ICC
Iranian Chemical Communication
Original Research Article
Payame Noor University http://icc.journals.pnu.ac.ir

Multicomponent reaction for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones using isatoic anhydride, aldehydes and NH_4OAc catalyzed by $SnCl_2.2H_2O$ under solvent-free conditions

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Received: 24 November 2018, Accepted: 19 January 2019, Published: 1 July 2019

Abstract

Nowadays, Quinazolinone derivatives are well-recognized as valuable scaffolds in the drug discovery. In this manuscript an improved multicomponent process for the chemical synthesis of 2,3-dihydroquinazolin-4(1*H*)-one derivatives is described. Isatoic anhydride and aromatic aldehydes with ammonium acetate have been subjected to a three-component reaction under solvent-free conditions and catalysis of $SnCl_2$ dihydrate at 110 °C. All of the products are known and characterized using melting point, ^{1}HNMR and infrared spectra (FT-IR), and also can be compared to the trusty references. The present methodology offers several advantages, such as cost efficiency, easy experimental workup procedure, mild reaction conditions, short reaction time, good to high yields and synthesis of wide range of products.

Keywords: Isatoic anhydride; 2,3-dihydroquinazolin-4(1*H*)-one; $SnCl_2.2H_2O$; solvent-free conditions.

Introduction

Multicomponent reactions (MCRs) combine three or more starting materials in a single chemical event to form a product containing most of the starting materials atoms. Efficiency, atom economy, straightforward reaction design, mild conditions, high convergence and concomitant step economy of modern MCRs in combination with their general compatibility with green solvents would justify a central place in the toolbox of sustainable synthetic methodologies [1–5].

Due to importance of heterocyclic compounds, several methods have been reported for the synthesis of these compounds *via* multicomponent condensation reactions [5–10].

Quinazolinone derivatives are among the privileged class of heterocyclic scaffolds known to exhibit a broad range of biological and pharmacological activities, such as anti-inflammatory and analgesic activity, as well as antimicrobial activity [11–15]. Likewise, quinazolinones are an important subunit of many natural and synthetic compounds that exhibit biological activity. Some quinazolinone compounds constitute the basic skeleton of a number of alkaloids such as rutaecarpine and luotonins (Figure 1), [11–15].

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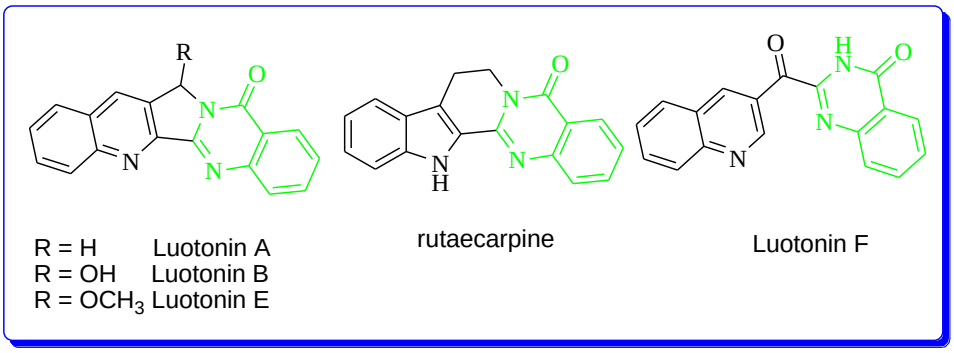


Figure 1. Quinazolinone based alkaloids

In the community of the fused heterocyclic compounds, 2, 3-Dihydroquinazolinone DHQZ is omnipresent and has been referred to as "core structure" in drug discovery. Further, DHQZs are also known to display a broad range of pharmacological properties such as antimicrobial, antiviral, antitumor, and analgesic activities [16–20]. Some structure of the important commercial drugs with quinazolinones core is shown in Figure 2 [16–20].

