

Electrooxidative Hofmann Rearrangement of Phthalimides to Access Anthranilate Derivatives

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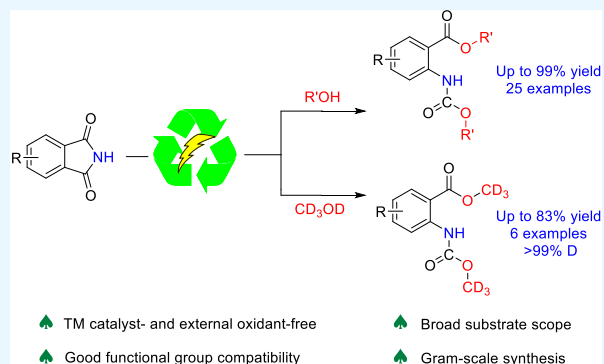
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ABSTRACT: A simple and efficient electrooxidative Hofmann rearrangement reaction of phthalimides was developed. Anthranilate derivatives were synthesized in moderate to good yields under green and mild conditions using phthalimides as a rearrangement precursor. This approach not only provides a strategy for synthesizing anthranilates and deuterated anthranilate derivatives with high deuteration efficiency but also realizes efficient conversion at the gram scale. A possible reaction mechanism is proposed.



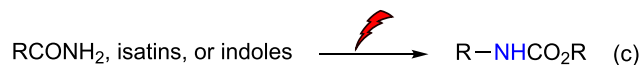
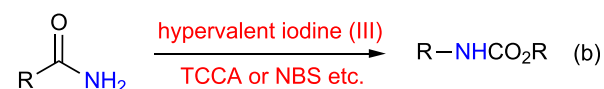
INTRODUCTION

Hofmann rearrangement plays an important role in synthetic chemistry and is a very effective method for the preparation of secondary amines, carbamates, anthranilic acids, ureas, and other related nitrogen-containing compounds based on the particular starting materials and reaction conditions.¹ This reaction generally occurs through the formation of an isocyanate intermediate. The traditional Hofmann rearrangement reaction process requires harsh conditions (e.g., Br₂ and NaOH), which limits the substrate scope and leads to some unwanted byproducts (Scheme 1a).² Therefore, many different reagents such as hypervalent iodine reagents generated in situ or added,³ trichloroisocyanuric acid (TCCA),⁴ *N*-bromosuccinimide (NBS),⁵ KBr,⁶ difluorobromane,⁷ and others⁸ have been used to promote the Hofmann rearrangement reactions (Scheme 1b). However, these methods regularly involve large amounts of external oxidants and other additives, which are undesirable from an environmental standpoint.

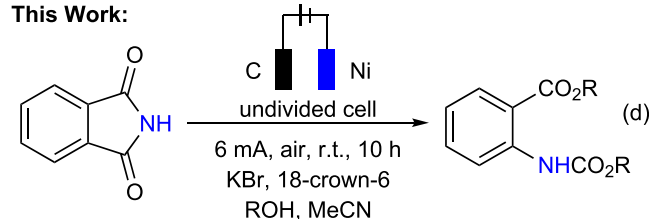
In the past few years, the revival of electrochemical organic synthesis has provided a new opportunity and alternative for greener and more sustainable synthetic procedures.⁹ Recently, some remarkable progress in electrochemically enabled Hofmann rearrangement reactions has made a deep impression on the community of synthetic chemistry, making Hofmann rearrangement reactions an environmentally friendly strategy without using traditional chemical oxidants (Scheme 1c).^{10–12} In 2018, Xu and Zhang reported the preparation of carbamates by NaBr-mediated electrochemical Hofmann rearrangement of benzamides and aliphatic amides.¹⁰ In 2021, an adjustable methodology for the synthesis of anthranilic acids via the electrochemical Hofmann ring-opening of isatins was demon-

Scheme 1. Versatile Methods (a–d) for the Hofmann Rearrangement

Previous work:



This Work:

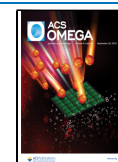


strated by the Wang group.¹¹ Notably, Guo and co-workers developed the construction of anthranilic acid derivatives

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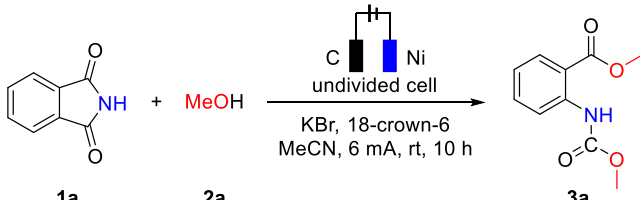
through the electrochemical oxidative ring-opening reactions of indoles with alcohols.¹²

