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C−C Bond Acylation of Oxime Ethers via NHC Catalysis

Hai-Bin Yang* and Dan-Hong Wan

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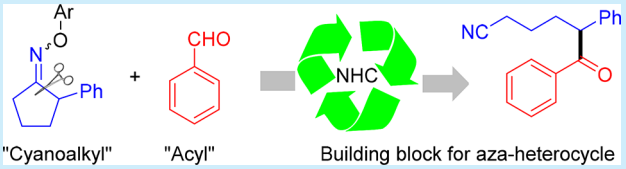
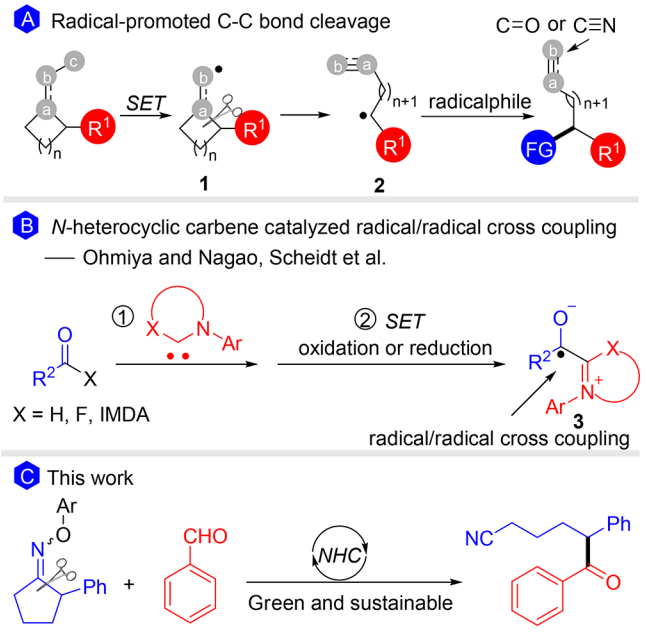
ABSTRACT: An *N*-heterocyclic carbene (NHC)-catalyzed strategy has been developed to address the issue of using toxic transitional metals in the field of C−C bond activation. The novel reaction mode enables an efficient docking between the cyanoalkyl from the cycloketone oxime derivative and the acyl group from the aldehyde, affording ketonitrile in moderate to good yields, which is one kind of useful building block for synthesizing nitrogen-containing pharmacophores.



In contrast with C−H bond functionalization as a method of modifying the periphery of molecules, C−C bond functionalization provides a powerful tool to modify their core framework. It endows the medicinal chemistry community with a new tactic for discovering lead compounds from known bioactive molecules via late-stage skeletal diversification, a concept referred to as "scaffold hopping".[1] This appealing chemistry is prevailingly achieved under the catalysis of a transitional metal (Rh, Ru, Pd) via a two-electron pathway,[2] but the radical-promoted C−C bond cleavage affords a unique and complementary strategy.[3] The essence of synthetic protocols through an open-shelled intermediate is harnessing the β-scission of a radical at the exocyclic α-position to afford an alkyl radical that is trapped by a radicalphile; meanwhile, the targeted functional group is introduced (Scheme 1A, **1** → **2**). However, toxic transitional-metal catalysts such as nickel are usually required to enable these radical process.[4] In this scenario, it is highly desirable to develop a new strategy to break the C−C bond in a green and sustainable manner.[5]

It is well known that *N*-heterocyclic carbene (NHC) catalysis can access umpolung reactivity of carbonyls as acyl anions, enolates, and homoenlates, which leads to the reaction with electrophiles through ionic pathways.[6] On the contrary, NHC catalysis that proceeds via single-electron transfer (SET) processes involving the coupling of radical intermediates is also known, although it is far less explored in organic synthesis.[7] As early as in 2008, a SET process between a Breslow intermediate and TEMPO was proposed in the oxidative esterification of aldehyde reported by Studer.[8] Rovis, Chi, Ye, and Sun developed diverse NHC-catalyzed β-, γ-, or ε-functionalizations of enals via radical/radical coupling[9] or radical addition[10] mechanisms thereafter. Although great progress has been made, these reactions mainly realized the carbon−carbon and carbon−heteratom bond formation at the remote position of the carbonyl group through an open-shelled radical intermediate. Very recently, Nagao and Ohmiya

