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N-Heterocyclic carbene catalyzed *C*-acylation reaction for access to linear aminoenones

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ABSTRACT

An *N*-heterocyclic carbene (NHC)-catalyzed carbonyl nucleophilic substitution reaction between 1-cyclopropylcarbaldehydes and *N*-sulfonyl imines is developed for access to linear β-aminoenone products. The β-aminoenones containing cyclopropyl fragments can be afforded in moderate to excellent yields under mild conditions. The reaction features excellent *trans*-diastereoselectivities and the desired aminoenone products are all afforded as *Z*-isomers.

Cyclopropyl carbonyl derivatives are widely found in natural products with proven biological activities and have significant applications in medicinal research and pesticide development (Fig. 1) [1–11]. For example, *S*-Bioallethrin and Cyhalothrin are commercially available insecticides that are widely used on crops for pest control. Cilastatin sodium is a popular antibiotic drug for the treatment of various infections. Milnacipran hydrochloride is an antidepression drug and can also be used for the treatment of fibromyalgia symptom. Prasugrel is an ADP (adenosine diphosphate) receptor (P2Y12) antagonist that can inhibit platelet aggregation and decrease the risk of coronary syndromes and stroke in patients. Therefore, the development of simple and efficient approaches for the synthesis of cyclopropyl carbonyl derivatives with multiple functionalities have received considerable attention.

N-Heterocyclic carbenes (NHCs) have been extensively explored as organocatalysts for the preparation of various functional molecules [12–29]. Ketones are one class of the most significant functional molecules in both synthetic chemistry [30–34] and biological research [35–37]. They can be efficiently afforded from NHC organocatalytic reactions (Scheme 1). For instance, linear ketones can be obtained from NHC-catalyzed single-electron transfer processes (SET) by using carbaldehyde/carboxylic acid derivatives as the starting materials [38–52]. An alkyl radical precursor bearing a redox active leaving group is generally involved in these processes (Scheme 1a(1)). They can also be efficiently afforded *via* Stetter reactions using aldehydes and various electron-deficient alkene/alkyne molecules as the reaction substrates (Scheme 1a(2)) [53–57]. NHC-catalyzed ionic carbonyl nucleophilic substitution reactions can be used as effective tools for the synthesis of ketone molecules as well (Scheme 1b(1)) [58–75]. Cyclic ketones bearing multiple functional groups are generally formed in this case. For example, the α,β-unsaturated aldehyde can be activated by an NHC catalyst as a nucleophile to react with an electron-deficient alkene substrate *via* a homoenolate [3 + 2] cycloaddition reaction to give the multi-substituted cyclopentanone as the final product (Scheme 1b(1)) [58–63]. The oxidative [4 + 2] cycloaddition reaction between the β-methyl-α,β-unsaturated aldehyde and the electron-deficient alkene substrate promoted by an NHC catalyst can also be used for cyclic unsaturated ketone syntheses (Scheme 1b(2)) [64–71]]. The α,β-unsaturated aldehyde can react as a dielectrophile with the tetra-substituted electron-deficient alkene substrate under NHC-catalyzed oxidative conditions to give the multi-functionalized cyclohexadienone product (Scheme 1b(3)) [72,74]. To the best of our knowledge, the formation of linear ketone molecules *via* NHC-catalyzed carbonyl nucleophilic substitution reactions has never been reported.

Herein, we disclose an NHC-catalyzed oxidative *C*-acylation reaction between the 1-cyclopropylcarbaldehydes **1** [76–78] and the aryl methyl ketimine substrates **2** [79–81] to give the α,β-unsaturated ketones **3** bearing a β-amino group in moderate to excellent yields as single diastereomers (Scheme 1c).

