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One-Pot Construction of Diverse Products using Versatile Cyclopropenones

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Abstract: Tunable C–H activation cascade reactions between quinazolinones and cyclopropenones have been developed. Notably, cyclopropenones, acting as multi-functional building blocks, could be assembled to construct up to 10 distinct heterocyclic scaffolds in a one-pot manner.

Keywords: One-pot construction; C–H activation; Cascade protocol; Quinazolinone; Cyclopropenone

Small molecules with structural complexity and diversity play a crucial role in the fields of drug discovery and chemical biology research.[1] The quest for efficient strategies that provide facile access to new, complex, and diverse molecules continues to be an important and stimulating thrust to academic and industrial research.[2] In the past decades, diversity-oriented synthesis (DOS) has imparted a paradigm shift in the construction of structurally diverse molecular libraries.[3] However, a consensus has emerged that DOS strategy usually demands multiple reaction steps and diverse substrates. The efficient creation of diverse molecules with limited substrates in a one-pot manner remains a formidable challenge.

Featuring step- and atom-economy, transition metal (TM)-catalyzed C–H functionalization represents an appealing strategy to construct molecules, dramatically reshaping the logic of synthetic chemistry.[4] Directing groups (DGs) that direct metal catalysts close to target C–H bonds, facilitating selective bond cleavage and functionalization processes have been widely used in C–H activation (Figure 1A).[5] Notably, The C–H functionalization cascade has emerged as a powerful strategy to construct complex molecules starting from limited feedstocks.[6] However, its potential in scaffold diversity synthesis remains underexplored, largely due to the difficulty in precisely controlling the degree and selectivity of reaction cascades that involve cleavage and construction of multiple chemical bonds.

We hence hoped to develop a tunable C–H activation cascade strategy to realize diversity synthesis using as few substrates as possible in a one-pot manner. As shown in Figure 1, we envisioned that this goal could be achieved by using a versatile coupling reagent. A few prerequisites must be fulfilled concurrently for a proof of concept: 1) the coupling partner is feasible for further transformations and rapid participation in subsequent C–H activation processes (Figure 1B); 2) Multiple C–H bonds can be activated continuously in a controllable manner (Figure 1C); 3) the functionalization sequences of multiple coupling partners are tunable (Figure 1D). Notably, we proposed to finely tune all above-mentioned approaches by solely manipulating reaction conditions, including catalysts, substrate ratio, solvent, etc. The aforementioned maneuvers have not fully been developed, so it is rather challenging to fully integrate them to make more progress in diversity by precisely controllable conditions. For example, in fact, the first step of C–H bond functionalization may lead to significant changes in steric and electronic properties of the substrate, which hampers the efficiency of the succedent C–H bond functionalization. In addition, it is often incompatible for multiple reaction conditions involved in the continuous introduction of different groups.

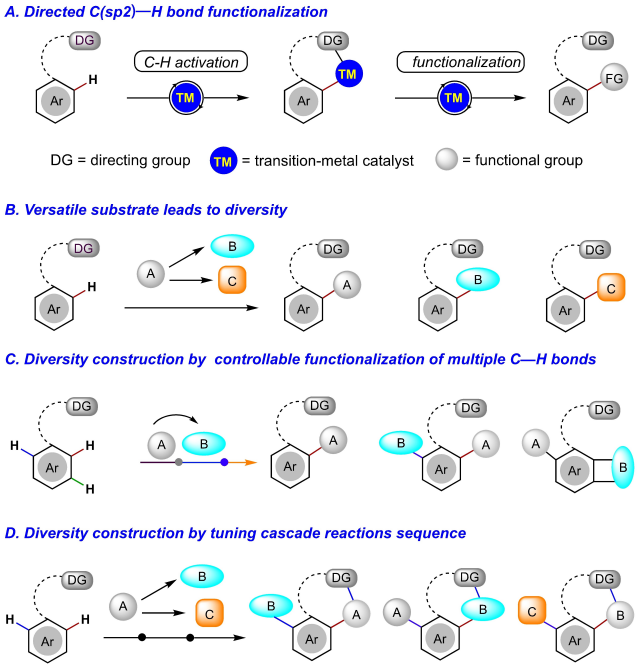

Figure 1. Diversity synthesis design via C–H functionalization.

