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Redox-Neutral Access to Isoquinolinones via Rhodium(III)-Catalyzed Annulations of *O*-Pivaloyl Oximes with Ketenes

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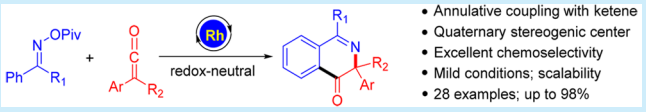
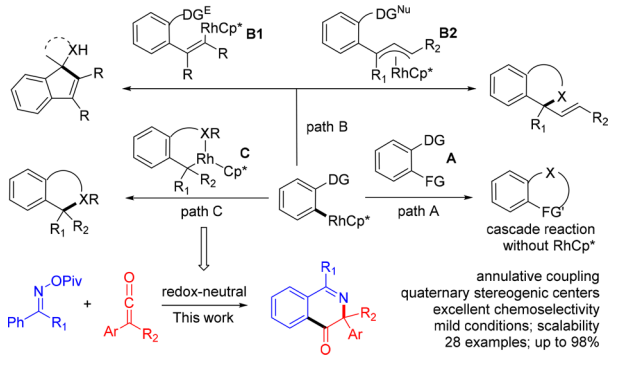
Supporting Information

ABSTRACT: A mild and redox-neutral [4 + 2] annulation of *O*-pivaloyl oximes with ketenes has been realized for synthesis of quaternary-carbon-stereocenter-containing (QCSC) isoquinolinones, where the *N*-OPiv not only acts as an oxidizing group but also offers coordination saturation to inhibit protonolysis. The reaction mechanism has been studied by DFT calculations.



In the past several decades, metal-catalyzed direct C−H activation of arenes has witnessed tremendeous progress.[1] In particular, C−H activation and annulation catalyzed by Cp*Rh(III) complexes has emerged as a powerful strategy to construct complex cyclic scaffolds in a streamlined and step-economical fashion.[2] Among those cyclic scaffolds, the synthesis of cyclic products bearing a quaternary carbon stereocenter, which provides important bioactive molecules for biological studies,[3] has been realized. To date, the synthetic strategies can be classified into three categories on the basis of the intrinsic differences in reaction pathways. In the first category (Scheme 1, path A), following the Rh(III)-catalyzed

Scheme 1. Construction of Stereogenic Quaternary Centers via C−H Activation and Annulation



ortho C−H activation that gives an organorhodium intermediate, the Rh−C bond underwent functionalization by a coupling partner to give a multifunctional organic intermediate **A**. Subsequently, intramolecular cyclization/functionalization took place to give a quaternary-carbon stereocenter without the participation of any rhodium species.[4] In the second category (path b), the quaternary-carbon stereocenter was accessed either through nucleophilic attack of a Rh−C species at the (electrophilic) directing group (**B1**)[5] or by the nucleophilic attack of a directing group at a proximal (electrophilic) π-allyl group (**B2**),[6] where the allyl moiety was generated via insertion of an alkyne or allene in C−H activation ststems. In the third strategy (path c),[7] the organorhodium intermediate undergoes migratory insertion into an unsaturated partner to deliver a sterically congested Rh−C(tertiary) species (**C**). Subsequently, the tertiary carbon and an anionic group reductively eliminate from **C** to deliver the final product. Despite these achievements, the coupling partners are mostly limited to reactive diazo compounds and alkynes.

