C–H Activation

DOI: 10.1002/ange.201207204

Rhodium(III)-Catalyzed Oxidative C–H Functionalization of Azomethine Ylides**

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Metal-catalyzed oxidative C–H olefination of arenes has emerged as a powerful alternative to the traditional Heck coupling for new C–C bond formation.[1] The significance of this reaction has been elegantly demonstrated in many new synthetic methods as well as in the total synthesis of natural products.[2] Whereas oxidative olefination is predominantly catalyzed by palladium complexes, rhodium(III)-catalyzed olefination of C–H bonds has increasingly received attention owing to high efficiency, broad scope, and good functional group tolerance.[3] Rh^{III}-catalyzed olefination of arenes bearing protic directing groups has allowed the synthesis of lactams[4] and oxygen-containing heterocycles.[5] Moreover, oxidative coupling of arenes with alkynes can lead to the construction of a broad spectrum of heterocycles such as isocoumarins,[5] indoles or pyrroles,[6] pyridines,[7] isoquinolines,[8] isoquinolones,[9] quinolones,[10] and pyridones.[11] Some of these strategies have been successfully applied to the synthesis of natural products.[12] Despite these successes, it is necessary to explore the C–H activation of arenes facilitated by directing groups that not only coordinate to metals, but also participate in subsequent reactions to provide novel tandem transformations. This serves to both broaden the scope of rhodium-catalyzed C–H functionalization reactions and to achieve molecular complexity.

We reasoned that azomethine ylides of (hetero)arylaldehydes such as **1a** are suitable substrates for C–H activation under chelation assistance although C–H activation of these substrates has not been reported. Furthermore, the possible cleavage of C=N, C–N, and C–C bonds in the pyrazolidinone ring may be coupled with C–H activation, leading to versatile and unique products (Figure 1). We now report the rich chemistry of Rh^{III}-catalyzed oxidative C–H olefination of azomethine imines, where cleavage of all of these bonds are involved under different conditions.

We initiated our studies with a screen of the conditions for the coupling of **1a** with benzyl acrylate (**2a**) using $[RhCp^*(MeCN)_3](SbF_6)_2$ as a catalyst (Cp^* = pentamethylcyclopentadienyl; 4 mol %). $Cu(OAc)_2$ proved to be a less effective oxidant, and two functionalized 1,2-dihydrophthalazines, **3aa** and **4aa**, were isolated in low yields. In contrast, AgOAc proved to be effective as an oxidant and monoolefination/cyclization product **3aa** was isolated in 88% yield when the reaction was optimized by using a slight excess of the olefin and the oxidant [conditions A, Eq. (1); Bn = benzyl]. Furthermore, the yield of isolated **4aa** was optimized to 65% by using the olefin and AgOAc in greater excess (conditions B). In contrast, only [3+2] dipolar addition occurred when the rhodium catalyst was omitted (see the Supporting Information).

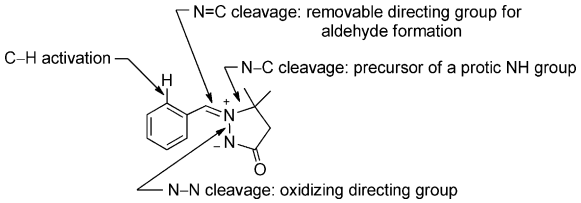
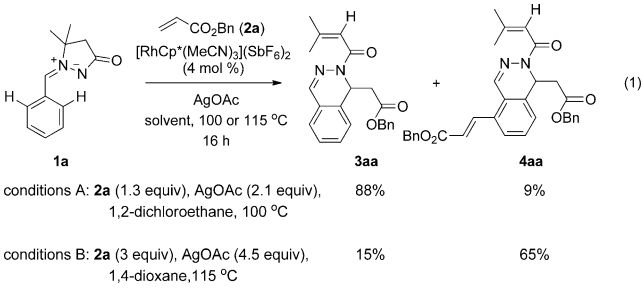


Figure 1. Versatile reactivity of azomethine imines under C–H activation conditions.



