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A Solvent-Free Process for Synthesis of Imines by Iron-Catalyzed Oxidative Self- or Cross-Condensation of Primary Amines Using Molecular Oxygen as Sole Oxidant

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Abstract The synthesis of imines by oxidative self- or cross-condensation of primary amines has been achieved using iron(II) bromide as the catalyst. The protocol is ligand-free, additive-free, solvent-free, and high-yielding and utilizes renewable oxygen as sole oxidant. The reaction tolerated a wide range of functionalities.

Graphical Abstract

Keywords Oxidative condensation · Iron(II) bromide · Molecular oxygen · Imines · Primary amines

Dedicated to Professor Pavan Mathur on the occasion of his 65th birthday.

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1 Introduction

Oxidative condensation reactions are important transformations in organic synthesis, which are largely promoted by metals such as Pd, Ru, Cu, and Ni catalysts [1, 2]. Recently, we reported analogous C–C, C–N, C–O, and C–S bond-forming processes using iron compounds as the catalysts [3, 4]. In general, iron compounds are of great synthetic interest because they possess benign character and abundant [5–11].

Imines are versatile intermediates for the synthesis of pharmaceutically and biologically active compounds, and fine chemicals [12–14]. The C=N bond in imines is also widely used in organic transformations such as addition, reduction, cyclization, and aziridination reactions [15–20]. Imines have traditionally been synthesized through condensation of primary amines with aldehydes or ketones, but alternative mild and practical catalytic methods [21–24], including oxidative coupling of alcohols and amines [25–30] together with oxidation of secondary amines [31–33] have also been developed. For a long time, little

attention had been given to the oxidation of primary amines probably due to poor product selectivity [34]. The discovery of oxidative catalytic systems that operate effectively with molecular oxygen or air as the sole oxidant has contributed to revitalize interest in this area [35]. Recently, non-precious transition-metal catalysis [36–42], metal-free catalysis [43–51], bioinspired catalysis [47–51], and photocatalysis [52–59] have led to homo-coupled imines in high yields and selectivity. However, these coupling reactions suffer from two major draw backs that limits the synthetic applications of imines. Firstly, most of these reactions are performed in solvent media. Solvents are perhaps the most crucial parameters in industrial chemical synthesis; they represent an important challenge because solvents often account for the vast majority of mass waste in the processes [60]. Secondly, the condensation reactions of amines are not efficient methods to prepare cross-coupled imines due to the intrinsic self-coupling properties of the substrates [36, 43, 61, 62]. To the best of our knowledge, only a few efficient and selective (>95 %) oxidative cross-coupling protocols for diverse amines have been reported to date [48, 63–65].

In the course of our research on the development of new methods for nitrogen heterocycles syntheses [3, 4], we found that N-benzylidenebenzylamines can readily be obtained from the iron-catalyzed oxidative self-condensation (homo-coupling) of benzylamines. We then, intended to explore a generalized method for the preparation of various functionalized imines. Herein, we report an efficient solvent-free process for the synthesis of imines by oxidative self and/or cross-condensation of primary amines using iron(II) bromide as catalyst and molecular oxygen as sole oxidant.

2 Results and Discussion

The oxidative self-condensation of benzylamine was chosen as a model reaction to optimize the reaction conditions (Table 1). Taking into account the observations made in the oxidative condensation of benzylamines with o-phenylenediamines [3], a reagent system of iron(II) bromide, molecular oxygen and chlorobenzene was tested (entry 1; Table 1). To our delight, amine 1a self-coupled smoothly at 110 °C to give N-benzylidenebenzylamine (2a) in 90 % yield. Comparing with many other transition metal-catalyzed reactions [36–42], this reaction system was quite simple because no ligand or additives were required. A quick survey of different iron salts indicated that iron(II) bromide was superior to other catalysts (entries 2–10; Table 1). Strikingly, the yields of imine 2a were slightly higher in solvent-free conditions than the chlorobenzene solution by simply testing for some iron salts (entries 2,

