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One-Pot, Borax-mediated synthesis of structurally diverse N, S-heterocycles in water

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ABSTRACT

Herein, a borax–mediated convenient and efficient strategy for the synthesis of prominent structurally diverse N, S-heterocycles such as quinazolin-4(3H)-one, benzothiadiazine 1,1-dioxide, benzothioazole, and benzoxazoles from readily available 2-aminobenzamide/2-aminobenzenesulfonamide/2-aminothiophenol/2-aminophenol with α,α,α-trihalotoluenes at 100 ℃ in water is elaborated. Upon using aldehydes instead of α,α,α-trihalotoluenes, the reactions proceed through domino fashion under the catalytic effect borax to yield 2,3-dihydroquinazolin-4(1H)-one, 3,4-dihydrothiadiazine 1,1-dioxide, and benzothiazoles in one-pot at 60 ℃. The advantages of this protocol are practical simplicity, large substrate scope, moderate to excellent yields, and the use of water as the solvent.

Introduction

Nitrogen-Sulfur heterocyclic compounds have raised the great interest of organic chemists for several decades, due to their innumerable importance in natural products, advanced materials, and designing bioactive molecules used for various pharmaceutical necessities [1]. Specifically, quinazolinones, benzothiadiazole-1,1-dioxide, and its polycyclic motifs are the very important building block of heterocycles because of their biological and pharmaceutical activities (Fig. 1) including antibacterial [2], antiviral [3], antimalarial [4], anticancer [5], antihypertensive [6], antitubercular [7], and they also act as inhibitors of various enzymes [8]. Additionally, quinazolinones are acting as ligands for AMPA and benzodiazepine receptors in the CNS organism or as DNA binders [9].

Owing to its long-standing recognition, chemists paid a great deal of attention to find a straightforward synthetic approach to quinazolin-4(3H)-one, and benzothiadiazine1,1-dioxides [10]. A literature survey reveals numerous studies on synthetic approach to quinazoline-4(3H)-one and benzothiadiazine1,1-dioxides from 2-aminobenzamide/2-aminobenzenesulfonamide with simple aryl precursors (aldehydes [11], benzyl alcohols [12], and benzoic acids [13]) used under metal or metal-free conditions. In particular, quinazolin-4(3H)-ones can also be prepared by the condensation reactions of 2-aminobenzamide with carbonyl equivalents, such as benzyl amines [14], benzyl halides [15], methyl arenes [16], benzoyl chlorides [17], aryl ketones [18], CO/aryl halides [19], and aryl halides with isocyanides insertion [20] (Scheme 1). Many of these methods, some of which have drawbacks, such as low or varying yields and time, use of costly chemicals, less substrate tolerance, use of coupling agents or metal catalysts, frequently require harsh reaction conditions and time-consuming work-up procedures. Therefore, a simple and an atom economy approach for the synthesis of quinazolinones and benzothiadiazine1,1-dioxides from easily available starting materials is still desirable.

Borax ($Na_2B_4O_7$) has been recently advanced as a mild, environmentally benign, non-toxic, and naturally occurring substance that provides hydroxyl anion (Brønsted base) and boric acid (Lewis acid) in aqueous solution (pH value 9.5). It has shown chemo, and stereoselectivity in many organic transformations such as Nef reaction, substitution, condensation, addition (including hetero-Michael reaction), etc. [21]. On the other side, the literature survey indicated that the groups of α,α,α-trihalotoluenes have been used to prepare a variety of heterocycles with di-nucleophiles [22]. Our ongoing study on the use of borax as catalyst and reagent in a variety of reactions to synthesize the potentially bioactive nitrogen and sulfur heterocycles is still on [21f-h].

In the current work, we have developed a new synthetic protocol in water for the synthesis of quinazolin-4(3H)-one/benzothiadiazine 1,1-dioxides/benzothiazole/benzoxazoles from a simple and easily accessible 2-aminobenzamide/2-aminobenzenesulfonamide/2-aminothiophenol/2-aminophenol and α,α,α-trihalotolue-

