



## Recent investigations into direct epoxyacylation of alkenes

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### ABSTRACT

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This review highlights recent progress in the direct synthesis of  $\alpha,\beta$ -epoxy ketones via oxidative coupling of alkenes and aldehydes, with a particular focus on mechanistic features of the reactions. For clarity, the review is organized into two sections: metal-catalyzed and metal-free epoxyacylations, covering literature up to the end of May 2025.

## 1. Introduction

Epoxides play a vital role in chemistry, functioning not only as versatile intermediates but also as fundamental building blocks in a wide range of synthetic transformations [1, 2]. Their unique reactivity makes them indispensable in constructing complex molecules, which has driven their widespread application across diverse industries, including pharmaceuticals, agrochemicals, and advanced materials [3-5].  $\alpha,\beta$ -Epoxy ketones are among the most important members of this family of compounds, serving not only as key precursors to numerous valuable compounds but also being present in various natural products, drug-like molecules, as well as FDA-approved drugs (Scheme 1a) [6].

Alkenes constitute one of the most fundamental and versatile classes of unsaturated compounds in organic chemistry. Owing to their abundance, structural diversity, and inherent reactivity toward both electrophilic and

radical species, they serve as indispensable building blocks for numerous functionalization and difunctionalization transformations [7, 8].

Owing to their wide importance across diverse fields, a variety of synthetic approaches have been developed for their preparation [9-11]. Despite their significance, the majority of reported syntheses of  $\alpha,\beta$ -epoxy ketones still rely on a two-step process involving intermediate workup: first, the generation of  $\alpha,\beta$ -unsaturated ketones, followed by their epoxidation using hydroperoxides under basic conditions [12, 13].

Therefore, the development of direct and one-pot methods for synthesizing  $\alpha,\beta$ -epoxy ketones from readily available starting materials is highly desirable, as it simplifies the synthetic route, enhances efficiency, and minimizes waste generation.

Recently, the direct epoxyacylation of alkenes with aldehydes has emerged as a highly promising

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approach for synthesizing  $\alpha,\beta$ -epoxy ketones, benefiting from readily available simple substrates, high atom and step economy, and operational simplicity (Figure 1). Conceptually, direct epoxyacylation can be categorized within the broader class of alkene difunctionalization reactions, as exemplified by recent studies on alkoxyulfenylation processes, which illustrate the potential of concurrent C–C and C–X bond formation [14]. To the best of our knowledge, this methodology has not been reviewed thus far. Therefore, herein, we present a detailed overview of recent advances in the direct epoxyacylation of alkenes with aldehydes, with an emphasis on the reaction mechanisms. The review is organized into two main sections: the first concentrates on metal-catalyzed reactions, and the second addresses metal-free reactions.

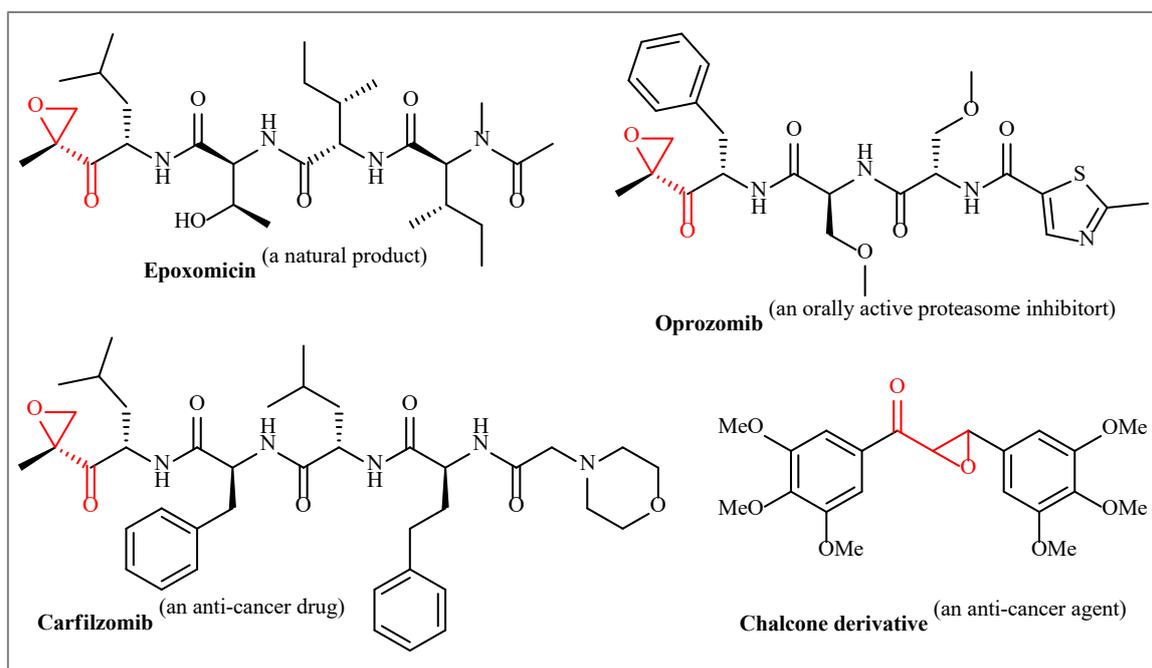
## 2. Metal-catalyzed epoxyacylations

Drawing inspiration from the pioneering work by Li and co-workers on the iron-catalyzed direct carbonylative peroxidation of olefinic double bonds using aldehydes

