Chinese Chemical Letters 37 (2026) 111484

Contents lists available at ScienceDirect

Chinese Chemical Letters

journal homepage: www.elsevier.com/locate/cclet

γ-C(sp^3)-H acylation of aliphatic amines enabled by cooperative photoredox NHC/Pd catalysis

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ARTICLE INFO

Article history:
Received 14 January 2025
Revised 12 June 2025
Accepted 17 June 2025
Available online 18 June 2025

Keywords:
N-Heterocyclic carbene
Palladium
Cooperative catalysis
C(sp^3)-H acylation
Aliphatic amine

ABSTRACT

The site-selective C(sp^3)-H functionalization is of great importance in synthetic chemistry. However, γ-amino C(sp^3)-H functionalization of aliphatic amines remains challenging. Herein, we develop an efficient γ-C(sp^3)-H acylation of aliphatic amines by cooperative photoredox NHC/Pd catalysis. The process entails the following key steps: (i) photoinduced palladium-promoted formation of aryl radical, (ii) generation of transient γ-amino alkyl radical through aryl radical-mediated 1,7-HAT, (iii) single-electron oxidation of Breslow enolate intermediate to persistent ketyl radical, and (iv) radical/radical coupling of γ-amino alkyl radical with ketyl radical. The synthetic utility of this γ-amino C(sp^3)-H acylation is illustrated by the conversion of readily available aliphatic amines to a diverse collection of γ-aminoketones, which serve as versatile building blocks to enable the synthesis of pyrrolines of interest in medicinal chemistry. The radical mechanism is supported by the results of various control experiments, *in situ* EPR analysis, radical trapping experiment, and isotopic labeling studies.

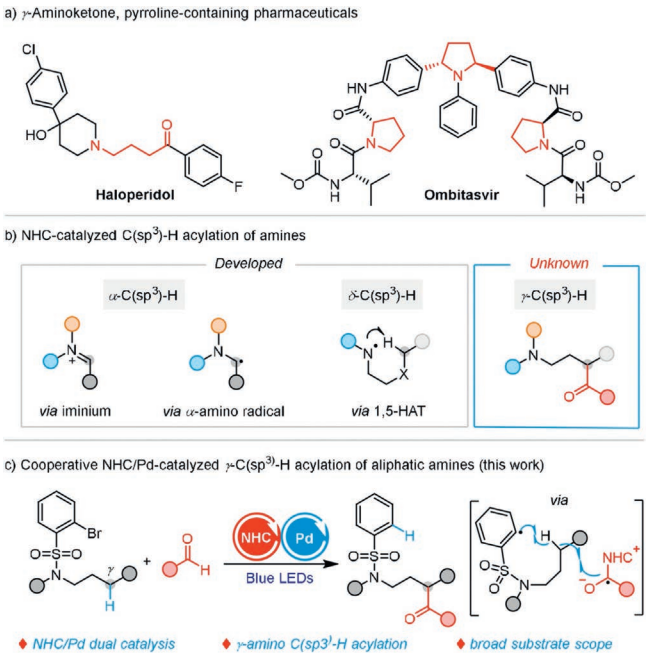
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An updated analysis of U.S. FDA-approved small-molecule drugs in the past 11 years (2013–2023) reveals that >82 % of drugs contain at least a nitrogen heterocycle or an amine moiety [1]. In this context, the continuous development of new methods for the precise synthesis of amine derivatives has emerged as a crucial research field in organic synthesis and medicinal chemistry. Among them, selective C(sp^3)-H functionalization of readily available aliphatic amines could streamline the synthesis of nitrogen-containing heterocycles and complex amine architectures. For example, different strategies for the α-C(sp^3)-H functionalization of aliphatic amines have been well developed *via* oxidative process [2–7] or hydrogen atom transfer (HAT) [8–13]. In addition, δ-C(sp^3)-H functionalization based on amidyl radical-mediated 1,5-HAT has been proven to be a robust strategy to access remote functionalized amines and the related nitrogen-containing heterocycles [14–17]. In contrast, γ-amino C(sp^3)-H functionalization of aliphatic amines remains challenging. Current successful γ-amino C–H functionalization methods mainly rely on directing group-assisted transition metal catalysis [18–27]. Alternatively, radical C(sp^3)-H functionalization of amines *via* γ-selective HAT process has enabled a new approach to the synthesis of amine derivatives [28–33]. However, γ-C(sp^3)-H acylation of aliphatic amines remains unaccessible, while the products will simplify the synthesis of γ-aminoketones, δ-amino alcohols, and pyrrolidines of interest in medicinal chemistry (Scheme 1a) [34–37].

