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View Article Online
DOI: 10.1039/C6CC09201B

Journal Name

COMMUNICATION

Cu(II)-Catalyzed Sulfide Construction: Both Aryl Groups Utilization of Intermolecular and Intramolecular Diaryliodonium Salt

Received 00th January 20xx,
Accepted 00th January 20xx

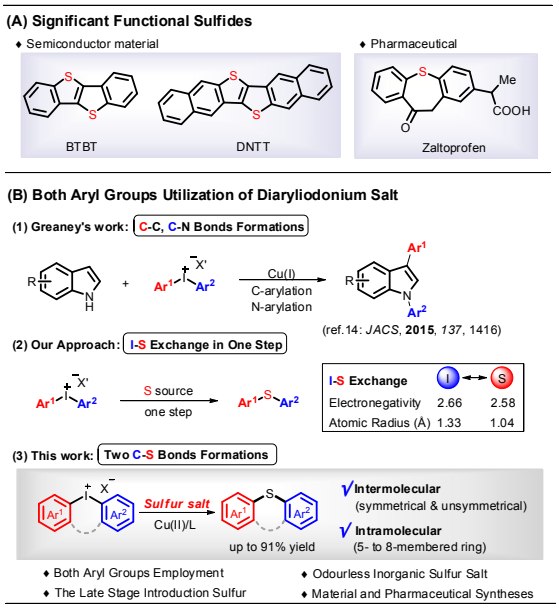
DOI: 10.1039/x0xx00000x

www.rsc.org/

Ming Wang,[a] Jianpeng Wei,[a] Qiaoling Fan[a] and Xuefeng Jiang*[ab]

A sulfur-iodine exchange protocol of diaryliodonium salts with inorganic sulfur salt was developed. Both aryl groups in diaryliodonium salt were fully exerted in this transformation. Five- to eight-membered sulfur-containing heterocycles were achieved. Notably, key unit of [1]benzothieno-[3,2-b][1]benzothiophene (BTBT) as organic field-effect transistor (OFET) material and Zaltoprofen were efficiently established through this method.

Sulfur-containing molecules extensively exist in materials,[1] natural products,[2] pharmaceuticals,[3] and even food.[4] In organic photoelectric materials, sulfur atom plays the unique role owing to its higher resonance energy than other heteroatoms.[1c] For example, thienothiophenes, thienoacenes and their derivatives are well-known organic functional material structure for thin film transistors.[1] More than 20% of top 200 U.S. prescriptions drugs were sulfur-containing drugs, and the percentage is still fleetly increasing.[3a] Thiophene, thiazepine and thiazine structures are typically occured in several famous pharmaceuticals (Scheme 1, A).[3] Traditionally, these widely existent heterocyclic sulfide structures were synthesized through cross-coupling between thiophenols and aryl halides followed by cyclization.[1c, 5] Nevertheless, introduction of sulfur in the early stage of synthetic process confronts with several conundrums: 1) Poison effect toward catalysts in the following steps due to strong coordination from sulfur to transition metals. 2) Over-oxidization to disulfide, sulfoxides, and sulphones due to sensitivity to oxidative conditions. 3) Environmental-unfriendly poison and smelly thiophenol application. Since extensive application of sulfides mentioned above, the methodology of using odourless, stable and abundant inorganic sulfur source for sulfur introduction in the late stage is highly desirable. Diaryliodonium salt is an air-/moisture-stable and well-established reagent reported by Hartmann and Meyer since 1894,[6] which is prepared easily by one step process[7] and has shown to be one of the most efficient arylation reagent in organic synthesis.[8] Cross-coupling reactions involving diaryliodonium salts with arenes,[9] alkynes,[10] alkenes,[11] heteroatoms[12] and carbonyl compounds[13] have been well developed, in which diaryliodonium salt was generally applied as a single arylation reagent. The applications of both aryl groups in diaryliodonium salt were concerned recently. Greaney group reported the first intermolecular diaryliodonium salts for double arylation of indole (Scheme 1, B1).[14] There is no other example describing the both aryl groups transformation of intermolecular diaryliodonium salt. Despite some elegant works of intramolecular diaryliodonium salts were explored,[15] it will be challenge to avoid the homo-



Scheme 1 Functional sulfides and diaryliodonium salts chemistry.

