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An unusual carbon radical-mediated ring-opening/amination or esterification cascade of unstrained α-haloalkyl cycloketone derivatives

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ABSTRACT

The carbon-centered radical induced C–C bond cleavage in strained cyclopropane is a well-known process. However, achieving similar transformations in unstrained rings is still challenging. Herein, a novel inexpensive Cu-catalyzed strategy for the carbon-centered radical-mediated ring-opening/amination or esterification cascade of unstrained α-haloalkyl cycloketone derivatives is presented. Differ from the classical Dowd-Beckwith ring expansion reaction, a ring opening cascade occurred successfully under a simple copper/base catalytic system, yielding useful distal unsaturated amides and esters in good to excellent yields. Mechanistic studies and DFT calculations indicate that the halogen-bonding interaction between halide and base is crucial for the activation of the C–Br bond. Furthermore, the formation of a bicyclic Cu-complex *via* bi-dentate coordination, including O–Cu–$^{\bullet}CH_2$, seems to be a crucial factor that promotes ring opening rather than the conventional ring expansion. This work would not only bring new vitality for the radical-mediated C–C bond cleavage, but also highlight the potential of protocol for synthesizing bioactive molecules.

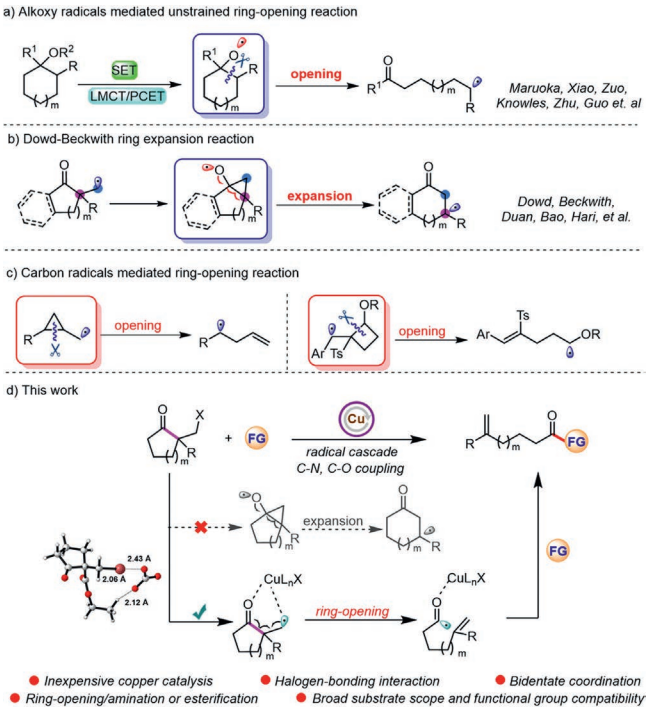
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The direct activation/functionalization of carbon-carbon σ bond has always been considered as one of the hot topics in organic synthesis [1–7]. In this field, the cleavage of carbon-carbon bonds in unstrained cyclic ketone has long been challenging and has attracted considerable attention from chemists [8–12]. In recent years, alkoxy radical-mediated cyclic C–C bond cleavage has significantly advanced. To access reactive alkoxy radical intermediates, various strategies have been reported, such as the direct activation of O–H and O-heteroatom bonds in alcohol and their derivatives (Scheme 1a) [13–23]. Additionally, the radical addition of carbon-centered radicals to the carbonyl group provided another strategy [24]. Among these, the most famous example is the Dowd-Beckwith ring expansion reaction (Scheme 1b) [25–31]. Recently, our group [32,33], Hari's group [34] and Bao's group [35] have successfully achieved the Dowd-Beckwith ring expansion/coupling reaction under the conditions of precious metal catalysis (Pd, Ir) or radical initiator, respectively, which produced the medium cyclic ketones with various functional groups.

As we know, the C–C bond cleavage initiated by the carbon-centered radical is a classical transformation in radical chemistry [36–39]. In particular, the ring-opening reactions of cyclopropylmethyl radicals have been commonly used to verify a radical pathway for mechanistic investigation. In addition, Zhang and co-workers [40,41] reported the carbon-centered radical mediated ring-opening/functionalization of 4-membered ring by strain release, providing a facile approach to the γ,δ-unsaturated aldehydes of ketones (Scheme 1c). However, the 5- and 6-membered ring analogues did not undergo such a transfer probably due to the lower ring strain and the rapid reverse cyclization [42,43]. We wonder whether unstrained ring opening could be facilitated through carbon-centered radical-mediated strategy. Herein, we disclose an unusual copper-catalyzed ring opening/amination and esterification of unstrained α-haloalkyl cycloketone derivatives, which provides a variety of distal unsaturated amides and esters in good yields (Scheme 1d). Mechanistic studies suggest a radical pathway for this transformation and halogen-bonding interaction is critical for initiating the reaction. Moreover, we speculate

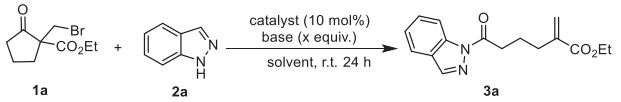
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Scheme 1. Radical-mediated C–C bond cleavage.

