ORIGINAL RESEARCH PAPER

Science

KEY WORDS:

SUSTAINABLE PHOTOCHEMICAL CARBONYLATION STRATEGIES TOWARDS EFFICIENT SYNTHESIS: RECENT ADVANCES AND APPLICATIONS.

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ABSTRACT

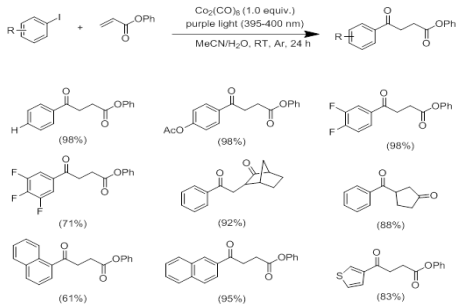
Visible-light-driven carbonylation reactions provide an attractive platform for the sustainable synthesis of carbonyl-containing compounds under mild conditions. Traditional carbonylation methods often rely on noble-metal catalysts, high carbon monoxide pressures, and harsh reaction conditions, limiting their practicality. This review highlights key developments in a three-component photochemical carbonylation of aryl halides. These methodologies exploit visible-light irradiation to generate reactive radical intermediates, facilitating carbon–carbon bond formation without external photosensitizers or precious metals. Mechanistic studies reveal diverse pathways, including radical, acylium ion, and electron donor–acceptor complex-mediated processes, providing insights into regio- and chemoselectivity control. This review highlights the progress, mechanistic understanding, and practical applications of sustainable visible-light carbonylation of aryl halides, underscoring its potential in modern organic synthesis. The reaction tolerates a broad range of substituents and diverse alkenes, demonstrating high functional-group compatibility. Recent advances have demonstrated that cobalt carbonyl based systems can rival traditional noble metal catalysts in terms of activity, selectivity, and substrate scope. This methodology offers an efficient and sustainable approach for constructing structurally diverse carbonyl compounds using visible light and an abundant metal source.This protocol highlights the potential of visible-light-mediated strategies using earth-abundant metals for constructing structurally diverse carbonyl-containing compounds in a mild and sustainable manner.

INTRODUCTION

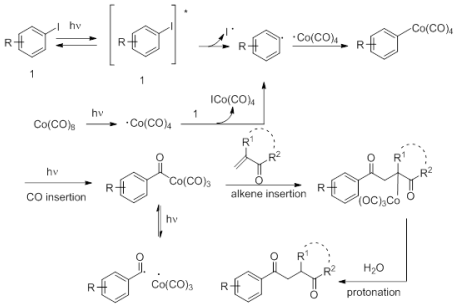
Carbonylation reactions represent one of the most powerful and versatile strategies in organic synthesis for the construction of carbonyl-containing compounds, which are ubiquitous in pharmaceuticals, agrochemicals, and functional materials. Traditional carbonylation methods often rely on noble-metal catalysts, high pressures of carbon monoxide, and harsh reaction conditions, which limit their sustainability and practical applicability. Consequently, the development of mild, efficient, and environmentally benign carbonylation protocols using earth-abundant metals has attracted significant attention in recent years.

Photochemical activation has emerged as an effective tool for enabling unconventional reactivity under mild conditions by harnessing visible light as a clean energy source. In particular, visible-light-induced radical processes have opened new avenues for carbon–carbon bond formation, allowing carbonylation reactions to proceed without the need for external photosensitizers or precious-metal catalysts. Among earth-abundant transition metals, metal carbonyl complexes such as cobalt carbonyls are especially attractive due to their dual role as both carbon monoxide sources and reactive intermediates under light irradiation.

In this context, Liu and co-workers reported a visible-light-driven three-component carbonylation of aryl halides using cobalt carbonyl under purple LED irradiation. This strategy enables the efficient construction of 1,4-dicarbonyl frameworks through a radical pathway involving aryl halides, carbon monoxide, and alkenes. The method demonstrates broad substrate scope, high functional-group tolerance, and excellent yields under mild conditions, providing a valuable contribution to sustainable photochemical carbonylation chemistry. Liu and co-workers fabricated three-component carbonylation reaction of aryl-halides which is initiated by the absorption of light by using ample metal carbonyl in 2024. They conducted the carbonylation reaction between iodo-benzene and 1.0 equiv. $Co_2(CO)_8$ by using methyl acrylate and then irradiated with 395-400 nm purple LED (**Scheme 1**). As a result, 1,4-dicarbonyl product with moderate to excellent yield. If the amount of aryl-halide was decreased, then 98% yield was attained. Aryl-iodides having electron-with-drawing and electron-releasing substituents, (VinylSulfonyl)benzene, dimethyl maleate, cyclopent-2-en-1-one and bridged 3-methylene-2-norbornanone were converted efficiently into the corresponding products. The reaction was started by the creation of photoexcited aryl-iodide from aryl-iodide by the irradiation of purple light and then C-I bond homolysis was takes place. Insertion of CO and alkene to give intermediate, later the product with opened ring was generated and it gives the desired product (**Scheme 2**). This method is useful for the synthesis of wide variety of organic structures.

