

## *o*-Iodoxybenzoic Acid Mediated *N*-Arylation of Aromatic Amines by Using Arylhydrazines as the Arylating Counterpart

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Through free-radical trapping experiments we have established, for the first time, the combination of arylhydrazines with *o*-iodoxybenzoic acid (IBX) for the generation of aryl free radicals. On the basis of this finding, a method was developed for the *N*-arylation of aromatic amines under mild

conditions (base-free,  $-5\text{ }^{\circ}\text{C}$ ) by using arylhydrazines as the arylating counterpart and arylamines. The scope of this method was demonstrated by using a number of arylhydrazines and arylamines, which gave the *N*-arylated amines in good yields.

### Introduction

The *N*-arylation of amines is an active area of research in organic synthesis, because *N*-arylated compounds are used in the synthesis of numerous substances including natural products, agrochemicals, dyes, and pharmaceuticals.<sup>[1]</sup> In the literature, many methods for the *N*-arylation of amines are reported. For instance, the coupling of electrophilic aryl halides and nucleophilic primary or secondary amines by using Pd and Cu catalysts, pioneered by Buchwald, Hartwig, and others, is a hallmark reaction in this field.<sup>[2]</sup> Another method, independently developed by Chan, Evans, and Lam, based on the Cu-mediated oxidative amination of arylboronic acids with amines and other nucleophiles, provides a valuable alternative in the construction of carbon–heteroatom bonds.<sup>[3]</sup> The drawbacks of Pd- and Cu-catalyzed *N*-arylation reactions include the requirement of high temperatures, long reaction times, the need for ligands and bases, and the generation of corrosive halide ions in many cases. By utilizing diaryliodonium salts, Beringer<sup>[4]</sup> established yet another method for *N*-arylation; however, owing to difficulties in the synthesis of these salts, the applicability of this method is limited. In the presence of copper salts, phenyllead triacetate<sup>[5]</sup> and triphenylbismuth<sup>[6]</sup> have found use in the synthesis of arylamines, but these reagents suffer from nonreactivity towards substrates containing electron-withdrawing substituents, and they are not eco-friendly, and, therefore, they are less popular. Recently, Lutz and Thomson<sup>[7]</sup> reported a hypervalent iodine initiated fragment-coupling cascade reaction of *N*-allylhydrazones,

aldehydes, and alcohols leading to the formation of disubstituted alkenes with vicinal stereocenters. In light of all these points and the requirement for *N*-arylated amines, there is a scope for new methods. Our research group, attracted by the mild and chemoselective nature of hypervalent iodine(V) reagents,<sup>[8]</sup> is extensively exploring new applications of these reagents, and the present work, which offers a new method for the *N*-arylation of amines, is an outcome of one such exploration.

Aryldiazonium salts produce aryl free radicals, very reactive unstabilized  $\sigma$  radicals, upon heating or upon reaction with metals such as Al, Cu, Pd, Ag, Pt, Au, and so on.<sup>[9]</sup> Similarly, aryl free radicals can also be produced by using arylhydrazines in the presence of oxidizing agents.<sup>[10]</sup>

Hypervalent iodine(V) reagents, mainly *o*-iodoxybenzoic acid (IBX) and Dess–Martin periodinane (DMP), have become alternative oxidizing agents for various metal oxidants.<sup>[11]</sup> The versatility of these reagents has been firmly established by various transformations they bring about, such as oxidative C–C coupling, oxidative cyclization, oxidative rearrangements, oxidative deoxygenation, oxidative ring expansion and contraction, C–N bond formation, oxidation of alcohols, and others.<sup>[12]</sup> Because of their mild nature and their ability to be chemoselective, these reagents are preferred in the total synthesis of complex molecules, including natural products.<sup>[13]</sup>