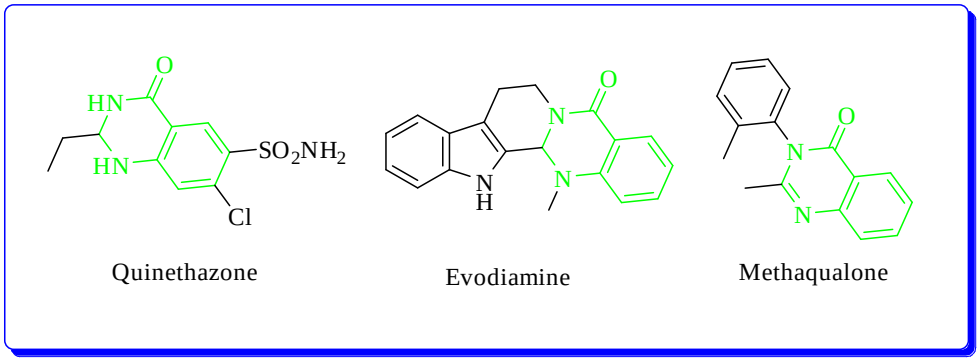
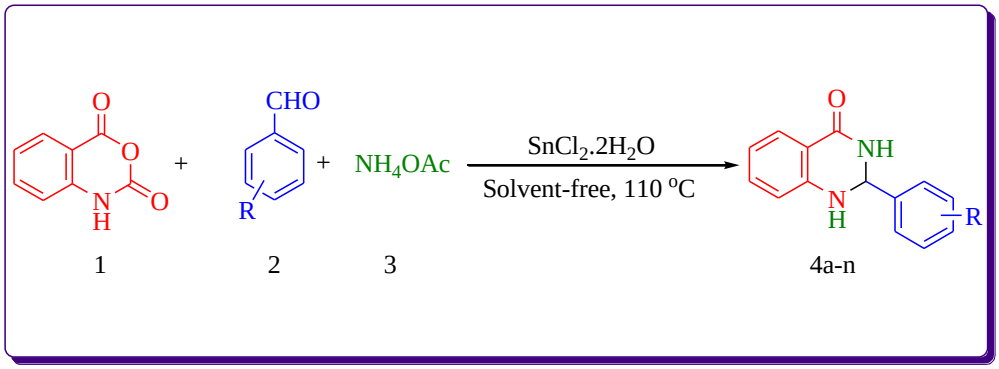


Figure 2. Examples of the bioactive quinazolinone skeleton with pharmacological activities

In view of their widely increased application value, a number of classical methods for the synthesis of DHQZs have been reported in the literature. Previous methods for the synthesis of DHQZs, involve: (i) Condensation of anthranilamide with aldehydes or ketones using different catalysts and conditions [21]; (ii) reductive cyclization of 2-nitrobenzamide/ or 2-azidobenzamide with aldehydes or ketones using $SnCl_2$/or metallic Sm in the presence of iodine or SmI_2 [22]; (iii) Condensation of anthranilamide with gem-dibromomethylarenes in in Pyridine/t-BuOK/DMF [23]; (iv) Condensation of 2-aminobenzonitriles and aromatic aldehydes in the presence of [PEG-TEA]OH, choline hydroxide or K_3PO_4 as catalysts [24]; (v) intramolecular cyclization of 2-amino-*N*-alkyl-*N*-allenyl benzamides using $Pd(OAc)_2$ as catalyst [25]; (vi) Reductive desulfurization of 2-thioxo-2, 3-dihydroquinazolinones with $NiCl_2/NaBH_4$ [26]; (vii) The reaction between isatoic anhydride, primary amines, and benzylic alcohols mediated by I_2/K_2CO_3 in water or HBr in DMSO [27]; (viii) The three-component reaction between isatoic anhydride, primary amines, and dialkyl acetylene dicarboxylates in the presence of *N*-sulfonic acid pyridinium chloride [28];

and (ix) oxone-mediated reaction of sec-amines via imine-*N*-oxides with 2-amino-N-substituted benzamides [29]. Continuous studies on the improvement the ability of synthetic chemists in order to improvements in the synthesis of target compounds [30-40] have been lead to introducing other useful and practical methods such as one-pot three-component condensation of isatoic anhydride, aldehydes and ammonium acetate [40-45].

