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光催化下 β-萘酚与二芳基膦氧化物区域选择性邻位膦酰化研究

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摘要 以荧光素为有机光催化剂, $K_2S_2O_8$ 为氧化剂, 在室温氮气保护下研究了 β-萘酚与二芳基氧化膦的区域选择性邻位 C—H 膦酰化反应. 该反应条件温和, 官能团适用范围广, 产率高, 区域选择性好, 是 β-萘酚改性的理想而实用的选择. 实验结果揭示反应通过自由基途径进行.

关键词 β-萘酚; 二芳基膦氧化物; 光催化; 膦酰化; 自由基反应

Visible-Light-Induced Regioselective *ortho*-C—H Phosphonylation of β-Naphthols with Diarylphosphine Oxides

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Abstract A metal-free visible-light-induced regioselective *ortho*-C—H phosphonylation of β-naphthols with diarylphosphine oxides has been developed using fluorescein as an organophotoredox catalyst in the presence of $K_2S_2O_8$ at room temperature under N_2 atmosphere. This feature along with mild reaction conditions, sensitive functional group tolerance, good to excellent yields, high regioselectivity, and scale-up synthesis makes it an ideal and practical alternative modification of β-naphthols. The experimental result suggests that a radical pathway is involved in the reaction.

Keywords β-naphthols; diarylphosphine oxide; visible-light catalysis; phosphonylation; radical reaction

1 Introduction

β-Naphthols are readily available raw materials and possess multiple reactive sites, which makes them versatile precursors for the synthesis of biologically active natural products and pharmaceuticals.[1] β-Naphthol derivatives are also valuable pharmacologic compounds possessing various important biological properties such as anti-inflammatory, antibacterial, hypotensive, bradycardiac, and anticancer activities.[2] Recently, the C(1) position of β-naphthol may be functionalized via a number of reaction processes, which include the C—H bond alkylation,[3] acylation,[4] arylation,[5] sulfonylation,[6] amination,[7] chalcogenation,[8] *etc*. Because the substituents of β-naphthols have a great influence on their properties, the direct C—H functionalization of β-naphthol has been paid attention for researchers.

In the past decades, phosphine-containing aromatic compounds are found to be widely applicable in organic synthesis, materials science, and inorganic chemistry, as the presence of phosphorus controls the physical, chemical, and

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biological properties of the adjacent arenes.[9] Some phenolic compounds containing phosphoryl functionalities have wide applications in chiral ligands in asymmetric synthesis, initiators or catalysts for polymerization processes, and optoelectronic materials (Figure 1).[10] Moreover, a number of biologically active compounds containing the carbon-phosphorus bond acquire a huge capacity to influence physiological and pathological processes with broad applications in the medicinal field.[11] Although *β*-naphthols containing phosphoryl functionalities have wide functions, convenient and practical synthetic tools are still lacking. This has stimulated a great interest in developing new reactions for C—P bond formation of *β*-naphthols.

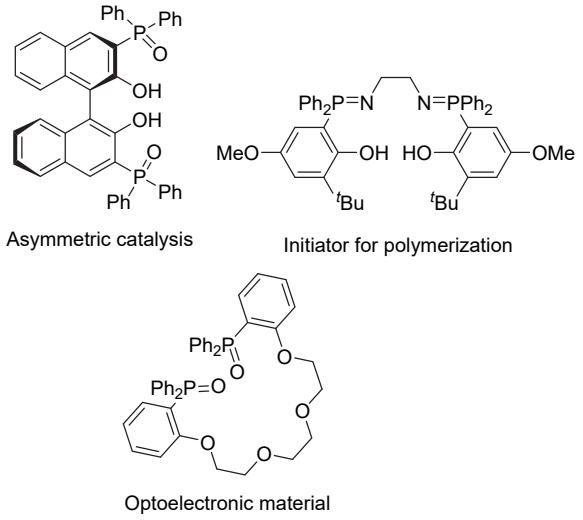
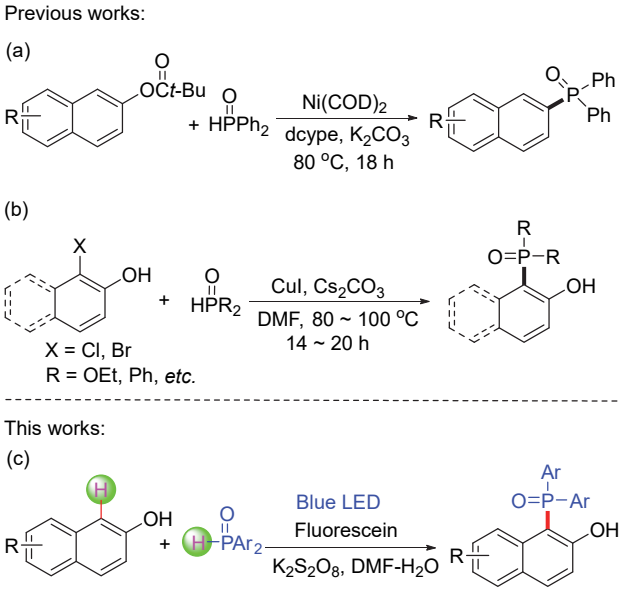


Figure 1 Functional *ortho*-phosphoryl phenol derivatives

Given the unique properties of phosphorus-containing aromatic molecules, it is not surprising that many synthetic methods have been developed for the formation of arene-phosphorus bonds.[12] Classically, the nucleophilic substitution of arylmetals with phosphine chlorides had been used for the synthesis of aromatic phosphorus compounds.[13] Along with the development of efficient synthetic methods, transition-metal-catalyzed strategies of C—P bond formation involving the coupling of phosphonate esters or phosphine oxides with electrophiles has been recognized as one of the most efficient and promising approaches.[14] In 2015, Han and his co-workers disclosed an efficient inert C—O/P—H cross-coupling to produce phosphorus-containing naphthol derivatives using $Ni(COD)_2$ catalyst and 1,2-bis(dicyclohexylphosphino)ethane (dcype) as a ligand at 80 ℃ for 18 h (Scheme 1a).[15] A protocol that allows for one-pot construction of C—P bonds via the cross-coupling of *α*-naphthols and phosphine oxide or phosphite in the presence of a nickel catalyst at 100 ℃ for 18 h was also presented by Han's groups,[16] and diethyl(1-hydroxy-2-naphthyl)phosphonate was obtained using diethyl 1-naphthyl phosphate as a substrate on treatment with excess lithium diisopropylamide (LDA) at −8 ℃ undergoing clean rearrangement.[17] Furthermore, Han's groups reported a copper-catalyzed cross-coupling reaction of *H*-phosphinates with 2-naphthol halides in the presence of Cs_2CO_3 at 80～100 ℃ for 14～20 h (Scheme 1b).[18] However, these transformations are associated with one or more limitations such as toxic or stoichiometric transition metals, air-sensitive ligands, additives, elevated temperatures, and longer reaction time, and so on. Therefore, we became interested in developing an efficient, environmentally benign, and regioselective method for the direct construction of the C(sp^2)—P bond using free *β*-naphthols as substrates under mild conditions.



Scheme 1 Synthesis of phosphonylated *β*-naphthol derivatives

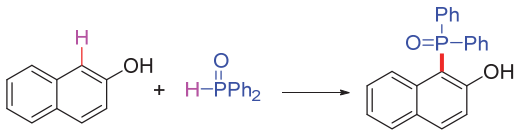
Visible light as renewable and clean energy has been widely used in synthetic chemistry and a variety of organic transformations can be achieved under very mild conditions. In recent years, great efforts have been devoted to the development of visible-light-mediated C—C and C—X formations by direct C—H functionalization.[19] Recently, photoredox-based P-radical reactions have been developed to synthesize a range of organophosphorus aromatic compounds.[20] For representative examples, Wu and co- workers[21] demonstrated a visible-light photocatalyzed C—P formation reaction of diphenylphosphine oxide with thiazole derivatives using eosin B as a catalyst. Remarkably, Hajra and co-workers have also described a strategy to produce phosphonylated 2*H*-indazole derivatives from diphenylphosphine oxide with 2*H*-indazoles employing rose bengal as a catalyst in the presence of 1,5-diazabicyclo[4.3.0]non-5-ene (DBU) under blue LED (light emitting diode).[22] Inspired by these leading researches, we envisaged that P-centered radical might be able to couple with *β*-naphthols under visible-light-mediated photoredox catalysis. As part of our continuing interest in synthesis and application of organophosphorus heterocyclic moieties.[23] Herein, we develop a direct and environmentally friendly approach for the C—H phosphonylation of free *β*-naphthols using fluorescein as an organophotoredox catalyst irradiated

with blue LED and employing O_2 as the sole oxidant at room temperature (Scheme 1c). To the best of our knowledge, this is the first example of direct and regioselective C—H phosphonylation of free *β*-naphthol derivatives by organic dye-sensitized photocatalysis without metal, base, and additive.

