ORIGINAL PAPER



# An efficient and practical synthesis of benzazolo[2,1-*b*] quinazolinones and triazolo[2,1-*b*]quinazolinones catalyzed by nano-sized NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>)·4Cl

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**Abstract** In this study, 4,4'-(butane-1,4-diyl)bis(1-sulfo-1,4-diazabicyclo[2.2.2]octane-1,4-diium)tetrachloride (NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>)·4Cl is used as a nano-sized Brönsted acid catalyst for the efficient and preferment synthesis of benzazolo[2,1-*b*]quinazolinones and triazolo[2,1-*b*]quinazolinones derivatives. This protocol has many advantages including mild reaction conditions, acceptable reaction times, high yields and easy work-up of the products and reusability of the catalyst.

**Keywords** Nano-catalyst · Multi-component reactions · Solvent-free conditions · Benzazolo[2,1-*b*]quinazolinones · Triazolo[2,1-*b*]quinazolinones

#### Introduction

Nowadays, designing and formulation of fast and facile organic reactions, which target products can be easily separated and purified in high yields, are significant purposes in current drug discovery. From this point of view, multi-component reactions (MCRs) are playing a key role in combinatorial chemistry. In MCRs, target compounds are synthesized with greater efficiency and atom economy because in these reactions the structurally complex products are generated in a single step by several reactants [1, 2].

Heterocyclic compounds have various biological activities which are essential and needed for everyday life.

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Among heterocyclic compounds, quinazolines have a broad scope of pharmacological and biological activities such as anticancer [3], antitumor [4], antidiabetics [5], antiinflammatory [6], antihypertensive [7], antihistamines [8], antiviral [9], antimicrobial [10], antineoplastic [11], analgesic and anti-HIV [12] activities. Employing as a propyl hydroxylase inhibitor [13] and potent immunosuppressive agents [14] are other applications of these compounds. Some antifungal medications are adequate examples which possess quinazolines motif in their structures (Fig. 1).

Benzazolo[2,1-*b*]quinazolinones and triazolo[2,1-*b*] quinazolinones are important derivatives of quinazolines. They are commonly synthesized from the condensation of substituted aldehydes with 2-aminobenzimidazole or 3-amino-1,2,4-triazole as amine sources, and  $\beta$ -diketone. For this purpose, a variety of conditions and catalysts have been used [15–35]. Although these methods are useful, they have some disadvantages such as harsh reaction conditions, long reaction times, low yields of products, need excess amounts of the catalyst, use of toxic solvents and non-recoverable of the catalyst. Hence, there are still needs for introducing easy, efficient and mild procedures to overcome these problems.

## **Experimental**

#### General

The chemical materials and solvents were purchased from Merck, Fluka and Sigma-Aldrich chemical companies. The purity determinations of reaction monitoring were accompanied by TLC on silicagel polygram SILG/UV 254 plates. The products were characterized by comparison of their physical constants such as melting point, use of FT-IR

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Fig. 1 Some antifungal medications possessing the quinazolines nucleus

and/or NMR spectroscopy. The FT-IR spectra were run on a VERTEX 70 Bruker company (Germany) using KBr disk. The <sup>1</sup>H NMR and <sup>13</sup>C NMR were run on a 400-MHz Bruker Avance in DMSO- $d_6$  using TMS as an internal standard. The melting points of all products were defined using an Electrothermal 9100 apparatus. The MS were measured on an Agilent Technology (HP) manufacturer company under 70 eV conditions.

# General procedure for the synthesis of benzazolo[2,1-*b*] quinazolinones and triazolo[2,1-*b*]quinazolinones derivatives

A mixture of the requested benzylic aldehyde (1 mmol), 2-aminobenzimidazole or 3-amino-1,2,4-triazole (1 mmol),  $\beta$ -diketone (1 mmol) and NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>·4Cl (20 mg, 10 mol %) was heated at 90 °C under solvent-free conditions. The progress of the reaction was monitored by TLC (*n*-hexane: EtOAc, 70:30). After completion of the reaction, the reaction mixture was cooled to room temperature and 3 mL of water was added to it. The catalyst was dissolved in water and filtered for separation of the crude product. The separated product was washed twice with water (2 × 5 mL), and the crude product was purified by recrystallization in ethanol.

