ACS Catalysis

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Ruthenium-Catalyzed Hydroarylations of Oxa- and Azabicyclic Alkenes

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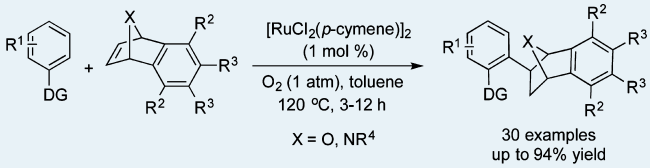
Supporting Information

ABSTRACT: A ruthenium-catalyzed arylation reaction of oxa- and azabicyclic alkenes with (hetero)arenes by C−H bond activation has been discovered. The reaction does not require additives and utilizes dioxygen in realizing the catalytic cycle leading to monosubstituted 7-oxa and 7-azabenzonorbornane derivatives.

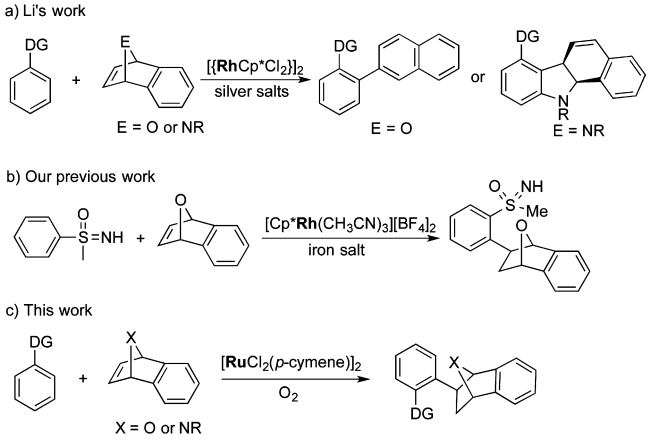


KEYWORDS: *ruthenium, alkylation, oxa- and azabicyclic alkenes, oxygen, C−H bond activation*

Transition-metal-catalyzed C−H bond functionalization[1] provides an effective access to natural products[2] and compounds with pharmaceutical relevance.[3] The use of molecules with directing groups (DG) allows the site-selective construction of C−C[4] and C−heteroatom bonds.[5−8] For the former transformations, the pioneering studies of arene-to-olefin additions leading to alkylated aromatic systems by Murai,[9] Chatani,[10] Bergman, Ellman,[11] Ackermann,[12] and Fagnou[13] have proven most stimulating because they opened new synthetic opportunities following atom-economical strategies.

Recently, Li reported rhodium(III)-catalyzed additions of arenes onto heterobicyclic alkenes in the presence of silver salts leading to ortho-naphthylated products and *cis*-fused dihydrocarbazole derivatives (Scheme 1a).[14] We applied the same bicyclic starting materials and achieved sulfoximine additions across the double bonds retaining the bicyclic scaffolds.[15] In addition, those C−H functionalization reactions were rhodium(III)-catalyzed, and in this case, the presence of $Fe(OAc)_2$ proved beneficial (Scheme 1b). In light of Ackermann's recent reports on ruthenium(II)-catalyzed couplings between linear alkenes and (hetero)arenes or pyrrolidines with potassium carboxylates and BINAP as cocatalytic additives,[16] we wondered about the effects of such ruthenium-based catalyst systems in the aforementioned cross-coupling reactions. The results of this investigation, which led to the development of hydroarylations of strained oxa- and azabicyclic alkenes without the necessity to add a metal salt (Scheme 1c), is reported here.

Scheme 1. Metal-Catalyzed C−H Bond Functionalizations of Oxa- and Azabicyclic Alkenes



