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Communication

Silver-catalyzed decarboxylative C–H functionalization of cyclic aldimines with aliphatic carboxylic acids

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ABSTRACT

Silver-catalyzed decarboxylative C–H alkylation of cyclic aldimines with abundant aliphatic carboxylic acids has been realized under mild reaction conditions generating the corresponding products in moderate to good yields (32%–91%). In addition, a gram-scale reaction, late-stage modification of drug, synthetic transformation of the product, and further application of the catalytic strategy were also performed. Preliminary studies indicate that the reaction undergoes a radical process.

Compounds containing a sulfamate moiety are important synthetic targets because they have a broad spectrum of biological activities and serve as preeminent synthetic reagents or intermediates in organic synthesis [1]. For example, readily accessible and stable cyclic sulfamate aldimines have been widely employed in organic synthesis. Among these transformations on the elaboration of cyclic sulfamate aldimines, nucleophilic addition reactions (Scheme 1a) [2–7], annulation reactions (Scheme 1b) [8], ring-expansion reactions (Scheme 1c) [9] and others have been intensively investigated [10–12]. Recently, we have disclosed a silver-catalyzed direct C–H functionalization of cyclic sulfamate aldimines with tertiary cycloalkanols *via* radical-mediated C–C bond cleavage process (Scheme 1d) [13]. Although a vast array of prominent transformations of cyclic sulfamate aldimines have been excavated, direct decarboxylative C–H functionalization of cyclic aldimines with aliphatic carboxylic acids through transition-metal catalyzed radical process has not been reported so far.

Silver-catalyzed decarboxylative radical functionalization of heterocycles is well-known as Minisci reaction [14]. In the past decades, the flourishing Ag-catalyzed decarboxylative strategies have been employed in multitudinous substrates. Among these splendid transformations, aliphatic carboxylic acids are the most frequently used alkyl precursors owing to the advantages of readily available starting materials, low cost, effectiveness, and ease of use in organic synthesis [15]. Since the pioneering report on silver-catalyzed decarboxylative chlorination of aliphatic carboxylic acids developed by Li's group in 2012 [16], a vast array of radical transformations of aliphatic carboxylic acids has been successfully achieved [17,18]. Despite these impressive advances, the development of more robust and readily available methods for the direct C—H functionalization through the decarboxylative cross-coupling of aliphatic carboxylic acids to construct valuable motifs with CO_2 as the only byproduct still remains desirable. Herein, we disclose the first silver-catalyzed decarboxylative C—H functionalization of cyclic aldimines for the assembly of cyclic ketimines *via* a radical pathway (Scheme 1e).

At the outset, cyclic aldimines (**1a**) and cyclohexanecarboxylic acid (**2a**) were selected as model substrates to identify the optimal conditions (Table 1). Pleasingly, the desired product **3a** was obtained in 52% yield with $AgNO_3$ (20 mol%) as catalyst and $K_2S_2O_8$ (3 equiv.) as oxidant in CH_3CN/H_2O (1:1) at 40 °C (entry 2). Further investigation of reaction temperature (entries 2–4) revealed that

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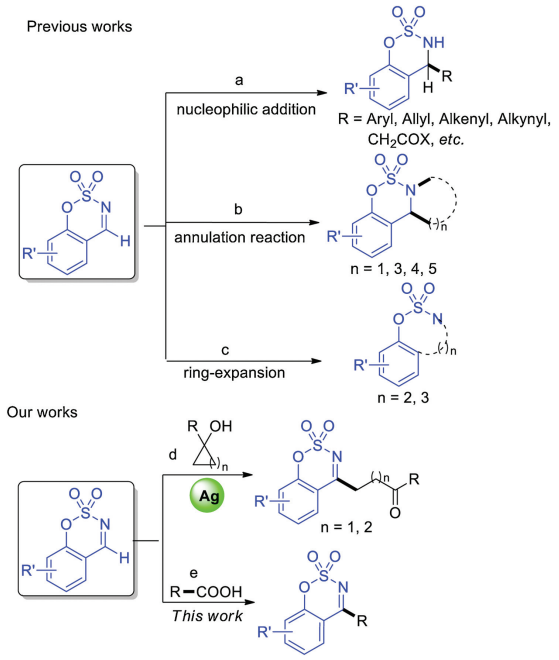
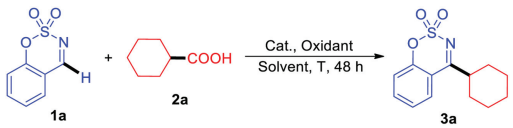


