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Review

One Electrifying Decade of Organic Syntheses: A Literature Survey Based on Different Electrode Combinations

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Abstract

Synthetic organic chemistry offers an endless scope in various domains of science, and electro-organic synthesis has grown as a green alternative to synthetic organic chemistry, obviating the necessity for harmful oxidants and reductants. Electro-organic reactions have attracted considerable attention globally in both academia and industry. This study analyzes the electrochemical organic synthesis towards the synthesis of valuable organic compounds. Electro-organic methodologies encompass C—P/C—O/C—N/N—N bond formation, C—H/N—H functionalization, C—N/C—S cross—coupling, ring expansion, oxidation dearomatization/annulation, C—H thiolation, dehydrogenative C(sp^2)—H amination, C(sp^3)—H/C(sp^2)—H functionalization, sulfur insertion, Henry reaction, and Buchwald–Hartwig amination. This review emphasizes the synthesis of diversified organic compounds that possess remarkable biological features and show considerable potential in medicinal chemistry. The current review summarizes the reports from the recent decade that represent electro-organic synthesis for the development of diversified organic compounds.

Keywords: electro-organic synthesis; green methodology; diverse electrodes; diverse organic compounds

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1. Introduction

Researchers frequently utilize thermal, photochemical, microwave, and ultrasonic activation modes in organic synthesis [1–3]. The term "electrification" in organic synthesis is emerging as a stable designation for a new era characterized by increasing opportunities and advantages [4] (Figure 1). An electrochemical method for synthesizing or-

ganic molecules for value-added goods is re-emerging as a novel synthetic tool and serves as an environmentally friendly alternative to existing chemical procedures. Electrochemical oxidation/reduction does not depend on stoichiometric oxidizing or reducing agents (such as OsO_4, $Pb(OAc)_4$, or metal and metal hydride sources), which have significant economic and environmental ramifications. From an ecological footprint perspective, replacing stoichiometric quantities of oxidants and reductants with electric current is an inherently more sustainable approach than conventional approaches. In organic chemistry, electrochemical synthesis has advanced rapidly in recent years, offsetting the previously gradual progression [5]. Electrosynthesis is regarded as one of the most cost-effective and environmentally sustainable methods, as it facilitates oxidations and/or reductions without requiring further chemical reagents [6]. The reduction in the number of processes is favorable for the environmental and economic evaluation of manufacturing and leads to energy savings [7]. Current trends in synthetic organic electrocatalysis involve the integration of electrosynthesis with established and novel process techniques, e.g., flow chemistry for process scaling, to improve efficiency and sustainability, as well as the use of new synthetic feedstocks [8,9].

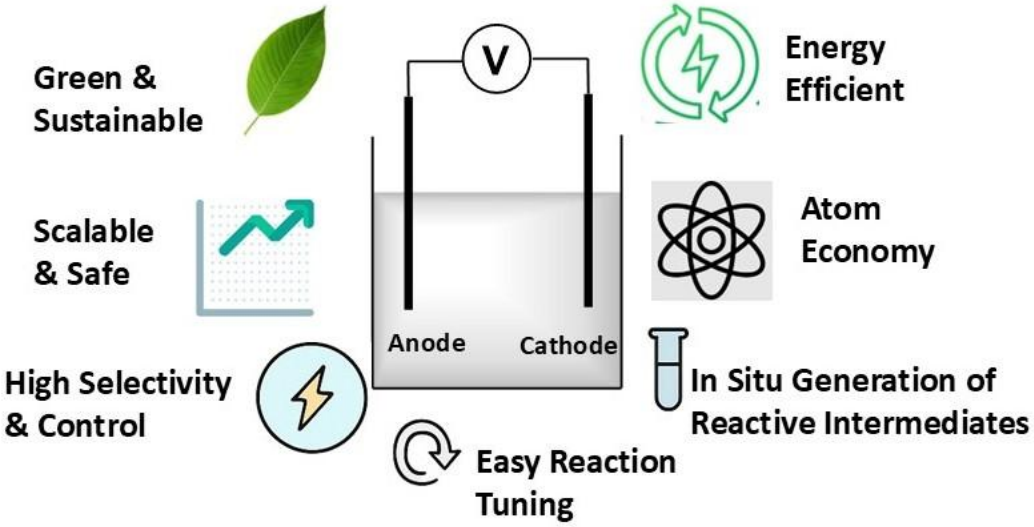


Figure 1. Features of electro-organic synthesis.

Organic heterocycles constitute a highly relevant category of molecules in contemporary chemistry, owing to their remarkable structural diversity and extensive functional potential [10]. Their significance stems from the inclusion of heteroatoms like nitrogen, oxygen, or sulfur in the cyclic structure, which greatly affects their electronic distribution, reactivity, and molecular interactions. Heterocycles are essential in medicines, with over 70% of FDA-approved treatments, including anticancer, antiviral, antibacterial, and CNS-active compounds, using heterocyclic scaffolds such as pyridine, indole, quinoline, pyrazole, and thiazole [11]. Their capacity for $\pi-\pi$ stacking, metal coordination, hydrogen bonding, and biological recognition facilitates accurate molecular targeting and enhanced pharmacokinetics. In addition to medicinal chemistry, heterocycles are essential in modern material science, functioning as organic semiconductors, OLED components, photovoltaic materials, and energy—storage interfaces due to their adjustable optoelectronic and redox characteristics [12]. In catalysis, heterocyclic ligands like *N*—heterocyclic carbenes (NHCs) improve catalytic durability and reactivity, facilitating effective C—H activation, cross—coupling, and asymmetric synthesis [13]. Their applicability includes agrochemicals, sensing technologies, supramolecular structures, and polymer science [14–17]. The advancement of synthetic techniques, such as metal catalysis, photoredox procedures,

and C-—H functionalization, underscores the ongoing pursuit of designing innovative heterocycles that exhibit improved functionality, selectivity, and sustainability, representing a significant challenge in chemical and transdisciplinary research.

2. A Brief History of Electro-Organic Synthesis

The use of electricity to produce a chemical reaction has been studied since the 1830s. In the experiment, Faraday used an electric current in an acetate solution for the synthesis of hydrocarbons [18]. The Kolbe electrolysis, allowing the formation of C—C bond, was studied in detail. In Kolbe electrolysis, the oxidation of the carboxylate anion occurs at the anode [19]. Then, the carbon-centered free radicals are formed, which leads to the CO_2 release and C—C bond formation by homo-coupling. Still, this reaction represents a diverse strategy for C—C bond formation [20]. Electrochemistry has great industrial importance, as shown by the Hall–Héroult process, and the synthesis of ethylene glycol, and adiponitrile [21]. The next step is to search for a redox mediator that, after electron transfer at the anode, forms an electrochemically generated species capable of catalyzing the formation of the desired product [22]. The most common redox mediators introduced in the last four decades are nitrosyl radicals [23] and triarylamines [24]. In addition, the presence of these functions enables a more selective electron transfer and decreases the probability of over-reduction or over-oxidation of the target product. Other notable advances in the 21st century include Waldvogel's selective biaryl cross-coupling [25] and Yoshida's cation-pool strategy for C—H functionalization of aromatic compounds [26].

3. Scope of the Review

In the past few years (2021–2025), several review articles have been published on the utilization of electro-organic synthesis towards the development of organic heterocycles (Figure 2a). In this direction, in 2021 Ackermann et al. [27] reviewed the application of electrochemistry in the synthesis of molecules. After that, Batanero and coworkers [28] highlighted the green feature and fundamentals of electro-organic chemistry. In the same year, Wirth et al. [29] reviewed the development of organosulfur compounds through electro-organic methods. In 2022, several review reports were published to focus on transition-metal-catalyzed electro-organic synthesis [30], electro—organic conversions (Heck reaction) [31], the combination of transition—metal and electrochemistry [32], electrochemical reactions for *N*—heterocycles [33], the basis of electrochemistry [34], green alternatives for heterocycles [35], electrochemical methods for nitrogen-enriched materials [36], tutorials of electro-organic reactions [37] and green alternatives for organic compounds [38]. In the next year (2023), researchers reviewed unexplored areas, such as electro-organic synthesis with heterogeneous catalysis [39], multicomponent electro-organic methods [40], and electrochemical syntheses with transition metal catalysis [41]. Recently (in 2024), Hosseini's group [42] published a report on the application of a modified working electrode that also worked well for the synthesis of organic compounds. In the same year, Guijarro and coworkers [43] reviewed the role of fluorinated solvents in electro-organic reactions. Salinas's research team [44] revealed a previously unknown aspect of wireless electro-organic synthesis in the construction of organic heterocycles. Moreover, Röckl et al. [45] disclosed the future perspectives of electrochemical methods in the drug-discovery program. In the same year, Nagarajan et al. [46] reviewed the synthesis of heterocyclic compounds via electro-organic synthesis.

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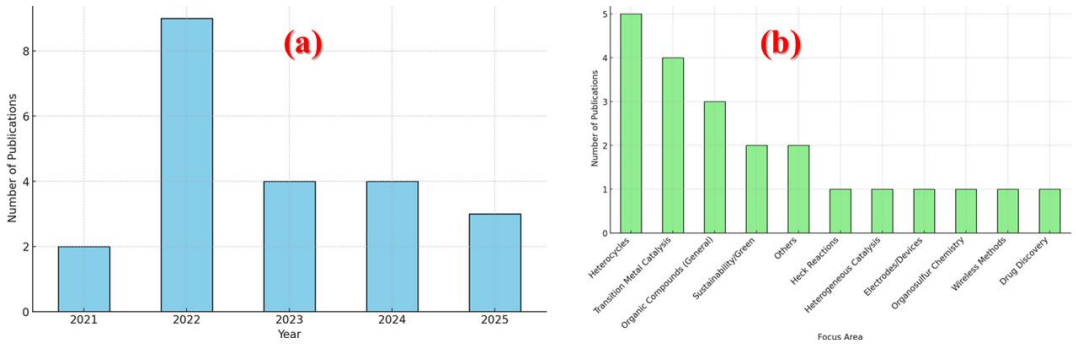


Figure 2. (**a**) Number of published reports on electro-organic synthesis (2021–2025); (**b**) focused area of published reports (2021–2025).

Although several reviews have been published, researchers have not yet explored several potentially interesting areas (Figure 2b). Reviews on specific aspects of this technology have been reported, e.g., general overviews of the basics of green electrosynthesis, applications in transition-metal-catalyzed reactions, synthesis of specific classes of compounds like N-heterocycles, or investigation of specific methodologies such as wireless methods or heterogeneous catalysts.

But despite this wealth of information, there is a critical gap in the literature. So far, no review has been available that classifies and discusses the wide field of modern electro-organic synthesis according to the electrode combination employed. The choice of anode and cathode materials is a key parameter that determines reaction feasibility, selectivity and efficiency but is often discussed in a fragmented way.

This review fills that particular gap, offering a novel and systematic analysis of the electrochemical literature of the last decade (2015–2025) explicitly organized by the type of electrode pair employed (i.e., C—C, Pt—Pt, C—Pt, C—Ni, etc.). This structure of analysis provides readers with a unique perspective, which highlights:

- ✓ The synthesis capabilities and limitations of specific electrode materials;
- ✓ Novel trends in the development and implementation of innovative cost-effective electrode systems;
- ✓ The direct relationship between electrode arrangement and the type of chemical conversions that can be achieved.

Such work goes beyond summarizing what has been synthesized to provide a fundamental understanding of how the choice of electrode enables these transformations. The present singular electrode-focused approach is designed as a practical guide for synthetic chemists to more efficiently select and design electrochemical systems for their own research purposes.

The aim of this review report is to encourage researchers to explore and adopt electro-organic synthesis, a technique with considerable potential to enhance the general synthetic organic toolbox. The current review is categorized on the basis of cell construction, with C—C, Pt—Pt, C—Pt, C—Ni, C—Fe, C—hybrid material, RVC—Pt, Mg—Pt and miscellaneous-based electrodes used for the organization of the review.

4. Review Methodology

This literature review was conducted as a systematic survey and analysis of the progress in electro-organic synthesis of various organic compounds with special emphasis on the electrode combinations used. A structured search strategy was followed to ensure a comprehensive and relevant literature collection. The primary scientific databases searched for this review were Scopus and Web of Science. The literature search was limited to reports published in the past decade, from January 2015 to 2025. This time frame was selected

to include the most recent and "electrifying" developments in the field, as per the title and aim of the review. A combination of keywords and their variants was used to identify relevant articles. The core search terms included electro-organic synthesis, electrosynthesis, electrochemical synthesis, heterocycle synthesis and organic compounds. This review is mainly based on original research articles and communications describing new synthetic methodologies of organic compounds *via* electrochemical techniques. Reports were excluded when they were primarily dedicated to purely theoretical calculations, materials science (where electrodes were developed but not employed in the synthesis), analytical methods or polymer synthesis, unless they involved a significant and novel organic transformation. The systematic method guarantees that the review presents a focused and relevant analysis of the state-of-the-art in preparative electro-organic chemistry organized according to the novel framework of electrode combinations.

5. Basic Principle

Electro-organic synthesis relies on the notion of utilizing electrical energy to facilitate organic chemical reactions via electron transfer at electrodes. In contrast to traditional procedures employing chemical oxidizing or reducing agents, electro-organic synthesis uses electricity as a clean reagent, rendering it an environmentally sustainable approach in contemporary organic chemistry. Oxidations happen at the anode, while reductions that involve transferring a single electron to a substrate occur at the cathode. Typically, chemically and electrochemically inert solvents, in conjunction with supportive electrolytes (such as salts, acids, or bases), enhance optimal electrical conductivity. The direction of the electron flow from the electrode can be inverted in any orientation using a power supply. Therefore, electric current is a crucial instrument in the synthesis of organic processes. The incorporation of electrons into the LUMO of a molecule induces cathodic reduction, whereas the removal of electrons from the HOMO of that molecule causes anodic oxidation [47]. The effectiveness of the electrochemical process is determined by parameters such as applied charge (reagent equivalents), current density, and temperature.

6. Scope of Various Electrodes

The bar chart provides a graphic representation of a comparative overview of various electrode types based on the number of studies that have been reported (Figure 3). Among all of the electrodes, C—Pt electrodes have the highest number of reports, demonstrating that they have superior electrocatalytic performance and may be used practically everywhere. Pt—Pt electrodes also show extensive usage, owing to the dependability of platinum-based systems, despite worries regarding cost. Because of its large surface area and improved mass transfer capabilities, RVC—Pt holds a prominent place in the engineering industry. According to this, C—C, C—Fe, and C—hybrid materials have a moderate utilization rate, which indicates that there is a growing interest in alternatives that are both cost-effective and environmentally friendly. The very low number of studies on C—Ni electrodes indicates either a lack of research or poor efficiency. This could be because of compatibility or stability problems, but Mg—Pt exhibits the least utilization. In general, the trend demonstrates a significant reliance on combinations based on platinum, but carbon-supported and hybrid systems are increasingly emerging as attractive and cost-effective alternatives in the design of electrodes.

