5	30
4	3	Ethanol	RT	4	75
5	1	DMF	Reflux	5	50
6	2	Methanol	Reflux	4	35
7	**3**	**Ethanol**	**Reflux**	**1.5**	**96**
8	4	Ethanol	Reflux	1.5	95
9	5	Ethanol	Reflux	1.5	94

[a]Reaction condition (3a): Anthranilamide (1) (1mmol), 4-hydroxybenzaldehyde (2a) (1mmol) , solvent , PANI-ZnO (mol%) reflux 1.5-9 h. [b]Isolated yield.

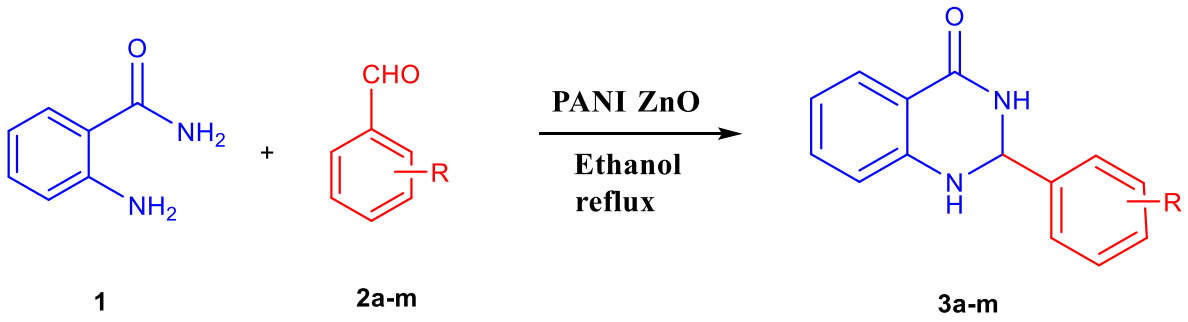
We have performed the effect of solvent on the synthesis of compound **(3a),** shown in Table 2. The solvent plays a key role in the catalyst's activity and results. Here, we have observed that ethanol is a solvent over a PANI-ZnO catalyst, which allowed us to evaluate catalyst loading further. The results are summarized in Table 2. The results reveal that (entry 7) 3 mol % of the PANI-ZnO catalyst loading is sufficient for the required yield. In the case of 4 mol% (entry 8) and 5 mol% (entry 9), PANI-ZnO loading did not show any considerable increase in the yield of the desired product. This result shows the scope of methodology for synthesizing 2,3-dihydroquinazolin-4(1H)-one from aldehyde and anthranilamide in optimized reaction conditions. The 3 mol% of PANI-ZnO catalyst and ethanol as a solvent is standard protocol to carry out further reactions.

Table 3. Recycling of PANI-ZnO for the synthesis of compound (3a).

Sr. No.	Run	Catalyst recovery (%)	Time (h)	Product Yield
1	1	97	1	98
2	2	96	1	94
3	3	95	1	88
4	4	94	1	86

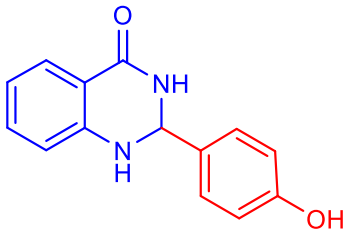
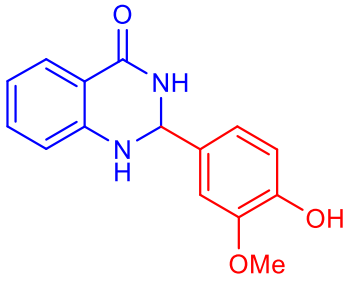
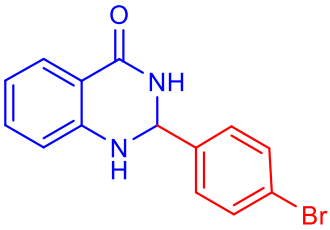
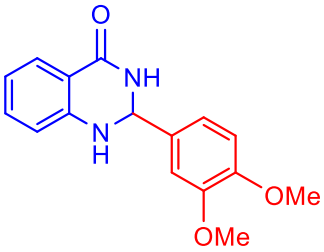
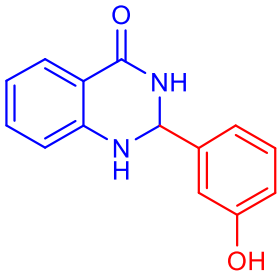
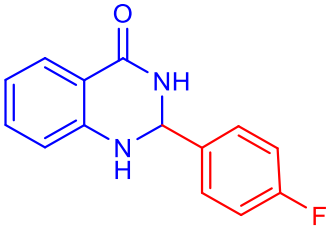
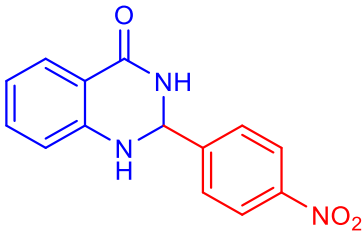
3.2. Recycling of the catalyst.

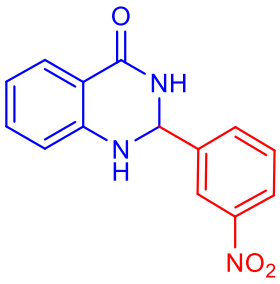
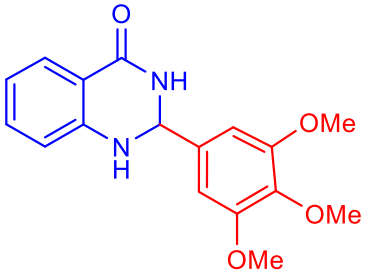
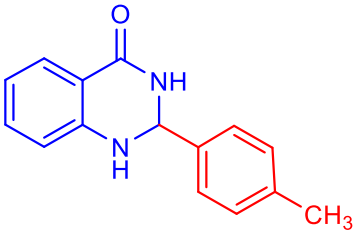
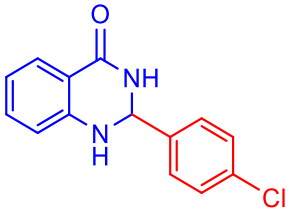
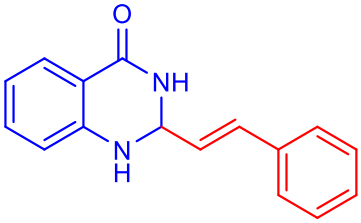
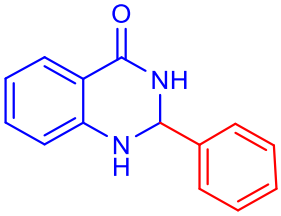
We have also found that PANI-ZnO catalyst recyclability for the given reaction of aldehyde (2a), anthranilamide (1) in ethanol solvent at reflux for 1.5 h. and the result were depicted in Table 3. Polyaniline-supported zinc oxide catalyst was recovered by filtration and reused it without loss of catalytic activity.



Scheme 2. Synthesis of 2-(4-chlorophenyl)-2,3-dihydroquinazoline-4(1H)one. Reaction condition *(3a-m):* Anthranilamide (1) (1mmol), substituted benzaldehyde (2a-m) (1mmol), ethanol, polyaniline supported zinc oxide (4%), reflux 1-2 hr.

Table 4. Physical data of synthesized compounds (3a-m).

Entry	R	Product	Yield (%)[b]	Time (h)	M. P. (°C)
3a	4-OH		98	1	182
3b	3-OMe,4-OH		89	2	185
3c	4-Br		92	1.5	196
3d	3-OMe,4-OMe		89	1	181
3e	3-OH		87	1	207
3f	4-F		90	2	193
3g	4-NO_2		88	1	213

Entry	R	Product	Yield (%)[b]	Time (h)	M. P. (ºC)
3h	3-NO_2		89	2	211
3i	3,4,5-OMe		91	1.5	185
3j	4-Me		90	2	226
3k	4-Cl		90	2	207
3l	PhCH=CH		89	2	195
3m	4-H		88	2	216

Reaction condition (3a-m): Anthranilamide (1) (1mmol), substituted benzaldehyde (2a-m) (1mmol), ethanol, polyaniline supported zinc oxide (4%), reflux 1-2 hr.

3.3. X-ray diffraction study.

The XRD spectrum of polyaniline (PANI) is shown in Figure 1. It observed that a broad and diffused peak at around 2Θ = 25 degrees. This shows polyaniline is amorphous in nature. Figure 1 shows the X-ray diffraction pattern of PANI-ZnO nanocomposites for 4 wt %. The FTIR spectra of PANI-ZnO nanocomposite for 4 wt % are shown in Figure 2

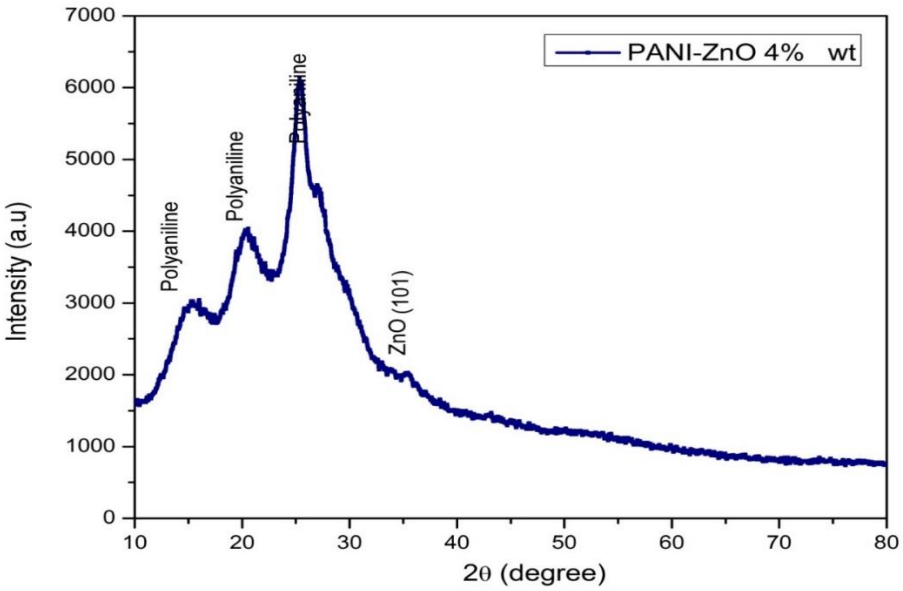


Figure 1. XRD pattern of PANI-ZnO nanocomposite 4 wt %.

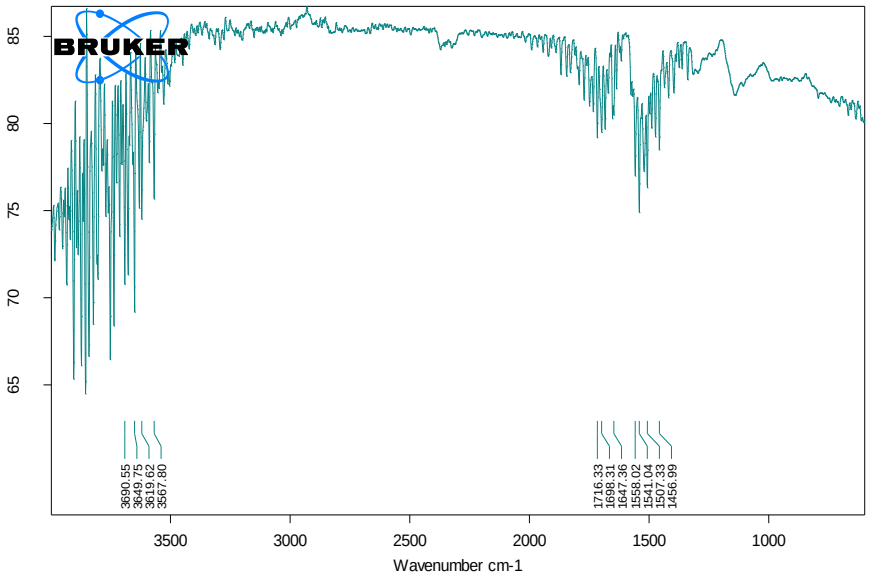


Figure 2. FTIR spectra of PANI-ZnO nanocomposite 4 wt %.

