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Advances in Contemporary Research

Visible-light photocatalyzed radical multicomponent reactions in organic synthesis: A short review

Debasree Saha*[a] & Brindaban C Ranu*[b]

[a] Department of Chemistry, Raidighi College (Affiliated to University of Calcutta), 24 Parganas (South), West Bengal 743 383, India
[b] School of Chemical Sciences, Indian Association for the Cultivation of Science, Jadavpur, Kolkata 700 032, India
E-mail: debasree.saha@raidighicollege.org.in, debasree27@gmail.com, ocbcr@iacs.res.in, bcranu@gmail.com

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This review presents recent advancements in multicomponent reactions driven by visible light photocatalysis, encompassing both metal-free and transition-metal-catalyzed strategies. It explores current challenges, promising opportunities, future directions, and real-world applications in the visible-light-mediated synthesis of organic and bioactive molecules. Practical aspects including room-temperature protocols, continuous-flow synthesis, and scalable green methodologies are discussed. By unifying these transformations under a mechanistic framework, this review provides a comprehensive and methodology-centered perspective, complementing recent heterocycle-focused reviews. Finally, this work aims to propel further innovation in photocatalytic methodologies for constructing valuable organic frameworks.

Keywords: Visible light, Multicomponent reactions, Photoinduced process, Sustainable operation

Multicomponent reactions have always been an integral part of organic synthesis as they offer atom and step economies, greater efficiency, lower energy and reduced time consumption and greater structural diversity. As such, multicomponent reactions have wide applicability in pharmaceutical and agricultural industries[1-11].

Photocatalysis in organic synthesis has emerged as one of the most important tools in the last decade as it provides a plethora of advantages like sustainable and mild reaction conditions, easy to perform methodologies, convergent atom economical approach, broader substrate scope and chemoselectivity[12-19].

Photocatalysis usually follows three distinct pathways.. Pathway one involves a photoredox catalyst, pathway two features a photoredox catalyst and a co-catalyst which may or may not be transition metal. The third pathway is visible light induced transition metal catalysis which mandates the presence of transition metal. Several reviews have highlighted the use of these pathways in organic synthesis[20-32]. Visible-light-mediated multicomponent reactions have also appeared in a few recent reviews[18,33-35]. However, many of these are either extensive and descriptive, focus predominantly on heterocycle synthesis, highlight a particular class of product or discuss multicomponent reactions as part of broader photoredox catalysis overviews.

In contrast, in this review we have adopted a concise and focused approach, emphasizing methodological development rather than product classification. The review deals with systematic organized representative multicomponent transformations — including Ugi-type reactions, trifluoromethylation, dithiocarbamate synthesis, carbofunctionalization of conjugated dienes, and fluorination of secondary amines — within a unified radical mechanistic framework. This approach facilitates a clearer understanding of reaction design principles and visible-light-induced radical pathways across different transformations.

Given the growing emphasis on sustainable chemistry, we propose to categorize the methodologies based on the involvement of transition metal photocatalysts. This classification will allow readers to readily identify more sustainable, metal-free approaches, while still acknowledging the contributions of transition metal-based systems in this field.

Accordingly, the review will be structured under two principal categories: (1) multicomponent reactions under transition metal-free visible light photocatalysis, and (2) multicomponent reactions under visible light-induced transition metal catalysis. The efficiency, merits and demerits of both the procedures will be discussed along with their potential for future synthetic applications.

Multicomponent reactions under transition metal free visible light photocatalysis

Transition metal free organic synthesis has attracted significant attention due to the economic feasibility and environmental sustainability of the reaction methodology. Moreover, the final product has zero chances of metal contaminant which is a great advantage especially in case of pharmaceutical industries. In this section a few metal free visible light photocatalyzed multicomponent reactions will be discussed.

In 2013, Rueping and his group developed a continuous flow procedure for the α-functionalization of tertiary amines under visible light photocatalysis[36]. Rose Bengal served as the organo-photocatalyst which effectively catalyzed the multicomponent reaction leading to the formation of C-C and C-P bond. Nitroalkanes, TMSCN, dialkyl malonates, and dialkyl phosphites were made to react with several N-aryl tetrahydroisoquinolines to produce the corresponding products in quite high yields. In the same pattern Ugi multicomponent reaction was carried out with N, N, dimethyl anilines1, isocyanides 2 and water to produce the desired products 3 efficiently (Scheme 1).