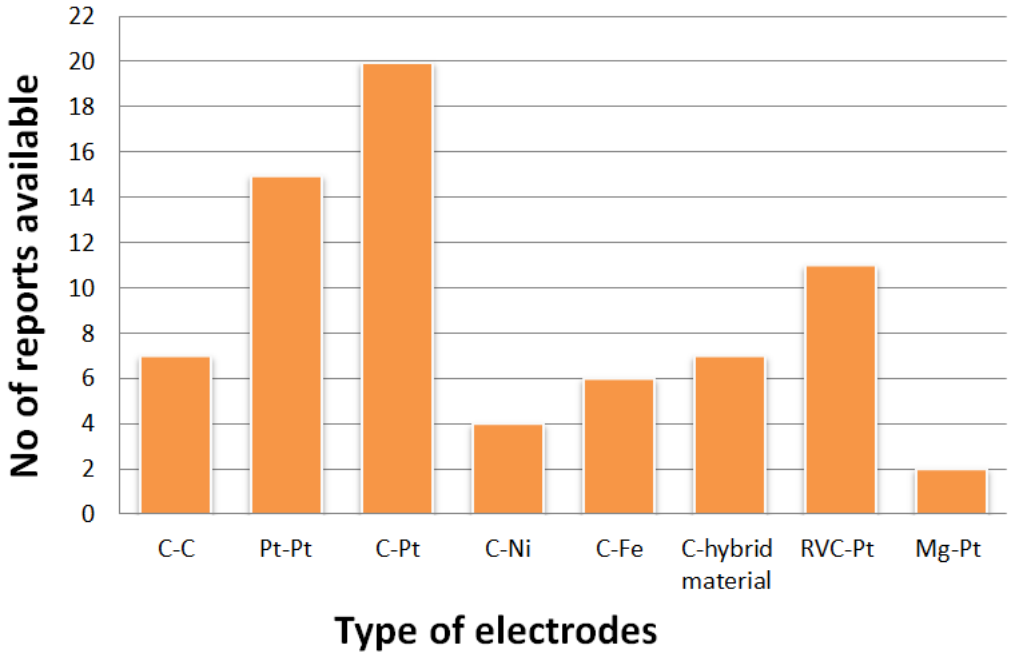


Figure 3. Scope of various electrodes.

7. Electro-Organic Synthesis of Organic Compounds by Using Various Electrodes Types

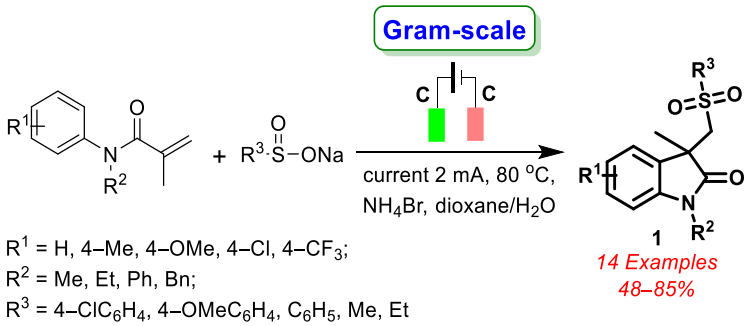
Here, we have summarized various methodologies for the construction of diversified organic compounds using different combinations of electrodes. This section is subdivided into 7.1. C—C-based electrode, 7.2. Pt—Pt-based electrode, 7.3. C—Pt-based electrode, 7.4. C—Ni-based electrode, 7.5. C—Fe-based electrode, 7.6. C—hybrid material-based electrode, 7.7. RVC—Pt-based electrode, 7.8. Mg—Pt-based electrode, and 7.9. Miscellaneous. A detailed discussion is presented here.

7.1. C—C-Based Electrode

Metal-free and sustainable electro-organic production relies on C-—C electrode systems, in particular those based on inexpensive graphite. Several research groups have widely explored C—C electrode systems owing to their low-cost, metal-free nature and symmetric electrode system. Moreover, the touch of green chemistry is proved by the use of mild conditions, high atom economy and the application of novel recyclable deep eutectic solvents (DES) makes C—C electrodes to be highly versatile and adoptable by the scientific community. In this section, we review the different methodologies for the construction of various organic compounds using C—C electrodes. This system can afford a wide variety of oxindoles, α-amino ketones, pyrrolidines, carbazoles and other N/S—containing organic compounds. Redox-mediated C—H functionalization, cycloadditions, and multicomponent reactions have been successfully catalyzed by C—C electrode systems.

In 2016, Zeng's research team [48] reported an electrolysis of a sodium sulfate and acrylamide mixture to afford oxindoles. The process introduced a NH_4Br (redox catalyst) in the presence of a C-C undivided cell. Notably, the technique has been shown to be effective for various substrates. Delightedly, both the electron-donating groups (CH_3^-/CH_3O^-) as well as the electron-withdrawing groups (Cl/CF_3^-) produced the desired products **1** in satisfactory yields. Surprisingly, both the electron-donating groups CH_3- and CH_3O- as well as the electron-withdrawing groups Cl- and CF_3- produced the expected products with yields of 70%, 70%, 54%, and 48%, respectively. Significantly,

when the potent electron-withdrawing group was the NO_2—group, the resultant adduct was not produced. Nevertheless, the presence of a chloro substituent at the ortho position of the aryl-acrylamide impeded the formation of the target compound under conventional conditions due to steric hindrance; instead, *N*—(2—chlorophenyl)—*N*—methyl—2—(tosylmethyl)acrylamide was obtained with a yield of 50%. In the instances of sodium methanesulfinate and sodium ethanesulfinate, the respective adducts were obtained with yields of 85% and 77%. The sodium mesitylenesulfinate failed to yield the intended product possibly owing to steric hindrance. Moreover, gram-scale studies demonstrate its industrial practicality (Scheme 1).



Scheme 1. Difunctionalization of acrylamide via electrochemical methods.

The reaction initiates with the anodic oxidation of bromide ions to generate molecular bromine, which subsequently reacts with sodium sulfinate to yield sulfonyl bromine (**I**). Next, the sulfonyl bromine (**I**) breaks apart in a way that creates a bromine radical and an oxygen-centered radical (**II**). Thereafter, the oxygen-containing radical (**II**) transitions to the more stable sulfonyl radical (**III**), while the bromine radical transfers to hydrobromide by abstracting a hydrogen atom. Further incorporation of the sulfonyl radical (**III**) into acrylamide produces radical (**IV**). The intramolecular cyclization of intermediate (**IV**) yields the radical intermediate (**V**), which is followed by oxidation to produce the final product (Figure 4).

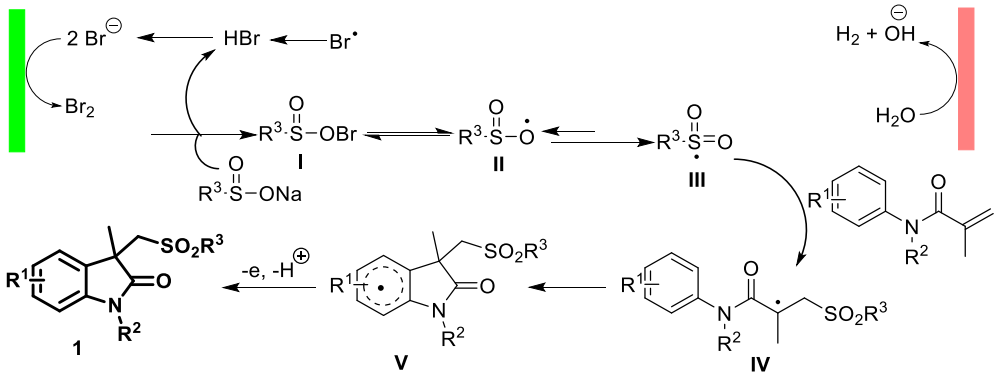
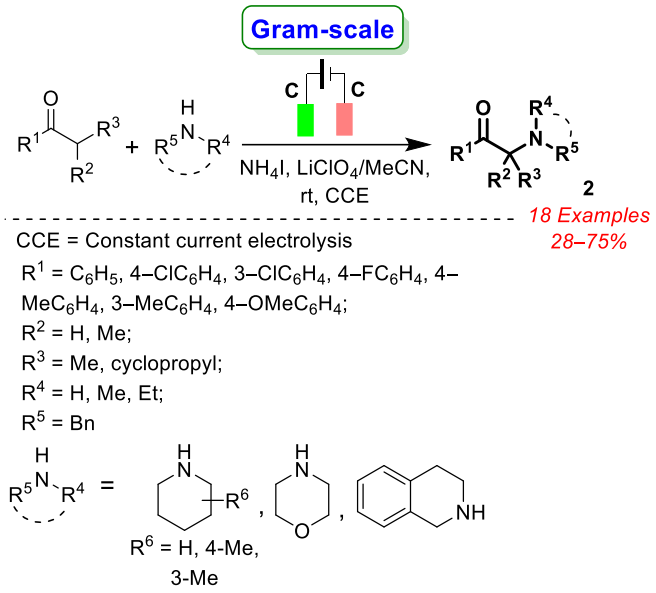


Figure 4. Proposed mechanism.

In 2016, Zeng et al. [49] also developed a C—C electrode-based electrolytic method towards the synthesis of *α*-amino ketones **2**. The reaction was done using a method that combined secondary amines and ketones through oxidation. In this work, the electrochemistry is carried out under a constant current of an undivided cell with NH_4I serving as a redox catalyst and low-cost graphite plates serving as electrodes. The final products **2** were afforded in 28–75% yields at room temperature. Nonetheless, 2—methylpiperidine proved to be an unsuitable coupling partner due to steric hindrance, and its reaction with propiophenone failed to yield the similar adduct. However, electron-poor and elec-

tron—rich propiophenones reacted smoothly with piperidine. The direct amination process demonstrated optimal efficiency when an electron-withdrawing substituent was attached to the benzene ring of propiophenone (Scheme 2).



Scheme 2. Electrochemical synthesis of α-amino ketones.

In the mechanism, the process initiates with the anodic oxidation of iodide, which delivers molecular iodine that further reacts with ketone (**I**) to produce α—iodo ketone (**III**). Next, it rapidly engages in a nucleophilic substitution reaction with an amine to form an α—amination product (**2**). Concurrently, the in situ produced HI is diminished to release H_2 on the cathode's surface (Figure 5).

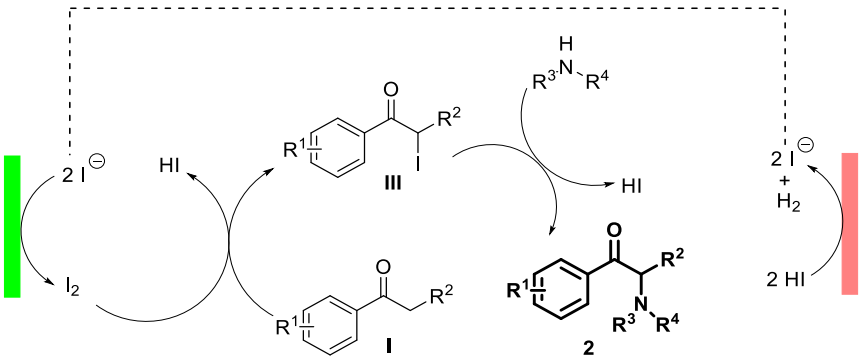
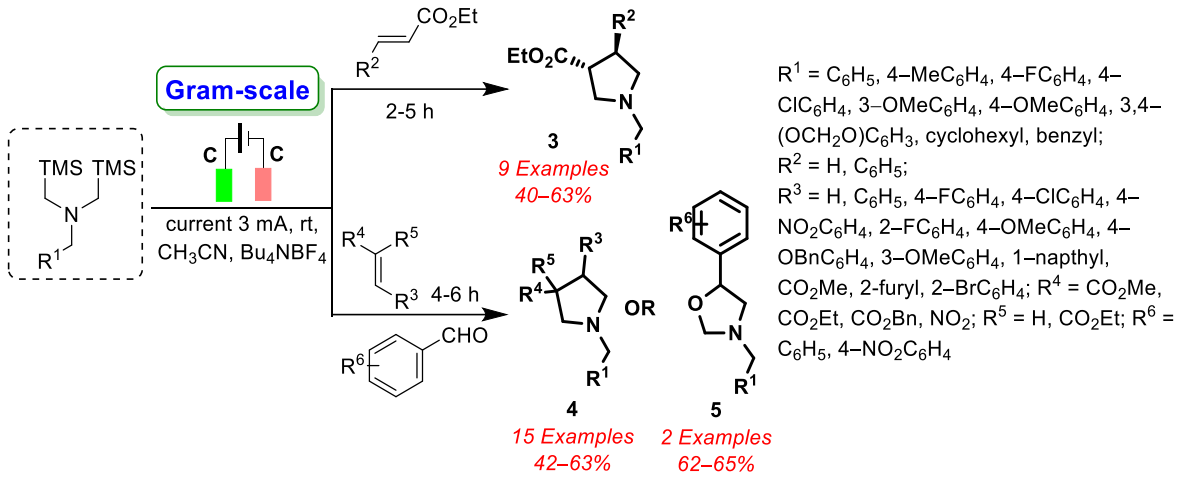


Figure 5. Proposed reaction mechanism.

Later on, Banerjee's research team [50] explored an electrolytic technique towards the formation of 3-/3,4—disubstituted pyrrolidines **3-4**. Specifically, an effective and ecologically friendly electrochemical method for constructing a nonstabilized azomethine was performed under metal-free and external oxidant-free conditions (Scheme 3). Next, the 1,3-dipole proceeds through a [3+2] cycloaddition process with olefins. The methodology also works well with benzaldehyde derivatives and provides access to oxazolidine with moderate yields. The synthetic approach elaborated anodic oxidation followed by double desilylation to produce a 1,3-dipole, which is subsequently trapped with different alkenes as dipolarophiles to form the cycloadduct.



Scheme 3. Formation of substituted pyrrolidines by using C-C electrode.

Initially, azomethine feels anodic oxidation through single electron transfer (SET) to generate radical cation species (**I**). This radical cation species loses a silane group, and then it undergoes another oxidation that creates an iminium cation (**II**). The further desilylation of this iminium cation (**II**) led to the synthesis of azomethine ylide (**III**), followed by the anticipated [3+2] cycloaddition with (**IV**), yielding the desired product (Figure 6).

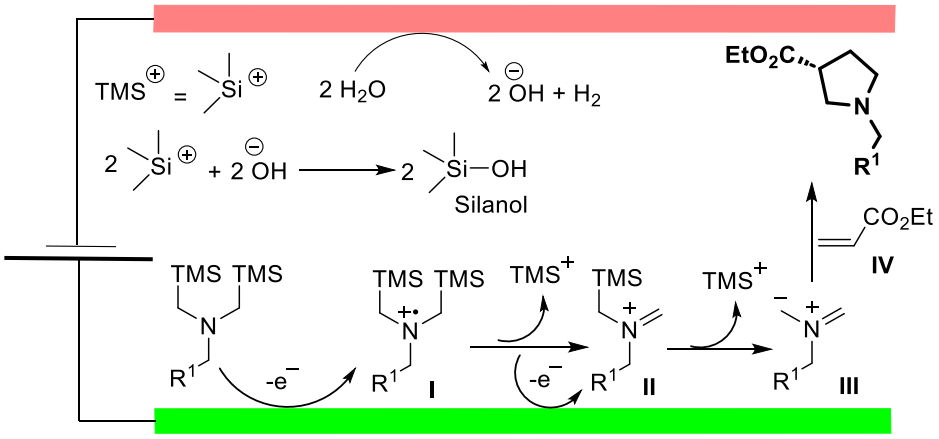
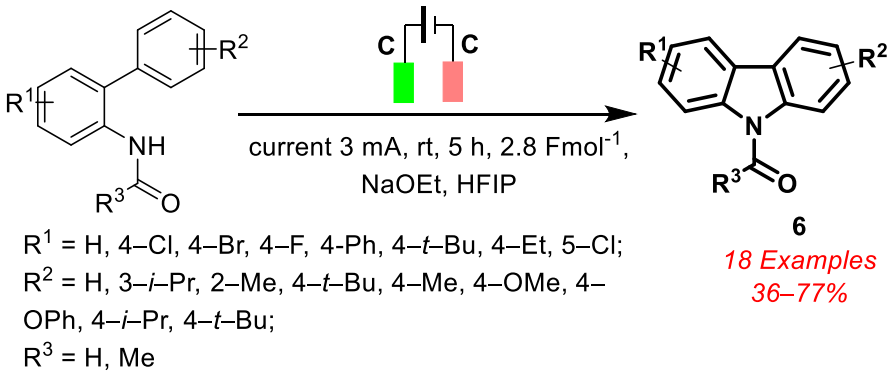


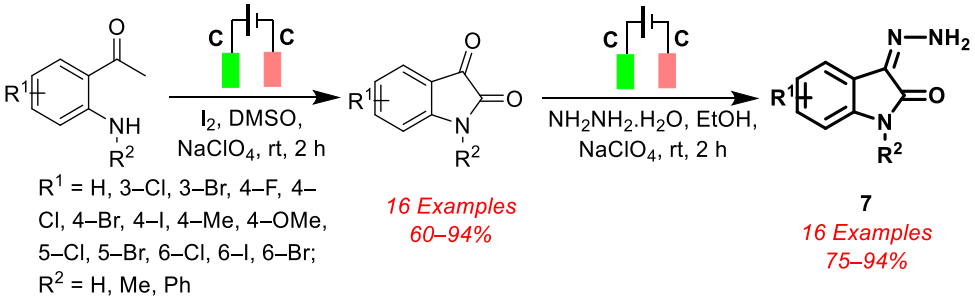
Figure 6. Plausible mechanism.

