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Electrochemical diazenylation of active methylene compounds with aryldiazonium salts: efficient synthesis of β-dicarbonyl benzoylhydrazones

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In this study, an electrochemical synthetic strategy is reported for the diazenylation of various active methylene compounds through their reaction with aryldiazonium salts, leading to the efficient formation of β-dicarbonyl benzoylhydrazones. The reaction efficiency and selectivity are significantly enhanced by the promotion of LiBr, along with the rational selection of electrode materials to effectively suppress the formation of a solid electrolyte interphase (SEI) layer on the electrode surface. The developed methodology exhibits remarkable substrate versatility, accommodating a broad range of substrates including aliphatic, cyclic, and aromatic diketones, as well as malononitrile and malonate derivatives. This work provides a green, cost-effective, and highly efficient approach for the synthesis of valuable benzoylhydrazone derivatives.

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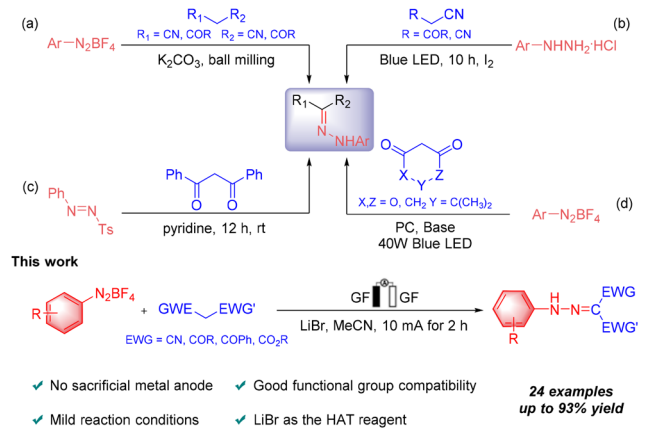
Introduction

The formation of carbon–nitrogen (C–N) bonds is of paramount importance in organic chemistry, facilitating the construction of essential nitrogen-containing organic compounds. These compounds are invaluable in organic synthesis, natural products, and medicinal chemistry.[1] Among the vast array of nitrogen-containing molecules, acylhydrazones stand out due to their structural diversity, a feature that grants them high adaptability in both chemical and pharmaceutical applications.[2] Specifically, benzoyl hydrazones play a pivotal role in the synthesis of various heterocyclic compounds, including indoles, pyrazolines, pyrazoles, and triazoles.[3] Numerous hydrazone derivatives are also utilized for therapeutic purposes, such as in the treatment of Alzheimer's disease,[4] hemostasis,[5] cancer therapy,[6] and as antifungal, antiviral, and antibacterial agents.[7] Consequently, the quest for a straightforward and efficient method to synthesize aryl hydrazone derivatives has attracted increasing attention from researchers in the field.

Among the methodologies for synthesizing phenylhydrazone compounds, diazenylation reactions have emerged as a cornerstone approach.[8] These reactions involve the incorporation of diazo alkyl or aryl groups into active methylene compounds or other nucleophilic reagents, thereby facilitating highly efficient synthesis of phenylhydrazone compounds and their analogs.[9] For instance, in 2014, Zhu's group described the synthesis of phenylhydrazone derivatives through the reaction of acetylacetone derivatives with aryl diazonium salts under ball-milling conditions. This approach, however, is dependent on specialized and costly equipment, necessitates base catalysis, and is restricted to aliphatic compounds, thereby limiting its suitability for scale-up applications (Scheme 1a).[10] Subsequently, Batra's group described a pro-



Scheme 1 Diazenylation reaction for the preparation of phenylhydrazone compounds.