7,10). Gratifyingly, the iron(II) bromide reaction in the absence of solvent produced the highest yield (97 %, entry 11; Table 1). The yield of imine 2a was decreased when the reaction was carried out in air atmosphere or at low temperature due to the incomplete reaction (entries 12, 13; Table 1). Investigation of the catalyst loading showed that 5 mol% of iron(II) bromide was required. The yield of 2a was reduced to 71 % when 2 mol% iron(II) bromide was used (entry 14; Table 1). In the absence of iron catalyst, the reaction proceeded very slowly, and the conversion of benzylamine to N-benzylidenebenzylamine reached 53 % in seven days (entry 15; Table 1). Furthermore, the reaction failed to give the desired imine compound when the process was carried out under a N_2 atmosphere. The best catalytic activity of $FeBr_2$ under solvent-free conditions, compared with the other iron salts, is possibly associated with the fact that the bromide is a better leaving group than chloride. This leads to a vacant coordination site on iron(II), thus allowing easy coordination of substrate to the iron(II) centre. Furthermore, the catalytic system requires a one electron change in oxidation state from iron(II) to iron(III), which is more feasible than the conversion of iron(III) to iron(IV), thus making the catalyst iron(III) bromide less useful than iron(II) bromide. Therefore, we decided to perform the subsequent reactions of the benzylamines in the presence of iron(II) bromide as catalyst (5 mol%) and molecular oxygen as oxidant at 110 °C under solvent-free conditions.

The scope of the oxidative self-condensation of benzylamines under optimized conditions is outlined in Table 2. Substituents at the *ortho*, *meta*, and *para* positions of the phenyl ring of the amine (entries 1–7; Table 2) are easily accommodated. The reaction is also tolerant of electronic perturbation: oxidative self-condensation of benzylamines bearing electron-donating (entries 1–3; Table 2) and electron-withdrawing substituents (entries 4–7; Table 2) proceeded in excellent yields in short times. Functional groups such as methoxy (entry 2; Table 2), acetal (entry 3; Table 2), halogens (entries 4–6; Table 2), and trifluoromethyl (entry 7; Table 2) were shown to be highly compatible with the present reaction conditions. It is noteworthy that the halogen-substituted benzylamines performed well, leading to the halogen-substituted imine compounds (entries 4–6; Table 2), which could be used for further transformations along with the imine functionality [66]. Sterically hindered substrate such as (±)-α-methylbenzylamine also underwent efficient oxidative self-condensation (entry 8; Table 2). Furthermore, the bulkier 1-naphthalenemethylamine could be used as the substrate, leading to formation of the corresponding imine product 2j in 89 % yield (entry 9; Table 2). Heterocyclic methylamines namely furfurylamine and 2-thiophenemethylamine, could also be converted into the corresponding

Table 1 Screening of catalysts and reaction conditions for the oxidative self-condensation of benzylamine
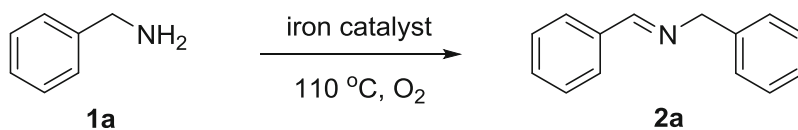
Entry	Catalyst	Solvent	Time (h)	Yield (%)[a,b]
1	FeBr$_2$	chlorobenzene	6	90
2	FeCl$_2$	chlorobenzene	24	76 (81)
3	FeF$_2$	chlorobenzene	24	51
4	Fe(OAc)$_2$	chlorobenzene	24	39
5	FeSO$_4$ ·7H$_2$O	chlorobenzene	24	trace
6	Fe(ClO$_4$)$_2$·xH$_2$O	chlorobenzene	24	67
7	FeBr$_3$	chlorobenzene	24	58 (60)
8	FeCl$_3$	chlorobenzene	24	43
9	Fe(acac)$_3$	chlorobenzene	24	24
10	Fe(NO$_3$)$_3$	chlorobenzene	24	71 (75)
11	FeBr$_2$	–	4	97
12[c]	FeBr$_2$	–	24	74
13[d]	FeBr$_2$	–	24	59
14[e]	FeBr$_2$	–	24	71
15	–	–	7 days	53

