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Insertion of Arynes into Arylphosphoryl Amide Bonds: One-Step Simultaneous Construction of C–N and C–P Bonds

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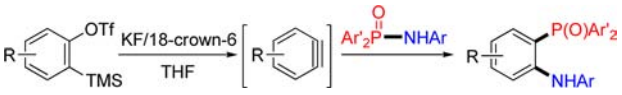
ABSTRACT



The insertion of arynes into arylphosphoryl amide bonds to synchronously construct C–P and C–N bonds is described. Arynes generated in situ from *o*-triflate arylsilanes under fluoride-promoted conditions insert into relatively inert P–N bonds, producing *o*-amine-substituted arylphosphine oxides. This process provides a simple pathway for the preparation of precursors for a number of bidentate aminophosphine ligands.

Arylphosphines and their derivatives are widely used in organic synthesis,[1] polymers,[2] and functional materials.[3] Particularly in organic synthesis, arylphosphines with bulky *ortho*-substituted groups such as Buchwald-type ligands play important roles in organometallic catalysis.[4] Their preparation usually requires transition-metal, moisture-sensitive reagents and oxygen-free conditions.[5] The transition-metal-catalyzed C–P cross-coupling reaction is one of the most important methods to prepare arylphosphines and their derivatives.[6] Because of its intrinsic properties, it is not applicable to the preparation of arylphosphines with bulky *ortho*-substituted functional groups. As a continuation of our research interests concerning the construction of sp^2 C–P bonds,[6b,d] we intended to develop a simple method to prepare arylphosphine oxides with bulky *ortho*-substituted groups.

As a useful and highly reactive intermediate in organic synthesis, the aryne group generated in situ from 2-(trimethylsilyl)aryl trifluoromethanesulfonate under fluoride-promoted conditions[7] has been used in numerous reactions for the construction of various bonds.[8] Due to its prominent character for the simultaneous construction of two different bonds in one reaction, insertion of arynes into certain bonds is

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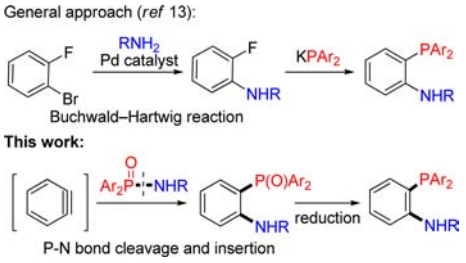
a powerful strategy that can be used to synthesize a number of different structures.[9] To the best of our knowledge, aryne insertion into P−N bonds remains unexplored. To date, there are only a handful of reported examples concerning the preparation of arylphosphines and aryl quaternary phosphonium salts via nucleophilic addition of phosphines on arynes.[10] Recently, the P-arylation of aryne with nucleophilic trialkyl phosphates was also reported.[11] These methods are not suitable for the synthesis of arylphosphines with bulky *ortho*-substituted functional groups. On the other hand, bidentate arylphosphine amine ligands have been widely applied in transition-metal-catalyzed reactions.[5f,12] Developing efficient pathways to prepare them has become a significant issue. Our study has forcused on the direct preparation of *o*-aniline-substituted arylphosphine oxides via the cleavage of an arylphosphoryl amide bond and aryne insertion (Scheme 1). The reaction involving the simultaneous construction of C−P and C−N bonds can produce various *o*-aniline-substituted arylphosphine oxides, some of which are versatile intermediates for the preparation of bidentate aminophosphine ligands.[13] The general approach to these bidentate aminophosphine ligands usually requires a transition metal, ligand, and anhydrous oxygen-free conditions.[13] To some extent, their preparation can be simplified by the methodology developed in this work.

