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Electrochemical Di-functionalization: Oxidative Amination and Oxygenation of 4-Hydroxy-α-Benzopyrones under Ring Contraction

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ABSTRACT

We report a catalyst-free, singlet oxygen-mediated electrochemical rearrangement and di-functionalization protocol for the green synthesis of biorelevant 2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamides from 4-hydroxy-α-benzopyrones. The transformation is carried out using lithium perchlorate as a cost-effective and eco-friendly electrolyte in a 1,4-dioxane:water solvent system at ambient temperature. The proposed mechanism is well supported by systematic control experiments. This one-pot strategy features a broad substrate scope, mild reaction conditions, and delivers good to excellent yields without the need for external catalysts. Key advantages of this method include operational simplicity, environmental sustainability, and high energy efficiency, making it a valuable addition to the toolbox of green synthetic methodologies.

Introduction

Benzofuranones are privileged scaffolds widely embedded in both natural products and synthetic compounds, renowned for their diverse and potent biological and pharmacological activities [1]. Moreover, benzofuranones serve as valuable synthetic intermediates in the construction of complex natural products and their structurally diverse derivatives [2]. Within the benzofuranone class, benzofuran-3(2*H*)-ones stand out as a particularly significant subclass – especially their 2,2-disubstituted derivatives [3], which are frequently encountered in bioactive natural products exhibiting a wide spectrum of pharmacological properties, including antibiotic, antioxidant, antipsychotic, antidiabetic, anticancer, anti-HIV activities, and more [4]. Fig. 1 presents key naturally occurring 2,2-disubstituted benzofuran-3(2*H*)-ones known for their biological relevance.

The impressive pharmacological profile and wide-ranging bioactivities of this class of compounds have garnered growing attention from the synthetic chemistry community, inspiring numerous efforts to construct 2,2-disubstituted benzofuranone frameworks [5]. Yet, many of the existing methods are burdened by drawbacks such as multistep operations, toxic reagents or solvents, expensive catalysts, and harsh reaction conditions. In response to these challenges, we recently introduced a photocatalytic protocol enabling the efficient synthesis of 2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamides under mild, ambient conditions [6]. Despite its advantages, the photochemical method presents certain limitations, including a narrow substrate scope, extended reaction times, and limited scalability. To address these challenges, our ongoing research in green chemistry [7] has been directed toward developing a more versatile and broadly applicable strategy for synthesizing this class of biologically significant compounds. In pursuit of a more practical and sustainable approach, I have now developed an efficient electrosynthetic strategy for the preparation of a wide array of structurally diverse 2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamides. This transformation proceeds *via* an electrochemical rearrangement involving ring contraction and di-functionalization of 4-hydroxy-α-benzopyrones upon reaction with amines in an undivided electrochemical cell. The reaction employs stainless steel plates as both the anode and cathode, operates under a constant direct current (10 mA), and utilizes lithium perchlorate ($LiClO_4$) as the electrolyte in a 1,4-dioxane:water solvent system. Notably, the process is conducted in the presence of oxygen gas at ambient temperature (25–28 °C), offering a green and operationally simple route to access this valuable class of compounds (Scheme 1). This newly developed electrochemical protocol offers significant advantages over our previously reported photochemical approach, notably in terms of higher yields, shorter reaction times, and improved scalability. Electrochemistry has emerged as a powerful