2 Results and discussion

Initially, the reaction of *β*-naphthol **1a** with diphenylphosphine oxide **2a** was chosen to screen the reaction conditions employing $K_2S_2O_8$ as an oxidant in DMF-H_2O co-solvent irradiated with 3 W blue LED at room temperature. (2-Hydroxynaphthalen-1-yl)diphenylphosphine oxide (**3a**) was only obtained in 11% yield after 16 h in the absence of photocatalyst (Table 1, Entry 1). Then, the effect of photocatalysts such as rhodamine B, eosin Y, eosin B, fluorescein, and tris(2,2'-bipyridyl)ruthenium(II) chloride hexahydrate (ruthenium) was checked (Table 1, Entries 2～6). To our delight, investigations revealed that the yield of **3a** could be improved to 65% when fluorescein was employed as the photocatalyst. As a comparison, when the classical metal ruthenium photocatalyst was employed for this transformation, a trace amount product was afforded, which evidenced the superiority of organic dyes over the transition-metal-derived counterpart in this type of C—H functionalization. Further screening of the loading of fluorescein revealed that 0.05 equiv. fluorescein was the best option, affording the product **3a** in 80% yield (Table 1, Entries 7～9). More photocatalysts make the yield of the product **3a** decrease, which is probably due to formation of

Table 1 Optimization of reaction conditions[a]



1a **2a** **3a**

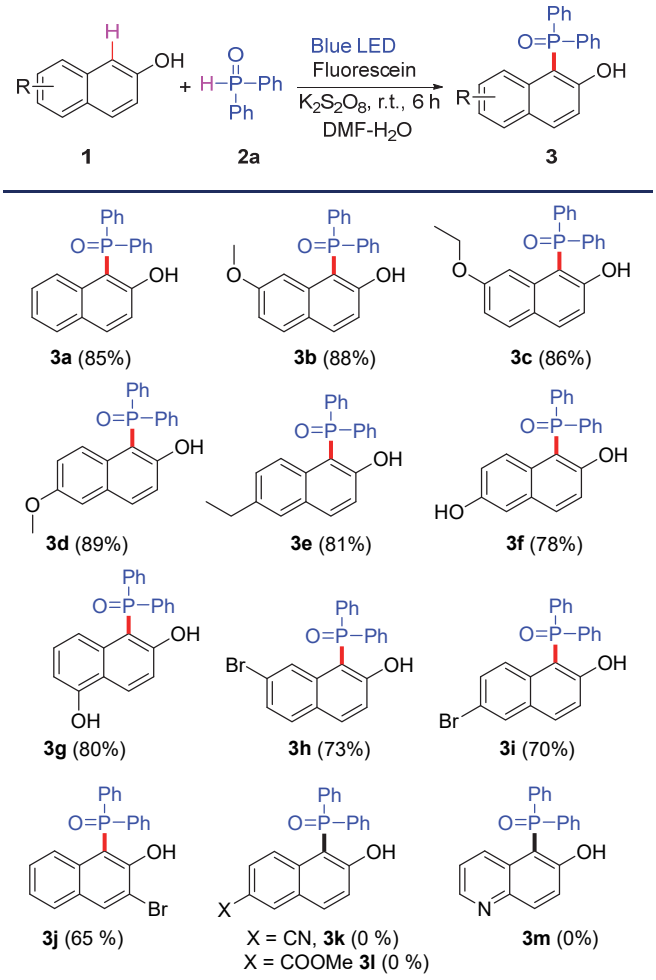
Entry	Catalyst (mol%)	Oxidant (equiv.)	Solvent	Time/h	Yield[b]/%
1	—	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	16	11
2	Rhodamine B (0.1)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	16	0
3	Eosin Y (0.1)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	16	Trace
4	Eosin B (0.1)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	16	Trace
5	Fluorescein (0.1)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	16	65
6	Ruthenium (0.1)[c]	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	16	Trace
7	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	16	80
8	Fluorescein (0.15)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	16	45
9	Fluorescein (0.2)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	16	40
10	Fluorescein (0.05)	$(NH_4)_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	16	70
11	Fluorescein (0.05)	TBPB (2.0)	DMF-H_2O (V∶V=1∶1)	16	15
12	Fluorescein (0.05)	DTBP (2.0)	DMF-H_2O (V∶V=1∶1)	16	21
13	Fluorescein (0.05)	BPO (2.0)	DMF-H_2O (V∶V=1∶1)	16	26
14	Fluorescein (0.05)	$K_2S_2O_8$ (1.0)	DMF-H_2O (V∶V=1∶1)	16	48
15	Fluorescein (0.05)	$K_2S_2O_8$ (1.5)	DMF-H_2O (V∶V=1∶1)	16	67
16	Fluorescein (0.05)	$K_2S_2O_8$ (2.5)	DMF-H_2O (V∶V=1∶1)	16	78
17	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	DMSO	16	12
18	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	MeCN	16	25
19	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	DCE	16	20
20	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	Dioxane	16	34
21[c]	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	DMF	16	67
22[d]	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	H_2O	16	0
23	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	2	50
24	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	4	71
25	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	6	83
26	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	8	78
27[d]	**Fluorescein (0.05)**	**$K_2S_2O_8$ (2.0)**	**DMF-H_2O (V∶V=1∶1)**	**6**	**85**
28[e]	Fluorescein (0.05)	$K_2S_2O_8$ (2.0)	DMF-H_2O (V∶V=1∶1)	6	0
29[f]	Fluorescein (0.05)	—	DMF-H_2O (V∶V=1∶1)	6	0

[a] Reaction conditions: *β*-naphthol (**1a**) (0.2 mmol, 28.8 mg), diphenylphosphine oxide (**2a**) (0.4 mmol, 80.8 mg), catalyst, oxidant, and solvent (2.0 mL), 3 W blue LED at room temperature. [b] Isolated yield. [c] Tris(2,2'-bipyridyl)ruthenium(II) chloride hexahydrate. [d] Under N_2 atmosphere. [e] In the dark. [f] In the absence of $K_2S_2O_8$.

byproducts. Then, various oxidants including $(NH_4)_2S_2O_8$, TBPB (*tert*-butyl peroxybenzoate), DTBP (di-*tert*-butyl peroxide), and BPO (benzoyl peroxide) were investigated, $K_2S_2O_8$ was found to be the best option (Table 1, Entries 7, 10～13). The loading of oxidant was also screened, and 2.0 equiv. of $K_2S_2O_8$ was the best choice (Table 1, Entries 7, 14～16). Subsequently, the effect of other solvents including dimethyl sulfoxide (DMSO), MeCN, 1,2-dichloroethane (DCE), dioxane, *N*,*N*-dimethylformamide (DMF), H_2O, and co-solvent (DMF-H_2O) was checked, and the binary solvent DMF-H_2O (1 mL/1 mL) mixture was found to be the best (Table 1, Entries 7, 17～22). Water can be considered, in many cases, the greenest solvent. The loading H_2O of binary solvent was also screened, and the best volume ratio of DMF and H_2O was 1∶1. Moreover, the molar ratio of **1a** and **2a** was also explored. When the ratio of **1a** and **2a** was 1∶1.5, the best yield (80%) could be obtained. In addition, the effect of reaction time was also tested, and 6.0 h was proved be optimal time to afford the product **3a** in 83% yield (Table 1, Entries 7, 23～26). The yield of **3a** could increase to 85% when the reaction proceeded under N_2 atmosphere (Table 1, Entry 27). Additionally, the reaction was carried out in the dark, and the phosphonylated product **3a** was not generated (Table 1, Entry 28). The product **3a** was also obtained in the absence of $K_2S_2O_8$ (Table 1, Entry 28). These results indicate that light, photocatalyst, and oxidant are all essential to achieve the reaction. Finally, the maximum yield (85%) of the desired product was afforded in the optimized condition: The molar ratio of **1a** and **2a** with 1∶1.5, 5 mol% fluorescein with 2.0 equiv. of $K_2S_2O_8$ in DMF-D_2O (V∶V= 1∶1) irradiated with 3 W blue LED for 6.0 h under N_2 atmosphere at room temperature.

