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Copper-catalyzed synthesis of 2-acylbenzo[*b*]thiophenes from 3-(2-iodophenyl)-1-arylpropan-1-ones and potassium sulfide under aerobic conditions†

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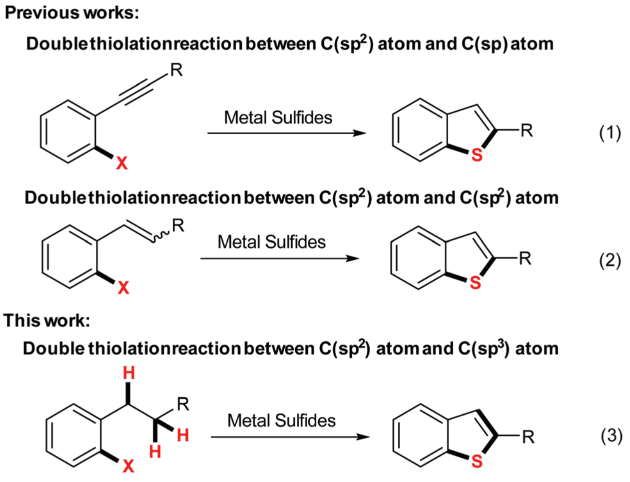
A method was developed for the synthesis of 2-acylbenzo[*b*]thiophenes *via* a copper-catalyzed sulfuration of 3-(2-iodophenyl)-1-arylpropan-1-ones with K_2S under aerobic conditions. Mechanistically, this procedure was proved to involve the formation of a dihydrobenzo[*b*]thiophene intermediate.

Benzo[*b*]thiophenes are one of the most important classes of sulfur-containing heterocyclic compounds, which constitute the key structures of many natural products, pharmaceutical drugs, and functional materials.[1–3] Therefore, a number of efficient strategies have been devised for the synthesis of benzo[*b*]thiophenes *via* the formation of C–S bonds.[4–7] In particular, inorganic sulfides as a readily available, stable, and non-toxic sulfur source were used for the synthesis of benzo[*b*]thiophenes *via* double thiolation, which has become a hot research field for chemists.[5–7] Most of these representative approaches have focused on: (a) the coupling cyclization reaction of *o*-halo alkynylbenzenes (Scheme 1, eqn (1)),[6] and (b) the coupling cyclization reaction of *o*-halo alkenylbenzenes (Scheme 1, eqn (2)).[7] These methods have realized double thiolation reactions of inorganic sulfides between C(sp^2) and C(sp) atoms and between C(sp^2) and C(sp^2) atoms, which lead to the efficient synthesis of benzothiophenes. To the best of our knowledge, double thiolation reactions of inorganic sulfides between C(sp^2) and C(sp^3) atoms for the synthesis of benzo[*b*]thiophenes have been rarely reported.[8] Accordingly, the synthesis of benzo[*b*]thiophenes *via* the construction of double C–S bonds between C(sp^2) and C(sp^3) atoms represents a significantly bigger challenge (Scheme 1, eqn (3)).

2-Acylbenzo[*b*]thiophenes, as an important derivatives of benzo[*b*]thiophenes, show their good biological activities, such as antimitotic,[9] antitrypanosomal,[10] anti-inflammatory activities.[11] Thus, developing efficient methods for the synthesis of 2-acylbenzo[*b*]thiophenes is of great value. Generally, the synthesis of 2-acylbenzo[*b*]thiophenes by the transition metal-catalyzed cross-coupling reaction from benzo[*b*]thiophene-2-yl-boronic acids was developed.[12] However, these methods were limited by the difficult preparation of starting materials and use of expensive metal catalysts. Recently, Nguyen developed an unusual DIPEA-promoted reaction of 2-nitrochalcones with elemental sulfur for the synthesis of 2-benzoylbenzothiophenes. Sekar and coworkers reported an efficient synthetic method of 2-acylbenzo[*b*]thiophenes *via* Cu-catalyzed C(sp^2)–H functionalization of 2-halochalcones.[7c] Both of them achieved the double thiolation reaction of 2-substituted chalcones between C(sp^2) and C(sp^2) atoms. As part of our ongoing research towards the copper-catalyzed double thiolation reaction *via* the cleavage of the C–X bond and C(sp^3)–H



Scheme 1 Synthesis of benzo[*b*]thiophenes.