[a] Shanghai Key Laboratory of Green Chemistry and Chemical Process, School of Chemistry and Molecular Engineering, East China Normal University, 3663 North Zhongshan Road, Shanghai, 200062, P. R. China. E-mail: xfjiang@chem.ecnu.edu.cn
[b] State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 345 Lingling Lu, Shanghai 200032, P. R. China.

Electronic Supplementary Information (ESI) available: [details of any supplementary information available should be included here]. See DOI: 10.1039/x0xx00000x

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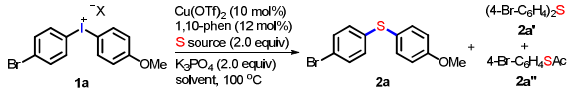
coupling product in the both utilization of intermolecular diaryliodonium salt. Continuous with our concept of sulfur atom transfer study,[16] it was envisioned that sulfur-iodine exchange for sulfide construction can stem from their similar electronegativity and atomic radius property by electron equilibration between sulfur and iodine(III) (Scheme 1, B2). Herein, we reported a new strategy for highly efficient construction of sulfides through double arylation of sulfur atom. This strategy, taking potassium thioacetate as sulfur source in the late stage of functional sulfide synthesis, was compliant with both intermolecular and intramolecular sulfur-iodine exchange (Scheme 1, B3).

Table 1 Conditions optimization[a]



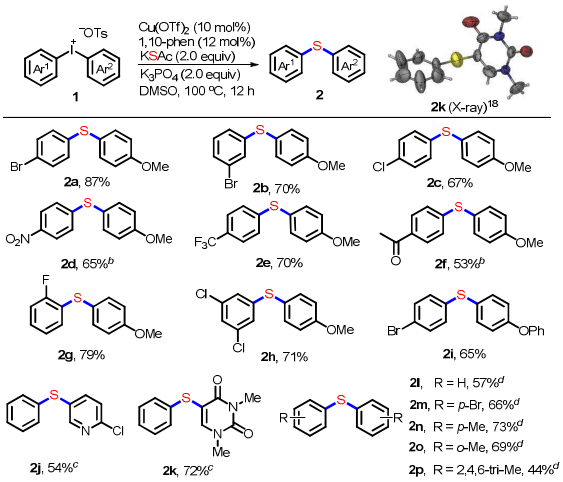
Entry	S Source	Solvent	X	Yield (%)[b]
1[c]	S_8	MeOH	BF_4	trace
2[c]	$Na_2S_2O_3$	MeOH	BF_4	trace
3[c]	$(NH_2)_2C=S$	MeOH	BF_4	trace
4[c]	Na_2S	MeOH	BF_4	15
5[c]	K_2S	MeOH	BF_4	18
6[c]	KSAc	MeOH	BF_4	23
7[c]	KSAc	DMF	BF_4	32
8	KSAc	DMF	BF_4	71
9	KSAc	DMA	BF_4	56
10	KSAc	Dioxane	BF_4	27
11	KSAc	DMSO	BF_4	77
12	KSAc	DMSO	OTf	72
13	***KSAc***	***DMSO***	***OTs***	***87***
14	KSAc	DMSO	I	69

[a] The reactions were carried out with 0.1 mmol of **1a** in 1 mL of solvent. [b] Isolated yields of **2a**. [c] Performed at room temperature.

We commenced our study with unsymmetric diaryliodonium salt **1a** and different inorganic sulfur sources catalyzed by copper in methanol at room temperature. Potassium thioacetate,[17] possessing electron-withdrawing acetyl on sulfur, was shown to be the best result and generated desired unsymmetrical aryl sulfides **2a** in 23% yield (Table 1, entries 1-6). *N*, *N*-Dimethylformamide was chosen as solvent instead of methanol to restrain the formation of symmetrical aryl sulfides **2a'** as by-product (Table 1, entry 7), in order to averting alcoholysis of acetyl. Higher temperature helped to promote the transformation to 71% (Table 1, entry 8). Dimethyl sulfoxide was fixed to be the solvent giving better yield of **2a** in 77% (Table 1, entries 9-11). Counter anions in diaryliodonium salts also played a crucial role for **2a** formation. 87% of unsymmetric sulfide **2a** was achieved when tosyloxy served as anion (Table 1, entries 12-14).

