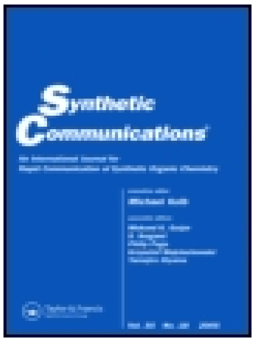

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Visible light-mediated photocatalytic phosphorylation of vinyl azides: A mild synthesis of β-ketophosphine oxides

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Visible light-mediated photocatalytic phosphorylation of vinyl azides: A mild synthesis of β-ketophosphine oxides

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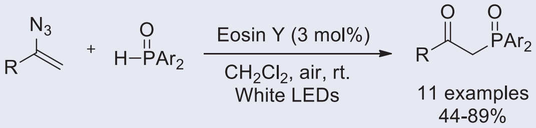
ABSTRACT

The photoredox-catalyzed phosphorylation of vinyl azides is described in this paper. The reaction proceeded smoothly using an inexpensive and easily-accessible Eosin Y as a photocatalyst under mild reaction conditions without any other oxidant, metal, or additive. This synthetic method affords a convenient way to prepare β-ketophosphine oxides.

GRAPHICAL ABSTRACT



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β-ketophosphine oxides; photoredox reaction; radical process; vinyl azides

Introduction

Organophosphorus compounds are attracting considerable attention in medicinal chemistry and material science due to their chemical and biological properties.[1] They can also be used as synthetic intermediates in organic chemistry.[2] Among the various organophosphorus compounds available, β-ketophosphine oxides are highly valuable organic compounds that serve as versatile building blocks in organic synthesis[3] and potential ligands in various complexes owing to their coordination property.[4] Therefore, the development of practical and novel methods for their synthesis has been a subject of intensive research. Generally, β-ketophosphine oxides are synthesized by the Arbuzov-type reactions of α-halogenated ketones[5] or the α-acylation of alkylphosphine oxides.[6] A number of methods have been reported based on the transition metal-catalyzed phosphorylation of alkenes[7] and their derivatives such as cinnamic acids,[8] allyl alcohols,[9] cinnamyl/alkynyl carboxylates,[10] carbonyl compounds,[11] and alkynes,[12] Recently, Yu et al. reported on the Mn-catalyzed phosphonylation of vinyl azide with phosphine oxides to afford β-ketophosphine oxides (Scheme 1a).[13] Despite the above achievements, more practical, novel, and environmentally benign protocols for the synthesis of β-ketophosphine oxides are still highly desired.

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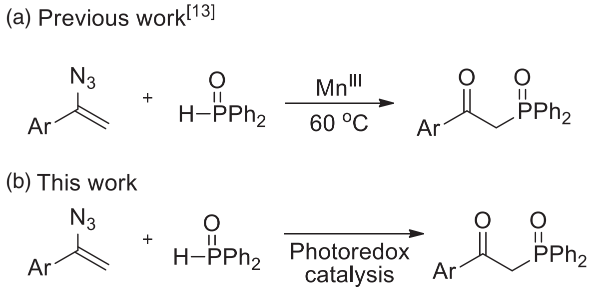


Scheme 1. Strategy for photoredox-catalyzed phosphorylation of vinyl azides.

The visible light-mediated photocatalysis has become an efficient protocol for organic chemists to introduce the functional group to organic molecules due to its environmental friendliness and potential application in industry.[14] To the best of our knowledge, photocatalytic phosphorylation of vinyl azides with phosphine oxides has not yet been reported. Thus, we envisioned the transformation of vinyl azides into β-ketophosphine oxides by the visible light-mediated photocatalysis with diphenylphosphine oxide as the phosphorus radical precursor (Scheme 1b).