Table 1
Optimization of the reaction conditions.[a]

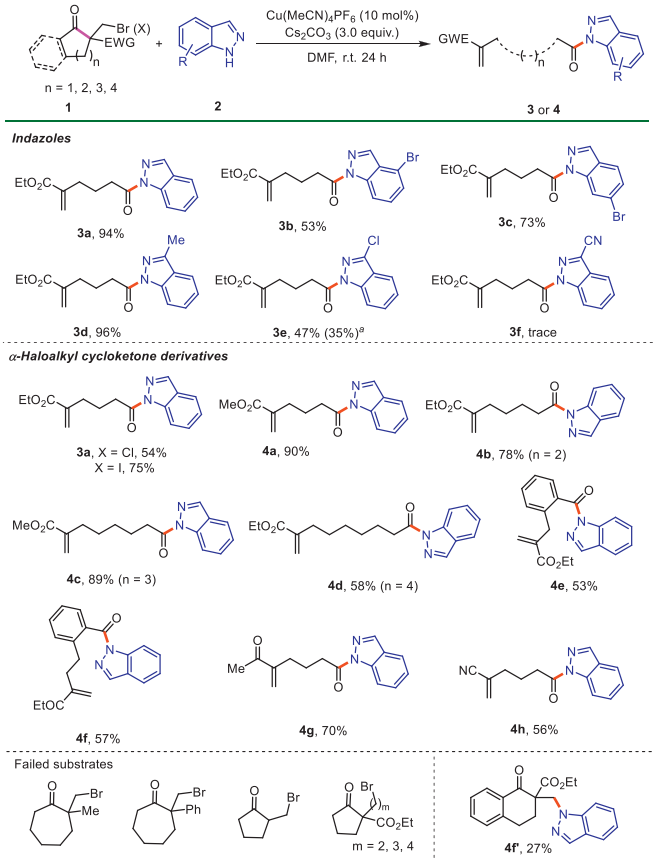


Entry	Catalyst	Solvent	Base (equiv.)	Yield (%)[b]
1	$Cu(MeCN)_4PF_6$	DMF	Cs_2CO_3 (2)	83
2	CuBr	DMF	Cs_2CO_3 (2)	53
3	$Cu(OTf)_2$	DMF	Cs_2CO_3 (2)	45
4	$CuBr_2$	DMF	Cs_2CO_3 (2)	41
5	$Fe(OTf)_2$	DMF	Cs_2CO_3 (2)	68
6	$ZnCl_2$	DMF	Cs_2CO_3 (2)	Trace
7	$Cu(MeCN)_4PF_6$	DMF	DBU (2)	34
8	$Cu(MeCN)_4PF_6$	DMF	*t*-BuOK (2)	30
9	$Cu(MeCN)_4PF_6$	DMF	K_2CO_3 (2)	37
10	$Cu(MeCN)_4PF_6$	DMA	Cs_2CO_3 (2)	70
11	$Cu(MeCN)_4PF_6$	DMSO	Cs_2CO_3 (2)	12
12	$Cu(MeCN)_4PF_6$	Toluene	Cs_2CO_3 (2)	28
13	$Cu(MeCN)_4PF_6$	DMF	Cs_2CO_3 (3)	94 (trace)[c]
14	$Cu(MeCN)_4PF_6$	DMF	–	n.r.
15	–	DMF	Cs_2CO_3 (3)	Trace

[a] Reaction conditions: **1a** (0.3 mmol, 1.5 equiv.), **2a** (0.2 mmol, 1.0 equiv.), catalyst (10 mol%), base (x equiv.), solvent (2 mL), r.t., for 24 h, under N_2.
[b] Yield of isolated product.
[c] K_2CO_3 (4 equiv.), DMAP (20 mol%), DCM, reflux, 72 h.

that copper engaged in bidentate coordination with both the carbonyl group and the carbon radical to form a bicyclic Cu-complex intermediate, which not only effectively inhibits ring expansion, but also activates the pre-cleavage carbon-carbon bond. Furthermore, the formation of stable acyl radicals and α,β-unsaturated fragments are important driving forces for this C–C bond cleavage. Remarkably, this protocol can be applied to the modification of drug molecules and fragments.

