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Review

Recent advances in *N*-heterocyclic carbene-catalyzed radical reactions

Lei Dai[a,b], Song Ye[a,b,*]

[a] *Beijing National Laboratory for Molecular Sciences, CAS Key Laboratory of Molecular Recognition and Function, CAS Research/Education Center for Excellence in Molecular Sciences, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, China*
[b] *University of Chinese Academy of Sciences, Beijing 100049, China*

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ABSTRACT

The *N*-heterocyclic carbene (NHC)-catalyzed reactions involving two-electron reaction pathway have been well-established. However, the development of NHC-catalyzed radical reactions is still in its infancy in terms of reaction types and enantioselectivity. In the past decade, several elegant NHC-catalyzed radical reactions have been developed, including NHC-catalyzed oxidation of aldehydes to esters, reductive coupling reactions using Breslow intermediate as SET reductant and NHC-catalyzed reactions *via* radical homoenolates, dienoaltes and trienolates. This review summarizes the recent advances in NHC-catalyzed reactions involving radical intermediates.

1. Introduction

Over the past few decades, N-heterocyclic carbenes (NHCs) have witnessed great success in the field of organocatalysis [1–10]. Since the NHC-catalyzed umpolung of aldehyde *via* Breslow intermediate [11], NHC-bound intermediates have been extensively studied, which can be divided mainly into two types, the electron-rich and electron-deficient intermediates (Fig. 1) [5]. These various intermediates offer many possibilities for carbene catalysis *via* two-electron reaction pathway.

Compared to the well-established NHC-catalyzed two-electron reaction pathway, the NHC-catalyzed reactions *via* single-electron transfer (SET) are far less developed [12–17]. Although the NHC-catalyzed SET reaction has been elucidated in biochemistry for the decarboxylation of pyruvate [18], NHC-catalyzed SET radical pathways remain challenging and much less developed in synthetic chemistry. In recent years, several reports have demonstrated that NHC-catalyzed radical reactions are feasible. This review aims to give a brief summary on this issue.

2. NHC-catalyzed oxidation of aldehydes to esters

In nature, thiamine diphosphate (ThDP), a NHC precursor, works as coenzyme to catalyze the oxidative decarboxylation of pyruvate *via* pyruvate ferredoxin oxidoreductase [19–22]. This process has been extensively studied, which involves mainly two steps, the decarboxylation *via* two electron transfer and two SET oxidation steps (Scheme 1). In the late 1990s, the one-electron-oxidation potential of the anion of Breslow intermediate was determined ($E^{o}_{ox} = -0.77 \sim -0.98$ V) [23–26].

Inspired by oxidative decarboxylation of pyruvate in biological system, Studer and co-workers reported the pioneering work of the oxidation of aldehydes to esters *via* two SET oxidation steps of Breslow intermediate using the (2,2,6,6-tetramethylpiperidin-1-yl)oxy radical (TEMPO) as the oxidant (Scheme 2) [27].

After this pioneering work, there are many reports using SET oxidative strategy to convert aldehydes or enals into various products, such as esters and cycloaddition products [28–35].

3. Breslow intermediate as SET reductant

In 2014, Chi and co-workers reported an elegant *β*,*β*-coupling of nitroalkenes *via* Breslow intermediate as SET reductant (Scheme 3) [36]. The coupling reaction is initiated by one electron oxidation of the Breslow intermediate **17** by nitroalkene **13** to form radical **19** which was characterized by electron paramagnetic resonance (EPR) spectroscopy. The radical **19** is coupled with nitroalkene **13** to form anionic radical intermediate **20** which then undergoes subsequent oxidation and protonation to form *β*,*β*-homo-coupling

* Corresponding author at: Beijing National Laboratory for Molecular Sciences, CAS Key Laboratory of Molecular Recognition and Function, CAS Research/Education Center for Excellence in Molecular Sciences, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, China.
E-mail address: songye@iccas.ac.cn (S. Ye).

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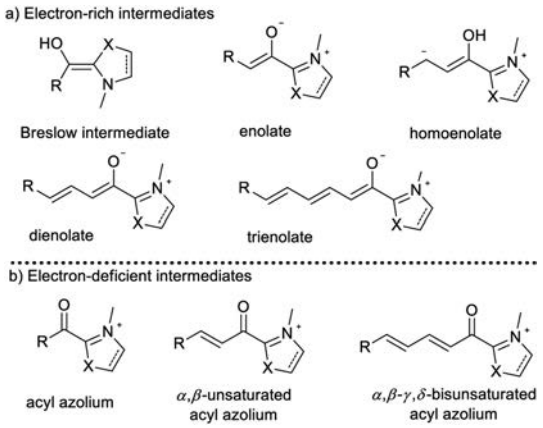
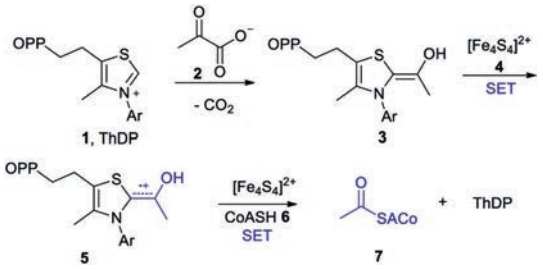
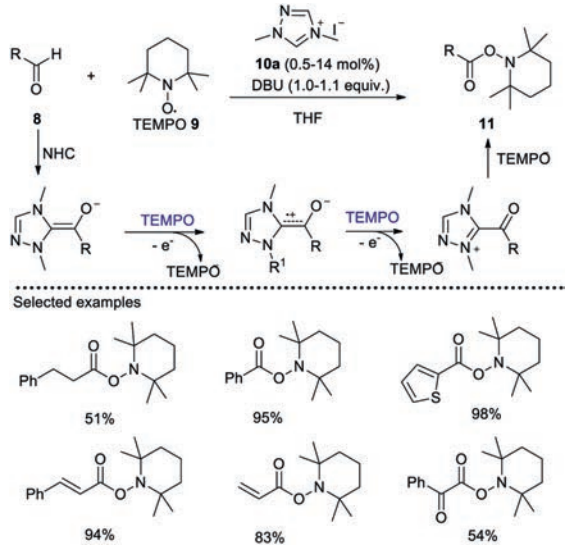


Fig. 1. Intermediates in NHC catalysis *via* two-electron pathway.



