

Regioselective Formal [4 + 2] Cycloadditions of Enaminones with Diazocarbonyls through Rh^{III}-Catalyzed C–H Bond Functionalization

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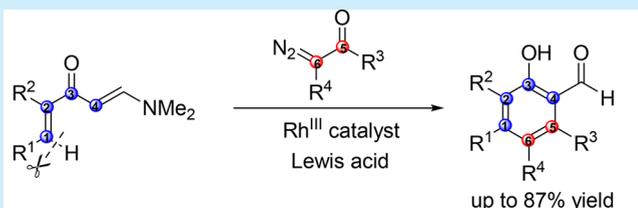
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Supporting Information

ABSTRACT: A regioselective formal [4 + 2] cycloaddition for the assembly of highly functionalized benzene rings was successfully developed. In this reaction, olefinic C–H bond functionalization/cyclization cascade reaction followed by rearomatization led to the desired molecules in one step under mild reaction conditions. This protocol also displays a broad substrate scope and good tolerance to a wide range of functional groups. Additionally, the potential utility for the synthesis of highly conjugated polybenzenes and diversification of natural products was also demonstrated.



- A reactive functional group introduced by C–H bond functionalization strategy
- One-pot assembly of tetra, penta, and hexa-substituted benzene rings
- Double annulation for the synthesis of highly conjugated polybenzenes
- Applications for the diversification of natural products

Benzene rings and their derivatives, especially those containing multifunctionalities, are core structures of many natural products and functional materials¹ (Figure 1a). Therefore, highly efficient methods to construct benzene rings have always received wide attention.² The conventional nucleophilic and electrophilic substitution reactions of benzene derivatives and catalytic cross-coupling reactions were commonly utilized synthetic methods to introduce the desired functional group on the aromatic ring.³ However, there are still deficiencies in these approaches such as the need for prefunctionalized substrates.⁴ In view of this, much effort has been focused on the directed C–H functionalization of aromatic molecules.⁵ Meanwhile, oxidative dehydrogenation of a six-membered carbocycle was also exploited as an effective approach for access to the aromatic skeletons with less substituents.⁶ The lack of efficiency in the sequential functionalization of benzene ring and the interaction of different functional groups restricted such methods from being applied to the construction of poly-substituted aromatic molecules. Nevertheless, it is still a very valuable and challenging task to develop new methods for the construction of the highly functionalized benzene architectures, such as salicylaldehyde derived motifs especially with multi-substituents, which widely exist as the active core in diverse functional molecules.⁷

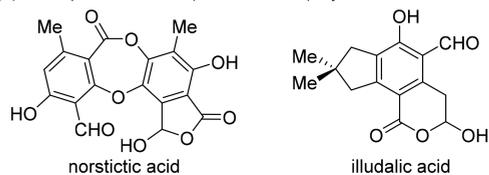
Recently, the emerging metal-catalyzed chelation-controlled C–H bond functionalization of alkenes is shown to be an effective method for the synthesis of diverse linear or *N* (*O*)-heterocyclic functional intermediates (Figure 1b).⁸ However, the regioselective β -C–H bond functionalization of the readily

prepared dialkenyl ketones and analogs has not been fully explored. Also, the cycloaddition pattern for the assembly of benzene rings has a greater advantage in the control of the diversification of the functional groups.⁹ We envisage that the development of the novel and practical [4 + 2] annulation via selective C–H bond functionalization of alkene as the key step may be feasible for the regiospecific synthesis of polysubstituted benzene rings in a one-step manner. For the achievement of this conception, the site-selective carbon insertion into the β -C–H bond of dialkenyl ketones with diazo compounds assisted by weak coordination of ketone to control the substitution patterns remains a great challenge on account of the different C_{sp^2} -H bonds of material molecules and possible competition with the cyclopropanation of alkenes. Additionally, the selection of the appropriate coupling partner carrying a reactive functional group, such as diazocarbonyls, and the identification of the active catalytic system are also challenging tasks for realizing the C–H bond functionalization of the alkene/cyclization cascade reaction followed by rearomatization in a one-step reaction (Figure 1c).