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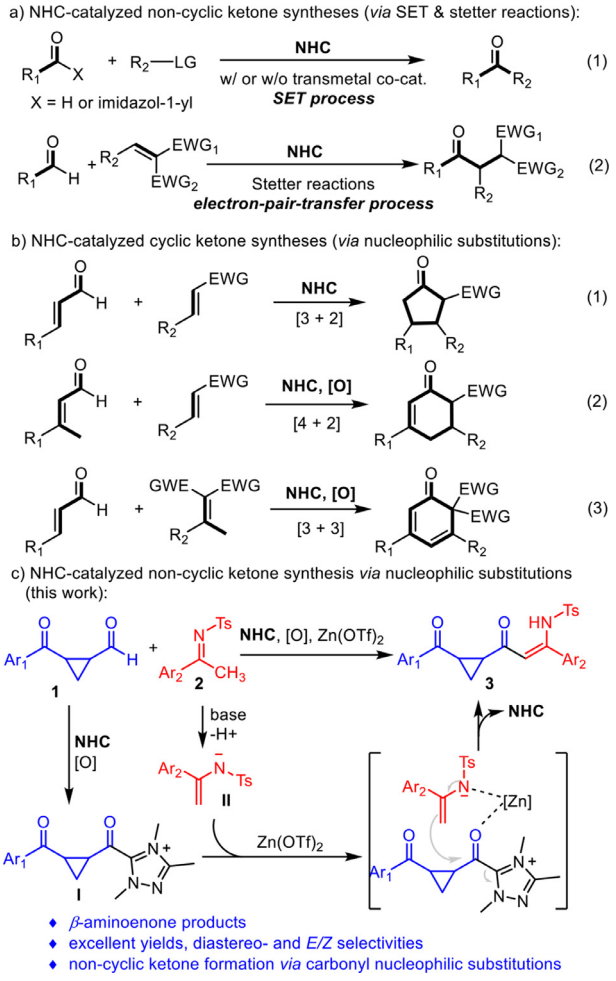


Scheme 1. NHC-catalyzed ketone syntheses.

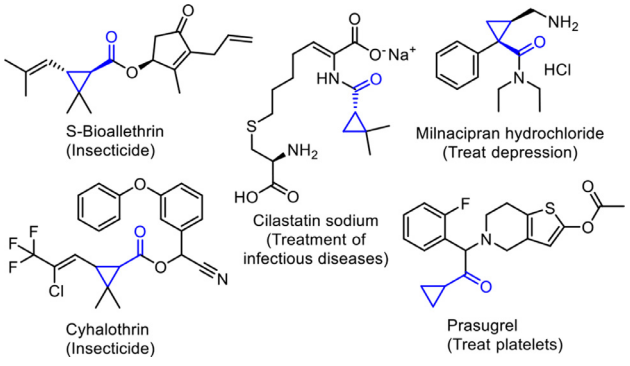


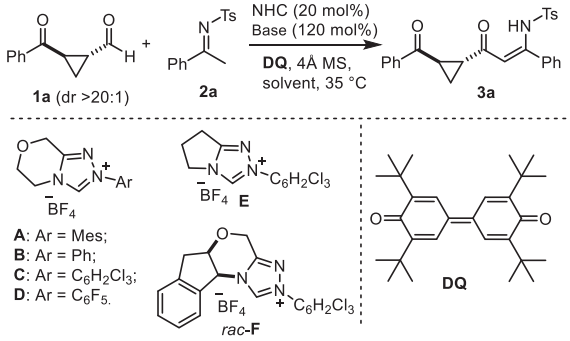
Fig. 1. Bioactive molecules containing cyclopropyl carbonyl skeleton.

Mechanistically, the aldehyde substrate **1** can react with NHC catalyst under oxidative conditions to generate the acyl azolium intermediate **I**. The Lewis acidic $Zn(OTf)_2$ can help bring affinities between the acyl azolium intermediate **I** and the enamine intermediate **II** (generated from deprotonation of the imine substrate **2**). An electrophilic enamine *C*-acylation/proton transfer cascade process leads to the formation of the target enone product, with the free NHC catalyst released for additional catalytic cycles.

The racemic 1-cyclopropylcarbaldehyde **1a** was chosen as the model substrate to react with the *N*-sulfonyl imine **2a** to test the reaction conditions, with key results summarized in Table 1. We initially tested the reaction using various NHC catalysts in the presence of Cs_2CO_3 and **DQ** oxidant in THF at 35 °C for 17 h (Table 1, entries 1–6). The non-chiral NHC catalysts bearing electron-rich *N*-aryl substituents (*e.g.*, **A** & **B**) were not efficient for this transformation (entries 1 and 2). The target product **3a** can be afforded in a promissing yield when using the NHC catalyst **C** bearing *N*-2,4,6-trichlorophenyl group (entry 3). Further decreasing the electron density of the NHC catalyst resulted in no formation of the desired product (entry 4, **D**). Therefore, different catalyst scaffolds were evaluated with the 2,4,6-trichlorophenyl group used as the NHC *N*-substituent (entries 5 and 6). To our delight, the target product **3a** can be afforded in a moderate yield when using the aminoindanol-derived NHC catalyst **F** (entry 6) [82]. Changing the basic additive into other organic or inorganic bases could not improve the reaction yields (entries 7–9). The reaction could also be carried out in a variety of organic solvents (entries 10–12), and the yield of product **3a** could be futher improved to 62% when using DCM as the reaction solvent (entry 10). Lewis acids have been proven to be beneficial to a number of NHC-catalyzed transformations in both reaction efficiency and stereoselectivity. In this regard, a diversity of Lewis acids were tested and $Zn(OTf)_2$ [83–89] was found as the most suitable promoter for the transformation, with the product yield dramatically improved to 91% (entry 14). The Lewis acidic $Zn(OTf)_2$ was believed to act as coordinators that could bring both of the reactive intermediates close to each other to facilitate the catalytic reaction process.