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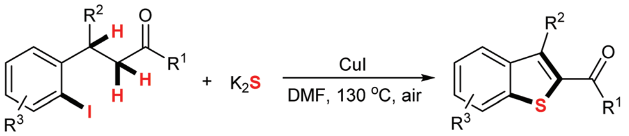
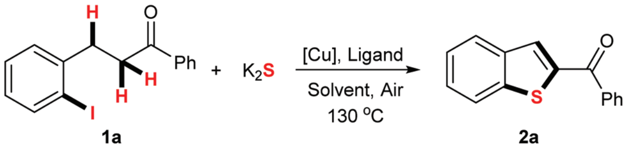


Scheme 2 Synthesis of 2-acylbenzo[*b*]thiophenes.

functionalization,[13] this synthetic strategy would be applied to the synthesis of benzo[*b*]thiophenes. Herein, we report a copper-catalyzed synthesis of 2-acylbenzo[*b*]thiophenes from the C(sp^3)–H functionalization of 3-(2-iodophenyl)-1-arylpropan-1-one (Scheme 2).

To validate our supposition, the reaction of 3-(2-iodophenyl)-1-phenylpropan-1-one **1a** and potassium sulfide was chosen as a model reaction for screening the optimized conditions (Table 1). First of all, we isolated **2a** in 43% yield by simply heating a DMF solution of **1a** and potassium sulfide (130 °C) in the presence of Cu(OAc)$_2$ as the catalyst under a nitrogen atmosphere (entry 1). When the reaction atmosphere was replaced with oxygen and air, the desired product **2a** was afforded in 61% and 88% yields, respectively (entries 2 and 3). Encouraged by these results, a series of copper salts (CuI, CuBr, CuCl, CuBr$_2$, and CuCl$_2$) were evaluated (entries 4–8), and CuI was found to be the best catalyst, and could give 92% yield of **2a**. Continuously, the ligands, including TEMED, DMEDA, and 1,10-phenanthroline, were examined. However, these ligands could not promote the coupling cyclization reaction (entries 9–11). Subsequently, solvents, such as DMSO, NMP, and CH$_3$CN, were also evaluated, and the yields of **2a** decreased slightly (entries 12–14). To improve the efficiency of the coupling cyclization reaction, different sulfur sources, such as S$_8$, Na$_2$S, Li$_2$S, and NaS$_2$O$_3$, were screened (entries 15–18). The results indicated that K$_2$S was the best sulfur source for this reaction. Finally, it was found that the lower temperature led to a decrease in the yields (entry 19). The yield of **2a** showed no significant change when the catalyst loading of CuI reduced to 10 mol% (91% yield, entry 20). Thus, the optimized reaction conditions are as follows: **1a** (0.2 mmol), K$_2$S (0.6 mmol), and CuI (10 mol%), in DMF (2 mL) under air atmosphere at 130 °C.

Table 1 Optimization of reaction conditions[a]



