ELSEVIER
ScienceDirect
Tetrahedron Letters 48 (2007) 7845–7848
Tetrahedron Letters

Solvent-free copper/iron co-catalyzed N-arylation reactions of nitrogen-containing heterocycles with trimethoxysilanes in air

Ren-Jie Song, Chen-Liang Deng, Ye-Xiang Xie and Jin-Heng Li*

Key Laboratory of Chemical Biology and Traditional Chinese Medicine Research (Ministry of Education), College of Chemistry and Chemical Engineering, Hunan Normal University, Changsha 410081, China

Received 13 May 2007; revised 30 August 2007; accepted 31 August 2007
Available online 5 September 2007

Abstract—A solvent-free copper/iron-catalyzed N-arylation of nitrogen-containing heterocycles with trimethoxysilanes method for the formation of C–N bonds has been developed. In the presence of Cu, $FeCl_3$, TBAF, and air, a variety of nitrogen-containing heterocycles including imidazoles and triazoles were coupled with aryltrimethoxysilanes and vinyltrimethoxysilane to afford the corresponding products in moderate to excellent yields. It is noteworthy that the reaction is conducted under solvent-free and relatively low Cu/$FeCl_3$ loadings conditions.

The transition metal-catalyzed N-arylation of nitrogen-containing heterocycles (e.g., imidazoles and triazoles) reactions has received considerable attention in recent years due to their successful uses in the preparation of *N*-aryl compounds in pharmaceuticals, crop-protection chemicals, and material sciences.[1] Among these N-arylation transformations, the nucleophilic aromatic reagents are usually aryl halides and pseudo-aryl halides.[2] Organometals, such as aryllead triacetates,[3] arylboronic acids,[4] arylbismuths,[5] diaryl iodonium salts,[6] arylstannanes,[7] and arylsilanes,[8] are also found to be efficient reaction partners for the N-arylation reaction with the nitrogen-containing heterocycles. Although arylsilanes are widely used as the reaction partners for the palladium-catalyzed Hiyama cross-coupling reactions with aryl halides because they are readily available and/or less toxic,[9] only one paper has been reported on the N-arylation reactions of the nitrogen-containing heterocycles with arylsilanes.[8] In the presence of $Cu(OAc)_2$ and TBAF, a variety of imidazoles, amines, and amides were coupled with silanes under air atmosphere in moderate to good yields. However, the method was conducted in harmful organic solvents besides the requirement of excess stoichiometric amount of $Cu(OAc)_2$ promoter. The development of a new and efficient method for the N-arylations of the nitrogen-containing heterocycles with arylsilanes using a catalytic amount of catalyst under solvent-free conditions became attractive. After a series of trials, we found that a catalytic amount of Cu combined with a catalytic amount of $FeCl_3$ as co-catalysts could be effective for the N-arylations of the nitrogen-containing heterocycles with aryltrimethoxysilanes and vinyltrimethoxysilane in the presence of TBAF and air. Moreover, the reaction was carried out under solvent-free conditions (Eq. 1).

$$\text{Het}-\text{NH} + \text{RSi(OR')}_3 \xrightarrow[\text{TBAF, 50 °C, Air}]{\text{Cu/FeCl}_3} \text{Het}-\text{N}-\text{R} \tag{1}$$

Het−NH = imidazoles, triazoles
R = aryl, vinyl

The N-arylation of 1*H*-benzo[*d*]imidazole (**1a**) with trimethoxy(phenyl)silane (**2a**) in the presence of 3 equiv of $TBAF{\cdot}3H_2O$ was first studied to seek a suitable catalyst (Table 1). We have recently demonstrated that TBAF (n-Bu_4NF) is an effective base for the solvent-free Pd or Cu-catalyzed cross-coupling reactions.[2u,9i,10] Thus, we expected to perform this reaction under solvent-free conditions using TBAF as base. As expected, the N-arylation of **1a** with silane **2a** proceeded successfully under solvent-free conditions using Cu/$FeCl_3$ as the co-catalysts and TBAF as the base in air atmosphere. Treatment of **1a** with silane **2a**, a stoichiometric amount of $Cu(OAc)_2$ and TBAF after 23 h afforded the corresponding product **3** in 52% yield (entry 1). Identical results were obtained using a stoichiometric amount of Cu_2O instead of $Cu(OAc)_2$ (entry 2). In the presence

