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Rh(III)-Catalyzed Diastereoselective Annulation of Amides with Quinone Monoacetals: Access to Bridged Nine-Membered Heterocycles via C−H Activation

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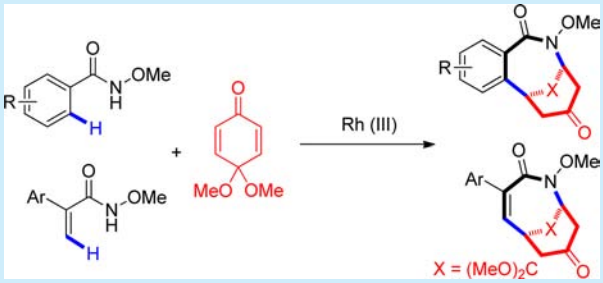
Supporting Information

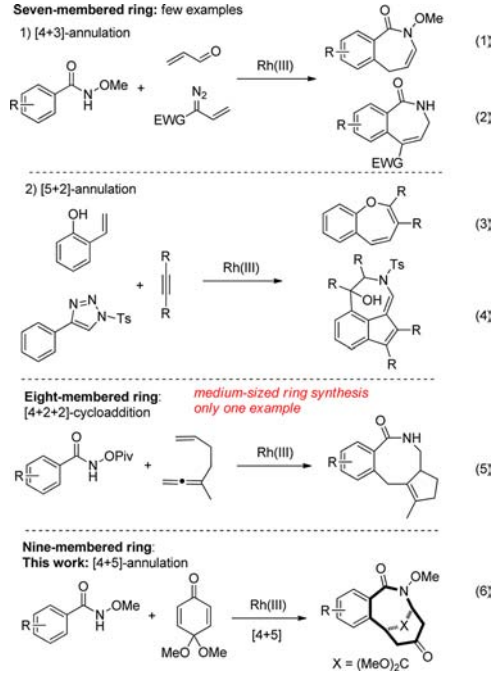
ABSTRACT: An unprecedented Rh(III)-catalyzed annulation of various benzamides and acrylamides with quinone monoacetals was developed for the facile and efficient one-pot synthesis of bridged nine-membered benzo[*c*]azonine-1,5(2*H*)-diones and 2-azabicyclo[4.3.1]dec-4-ene-3,8-diones. It is the first example of synthesis of nine-membered heterocycles through Rh(III)-catalyzed C−H bond functionalization, and both aryl and vinyl C−H bonds are tolerant in this reaction. A plausible mechanism is proposed on the basis of control experiments.



Recently, Rh(III)-catalyzed C−H activation has proven to be a powerful and environmentally friendly tool for the construction of nitrogen-containing heterocycles because it obviates the need for prefunctionalization of the starting materials and possesses high functional-group tolerance.[1] In this context, [3 + 2]-,[1,2] [4 + 1]-,[1,3] [4 + 2]-,[1,4] [5 + 1]-,[5] and [3 + 3]-annulations[6] have been well developed for the synthesis of five- and six-membered heterocycles. In contrast, only a few examples on the synthesis of seven-membered and medium-sized rings have been reported.[1,7−10] In 2013, the research groups of Glorius and Cui independently disclosed elegant rhodium-catalyzed [4 + 3]-annulations of *N*-methoxybenzamides with *α,β*-unsaturated aldehydes[7] or vinylcarbenoids[8] to construct azepinones, respectively (Scheme 1, eqs 1 and 2). In 2014, Mascareñas and Gulías documented a formal [5 + 2]-annulation between *o*-vinylphenols and alkynes for the practical and versatile synthesis of benzoxepines (Scheme 1, eq 3).[9] One year later, a rhodium(III)-catalyzed domino [3 + 2]/[5 + 2]-annulation of 4-aryl-1-tosyl-1,2,3-triazoles with internal alkynes was reported for the construction of indeno[1,7-*cd*]azepines by Li and co-workers (Scheme 1, eq 4).[10] For the preparation of medium-sized rings via Rh(III)-catalyzed C−H annulation reactions, there is only one example so far. Last year, a formal [4 + 2 + 2]-cycloaddition of *N*-(pivaloyloxy)benzamides with 1,6-allene−enes for the preparation of eight-membered lactams was successfully developed by Ma and co-workers (Scheme 1, eq 5).[11] To date, the assembly of nine-membered rings by means of related C−H bond functionalization remains unexplored.