(1)

With the optimized conditions in hand, we next explored the scope of this reaction. Acrylates smoothly coupled with azomethine **1a** under conditions A (Scheme 1), and products **3aa**–**3ae** were isolated as the major products in 68-88% yield, whereas the the corresponding diolefination product were isolated in yields of 56–79% under conditions B. Acrylonitrile is also a viable coupling partner, but only for monoolefination/cyclization (**3af**). The scope of the azomethine substrate was next explored, using *n*-butyl or benzyl acrylate. Electron-donating, electron-withdrawing, and halogen (**3ha** and **3ia**) groups at the *para* position of the benzene ring are all well tolerated under both sets of conditions. When the ortho position was blocked by a hologen group or fused with an extra ring, selective monoolefination/cyclization was obtained. Azomethines with substituents in the *meta* position

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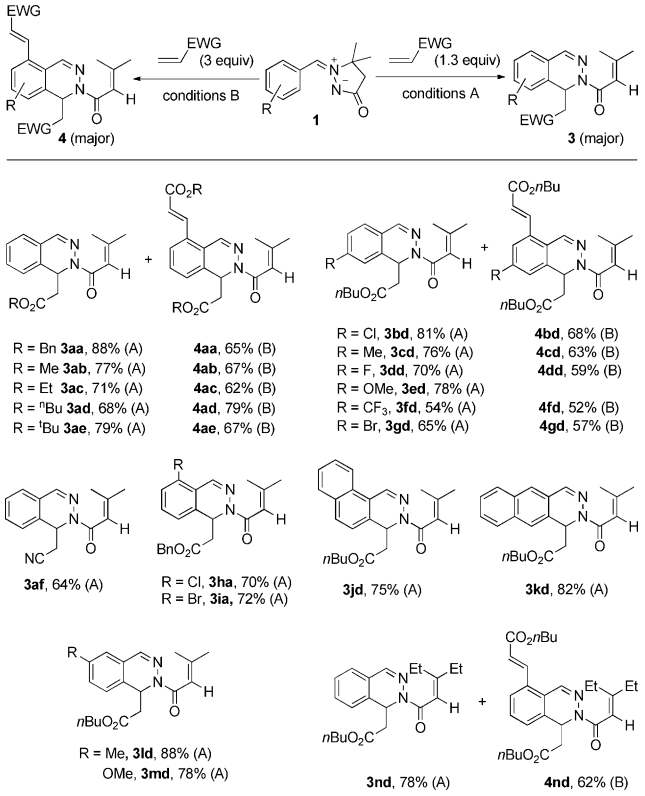
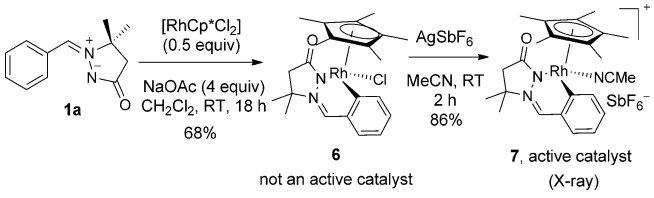
[**] This work was supported by the Dalian Institute of Chemical Physics and the Chinese Academy of Sciences.
Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/anie.201207204.



Scheme 1. Coupling of azomethines with olefins. Conditions A: azomethine (0.50 mmol), olefin (0.65 mmol), $[RhCp^*(MeCN)_3](SbF_6)_2$ (4 mol%), AgOAc (1.05 mmol), DCE (4 mL), 100°C, 16 h. Conditions B: azomethine (0.5 mmol), olefin (1.5 mmol), $[RhCp^*(MeCN)_3](SbF_6)_2$ (4 mol%), AgOAc (2.25 mmol), 1,4-dioxane (4 mL), 115°C, 16 h. Yields shown are of isolated products. DCE = 1,2-dichloroethane, EWG = electron withdrawing group.

are well-tolerated (**3ld** and **3md**), and the C–H functionalization preferentially occurred at the less hindered site. Moreover, a gem-diethyl analogue reacted smoothly with comparable reactivity and selectivity (**3nd** and **4nd**). In contrast to the smooth reactions of these activated olefins, *N*,*N*-dimethylacrylamide and styrene were less effective and reacted with different selectivity (see the Supporting Information). Phthalazines and dihydrophthalazines are known to be potentially bioactive.[13] Dihydrophthalazines are less accessible, and they are typically synthesized by nucleophilic addition to phthalazines.[14]