Known as a highly efficient coupling reagent, cyclopropenone has been widely used in the field of C–H activation.[7] It could be used as a versatile substrate in the construction of different scaffolds.[8] In our previous work, we successfully realized a divergent synthesis strategy to obtain three kind of compounds between cyclopropenones and *N*-nitrosoanilines.[9] Quinazolinones have exhibited a wide range of biological activities[10] and have been used as an excellent DG.[11] Herein, we hoped to explore the C–H activation reactions of cyclopropenones and quinazolinones to realize the above designs.

We initially selected 2-phenylquinazolin-4-(3*H*)-one **1 a** and diphenylcyclopropenone **2 a** as model substrates and ran the reaction in the presence of different catalysts, Ag salts, additives, oxidants, and solvents. To our delight, a tertiary indenol product **3 a** was obtained in 85% yield in the presence of $[RhCp^*Cl_2]_2$ and $AgSbF_6$ in DCM at 100 °C under air (Figure 2B) (for a comprehensive investigation of the conditions, see SI). Indenols are an important class of organic compounds known for several important biological activities such as antibacterial and insecticidal properties (Figure 2A).[12] Thus, the synthesis of indenols also attracted the attention of chemical workers.[13] We investigated the substrate scope and limitations for the indenol product synthesis (Figure 2B). The C6 or C7-(OMe, Me, Cl)-substituted 2-phenylquinazolin-4-ones treated with **2 a** gave quinazolinone derivatives **3 b**–**3 d** in good yields. Quinazolinones bearing various substitutions on the 2-phenyl ring such as 4-*t*Bu, 4-F, 4-Br, 4-CF_3, 3-OCF_3, and 2-OMe reacted effectively

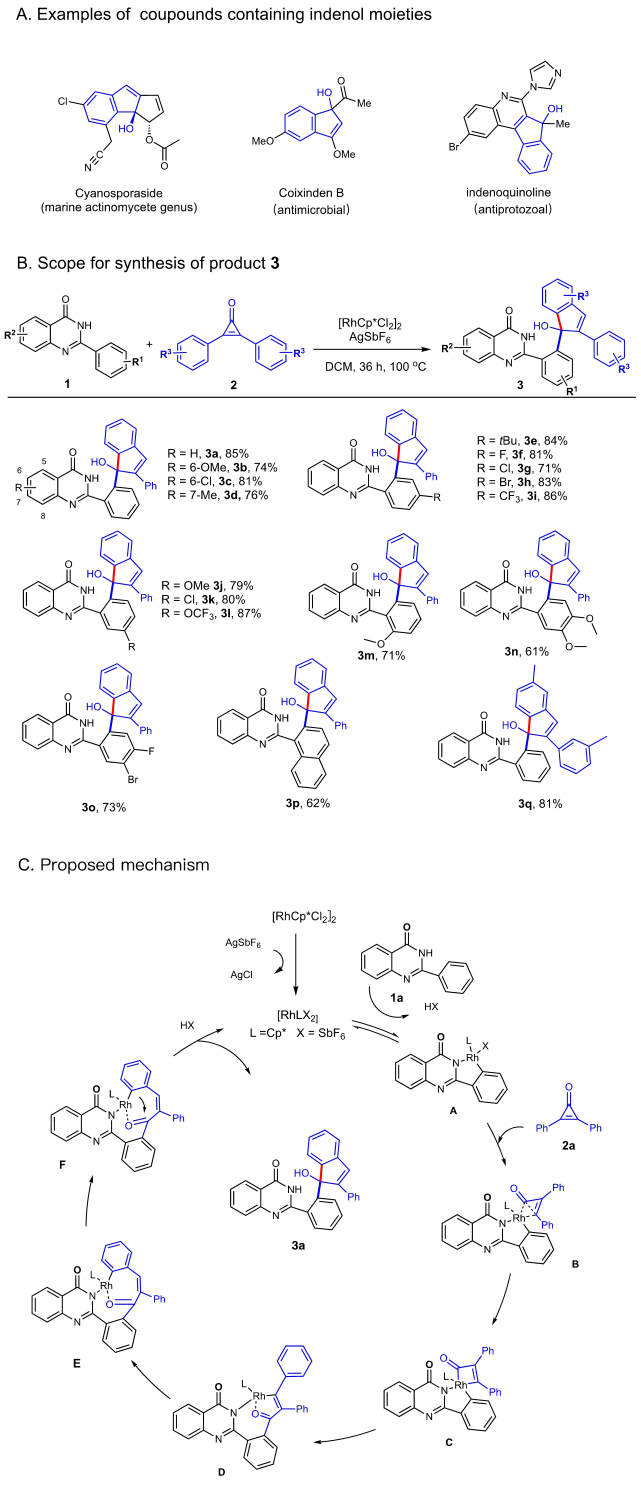

Figure 2. Examples, substrate scope and mechanism.