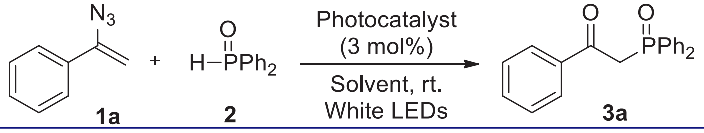
Results and discussion

In connection with our ongoing research program on investigating the C–H bond functionalization via redox reactions,[15] we recently represented the visible light-mediated photoredox functionalization of alkenes and aromatics.[16] Herein, we report visible light-mediated photocatalytic phosphorylation of vinyl azides with phosphine oxides.

To determine the optimized reaction conditions for the photocatalytic phosphorylation of vinyl azides, we examined the reaction of (1-azidovinyl)benzene (**1a**) and diphenylphosphine oxide (**2**) in dichloromethane in the presence of 3 mol% of photocatalyst with 10 W white LED. We conducted this reaction with various metal photocatalysts and organic dyes (Table 1, entries 1–8). By screening photocatalysts, Eosin Y was found to be the optimal photocatalyst for this phosphorylation. (82% yield, Table 1, entry 4). Various commonly used solvents such as dichloromethane, chloroform, THF, ethyl acetate, 1,4-dioxane, acetonitrile, DMF, DMSO, methanol, and water were examined (Table 1, entries 4 and 9–18). The results showed that the reaction gave the highest yield in dichloromethane (entry 4). Reducing the loading of photocatalyst (Eosin Y) leads to 2 mol% slightly reduced the yield of product **3a** (Table 1, entry 19). Furthermore, when the visible light sources were switched to green or blue LEDs, a lower yield was obtained (Table 1, entries 20–21). The controlled experiments demonstrated the critical roles of the photocatalyst and visible light in this phosphorylation (Table 1, entries 22–23).

With the optimal reaction conditions determined, we turned our attention to study the substrate scope of vinyl azides **1** with diphenylphosphine oxide (**2**) (Table 2). The reactions of vinyl azides **1a**–**1g** showed that a variety of functional groups were well tolerated, including methyl, fluoro, chloro, and bromo substituents. The corresponding β-ketophosphine oxides **3a**–**3g** were obtained with 58–89% yields (Table 2).

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



Entry	Photocatalyst	Solvent	Time (h)	Yield (%)[b]
1	$Ru(bpy)_3(PF_6)_2$	CH_2Cl_2	23	46
2	*fac*-$Ir(ppy)_3$	CH_2Cl_2	23	48
3	Na_2Eosin Y	CH_2Cl_2	25	73
4	Eosin Y	CH_2Cl_2	20	82
5	Eosin B	CH_2Cl_2	22	61
6	Rose Bengal	CH_2Cl_2	22	58
7	Rhodamine B	CH_2Cl_2	22	68
8	Rhodamine 6G	CH_2Cl_2	22	61
9	Eosin Y	$CHCl_3$	24	24
10	Eosin Y	DCE	24	28
11	Eosin Y	THF	24	29
12	Eosin Y	EtOAc	24	50
13	Eosin Y	1,4-dioxane	24	38
14	Eosin Y	MeCN	24	17
15	Eosin Y	DMF	24	15
16	Eosin Y	DMSO	24	45
17	Eosin Y	MeOH	24	26
18	Eosin Y	H_2O	24	24
19[c]	Eosin Y	H_2O	24	63
19[d]	Eosin Y	CH_2Cl_2	24	45
20[e]	Eosin Y	CH_2Cl_2	24	62
21[f]	Eosin Y	CH_2Cl_2	24	Trace
22[g]	–	CH_2Cl_2	24	Trace
23[h]	Eosin Y	CH_2Cl_2	24	Trace

[a] Reaction conditions: (1-azidovinyl)benzene (**1a**, 0.1 mmol), diphenylphosphine oxide **2** (0.3 mmol), photocatalyst (0.003 mmol), solvent (1 mL), white LEDs, room temperature under air.
[b] Isolated yield.
[c] 2 mol% Eosin Y loading.
[d] Green LEDs instead of white LEDs.
[e] Blue LEDs instead of white LEDs.
[f] The reaction was performed in the dark.
[g] The reaction was performed without Eosin Y.
[h] The reaction was performed in the presence of TEMPO (5 equiv).

