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Electrochemical Approach for Direct C–H Phosphonylation of Unprotected Secondary Amine

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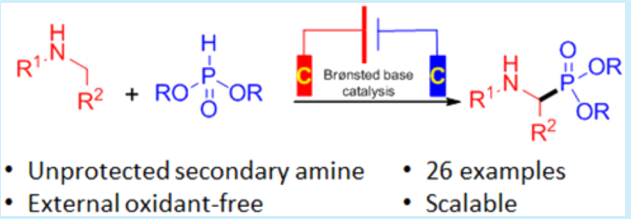
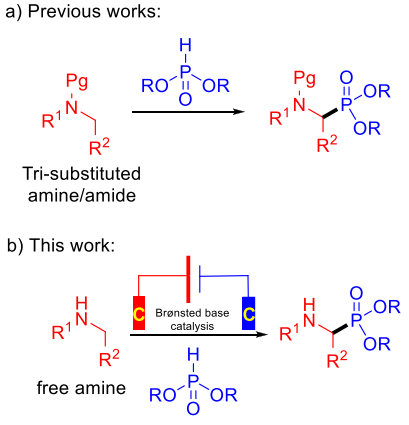
Supporting Information

ABSTRACT: Direct α-phosphonylation of an unprotected secondary amine in a single step is of practical importance to amino phophophates. However, this protocol is limited due to the high redox barrier of unprotected amine. In this paper, we report C–H phosphonylation of an unprotected secondary amine via an electrochemical approach in the presence of catalytic carboxylate salt. This metal-free and exogenous oxidant-free method furnishes diverse target molecules with satisfactory yield under mild reaction conditions. Successful application of the protocol in a gram-scale experiment demonstrates the potential utility for further functionalization.



Direct α-phosphonylation of amine could provide amino phosphonates, mimics of amino acids with broad pharmaceutical applications.[1] It was, however, unexpected that most of the direct phosphonylation protocols require tertiary amine or amide as starting materials, in the presence of various catalysts and stoichiometric oxidant[2] (Scheme 1a).

Scheme 1. Directed C–H Phosphonylation of Amine



Two possible reasons could contribute to this substrate preference: (i) the trisubstituted amine was more electron-donating and could more readily undergo single electron transfer with oxidative reagent or catalyst, and (ii) the iminium ion intermediate of amide or trisubstituted amine was highly reactive toward nucleophiles, facilitating the intermolecular bond formation. In comparison, the similar conversion involving unprotected amine remains unexplored. As the unprotected amine could provide broad diversity during derivation, a direct phosphonylation of unprotected secondary amine would be desired.

Electrochemical organic synthesis has recently experienced tremendous growth, leading to a variety of novel and unexpected transformations[3] with transitional-metal catalyst,[4] organocatalyst,[5] and even catalyst-free[6] conditions. Compared to conventional synthetic methods, the electrochemical strategy could also be conveniently employed to construct C–C,[7] C–O,[8] C–N,[9] and C–S[10] bonds under much more mild conditions. Despite the progress on the electrochemical C–P bond formation from trisubstituted amine and imine made in recent years through electrochemical strategies,[11] the C–H phosphonylation from unprotected secondary amine has been left unexplored. Lei[12] and co-workers demonstrated electrochemical dehydrogenative aromatization of a series of N-heterocycles. In this chemistry, a mediated dehydrogenation of unprotected cyclic amine was proposed as the operative mechanism, whereas the dehydrogenation took place at the Pt anode to produce aromatized compounds as the major product. Inspired by such correlation between anodic materials and the electron transfer pathway, we achieved a readily anodic oxidation of secondary free amine, affording the C–H phosphonylation by the combination of a suitable anode and Brønsted base catalyst. Mechanistic investigations further show that the electro-organic reaction follows a Brφnsted base catalysis pathway (Scheme 1b).

We started the study using 1,2,3,4-tetrahydroisoquinoline (**1a**) as the starting material and diethyl phosphate (**2a**) as a model nucleophile to trap the dehydrogenative intermediate. Graphite felt was employed as both the anode and the cathode

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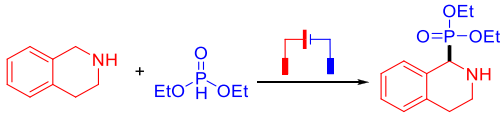
material with controlled cell potential. At first, $LiClO_4$ was applied as an electrolyte, and desired product **3a** was detected, giving 46% yield. Due to the property of $LiClO_4$ as a Lewis acid, we evaluated $Cu(OTf)_2$ as an additional Lewis acid additive. Unexpectedly, with 10 mol % of $Cu(OTf)_2$ loading, the desired **3a** was not detected at all. So, we subjected a catalytic amount of base into the reaction, and NaOAc was proven as the optimum choice, giving **3a** in 76% isolated yield. Several tetraalkylammonium salts were also employed as alternative electrolytes instead of $LiClO_4$, and Bu_4NClO_4 could give an acceptable yield of 64%. Subsequently, other solvents (including DMF, DCM, and MeOH) and electrode materials were tested, as summarized in Table 1.