In 2021, Antonchick et al. [51] pioneered a novel transition-metal-free direct electrolytic protocol for the synthesis of carbazole derivatives **6** using graphite electrodes. The method devised is devoid of any need for organoiodine catalysts and exhibits simplicity in its execution. Furthermore, the authors illustrated the feasibility of the in situ generation of a supporting electrolyte for electrolysis by employing widely available inorganic bases in acidic solvents, such as hexafluoroisopropanol. A new and effective method for C—H amination, both within and between molecules, has been successfully implemented using a straightforward reaction setup (Scheme 4).



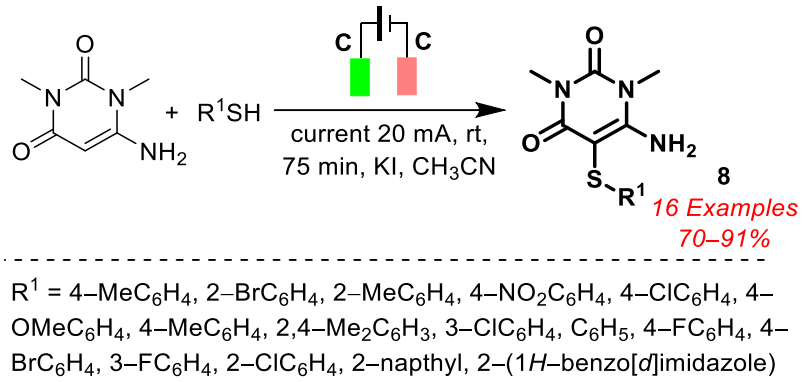
Scheme 4. Substrate scope of intramolecular amination.

In 2023, the research group of Sagar [52] reported an environmentally friendly method for creating isatin derivatives **7** via a two—step process. It was stated here that isatin derivatives **7** were synthesized by a free radical mechanism in a shorter reaction time using an electro-organic technique aided by iodine in DMSO solvent through C—N cross-coupling and C(sp^2)—H/C(sp^3)—H functionalization. Thereafter, isatin derivatives **7** were subjected to treatment with hydrazine hydrate in an undivided cell, including graphite carbon functioning as both an anode and a cathode at room temperature, with a constant voltage applied under ambient conditions for 30 min. This treatment resulted in the formation of isatin-based hydrazone derivatives **7**. The isatins and corresponding hydrazones were constructed in good to high yields by these transformations, which also tolerated the EDG and EWG substituents on the core molecules (Scheme 5).



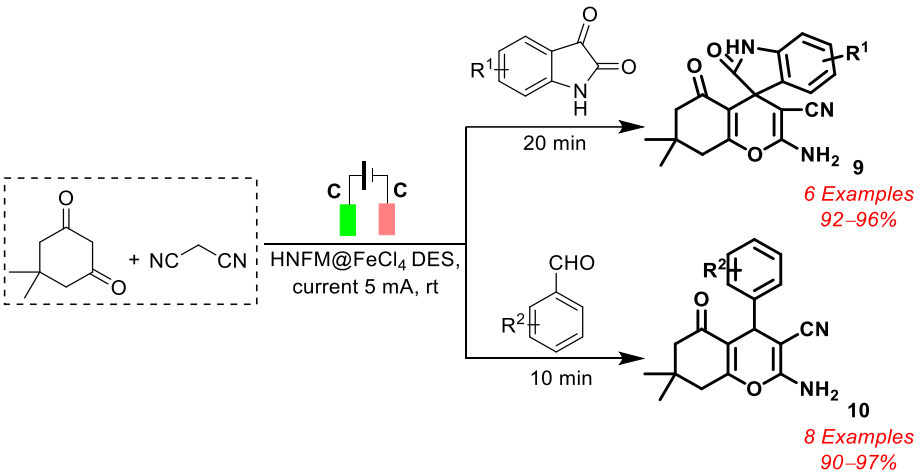
Scheme 5. Electro-organic synthesis of hydrazones.

Next (in 2023), Joshi et al. [53] synthesized thiol-tethered pyrimidine derivatives **8** as anticancer agents through a sulfur insertion approach by the application of C—C electrode-based electro-organic synthesis. The treatment of pyrimidine and thiols delivered a diversified library of C—5 sulfenylated amino uracils **8** in the presence of potassium iodide and acetonitrile. The final products **8** were achieved in high yields (70–91%) within only 75 min. Interestingly, substituting bromo and chloro groups at the second position led to a reduced yield due to the ortho effect. 2—Mercapto benzimidazole and naphthalene thiol were likewise effectively introduced under the established reaction conditions, resulting in the required products with yields of 75% and 91%, respectively. Moreover, the current methodology has additional advantages, such as broad substrate scope (16 examples), high atom economy, high yields (70–91%), and green reaction conditions (Scheme 6).



Scheme 6. Electro-organic synthesis of C5—sulfenylated amino uracil derivatives.

More recently (in 2025), Afzal's research team [54] introduced a novel recyclable DES instead of organic solvents in C—C electrode-based electrolytic system. This green methodology successfully synthesized spirooxindoles **9** (92–96%) and 4*H*—pyran derivatives **10** (90–97%) showcasing the effectiveness of the DES in facilitating rapid reactions under mild conditions. Usually, the reaction was conducted at room temperature under a 5 mA current using graphite rods of low-cost and readily available anodes and cathodes, completing the process in just 10–20 min only. The new 4—formylmorpholin—4—ium iron (**III**) chloride (HNFM@$FeCl_4$) DES not only makes electrochemical processes more efficient, but it also solves problems with traditional solvents, catalysts, and electrolytes that affect the environment (Scheme 7).



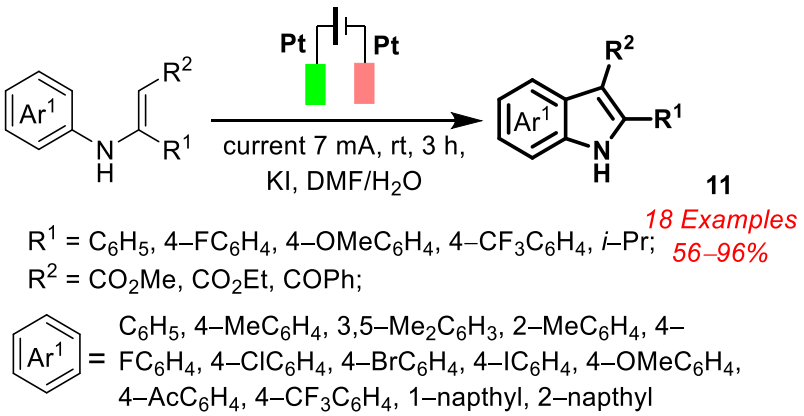
Scheme 7. Electro-organic formation of spirooxindole and 4*H*—pyrans.

7.2. Pt-Pt-Based Electrode

Here, we report Pt—Pt electrode systems as a robust and highly effective platform for a broad range of complex organic transformations. Pt—Pt electrodes are widely used by researchers due to their excellent chemical stability and superior electrocatalytic kinetics (especially for the hydrogen evolution reaction). This allows them to promote very challenging transformations such as intramolecular C—H/C—H cross-couplings, cyclizations and annulations without the use of harsh chemical oxidants and to deliver high yields and excellent functional group tolerance under mild conditions. This section presents the synthesis of a variety of compounds, including indoles, oxime derivatives, polycyclic aromatic hydrocarbons (PAHs), quinolines, and various S/P-containing organic compounds. The high efficiency, excellent yields and broad functional group tol-

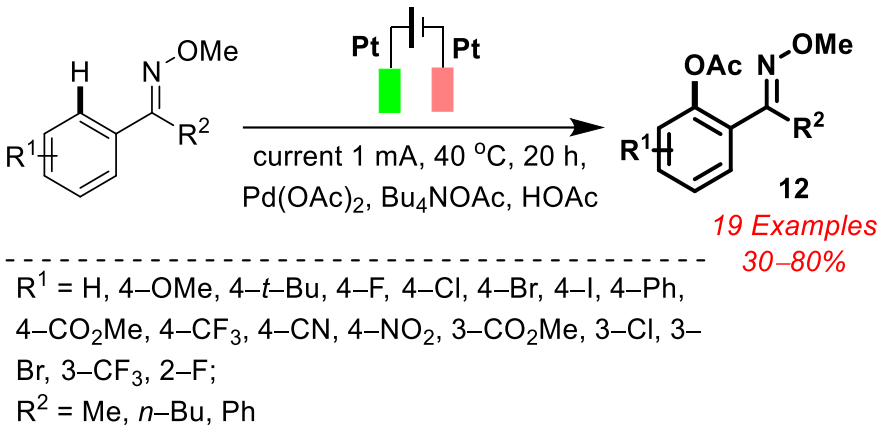
erance of these methods are discussed, and they often employ metal co-catalysts or halide mediators to further improve their synthetic utility.

In this direction, in 2017, the research group of Lei [55] established, for the first time, an electrocatalytic intramolecular annulation of *N*—aryl enamines by using a Pt—Pt cell. It provides a straightforward and effective method for the synthesis of indoles **11** within an undivided cell. Favorable to exceptional yields (56–96%) were achieved in the absence of oxidants and transition metals. Besides *N*—phenyl enamines, *N*—naphthyl and *N*—pyridyl enamines were also suitable for this transformation, affording the corresponding products **11** in good yields. *N*-Phenyl enamines exhibited remarkable reactivity in the production of indoles. Methyl groups located at the para or ortho positions did not influence the reaction efficiency, whereas the ortho-methyl substituent resulted in a marginal reduction in reaction efficiency. Halide substituents (F/Cl/Br/I) were all effectively accommodated under the electrochemical conditions. Both robust electron-rich and electron-poor *N*—phenyl enamines successfully yielded the corresponding indoles. In addition to *N*—phenyl enamines, *N*—pyridyl enamines and *N*—naphthyl enamines were also effective in this reaction, yielding the required indole in favorable yields. *N*—Phenyl enamines with either electron-donating or electron-withdrawing groups on the C-2 aryl moiety exhibited favorable reactivity for the production of indoles. Altering the C—2 substituent from a phenyl group to an isopropyl group resulted in a reduced reaction yield. It is interesting to note that KI not only works as an electrolyte, but it also takes part in the redox processes of the oxidative annulation (Scheme 8).



Scheme 8. Intramolecular dehydrogenative C—H/C—H cross-coupling reaction.

In 2017, Zhang et al. [56] targeted an efficient Pt—Pt electrode-based electrochemical reaction for the palladium-catalyzed C(sp^2)—*H* bond acetoxylation. The authors presented an anodic oxidation strategy to accomplish arene C(sp^2)—*H* acetoxylation by using aromatic oxime. Acetoxylated products **12** were obtained in 30–80% yields in the presence of acetic acid and Bu_4NOAc. A diverse library of derivatives (**12**) was generated to prove the synthetic versatility of the protocol. This reaction was carried out at a mild temperature without the use of hazardous oxidants, overcoming some of the disadvantages of conventional acetoxylation techniques employing stoichiometric oxidants (Scheme 9).



Scheme 9. Substrate scope of oxime derivatives.

The nitrogen atom of the oxime first binds to Pd(II) and then undergoes rate-determining C—H activation to give intermediate (**I**). Then the Pd(II) complex is oxidized at the anode to afford the Pd(IV) complex. In addition, the intermediate (**II**) then proceeds with reductive elimination to give the desired product (Figure 7).

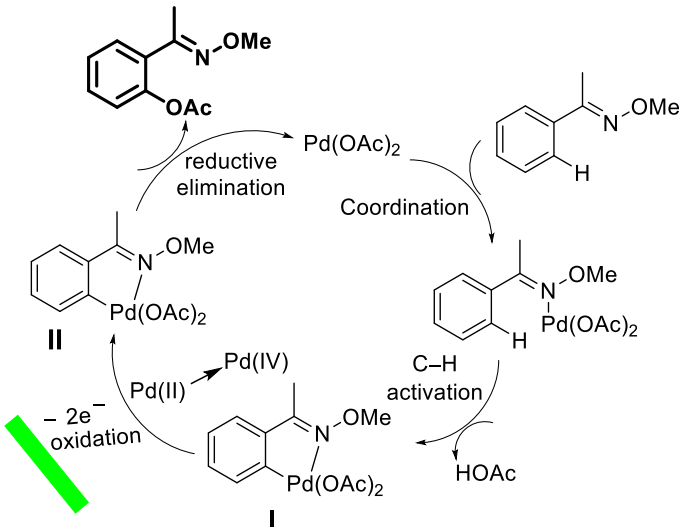
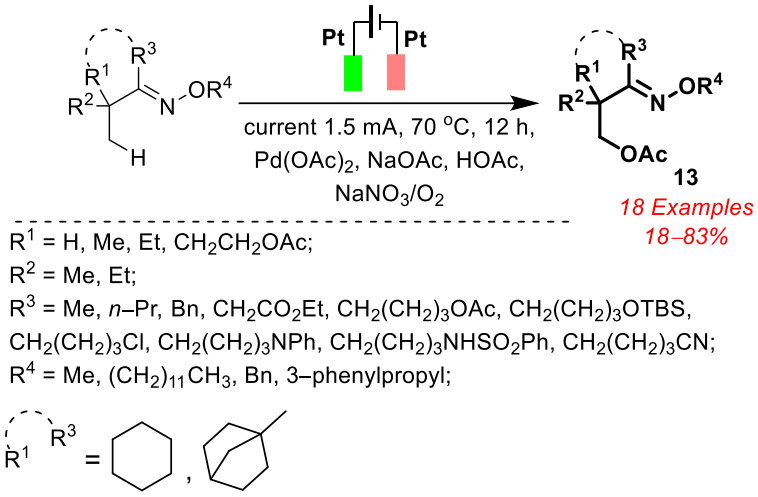


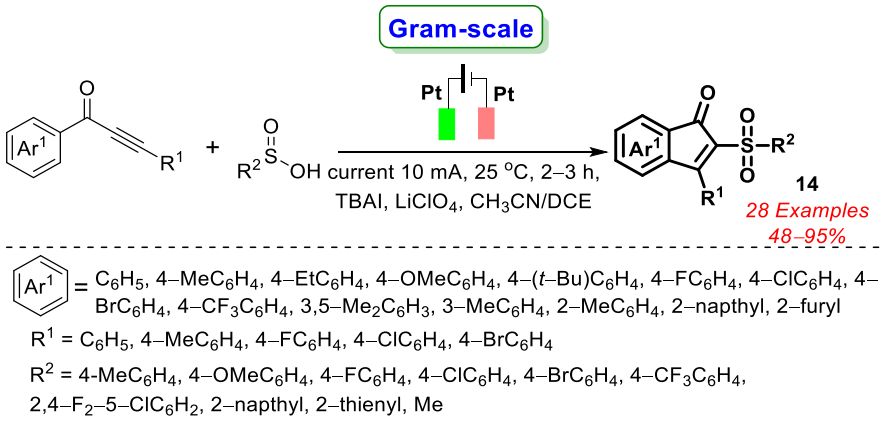
Figure 7. Tentative catalytic cycle.

