

I₂/叔丁基过氧化氢(TBHP)促进烯烃和 *N,N*-二甲基甲酰胺的氧化-酰胺化反应: 一种制备芳基- α -酮酰胺衍生物的简易方法

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摘要 以 *N,N*-二甲基甲酰胺(DMF)为溶剂和二甲胺源, 发展了用于合成芳基- α -酮酰胺衍生物的烯烃双官能化反应. 该过程采用 I₂/叔丁基过氧化氢(TBHP)为催化氧化体系, 一系列芳基- α -酮酰胺衍生物以良好的收率合成得到.

关键词 氧化-酰胺化; 烯烃; *N,N*-二甲基甲酰胺; 芳基- α -酮酰胺

I₂/*t*-Butylhydroperoxide (TBHP)-Mediated Oxo-amidation of Alkenes with *N,N*-Dimethylformamide: A Facile Access to Aryl- α -ketoamide Derivatives

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Abstract Difunctionalization of alkenes providing for the synthesis of aryl- α -ketoamide derivatives is developed by using *N,N*-dimethylformamide (DMF) as the solvent and the source of dimethylamine. This procedure is catalyzed by I₂ and *t*-butylhydroperoxide (TBHP) as oxidant. A wide range of aryl- α -ketoamide derivatives are generated in good yields.

Keywords oxo-amidation; alkenes; *N,N*-dimethylformamide; aryl- α -ketoamide

1 Introduction

α -Ketoamides are attractive in pharmacology because they are characteristically present in a wide range of bioactive molecules^[1]. Moreover, they serve as valuable synthetic intermediates in functional group transformations.^[2] Plenty of the strategies for the synthesis of α -ketoamides have been developed. Approaches for the synthesis of α -ketoamides mainly involve direct condensation of α -keto acids or α -keto acyl halides with amines,^[3] oxidations of α -hydroxyamides and α -aminoamides,^[4] dimethyl sulfoxide (DMSO)-mediated oxidative amidation of α -ketoaldehydes,^[5] oxidative amidation of aryl methyl ketones,^[6] *t*-butylhydroperoxide (TBHP)/I₂-promoted reactions of β -diketones with amines,^[7] and others.^[8] Nevertheless,

many of these methods suffer from complicated starting materials, multiple-step processes, and harsh conditions.

Alkenes, as one of the most useful chemical feedstocks in organic synthesis, possess the advantages of wide ubiquity, simplicity, and cheap cost. The vicinal difunctionalization of alkenes with two distinct functional groups in a single operation has become an important area due to its applicability to the pharmaceuticals, agrochemicals, and fine chemicals industries.^[9] This process could be carried out by metal or metal-free catalysis, reducing waste generation and purification activity, which often leads to a wide range of functional products with structural diversity, thus contributing to step and atomic economy. Oxo-functionalization such as oxo-halogenation, oxo-hydroxylation, oxo-nitrogenation, oxo-acyloxylation and oxo-amidation of alkenes is

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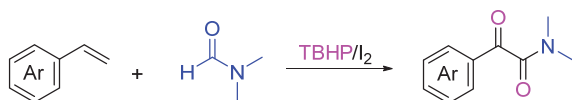
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an ideal and convenient method in the synthesis of α -functionalized carbonyl derivatives.^[10] *N,N*-Dimethylformamide (DMF) as a ubiquitous solvent, possesses the advantages of wide ubiquity and low cost. Moreover, DMF has been widely used as a functional reagent to introduce various functional groups, such as CHO, O, CO, CON(CH₃)₂, N(CH₃)₂, CN, and H moieties in organic chemistry.^[11]

In previous work, our group focused on the TBHP-mediated difunctionalization of alkenes for the construction of various complex difunctionalized molecules.^[12] We have also been investigating the function of DMF as an oxygen source, or the surrogate of CHO, and dimethylamine in organic chemistry.^[13] Thus, as the continuation of our work, we were interested in exploring the reaction of alkenes with DMF for the synthesis of aryl- α -ketoamides. Herein, we report an I₂/TBHP-mediated *oxo*-amidation protocol for the synthesis of aryl- α -ketoamides from aryl alkenes by using DMF as both the solvent and the source of dimethylamine (Scheme 1).



