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# An efficient zinc acetate dihydrate-catalyzed green protocol for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

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**Abstract:** By condensation from substituted carbonyl compounds and anthranilamide under toluene reflux conditions, a wide range of 2,3-dihydroquinazolin-4(1*H*)-ones were produced in fair to good yields with the use of a Lewis acid catalyst $Zn(OAc)_2 \cdot 2H_2O$ (10 mol%), which is inexpensive, accessible, and environmentally friendly. All the synthesized compounds were properly described using melting point, IR, NMR, and mass spectral studies, and the findings were compared with information from the earlier literature. The new method has a number of advantages over the traditional methods for the synthesis of divergent 2,3-dihydroquinazolin-4(1*H*)-ones, including a higher product conversion, a wide substrate range, and the absence of undesirable side products. Aliphatic, heteroaromatic and aromatic carbonyl compounds were well tolerated under the optimized reaction conditions.

**Keywords:** Quinazoline; 2,3-dihydroquinazolin-4(1H)-ones; catalysis; zinc acetate dihydrate (Zn(OAc)2.2H2O) and cyclization. 

## 1. Introduction

Due to its astoundingly broad spectrum of pharmacological properties, the quinazoline scaffold (QZ) has taken a distinctive position in heterocycles containing nitrogen[1-7]. One of the key quinazoline analogues, 2,3-Dihydroquinazolin-4(1*H*)-ones (DHQZ) (Figure 1), particularly the 2-aryl substituted derivatives, have been found to have a wide range of biological activities, including anticancer, antifungal, anti-fertility, diuretic, antifibrillatory, and choleretic activities (Figure 2)[8-10]. Some isolated alkaloids from traditional Chinese medicine share the same scaffold. Quinazolin-4(3*H*)-ones are another class of compounds having different pharmacological actions from those of their dihydro counterparts, and they are easily converted from 2,3-dihydroquinazolin-4(1*H*)-ones[11-12].

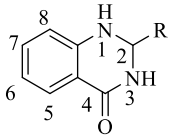


**Figure 1**. Structure of 2,3-dihydroquinazolin-4(1*H*)-ones (DHQZ)

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Zinc acetate dihydrate-catalyzed green protocol