In the past decades, *N*-heterocyclic carbene (NHC) catalysis has emerged as a powerful strategy for the synthesis of structurally diverse molecules [38–49]. In 2012, Rovis and co-workers developed cooperative NHC and photocatalysis for asymmetric acylation of α-C(sp^3)-H bond in *N*-aryl tetrahydroisoquinoline *via* nucleophilic addition of Breslow intermediate to the oxidatively generated iminium (Scheme 1b) [50]. Driven by the development of radical NHC catalysis [51–58], the direct acylation of C(sp^3)-H bonds has aroused great interest [59]. Limited successful examples mainly focus on α- or δ-C(sp^3)-H acylation of amines. For example, the NHC-catalyzed α-C(sp^3)-H acylation of amines has been established through a cascade generation of α-amino radical *via* HAT process and the following coupling with ketyl radical [60–63]. On the other hand, the remote δ-C(sp^3)-H acylation has been achieved *via* amidyl radical-mediated 1,5-HAT [64]. However, γ-amino C(sp^3)-H acylation is highly desirable but still unexplored to date.

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https://doi.org/10.1016/j.cclet.2025.111484
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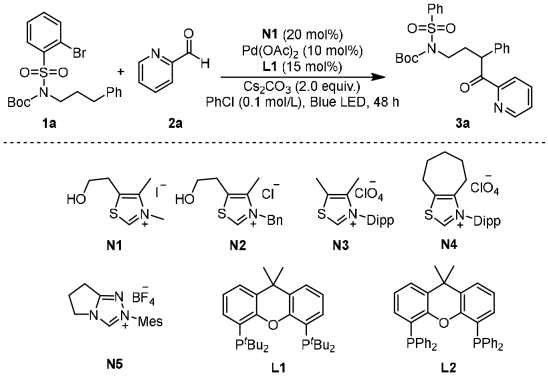
Scheme 1. NHC-catalyzed C(sp^3)-H acylation of amines.

Herein, we report the γ-selective C(sp^3)-H acylation of aliphatic amines with aldehydes as acyl source under cooperative NHC and photoinduced palladium catalysis (Scheme 1c). 2-Bromophenyl sulfone is used as γ-selective HAT auxiliary and amino-protecting group [28,65]. The photoinduced Pd-promoted generation of aryl radical and the subsequent 1,7-HAT is the key to the success of this methodology. This approach enables direct conversion of a wide array of readily available aliphatic amines to γ-aminoketones in moderate to good yields with excellent and predictable regioselectivity. The easy conversion of the products to δ-aminoalcohols and pyrrolidines of interest to medicinal chemists further demonstrates the potential application of the developed method in drug discovery campaigns.