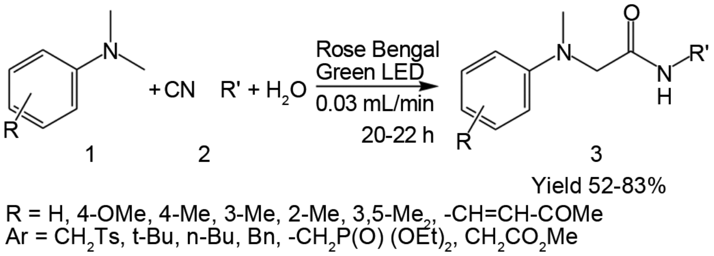
The authors employed a reasonably inexpensive organic dye like Rose Bengal for the operation. The methodology has several advantages like use of inexpensive reagents and photocatalyst, transition metal free sustainable phenomenon and generation of products in significantly high yields.

Xiao and co-workers employed Fluorescein dye as a photoredox catalyst for the alkoxycarboxylation of aryl diazonium salts 4 using CO gas under visible light (Scheme 2)[37]. The reaction is carried out at room temperature with very low loading of the fluorescein photocatalyst without the presence of any metal, thus making the protocol highly environment friendly. Several aryl diazonium salts 4 participated in the reaction leading to a range of product esters 5 in good yields. The protocol was extended successfully to include reaction of aryl diazonium salts with chiral alcohols resulting in chiral products.

In 2020, a phenothiazine core based conjugated microporous polymer (CMP CSU8) was developed by Yu and his group who applied it as a photoredox catalyst for the Ugi type multicomponent reaction between dimethylanilines 7, isocyanides 8 and water/carboxylic acid **9** (Scheme 3)[38]. A wide array of substituted dimethylanilines participated in the reaction to produce the corresponding products **10** in reasonably high yields.

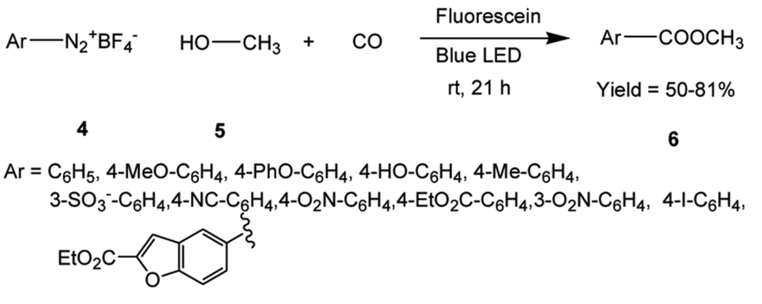
The authors also extended the methodology towards selenation of indoles. The CMP catalyst used was found to be highly efficient as well as reusable in successive cycles.

He, Zhao, Wei and co-workers developed a metal free visible light promoted method for the synthesis of S-alkyl-dithiocarbamates **14** through the multi-component reaction of α-diazoesters **11**, amines **12** and carbon disulphide **13** in 1,4-dioxane solvent



R = H, 4-OMe, 4-Me, 3-Me, 2-Me, 3,5-Me_2, -CH=CH-COMe
Ar = CH_2Ts, t-Bu, n-Bu, Bn, -$CH_2P(O)(OEt)_2$, CH_2CO_2Me

Scheme 1 — Ugi multicomponent reaction under metal free visible light photocatalysis



Ar = C_6H_5, 4-MeO-C_6H_4, 4-PhO-C_6H_4, 4-HO-C_6H_4, 4-Me-C_6H_4, 3-SO_3^--C_6H_4,4-NC-C_6H_4,4-O_2N-C_6H_4,4-EtO_2C-C_6H_4,3-O_2N-C_6H_4, 4-I-C_6H_4,

Scheme 2 — Alkoxycarbonylation of aryl diazonium salts through visible light photocatalysis

(Scheme 4)[39]. Several diversely substituted α-diazoesters successfully participated in the reaction. Both electron withdrawing and electron donating groups on the phenyl ring worked well. On the amine side, both cyclic and acyclic amines were compatible with the reaction leading to the formation of a range of dithiocarbamates. The authors also detected the formation of a byproduct **15** in trace amount in the four component coupling using dioxane as solvent.

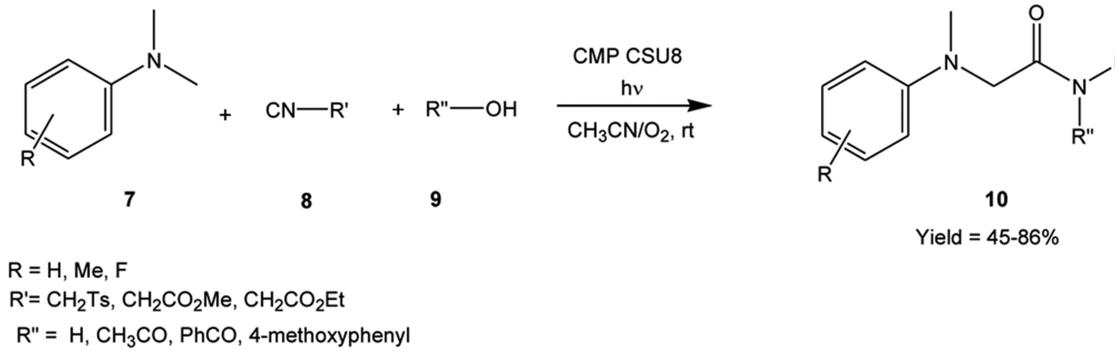
Strikingly, when the reaction was carried out in THF, four component coupling products were obtained in quite good yields which could be utilized for synthetic purposes. This serves as an added bonus in the potential applications of the methodology.

