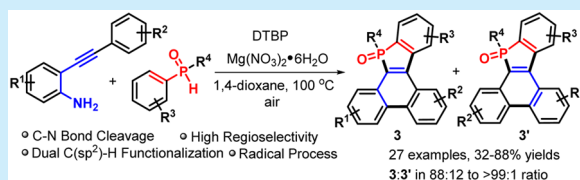


Synthesis of Tribenzo[*b,e,g*]phosphindole Oxides via Radical Bicyclization Cascades of *o*-ArylalkynylanilinesJie Li,<sup>†</sup> Wen-Wen Zhang,<sup>†</sup> Xiao-Jing Wei,<sup>†</sup> Wen-Juan Hao,<sup>\*,†</sup> Guigen Li,<sup>‡</sup> Shu-Jiang Tu,<sup>\*,†</sup> and Bo Jiang<sup>\*,†</sup><sup>†</sup>School of Chemistry & Materials Science, Jiangsu Normal University, Xuzhou 221116, P. R. China<sup>‡</sup>Institute of Chemistry & BioMedical Sciences, Nanjing University, Nanjing 210093, P. R. China

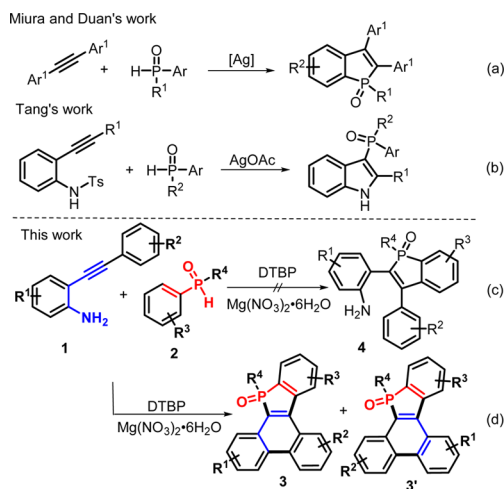
## S Supporting Information

**ABSTRACT:** A new DTBP/Mg(NO<sub>3</sub>)<sub>2</sub>-mediated bicyclization cascade of *o*-arylalkynylanilines with secondary arylphosphine oxides has been developed, enabling dual C(sp<sup>2</sup>)-H functionalization along with the cleavage of the C-N bond. The combination between regioselective P-centered radical-triggered [3 + 2] cyclization and C-centered radical-induced cross-coupling in a one-pot manner delivered 27 examples of tribenzo[*b,e,g*]phosphindole oxides with generally high regioselectivity. A reasonable mechanism for forming such products involving radical addition-cyclization cascade is proposed.



As a highly important and valuable class of phosphorus-containing heterocycles, fused benzophosphole derivatives have attracted considerable attention in organic chemistry and material science due to their unique physical and optical properties,<sup>1</sup> which show great potential in optoelectrochemical materials.<sup>2</sup> Consequently, substantial effort has been made toward identifying efficient methods for the preparation of such compounds. Generally, the vast majority of well-established synthetic strategies for benzophosphole syntheses include nucleophilic substitution of a P-X bond with organolithium or organomagnesium reagents,<sup>3</sup> metal-catalyzed [2 + 2 + 2] cycloaddition of dialkynylphosphines with polyynes,<sup>4</sup> intramolecular cross-coupling of aryl halides with hydrophosphines,<sup>5</sup> dehydrogenative cyclization of hydrophosphine oxides,<sup>6</sup> palladium-catalyzed cyclization of triarylphosphines,<sup>7</sup> and multiple cyclizations of *o*-halide-substituted arylalkynes with phenylphosphorus compounds.<sup>8</sup> Specifically, the group of Miura<sup>9a</sup> and Duan<sup>9b</sup> independently reported the Ag-mediated arylphosphine oxide radical-triggered cyclization of symmetrical diarylalkynes, providing direct approaches toward benzo[*b*]phosphole oxides in an atom-economic fashion (Scheme 1a). Later, various catalytic variations of such reactions were developed using different oxidants.<sup>10</sup> However, studies in the reactivity of unsymmetrical diarylalkynes have been almost ignored. Recently, Tang et al. described a silver-mediated cycloaddition between *N*-Ts-2-alkynylanilines and secondary phosphine oxides to access 3-phosphinoylindole derivatives (Scheme 1b).<sup>11</sup> To further investigate the regioselectivity of this radical cyclization and expand the family of benzophospholes, *N*-unprotected *o*-arylalkynylanilines were subjected to a reaction with diarylphosphine oxides in order to establish a regioselective radical [3 + 2] cyclization (Scheme 1c). Unexpectedly, regioisomers of pentacyclic tribenzo[*b,e,g*]phosphindole oxides 3 and 3' were generated in a functional-group-compatible manner through

