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Metal-Free One-Pot Synthesis of 3-Phosphinoylbenzofurans via Phospha-Michael Addition/Cyclization of H-Phosphine Oxides and *in Situ* Generated *ortho*-Quinone Methides

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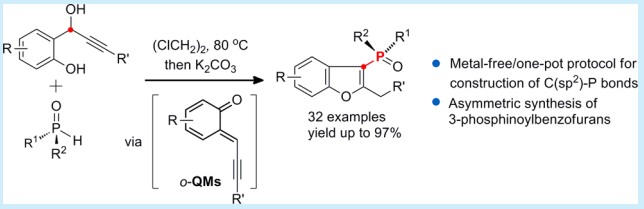
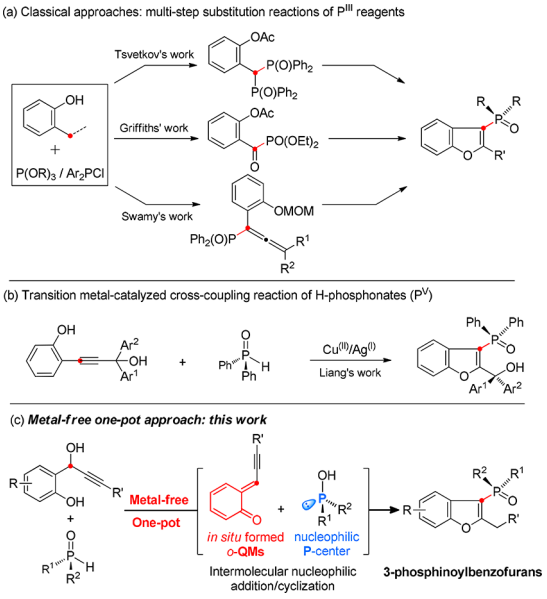
Supporting Information

ABSTRACT: A novel metal-free one-pot protocol for the effective and efficient synthesis of 3-phosphinoylbenzofurans via a phospha-Michael addition/cyclization of H-phosphine oxides and *in situ* generated *ortho*-quinone methides is described. Based on the expeditious construction of C(sp^2)−P bonds, asymmetric synthesis of optically pure 3-phosphinoylbenzofurans containing chiral P-stereogenic center has also been probed by using chiral R_P-(−)-menthyl phenylphosphine oxide.



Aromatic organophosphorus compounds and their derivatives are of particular interest in organic chemistry due to their biological properties and synthetic value.[1] For example, some structurally related organophosphorus compounds have been used as biologically active molecules in medicinal chemistry,[1b,d] as phosphorus ligands in asymmetric catalysis,[1a,f] and as buildings blocks in organic synthesis.[1c] In recent decades, focusing on the synthesis of phosphorylated heterocycles through C(sp^2)−P bond formation, several methodologies have been established on the basis of classical reactions of halophosphine electrophiles with active carbon nucleophiles such as organometallic reagents,[2] transition-metal-catalyzed cross-coupling reactions of phosphines with aryl (pseudo) halides,[3,4] Friedel−Crafts reaction,[5] and P-centered radicals addition to unsaturated systems.[6] Despite significant progress in the construction of C(sp^2)−P bonds, only a few approaches (Scheme 1) have been developed for the synthesis of 3-phosphinoylbenzofurans.[7] In approach (a), for example, Tsvetkov and Griffiths have developed the protocols for the synthesis of 3-phosphinoylbenzofurans using bisphosphine oxides and dialkyl benzoylphosphonates through Wittig−Horner and rearrangement reactions, respectively.[7a,c] In 2012, Swamy and co-workers reported a three-step synthetic procedure using allenylphosphine oxides as key intermediates.[7b,d] Among the above-mentioned approaches, notably, the C(sp^2)−P bonds were constructed through traditional substitution reactions using water-sensitive P^{III} reagents. Recently, Liang and co-workers have reported a novel synthesis of 3-phosphinoylbenzofuran derivatives via a copper(II) catalyzed intermolecular cascade annulation reaction of nucleophilic diphenylphosphine oxide and propargylic alcohols.[7e] However, the above-mentioned methodologies generally either require expensive metal catalysts or suffer from multistep preparation of the reaction precursors in some cases.

