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Article

Substrate-promoted copper-catalyzed *N*-arylation of amino alcohols with aryl iodides in water

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ABSTRACT

An efficient method has been developed for the *N*-arylation of a variety of water-soluble amino alcohols (1.2 mmol) with aryl iodides (1.0 mmol) in water under CuI-catalyzed conditions. The reaction was conducted via substrate-promoted action and did not require an additional ligand or phase-transfer catalyst, and afforded the desired *N*-aryl amines in acceptable to excellent yields (64%–93%) under mild reaction conditions with a small excess of the amino alcohol.

1. Introduction

Arylamines are an important class of compounds that possess a range of significant biological and pharmaceutical activity [1–4]. Extensive research efforts have been directed towards developing effective new methods for the synthesis of compounds of this type, with particular emphasis on the transition-metal-catalyzed *N*-arylation of amines. Traditional protocols available for the construction of C–N bonds include the copper-mediated Ullmann and palladium-catalyzed Buchwald-Hartwig reactions [5–11]. The application of these methods, however, can sometimes be limited by their requirement for elevated temperatures, which can be as high as 150 °C, and a high-boiling point polar solvent such as *N*,*N*-dimethylformamide (DMF) [12], dimethyl sulfoxide (DMSO) [13], nitrobenzene or *N*-methylpyrrolidone [14–16]. In addition, the toxic and air-sensitive palladium catalysts required of the Buchwald-Hartwig reaction can be damaging to the environment. These drawbacks have undoubtedly limited the wider application of aryl amination strategies for the construction of *N*-arylamine. Following on from Buchwald's discovery that the Cu-catalyzed *N*-arylation of nitrogen-containing heterocyclic with aryl halides could be achieved in good yields under mild reaction conditions in the presence of bidentate *N*,*N*-ligands [17,18], there has been considerable interest in the development of novel and more efficient ligands for these cross-coupling reactions. As a result, a variety of different ligands have been reported in the literature as effective catalysts for this type of C–N bond forming reaction, including organic phosphanes [19], diamines [20,21], diols [5], salicylamides [22], imines [23], amino acids [24], amino phosphates [25], *N*-containing aromatic heterocycles [26], triols [27], *β*-diketones [28], rac-binols [7], *β*-keto esters [29], and diazaphospholanes [30]. It is noteworthy, however, that the con-

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struction of the external ligands described above can sometimes be time-consuming and expensive. The development of analogous reactions that do not require a ligand or additive, therefore, represents an attractive challenge to organic chemists.

In recent years, chemists have developed an interest in using water as a solvent for copper-catalyzed *N*-arylation reactions, and several methods have been reported in this area involving the use of phase-transfer catalysts or microwave radiation to facilitate the reactions [31–34]. Procedures that operate in aqueous media offer significant advantages over those that are conducted in organic solvents, in terms of their overall cost, safety, and large-scale industrial application.

We recently became interested in developing a mild and effective method for the *N*-arylation of amine derivatives [35,36]. Based on the structural characteristics of some *N*,*O*-containing coordinative substrates, it was envisaged that amino alcohols, which are small bi-soluble (inorganic and organic) molecules, could play an additional role as ligands in this *N*-arylation process. Our recent work demonstrated that the copper-catalyzed *N*-arylation of amino alcohols and diamines with aryl halides could be accomplished in good to excellent yields under ligand-free and solvent-free conditions, although the requirement of this procedure for three equivalents of the amino alcohols represented a significant limitation to the general application of this approach [35]. Herein, we describe the development of a new catalytic system that can be applied to the *N*-arylation of amino alcohols using CuI in water according to a substrate-promoted action, without the need for an additional ligand or phase transfer catalyst.

2. Experimental

Copper(I) iodide (97%, fine powder) was purchased from Shanghai Sinpeuo Fine Chemical (Shanghai, China). Potassium hydroxide was purchased from Sinopharm Chemical Reagents (Shanghai, China). All of the aryl iodides and amines were purchased from Aladdin Chemistry Co. Ltd. (Shanghai, China). All of the commercially sourced materials were used without further purification. Flash column chromatography was performed with silica gel (230–400 mesh). ^{1}H and ^{13}C NMR spectra were recorded on a Varian 300 or 500 MHz instrument (Palo Alto, America) with chemical shifts reported relative to the residual deuterated solvent peaks. All of the yields reported in the current study represent an average of at least two independent runs. The known compounds were characterized by comparing their ^{1}H NMR data with those previously reported in the literature.