Spectral data of new compounds are presented below:

9-(2-Nitrophenyl)-5,6,7,9-tetrahydro-[1,2,4] triazolo[5,1-*b*]quinazolin-8(4*H*)-one (Table 2, entry 30): White powder; M.p. = 300–304 °C; IR (KBr, cm<sup>-1</sup>)  $\nu_{max}$ : 3444, 3213, 2910, 1643, 1569, 1471, 1410, 1357; MS: m/z = 311 (M<sup>+</sup>); <sup>1</sup>HNMR (400 MHz, DMSO- $d_6$ ):  $\delta$  (ppm) 1.88–1.99 (m, 2H, CH<sub>2</sub>), 2.17–2.27 (m, 2H, CH<sub>2</sub>), 2.64–2.67 (m, 2H, CH<sub>2</sub>), 6.98 (s, 1H, CH), 7.30 (d, 1H, J = 1.2 Hz), 7.49 (dt, 1H,  $J_1 = 7.6$  Hz,  $J_2 = 0.8$  Hz), 7.61 (dt, 1H,  $J_1 = 7.6$  Hz,  $J_2 = 1.2$  Hz), 7.73 (s, 1H, CH), 7.86 (dd, 1H,  $J_1 = 8$  Hz,  $J_2 = 1.2$  Hz), 11.32 (s, 1H, NH); <sup>13</sup>CNMR (100 MHz, DMSO- $d_6$ ):  $\delta$  (ppm) 21.1, 26.8, 36.4, 53.4, 106.19, 124.4, 129.4, 129.8, 133.8, 135.3, 147.2, 149.0, 150.8, 153.7, 193.9.

9-(4-Bromophenyl)-5,6,7,9-tetrahydro-[1,2,4] triazolo[5,1-*b*]quinazolin-8(4*H*)-one (Table 2, entry 32): White powder; M.p. = 306–308 °C; IR (KBr, cm<sup>-1</sup>)  $\nu_{max}$ : 3429, 3129, 2883, 1648, 1578, 1478, 1411, 1362; MS: *m*/*z* = 344 (M<sup>+</sup>); <sup>1</sup>HNMR (400 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 1.89–2.01 (m, 2H, CH<sub>2</sub>), 2.22–2.34 (m, 2H, CH<sub>2</sub>), 2.61–2.71 (m, 2H, CH<sub>2</sub>), 6.23 (s, 1H, CH), 7.18 (d, 2H, *J* = 8.4 Hz), 7.49 (d, 2H, *J* = 8.4), 7.71 (s, 1H, CH), 11.21 (s, 1H, NH);); <sup>13</sup>CNMR (100 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 21.1, 26.8, 36.7, 44.4, 57.7, 106.6, 121.3, 129.7, 131.6, 141.3, 147.1, 150.6, 153.2, 193.8.

## **Results and discussion**

Very recently and in continuation of our continuous research program on the development of new catalysts for the promotion of the organic transformations [37–41], we have reported the preparation of nano-sized 4,4'-(butane-1,4-diyl)bis(1-sulfo-1,4-diazabicyclo[2.2.2]octane-1,4-diium) chloride ([NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>)]•4Cl) and its



Fig. 2 Structure of NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>·4Cl

 Table 1
 Optimization of the

application for the synthesis of polyhydroquinoline derivatives via Hantzsch condensation (Fig. 2) [36].

Herein and in continuation of these studies, we wish to report the applicability of this nano-catalyst in the promotion of the synthesis of benzazolo[2,1-b] triazolo[2,1-b]quinazolinones quinazolinones and derivatives.

