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Electrochemical oxidative decarboxylative of α-oxocarboxylic acids towards the synthesis of quinazolines and quinazolinones†

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A mild and environmentally electrochemical method for the synthesis of quinazolines and quinazolinones has been developed through anodic oxidation decarboxylative of α-oxocarboxylic acids. The present reaction was efficiently conducted by using simple and cheap NH_4I as the N-source and electrolyte in an undivided cell. The desired products, quinazolines and quinazolinones, were isolated in high yield under chemical oxidant free conditions.

Nitrogen-containing heterocycles,[1] like quinazolines[2] and quinazolinones,[3] represent important classes of compounds that are widely found in natural products and biologically active molecules. Furthermore, some compounds such as prazosin, iressa, methaqualone, rutaecarpine, bouchardatine *etc.*, containing quinazolines and quinazolinones scaffolds, exhibit various pharmaceutical activities and have been used in daily life (Fig. 1).[4] Due to their importance, various methods for the synthesis of quinazolines[5] and quinazolinones[6] have been developed. Although several good results have been achieved, most of these methods require either metal catalysts or chemical oxidants. Recently, Wang's[7] group reported a prominent research study on the electrosynthesis of quinazolines and quinazolinones using tetramethylethane-1,2-diamine as the methyl source (Scheme 1, I). However, the long reaction times and finite substrate scope have limited the application of the reaction.

α-Oxocarboxylic acids, known for their low toxicity, high stability, and ease of storage, have been widely used as acyl reagents or C1 synthon through decarboxylation in the field of organic synthesis.[8] In 2019, Yan and Wang reported a copper-catalyzed oxidative decarboxylative amination of glyoxylic acid for synthesis of quinazolines and quinazolinones[9] (Scheme 1, II). Nevertheless, this method involves metal catalysts, chemical oxidants and long reaction time.

Since the discovery of Kolbe electrolysis,[10] electrochemical oxidative[11] decarboxylation has emerged as a practical tool for constructing C–C or C–heteroatom bonds, as it circumvents the need for chemical oxidants.[12] Recently, Huang, Zeng, Xu, Lei, Wei and others reported a series of electrochemical decarboxylation reactions of α-oxocarboxylic acids for synthesis of formamides,[13] ketone,[14] 3-formylindoles,[15] β-substituted desaturated cyclic amines,[16] α-ketoamides,[17] 2-substituted benzimidazoles,[18] quinazolinones,[19] 2,5-disubstituted 1,3,4-oxadiazoles,[20] enaminones[21] *etc.*[22] (Scheme 1, III). To further expand the application of electrochemical oxidative decarboxylation of α-oxocarboxylic acids, we hereby report an environmentally friendly electrochemical oxidative decarboxylation protocol for the synthesis of quinazolines and quinazolinones (Scheme 1, IV).

We commenced our studies by using (2-aminophenyl)(phenyl)methanone **1** and glyoxylic acid **2** as the model substrates to optimize the reaction conditions (Table 1). The desired product **3** was isolated in 94% yield by conducted the electrolysis in an undivided cell equipped with a carbon rod

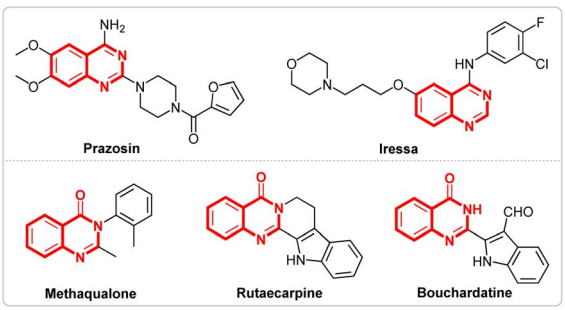

Fig. 1 Structures of selected bioactive quinazolines and quinazolinones.

