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Hypervalent Iodine(III) in Direct Oxidative Amination of Arenes with Heteroaromatic Amines

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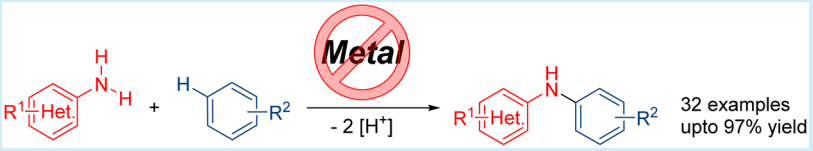
Supporting Information



ABSTRACT: A novel, mild, and practical method of amination of simple nonfunctionalized arenes under metal free conditions has been developed. The approach allows coupling of electron-rich arenes with amino derivatives of electron-deficient heterocycles providing rapid access to scaffolds of bioactive compounds and is based on the application of the hypervalent iodine(III) reagent as an oxidant. Regioselective functionalization of C−H bonds of arenes by the formation of C−N bonds under organocatalytic conditions was demonstrated.

The C−N bond forming reactions are pivotal transformations in the synthesis of various products of interest and have found broad applications.[1] Selective N-arylation of nitrogen-containing heterocycles (Figure 1) is of certain interest because the products represent important structural motifs of natural products, biologically active compounds, pharmaceutical agents, and materials.[2]

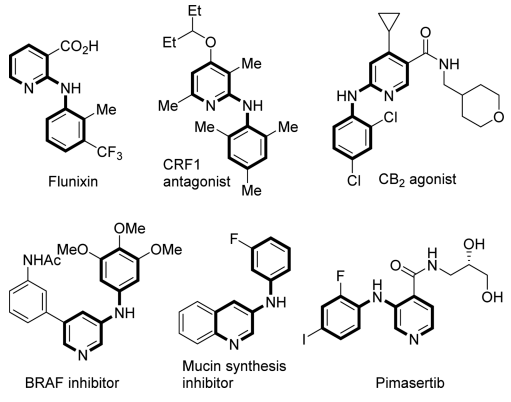
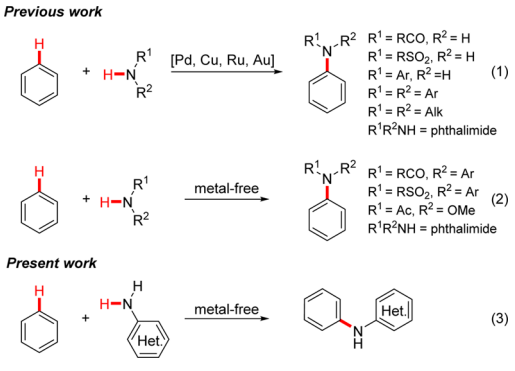


Figure 1. Selected bioactive N-arylated pyridines.

Despite the significant progress made in the development of C−N bond forming reactions using Ullmann reaction[3] and Buchwald-Hartwig amination,[4] there still exists a need for new methods that involve cheap, straightforward and environmentally friendly reaction conditions. Over the past decades, numbers of C−N bond forming methods have been developed employing transition metal catalysts for the N-arylation of nitrogen-containing heterocycles with prefunctionalized compounds such as aryl halides and arylboronic acids.[5] Recently, the intermolecular formation of C−N bonds by direct C−H bond functionalization of arenes using N−H bond containing reagents (cross-dehydrogenative couplings) was studied.[6] Transition metals found application as catalysts in the cross-dehydrogenative C−N bond formation with various arenes in the presence of external oxidants (Scheme 1, eq 1).[7] Recently, independently of our group, Chang's and DeBoef's groups reported a hypervalent iodine reagent mediated direct intermolecular oxidative amination (Scheme 1, eq 2).[8] Soon after, we developed the organocatalytic reaction conditions which give access to aminated benzenes at ambient temperature

