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## Efficient synthesis of $\alpha$ -substituted ethylphosphonates via CuH-catalyzed conjugate reduction of terminal alkenylphosphonate

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### ABSTRACT

An unprecedented approach toward synthesis of  $\alpha$ -substituted ethylphosphonates based on CuH-catalyzed conjugate reduction of vinylphosphonates has been successfully developed. This protocol features mild conditions, broad substrate scope, good functional group compatibility, high overall efficiencies, and easy gram-scale synthesis. The Cu-catalyzed reduction takes place in a highly selective manner on the phosphono substituted C=C bond in the case of the reaction of alkenylphosphonates bearing both phosphono and alkyl or aryl substituted alkene moieties. Furthermore, the result of competitive reaction indicates that the Cu-catalyzed conjugate reduction of vinylphosphonate is more challenging and reproducible than the corresponding acrylate's reaction.

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### Introduction

Amongst the various phosphorus compounds,  $\alpha$ -substituted ethylphosphonates continue to receive great attention because they could be considered as phosphorus-containing analogues of  $\alpha$ -substituted propionic acids, well-known non-steroidal anti-inflammatory drugs such as naproxen and ibuprofen.<sup>1</sup> Therefore,  $\alpha$ -substituted ethylphosphonates may find applications in the search for new potent antipsychotic drugs. Moreover,  $\alpha$ -arylalkylphosphonates have been demonstrated to exhibit negative inotropic and calcium antagonistic activity.<sup>2</sup> They were also widely employed as haptens for reactive immunization.<sup>3</sup> As a consequence, significant progress has been achieved for the preparation of  $\alpha$ -substituted ethylphosphonates.

Compared to the Arbuzov reaction of triethyl phosphite with  $\alpha$ -arylethyl bromides<sup>4</sup> and other noncatalytic protocols,<sup>5</sup> the catalytic approaches toward the synthesis of  $\alpha$ -substituted ethylphosphonates are undoubtedly more powerful and appealing. In the last

several years, many efforts have been devoted to developing efficient metal-catalyzed methods. As for the Ni-catalyzed Hiyama coupling,<sup>6</sup> only one example was reported on the coupling of  $\alpha$ -brominated ethylphosphonate with *p*-tolyl silane. Furthermore, in the case of Cu-catalyzed reductive coupling of *N*-tosylhydrazones and *H*-phosphonates,<sup>7</sup> moderate to high yields were observed under somewhat harsh conditions. Additionally, the method based on the Pd-catalyzed hydrophosphorylation of olefins is attractive because of its high atom economy and efficiency. However, the expected Markovnikov products were always contaminated by the anti-Markovnikov adducts.<sup>8</sup> In comparison with the above-mentioned methods, hydrogenation of  $\alpha$ -substituted vinylphosphonates represents the versatile and straightforward approach. These methods fall into the following three categories based on the resource of hydrogen: (i) hydrogen pressure reduction<sup>9</sup>; (ii) hydrogen transfer reduction<sup>9c,10</sup>; (iii) diimide reduction.<sup>11</sup>

Given the dangerous handling of gaseous hydrogen and the employment of a large excess amount of hydrazine in noncatalytic diimide reduction, reduction of alkenes based on catalytic hydrogen transfer offers advantages such as operational simplicity, low requirement of equipment, and economical fashion. However, in the case of the hydrogen transfer reduction using a HCOONH<sub>4</sub>/Pd/C system, hydrodehalogenation as a side reaction often occurs over most metal catalysts employed.<sup>9c</sup> We hypothesized that the

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above mentioned drawbacks could be addressed via CuH-catalyzed conjugate reduction with silanes as the hydrogen source. Since the pioneering works of Stryker and co-workers,<sup>12</sup> Stryker's reagent, [(Ph<sub>3</sub>P)CuH]<sub>6</sub> effect highly regioselective conjugate reductions of various carbonyl derivatives, including unsaturated ketones, esters, aldehydes, and related substrates.<sup>13</sup> Surprisingly, there is no precedent for the copper-catalyzed reduction of the terminal alkenylphosphonates. Herein, as a part of our program aiming at developing practical methods for the synthesis of  $\alpha$ -substituted phosphonates,<sup>10a,11,14</sup> we describe a straightforward and efficient approach toward the synthesis of  $\alpha$ -substituted ethylphosphonates based on CuH-catalyzed reduction under mild conditions.