Scheme 1. Insertion of Benzyne into the Arylphosphoryl Amide Bond



Our studies began with the reaction of *N*-phenyl diphenylphosphinic amide **2a** with benzyne generated in situ from 2-(trimethylsilyl)phenyl triflate **1a** under fluoride-promoted condititons. To identify the optimal conditions for the formation of the reaction product **3a**, we investigated the effect of fluoride salts, aprotic solvents, reaction temperature, and time on the reaction yield. Considering that the addition of base can accelerate the deprotonation of *N*-phenyl diphenylphosphinic amide **2a**, which would enhance the nucleophilicity of the arylphosphoryl amide, several kinds of bases were also employed as the reaction additive (detailed results in Table S1 of Supporing Information). Based on these results, we found that the use of 2.0 equiv of KF along with 2.0 equiv of 18-crown-6 and 2.0 equiv of Cs_2CO_3 in THF provided the optimal conditions for this reaction (Scheme 2). The reaction time is 8 h and reaction temperature is 80 °C. The desired product was obtained in 44% yield, with the remainder of the recovered material being unreacted **2a**. It was suggested that the low yield resulted from some side reactions that consumed very reactive benzyne.

Scheme 2. Reaction of *N*-Phenyl Diphenylphosphinic Amide **2a** with Benzyne

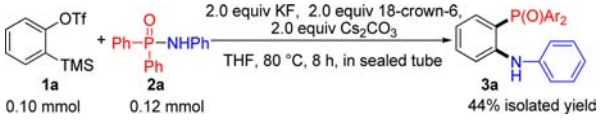


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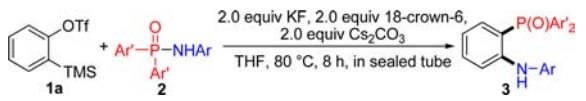
Table 1. Scope of Diphenylphosphinic Amide Substrates[a]



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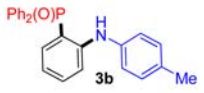
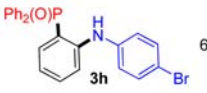
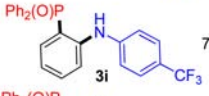
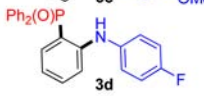
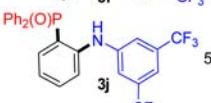
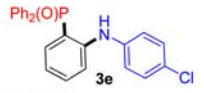
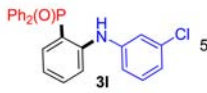
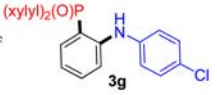
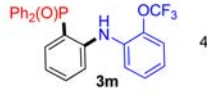
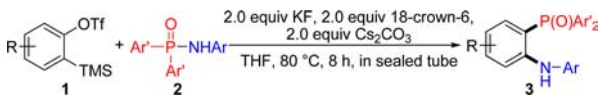
1	 **3b**	47%	7	 **3h**	63%
2	 **3c**	42%	8	 **3i**	72%
3	 **3d**	69%	9	 **3j**	56%
4	 **3e**	71%	10	 **3k**	53%
5	 **3f**	70%	11	 **3l**	55%
6[c]	 **3g**	68%	12	 **3m**	40%

[a] Unless otherwise stated, the reaction was conducted on a 0.10 mmol scale (0.10 mmol of **1a**, 0.12 mmol of **2**, 0.20 mmol of KF, 0.20 mmol of 18-crown-6 and 0.20 mmol of Cs_2CO_3) with 3.0 mL of THF in a sealed tube with a Teflon plug valve. Reaction time was 8 h. [b] Isolated yield. [c] Xylyl = 3,5-dimethylphenyl.

Table 2. Scope of 2-(Trimethylsilyl)aryl Triflate Substrates[a]



