

# One-Pot Three-Component Synthesis of 2,3-Dihydroquinazolin-4(1H)-Ones in the Presence of a Molecular Sieve Supported Lanthanum Catalyst

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**Abstract** A series of 2,3-dihydroquinazolin-4(1H)-ones have been synthesized with good to excellent yields by one-pot reaction using isatoic anhydride, aldehydes, and ammonium acetate or amines in acetonitrile in the presence of 4 Å molecular sieve modified with lanthanum(III) as an efficient heterogeneous catalyst. The catalyst could be reused without evident loss of activity.

**Graphical Abstract** 

inflammatory and analgesic, antitumor, anticancer, antibacterial, and diuretic activities [1-5]. In addition, these compounds can be oxidised to their corresponding quinazolin-4(3H)-one analogues, which are also biologically important heterocycles and can be found in some natural products [6]. Due to their importance, several strategies have been developed for the synthesis of 2,3-dihydroquinazolinones. One possible route is the conden-



**Keywords** Heterogeneous catalysis · Quinazolinones · Lanthanum · Molecular sieve

## **1** Introduction

2,3-Dihydroquinazolinons represent a highly important group of heterocycles possessing a wide range of pharmacological and biological properties such as anti-

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sation of aryl, alkyl, and heteroaryl aldehydes with anthranilamide in the presence of p-toluenesulfonic acid as a catalyst [7, 8]. The synthesis of 2,3-dihydroquinazolinones was also achieved by reductive cyclisation of o-nitrobenzamide or o-azidobenzamide [9, 10]. Other methods including desulfurization of 2-thioxo-4(3H)-quinazolinones [11], reaction of isatoic anhydride with Schiff bases [12], condensation of anthranilamide with benzyl [8], and a two-step synthesis starting from isatoic anhydride and amines, followed by annulation with ketones [13] was also reported. A more attractive and atom-efficient strategy for the preparation of 2,3-dihydroquinazolinones is through a one-pot, three-component reaction of isatoic anhydride, amines, and aldehydes. Multicomponent reactions (MCRs) have some advantages over classical reaction strategies, such as lower costs, shorter reaction time, and less side

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products, as well as environmentally more friendly aspects. In 2005, Salehi and Dabiri reported first an MCR method for the synthesis of 2,3-dihydro-4(1H)-quinazolinones from isatoic anhydride, aldehydes, and amines [14, 15]. Numerous protocols have been developed recently for the synthesis using homogeneous catalysts such as strontium chloride [16], Zn(PFO)<sub>2</sub> [17], Ga(OTf)<sub>3</sub> [18], molecular iodine [19], copper-benzenesulfonate [20], aluminiummethanesulfonate [21]. The use of ionic liquids was also reported for this conversion [22-24]. Recently solid acid catalysts, such as cation exchange resin [25], starch sulfate [26], silica sulfuric acid [27], silica-bonded S-sulfonic acid [28], PTSA-formaldehyde copolymer [29], β-cyclodextrin-SO<sub>3</sub>H [30], magnetic Fe<sub>3</sub>O<sub>4</sub> nanoparticles [31, 32], Al/ Al<sub>2</sub>O<sub>3</sub> nanoparticles [33], CuO nanoparticles using sonication [34], nano indium-oxide [35], montmorillonite K-10 [36], and Amberlyst-15 microwave-assisted [37] have also been reported. However, some of these procedures have certain limitations such as harsh reaction conditions, expensive or large amount of catalyst, long reaction time, high reaction temperature, tedious work-up, and low yields. The catalysts described in the literature are mainly acidic what can cause problems in the case of acid sensitive compounds. Thus, the development of novel methods for the synthesis of dihydroquinazolin-4(1H)-ones might have great importance because of their potential biological and pharmaceutical activities.

Our research group works on the development of new heterogeneous catalytic methods for the preparation of organic compounds using supported metal catalysts. During this work several metals, such as palladium [38–41], nickel [42], copper [43–45] or titanium [46] on different supports (Mg:La 3:1 mixed oxide, 4 Å molecular sieve) were used successfully in different organic syntheses. In this paper we report a method for the one-pot three-component synthesis of 2,3-dihydroquinazolin-4(1H)-ones promoted by a heterogeneous, 4 Å molecular sieve supported lanthanum catalyst under mild basic conditions.

