

## CHAPTER 4

# Decarboxylative C-H Activation Reactions: A Modern Synthetic Strategy Towards Sustainable Development

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**Abstract:** Transition metal-catalyzed decarboxylative C-H activation reactions have appeared as a powerful and efficient strategy for the selective functionalization of C-H bonds which offers a promising route to sustainable chemical transformation. This method couples the decarboxylation of carboxylic acids with the simultaneous activation of inert carbon-hydrogen bonds which enables the direct incorporation of functional groups into organic scaffolds without the need for pre-functionalized activated substrates. The application of metal catalysts, including palladium, nickel, silver, rhodium and copper in this approach has made the process efficient and work selectively under benign conditions with nominal waste generation. Furthermore, the use of carboxylic acids, which are readily available, structurally diverse, cost-effective, air- and moisture-stable, as well as easy to store and handle, has recognized this approach as a valuable tool for promoting sustainable developments in chemical synthesis. Therefore, this modern decarboxylative strategy has revealed significant potential for the construction of complex molecular frameworks, including pharmaceuticals, bioactive compounds and cutting-edge materials. This review emphasizes on various proficient decarboxylative carbon-hydrogen

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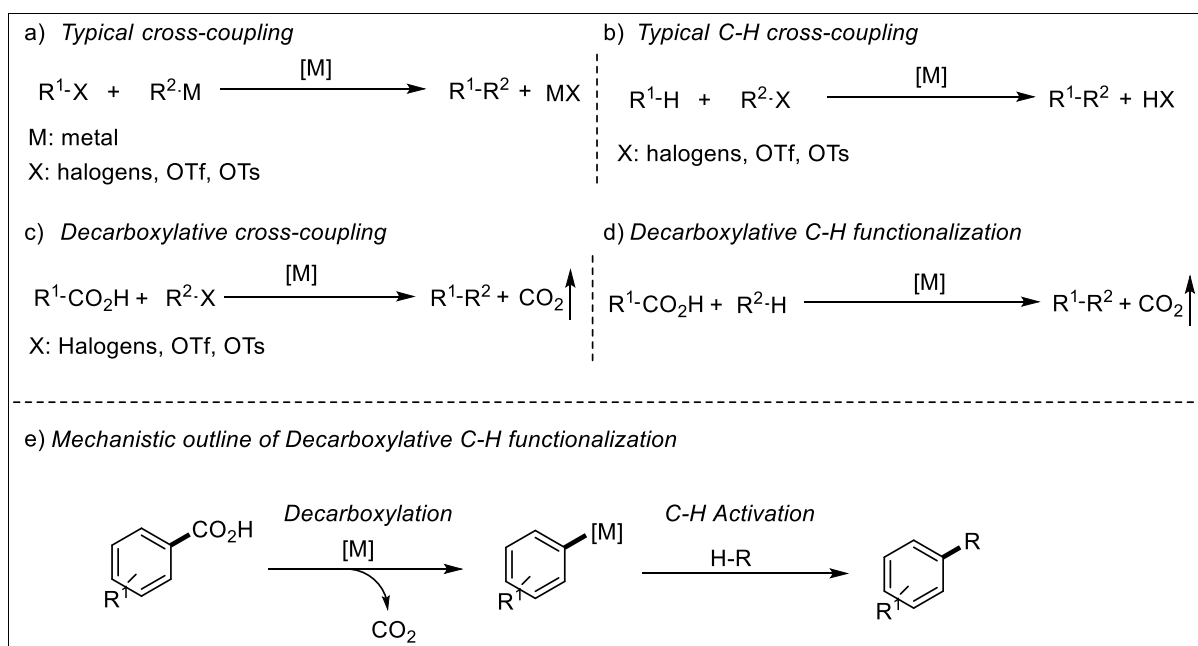
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bond functionalization reactions through a productive and environmentally viable approach including the mechanistic aspects, catalyst design and the scope of substrates for the reactions.

**Keywords:** Decarboxylation, Transition Metal Catalysts, Cross-Coupling Reactions, C-H Activation.

## 1. Introduction

Transition metal-catalyzed cross-coupling reactions with different organometallic reagents have been developed and extensively used for the preparation of bioactive compounds, natural products, polymers, and functionalized advanced materials.<sup>1</sup> Most of the organometallic reagents are air and moisture sensitive and difficult to handle. Therefore, specialized reaction setup and techniques are necessary for the typical cross-coupling reactions.<sup>2</sup> To address these limitations, direct activation of carbon-hydrogen bond has recently added notable attention for coupling reactions.<sup>3</sup> However, traditional C-H activation methods typically need organometallic reagents or halo arenes as coupling counterparts, leading to the generation of stoichiometric metal or acid waste. In the past few decades, cross-coupling reaction via decarboxylation has arisen as a viable substitute for traditional cross-coupling and also typical C-H functionalization. This approach utilizes carboxylic acids as reaction counterparts which are readily available, cost-effective and resistant to both air and moisture. Also, in the decarboxylative coupling reactions the byproduct is stoichiometric amount of CO<sub>2</sub> which is non-toxic, non-flammable, and can be easily removed from the reaction system that is a crucial factor in achieving sustainable development for chemical synthesis.<sup>4</sup> In these reactions, a uniquely metalation occur via decarboxylative process replaces oxidative addition or transmetalation, forming organometallic species that serve as alternatives to aryl halides or organometallic reagents. These species then react with electrophiles to yield the desired coupling products (**Scheme 1**).<sup>5</sup> In recent years, in situ-generated organometallic compounds from the novel decarboxylative metalation step have been utilized in carbon-hydrogen bond activation to generate novel carbon-carbon bonds. By integrating the benefits of both C-H bond activation and decarboxylation, carbon-hydrogen functionalization reactions through decarboxylation have become an innovative synthetic approach for chemical transformations (**Scheme 1**). Two distinct mechanisms have been identified for the process of removing the carboxyl group from carboxylic acids in decarboxylative carbon-hydrogen bond activation reactions. The first is decarboxylation through redox-neutral, which generates an organometallic species without altering the oxidation state of the involved metal ions. The second is oxidative decarboxylation, where radical intermediates are formed, and the metal ions facilitating the process undergo a change in oxidation state. Over the past few years, significant advancements have been attained in transition metal catalyzed decarboxylative carbon-hydrogen bond activation reactions, highlighting their substantial synthetic utility.<sup>6</sup> This review discusses several efficient decarboxylative C-H functionalization reactions along with their mechanistic insights.



