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Scalable, Electrochemical Dehydrogenative Cross-Coupling of P(O)H Compounds with RSH/ROH

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A practical, scalable electrochemical dehydrogenative crosscoupling of P(O)H compounds with thiols, phenols and alcohols in both undivided cell and continuous-flow setup is disclosed. The broad substrate scope (>50 examples), good functional-group tolerance and scalability (>10 g) show the potential for the practical synthesis. A preliminary mechanistic study suggests the phosphorus radicals are involved in the catalytic cycle.

as Organophosphorus compounds serve important intermediates in organic synthesis, medicinal chemistry, materials and agrochemistry.¹⁻² Traditional routes for the construction of P-S and P-O bonds generally (such as the Atherton-Todd-type reactions) proceed by nucleophilic substitution of the toxic and moisture sensitive halides RP(O)Cl with RSX/ROH in presence of strong bases and toxic solvents, in which with limited functional-group tolerance and multistep reaction progress.³⁻⁴ Additionally, a variety of oxidative methods including metal-catalyzed,⁵ photoredox-catalyzed⁶ and chemical oxidants catalyzed⁷ cross-coupling of P(O)-H compounds with RSH or ROH has been developed in recent years (Scheme 1a). In the meantime, organic radical reactions have emerged as a powerful strategy for the synthesis of versatile compounds.8 Given the accessible bond dissociation energies (BDE \approx 90 kcal mol⁻¹) of the P-H bond of secondary phosphine oxides and related structures,9 several radical strategies¹⁰ by using stoichiometric amounts of metal oxides Ag(I)^{10a-c} and Mn(III) reagents^{10d}; photocatalyst^{10e-g} or other reagents^{10h} to generate phosphorus radicals, have been reported for the synthesis of organophosphorus compounds (Scheme 1b). Despite significant advances that have been made in this field, the development of efficient, environmentally friendly, scalable and facile approach is still highly desired, especially in a oxidant-free fashion.





Mild reaction conditions
 Broad substrate scopes
 gram-scale synthesis
 Scheme 1 Methods for construction of P-S and P-O bonds

Electrochemistry offers a green and efficient alternative to conventional chemical approaches for redox transformations, electrons as "reagents" are tunable in the activation of organic molecules to generate reactive intermediates, which provides new opportunities for the construction of important organic molecule in environmentally friendly way.11 an Recently. efficient electrochemical protocols have been achieved by groups of Baran, Waldvogel, Lei, Xu, Ackermann, Stahl and others.¹²⁻¹³ Herein, we report a new strategy for the generation of phosphorus radicals through electrochemical and its application in the construction of P-S and P-O bonds (Scheme 1c). Our strategy provides an efficient and facile approach for the synthesis of organophosphates and sulphurcontaining organophosphorus compounds under the oxidant-free conditions. In addition, the reaction can be easily scaled up to 10 g scale by using a continuous-flow system under mild conditions.

To start our investigation, diphenylphosphine oxide **1** and 4methylbenzenethiol **2** were selected as the model substrates for optimization studies. Initial screening revealed that using $^{n}Bu_{4}NBF_{4}$ as the electrolyte and acetonitrile as the solvent, combined with the use of platinum anode and cathode with a constant current density of 3.3 mA cm⁻² after 4 hours in the ambient atmosphere, resulted in

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⁺ Footnotes relating to the title and/or authors should appear here.

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Table 1 Optimization of reaction conditionsa

4NBF4 (0.18 M) MeCN, rt, air, 4 h i = 3.3 mA/cm 3 Entry Deviation from standard conditions Yield (%)b 1 84 None 2 LiClO4 as electrolyte 62 3 nBu4NPF6 as electrolyte 71 4 DMF as solvent 83 5 MeOH as solvent 49 RVC as anode 6 68 7 GC as anode 58 8 $j = 2.7 \text{ mA/cm}^2, 5 \text{ h}$ 64 9 *j* = 4.7 mA/cm², 3 h 56 Without current 10 n.d.