## Scheme 1. Profiles for P-Centered Radical Cyclization



dual C(sp<sup>2</sup>)-H functionalization along with the cleavage of the C-N bond without observation of the desired benzo[*b*]phosphole oxides 4 as we originally planned (Scheme 1d). This protocol represents the first bicyclization procedure for the direct synthesis of these new pentacyclic tribenzo[*b,e,g*]phosphindole oxides by merging a regioselective P-centered radical-triggered [3 + 2] cyclization with C-centered radical-induced cross-coupling in a one-pot manner. Herein, we would report this serendipitous and interesting radical transformation.

Phosphine oxide radicals from HP(=O)R<sup>1</sup>R<sup>2</sup> precursor show high reactivity with unsaturated bonds and behave as ideal radical partners for constructing organophosphorus molecules.<sup>12</sup> At the

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outset of our studies, diphenylphosphine oxide **2a** was selected as a phosphine oxide radical donor and subjected to the reaction of 2-((4-chlorophenyl)ethynyl)aniline **1a** as unsymmetrical diarylalkyne substrate using silver nitrate ( $\text{AgNO}_3$ , 2.0 equiv) as a promoter. The reaction smoothly proceeded in 1,4-dioxane at 100 °C under air conditions. Instead of the desired benzo[*b*]-phosphole oxide **4a**, unexpected inseparable tribenzo[*b,e,g*]-phosphindole oxide isomers **3a** and **3a'** in a 98:2 ratio were provided in total 56% yield, which were fully characterized by NMR spectroscopic analysis (Table 1, entry 1). In view of this

Table 1. Optimization of Reaction Conditions<sup>a</sup>

entry	oxidant (equiv)	solvent	temp (°C)	yield <sup>b</sup> (%)
1 <sup>c</sup>	$\text{AgNO}_3$ (2.0)	1,4-dioxane	100	56
2 <sup>c</sup>	$\text{AgOAc}$ (2.0)	1,4-dioxane	100	trace
3	$\text{AgOAc}$ (2.0)	1,4-dioxane	100	41
4	$\text{AgOAc}$ (2.0)	1,4-dioxane	100	18 <sup>d</sup>
5	$\text{AgOAc}$ (2.0)	1,4-dioxane	100	13 <sup>e</sup>
6	$\text{AgOAc}$ (2.0)	1,4-dioxane	100	trace <sup>f</sup>
7	$\text{AgOTf}$ (2.0)	1,4-dioxane	100	51
8	DTBP (2.0)	1,4-dioxane	100	72
9	TBHP (2.0)	1,4-dioxane	100	58
10	TBPP (2.0)	1,4-dioxane	100	54
11	$\text{K}_2\text{S}_2\text{O}_8$ (2.0)	1,4-dioxane	100	49
12	DTBP (2.0)	$\text{CH}_3\text{CN}$	100	trace
13	DTBP (2.0)	THF	100	trace
14	DTBP (2.0)	DCE	100	trace
15	DTBP (2.0)	toluene	100	48
16	DTBP (2.0)	1,4-dioxane	80	ND <sup>g</sup>
17	DTBP (2.0)	1,4-dioxane	90	39
18	DTBP (2.0)	1,4-dioxane	110	66

<sup>a</sup>Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), oxidant (2.0 equiv),  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (2.0 equiv), solvent (5.0 mL), under air conditions. <sup>b</sup>Isolated total yield based on substrate **1a**. <sup>c</sup>Without  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ . <sup>d</sup>Use of  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (2.0 equiv). <sup>e</sup>Use of  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (2.0 equiv). <sup>f</sup>Use of  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  (2.0 equiv).