The naphthyl- and heteroaryl-substituted vinyl azides **1h** and **1i** provided the products with moderate yields (70–71%, Table 2, for **3h**–**3i**).

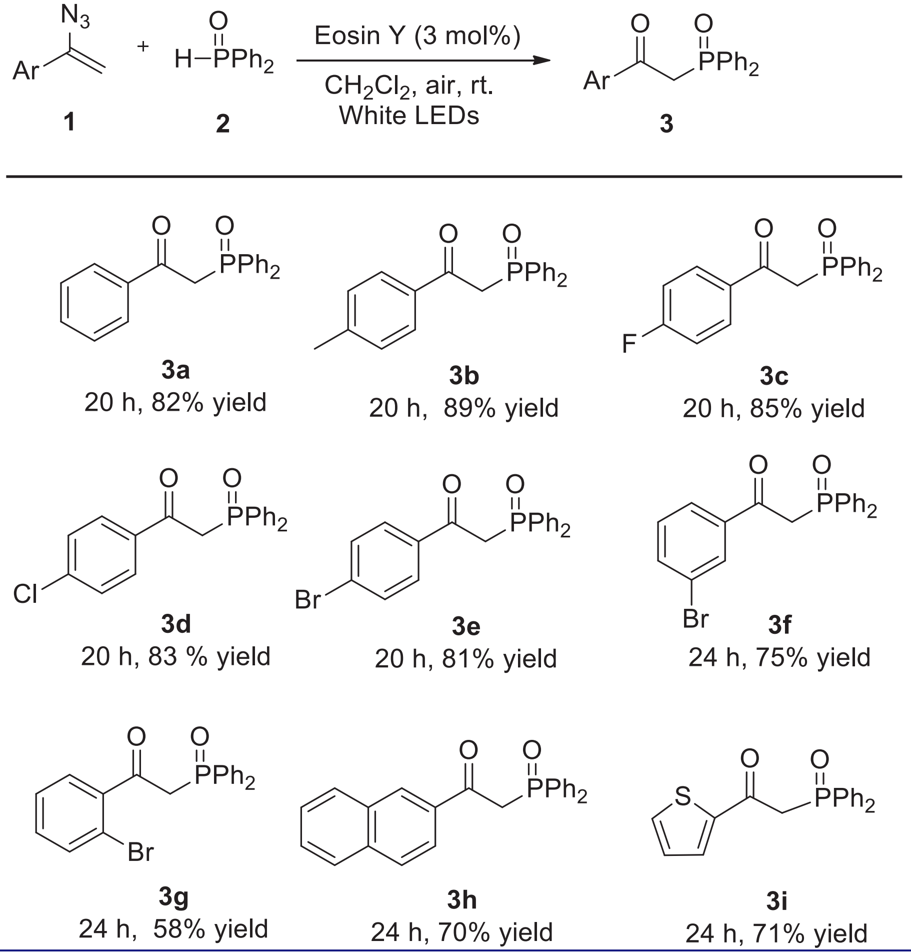
Then 2-azidohex-1-ene was used as the substrate, corresponding *β*-ketophosphine oxide **5** was obtained in 44% yield (Scheme 2).

The reaction of (1-azidovinyl)benzene **1a** with di-*p*-tolylphosphine oxide **6** was also compatible under the optimal reaction conditions to afford the corresponding *β*-ketophosphine oxide **7** in 71% yield (Scheme 3).

To demonstrate the practicality of this photocatalytic phosphorylation of vinyl azides, the gram-scale reaction was conducted. As shown in Scheme 4, the reaction of (1-azidovinyl)benzene (**1a**) with diphenylphosphine oxide (**2**) under the optimized reaction conditions afforded the corresponding *β*-ketophosphine oxide **3a** with 81% yield.