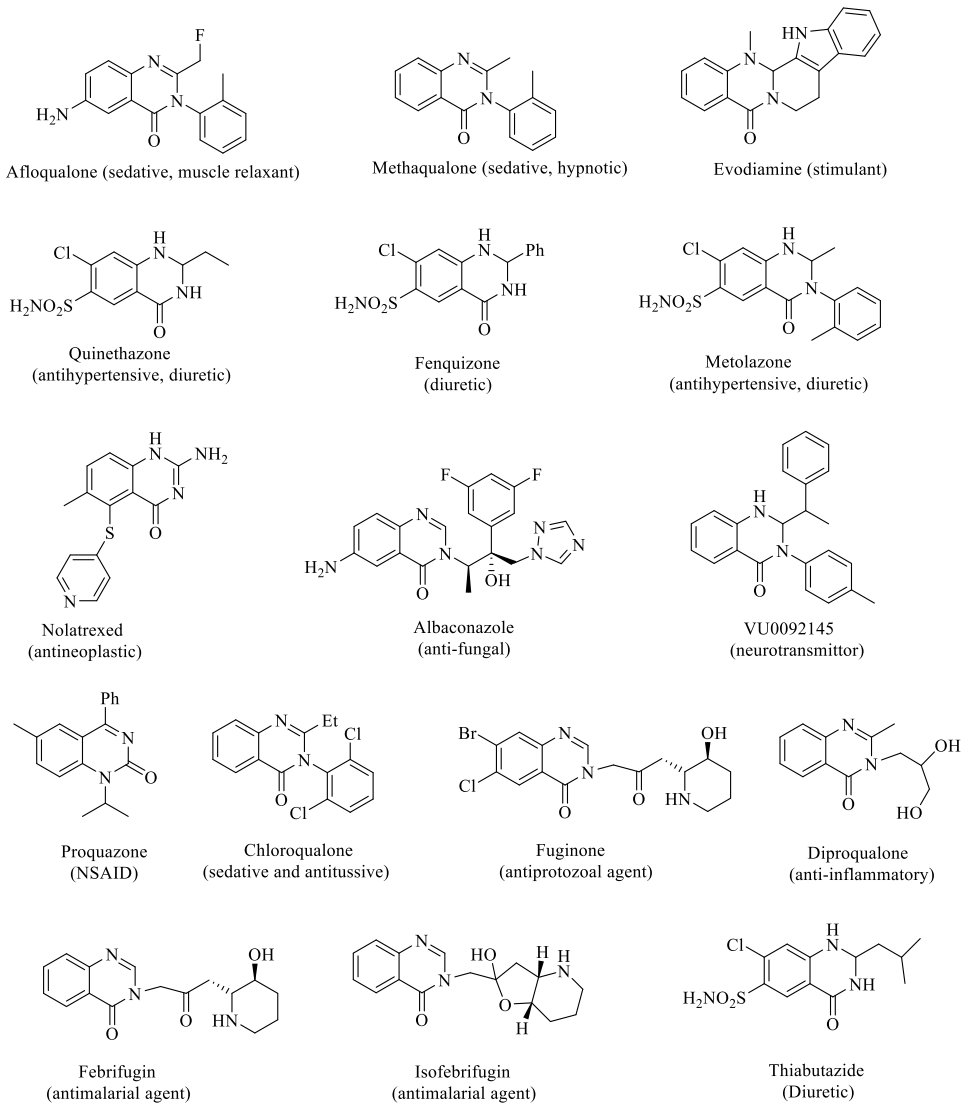


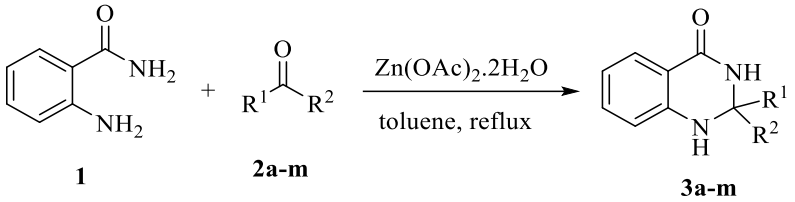

**Figure 2.** The privileged scaffold **'**DHQZ' in marketed drugs.

Due to their unique biodynamic and pharmacological characteristics, 2,3-dihydroquinazolin-4(1*H*)-ones have recently become particularly sought-after candidates in synthetic chemistry. There have been numerous procedures reported for making quinazolines, but three common methods stand out: (1) One-pot reactions using isatoic anhydride, ammonium, and aldehyde as reactants; (2) Reductive cyclization of *o*-nitrobenzamide using metallic reagents, a procedure that has never been particularly eco-friendly; (3) A condensation followed by a cyclodehydration between anthranilamide and aldehydes. However, the majority of the described protocols have laborious steps, poor yields, or demanding circumstances. As a result, organic chemists have faced a significant challenges in developing effective methods for the synthesis of target heterocyclic molecules for more than a century. This latter method was described by a number of researchers who used a variety of catalysts, including *p*-TSA, cellulose-$SO_3H$, $TiCl_4$, $CuCl_2$, $NH_4Cl$, ionic liquids, amberlyst-15, TFA, chiral phosphonic acids, alum, silica sulfuric acid, $H_3BO_3$, $Sc(OTf)_3$, $SrCl_2.6H_2O$, $ZrCl_4$, $Al(H_2PO_4)_3$, ZnO nanoparticles, etc[13-26].