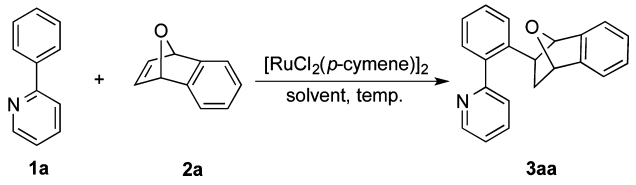
For the initial reactivity study, 2-phenylpyridine (**1a**) and oxabicyclic alkene **2a** were selected as representative starting materials. Using a catalyst system consisting of $[RuCl_2(p\text{-cymene})]_2$ (2 mol %) and $Cu(OAc)_2{\cdot}H_2O$ (2 equiv) in air gave a promising 10% of the desired alkylated product **3aa** (Table 1, entry 1).[17] The addition of $AgSbF_6$ (10 mol %) (with a concomitant solvent switch from toluene to dichloroethane) inhibited the catalysis (Table 1, entry 2). Combinations of 2 mol % of $[RuCl_2(p\text{-cymene})]_2$ and 20 mol % of AgOAc, Ag_2CO_3, or $Fe(OAc)_2$ led to unsatisfying results (Table 1, entries 3−5). The yield of **3aa** could be increased using $Fe(OAc)_2$ under oxygen instead of air (Table 1, entry 6).

Raising the temperature from 100 to 120 °C proved beneficial as well (Table 1, entry 7). That the ruthenium catalyst was needed was confirmed by a test experiment performed without $[RuCl_2(p\text{-cymene})]_2$ (Table 1, entry 8). To our surprise and contrasting previous observations made in the rhodium-catalyzed hydroarylations,[15] the reactions proceeded better in the absence of metal-based additives (Table 1, entries 9−13). The optimal result was obtained in a catalysis with 1 mol % of $[RuCl_2(p\text{-cymene})]_2$ in toluene at 120 °C under an

Received: February 6, 2015
Revised: March 23, 2015
Published: March 25, 2015

DOI: 10.1021/acscatal.5b00258
ACS Catal. 2015, 5, 2770−2773

Table 1. Optimization of Reaction Conditions for the Ru(II)-Catalyzed Hydroarylation[a]



1a 2a 3aa

entry	catalyst loading (mol %)	oxidant	additive	temp (°C)	yield (%)
1[b]	2.0	air	$Cu(OAc)_2·H_2O$	100	10
2[c]	2.0	air	$Cu(OAc)_2·H_2O$ $AgSbF_6$	100	0
3[d]	2.0	air	AgOAc	100	14
4[d]	2.0	air	Ag_2CO_3	100	20
5[d]	2.0	air	$Fe(OAc)_2$	100	21
6[d]	2.0	O_2	$Fe(OAc)_2$	100	35
7[d]	2.0	O_2	$Fe(OAc)_2$	120	48
8[d]	0	O_2	$Fe(OAc)_2$	120	0
9	2.0	O_2		120	61
10	5.0	O_2		120	10
11	**1.0**	**O_2**		**120**	**92**
12	0.5	O_2		120	74
13[e]	0.1	O_2		120	21
14	1.0	argon		120	5
15	1.0	air		120	35

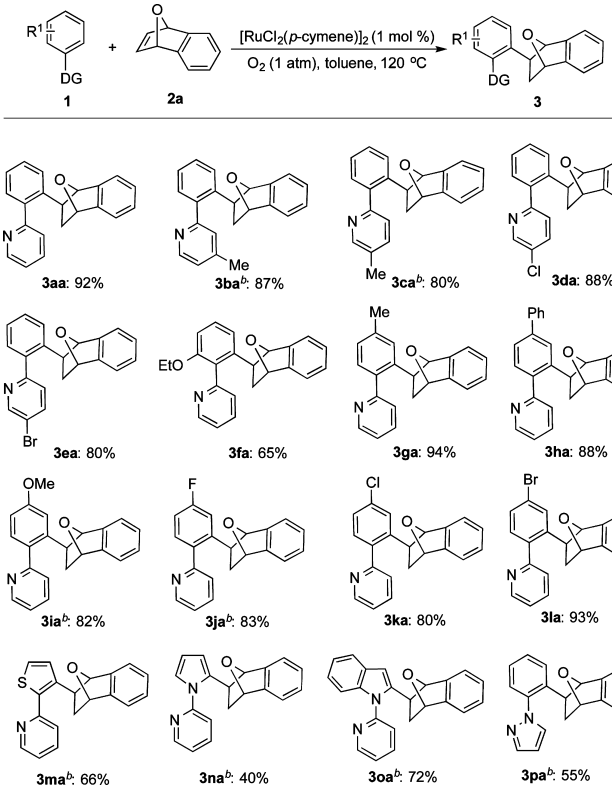
[a]Reaction conditions: **1a** (0.30 mmol), **2a** (0.60 mmol), $[RuCl_2(p\text{-cymene})]_2$ (*x* mol %) in dry toluene (1.5 mL) at indicated temperature for 3 h. [b]$Cu(OAc)_2·H_2O$ (2.0 equiv). [c]$Cu(OAc)_2·H_2O$ (2.0 equiv) and $AgSbF_6$ (10 mol %) in DCE (1.5 mL). [d]Reaction for 1 h with 20 mol % of additive. [e]Reaction time for 12 h.

