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Rhodium-catalyzed direct coupling of biaryl pyridine derivatives with internal alkynes†

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Axially chiral biaryls were synthesized by an isoquinoline or 2-pyridine-directed Rh(III)-catalyzed dual C–H cleavage and coupling with internal alkynes in good to excellent yields. Oxidation of isoquinoline derivatives with *m*-CPBA furnished their corresponding *N*-oxides, which could be utilized as Lewis base catalysts in asymmetric reactions.

Axially chiral biaryl units are embedded in many important natural products[1] and widely applied as chiral auxiliaries, ligands and catalysts in asymmetric syntheses.[2] The rapidly increasing interest in axially chiral biaryls has led to the development of a great variety of successful methods for their atroposelective construction.[3] However, the vast majority of these methods are focused on the synthesis of axially chiral biaryls *via* Suzuki couplings.[4] Some other approaches involve the desymmetrization of prochiral biaryl compounds,[5] atroposelective cleavage of the biaryl lactones with chiral nucleophiles,[6] asymmetric oxidative coupling of 2-naphthalenol derivatives[7] and asymmetric [2+2+2] cycloaddition of an α,ω-diyne and monoalkynes.[8] As an alternative method for atroposelective biaryl synthesis, the functionalization of achiral biaryl compounds *via* C–H bond cleavage is highly efficient,[9] but less explored. In this context, Murai and co-workers reported the atroposelective alkylation of naphthyl pyridines and naphthyl isoquinolines by a Rh(I)-catalyzed C–H activation reaction.[10] The same group also described a Ru-catalyzed silylation of 2-(1-naphthyl)-3-methylpyridine in 2003.[11] Later Lassaletta and co-workers reported Ir(III)-catalyzed nitrogen-directed borylations of 2-arylpyridines and 1-arylisoquinolines.[12] Pd(II)-catalyzed intermolecular C–H phosphorylation of 1-(naphthalen-1-yl)isoquinoline was reported by Yu and co-workers in 2013.[13] In addition, by taking advantage of a chiral sulfoxide moiety as the directing group and chiral auxiliary, the group of Colobert realized the direct atropodiastereoselective C–H olefination of biaryl compounds.[14] Very recently Yang and co-workers succeeded in palladium-catalyzed C–H acetoxylation of optically pure 2-diphenylphosphine oxide-1,1′-binaphthyl, which could lead to the synthesis of (*R*)-MeO-MOP.[15] Despite the above elegant methods for the construction of atroposelective scaffolds, novel approaches to build axially chiral biaryls are still highly demanded.

Usually, increasing the steric hindrance of axially chiral biaryls could have an enormous impact on their performance in asymmetric catalysis.[16] However, the introduction of arylated naphthalene and anthracene units to axially chiral biaryl scaffolds is far from developed. Although numerous methods for construction of polyarylatedarenes have been developed in the past decades,[17] transition metal-catalyzed C–H bond activation in an aromatic substrate followed by coupling with alkynes has been considered as a particularly useful tool.[18] In this regard, Miura, Sato and co-workers reported an effective aromatic homologation by Rh(III)-catalyzed oxidative annulations of the phenylazoles and 2-phenylpyridine with diarylacetylenes.[19] Herein, we report the synthesis of axially chiral biaryl compounds from 2-arylpyridines or 1-arylisoquinolines and internal alkynes through Rh(III)-catalyzed dual C–H functionalization/cycloaromatization.

Our studies commenced with the reaction between 1-(naphthalen-1-yl)isoquinoline **1a** and diphenyl acetylene **2a** using $[RhCp^*Cl_2]_2$ as the catalyst and $Cu(OAc)_2$ as the oxidant (Table 1). The examination of several silver salts revealed that $AgSbF_6$ is optimal (for detailed studies, see the ESI†). The desired product **3a** was obtained in 99% yield (entry 1). After acidification of **3a**, the structure of protonated **3a** was determined by single crystal X-ray diffraction analysis (see the ESI† for details). The presence of 1 equiv. of $Cu(OAc)_2$ was enough for this reaction, affording **3a** in 99% yield (entry 2). Other oxidants such as benzoquinone or oxygen were ineffective (entries 3 and 4). The choice of solvent was also crucial for this reaction. The reaction in *tert*-amyl alcohol gave the best result, in which **3a** was obtained in almost quantitative yield. Other solvents such as $ClCH_2CH_2Cl$, toluene, DMA and *t*BuOH were