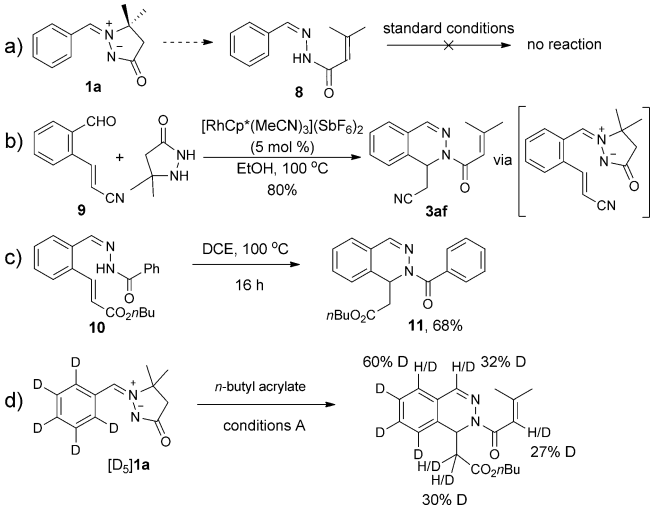
We next focused on the mechanistic aspects of this chemistry. Azomethine **1a**, $[RhCp^*Cl_2]_2$, and NaOAc reacted smoothly to give rhodacycle **6** in 68% yield (Scheme 2). Both ^1H and ^{13}C NMR analysis of **6** pointed to cyclometalation. Chloride abstraction (by $AgSbF_6$) from complex **6** afforded



Scheme 2. Rhodium(III) intermediates in catalysis.

complex **7**, which was further characterized by X-ray crystallography.[15] Only complex **7** successfully catalyzed the coupling of **1a** with ethyl acrylate (**2c**) under monoolefination/cyclization conditions, and **3ac** (68%) and **4ac** (12%) were isolated in essentially the same yields as those obtained using $[RhCp^*(MeCN)_3](SbF_6)_2$, which suggests that a vacant coordination site is necessary. Although Rh^{III} catalysts have been widely applied in C–H activation, reports on isolated stable organorhodium(III) intermediates are still limited.[16]

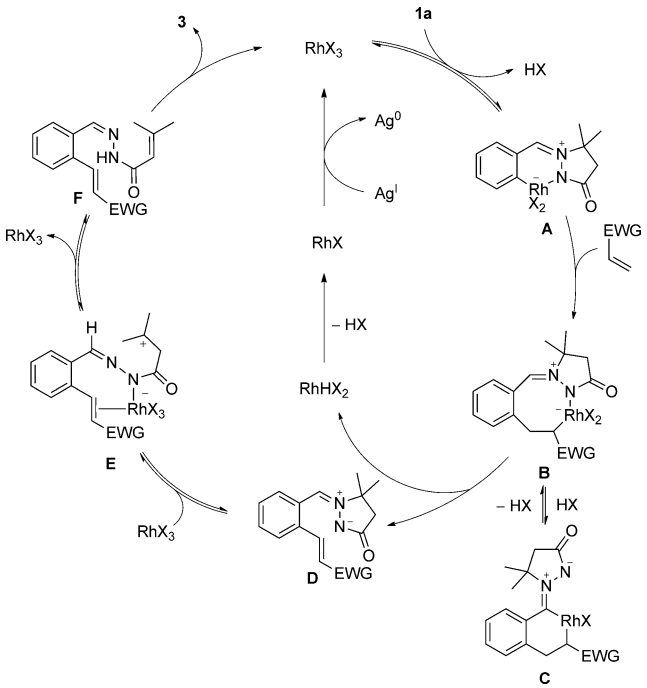
Secondly, the sequencing of the ortho C–H olefination and scission of the pyrazolidinone ring was probed. Subjecting hydrazone[17] **8** to conditions A with ethyl acrylate only afforded slight decomposition, which indicates that the C–H olefination should occur prior to any opening of the pyrazolidinone ring (Scheme 3a). To study the sequencing



Scheme 3. Mechanistic studies. Conditions A: azomethine (0.50 mmol), olefin (0.65 mmol), $[RhCp^*(MeCN)_3](SbF_6)_2$ (4 mol%), AgOAc (1.05 mmol), DCE (4 mL), 100°C, 16 h.

of the 2nd olefination and the scission of the pyrazolidinone ring, an isolated sample of **3aa** was subjected to conditions B (benzyl acrylate), but no reaction occurred. This suggests that the 2nd olefination should occur prior to cyclization, and that it is also chelation assisted. Based on these results, a sequence of 1) mono- or diolefination of azomethine, 2) opening of pyrazolidinone ring, and 3) *6-exo-trig* cyclization was proposed (Scheme 4).