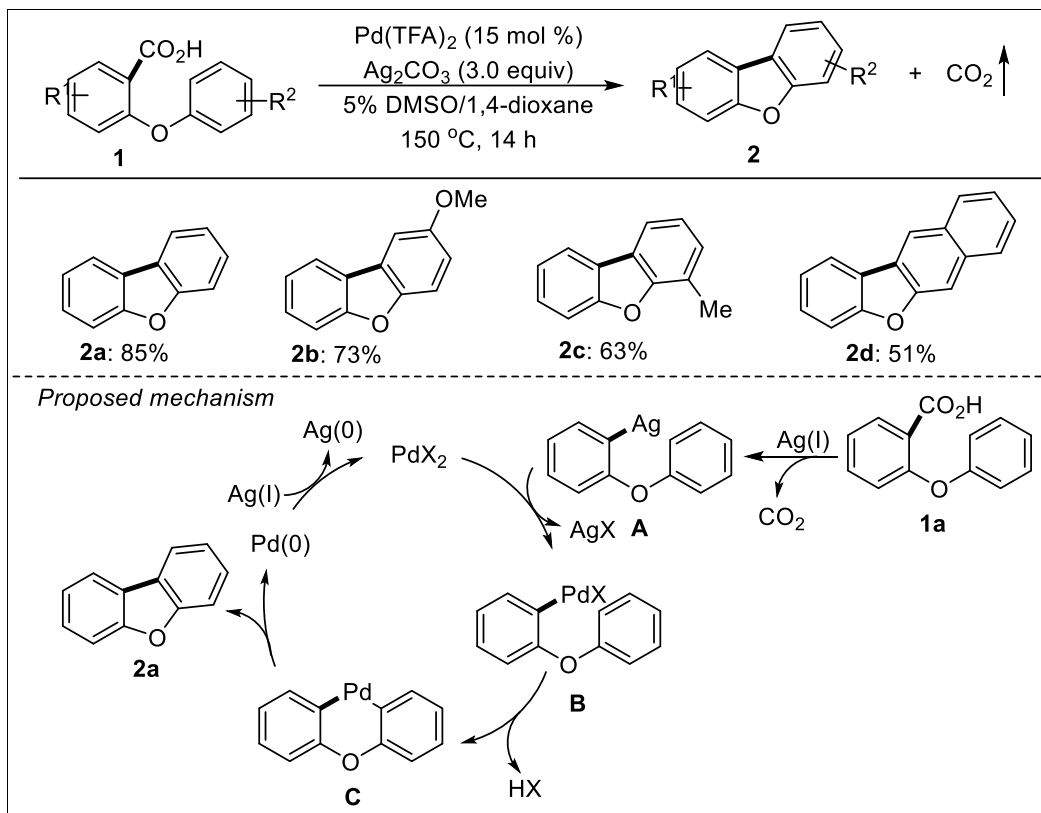
**Scheme 1:** Typical cross-coupling vs decarboxylative cross-coupling reaction

## 2. Decarboxylative C-H functionalization Reactions

### 2.1. Palladium catalyzed intramolecular decarboxylative arylation of $sp^2$ C-H bond

The C-H arylation reaction of arenes via decarboxylation was first disclosed by the Crabtree group in 2008. They employed anisole and 2,6-dimethoxybenzoic acid as reaction counterparts, yielding a combination of meta- and para-arylated products in a 3:1 proportion. In addition to the targeted coupling products, a significant amount of 1,3-dimethoxybenzene was also isolated from 2,6-dimethoxybenzoic acid.<sup>7</sup>

Consequently, Glorius and his team were successfully reported a regioselective palladium(II)-catalyzed arylation reaction with ether linked benzoic acids via sequential intramolecular activation of C-H bond followed by decarboxylation providing dibenzofuran framework (**Scheme 2**).<sup>8</sup> From the mechanistic perspective of the reaction, an aryl-silver species (**A**) is produced after silver-assisted decarboxylation of benzoic acid. This intermediate then undergoes transmetalation with a Pd(II) complex, forming an palladium(II)-aryl species (**B**), which subsequently undergoes intramolecular C-H activation to produce a cyclic palladium complex (**C**). Shortly, the targeted product is obtained via reductive elimination and regenerating the catalyst Pd(II) from Pd(0) via oxidation with silver(I) carbonate for the next catalytic sequence (**Scheme 2**).