#### 2 Experimental

Morphology of the catalyst samples was investigated by a JEOL 6380LVa (JEOL, Tokyo, Japan) type scanning electron microscope and elemental mapping was also accomplished using the energy-dispersive X-ray detector of the equipment. Each specimen was fixed by conductive double-sided carbon adhesive tape and sputtered by gold (using a JEOL 1200 instrument). Applied accelerating voltage and working distance were between 15 and 30 kV and 10 and 12 mm, respectively. In the case of the comparison of the fresh and the used catalysts, the samples

were examined without sputtering with gold and weren't fixed on a carbon tape.

XRF investigation was made on a Canberra isotope excitation instrument with SCD, using  $^{125}I$  as exciting source.

GC–MS measurements were performed on an Agilent 6890 N-GC-5973 N-MSD chromatograph, using a 30 m  $\times$  0.25 mm Restek, Rtx-5SILMS column with a film layer of 0.25 µm. The initial temperature of column was 45 °C for 1 min, followed by programming at 10 °C/min up to 310 °C and a final period at 310 °C (isothermal) for 17 min. The temperature of the injector was 250 °C. The carrier gas was He and the operation mode was splitless. <sup>1</sup>H NMR spectra were made on BRUKER Avance-300 instrument using TMS as internal standard in DMSO-D<sub>6</sub> or CDCl<sub>3</sub>.

Melting points were determined on Gallenkamp apparatus and were uncorrected.

#### 2.1 Preparation of the Catalyst

 $4\text{\AA}$  molecular sieve (4A) was impregnated with La(NO<sub>3</sub>)<sub>3</sub> × 6H<sub>2</sub>O as follows: 1 mmol of the metal salt was dissolved in 100 mL of deionised water and stirred with 1 g 4A at room temperature for 24 h. The solid was filtered, washed with deionised water and with acetone, then dried in an oven at 150 °C for 1 h.

#### 2.2 Determination of the pH of the Catalyst

The catalyst (1 g) was stirred in 30 mL deionised water under continuous measuring of the pH. The values were accepted after reaching a constant value at least during 10 min.

#### 2.3 Typical Reaction Conditions

## 2.3.1 General Procedure for the One-Pot Synthesis of Quinazolin-4(3H)-Ones from Aldehydes

A typical reaction was carried out in a 10 mL flask. Isatoic anhydride (2 mmol), aldehyde (2 mmol), ammonium acetate (2.4 mmol),  $La^{3+}/4A$  (0.2 g) and acetonitrile (3 mL) were stirred at reflux temperature for 24 h. The progression of the reaction was monitored by TLC. After completion, the solid was filtered, and washed with acetone, then the filtrate was evaporated. The residue was suspended in diethyl ether, the precipitated solid was filtered and subjected to GC–MS analysis and/or <sup>1</sup>H NMR spectroscopy.

## 2.3.2 General Procedure for the One-Pot Synthesis of Quinazolin-4(3H)-Ones from Acetals

To a solution of isatoic anhydride (2 mmol), benzaldehyde dimethylacetal (2 mmol), ammonium acetate (2.4 mmol) or benzylamine (2 mmol) in 3 mL acetonitrile  $La^{3+}/4A$  (0.2 g) was added. The mixture was stirred for 24 h at reflux temperature. The progression of the reaction was monitored by TLC. After completion, the solid was filtered, and washed with acetone, then the filtrate was evaporated. The residue was suspended in diethyl ether, the precipitated solid was filtered and subjected to <sup>1</sup>H NMR spectroscopy.

### 2.3.3 General Procedure for the One-Pot Synthesis of Quinazolin-4(3H)-Ones from Ketones

A mixture of isatoic anhydride (2 mmol), ketone (2 mmol), ammonium acetate (2.4 mmol) or benzylamine (2 mmol) and  $La^{3+}/4A$  (0.2 g) in acetonitrile (3 mL) were stirred at reflux temperature for 24 h. The progression of the reaction was monitored by TLC. After completion, the solid was filtered, and washed with acetone, then the filtrate was evaporated. The residue was suspended in diethyl ether, the precipitated solid was filtered and subjected to GC–MS analysis and <sup>1</sup>H NMR spectroscopy.

All products have satisfactory spectral data (<sup>1</sup>H NMR, MS). The spectral data of the known compounds were identical with those reported in the literature.

## 2.3.4 Representative Physical and Spectroscopic Data of the Products

2.3.4.1 2-(4-Chlorophenyl)-2,3-Dihydro-4(1H)-Quinazolinone (4a) White solid. <sup>1</sup>H NMR, DMSO- $d_6$ :  $\delta = 8.32$  (s, 1 H), 7.60 (d, J = 6.9 Hz, 1 H), 7.49 (d, J = 8.4 Hz, 2 H), 7.47 (d, J = 8.4 Hz, 2 H), 7.25 (t, J = 8.4 Hz, 1 H), 7.13 (s, 1 H), 6.76 (d, J = 8.1 Hz, 1 H), 6.68 (t, J = 7.8 Hz, 1 H), 5.77 (s, 1 H).

