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Photoelectrochemical cell for P-H/C-H cross-coupling with hydrogen evolution

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Photoelectrochemistry enables formation of a variety of active intermediates for organic synthesis in an environmental friendly manner. Herein, a photoelectrochemical cell is fabricated to realize activation of P-H/C-H bonds for cross-coupling hydrogen evolution. As compared with electrochemical cell, nearly 90% external bias input is saved to drive the C-P bond construction with good to excellent yields.

In view of the increasingly serious energy crisis and environmental problems, the use of photoelectrochemical (PEC) cells to realize water splitting^{1,2} and carbon dioxide (CO₂) reduction³ has received considerable attention across multiple discipline. Upon irradiation the photoexcited electrons from the light-sensitive electrode can be used to drive reduction reaction,⁴ and the holes left can be employed for oxidation reactions (Scheme 1).^{5,6} In this regard, electrochemical study has made a rapid progress in organic synthesis.⁷ A large number of dehydrogenation cross-coupling reactions via electrochemistry have been developed to construct C-C, C-N, C–O, C–S and C–P bonds. $^{8\mathchar`-11}$ Through precise control of the voltage used,¹² the active intermediates are able to form at electrode surface via single electron transfer. It is anticipated that PEC can conduct the organic conversion in a more energysaving way because of contribution of light energy. By modulating the composition, band position and morphology of photoelectrode, it is subsequently simple and easy to control the reactivity and selectivity.

With this in mind, we initiated to build PEC for chemical reaction. Herein, P-H/C-H dehydrogenation cross-coupling to construct C-P bond is chosen as a model reaction because organophosphorus compounds are important structural motifs

widely existing in agrochemical, material chemistry, and biochemistry.¹³ As the analogues of natural α -amino acid, there has been numerous advances in constructing the α amino phosphonates. Traditional synthetic methods (e.g., transition metal catalysis,¹⁴ chemical oxidation¹⁵ and photocatalysis¹⁶⁻²⁰) usually require directing groups, stoichiometric oxidants or metal catalysts. Our results show that BiVO₄ photoanode is able to construct C-P bond in good to excellent yields with no aid of any external oxidants or metal catalysts. For comparison, we also constructed a EC cell composed of a glassy carbon anode, Pt plate counter electrode and Ag/AgCl reference electrode for the same reaction. The results demonstrate that the applied bias to produce a (photo)-current of 5.0 mA is 0.1 V and 1.5 V for BiVO₄ PEC system and glassy carbon EC system, respectively. These potentials afford the target products in very similar yields (i.e., 90% in PEC Cell and 88% in EC Cell) after 12 h of (photo)electrolysis, indicating the great potential of more energy-saving PEC for organic synthesis.

The BiVO₄ electrode was prepared by electrodeposition in a three-electrode cell using a constant potential of -0.1 V vs Ag/AgCl for 10 min at room temperature (FTO as working electrode, Pt plate as the counter electrode and Ag/AgCl as the reference electrode). Then, N-phenyl tetrahydroisoquinoline **1a** and diphenyl-phosphine oxide **2a** were used as model substrates to perform the organic transformation. Solvents and electrolytes were firstly screened in an undivided cell via electrocatalysis. By utilizing "Bu₄NBF₄ as electrolyte and



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	$ \begin{array}{c} & & \\ & & $	Ph + H₂
	1a 2a Ph 3aa	
Entry	Electrolysis conditions	Yield(%)
1	RVC(+)/Pt(-), 10 mA / 2 h, ⁿ Bu ₄ NBF ₄ , MeCN, rt, Ar	90
2	ⁿ Bu ₄ NPF ₆ as electrolyte	79
3	ⁿ Bu ₄ NClO ₄ as electrolyte	83
4	MeOH as solvent	53
5	DMF as solvent	48
6	THF as solvent	n.d.
7	Under air	32
8	No electric current	n.d.
9	BiVO₄ as photoanode, E= 0.5 V <i>vs</i> Ag/AgCl	39
10	BiVO₄ as photoanode, NHPI as mediator, E= 0.1 V vs Ag/AgCI	93
11	No irradiation under PEC cell	n.d.
Reaction conditions: RVC anode (500 PPI, 1.0 cm × 1.0 cm × 0.5 cm), Pt plate		

cathode (0.5 cm \times 1.0 cm), **1a** (0.2 mmol), **2a** (0.2 mmol), "Bu₄NBF₄" (0.1 M), MeCN (5.0 mL), argon. Yield determined by ¹H NMR analysis using diphenylacetonitrile as the internal standard.

acetonitrile as solvent, the desired product 3aa was obtained in 90% yield with reticulated vitreous carbon (RVC) anode and Pt plate cathode (Table 1, entry 1). Slightly decreased reaction yields were obtained with "Bu₄NPF₆ or "Bu₄NClO₄ as electrolytes (Table 1, entries 2-3). By using "Bu₄NBF₄ as electrolyte, we replaced acetonitrile with other solvents such as methanol (MeOH), N,N-dimethylformamide (DMF) and tetrahydrofuran (THF). However, all of them resulted in lower reaction yields (Table 1, entries 4-6). Remarkably, a significant drop in yield was observed under air atmosphere (Table 1, entry 7). Moreover, control experiment showed that electric current was essential to this reaction (Table 1, entry 8). With the validated electrochemical results, we explored the possibility of PEC catalytic P-H/C-H cross-coupling under much reduced external bias input. The initial PEC experiments were carried out in an undivided cell equipped with BiVO₄ as the working electrode, Pt plate as the counter electrode and Ag/AgCl as the reference electrode. With a constant potential of +0.5 V vs Ag/AgCl, however, the coupling product was obtained in 39% yield after 12 h of photoelectrolysis (Table 1, entry 9). Introduction of 0.2 equivalent N-hydroxyphthalimide (NHPI) mediator dramatically enhanced the photocurrent density, thus leading to an excellent yield of desired product (Table 1, entry 10).

