RSC Advances

COMMUNICATION

Cite this: *RSC Adv.*, 2015, **5**, 37060

Received 13th February 2015
Accepted 15th April 2015

DOI: 10.1039/c5ra05244k

www.rsc.org/advances

Ligand-free Cu-catalyzed odorless synthesis of unsymmetrical sulfides through cross-coupling reaction of aryl/benzyl/alkyl halides with an aryl boronic acid/S_8 system as a thiolating agent in PEG†

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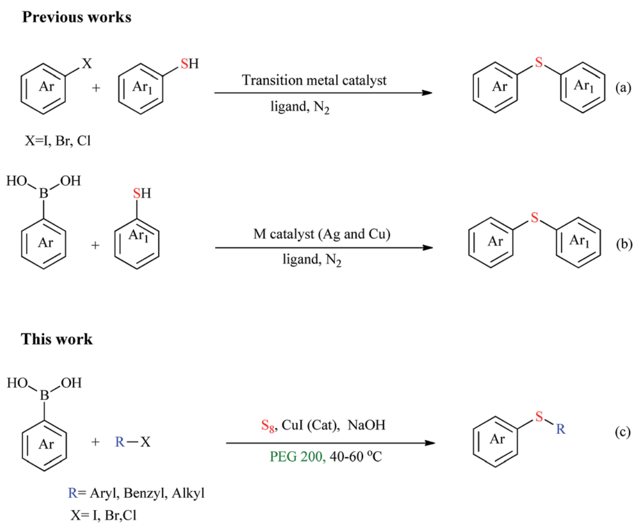
In this article, we have presented a novel, efficient and environmentally benign method for one-pot, one-step and odorless synthesis of a wide range of unsymmetrical sulfides from the reaction of aryl/benzyl/alkyl halides with aryl boronic acids in the presence of S_8, NaOH and a catalytic amount of CuI in PEG200 as green solvent at 40–60 °C. The products were obtained in moderate to excellent yields. More importantly, this reaction is applicable for the gram-scale preparation of the desired sulfides.

Aryl alkyl sulfide and diaryl sulfide compounds are important and useful in chemistry, the pharmaceutical industry and as intermediates in organic synthesis.[1–4] Numerous examples of drugs applied for diabetes, Alzheimer's and Parkinson's diseases, inflammatory and immune diseases have sulfide moieties in their structure.[5] Therefore, protocols leading to C–S bond generation, which are efficient, cheap and practical have attracted a great deal of attention.[6] The cross-coupling reactions of aryl halides with thiols are one of the most synthetic methods for the preparation of these valuable materials. However, these reactions require elevated temperatures and high boiling point, toxic and polar solvents.[7,8] To solve these drawbacks, a number of transition metal catalysts have been reported for the cross-coupling reactions between thiols and aryl halides (Scheme 1 eqn (a)).[9–13] Chan–Lam type S-arylation of thiols with boronic acids is an efficient and valuable alternative to traditional cross-couplings in the construction of C–S bond (Scheme 1 eqn (b)).[14]

Although, these efforts have led to significant progress in improving reaction conditions and substrate scope, the main limitations of these protocols are still using of volatile and foul-smelling thiols as substrate which lead to the serious safety and environmental problems. In addition, thiols are prone to form undesired disulfides, and can act as metal deactivators. In order to overcome these difficulties, various sulfur reagents such as thiourea,[15] thiolates,[16] metal sulfides,[17] ethyl xanthogenate,[18] thiocyanate,[19] carbon disulfide,[20] sulfur powder,[21] sodium thiosulfate[22] and thioimide[23] have been applied in the synthesis of sulfides. Among sulfur reagents, S_8 shows particular promise for S-arylation, due to its low cost and being odorless.

However, these procedures still require the use of Cu/N-ligand and Pd/P-ligand as the catalyst,[17–20] long reaction times,[18] using harmful organic solvents,[18] foul-smell of sulfur source[17,20] and more importantly, limited to synthesis of symmetrical diaryl sulfides[15*b*,18–21] or alkyl aryl thioethers.[15*a*,17] Therefore, there is still a great interest to find a new reagent as an arenethiol alternative to afford unsymmetrical sulfides under ligand-free and mild reaction conditions.

In the past years, aryl boronic acids were used as a source of aryl for C–C and C–hetroatom bond formation.[24] These reports prompted us to explore the prospects of this commercially



Scheme 1 Approaches to C–S bond formation.

