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Electrochemical Synthesis of α-Ketoamides under Catalyst-, Oxidant-, and Electrolyte-Free Conditions

Jin-Yang Chen, Hong-Yu Wu, Qing-Wen Gui, Xiao-Ran Han, Yan Wu, Kui Du, Zhong Cao, Ying-Wu Lin, and Wei-Min He*

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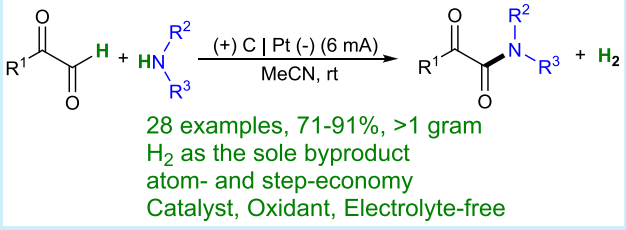
ABSTRACT: A catalyst-, oxidant-, electrolyte-free method for the preparation of α-ketoamides through the direct electrochemical amidation of α-ketoaldehydes and amines with innocuous hydrogen as the sole byproduct at ambient temperature was developed. The present reaction features clean and mild conditions, excellent functional-group tolerance, and high atom economy and scalability, enabling facile applications in pharmaceutical chemistry.

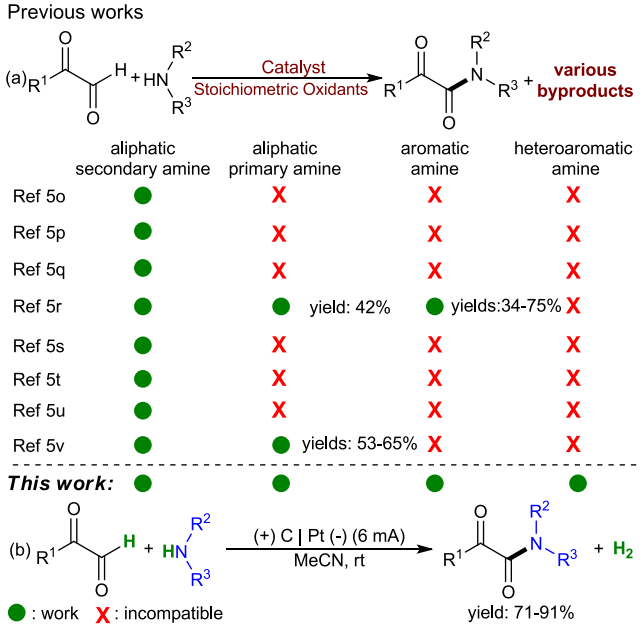


The continuous understanding of sustainable development has prompted chemists to minimize the generation of chemical waste in order to comply with the 12 principles of green chemistry.[1] Traditional redox reactions usually require the use of transition metal/organic catalysts (and/or relevant promoters) and a stoichiometric amount of oxidants to achieve an overall redox-neutral process, during which environmental hazardous waste is usually generated. Electrochemical synthesis, which solely uses sustainable electric current as a green and traceless reagent for driving the redox process, can efficiently avoid the usage of stoichiometric amounts of often harmful oxidants or reducing agents.[2] Therefore, electrochemical synthesis is considered to be sustainable, eco-friendly, and safe.[3]

α-Ketoamides not only are valuable structural motifs present in a broad range of natural products and synthetic pharmaceuticals but also are used as versatile intermediates and building blocks in organic synthesis.[4] In recent years, much effort has been dedicated to constructing such scaffolds.[5] The cross-dehydrogenation coupling (CDC) of α-keto aldehydes with amines is regarded as one of the most efficient and straightforward protocols.[5o−v] However, the practicability and atomic economy of these CDC reactions are impaired by the required catalysts, cocatalysts, and stoichiometric oxidants, because they inevitably lead to undesirable catalyst residues and chemical wastes (Scheme 1a). Moreover, most of these approaches do not work with primary amines, regardless if they are aliphatic or (hetero)aromatic ones.

Very recently, Ke and co-workers reported a novel method for the electrochemical synthesis of amide bond through radical amidation of carboxylic acids with amines.[6] However, both a strong base (Cs_2CO_3) and supporting electrolyte (tetrabutylammonium bromide) were needed for the electrochemical reaction to proceed. To the best of our knowledge, the direct electrochemical synthesis of α-ketoamides under base- and electrolyte-free conditions has never been reported. With our continuing interest in green organic synthesis,[7] herein, we disclose a clean and practical electrochemical cross-

Scheme 1. Amidation of α-Ketoaldehydes with Amines



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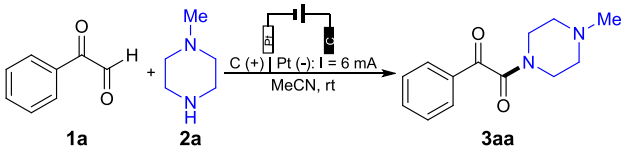
dehydrogenative coupling reaction of α-ketoaldehydes and amines at ambient temperature, by which a series of α-ketoamides could be synthesized under catalyst-, oxidant-, electrolyte-free and mild conditions (Scheme 1b). In addition, the reaction proceeded via an ionic mechanism and only generated innocuous hydrogen as the sole side product.

