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Efficient silver-catalyzed direct sulfenylation and selenylation of rich arenes†

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Received 18th September 2014, Accepted 3rd October 2014 DOI: 10.1039/c4ob01992j An efficient protocol for silver/copper-cocatalyzed direct sulfenylation and selenylation of arenes with aryl disulfides and diselenides has been developed. This strategy exhibits excellent functional group tolerance and high regioselectivity. Mono sulfenylation and selenylation products can be exclusively achieved. This reaction provides a simple and practical route to the preparation of aryl sulfides and selenides.

Organochalcogenides are very important molecules frequently found in the biological and pharmaceutical fields.¹ Apart from the conventional methods of coupling aryllithiums/aryl Grignard reagents with chalcogenide precursors towards formation of carbon-chalcogen (S, Se) bond,² one of the more powerful approaches is based on transition-metal-catalyzed coupling of aryl halides,³⁻¹¹ triflates^{4d,12} or boronic acids¹³ with a suitable chalcogenide precursor. Complexes and salts of Pd^{4,12} Ni,⁵ Cu,^{6,13} Fe,⁷ Co,⁸ Rh,⁹ In,¹⁰ and Ru¹¹ have been introduced as efficient catalysts for this type of reaction. However, prefunctionlization of the starting material can be considered a drawback of this approach in comparison to another approach through the direct C-H bond activation process. While in the area of C-H bond functionalization, much attention has been paid to transition metal catalyzed C-C, C-O, and C-N bond-forming reactions,¹⁴ the formation of intermolecular C-S/C-Se bonds through transition-metal catalyzed direct C-H activation is relatively rare.15 Metal-free C-S bond formation is also reported but to a very small extent.¹⁶

In general, the metal-catalyzed processes involve predominantly the use of thiols or disulfides as coupling partners. However, thiols are prone to undergo oxidative S–S coupling reactions, resulting in the undesired formation of disulfides. Therefore, the use of dichalcogenides is preferred. Since the pioneering Cu-mediated direct intermolecular thioetherification of the arene C–H bond reported by Yu and co-workers,^{15*a*} there have been a few reports on chelation-assisted C–S bond formation.^{15*b*-*f*} Though these methods are highly regioselective, success requires a heteroatom in the molecule that acts as a directing group. The direct C–S/Se bond formation of non acidic C–H bonds of arenes is very challenging in comparision to acidic heterocycles.^{15*g*,*h*,*j*,*n*,*o*} Cheng *et al.*^{15*k*} and Beller^{15*m*} reported a nonchelation-assisted C(sp²)–H functionalization of electron-rich arenes using Cu and Pd respectively as catalysts. With these methods, the yields of the products were found to be moderate and corresponding selenide analogues are less available. Recently, Lee and co-workers¹⁵^p reported the Cu-catalyzed, highly regioselective synthesis of aryl chalcogenides using arenes as coupling partners. However, it proceeded through tandem C–H borylation and copper-catalyzed C–S coupling reactions. Continuing the study on metal catalyzed C–S/Se bond formation of arenes, in this paper we have introduced silver in combination with copper as cocatalysts that can efficiently carry out the formation of C–S/Se bond in the presence of various dichalcogenides.

