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Rhodium-Catalyzed Intermolecular Oxidative Cross-Coupling of (Hetero)Arenes with Chalcogenophenes

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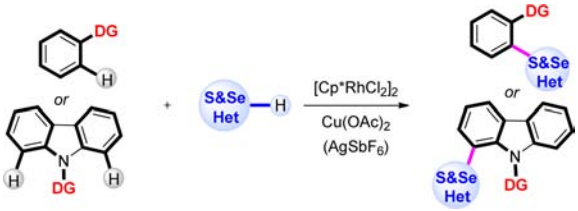
ABSTRACT



A straightforward and efficient method for the rhodium-catalyzed intermolecular oxidative cross-coupling of arenes and heteroarenes with thio- and selenophene derivatives (chalcogenophenes) via double C−H bond cleavage has been developed by using $Cu(OAc)_2/AgSbF_6$ as an oxidant. The reaction is applicable to a wide range of (hetero)arenes carrying a directing group and chalcogenophenes to yield substituted biaryl heterocyclic derivatives and oligothiophene derivatives in moderate to high yields.

π-Conjugated organic molecules containing bi-, tri-, tetra-, and polyheteroaryls constitute an important class of heterocyclic compounds having unique biological activities or physical properties. Among them, thiophene, selenophene, and thienylcarbazole derivatives have attracted considerable attention as promising advanced materials for electroconductive organic materials, semiconductors, organic light-emitting diodes (OLEDs), organic field effect transistors (OFETs), organic solar cells, lasers, dyes, liquid crystals, and so on.[1]

Transition-metal-catalyzed cross-coupling reactions between Ar−m and Ar′−X provide a practical and reliable access to biaryls Ar−Ar′.[2] Direct arylation of arenes Ar−H via C−H bond cleavage with activated arenes Ar′−m[3a,4a−e] or Ar′−X[3b,c,4a−f] affords an advanced route to biaryls. A more versatile transformation for biaryl synthesis would be oxidative cross-coupling between Ar−H and Ar′−H.[4] After the pioneering work for the Pd-catalyzed formation of $C(sp^2)-C(sp^2)$ bonds via double C−H bond cleavage,[5] such

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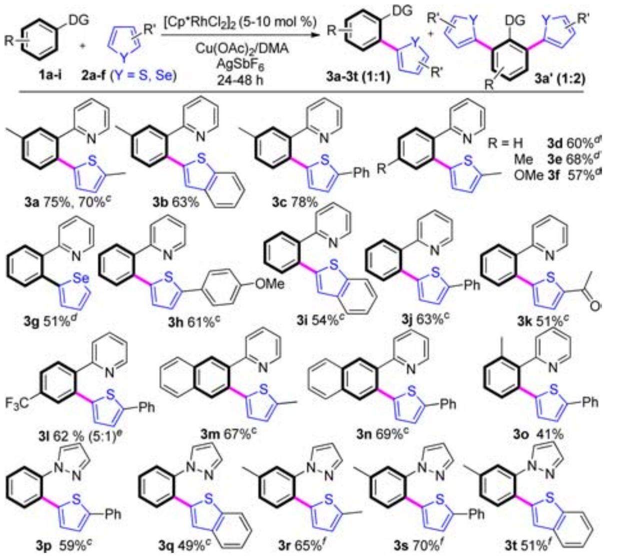
transformations have been extensively studied using Pd as the catalyst and have become useful synthetic tools for the construction of biaryl units.[4,6,7] Recently, this synthetic strategy for connecting aryl rings has been expanded to other metals such as copper,[8] rhodium,[9] and ruthenium.[10] These successful developments prompted us to study the Rh-catalyzed intermolecular[11] oxidative cross-coupling of heteroarenes with chalcogenophenes.[12] Several heterocyclic derivatives such as oxazoles,[7h,8c−e,12] thiazoles,[7h,8d,12] imidazoles,[8c,d,12] furans,[7c,f,9c,9d,12] thiophenes,[7i,9c,d,12] and pyrroles[7a−d,f,8e,12b] have been studied to achieve C−H bond functionalization. However such a transformation of selenophenes and unsubstituted thiophene[13] has not been accomplished to date. Herein we summarize our results on the Rh-catalyzed oxidative cross-coupling of arenes and heteroarenes with chalcogenophenes.