Initially, the model reaction of 2-bromophenyl sulfonamide **1a** and commercially available picolinaldehyde **2a** was investigated under photoredox cooperative NHC/Pd catalysis (Table 1). We were pleased to find that the reaction gave the desired product **3a** in 62% yield when carried out in the presence of 20 mol% thioazolium preNHC **N1**, 10 mol% of $Pd(OAc)_2$ with ^{t}Bu-Xantphos **L1** (15 mol%) as ligand and Cs_2CO_3 (2.0 equiv.) as base in PhCl under blue LED irradiation at room temperature (entry 1). The screening of preNHC catalysts revealed that *N*-benzyl and *N*-aryl substituted thioazolium preNHC **N2**-**N4** resulted in some loss of yields (entries 2 and 3), while triazolium preNHC **N5** did not work for this reaction (entry 4). Other palladium catalysts such as $PdCl_2$ and $Pd(PPh_3)_4$ were less effective (entries 5 and 6). The use of ligand Xantphos resulted in decreased yield (entry 7), and PCy_3 was ineffective (entry 8). Solvent screening revealed that the reaction in $PhCF_3$, THF or DCM did not give better yields (entries 9 and 10). Evaluation of bases showed that other inorganic bases, such as K_2CO_3 and CsF, and organic base led to decreased yields (entries 11 and 12). The improvement of the yield was achieved by reducing reaction concentration to 0.05 mol/L (entry 13). The reaction with other photocatalysts, such as 4CzIPN, *fac*-Ir(ppy)$_3$, Ru(phen)$_3$(PF_6)$_2$, gave the desired product in <7% yield (entries 14–16).

With the optimized reaction conditions in hand, the scope of aldehydes was evaluated (Scheme 2). It was found that picolinaldehydes bearing electron-donating or electron-withdrawing groups such as OMe, Me, F, and Cl at various positions of pyridine ring

Table 1
Optimization of the reaction conditions.[a]