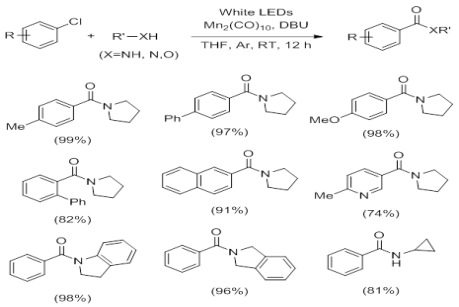


Scheme 1. A three-component carbonylation reaction of aryl-halides initiated by the absorption of light between iodo-benzene and 1.0 equiv. $Co_2(CO)_8$ with methyl acrylate and then irradiated with 395-400 nm purple LED.

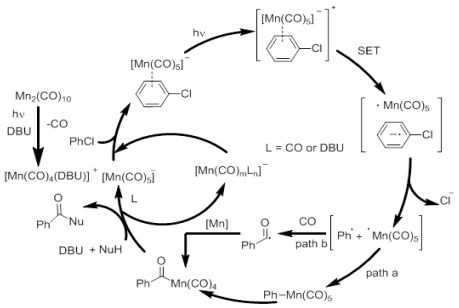


Scheme 2. Proposed Mechanism for the Visible-Light-Driven Three-Component Carbonylation of Aryl Halides Leading to 1,4-Dicarbonyl Products

In 2025, Xue and co-workers developed carbonylation process of (hetero)aryl-chloride catalysed by $n_2(CO)_{10}$ which is triggered by visible light. They conducted the amino-carbonylation reaction of chlorobenzene with pyrrolidine by using 20 mol% $n_3(CO)_{10}$ and 2.0 equiv. DBU in 1.0 mL THF and it was irradiated by 10W LEDs at normal temperature in argon for a period of 12 h, N-benzoylpyrrolidine, product of aminocarbonylation was formed with moderate to excellent yield **(Scheme 3)**. An electron-releasing group attached to para position of aryl chlorides, ortho compounds were efficiently transformed into amides. The desired products were obtained by incorporating the optimizing conditions with heteroaryl-chlorides and naphthyl chlorides, pyridine and quinoxaline. The possible mechanism for the given reaction is SRN1. $Mn_2(CO)_{10}$ reacts with amine and the visible light induced to release CO, forming a manganese anion. This anion binds to aryl chloride, undergoes single electron transfer (SET), and loses a chloride ion, creating an aryl radical and manganese radical pair. These radicals combine, insert CO, and form a species of acyl manganese, which reacts with a nucleophile to produce the desired carbonylated product **(Scheme 4)**. This approach facilitates the aminocarbonylation and alkoxycarbonylation of unreactive aryl chlorides under ambient conditions, offers broad applicability to various substrates, and produces the desired carbonylated compounds with excellent efficiency.

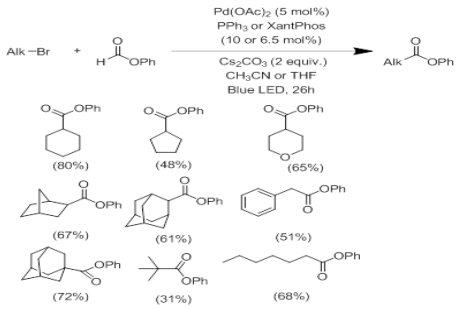


Scheme 3. Selective and efficient activation of carbonylation process of (hetero) aryl-chloride under Mild reaction conditions.