2.3.4.2 2,3-Dihydro-2-(3-Methoxyphenyl)-4(1H)-Quinazolinone (4e) White solid. <sup>1</sup>H NMR, DMSO- $d_6$ :  $\delta = 8.29$  (s, 1 H), 7.60 (d, J = 4.5 Hz, 1 H), 7.29 (t, J = 5 Hz, 1 H), 7.25 (t, J = 5 Hz, 1 H), 7.13 (s, 1 H), 7.06 (m, 2H), 6.90 (dd, J<sub>1</sub> = 6 Hz, J<sub>2</sub> = 1 Hz, 1 H), 6.75 (d, J = 4.8 Hz, 1 H), 6.68 (t, J = 4.2 Hz, 1 H), 5.72 (s, 1 H), 3.74 (s, 3H).

2.3.4.3 2,3-Dihydro-2-(4-Methoxyphenyl)-4(1H)-Quinazolinone (4e) White solid. <sup>1</sup>H NMR, DMSO- $d_6$ :  $\delta = 8.17$  (s, 1 H), 7.61 (d, J = 6.9 Hz, 1 H), 7.42 (d, J = 8.7 Hz, 2 H), 7.25 (t, J = 8.4 Hz, 1 H), 7.00 (s, 1 H), 6.93 (d, J = 8.7 Hz, 2 H), 6.73 (d, J = 8.1 Hz, 1 H), 6.67 (t, J = 7.5 Hz, 1 H), 5.70 (s, 1 H), 3.74 (s, 3H). 2.3.4.4 2,3-Dihydro-2-(2-Fluorophenyl)-4(1H)-Quinazolinone (4f) White solid. <sup>1</sup>H NMR, DMSO- $d_6$ :  $\delta = 8.24$  (s, 1 H), 7.64 (d, J = 9.9 Hz, 1 H), 7.54 (t, J = 7.2 Hz, 1 H), 7.42 (q, J = 8.1 Hz, 1 H), 7.23 (q, J = 7.2 Hz, 3 H), 7.04 (s, 1 H), 6.67-6.76 (m, 2 H), 6.06 (s, 1 H).

2.3.4.5 2-(3-Bromophenyl)-2,3-Dihydro-4(1H)-Quinazolinone (**4g**) White solid. <sup>1</sup>H NMR, DMSO- $d_6$ :  $\delta = 8.38$  (s, 1 H), 7.67 (s, 1 H), 7.61 (d, J = 6.9 Hz, 1 H), 7.53 (d, J = 8.1 Hz, 1H), 7.50 (d, J = 15.1 Hz, 1 H), 7.35 (t, J = 7.8 Hz, 1 H), 7.20–7.28 (m, 2 H), 6.76 (d, J = 8.4 Hz, 1 H), 6.68 (t, J = 7.5 Hz, 1 H), 5.77 (s, 1 H).

2.3.4.6 2,3-Dihydro-2-(3-Nitrophenyl)-4(1H)-Quinazolinone (4j) Pale yellow solid. <sup>1</sup>H NMR, DMSO- $d_6$ :  $\delta = 8.54$  (s, 1 H), 8.37 (s, 1 H), 8.21 (d, J = 4.8 Hz, 1 H), 7.95 (d, J = 4.8 Hz, 1 H), 7.71 (t, J = 4.8 Hz, 1 H), 7.63 (d, J = 4.8 Hz, 1 H), 7.35 (s, 1 H), 7.27 (d, J = 4.8 Hz, 1 H), 6.79 (d, J = 4.8 Hz, 1 H), 6.70 (t, J = 4.8 Hz, 1 H), 5.95 (s, 1 H).

2.3.4.7 2,3-Dihydro-2-[4-(Dimethylamino)phenyl)]-4(1H)-Quinazolinone (4l) White solid. <sup>1</sup>H NMR, DMSO- $d_6$ :  $\delta = 8.05$  (s, 1 H), 7.60 (d, J = 6.9 Hz, 1 H), 7.30 (d, J = 8.7 Hz, 2 H), 7.22 (t, J = 6.9 Hz, 1H), 6.90 (s, 1 H), 6.63 - 6.74 (m, 4 H), 5.63 (s, 1 H), 2.87 (s, 6 H).