Initially, we carried out sulfenylation of 1-methoxynaphthalene using diarydisulfide (2a) with AgSbF₆ as a Lewis acid catalyst at room temperature (Table 1, entry 1). The reaction was carried out in 1,2-dichloroethane (DCE) as solvent, but only trace amounts of product were detected by GC-MS. To our delight, increasing the temperature to 100 °C (Table 1, entry 2), the sulfenylation was found to occur with 47% yield (product 3a). The NMR spectral data revealed the sulfenylation process at the para position of the naphthalene ring. We then changed the Ag source under this improved reaction condition to get a better yield (Table 1, entries 3-5) but the reaction did not occur in all cases. In an attempt using $Cu(OAc)_2 \cdot H_2O$ as an additive^{15k} (Table 1, entry 6) at 100 °C, the product 3a was isolated with excellent yield (85%) within 12 h of reaction time. However, Cu(OAc)₂·H₂O by itself cannot provide the sulfide product 3a. (Table 1, entry 7). So the use of this additive is essential for the Ag catalyzed process. $\text{Cu}(\text{OAc})_2{\cdot}\text{H}_2\text{O}$ is an oxidant and was used in catalytic amount in the presence of air. Then, we examined the reaction using $Cu(OAc)_2 \cdot H_2O$ at other temperatures (Table 1, entries 11 and 12) but low yield in comparision to entry 6 was recorded. Screening of different solvents had shown that toluene (Table 1, entry 8) can provide 68% yield while in DMF (Table 1, entry 9) and DMSO (Table 1, entry 10), the desired product could not be obtained. Though decreased amounts of diarydisulfide (2a) results in compara-

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^{*a*} Unless otherwise noted, the reaction conditions are: **1a** (0.3 mmol), **2a** (0.3 mmol), catalyst (0.15 eq.), solvent (2 mL), 12 h. ^{*b*} Isolated yield. ^{*c*} Cu(OAc)₂·H₂O (0.4 eq.), 12 h. ^{*d*} **2a** (0.2 mmol). ^{*e*} AgSbF₆ (0.3 eq.).

tively lower yield during the process (Table 1, entry 13), with higher loading of the catalyst (Table 1, entry 14), the system enables the use of two RS in (RS)₂. Hence the catalyst AgSbF₆ in combination with Cu(OAc)₂·H₂O at 100 °C using DCE as solvent was found to be the best reaction condition (Table 1, entry 6)[±].

Having identified the optimal reaction conditions, this approach was then applied to the coupling of 1-methoxynaphthalene to a variety of disulfides, the results of which are shown in Table 2. The electronic effects of the substituents on the aromatic ring of disulfides hardly influenced the reactivity (Table 2, entries 1-10) and provided the sulfides in good to excellent yield. However, the NO2 substituent on the aromatic ring of the disulfide provided only 21% yield of the product (3k), while the amine group (product 3l) provided a trace amount. The position of the substituent on the benzene ring had a slight impact on the reaction yield. Substituents at the para position provided more yield in comparision to ortho and meta. This is due to steric restriction of the disulfides. No regioisomeric products or disulfenation products were observed by ¹H NMR spectroscopy. The process was also extended to disulfides bearing heteroarenes (entries 14 and 15), but only a trace amount of product was obtained.

 Table 2
 Direct sulfenylation of 1-methoxynaphthalene with disulfides^a

	OMe + RSSR ·	15 mol% AgSbF ₆ 0.4 eq. Cu(OAc) ₂ ·H ₂ O DCE, 100°C, 12h	MeO
1a	2а-р		За-р
Entry	RSSR (2a-p)	Product (3	$\mathbf{a-p}) \qquad \text{Yield}^{b}(\%)$
1	$R = 4 - ClC_6H_4$ 2	2a 3a	85
2	$R = 3 - ClC_6H_4$ 2	2b 3b	79
3	$R = 2 - ClC_6H_4 2$	2c 3c	69
4	$R = 4 - BrC_6H_4$	2d 3d	78
5	$R = 2 - BrC_6H_4$	2e 3e	67
6	$R = 4 - FC_6 H_4 2f_6$	f 3f	83
7	$R = 2 - FC_6 H_4 2g$	g 3g	69
8	$R = 4 - MeC_6H_4$	2h 3h	95
9	$R = 3-MeC_6H_4$	2i 3i	78
10	$R = 2 - MeC_6H_4$	2j 3j	64
11	$R = 4 - NO_2 C_6 H_2$	4 2k 3k	21
12	$R = 2 - NH_2C_6H$	4 2l 3l	Trace
13	$R = C_6 H_5 2m$	3m	80
14	R = 2-Pyridiny	l 20 30	Trace
15	R = 4-Pyridiny	l 2p 3p	Trace





Scheme 1 Scope of the sulfenylation process of rich arenes with diaryldisulfide (2a).