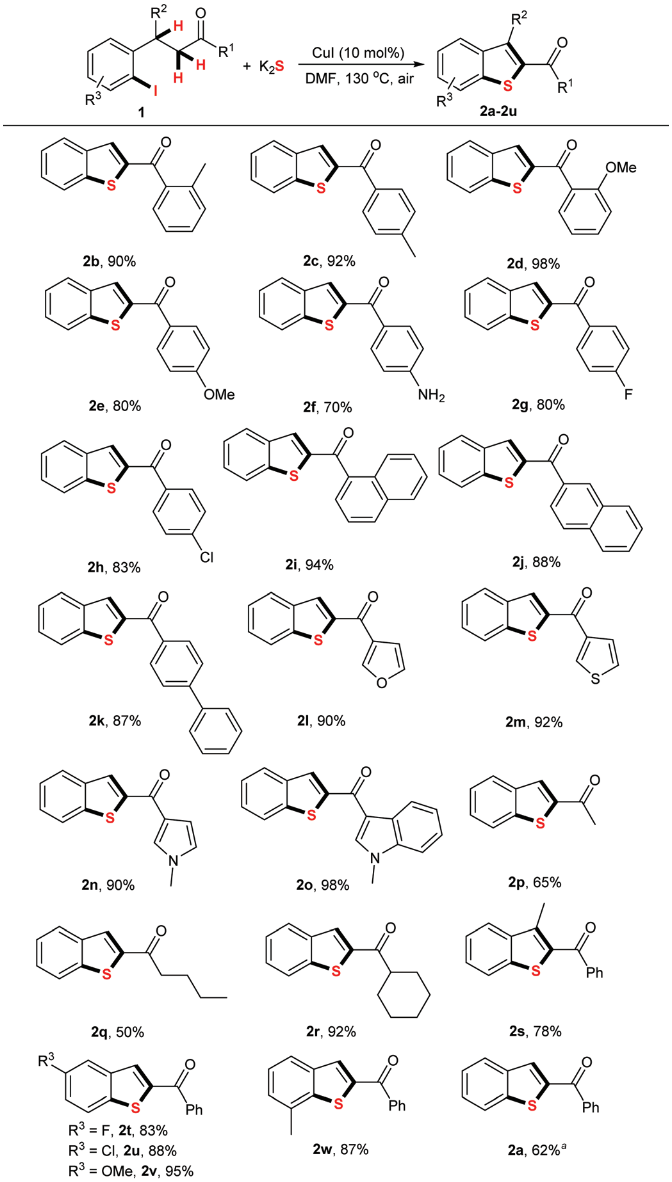
1a **2a**

Entry	Catalyst	Ligand	[S]	Solvent	Yield[b] (%)
1[c]	Cu(OAc)$_2$	—	K$_2$S	DMF	43
2[d]	Cu(OAc)$_2$	—	K$_2$S	DMF	61
3	Cu(OAc)$_2$	—	K$_2$S	DMF	88
4	CuI	—	K$_2$S	DMF	92
5	CuBr	—	K$_2$S	DMF	89
6	CuCl	—	K$_2$S	DMF	90
7	CuBr$_2$	—	K$_2$S	DMF	89
8	CuCl$_2$	—	K$_2$S	DMF	88
9	CuI	TEMED	K$_2$S	DMF	92
10	CuI	DMEDA	K$_2$S	DMF	86
11	CuI	1,10-Phen	K$_2$S	DMF	88
12	CuI	—	K$_2$S	DMSO	79
13	CuI	—	K$_2$S	NMP	78
14	CuI	—	K$_2$S	CH$_3$CN	88
15	CuI	—	S	DMF	Trace
16	CuI	—	Na$_2$S	DMF	Trace
17	CuI	—	Li$_2$S	DMF	21
18[e]	CuI	—	Na$_2$S$_2$O$_3$	DMF	57
19[e]	CuI	—	K$_2$S	DMF	86
20[f]	CuI	—	K$_2$S	DMF	91
21[g]	CuI	—	K$_2$S	DMF	86

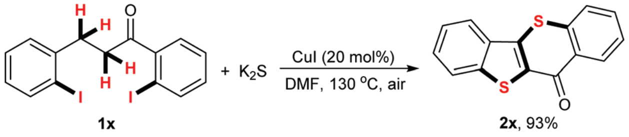
[a] Reaction conditions: **1a** (0.2 mmol), K$_2$S (0.6 mmol), catalyst (20 mol%), ligand (40 mol%), and DMF (2 mL) under air atmosphere in a sealed Schlenk tube, at 130 °C for 12 h. [b] Isolated yields. [c] Under N$_2$ atmosphere. [d] Under O$_2$ atmosphere. [e] At 100 °C. [f] CuI (10 mol%). [g] CuI (5 mol%).