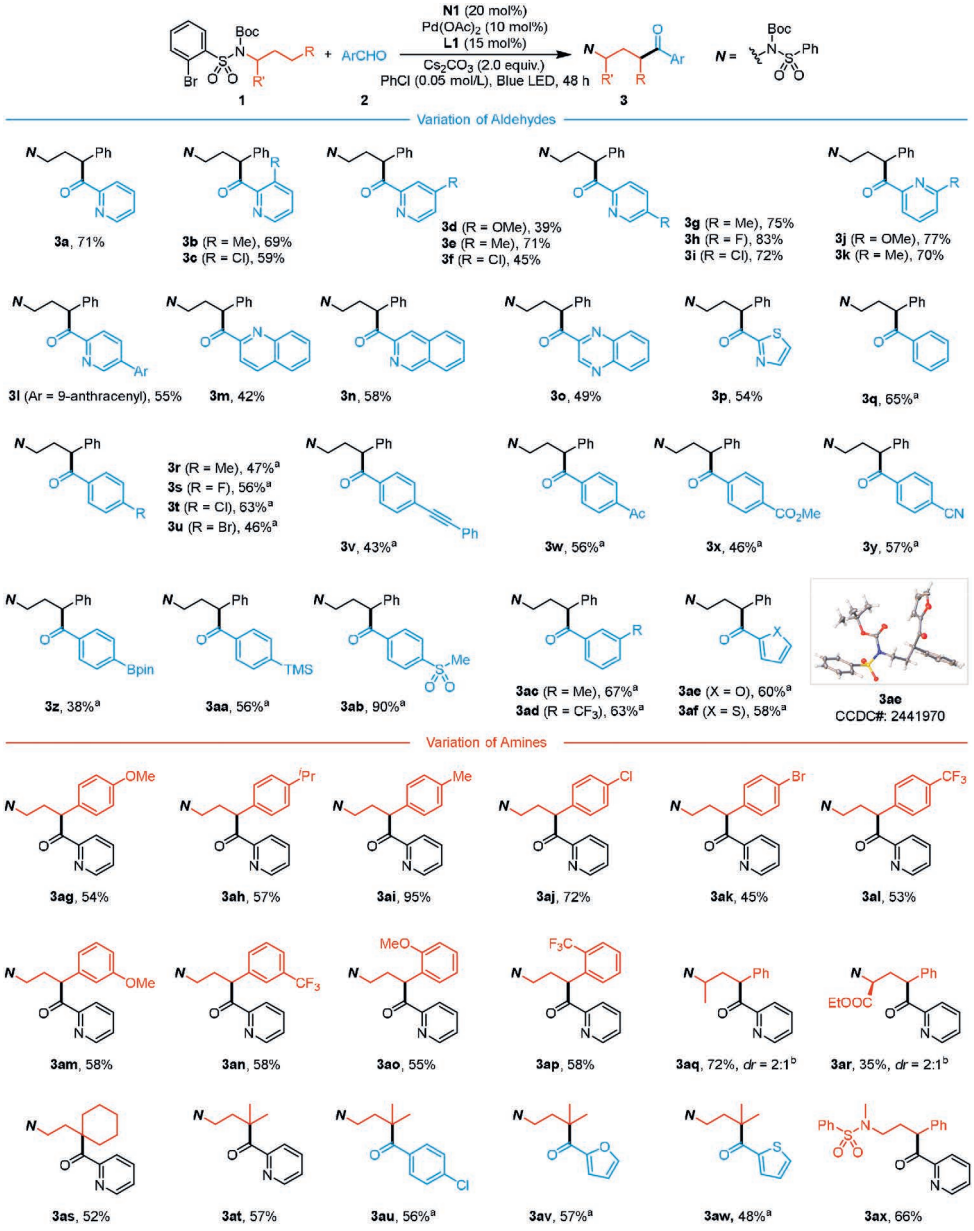


Entry	Variation from standard conditions	Yield (%)[b]
1	None	62
2	**N2**, **N3** instead of **N1**	48, 52
3	**N4** instead of **N1**	57
4	**N5** instead of **N1**	Trace
5	$Pd(PPh_4)_3$ instead of $Pd(OAc)_2$	35
6	$PdCl_2$ instead of $Pd(OAc)_2$	ND
7	**L2** instead of **L1**	16
8	PCy_3 instead of **L1**	ND
9	$PhCF_3$ instead of PhCl	51
10	THF, DCM instead of PhCl	Trace
11	K_2CO_3, CsF instead of Cs_2CO_3	54, 53
12	DBU instead of Cs_2CO_3	22
13	0.05 mol/L instead of 0.1 mol/L	74 (71)[c]
14	4CzIPN instead of $Pd(OAc)_2$/**L1**	Trace
15	*fac*-Ir(ppy)$_3$ instead of $Pd(OAc)_2$/**L1**	7
16	Ru(phen)$_3$(PF_6)$_2$ instead of $Pd(OAc)_2$/**L1**	Trace

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), preNHC (0.04 mmol), $Pd(OAc)_2$ (0.02 mmol), ^{t}Bu-Xantphos (0.03 mmol), Cs_2CO_3 (0.4 mmol) and PhCl (2 mL), 36 W blue LED at room temperature for 48 h under N_2.
[b] Yields of **3a** determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as internal standard. ND = not detected.
[c] Isolated yield in parenthesis.

all worked well to afford desired ketones **3b**–**3k** in moderate to good yields. Picolinaldehyde bearing 9-anthracenyl at 5-position was compatible for this reaction giving the desired product **3l** in 55% yield. The reactions of other heteroaromatic aldehydes such as quinoline-2-carbaldehyde, isoquinoline-3-carbaldehyde, quinoxaline-2-carbaldehyde and thiazole-2-carbaldehyde all went smoothly to furnish the desired ketones **3m**–**3p**. However, the reaction of benzaldehyde gave no desired product which promoted us reoptimized the conditions of the reaction (see Supporting information for details). It was found that the use of preNHC **N4** afforded the desired ketone **3q** in 65% yield. The scope of benzaldehydes was then examined. Benzaldehydes bearing electron-donating (Me) or electron-withdrawing groups (F, Cl, and Br) on the *para*-position all worked well, leading to ketones **3r**–**3u** in moderate yields. It is worth noting that functional groups, such as internal alkyne, acetyl, carbomethoxy, cyano, pinacol boronic ester (Bpin), trimethylsilyl (TMS) and methylsulfonyl, were all tolerable to afford the corresponding ketones **3v**–**3ab**. *meta*-Substituted benzaldehyde bearing Me or CF_3 groups gave the products **3ac**–**3ad** in good yields. Besides, the reaction of furfural and 2-thenaldehyde also proceeded smoothly to give ketones **3ae**–**3af** in good yields. The structure of the product **3ae** was confirmed by single crystal X-ray diffraction analysis. However, cinnamaldehyde and aliphatic aldehydes, such as pivaldehyde and benzenepropanal, did not work for this reaction under current reaction conditions.