After C(sp²)—*H* activation, Li's research team [57] in the same year demonstrated the Pd(II)—catalyzed C(sp³)—*H* oxygenation via anodic oxidation. The authors presented an electrochemical method that utilizes various oxyanion coupling partners to selectively generate C—O reductive elimination through the anodic oxidation of Pd(II). In addition, this strategy provides an alternative to current procedures that depend on strong chemical oxidants and is a benign natural tool for connecting distinct oxygen anions such as alkoxides acetate, and tosylate to C(sp³)—H bonds (Scheme 10).



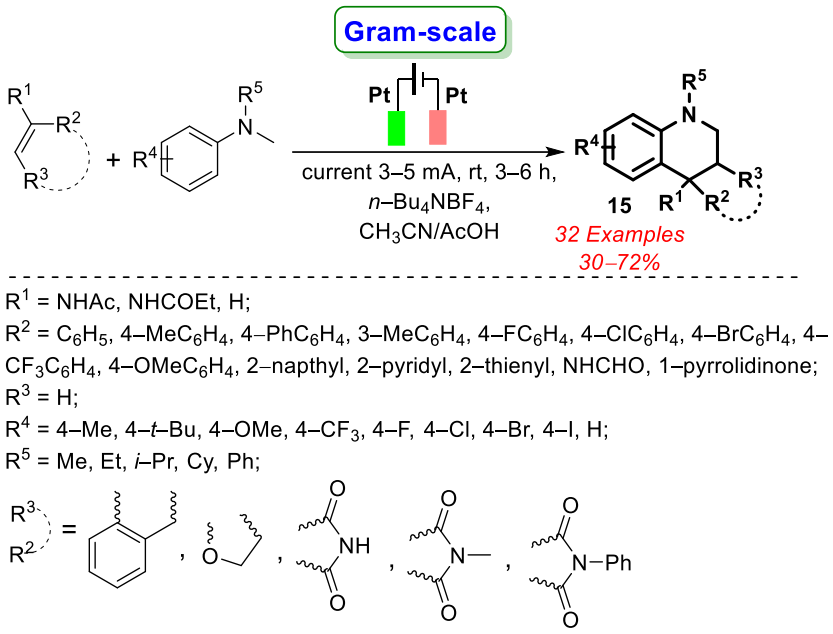
Scheme 10. Pd(II) —Catalyzed C—H functionalization.

In 2017, Xu et al. [58] disclosed a cyclization reaction introducing readily accessible urea-tethered diynes **14** (Scheme 11). Effective cyclization was completed by the use of Cp_2Fe as a mild and selective catalyst, which prevents the PAH products from being over-oxidized. The electrosynthetic method encompasses a remarkable amidyl radical cyclization strategy, enabling the formation of three rings in a singular operation. Additionally, this transformation uses readily available substrates, has a wide range of substrate compatibility and compatibility with various functional groups, and makes it simple to synthesize structurally specified polyaromatics with a variety of substitution patterns.



Scheme 11. Electro—organic arylsulfonylation of ynones with sulfinic acids.

Furthermore (in 2018), Lei's research team [59] devised a Pt—Pt-based electrolytic approach to afford six-membered heterocycles such as tetrahydroquinolines **15** (Scheme 12). Under this work, the authors performed electrochemical oxidative [4+2] annulation of tertiary anilines and alkenes by using a metal- and oxidant-free approach. Along with heterocyclic enamines (pyridyl enamine/thienyl enamine) and phenyl enamines/2-naphthyl enamine showed significant compatibility. It was observed that the reaction predominantly occurs on the *N*-methyl group. Substitution of methyl groups in *N*,*N*-dimethylaniline with ethyl/isopropyl/cyclohexyl, it demonstrates significant steric hindrance; the anticipated compounds **15** were obtained in low yields. A diversified library of tetrahydroquinoline derivatives (**15**) was produced with good yields (30–72%) using an undivided cell.



Scheme 12. Electro—organic oxidative [4+2] annulation strategy.

Initially, the reactant (**I**) is oxidized at the anode to give a radical cation which can be stabilized by AcOH. The radical cation undergoes resonance and subsequent deprotonation to form the α—amino carbon radical. The radical may subsequently participate in a radical addition reaction involving compound (**II**). The resulting radical species may undergo an intramolecular cyclization process, followed by anodic oxidation to provide the desired product (Figure 8).

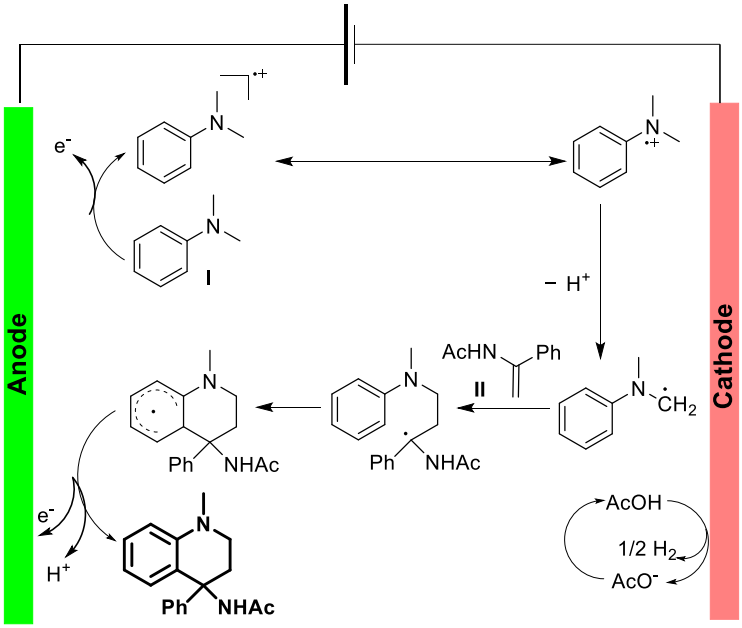
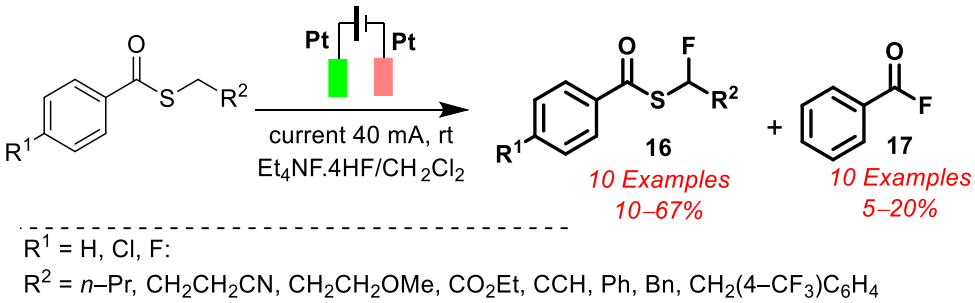


Figure 8. Tentative reaction mechanism.

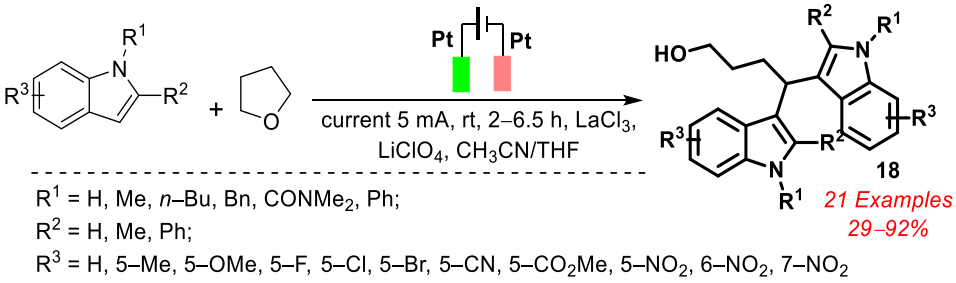
In 2018, Fuchigami et al. [60] reported a regioselective anodic fluorination of various *S*—alkyl **16** and *S*—substituted alkyl benzothioate derivatives **17** in $Et_4NF.4HF/CH_2Cl_2$ and a fluorine atom was selectively added in the α-position to the sulfur atom. Interestingly, sulfur and fluorine containing aromatic ketones **16** were obtained in 10–67% yields, whereas sulfur was replaced in benzoyl fluoride. Dichloromethane with $Et_4NF.4HF$ was found to be the best choice as a solvent, helping with salt

and fluorine needed for anodic fluorination. Pt—Pt electrode-based electrochemical strategy worked well for fluorination methodology. No relationship between the substituents and the oxidation potentials could be observed (Scheme 13).



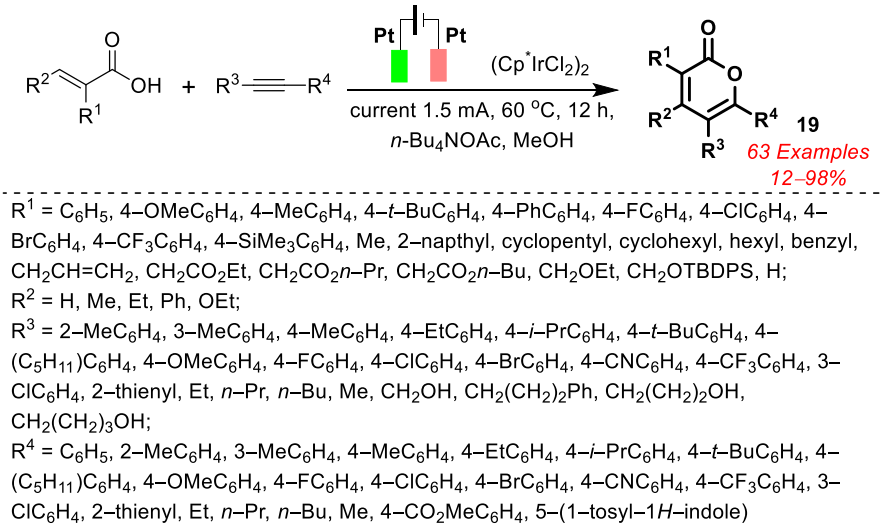
Scheme 13. Electrochemical fluorination of *S*—butyl benzothioates.

Another Pt-Pt electrode-based electrochemical synthesis of bis—indole methane derivatives from a broad range of indoles and ethers has been developed by Huang's research team [61] in 2018. The reaction of substituted indole and furan successfully delivered bis-indole methane derivatives **18**. The final products **18** were obtained in 29–92% yields at room temperature. The authors prepared a total of 21 examples to demonstrate the generality of the protocol. The reaction has high functional group tolerance and substrate scope at room temperature in the absence of chemical oxidants. This reaction was well tolerated with both electron-donating and electron-withdrawing groups in general (Scheme 14).



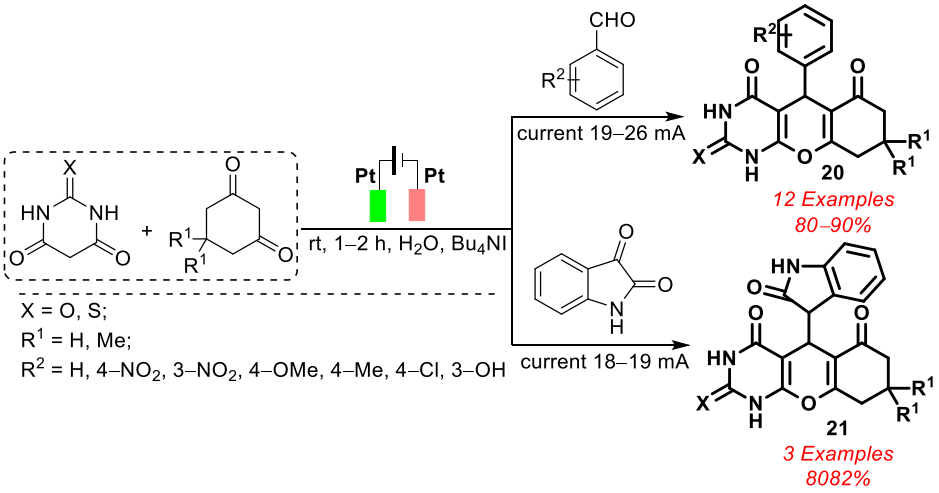
Scheme 14. One—pot synthesis of bis-indole methane.

The Mei's group [62] demonstrated the site-selective C—H functionalization by a Pt—Pt electrode-based electrolytic method (Scheme 15). Here, they report on the development of electrochemical vinylic C—H functionalization of acrylic acids with alkynes. The procedure examined an iridium catalyst to permit C—H/O—H functionalization for alkyne annulation. The reaction resulted in the synthesis of α—pyrones **19** with good to excellent yields (12–98%) in an undivided cell. This protocol is preferable to Co- or Ru-catalyzed annulation protocols using chemical oxidants to obtain highly selective regioselectivity with unsymmetrical internal alkynes. Note that the traditional chemical oxidants such as Ag(I) or Cu(II) failed to afford a significant amount of desired product **19** in the absence of electricity.



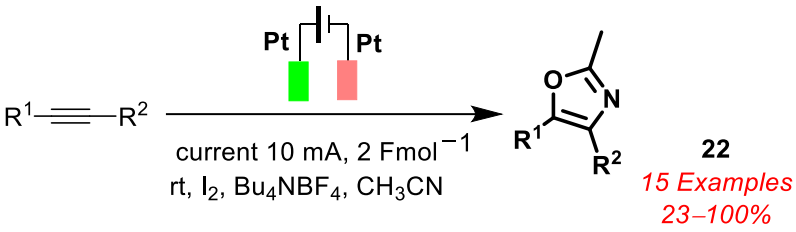
Scheme 15. C—H Activation for the construction of α—pyrones.

In 2020, using the same electrode, an alternate protocol was developed by Singh and coworkers [63] for the synthesis of novel chromeno[2,3—*d*]pyrimidine scaffolds (Scheme 16). One-pot multicomponent reactions assembled barbituric acids, 1,3 cyclic diketones, and substituted benzaldehydes in the presence of TBAI as an electrolyte in an aqueous medium. Accordingly, chromeno[2,3—*d*]pyrimidines (**20**) were achieved in high yields (80–90%). In contrast, isatin was employed instead of benzaldehydes under similar reaction conditions to afford isatin-tethered chromeno[2,3—*d*]pyrimidines **21** in 80–82% yields. It also proved advantageous for gram-scale synthesis.



Scheme 16. Development of chromeno[2,3—*d*]pyrimidine scaffolds.

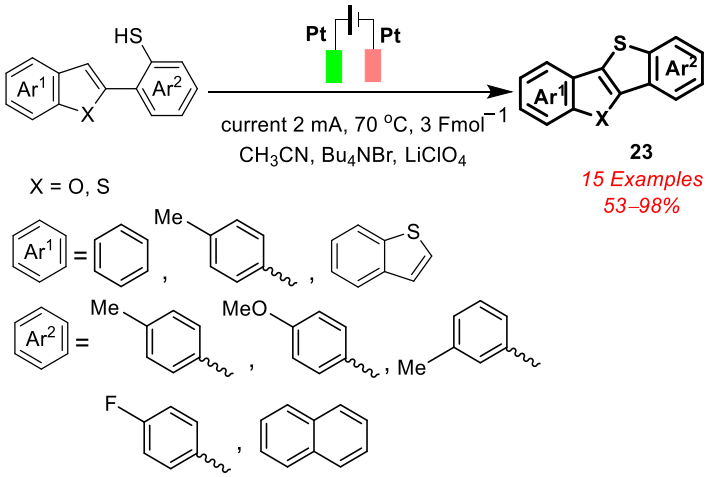
In 2020, Hilt et al. [64] devised a synthesis of 1,3—oxazoles **22** from symmetrical and unsymmetrical alkynes. The present synthesis was achieved using iodonium cation-pool electrolysis of I_2 in acetonitrile with a precisely controlled water content. As a result, 1,3-—oxazoles **22** were obtained as cyclized products in 23–100% yields. A specific selection of unsymmetrical alkynes was explored, resulting in the formation of a single regioisomer, the structure of which was elucidated through $^{1}H/^{15}N$ correlation NMR spectroscopy. Unfortunately, the transformation was unsuccessful for electron-deficient diethyl but—2—ynedioate and for terminal alkynes, including conjugated 1,3—diyne (1,4—diphenylbuta—1,3—diyne) and phenylacetylene, resulting in multiple side products (Scheme 17).



