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Communication

# Metal-free photo-induced radical C-P and C-S bond formation for the synthesis of 2-phosphoryl benzothiazoles

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Graphical abstract

In this work, an efficient method for the synthesis of phosphoryl nitrogen-containing heterocycles was developed from isocyanides and *H*-phosphorus oxides *via* radical cascade cyclization reaction.

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ABSTRACT

We reveal here a visible-light promoted phosphorylation of 2-isocyanoaryl thioethers for the first time with concomitant C(sp$^3$)-S bond cleavage and imidoyl C-S formation. Additionally, this method features the use of 3 mol% organic dye Rose Bengal as the photocatalyst without external transition-metal or peroxide oxidants, and provides a novel and environmentally friendly approach for the preparation of a variety of 2-phosphoryl benzothiazoles in moderate to good yields.

*Keywords:*
Dearomatization
Spiro compounds
Isocyanides
Radical cascade reaction
*H*-Phosphorus oxides

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Organophosphorus compounds have wide applications in organic synthesis, biochemistry and medicinal chemistry [1], where phosphorus substituents play as ligands, directing group for transition metal catalysis or $\pi$-conjugated materials [2]. As a consequence, the introduction of organophosphorus groups in convenient means continues to stimulate methods for their synthesis [3]. Among which, the addition of P-centered radicals to unsaturated bonds has become one of the most used reactions for the construction of phosphorus-containing compounds, but an extra equivalent of promoter (such as Ag(I), Mn(III), or peroxides) or catalytic quantity of metal catalyst and high reaction temperature were usually utilized, resulting in a waste of energy and limitations of the utilization [4]. For instance, Yang and coworkers reported a silver-catalyzed radical carbon phosphorylation of alkenes with *H*-phosphorus oxides for construction of

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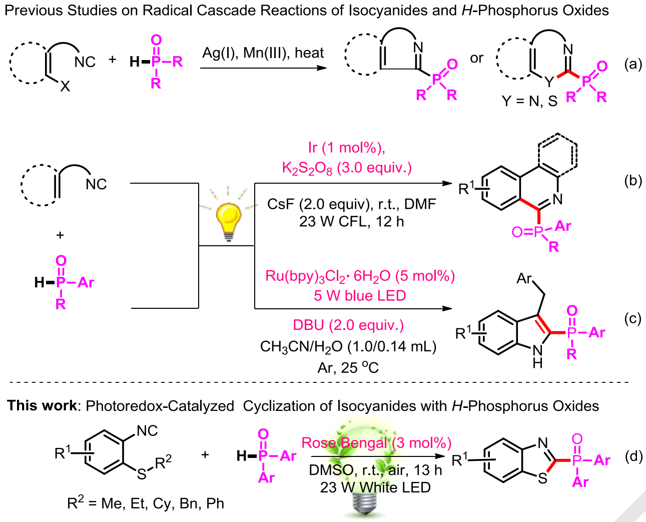
phosphorylated oxindoles [4f]. For these reasons, more and more attentions have been paid to search for green and efficient approaches for introduction of organophosphorus backbones during the past decade. Meanwhile, the application of visible-light photoredox catalysis has regained substantial attention during the last decade due to its operational simplicity to carry out synthetic transformations in an environ-mentally benign and sustainability [5]. This photocatalytic transformation meets mild reaction conditions and green chemistry, providing an alternative for assembling phosphorus-containing molecules [6].

Isocyanides belong to a valuable class of organic intermediates and their versatility in the synthesis of heterocycles (phenanthridine, quinolone, quinoxaline, indole, benzimidazole, and benzothiazole) has been adequately demonstrated in radical involved cascade reactions, including P-radical [7]. A literature investigations found that Ag(I) or Mn(III) salts are necessary in the early radical cascade reactions between isocyanides with phosphine oxides (Scheme 1a) [8]. For example, in 2017, a Mn(III) salt promoted denitrogenative imidoyl radical cyclization with diphenylphosphine oxides to access 2-phosphoryl-substituted benzoimidazoles derivatives was established by the Zhu's group [8b]. Additionally, visible-light induced C-P bonds formations have greatly responded to the demands of environmental friendliness since the light source is inexhaustible [9]. Although two research groups independently reported the radical cascade insertion/cyclization from phosphine oxides and isocyanides *via* visible-light irradiation, the use of noble-metal catalysts or extra oxidants limited their application in pharmaceutical chemistry and advanced functional materials (Schemes 1b and 1c) [10]. As a result, the development of an oxidant (perxides) and metal-free strategy for radical cascade reaction of isocyanides with phosphine oxides is still a challenging but highly attractive task.