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cedure for the photocatalytic diazenylation of active methylene compounds and N-heterocyclic compounds using arylhydrazine hydrochlorides in the presence of iodine and a base, aimed at synthesizing phenylhydrazone derivatives. Nonetheless, this method relies on the use of stoichiometric amounts of base and is restricted to nitrile substrates (Scheme 1b).[11] In 2020, Wang Lei's group reported a pyridine-catalyzed condensation of β-diketones with arylazosulfones to prepare β-diketone phenylhydrazone products. This method relies on the presence of a tosyl group and is only applicable to β-diketone substrates (Scheme 1c).[12] In 2023, Das and colleagues introduced a method for the diazenylation of 1,3-diones and cyclic esters using organocatalysts and aryldiazonium tetrafluoroborates under photoredox conditions. However, this method depends on high-power light sources and requires the addition of stoichiometric amounts of base (Scheme 1d).[13]

Although these methods have successfully enabled the synthesis of phenylhydrazone compounds, their practical application is often hindered by several limitations, including a narrow substrate scope, the need for additional catalysts, or the requirement of strong bases. Consequently, the development of an environmentally benign and broadly applicable method for synthesizing hydrazone compounds remains a highly desirable goal in synthetic chemistry. In recent years, organic electrochemistry has emerged as a more efficient and sustainable alternative in the realm of organic synthesis.[14] In comparison to conventional organic synthesis, organic electrochemical methods present distinct benefits, such as the avoidance of exogenous redox reagents and the utilization of more benign reaction conditions.

Building on our ongoing research in electrochemical synthesis[15] and motivated by the goal of expanding the scope of diazenylation reactions, we herein report a novel electrochemically-driven strategy for diazenylation. This approach enables the efficient synthesis of β-dicarbonyl benzoylhydrazones through the electrochemical reaction of active methylene compounds with aryl diazonium salts. The developed method exhibits remarkable substrate versatility, accommodating a wide range of substrates including aliphatic, cyclic, and aromatic diketones, as well as malononitrile and malonates. To the best of our knowledge, this protocol currently represents the most extensive substrate scope reported for such transformations. This advancement provides a green, cost-effective, and efficient route for the synthesis of benzoylhydrazone derivatives.

Results and discussion

The reaction between acetylacetone (**1c**) and 4-methoxyphenyl diazonium tetrafluoroborate (**2a**) was selected as the prototype to investigate the optimal reaction conditions, and the results are summarized in Table 1.

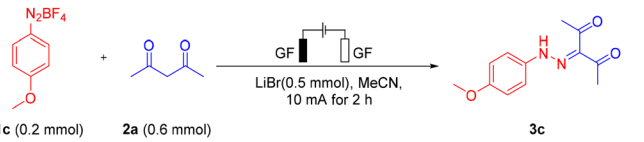
Initially, the reaction was conducted in MeCN using a graphite felt (10 × 10 × 1.0 mm^3) as both the anode and cathode, under a constant current of 10 mA, with LiBr (0.5 mmol) used as the supporting electrolyte. The desired product (**3c**) was obtained in 93% yield (Table 1, entry 1). Subsequently, when the supporting electrolyte was replaced with *n*-Bu_4NBF_4 or *n*-Bu_4NI instead of LiBr, the reaction was completely inhibited (Table 1, entries 2 and 3). Utilizing other bromide salts, such as *n*-Bu_4NBr and NaBr, as supporting electrolytes led to a decrease in yield by approximately 10% (Table 1, entries 4 and 5), indicating that bromide ions may play a critical role in this reaction.

Given that previous studies have shown the reaction to be highly base-dependent,[10–13] we carried out the reaction in the presence of stoichiometric amounts of base, using *n*-Bu_4NBF_4 in place of LiBr as the supporting electrolyte. However, no formation of the desired product was observed (Table 1, entry 6).

Additionally, several basic additives were introduced under standard reaction conditions; however, the reaction was significantly inhibited (Table 1, entries 7–9). Collectively, these results suggest that the reaction exhibits minimal dependence on the presence of a base. Moreover, the addition of various acidic additives also inhibited the reaction, resulting in a significant decrease in yield (Table 1, entries 10 and 11).

