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# Dual Directing-Groups-Assisted Redox-Neutral Annulation and Ring Opening of *N*-Aryloxyacetamides with 1-Alkynylcyclobutanols via Rhodium(III)-Catalyzed C−H/C−C Activations

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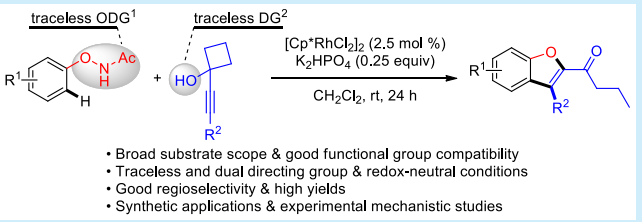
Supporting Information

**ABSTRACT:** A cascade [3 + 2] annulation and ring opening of *N*-aryloxyacetamides with 1-alkynylcyclobutanols via Rh(III)-catalyzed redox-neutral C−H/C−C activations using internal oxidative O−NHAc and −OH as the dual directing groups has been achieved. This reaction provided an efficient and regioselective approach to benzofuran derivatives with good functional group compatibility and high yields.



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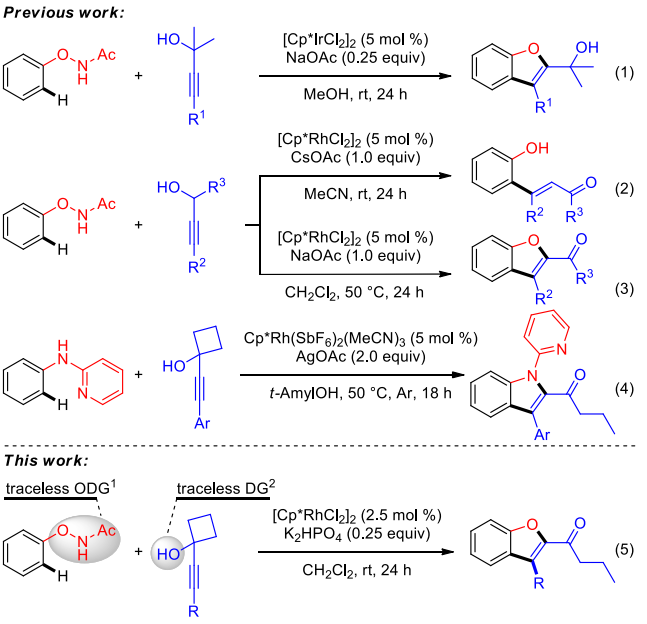
Transition-metal-catalyzed C−H bond activation and functionalization has been emerging as an area of great interest to chemists.[1] The past decade has witnessed the development of transition-metal-catalyzed C−H functionalization enabled by oxidizing directing groups (ODGs), which avoid the use of stoichiometric amounts of external oxidants.[2] In 2013, Liu and Lu pioneered an efficient synthesis of benzofuran and enamide derivatives via a Cp*Rh(III)-catalyzed coupling between *N*-aryloxyacetamides and alkynes with tunable selectivity under mild conditions.[3] Afterward, increasing attention has been devoted to transition-metal-catalyzed C−H functionalization using *N*-aryloxyacetamides as the privileged substrates for the redox-neutral coupling with different coupling partners, such as alkynes,[4] alkenes,[5] diazos,[6] and others.[7]

Among the different functionalized alkynes, alkynyl alcohols are widely used as important building blocks in synthetic chemistry.[4e−h,8] In 2018, Yi revealed an efficient and mild Ir(III)-catalyzed C−H annulation of *N*-aryloxyacetamides with tertiary propargyl alcohols to deliver benzofurans (Scheme 1, eq 1).[4f] In the same year, Yi developed the Rh(III)-catalyzed and solvent-controlled C−H functionalization of *N*-aryloxyacetamides with secondary or primary propargyl alcohols for the divergent synthesis of chalcones and benzofurans (Scheme 1, eqs 2 and 3).[4g] Almost simultaneously, Deng reported a similar Rh(III)-catalyzed coupling of *N*-aryloxyacetamides with secondary propargyl alcohols to construct chalcones under redox-neutral conditions.[4h] In 2017, Zeng described a Rh(III)-catalyzed C−H/N−H annulation of 1-alkynylcyclobutanols with arylamines to afford indole skeletons through a sequential C−H/C−C cleavage (Scheme 1, eq 4).[9]