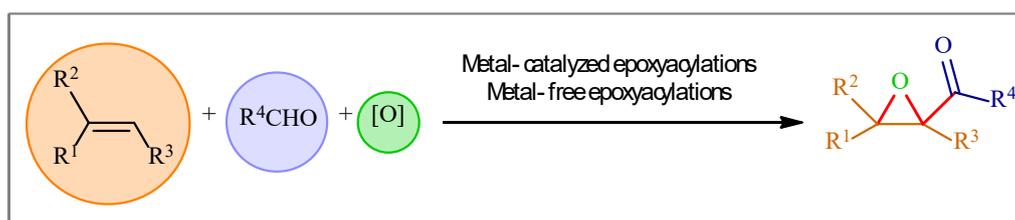
and hydroperoxides as sources of carbonyl and peroxide groups, respectively [15, 16], the first general method for the synthesis of  $\alpha,\beta$ -epoxy ketones *via* direct epoxyacylation of alkenes was reported in 2015 by Li and Wang [17]. By employing 4-chlorostyrene and benzaldehyde as model substrates,  $\text{Ru}(\text{bpy})_3\text{Cl}_2$  as the photocatalyst, and a 45 W household bulb as the visible light source, a range of oxidants, bases, additives, and solvents were carefully screened. Ultimately, the combination of  $\text{Ru}(\text{bpy})_3\text{Cl}_2$ ,  $\text{Cs}_2\text{CO}_3$ , and 4 Å MS was identified as the most effective catalytic system, with *tert*-butyl hydroperoxide (*t*BuOOH) and MeCN selected as the optimal oxidant and solvent, respectively. Under the optimized conditions, various styrene derivatives 1 smoothly engaged with a series of (hetero)aromatic aldehydes 2 by an oxidative coupling process to generate desired  $\alpha,\beta$ -epoxy ketones 3 in moderate to good yields (Scheme 2).

Interestingly, the reaction efficiency was not significantly influenced by the electronic or steric effects of substituents on the aromatic rings.

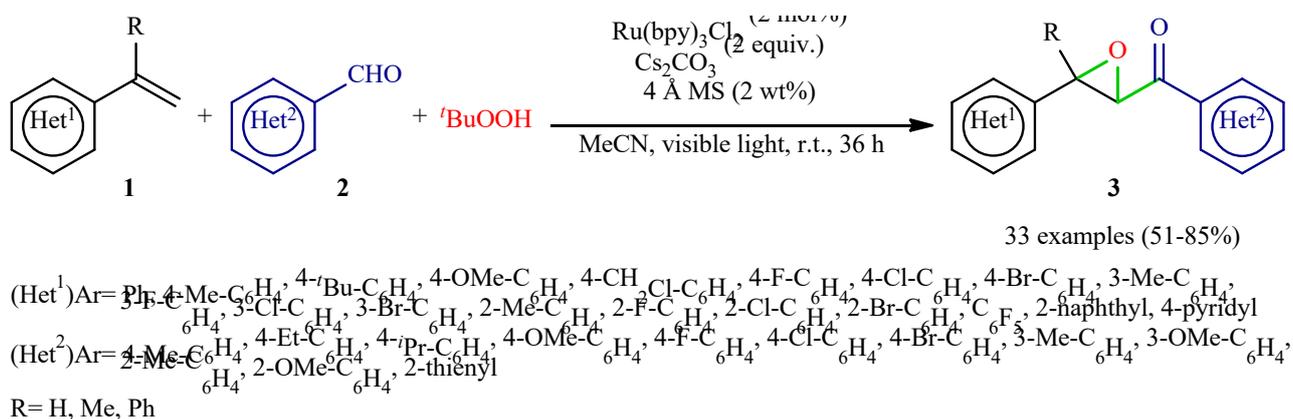
As a result, both coupling partners bearing a wide range of electron-donating or electron-withdrawing groups at the *ortho*-, *meta*-, or *para*-positions of the aryl rings were well tolerated in this transformation.



**Scheme 1.** Selected examples of natural products and synthetic pharmaceuticals containing an  $\alpha,\beta$ -epoxy ketone moiety.



**Fig. 1.** Oxidative coupling of alkenes with aldehydes.



**Scheme 2.** Ru-catalyzed synthesis of  $\alpha,\beta$ -epoxy ketones 3 from alkenes 1, aldehydes 2, and  $t$ BuOOH.

Remarkably, the reaction also accommodated 1,1-disubstituted alkenes as well as alkenes bearing heteroatoms or perfluorinated aryl groups. Furthermore, a gram-scale synthesis of  $\alpha,\beta$ -epoxy ketones was successfully carried out under the standard reaction conditions, affording the desired product in 66% yield (1.1 g) without effect on the reaction outcome. A plausible mechanism to this epoxyacylation is shown in Scheme 3: Initially, the ground state photocatalyst Ru<sup>II</sup> undergoes photoexcitation under visible-light irradiation to produce the excited state [Ru<sup>II</sup>]\*.

This excited species then transfers an electron to  $t$ BuOOH, promoting the cleavage of its weak O–O bond to generate a hydroxyl anion (HO<sup>−</sup>) and *tert*-butoxy radical ( $t$ BuO<sup>•</sup>). The  $t$ BuO<sup>•</sup> radical then abstracts a hydrogen atom from aldehyde 2 to generate the key acyl radical A, which can be captured by alkene 1 to form benzyl radical intermediate B. Meanwhile, HO<sup>−</sup> abstracts a proton from another molecule of  $t$ BuOOH to generate  $t$ BuOO<sup>−</sup>, that after a single-electron-transfer (SET) process provides  $t$ BuOO<sup>•</sup>.

