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Electrochemical synthesis of hemiaminal from protected amino acids: A facile alternative way to access pro-drugs and ADC linkers via decarboxylative hydroxylation.☆

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ABSTRACT

We report a novel and efficient electrochemical method for the synthesis of hemiaminals and related analogues, which are important intermediates in pro-drug and antibody–drug conjugate (ADC) linker design. The protocol employs a decarboxylative hydroxylation strategy that provides a practical alternative to conventional synthetic approaches, enabling broad substrate scope and excellent functional-group tolerance. Notably, challenging secondary and tertiary substrates are readily accommodated. Reaction optimization minimizes the requirement for excess nucleophile, improving atom economy and operational simplicity. The robustness and scalability of the method are further demonstrated by its extension to diverse nucleophiles, including carboxylic acids and lactams, thereby expanding its utility in linker conjugation for pro-drug and ADC applications.

Introduction

Hemiaminals (also known as carbinolamines) are considered as key intermediates formed by the nucleophilic addition of amines to carbonyl compounds, resulting in a functional group that contains both a hydroxyl and an amine attached to the same carbon center [1]. Their transient yet pivotal role in organic transformations underpins numerous synthetic pathways, particularly those involved in imine formation and downstream derivatization strategies used in medicinal chemistry [2,3]. Apart from the widespread utility in heterocycle synthesis, hemiaminals also appear in biochemical transformations and in natural product biosynthesis, where they facilitate key bond-forming events [4].

The strategic integration of hemiaminals into drug discovery becomes even more impactful when considering prodrug design, an established strategy for overcoming limitations in pharmacokinetics, solubility, stability, and targeted delivery [5,6]. Moreover, hemiaminals can act as transient linkers, masked intermediates, or modifiable functional groups, they offer unique opportunities for developing cleavable motifs, tailored release mechanisms, and novel activation pathways. Their inherent reactivity provides a platform for engineering innovative prodrug constructs capable of improved pharmacokinetic profiles, selective tissue targeting, and controlled activation under specific physiological conditions [7]. 'Aripiprazole lauroxil' is an example of a prodrug of a well-known anti-psychotic medication, which is synthesized from hemiaminal intermediate, hydroxymethyl aripiprazole (Scheme-1) [8].

In addition, hemiaminals have been found to be excellent intermediates for accessing substituted chloromethanamines through straightforward chemical transformation either with PCl_5 in chloroform or with thionyl chloride in THF [9,10] or using chloro-trimethyl-silane in solvents like dimethyl sulfoxide [11], which can easily be transformed to synthesize pro-drugs [12]. The example in Scheme 2 shows how the hemiaminal intermediates are employed to construct self immolative linkers for the syntheses of Stimuli-Responsive PROTACs [13], and other degrader antibody conjugates (DAC) [14].

With the increasing emphasis on precision medicine and rational design of drug-delivery systems, understanding and exploiting hemiaminal intermediates is likely to play an increasingly influential role in next-generation therapeutic development. Consequently, many scientific groups have started investigating various new modalities, where linker syntheses are primarily designed using hemiaminal intermediates

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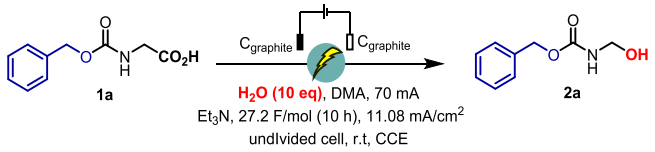
Table 1
Optimization of the reaction parameters.



Entry	Deviations from the standard conditions	%yield[b]
1	None	84[a]
2	Water (2 eq) in DMA	23
3	Water (10 eq) in DMF	75
4	Water (10 eq) in MeCN	62
5	Water (10 eq) in DMSO	59
6	2 h instead of 10 h	15
7	Stainless steel plate as cathode	67
8	Nickel plate as cathode	60
9	Pt plate as cathode	72
10	*n*-Bu_4NBF_4 (0.5 eq) as supporting electrolyte	69
11	K_2CO_3 instead of Et_3N	52
12	Cs_2CO_3 instead of Et_3N	64
13	No current	n.r.