**Scheme 1. Transition-Metal-Catalyzed C−H Functionalization with Alkynyl Alcohols**




Enlightened by the above elegant work and as part of our ongoing interest in transition-metal-catalyzed C−H functionalization,[4i,10] herein, we present our new development of Rh(III)-catalyzed cascade [3 + 2] annulation and ring opening

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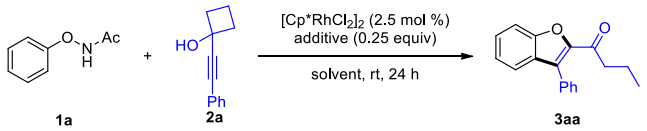
of *N*-aryloxyacetamides with 1-alkynylcyclobutanols. In this reaction, O−NHAc served as both the directing group and the internal oxidant. Moreover, O−NHAc and −OH, which avoided extra steps for removal of undesired directing groups, were traceless in the products. Notably, this methodology provided an efficient and regioselective approach to 1-(benzofuran-2-yl)butan-1-ones with good functional group tolerance and high yields under mild conditions (Scheme 1, eq 5).

*N*-Phenoxyacetamide **1a** and 1-(phenylethynyl)cyclobutanol **2a** were chosen as the model substrates for optimization of the reaction parameters, and the results were summarized in Table 1. Initially, the substrates was treated with $[Cp^*RhCl_2]_2$ (2.5 mol %) and CsOAc (0.25 equiv) in dichloromethane (0.1 M) at room temperature for 24 h, and the desired product **3aa** was obtained in 19% yield (entry 1). Intrigued by the isolation of **3aa**, we then attempted to investigate different additives, including basic acetates, bicarbonates, carbonates, phosphates, and hydrogen phosphates. The results suggested that $K_2HPO_4$ was more practical and provided a better yield. No target product was generated when $KH_2PO_4$ or no additive was used (entries 2−13). Next, a survey of solvents indicated that 1,2-dichloroethane gave an identical yield and that the reaction was sluggish when polar solvents were employed. Especially, no desired product was observed in MeOH (entries 14−17). To further improve the conversion of **2a**, the amount of **1a** was increased to 1.2 equiv and finally we were pleased to find that the desired product **3aa** could be isolated in 93% yield (entry 18). Notably, the reaction was not sensitive to the air atmosphere and moisture. Control experiments showed that Cp*Rh(III) was crucial toward the success of this reaction, as its omission led to no formation of **3aa**. Other representative Cp*M catalysts (M = Co, Ir, or Ru) did not show any catalytic activity. Under the optimal reaction conditions, *N*-phenoxypivalamide did not afford **3aa**, and neither 1-(phenylethynyl)-cyclopropanol nor 1-(phenylethynyl)cyclopentanol gave the corresponding products.

