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Copper-mediated construction of benzothieno [3,2-*b*]benzofurans by intramolecular dehydrogenative C–O coupling reaction†

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An efficient method to synthesize benzothieno[3,2-*b*]benzofurans *via* intramolecular dehydrogenative C–H/O–H coupling has been developed. Good to excellent yields (64–91%) could be obtained no matter if the substituted group is electron-donating or electron-withdrawing. Notably, three-to-six fused ring thienofuran compounds could be constructed using this method. A reaction mechanism study showed that 1,1-diphenylethylene can completely inhibit the reaction. Therefore, it is a radical pathway initiated by single electron transfer between the hydroxyl of the substrate and the copper catalyst.

Introduction

The research on polycyclic aromatic hydrocarbons has achieved substantial advances in the past decades, which have led to the development of various materials for organic light-emitting diodes (OLEDs), organic field-effect transistors (OFETs) and photovoltaics (OPVs).^{1–10} Ladder-type thienoacenes such as benzothieno[3,2-*b*]benzothiophene (BTBT) have played a key role in the development of high-performance optoelectronic devices such as OFETs and OPVs.¹¹ 2,7-Dioctyl[1]benzothieno[3,2-*b*][1]benzothiophene (C8-BTBT) showed an ultra-high maximum hole mobility of 43 cm² V^{−1} s^{−1} in thin film transistors,¹² which is among the highest values reported to date for all organic molecules. Inspired by the great success of BTBT derivatives in OFETs, we have recently designed and synthesized benzo[4,5]thieno[3,2-*b*]benzofuran (BTBF) derivatives^{13,14} and benzofuro[3,2-*b*]benzofuran (BFBF) derivatives¹⁵ with either one or two furan rings substituting for thiophene rings, and investigated their packing mode, photophysical and charge transport properties.

As we all know, an oxygen atom and a sulfur atom have a similar valence electron configuration; the difference is that the oxygen atom has a smaller atomic radius and a larger electronegativity. The smaller atomic radius of the oxygen atom may decrease the intermolecular distance and increase the intermolecular π -orbital overlap, which facilitates the charge transport property in the solid state. Thus, furan-fused π -conjugated molecules are being viewed as very promising materials in organic electronics.^{9,15–21} BTBF motif is believed to

be a promising candidate for material applications as it possesses a similar structure and energy profile with those of BTBT according to the density functional theory (DFT) calculations.¹³ However, there are only a few examples for BTBFs used in optoelectronic devices.^{13,22–24} One major problem restraining the applications of BTBFs can be attributed to the lack of facile synthetic routes toward these oxygen-containing heteroacenes as oxygen atom has only one valence state of −2 to construct the furan ring. Thus, developing a new and efficient synthetic method toward BTBFs is paramount for their material applications.

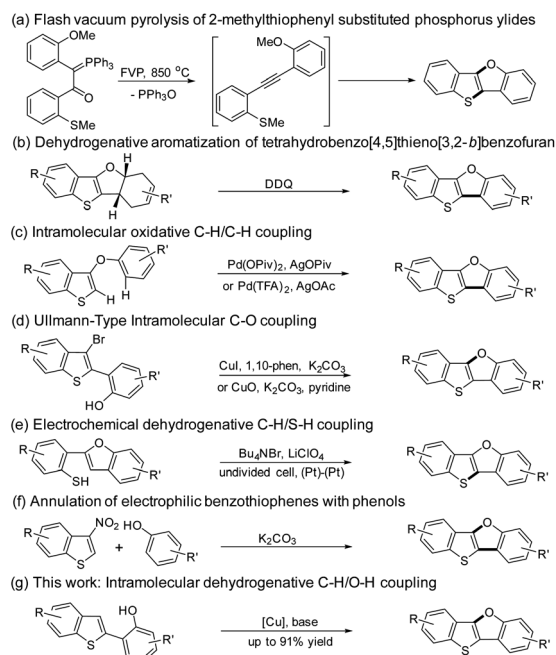
The synthesis of BTBF was firstly realized by Aitken group using flash vacuum pyrolysis of 2-methylthiophenyl substituted phosphorus ylides in 1995 (Scheme 1a).^{25,26} By the reaction of benzothieno[3,2-*b*]furan with substituted dienes, Svoboda group synthesized tetrahydro[1]benzothieno[3,2-*b*][1]benzofuran derivatives which were transformed into BTBFs by dehydrogenative aromatization (Scheme 1b).²⁷ A Pd-catalyzed intramolecular oxidative C–H/C–H coupling of 3-aryloxybenzo[*b*]thiophenes was independently reported by Kuninobu²⁸ and Miura group²⁹ in 2015 (Scheme 1c). Then a Cu-catalyzed Ullmann-type intramolecular C–O bond coupling reaction toward BTBFs was developed by our group and coworkers (Scheme 1d).¹³ Later on, You group used a similar strategy to synthesize these compounds.³⁰ Recently, Mitsudo group have reported an electrochemical synthesis of this type of compounds from 2-(benzo[*b*]furan-2-yl)benzenethiol *via* a dehydrogenative C–H/S–H coupling (Scheme 1e).³¹ Most recently, John group have developed a mild metal-free synthetic route toward BTBFs by the annulation of 3-nitrobenzothiophene with phenols (Scheme 1f).³² However, all these methods required metal catalysts or harsh conditions, and multistep transformations for the synthesis of BTBFs with limited scopes.