**Scheme 1**. Radical cascade reactions of isocyanides with *H*-phosphorus oxides.

Benzothiazole, a fundamental building block in synthetic intermediates, often have surprisingly complex biological and pharmacological properties [11]. Among which, 2-substituted benzothiazoles are especially important for their existence in many naturally-occurring molecules and drugs. This ever demanding need in medicinal chemistry stems from the desire to establish mild and efficient methods for the synthesis of 2-substituted benzothiazoles [12]. And thioanisole derivatives offer an opportunity to build benzothiazoles because the methylthio motif could play as the radical acceptor in radical cascade reactions [13]. Recently, our group disclosed a novel way to synthesize 2-phosphorylated benzothiazoles *via* radical cyclization of 2-isocyanoaryl thioethers [8g]. However, the cascade reaction was achieved depending on the use of equivalent of metal promoters and high temperature. With our continuing research interest in C-X bonds formation and radical chemistry [14], we herein report for the first time an eco-friendly visible-light induced Rose Bengal catalyzed cyclization of isocyanides with *H*-phosphorus oxides *via* C–P and C–S bond formation (Scheme 1d). The transformation was performed at room temperature under oxidant- and additive-free conditions to generate 2-phosphorylated benzothiazole derivatives with wide functional group tolerance, good yields and high efficiency.

To identify the optimal reaction conditions, we commenced our investigation with the synthesis of **3a** by the treatment of (2-isocyanophenyl)(methyl)sulfane (**1a**) and diphenylphosphine oxide (**2a**) at room temperature under an air atmosphere (Table 1). After irradiation with 23 W white LED light for 13 h, an initial attempt found that the cascade reaction gave the target product **3a** in 32% yield when using Rose Bengal as photocatalyst and DMF as solvent. Motivated by this result, a screening of the solvents was further performed to enhance reactivity (entries 2-6). The results revealed that DMSO is the best solvent for this transformation. Afterward, various photocatalysts including Eosin Y, Eosin Y disodium, Eosin B, Rhodamine B, and Fluorecein were examined under the same conditions, affording **3a** in lower yields than 86% (entries 7-11). In addition, replacing organic photocatalysts with metal-photocatalyst shows less activity (entry 12). Interestingly enough, the two substrates successfully transform to **3a** in 59% yield when utilizing Eosin Y/TBHP as catalytic system [6h]. Besides, switching conditions to dark at 90 °C shut down the reaction (entry 14, condition e). Lower yields were obtained when shortening the reaction time to 7 h or changing light source to sunlight (34% and 16% corresponding). Finally, two control experiments were conducted to prove that photocatalyst and visible light are essential for the concomitant $C(sp^3)$-S bond cleavage and imidoyl C-S formation in the absence of Rose Bengal or visible light.

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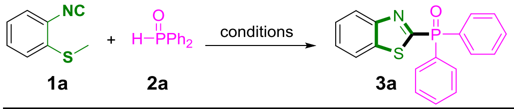
**Table 1**

Optimization of the reaction conditions.[a]



| Entry | Catalyst (3 mol%) | Solvent | Yield (%)[b] |
|---|---|---|---|
| 1 | Rose Bengal | DMF | 32 |
| 2 | Rose Bengal | Toluene | 24 |
| 3 | Rose Bengal | MeCN | trace |
| 4 | Rose Bengal | THF | 17 |
| 5 | Rose Bengal | DCE | 36 |
| 6 | Rose Bengal | DMSO | 86 |
| 7 | Eosin Y | DMSO | 45 |
| 8 | Eosin Y disodium | DMSO | trace |
| 9 | Eosin B | DMSO | 70 |
| 10 | Rhodamine B | DMSO | 38 |
| 11 | Fluorecein | DMSO | 72 |
| 12 | $Ru(bpy)_3Cl_2$ | DMSO | 41 |
| 13[c] | Eosin Y, TBHP | DMSO | 59 |
| 14[d,e] | Rose Bengal | DMSO | N.R. |
| 15 | -- | DMSO | N.R. |

[a] Reaction conditions: (2-isocyanophenyl)(methyl)sulfane (**1a**, 0.15 mmol), diphenylphosphine oxides (**2a**, 0.3 mmol), 1.5 mL of solvent, photocatalyst, 23 W white LED for 13 h under air.