Scheme 1. Cross-Dehydrogenative Amination of Arenes



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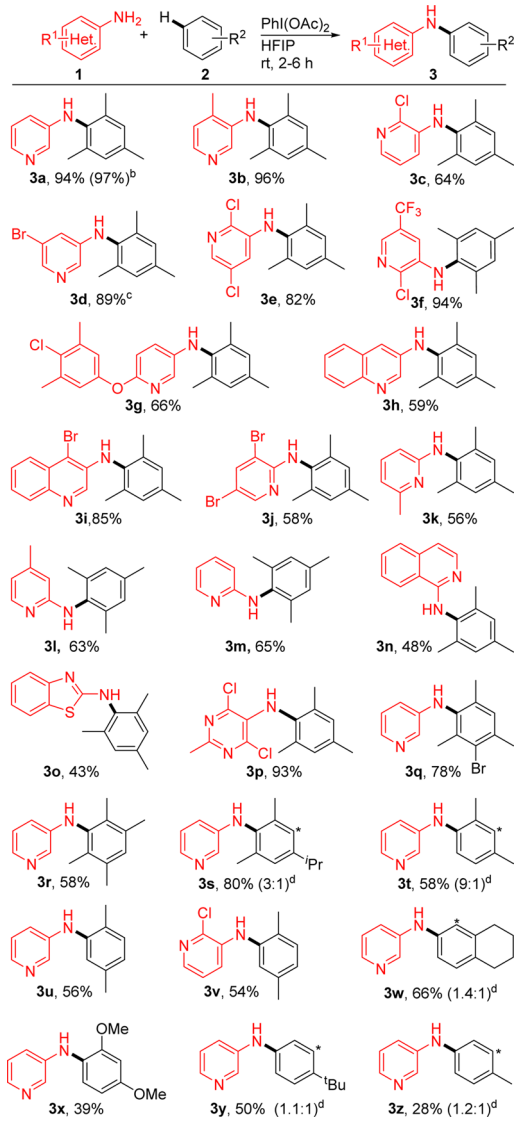
and mild reaction conditions.[9] Very recently, the oxygen-mediated dehydrogenative amination of phenols with phenoxazine and phenothiazine was developed by Patureau's group.[10] To the best of our knowledge, metal-free[11] approaches of C−H bond amination of nonfunctionalized arenes with aminoheterocycles for the synthesis of bioactive compounds scaffold have never been reported.[12]

Recently, we have developed a metal-free intermolecular amination of nonfunctionalized arenes with amine derivatives employing hypervalent iodine(III) reagents or organocatalysis.[8c,9] The amination of nonprefunctionalized arenes with amines has one limitation: a protected (disubstituted) amine is required to avoid the over oxidation and dimerization. Interested in the synthesis and modification of heterocycles,[13] we have been focusing on the application of hypervalent iodine(III) reagents[14] in the cross-dehydrogenative amination of arenes with aminoheterocycles. Herein, we report the first metal-free and organocatalytic approach of arene amination with aminoheterocycles (Scheme 1, eq 3).

We initiated our investigation by exploring the iodosobenzene diacetate mediated arylation of 3-aminopyridine **1a** which is a widely present and privileged scaffold in bioactive compounds. As second coupling partner we used an excess (10 equiv) of mesitylene **2a** due to its high reactivity in electrophilic aromatic substitution. Using 1.1 equiv of $PhI(OAc)_2$ at ambient temperature in CH_3CN, we isolated the desired product **3a** of direct C−H bond amination in 48% yield (see Supporting Information). Afterward, various solvents such as 2,2,2-trifluoroethanol (TFE), 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP), $MeNO_2$, $CHCl_3$, dichloromethane and DMF were tested, and the reaction showed strong solvent dependence (see Supporting Information). To our delight, the yield of target product was increased to 94% using HFIP as solvent. Various hypervalent iodine(III) reagents such as $PhI(OAc)_2$, $PhI(OCOCF_3)_2$, PhIO, and PhI(OH)OTs (Koser's reagent) were screened. Subsequently, we discovered that the application of $PhI(OAc)_2$ as oxidant provides the aminated product **3a** in highest yield at room temperature.