With the optimized conditions in hand, we evaluated the nature of free *β*-naphthol derivatives that could participate in the photoredox cross-coupling with diphenylphosphine oxide, and the results are summarized in Table 2. *β*-Naphthols with an electron-donating and electron- withdrawing groups at naphthalene ring performed as well in good to excellent yields under the standard conditions (**3a**～**3j**). Generally, *β*-naphthols with electron-donating groups (**3b**～**3g**) gave higher yields than those with electron-withdrawing (**3h**～**3j**). Notably, *β*-naphthols bearing electron-donating groups such as Me, Et, OMe, and OEt at the phenyl ring efficiently reacted to provide excellent yields (86%～93%) of the desired products **3a**～**3e**. To our delight, when naphthalene-2,6-diol and naphthalene-1,6-diol were employed for this transformation, the desired products **3f** and **3g** could also be afforded in good yields (83% and 85%), respectively. No other phosphorylated *β*-naphthol derivatives were found, and only C(1) position of *β*-naphthol derivatives was phosphorylated, which showed that the cross-coupling reaction has high regioselective. *β*-Naphthols containing halogens such as 3-Br, 6-Br, and 7-Br successfully reacted under the present reaction conditions to give the desired products **3h**～**3j** in good yields. Interestingly, 3-bromonaphthalen-2-ol could also react with **2a** under the standard conditions affording the desired product **3j** in 70% yield. Unfortunately, *β*-naphthol derivatives having a strong withdrawing group (CN, COOMe), 6-hydroxy-2-naphthonitrile and methyl 6-hydroxy-2-naphthoate, were inapplicable even after systematic screening, which might be attributed to the low reactivity. Regrettably, this transformation could also not proceed when quinolin-6-ol was employed under the standard conditions.

Table 2 Scope of *β*-naphthols for the phosphorylation[a,b]



[a] Reaction conditions: *β*-naphthol **1** (0.2 mmol), diphenylphosphine oxide **2a** (0.3 mmol, 60.6 mg), fluorescein (0.01 mmol, 3.32 mg), and $K_2S_2O_8$ (0.4 mmol, 108 mg) in DMF (1.0 mL) and H_2O (1.0 mL), 3 W blue LED at room temperature for 6 h under N_2 atmosphere. [b] Isolated yield.

Next, the compatibility of the reaction of protocol with various diarylphosphine oxides was examined. As shown in Table 3. Electronically diverse diarylphosphine oxides were investigated as coupling partners. To our delight, diarylphosphine oxides with electron-donating and electron-withdrawing groups could provide the desired products in good yields (**3n**～**3t**). A preference for electron-rich diarylphosphine oxides was observed for coupling with *β*-naphthols (**3n** and **3o**). Regrettably, when diarylphosphine oxide with electron-withdrawing group at the *para* position, such as trifluoromethyl, was employed, the reaction could not proceed. Furthermore, the steric effect

Table 3 Substrate scopes with a variation of diarylphosphine oxides[a,b]

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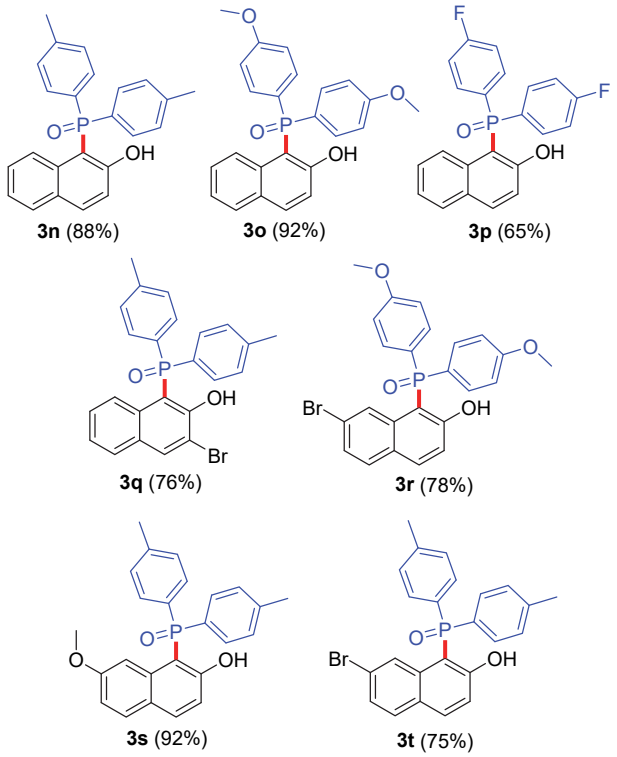

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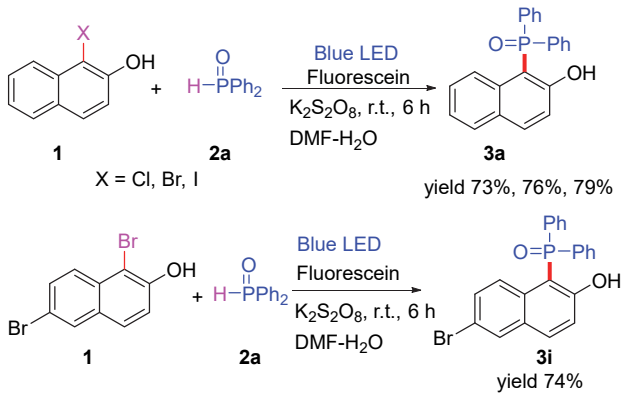
could affect the reaction, and on using di(naphthalen-1-yl)phosphine oxide the corresponding product was not delivered. In addition, dialkylphosphine oxides and dialkyl H-phosphonates exhibited no reactivity toward the standard conditions.

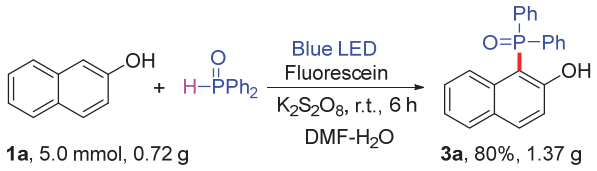
From the Tables 2 and 3, we could realize that the photoredox cross-coupling reaction is a type of highly regioselective reaction. *α*-Position of *β*-naphthol derivatives could be regioselectively phosphorylated with diarylphosphine oxides under the standard conditions. Further, in order to investigate the regioselectivity of this transformation, some experiments were designed in Scheme 2. If *α*-substituted *β*-naphthols were used as substrates, could this reaction proceed under standard conditions? Then, 1-halogeno naphthalen-2-ols including 1-chloronaphthalen-2-ol, 1-bromonaphthalen-2-ol, and 1-iodonaphthalen-2-ol, were employed for this transformation. Interestingly, the experimental results showed that the same desired product **3a** was afforded under standard conditions in good yields (73%～79%). Subsequently, the reaction of 1,6-dibromo-



