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Synthesis of Dibenzophosphole Oxides *via* Palladium-Catalyzed Intramolecular Direct Arylation Reactions of *ortho*-Halodiarylphosphine Oxides

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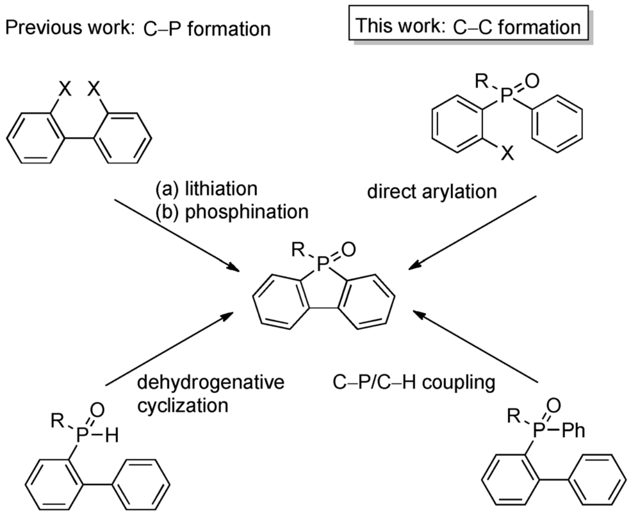
Abstract: A palladium-catalyzed intramolecular direct arylation reaction was developed and two efficient one-pot sequential direct arylation/Suzuki–Miyaura coupling and intra/intermolecular direct arylation reations were also realized. The method provides a simple and straightforward procedure for the synthesis and further functionalization of dibenzophosphole oxides from easily accessible *ortho*-halodiarylphosphine oxides in good to excellent yields.

Keywords: catalysis; C–H activation; direct arylation; heterofluorenes; palladium

In the field of organic materials, heterofluorenes have attracted considerable attention due to a growing interest as components of optical and electronic devices. The particular interaction between the heteroatoms (nitrogen,[1,2] silicon,[1,3] boron,[4,5] sulfur,[6] germanium,[7] etc.) and the π-conjugated polyphenylene-like framework in the oligomers and polymers determines their electronic nature which will play the key role in the performance of the final materials. To achieve the desired function such as electroluminescence and high charge mobility, the properties of π-conjugated oligomers and polymers need to be tailored at the molecular level. However, the facile synthesis of differently substituted heterofluorenes is still a challenge for chemists, limiting the application of heterofluorenes. In recent years, direct arylation reactions that allow direct C–C formation by C–H activation have emerged as a powerful tool for the construction of aryl-aryl bonds and thus have many advantages over the more commonly employed cross-coupling reactions.[8] A few of heterofluorenes including carbazoles, dibenzofurans, dibenzothiophenes, and silafluorenes have been synthesized *via* intramolecular direct arylation.[3b,9] To the best of our knowledge, the synthesis of dibenzophospholes *via* direct arylation of *ortho*-halodiarylphosphines has never been reported and the difficulty may be due to the instability and undesired coordination of the metal with the phosphine, although the dibenzophospholes are valuable building blocks for tailoring organic π-conjugated materials and their photophysics and redox properties can be efficiently tuned through the diverse and reversible phosphorus chemistry.[10]

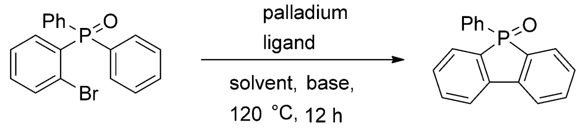
The previously used methods[11] to synthesize dibenzophospholes often required the synthesis of functionalized biaryl subunits and/or suffered from the modest functional group tolerance, particularly in the many cases using aryllithium reagents. On the other hand, the frequently used procedure through metathesis reaction between 2,2′-dilithiated biaryls and $RPCl_2$ is suitable for the synthesis of the symmetrical dibenzophosphole oxides, only being restricted by the availability of the dihalobiaryls. To overcome the issues mentioned above and increase the steric effect between the two phenyl groups, we attempted a palladium-catalyzed intramolecular direct arylation as a new synthetic route to symmetrical and unsymmetrical dibenzophosphole oxides from the easily accessible substrates (Scheme 1). These oxides can be easily reduced to phosphines by treatment with $HSiCl_3$ and further functionalization (sulfuration, borylation, methylation, metalation, etc.) can be carried out following established procedures.[12]

Considering the significant potential of palladium in tandem catalysis, we sought to investigate the possibility of a one-pot sequential coupling protocol for the synthesis of functionalized dibenzophosphole oxides. Fagnou reported the tandem Heck/direct arylation and Heck/direct arylation/hydrogenation reactions and the key factor influencing reaction success was the use of bromochloro substrates to ensure good



Scheme 1. Syntheis of dibenzophosphole oxides.