Table 1. Optimization of Reaction Conditions



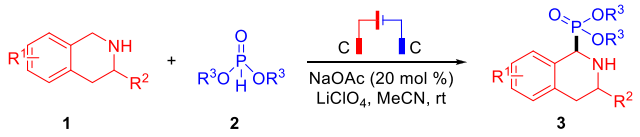
1a 2a 3a

entry	electrolyte	additive (20 mol %)	solvent	electrode	yield[b] (%)
1	$LiClO_4$		MeCN	C+/C−	46
2	$LiClO_4$	$Cu(OTf)_2$	MeCN	C+/C−	ND
3	$LiClO_4$	NaOAc	MeCN	C+/C−	88 (76[c])
4	$LiClO_4$	K_3PO_4	MeCN	C+/C−	76
5	Bu_4NClO_4	NaOAc	MeCN	C+/C−	64
6	Bu_4NBr	NaOAc	MeCN	C+/C−	32
7	Bu_4NF	NaOAc	MeCN	C+/C−	35
8	$LiClO_4$	lutidine	MeCN	C+/C−	48
9	$LiClO_4$	NaOAc	DMF	C+/C−	15
10	$LiClO_4$	NaOAc	CH_2Cl_2	C+/C−	ND
11	$LiClO_4$	NaOAc	MeOH	C+/C−	22
12	$LiClO_4$	NaOAc	MeCN	C+/Pt−	30
13	$LiClO_4$	NaOAc	MeCN	Pt+/C−	27
14	$LiClO_4$	NaOAc	MeCN	Pt+/Pt−	20

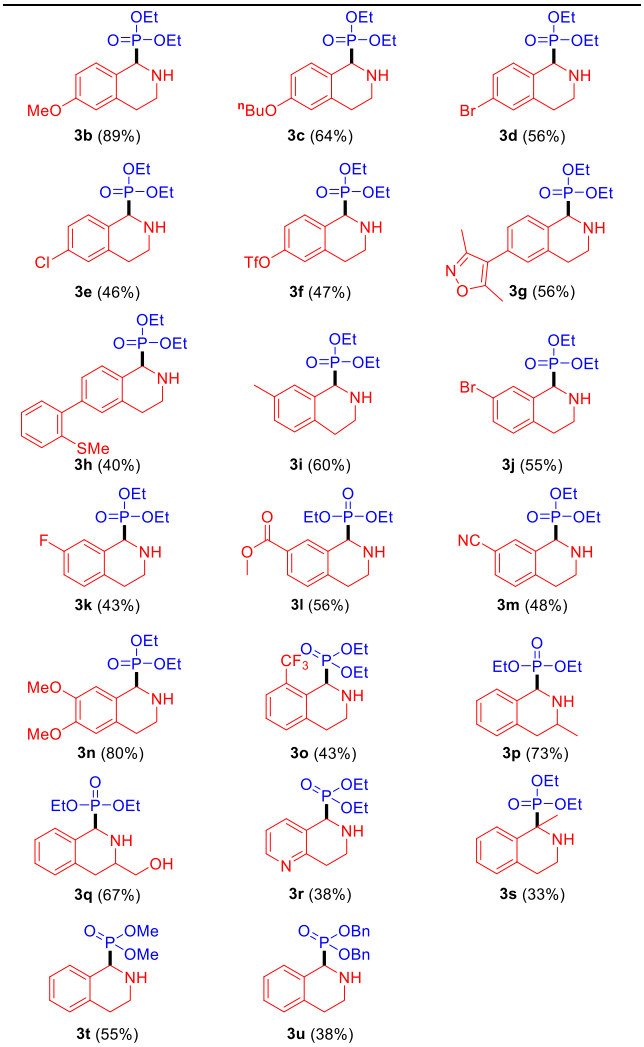
[a]Conditions: **1a** (0.5 mmol), **2a** (1 mmol), supporting electrolyte (0.1 mmol), solvent (20 mL), graphite felt (C) anode and cathode, rt, 6.6 V, 6 h. [b]Yields were determined by ^{31}P NMR using trimethyl phosphate as internal standard. [c]Isolated yield.

