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Photoinduced deacylative epoxidation of ketone derivatives with allylic peroxides

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Epoxide compounds represent a privileged scaffold with diverse applications in organic synthesis, pharmaceuticals, agrochemicals, and materials science. However, the available routes to obtain such high-value structures from feedstocks, such as ketones, remain less developed. In this work, we report an alternative deacylative epoxidation of ketones using allylic peroxides to produce fused epoxides via the photoinduced radical cyclization of ketone-derived pro-aromatic dihydroquinazolinones. This system operates under very mild conditions and showcases a broad substrate scope with excellent functional group tolerance (over 50 examples). Its gram-scale preparation and late-stage transformations emphasize its potential in drug discovery. A possible mechanism has been proposed based on mechanistic studies.

Introduction

Strained three-membered oxygen heterocycles, known as epoxides or oxiranes, are highly valuable motifs in the drug industry with remarkable biological and pharmaceutical activities and are commonly observed in naturally occurring substances, pharmaceutical compounds, materials and synthetic molecules, including 14 FDA-approved drugs (Scheme 1a).¹ In addition, spring-loaded oxiranes have inherent angle strain and two polar bonds, making them highly reactive and rendering them excellent reactive intermediates for constructing new bonds in larger and more complex molecules through nucleophilic ring-opening reactions in the presence of different nucleophiles, such as organolithium, amine, cyanides, thiols, halides and azides.^{2,3} The global market for oxiranes is expected to reach \$78 billion by 2025, fueled by various applications, such as the production of epoxy resins, surfactants, adhesives, coatings, agrochemicals, and pharmaceuticals.⁴

Therefore, the efficient incorporation of oxirane units into organic molecules is an important and challenging objective, particularly when utilizing abundant feedstock chemicals under simple conditions. However, the majority of state-of-the-art methodologies for synthesizing oxiranes are limited to the epoxidation of specific single alkenes using strong oxidizing agents, such as peroxy acids or hydrogen peroxide.^{1,5} To explore more readily available substrates and facilitate operational

processes, as well as to achieve greater structural diversity in oxiranes, it would be immensely beneficial to develop a straightforward method for converting libraries of cheap feedstocks into their corresponding oxiranes, addressing growing demands in chemical biology and drug discovery.