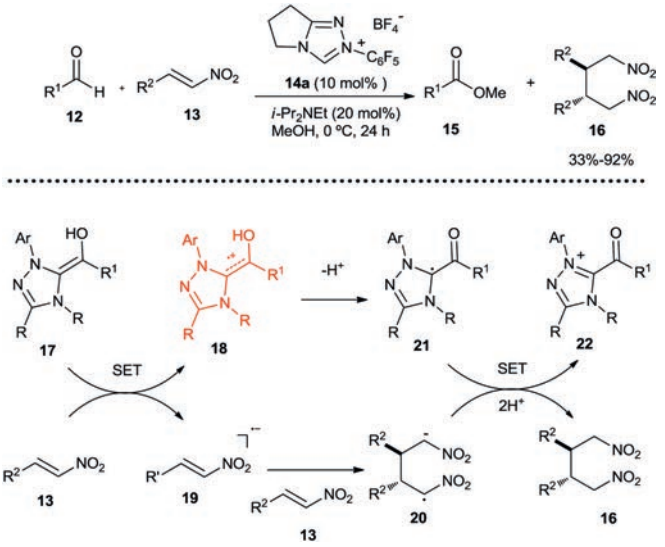
Scheme 1. Oxidative decarboxylation of pyruvate *via* thiamine diphosphate (ThDP).



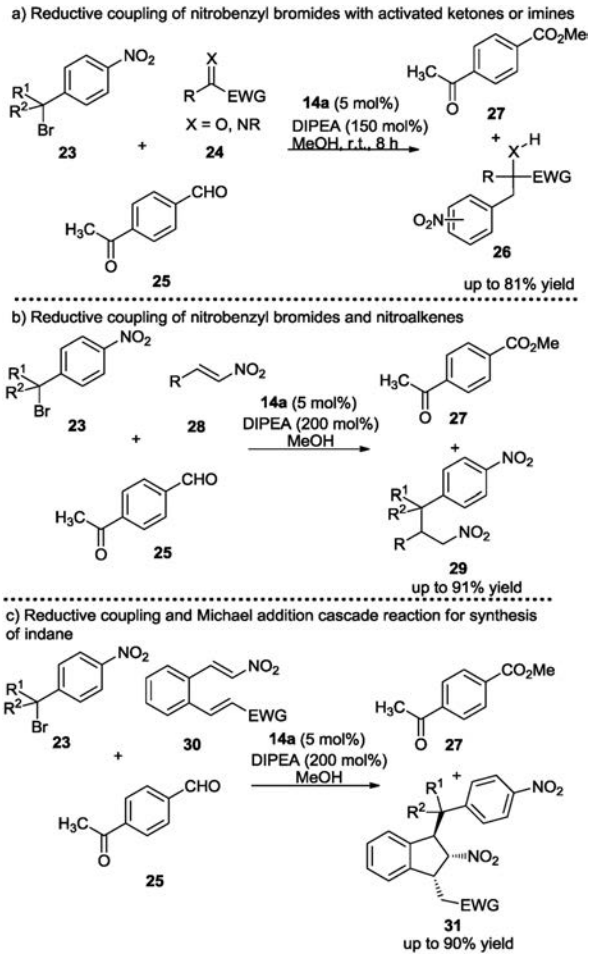
Scheme 2. Biomimetic carbene-catalyzed oxidations of aldehydes to esters.

of nitroalkenes **16**. At the same time, the Breslow intermediate undergoes two SET oxidation steps to form aycl azolium **22** which is trapped by methanol to form ester **15**.

Subsequently, using this strategy, the same group further developed some interesting reductive coupling and cascade reactions (Scheme 4). For example, the reductive coupling of nitrobenzyl bromides **23** with activated ketones or imines **24** was established (Scheme 4a) [37]. They realized NHC-catalyzed reductive 1,4-addition of nitrobenzyl bromides **23** to nitroalkenes **28** with good yields (Scheme 4b) [38]. An elegant NHC-catalyzed



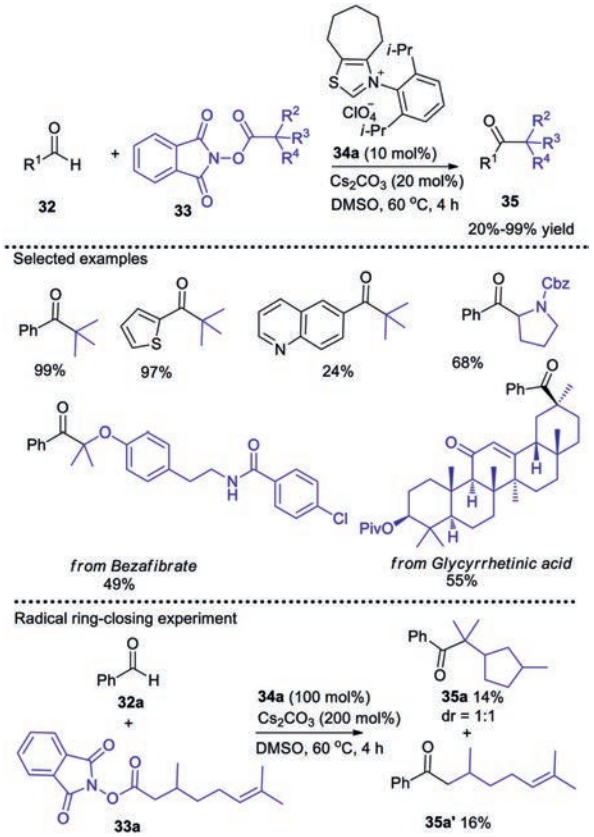
Scheme 3. β,β-Coupling of nitroalkenes.