Although these methods have their own merits, some of these procedures have noticeable disadvantages such as low product yields, long reaction times, harsh reaction conditions, extractive product isolation with toxic organic solvents, tedious procedures for preparation of catalysts and use of toxic and expensive catalysts or media. Therefore, there is still a demand for simple, economic and facile synthetic methods in order to obtain DHQZs under mild reaction conditions. With this in mind, and as part of our continuing interest in the development of multi-component reactions, I report herein an efficient and convenient procedure for the synthesis of DHQZs **4** through the $SnCl_2$ dihydrate catalyzed reaction of isatoic anhydride **1**, aldehydes **2**, and NH_4OAc **3** under solvent-free conditions at 110 °C (Scheme 1).



Scheme 1. Synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

Experimental

General remarks

Melting points were determined in open capillaries using an Electrothermal 9100 apparatus and were uncorrected. FT-IR spectra were recorded on a VERTEX 70 spectrometer (Bruker) in the transmission modein spectroscopic grade KBr pellets for all the powders. The ^{1}HNMR spectra were recorded on a Bruker Avance 400-MHz using DMSO as the solvent and TMS as the internal standard. The chemicals were commercially purchased from Merck (Germany), Sigma-Aldrich, and Fluka (Switzerland), they were used without further purification. Thin layer chromatography (TLC) on commercial aluminum-backed plates of silica gel 60 F_{254} was used to monitor the progress of reactions.

*General procedure for the synthesis of DHQZs (**4a–n**)*

A test tube, equipped with a magnetic stir bar, was charged with a mixture of isatoic anhydride (2 mmol), benzaldehyde (2 mmol), NH_4OAc (2.2 mmol), and $SnCl_2.2H_2O$ (20 mol%). The mixture was stirred for a certain period as indicated in Table 3 under solvent-free conditions at 110 °C until the reaction was complete. After completion of the reaction which confirmed by TLC (eluent: *n*-hexane/ethyl acetate: 2/1), water was added to the mixture, and the resulting

precipitate was filtered off. DHQZs which were obtained in good to high yield were purified by recrystallization from ethanol. All products are known compounds and their structures were established from their IR and ^{1}HNMR spectral data and melting points as compared with authentic samples or literature values [21-45].

Selected spectral data of some products:

2-Phenyl-2,3-dihydroquinazolin-4(1*H*)-one (4a)

White solid; ^{1}HNMR (400 MHz, DMSO-d_6): δ = 8.30 (s, 1 H), 7.61 (t, *J* = 6.8 Hz, 1 H), 7.50 (d, *J* = 7.2 Hz, 2 H), 7.32 – 7.41 (m, 3 H), 7.22 – 7.26 (m, 1 H), 7.12 (s, 1 H), 6.75 (d, *J* = 8.0 Hz, 1 H), 6.67 (t, *J* = 7.2 Hz, 1 H), 5.77 (s, 1 H). IR (KBr) cm^{-}1: 3300, 3168, 3060, 2930, 1658, 1609, 1439, 1299, 1165, 745, 640.

2-(4-Methoxyphenyl)-2,3-dihyroquinazolin-4(1*H*)-one: (4c)

White solid; ^{1}H NMR (400 MHz, DMSO- d_6): δ = 8.19 (s, 1 H), 7.61 (d, *J* = 7.2 Hz, 1 H), 7.42 (d, *J* = 8.4 Hz, 2 H), 7.24 (t, *J* = 7.2 Hz, 1 H), 7.01 (s, 1 H), 6.94 (d, *J* = 7.6 Hz, 2 H), 6.74 (d, *J* = 8.0 Hz, 1 H), 6.67 (t, *J* = 7.2 Hz, 1 H), 5.71 (s, 1 H), 3.74 (s, 3H). IR (KBr) cm^{-}1: 3297, 3176, 3046, 2942, 1735, 1655, 1608, 1452, 1302, 1246, 1171, 1029, 802,755, 671.

2-(2,4-Dichlorophenyl)-2,3-dihyroquinazolin-4(1*H*)-one: (4d)

White solid; ^{1}H NMR (400 MHz, DMSO- d_6): δ = 8.10 (s, 1 H), 7.74-7.65 (3 H), 7.58-7.57 (1 H), 7.30-7.26 (1 H), 7.05 (s, 1 H), 6.50 (t, *J* = 7.6 Hz, 1 H), 6.12 (s, 1 H). IR (KBr) cm^{-}1: 3354, 3286, 3179, 3059, 2917, 1603, 1465, 1385, 1317, 1249, 1184, 1115, 1036, 807, 739, 615.