2.3.4.8 2,3-Dihydro-2-Phenyl-3-(Phenylmethyl)-4(1H)-Quinazolinone (6) White solid. <sup>1</sup>H NMR, DMSO- $d_6$ :  $\delta = 7.72$  (d, J = 4.8 Hz, 1 H), 7.22–7.40 (m, 12 H), 6.65–6.69 (m, 2 H), 5.75 (s, 1 H), 5.34 (d, J = 12.6 Hz, 1 H), 3.83 (d, J = 9.3 Hz, 1 H).

2.3.4.9 2,3-Dihydro-2-Ethyl-2-Methyl-4(1H)-Quinazolinone (8a) <sup>1</sup>H NMR, DMSO- $d_{6_1}$  selected data:  $\delta = 7.83$  (dd, J<sub>1</sub> = 1.2 Hz, J<sub>2</sub> = 7.8 Hz, 1 H), 7.35 (dd, J<sub>1</sub> = 1.2 Hz, J<sub>2</sub> = 7.8 Hz, 1 H), 1.80 (q, J = 7.5 Hz, 2 H), 1.48 (s, 3 H), 0.98 (t, J = 7.2 Hz, 3 H).

#### **3** Results and Discussion

The structure of the  $La^{3+}/4A$  catalyst was investigated by scanning electron microscopy. The characteristic cuboctahedron shape of the molecular sieve support can be seen on Figs. 1, 2. The particles are well defined both in shape and size. The lanthanum is evenly distributed on the surface of the support. EDS showed 3.65 w/w% lanthanum on the surface. The lanthanum content determined by ICP-OES was 3.88 w/w%. The catalyst has slightly basic properties, its pH value is 8.40 (see Experimental).

From the nitrogen adsorption/desorption measurements the specific surface of the catalyst is  $35 \text{ m}^2/\text{g}$ .



Fig. 1 SEM image of the catalyst

The catalyst was tested in the multicomponent reaction of isatoic anhydride, aldehydes and ammonium acetate yielding 2,3-dihydroquinazolinones. This reaction has been described as an acid-catalysed cyclisation. In the literature there are examples for the use of a lanthanum-compound as the catalyst for this cyclisation such as lanthanum-triflate [18], lanthanum-benzenesulfonate [20] and lanthanummethanesulfonate [21], these catalysts are both acidic in nature. To study the effect of the solvent on the condensation reaction, isatoic anhydride, 4-chlorobenzaldehyde and ammonium acetate were used in a 1:1:1,2 molar ratio and were reacted in the presence of 0.2 g  $La^{3+}/4A$  catalyst in 3 mL of different solvents. The results are shown in Table 1.

Acetonitrile gave the best yield in the reaction (entry 5). The product has a weak solubility in ethanol, thus we tried to increase its amount in the reaction mixture. But in this case the yield of **4a** decreased significantly, and a new product appeared. GC–MS investigation of the reaction mixture showed that this was 4-chlorobenzaldehyde diethylacetal. Toluene and xylene showed weaker results. Thus, in the further experiments we used acetonitrile in a 24 h reaction.

Next, we studied the reaction of different aldehydes. The results are summarized in Table 2.

The reactions gave generally good to excellent isolated yields. In the case of 2-nitro- and 4-nitro-benzaldehyde (entries 9 and 11) the lower yields may be explained by the weak solubility of the products, thus they remained at the surface of the catalyst during the workup. The use of acetonitrile in regard of 4-dimethylamino-benzaldehyde didn't prove to be efficient, higher yield could be obtained using ethanol as solvent (entry 12). In the reaction of salicylaldehyde the lower yield obtained in ethanol might be



**Fig. 2** SEM image of a catalyst (*upper left*), distribution of the lanthanum on the particle (*lower left*) and the standard quantitative analysis of the catalyst (*right*) (magnification: ×5000)



Table 1 Reaction of isatoic anhydride, 4-chloro-benzaldehyde and ammonium acetate in different solvents

2 mmol isatoic anhydride, 2 mmol benzaldehyde, 2.4 mmol ammonium acetate, 0.2 g catalyst, 3 mL solvent, reflux temperature, 14 h

<sup>a</sup> Isolated yield

<sup>b</sup> 24 h reaction time

explained by the formation of a H-bond between aldehyde function and the hydroxyl group (entry 13).

Although the reaction has been described generally with acidic catalysts, in our case the slightly basic heterogeneous catalyst gave good results. As the EDS measurement showed, the lanthanum is located on the surface of the support, forming potentially acidic sites. The reaction may happen on these parts of the catalysts. Meanwhile the bulk basic phase may help to avoid the potential disadvantageous, acid-catalysed side reactions or a possible decomposition if an acid-sensitive compound or functional group is present.