Because of entropic and enthalpic reasons, the synthesis of nine-membered rings from acyclic precursors is quite challenging.[12] While some synthetic methods for the construction of

Scheme 1. Rh(III)-Catalyzed Annulation of C−H Bonds for Medium-Sized Ring Synthesis



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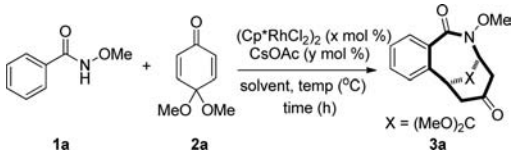
these ring systems through transition-metal-catalyzed cyclization reactions have been reported,[12,13] the development of conceptually new synthetic approaches, especially those involving inert C−H bond activation, is still of great interest. Quinone derivatives are versatile coupling partners in the Rh(III)-catalyzed C−H functionalization reactions.[14−17] In 2014, Lin and co-workers reported elegant annulations between alkyne-tethered cyclohexadienone and *N*-methoxybenzamides or *N*-(pivaloyloxy)benzamides to afford hydrobenzofurans and isoquinolones, respectively.[14] In the same year, Li and co-workers developed a Rh(III)-catalyzed redox-economic arylation of arenes using 4-hydroxycyclohexa-2,5-dienones as the arylating reagents.[15] During our investigation of Rh(III)-catalyzed C−H functionalization,[18] we observed that quinones were versatile coupling partners in the chemoselective [4 + 2][16] and [3 + 3][17] annulation. The aromatization potential of the quinone derivatives plays a vital role in these selective transformations. Based on this observation and combined with our continuous interest in the synthesis of medium-sized rings,[19] we became interested in the annulation between the nonaromatizable quinone monoacetals and *N*-methoxybenzamides. Herein, we report an unprecedented Rh(III)-catalyzed diastereoselective annulation of amides with quinone monoacetals for the construction of bridged nine-membered heterocycles through C−H activation from simple precursors in a single operation (Scheme 1, eq 6).

Initially, the reaction of *N*-methoxybenzamide **1a** with quinonemonoacetal **2a** was employed to screen the reaction conditions for the annulation (Table 1). When the reaction was

Table 1. Optimization of the Reaction Conditions[a]



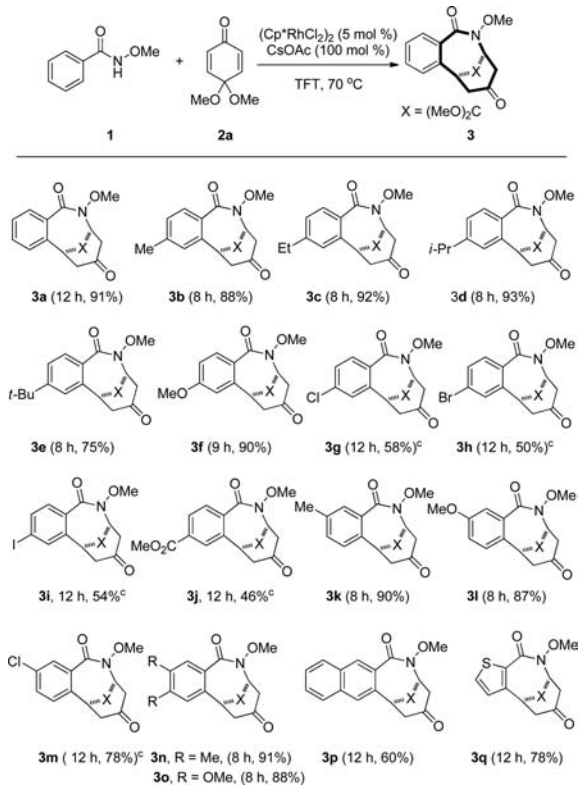
entry	*x*	*y*	solvent	temp (°C)	time (h)	yield[b] (%)
1	2.5	30	CH_3OH	70	12	29
2	2.5	30	CH_3OH	90	12	16
3	2.5	30	CH_3OH	50	12	trace
4	2.5	30	C_2H_5OH	70	12	8
5	2.5	30	DCE	70	12	trace
6	2.5	30	acetone	70	12	10
7	2.5	30	toluene	70	12	21
8	2.5	30	TFT[c]	70	12	59
9	5	30	TFT[c]	70	12	70
10	5	100	TFT[c]	70	12	91
11	2.5	100	TFT[c]	70	24	60

[a]Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), $(Cp^*RhCl_2)_2$, and CsOAc in solvent (2.0 mL). [b]Isolated yields. [c]TFT = trifluorotoluene.