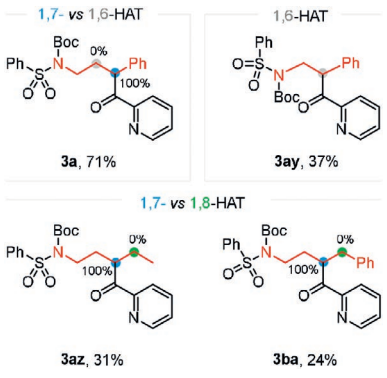
Then, various amines were examined. 3-Phenylpropylamines bearing OMe, ^{i}Pr, Me, Cl, Br, and CF_3 on the *para*-position afforded



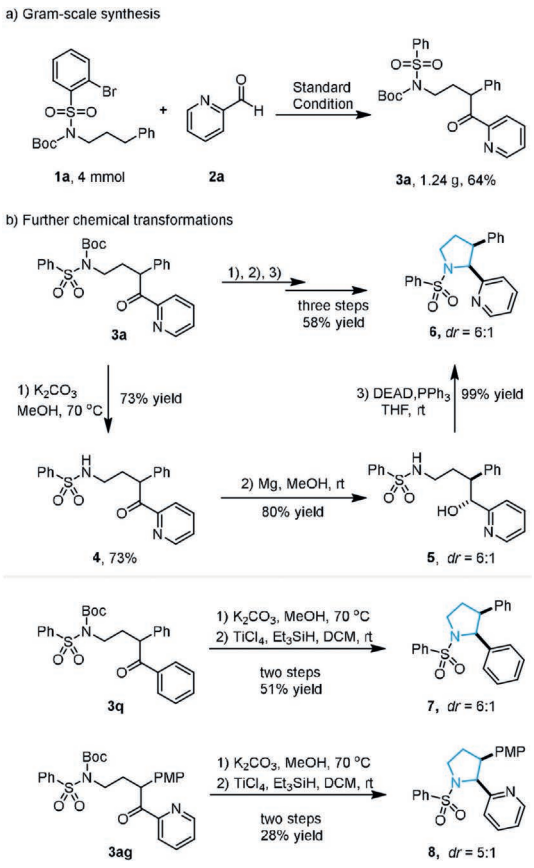
Scheme 2. NHC/Pd-catalyzed γ-C(sp^3)-H acylation of amines. Reaction conditions: **1** (0.2 mmol), **2** (0.3 mmol), preNHC **N1** (0.04 mmol), $Pd(OAc)_2$ (0.02 mmol), **L1** (0.03 mmol), Cs_2CO_3 (0.4 mmol) and PhCl (4 mL), 36 W blue LED at room temperature for 48 h under N_2. Isolated yields. [a] preNHC **N4** was used. [b] Diastereomeric ratio was determined by ^1H NMR.

corresponding ketones **3ag**-**3al** in moderate to good yields. The reaction of 3-phenylpropylamines bearing OMe and CF_3 at *meta*- or *ortho*-position all worked well, leading to products **3am**-**3ap** in moderate yields. 3-Phenylpropylamines bearing Me and ester at amino α-position were also tolerable (**3aq**-**3ar**). Besides acylation of benzyl C(sp^3)-H bond, acylation of unactivated aliphatic C(sp^3)-H bond with various (hetero)aromatic aldehydes could also be achieved on this catalytic platform (**3as**–**3aw**). The protecting group Boc could be replaced by methyl without affecting the reaction efficiency (**3ax**). However, unsubstituted substrate **1** did not work for this reaction under current reaction conditions.