Phthalimide and its analogues are widely used in organic synthesis as versatile building blocks,¹³ as well as in the field of pharmaceutical chemistry due to their rich biological activities.¹⁴ Recently, we realized the electrosynthesis of *N*-acylsulfenamides by the direct thiolation of amides (phthalimides, isatins, and aliphatic amides) with mercaptans through the formation of N–S bonds.¹⁵ Inspired by the acquired advances in electrochemical Hofmann reactions and our continuous interest in heterocyclic transformation¹⁶ and organic electrochemistry,¹⁷ we herein report a Hofmann rearrangement scheme for the direct conversion of phthalimides to the corresponding anthranilate derivatives via alcoholysis under electrooxidative conditions in the absence of transition-metal catalysts and excessive extra oxidants (Scheme 1d).

RESULTS AND DISCUSSION

Phthalimide (**1a**) and methanol (**2a**) were initially employed as model substrates to screen the optimal conditions for the electrochemically enabled Hofmann rearrangement reaction under constant current mode (Table 1, see Figures S1–S5 in the

Table 1. Optimization of Reaction Conditions^a



entry	variation from the standard conditions	yield ^b (%)
1	none	99
2	NaCl instead of KBr	trace
3	TBAI instead of KBr	NR
4	TBAB instead of KBr	49
5	ⁿ Bu ₄ NHSO ₄ instead of KBr	NR
6	4 mA instead of 6 mA	76
7	8 mA instead of 6 mA	82
8	MeOH (4 mL)/MeCN (0 mL)	68
9	MeOH (1 mL)/DCM (3 mL)	53
10	MeOH (1.5 mL)/DCM (2.5 mL)	87
11	C(+)/Pt(−) instead of C(+)/Ni(−)	31
12	without KBr	NR
13	without 18-crown-6	trace
14	without current	NR

^aReaction conditions: **1a** (0.2 mmol), **2a** (1.0 mL), KBr (0.2 mmol), 18-crown-6 (0.2 mmol), MeCN (3.0 mL), C plate (15 mm × 10 mm × 2.0 mm) anode, Ni gauze (10 mm × 10 mm × 1.0 mm) cathode, the distance between the electrodes (10 mm), constant current = 6 mA, rt, 10 h, undivided cell. ^bIsolated yields. TBAI = tetrabutylammonium iodide. TBAB = tetrabutylammonium bromide. NR = no reaction.

Supporting Information (SI) for details). To our delight, a quantitative yield of the target product **3a** was obtained by constant current electrolysis of 6 mA at room temperature for 10 h with KBr and 18-crown-6 in MeCN (entry 1). Only electrolytes containing bromine ions can realize the Hofmann rearrangement of phthalimide (entries 2–5). Varying the current strength resulted in lower reaction efficiencies (entries 6–7). Then, the effect of the solvent was explored; changing the

