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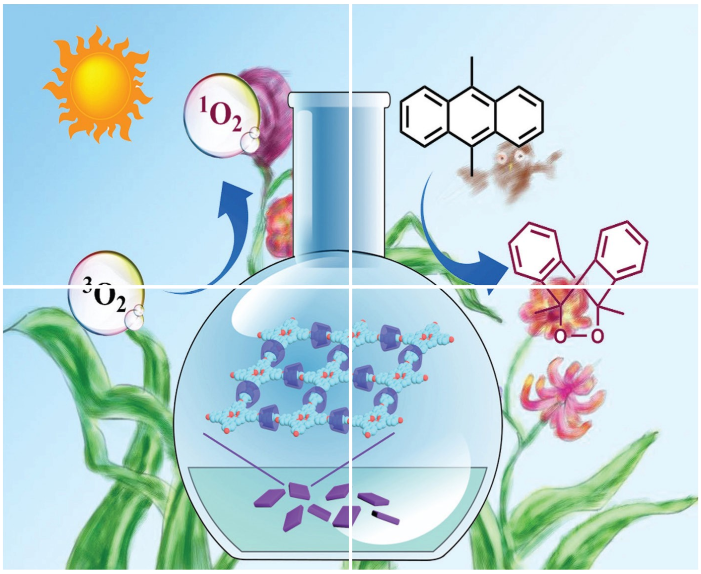

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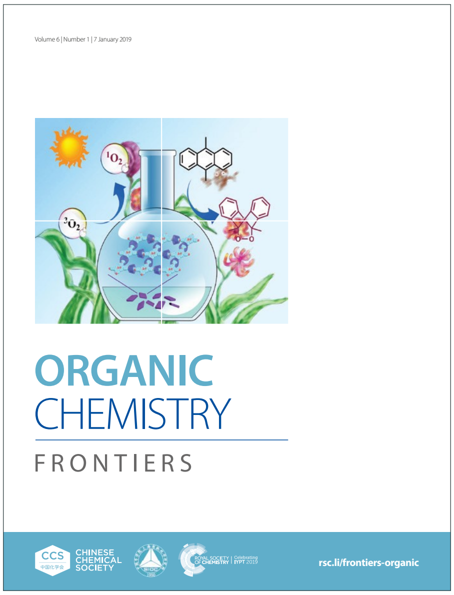

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ARTICLE

Recent advances in visible-light driven phosphine-mediated transformations

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Among the large literature on the use of trivalent phosphines in the activation of electrophilic unsaturated reagents, but the application of such derivatives for the generation of radical intermediates has received attention only in the recent decade. The present review aims to provide a detailed description of the phosphine-mediated photocatalyzed and photoredox catalyzed protocols for the formation of both carbon-carbon and carbon-heteroatom bonds proposed in the last decade.

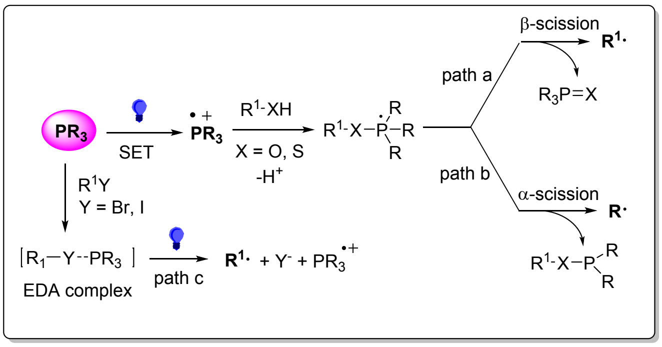
1. Introduction

Since the seminal experiments published in early '60s, the potentialities of trivalent phosphines as organocatalysts in synthesis have been intensively exploited. [1] However, in a large part of the literature available, the nucleophilic addition of the phosphine moiety on an electrophilic reactant (e.g. olefins, alkynes or allenes) promotes the formation a reactive zwitterion specie, which is then converted into the desired product[2-10] via electron-pair-transfer reactions· On the other hand, only few strategies based on a single-electron-transfer (SET) mechanism have been proposed,[11] but the same SET strategies are on the basis of the successful application of phosphines as redox active cocatalysts in photoredox catalyzed processes.[12] Such premises increased the attention for the development of phosphine-promoted visible light driven protocols, enabling chemical paths that are poorly or not accessible via thermal approaches. As described in Scheme 1, phosphine photo-organocatalysis has been reported to occur via three different paths, namely:

a) β-scission of a phosphoranyl radical (path a). Trivalent phosphine undergoes photoinduced Single Electron Transfer (SET) and the resulting radical cation $PR_3^{\bullet+}$ is trapped by a suitable (thio)alcohol R-XH. The resulting phosphoranyl radical releases a carbon centred radical via β-scission of the C-X bond. The process is thermodynamically driven by the stoichiometric formation of a stable phosphine oxide or sulphide as the by-product.

b) α-scission of a phosphoranyl radical (path b). The α-scission occurring in the phosphoranyl radical described in path b releases, via C-P bond homolytic cleavage, a carbon centred radical. Such behavior is kinetically favored by the presence of an unpaired electron occupying a P-X antibonding orbital. The competition between paths a and b was described to strictly depend on the relative bond strengths of the C-X and P-C bonds.[13]

c) Formation of an Electron Donor Acceptor (EDA) complex. In some case, a visible light absorbing EDA complex[14] could be formed via the in-situ interaction of electron-poor alkyl halides (e.g. perfluoroalkyl iodides) and trivalent phosphines via single electron transfer process. The photoinduced homolytic cleavage of the C-halogen bond then affords the desired alkyl radical.[14]



Scheme 1. Conceptual overview of phosphine-mediated photoredox processes.

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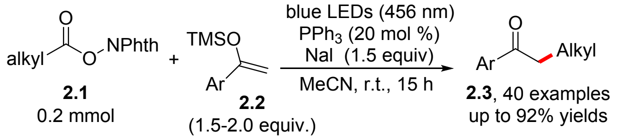
Since all the examined paths have been applied to organic synthesis, the present review aims to provide the readership with an overview of the most recent applications of phosphines in the visible light driven formation of Carbon-Carbon and Carbon-Heteroatom bonds. The classification is based on the first chemical bond formed, in accordance with the proposed mechanism. The use of continuous flow conditions as well as the application of the developed procedures in late stage functionalization approaches have been likewise considered and discussed in the following sections.

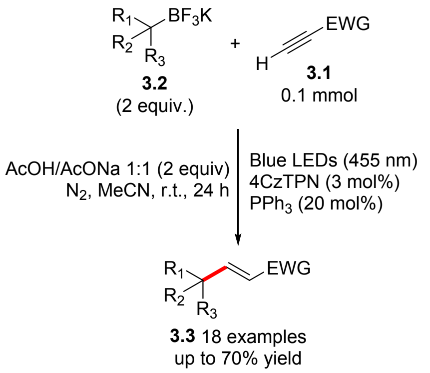
2. Carbon-Carbon bond formation.