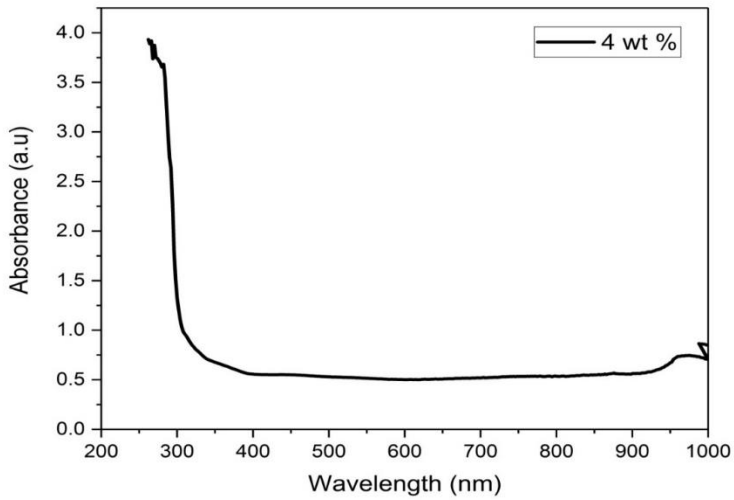


Figure 3. UV-Visible absorption spectra of PANI-ZnO nanocomposite 4 wt %.

4. Conclusions

In conclusion, an environmentally and highly efficient green protocol has been established for the synthesis of 2,3-dihydroquninazolin-4(1H)-one derivative by using polyaniline-supported zinc oxide nanocomposite (**3a-m**) using an inexpensive and recoverable polyaniline zinc oxide (PANI–ZnO) catalyst with ethanol solvent condition. In this method, our protocol is convenient. It offers several advantages, such as higher yield, shorter reaction time, clean reaction profiles, easy workup procedure, recycling of the catalyst, and eco-friendliness.

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Conflicts of Interest

The authors declare no conflict of interest.

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Supplementary materials

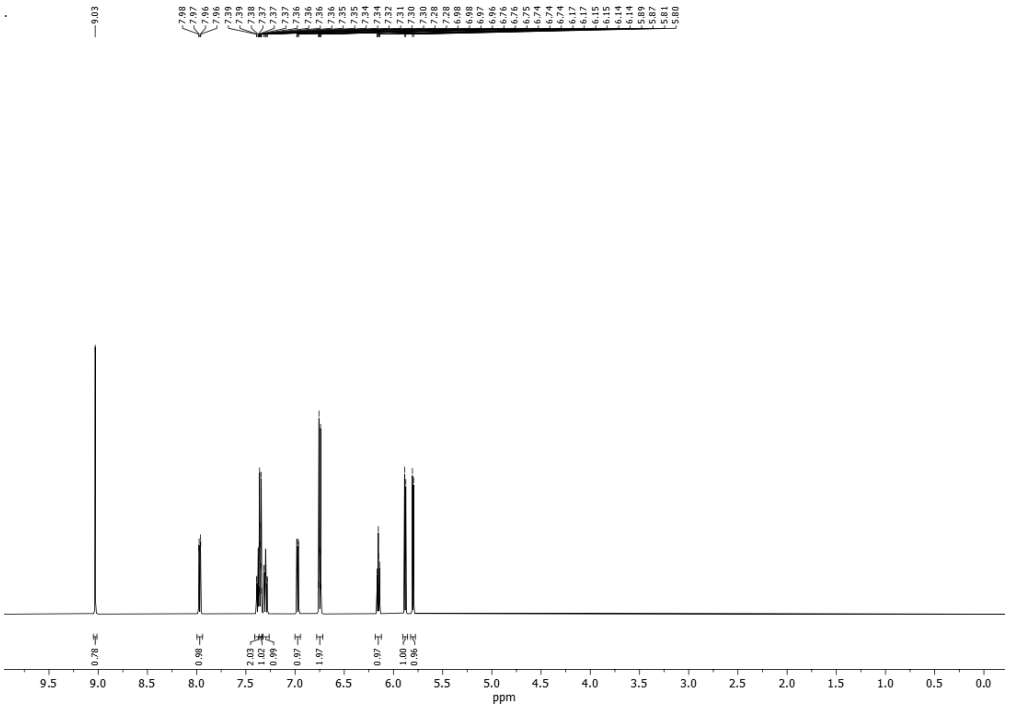

Figure S1. [1]H NMR spectra of 2-(4-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one.

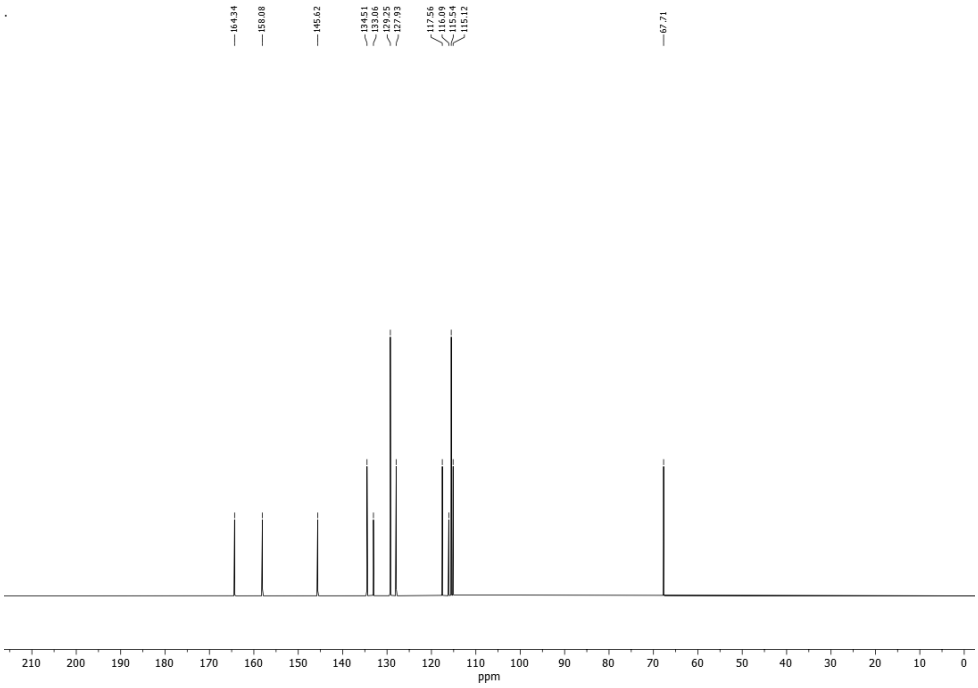

Figure S2. [13]C NMR spectra of 2-(4-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one

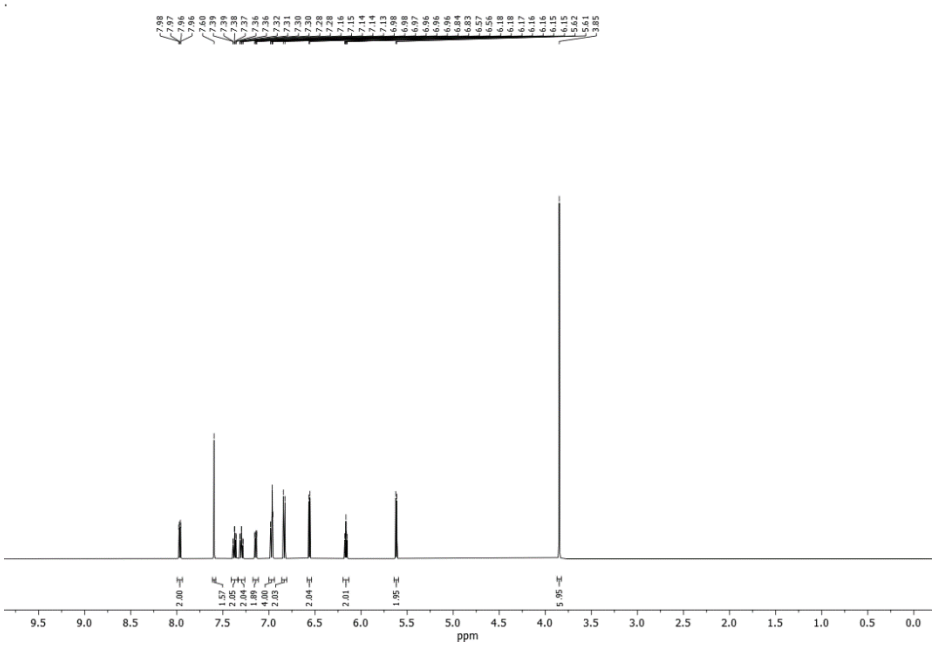

Figure S3. [1]H NMR spectra of 2-(4-hydroxy-3-methoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one.

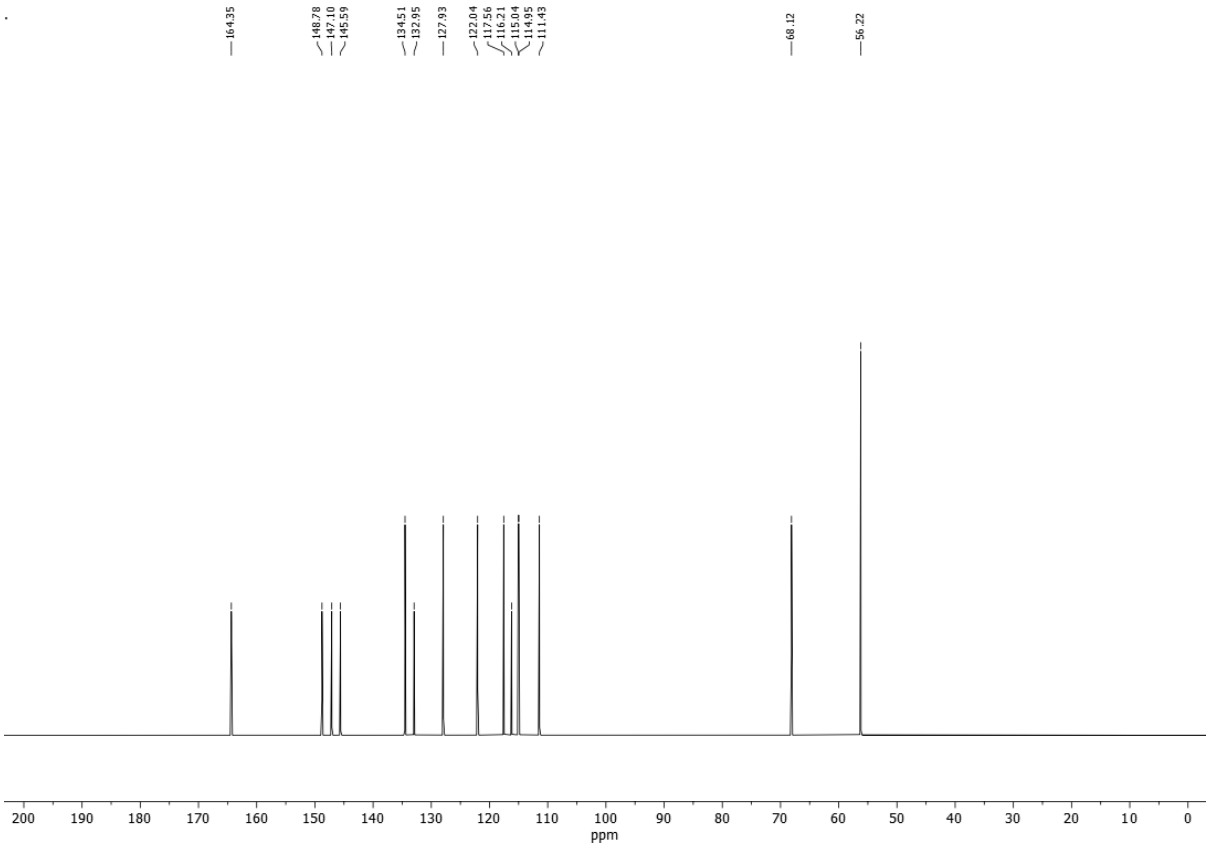

Figure S4. [13]C NMR spectra of 2-(4-hydroxy-3-methoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one.