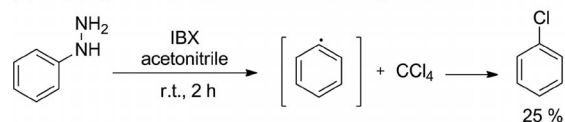
### Results and Discussion

It has been shown that IBX reactions proceed through a single-electron transfer (SET) mechanism.<sup>[14]</sup> IBX reacts violently with hydrazine and is reduced to *o*-iodobenzoic acid. We hypothesized that, on the same lines, IBX would oxidatively decompose phenylhydrazine to produce the phenyl radical by expelling the oxidized hydrazine group as nitrogen gas. To prove this hypothesis, we undertook two

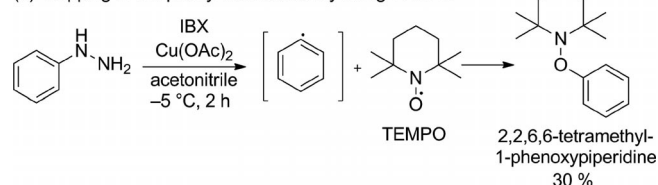
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free-radical trapping experiments and were successful. In the first experiment, phenylhydrazine was treated carefully at room temperature with IBX in acetonitrile in the presence of  $\text{CCl}_4$  as a radical trap. After workup, chlorobenzene was isolated in 25% yield (Scheme 1a). In the second experiment, performed under the same reaction conditions as those used in the first experiment, (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO), a compound already in the free-radical state, was used as the radical trapping agent in the presence of a catalytic amount of  $\text{Cu}(\text{OAc})_2$ ; the expected product, 1-[(2,2,6,6-tetramethylpiperidinyl)oxy]benzene, was formed in 30% yield (Scheme 1b).

(a) Trapping of the phenyl free radical by using  $\text{CCl}_4$ 

(b) Trapping of the phenyl free radical by using TEMPO



Scheme 1. Trapping of the phenyl free radical.

At that point, having established the generation of the free radical, experiments were carried out by adding phenylhydrazine to a mixture of IBX and an arylamine in the presence of a catalytic amount of  $\text{Cu}(\text{OAc})_2$ . It was anticipated that the generated phenyl free radical would be trapped by the amine to form an *N*-arylamine, and this would offer a new method for the *N*-arylation of amines. This, in fact, happened, and the details are discussed below.

Table 1. Optimization of reagents and reaction conditions.<sup>[a]</sup>

Entry	Reaction		Yield [%]			
	1a	2a	3a	4a		
1	IBX (1.5)	0.1	$\text{CH}_3\text{CN}$	-5	30	05
2	IBX (3)	0.1	$\text{CH}_3\text{CN}$	-5	78	10
3	IBX (3)	0.1	$\text{CH}_3\text{CN}$	r.t.	50	20
4	IBX (3)	0.1	$\text{CH}_3\text{CN}$	-20	-	25
5	IBX (3)	0.1	$\text{CH}_3\text{CN}$	-5	78 <sup>[c]</sup>	10 <sup>[c]</sup>
6	IBX (4)	0.1	$\text{CH}_3\text{CN}$	-5	78	10
7	IBX (3)	0	$\text{CH}_3\text{CN}$	-5	10	15
8	IBX (0)	3	$\text{CH}_3\text{CN}$	-5	20	10
9	IBX (3)	0.1	DMF	-5	60	15
10	DMP (3)	0.1	$\text{CH}_3\text{CN}$	-5	-	05
11	DIB (3)	0.1	$\text{CH}_3\text{CN}$	-5	-	20

[a] All reactions were carried out on a 5 mmol scale with aniline (1 equiv.) and phenylhydrazine (1.5 equiv.). [b] Yield of isolated product after chromatography (silica gel #60-120; ethyl acetate/hexane, 2:98); reaction time 3 h. [c] Reaction time 7 h.

To establish the reaction conditions, preliminary experiments were carried out by using aniline and phenylhydrazine as model substrates. Treatment of phenylhydrazine (**2a**, 1.5 equiv.) with IBX (3 equiv.) in the presence of aniline (**1a**, 1.0 equiv.) and  $\text{Cu}(\text{OAc})_2$  (0.1 equiv.) as the catalyst in ace-

Table 2. *N*-Arylation of amines by using phenylhydrazine (**2a**) as the arylating counterpart.