Reaction conditions: **1a** (3.0 mmol), catalyst (5 mol %), chlorobenzene (2 ml), 110 °C, molecular oxygen (O$_2$ balloon) atmosphere unless otherwise noted

[a] Yield of product after chromatography

[b] Values in parenthesis belongs to the yields at solvent-free conditions

[c] Reaction under air atmosphere

[d] Reaction was performed at 80 °C

[e] Using 2 mol % of catalyst

self-coupled imines in high yields indicating a good tolerance of heteroatoms (entries 10, 11; Table 2). In addition, aliphatic amines such as *n*-octylamine and cyclohexylamine were quite effective in the reaction, affording the desired products **2m** and **2n** in 53 and 61 % yields, respectively (entries 12, 13; Table 2). Interestingly, FeBr$_2$/O$_2$ catalytic system could efficiently oxidize the secondary amines viz, dibenzylamine and 1,2,3,4-tetrahydroisoquinoline to the corresponding imines in high yields (92–95 %), which represents the versatility of the present reaction protocol (entries 14, 15; Table 2).

Recently, Xu and co-workers reported Fe(NO$_3$)$_3$/TEMPO-catalyzed aerobic oxidation of amines to imines in toluene with 5–10 mol% catalyst loading.[40] This [Fe]/TEMPO catalyst system could oxidize benzylic amines to imines efficiently, but insignificant reaction or failed to oxidize hetero aryl methylamines and aliphatic amines. Moreover, in this catalytic system the reaction efficiency was largely affected by substituents on the phenyl ring of amine, which is not observed in our method. Also, for the

oxidation of *ortho*-substituted benzylamines, the yields for the imine products were not notable. However, a longer reaction time for all the conversions was required compared with the system reported here. Besides the above limitations, an expensive TEMPO was used as a co-catalyst, which is not recovered and reused.

Encouraged by excellent yields of the oxidative self-condensation of benzylamines, our efforts were next focused to explore the scope of cross-condensation between benzylamines **1** and primary amines **3** (Table 3). First, the reaction between various benzylamines and aniline was investigated under standard conditions (entries 1–6; Table 3). All the cross-condensation reactions were carried out with one molar equivalent of benzylamines and 1.2 molar equivalents of primary amines. To our delight, benzylamines reacted smoothly with aniline furnished the *N*-benzylideneanilines **4a–f** in good to excellent yields. As previously reported by our group [3], it is likely that the self-condensation products *N*-benzylidenebenzylamines were formed first and then reacted with aniline to form *N*-benzylideneanilines by

Table 2 Iron-catalyzed oxidative self-condensation of amines

Entry	Substrate 1	Time (h)	Product 2		Yield[a] (%)
1	Me—C6H4—CH2NH2	4	Me—C6H4—CH=N—CH2—C6H4—Me	2b	97
2	MeO—C6H4—CH2NH2	4	MeO—C6H4—CH=N—CH2—C6H4—OMe	2c	98
3	(methylenedioxyphenyl)—CH2NH2	5	(methylenedioxyphenyl)—CH=N—CH2—(methylenedioxyphenyl)	2d	97
4	F—C6H4—CH2NH2	5	F—C6H4—CH=N—CH2—C6H4—F	2e	95
5	Cl,Cl—C6H3—CH2NH2	3.5	Cl,Cl—C6H3—CH=N—CH2—C6H3—Cl,Cl	2f	95
6	Cl—C6H4—CH2NH2	7	Cl—C6H4—CH=N—CH2—C6H4—Cl	2g	93
7	F3C,CF3—C6H3—CH2NH2	5	F3C,CF3—C6H3—CH=N—CH2—C6H3—CF3,CF3	2h	90
8	Ph—CH(CH3)—NH2 (±)	12	Ph—C(CH3)=N—CH(CH3)—Ph	2i	94
9	naphthyl—CH2NH2	8	naphthyl—CH=N—CH2—naphthyl	2j	89
10	furyl—CH2NH2	9	furyl—CH=N—CH2—furyl	2k	88
11	thienyl—CH2NH2	8	thienyl—CH=N—CH2—thienyl	2l	92
12	CH3(CH2)6CH2NH2	18	CH3(CH2)6CH=N—CH2(CH2)6CH3	2m	53
13	cyclohexyl—NH2	18	cyclohexylidene=N—cyclohexyl	2n	61
14	Ph—CH2—NH—CH2—Ph	3	Ph—CH=N—CH2—Ph	2a	95
15	tetrahydroisoquinoline (NH)	6	dihydroisoquinoline (N)	2o	92