Recently, in 2024 Shen and his group presented a novel method for the synthesis of trifluoromethylated 3-indolequinoxalin-2(1H)-ones **19** *via* the three component reaction of quinoxalin-2(1H)-ones **16**, indoles **17**, and CF_3SO_2Na **18** under photocatalysis in mild acidic condition (Scheme 5)[40]. A library of trifluoromethylated 3-indolequinoxalin-2(1H)-ones was successfully obtained.

The methodology could be scaled up and carried out in gram scale which depicted the practicability of the protocol.

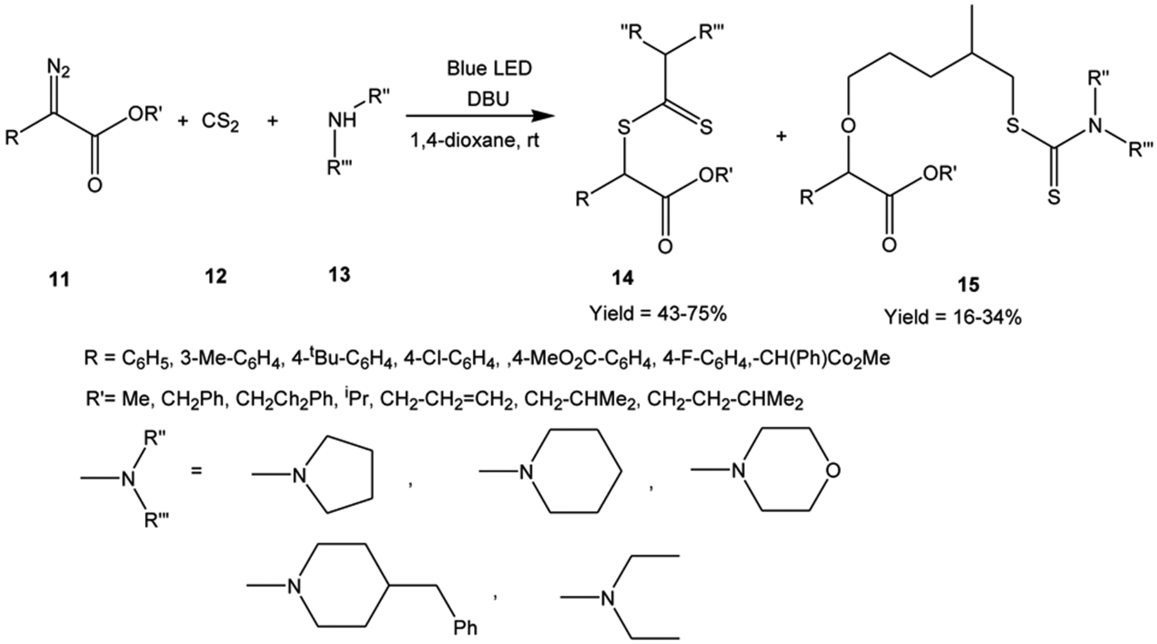
Multicomponent reactions under visible light induced transition metal catalysis

Recently, visible light induced transition metal catalysis has received much attention[17]. The transition metal complex carries out a dual purpose of reaping photon energy as well as initiating the bond making/bond breaking process. In this subsection we will discuss the recent advances of this particular emerging field in the catalysis of multicomponent reactions.