[a] optimized condition- **1a** (0.4 mmol) in DMA (3 ml), methanol (10 eq), Et_3N (5 Mol%), constant current (70 mA), undivided cell, graphite as anode and cathode.
[b] Isolated yield (no side product was found to form; lower yield corresponds to poor conversion from the starting materials). n.r. = no reaction.

[15,16]. The most fundamental synthetic route to hemiaminals involves nucleophilic addition of an amine to an aldehyde or ketone, generating a carbinolamine intermediate.

The classical pathway generally represents the initial stage of imine formation and provides the mechanistic basis for numerous downstream transformations, including Schiff base formation, Mannich-type reactions, and heterocycle assembly [17].

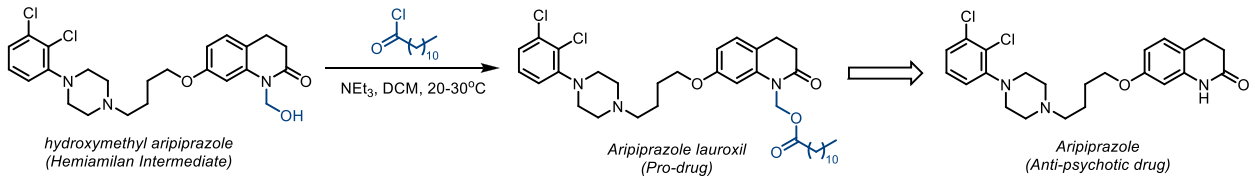
Bois group demonstrated an efficient stereoselective total synthesis of (+)-Saxitoxin, an agent associated with red tides and paralytic shellfish poisoning, by employing crucial transannular nucleophilic addition of amine N to the carbonyl group thereby forming the hemiaminal intermediate (Scheme 3, entry a) [18]. In the year of 2012, Scherr et al. reported synthesis of 4,5-dihydroxyimidazolidin-2-one from urea and glyoxal via a two-fold intermolecular nucleophilic addition reaction (Scheme 3, entry b) [19]. Another well-known method for synthesizing stable hemiaminal synthesis was reported by Dogan and coworkers in 2016, where $LiAlH_4$ mediated carbonyl bond reduction of thiazolidin-4-one afforded a stable hemiacetal derivative with a satisfactory yield [20]. After some years, the same group successfully synthesized a series of 3-(pyridin-2-yl)-2-(pyridin-2-ylimino)thiazolidin-4-ol derivatives regio-selectively from 2-iminothiazolidin-4-ones by the application of the previous method of carbonyl reduction in presence of $LiAlH_4$ (Scheme 3, entry c) [21].

Nevertheless, these protocols are well structured to synthesize corresponding hemiaminals effectively, these methods rely on the direct nucleophilic addition of amines to carbonyl compounds, a process that is often equilibrium-limited and highly sensitive to steric and electronic effects and frequently suffer from poor selectivity and yields. Requirement of stringent anhydrous conditions, cryogenic temperatures, or the use of activated carbonyl substrates to achieve appreciable conversion and limiting substrate scope are some of the prominent drawbacks of these methods. As a result, the synthesis of stable, isolable hemiaminals through conventional condensation methods remains challenging, thereby motivating the need for alternative strategies such as electrochemical, decarboxylative, or radical-based approaches.

