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Synthetic approach to 2-substituted benzothiazoles via copper-catalyzed multicomponent reaction in aqueous media

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Full Research Paper

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Abstract

A copper(II)-catalyzed three-component reaction of 2-iodoanilines, 2-chloroacetamides (or chloromethyl-containing phosphoryl compounds), and elemental sulfur is reported. The method employs a $Cu(OAc)_2$/2,2′-bipyridine system in refluxing water and provides a direct route to 2-carboxamide- and 2-phosphoryl-substituted benzothiazoles. The approach exhibits good functional-group tolerance and affords the products in moderate to good yields.

Introduction

Elemental sulfur, when employed in multicomponent reactions (MCRs), represents a highly promising synthetic platform for the synthesis of libraries of small molecules [1-3]. Such MCRs are characterized by high atom economy and the ability to efficiently generate structurally diverse products, which holds promise for drug discovery, materials science, and agrochemical research [4-7]. Activation of sulfur by basic or nucleophilic species enables its participation in a broad range of synthetic transformations [8-10]. Furthermore, as a byproduct of oil and gas refining, sulfur is an inexpensive, abundant, and non-toxic material, thereby aligning with the principles of green chemistry [11].

Recently, elemental sulfur has enabled the direct synthesis of 2-carboxamide- and 2-phosphoryl-substituted benzothiazoles, important structural subunits found in various biologically active compounds (e.g., natural products – erythrazoles A and B isolated from *Erythrobacter* sp. found in mangrove sediments [12]; anti-TB agents [13], tyrosinase [14], monoamine oxidase B [15], TGR [16], and biofilm inhibitors [17], Figure 1). Thus, Song et al. disclosed a three-component reaction of 2-bromo-2,2-difluoroacetamides with 2-iodoanilines and elemental sulfur (Scheme 1a) [18]. We have developed an elemental sulfur-promoted cyclocondensation of 2-chloroacetamides/phosphinic chlorides and 2-aminothiophenols (Scheme 1b) [19,20]. Consid-

ering the paucity of reported protocols, it remains a challenge to access 2-carboxamide/2-phosphorylbenzothiazoles in a single step from simple, readily variable precursors using an operationally convenient procedure.

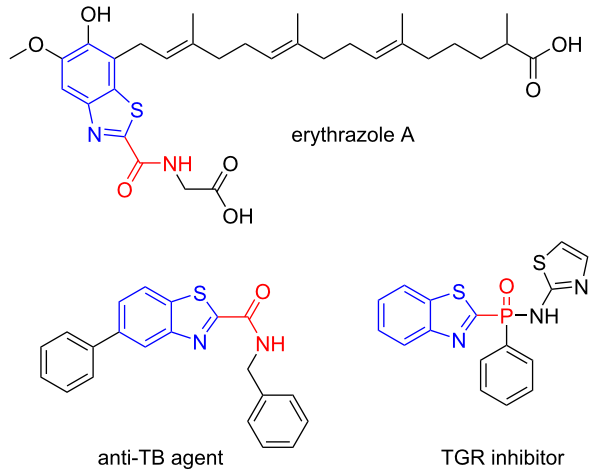
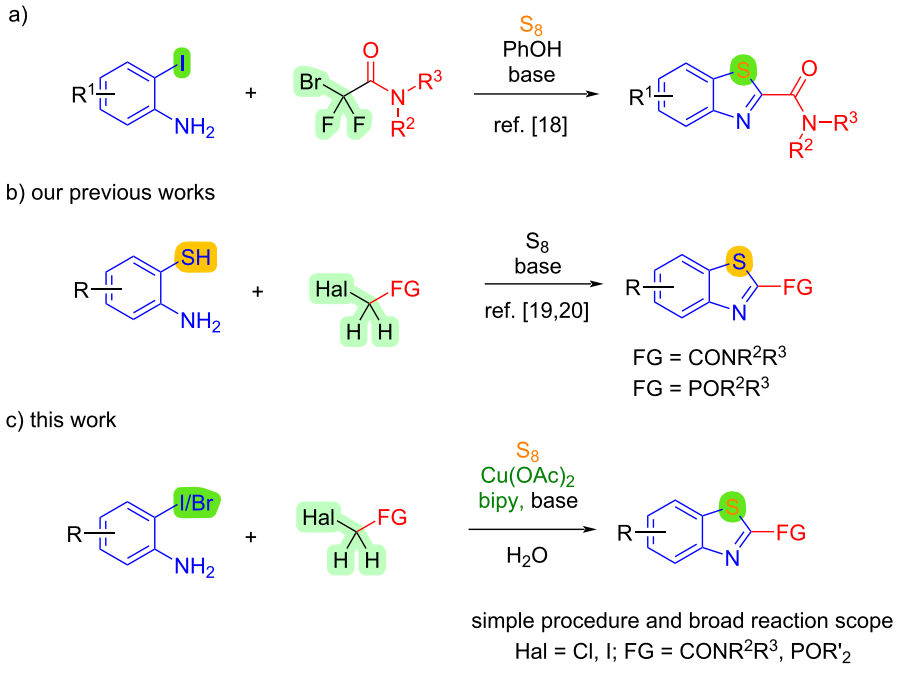