Longer reaction times, stringent conditions, homogeneous catalyst nature, which makes the process very expensive, usage of expensive, water-sensitive catalysts, and particular efforts needed to manufacture the catalyst are all limitations of the current protocols. Most of the developed protocols worked well with aromatic aldehdyes. Ketones and heterocyclic aldehydes, on the other hand, require longer reaction durations and yield lesser amounts. The main drawback of this tactic is its inability to react with aromatic ketones.

## 2. Background

The Zn-based compounds have previously been employed in a number of catalytic processes[27]. Among the several zinc complexes, zinc acetate ($Zn(OAc)_2$) is easily accessible and stable in the presence of moisture and air at room temperature. After a thorough study of the literature, we discovered that $Zn(OAc)_2$•$2H_2O$ has not been used for the intended reaction. We are disclosing here our findings for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones (**3a-m**) with reference to other relevant recent literature reports[28-34] and our earlier successful result with $Zn(OAc)_2.2H_2O$ (Scheme 1)[28].




**Scheme 1.** $Zn(OAc)_2$•$2H_2O$-catalyzed synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

## 3. Experimental

### *2.1. Material and Methods*

All of the compounds were obtained from commercial sources and utilized without undergoing any further purification processes. Before being used, the solvents for chromatography go through the distillation process. In DMSO-d6, $^1H$ and $^{13}C$ nuclear magnetic resonance (NMR) spectra were recorded using Bruker UXNMR FT-400 MHz (Avance) devices. The tetramethylsilane (δ 0.0) internal standard serves as the reference point against which chemical shifts are compared and represented as parts per million. At a temperature of 200 $^0$C and an energy of 70 eV, EI-MS were obtained using a VG 7070H Micromass mass spectrometer. For the purpose of recording melting points, an electrothermal melting point equipment has been utilized. The IR spectra were obtained by employing KBr pellets and a Perkin Elmer 240-C instrument in the collection process. The analytical TLC for all reactions was performed on plates that had been pre-coated by Merck (silica gel 60F-254 on glass). In order to perform column chromatography, acme silica gel was utilized (100-200 mesh)

### *2.2. General Procedure for Preparation of 2,3-dihydroquinazolin-4(1H)-ones (Table 3, enries* **3a-m***)*

Anthranilamide (1 mmol) and substituted aromatic aldehydes (1 mmol) were added to a solution of zinc acetate (10 mol%) in toluene. The resulting mixture was stirred for the specified period of time while in reflux conditions (Table 3). Through thin layer chromatography (eluent: *n*-hexane/ethyl acetate: 2:1), the reaction was observed. After the reaction was finished, the precipitate was filtered and the corresponding pure product was recrystallized from the ethanol.

### *2.3. Spectral Data for the Selected Compounds*

*2,2-Dimethyl-2,3-dihydroquinazolin-4(1H)-one* (***3i***): White solid, $^1H$ NMR (400 MHz, DMSO-*d6*) $\delta$ = 7.95 (s, 1H), 7.59 (dd, *J*= 8.0 Hz, $J_2$ = 1.6 Hz, 1H),7.23 (dt, $J_1$ = 7.6 Hz, $J_2$ = 1.6 Hz, 1H), 6.67-6.61(m, 3H), 1.39 (s, 6H). $^{13}C$ NMR (100 MHz, DMSO-*d6*) $\delta$ = 163.5, 147.5, 133.7, 127.6, 116.9, 114.7, 114.3, 67.3, 29.5.