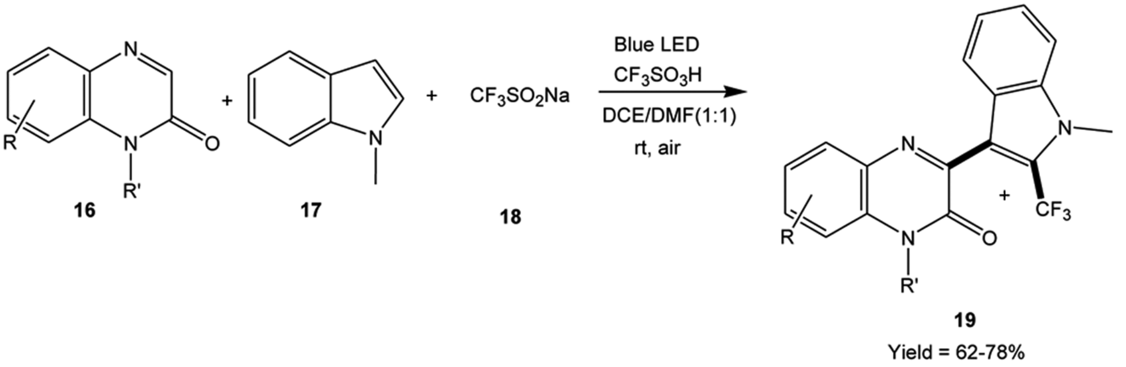
R = H, Me, F
R'= CH_2Ts, CH_2CO_2Me, CH_2CO_2Et
R" = H, CH_3CO, PhCO, 4-methoxyphenyl

Scheme 3 — Ugi multicomponent reaction catalysed by conjugated microporous polymers under visible light photocatalysis



R = C_6H_5, 3-Me-C_6H_4, 4-^{t}Bu-C_6H_4, 4-Cl-C_6H_4, ,4-MeO_2C-C_6H_4, 4-F-C_6H_4,-CH(Ph)Co_2Me
R'= Me, CH_2Ph, CH_2Ch_2Ph, ^{i}Pr, CH_2-CH_2=CH_2, CH_2-$CHMe_2$, CH_2-CH_2-$CHMe_2$

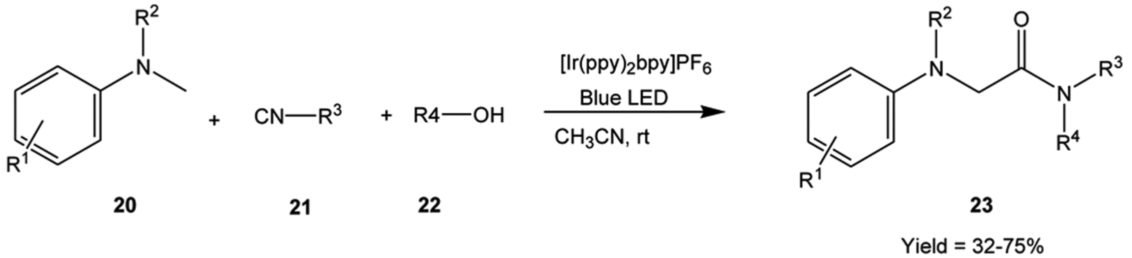
Scheme 4 — Metal free visible light promoted synthesis of dithiocarbamates through multicomponent reaction



R = H, Me, OMe, Me_2,F, Cl

R'= Me, Et, ^nPr, CH_2-CH=CH_2, CH_2-C≡H, CH_2Ph

Scheme 5 — Synthesis of trifluoromethylated 3-indolequinoxalin-2(1*H*)-ones *via* multicomponent reaction under visible light photocatalysis



R^1= H, 4-Me, 3-Me, 3,5-Me_2, 4-Cl, 4-Br, 3-Br

R^2 = Me, Et

R^3 = CH_2Ts, Bu, cyclohexyl, CH_2CO_2Me, CH_2Ph

R^4 = H, COPh, CO(1-naphthyl), CO(3-furyl), COBn, CO(3-fluorophenyl),CO(3-fluorophenyl), CO(CH_2-propargyl)

Scheme 6 — Ugi multicomponent reaction under visible light induced Iridium metal catalysis

Rueping *et al*. reported synthesis of α-amino amides/imides **23** through oxidative multicomponent reaction of dialkyl anilines **20**, isocyanides **21** and carboxylic acids/water **22** under visible light photocatalysis using Iridium complex as photoredox catalyst (Scheme 6)[41]. Both electron donating as well as electron withdrawing substituents on the aniline moiety were compatible with the procedure resulting in the corresponding products in reasonably good yields. Various isocyanides worked well thus depicting the versatility of the methodology.