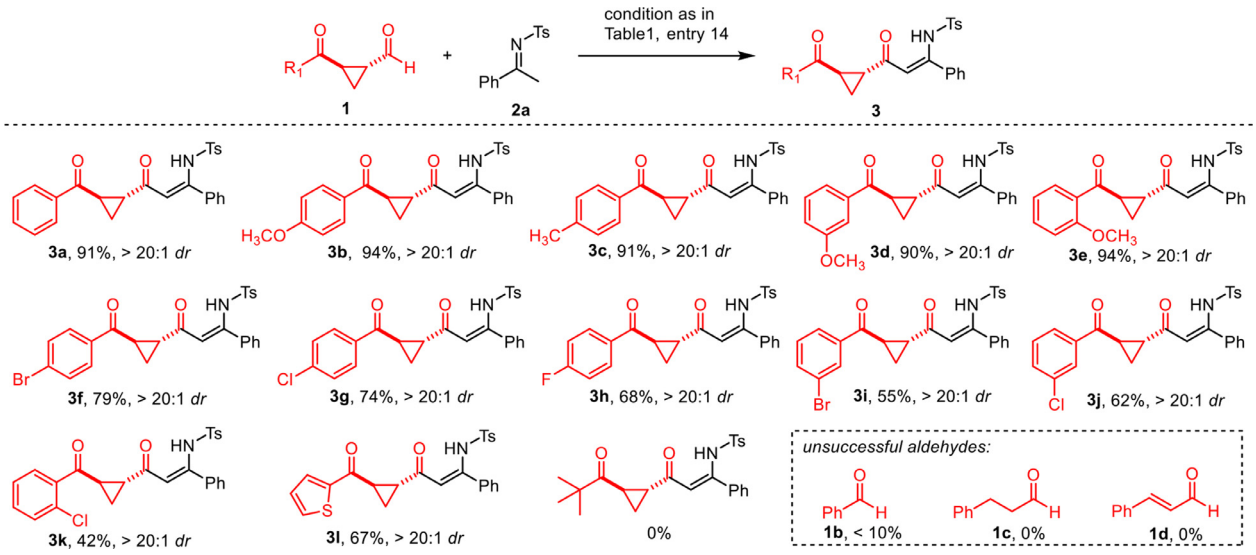
With the optimal reaction conditions at hand (Table 1, entry 14), we next examined the reaction scope of the 1-cyclopropylcarbaldehydes **1** bearing different substituents and substitution patterns (Scheme 2). Electron-donating groups are well tolerated on each position of the phenyl ring of the

Table 1
Condition optimization.[a]