with **2 a** to afford corresponding products **3 e**–**3 m** in 71–87% yields. No obvious steric or electronic effect was observed. It's worth noting that multi-substituted substrates and 2-(naphthalen-1-yl)quinazolinone could also furnish the targeted molecules in good yields under the standard conditions (**3 n**–**3 p**). Substituted

cyclopropenone was also proved to be good material for this transformation (**3 q**).

Plausible mechanism was proposed to reveal how the product **3 a** was synthesized, based on the corresponding mechanism experiments (see SI for details) and the reported C–H activation works (Figure 2C).[11e,14] Initial anion exchange affords the active [RhCp*X_2] species, which might involve the coordination of the nitrogen atom of **1 a** to the metal centre. Followed by displacement of aromatic C–H bond, the five-membered metal-cycle **A** would be achieved. Intermolecular oxidative addition by cyclopropenone then generates the cyclobutanone Rh(V) intermediate **C**. Subsequent reductive elimination generates C-(benzyl)–C(carbonyl) bond in the intermediate **D. E** will be achieved rapidly by the second C–H activation through the 1,3-migration of the Rh catalyst from the vinyl to aryl. Finally, the product **3 a** is generated through the intramolecular nucleophilic attack and protonation, while releasing the active catalyst for the next catalytic cycle.

Besides, diphenylcyclopropenone could efficiently convert into diphenylacetylene when exposed to ultraviolet.[15] Indisputably, the preparation of symmetric and asymmetric arylacetylene compounds remains to be tedious. Diarylacetylene compounds are usually synthesized from aldehydes and compounds with active hydrogen by bromination and debromination.[16] They can also be obtained by metal-catalyzed coupling methods, which inevitably require the use of halogenated compounds.[17] Because of above difficulties, decarbonylation of cyclopropenones under high-temperature pyrolysis, photolysis or in the presence of certain catalysts (such as [Rh(COD)Cl]$_2$) has become an important synthesis strategy for symmetrically or asymmetrically substituted alkynes.[18] Thus directly using cyclopropenones as the precursor of alkynes to participate in the C–H olefination/cyclization reaction in a one-pot manner is a desired cost-effective process. Here we successfully synthesized a fused tetracyclic heteroarene **4 a**, which was reported in our recent work, by C–H olefination/cyclization cascade between **1 a** and **2 a** under the ultraviolet without any photocatalysts (Figure 3). We also found that the cyclopropenone can be transformed into 2-phenylacetophenone efficiently under the catalysis of 5 mol% K_2CO_3. 2-Phenylacetophenone generated from **2 a** could be used as a benzoyl radical donor to react with **1 a** co-catalyzed by $Cu(OAc)_2$ (5 mol%) and $Pd(OAc)_2$ (5 mol%), affording the product **5 a** (Figure 3). A wide range of quinazolinones and cyclopropenones could be well tolerated under the standard conditions to synthesize product **4** and **5** (see SI for details).

To achieve the goal mentioned before, we further attempted to realize diversity synthesis by one-pot sequential unsymmetrical twofold C–H functionalization. Just as designed, the diverse new products **6** and

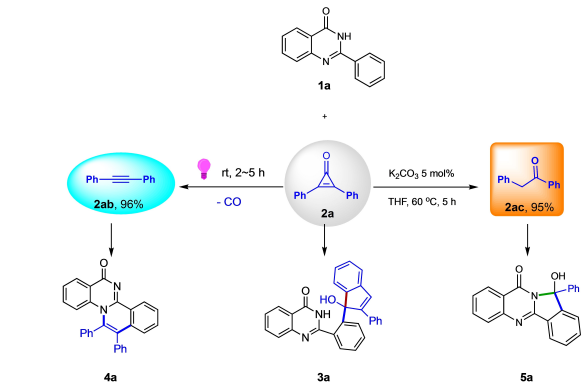

Figure 3. Transformation of cyclopropenone and reactions.