The construction of furan-fused π -conjugated molecules through dehydrogenative C–H/O–H coupling has been studied

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Scheme 1 Synthetic strategies toward BTBFs.

in the past decade. Liu³³ and Yoshikai group³⁴ independently reported the Pd-catalyzed synthesis of dibenzofuran derivatives through dehydrogenative C–H/O–H coupling. Zhu group discovered that copper catalysts could also promote dehydrogenative etherification to form dibenzofurans.^{35,36} Hong *et al.* reported the synthesis of heterocyclic-fused benzofurans *via* dehydrogenative C–H/O–H coupling of flavones and coumarins.³⁷ Shimada and coworkers reported that dual C–H/O–H coupling of binaphthols occurred to furnish *peri*-xanthenoxanthenes.³⁸ To be noted, the products by the methods mentioned above have been limited to aryl ethers and lactones.

Inspired by the results mentioned above, we hypothesized that BTBFs could be constructed *via* intramolecular dehydrogenative C–H/O–H coupling reaction. Herein, due to our continuing interest in Cu-catalyzed synthesis of furan-fused π -conjugated molecules,^{13–15} we have been motivated to investigate the construction of BTBFs *via* dehydrogenative C–H/O–H coupling reaction as shown in Scheme 1g. The advantages of this novel approach compared with our previous strategy (Scheme 1d) are as followed: (1) the bromination of β -position of thiophene is unnecessary which simplifies the reaction; (2) the debromination side reaction which was found in our previous strategy could be avoided which helps to improve the reaction yield.

Results and discussion

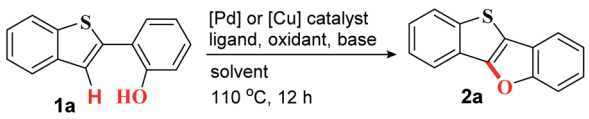
Our investigations commenced with the synthesis of 2-(benzo[*b*]thiophen-2-yl)phenol derivatives in Suzuki coupling reaction, and the yields were very high (75–98%, see ESI†). With the above compounds in hand, we started to investigate the construction of BTBFs *via* intramolecular

dehydrogenative C–H/O–H coupling reaction. Initially, intramolecular dehydrogenative C–H/O–H coupling reaction of 2-(benzo[*b*]thiophen-2-yl)phenol (**1a**) was selected as the model reaction. We first tried Pd(OAc)₂ as the catalyst, 1,3-bis(2,6-diisopropylphenyl)-2,3-dihydro-1*H*-imidazole (IPr) as the ligand, Cs₂CO₃ as the base, toluene as the solvent, and the reaction was performed under air (*i.e.*, air as the oxidant), but no target product BTBF (**2a**) was observed (see Table 1, entry 1). When the oxidant was replaced by 3 equivalent of Cu(OAc)₂, the target product was produced in 8% nuclear magnetic resonance (NMR) yield (see Table 1, entry 2). Then the ligand was replaced by pyridine (Table 1, entry 3), the yield of BTBF (**2a**) increased to 11%; if pyridine was used as the ligand and solvent, the yield was further increased to 21% (Table 1, entry 4). However, if a catalytic amount of Cu(OAc)₂ (0.2 equivalent) was used as the catalyst instead of Pd(OAc)₂, and the reaction was performed under air, only a trace amount of target compound was observed either with or without ligands (Table 1, entries 5–7). To our delight, when the amount of Cu(OAc)₂ was gradually increased to 0.5, 1 and 3 equivalent, the yield increased to 32%, 47%, 51% respectively (Table 1, entries 8–10). When the reaction was performed under pure oxygen, the yield dropped to 35% (Table 1, entry 11), which is probably due to oxidation of starting material by oxygen. Intriguingly, when 3 equivalent of Cu(OAc)₂ was used, and the reaction was performed under nitrogen, the NMR yield increased to 90% (86% isolated yield) (Table 1, entry 12). Actually, Cu(OAc)₂ was used as a catalyst and oxidant under this condition. Afterward, we screened a variety of copper oxidants (entries 13–20), bases (entries 21–23) and solvents (entries 24–28), the yields were much lower than the above yield (Table 1, entry 12).

