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TEMPO-Catalyzed Aminophosphinoylation of Ethers via Tandem C(sp^3)−H and C(sp^3)−O Bond Cleavage

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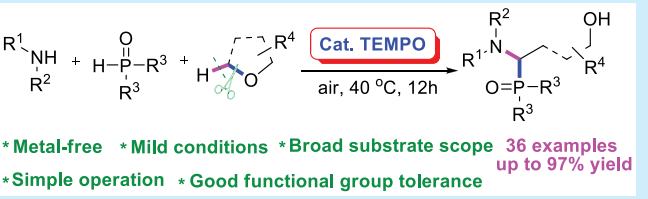
Supporting Information

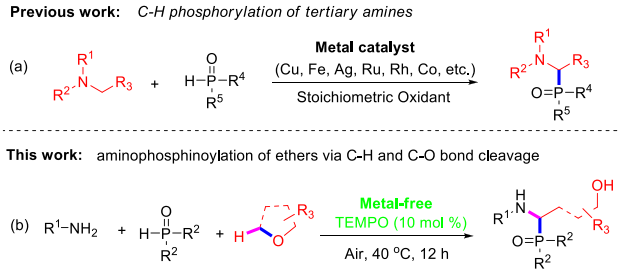
ABSTRACT: A new TEMPO-catalyzed aminophosphinoylation of ethers with amines and H-phosphine oxides was developed for the synthesis of α-aminophosphine oxides. This metal-free aminophosphinoylation reaction could be conducted under mild conditions through tandem C(sp^3)−H and C(sp^3)−O bond cleavage. The present method offers a facile and efficient approach to broad range of α-aminophosphine oxide derivatives in moderate to good yields with excellent functional group tolerance.



Organic phosphorus compounds have attracted considerable attention of chemists owing to their wide applications as ligands in synthetic chemistry and their important biological and pharmacological characteristics.[1] In particular, α-aminophosphine oxides exhibit various intriguing biological activities such as antiviral,[2] antitumor,[3] antibacterial,[4] and enzyme-inhibiting[5] properties. Consequently, much effort has been devoted to develop efficient methods for the construction of α-aminophosphine oxides. Generally, α-aminophosphine oxides are prepared by the Lewis acid catalyzed nucleophilic addition reaction of H-phosphine oxides with imines (Pudovik reaction)[6] and the three-component reaction of aldehydes, amines, and H-phosphine oxides (Kabachnik−Fields reaction).[7] Nevertheless, these traditional methods usually encountered limitations on the substrate scope or reaction applicability because of the poor availability and stability of imines. To overcome this problem, the oxidative α-C(sp^3)−H phosphinoylation of tertiary amines is undoubtedly the most direct protocol for the synthesis of α-aminophosphine oxides via direct C−P bond formation.[8,9] Since the pioneering work of Li and co-workers,[8] various transition-metal-catalyzed oxidative phosphinoylations of C-(sp^3)−H adjacent to the N atom of amines with H-phosphine oxides or H-phosponates have been reported (Scheme 1a).[9] Despite some great advantages, the harsh reaction conditions and the use of toxic metal catalysts and stoichiometric quantities of oxidants have stimulated chemists to develop more mild, safe, and efficient approaches.

Recently, the cross-coupling reaction involving α-C−H functionalization of ether has emerged as a powerful and attractive protocol for construction of the C−X (X = C, S, N, P) bond.[10] In most cases, the C−O bond cleavage of ether does not occur, and the ether structural unit was introduced to the organic framework via C−H functionalization of ethers.[10,11] So far, a few synthetic methods have been reported for difunctionalization of ethers via tandem C−H and C−O bond cleavage, in which transition-metal catalysts, strong oxidants, or high reaction temperature are usually required.[12−15] For example, Li and co-workers described Fe-catalyzed reactions of indoles with ethers for the synthesis of 1,1-bis-indolylmethane derivatives via C−H bond oxidation and C−O bond cleavage in the presence of (*t*-BuO)$_2$.[13] Wang reported Mn-catalyzed three-component synthesis of 1,5-amino/keto alcohols from Grignard reagents, imines/nitriles, and tetrahydrofuran (THF) through C−H and C−O bond cleavage of THF.[14] Sun presented the three-component reaction of 1-(2-aminoaryl)pyrroles, elemental sulfur, and ethers to access N-heterocycle-fused 1,3,6-benzothiadiazepines in the presence of the *n*-Bu$_4$NI/TBHP system.[15] As a

Scheme 1. Synthetic Strategies toward α-Aminophosphine Oxides via C(sp^3)−H Functionalization



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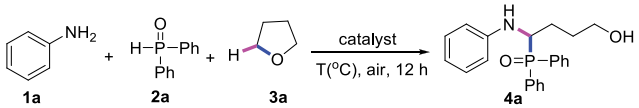
continuation of our interest in constructing phosphorus-containing compounds[16] and green organic synthesis,[17] here, we report a new and efficient method for the synthesis of various α-aminophosphine oxide derivatives via TEMPO-catalyzed aminophosphinoylation of ethers with amines and H-phosphine oxides, in which a mild and selective cleavage of the C(sp^3)−H and C(sp^3)−O bonds of ethers is achieved under metal- and external oxidant-free conditions.