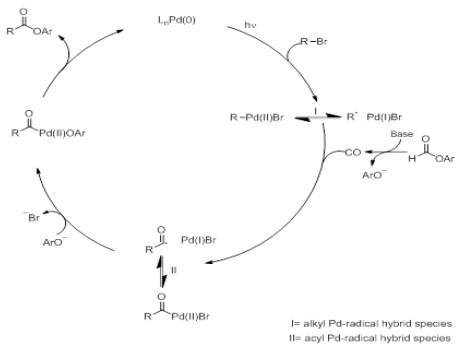


Scheme 4. Suggested Mechanism for theaminocarbonylation and alkoxycarbonylation of unreactive aryl chlorides under ambient conditions.

In 2024, Chuentragool and co-workers developed a mild one-pot carbonylation reaction of alkyl-halides which was driven by visible light and the aryl-formates was used as the source of phenoxide and a CO substitute. They reacted aryl-halide with aryl-formate using 5 mol% $Pd(OAc)_2$, 10 mol% PPh3 and 2 equiv. Cs_2CO_3 in CH_3CN by the irradiation of blue LED with 40 W for 26 h and isolated moderate to high yields of desired esters **(Scheme 5)**. The phenyl rings bearing electron-withdrawing groups, tricyclic and bicyclic secondary bromoalkanes, iodocyclohexane and bromocyclohexane gives the desired esters efficiently. The mechanism put forward was a pd-radical pathway. Alkyl halides undergo an oxidative addition which is driven by a light to give hybridized species of alkyl Pd-radical. CO insertion and reductive elimination were held, later a nucleophile was incorporated to afford the desired ester **(Scheme 6)**. This gives an alternate method for the conversion of alkyl-halides which were inactivated to the esters.

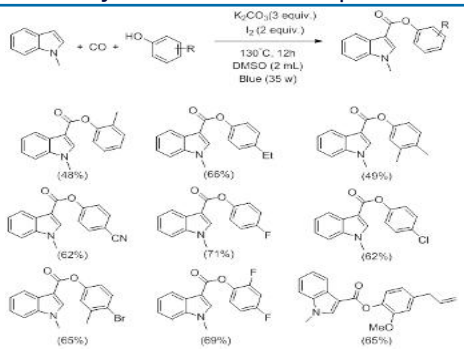


Scheme 5. One-pot carbonylation reaction of alkyl-halides driven by visible light using 5 mol% $Pd(OAc)_2$, 10 mol% PPh3 and 2 equiv. Cs_2CO_3 in CH_3CN by the irradiation of blue LED with 40 W for 26 h and isolated moderate to high yields of desired esters

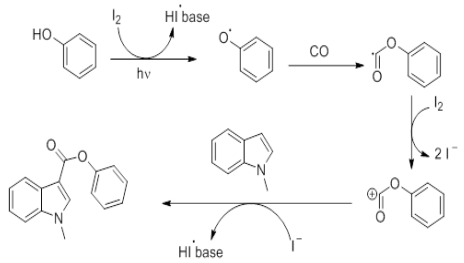


Scheme 6. The suggested mechanism for the Visible-Light-Induced Pd-Radical Pathway for Alkyl Halide Carbonylation.

In 2021, Peng and co-workers synthesized indole-3-carboxylates by the carbonylation reaction of indoles at metal-free conditions with phenols which was induced by visible light. They reacted N-methyl indole with phenol using 2 equiv. I_2 and 3 equiv. K_2CO_3 in 2 mL DMSO at 120°C for 12h, by irradiating with a white LED of 35 W and isolated moderate to high yields **(Scheme 7)**. Many Phenolic derivatives bearing electron-withdrawing and donating groups efficiently results the desired product in variety of yields. Phenols with electron-withdrawing substituents provided higher yields comparably. This carbonylation process was compatible with various functional groups such as cyano, acetyl, halide and ether groups. A carbonylation reaction via radical pathway was proceeded here. A phenol radical was generated by the oxidation of a single-electron of phenol under the irradiation of visible-light with I_2. Benzoyl radical formation by CO trapping, then it was oxidized to form acylium ion by I_2. Nucleophilic attack gives the corresponding product

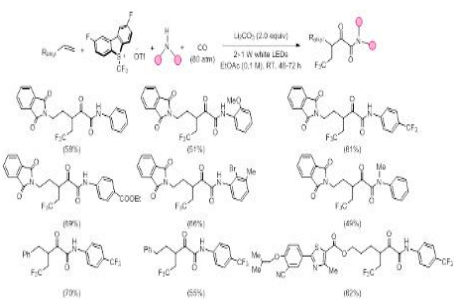


Scheme 7. A Metal-Free Visible-Light Strategy for Indole-3-Carboxylate Synthesis

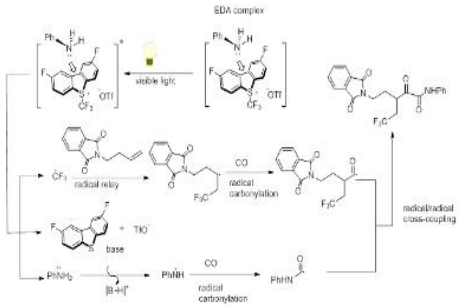


Scheme 8. A Proposed Mechanism for the Radical-Mediated Metal-Free Carbonylation of Indole.

