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An Approach to P=N Bond Formation: Straightforward Synthesis of Arylurea-Derived Phosphazenes via Condensation of $Ph_3P=O$ with *N*-Monosubstituted Arylureas

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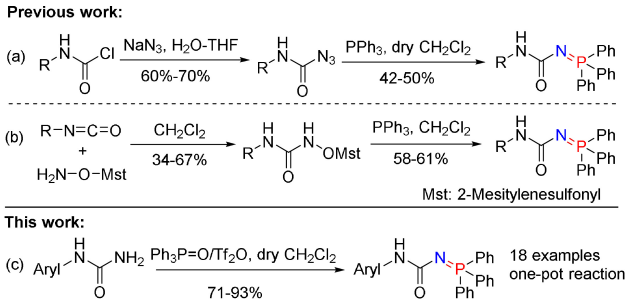
The Hendrickson's reagent-mediated formation of the P=N bond provides access to arylurea-derived phosphazenes compounds using readily available *N*-monosubstituted arylureas and $Ph_3P=O$. Various functional groups were tolerated to give arylurea-derived phosphazenes in the yield of 71–93%. The reaction also provides an alternative strategy for constructing compounds containing P=N bond. In this transformation, the Hendrickson's reagent unprecedented selectively reacts with the free amine group instead of the oxygen atom of *N*-monosubstituted arylurea. In addition, we found that thioureas could act as an effective thionating agent by converting $Ph_3P=O$ to $Ph_3P=S$.

Introduction

Compounds containing P=N bond are known as phosphazenes and have extensive and important applications in the pharmaceutical and biochemical industries.[1] Normally, phosphazenes are versatile synthetic intermediates, which can be used for reactions with carbonyl compounds (aza-Wittig reaction),[2] carboxylic acids,[3] acyl halides,[4] nitro compounds,[5] and water.[6] Furthermore, the Staudinger reaction has recently attracted renewed interest as a highly efficient bioorthogonal reaction.[7] In recent years, iminophosphoranes (IPs) have been extensively studied as protecting/directing groups for metal-mediated C–H bond activation reaction, and their synthetic applications have been extensively investigated.[8]

Two general preparative methods to access phosphazenes include the Staudinger reaction[9] and the Kirsanov reaction.[10] More recently, Yavari,[11] Norris,[12] Sundberg,[13] Onys' ko,[14] Amstrong,[15] Cristau,[16] and Morita[17] have developed methods for phosphazene preparation. Although these methods have been extensively studied, access towards arylurea-derived phosphazenes remains a challenging endeavor. Notably, few synthetic methods targeting $Ph_3P=NC(O)NH$-derivatives have been developed (Scheme 1).[8d,18–19] Current approaches require explosive organic azides (Scheme 1, a).[20] Moreover, all of these approaches involve two-step or multiple-step synthetic routes;



Scheme 1. Synthetic approach to arylurea-derived phosphazenes derivatives.

therefore, resulting in low atom economy, high cost, tedious work-up and environmental pollution (Scheme 1, a and b). In this regard, the development of a rapid and straightforward strategy to build arylurea-derived phosphazenes is highly meaningful and desirable.

$Ph_3P=O$ is a stable chemical substance owing to thermodynamically highly stable P=O bond;[21] therefore, a significant amount of $Ph_3P=O$ tends to be stored as industrial waste.[22] It is both important and challenging to develop a new methodology to enable facile recycling of $Ph_3P=O$ to functionalized molecules. As a consequence, efforts have been dedicated towards method development utilizing $Ph_3P=O$, such as the reduction to Ph_3P,[23] conversion into a dielectric material as flame retardant.[24] Despite these impressive advances, the use of $Ph_3P=O$ as a precursor via P=O cleavage remains a specific interest. Consequently, the direct conversion of $Ph_3P=O$ into useful scaffolds via easy operation has garnered increased interest in our research group.