Hydroalkylation reactions. The photocatalytic NaI-PPh_3 mediated decarboxylative alkylation of silyl enol ether **2.2** and redox-active esters **2.1** affords α-substituted acetophenones **2.3** in discrete to satisfactory yield. The reaction occurs under transition metal-free conditions (Scheme 2), in the presence of a stoichiometric excess of NaI.[15] The same strategy also promotes the Minisci-type alkylation of *N*-heterocycles and an analogous enantioselective α-amino alkylation with the assistance of a chiral phosphoric acid in catalytic amounts.



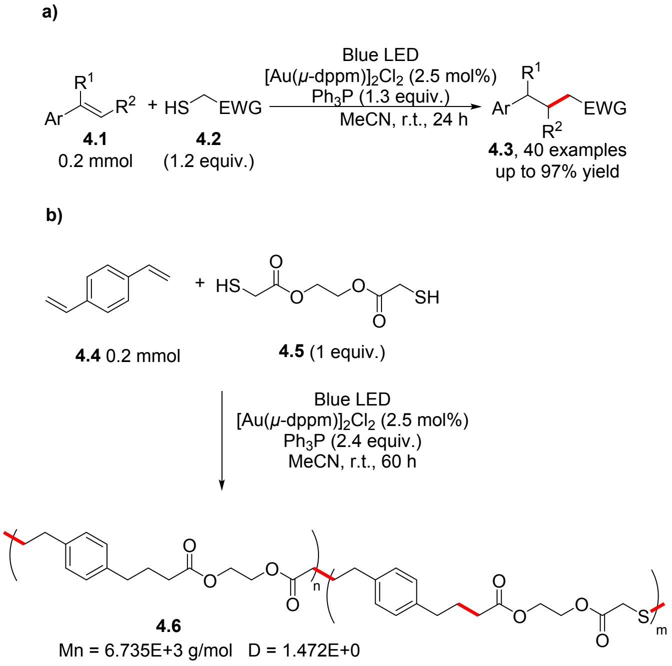
Scheme 2. Light driven, photocatalytic NaI-PPh_3 mediated decarboxylative alkylation of silyl enol ethers and nitrogen-based heterocycles.

Shi and co-workers applied a photoredox-promoted hydroalkylation strategy to the preparation of a library of DNA-tagged alkenes by using xanthates as the alkyl radical precursors in the presence of $Ir[FCF_3(CF_3)ppy]_2(dtbbpy)PF_6$ as the photoredox catalyst.[16] Phosphine organocatalysis was successfully merged with photoredox catalysis in the conversion of ynoates **3.1** to electron-poor olefins **3.3** under metal-free conditions, as described in Scheme 3.[17] The procedure involves a Giese-like coupling of **3.1** with alkylating agents trifluoroborate salts **3.2** in the presence of 4-CzTPN as the photoredox catalyst.



Scheme 3. Giese-like functionalization of ynoates **3.2** by dual phosphine organocatalysis and photoredox catalysis.

Mercaptans **4.2** have been recently proposed as electron-deficient precursors of carbon centred radicals in the hydroalkylation of styrenes **4.1** under blue light irradiation and in the presence of transition metal based species $[Au_2(\mu\text{-dppm})_2]Cl_2$ (Scheme 4a).[18] Such versatile strategy was successfully applied to the functionalization of small amino acids fragments as well as the preparation of polymers (an example in Scheme 4b). The same target was achieved under photocatalyst free conditions by Su and co-workers,[19] and exploited the partial oxidation of triphenyl phosphine to the corresponding triphenylphosphine oxide, that in turn forms an EDA complex with the yet unreacted PPh_3; the species is responsible for both the absorption of light and the initiation of the radical chain process.[19]



Scheme 4. Visible light driven Gold-catalyzed hydroalkylation of styrenes **4.1** in a) synthesis and b) in polymerization processes.

Perfluoroalkyl radicals have been generated and in turn employed for the hydroalkylation of electron-poor olefins (including fluoxetine and sertraline derivatives) from the hydrated form of hexafluoroacetone (**5.2**, Scheme 5) and trifluoro-acetaldehyde.[20] In this case, a key role is played by phosphoranyl radical **5.4**, arising from the coupling between triphenylphosphine radical cation and **5.2**.[20]

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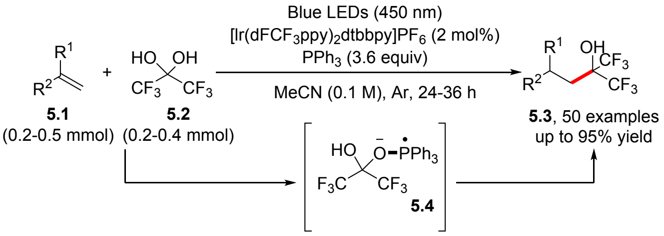
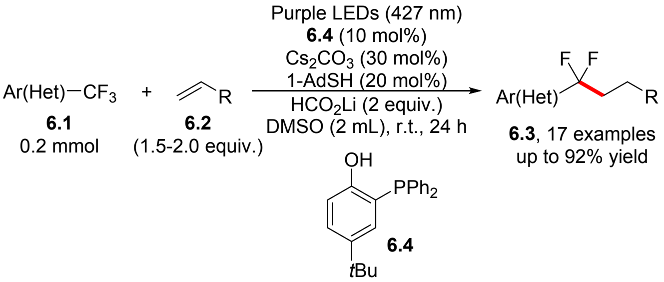


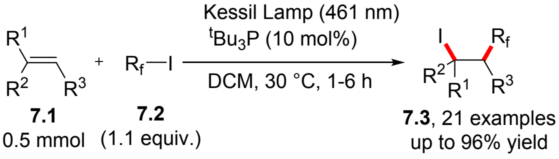
Scheme 5. Photoredox-catalyzed phosphine-mediated bifunctionalization of electron-deficient alkenes.