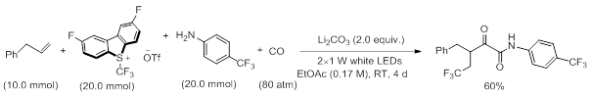
In 2023, Chen and co-workers developed a photochemical five-component radical cascade for alkene functionalization via aminocarbonylation. They developed the transformation involving unactivated alkene, aniline, and Umemoto's reagent in a 2:1:2 ratio under 80 atm of CO atmosphere by using 2.0 equiv. Li_2CO_3 in EtOAc in stainless-steel autoclave fitted with dual quartz windows, allowing it to withstand pressure while being exposed to light from blue LEDs of 2×1 W and enables efficient synthesis of significant -trifluoromethyl -ketoamides, offering good yields and excellent chemoselectivity **(Scheme 9)**. The chemoselectivity and efficiency of the reaction seem to be influenced by the choice of base. Various primary aniline compounds with electron-withdrawing or electron-donating substituents at the different positions on the benzene effectively took part in this reaction. A photoactive EDA complex forms between Umemoto-type reagent and aniline, which take part a visible light-induced single electron transfer (SET) in its excited state, generating an anilinium cation and a CF_3 radical. The CF_3 radical then adds to the alkene, forming an alkyl radical that reacts with carbon monoxide to produce an acyl radical. Simultaneously, the anilinium cation yields a carbamoyl radical through sequential deprotonation followed by carbonylation. Phenyl isocyanate may arise from the carbamoyl radical via oxidative pathway **(Scheme 10)**. Ultimately, a cross-coupling between the acyl and carbamoyl radicals delivers -ketoamide in high selectivity. The gram-scale reaction successfully synthesizes 2.42 grams of the desired product with a 60% yield **(Scheme 11)**. Crucial was the discovery that amine coupling partners first enable the creation of photochemically active EDA complexes by donating electrons to radical precursors, and later facilitate carbamoyl radical generation by accepting CO.



Scheme 9. Photochemical five-component radical cascade for alkene functionalization via aminocarbonylation.

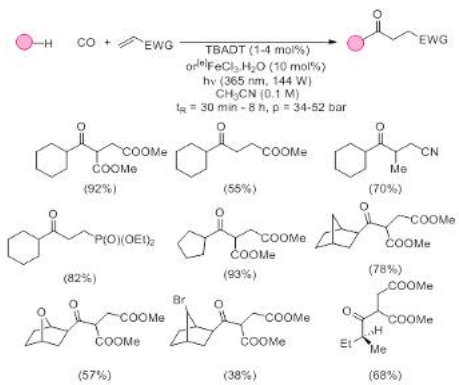


Scheme 10. Suggested Mechanism for the Photochemically Triggered Multicomponent Radical Cascade for Alkene Aminocarbonylation.



Scheme 11. Gram-scale reaction for the synthesis of the Multicomponent Aminocarbonylation of Alkenes.

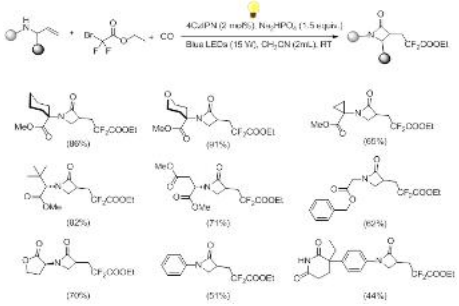
In 2023, Noel and co-workers developed carbon monoxide-mediated functionalization of C(sp^3)–H bonds via HAT (Hydrogen-Atom-Transfer) enabled photocatalysis in flow systems. They performed alkane functionalization using carbon monoxide alongside a range of olefinic substrates in flow by using 1 to 4 mol% TBADT and 10 mol% $FeCl_3.H_2O$ in 0.1 M CH_3CN for 30min to 8 h at 34 to 52bar under the irradiation of 365 nm, 144 W and isolated good yields of unsymmetrical ketones (**Scheme 12**). Mono-substituted olefins, conjugated enones can be involved in the reaction gives the desired product efficiently. The standard reaction conditions were compatible with a range of functional groups including nitriles, phosphonates, sulfones, protected alcohols, and ketones. A variety of bridged-bicyclic alkanes and electron-deficient alkenes were tolerated. Flow technology played a pivotal role in achieving efficient transfer of gas–liquid mass and rapid reaction kinetics, essential for suppressing side reactions while also providing an up-scalable and inherently safe platform.