To examine the generality of the PEC strategy, the scope of the substrates was investigated using BiVO₄ photoelectrode under the standard conditions (Table 2). Firstly, we studied the reactivity of diverse tertiary amines. The derivatives bearing methoxyl and methyl at the *para*-position of the aromatic ring showed good reactivity in this transformation (**3ba-3ca**). The strong electron-withdrawing group trifluoromethyl was well tolerated to afford products in good yields (**3da**). In the case of *ortho*- or *meta*-substituents on the phenyl ring, the reaction



 o Reaction conditions: BiVO₄ photoanode as the working electrode (1.0 cm \times 2.0 cm), Pt plate as the counter electrode (0.5 cm \times 1.0 cm), Ag/AgCl as the reference electrode, **1** (0.2 mmol), **2** (0.2 mmol), NHPI (0.2 equiv.), 2,6-Lutidine (1.5 equiv.), "Bu₄NBF₄ (0.1 M), MeCN (5.0 mL), E= 0.1 V vs Ag/AgCl, 12h, argon, Blue LEDs (λ = 450 nm). Isolated yields are shown.

could proceed smoothly with good yields (**3ea-3fa**). Subsequently, the scope of P(O)H compounds was also studied. A variety of P(O)H substrates with electron-rich or electrondeficient groups were all tolerated in this reaction (**3ab-3ad**). Moreover, the reaction took place efficiently when the *meta*position of the phenyl ring was substituted with two methyl group (**3ae**). Other substrates, such as di(naphthalen-1-yl) phosphine oxide and phosphite esters, also generated the desired products in moderate yields (**3af-3ak**).

In the absence of light irradiation, we found electrochemistry catalytic P-H/C-H cross-coupling with a significant increase in external bias input (Table 3). The tertiary amines derivatives with either electron-donating or electronwithdrawing groups were well tolerated and afforded products in moderate to excellent yields (3aa-3la). Moreover, various substituents of P(O)H compounds were also tolerated in the EC cell (3ab-3ag). However, an electron-deficient chloro group at the para-position of the aromatic ring led to a lower reaction yield (3ah). Importantly, phosphite esters were found suitable for EC system, but the yield gradually decreased with the increase of chain length (3ai-3ak). From the above results, we inferred that both PEC and EC systems can work well for C-P bond construction.

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Table 3. EC phosphorylation of C-H bond^a



In the PEC cell, NHPI play a key role in this transformation. When NHPI was added, the bias required to produce 5.0 mA photocurrent negatively shifted from 0.5 to 0.1 V (Figure 1a), the yield of final product increased from 39% to 93% after 12 h photoelectrolysis. Furthermore, the cyclic voltammetry (CV) was also performed to explore the reaction mechanism. Initially, the oxidation peak of N-phenyl tetrahydroisoquinoline (1a), diphenyl-phosphine oxide (2a) and NHPI was observed at 0.83 V, 1.05 V and 1.62 V (Figure 1b). After adding 2,6-lutidine in the NHPI solution, a redox couple were appeared at 0.82 V (half-wave potential). Then, upon the addition of 1a, the anodic current was enhanced and a significant decrease in the cathodic current, indicating the regeneration of NHPI via the reaction between phthalimide-N-oxyl (PINO) radical and 1a.21 These results also indicated that NHPI was involved in the reaction as a redox mediator.





Fig. 1 (a) LSVs of a BiVO₄ photoanode obtained under Blue LEDs (λ = 450 nm) illumination (red) and containing NHPI (green); under dark (black) and containing NHPI (blue). (b) CVs obtained in MeCN with 0.1 M "Bu₄NBF₄ containing 5 mM **1a** (black), 5 mM **2a** (red), 5 mM NHPI (blue); 5 mM NHPI solution containing 10 mM 2,6-Lutidine (green); 5 mM NHPI solution containing 10 mM 2,6-Lutidine and 5 mM **1a** (pink). Scan rate: 100 mV s⁻¹.

Based on the aforementioned results, a plausible mechanism for photoelectrochemical P-H/C-H cross-coupling is outlined (Scheme 2). Initially, $BiVO_4$ photoelectrode is excited by the incident photons to generate high energy electron-hole pairs. Then, the holes reach the surface of $BiVO_4$ to oxidize **1a** into radical cation intermediate (**1a**^{*+}). At the same time, the deprotonated NHPI by 2,6-lutidine goes through an anodic oxidation to give the corresponding PINO radicals. Subsequently, PINO radical extracts a hydrogen atom from **1a**^{*+} to regenerate NHPI, and the **1a**^{*+} is further oxidized to afford an iminium ion intermediate (**1a**⁺), which reacts with diphenyl-phosphine oxide *via* the nucleophilic attack to enable the formation of final product with C-P bond. Simultaneously, the released protons are reduced to evolve molecular hydrogen at the surface of counter electrode.

In summary, we have realized P-H/C-H cross-coupling with hydrogen evolution via PEC and EC systems. The approaches show good functional-group tolerance, broad substrate scope and good to excellent yield. In contrast to the EC system, PEC system can considerably decrease the applied voltage (i.e., >90% external bias) for the C-P bond construction because of combining visible light energy. From the perspective of energy saving, PEC has significant advantages over EC that requires a higher applied potential and electric energy input. Further extension of PEC to diverse organic synthesis are actively ongoing in our laboratory.

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Conflicts of interest

There are no conflicts to declare.

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