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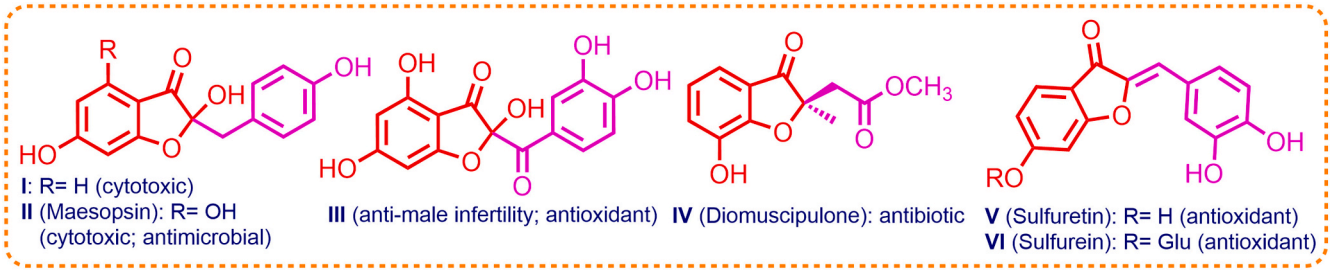
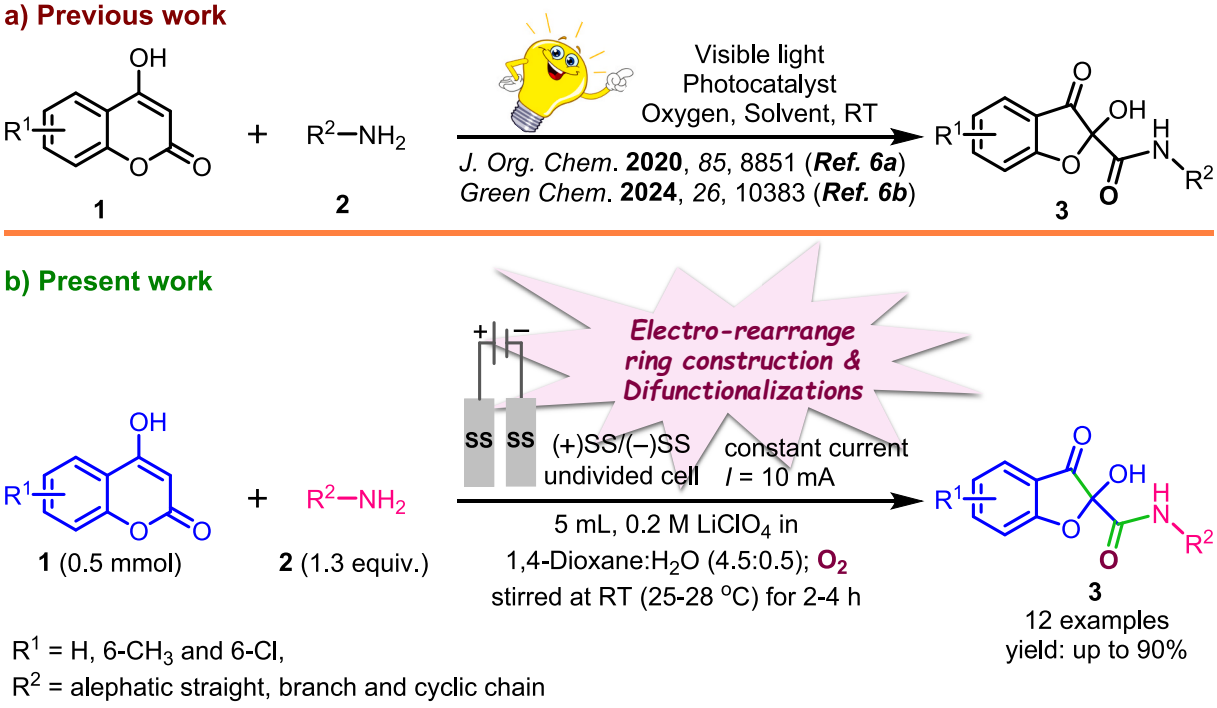


Fig. 1. Structurally diverse, bioactive 2,2-disubstituted benzofuran-3(2*H*)-ones found in nature.



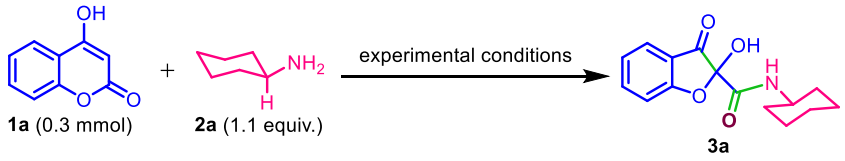
Scheme 1. Electrochemical synthesis of substituted 2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxylates (**3**).

and versatile tool in contemporary synthetic chemistry, gaining traction in both academic and industrial settings for its energy efficiency, mild reaction conditions, and environmentally benign nature [8].