Initially, the reaction of aniline (**1a**) and diphenylphosphine oxide (**2a**) was investigated in tetrahydrofuran (THF, **3a**) at room temperature under air. Interestingly, the product **4a** was formed in 23% isolated yield (Table 1, entry 1). We then tried

Table 1. Optimization of the Reaction Conditions[a]



1a 2a 3a 4a

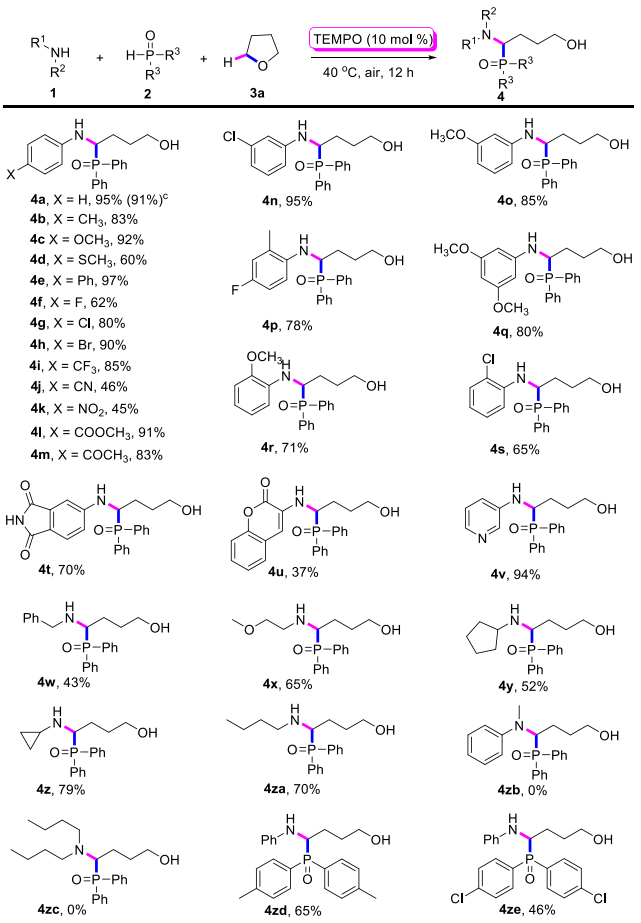
entry	catalyst (mol %)	T (°C)	yield[b] (%)
1		25	23
2	$CuBr_2$ (20)	25	6
3	$FeBr_3$ (20)	25	11
4	$Cu(OAc)_2·H_2O$ (20)	25	9
5	$AgNO_3$ (20)	25	24
6	AgOAc (20)	25	19
7	TEMPO (20)	25	82
8	TEMPO (50)	25	60
9	TEMPO (100)	25	67
10	TEMPO (15)	25	83
11	TEMPO (10)	25	87
12	TEMPO (5)	25	83
13	**TEMPO (10)**	**40**	**95**
14	TEMPO (10)	50	85
15	TEMPO (10)	60	76
16	TEMPO (100)	40	46[c]

[a]Reaction conditions: **1a** (0.5 mmol), **2a** (1.0 mmol), TEMPO (5−100 mol %), THF **3a** (2 mL), 12 h, air, 25−60 °C. [b]Isolated yield based on **1a**. [c]Under N_2.

to optimize the reaction conditions to improve the yield of **4a** (Table 1). As shown in entries 2−6, when various transition-metal catalysts such as Cu, Fe, and Ag salts (20 mol %) were added in the model reaction system, the reaction efficiency was not obviously improved. To our delight, significantly improved reaction efficiency was obtained when TEMPO was used as the catalyst (Table 1, entry 7). Encouraged by this result, the effects of TEMPO loading were further examined (Table 1, entries 8−12). The increase of TEMPO loading to 50 or 100 mol % did not improve the reaction efficiency (Table 1, entries 8 and 9). In contrast, the reaction efficiency was slightly improved along with the reduction of catalyst loading, and 10 mol % of TEMPO was found to be the best choice (Table 1, entry 11). Further investigation on the effect of the reaction temperature found that the highest isolated yield (95%) was obtained when reaction was conducted at 40 °C (Table 1, entry 13). Moreover, the desired product **4a** was only obtained in 46% yield when the reaction was carried out in the presence of stoichiometric amounts of TEMPO under N_2 (Table 1, entry 16).

