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Base-promoted [3 + 3] cyclization of cyclopropenones and cyclopropenethiones with amides for the synthesis of 6*H*-1,3-oxazin-6-ones and 6*H*-1,3-thiazin-6-ones†

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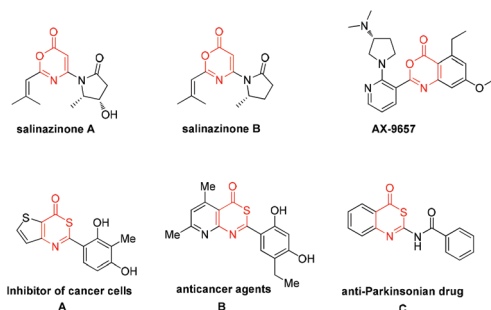
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A facile synthetic method to access 6*H*-1,3-oxazin-6-ones and 6*H*-1,3-thiazin-6-ones has been disclosed via a base-promoted [3 + 3] cyclization reaction of cyclopropenones and cyclopropenethiones with amides. These reactions exhibited excellent yields and good functional group tolerance under metal free and mild conditions.

6*H*-1,3-Oxazin-6-one frameworks have been recognized as important core structures that widely exist in medicinal agents, pharmaceuticals and biologically active molecules,¹ such as salinazinone A, salinazinone B and AX-9657, as shown in Scheme 1. Consequently, synthetic methods of 6*H*-1,3-oxazin-6-ones have garnered much attention over the past few years and various useful methods have been developed for the synthesis of 6*H*-1,3-oxazin-6-ones.² For example, in 2013,

Guan and co-workers achieved palladium-catalyzed oxidative carbonylation of enamides with equivalent Cu(OAc)₂ as an oxidant for the construction of 6*H*-1,3-oxazin-6-ones (Scheme 2, eqn (1)).^{2a} In 2015, Liu's group described a gold-catalyzed cycloaddition reaction of *tert*-butyl propiolates with nitriles (Scheme 2, eqn (2)).^{2b} More recently, Liu and co-



Scheme 1 Biologically active molecules containing 6*H*-1,3-oxazin-6-ones and 6*H*-1,3-thiazin-6-one.

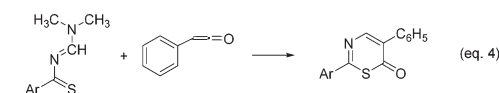
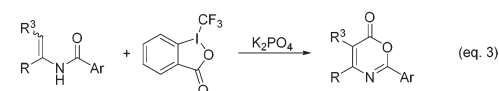
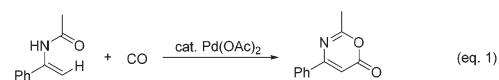
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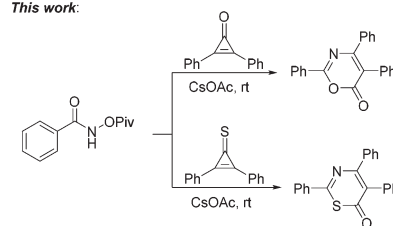
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Previous work



This work:



Scheme 2 Previous work for the synthesis of 6*H*-1,3-oxazin-6-ones and 6*H*-1,3-thiazin-6-one.

workers have successfully realized the formal carbonylation reaction of enamides with Togni's reagent as the CO surrogate for the generation of 6*H*-1,3-oxazin-6-ones (Scheme 2, eqn (3)).^{2c} Obviously, these reaction procedures are often accompanied by transition metal catalysts and the use of complex starting materials is unavoidable. Sometimes, undesired side products were also formed in these reactions. Therefore, the exploration of a simple, more general and convenient synthetic protocol for the synthesis of 6*H*-1,3-oxazin-6-ones is still highly desirable.

