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Recent advances in catalytic asymmetric Büchner reaction☆

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ABSTRACT

Büchner reaction, as a unique type of expansive dearomatization, has become a practical strategy for the straightforward assembly of valuable functionalized cycloheptatrienes from ubiquitous aromatic precursors. Although the asymmetric version has been investigated since the early 1990s, enantioselective Büchner reaction is still limited by the catalyst type and substrate scope. This review aims to propose the limitation and possible development direction of this field by summarizing the evolution of catalytic asymmetric Büchner reaction, which is organized on the basis of intra- and intermolecular reactions. Considering the different metal carbene precursors, the reactions are further classified by carbene sources.

1. Introduction

Since Büchner reaction was first reported by Büchner and Curtius in 1885 [1], it has attracted widespread attention due to its privileges in dearomatization reactions and construction of valuable seven-membered carbocycles. Typically, Büchner reaction refers to the cyclopropanation of a benzenoid double bond with metal carbene to generate a norcaradiene intermediate, which subsequently transforms into a more stable cycloheptatriene through electrocyclic rearrangement (Scheme 1). Moreover, this elegant strategy has been successfully applied to the synthesis of natural products [2,3].

The non-enantioselective Büchner reaction has been well-established during the recent decades [4–18], and a series of metal catalysts, such as Rh [4–8,10,11], Cu [12–14], Ru [9], Co [15], Ag [16] and Au [17,18] were screened, which demonstrated different reactivity and selectivity of this ring expansion reaction. Furthermore, besides transition-metal catalysis, some metal-free protocols have also been developed recently [19,20]. However, the asymmetric Büchner reaction was much less explored due to the racemization of products and low chemoselectivity. In 1990, McKervey reported the first example of asymmetric Büchner reaction by employing chiral rhodium(II) carboxylate, affording bicyclic trienone with 33% *ee* [21]. Afterwards in 1992, the same group employed a new rhodium(II) phosphate as the catalyst, which led to 60% *ee* in the case of a biphenyl substrate [22]. Both aforementioned studies only involved a few examples of diazoketone substrates, and high enantioinduction was not obtained. Thus, systematic exploration of the asymmetric Büchner reaction, especially in a highly enantioselective manner, is greatly desired. This review focuses on the recent examples of asymmetric Büchner reactions, and we will summarize these advancements systematically on the basis of intra- and intermolecular reactions. Considering the different metal carbene precursors, the reactions are further classified by carbene sources. The aim of this review is to provide latest and comprehensive introduction of catalytic enantioselective Büchner reaction by highlighting the product diversity, regio- and stereoselectivity as well as the reaction mechanism.

2. Intramolecular asymmetric Büchner reactions

2.1. Diazo compounds as carbene precursors

Bisoxazoline (BOX)-copper(I) catalytic system was first applied to asymmetric cyclopropanation reactions based on diazo compounds and alkenes in 1990 [23], which has become an important strategy in the field of asymmetric catalysis [24–27]. As an extension of the cyclopropanation reaction, Büchner reaction was also chosen as a model reaction to optimize the catalytic conditions of BOX-copper(I) complex.

☆ Dedication to Prof. Lixin Dai on the Occasion of His Centenary Birthday.

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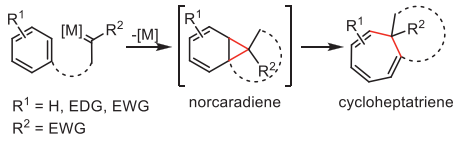
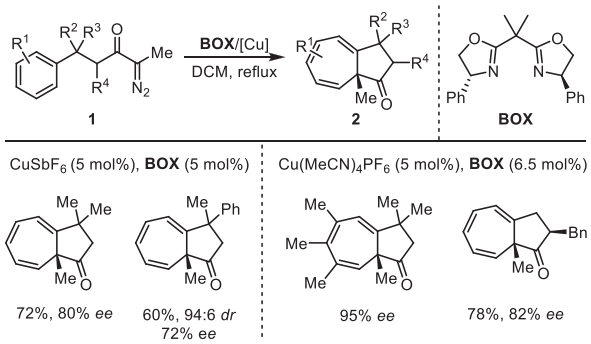
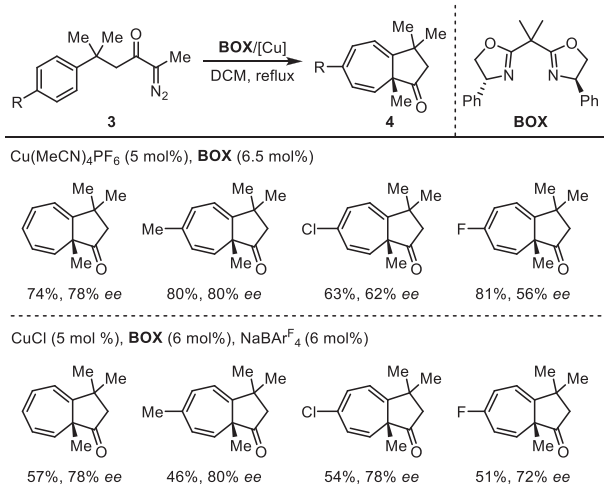