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



1a **2a**

Entry	x	y	Base	Solvent	Yield [%][b]
1	3	6	K_2CO_3	DMF	88
2	3	6	K_2CO_3	dioxane	86
3	3	6	K_2CO_3	THF	55
4	3	6	K_2CO_3	toluene	94
5	3	6	K_2CO_3	DMAc	98
6	3	6	Cs_2CO_3	DMAc	83
7	3	6	NEt_3	DMAc	20
8	3	6	KOAc	DMAc	94
9[c]	3	6	K_2CO_3	DMAc	82
10	1	2	K_2CO_3	DMAc	93
11	3	–	K_2CO_3	DMAc	93
12	3	–	KOAc	DMAc	88
13[d]	3	–	K_2CO_3	DMAc	81
14	1	–	K_2CO_3	DMAc	92

[a] Unless otherwise specified, the reaction was carried out using **1a** (1.0 mmol), base (1.5 mmol), $Pd(OAc)_2$ (x mol%), and $PCy_3·HBF_4$ (y mol%) in solvent (3 mL) at 120 °C for 12 h under N_2.

[b] Isolated yields.

[c] At 90 °C.

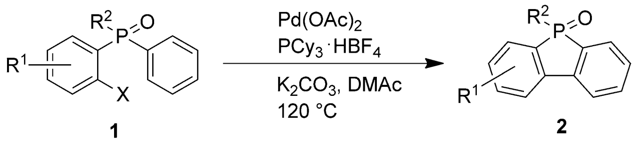
[d] $Pd_2(dba)_3$ was used.

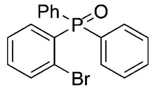
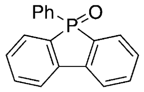
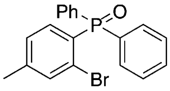
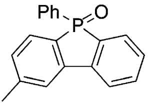
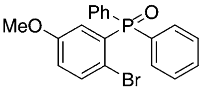
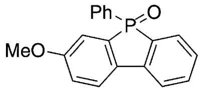
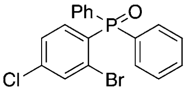
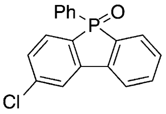
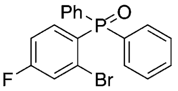
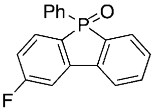
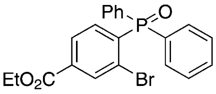
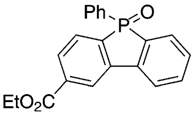
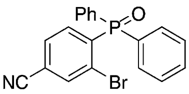
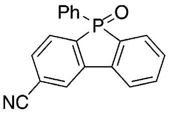
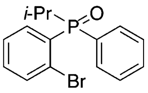
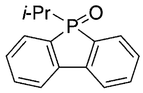
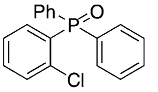
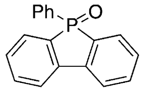
site-selectivity.[13] In fact, it will be a tough task to use dihalo-substituted substrates in the tandem reactions in spite of their being prepared readily compared with those bearing the different reactive groups. Herein, we present a new synthetic method for the synthesis of dibenzophosphole oxides using the intramolecular direct arylation as the key transformation. Based on the good compatibility of palladium catalysts, the palladium-catalyzed sequential direct arylation/Suzuki–Miyaura coupling and intra-/intermolecular direct arylation reactions were also realized.

Initial reaction optimization was performed with $OPPh_2(o\text{-}C_6H_4Br)$ **1a** as a model substrate (Table 1). To our delight, reactions in DMF at 120 °C using a $Pd(OAc)_2/PCy_3·HBF_4$ catalyst system and K_2CO_3 as the base lead to the desired dibenzophosphole oxide product **2a** in 88% yield (entry 1). Next, we investigated the effects of different solvents, bases, and temperature on the reaction outcome. Among the selected solvents, DMAc was superior to other solvents such as dioxane, toluene, and THF, providing the product **2a** in almost quantitative yield (entries 2 to 5). With DMAc as the solvent, a survey of different bases including Cs_2CO_3, NEt_3, and KOAc revealed that no improvement was observed, suggesting that K_2CO_3 is the best choice for this transformation (entries 6 to 8). Attempts to lower the reaction temperature from 120 °C to 90 °C led to inferior results, producing **2a** in only 82% yield (entry 9). Interestingly, no significant drop of the yield (93%) was observed when using a catalyst loading as low as 1 mol% (entry 9). It should be noted that the reaction can be performed in the absence of phosphine ligand and similar results were obtained (entries 10 and 14), reaching 92% yield. For these cases, the palladium colloids or nanoparticles generated from $Pd(OAc)_2$ under the reaction conditions could behave as the active catalyst.[14]