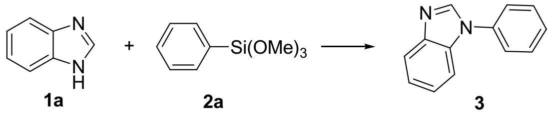
---

Keywords: Copper; $FeCl_3$; N-Arylation; Imidazole; Trimethoxysilane.

* Corresponding author. Tel.: +86 731 8872530; fax: +86 731 8872531; e-mail: jhli@hunnu.edu.cn

0040-4039/$ - see front matter © 2007 Elsevier Ltd. All rights reserved.
doi:10.1016/j.tetlet.2007.08.117

Table 1. Screening the optimum conditions[a]



Entry	Catalysis (mol %)	Time (h)	Isolated yield (%)
1	$Cu(OAc)_2$ (100)	23	52
2	Cu_2O (100)	23	51
3	Cu (100)	21	58
4	Cu (40)	21	61
5	Cu (20)	24	48
6	Cu (20)/$Cu(OAc)_2$ (20)	24	51
7	Cu (10)/$Cu(OAc)_2$ (10)	23	53
8	Cu (10)/$CuBr_2$ (10)	23	Trace
9	$FeCl_3$ (40)	23	60
10	$FeCl_3$ (20)	24	51
11	Cu (40)/$FeCl_3$ (40)	23	60
12	Cu (50)/$FeCl_3$ (50)	17	52
13	Cu (30)/$FeCl_3$ (30)	24	63
14	Cu (20)/$FeCl_3$ (20)	15	71
15	Cu (5)/$FeCl_3$ (5)	24	82
16	***Cu (3)/$FeCl_3$ (3)***	***24***	***82***
17	Cu (1)/$FeCl_3$ (1)	28	81
18[b]	Cu (3)/$FeCl_3$ (3)	24	62
19[c]	Cu (3)/$FeCl_3$ (3)	24	Trace
20[d]	Cu (3)/$FeCl_3$ (3)	24	32
21[e]	Cu (3)/$FeCl_3$ (3)	24	78
22[f]	Cu (3)/$FeCl_3$ (3)	24	52

[a] Reaction conditions: **1a** (1.0 mmol), **2a** (2.0 mmol), and TBAF·$3H_2O$ (3 equiv) in air atmosphere at 50 °C.

[b] At room-temperature.

[c] In Ar atmosphere.

[d] In O_2 atmosphere.

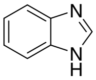
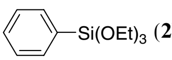
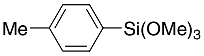
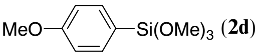
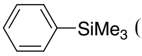
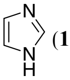
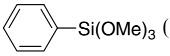
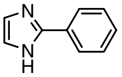
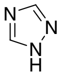
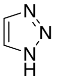
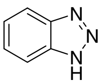
[e] TBAF·$3H_2O$ (1 equiv).

[f] TBAF·$3H_2O$ (2 equiv).