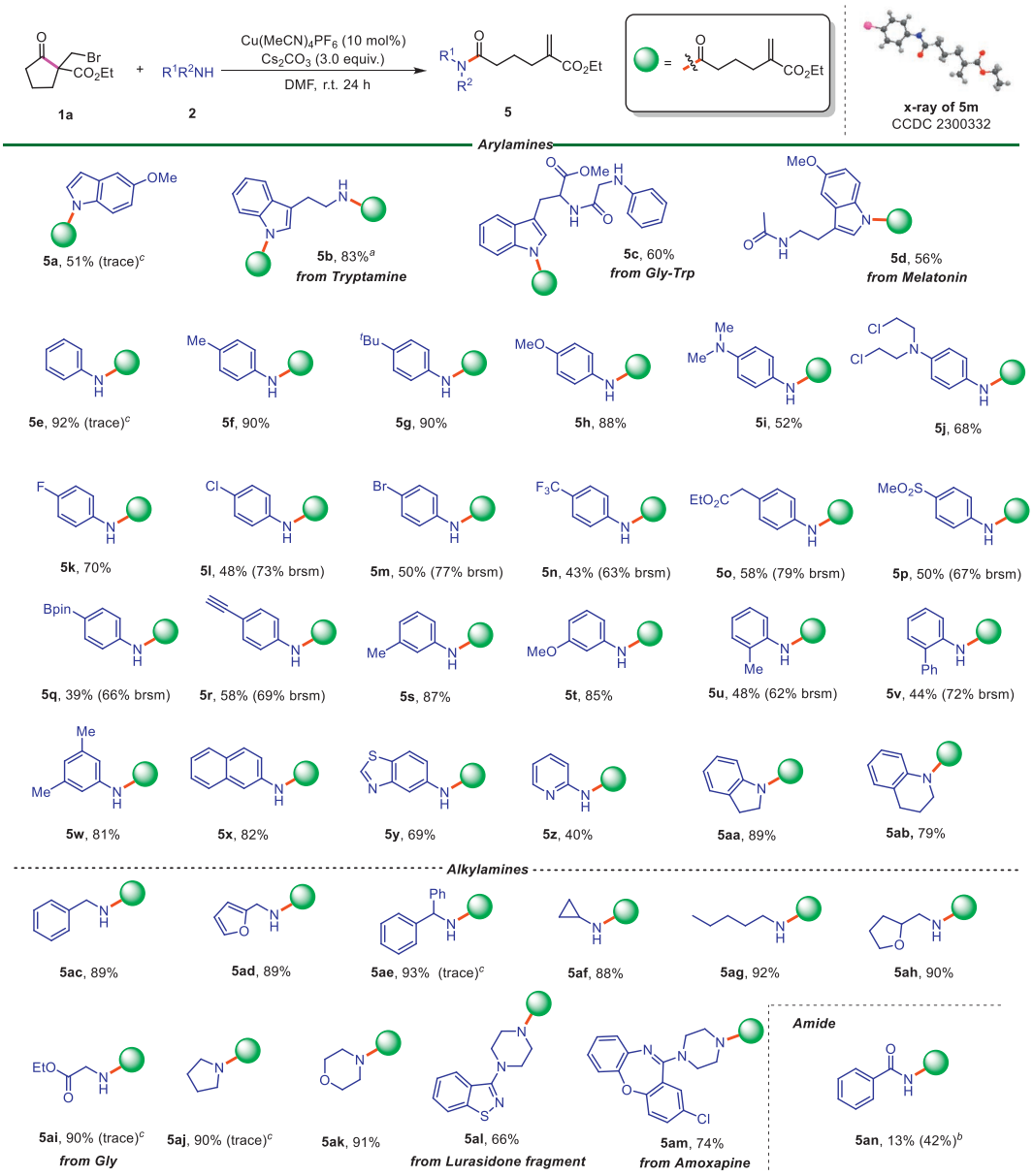
Initially, the ethyl 1-(bromomethyl)-2-oxocyclopentane-1- carboxylate **1a** and indazole **2a** were chosen as model substrates to find the optimal reaction conditions (Table 1). To our delight, it was found that the reaction proceeded smoothly in the presence of $Cu(MeCN)_4PF_6$ (10 mol%) and Cs_2CO_3 (2 equiv.) in DMF for 24 h,



Scheme 2. Scope of indazoles and α-haloalkyl cycloketone derivatives. Reaction conditions: **1** (0.3 mmol, 1.5 equiv.), **2** (0.2 mmol, 1.0 equiv.), 10 mol% of $Cu(MeCN)_4PF_6$, Cs_2CO_3 (0.6 mmol, 3.0 equiv.), DMF (2.0 mL), r.t., for 24 h, under N_2. Isolated yields. [a] Yield of **3a**.

yielding the ring opening product **3a** in 83% yield (entry 1). Other copper salts, such as CuBr, $Cu(OTf)_2$ and $CuBr_2$ were all efficient catalysts for the reaction, but gave lower yields than $Cu(MeCN)_4PF_6$ (entries 2–4). The use of $Fe(OTf)_2$ as catalyst also afforded the product **3a** in 68% yield, while Lewis acid $ZnCl_2$ resulted in only a trace amount of **3a** (entries 5 and 6). A variety of organic and inorganic bases were tested, with Cs_2CO_3 proving to be the best base (entries 7–9). Solvent screening revealed that polar solvents were more suitable for this transformation, and DMF was still the optimal one (entries 10–12, for other solvents see Supporting information). Fortunately, increasing the amount of Cs_2CO_3 from 2.0 equiv. to 3.0 equiv. improved the yield of **3a** up to 94% (entry 13). In addition, the reaction yield was poor when the reaction was carried out with K_2CO_3 and DMAP under reflux in DCM for 72 h (entry 13) [44]. Finally, control experiment revealed that base and catalyst are essential for this reaction (entries 14 and 15).