Scheme 4. The reductive coupling reactions with Breslow intermediate as SET reductant.

cascade reaction for the synthesis of multi-substituted indane derivatives **31** was also developed (Scheme 4c) [39].

It is interesting that only electron deficient NHC-catalysts bearing *N*-C_6F_5 group were effective for the reaction, which may



Scheme 5. NHC-catalyzed decarboxylative alkylation.

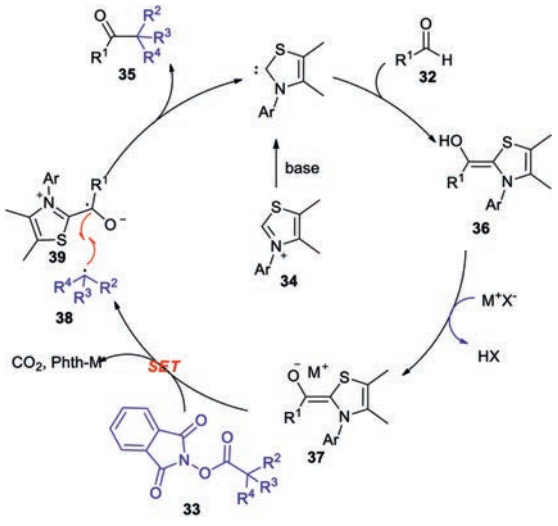
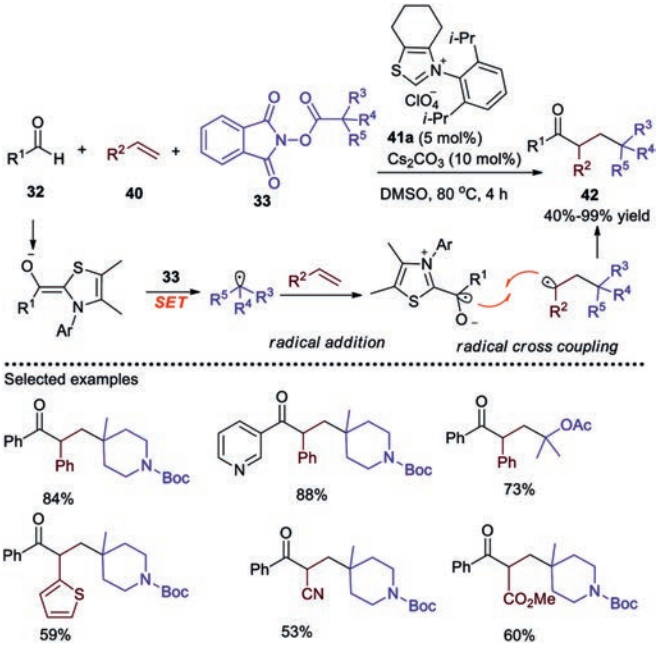


Fig. 2. Possible mechanism for decarboxylative alkylation.

owe to their suitable oxidation potential of the Breslow intermediates.

Very recently, Ohmiya and Nagao *et al.* reported the NHC-catalyzed decarboxylative alkylation of aryl aldehydes for construction of $C(sp^2)$-$C(sp^3)$ bond *via* cross of coupling Breslow intermediate type radicals with tertiary or secondary alkyl radicals (Scheme 5) [40]. This methodology allowed efficient synthesis of functionalized ketones including late stage modification of pharmaceutical drugs and natural products. The reaction of **32a** with citronellic acid-derived **33a** obtained radical cyclization



Scheme 6. NHC-catalyzed alkylacylation of alkenes *via* radical relay.

product **35a** in 14% yield. This radical ring-closing experiment strongly suggested that a carbon-centred radical was involved in this reaction.

Based on a series of mechanistic experiments, they proposed the reaction mechanism as depicted in Fig. 2. The reaction is initiated by formation of the Breslow intermediate **36**, and then deprotonation of **36** by a base generates the intermediate **37**. Subsequently, the SET between **37** and **33** gives alkyl radical **38** and Breslow intermediate type radical **39**, respectively. Eventually, the radical–radical recombination gives the desired ketones **35** and regenerates the carbene catalyst.

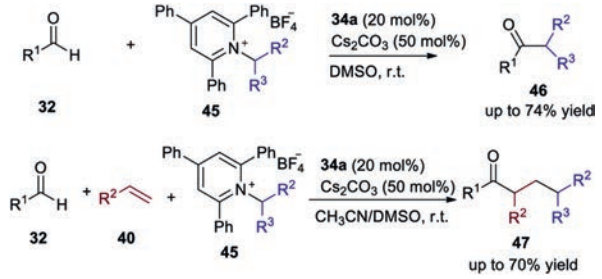
Subsequently, the same group reported an interesting carbene-catalyzed vicinal alkylacylation of alkenes *via* radical relay strategy (Scheme 6) [41]. They demonstrated that NHC-catalyzed radical relay strategy was a powerful method for vicinal alkylacylation of styrenes, acrylates and acrylonitriles.

Recently, this strategy was successfully applied for the NHC-catalyzed acylfluoroalkylatoin of alkenes by Li [42], Wang [43] and Wu *et al.* [44] independently (Scheme 7).

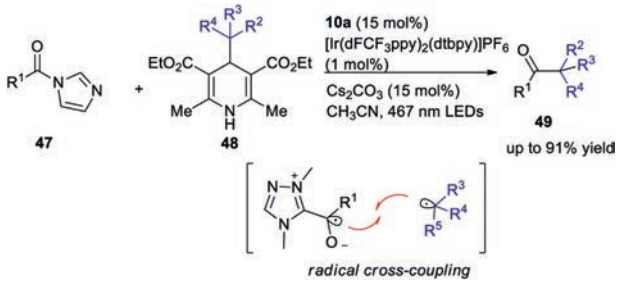
The Katritzky pyridinium salts was used successfully as the alkyl sources for the NHC-catalyzed alkylation of aldehyde and alkylacylation of alkene by Hong *et al.* (Scheme 8) [45].