of a stoichiometric amount of Cu, the yield was enhanced slightly to 58% (entry 3). To our delight, the same yield of **3** was still obtained when a catalytic amount of Cu was used as catalyst (40 mol % Cu; entry 4). However, further decreasing amount of Cu reduced the yield (20 mol % Cu; entry 5). Unfortunately, attempts to the use of some Cu co-catalysts were unsuccessful (entries 6–8). Very recently, the use of iron as a co-catalyst was found to improve the copper-catalyzed N-arylations of nitrogen-containing heterocycles with aryl halides.[2x] Consequently, iron as the catalyst for the reaction of substrate **1a** with silane **2a** was examined. We were happy to find that a catalytic amount of $FeCl_3$ could also catalyze the reaction of substrate **1a** with silane **2a** and TBAF smoothly to provide the target product **3** in a moderate yield (entries 9 and 10). Encouraged by these results, we subsequently evaluated the effect of the Cu/$FeCl_3$ co-catalysts on the reaction. The results showed that the amount of both Cu and $FeCl_3$ played a crucial role on the reaction. In the presence of 40 mol % of Cu and 40 mol % of $FeCl_3$, the same yield of **3** was isolated as that of either Cu or $FeCl_3$ as the catalyst (entries 4, 9, and 11). Further increasing amount of both Cu and $FeCl_3$ resulted in decreasing the yield of **3** to some extent (entry 12). It is interesting to observe that the reduction of both Cu and $FeCl_3$ favored the reaction, and the loadings of both Cu and $FeCl_3$ are 3 mol % providing the best result in view of the reaction yield and rate (entries 13–17). The reaction temperature was also tested, and the yield of **3** was reduced to 62% at room temperature (entries 16 and 18). It is noteworthy that the reaction gave unsatisfactory results when it was carried out in argon atmosphere or oxygen atmosphere (entries 19 and 20). The results showed that the yield was reduced to some extent in the presence of either 2 equiv or 1 equiv of TBAF (entries 21 and 22).

We then explored the scope of the reaction of the nitrogen-containing heterocycles including imidazoles and triazoles, and the results are summarized in Table 2.[11] It was found that 1*H*-benzo[*d*]imidazole (**1a**) could also undergo the N-arylation with other silanes **2b–e** in moderate to good yields under the standard conditions (entries 1–4). For example, substrate **1a** was reacted with trimethoxy(*p*-tolyl)silane (**2c**) Cu, $FeCl_3$, and TBAF·$3H_2O$ efficiently to afford the corresponding product **4** in a 93% yield (entry 2). However, the other silanes **2f**

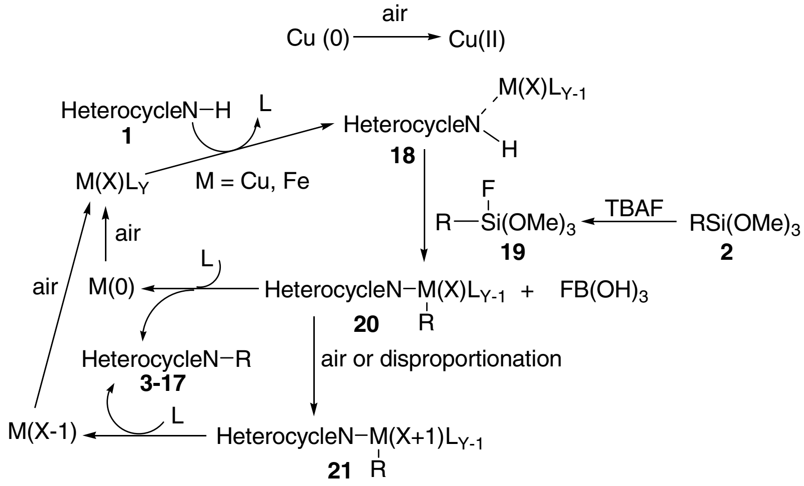
Table 2. N-Arylations of imidazoles and triazoles with trimethoxysilanes in the presence of $FeCl_3$, Cu, and TBAF[a]

Entry	Substrate	Silane	Yield[b] (%)
1	 (**1a**)	 (**2b**)	66 (**3**)
2	(**1a**)	 (**2c**)	93 (**4**)
3	(**1a**)	 (**2d**)	80 (**5**)
4	(**1a**)	 (**2e**)	66 (**6**)
5[c]	(**1a**)	 (**2f**)	Trace (**7**)
6[c]	(**1a**)	 (**2g**)	Trace (**3**)
7	 (**1b**)	 (**2a**)	45 (**8**)
8	(**1b**)	(**2c**)	58 (**9**)
9	(**1b**)	(**2e**)	41 (**10**)
10[c]	 (**1c**)	(**2c**)	Trace (**11**)
11	 (**1d**)	(**2a**)	68 (**12**)
12	(**1d**)	(**2c**)	96 (**13**)
13	(**1d**)	(**2e**)	33 (**14**)
14	 (**1e**)	(**2a**)	52 (**15**)
15	(**1e**)	(**2c**)	57 (**16**)
16	 (**1f**)	(**2c**)	Trace (**17**)