Results and discussion

To optimize the reaction conditions for this electro-rearrangement-based ring contraction and difunctionalization process, we initiated our study with a series of trial reactions using 4-hydroxycoumarin (**1a**) and cyclohexylamine (**2a**) as the model substrates. As a starting point, we stirred a mixture of **1a** and **2a** in 1,4-dioxane:water (4:1) under ambient conditions and open air. However, no reaction was observed even after 6 h (Table 1, entry 1). The addition of 0.1 M lithium perchlorate ($LiClO_4$) to the reaction mixture did not alter the outcome, as no noticeable reaction progress was observed under the same conditions (Table 1, entry 2). Next, we explored an electrochemical approach for the model reaction using stainless steel (SS/SS) electrodes in an undivided cell, applying a constant direct current (DC) of 10 mA. The reaction was conducted under ambient conditions in open air, with 0.1 M $LiClO_4$ as the supporting electrolyte (Table 1, entry 3). To our satisfaction, the reaction proceeded smoothly and afforded the target product, *N*-cyclohexyl-2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamide (**3a**), in 48 % yield within 4 h. We then investigated the influence of water content in the 1,4-dioxane solvent system and found that adjusting the ratio to 4.5:0.5 (dioxane:water) impacted the reaction outcome (Table 1, entries 4–6). We further evaluated the effect of electrolyte concentration on the model reaction using SS/SS electrodes in an undivided cell under a constant DC current of 10 mA. When the reaction was performed in acetonitrile under open-air conditions, the desired product 3a was obtained in 76 % and 74 % yields at 4 h using 0.2 M and 0.3 M $LiClO_4$, respectively (Table 1, entries 7–8). Interestingly, when the reaction was carried out under a nitrogen atmosphere while keeping all other conditions constant, no product formation was observed even after 6 h (Table 1, entry 9). In contrast, performing the same reaction under an oxygen atmosphere led to a smooth transformation, affording the desired product **3a** in 80 % yield within just 3 h (Table 1, entry 10). Altering the applied direct current from the optimal value led to diminished product yields. A lower current of 6 mA afforded a 65 % yield after 6 h (Table 1, entry 11), while increasing the current to 15 mA gave a slightly improved but still suboptimal yield of 79 % at 3 h (Table 1, entry 12). To further refine the reaction conditions, we conducted additional trials of the model reaction using various supporting electrolytes, including $LiC_2O_4BF_2$, $^nBu_4NClO_4$, $NaClO_4{\cdot}H_2O$, $^nBu_4NBF_4$, Me_4NBF_4, $^nBu_4NPF_6$, $^nBu_4NI$, KI, KBr, and KCl. Each was tested at a concentration of 0.2 M in a 1,4-dioxane:water mixture (4.5:0.5) under galvanostatic conditions (SS/SS electrodes, I = 10 mA) in an oxygen atmosphere at ambient temperature (25–28 °C). However, none of these electrolytes facilitated the formation of the desired product 3a within 6 h

Table 1
Optimization of reaction conditions for the synthesis of *N*-cyclohexyl-2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamide (**3a**).



Entry	Electrolyte (M)	Cell (+ | -)	Solvent (5 mL)	Atmosp.	Current (mA)	Time (h)	Yield (%)[a,b]
1	–	SS | SS	Dioxane:H_2O (4:1)	Air	–	6	–
2	$LiClO_4$ (0.1)	SS | SS	Dioxane:H_2O (4:1)	Air	–	6	–
3	$LiClO_4$ (0.1)	SS | SS	Dioxane:H_2O (4:1)	Air	10	4	48
4	$LiClO_4$ (0.1)	SS | SS	Dioxane:H_2O (1:4)	Air	10	6	–
5	$LiClO_4$ (0.1)	SS | SS	Dioxane	Air	10	6	–
6	$LiClO_4$ (0.1)	SS | SS	Dioxane:H_2O (4.5:0.5)	Air	10	4	59
7	$LiClO_4$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	Air	10	4	76
8	$LiClO_4$ (0.3)	SS | SS	Dioxane:H_2O (4.5:0.5)	Air	10	4	74
9	$LiClO_4$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	N_2	10	6	–
10	$LiClO_4$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	3	80
11	$LiClO_4$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	6	6	65
12	$LiClO_4$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	15	3	79
13	$LiC_2O_4BF_2$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	6	Trace
14	$^nBu_4NClO_4$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
15	$NaClO_4{\cdot}H_2O$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	3	Trace
16	$^nMe_4NBF_4$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
17	$^nBu_4NBF_4$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
18	$^nBu_4NPF_6$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
19	$^nBu_4NI$ (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
20	KI (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
21	KBr (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
22	KCl (0.2)	SS | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
23	$LiClO_4$ (0.2)	SS | Pt	Dioxane:H_2O (4.5:0.5)	O_2	10	6	Trace
24	$LiClO_4$ (0.2)	Pt | SS	Dioxane:H_2O (4.5:0.5)	O_2	10	6	Trace
25	$LiClO_4$ (0.2)	SS | C	Dioxane:H_2O (4.5:0.5)	O_2	10	6	Trace
26	$LiClO_4$ (0.2)	SS | cu	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
27	$LiClO_4$ (0.2)	SS | Zn	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
28	$LiClO_4$ (0.2)	SS | au	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
29	$LiClO_4$ (0.2)	SS | cu	Dioxane:H_2O (4.5:0.5)	O_2	10	6	–
30	$LiClO_4$ (0.2)	Pt | Pt	Dioxane:H_2O (4.5:0.5)	O_2	10	6	Trace
31	$LiClO_4$ (0.2)	C | C	Dioxane:H_2O (4.5:0.5)	O_2	10	6	Trace
32	$LiClO_4$ (0.2)	SS | SS	CH_3CN:H_2O (4.5:0.5)	O_2	10	6	–
33	$LiClO_4$ (0.2)	SS | SS	DMSO:H_2O (4.5:0.5)	O_2	10	6	–
34	$LiClO_4$ (0.2)	SS | SS	DMF:H_2O (4.5:0.5)	O_2	10	6	–
35	$LiClO_4$ (0.2)	SS | SS	DCE:H_2O (4.5:0.5)	O_2	10	6	–