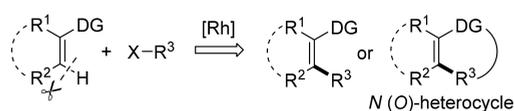
Herein, we report a novel method for straight access to highly functionalized benzene rings by regioselective formal [4 + 2] cycloadditions of enaminones with diazocarbonyls. This reaction smoothly proceeded with a broad range of substrates under mild conditions, affording the desired products in good to excellent yields. Deuterium labeling experiments support the

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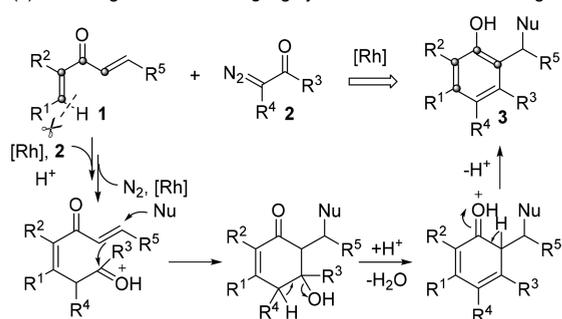
(a) Examples of natural products with polysubstituted benzene



(b) Previous work: olefinic C-H bond functionalization

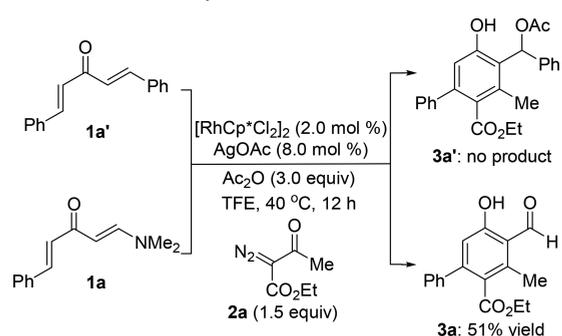


(c) Our design for constructing highly functionalized benzene rings

**Figure 1.** Strategy for the construction of highly functionalized benzene rings.

cycloaddition reaction involving the key step of weakly directed site-selective alkenyl C–H bond functionalization. In addition, this strategy of [4 + 2] annulation was also successfully applied to the synthesis of highly conjugated polybenzenes and diversification of natural products.

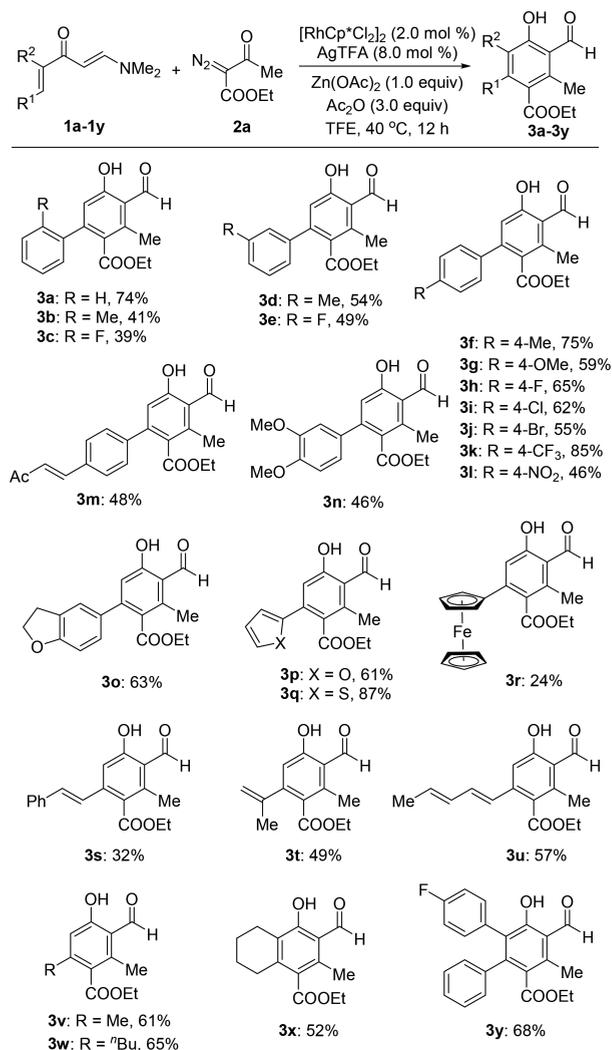
In order to validate the feasibility of the elaborate conception for the synthesis of the multiply substituted benzene rings, we initially explored the reaction of varied dialkenyl ketone analogs with α -diazo- β -ketoester (**2a**) in the presence of Ac_2O using $[\text{Cp}^*\text{RhCl}_2]_2$ and AgOAc as the catalytic system. Interestingly, the benzannulation product (**3a**), which was identified by single-crystal X-ray diffraction, was obtained in 51% isolated yield when the enaminone (**1a**) was exploited (Scheme 1).