Initially, the reaction of 2-(*m*-tolyl)pyridine (**1a**) with 2-methylthiophene (**2a**) was chosen as a model reaction to optimize the reaction conditions.[14] After screening several parameters, it was revealed that the desired cross-coupling product was formed in a high yield when 10 mol % of $[Cp^*RhCl_2]_2$ was employed as a catalyst in combination with $Cu(OAc)_2$ (2.8 equiv) and $AgSbF_6$ (10 mol %) at 140 °C for 24 h. The desired cross-coupling products were obtained in almost similar yields with 5 mol % of $[Cp^*RhCl_2]_2$ along with $Cu(OAc)_2$ (3.2 equiv) and $AgSbF_6$ (20 mol %) as the oxidant at 140 °C for 48 h. Under the optimized conditions, the reaction of 2-phenylpyridine and *N*-phenylpyrazole derivatives with various thiophenes and selenophene was examined to explore the scope of this rhodium-catalyzed intermolecular oxidative cross-coupling. As shown in Scheme 1, these reactions provided the corresponding cross-coupling products in moderate to good yields. The reaction of 2-(*m*-tolyl)pyridine (**1a**) with 2-methylthiophene (**2a**) afforded the corresponding coupling product **3a** in 75% yield. In this reaction, starting material **1a** was completely consumed. Simple 2-phenylpyridine (**1b**) reacted with 2-methylthiophene (**2a**) to afford the corresponding coupling product **3d** in 60% yield. Electron-rich

Scheme 1. Rhodium-Catalyzed Oxidative Cross-Coupling of Pyridinyl and Pyrazole Directing Groups[a,b]



[a] Reaction conditions: **1a−i** (0.25 mmol), **2a−f** (0.75 mmol), $[Cp^*RhCl_2]_2$ (10 mol %), $Cu(OAc)_2$ (2.8 equiv), $AgSbF_6$ (10 mol %), and DMA (1.0 mL), 24 h, 140 °C. [b] Yields of isolated products. [c] $[Cp^*RhCl_2]_2$ (5 mol %), $AgSbF_6$ (20 mol %), $Cu(OAc)_2$ (3.2 equiv), DMA (1.0 mL), 48 h, 140 °C. [d] 5−10% of **3′** was observed by ^1H NMR. [e] Isolated as mixture (ratio was determined by ^1H NMR). [f] Without $AgSbF_6$.

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(11) Intermolecular aryl−aryl, heteroaryl−heteroaryl, and aryl−heteroaryl cross-couplings catalyzed by Rh have been reported by Glorius in refs 9b, 9c, and 9d, respectively. In ref 9c, several examples of the reaction of thiophenes using $Cu(OAc)_2$ as the oxidizing reagent are presented.

(12) During preparation of our manuscript, two similar reports appeared: (a) Dong, J.; Long, Z.; Song, F.; Wu, N.; Guo, Q.; Lan, J.; You, J. *Angew. Chem., Int. Ed.* **2013**, *52*, 580. (b) Qin, X.; Liu, H.; Qin, D.; Wu, Q.; You, J.; Zhao, D.; Guo, Q.; Huang, X.; Lan, J. *Chem. Sci.* **2013**10.1039/C3SC22241A.

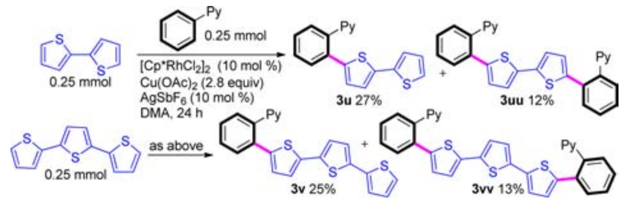
(13) This is partly because thiophene tends to undergo oligomerization. See for instance ref 9c.

(14) See Supporting Information.

phenylpyridine derivatives carrying an Me or OMe substituent gave similar results and produced desired products **3e** and **3f** in 68% and 57% yields, respectively. The reaction of 2-phenylpyridine with various thiophene derivatives and selenophene gave the desired products **3g**−**k** in moderate to good yields, along with a trace amount of the corresponding 1:2 coupling products **3′**. However, the reaction between 2-(4-(trifluoromethyl)phenyl)pyridine with 2-phenylthiophene gave a mixture of **3l** and **3l′** with a 5:1 ratio in a total yield of 62%. 2-Naphthylpyridine reacted site selectively with thiophenes at the less congested position to give **3m** and **3n** in good yields as sole coupling products. The reaction of *ortho*-substituted phenylpyridine with phenylthiophene gave the desired product in moderate yields, and *meta*-substituted phenylpyridine derivatives did not affect the reaction giving rise to **3a**−**c** in good yields. The reaction of *N*-phenylpyrazole with various thiophene derivatives gave the desired products **3p**−**q** in moderate to good yields, along with a trace amount of the corresponding 1:2 coupling products **3′**. The cross-coupling of 1-(*m*-tolyl)-1*H*-pyrazole with thiophenes proceeded to afford the corresponding products **3r**−**t** in 51% to 70% yields.