**Table 1. Optimization of the Reaction Conditions**[a,b]

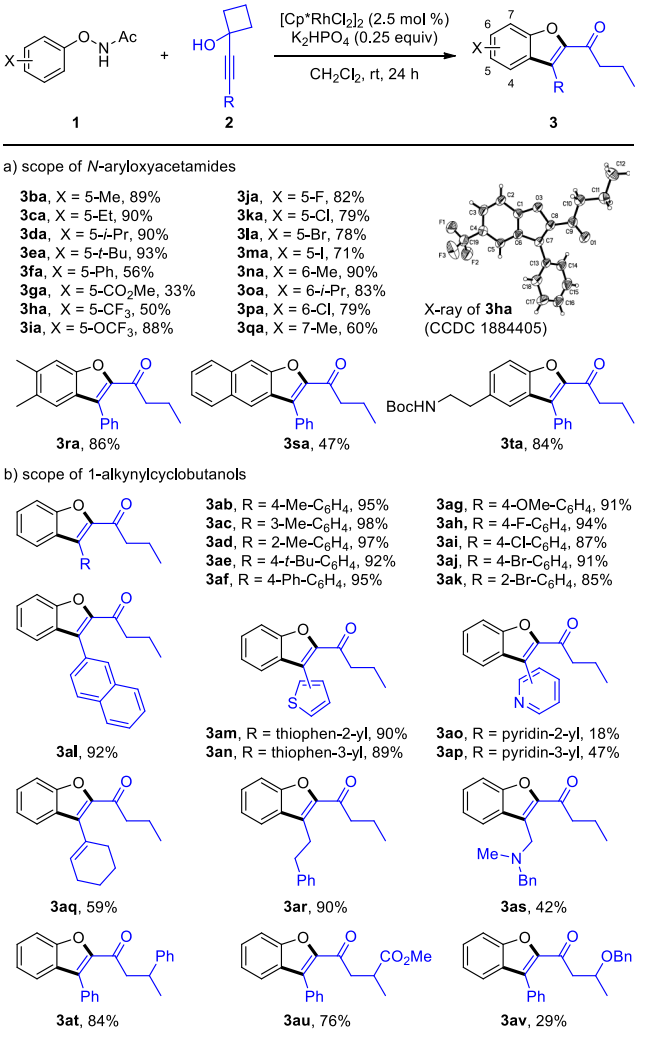



| entry | solvent | additive | yield (%) |
|---|---|---|---|
| 1 | $CH_2Cl_2$ | CsOAc | 19 |
| 2 | $CH_2Cl_2$ | KOAc | 25 |
| 3 | $CH_2Cl_2$ | NaOAc | 36 |
| 4 | $CH_2Cl_2$ | $NaHCO_3$ | 58 |
| 5 | $CH_2Cl_2$ | $KHCO_3$ | 83 |
| 6 | $CH_2Cl_2$ | $Na_2CO_3$ | 83 |
| 7 | $CH_2Cl_2$ | $K_2CO_3$ | 81 |
| 8 | $CH_2Cl_2$ | $Cs_2CO_3$ | 31 |
| 9 | $CH_2Cl_2$ | $Na_3PO_4$ | 79 |
| 10 | $CH_2Cl_2$ | $K_3PO_4$ | 81 |
| 11 | $CH_2Cl_2$ | $K_2HPO_4$ | 85 |
| 12 | $CH_2Cl_2$ | $KH_2PO_4$ | 0 |
| 13 | $CH_2Cl_2$ | – | 0 |
| 14 | $ClCH_2CH_2Cl$ | $K_2HPO_4$ | 85 |
| 15 | THF | $K_2HPO_4$ | 78 |
| 16 | MeCN | $K_2HPO_4$ | 39 |
| 17 | MeOH | $K_2HPO_4$ | 0 |
| **18**[c] | **$CH_2Cl_2$** | **$K_2HPO_4$** | **93** |

[a]Reaction conditions: **1a** (0.20 mmol), **2a** (0.20 mmol), $[Cp^*RhCl_2]_2$ (2.5 mol %), and additive (0.25 equiv) in solvent (2.0 mL) at rt for 24 h. [b]Isolated yield calculated based on **2a**. [c]1.2 equiv of **1a** was used.

With the optimized reaction conditions in hand, we first examined the scope and limitations of *N*-aryloxyacetamides in the present Cp*Rh(III)-catalyzed redox-neutral cascade [3 + 2] annulation and ring-opening reaction. As shown in Scheme 2, *para*-substituted *N*-aryloxyacetamides bearing alkyl, phenyl, trifluoromethoxy, and halogens led to the formation of the desired products in good yields (**3ba**−**3fa**, **3ia**−**3ma**). However, electron-deficient substrates were less reactive, affording the target products in 33% and 50% yield, respectively (**3ga** and **3ha**). For *meta*- substituted substrates, the reaction proceeded smoothly at the less sterically hindered position with excellent regioselectivity (**3na**−**3pa**). Substrate containing methyl at the *ortho*-position gave **3qa** in moderate yield. Additionally, 3,4-disubstituted and 2-naphthalenyl substrates were well-tolerated (**3ra** and **3sa**), and the reaction could be successfully applied to late-stage modification of a

**Scheme 2. Substrate Scope**[a,b]




[a]Reaction conditions: **1** (0.24 mmol), **2** (0.20 mmol), $[Cp^*RhCl_2]_2$ (2.5 mol %), and $K_2HPO_4$ (0.25 equiv) in $CH_2Cl_2$ (2.0 mL) at rt for 24 h. [b]Isolated yield calculated based on **2**.



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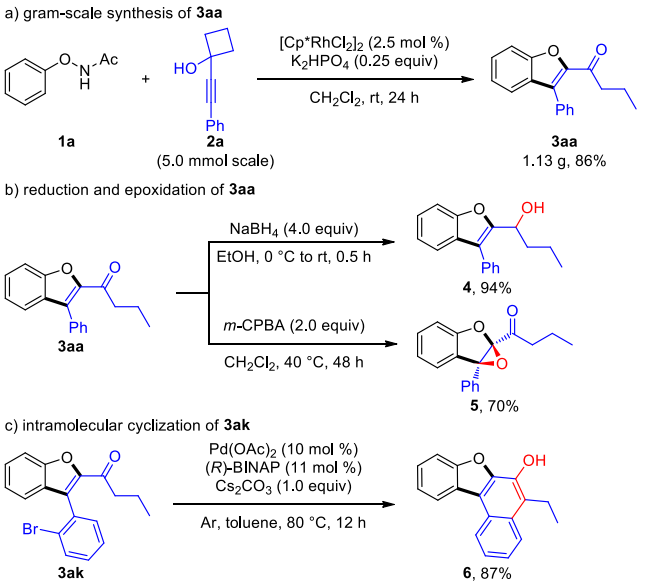
bioactive dopamine derivative, as exemplified by the construction of **3ta** with good yield and functional group compatibility. The structure of **3ha** was unambiguously confirmed by X-ray diffraction analysis.