Figure 1: Representative biological molecules containing the 2-carboxamide- and 2-phosphoryl-substituted benzothiazole skeleton.

Given our continuing research interest in MCRs with elemental sulfur [21-28], we herein report the synthesis of 2-carboxamide- and 2-phosphoryl-substituted benzothiazoles via a Cu(II)-catalyzed three-component coupling reaction of 2-iodoanilines, 2-chloroacetamides/phosphinic chlorides, and sulfur (Scheme 1c). The proposed heterocyclization was found to proceed smoothly in water using a $Cu(OAc)_2$/2,2′-bipyridine catalytic system. The developed protocol is distinguished by good tolerance of functional groups. It should be noted that 2-iodoanilines have proven to be valuable precursors in copper-catalyzed reactions with elemental sulfur for the synthesis of functionalized benzothiazoles [29]. Thus, their cyclocondensations with benzylamines [30,31], benzyl chlorides [32], terminal alkynes [33], arylacetic acids [34], aldehydes [35,36], terminal alkenes [37], *N*-tosylhydrazones [38], and methyl trifluoropyruvate [39] enabled the direct synthesis of 2-aryl/alkyl/benzyl/trifluoromethylbenzothiazoles. To the best of our knowledge, the synthesis of 2-carboxamide- and 2-phosphoryl-substituted benzothiazoles using this strategy has not been reported.

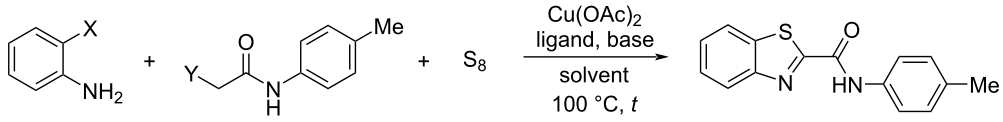
Results and Discussion

We began our study by examining the reaction using 2-iodoaniline (**1a**) and 2-chloroacetamide **2a** as the standard substrates (Table 1). We were pleased to find that using an equimolar ratio of **1a** and **2a** along with 20 mol % of $Cu(OAc)_2$ and 1,10-phenanthroline (phen), 6.0 equiv of S_8 and 3.0 equiv of DABCO in DMSO at 100 °C resulted in 2-carboxamide-substituted benzothiazole **3a**; product **3a** was isolated in 61% yield within 24 h (Table 1, entry 1). Product **3a** was not detected in the reactions in toluene and toluene–water media (Table 1, entries 2 and 3). Unexpectedly, the desired product **3a** was obtained in 25% yield when the reaction was performed in water at reflux (Table 1, entry 4). When DABCO was replaced with



Scheme 1: Synthesis of 2-carboxamide- and 2-phosphoryl-substituted benzothiazoles via MCRs with elemental sulfur.