With the optimal reaction conditions for the synthesis of 2-acylbenzo[*b*]thiophenes in hand, the substrate scope was investigated (Scheme 3). Initially, under the optimal conditions, an array of substituents on the aryl ring of the 3-(2-iodophenyl)-1-arylpropan-1-ones were screened. The results demonstrated that substrates **1b–1h** bearing electron-donating groups (Me, OMe, and NH$_2$) and electron-withdrawing groups (F and Cl) could be smoothly transformed into the desired products **2b–2h**. The steric effect of the methyl and methoxyl groups at the *para*- or *ortho*-position of the phenyl group did not obviously affect the yield of the product (**2b–2e**). Notably, amino substituted 3-(2-iodophenyl)-1-arylpropan-1-one was a competent reaction partner in this transformation, leading to **2f** in 70% yield. Halo-substituted **1g** and **1h** survived well, and the corresponding products were obtained in good yields. Moreover, when the aryl group of 3-(2-iodophenyl)-1-arylpropan-1-ones was displaced with 1-naphthyl, 2-naphthyl, and 4-biphenyl groups, the desired products **2i–2k** were obtained in 94%, 88%, and 87% yields, respectively. To our delight, 3-acylfuran, 3-acylthiophene, 3-acylpyrrole, and 3-acylindole derived 3-(2-iodophenyl)-1-arylpropan-1-ones could be successfully converted into the corresponding heterocycle substituted products **2l–2o** in excellent yields.

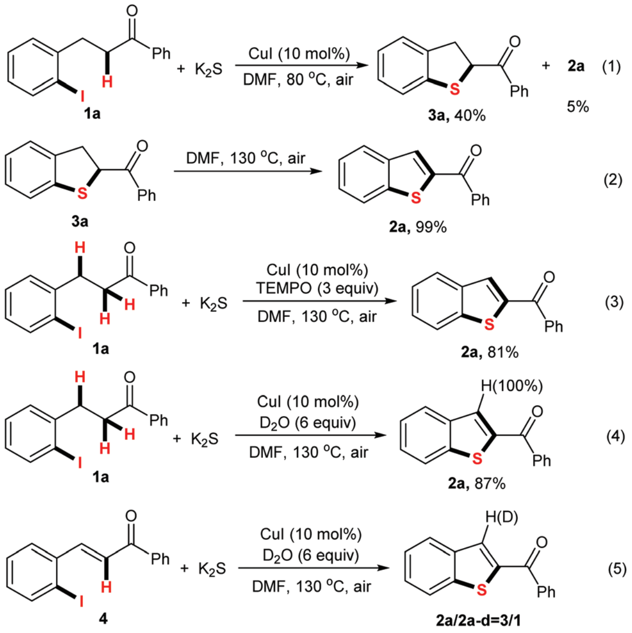
In addition, 3-(2-iodophenyl)-1-alkylpropan-1-ones could react with K$_2$S under the standard conditions, and 2-acetylbenzo[*b*]thiophenes (**2p**), 2-valerylbenzo[*b*]thiophenes (**2q**) and 2-cyclohexanecarbonylbenzo[*b*]thiophenes (**2r**) were afforded in moderate to good yields. It is noteworthy that 3-methyl-2-benzoyl benzo[*b*]thiophene could be isolated in 78% yield. Finally, both the electron-deficient group (F and Cl) and the electron-rich group (OMe and Me) on the aromatic ring of the iodobenzene moiety were well-tolerated under the reaction conditions, and the corresponding products **2t–2w** were generated in good to excellent yields. These results indicated that the electronic effect of the substituents on the iodobenzene moiety did not play a significant role in regulating the reaction, and revealed the inherent high reactivity of 2-iodophenyl. Importantly, 3-(2-bromophenyl)-1-phenylpropan-1-one could also react with K$_2$S to give **2a** in 62% yield.



Scheme 3 Synthesis of 2-acylbenzo[*b*]thiophenes. Reaction conditions: **1a** (0.2 mmol), K_2S (0.6 mmol), CuI (10 mol%), and DMF (2 mL) in a sealed Schlenk tube, at 130 °C for 12 h. Isolated yields. [a] 3-(2-Bromophenyl)-1-phenylpropan-1-one in place of 3-(2-iodophenyl)-1-phenylpropan-1-one.



Scheme 4 Synthesis of 11*H*-benzo[4,5]thieno[3,2-*b*]thiochromen-11-one.