Reaction conditions: amine **1** (3.0 mmol), FeBr$_2$ (5 mol%), molecular oxygen (O$_2$ balloon) atmosphere, 110 °C

[a] Isolated yield

Table 3 Iron-catalyzed oxidative cross-condensation of primary amines

$$R^1 \diagdown NH_2 \quad + \quad H_2N{-}R^2 \quad \xrightarrow[\text{O}_2,\ 110\ ^\circ\text{C}]{\text{FeBr}_2\ (5\ \text{mol }\%)} \quad R^1 \diagup N \diagdown R^2$$

$$\mathbf{1} \qquad\qquad \mathbf{3} \qquad\qquad\qquad\qquad\qquad \mathbf{4}$$

Entry	R^1 (1)	R^2 (3)	Time (h)	Yield (%)^a (4)
1	C_6H_5	C_6H_5	6	95 **4a**
2	4–MeC_6H_4	C_6H_5	6	95 **4b**
3	4–$MeOC_6H_4$	C_6H_5	6	99 **4c**
4	4–FC_6H_4	C_6H_5	10	92 **4d**
5	2–ClC_6H_4	C_6H_5	12	89 **4e**
6	3,4–$Cl_2C_6H_3$	C_6H_5	8	94 **4f**
7	C_6H_5	4-MeC_6H_4	6	96 **4g**
8	C_6H_5	2-MeC_6H_4	6	91 **4h**
9	C_6H_5	4-$iPrC_6H_4$	5	95 **4i**
10	C_6H_5	4-$MeOC_6H_4$	5	97 **4j**
11	C_6H_5	4-FC_6H_4	8	90 **4k**
12	C_6H_5	4-ClC_6H4	8	86 **4l**
13	C_6H_5	4-BrC_6H_4	8	85 **4m**
14	C_6H_5	3,5-$Cl_2C_6H_3$	9	80 **4n**
15	C_6H_5	1-naphthyl	12	91 **4o**
16	C_6H_5	n-octyl	18	78 **4p**
17	C_6H_5	n-butyl	18	69 **4q**
18	C_6H_5	cyclohexyl	18	75 **4r**

Reaction conditions: amine **1** (3.0 mmol), amine **3** (3.6 mmol), FeBr$_2$ (5 mol %), molecular oxygen (O$_2$ balloon) atmosphere, 110 °C

^{a} Isolated yield

transimination. The yields of the reactions of benzylamines bearing electron-donating groups with aniline were slightly higher in comparison with electron-withdrawing amines.