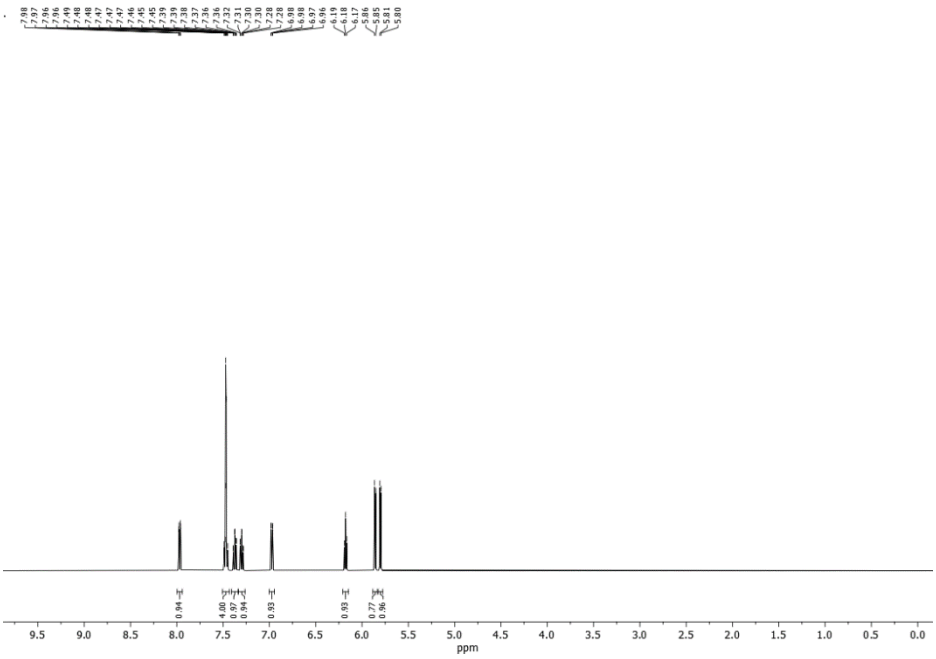

Figure S5. ^{1}H NMR spectra of 2-(4-bromophenyl)-2,3-dihydroquinazolin-4(1H)-one.

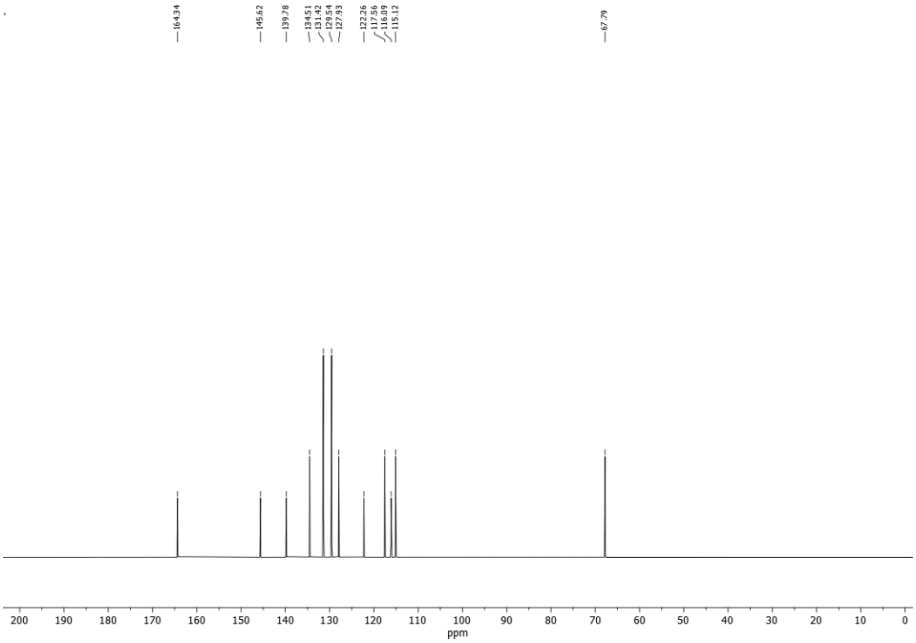

Figure S6. ^{13}C NMR spectra of 2-(4-bromophenyl)-2,3-dihydroquinazolin-4(1H)-one.

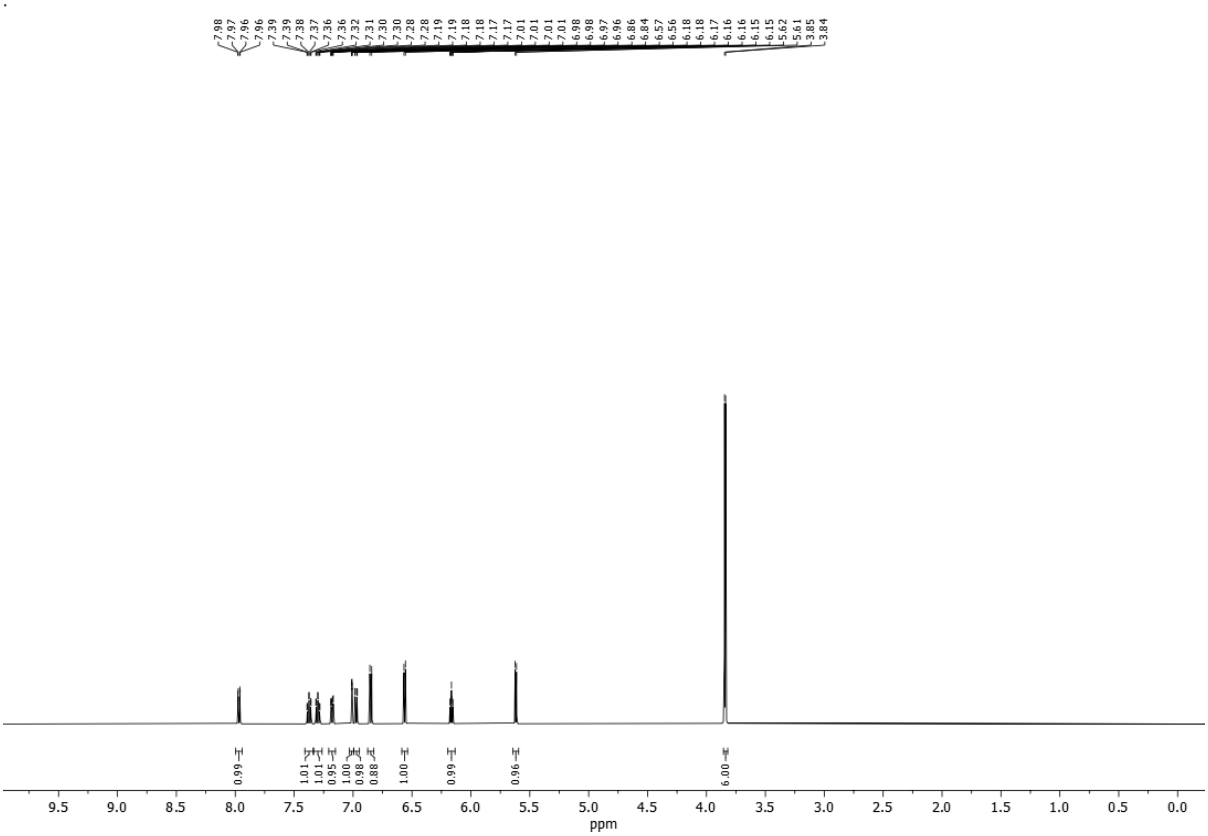

Figure S7. [1]H NMR spectra of 2-(3,4-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one.

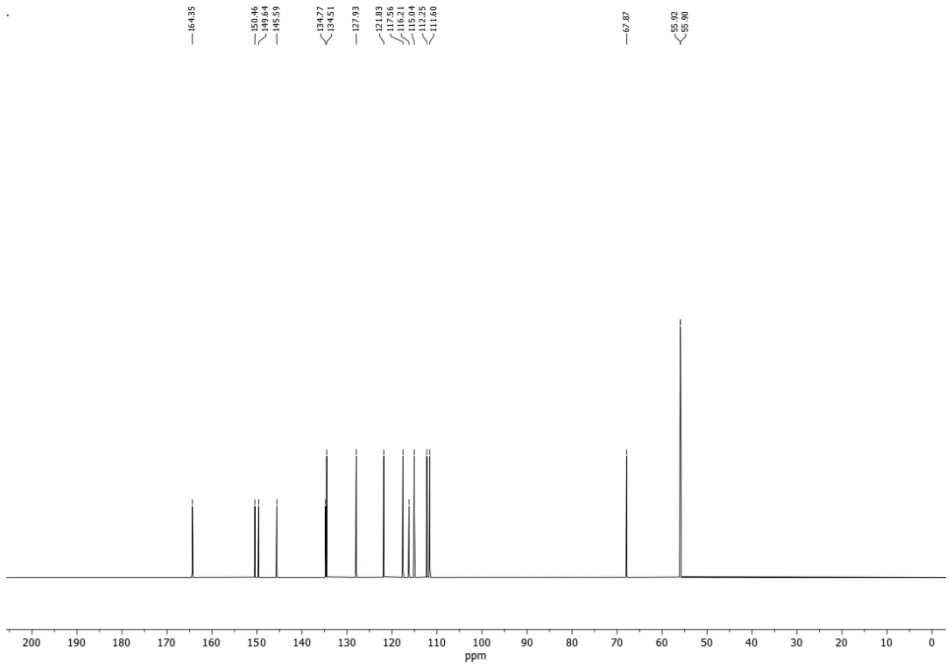

Figure S8. [13]C NMR spectra of 2-(3,4-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one.