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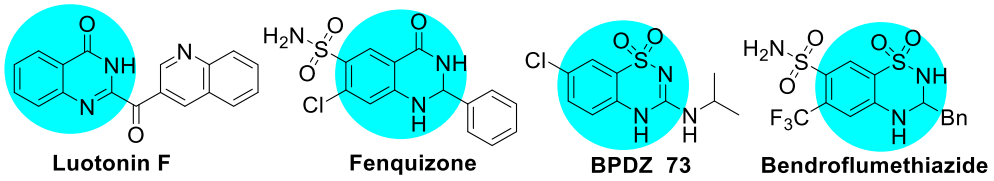
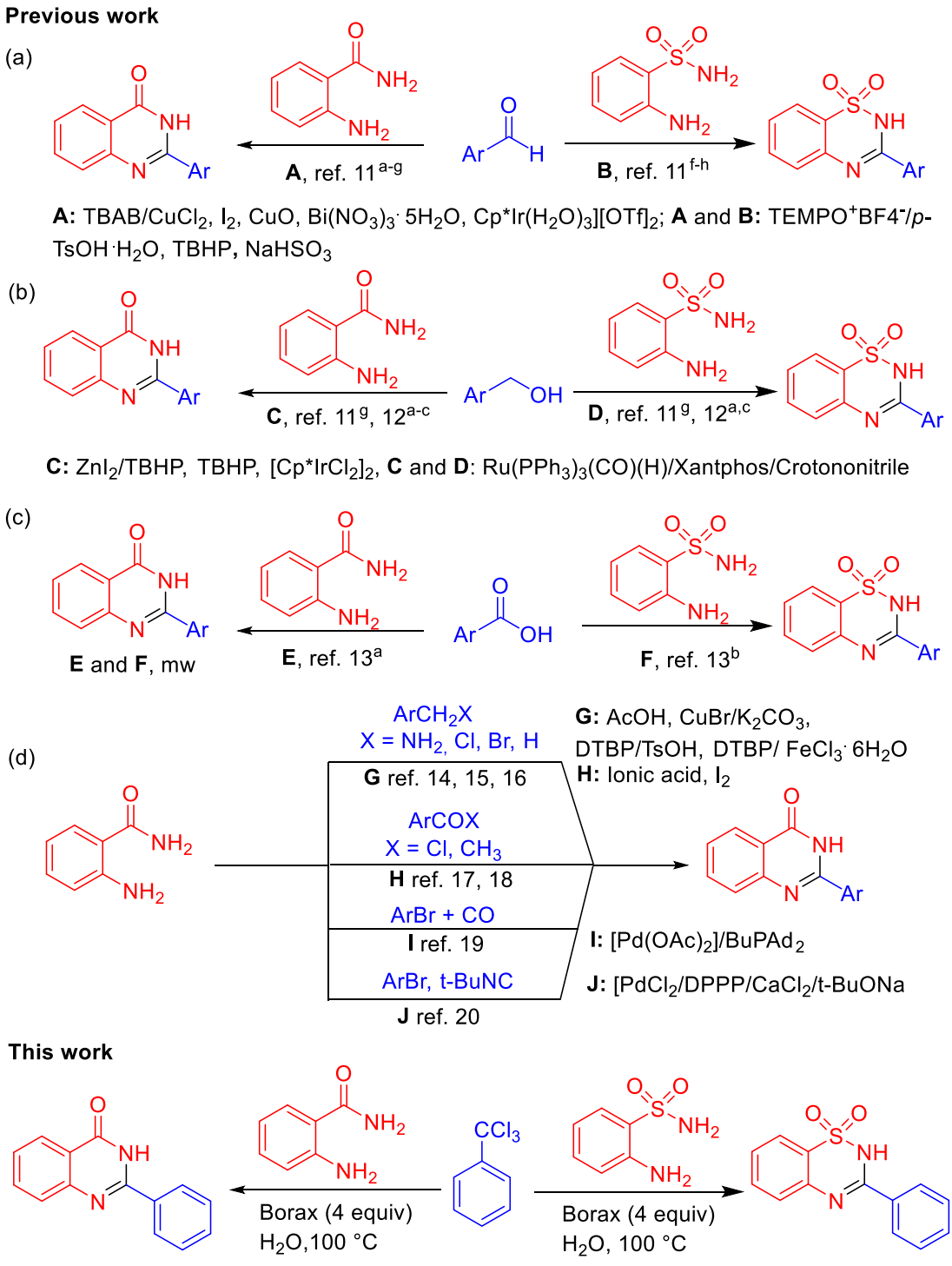


Fig. 1. Examples of biologically and pharmaceutically important N, S-heterocycles.



