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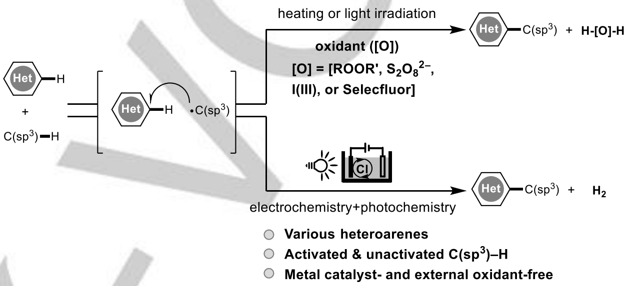
Scalable Photoelectrochemical Dehydrogenative Cross-Coupling of Heteroarenes with Aliphatic C–H Bonds

Pin Xu†, Peng-Yu Chen†, Hai-Chao Xu*

Abstract: Heteroarenes are structural motifs found in many bioactive compounds and functional materials. Dehydrogenative cross-coupling of heteroarenes with aliphatic C–H bonds provides straightforward access to functionalized heteroarenes from readily available materials. Established methods employ stoichiometric chemical oxidants under conditions of heating or light irradiation. By merging electrochemistry and photochemistry, we have achieved efficient photoelectrochemical dehydrogenative cross-coupling of heteroarenes and C(sp^3)–H donors through H_2 evolution, without the addition of metal catalysts or chemical oxidants. Mechanistically, the C(sp^3)–H donor is converted to a nucleophilic C-radical through H-atom transfer with chlorine atom, which is produced by light irradiation of anodically generated Cl_2 from Cl^-. The C-radical then undergoes radical substitution to the heteroarene to afford alkylated heteroarene products.

Dehydrogenative C–H/C–H cross-coupling is one of the most powerful methods for forging C–C bonds because of its high step and atom economy, as well as the easy availability of starting materials.[1] In this context, there has been significant interest in the cross-coupling of heteroarenes with aliphatic C–H donors for the synthesis of functionalized heteroarenes (Scheme 1, top), which are prevalent motifs in pharmaceuticals, natural products and functional materials.[2] These methods generally involve a Minisci-type mechanism,[3] in which C–C bond formation is achieved by the addition of a carbon-centered radical to the heteroarene. The key C-radical intermediate can be generated from the aliphatic C–H donor through hydrogen atom transfer (HAT) using oxygen- or nitrogen-centered radicals, which are in turn produced by heat- or light-induced cleavage of a chemically labile oxidant such as peroxide (ROOR'),[4] persulfate ($S_2O_8^{2-}$),[5] I(III) reagent,[6] or Selectfluor.[7] However, the use of these chemical oxidants reduces the atom economy of the whole process and may cause safety concerns, especially for large scale production.[8]

Organic electrochemistry, which achieves redox transformations with traceless electricity instead of oxidants or reductants, has gained considerable traction in the past few years due to its inherent sustainability and tunability.[9] The simultaneously occurrence of anodic oxidation and cathodic proton reduction allows the dehydrogenative reactions to proceed through H_2 evolution, obviating the need for stoichiometric electron or proton acceptors. Although electrochemically-driven radical C–H fluoroalkylation and acylation of heteroarenes have been achieved,[10] an efficient electrochemical coupling of electron-deficient heteroarenes with various C(sp^3)–H donors remains unknown probably because both coupling partners are rather difficult to oxidize and electron-rich alkyl radicals are prone to oxidation into carbocations under typical electrochemical conditions.[11]



Scheme 1. Dehydrogenative cross-coupling of heteroarenes with aliphatic C–H bonds.

While photochemical dehydrogenative cross coupling via H_2 evolution have been reported, the requisite Co-based catalyst causes reduction and elimination of alkyl radicals, limiting the scope of the photochemical methods.[12] The merger of electrochemistry with photochemistry has the potential to unlock access to reactivities that are difficult to achieve with electrochemistry or photochemistry alone.[13] Very recently, Lambert and coworkers reported an elegant electrophotocatalytic cross-coupling of heteroarenes with ethers.[14] We have been interested in chemical-oxidant free Minisci-type reactions and have reported photoelectrochemical alkylation of heteroarenes with organotrifluoroborates and carboxylic acids.[15] The use of various C(sp^3)–H donors with different bond dissociation energies (BDE) as coupling partners in these reactions require the development of new mechanistic paradigms. We report herein an unprecedented photoelectrochemical method that achieves dehydrogenative cross-coupling of a wide variety of heteroarenes with various activated and unactivated C(sp^3)–H donors (Scheme 1, bottom).