The synergistic merger of NHC catalysis and photoredox catalysis for benzylation of acyl azolium was reported by Scheidt *et al.* (Scheme 9) [46]. Oxidation of the Hantzsch ester **48** by excited-photocatalyst (Ir^{III*}) gives benzyl radical, and SET reduction of the acyl triazolium provides the Breslow-derived ketyl radical by Ir^{II} photocatalyst. Eventually, the radical–radical cross coupling affords the desired ketones **49** and regenerates the carbene catalyst.

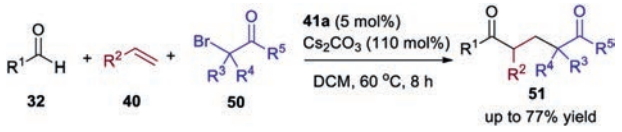
Scheme 7. NHC-catalyzed acylfuoroalkylation of alkenes *via* radical relay.



Scheme 8. NHC-catalyzed deaminative cross-coupling of aldehydes with Katritzky pyridinium salts.



Scheme 9. Photo/NHC-cocatalyzed synthesis of ketones.



Scheme 10. NHC-catalyzed synthesis of δ-ketocarbonyls *via* radical relay.

Very Recently, Ohmiya and Nagao *et al.* reported NHC-catalyzed synthesis of δ-ketocarbonyls from enals, alkenes and tertiary α-bromocarbonyls (Scheme 10) [47].

Notably, *N*-2,6-diisopropylphenyl thiazolium salts were used as the catalysts for most of these reactions, which implies the sterically hindered and electron-rich property are crucial for the efficient NHC catalysts.

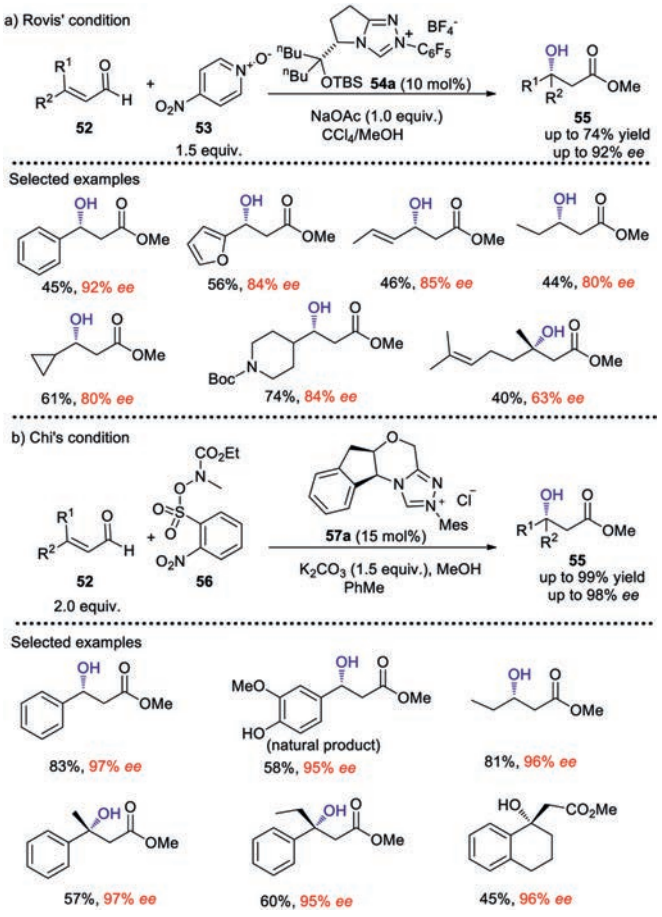
4. Reactions *via* radical from homoenolate

In recent years, several reports have demonstrated that NHC-catalyzed reactions involving homoenolate radical as key bond formation are feasible, including the formation of C(sp^3)-O and C (sp^3)-C(sp^3) bonds.

4.1. β-Hydroxylation of enals

Recently, Rovis group [48] (Scheme 11a) and Chi group [49] (Scheme 11b) more or less simultaneously independently reported enantioselective β-hydroxylation of enals *via* nitrobenzene as SET oxidant. These two interesting reports are considered as significant breakthrough, because the homoenolate type radical was achieved for the first time *via* the oxidation of NHC-bound homoenolate intermediate. The SET oxidant is crucial for this process, nitropyridine N-oxide **53** and nitrobenzenesulfonic carbamate **56** were used, respectively.

The possible mechanism for β-hydroxylation of enals is depicted in Fig. 3. This reaction starts from formation of the homoenolate intermediate **58**, which undergoes oxidation with nitrobenzene to



Scheme 11. Enantioselective β-hydroxylation of enals.

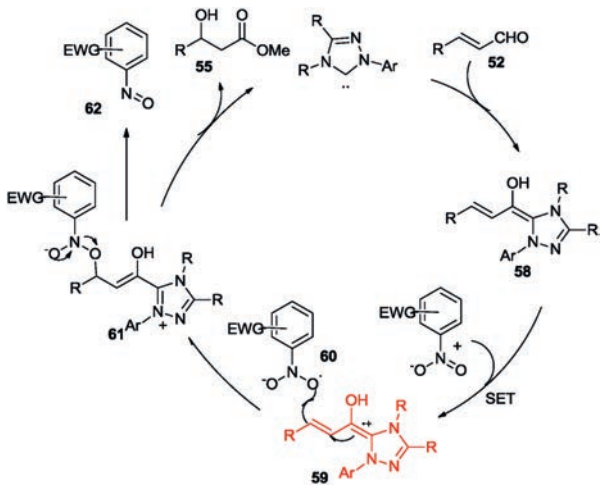
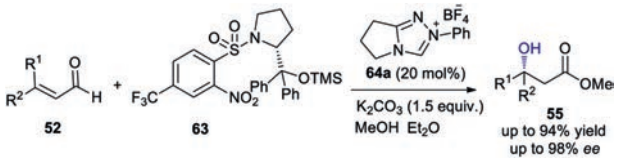