Scheme 1. Strategies for the synthesis of quinazoline-4(3H)-one and benzothiadiazine1,1-dioxides.

nes, and also one-pot cascade synthesis of 2,3-dihydroquinazolin-4(1H)-one/3,4-dihydrothiadiazine 1,1-dioxide/benzothiazoles from 2-aminobenzamide/2-aminobenzenesulfonamide/2-aminothiophenol and aldehydes in the presence of borax (Scheme 1).

Results and discussion

For the initial study, the synthesis of quinazolin-4(3H)-one (**3a**) was chosen as a model reaction and optimization was done with 2-aminobenzamide (**1a**) and α,α,α-trichlorotoluene (**2a**), in the presence of 3 equivalent of borax and the optimized conditions are shown in Table 1. We observed when solvents either aprotic solvents such as acetonitrile and toluene or protic solvents ethanol and methanol were used in the reaction, only the desired product **3a** was obtained in fewer yields (Table 1, entries 1–4). It is gratifying, while the reaction conducted in water gives 68% of **3a** was obtained at 80 °C for 14 h (Table 1, entry 5). Further, increasing the temperature to 100 °C, the amount of borax to 4 equivalent, and time, the reaction yield is increased to 78% (Table 1, entry 6). A further increase and decrease in the amount of borax were optimized and the results are summarized in Table 1 (entries, 7–9). The reaction proceeds in a trace amount at room temperature and the reaction did not proceed at all in the absence of borax (Table 1, entries 10 and 11). The formation of compound **3a** was confirmed by comparing FT-IR, ^{1}H, and ^{13}C NMR spectroscopic data with literature data. The optimal reaction conditions were, therefore, achieved with 2-aminobenzamide (**1a**) (0.3 mmol), α,α,α-trichlorotoluene (**2a**) (0.36 mmol) and 4 equiv of borax in water at 100 °C for 14 h (Table 1, entry 6).

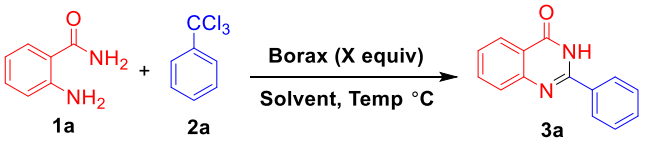
The protocol's applicability was checked under optimal conditions. As shown in Table 2, the scope of the reaction involving α,α,α-trichlorotoluene was first investigated. The reactions of α,α,α-trichlorotoluene substituted with electron-withdrawing groups (F, Cl) at various positions in the aryl ring furnished the corresponding products in moderate to satisfactory yields (Table 2, 60–74%, **3b**-**3d**). Substituted 2-aminobenzamide with electron-withdrawing and electron-donating groups at the 5-position, such as fluoro, chloro, nitro, and methyl groups, is then investigated. Electron-donating groups were found to yield products better than electron-withdrawing groups respectively (Table 2, 69–75%, **3e**-**3h**).

After the successful conversion to the desired product, the scope of this reaction was further extended to the synthesis of benzothiadiazine 1,1-dioxides. Instead of 2-aminobenzamide, 2-aminobenzenesulfonamide was used. The reactions proceed smoothly and the results are in Table 2. It is noteworthy that substituted benzothiadiazine 1,1-dioxides derivatives could be prepared in good yields (Table 2, **5a-c**. Furthermore, to demonstrate the utility of the current method, benzothiazole, and benzoxazole derivatives were synthesized under optimal conditions using 2-aminothiophenol and 2-aminophenol in place of 2-aminobenzamide. The corresponding products were obtained in 71–85% yields (Table 2, **7a-c**, **9a-c**).