The photoelectrochemical method that we employed in the current study involves the conversion of C(sp^3)–H to C-radicals by chlorine radicals (Cl•). The relatively large BDE of HCl (102 kcal mol^{-1}) ensures that Cl• can react with a wide range of activated and unactivated aliphatic C–H bonds (Scheme 2a).[16,17] Specifically, Cl• is first generated by the anodic oxidation of Cl^- and the subsequent light-irradiation promoted homolytic cleavage of the resultant Cl_2 (Scheme 2b). The C-radical generated through hydrogen atom transfer could then add to a protonated N-heteroaromatic compound to furnish a radical cation intermediate, which could then undergo rearomatization with the assistance of Cl•. The continuous anodic generation of low concentration of Cl_2 avoids the use of hazardous Cl_2 gas and reduces unwanted formation of alkyl chloride through Cl_2

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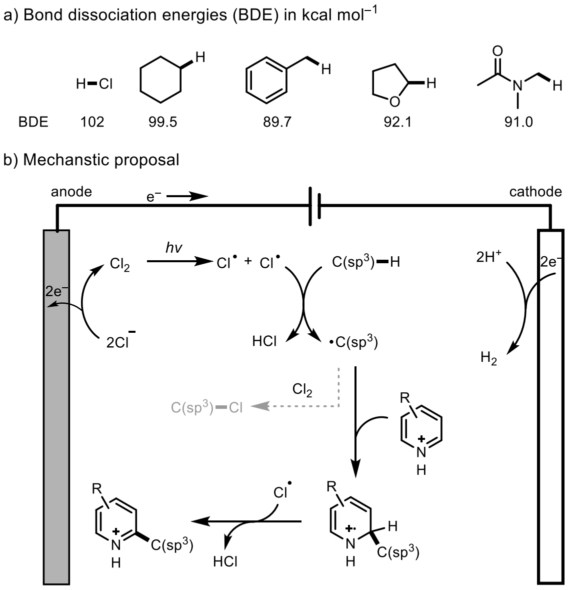
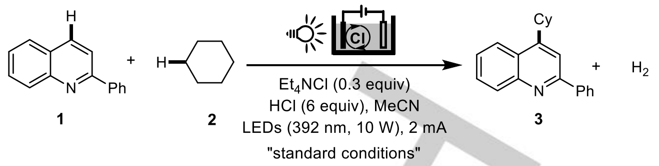
trapping of the alkyl radical.[18] The electrons and protons lost during the C–C cross-coupling are combined at the cathode to generate H_2.



Scheme 2. Proposed reaction design.

We first investigated the dehydrogenative cross-coupling of 2-phenylquinoline (**1**) and cyclohexane (**2**) as a model reaction (Table 1). After initial optimization, the desired product **3** was obtained in 92% yield by conducting the photoelectrochemical reaction under the irradiation of LEDs (392 nm, 10 W) and in an undivided cell equipped with a reticulated vitreous carbon (RVC) anode and a Pt cathode (entry 1). The reaction mixture consisted of Et_4NCl (0.3 equiv) and HCl (6 equiv) as chloride source and acid, respectively, in MeCN. Performing the reaction in the absence of light (entry 2), electricity (entry 3) or acid (entry 4) led to the complete abolition of product formation, whereas eliminating Et_4NCl (entry 5) resulted in a moderate drop in the yield of **3**. The match of the rate of the electrochemical process with the photochemical reaction is essential for optimal results. Increasing the wavelength and power of the LED light to 398 nm (20 W) or 450 nm (20 W) or the electric current to 4 or 6 mA similarly caused varying degrees of yield reduction (entries 6–9). In addition, lowering the amount of HCl to 2 equiv (entry 10) or cyclohexane to 5 equiv (entry 12), or replacing HCl with TFA (entry 11) were also found to be less optimal. Although stringent removal of oxygen was not necessary, running the reaction under air resulted in moderate yield reduction to 80% (entry 13).