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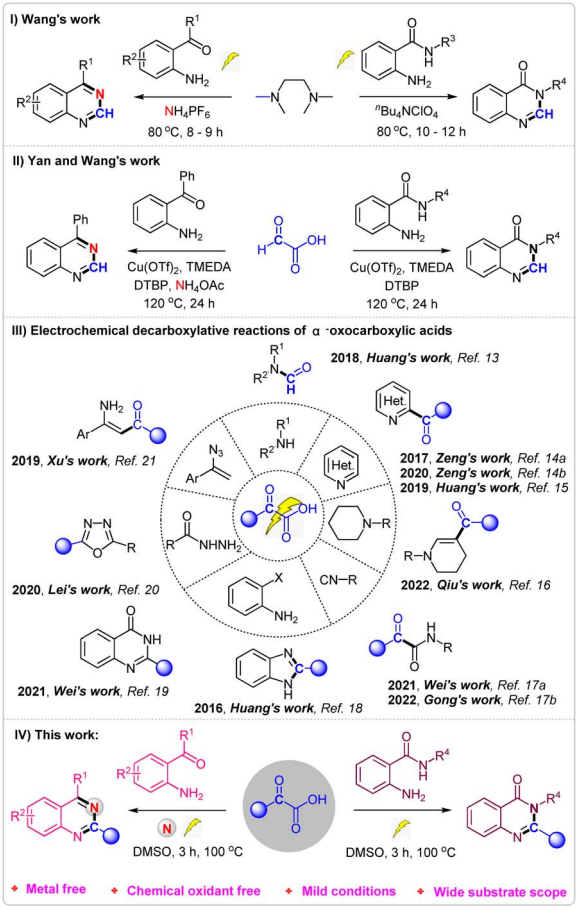
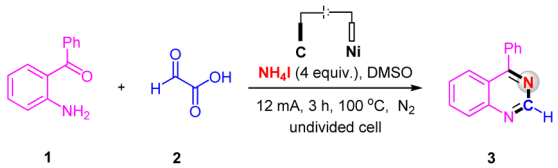


Scheme 1 Various methods for the synthesis of quinazolines and quinazolinones and electrochemical decarboxylative reactions of α-oxocarboxylic acids.

anode and a nickel plate cathode with a solvent of DMSO containing NH_4I as the N-source and electrolyte at 100 °C under a constant current of 12 mA for 3 h (Table 1, entry 1). Control experiment has confirmed the essential role of electricity, no desired product without current (Table 1, entry 2). Compared with NH_4I as the N-source and electrolyte, the yield dropped significantly when employing NH_4Cl and CH_3COONH_4 as the N-source and electrolyte (Table 1, entries 3 and 4). Furthermore, increasing the current to 15 mA did not improve the reaction yield, whereas when the current was decreased to 8 mA, a slight decrease in yield was observed (Table 1, entries 5 and 6). As for the reaction temperature, higher temperature has no effect on the reaction (Table 1, entry 7), but the yield decreased below 100 °C (Table 1, entry 8). Neither prolonging nor shortening the reaction time could improve yield of **3** (Table 1, entries 9 and 10). Different electrode couples such as C(+)/C(−), C(+)/Pt(−) and C(+)/Fe(−) were examined, from which the couple electrode of C(+)/Ni(−) was the best choice for this reaction (Table 1, entries 1 and 11–13). When switching DMSO to other solvents, such as CH_3CN and DMF, the yield of **3** was obtained in only 11% and 35%, respectively (Table 1, entries 14 and 15). Only 41% yield of product **3** was obtained, when the reaction was performed under air (Table 1, entry 16).