*2-Methyl-2-phenyl-2,3-dihydroquinazolin-4(1H)-one (**3k**)*: White solid, $^1H$ NMR (400 MHz, DMSO-*d6*) $\delta$ = 8.77 (d, *J* = 1.6 Hz, 1H), 7.64 (d, *J* = 1.6 Hz, 1H), 7.50-7.46 (m, 3H), 7.26 (dt, $J_1$ = 7.6 Hz, $J_2$ = 2.0 Hz, 2H), 7.23-7.17 (m, 2H), 6.76 (dd, $J_1$ = 8.0 Hz, $J_2$ = 0.8 Hz, 1H), 6.56 (dt, $J_1$ = 7.6 Hz, $J_2$ = 1.2 Hz, 1H), 1.63 (s, 3H). $^{13}C$ NMR (100 MHz, DMSO-*d6*) $\delta$ = 164.3, 148.2, 147.7, 133.8, 128.4, 127.7, 127.5, 125.6, 117.3, 115.5, 114.8, 70.6, 31.2. Please see the spectra in supporting information file of the paper.

## 4. Present Study

As a control reaction, we synthesized the corresponding 2,3-dihydroquinazolinone-4(1*H*)-one with a 64% yield by reacting anthranilamide (**1**, 1 mmol), benzaldehyde (**2a**, 1.2 mmol), and $Zn(OAc)_2•2H_2O$ (5 mol%) in toluene under reflux conditions. The reaction was carried out in several solvents and in solvent-free conditions to optimize the reaction conditions (Table 1, entries 1-6). It was noted that toluene had a good output (Table 1, entry 1). The formation of the product (**3a**) was confirmed by the disappearance of aldehyde proton (aliquots taken in regular intervals, IR monitoring) and formation of methylene proton (NH-C**H**-NH) at δ 5.88 ($^1$H NMR) and δ 160.08 (-**C**ONH) in $^{13}$C NMR.

**Table 1. Solvent studies**

| Entry | Solvent | Yield[a] (%) |
|---|---|---|
| 1 | **Toluene** | **64** |
| 2 | Acetone | 42 |
| 3 | Ethanol | 58 |
| 4 | Acetonitrile | 31 |
| 5 | Chloroform | 60 |
| 6 | Neat | 45 |

[a]Isolated yields

The superior catalytic activity of $Zn(OAc)_2•2H_2O$ tested in terms of isolated yields and reaction time for the intended reaction. Optimization has not been investigated or performed for the effective surface area studies based on BET measurement. By carrying out the reaction at various concentrations, the impact of $Zn(OAc)_2•2H_2O$ concentration (mol%) was evaluated (Table 2, entries 1-5). It was shown that 10 mol% was adequate for superior outcomes (88% isolated yield, Table 2, entry 3).

**Table 2. Study of catalyst loading**

| Entry | Mol (%) | Yield (%)[a] |
|---|---|---|
| 1 | 5 | 64 |
| 2 | 7.5 | 80 |
| 3 | **10** | **88** |
| 4 | 15 | 88 |
| 5 | 20 | 85 |

[a]Isolated yields

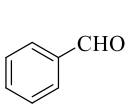
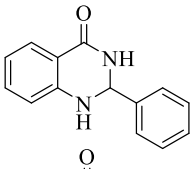
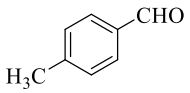
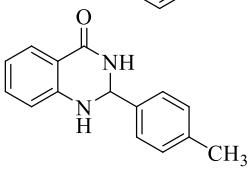
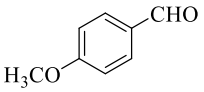
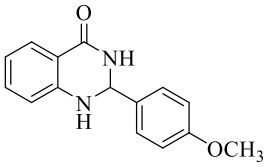
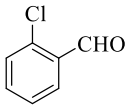
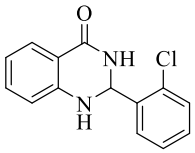
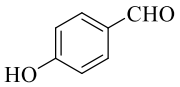
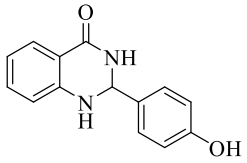
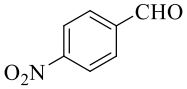
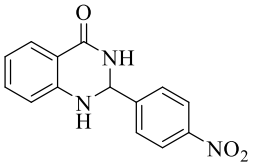
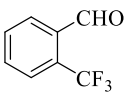
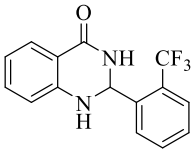
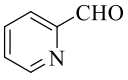
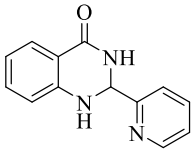
The improved system was used for the synthesis of further derivatives to demonstrate the generality of the current methodology. Table 3 provides a summary of many examples illustrative of this unique and versatile process for the synthesis of 2,3-dihydro-4(1*H*)-quinazolinones. With yields ranging from 62 to 91%, various aldehydes with various functionalities, including halogen, methoxy, hydroxyl, and nitro groups, produced the corresponding substituted 2,3-dihydro-4(1*H*)-quinazolinones (Table 3). It is significant to note that the absence of $Zn(OAc)_2•2H_2O$ prevented the synthesis of 2,3-dihydro-4(1*H*)-quinazolinones. The structures of all the products were confirmed by comparison with their known physical (melting point) and spectral (IR, NMR, and mass) data reported in literature. All the synthesized products are known compounds.