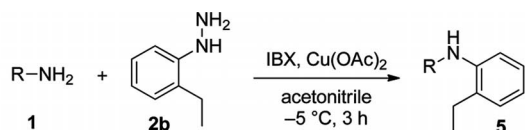
Entry	Reaction		Yield <sup>[b]</sup> [%]
	Substrate 1	Product 3	
1	<b>1a</b>	<b>3a</b>	78
2	<b>1b</b>	<b>3b</b>	76
3	<b>1c</b>	<b>3c</b>	74
4	<b>1d</b>	<b>3d</b>	75
5	<b>1e</b>	<b>3e</b>	73
6	<b>1f</b>	<b>3f</b>	71
7	<b>1g</b>	<b>3g</b>	76
8	<b>1h</b>	<b>3h</b>	72
9	<b>1i</b>	<b>3i</b>	73
10	<b>1j</b>	<b>3j</b>	67
11	<b>1k</b>	<b>3k</b>	65
12	<b>1l</b>	<b>3l</b>	63

[a] All reactions were carried out on a 5 mmol scale with the amine (1 equiv.), phenylhydrazine (1.5 equiv.), IBX (3 equiv.), and  $\text{Cu}(\text{OAc})_2$  (0.1 equiv.). [b] Yield of isolated product after chromatography (silica gel #60-120; ethyl acetate/hexane, 2:98).

tonitrile as the solvent at  $-5\text{ }^{\circ}\text{C}$  led to the formation of diphenylamine (**3a**) and azobenzene (**4a**) in 78 and 10% yield, respectively (Table 1, Entry 2). To study the effect of temperature, the reactions were repeated separately at room temperature and at  $-20\text{ }^{\circ}\text{C}$ . In both cases, the yields of the products were affected: a decrease in the yield of **3a** to 50% and an increase in the yield of **4a** to 20% was observed at room temperature (Table 1, Entry 3), whereas at the lower temperature of  $-20\text{ }^{\circ}\text{C}$ , **3a** was not formed, but **4a** was obtained in 25% yield (Table 1, Entry 4). There was no improvement in the yield with a prolonged reaction time (Table 1, Entry 5) or an increase in the amount of IBX to 4 equiv. (Table 1, Entry 6); however, a decrease in the amount of IBX to 1.5 equiv. resulted in a lower yield of

30% for **3a** (Table 1, Entry 1). To ascertain the role of  $\text{Cu}(\text{OAc})_2$ , a reaction was carried out in the absence of  $\text{Cu}(\text{OAc})_2$ , and **3a** was formed in a very low yield of 10% (Table 1, Entry 7). Copper salts are known to oxidize phenylhydrazine,<sup>[15]</sup> and therefore, a reaction was performed by using  $\text{Cu}(\text{OAc})_2$  (3.0 equiv.) as the sole oxidizing agent. Product **3a** was formed but only in a very low yield of 20% (Table 1, Entry 8). These results indicated that either IBX or  $\text{Cu}(\text{OAc})_2$  alone was not efficient for the reaction. DMF, screened as an alternative solvent, was found to be viable, but it delivered **3a** in an inferior yield of 60% (Table 1, Entry 9). Other hypervalent iodine reagents such as DMP and (diacetoxyiodo)benzene (DIB) were also studied under the optimized reaction conditions. In both cases, the formation of **3a** was not observed; instead, these reactions resulted in the formation of a complex mixture in which small amounts of **4a** were present (Table 1, Entries 10 and 11).

Table 3. *N*-Arylation of amines by using (2-ethylphenyl)hydrazine (**2b**) as the arylating counterpart.