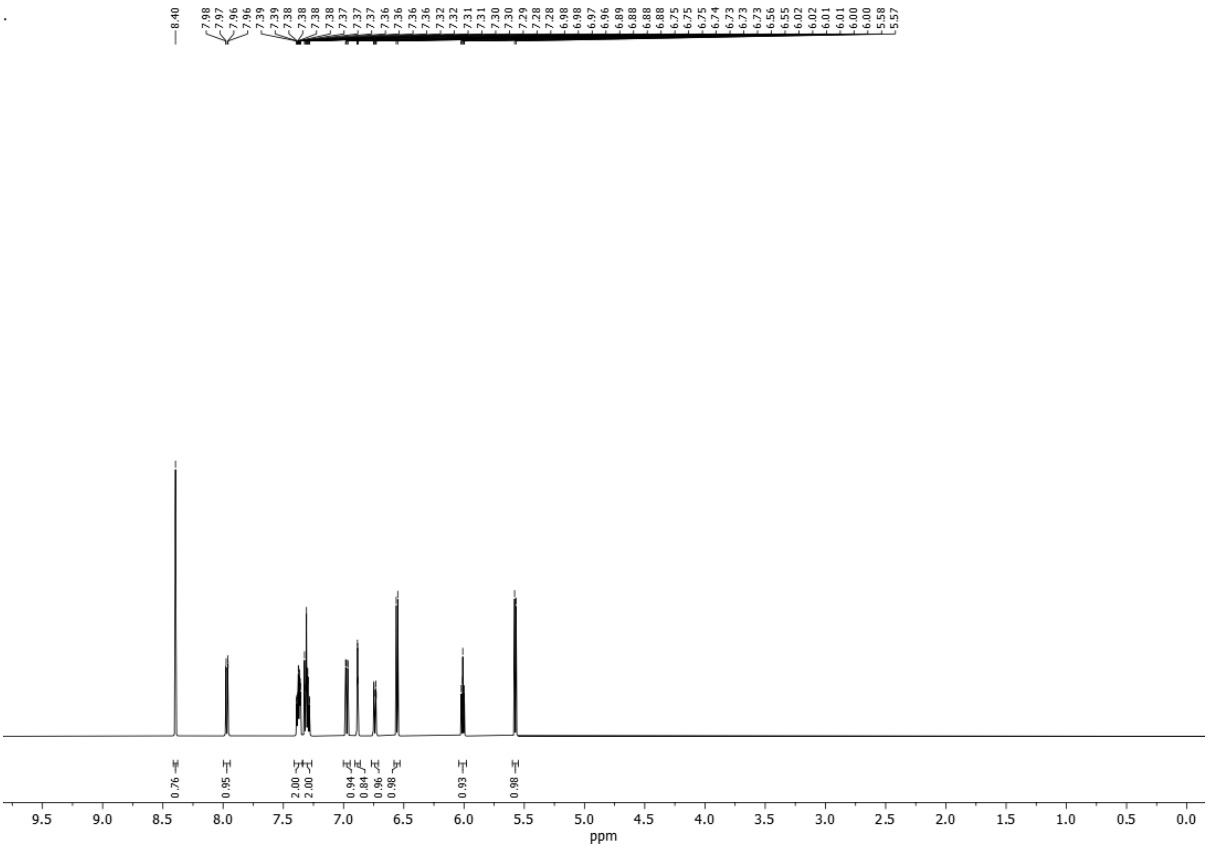

Figure S9. ^{1}H NMR spectra of 2-(3-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one.

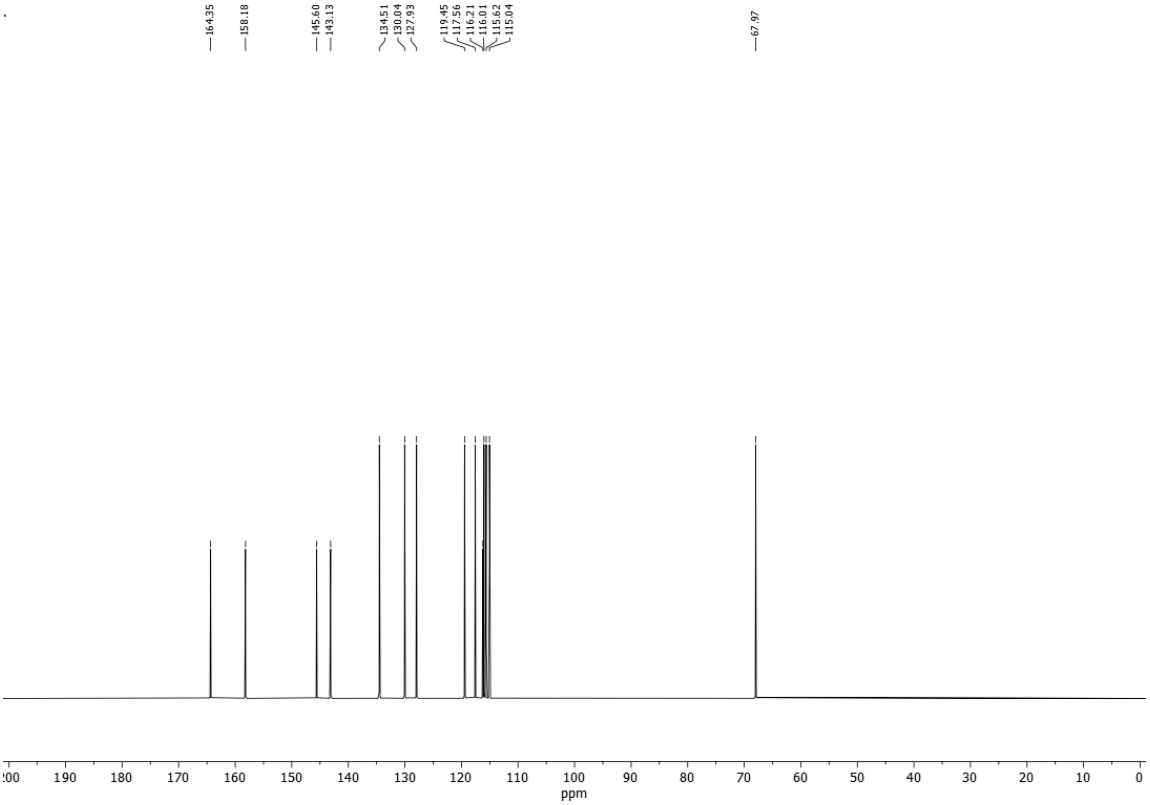

Figure S10. ^{13}C NMR spectra of 2-(3-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one.

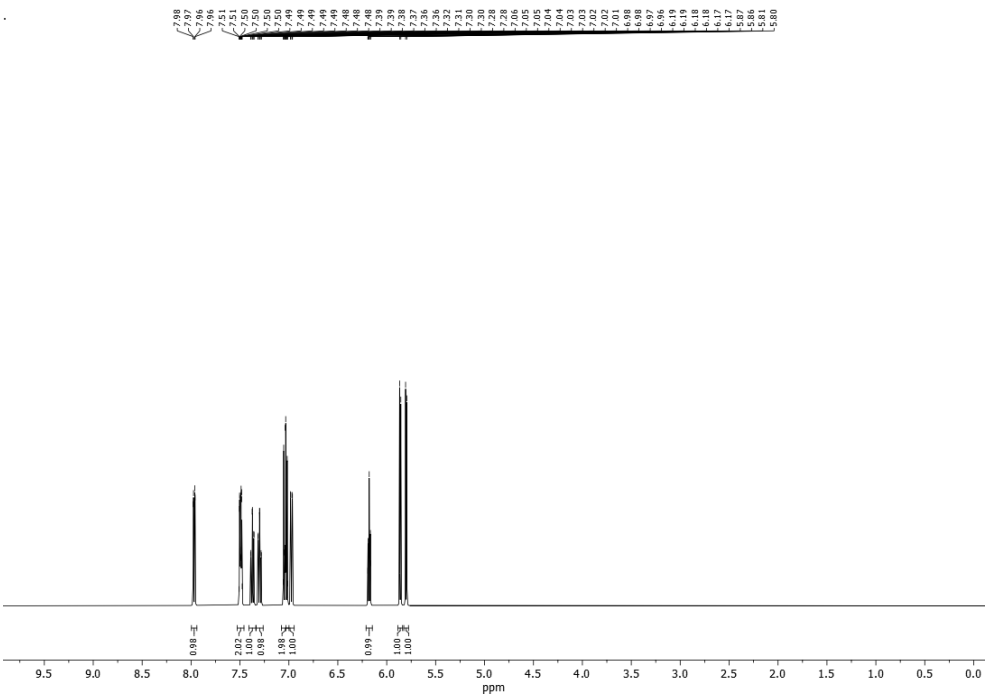

Figure S11. [1]H NMR spectra of 2-(4-fluorophenyl)-2,3-dihydroquinazolin-4(1H)-one

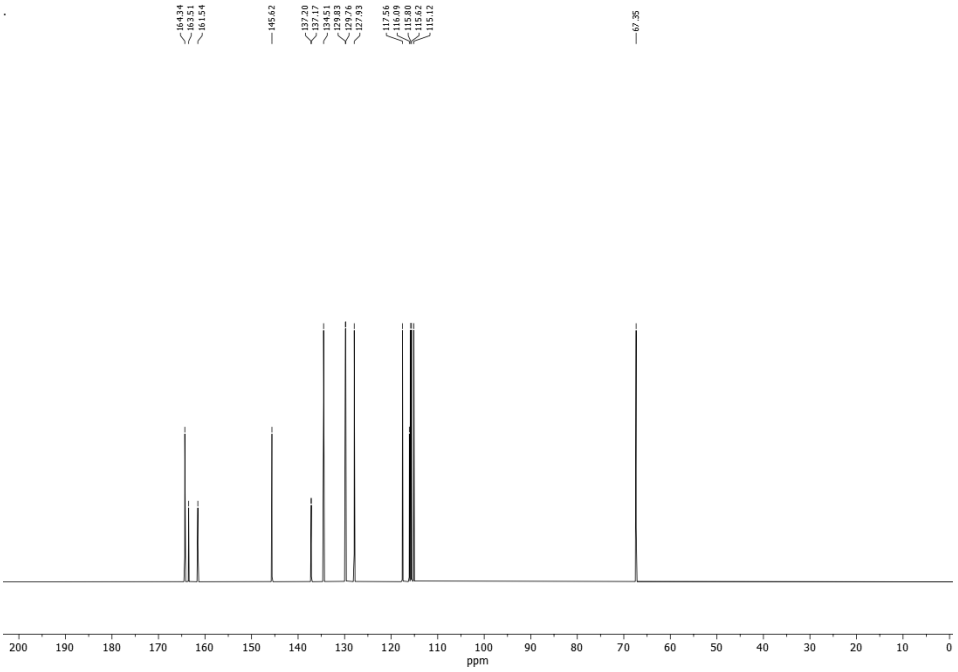

Figure S12. [13]C NMR spectra of 2-(4-fluorophenyl)-2,3-dihydroquinazolin-4(1H)-one.

