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Letter

Ag(I)-Catalyzed Addition of Cyclopropenones and Nitrones to Access Imides

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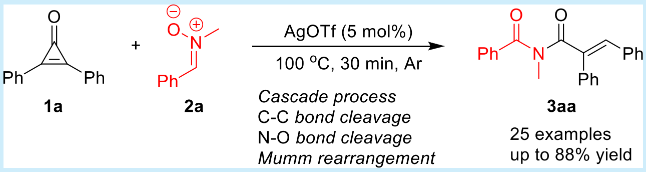
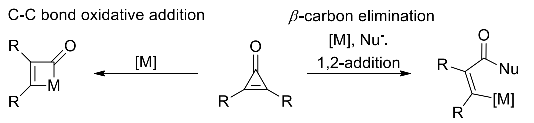
ABSTRACT: An unprecedented Ag-catalyzed addition reaction of cyclopropenones and nitrones to access imides was developed. Sequential C−C bond cleavage, N−O bond cleavage, and Mumm rearrangement were uncovered in this process. This protocol exhibited high efficiency, regioselectivity, good yields, and a broad tolerance of various functional groups.



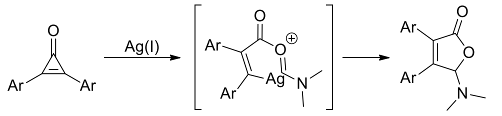
For the past few decades, significant advances have been made in the field of C−C bond activation, which is an intriguing process from both mechanistic and synthetic standpoints.[1] As the smallest of the compounds exhibiting Hückel aromaticity, cyclopropenones are ideal candidates for C−C bond cleavage.[2,3] Because of their ambiphilic properties, cyclopropenones can serve as excellent 3C synthons, which provide wide utility in numerous chemical transformations.[4−6] Generally, there are two different modes for metal-catalyzed ring-opening of cyclopropenones, and *β*-carbon elimination is one of the elementary ways for cleavage of the C−C(O) bond (Scheme 1a).[6] However, few examples have been documented in this field, which represents a highly desirable but unmet goal. Previously, Gleiter pioneered Cu-promoted dimerization reactions with cyclopropenones.[6a] Afterward, Matsuda made important progress in the alkynylation and cycloaddition reactions of cyclopropenones.[6b,c] Li and co-workers have made significant achievements in C−H acylation reactions with cyclopropenones[6f,g] and further transformations.[6e] Recently, our group has successfully realized an unprecedented Ag-catalyzed [3 + 2]-annulation of cyclopropenones.[6h] Despite these successes, it is still necessary to explore new chemistry featuring *β*-carbon elimination, thus broadening the scope and applications of the ring-opening of cyclopropenones.

Scheme 1. Metal-Catalyzed Ring-Opening of Cyclopropenones

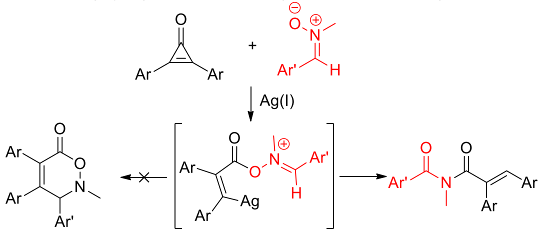
a) Transition-metal catalyzed ways of ring-opening of cyclopropenones



b) Our previous work: cycloaddition of cyclopropenone to *N*,*N*-dimethylformamide



c) This work: highly regioselective coupling via C-C /N-O cleavage/ Mumm rearrangement



In our previous studies, we represented an active C−Ag intermediate generated by *β*-carbon elimination, which subsequently underwent intramolecular nucleophilic addition (Scheme 1b).[6h] We became intrigued by the possibility of using the active C−Ag intermediate to initiate a new reaction to access diverse skeletons. Such a strategy may suffer from major two challenges: (a) the initial attack of the nucleophilic atom to C-1 (not C-2) of cyclopropenone and (b) to find a suitable partner driving C−Ag intermediate into a new chemistry (avoiding direct cycloaddition). Herein, we report our success in the Ag-catalyzed ring-opening of cyclopropenone with nitrones[7] and subsequent transformation into imides[8] via domino rearrangements (Scheme 1c).