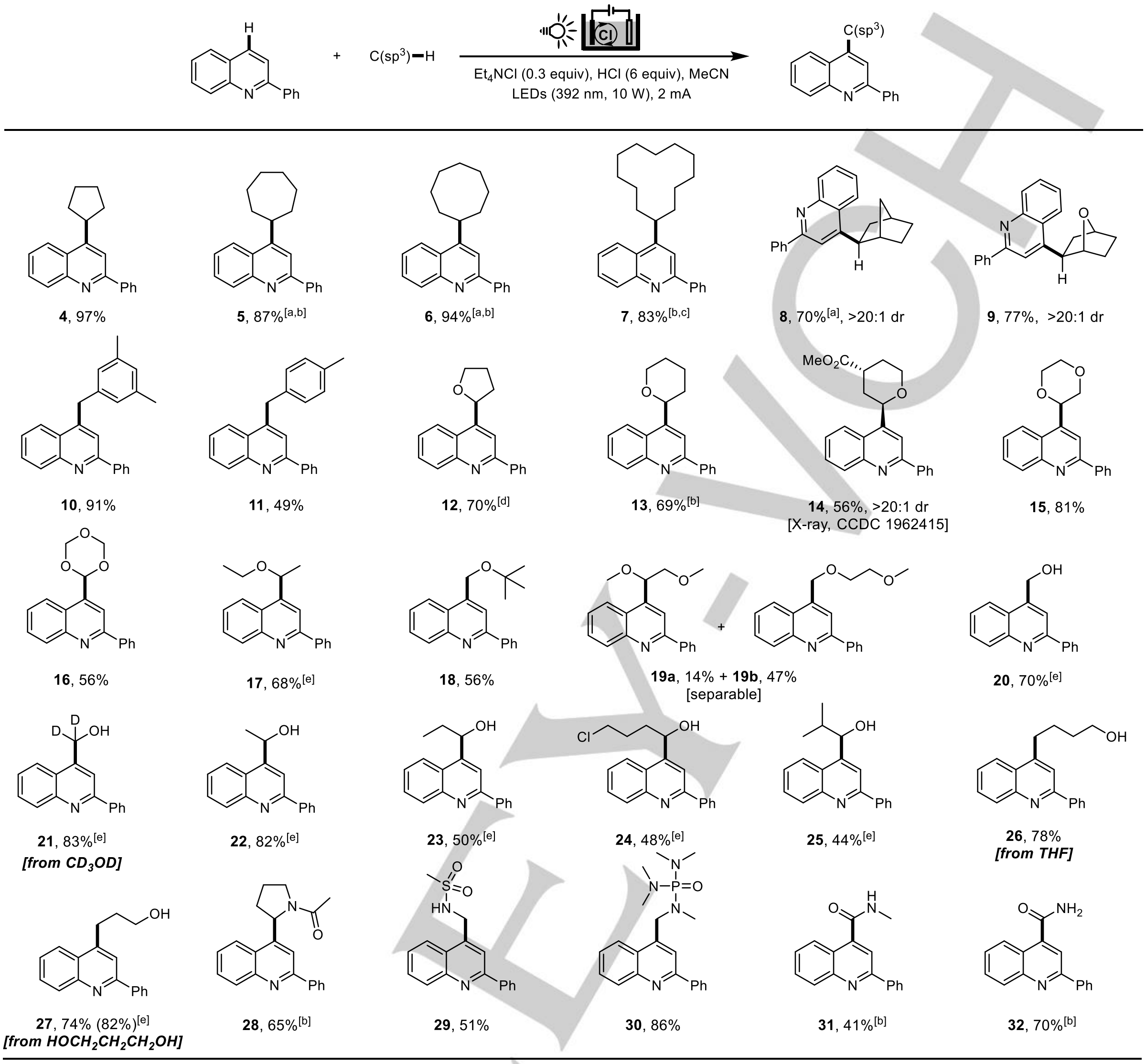
Table 1. Optimization of reaction conditions.[a]



Entry	Deviation from standard conditions	Yield [%][b]
1	none	92[c]
2	No electricity	0 (74)[d]
3	No LED	0 (84)[e]
4	No HCl	<5 (68)
5	No Et_4NCl	76 (17)
6	LEDs (398 nm, 20W)	84 (6)
7	LEDs (450 nm, 20W)	55 (34)
8	4 mA, 9 h	71 (13)
9	6 mA, 6 h	66 (17)
10	HCl (2 equiv)	87 (8)
11	TFA (2 equiv) instead of HCl	82
12	5 equiv of **2**	68 (9)
13	Under air	80