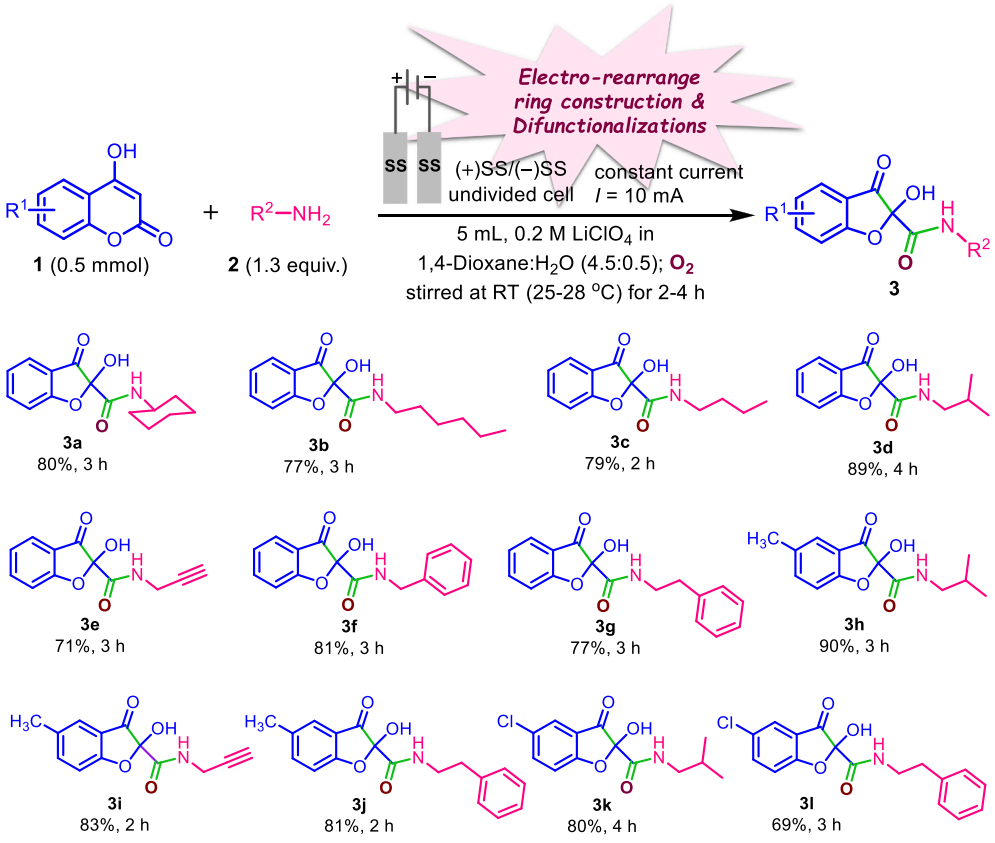
[a] Reaction conditions: A mixture of 4-hydroxycoumarin (**1a**; 0.5 mmol) and *c*-hexylamine (**2a**; 1.3 equiv.) dissolved in a different electrolyte solution(s) at a constant current in an undivided cell at 0.5 cm distance at room temperature (25–28 °C). Electrode size: graphite, Pt, SS, Cu, Zn, and Ag plates: 0.7 cm × 0.7 cm × 0.2 cm. [b]Isolated yields. M = molarity.