Scheme 1. Büchner reaction.



Scheme 2. Copper-catalyzed asymmetric Büchner reaction based on BOX ligands.



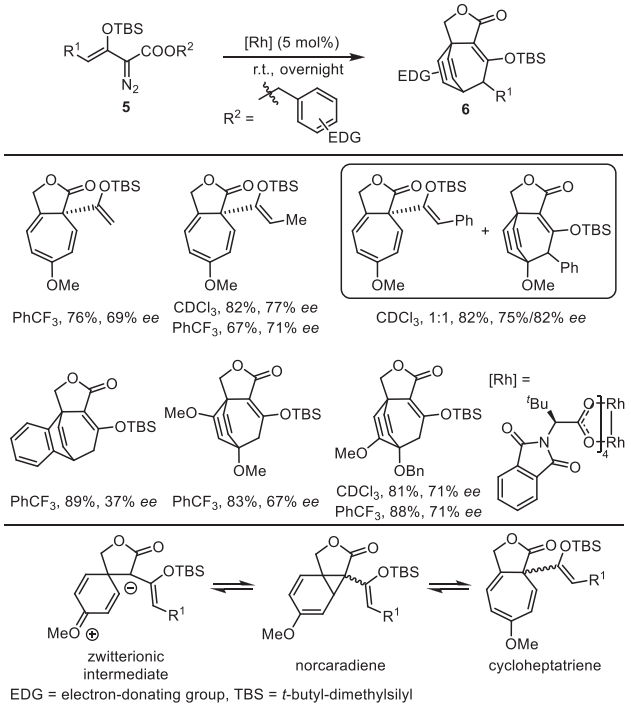
Scheme 3. Enhancement of enantioselectivity in the copper-catalyzed Büchner reaction by variation of the counterion.

In 2007, Maguire group realized the asymmetric Büchner reaction with high enantioselectivities by using copper bisoxazoline complexes, and azulenones **2** were obtained efficiently from α-diazoketones **1**, which were strongly dependent on the catalysts (Scheme 2) [28]. However, some key data, such as yield, diastereoisomer ratio and relative configuration, were not exhibited in individual cases. This work is the first example that the BOX-copper(I) catalytic system participated in Büchner reaction.

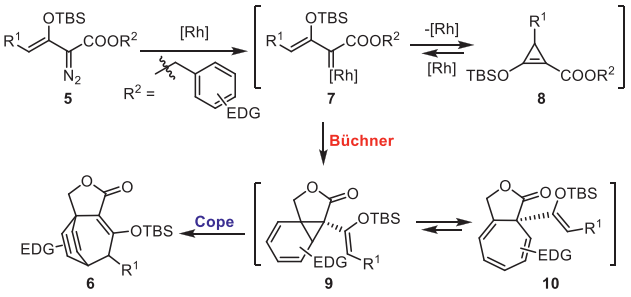
On the basis of the above reports, Maguire and co-workers further evaluated the substrate scope of the asymmetric Büchner reaction, including the electronic effect of the aryl substituents and the nature of the counterions [29]. Four α-diazoketones **3** bearing different electron-donating and -withdrawing groups were tested under two copper-catalyzed conditions, which displayed enhanced enantioselection by the use of $NaBAr^F_4$ as counterion-exchange reagent (Scheme 3). In 2012, Maguire group further demonstrated the key role of the alkali metal cation in this copper-catalyzed asymmetric Büchner reaction in their subsequent work [30]. Then in 2018, the same group achieved the asymmetric Büchner reaction by immobilizing BOX-copper(I) in batch or continuous flow [31]. It was observed that the heterogeneous catalysts could be reused seven times without deterioration in asymmetric catalytic activity, which provided a safe method for the large-scale asymmetric reactions of potentially hazardous reagents like diazo compounds. Although copper-catalyzed asymmetric Büchner reaction with relatively good enantioselectivity was initially reported, it has not been developed more deeply to date, especially in tolerance of substrates. Namely, Büchner reaction was just selected as a model reaction to explore the practicality of BOX ligand and investigate the effect of additive in the BOX-copper(I) catalytic system by Maguire and co-workers. However, the limited scope and stereoselectivity demonstrated here opened up new horizons for the development of catalytic asymmetric Büchner reaction.