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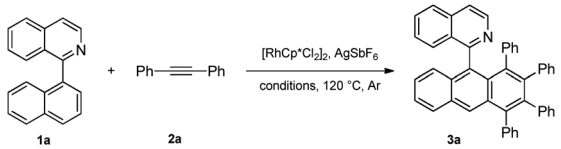
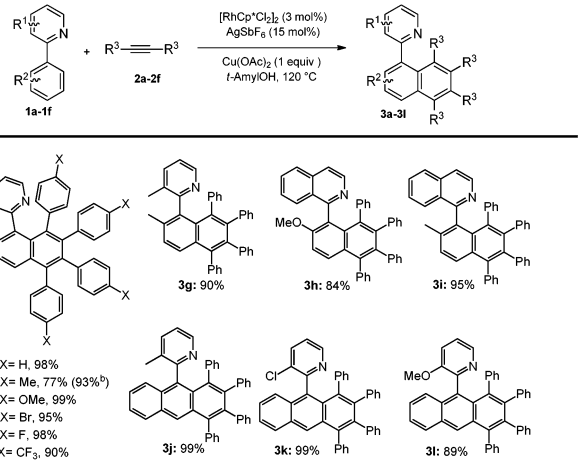
Table 1 Optimization of reaction conditions for biaryl construction *via* dual C–H bond cleavage[a]



Entry	Solvent	Oxidant	Yield[d] (%)
1[b]	*t*-AmylOH	$Cu(OAc)_2$	99
2	*t*-AmylOH	$Cu(OAc)_2$	99
3	*t*-AmylOH	BQ	Trace
4[g]	*t*-AmylOH	O_2	Trace
5	$ClCH_2CH_2Cl$	$Cu(OAc)_2$	32
6	Toluene	$Cu(OAc)_2$	77
7	DMA	$Cu(OAc)_2$	79
8	*t*-BuOH	$Cu(OAc)_2$	84
9[c]	*t*-AmylOH	$Cu(OAc)_2$	98
10[h]	*t*-AmylOH	$Cu(OAc)_2$	97
11[e]	*t*-AmylOH	$Cu(OAc)_2$	23
12[f]	*t*-AmylOH	$Cu(OAc)_2$	NR

[a] Unless otherwise noted, all reactions were carried out under the following conditions: $[RhCp^*Cl_2]_2$ (5 mol%), $AgSbF_6$ (25 mol%), **1a**/**2a**/$Cu(OAc)_2$ = 1/2.2/1, 0.1 mol L^{-1}, and *t*-amyl alcohol (1 mL) in a sealed tube at 120 °C for 12 h. [b] $Cu(OAc)_2$ (2 equiv.) was used. [c] 3 mol% $[RhCp^*Cl_2]_2$ and 15 mol% $AgSbF_6$ were used. [d] Isolated yield. [e] 1 mol% $[RhCp^*Cl_2]_2$ and 5 mol% $AgSbF_6$ were used. [f] W/O $[RhCp^*Cl_2]_2$. [g] 1 atm O_2 was used. [h] 2 mmol **1a** and 4.1 mmol **2a** were used.

Table 2 Rhodium(III) catalyzed cyclization of 2-arylpyridines and 1-arylisoquinolines (1) with alkynes (2)[a]



[a] Unless otherwise noted, all reactions were run in **1** (0.2 mmol), **2** (0.44 mmol), $[RhCp^*Cl_2]_2$ (3.0 mol%), $AgSbF_6$ (15.0 mol%), $Cu(OAc)_2$ (0.2 mmol) and *t*-amyl alcohol (2 mL) in a sealed tube at 120 °C. Isolated yields are reported. [b] Reaction was run on a 0.1 mmol scale with 5 mol% of $[RhCp^*Cl_2]_2$.

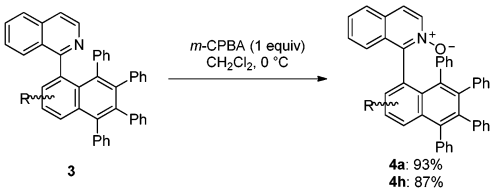
less effective (32–84% yields, entries 5–8). Finally, reducing the loading of $[RhCp^*Cl_2]_2$ from 5 mol% to 3 mol% led to the isolation of **3a** in 98% yield (entry 9). Furthermore, the reaction on a 2 mmol scale (**1a**) proceeded smoothly without notable erosion in yield (97%, entry 10). However, further reducing the loading of $[RhCp^*Cl_2]_2$ to 1 mol% resulted in a dramatically decreased yield (23% yield, entry 11). As expected, no reaction occurred in the absence of $[RhCp^*Cl_2]_2$ (entry 12). Thus, the optimal conditions were identified as the following: 3 mol% $[RhCp^*Cl_2]_2$, 15 mol% $AgSbF_6$, 2.2 equiv. of biphenyl acetylene, 1 equiv. of $Cu(OAc)_2$ in *tert*-amyl alcohol at 120 °C for 12 h, and under these conditions product **3a** was obtained in 98% yield.

Under the optimal reaction conditions described above, various substituted symmetrical alkynes (**2b–f**) were treated with 1-(naphthalen-1-yl)isoquinoline **1a** and gave the desired polyarylated anthracene products (**3b–f**) in excellent yields (Table 2). When 1,2-di-*p*-tolylethyne (**2b**) was used, **3b** was obtained in 77% yield. Using 5 mol% of $[RhCp^*Cl_2]_2$, product **3b** was obtained in 93% yield. Unfortunately, when 5-decyne and 1-phenyl-1-propyne were tested with 1-(naphthalen-1-yl)isoquinoline under the optimized reaction conditions, only a trace amount of the desired product was observed in both cases.

