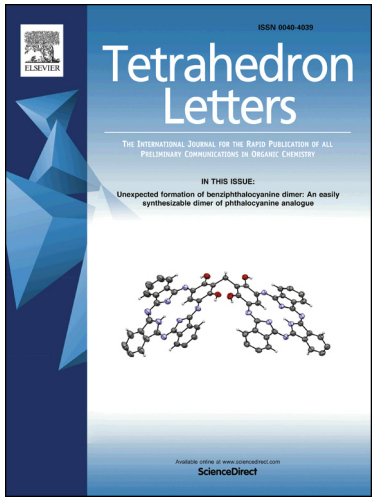

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Loukrakpam Dineshwori Chanu, Prodeep Phukan

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Graphical Abstract

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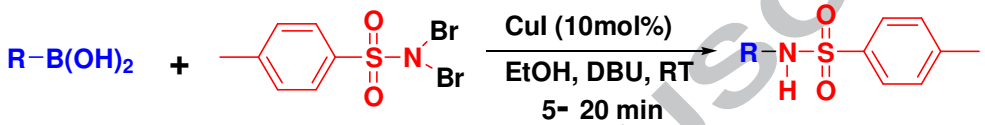

CuI catalyzed sulfamidation of arylboronic acid using $TsNBr_2$ at room temperature

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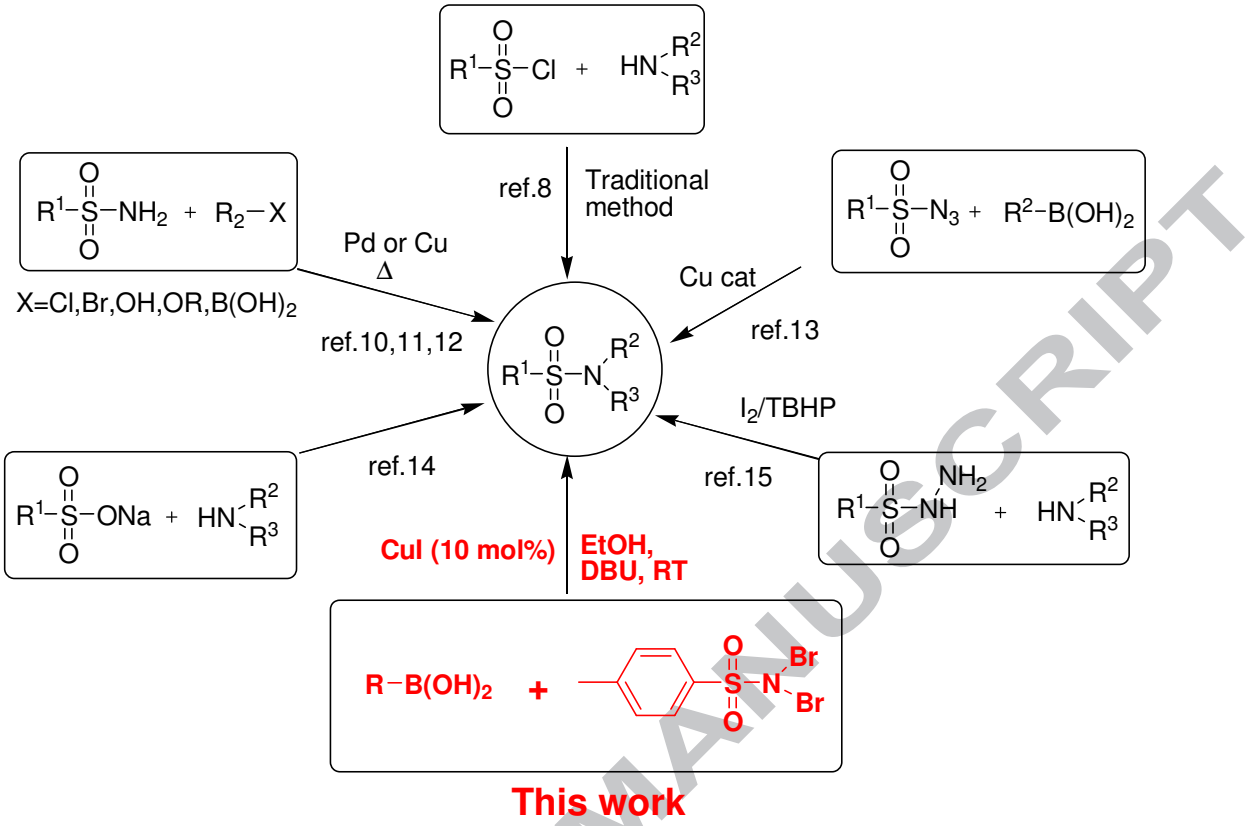
Abstract: An expeditious protocol for amidation arylboronic acid has been developed using $TsNBr_2$ as the nitrogen source in presence of a CuI as catalyst. Various arylboronic acids could be transformed into corresponding *N*- arylsulfonamide derivatives within a very short time using CuI as catalyst in presence of DBU at room temperature.

