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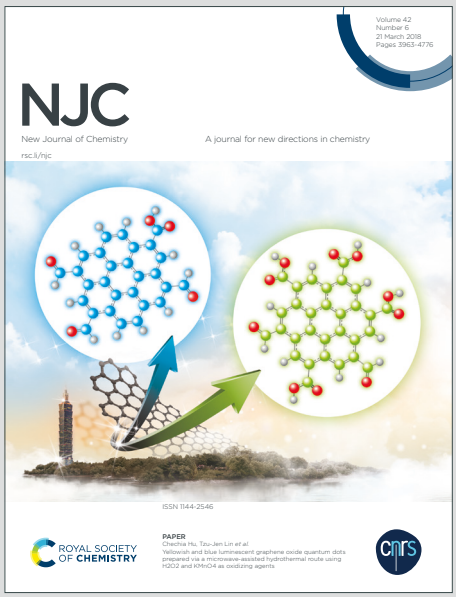

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COMMUNICATION

Silver-Mediated Radical Phosphorylation/Cyclization of *N*-Allylbenzamides to Access Phosphoryl-Substituted Dihydroisoquinolones

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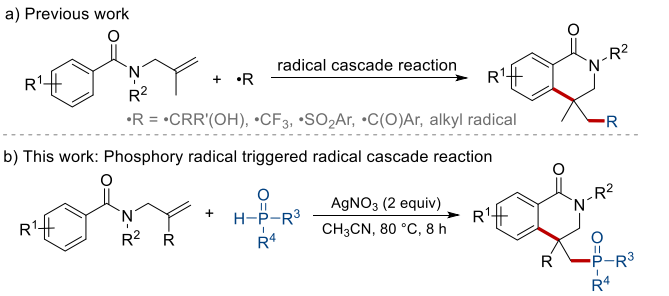
Xiao-Ceng Liu,[a,#] Kai Sun,[a,#] Qi-Yan Lv,[a,b] Xiao-Lan Chen,[a,*] Yuan-Qiang Sun,[a,*] Yu-Yu Peng,[c] Ling-Bo Qu,[a] Bing Yu,[a,b*]

A silver-mediated phosphorylation/cyclization of *N*-allylbenzamides with phosphine oxides for the synthesis of phosphoryl-substituted dihydroisoquinolones was developed. This protocol features broad substrate scope, experimental simplicity, high efficiency, step-economy, and facile scalability.

Dihydroisoquinolones, as privileged structure of alkaloids, have been widely found in complex natural products, biomolecules and pharmaceuticals, which display extraordinary promising biological activities such as anti-tumor, anti-nausea, anti-inflammatory and even their wide applications in cancer diagnosis.[1] Consequently, increasing efforts have been devoted to the development of novel and efficient methods to access diverse functionalized dihydroisoquinolones in recent years.[2] Among the reported of methods, radical cascade cyclization strategies[3] have proved to be efficient and attractive routes for the synthesis of dihydroisoquinolones from *N*-allylbenzamides with simple operation and high step-economy.[4] For example, radicals including •CRR'(OH), •CF_3, •SO_2Ar, •C(O)Ar, and alkyl radical have been successfully applied for the radical cascade reactions (Scheme 1a).[5] These above-mentioned elegant strategies suggested that *N*-allylbenzamides has tremendous potential for the construction of novel dihydroisoquinolones derivatives.

Phosphorus-containing groups as important structural moieties are widespread in drugs, active molecules, pesticides and functional materials.[6] It is well known that medicinal properties, biological responses as well as material functions could be effectively modified by introducing phosphorus substituents.[7] Consequently, the installation of phosphorus substituents into valuable frameworks has attracted considerable attention of synthetic chemists and pharmacologists.[8] Inspired by previous elegant works and our interest in green chemistry and radical reactions,[9] we herein present a novel and efficient silver-mediated strategy, by which a large variety of phosphoryl-substituted dihydroisoquinolones were prepared *via* the reaction of *N*-allylbenzamides and phosphine oxides (Scheme 1b).