Scheme 2 Scope of 1-halogenonaphthalen-2-ol for the phosphorylation

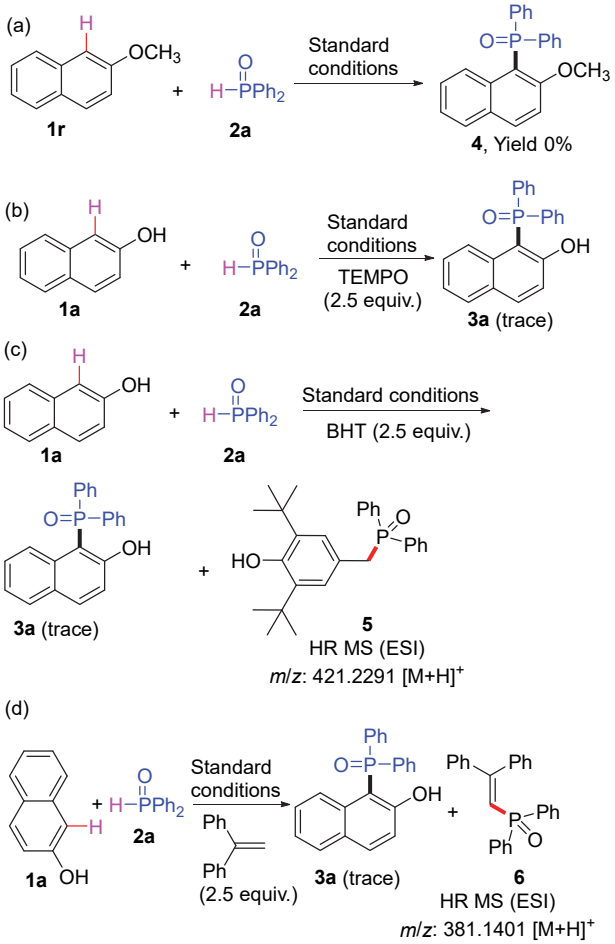
naphthalen-2-ol with diphenylphosphine oxide (**2a**) was also investigated. To our delight, the corresponding product (6-bromo-2-hydroxynaphthalen-1-yl)-diphen- ylphosphine oxide was obtained under standard conditions. These facts indicate that this reaction features high regioselectivity at the C(1) position under mild and simple conditions.

To further evaluate the utility of this approach, a gram-scale reaction was conducted using the photocatalyzed C—P cross-coupling procedure with **1a** (5.0 mmol, 0.72 g) and **2a**. As expected, the reaction proceeded well to produce the desired product **3a** with reasonable yield (80%) (Scheme 3).



Scheme 3 Gram-scale reaction of *β*-naphthol **1a** with diphenylphospine oxide **2a**

More experiments were subsequently designed and conducted for understanding the related mechanisms. Initially, 2-methoxynaphthalene (**1r**) was employed to react with diphenylphosphine oxide (**2a**) under standard conditions, and this transformation could not proceed, which indicated that the hydroxyl group of *β*-naphthol maybe be involved in the photocatalyzed C—P cross-coupling procedure (Scheme 4a). Then, it was found that the reaction of *β*-naphthol **1a** with diphenylphosphine oxide (**2a**) failed to give the corresponding phosphonylation product **3a** in the presence radical scavenger TEMPO (2,2,6,6-tetramethyl-1-piperidinyl-oxy) (Scheme 4b). Then, BHT (2,6-di-*tert*-butyl-4-methyl phenol, 2.5 equiv.) was added to the reaction of **1a** with **2a** under the standard conditions, and a trace of **3a** was obtained. Notably, the adduct of BHT and **2a** was observed by HRMS (Scheme 4c). In addition, when ethene-1,1-diyldibenzene (2.5 equiv.) was added to the reaction system under the standard conditions, the adduct **6** of ethene-1,1-diyldibenzene and **2a** was found by HRMS spectrum (Scheme 4d). Furthermore, the mechanism was also studied by Stern-Volmer quenching experiment. The



Scheme 4 Some control experiments for the mechanism study

fluorescence of fluorescein was dramatically quenched by diphenylphosphine oxide **2a**. In contrast, no quenching took place with the addition of *β*-naphthol **1a** into the same solution of fluorescein. On the basis of the result, we speculated that a single-electron transfer (SET) process should

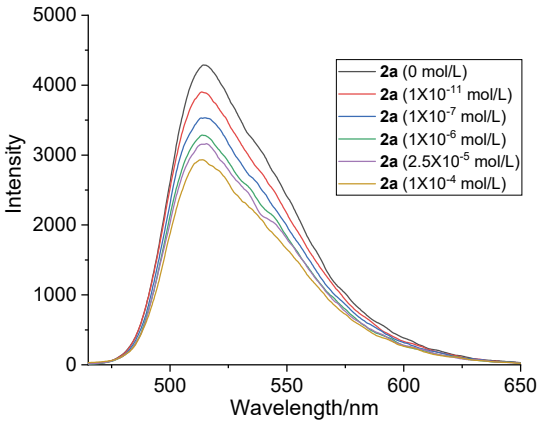
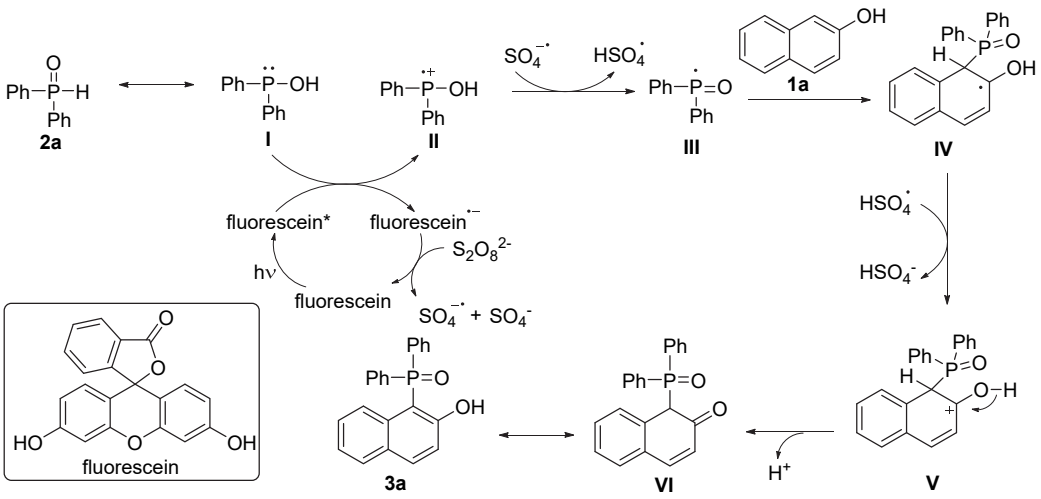


Figure 2 Luminescence quenching study

be involved between diphenylphosphine oxide **2a** and fluorescein in this reaction (Figure 2). All these observations imply that the reaction may occur through a radical mechanism.

Based on the control experiments and relevant reports,[21,22,24,25] a plausible catalytic cycle was proposed in Scheme 5. Initially, fluorescein is irradiated by blue LED light to produce the excited-state fluorescein*. Excited-state fluorescein* obtains an electron from the tautomer **I** of diphenylphosphine oxide **2a** affording the fluorescein$^{\bullet -}$ radical anion and radical-cation intermediate **II**. Then, the oxidation of fluorescein$^{\bullet -}$ by $S_2O_8^{2-}$ generates the ground state fluorescein, $SO_4^{-\bullet}$, and SO_4^{-}. Subsequently, radical-cation intermediate **II** by $SO_4^{-\bullet}$ delivers *P*-centered radical **III** and $HSO_4^{\bullet}$. Next, *β*-naphthol **1a** can capture the nucleophilic *P*-centered radical **III** and generates the radical adduct **IV**. The intermediate **IV** would furnish the corresponding carbon cation **V** via single-electron transformation (SET) process. The carbon cation **V** loses a proton to obtain the compound **VI**. Finally, the expected coupling product **3a** is provided by the tautomerization of **VI**.



Scheme 5 Proposed reaction mechanism for this phosphonylation of *β*-naphthols

3 Conclusions

In summary, we have developed a metal-free visible-light-induced photoredox-catalyzed radical phosphonylation of *β*-naphthol using fluorescein as a photocatalyst in the presence of $K_2S_2O_8$ under N_2 atmosphere. A variety of phosphonylated *β*-naphthols have been regioselectively synthesized in good to excellent yields. To the best of our knowledge, this is the first report of visible-light-promoted functionalization of *β*-naphthols. The further development of related photo-induced oxidative C-heteroatom bond forming reactions is ongoing in our laboratory.