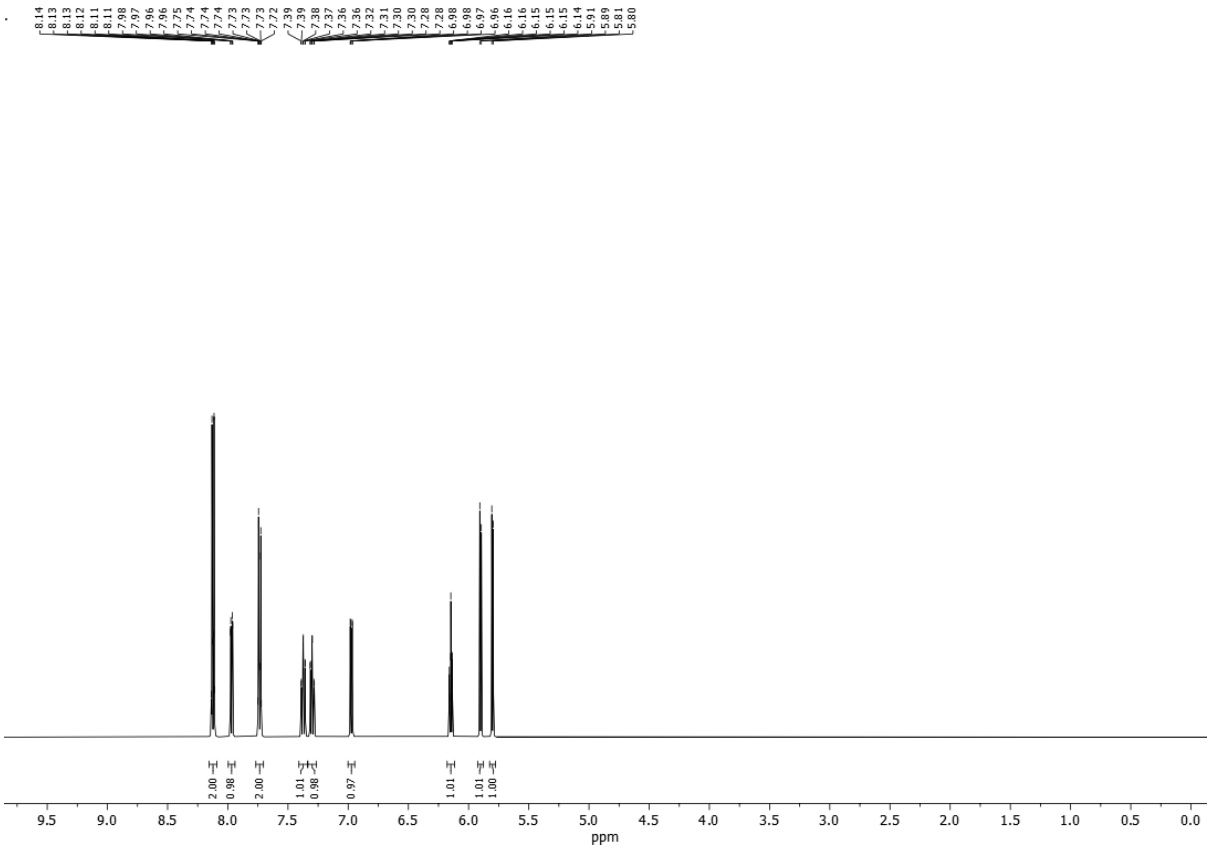

Figure S13. ^{1}H NMR spectra of 2-(4-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one.

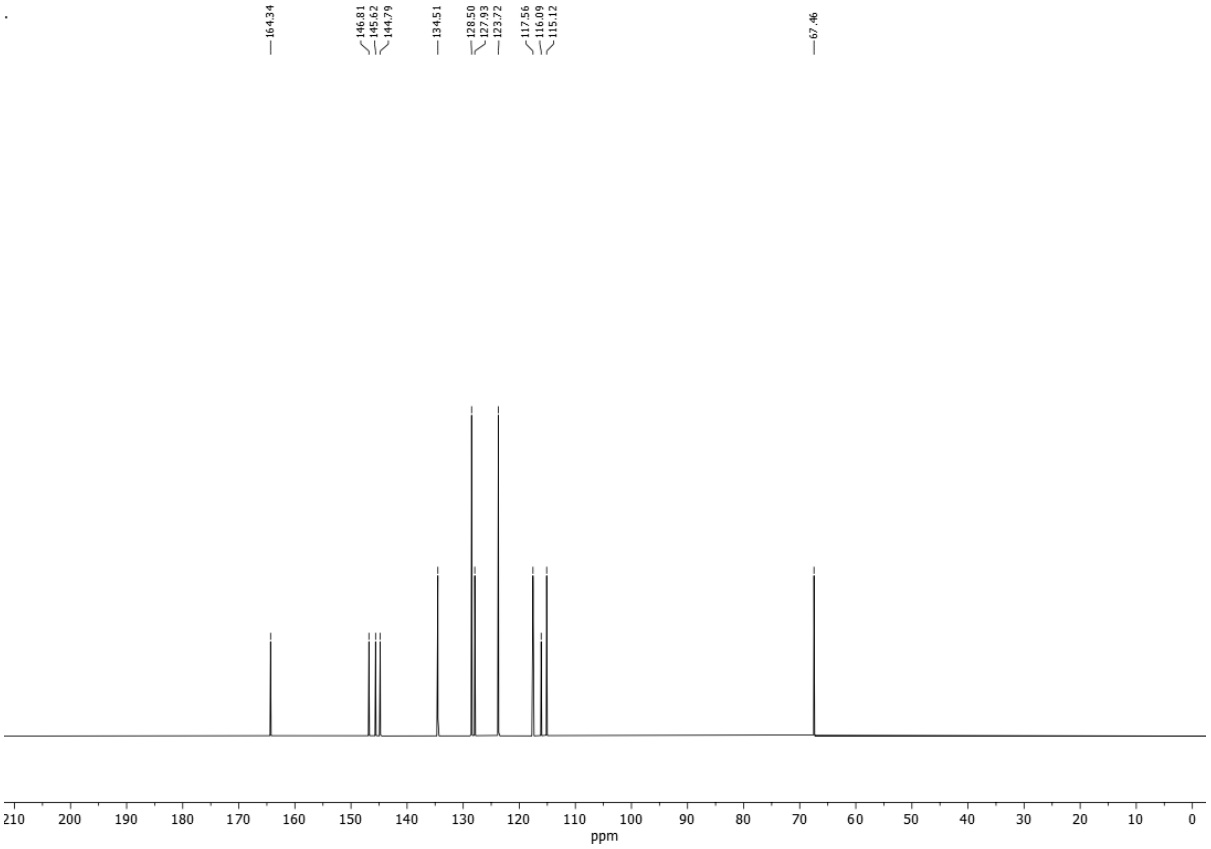

Figure S14. ^{13}C NMR spectra of 2-(4-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one.

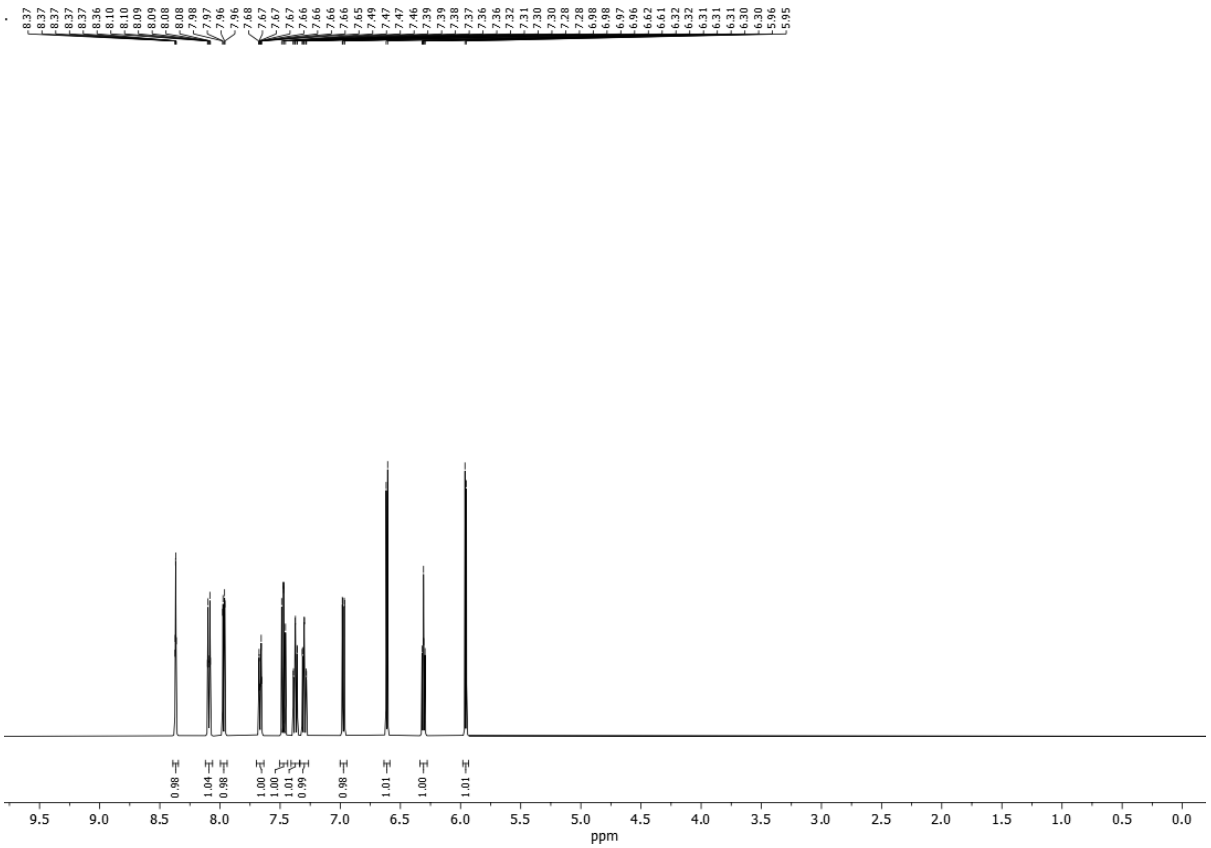

Figure S15. [1]H NMR spectra of 2-(3-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one.

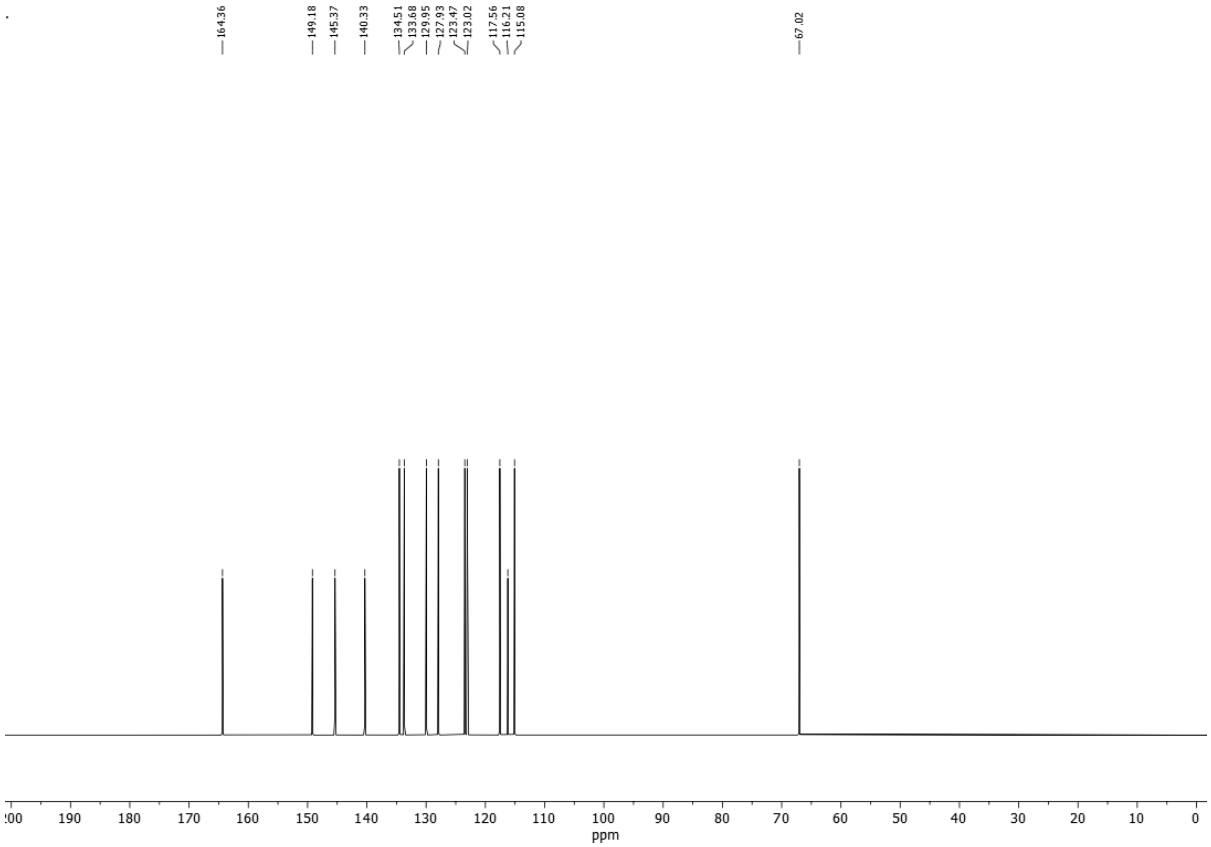

Figure S16. [13]C NMR spectra of 2-(3-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one.