To clarify the reaction scope, we next devoted our efforts to test a variety of 2-(benzo[*b*]thiophen-2-yl)phenol derivatives **1** under the optimized reaction conditions (Scheme 2). Firstly, we selected *o*-, *m*- or *p*-substituted 2-(benzo[*b*]thiophen-2-yl)phenols as the substrates, and found that all the yields (**2b–k**, 64–91%) were good to excellent no matter the substituted group is electron-donating or electron-withdrawing. The substrates with weak electron-donating groups such as alkyl chains afforded very high yields (**2b–d**, 85–91%), however, the substrate with a strong electron-donating group such as methoxy group gave comparably low yield (**2e**, 64%). For a specific substituent, the substituting position affects the yield: *p*-fluoro substituted BTBF (**2f**, 85%) > *m*-substituted BTBF (**2g**, 78%) > *o*-substituted BTBF (**2h**, 68%), probably due to the different steric hindrance of fluoro substituent in different position.

Next, we tested phenols with substituted benzo[*b*]thiophene moieties, and also got excellent yields (**2l–2m**, 81–91%). For more π -extended 3-(benzo[*b*]thiophen-2-yl)naphthalen-2-ol as the substrate, the yield only slightly dropped, affording benzo[4,5]thieno[3,2-*b*]naphtho[2,3-*d*]furan (**2n**, BTNF, 63%). Although substrates having a thiophene unit (**1o**) or bithiophene unit (**1p**) could also be used in this reaction, the yields significantly dropped (**2o**, 45%; **2p**, 40%), probably because the cation intermediate could not be effectively stabilized without the resonance with phenyl group (see proposed mechanism,

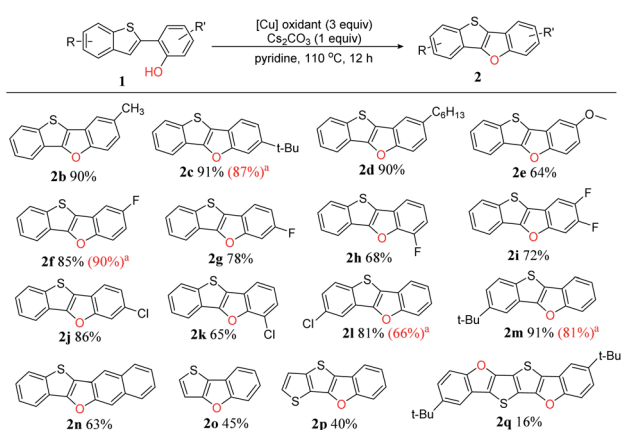


Table 1 Screening reaction conditions for the synthesis of BTBFs via intramolecular dehydrogenative C–O coupling reaction^a