Scheme 1 *Oxo*-amidation protocol for the synthesis of aryl- α -ketoamides from aryl alkenes and DMF

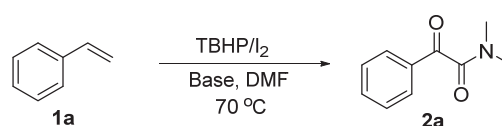
2 Results and discussion

Our study commenced with the reaction of styrene (**1a**), TBHP (4 equiv.) and Na₂CO₃ (1 equiv.) in the presence of iodine (0.1 equiv.) and DMF at 70 °C. As expected, the reaction afforded the desired product **2a** in 75% yield (Table 1, Entry 1). A series of base additives, including organic and inorganic ones, were investigated (Entries 1~5). Based on the in hand data, Na₂CO₃ was identified as amongst the best additive (Entry 1). In this approach, DMF was essential to the success formation of the product **2a**. To illustrate the role of DMF, a control experiment was carried out. Styrene (**1a**) reacted with 3 equiv. of DMF in the solvent of dioxane under the catalysis of TBHP/I₂, the main product **2a** could be observed with the yield of 30% (Entry 6). It illustrated that DMF was not only used as the reactant but also as the solvent. Furthermore, we observed a remarkable increase in the product yield with favourable yield of 88% when the amount of TBHP was increased to 6 equiv. (Entry 7).

With the optimized conditions in hand, the scope of the substrates was subsequently examined (Table 2). A variety of aryl olefins with substituents involving electron-donating and electron-withdrawing groups proceeded smoothly to give the desired products in moderate to good yields (**2b**~**2q**). It should be noted that group with proper electron absorption ability on the phenyl ring could facilitate the reaction (**2g** vs. **2h**). In addition, the position of substituent on the phenyl ring (**2i**, **2k**, **2l**) and the steric hindrance of the substituents (**2c**, **2d**) had no substantial effect on the reaction yields.

Based on our previous work and the reports from the li-

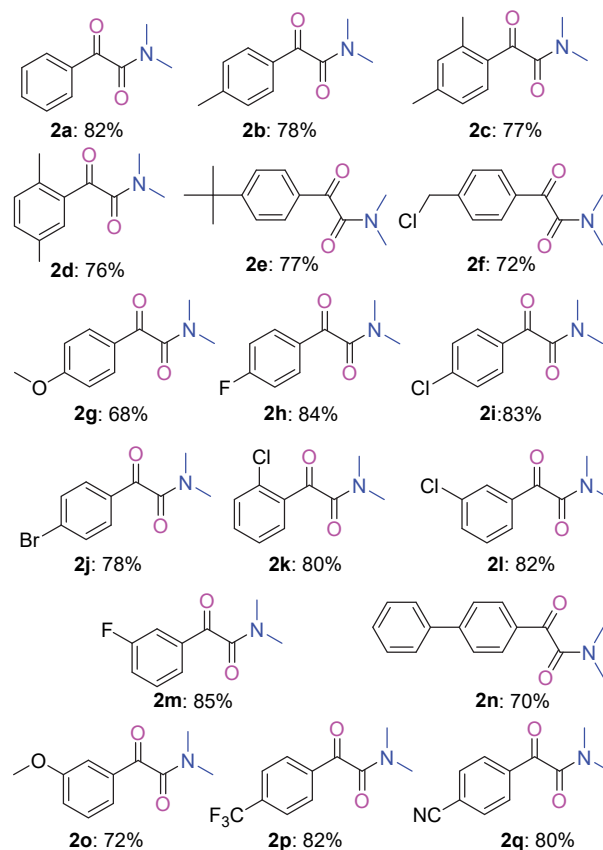
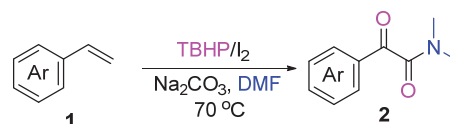
Table 1 Optimization of reaction conditions^a



Entry	Peroxide (equiv.)	Base	Solvent	Yield ^b /%
1	TBHP (4.0)	Na ₂ CO ₃	DMF	75
2	TBHP (4.0)	NaHCO ₃	DMF	72
3	TBHP (4.0)	<i>t</i> -BuOK	DMF	70
4	TBHP (4.0)	Et ₃ N	DMF	58
5	TBHP (4.0)	DBU	DMF	45
6 ^c	TBHP (4.0)	Na ₂ CO ₃	Dioxane	30
7	TBHP (6.0)	Na₂CO₃	DMF	88

^a Reaction conditions: **1a** (0.5 mmol), I₂ (0.1 equiv.), TBHP (4.0 equiv.), Na₂CO₃ (1.0 equiv.), solvent (2.0 mL), 70 °C for 24 h; ^b GC yield; ^c DMF (3 equiv.).