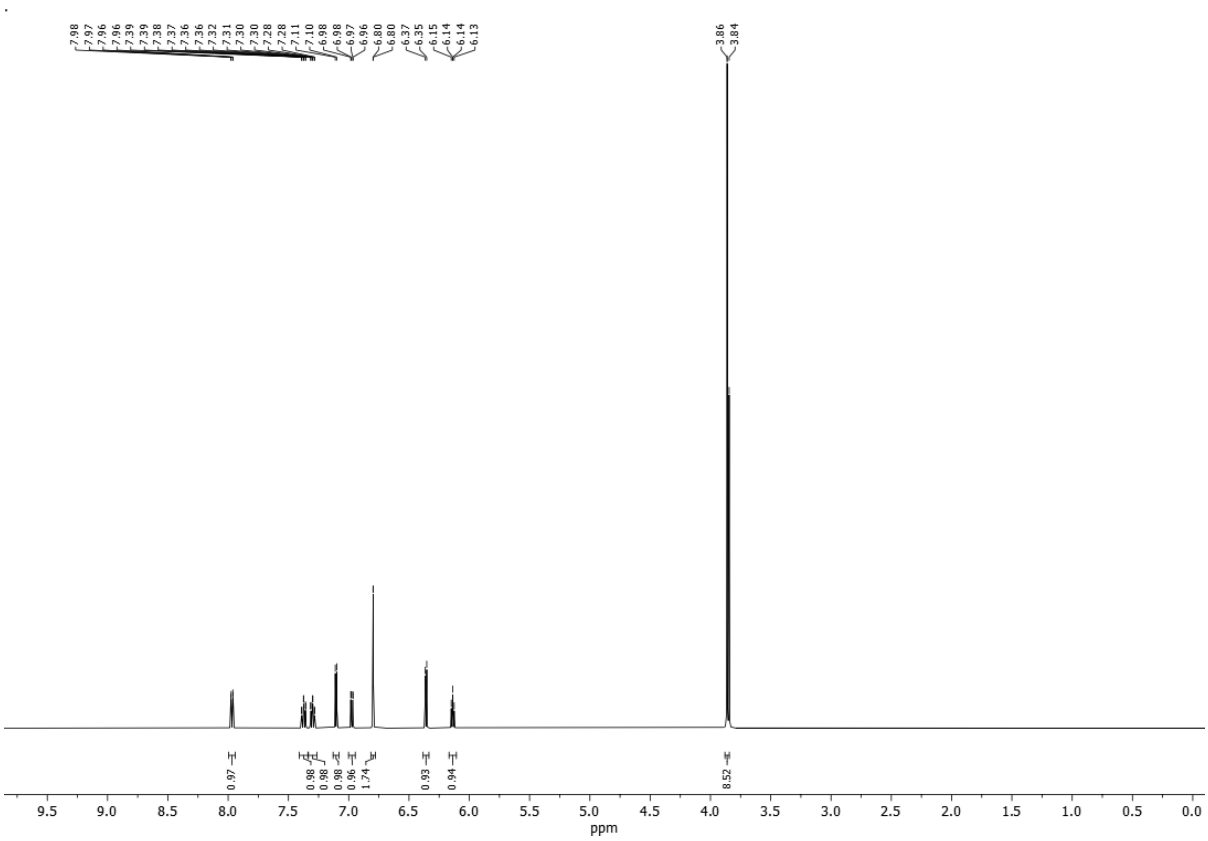

Figure S17. [1]H NMR spectra of 2-(3,4,5-trimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one.

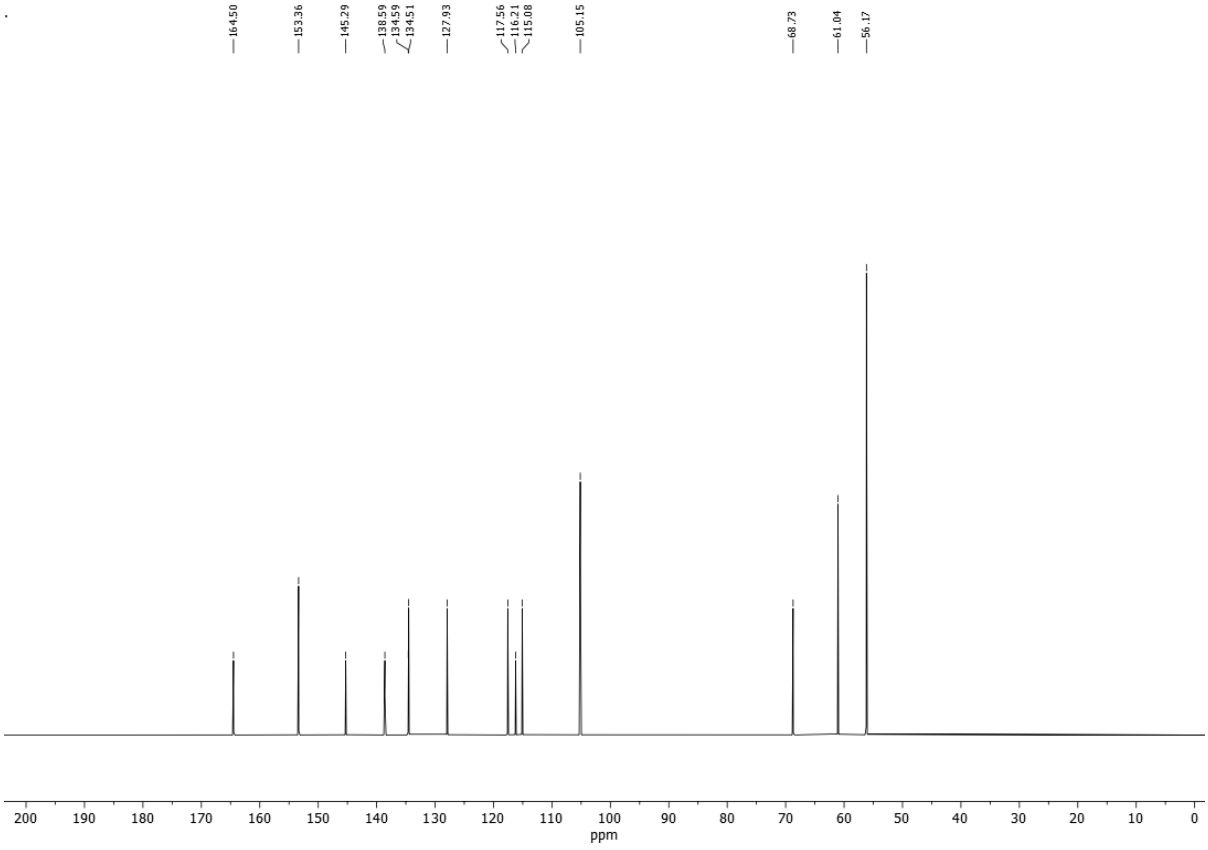

Figure S18. [13]C NMR spectra of 2-(3,4,5-trimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one.

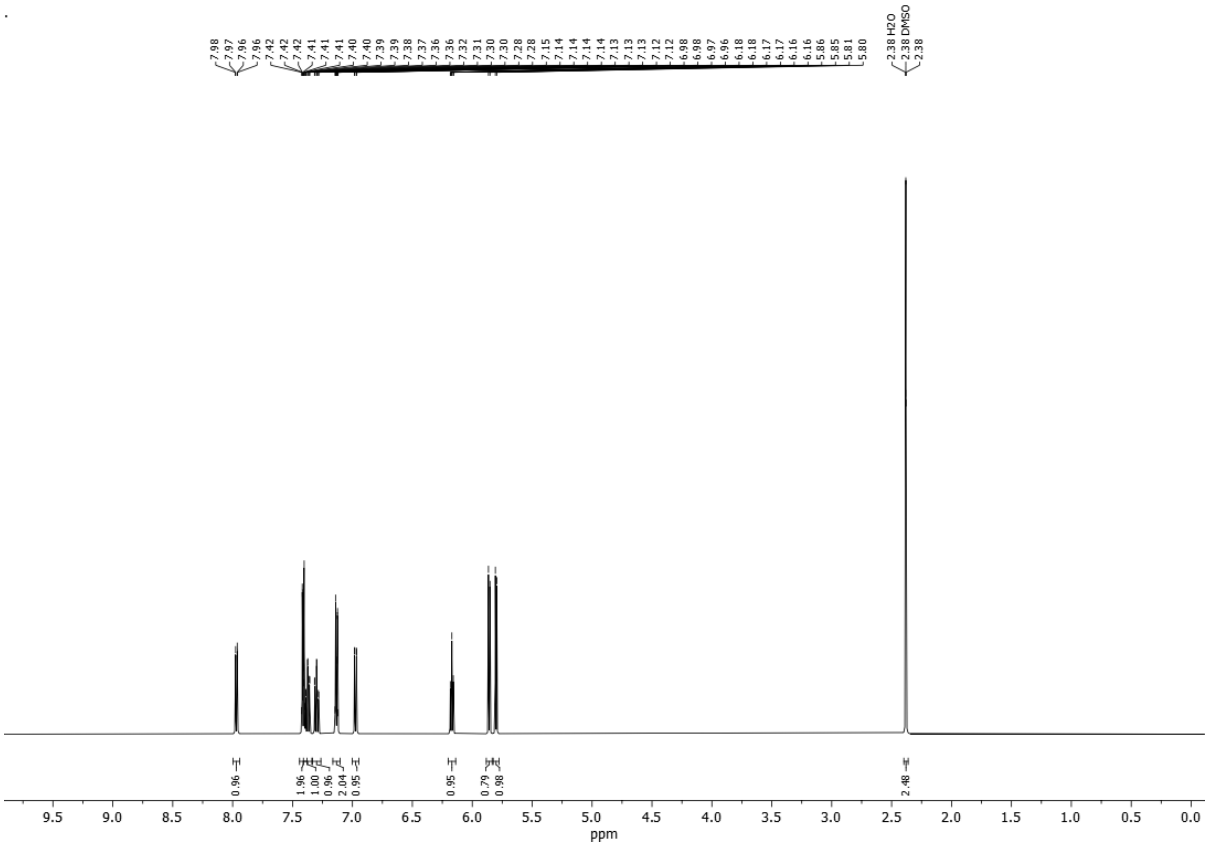

Figure S19. [1]H NMR spectra of 2-(p-tolyl)-2,3-dihydroquinazolin-4(1H)-one.