Scheme 1. Catalytic reaction types of cyclic sulfamate aldimines.

Table 1
Optimization of reaction conditions.[a]



Entry	Cat.	Oxidant	Solvent (1:1)	T (°C)	Yield (%)[b]
1	$AgNO_3$	$K_2S_2O_8$	CH_3CN/H_2O	25	trace
2	$AgNO_3$	$K_2S_2O_8$	CH_3CN/H_2O	40	52
3	$AgNO_3$	$K_2S_2O_8$	CH_3CN/H_2O	60	83
4	$AgNO_3$	$K_2S_2O_8$	CH_3CN/H_2O	80	76
5	AgF	$K_2S_2O_8$	CH_3CN/H_2O	60	65
6	AgOAc	$K_2S_2O_8$	CH_3CN/H_2O	60	71
7	$AgNO_3$	$Na_2S_2O_8$	CH_3CN/H_2O	60	80
8	$AgNO_3$	$(NH_4)_2S_2O_8$	CH_3CN/H_2O	60	73
9	$AgNO_3$	$K_2S_2O_8$	Acetone/H_2O	60	46
10	$AgNO_3$	$K_2S_2O_8$	DMSO/H_2O	60	77
11	$AgNO_3$	$K_2S_2O_8$	CH_3CN	60	72
12[c]	$AgNO_3$	$K_2S_2O_8$	CH_3CN/H_2O	60	76
13[d]	$AgNO_3$	$K_2S_2O_8$	CH_3CN/H_2O	60	74
14[e]	$AgNO_3$	$K_2S_2O_8$	CH_3CN/H_2O	60	82
15	$AgNO_3$	–	CH_3CN/H_2O	60	trace
16	–	$K_2S_2O_8$	CH_3CN/H_2O	60	n.d.

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), catalyst (20 mol%), oxidant (0.6 mmol), solvent (2 mL), 48 h.
[b] Isolated yield.
[c] 10 mol% of catalyst was used.
[d] 2.0 equiv. $K_2S_2O_8$ was used.
[e] 4.0 equiv. $K_2S_2O_8$ was used.