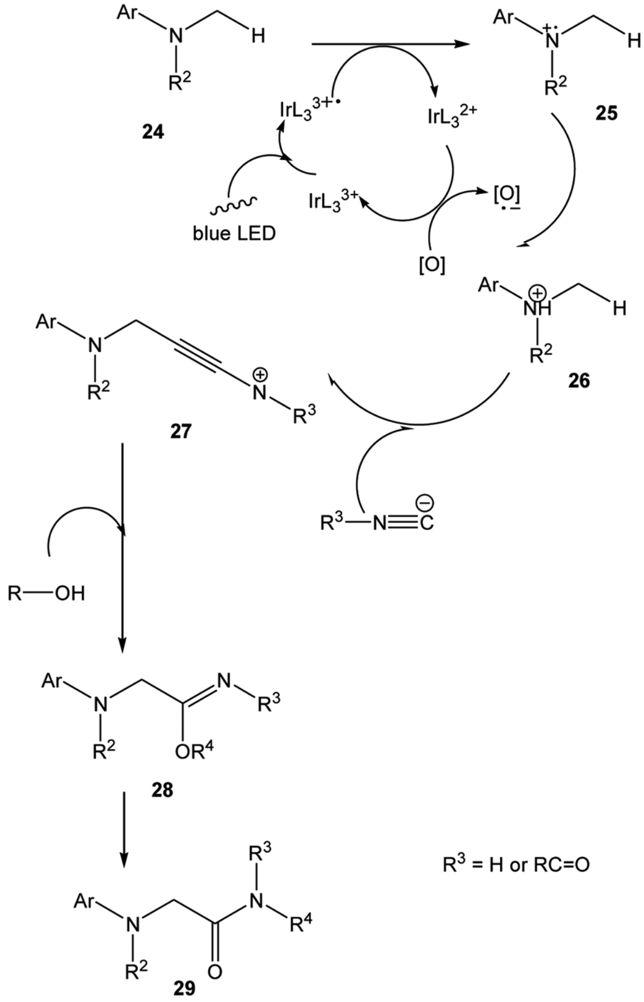
The authors proposed a plausible mechanism (Scheme 7) for the reaction *via* SET (single electron transfer) oxidation which involved the formation of a nitrilium ion intermediate **27**.

In 2015 Feng and his group employed ruthenium bipyridyl complex as a photoredox catalyst for the sp^3 C-H bond functionalization of 1,2,3,4 tetrahydroisoquinolines *via* three component Ugi type reaction under visible light in aerobic conditions (Scheme 8)[42]. Differently substituted 1,2,3,4 tetrahydroisoquinolines **30**, isoinitriles **31** and carboxylic acids **32** were compatible with the reaction providing high yields of the corresponding products **33**.

CH_3CN served as a crucial solvent and excellent yields were obtained when CH_3CN was used as solvent in comparison to toluene, dichloromethane or THF.

Titanium dioxide was employed as a photocatalyst by Rueping and his group for the C-H activation of tertiary amines **34** resulting in a variety of α-amino acids **36** (Scheme 9)[43]. The methodology proceeded *via* visible light mediated Ugi-type three component reaction. Several substituted N,N-dimethylanilines were well tolerated in the reaction leading to a wide range of products. A variety of isocyanides bearing functional groups likes esters and phosphonates participated successfully in the reaction.

The titanium dioxide catalyst could easily be separated by centrifugation and recycled for five cycles without significant loss in catalytic activity or selectivity, thus making the protocol environmentally as well as economically sustainable.

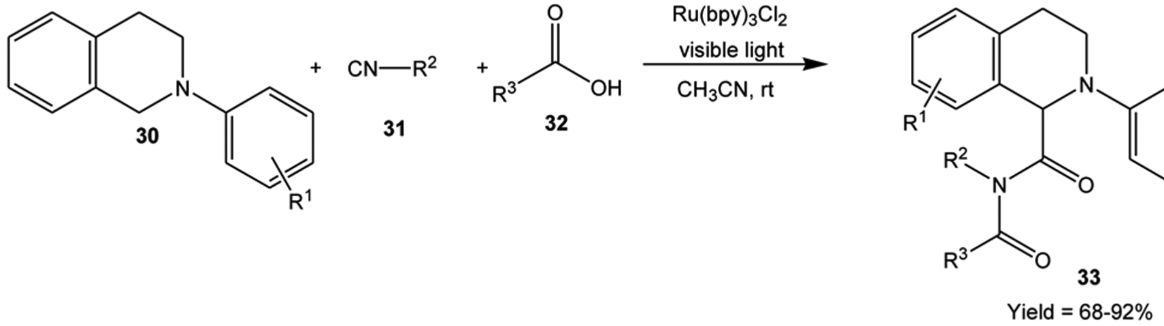


Scheme 7 — Plausible mechanism for Ugi multicomponent reaction under visible light induced Iridium metal catalysis

In 2020, Gevorgyan and his co-workers reported multicomponent reaction of 1,3-dienes **37**, alkyl iodides **38**, and amines **39** resulting in the formation of a broad range of allylic amines *via* photoinduced palladium catalyzed carbofunctionalization of conjugated dienes (Scheme 10)[44]. Several aryl and hetero-aryl substituted dienes were compatible with the reaction. Both electron rich as well as electron deficient dienes participated in the reaction though yields were found to be better for the former. Alkyl substituted dienes also underwent the reaction. A variety of alkyl iodides and amines reacted quite well leading to a range of 1,2-carboamination products **40**.