In situ deprotonation of phenol **6.4** generates o-phosphinophenolate anion that acts as a photoredox organocatalyst for activating trifluoromethyl groups in trifluoroacetamides, acetates, trifluoromethyl (hetero)arenes (**6.1**, Scheme 6) and even trifluoroalkanes. This approach enables the difluoroalkylation of non-activated alkenes and polyfunctionalized aromatics, including the antipsychotic compound trifluoroperazine.[21]



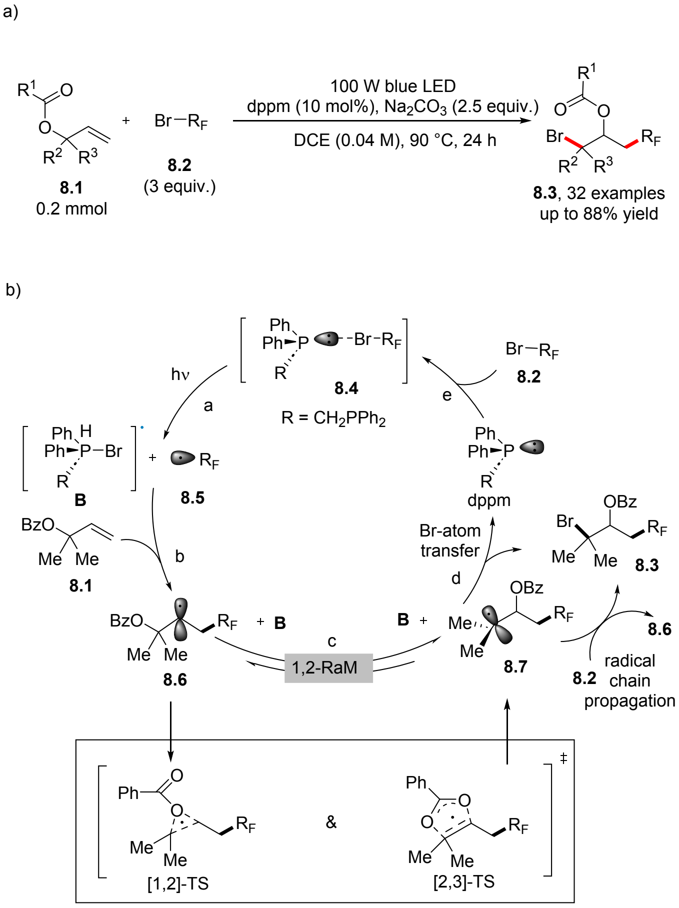
Scheme 6. Photoredox organocatalyzed difluoro(hetero)arylation and difluoroalkylation of non-activated olefins.

The difunctionalization of multiple bonds is currently one of the the most widely adopted approaches for constructing structurally complex molecules.[22-25] In this context, the 1,2-iodoperfluoroalkylation of alkenes via C-C and C-I bond formation was described to occur under visible light (461 nm) irradiation of a mixture of olefin **7.1** (Scheme 7), perfluoroiodoalkanes **7.2** and a catalytic amount of tri-tertbutylphosphine. The reaction mechanism involves an Atom Transfer Radical Addition (ATRA) chain path initiated by the formation of an EDA complex between **7.2** and the $^tBu_3P$ organocatalyst.[26] The same synthetic target was achieved by having recourse to a merged photoredox/copper catalysed strategy, in the presence of fac-Ir(ppy)$_3$ as the photoredox catalyst and Cu(MeCN)$_4^-$ as the cocatalyst, respectively.[27]



Scheme 7. Visible light induced photochemical iodo perfluoroalkylation of alkenes.

A 1,3-carbobromination strategy for the conversion of allyl carboxylates **8.1** to isopropyl carboxylates (sIPC) **8.3** was recently reported in literature (Scheme 8a).[28] The suggested non-chain-radical mechanism is shown in Scheme 8b and involves the formation of a visible light absorbing EDA complex **8.4**, between the phosphine (dppm) and **8.2**. Upon irradiation, **8.4** decompose in the bromo-phosphine **B** and alkyl radical **8.5** (path a). The latter is trapped by the olefin moiety in allyl carboxylate **8.1** (path b) and the obtained secondary alkyl radical undergoes 1,2-radical migration (1,2-RaM) to generate the more stable intermediate **8.7** (path c). Subsequently, bromo-atom transfer process from **B** to **8.7** affords the desired product (path d).[28]



Scheme 8. The photoinduced and phosphine-catalyzed difunctionalization of allyl carboxylates.

Acylation reactions. Aliphatic ketones play a crucial role in post-transformation and late-stage functionalization processes, making them privileged targets in organic synthesis.[29-30] Symmetric and asymmetric dialkyl ketones **9.3** have been obtained via Ir(III) photocatalyzed hydroacylation of styrenes (**9.1**, Scheme 9) by aliphatic acids **9.2** in the presence of diphenyl methyl phosphine as reported in Scheme 9.[31] In this case, a key role is played again by phosphoranyl radical **9.4** that in turn undergoes β-scission to release the desired acyl radical. Analogously, aromatic carboxylic acids were employed as precursors of acyl radicals in the preparation of α,α'-diaryl carbonyls via photoredox catalyzed acylation of 1,6-conjugate addition of *p*-quinone derivatives.[32] By following an analogous pathway, acyl oximes have been employed as the acyl radical

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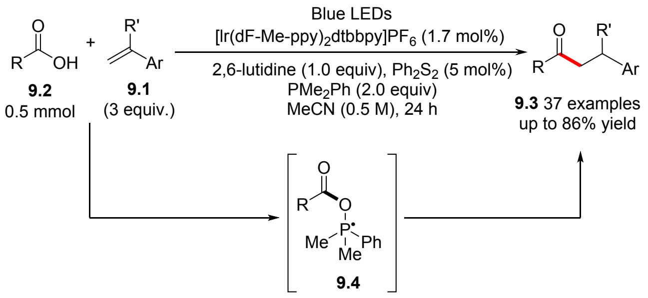
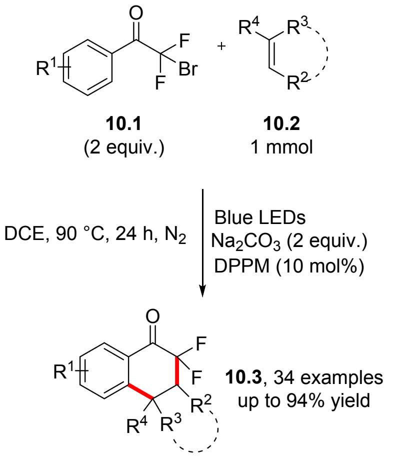
precursors in the hydroacylation of 1,1-diarylethylenes and α-trifluoromethyl styrenes.[33]



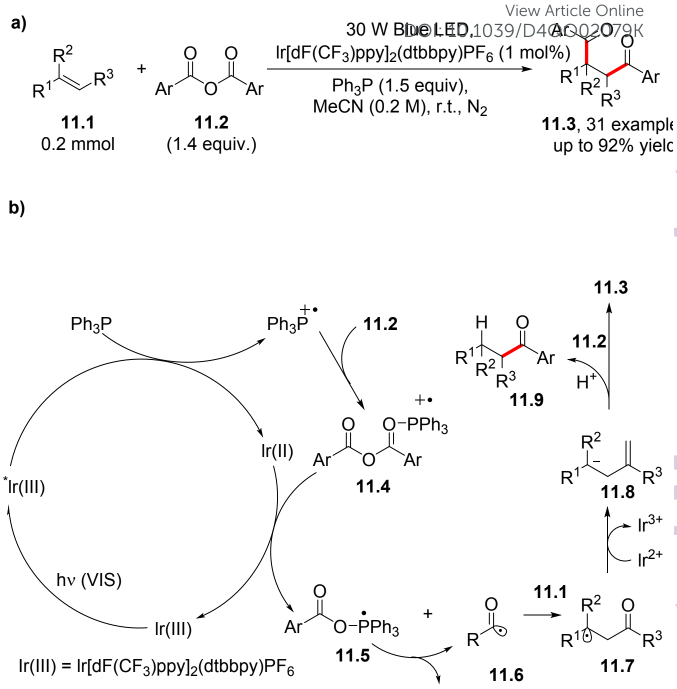
Scheme 9. Hydroacylation of styrenes **9.1** by carboxylic acids.