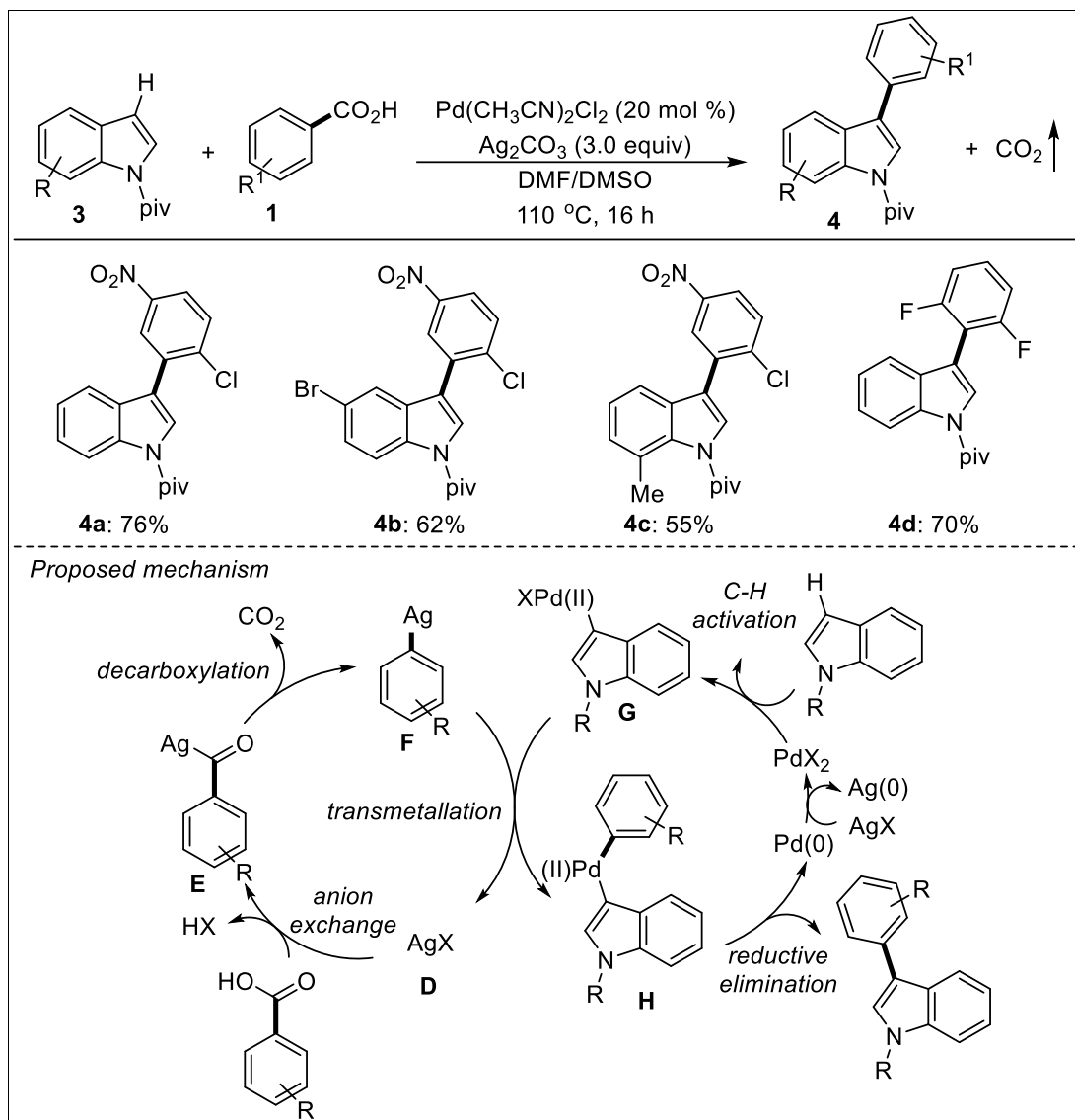


**Scheme 2:** Intramolecular palladium(II)-catalyzed arylation reaction via decarboxylation

## 2.2. Decarboxylative C3-H arylation of indoles

For C-H bond functionalization in heteroaromatic systems, the Larrosa research group established a palladium/silver catalyst system for the C3-H arylation of indoles by the use of benzoic acids through decarboxylation mode (**Scheme 3**).<sup>9</sup> In the reaction,  $\text{Pd(MeCN)}_2\text{Cl}_2$  and  $\text{Ag}_2\text{CO}_3$  were used as catalyst and oxidant respectively. Notably, the reaction exhibited excellent regioselectivity and exclusively yielded C3-aryl substituted products of *N*-protected indoles.

Mechanistically, silver facilitates the decarboxylation process, while palladium shows a crucial role for the activation of C-H bonds. Both metals were found to be essential for the C3-H arylation of C-3 position. The C3-H bond in indole is activated by palladium, leading to the formation of an aryl-palladium species (**G**) that readily reacts with an aryl-silver species (**F**) obtained through the process of decarboxylation to yield a diaryl-palladium complex (**H**) via transmetalation process. From the diaryl-palladium complex (**H**), the coupling biaryl product is provided via reductive elimination pathway. The resulting Pd(0) catalyst may be reoxidized to Pd(II) by the oxidant  $\text{Ag}_2\text{CO}_3$ , allowing for the subsequent catalytic cycle (**Scheme 3**).