At the outset of the investigation, the reaction between 2-oxo-2-phenylacetaldehyde (**1a**) and 1-methylpiperazine (**2a**) was taken as the template reaction to optimize the reaction conditions (Table 1). The initial coupling reaction was

Table 1. Optimization of Reaction Conditions[a]



1a 2a 3aa

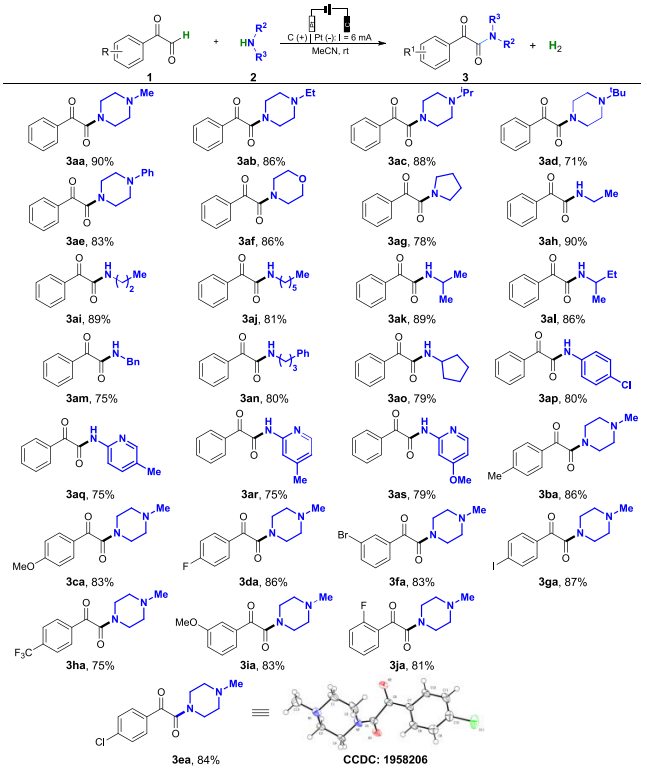
Entry	Variation from the standard reaction conditions	Yield (%)[b]
1	None	92
2	DMSO as the solvent	32
3	H_2O as the solvent	trace
4	C(+)|Ni(−) instead of C(+)|Pt(−)	68
5	C(+)|C(−) instead of C(+)|Pt(−)	36
6	Pt(+)|Pt(−) instead of C(+)|Pt(−)	85
7	RVC(+)|RVC(−) instead of C(+)|Pt(−)	64
8	*n*-Bu_4NBr	19
9	*n*-Bu_4NBF_4	16
10	$LiClO_4$	18
11	4 mA instead of 6 mA	75
12	8 mA instead of 6 mA	92
13	1.5 mmol of **2a** was used	92
14	50 °C instead of rt	92
15	No electric current	N.R.

[a]Conditions: Pt plate (15 mm × 15 mm × 0.3 mm) cathode, graphite rod anode (Φ 6 mm), constant current = 6 mA, **1a** (0.5 mmol), **2a** (0.5 mmol), MeCN (8 mL), in air, rt, 23 h, undivided cell. [b]Estimated by GC-MS. NR: no reaction.

conducted in an undivided cell with a graphite rod as the anode and a platinum plate as the cathode under 6 mA constant current at ambient temperature with MeCN as the solvent. Delightedly, the targeted product **3aa** was formed in 92% GC yield (Table 1, entry 1). When other solvents, such as DMSO or water, were used instead of MeCN, a low or trace yield of **3aa** was observed (entries 2 and 3). The influence of electrode effect was next investigated (entries 4−7), and the results revealed that the graphite rod anode and platinum plate cathode were the best choice for the electrode materials. When supporting electrolytes such as *n*-Bu_4NBr, *n*-Bu_4NBF_4, or $LiClO_4$ were added to the reaction system, the yield of **3aa** was severely reduced (entries 8−10). Decreasing the constant current to 4 mA resulted in the formation of **3aa** in a lower yield (entry 11). No improvement in the transformation was obtained when 8 mA constant current was employed (entry 12). Increasing the loading of **2a** as well as reaction temperature did not provide improved reaction efficiency (entries 13 and 14). Changing the air atmosphere to a nitrogen atmosphere had virtually no effect on the yield of **3aa**. No reaction occurred without a constant current, and the substrate **1a** was quantitatively recovered (entry 15).