After successful accomplishment of the reaction of α,α,α-trichlorotoluenes, and 2-aminobenzamide, we noted the importance of 2,3-dihydroquinazolin-4(1*H*)-ones [23], which can also be prepared from 2-aminobenzamide. In recent years, several methods have been reported for the preparation of 2,3-dihydroquinazolin-4(1*H*)-ones using a variety of homogeneous or heterogeneous catalysts such as I_2, $ZrCl_4$, $SbCl_3$, cyanuric chloride, TFA, morpholinoethanesulfonic acid, $B(C_6F_5)_3$, $Y(OTf)_3$, $[Al(H_2PO_4)_3]$, ZrO_2–Al_2O_3, L-proline nitrate, amberlyst-15, α-chymotrypsin, graphene oxide, heteropoly acids, ionic liquids, β-cyclodextrinSO_3H, PEG-400 and magnetic nanoparticles [24]. The catalysts that have been used are either expensive or relatively more toxic. Because of the above, we further stretched the use of borax for the preparation of 2,3-dihydroquinazolinones, which could be produced by simply replacing α,α,α-trichlorotoluenes with aldehydes as shown in Scheme 2. The 20 mol% borax is sufficient to complete the reaction with high yields at 60 °C. It was noticed that the methodology is tolerable with a variety of substituents on the substrate. The observed corresponding products in each result were obtained in high yields and short reaction time of 2–3 h (Table 3). The substituent on the aromatic aldehydes showed slightly different effects on the yields. The reactions of aromatic aldehydes with electron-withdrawing groups afforded little better yields of products than those with the electron-donating groups (Table 2, 11a-**g**). Moreover, the reactions of heterocyclic aldehydes with thiophene also gave a good yield (Table 2, 11h). To further extend the scope of the reaction, a few substituted 2-aminobenzamides (5-methyl and 5-chloro) were used to carry out reaction with aldehydes. It also gave 87–89% yields (Table 2, 11i and 11j). Unfortunately, propionaldehyde and acetophenone failed to produce the desired product (Table 2, 11k and 11l).

Later, the above reaction conditions were then applied towards the synthesis of 3,4-dihydrobenzothiadiazine 1,1-dioxides (Table 3). 2-aminobenzenesulfonamide reacts with benzaldehyde to produce 3,4-dihydrobenzothiadiazine 1,1-dioxide **12a** in 87%

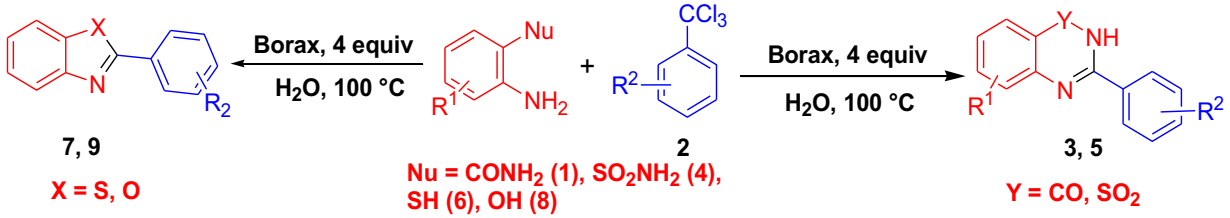
Table 1
Optimization of reaction conditions.[a]



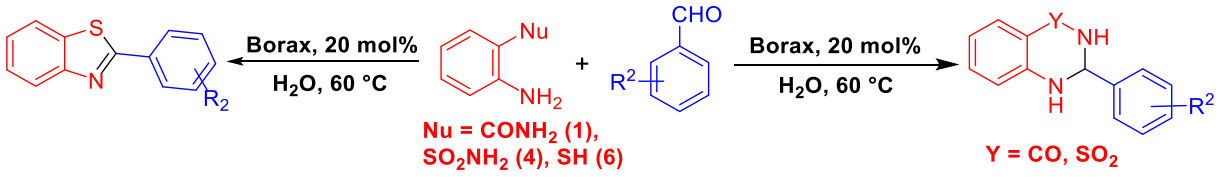
Entry	Reagent	Solvent	Temp. (°C)	Time (hours)	Yield (%)[a]
1.	Borax (3equiv)	CH_3CN	80	14	25
2.	Borax (3 equiv)	Toluene	80	14	10
3.	Borax (3 equiv)	EtOH	80	14	45
4	Borax (3 equiv)	MeOH	60	14	34
5.	Borax (3 equiv)	H_2O	80	14	68
6.	**Borax (4 equiv)**	**H_2O**	**100**	**14**	**78**
7.	Borax (5 equiv)	H_2O	100	14	79
8.	Borax (2 equiv)	H_2O	100	14	51
9.	Borax (1 equiv)	H_2O	100	14	22
10.	Borax (4 equiv)	H_2O	r.t	14	Trace
11.	–	H_2O	100	16	–

[a]Isolated yields.

Table 2
Substrate scope.