After successful investigation of the sulfenylation of 1-methoxynaphthalene, we also observed the efficiency of the process with different aromatic systems as coupling partner to **2a.** However, it was revealed that only electron rich arenes (Scheme 1) are suitable for the sulfenylation process. Disappointingly, the scope of the arenes is limited.

We next turned our attention towards the formation of C-Se bonds. Under similar reaction conditions using diaryldi-

[‡]Typical procedure for the sulfenylation and selenylation of rich arenes: Under air atmosphere, 1-methoxynaphthalene (47.4 mg, 0.3 mmol), diaryl disulfides or diaryl diselenides (0.3 mmol), AgSbF₆ (0.15 eq. or 0.20 eq.), Cu(OAc)₂·H₂O (0.4 eq. or 1.0 eq.) were added to a screw-capped vial, followed by addition of a stir bar and DCE (2 mL). The reaction vial was placed in a temperature-controlled aluminium-heating block set at 100 °C. After 12 h of stirring, the vial was removed from the heating block and was left at ambient temperature. The solvents were removed under reduced pressure and the crude reaction mixture was purified by silica gel column chromatography with Petroleum ether/EtOAc as an eluent to give the desired product.

 Table 3
 Direct selenylation of 1-methoxynaphthalene with diaryl

 diselenides^a
 Image: selenylation of 1-methoxynaphthalene with diaryl



^{*a*} The reaction conditions are as: **1a** (0.3 mmol), **4a-f** (0.3 mmol), AgSbF₆ (0.20 eq.), Cu(OAc)₂·H₂O (1.0 eq.), DCE (2 mL), 120 °C, 12 h. ^{*b*} Isolated yield.

selenides, selenide products of 1-methoxynaphthalene (Table 3, **5a–5f**) were isolated in very good yield of 60–87%. However, use of 1 equivalent $Cu(OAc)_2 \cdot H_2O$ at 120 °C was found to be the best condition for the selenylation process and also required slightly more catalyst loading (20 mol%) in comparision to the sulfenylation process. Both electron withdrawing and electron donating substituents on the aryl ring of the diselenide are equally effective and do not exhibit significant differences. The extension of the process with another electron rich arene was found to be effective under the reaction conditions (Scheme 2).

The exact mechanistic path of the reaction is not clear at this moment. We believe there is a radical reaction path since the use of TEMPO (Scheme 3), a radical-trapping reagent, completely suppressed the formation of the desired product **3a**. However, under the reaction conditions it was not possible to detect the formation of the TEMPO adduct.

On the basis of our observations, a plausible mechanism has been proposed (Fig. 1). Initially, the interaction between 1-methoxynaphthalene and Ag(1) salt generated aryl-Ag(1)species **A** at 4-position of the ring, which reacted with diarydisulfide to afford intermediate **B** through a single-electron



Scheme 2 Direct selenylation of 1,3,5-trimethoxybenzene with 1,2-di*p*-tolyldiselane.



Scheme 3



Fig. 1 Proposed mechanism of the reaction.

transfer process. Subsequent reductive elimination of intermediate **B** gave the desired product, along with Ag (0) species which can be oxidized to Ag(1) by Cu(11) and/or air to complete the catalytic cycle. Therefore, Ag(1) is used as a Lewis acid through the classical electrophilic substitution providing highly regioselective sulfenylation of 1-methoxynaphthalene at the 4-position of naphthalene ring and only electron-rich arenes were suitable under the reaction conditions. In addition, the use of oxidant Cu(OAc)₂ in catalytic amount improves the reaction yield. We speculate the reduced Cu(1) is oxidized back to Cu(11) in the presence of air.

In conclusion, we have developed a direct sulfenylation and selenylation process of arenes using silver in combination with copper as cocatalysts. The present method is very effective with a number of dichalcogenides. However, only electron rich arenes were found to be successful under the reaction conditions.

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