At first, we focused our attention on the synthesis of benzazolo[2,1-b]quinazolinones. For optimization of the

Yield (%) <sup>a</sup>
40
35
85
Trace
95
95
93
92
94
93
65
92

<sup>a</sup>Isolated yields



Scheme 1 Synthesis of benzazolo[2,1-b]quinazolinones derivatives catalyzed by NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>-4Cl



Scheme 2 Synthesis of triazolo[2,1-b]quinazolinones derivatives catalyzed by NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>·4Cl

	A 1 J - 1 J -		Time	Yield	M.p. (°C)		
Entry	Aldehyde	Product	(min.)	(%) <sup>a</sup>	Found	Reported [Ref.]	
1	СНО		13	92	>300	368 [19]	
2	CHO CI		22	89	>300	>300 [20]	
3	CHO CH <sub>3</sub>	H <sub>3</sub> C- N- H	30	88	>300	>300 [20]	
4	CHO CI		15	92	>300	>300 [20]	
5	CHO OMe		20	93	>300	>300 [20]	
6	CHO NO <sub>2</sub>	$\begin{array}{c} O_2 N \\ \hline \\ N \\ H \end{array}$	16	95	>300	>300 [20]	
7	CI CHO		10	94	>300	393 [19]	

	Table 2	Synthesis of	benzimidazoquinazol	none and triazoloquinaz	olinone derivatives using	C <sub>4</sub> (DABCO-S	$(O_3H)_2 + 4Cl$ as the catalyst
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Entre	Aldebude	Droduot	Time	Yield	M.p. (°C)		
Entry	Aldenyde	Product	(min.)	(%) <sup>a</sup>	Found	Reported [Ref.]	
8	МеО		25	95	>300	389 [19]	
9	O <sub>2</sub> N CHO		10	94	>300	335 [19]	
10	NC CHO		15	95	340-342	340-342 [34]	
11	СНО		22	93	>300	344-346 [34]	
12	СНО		35	92	>300	347-349 [34]	
13	СНО	OEt N H	30	91	280-282	281–283 [35]	

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# Table 2 continued

# Table 2 continued

Enuy	/ /////////////////////////////////////	Draduat	rime	Yield M.p		vi.p. ( C)
	Aldenyde	Floduct	(min.)	(%) <sup>a</sup>	Found	Reported [Ref.]
14	CHO Cl	$Cl \rightarrow 0$	15	92	263-265	263–266 [35]
15	CHO CHO OMe	MeO O N N H O OEt	25	93	208-210	210–213 [35]
16	CHO NO <sub>2</sub>	$O_2N$ $O_2N$ $O$	35	92	297-299	296–298 [35]
17	СІСНО	$ \begin{array}{c} Cl \\ O \\ O \\ O \\ H \end{array} $ $ \begin{array}{c} O \\ O \\$	13	93	296-298	297–300 [35]
18	МеО		25	88	254-256	256–258 [35]
19	O <sub>2</sub> N CHO	$ \begin{array}{c}                                     $	20	93	>300	300–302 [35]

Enters	Aldebyde	Product	Time	Yield	M.p. (°C)		
Entry	Aldenyde	Product	(min.)	(min.) (%) <sup>a</sup>		Reported [Ref.]	
20	СНО		20	95	248-250	248-250 [16]	
21	CHO CH <sub>3</sub>		28	92	>300	>300 [20]	
22	CHO NO <sub>2</sub>	$O_2N$	25	92	291-292	290-292 [28]	
23	CHO OMe	MeO N N N H	30	93	>300	>300 [20]	
24	CHO NO <sub>2</sub>	$O_2 N \qquad O \qquad$	30	91	265-266	266-269 [24]	
25	CI CHO		18	94	301-303	303–305 [25]	
26	МеО	OMe OMe NNN H	30	92	222-224	222-224 [16]	