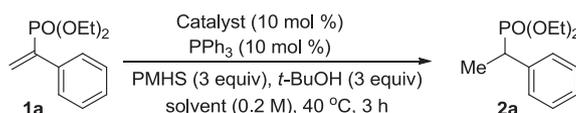
## Results and discussion

At the outset, we selected  $\alpha$ -phenyl ethenylphosphonate **1a** as the model substrate to optimize the reaction conditions and the results are listed in Table 1. Subjecting **1a** to a solution of CuH catalyst (generated from Cu(OAc)<sub>2</sub>·H<sub>2</sub>O, PPh<sub>3</sub>, and a stoichiometric amount of poly(methylhydrosiloxane) (PMHS) as the hydride source)<sup>15</sup> resulted in the formation of the desired product **2a** in 22% yield. Gratifyingly, the presence of 1 equiv *t*-BuONa as the additive accelerated the conversion greatly,<sup>16</sup> affording the **2a** in 81% yield (entry 2). The yield could be further improved to 95% with 3 equiv of *t*-BuOH as the additive<sup>13c</sup> (entry 3). Reducing the amount of the catalyst to 5 mol% resulted in a lower yield of 87% (entry 4). Changing the solvent to THF, dioxane, or CH<sub>3</sub>CN dramatically decreased the efficiency (entries 5–7). Furthermore, the significance of the copper source in this reaction was demonstrated by control experiments. Similar yield was obtained using anhydrous Cu(OAc)<sub>2</sub> in place of Cu(OAc)<sub>2</sub>·H<sub>2</sub>O as the

catalyst (entry 8). Significant drop of the yield was observed with CuCl or Cu(acac)<sub>2</sub> as the copper source (entries 9–10). Other common copper complexes like CuI, CuCN, CuCl<sub>2</sub>·2H<sub>2</sub>O, CuBr<sub>2</sub>, and CuSO<sub>4</sub> did not catalyze the reaction at all (entries 11–15). Expectedly, control experiments indicated that both Cu(OAc)<sub>2</sub>·H<sub>2</sub>O and PPh<sub>3</sub> were required for this reductive protocol (entries 16–17). On the basis of the screening, the combination of 10 mol% Cu(OAc)<sub>2</sub>·H<sub>2</sub>O, 10 mol% PPh<sub>3</sub>, 3 equiv of *t*-BuOH and 3 equiv of PMHS in toluene at 40 °C for 3 h emerged as the best reaction conditions.

With the optimized reaction conditions in hand, we then set out to examine the scope of the reduction with respect to the  $\alpha$ -aryl ethenylphosphonates. As shown in Table 2, the copper-catalyzed conjugate reduction of various terminal vinylphosphonates proceeded smoothly and furnished  $\alpha$ -aryl ethylphosphonates in good to excellent yields.  $\alpha$ -Aryl ethylphosphonates possessing electron-donating groups (Me, OMe) (**2b–f**) at the phenyl ring were efficiently accessible in 82–95% yields. The reactions of vinylphosphonates having electron-withdrawing groups (CO<sub>2</sub>Me, NO<sub>2</sub>, CN) at the phenyl ring proceeded efficiently to provide the products **2g–i** in 87–91% yields. Notably, the highly chemoselective reduction of acetyl-containing alkenylphosphonate could be realized,<sup>16a</sup> giving the product **2j** in a 87% isolated yield. The reaction of alkenylphosphonate containing biphenyl group afforded **2k** in 94% yield. However, the 2-naphthyl substituted vinylphosphonate was reluctant to be reduced under the standard conditions. Fortunately, the conjugate reduction could undergo smoothly when PPh<sub>3</sub> was replaced with 1,2-bis(diphenylphosphino)benzene (BDP),<sup>13c</sup> delivering **2l** in 96% yield. Furthermore, the hydrogenation of alkene also took place when  $\alpha$ -thienyl vinylphosphonate was used as substrate to give the corresponding product **2m** in 96% yield. Likewise, the dimethyl