Afterwards, a radical–radical coupling between the  $t$ BuOO<sup>•</sup> and benzyl radical intermediate B affords the  $\beta$ -peroxy ketone C. Finally, in the presence of base, intermediate C undergoes elimination of  $t$ BuOH to produce the observed  $\alpha,\beta$ -epoxy ketone product 3.

Shortly thereafter, the Tang laboratory designed and synthesized a series of Fe-, Co-, Ni-, and Cu-doped zeolite X catalysts (Fe-/Co-/Ni-/Cu-X) with Lewis basic sites, which were applied as efficient catalysts for the oxidative coupling of simple styrene with benzaldehyde [18].

Interestingly, all the metal-doped zeolite catalysts exhibited higher activity than the single basic zeolite X catalyst, affording the target product in yields of 84%, 90%, 93%, and 75%, respectively.

Although only a single example was reported, this study represents the first demonstration of the utility of functional mesoporous materials as catalysts in the direct epoxyacylation of alkenes and may serve as an inspiration

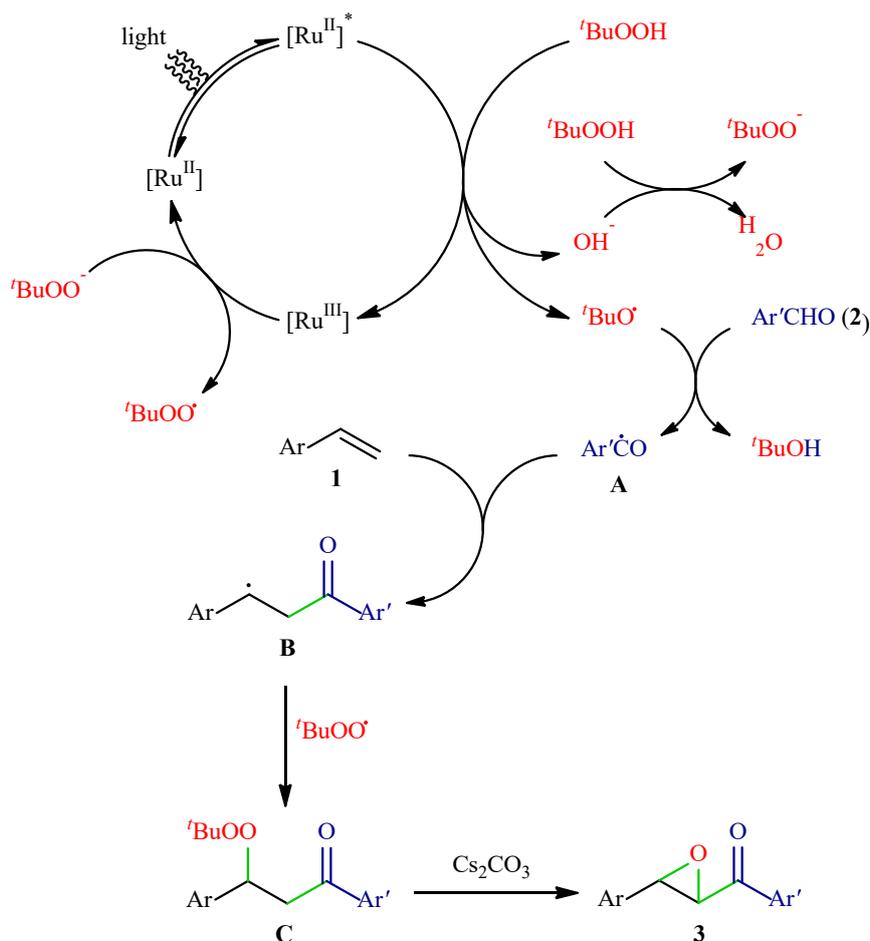
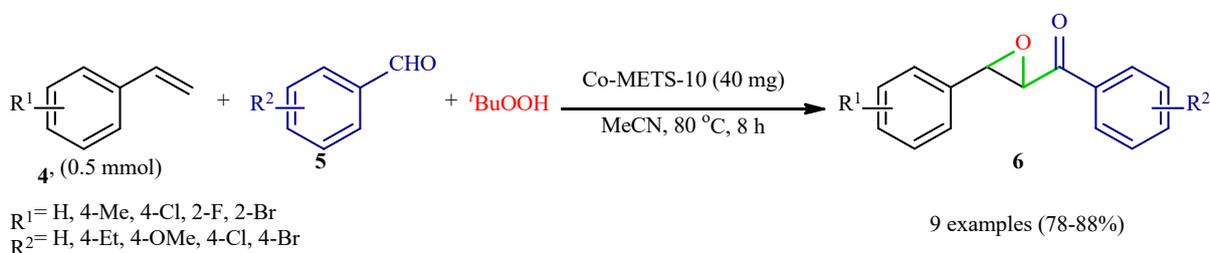
for further research in this area. Subsequently, this innovative research group disclosed the usefulness of bi-functional Co-containing zeolite ETS-10 (Co-METS-10) as the heterogeneous catalyst for the same oxidative coupling reaction [19].

Thus, in the presence of a catalytic amount of Co-METS-10, three component reaction of a series of styrene derivatives 4, aromatic aldehydes 5, and  $t$ BuOOH furnished the corresponding  $\alpha,\beta$ -epoxy ketones 6 in good to high yields, ranging from 78% to 88% (Scheme 4). Unfortunately, the authors did not provide any information regarding the recovery and reusability of the catalyst, nor the applicability of  $\alpha$ - and  $\beta$ -substituted styrenes as starting materials in their methodology.