The photoinduced phosphine-catalyzed cyclization of bromodifluoroacyl arenes **10.1** with olefins has been recently applied to the preparation of a wide range of cyclic gem-difluoroacyl compounds **10.3** under photocatalyst-free conditions (Scheme 10).[34]



Scheme 10. Blue light mediated, photocatalyst-free preparation of cyclic ketones **10.3**.

Anhydrides **11.2** have been employed as acylating radical agents in the photocatalyzed 1,2 conversion of alkenes **11.1** into 1,4-Dicarbonyl derivatives (**11.3**, Scheme 11). In the proposed mechanism, the photoexcited photocatalyst (Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$)* undergoes reductive quenching by triphenyl phosphine, and the resulting radical cation PPh$_3^{\bullet+}$ is then trapped by **11.2** to form the complex **11.4** which allows the otherwise unfavorable reduction of the carboxylic moiety in **11.2** ($E_{1/2}^{red}$ = -1.41 V vs SCE) by Ir(II). Acyl radical **11.6** arising from the decomposition of intermediate **11.5** is in turn trapped by the C=C double bond of **11.1**, and the resulting radical **11.7** undergoes reduction to carbanion **11.8**, which reacts with another molecule of **11.2** to form the diacylated product. In the presence of a proton source (e.g. water), **11.8** is finally converted into the corresponding hydroacylated derivative **11.9**. [35]



Scheme 11. A) Visible light driven photoredox-catalyzed 1,2-difunctionalization of olefins; b) suggested mechanism.

1,6-Dicarbonyl derivatives **12.3**[36-38] are suitable via a regioselective 1,6-addition of acyl radicals (in turn generated from carboxylic acids **12.1** under Ir(II) photoredox catalyzed conditions) onto electron-deficient 1,3-dienes (**12.2**, Scheme 12a).[39] When exposed to visible light, the excited photoredox catalyst undergoes oxidative quenching by **12.2**, generating the radical anion **12.6** (Scheme 12b, paths a and b). As a result, triarylphosphine is oxidized through a single-electron transfer process, while restoring the starting photoredox catalyst (path c) and the resulting phosphoranyl radical intermediate **12.4** (path d) decomposes and releases acyl radical **12.5** (path e) which then couples with **12.6** (path f). Protonation of the so-generated carbanion **12.7** by water (path g) affords the desired product **12.3.**[39]

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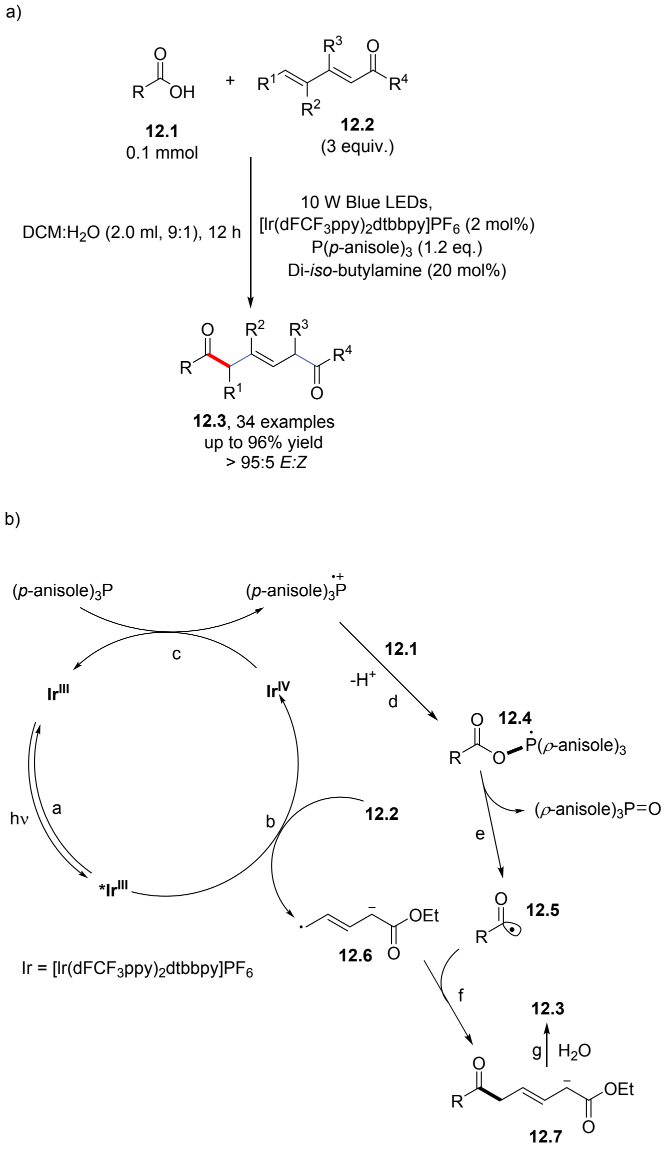
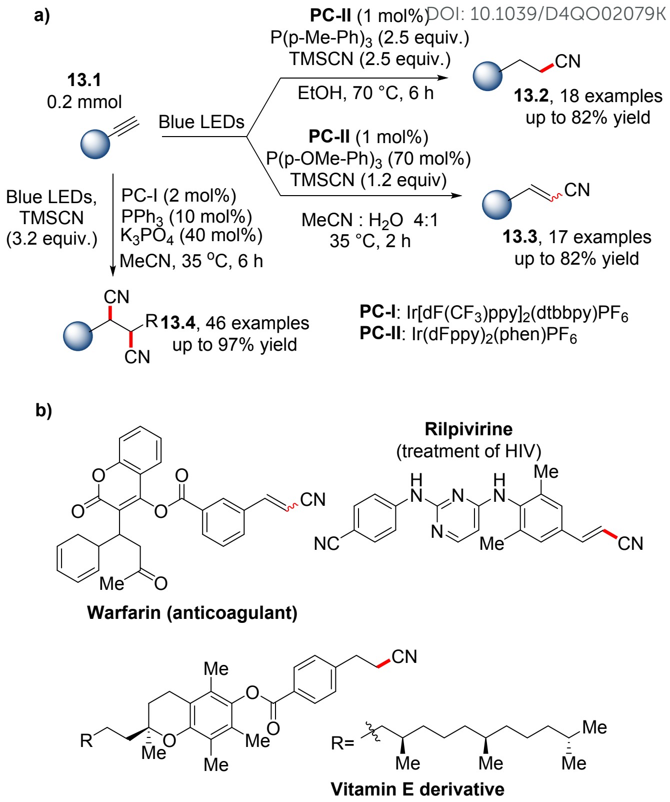


Scheme 12. Regioselective, photoredox catalyzed decarboxylative acylation of electron poor 1,3-dienes **12.2**.