Scheme 1. Strategies of the Radical-Promoted C−C Bond Activation



demonstrated a novel C−CO bond construction strategy via a persistent ketyl-type radical **3** that resulted from the oxidation of the Breslow intermediate (Scheme 1 B).[11] Scheidt disclosed that the reduction of the acyl azolium intermediate could also provide an analogous ketyl-type radical, which was

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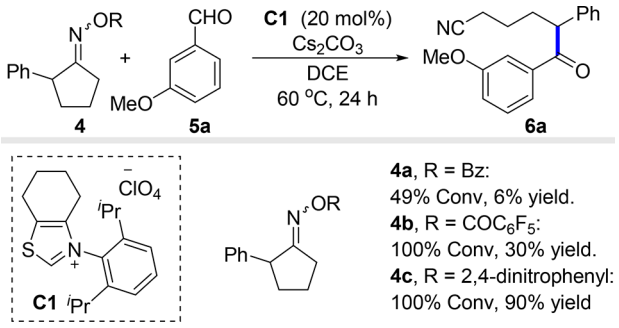
applied to the ketone synthesis.[12] Following their works, diverse coupling reactions between carbonyl compounds and radical precursors have been developed for synthesizing functionalized ketones.[13] Inspired by these excellent precedents involving the ketyl-type radical **3**, we considered that the goal of cleaving an inert C−C bond in a green and sustainable manner would be attained via NHC catalysis. Moreover, the cyclic oxime derivative is an excellent platform for C−C bond activation reactions.[14] We envisaged that a ring-opening coupling between a cyclic oxime derivative and an aldehyde could occur under the catalysis of NHC, affording ketonitrile, which is one kind of useful building block for constructing nitrogen-containing pharmacophores such as piperidine, pyridine, pyridin-2(1*H*)-one, and 2-aminothiophene (Scheme 1C).[15]

We initiated our investigation by examining the reaction of 2-phenylcyclopentan-1-one oxime derivative **4** and 3-methoxybenzaldehyde **5a** in the presence of 20 mol % thiazolium **C1**. Preliminary condition optimization showed that the reaction was very sensitive to the leaving group of 2-phenylcyclopentan-1-one oxime derivative **4** (Scheme 2). When *O*-benzoyl oxime

Scheme 2. Evaluation of the Effects of the Leaving Group



4a (R = Bz) was employed as a substrate, the corresponding product **6a** was obtained in 6% yield with 51% of starting material **4a** remaining, which indicated that the reactivity of **4a** was not strong enough for this thiazolium-based carbene catalytic system. It is foreseeable that decreasing the electron density of the aryl motif in the leaving group can increase the reductive potential of the substrate; then, the reactivity will be greatly improved. Next, we synthesized *O*-perfluorobenzoyl oxime **4b**, and performing the reaction with **4b** provided **6a** in 30% yield. However, the reaction mass balance was not ideal, and we speculated that oxime ester was unstable under basic conditions. Therefore, we considered using the more stable oxime ether **4c** as the substrate, where the 2,4-dinitrophenol motif is also a good redox-active leaving group.[16] Nevertheless, a SET process between the nitro group and a Breslow intermediate might occur to produce a persistent N−O radical, which might interrupt the designed reaction.[9] To our delight, the reaction proceeded smoothly to provide **6a** in 90% NMR yield. After a further screening of other parameters such as thiazolium **C**, temperature, solvent, and base, we established the optimal conditions using 10 mol % of carbene precursor **C1** and 1.0 equiv of Cs_2CO_3 as a base in DCE at 60 °C to provide product **6a** in 82% isolated yield. (For details, see the Supporting Information.)