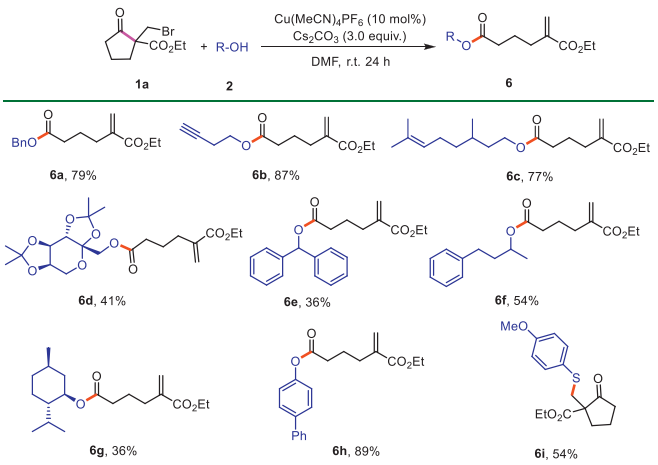
With the optimized conditions established, we firstly investigated the scope of indazoles **2** using ethyl 1-(bromomethyl)-2-oxocyclopentane-1-carboxylate **1a** as model substrate (Scheme 2). Among them, 4–bromo-1*H*-indazole and 6–bromo-1*H*-indazole afforded the products **3b** and **3c** in 53% and 73% yields, respectively. Substrates with an electron-donating group on heterocyclic ring gave better yields than those with an electron-withdrawing group (**3d** *vs.* **3e**, **3f**). Notably, in addition to **3e**, the dechlorination product **3a** was also isolated in 35% yield when 3–chloro-1*H*-indazole was used as substrate. Subsequently, we explored the scope of α-haloalkyl cycloketone derivatives **1** using indazole **2a** as a nucleophile under the optimized conditions (Scheme 2). The desired product **3a** was obtained from both the analogous chloride



Scheme 3. Scope of structurally diverse amines. Reaction conditions: **1a** (0.3 mmol, 1.5 equiv.), **2** (0.2 mmol, 1.0 equiv.), 10 mol% of $Cu(MeCN)_4PF_6$, Cs_2CO_3 (0.6 mmol, 3.0 equiv.), DMF (2.0 mL), r.t., for 24 h, under N_2. Isolated yields. [a] **1a** (0.4 mmol, 2.0 equiv.) was used. [b] *t*-BuOK (3.0 equiv.) instead of Cs_2CO_3. [c] K_2CO_3 (4 equiv.), DMAP (20 mol%), DCM, reflux, 72 h.

and iodide in 54% and 75% yields, respectively. A range of bromides with five- to eight-membered rings also afforded the corresponding products **4a**-**4d** in moderate to excellent yields. The 1-indanone-derived substrate also gave the product **4e** in 53% yield. With the 1-tetralone-derived substrate, both the desired product **4f** and a by-product **4f'** were isolated. In addition, the **4f'** could not transfer to **4f**. Notably, substrate **1** with an acetyl group instead of the ester group also worked well, giving the target product **4 g** in 70% yield. Substrate with a cyano group was also compatible with the reaction system, giving the desired product **4h** in 56% yield. However, replacing the ester with a phenyl or methyl group resulted in no formation of the desired products, with 81% and 78% of **2a** recovered, respectively. Unfortunately, 2-(bromomethyl)cyclopentan-1-one and substrates with two to four-carbon side chain did not undergo this ring-opening reaction.

Next, we continued to evaluate the scope of amines **2** using bromide **1a** as model substrate (Scheme 3). Both aromatic and aliphatic amines reacted well with **1a** to afford the chain-unsaturated amides **5** in moderate to excellent yields. The structure of product **5m** was further verified by X-ray crystal diffraction. 5-Methoxyindole furnished the target product **5a** in 51% yield. Remarkably, the tryptamine was also amenable, furnishing the diacylated product **5b** in 83% yield when 2 equiv. of **1a** was used. The dipeptide Gly-Trp afforded product **5c** in 60% yield. The natural product melatonin afforded the desired product **5d** in acceptable yield. In addition, it was found that both electronic and steric effects on the aromatic ring had an obvious influence on the reaction efficiency. Aromatic amines with electron-donating groups at the para position of benzene ring gave better yields than those with an electron-withdrawing group (**5f**-**5j** *vs.* **5k**-**5n**). Arylamines with *o*-methyl and *o*-phenyl groups gave the products **5u** and **5v** in relatively lower yields. Notably, a variety of functional groups, including ester (**5o**), sulfonyl (**5p**), Bpin (**5q**) and alkynyl (**5r**) were well preserved under this copper catalyst system. 3,5-Dimethylaniline