[a] Reaction conditions: **1** (1 mmol), **2** (2 mmol), Cu (3 mol %), $FeCl_3$ (3 mol %), and TBAF·$3H_2O$ (3 mmol) in air at 50 °C for 24 h.

[b] Isolated yield.

[c] >95% of the starting substrate was recovered.



Scheme 1. A working mechanism.

and **2g** were not suitable substrates for the reaction with **1a** (entries 5 and 6). Substrate **1b**, another imidazole, provided moderate yields from the N-arylation with the three silanes **2a**, **2c**, and **2e**, respectively, under the same conditions (entries 7–9). Unfortunately, no reaction was observed employing the hindered imidazole **1c** as the substrate (entry 10). To our delight, the standard conditions were also suitable for the reaction of triazoles (entries 11–16). In the presence of Cu, FeCl$_3$, and TBAF·3H$_2$O, the couplings of triazoles **1d**,**e** with silanes **2a**, **2c**, or **2e**, respectively, proceeded smoothly to produce the target products **12**–**16** in moderate to excellent yields (entries 11–15). Unfortunately, the reaction between another triazole **1f** and silane **2c** gave trace amount of the desired product under the same conditions (entry 16).

A working mechanism was proposed as outlined in Scheme 1 based on the previously proposed mechanism.[3–8,12] Trimethoxysilane can readily react with TABF to afford a pentavalent silicate intermediate **19**.[9,13] The pentavalent silicate **19** undergoes transmetalation with intermediate **18**, which was obtained from the coordination of nitrogen atom with catalyst (Cu(II) and/or Fe(III)), to generate intermediate **20**. Finally, the reductive elimination of intermediate **20** takes place directly to give the target product **3**–**17** and a lower-valent species (Cu(0) and/or Fe(II)). Alternatively, oxidation or disproportionation of intermediate **20** can occur with the aid of oxidant to give the corresponding higher oxidation-state intermediate **21**, which is more efficiently reductive elimination than intermediate **20**.[8] In view of the mechanism, there are two roles of air in the present process including (i) oxidation of the Cu(0) and M(X − 1) species to the reactive Cu(II) and M(X) species and (ii) oxidation of intermediate **20** to intermediate **21**. The presence of pure oxygen may induce other side-reactions resulting in a low yield of the desired product. We deduce that FeCl$_3$ can also play as an oxidant to regenerate the active catalyst species beside as catalyst itself.[12]

In summary, we have developed a catalytic and efficient protocol for the solvent-free N-arylations of some nitrogen-containing heterocycles including imidazoles and triazoles with aryltrimethoxysilanes using Cu/FeCl$_3$ as the co-catalysts and TBAF as the base under air atmosphere. In the presence of Cu, FeCl$_3$, and TBAF·3H$_2$O, a variety of imidazoles and triazoles worked well with aryltrimethoxysilanes and vinyltrimethoxysilane in moderate to excellent yields. Compared with the previously report, several features are established: (1) The reaction employs relatively low Cu and FeCl$_3$ catalyst loadings, and the loadings of both Cu and FeCl$_3$ affected the yields of the reactions. (2) The reaction was performed under solvent-free conditions. (3) The presence of argon or oxygen disfavored the reaction, but the best results were obtained under air atmosphere. Further applications of the present system in organic synthesis and the studies of the detailed mechanism are underway.