Table 1 Optimization studies and control experiments

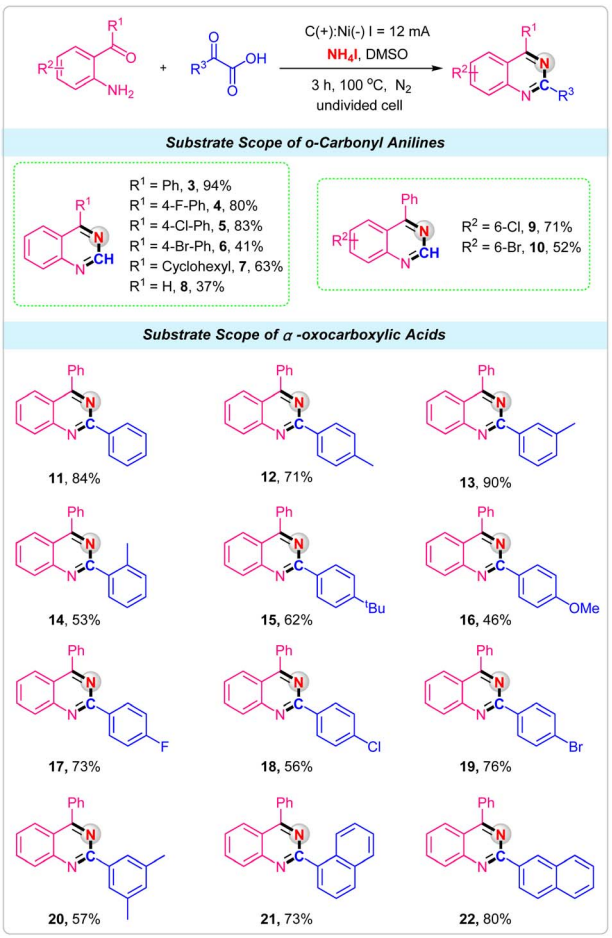


1 2 3

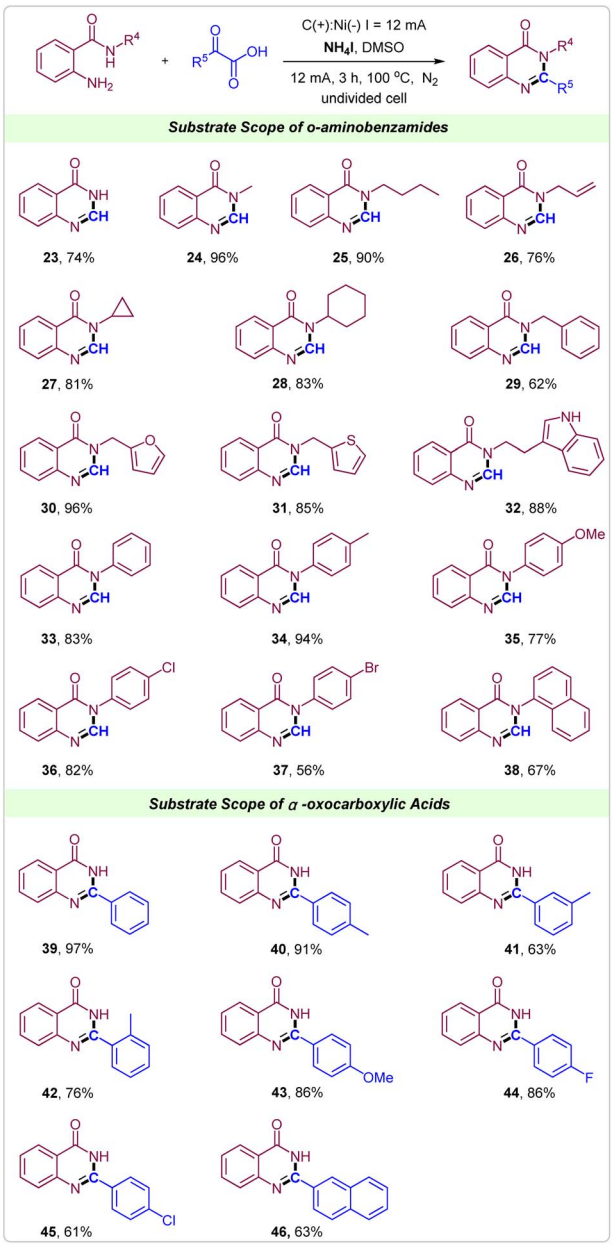
Entry	Variation from the standard conditions	Yield of[a] **3** (%)
1	None	94
2	No electric current	Trace
3	NH_4Cl instead of NH_4I	45
4	CH_3COONH_4 instead of NH_4I	72
5	15 mA instead of 12 mA	81
6	8 mA instead of 12 mA	77
7	120 °C instead of 100 °C	93
8	80 °C instead of 100 °C	61
9	3.5 h instead of 3 h	88
10	2.5 h instead of 3 h	76
11	Graphite rod instead of nickel plate	80
12	Platinum plate instead of nickel plate	86
13	Iron plate instead of nickel plate	67
14	CH_3CN instead of DMSO	11
15	DMF instead of DMSO	35
16	Air instead of N_2	41

[a] Reaction conditions: C anode, Ni cathode, constant current = 12 mA, **1** (0.2 mmol), **2** (0.4 mmol, 50% in water), NH_4I (0.8 mmol), in 10.0 mL DMSO at 100 °C under N_2 for 3 h. Yields shown are of isolated products.

