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COMMUNICATION

Elemental sulfur mediated 2-substituted benzothiazole formation from 2-aminobenzenethiols and arylacetylenes or styrenes under metal-free condition

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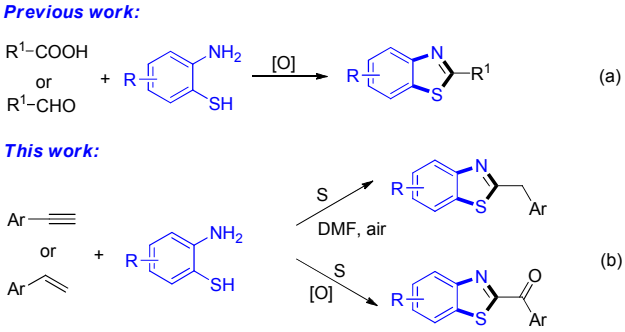
An oxidative cyclization of 2-aminothiophenols and arylacetylenes or styrenes for the synthesis of 2-alkyl benzothiazoles and 2-acylbenzothiazoles has been developed. Elemental sulfur was used as the effective oxidant to give the corresponding product in good yield under metal-free conditions.

Benzothiazole derivatives are privileged motif frequently found in pharmaceuticals and many other biologically active products.[1] Moreover, benzothiazole moieties are also found in many functional molecules such as ligands for catalytic reactions.[2] Therefore, the development of efficient methods for construction of benzothiazole derivatives attracted considerable interest. Over the past few decades, tremendous progress has been made to synthesize 2-substituted benzothiazoles, an important class of benzothiazole derivatives.[3] The condensation reaction of 2-aminothiphenols with carboxylic acids or aldehydes under oxidative conditions is the most common used method to construct various 2-substituted benzothiazoles (Scheme 1, a).[4] However, most of these transformations involve the use of harsh reaction conditions, such strong acidic or oxidative conditions. Recently, the Bao and Yu developed a Brϕnsted acids catalyzed 2-substituted benzothiazoles formation from 2-aminothiophenols and *β*-ketones under relative mild conditions.[5] The transition-metal-catalyzed intramolecular cyclization of 2-haloanilides/analogues provided an efficient alternative approach.[6] The reaction of *o*-halonitrobenzenes, elemental sulfur and another reducing partners such as benzylamines, benzaldehydes, methylhetarenes, acetophenones and phenylacetic acids provided a convenient approach by using nitroarenes as the substrates.[7] In recent years, great attention has been paid on the transition-metal-catalyzed direct functionalization of the benzothiazole core for 2-substituted benzothiazole preparation.[8]

In most cases, however, the aforementioned synthetic methods involve 2-aminothiophenol starting materials required strong oxidant or high temperature, which is a challenging for functional group tolerance. In addition, arylacetic acids or aryl acetaldehydes were required for 2-benzylic benzothiazole formation.[9] The substrate scope is limited for this kind of chemicals. Therefore, the development of efficient method for 2-substituted benzothiazoles under metal-free and mild conditions with good functional group compatibility is still highly desirable. Elemental sulfur widely exists in nature, and due to its nontoxicity and stability under normal conditions, it was employed for the vulcanization of rubber and the synthesis of sulfuric acids and as sulfur source for preparing other sulfur-containing compounds.[10-11] Moreover, elemental sulfur could serve as an effective oxidant in organic synthesis to enable oxidative coupling reactions.[12] Recently, the Nguyen group,[7,13] we[14] and others[15] have developed various methods for sulfur-containing heterocycle synthesis using elemental sulfur as the sulfur source under transition-metal-free conditions. As our continuing interest in heterocycle preparation from readily available starting materials under transition-metal free conditions,[16] herein, we report a metal-free oxidative cyclization reaction of 2-aminothiaphenols and arylacetylenes or styrenes for the facile synthesis of 2-alkyl benzothiazoles and 2-acylbenzothiazoles, in which elemental sulfur was found to be the key for the success of this aerobic cyclization process (Scheme 1, b).



Scheme 1 Different methods for 2-substituted benzothiazole formation from 2-aminobenzenethiols.