Next, we turned our attention to study the effect of the reactions of benzylamine with various primary amines under standard conditions (entries 7–18; Table 3). In these reactions, the electronic properties of groups on the phenyl ring of anilines had some effects on the catalytic activities. Both electron-donating (e.g., Me, iPr, OMe) and electron-withdrawing (e.g., F, Cl, Br) substituents were tolerated in the reaction. Generally, anilines having an electron-donating substituent on the phenyl moiety gave slightly higher yield of cross-coupled imine products than the electron-withdrawing ones (entries 7–15; Table 3). This is likely due to the reduced basicity of the anilines containing electron-withdrawing groups which makes the transimination step sluggish. Steric effects of substituents had little effect on the yield of the reaction. For example, the reaction of 4- and 2-methylanilines with benzylamine resulted in the formation of *N*-benzylidene-4-methylaniline (**4g**) and *N*-benzylidene-2-methylaniline (**4h**) in 96 and 91 % yields, respectively (entries 7, 8; Table 3). Furthermore, aliphatic amines such as *n*-octylamine, *n*-butylamine and

cyclohexylamine were performed well in the cross-condensation reaction, furnished the corresponding imines **4p–r** in 78, 69 and 75 % yields, respectively (entries 16–18; Table 3).

To examine the feasibility of a large-scale experiment, the reaction of 4-fluorobenzylamine with aniline was investigated. The reaction could afford 1.81 g of imine **4d** in 91 % yield without any significant loss of its efficiency (Scheme 1). Therefore, this protocol could be used as a practical method to synthesize the precursors of some important biologically active molecules, such as triaryl-1,2,4-oxadiazole **5**, which shows significant inhibitory cytotoxicity against MCF7 and K562 cell lines [67].

To explore the reaction mechanism, the following experiments were performed as shown in Scheme 2. Benzylamine treated with iron(II) bromide and molecular oxygen to provide homo-coupled imine **2a** in 97 % yield (Scheme 2A). Whereas, only a trace amount of **2a** was observed in the absence of Fe source (Scheme 2B). This experiment demonstrates the essential role of the iron catalyst for this reaction. Next, the reaction of imine **2a** with aniline in the presence of iron catalyst afforded the cross-coupled imine **4a** in 98 % yield in 3 h (Scheme 3A).

Scheme 1 The large-scale reaction for the preparation of N-(4-fluorobenzylidene)aniline
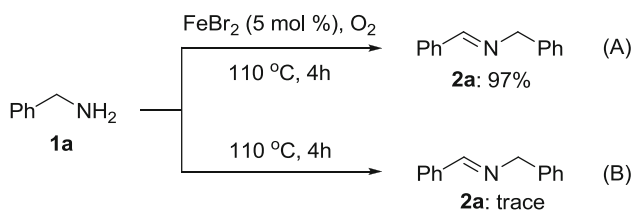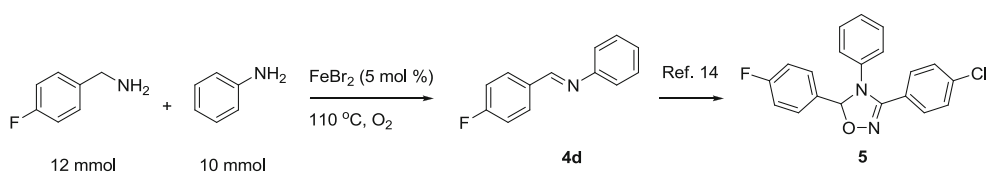
Scheme 2 Oxidative self-condensation of benzylamine

Unfortunately, the transimination reaction to form imine **4a** did not occur in the absence of catalyst in 3 h (Scheme **3**B). However, after a prolonged reaction time (40 h), investigating the reaction mixture showed that the imines **2a** and **4a** are present in 3:1 ratio by ^{1}H NMR analysis (Scheme **3**B). Similarly, the transimination of **2a** with *n*-

octylamine in the presence of FeBr$_2$ led to the corresponding cross-coupled imine in 57 % yield in 12 h, whereas no cross-imine compound was detected in the absence of catalyst at the same interval. Thus, the iron catalyst would be essential to promote both the self- and cross-condensation reactions of amines. We presumed that the cross-condensation reaction take place through the homo-coupled imine.