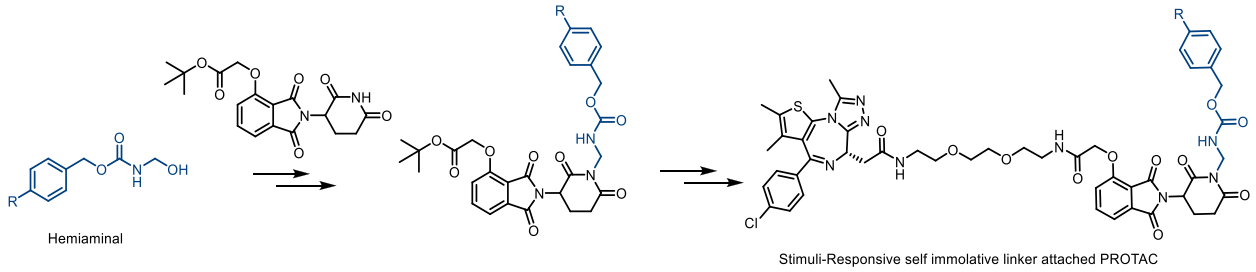
Decarboxylative hydroxylation reaction is particularly attractive for the modification of protected amino acids, where temporary protection of the amino and carboxyl functional groups ensures selective reactivity at the side chain, thus avoiding undesired side reactions. The ability to selectively introduce hydroxyl groups into amino acid derivatives expands the scope of synthetic methods available for peptide and protein engineering, providing access to a diverse array of modified compounds with enhanced bioactivity, stability, or specificity [22]. Consequently, a large number of synthetic groups have started investigating various methods of selective decarboxylative hydroxylation of carboxylic acids and amino acid. Among them, employment of molecular oxygen as the oxidant along with the application of high valent metal ions like Pb (IV), Ti (III), Mn (III), and Ce (IV) in stoichiometric amount as co-oxidant has become popular for an effective decarboxylation of carboxylic acid moiety (Scheme 4, entry a) [23]. In addition, Barton et al. reported indirect decarboxylation process of carboxylic acid by transforming it into a crucial thiohydoxamate ester intermediate (Scheme 4, entry b) [24–25].

In 2016, Lu group reported the first example of visible light induced direct transformation of carboxylic acid to respective alcohol scaffolds in presence of molecular oxygen as the oxidant. This protocol, being metal-free in nature, efficiently performed efficient decarboxylative hydroxylation on several structurally diverse carboxylic acids (Scheme 4, entry c). However, this method was found to be completely unsuccessful to produce corresponding hydroxylated compound from Boc protected L-proline [26]. Recently, Yoshimi group reported another elegant photo-induced method of decarboxylative hydroxylation of α-amino acids to form amino alcohol in presence of 9,10-diphenylanthacene as an electron donor and Selectfluor as both an electron acceptor and oxidant in presence of acetonitrile and water (7:3) co-solvent system (Scheme 4, entry d) [27]. Despite of the mild nature, this process endures with a very limited substrates scope.