Prepared in situ from $Ph_3P=O$ and trifluoromethanesulfonic anhydride (Tf_2O), Hendrickson's reagent was developed as a potent oxophile reagent for reactions involving net loss of water such as alcohol and aldoxime dehydration and carboxylic

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acid activation towards nucleophilic attack.[24] Recently, the synthetic utility of the Hendrickson's reagent as an effective amide activator has been extensively studied and its many proven applications are well documented.[25] Based on the mechanism previously described, the attack of the phosphorus atom in Hendrickson's reagent can occur from the inert amide oxygen.[26] Previously, Hendrickson disclosed the use of Hendrickson's reagent for the direct formation of phosphinimines with NH_3.[27] Based on the azophilicity and the Lewis acidity of the phosphonium salts of Hendrickson's reagent, we envisioned that anilines, arylureas, and benzamides could function as substrates to achieve similar transformations. To test this hypothesis, a number of commercially available or easily prepared anilines, *N*-monosubstituted arylureas and benzamides were evaluated. As a result, we found that anilines and benzamides did not react as predicted; however, *N*-monosubstituted phenylureas, which are less nucleophilic than anilines, unprecedentedly produced PhNHC(O)N=PPh_3 (**3 a**), whose structure was confirmed by single-crystal X-ray diffraction (Figure 1). Herein, we report an efficient and simple one-pot

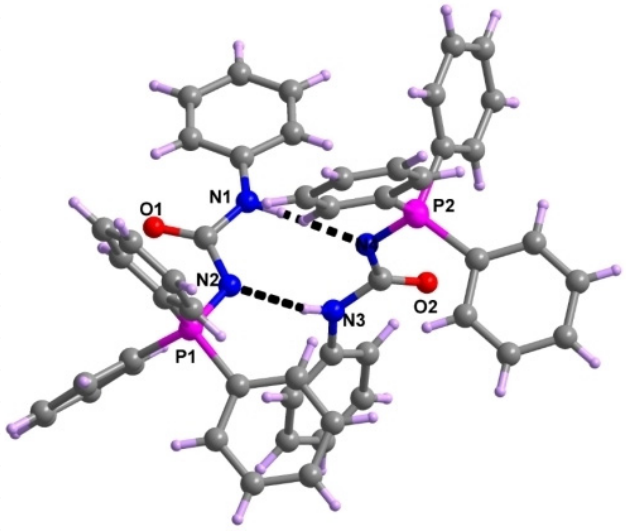


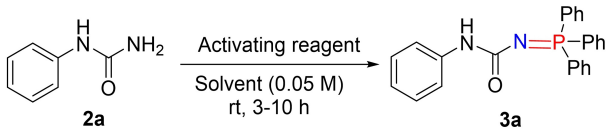
Figure 1. Structure of product **3 a**.

reaction for the preparation of ArNHC(O)N=PPh_3 with *N*-monosubstituted urea (Scheme 1, c).

Results and Discussion

Our investigations were initiated with the reaction of *N*-monosubstituted phenylurea (**1 a**) and Hendrickson's reagent according to Kelly's method[26] for reaction condition optimization (Table 1). First, the reaction was performed in dry CH_2Cl_2 for 0.5 h at 0 °C and quenched with 10% aqueous $NaHCO_3$ solution, affording **3 a** in 69% yield (Table 1, Entry 1). Notably, if the reaction was directly concentrated in vacuo instead of quenching the reaction with aqueous $NaHCO_3$ solution, only an 18% yield was obtained (Table 1, Entry 2), resulting from decomposition of **3 a** from excess Hendrickson's reagent. We

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



2a **3a**

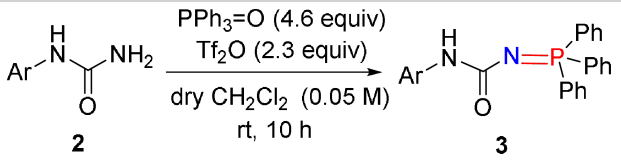
Entry	Tf_2O (equiv)	$Ph_3P=O$ (eqviv)	Solvent	*T.* [°C]	Time [h]	**3a** [%][b]
1	1.5	3	CH_2Cl_2	0	0.5	69
2[c]	1.5	3	CH_2Cl_2	0	0.5	18
3	1.5	–	CH_2Cl_2	0	3	0
4	–	3	CH_2Cl_2	0	3	0
5	1.5	3	Et_2O	0	3	52
6	1.5	3	THF	0	3	NR
7	1.5	3	CH_3CN	0	3	45
8	1	2	CH_2Cl_2	0	3	46
9	1.75	3.5	CH_2Cl_2	0	3	72
10[d]	1.75	3.5	CH_2Cl_2	0	3	43
11[e]	1.75	3.5	CH_2Cl_2	rt	3	45
12	2	4	CH_2Cl_2	rt	10	83
13	2.3	4.6	CH_2Cl_2	rt	10	88
14	3	6	CH_2Cl_2	rt	10	88
15	1.5	TPP (3)	CH_2Cl_2	0	3	32
16	-	TPP (3)	CH_2Cl_2	0	3	NR