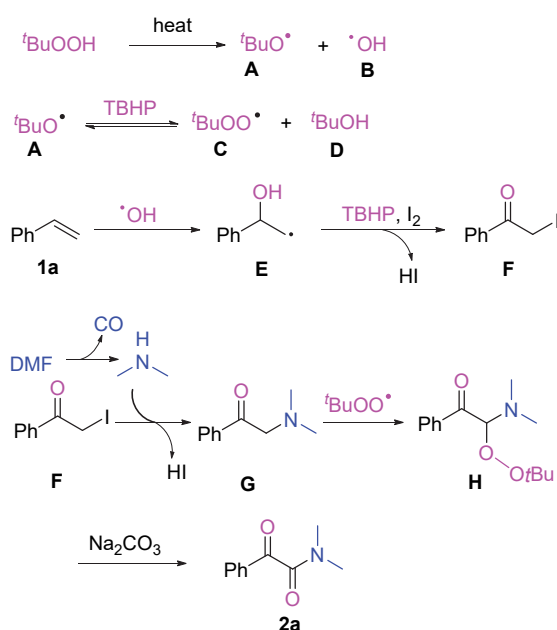
Table 2 Substrate scope for the synthesis of aryl- α -ketoamide derivatives^{a,b}



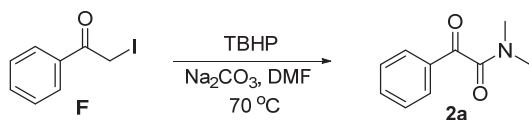
^a All of these reactions were carried out on a 2.0 mmol scale using DMF (2.0 mL) as a solvent. ^b Isolated yields.

terature, a plausible mechanism for the current *oxo*-amidation reaction was proposed as shown in Scheme 2. Initially, a hydroxyl radical and a *t*-butoxy radical **A** were

generated from a homolytic cleavage of TBHP under heat.^[14] The *t*-butoxy radical reacted with another molecule of TBHP to provide *t*-BuOO• (C).^[12c] Then, the hydroxy radical attacked on styrene to give intermediate E, which underwent further oxidation and iodination in presence of I₂/TBHP to give species F.^[15] In order to prove the existence of intermediate F, a control experiment was carried out. Intermediate F as the raw material, the reaction was conducted with TBHP in DMF, product 2a was observed with excellent yield of 97% (Scheme 3). Then, intermediate F reacted with dimethylamine, which was generated *in situ* from *N,N*-dimethylformamide^[16], to generate intermediate G. Intermediate G combined with *t*-BuOO• to afford the *tert*-butyl peroxide intermediate H,^[15] and finally followed by a Kornblum-DeLaMare rearrangement^[17] to give 2a in the presence of Na₂CO₃.



Scheme 2 Possible mechanism for the synthesis of 2a



Scheme 3 Control experiment of intermediate F with TBHP in DMF

3 Conclusions

In conclusion, we have developed a convenient one-pot protocol for the construction of a series of aryl- α -ketoamide *via* oxo-amidation of aryl alkenes. This transformation catalyzed by I₂ and oxidized by TBHP in DMF, which works not only as the solvent but also as the source of dimethylamine. The protocol permits the use of simple starting materials, mild reaction conditions, and wide substrate scopes and also offers operational simplicity. Further in-

vestigations into the oxo-amidation of aryl alkenes are currently underway in our laboratory.

4 Experimental section

4.1 General experimental information

All the reactions were carried out at room temperature for 24 h in a round-bottom flask equipped with a magnetic stir bar. Unless otherwise stated, all reagents and solvents were purchased from commercial suppliers and used without further purification. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker model Bruker AV-400 spectrometer in solutions of CDCl₃ using tetramethylsilane as the internal standard.

4.2 General procedure for the synthesis of aryl- α -ketoamide

A mixture of styrene (1a, 208 mg, 2.0 mmol), I₂ (51 mg, 0.2 mmol), TBHP (1.5 g, 12.0 mmol, 70% in water), and DMF (2.0 mL) was added successively in a round-bottom flask, and the resulting solution was stirred for 24 h at 70 °C. The mixture was purified by column chromatography on silica gel to afford product 2a with ether/ethyl acetate (*V* : *V* = 2 : 1) as the eluent.