We started our investigation using phenylacetylene (**1a**) and 2-aminothiophenol (**2a**) as the model system. No desired product **3aa** was observed when the reaction was carried out in NMP (N-methyl pyrrolidone) in the absence of oxidant (Table 1, entry 1). TBHP and H_2O_2 both were not effective oxidant for this kind of transformation (entries 2 and 3). The desired product could be

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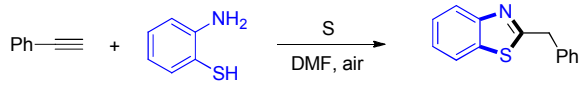
observed when $K_2S_2O_8$ and DDQ were used as the oxidants (entries 4 and 5). To our delight, **3aa** was obtained in 32% yield when 1 equiv. of sulfur powder was used as the oxidant (entry 6). Encouraged by this discovery, we screened several organic solvents for this reaction using sulfur powder as the oxidant. Better yield was obtained when the reaction was carried out in DMF (entry 7). However, much lower yield was observed when the reaction was carried out in toluene and chlorobenzene (entries 8 and 9). The use of DMSO as the solvent completely inhibited the reaction (entry 10). The reaction yield could be improved to 87% when increasing the sulfur powder to 2 equiv. (entry 11). Further increasing the sulfur powder to 3 equiv. could not increase the reaction yield (entry 12). Similar yield was obtained when the reaction was carried out in oxygen or nitrogen (entries 12 and 14). This indicates that sulfur powder should acted as the oxidant solely for this kind of reaction.

Table 1 Optimization of the reaction conditions[a]



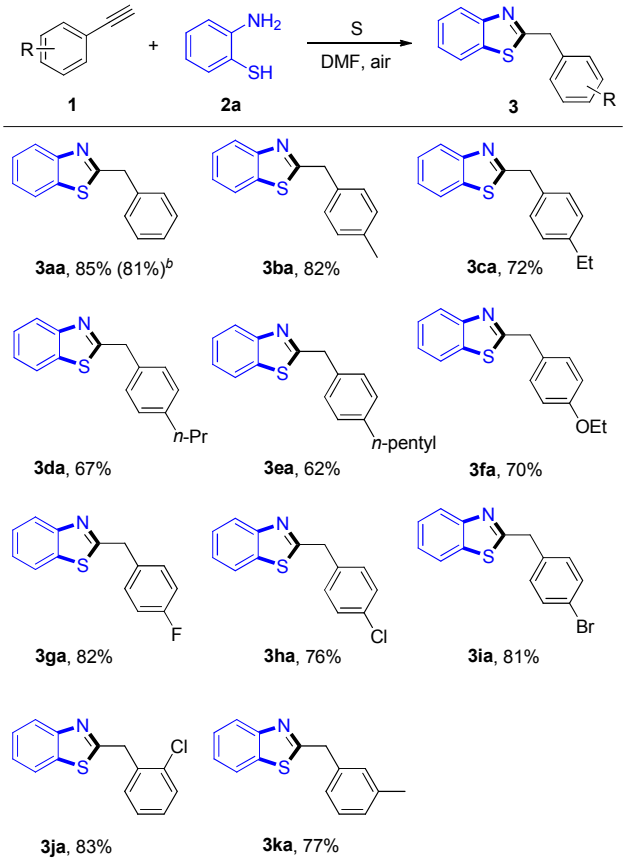
1a **2a** **3aa**

entry	oxidant	solvent	yield[b] (%)
1	—	NMP	ND
2	TBHP	NMP	trace
3	H_2O_2	NMP	trace
4	$K_2S_2O_8$	NMP	5
5	DDQ	NMP	6
6	S	NMP	32
7	S	DMF	51
8	S	toluence	12
9	S	PhCl	10
10	S	DMSO	trace
11	S (0.4 mmol)	DMF	87
12	S (0.6 mmol)	DMF	86
13[c]	S (0.4 mmol)	DMF	86
14[d]	S (0.4 mmol)	DMF	86

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), oxidant (0.2 mmol), solvent (0.6 mL), S (0.2 mmol), 110 °C, under air, 15 h. [b] GC yield. [c] Under N_2. [d] Under O_2.

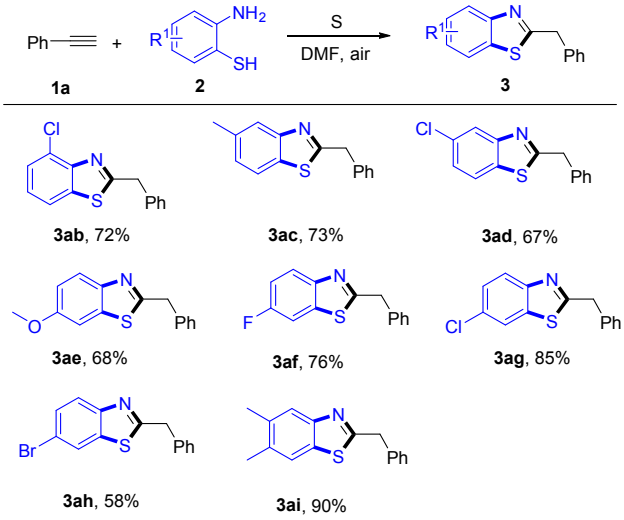
With the optimized reaction conditions in hand, we then investigated the substrate scope of the reaction with respect to various arylacetylenes (Table 2). Slightly lower yields were obtained when alkyl substituents were presented at the C4 position of phenylacetylene especially for those bulky groups (**3ba-3ea**). Halogens such as fluoro, chloro and bromo were compatible to give the corresponding products in high yield (**3ga-3ia**). Interestingly, the desired product **3ja** was obtained in 83% yield when 2-chlorophenylacetylene was used. In addition, 3-substituted phenylacetylene also suitable substrate in this system to give the corresponding product **3ka** in good yield.