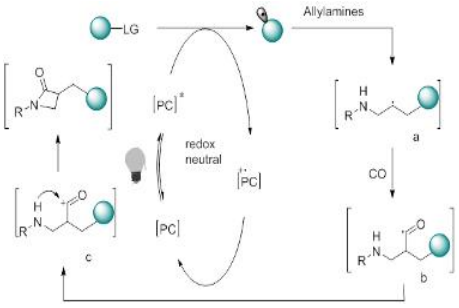
Scheme 12. Carbon monoxide-mediated functionalization of C(sp^3)–H bonds via HAT (Hydrogen-Atom-Transfer) enabled photocatalysis in flow systems.

In 2025, Wu and co-workers developed a mild light-driven carbonylative annulation for the synthesis of -lactams. They reacted allylamine and ethyl-difluorobromoacetate as electrophile-based radical source using 2 mol% 4CzIPN and 1.5 eq. Na_2HPO_4 in 2 mL CH_3CN by the irradiation of 15 W blue LEDs at ambient conditions under CO atmosphere and isolated excellent yield of -Lactams (**Scheme 13**). Sterically hindered groups, cycloalkylsubstituted, and isopropyl substrates gave the desired product effectively. The presence of an ortho-positioned isopropyl group enhanced the

efficiency of -lactam formation. Once excited, the photosensitizer transfers an electron to the radical precursor, forming a reactive fluoroalkyl radical. The double bond of allylamine reacts to form the alkyl carbon radical a. CO was inserted to generate -aminoacyl-radical intermediates b. Then it was quickly oxidized by the photocatalyst to give an acyl-cation c. An intramolecular nucleophilic attack by the amine then triggers cyclization, yielding the -lactams (**Scheme 14**). The mild reaction conditions and broad substrate compatibility highlight the potential of this method for pharmaceutical innovations.

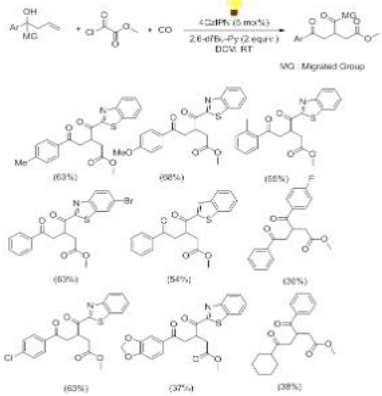


Scheme 13. Visible-Light-driven carbonylative annulation for the synthesis of β-lactam Frameworks.



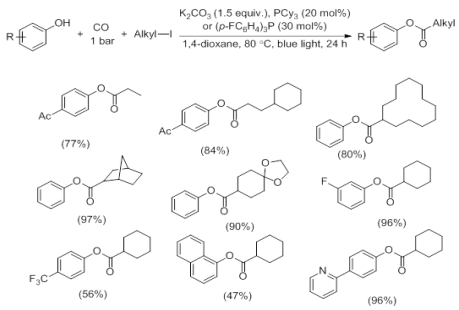
Scheme 14. Suggested Mechanism of the Light-Driven Carbonylative Annulation for the Synthesis of -Lactams.

In 2025, Wu and co-workers developed a mild migration strategy facilitated by alkoxycarbonyl radical and carbon monoxide for the incorporation of carbonyl groups into unactivated alkenes. They reacted methyl-oxalyl chloride as a source of alkoxycarbonyl radical and an unactivated alkene using 5 mol% CzIPN and 2.0 equiv. 2,6-di^{t}Bu-Py in 1.0 mL DCM under CO atmosphere by the irradiation of 15W blue LED and isolated moderate to high yields of asymmetric 1, 4-dicarbonyl compounds (**Scheme 15**). Substrates bearing para -Me, -^{t}Bu, -OMe substituents, and various benzo-heterocycles including benzofuran and benzoxazole afford the desired products efficiently. This method was successfully utilized for the final stage modification of a range of pharmaceutical compounds and natural products. For DL-menthol, cariniol, isoborneol, fructose, and epiandrosterone, the targeted products were effectively obtained.