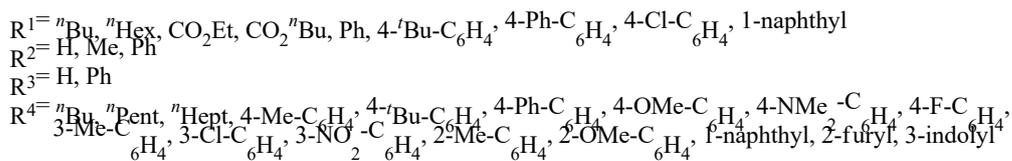
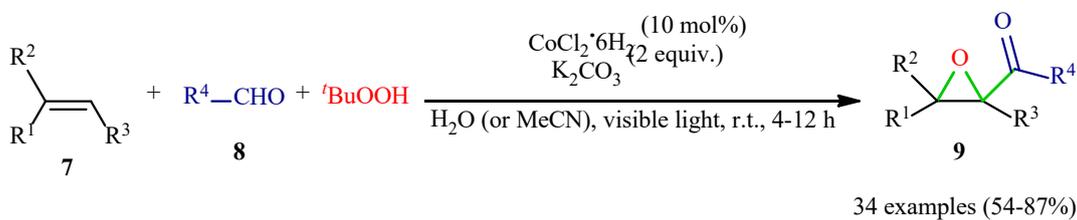
Very recently, with the objective of developing a more general and practical methodology for the synthesis of  $\alpha,\beta$ -epoxy ketones through direct epoxyacylation of the respective alkenes, Thakur and co-workers were able to demonstrate that a diverse range of highly substituted  $\alpha,\beta$ -epoxy ketones 9 could be obtained in modest to high yields from the reaction of alkenes 7 (terminal and internal) with aldehydes 8 (aromatic and aliphatic) and  $t$ BuOOH employing relatively cheap commercially available CoCl<sub>2</sub>·6H<sub>2</sub>O as a catalyst and K<sub>2</sub>CO<sub>3</sub> as the base in the most environmentally friendly solvent, water, at room temperature [20].

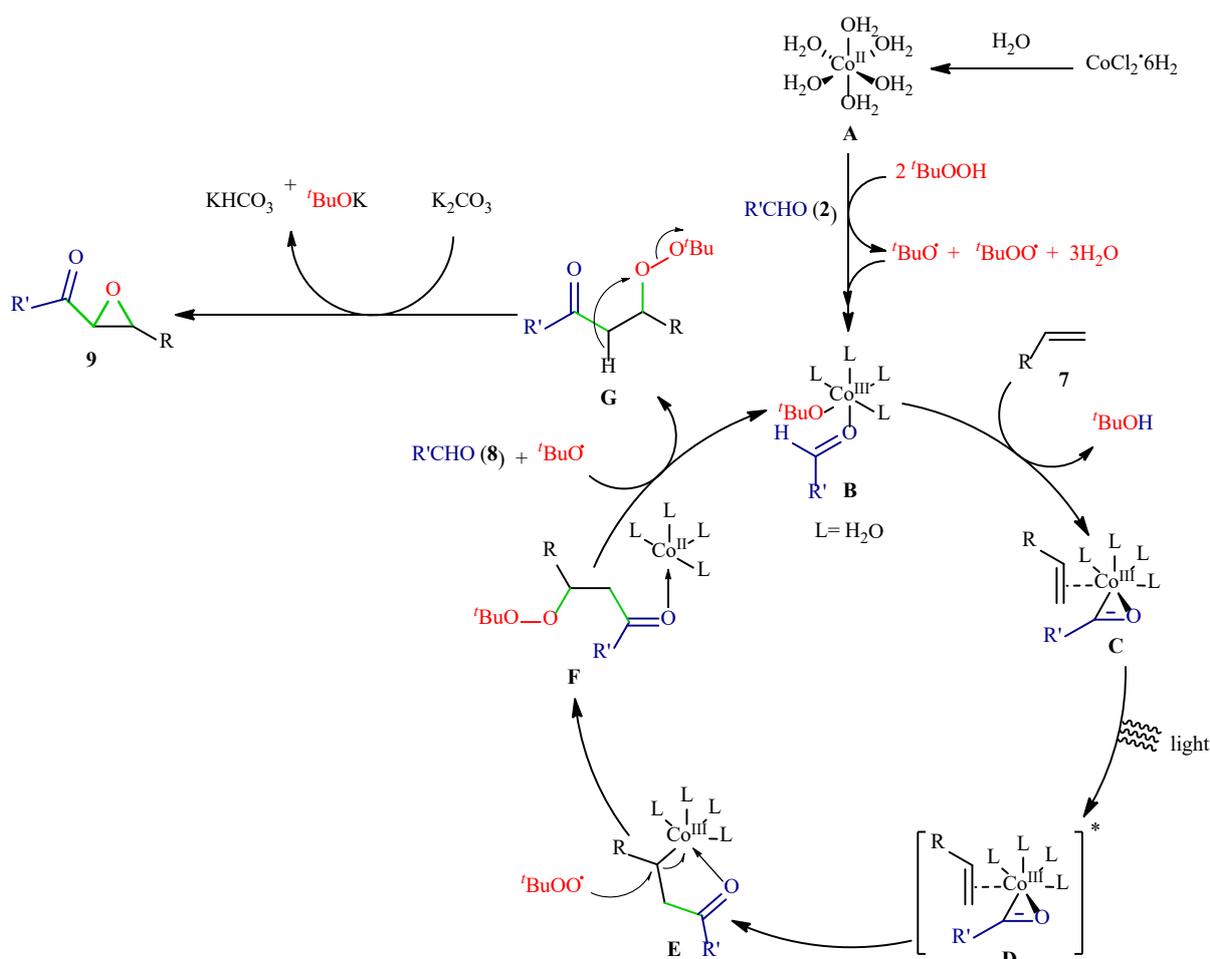
As shown in Scheme 5, a broad range of aromatic and aliphatic alkenes as well as  $\alpha,\beta$ -unsaturated carbonyl compounds along with (hetero)aromatic and aliphatic aldehydes, were employed to demonstrate the general applicability of this synthetic strategy.