3a, 78%, 14 h	**3b**, 74%, 14 h	**3c**, 69%, 14 h	**3d**, 60%, 16 h
3e, 73%, 14 h	**3f**, 70%, 14 h	**3g**, 69 %, 14 h	**3h**, 75 %, 14 h
5a, 82%, 14 h	**5b**, 88%, 14 h	**5c**, 76%, 16 h	**7a**, 85%, 10 h
X = F, **7b**, 76%, 10 h X = Cl, **7c**, 81%, 10 h	**9a**, 80%, 10 h	**9b**, 78%, 10 h	**9c**, 71%, 10 h



Scheme 2. Synthesis of 2,3-dihydroquinazolinone, 3,4-dihydrobenzothiadiazine 1,1-dioxide and benzothiazoles.

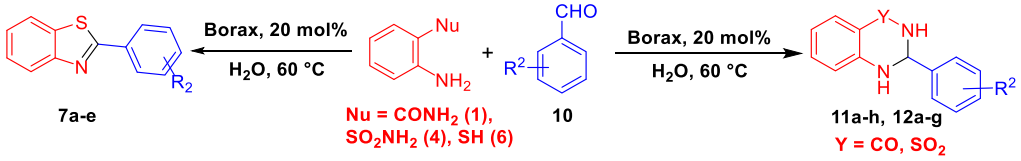
yield. Whereas halo, nitro, methoxy-substituted benzaldehyde, and 2-napthaldehydes offered yields ranging from 80 to 90% (**12b-f**). As well as heterocyclic 2-thiophene carboxaldehyde furnished product **(12g)** in 85% yield.

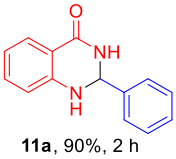
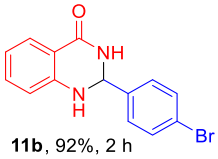
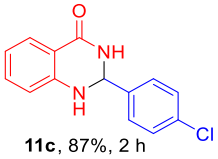
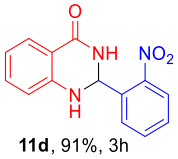
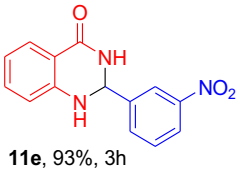
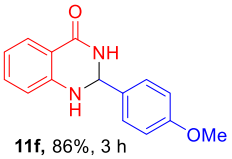
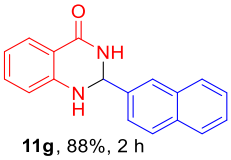
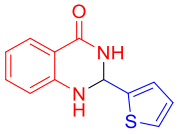
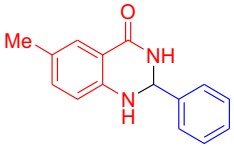
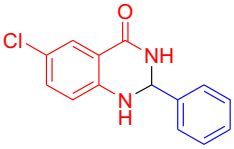
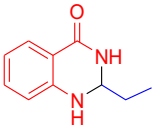
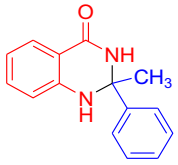
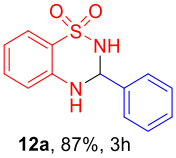
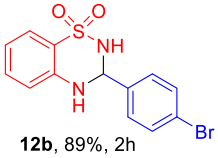
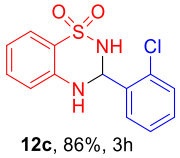
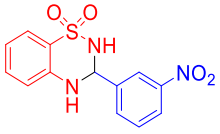
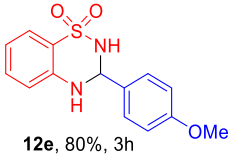
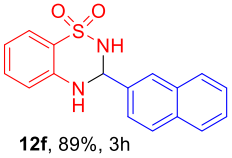
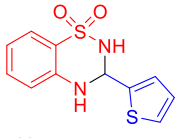
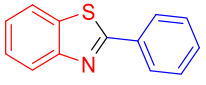
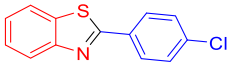
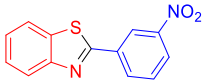
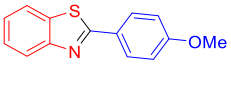
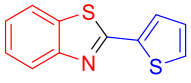
Further, the reaction is applied to synthesize benzothiazole derivatives under a similar condition from the reaction of 2-aminothiophenol with aromatic aldehydes. Aldehydes with different substituents like *p*-Cl, *m*-NO_2, *p*-OMe or 2-thiophene carboxaldehyde afforded the expected products in 80–87% yields (Table 3, 7a, and 7c-f).