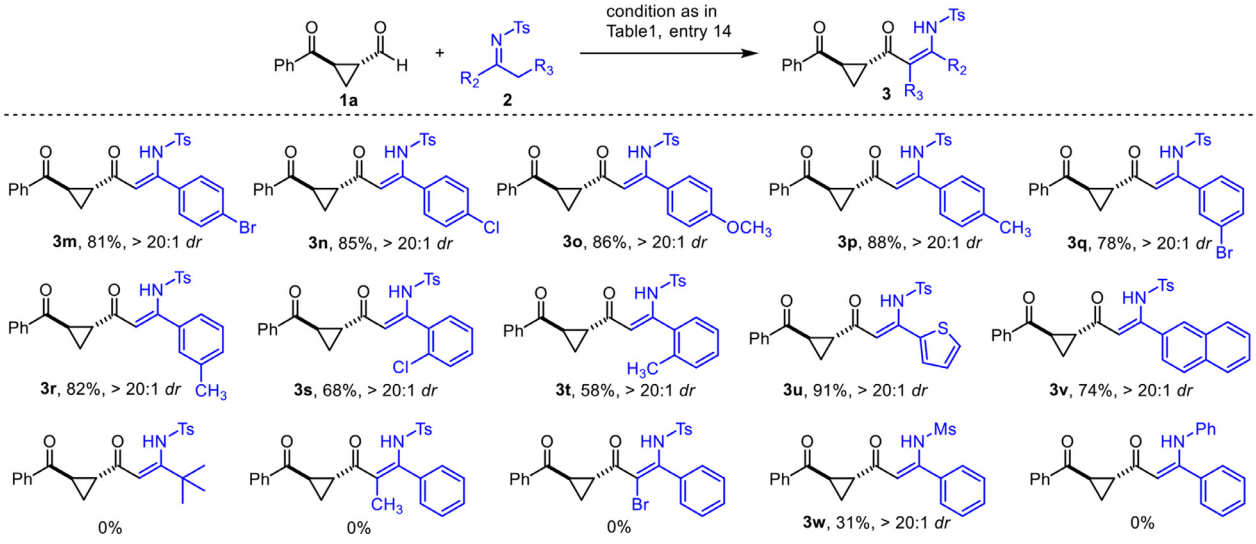


Entry	NHC	Base	Solvent	Yield (%)[b]	*dr*[c]
1	**A**	Cs_2CO_3	THF	<5	
2	**B**	Cs_2CO_3	THF	<5	
3	**C**	Cs_2CO_3	THF	13	>20:1
4	**D**	Cs_2CO_3	THF	0	
5	**E**	Cs_2CO_3	THF	11	>20:1
6	**F**	Cs_2CO_3	THF	47	>20:1
7	**F**	DBU	THF	11	>20:1
8	**F**	Et_3N	THF	0	
9	**F**	*t*-BuOK	THF	28	>20:1
10	**F**	Cs_2CO_3	DCM	62	>20:1
11	**F**	Cs_2CO_3	EA	32	>20:1
12	**F**	Cs_2CO_3	Dioxane	27	15:1
13[d]	**F**	Cs_2CO_3	DCM	46	>20:1
14[e]	**F**	Cs_2CO_3	DCM	91	>20:1

[a] General conditions (unless otherwise specified): **1a** (0.20 mmol), **2a** (0.10 mmol), NHC (0.02 mmol), base (0.12 mmol), **DQ** (0.20 mmol), 4 Å MS (100 mg), and solvent (2.0 mL) at 35 °C for 17 h.
[b] Isolated yield of **3a**.
[c] The *dr* values were determined *via* ^{1}H NMR on the crude reaction mixture.
[d] $Mg(OTf)_2$ (0.02 mmol) as additive.
[e] $Zn(OTf)_2$ (0.02 mmol) as additive.



Scheme 2. Scope of the 1-cyclopropylcarbaldehydes **1.** Reaction conditions as stated in Table 1, entry 14. Yields were isolated yields after purification *via* SiO_2 column chromatography. The *dr* values were determined *via* ^1H NMR on the crude reaction mixture.



Scheme 3. Scope of the *N*-sulfonyl imines **2**. Reaction conditions as stated in Table 1, entry 14. Yields were isolated yields after purification *via* SiO_2 column chromatography. The *dr* values were determined *via* ^1H NMR on the crude reaction mixture.

1-cyclopropylcarbaldehyde **1a**, with the target functional β-aminoenone products (**3b**-**3e**) afforded in excellent yields as single diastereoisomers. In contrast, substitutions on the phenyl ring of the cyclopropylcarbaldehyde **1a** with electron-withdrawing groups generally give the products in relatively lower yields, regardless of their substitution positions (**3f**-**3k**). The phenyl ring of the substrate **1a** could be also switched to a heteroaromatic thiophenyl group, with the desried β-aminoenone product **3l** afforded in 67% yield as a single diastereomer. Replacing the phenyl group of **1a** with an alkyl *t*Bu group led to no desired product, with the imine substrate remained unchanged. Noteworthily, aldehyde substrates without an electron-deficient 1-cyclopropyl group are not efficient in this transformation.

