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Fe/S-Catalyzed Synthesis of 2-Benzoylbenzoxazoles and 2-Quinolylbenzoxazoles via Redox Condensation of *o*-Nitrophenols with Acetophenones and Methylquinolines

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Fe/S catalyst generated *in situ* from FeCl₂•4H₂O and elemental sulfur S₈ in the presence of a tertiary amine as a base was found to catalyze efficiently a 6e⁻ redox condensation of *o*-nitrophenols with acetophenones and methylquinolines. The condensed products 2-benzoyl benzoxazoles and 2-quinolyl benzoxazoles were obtained in reasonable yields with water as the only byproduct at temperature as low as 80 °C.

2-Benzoylbenzoxazoles¹ and 2-quinolylbenzoxazoles represent versatile building blocks for building diverse molecular scaffolds, including bioactive molecules I–III² and fluorescence probes IV and V.³ In fact, I and III were identified to be an antiproliferative agent,^{3c} while II was found as a potent inhibitor of fatty acid amide hydrolase (Scheme 1).^{2a}

Consequently, a number of synthetic methods have been developed. Benzoxazole functionalization allows access to complex derivatives from simpler starting materials such as 2H-benzoxazoles,⁴ benzoxazoles 2-substituted⁵ by suitable activating groups or 2-benzyl benzoxazoles.⁶ The benzoxazole ring could be created from 2-aminophenols via non redox condensation,⁷ oxidative coupling⁸ or cyclization⁹ reactions. In parallel, a condensation between two starting materials proceeding with change in oxidation states and removal of small molecules, i.e. redox condensation, is an interesting strategy to benzoxazole cores. Beifuss suggested an original approach to 2-benzoylbenzoxazole based on redox condensation of *o*-nitrosophenols which act as oxidant components with 2-bromoacetophenones as reducing components (Scheme 2).¹⁰

This base-promoted reaction proceeded without any added reducing or oxidizing agent. However, the main drawbacks of this approach are limited availability of both starting substrates, especially *o*-nitrosophenols along with lachrymatory nature of phenacyl bromides.

Scheme 1. 2-Benzoyl and 2-Quinolyl Benzoxazoles

In general, all known methods for the synthesis of 2-benzoylbenzoxazole require substrates that are more or less functionalized with activating or protecting groups, the efficiency of the global processes in terms of atom-, step-, and redox- economies could be further improved. For example, while 2-aminophenols are the precursor of choice for most benzoxazoles, they are generally obtained by 6e⁻ reduction of readily available *o*-nitrophenols 1. In an ideal scenario, a synthesis of 3 via redox condensation with 1 as an oxidizing partner would involve acetophenones 2 as a reducing partner. Such a shortcut approach would produce two molecules of water as the only byproduct.

In living systems, the redox transformations are frequently orchestrated by the metalloenzymes.¹¹ Among the most representative examples, iron–sulfur clusters, ubiquitous in cellular proteins, are best known for their involvement in a wide range of in the oxidation-reduction reactions.¹²

The last decades have seen a tremendous development in the understanding of the mechanism of these enzymes.¹³ However,

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their application in organic synthesis has so far not been adequately explored due to both structural and electronic complexity of these systems. Recently, the bioinspired Fe/S-based catalytic systems were developed for the redox condensation between nitro groups with different reducing substituents.^{14,15} Using iron sources such as metallic iron or simple iron salts and elemental sulfur as the sulfur source for *in situ* formation of Fe-S catalytic species, we and others have succeeded in condensing aromatic nitro compounds with methylhetarenes,^{14a} amines,^{14b,c} phenylacetic acids,^{14d} alcohol,^{14e} benzyl cyanides,^{14f} dibenzyl disulfides^{15a} and *p*-methylphenols,^{15b-d} enabling the synthesis of a wide range of nitrogen heterocycles.

Beifuss method: NO/CH₂ 4e⁻ redox condensation

drawbacks: - limited availability of *o*-nitrosophenols (stabilized by a 5-EDG)
- lachrymatory phenacyl bromides
- use of base to remove HBr

Our method: NO₂/CH₃ 6e⁻ redox condensation using bioinspired Fe/S catalyst

advantages + inexpensive, readily available substrates
+ user friendly substrates
+ water as the only byproduct
+ Fe/S catalyst from simple and inexpensive precursors

Fe/S clusters:

biological redox reactions

Scheme 2. Redox Condensation to Benzoxazoles 3

In continuing our work, we report here our extension to 6e⁻ redox condensation of **1** and **2** to **3**.