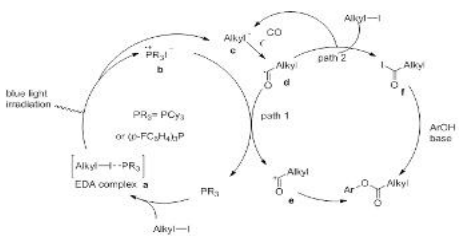


Scheme 15. Mild migration strategy facilitated by alkoxycarbonyl radical and carbon monoxide for the incorporation of carbonyl groups into unactivated alkenes

In 2023, Wu and co-workers proposed a light-activated phosphine-catalyzed alkoxycarbonylation process of alkyl-iodides utilizing 1,4-dioxane and phenols via a charge-transfer pathway. They reacted phenols and alkyl-iodides using 20 mol% PCy_3 and 1.5 equiv. K_2CO_3 in 0.5 mL 1,4-dioxane under a CO pressure of 1 bar by the irradiation of light, at 80 °C and isolated high to excellent yields of alkylphenol esters (**Scheme 16**). This reaction shows strong compatibility with various functional groups and delivers outstanding chemoselectivity. A wide range of phenols, phenols containing both electron-withdrawing and electron-donating groups, heterocycles, cyclic substrates effectively took part in the reaction, yielding the targeted products. Initially, PCy_3 and the alkyl-iodide form an electron donor–acceptor complex a, which becomes photoactivated upon exposure to blue light. Upon photoactivation, the EDA complex undergoes fragmentation to yield a Phosphinium-based radical ion-pair b and an alkyl radical c. The resulting alkyl radical c then captures carbon monoxide to form an acyl-radical d. From this point, two possible reaction mechanisms may occur: (Path 1) intermediate b oxidizes the acyl-radical d, resulting in the formation of acyl cation e, which subsequently undergoes reaction with phenol in a basic environment to afford the corresponding alkylphenol ester. (Path 2) Alternatively, the acyl-radical d may engage in an iodine atom transfer with an alkyl iodide, regenerating the alkyl radical c and forming an acyl iodide f, which leads to the formation of the intended alkylphenol ester (**Scheme 17**). It shows potential for scalability and further practical applications.



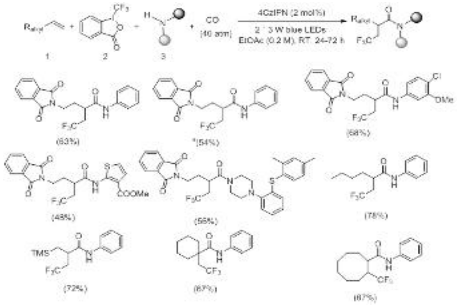
Scheme 16. light-activated phosphine-catalyzed alkoxycarbonylation process of alkyl-iodides utilizing 1,4-dioxane and phenols via a charge-transfer pathway.



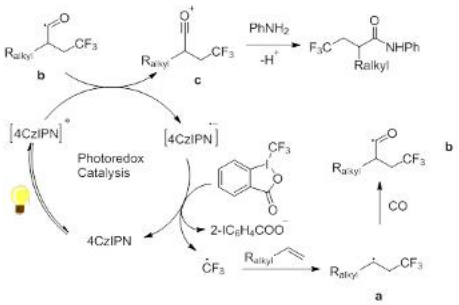
Scheme 17. Plausible Mechanism for the EDA-Complex-Mediated Carbonylative Ester Formation of Alkyl Iodides with Phenols via the Visible-Light.