We then examined the scope of the *N*-sulfonyl imine substrates **2** (Scheme 3). Substituents with various electronic properties could be installed on each position of the phenyl rings of the *N*-sulfonyl imines **2**, with the corresponding products obtained in good to excellent yields as single diastereoisomers (Scheme 3, **3m**-**3t**). Replacing the phenyl group with a thiophenyl group in the *N*-sulfonyl imine **2a** could afford the desired product **3u** in 91% yield. Note that, the phenyl ring of the *N*–sulfonyl imine **2a** could also be replaced by a naphthyl ring, and the yield of the target product **3v** was slightly decreased. However, switching the phenyl group of the *N*-sulfonyl imine **2a** into an aliphatic *t*Bu group resulted in no formation of the desired product, with the imine substrate remained unchanged. It is also worthy to note that no desired product could be observed when the R^3 on the *N*-sulfonyl imine **2** was a methyl or a Br group. As a technical note, all the enone products are afforded as *Z*-isomers from our approach, which is probably due to the stablization effects by the intramolecular H-bonding interaction. Noteworthily, the *N*-Ts group of the imine **2a** could also be converted to an *N*-Ms group, with the desired product **3w** obtained in 31% yield under the currently optimized condition. However, no target product was observed when the *N*-Ts group of the imine **2a** was replaced by an *N*-Ph group.

The NHC-catalyzed C-acylation reaction between **1a** and **2a** can be carried out at 1.0-mmol scale without much erosion on the product yield (Fig. 2a). We have also explored the enantioselective dynamic kinetic resolution (DKR) reaction between **1a** and **2a** using various chiral NHC catalysts (*e.g.*, **G**) (Fig. 2b). The

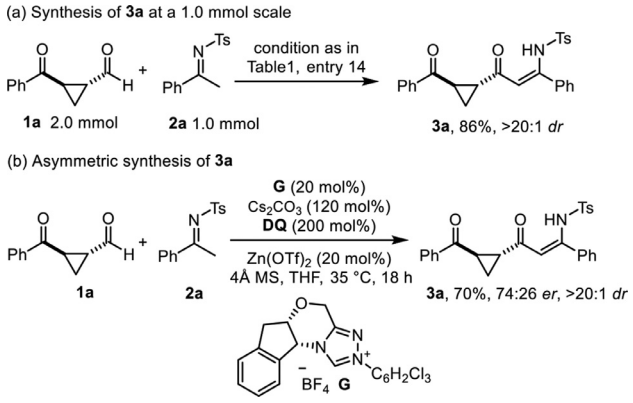
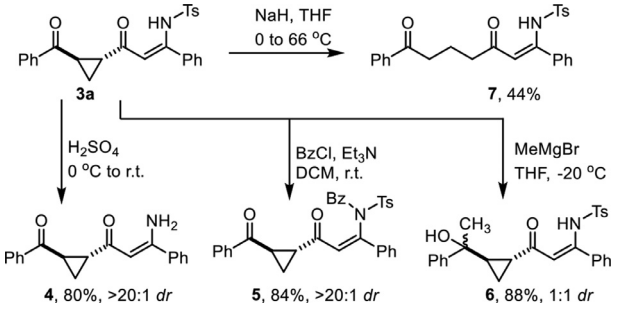


Fig. 2. A 1.0-mmol scale synthesis and an enantioselective synthesis of **3a**.



Scheme 4. Synthetic transformation of **3a**.

β-aminoenones could be obtained in the a moderate *er* value at the current stage.

The β-aminoenones **3** containing cyclopropyl fragments can be easily transformed into novel multifunctional molecules (Scheme 4). For instance, the Ts group of the β-aminoenone **3a** can be removed by sulfuric acid to give the product **4** in an excellent yield as a single diastereoisomer. The β-aminoenone **3a** can react with BzCl to afford the product **5** in 84% yield. The carbonyl group of **3a** can be methylated by the Grignard reagent, with the product **6** afforded in 88% yield. Besides, the cyclopropyl fragments of **3a** can also undergo a ring-opening reaction to obtain the linear ketone product **7** in moderate yield.

In summary, we have developed the first NHC-catalyzed carbonyl nucleophilic substitution reaction for quick and efficient access to linear ketone molecules. β-Aminoenones bearing electron-deficient cyclopropyl fragments are afforded in moderate to excellent yields as single diastereomers. The products feature *trans*-diastereoselective substitutions on the cyclopropyl ring motif and *Z* substitutions around the C=C double bond. Further investigations towards highly enantioselective approaches for the DKR *C*-acylation reaction and the applications of the β-aminoenone products in novel pestcide development are in progress in our laboratories.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.05.084.

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