Keywords: $TsNBr_2$, CuI, arylboronic acid, arylsulfonamide

Introduction

Sulfonamides are important structural motif which is frequently encountered in pharmaceuticals, bioactive compounds and natural products.[1] Sulfonamide derivatives became popular ever since the discovery of their activities like anticancer,[2] HIV protease inhibitor,[3] anticonvulsant,[4] antimicrovial,[5] antibacterial and antifungal,[6] HCV NS5B polymerase inhibitors for acute hepatitis and chronic liver disease.[7] Pharmaceutically important examples of sulfonamides include celecoxid (analgesic), amprenavir (HIV protease inhibitor), pazopanid (antitumor), sildenafil (anti-impotance) and solfamethoxazole (antibiotic).[1-7] Owing to their wide range of applications, various methods have been developed for the synthesis of sulfonamides (Scheme 1).



Scheme 1. Different synthetic approaches towards the synthesis of sulfonamides

Generally, sulfonamides are prepared by the reaction of a primary amine (ammonia), or a secondary amine with sulfonyl chloride in presence of a base.[8] However, this methodology has limited application in pharmaceutical manufacturing since both the sulfonyl chloride and aromatic amine are genotoxic alerting structures having a very low threshold (ppm level) of residual tolerance in active pharmaceutical ingredients (API).[9] Alternative routes to N-arylsulfonamides are the transition metal catalyzed cross coupling reaction of sulfonamides with organohalides or boronic acids[10] and metal catalyzed aminosulfonation of benzylic and allylic hydrocarbons.[11] Sulfonamides can also be synthesized from arylthiols through an oxygen-activated radical protocol in presence of copper catalyst,[12] Chan-Lam coupling of sulfonyl azides and boronic acids under copper catalyst,[13] reaction of sodium sulfinates with amines under metal and metal free conditions.[14] Another synthetic approach include sulfonylation of amines or nitroarenes with arylsulfonyl hydrazides under metal-mediated or metal-free conditions.[15] Among the transition metals, palladium and copper have been generally used due to their

efficiency and compatibility with functional groups. However, toxicity and high cost of palladium catalyst restrict their use on large scale industrial application. Thus, researchers have shifted their attention towards the used of less expensive, less toxic, and more efficient metals to replace palladium catalyst. Lam *et al*., (2001) reported the synthesis of N-arylsulfonamides employing sulfonamides and arylboronic acids.[16] Since then, many extensions and modifications of the Chan–Lam coupling reaction of sulfonamides with arylboronic acids have been demonstrated.[17] Although, there are several existing methods for the synthesis of N-arylsulfonamides, yet certain significant drawbacks viz. use of non-stable, hazardous and mutagenic starting materials (e.g. sulfonyl chlorides and organic azides), generation of a large quantity of toxic waste, difficulty in handling and storing, the requirement of harsh reaction conditions and a prolonged reaction time are usually associated with them. While preparing this manuscript, we have come across a procedure for arylsulfonamide synthesis by coupling arylboronic acids with chloramine-T in presence of $Cu(OAc)_2$ as catalyst under the influence of potassium *tert*-butoxide.[18] Of late, various workers have reported the used of *N,N*-dibromo-*p*-toluenesulfonamide ($TsNBr_2$) as an efficient reagent for various organic transformation.[19] In continuation of our work on $TsNBr_2$,[20] we report herein a new synthetic approach for the preparation of *N*-arylsulfonamide using $TsNBr_2$ as the nitrogen source in presence of CuI catalyst and base under mild reaction conditions within a short reaction time. To the best of our knowledge, the present study is the first to use $TsNBr_2$ as the nitrogen sources in the synthesis of *N*-arylsulfonamide.

