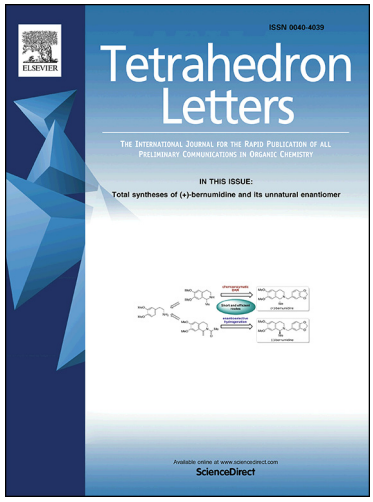

Copper-catalyzed cross-coupling of chloramine salts and arylboronic acids in water: a green and practical route to *N*-arylsulfonamides

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Copper-catalyzed cross-coupling of chloramine salts and arylboronic acids in water: a green and practical route to *N*-arylsulfonamides

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ABSTRACT

A green and practical method for the synthesis of *N*-arylsulfonamides from chloramine salts and arylboronic acids is herein developed. The reaction proceeds readily in the presence of 5 mol% of CuI and 2.5 equiv. K_2CO_3 in water at room temperature, generating a variety of *N*-arylsulfonamides in moderate to good yields with good functional group tolerance.

Introduction

Chloramine salts have been recognized as an important source for *N*-building blocks due to its ready commercial availability. Significantly, we found chloramine salts have highly attractive practical characteristics: easy and amenable preparation on large scales, nontoxic byproducts, high stability to air and heat, and excellent reactivity as a sulfonamidation reagent.[1] By using chloramine salts, chlorosulfonamidation, aminohydroxylation, or aziridination of olefins, sulfonamidation of relatively highly reactive alkanes, and decarboxylative sulfonamidation of carboxylic acids can be performed to give chlorosulfonamidated products,[2] *β*-sulfonamido alcohols,[3] aziridines[4] and sulfonamidated compounds,[5] respectively. In these procedures, nitrene species derived from chloramine salts by metal- or nonmetal-assisted removal of sodium chloride are generally regarded as intermediates. In 2011, Chandrasekaran and co-workers found that chloramine salts can behave as a dual nucleophile when its chlorine atom is removed in the form of BrCl.[6] Recently, it was further found that chloramine salts can react with arylboronic acids in the present of a copper catalyst to generate *N*-arylsulfonamides. These results indicated that chloramine salts can also serve as an electrophile to undergo oxidative addition at the N-Cl bond .[7]

N-Arylsulfonamide is a kind of common structural motif in pharmaceutical agents and agrochemicals.[8] Traditionally, *N*-arylsulfonamides were prepared by nucleophilic substitution of sulfonyl chlorides with aromatic amines. However, this classical approach requires the use of mutagenic sulfonyl chlorides and genotoxic aryl amines. As an alternative, the metal-catalyzed *N*-arylation of sulfonamides with aromatic halides allows the synthesis of *N*-arylsulfonamides free of the use of sulfonyl chloride, but these reactions generally require harsh reaction conditions due to the low nucleophilicity of sulfonamides (Scheme 1-a).[9] To avoid harsh reaction conditions, Chan and Lam etc. developed the copper-catalyzed cross-coupling of sulfonamides with arylboronic acids, which generally go through under relatively mild reaction conditions with short reaction time,[10] but the Chan–Lam coupling reaction typically requires a stoichiometric amount of copper salt and suffers from the limited substrate scope (Scheme 1-b). Although significant progress has been achieved in the transition metal-catalyzed direct sulfonamidation of C-H bonds using sulfonyl azides as the amine source recently, these reactions involve precious Rh, Ir or Ru catalysts and toxic organic solvents (Scheme 1-c).[11] In 2014, Kim and co-workers developed a copper-catalyzed cross-coupling between arylboronic acids and sulfonyl azides to give *N*-arylsulfonamides in the absence of ligands and bases.[12] This reaction proceeds efficiently under mild conditions