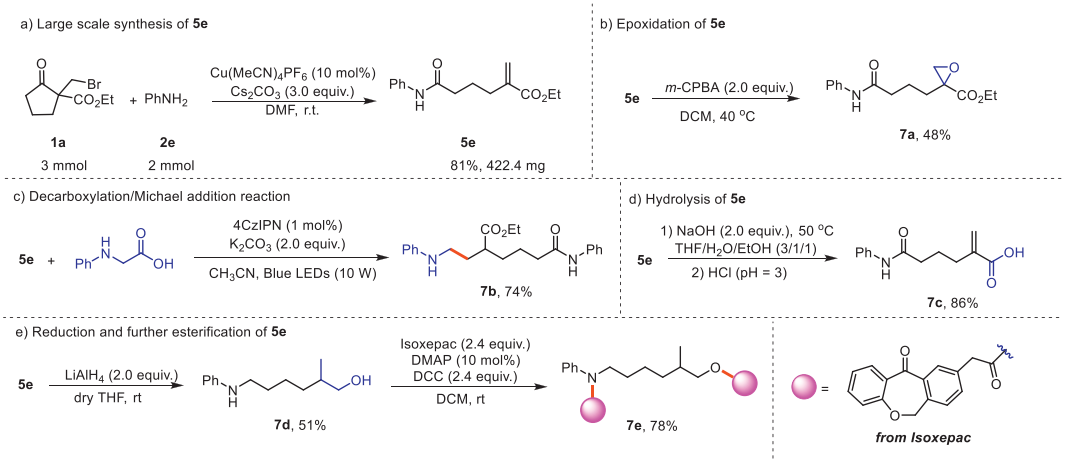
Scheme 4. Scope of structurally diverse alcohols. Reaction conditions: **1a** (0.3 mmol, 1.5 equiv.), **2** (0.2 mmol, 1.0 equiv.), 10 mol% of $Cu(MeCN)_4PF_6$, Cs_2CO_3 (0.6 mmol, 3.0 equiv.), DMF (2.0 mL), r.t., for 24 h, under N_2. Isolated yields.

and 2-naphthylamine were also converted into the corresponding products **5w** and **5x** in excellent yields. Heteroaryl amines, such as 5-benzothiazolamine and 2-aminopyridine, also participated well in this reaction, giving the products **5y** and **5z** in moderate yields. When indoline and tetrahydroquinoline were employed, **5aa** and **5ab** were obtained in good yields. The alkyl amines were then evaluated. Satisfactorily, both primary and secondary alkyl amines were engaged well in this reaction, giving the expected products **5ac**-**5ak** in good to excellent yields. Drug molecules and fragments, such as lurasidone and amoxapine, were successfully acylated by the ring-opening of **1a** to give the desired products in satisfactory yields (**5al**-**5am**). In addition, benzamide also furnished desired product **5an** in 42 % yield with *t*-BuOK as base. In particular, in this study we successfully converted various amines, including aromatic (hetero) amines and aliphatic amines (such as **5a**, **5e**, **5ae**, **5ai**, **5aj**), which were inaccessible with a previous K_2CO_3/DMAP-catalyzed method. These results suggest that the main reaction pathway differ from Grob fragmentation [44].

