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UV Assisted High-efficient Synthesis of α-Ketoamides Using Air Promoted by A Non-metal Catalyst in Aqueous Solution

Jianhui Li,[a]† Shaopo He,[a]† Kuan Zhang,[a]† Ziyi Quan,[a] Qiheng Shan,[a] Zhongliang Sun,[a] and Bo Wang*[a,b,c]

Dedication ((optional))

Abstract: Presented here is the first example of UV (λ = 210 nm) promoted procedure proceeding in aqueous media at r.t. using ambient air as the oxidant for efficient synthesis of an array of α-ketoamides of all types using a non-metal catalyst N-iodosuccinimide with a loading of 20mol%. With UV, oxygen in the air was efficiently utilized as the green oxidant, some control experiments were carried out and a plausible mechanism was proposed, disclosing that in aqueous solution, the oxidation process was actually triggered by dioxygen radical anion ($O_2^{\cdot-}$), while not molecular oxygen. A variety of secondary amines and primary amines as well as ammonia were employed as the amine moieties, and the desired product primary-, secondary-, and tertiary α-ketoamides were afforded in good to excellent yields of up to 96%.

Introduction

α-Ketoamides and their related derivatives are a class of key structural skeletal scaffold in a plethora of natural products, as well as biologically relevant molecules useful precursors for functional group transformations.[1,2] Because of their wide applications, the development of practical approaches for synthesizing α-ketoamides has recently attracted considerable attentions, and there have been numerous methods developed for the synthesis of α-ketoamides starting from varied substrates, for instance, ethylarenes,[3] terminal alkenes,[4] terminal alkynes,[5] aryl methyl ketones,[6] aryl acetaldehydes,[7] 2-oxo aldehydes,[8] 1-arylethanols,[9] 2-arylethanols,[10] 2-oxo alcohols,[11] and *etc.* The most important factors in successful α-ketoamide synthesis are the oxidants and catalysts, including metal[12] and non-metal ones **(Figure 1)**.[13] For those procedures with metal catalysts, harsh reaction conditions are usually required, and for those using non-metal catalysts, in most conditions, large excess of oxidant were needed, and high costs and environmental problems are therefore associated when using metal catalysts or non-metal ones requiring large excess of chemical oxidants, for instance *tert*-butyl hydroperoxide (TBHP).

Molecular oxygen (air contains about 21% oxygen) is an ideal oxidant, and the utilization of ambient air for the construction of valuable oxygen-containing organic compounds is of greater interest over pure oxygen in both academia and industry due to its economic and environmentally benign properties, and also for the consideration of safety problems as the latter needs pressure vessels to transport and high-pressure reactor for reaction process.

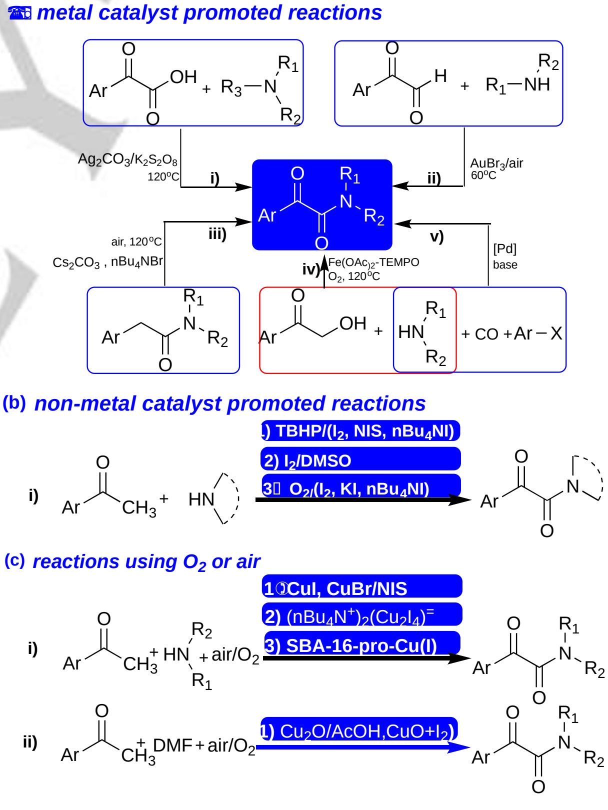

Figure 1. Selected examples of synthetic approaches for α-ketoamides

There have been several recent publications focused on molecular oxygen (air or O_2) oxidized protocols for the preparation of α-ketoamides from varied substrates[14,15] (**Figure 1**), though great achievements have been made, there still remains some to be solved such as involving metal catalyst, using large excess of chemical oxidants, limited substrate scopes (the majority of the developed methods could only produce one kind of α-ketoamide, tertiary α-ketoamides, some