Scheme 1. The construction of dihydroisoquinolones

We commenced our investigations using *N*-methyl-*N*-(2-methylallyl)benzamide (**1a**) and diphenylphosphine oxide (**2a**) as model substrates to optimize the conditions, as summarized in Table 1. Gratifyingly, by employing 2 equiv of $AgNO_3$, the reaction of substrate **1a** with diphenylphosphine oxide **2a** was smoothly performed in DMSO for 8 h to afford the desired phosphoryl-substituted dihydroisoquinolones **3a** in 46% yield (entry 1). Encouraged by this exciting result, several different solvents, including CH_3CN, DCM, THF, MeOH, toluene, acetone, 1,4-dioxane as well as DMF, were examined to improve the reaction efficiency (entries 2-9). Much to our delight, when the reaction was performed in CH_3CN, a satisfactory yield (76%) was obtained (entry 2). Furthermore, different silver salts, including AgOAc, $AgSbF_6$, Ag_2O, AgF_2, AgOTf, and Ag_3PO_4 were screened (entries 10-16). As a result, we found that $AgNO_3$ was the most effective reagent for this reaction among all the Ag salts tested. Afterward, the amount of $AgNO_3$ was investigated. It was found that the yield markedly increased from 30% to 76% along with the increase of the amount of $AgNO_3$ from 1 equiv to 2 equiv (entry 17 vs and entry 2), and then, a slight decrease was observed by

[a.] *College of Chemistry, Zhengzhou University, Zhengzhou 450001, China. Email: chenxl@zzu.edu.cn, bingyu@zzu.edu.cn.*
[b.] *Henan Nonferrous Metals Geological Exploration Institute, Zhengzhou 450052, Henan Province, China*
[c.] *Hunan Provincial Key Laboratory of Materials Protection for Electric Power and Transportation, Changsha University of Science & Technology, Changsha 410114, China*
# Xiao-Ceng Liu and Kai Sun contributed equally
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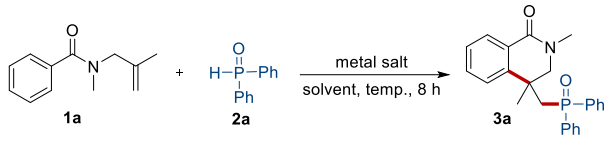
continuous increasing the amount of $AgNO_3$ to 3 equiv (entry 18). Investigation of temperature and reaction time was also conducted, and the results showed that 80 °C and 8 h were still the best choices (entries 19-21). The experiment without silver salt implied that $AgNO_3$ is necessary for this transformation (entry 22). Attempts to lower the loading of $AgNO_3$ failed and the yield of **3a** decreased significantly (See Supporting Information for details).[10] After extensive experimentation, the optimized reaction conditions were established as follows: **1a** (0.5 mmol), **2a** (1 mmol), $AgNO_3$ (2 equiv), CH_3CN (3 mL) under 80 °C for 8 h.

Table 1. Optimization of the reaction conditions[a,b]