conducted in methanol in the presence of $[Cp^*RhCl_2]_2$ (2.5 mol %) and CsOAc (30 mol %) at 70 °C for 12 h, tetrahydro-1*H*-3,7-methanobenzo[*c*]azonine-1,5(2*H*)-dione (**3a**) was obtained in 29% yield (Table 1, entry 1). Elevating the reaction temperature to 90 °C led to a lower yield of **3a** (Table 1, entry 2), whereas decreasing the temperature to 50 °C produced trace amount of **3a** (Table 1, entry 3). Solvent screening revealed that the reaction in trifluorotoluene (TFT) gave a higher yield of **3a** (Table 1, entries 1, 4−8). To our delight, when $[Cp^*RhCl_2]_2$ (5 mol %) and CsOAc (100 mol %) were used, the yield of **3a** was improved to 91% (Table 1, entry 10 versus entries 9 and 11). The structure of **3a** was unambiguously confirmed by single-crystal X-ray analysis.[20]

With the optimal conditions in hand (Table 1, entry 10), we surveyed various substrates to determine the scope of the reaction. The annulation reactions proceeded smoothly to afford various bridged nine-membered benzo[*c*]azonine-1,5(2*H*)-diones **3** in good to excellent yields (Scheme 2). A wide range of

Scheme 2. Synthesis of Benzo[*c*]azonine-1,5(2*H*)-diones[a,b]



[a]Reaction conditions: **1** (0.2 mmol), **2a** (0.3 mmol), $(Cp^*RhCl_2)_2$ (5 mol %) and CsOAc (100 mol %) in TFT (2.0 mL) at 70 °C. [b]Isolated yields. [c]$(Cp^*RhCl_2)_2$ (10 mol %) was used.

important functional groups on the aryl moieties of benzamides **1**, such as chloro, bromo, iodo, and ester groups, were well tolerated under the reaction conditions, and these groups can be used as handles for further diversification. Substrates **1** with both electron-donating (**1a−f**) and electron-withdrawing groups (**1g−j**) at the *para* position of the aryl groups participated in this reaction; electron-rich substrates generally reacted faster and gave higher yields of products **3** than electron-deficient ones (**3a−f** versus **3g−j**). Reactions of benzamides with both electron-donating and -withdrawing *meta*-substituents smoothly proceeded to give the corresponding products **3k−m** in good yields in a regiospecific manner. 3,4-Disubstituted benzamides smoothly reacted to provide the corresponding nine-membered heterocycles **3n** and **3o** in high yields. In the case of *β*-naphthamide **1p**, naphtho[2,3-*c*]azonine-1,5(2*H*)-dione **3p** was obtained in good yield. Furthermore, the reaction was also tolerated with heteroaryl carboxamides, and when thiophene-2-carboxamide **1q** was treated with **2a** under the optimal conditions, thieno[2,3-*c*]azonine-6,10(5*H*,7*H*)-dione **3q** was obtained in good yields. When substituted quinone monoacetals, such as 4,4-dimethoxy-2-methylcyclohexa-2,5- dienone (**2b**) and

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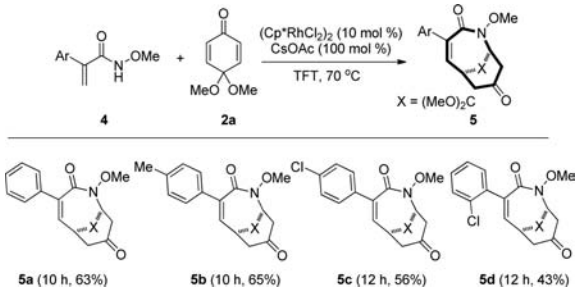
2-chloro-4,4-dimethoxycyclohexa-2,5-dienone (**2c**), were employed in this annulation under the standard conditions (Table 1, entry 10), only a trace of the corresponding bridged nine-membered benzo[*c*]azonine-1,5(2*H*)-diones could be detected. It is worth mentioning that the annulation proceeded diastereoselectively on the basis of ^{1}H and ^{13}C NMR spectroscopy data of products **3** (no diastereoisomers of **3a**−**q** were detected). The configurations of **3b**−**q** were assigned according to the X-ray diffraction analysis of **3a**.

Gratifyingly, the annulation was not restricted to aromatic amides. Olefinic carboxamides provided the expected bridged nine-membered 2-azabicyclo[4.3.1]dec-4-ene-3,8-diones under the same conditions while elevating the amount of catalyst to 10 mol %. Some selected examples are shown in Scheme 3. The

Scheme 3. Synthesis of 2-Azabicyclo[4.3.1]dec-4-ene-3,8-diones 5[a,b]