In addition, three scale-up experiments (1 g) and several postsynthetic modifications of the prepared compounds using known reactions further highlighted the practical utility of this methodology. Based on the several control experiments, such as radical trapping studies, intermediate detection by GC-MS, Hammett analysis, and DFT calculation, the authors proposed a plausible mechanistic cycle for the reaction as illustrated in Scheme 6.

Scheme 3. Mechanistic proposal for the formation of  $\alpha,\beta$ -epoxy ketones 3.

Scheme 4. Oxidative coupling of styrenes 4 with aldehydes 5 catalyzed by Co-METS-10.

Scheme 5. Thakur's synthesis of  $\alpha,\beta$ -epoxy ketones 9.



**Scheme 6.** Mechanistic proposal for the formation of  $\alpha,\beta$ -epoxy ketones **9**.

### 3. Metal-free epoxyacylations

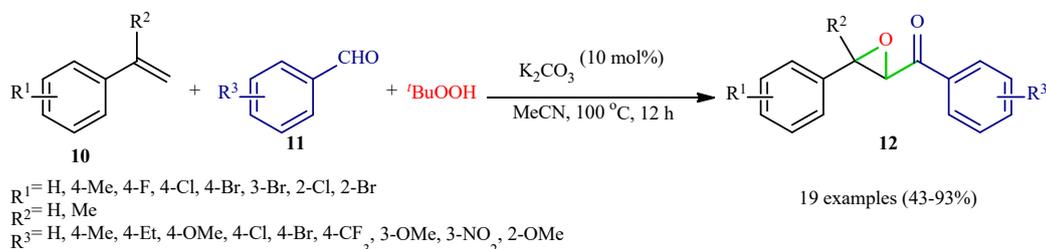
In 2015, Ke *et al.* disclosed for the first time the possibility of synthesizing  $\alpha,\beta$ -epoxy ketones *via* direct epoxyacylation of alkenes under metal-free conditions [21]. They revealed that the treatment of aromatic alkenes **10** with 2 equiv. of aromatic aldehydes **11** and  $t\text{-BuOOH}$  in the presence of a catalytic amount of  $\text{K}_2\text{CO}_3$  in MeCN at  $100^\circ\text{C}$  resulted in the corresponding  $\alpha,\beta$ -epoxy ketones **12** in fair to excellent yields within 12 h (Scheme 7). Regarding the substrate scope, both electron-rich and electron-deficient substituents on either coupling partner had only a minor influence on the reaction outcome. As a result, a wide variety of functional groups were well tolerated under the reaction conditions. However, aliphatic alkenes were unfortunately found to be unsuitable substrates for this reaction. In addition, hexanal exhibited poor reactivity, and as a result, no other aliphatic aldehydes were evaluated under the reported conditions. It is worth noting that the specified reaction temperature is critical, as both lower and higher temperatures resulted in a dramatic decrease in yield. Additionally, the choice of base plays a crucial role in the reaction outcome. Replacing  $\text{K}_2\text{CO}_3$  with other bases such as NaOH, KOH,  $\text{Na}_2\text{CO}_3$ , or  $\text{Cs}_2\text{CO}_3$  led to significantly lower yields or even complete suppression

of the desired product formation. Mechanistically, the authors proposed that this epoxyacylation reaction proceeds through the following key steps (Scheme 8): (i) thermal decomposition of  $t\text{-BuOOH}$  to generate  $t\text{-BuO}^\bullet$  and  $\text{HO}^\bullet$  radicals; (ii) abstraction of a hydrogen atom from the aldehyde **11** by  $t\text{-BuO}^\bullet$  to form the corresponding acyl radical **A**; (iii) addition of acyl radical **A** to alkene **10** to afford a new carbon-centered radical intermediate **B**; (iv) capture of  $t\text{-BuOO}^\bullet$  by radical **B** to generate  $\beta$ -peroxy ketone **C**; and (v) intramolecular cyclization of  $\beta$ -peroxy ketone **C** *via* base-mediated elimination of  $t\text{-BuOH}$  to produce the observed  $\alpha,\beta$ -epoxy ketones **12**.