Alternatively, dirhodium catalysts have proven to be superior to copper catalysts for the asymmetric Buchner reaction, and significant developments have been achieved in recent years. In 2015, Xu and Doyle reported an interesting asymmetric Büchner reaction of benzyl enol diazoacetates **5** for the synthesis of bicyclo[3.2.2]nonatrienes **6** *via* a dirhodium-catalyzed intramolecular cascade process (Scheme 4) [32]. Of note, no absolute configuration of compound **6** was mentioned in this work, although the enantiomeric excess values were tested.

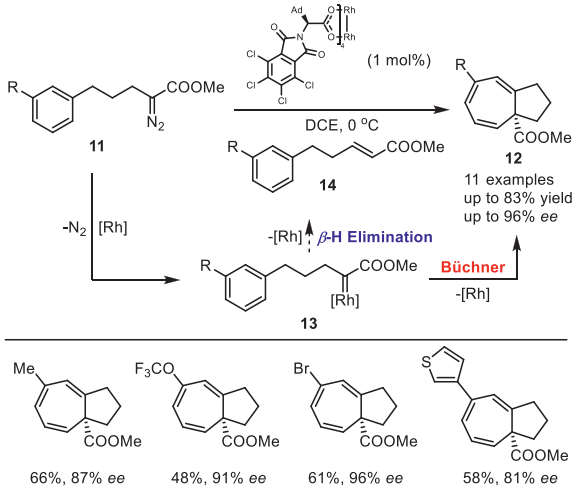
A plausible mechanism involving tandem cyclopropanation/Büchner reaction/Cope rearrangement is shown in Scheme 5.



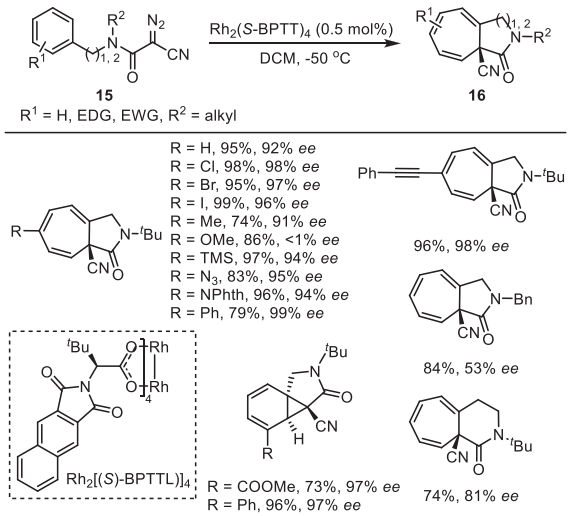
Scheme 4. Rhodium-catalyzed asymmetric tandem reaction of benzyl enol diazoacetates **5**.