Our initial investigation focused on the reaction of cyclopropenone (**1a**) with nitrone (**2a**) as model substrates (Table 1). To our delight, using 10 mol % AgSbF$_6$ as a catalyst, a very interesting imide **3aa** was identified in dioxane, with no annulation product observed.[9] Encouraged by this result, a series of reaction parameters were next explored. Catalyst screening indicated that AgOTf was optimal, affording the desired product in 71% yield (entries 1−5). Control

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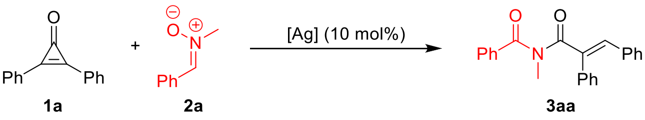
Table 1. Screening of Reaction Conditions[a]



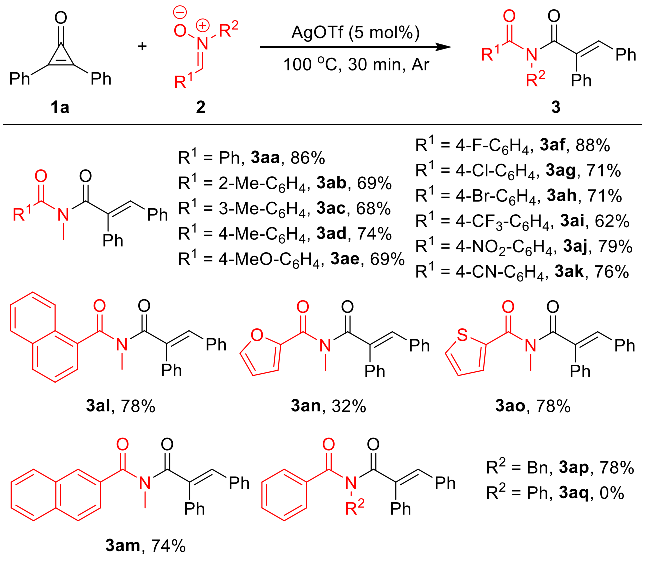
entry	catalyst	solvent	yield (%)[b]
1	$AgSbF_6$	dioxane	40
2	AgOTf	dioxane	71
3	$AgBF_4$	dioxane	65
4	AgOAc	dioxane	39
5	$AgNTf_2$	dioxane	66
6		dioxane	n.r.
7	AgOTf	THF	66
8	AgOTf	DME	75
9	AgOTf	toluene	67
10	AgOTf	DCE	64
11[c]	AgOTf	DME	87
12[c,d]	AgOTf	DME	87
13[e]	AgOTf	DME	86
14[c,f]	AgOTf	DME	69

[a]Conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), [Ag] (10 mol %) in 1.0 mL of solvent at 80 °C under Ar for 8 h. [b]Isolated yields. [c]100 °C, 30 min. [d]5 mol % of AgOTf was used. [e]120 °C. [f]Under air condition. n.r. = no reaction.

experiments indicated that silver salt was crucial for this transformation (entry 6). Different solvents were next tested; the reaction proceeded well in most of solvents, but DME was the best among all (entries 7−10). Furthermore, increasing the temperature to 100 °C and reducing the reaction time to 30 min showed a positive effect, improving the yield to 87% (entry 11).[10] Satisfyingly, reduction of the catalyst loading to 5% had no impact on the overall yield (entry 12).

With the optimized reaction conditions in hand, we examined the scope of nitrones for this reaction. As exemplified in Scheme 2, a wide range of nitrones can be engaged in this transformation, affording the desired products in good yields. Nitrones substituted at the *ortho-*, *meta-*, and *para*-position with methyl group proceed smoothly, showing that no obvious steric effect was observed (**3ab**−**3ad**). Both electron-rich and electron-deficient aryl substituents of the nitrones can be tolerated, giving the corresponding imides in yields of 62−88% (**3ae**−**3ak**). Naphthyl-substituted nitrones underwent the present reaction, and the rearrangement products **3al** and **3am** were isolated in 78% and 74% yields, respectively. This chemistry can be extended to heteroaromatic substituted nitrones (**3an** and **3ao**), thus highlighting the generality of the current methodology. Moreover, changing the methyl group on N atom to a benzyl group also proved the compatibility, while no reaction proceeded using *N*-phenyl nitrone (**3ap** and **3aq**).