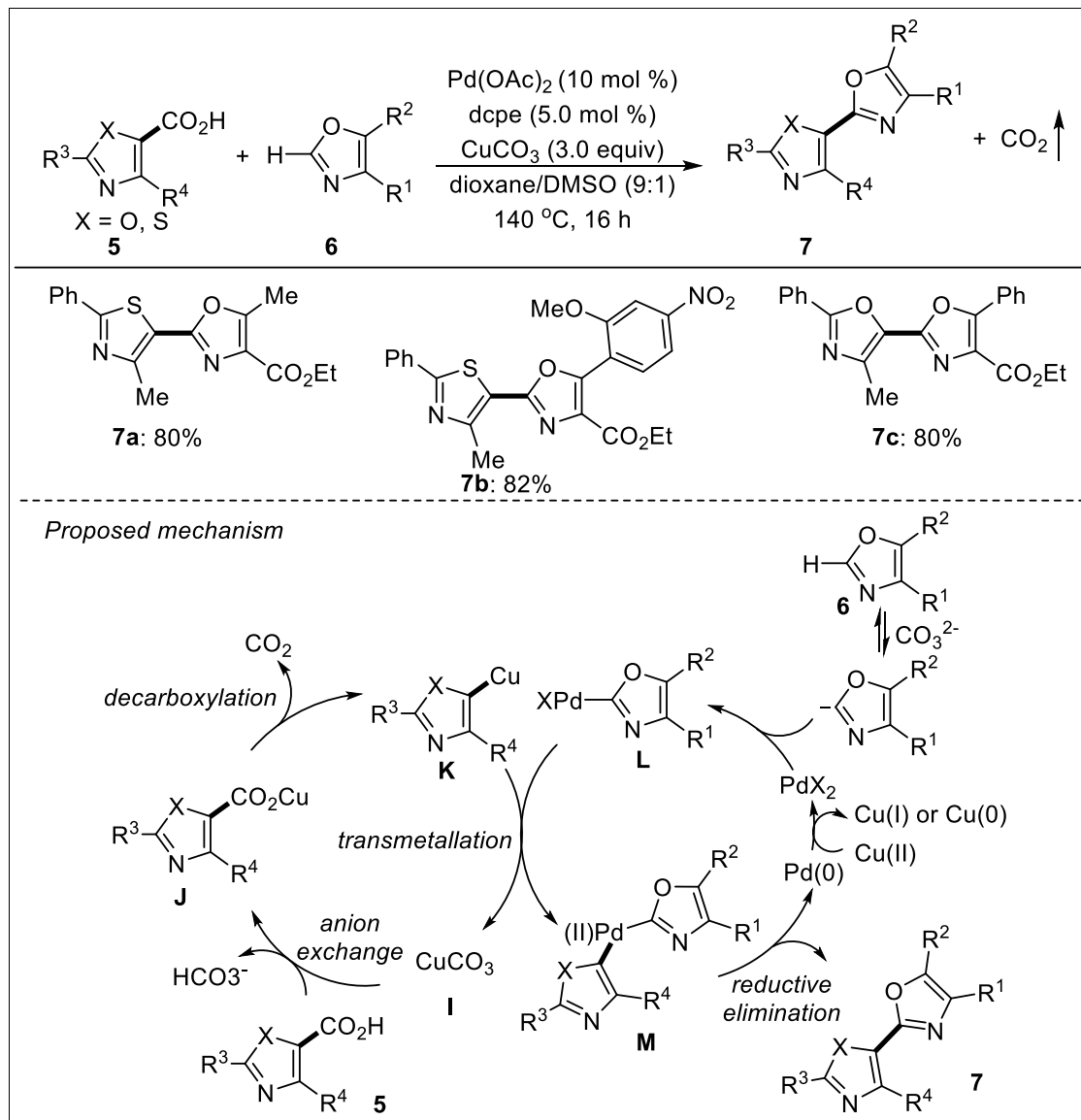


**Scheme 3:** Palladium-catalyzed C3-H arylation of indoles via decarboxylation

### 2.3. Decarboxylative C-H hetero-arylation of azoles

A copper/palladium-based catalytic scheme have reported for the C-H heteroarylation of azoles via decarboxylation by the Greaney and coworkers for the construction of biologically active scaffolds such as oxazoles, thiazoles, and imidazoles using heteroaromatic acids (**Scheme 4**).<sup>10</sup> The reaction takes place using  $\text{Pd}(\text{OAc})_2$  as a catalyst with stoichiometric amounts of copper salts and a sterically hindered bidentate bis(dicyclohexylphosphino)ethane(dcppe) ligand in dioxane/DMSO solvent. Mechanistically, a copper-aryl species (**K**) is made from the heteroaromatic acids through Cu-assisted decarboxylation. Simultaneously, the C-H bond of oxazole reacts with a base carbonate ( $\text{CO}_3^{2-}$ ) followed by Pd(II),

afforded a palladium(II)-aryl intermediate (**L**). Then this species undergoes transmetalation with the copper-aryl intermediate, forming a palladium(II)-diaryl complex (**M**). Lastly, the product hetero-biaryl along with the Pd(0) catalyst are made by reductive elimination. Subsequently the Pd(0) is oxidized again to Pd(II) by Cu(II) salt, completing the cycle (**Scheme 4**).