With the optimized conditions in hand, we then investigated the scope of the direct arylation reactions and the results are summarized in Table 1. We focused on $Pd(OAc)_2/PCy_3·HBF_4$ as the catalyst system because it gave very good yields of the products and is air stable and inexpensive. As shown in Table 2, the substrates with electron-rich and poor functional groups can be tolerated to give the desired products in good to excellent yields. For electron-donating groups, methyl- and methoxy-substituted products **2b** and **2c** were obtained in 85% and 96% yields, respectively (entries 2 and 3). In addition, various electron-withdrawing groups are suitable for these transformations. We were pleased to find that the chloro-substituted substrate could be successfully employed to give **2d** in 94% yield without any problem (entry 4). In the cases of **2c** and **2d**, the methoxy and chloro groups could be used to extend the π-conjugated systems of dibenzophosphole oxides, applying the previously reported coupling methods.[3b,15] Fluorodibenzophosphole oxide **2e** was also formed in excellent yield, up to 97% (entry 5). Furthermore, two kinds of ester- and nitrile-substituted products **2f** and **2g** were first synthesized in very good yields under our conditions which cannot be prepared directly using the most

Table 2. Syntheis of dibenzophosphole oxides **2**.[a]

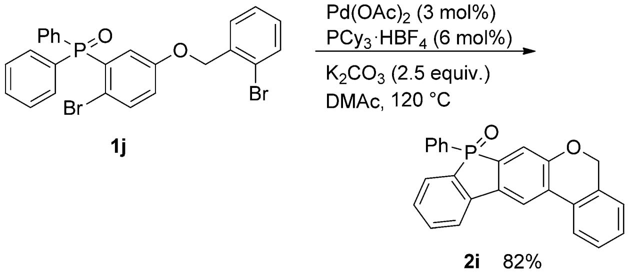


Entry	Boronic acid	Product	Yield [%]
1	 **1a**	 **2a**	98
2	 **1b**	 **2b**	85
3	 **1c**	 **2c**	96
4	 **1d**	 **2d**	94
5	 **1e**	 **2e**	97
6	 **1f**	 **2f**	90
7	 **1g**	 **2g**	83
8	 **1h**	 **2h**	94
9	 **1i**	 **2a**	97

[a] Unless otherwise specified, the reaction was carried out using **1** (1.0 mmol), K_2CO_3 (1.5 mmol), $Pd(OAc)_2$ (3 mol%), and $PCy_3{\cdot}HBF_4$ (6 mol%) in DMAc (3 mL) at 120°C for 12 h under N_2.

common organolithium method due to their sensitivities to electrophilic attack (entries 6 and 7). On changing the phenyl to an isopropyl group on the phosphorus atom, the reaction proceeded smoothly to produce the corresponding product **2h** in 94% yield (entry 5).

Taking into account of the wide generality of the direct arylation coupling method as for *ortho*-bromodiarylphosphine oxides, we turned our attention to the more challenging chloro-substituted substrates. As demonstrated by entry 9, the desired product **2a** was formed in high yield up to 97% from *ortho*-chlorodiarylphosphine oxide **1i**. Such a result constitutes one of the rare examples in the direct arylation of chloroarenes in very high yields.[16] Through a rational design of the substrate structure, this method could be applied to the synthesis of polycyclic dibenzophosphole oxides in the tandem reaction. Polycyclic phosphole oxide **2i** was prepared in 82% yield *via* double direct arylation reactions and the use of two distinct arylation couplings endowed a high degree of modularity to this strategy (Scheme 2). The structure of new compound **2i** was determined by X-ray analysis, which confirmed that regioselective direct arylation procceeded preferentially at the most sterically accessible site to afford **2i** (Figure 1).[17]



Scheme 2. Synthesis of **2i** by double direct arylation.