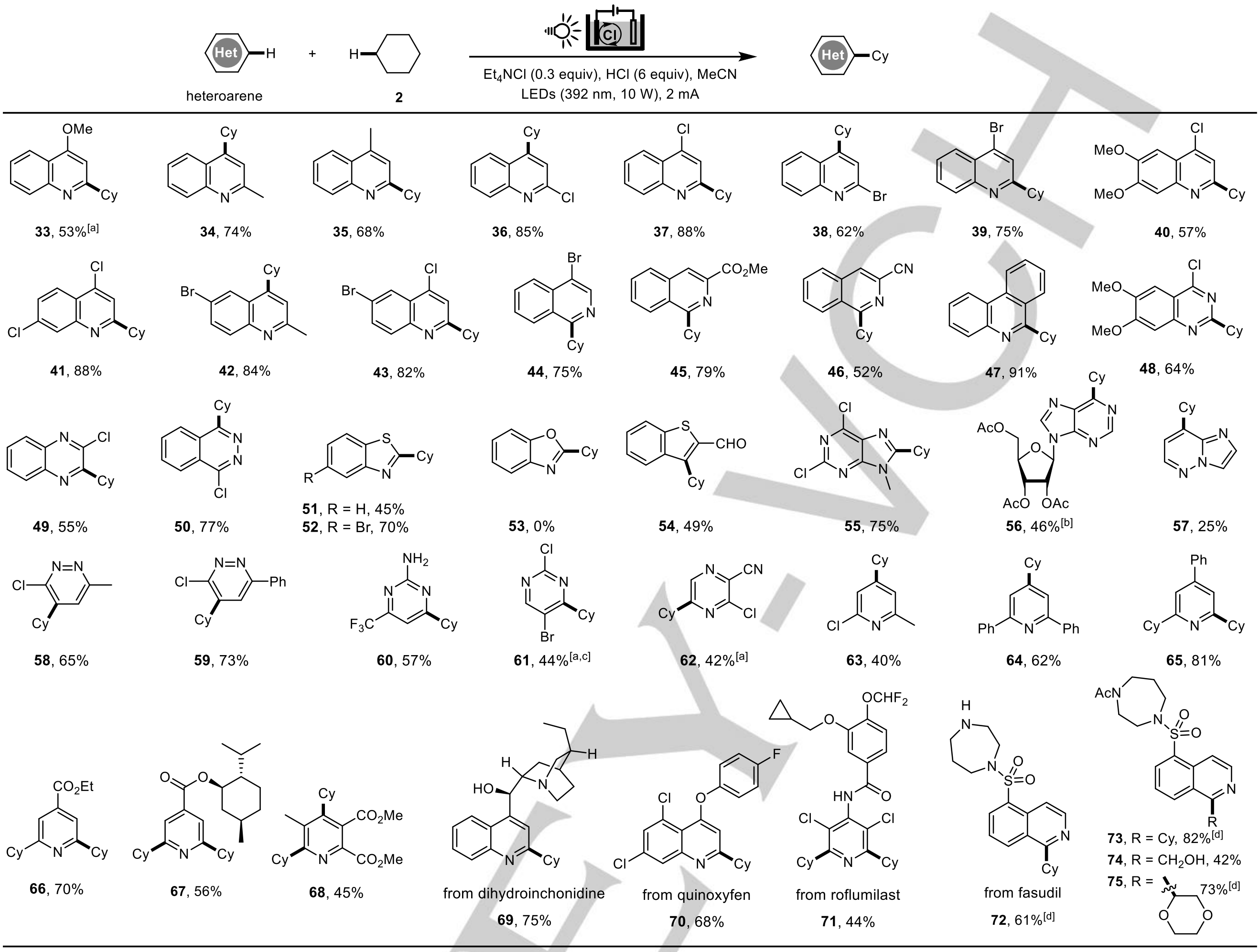
[a] Reaction conditions: RVC anode, Pt cathode, **1** (0.3 mmol), **2** (1 mL), MeCN (6 mL), Et_4NCl (0.09 mmol), HCl (1.8 mmol), 40 °C (internal temperature), 18 h (4.5 F mol^{-1}). [b] Determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as the internal standard. [c] Isolated yield. [d] Unreacted **1** in parenthesis. [e] Reaction at 40 °C in dark.