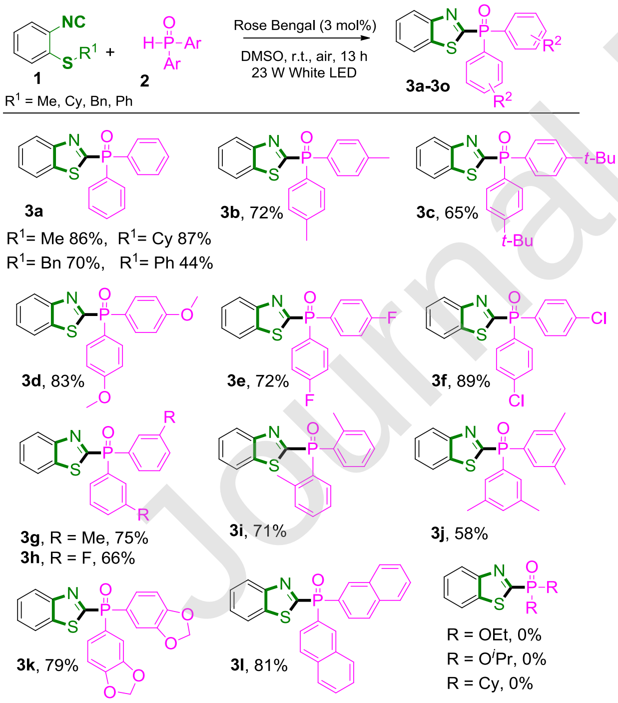
[b] Isolated yields, N.R. = no reaction.

[c] Eosin Y (3 mol%) TBHP (2.0 equiv.).

[d] Dark.

[e] Dark at 90 °C.

Encouraged by the preliminary results, we next investigated the generality of this protocol with various H-phosphorus oxides and isocyanides. As shown in Scheme 2, no matter whether the ring of diarylphosphine oxides is substituted with either electron-deficient, electron-rich or steric-hindered groups, all of them were well-tolerated to the system delivering the expected products (**3a**-**3i**) in medium to good yields. Importantly, this present protocol could be applied to piperonyl- and naphthyl-derivated phosphine oxides as well, affording the target products (**3k**-**3l**) in 79% and 81% yields. Additionally, dimethyl-substituted *H*-phosphorus oxide was also utilized in this reaction to give the corresponding **3j** in moderate yield. Unfortunately, phosphonate esters of ethyl and isopropyl and dicyclohexylphosphine oxide were proven to be not suitable substrates.



**Scheme 2.** Substrates scope on *H*-phosphorus oxides. Reaction conditions: 2-isocyanothioanisoles (**1**, 0.15 mmol), *H*-phosphorus oxides (**2**, 0.3 mmol), Rose Bengal (3 mol%), 1.5 mL of DMSO, 23 W white LED for 13 h under air.

Next, the feasibility of utilizing substituted isocyanides in the current system were investigated with the optimized conditions, and Scheme 3 illustrates the scope of iscyanides compatible. The results revealed that most of the various functional groups, covering electron-rich groups (−OMe and −Me) and electron-poor groups (−F, −Cl, −Br, and $–SO_2Me$) substituents on the benzene rings in 2-

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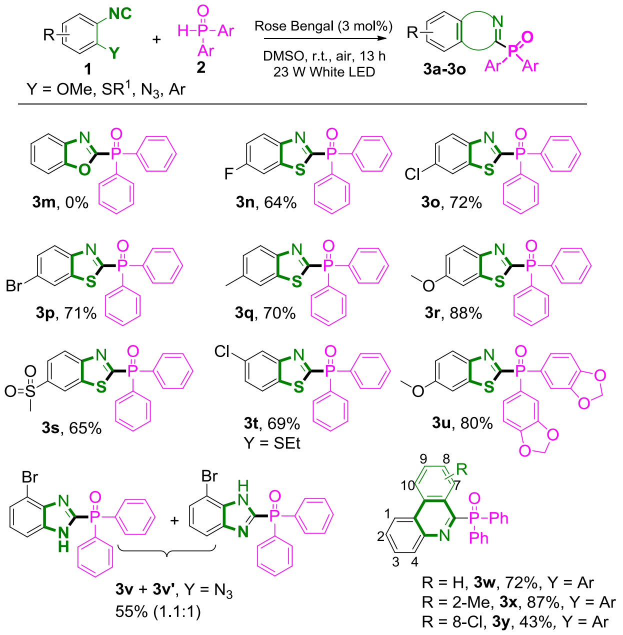
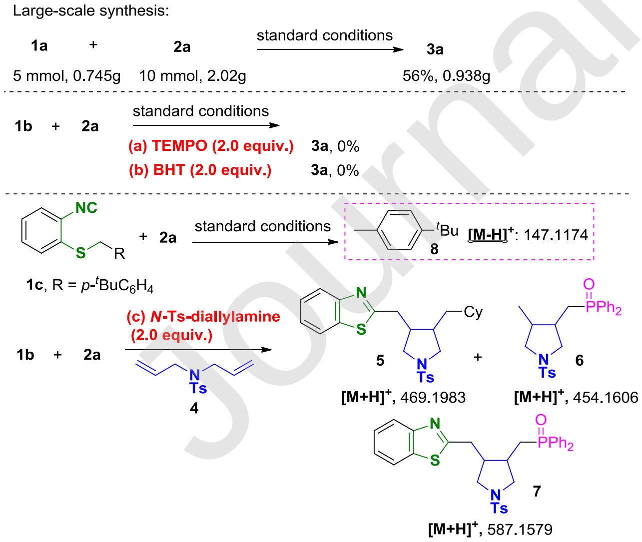
isocyanoaryl thioethers, were well tolerated in the present reactions and provided the desired products in good yields. Analogues of OMe were also tested, however, no **3m** was obtained. Notably, other functionalized isocyanides were synthesized to expand the application of this protocol for constructing 2-substituted benzimidazole and phenanthridine derivatives in the fields from 55% to 87%. For example, 1-azido-2-isocyanoarene was also compatible with the standard condition, furnishing **3v** as a mixture of isomers *via* a denitrogenative imidoyl radical cyclization.