Encouraged by the facile synthesis of functionalized dibenzophosphole oxides through a second intramolecular biaryl coupling reacion, we then investigated the viability of one-pot sequential coupling reactions using arylboronic acids and heteroarenes as the second coupling partners respectively. In 2004, Xiao reported the Suzuki coupling reactions of **1a** with a variety of arylboronic acids for the synthesis of biarylphosphine oxides.[18] When 2,5′-dibromo-substituted substrate **1k** was used, it is very important to control the reaction conditions to suppress the undesired coupling at 2-position with boronic acids (Table 3). It was not surprising to find that **2j** was only isolated in 49%

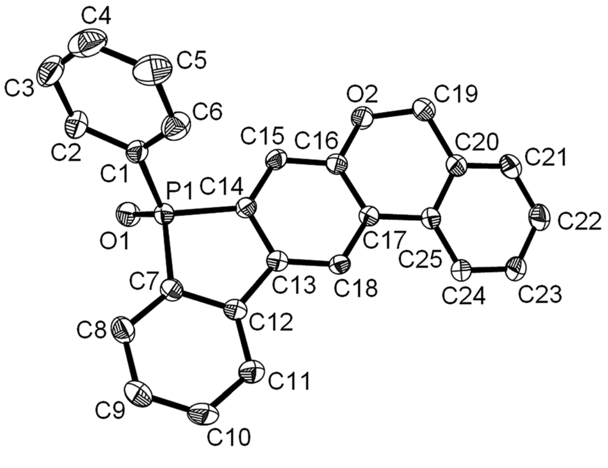
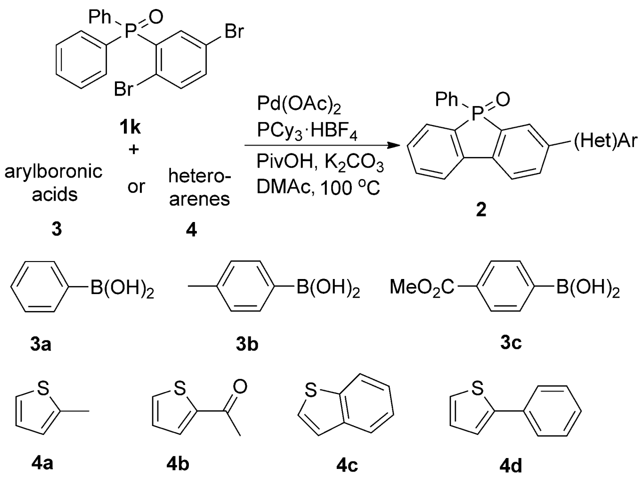
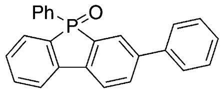
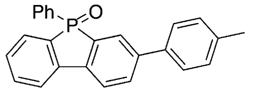
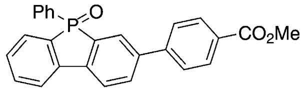
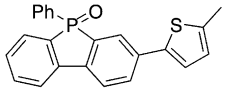
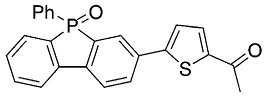
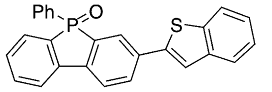
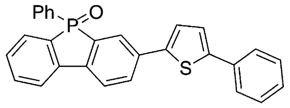


Figure 1. ORTEP representation of **2i**.

Table 3. Synthesis of 3-(het)aryldibenzophosphole oxides **2**.[a]



Entry	**3** or **4**	Product		Yield [%]
1	**3a**		**2j**	87; 49[b]
2	**3b**		**2k**	82
3	**3c**		**2l**	81
4	**4a**		**2m**	86
5	**4b**		**2n**	83
6	**4c**		**2o**	75
7	**4d**		**2p**	85

[a] *Reaction conditions:* For Suzuki coupling, **3** (1.1 mmol) in DMAc (2 mL) was added dropwise to a solution of **1k** (1.0 mmol), K_2CO_3 (2.5 mmol), PivOH (30 mol%), $Pd(OAc)_2$ (3 mol%), and $PCy_3{\cdot}HBF_4$ (6 mol%) in DMAc (2 mL) at 100 °C within 2 h and then stirred for 12 h under N_2 after addition. For direct heteroarylation, **4** (2 mmol), **1k** (1.0 mmol), K_2CO_3 (2.5 mmol), PivOH (30 mol%), $Pd(OAc)_2$ (3 mol%), and $PCy_3{\cdot}HBF_4$ (6 mol%) in DMAc (2 mL) were stirred at 100 °C for 20 h under N_2.