Acknowledgments

The authors thank the Program for the New Century Excellent Talents in University (No. NCET-06-0711), National Natural Science Foundation of China (No. 20572020), Fok Ying Tung Education Foundation (No. 101012), Key Project of Chinese Ministry of Education (No. 206102), Hunan Provincial Natural Science Foundation of China (No. 05JJ1002), and Specialized Research Fund for the Doctoral Program of Higher Education (No. 20060542007) for financial support.

Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tetlet.2007.08.117.

References and notes

1. For selected recent examples, see: (a) Qiao, J. X.; Cheng, X.; Modi, D. P.; Rossi, K. A.; Luettgen, J. M.; Knabb, R. M.; Jadhav, P. K.; Wexler, R. R. *Bioorg. Med. Chem. Lett.*

2005, *15*, 29; (b) Lange, J. H. M.; van Stuivenberg, H. H.; Coolen, H. K. A. C.; Adolfs, T. J. P.; McCreary, A. C.; Keizer, H. G.; Wals, H. C.; Veerman, W.; Borst, A. J. M.; de Looff, W.; Verveer, P. C.; Kruse, C. G. *J. Med. Chem.* **2005**, *48*, 1823; (c) Quan, M. L.; Lam, P. Y. S.; Han, Q.; Pinto, D. J. P.; He, M. Y.; Li, R.; Ellis, C. D.; Clark, C. G.; Teleha, C. A.; Sun, J.-H.; Alexander, R. S.; Bai, S.; Luettgen, J. M.; Knabb, R. M.; Wong, P. C.; Wexler, R. R. *J. Med. Chem.* **2005**, *48*, 1729; (d) Barchéchath, S. D.; Tawatao, R. I.; Corr, M.; Carson, D. A.; Cottam, H. B. *J. Med. Chem.* **2005**, *48*, 6409; (e) Voets, M.; Antes, I.; Scherer, C.; Müller-Vieira, U.; Biemel, K.; Barassin, C.; Marchais-Oberwinkler, S.; Hartmann, R. W. *J. Med. Chem.* **2005**, *48*, 6632; (f) Wiglenda, T.; Ott, I.; Kircher, B.; Schumacher, P.; Schuster, D.; Langer, T.; Gust, R. *J. Med. Chem.* **2005**, *48*, 6516; (g) Jiang, W.; Guan, J.; Macielag, M. J.; Zhang, S.; Qiu, Y.; Kraft, P.; Bhattacharjee, S.; John, T. M.; Haynes-Johnson, D.; Lundeen, S.; Sui, Z. *J. Med. Chem.* **2005**, *48*, 2126; (h) Hutzler, J. M.; Melton, R. J.; Rumsey, J. M.; Schnute, M. E.; Locuson, C. W.; Wienkers, L. C. *Chem. Res. Toxicol.* **2006**, *19*, 1650; (i) Wiglenda, T.; Gust, R. *J. Med. Chem.* **2007**, *50*, 1475; (j) Davey, D. D.; Adler, M.; Arnaiz, D.; Eagen, K.; Erickson, S.; Guilford, W.; Kenrick, M.; Morrissey, M. M.; Ohlmeyer, M.; Pan, G.; Paradkar, V. M.; Parkinson, J.; Polokoff, M.; Saionz, K.; Santos, C.; Subramanyam, B.; Vergona, R.; Wei, R. G.; Whitlow, M.; Ye, B.; Zhao, Z. S.; Devlin, J. J.; Phillips, G. *J. Med. Chem.* **2007**, *50*, 1146.