Table 2 Substrate scope with respect to various arylacetylenes[a]



[a] Reaction conditions: **1** (0.2 mmol), **2a** (0.4 mmol), solvent (0.6 mL), S (0.4 mmol), 110 °C, under air, 15 h. [b] 6 mmol scale reaction.

Table 3 Substrate scope with various 2-aminobenzenethiols[a]



[a] Reaction conditions: **1a** (0.2 mmol), **2** (0.4 mmol), solvent (0.6 mL), S (0.2 mmol), 110 °C, under air, 15 h.

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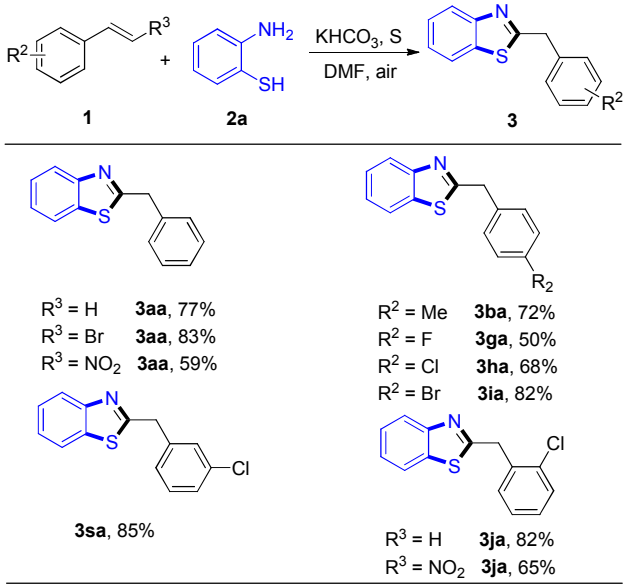
The substrate scope with respect to various substituted 2-amonothiophenols were further evaluated (Table 3). The position of the substituent slightly affected the reaction yield (**3ab**, **3ad** and **3ag**) and the desired product **3ag** could be obtained in 85% yield when 5-chloro-2-amonothiophenol was used. Active bromo substituent was tolerated under the give conditions to give the corresponding product **3ah** in moderate yield. Interestingly, high yield could be achieved when 4,5-dimethyl-2-amonothiophenol was used as the substrate.

To identify the best reaction conditions, styrene (**1m**) and 2-aminobenzenethiol (**2a**) were initially used as the standard substrates under different conditions. To get a better substrate scope for this kind of transformation, we investigated the styrene substrate under the aforementioned optimized conditions (Table 4). However, much lower yield was obtained as comparing with the arylacetylene substrate. We then re-optimized the reaction conditions for styrene substrate and found that the addition of $KHCO_3$ could significantly improve the reaction yield (Table S1 in ESI†). With $KHCO_3$ as the additive and sulfur powder as the oxidant, the desired product **3aa** could be obtained in 77% yield. Interestingly, substituted styrenes such as (*E*)-(2-bromovinyl)benzene and (*E*)-(2-nitrovinyl)benzene also suitable coupling partners to give the same product in 83% and 59% yield, respectively. Substituent at the phenyl ring of styrene significantly affected the reaction yield. Good yield was obtained when a methyl group presented at the *para* position (**3ba**). Functional groups such as fluoro, chloro and bromo were well tolerated. Interestingly, **3ia** could be obtained in 82% yield although a reactive bromo group is existed. In addition, position of substituent has profoundly affect the reaction yield and better yields were obtained when the chloro group was located at the *meta* and *ortho* position (**3sa** and **3ja**).