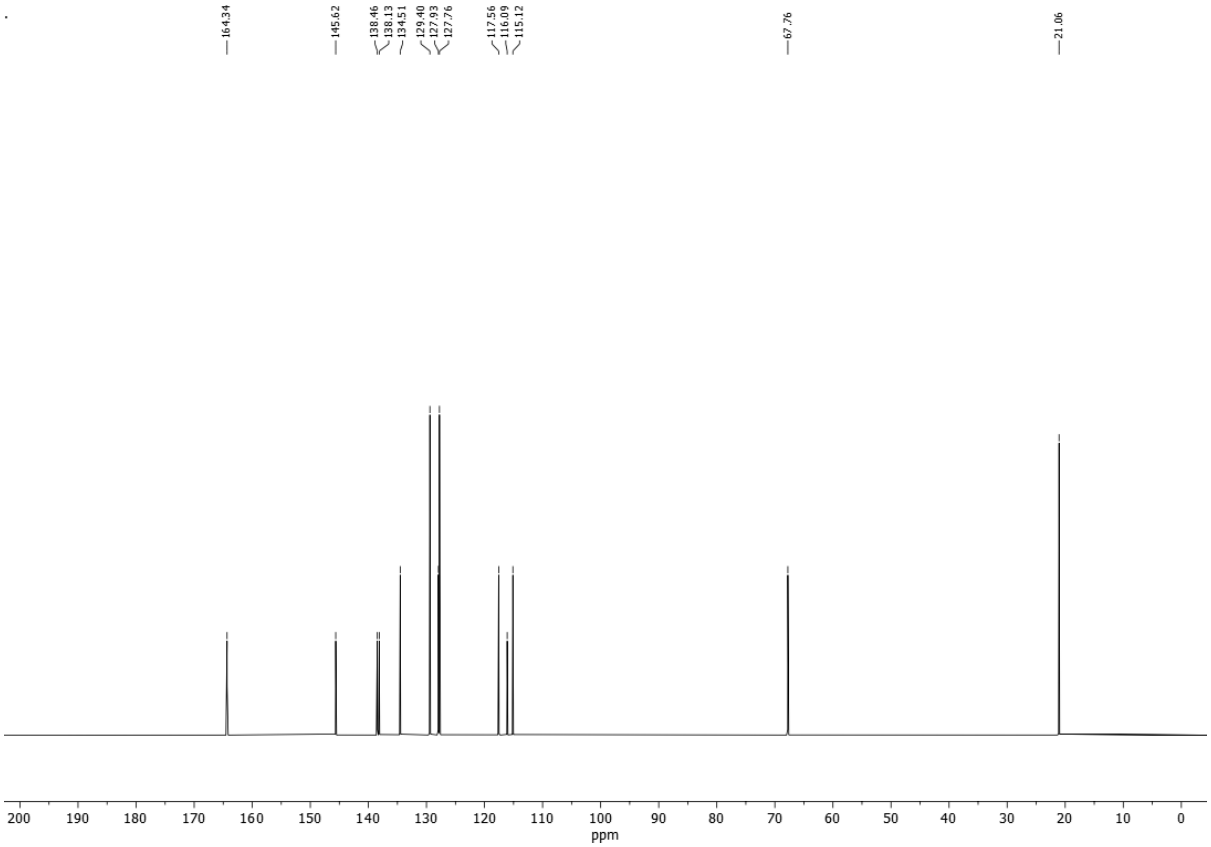

Figure S20. [13]C NMR spectra of 2-(p-tolyl)-2,3-dihydroquinazolin-4(1H)-one.

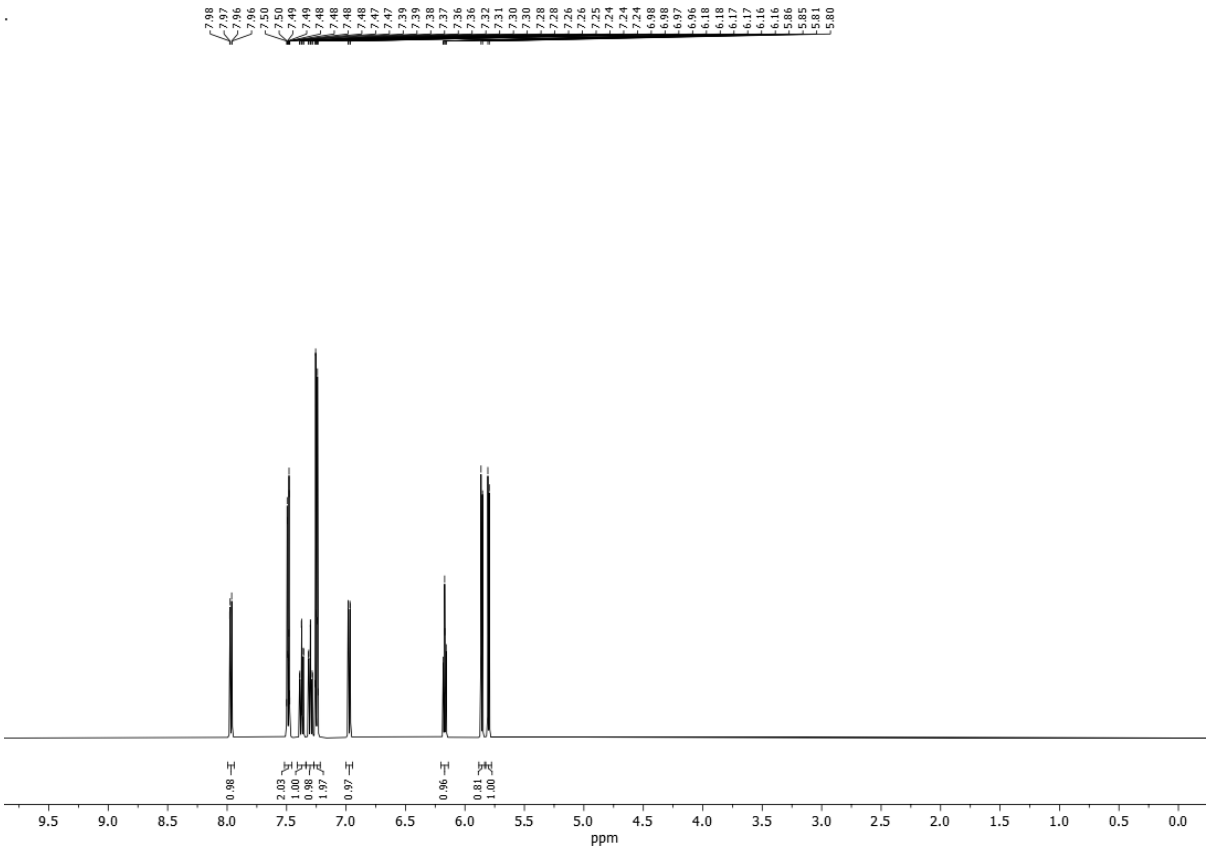

Figure S21. [1]H NMR spectra of 2-(4-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one.

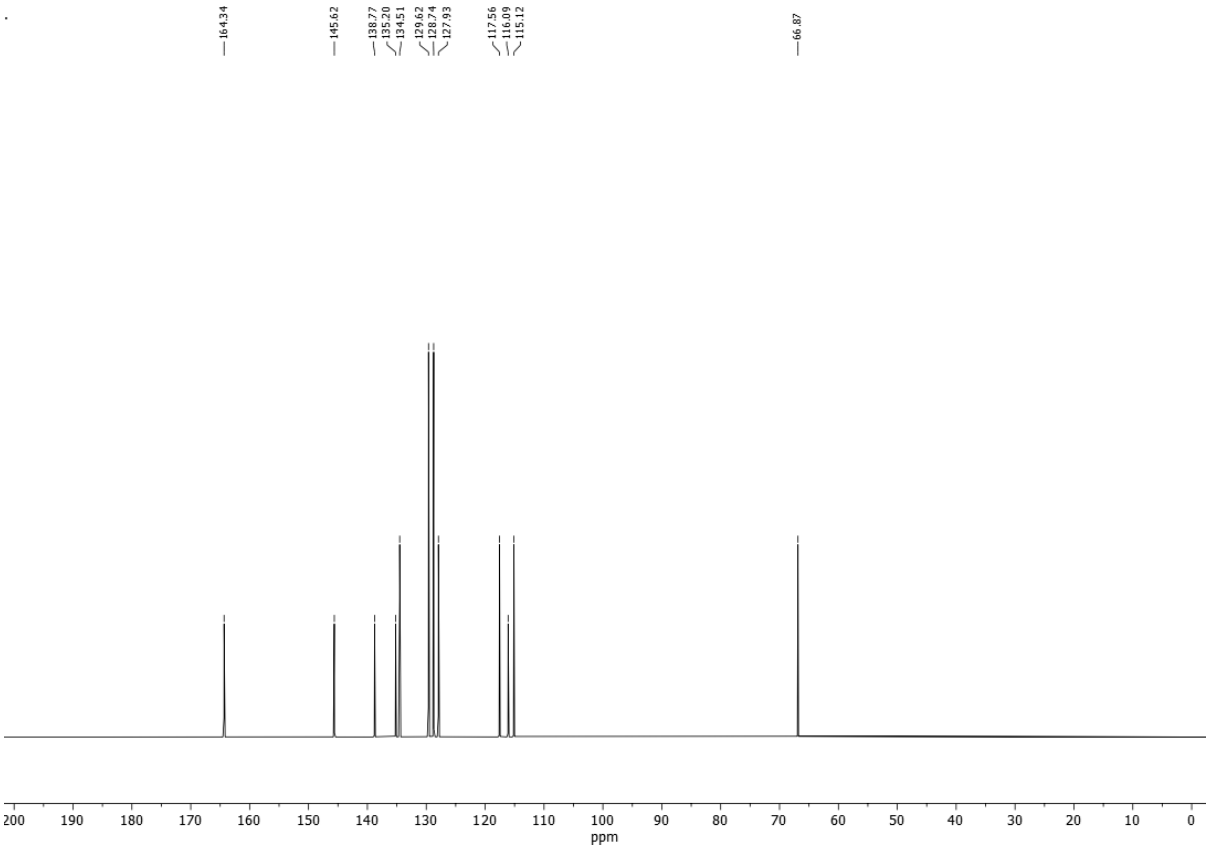

Figure S22. [13]C NMR spectra of 2-(4-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one.

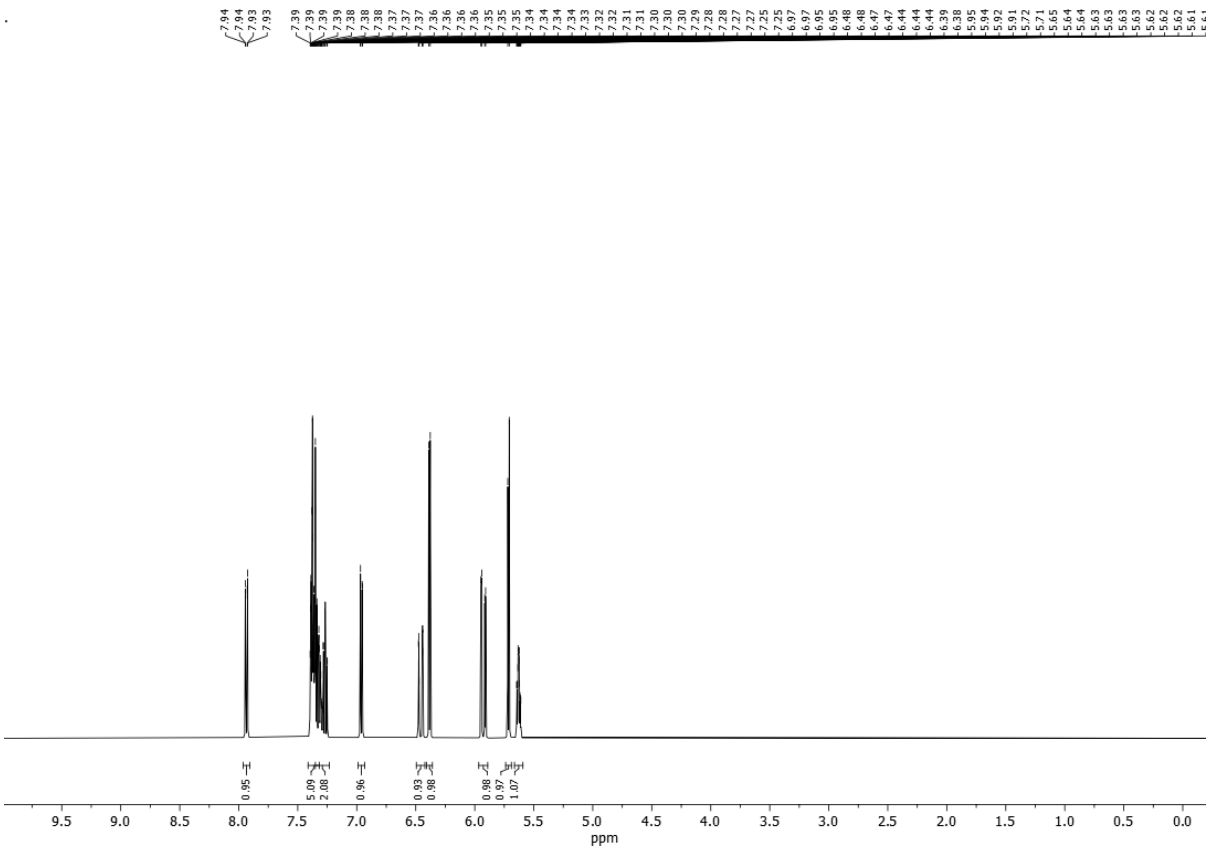

Figure S23. [1]H NMR spectra of (E)-2-styryl-2,3-dihydroquinazolin-4(1H)-one.