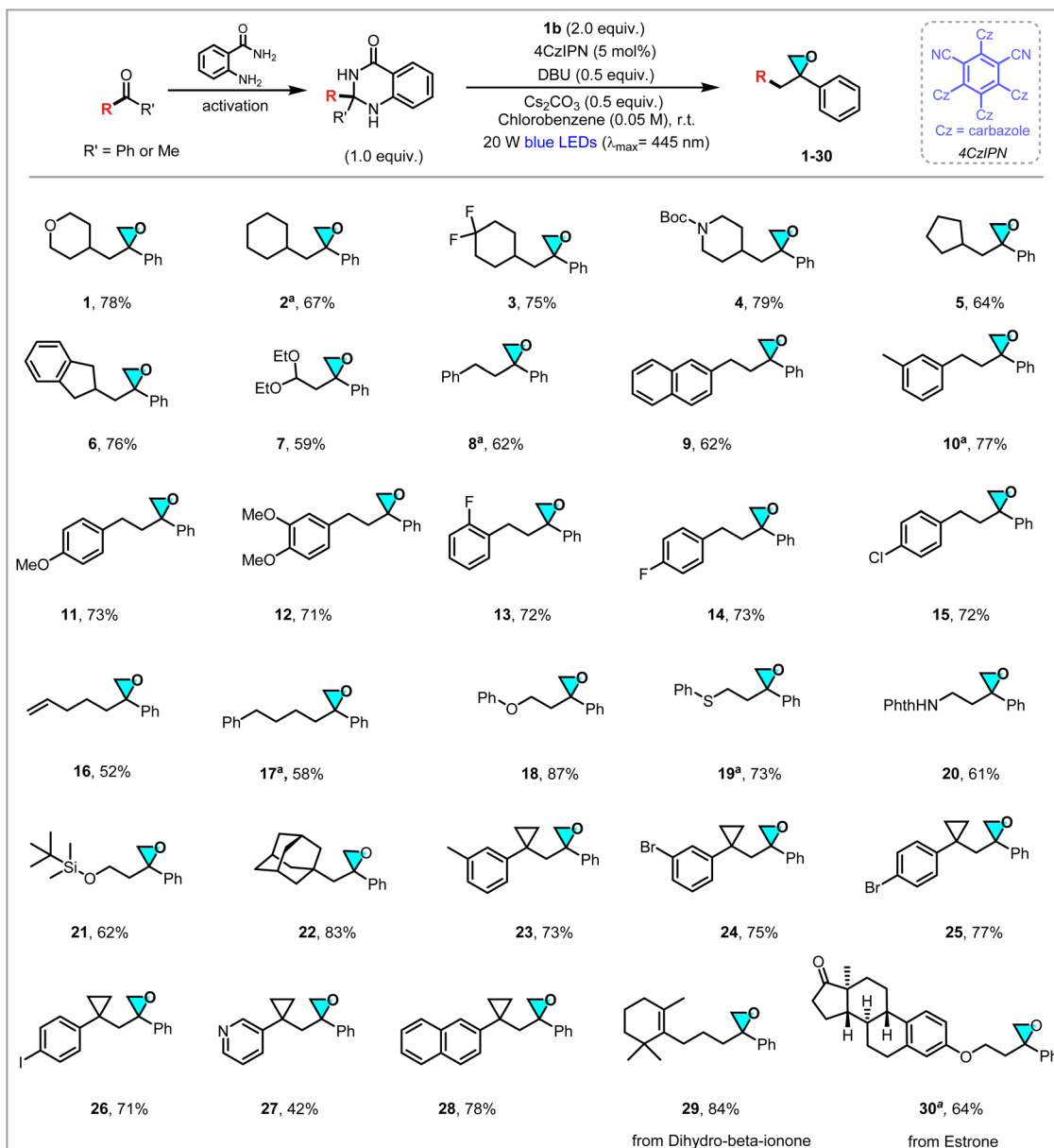
Among them, ketones (carbonyl compounds) are one of the most common and widely used fundamental skeletons found in many industrial chemicals and pharmaceutical architectures.⁶ For example, Darzens condensation involves the reaction of aldehydes or ketones with α -mono- or dihalocarbonyl compounds in the presence of bases to form epoxides (Fig. 1b, left),⁷ and the Johnson–Corey–Chaykovsky method⁸ is based on the reaction of carbonyl compounds with sulfonium and sulfoxonium ylides, which are often derived *in situ* from sulfonium or sulfoxonium halides containing the mono- or disubstituted methyl group (Fig. 1b, right). However, these well-known reactions still suffer from limitations, such as the requirement for strong bases, aldehyde self-condensation, and the failure to achieve tri-alkyl sulfur ylides. The further elaboration of complex functionalities associated with ketone-containing molecules, such as amino acids, peptides and sensitive drug candidates, poses a synthetic challenge for epoxidation reactions, primarily due to the limited number of reliable methods currently available and their poor compatibility under harsh conditions.

Despite this outstanding work, the exploration of conceptually distinct strategies—especially those avoiding harsh reaction conditions, such as high temperature, excessive additives, long reaction times and limited substrate scope—remains an urgent issue. Developing alternative methods for the direct activation and functionalization of these carbonyl compounds to oxiranes is crucial for improving the quality of chemical raw materials

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Scheme 1 Substrate scope for ketones. Reaction conditions: ketone derivatives (0.2 mmol, 1.0 equiv.), **1b** (0.4 mmol, 2.0 equiv.), 4CzIPN (5 mol%), DBU (0.1 mmol, 0.5 equiv.), Cs₂CO₃ (0.1 mmol, 0.5 equiv.), chlorobenzene (0.05 M), room temperature, under 20 W blue LEDs ($\lambda_{\text{max}} = 445 \text{ nm}$), 24 h, in a vial. Isolated yield. The R' group of the ketone used as the raw material is a phenyl (R' = Ph) unless otherwise noted. The specific reaction raw materials are referenced in the Supporting Information. ^aThe R' group of the ketone used as the raw material is a methyl group (R' = Me).

and for the late-stage modification of complex natural products and pharmaceuticals. However, due to the strong bond dissociation energy (BDE) of the C=O double bond, deoxygenation transformations of these functional groups present considerable challenges, especially for unstrained common ketones. Based on Dong's pioneering work on the deconstructive transformations of ketones,⁹ alkyl radicals can be generated *via* decylation through pre-aromatization intermediates like

dihydropyrazoles and dihydrotriazoles. Recently, innovative methods have emerged for the deconstructive reactions of ketones, involving their transformation into pro-aromatic compounds known as dihydroquinazolinones (DHQZs), with related studies being reported consecutively (Fig. 1c).^{10,11} Seeking a complementary route that would allow the direct transformation of unstrained ketones to oxiranes, we