The authors also extended the protocol to oxygen and carbon nucleophiles such as phenol and malonate derivatives leading to the formation of the corresponding carbofunctionalization products. The methodology was also applied for synthesizing drug analogues.

Wu and co-workers employed an iridium catalyst for the visible light induced multicomponent reaction of aromatic aldehydes **41**, amines **42** and sodium difluoromethanesulfinate **43** leading to the formation of fluorinated secondary amines **44** at room temperature (Scheme 11)[45]. The methodology provides a convenient way to introduce difluoromethyl group into imines. Various substituted aromatic aldehydes and amines participated in the reaction, thus leading to a diverse range of fluorinated secondary amines. The synthetic utility of the protocol was demonstrated by the gram scale application of the reaction. The reaction

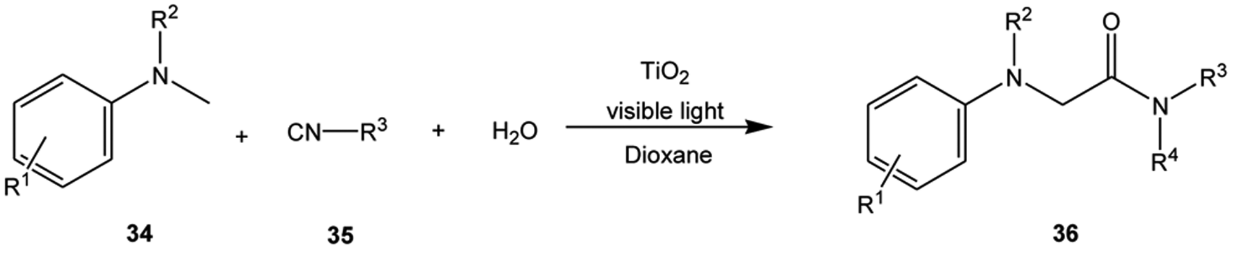


R^1= H, 4-OMe, 3-OMe, 4-Me, 3-F, 4-cl, 4-Br

R^2 = Cy, Bn, 4-MeO-C_6H_4, 4-NC-C_6H_4,4-Cl-C_6H_4,3-Cl-C_6H_4,4-Br-C_6H_4,4-F_3CO-C_6H_4

R^3 = Me, -CH=CH_2, -CH=Ch-Ph, 2-I-C_6H_4, 2-MeO-C_6H_4, 4-MeO-C_6H_4,3-F_3O-C_6H_4, 2-Br-4-F-C_6H_3, 3-O_2N-C_6H_4
4-(CH=CH_2)-C_6H_4, 3-pyridyl, 2-furyl

Scheme 8 — Multicomponent reaction under visible light induced Ruthenium metal catalysis