(Table 1, entries 13–22). Changing the electrode pairs to alternatives such as SS/Pt, Pt/SS, SS/C, SS/Cu, SS/Zn, SS/Au, Pt/Pt, and C/C, while keeping all other reaction parameters constant, also proved ineffective (Table 1, entries 23–31). We also tested four additional solvent systems – CH_3CN:H_2O, DMSO:H_2O, DMF:H_2O, and DCE:H_2O – while keeping all other reaction conditions unchanged. However, none of these reactions showed any progress after 6 h (Table 1, entries 32–35). Ultimately, the optimal conditions for the electrochemical reaction between 4-hydroxycoumarin (**1a**) and cyclohexylamine (**2a**) were established based on yield and reaction time. The best result was obtained using stainless steel (SS) plates as both the anode and cathode in an undivided cell, with 0.2 M lithium perchlorate ($LiClO_4$) as the supporting electrolyte in a 1,4-dioxane:water (4.5:0.5) solvent system. The reaction was conducted under an oxygen atmosphere at ambient temperature (25–28 °C) with a constant direct current (DC) of 10 mA. Under these conditions, the desired product, *N*-cyclohexyl-2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamide (**3a**), was obtained in 80 % yield within 3 h (Table 1, entry 10). A comprehensive summary of all the electrosynthetic trials is presented in Table 1. Compound **3a** was fully characterized by detailed spectroscopic analyses, including ^1H NMR, ^{13}C NMR, DEPT-135, and HRMS, and its physical and spectral data were found to be consistent with those reported in the literature [6]. (*see Supplementary material*).

With the optimized reaction conditions established, we proceeded to evaluate the scope of the protocol by performing fifteen reactions between 4-hydroxycoumarin (**1a**) and various aliphatic amines, including *n*-hexylamine (**2b**), *n*-butylamine (**2c**), and isobutylamine (**2d**). All reactions proceeded smoothly under the standard conditions, affording the corresponding products – *N*-hexyl-2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamide (**3b**), *N*-butyl-2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamide (**3c**), and 2-hydroxy-*N*-isobutyl-3-oxo-2,3-dihydrobenzofuran-2-carboxamide (**3d**) – in good yields ranging from 77 %, 79 % and 89 % respectively, within 2–4 h (Table 2, compounds **3b-3d**). Propargylamine (**2e**) also participated effectively in the reaction with 4-hydroxycoumarin (**1a**) under the same optimized conditions, yielding 2-hydroxy-3-oxo-*N*-(prop-2-yn-1-yl)-2,3-dihydrobenzofuran-2-carboxamide (**3e**) in 71 % yield after 3 h (Table 2, compounds **3e**). Furthermore, the protocol was extended to benzylamine (**2f**) and phenylethylamine (**2 g**), both of which reacted efficiently with 4-hydroxycoumarin (**1a**) under the standard conditions. The reactions furnished the desired products – *N*-benzyl-2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamide (**3f**) and 2-hydroxy-3-oxo-*N*-phenethyl-2,3-dihydrobenzofuran-2-carboxamide (**3 g**) – in excellent yields of 81 % and 77 %, respectively, within 3 h (Table 2, compounds **3f-3 g**).

Building on these results, we next explored the reactivity of

Table 2
Electrochemical synthesis of diversely functionalized 2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamides (3)[a,b].

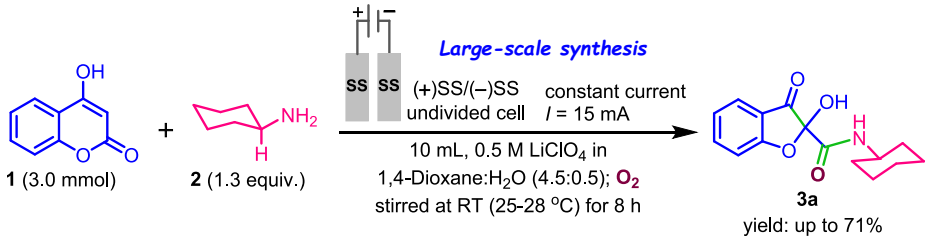


[a]Standard conditions: A mixture of 4-hydroxycoumarins (**1**; 0.5 mmol) and amones (**2**; 0.65 mmol) was electrolyzed in the presence of 0.1 M lithium perchlorate ($LiClO_4$) in 1,4-dioxane:water (4.5:0.5; 5.0 mL) at a constant direct current (I = 10 mA) in an undivided cell using stainless steel (SS) plates as both anode and cathode) at a distance of 0.5 cm under an oxygen atmosphere at ambient temperature (25–28 °C). [b]Isolated yield.