A divergent approach for the selective preparation of alkyl nitriles (**13.2**, Scheme 13a), alkenyl nitriles (**13.3**) and alkyl dinitriles (**13.4**) from alkynes has been proposed by Chu and co-workers.[40] As pointed out in Scheme 13a, the choice of phosphine is critical in determining the reaction outcome. Notably, a wide range of polyfunctionalized substrates, including Warfarin analogues, pharmaceutics, sugars and Vitamin E can be selectively functionalized under the tunable conditions (some examples are depicted in scheme 13b).



Scheme 13. a) Phosphine/photoredox catalyzed divergent cyanation of alkynes and b) selected applications.

Arylations. The incorporation of fluorine atoms and fluorine-containing functional groups into complex (hetero)aromatic cores is a flourishing research area, in view of the opportunity to fine-tune key physicochemical properties including, among the others, solubility, metabolic stability and bioavailability.[41-48] In this context, the use of phosphine as radical initiator in the perfluoroalkylation of electron rich heterocycles **14.1** (including indoles) has been proposed only recently. In the case described by Jin and co-workers,[49] and suitable at Scheme 14a, an EDA complex (**14.4**) between the chosen perfluoroalkyl iodide **14.2** and the phosphine catalyst is involved. Visible light irradiation promotes a single electron transfer, and the so generated perfluoroalkyl radical $\mathbf{R_F}^{\bullet}$ which is in turn trapped by the heteroaromatic core. The obtained radical species **14.5** is thus oxidized by another molecule of **14.2** to release, after deprotonation of the Wheland intermediate **14.6**, the desired product **14.3** and so restarting, at the same time, the radical chain. The protocol was found successful also when starting from indoles conjugated to bioactive fragments such as Zomipitran and Melatonine.[49]

Analogously, the introduction of *gem*-difluoromethyl group into alkenes and (hetero)cyclic cores (including dimethyl uracil) has been reported to occur via a visible-light promoted phosphine-catalyzed difluoroalkylation strategy. [50]

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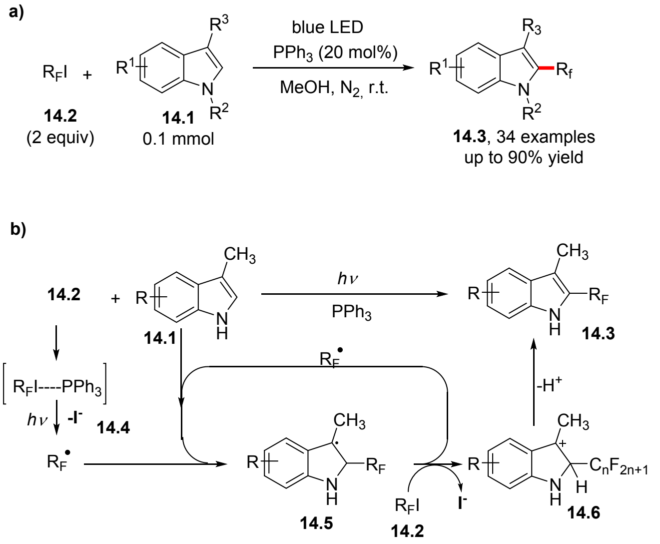
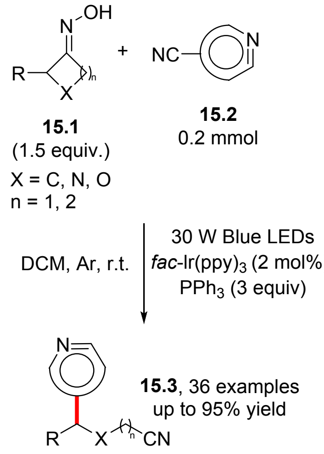


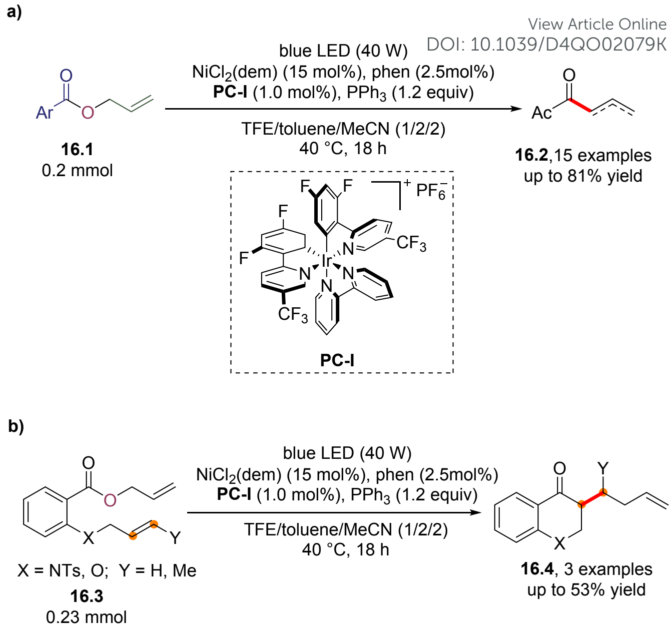
Scheme 14. Visible light-driven phosphine-catalyzed perfluoroalkylation of indoles.