4 Experimental section

4.1 Instruments and reagents

All commercial reagents and solvents were used without purification. Column chromatography was performed using 300～400 mesh silica with the indicated solvent system according to standard techniques. Nuclear magnetic resonance spectra were recorded on a Bruker Avance 400 MHz spectrometer. Chemical shifts for ^{1}H NMR and ^{13}C NMR spectra are recorded from tetramethylsilane. Chemical shifts for ^{19}F NMR spectra were recorded with fluorobenzene as external standard. High resolution mass spectra (HRMS) were obtained on a Thermo Scientific LTQ Orbitrap XL instrument using the ESI technique. IR spectra were recorded on a Shimadazu IR-408 Fourier transform infrared spectrophotometer using a thin film supported on KBr pellets. Melting points were measured on an XT4A microscopic apparatus uncorrected.

4.2 General procedure for the synthesis of phosphonylated *β*-naphthols (**3**)

β-Naphthol **1** (0.2 mmol), diarylphosphine oxide **2** (0.3 mmol), fluorescein (0.01 mmol, 3.32 mg), and $K_2S_2O_8$ (0.4 mmol, 108 mg) were placed a reaction vessel equipped with a magnetic stir bar. Then DMF (1.0 mL) and H_2O (1.0 mL) co-solvent was added, and the reaction mixture was irradiated using 3 W blue LED at room temperature under N_2 atmosphere for 6.0 h. After completion, the solvent was distilled under vacuum. Then, the resulting mixture was dissolved with ethyl acetate (10 mL), washed with saturated sodium chloride solution (10 mL×2). The organic phase was dried over anhydrous Na_2SO_4 and concentrated under vacuum. The crude residue was purified by column chromatography on silica gel using ethyl acetate/petroleum ether as an eluent to afford the pure products **3**.

(2-Hydroxynaphthalen-1-yl)diphenylphosphine oxide (**3a**): Light yellow crystal, m.p. 188～189 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 13.32 (s, 1H), 7.92 (d, $J_{H\text{-}H}$=9.0 Hz, 1H), 7.82～7.77 (m, 4H), 7.71 (d, $J_{H\text{-}H}$=8.0 Hz, 1H), 7.56 (td, $J_{H\text{-}H}$=7.2, 1.2 Hz, 2H), 7.48～7.44 (m, 4H), 7.25～7.18 (m, 3H), 7.08 (td, $J_{H\text{-}H}$=7.7, 1.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 167.1 (d, $J_{P\text{-}C}$=2.9 Hz), 136.1 (d, $J_{P\text{-}C}$=2.2 Hz, CH), 133.5 (d, $J_{P\text{-}C}$=8.9 Hz), 132.5 (d, $J_{P\text{-}C}$=3.0 Hz, CH), 132.0 (d, $J_{P\text{-}C}$=10.3 Hz, CH), 132.1 (d, $J_{P\text{-}C}$=104.3 Hz), 129.1 (d, $J_{P\text{-}C}$=1.5 Hz, CH), 128.8 (d, $J_{P\text{-}C}$=12.3 Hz), 128.3 (d, $J_{P\text{-}C}$=8.8 Hz, CH), 126.8 (CH), 124.9 (d, $J_{P\text{-}C}$=6.3 Hz, CH), 123.0 (CH), 121.4 (d, $J_{P\text{-}C}$=8.8 Hz, CH), 99.2 (d, $J_{P\text{-}C}$=102.2 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) *δ*: 42.8; IR (KBr) *ν*: 1567, 1463, 1436, 1414, 1370, 1239, 1119 cm^{-1}. HRMS (ESI) calcd for $C_{22}H_{18}O_2P$ $[M+H]^+$ 345.1039, found 345.1042,

(2-Hydroxy-7-methoxynaphthalen-1-yl)diphenylphosphine oxide (**3b**): Light yellow crystal, m.p. 194～195 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) *δ*: 12.71 (s, 1H), 8.00 (d, $J_{H\text{-}H}$=8.9 Hz, 1H), 7.77～7.69 (m, 5H), 7.68～7.64 (m, 2H), 7.60～7.55 (m, 4H), 7.00～6.97 (m, 2H), 6.92 (dd, $J_{H\text{-}H}$=8.8, 2.4 Hz, 1H), 3.36 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) *δ*: 165.6 (d, $J_{P\text{-}C}$=3.0 Hz), 158.3, 136.4 (d, $J_{P\text{-}C}$=2.0 Hz, CH), 135.7 (d, $J_{P\text{-}C}$=7.5 Hz), 133.0 (d, $J_{P\text{-}C}$=2.7 Hz), 132.7 (d, $J_{P\text{-}C}$=104.1 Hz, CH), 131.8 (d, $J_{P\text{-}C}$=10.6 Hz, CH), 131.0 (CH), 129.5 (d, $J_{P\text{-}C}$=12.0 Hz, CH), 123.6 (d, $J_{P\text{-}C}$=8.9 Hz), 117.9 (d, $J_{P\text{-}C}$=8.3 Hz, CH), 115.2 (CH), 105.3 (d, $J_{P\text{-}C}$=6.1 Hz, CH), 100.0 (d, $J_{P\text{-}C}$=101.3 Hz), 55.2 (CH_3); ^{31}P NMR (162 MHz, DMSO-d_6) *δ*: 39.3; IR (KBr) *ν*: 2922, 2850, 1615, 1514, 1463, 1434, 1362, 1221, 1117 cm^{-1}. HRMS (ESI) calcd for $C_{23}H_{20}O_3P$ $[M+H]^+$ 375.1145, found 375.1142.

(7-Ethoxy-2-hydroxynaphthalen-1-yl)diphenylphosphine oxide (**3c**): Brown crystal, m.p. 121～122 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 13.18 (s, 1H), 7.87 (d, $J_{H\text{-}H}$=9.0 Hz, 1H), 7.82～7.76 (m, 4H), 7.57～7.53 (m, 2H), 7.51～7.44 (m, 5H), 7.18～7.14 (m, 2H), 6.96 (dd, $J_{H\text{-}H}$=8.7, 1.8 Hz, 1H), 2.66 (q, $J_{H\text{-}H}$=7.6 Hz, 2H), 1.22 (t, $J_{H\text{-}H}$=7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 166.5 (d, $J_{P\text{-}C}$=2.4 Hz), 138.8, 135.7 (d, $J_{P\text{-}C}$=2.1 Hz, CH), 132.4 (d, $J_{P\text{-}C}$=2.7 Hz, CH), 132.2 (d, $J_{P\text{-}C}$=104.2 Hz), 132.0 (d, $J_{P\text{-}C}$=10.7 Hz, CH), 131.7 (d, $J_{P\text{-}C}$=8.7 Hz), 128.8 (d, $J_{P\text{-}C}$=12.4 Hz, CH), 128.5 (d, $J_{P\text{-}C}$=8.9 Hz), 127.8 (CH), 127.0 (CH), 124.8 (d, $J_{P\text{-}C}$=6.4 Hz, CH), 121.2 (d, $J_{P\text{-}C}$=8.9 Hz, CH), 98.6 (d, $J_{P\text{-}C}$=72.1 Hz), 28.2 (CH_2), 15.1 (CH_3); ^{31}P NMR (162 MHz, $CDCl_3$) *δ*: 42.7; IR (KBr) *ν*: 3338, 2973, 2871, 1468, 1437, 1360, 1234, 1047 cm^{-1}. HRMS (ESI) calcd for $C_{24}H_{22}O_3P$ $[M+H]^+$ 389.1301, found 389.1303.