Subsequently, we explored a wide range of 1-alkynylcyclobutanols under standard conditions. To our delight, various functionalities on the *para*-, *meta*-, or *ortho*-positions of the aromatic rings were compatible, giving the corresponding products in good to excellent yields (**3ab**−**3ak**). 2-Naphthalenyl, 2- or 3-thiophenyl substrates were well-tolerated, while 2- or 3- pyridinyl substrates gave unsatisfactory yields, probably due to the coordination of nitrogen with the catalyst (**3al**−**3ap**). Moreover, the reaction could be extended to alkenyl or alkanyl enthynylcyclobutanols (**3aq**−**3as**). Substrates bearing substituents, such as phenyl, methoxycarbonyl, and benzyloxy, at the 3-position of cyclobutanols showed different reaction activities, generating the corresponding products in yields ranging from 29% to 84% (**3at**−**3av**).

To demonstrate the practicality of this method in organic synthesis, a scaled-up reaction of **1a** (6 mmol) and **2a** (5 mmol) was performed under the optimal reaction conditions and the product **3aa** was isolated in 86% yield (Scheme 3a).

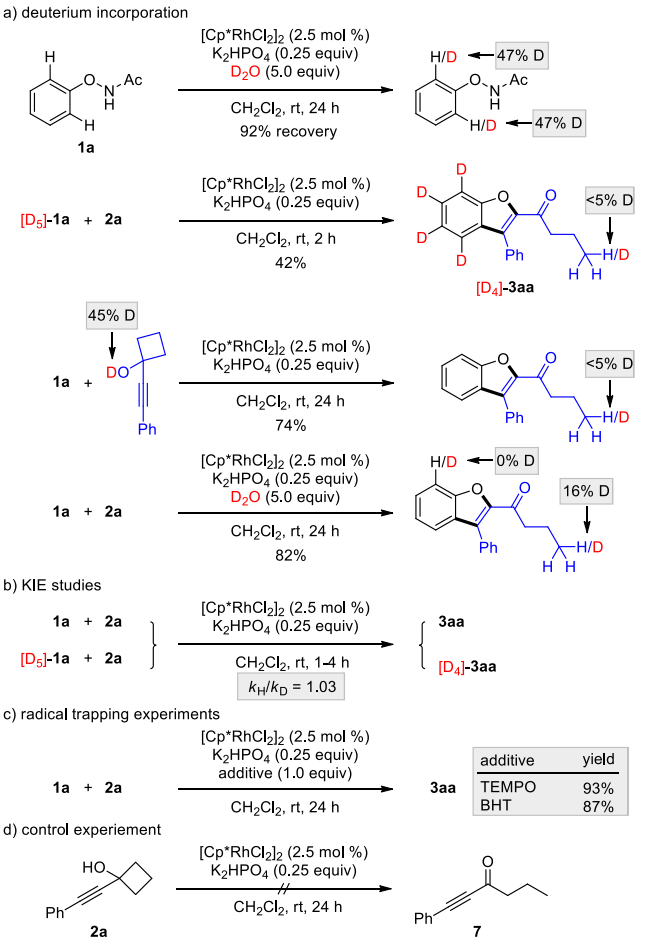
**Scheme 3. Synthetic Applications**




Further transformations of the products were subsequently conducted to prove their versatility. Reduction of **3aa** was readily available using $NaBH_4$ as a reductant, affording alcohol **4** in 94% yield. Epoxidation of **3aa** with *m*-CPBA smoothly gave **5** in 70% yield (Scheme 3b). Intramolecular cyclization of **3ak** was possible via a palladium-catalyzed cascade arylation−aromatization, delivering 5-*β*-naphthol derivative **6** in 87% yield (Scheme 3c).[11]