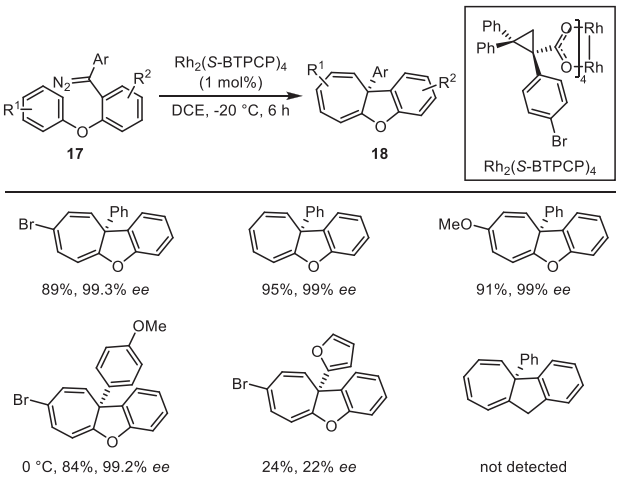
Scheme 5. Plausible mechanism for rhodium-catalyzed asymmetric tandem cyclopropanation/Büchner reaction/Cope rearrangement.



Scheme 6. Rhodium-catalyzed intramolecular asymmetric Büchner reaction for the regioselective synthesis of dihydroazulenes **12**.



Scheme 7. Rhodium-catalyzed intramolecular asymmetric Büchner reaction based on β-cyano α-diazoacetamides **15**.



Scheme 8. Rhodium-catalyzed intramolecular asymmetric Büchner reaction based on diaryl diazomethanes **17**.

The authors, who provided the first discussion of the racemization of norcaradienes **9** *via* isomerization into the corresponding zwitterionic intermediates and cycloheptatriene compounds **10**, pointed out that the generation of the methoxy stabilized zwitterionic intermediates was the most likely reason for the racemization of the cycloheptatrienes. Notably, no loss of enantioselectivity was observed in these cope rearrangement products even under heated conditions.

As an interesting extension of this transformation, the asymmetric Buchner reaction of simpler diazoacetate substrates was developed by using Rh_2(*S*-TCPTAD)$_4$ as catalyst. In 2019, Yamaguchi and co-workers developed a rhodium-catalyzed asymmetric intramolecular Büchner reaction of α-alkyl-α-diazoesters **11** with good chemo-, regio- and enantioselectivity, providing a direct entry to a series of chiral dihydroazulenes **12** (Scheme 6) [33]. Of note, both the substrates bearing meta-substituted phenyl rings and the selection of rhodium catalyst contributed to the minimization of β-hydride elimination, avoiding the generation of 5-phenyl-2-pentenoic acid methyl esters **14** from rhodium carbene intermediate **13**.

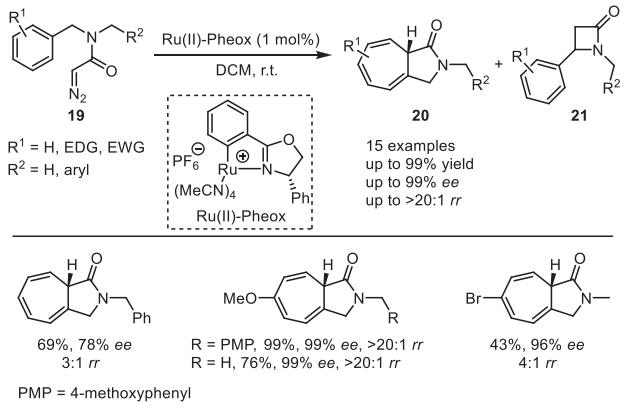
Besides diazoacatate substrates, cyano-substituted diazo derivatives were also found to be suitable substrates for the enantioselective Büchner reaction. In 2021, Darses realized a rhodium-catalyzed asymmetric intramolecular Büchner reaction of benzyl β-cyano α-diazoacetamides **15**, affording the corresponding γ-lactams **16** in excellent yields and enantioselectivities, which bore all-carbon quaternary stereogenic centers and cycloheptatriene skeletons (Scheme 7) [34]. Interestingly, the use of *meta*-substituted substrates only generated norcaradienes without decrease of the enantioselectivities. It worth noting that these products were observed as the mixture of cycloheptatrienes and norcaradienes in their crystalline structures, which was supported by the X-ray analysis and density functional theory (DFT) calculations.