The general procedure for the *N*-arylation of amino alcohols with iodobenzenes was as follows.

Iodobenzene (1.0 mmol), amino alcohol (1.2 mmol), CuI (0.1 mmol), KOH (2.0 mmol), and H_2O (1 ml) were added to a screw-capped test tube, and the resulting mixture was stirred at 100 °C for 12 h. Upon completion of the reaction, determined by TLC, the reaction mixture was cooled to ambient temperature and extracted with ethyl acetate (3 × 5 ml). The combined organic phases were then dried over anhydrous Na_2SO_4 before being concentrated under reduced pressure to give the crude product as a residue, which was purified by flash column chromatography over silica gel to afford the desired products **3a–3m**.

2-Phenylamino-1-ethanol (**3a**) [37] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22 (t, 2H, *J* = 7.8 Hz), 6.78 (t, 1H, *J* = 7.3 Hz), 6.71 (d, 2H, *J* = 7.8 Hz), 3.85 (t, 2H, *J* = 5.3 Hz), 3.33 (t, 2H, *J* = 5.3 Hz).

2-(4-Bromophenylamino)ethanol (**3b**) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 (m, 2H), 6.52 (m, 2H), 3.82 (t, 2H, *J* = 5.2 Hz), 3.26 (t, 2H, *J* = 5.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) 46.3, 70.0, 109.8, 115.0, 132.0, 146.7.

2-(3-Bromophenylamino)ethanol (**3c**) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.00 (t, 1H, *J* = 8.0 Hz), 6.82 (m, 1H), 6.74 (t, 1H, *J* = 2.0 Hz), 6.52 (m, 1H), 3.76 (t, 2H, *J* = 5.2 Hz), 3.21 (t, 2H, *J* = 5.2 Hz); ^{13}C NMR (75 M Hz, $CDCl_3$) 45.7, 60.9, 111.9, 115.6, 120.5, 123.2, 130.5, 149.3.

2-(3-Tolylamine)ethanol (**3d**) ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.05 (t, 1H, *J* = 7.6 Hz), 6.55 (d, 1H, *J* = 7.4 Hz), 6.44 (d, 2H, *J* = 7.5 Hz), 3.75 (t, 2H, *J* = 5.2 Hz), 3.22 (t, 2H, *J* = 5.2 Hz), 2.26 (s, 3H).

2-[(4-Methoxyphenyl)amino]ethanol (**3e**) [33] ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.81 (s, 4H), 3.85 (t, 2H, *J* = 5.2 Hz), 3.74 (s, 3H), 3.30 (t, 2H, *J* = 5.2 Hz).

3-Phenylamino-1-propanol (**3f**) [38] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.17 (t, 2H, *J* = 7.5 Hz), 6.71 (t, 1H, *J* = 7.5 Hz), 6.62 (d, 2H, *J* = 7.5 Hz), 3.74 (t, 2H, *J* = 6.1 Hz), 3.22 (t, 2H, *J* = 6.1 Hz), 1.82 (m, 2H).

4-Phenylamino-1-butanol (**3g**) [39] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.16 (t, 2H, *J* = 7.8 Hz), 6.7 (t, 1H, *J* = 7.8 Hz), 6.59 (d, 2H, *J* = 7.8 Hz), 3.64 (t, 2H, *J* = 6.1 Hz), 3.14 (t, 2H, *J* = 6.1 Hz), 1.67 (m, 4H).

1-Phenylamino-2-propanol (**3h**) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 (t, 2H, *J* = 7.5 Hz), 6.72 (t, 1H, *J* = 7.5 Hz), 6.64 (d, 2H, *J* = 7.5 Hz), 4.00 (m, 1H), 3.20 (d, 1H, *J* = 12.0 Hz), 2.98 (dd, 1H, *J* = 8.5 Hz, *J* = 12.0 Hz), 1.24 (d, 3H, *J* = 6.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) 20.7, 51.4, 66.1, 113.1, 117.6, 129.1, 148.0.