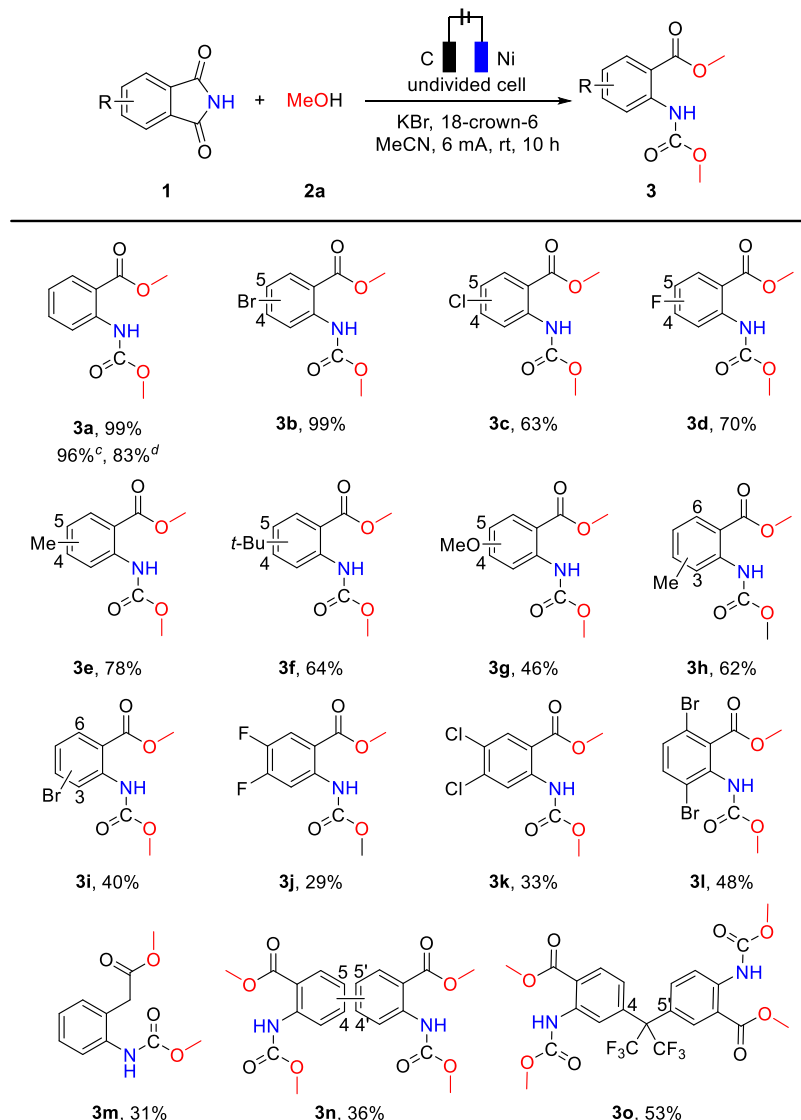
cosolvent or their loading also led to the reduction of isolated yields (entries 8–10). Only a moderate yield of **3a** was obtained when a platinum plate was utilized as the cathode (entry 11). The blank experiment showed that electrolyte KBr is indispensable for this rearrangement reaction (entry 12). The voltage of the reaction system increased significantly in the absence of 18-crown-6, and the Hofmann rearrangement hardly occurred (entry 13). More importantly, no desired product was observed when the reaction proceeded without current, manifesting that this rearrangement procedure was driven by electricity (entry 14).

With the optimal reaction conditions in hand, the scope and limitations of phthalimide **1** were subsequently investigated. The electronic properties of substituents were studied by using monosubstituted phthalimides as substrates (**3b–3i**). As shown in Scheme 2, the reactions of methanol with 4-substituted phthalimides attached to electron-withdrawing groups such as Br, Cl, and F gave the corresponding monosubstituted anthranilate derivatives **3b–3d** in good yield (63–99%). The reaction activities of electron-donating group (methyl, *tert*-butyl, and methoxy)-substituted phthalimides gradually decreased with the increase of electron-donating abilities of functional groups, and the target rearrangement products were obtained in yields of 78% (**3e**), 64% (**3f**), and 46% (**3g**), respectively. 3-Methylphthalimide and 3-bromophthalimide were also smoothly converted into their corresponding products **3h** and **3i** in moderate yields. For symmetrical phthalimide derivatives, for example, 4,5-difluoro-, 4,5-dichloro-, and 3,6-dibromophthalimides, the electrochemical rearrangement reactions proceeded well to assemble the target products **3j–3l** in acceptable yields. Interestingly, when isoquinoline-1,3(2*H*,4*H*)-dione was used as the substrate, only the single product **3m** was obtained. Under standard conditions, using the dimer of phthalimides resulted in the rearrangement of products **3n–3o** in moderate yields. Regrettably, no desired products were detected when maleimide and succinimide were involved in this reaction system under standard conditions.

Having well-constructed structurally diverse anthranilates from varied substituted phthalimides, plenty of aliphatic alcohols were also examined (Scheme 3). The use of other alcohols instead of MeOH as solvents for the electrochemical rearrangement of **1a** provided the corresponding products **4a–4j** in moderate to good yields (39–75%). Aliphatic primary alcohols with the C₂–C₁₀ alkyl chains can be successfully involved as the reaction medium and starting materials. It is worth noting that phthalimide in ethylene glycol can also undergo Hofmann rearrangement to obtain the desired product **4j** in 59%. Under standard conditions, other oxygen-containing nucleophiles such as benzyl alcohol, phenol, and diphenyl carbinol can not deliver the Hofmann rearrangement products.

Compounds marked with deuterium atoms play a very important role in the research of pharmaceutical chemistry and organic chemistry mechanisms. It was found that under standard conditions when deuterated methanol was used as the solvent, the corresponding deuterated anthranilates **6a–6f** were generated with good yields (44–83%) and equal deuteration rates (Scheme 4).