On the other hand, their sulfur-containing analogues, 6*H*-1,3-thiazin-6-ones, are also an important class of molecules that are widely used in various fields, including the pharmaceutical industry, agrochemistry and materials sciences.³ For example, molecule **A** has the ability to suppress DNA synthesis in cancer cells,^{3a} especially in C6 cells (Scheme 1). Compound **B** also has anticancer activity *in vitro* (Scheme 1).^{3a} Molecule **C**, structurally neither related to xanthenes nor to adenine, has been identified as an anti-Parkinsonian drug (Scheme 1).^{3b} However, until now, a lack of methodological investigation on the construction of 6*H*-1,3-thiazin-6-ones was a gap in the organic synthetic chemistry domain. Thus far, easily available methods for synthesizing the 6*H*-1,3-thiazin-6-one unit were very limited and have a lot of drawbacks,⁴ such as substrate dependence, poor yields, harsh reaction conditions and so on. For example, in 1975, Quiniou and co-workers synthesized 6*H*-1,3-thiazin-6-one through an intermolecular cyclization reaction of *N*-methylenebenzothioamide with ketene (Scheme 2, eqn (4)).^{4a} Obviously, complex and labile starting materials were used. Undoubtedly, the development of new methods to access 6*H*-1,3-thiazin-6-ones in a simple and efficient way is also very meaningful in the agrochemical field and in medicinal chemistry.

In recent years, the chemistry of strained small rings, particularly three-membered rings,⁵ has been extensively investigated as a class of activated coupling partners.⁶ Among them, cyclopropanones, a kind of representative highly reactive molecules, have also been broadly used in organic synthesis,⁷ because of their unique chemical properties that can react readily with both nucleophilic and electrophilic reagents. Thus, we envisioned whether cyclopropanones could react with amides to afford the desired 6*H*-1,3-oxazin-6-one or 6*H*-1,3-thiazin-6-one scaffold (Scheme 2, this work).

We initially investigated the reaction outcome of 4-bromo-*N*-(pivaloyloxy)benzamide **1a** with diphenylcyclopropanone **2a**. As shown in Table 1, we first tested a set of representative bases, such as Cs₂CO₃, NaOH, DBU and K₂CO₃, using methanol as a solvent, but none of the desired transformations was observed (Table 1, entries 1–4). To our delight, when the reaction mixture was treated with PhCOONa, the desired cyclization product **3a** was obtained in 70% yield (Table 1, entry 5). The use of Et₃N to replace PhCOONa gave **3a** in 85% yield under otherwise identical conditions (Table 1, entry 6). Gratifyingly, the yield was further improved to 90% when CsOAc was used as a base (Table 1, entry 7). The examination of the solvent effect revealed that no better result could be

Table 1 Optimization of the reaction conditions^a

| Entry ^a | Base | Solvent | Yield ^b /% |
|--------------------|---------------------------------|---------------------------------|-----------------------|
| 1 | Cs ₂ CO ₃ | MeOH | nr |
| 2 | NaOH | MeOH | nr |
| 3 | DBU | MeOH | nr |
| 4 | K ₂ CO ₃ | MeOH | nr |
| 5 | PhCOONa | MeOH | 70 |
| 6 | NEt ₃ | MeOH | 85 |
| 7 | CsOAc | MeOH | 90 |
| 8 | CsOAc | CH ₂ Cl ₂ | 60 |
| 9 | CsOAc | Toluene | 72 |
| 10 | CsOAc | DCE | 90 |
| 11 | CsOAc | THF | 85 |
| 12 ^c | CsOAc | DCE | 42 |

^a The reactions were carried out using **1a** (0.2 mmol), **2a** (0.2 mmol), base (1.0 equiv.), and solvent (2.0 mL) in a Schlenk tube. ^b Isolated yields. ^c CsOAc (0.2 equiv.).

obtained (Table 1, entries 8–11). However, when the reaction was carried out in 1,2-dichloroethane (DCE), we found that the reaction proceeded more cleanly and smoothly. The use of 0.2 equiv. CsOAc afforded the desired product **3a** in 42% yield (Table 1, entry 12). Therefore, the reaction should be carried out in DCE and 1.0 equiv. CsOAc should be used as a base.