2-Phenylamino-1-butanol (**3i**) [32] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14 (t, 2H, *J* = 7.8 Hz), 6.68 (t, 1H, *J* = 7.8 Hz), 6.63 (d, 2H, *J* = 7.8 Hz), 3.72 (dd, 1H, *J* = 5.1 Hz, *J* = 10.9 Hz), 3.5 (dd, 1H, *J* = 5.1 Hz, *J* = 10.9 Hz), 3.38 (m, 1H), 1.56 (m, 2H), 0.94 (t, 3H, *J* = 7.5 Hz).

2-(3-Bromophenylamino)-1-butanol (**3j**) ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.99 (t, 1H, *J* = 8.0 Hz), 6.82 (m, 1H), 6.78 (t, 1H, *J* = 2.0 Hz), 6.55 (m, 1H), 3.72 (m, 1H), 3.53 (m, 1H), 3.35 (m, 1H), 1.57 (m, 2H), 0.96 (t, 3H, *J* = 7.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) 10.5, 24.7, 56.5, 63.8, 112.2, 116.1, 120.4, 123.3, 130.5, 149.3; ESI-MS *M*r/Z 244.9/246.9 (1:1) [MH$^+$].

2-(3-Tolylamino)butan-1-butanol (**3k**) ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.05 (m, 1H), 6.54 (d, 1H, *J* = 7.6 Hz), 6.46 (d, 2H, *J* = 7.6 Hz), 3.72 (dd, 1H, *J* = 4.2 Hz, *J* = 10.8 Hz), 3.5 (dd, 1H, *J* = 5.9 Hz, *J* = 10.8 Hz), 3.38 (m, 1H), 2.28 (s, 3H), 1.54 (m, 2H), 0.95 (t, 3H, *J* = 7.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) 10.5, 24.9, 29.7, 56.7, 64.1, 110.8, 114.5, 118.8, 129.2, 139.0, 148.5; ESI-MS *M*r/Z 180.2 [MH$^+$].

N,*N*-Dimethyl-*N*′-phenylethylene-1,2-diamines (**3l**) [40] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.19 (d, *J* = 7.5 Hz, 1H), 6.77 (t, *J* = 7.2 Hz, 1H), 6.73 (d, *J* = 7.9 Hz, 1H), 4.09 (t, *J* = 6.8 Hz, 1H), 3.67 (d, *J*

= 5.1 Hz, 1H), 3.31 (t, *J* = 5.3 Hz, 1H), 2.86 (s, 3H).

2-(Phenylamino)phenol (**3m**) [41] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.19 (m, 3H), 7.09 (t, *J* = 8.0 Hz, 1H), 6.98 (d, *J* = 8.0 Hz, 1H), 6.87 (m, 2H), 6.77 (d, *J* = 7.6 Hz, 2H), 5.76 (br, 1H), 5.24 (br, 1H).

2-Phenoxybenzenamine (**3m'**) [41] ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.29 (br, 2 H), 6.14 (dd, 1H, *J* = 6.8, *J* = 2.4 Hz), 6.19 (d, 1H, *J* = 2.4 Hz), 6.33 (d, 1H, *J* = 8.0 Hz), 6.97–7.01 (m, 3H), 7.09 (t, 1H, *J* = 7.2 Hz), 7.36 (t, 2H, *J* = 7.6 Hz).

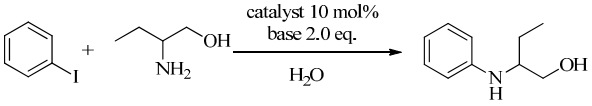
3. Results and discussion

3.1. Optimization of conditions

The current work focused initially on the coupling of 2-amino-1-butanol with iodobenzene as a model reaction to optimize the reaction conditions. A variety of different bases (2 equiv.) and Cu sources (10 mol%) were screened against the standard coupling procedure, which involved the reaction of iodobenzene (1 equiv.) with 2-amino-1-butanol (1.2 equiv.) in water (1 ml). The results of these screening experiments are shown in Table 1.