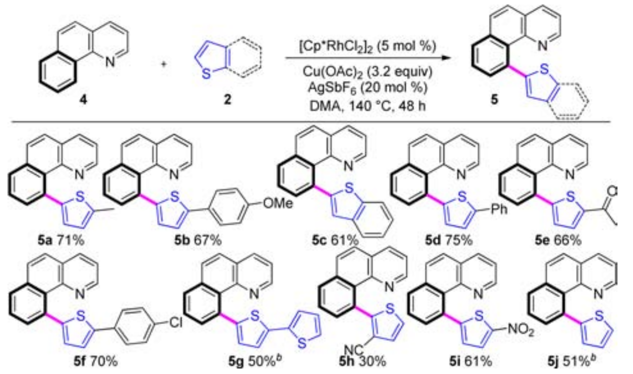
Oligothiophene derivatives were synthesized directly via double C−H bond cleavage using the present catalyst. Intermolecular oxidative cross-coupling of 2,2′-bithiophene and 2,2′:5′,2″-terthiophene with 2-phenylpyridine gave coupling products **3u** and **3v** as the major products and **3uu** and **3vv** as the minor products as shown in Scheme 2. Poly- and oligoarene compounds involving a thiophene unit have attracted much attention as the organic components of electronic devices[15] and fluorescent materials.[16] The present catalyst provides a straightforward route to access such oligothiophenes.

Scheme 2. Synthesis of Oligothiophene



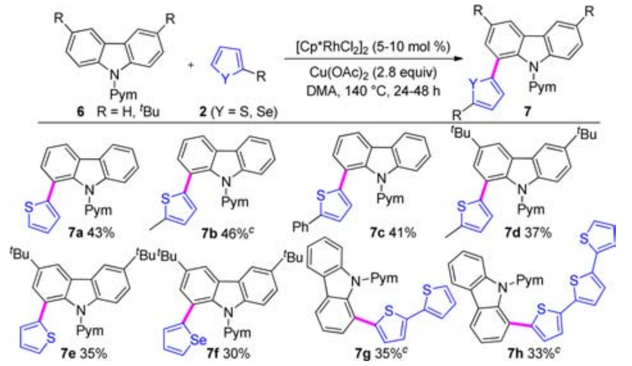
Benzo[*h*]quinoline (**4**) was also employed for this transformation, and the results are shown in Scheme 3. The reaction between **4** with various substituted thiophenes gave the corresponding coupling products in good yields (Scheme 3). The reaction of **4** with thiophene-3-carbonitrile gave the desired product **5h** in low yield due to the steric hindrance. The reaction of benzoquinoline with 2,2′-bithiophene and thiophene gave **5g** and **5j** as the major

Scheme 3. Coupling of Benzo[*h*]quinoline with Thiophenes[a]



[a] Reaction conditions: **4** (0.25 mmol), **2a**−**j** (0.75 mmol), $[\mathrm{Cp^*RhCl_2}]_2$ (5 mol %), $\mathrm{Cu(OAc)_2}$ (3.2 equiv), $\mathrm{AgSbF_6}$ (20 mol %), and DMA (1.0 mL), 48 h, 140 °C, isolated yields. [b] $[\mathrm{Cp^*RhCl_2}]_2$ (10 mol %), $\mathrm{Cu(OAc)_2}$ (2.8 equiv), $\mathrm{AgSbF_6}$ (10 mol %), 24 h.

Scheme 4. Coupling of Carbazoles **6** with Chalcogenophenes **2**[a,b]



[a] Reaction conditions: **6** (0.25 mmol), **2** (1.0 mmol), $[\mathrm{Cp^*RhCl_2}]_2$ (10 mol %), $\mathrm{Cu(OAc)_2}$ (2.8 equiv), and DMA (2.0 mL), 24 h, 140 °C. [b] Yields of isolated products. < 5% of 1:2 coupling product **7′** was observed in crude mixture by ^{1}H NMR. [c] $[\mathrm{Cp^*RhCl_2}]_2$ (5 mol %), $\mathrm{Cu(OAc)_2}$ (3.2 equiv), $\mathrm{AgSbF_6}$ (20 mol %), DMA (2.0 mL), 48 h, 140 °C. Pym: pyrimidin-2-yl.

coupling products, and a trace amount of 1:2 coupling products was formed (Scheme 3, **5g** and **5j**).