Ketenes ($R^1R^2C{=}C{=}O$) are reactive reagents in synthetic and polymer chemistry.[8] In particular, ketenes have been widely used in the synthesis of complex carbonyl compounds in metal-catalyzed addition and cross-coupling reactions. Despite their high activity, the corresponding catalytic transformations in C−H activation chemistry largely lag behind. The insertion of arene C−H bonds into polar unsaturated bonds via C−H activation has been well-studied,[9] and we have also successfully achieved acylation of arenes via insertion of a C−H bond into the C=C bond of ketenes under redox-neutral conditions.[10] Recently, the Zeng group reported cyclization of *N*-nitrosoanilines with α-diazo-β-ketoesters through a cobalt(III)-catalyzed C−H activation/Wolff rearrangement, in which a ketene was established as a key intermediate via a Co-mediated rearrangement of carbenes.[11] Very recently, we also realized oxidative [4 + 2] annulation of ketenes with 2-phenylindoles, a specific class of arene, by taking advantage of the high reactivity of the C(3) of the indole.[10b] However, oxidative conditions are required. Encouraged by these reports, we aimed to further explore redox-neutral synthesis of quaternary-carbon-stereocenter-containing (QCSC) heterocycles via reductive elimination that involves a sterically hindered enolate tertiary carbon (path C). To achieve this annulation, several challenges nevertheless

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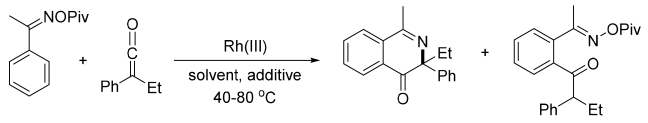
exist: (1) the annulation requires sufficient suppression of protonolysis[12] of the Rh(III)-C(tertiary) species and (2) reductive elimination involving a sterically congested tertiary carbon generated from the insertion of ketene into a Rh−C(aryl) bond generally requires a higher activation barrier. Given the electrophilic nature of ketenes, as well as their tendency toward dimerization or decomposition, introduction of additives or high temperature may accelerate background reactions. We now report the Rh(III)-catalyzed efficient synthesis of QCSC isoquinolinones using ketene as a C_2 synthon under redox-neutral conditions.

To ensure the high activity and selectivity of arenes and to realize annulative coupling under the relatively simple, redox-neutral conditions, our strategy is to take advantage of the coordinating and oxidizing nature of an *N*-pivaloxy directing group.[7,13] The chelation of the pivaloyl oxygen saturates the coordination sphere, so as to inhibit undesired protonation, as well as to accelerate reductive elimination.[7,13] Thus, we initiated our studies by reacting *O*-pivaloyl acetophenone oxime **1a** with ethyl phenyl ketene **2a** under redox-neutral conditions with $[Cp^*RhCl_2]_2/AgSbF_6$ as a catalyst (Table 1). No reaction took place until a base with a small ionic radius was employed (entries 1−6). Among those bases, NaOAc gives a good yield

Table 1. Optimization Studies[a]