As mentioned earlier, nitrile is one of the most prevalent and versatile units in organic synthesis, acting as a precursors of different functional groups including amines, aldehydes, amidines, ketones, carbamates and carboxylic acids.[51] The research group of Yi accomplished an intriguing photoredox-neutral ring-opening pyridylation of cyclic oximes **15.1** (Scheme 15) by cyanopyridines **15.2**, to afford a wide array of pyridyl-alkylnitriles **15.3** with satisfactory selectivity (Scheme 15).[52]



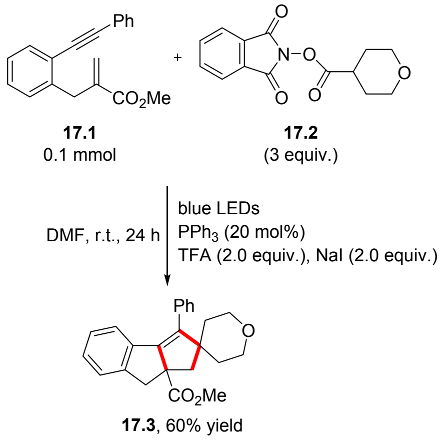
Scheme 15. Photoredox-neutral ring-opening pyridylation of cyclic oximes.

Extrusion reactions. In 2023, Tobisu and co-workers described a nickel/Ir(II) photoredox/phosphine promoted intramolecular C-C coupling occurring in allyl esters **16.1** for the preparation of asymmetric ketones **16.2**. in good yield (Scheme 16a).[53] The same conditions have been applied to obtain cyclic ketone (**16.4**, Scheme 16b) via functionalization of allyl esters bearing a tethered alkene moiety (**16.3**). [53]



Scheme 16. Nickel/photoredox/phosphine catalysed preparation of ketones from allyl esters.

Xu and co-workers described in 2019 a single example of phosphine/iodide-based photocatalytic decarboxylative [2+2+1] annulation of 1,6-enynes and *N*-hydroxyphthalimide esters, providing the corresponding cyclopenta[a]indene **17.3** in 60% yield (Scheme 17). The procedure can be considered as a transition metal-free alternative to fac-Ir(ppy)$_3$ catalyzed approach described in the same paper. [54]



Scheme 17. Preparation of spyroderivative **17.3** via phosphine/iodide-based photocatalytic decarboxylative [2+2+1] annulation.

3. Carbon-Heteroatom bond formation

C-S bond formation. The incorporation of a trifluoromethylthio moiety (-SCF$_3$) in pharmaceuticals and agrochemicals is able to finely tune their lipophilicity and electronic properties.[44,47,55] Qing and co-workers disclosed a facile and mild strategy for the Ir (II)-photoredox catalyzed hydrotrifluoromethylthiolation of unactivated alkenes

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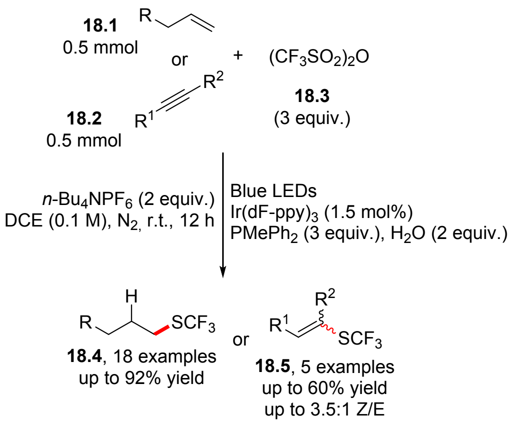
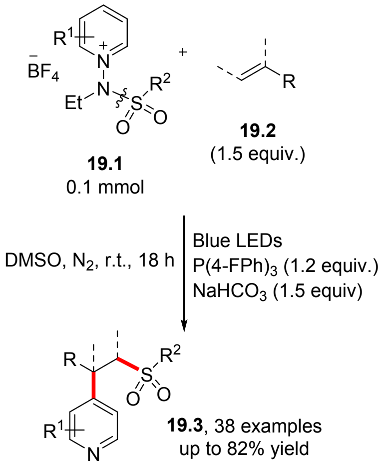
(**18.1**) or alkynes (**18.2**) by using trifluoromethanesulfonic anhydride (Tf_2O) as the SCF_3 radical precursor, in the presence of H_2O as the proton source (Scheme 18).[56]



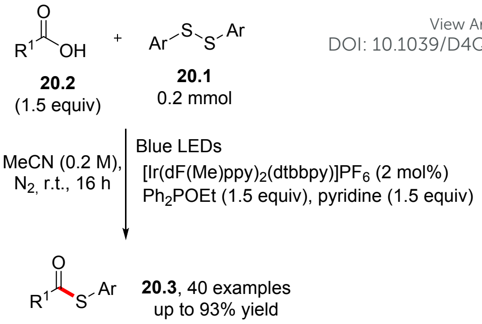
Scheme 18. Visible light mediated hydrotrifluoromethylthiolation of unactivated alkenes and alkynes.

The visible light-induced hydrosulfonylation of alkenes in the presence of *N*-amidopyridium salts **19.1** as precursors of sulfonyl radical which has been recently proposed in a photocatalyst free fashion, via the formation of a photoreactive EDA complex between **19.1** and triphenyl phosphine (or HCO_3^- anion, Scheme 19). Notably, the protocol was extended to the functionalization of bioactive molecules including estrone and fructopiranose.[57]



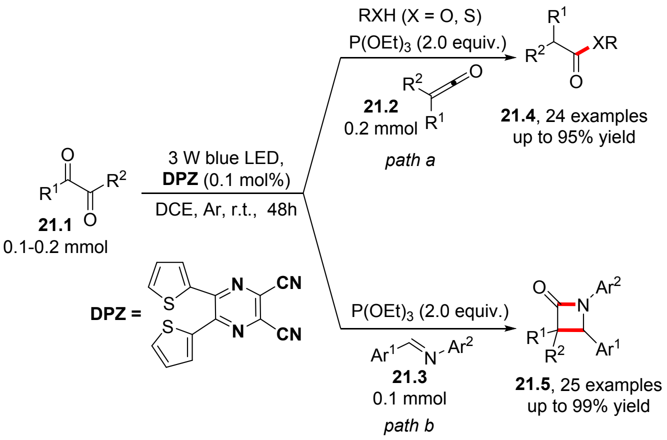
Scheme 19. 1,2-difunctionalization of olefins via blue light driven C-aryl and C-S bond formation**.**

Disulfides **20.1** have been employed as sulfur source in the photoredox catalyzed, alkoxy phosphine-mediated thioesterification of carboxylic acids **20.2** that was successfully applied to the functionalization of complex pharmaceutical-derived carboxylic acids including cholesteryl ester transfer protein inhibitor Dalcetrapib (Scheme 20).[58]



Scheme 20. Phosphine-mediated thioesterification of carboxylic acids **20.2**.

In 2021, Jiang and co-workers reported the photoredox catalytic phosphite-mediated deoxygenation and Wolff rearrangement of 1,2-diketones via ketenes to afford the corresponding (thio)esters (**21.4**, Scheme 21, path a) by using Pyrazine derivative DPZ as the visible light absorbing photocatalyst.[59] The same approach is on the basis of the preparation of β-lactams **21.5** (path b) via a Staudinger [2+2] cycloaddition with imines.[60]



Scheme 21. Photoredox catalytic phosphite-mediated deoxygenation and Wolff rearrangement of α-diketones.