a Standard condition A: Pt plate anode (15 mm×10 mm×0.1 mm), Pt plate cathode (15 mm×10 mm×0.1 mm), 1 (0.3 mmol), 2 (0.9 mmol), ⁿBu₄NBF₄ (0.18 M), MeCN (5.0 mL), rt, I= 5 mA (*j* = 3.3 mA/cm²), 4 h. b Isolated yield. RVC = Reticulated Vitreous Carbon; GC = graphite rod; n.d. = not detected

the highest yield for the desired product 3 (84% yield; Table1, entry1). when using LiClO₄ or ⁿBu₄NPF₆ instead of ⁿBu₄NBF₄ as electrolyte, lower yields were observed (Table 1, entries 2 and 3). The solvent DMF showed no negative effect, and MeOH afforded the product 3 in only 49% yield (Table1, entries 4 and 5). Among the anode electrode materials screened, platinum anode proved to be the most effective at facilitating transformation. Either using RVC (Reticulated Vitreous Carbon) or GC (graphite rod) to replace the platinum led to a decreased yield, respectively (Table1, entries 6 and 7). The current density had a great influence on the reaction, increase or decrease of electric current gave lower yields (Table1, entries 8 and 9). Control experiment indicated that the electricity was essential for this reaction (Table1, entry 10).

With the optimal conditions in hand, the scope of the reaction was investigated. As shown in Table 2, methyl-substituted benzenethiols showed good reactivities in this transformation (3-6). Using flow electrochemical reactor, the product 3 was obtained in 81 % yield with a shorter reaction time (25 mins vs 4 h). Thiophenols bearing halide (Cl, Br, F) substituents were successfully coupled with 1, affording the products 6-10 in 66-86% yields. In addition, the electron-rich or electron-neutral substituents at different positions of benzene ring of thiophenols gave the desired products 10-13 in good yields. Naphthalenethiol afforded the desired product 14 in 47% yield (46% yield in 25 mins with the flow system). Moreover, aliphatic thiols were also suitable substrates for this P-S bond formation reaction. Phenylmethanethiol and 1-phenylethane-1-thiol were tolerable, giving the products 15-16 in moderate yields. cyclohexanethiol, butane-2-thiol, pentane-1-thiol and methyl 2mercaptoacetate furnished the desired products in high yields (up to 90% yield; 17-20). The scope of diphenylphosphine oxide was next investigated, substrates bearing methyl, fluoro and di-methyl groups at phenyl ring were able to afford desired products 21-23 in 53-84% yields.

Next, we further applied this electrochemical system to the crosscoupling of various phenols and alcohols under similar conditions (for more details, see SI, Table S2). As shown in Table 3, with minor alterations of anode electrode material (GC-Pt) and electrolyte (KI),

Table 2 Scope of P-S bond formation with various thiols and different P(O)H compounds^a

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^a Standard condition A: the same to table 1. Isolated yield. ^b Using continuousflow setup as reaction instrument, 25 mins. ^c Gram-scale synthesis. ^d Using DMF as solvent. e Reaction time = 6 h

using DABCO as the additive, the cross-coupling process proceeded efficiently to generate the desired organophosphinates in high yields (up to 93% yield, 24-57). The results indicated this method exhibited excellent functional group tolerance, and a wide variety of phenols bearing different substituents including OMe (24), Me (26-27), t-Bu (28), F (30-31), Cl (32), Br (33-34), I (35) on the benzene ring had been successfully applied to the reaction to produce the corresponding products in moderate to high yields. A substrate bearing a highly coordinative SMe group was successfully subjected to the reaction, giving the organophosphinate 29 in 65% yield. Other functional groups such as SO₂Me, Ac and CO₂Me, as well as strongly electronwithdrawing NO₂, easily hydrolyzed OCF₃, easily oxidized CHO groups were also well-tolerable, affording the corresponding products in high yield (36-42). The reaction of Bpin substituted phenol (43) gave a slightly decreased yield. Naphthols and disubstituted phenols were also proved to be suitable substrates in the reaction (44-51). To our delight, this method can be applied to the coupling of alcohols, and various alcohols such as methanol, ethanol and isopropanol were proven tolerable by using RVC as anode (52-54). Diphenylphosphine oxide were also investigated, finishing the desired products 55-57 in 30-69% yields. The reaction under flow condition was performed using Pt as anode, the products 52-54 were obtained with superior outcomes. To further demonstrate the synthetic utility of this method, the reaction was conducted on gram scale under the optimal conditions. 8 mmol 1 reacted with 2 to furnish the desired

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 Table 3 Scope of P-O bond formation with various Phenols/alcohols and different P(O)H compounds^o



^{*a*} Standard condition B: GC anode (φ 6 mm), Pt plate cathode (15 mm×10 mm×0.1 mm), 1 (0.3 mmol), Phenols (0.9 mmol), DABCO (50 mol%), KI (0.12 M), MeCN (5.0 mL), rt, *j* = 3.3 mA/cm² 3 h. Isolated yields. ^{*b*} Using ElectraSyn 2.0 as reaction instrument. ^{*c*} Gram-scale synthesis. ^{*d*} Using continuous-flow setup as reaction instrument. DABCO = 1,4-Diaza[2.2.2]bicyclooctane. ^{*e*} RVC anode (100 PPI, 15 mm×10 mm×10 mm), MeCN : alcohol = 4 : 1 (5 mL), without DABCO.