We next explored the substrate scope of the photoelectrochemical cross-coupling by first reacting a variety of $C(sp^3)$–H species with 2-phenylquinoline (Scheme 3). Besides cyclohexane, other cyclic alkanes, including cyclopentane (**4**), cycloheptane (**5**), cyclooctane (**6**) and cyclododecane (**7**), were also suitable coupling partners. Notably, norbornane (**8**) and 1,4-epoxycyclohexane (**9**) both reacted with 2-phenylquinoline in a regio- and stereoselective manner. It should be emphasized that 1,2-dichloroethane (DCE) was added as a cosolvent in the synthesis of **5**–**8** to facilitate the dissolution of the alkane substrates. To our gratification, our method was also found to be broadly compatible with activated $C(sp^3)$–H bonds such as toluene derivatives (**10** and **11**), ethers (**12**–**19**), primary alcohols (**20**–**25**), amide (**28**), sulfonamide (**29**) and phosphoramide (**30**), most of which would give rise to radical intermediates that are prone to oxidation into carbocations. Cleavage of the benzylic C–O bond was observed for the reactions of tetrahydrofuran (THF), diether ether and the primary alcohols under the standard reaction conditions. For example, the photoelectrochemical reaction of THF afforded alcohol **26** as the final product in 78% yield. Pleasingly, this type of transformations, which has been reported under photochemical conditions,[19] was avoided by reducing the acid concentration (**12**) or by the addition of a catalytic amount of $CeCl_3$ (**20**–**25**). For the reaction of 1,2-propanediol, the mono alcohol **27** was obtained as the only identifiable product with or without $CeCl_3$. The mechanism of the additive $CeCl_3$ to stop C–O cleavage remains unclear. Finally, the formyl $C(sp^2)$–H bond in formamide could also be activated, ultimately resulting in the installation of an aminocarbonyl group on the heteroarenes (**31** and **32**).

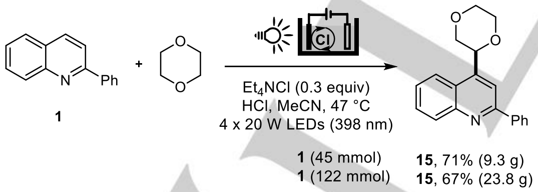


Scheme 3. Scope of C(sp^3)–H species. Reaction conditions: RVC anode, Pt cathode, 2-phenylquinoline (0.3 mmol), C(sp^3)–H species (1 mL for liquid or 20 equiv for solids), MeCN (6 mL), 33–46 °C (internal temperature). [a] MeCN/DCE (6 mL/1mL) as solvent. [b] Irradiated with 398 nm LEDs (20 W). [c] MeCN/DCE (6 mL/4 mL) as solvent. [d] 2 equiv of HCl. [e] Reaction in the presence of 2 mol % of $CeCl_3$.

Reaction compatibility with different heteroarenes was next explored using cyclohexane (**2**) as the coupling partner (Scheme 4). Quinolines substituted at 2- or 4-position with OMe (**33**), Me (**34** and **35**), Cl (**36** and **37**), or Br (**38** and **39**) were all shown to be well tolerated. Quinoline substrates carrying an OMe- (**40**), Cl- (**41**), or Br-functionalized (**42** and **43**) benzo ring also demonstrated satisfactory reaction efficiency. Note that quinoline reacted to afford a mixture of C2 (28%) and C4 (29%) alkylation product along with C2,C4-bisalkylated product (20%). Not surprisingly, cross-coupling between cyclohexane and other benzo-fused heteroaromatic scaffolds, including isoquinoline (**44**–**46**), phenanthridine (**47**), quinazoline (**48**), quinoxaline (**49**), phthalazine (**50**), benzothiazole (**51** and **52**), and benzothiophene (**54**), also occurred in a regioselective manner on the heterocyclic ring. Benzoxazole (**53**) underwent decomposition and failed to afford any desired product. Purine-derivatives (**55** and **56**) and imidazo[1,2,-*b*]pyridazine (**57**) were also alkylated regioselectively. We also succeeded in converting a variety of monocyclic heteroaromatics, such as pyridazine (**58** and **59**), pyrimidine (**60** and **61**), pyrazine (**62**) and the more challenging pyridine-based compounds (**63**–**68**). In addition, the photoelectrochemical method was applied to the alkylation of dihydrocinchonidine (**69**) and bioactive compounds such as quinoxyfen (**70**), roflumilast (**71**), fasudil (**72**) and acylated fasudil (**73–75**). Note that H_2O were added to the reactions of fasudil and acylated fasudil to increase their solubility in the reaction mixture.