2. For selected recent papers on the N-arylations of nitrogen-containing heterocycles with aryl halides, see: Pd: (a) Hartwig, J. F. In *Handbook of Organopalladium Chemistry for Organic Synthesis*; Negishi, E., Ed.; Wiley-Interscience: New York, 2002; p 1051; (b) Jiang, L.; Buchwald, S. L. In *Metal-Catalyzed Cross-Coupling Reactions*; de Meijere, A., Diederich, F., Eds.; Wiley-VCH: Weinheim, 2004; p 699; (c) Bellina, F.; Cauteruccio, S.; Mannina, L.; Rossi, R.; Viel, S. *J. Org. Chem.* **2005**, *70*, 3997; (d) Anderson, K. W.; Tundel, R. E.; Ikawa, T.; Altman, R. A.; Buchwald, S. L. *Angew. Chem., Int. Ed.* **2006**, *45*, 6523; Cu: (e) Beletskaya, I. P.; Cheprakov, A. V. *Coord. Chem. Rev.* **2004**, *248*, 2337; (f) Ley, S. V.; Thomas, A. W. *Angew. Chem., Int. Ed.* **2003**, *42*, 5400; (g) Kuil, M.; Bekedam, E. K.; Visser, G. M.; van den Hoogenband, A.; Terpstra, J. W.; Kamer, P. C. J.; Kamer van Leeuwen, P. W. N. M.; van Strijdonck, G. P. F. *Tetrahedron Lett.* **2005**, *46*, 2405; (h) Altman, R. A.; Buchwald, S. L. *Org. Lett.* **2006**, *8*, 2779; (i) Hosseinzadeh, R.; Tajbakhsh, M.; Alikarami, M. *Synlett* **2006**, 2124; (j) Alcalde, E.; Dinarès, I.; Rodríguez, S.; Garcia de Miguel, C. *Eur. J. Org. Chem.* **2005**, 1637; (k) Antilla, J. C.; Baskin, J. M.; Barder, T. E.; Buchwald, S. L. *J. Org. Chem.* **2004**, *69*, 5578; (l) Jerphagnon, T.; van Klink, G. P. M.; de Vries, J. G.; van Koten, G. *Org. Lett.* **2005**, *7*, 5241; (m) Ma, D.; Cai, Q. *Synlett* **2004**, 128; (n) Zhang, H.; Cai, Q.; Ma, D. *J. Org. Chem.* **2005**, *70*, 5164; (o) Lv, X.; Wang, Z.; Bao, W. *Tetrahedron* **2006**, *62*, 4756; (p) Liu, L.; Frohn, M.; Xi, N.; Dominguez, C.; Hungate, R.; Reider, P. J. *J. Org. Chem.* **2005**, *70*, 10135; (q) Rao, H.; Fu, H.; Jiang, Y.; Zhao, Y. *J. Org. Chem.* **2005**, *70*, 8107; (r) Xu, L.; Zhu, D.; Wu, F.; Wang, R.; Wan, B. *Tetrahedron* **2005**, *61*, 6553; (s) Zhang, Z.; Mao, J.; Zhu, D.; Wu, F.; Chen, H.; Wan, B. *Tetrahedron* **2006**, *62*, 4435; (t) Choudary, B. M.; Sridhar, C.; Kantam, M. L.; Venkanna, G. T.; Sreedhar, B. *J. Am. Chem. Soc.* **2005**, *127*, 9948; (u) Xie, Y.-X.; Pi, S.-F.; Wang, J.; Yin, D.-L.; Li, J.-H. *J. Org. Chem.* **2006**, *71*, 8324; (v) Lv, X.; Bao, W. *J. Org. Chem.* **2007**, *72*, 3863; (w) Zhu, L.; Cheng, L.; Zhang, Y.; Xie, R.; You, J. *J. Org. Chem.* **2007**, *72*, 2737; Fe/Cu: (x) Taillefer, M.; Xia, N.; Ouali, A. *Angew. Chem., Int. Ed.* **2007**, *46*, 934.
3. Lopez-Alvarado, P.; Avendaño, C.; Menéndez, J. C. *J. Org. Chem.* **1995**, *60*, 5678.