2-(4-Chlorophenyl)-2,3-dihyroquinazolin-4(1*H*)-one: (4e)

White solid; ^{1}H NMR (400 MHz, DMSO- d_6): δ = 8.37 (s, 1 H), 7.64 (d, *J* = 7.6 Hz, 1 H), 7.52 (d, *J* = 8.0 Hz, 2 H), 7.48 (d, *J* = 8.0 Hz, 2 H), 7.26 (t, *J* = 7.6 Hz, 1 H), 7.17 (s, 1 H), 6.76 (d, *J* = 8.0 Hz, 1 H), 6.70 (t, *J* = 7.6 Hz, 1 H), 5.79 (s, 1 H). IR (KBr) cm^{-}1: 3300, 3175, 3057, 2929, 1655, 1599, 1473, 1429, 1377, 1290, 1085, 795, 748, 660.

2-(4-Methylphenyl)-2,3-dihyroquinazolin-4(1*H*)-one: (4f)

White solid; ^{1}H NMR (400 MHz, DMSO- d_6): δ = 8.26 (s, 1 H), 7.62 (d, *J* = 7.6 Hz, 1 H), 7.38 (d, *J* = 8.0 Hz, 2 H), 7.17 – 7.25 (m, 3 H), 7.07 (s, 1 H), 6.75 (d, *J* = 8.0 Hz, 1 H), 6.66 (t, *J* = 7.6 Hz, 1 H), 5.72 (s, 1 H), 2.31 (s, 3 H). IR (KBr) cm^{-}1: 3059, 2855, 1656, 1601, 1433, 1373, 1294, 1163, 1015, 743, 648, 601.

Results and discussion

In order to optimize the reaction conditions, initially, we selected the reaction of isatoic anhydride (1 mmol), *p*-Cl-benzaldehyde (1 mmol), and NH_4OAc (1.2 mmol) as a model reaction. We studied the effect of different amounts of the catalyst, temperature, and solvent on the model reaction. The obtained results from the model reaction to determine the optimum amount of catalyst are presented in Table 1. The model reaction was tested in the absence of the catalyst. As shown in Table 1, the reaction could not proceed even at 110 °C after 90 min. In order to improve the yield and optimize reaction conditions, the same reaction was carried out in the presence of $SnCl_2.2H_2O$ as catalyst under similar conditions. Surprisingly, a significant improvement was observed in the yield of the product. The best result was obtained by using 20 mol % of $SnCl_2.2H_2O$ as catalyst under solvent-free conditions at 110°C (Table 1).

Table 1. Effect of the amount of the catalyst and temperature on the reaction of isatoic anhydride, *p*-Cl-benzaldehyde, and NH_4OAc

Entry	Catalyst (mol%)	Temp °C	Time (min)	Yield (%)
1	-	110 °C	90	Trace
2	5	110 °C	80	59
3	10	110 °C	60	68
4	15	110 °C	50	80
5	**20**	**110 °C**	**40**	**88**
6	25	110 °C	40	88
7	30	110 °C	40	88
8	20	120 °C	40	88
9	20	90 °C	50	73
10	20	80 °C	60	60
11	20	25 °C	60	Trace

Reaction conditions: $SnCl_2.2H_2O$ (20 mol%), isatoic anhydride 1 mmol, *p*-Cl-benzaldehyde 1 mmol, and NH_4OAc 1.2 mmol.

In the next step, to compare the solvent-free condition versus solvent condition, some different solvents such as *n*-Hexane, CH_2Cl_2, CH_3CN, H_2O, and EtOH were tested in this reaction (Table 2). Based on the results, solvent-free condition was found to be more effective than with the solvent in the condensation of isatoic anhydride, *p*-Cl-benzaldehyde and NH_4OAc (Table 2).