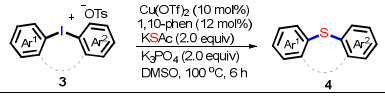
With optimized conditions in hand, intermolecular construction of unsymmetrical diaryl sulfides were sysmatically probed (Table 2). Different kinds of unsymmetrical diaryl sulfides were efficiently achieved through corresponding diaryliodonium salts derived from aryl iodides with various substituents (**2a**-**2h**). The unsymmetrical diaryl sulfides **2i** could be obtained as well when 4-phenoxyl was installed instead of 4-methoxyl. Notably, phenyl-pyridine and phenyl-uracil iodonium salts were good candidates for corresponding aryl sulfides **2j-2k** construction.[18] Logically, symmetrical diphenyl-sulfide (**2l**) was readily achieved through exchanging between diphenyliodonium salt and potassium thioacetate. Both electron-withdrawing (**2m**) and –neutral substituent (**2n**) on phenyl rings formed desired products smoothly. What needs to be pointed out, *ortho*-position substituted with methyl did not impede the exchanging, affording sulfides **2o** and **2p** in 69% and 44% yield respectively.

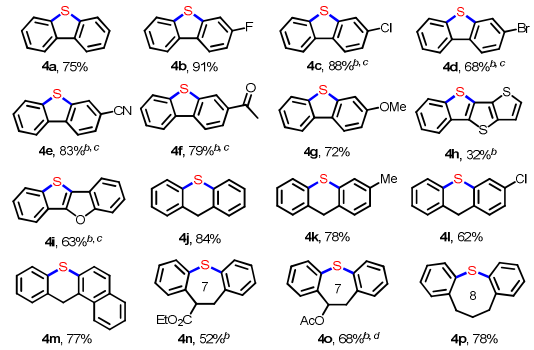
Table 2 Intermolecular sulfur-iodine exchange[a]



[a]The reaction conditions: **1** (0.1 mmol), KSAc (0.2 mmol), $Cu(OTf)_2$ (0.01 mmol), 1, 10-phenanthrolin (0.012 mmol), K_3PO_4 (0.2 mmol), DMSO (1 mL). Isolated yields. [b]Performed at 80 °C. [c]Triflate was instead of tosyloxy. [d]Tetrafluoroborate was instead of tosyloxy.

Table 3 Intramolecular sulfur-iodine exchange [a]





[a]The reaction conditions: **3** (0.1 mmol), KSAc (0.2 mmol), $Cu(OTf)_2$ (0.01 mmol), 1, 10-phen (0.012 mmol), K_3PO_4 (0.2 mmol), DMSO (1 mL). Isolated yields. [b]Triflate was instead of tosyloxy. [c]80 °C. [d]60 °C.

Following success of intermolecular sulfur-iodine exchange, intramolecular diaryl sulfides were further studied for widely existing sulfur-containing heterocycles in materials and pharmaceuticals (Table 3). Five-membered sulfur