Having established the optimized reaction conditions, we next explored the substrate scope with various 3-aminopyridine derivatives with different functional groups in direct coupling with mesitylene **2a**. We were pleased to find that various amine derivatives could be transferred into the amination derivatives in moderate to good yields. Electron-withdrawing groups such as chloro-, bromo- and trifluoromethyl groups and electron-donating groups such as methyl and phenoxy groups in 3-aminopyridine were well tolerated under the metal free reaction conditions (**3b**−**3g**) (Scheme 2) and the corresponding products of direct amination were obtained with moderate to good yields. To our delight, disubstituted 3-aminopyridines were well tolerated and the desired products (**3e** and **3f**) were generated with good yield. It is notable that the application of 3-aminoquinolines did provide the desired products (**3h**, **3i**) with good yields. Encouraged by these results, we further proceeded to explore the amination reaction with various 2-aminopyridines, and interestingly, we found that various functionalized 2-aminopyridines can be obtained smoothly under the metal-free reaction conditions. Nevertheless, the yield of the arylated 2-aminopyridines (**3j**−**3m**) was lower compared to that of the 3-aminopyridine derivatives. It is noteworthy that 1-aminoisoquinoline was also a suitable substrate for the corresponding amination with good yield (**3n**). However, the application of 4-aminopyridine in direct

Scheme 2. Metal-Free Oxidative Amination[a]



[a]Reaction conditions: **1** (0.25 mmol), arene **2** (2.5 mmol, 10 equiv), (diacetoxy)iodobenzene (0.275 mmol) in HFIP (0.25 M). [b]HFIP and dichloromethane (1:1) were used. [c]Reaction carried out at 0 °C. [d]Major isomer is shown, minor is indicated with star, and the yields were reported for mixture of isomers.

arylation was not successful. The formation of a dimerization product of 4-aminopyridine was observed to be similar to the reported[15] dimerization products of aniline derivatives. The developed reaction conditions were suitable for the metal-free arylation of benzo[*d*]thiazol-2-amine with moderate yield (**3o**). Interestingly, a highly substituted pyrimidine could be efficiently converted into its corresponding aminated product **3p** in 93% yield.

Having success in direct arylation of various aminoheterocycles, we further explored the amination of various simple nonfunctionalized arenes under the developed reaction conditions. Gratifyingly, the presence of various functional groups at the arene allowed the formation of the corresponding desired products. Interestingly, multisubstituted arenes were successfully utilized in the synthesis of the desired products (**3q**−**3x**) with moderate to good yields. Those products were obtained regioselectively and the new C−N bonds were formed

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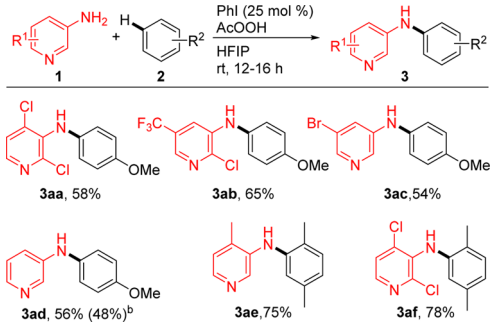
at the most nucleophilic and least sterically demanding positions. In general, the amination of more nucleophilic arenes provided the products in higher yields compared to less nucleophilic arenes. Nevertheless, the reaction with nucleophilic 1,3-dimethoxybenzene gave the target product **3x** in low yield due to side oxidation reaction of 1,3-dimethoxybenzene by iodosobenzene diacetate. Monosubstituted arenes were providing the amination products with lower yield and regioselectivity, due to the lower nucleophilicity of arenes (**3y**, **3z**). Unfortunately, the application of arenes with electron-withdrawing groups did not result in the desired product formation under the mild reaction conditions. Products of functionalization of electron rich heteroaromatics such as indole and benzofuran were not obtained due to their dearomatization in the presence of the iodine(III) reagent. A notable feature of the developed method is the sole formation of monoarylated aminoheterocycles.