With the optimal condition in hand, we investigated the scope of this organocatalyzed ring-opening cross-coupling between the oxime derivative and the aryl aldehyde (Scheme 3). At first, a number of aromatic aldehydes were examined. Benzaldehyde, which bears a substituent such as chlorine at the meta position, was an effective coupling partner, affording the corresponding product **6b** in 80% yield. When unsubstituted benzaldehyde was employed as the substrate, ketonitrile **6c** was obtained in 74% yield. Benzaldehydes, which bear substituents at the para position, including acetyl and cyano, were tolerated, and the reaction provided compounds **6d** and **6e** in 62 and 53% yields. Treating oxime ether **4c** with fused aromatic aldehydes such as 2-naphthaldehyde and 2-quinolinecarboxaldehyde, the corresponding products **6f** and **6g** were, respectively, obtained in 80 and 69% yields. An electron-deficient heteroaryl aldehyde such as nicotinaldehyde is a better coupling partner than an electron-rich heteroaryl aldehyde such as 2-furfural, and the corresponding products **6h** and **6i** were, respectively, delivered in 88 and 43% yields. However, the current reaction condition does not work for other types of aldehydes such as aliphatic and *α*,*β*-unsaturated aldehydes. Next, we focused on investigating the scope of oxime ethers **4**. The reaction proceeded smoothly to provide ketonitriles **6j**−**n** in 50−82% yields for various cyclopentanone oxime ethers **4** bearing a scope of para-, meta-, and ortho-substituted phenyl rings at the endocyclic *α*-position. Meanwhile, both electron-donating groups, such as methyl and methoxy, and electron-withdrawing groups, such as fluoro and ester, were tolerated in this mild catalytic system. Remarkably, the cyclopentanone oxime ethers bearing other types of aromatic substituents such as 2-naphthyl and benzo[*b*]-thiophen-5-yl were also compatible with this catalytic system, affording compounds **6o** and **6p** in 87 and 51% yields. Furthermore, cyclobutanone oxime ethers were more reactive substrates, and the coupling products **6q** and **6r** after the ring-opening process were obtained in 82 and 65% yields at room temperature. Although the oxidative radical cyanomethylation of allylic alcohol provides an effective strategy for synthesizing 1, 5-ketonitrile derivatives, this protocol will not produce a regioisomer due to a mechanistic discrepancy ($Ar^3 \neq Ar^2$).[17] Compared with the four-membered ring and five-membered ring counterparts, this organocatalyzed ring-opening acylation became less favorable for cyclohexanone-derived oxime ether, affording compound **6s** in 54% yield at 80 °C (**6c** and **6q** vs **6s**). An attempt to construct quaternary stereogenic center through the ring-opening benzoylation of 2-methyl-2-phenylcyclopentan-1-one-derived oxime ether was unsuccessful, and the corresponding product could not be detected via ^{1}H NMR analysis.

To demonstrate the synthetic potential of the current methodology, a formal synthesis of CWJ-a-5, which is a potent topoisomerase I inhibitor, was conducted (Scheme 4).[18] Treating oxime derivative **4d** with benzaldehyde under standard conditions, the reaction proceeded smoothly to afford compound **6t** in 70% yield, which could be converted to CWJ-a-5 according to the reference method.[19] Considering the ease availability of substituted aromatic aldehydes, this route is convenient for synthesizing CWJ-a-5 analogues.

A possible mechanism for this organocatalyzed ring-opening acylation of cyclic oxime derivatives is depicted in Scheme 5.[20] The catalytic cycle begins with the addition of the NHC to the benzaldehyde in the presence of cesium carbonate. The resulting Breslow intermediate **8** (E^{red} = −0.7 to −1.0 V vs SCE)[21] undergoes a SET process with cyclic oxime ether **4c** (acetophenone-derived analogue, E^{red} = −0.55 V vs SCE)[16a] to form the key intermediates **10** and **9**. Unlike the other N-