7 were smoothly produced in moderate yield by further reaction between the intermediate **3 a** and diphenylacetylene generated from **2 a** (Figure 4). Interestingly, **3 a** underwent a Rh-catalyzed C–H olefination/cyclization process while its indenol moiety proceeded a ring-opening process to form a chalcone side chain at a high temperature (120 °C). The structure of product **6 d** was unambiguously confirmed by the single crystal X-ray diffraction analysis (Figure 4, **6 d**). At a relatively low temperature (70 °C), the succeeding steps could not take place, which may be due to unfavourable steric factors. Consequently, an alternative pathway is followed. The *meta* C–H bond of intermediate **3 a** was activated to further afford a naphthalene ring product

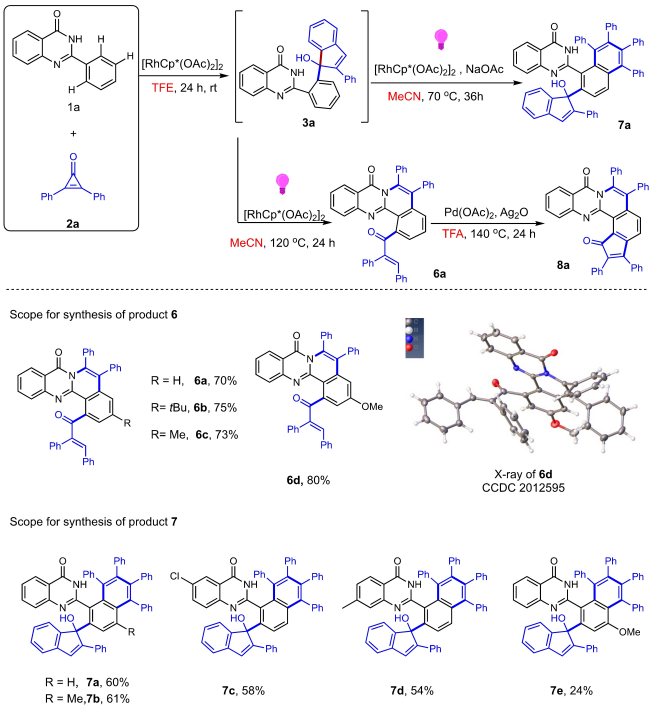

Figure 4. Scope for synthesis of product 6 and 7.

7a. When 1.0 equiv. of NaOAc was used as the additive, the yield of **7a** was further improved. Moreover, product **8a** was obtained by a further Pd-catalyzed coupling of the double bond of chalcone moiety with the C–H bond of the benzene ring (Figure 4).

Subsequently, we tried to tune the sequence of cascade reactions. Gratifyingly, the product **4a** was proved to be an effective intermediate in the following exploration. Through controlling the introduction sequences of diverse functional groups, modules of cascade reactions could be flexibly assembled like Lego, generating more different scaffolds (products **9a**–**12a**) (Figure 4). At first, cyclopropenone **2a** was added to the reaction solution containing **4a**, and compound **9a** was attempted to obtain under the catalysis of $[RhCp^*(OAc)_2]_2$. As expected, compound **9a** was successfully produced at room temperature in considerable yield. Next, the product **10a** was also smoothly obtained by a further Pd-catalyzed coupling of the double bond of chalcone moiety with the C–H bond of the benzene ring. In the presence of $[Ru(p\text{-cymene})Cl_2]_2$, compound **11a** was formed by an unprecedented sequential C–H functionalization on 3- and 2-positions of the benzene ring. The unusual structures of product **11** were confirmed by a single crystal X-ray diffraction analysis of **11c**. Finally, compound **12a** was also successfully obtained by further reaction of the 2-phenylacetophenone transformed from **2a** with the intermediate **4a**.

We surveyed the scope and limitation of substrates for the synthesis of product **6**–**12**. As summarized in Figure 4 and Figure 5, quinazolinones **1** or cyclopropenones **2** substituted with different functional groups at different positions could proceed smoothly under optimal conditions of different reactions, providing the corresponding products in moderate to good yields. Finally, we investigated the mechanism of these reactions, and the possible pathways were proposed in the SI.