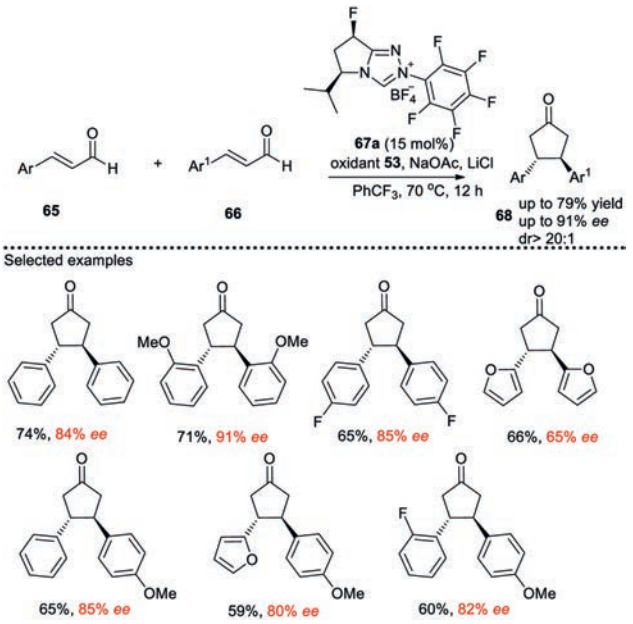
Fig. 3. Possible mechanism for β-hydroxylation of enals.

furnish homoenolate type radical **59** and nitrobenzene-derived radical anion **60**. Subsequently, the cross coupling of radicals **59** and **60** is the key step for bond formation to form **61** which collapses to expel an NHC-bound alkoxide which is trapped by methanol to give β-hydroxylation ester **55** and nitrosobenzene **62**.

Very recently, Chi and co-workers reported an alternative method for enantioselective β-hydroxylation of enals which combined chiral nitrobenzene and achiral carbene (Scheme 12) [50].



Scheme 12. Chiral nitrobenzene mediated enantioselective β-hydroxylation of enals.



Scheme 13. NHC-catalyzed enantioselective homo- and cross-coupling of the two homoenolates.

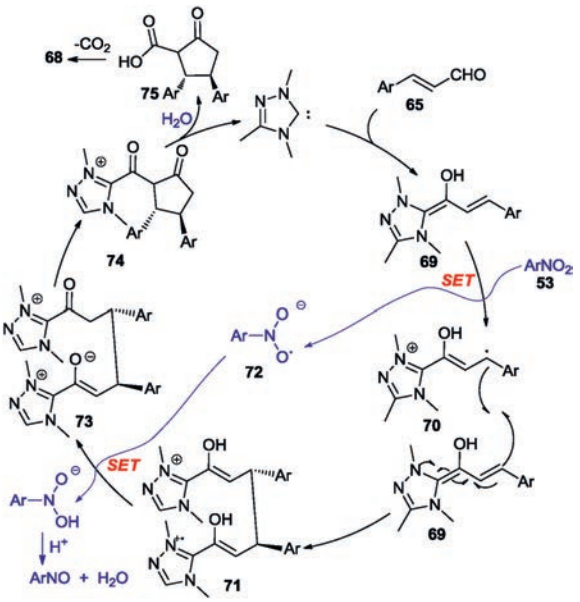
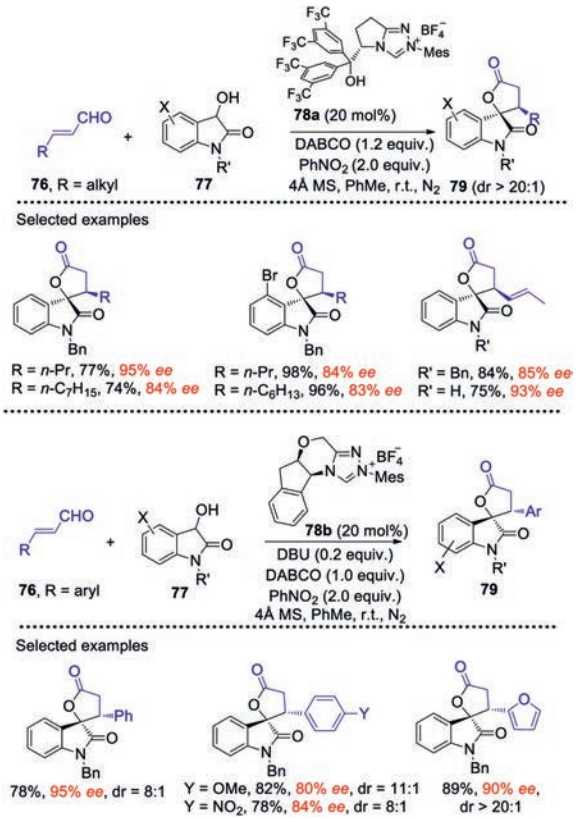


Fig. 4. Possible mechanism for enantioselective homo- and cross-coupling of the two homoenolates.

4.2. Homo- and heterocoupling of enals

Rovis and co-workers reported the pioneering work of enantioselective homo- and cross-coupling of two homoenolates for construction of C(sp^3)-C(sp^3) bond in 2015 (Scheme 13) [51]. In their previous work, they developed enantioselective α-hydroxylation of enals using nitrobenzene as SET oxidant [48]. During the investigations, they observed cyclopentanone products when the reaction was run in a non-nucleophilic solvent. After a series of condition screening, they found $PhCF_3$ to be the best solvent, giving 3,4-disubstituted cyclopentanones with good yields and enantioselectivity.