Scheme 1. Previous Methods for C(sp^2)−P Bond Construction and Our Design for the Synthesis of 3-Phosphinoylbenzofurans



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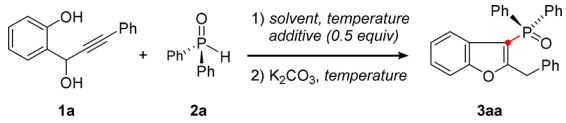
Recently, metal-free C(sp^2)−P bond construction reactions have attracted much attention as appealing alternatives to the metal-mediated and environmentally friendly processes.[8] For example, Miura et al. developed a metal-free approach to phosphorylated heterocycles using Tf_2O through electrophilic phosphination/cyclization of alkynes.[8h] Despite these elegant improvements, there is still high demand for novel methodologies to efficiently and effectively construct C(sp^2)−P bonds for the synthesis of 3-phosphinoylbenzofuran compounds.

ortho-Quinone methides (*o*-QMs) are highly reactive as well as ephemeral intermediates which have been employed for decades in the synthesis of natural products and bioactive molecules.[9] Due to their intrinsic electrophilic reactivity, *o*-QMs have been generally used in a series of 1,4-conjugate addition, [4 + *n*]-cycloaddition, intramolecular [5 + 2]-cycloaddition, and oxa-6π-electrocyclization.[10] Very recently, the Kang group developed a novel organocatalytic phosphonylation through 1,4-conjugate addition of *in situ* formed *o*-QMs with trialkylphosphites for the construction of diaryl phosphonates.[11] To our knowledge, however, there are few reports on the reaction of *o*-QMs as nucleophile acceptors with H-phosphine oxides. In light of the importance of 3-phosphinoylbenzofurans compounds and our interest in the development of transformations with *o*-QMs, herein we report an effective one-pot metal-free method for C(sp^2)−P bond formation through intermolecular phospha-Michael addition/intramolecular cyclization reaction (Scheme 1), wherein we envision that both nucleophilic P-center and electrophilic *o*-QMs would be generated *in situ* from H-phosphine oxides and chemically stable *o*-hydroxyl-benzyl alcohols (*o*-HBAs) under metal-free conditions, respectively.[12]

With the above-mentioned consideration, we initiated our screenings for the model reaction of *o*-hydroxyl-benzyl alcohol (*o*-HBA) **1a** and diphenylphosphine oxide **2a** under metal-free reaction conditions (Table 1). Initially, several acids such as TsOH, AcOH, and TFA were chosen for this transformation at ambient temperature in CH_3CN to facilitate the generation of *o*-QM intermediates. Pleasingly, the desired product **3aa** was obtained, despite low yields (entries 1−3). By considering the acidic instability of *in situ* formed *o*-QM intermediates, this model reaction was examined without an acid additive at increased temperature (80 °C). Surprisingly, the titled reaction proceeded smoothly and gave an improved yield of 82%, clearly demonstrating that the temperature enhancement could positively promote the formation of *o*-QMs (entry 4). In an attempt to further improve the yield, several solvents were examined (entries 5−9). With exception of polar DMF (entry 9), the desired product **3aa** could be obtained in good yields using toluene, EtOAc, $(ClCH_2)_2$, CH_3CN, and THF, in which $(ClCH_2)_2$ as the optimal solvent afforded **3aa** in 92% yield (entry 5).

With the optimal reaction conditions in hand, the scope of *in situ* generated *o*-QMs in our metal-free, one-pot reaction was first examined. A series of *o*-HBAs as *o*-QM precursors were tested in the presence of diphenylphosphine oxide (**2a**) (Scheme 2). The substrates bearing electron-donating sub-

Table 1. Optimization of the Reaction Conditions[a]

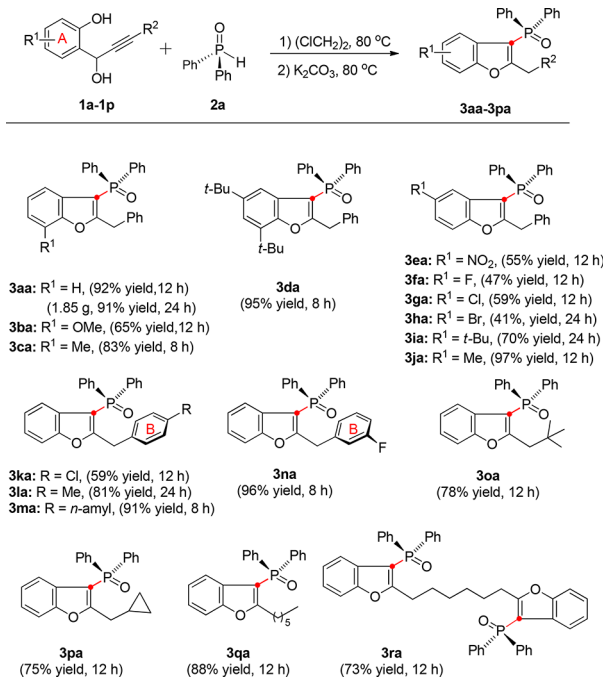


1a 2a 3aa

entry	additive	solvent	temp (°C)	yield (%)[b]
1	TsOH	CH_3CN	25	14
2	AcOH	CH_3CN	25	29
3	TFA	CH_3CN	25	42
4	–	CH_3CN	80	82
5	–	**$(ClCH_2)_2$**	**80**	**92**
6	–	toluene	80	75
7	–	EtOAc	80	76
8	–	THF	80	79
9	–	DMF	80	0