According to the above observations, a tentative mechanism for the formation of homo and cross-coupled imines was proposed based on previous reports in the literature [3, 63–65]. As shown in Scheme **4**, the first step may involve the oxidative addition of iron to the amine forming the complex **6**. Further oxidation of **6**, provides benzylimine (**7**) and regenerate the iron(II) bromide for

Scheme 3 Reaction of N-benzylidenebenzylamine with aniline
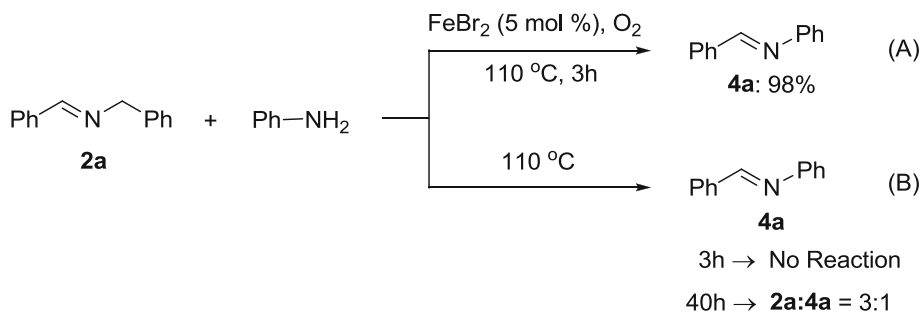
Scheme 4 Proposed reaction mechanism for the iron-catalyzed oxidative condensation of primary amines to imines
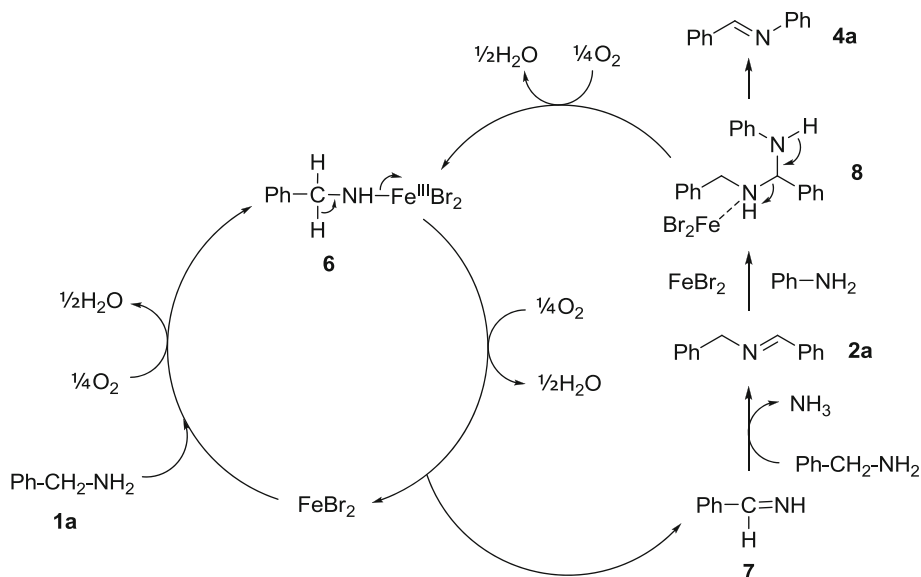
catalytic cycle. Reaction of imine **7** with another benzylamine leads to the homo-coupled product **2a** with liberation of ammonia. Next, the nucleophilic addition of aniline to imine **2a** takes place in the presence of iron catalyst to give iron-diamine complex **8**. Finally, diamine complex **8** breaks down to the cross-imine product **4a** and iron-amine complex **6** (regenerated).

3 Conclusions

In summary, a practical and convenient synthetic method in solvent-free conditions using iron(II) bromide as the catalyst (5 mol%) and molecular oxygen as the oxidant has been developed for the facile synthesis of functionalized homo- and cross-coupled imines from primary amines. The operational simplicity, broad substrate scope, and excellent yields of the products are the main advantages of this method. The tolerance of diverse functional groups makes the present protocol more attractive. Furthermore, this procedure is cheap, safe and environmentally benign.

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