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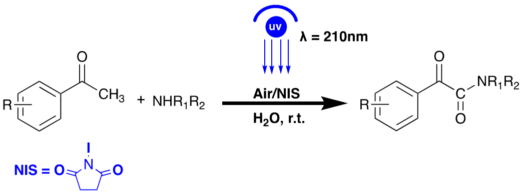
Supporting information for this article is given via a link at the end of the document.((Please delete this text if not appropriate)) could afford one or two types of α-ketoamides, secondary-or/and tertiary- α-ketoamides. The one applicable for all three types α-ketoamides (primary-, secondary- and tertiary- α-ketoamides), especially those proceeding in aqueous solution at mild conditions, has been less studied), the employment of organic solvent, and low product yields.

We have recently published some paper on the synthesis of α-ketoamides starting from different substrates.[3,13c,16] And as a continuous work on clean synthesis of α-ketoamide and inspired by the formation of ozone in the air with O_2 and UV light from the sun,[18] and the fact that ozone can decompose into O_2 and $O_2^{\cdot -}$ when meeting H_2O,[17] we envisioned an UV-assisted formation of ozone using air and further generation of $O_2^{\cdot -}$ in water, and the application of which for efficient and clean synthesis of α-ketoamides.



R = H, p-NO_2, p-CH_3, p-Cl, p-F, p-Br, m-CH_3, m-CH_3O, m-Br, o-Br, o-CH_3

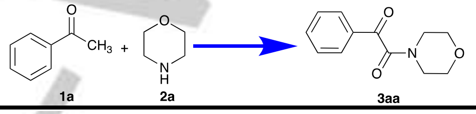
Scheme 1 UV-assisted aerobic oxidative of methyl ketones and amines or aqueous ammonia

Herein, we present a UV-assisted metal-free aerobic oxidative synthetic protocol for the preparation of an array of α-ketoamides in aqueous media, *N*-iodosuccinimide (NIS) was used as the catalyst, both primary- and secondary amines, and as well as ammonia (dissolved in water) were employed as the amine moieties. All reactions were proceeded smoothly well in water at room temperature (**Scheme 1).** To the best of our knowledge, this represents the first example of UV assisted metal-free approach proceeding in water at r.t. using air as the green oxidant to provide α-ketoamides of all types.

Results and Discussion

Initial evaluations for suitable reaction conditions were performed on a 2mL scale employing acetophenone (1a) and morpholine (2a) as the model substrates. Reaction parameters, such as solvent (H_2O, DMSO, ethanol (neat), toluene, and CH_3CN), catalyst (I_2, NIS, KI, and nBu_4NI), oxidant (air, and O_2) and temperatures (r.t., 40°C, and 50°C) were investigated and the results were summarized in **Table 1**. When reactions were carried out under N_2 (here we use N_2 to avoid the possible contact between air/O_2 and substrates to test whether NIS or I_2 can be used as the oxidant) firstly in water, both I_2 and NIS were tested. However, the proposed product **3aa** was not detected (**Entries 3,4**, Table 1) during or after reactions, indicating that both I_2 and NIS was incapable of oxidizing substrate **1a** to form product **3aa**, therefore they could not be used as the oxidant. When we replaced N_2 with air or O_2, and ran these reactions in varied organic solvents, to our interested, product **3aa** was detected and confirmed though not satisfied results were provided (**Entries 11-14**, Table 1), it should be noted that when the reaction was carried out in neat organic solvent ethanol (**Entry 12**, Table 1), only trace of product **3aa** were generated, however, when performing reaction in toluene, DMSO, or CH_3CN, poor yields of 25%, 12%, and 19% were provided respectively, it should be noted that these three organic solvents were used as purchased. Continuous work using water instead of organic solvent was performed and evaluated, to our satisfied that when use air as the oxidant, the proposed product 3aa was obtained in the yield of 96%, and with O_2, the yield was 97% (**Entry 8,9**, Table 1), a slight increase in product yield. For the consideration of meeting more principles of green chemistry and economic problems, air was chosen as the ideal oxidant (though a slight increase in product yield was achieved when using O_2) for further evaluations due to its abundant source and low cost. Higher temperatures were also tested, nevertheless, the respective lower yields of 87%, and 75% were obtained when carried out at 40°C and 50°C (**Entry 15,16**, Table 1), indicating that a higher temperature would not make positive contributions to product formation. Simply increasing the concentrations of substrates would not bring a better conversion, on the contrary, there was a slight decrease (from 87% to 82%) in the yield of product **3aa** (**Entry 17,18**, Table 1).