Adjusting the amount of supporting electrolyte LiBr, either increasing or decreasing it, also resulted in reduced yields (Table 1, entries 12 and 13). Since lithium salt was chosen as the supporting electrolyte in the reaction, the selection of elec-

Table 1 Optimization of the reaction conditions[a]



Entry	Variation of conditions	Yield[b] (%)
1	None	93
2	*n*-Bu_4NBF_4 instead of LiBr	0
3	*n*-Bu_4NI instead of LiBr	0
4	*n*-Bu_4NBr instead of LiBr	82
5	NaBr instead of LiBr	80
6[c]	NaOH (1 eq.)	0
7	DABCO (1 eq.)	9
8	Na_2CO_3 (1 eq.)	25
9	KOH (1 eq.)	31
10	AcOH (20 mmol%)	40
11	TFA (20 mmol%)	70
12	LiBr (1 eq.)	34
13	LiBr (4 eq.)	70
14	Ni (−) instead of GF (−)	0
15	Pt (−) instead of GF (−)	76
16	C (−) instead of GF (−)	88
17	**1c : 2a** = 1 : 1	18
18	**1c : 2a** = 1 : 4	68
19	5 mA for 4 h	60
20	15 mA for 1 h	81
21	Without electricity	5

[a] Reaction conditions: **1c** (0.2 mmol), **2a** (0.6 mmol), LiBr (0.5 mmol), MeCN (6.0 mL), in air, graphite felt (10 × 10 × 1.0 mm^3) as the anode and cathode, constant current of 10 mA, 2 h. [b] Isolated yields. [c] *n*-Bu_4NBF_4 (0.5 mmol) as the supporting electrolyte.

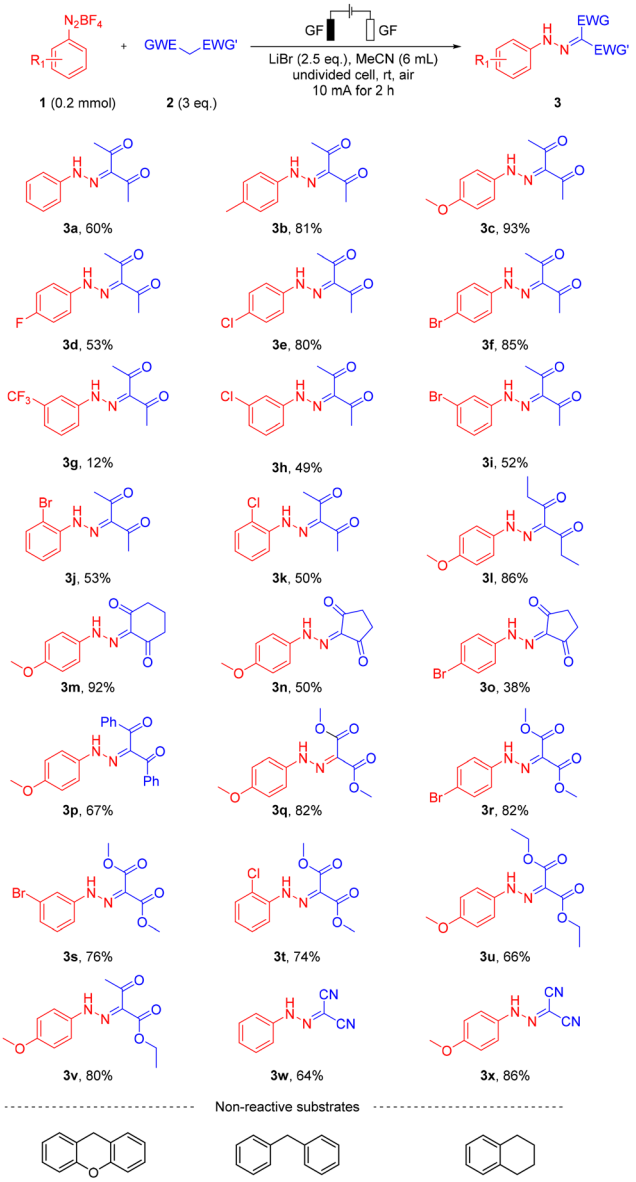
trode material significantly impacted the reaction efficiency. We tested various metal cathodes, including nickel foam and platinum sheets, and observed the formation of a distinct solid electrolyte interphase (SEI) film on their surfaces (Fig. S1).[16] The formation of the SEI film caused a sharp voltage increase, which in turn reduced the yield (Table 1, entries 14 and 15). Using a non-metal carbon rod electrode as the cathode also reduced the yield slightly (Table 1, entry 16).