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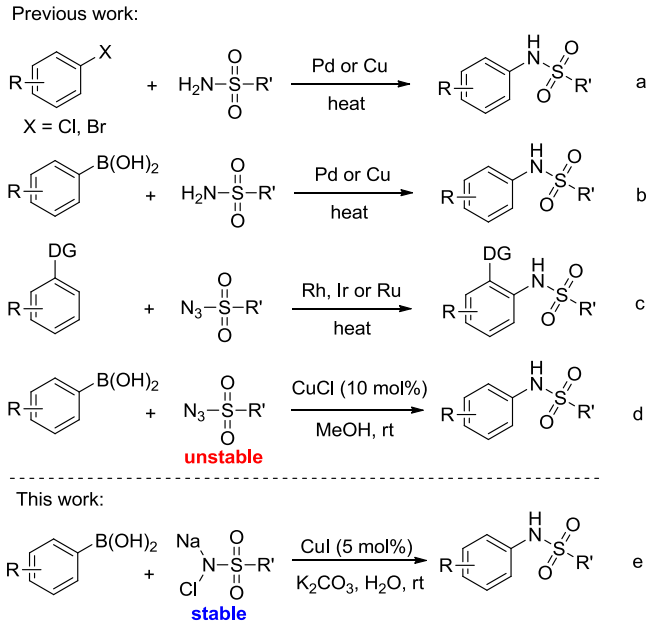


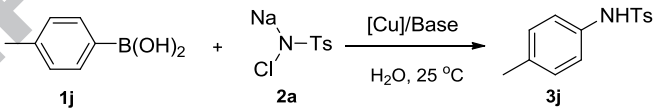
Scheme 1 Selected examples for synthesis of *N*-arylsulfonamides

with a broad functional groups tolerance, but sulfonyl azides are not shelf stable and require precautions in handling (Scheme 1-d). Very recently, we described a copper catalyzed cross-coupling between arylboronic acids and chloramine salts with low catalyst loading at room temperature, but this procedure suffers from some disadvantages such as the use of an organic solvent and a strong base.[7a] Compared to sulfonyl azides, chloramine salts are stable and readily accessible. Therefore, we reason that it will be advantageous that chloramine salts serving as an alternative to sulfonyl azides react with arylboronic acids to give *N*-arylsulfonamides. In this work, we describe an improved approach to furnish *N*-arylsulfonamides from arylboronic acids and chloramine salts with weak base in water at room temperature (Scheme 1-e).

Results and discussions

Initially, the reaction of *p*-tolylboronic acid (**1j**) and chloramine T (**2a**) was employed as the model reaction for condition optimization. To our delight, the desired *N*-arylsulfonamide **3j** was obtained when the model reaction was treated with 5 mol% $Cu(OAc)_2$ and 1.3 equiv. K_2CO_3 with H_2O as solvent at room temperature (Table 1, entry 1). Encouraged by this positive result, further optimization of the reaction conditions was carried out. The results were presented in Table 1. For copper catalysts, the results demonstrated that CuI is the optimal catalyst for this transformation (Table 1, entries 1-9). When precious $Pd(OAc)_2$ was used as the catalyst, a homocoupling product of *p*-tolylboronic acid was obtained in 65% yield (Table 1, entry 10), whereas no desired product were observed. In the absence of a metal catalyst, a complex mixture was obtained with very low conversion (Table 1, entry 11). Subsequently, various bases were explored. The results revealed that K_2CO_3 was the best base, producing the desired *N*-arylsulfonamide **3j** in 63% isolated yield (Table 1, entry 7), while other bases including $AcONa{\cdot}3H_2O$, NaOH, KOH and Na_2CO_3 did not give better yields (Table 1 entries 12-15). Interestingly, increasing the catalyst loading from 5 mol% to 10 mol% did not show any improvement in the reaction efficiency (Table 1, entry 16). Gratifyingly, when the amount of K_2CO_3 was increased to 2.5 equiv., the yield reached 85% (Table 1, entry 20). However, further increase of the amount of base did not improve the isolated yield (Table 1, entry 21). Finally, when 4-methylphenylboronic acid pinacol ester was used instead of 4-methylphenylboronic acid (**1j**) to react with chloramine T, *N*-arylsulfonamide **3j** was isolated only in 55% yield.