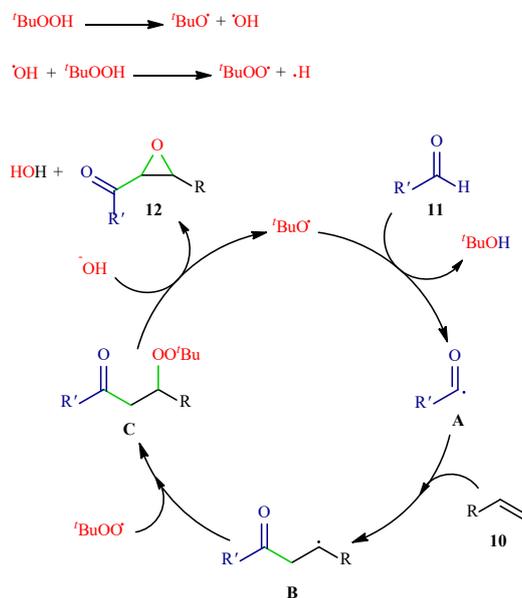
Almost concurrently, in a closely related investigation, Li and colleagues reported the conversion of a series of terminal alkenes **13** into the corresponding  $\alpha,\beta$ -epoxy ketones **15** through oxidative coupling with various aliphatic and aromatic aldehydes **14**, using a catalytic amount of  $t\text{-BuOK}$  in refluxing MeCN (22 examples with average yield of 71%) [22]. As shown in Scheme 9a, various aliphatic, aromatic, and heteroaromatic alkenes with either electron-donating or electron-withdrawing substituents were well tolerated under these reaction conditions. The observed order of reactivity was: aromatic alkenes  $\geq$  aliphatic alkenes  $>$  heteroaromatic alkenes. It is worth mentioning that this

approach demonstrated superior yields compared to the methodology developed by Ke's group, highlighting the significant impact of the base choice. In addition to the choice of base, the nature of the solvent also played a critical role in the reaction outcome. For instance, conducting the reaction in toluene, DMSO, or 1,4-dioxane resulted in very low (5-10%) to negligible product formation. In the same year, Tang and co-

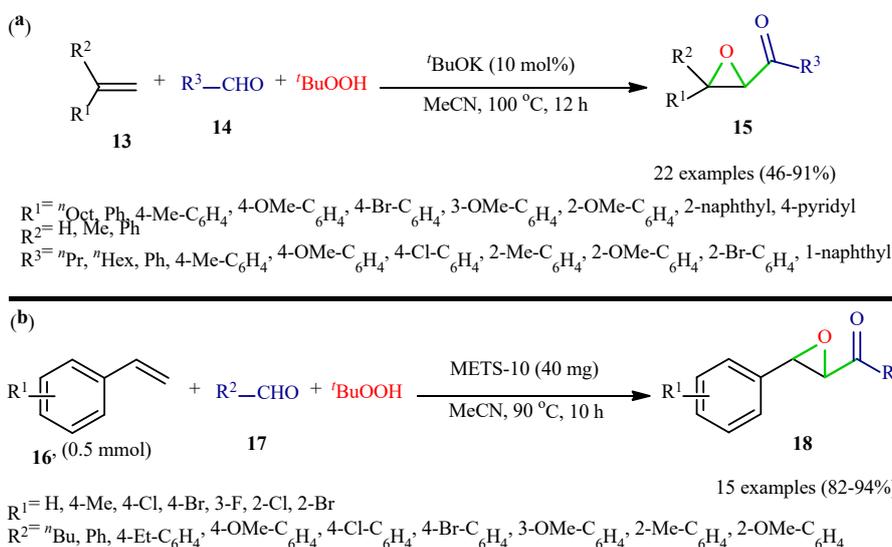
workers published their result on a mesoporous Zeolite ETS-10 (METS-10) catalyzed direct epoxyacylation alkene substrates [23]. They demonstrated that styrene derivatives **16** underwent oxidative coupling with various aliphatic and aromatic aldehydes **17** using <sup>t</sup>BuOOH as an oxidant and a catalytic amount of heterogeneous METS-10 to form corresponding  $\alpha,\beta$ -epoxy ketones **18** in high to excellent yields within 10 h (Scheme 9b).



**Scheme 7.** Ke's synthesis of  $\alpha,\beta$ -epoxy ketones **12**.



**Scheme 8.** Plausible mechanistic cycle for the formation of  $\alpha,\beta$ -epoxy ketones **12**.

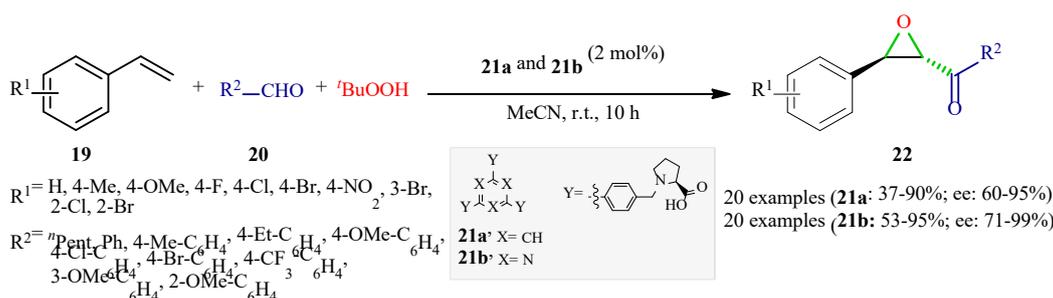


**Scheme 9.** (a) Li's synthesis of  $\alpha,\beta$ -epoxy ketones **15**; (b) Tang's synthesis of  $\alpha,\beta$ -epoxy ketones **18**.