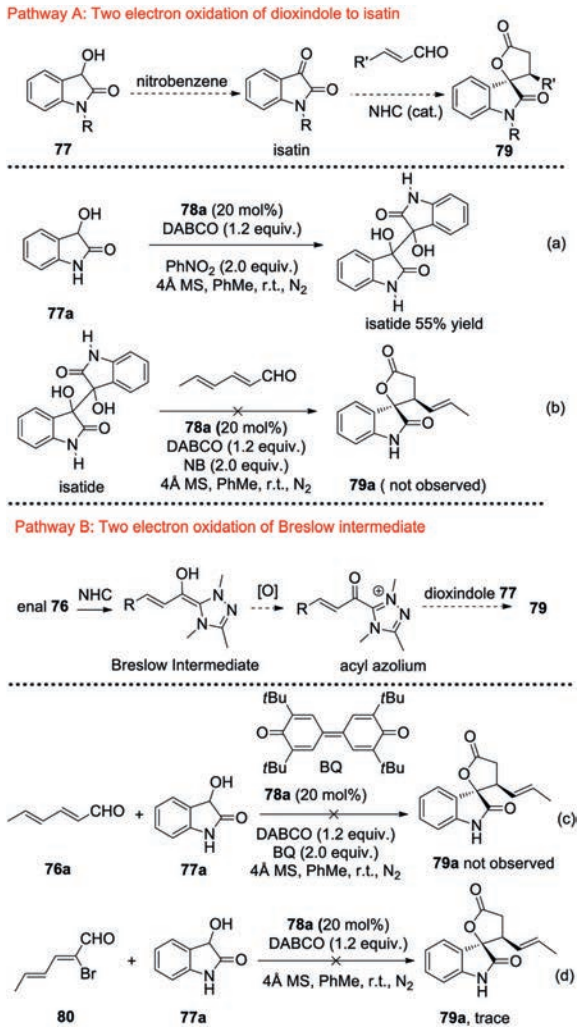
The possible mechanism for this reaction is depicted in Fig. 4. The reaction starts from formation of the homoenolate intermediate **69**, which undergoes SET oxidation with nitrobenzene to give homoenolate radical **70**. Subsequently, the cross coupling of homoenolate radical **70** and homoenolate **69** is the key step for bond formation to form **71** which undergoes a second SET oxidation to form acyl azolium **73**. The acyl azolium **73** is attacked by the enolate to form cyclopentanone **74**. Then the cyclopentanone **74** is trapped by water to give β-ketoacid **75**. The final product **68** is obtained from decarboxylation of β-ketoacid **75**.



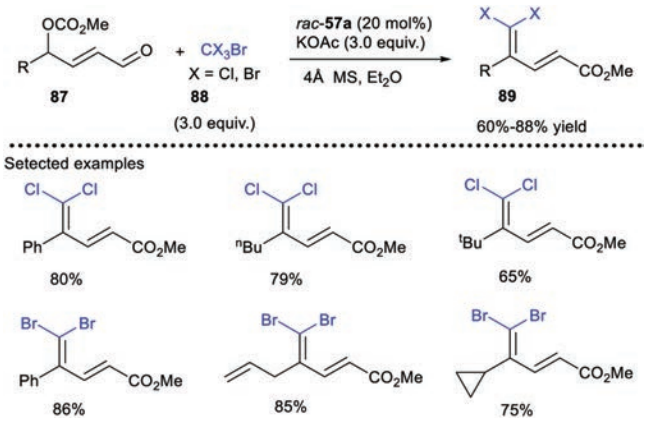
Scheme 14. NHC-catalyzed enantioselective cross coupling of homoenolate and enolate.

4.3. Radical coupling of homoenolate and enolate

Recently, our group reported the first example of NHC-catalyzed enantioselective cross coupling of homoenolate and enolate (Scheme 14) [52,53]. The NHC-catalyzed oxidative [3 + 2] annulation of dioxindole and enals gave the spirocyclic oxindole-γ-lactones in good yields with high to excellent diastereo- and enantioselectivities. It is worth noting that the challenging aliphatic enals also worked effectively in this reaction. A series of mechanistic investigations were conducted to clarify the possible mechanisms (Scheme 15). Pathway A is the oxidation of dioxindole to isatin followed by annulation with enals and pathway B is the two-electron oxidation of Breslow intermediate to acyl azolium followed by annulation with dioxindole. These two pathways were all ruled out by the control experiment. Next, we



Scheme 15. Control experiments of enantioselective cross coupling of homoenolate and enolate.



Scheme 16. NHC-catalyzed γ-dihalomethylenation of enals.

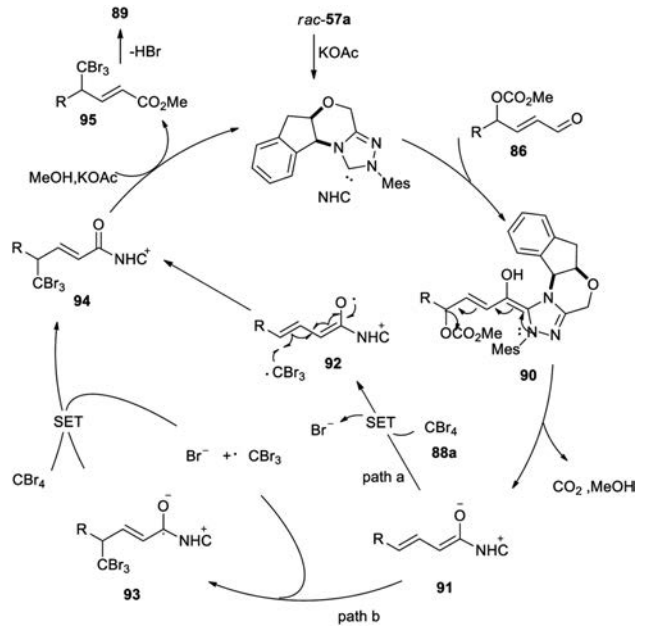


Fig. 6. Possible mechanism for γ-dihalomethylenation of enals.