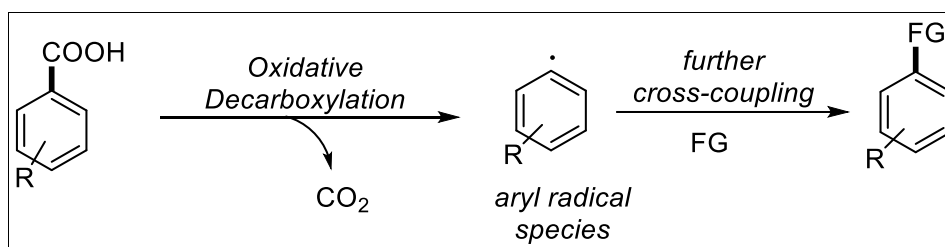


**Scheme 4:** Palladium catalyzed C-H hetero-arylation of azoles through decarboxylation

#### 2.4. Decarboxylative C-H arylation reaction through formation of an aryl radical

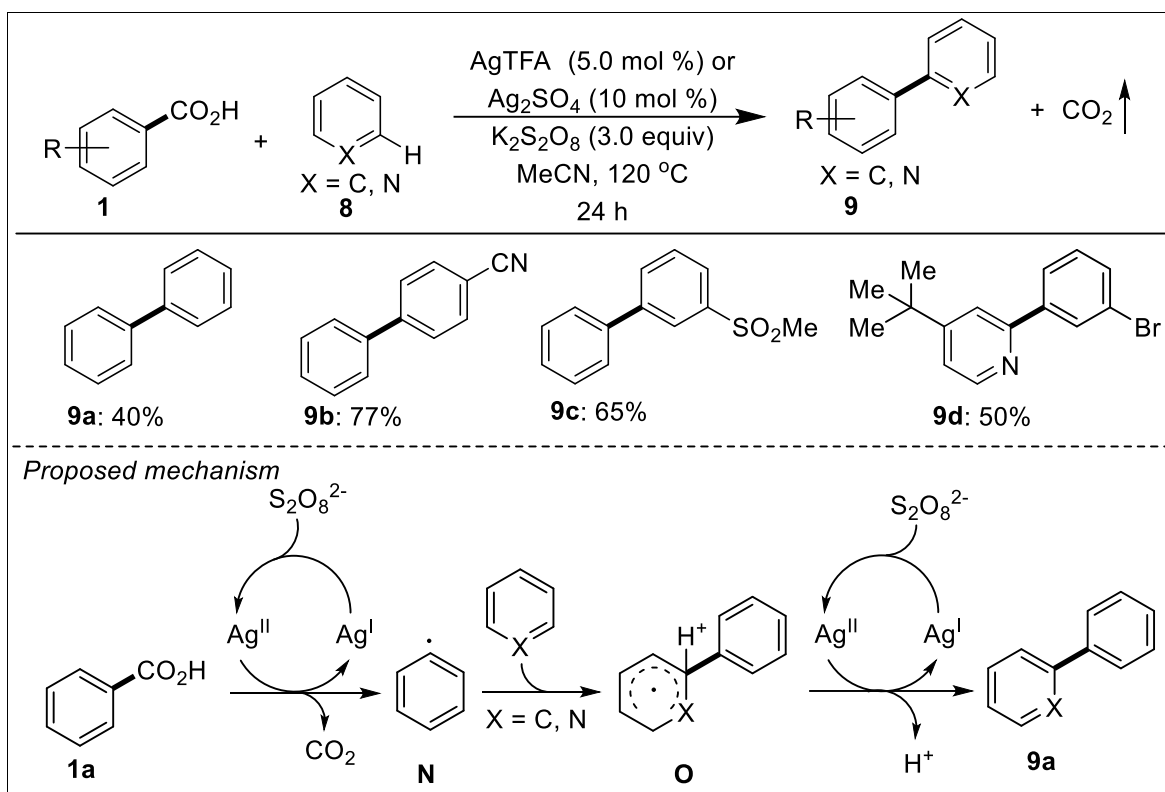
Aryl radicals are well-recognized as key synthetic intermediates in chemical transformations.<sup>11</sup> Owing to their high reactivity, they can be generated from only a few functional groups, including aryl

boronic acids,<sup>12</sup> aryl diazonium salts,<sup>13</sup> and aryl halides.<sup>14</sup> However, such approaches are not useful because of instability, high cost, and limited availability of the starting materials. Subsequently, the Li group introduced a straightforward and effective methodology for generating aryl radicals from more affordable aryl triflates.<sup>15</sup> Thus, adopting the full benefits of decarboxylation reactions, Su and his coworkers reveals the synthesis of aryl radicals from the unactivated carboxylic acids through a radical decarboxylation pathway (**Scheme 5**).<sup>16</sup> Shortly, the Glorius research group introduced a visible light-driven decarboxylation process of arene carboxylic acids, leading to the formation of aryl radical intermediates.<sup>17</sup> In these methods, aryl radical intermediates are generated by the expulsion of CO<sub>2</sub> from the corresponding carboxylic acids, followed by their coupling with arenes through a Minisci-like reaction (**Scheme 6**).<sup>18</sup>