substituted 4-hydroxycoumarins by conducting three additional reactions using 4-hydroxy-6-methylcoumarin (**1b**) with different amines – namely isobutylamine, propargylamine, and phenylethylamine – under the optimized conditions. All reactions proceeded smoothly, delivering the corresponding products: 2-hydroxy-*N*-isobutyl-5-methyl-3-oxo-2,3-dihydrobenzofuran-2-carboxamide (**3 h**), 2-hydroxy-5-methyl-3-oxo-*N*-(prop-2-yn-1-yl)-2,3-dihydrobenzofuran-2-carboxamide (**3i**), and 2-hydroxy-5-methyl-3-oxo-*N*-phenethyl-2,3-dihydrobenzofuran-2-carboxamide (**3j**), in good to excellent yields of 90 %, 83 %, and 81 %, respectively, within 2–3 h (Table 2, compounds **3 h**-**3j**). We next explored the reactivity of a halogenated coumarin derivative, 6-chloro-4-hydroxycoumarin (**1c**), under the established electrochemical conditions. Reactions with isobutylamine and phenylethylamine proceeded smoothly, delivering the corresponding products – 5-chloro-2-hydroxy-*N*-isobutyl-3-oxo-2,3-dihydrobenzofuran-2-carboxamide (**3 k**) and 5-chloro-2-hydroxy-3-oxo-*N*-phenethyl-2,3-dihydrobenzofuran-2-carboxamide (**3 l**) – in good to excellent yields of 80 % and 69 %, respectively, within 3–4 h (Table 2, compounds **3 k**-**3 l**). However, aromatic amines such as aniline and *p*-toluidine failed to undergo the reaction under the optimized conditions. This observation is consistent with our proposed mechanism (Scheme 4), which suggests that the aromatic ring in these amines, through its strong +M (mesomeric) effect, stabilizes the lone pair on the nitrogen atom and thereby inhibits the formation of the amine-derived radical cation (**2′**) *via* single-electron oxidation at the anode. These synthetic outcomes are collectively summarized in Table 2.

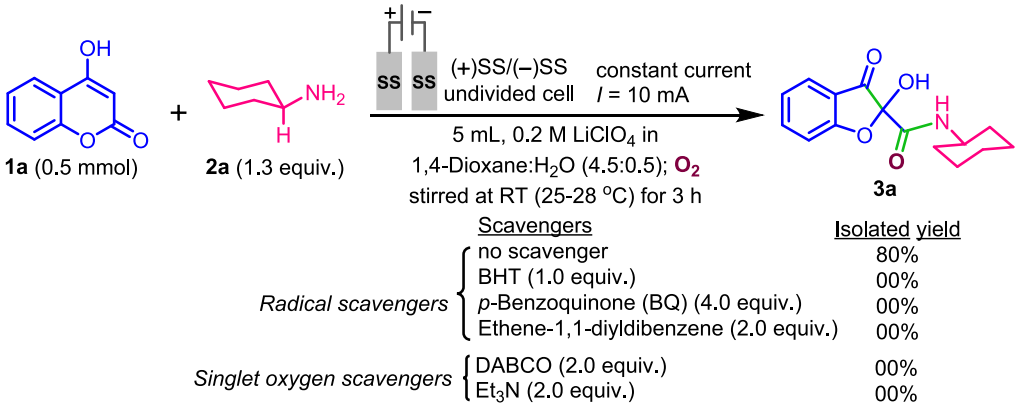
All synthesized compounds **3** (**3a**-**3 k**; 12 compounds in total) were purified by flash column chromatography. As all the target compounds are known, each was characterized by spectral analysis, including ^{1}H NMR and high-resolution mass spectrometry (HRMS), to confirm their identities (*see Supplementary material*).

We also explored the scalability of this protocol by conducting a

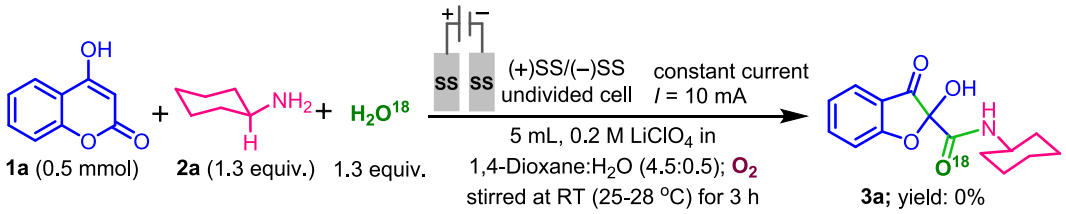


Scheme 2. Larger-scale synthetic applications.