Table 1 Optimization of reaction conditions[a]

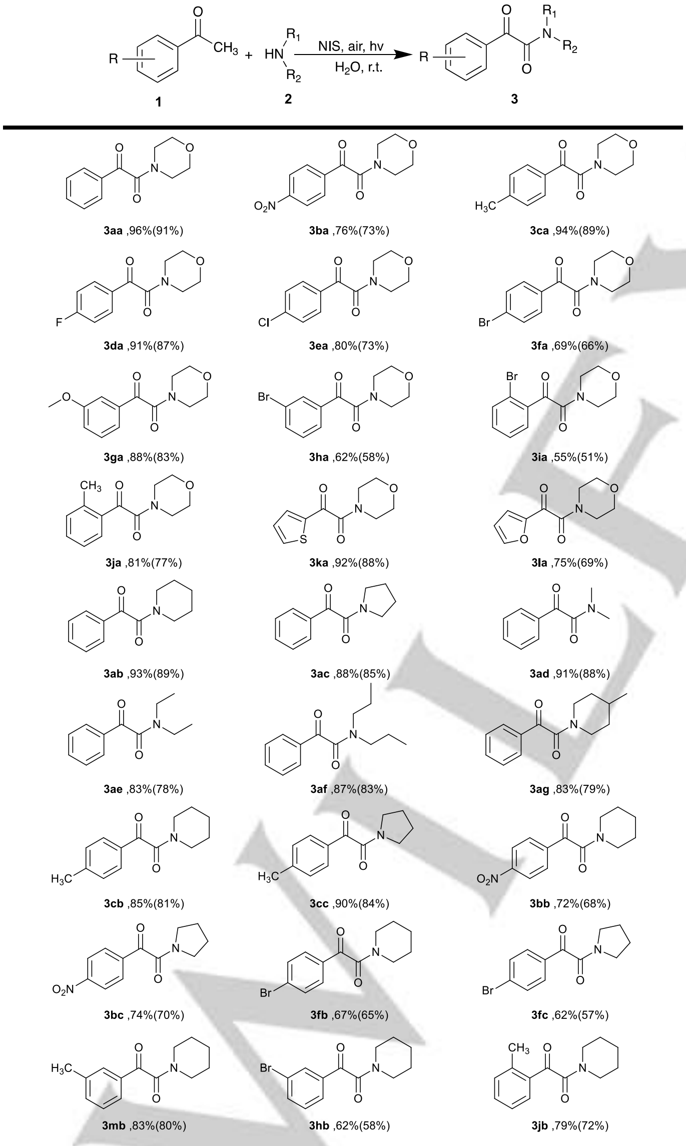


Entry	Catalyst	Oxidant	Solvent	Temp. (°C)	Yield (%)
1	I_2	O_2	H_2O	r.t.	31[b]
2	I_2	Air	H_2O	r.t.	25[b]
3	I_2	N_2	H_2O	r.t.	n.d.
4	NIS	N_2	H_2O	r.t.	n.d.
5	I_2	O_2	H_2O	r.t.	85
6	I_2	Air	H_2O	r.t.	82
7	nBu_4NI	Air	H_2O	r.t.	23
8	NIS	Air	H_2O	r.t.	96
9	NIS	O_2	H_2O	r.t.	97
10	KI	Air	H_2O	r.t.	27
11	NIS	Air	DMSO	r.t.	12
12	NIS	Air	C_2H_5OH	r.t.	trace
13	NIS	Air	Toluene	r.t.	25
14	NIS	Air	CH_3CN	r.t.	19
15	NIS	Air	H_2O	40	87
16	NIS	Air	H_2O	50	75
17	NIS	Air	H_2O	r.t.	87[c]
18	NIS	Air	H_2O	r.t.	82[d]

[a]Reaction conditions: 1a (0.1 mmol), 2a (0.25 mmol), catalyst (20 mol%), solvent (2 ml), reaction time, 16h. Yield was determined by HPLC analysis. [b]Reaction was carried out with no UV. [c]1a (0.5 mmol), 2a (1.25 mmol). [d]1a (1 mmol), 2a (2.5 mmol).