In summary, we designed and developed a series of C–H activation reactions which enable direct access to up to 10 heterocyclic scaffolds using cyclopropenone as a multifunctional coupling reagent. The good regio-/chemoselectivity and wide substrate scope for both quinazolinones and cyclopropenones demonstrated the applicability of these reactions. We also investigated and proposed the corresponding mechanism of these reactions. This work highlights the tremendous potential of tunable cascade reactions to deliver compound libraries enriched in structural diversity from versatile substrates. Meanwhile, this one-pot divergent synthesis strategy starting from limited starting materials would provide insights for the improvement of DOS.

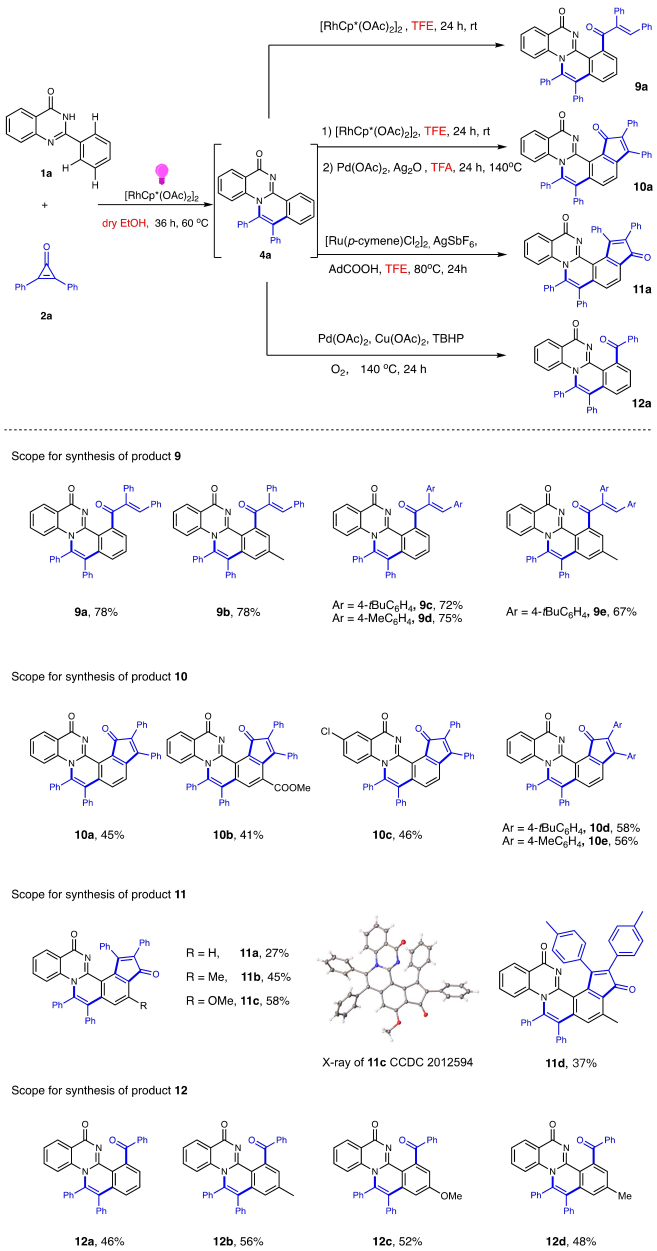

Figure 5. Scope for synthesis of product **9**, **10**, **11** and **12**.

Experimental Section

General procedure for the synthesis of compound **3** (**3a** as an example).

Quinazolinone **1a** (0.10 mmol), diphenylcyclopropenone **2a** (0.105 mmol), $[RhCp^*Cl_2]_2$ (0.05 mmol), $AgSbF_6$ (0.3 mmol) were charged into a pressure tube, to which was added DCM (2.0 mL) under air. The reaction mixture was stirred at 100 °C for 36 h. After completion of the reaction (monitored by TLC), the reaction mixture was cooled to room temperature and the solvent was removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA (about 20/1 ~ 2/1) to afford compound **3a** as a white solid.

CCDC-2012594 and 2012595 contain 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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