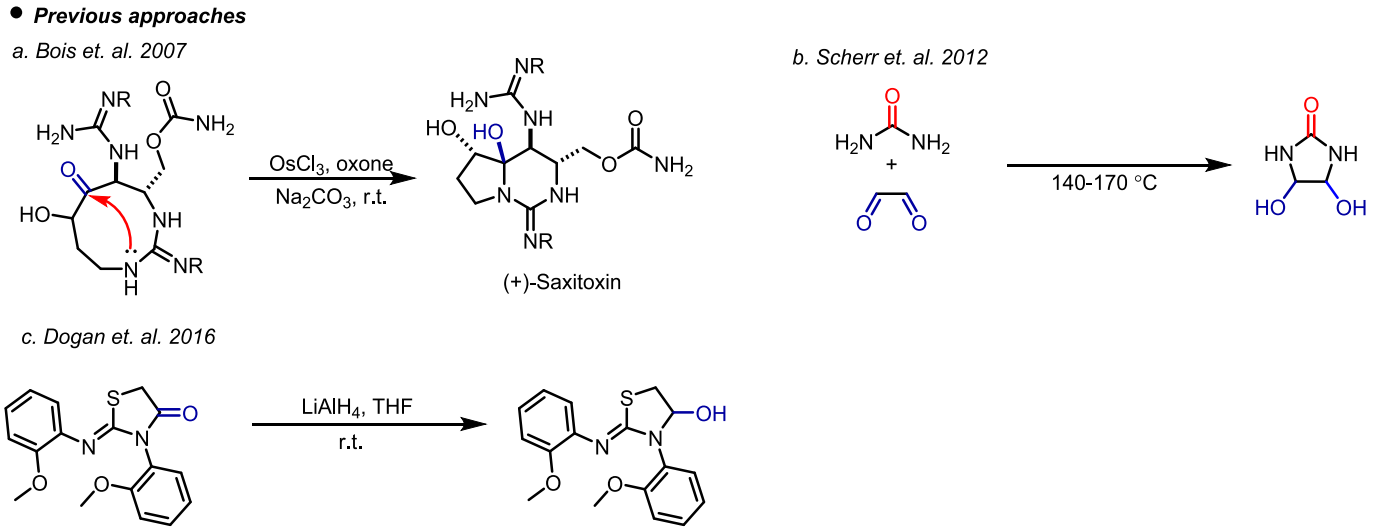
In view of the limitations associated with the previously reported methods, we sought to develop a robust yet mild protocol for the decarboxylative hydroxylation of protected amino acids by exploiting the unique reactivity and selectivity offered by electrochemical synthesis [28]. Recently, our group reported the electrochemical synthesis of self-immolative methylene alkoxy carbamates (MACs) from a variety of alcohols [29]. Inspired by these findings, we envisioned that electrochemistry could provide an efficient platform for the decarboxylative hydroxylation of amino acids. To validate this hypothesis, we undertook a systematic investigation of the electrochemical decarboxylative hydroxylation of N-Cbz-protected amino acids, employing them as model substrates.Herein, we reveal the early findings of synthesizing stable, isolable hemiaminal derivative via an electrochemical decarboxylative



Scheme 1. Preparation of pro-drug Aripiprazole lauroxil



Scheme 2. Application of hemiaminals in the construction of self immolative linker for the synthesis of Stimuli-Responsive PROTACs



Scheme 3. Previous and present synthetic approaches to stable Hemiaminals

hydroxylation on amino acid analogue and further modification of the protocol towards successful application in decarboxylative nucleophile addition to amino acids (Scheme 4).

Results and discussion

In quest of the optimized reaction condition for electrochemical decarboxylative hydroxylation, N-Cbz glycine **1a** was taken as the model substrate. Optimum formation of the hydroxyl amino acid **2a** was achieved by electrolyzing the DMA solution of **1a** at constant current of 70 mA (27.2 F/ mol) in presence of 10 eq of water and catalytic amount of triethylamine. The electrolysis was performed in an undivided cell employing Graphite plate electrode both as anode and cathode at ambient temperature for 10 h.