(2-Hydroxy-6-methoxynaphthalen-1-yl)diphenylphosphine oxide (**3d**): Pink crystal, m.p. 178～179 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) *δ*: 12.34 (s, 1H), 7.77 (d, $J_{H\text{-}H}$=9.3 Hz, 1H), 7.73～7.67 (m, 4H), 7.65～7.61 (m, 2H), 7.57～7.53 (m, 4H), 7.32 (bs, 1H), 7.13～7.10 (m, 1H), 6.94 (dd, $J_{H\text{-}H}$=9.3, 2.8 Hz, 1H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) *δ*: 162.7 (d, $J_{P\text{-}C}$=3.0 Hz), 155.5, 135.4 (d, $J_{P\text{-}C}$=2.0 Hz, CH), 133.1 (d, $J_{P\text{-}C}$=104.2 Hz), 132.8 (d, $J_{P\text{-}C}$=2.3 Hz, CH), 131.8 (d, $J_{P\text{-}C}$=10.6 Hz, CH), 129.8 (d, $J_{P\text{-}C}$=9.0 Hz), 129.3 (d, $J_{P\text{-}C}$=12.1 Hz), 129.0 (d, $J_{P\text{-}C}$=8.2 Hz, CH), 126.4 (d, $J_{P\text{-}C}$=5.3 Hz, CH), 120.8 (d, $J_{P\text{-}C}$=8.4 Hz, CH), 119.1 (CH), 108.7 (CH), 101.8 (d, $J_{P\text{-}C}$=100.5 Hz), 55.5 (CH_3); ^{31}P NMR (162 MHz, DMSO-d_6) *δ*: 38.5; IR (KBr) *ν*: 1601, 1514, 1435, 1372, 1234, 1116 cm^{-1}. HRMS (ESI) calcd for $C_{23}H_{20}O_3P$ $[M+H]^+$ 375.1145, found 375.1148.

(6-Ethyl-2-hydroxynaphthalen-1-yl)diphenylphosphine oxide (**3e**): Light yellow crystal, m.p. 214～215 ℃; ^{1}H

NMR (400 MHz, $CDCl_3$) δ: 13.14 (s, 1H), 7.79～7.76 (m, 5H), 7.59～7.53 (m, 3H), 7.48～7.43 (m, 4H), 7.04～7.01 (m, 1H), 6.83 (dd, $J_{H\text{-}H}$=8.8, 4.1 Hz, 1H), 6.48 (d, $J_{H\text{-}H}$=2.1 Hz, 1H), 3.28 (q, $J_{H\text{-}H}$=7.0 Hz, 2H), 1.10 (t, $J_{H\text{-}H}$=7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.6 (d, $J_{P\text{-}C}$=2.7 Hz), 157.5, 135.9 (d, $J_{P\text{-}C}$=2.0 Hz, CH), 135.2 (d, $J_{P\text{-}C}$=8.4 Hz), 132.5 (d, $J_{P\text{-}C}$=2.5 Hz, CH), 132.2 (d, $J_{P\text{-}C}$=104.0 Hz), 131.7 (d, $J_{P\text{-}C}$=10.7 Hz, CH), 130.4 (CH), 128.8 (d, $J_{P\text{-}C}$=12.4 Hz, CH), 123.2 (d, $J_{P\text{-}C}$=9.0 Hz), 118.5 (d, $J_{P\text{-}C}$=8.5 Hz, CH), 115.6 (CH), 105.7 (d, $J_{P\text{-}C}$=6.8 Hz, CH), 98.5 (d, $J_{P\text{-}C}$=103.0 Hz), 63.1 (CH_2), 14.4 (CH_3); ^{31}P NMR (162 MHz, $CDCl_3$) δ: 42.4; IR (KBr) ν: 2920, 1615, 1470, 1435, 1359, 1234, 1117 cm^{-1}. HRMS (ESI) calcd for $C_{24}H_{22}O_2P$ $[M+H]^+$ 373.1352, found 373.1348.

(2,6-Dihydroxynaphthalen-1-yl)diphenylphosphine oxide (**3f**): Pink crystal, m.p. 221～223 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 12.34 (s, 1H), 9.52 (s, 1H), 7.88 (d, $J_{H\text{-}H}$=9.0 Hz, 1H), 7.73～7.68 (m, 4H), 7.66～7.53 (m, 7H), 7.10 (t, $J_{H\text{-}H}$=2.0 Hz, 1H), 7.07～7.03 (m, 1H), 6.81 (dd, $J_{H\text{-}H}$ = 9.2, 2.6 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 162.4 (d, $J_{P\text{-}C}$=3.0 Hz), 153.5, 135.0 (d, $J_{P\text{-}C}$=1.8 Hz, CH), 133.1 (d, $J_{P\text{-}C}$=104.0 Hz), 132.8 (d, $J_{P\text{-}C}$=2.4 Hz, CH), 131.8 (d, $J_{P\text{-}C}$=10.6 Hz, CH), 130.1 (d, $J_{P\text{-}C}$=9.1 Hz), 129.3 (d, $J_{P\text{-}C}$=12.2 Hz, CH), 127.8 (d, $J_{P\text{-}C}$=8.0 Hz), 126.4 (d, $J_{P\text{-}C}$=5.8 Hz, CH), 120.7 (d, $J_{P\text{-}C}$=8.3 Hz, CH), 119.3 (CH), 111.2 (CH), 101.3 (d, $J_{P\text{-}C}$=100.7 Hz, CH); ^{31}P NMR (162 MHz, DMSO-d_6) δ: 38.9; IR (KBr) ν: 1519, 1433, 1366, 1308, 1234, 1119 cm^{-1}. HRMS (ESI) calcd for $C_{22}H_{18}O_3P$ $[M+H]^+$ 361.0988, found 361.0985.

(2,5-Dihydroxynaphthalen-1-yl)diphenylphosphine oxide (**3g**): Colorless solid, m.p. 213～214 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 12.93 (s, 1H), 10.29 (s, 1H), 8.33 (d, $J_{H\text{-}H}$=9.2 Hz, 1H), 7.73～7.63 (m, 6H), 7.59～7.54 (m, 4H), 7.05 (dd, $J_{H\text{-}H}$=8.2, 4.2 Hz, 1H), 7.02～6.95 (m, 2H), 6.64 (dd, $J_{H\text{-}H}$=7.2, 1.0 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 165.7 (d, $J_{P\text{-}C}$=2.9 Hz), 154.5, 135.4 (d, $J_{P\text{-}C}$=8.4 Hz), 133.2, 132.9 (d, $J_{P\text{-}C}$=1.9 Hz, CH), 132.1 (d, $J_{P\text{-}C}$=3.1 Hz), 131.9 (d, $J_{P\text{-}C}$=10.6 Hz, CH), 130.7 (CH), 129.4 (d, $J_{P\text{-}C}$=12.3 Hz, CH), 128.5 (CH), 119.4 (d, $J_{P\text{-}C}$=9.1 Hz, CH), 119.0 (d, $J_{P\text{-}C}$=8.1 Hz, CH), 106.7 (CH); ^{31}P NMR (162 MHz, DMSO-d_6) δ: 40.5; IR (KBr) ν: 3056, 1521, 1437, 1372, 1310, 1238, 1120 cm^{-1}. HRMS (ESI) calcd for $C_{22}H_{18}O_3P$ $[M+H]^+$ 361.0988, found 361.0986.

(7-Bromo-2-hydroxynaphthalen-1-yl)diphenylphosphine oxide (**3h**): Colorless crystal, m.p. 195～196 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 12.44 (s, 1H), 8.13 (t, $J_{H\text{-}H}$=1.7 Hz, 1H), 8.08 (t, $J_{H\text{-}H}$=8.7 Hz, 2H), 7.73～7.68 (m, 4H), 7.65～7.61 (m, 2H), 7.56～7.52 (m, 4H), 7.42 (dd, $J_{H\text{-}H}$=9.1 Hz, $J_{H\text{-}H}$=2.2 Hz, 1H), 7.20～7.17 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 163.9, 135.6 (CH), 133.5 (d, $J_{P\text{-}C}$=7.6 Hz), 133.1 (d, $J_{P\text{-}C}$=104.9 Hz), 132.7 (d, $J_{P\text{-}C}$=2.3 Hz, CH), 131.8 (d, $J_{P\text{-}C}$=10.4 Hz, CH), 131.1 (CH), 130.3 (CH), 130.1 (d, $J_{P\text{-}C}$=8.9 Hz), 129.3 (d, $J_{P\text{-}C}$=12.2 Hz, CH), 127.2 (d, $J_{P\text{-}C}$=5.2 Hz, CH), 121.4 (d, $J_{P\text{-}C}$=7.9 Hz, CH), 116.6; ^{31}P NMR (162 MHz, DMSO-d_6) δ: 37.2; IR (KBr) ν: 1611, 1496, 1446, 1434, 1387, 1233, 1133 cm^{-1}. HRMS (ESI) calcd for $C_{22}H_{17}BrO_2P$ $[M+H]^+$ 423.0144, found 423.0146.