In addition, the obtained rearrangement product **3a** can be utilized as a feedstock for some meaningful derivatization through hydrolysis, transesterification, and cyclization, producing the corresponding products 7–9 with good yields (Scheme 5). These diverse transformations highlight the synthetic value of electrogenerated anthranilate derivatives.

Scheme 2. Substrate Scope of Phthalimides 1^{a,b}

^cGram-scale at 10 mmol. ^dGram-scale at 50 mmol. ^aReaction conditions: **1** (0.2 mmol), **2a** (1.0 mL), KBr (0.2 mmol), 18-crown-6 (0.2 mmol), MeCN (3.0 mL), C plate (15 mm × 10 mm × 2.0 mm) anode, Ni gauze (10 mm × 10 mm × 1.0 mm) cathode, the distance between the electrodes (10 mm), constant current = 6 mA, rt, 10 h, undivided cell. ^bIsolated yields.

Cyclic voltammetry experiments were performed to probe the reaction procedure (Figures 1 and 2). A significant increase in anodic peak current was observed with the addition of 18-crown-6 to KBr (Figure 1, green curve). The reduction peak of MeOH (**2a**) at 0.23 V was observed (Figure 2, blue curve). In contrast, no obvious new peak was observed in the CV of **1a** (Figure 2, red curve).

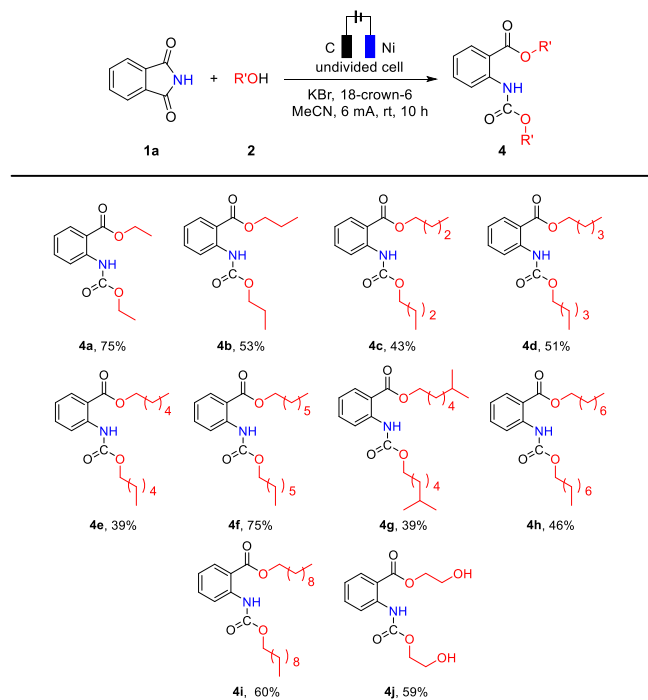
Based on the above experimental results and some related literature, a possible reaction mechanism is proposed (Scheme 6). A bromide anion is initially oxidized at the anode to Br₂, which interacts with phthalimide **1** to form the reaction intermediate **I** by the formation of the N–Br bond. Meanwhile, alkoxide anion **II** and hydrogen gas are produced by the cathodic reduction of R'OH **2**. Then, the ring-opening of intermediate **I** by alcoholysis delivers isocyanate **III**, which is further attacked by the second alkoxide anion and then protonation to give the desired rearrangement product **3**.

CONCLUSIONS

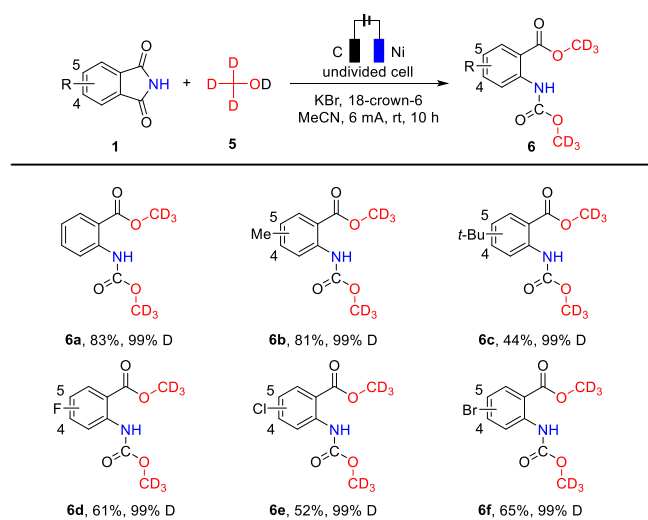
In summary, an electrochemical Hofmann rearrangement is developed with the mediation of KBr and 18-crown-6. This efficient and green approach exhibits high atom economy, good functional group tolerance, and operational simplicity under external oxidant- and transition-metal catalyst-free conditions. The combination of phthalimides and alcohols constructs a variety of useful aminobenzoate building blocks, especially for the efficient introduction of deuterium atoms, and this transformation can be a valuable tool for the preparation of potentially biologically active compounds. More importantly, the practicability of the methodology is preliminarily proven by synthetic transformations and scale-up experiments.