Under the optimized conditions (Table 1, entry 10), we next focused our attention on the investigation of the scope of amides in the reaction with cyclopropanone **2a**. As shown in

Table 2 Substrate scope for the synthesis of oxazinones **3**^{a,b}

| Structure | Yield (%) |
|-----------|-----------|
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Table 2, when R¹ is an aromatic ring and the leaving group is the OPiv anion, we first examined the electronic effect at the *para*-position of the aromatic ring. As for substrates **1b–1f**, regardless of whether an electron-donating or electron-withdrawing substituent was introduced, the reaction proceeded smoothly, giving the desired products **3b–3f** in good to excellent yields ranging from 65 to 98%. In the case of the *ortho*-substituted substrate (2-methyl) or disubstituted substrate (3,5-dimethyl), the reaction also performed very well, providing the corresponding products **3g** and **3h** in 65% and 89% yields, respectively. Afterwards, we screened a set of leaving groups such as OBz, OMe, OBoc and OFmoc when R¹ is a phenyl group. As can be seen from Table 2, all of them afforded the desired product **3d** in good yields. The heteroaryl-substituted amide **1i** was also compatible, affording the corresponding product **3i** in 65% yield. The structure of **3a** was determined by X-ray diffraction and its ORTEP drawing is shown in Table 2.⁸

To make this cyclization reaction even more integrated, next, R¹ was switched from an aryl group to an alkyl group and R² was changed to a phenoxy group. We found that these substrates were also well tolerated when R¹ is a methyl, an ethyl or an isopropyl group, furnishing the target products **3d**, **3j**, **3k** and **3l** in excellent yields varying from 91–92% regardless of the electronic nature of the phenoxy leaving group.

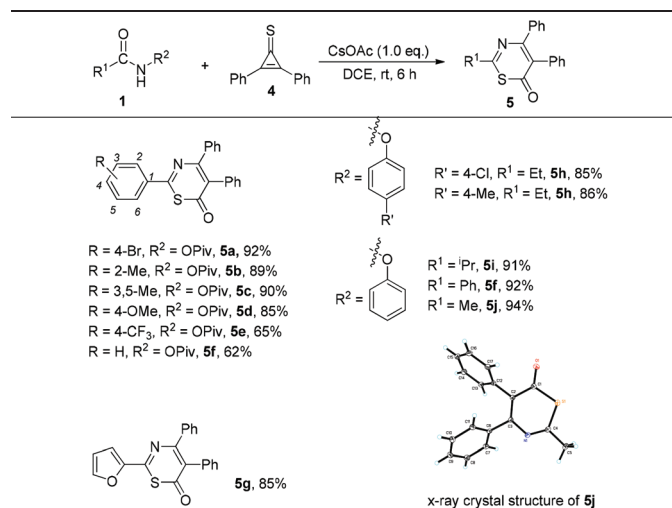
Encouraged by the above results, we next tried to replace the cyclopropanone with cyclopropanethione **4**. Initially, we commenced the investigation in the reaction of *N*-phenoxyacetamide **1j** (R¹ = Me, R² = OPh) with cyclopropanethione **4** under the optimal conditions. We found that 6*H*-1,3-thiazin-6-one **5j** was obtained in 94% yield and its structure was unambiguously determined by X-ray diffraction⁹ (Table 3). After that, we further explored the substrate scope of this cycloaddition reaction and the results are shown in Table 3. Firstly, when cyclopropanethione **4** was treated with various benza-

mides, the desired products **5a–5f** were obtained in 62–92% yields regardless of whether an electron-donating or electron-withdrawing substituent was introduced at different positions of the aromatic ring. The use of *N*-phenoxyacetamides **1h–1j** as substrates also gave the corresponding products **5h–5j** in good yields ranging from 85 to 92%. Heteroaryl-substituted amide was tolerated in this case, giving the desired product **5g** in 85% yield under the standard conditions.

The substrate scope of cyclopropanones was also explored, using two representative amides, 4-bromo-*N*-(pivaloyloxy)benzamide **1a** and *N*-phenoxyacetamide **1j** (R¹ = Me, R² = OPh), for this cyclization reaction. Upon treatment of 4-bromo-*N*-(pivaloyloxy)benzamide **1a** with 2-ethyl-3-phenylcyclopropanone **2b** at 60 °C under the standard conditions provided the desired product **6ab** in 71% yield. Its crystal structure has been determined by X-ray diffraction and the ORTEP drawing is shown in Table 4.¹⁰ On the other hand, the reaction of **1j** with **2b** proceeded smoothly at room temperature, affording the target product **6jb** in 75% yield. However, the reaction of **1j** with 2,3-dibutylcyclopropanone **2c** should be carried out at 60 °C, giving the desired product **6jc** in 51% yield, suggesting that the phenoxy group might be a better leaving group in this transformation. When R³ and R⁴ are different aryl groups, a cycloadduct mixture is formed under the standard conditions. All these results indicated a wide substrate scope in this base-promoted cyclization reaction.