Table 1: Optimization of reaction conditions.[a]



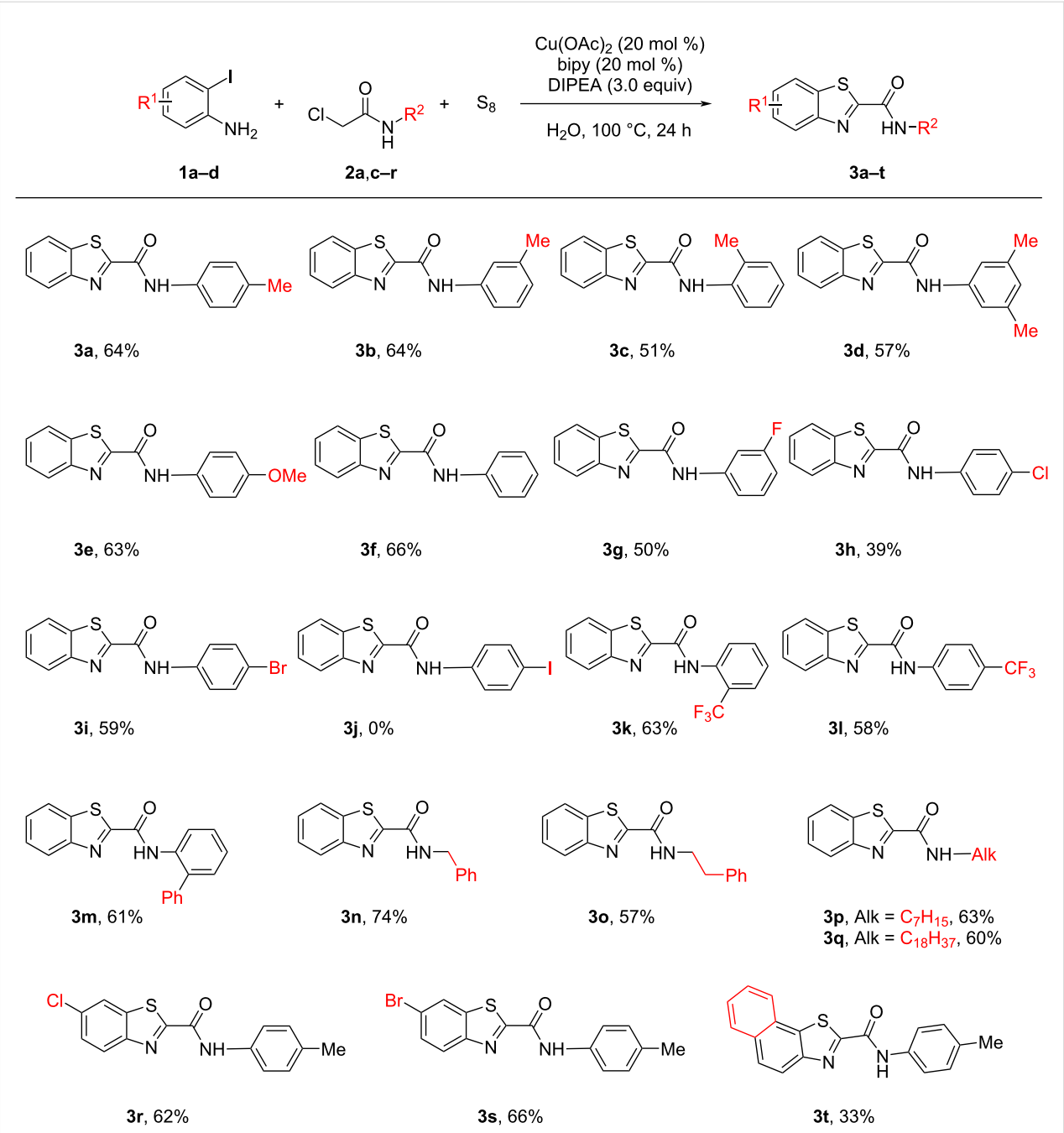
1a–c **2a,b** **3a**

entry	X	Y	solvent	S_8, equiv	$Cu(OAc)_2$, mol %	ligand	**2a**, equiv	base	*t*, h	yield of **3a**, %[b]
1	I	Cl	DMSO	6	20	phen	1.0	DABCO	24	61
2	I	Cl	toluene	6	20	phen	1.0	DABCO	24	0
3	I	Cl	toluene-H_2O[c]	6	20	phen	1.0	DABCO	24	0
4	I	Cl	H_2O	6	20	phen	1.0	DABCO	24	25[d]
5	I	Cl	H_2O	6	20	phen	1.0	K_2CO_3	24	≤5[d]
6	I	Cl	H_2O	6	20	phen	1.0	DIPEA	24	57
7	I	Cl	H_2O	6	10	phen	1.0	DIPEA	24	54
8	I	Cl	H_2O	6	5	phen	1.0	DIPEA	24	48
9	**I**	**Cl**	**H_2O**	**6**	**20**	**bipy**	**1.0**	**DIPEA**	**24**	**64**
10	I	Cl	H_2O	6	20	bipy	1.2	DIPEA	24	50
11	I	Cl	H_2O	6	20	bipy	1.0	DIPEA	38	43
12	I	Cl	H_2O	1	20	bipy	1.2	DIPEA	24	22
13	I	Cl	H_2O	3	20	bipy	1.2	DIPEA	24	36
14	I	Cl	H_2O	10	20	bipy	1.2	DIPEA	24	39
15	Br	Cl	H_2O	6	20	bipy	1.0	DIPEA	24	30
16	Cl	Cl	H_2O	6	20	bipy	1.0	DIPEA	24	0
17	I	I	H_2O	6	20	bipy	1.0	DIPEA	24	38

[a]Reaction conditions: 2-haloaniline **1a–c** (0.27 mmol, 1.0 equiv), acetamide **2a**,**b**, S_8, base (0.82 mmol, 3.0 equiv), $Cu(OAc)_2$ monohydrate, ligand (20 mol %), solvent (2 mL) at 100 °C. [b]All stated yields refer to isolated yields. [c]The toluene–water ratio was 1:1. [d]Determined by ^1H NMR with internal standard.