N,N-Dimethyl-2-oxo-2-phenylacetamide (2a):^[18] 82% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.91 (d, *J* = 8.0 Hz, 2H), 5.9 (t, *J* = 8.0 Hz, 1H), 7.48 (t, *J* = 8.0 Hz, 2H), 3.08 (s, 3H), 2.92 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 191.8, 167.0, 134.7, 133.0, 129.6, 129.0, 37.0, 33.9.

N,N-Dimethyl-2-oxo-2-*p*-tolylacetamide (2b):^[19] 78% yield. ¹H NMR (400 MHz, CDCl₃) δ : 7.84 (d, *J* = 7.6 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.09 (s, 3H), 2.92 (s, 3H), 2.44 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 191.1, 167.4, 146.2, 131.3, 130.5, 129.8, 129.7, 37.1, 34.0, 21.7.

N,N-Dimethyl-2-(2,4-dimethylphenyl)-2-oxoacetamide (2c):^[19] 77% yield. ¹H NMR (400 MHz, CDCl₃) δ : 7.62 (d, *J* = 8.0 Hz, 1H), 7.15 (d, *J* = 7.6 Hz, 2H), 3.11 (s, 3H), 2.93 (s, 3H), 2.66 (s, 3H), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 192.8, 168.1, 144.8, 141.6, 133.4, 133.0, 128.9, 126.9, 37.1, 33.9, 21.9, 21.7.

N,N-Dimethyl-2-(2,5-dimethylphenyl)-2-oxoacetamide (2d):^[20] 76% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.43 (s, 1H), 7.28 (d, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 3.11 (s, 3H), 2.97 (s, 3H), 2.60 (s, 3H), 2.35 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 193.8, 167.9, 138.3, 135.8, 134.5, 132.9, 132.4, 131.3, 37.0, 34.0, 21.2, 20.7.

2-(4-*tert*-Butylphenyl)-*N,N*-dimethyl-2-oxoacetamide (2e):^[21] 77% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.84 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H), 3.07 (s, 3H), 2.92 (s, 3H), 1.30 (s, 9H); ¹³C NMR (CDCl₃, 100 MHz) δ : 191.5, 167.2, 158.8, 130.4, 129.6, 126.0, 37.0, 35.3, 33.9, 30.9.

2-(4-(Chloromethyl)phenyl)-*N,N*-dimethyl-2-oxoacetamide (2f): 72% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.94 (d, *J* = 8.0 Hz, 2H), 7.53 (d, *J* = 8.0 Hz, 2H), 4.62 (s, 2H), 3.11 (s, 3H), 2.95 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 191.0, 166.7, 144.1, 132.8, 130.0, 129.0, 45.1, 37.0, 34.0.

2-(4-Methoxyphenyl)-*N,N*-dimethyl-2-oxoacetamide

(**2g**):^[18] 68% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.90 (d, $J=8.0$ Hz, 2H), 6.96 (d, $J=8.0$ Hz, 2H), 3.88 (s, 3H), 3.09 (s, 3H), 2.94 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 190.4, 167.3, 164.8, 132.1, 126.1, 114.3, 55.6, 37.1, 33.9.

2-(4-Fluorophenyl)-*N,N*-dimethyl-2-oxoacetamide (**2h**):^[21] 84% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.97 (dd, $J=5.6, 8.4$ Hz, 2H), 7.16 (t, $J=8.4$ Hz, 2H), 3.09 (s, 3H), 2.94 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 190.2, 165.6 (d, ¹ $J_{C-F}=224.0$ Hz), 166.6, 132.5 (d, ³ $J_{C-F}=40.0$ Hz), 129.5 (d, ⁴ $J_{C-F}=12.0$ Hz), 116.3 (d, ² $J_{C-F}=88.4$ Hz), 37.0, 34.0.

2-(4-Chlorophenyl)-*N,N*-dimethyl-2-oxoacetamide (**2i**):^[19] 83% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.88 (d, $J=8.4$ Hz, 2H), 7.47 (d, $J=8.4$ Hz, 2H), 3.10 (s, 3H), 2.95 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 190.3, 166.4, 141.3, 131.4, 131.0, 129.3, 37.0, 34.0.

2-(4-Bromophenyl)-*N,N*-dimethyl-2-oxoacetamide (**2j**):^[21] 78% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.79 (d, $J=8.0$ Hz, 2H), 7.63 (d, $J=8.0$ Hz, 2H), 3.09 (s, 3H), 2.93 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 190.5, 166.4, 138.3, 132.3, 131.0, 130.1, 37.0, 34.0.