Apart from the electron-withdrawing group containing diazo compounds, relatively less-reported diaryl diazomethanes have also been discovered to be compatible for the enantioselective Büchner reaction. In 2022, Zhu and Chen employed diaryl diazomethanes **17** as the carbene precursors to access a variety of structurally novel polycyclic chiral cycloheptatrienes **18** in excellent yields and enantioselectivities *via* donor-donor rhodium carbene intermediates (Scheme 8) [35]. The reaction showed good compatibility for aromatic rings with different electronic properties. Nevertheless, the CH_2-tethered diazo compound was found to be unreactive for Büchner reaction, only delivering carbene dimerization byproduct.

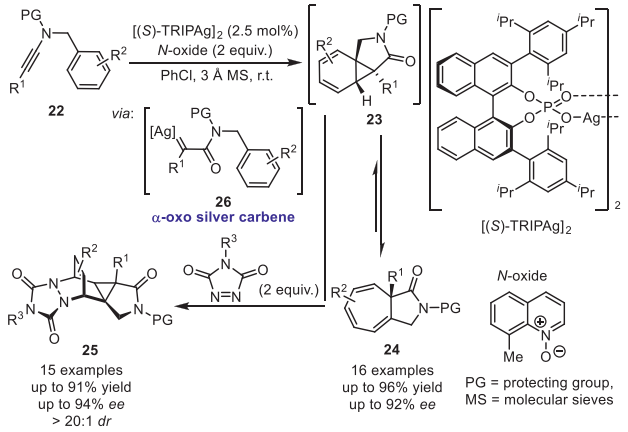
In addition to chiral copper and dirhodium-catalyzed asymmetric intramolecular Büchner reaction, ruthenium complex also exhibited high efficiency in the transformation. In 2019, Iwasa and co-workers utilized chiral ruthenium complex developed by themselves to catalyze an intramolecular asymmetric Büchner reaction, furnishing the corresponding γ-lactam fused 5,7-bicyclic-heptatrienes **20** in excellent yields with excellent enantioselectivities, as demonstrated in Scheme 9 [36]. It should be mentioned that the β-lactams **21** as direct C–H insertion byproducts were unavoidable in some cases.

2.2. Alkynes as carbene precursors (non-diazo method)

During the past decades, the generation of metal carbenes from readily available alkynes represents a significant advance in metal carbene chemistry [37–42]. The replacement of traditional diazo compounds with alkynes allows a safe and convenient pathway for the generation of metal carbenes. The catalytic oxygen transfer reactions of alkynes have been extensively explored in recent years, for the efficient and divergent formation of α-oxo metal



Scheme 9. Ruthenium-catalyzed asymmetric Büchner reaction.

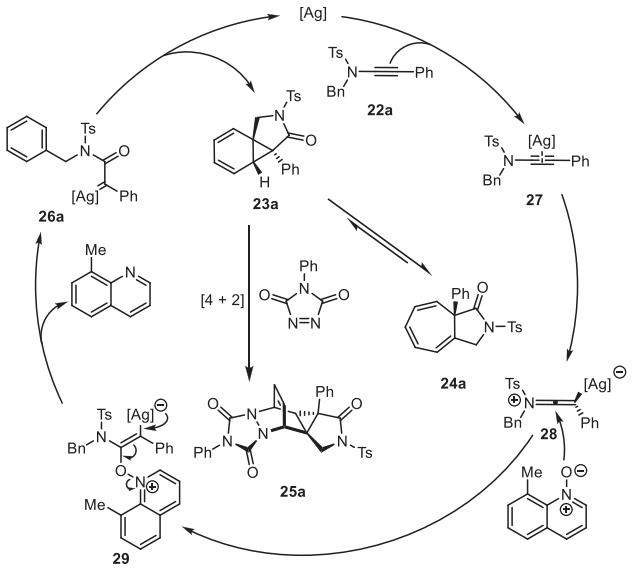


Scheme 10. Silver-catalyzed asymmetric Büchner reaction.