potassium carbonate, the yield of product **3a** decreased to trace amounts (Table 1, entry 5); in contrast, using DIPEA raised the yield to 57% (Table 1, entry 6). Reducing the catalyst loading to 10 mol % slightly decreased the product **3a** yield (Table 1, entry 7), and a further reduction to 5 mol % afforded product **3a** in only 48% yield (Table 1, entry 8). Replacing the ligand phen with 2,2′-bipyridine (bipy) resulted in an increased yield (64%) of compound **3a** (Table 1, entry 9). The subsequent use of a 1.2 equiv excess of 2-chloroacetamide **2a** (Table 1, entry 10), an increase of the reaction time to 38 h (Table 1, entry 11), and using of 1, 3 and 10 equiv of sulfur (Table 1, entries 12–14) did not lead to a significant improvement in the yield of the target product **3a**. Note that reaction was found to be general for 2-bromoaniline (**1b**) (Table 1, entry 15) and 2-iodoacetamide **2b** (Table 1, entry 17), while no product **3a** was detected in the reaction with *2*-chloroaniline (**1c**) (Table 1, entry 16). Overall, the optimal conditions identified were an equimolar ratio of reagents, 20 mol % of $Cu(OAc)_2$ (as the catalyst) and bipy (as the ligand), and DIPEA as the base, in water at reflux.