With all the optimized conditions in hand, some methyl ketones and secondary amines listed in Table 2 were subjected, and the results were summarized in Table 2. All these reactions were proceeded well and the desired product α-ketoamides were obtained with yields in good to excellent of up to 96%. Results indicated that in most conditions, when aryl methyl ketone bearing an electron-rich substituent(s) on aromatic ring, the corresponding secondary α-ketoamide could be afforded in better yields than those carrying an electron-deficient group(s) (**3ca, 3da, 3ga, 3ha, 3ia, 3ja, 3cb, 3bb,** Table 2). It seems that steric hinderance could also make negative affections on product yields, more steric hindered amines usually resulted in lower conversions (**3aa, 3ca, 3bb, 3bc, 3cb, 3cc,** Table 2). Two heterocyclic ketones were also tested to check the generality of this procedure, to our delighted, satisfied results were afforded (**3ka, 3la**, Table 2), the yields of product **3ka** and **3la** were 92% and 75%, respectively.

Table 2 Synthesis of tertiary α-ketoamides using secondary amine as the amine moiety[a]

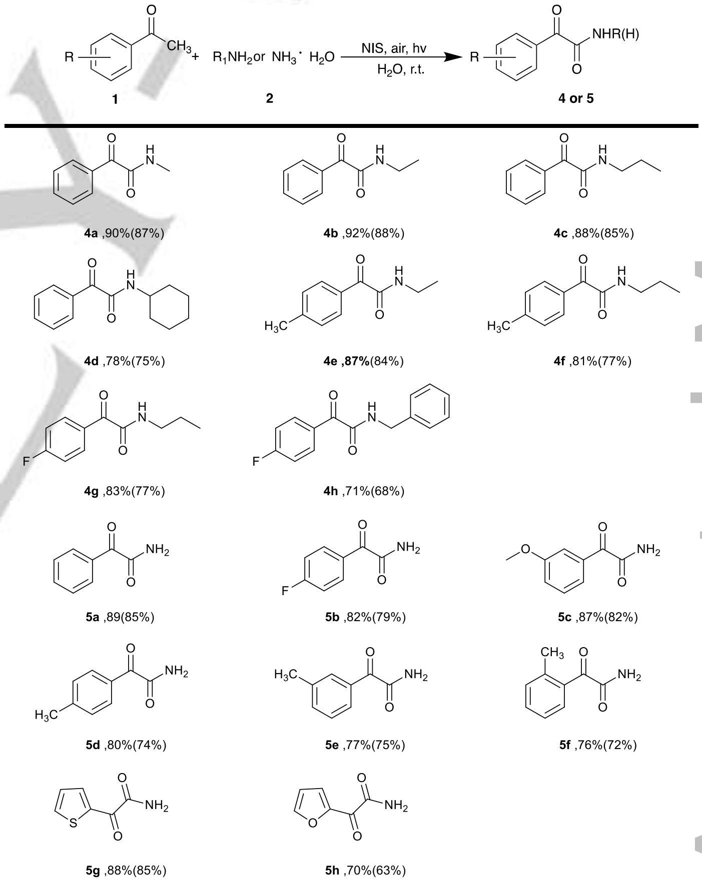


3aa ,96%(91%)
3ba ,76%(73%)
3ca ,94%(89%)
3da ,91%(87%)
3ea ,80%(73%)
3fa ,69%(66%)
3ga ,88%(83%)
3ha ,62%(58%)
3ia ,55%(51%)
3ja ,81%(77%)
3ka ,92%(88%)
3la ,75%(69%)
3ab ,93%(89%)
3ac ,88%(85%)
3ad ,91%(88%)
3ae ,83%(78%)
3af ,87%(83%)
3ag ,83%(79%)
3cb ,85%(81%)
3cc ,90%(84%)
3bb ,72%(68%)
3bc ,74%(70%)
3fb ,67%(65%)
3fc ,62%(57%)
3mb ,83%(80%)
3hb ,62%(58%)
3jb ,79%(72%)

[a]Conditions: 1a (0.1 mmol), 2a (0.25 mmol), catalyst (20 mol%) and water (2 ml), reaction time, 16h. Yields was determined by HPLC analysis, and the yields in brackets were isolated ones.