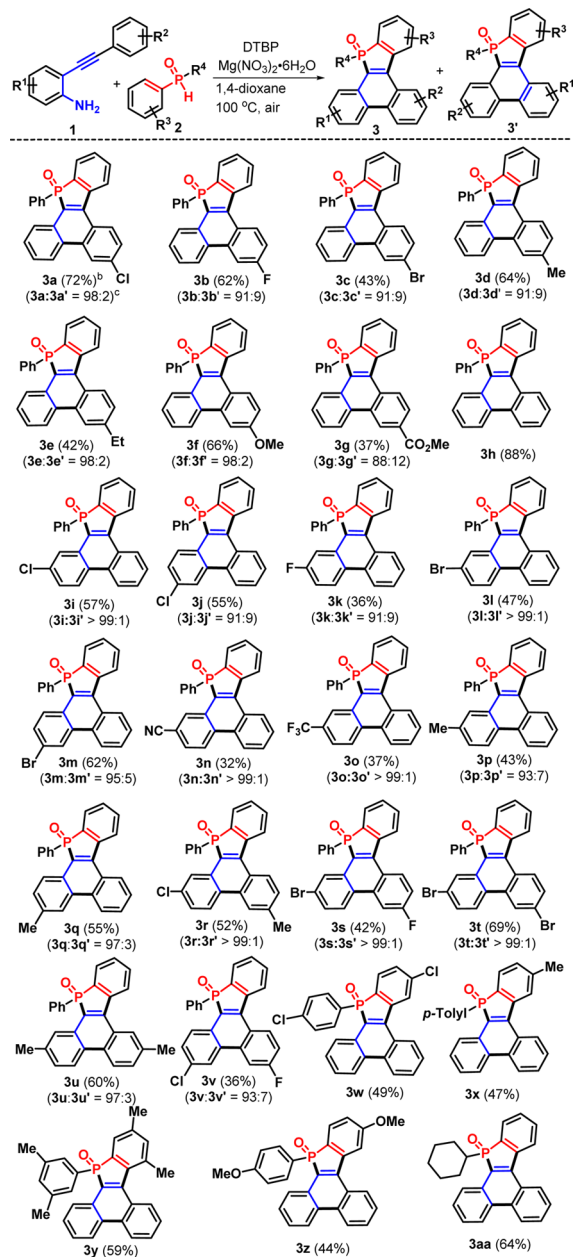
<sup>g</sup>ND = not detected.

interesting result, we worked to improve the efficiency of this deaminated bicyclization reaction. Exchanging  $\text{AgNO}_3$  for  $\text{AgOAc}$  completely suppressed the formation of **3a** and **3a'**, but 3-phosphinoindole **5a** (21%) was observed,<sup>11</sup> indicating that nitrate anions play a key role in this transformation (entry 2). Next, we considered employing magnesium nitrate as nitrate anion source to investigate the feasibility of this transformation. As we expected, the reaction in the presence of 2.0 equiv of  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  gave a 41% yield of **3a** and **3a'** (entry 3). Other nitrate salts including  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , and  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  gave very inferior outcomes<sup>13</sup> (entries 4–6). Employment of silver trifluoromethanesulfonate ( $\text{AgOTf}$ ) resulted in 51% yield of isomers **3a** and **3a'** but still relatively low compared with  $\text{AgNO}_3$  (entry 7 vs entry 1). Instead of silver salt oxidants, four other oxidants including di-*tert*-butyl peroxide (DTBP), *tert*-butyl hydroperoxide (TBHP), *tert*-butyl perox-

ybenzoate (TBPB), and  $\text{K}_2\text{S}_2\text{O}_8$  were chosen to evaluate improvement in the yield of isomers. The experimental results indicated that all these oxidants can drive the reaction to access products **3a** and **3a'** (entries 8–11), in which DTBP was proven to be most efficient for this oxidative system (72%, entry 8). By taking the combination of DTBP with  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , we then investigated the solvent effect by using other aprotic solvents, such as acetonitrile ( $\text{CH}_3\text{CN}$ ), tetrahydrofuran (THF), 1,2-dichloroethane (DCE), and toluene, showing that all these solvents have no positive effect on the yield of **3a** as compared with 1,4-dioxane (entries 12–15 vs entry 8). The reaction efficiency was found to show an important temperature dependence. With the temperature at 80 °C, this transformation hardly occurred to deliver the desired product (entry 16), whereas the relatively lower conversion into isomers **3a** and **3a'** was observed as the reaction was conducted at either 90 or 110 °C (entries 17 and 18).