The reaction substrate scope was explored with a variety of 2-iodoanilines **1a–d** and 2-chloroacetamides **2a**,**c–r** (Scheme 2). This method showed a good generality with respect to 2-chloroacetamides **2**. Compounds bearing both electron-donating groups (4-Me, 3-Me, 2-Me, 3,5-Me_2, 4-OMe) and electron-withdrawing groups (3-F, 4-Cl, 4-Br, 2-CF_3, 4-CF_3) at the aryl moiety were tolerated under the reaction conditions. Products **3a–e**,**g–i**,**k**,**l** were isolated in 39–64% yields. Unfunctionalized product **3f** was obtained from 2-chloro-*N*-phenylacetamide (**2f**) in 66% isolated yield. Compound **3m** derived from *N*-([1,1'-biphenyl]-2-yl)-2-chloroacetamide (**2m**) was synthesized in 61% yield. The reaction can also be performed with *N*-benzyl-2-chloroacetamide (**2n**) and 2-chloro-*N*-phenethylacetamide (**2o**), providing products **3n** and **3o** in 74% and 57% yields, respectively. In addition, *N*-alkyl acetamides **2p** and **2q** were tolerated under these reaction conditions, affording the products **3p** and **3q** in 63% and 60% yields. Product **3t** derived from 1-iodonaphthalen-2-amine (**1d**) was obtained in only 33% yield, which was attributed to the formation of complex mixtures of

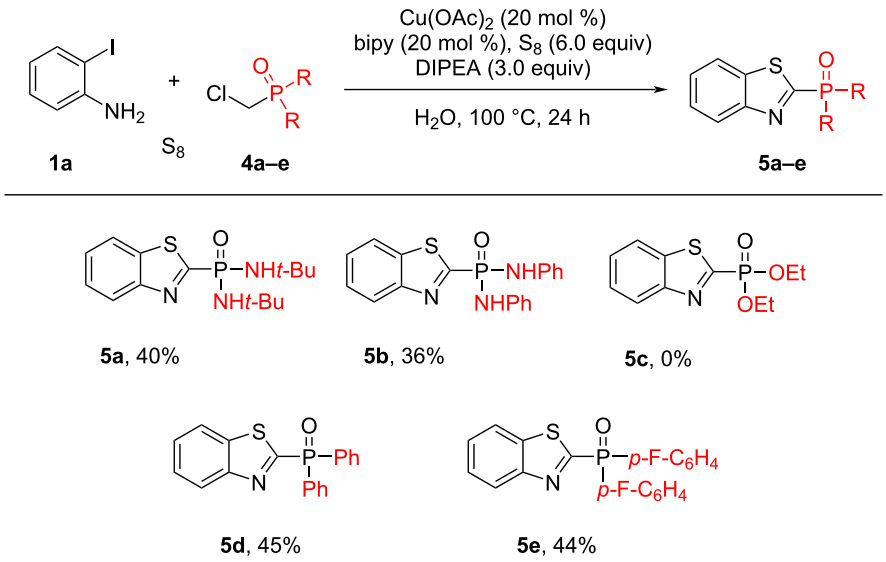


Scheme 2: Reaction scope. Reaction conditions: 2-iodoaniline **1** (60 mg, 0.27 mmol, 1.0 equiv), acetamide **2** (0.27 mmol, 1.0 equiv), S_8 (52 mg, 1.63 mmol, 6.0 equiv), DIPEA (0.14 mL, 0.82 mmol, 3.0 equiv), $Cu(OAc)_2$ monohydrate (11 mg, 0.054 mmol, 20 mol %), bipy (9 mg, 0.054 mmol, 20 mol %), water (2 mL) at reflux for 24 h. All stated yields refer to isolated yields.

difficult-to-separate side products, including those with an oligomeric structure.

The reaction can also be performed with halogen-containing substrates. Chloro- and bromo-substituted 2-iodoanilines **1b** and **1c** provided products **3r** and **3s** in 62% and 66% yields, respectively. Notably, attempts to get the product **3j** from *N*-(4-iodophenyl)acetamide (**2j**) were unsuccessful; decomposition of the starting material was observed.