R^1 = C_6H_5, 2–MeC_6H_4, 3–MeC_6H_4, 4–MeC_6H_4, 4–$NO_2C_6H_4$, 4–$CF_3C_6H_4$, 2–$CO_2EtC_6H_4$, 4–FC_6H_4, 4–ClC_6H_4, 4–BrC_6H_4, 4–CNC_6H_4, 4–$OMeC_6H_4$, C_6H_{13};
R^2 = Me, C_6H_5, 2–MeC_6H_4, 3–MeC_6H_4, 4–MeC_6H_4, 4–$NO_2C_6H_4$, 4–$CF_3C_6H_4$, 2–$CO_2EtC_6H_4$, 4–FC_6H_4, 4–ClC_6H_4, 4–BrC_6H_4, 4–CNC_6H_4, 4–$OMeC_6H_4$, C_6H_{13}

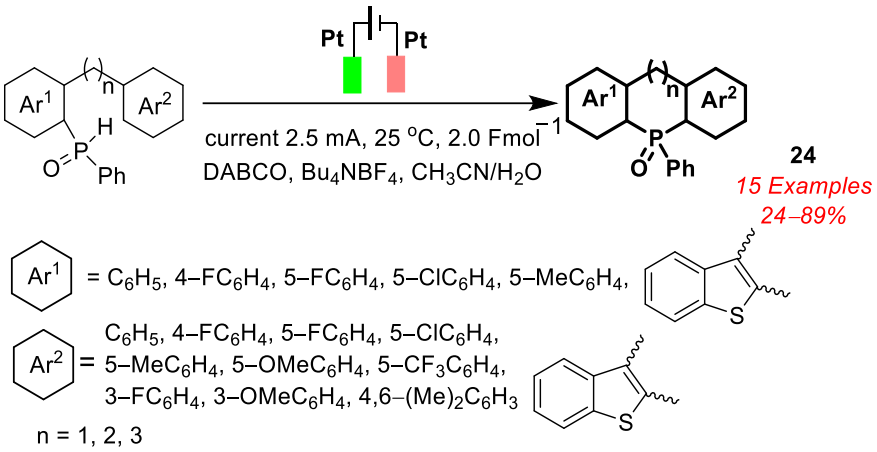
Scheme 17. Electro—organic synthesis of oxazoles.

In 2020, Suga and coworkers [65] published the first electrochemical dehydrogenative C—S bond formation to afford the thienoacene derivatives **23**. A number of thienoacene derivatives were created through the dehydrogenative coupling of C—H/S—H. This approach allows for the production of π-expanded thienoacenes under mild conditions, without the use of transition metals and with only a catalytic amount of a halogen mediator. The cyclized products **23** were obtained in 53–98% yields with 15 examples. All the substituted starting reactants worked well to smoothly conduct the reaction. It was crucial to add *n*—Bu4NBr, a halogen mediator that catalytically accelerated the process (Scheme 18).



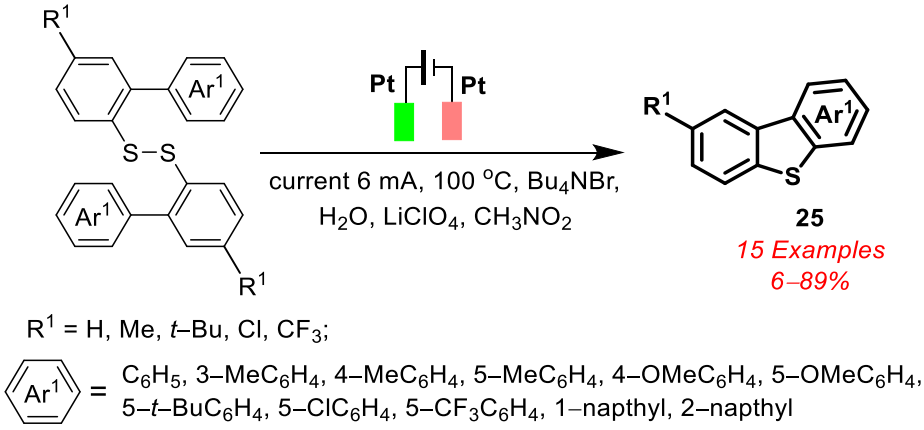
Scheme 18. Electro—oxidative dehydrogenative cyclization.

In 2021, Suga's research team [66] also investigated an electrochemical method towards the synthesis of diarylphosphole oxides **24**. The practical protocol uses readily accessible and cost-effective DABCO as a H—transfer mediator, resulting in a range of diarylphosphole oxides **24** with high yields (24–89%). Pleasingly, this method was also applicable to the synthesis of six-membered phosphacycles (phenophosphazines). Note that the control experiments indicated that a phosphinyl radical was produced in situ, and the strategy is likely to follow a radical route (Scheme 19).



Scheme 19. Construction of diarylphosphole oxides.

In the upcoming year, Suga et al. [67] extended our study towards the formation of dibenzothiophenes **25** introducing the same cell and different reaction conditions. They explored Br^- as a halogen mediator to create an electro-oxidative C—S coupling for the production of dibenzothiophenes **25**. The reaction required the use of Bu_4NBr and CH_3NO_2, and under ideal conditions, a wide range of dibenzothiophene derivatives **25** were produced. A wide variety of dibenzothiophene derivatives **25** were constructed in good yields (6–89%) (Scheme 20).



Scheme 20. Electroorganic construction of dibenzothiophenes.

Initially, Br^- from Bu_4NBr would undergo oxidation to produce [Br^+]. Next, the electrochemical process was significantly enhanced in the presence of Li^+, perhaps due to the resultant low-concentration Br_2 oxidizing compound (**I**) to biphenylsulfanyl bromide (**II**). Also, intramolecular nucleophilic attack of the benzene ring on the sulfur atom would give intermediate (**III**). Then, the next deprotonation of intermediate (**III**) would give the required molecule (Figure 9).

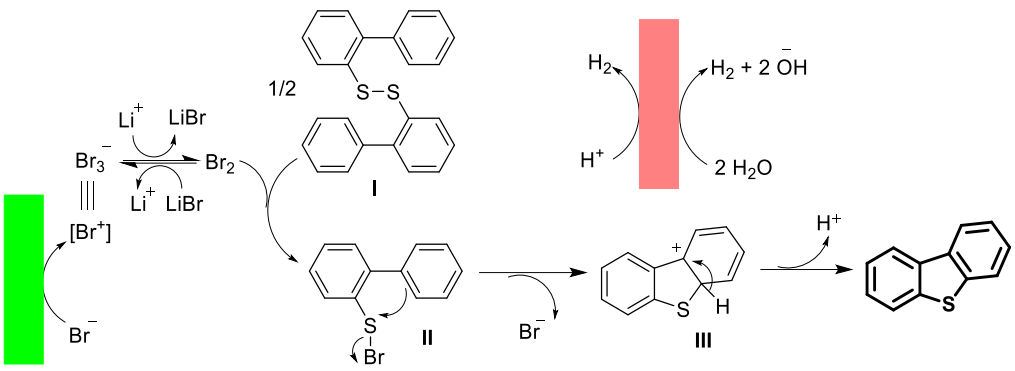
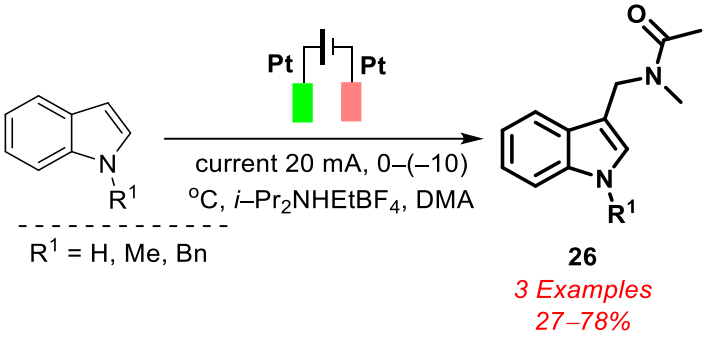


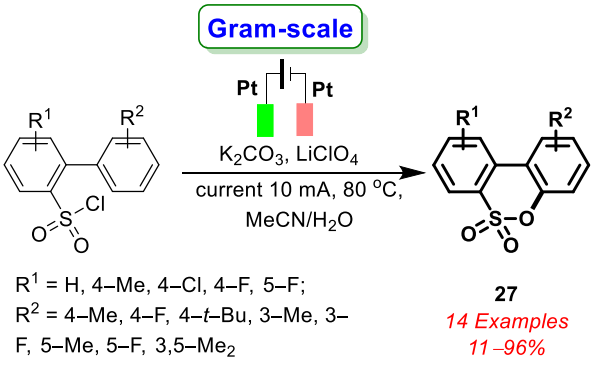
Figure 9. Plausible reaction mechanism.

Later that year, the research team of Senboku [68] in 2022 described a new electrochemical oxidation system through a quasi-divided Pt—Pt-based cell. In this procedure, trial-kylammonium tetrafluoroborates were used as a supporting electrolyte and a source of protons for the cathodic reduction process. In an electrolysis of 1,3,5—trimethoxybenzene/indoles in DMA containing *i*—$Pr_2NHEtBF_4$ in a quasi-divided cell with a Pt wire anode and a Pt plate cathode, *N*—acyliminium ions were selectively generated at the anode. These ions interacted with arenes to provide the corresponding amidomethylated indole products **26** in high yields (27–78%) (Scheme 21).



Scheme 21. Amidomethylation of indoles via electrochemical method.

In 2023, Mitsudo et al. [69] focused their work on the electrochemical C—O bond formation approach by using the same cell. In the presence of K_2CO_3 and H_2O, the constant current electrolysis of [1,1′—biphenyl]—2—sulfonyl chloride quantitatively delivered aryl-fused sultone scaffolds. A diverse array of sultone derivatives **27** was obtained using diverse starting materials. Under mechanistic investigation, it was revealed that the protocol progressed through a radical mechanistic pathway (Scheme 22).

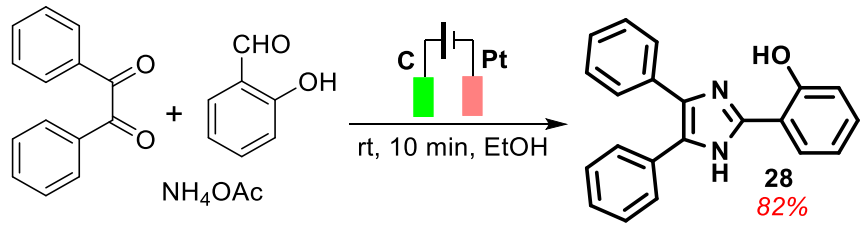


Scheme 22. Pt—Pt-based electrochemical method to access sultones.

7.3. C—Pt-Based Electrode

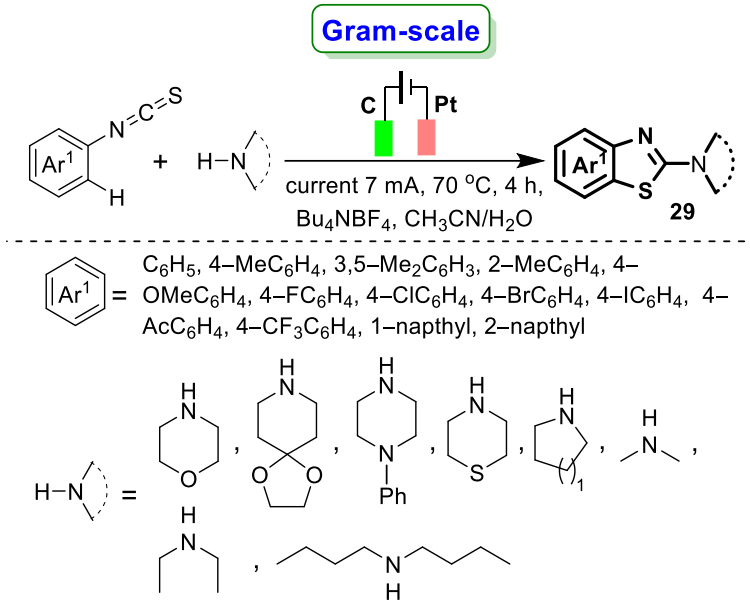
In this section, we describe the remarkable adaptability and efficiency of C—Pt electrodes in modern electro-organic synthesis. These systems combine the stability and conductivity of carbon with the catalytic power of platinum to provide a cost-effective and balanced strategy for the synthesis of a wide range of organic compounds. Further, C—Pt electrode systems are compatible with flow chemistry and photochemical techniques. The literature discussed shows the ability of C—Pt electrodes to promote multicomponent reactions, dehydrogenative cross-couplings, cyclizations and ring expansions under mild and environmentally benign conditions.

In 2017, Sarala's research team [70] illustrated a C—Pt electrode-based multicomponent electro-organic synthesis of 2—(4,5—diphenyl-—1*H*—imidazol—2—yl)phenol **28** (Scheme 23). The electrochemical method was performed through the cyclocondensation reaction of benzil and 2—hydroxybenzaldehyde using a C—Pt two-electrode setup. Interestingly, it was observed that the reaction was completed within only 10 min and delivered the final product **28** in 82% yield. Additionally, density functional theory (DFT) studies of the created derivative were conducted to theoretically determine the band gap energy. These results indicate that a 2—(4,5—diphenyl—1*H*—imidazol—2—yl)phenol monomer-based electrode is a viable alternative to electrochemical supercapacitor applications without necessitating modification.



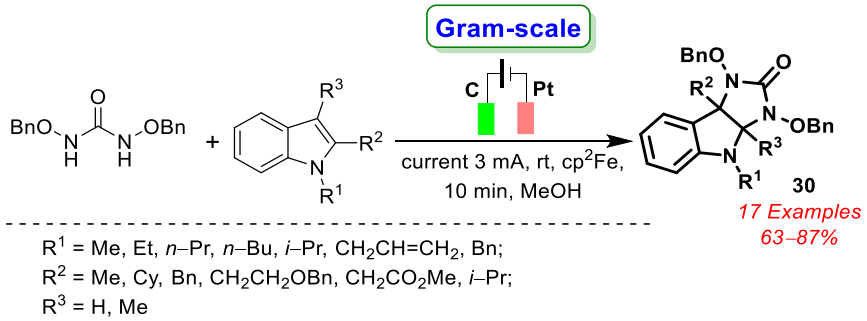
Scheme 23. Synthesis of substituted imidazole by using C-Pt electrode.

In the same year, Lei and coworkers [71] also published a C—Pt electrode-based strategy for the development of 2—aminobenzothiazoles **29** (Scheme 24). Under this methodology, an external oxidant-free intramolecular dehydrogenative C—S cross-coupling was conducted. Numerous 2—aminobenzothiazoles **29** were synthesized by directly combining aryl isothiocyanates and amines. Phenyl isothiocyanates with electron-donating groups (methyl and methoxyl) resulted in marginally reduced reaction yields. Halide substituents (F/Cl/Br/I) were effectively accommodated, yielding the corresponding benzothiazoles in favorable to excellent yields. Electron-deficient phenyl isothiocyanates exhibited remarkable reactivity in this transformation. Additionally, both 1-isothiocyanatonaphthalene and 2—isothiocyanatonaphthalene successfully produced the targeted compounds in satisfactory yields. Subsequently, other secondary amines were explored as substrates in the synthesis of benzothiazoles. Cyclic amines (thiomorpholine, 1,4—dioxa—8—azaspiro[4.5]decane, and 1—phenylpiperazine) exhibited remarkable reactivity in the production of 2-aminobenzothiazoles. Primary amines may react with aryl isothiocyanates, but no cyclization product can be achieved under typical conditions. It is delightful to note that a two-component reaction can be performed on a gram scale as well.



Scheme 24. Scope of benzothiazoles synthesis.