atmosphere of dioxygen, which afforded **3aa** in 92% yield (Table 1, entry 11). Both raising and lowering the catalyst amount led to a decrease in yield of **3aa**.[18] Notably, the dioxygen atmosphere was crucial for the progression of the reaction as shown by experiments performed under argon and air, which led to **3aa** in only 5% and 35%, respectively (Table 1, entries 14 and 15).[19]

Under the optimized conditions (Table 1, entry 11) the hydroarylation of a number of substituted (hetero)arenes with **2a** as olefinic partner was studied next. As shown in Scheme 2, 2-arylpyridines with both electron-donating and -withdrawing groups on the arene reacted well affording the corresponding products (**3aa**−**3la**) in yields between 65% and 94%. The substrate palette included two picoline derivatives (**3ba**, **3ca**). The moderate yield in the formation of **3fa** (65%) was attributed to a steric compression induced by the *ortho*-ethoxy group on the arene. Pyridines with thiophenyl, pyrrolyl, and indolyl substituents gave products **3ma**, **3na**, and **3oa** in yields of 66%, 40%, and 72%, respectively. The structure of **3ma** was analyzed by single-crystal X-ray diffraction, which confirmed the formation of the *exo* product (for details, see Supporting Information). Finally, *N*-phenyl pyrazole was applied, which led to addition product **3pa** in 55% yield. 7,8-Benzoquinoline did not react with **2a**.

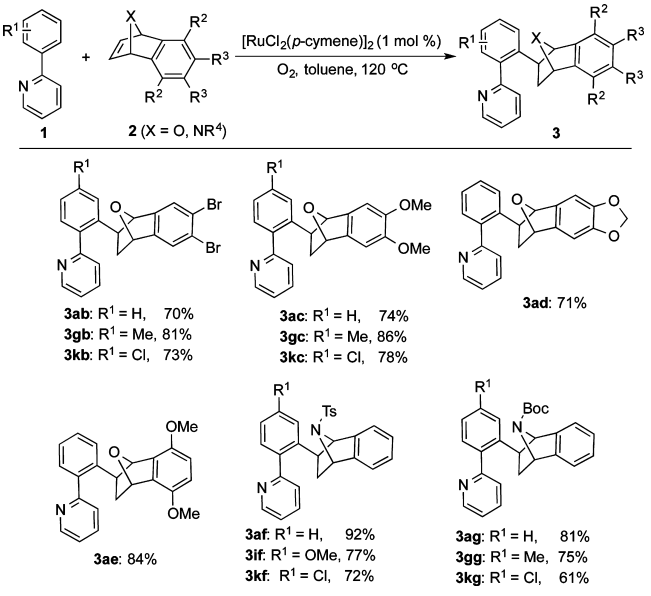
Subsequently, the olefinic component was varied, and additions of 2-arylpridines onto a range of oxa- and azabicyclic alkenes were examined (Scheme 3). In all cases, the reactions proceeded well affording the corresponding products in yields between 61% and 92%. Electronic effects on both the arene and the alkene were insignificant.

Scheme 2. Scope of (Hetero)Arenes[a]



[a]Reaction conditions: **1a** (0.30 mmol), **2a** (0.60 mmol), $[RuCl_2(p\text{-cymene})]_2$ (1 mol %) in dry toluene (1.5 mL) at 120 °C for 3−12 h. [b]0.80 mmol of **2a**.