MATERIALS AND METHODS

¹H, ¹³C, and ¹⁹F NMR spectra were recorded on a Varian Inova-400 spectrometer. ¹H and ¹³C NMR chemical shifts were determined relative to internal standard TMS at δ 0.0 ppm or

Scheme 3. Substrate Scope of Alcohols 2^{a,b}

^aReaction conditions: **1a** (0.2 mmol), **2** (1.0 mL), KBr (0.2 mmol), 18-crown-6 (0.2 mmol), MeCN (3.0 mL), C plate (15 mm × 10 mm × 2.0 mm) anode, Ni gauze (10 mm × 10 mm × 1.0 mm) cathode, the distance between the electrodes (10 mm), constant current = 6 mA, rt, 10 h, undivided cell. ^bIsolated yields.

Scheme 4. Synthesis of Deuterated Anthranilates^{a,b}

^aReaction conditions: **1** (0.2 mmol), **5** (1.0 mL), KBr (0.2 mmol), 18-crown-6 (0.2 mmol), MeCN (3.0 mL), C plate (15 mm × 10 mm × 2.0 mm) anode, Ni gauze (10 mm × 10 mm × 1.0 mm) cathode, the distance between the electrodes (10 mm), constant current = 6 mA, rt, 10 h, undivided cell. ^bIsolated yields.

CDCl₃ (δ (¹H), 7.26 ppm; δ (¹³C), 77.16 ppm). Chemical shifts (δ) are reported in ppm, and coupling constants (*J*) are reported in Hertz (Hz). The following abbreviations are used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broadened. The high-resolution mass spectrometry (HRMS) analysis was performed on an Agilent

Scheme 5. Synthetic Transformations

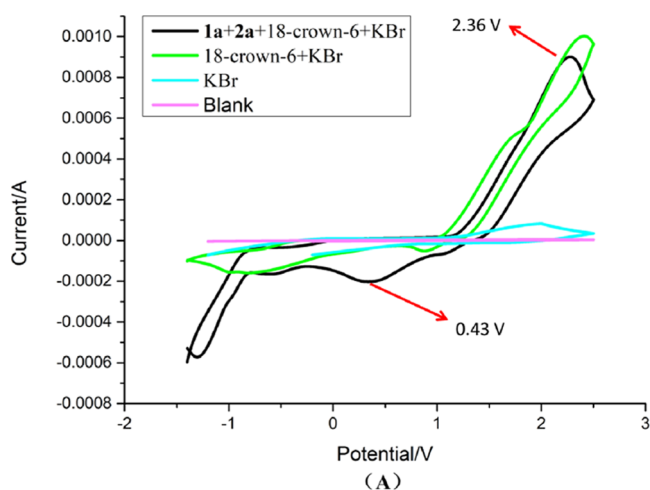
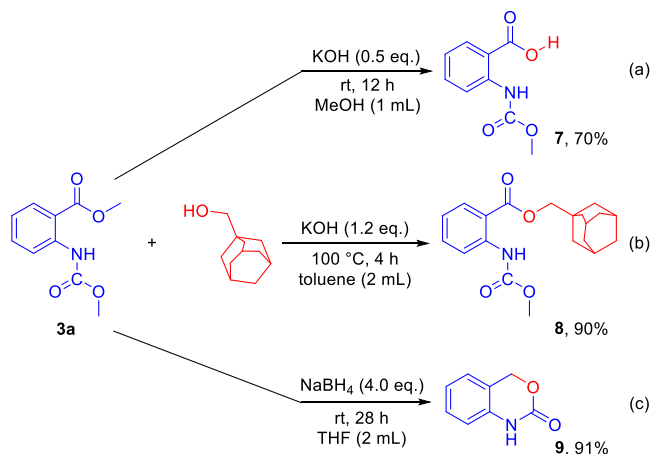


Figure 1. Cyclic voltammograms of **1a** and **2a** in MeCN with KBr (0.05 M) and 18-crown-6 (0.05 M) under air. Cyclic voltammograms were performed using a glassy carbon disk as the working electrode and a Pt disk and Ag/AgCl as the counter and reference electrode, respectively, at a 100 mV/s scan rate.