The effect of varying the substrate ratio on the reaction was also investigated. It was found that changes in the substrate ratio did not enhance the yield (Table 1, entries 17 and 18). Increasing or decreasing the reaction current led to a slight decrease in the yield of **3c** (Table 1, entries 19 and 20). Without applying an electric current, the reaction yield was only 5% (Table 1, entry 21).

After optimizing the reaction conditions, we tested the substrate scope of the reaction (Scheme 2). Phenyl diazonium tetrafluoroborate exhibited good compatibility during the reaction, generating the target product (**3a**) smoothly with a 60% yield. It was observed that aryl diazonium salts bearing electron-donating groups (Me, OMe) at the *para*-position of the aromatic ring exhibited significantly higher reactivity compared to those with electron-withdrawing groups, yielding the corresponding products in 81% and 93% yields, respectively (**3b–3c**). Aryl diazonium salts with electron-withdrawing groups (F, Cl, Br) at the *para*-position of the aromatic ring also underwent the transformation smoothly, affording the desired products in moderate to good yields (**3d–3f**). However, when a strong electron-withdrawing group (CF_3) was introduced at the *meta*-position, the yield of the desired product (**3g**) was relatively low due to the lower conversion of diketone, and no other byproducts were observed. Aryl diazonium salts with electron-withdrawing substituents (Cl, Br) at the *meta*-position provided the corresponding products in yields of 49% and 52%, respectively (**3h–3i**). Notably, the yields of products obtained from *ortho*-substituted aryl diazonium salts were comparable to those from *meta*-substituted salts (**3j–3k**), suggesting that the reaction is slightly influenced by steric hindrance.

To further demonstrate the generality of this reaction, the scope of active methylene compounds was also investigated (**3l–3x**). In addition to acetylacetone, long-chain aliphatic 1,3-diketones, such as 3,5-heptanedione, generated the expected product **3l** in 86% yield, indicating minimal impact of steric hindrance on the reaction. Cyclic 1,3-diketones, including cyclohexane-1,3-dione and its analogues, were successfully utilized, affording the desired products in moderate to excellent yields (**3m–3o**). Notably, aromatic 1,3-diketones with significant steric hindrance, such as dibenzoylmethane, underwent the transformation smoothly, affording the desired product in 67% yield (**3p**).

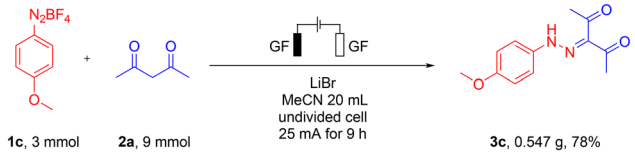
Furthermore, various malonates, including diethyl malonate and dimethyl malonate, exhibited good tolerance when reacted with diverse aryl diazonium salts, yielding the expected products in 66–82% yields (**3q–3u**). Dimethyl malonate reacted efficiently with aryl diazonium salts bearing electron-withdrawing groups at the *ortho*, *meta*, or *para* positions, and up to 82% yield was achieved (**3r–3t**). The unsymmetrical keto ester, ethyl acetoacetate, also underwent the reaction smoothly, yielding the desired product **3v** in 80% yield. Additionally, malononitrile proved to be effective in the reaction with aryldiazonium salts, achieving yields of up to 86% (**3w–3x**). Based on the aforementioned studies, we attempted to extend the reaction to other methylene compounds with similar structures but weaker acidity, such as xanthene, diphenylmethane, and acetophenone. Unfortunately, these substrates did not undergo the reaction (Scheme 2). This observation suggests that the presence of two electron-withdrawing groups is crucial for the success of the reaction.