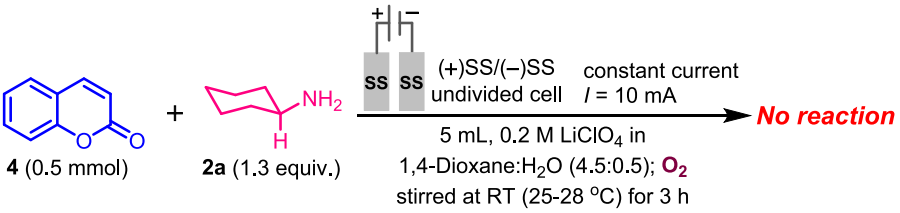
a) Control experiments with radical and singlet oxygen scavengers



b) Control experiment for the source of the inserted oxygen atom



c) Control experiment to establish the role of C_4-hydroxy group of coumarin moiety



Scheme 3. Control experiments.

larger-scale reaction (3.0 mmol; six-fold increase) for the representative compound **3a** (Scheme 2). The reaction proceeded smoothly, affording the product in 71 % yield after 8 h (*see Supplementary material*). The yield was comparable to that obtained on the millimolar scale, although a slightly extended reaction time was required for completion.

At this point, we aimed to gain insight into the possible mechanism underlying the electro-rearrangement ring contraction and difunctionalization reaction between 4-hydroxycoumarins (**1**) and amines (**2**), which affords 2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamides (**3**). To probe the mechanistic pathway, we conducted a series of carefully designed control experiments using the model reaction system (Scheme 3). When the model reaction was carried out under the standard conditions in the presence of various radical scavengers – such as BHT, *p*-benzoquinone, and ethene-1,1-diyldibenzene – as well as singlet oxygen quenchers like DABCO and triethylamine (Et_3N), the formation of the desired product **3a** was completely suppressed (Scheme 3a). These results strongly support the involvement of a radical-mediated pathway and indicate the participation of singlet molecular oxygen in the reaction mechanism. Further insight into the oxygen source was gained through an isotope labeling experiment using H_2O^{18} (1.3 equiv), which failed to yield the product under otherwise identical conditions (Scheme 3b). This result indicates that the incorporated oxygen atom does not originate from water, but exclusively from molecular oxygen, which is presumed to be converted from its triplet ground state to the reactive singlet state under electrochemical conditions. Moreover, the C_4-hydroxy group of the coumarin framework was found to be crucial for the reaction, as evidenced by the failure of unsubstituted parent coumarin

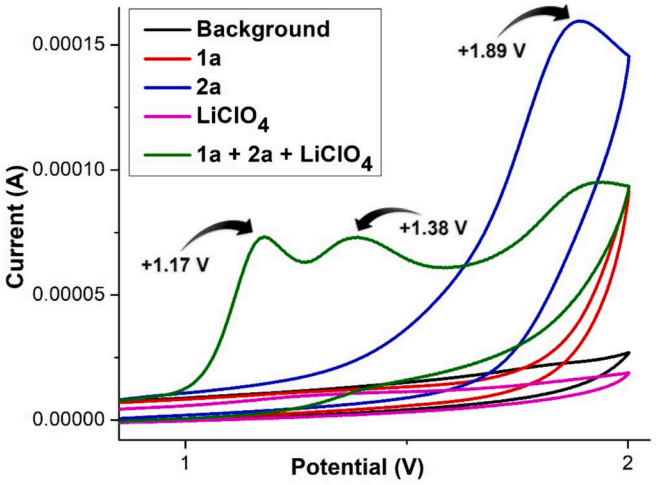
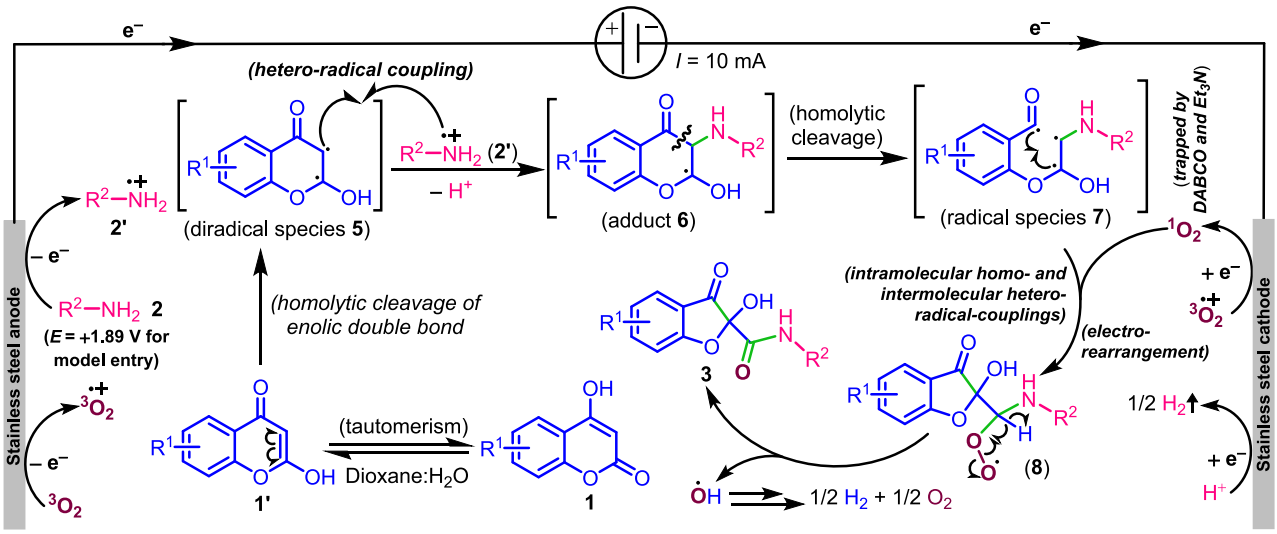