In 2024, Chen and co-workers developed a light-induced radical relay-mediated four-component aminocarbonylation process for unactivated alkenes. They reacted unactivated alkenes, amines and Togni reagent II using 2 mol% 4CzIPN in EtOAc under CO atmosphere by the irradiation of 2 x 3 W blue LEDs for 24 to 72 h to form -perfluoroalkylated amides with good yields (**Scheme 18**). A series of aryl-amines containing substituents with a broad range of electronic properties at the various positions, heteroaryl amines featuring thiophene and pyridine and primary alkyl-amines were also amenable to the reaction conditions. Aliphatic alkenes, along with substrates containing ester, bromide, or ketone functionalities took part

in the process and gives the desired products efficiently. The radical anion generated from 4CzIPN is formed when the photoexcited 4CzIPN accepts an electron from an amine. This radical anion then reacts with a fluoroalkyl precursor to give a fluoroalkyl radical, which combines with an olefin and carbon monoxide to form an acyl radical b. The photoexcited 4CzIPN then oxidizes this acyl radical into an acyl cation c, while regenerating the 4CzIPN radical-anion. Finally, an amine reacts with the acyl cation c through nucleophilic addition, followed by deprotonation, to produce the final product (**Scheme 19**). Gram-scale reaction was conducted. This reaction enables easy synthesis of a range of useful -perfluoroalkyl amides.

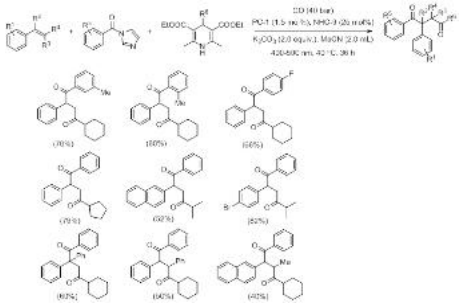


Scheme 18. Light-induced radical relay-mediated four-component aminocarbonylation process for unactivated alkenes. [a] Gram-scale reaction condition: 1(14.0 mmol), 2(7.0 mmol), 3(7.1 mmol), 4CzIPN (0.5 mol%), CO (40 atm), EtOAc (44 mL), 2 × 40W blue LEDs (λmax = 456 nm), 48 h.

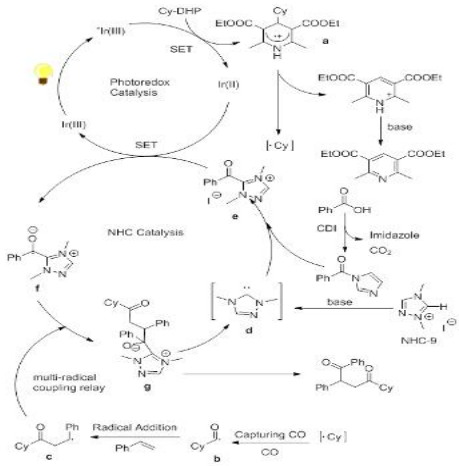


Scheme 19. Possible Mechanism for the Light-Induced Radical Relay-Mediated Four-Component Aminocarbonylation of Unactivated Alkenes.

In 2025, Wu and co-workers developed an alkene carbonylation with regioselective control enabled by N-heterocyclic carbene and photoredox catalysis. They reacted alkene, Hantzsch ester and acyl imidazole using 25 mol% NHC-9 and 2.0 equiv. K_2CO_3 in 2.0 mL MeCN under a CO pressure of 40 bar under the irradiation of light, at 40 °C and isolated excellent to moderate yields of 1,4-dicarbonyl compounds (**Scheme 20**). Various substituted alkenes like 5-vinylbenzo[d][1,3]dioxole, fluorine-substituted styrenes, , -disubstituted styrenes were efficiently gives the desired product. It has wide substrate scope and the electron-donating groups improve the efficiency of the reaction. Light irradiation activates the photosensitizer, which then oxidizes the Hantzsch ester, resulting in the formation of intermediate a. This process leads to the creation of cyclohexyl-radical, accompanied by the liberation of diethyl 2,6-dimethylpyridine-3,5-dicarboxylate following further deprotonation. The cyclohexyl-radical then captures carbon monoxide to generate acyl radical b, which adds to styrene via a radical mechanism, producing intermediate c. Concurrently, NHC-9 is deprotonated by a base to yield the active carbene species d. This carbene intermediate facilitates the transformation of acyl imidazole into the corresponding acyl azolium intermediate e. The chemically reactive intermediate f was generated through a photocatalytic single-electron transfer process. Finally, the coupling of intermediates f and c produces intermediate g, which undergoes transformation to deliver the desired compound, concurrently regenerating the carbene catalyst (**Scheme 21**). The synthetic applicability of the protocol was showcased by the efficient synthesis of valuable heterocycles including pyrrole, furan, and pyridazine and 1,4-diol derivatives.