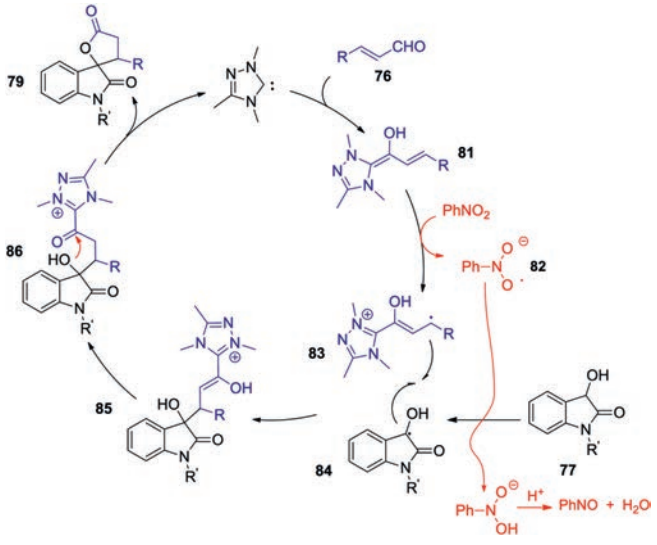


Fig. 5. Plausible catalytic cycle.

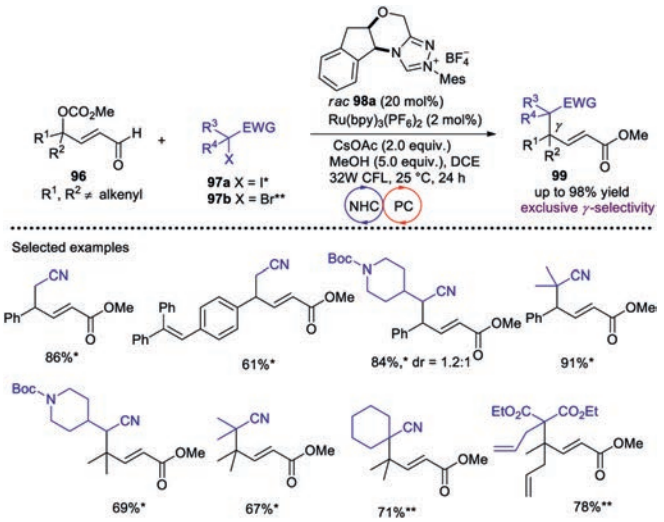
also conducted some TEMPO-trapping and EPR experiments to further clarify this reaction mechanism. We suggested that the cross coupling of homoenolate radical and enolate radical was the key step for this reaction.

The plausible catalytic cycle for this reaction is depicted in Fig. 5. The reaction starts from formation of the homoenolate intermediate **81**, which undergoes nitrobenzene oxidation to give homoenolate radical **83** and nitrobenzene-derived radical anion **82**. Subsequently, the radical **82** could undergo hydrogen atom transfer from dioxindole **77** to obtain its enolate radical **84**. The cross-coupling of the homoenolate radical **83** and the enolate radical **84** gives adduct **85**, which is protonated to furnish acyl azolium **86**. The lactonization of acyl azolium **86** gives the spirocyclic oxindole-γ-lactones **79** and regenerates the carbene catalyst.

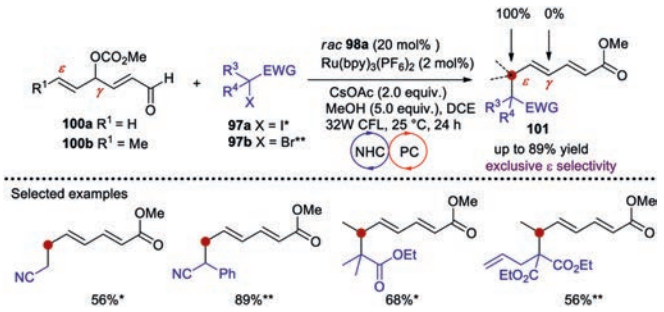
5. Reactions *via* radicals from dienolate and trienolate

5.1. γ-Dihalomethylenation of enals

Sun and co-workers developed an elegant NHC-catalyzed γ-dihalomethylenation of enals *via* radicals from dienolaes with



Scheme 17. γ-Alkylation of enals with simple alkyl radicals *via* photo/NHC cocatalysis.



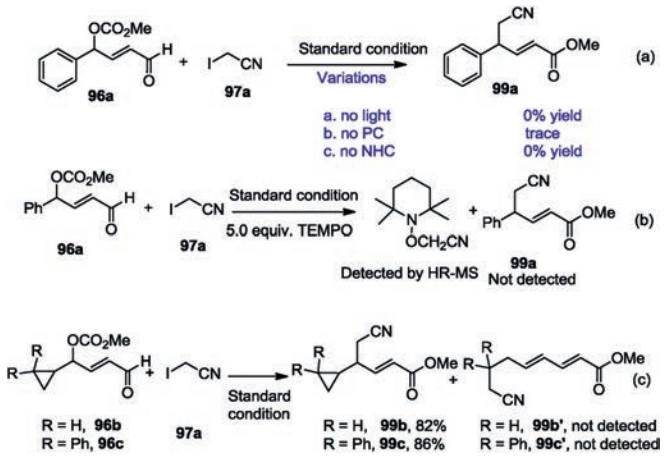
Scheme 18. ε-Alkylation with radicals.

CCl_3Br and CBr_4 as SET oxidant (Scheme 16) [54]. Notably, one example of NHC/photo cocatalyzed γ-dichlomethylenation of enals gave the product in 42% yield using CCl_4 as terminal SET oxidant. However, further efforts to improve the yield proved fruitless. Luckily, they found CCl_3Br and CBr_4, which have more oxidation capacity, were efficient SET oxidants for this transformation without photocatalyst and light.