With the optimized conditions (Table 1, entry 3), we explored the substrate scope for this dehydrophosphonlyation reaction with a series of unprotected secondary amines (Scheme 2). Initially, the substrates with both electron-donating and -withdrawing groups at position 6 were proved feasibly, affording the desired products (**3b**−**3h**) in moderate to excellent yield. We found that N-heterocycles with an electron-donating group can achieve a relatively higher yield (**3b**−**3f**). In addition, N-heterocycles substituted with 3,5-dimethylisoxazole and *o*-methyl(phenyl)sulfane at position 6 also lead to favorable yields (**3g**, **3h**). These results indicate that there is no inhibition by the heterocyclic substituent in this electro-organic system. Different groups were introduced into position 7 of N-heterocycles. The result shows that electron-donating groups favor a higher yield, which agrees with that of position 6. In addition, the halogen atoms Br and F are well-tolerated, and target molecules (**3j**, **3k**) were easily achieved with a moderate yield. For the substitution of methyl, methyl formate, and cyanide, the desired molecules can also be achieved with moderate yield (**3i**, **3l**, **3m**). At a specific length, a trifluoromethyl at position 8 was able to furnish the

Scheme 2. Substrates of Unprotected Cyclic Secondary Amine[a]



1 2 3



[a]Reaction conditions: **1** (0.5 mmol), **2** (1.0 mmol), NaOAc (0.1 mmol), $LiClO_4$ (0.1 mmol), CH_3CN (20 mL), 6.6 V, rt, graphite felt. Isolated yields.

corresponding product (**3o**), and two methoxyls at positions 6 and 7 (**3n**) even gave an excellent yield up to 80%. Simultaneously, some substrates with substitution at position 2 were investigated. In these cases (**3p**, **3q**), the yield was 73 and 67%, respectively. A substrate changing its aromatic ring from benzene into pyridine (**3r**) was amenable to this transformation, as well. In this case, even with a bulkier steric hindrance at the α-position, the target C−P was constructed successfully with an acceptable yield (**3s**). Finally, the small steric hindrance of dimethyl phosphonate (**3t**) suited the system well and afforded the field at a close level with that of the standard reaction. Because of the bulky steric hindrance of dibenzyl phosphite, **3u** afforded a relatively low yield.

The dehydrophosphonlyation approach was then applied to acyclic molecules (Scheme 3). Target products were

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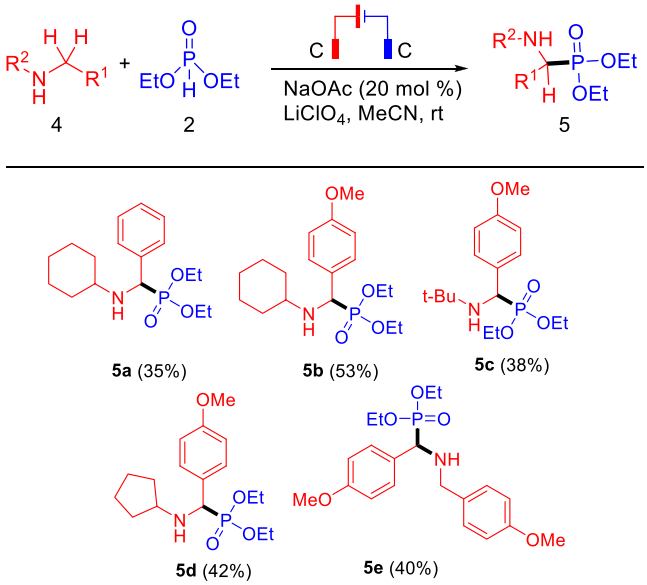
Scheme 3. Substrates of Unprotected Acyclic Secondary Amine[a]



[a]Reaction conditions: **4** (0.5 mmol), **2** (1.0 mmol), NaOAc (0.1 mmol), $LiClO_4$ (0.1 mmol), CH_3CN (20 mL), 6.6 V, rt, graphite felt. Isolated yields.

successfully achieved (**5a**−**5e**). First, *N*-benzylcyclohexanamine (**4a**) was subjected to the reaction and afforded **5a** in 35% yield. Then the methoxy was introduced to the aryl *para*-position. For the substrate *N*-(4-methoxybenzyl)-cyclohexanamine, the reaction occurred and provided an acceptable yield of 53% (**5b**). Cyclohexyl connected to N was next replaced by tertiary butyl (**5c**) and cyclopentyl (**5d**), which also led to a desired product at the same level as **5a**. Specifically, the primary amine (4-methoxyphenyl)-methanamine was employed to the system, and the target secondary amine product was achieved (**5e**).