Results and discussion

We began our studies by selecting phenylboronic acid as the model reactant to find out the optimized condition. The results are shown in Table 1. Initially, the reaction was carried out by treating phenyl boronic acid (1 mmol) with $TsNBr_2$ (1 mmol) in presence of 3 mol equivalent of cesium carbonate in methanol at room temperature under inert atmosphere. To our disappointment, the reaction did not proceed at all even after 24h of reaction. Thereafter, the reaction was carried out using 10 mol% of $Cu(OAc)_2$ which resulted in a moderate yield of 68% after 4h of reaction. When the reaction was carried out by using palladium acetate catalyst, biphenyl was formed instead of the desired N-arylated product. On modification of nature of the

copper source, CuI was found to be the most efficient catalyst giving the desired N-arylated product in 75 % yield. Further study with 1.2 equivalent of $TsNBr_2$ could impart a slight improvement in the result. However, when the reaction was examined in presence of DBU (3 mol equivalent base on the substrate) a dramatic improvement of yield and reaction rate was observed. The reaction produced the desired *N*-tosyl aniline in 86% yield within 5 min. The reaction was found to be marginally better in ethanol as a solvent. When the study was carried out in presence of K_2CO_3 instead of DBU, the reaction took much longer time for completion (8h) with relatively lower yield (Table 1, entry 9). Increasing or decreasing the amount of copper catalyst showed no substantial improvement in the yield. Finally, the use of 1 mmol of phenylboronic acid, 10 mol% of CuI, 1.2 equiv. of $TsNBr_2$ and 3 mmol of DBU was considered as the optimum condition for achieving the best result (Table 1, entry 8). The eco-friendly solvent ethanol was chosen as the medium for carrying out this coupling reaction.

Table 1. CuI catalyzed sulfamidation reaction under different conditions[a]

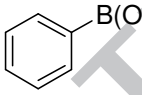
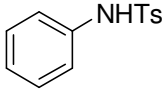
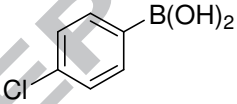
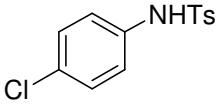
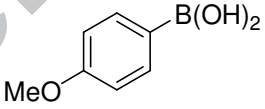
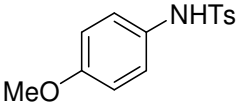
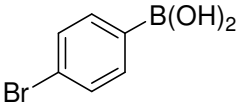
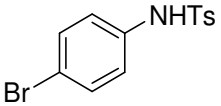
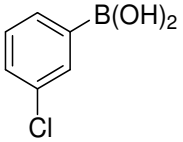
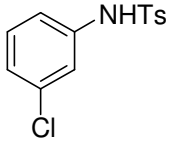
Entry	$TsNBr_2$ (mmol)	Catalyst	Catalyst amount (mol%)	Base	Solvent	Time	Yield[b] (%)
1	1	Nil	_	Cs_2CO_3	MeOH	24 h	Nil
2	1	$Cu(OAc)_2$	10	Cs_2CO_3	MeOH	4 h	68
3	1	$Pd(OAc)_2$	10	Cs_2CO_3	MeOH	4 h	_[c]
4	1	$CuCl_2.2H_2O$	10	Cs_2CO_3	MeOH	4 h	66
5	1	CuI	10	Cs_2CO_3	MeOH	4 h	75
6	1.2	CuI	10	Cs_2CO_3	MeOH	4 h	78
7	1.2	CuI	10	DBU	MeOH	5 min	86
8	**1.2**	**CuI**	10	**DBU**	**EtOH**	**5 min**	**88**
9	1.2	CuI	10	K_2CO_3	EtOH	8 h	80
10	1.5	CuI	10	DBU	EtOH	5 min	88

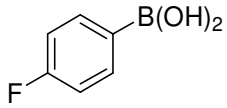
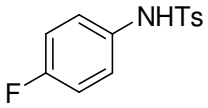
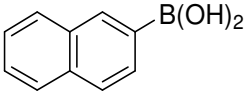
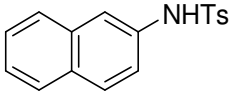
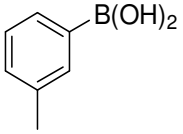
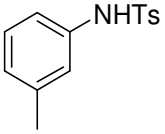
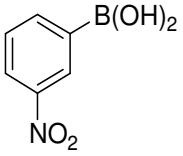
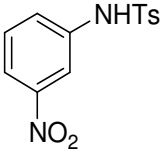
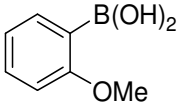
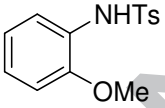
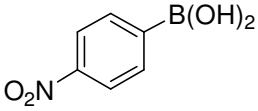
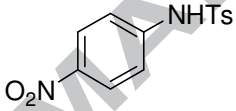
11	1.2	CuI	5	DBU	EtOH	10 min	80
12	1.2	CuI	15	DBU	EtOH	10 min	87