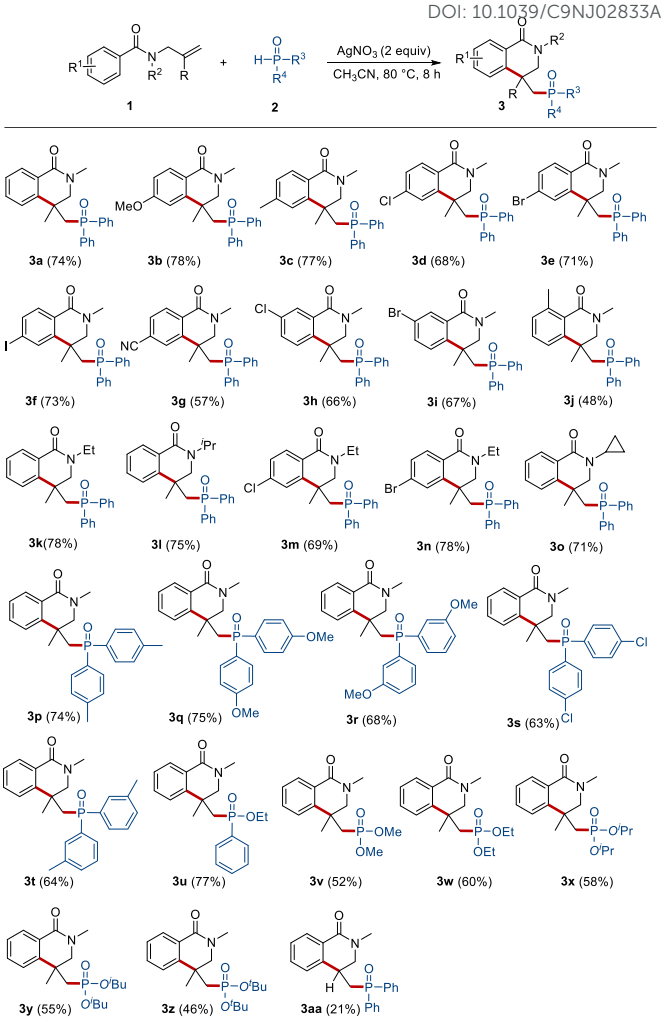


entry	metal salt (equiv)	Solvent	Yield (%)
1	$AgNO_3$ (2)	DMSO	46
2	$AgNO_3$ (2)	CH_3CN	76 (74)[b]
3	$AgNO_3$ (2)	DCM	11
4	$AgNO_3$ (2)	THF	20
5	$AgNO_3$ (2)	MeOH	N.D.
6	$AgNO_3$ (2)	toluene	N.D.
7	$AgNO_3$ (2)	acetone	N.D.
8	$AgNO_3$ (2)	1,4-dioxane	26
9	$AgNO_3$ (2)	DMF	trace
10	Ag_2CO_3 (1)	CH_3CN	trace
11	AgOAc (2)	CH_3CN	trace
12	$AgSbF_6$ (2)	CH_3CN	trace
13	Ag_2O (1)	CH_3CN	21
14	AgF_2 (2)	CH_3CN	19
15	AgOTf (2)	CH_3CN	N.D.
16	Ag_3PO_4 (0.67)	CH_3CN	N.D.
17	$AgNO_3$ (1)	CH_3CN	30
18	$AgNO_3$ (3)	CH_3CN	72
19[c]	$AgNO_3$ (2)	CH_3CN	55
20[d]	$AgNO_3$ (2)	CH_3CN	25
21[e]	$AgNO_3$ (2)	CH_3CN	68.
22	----	CH_3CN	N.D.

[a] Reaction conditions: **1a** (0.5 mmol), **2a** (1 mmol), silver salt, solvent (3 mL) under 80 °C for 8 h. N.D. = Not detected. Yields were determined by ^{31}P NMR using trioctylphosphine oxide as internal standard. [b] Isolated yield. [c] At 60 °C. [d] For 4 h. [e] For 12 h.

With the optimized reaction conditions in hand, we next evaluated the substrate scope of this silver-mediated transformation by examining various *N*-allylbenzamides **1** and phosphine oxide components **2**, and the results are summarized in Table 2. As can be seen, different *N*-allylbenzamides, with either electron-donating groups (-Me, -OMe) or electron-withdrawing groups (-Cl, -Br, -I, -CN), reacted smoothly with diphenyl phosphine oxide **2a** to access the corresponding phosphoryl-substituted dihydroisoquinolones **3a-j** in moderate to excellent yields (48-84%). It should be noted that *N*-allylbenzamides which bear electron-donating groups gave slightly higher yields compared to those with electron-withdrawing substituents. Additionally, different *N*-substituted allylbenzamides

Table 2. Substrate scope for phosphoryl-substituted dihydroisoquinolones[a]

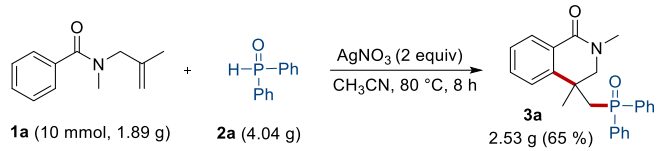


[a] Reaction conditions: **1** (0.5 mmol), **2** (1 mmol), $AgNO_3$ (2 equiv), CH_3CN (3 mL) under 80 °C for 8 h. Isolated yields were given.

bearing methyl, ethyl, *iso*-propyl, and cyclopropyl groups on N-position and halides on phenyl ring were also converted into the target products **3k-o** in good yields (69-78%). Afterward, *N*-methyl-*N*-(2-methylallyl)benzamide (**1a**) was employed to react with various organophosphorus reagents. To our delight, diarylphosphine oxides, bearing different substituents were smoothly react with **1a** to access the desired products **3p-t** in moderate to good yields (63-75%). When ethyl phenylphosphinate was employed as phosphorus reagent, the desired product **3u** was isolated in 77% yield. Moreover, five dialkyl *H*-phosphonates, including dimethyl *H*-phosphonate, diethyl *H*-phosphonate, di-*iso*-propyl *H*-phosphonate, di-*iso*-butyl *H*-phosphonate as well as di-*tert*-butyl *H*-phosphonate were found to be suitable in this protocol, giving the corresponding target products **3v-z** in moderate yields (46-60%). However, when *N*-allyl-*N*-methylbenzamide was employed as reaction substrate, the desired products **3aa** were only obtained in 21% yield. Moreover, *N*-methyl-*N*-(2-phenylallyl)benzamide was not suitable for this reaction.