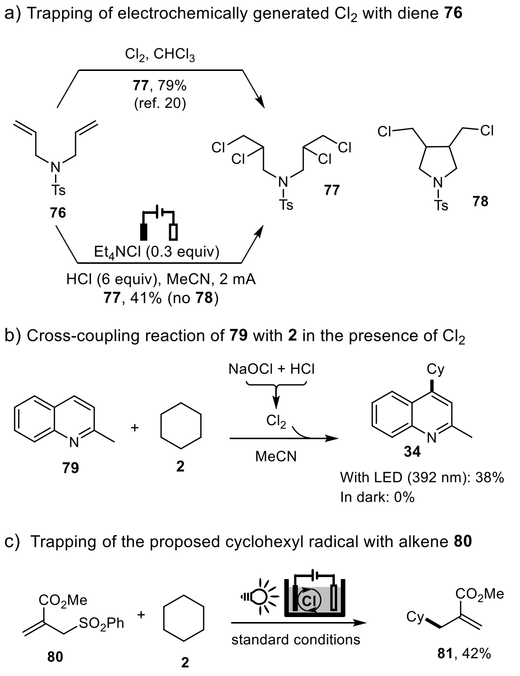
Scheme 4. Scope of heteroarenes. Reaction conditions: RVC anode, Pt cathode, heteroarene (0.3 mmol), **2** (1 mL), MeCN (6 mL), 34–42 °C (internal temperature). [a] Irradiated with 398 nm LEDs (20 W). [b] 2 equiv of HCl. [c] An inseparable mixture of mono- and bisalkylated products (67% yield, 1:3 ratio) was obtained under the standard conditions. [d] H_2O (0.5 mL) was added.



Scheme 5. Gram scale synthesis of compound **15**.

Reaction scale up for photoelectrochemical reactions is hampered by low surface area-to-volume ratio of batch reactors, which severely limits light penetration through the reaction media and thus makes it difficult to kinetically balance the photochemical and electrochemical steps. To our gratification, the synthesis of **15** could be conducted on a gram or even decagram scale by employing a flow setup (Scheme 5).

Initial mechanistic investigations showed that, under standard reaction conditions, the anode potential for the cross-coupling between **1** and **2** gradually increased from 0.92 V to 1.04 V vs. Fc^+/Fc (Figure S6a). These potentials are positive enough to oxidize Cl^- to Cl_2 [E_p (Et_4NCl) = 0.79 V vs Fc^+/Fc, $MeCN/H_2O$ = 40:1, Figure S6b]. It has been reported that diene **76** can react with Cl_2 to generate tetrachlorinated **77** instead of the radical cyclization-derived adduct **78** (Scheme 6a, top).[20] Consistent with this, the electrolysis of a solution of **75**, Et_4NCl and HCl in MeCN produced **77** in 41% yield without the detection of **78** (Scheme 6a, bottom). Additionally, the cross-coupling of **79** with **2** in the presence of Cl_2, generated by mixing NaOCl with HCl, furnished the intended target molecule **34** under LED irradiation, whereas no product formation was observed when the reaction mixture was shielded from light (Scheme 6b). Combined, these results indicated that Cl_2 was electrochemically generated and critical for promoting the subsequent dehydrogenative alkylation reaction. Meanwhile, the photoelectrochemical reaction of electrophilic alkene **80** with cyclohexane afforded compound **81** in 42% yield,[17d] providing support for the involvement of alkyl radicals as intermediates (Scheme 6c).


a) Trapping of electrochemically generated Cl_2 with diene **76**

b) Cross-coupling reaction of **79** with **2** in the presence of Cl_2

c) Trapping of the proposed cyclohexyl radical with alkene **80**

Scheme 6. Mechanistic experiments.

In summary, we have achieved efficient dehydrogenative cross-coupling of heteroarenes with aliphatic C–H bonds by merging electrochemistry with photochemistry. The method obviates the need for metal catalysts or chemical oxidants and demonstrates broad compatibility with a diverse range of heteroarenes as well as activated and unactivated $C(sp^3)$–H donors.

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Keywords: electrochemistry • photoelectrochemistry • heterocycles • C-H functionalization • radical reactions

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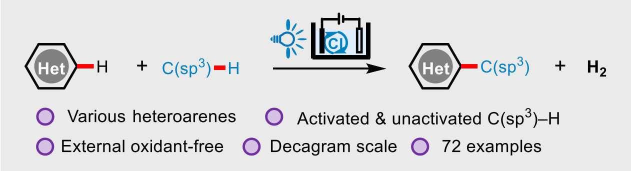

Report herein is an efficient photoelectrochemical dehydrogenative cross-coupling of heteroarenes with C(sp^3)–H species through H_2 evolution. Chlorine atoms, which are produced through light irradiation of anodically generated Cl_2 from Cl^-, abstract a hydrogen atom from C(sp^3)–H bonds to afford C-radicals. The latter undergo Minisci alkylation to afford the final functionalized heteroarene products.

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