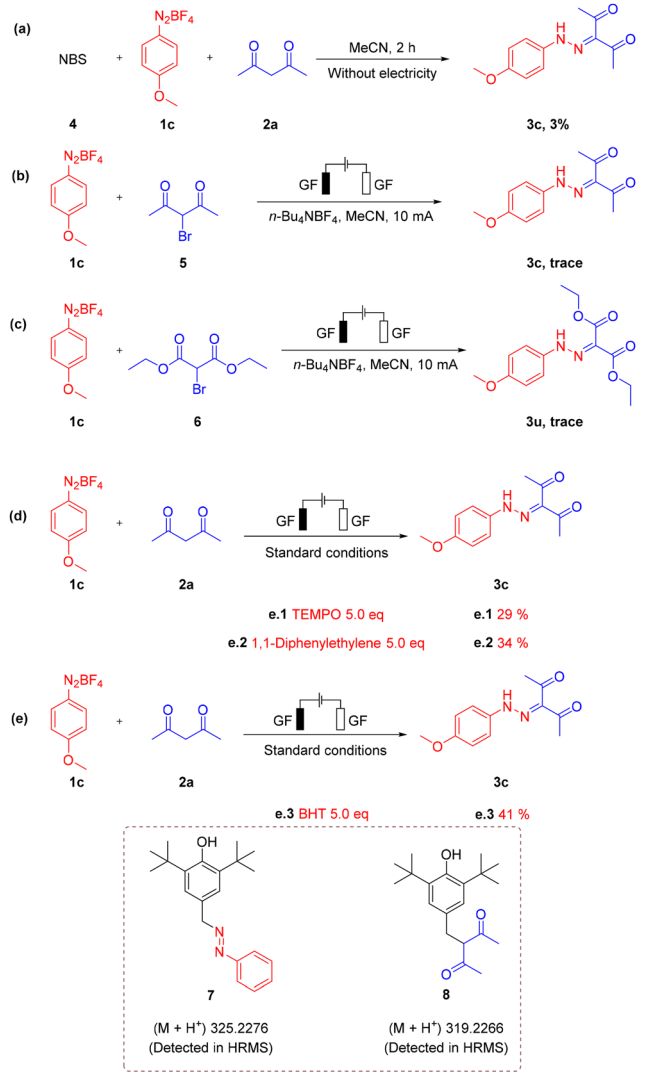
Scheme 2 Substrate scope. Reaction conditions: **1** (0.2 mmol), **2** (0.6 mmol), LiBr (0.5 mmol), MeCN (6.0 mL), in air, graphite felt (10 × 10 × 1.0 mm^3) as the anode and cathode, constant current of 10 mA, 2 h.

To validate the synthetic practicability of this method, we performed a gram-scale synthesis of compound **3c** using the model substrates **1c** and **2a**, achieving the desired product in 78% isolated yield (Scheme 3).

To gain insight into the reaction mechanism, a series of control experiments were carried out (Scheme 4). Initially, in the absence of an electric current and upon addition of a stoichiometric amount of NBS (**4**), the reaction between **1c** and **2a** yielded only 3% of the desired product, **3c** (Scheme 4a). Subsequently, the possible brominated intermediates, 3-bromopentanedione (**5**) and diethyl bromomalonate (**6**), were independently subjected to the electrochemical reaction with **1c**. However, no target product was observed under these conditions, suggesting that the transformation does not proceed through a bromination pathway (Scheme 4b and c).



Scheme 3 Gram scale of the reaction.



Scheme 4 Mechanism experiments.

Under the standard reaction conditions, several radical scavengers, such as 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO), 1,1-diphenylethylene, and butylated hydroxytoluene (BHT), were introduced into the reaction between **1c** and **2a**. The yield of **3c** was reduced from 93% to 29–41%, respectively (Scheme 4d and e), suggesting that a radical mechanism may be involved.[17] Moreover, from the reaction mixture with BHT as an additive, radical trapping adducts **7** and **8** were detected by high-resolution mass spectrometry (HRMS) (Scheme 4e).[18] These observations further indicated that the electrochemical coupling reaction may proceed *via* the coupling of the diazenyl radical ($Ar–N_2^{\bullet}$)[18] and the β-diketo radical.

