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Radical Cation Salt-initiated Aerobic C-H Phosphorylation of *N*-Benzylanilines: Synthesis of α-Aminophosphonates

Xiaodong Jia*, [a], Xiaofei Liu, [b] Yu Yuan, [a] Pengfei Li, [b] Wentao Hou, [b] and Kaixuan He [a]

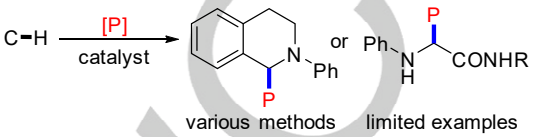
Dedication ((optional))

Abstract: A radical cation salt-initiated phosphorylation of *N*-benzylanilines was realized through the aerobic oxidation of sp^3 C-H bond, providing a series of α-aminophosphonates in high yields. The investigation of the reaction scope revealed that this mild catalyst system is superior in good functional group tolerance and high reaction efficiency. The mechanistic study implied that the cleavage of the sp^3 C-H bond was involved in the rate-determining step.

Due to the structural resemblance to natural and unnatural amino acids, α-aminophosphonates and their corresponding α-aminophosphonic acids exhibit various biological activities,[1, 2] such as antibacterial, [3] antifungal [4] and others.[5] They are also ubiquitous building blocks or ligands, and widely utilized in transition-metal catalysis and organocatalysis.[6] Therefore, the construction of C-P bond by concise and efficient methods is highly desirable in organic synthesis. Recently, with the development of C-H bond functionalization,[7, 8] direct phosphorylation of C-H bond has provided a more straightforward and efficient way for C-P bond formation. [9, 10, 11] In particular, direct phosphorylation of sp^3 C-H bond received more and more attention.[10, 11, 12] In 2009, Li reported an elegant copper-catalyzed phosphorylation of *N*-aryl tetrahydroisoquinolines (THIQ) via cross-dehydrogenative-coupling (CDC), using dialkyl phosphonates as the phosphorus source.[10a] Then, a series of catalyst systems were developed to promote the phosphorylation of *N*-aryl tetrahydroisoquinolines, such as copper complex, visible light, DDQ, iodine, gold complex, CBr_4 and so on.[10] In sharp contrast, investigation of more general sp^3 C-H bond phosphorylation remains limited. Yang and co-workers realized an oxidative coupling between α-amino carbonyl compounds and diphenylphosphine oxide to afford imidoylphosphonates, using TBHP (*tert*-butyl hydroperoxide) as a stoichiometric oxidant.[11] In 2016, Li's group achieved the first phosphorylation of glycine derivatives by copper catalysis, [12a] however, the reaction scope was not desirable, and only *N*-PMP glycine amides exhibited good reactivity.

Since 2012, our group has achieved a series of sp^3 C-H functionalization by using $TBPA^{+\cdot}$ [tris(4-bromophenyl)aminium hexachloroantimonate]/O_2 catalyst system, constructing various heterocycles in high efficiency.[13] In this research, $TBPA^{+\cdot}$ exhibited good reactivity to promote the aerobic oxidation of sp^3 C-H bond, and in most cases, a variety of functional groups can be tolerated. Recently, this catalyst system was further applied to the phosphorylation of glycine derivatives, [12b] and the results showed higher functional group tolerance and wider reaction scope, compared with reported approaches. So we questioned whether this mild catalyst system could be applied to more general substrates to realize efficient sp^3 C-H bond phosphorylation. Additionally, in our previous research, *N*-benzylanilines could be readily functionalized by $TBPA^{+\cdot}/O_2$ catalysis, [13c] which inspired us to investigate the aerobic oxidative phosphorylation of *N*-benzylanilines to construct aminophosphonate skeleton. Herein, we report the first direct phosphorylation of *N*-benzylanilines through a radical cation salt-induced aerobic oxidation of sp^3 C-H bond.

a) Popular substrates of C-H phosphorylation:



b) First phosphorylation of *N*-benzylanilines: ***This work***

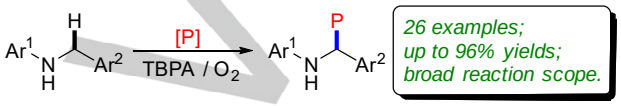


Figure 1. Phosphorylation of sp^3 C-H Bond

Initially, *N*-benzyl-*p*-toluidine **1a** was chosen as the model substrate to test the possibility of this direct C-H phosphorylation. Based on our previous reaction conditions,[12b] various commercially available phosphorus-containing compounds **2a**-**2d** were tested in the presence of 10 mol % $TBPA^{+\cdot}$ in MeCN under 1 atm dioxygen atmosphere at 60 °C. The results show that only diethyl phosphonate **2d** exhibited good reactivity, affording the desired phosphorylated product in excellent yield (entry 4). Then a brief solvent screen was performed (entries 5-7), and MeCN still gave the best result.