All the compounds were purified by column chromatography. The structure of all products was confirmed by comparing spectroscopic data with literature values. The structure of 3-(4-methoxyphenyl)-3,4-dihydro-2H-benzo[e][1,2,4]thiadiazine 1,1-dioxide dimethyl sulfoxide mono solvate (**12e•DMSO)** was unambiguously proved by X-ray single-crystal studies (Fig. 2).

A probable mechanism for the synthesis of quinazolin-4(1H)-one **3a** and 2,3-dihydroquinazolinones **11a** is illustrated in Scheme 3 [21,22]. The reaction seems to be initially occurring by the partial hydrolysis α,α,α-trichlorotoluene by OH^- (produced

Table 3
Substrate scope.



 **11a**, 90%, 2 h	 **11b**, 92%, 2 h	 **11c**, 87%, 2 h	 **11d**, 91%, 3h
 **11e**, 93%, 3h	 **11f**, 86%, 3 h	 **11g**, 88%, 2 h	 **11h**, 86%, 3 h
 **11i**, 89%, 3h	 **11j**, 87%, 3h	 **11k**, 0%, 6 h	 **11l**, 0%, 6 h
 **12a**, 87%, 3h	 **12b**, 89%, 2h	 **12c**, 86%, 3h	 **12d**, 90%, 3h
 **12e**, 80%, 3h	 **12f**, 89%, 3h	 **12g**, 85%, 3h	 **7a**, 87%, 6h
 **7c**, 83%, 6h	 **7d**, 86%, 5h	 **7e**, 85%, 6h	 **7f**, 80%, 6h

from aqueous borax) leads to the formation of imine (**A**) from 2-aminobenzamide. Then, the presence of $B(OH)_3$ and OH^- in the reaction medium help in intramolecular cyclization through (**B**) to the desired product **3a**. Whereas, for the synthesis of 2,3-dihydroquinazolin-4(1H)-one (**11a**), the formation of imine takes place

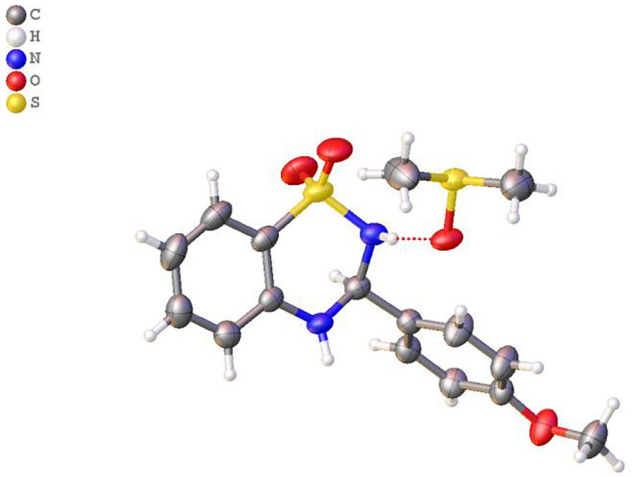


Fig. 2. Single-Crystal XRD structure of **12e** (CCDC 2063586).

initially (**A'**), which cyclizes in the presence of borax to give the final product (Scheme 2b).

Conclusions

In summary, we have developed a convenient and straightforward borax-mediated synthetic protocol for the synthesis of biologically relevant quinazoline-4(3H)-one, benzothiadiazine 1,1-dioxide, benzothiazole, and benzoxazoles from simple and easily accessible 2-aminobenzamide/2-aminobenzenesulfonamide/2-aminothiophenol/2-aminophenol and α,α,α-trihalotoluenes at 100 ℃. In addition, one-pot, borax catalyzed a domino strategy for the synthesis of 2,3-dihydroquinazolin-4(1H)-one, 3,4-dihydrothiadiazine 1,1-dioxide, and benzothiazoles from both aromatic and hetero-aromatic aldehydes with 2-aminobenzamide/2-aminobenzenesulfonamide/2-aminothiophenol at 60 ℃. Moderate to high yields of the desired products and green solvents such as water are important features of this protocol.