To demonstrate the synthetic application of this electrochemical protocol, a scale-up reaction was carried out. The reaction was designed using 5 mmol **1a** and 10 mmol **2a**. All of the additives were scaled up corresponding to the amount we optimized. The reaction time was prolonged to 28 h, and the reaction proceeded smoothly. Finally, the target product **3a** was gained with a 67% isolated yield.

To gain further mechanistic insights about this electro-organic system, we studied the voltammogram properties of **1a**, **2a**, NaOAc, and the reaction mixture to provide key electrochemical parameters and information regarding the reaction pathway. As shown in Figure 1, diethyl phosphonate **2a** was electrochemically inert whether alone (blue curve) or with 20 mol % of NaOAc (green curve). However, substrate **1a** underwent an anodic oxidation with an obvious anodic current after the onset of ~0.75 V vs Ag/AgCl, and a remarkably increasing Faradaic current was observed with an increasing oxidative potential. Compared with **1a** (red curve), the anodic current of the mixture of **1a** and NaOAc (magenta curve) was generally enhanced with similar onset potentials, indicating the benefit of NaOAc addition to slightly promote the electron transfer.

Based on our synthetic and mechanistic investigations, a plausible reaction pathway is summarized and outlined in Scheme 4. Initially, **1a** is oxidized at the anode electrode, giving

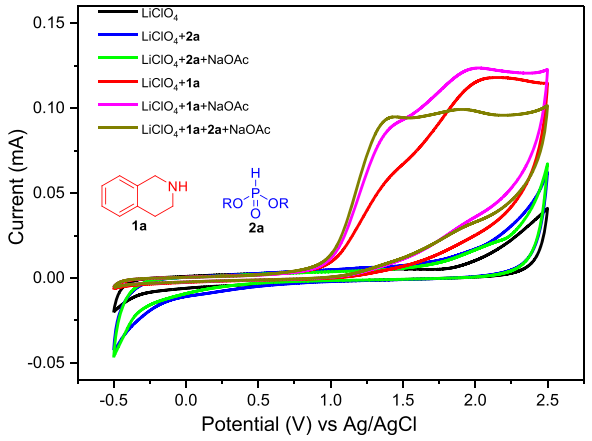
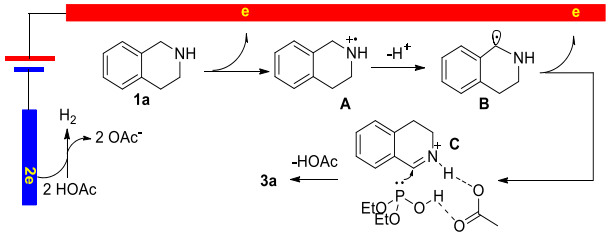


Figure 1. Cyclic voltammogram (CV) investigations of direct electrochemical C−H phosphonylation. CV characteristics of the reaction background (solvent MeCN and electrolyte $LiClO_4$), phosphate **2a**, **2a** in NaOAc, amine **1a**, **1a** in NaOAc, and a mixture of **1a** and **2a** in NaOAc (best mimicking the reaction conditions).

Scheme 4. Plausible Reaction Pathway



rise to a radical cation intermediate **A**. After consequent deprotonation, carbon radical **B** is formed and further undergoes a second electron transfer to give intermediate **C**, which is attacked by phosphite **2a** catalyzed by Bronsted base sodium acetate and furnished final product **3a**. On the cathode, hydrogen evolution occurs and an acetate anion is regenerated, producing the high value hydrogen gas which is detected by a gas detector (see Supplementary Figure S3) and giving the atomic economy to the whole electro-organic reaction.

In summary, a green and direct electrochemical C−H phosphonylation of unprotected secondary amine was developed under mild react conditions without transition metal, catalyst, and exogenous oxidant. For the substrate scope, a series of cyclic and chain secondary amines were well compatible. The scale-up experiment also showed the promising use of this protocol in mass production. An acetate anion was important to facilitate the electron transfer and nucleophilic addition of phosphite.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b02707.

Synthesis of starting compounds, detail process of electrochemistry reaction, characterization data (PDF)

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Notes

The authors declare no competing financial interest.

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