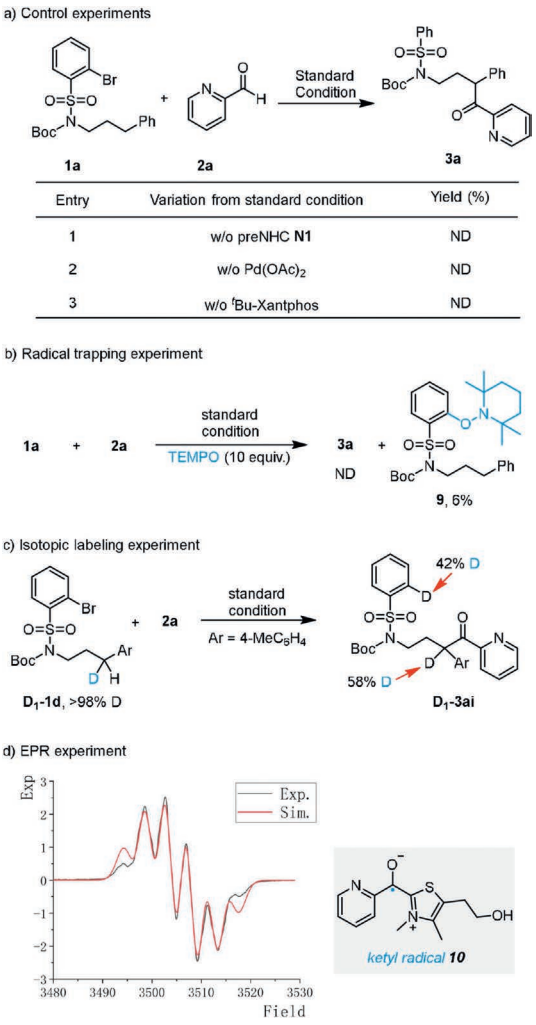
The excellent γ-selectivity observed in these reactions led us to further investigate the limits of the regioselectivity in the key HAT step (Scheme 3). β-C(sp^3)-H acylation occurs *via* 1,6-HAT to furnish β-aminoketones **3ay** in moderate yield, when no γ-C(sp^3)-H is present in the substrate. The reaction of *n*-amylamine exhibits



Scheme 3. Regioselectivity probes of intramolecular HAT.



Scheme 4. Gram-scale synthesis and synthetic transformations.



Scheme 5. Mechanistic study.

exclusive γ-regioselectivity, and β- and δ-C(sp^3)-H are untouched (**3az**). The γ-C(sp^3)-H acylation of aliphatic amine bearing benzyl δ-C(sp^3)-H further confirms 1,7-HAT is highly favored over 1,8-HAT (**3ba**). The excellent regioselectivity may be attribute to the rigid sulfonamide tether, which makes 1,7-HAT favored over the corresponding 1,5-, 1,6- or 1,8-HAT [28,66,67].

To further demonstrate the synthetic potential of this method, gram-scale synthesis and synthetic transformations were carried out (Scheme 4). The efficiency of this reaction was maintained in gram-scale synthesis, affording 1.24 g of ketone **3a** in 64% yield. The removal of the Boc protective group of ketone **3a** gave sulfonamide **4**, which was reduced to afford δ-aminoalcohol **5**. Pyrrolidine **6** was obtained *via* the following intramolecular Mitsunobu reaction. Similar deprotection of ketone **3q** and the following reductive amination afforded pyrrolidine **7** in good yield with 6:1 *dr*. Similarly, ketone **3ag** could be converted into pyrrolidine **8**, which is the key intermediate for the synthesis of BIRZ227, a potentially useful therapeutic agent for treating inflammatory disorders [68].

To go insight into the mechanism, a series of control experiments were carried out (Scheme 5). It is found that preNHC, palladium and ligand are all crucial for the reaction (Scheme 5a). The light on/off experiments reveals that light is essential (see Supporting information for details). When the radical scavenger TEMPO was added, the reaction was completely inhibited and the adduct **9** of aryl radical with TEMPO could be isolated in 6% yield (Scheme 5b). When introducing a deuterium at benzyl position, only the benzene ring of sulfonyl group was partially deuterated, demonstrating the 1,7-HAT between aryl radical and γ-C(sp^3)-H (Scheme 5c). The *in situ* electron paramagnetic resonance (EPR) analysis of the model reaction mixture suggests the generation of ketyl radical **10** (Scheme 5d). In addition, Stern-Volmer quenching studies demonstrated that **1a** quenched the excited palladium catalyst (see Supporting information for details).