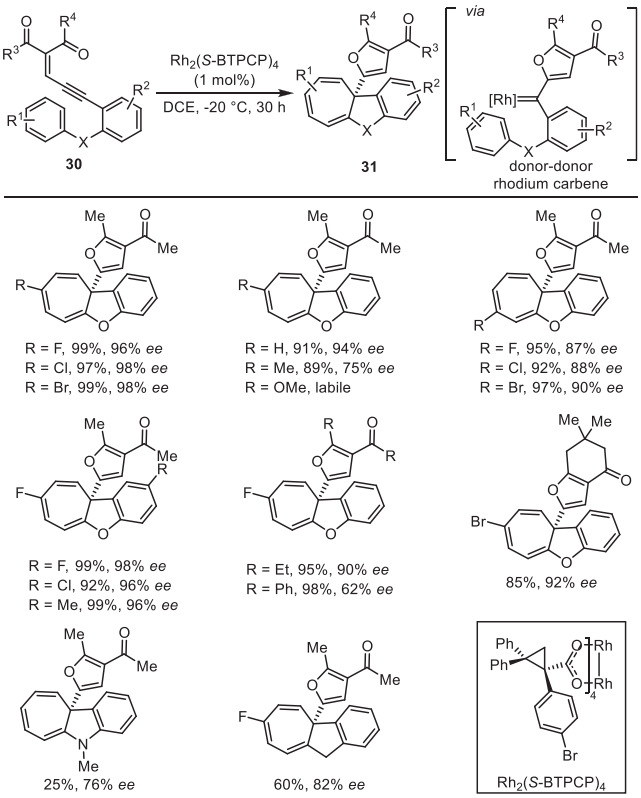
carbene intermediates [43–47]. In 2021, Nemoto and Harada reported the first example of asymmetric Büchner reaction based on non-diazo carbene precursors, as depicted in Scheme 10 [48] Inspired by the transition metal-catalyzed alkyne oxidation reactions *via* α-oxo metal carbene intermediates, ynamides **22** were chosen as the carbene precursors to participate in this reaction catalyzed by chiral phosphoric acid-derived silver salt [49–56]. This strategy not only synthesized a range of chiral cycloheptatrienes **24**, but also afforded polyheterocycles **25** with five consecutive stereogenic centers *via* the intermolecular [4 + 2]-cycloaddition between dienophiles and norcaradiene intermediates **23**, and both **24** and **25** were formed in excellent yields and stereoselectivities, respectively.

The mechanism shown in Scheme 11 was proposed to rationalize the reaction process. First, ynamide **22a** is activated by the silver salt to form the keteneiminium intermediate **28**. Subsequently, intermediate **28** undergoes nucleophilic attack by *N*-oxide to give a vinyl silver intermediate **29**, which can generate α-oxo silver carbene intermediate **26a** upon cleavage of the N–O bond. Finally, silver carbene **26a** is trapped by the phenyl ring to form the norcaradiene **23a**, which will transform into cycloheptatriene **24a** or further react with dienophile to furnish the bridged compound **25a**.

Alternatively, enynone cycloisomerization has been proven as one of the most efficient methods for the generation of metal carbene intermediates *via* non-diazo approach. In the previously described work by Zhu, Chen and co-workers, aryloxy enynones **30** were developed as the carbenes precursors and facilitated the catalytic asymmetric Büchner reaction, for the assembly of di-

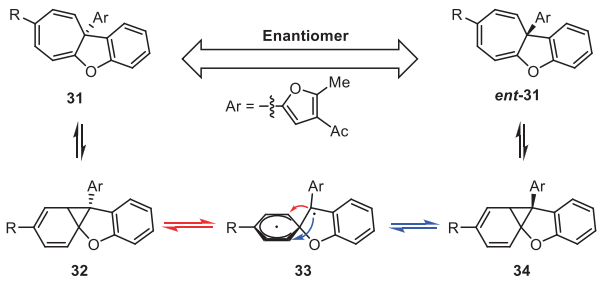


Scheme 11. Plausible mechanism for silver-catalyzed asymmetric Büchner reaction.

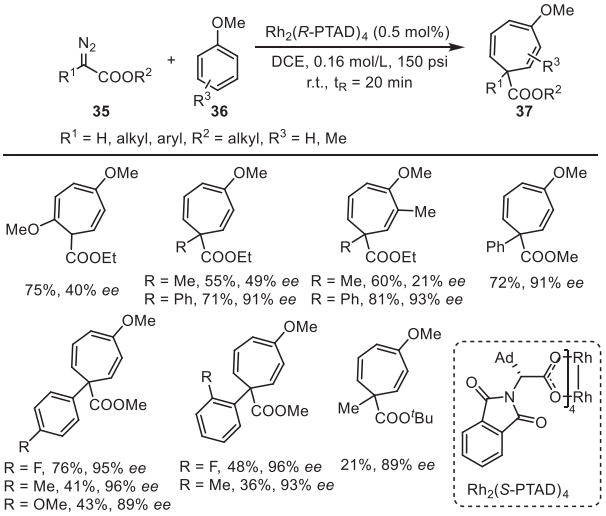


Scheme 12. Rhodium-catalyzed intramolecular asymmetric Büchner reaction based on aryloxyenynones **30**.