Table 2. The synthesis of DHQZ under different solvents

Entry	Solvent	Temp °C	Time (min)	Yield (%)
1	*n*-Hexane	Reflux	50	30
2	CH_2Cl_2	Reflux	50	35
3	CH_3CN	Reflux	50	74
4	H_2O	Reflux	50	79
5	EtOH	Reflux	50	83
6	-	110 °C	40	88

Reaction conditions: $SnCl_2.2H_2O$ (20 mol%), isatoic anhydride 1 mmol, *p*-Cl-benzaldehyde 1 mmol, and NH_4OAc 1.2 mmol.

Due to the success of the above mentioned pilot reaction, we explored the scope and limitations of this promising reaction by varying the structure of aryl aldehyde component. As demonstrated in Table 3, the reactions proceed cleanly under mild and solvent-free reaction conditions. The generality of this three-component one-pot synthesis of target compounds is well-illustrated with structurally diverse aldehydes. In almost all of the cases, this protocol gave good results at 110 °C. The reaction proceeded smoothly and equally well for electron-donating as well as electron-ithdrawing aryl aldehydes to afford the corresponding products in good to high yields (Table 3).

Table 3. Synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

Entry	R	Time (min)	Yield (%)[a]	Melting Point (°C)	Reported Melting Point[ref] (°C)
4a	H	40	92	218–221	218–220 [39]
4b	2,4-$(OCH_3)_2$	50	89	222-225	224–227 [39]
4c	4-OCH_3	48	90	190–192	188–191 [39]
4d	2,4-$(Cl)_2$	52	82	163–165	162–164 [39]
4e	4-Cl	40	88	208–211	207–211 [39]
4f	4-CH_3	40	91	226-229	225–227 [39]
4g	4-Br	45	90	198–200	197–199 [39]
4h	2- Cl	51	80	204–207	203–205 [39]
4i	4-F	35	92	192–195	193–195 [21]
4j	3-Br	40	91	185–188	184–187 [21]
4k	4-OH	50	83	277-280	278-280 [21]
4l	3-OH	50	84	224–227	226–228 [24a]
4m	3-Cl	40	91	185-188	186-189 [24a]
4n	2-OH	55	80	208–211	210–213 [24a]

[a]Yields refer to isolated pure products.

Finally, the efficiency of the present protocol for the synthesis of DHQZs was compared to different type of catalysts and conditions previously reported in the literature [30-38]. The results are shown in Table 4. It can be seen from Table 4 that $SnCl_2.2H_2O$ gives better yields in shorter reaction times than some the other catalysts.

Table 4. Comparison of different catalysts

Entry	Catalyst	Condition	Time(min)	Yield (%)[a]	Ref
1	β- CD [b]	H_2O, 60 °C	90	78–92	30
2	CH_3COOH	rt	240	35–49	31
3	$Ce(SO_4)_2.H_2O$	Solvent-free, 120 °C	30–50	85–97	32
4	SiO_2–$ZnCl_2$	Solvent-free, 100 °C	6–80	51–95	33
5	SPC [c]	Solvent-free, 70 °C	150–210	78–86	34
6	SSA [d]	EtOH, reflux	180–420	73–92	35
7	Al/Al_2O_3 NPs	Solvent-free, 115 °C	8–30	65–98	36
8	Starch sulfate	Solvent-free, 100 °C	5–55	75–96	37
9	Co-CNTs [e]	Solvent-free, MW	10–35	75–98	38
10	$SnCl_2.2H_2O$	Solvent-free, 100 °C	35–55	80–92	-