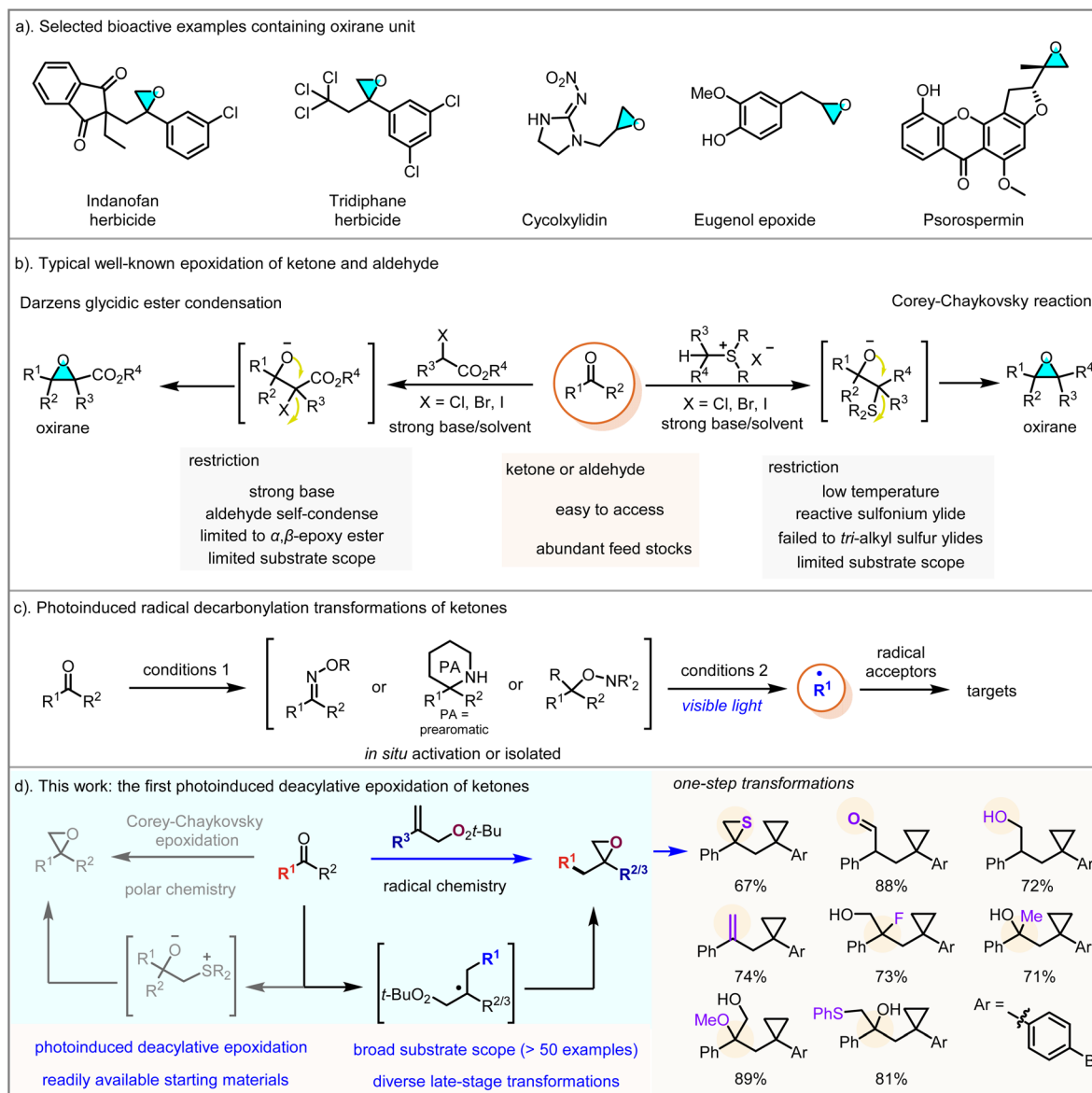


Fig. 1 Background for the synthesis of epoxides and our design. (a) Selected bioactive examples containing oxirane unit. (b) Typical well-known epoxidation of ketone and aldehyde. (c) Photoinduced radical decarbonylation transformations of ketones. (d) This work: the first photoinduced deacylative radical epoxidation of ketones.

questioned whether deacylative epoxidation could occur through the aromatization-driven deconstructive reaction of ketones.