Table 1. Optimization of Reaction Conditions [a]

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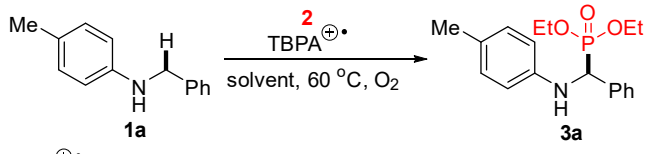
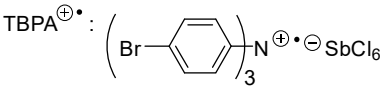


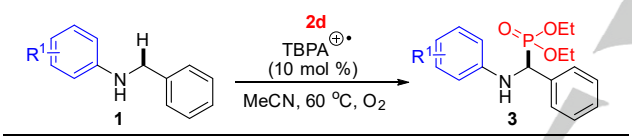


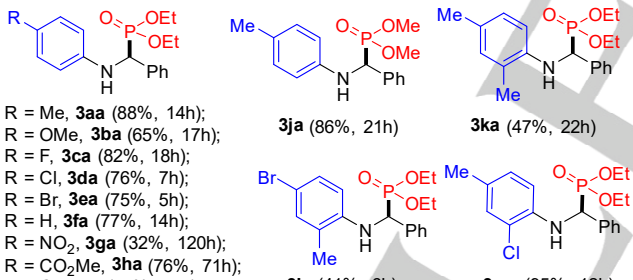
2a: $P(OEt)_3$; **2b**: $P(OEt)_3$; **2c**: $HP(O)Ph_2$; **2d**: $HP(O)(OEt)_2$

Entry	[P]	Solvent	Time (h)	Yield (%) b
1	2a	MeCN	24	trace
2	2b	MeCN	24	N. R.
3	2c	MeCN	24	trace
4	2d	MeCN	6	96
5	2d	PhOMe	5	60
6	2d	1,4-dioxane	7	82
7	2d	DCE	4	74

[a] Unless otherwise specified, the reaction was carried out with **1a** (0.1 mmol) and **2** (0.2 mmol) in the presence of $TBPA^{+\cdot}$, and anhydrous solvent (1.0 mL). [b] Yield of crude product ^1H NMR using 1,3,5-trimethoxylbenzene as internal standard.

Scheme 1. Scope of Aryl Groups on Anilines [a]

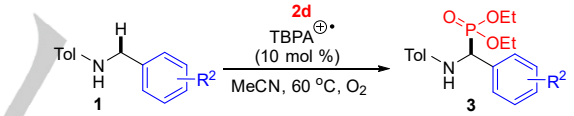


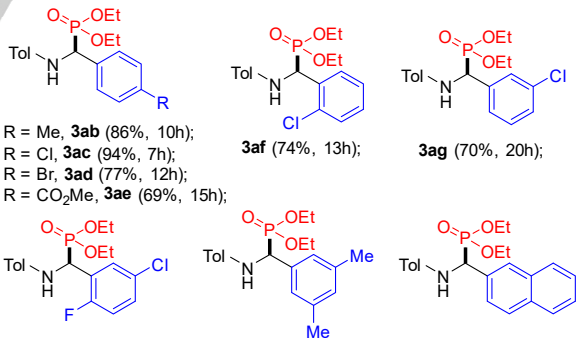


[a] Reaction conditions: **1** (1 mmol), **2d** (2 mmol), $TBPA^{+\cdot}$ (10 mol %), MeCN 5 mL), 60 °C under O_2, isolated yield.