With the optimal conditions in hand, the substrate scope of the electrochemical amidation reaction was explored with respect to both amines and α-ketoaldehydes (Scheme 2). A variety of aliphatic and aromatic amines were allowed, and the

Scheme 2. Reaction Scope[a,b]



[a]Reaction conditions: **1** (0.5 mmol), **2** (0.5 mmol), MeCN (8.0 mL), Pt plate (15 mm × 15 mm × 0.3 mm) cathode, graphite rod (6 mm) anode, *I* = 6.0 mA, in air, rt, 23 h. [b]Isolated yields.

isolated yields were mostly ≥80%. A series of bulky heterocyclic cyclic secondary amines, including *N*-substituted piperazines (**3aa**−**3ae**), morpholine (**3af**), and pyrrolidine (**3ag**), were well compatible in the present reaction and provided the target products in good to excellent yields. In the reported method, it was hard to synthesize α-ketoamides from primary aliphatic amines, likely due to the fact that the primary amines are less reactive than the secondary amines. To our delight, the aliphatic primary amines containing diverse chain lengths and isomeric alkyl, phenyl, and cyclic alkyl substituents underwent the amidation smoothly, delivering the desired products in 79−90% yields (**3ah**−**3ao**). In addition, both the aniline and substituted 2-aminopyridines are suitable substrates for this transformation (**3ap**−**3as**), which are incompatible with the conventional thermal method and the visible light induced protocol. Next, various aromatic α-ketoaldehydes were investigated under the optimal conditions to react with 1-methyl piperazine. 2-Oxo-2-phenylacetaldehydes, whether the phenyl ring was substituted with an electron-donating, an electron-withdrawing, or a sterically hindered group, provided the desired products in high yields (**3ba**−**3ja**). Among these, the resulting products bearing halogens such as F, Cl, Br, and I could be further utilized for the synthesis of more complex organic compounds. The structure of α-ketoamides **3ea** (CCDC: 1958206) was confirmed using single crystal X-ray diffraction as shown in Scheme 2.

The applicability of the present electrochemical reaction was further tested (Scheme 3). In the small-scale synthesis, a

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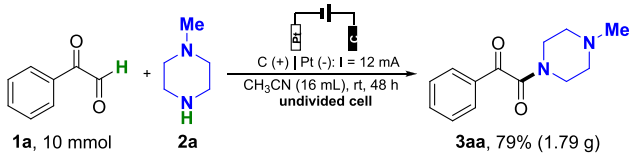
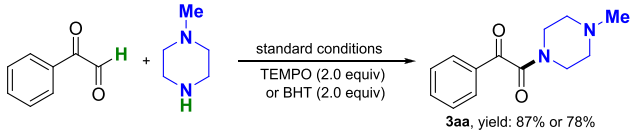
Scheme 3. Large-scale Synthesis of 3aa



concentration of 0.0625 M of the α-ketoaldehydes was required, and the increase of reaction substrate in concentration would reduce the yield of the target product. Pleasingly, the amidation reaction of **1a** with a 10-fold increase in the concentration of **1a** gave the target product **3aa** in 79% yield.

To understand the reaction mechanism, the radical capture experiments were conducted. The addition of 2 equiv of radical scavenger (TEMPO or BHT) had virtually no effect on the reaction efficiency (Scheme 4), suggesting that a free-radical

Scheme 4. Radical-Capturing Experiments



process might not be involved in the transformation. Moreover, the redox potential of the substrates and reaction mixture were investigated. As shown in Figure 1, the oxidation