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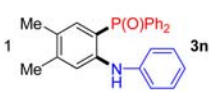
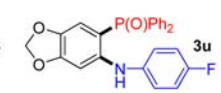
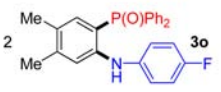
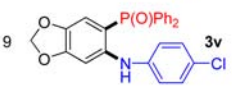
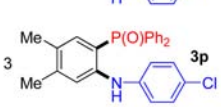
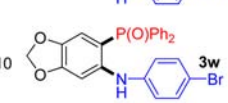
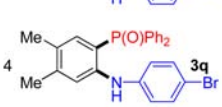
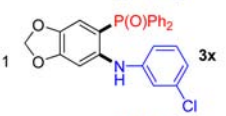
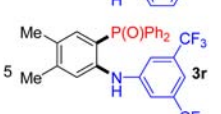
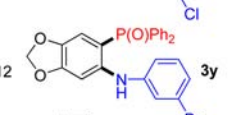
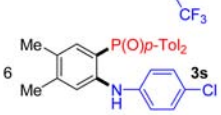
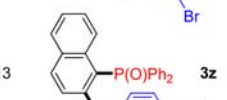
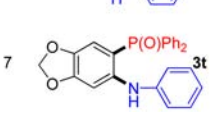
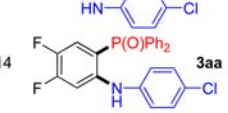
1	 **3n**	42%	8	 **3u**	59%
2	 **3o**	60%	9	 **3v**	62%
3	 **3p**	64%	10	 **3w**	56%
4	 **3q**	57%	11	 **3x**	46%
5	 **3r**	55%	12	 **3y**	45%
6	 **3s**	67%	13	 **3z**	NR
7	 **3t**	39%	14	 **3aa**	ND

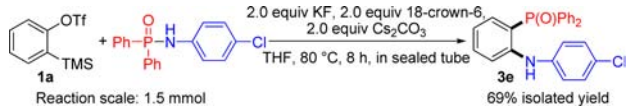
[a] Unless otherwise stated, reaction was conducted on a 0.10 mmol scale (0.10 mmol of **1**, 0.12 mmol of **2**, 0.20 mmol of KF, 0.20 mmol of 18-crown-6, and 0.20 mmol of Cs_2CO_3) with 3.0 mL of THF in a sealed tube with a Teflon plug valve. Reaction time was 8 h. [b] Isolated yield. NR = no reaction, no consumption of *N*-(4-chlorophenyl)diphenylphosphinic amide. ND = trace, not detected.

The scope of the reaction was next investigated by subjecting various *N*-aryl diphenylphosphinic amides to the optimized reaction conditions (Table 1). Compared with the result obtained for the reaction between **1a** and **2a** (Scheme 1), reactions of *N*-aryl diphenylphosphinic amides with *para*-substituted electron-netrual or electron-rich groups showed similar results with respect to yield (Table 1, entries 1 and 2). However, when the *para*-substituted group on the aryl ring was switched to an electron-deficient group such as fluoride, chloride, or trifluoromethyl substituent, a significant increase in yield occurred (entries 3–8). This may be attributed to the enhanced acidity of the *N*-aryl diphenylphosphinic amide.[9m] The introduction of electron-deficient groups at the aryl ring's *meta*-position also promoted an increase in yield (entries 9–11). The *ortho*-substituted electron-withdrawing group influences the yield of product via steric hinrance (entry 12) because it hinders the nucleophilic attack of the amide to the benzyne.[9g,m] Using the same protocol, we also examined the reaction between 2-(trimethylsilyl)phenyl triflate and *N*-butyl diphenylphosphinic amide. Only a trace amount of product was observed.

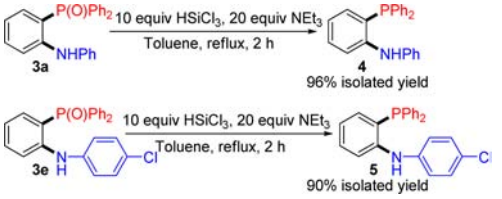
After we discovered that *N*-aryl diphenylphosphinic amides with electron-deficient groups provided better results for the reaction, we further explored the scope of 2-(trimethylsilyl)aryl triflate substrates with *N*-aryl diphenylphosphinic amides possessing *para*- or *meta*-substituted electron-deficient groups (Table 2). Yield obtained from the reaction of the symmetrically electron-neutral substituted aryne precursor, 4,5-dimethyl-2-(trimethylsilyl)-phenyl triflate, with *N*-aryl diphenylphosphinic amides and *N*-aryl di(*p*-tolyl)phosphinic amide with *para*- or *meta*-substituted electron-deficient groups (entries 2–6) gave higher yields than reactions involoving simple *N*-phenyl diphenylphosphinic amide (entry 1). For the symmetrically substituted electron-deficient aryne precursor, 6-(trimethylsilyl)benzo[*d*][1,3]dioxol-5-yl triflate, a similar phenomenon was also observed between the reaction with *N*-aryl diphenylphosphinic amides possessing *para*- or *meta*-substituted electron-deficient groups (entries 8–12) and the reaction with the simple *N*-phenyl diphenylphosphinic