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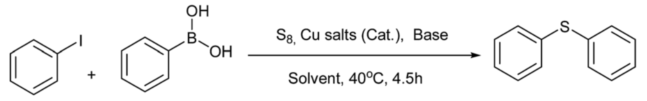
† Electronic supplementary information (ESI) available: Detailed experimental methods, and copies of the ^{1}H NMR and ^{13}C NMR spectra. See DOI: 10.1039/c5ra05244k

available, easy workable, chemically stable and eco-friendly aryl group source for other demanding reactions. To the best of our knowledge there has been no report on the C–S cross-coupling reaction of aryl boronic acid/S_8 system as a thiolating agent with aryl halides catalyzed by copper.

Herein, we present for the first time the cross-coupling reactions of aryl/benzyl/alkyl halides with aryl boronic acid/S_8 in the presence of copper as a catalyst *via* a process releasing from the unclean smell of thiols in environment-friendly solvent (Scheme 1 eqn (c)). Initially, the reaction between iodobenzene and phenyl boronic acid in the presence of S_8 at 40 °C, was selected as a model reaction, and various parameters were optimized to develop the scope of this reaction further (Table 1).

As shown in Table 1, the nature of the base was important affecting the yield of the desired product (Table 1, entries 1–7). Among organic and inorganic bases, the most effective one was found to be NaOH (Table 1, entries 7 and 8). It should be noted that the use of $Cu(OAc)_2$ instead of CuI give lower yield of diphenyl sulfide (Table 1, entry 9). In order to optimize the solvent, we have conducted the reaction in PEG200, DMF, dioxane, CH_3CN and aqueous nBu$_4$NOH (20%) (Table 1, entries 7–13). Among these solvents, PEG200 and DMF both gave the best yields compared with other solvents (Table 1, entries 7 and 8). No improvement of the yield of the desired product was observed when other S-sources, such as $Na_2S_2O_3$, Na_2S and thiourea, were used as sulfur reagents (Table 1, entries 16–18). However, the results showed that when the reaction was conducted with CuI as the catalyst, NaOH as both inorganic base and sulfur activator and PEG200 or DMF as the solvent, the desired diphenyl sulfide was obtained in excellent yield (Table 1, entries 7 and 8).

Table 1 Optimization of the reaction conditions between iodobenzene and phenyl boronic acid[a]



Entry	Base	S-source	Solvent	Cu salts	GC yield (%)
1	K_2CO_3	S_8	PEG200	CuI	0
2	Cs_2CO_3	S_8	PEG200	CuI	15
3	Et_3N	S_8	PEG200	CuI	0
4	DABCO	S_8	PEG200	CuI	0
5	KOAc	S_8	PEG200	CuI	65
6	NaOtBu	S_8	PEG200	CuI	78
7	NaOH	S_8	PEG200	CuI	100
8	NaOH	S_8	DMF	CuI	100
9	NaOH	S_8	PEG200	$Cu(OAc)_2$	89
10	NaOH	S_8	PEG200	CuI	78[b]
11	NaOH	S_8	CH_3CN	CuI	36
12	NaOH	S_8	Dioxane	CuI	35
13	—	S_8	nBu$_4$NOH (20% aq.)	CuI	78
14	NaOH	S_8	nBu$_4$NOH (20% aq.)	CuI	75
15	NaOH	S_8	nBu$_4$NOH (20% aq.)	CuI	65[c]
16	NaOH	Na_2S	PEG200	CuI	57
17	NaOH	$Na_2S_2O_3$	PEG200	CuI	0
18	NaOH	Thiourea	PEG200	CuI	0

[a] Reaction conditions: iodobenzene (1 mmol), phenyl boronic acid (1.1 mmol), S_8 (1 mmol), Cu salts (10 mol%), NaOH (4 mmol) and solvent (2 mL). [b] NaOH (3 mmol) was used. [c] The reaction was occurred at 70 °C for 8 h.

In order to generalize the scope of the reaction, a series of structurally diverse aryl/benzyl/alkyl halides was subjected to the reaction with aryl boronic acids under the optimized reaction conditions in PEG200 as the solvent. The reactions proceeded well to produce the corresponding unsymmetrical sulfides in moderate to excellent yields ranging from 65 to 98% (Table 2). It was observed that aryl halides with electron-withdrawing groups show greater activities than those having electron-donating groups (Table 2, entries 4–20). Also, p-tolyl boronic acid is less reactive than phenyl boronic acid in these reactions (Table 2, entries 1–11).