Based on our continuing interest in environmentally friendly visible-light catalytic cyclization,¹² herein, we report our efforts to develop a new method for the direct deacylative epoxidation of unstrained ketones under exceptionally mild and highly compatible conditions, providing an alternative avenue to access diverse epoxides (Fig. 1D). This versatile protocol requires no additional oxidants or organometallic reagents and features considerable substrate scope, including not only

primary, secondary, and tertiary ketones, but also pharmaceutical molecules. Furthermore, the synthetic utility of this protocol is highlighted by its ability to convert epoxide into various valuable functional groups (thiirane, aldehyde, alcohol and other bulky hindered skeletons with quaternary carbon), highlighting its potential applications.

Results and discussion

At the outset of the reaction, DHQZ substrate **1a**, synthesized from a ketone feedstock through a single condensation step,



Table 1 Optimization of the reaction conditions^a

Entry	Variation from standard conditions	Yield of 1 (%) ^b	Conversion of 1a (%) ^b	Explored photocatalysts
1	None	81	100	
2	Ir[dF(CF ₃)ppy] ₂ (dtbbpy)PF ₆	32	65	
3	Ir(ppy) ₂ (dtbbpy)PF ₆	25	68	
4	Ru(bpy) ₂ Cl ₂	9	42	
5	Eosin Y	13	55	
6	fac-Ir(ppy) ₃	17	60	
7 ^c	Other solvents	<29	<57	
8 ^c	Other bases	<32	<64	
9	Without DBU	60	100	
10	Without Cs ₂ CO ₃	38	100	
11	Without light	0	0	
12	Without 4CzIPN	0	0	
13	Under air	31	84	

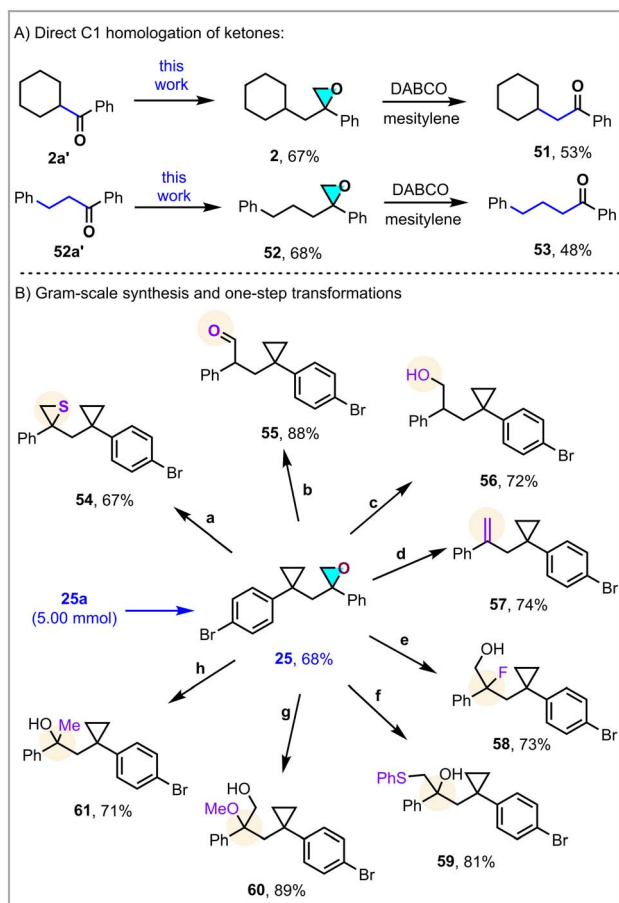
^a Reaction conditions: **1a** (0.1 mmol, 1.0 equiv.), **1b** (0.2 mmol, 2.0 equiv.), photocatalyst (5 mol%), DBU (0.05 mmol, 0.5 equiv.), Cs₂CO₃ (0.05 mmol, 0.5 equiv.), chlorobenzene (0.05 M), room temperature, under 20 W blue LEDs ($\lambda_{\max} = 445$ nm), 24 h, in vial. ^b ¹H NMR yield using 1,2-diethyl phthalate as the internal standard. ^c See SI for details.