After these successful results for phenylpyridine, *N*-phenylpyrazole, and benzo[*h*]quinoline derivatives, we applied this catalytic reaction to 9-(pyrimidin-2-yl)-9*H*-carbazole (**6**) and found that it reacted with thiophene derivatives to give moderate yields of products under similar conditions (Scheme 4, **7a**−**7c**). Reaction of the 3,6-di-*tert*-butyl derivative with thiophenes and a selenophene led to a slight decrease in yields (Scheme 4, **7d**−**7f**). The reaction of selenophene with **6b** gave the corresponding coupling product **7f** in 30% yield. The reaction of oligothiophene derivatives with **6a** gave the desired cross-coupling products in 35% and 33%

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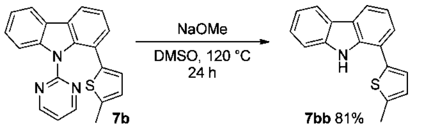
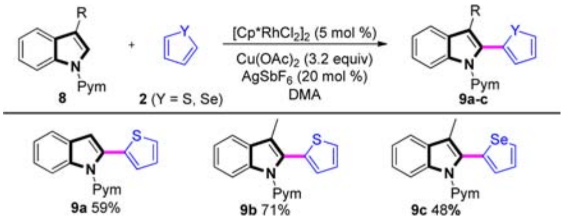
Scheme 5. Removal of 2-Pyrimidyl Directing Group

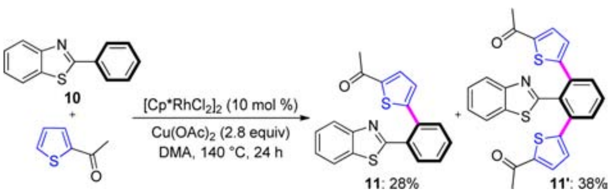


Scheme 6. Coupling of Indole **8** with Chalcogenophenes **2**[a,b]



[a] Reaction conditions: **8** (0.25 mmol), **2** (0.75 mmol), [Cp*RhCl$_2$]$_2$ (5 mol %), Cu(OAc)$_2$ (3.2 equiv), AgSbF$_6$ (20 mol %), and DMA (1.0 mL), 24 h, 140 °C. [b] < 5% of **9aa**−**cc** was observed by ^{1}H NMR.

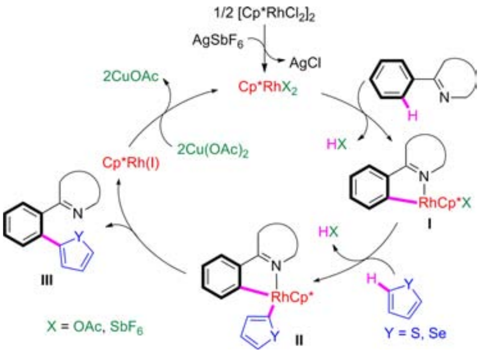
Scheme 7. Coupling of 2-Phenylbenzo[*d*]thiazole (**10**) with **2**



yields, respectively (Scheme 4, **7g**−**7h**). The directing group could be removed from the coupling products (Scheme 5).[14]

Reactions of 1-(pyrimidin-2-yl)-1*H*-indole derivatives **8** and 2-phenylbenzo[*d*]thiazole (**10**) with chalcogenophenes were also tested, and the results are shown in Schemes 6 and 7, respectively. Thiophene readily reacted with 1-(pyrimidin-2-yl)-1*H*-indole (**8a**) to give the desired cross-coupling product in 59% yield (Scheme 6, **9a**). Substitution with an electron-donating Me group at the 3-position of indole improved the reaction efficiency (Scheme 6, **9b**). Selenophene reacted with **8b** to give the desired product in 48% yield (Scheme 6, **9c**). The reaction of **10** with 2-acetylthiophene gave a mixture of **11** and **11′** with a nearly 1:1.4 ratio under the conditions employed as shown in Scheme 7.

Scheme 8. A Plausible Mechanism for the Reaction of Rhodium-Catalyzed Oxidative Cross-Coupling Reaction



A plausible reaction pathway is illustrated in Scheme 8. The Rh complex would be activated by AgSbF$_6$ to generate the electrophilic cationic complex.[9a−d] The catalytic cycle starts initially where arene reacts with the thus formed Cp*Rh(III) to generate rhodacyclic intermediate **I** stabilized by N−Rh coordination.[9b,12,17] Subsequently, intermediate **I** reacts with chalcogenophenes to provide intermediate **II**, which undergoes reductive elimination to generate the product **III** and an Rh(I) species, and the latter is oxidized by Cu(II) to complete the catalytic cycle.

In conclusion, we have developed a novel and efficient Rh(III)-catalyzed intermolecular oxidative cross-coupling of directing-group-containing (hetero)arenes with chalcogenophenes including oligothiophenes. This protocol exhibits a broad substrate scope with respect to (hetero)arenes such as phenylpyridine, *N*-phenylpyrazole, benzoquinoline, carbazole, indole, and 2-phenylbenzothiazole derivatives, and the yields are moderate to good.

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Supporting Information Available. Full experimental details, characterization data, ^{1}H and ^{13}C NMR spectra for new products. This material is available free of charge via the Internet at http://pubs.acs.org.

The authors declare no competing financial interest.