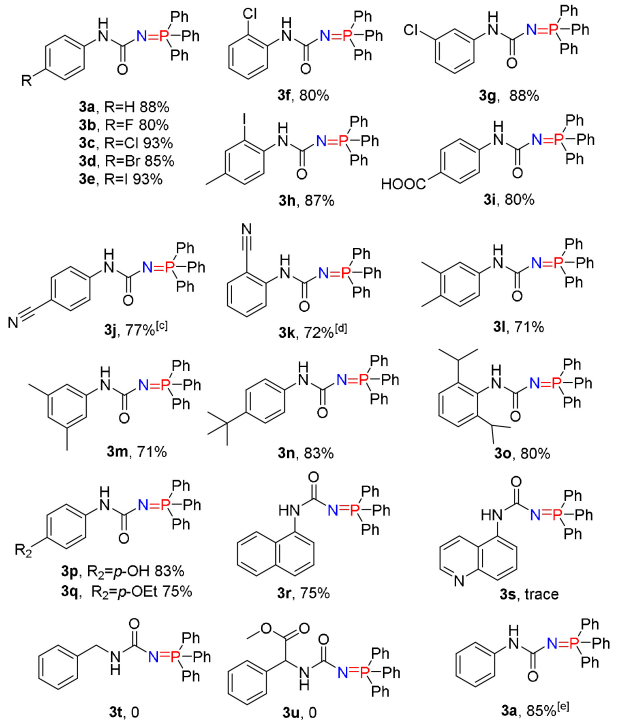
[a] Reaction conditions: Tf_2O (50 µL, 0.3 mmol) was added slowly to a solution of $Ph_3P=O$ (167 mg, 0.6 mmol) in dry CH_2Cl_2 (4 mL) at 0 °C under nitrogen atmosphere. The reaction mixture was stirred for 10 min at 0 °C followed by addition of the *N*-monosubstituted urea (0.2 mmol) under nitrogen atmosphere. The reaction progress was monitored by TLC. [b] Yield of isolated product. [c] Reaction complex was directly concentrated in vacuo after the reaction. [d] Reaction was conducted under an air atmosphere. [e] Reaction was conducted at room temperature.

then investigated the effects of $Ph_3P=O$ and Tf_2O and observed no reaction in the absence of $Ph_3P=O$ or Tf_2O (Table 1, Entries 3–4). Furthermore, screening of the reaction solvent revealed that dry CH_2Cl_2 was optimal compared to other solvents such as Et_2O, THF and CH_3CN (Table 1, Entry 1 vs. Entries 5–7). Decreasing Hendrickson's reagent loading reduced the yield to 46%; however, increasing the loading to 1.75 equiv. resulted in the formation of **3 a** in 72% yield (Table 1, Entries 8–9). Lower yields of 43% or 45% yield were obtained in an air atmosphere or at room temperature respectively, indicating that both an inert atmosphere and low temperature were necessary for the transformation (Table 1, Entries 10–11). Further increasing Hendrickson's reagent loading from 2 equiv. to 2.3 equiv. and prolonging the reaction time from 3 h to 10 h increased the yield from 83% to 88% respectively (Table 1, Entries 12–13). However, when 3 equiv. of Hendrickson's reagent was employed, no further yield improvement was observed (Table 1, Entry 14). More importantly, when Ph_3P was used instead of $Ph_3P=O$, 32% yield of **3 a** was obtained, suggesting that Ph_3P and Tf_2O form a reagent in situ similar to Hendrickson's reagent (Table 1, Entry 15).[25b] Moreover, when only Ph_3P, was used, no reactivity was observed (Table 1, Entry 16). Entry 13 indicates the optimized reaction conditions for further exploration.