Aldehydes bearing either electron-donating or electron-withdrawing groups were used to yield the corresponding products, and the reaction proceeded without any problems. Aldehydes containing electron-donating groups (-Me, -OMe and OH) (Table 3, entries **3b**, **3c**, and **3e**) and halogen-substituted (-Cl) aldehydes (Table 3, entry **3d**) with strong electron-withdrawing groups ($-NO_2$ and $-CF_3$) may boost the reaction and offered greater yields (Table 3, entries **3f-g**). Additionally, the corresponding products were produced in good yields by heterocyclic aldehyde such as pyridine-2-carbaldehyde (Table 3, entry **3h**).

We have then focused on aliphatic carbonyl compounds after being inspired by the outcomes of aromatic aldehydes. Table 3, entries **3i-j**, showed that acetone and 2-butanone produced corresponding

compounds in good quantities. Using this procedure, acetophenone (Table 3, entry **3k**) produced a moderate yield. Cyclic ketones like cyclopentanone and cyclohexanone responded favourably and generated yields of 78 and 82%, respectively (Table 3, entries **3l-m**). Compared to aliphatic or alicyclic ketones, it was observed that aromatic ketones were less reactive.

**Table 3.** Zinc acetate dihydrate-catalyzed synthesis of DHQZs from carbonyl compounds

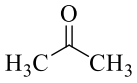
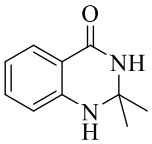
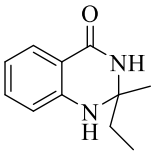
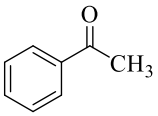
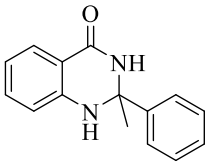
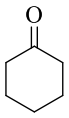
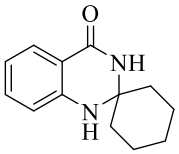
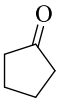
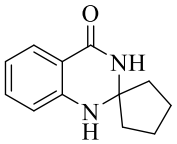
| Entry | Carbonyl compound | Product | Time (h) | Yield (%) | Melting point ($^0$C)$^{ref}$ |
|---|---|---|---|---|---|
| **3a** |  |  | 2.5 | 88 | 219-220[27] |
| **3b** |  |  | 2 | 90 | 221-223[27] |
| **3c** |  |  | 2 | 91 | 189-191[27] |
| **3d** |  |  | 3.5 | 78 | 210-212[25] |
| **3e** |  |  | 2.5 | 80 | 182-183[27] |
| **3f** |  |  | 3.5 | 75 | 159-161[25] |
| **3g** |  |  | 2 | 72 | 188-190[25] |
| **3h** |  |  | 3 | 78 | 172-174[25] |