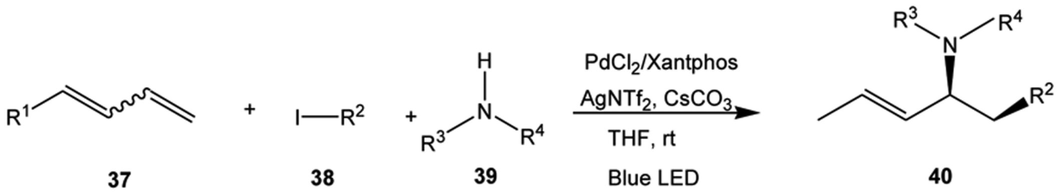
Yield = 51-82%

R ^{1}= H, 4-Me, 3-Me, 3,5-Me$_2$, 4-Cl, 4-Br, 3-Br, 4-COPh, 4-CH=CH-CHO

R^2 = Me, Et

R^3 = CH$_2$Ts, Bn, CH$_2$CO$_2$Me, CH$_2$P(O)(OEt)$_2$

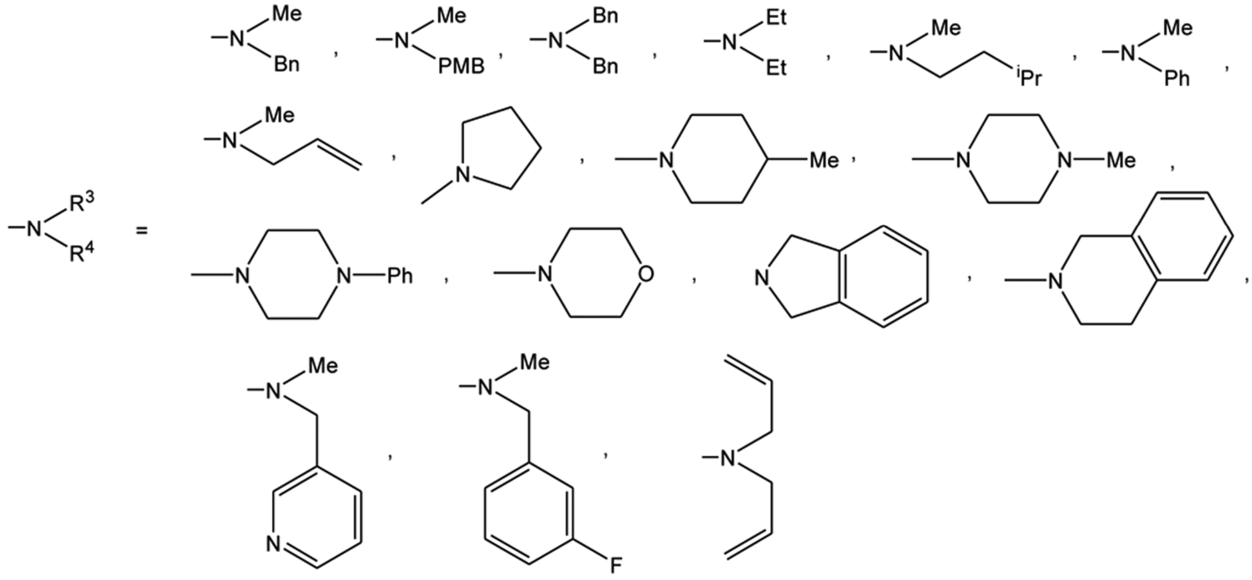
Scheme 9 — C–H functionalization of tertiary amines under visible light induced Titanium metal catalysis



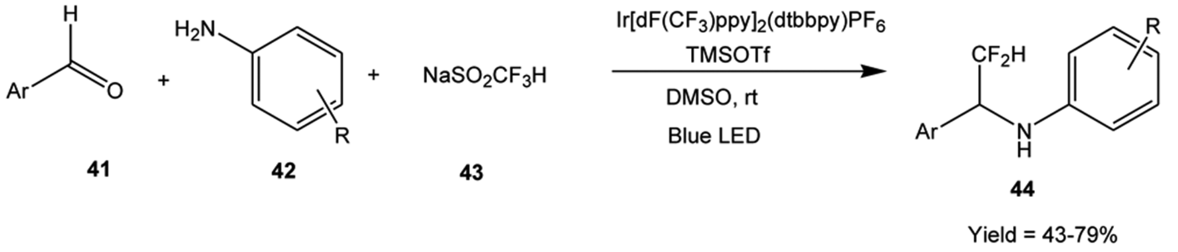
Yield = 30-83%

R^1= 4-MeO-C$_6$H$_4$, 4-Me-C$_6$H$_4$, 4-HO-C$_6$H$_4$, 5-(2,3-dihydrobenzofuran), 4-Me$_2$N-C$_6$H$_4$, 4-F$_3$C-C$_6$H$_4$, 4-O$_2$N-C$_6$H$_4$, 4-NC-C$_6$H$_4$, 4-(trimethylsilylacetylide)-C$_6$H$_4$, 4-F-C$_6$H$_4$, 4-Cl-C$_6$H$_4$, 4-Br-C$_6$H$_4$, 3-Me-C$_6$H$_4$, 2-Me-C$_6$H$_4$, 2-Ph-C$_6$H$_4$, 2,4,6-Me$_3$-C$_6$H$_2$, 2-Ferrocenyl,3-Pyridyl, 2-Pyridyl, 3-(N-methyl idolyl), 3-(N-Boc-inidolyl),3-benzothienyl, cyclohexyl, n-Pentyl, Ph-CH=CH$_2$-, Heptenyl, Cinnamyl, CO$_2$Et, trans-Ph-CH-C(Me)

R^2 = CH$_2$-CH$_2$-TMS, CH$_2$-TMS, CH$_2$-SiMe$_2$Ph, CH$_2$-SiMe$_2$(O^{t}Bu),CH$_2$-SiMe$_3$, CH$_2$-^{t}Bu, CH$_2$-Cp, CH$_2$-^{i}Pr, CH$_2$-Bu, CH$_2$-(TMS)$_2$, ^{t}Bu, Cp, Cy, Oxetanyl, tetrahydropyranyl, N-Boc-piperidinyl



