Silver-Catalyzed Acyl Nitrene Transfer Reactions Involving Dioxazolones: Direct Assembly of *N*-Acylureas

Zheng-Lin Yang,^[a] Xin-Liang Xu,^[b] Xue-Rong Chen,^[a] Zhi-Feng Mao,^{*[a]} and Yi-Feng Zhou^{*[a]}

Dioxazolones and isocyanides are useful synthetic building blocks, and have attracted significant attention from researchers. However, the silver-catalyzed nitrene transfer reaction of dioxazolones has not been investigated to date. Herein, a silver-catalyzed acyl nitrene transfer reaction involving dioxazolones, isocyanides, and water was realized in the presence of Ag_2O to afford a series of *N*-acylureas in moderate to good yields.

The incorporation of an amide moiety into a molecule is of considerable importance because of the presence of this substructure in many biologically active molecules and natural products.^[1] In the late 1960s, 1,4,2-dioxazol-5-ones (dioxazolones) 1 were identified by Sauer and Mayer as safe alternatives to acyl azides for *N*-acyl nitrene formation.^[2] Owing to their low cost, ready availability, and environmental friendliness, 1,4,2dioxazol-5-ones have been extensively studied as amidating reagents in the past several years. In 2009, Dubé et al. published a pioneering method to prepare isocyanates by employing 1,4,2-dioxazol-5-ones.^[3] Later, Bolm and co-workers reported the first light-induced ruthenium-catalyzed N-acyl nitrene transfer to sulfides and sulfoxides by the decarboxylation of 1,4,2dioxazol-5-ones (eq 1, Scheme 1).^[4] Subsequently, dioxazolones were reported as amidating reagents for Rh-catalyzed C-H amidation by the Chang group.^[5] Thereafter, a series of Rh-,^[6] Co-,^[7] and Ir- catalyzed^[8] C–H amidation reactions were developed using this amidating reagent (eq 2, Scheme 1). Furthermore, Li, Zhu, Glorius, Ackermann, and other research groups described the cascade C-H amidation and cyclization reactions for the syntheses of various heterocycles.^[9] Bruin and co-workers recently reported the fast N-acyl amidations via Cucatalyzed three-component reaction of aryl acetylenes, amines, and 1,4,2-dioxazol-5-ones (eq 3, Scheme 1).^[10] However, to the best of our knowledge, the silver-catalyzed reaction of 1,4,2dioxazol-5-one via acvl nitrene has not been reported in the literature.

[a]	ZL. Yang, XR. Chen, Dr. ZF. Mao, Dr. YF. Zhou
	Department of Pharmacy, College of Life Sciences
	China Jiliang University
	Hangzhou 310018, China
	E-mail: zhouyifeng@cjlu.edu.cn
	18a0904100@cjlu.edu.cn
[b]	XL. Xu
	Apeloa Pharmaceutical Co., Ltd.
	Hengdian 322118, China
	E-mail: xinliang.xu@apeloa.com

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(a) Transition-metal-catalyzed reactions involving dioxazolones



(c) This work



Scheme 1. Background for this research.

Isocyanides, as versatile building blocks with diverse reactivities, have been extensively investigated in the transitionmetal-catalyzed transformations to construct numerous valuable nitrogen-containing compounds.[11] Among the studied transition-metal-catalysts investigated, silver catalyst exhibits a remarkable capacity for the activation of isocyanides, allowing their reactions with many different types of electrophilic reagents, such as carbonyls,^[12] imines,^[13] alkenes,^[14] α -imino esters,^[15] allenoates,^[16] fulvenes,^[17] and alkynes^[18] for constructing diverse heterocyclic compounds.^[19] Additionally, silver salts have been also utilized in the radical chemistry of 2isocyanobiphenyls for the construction of 6-substituted phenanthridines.^[11h] Furthermore, the reactions between two different isocyanides catalyzed by silver salts have been reported.^[20] Despite these elegant strategies, the reactions of isocyanides with acyl nitrene precursors, i.e., dioxazolones, have



not been reported. Recently, Wei and co-workers reported the syntheses of pyrimidinediones via the Ag₂O catalyzed difunctionalization of an isocyano group with cyclic oximes (eq 4, Scheme 1).^[21] Inspired by their work and considering that dioxazolones and cyclic oximes have structural similarities, it was speculated that the reactions between dioxazolones and isocyanides may proceed by silver catalysis. Herein, the one-pot syntheses of a series of *N*-acylureas via silver-catalyzed acyl nitrene transfer reactions involving dioxazolones, isocyanides, and water are reported for the first time (eq 5, Scheme 1).