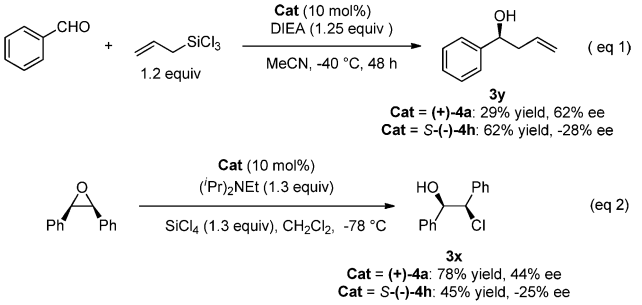
On the other hand, various substituted 2-arylpyridines or 1-arylisoquinolines (**1b–f**) reacted smoothly with diphenyl acetylene **2a** to give their corresponding polyarylated naphthalene and anthracene derivatives (**3g–l**) in good to excellent yields (Table 2).

The development of novel chiral pyridine *N*-oxides as Lewis base catalysts has been one active project in asymmetric synthesis.[20] To test the utility of products obtained here, the oxidation of **3a** and **3h** with *m*-CPBA furnished the desired pyridine *N*-oxides **4a** and **4h**, respectively, in good to excellent yields (Scheme 1). The enantiomers of **4a** and **4h** were easily separated by chiral preparative HPLC methods. Fortunately, the absolute configuration of product (+)-**4h** was assigned as *R* by an X-ray crystallographic analysis of a single crystal of the enantiopure sample (see the ESI† for details).

The enantiopure *N*-oxides **4a** and **4h** were tested in the asymmetric allylation of benzaldehyde with allyltrichlorosilane, as shown in Scheme 2. After a preliminary examination, both *N*-oxides **4a** and **4h** could catalyze this reaction but with moderate reactivity and enantioselective control. 1-Phenylbut-3-en-1-ol was obtained in 29% yield, 62% ee by using (+)-**4a** and 62% yield,



Scheme 1 Oxidation of **3a** and **3h**.



Scheme 2 Application of enantiopure *N*-oxides **4a** and **4h**.

28% ee by using *S*-(−)-**4h** (eqn (1)). These chiral *N*-oxides also sufficiently catalyzed the ring-opening reaction of *cis*-stilbene oxide with $SiCl_4$, affording the corresponding chlorohydrin with moderate ee values (eqn (2)). These enantiopure *N*-oxides were also found to be suitable catalysts for asymmetric addition of diethylzinc to benzaldehyde and allenylation of aldehydes with propargyl trichlorosilane, but with only moderate results (see the ESI† for details).

A plausible mechanism is proposed to account for the reaction of **1a** with alkyne **2a**. The catalytic cycle starts with the removal of chloride in $[RhCp^*Cl_2]_2$ by using $AgSbF_6$. The isoquinoline nitrogen of **1a** coordinates to the rhodium center, and subsequently the *ortho* C–H bond is cleaved to form a five-membered rhodacycle **I**. In the second step, insertion of alkyne **2a** into the rhodium–carbon bond gave rhodium species **II**. Then intermediate **II** undergoes further concerted-metallation–deprotonation[21] to afford intermediate **III**. After insertion of alkyne **2a** once again and subsequent reductive elimination, product **3a** is obtained and the reduced rhodium species can be oxidized by $Cu(OAc)_2$ to form the active catalyst (Scheme 3).[22]

In conclusion, we have demonstrated that the axially chiral biaryl compounds could be effectively constructed through a rhodium-catalyzed, chelating-assisted dual C–H functionalization/cycloaromatization reaction. In addition, these biaryl compounds could be easily converted to novel *N*-oxides, which were demonstrated to be suitable organocatalysts. Further applications of this method in ligand design, detailed mechanistic investigation, and the development of asymmetric reactions are currently underway in our laboratory.

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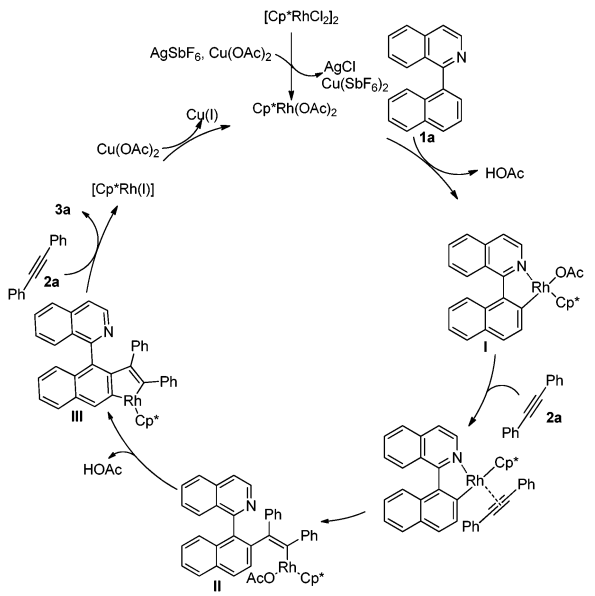

Scheme 3 Plausible mechanism for the reaction of **1a** with **2a**.

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