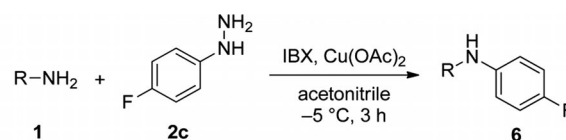
Entry	Substrate 1	Product 5	Yield <sup>[b]</sup> [%]
1			72
2			71
3			69
4			70
5			68
6			67
7			63
8			60

[a] All reactions were carried out on a 5 mmol scale with the amine (1 equiv.), (2-ethylphenyl)hydrazine (1.5 equiv.), IBX (3 equiv.), and  $\text{Cu}(\text{OAc})_2$  (0.1 equiv.). [b] Yield of isolated product after chromatography (silica gel #60-120; ethyl acetate/hexane, 5:95).

To explore the generality of the reaction, various substituted anilines and different arylhydrazines were transformed into *N*-arylamines under the optimized reaction conditions, and the results are recorded in Tables 2, 3, and 4.

Reactions were carried out with phenylhydrazine (**2a**) as the arylating counterpart with various substituted anilines (Table 2). In all cases, the expected *N*-arylanilines were obtained. Substrates containing electron-donating groups gave

Table 4. *N*-Arylation of amines by using (4-fluorophenyl)hydrazine (**2c**) as the arylating counterpart.



Entry	Substrate 1	Product 6	Yield <sup>[b]</sup> [%]
1			68
2			66
3			67
4			68
5			60
6			60

[a] All reactions were carried out on a 5 mmol scale with the aniline (1 equiv.), (4-fluorophenyl)hydrazine (1.5 equiv.), IBX (3 equiv.), and  $\text{Cu}(\text{OAc})_2$  (0.1 equiv.). [b] Yield of isolated product after chromatography (silica gel #60-120; ethyl acetate/hexane, 5:95).

the products in good yields of 78–71% (Table 2, Entries 1–9), whereas substrates containing electron-withdrawing groups gave the products in lower yields of 67–63% (Table 2, Entries 10–12).

Reactions with (2-ethylphenyl)hydrazine (**2b**) as the arylating agent also gave good yields of the *N*-arylated products (Table 3). Here again, substrates containing electron-donating groups gave the products in good yields of 72–67% (Table 3, Entries 1–6), whereas substrates containing electron-withdrawing groups gave the products in lower yields of 63–60% (Table 3, Entries 7 and 8).

Similarly, all the reactions attempted with (4-fluorophenyl)hydrazine (**2c**) as the arylating counterpart with a variety of anilines were successful, and the same trend in the yield of the *N*-arylated products was observed with respect to the nature of the substituents. Substrates containing electron-donating groups gave good yields of the products in the range of 68–66% (Table 4, Entries 1–4), whereas lower yields of 60% were observed with substrates containing electron-withdrawing groups (Table 4, Entries 5 and 6).

To study the scope of the arylhydrazine counterpart, other arylhydrazines such as (2-chlorophenyl)hydrazine (**2d**), (3,5-dichlorophenyl)hydrazine (**2e**), and (2-bromo-

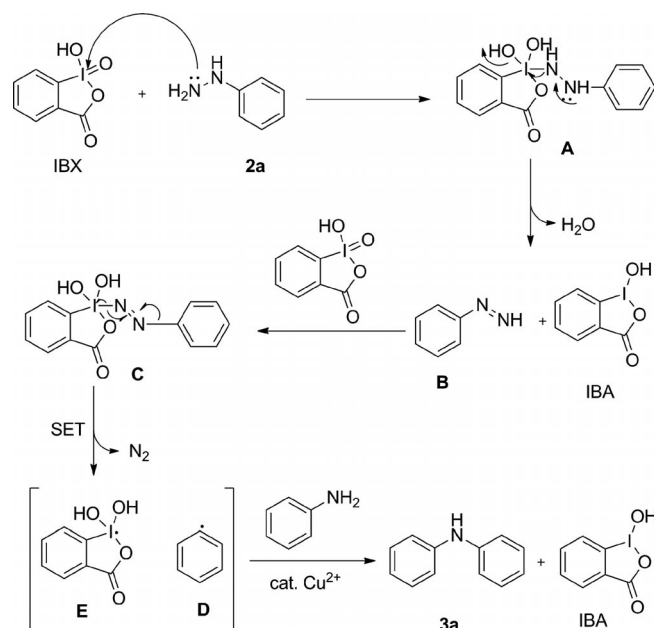
phenyl)hydrazine (**2f**) in addition to **2a–c** were evaluated in the reaction with aniline (Table 5). In all cases, the reactions were smooth, and the *N*-arylated products were obtained in 78–66% yield (Table 5, Entries 1–6).