Notably, N-acylureas, a class of acyclic compounds, have been widely applied in agricultural and pharmaceutical chemistry.^[22] Therefore, a number of strategies for the construction of N-acylurea compounds have been developed. A typical approach relies on the reactions of isocyanates with amides (Scheme 2, path A).^[23] Another strategy involves the Nacylation of substituted ureas (Scheme 2, path B, and C).^[22a,24] The reactions of carboxylic acids with carbodiimides have also been frequently used for the syntheses of N-acylureas (Scheme 2, path D).^[25] Recently, Zhu et al. reported a method to prepare N-acylureas via the C–H/N–H bond activation between N-alkoxy aromatic amides and N,N-disubstituted formamides (Scheme 2, path E).^[26] Yadav and co-workers reported the syntheses of N-acylureas via the reactions of two different carboxamides (Scheme 2, path F).^[27] Some other methods to generate N-acylureas have also been reported.^[28] Herein, a different pathway toward preparing N-acylurea derivatives utilizing 3-substituted-1,4,2-dioxazol-5-ones, isocyanides, and water as the starting materials is reported.

First, 3-phenyl-1,4,2-dioxazol-5-one (**1a**) and ethyl isocyanoacetate (**2a**) were selected as the model substrates to investigate the reaction conditions. The resulting product *N*acylurea **3aa** was isolated in 57% yield (entry 1, Table 1) after the reaction using 10 mol% Ag₂O as a catalyst in 1,4-dioxane for six hours at 80°C in air. Considering the presence of two H atoms and one O atom in the product, it was speculated that residual H₂O in undried solvent could participate in this reaction to provide the *N*-acylurea product. Based on this preliminary result, further optimization of the reaction conditions was performed. Investigation of various solvents (entries 2–7, Table 1) led to the identification of DMSO as an optimal solvent



Scheme 2. The approaches for the synthesis of *N*-acylureas.



[a] Reaction conditions: All the solvents were commercially available and used as received. **1a** (1.1 equiv), **2a** (0.5 mmol), catalyst, solvent (2 mL), temperature, stirred for 6 h in air. [b] Isolated yield. NR = no reaction. [c] Reaction was performed in DMSO/H₂O (10/1, v/v). [d] 0.5 g of 4 Å MS was added and the reaction was performed under N₂ atmosphere. [e] 15 mol% Ag₂O was added.

(73% yield, entry 7, Table 1). When ethanol was used as the solvent, the yield of the target product decreased significantly owing to the formation of some unidentified by-products (entry 5, Table 1). In the absence of Ag₂O, no reaction occurred (entry 8, Table 1). Several commercially available Ag salts were then tested as catalysts. The reactions proceeded to afford product 3 aa in acceptable yields (AqOAc, 65% yield, entry 9; Ag₂CO₃, 63% yield, entry 10; AgCO₂CF₃ 52% yield, entry 11). When AqNO₃ was used, the target product **3 aa** was obtained in only 31% yield. To determine the effect of water on the reaction, its content was varied. When DMSO/H₂O (10:1) was used as the solvent, the yield of the product decreased to some extent (entry 13, Table 1) along with a side product, symmetrical N,N'-diphenyl urea.^[2,3,4] When the reaction was performed in strictly dry DMSO under N₂, the reaction yield decreased dramatically, and an unknown side product was formed (entry 14, Table 1). The above results indicated that water traces in the reaction mixture were necessary for the reaction. Further screening of the reaction temperature and catalyst amount showed that 80°C and 10 mol% catalyst loading were appropriate. Considering these results, the optimal conditions included 10 mol% Ag₂O in DMSO at 80 °C in air.