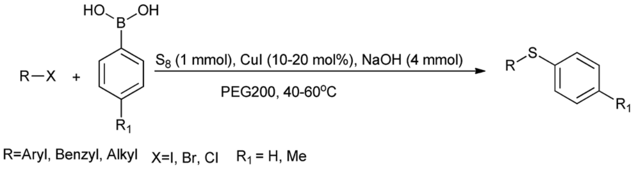
Among aryl halides the most reactive are aryl iodides (Table 2, entries 1–20). Interestingly, aryl bromides and chlorides which are relatively unreactive substrates were also converted to the corresponding unsymmetrical sulfides in moderate to good yields under the same reaction conditions (Table 2, entries 12–20).

Another important aspect of this method is the successful reaction of sterically demanding substrates to give the desired products in good yields (Table 2, entries 7 and 14). *Ortho*-substituted aryl halides require longer reaction times and gave relatively lower product yields than *para*-substituted aryl halides (Table 2, entries 6, 7, 13 and 14). The reaction of 1-bromo-4-chlorobenzene as dihalogenated aryl halide was also subjected to investigate the selectivity of the method; the bromide showed more reactivity (Table 2, entry 20). This selectivity allows remaining an active halide site for further functionalization. Benzyl bromide and benzyl chloride as two model compounds for benzylic halides were satisfactorily subjected to C–S bond formation as well (Table 2, entries 21 and 22). The cross-coupling reactions of butyl bromide, as a model for alkyl halides, with phenyl boronic acid or p-tolyl boronic acid were also conducted. The reactions proceeded well to produce the corresponding unsymmetrical sulfides in good yields (Table 2, entries 23 and 24).

It was shown that the method was suitable for large scale reactions. For this purpose, the reaction of 1-bromo-4-nitrobenzene with phenyl boronic acid was scaled-up to 10 times under similar reaction conditions. The reaction proceeded well and the desired sulfide was isolated in 89% yield (Table 2, entry 15).

In order to prove our hypothesis that, in the synthesis of unsymmetrical diaryl sulfide, $ArB(OH)_2/S_8$ act as a thiolating agent, the reaction of iodobenzene with S_8 was studied under the same reaction conditions. It was observed that the iodobenzene remained intact during the reaction. However, when the reaction temperature was increased to 100 °C two products including thiophenol and diphenyl sulfide from the coupling reaction of aryl halide and S_8 were obtained. The results showed

Table 2 C–S bond formation of aryl/benzyl/alkyl halides with aryl boronic acids using S_8 catalyzed by CuI for the synthesis of unsymmetrical sulfides[a]



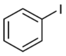
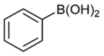
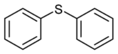
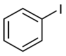
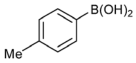
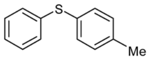
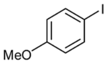
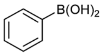
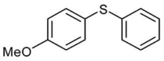
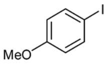
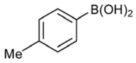
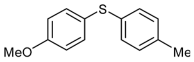
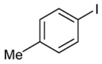
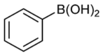
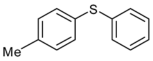
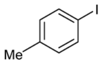
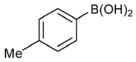
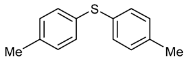
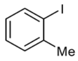
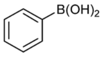
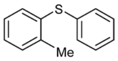
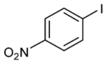
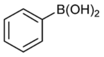
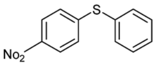
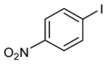
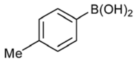
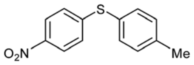
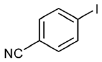
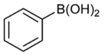
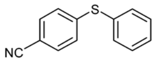
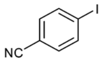
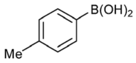
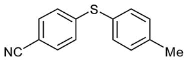
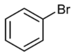
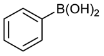
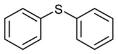
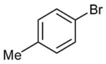
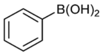
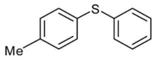
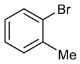
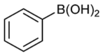
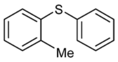
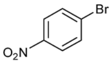
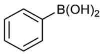
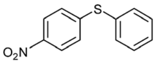
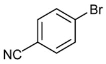
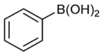
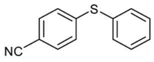
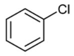
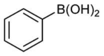
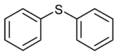
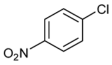
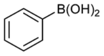
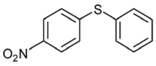
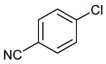
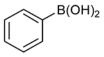
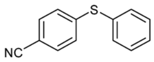
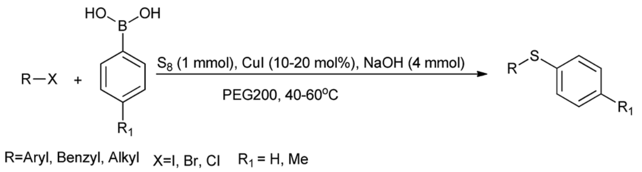
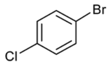
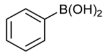
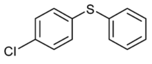
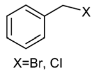
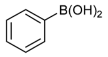
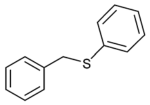
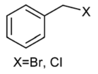
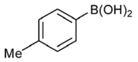
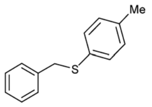
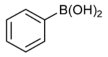
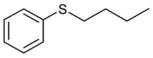
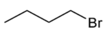
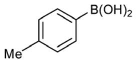
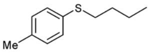
Entry	RX	ArB(OH)$_2$	Product	Time (h) : yield (%)[b]
1				4.3 : 93
2				5 : 90
3				9 : 89
4				11 : 85
5				9 : 89
6				10 : 85
7				12 : 78
8				1.3 : 94
9				3 : 98
10				2.3 : 95
11				4 : 97
12				13 : 93
13				20 : 89
14				23 : 79
15				5 : 91(89)[c]
16				6.3 : 89
17				35 : 65
18				17 : 89
19				18 : 86