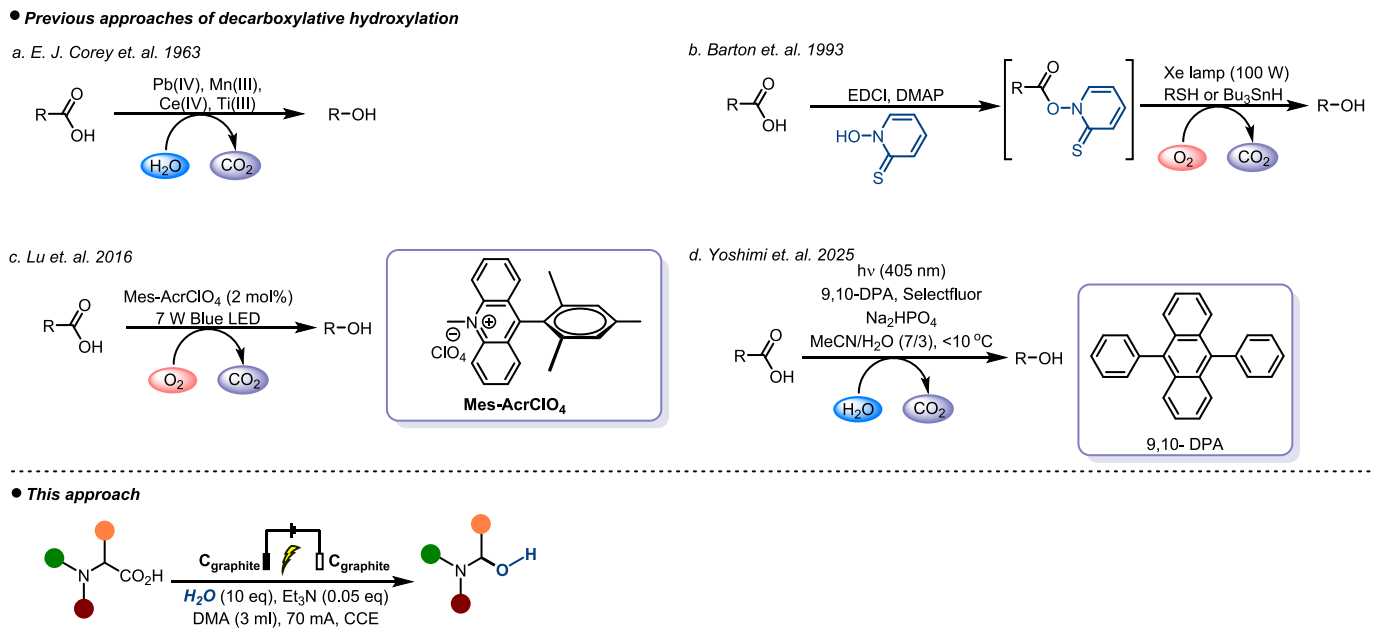
When the equivalence of water was reduced to 2 eq, a drastic decrease in the product yield was observed. (Table 1, entry 2). Electrolysis of the **1a** with 10 eq of water in polar aprotic solvents like DMF, MeCN, and DMSO didn't bring any improvement in the product yield. (Table 1, entry 3–5). A prominent negative effect in the yield of **2a** was observed when the reaction time was reduced to 2 h (Table 1, entry 6).

Metal plate electrodes like stainless steel, Ni plate, and Pt plate were checked in place of graphite. However, product yield was found to be less, probably because of the higher rate of HER reaction on metal electrode surface (Scheme 1, entry 7–9) [30–31].

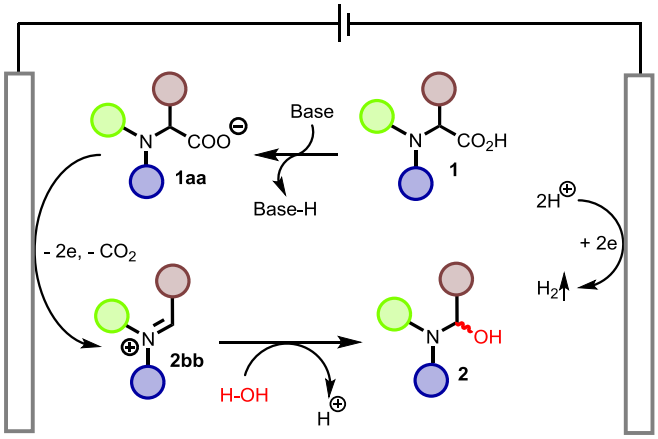
We were curious to know if supporting electrolyte had any beneficial role in this reaction. To check this, non-interfering electrolyte n-Bu_4NBF_4 was employed along with water. Hemiaminal **2a** was obtained with lesser yield. This negative role of supporting electrolyte could be associated with the higher degree of ionization of n-Bu_4NBF_4 than the existing nucleophile (Table 1, entry 10).