The proposed *ortho*-olefinated azomethine intermediate was next probed. Although we failed to prepare an azomethine bearing an *ortho* (*E*)-CH=CHCN group as a starting material, the rhodium-catalyzed one-pot reaction of aldehyde **9** with 5,5-dimethylpyrazolidinone afforded 1,2-dihydrophthalazine **3af** in high yield (Scheme 3b), which indicates the intermediacy of an *ortho* olefinated azomethine. The rhodium catalyst is necessary in this reaction, as omission of the catalyst afforded a mixture of unidentifiable products, which also suggests that the rhodium catalyst promotes the scission of the pyrazolidinone ring and/or the subsequent Michael cyclization. The intermediacy of an olefinated hydrazone was next explored. Hydrazone **10** cyclized smoothly to give 1,2-



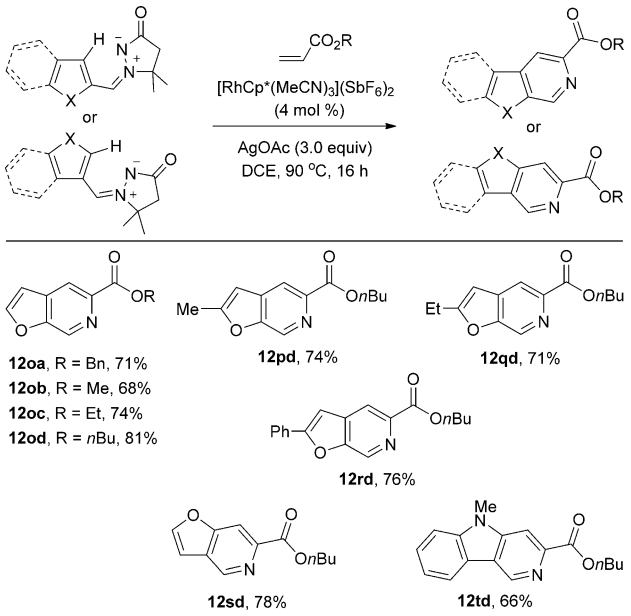
Scheme 4. Proposed catalytic cycle.

dihydrophthalazine **11** (68% yield) even under uncatalyzed conditions (Scheme 3c). As well as supporting the intermediacy of **10**, this result also indicates that the scission of the pyrazolidinone ring is rhodium-mediated.

Additional information on the catalytic cycle was obtained from deuterium-labeling studies using [D_5]**1a** and *n*-butyl acrylate (Scheme 3d). ^{1}H NMR analysis of the isolated product revealed decreased deuteration at the position *ortho* to the imine group, and deuterium scrambled to the imine C–H (28%), the olefinic C–H (26%), and the α methylene positions (30%).[18] The observed loss of deuterium in the *ortho* position of the product agrees with reversible cyclometalation, and this metalation/proto-(deutero)demetalation should be faster than any subsequent reactions.[6a] Therefore, C–H cleavage cannot be turnover-limiting.[19]

A proposed mechanism for the formation of product **3** is given in Scheme 4. Reversible cyclometalation affords a rhodium(III) intermediate **A** and an acid (HX, or DX for [D_5]**1a**). Insertion of an olefin gives metallacycle **B**, which may undergo off-loop nitrogen dissociation followed by reversible cyclometalation to give **C**, which can account for the H/D exchange on the imine, because both HX and DX can cleave the Rh-C_{imine} bond in intermediate **C** when [D_5]**1a** is used. Subsequent β-hydrogen elimination from **B** generates *ortho*-olefinated azomethine **D** and a rhodium(III) hydride, which reductively eliminates an HX followed by Ag^I oxidation to regenerate the active Rh^{III} catalyst. Intermediate **D** is proposed to undergo Rh^{III}-catalyzed reversible scission of the pyrazolidinone ring to afford protic hydrazone **F**. With the reversible C–N scission, deuterium is incorporated into the pyrazolidinone ring and eventually into the olefinic position of the product when the *N*-deuterated hydrazone cyclizes back to a pyrazolidinone. Uncatalyzed cyclization of protic hydrazone **F** yields the final product, and deuterium incorporation into the α-methylene group is expected when *N*-deuterated **F** cyclizes.

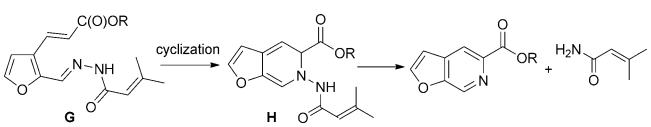
To better define the scope of the azomethine substrate, an azomethine functionalized with a 2-furanyl group was used (Scheme 5). Significant substrate effects were observed and



Scheme 5. Formation of pyridine-fused heterocycles. Conditions: azomethine (0.50 mmol), olefin (0.65 mmol), [RhCp*$(MeCN)_3$]$(SbF_6)_2$ (4 mol%), AgOAc (1.50 mmol), DCE (4 mL), 90°C, 16 h. Yields shown are of isolated products.

a furo[2,3-c]pyridine was isolated and identified as a result of C–H olefination and N–N cleavage. Azomethines with alkyl- and aryl-substituted furans coupled smoothly with acrylates (68–81%). The azomethine is not limited to C2-substitution in the furan ring. Thus, under the same conditions a C3-substituted substrate undergoes C2–H bond cleavage with high selectivity and only a single isomeric product (**12sd**) was observed. Furthermore, indole-functionalized azomethine smoothly coupled with *n*-butyl acrylate to afford an indole-fused pyridine (**12td**) in good yield. These fused heterocycles are widely present as the cores of natural products.[20] The synthesis of these less accessible heterocycles through C–H activation is unprecedented.