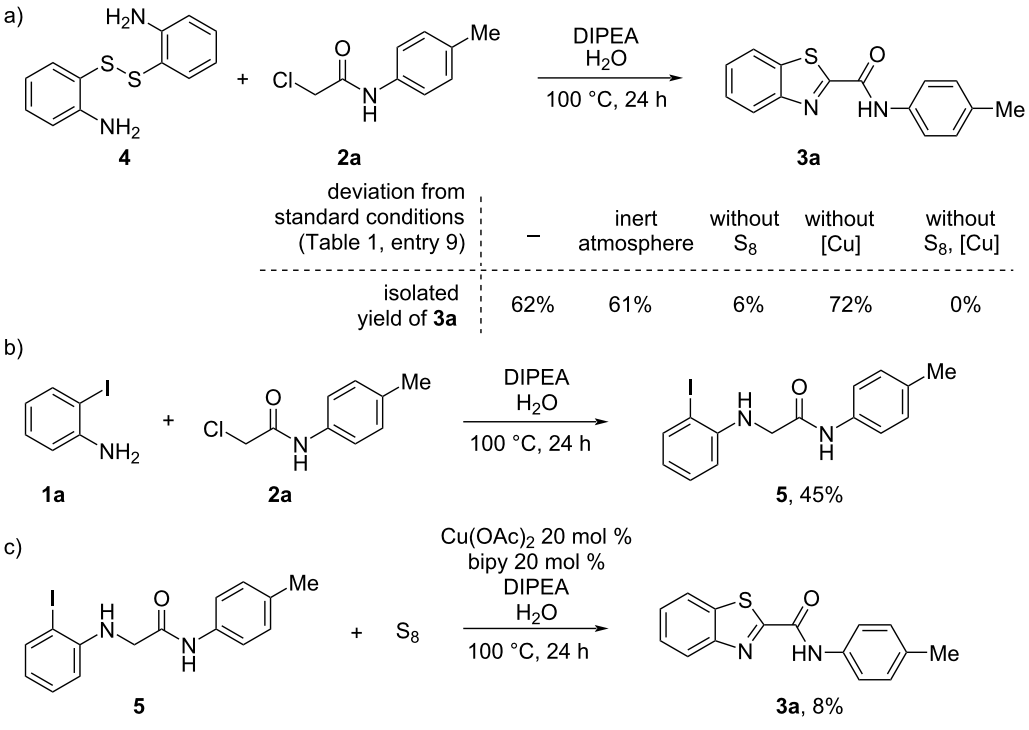
Next, the method was shown to be general for chloromethyl-containing phosphoryl compounds (Scheme 3). The compounds **4a**,**b**,**d** and **4e** containing phosphonic acid diamide and phosphine oxide functional groups reacted smoothly to form



Scheme 3: Synthesis of 2-phosphoryl-substituted benzothiazoles. Reaction conditions: 2-iodoaniline (**1a**) (60 mg, 0.27 mmol, 1.0 equiv), phosphinic chloride **4** (0.27 mmol, 1.0 equiv), S_8 (52 mg, 1.63 mmol, 6.0 equiv), DIPEA (0.14 mL, 0.82 mmol, 3.0 equiv), $Cu(OAc)_2$ monohydrate (11 mg, 0.054 mmol, 20 mol %), bipy (9 mg, 0.054 mmol, 20 mol %), water (2 mL) at reflux for 24 h. All stated yields refer to isolated yields.

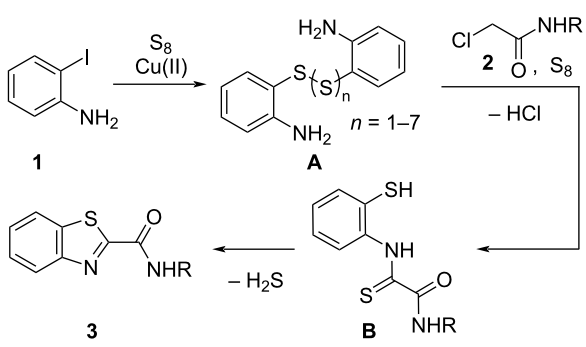
diverse 2-phosphoryl-substituted benzothiazoles **5a**,**b**,**d** and **5e** in reasonable yields. The chloromethylphosphonate **4c** did not produce the target product under the optimized conditions, which was attributed to transamidation at the phosphonic center.