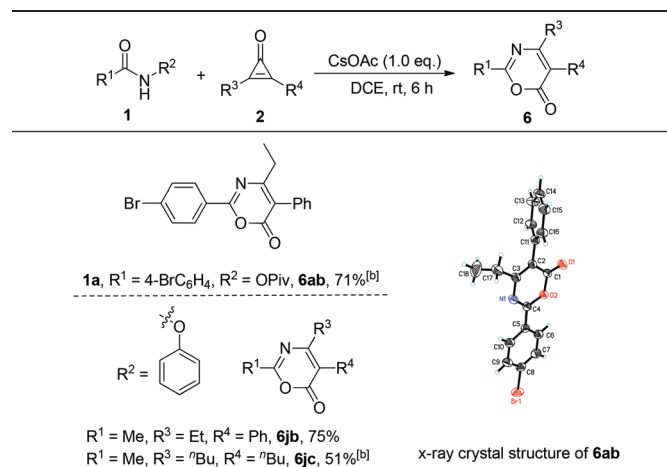
Based on the above results, a plausible mechanism has been outlined in Scheme 3. As for the synthesis of 6*H*-1,3-oxazin-6-ones (X = O), it is commonsense that the intermediate **A** is formed upon treatment of **1d** with CsOAc, which undergoes an isomerization to give the intermediate **B**. The reaction of the intermediate **B** with **2a** provides the intermediate **C**, which undergoes a ring-opening process to yield the intermediate **D**. Then, the desired product **3d** is formed through an intramolecular nucleophilic attack reaction along with the

Table 3 Reaction scope for the synthesis of thiazinones 5^a

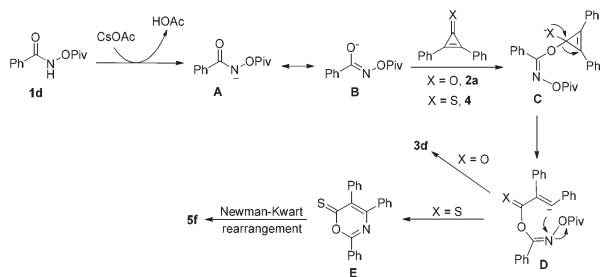


^a Reaction conditions: **1** (0.2 mmol), **4** (0.2 mmol), CsOAc (1.0 equiv.), DCE (2.0 mL). NR = no reaction.

Table 4 Substrate scope for cyclopropanones^a



^a Reaction conditions: **1** (0.2 mmol), **4** (0.2 mmol), CsOAc (1.0 equiv.), DCE (2.0 mL). NR = no reaction. ^b T = 60 °C.



Scheme 3 A plausible reaction mechanism.

release of the OPiv anion. On the other hand, as for the synthesis of 6*H*-1,3-thiazin-6-one ($X = S$), we believe that the intermediate **E** undergoes a Newman–Kwart rearrangement¹¹ to produce a thermodynamically more stable product **5f**.

In summary, we have developed a novel and efficient synthetic protocol to easily access 6*H*-1,3-oxazin-6-ones and 6*H*-1,3-thiazin-6-ones *via* a base-promoted [3 + 3] cyclization reaction of cyclopropenones and cyclopropenethiones with amides. The reaction exhibits a wide substrate scope using easily available starting materials, excellent yields and good functional group tolerance under metal free and mild conditions. The potential utilization and extension of the scope of this new synthetic methodology are currently under investigation in our laboratory.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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- 8 The crystal data of **3a** have been deposited at CCDC with the number 1535814.†
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- 10 The crystal data of **6ab** have been deposited at CCDC with the number 1576769.†
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