Using the optimized reaction conditions, we subsequently investigated the substrate scope. Table 2 demonstrates that most of the *N*-monosubstituted arylureas examined provided

Table 2. Substrate scope.[a] [b]



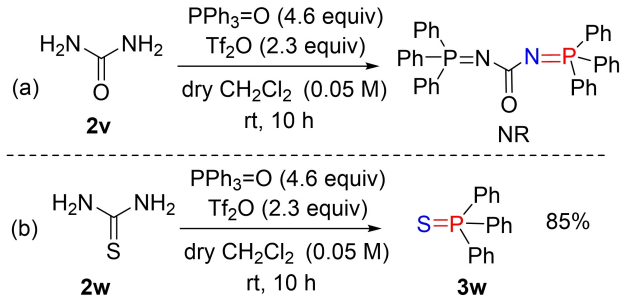


[a] Reaction conditions: Tf_2O (77 µL, 0.46 mmol) was added slowly to a solution of $Ph_3P{=}O$ (255 mg, 0.92 mmol) in dry CH_2Cl_2 (4 mL) at 0 °C under nitrogen atmosphere. The reaction mixture was stirred for 10 min at 0 °C followed by addition of the *N*-monosubstituted urea (0.2 mmol) under nitrogen atmosphere. The reaction progress was monitored by TLC. [b] Isolated yield. [c] 4-ureidobenzamide (**2j**) was used as the starting reagent. [d] 2-ureidobenzamide (**2k**) was used as the starting reagent. [e] 2.5 mmol scale.

good to excellent yields. The reaction was tolerant towards both electron-withdrawing and electron-donating substituents onto the aryl moiety of the *N*-monosubstituted arylurea. *N*-monosubstituted arylurea bearing electron-withdrawing halogens

(F, Cl, Br, I) in the *para*, *meta* or *ortho* position of the aryl afforded **3b**-**3h** in good to excellent yields (80-93%). Next, the scope of other electron-withdrawing groups (COOH, $CONH_2$) was investigated. Different *N*-monosubstituted arylurea **2i**-**2k** reacted smoothly to provide access to structurally diverse $ArNHC(O)N{=}PPh_3$ products in good to high yields; however, the amide substituent underwent dehydration to form cyanide groups.[25b] In the case of electron-donating groups (Me, i-Pr, tert-butyl, OH, OEt), the corresponding products **3l**-**3q** were generated in 71–83% yields. Even the sterically hindered substrate **2o** yielded the corresponding products in 80% yield. Additionally, fused rings such as **2r** were also efficient, providing the desired product **3r** in 75% yield. Notably, we failed to form the desired products **3s**-**3u** in the case of *N*-monosubstituted heteroaromatic ureas or *N*-monosubstituted alkylureas. Finally, to illustrate the synthetic potential of this method, we synthesized **3a** on a 2.5 mmol scale in 85% isolated yield.

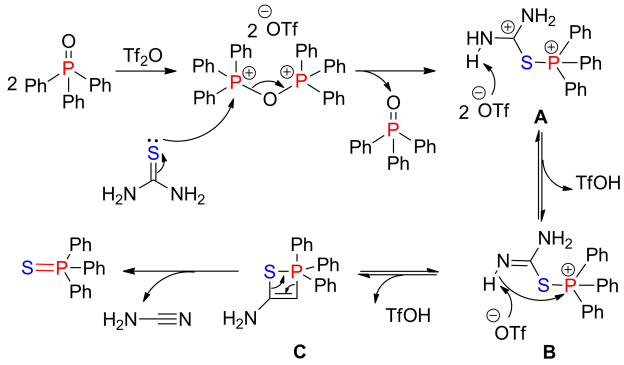
To investigate the reactivity of urea and thiourea in this reaction, two other experiments were conducted (Scheme 2).



Scheme 2. The reaction of urea or thiourea with Hendrickson's reagent.