To get a better understanding of the reaction mechanism, a series of experiments were then carried out. First, **1a** was treated with $D_2O$ under the optimal reaction conditions, and 47% deuterium incorporation was observed at the *ortho* position of the directing group O−NHAc in the recycled substrate. This experiment demonstrated that C−H bond activation was largely reversible. Next, no evident deuterium incorporation was found in the terminal methyl group of **3aa** either in the reaction of [$D_5$]-**1a** and **2a** or in the reaction of **1a** and **2a** with a 45% deuterated hydroxyl group. In contrast, the reaction between **1a** and **2a** was performed smoothly in the presence of $D_2O$, and 16% deuterium was incorporated. These results indicated that one hydrogen in the terminal methyl group of **3aa** did not directly come from either aromatic ring of **1a** or the hydroxyl group of **2a**, and that metal protonation was likely to be involved in the reaction.[9] (Scheme 4a). The KIE

**Scheme 4. Experimental Mechanistic Investigations**




value were determined to be 1.03, which suggested that C−H bond cleavage might not be involved in the rate-determining step (Scheme 4b). In the presence of equivalent 2,2,6,6-tetramethylpiperidine-*N*-oxyl (TEMPO), or 2,6-di-*tert*-butyl-4-methylphenol (BHT) as a radical scavenger, **3aa** could still be isolated in 93% and 87% yields, respectively. These experiments implied that the reaction might not proceed in a radical pathway (Scheme 4c). A control experiment showed that **2a** could not converted to ketone **7** under the standard conditions, and substrate **2a** was recovered in 90% yield. This experiment illustrated that [3 + 2] annulation of **1a** and **7** was not involved in this methodology.

Based on the aforementioned experimental results, together with precedent literature,[4f,g,12] we have proposed a plausible mechanism for the Cp*Rh(III)-catalyzed cascade [3 + 2] annulation and ring opening, as depicted in Scheme 5. Initially, the reaction was triggered by the reversible N−H deprotonation and C−H activation processes of **1a** with the active Cp*Rh(III) species to give a five-membered rhodacycle **A** via a concerted metalation−deprotonation (CMD) process. The



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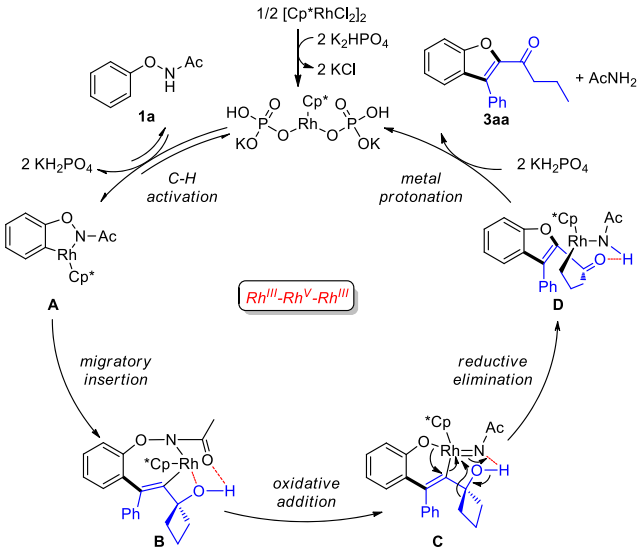
**Scheme 5. Proposed Catalytic Pathway**




regioselective migratory insertion of **2a** into the C−Rh bond stemmed from the formation of hydrogen bonding between the dual directing groups to afford a seven-membered rhodacycle **B**. The oxidative addition of Rh(III) into the N−O bond generated the Cp*Rh(V) nitrenoid **C**, which afforded **D** through the reductive elimination and rhodium insertion into the C−C bond. The following metal protonation led to the formation of **3aa** and the regeneration of the active Cp*Rh(III) species for the next catalytic cycle.

In summary, we have developed efficient and regioselective [3 + 2] annulation/ring opening cascade reactions of readily available *N*-aryloxyacetamides and 1-alkynylcyclobutanols via rhodium(III)-catalyzed redox-neutral C−H/C−C activations using the internal oxidative O−NHAc and −OH as the dual directing groups, which are traceless in the final benzofuran derivatives. This methodology features good functional group compatibility and high yields to provide substituted benzofuran derivatives. Further studies on the synthetic applications of this method and developments of new transition-metal-catalyzed C−H functionalizations with other coupling partners are underway in our laboratory.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b00812.

Experimental procedures, characterization data, and copies of NMR spectra (PDF)

### Accession Codes

CCDC 1884405 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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### Notes

The authors declare no competing financial interest.

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