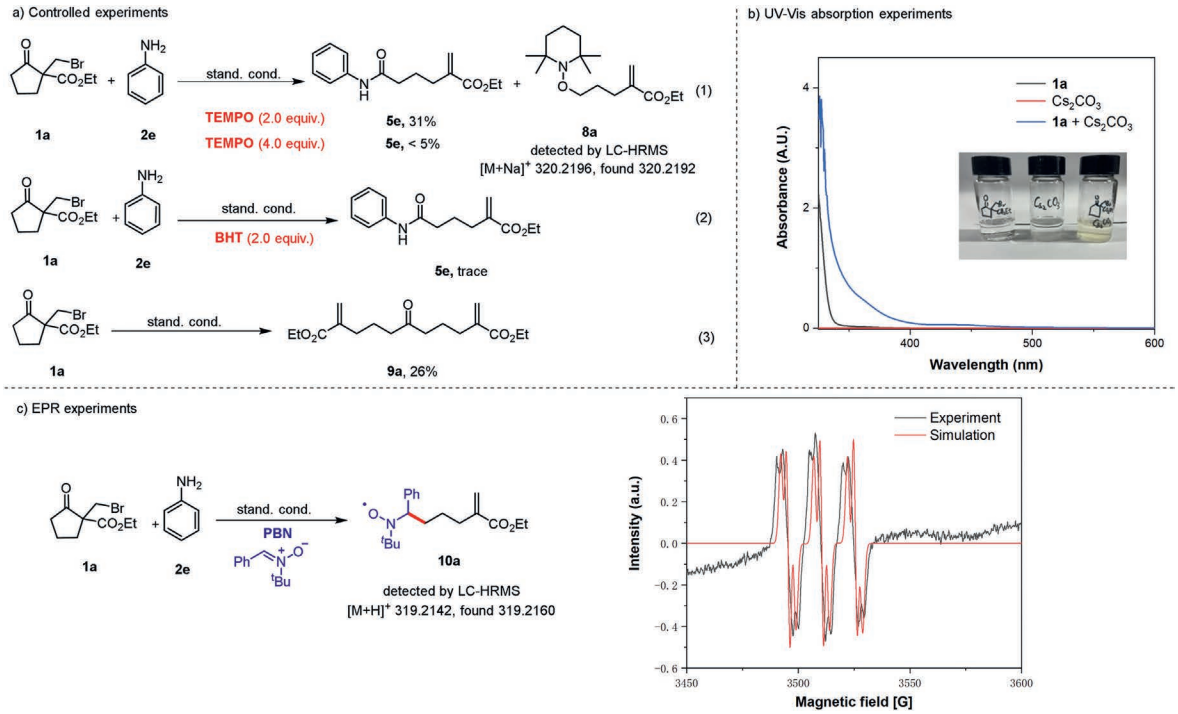
Encouraged by the above results, we explored a structurally diverse range of alcohols as nucleophiles for this reaction (Scheme 4). Benzyl alcohol reacted well to afford the expected ester **6a** in 79 % yield. Alkenyl and alkynyl alcohols also successfully produced the products **6b** and **6c** in 87 % and 77 % yields, respectively. Diacetonefructose was also applicable and gave the product **6d** in 41 % yield. Additionally, secondary alcohols, including diphenylmetanol (**6e**), 4-phenyl-2-butanol (**6f**) and menthol (**6 g**), provided the target products in moderate yields. Reasonably, sterically bulky tertiary alcohol, such as butanol, failed to yield the expected product (not shown). Notably, *p*-phenylphenol was a viable substrate, furnishing the desired product **6h** in excellent yield. Unfortunately, thiophenol was not compatible with this reaction, leading to the formation of thioether **6i** as a byproduct.

To demonstrate the application of this protocol, a 2.0 mmol scale model reaction was conducted, yielding **5e** in 81 % yield after extending the reaction time (Scheme 5a). Subsequently, various derivations of product **5e** were explored. The epoxidation of **5e** with *m*-CPBA resulted in the product **7a** in a 48 % yield (Scheme 5b). A visible light-mediated decarboxylative Michael addition of **5e** with *N*-phenylglycine also proceeded efficiently to provide product **7b** in good yield (Scheme 5c). Product **5e** can be hydrolyzed chemoselectively to afford the carboxylic acid **7c** in 86 % yield (Scheme 5d). Notably, treatment of **5e** with $LiAlH_4$ provided the alcohol **7d** in moderate yield, which could be further esterified with the drug molecule isoxepac to give the aminyl-containing ester **7e** in 78 % isolated yield (Scheme 2e). In addition, the effect of product **5j** on the cell viability of HeLa cells was examined, MTT assay shown **5j** has potential *in vitro* anticancer activity against HeLa cells and significant reduction in MTT activity with increasing concentrations of **5j** (Fig. S1 in Supporting information).

To elucidate the mechanism of this reaction, several control experiments were performed (Scheme 6). Addition of different amounts of TEMPO led to obviously decreased yields of **5e** and a decarbonylative TEMPO-adduct **8a** was detected by LC–HMRS (Scheme 6a, Eq. 1). The addition of BHT (2.0 equiv.) also resulted in a significant inhibition of this reaction (Scheme 6a, Eq. 2). These results suggest that a radical pathway may be involved in this transformation. Furthermore, in the absence of **2e**, substrate **1a** decomposed to the compound **9a** under standard conditions (Scheme 6a, Eq. 3), and the release of CO could be detected by GC (for details, see Supporting information). These findings imply that an acyl radical might be generated in this transformation. To clarify the role of the base, NMR titration experiments of **1a** were performed with different amounts of Cs_2CO_3 (Scheme 6b). It was observed that the signals of α-H or α-C of bromide **1a** shifted downfield in the ^1H NMR and ^{13}C NMR spectra, suggesting the possible formation of a halogen-bonding complex [40,41]. Additionally, UV–vis analysis indicated the formation of a complex between bromide **1a** and Cs_2CO_3 (Scheme 6c). Notably, the EPR experiment using the spin trapping agent *N*–*tert*–butyl–α-phenylnitrone (PBN) confirmed the formation of a radical intermediate in this transforma-



Scheme 5. Large-scale synthesis and derivatizations.