**Table 1**  
Optimization of reaction conditions.<sup>a</sup>



entry	catalyst	ligand	additive	solvent	yield of <b>2a</b> <sup>b</sup> (%)
1 <sup>c</sup>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	PPh <sub>3</sub>	–	toluene	22
2 <sup>d</sup>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	PPh <sub>3</sub>	<i>t</i> -BuONa	toluene	81
3	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	95
4 <sup>e</sup>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	87
5 <sup>c</sup>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	PPh <sub>3</sub>	<i>t</i> -BuOH	THF	23
6 <sup>c</sup>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	PPh <sub>3</sub>	<i>t</i> -BuOH	dioxane	trace
7 <sup>c</sup>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	PPh <sub>3</sub>	<i>t</i> -BuOH	CH <sub>3</sub> CN	trace
8	Cu(OAc) <sub>2</sub>	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	92
9 <sup>c</sup>	Cu(acac) <sub>2</sub>	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	33
10 <sup>c</sup>	CuCl	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	20
11 <sup>c</sup>	CuI	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	trace
12 <sup>c</sup>	CuCN	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	trace
13 <sup>c</sup>	CuCl <sub>2</sub> ·2H <sub>2</sub> O	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	trace
14 <sup>c</sup>	CuBr <sub>2</sub>	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	trace
15 <sup>c</sup>	CuSO <sub>4</sub>	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	trace
16 <sup>c</sup>	–	PPh <sub>3</sub>	<i>t</i> -BuOH	toluene	0
17 <sup>c</sup>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	–	<i>t</i> -BuOH	toluene	0

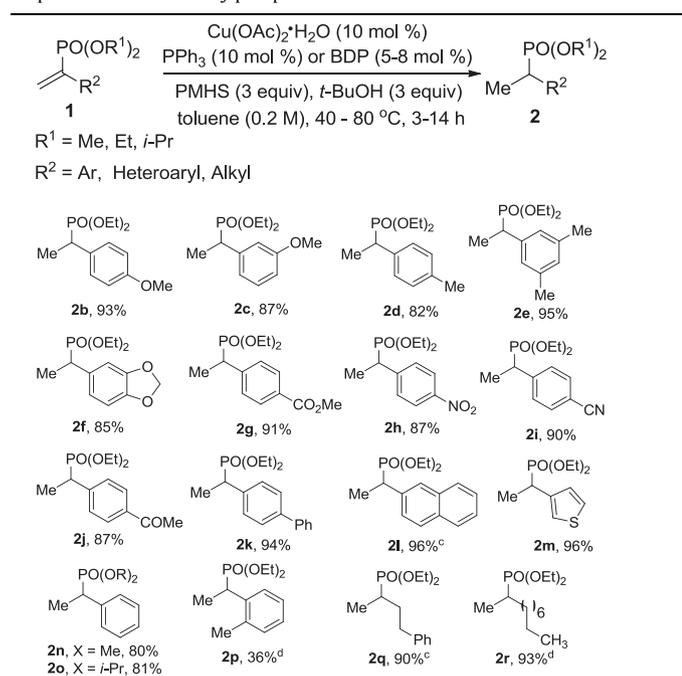
<sup>a</sup> Reaction conditions: a reaction mixture of **1a** (0.4 mmol), copper catalyst (0.04 mmol), PPh<sub>3</sub> (10.5 mg, 0.04 mmol), PMHS (266  $\mu$ L, 1.2 mmol), *t*-BuOH (127  $\mu$ L, 1.2 mmol), and solvent (2 mL) was stirred at 40 °C for 3 h.

<sup>b</sup> Yield of the isolated product.

<sup>c</sup> The yield was determined using <sup>1</sup>H NMR spectroscopy with 4-nitroacetophenone as an internal standard.

<sup>d</sup> With 1 equiv of *t*-BuONa as the additive in place of *t*-BuOH.