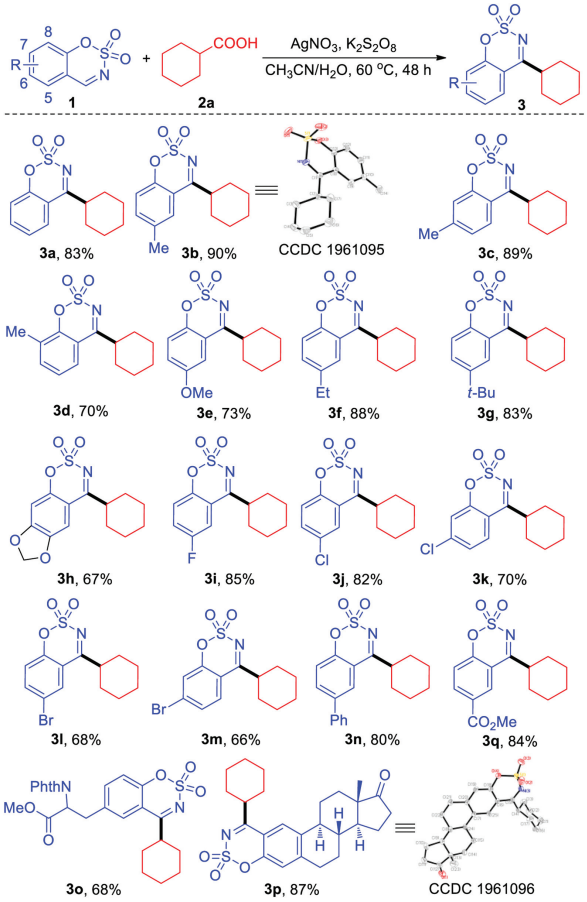
60 °C was the optimal choice (entry 3). Other Ag salts such as AgF and AgOAc could also promote the reaction albeit in slightly lower yields (entries 5 and 6). Subsequently, other oxidants were screened and the results showed that $K_2S_2O_8$ was the optimal oxidant (entries 7 and 8). Next, solvent examination indicated that the reaction worked unfavorably in mixed solvents such as acetone/H_2O and DMSO/H_2O (entries 9 and 10). Using CH_3CN as reaction medium, a decreased yield of **3a** was obtained (entry 11). Then, the amount of catalyst and oxidant were explored. The results showed that changing the amount of catalyst and oxidant led to negative results (entries 12–14). Control experiments indicated that the reaction did not work in the absence of catalyst or oxidant (entries 15 and 16). Thus, based on the above experiments, the optimized conditions were determined to be **1a** (0.2 mmol), **2a** (0.3 mmol), $AgNO_3$ (20 mol%), $K_2S_2O_8$ (0.6 mmol) in CH_3CN/H_2O (1:1) at 60 °C for 48 h.

With the optimized reaction conditions in hand, we set out to explore the substrate scope of this reaction. First, different cyclic aldimines were investigated. As shown in Scheme 2, better results were achieved when substituent was 6-Me or 7-Me (**3b**, **3c**) and the structure of **3b** was confirmed through X-ray crystallographic data. 8-Me-substituted cyclic aldimine gave a relatively lower yield of **3d**, owing to the electronic effect. Substrates with electron-donating substituents such as OMe, Et, ^{t}Bu, 6,7−OCH_2O displayed good reactivity to afford the products **3e-3h** in decent yields. In addition, cyclic aldimines with halogen substituents were well compatible in this transformation, delivering the products in moderate to good yields (**3i-3m**). Cyclic aldimine with 6-Ph was also a good candidate, giving the desired product **3n** in 80% yield. In addition, the cyclic aldimine originated from tyrosine was suitable for this transformation, delivering the expected product **3o** in 68% yield. It is noteworthy that estrone-derived cyclic aldimine could also undertook the radical process to give the desired product **3p** in 87% yield without affecting the ketone group. The structure of **3p** was confirmed through X-ray crystallographic data. The product **3o** and **3p** arguably highlight the current protocol could be applied for late-stage modification of bioactive molecules. To our delight, ester-substituted imine was amenable to this transformation, providing **3q** in 84% yield.

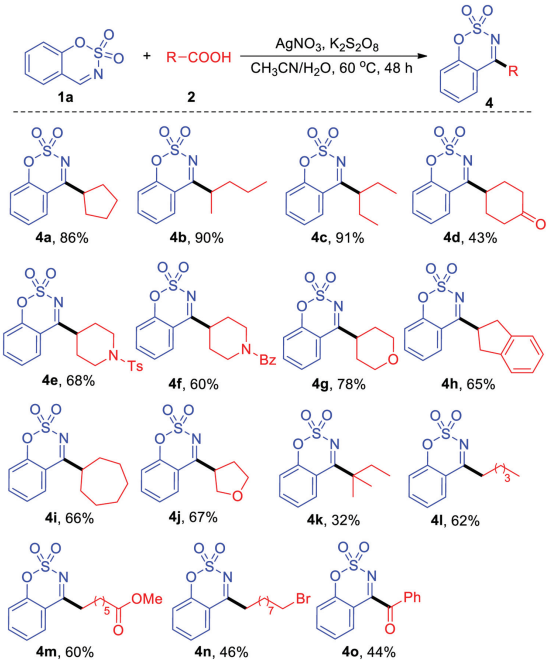
Next, we started to investigate the scope of aliphatic carboxylic acids. As depicted in Scheme 3, secondary cyclic or noncyclic aliphatic carboxylic acids were proved to be suitable alkyl donors, giving the corresponding products **4a-4j** in moderate to excellent yields. However, tertiary aliphatic carboxylic acid exhibited low reactivity probably owing to the steric hindrance (**4k**). Moreover, primary aliphatic carboxylic acids and 2-oxo-2-phenylacetic acid could be engaged in this transformation to give the desired products in moderate yields (**4l-4o**).