The possible mechanism for γ-dihalomethylenation of enals is depicted in Fig. 6. The reaction begins with formation of the Breslow intermediate **90**, which undergoes elimination of the leaving group to form dienolate intermediate **91**. Subsequently, **91** may experience two possible pathways. In path a, it may undergo SET oxidation to deliver radical cation **92** and tribromomethyl radical. Thereafter, radical–radical recombination gives the α,β-unsaturated aycl azolium **94**. Finally, **94** is trapped by methanol with regeneration of the carbene catalyst and delivery of compound **95**, which leads to the final product **89** by HBr elimination. In path b, dienolate **91** could be attacked by electrophilic tribromomethyl radical to obtain zwitterion radical **93**, which then experiences SET oxidation by CBr_4 giving the intermediate **94**. At present, they could not rule out either pathway.

5.2. γ- And ε-alkylation of γ-preoxidized enals

NHC-catalyzed radical reactions with specific oxygen or functionalized carbon-centered radicals prompted us to develop more general NHC-catalyzed γ-alkylation with alkyl radicals. The visible-light-mediate photoredox catalysis is a very powerful



Scheme 19. Mechanism studies of photo/NHC-cocatalyzed remote alkylation.

strategy to generate radicals [55]. Very recently, our group successfully realized the merging of photoredox catalysis with NHC catalysis for efficient construction of $C(sp^3)$-$C(sp^3)$ bond with alkyl radicals (Schemes 17 and 18) [56]. The reaction of enals with alkyl halides gave the γ-multisubstituted-α,β-unsaturated esters with exclusive γ-selectivity, including those with challenging vicinal all-carbon quaternary centers (Scheme 17). NHC-catalyzed remote functionalization is still a challenge. In this work we reported the first example of NHC-catalyzed ε-functionalization [57,58] *via* our rational design which was a series of enals bearing vinyl substituent at the γ position that can generate trienolate intermediate *via* NHC catalysis (Scheme 18).

Several mechanism studies were carried out (Scheme 19). The reaction without light, PC, or carbene gave no or trace desired product (Scheme 19a). The reaction was totally inhibited when TEMPO was added and radical coupling product between cyanomethyl radical and TEMPO was detected by HR-MS (Scheme 19b). The reaction of enal **96b** and **96c**, bearing a cyclopropyl group at the γ position, with iodoacetonitrile gave the desired products **99b** and **99c** in excellent yield without the ring-opening product **99b′** and **99c′** (Scheme 19c), which suggests the reaction pathway of alkyl radical addition to dienolate intermediate.

The proposed mechanism is depicted in Fig. 7. The alkyl radical **102** generated from alkyl halide **97** *via* photoredox catalysis, was added to the dienolate intermediate **103**, generated from enals **96** *via* carbene catalysis, to give homoenolate radical **104**. The following SET oxidation of homoenolate radical **104** by the radical cation **105** of photocatalyst gives α,β-unsaturated acyl azolium

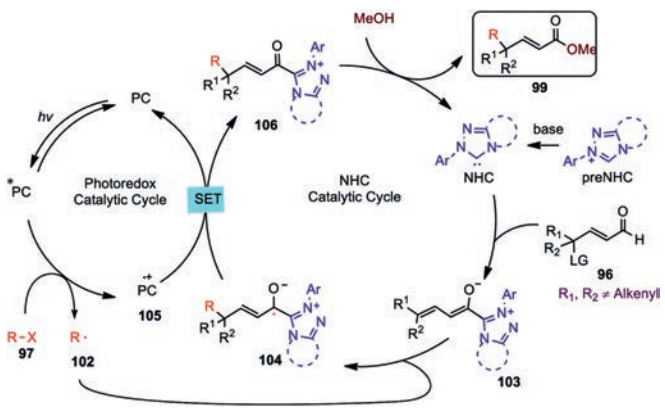
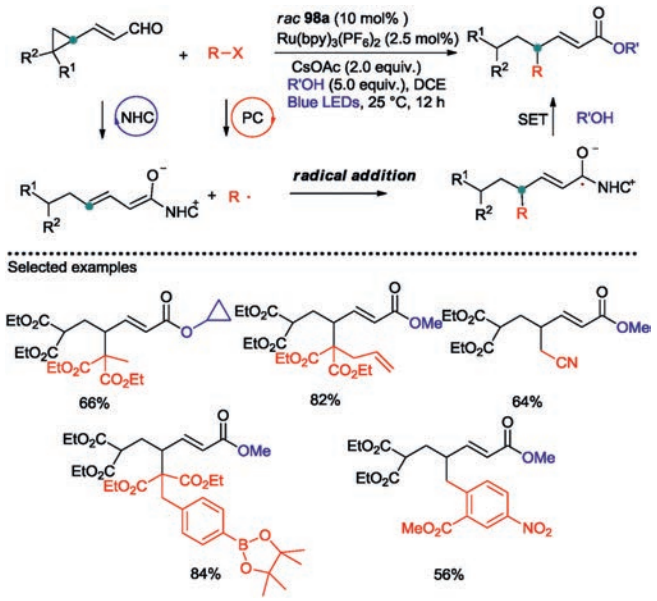


Fig. 7. Proposed mechanism.



Scheme 20. Ring-opening and γ-alkylation of cyclopropane enal.

intermediate **106**. The acyl azolium intermediate **106** is attacked by methanol to afford the final products **99** and regenerates the NHC catalyst.

5.3. Ring-opening and γ-alkylation of cyclopropane enal

Very recently, our group successfully realized photo/NHC-cocatalyzed ring-opening C—C bond cleavage of cyclopropane enal and subsequent γ-alkylation with alkyl electrophiles, giving the corresponding γ-alkylated α,β-unsaturated esters in good yields (Scheme 20) [59].

6. Conclusions

Several elegant transformations *via* NHC-catalyzed radical reactions have been reported in the past decade. However, the development of this area is still in its infancy in terms of reaction types and enantioselective radical reactions. Considering the invaluable potential of this area, many more NHC-catalyzed radical reactions may be expected in the future. Especially, NHC-catalyzed enantioselective radical reactions and the merging of photoredox catalysis with NHC catalysis will be very valuable and arouse our attention.

Declaration of competing interest

The authors report no declarations of interest.

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