With the optimized conditions in hand, we next explored the scope of various *o*-carbonyl anilines and α-oxocarboxylic acids to illustrate the robustness of the electrochemical reactions for the synthesis of quinazolines. First, we examined the effect of *o*-carbonyl anilines with different substituents. It was observed that substituent R1 was an aryl group with electron-withdrawing such as F, Cl and Br attached to the phenyl ring were well tolerated, giving the corresponding quinazolines products **4–6** in good yields. When R1 was cyclohexyl, a 73% yield was obtained in this electrochemical strategy (Scheme 2, **7**). Unfortunately, only 37% yield was obtained by employing 2-aminobenzaldehyde as the reactant (Scheme 2, **8**). For the substrates of Cl and Br substituents on the 5-position of 2-aminobenzophenone, reactions also proceeded smoothly with glyoxylic acid and NH_4I to deliver the desired products **9** and **10** in good yields. Then, the α-oxocarboxylic acids were investigated under the standard conditions. For phenylglyoxylic acid substrate, the product **11** was obtained in 84% isolated yield. *Ortho*-, *meta*-, and *para*-methyl substituted phenylglyoxylic acids were effectively converted into corresponding quinazolines product with good to excellent yields (Scheme 2, **12–14**). Other electron-donating substituents, such as *tert*-butyl and methoxy groups, were also tolerated (Scheme 2, **15–16**). The halogen group such as F, Cl and Br had little influence on the reaction, providing the products **17**, **18** and **19** in 56–76% yields. For multisubstituted substrate 2-(3,5-dimethylphenyl)-2-oxoacetic acid, the product **20** was obtained in 57% isolated yield. Finally, the 2-(naphthalen-1-yl)-2-oxoacetic acid and 2-



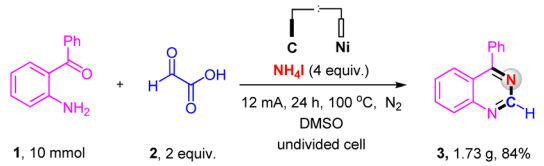
Scheme 2 Substrate scope of electrochemical decarboxylative reactions of α-oxocarboxylic acids for synthesis of quinazolines. Reaction conditions: C anode, Ni cathode, constant current = 12 mA, *o*-carbonyl anilines (0.2 mmol), α-oxocarboxylic acids (0.4 mmol), NH_4I (0.8 mmol), in 10.0 mL DMSO at 100 °C under N_2 for 3 h. Yields shown are of isolated products.



Scheme 3 Substrate scope of electrochemical decarboxylative reactions of α-oxocarboxylic acids for synthesis of quinazolinones. Reaction conditions: C anode, Ni cathode, constant current = 12 mA, *o*-carbonyl anilines (0.2 mmol), α-oxocarboxylic acids (0.4 mmol), NH_4I (0.8 mmol), in 10.0 mL DMSO at 100 °C under N_2 for 3 h. Yields shown are of isolated products.

(naphthalen-2-yl)-2-oxoacetic acid were successfully used as the substrates, and the products **21** and **22** were produced in 73% and 80% yields, respectively.

It is noteworthy to emphasize that the current tactics could also be used to synthesize quinazolinones under the same reaction conditions. Gratifyingly, the reaction of 2-aminobenzamide with glyoxylic acid proceeded smoothly, delivering the desired products **23** in a 74% yield. Then, 2-amino-*N*-alkylbenzamides were attempted. As shown, when the alkyl was methyl, *n*-butyl, allyl group, cyclopropyl, cyclohexyl or benzyl showed good to excellent reactivity in the synthesis of quinazolinones (Scheme 3, **24–29**). Notably, the heterocyclic substituents such as 2-methylfuran, 2-methylthiophene and tryptamine were well tolerated in this electrochemical reaction giving the corresponding quinazolinones in excellent yields (Scheme 3, **30–32**). Similarly, for 2-amino-*N*-phenylbenzamides, reactions also proceeded well to deliver the corresponding quinazolinones products in moderate to excellent yields, in which electron-donating and electron-withdrawing groups on the benzene ring of 2-amino-*N*-phenylbenzamides could be well tolerated (Scheme 3, **33–37**). 2-Amino-*N*-(naphthalen-1-yl)benzamide was also suitable substrate to give product **38** in 67% yield. Next, various α-oxocarboxylic acids were tested to reacted with 2-aminobenzamide for synthesis of 2-aryl quinazolinones. From the reaction between 2-aminobenzamide and 2-oxo-2-phenylacetic acid, the product 2-phenylquinazolin-4(3*H*)-one **39** was achieved in 97% yield. The substituent position did not strongly impact the efficiency of this protocol (Scheme 3, **40–42**). When methoxy, F and