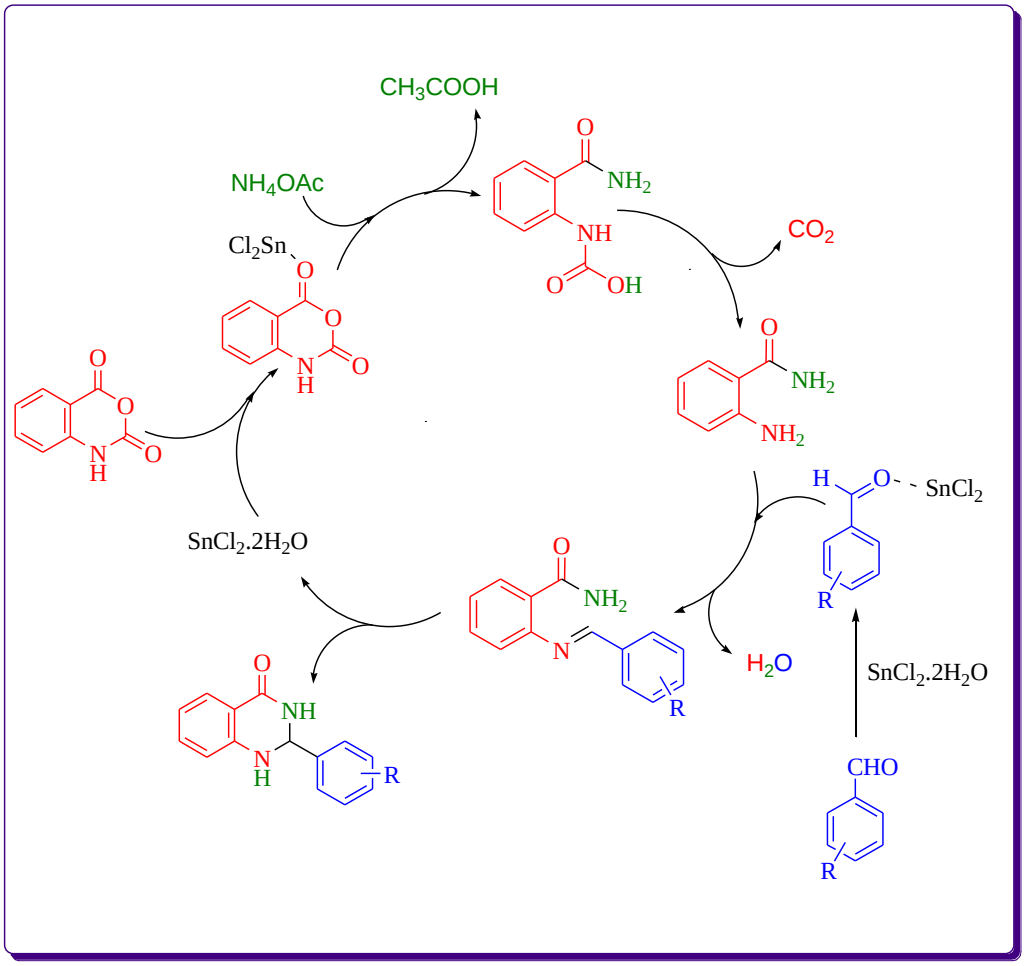
[a]Isolated yield
[b]β-Cyclodextrin
[c]Sulfonated Porous Carbon
[d]Silica sulfuric acid
[e]Cobalt carbon nanotubes

A possible mechanistic pathway for the formation of DHQZs is outlined in Scheme 2. Initially, condensation of isatoic anhydride with ammonium acetate followed by decarboxylation to yield the corresponding anthranilamide. Then condensation of the arylaldehyde with the amino group of anthranilamide gives imine which undergoes cyclization to afford the DHQZs.

Conclusion

In summary, an effective, easy, and rapid procedure has been developed for the preparation of DHQZs using isatoic anhydride, NH_4OAc and benzaldehydes in the presence of $SnCl_2.2H_2O$. This simple and atom-economic protocol represents an attractive approach to a diverse range of DHQZs with a wide substrate scope. Use of readily

available $SnCl_2$ dihydrate, clean reaction profiles, simplicity, and efficiency of the reaction, mild conditions, no use of any solvent, low reaction times, and good to high yields of the products are the main advantages of this protocol.



Scheme 2. Proposed mechanism for the synthesis of DHQZs

Acknowledgments

This research was funded by a grant (No. 1/3314) from the Institute for Science and High Technology and Environmental Sciences, Graduate University of Advanced Technology, Kerman, Iran.

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How to cite this manuscript: Asadollah Hassankhani. Multicomponent reaction for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones using isatoic anhydride, aldehydes and NH_4OAc catalyzed by $SnCl_2.2H_2O$ under solvent-free conditions. *Iranian Chemical Communication*, 2019, 7(3), 248-256.