To evaluate the synthetic potential of this methodology, the gram-scale reaction, late-stage modification of drug, transformations of the product, and further application of the catalytic strategy were performed. The product **3a** was readily obtained in 78% yield (1.24 g) on a gram-scale reaction by prolonged reaction time (Scheme 4a). Under the standard conditions, fenbufen-a nonsteroidal antiinflammatory drug, and dehydrocholic acid-a choleretic drug, could be modified to corresponding cyclic ketimine derivatives **4p**, **4q** (58%, 62%) without affecting the ketone group (Scheme 4b). To further demonstrate the synthetic utility of this protocol, synthetic transformations of the product **3a** were conducted (Scheme 4c). Reduction of **3a** with 4 equiv. of $LiAlH_4$ gave cyclic sulfamate **5** in 94% yield. When $LiAlH_4$ was increased to 10 equiv., the ring of sulfonylimine was opened and *β*-aminophenol **6** was obtained in 82% yield after trapping with TsCl. Subsequently, we extended this strategy to other cyclic imines (Scheme 4d). To our delight, the reaction of five- and seven-membered cyclic aldimines efficiently proceeded under the standard conditions, and the corresponding product **7a** and **8a** were obtained in 76% and 68% yields, respectively [19].

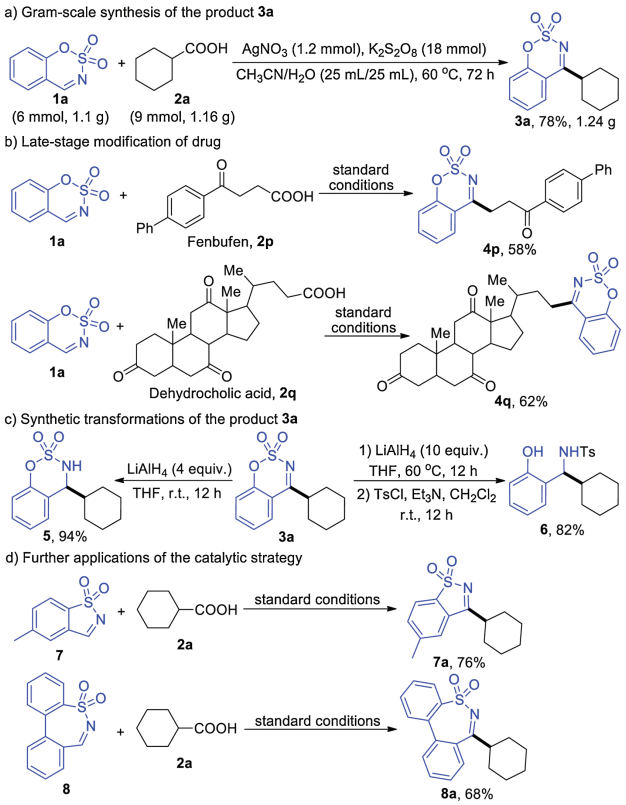
To confirm that radical intermediates might be involved in the reaction, radical-trapping experiments were performed (Scheme 5). When radical scavenger TEMPO was added to mixture of **1a** and **2a** under the standard conditions, the reaction was totally suppressed and the substrates were recovered (Scheme 5a). With