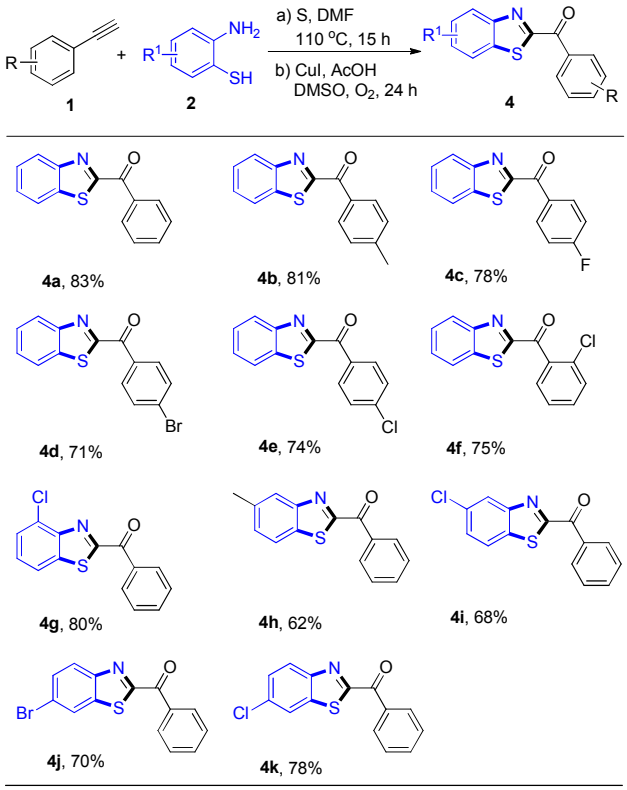
Table 4 Substrate scope with respect various styrenes [a]



[a] Reaction conditions: **1** (0.2 mmol), **2a** (0.4 mmol), $KHCO_3$ (0.4 mmol), solvent (0.6 mL), S (0.6 mmol), 110 °C, under air, 15 h.

After we have realized 2-alkylbenzothiazole synthesis using elemental sulfur as the oxidant, we turned our attention to 2-acylbenzothiazole formation from phenylacetylene (**1a**) and 2-aminothiophenol (**2a**) via "one-pot, two-step" process (Table 5). After systematic optimization of the reaction conditions, we got the optimized conditions for 2-acylbenzothiazole: phenylacetylene reacted with 2-aminothiophenol at 110 °C for 15 h using sulfur as the oxidant then CuI and AcOH/DMSO were added to the reaction and the resulted mixture was further stirred for 24 h. For the model reaction, **4a** was isolated in 83% yield. Various 4-substituented phenylacetylenes could smoothly reacted with **2a** to give the corresponding products in good yields (**4b**-**4e**). Halogens such as fluoro, chloro and bromo were compatible at the given conditions. The position of substituent slightly affected the reaction yield (**4e**-**4g, 4i**, **4k**) and the desired product **4g** was obtained in 80% yield when 3-chloro-2-amonothiophenol was used as the substrate. Unfortunately, aliphatic alkynes and alkenes were not suitable substrates for this kind of reaction.

Table 5 Substrate scope with respect to various arylacetylenes and 2-aminobenzenethiols[a]



[a] Reaction conditions: **1** (0.2 mmol), **2** (0.4 mmol), S (0.4 mmol), 110 °C, CuI (20 mol%), AcOH (0.4 mmol), DMF (0.5 mL), DMSO (0.5 mL).

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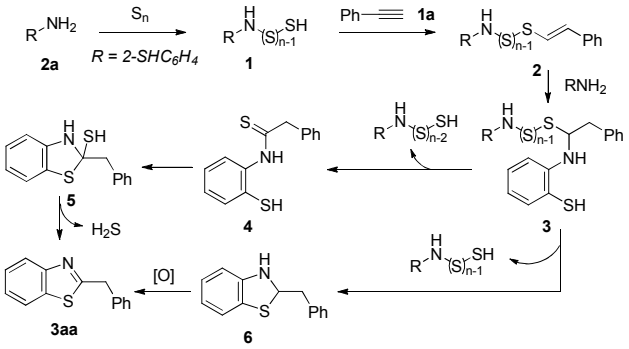

Scheme 2 Possible reaction pathway.

Although the exact reaction mechanism is unknown at this stage, based on the experimental results and relative literatures,[17] we proposed a possible reaction pathway to illustrate this transformation. Reaction of 2-aminothiophenol (**2a**) with elemental sulfur generates an intermediate **1**.[11a, 11b, 12c] Addition reaction of **1** with phenylacetylene (**1a**) affords an alkene intermediate **2**, which can further react with another equiv. of **2a** to give compound **3**. A thioamide intermediate **4** is generated from **3** via S-S bond cleavage process and cyclization of **4** provides an intermediate **5**, which can give the final product **3aa** via extrusion H_2S. Meanwhile, there is another possible reaction pathway for conversion of **3** into the desired product **3aa**. 2-Benzyl-2,3-dihydrobenzo[d]thiazole (**6**) can be produced from **3** via S-S bond cleavage and cyclization process. This compound can convert into the final product **3aa** under oxidative conditions.

In summary, we have developed a novel approach for the synthesis of 2-substituted benzothiazoles from 2-aminobenzenethiols and arylacetylenes or styrenes under metal-free conditions. In this transformation, elemental sulfur was used as the efficient oxidant to promote the reaction under mild conditions. This method affords a simple approach for benzothiazoles under metal-free conditions. The mechanism and the further synthetic applications of this reaction are in progress in our laboratory.

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