Scheme 3. Scope of Oxa- and Azabicyclic Alkenes[a]



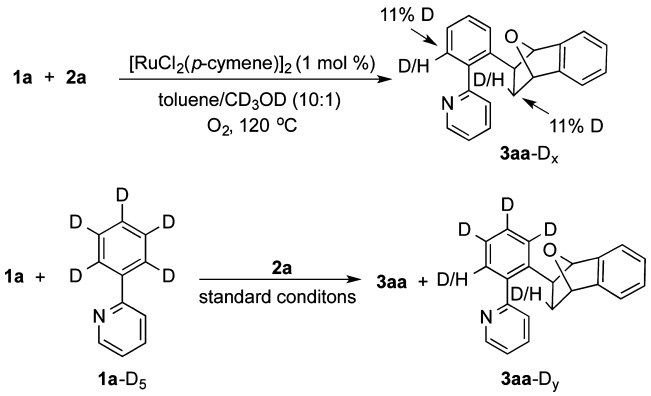
[a]Reaction conditions: **1a** (0.30 mmol), **2a** (0.60 mmol), $[RuCl_2(p\text{-cymene})]_2$ (1 mol %) in dry toluene (1.5 mL) at 120 °C for 6−12 h.

To probe the reaction mechanism, two experiments were carried out. First, hypothesizing that protonation events were relevant for the catalysis, **1a** was treated with alkene **2a** in a solvent system consisting of toluene and fully deuterated methanol (in a 10:1 ratio). As a result, partially labeled **3aa** with

DOI: 10.1021/acscatal.5b00258
ACS Catal. 2015, 5, 2770−2773

11% deuterium each at the arene and the newly formed alkyl substituent was obtained in 31% yield (Scheme 4, top). This

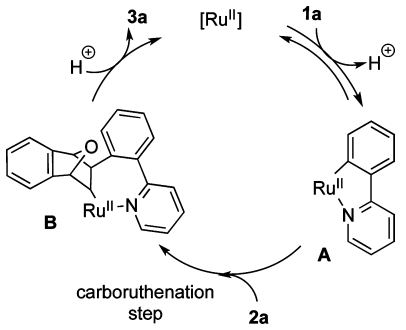
Scheme 4. Deuterium-Labeling Experiments



observation was interpreted as support for the proposed mechanistically relevant proton transfer and as indication for a reversible C−H bond metalation step. The latter was strengthened by an isotope analysis of the recovered starting material, which showed a significant deuterium incorporation into **1a** after the catalysis (see Supporting Information for details). In a second experiment, a mixture of 2-phenylpyridine (**1a**) and its isotopically labeled analogue **1a**-D_5 was subjected to the reaction with alkene **2a** (Scheme 4, bottom). From this catalysis, a kinetic isotope effect (KIE) of $k_H/k_D \approx 2.57$ was determined.[20,21]

On the basis of the aforementioned experimental evidence, we propose a mechanistic path as shown in Scheme 5. The

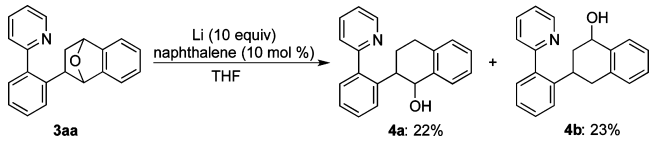
Scheme 5. Plausible Catalytic Cycle



ruthenium(II) catalyst inserts (reversibly) into the C−H bond of the arene, which upon loss of a proton forms ruthenacycle **A**.[22] The reaction with the bicyclic olefin (carboruthenation) leads to a new ruthenium complex **B**. Protonation of **B** provides the product and regenerates the initial ruthenium species, which reenters the catalytic cycle. This scenario explains the formation of the hydroarylation product, but a few facts remain obscure. First, it is surprising that no additive is required for the catalyst activation, as needed by Ackermann in his ruthenium-catalyzed carboxylate-assisted olefin hydroarylation reactions.[16] Second, the significant activation effect by dioxygen, which appears critical for the entire catalysis, is not accounted for. Both effects deserve attention in subsequent more detailed mechanistic analyses.[23,24]