Table 1 Optimization of reaction conditions[a]



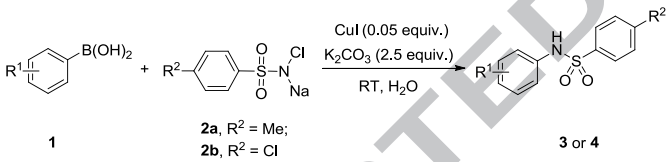
Entry	Catalyst	Base	Yield%[b]
1	$Cu(OAc)_2$	K_2CO_3	60
2	$Cu(OTf)_2$	K_2CO_3	44
3	$CuCl_2$	K_2CO_3	45
4	CuCl	K_2CO_3	47
5	$CuBr_2$	K_2CO_3	60
6	CuBr	K_2CO_3	58
7	CuI	K_2CO_3	63
8	$CuSO_4{\cdot}5H_2O$	K_2CO_3	56
9	CuTC	K_2CO_3	59
10	$Pd(OAc)_2$	K_2CO_3	0
11	/	K_2CO_3	0

12	CuI	$AcONa{\cdot}3H_2O$	32
13	CuI	NaOH	49
14	CuI	KOH	51
15	CuI	Na_2CO_3	54
16[c]	CuI	K_2CO_3	63
17[d]	CuI	K_2CO_3	53
18[e]	CuI	K_2CO_3	69
19[f]	CuI	K_2CO_3	77
20[g]	**CuI**	**K_2CO_3**	**85**
21[h]	CuI	K_2CO_3	85
22[i]	CuI	K_2CO_3	55

[a] Reaction condition: 0.3 mmol TsNNaCl, 1.2 equiv 4-Me-PhB$(OH)_2$, 1.3 equiv base, 0.05 equiv catalyst, 1.5 mL H_2O, 25 °C; 18 hrs; [b] Isolated yield; [c] 0.1 equiv CuI was used; [d] 1.0 equiv K_2CO_3 was used; [e] 1.5 equiv K_2CO_3 was used; [f] 2.0 equiv K_2CO_3 was used; [g] 2.5 equiv K_2CO_3 was used; [h] 3.0 equiv K_2CO_3 was used, [i] 4-methylphenylboronic acid pinacol ester was used instead of 4-Me-PhB$(OH)_2$.

With the optimized conditions in hand (5 mol% CuI and 2.5 equiv. K_2CO_3 in H_2O at room temperature), we then examined the scope and generality of the reaction with respect to the arylboronic acid. As shown in Table 2, a broad range of arylboronic acids **1** with electron-donating or -withdrawing substituents in the *para* or *meta* position of the aryl ring are compatible with this transformation, delivering the corresponding *N*-arylsulfonamides in moderate to good yields. However, this reaction does not seem to work with *ortho*-substituted arylboronic acids presumably due to the steric hindrance. For example, the reaction of (4-fluorophenyl)boronic acid (**1b)** and (3-fluorophenyl)boronic acid (**1c)** with chloramine T (**2a**) offered corresponding *N*-arylsulfonamides **3b** and **3c** in 76% and 67%

Table 2 Substrate scope.[a]

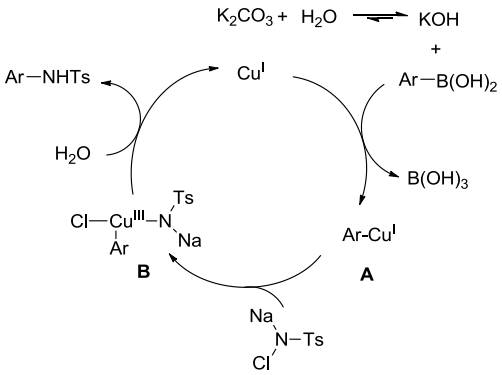


Entry	R^1	R^2	Product	yield (%)[b]
1	H	Me	**3a**	69
2	4-F	Me	**3b**	76
3	3-F	Me	**3c**	67
4[c]	2-F	Me	**3d**	15
5	4-CF_3	Me	**3e**	30
6	4-Cl	Me	**3f**	65
7	3-Cl	Me	**3g**	51
8	3,5-Cl	Me	**3h**	49
9	4-Br	Me	**3i**	54
10	3-CH_2OH	Me	**3j**	61
11	3-CHO	Me	**3k**	52
12[d]	4-COOMe	Me	**3l**	38
13[e, f]	4-COOH	Me	**3m**	41
14	4-Me	Me	**3n**	85