2-(2-Chlorophenyl)-*N,N*-dimethyl-2-oxoacetamide (**2k**):^[21] 80% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.84 (d, $J=7.6$ Hz, 1H), 7.49 (t, $J=8.0$ Hz, 1H), 7.42~7.36 (m, 2H), 3.06 (s, 6H); ¹³C NMR (CDCl₃, 100 MHz) δ : 190.1, 166.9, 134.3, 133.7, 133.3, 132.2, 130.7, 127.3, 37.0, 34.5.

2-(3-Chlorophenyl)-*N,N*-dimethyl-2-oxoacetamide (**2l**):^[18] 82% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.92 (s, 1H), 7.82 (d, $J=7.6$ Hz, 1H), 7.59 (d, $J=8.0$ Hz, 1H), 7.43 (t, $J=8.0$ Hz, 1H), 3.12 (s, 3H), 2.96 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 190.1, 166.2, 135.3, 134.7, 134.6, 130.3, 129.4, 127.8, 37.0, 34.1.

2-(3-Fluorophenyl)-*N,N*-dimethyl-2-oxoacetamide (**2m**):^[22] 85% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.69 (d, $J=8.0$ Hz, 1H), 7.62 (d, $J=8.8$ Hz, 1H), 7.50~7.45 (m, 1H), 7.33~7.29 (m, 1H), 3.09 (s, 3H), 2.94 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 190.2, 165.6 (d, ¹ $J_{C-F}=228.0$ Hz), 161.6, 130.7 (d, ³ $J_{C-F}=28$ Hz), 130.7 (d, ³ $J_{C-F}=32.0$ Hz), 125.6 (d, ⁴ $J_{C-F}=12.0$ Hz), 121.8 (d, ² $J_{C-F}=86.0$ Hz), 115.8 (d, ² $J_{C-F}=92.0$ Hz), 36.9, 34.9.

2-(4-Phenylphenyl)-*N,N*-dimethyl-2-oxoacetamide (**2n**):^[23] 70% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 8.02 (d, $J=8.0$ Hz, 2H), 7.73 (d, $J=8.0$ Hz, 2H), 7.62 (d, $J=8.0$ Hz, 2H), 7.49~7.41 (m, 3H), 3.15 (s, 3H), 3.00 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 191.4, 167.0, 147.5, 139.5, 131.8, 130.2, 129.0, 128.5, 127.6, 127.3, 37.1, 34.0.

2-(3-Methoxyphenyl)-*N,N*-dimethyl-2-oxoacetamide (**2o**):^[18] 72% yield. ¹H NMR (CDCl₃, 400 MHz) δ : 7.41 (s, 1H), 7.40 (d, $J=8.4$ Hz, 1H), 7.33 (t, $J=8.0$ Hz, 2H), 7.11 (d, $J=8.4$ Hz, 1H), 3.72 (s, 3H), 3.03 (s, 3H), 2.87 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 191.6, 166.9, 160.0, 134.2, 130.0, 122.6, 121.4, 112.8, 55.4, 36.9, 33.8.

N,N-Dimethyl-2-oxo-2-(4-(trifluoromethyl)phenyl)acetamide (**2p**):^[20] 82% yield. ¹H NMR (400 MHz, CDCl₃) δ : 7.69 (d, $J=8.1$ Hz, 1H), 7.54 (d, $J=8.0$ Hz, 1H), 3.14 (s, 2H), 2.98 (s, 2H); ¹³C NMR (101 MHz, CDCl₃) δ : 170.14, 139.88, 131.93, 131.64, 131.31, 131.00, 127.39, 125.54, 125.50, 125.46, 125.43, 125.10, 122.39, 39.40, 35.32.

2-(4-Cyanophenyl)-*N,N*-dimethyl-2-oxoacetamide (**2q**):^[24] 80% yield. ¹H NMR (400 MHz, CDCl₃) δ : 7.69 (d, $J=8.1$ Hz, 1H), 7.54 (d, $J=8.0$ Hz, 1H), 3.14 (s, 2H), 2.98 (s, 2H); ¹³C NMR (101 MHz, CDCl₃) δ : 169.53, 140.67, 132.35, 127.75, 118.17, 113.35, 39.36, 35.36.

Supporting Information The ¹H NMR and ¹³C NMR spectra of all products. The Supporting Information is available free of charge via the Internet at <http://sioc-journal.cn>.

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