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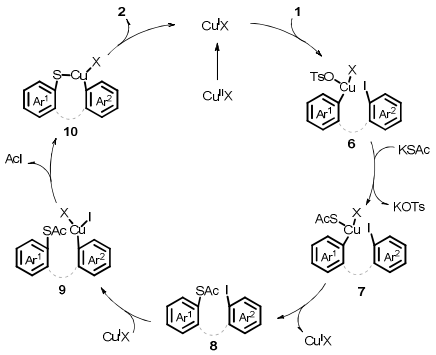
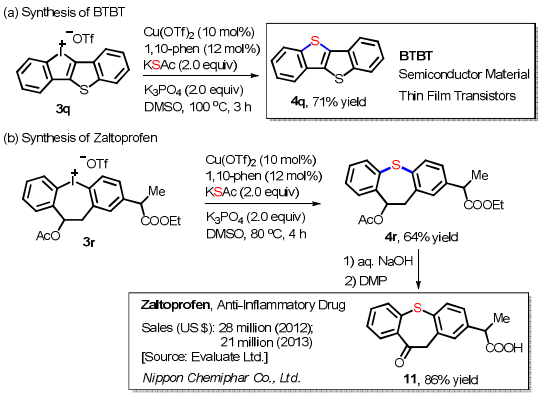
heteroaromatics with diverse electronic properties were afforded in good to excellent yields (**4a**-**4g**), which is a core structure in OFET materials.[1] Heteoaryl sulfides, derived from corresponding iodonium salts, could be also accommodated and furnished important multi-heterocyclic material structures **4h** and **4i**. Six-membered sulfur-containing heterocyclic, which are of great importance in biological active lead compound structures,[19] were obtained in 62-84% yields (**4j**-**4m**). Remarkably, even seven- and eight-membered diaryliodonium salts efficiently formed the corresponding seven- and eight-membered sulfur-containing heterocyclic products (**4n**-**4p**) in good yields, which were kernel structures of pharmaceutical intermediate.[5f] These results indicated that sulfur-iodine exchange processed predominant compatibility for different size ring formation.



Scheme 2 The plausible mechanism.

In order to figure out the mechanism of sulfur-iodine exchange reaction, we tried to detect key intermediates through decreasing the temperature to 25 °C, which gave the desired aryl sulfides **2a** in only 29% yield with aryl thioacetate **2a''** in 50% yield (SI, Scheme S1a). Further, 47% of homo-coupling product of **2a'** was isolated with only 25% of product **2a** under standard conditions when aryl thioacetate **2a''** was subjucted to diaryliodonium salt **1a**. However, intermediate **2a''** and aryl iodide **5** could afford the desired product **2a** in 88% yield under standard conditions (SI, Scheme S1b). It illustrated that aryl thioacetate is the key intermediate during sulfur-iodine exchange process. Thus, a postulated reaction pathway is depicted in Scheme 2. Intermediate **6** was afforded through oxidative addition of diaryliodonium salt **1** with Cu(I) species which had been proposed and investigated in Cu(II)-catalyzed reactions of diaryliodonium salts.[9d,12k] Subsequently, ligand exchange of **6** with potassium thioacetate formed complex **7**, followed by reductive elimination affording aryl thioacetate **8**. Oxidative addition of aryl iodine with Cu(I) catalyst provided Cu(III) aryl species **9** as Ullman type intermediate, which underwent intramolecular ligand exchange to generate intermediate **10**. Reductive elimination of **10** afforded desired product **2** and regenerated Cu(I) catalyst (Scheme 2).

BTBT (**4q**), a promising organic semiconductor structure exhibiting outstanding electron mobility in thin-film transistor settings,[20] could be afforded in 71% yield from diaryliodonium salts **3q** through this sulfur-iodine exchange approach (Scheme 3a). Non-steroidal anti-inflammatory drug Zaltoprofen (**11**) was highly efficiently synthesized from diaryliodonium salt **3r**, followed by only two steps of hydrolysis and oxidation (Scheme 3b), which provided a new synthetic route for Zaltoprofen, different from the traditional strategy.[21]



Scheme 3 Application in the syntheses of BTBT and Zaltoprofen.

In summary, an efficient sulfur-iodine exchange protocol between odourless safe sulfur salt and readily available diaryliodonium salts was developed. Both intermolecular and intramolecular sulfur-iodine exchanging process performed well to afford different types of functional sulfur-containing structures, which introduce sulfur in the late stage as a practical approach for the construction of sulfides. New pathways of the semiconductor material BTBT and non-steroidal anti-inflammatory drug Zaltoprofen exhibited great potential of this method. Further explorations of diaryliodonium salts are in progress in our laboratory.

We are grateful for financial support provided by NSFC (21672069, 21472050, 21272075), NSFC (21502054 for MW), DFMEC (20130076110023), Fok Ying Tung Education Foundation (141011), Program for Shanghai Rising Star (15QA1401800), Professor of Special Appointment (Eastern Scholar) at Shanghai Institutions of Higher Learning, and the National Program for Support of Top-notch Young Professionals.

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