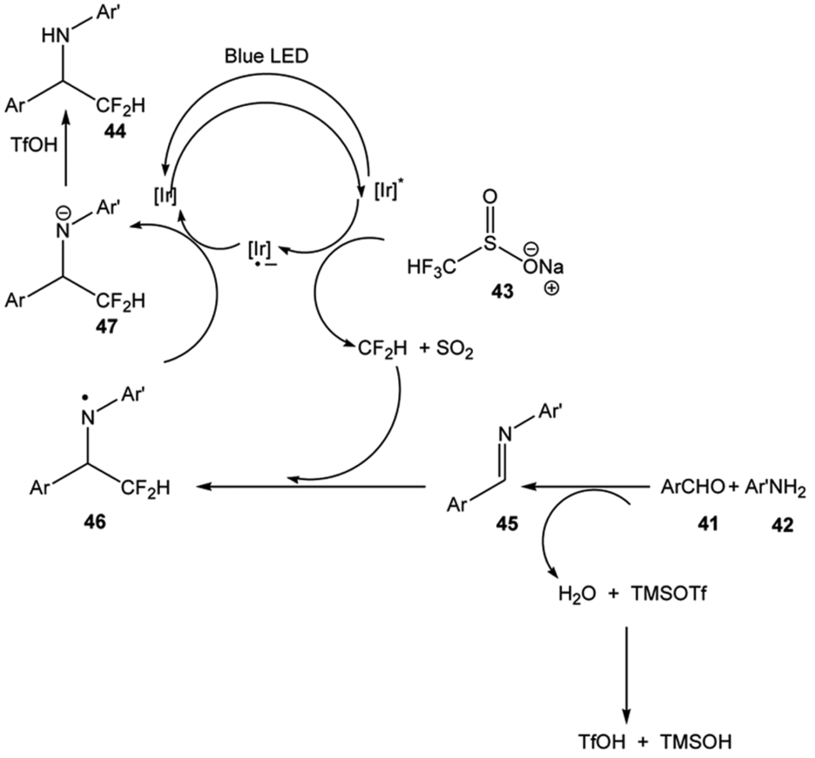
Scheme 10 — Palladium catalysed carbofunctionalization of conjugated dienes under visible light



Ar = 4-Me-C_6H_4, 3-Me-C_6H_4, 2-Me-C_6H_4, 4-^{t}Bu-C_6H_4, 4-MeO-C_6H_4, 4-HO-C_6H_4, 4-O_2N-C_6H_4, 4-F-C_6H_4, 4-Cl-C_6H_4, 4-Br-C_6H_4, 1-naphthyl, 3-pyridyl

R = 4-Me, 3-Me, 2-Me, 4-OMe, 4-CN, 4-CF_3, 4-CO_2Me, 4-OCF_3, 4-F, 4-Cl, 4-Br, 3,4-Me_2, 4 Ph

Scheme 11 — Synthesis of fluorinated secondary amines *via* multicomponent reaction under visible light induced Iridium metal catalysis



Scheme 12 — Plausible mechanism for synthesis of fluorinated secondary amines *via* multicomponent reaction under visible light induced Iridium metal catalysis

was also found useful for the late stage synthetic modification of biologically active compounds.

The authors proposed a plausible mechanism (Scheme 12) for the reaction based on control experiments where the reaction has been depicted to proceed *via* single electron transfer leading to the formation of aminyl radical **46** which generates the product **44** *via* subsequent reduction followed by protonation.

Conclusion

Visible light-driven chemical reactions have emerged as powerful tools in synthetic chemistry, offering enhanced efficiency and enabling the construction of mechanistically novel synthetic pathways to access target molecules that are often unattainable *via* traditional thermal methods. Modern synthetic strategies are increasingly focused on developing milder, greener reaction conditions to build complex molecular architectures in an environmentally sustainable manner.

This review explores both the advantages and challenges associated with visible light-induced multicomponent reactions. It presents a comprehensive overview of metal-free and transition metal-catalyzed photoinduced processes, highlighting their scope and impact.

The key photocatalytic transformations are examined, including the Ugi reaction, alkoxycarbonylation of diazonium salts, synthesis of dithiocarbamates, trifluoromethylation, carbofunctionalization of conjugated dienes, and fluorination of secondary amines. These examples showcase the breadth of visible light-enabled multicomponent methodologies.

Although still an emerging field, visible light photocatalysis holds significant promise in drug discovery, particularly in facilitating the synthesis of complex heterocyclic frameworks with therapeutic potential. Furthermore, the adoption of continuous flow chemistry has begun to address the limitations of scaling up photoredox reactions efficiently. Room-temperature, light-mediated synthesis using readily available solvents offers a compelling route to gram-scale production of structurally sophisticated heterocycles.

Looking ahead, a good mechanistic understanding of these transformations is essential. Detailed investigations will aid in elucidating reaction pathways and accelerate the development of novel photocatalytic strategies for heterocycle construction.

Ultimately, this review aspires to serve as a valuable resource for researchers engaged in sustainable synthesis under visible light, reflecting the convergence of innovation, efficiency, and environmental consciousness.

Acknowledgements

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Conflict of Interest

The authors declare that there is no conflict of interest related to this article.

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