C-N bond formation. A photoredox catalyzed anti-*Markovnikov* hydroamination of olefins **22.2** by sulphonamides **22.1** in the presence of tricyclohexylphosphine and TRIP-SH as the cocatalysts was recently described (Scheme 22a).[61] As illustrated in Scheme 22b, the mechanism involves a merged photoredox/hydrogen Atom Transfer (HAT)/ phosphine organocatalysis cycle, where P-N bond α-cleavage occurring in the radical intermediate **22.4**, in turn generated by coupling of $PCy_3^{•+}$ with **22.1** plays a key role in the generation of the amido radical **22.5**, that is in turn trapped by **22.2**, to afford intermediate **22.6**. The latter undergoes hydrogen atom transfer with TRIP-SH to release the desired product.[61] A similar visible light driven anti-*Markovnikov* hydroamination of alkenes (including cyclic olefins and vinyl ethers) was carried out also by using *N*-hydroxyphthalimide as the functionalizing agent and triethyl phosphite as the cocatalyst.[62]

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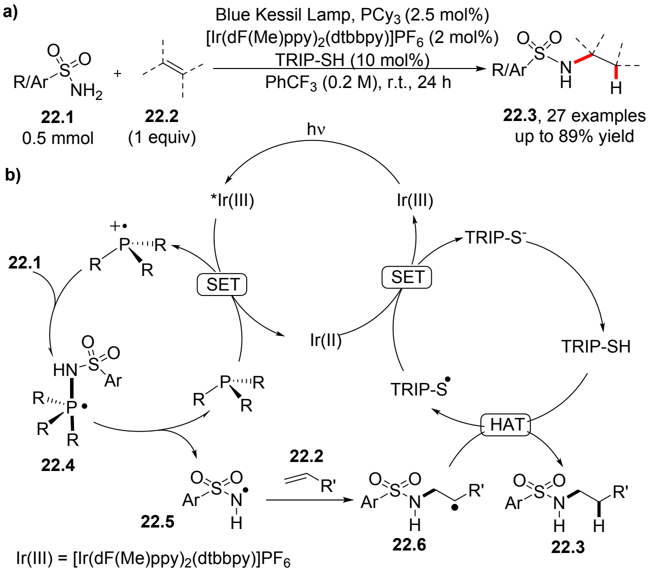
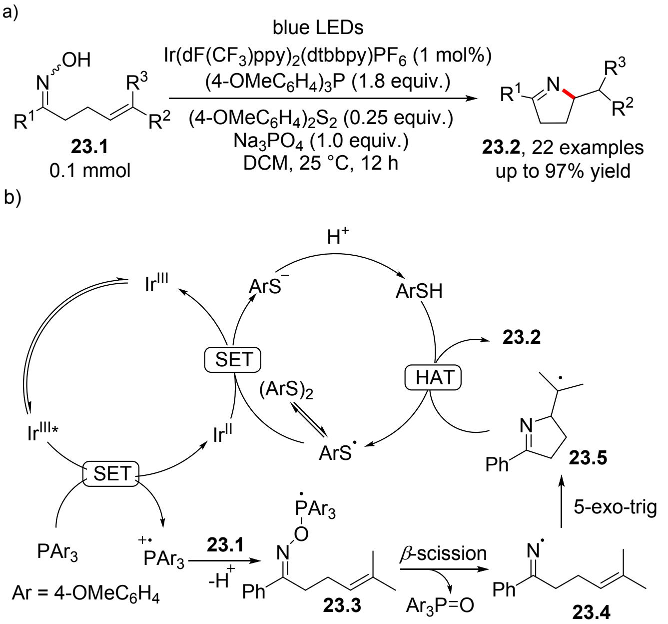


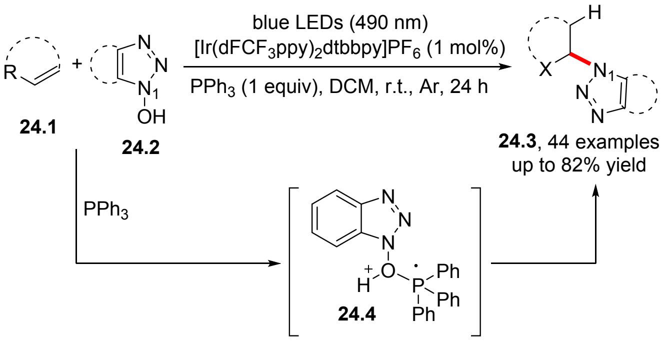
Scheme 22. Visible light driven anti-*Markovnikov* hydroamination of olefins.

The synthesis of pyrrolines from γ,δ-unsaturated oximes (Scheme 23) was recently described to take place in the presence of $Ir(dF(CF_3)ppy)_2(dtbbpy)PF_6$, tris(4-methoxyphenyl)phosphine and 4-methoxyphenyl disulphide as the photoredox catalysts and the cocatalysts, respectively.[63] Such strategy is based on the reductive quenching of the photoexcited iridium catalyst by tris(4-methoxyphenyl)phosphine ($E_{p/2}[Ar_3P^{\bullet+}/Ar_3P] = +0.87$ V vs. SCE), that is converted in the corresponding radical cation which add itself onto the oxime moiety. The so generated phosphoranyl radical **23.3** undergoes N-O bond β-cleavage, to deliver iminyl radical **23.4** that in turn undergoes intramolecular 5-exo-trig cyclization. Hydrogen atom transfer (HAT) from 4-methoxythiophenol (arising from the corresponding disulphide) to carbon centered radical **23.5** afford the desired product.



Scheme 23. Light driven intramolecular hydroimination of oximes **23.1**.

N-hydroxyl azoles **24.2** (Scheme 24) were successfully employed in the regioselective hydroazolylation of alkenes under Iridium photoredox catalysis conditions, in the presence of triphenylphosphine. The N-O bond cleavage that occurs in the phosphoranyl radical cation **24.4** has been suggested as the key step in the reaction mechanism. An analogous mechanism was exploited for the preparation of *N*-arylphthalimides via photocatalyzed radical *N*-arylation of (hetero)aryl derivatives by *N*-hydroxyphthalimides.[64]



Scheme 24. The photoredox catalyzed hydroazolylation of alkenes.