Entry	[Pd]/[Cu]	Ligand	Base	Oxidant	Solvent	Yield ^b (%)
1	Pd(OAc) ₂ , 0.2 equiv.	IPr	Cs ₂ CO ₃	Air	Toluene	0
2	Pd(OAc) ₂ , 0.2 equiv.	IPr	Cs ₂ CO ₃	Cu(OAc) ₂	Toluene	8
3	Pd(OAc) ₂ , 0.2 equiv.	Pyridine	Cs ₂ CO ₃	Cu(OAc) ₂	Toluene	11
4	Pd(OAc) ₂ , 0.2 equiv.	Pyridine	Cs ₂ CO ₃	Cu(OAc) ₂	Pyridine	21
5	Cu(OAc) ₂ , 0.2 equiv.	<i>o</i> -Phen	Cs ₂ CO ₃	Air	Pyridine	Trace
6	Cu(OAc) ₂ , 0.2 equiv.	2,2'-Bipyridine	Cs ₂ CO ₃	Air	Pyridine	Trace
7	Cu(OAc) ₂ , 0.2 equiv.	—	Cs ₂ CO ₃	Air	Pyridine	Trace
8	Cu(OAc) ₂ , 0.5 equiv.	—	Cs ₂ CO ₃	Air	Pyridine	32
9	Cu(OAc) ₂ , 1 equiv.	—	Cs ₂ CO ₃	Air	Pyridine	47
10	Cu(OAc) ₂ , 3 equiv.	—	Cs ₂ CO ₃	Air	Pyridine	51
11	Cu(OAc) ₂ , 3 equiv.	—	Cs ₂ CO ₃	O ₂	Pyridine	35
12	Cu(OAc) ₂ , 3 equiv.	—	Cs ₂ CO ₃	—	Pyridine	90 (86) ^c
13	Cu(OAc) ₂ ·H ₂ O, 3 equiv.	—	Cs ₂ CO ₃	—	Pyridine	64
14	CuOAc, 3 equiv.	—	Cs ₂ CO ₃	—	Pyridine	51
15	Cu ₂ S, 3 equiv.	—	Cs ₂ CO ₃	—	Pyridine	7
16	CuS, 3 equiv.	—	Cs ₂ CO ₃	—	Pyridine	Trace
17	CuCl, 3 equiv.	—	Cs ₂ CO ₃	—	Pyridine	0
18	CuCl ₂ , 3 equiv.	—	Cs ₂ CO ₃	—	Pyridine	0
19	CuBr ₂ , 3 equiv.	—	Cs ₂ CO ₃	—	Pyridine	0
20	CuI, 3 equiv.	—	Cs ₂ CO ₃	—	Pyridine	0
21	Cu(OAc) ₂ , 3 equiv.	—	K ₂ CO ₃	—	Pyridine	34
22	Cu(OAc) ₂ , 3 equiv.	—	NaOAc	—	Pyridine	59
23	Cu(OAc) ₂ , 3 equiv.	—	—	—	Pyridine	20
24	Cu(OAc) ₂ , 3 equiv.	Pyridine	Cs ₂ CO ₃	—	Toluene	30
25	Cu(OAc) ₂ , 3 equiv.	—	Cs ₂ CO ₃	—	DMSO	44
26	Cu(OAc) ₂ , 3 equiv.	—	Cs ₂ CO ₃	—	DMF	Trace
27	Cu(OAc) ₂ , 3 equiv.	—	Cs ₂ CO ₃	—	Isopropanol	0
28	Cu(OAc) ₂ , 3 equiv.	—	Cs ₂ CO ₃	—	<i>t</i> -Butanol	0

^a Reaction conditions: **1a** (0.20 mmol), Pd(OAc)₂ or Cu catalyst (0.2–3 equiv., 0.04–0.6 mmol), ligand (0.4 equiv., 0.08 mmol), base (1 equiv., 0.20 mmol), oxidant (for entries 2–4, 3 equiv., 0.60 mmol), solvent (4 mL), 110 °C, the reactions were performed under nitrogen if the oxidant is neither air nor oxygen. ^b Determined by ¹H NMR with 1,3,5-trimethoxybenzene as an internal standard. ^c Isolated yields.

vide infra). Thus, not surprisingly, the yield of **2q** is even lower (16%) because it is obtained *via* two subsequent intramolecular dehydrogenative C–H/O–H couplings. Thus, three-to-six fused



Scheme 2 Reaction scope for the intramolecular dehydrogenative C–O coupling reaction under optimized conditions. ^a The yields in the parenthesis reported in our previous paper are shown for comparison with this work.¹³

ring thienofuran compounds could be constructed. Notably, the yields of BTBFs are comparable or even higher than those reported in our previous paper (**2c** 91% vs. 87%, **2f** 85% vs. 90%, **2l** 81% vs. 66%, and **2m** 91% vs. 81%),¹³ indicating advantage of the new synthetic approach.

Subsequently, we investigated the gram-scale synthesis of copper-mediated construction of benzothieno[3,2-*b*]benzofurans by intramolecular dehydrogenative C–O coupling reaction. 2-(Benzo[*b*]thiophen-2-yl)phenol derivatives **1a** and **1f** (6 mmol) reacted under the optimised reaction conditions, affording their corresponding benzothieno[3,2-*b*]benzofurans **2a** and **2f** in high yields which are comparable with those obtained on a 0.2 mmol scale (Fig. 1).