[a]A Schlenk tube (15 mL) charging with a magnetic stirring bar and **1a** (0.2 mmol, 1.0 equiv), **2a** (0.3 mmol, 1.5 equiv), solvent (2 mL) and additive (0.1 mmol, 0.5 equiv) (if applicable) was sealed without degassing and heated at the indicated temperature until **1a** was disappeared; and then K_2CO_3 as base (0.6 mmol, 3.0 equiv) was added to the resulting mixture and stirred at the indicated temperature. [b]Yield of isolated product. TFA = trifluoroacetic acid.

Scheme 2. Scope of *o*-Hydroxyl Benzyl Alcohols (*o*-HBAs)[a,b]



[a]Unless otherwise specified, reactions were carried out using **1** (0.2 mmol) and **2a** (0.3 mmol) in $(ClCH_2)_2$ (2 mL) at 80 °C until **1** disappeared, and then K_2CO_3 (0.6 mmol) was added. [b]Times listed were for the whole one-pot process.

stituents including methyl (**3ca**, **3ja**), *tert*-butyl (**3da**, **3ia**), and methoxy (**3ba**) groups at ring A afforded the corresponding products in 65−97% yields. The nitro group and halogen (F, Cl, and Br) containing substrates at ring A gave **3ea**−**3ha** in moderate yields under the standard reaction conditions, providing possibilities for late-stage chemical transformations of the products. Besides, aryl and alkyl acetylene based substrates were also screened, affording products (**3ka**−**3ra**) in good to excellent yields. Substrates with different aryl (e.g., 4-MeC_6H_4, 4-ClC_6H_4, 4-*n*-amyl-C_6H_4, 3-FC_6H_4) substituted acetylene motifs were further investigated, and the desired 3-phosphinoylbenzofurans **3ka**−**3na** were afforded in 59−96% yields. Besides, the substrates having alkyl (e.g., *tert*-butyl,

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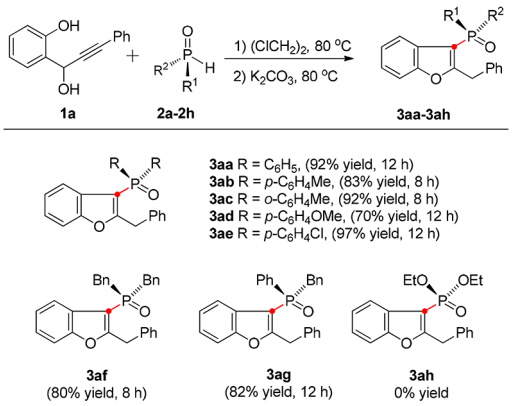
cyclopropyl, and *n*-amyl) substituted acetylene units were also tolerable under standard reaction conditions, giving the desired products **3oa** (78%), **3pa** (75%), and **3qa** (88%), respectively. Additionally, one example using the symmetric *o*-HBA substrate **1r** was conducted, and interestingly bis-3-phosphinoylbenzofuran product **3ra** was smoothly delivered in 73% yield. To test the synthetic potential of this protocol, we carried out a gram-scale reaction with 5 mmol (1.12 g) of **1a**, and the titled product **3aa** was obtained with analogous reactivity in good yield (91%, 1.85 g).

Next, we explored the scope with respect to the H-phosphine oxides (Scheme 3). Gratifyingly, the reaction of H-phosphine

Scheme 3. Scope of H-Phosphine Oxides[a,b]

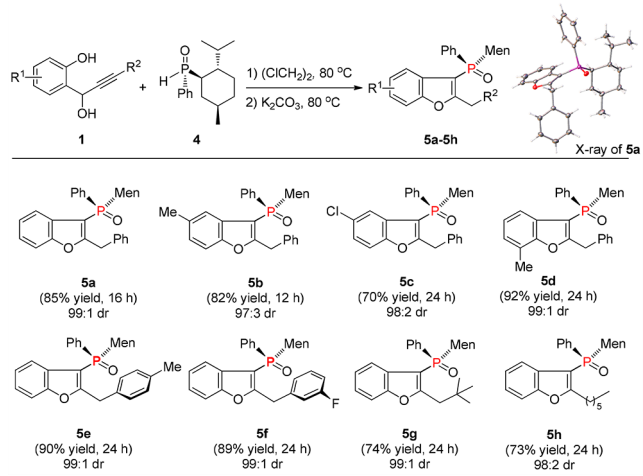


[a]Unless otherwise specified, reactions were carried out using **1a** (0.2 mmol) and **2** (0.3 mmol) in $(ClCH_2)_2$ (2 mL) at 80 °C until **1a** disappeared, and then K_2CO_3 (0.6 mmol) was added. [b]Times listed were for the whole one-pot process.