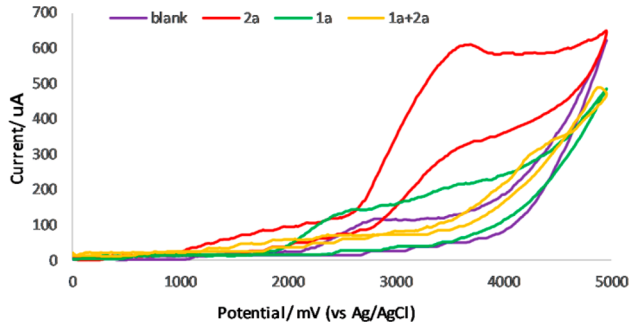


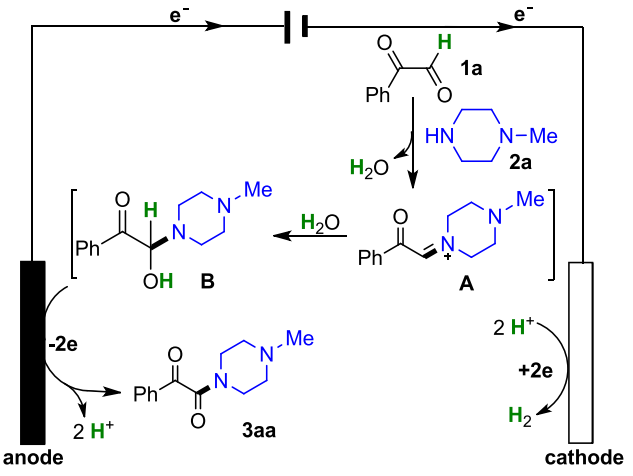
Figure 1. Cyclic voltammetry experiments.

peak of *N*-methylpiperazine (**2a**) (1.095 V, vs Ag/AgCl) was lower than that of 2-oxo-2-phenylacetaldehyde (**1a**) (2.533 V vs Ag/AgCl), which indicated that the oxidation of 1-methylpiperazine (**2a**) should occur first under the galvanostatic mode. In addition, the oxidation peak (1.09 V, vs Ag/AgCl) was also observed in the cyclic voltammetry experiment of mixture of **1a** and **2a**.

On the basis of the mechanistic studies above and the literature,[2e,5p,8] a plausible mechanism is outlined in Scheme 5. Initially, 2-oxo-2-phenylacetaldehyde (**1a**) reacted with 1-methylpiperazine (**2a**) to form an iminium intermediate **A**, which underwent water addition to generate a hemiaminal intermediate **B**. Next, the intermediate **B** was oxidized to target product **3aa** via Shono oxidation by the anode. On the cathode, the proton was reduced to the hydrogen gas.

In summary, we have developed a general and efficient protocol for the clean preparation of various α-ketoamides (28 examples, 71−91%) through the direct electrochemical amidation of α-ketoaldehydes and amines at ambient temperature. Moreover, the amidation reaction can be conducted on a

Scheme 5. Proposed Mechanism



gram scale with excellent efficiency. The present method has favorable characteristics over the previous synthetic methods: (1) the well-matched reactivity of α-ketoaldehydes and amines avoids the usage of supporting electrolyte and traditional heating; (2) the use of current as a green and traceless oxidant leads to simple and clean conditions (catalyst- and oxidant-free conditions) and harmless H_2 as the sole side product; (3) in comparison with the previous methodologies, both aliphatic and aromatic primary amines, as well as heteroaromatic amines, were suitable for cross-dehydrogenative coupling reaction.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications Web site. The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c00387.

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CCDC 1958206 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

■ AUTHOR INFORMATION

Corresponding Author

Wei-Min He − *Hunan Provincial Key Laboratory of Materials Protection for Electric Power and Transportation, Changsha University of Science and Technology, Changsha 410114, China*; orcid.org/0000-0002-9481-6697; Email: weiminhe2016@yeah.net

Authors

Jin-Yang Chen − *College of Chemistry and Chemical Engineering, Yangtze Normal University, Chongqing 408000, China*

Hong-Yu Wu − *College of Chemistry and Chemical Engineering, Yangtze Normal University, Chongqing 408000, China*

Qing-Wen Gui − *School of Chemistry and Chemical Engineering, Hunan University of Science and Technology,*

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Xiangtan 411201, China; Hunan Provincial Key Laboratory of Materials Protection for Electric Power and Transportation, Changsha University of Science and Technology, Changsha 410114, China; orcid.org/0000-0003-4847-7070

Xiao-Ran Han − *College of Chemistry and Chemical Engineering, Yangtze Normal University, Chongqing 408000, China*

Yan Wu − *College of Chemistry and Chemical Engineering, Yangtze Normal University, Chongqing 408000, China*

Kui Du − *College of Chemistry and Chemical Engineering, Yangtze Normal University, Chongqing 408000, China*

Zhong Cao − *Hunan Provincial Key Laboratory of Materials Protection for Electric Power and Transportation, Changsha University of Science and Technology, Changsha 410114, China*

Ying-Wu Lin − *School of Chemistry and Chemical Engineering, University of South China, Hengyang 421001, China;* orcid.org/0000-0002-2457-0871

Complete contact information is available at: https://pubs.acs.org/10.1021/acs.orglett.0c00387

Notes

The authors declare no competing financial interest.

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