Scheme 1. Initial Study

Encouraged by this result, we commenced our investigations into the optimization of reaction conditions by utilizing enaminone (**1a**) and α -diazo- β -ketoesters (**2a**) as the reaction partners. Extensive screening of various reaction parameters revealed that the optimal conditions for the [4 + 2] annulation reaction were the use of $[\text{Cp}^*\text{RhCl}_2]_2$ (2.0 mol %), AgTFA (8.0 mol %), Ac_2O (3.0 equiv), and $\text{Zn}(\text{OAc})_2$ (1.0 equiv) in

the medium of 2,2,2-trifluoroethanol (TFE) at 40 °C under a N_2 atmosphere for 12 h (see the Supporting Information for more details).

With the reaction conditions established, we next evaluated the scope of enaminones (**1**) for the synthesis of multi-substituted salicylaldehydes with another coupling partner, α -diazo- β -ketoester (**2a**). It is worth mentioning that a simple condensation operation to access the diverse enaminones using commercially available chemicals is a practical advantage of this method.¹⁰ As shown in Scheme 2, a number of aryl-substituted

Scheme 2. Scope of Enaminones^{a,b}

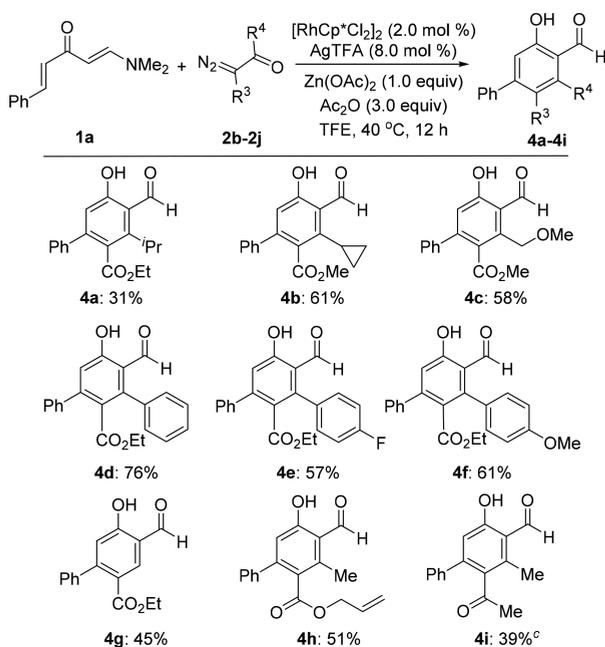
^aReaction conditions: enaminone **1** (0.3 mmol, 1.0 equiv), α -diazo- β -ketoester **2a** (0.45 mmol, 1.5 equiv), Ac_2O (0.9 mmol, 3.0 equiv), $\text{Zn}(\text{OAc})_2$ (0.3 mmol, 1.0 equiv), TFE (1.5 mL), $[\text{Cp}^*\text{RhCl}_2]_2$ (2.0 mol %), AgTFA (8.0 mol %), 40 °C, 12 h. ^bIsolated yields.

salicylaldehydes were obtained in moderate to excellent yields while varying the substituents on the R^1 group (R^1 = aryl) and their different positions. Additionally, the optimal catalytic system was compatible with a variety of functional groups including halide, ether, ester, CF_3 , and NO_2 . For the same group, such as Me, F on the aryl ring, the *para*-substituted substrate (**1f**) showed better reactivity than the *ortho*- and *meta*-position (**1b** and **1d**). The probable reason leading to the activity order (*para* > *meta* > *ortho*) may be steric hindrance.