**Scheme 5:** Formation of an aryl radical through decarboxylation process

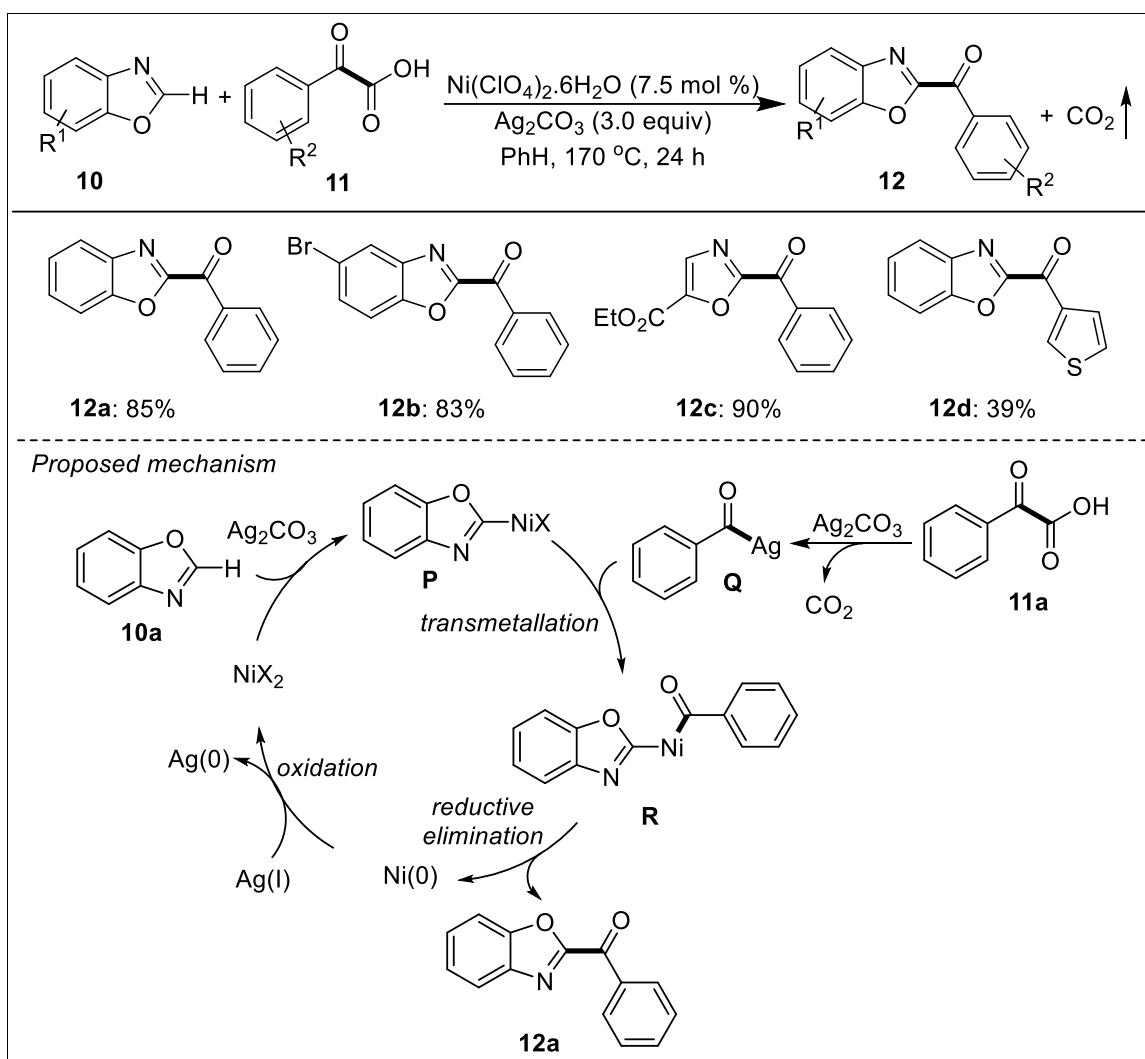
With respect to the aryl radical generation from arene carboxylic acids, the Su group successfully developed a method using a catalytic quantity of a cost-effective silver salt as the catalyst and an equivalent proportion of K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> as the oxidant.<sup>16</sup> Mechanistically, the process begins through the oxidation of silver(I) to silver(II) with persulfate anion (S<sub>2</sub>O<sub>8</sub><sup>2-</sup>). Then the silver(II) facilitates oxidation of the arene carboxylic acid, triggering decarboxylation and forming an aryl radical intermediate (**N**). Afterwards, the generated aryl radical interacts with benzene or pyridine, leading towards the formation of a cyclohexadienyl radical species (**O**). Lastly, transient species (**O**) undergoes oxidation by the silver(II) species, restoring aromaticity and producing the targeted arylation product (**Scheme 6**).



**Scheme 6:** C-H arylation reaction with simple arenes via novel decarboxylation process

### 2.5. Decarboxylative acylation reactions of $sp^2$ C-H bonds

2-Oxocarboxylic acids have been investigated for transition metal-catalyzed activation of C-H bond through the novel decarboxylative pathway.<sup>19</sup> In these reactions,  $\alpha$ -ketocarboxylic acids serve as acyl group equivalents for ketones synthesis. Subsequently, the Ge group successfully reported acylation reaction of azoles using  $\alpha$ -ketocarboxylic acids and nickel as catalyst (**Scheme 7**).<sup>20</sup> Mechanistically, the catalytic sequence begins with the nickelation of the azole with the help of  $\text{Ag}_2\text{CO}_3$ , resulting a Ni(II)-aryl species (**P**). This is followed by a transmetalation process between the **P** intermediate and the Ag-acyl intermediate (**Q**) that is generated via silver-assisted decarboxylation of the acid, making an acyl-aryl-Ni(II) species (**R**). Lastly, the **R** species undergoing reductive elimination process generates the expected ketone product and restores the catalyst Ni(0). Further this catalyst can undergo reoxidation to Ni(II) by the oxidant  $\text{Ag}_2\text{CO}_3$  and be used for the subsequent cycle (**Scheme 7**).