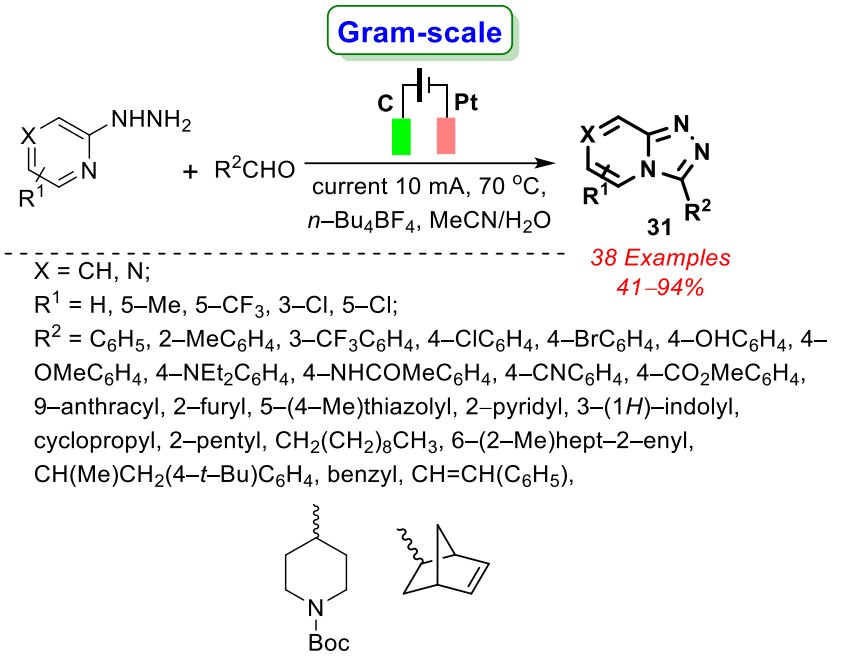
Furthermore (in 2018), the electro-oxidative generation of the diaza—oxyallyl cation and its application in the synthesis of fused indole-based frameworks **30** have been examined by Luo's research team [72] (Scheme 25). The current electrochemical technique eliminated the necessity for a supporting electrolyte and external base, avoiding the use of chemical oxidant. As a result, current electro-organic synthetic methods successfully completed the reaction in only 10 min and delivered 30 final products with yields ranging from 63% to 87%. The protocol assembled 1,3—bis(benzyloxy)urea and substituted indole as starting precursors in the presence of MeOH. The C5 position of indole was also well tolerated with electron-withdrawing groups. In addition, the reaction was compatible with a diverse variety of functional groups at the C3 position of indole.



Scheme 25. Two-component electrochemical formation of indole-based frameworks.

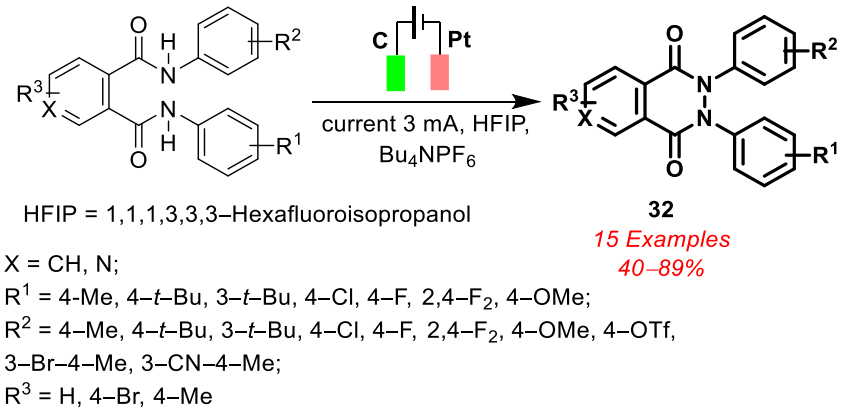
Alternatively, an electrolytic process for dehydrogenative C—N cross-coupling was established by the group of Zhang and their coworkers [73] (Scheme 26). This green methodology was conducted by using aliphatic/(hetero)aromatic aldehydes and 2—hydrazinopyridines at 70 °C. 1,2,4—Triazolo[4,3—*a*]pyridines **31** were achieved in 41–94% yields. Moreover, this straightforward transition required neither metal catalysts nor chemical oxidants, and diverse functional groups are compatible with these mild reaction conditions. Interestingly, this reaction can be performed on a gram scale and used for the manufacture of the highly marketed medication Xanax and its biologically pertinent counterparts. It is remarkable that numerous functional groups including hal-

ogens, free alcohols and strong electron-donating or electron—withdrawing groups were well tolerated.



Scheme 26. C—Pt electrode-assisted synthesis of 1,2,4—triazolo[4,3—*a*]heterocycles.

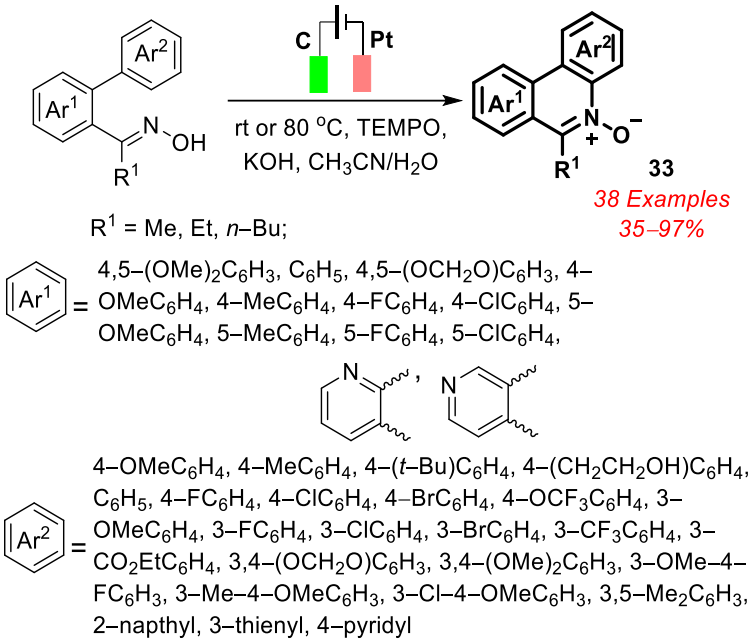
In later work, Waldvogel et al. [74] in 2018 checked a sustainable method for obtaining phthalazin—1,4—diones **32** using C—Pt cell. The reaction assembled aryl-substituted diamide compounds in the presence of HFIP. Phthalazin—1,4—diones were obtained in 40–89% yields by generating 15 examples. It is delightful to share that diversified starting substrates were tolerated well for the current intramolecular cyclization approach. Straightforward and sustainable access to this class of substrates is made possible by a very simple setup, conditions free of metal catalysts and oxidizers, and scalable and durable electrode materials. Both electron-withdrawing and electron-donating substrates are compatible and deliver favorable results. The capability to convert non-symmetric dianilides underscores the substantial significance of this technology (Scheme 27).



Scheme 27. Strategy for the synthesis of phthalazin—1,4—diones.

In 2018, Zhao and coworkers [75] also employed C—Pt cell to conduct the electrochemical C—H activation of biaryl ketoximes **33**. Dehydrogenative cyclization of the oxime substrates with a Pt cathode provided unparalleled access to a variety of

N—heteroaromatic N—oxides. The transformation was accomplished by using TEMPO, KOH, and a mixture of acetonitrile and water. The authors prepared a huge library of 38 examples to demonstrate the protocol's versatility. Pleasingly, the final products **33** were obtained in high yields (35–97%) (Scheme 28).



Scheme 28. Substrate scope for the dehydrogenative cyclization.

The electrolysis initiates with the anodic oxidation of TEMPO to form $TEMPO^+$, which subsequently interacts with (**I**) to yield an iminoxyl radical (**II-III**). Next, the N—cyclization of the iminoxyl radical onto the phenyl ring, and then restoring its aromatic structure, produces the N—oxide (**IV**). Conversely, employing a Pb cathode, characterized by a greater overpotential for H_2 development, would promote the generation of the final product through the cathodic breakage of the N—O link in (**IV**) (Figure 10).

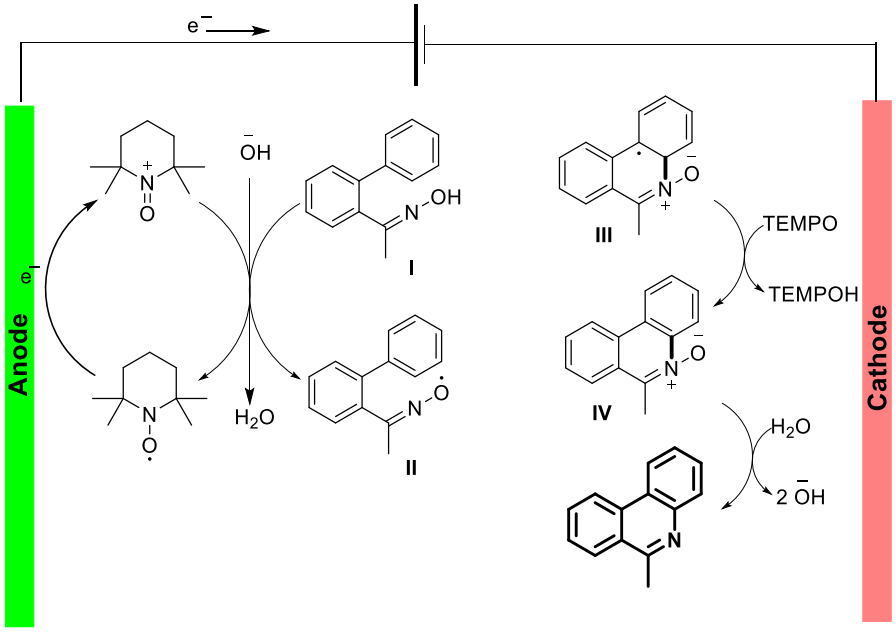
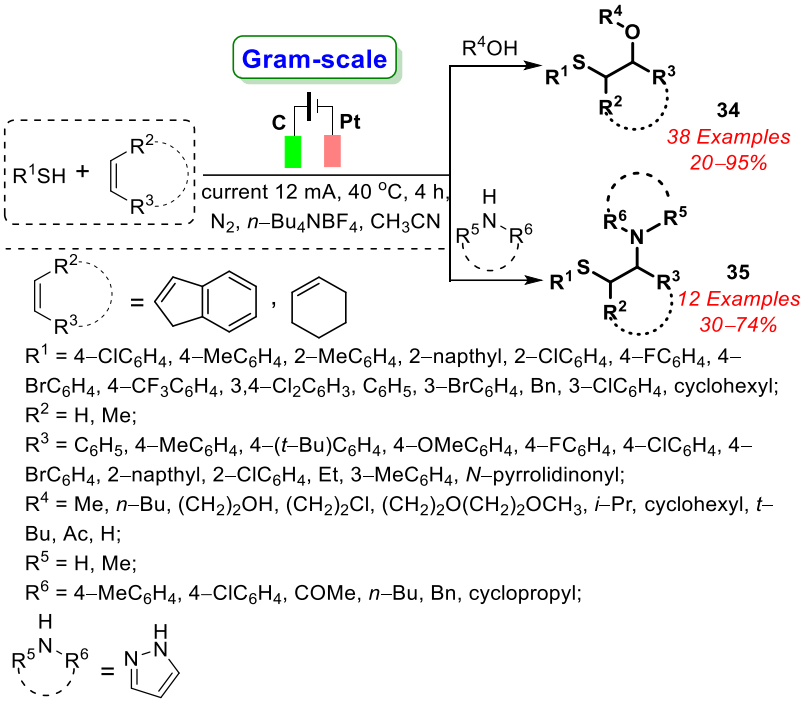


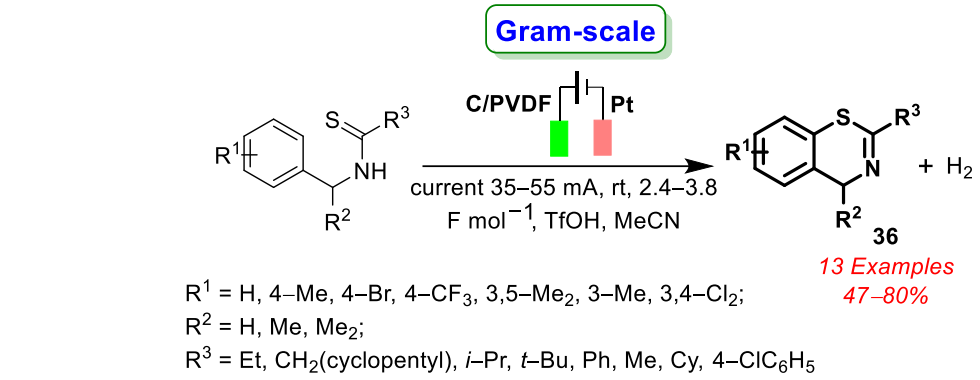
Figure 10. Proposed mechanistic study.

In 2018, Lei's research team [76] reported an electrochemical oxidative oxysulfenylation and aminosulfenylation of alkenes with good selectivity directly employing thiophe-nols/thiols as thiolating agents. Electrochemical approach based on C—Pt electrode has been successfully used for the synthesis of *β*—alkoxy sulfides **34** and β-amino sulfides **35**. Under electrochemical oxidation conditions, a number of *β*—alkoxy sulfides and *β*-amino sulfides were formed in good to outstanding yields (**34**, 20–95%; **35**, 30–74%). The required compounds were obtained in moderate to good yields using various thiophenols containing electron-withdrawing and electron-neutral groups. It is worth to remark that other O—nucleophiles such as acetic acid and water were equally efficient for this reaction affording the respective acyloxysulfenylation and hydroxysulfenylation products (Scheme 29).



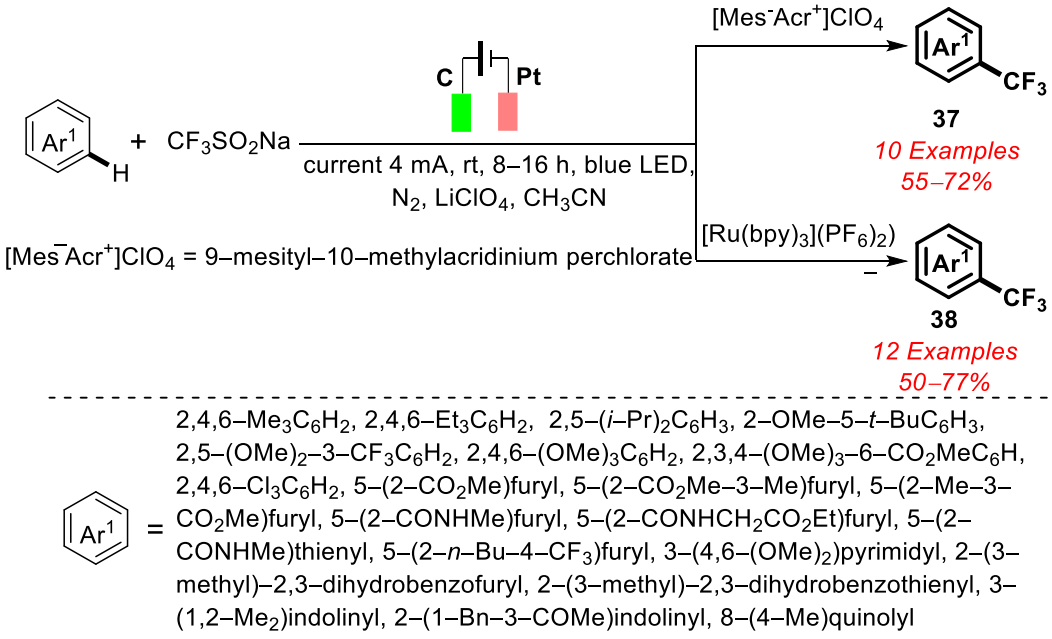
Scheme 29. One-pot difunctionalization of alkenes.