The effect of inorganic bases, including K_2CO_3 and Cs_2CO_3, in place of Et_3N was investigated; however, these conditions failed to surpass the optimum yield of **2a** (Table 1, entries 11 & 12). Formation of **2a** was not observed in absence of constant current, which proves the indispensable role of the electricity in this reaction (Table 1, entry 13).

After successfully establishing the optimized condition for electrochemical decarboxylative hydroxylation, we focused our interest on substrate scope exploration. Cbz protected amino acids derived from glycine, *L*-phenyl alanine, *L*-alanine, and *L*-proline respectively were successfully converted to their corresponding hydroxymethylcarabamates



Scheme 4. Decarboxylative hydroxylation approaches



Scheme 5. Plausible mechanism

2a-2d (Table 2, entry 2a-2d) with good to excellent yield.

Scale-up reaction was also performed on Cbz protected glycine **1a** (4.78 mmol) and the desired product **2a** was obtained with slight decreased yield after electrolyzing the DMA solution of **1a** at constant current of 140 mA (Table 2, entry 2a).

Moving forward, the feasibility of the protocol was also checked on the sterically hindered strained cyclopropyl tethered amino acid **1e**, which was transformed to the corresponding strained hydroxylcyclopropyl carbamate **2e** (Table 2, entry 2e). Non-electrochemical conventional decarboxylative transformation required harsh non-environment friendly conditions like refluxing in benzene with $(CF_3CO_2)_2IPh$ and pyridine [32], in contrast, the novel electrochemical condition is proven to be mild and environment friendly.

In the transformation, to check if there is any potential role of the 'H' attached to the Nitrogen of the amino acid, *N*-methyl analogue **1f** was subjected to the reaction under the optimized condition to afford hydroxymethyl carbamate **2f** with satisfactory yield (Table 2, entry 2e), suggesting the robust nature of the process. Likewise, upon application of the optimized condition on the model compound **1 g**, hydroxyl methyl carbamate **2 g** with an azide substitution on the aryl ring was isolated with good yield (Table 2, entry 2f). This model reaction with an aryl azide was planned in order to extend the application to construct PABC linkers ideally suitable for syntheses of pro-drugs and ADC compounds, converting the azide functionality to amine followed by amidation using Staudinger ligation condition.

To further establish the method's generality, dipeptide molecule, Fmoc protected Val-ala-OH **1 h** was electrolyzed by dissolving in DMA solution in presence of 2 eq of triethylamine and 10 eq of water to obtain **2 h** in moderate yield (Table 2, entry 2 g). Successful formation of **2 h** undoubtedly confirms the functional group tolerance and feasibility of the method.

Polyhydric alcohol like ethylene glycol was employed with the Cbz glycine to afford the carbamate with a terminal hydroxyl moiety **2i** with excellent yield (Table 2, entry 2i).