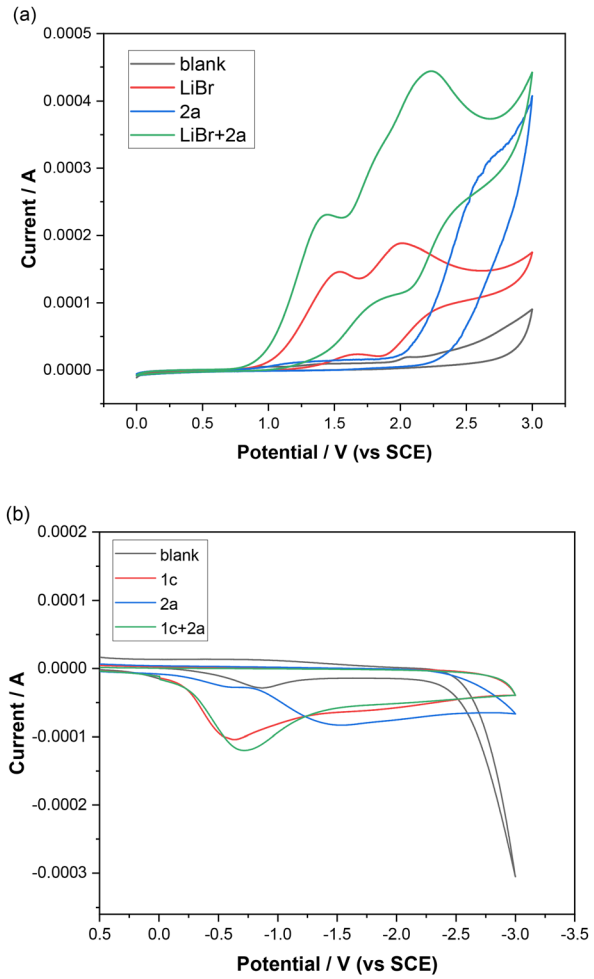
To gain deeper insight into the electrochemical mechanism of this reaction, cyclic voltammetry (CV) experiments were carried out on the reactants 2,4-pentanedione (**2a**) and LiBr, as well as their mixture. It was observed that **2a** exhibited no significant oxidation peaks within the potential range of 0–3 V. In contrast, LiBr displayed oxidation peaks at 1.51 V and 1.92 V. Notably, the mixture of LiBr and **2a** showed oxidation peaks at 1.42 V and 2.23 V, suggesting that LiBr is preferentially oxidized at the anode surface to generate bromine radicals (Scheme 5a).

Furthermore, as shown in Scheme 5b, *p*-methoxyphenyl diazonium tetrafluoroborate (**1c**) exhibited a single reduction peak at −0.64 V, whereas 2,4-pentanedione (**2a**) showed no distinct reduction peaks. The mixture of **1c** and **2a** also displayed only one prominent reduction peak at −0.72 V, with only a slight shift in peak position compared to that of **1c** alone. These CV results indicate that *p*-methoxyphenyl diazonium tetrafluoroborate (**1c**) possesses a relatively low reduction potential and is likely to be preferentially reduced during the cathodic process (Scheme 5b).

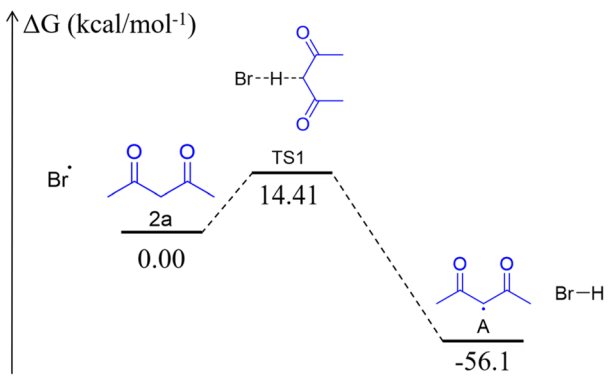
Based on the results obtained from Scheme 4e and 5a, we hypothesized that the β-diketo radical captured in compound **8** could originate from the reaction between bromine radicals and **2a**. In other words, LiBr might function as a hydrogen atom transfer (HAT) reagent.