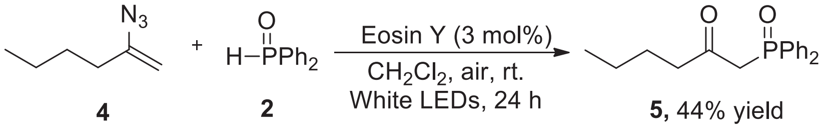
To further elucidate the reaction mechanism, some controlled experiments were performed. The results showed that the absence of either the visible light or photocatalyst completely shut down the reactivity (Table 1, entries 22–23). These reaction factors play an important role in this phosphorylation reaction. The addition of 5 equiv of 2,2,6,6-tetramethylpiperidine *N*-oxide (TEMPO) per vinyl azide **1a** under the standard

Table 2. Substrate scope.[a,b].

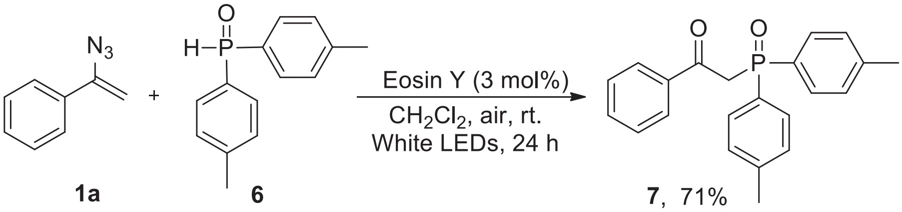


[a] Reaction conditions: vinyl azide **1** (0.1 mmol), diphenylphosphine oxide (**2**, 0.3 mmol), Eosin Y (0.003 mmol), CH_2Cl_2 (1 mL), white LEDs, room temperature under air.

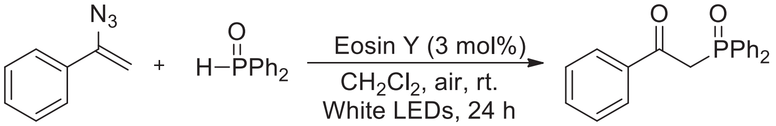
[b] Isolated yield.



Scheme 2. Photocatalytic phosphorylation of alkyl-substituted vinyl azide **4**.



Scheme 3. Photocatalytic phosphorylation with di-*p*-tolylphosphine oxide **6**.



Scheme 4. Gram-scale synthesis of **3a**.

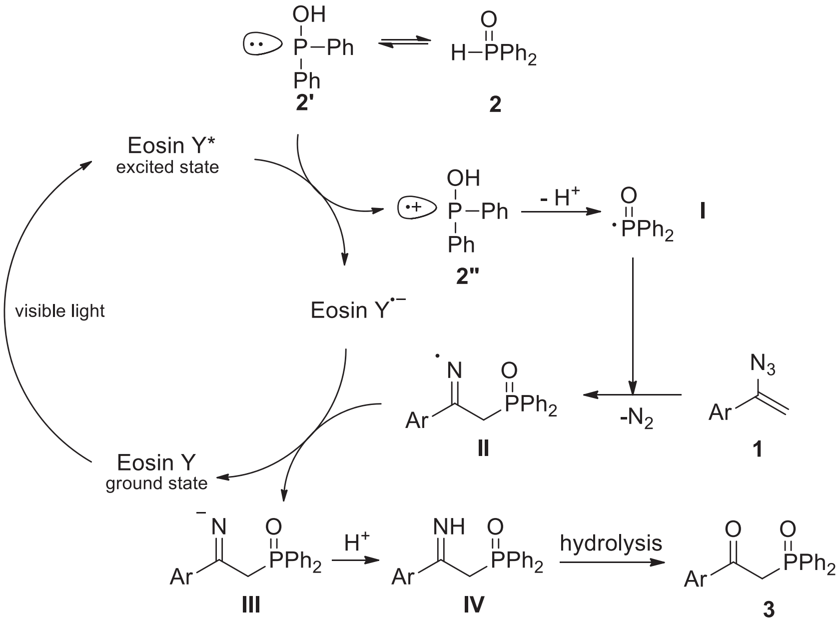


Figure 1. Plausible reaction pathway.

reaction conditions resulted in a trace amount of the desired *β*-ketophosphine oxide **3a** (Table 1, entry 24). This result is likely supporting a radical pathway to be operative in the present reaction. Based on our preliminary results as well as related literature,[11] we propose the plausible reaction pathway shown in Figure 1.