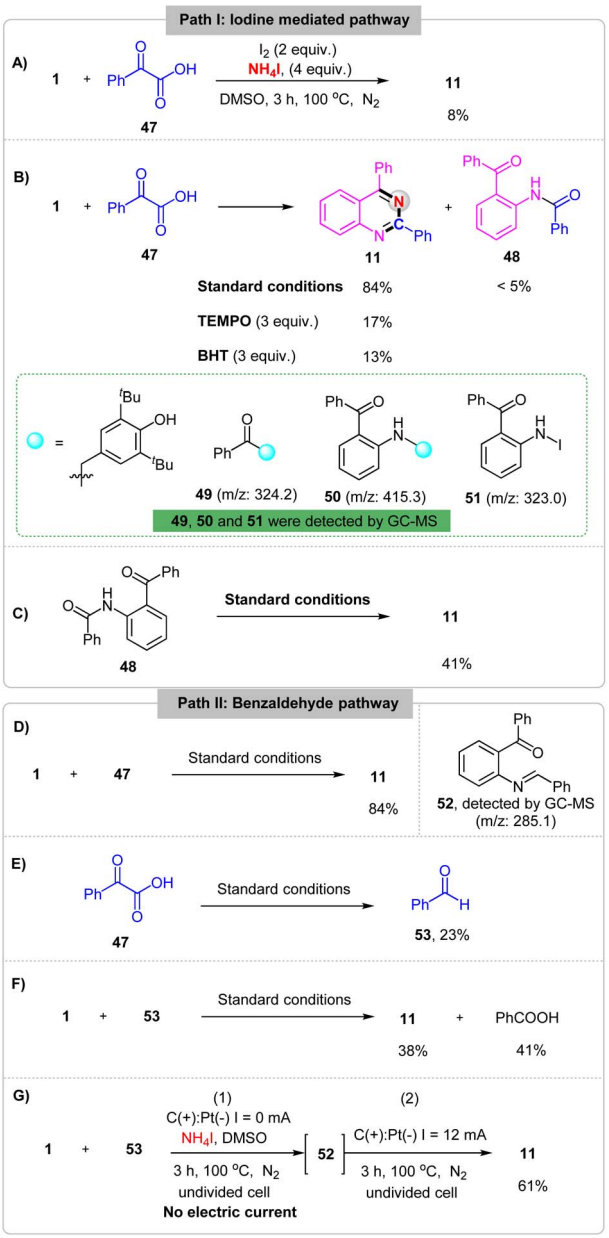
Scheme 4 Gram scale synthesis.

Cl groups were introduced into the *para*-position of 2-oxo-2-phenylacetic acid the products **43**, **44** and **45** were obtained in good to excellent yields. Notably, 2-(naphthalen-2-yl)-2-oxoacetic acid could also react well with 2-aminobenzamide to form the target product **46** in 63% yield.

The scalability of the electrochemical oxidative decarboxylative of α-oxocarboxylic acids for synthesizing nitrogen-containing heterocycles was evaluated on a 10.0 mmol scale (Scheme 4). The reaction afforded the corresponding product in an 84% yield, substantiating the great applied potential of this electrochemical reaction.