With the optimum reaction conditions in hand, a wide range of 3-substituted-1,4,2-dioxazol-5-ones 1 was reacted with ethyl isocyanoacetate (2 a) to examine the generality of the reaction (Scheme 3). The reactions of 3-substituted-1,4,2-dioxazol-5-ones containing *para*-methoxyphenyl and *meta*-meth-ylphenyl groups proceeded smoothly to afford the desired products (3 ba-3 ca) in good yields (63–76% yields), and the structure of 3 ba was confirmed by X-ray crystallography (CCDC 1995067, for details, see Supporting Information). The sterically hindered *ortho*-methylphenyl group was also compatible in this reaction, albeit affording a lower yield (3 da, 31% yield). The 2-substituted-1,4,2-dioxazol-5-ones with electron-withdrawing



Scheme 3. Reaction substrate scope: dioxazolones. [a] Reaction conditions: The solvent was commercially available and used as received. 1 (1.1 mmol), 2a (1.0 mmol), Ag₂O (10 mol%), DMSO (4 mL), 80 °C, 6 h in air. [b] Isolated yield of 3 aa–3 pa.

substituents (*p*-Cl, *p*-Br, *p*-CF₃; **1e**-**1g**) reacted to provide the corresponding products (**3ea**-**3ga**) in good yields (71–73% yields). Dioxazolones bearing a heteroarene substituent at the 3-position also reacted with **2a** to afford the desired products in moderate yields (furan, **3ha**, 53%; thiophene, **3ia**, 57%). Interestingly, when 3-naphthyl-1,4,2-dioxazol-5-one (**1***j*) was utilized, the reaction occurred smoothly in good yield (82%). Fortunately, aliphatic 1,4,2-dioxazol-5-ones were also identified as suitable substrates resulting in the desired products (**3ka**-**3na**) in good yields. Furthermore, styryl-substituted 1,4,2-dioxazol-5-one **1o** furnished the corresponding product **3oa** in 69% yield. Interestingly, the drug-like product **3pa** could be also prepared from **1p** in good yield.

Thereafter, the reactions of **1a** with various isocyanides were also examined (Scheme 4). Isocyanoacetates bearing different ester groups were all suitable substrates, affording products **3ab–3ad** in moderate to good yields (48–71%). Surprisingly, when methyl 2-isocyano-3-phenylpropanoate **2d** was used as the substrate, the reaction proceeded to afford the desired product **3ad** in moderate yield (48%). Unfortunately, when 2-isocyano-1-morpholinoethan-1-one **2e** and tosylmeth-ylisocyanide (TosMIC) **2f** were tested in the reactions with **1a**, the desired products were not obtained under any optimized condition. Similarly, 1-isocyano naphthalene **2g** failed to yield the desired product in the reaction.

To test the practicality of the methodology, a gram-scale synthesis of *N*-acylurea was performed under standard conditions. Here, 8.8 mmol of ethyl 2-isocyanoacetate 2a was reacted with 9.7 mmol of (italic E)-3-styryl-1,4,2-dioxazol-5-one 1o and water to afford 1.82 g of the corresponding *N*-acylurea 3oa in 75% yield (Scheme 5a). To investigate the mechanism of



Scheme 4. Reaction substrate scope: isocyanides. [a] Reaction conditions: The solvent was commercially available and used as received. 1 a (1.1 mmol), 2 (1.0 mmol), Ag₂O (10 mol%), DMSO (4 mL), 80 °C, 6 h in air. [b] Isolated yield of 3 ab–3 ag.



Scheme 5. (a) Gram-scale synthesis of 3 oa and (b) Isotopic labeling experiment.

the reaction, O^{18} -labelled water was added to the reaction mixture to determine the oxygen atom source of the urea moiety (Scheme 5b). Mass spectrometry analysis of the target product revealed that the O^{18} atom was incorporated into the product [O^{18}]**3aa**.

Based on the results and previously reported studies,^[5,29] a possible reaction mechanism shown in Scheme 6 is postulated. Through one of the heteroatoms, 3-substituted-1,4,2-dioxazol-5-one 1 interacts with the silver(I) salt to generate silver complex A.^[5] The subsequent loss of CO₂ provides silver *N*-acyl nitrene complex B.^[29] Then, the interaction of isocyanide 2 with *N*-acyl nitrene complex B allows an *N*-acyl nitrene transfer reaction via complex C, generating intermediate D and active silver(I) catalyst for the next cycle. The intermediate D is then converted into the desired product 3 by reaction with H_2O . However, the reason for the limitation of the substrate scope to isocyanoacetates remains unclear.



Scheme 6. Proposed mechanism.

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Deposition Number 1995067 (for **3ba**) contains the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service www.ccdc.cam.ac.uk/structures.

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Conflict of Interest

The authors declare no conflict of interest.

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