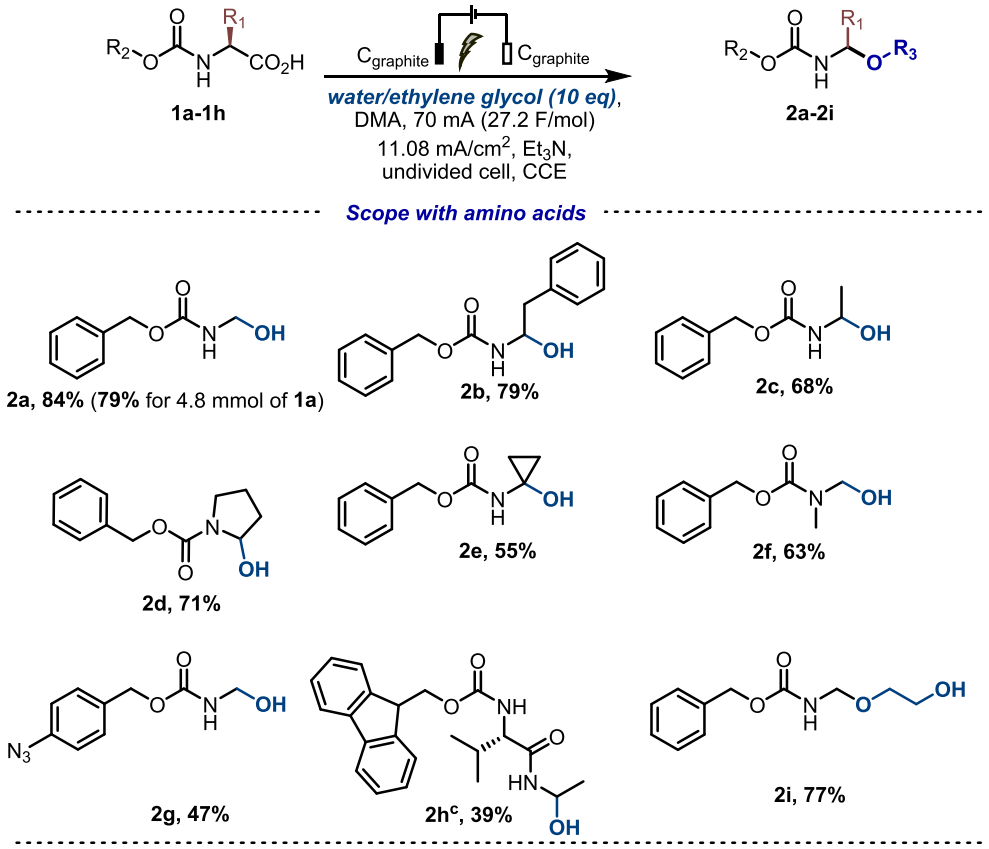
After successfully exploring the substrate scope of electrochemical decarboxylative hydroxylation, we further extended our study in field of decarboxylative nucleophilic addition. Recently, Cantillo et al. reported a solvent mediated efficient decarboxylative acetoxylation [33]. However, the acid scope of the reaction was limited to acetic acid, that too when used as solvent. We carried out thorough investigations to optimize the condition to have a general strategy using solvent like DMA and extending the method for other acids as well. (See *SI*, Page S5).

After obtaining the optimized conditions, Cbz and benzoyl protected amino acids **3a** and **3b** derived from glycine were electrolyzed to synthesize corresponding acetoxy analogues **4a** and **4b** respectively (Table 3, entry 4a-4b). This protocol was further tested on the aryl azide **3c**, and respective methyl acetate **4c** was synthesized with an excellent yield (Table 3, entry 4c). Method's versatility was further manifested when cyclopentane carboxylic acid was employed as the reacting nucleophile with the Cbz glycine to produce **4d** with a good yield (Table 3, entry 4d).

Mechanistically, the electrochemical conversion of amino acids into hemiaminals follows the classical non-Kolbe electrolysis pathway [34].

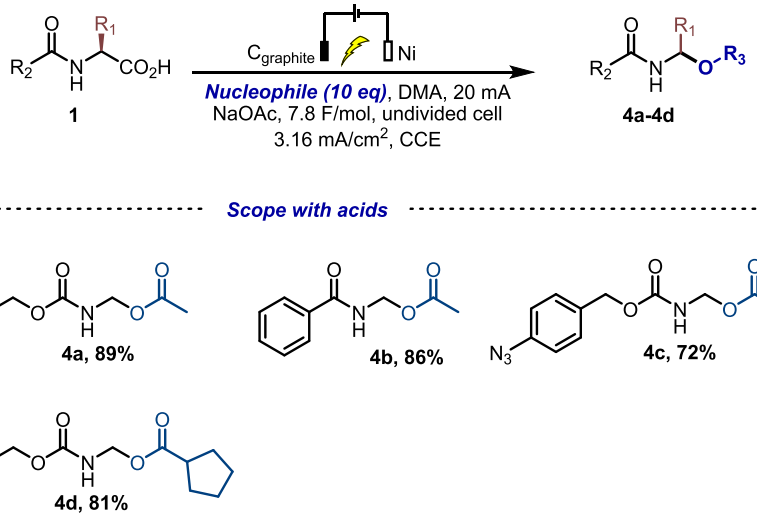
The reaction is triggered by anodic oxidation of the carboxylate species **1aa**, leading to rapid decarboxylation and generation of a resonance-stabilized iminium intermediate **2bb** [35]. The ionic nature of the process was supported by two control experiment conducted in the presence of 2 equivalents of TEMPO and 1,1-diphenylethylene, wherein the desired product was obtained in a comparable yield (Table 4). Subsequently, the electrophilic intermediate **2bb** is trapped by water O atom, with nucleophilic attack occurring at the sp^2-