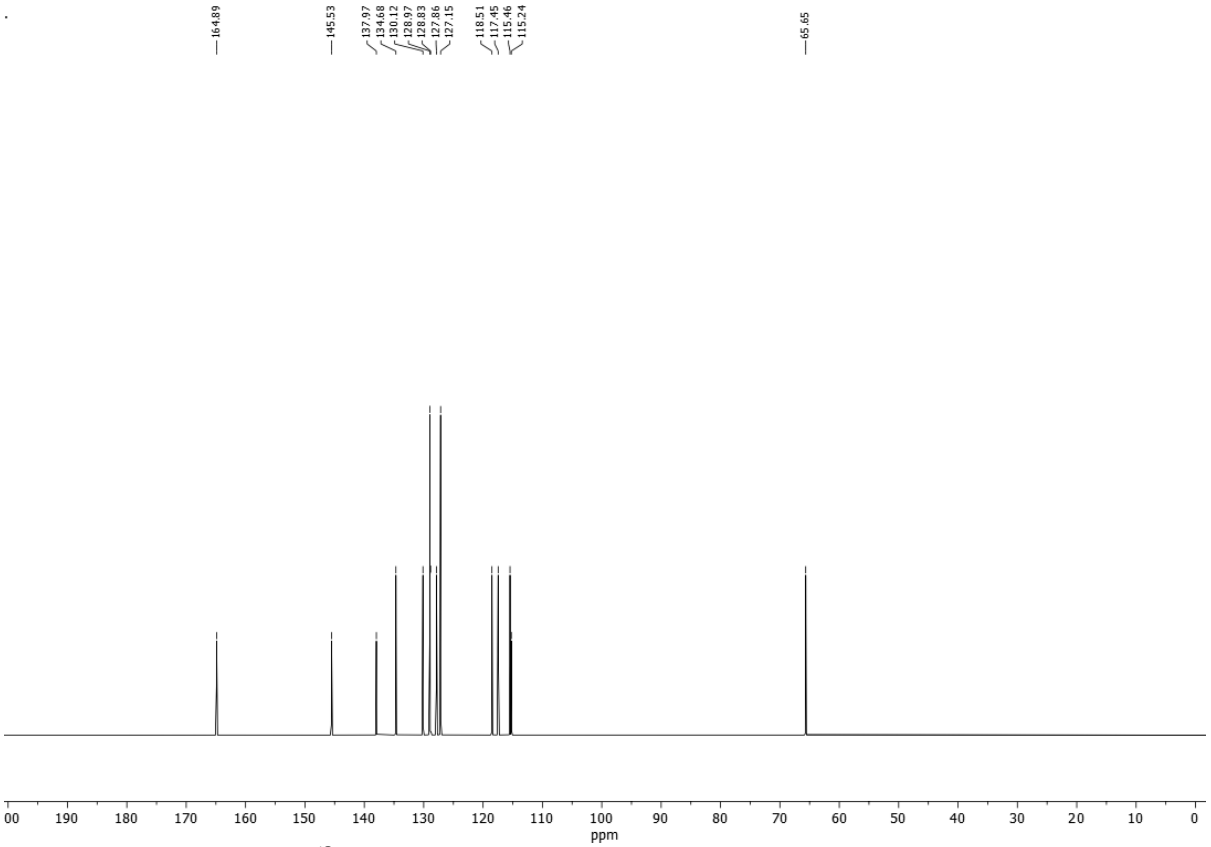

Figure S24. [13]C NMR spectra of (E)-2-styryl-2,3-dihydroquinazolin-4(1H)-one.

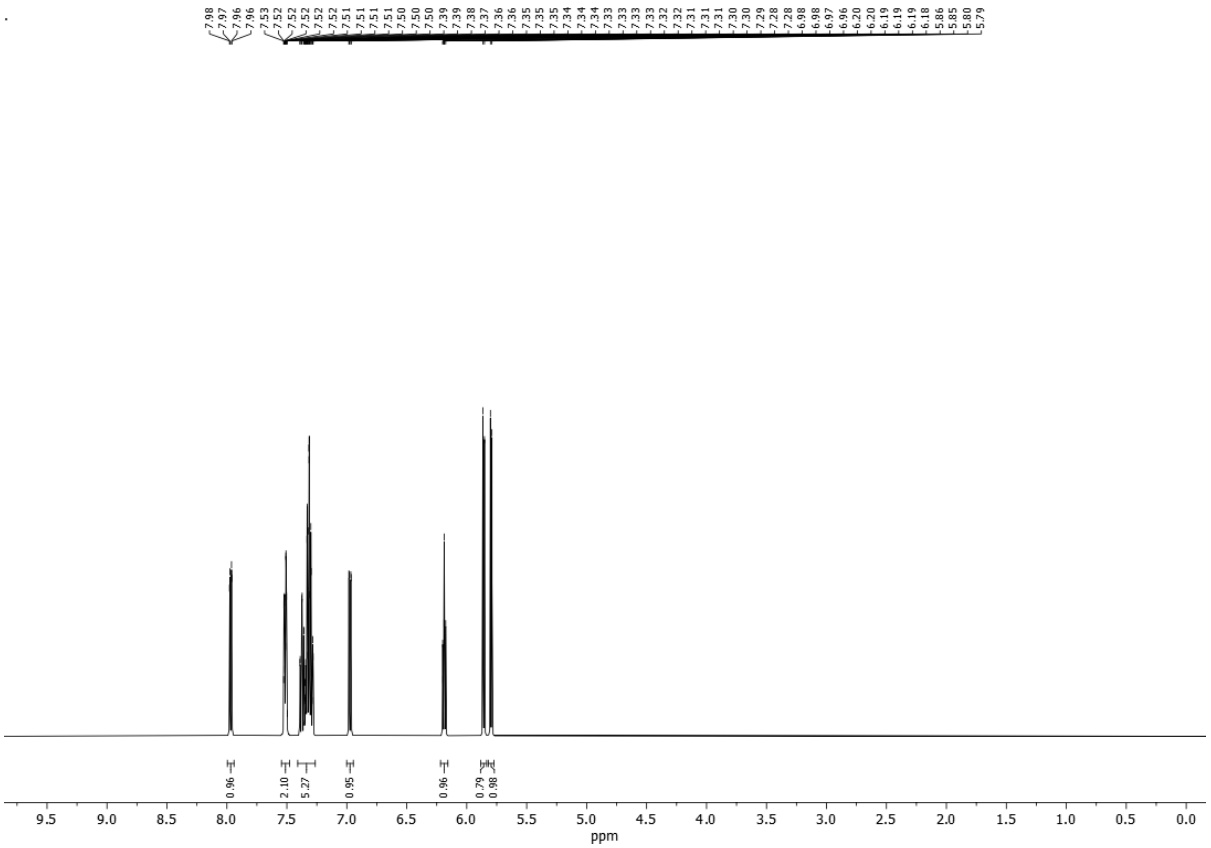

Figure S25. [1]H NMR spectra of 2-phenyl-2,3-dihydroquinazolin-4(1H)-one.

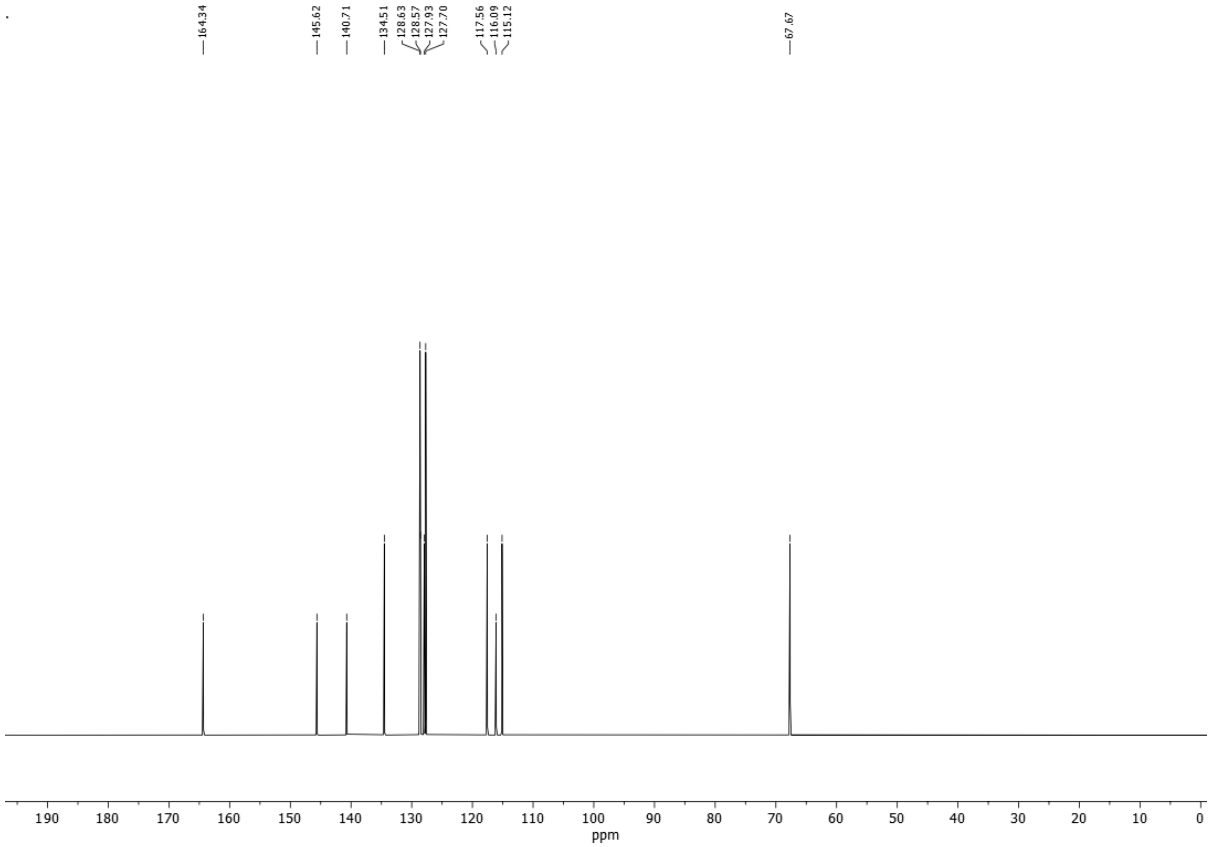

Figure S26. [13]C NMR spectra of 2-phenyl-2,3-dihydroquinazolin-4(1H)-one.