The development of innovative strategies for the amidation of carboxylic acids is yet of interest in organic synthesis, due to the widespread presence of amide moieties in fine chemicals, in peptides as well in polymeric materials.[65-70]

In this context, we noticed the preparation of differently substituted amides via a merged photoredox/cobaloxime catalyzed deoxygenative activation of carboxylic acid.[71] The protocol, that requires PPh_3 as the redox active cocatalyst, has been performed on different biorelevant substrates and was considered for late-stage functionalization. Furthermore, the process has been optimized also under continuous flow conditions, for the preparation of peptides in satisfactory yield (Scheme 25).[71,72] Carboxylic acids have been used also as acyl radical precursor in the preparation of differently substituted acyl hydrazides by deoxygenative hydroacylation of azobenzenes.[73]

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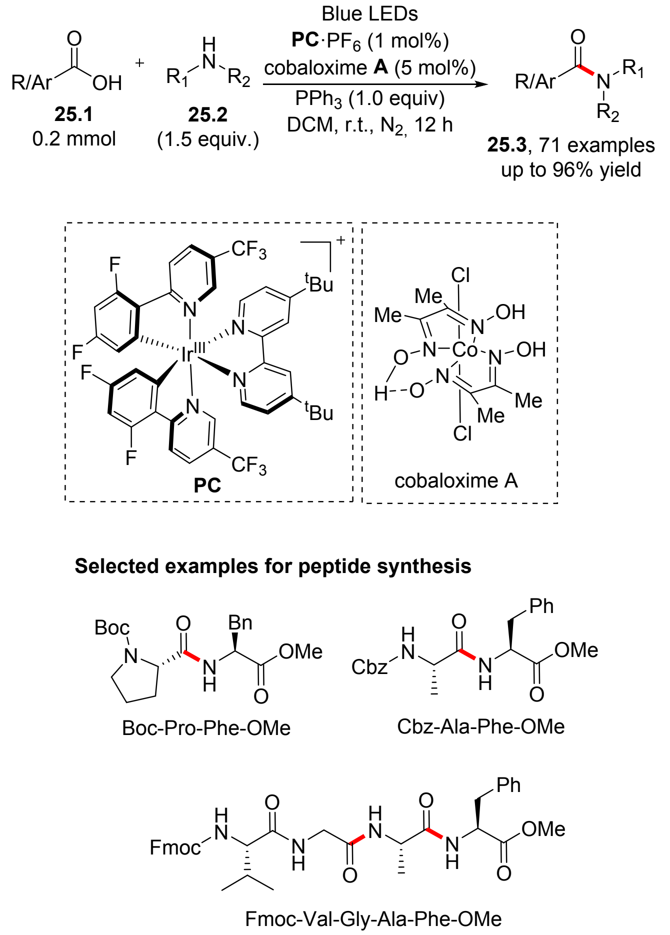
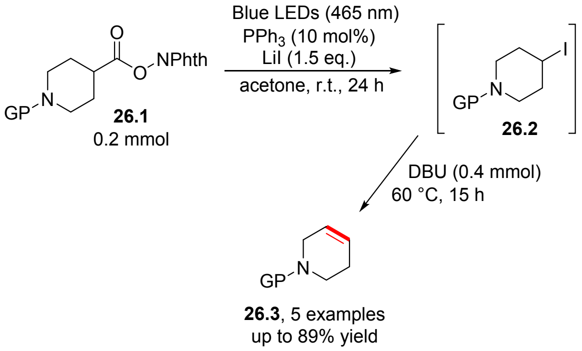


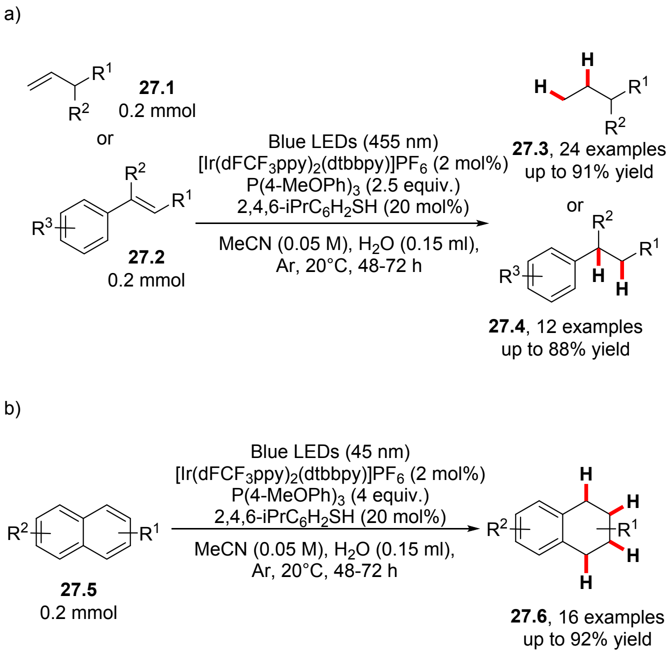
Scheme 25. Merged photoredox and cobaloxime preparation of amides via deoxygenative amidation of carboxylic acids.

Carbon-halogen bond formation. Carboxylic acid derivatives have been employed in the preparation of halogenated substrates via acyl radical. *N*-(acyloxy)phthalimides have been subject to triphenylphosphine photocatalyzed iododecarboxylation by using Lithium Iodide as the iodine source, to afford a wide range of both cyclic and acyclic alkyl iodides (**26.2**, Scheme 26).[74] Notably, when an organic base (DBU) is added, **26.2** undergoes elimination to afford selectively the corresponding alkene in a one pot fashion.[74]



Scheme 26. Triphenylphosphine photocatalyzed iododecarboxylation of *N*-(acyloxy)phthalimide.

Carbon-Hydrogen bond formation. In 2023, Studer and co-workers established the photocatalyzed activation of water by the interaction of phosphine radical, which could provide H atom to further reacted with alkenes (**27.1** or **27.2**) under mild condition (Scheme 27).[75] This transfer hydrogenation methodology have been applied also to the reduction of naphthalenes **27.5**. Furthermore, photochemical skeletal editing of 2-substituted quinolines can be achieved to afford the 2,3-disubstituted indoles via a hydrogenative rearrangement in slightly modified conditions.[75]



Scheme 27. The photocatalyzed activation of water and further application.

Conclusions

In recent years, radical phosphine chemistry has emerged as an elegant and prominent strategy for broadening the horizons of (visible) light driven methodology, with attention to the design of late stage functionalization protocols for pharmaceutical design and bioactive and natural molecules synthesis. Despite the nice advancements reported in literature, however, there are still several drawbacks that has not been solved yet, since the design of stereoselective processes is still underdeveloped and the use of triarylphosphines in stoichiometric excess[56,75] involves the release of rather noxious phosphorous based by-products. In this context, researches should see to methodologies that employ trisubstituted phosphines as effective organocatalysts (e.g. in the cases where an EDA complex with an electron poor alkyl halide is formed)[28] as a promising strategy to overcome these limitations.

Conflicts of interest

There are no conflicts to declare.

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Data Availability Statement

No primary research results, software or code have been included and no new data were generated or analysed as part of this review.