Under the optimized conditions, the substrate scope of various amines and H-phosphine oxides were investigated. As summarized in Scheme 2, various aniline derivatives were first

Scheme 2. Scope of Amines and H-Phosphine Oxides[a,b]



[a]Reaction conditions: **1** (0.5 mmol), **2** (1.0 mmol), THF **3a** (2 mL), TEMPO (10 mol %), 12 h, air, 40 °C. [b]Isolated yield based on **1**. [c]**1a** (2 mmol), **2a** (4.0 mmol), THF **3a** (8 mL).

examined in this aminophosphinoylation reaction. Generally, *para*-substituted anilines bearing electron-rich groups or electron-withdrawing groups on the benzene rings reacted readily with **2a**, affording the corresponding products **4b−m** in moderate to good yields. It is worth noting that a wide range of functionalities such as halogen, trifluoromethyl, cyano, nitro, ester, and carbonyl groups were well tolerated in this procedure to give the desired products **4f−m**, which could be utilized for further modification. *Meta*-substituted anilines also worked well in this reaction to deliver the products **4n−q** in good yields. The steric hindrance of substituents on the benzene ring had a slightly effect on the reaction efficiency, and the corresponding products **4r** and **4s** were obtained in moderate yields. Notably, aromatic heterocyclic amine (i.e., pyridin-3-amine) and some complex amines (i.e., 5-amino-isoindoline-1,3-dione and 3-amino-2H-chromen-2-one) were also suitable substrates to generate the products **4t−v**. Furthermore, various aliphatic primary amines could also react smoothly to yield the corresponding products **4w−za**. Nevertheless, none of the desired products were detected when secondary amines were used in this reaction system (**4zb** and **4zc**). In addition, the methyl- and chlorine-substituted H-phosphine oxides were also examined under the optimized conditions, and the corresponding products **4zd** and **4ze** could be isolated in moderate yields. Unfortunately, H-phosphonates

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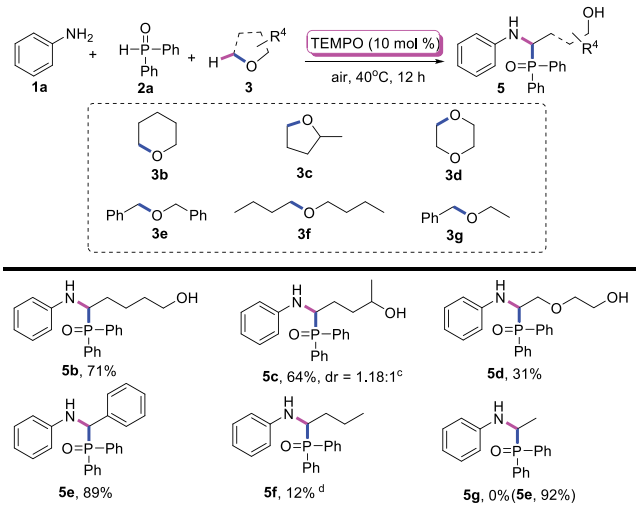
such as diethyl phosphite and dimethyl phosphite failed to provide the corresponding aminophosphonylation products. Moreover, when other nucleophiles such as indole, 1-cyclohexenylpiperidine, and 1,2,3-trimethoxybenzene were examined under the standard conditions, none of the desired product was detected.

Subsequently, the scope of ethers was examined under the optimal reaction conditions. As shown in Scheme 3, in

Scheme 3. Scope of Ethers[a,b]



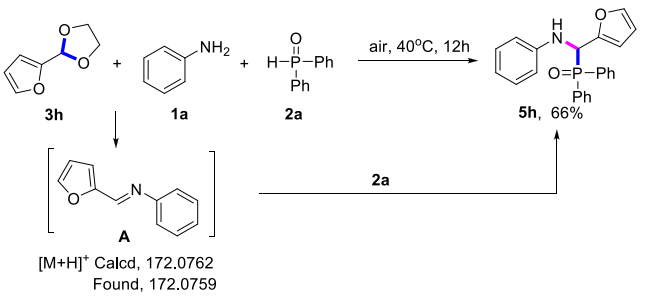
[a]Conditions: **1a** (0.5 mmol), **2a** (1.0 mmol), **3** (1 mL), TEMPO (10 mol %), 12 h, 40 °C, air. [b]Isolated yield based on **1**. [c]dr valueswere determined by ^{1}H NMR analysis of the mixture. [d]**3f** (2 mL), 60 °C.

addition to tetrahydrofuran, other cyclic ethers such as tetrahydro-2H-pyran, 2-methyltetrahydrofuran, and 1,4-dioxane were also suitable for this protocol, and the corresponding aminophosphinoylation products were obtained in moderate to good yields (**5b**−**d**). Moreover, linear ethers such as dibenzyl ether **3e** and dibutyl ether **3f** were compatible with this reaction, leading to the desired products **5e** and **5f**. It was found that the product **5e** was selectively obtained in 92% yield, and no product **5g** was detected when asymmetric ethyl benzyl ether **3g** was employed as the substrate.