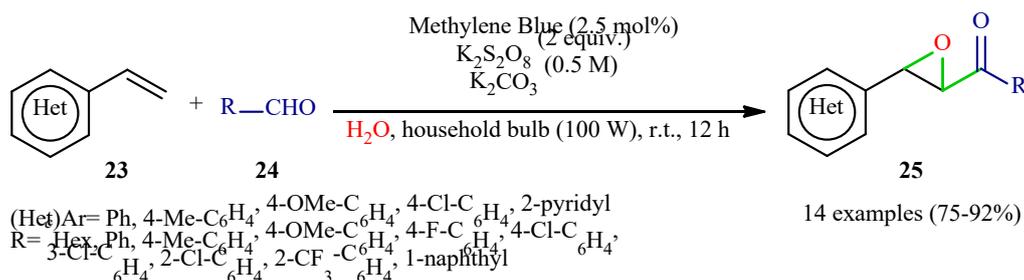
Two years later, Ashokkumar and Siva extended this chemistry to the enantioselective synthesis of  $\alpha,\beta$ -epoxy ketones by employing two proline based chiral C3-symmetric organocatalysts, **21a** and **21b** [24]. Their approach enabled the efficient synthesis of a panel of 20 chiral epoxy ketones **22** in moderate to excellent yields and with high enantioselectivity from the corresponding alkenes **19** and aldehydes **20** under ambient conditions (Scheme 10). While both catalysts demonstrated effective catalytic performance, catalyst **21b** proved to be superior to **21a** with respect to both product yield and enantioselectivity.

In 2018, Souza *et al.* reported an interesting visible-light-induced epoxyacylation of terminal alkenes with aldehydes using water as the reaction medium and the oxygen source [25]. The transformation was conducted under 100 W visible light irradiation at room temperature, employing a catalytic system composed of methylene blue (MB),  $K_2S_2O_8$  and  $K_2CO_3$ . The protocol demonstrated relatively broad substrate scope, accommodating various (hetero)aromatic alkenes **23** and both aromatic and aliphatic aldehydes **24**, and efficiently afforded the desired  $\alpha,\beta$ -epoxy ketones **25** in good to excellent isolated yields within 12 h (Scheme 11). Notably, the reaction was also readily scalable to the gram scale, delivering high yields (up to 84%) when conducted under sunlight irradiation for 6 hours. Interestingly, when nonconjugated alkenes were subjected to the same reaction conditions, the reaction proceeded *via* a hydroacylation pathway, yielding the corresponding ketones as the sole products. Mechanistic investigations using radical scavengers suggested the involvement of a radical pathway in this epoxyacylation reaction. Based on control experiments and prior studies, particularly considering that persulfate rapidly

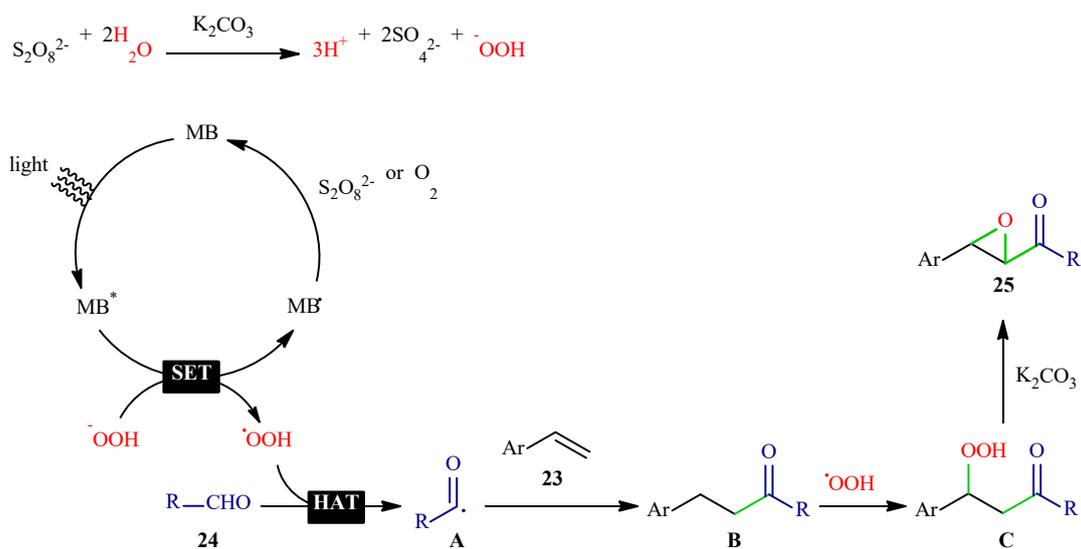
decomposes into hydroperoxide anion and sulfate under basic conditions, the authors proposed a catalytic mechanism for the formation of  $\alpha,\beta$ -epoxy ketones, as illustrated in Scheme 12. In a significant contribution in this field, Sudalai's research team developed a robust and unprecedented regioselective oxidative coupling of alkenes with aldehydes for the synthesis of  $\alpha,\beta$ -epoxy ketones employing DMSO as both the solvent and the oxygen source [26]. The authors identified a combination of *N*-heterocyclic carbene (NHC-1), NBS, and DBU as the optimal system for this transformation. Under these conditions, eleven  $\alpha,\beta$ -epoxy ketones **28** were synthesized in moderate to good yields *via* the reaction of corresponding styrene derivatives **26** with aldehydes **27** at room temperature (Scheme 13). While the method demonstrated a relatively broad scope with respect to the aldehyde component, it showed limited substrate tolerance for alkenes. Unfortunately, the reaction proved ineffective for internal alkenes and terminal disubstituted alkenes. Furthermore, aliphatic alkenes exhibited low reactivity under the optimized conditions, making them poorly suited for this protocol. In the proposed mechanistic pathway (Scheme 14), the authors suggested that this epoxyacylation reaction proceeds through the following key steps: (i) reaction of styrene **26** with NBS to form bromonium ion **A**; (ii) regioselective ring opening of **A** by DMSO to generate the cationic intermediate **B**; (iii) elimination of Me<sub>2</sub>S from **B** to afford phenacyl bromide **C**; (iv) reaction of NHC-1 with **C** to form the intermediate salt **D**; (v) elimination of HBr from **D** to yield the ketodeoxy Breslow intermediate **E**; (vi) nucleophilic addition of **E** to aldehyde **27** to give intermediate **F**; and (vii) release of the NHC catalyst from **F** to afford the thermodynamically stable *trans*- $\alpha,\beta$ -epoxy ketones **28**.