To further elucidate the mechanism, we performed density functional theory (DFT) calculations, with the results presented in Scheme 6. The energy barrier for this hydrogen atom transfer (HAT) process is only 14.41 kcal mol^{-1}, indicating that the reaction can proceed very easily at room temperature[19] (also, the BDE of comparable HBr Br–H = 87 kcal mol^{-1} and malononitrile C(sp^3)–H bond BDE = 87 ± 2 kcal mol^{-1}).[20] These results suggest that bromine radicals, generated from the anodic oxidation of lithium bromide, can readily abstract a hydrogen atom from the labile methylene group of acetylacetone at room temperature.

Based on the experimental results and relevant literature reports, a plausible reaction mechanism is proposed (Scheme 7).[10–13,17,21] Initially, at the anode surface, bromide

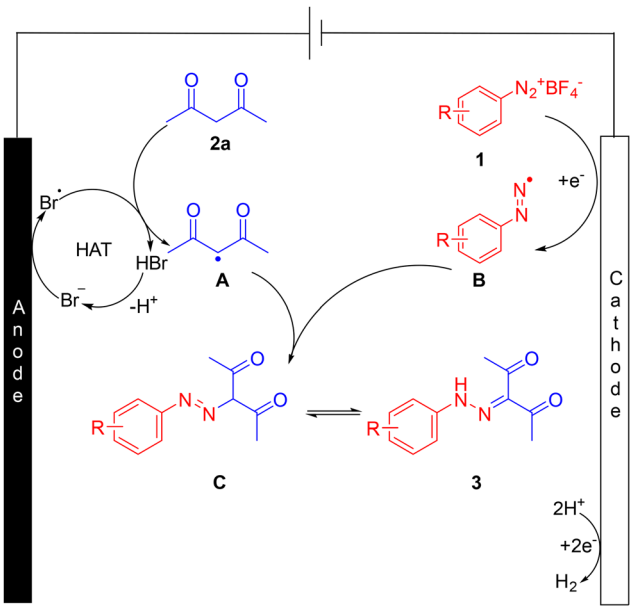


Scheme 5 Cyclic voltammetry studies. (a) Cyclic voltammograms of *n*-Bu_4NBF_4 (0.1 M) and related compounds in acetonitrile (20.0 mL) using glassy carbon working electrode, platinum plate and saturated calomel electrode as counter and reference electrode at 100 mV s^{-1} scan rate. (b) Cyclic voltammograms of LiBr (0.1 M) and the remaining conditions are the same as (a).



Scheme 6 DFT calculation.

ions (Br^-) undergo a single-electron transfer (SET) process to generate bromine radicals. These bromine radicals then abstract a hydrogen atom from **2a** *via* a hydrogen atom transfer



Scheme 7 Proposed reaction mechanism.

(HAT) process, forming the carbon radical intermediate **A** and hydrobromic acid (HBr). Thereafter, the HBr undergoes deprotonation to regenerate bromide ions, thereby completing a cycle.

Simultaneously, at the cathode surface, the aryl diazonium tetrafluoroborate **1** is reduced to a diazenyl radical ($Ar–N_2^{\bullet}$) intermediate **B**.[18] Subsequently, the carbon radical intermediate **A** couples with the diazenyl radical intermediate **B** to form intermediate **C**, which undergoes intramolecular rearrangement to yield the target product **3**. Concurrently, a hydrogen evolution reaction occurs at the cathode.

Conclusions

In conclusion, we have developed a mild and efficient electrochemical dehydrogenative diazenylation protocol for the synthesis of a variety of β-dicarbonyl benzoylhydrazone derivatives. This method proceeds smoothly without the need for any metal catalysts, ligands, or acidic or basic additives, and features a broad substrate scope and good functional group tolerance. Lithium bromide plays a multifaceted role in the reaction system, acting both as a supporting electrolyte and a hydrogen transfer reagent. By carefully selecting suitable electrode materials, the formation of a SEI layer on the electrode surface can be effectively suppressed, thereby significantly enhancing both the efficiency and selectivity of the reaction. Further mechanistic investigations are currently ongoing in our laboratory.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the SI.

Characterization and NMR spectrum of products, and experimental details are provided. See DOI: **https://doi.org/10.1039/d5ob01002k**

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