Our initial work focused on screening different bases against the model *N*-arylation reaction in the presence of CuI (0.1 equiv.). Pleasingly, the reaction proceeded well when KOH was used as the base and gave the desired product in a yield of 75% following 12 h at 85 °C (Table 1, entry 5). Unfortunately, the use of other bases such as K_3PO_4, K_2CO_3, Cs_2CO_3, and NaOH resulted in lower yields (Table 1, entries 1–4). Several readily available copper salts, including CuI, Cu_2O, CuBr and CuCl, were then evaluated under the KOH conditions (Table 1, entries 5–8), and CuI was found to provide the best results (Table 1, entry 5). We then proceeded to investigate the effects of the temperature and the reaction time on the outcome of the reaction (Table 1, entries 8–10). Pleasingly, when the reaction was conducted at 100 °C, the desired amination product was obtained in 89% yield (Table 1, entry 9), representing a significant increase from the yield obtained when the reaction was

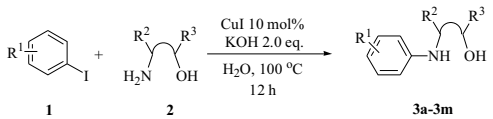
Table 1
Screening of the reaction conditions for the *N*-arylation of 2-amino-1-butanol with iodobenzene.

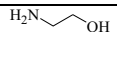
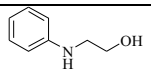
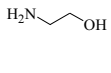
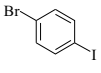
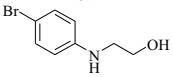
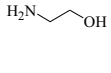
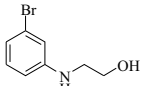
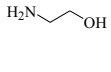
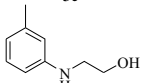
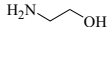
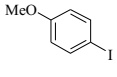
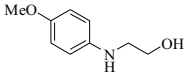
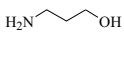
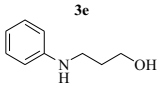
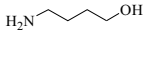
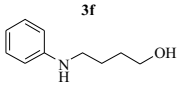
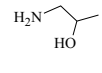
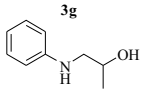
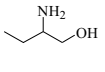
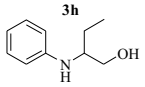
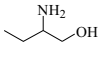
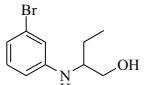
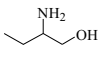
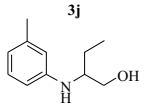
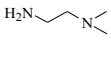
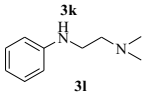
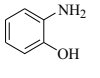


Entry	Catalyst	Base	Temperature (°C)	Time (h)	Yield[a] (%)
1	CuI	K_3PO_4	85	12	36
2	CuI	K_2CO_3	85	12	31
3	CuI	Cs_2CO_3	85	12	43
4	CuI	NaOH	85	12	61
5	CuI	KOH	85	12	75
6	Cu_2O	KOH	85	12	61
7	CuBr	KOH	85	12	67
8	CuCl	KOH	85	12	72
9	CuI	KOH	100	12	89
10	CuI	KOH	100	20	89

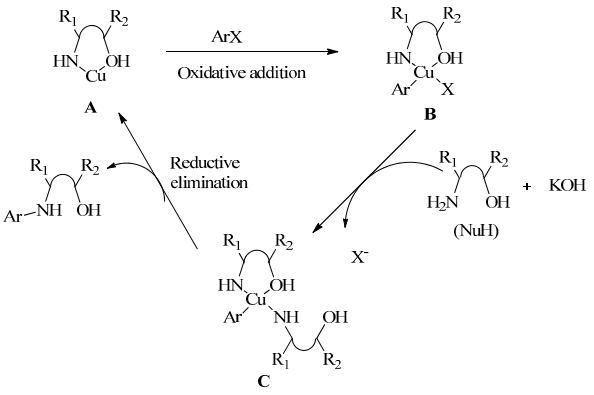
Reactions performed with iodobenzene (1.0 mmol) and 2-amino-1-butanol (1.2 mmol) in water (1 ml).
[a] Isolated yield.

Table 2
N-arylation of amino alcohols with iodobenzenes in water.



Entry	Amine (**2**)	Aryl halide (**1**)	Product (**3**)	Yield [a] (%)
1			 **3a**	93
2			 **3b**	86
3			 **3c**	90
4			 **3d**	86
5			 **3e**	84
6			 **3f**	91
7			 **3g**	67
8			 **3h**	76
9			 **3i**	89
10			 **3j**	92
11			 **3k**	64 (65%[b], 98%[c])
12			 **3l**	46 (48%[b], 95%[c])
13			**3m** + **3m'**	18 (2:1) (19%[b], 96%[c])

Reactions performed with ArI (1.0 mmol) and amino alcohols (1.2 mmol) in water (1 ml).
[a] Isolated yield.
[b] Conversion of the substrate.
[c] Selectivity for the product.