Encouraged by efficient synthesis of an array of tertiary α-ketoamides using secondary amines as the amine moieties, we would like to extend the possible scope of substrate by employing eight primary amines and ammonia. To the best of our knowledge, there has been limited examples for the synthesis of primary- (ammonia as the amine moiety) and secondary (primary amine as the amine moiety) α-ketoamides. For such purpose, some reactions were carried out under the same conditions. As expected, most of the reactions proceeded well and the results were summarized in Table 3. Results indicated that more steric hindered substrates usually resulted in lower product yields (**4a-h**, Table 3) when using ammonia, similar to those reactions for tertiary α-ketoamides synthesis, and when employ primary amines, in most conditions, similar results were provided as well. The generality of this procedure was also tested by employing two heterocyclic methyl ketones (**5g, 5h,** Table 3), and the corresponding products were afforded in the yields of 88% and 71%, respectively.

Table 3 Synthesis of secondary/primary α-ketoamides using methyl ketones and primary amines/ammonia[a].



4a ,90%(87%)
4b ,92%(88%)
4c ,88%(85%)
4d ,78%(75%)
4e ,**87%**(84%)
4f ,81%(77%)
4g ,83%(77%)
4h ,71%(68%)
5a ,89(85%)
5b ,82%(79%)
5c ,87%(82%)
5d ,80%(74%)
5e ,77%(75%)
5f ,76%(72%)
5g ,88%(85%)
5h ,70%(63%)

[a]Conditions: 1a (0.1 mmol), 2a (0.25 mmol), catalyst (20 mol%) and water (2 ml), reaction time, 16h. Yields was determined by HPLC analysis, and the yields in brackets were isolated ones.

Based on the above results and some relevant publications,[13a,14a-c,16] a plausible mechanism was proposed, as shown in **Figure 3**. With UV at λ = 210nm, molecular oxygen in the air was transformed to ozone (O_3), which then underwent gradual dissolution in aqueous media, and decomposed immediately upon meeting with hydroxyl anion (OH^-) to form superoxide radical anion ($O_2^{\cdot -}$), see **Figure 2**. Acetophenone **1** was attacked by amine to form the intermediate **3** in water, and the intermedia **3** reacted with NIS molecule to form the intermediate **4**. Then intermediate **5** was formed by the attack of $O_2^{\cdot -}$ and**,** then the cleavage of an O-O bond of **5** took place to form aryl glyoxal **6,** which react with amine **2** to give an intermediate **8**. Finally, the intermedia **8** was oxidized with O_2 promoted by NIS to fast form the desired product.