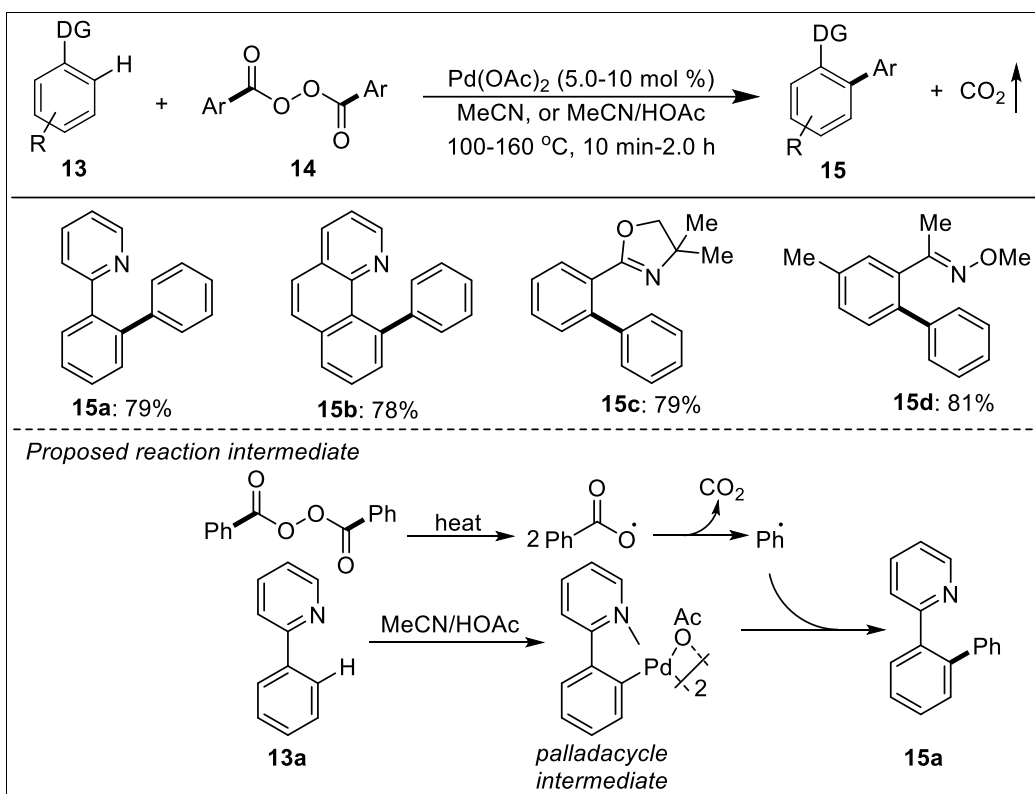


**Scheme 7:** Nickel-catalyzed acylation of  $\text{sp}^2$  C-H bond of azoles via decarboxylation

## 2.6. Directing group assisted C-H arylation reactions via novel decarboxylation process

Heteroatoms like Nitrogen (N), Oxygen (O), and Sulfur (S) containing functional groups are mostly employed as directing groups for the transition metal-catalyzed activation of unreactive carbon-hydrogen bonds. In this process, directing groups guide the metal to reside in specific positions in the substrate enabling selective C-H functionalization.<sup>3</sup> Additionally, directing groups can increase the stability of organometallic species produced during breaking of C-H bond, like palladacycle and rhodacycle intermediates. Moreover, these intermediates can undergo chemical transformations to produce the intended coupling compounds. Subsequently, a variety of directing groups has been successfully explored for the decarboxylative C-H bond cross-couplings.