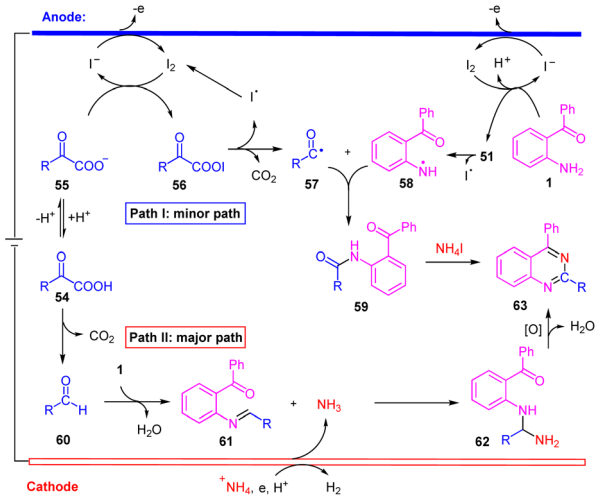
To gain more insight into the electrochemical oxidative reaction, a set of mechanistic studies was performed. First, the cyclic voltammetry (CV) experiments on NH_4I, **1** and **47** have been carried out and showed that the oxidation peaks of NH_4I, **1** and **47** in acetonitrile were 0.60 V, 1.62 V and 1.58 V (Fig. S5†), respectively. Then, the reaction between **1** and **47** was performed with chemical oxidants I_2 in the absence of an electrical input and the desired product **11** was generated in 8% yield (Scheme 5A). These results implicated that this reaction probably proceeds by an iodine mediated process. Second, a 3 equiv. radical scavenger TEMPO (2,2,6,6-tetramethylpiperidin-1-oxyl) or BHT (2,6-di-*tert*-butyl-4-methylphenol) was added in this reaction under standard conditions, the yield of desired product **11** was obtained in yields of 17% and 13%, respectively. And the compounds **49**, **50** and **51** were detected by GC-MS (Scheme 5B). These results implicated that the reaction may proceed by a radical pathway with acyl radical and nitrogen radical generating. Additionally, a constant amount of compound **48** was observed under the standard conditions. When, the **48** was test in the absence of **1** and **47**, the product **11** was obtained in 41% yield (Scheme 5C), which showed that compound **48** may the inter-mediate in this reaction. Furthermore, compound **52** was detected by GC-MS (Scheme 5D) and benzaldehyde **53** was formed from 2-oxo-2-phenylacetic acid **47** (Scheme 5E) under standard conditions. Last but not least, replacing **1** with **53** under the standard conditions, **11** was obtained in 38% yield (Scheme 5F). However, when the reaction was reacted under the standard conditions without electric current for 3 h and then reacted under the standard conditions the yield of **11** was improved to 61% (Scheme 5G). These results indicating that the aldehyde intermediate may be generated in this electrochemical reaction.

Taken the above results and the previous literature precedent, we propose two plausible mechanistic pathways for this electrochemical transformation. In path I, iodide ion generates I_2 by anodic oxidation.[14a,21] Then I_2 react with α-keto carboxylate



Scheme 5 Mechanistic investigations. (A) I_2 oxidation experiment. (B) Radical-trapping experiment. (C) Intermediate **48** validation experiment. (D) Intermediate **52** detection experiment. (E) Benzaldehyde **53** generation experiment. (F) Intermediate **53** validation experiment. (G) Intermediate **52** validation experiment.

anion **55** to form acyl hypoiodite **56** along with iodide ion.[14a,21] At the same time, the substrate **1** reacted with I_2 to generated **51**. Followed by the **56** and **51** undergoes hemolytic dissociation to give iodine radical, aroyloxy radical **57** [14a,21] and nitrogen radical **58**. Subsequently, intermediate **59** is generated through radical/radical coupling between **57** and **58**. Finally, intermediate **59** react with NH_4I to generate the target product **63**.[5b] In path II, α-oxocarboxylic acid products aldehyde **60** with decarboxylation.[18] Then, imine **61** is formed from **60** and **1** by dehydration condensation.[7] On the other hand, $^+NH_4$ was reduced on the cathode to generate NH_3.[7,23] Subsequently, nucleophilic



Scheme 6 Proposed mechanism.

addition of NH_3 to imine **61** provides an intermediate **62**, which *via* a tandem condensation-oxidation process to generate the target product **63**.[5a,7] Noteworthily, acyl radical (**57**) and primary amine-based radical (**58**) were both transient radicals, making it difficult to generate **59** from the cross coupling of **57** and **58**. Therefore, we propose that path II is the more favorable process for our reaction (Scheme 6).

Conclusions

In conclusion, we have developed a mild and environmentally friendly electrochemical method for the synthesis of quinazolines and quinazolinones *via* electrochemical oxidative decarboxylation of α-oxocarboxylic acids. This protocol utilizes NH_4I as the N-source and electrolyte, which is simple and inexpensive, eliminating the need for transition metals and chemical oxidants. A wide range of substituted quinazolines and quinazolinones were synthesized with good to excellent yields.

Author contributions

J. Wu, M. Zhang, J. He and K. Li, conducted the experiments. J. Wu, L. Ye and X. Xu conceived the project, wrote the manuscript and prepared the ESI. J. Zhou prepared part of the starting materials. Z. Li and H. Xu supervised the work and revised the manuscript.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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