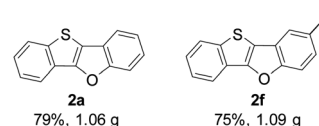
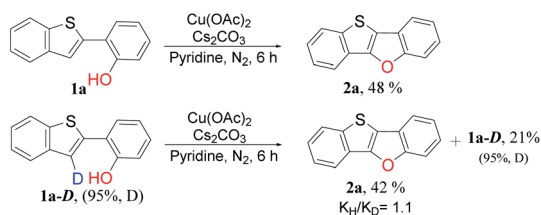
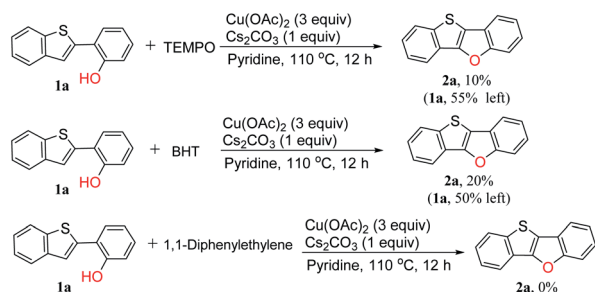


Fig. 1 Gram-scale synthesis.





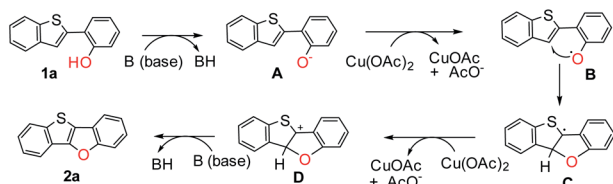
Scheme 3 Kinetic isotope effect study. Reaction conditions: **1a** and **1a-D** (0.20 mmol), Cu catalyst (3 equiv., 0.6 mmol), base (1 equiv., 0.20 mmol), solvent (4 mL), 110 °C, 6 h under nitrogen.



Scheme 4 Free radical capture experiment.

To gain insight into the reaction mechanism, some deuterium-labeling experiments were conducted with deuterated 2-(benzo[*b*]thiophen-2-yl-3-*d*)phenol (**1a-D**) (Scheme 3). The rate measurements revealed kinetic isotope effects (KIEs) of 1.1, indicating that the C3–H bond cleavage is not necessarily involved in the turnover limiting step of the reactions. The deuterated percentage in residual compounds did not change obviously, which indicated that cleavage of C3–D or C3–H bond is irreversible and thus phenolic hydroxyl played an important role in C–H functionalization.³⁷

A radical inhibition test was carried out in order to gain insight into whether the reaction proceeded through radical intermediates (Scheme 4). We selected 2,2,6,6-tetramethylpiperidine 1-oxyl (TEMPO), butylated hydroxytoluene (BHT) and 1,1-diphenylethylene as the radical scavenger which are widely used in free radical capture experiments.^{39–43} It is found that the NMR yield dropped to 10% in the presence of TEMPO, and 55% of the starting material was still left. In the presence of BHT, the yield of the target product dropped to 20%. While the desired cyclization reaction was completely inhibited by 1,1-diphenylethylene. These results clearly indicate that the reaction pathway is a radical mechanism. Unfortunately, all the efforts to detect the adducts between the substrate and radical intermediates failed.



Scheme 5 Proposed mechanism.

Although the detailed mechanism remains to be elucidated, a possible reaction pathway is proposed based on the above observations (Scheme 5). At first, **1a** reacts with a base, generating anionic intermediate **A**. Then phenoxy radical **B** is produced *via* single electron transfer with Cu(OAc)₂, followed by intramolecular C–O cyclization to generate radical intermediate **C**. Afterward, cationic intermediate **D** is formed by oxidation with Cu(OAc)₂. Finally, the target product **2a** is obtained by the abstraction of a proton with the base.

Conclusions

In summary, we have developed an efficient method to synthesize benzothieno[3,2-*b*]benzofurans *via* intramolecular dehydrogenative C–H/O–H coupling in up to 91% yield. Notably, three-to-six fused ring thienofuran compounds could be constructed. The advantages of this novel approach include unnecessary bromination of β-position of thiophene and thus inhibition of the debromination side reaction and high yield. Reaction mechanism study showed that it is a radical pathway initiated by single electron transfer between substrate and copper catalyst.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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