1a 2a 3aa 4aa

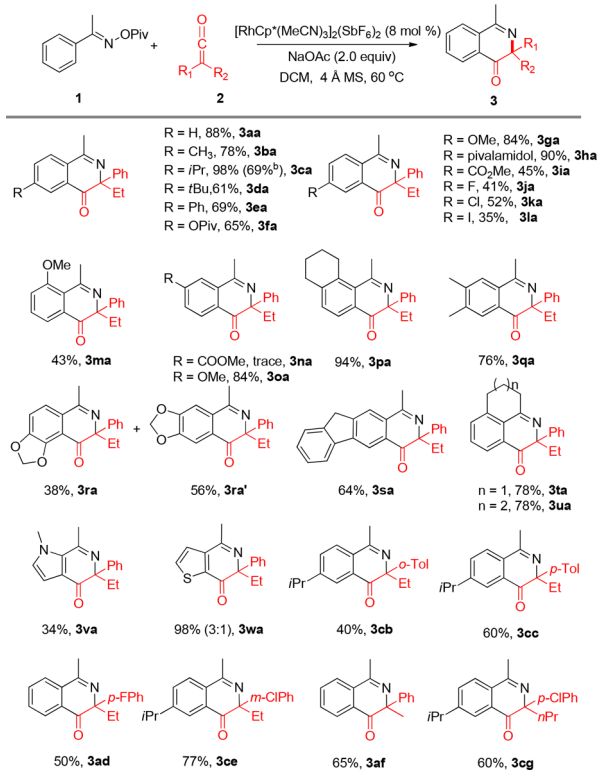
entry	additive	solvent	temp (°C)	yield (%)[b] 3aa	4aa
1	CsOAc	DCE	60	ND	
2	KOAc	DCE	60	ND	
3	NaOPiv	DCE	60	trace	
4	NaOAc	DCE	60	70	<5
5	Na_2CO_3	DCE	60	62	0
6	LiOAc	DCE	60	75	12
7	NaOAc	DCE	80	60	trace
8	NaOAc	DCE	40	60	13
9[c]	NaOAc	DCE	60	66	trace
10[d]	NaOAc	DCE	60	59	<5
11	LiOAc	DCM	60	72	10
12	NaOAc	DCM	60	82	<5
13	NaOAc	CH_3CN	60	<5	0
14	NaOAc	$PhCH_3$	60	23	20
15	NaOAc	THF	60	10	0
16	NaOAc	1,4-dioxane	60	72	<5
17[e]	NaOAc	DCM	60	NR	
18[f]	NaOAc	DCM	60	82	<5
19[fg]	NaOAc	DCM	60	86	<5
20[h]	NaOAc	DCM	60	ND	ND
21[i]	NaOAc	DCM	60	28	<5
22[j]	NaOAc	DCM	60	48	<5

[a]Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), $[RhCp^*Cl_2]_2$ (4 mol %), $AgSbF_6$ (16 mol %), base (0.4 mmol), solvent (2.0 mL) under N_2 for 15 h. [b]Isolated yield after column chromatography. [c]PivOH (0.2 equiv). [d]AgOAc (0.2 equiv). [e]No rhodium or $AgSbF_6$ was used. [f]$[RhCp^*(MeCN)_3]_2(SbF_6)_2$ (8 mol %) was used instead of $[RhCp^*Cl_2]_2/AgSbF_6$. [g]4 Å MS (50 mg) was introduced. [h]Acetophenone oxime was used instead of **1a**. [i]*O*-Acetyloxime was used. [j]*O*-Benzoyl oxime was used.

of the desired annulation product (**3aa**), together with the simple acylation side product **4aa** (entries 4−6). In comparison, lowering or increasing the reaction temperature all resulted in inferior results (entries 7, 8). Introduction of PivOH and AgOAc also decreased the reaction efficiency (entries 9, 10). DCM seems to the optimal solvent among the various solvents examined (entries 12−16), and control experiments verified that both $[RhCp^*Cl_2]_2$ and $AgSbF_6$ were necessary (entry 17). Furthermore, replacement of the catalyst by $[RhCp^*(MeCN)_3]_2(SbF_6)_2$ improved the yield to 86% in the presence of 4 Å molecular sieves (MS, entries 18−19). Examination of the oxime moiety revealed that simple oxime or other oxime esters only coupled with poor efficiency under the optimized conditions (entries 20−22), and these results were consistent with our hypothesis.

With the optimized conditions in hand, the scope of annulation between *O*-pivaloyl oximes and ketenes was then explored (Scheme 2). Introduction of diverse electron-

Scheme 2. Scope of Coupling of *O*-Pivaloyl Oximes with Ketenes[a]



[a]Conditions: arene (0.2 mmol), ketene (0.4 mmol), $[RhCp^*(MeCN)_3]_2(SbF_6)_2$ (8 mol %), NaOAc (0.4 mmol), 4 Å MS (50 mg), and DCM (2.0 mL) at 60 °C for 15 h. [b]The reaction was conducted at a 4 mmol scale with $[RhCp^*Cl_2]_2/AgSbF_6$ (4 mol %/16 mol %).