oxides bearing the electron-donating groups including methyl (**2b**, **2c**) and methoxy (**2d**) at the *para* or *ortho* position of the phenyl ring with *o*-HBA (**1a**) under optimal conditions readily gave products **3ab**–**3ad** in 83%, 92%, and 70% yields, respectively. The substrate **2e** with electron-withdrawing chlorine at the phenyl ring could also proceed smoothly, affording the desired product **3ae** in 97% yield. While di(benzyl)phosphineoxide (**2f**) was introduced as the reactant, the product **3af** was obtained in good yield (80%). Moreover, the unsymmetrical phosphine oxide (**2g**) was also effective for this transformation, giving the desired product **3ag** in 82% yield. Unexpectedly, diethyl phosphonate (**2h**) was ineffective in this reaction following the decomposition of the starting materials.

Recently, optically pure R_P-(−)-menthyl phenylphosphine oxide **4** was used as an excellent building block for the intramolecular rearrangement reaction and intermolecular addition reaction in our lab, leading to various optically pure tertiary phosphine oxides compounds.[13] Considering the importance of chiral P-stereogenic phosphorus compounds in organic synthesis, the reactions using the chiral H-phosphine oxide were further explored in the asymmetric synthesis of chiral 3-phosphinoylbenzofurans through phospha-Michael addition/cyclization reactions. As shown in Scheme 4, various *o*-HBAs were examined in this metal-free, one-pot stereoselective reaction. Generally, all the reactions gave the corresponding chiral 3-menthylphenylphosphinoylbenzofurans (**5a**–**5h**) with very high diastereoselectivity in good yields (up to 92% yield). The absolute configuration of **5a** was

Scheme 4. Stereoselective Synthesis of Chiral 3-Menthylphenylphosphinoylbenzofurans[a,b,c]

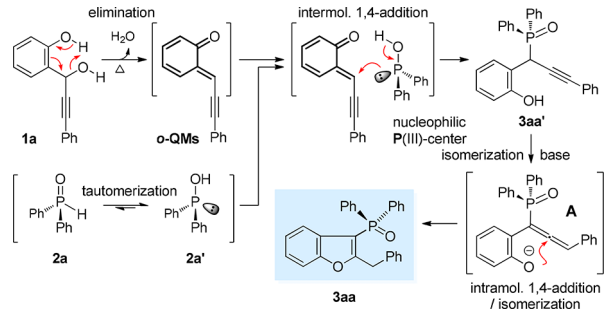


[a]Unless otherwise specified, reactions were carried out using **1** (0.2 mmol) and **4** (0.3 mmol) in $(ClCH_2)_2$ (2 mL) at 80 °C until **1** disappeared, and then K_2CO_3 (0.6 mmol) was added. [b]Times listed were for the whole one-pot process. [c]The diastereomeric ratio were detected by ^{31}P NMR spectroscopy.

unambiguously established by X-ray crystallographic analysis (CCDC 1568383).

Based on the above results and previous reports, a plausible mechanism was proposed as shown in Scheme 5. The thermal

Scheme 5. Proposed Mechanism



dehydration of *o*-HBA **1a** first resulted in the generation of *o*-QM intermediate, and then an intermolecular phospha-Michael addition of nucleophilic phosphinous acid **2a′** tautomerized from **2a** produced the alkynylphosphine oxide intermediate **3aa′** (CCDC 1568384). Following the isomerization of **3aa′**, *in situ* formed allenylphosphine oxide intermediate **A** quickly underwent an intramolecular 1,4-addition/isomerization to afford the final product 3-phosphinoylbenzofuran **3aa**.

In summary, we have developed a novel metal-free one-pot protocol featuring phospha-Michael addition/cyclization of H-phosphine oxides with *in situ* generated *o*-QMs, leading to an effective method for the construction of C(sp^2)–P bonds in various structurally interesting 3-phosphinoylbenzofurans. Importantly, asymmetric synthesis of 3-phosphinoylbenzofurans containing chiral P-stereogenic centers has been explored by using optically pure R_P-(−)-menthylphenylphosphine oxide. Further investigations on the development of phosphorus-directed methodologies initiated by such type of metal-free phospha-Michael addition are currently underway in our group.

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■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b03863.

Experimental procedure and spectra data (PDF)

Accession Codes

CCDC 1568383−1568384 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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