In 2019, intramolecular C—S cross-coupling was performed by Xu et al. [77] to afford the synthesis of 1,3—benzothiazines **36**. In a flow electrolysis cell (C/PVDF—Pt), N—benzyl thioamides undergo electrochemical dehydrogenative cyclization. The electrosynthesis has overcome the drawbacks of prior alkylthioamide cyclization procedures and provides transition metal- and oxidizing reagent-free access to functionalized 1,3—benzothiazines **36** in good yields (47–80%). This electrochemical protocol can be extended to include the formation of a seven-membered ring. No auxiliary salt was required, as the incorporation of TfOH enhanced the conductivity of the reaction solution. Interestingly, the electrochemical dehydrogenative cyclization reaction also worked well for making seven-membered rings (Scheme 30).



Scheme 30. One—pot dehydrogenative cyclization of thioamides.

Later work by Ackermann and coworkers [78] in 2020 developed the first C—Pt electrode-based electrochemical C—H trifluoromethylation enabled by the cooperative action of photochemistry and electrochemistry. The reaction was conducted by using substituted arenes/heteroarenes and sodium trifluoromethanesulfonate at room temperature. C—H activation provided trifluoromethyl derivatives of arenes/heteroarenes **37-38** in the presence of [Mes$^-$Acr$^+$]ClO_4 and [Ru(bpy)$_3$]$(PF_6)_2$), respectively. The strategy featured a broad substrate scope (**37**; 55–72%, 10 examples, and **38**; 50–77%, 12 examples) and a high functional group tolerance. A diverse array of heteroarenes demonstrated compatibility with the optimized electrophotochemical transformation, like benzofuran, furan, thiophene, quinoline, benzo[*b*]thiophene, indole, and pyrimidine, affording the desired trifluoromethylated products with high selectivity (Scheme 31).



Scheme 31. Electrophotochemical C—H trifluoromethylation of heteroarenes and arenes.

The first step, irradiation of the organic dye Mes-Acr$^+$, gives the excited state oxidized, Mes-Acr^{+*}. A SET process between the sulfinate anion and Mes-Acr^{+*} leads to the formation of the acridinyl radical and the CF_3SO_2 radical. The latter is followed by anodic electrooxidation of the acridinyl radical to restore the ground-state catalyst Mes-Acr$^+$. Meanwhile, the trifluoromethyl radical adds to the substrate (**I**) to give intermediate radical that is oxidized by single electron transfer to give the cationic Wheland complex intermediate (**II**). At the same time, (**I**) may be immediately oxidized at anode. The pro-

tons are eventually abstracted to create the target product and are reduced to give H_2 at the cathode (Figure 11).

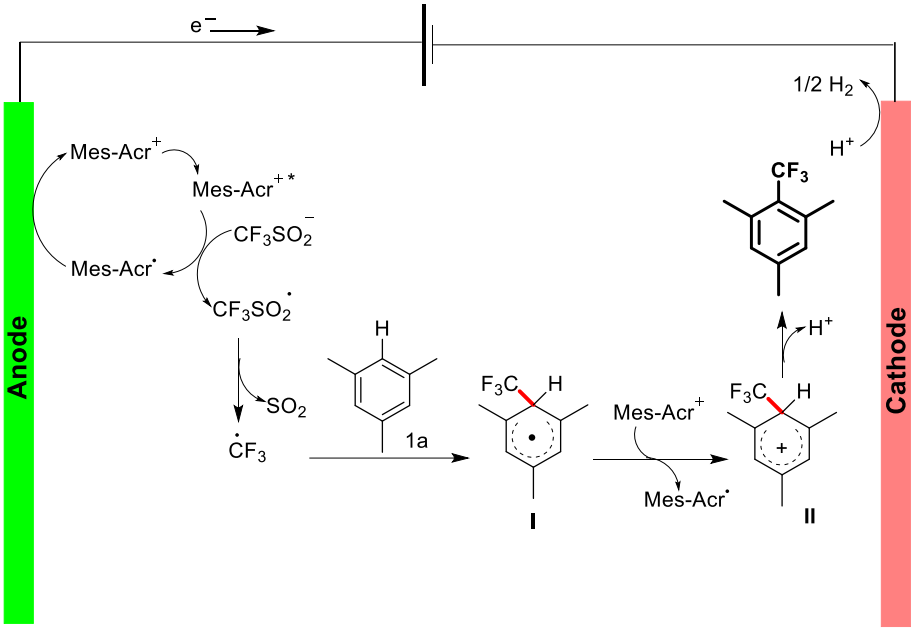
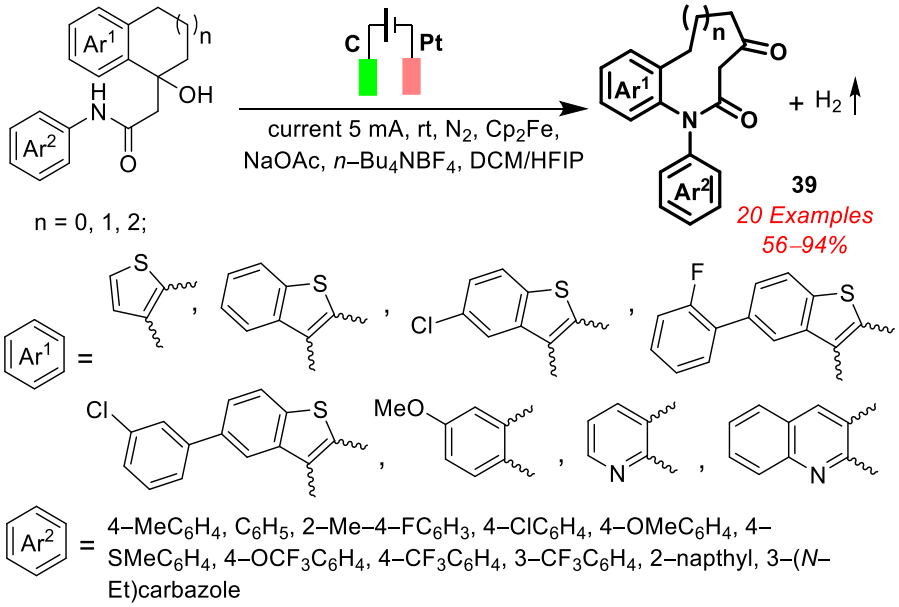


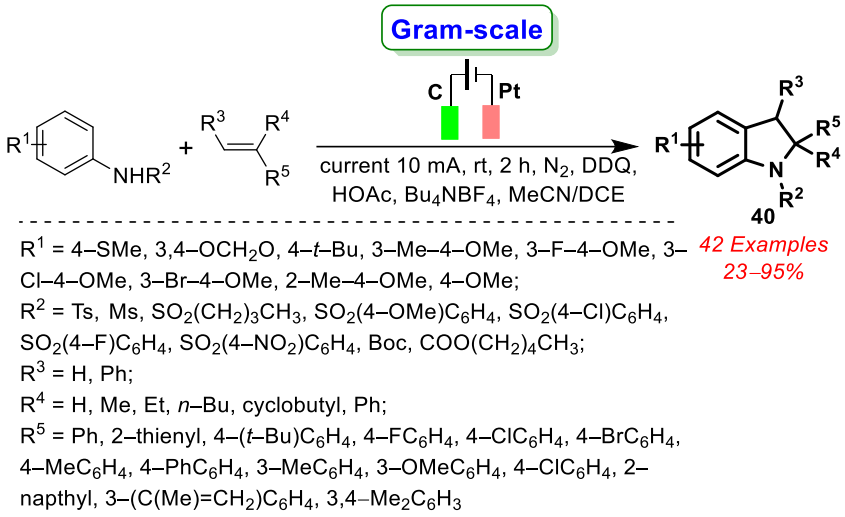
Figure 11. Putative reaction mechanism.

In 2020, the research group of Liu [79] reported a feasible electrochemical ring expansion methodology using commercial benzocyclic amides. A series of highly functionalized eight- to eleven-membered lactams **39** was successfully synthesized with high yields (56–94%). This transition exhibits remarkable tolerance to both very electron-deficient and electron-rich benzo-cyclic ketone substrates. Significantly, satisfactory reaction yields (56–94%) were achieved using gram-scale one-pot synthesis. The practical applicability of this procedure was further shown by the successful expansion of substrates, consisting of fused arenes within the backbones of the expanding rings. Mechanistic investigations reveal that this ring enlargement occurs via a distinctive remote amidyl radical movement that facilitates C—C bond breaking (Scheme 32).



Scheme 32. Electrochemical synthesis of lactams.

By applying the same electrode further, a regioselective intermolecular electro—oxidative cyclization was explored by Lei's research team [80] to afford the formation of indolines **40** (Scheme 33). Accordingly, indolines **40** were achieved in high yields (23–95%) within 2 h of reaction time. Amazingly, electrolyte-free reaction environments were achieved in an electrochemical flow cell of C—Pt. It is intriguing to note that before the electrochemical intermolecular oxidative [3+2] annulation of alkenes and anilines for indoline synthesis, external oxidants were removed. The indoline derivatives were successfully synthesized in electrolyte-free conditions with merely 5 mol% DDQ, indicating the method's potential for the application. Numerous alkenes (e.g., heterocyclic olefins/1,1—diphenylethylene/α—methylstyrenes/styrene) exhibited considerable tolerance. Moreover, a diverse array of functional groups was tested and delivered to a library of 42 examples.



Scheme 33. Electro—organic synthesis to access indolines.

In the mechanistic investigation, the reaction commences with the anodic oxidation of aniline (**I**). The next step of losing a proton might create N-radical species (**II**), which can change to release C-radical species (**III**). The intermediate (**IV**) may be synthesized via the radical addition of **III** and alkene (**IV**). Thereafter, intermediate (**V**) undergoes oxidation by anodic oxidation. The target scaffold is constructed via an intermolecular approach. The simultaneous cathodic reduction in protons results in the production of dihydrogen (Figure 12).

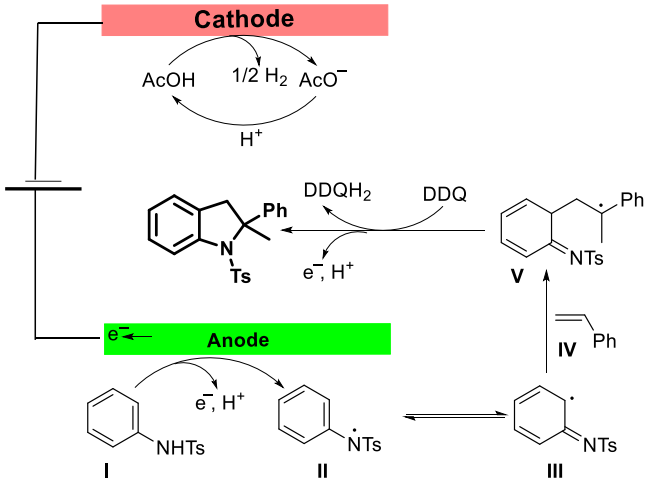
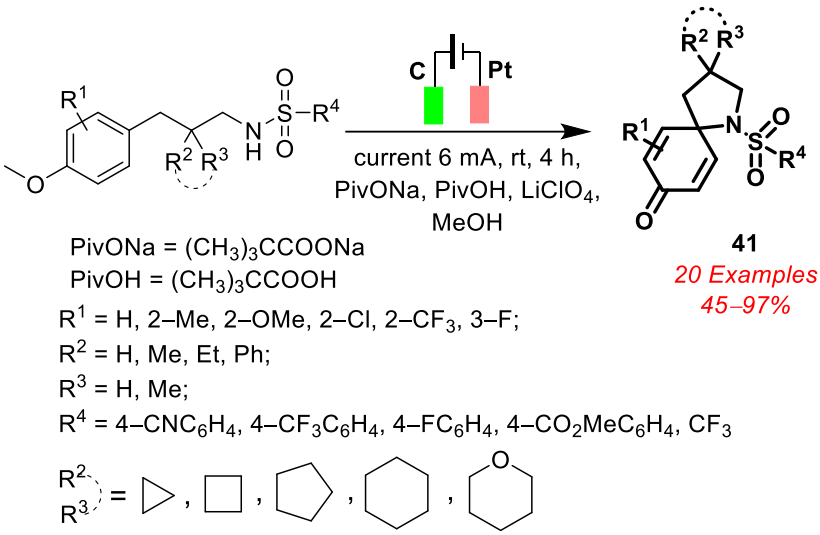


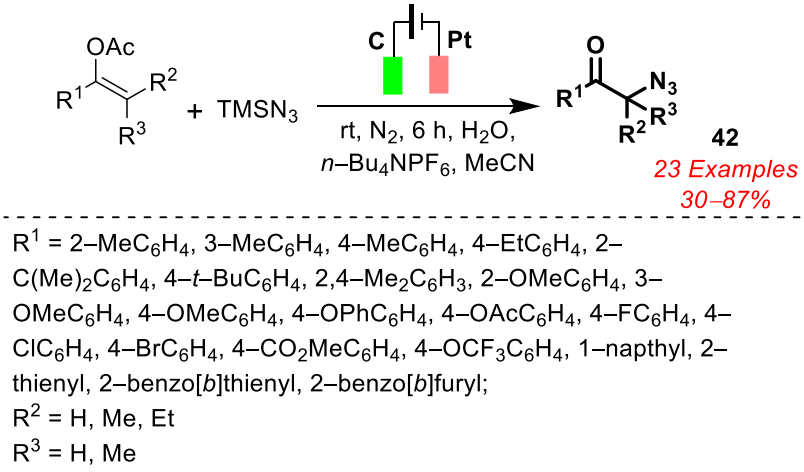
Figure 12. Plausible mechanism.

In 2021, Zhang et al. [81] presented a strategy for anodic oxidation spiroamination and spirolactonization of anisole derivatives, accompanied by the cathodic reduction in protons, without the need for hypervalent iodine reagents. Delightedly, anisole derivatives containing electron-withdrawing groups were also viable in this electrochemical system, which posed significant challenges under chemical oxidation conditions. Electron-withdrawing groups (chloro, trifluoromethyl, and fluoro) as well as electron-donating groups (methyl and methoxy) were all compatible with this transformation. A varied array of spiropyrrolidines **41** with various functional groups served as effective scaffolds in this transformation, achieving yields of up to 97% (Scheme 34).



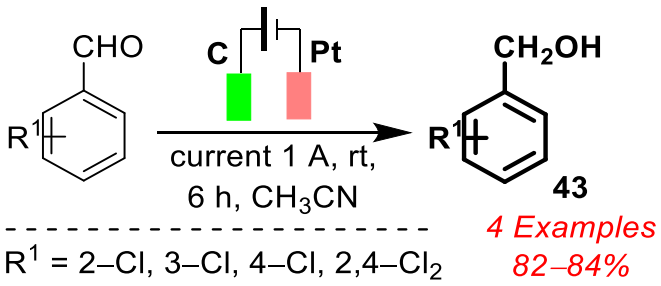
Scheme 34. Electrochemical cyclization to access spiro derivatives.

In 2022, Shen et al. [82] devised a C—Pt electrode-based electro-organic reaction to afford α—azidoketones **42**. The reaction was completed by oxyazidation of α—arylvinyl acetates to access diverse vicinal α—azidoketones **42**. A library of 30 examples was generated in 30–87% yields to check the versatility and generality of the protocol. The protocol tolerates a broad substrate scope and employs an experimentally simple undivided electrochemical cell. Interestingly, naphthalene/thiophene/benzothiophene/benzofuran were all amenable to this transformation. Moreover, the produced α—azidoketones have been proven to be useful building blocks for the production of biologically relevant heterocycles (Scheme 35).