(13) (a) Liang, L.-C.; Lee, W.-Y.; Hung, C.-H. *Inorg. Chem.* **2003**, *42*, 5471. (b) Liang, L.-C.; Huang, M.-H.; Hung, C.-H. *Inorg. Chem.* **2004**, *43*, 2166. (c) Liang, L.-C.; Chen, F.-Y.; Huang, M.-H.; Cheng, L.-C.; Lia, C.-W.; Lee, H. M. *Dalton Trans.* **2010**, *39*, 9941. (d) Eggenstein, M.; Thomas, A.; Theuerkauf, J.; Franciò, G.; Leitner, W. *Adv. Synth. Catal.* **2009**, *351*, 725. (e) Lindner, R.; van den Bosch, B.; Lutz, M.; Reek, J. N. H.; van der Vlugt, J. I. *Organometallics* **2011**, *30*, 499.

Scheme 3. Scale-up of the Reaction



Scheme 4. Derivation of Reaction Products



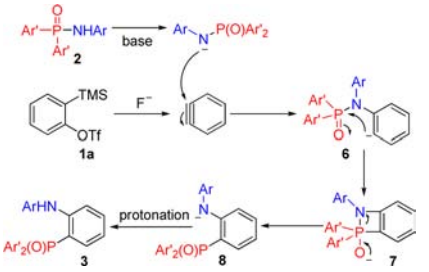
amide (entry 7). For the unsymmetrically substituted aryne precursor, 1-(trimethylsilyl)naphthalen-2-yl triflate, the desired product **3z** was not obtained (entry 13). This may be due to the steric hindrance of the bulky diphenylphosphinic group. Although both of the substrates were completely consumed in the reaction between 4,5-difluoro-2-(trimethylsilyl)phenyl triflate and substrate *N*-(4-chlorophenyl) diphenylphosphinic amide, no desired product **3aa** was detected (entry 14). We surmised that this outcome was related to the reactivity of the highly electron-deficient 4,5-difluorobenzyne which probably triggered some undesired side reactions.

The scale-up of the reaction between aryne precusor **1a** and *N*-(4-chlorophenyl)diphenylphosphinic amide was also attempted. When we increased the scale of the reaction from 0.10 to 1.5 mmol, the yield only slightly decreased (Scheme 3). Arylphosphine **4** and **5** could be easily obtained in high yield via the reduction of product **3a** and **3e**, respectively (Scheme 4). The compound **4** is a bidentate aminophosphine ligand[13a−c] and the intermediate of some versatile chiral ligands.[13d]

Based on previous reports,[9g,j,k,m] we propose the following mechanism for the formation of **3** (Scheme 5), The aryl

(14) Slana, G. B. C. A.; de Azevedo, M. S.; Lopes, R. S. C.; Lopes, C. C.; Cardoso, J. N. *Beilstein J. Org. Chem.* **2006**, *2*, 1.

Scheme 5. Proposed Mechanism for the Reaction



anion intermediate **6**, formed after *N*-arylation of the arylphosphoryl amide, undergoes a formal anionic Fries rearrangement[14] to the anion **8** via a four-membered ring intermediate **7**. The product **3a** is then formed by the protonation of **8**. The low/moderate yield of this reaction is probably due to the steric hindrance during the formation of **7** from **6**.

In summary, a simple and easily operated method to directly synthesize *ortho* aniline-substituted arylphosphine oxides has been developed via insertion of in situ generated arynes into the P−N bonds of arylphosphoryl amides. The product can be converted to *ortho*-amine-substituted arylphosphines which provide a new approach to accessing arylphosphines with a bulky *ortho*-substituted group.

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Supporting Information Available. General experimental procedure and characterization details. This material is available free of charge via the Internet at http://pubs.acs.org.

The authors declare no competing financial interest.