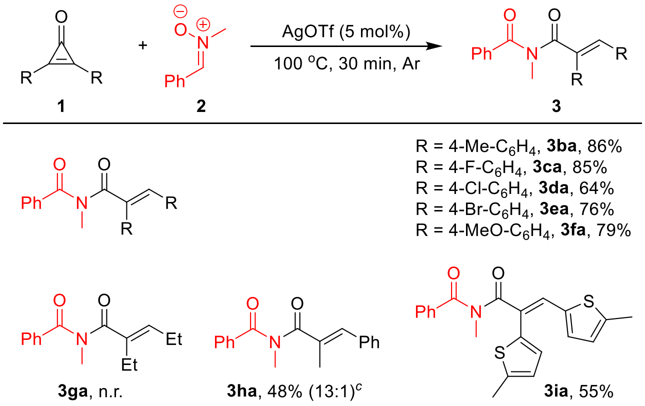
Scheme 2. Scope of Nitrones[ab]



[a]Conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), AgOTf (5 mol %) in 1.0 mL of solvent at 100 °C under Ar for 30 min. [b]Isolated yields.

The reaction scope with respect to cyclopropenones **2** was then examined (Scheme 3). Gratifyingly, substrates carrying methyl, fluoro, chloro, bromo, and methoxyl were all compatible, giving good to excellent yields (**3ba**−**3fa**). The employment of cyclopropenone with thienyl moiety generated the product **3ia** in moderate yield. However, when dialkyl-substituted cyclopropenone **1g** was exposed under the optimal reaction conditions, no product was detected.[11] Interestingly, nonsymmetrical cyclopropenone **1h** can be successfully reacted with nitrone, showing that the C(O)−C(Ph) bond was preferentially cleaved.

Scheme 3. Scope of Cyclopropenones[ab]

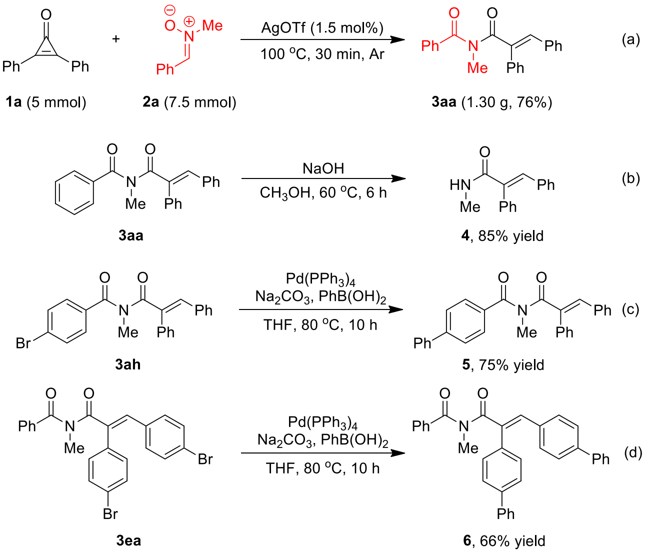


[a]Conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), AgOTf (5 mol %) in 1.0 mL of solvent at 100 °C under Ar for 30 min. [b]Isolated yields. [c]Ratio of 3ha to 3ha′.

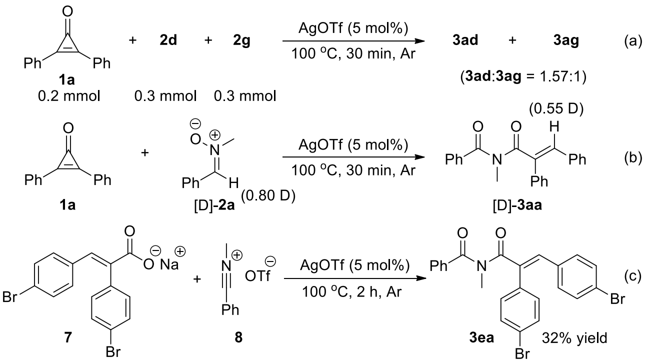
To further confirm the synthetic practicality and utility of this novel methodology, gram-scale synthesis of **3aa** was performed under the established reaction conditions (Scheme 4a). When 5.0 mmol scale of cyclopropenone **1a** was involved, the corresponding imide **3aa** was isolated in 76% yield with only 1.5 mol % loading of catalyst, which can be further transformed into (*E*)-*N*-methyl-2,3-diphenylacrylamide **4** under hydrolysis condition (Scheme 4b). Moreover, imide **3ah** and **3ea** could work well under Suzuki reaction conditions, and the coupling products **5** and **6** were obtained in satisfactory yields (Scheme 4c and d).