[a]Reaction conditions: Phenylboronic acid (1 mmol), base (3 mmol), solvent (2 ml), room temperature; [b] Isolated yield; [c] biphenyl was found as the sole product

With the optimized condition in hand, we investigated the scope of the Cu-catalyzed sulfamidation of various arylboronic acids using $TsNBr_2$ as the nitrogen source. As shown in Table 2, most of the arylboronic acids could be transformed into corresponding *N*-arylsulfonamide under the optimal condition within very short time at room temperature.[21] In our system, generally the electron rich arylboronic acid delivered the products with greater yield (Table 2, entry 3, 7, 8 & 10) then the electron deficient analogs (Table 2, entry 6, 9 & 11). It is evident that this method is reasonably general and can be applied to several kinds of arylboronic acids. In all cases, the reaction gives the corresponding sulfonamides in good to excellent yields under the reaction conditions.

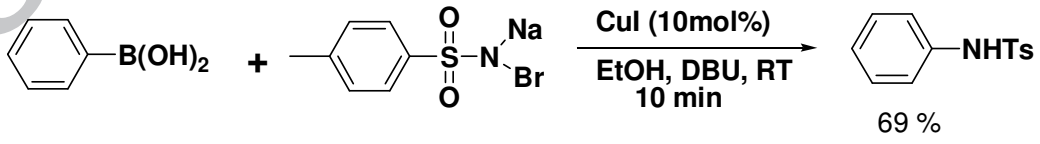
Table 2: CuI catalyzed amidation of arylboronic acids using $TsNBr_2$

Entry	Boronic acid	Product	Time (min)	Yield (%)
1			5	88
2			15	82
3			5	85
4			15	83
5			15	84

6			15	77
7			5	88
8			5	84
9			15	76
10			5	87
11			20	78

[a]Boronic acid (1 mmol), $TsNBr_2$ (1.2 mmol), CuI (10 mol%), DBU (3 mmol), EtOH (2 ml), rt; [b] Isolated yield

We have also tested the feasibility of using bromamine-T[22] as an alternative sulfonamide source. Bromamine-T was prepared from $TsNBr_2$ following a literature procedure.[22] When the reaction was carried out by treating phenyl boronic acid (1 mmol) with bromamine-T (1.2 mmol) in presence of CuI (0.1 mmol) and DBU (3 mmol) in ethanol, the reaction produced corresponding *N*-phenyl sulfonamide in 69 % yield within 10 min of reaction (Scheme 2).



Scheme 2. Synthesis of N-phenyl sulfonamide using bromamine-T

Conclusion

In summary, we have successfully developed a new simple method for the synthesis of N-arylsulfonamide through CuI catalyzed amination of arylboronic acids with *N,N*-dibromo-*p*-toluenesulfonamide as the nitrogen source in presence of DBU at room temperature. The important feature of this method is the use of $TsNBr_2$ for the first time as the nitrogen source along with inexpensive copper catalyst, resulting in higher yields within a short period of reaction time at room temperature. Further work in this area is in progress.

Acknowledgement

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21. **General procedure for the synthesis of N-arylsulfonamide**: A 10 mL round bottom flask was charged with arylboronic acid (1 mmol), DBU (3 mmol), dry ethanol (2 mL) then CuI (10 mol%) and $TsNBr_2$ (1.2 mmol) was added and stirred for appropriate time (TLC) at room temperature. After completion of the reaction as indicated by TLC, the reaction mixture was quenched by adding sodium thiosulphate (200 mg approximately), and stirring was continued for nearly 20 min. The reaction mixture was then extracted with ethyl acetate (3x20 ml), dried (Na_2SO_4) and concentrated. The crude product was purified by flash chromatography on silica gel (230-400) mesh with petroleum ether-ethyl acetate as eluent.

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Research Highlight

1. New source for sulfamidation of boronic acid
2. New pathway for C-N bond formation
3. Expeditious process-Short reaction time
4. Excellent yield of Aryl sulfonamide
5. Works well with a wide range of boronic acids