Table 2
Electrochemical decarboxylative hydroxylation with amino acids [a].



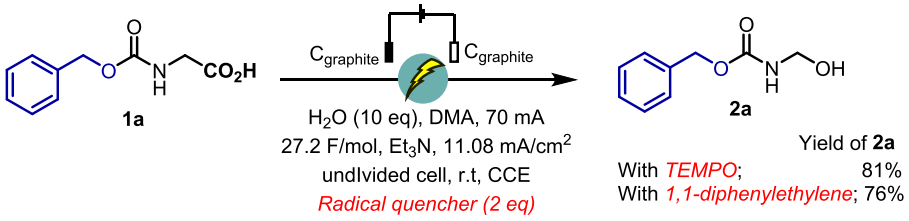
[a] **Optimized condition: 1a-1 h** in DMA, 10 eq of water/ethylene glycol, Et_3N (0.05 eq), Undivided cell, constant current electrolysis 70 mA (27.2 F/mol). $C_{graphite}(+)|C_{graphite}(-)$ as electrodes. [b] Condition for scale up: **1a** in DMA, 10 eq of water, Et_3N (0.05 eq), Undivided cell, constant current electrolysis 140 mA (10.92 F/mol), [c] with 0.02 eq of Et_3N.

Table 3
Electrochemical decarboxylative addition with other nucleophiles [c].



[c] **Optimized condition: 1** in DMA, 10 eq of acid, NaOAc (2 eq), Undivided cell, constant current electrolysis 20 mA (7.8 F/mol). $C_{graphite}(+)|$Ni plate $(-)$ as electrodes.

Table 4
Control experiments [a].



[a] **Standard condition: 1a** in DMA, 10 eq of water, Et_3N (0.05 eq), radical quencher (2 eq), Undivided cell, constant current electrolysis 70 mA for 10 h. $C_{graphite}(+)|C_{graphite}(-)$ as electrodes.

hybridized carbon of the iminium ion. This step is followed by deprotonation, ultimately affording the hemiaminal product **2** (Scheme 5).

Conclusion

We have developed a novel and facile electrochemical strategy for the synthesis of hemiaminals and related analogues, which are key intermediates in pro-drug and ADC linker development. This electrochemical decarboxylative hydroxylation protocol offers a practical and efficient alternative to conventional synthetic methods, displaying broad substrate compatibility, including sterically demanding secondary and tertiary substrates. The optimized reaction conditions minimize excess nucleophile usage, rendering the process atom-economical and operationally simple. Furthermore, the methodology has been successfully extended to diverse nucleophiles such as carboxylic acids and lactams, highlighting its versatility for linker conjugation in pro-drug and ADC applications.

CRediT authorship contribution statement

Manas Bandyopadhyay: Writing – original draft, Validation, Investigation. **Suvadeep Nath:** Writing – review & editing, Supervision. **Chandrasekhar Korapala:** Supervision. **Subhendu K. Mohanty:** Writing – review & editing.

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.2026.156233.

Data availability

Data are given in the Supporting Information file published along with the supporting information file.

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