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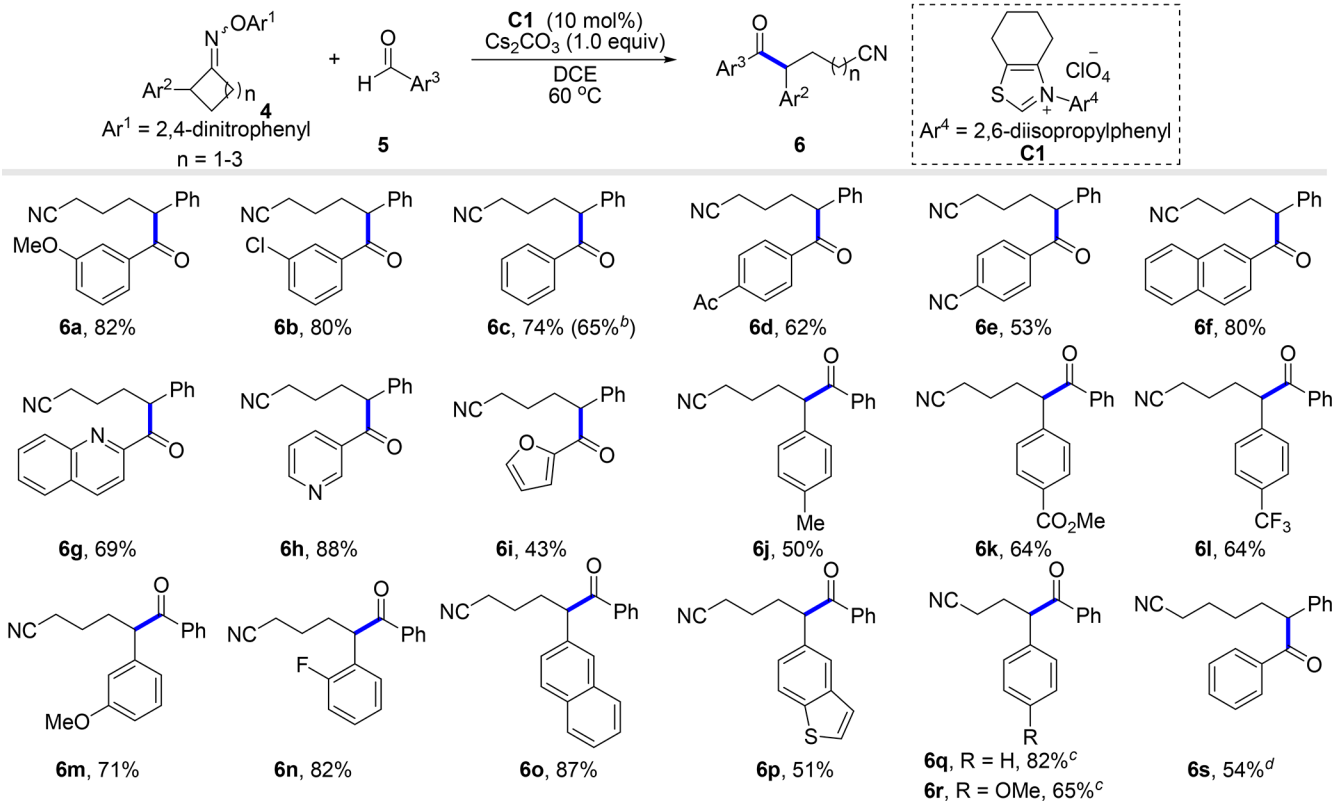
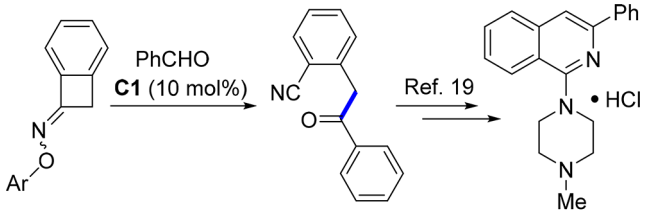
Scheme 3. NHC-Catalyzed C−C Bond Activation: Scope of Ketonitrile Products[a]