verse structurally novel polycyclic chiral cycloheptatrienes **31** in excellent yields and enantioselectivities. Of note, when enynone tethered aryloxy substrates bearing electron-withdrawing groups (R^1 = EWG) were used as carbene precursors in this Rh_2(II)-catalyzed enantioselective intramolecular Büchner reaction, better enantioselectivities could be achieved. It should be pointed out that this report provided a quick access to the relatively less reported donor-donor rhodium carbene intermediates (Scheme 12) [35].



Scheme 13. Probable mechanism for the racemization process.



Scheme 14. Rhodium-catalyzed intermolecular asymmetric Büchner reaction in a continuous flow system.

In addition, the authors observed a slowly decreased enantioselectivity in some special furyl-substituted chiral cyclohepta[*b*]benzofurans **31** generated from the *para*-substituted phenoxy enynones, and a diradical process was proposed as the probable mechanism for the racemization of these compounds (Scheme 13), which was supported by several control experiments.

3. Intermolecular asymmetric Büchner reactions

Although significant advancements have been achieved in asymmetric Büchner reaction, these transformations were limited to intramolecular manner for long time, until the introduction of flow conditions. In 2017, Beeler realized the first example of intermolecular asymmetric Büchner reaction by the Rh-catalyzed reaction of α-diazoacetates **35** with anisole derivatives **36**, which was conducted in flow with high regioselectivity and enantioselectivity (Scheme 14) [57]. The position of ring expansion tends to occur away from the substituent group of the arene substrates, and the regioselectivity was highly dependent on the flow rate and residence time of the continuous flow system. This work provided a novel method for the catalytic asymmetric dearomatization (CADA) reactions of non-activated simple phenyl rings. It should be pointed out that, this report by Beeler and co-workers is the only example of intermolecular asymmetric Büchner reaction till now. The inherent challenges include largely excess amounts of arenes, limited substrates, and problematic regioselectivity. We anticipate that further improvement based on better catalytic systems or other new technologies will expand the scope of intermolecular asymmetric Büchner reaction.

4. Conclusions and future outlook

In summary, catalytic asymmetric Büchner reaction has experienced significant progress since the 21st century, and various transition-metal-catalyzed transformations of distinct carbene precursors have been successively developed. Many advancements, including intermolecular and intramolecular reactions, new catalytic systems, new carbene precursors as well as flow conditions have been developed, and some intrinsic limitations have been overcome owing to these efforts. For example, the rhodium catalysts were found to be more efficient compared with initially developed copper catalysts; intermolecular asymmetric Büchner reaction was realized using continuous flow system; alkynes have been developed as new carbene precursors. In addition, the causes of racemization of some special Büchner products have been systematically explained by the combination of control experiments and DFT calculations.

Despite these considerable achievements, there is still space for further exploration that includes but is not limited to the following: (a) In particular, there are obvious limitations in the tolerance of functional groups on the reactive aromatic rings especially electron-rich ones, and low enantioselectivities or chemoselectivities are observed in some cases. It's anticipated that a deep and systematic mechanistic understanding of asymmetric Büchner reaction could guide the exploration of more efficient catalyst systems. (b) Although the mechanism of racemization of some special Büchner products has been explained reasonably, there is still a lack of effective method to avoid the phenomenon especially during the reaction. The research for the development of other carbene precursors that lead to divergent synthesis of stable chiral Büchner products is highly desirable. (c) Novel catalytic systems involving non-noble metal catalysts or non-diazo carbene precursors need to be further explored. There is enough reason to believe that research on this remarkable area will attract more organic chemists to devote endeavors to this unique CADA [58–63] and make it more important both academically and industrially.

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.

Acknowledgments

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