donating, -withdrawing, and halogen groups into the *para* position of the acetophenone *O*-pivaloyl oxime was fully tolerated (**3aa**−**3la**, 35−98%) although the reaction efficiency was obviously lower with installation of electron-withdrawing and halogen groups. In addition, a gram-scale synthesis of product **3ca** has been realized in 69% yield. Oxime esters bearing an *o*-OMe group also coupled with moderate efficiency (**3ma**), indicating tolerance of steric hindrance. While the electron-drawing group at the meta position of the oxime esters

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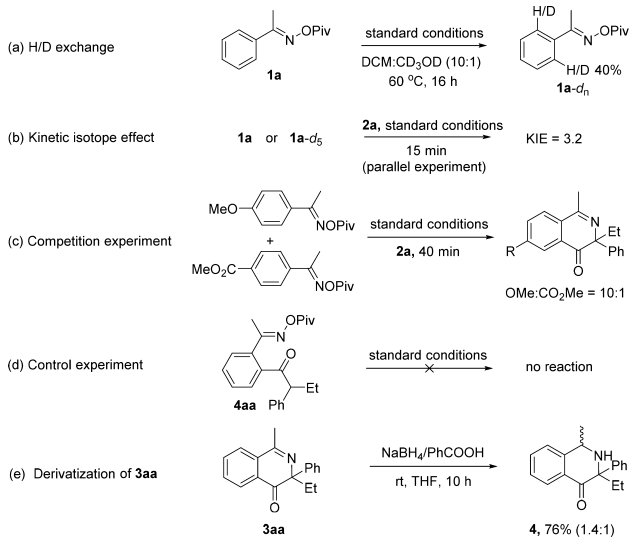
almost lead to no activity (**3na**), *meta* electron-donating group substituted arenes exhibited high reactivity and regioselectivity at the less hindered site (**3oa**−**3qa**, **3sa**), except for isomeric products **3ra** and **3ra′**, likely due to the secondary coordination effect of the oxygen group. Of note, *O*-pivaloyl oximes in cyclic frameworks were also applicable, and the desired products **3ta** and **3ua** were isolated both in 78% yields. The C−H activation was not limited to a benzene ring (**3va**, **3wa**); both *ortho* positions of a thiophene ring underwent C−H activation with moderate regioselectivity (**3wa** and **3wa′**). We next evaluated the generality of ketenes in the coupling with several *O*-pivaloyl oximes. Introductions of a chloro, fluoro, or methyl group into the benzene ring of ethylaryketenes were well tolerated (**3cb**−**3ce**, **3ad**), and the reaction efficiency was only slightly affected by steric hindrance of the ketene. In addition to using an ethyl substituent, arylketenes substituted by methyl and *n*-propyl also coupled with comparably high efficiency (**3af**, **3cg**).

To gain insight into the mechanism of the coupling reaction, experimental studies have been performed. H/D exchange between oxime ester **1a** and CD_3OD under the standard conditions (Scheme 3a) revealed significant H/D exchange at

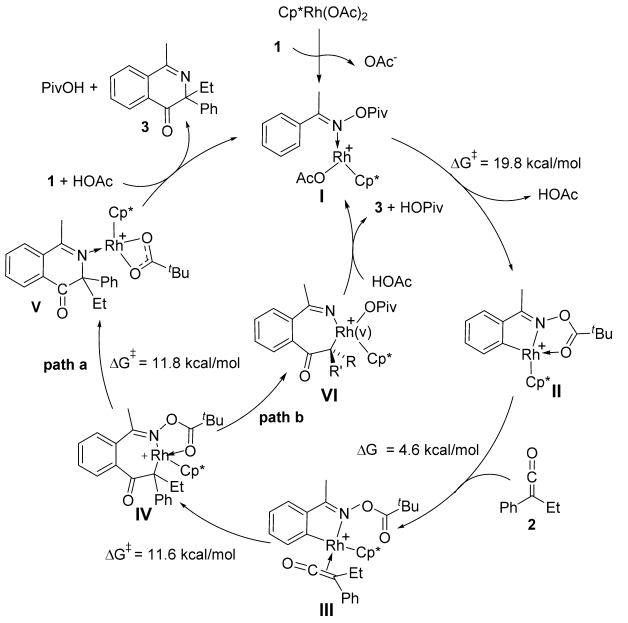
Scheme 3. Mechanistic Studies and a Derivatization Reaction