When the reaction was performed with urea (**2v**) under standard reaction conditions, no product was detected (Scheme 2, a). To our surprise, $Ph_3P{=}S$ (**3w**) could be directly obtained in 85% yield from thiourea (**2w**) (Scheme 2, b). Although several thionating agents for the preparation of $Ph_3P{=}S$ from $Ph_3P{=}O$ such as silver sulfide,[28a] phosphorus pentasulfide,[28a] phosphorothioic trichloride[28a] and other special thionating agents[28b] have been reported, thioureas are nontoxic, easily available and environmental friendly thionating reagents for the challenging thionation of $Ph_3P{=}O$. Further, this reaction provides another synthetic way to recycle phosphine resources.

Scheme 3 illustrates a plausible mechanism for the reaction of thiourea with Hendrickson's reagent. Initially, the lone pair

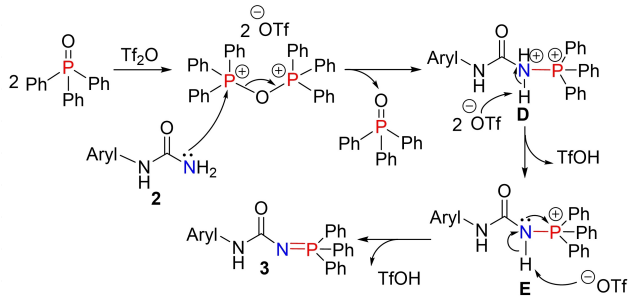


Scheme 3. Plausible reaction mechanism of thiourea with Hendrickson's reagent.

electrons on the thiourea attack the positively charged phosphorus atom in Hendrickson's reagent generated in situ

from $Ph_3P{=}O$ and Tf_2O.[25] Meanwhile, one molecule of $Ph_3P{=}O$ is removed to form intermediate **A**. Deprotonation of intermediate **A** by TfO^- generates intermediate **B**. Then, deprotonation of intermediate **B** generates four- intermediate **C** with a four-membered ring, which is a Wittig-type intermediate. Elimination of cyanamide from **C** gives the desired $Ph_3P{=}S$ (**3 w**).

Scheme 4 illustrates a plausible mechanism for this transformation of $Ph_3P{=}O$ with *N*-Monosubstituted Arylurea. Initially,



Scheme 4. Plausible reaction mechanism *N*-monosubstituted arylurea with Hendrickson's reagent.

the lone pair electrons from the free amino group of *N*-monosubstituted arylurea **2** attack the positively charged phosphorus atom in Hendrickson's reagent. Meanwhile, one molecule of $Ph_3P{=}O$ is removed to form intermediate **D**. Deprotonation of intermediate **D** by TfO^- generates intermediate **E**. Secondly, deprotonation of intermediate **E** generates product **3**. It is well-known that the Hendrickson's reagent can activate amides via nucleophilic substitution reaction with the oxygen of amide. The amides can then reacts with other nucleophilic groups.[26] Notably, Hendrickson's reagent selectively reacts with the free amine group of *N*-monosubstituted arylurea instead of the oxygen of *N*-monosubstituted arylurea, due to the stronger nucleophilicity of the free amino groups on *N*-monosubstituted arylurea compared to the oxygen or nitrogen atoms of the amide or the oxygen atom of the *N*-monosubstituted arylurea. To the best of our knowledge, such selectivity is unique. In addition, it is possible that the products obtained from *N*-monosubstituted alkylurea or aniline decomposed under the reaction conditions. Further studies to expand the application of Hendrickson's reagent-mediated formation of the P=N bond reaction are underway.

Conclusions

In conclusion, we have developed an efficient way to construct the N=P bond, which enables the condensation of *N*-monosubstituted arylureas with $Ph_3P{=}O$ to afford $ArNHC(O)N{=}PPh_3$. Key reaction features include readily available and safer starting materials, easy operational procedure, good to excellent yields, and broad substrate scopes. This is a useful strategy for treatment of the useless $Ph_3P{=}O$. Further, readily available reagents ($Tf_2O/Ph_3P{=}O$) act as an effective promoters for the direct preparation $R{-}N{=}PPh_3$ and $AlkylNHC(O)N{=}PPh_3$ are being conducted in our laboratory.

Supporting Information Summary

Experimental details and characterization data of all compounds.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords: Hendrickson's reagent · *N*-monosubstituted arylurea · P=N Bond · Phosphazene · Triphenylphosphine oxide

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