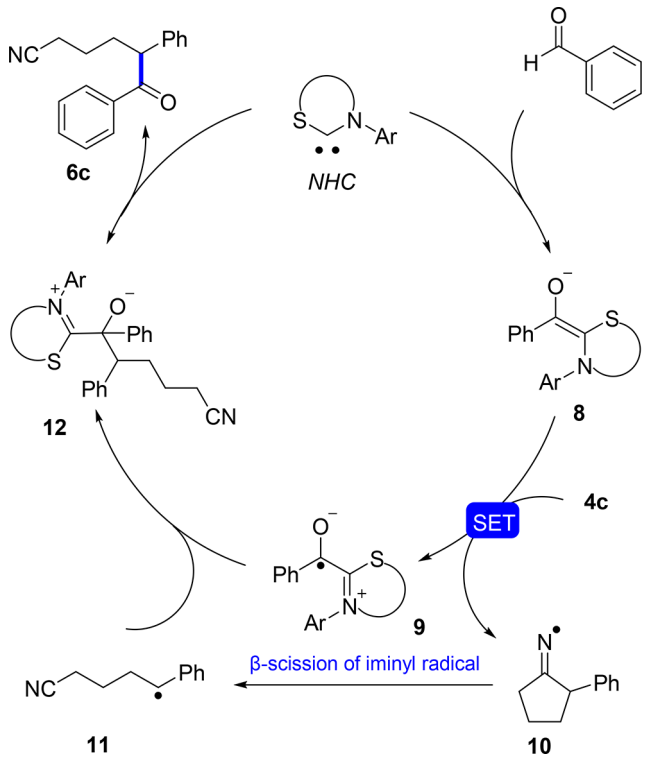


[a]Reaction was performed with carbene catalyst precursor **C1** (10 mol %), aryl aldehyde **5** (0.3 mmol, 1.5 equiv), cyclic oxime ether **4** (0.2 mmol, 1.0 equiv), and cesium carbonate (1.0 equiv) in DCE at 60 °C. All yields are isolated yields. [b]Isolated yield on a 1.0 mmol scale. [c]Carried out at room temperature. [d]Performed with 20 mol % of **C1** at 80 °C.

Scheme 4. Formal Synthesis of CWJ-a-5



Scheme 5. Reaction Mechanism for NHC-Catalyzed C−C Bond Cleavage



centered radicals, iminyl radical **10** is a σ-type species with its unpaired electron in the orbital on the N atom in the nodal plane of the C=N π-system.[22] The hyperconjugation effect between the unpaired-electron-occupied orbit on the N atom and the σ orbital of the α-C−C bond would lead to a rapid radical-type ring-opening process, affording the nucleophilic alkyl radical **11**. The cross-coupling between the transient radical **11** and the persistent radical **9** gives reaction species **12**, which undergoes an intramolecular fragmentation to yield product **6c** and regenerate carbene catalyst.

To further understand the reactivity of the iminyl radical in NHC catalysis, we explored the reaction outcome for oxime ether, where α-C−C bond cleavage is an unfavorable process (Scheme 6). Using benzophenone-derived oxime ether **4e** as a substrate, 32% of N-coupled product **13** was detected in the presence of 20 mol % **C1**.[23] The *N*-acylation product **13** is possibly formed as follows: (1) radical/radical cross-coupling between iminyl radical **14** and captodative radical **9** and (2)

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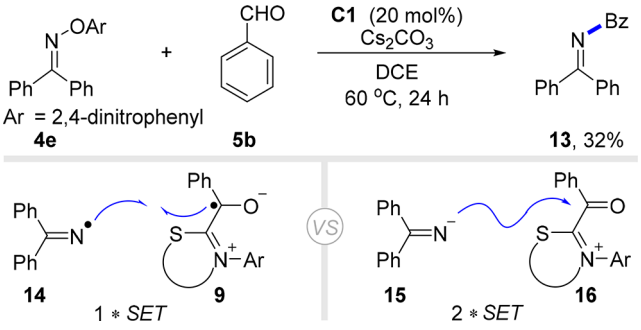
Scheme 6. Acyl-Transfer Mechanism of Noncycloketone-Derived Oxime Ether



nucleophilic substitution of iminyl anion **15** to acylthiazolium **16**.[8] Captodative radical **9** is also a strong reductant (E^{red} = −0.5 V vs SCE). In addition, the N-centered radical prefers to accept an electron because the electronegativity of the N atom is higher than that of the C atom.[24] The SET process between iminyl radical **14** and captodative radical **9** should be fast, yielding iminyl anion **15** and acylthiazolium **16**. So, we are inclined to think that the latter one is more possible.

In conclusion, we have introduced a strategy for NHC-catalyzed C−C bond functionalization. Under the guidance of this strategy, a radical-type cross-coupling between cyclic oxime ether and an aromatic aldehyde has been developed. This protocol demonstrated several good synthetic features including environmentally friendliness, easily available starting materials, and good yields. The development of an enantioselective version of this C−C bond acylation is underway in our lab.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c04248.

Experimental procedures, characterization data, and ^{1}H and ^{13}C NMR spectra of all new compounds (PDF)

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Notes

The authors declare no competing financial interest.

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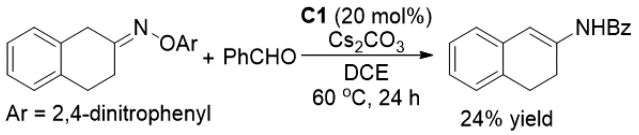

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