4. (a) Lam, P. Y.; Clark, C. G.; Sauber, S.; Adams, J.; Winters, M. P.; Chan, D. M.; Combs, A. *Tetrahedron Lett.* **1998**, *39*, 2941; (b) Collman, J. P.; Zhong, M. *Org. Lett.* **2000**, *2*, 1233; (c) Collman, J. P.; Zhong, M.; Zeng, L.; Costanzo, S. *J. Org. Chem.* **2001**, *66*, 1528; (d) Collman, J. P.; Zhong, M.; Zhang, C.; Costanzo, S. *J. Org. Chem.* **2001**, *66*, 7892; (e) Lan, J.-B.; Chen, L.; Yu, X.-Q.; You, J.-S.; Xie, R.-G. *Chem. Commun.* **2004**, 188; (f) Kantam, M. L.; Venkanna, G. T.; Sridhar, C.; Sreedhar, B.; Choudary, B. M. *J. Org. Chem.* **2006**, *71*, 9522.
5. Fedorov, A. Y.; Finet, J.-P. *Tetrahedron Lett.* **1999**, *40*, 2747.
6. Kang, S.-K.; Lee, S.-H.; Lee, D. *Synlett* **2000**, 1022.
7. Lam, P. Y. S.; Clark, C. G.; Saubern, S.; Adams, J.; Averill, K. M.; Chan, D. M. T.; Combs, A. *Synlett* **2000**, 674.
8. Lam, P. Y. S.; Deudon, S.; Averill, K. M.; Li, R.; He, M. Y.; DeShong, P.; Clark, C. G. *J. Am. Chem. Soc.* **2000**, *122*, 7600.
9. For selected reviews and papers on the Hiyama cross-coupling reaction, see: (a) Diederich, F.; Stang, P. J. *Metal-Catalyzed Cross-coupling Reactions*; Wiley-VCH: Weinheim, 1998; (b) Miyaura, N. *Cross-Coupling Reaction*; Springer: Berlin, 2002; (c) de Meijere, A.; Diederich, F. *Metal-Catalyzed Cross-Coupling Reactions*; Wiley-VCH: Weinheim, 2004; (d) Li, J.-H.; Deng, C.-L.; Liu, W.-J.; Xie, Y.-X. *Synthesis* **2005**, 3039; (e) Wolf, C.; Lerebours, R. *Org. Lett.* **2004**, *6*, 1147; (f) Seganish, W. M.; DeShong, P. *J. Org. Chem.* **2004**, *69*, 1137; (g) Lerebours, R.; Wolf, C. *Synthesis* **2005**, 2287; (h) Alacid, E.; Nájera, C. *Adv. Synth. Catal.* **2006**, *348*, 945; (i) Li, J.-H.; Deng, C.-L.; Xie, Y.-X. *Synthesis* **2006**, 969.
10. (a) Liang, Y.; Xie, Y.-X.; Li, J.-H. *J. Org. Chem.* **2006**, *71*, 379; (b) Deng, C.-L.; Xie, Y.-X.; Yin, D.-L.; Li, J.-H. *Synthesis* **2006**, 3370; (c) Xie, Y.-X.; Deng, C.-L.; Pi, S.-F.; Li, J.-H.; Yin, D.-L. *Chin. J. Chem.* **2006**, *24*, 1290.
11. *Typical experimental procedure for the Cu/FeCl$_3$-catalyzed N-arylations of some nitrogen-containing heterocycles with aryltrimethoxysilanes*. A mixture of 1 (1 mmol), aryltrimethoxysilanes 2 (2 mmol), Cu (3 mol %), FeCl$_3$ (3 mol %), and TBAF·3H$_2$O (3 mmol) was stirred in air atmosphere at 50 °C for 24 h until complete consumption of starting material as monitored by TLC. After the reaction was finished, the mixture was poured into ethyl acetate, which was washed with brine. The aqueous layer was extracted with ethyl acetate and the combined organic layers were dried over anhydrous Na$_2$SO$_4$ and evaporated under vacuum. The residue was purified by flash column chromatography (hexane/ethyl acetate or CH$_2$Cl$_2$/CH$_3$OH) to afford the desired product.
12. For a review on the use of iron as catalysts, see: Bolm, C.; Legros, J.; Le Paih, J.; Zani, L. *Chem. Rev.* **2004**, *104*, 6217.
13. Mowery, M. N.; DeShong, P. *Org. Lett.* **1999**, *1*, 2137.