<sup>e</sup> A combination of Cu(OAc)<sub>2</sub>·H<sub>2</sub>O (5 mol%)/PPh<sub>3</sub>(5 mol%) was used.

**Table 2**  
Scope of  $\alpha$ -substituted vinylphosphonates.<sup>a,b</sup>

<sup>a</sup> Reaction conditions: a reaction mixture of **1** (0.4 mmol),  $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$  (0.04 mmol),  $\text{PPh}_3$  (10.5 mg, 0.04 mmol), PMHS (266  $\mu\text{L}$ , 1.2 mmol), *t*-BuOH (127  $\mu\text{L}$ , 1.2 mmol), and toluene (2 mL) was stirred at 40 °C for 3 h.

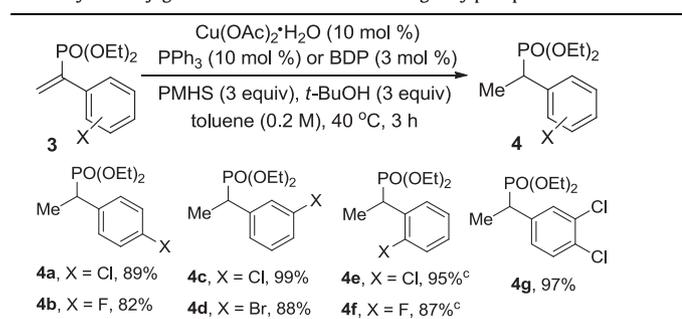
<sup>b</sup> Yield of the isolated product.

<sup>c</sup> The reaction was conducted in the presence of  $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$  (10 mol%) and BDP (5 mol%) at 60 °C for 12 h.

<sup>d</sup> Reaction run at 80 °C for 14 h in the presence of  $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$  (10 mol%) and BDP (8 mol%).

ethylphosphonates **2n** and diisopropyl ethylphosphonate **2o** could be accessed efficiently under the standard reaction conditions. As a limitation of this method, this Cu-catalyzed reduction is sensitive to steric hindrance. In the case of the reduction of  $\alpha$ -2-tolyl ethenylphosphonate, only moderate yield of **2p** could be achieved. Of note, with BDP as the ligand, this protocol could be extended to the reduction of  $\alpha$ -alkyl substituted vinylphosphonates, albeit under somewhat harsh reaction conditions, producing **2q** and **2r** in 90% and 93% yield, respectively.

The halo group is a kind of fundamental and important functional groups, which offers the opportunity for further elaboration

**Table 3**  
Cu-catalyzed conjugate reduction of halo-containing vinylphosphonates.<sup>a,b</sup>

<sup>a</sup> Reaction conditions: a reaction mixture of **3** (0.4 mmol),  $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$  (0.04 mmol),  $\text{PPh}_3$  (10.5 mg, 0.04 mmol), PMHS (266  $\mu\text{L}$ , 1.2 mmol), *t*-BuOH (127  $\mu\text{L}$ , 1.2 mmol), and toluene (2 mL) was stirred at 40 °C for 3 h.

<sup>b</sup> Yield of the isolated product.

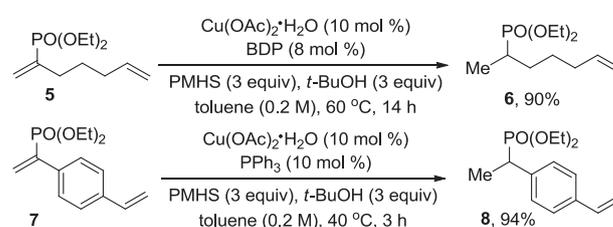
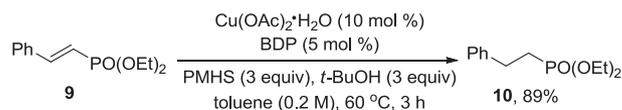
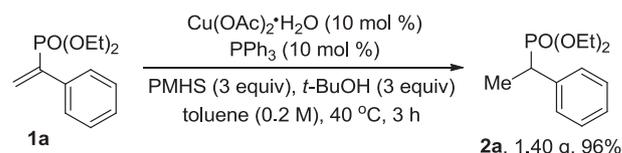
<sup>c</sup> BDP (3 mol%) was used.