and easily accessible **1b** were chosen as the model substrates to explore epoxidation conditions, and selected results are displayed in Table 1. Delightfully, after a survey of reaction conditions, it was found that when a solution of **1a** and **1b** with 0.5 equivalents of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) and cesium carbonate (Cs₂CO₃) in the presence of 5 mol% of 4,5,6-tetra(9*H*-carbazol-9-yl)isophthalonitrile (4CzIPN) in chlorobenzene was stirred for 24 h under irradiation by 20 W blue LEDs ($\lambda_{\max} = 445$ nm) at ambient temperature under N₂ atmosphere, the desired oxirane **1** was obtained in 81% yield (Table 1, entry 1). Other common photocatalysts, such as Ir[dF(CF₃)ppy]₂(dtbbpy)PF₆, Ir[(ppy)₂(dtbbpy)]PF₆, Ru(bpy)₂Cl₂, Eosin Y or *fac*-Ir(ppy)₃, were screened, but provided no improvement over 4CzIPN (Table 1, entries 2–6). The screening of solvents, such as DMSO, DMC, DMA, and NMP, showed that MeCN was more suitable than others (Table 1, entry 7; for more details please see the SI). In addition, different organic or inorganic bases were examined, but no further improvement in yield was obtained (Table 1, entry 8). The developed approach still worked without the involvement of bases DBU or Cs₂CO₃, however, both leading to significantly lower efficiency (Table 1, entries 9 and 10), control experiments revealed that visible light and

a photocatalyst are indispensable to the success of the reaction (Table 1, entries 11 and 12). When the reaction was carried out under air conditions, it led to **4a** in lower yield (Table 1, entry 13).

With the optimal conditions in hand for the deacylative epoxidation reaction, we first sought to explore the scope of ketones. As presented in Scheme 1, a range of structurally diverse methyl and phenyl ketones underwent epoxidation site-selectively at the nonmethyl/phenyl side with allylic peroxide to easily afford the corresponding oxiranes in generally moderate to good yields. First, various carbon cyclic and heterocyclic methyl ketones with various ring sizes reacted smoothly to generate secondary alkyl radicals and to form the desired oxiranes **1–5** in 64–79% yields. Benzo-fused cyclic methyl ketone as an indanyl radical precursor and acetal-derived 2,2-diethoxyacetophenone as a masked formyl radical source proved to be competent substrates, to give the corresponding oxiranes **6** and **7** efficiently in 76% and 59% yields, respectively. The protocol successfully accommodated different benzyl methyl ketones, and the substituted benzyl methyl ketone bearing groups, such as Me, OMe, F and Cl, on the aromatic ring were applied, yielding the corresponding oxiranes **8–15** in 56–77%





Scheme 3 Gram-scale synthesis and synthetic derivatizations of epoxide. Reaction conditions: (A) 51 : 2 (1.0 equiv.), DABCO (4.0 equiv.), mesitylene, 165 °C. 53 : 52 (1.0 equiv.), DABCO (4.0 equiv.), mesitylene, 165 °C. (B) (a) Thiourea (2.0 equiv.), 25 (0.20 mmol, 1.0 equiv.), MeOH (1.0 mL), r.t., 18 h. (b) ZnCl₂ (0.5 equiv.), 25 (0.20 mmol, 1.0 equiv.), toluene (2.0 mL), 110 °C, 4 h. (c) Et₂SiH (2.0 equiv.), TfOH (0.05 equiv.), 25 (0.20 mmol, 1.0 equiv.), HFIP (1.0 mL), r.t., 1 h. (d) PPh₃ (1.2 equiv.), 25 (0.20 mmol, 1.0 equiv.), *i*-PrOH (1.0 mL), 120 °C, 36 h. (e) Et₃N·3HF (2.0 equiv.), BF₃·Et₂O (0.2 equiv.), 25 (0.20 mmol, 1.0 equiv.), DCM (2.0 mL), r.t., 12 h. (f) C₆H₅SnA (1.5 equiv.), 25 (0.20 mmol, 1.0 equiv.), DMF (4.0 mL), 40 °C, 12 h. (g) HCl (37% in H₂O, 0.005 equiv.), 25 (0.20 mmol, 1.0 equiv.), MeOH (2.0 mL), r.t., 2 h. (h) LiAlH₄ (2.0 equiv.), 25 (0.20 mmol, 1.0 equiv.), THF (2.0 mL), r.t., 4 h.

withdrawing nor electron-donating substituents has a significant influence on the reaction. Nevertheless, *ortho*-substituted styrene peroxides markedly reduce the reaction yield, presumably owing to steric hindrance impeding the reaction pathway, as evidenced by products **40** and **47**. Heteroaryl peroxide could also be involved to form oxirane **49** in satisfactory yield (68%) under the current conditions. However, alkyl allylic peroxide resulted in a significantly reduced yield of **50**, likely due to the relatively weak reactivity of alkyl peroxides.