(6-Bromo-2-hydroxynaphthalen-1-yl)diphenylphosphine oxide (**3i**): Colorless crystal, m.p. 217～218 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 11.95 (s, 1H), 8.08 (d, $J_{H\text{-}H}$=9.0 Hz, 1H), 7.82 (dd, $J_{H\text{-}H}$=8.7, 1.1 Hz, 1H), 7.71～7.66 (m, 4H), 7.64～7.60 (m, 2H), 7.56～7.52 (m, 4H), 7.45 (dd, $J_{H\text{-}H}$=8.6, 1.8 Hz, 1H), 7.17～7.13 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 162.9, 136.8 (d, $J_{P\text{-}C}$=7.2 Hz), 136.3 (d, $J_{P\text{-}C}$=2.0 Hz, CH), 133.8 (d, $J_{P\text{-}C}$=105.4 Hz), 132.5 (d, $J_{P\text{-}C}$=2.4 Hz, CH), 131.6 (d, $J_{P\text{-}C}$=10.5 Hz, CH), 131.4 (CH), 129.1 (d, $J_{P\text{-}C}$=12.1 Hz, CH), 127.3 (d, $J_{P\text{-}C}$=8.8 Hz), 127.2 (d, $J_{P\text{-}C}$=4.6 Hz, CH), 126.6 (CH), 121.5, 120.2 (d, $J_{P\text{-}C}$=7.7 Hz, CH), 103.1 (d, $J_{P\text{-}C}$=100.1 Hz); ^{31}P NMR (162 MHz, DMSO-d_6) δ: 35.0; IR (KBr) ν: 3053, 1588, 1505, 1457, 1437, 1383, 1297, 1236, 1119, 693 cm^{-1}. HRMS (ESI) calcd for $C_{22}H_{17}BrO_2P$ $[M+H]^+$ 423.0144, found 423.0145.

(3-Bromo-2-hydroxynaphthalen-1-yl)diphenylphosphine oxide (**3j**): Light yellow crystal, m.p. 175～176 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 14.22 (s, 1H), 8.28 (s, 1H), 7.81～7.76 (m, 4H), 7.64 (d, $J_{H\text{-}H}$=7.9 Hz, 1H), 7.59～7.55 (m, 2H), 7.48～7.46 (m, 4H), 7.25～7.20 (m, 2H), 7.10 (t, $J_{H\text{-}H}$=7.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.6 (d, $J_{P\text{-}C}$=3.4 Hz), 138.6 (d, $J_{P\text{-}C}$=2.1 Hz, CH), 132.8 (d, $J_{P\text{-}C}$=2.8 Hz, CH), 132.6 (d, $J_{P\text{-}C}$=8.5 Hz), 132.0 (d, $J_{P\text{-}C}$=10.8 Hz, CH), 130.9 (CH), 128.9 (d, $J_{P\text{-}C}$=12.5 Hz, CH), 128.5 (d, $J_{P\text{-}C}$=10.0 Hz), 128.3 (CH), 127.1 (CH), 124.7 (d, $J_{P\text{-}C}$=6.4 Hz, CH), 123.8 (CH), 115.3 (d, $J_{P\text{-}C}$=11.9 Hz, CH), 100.9 (d, $J_{P\text{-}C}$=99.1 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ: 43.1; IR (KBr) ν: 1558, 1511, 1468, 1437, 1405, 1236, 1125 cm^{-1}. HRMS (ESI) calcd for $C_{22}H_{17}BrO_2P$ $[M+H]^+$ 423.0144, found 423.0146.

(2-Hydroxynaphthalen-1-yl)di-*p*-tolylphosphine oxide (**3n**): Pink crystal, m.p. 137～138 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 13.42 (s, 1H), 7.89 (d, $J_{H\text{-}H}$=9.0 Hz, 1H), 7.70～7.64 (m, 5H), 7.28～7.24 (m, 5H), 7.20～7.16 (m, 2H), 7.10～7.06 (m, 1H), 2.37 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.9 (d, $J_{P\text{-}C}$=2.8 Hz), 143.0 (d, $J_{P\text{-}C}$=2.7 Hz), 135.9 (d, $J_{P\text{-}C}$=2.0 Hz, CH), 133.6 (d, $J_{P\text{-}C}$=8.7 Hz), 132.0 (d, $J_{P\text{-}C}$=10.9 Hz, CH), 129.5 (d, $J_{P\text{-}C}$=12.8 Hz, CH), 129.0 (CH), 128.7 (d, $J_{P\text{-}C}$=61.0 Hz, CH), 128.2 (d, $J_{P\text{-}C}$=8.9 Hz), 126.7 (CH), 125.0 (d, $J_{P\text{-}C}$=6.6 Hz, CH), 122.9 (CH), 121.4 (d, $J_{P\text{-}C}$=8.7 Hz, CH), 99.7 (d, $J_{P\text{-}C}$=102.1 Hz), 21.6 (CH_3); ^{31}P NMR (162 MHz, $CDCl_3$) δ: 42.7; IR (KBr) ν: 2921, 1851, 1598, 1470, 1449, 1418, 1366, 1236, 1113 cm^{-1}. HRMS (ESI) calcd for $C_{24}H_{22}O_2P$ $[M+H]^+$ 373.1352, found 373.1353.

(2-Hydroxynaphthalen-1-yl)bis(4-methoxyphenyl)phosphine oxide (**3o**): Pink crystal, m.p. 173～174 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 13.34 (s, 1H), 8.07 (d, $J_{H\text{-}H}$=9.0 Hz, 1H), 7.83 (d, $J_{H\text{-}H}$=7.8 Hz, 1H), 7.66～7.61 (m, 4H), 7.46 (d, $J_{H\text{-}H}$=8.1 Hz), 7.27～7.19 (m, 2H), 7.16～7.13 (m, 1H), 7.12～7.09 (m, 4H), 3.80 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 165.7 (d, $J_{P\text{-}C}$=3.0 Hz), 162.8 (d, $J_{P\text{-}C}$=2.7 Hz), 136.5 (CH), 134.0 (d, $J_{P\text{-}C}$=12.1 Hz, CH), 133.7 (d, $J_{P\text{-}C}$=8.3 Hz), 129.6 (CH), 128.4 (d,

$J_{P\text{-}C}$=8.8 Hz, CH), 127.5 (CH), 124.9 (d, $J_{P\text{-}C}$=6.2 Hz, CH), 123.6 (d, $J_{P\text{-}C}$=110.9 Hz), 123.5 (CH), 121.0 (d, $J_{P\text{-}C}$=8.6 Hz, CH), 115.0 (d, $J_{P\text{-}C}$=13.2 Hz, CH), 101.1 (d, $J_{P\text{-}C}$=101.3 Hz), 55.9 (CH_3); ^{31}P NMR (162 MHz, DMSO-d_6) δ: 39.9; IR (KBr) ν: 1594, 1505, 1417, 1372, 1242, 1118 cm^{-1}. HRMS (ESI) calcd for $C_{24}H_{22}O_4P$ [M+H]$^+$ 405.1250, found 405.1247.

Bis(4-fluorophenyl)(2-hydroxynaphthalen-1-yl)phosphine oxide (**3p**): Light yellow solid, m.p. 190～191 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 13.14 (s, 1H), 7.94 (d, $J_{H\text{-}H}$=9.0 Hz, 1H), 7.82～7.72 (m, 4H), 7.23～7.12 (s, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.2 (d, J=2.1 Hz), 165.3 (dd, J=3.0, 252.9 Hz), 136.5 (d, J=2.2 Hz, CH), 134.6 (dd, J=8.7, 12.4 Hz, CH), 133.2 (d, J=8.7 Hz), 129.3 (CH), 128.3 (d, J=8.5 Hz), 128.0 (dd, J=3.6, 108.4 Hz), 127.1 (CH), 124.4 (d, J=6.6 Hz, CH), 123.2 (CH), 121.4 (d, J=9.5 Hz, CH), 116.4 (d, J=12.5 Hz, J=20.6 Hz, CH), 98.7 (d, J=104.3 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ: 41.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −104.8; IR (KBr) ν: 2920, 1602, 1509, 1432, 1400, 1353, 1240, 1112 cm^{-1}. HRMS (ESI) calcd for $C_{22}H_{16}F_2O_2P$ [M+H]$^+$ 381.0850, found 381.0848.