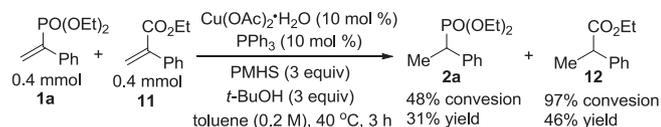
of these versatile functional groups by transition-metal-catalyzed cross-coupling reactions. However, in the case of reduction of alkenes under hydrogen transfer conditions in the presence of carbon-supported metal catalysts, hydrodehalogenation is a competing reaction. Considering the mild reaction conditions, we speculate that our protocol could be extended to the reduction of halo-containing vinylphosphonates (Table 3). To our delight, aryl chlorides, aryl bromide, and aryl fluorides turned out to be compatible. In contrast to the moderate yield of **2s**, high yields of 2-chloro- and 2-fluorophenyl substituted ethylphosphonates (**4e** and **4f**) were observed with BDP as the ligand.

One limitation of NBSH mediated reduction of alkenes arises from the difficulties associated with differentiation between phosphono and aliphatic olefins.<sup>11</sup> To determine whether this method displays acceptable levels of olefin selectivity, conjugate reduction of **5**, which contain both phosphono and alkyl substituted alkene moieties, was investigated (Scheme 1). The exclusive formation of the respective ethylphosphonate **6** indicates that CuH-catalyzed conjugate reduction takes place in a highly selective manner on the phosphono substituted C=C bond. A similar result occurs when **7** bearing two *exo* C=C bonds was subjected to the standard reaction conditions. In this case, the phosphono substituted olefin was reduced exclusively while the other aryl substituted C=C bond remained intact.

Interestingly, in addition to the 1-aryl ethenylphosphonate, the above CuH-catalyzed conjugate reduction could be extended to 2-(aryl)vinylphosphonate. As demonstrated in Scheme 2, with BDP (5 mol%) as the ligand at 60 °C, styrene phosphonate **9** was nicely reduced to give the diethyl 2-phenylethylphosphonate **10** in 89% yield. This result further expanded the substrate scope of the reaction.

Having learned the scope of the conjugate reduction, we sought to explore the effect of increasing the scale of this reductive reaction (Scheme 3). Subjecting 1.44 g of **1a** yielded 1.40 g (96% yield) of the desired reduced product **2a** without decreasing the efficiency compared with the scale used in the optimization studies. This gram-scale reaction highlights the practicality of this new Cu-catalyzed conjugate reduction of vinylphosphonates.

**Scheme 1.** Chemoselective reduction of C=C double bonds.**Scheme 2.** Reduction of 2-(phenyl)vinylphosphonate.**Scheme 3.** Gram-scale synthesis.



Scheme 4. Competitive conjugate reduction.

Finally, in competition study (Scheme 4),  $\alpha$ -phenyl ethyl acrylate **11** reacted preferentially over  $\alpha$ -phenyl ethenylphosphonate **1a**, giving **12** and **2a** in 46% and 31% yield, respectively. This result suggests that vinylphosphonate is more challenging substrate compared to the corresponding acrylate in this Cu-catalyzed conjugate reduction reaction. Meanwhile, vinylphosphonates are much less liable to polymerize in comparison with the corresponding conjugated carbonyl compounds. Consequently, their reductive reactions would be much reproducible.

In summary, we have developed a general and robust protocol for the synthesis of  $\alpha$ -substituted ethylphosphonates based on CuH-catalyzed conjugate reduction of vinylphosphonates under mild reaction conditions. This protocol features broad substrate scope, good functional group compatibility, high overall efficiencies, and easy gram-scale synthesis. Furthermore, the result of competitive reaction indicates that the Cu-catalyzed conjugate reduction of vinylphosphonate is more challenging and reproducible than the corresponding acrylate's reaction.

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## A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <https://doi.org/10.1016/j.tetlet.2017.10.045>.

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