Scheme 1. Proposed substrate-promoted mechanism for copper-catalyzed *N*-arylation of amino alcohols.

conducted at 85 °C (Table 1, entry 5). The extension of the reaction time to 20 h, however, did not result in any further increase in the reaction yield (Table 1, entry 10). Taken together, these results therefore indicated that the optimized procedure for the *N*-arylation of 2-amino-1-butanol required the application of CuI as a catalyst and KOH as a base in water at 100 °C (Table 1, entry 9).

3.2. Substrate scope of amino alcohols and aryl iodides

With the optimized reaction conditions in hand, we proceeded to investigate the scope of this reaction using a variety of different amino alcohols and aryl iodides. The results of this work are summarized in Table 2. All of the products resulting from these experiments were characterized by ^{1}H NMR and MS, and were consistent with the literature data. The results in Table 2 clearly demonstrate the broad scope of the current procedure, with a variety of different amino alcohols, such as ethanolamine and 3-amino-1-propanol, giving the desired products in high yields (Table 2, entries 1–6). When 4-aminobutanol was used as a substrate, a lower yield of 67% was obtained (Table 2, entry 7). Even amino alcohols bearing branched alkyl chains afforded the desired products in high yields (Table 2, entries 8–11). As for the aryl iodides, a range of different substituents was tolerated, including bromo, methyl, and methoxy groups. When 2-aminobutanol was subjected to the optimized reaction conditions, significant electronic effects were observed for the electron-poor and electron-rich substituted aryl iodides (Table 2, entries 10 and 11). The presence of an electron-donating methyl or methoxy group led to lower levels of reactivity than that observed in the presence of an electron-withdrawing bromo substituent. Based on these interesting results, the decision was taken to further investigate the scope of the reaction using different substrates. Thus, when *N*,*N*-dimethylethlenediamine was used as substrate, **3l** was obtained in 46% yield (Table 2, entry 12). In addition, when 2-aminophenol was selected as a substrate, the corresponding *N*-arylation product **3m** and *O*-arylation product **3m'** were obtained in yields of 12% and 6%, respectively (Table 2, entry 13). Morpholine, imidazole and benzimidazole were also investigated as substrates. In these cases, however, no products were obtained likely because of the poor water solubility of these substrates, which would make it difficult for them to coordinate to the Cu center.

3.3. Mechanistic consideration

Based on the results provided above, we have proposed a substrate (amino alcohol)-promoted catalytic mechanism for the current ligand-free aqueous-phase reactions (Scheme 1). Thus, the amino alcohol is bis-nucleophilic and capable of forming chelator **A** with CuI. Subsequent oxidative addition of an aryl halide to chelator **A** would lead to the formation of intermediate **B**, which could undergo nucleophilic substitution in the presence of base to give complex **C**. Reductive elimination of **C** could then give rise to the *N*-arylation product [42].

4. Conclusions

We have successfully developed a new method for the aqueous phase copper-catalyzed *N*-arylation of amino alcohols that does not require an additional ligand, phase-transfer catalyst, or microwave irradiation. The reactions proceeded smoothly and efficiently in most cases to afford the *N*-arylation products in high yields under mild reaction conditions with only a small excess of the amino alcohol. Further studies aimed at expanding the scope of this protocol to other nitrogen nucleophiles, as well as developing a deeper understanding of the reaction mechanism, are currently underway in our laboratory.

Acknowledgments

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Graphical Abstract

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Substrate-promoted copper-catalyzed *N*-arylation of amino alcohols with aryl iodides in water

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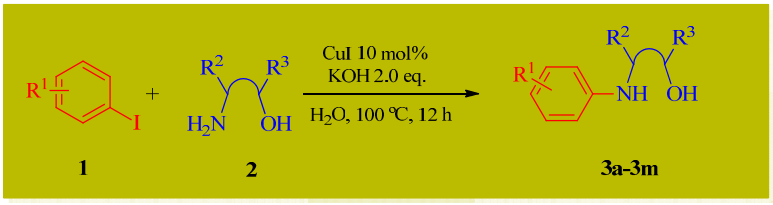

This work describes the discovery of a new catalytic system for *N*-arylation of amino alcohols using CuI in water via substrate-promoted action without the need for an additional ligand or phase transfer catalyst.

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