#### J IRAN CHEM SOC

# Table 2 continued

# Table 2 continued

 Enter	Aldehyde	Product	Time	Yield (%) <sup>a</sup>	M.p. (°C)		
 Entry			(min.)		Found	Reported [Ref.]	
27	O <sub>2</sub> N CHO	$NO_{2}$	20	91	285-286	284-285 [16]	
28	СНО		35	92	286-288	287-290 [24]	
29	СНО		25	95	>300	>300 [20]	
30	CHO NO <sub>2</sub>	$O_2N$ $O$ $O$ $N$ $N$ $N$ $N$ $N$ $N$ $H$	35	84	300-304	New	
31	CHO NO <sub>2</sub>	$O_2N$ $O_2N$ $O$ $N$ $N$ $H$	30	91	>300	>300 [20]	
32	Br	Br O N N H	20	94	306-308	New	
33	МеО		35	92	306-308	>300 [20]	

<sup>a</sup> Isolated yields

Scheme 3 Proposed mechanism for the synthesis of benzimidazoquinazolinone and triazoloquinazolinone derivatives in the presence of NS- $C_4$ (DABCO-SO<sub>3</sub>H)<sub>2</sub>·4Cl



reaction conditions, the condensation of 4-chlorobenzaldehyde with 2-aminobenzimidazole and dimedone in the presence of NS-C<sub>4</sub> (DABCO-SO<sub>3</sub>H)<sub>2</sub>·4Cl was selected as a model reaction, and various conditions including different amounts of the catalyst, temperatures and solvent were examined (Table 1). After careful studies, the optimal reaction conditions were selected as shown in Scheme 1. Also 3-amino-1,2,4-triazole was another amine source which could be successfully produced the corresponding products under this optimized reaction condition (Scheme 2).

After optimization of the reaction conditions and in order to establish the effectiveness and the acceptability of the method, we have extended it to the three-component condensation of various substituted benzaldehydes, different type of  $\beta$ -diketones (dimedone, cyclohexanedione and ethyl acetoacetate) and/or 2-aminobenzimidazole or 3-amino-1,2,4-triazole (Table 2). It was observed that a wide range of aromatic aldehydes containing electron-withdrawing as well as electron-donating groups such as Cl, CH<sub>3</sub>, OCH<sub>3</sub>, NO<sub>2</sub> and CN in the *ortho*, *meta* and *para* positions of the benzene ring were easily converted to their corresponding benzimidazoquinazolinone and/or triazoloquinazolinone in short reaction times with good to excellent isolated yields. It is interesting to note





Table 3 Compared performance of various catalysts with NS-C4(DABCO-SO3H)2.4Cl in the synthesis of quinazolinone derivatives

Entry	Compound	Catalyst (mol %)	Conditions	Time (min.)	Yield (%) <sup>a</sup>	References
1		- (-)	EtOH/reflux	90	64	[18]
	N N N					
2		- (-)	Solvent free/110 °C	420	85	[20]
3		$H_6P_2W_{18}O_{62} \cdot H_2O(1)$	CH <sub>3</sub> CN/reflux	15	96	[24]
4		Sulfamic acid (0.005)	CH <sub>3</sub> CN/80 °C	15	94	[25]
5		Iodine (10)	CH <sub>3</sub> CN/reflux	10	84	[28]
6		Fe <sub>3</sub> O <sub>4</sub> @chitosan (2 mg)	EtOH/40 °C	90	90	[29]
7		<i>p</i> -TSA (15)	CH <sub>3</sub> CN/40-50 °C	25	95	[30]
8		- (-)	Acetic acid/60 °C	20	94	[31]
9		C <sub>4</sub> (DABCO-SO <sub>3</sub> H) <sub>2</sub> ·4Cl (10)	Solvent free/100 °C	13	92	This work
10	$\bigtriangleup$	- (-)	DMF/reflux	30	76	[16]
11		- (-)	Solvent free/110 °C	240	93	[20]
12		$H_6P_2W_{18}O_{62} \cdot H_2O(1)$	CH <sub>3</sub> CN/reflux	30	95	[24]
13		Sulfamic acid (0.005)	CH <sub>3</sub> CN/80 °C	30	95	[25]
14		[Bmim]Br (0.1)	Solvent free/100 °C	20	93	[27]
15		Iodine (10)	CH <sub>3</sub> CN/reflux	10	81	[28]
16		<i>p</i> -TSA (15)	CH <sub>3</sub> CN/40-50 °C	30	96	[30]
17		- (-)	Acetic acid/60 °C	25	95	[31]
18		$C_4(DABCO-SO_3H)_2 \cdot 4Cl (10)$	Solvent free/100 °C	20	95	This work