The electronic effects also play an important role in this reaction. Compared with Me and CF₃ groups, more strongly electron-donating (OMe, **3g**) or electron-withdrawing (NO₂, **3l**) groups on the *para*-position decreased the activity, only affording the desired products in moderate yields. The compatibility of the enone (**3m**) is a significant feature, which revealed that simple enone as the weakly directing group is difficult to chelate with a rhodium catalyst to activate the alkenyl C–H bond. Moreover, the reaction of the enaminones bearing multisubstituted groups on the aryl ring with **2a** can smoothly proceed to give the target products (**3n**, **3o**) in good yields. For the other heterocyclic substituents, namely, furyl, thiophenyl, and ferrocenyl (**3p–3r**), this protocol also showed good tolerance under the standard reaction conditions. In view of the salicylaldehyde derivatives widely exhibited in drug synthesis, alkenyl- and alkyl-substituted salicylaldehydes were further pursued. Gratifyingly, when R¹ was changed to alkenyl, dienyl, and alkyl groups, respectively, the corresponding products (**3s–3w**) were successfully generated. More importantly, representative examples of the disubstituted enaminones (**1x**, **1y**) were prepared for this reaction, affording the desired hexasubstituted salicylaldehydes (**3x**, **3y**) in good yield. These have important potential applications in pharmaceutical synthesis and materials science.

Subsequently, with enaminone (**1a**) as one model coupling partner, a variety of diazocarbonyls (**2**) were then used to explore the effect of substituents on the reaction. As revealed in Scheme 3, in nearly all cases, the reaction performed smoothly to provide polysubstituted salicylaldehydes (**4**) in moderate to good yields. The alteration of the R⁴ substituent ranging from alkyl to aryl group led to the corresponding products (**4a–4f**), and good compatibility was exhibited with bulky isopropyl, cyclopropyl, F, and ether. Further expansion of the scope to

Scheme 3. Scope of Diazocarbonyls^{a,b}

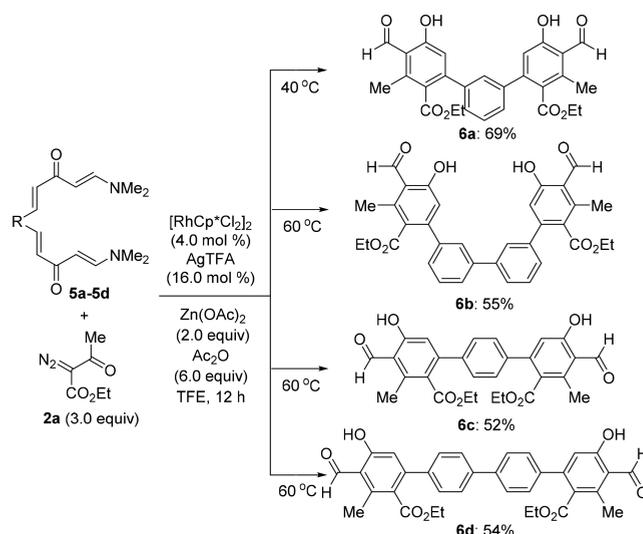


^aReaction conditions: enaminone **1a** (0.3 mmol, 1.0 equiv), diazocarbonyl **2** (0.45 mmol, 1.5 equiv), Ac₂O (0.9 mmol, 3.0 equiv), Zn(OAc)₂ (0.3 mmol, 1.0 equiv), TFE (1.5 mL), [Cp*⁺RhCl₂]₂ (2.0 mol %), AgTFA (8.0 mol %), 40 °C, 12 h. ^bIsolated yields. ^c24 h.

diazocarbonyl ester as a coupling reagent was also successful in achieving the installation of a tetrasubstituted benzene ring (**4g**). In addition, a different R³ group, such as allyl ester (**4h**) and acetyl (**4i**), was also suitable under this catalytic system.