15	3-Me	Me	**3o**	74
16	4-OMe	Me	**3p**	53
17	4-OH	Me	**3q**	0
13	H	Cl	**4a**	75
14	4-Me	Cl	**4b**	71
15	3-Me	Cl	**4c**	66
16	4-F	Cl	**4d**	65
17	3-F	Cl	**4e**	64
18	4-Cl	Cl	**4f**	57
19	4-Br	Cl	**4g**	59

[a] Reaction condition: 0.3 mmol TsNNaCl, 1.2 equiv. arylboronic acids, 2.5 equiv. K_2CO_3, 0.05 equiv. CuI, 1.5 mL H_2O, 18 hrs; [b] Isolated yield; [c] *p*-Toluenesulfonamide was isolated in 55% yield; [d] Methyl 4-hydroxybenzoate was obtained in 43% yield; [e] 4-Hydroxybenzoic acid was isolated in 36% yield; [f] 3.5 equiv K_2CO_3 was used.

yields, respectively, whereas the use of a *ortho*-substituted arylboronic acid such as (2-fluorophenyl)boronic acid (**1d**) afforded *p*-toluenesulfonamide instead of the expected *N*-arylsulfonamide as the major product, presumably by a Cu-catalyzed reducing reaction of chloramine T. When a trifluoromethyl-substituted arylboronic acid was employed, it showed less reactivity and provided a relatively lower yield. Moreover, chloro-substituted arylboronic acids were tolerated, providing the corresponding products **3f**-**3h** in moderate yields. Particularly, a bromo substituent survived the reaction, which is valuable for further transformations. The reaction of (4-bromophenyl)boronic acid with chloramine T (**2a**) produced *N*-arylsulfonamide **3i** in 54% yield. Furthermore, alcohol and formyl group were also tolerated. (3-(Hydroxymethyl)phenyl)boronic acid (**1j**) and (3-formylphenyl)boronic acid (**1k**) reacted with chloramine T smoothly affording the corresponding *N*-arylsulfonamides in 61% and 52% yield respectively. Unexpectedly, carbomethoxy- and carboxyl-substituted arylboronic acids **1l** and **1m** showed low efficiency in this reaction due to the oxidative hydroxylation.



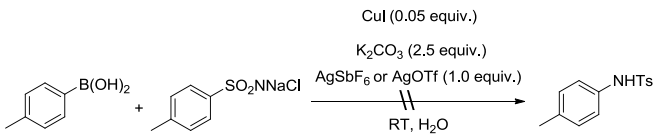
Scheme 3 Proposed reaction pathway

Electron-donating group-attached arylboronic acids were also efficient for the reactions with chloramine T (**2a**) to produce the corresponding *N*-arylsulfonamides **3n**-**3p**. For example, the reactions of methyl-substituted arylboronic acid **1n** and **1o** gave the *N*-arysulfonamide **3n** in 85% yield and **3o** in 74% yield, respectively. In addition, (4-methoxyphenyl)boronic acid (**1p**) was also suitable for this reaction, affording the corresponding *N*-arylsulfonamides **3p** in 53% yield. Unfortunately, free phenol moiety was not tolerated due to its high reducibility. The reaction of (4-hydroxyphenyl)boronic acid (**1q**) with chloramine T gave a complex mixture, and no desired product was observed.