With the optimized reaction conditions established, the investigation of the reaction scope was then performed. First, a variety of substituted anilines were tested (Scheme 1). Generally, anilines with various *para*-substituents were well tolerated, giving the desired product in high yields (**3aa-3ia**). Neither electron-donating such as Me and MeO nor electron-withdrawing groups such as halogen, ester and CN affected the reaction efficiency, affording the phosphorylated anilines in 65% to 96% yields. Only the strongly electron-withdrawing nitro group provided the expected **3ga** in lower yield with poor starting material conversion (35%). This is attributed to the lower electron density on nitrogen, decreasing the activity of the sp^3 C-H bond adjacent to nitrogen atom. When dimethyl phosphonate was employed, the reaction efficiency was not decreased, providing the corresponding α-aminophosphonate **3ja** in 86% yield. Then the more sterically hindered anilines were evaluated (**3ka-3oa**). When a bulky methyl group exists on the *ortho*-position of the phenyl ring, obviously lower yields were observed (compared **3ka** and **3la** with **3ma**), suggesting that this C-H bond phosphorylation was greatly affected by the steric hindrance. When *ortho*-isopropylaniline was employed, the C-H phosphorylation was thoroughly inhibited, and no phosphorylated product was detected (**3ra**). The reaction of *p-tert*-butyl group substituted aniline was also performed, and the desired product **3qa** were isolated in lower yield. In particular, when the reaction of 1,3,5-trimethylaniline was performed under the standard reaction conditions, the desired product **3oa** was isolated in only 21% yield, together with the decomposed products, 2,4,6-trimethylaniline and benzaldehyde. Halogen substituents on the *ortho* position did not exert a negative effect on this reaction, and the corresponding phosphorylated compounds were obtained in high yields (**3ma-3na**). Naphthylamine was also compatible with this reaction, generating the desired product in 76% yield (**3pa**).

Scheme 2. Scope of Substituents on Benzyl Groups [a]





[a] Reaction conditions: **1** (1 mmol), **2d** (2 mmol), $TBPA^{+\cdot}$ (10 mol %), MeCN (5 mL), 60 °C under O_2, isolated yield.

With the success of reaction between dialkyl phosphonate and various *N*-benzyl anilines, the substituents on the benzyl group were then investigated (Scheme 2). Overall, the substituents on the benzyl ring did not affect the reaction efficiency significantly. When *para*-substituted benzyl groups exist (**3ab-3ae**), good yields were obtained, and only an ester group gave slightly a lower yield (**3ae**). The substrates with *ortho*- and *meta*- groups react smoothly with diethyl phosphonate, providing the desired α-

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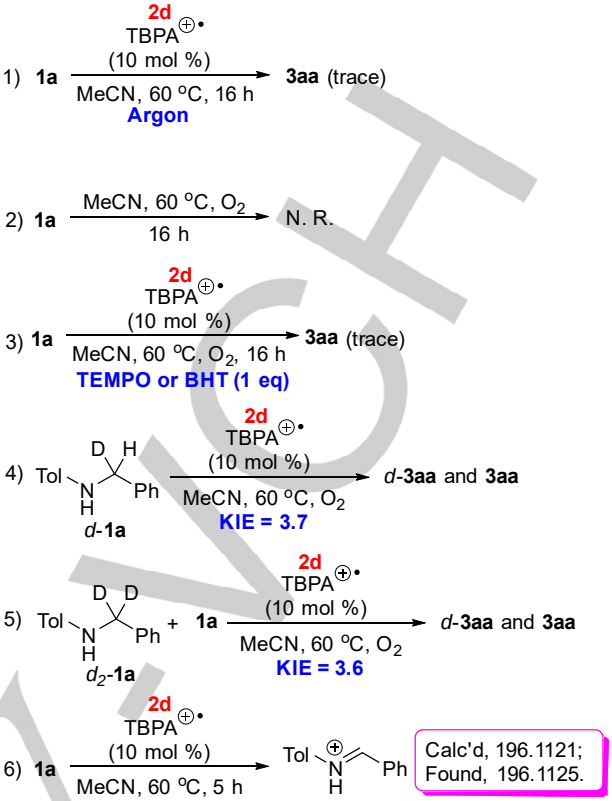
aminophosphonate in good yields (**3af-3ag**). When disubstituted *N*-tolylbenzylamines were employed, the reaction efficiency was not influenced, and the phosphorylated products were isolated in comparable yields (**3ah-3ai**). Due to the electron-rich nature, the α-position of the naphthalene ring tends to be oxidized under oxidative conditions. We then examined the reaction of 4-methyl-*N*-(naphthalen-2-ylmethyl)aniline. To our satisfaction, the C-H bond adjacent to nitrogen was phosphorylated selectively, and the naphthalene ring remained unchanged, highlighting this mild oxidation (**3aj**).