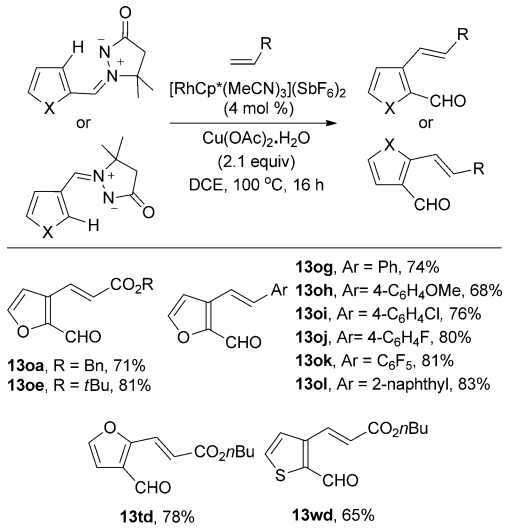
A simplified pathway for this transformation, starting from putative heteroaryl hydrazone intermediate **G** is proposed in Scheme 6. Instead of undergoing any intramolecular Michael reaction, **G** undergoes 6-e cyclization[21] to give **H** as a result of stereoelectronic effects of the heterocyclic scaffold. The final product was produced upon the elimination of a



Scheme 6. Proposed formation of pyridine-fused furan.

3-methylbutenamide. In this system, the azomethine functions as a oxidizing directing group, and this system is an oxidative olefination process that uses an unprecedented combination of external and internal oxidants. Although there are reports of Rh^{III}- and Ru^{II}-catalyzed couplings of arene C–H bonds with alkenes and alkynes using N–OR oxidizing directing groups,[22] the strategy of using oxidizing N–N bonds, has not been reported for C–H activation, likely owing to limited bond polarization.[22b]

The azomethine group may also act as a potential removable directing group[23] to produce a sequence of C–H olefination and subsequent C=N bond hydrolysis. Indeed, when the oxidant is simply switched to $Cu(OAc)_2{\cdot}H_2O$, the coupling of 2-furanyl functionalized azomethine with *tert*-butyl acrylates proceeded with complimentary selectivity and olefinated aldehyde **13oe** was isolated in 81% yield (Scheme 7). Moreover, the olefin substrate can be extended to various styrenes (**13og–13ol**). The heterocycle is not



Scheme 7. Formation of *ortho*-olefinated heteroaryladehydes. Conditions: azomethine (0.50 mmol), olefin (0.7 mmol), $[RhCp^*(MeCN)_3](SbF_6)_2$ (4 mol%), $Cu(OAc)_2{\cdot}H_2O$ (1.05 mmol), DCE (4 mL), 100°C, 16 h. Yields shown are of isolated products.

limited to the furan ring (**13wd**), nor is the substitution of the azomethine limited to the C2 position (**13td**). In most cases, the *ortho*-olefinated aldehyde was isolated in high yield (65–83%). Here the azomethine acts as a removable directing group to give a different selectivity under oxidant control. Aldehyde is often a poor directing group for oxidative C–H olefination and high catalyst loading is necessary to produce even a low yield of product.[24] Indeed, essentially no desired product was detected when 2-furaldehyde was subjected to the same conditions.

In summary, we have achieved the Rh^{III}-catalyzed oxidative coupling of azomethine ylides with olefins. With AgOAc as an oxidant, azomethines of benzaldehydes undergo selective
C–H and C–N cleavage to produce 1,2-dihydrophthalazines; several intermediates in this reaction were studied to better understand the mechanism. In sharp contrast, the reaction of azomethines of heteroaldehydes (such as furaldehydes) afforded heteroarene-fused pyridines as a result of C–H and N–N cleavage. In addition, using $Cu(OAc)_2$ as the oxidant, the olefination of azomethines of these heteroarldehydes gave *ortho*-olefinated aldehydes. Given the versatility of this reaction system and the diversity of the products, this method is likely to find applications in the synthesis of complex structures.

Received: September 5, 2012
Published online: October 19, 2012

Keywords: azomethines · C-H activation · olefination · oxidative coupling · rhodium

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