To verify the practical application of the visible-light promoted radical cyclization, the reaction of **1a** and **2a** was conducted on the 5 mmol scale (Scheme 4). The cascade reaction performed well and 0.93 g of the final product was obtained in 56% yield after isolation.



**Scheme 3.** Substrates scope on isocyanides. Reaction conditions: Isocyanides (**1**, 0.15 mmol), *H*-phosphorus oxides (**2**, 0.3 mmol), Rose Bengal (3 mol%), 1.5 mL of DMSO, 23 W white LED for 13 h under air, isolated yields, Y = SMe (unless stated).

In order to gain further insight into the product-forming profile, several control experiments were carried out as shown in Scheme 4. Firstly, the standard reactions were performed in the presence of TEMPO or BHT, and no product **3a** was detected, implying that radical species might exist in this transformation (Scheme 4a,4b). A well-known radical capturer **4**, used in our early and Song's papers [8g,13a], was added to the standard conditions, the radical cyclization products **5-7**, were detected by HR-MS, indicating that phosphonyl and cyclohexyl radical intermediates were involved in the process ((Scheme 4c, and details see Supporting information). When **1a** was replaced by **1c**, 1-(*tert*-butyl)-4-methylbenzene was detected by HR-MS.



**Scheme 4**. Large-scale synthesis and radical inhibiting experiments.

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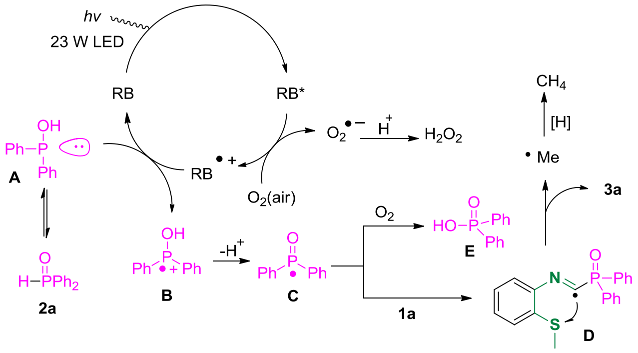

**Scheme 5.** Proposed mechanism.

Based on the mechanistic study as well as previous reports [13,15], the possible pathway for the visible-light initiated cyclization of isocyanides was proposed as shown in Scheme 5. Mechanistically, diarylphosphine oxide **2a** exists in equilibrium with its trivalent tautomer phosphinous acid **A** [6c,6i,15]. Firstly, a radical cation intermediate **B** is generated from corresponding electron-rich phosphinous acid **A** *via* a reductive quenching of the RB radical cation ($RB^{\bullet+}$) [6c], which comes from the reaction of excited state ($RB^*$) and oxygen from air. Then, the deprotonation occurred with the affording of phosphinoyl radical **C** [6i], which subsequently adds to the isocyanide functionality in **1a** to form intermediate radical **D** [7a,8g]. Meanwhile, the phosphinoyl radical could transfer to byproduct $Ph_2P(O)OH$ with an oxidation by $O_2$. Finnaly, The product **3a** was obtained by the addition of intermediate **D** to a sulfur atom in **1a** along with the release of a methyl group.

In conclusion, we have developed a versatile synthetic entry point to C2-phosphoryl benzothiazoles from easily prepared isocyanides and *H*-phosphorus oxides. This convenient and green method is initiated by 23 W White LED *via* Rose Bengal catalyzed cascade C-P bond formation and C-S bond cleavage under metal- and oxidant-free at room temperature. By simple variation of the isocyanides a wide range of nitrogen heterocycles including phenanthridines and benzimidazoles are obtained, leading to high product diversity and synthetic efficiency. Future studies will examine the application of this visible light mediated electron-transfer event in alternative spiro molecules forming transformations.

## Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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Journal Pre-proof