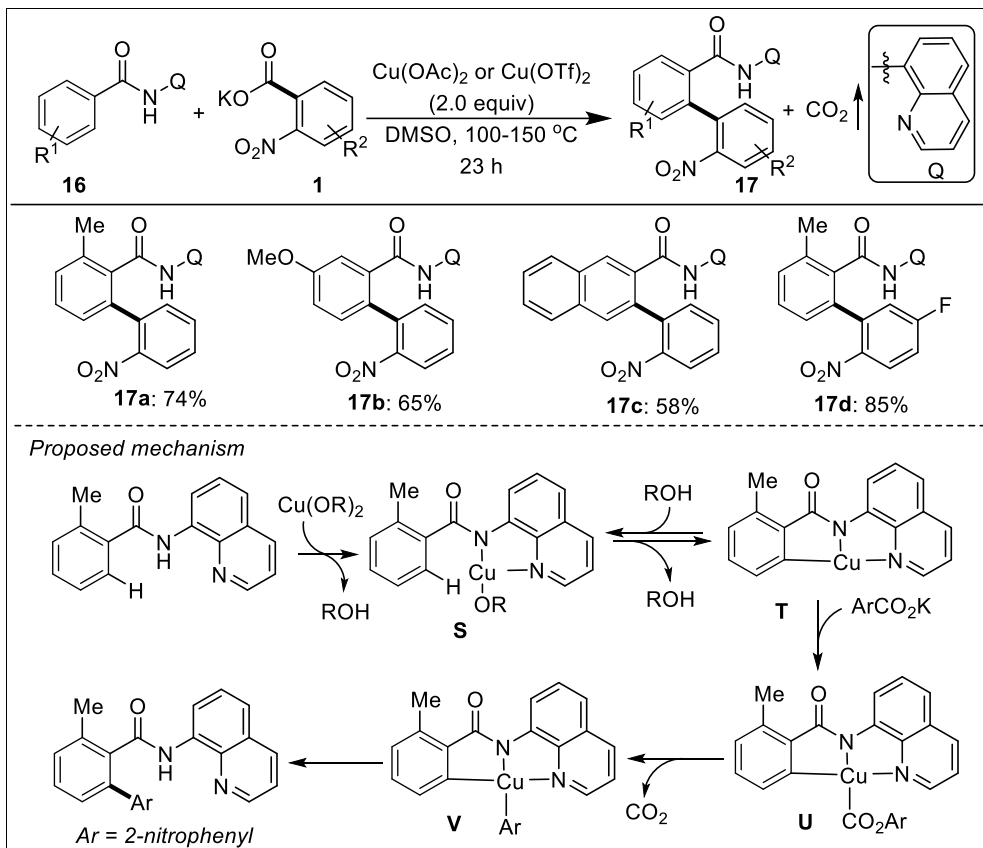
Regarding that perspective, Yu and co-workers successfully developed a chelation-guided decarboxylative arylation of  $sp^2$  C-H bond with aryl acyl peroxides (**Scheme 8**).<sup>21</sup> In this method, a range of *N*-containing directing groups, for example pyridine, oxime, and oxazole, have shown compatibility, exhibiting well reactivity and selectivity in palladium-catalyzed decarboxylative arylation of  $sp^2$  C-H bond. Mechanistically, pyridine-directed cyclopalladation of palladium(II) through an electrophilic palladation step, a five-membered dimeric palladium complex is produced. On the other hand, the coupling partner peroxides undergo dissociation under thermal conditions, followed by decarboxylation, ensuing in the formation of the arene radicals. The dimeric palladium complex then undergoes oxidative incorporation with the arene radical, finally yielding the final arylated product (**Scheme 8**).



**Scheme 8:** Directing group aided Pd(II)-catalyzed decarboxylative arylation

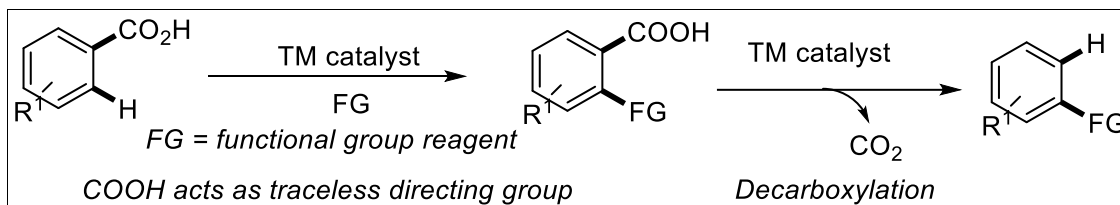
Subsequently, the Miura group introduced an 8-aminoquinoline-guided, copper-assisted decarboxylative arylation of  $sp^2$  C-H bond in benzamides, employing nitrobenzoic acids as coupling partners (**Scheme 9**).<sup>22</sup> In this reaction, the 8-aminoquinoline moiety in the substrate is crucial for the reaction outcome and the N-H bond in the amide is also essential. Other bidentate directing groups were found ineffective under the given reaction conditions. Mechanistically, the reaction initiates through deprotonation of the comparatively acidic N-H bond within the substrate and consequently forms the *N,N*-bidentate chelate compound (**S**) with Cu(II) salt. This intermediate (**S**) undergoes a successive

carbon-hydrogen bond cleavage and generates a five-membered copper (II) species (**T**). A Cu(III) intermediate (**U**) is formed shortly through a disproportionation reaction with the species (**T**) and a salt of carboxylic acid. Subsequently, copper-mediated decarboxylation of intermediate **U** produces the aryl-copper species (**V**). Finally, reductive elimination from intermediate **V** yields the corresponding arylated product (**Scheme 9**).



**Scheme 9:** 8-aminoquinoline-directed Cu(II)-assisted decarboxylative C-H arylation<sup>23</sup>

The carboxyl functional group also can act as a transient directing group for *ortho* selective C-H bond functionalization, followed by oxidative decarboxylation in a sequential manner within a unified process (**Scheme 10**).<sup>24</sup>



**Scheme 10:** Carboxylic acid-directed decarboxylative functionalization reactions

Over time, various research groups have reported several decarboxylative C-H activation reactions carried out under benign conditions, attaining high yields with wide range of substrate scopes.<sup>25</sup>

## Conclusion

Transition metal-catalyzed decarboxylative C-H functionalization has appeared as one of the greatest persuasive strategies for C-C bond foundation. This methodology not only offers a direct and effective pathway for activating inert C-H bonds but also minimizes the need for pre-functionalized activated substrates, toxic reagents, and harsh reaction conditions offering significant advantages over traditional coupling reactions or carbon-hydrogen functionalization reactions. Employing transition metal catalysts, including palladium, nickel, copper, and silver, has expanded the opportunity of this reaction, enabling the synthesis of complex molecular architectures with high selectivity and proficiency. The ability to employ abundant carboxylic acid derivatives and the minimal waste generation in the reaction have contributed to the sustainability of this method and also connecting with green chemistry principles. Despite significant advancements in this field, a major limitation of this method is that the reactions occurred at high temperatures ( $\geq 120$  °C). Therefore, further investigation into new catalyst design, mechanistic understanding, lowering reaction temperature and also substrate flexibility will enrich the practical application of this strategy in industrial processes.

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