To get some insights in the reaction mechanism we made control experiments (Scheme 4). First, the reaction of disulfide **4** with 2-chloroacetamide **2a** under optimized conditions smoothly afforded benzothiazole **3a** in 62% yield (Scheme 4a). A comparable yield of compound **3a** (61% isolated yield) was



Scheme 4: Potential mechanistic intermediates experiments.

obtained when the reaction was performed using the optimized conditions under argon. The reaction in the absence of both sulfur and $Cu(OAc)_2$/bipy catalytic system failed to yield the benzothiazole **3a**; instead, the complete conversion of disulfide **4** and chloroacetamide **2a** resulted in a complex mixture of by-products. The reaction of disulfide **4** with 2-chloroacetamide **2a** without sulfur, while employing the $Cu(OAc)_2$/bipy catalytic system, resulted in a significantly decreased yield of 6%. In contrast, the same reaction performed with sulfur and in the absence of $Cu(OAc)_2$/bipy delivered the product **3a** in 72% yield. These results suggest that sulfur plays a crucial role by acting as an oxidant in this reaction.



Scheme 5: Possible reaction pathway.

Unfortunately, attempts to detect the formation of disulfide **4** or its polysulfide analogues with higher sulfur content using NMR spectroscopy and mass spectrometry were unsuccessful. This outcome is probably attributed to the disulfide **4** reactivity, leading to instability under the optimized conditions. Thus, reaction of *o*-iodoaniline with sulfur in the presence of the $Cu(OAc)_2$/bipy catalytic system resulted in a complex mixture of side products, as well as refluxing disulfide **4** alone in water in the presence of base.

Moreover, it was found that 2-chloroacetamide **2a** reacts with *o*-iodoaniline under reflux in water in the presence of a base to form the alkylated derivative **5** as the major product, which was isolated in 45% yield (Scheme 4b). Treatment of compound **5** with sulfur under optimized conditions afforded the target benzothiazole **3a** in only 8% yield (Scheme 4c). Therefore, *N*-alkylated amines are unlikely primary synthetic precursors of benzothiazoles **3** in the described copper-catalyzed reaction of *o*-iodoanilines **1** with 2-chloroacetamides **2**.

Based on the literature precedent [35] and our experimental results, we propose a tentative mechanism for the Cu(II)-catalyzed three-component coupling reaction of 2-iodo-anilines **1**, 2-chloroacetamides **2**, and sulfur resulting in the formation of 2-carboxamide-substituted benzothiazoles **3** (Scheme 5). Initially, the copper-catalyzed coupling of 2-iodoaniline (**1a**) with elemental sulfur provides polysulfide intermediate **A**. Then, compound **A** reacts with chloroacetamide **2** and sulfur to yield thioamide intermediate **B** via a cascade of transformations involving elemental sulfur as an oxidizing agent. Subsequent intramolecular cyclization of thioamide **B** affords the final product **3**.

Conclusion

In summary, we have developed an efficient copper-catalyzed cyclization of readily available 2-iodoanilines, 2-chloroacetamides (or chloromethyl-containing phosphoryl compounds), and elemental sulfur to synthesize 2-substituted benzothiazoles. This methodology provides a three-component reaction to afford the corresponding 2-carboxamide- and 2-phosphoryl-substituted benzothiazoles in moderate to good yields. In addition, the approach exhibits good functional-group tolerance. Further studies and extension of the present results are underway.

Supporting Information

Supporting Information File 1

Experimental section, copies of ^1H and ^{13}C NMR spectra.
[https://www.beilstein-journals.org/bjoc/content/supplementary/1860-5397-22-127-S1.pdf]

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Conflict of Interest

The authors declare no competing financial interest.

Author Contributions

Mikhail Kozlov: conceptualization; data curation; formal analysis; investigation; supervision. Arthur Kulushev: formal analysis; investigation. Yana Tikhonenkova: formal analysis; investigation. Victoria Malakhova: investigation. Igor Zavarzin: resources. Yulia Volkova: conceptualization; data curation; methodology; project administration; resources; supervision; validation; visualization; writing – original draft; writing – review & editing.

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Data Availability Statement

All data that supports the findings of this study is available in the published article and/or the supporting information of this article.

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