At the outset of this study, we chose *o*-nitrophenol **1a** and acetophenone **2a** as the model substrates. Their reaction in the presence of FeCl₂·4H₂O (10 mol %) and sulfur¹⁶ (0.5 mmol based on atomic sulfur) was found to provide the expected 2-benzoylbenzoxazole **3aa** in a moderate yield in the presence of *N*-methylpiperidine (1 equiv) as a base at 80 °C (entry 1). While other bases such as DIPEA, NEt₃ or NPr₃ were found to be efficient as an additive (entries 2-4, respectively), no reaction was observed with weaker nitrogen bases such as *N*-methylmorpholine (entry 5) or pyridine (entry 6) or in the absence of a basic additive (entry 7). FeCl₃·6H₂O also displayed a similar result (entry 8), which could be understood by the reversibility of the Fe²⁺/Fe³⁺ interconversion in the presence of both oxidizing and reducing agents. The reaction occurred even with lower iron or sulfur catalyst loading (entries 9-12) but was found to be totally inhibited in the absence of either iron salt or

sulfur (entries 13-14). Interestingly, the reaction was found to proceed at temperature as low as 60 °C despite a low yield (entry 15). Lower yield was observed at a higher reaction temperature (entry 16), possibly due to loss of both starting materials by evaporation. Under the optimized condition of catalysts and base additive, we found that pyridine as a second additive was shown to be beneficial (entry 17 vs entry 9). Although further investigation is needed to understand the role of pyridine, this base could act as a stabilizing ligand for Fe/S complex and thus enhance the catalytic activity.

Finally, for comparison purpose, Co(OAc)₂·4H₂O and Ni(OAc)₂·4H₂O were intendedly used in place of iron salt. While Co salt displayed a weak catalytic activity, Ni salt was shown to be completely inefficient, the reaction resulted in both unchanged starting materials.

Table 1. Screening of Redox Condensation Conditions

Entry ^a	x	y	additive	T (°C)	yield (%) ^b
1	10	1	<i>N</i> -methylpiperidine	80	50
2	10	1	DIPEA	80	63
3	10	1	NEt ₃	80	46
4	10	1	NPr ₃	80	40
5	10	1	<i>N</i> -methylmorpholine	80	0 ^c
6	10	1	pyridine	80	0 ^c
7	10	1	-	80	0 ^c
8 ^d	10	1	DIPEA	80	52
9	5	1	DIPEA	80	67
10	2	1	DIPEA	80	57
11	1	1	DIPEA	80	45
12	5	0.5	DIPEA	80	55
13	0	1	DIPEA	80	0
14	1	0	DIPEA	80	0
15	5	1	DIPEA	60	28
16	5	1	DIPEA	100	65
17	5	1	DIPEA:Pyridine ^e	80	72
18 ^f	5	1	DIPEA:Pyridine ^e	80	27
19 ^g	5	1	DIPEA:Pyridine ^e	80	0 ^c

^a Reaction conditions: *o*-nitrophenol **1a** (1 mmol), acetophenone **2a** (1.2 mmol), FeCl₂·4H₂O (x mol %) sulfur (y mmol, 32 mg/mmol), additive (1 mmol), 80 °C, 16 h.

^b Isolated yield. ^c Determined by ¹H NMR of the crude mixture. ^d FeCl₃·6H₂O was used in place of FeCl₂·4H₂O. ^e DIPEA (1 mmol), pyridine (1 mmol). ^f Co(OAc)₂·4H₂O was used in place of FeCl₂·4H₂O. ^g Ni(OAc)₂·4H₂O was used in place of FeCl₂·4H₂O.

Encouraged by this initial result, we have then explored the generality of the Fe/S-catalyzed redox condensation. Pleasingly, the optimized condition for the model reaction in Table 1, entry 17, could be applied for the redox condensation of 22 different methyl ketones **2b-2w** with *o*-nitrophenol **1a**. In all cases, the 2-acylbenzoxazole derivatives **3b-3aw** were obtained in moderate to high yields (Scheme 3). Substituents such as methyl, methoxy, halogens were found to be suitable to deliver the expected products **3ab-3al** in comparable yields to

unsubstituted acetophenone **2a**. It should be noted that the reactions with sterically demanding ketone such as **2c** or with sparingly soluble ketone **2g** required heating at higher temperatures (100 °C and 90 °C, respectively). We emphasized that ketone **3af**, which was previously found to display anti-proliferative property (compound **I**, Scheme 1) could be synthesized directly by our method.

Scheme 3. Scope of Methyl Aryl Ketones **2**

Ketones bearing electron withdrawing groups such as trifluoromethyl, CN, NO₂, methyl ester were also competent substrates. Acetyl derivatives of polyaromatic or heteroaromatic substrates such as 2-naphthyl **2t**, thiophene **2u**, furan **2v** and benzofuran **2w** exhibited similar reactivity, providing ketones **3at-3aw** in reasonable yields.

Next, we focused our attention on the reactions of acetophenone **2a** with different substituted *o*-nitrophenols **1b-1g** (Scheme 4). In general, our reaction conditions were found

to be nicely applicable to substrates bearing various functional groups including alkyl **1b-1c**, alkoxy **1d-1e**, halogen **1f-1h**, ester **1i** and pyridine analogue **1j**. It is noteworthy that unlike *o*-aminophenols,^{8f} the corresponding *o*-nitrophenols substrates were commercially available and readily obtained by direct nitration of the corresponding phenols.