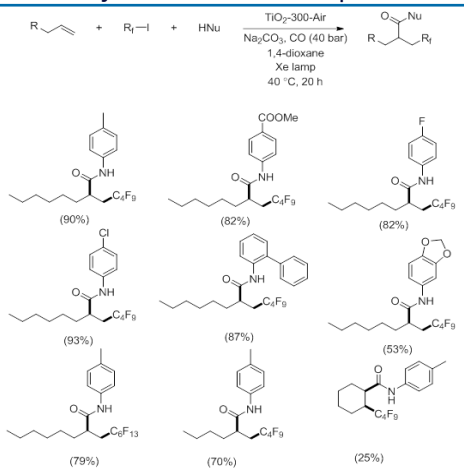


Scheme 20. Alkene carbonylation with regioselective control enabled by N-heterocyclic carbene and photoredox catalysis

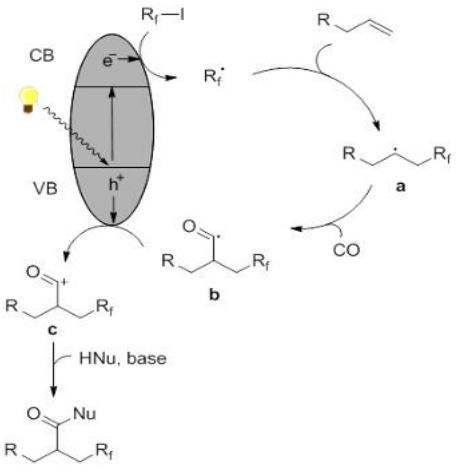


Scheme 21. Possible Mechanism for the Regioselective Alkene Carbonylation Enabled by N-Heterocyclic Carbene and Photoredox Catalysis.

In 2024, Chen and Co-workers developed photocatalytic light-driven perfluoroalkylative carbonylation process of unactivated alkenes by using recyclable catalyst. They reacted inactive olefins, perfluoroalkyl iodide and a nucleophile using TiO_2-300-Air and 2.0 equiv. Na_2CO_3 in 1.0 mL 1,4-dioxane under 40 bar CO atmosphere by the light irradiation from a xenon lamp at 40 °C and isolated moderate to excellent yields of -perfluoroalkylamide compounds (**Scheme 22**). It exhibits a wide substrate range and significant compatibility with various functional groups. Aniline substrates bearing various electron-withdrawing groups at the para position of the phenyl ring afford the desired products efficiently. Several alkyl-substituted alkenes and secondary amines were easily tolerated. Upon light irradiation, TiO_2 becomes photoexcited, electrons were promoted from the valence band (VB) into the conduction band (CB), resulting in the formation of electron–hole pairs. The valence band then donates an electron to perfluorobutyl iodide, generating a perfluoroalkyl radical. This radical adds to the olefin, forming radical intermediate a, which subsequently traps a molecule of CO to produce radical intermediate b. The latter undergoes oxidation by losing an electron to the conduction band, forming the corresponding intermediate c, which was transformed into the desired product via nucleophilic attack (**Scheme 23**). Employing TiO_2 offers several benefits, including an environmentally friendly, low cost and sustainable process. This catalyst minimizes the need for precious metals, eliminates the requirement for ligands, and can be conveniently isolated.



Scheme 22. photocatalytic light-driven perfluoroalkylative carbonylation process of unactivated alkenes by using recyclable catalyst.



Scheme 23. Suggested Mechanism for the TiO_2-Photocatalyzed Perfluoroalkylative Carbonylation of Alkenes with Amines.

CONCLUSION

Visible-light-driven carbonylation reactions have emerged as a sustainable and versatile platform for the construction of carbonyl-containing compounds under mild and environmentally benign conditions. Recent advances in three-component photochemical carbonylation of aryl halides demonstrate that earth-abundant metal systems, particularly cobalt carbonyl complexes, can rival traditional noble-metal catalysts in terms of efficiency, selectivity, and substrate scope. These reactions exhibit broad functional-group tolerance, accommodating both electron-donating and electron-withdrawing substituents, as well as diverse alkenes, enabling the synthesis of structurally complex 1,4-dicarbonyl products. Mechanistic studies indicate that these transformations proceed via radical pathways initiated by photoexcitation, followed by homolytic bond cleavage and carbon monoxide insertion, with potential involvement of acylium ions or electron donor–acceptor complexes. Overall, visible-light-mediated carbonylation using earth-abundant metals provides a practical, scalable, and sustainable strategy for the efficient assembly of structurally diverse carbonyl compounds, underscoring its significant potential in modern organic synthesis.

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