<sup>a</sup> Isolated yields

that 2-naphthaldehyde and fluorene-2-carbaldehyde as polycyclic aromatic aldehydes also provided the desired products in very good yields (Table 2, entries 11–12, 28). It should be mentioned that because of the formation of a mixture of unidentified products the method is not useful for the synthesis of the same products from aliphatic aldehydes.

The proposed mechanism for the synthesis of benzimidazoquinazolinone and triazoloquinazolinone derivatives in the presence of NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>·4Cl as a promoter is shown in Scheme 3. On the basis of this mechanism, NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>·4Cl catalyzes the reaction through: (a) the activation of the carbonyl groups for the nucleophilic attack of  $\beta$ -diketones, (b) dehydration of the intermediate (I), (c) acceleration of the nucleophilic attack of the amine group on (II) and (III) and (d) dehydration of (IV).

The reusability of catalyst is worthwhile and ecofriendly. For this purpose, we chose the synthesis of 12-(4-chlorophenyl)-3,3-dimethyl-3,4,5,12-tetrahydrobenzo[4,5]imidazo[2,1-b] quinazolin-1(2H)-one(Table 2, entry 17) as a model reaction to investigate thereusability of NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>·4Cl under the optimized reaction conditions. After completion of the reaction, the solid product was washed with water to separatethe catalyst (catalyst is soluble in water). Then, the filtrate was evaporated under vacuum up to 100 °C and theobtained catalyst was washed with diethyl ether, dried andreused for the same reaction. The recycled catalyst wasreused at least for 5 runs without considerable loss of itsactivity. The obtained results are demonstrated in Fig. 3.

To highlight the merits of our newly developed procedures, we have compared our results obtained for the synthesis of 3,3-dimethyl-12-phenyl-3,4,5,12-tetrahydrobenzo[4,5]imidazo [2,1-*b*]quinazolin-1(2*H*)-one (Table 2, entry 1) and 6,6-dimethyl-9-phenyl-5,6,7,9-tetrahydro-[1,2,4]triazolo[5,1-*b*]quinazolin-8 (4*H*)-one (Table 2, entry 20) using NS-C<sub>4</sub>(DABCO-SO<sub>3</sub>H)<sub>2</sub>·4Cl as the catalyst with other reported results for the same transformations (Table 3). The results showed that the newly expanded method avoids some of the disadvantages affiliated by the other procedures such as longer reaction time, large excesses of reagents, use of organic solvents and/or toxic reagents and low yields of the products.

## Conclusions

In the conclusion of this study, we have introduced NS- $C_4(DABCO-SO_3H)_2$ ·4Cl as a highly powerful nano-catalyst for the simple and efficient synthesis of benzimidazoquinazolinone and triazoloquinazolinone derivatives. The obtained results show that this method is convincingly superior to other reported procedures in terms of the reaction times and yields. Simple experimental procedure and use of a reusable catalyst with lower loading are other advantages of this method. Furthermore, this process avoids problems associated with organic solvents and liquid acids used, which makes it a useful and attractive strategy in view of economic and environmental advantages.

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