To gain more insights into the reaction mechanism, several experiments were designed and carried out (Scheme 5).[12] First, intermolecular competition experiments between **2d** and **2g** showed that electron-rich aryl nitrone was preferred. Next, when [D]-**2a** (80% deuterium) was tested in the reaction conditions, 55% deuterium incorporation at the position of

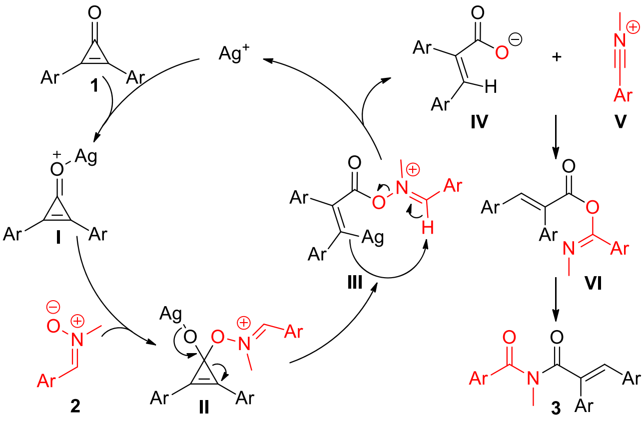
Scheme 4. Scale-Up of Reactions and Derivatization of Products



Scheme 5. Mechanistic Studies



Scheme 6. Possible Reaction Mechanism



terminal olefin of the imide was detected. The lower D content of [D]-**3aa** was attributed to trace water in the solvent. Moreover, we prepared sodium carboxylate **7** and nitrilium ion **8** to gain more information. Indeed, the reaction of **7** and **8** could afford **3ea** in DME at 100 °C, indicating this pathway may be involved in our established protocol.

On the basis of our previous work[6h] and the above experimental observations, a proposed reaction mechanism was outlined in Scheme 6. The reaction was initiated by the formation of intermediate **I** with assistance of Ag(I). The 1,2-addition of nitrone to intermediate **I** formed intermediate **II**, which converted to key intermediate **III** via β-carbon elimination promoted ring-opening process. Subsequently, intermediate **III** underwent sequential protonation and N−O bond cleavage to deliver carboxylate anion (**IV**) and nitrilium ion (**V**), accompanied by a silver salt. The nucleophilic addition of **IV** to **V** produced intermediate **VI**, and the consequent Mumm rearrangement[13] released the final imides **3**.

In summary, we have successfully developed the first Ag-catalyzed addition of cyclopropenones and nitrones for the synthesis of imides via C−C bond cleavage. In this strategy, the regioselectivity of nucleophilic addition, the trigger for subsequent transformation, and a series of rearrangements have been performed with excellent control. Furthermore, this protocol not only provides a new synthetic approach for various imides from diverse nitrones but also has broadened the application of cyclopropenones as 3C synthon. Further applications of this method and more mechanistic investigations are in progress.

■ ASSOCIATED CONTENT

SI Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c02099.

Experimental details and characterization data for all new compounds (PDF)

Accession Codes

CCDC 1966538 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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(10) A comparable yield (86%) can be achieved by increasing the temperature to 120 °C for 10 min. However, when various nitrones and cyclopropenones were exposed to these reaction conditions, some yields were not good enough.

(11) Most of the cyclopropenone **1g** was decomposed.

(12) (a) *N*-Methylbenzamide can be generated by some reactions of nitrone; however, it can not produce imide **3aa** with cyclopropenone under our established reaction conditions. (b) When N-methyl-1-phenylethan-1-imine was exposed under the reaction conditions, no reaction occurred.

(13) (a) Mumm, O. Umsetzung von Säureimidchloriden mit Salzen organischer Säuren und mit Cyankalium. *Ber. Dtsch. Chem. Ges.* **1910**, *43*, 886−893. (b) Mumm, O.; Hesse, H.; Volquartz, H. Zur Kenntnis der Diacylamide. *Ber. Dtsch. Chem. Ges.* **1915**, *48*, 379−391. (c) Curtin, D. Y.; Miller, L. L. 1,3-Acyl Migrations in Unsaturated Triad (Allyloid) Systems. Rearrangements of N-(2,4-Dinitrophenyl)-benzimidoyl Benzoates. *J. Am. Chem. Soc.* **1967**, *89*, 637−645. (d) Schwarz, J. S. P. Preparation of acyclic isoimides and their rearrangement rates to imides. *J. Org. Chem.* **1972**, *37*, 2906−2908. (e) Brady, K.; Hegarty, A. F. The isoimide−imide rearrangement. *J. Chem. Soc., Perkin Trans. 2* **1980**, *2*, 121−126. (f) Chen, J.; Shao, Y.; Ma, L.; Ma, M.; Wan, X. In situ generation of nitrilium from nitrile ylide and the subsequent Mumm rearrangement: copper-catalyzed synthesis of unsymmetrical diacylglycine esters. *Org. Biomol. Chem.* **2016**, *14*, 10723−10732.