According to the reported mechanisms both for homogeneous [16] and heterogeneous methods [31, 32], we propose a plausible mechanism for the cyclisation. Thus first isatoic anhydride is activated by the cation (here  $La^{3+}$ ). This is followed by *N*-nucleophilic attack of ammonium acetate (or the respective amine) on the carbonyl group. After a loss of carbon dioxide the yielded 2-amino-benzamide reacts with the activated aldehyde A affording the intermediate Β. the intramolecular cyclisation of this intermediate results intermediate C, finally 1,5-proton transfer gives the product 4 (Fig. 3). The intermediate 2-amino-benzamide could be shown in the GC-MS spectra of the reaction mixture of isatoic anhydride, ammonium acetate and benzaldehyde.

We examined the reaction with benzaldehyde dimethylacetal instead of benzaldehyde. The corresponding **4b** was formed with excellent yields (Fig. 4). Replacing ammonium acetate by benzylamine, the disubstituted derivative 6 was formed also with excellent yield. We investigated the reaction of different ketones instead of aldehydes, the results are shown in Table 3. In the case of ethyl methyl ketone (entry 1), based on the NMR spectra, regardless the reaction time the yield was approximately 50 %. Using ethyl methyl ketone in excess, the yield of the desired products decreased, probably due to aldol-type side reactions. Benzylamine instead of ammonium acetate gave low yield (entry 2). Acetophenone gave moderate yield even if the reaction time was 48 h (entry 3).

The reusability of the catalysts was examined in the reaction of isatoic anhydride, benzaldehyde and ammonium acetate. After the reaction the solid was filtered and washed with hot acetone, then heated at ca. 150 °C for 1 h. The yields in the 1st and 2nd runs were 85 and 76 %, respectively. We examined the crude product by XRF to determine whether the leached metal resulted the lower yield, but no lanthanum could be detected. We investigated the used catalyst by scanning electron microscopy and EDS to determine the background of the decreased activity of the catalyst. The SEM and EDS records showed rectangle shaped crystals with high carbon content (Fig. 5), subsequently the reason of the decreasing activity might be the weak solubility of the products. Sampling the catalyst particles the EDS results confirmed that there was no difference in the metal content of the fresh and the used catalyst. Thus we changed the recycling process of the catalyst. The used catalyst was stirred in boiling acetone for 5 h, filtered, then heated at ca, 150 °C for 1 h. The lanthanum content of this sample determined by ICP-OES was 3.79 w/w%. The thus treated catalyst was tested in the model reaction. There was no significant difference in the yields.



Table 2 The reaction of isatoic anhydride, different aldehydes and ammonium acetate

Table 2 continued

Entry	R	Product	Yield (%) <sup>a</sup>	Mp (°C)
7	3-Br	NH NH H	90	>220 (229–230 [33])
8	4-Br	4g	86	199–201 (205.7–206.3 [22])
9	2-NO <sub>2</sub>	4h	60	187–188 (193–194 [18])
10	3-NO <sub>2</sub>		90	213–215 (216–217 [18])
11	4-NO <sub>2</sub>	4j	47	(213–214 [18]) <sup>c</sup>
12	4-NMe <sub>2</sub>	4k	47, 80 <sup>b</sup>	202–204 (207–209 [25])
13	2-OH	41 • • • • • • • • • •	45 <sup>b</sup>	(218–219 [48]) <sup>c</sup>
		4m		

2 mmol isatoic anhydride, 2 mmol benzaldehyde, 2.4 mmol ammonium acetate, 0.2 g catalyst, 3 mL acetonitrile, reflux temperature, 24 h

<sup>b</sup> In ethanol

 $^{\rm c}\,$  Decomposed above 200  $^{\circ}{\rm C}$ 

<sup>&</sup>lt;sup>a</sup> Isolated yield



Fig. 3 Proposed mechanism of the reaction



Fig. 4 The condensation reaction with benzaldehyde dimethylacetal



Table 3 The reaction of isatoic anhydride, ammonium acetate/benzylamine and different ketones

<sup>a</sup> Conversion determined from the <sup>1</sup>H NMR spectra

<sup>b</sup> Based on the GC-MS examination of the product



Fig. 5 SEM image of the used catalyst

## 4 Conclusions

In conclusion, lanthanum on 4 Å molecular sieve support proved to be useful catalyst for the one-pot, three component synthesis of 2,3-dihydroquinazolin-4(1H)-ones under mild, slightly basic conditions, which is unprecedented in the literature. The catalyst could be prepared simply and can be reused with good result after a treatment with boiling acetone.

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