To further explore the potential of our methodology, chloramine salt **2b** was also tested for the synthesis of *N*-arylsulfonamide **4a**-**4g**. The results indicated chloramine salt **2b** was also normally efficient in the reaction of arylboronic acids, giving the desired *N*-arylsulfonamides in moderate to good yields. However, alkyl chloramine salts were not compatible with this transformation.

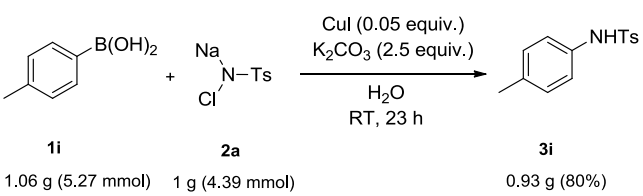
We then tried to gain some insights into the mechanism of this sulfonamidation reaction. Provided that the key step of the reaction involves a copper-nitrene complex, the reaction would be promoted by silver ion, which facilitates the expulsion of the chloridion in the form of AgCl. To test this assumption, $AgSbF_6$ and AgOTf was added to the standard reaction respectively. The result showed that a complex reaction mixture was obtained instead of the desired product. (Scheme 2) This result indicated that the transformation should not proceed via a copper-nitrene intermediate.

Based on above data and previous studies,[7c, 13] a plausible mechanism is illustrated in Scheme 3. Initially, transmetalation of the aryl group of the arylboronic acid with copper(I) under basic condition gives rise to complex **A**, followed by oxidative addition of N-Cl bond of chloramine T (**2a**) at the Cu(I) center to afford complex **B**. Finally, the C-N reductive elimination of complex **B** delivers the desired *N*-arylsulfonamides and regenerates the copper(I) catalyst.



Scheme 2 Preliminary mechanistic studies

To demonstrate the potential synthetic application of the present method, the sulfonamidation of **1j** on a gram scale was



Scheme 4 Gram-scale sulfonamidation of arylboronic acids

also examined. The desired product **3j** was obtained with a yield of 80% (Scheme 4).

Conclusions

In conclusion, we have developed a green and practical method for the synthesis of *N*-arylsulfonamides from easily available chloramine salts and arylboronic acids. In this process, the chloramine salt is assumed to serve as an electrophile to oxidize the copper catalyst through oxidative addition. Importantly, this reaction can be performed on a gram scale under mild conditions, which is important to industry applications. Further exploration based on this new method for the synthesis of well-known bioactive *N*-arylsulfonamides in a convenient and economical manner is ongoing in our lab.

Acknowledgments

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A. Supplementary data

Experimental procedure, characterization data, ^{1}H and ^{13}C NMR spectra of compounds **3** and **4** can be found, in the online version.

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Graphical Abstract

Copper-catalyzed cross-coupling of chloramine salts and arylboronic acids in water: a green and practical route to *N*-arylsulfonamides

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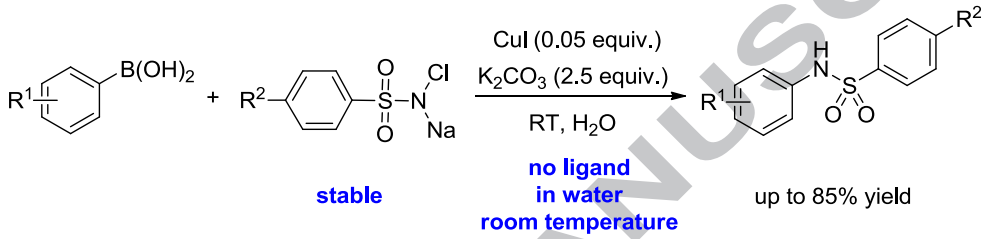
Banlai Ouyang [a, b, *], Yanxia Zheng [b], Yi Liu [b], Fei Liu [b], Juying Yao [b], Yiyuan Peng [a, *]



Highlights

1) A green and practical method for the synthesis of *N*-arylsulfonamides

2) Copper-catalyzed sulfonamidation reaction of arylboronic acids with chloramine salts

3) The reaction goes through in water at room temperature

4) A broad range of arylboronic acids are compatible with this transformation

5) This transformation can be conducted on a gram scale