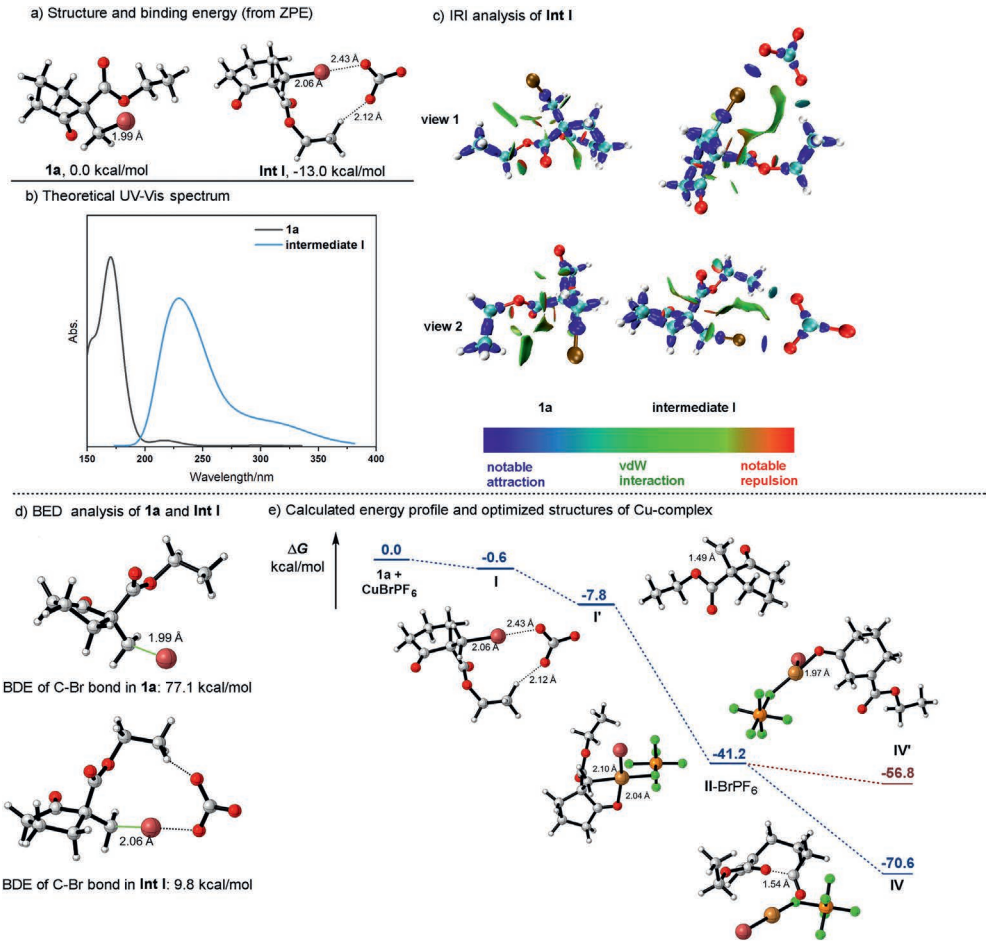
Scheme 6. Mechanistic studies.

tion (Scheme 6d). Finally, the Cu(III) complexes were successfully detected by LC–HMRS (for details, see Supporting information).

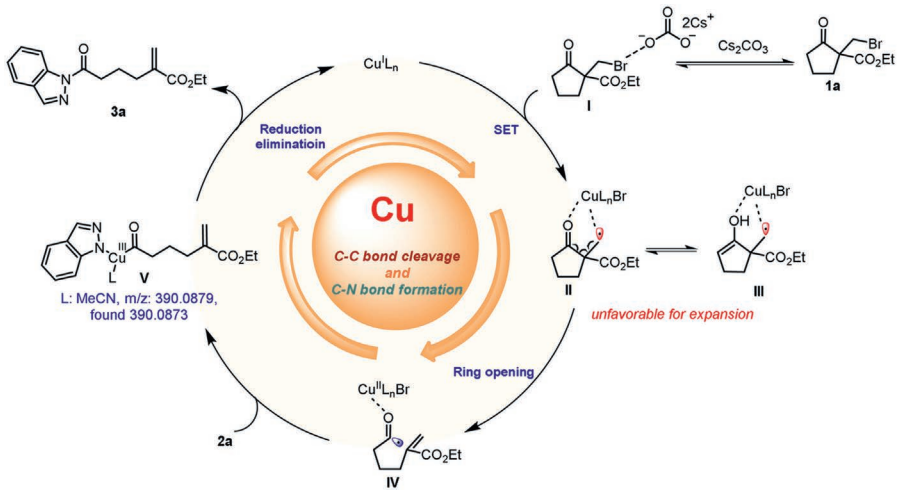
To gain a deeper understanding of the reaction mechanism, we employed density functional theory (DFT) for a comprehensive theoretical study of the intermediate **I** through halogen bond that formed by **1a** and CO_3^{2-} negative ion, was carried out, and the resulting structure and binding energy are presented in Scheme 7. The C–Br bond in intermediate **I** was found to be elongated by 0.07 Å (from 1.99 Å to 2.06 Å) compared to **1a**, suggesting a possible weakening of the C–Br bond. The distance between O atom and Br atom is 2.43 Å, and the distance between O atom and H atom is 2.12 Å, indicating a potential halogen bond between O atom and Br atom, as well as a non-covalent interaction between O atom and H atom. The favorable binding energy of intermediate **I**, measured at −13.0 kcal/mol, indicates its energetically favorable formation (Fig. 7a). Subsequently, we analyzed the interactions within intermediate **I** using the interaction region indicator (IRI) analysis, as shown in Scheme 7c. The analysis also revealed a robust halogen bond between the O atom and the Br atom, depicted in blue, and an additional interaction between the O atom and the H atom, with a strength comparable to that of a van der Waals interaction. Furthermore, time-dependent density functional theory (TD-DFT) was used to calculate the theoretical UV–vis spectra of **1a** and intermediate **I**, as illustrated in Scheme 7b. The UV–vis absorption of intermediate **I** exhibited an obvious redshift compared to **1a**, aligning with the experimental observations. Then, the FMOs analysis of **1a** and intermediate **I** were also performed and shown in Fig. S9a (Supporting information). We found that the energy gap of intermediate **I** was significantly lower than **1a**. Finally, we calculate the BDE of the C–Br bond and find it decreases significantly after combining the CO_3^{2-} (Scheme 7d), which may provide an explanation for the activation of substrate **1a** by the halogen bond.