The construction of highly functionalized π -conjugated frameworks is emerging as an effective means for the exploitation of new materials with exceptional electronic and magnetic properties.¹¹ As a further demonstration of the synthetic utility of this formal [4 + 2] cycloaddition protocol, the strategy of double annulation from bis-enaminone and diazocarbonyl is considered to be suitable for the synthesis of such a conjugated polybenzene system. Indeed, this double benzannulation reaction proceeded well, providing extended polybenzene systems (**6a–6d**) in good yields, respectively (Scheme 4).

Scheme 4. Double Annulation for Highly Conjugated Polybenzene Synthesis

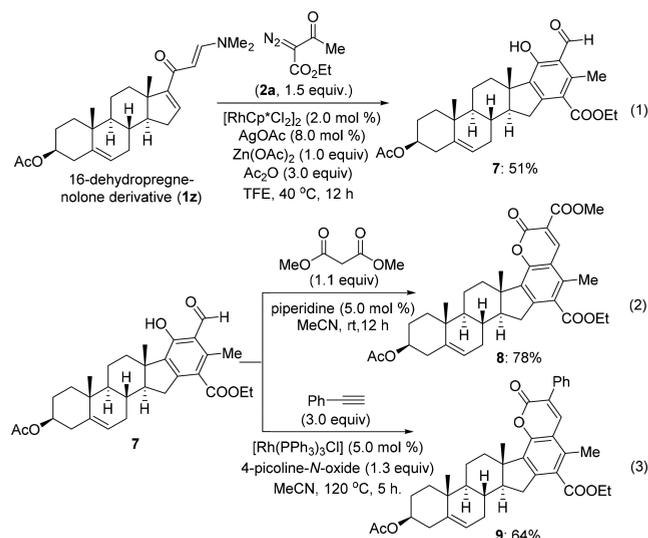


On the other hand, the potential utility of this regioselective formal [4 + 2] cycloaddition accompanying the alkenyl C–H bond functionalization protocol was also explored for improving or altering the biological and pharmaceutical activities of natural products and drugs by introducing highly functionalized benzene rings.¹² For example, when the 16-dehydropregnenolone derivative (**1z**) containing the structural motif of enaminone was subjected to the optimum conditions, the target product was obtained in 51% yield as expected (Scheme 5, eq 1). Additionally, diversification of such structures with both hydroxy and formyl groups was successfully demonstrated by Knoevenagel condensation and hydration–condensation cyclization of compound **7** for the installation of other six-membered rings (**8**, **9**) on the original framework (Scheme 5, eqs 2–3).^{10,13}

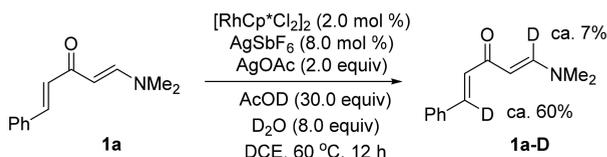
To gain insight into the coordination activation mode of the rhodium catalyst and enaminone, several deuterium labeling experiments were performed. As shown in Scheme 6, the C_{sp³}–H bond at the β -position of the carbonyl group was involved in this benzannulation reaction. It also accounts for the formation of a five-membered rhodacycle through the ketone directing group (please refer to SI for more details related to the mechanistic study and plausible mechanism).

In summary, we have developed a novel [4 + 2] annulation strategy for the synthesis of multisubstituted benzene

Scheme 5. Diversification of 16-Dehydropregnenolone



Scheme 6. Deuterium Labeling Experiments



architectures. This protocol adopted weakly directed regioselective C–H bond functionalization to control the substituent pattern of the benzene ring. The advantages of this transformation include readily prepared materials, mild reaction conditions, a broad substrate scope, and good tolerance to functional groups. In addition, the applications of this consecutive conversion in a one-step reaction to the synthesis of highly conjugated polybenzenes and diversification of natural products were also demonstrated. Further studies on the applications of this method in natural products and functional materials are in progress.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: [10.1021/acs.orglett.8b01540](https://doi.org/10.1021/acs.orglett.8b01540).

Synthetic procedures, mechanistic studies (PDF)
NMR data (PDF)

Accession Codes

CCDC 1836507 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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