An attempt was made to formulate a tentative mechanism for this reaction. Given that iodine is an electrophilic center in IBX, initial formation of IBX–phenylhydrazine adduct intermediate **A** is considered, which upon oxidative decomposition would form phenyldiazine **B** and *o*-iodosobenzoic acid (IBA). Phenyldiazine **B**, thus formed, reacts with a second molecule of IBX and undergoes oxidative cleavage through single-electron transfer to form phenyl free radical **D**, which is subsequently trapped by aniline in the presence of Cu(OAc)<sub>2</sub>, and this leads to the formation of *N*-phenylaniline (**3a**, Scheme 2). The role of Cu<sup>2+</sup> could be to stabilize the phenyl free radical and to bring together this radical and aniline through complexation;<sup>[16]</sup> this would lead to efficient trapping and consequently to a higher yield of the product.

Table 5. *N*-Arylation of aniline by using arylhydrazines **2** as the arylating counterpart.

Entry	Aryl hydrazine <b>2</b>	Product	Yield <sup>[b]</sup> [%]
1			78
2			72
3			68
4			68
5			70
6			66

[a] All reactions were carried out on a 5 mmol scale with aniline (1 equiv.), arylhydrazine (1.5 equiv.), IBX (3 equiv.), and Cu(OAc)<sub>2</sub> (0.1 equiv.). [b] Yield of isolated product after chromatography (silica gel #60-120; ethyl acetate/hexane, 2:98).



Scheme 2. Tentative mechanism depicted for *N*-phenylation of aniline by using **2a** as the arylating counterpart.

## Conclusions

Through free-radical trapping experiments we have established, for the first time, the combination of arylhydrazines with IBX for the generation of aryl free radicals. On the basis of this observation, a method was developed for the *N*-arylation of aromatic amines under mild conditions (base-free, –5 °C) by using arylhydrazines as the arylating counterpart. The scope of the reaction was demonstrated with a number of arylhydrazines and arylamines. This work may attract attention for further developments.



## Experimental Section

**General Procedure for the N-Arylation of Arylamines:** Copper(II) acetate (0.5 mmol) was added to a stirred solution of acetonitrile (20 mL) and the arylamine (5 mmol), and the mixture was cooled to  $-5\text{ }^{\circ}\text{C}$  in an ice/salt bath and stirred for 15 min. IBX (15 mmol) was added to this cold solution. The mixture was stirred for 5 min followed by the dropwise addition of arylhydrazine (7.5 mmol) in acetonitrile (5 mL) over a period of 20 min and then stirred until the reaction was complete. Upon completion of the reaction (3 h, by TLC), the reaction mixture was filtered, and the filtrate was concentrated under reduced pressure to remove acetonitrile. The residue was slurred by adding water (20 mL), neutralized with 10%  $\text{NaHCO}_3$  solution, and extracted with chloroform ( $2 \times 20\text{ mL}$ ). The organic layer was dried with anhydrous sodium sulfate, concentrated, and chromatographed (silica gel #60-120; ethyl acetate/hexane) to afford the pure product.

**CAUTION!** IBX was reported to be explosive under excessive heating or impact.<sup>[17]</sup> Therefore, the temperature of the water bath/oil bath of the rotary evaporator should not be more than  $50\text{ }^{\circ}\text{C}$  while concentrating the IBX-containing mixture.

**Supporting Information** (see footnote on the first page of this article): Experimental details, analytical data, and copies of the  $^1\text{H}$  NMR spectra.

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