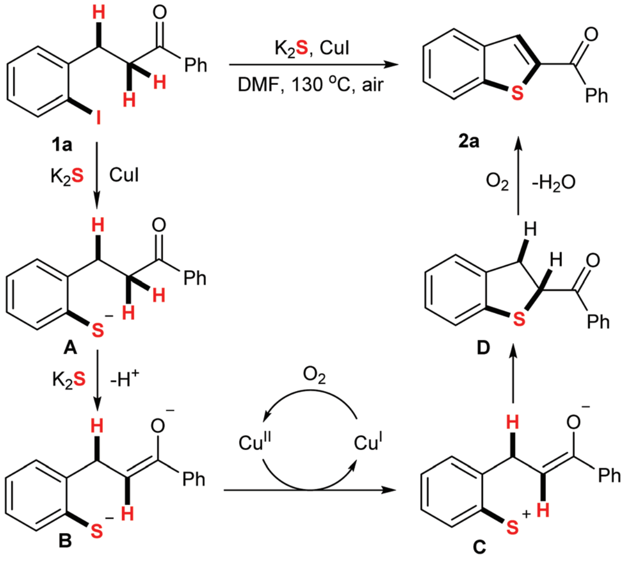


Scheme 5 Control experiments.

Notably, this approach was applicable to the construction of double sulfur-heterocyclic rings *via* double C(sp^3)–H functionalization in a step reaction, which provided an efficient route for the assembly of polycyclic sulfur-containing heterocycles. For example, 1,3-bis(2-iodophenyl)propan-1-one **1x** could efficiently react with K_2S in the presence of CuI (20 mol%) as the catalyst at 130 °C, and the desired product 11*H*-benzo[4,5]thieno[3,2-*b*]thiochromen-11-one **2x** was obtained in 93% yield (Scheme 4).

To gain insight into the mechanism of the reaction, two control experiments were performed, as shown in Scheme 5. When the reaction temperature of 3-(2-iodophenyl)-1-phenylpropan-1-one **1a** with K_2S was decreased to 80 °C, 40% of (2,3-dihydrobenzo[*b*]thiophen-2-yl)(phenyl)methanone **3a** was isolated (eqn (1)). To our delight, **3a** can be translated into the desired product in 99% yield under air conditions without CuI and K_2S (eqn (2)). These results indicated that the (2,3-dihydrobenzo[*b*]thiophen-2-yl)(phenyl)methanone **3a** should be the key intermediate in the reaction of 3-(2-iodophenyl)-1-phenylpropan-1-one **1a** with potassium sulfide. In addition, when the radical inhibitor TEMPO was added to the model reaction (eqn (3)), it was found that the yield of **2a** was only slightly decreased, which proved that this reaction could not undergo a radical process. Finally, the deuteration experiments indicated that 2-iodochalcone **4** should not be the intermediate (eqn (4) and (5)).

From the above experimental results and previous reports,[14] a possible mechanism for the formation of **2a** was proposed as shown in Scheme 6. First, intermediate **A** is afforded from the copper-catalyzed traditional coupling reaction of 3-(2-iodophenyl)-1-phenylpropan-1-one **1a** with K_2S, which is transformed into enolate **B** by base-promoting deprotonation. Then, enolate **B** undergoes the copper-catalyzed oxidation reaction and gives intermediate **C**. Subsequently, 2,3-dihydrobenzo[*b*]thiophene **D** is formed *via* the intramolecular electrophilic addition of intermediate **C**. Finally, an oxidative



Scheme 6 Possible mechanism for the formation of **2a**.

dehydrogenation of intermediate **D** gives the desired product benzo[*b*]thiophene **2a**.

In conclusion, we have developed an efficient approach for the construction of double C–S bonds *via* a copper-catalyzed coupling reaction of the C–X bond and functionalization of the C(sp^3)–H bond using K_2S as the sulfur source. In addition, the reaction has been proved to tolerate a wide variety of functional groups, and the corresponding products are obtained in good yields. This finding opens a new method which is the functionalization of the C(sp^3)–H bonds applied to the incorporation of sulfur into organic frameworks. Further applications of this method are currently underway.

Conflicts of interest

The authors declare no competing financial interest.

Acknowledgements

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