To reveal more details of the mechanistic process, several control experiments were performed, and the results are compiled in Scheme 3. In the absence of dioxygen, only a trace amount of the phosphorylated product was detected, implying that dioxygen was involved in this C-H phosphorylation (eq 1). However, when $TBPA^{+\cdot}$ was absent, no reaction occurred (eq 2). Therefore, dioxygen and $TBPA^{+\cdot}$ are both crucial to realize this C-H phosphorylation, and the aerobic oxidation was initiated by $TBPA^{+\cdot}$. Then we conducted the oxidative phosphorylation in the presence of the radical inhibitor TEMPO and BHT, respectively, and the reactions were totally inhibited (eq 3), supporting the existence of the radical intermediate. Subsequently, the intramolecular and intermolecular KIE experiments were conducted under the standard reaction conditions, which afforded KIE values of 3.7 and 3.6, respectively (eq 4 and 5). These KIE values indicate that the C–H bond cleavage might be involved in the rate-determining step. Next, to detect the intermediate, a HRMS experiment on the reaction mixture was performed (eq 6), and an iminium intermediate derived from the corresponding *N*-benzylaniline was successfully detected (calcd for $C_{14}H_{13}N + H^+$, 196.1121; found, 196.1125). This result verified that the C-P bond is constructed through nucleophilic addition between dialkyl phosphate and the generated iminium intermediate.

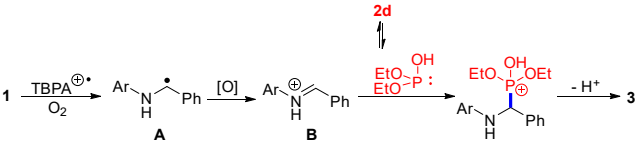
Scheme 3. Control Experiments



Based on the results of the control experiments and previous reports,[7] a possible mechanism was proposed in Scheme 4. In the presence of $TBPA^{+\cdot}$ and dioxygen, a radical intermediate **A** was generated, which was further oxidized to an iminium intermediate **B**. On the other hand, it is well-known that the dialkyl phosphonate **2d** is in equilibrium with the nucleophilic phosphite species.[14] Then the generated iminium intermediate was attacked by this reactive phosphite form, and after deprotonation, the desired α-aminophosphonate **3** was produced.

In conclusion, an efficient aerobic phosphonylation of *N*-benzylanilines is achieved by radical cation salt initiated sp^3 C-H bond oxidation. Compared with the reported catalyst system, this reaction is superior in better functional group tolerance, wider reaction scope and higher reaction efficiency. This method provides a simple way to construct biologically and synthetically important α-aminophosphonates from more general starting material. Further applications and more variants of direct C-H phosphonylation are still under investigation in our laboratory.

Scheme 4. Proposed Mechanism



Experimental Section

General Experimental Procedure

A solution of **1** (1 mmol)**, 2d** (2 mmol) in MeCN (5 ml) was mixed fully, then $TBPA^{+\cdot}$ (10 mol %) was added dropwise. The reaction solution was stirred at 60ºC under O_2 atmosphere. After completion monitored by TLC (by UV visualization), the reaction was quenched by addition of NEt_3 (1 ml). The mixture was poured into a separator funnel with the addition of excess DCM (10 ml), and then the crude organic solution was extracted three times with water to remove inorganic salts. The organic phase was then dried over anhydrous magnesium sulfate, filtered, and the solvent was removed under reduced pressure. The products were separated by silica gel column chromatography eluted with petroleum ether/acetone (v/v 5:1) to afford the products.

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Keywords: Radical cation salt • C-H functionalization • *N*-Benzylaniline • Phosphorylation • α-Aminophosphonate

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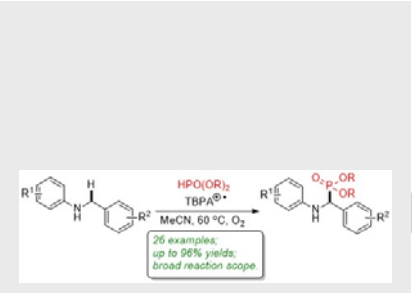

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Radical Cation Salt-initiated Aerobic C-H Phosphorylation of *N*-Benzylanilines: Synthesis of α-Aminophosphonates

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Page No. – Page No.

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