After the development of the oxidative amination of heteroarylamines with arenes by employing a stoichiometric amount of iodine(III) reagent, we focused on the development of an organocatalytic amination. Interestingly, we observed that a wide array of 3-aminopyridine derivatives were well tolerated under organocatalytic conditions resulting in moderate to good yields using iodobenzene as catalyst in the presence of peracetic acid as oxidant. In general, various groups at 3-aminopyridine were tolerated and the desired products were formed smoothly (Scheme 3). Unfortunately, the organocatalytic conditions were

Scheme 3. Organocatalytic Oxidative Amination[a]

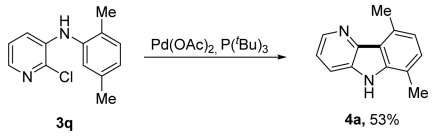


[a]Reaction conditions: **1** (0.25 mmol), arene **2** (2.5 mmol, 10 equiv), PhI (25 mol %), AcOOH (5 equiv, added portionwise in 6 h) in HFIP (0.25 M). [b]$PhI(OAc)_2$ (1.1 equiv) was used.

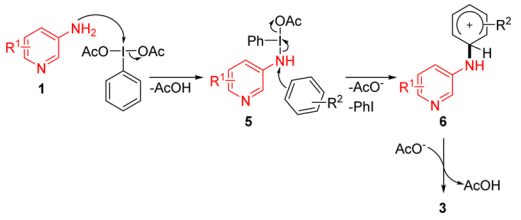
only suitable for arylation of electron-rich arenes, and the desired products were not obtained using electron deficient arenes and benzene. It is notable that selective functionalization of anisole at the para-position was observed under catalytic reaction conditions (**3aa**−**3ad**) and using a stoichiometric amount of iodosobenzene diacetate (**3ad**). However, using a stoichiometric amount of iodine(III) reduced the reagent yield of arylated product. We next turned our attention to the application of the heteroarylaminated products to the important carboline scaffold synthesis. Compound **4a** was obtained with 53% yield from **3q** employing a palladium catalyzed C−H activation (Scheme 4).

A plausible mechanism was described for the $PhI(OAc)_2$ mediated amination of arenes on the basis of our studies and previous reports[8c,9] (Scheme 5). In presence of $PhI(OAc)_2$, intermediate **5** was generated through ligand exchange of the hypervalent iodine reagent. Intermediate **5** underwent an electrophilic aromatic substitution via reductive elimination of

Scheme 4. Synthesis of Carbazole



Scheme 5. Plausible Mechanism



iodobenzene and an acetate anion, and formation of a nitrenium ion was stabilized by polar non-nucleophilic solvents such as HFIP which resulted in the formation of intermediate **6**. Subsequently, aromatization of intermediate **6** in the presence of the acetate anion led to the formation of the desired product **3**. We have elucidated the kinetic isotope effect (KIE) between **1a** and deuterated *p*-xylene with 3-aminopyridine under the optimized conditions and observed no kinetic isotope effect ($k_H/k_D \approx 1$). Therefore, the aromatization of intermediate **6** is not the rate-limiting step of the reaction.

In conclusion, we have developed a hypervalent iodine(III) mediated and organocatalytic amination of nitrogen-containing heterocycles and nonfunctionalized arenes under mild reaction conditions with broad scope. The presented method was employed in the coupling of simple readily available amino derivatives of electron-deficient heterocycles with electron-rich arenes via oxidative coupling by direct C−H bond functionalization. The described transformation is highly practical because diverse, readily available starting materials can be used for straightforward access to scaffolds of bioactive compounds.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b02320.

General information, optimization of reaction conditions, spectral data and copies of NMR spectra for compounds (PDF)

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Notes

The authors declare no competing financial interest.

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