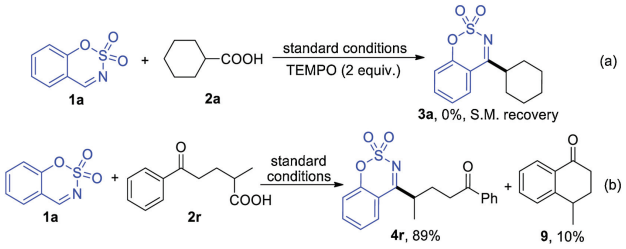
Scheme 2. Scope of cyclic aldimines. Reaction conditions: **1** (0.2 mmol), **2a** (0.3 mmol), $AgNO_3$ (20 mol%), $K_2S_2O_8$ (0.6 mmol), CH_3CN/H_2O (1 mL/1 mL), 48 h, isolated yields.



Scheme 3. Scope of aliphatic carboxylic acids. Reaction conditions: **1a** (0.2 mmol), **2** (0.3 mmol), $AgNO_3$ (20 mol%), $K_2S_2O_8$ (0.6 mmol), CH_3CN/H_2O (1 mL/1 mL), 48 h, isolated yields.



Scheme 4. Synthetic applications.

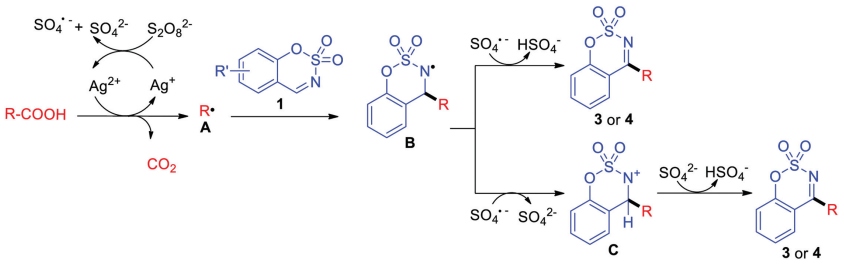


Scheme 5. Mechanistic studies.

aliphatic carboxylic acid **2r** as substrate, along with normal product **4r**, the cyclization by-product **9** was obtained in 10% yield (Scheme 5b). All these experiments suggest that a radical pathway might be involved in the reaction.

According to these mechanistic studies and other previous investigations, a plausible mechanism for the reaction is proposed in Scheme 6. First, Ag^{2+} intermediate is generated by oxidizing Ag^{+} specie with $K_2S_2O_8$, which undergoes the single-electron transfer (SET) with the aliphatic carboxylic acid to generate radical intermediate **A** *via* a decarboxylation. Then, radical **A** attacks cyclic aldimines to afford *N*-radical intermediate **B**, along with the formation of a new C—C bond. Finally, *N*-radical **B** loses a H atom to give the target product. In addition, sulfate radical anion might oxidize the intermediate **B** nitrogen centered radical *via* SET to generate nitrogen cation intermediate **C**, and then the deprotonation of the intermediate **C** occurs to give the desired product.

In conclusion, we have developed a novel, practical and straightforward silver-catalyzed decarboxylative radical reaction of aliphatic carboxylic acids with cyclic aldimines under mild



Scheme 6. Proposed mechanism of decarboxylative radical C–H functionalization.

reaction conditions. The desired products were obtained in moderate to good yields. This transformation features easy accessibility of aliphatic carboxylic acids and broad substrate scopes. In addition, late-stage modification of bioactive molecules, gram-scale synthesis and further synthetic transformations of the product indicate the synthetic potential of this approach. Moreover, five- and seven-membered cyclic aldimines were also applicable for this transformation. Mechanistic studies showed that the reaction might undergo a radical pathway. Further exploration of the synthetic utility of this transformation is ongoing in our laboratory.

Declaration of competing interest

The authors report no declarations of interest.

Acknowledgments

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.cclet.2021.03.011.

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