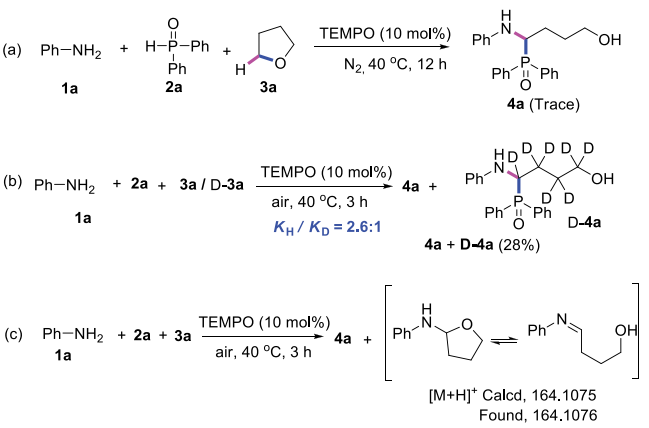
Notably, 2-(furan-2-yl)-1,3-dioxolane could react with aniline **1a** and diphenylphosphine oxide **2a** leading to the corresponding product **5h** in 66% yield in the absence of TEMPO catalyst (Scheme 4). The product **5h** might be generated from the addition of diphenylphosphine oxide **2a** with imine intermediate **A** that was detected by HRMS analysis.

Several control experiments were performed to understand the possible reaction process, and the results are compiled in Scheme 5. First, when the model reaction was carried out under N_2, only a trace amount of **4a** was detected, indicating that O_2 in air is essential for the present transformation (Scheme 5a). Subsequently, the intermolecular kinetic isotope experiment (KIE) was carried out under the standard reaction conditions, and the kinetic isotope effect (k_H/k_D = 2.6) was obviously observed (Scheme 5b). This result demonstrated that the C−H bond cleavage in tetrahydrofuran might be involved in the rate-determining step of this reaction (see the Supporting Information). Moreover, an HRMS experiment on the model reaction mixture was performed to detect the

Scheme 4. Formation of Product 5h in the Absence of TEMPO



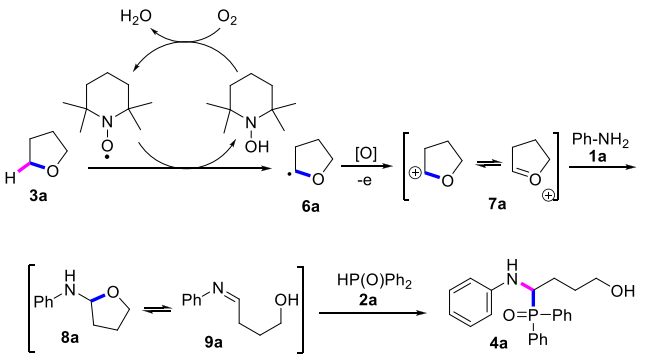
Scheme 5. Control Experiments



possible intermediate (Scheme 5c). In addition to the product **4a**, an imine intermediate derived from the corresponding aminal was detected (calcd for $C_{10}H_{14}NO$ $[M + H]^+$, 164.1075; found, 164.1076).

On the basis of the above observations and previous reports,[11c,12a,18] a plausible mechanism for the present transformation is described in Scheme 6. First, α-alkoxyalkyl

Scheme 6. Possible Reaction Mechanism



radical **6a** is produced via the hydrogen-transfer reaction between THF and TEMPO.[18a] The formed TEMPOH is oxidized by O_2 in air to regenerate TEMPO.[18b] Then the α-alkoxyalkyl radical **6a** undergoes further oxidation to give an electrophilic α-alkoxyalkyl cation **7a**.[11c,12a] Next, the nucleophilic attack of **1a** to **7a** affords unstable aminal **8a**, which will form imine **9a** owing to the electron-donating property of the nitrogen atom.[12a] Finally, the desired product **4a** was produced by nucleophilic addition of **2a** to **8a** or **9a**.

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In summary, we have successfully developed a new TEMPO-catalyzed aminophosphinoylation of ethers with amines and H-phosphine oxides under mild conditions via tandem C(sp^3)−H and C(sp^3)−O bond cleavage. The present methodology provides a facile and efficient approach to access a wide range of α-aminophosphine oxides in moderate to good yields from simple starting materials with excellent functional group tolerance. This reaction can be conducted under metal-free conditions without any added external oxidant, demonstrating great potential in practical applications. Further investigation of the detailed reaction mechanism and synthetic application are currently underway in our group.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b01081.

Experimental details and compound characterization (PDF)

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Notes

The authors declare no competing financial interest.

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