(3-Bromo-2-hydroxynaphthalen-1-yl)di-*p*-tolylphosphine oxide (**3q**): Colorless solid, m.p. 187～188 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 14.36 (s, 1H), 7.68～7.59 (m, 4H), 7.60 (d, $J_{H\text{-}H}$=8.0 Hz, 1H), 7.28～7.24 (m, 5H), 7.19 (t, $J_{H\text{-}H}$=7.6 Hz, 1H), 7.10 (t, $J_{H\text{-}H}$=7.4 Hz, 1H), 2.36 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.4 (d, $J_{P\text{-}C}$=3.5 Hz), 143.4 (d, $J_{P\text{-}C}$=2.7 Hz), 138.4 (CH), 132.6 (d, $J_{P\text{-}C}$=8.5 Hz), 132.1 (d, $J_{P\text{-}C}$=11.0 Hz, CH), 129.6 (d, $J_{P\text{-}C}$=13.0 Hz, CH), 128.5 (d, $J_{P\text{-}C}$=9.9 Hz), 128.2 (CH), 127.9 (d, $J_{P\text{-}C}$=107.6 Hz), 127.0 (CH), 124.8 (d, $J_{P\text{-}C}$=6.4 Hz, CH), 123.7 (CH), 115.3 (d, $J_{P\text{-}C}$=11.7 Hz), 101.0 (d, $J_{P\text{-}C}$=99.2 Hz), 21.6 (CH_3); ^{31}P NMR (162 MHz, $CDCl_3$) δ: 43.2; IR (KBr) ν: 2921, 1600, 1511, 1435, 1405, 1348, 1241, 1116, 1074, 804 cm^{-1}. HRMS (ESI) calcd for $C_{24}H_{21}BrO_2P$ [M+H]$^+$ 451.0457, found 451.0459.

(7-Bromo-2-hydroxynaphthalen-1-yl)di-*p*-tolylphosphine oxide (**3r**): Light yellow crystal, m.p. 207～208 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 13.47 (s, 1H), 7.84 (d, $J_{H\text{-}H}$=9.0 Hz, 1H), 7.67～7.62 (m, 4H), 7.54 (dd, $J_{H\text{-}H}$=8.6, 1.5 Hz, 1H), 7.43 (s, 1H), 7.31～7.27 (m, 4H), 7.17 (dd, $J_{H\text{-}H}$=9.0, 3.9 Hz, 1H), 2.41 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.4 (d, $J_{P\text{-}C}$=2.4 Hz), 143.4 (d, $J_{P\text{-}C}$=2.8 Hz), 135.5 (CH), 134.8 (d, $J_{P\text{-}C}$=8.0 Hz), 132.0 (d, $J_{P\text{-}C}$=11.0 Hz, CH), 130.3 (CH), 129.6 (d, $J_{P\text{-}C}$=12.7 Hz, CH), 129.0, 127.9, 127.2 (d, $J_{P\text{-}C}$=6.7 Hz, CH), 126.6 (d, $J_{P\text{-}C}$=8.7 Hz), 126.2 (CH), 121.8 (d, $J_{P\text{-}C}$=8.6 Hz), 121.3 (CH), 99.8 (d, $J_{P\text{-}C}$=101.5 Hz), 21.7 (CH_3); ^{31}P NMR (162 MHz, $CDCl_3$) δ: 42.3; IR (KBr) ν: 2919, 1613, 1501, 1443, 1388, 1233, 1113 cm^{-1}. HRMS (ESI) calcd for $C_{24}H_{21}BrO_2P$ [M+H]$^+$ 451.0457, found 451.0455.

(2-Hydroxy-7-methoxynaphthalen-1-yl)di-*p*-tolylphosphine oxide (**3s**): Pink crystal, m.p. 183～185 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 13.24 (s, 1H), 7.82 (d, $J_{H\text{-}H}$=8.9 Hz, 1H), 7.69～7.64 (m, 4H), 7.58 (dd, $J_{H\text{-}H}$=8.9, 1.4 Hz, 1H), 7.28～7.25 (m, 4H), 7.03 (dd, $J_{H\text{-}H}$=8.9, 4.0 Hz, 1H), 6.83 (dd, $J_{H\text{-}H}$=8.9, 2.3 Hz, 1H), 6.54 (d, $J_{H\text{-}H}$=2.0 Hz, 1H), 3.23 (s, 3H), 2.39 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.5 (d, $J_{P\text{-}C}$=2.3 Hz), 158.0, 143.0 (d, $J_{P\text{-}C}$=2.8 Hz), 135.5 (d, $J_{P\text{-}C}$=1.5 Hz, CH), 135.2 (d, $J_{P\text{-}C}$=8.0 Hz), 132.0 (d, $J_{P\text{-}C}$=10.9 Hz, CH), 130.3 (CH), 129.5 (d, $J_{P\text{-}C}$=13.2 Hz, CH), 129.1 (d, $J_{P\text{-}C}$=106.5 Hz), 123.3 (d, $J_{P\text{-}C}$=9.3 Hz), 118.6 (d, $J_{P\text{-}C}$=8.5 Hz, CH), 114.9 (CH), 105.3 (d, $J_{P\text{-}C}$=7.5 Hz, CH), 99.2 (d, $J_{P\text{-}C}$=102.9 Hz), 54.9 (CH_3), 21.6 (CH_3); ^{31}P NMR (162 MHz, $CDCl_3$) δ: 42.3; IR (KBr) ν: 2955, 2919, 2850, 1615, 1512, 1454, 1368, 1237, 1218, 1134 cm^{-1}. HRMS (ESI) calcd for $C_{25}H_{24}O_3P$ [M+H]$^+$ 403.1458, found 403.1455.

(7-Bromo-2-hydroxynaphthalen-1-yl)bis(4-methoxyphenyl) phosphine oxide (**3t**): Colorless solid, m.p. 196～197 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 13.50 (s, 1H), 7.83 (d, $J_{H\text{-}H}$=9.0 Hz, 1H), 7.71～7.66 (m, 4H), 7.53 (d, $J_{H\text{-}H}$=8.6 Hz, 1H), 7.45 (s, 1H), 7.26 (d, $J_{H\text{-}H}$=6.8 Hz, 1H), 7.16 (dd, $J_{H\text{-}H}$=9.0, 3.7 Hz, 1H), 6.97 (d, $J_{H\text{-}H}$=8.5 Hz, 4H), 3.82 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.3 (d, $J_{P\text{-}C}$=2.2 Hz), 163.0 (d, $J_{P\text{-}C}$=2.8 Hz), 135.4 (d, $J_{P\text{-}C}$=2.5 Hz, CH), 134.8 (d, $J_{P\text{-}C}$=7.8 Hz, CH), 134.0 (d, $J_{P\text{-}C}$=11.5 Hz, CH), 127.2 (d, $J_{P\text{-}C}$=6.6 Hz, CH), 126.6 (d, $J_{P\text{-}C}$=8.6 Hz), 126.1 (CH), 123.5, 122.4, 121.8 (d, $J_{P\text{-}C}$=8.6 Hz, CH), 121.3, 114.5 (d, $J_{P\text{-}C}$=12.9 Hz, CH), 99.7 (d, $J_{P\text{-}C}$=101.6 Hz), 55.4 (CH_3); ^{31}P NMR (162 MHz, $CDCl_3$) δ: 41.3; IR (KBr) ν: 2922, 2851, 1593, 1565, 1503, 1443, 1290, 1258, 1235, 1118 cm^{-1}. HRMS (ESI) calcd for $C_{24}H_{21}BrO_4P$ [M+H]$^+$ 483.0355, found 483.0357.

Supporting Information Copies of NMR spectra of products. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn/.

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