product **3** in 85% yield (2.2 g; Table 2). Similarly, a gram-scale reaction between 8 mmol **1** and **58** afforded the desired product **24** in 68% yield (1.7 g; Table 3). Notably, simple continuous-flow electrochemical reactor was assembled for the scale up to the 10 g scale, which showed potential for the practical synthesis (**52**, 93 % yield, 10.8 g/70 h; Table 3).

To shine light on the mechanism of this electrochemical dehydrogenative cross-coupling reaction, several control experiments were conducted (for more details, see SI). For the P-S bond formation reactions, when 2.0 equiv TEMPO (2,2,6,6-Tetramethylpiperidin-1-oxy) was added under the standard condition A, coupling product 3 was obtained in 72% yield with 24% radical trapping product 59 (Scheme 2a). In addition, 4methylbenzenethiol 2 reacted with styrene to afford the thioether in 78% yield when using styrene instead of substrate 1, and the disulfide was obtained in 96% yield without substrate 1 under the standard condition A (SI, Figure S4). These results suggested that thiyl radical intermediates were involved in the catalytic cycle. Notably, the radical trapping product 60 was isolated in 26% yield when 2.0 equiv TEMPO was added without 4-methylbenzenethiol 2 under the standard condition A, which not been detected under the condition without an electric current. These results indicated that the DOI: 10.1039/C9CC01378D



Scheme 2 Control experiments.



Figure 1. Proposed mechanism.

phosphorus radicals might be generated under the standard conditions (Scheme 2b). Furthermore, EPR experiments were also performed. Compare to no signal was observed under the condition without the electricity, the signal of phosphorus radicals further lent support to the hypothesis that the formation of phosphorus radicals in the reaction (SI, Figure S3). For the P-O bond formation reactions, when 1.0 equiv TEMPO was added under the standard condition B, the yield of the coupling product 24 decreased to 22% yield, and the adduct 60 was also detected by ESI-HRMS (Scheme 2b). The EPR data provided further support for the involvement of phosphorus radical in the P-O bond formation reaction (SI, Figure S3). In addition, no product was detected for the control experiment for the reaction without KI indicated that the iodine ion was essential for this reaction. Different from the previous report,¹⁴ only trace amount of **24** was obtained, when using I₂ instead of KI, suggesting that the I₂ was not involved in the catalytic cycle (SI, Figure S4).

Based on the above experimental results, a plausible mechanism for this electrochemical dehydrogenative cross-coupling reaction is proposed in Figure 1. Single-electron-transfer (SET) oxidation of the thiols by the anode leads to the formation of sulfur radicals,^{12c-d} followed by rapid dimerization of sulfur radicals to give disulfides **II**. In the meantime, the phosphorus radicals **III** are generated from the I by a hydrogen atom transfer (HAT) process. The phosphorus radical intermediates **III** can undergo either direct coupling with the sulfur radicals or disulfides to form the final products. Alternatively, the product also can be produced by nucleophilic attack of the P(O)H compound with the disulphide (SI, Figure S4). Meanwhile, H₂ is released by cathodic reduction at the cathode. Similarly, for the P-O bond formation reactions, iodine radical intermediates **IV** can be formed from iodide ions through anodic oxidation. Sequentially, the radical coupling reaction occurs between phosphorus radicals **III** and the iodine radicals **IV** to produce diphenylphosphinyl iodine intermediates **VI**, which easily react with phenols or alcohols anions **V** to form the P-O bonds. Moreover, several alternative mechanisms were also proposed in the SI (for more details, see Figure S4).

In summary, we developed an efficient electrochemical method to construct P-S and P-O bonds under oxidant-free conditions. Various cheap thiols, phenols and alcohols were suitable substrates for this transformation with excellent functional-group tolerance. Importantly, the reaction can be conducted on a gram scale with good reaction efficiency and a flow electrochemical method has been developed for >10 g scale up synthesis, which showed the potential for the practical synthesis. Further studies on synthetic application are ongoing in our laboratory.

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Conflicts of interest

There are no conflicts to declare

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A practical, scalable electrochemical dehydrogenative cross coupling of P(O)H compounds with thiols, phenols and alcohols