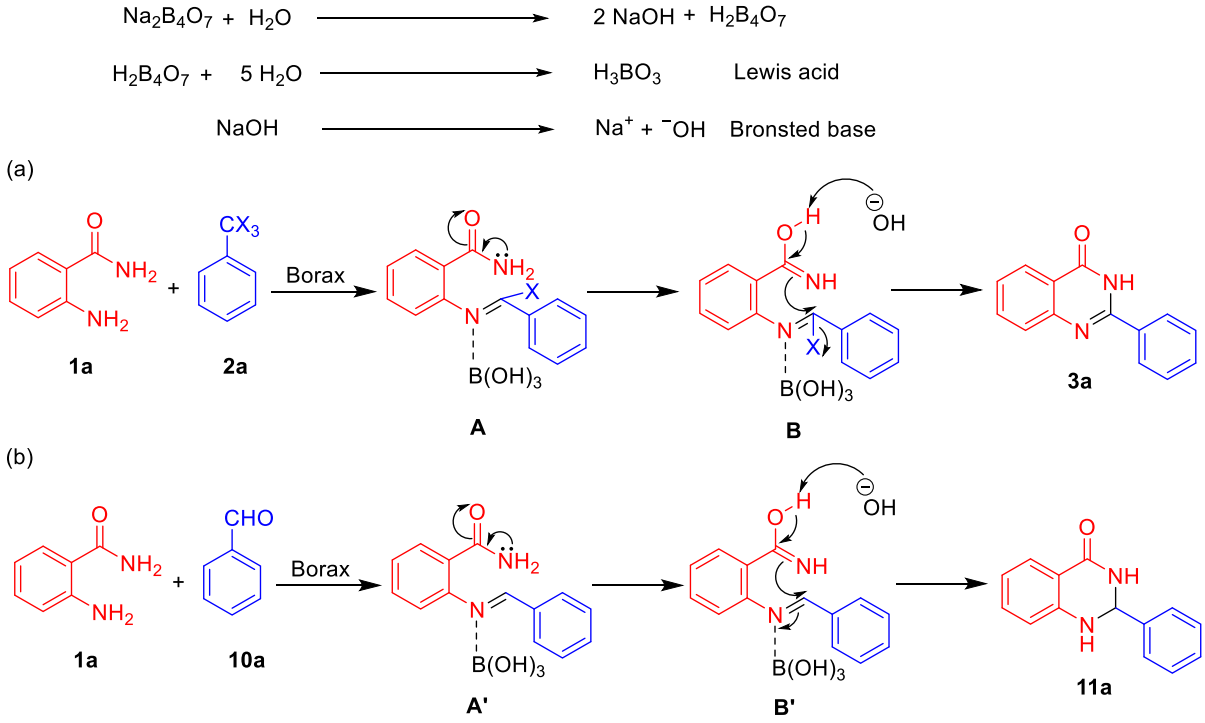
Experimental section

General procedure for the synthesis of quinazolin-4(3H)-one/benzothiadiazine 1,1-dioxides/benzothiazole/benzoxazoles (3, 5, 7, and 9)

2-Aminobenzamide/2-aminobenzenesulfonamide/2-aminothiophenol/2-aminophenol (0.3 mmol), α,α,α-trichlorotoluene (0.36 mmol), and borax (4 equiv) were taken in a 50 ml round bottom flask, and the reaction mixture was stirred in H_2O (3 ml) at 100 ℃ for a period of the time till the completion of the reaction (monitored by TLC). After completion of the reaction, it was extracted with ethyl acetate. The combined organic layers were washed with water and brine, dried over sodium sulfate, and concentrated under reduced pressure. The crude product was purified by column chromatography to give the desired product. All compounds were well characterized by FT-IR, ^{1}H, ^{13}C NMR, and melting point analysis.

General procedure for the synthesis of 2,3-dihydroquinazolinones/3,4-dihydrobenzothiadiazine 1,1-dioxide/benzothiazoles (11, 12, and 7)

2-Aminobenzamide/2-aminobenzenesulfonamide/2-aminothiophenol (0.3 mmol), aldehyde (0.3 mmol), and borax (20 mol %) were taken in a 50 ml round bottom flask, and the reaction mixture was stirred in H_2O (2 ml) at 60 ℃ for a period of time till the completion of the reaction (monitored by TLC). After completion of the reaction, it was extracted with ethyl acetate. The combined organic layers were washed with water and brine, dried over sodium sulfate, and concentrated under reduced pressure. The crude product was purified by column chromatography to give the desired



Scheme 3. Proposed mechanism for the synthesis of quinazoline-4(3H)-one (**a**) and 2,3-dihydroquinazolinones (**b**).

product. All compounds were well characterized by FT-IR, ^{1}H, ^{13}C NMR, and melting point analysis.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2021.153159.

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