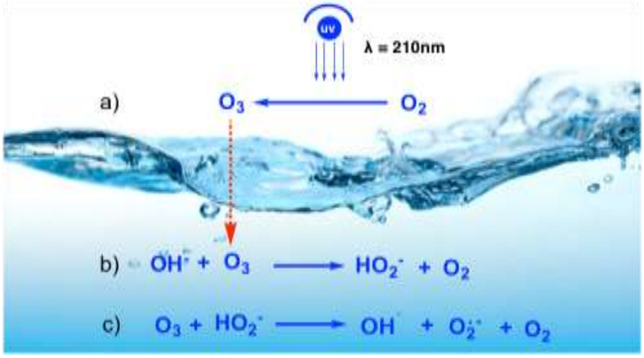


Figure 2. The formation of $O_2^{\cdot -}$ assisted by UV in water

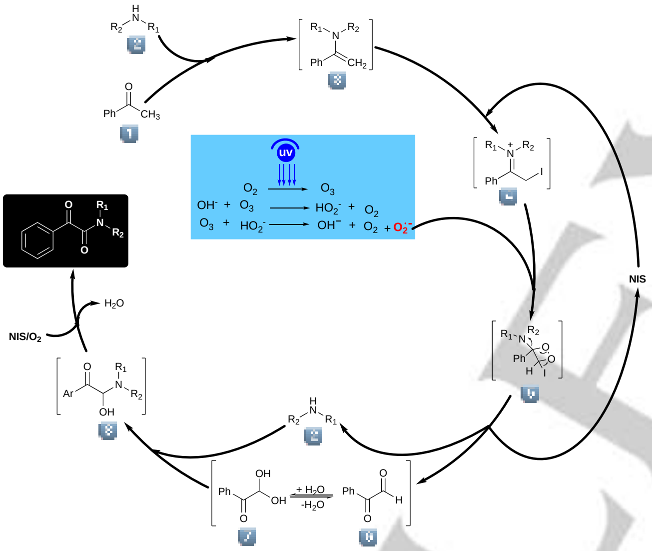
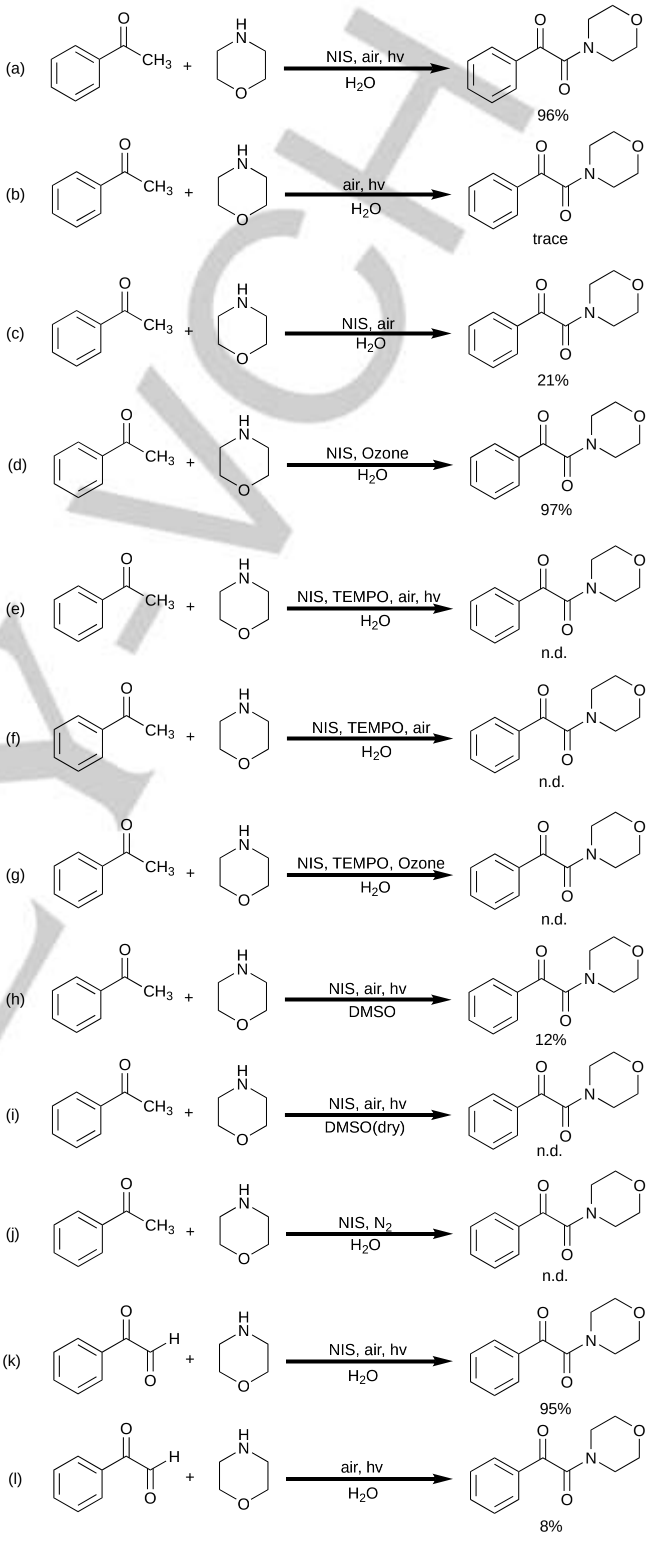