Irradiation of the photocatalyst Eosin Y with visible light leads to the excited-state Eosin Y*. Initial oxidation of phosphinous acid **2′** by Eosin Y*, via single-electron transfer process, should generate radical cation **2″**. Upon deprotonation, phosphinoyl radical **I** is formed and undergoes an addition with vinyl azides **1** to furnish iminyl radical intermediates **II**. Radicals **II** is reduced by Eosin Y·to afford the anions **III**. Protonation and hydrolysis lead to yield the *β*-ketophosphine oxides **3**.

Conclusion

In conclusion, we have developed a practical synthetic method for preparing *β*-ketophosphine oxide derivatives from the reaction between vinyl azide derivatives and diphenylphosphine oxides, accelerated by the inexpensive and easily-accessible Eosin Y as a photocatalyst under air. This protocol is practical and environmentally friendly by using shelf-stable diphenylphosphine oxide as phosphorus radical source and visible light as a source of energy. This reaction represents a convenient and mild method of preparation of β-ketophosphine oxides.

Experimental

General information

All commercial reagents and solvents were used without purification. TLC analyses were carried out on pre-coated silica gel plates with F_{254} indicator. Visualization was accomplished by UV light (254 nm). Purification of reaction products was carried out by flash chromatography using E. Merck silica gel 60 (230–400 mesh). ^{1}H NMR,^{13}C NMR, and ^{19}F NMR spectra were recorded at 400 MHz, 100 MHz, 376 MHz, respectively, on a Jeol ECS 400 MHz NMR spectrometer. Chemical shift values (δ) are reported in ppm relative to Me_4Si as the internal references and $PhCF_3$ as the external references. Mass spectra (MS-EI, 70 eV) were conducted on GC-MS Shimadzu QP2010. High-resolution mass spectra were measured on Jeol HX110/110A using electrospray ionization technique.

General procedure for the synthesis of β-ketophosphine oxide derivatives

An oven-dried flask was equipped with a magnetic stir bar, (1-azidovinyl)arene **1** (0.1 mmol) diphenylphosphine oxide **2** (0.3 mmol), Eosin Y (1.9 mg, 0.003 mmol), and methylene chloride (1 mL) under air atmosphere. The reaction mixture was allowed to stir for 20–24 h under irradiation using 10 W white LEDs. After the reaction was finished, the mixture was concentrated in vacuum and purified by chromatography on silica gel (ethyl acetate: *n*-hexane = 2:1) to afford the β-ketophosphine oxides **3**.

2-(Diphenylphosphoryl)-1-phenylethanone (3a): Yield: 82%; White solid; m.p. 136–138 °C; ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.99 (d, $J = 7.2$ Hz, 2H), 7.83–7.78 (m, 4H), 7.54–7.42 (m, 9H), 4.15 (d, $J = 15.2$ Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 192.8 (d, $J = 5.7$ Hz), 137.0, 133.6, 132.2 (d, $J = 2.8$ Hz), 131.9 (d, $J = 103$ Hz), 131.1 (d, $J = 9.5$ Hz), 129.3, 128.6 (d, $J = 12.4$ Hz), 128.5, 43.4 (d, $J = 57.2$ Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): δ 27.5; HRMS (ESI) calcd for $C_{20}H_{18}O_2P$ $[M+H]^+$ 321.1039; found 321.1041.

Supplementary data (copies of ^{1}H, ^{13}C, and ^{19}F NMR spectra of all the synthesized compounds) associated with this article can be found through the "Supplementary Content".

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