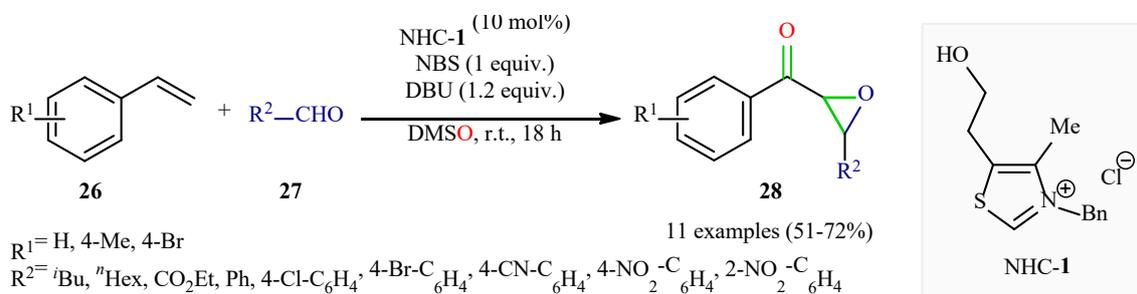
Scheme 10. Ashokkumar-Siva's synthesis of chiral  $\alpha,\beta$ -epoxy ketones **22**.



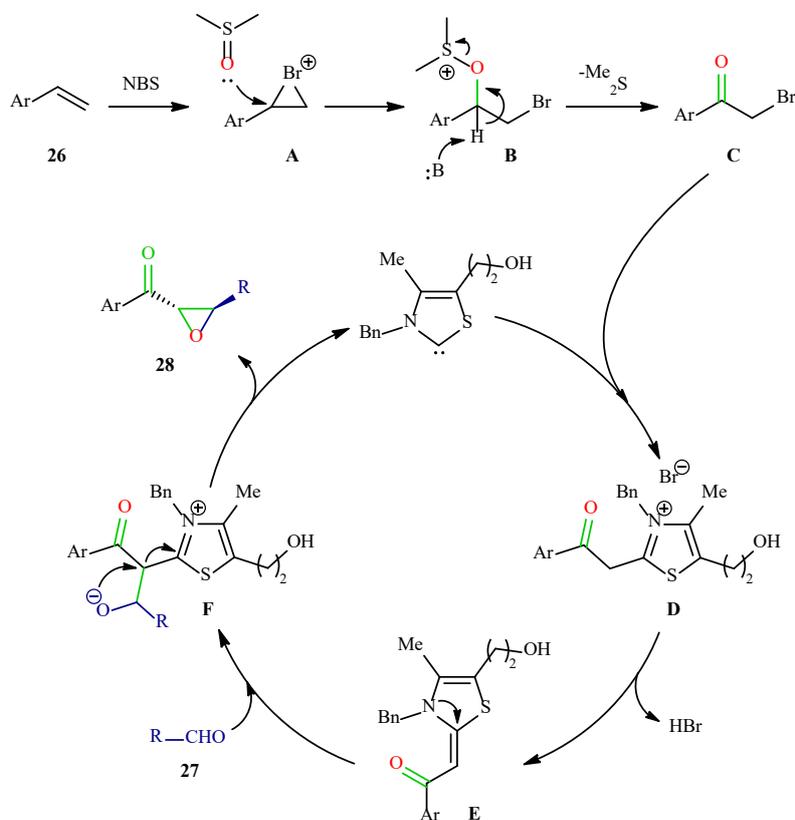
Scheme 11. Souza's synthesis of  $\alpha,\beta$ -epoxy ketones **25**.



**Scheme 12.** Plausible mechanism for the formation of  $\alpha,\beta$ -epoxy ketones 25.



**Scheme 13.** Sudalai's synthesis of  $\alpha,\beta$ -epoxy ketones 28.



**Scheme 14.** Proposed mechanistic pathway for the formation of  $\alpha,\beta$ -epoxy ketones 28.

#### 4. Conclusion

This review highlights that direct epoxyacylation of alkene substrates with aldehydes has received considerable attention in recent years, evolving into a versatile methodology that enables efficient access to  $\alpha,\beta$ -epoxy ketones with substantial synthetic and biological significance. The widespread availability and structural diversity of the starting materials, combined with the mild reaction conditions, underscore the scalability and practical potential of these transformations. However, the applicability of other unsaturated compounds, such as allenes and alkynes, has yet to be investigated in these reactions. Moreover, majority of these transformations rely heavily on *t*-BuOOH as the oxidant, which presents certain drawbacks, including flammability, potential explosiveness, and the need for careful handling and storage, factors that may limit its use and particularly in large-scale applications. Looking to the future, we anticipate a rapid expansion of the substrate scope, the development of milder oxidants, and advances in more efficient catalytic systems.

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