Fig. 2. Cyclic voltamogram.

(**4**) to undergo the transformation under identical conditions (Scheme 3c).

Drawing on the results from detailed control experiments and cyclic voltammetry analyses (Fig. 2), as well as literature support [8a,10] for the reaction components, we propose a plausible mechanism for the electrochemically driven ring contraction and difunctionalization



Scheme 4. Proposed mechanism for the electro-rearrangement ring contraction and difunctionalization reaction.

process, as illustrated in Scheme 4. Cyclic voltammetric analysis of the model substrates – 4-hydroxycoumarin (**1a**) and cyclohexylamine (**2a**) – showed that only cyclohexylamine exhibits a distinct oxidation peak at +1.89 V (*vs* Ag/Ag^+). This suggests that, of the two, the amine component (**2**) is preferentially oxidized at the anode, leading to the formation of an amine radical cation (**2′**). Meanwhile, 4-hydroxycoumarin (**1**) undergoes tautomerization in the reaction medium to form its enolic tautomer (**1′**), which subsequently experiences homolytic cleavage at the C═C bond, generating a diradical intermediate (**13**). The two *in situ*-formed radical species – **2′** (from the amine) and **5** (from the coumarin) – then engage in a hetero-radical coupling to afford intermediate **6** (displays well-defined oxidation signals at +1.17 V and + 1.38 V *versus* Ag/Ag^+). This intermediate undergoes further homolytic cleavage of its C(O)-C(NHR_2) bond, leading to the formation of a new radical species, **7**. It is proposed that molecular oxygen ($^3O_2$) undergoes anodic oxidation followed by cathodic reduction under electrochemical conditions, resulting in a spin-state transition from its triplet ground state to the reactive singlet state ($^1O_2$) [9]. The triradical intermediate **7**, generated earlier in the sequence, is then believed to engage in both intramolecular homo-radical and hetero-radical coupling reactions with the *in situ*-formed singlet oxygen. These radical interactions lead to the formation of a benzofuranone intermediate (**8**), which subsequently eliminates a hydroxyl radical (HO·) to afford the final product **3**. The liberated hydroxyl radical is further decomposed into water and molecular oxygen, while protons generated during the process are reduced at the cathode surface, releasing hydrogen gas (Scheme 4).

Conclusion

In summary, we have developed an efficient and operationally simple electrosynthetic protocol for the ring contraction and simultaneous difunctionalization of 4-hydroxy-α-benzopyrones. This unprecedented transformation provides direct access to a range of structurally diverse and biologically relevant 2-hydroxy-3-oxo-2,3-dihydrobenzofuran-2-carboxamides. The reaction proceeds *via in situ* generation of singlet oxygen from molecular oxygen under electrochemical conditions, using lithium perchlorate as a cost-effective supporting electrolyte and a 1,4-dioxane:water mixture as a green solvent system at ambient temperature (25–28 °C). A comprehensive mechanistic rationale has been established, substantiated by systematic control experiments that validate the proposed reaction pathway. Notably, the methodology operates without the need for any additional catalyst. Compared to previously reported photochemical approaches, this electrochemical one-pot strategy offers significant advantages in terms of product yield, shorter reaction times, scalability, and environmental sustainability.

CRediT authorship contribution statement

Indrajit Karmakar: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2025.155839.

Data availability

The data underlying this study are available in the Supplementary material.

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