Table 2 (*Contd.*)

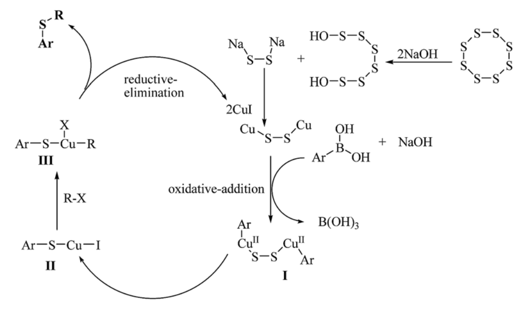


Entry	RX	$ArB(OH)_2$	Product	Time (h) : yield (%)[b]
20				21 : 79
21				X = Br 6 : 84, X = Cl 11 : 72
22				X = Br 9 : 87, X = Cl 14 : 76
23				12 : 89
24				14 : 88

[a] Reaction conditions: aryl/benzyl/alkyl halide (1 mmol), aryl boronic acid (1.1 mmol), CuI (10–20 mol%), S_8 (1 mmol), NaOH (4 mmol), 2 mL of PEG200, 40–60 °C. [b] Isolated yield after column chromatography. [c] In parenthesis the yield of the reaction performed with 10 mmol of 1-bromo-4-nitrobenzene, 10 mol% of CuI, 11 mmol of phenyl boronic acid, 40 mmol of NaOH, 20 mL of PEG200.

that aryl halides require elevated temperatures to act as thiolating agent.

We have also proposed a mechanism for this reaction which is presented in Scheme 2. On the basis of obtained results (Table 1, entries 1, 5, 6, 8, and 16) and previously reported mechanism for the formation of a stable copper disulfide complex from the reaction of S_8 with CuI,[21i] as well as formation of aryl organocopper intermediate from the reaction of aryl boronic acid with copper disulphide,[21h] we hypothesis that S_8 reacts with NaOH to give sodium disulfide. Then copper disulfide was formed from the reaction of sodium disulfide with CuI. Copper disulfide reacts with aryl boronic acid *via* oxidative-addition reaction to give aryl organocopper (intermediate I) which may convert to intermediate II. The intermediate II reacts with aryl/benzyl/alkyl halide to provide the key intermediate III. The deserted product could obtain from reductive elimination of the key intermediate.



Scheme 2 Proposed mechanism.

Conclusions

In summary, we have developed new efficient methodology for the synthesis of unsymmetrical sulfides *via* C–S bond formation reaction of aryl boronic acid with aryl/benzyl/alkyl halides using S_8 as a sulfur surrogate, NaOH as both base and sulfur activator and CuI as catalyst in the PEG200 as a green solvent. This procedure offers several major advantages; first, the synthesis of structurally diverse unsymmetrical sulfides has been reported in moderate to high yields in green solvent. Second, release from the unclean smell of thiols makes this method more eco-friendly and practical for C–S bond formation. Third, commercially available, cheap and chemically stable sulfur source, starting material and catalyst have been used.

Acknowledgements

We are grateful to the University of Kurdistan Research Councils for the partial support of this work.

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