Homologation is a general and valuable synthetic approach used for the elongation of a carbon chain.¹³ As demonstrated in Scheme 3A, one-carbon-inserted ketones **51** and **53** were obtained

in 53% and 48% yields, respectively, suggesting an alternative way to achieve the one-carbon homologation of carbonyl derivatives without modification of the backbone. Moreover, to demonstrate the scalability and robustness of the method, epoxide **25** was synthesized at gram scale (5.0 mmol) with comparable efficiency (68%) (Scheme 3B). In particular, strained oxiranes, as privileged heterocyclic building blocks, are very valuable motifs in organic synthesis and drug development. By the addition of thiourea, thiirane **54** was obtained in 67% yield. Selective oxidation by ZnCl₂ and reduction by Et₂SiH gave primary aldehyde **55** and alcohol **56** in 88% and 72% yields, respectively. Treatment with PPh₃ led directly to the generation of terminal alkene **57** in 74% yield. Ring-opening by Et₃N·3HF formed fluorinated product **58** in 73% yield. Other nucleophiles could open the ring to give compounds **59** and **60** under basic and acidic conditions. Sterically hindered tertiary alcohol **61** was delivered with 71% yield in the presence of LiAlH₄.

To probe the mechanism of this deacylative epoxidation, the following control experiments were conducted, as shown in Scheme 4. First, radical scavengers 2,2,6,6-tetramethylpiperidinoxy (TEMPO) and galvinoxyl fully inhibited the reaction, and cyclohexyl radical adduct **62** was detected by high-resolution mass spectrometry (Scheme 4a). Subjecting cyclopropane-containing dihydroquinazolinone **63** to a radical clock experiment, ring-opening product **16** was isolated in 52% yield (Scheme 4b). Using alkene **64a** as a radical acceptor, no radical addition product **64** was observed, and only a trace of cyclopropanation product **65** was observed, implying that perhaps no direct radical-polar crossover process may be involved (Scheme 4c). Direct irradiation of the reaction with UV light in the absence of a photocatalyst failed to work (Scheme 4d). In addition, short-term photo-irradiation experiments suggested that a radical chain process is unlikely (Scheme 4e). Stern–Volmer fluorescence quenching experiments revealed that dihydroquinazolinone **1a** is an effective quencher for excited-state 4CzIPN* (Scheme 4f). Cyclic voltammetry (CV) studies provided compelling evidence for the ability of the photocatalyst to activate the substrate, leading to the generation of radical species (Scheme 4g).

Based on the above results and previous reports,¹¹ a probable catalytic cycle is proposed (Scheme 4h). Initially, photocatalyst 4CzIPN reached excited-state 4CzIPN* by irradiation with visible light. Then, single-electron transfer (SET) between **1a** ($E_{1a^{\cdot+}/1a} = +1.29$ V vs. SCE) and 4CzIPN* ($E_{4CzIPN^*/4CzIPN^{\cdot-}} = +1.35$ V vs. SCE)¹⁴ generated 4CzIPN^{·-} and radical cation **I**, which underwent rapid homolytic cleavage into carbon radical **II** and quinazolinone **III** under basic conditions. Carbon radical **II** was subsequently captured by allylic peroxide to form open-shell intermediate **IV**. An intramolecular radical homolytic substitution (SH₂) on the oxygen atom of **IV** furnished target oxirane **1**, alongside the release of *t*-BuO[·] radical intermediate **V**. Finally, single-electron reduction of the *t*-BuO[·] radical to *t*-BuO⁻ by 4CzIPN^{·-} completed the catalytic cycle and regenerated the ground state of the photocatalyst. Meanwhile, a *t*-BuO[·]



Lin and M. Zhou developed the catalytic method. Z. Li, J. Duan, X. Liu and Y. Wang studied the substrate scope. All the authors were involved the analysis of results and discussions of the project.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: experimental procedures, characterization data, and nuclear magnetic resonance spectra. See DOI: <https://doi.org/10.1039/d6sc00898d>.

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