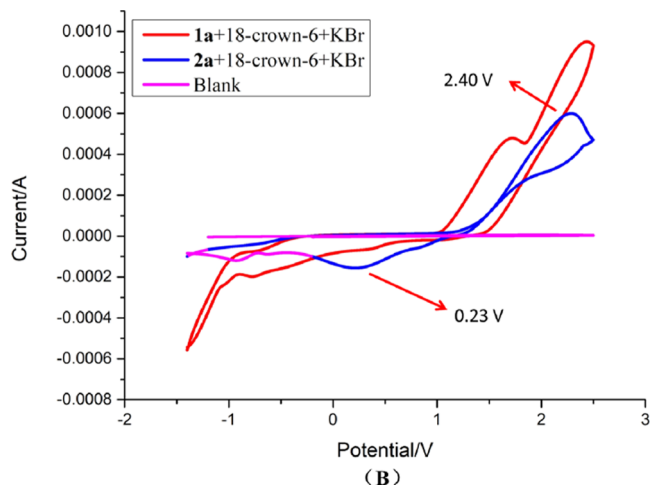
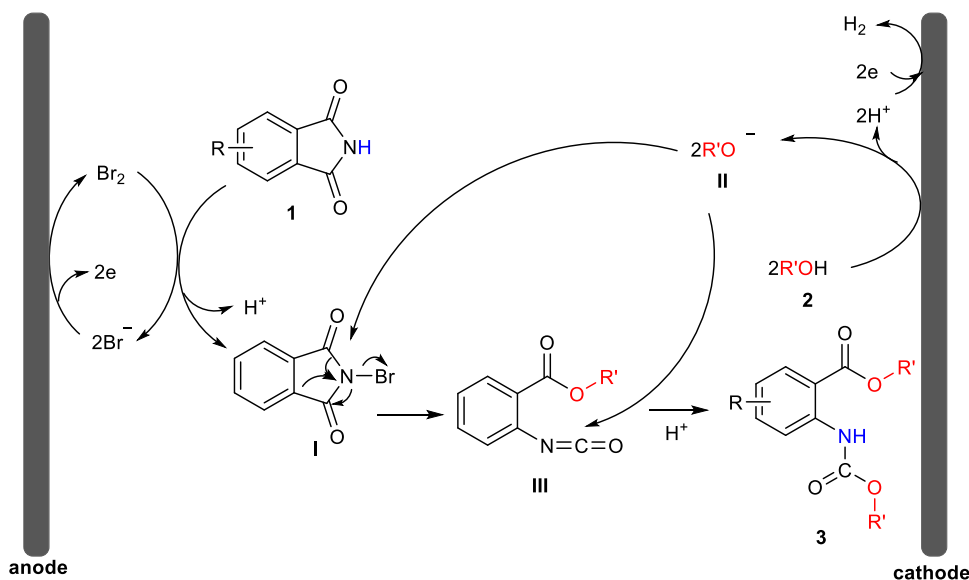


Figure 2. Cyclic voltammograms of **1a**, **2a**, and background. Cyclic voltammograms using a glassy carbon disk as the working electrode and Pt disk and Ag/AgCl as counter and reference electrodes, respectively, at a 100 mV/s scan rate.

Scheme 6. Proposed Mechanism



6540 UHD quadrupole time-of-flight (Q-TOF) mass spectrometer. The melting point was recorded on a BÜCHI (M-560) and uncorrected. Analytical thin-layer chromatography (TLC) was performed on 0.25 mm silica gel 60 F254 plates and viewed with a UV light (254 nm). Column chromatographic purification was performed by using 200–300 mesh silica gel. Electrochemical Hofmann rearrangement reactions were performed on a DC Power Supply (GPD-2303S). The H₂ detection experiment was conducted on an ES20B–H₂ gas detector (Shenzhen Eyesky Technology Co., Ltd.). Cyclic voltammetry (CV) was carried out on a CHI660E electrochemical workstation (CH Instruments, Ins). All commercial reagents and solvents were purchased from commercial sources and used as received unless otherwise indicated.

Detailed synthetic methods and characterization data are provided in the [Supporting Information](#).

■ ASSOCIATED CONTENT

SI Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.3c04797>.

Experimental materials and procedures and NMR of compounds ([PDF](#))

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Notes

The authors declare no competing financial interest.

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