Based on the above results and literature [44–52], a probable mechanism for the ring-opening/amination of the α-haloalkyl cycloketone derivatives is proposed (Scheme 8). Initially, substrate **1a** is activated by Cs_2CO_3, forming a halogen-bonding complex **I**. Then, a single electron transfer (SET) event occurs between Cu(I) and complex **I** to give the Cu-complexed radical intermediate **II** or the enolized complex **III**. Subsequently, intermediate **II** undergoes a homolytic C–C cleavage, likely driven by the formation of stable α,β-unsaturated fragments, to give the acyl radical intermediate **IV**. This intermediate rapidly recombines with Cu(II) in the presence of indazole **2a** to give the Cu(III) complex **V**. Finally, reductive elimination of Cu(III) complex **V** produces the desired product **3a** and regenerates the Cu(I) complex. For this unusual ring-opening transformation, we hypothesize that the formation of bicyclic Cu-complex intermediate **II** or **III** may effectively inhibit the ring expansion reaction. To gain more insight into these intermediates, we conducted the structure and energy analysis using DFT calculations. The findings of this study demonstrate that the carbon-centered radical intermediates produced by the SET process have the capacity to form stable complexes **II**-$BrPF_6$ with $CuBrPF_6$. Furthermore, the energy of the corresponding ring-opening product **IV** is significantly reduced to −70.6 kcal/mol, indicating that the ring-opening process of **II**-$BrPF_6$ is energetically favorable. In contrast, the energy of the Dowd-Beckwith ring expansion product **IV'** is −56.8 kcal/mol, which is significantly higher than that of **IV**. From the perspective of product stability, the Dowd-Beckwith ring expansion is not a favorable pathway (Scheme 7e). IRI analysis reveals the presence of notable attractive interactions between Cu—O and Cu—$^{\bullet}CH_2$, suggesting the potential for coordination between the Cu—O and the possibility of covalent bonding between Cu—C cannot be ruled out (Fig. S9b in Supporting information). Spin density analysis was performed to study the spin delocalization of Cu complex **II**-$BrPF_6$, the unpaired electrons on the Cu atoms are dispersed to the ketocarbonyl oxygen (Fig. S9c in Supporting information). In addition, the charge analysis also shown that the charge of the carbonyl carbon increased from 0.433 to 0.518 (Fig. S9d in Supporting information). These results revealed the pre-cleavage carbon-carbon bond was activated after formation Cu complex **II**-$BrPF_6$. Consequently, the generation of the stabilized Cu complex may be the pivotal factors in the ring-opening reaction.

In conclusion, we have developed a novel carbon-centered radical-mediated ring-opening/amination or esterification cascade



Scheme 7. Computational studies.



Scheme 8. Proposed mechanism.

of α-haloalkyl cycloketone derivatives under copper catalysis. A range of α-haloalkyl cycloketone derivatives underwent a unique C–C bond cleavage model, producing the chain-unsaturated amides and esters that are traditionally challenging to synthesize. This protocol is characterized by mild conditions, operational simplicity, broad substrate scope and excellent functional group compatibility. Mechanistic studies and DFT calculations indicate that the halogen-bonding interaction between halide and base is crucial for the activation of the C–Br bond. Furthermore, the formation of a bicyclic Cu-complex, including O–Cu–$^{\bullet}CH_2$, favors the ring-opening reaction over the typical ring expansion. This work would bring new vitality for the radical-mediated C–C bond cleavage.

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

Hong Xin: Writing – original draft, Methodology, Data curation, Conceptualization. **Shutao Wang:** Formal analysis, Data curation. **Li-Na Guo:** Writing – original draft. **Cheng Guan:** Methodology. **Jinyi Liao:** Methodology. **Zi-Hang Yuan:** Data curation. **Zhengze Zhang:** Software. **Xin-Hua Duan:** Project administration, Conceptualization.

Acknowledgments

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2025.111939.

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