With these optimal reaction conditions in hand, we then systematically studied the generality of this DTBP/ $\text{Mg}(\text{NO}_3)_2$ -mediated bicyclization cascade toward tribenzo[*b,e,g*] phosphindole oxides **3** by examining *o*-arylalkynylanilines and phosphine oxide components (Scheme 2). At first, *o*-arylalkynylanilines with diverse functionalities were evaluated in combination with phosphine oxide **2a**. Substituents with both electronically poor and rich properties at different positions of the arylalkynyl ( $\text{R}^2$ ) moiety would be accommodated, confirming the success of transformations, as the corresponding regioisomers **3a–g** and **3a'–g'** were afforded in 37–72% yields and 88:12–98:2 ratios. Functional groups such as chloride (**1a**), fluoride (**1b**), bromide (**1c**), methyl (**1d**), ethyl (**1e**), methoxy (**1f**), and ester (**1g**) were compatible with the oxidative conditions. Alternatively, 2-(phenylethynyl)aniline **1h** was an efficient reaction partner, enabling its radical-triggered [3 + 2] cyclization/cross-deaminated coupling cascade to access product **3h** as a sole isomer in 88% yield. The reaction can be extended to different functional groups such as chloride (**1i** and **1j**), fluoride (**1k**), bromide (**1l** and **1m**), cyano (**1n**), trifluoromethyl (**1o**), and methyl (**1p** and **1q**) located at the 4- or 5-positions of the aniline ring, and structurally diverse isomers **3i–q** and **3i'–q'** in 32–62% yields and 91:9 to >99:1 ratios were rendered under the DTBP/ $\text{Mg}(\text{NO}_3)_2$ -mediated conditions. Among them, the presence of strong electron-withdrawing groups such as fluoride, cyano, and trifluoromethyl proved to be more reluctant to undergo the bicyclization process, in which **3k** and **3n,o** were generated in substantially decreased yields of 32–37%. Moreover, the electronic nature of substituents on both the aniline ring ( $\text{R}^1$ ) and arylalkynyl ( $\text{R}^2$ ) moieties was also probed. The reaction occurred smoothly with a variety of functional groups on both aryl moieties, giving access to isomers **3r–v** and **3r'–v'** with yields ranging from 36% to 69%, some of which were offered with an excellent regioselectivity (**3r–t** in >99:1 ratio).

Next, the scope with respect to the phosphine oxide component was investigated. The bicyclization reaction can tolerate various phosphine oxides **2b–e** carrying both electron-deficient (Cl, **2b**) and electron-rich (Me, **2c** and **2d**; MeO, **2e**) groups at different positions of arene rings, leading to the formation of tribenzo[*b,e,g*]phosphindole oxides **3w–z** in moderate yields (Scheme 2). Cyclohexyl(phenyl)phosphine oxide **2f** was also found to be a suitable coupling partner and can be transformed into cyclohexyl-substituted product **3aa** in 64% yield. It is noteworthy that the current oxidative protocol represents a new and practical pathway for assembling richly decorated tribenzo[*b,e,g*]phosphindole oxides **3** with generally

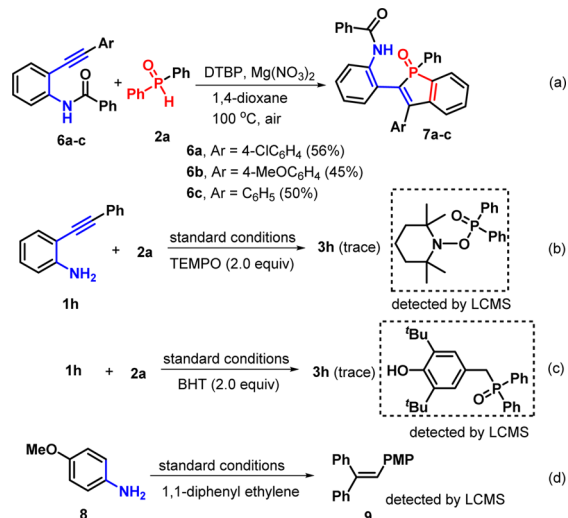
Scheme 2. Substrate Scope for Synthesis of Products 3<sup>a</sup>

<sup>a</sup>Reaction conditions: 1 (0.2 mmol), 2 (0.4 mmol), DTBP (2.0 equiv),  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (2.0 equiv), 1,4-dioxane (5.0 mL), under air conditions. <sup>b</sup>Isolated total yield based on substrate 1. <sup>c</sup>Regioisomer ratio based on the analysis of <sup>31</sup>P NMR.

excellent regioselectivity through DTBP/ $\text{Mg}(\text{NO}_3)_2$ -mediated bicyclization cascade involving deaminated  $\text{C}(\text{sp}^2)\text{--H}$  functionalization. In the case of 3j, its structure was unequivocally confirmed by carrying out single-crystal X-ray diffraction (see the Supporting Information).