To illustrate the synthetic applicability of the hydroarylation products, a derivatization of **3aa** was conducted (Scheme 6). In

Scheme 6. Synthesis of 1,2,3,4-Tetrahydronaphthalen-1-ols



the presence of lithium metal and naphthalene, **3aa** underwent reductive cleavage of the carbon−oxygen bond in tetrahydrofuran to give 1,2,3,4-tetrahydronaphthalen-1-ols **4a** and **4b** in 22% and 23% yields, respectively. As partially hydrogenated naphthalenes have attracted much attention,[25] we can envision applications of this methodology in medicinal chemistry.[26]

In summary, we developed a ruthenium-catalyzed C−H bond activation leading to additions of (hetero)arenes onto bicyclic olefins. As a result, synthetically useful 7-oxa and 7-azabenzonorbornanes are obtained that can be functionalized further. Interesting features are that no additives are required for the catalyst activation and that dioxygen plays a decisive, still to be uncovered role.

■ ASSOCIATED CONTENT

Supporting Information

The following file is available free of charge on the ACS Publications website at DOI: 10.1021/acscatal.5b00258.

Experimental details, characterizing data of compounds **3** and **4**, deuterium-labeling experiments, NMR spectra, X-ray crystal structure and data of **3ma** (PDF)

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

H.C., W.D., and Q.G. are grateful to the China Scholarship Council (CSC) for predoctoral stipends. S.D. acknowledges support by the Alexander von Humboldt Foundation. We thank Dr. Kanniyappan Parthasarathy for supplying 2-phenylpridine-D_5.

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(17) The aerobic atmosphere was chosen as it had proven suitable in the previously studied rhodium catalysis (ref 15).

(18) At catalyst loadings of 5 mol% and 2 mol%, the reactions were very fast, leading to a decomposition of **2a**. Phenylpyridine (**1a**) could be recovered. Degradation of **2a** was also observed when the reaction was performed in the absence of **1a** using 5 mol % of $[RuCl_2(p\text{-cymene})]_2$ at 120 °C for 3 h under dioxygen.

(19) Substituting $[RuCl_2(p\text{-cymene})]_2$ by $RuCl_2(PPh_3)_3$ (2 mol %) and $[Ru(CO)_3Cl_2]_2$ (1 mol %) gave **3aa** in 38% and 21% yield, respectively. No reaction was observed with $Ru(CO)_2Cl_2(PPh_3)_2$. Neither acetophenone nor *N*-phenylbenzaldimine could be applied instead of **1a** as coupling partner for **2a** under standard reaction conditions.

(20) (a) For a recent important paper asking for precautions when interpreting the H/D exchange reactions, see: Munz, D.; Webster-Gardiner, M.; Fu, R.; Strassner, T.; Goddard, W. A.; Gunnoe, T. B. *ACS Catal.* **2015**, *5*, 769−775. (b) For an important recent summary on the use of KIE, see: Simmons, E. M.; Hartwig, J. F. *Angew. Chem., Int. Ed.* **2012**, *51*, 3066−3072.

(21) The determination of the KIE values was complicated in this case as ESI MS indicated that product **3aa**-Dy had undergone multiple H/D exchange reactions which hampered the precise analysis by NMR spectroscopy.

(22) For an important kinetic study, see: Flegeau, E. F.; Bruneau, C.; Dixneuf, P. H.; Jutand, A. *J. Am. Chem. Soc.* **2011**, *133*, 10161−10170.

(23) Heating of $[RuCl_2(p\text{-cymene})]_2$ under an atmosphere of dioxygen for 1 h at 120 °C did not seem to affect the catalyst as suggested by ESI MS analysis. Changing the oxidant from dioxygen to di-*tert*-butylperoxide (DTBP, 2 equiv.) gave **3aa** in 23% yield. With *tert*-butylhydroperoxide (TBHP) as oxidant, no reaction occurred. The addition of TEMPO (2 equiv) to a reaction under standard conditions led to an inhibition of the catalysis affording **3aa** in <10% yield.

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DOI: 10.1021/acscatal.5b00258
ACS Catal. 2015, 5, 2770−2773