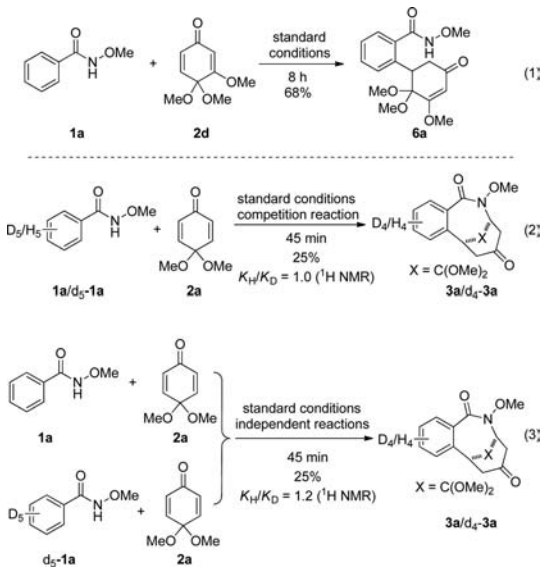
[a]Reaction conditions: **4** (0.2 mmol), **2a** (0.3 mmol), $(Cp^*RhCl_2)_2$ (10 mol %), and CsOAc (100 mol %) in TFT (2.0 mL) at 70 °C. [b]Isolated yields.

annulation of *N*-methoxy-2-arylacrylamides, bearing electron-nautral (**4a**), -rich (**4b**), and -deficient aryl (**4c** and **4d**) groups, with quinone monoacetal **2a** afforded the corresponding 2-azabicyclo[4.3.1]dec-4-ene-3,8-diones **5a**−**d** in good yields through the vinylic C−H activation.

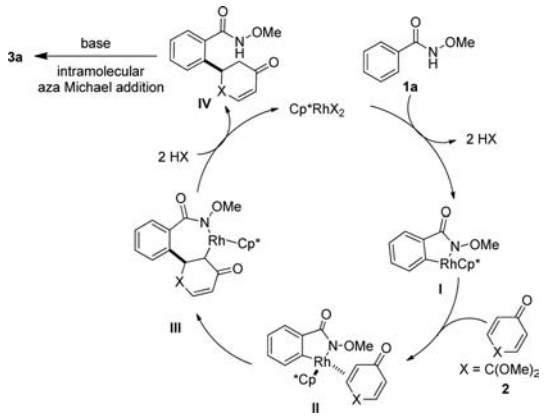
To shed light on the reaction mechanism of this annulation, control experiments were performed. When 3,4,4-trimethoxycyclohexa-2,5-dienone **2d** was used as the coupling partner, the adduct **6a** was obtained exclusively in 68% yield (Scheme 4, eq 1). This result indicates that the formation of the C−C bond is most likely earlier than the C−N bond in the annulation. Next, a deuterium-labeling experiment was carried out, which showed competition between protio and deutero *N*-methoxybenzamides **1a** with a 1:1 product ratio at early conversion (Scheme 4, eq 2). In addition, the kinetic isotope effect (KIE) was further measured from two side-by-side reactions using protio and deutero *N*-methoxybenzamides **1a** (Scheme 4, eq 3), and a KIE value of 1.2 was observed. These results demonstrate that the C−H bond cleavage process may not be involved in the rate-determining step of the annulation.

On the basis of our previous observations,[16,17] present results, and literature precedent,[15] a mechanistic pathway is proposed (Scheme 5, taking the reaction of amide **1a** with quinone monoacetal **2a** as an example). First, C−H bond cleavage of **1a** occurs to produce a five-membered rhodacycle intermediate **I**. Next, coordination of the quinone monoacetal **2a** affords intermediate **II**, which undergoes migratory insertion into the incipient Rh−C bond to form a seven-membered rhodacycle **III**.[15−17] Then, protonolysis delivers intermediate **IV** while concomitantly releasing the catalyst Cp^*RhX_2. Finally, intra-

Scheme 4. Control Experiments



Scheme 5. Proposed Mechanism



molecular aza-Michael addition of intermediate **IV** delivers the bridged nine-membered heterocycle **3a** under basic conditions.

In summary, an unprecedented Rh(III)-catalyzed annulation of various benzamides and acrylamides with quinone monoacetal was developed for the facile and efficient synthesis of bridged nine-membered benzo[*c*]azonine-1,5(2*H*)-diones and 2-azabicyclo[4.3.1]dec-4-ene-3,8-diones. This is the first example of synthesis of nine-membered heterocycles from Rh(III)-catalyzed C−H bond functionalization. The reaction features high efficiency, atom- and step-economy, broad substrate scope, and good functional group tolerance. Investigation of the application of this reaction is being carried out in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

Experimental procedures and characterization data for all compounds and X-ray data of **3a**. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b03777.

Experimental procedures and characterization data for all compounds and X-ray data for **3a** (PDF)

X-ray data for **3a** (CIF)

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Notes

The authors declare no competing financial interest.

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(20) CCDC 1517904 (**3a**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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