[a]Isolated yields after purification

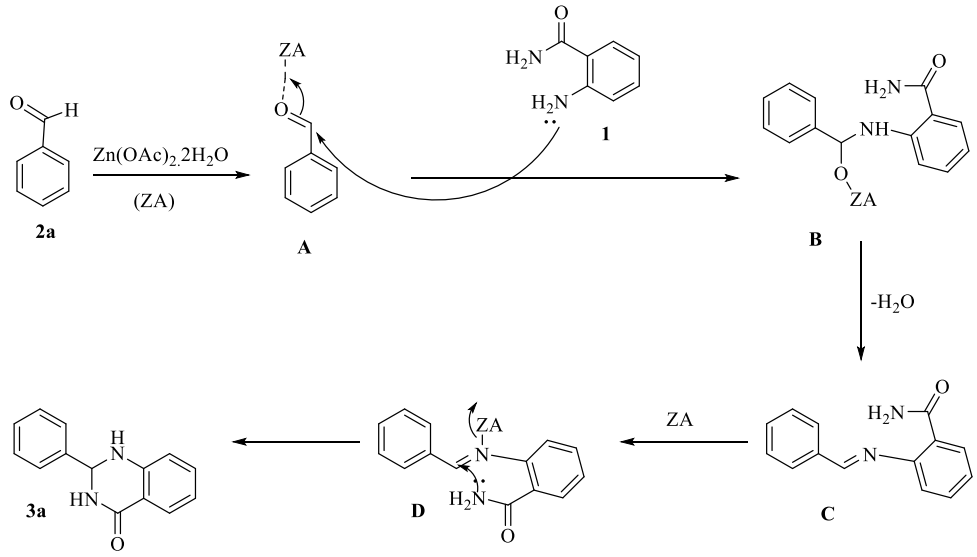
(Continued)

*Table 3 continued..*

Zinc acetate dihydrate-catalyzed green protocol

| Entry | Carbonyl compound | Product | Time (h) | Yield (%)[a] | Melting point ($^0$C)[ref] |
|---|---|---|---|---|---|
| **3i** |  |  | 2.5 | 88 | 182-183[27] |
| **3j** |  |  | 2 | 85 | 180-181[23] |
| **3k** |  |  | 4 | 62 | 226-228[27] |
| **3l** |  |  | 2.5 | 78 | 216-218[27] |
| **3m** |  |  | 2.5 | 82 | 257-259[21] |

[a]Isolated yields after purification




**Scheme 2.** Plausible mechanism for the $Zn(OAc)_2 \bullet 2H_2O$-catalyzed synthesis of DHQZs

In Scheme 2, the plausible mechanism for the synthesis of 2,3-dihydroquinazolin 4(1*H*)-ones using zinc acetate is depicted. In this instance, zinc acetate (ZA) generates a zinc-aldehyde intermediate (**A**) after coordinating with benzaldehyde (**2a**). After the dehydration and removal of zinc acetate, the condensation of the zinc acetate-aldehyde intermediate (**A**) with the anthranilamide (**1**) results in the imine intermediate (**C**). ZA again coordinates to intermediate (**C**), and the subsequent condensation of the imine with the amino group of anthranilamide created the desired product (**3a**).

## 5. Conclusion

The one-pot cyclocondensation of anthranilide and carbonyl compounds in refluxing toluene was accomplished in this work using $Zn(OAc)_2$•$2H_2O$ as the green, easily accessible, and cost-effective catalyst for the first time. The new approach has a number of advantages over the traditional method for synthesizing divergent 2,3-dihydroquinazolin-4(1*H*)-ones, including increased product conversion, wide range of substrate scope, and the absence of undesirable side products. We strongly hope that the present methodology will be a valuable addition to the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones.

## Supporting Information

Supporting information accompanies this paper on http://www.acgpubs.org/journal/organic-communications

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