Figure 3. The proposed mechanism for UV promoted reactions

To verify the mechanism proposed here, twelve control experiments were carried out using acetophenone/aryl glyoxal and morpholine as the model substrates (**Scheme 2**). When reactions were carried out in the absence of NIS, there was basically no products were generated (**Entries a,b**, Scheme 2), and when with NIS, the yield was boosted to 96%, indicating that NIS was dispensable for successful transformation to the desired product, acting as the catalyst, while not as the oxidant. However, when UV was removed from the reaction system, the yield of product decreased from 96% to 21% (**Entry c**, Scheme 2), while the conversion was about 29%, there was still product formations occurred though there was more by-product generated. It was assumed that even without UV, there reaction could still be feasible, however it seems that they (with or without UV) were not the same mechanism as the latter provided less products along with more by-products. And then, we performed a reaction using ozone (Entry **d**, **Scheme 2**) as the oxidant, as expected, an almost identical yield (97%) of product **3aa** were obtained. And next, we used 2,2,6,6-



Scheme 2 Control experiments

tetramethylpiperidinooxy (TEMPO) to selectively capture the generated $O_2\cdot^-$ during reaction (Entries **e,f,g Scheme 2**) and misfunctioned it, to our excited, in all reactions with or without UV, or solo using ozone, we did not detect the formations of the desired product, indicating that the actual oxidant was ozone, and also the reaction was triggered by $O_2\cdot^-$, while not O_2, and another fact was that in the presence of NIS, the dissolved O_2 could be converted to $O_2\cdot^-$,[17] that is why even with no UV, no formation of O_3, there was still some products generated. And another proof was that, when we tried to conduct the reaction in organic solvent, for instance, dry DMSO, no product formation was observed (Entry **i**, **Scheme 2**), however, when use commercial dimethylsulfoxide (DMSO) as the solvent, a yield of 12% was detected (Entry **h**, **Scheme 2**), it was found that there was actually some water dissolved in DMSO with a moisture measuring instrument, proved that water played a vital role in the formation of $O_2\cdot^-$ from O_3, which was well agreed with the fact that O_3 was decomposed to $O_2\cdot^-$ when dissolved in water,[16] and the formed $O_2\cdot^-$ further react with substrate methyl ketones to form α-ketoamides**.** When use aryl glyoxal, intermediate **6** in the reaction mechanism, to react with morpholine, a high yield of 95% was detected (Entry **k**, **Scheme 2**), which means aryl glyoxal is the intermediate, indeed. These results agreed well with those from the optimization in **Table 1**.

Conclusions

In summary, we have developed an efficient metal-free approach to prepare an array of primary-, secondary-, and tertiary α-ketoamides using NIS as the catalyst and ambient air as the oxidant in water. Reactions could be proceeded smoothly well under UV at λ = 210nm at room temperature. Easily commercial obtainable ketones were as the keto moiety, and primary-/secondary- amines or aqueous ammonia are used as the amine moieties. The desired products could be provided in good to excellent yields of up to 96%. Some control experiments were carried out, which disclosed that water played an essential role in successful product formation, and the oxidation was actually initiated by dioxygen radical anion generated in water, while not molecular oxygen. Based on these results, we proposed a plausible mechanism to explain how the desired products formed. This protocol represents the first UV-assisted example with a broad substrate scope using non-metal catalyst and air for efficient and clean synthesis of primary-, secondary-, and tertiary α-ketoamides, and therefore is potential for practical and sustainably industrial applications. Further investigations into the application scope of UV triggered $O_2\cdot^-$ formation, $O_2\cdot^-$ formation in the presence of NIS from O_2, and the synthetic applications are undergoing in our lab.

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Keywords: UV • superoxide radical anion • α-ketoamide • air of 1atm • non-metal catalyst

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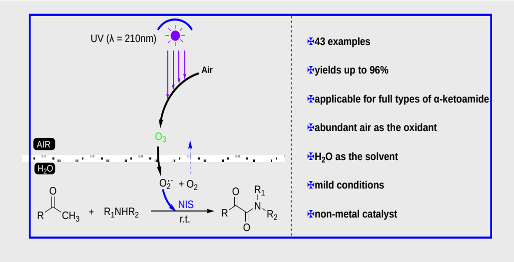
Entry for the Table of Contents (Please choose one layout)

Layout 1:

FULL PAPER

The first example of UV promoted procedure proceeding in aqueous media at r.t. using ambient air as the oxidant for efficient synthesis of an array of α-ketoamides of all types using a non-metal catalyst N-iodosuccinimide with a loading of 20mol%. A variety of α-ketoamides were afforded in good to excellent yields of up to 96%.



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UV Assisted High-efficient Synthesis of α-Ketoamides Using Air Promoted by A Non-metal Catalyst in Aqueous Solution