[b] **3a** was mixed with the other reagents.

yield under our standard conditions. To address the issue of competitive arylation resulting from the intra- and intermolecular coupling, the reaction was carried out in the presence of catalytic amounts of pivalic acid, which has been proved to promote direct arylation for a concerted metalation-deprotonation mechanism.[19] In addition, it was observed that slow addition of arylboronic acids is another critical factor to achieve satisfactory results. Finally, an efficient one-pot sequential tandem approach was successfully developed using arylboronic acids with different electronic properties. For example, the outcomes of the tandem coupling reaction were greatly improved to afford the desired product **2j** in 87% yield under our optimized conditions when phenylboronic acid was used as a coupling partner (entry 1). Analogously, **1k** also reacts smoothly with 4-methyl- and 4-methoxycarbonylphenylboronic acids and the corresponding products **2k** and **2l** were prepared in 82% and 81% yields, respectively (entries 2 and 3). Furthermore, we successfully synthesized a new family of thiophene-containing extended π-conjugated dibenzophosphole oxides through the sequential intra-/intermolecular direct arylation in a one-pot manner. This method is compatible with 2-methylthiophene (entry 4), 2-acetylthiophene (entry 5), benzothiophene (entry 6), and 2-phenylthiophene (entry 7) producing the corresponding products in good yields ranging from 75% to 86%.

In conclusion, we have developed a new method for the synthesis of dibenzophosphole oxides by palladium-catalyzed direct arylation reactions from the readily available *ortho*-halodiarylphosphine oxides. In addition, the utilities of this method were demonstrated by the facile access to extended π-conjugated systems of dibenzophosphole oxides combined with the second direct arylation or Suzuki–Miyaura coupling reacions in a tandem process. This strategy features simplicity, atom-economics, high yields, and good functional group compatibility, providing an efficient and versatile entrance to a variety of dibenzophosphole oxides applicable in materials. Studies on further applications of the reported method are currently underway.

Experimental Section

Representative Procedure for the Synthesis of Dibenzophosphole Oxide 2g

To a mixture of crushed K_2CO_3 (207 mg, 1.5 mmol), 3-bromo-4-(diphenylphosphinoyl)-benzonitrile (**1g**) (382 mg, 1.0 mmol), $Pd(OAc)_2$ (6.7 mg, 3 mol%), and $PCy_3{\cdot}HBF_4$ (22 mg, 6 mol%) in a 10-mL Schlenk tube equipped with a magnetic stir bar under a nitrogen atmosphere were added 3 mL of *N*,*N*-dimethylacetamide (DMAc). The contents of the Schlenk tube were stirred at 120 °C for 12 h. The reaction mixture was cooled to room temperature and quenched with water and then extracted with DCM. The extract was washed with brine and dried over Na_2SO_4. The solvent was then evaporated under vacuum and the residue was purified by using an SiO_2 column with ethyl acetate as eluent to afford the final product **2g**; yield: 250 mg, 83%; white solid; mp 269–271 °C. ^1H NMR (400 MHz, $CDCl_3$): $\delta=8.07$ (s, 1 H), 7.75–7.88 (m, 3 H), 7.59–7.70 (m, 4 H), 7.40–7.56 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta=142.5$ (d, $J=$ 10.9 Hz), 139.8 (d, $J=20.5$ Hz), 138.1 (d, $J=91.6$ Hz), 134.0 (d, $J=2.1$ Hz), 132.8 (d, $J=1.4$ Hz), 132.7 (d, $J=14.1$ Hz), 132.5 (d, $J=105.0$ Hz), 131.0 (d, $J=11.0$ Hz), 130.8 (d, $J=$ 11.3 Hz), 130.4, 130.4 (d, $J=18.5$ Hz), 129.3 (d, $J=$ 104.0 Hz), 129.0 (d, $J=12.8$ Hz), 124.5 (d, $J=9.9$ Hz), 121.7 (d, $J=10.0$ Hz), 117.9, 117.0 (d, $J=2.3$ Hz); ^{31}P NMR ($CDCl_3$): $\delta=33.4$; IR (film): $\nu=2229$, 1442, 1400, 1203, 1109, 839, 775, 752, 731, 692, 617, 601 cm^{-1}; HR-MS: $m/z=$ 302.0729, calcd. for $C_{19}H_{13}NOP$ $(M+H)^+$: 302.0729.

Crystallographic Data for 2i[17]

A single crystal of **2i** was obtained, grown by slow vapor diffusion of *n*-hexane into a solution of **2 i** in THF at room temperature. mp 210–212 °C.

Supporting Information

Experimental procedures, characherization and spectroscopic data are available in the Supporting Information.

Acknowledgements

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