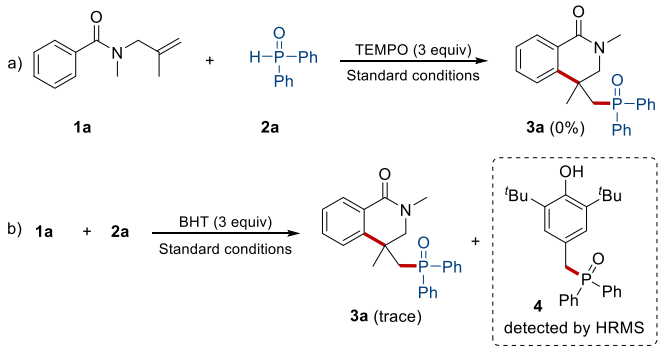
Furthermore, the scalability of this novel protocol was investigated by performing the model reaction in 10 mmol scale.

Pleasingly, it is found that the reaction proceeded smoothly by affording **3a** in 65% isolated yield (Scheme 2).



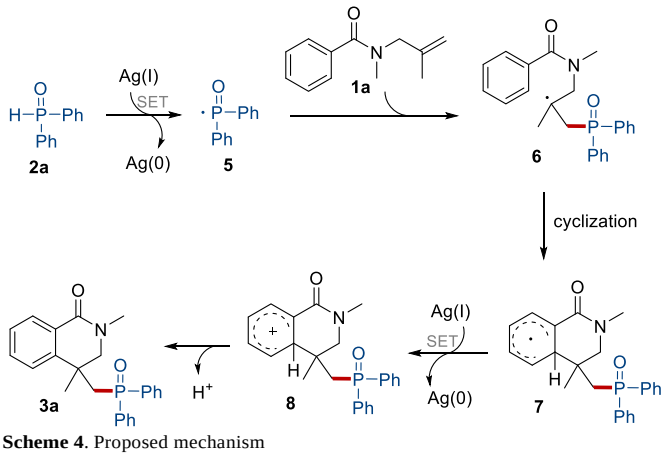
Scheme 2. Gram-scale synthesis of **3a**

To gain deep mechanistic insight into this transformation, several control experiments were conducted. As shown in Scheme 3, when the model reaction was performed respectively in the presence of two widely used radical scavengers like TEMPO (2,2,6,6-tetramethyl-1-piperidinyloxy) or BHT (2,6-di-*tert*-butyl-4-methylphenol), the reactions were remarkably suppressed, suggesting that a radical pathway might be involved in the reaction process. Moreover, when BHT was added into the model reaction under optimized reaction conditions, the adduct **4** was successfully detected by high-resolution mass spectrometry (HRMS), implying that diphenylphosphoryl radical was produced in these cyclization reactions.



Scheme 3. Control experiments

Based on our experimental results and previous reports,[11] a reasonable mechanism for this silver-mediated cascade cyclization was proposed as shown in Scheme 4. Initially, $AgNO_3$ reacted with diphenyl phosphine oxide **2a** to produce diphenylphosphine oxide radical **5**, which then added to the C=C bond of **1a** regioselectively to produce the radical intermediate **6** with the formation of C-P bond. Then, radical intermediate **6** underwent an intramolecular cyclization to generate the radical intermediate **7**, which was further oxidized to the carbocation intermediate **8** by Ag(I) *via* a single electron transfer (SET) process. Finally, intermediate **8** was immediately converted into the product **3a** by deprotonation.



Scheme 4. Proposed mechanism

Conclusions

In conclusion, we have developed a novel and straightforward protocol by which a large variety of phosphoryl-substituted dihydroisoquinolones were prepared *via* the radical cascade reaction of easily available *N*-allylbenzamides and phosphine oxides in the presence of $AgNO_3$. This simple and step-economy procedure has displayed broad substrate scope, good scalability and high efficiency under mild reaction conditions. Further exploration of this synthetic strategy will be conducted in due course.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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Xiao-Ceng Liu,[a,#] Kai Sun,[a,#] Qi-Yan Lv,[a,b] Xiao-Lan Chen,[a,*] Yuan-Qiang Sun,[a,*] Yu-Yu Peng,[c] Ling-Bo Qu,[a] Bing Yu,[a,b,*]

[a]*College of Chemistry and Molecular Engineering, Zhengzhou University, Zhengzhou 450001, China. Email: chenxl@zzu.edu.cn, bingyu@zzu.edu.cn.*

[b]*Henan Nonferrous Metals Geological Exploration Institute, Zhengzhou 450052, Henan Province, China*

[c]*Hunan Provincial Key Laboratory of Materials Protection for Electric Power and Transportation, Changsha University of Science & Technology, Changsha 410114, China*