Some control experiments were conducted to gain mechanistic insight into this bicyclization. *N*-Protected *o*-aryalkynylanilines 6a–c were treated with 2a under the standard conditions, and the corresponding benzo[*b*]phosphole oxides 7a–c as a single regioisomer were isolated in 45–56% yields. The regioselectivity may depend on its hydrogen bonds and steric effects (Scheme 3a), showing that protected amines inhibit cross-deaminated coupling; [3 + 2] cyclization occurred prior to the

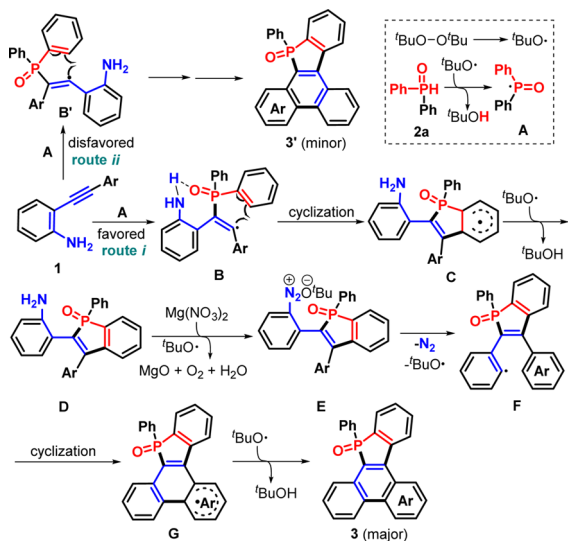
Scheme 3. Control Experiments



cross-deaminated coupling step. Notably, radical inhibitors 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) and butylhydroxytoluene completely suppressed the reaction and provided TEMPO-P and BHT-P adducts (detected by LC–MS analysis), respectively (Scheme 3b,c), suggesting that the [3 + 2] cyclization involves a radical mechanism. To confirm the mechanism of deaminated coupling, 4-methoxyaniline 8 was subjected to 1,1-diphenyl ethylene under the standard conditions with observation of the desired product 9 by LC–MS analysis (Scheme 3d). These outcomes supported a radical process for deaminated coupling.

On the basis of our own observations and literature survey,<sup>9–11</sup> a reasonable radical-triggered mechanism was proposed as depicted in Scheme 4. First, a P-centered radical A is generated in

Scheme 4. Plausible Reaction Pathway



situ from diphenylphosphine oxide 2a through single-electron transfer (SET) mediated by the homolysis of DTBP under heating conditions.<sup>14</sup> Next, regioselective addition of radical A to alkynyl unit of *o*-aryalkynylanilines 1 favors a vinyl radical B (route *i*), depending on hydrogen bonds and steric effect, followed by homolytic aromatic substitution (HAS B to D) to



generate benzo[*b*]phosphole oxide **D**. Intermediate **D** mediated by Mg(NO<sub>3</sub>)<sub>2</sub> and *tert*-butoxyl radical (<sup>t</sup>BuO•) may provide intermediate **E**, although this transformation is unclear for us so far, which gives the phenyl radical **F** by decomposition of itself with concurrent release of N<sub>2</sub> and <sup>t</sup>BuO•. Phenyl radical **F** undergoes a second HAS (**F** to **3**) to access final major product **3**. The formation of minor product **3'** undergoes a radical process very similar to that mentioned above (route *ii*).

In conclusion, we have established a new radical-triggered bicyclization of *o*-aryalkynylanilines with a large variety of functional groups by which a series of structurally diverse tribenzo[*b,e,g*]phosphindole oxides with generally high regioselectivity would be synthesized through double C(sp<sup>2</sup>)–H functionalization. This reaction merged P-centered radical-triggered [3 + 2] cyclization with C-centered radical-induced cross-coupling in a one-pot manner, resulting in multiple C–P and C–C bond-forming events along with C–N cleavage of aniline substrates. The protocol features bond-forming/annulation efficiency and functional group tolerance, providing a direct and powerful synthetic method for constructing phosphorus-containing heterocycles. Further investigation on the mechanism of this radical bicyclization is underway in our laboratory.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b02071.

Experimental procedures and spectroscopic data for all new compounds **3a–aa** and **7a–c** (PDF)

X-ray crystal data for **3j** (CIF)

X-ray crystal data for **7c** (CIF)

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### Notes

The authors declare no competing financial interest.

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