Based on the mechanistic experiments and previous reports [69–71], a plausible catalytic cycle is shown in Fig. 1. An inner-sphere single electron transfer from photo-excited $Pd^{(0)}L$ species to aryl bromide **1** produces aryl radical **I** and $Pd^{(I)}L$ complex. Site-selective γ-C(sp^3)-H activation through aryl radical-mediated 1,7-HAT affords transient alkyl radical **II**. Meanwhile, single electron oxidation of the Breslow enolate intermediate **III** by $Pd^{(I)}L$ complex gives persistent ketyl radical **IV** and regenerates $Pd^{(0)}L$ complex. The radical/radical coupling of ketyl radical **IV** and alkyl radical **II** furnishes the adduct **V**, which is fragmented to afford γ-amino C(sp^3)-H acylation products and release the NHC catalyst.

In conclusion, we have developed a practical acylation of γ-amino C(sp^3)-H bonds with aldehydes as acyl source through cooperative photoredox NHC/Pd catalysis. This approach enables direct conversion of a variety of readily available aliphatic amines to γ-aminoketones in moderate to good yields with excellent and predictable regioselectivity. Mechanistic studies of the γ-amino C(sp^3)-H acylation are consistent with a radical/radical coupling process that entails (a) photoinduced Pd-promoted formation of aryl radical, (b) generation of transient γ-amino alkyl radical through aryl radical-mediated 1,7-HAT, (c) single-electron oxidation of Breslow enolate intermediate to persistent ketyl radical, and (d) radical/radical coupling of γ-amino alkyl radical with ketyl rad-

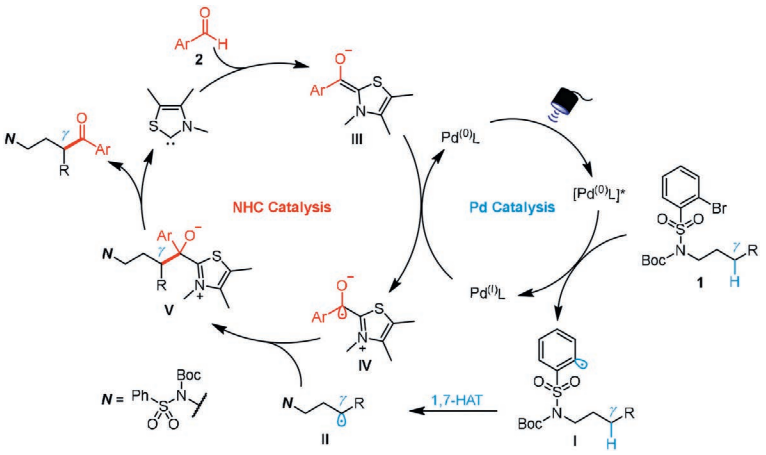


Fig. 1. Proposed catalytic cycle.

ical. Additionally, we have shown that the γ-aminoketones are easily manipulated to enable access to pyrrolines and δ-amino alcohols of interest in medicinal chemistry, thus highlighting the synthetic potential of this methodology.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Xin-Han Wang: Methodology, Investigation. **Ying Huang:** Investigation. **Chun-Lin Zhang:** Writing – review & editing, Writing – original draft, Supervision. **Song Ye:** Writing – review & editing, Project administration.

Acknowledgments

Financial support from the National Natural Science Foundation of China (Nos. 22271292; 21831008) and Beijing National Laboratory for Molecular Sciences (No. BNLMS-CXXM-202003), National Key R&D Program of China (No. 2023YFF0723900) and the Ministry of Science and Technology of China is greatly acknowledged.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2025.111484.

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