Finally, we extended our reactions to 2-methylquinoline and analogues **4** as stable methyl iminic substrates (Scheme 5). Since they all contain a pyridine ring, their reaction involved DIPEA as the only additive. 2-Methylquinoline **4a** was found to be an excellent substrate capable of reacting with a wide range of *o*-nitrophenols **1a-1k**. Among the resulting products, **5da** and **5ga**, represent the direct precursor or core structure of fluorescent probes **V** and **IV** (Scheme 1). Compared to our previous catalytic system based on metallic iron and sulfur which required high temperature up to 150 °C,^{14a} the present system using DIPEA as a base and FeCl₂·4H₂O as an iron source showed to be more reactive, allowing the reaction to proceed at only 80 °C.

Scheme 4. Scope of *o*-Nitrophenols **1**

Other isomer of **4a** bearing an active methyl group such as 4-methylquinoline **4b** and 1-methylisoquinoline **4c** were also competent substrates. On the other hand 2-methylquinoxaline **4d** reacted sluggishly under the standard conditions but provide the expected product **5ad**, which is a core structure of compound **III** presented in Scheme 1 as an anti-proliferative compound.

At this stage, although the detailed mechanism was not fully elucidated, we were eager to investigate some control reactions for a better understanding of the reaction pathway.

Since the phenol function was found to complex with Fe³⁺, we carried out experiments with a range of nitro substrates without an *o*-phenol function such as nitrophenol, *o*-methoxynitrophenol, *p*-nitrophenol with acetophenone **2a** (eq. 1). In all cases tested, the expected redox condensed products were not formed in detectable or significant amounts. This suggests the central role of the *o*-hydroxy group in the reaction, allowing the reaction to occur at a temperature as low as 80 °C.

Second, our optimized conditions were not applicable to aliphatic methyl ketones such as pinacolone, isopropyl methyl ketone and 2-pentanone (eq. 2). It is strongly possible that the reaction occurs via enol tautomer of the methyl ketone derivative **B** and such an intermediate is stabilized more effectively by an aryl group than an alkyl one.

Scheme 5. Scope of Methylquinolines **4**

Third, the redox condensation of **1a** and **2a** failed also when monosubstituted sulfide precursors such as dimethyl, dibenzyl or diphenyl disulfide as well as their thiol parents were used in place of elemental sulfur (eq. 3). It should be noted that, unlike these sulfur derivatives, elemental sulfur is capable of forming Fe/S clusters, suggesting the involvement of this kind of catalyst in the reactions.

Based on these observations as well as on our previous experiences on similar redox condensation, we tentatively proposed a reaction mechanism starting with the formation of Fe/S clusters **A** (in a simplified form without charge on iron atoms for clarity reason) from iron salt and sulfur. Although the exact structures of such catalytic species could not be determined in the reactions mixtures, the key features are that they contain many iron atoms capable of existing in a wide range of easily interchangeable oxidation states. These complexes are stabilized by both sulfur bridges and external sulfide ligands. Complexation of *o*-nitrophenolate and 2-mercaptoacetophenone (generated *in situ* from sulfur and acetophenone **2a**) with iron to form **B** followed by a cascade of intramolecular condensation of the methylene group to the nitro group would lead to nitrone **D** via **C**. Extrusion of benzoyl nitrone **E** from **D** will regenerate catalyst **A**. Deoxygenation of **E** with H₂S will provide the expected product **3aa**.

Reaction of 2-nitroaniline with acetophenone resulted in complex mixtures from which we could identify the presence of 2-phenylquinoxaline. Reaction of 2,2'-dinitrodiphenyl disulfide, a stable analogs of 2-nitrothiophenol, with acetophenone could proceed without iron catalyst to provide 2-benzoylbenzothiazole.¹⁷

Control experiments:

Proposed mechanism:

Scheme 6. Control Experiments and Proposed Mechanism

Conclusions

In conclusion, we have developed a direct access to 2-benzoylbenzoxazoles from 2-nitrophenols and acetophenones. Iron sulfur catalysts generated *in situ* from simple iron salts such as FeCl₂·4H₂O and elemental sulfur S₈ were found to be highly reactive, allowing the reactions to proceed at temperatures as low as 80 °C with high functional group tolerance for both substrates. Moreover, *o*-nitrophenols, acetophenones and methylquinoline analogues are inexpensive and readily available starting materials with high degree of structural diversity. Compared to known methods, our developed strategy is inarguably the cheapest and most convenient method to provide a library 2-benzoylbenzoxazoles and 2-quinolylbenzoxazoles in a green manner since water is the only by-product. We strongly believe that with suitable modifications, the Fe/S catalysts will find wider applications

with temperatures approaching that of physiological conditions. Such efforts are being pursued in our laboratory.

Conflicts of interest

There are no conflicts to declare.

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