the *ortho* positions, indicating reversibility of the C−H cleavage. The kinetic isotopic effect was then measured in two side-by-side reactions using **1a** and **1a-d_5** at a low conversion under the standard conditions, from which k_H/k_D = 3.2 was obtained by ^{1}H NMR analysis (Scheme 3b). This result indicates that a C−H activation process is probably involved in the turnover-limiting step. In addition, intermolecular competition has been performed for two arenes that differ in electronic effect, and the electron-rich arene exhibited significantly higher reactivity (Scheme 3c), which is consistent with higher nucleophilicity of the Rh−C bond. Then, we performed a control experiment by subjecting the simple acylation product **4aa** to the standard conditions (Scheme 3d), and no reaction was detected, suggesting that full C−H acylation could not lead to the final annulation. Encouraged by the wide abundance of piperidone in biologically active compounds, we turned our attention to the reduction of the C═N bond. Treatment of **3aa** with $NaBH_4$/PhCOOH led to chemoselctive reduction of the imine, affording piperidone **4** in 76% yield and in a 1.4:1 diastereomeric ratio (Scheme 3e).[14]

On the basis of these observations, previous reports,[7,13,15] and our DFT studies (see the Supporting Information for details), a plausible mechanism of the coupling of oxime ester **1a** and **2a** is proposed in Scheme 4. The catalytic cycle starts

Scheme 4. Proposed Pathways of Coupling of Oxime Esters with Ketenes (See the Supporting Information for Detailed DFT Studies)



from a cationic rhodium complex **I**, which could be generated by the ligand exchange with oxime ester **1**. With assistance of coordination of the oxime moiety, *ortho* C−H cleavage takes place via concerted metalation−deprotonation (CMD) to give a rhodacyclic intermediate **II** with an activation free energy of 19.8 kcal/mol. Coordination of a ketene is followed by migratory insertion of the Rh−C(alkyl) bond into the C═C bond of ketene, affording a fused rhodacycle **IV** with an activation free energy of 11.6 kcal/mol. The coordination saturation in rhodacycle **IV** serves to inhibit protonolysis of the Rh−C(alkyl) bond. Meanwhile, N−O bond cleavage and corresponding N−C bond formation might occur in either a concerted (pathway a) or stepwise (pathway b) fashion to furnish the coupled product **3**, together with regeneration of the active Rh(III) intermediate **I** by the ligand exchange of oxime ester **1a**. In the stepwise pathway b, a high oxidation state Rh(V) intermediate is generated prior to reductive elimination. Our DFT studies by an intrinsic reaction coordinate (IRC) indicated that the concerted pathway can readily occur, and is consequently a favored pathway, in which the reaction proceeds via all-Rh(III) intermediates. In particular, the concerted C−N formation/N−O cleavage takes place with an activation barrier of only 11.8 kcal/mol.

In summary, we have realized Rh(III)-catalyzed annulative couplings between arenes and ketenes. The coupling of *O*-pivaloyl oxime occurred under redox-neutral conditions to give isoquinolones, and the OPiv group played a key role in suppressing protonolysis of the Rh−C(alkyl) bond. DFT studies suggest that the C−N coupling/N−O cleavage occurs via a concerted all-Rh(III) process. This concise protocol to

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access QCSC scaffolds may find applications in the synthesis of complex biologically active products.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b00906.

Detailed experimental procedures, characterization of new compounds, DFT calculation data, and copies of NMR spectra (PDF)

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Notes

The authors declare no competing financial interest.

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