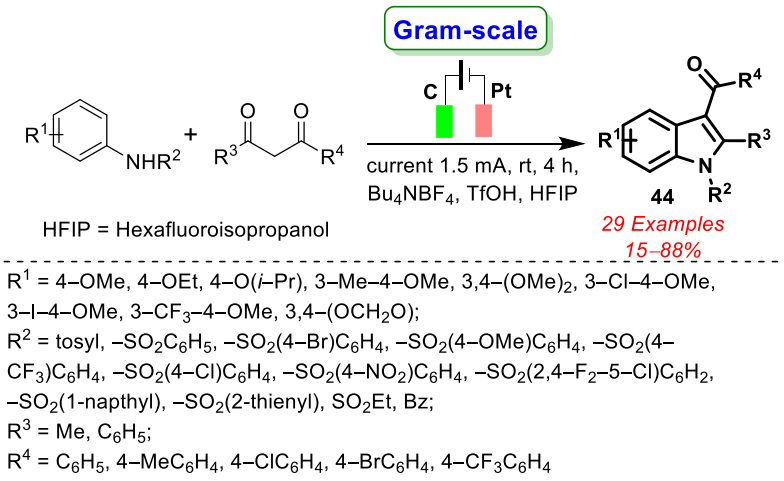
Scheme 35. Electrochemical conversion of vinyl acetates to α—azidoketones.

Electro-organic conversion of various chloro-substituted benzaldehydes into benzyl alcohols **43** disclosed by Saharan's research team [83] in 2022. The current interesting transformation was achieved via C-—Pt electrode-based electrochemical reduction using cyclic voltammetric and constant current electrolysis. C—Pt worked well for the conversion of aldehyde groups into alcohol in the presence of acetonitrile as a solvent at room temperature. The authors generated a total of four examples (**43**) in 82–84% yields to demonstrate the protocol's generality. It is a promising way as in these reactions electrons act as reagent which gives a green and sustainable way for the synthesis of value-added organic molecules (Scheme 36).



Scheme 36. Electrochemical synthesis of 2,4—dichlorobenzaldehyde.

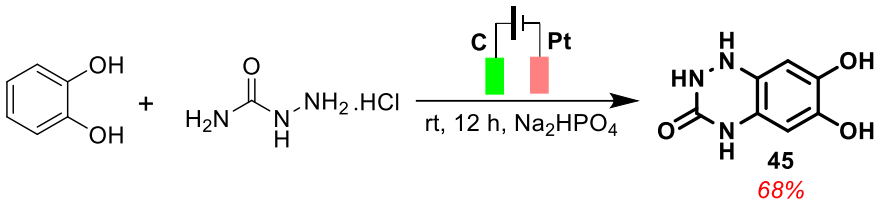
Here, Sun et al. [84] (in 2022) synthesized another indole derivative (**44**) by using different reaction conditions (Scheme 37). Under this work, the authors report the first intermolecular electro-oxidative cycloaddition of anilines and 1,3—dicarbonyl compounds for the formation of fused derivatives such as indoles **44**. A transition-metal catalyst or chemical oxidant is not needed to synthesize a wide spectrum of multisubstituted indoles using this approach. Intermolecular processes between simple anilines and other widely available synthons, however prevalent, have not been studied previously. However, when there was an electron-withdrawing group (NO_2 or CN) at the C—4 position, an indole was not obtained. A wide variety of starting substrates were investigated to prove the generality of the current green protocol, and it was observed that the protocol was well tolerated. A total of 29 examples were obtained in 15–88% yields.



Scheme 37. Synthetic route to indoles by using C—Pt cell.

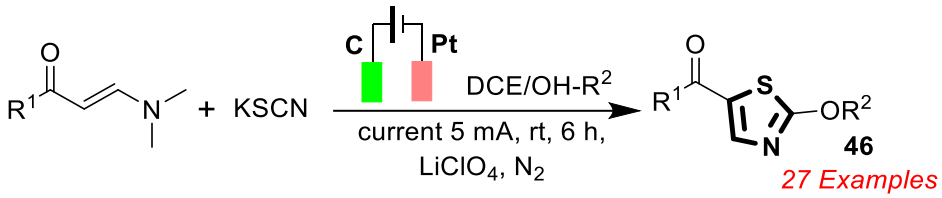
Next (in 2023), the green electrochemical synthesis of catechol oxidation to *o*—benzoquinone **7** was performed by Alsoghier and their coworkers [85] (Scheme 38). Noted semicarbazide as a nucleophile in ethanol/phosphate buffer with catechol yielded *o*—benzoquinone **45**. A new heterocyclic *o*—benzoquinone **45** product functionalized

with hydroxyl and amide groups was constructed with 68% yield. In this study, 1,2—Michael addition was conducted by using a phosphate-buffered solution with a pH of 7 and cyclic voltammetry and potential-controlled coulometry. Therefore, a phosphate-buffered solution was subsequently chosen as an optimal pH value to obtain a high yield (68%) of products.



Scheme 38. One-pot electro-organic synthesis of *o*-benzoquinone.

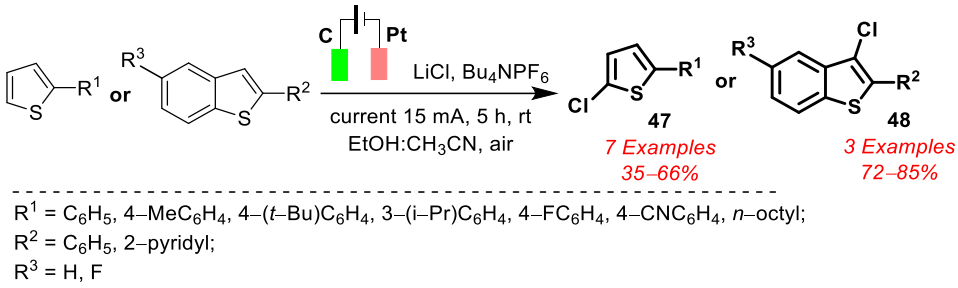
Wen's research group [86] in 2023 highlighted an electrochemical method for the formation of 2—alkoxythiazoles [49] (Scheme 39). A three-component cyclization of potassium thiocyanate and alcohols with enaminones was facilitated in the presence of a C—Pt electrode. The current technique provided an efficient and practical method for synthesizing diverse 2—alkoxythiazoles **46** in moderate to good yields (36–84%). The green methodology progressed efficiently via electrochemical oxidative C—H thiolation and nucleophilic tandem cyclization to form C—O/C—S/C—N bonds. Moreover, the procedure was compatible with heterocyclic enaminones (pyridine/furan/thiophene) yielding the required 2—methoxythiazole compounds (55–83% yields).



R^1 =NH, 4–Me, 4–OMe, C_6H_5, 4–ClC_6H_4, 4–BrC_6H_4, 4–IC_6H_4, 4–$SO_2MeC_6H_4$, 4–$CF_3C_6H_4$, 4–$NO_2C_6H_4$, 4–$NMe_2C_6H_4$, 4–$OCF_3C_6H_4$, 2–MeC_6H_4, 2–FC_6H_4, 2–ClC_6H_4, 3–MeC_6H_4, 3–BrC_6H_4, 3,4–$Me_2C_6H_3$, 3,4–$(OCH_2O)C_6H_4$, 3,4–$Cl_2C_6H_4$, 2–napthyl, 2–furyl, 2–thienyl, 2–pyridyl, 4–pyridyl, cyclopropyl;
R^2 = OMe, OEt

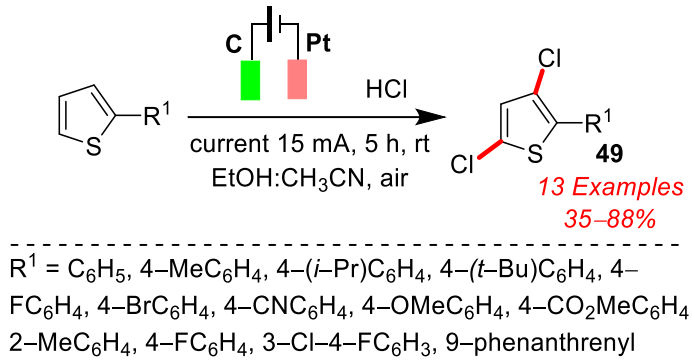
Scheme 39. Two-component synthesis of 2—alkoxythiazoles.

Recently (in 2024), Ye's research team [87] developed a green and efficient electrochemical strategy for the regioselective chlorination of 2—substituted thiophenes **47-48** (Scheme 40). The reaction of substituted thiophenes/benzothiophenes in the presence of C—Pt electrode delivered chlorinated products **47-48** in 35–66% and 72–85% yields, respectively. With this established protocol, monochlorination of 2—substituted thiophenes/benzothiophenes could be selectively achieved to produce 5—chlorinated/3—chlorinated thiophenes in moderate to excellent yields, respectively. Notably, the reaction worked well in mild conditions and tolerated functional groups well, yielding high yields and selectivity.



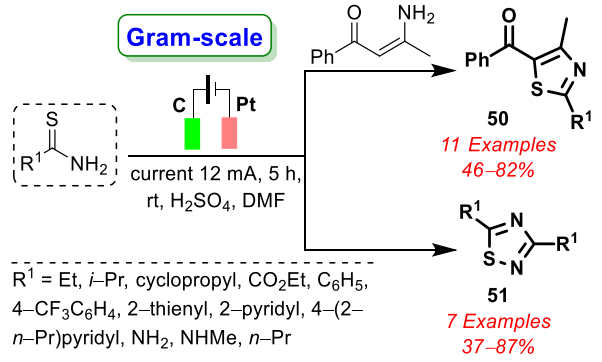
Scheme 40. Electrochemical-assisted chlorination of benzothiophenes.

In addition to the above work, the authors slightly changed the reaction conditions using HCl instead of LiCl and Bu_4NPF_6. It is interesting to share that the current reaction conditions afforded 3,5-dichlorinated thiophenes **49** in 35–88% yields. The protocol was also compatible with a wide variety of substituted thiophenes. Moreover, a library of 13 examples was generated to check the generality of the methodology (Scheme 41).



Scheme 41. Electrochemical-promoted dichlorination of thiophenes.

Recently (in 2024), Wan and their coworkers [88] reported a novel electrochemical synthetic route for the development of N/S—heterocycles such as benzothiazoles and 1,2,4—thiadiazoles (Scheme 42). A green electrochemical oxidative cyclization of enaminones was performed with thioamides under metal- and oxidant-free conditions. Accordingly, the substituted benzothiazoles **50** were obtained in 46–82% yields. On the other hand, electrochemical dimerization of thioamides without enaminones accessed 1,2,4—thiadiazoles under similar reaction conditions. 1,2,4—Thiadiazoles **51** were achieved in 37–87% yields across seven examples. Pleasingly, benzoyl enaminones containing various electron-deficient, electron-neutral, and electron-rich substituents in the phenyl moiety reacted effectively under normal circumstances, achieving the desired products **50-51** in moderate to good yields.

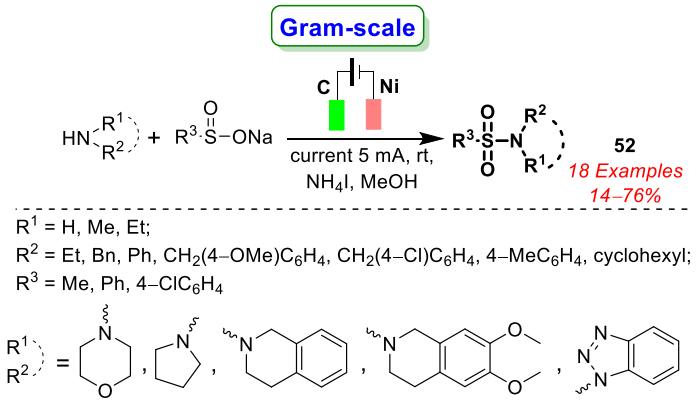


Scheme 42. Scope of thioamides in the synthesis of benzothiazoles/1,2,4—thiadiazoles.

7.4. C—Ni-Based Electrode

The section highlights the use of C—Ni electrodes as a remarkably efficient and cost-effective substitute for precious Pt-Pt/C-Pt systems for facilitating valuable synthetic transformations. C—Ni electro-organic methods are applicable under mild conditions and are often further improved by their combination with photocatalysis. The summarized reports reveal the excellent performance of C—Ni systems in promoting key reactions, such as oxidative amination, C—H sulfenylation and, especially, enantioselective cross-coupling reactions.

In 2016, Zeng et al. [89] highlighted a C—Ni electrode-based strategy towards the synthesis of sulfonamides from sodium sulfinates and substituted amines introducing electrochemical oxidative amination. The final products, sulphonamides **52,** were obtained in 14–76% yields at room temperature using a constant current of 5 mA. A substoichiometric quantity of NH_4I acted as a redox catalyst and a supporting electrolyte in a simple undivided cell. Aliphatic secondary amines (morpholine, pyrrolidine, and diethylamine) produced the respective adducts with yields of 64%, 71%, and 31%. Aromatic secondary amines were also compatible but reduced yields were noted. For instance, *N*—methylaniline produced a 14% yield of the respective product. Findings illustrate that benzylicamines, cyclohexanamine, and ethanamine produced the required products in moderate to favorable yields. The optimized conditions may also be utilized for aromatic amines, including *p*-chloroaniline and *p*—methylaniline, resulting in moderate yields of compounds. Delightedly, scaling up the experiment conducted by the authors demonstrated the industrial practicality of the protocol (Scheme 43).



Scheme 43. Amination of sodium sulfonate via electrochemical oxidation.

The reaction mechanistic pathway initiates with the anodic oxidation of the iodide ion to generate molecular iodine. Further, the in situ-generated molecular iodine can combine with amine (**I**) to deliver *N*—iodoamine (**II**). Upon additional interaction with sulfinate (**III**), sulfonamide is generated as a product (Figure 13).

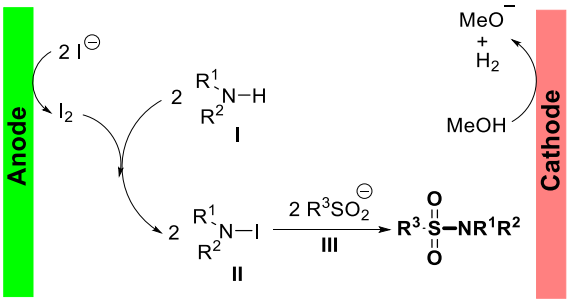
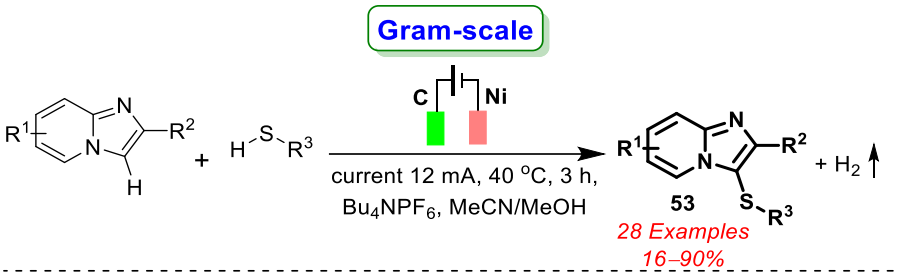


Figure 13. Plausible electrocatalytic cycle.

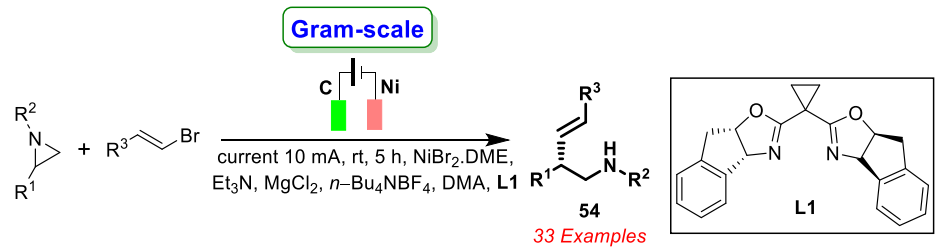
In order to explore the sulfur chemistry, Lei's research team [90] also performed selective oxidative C—H sulfenylation of imidazopyridine heterocycles in 2017. Imidazo[1,2—*a*]pyridines and thiols were subjected to the presence of Bu_4NPF_6, MeCN/MeOH at 40 °C. The sulfur insertion reaction delivered sulfur-linked imidazopyridines (**53**), with 16–90% yields. It is intriguing to share that both aryl and aliphatic thiols show high reactivity for C—S bond formation. Noted weak electron-donating substituents (methyl/isopropyl/*tert*—butyl groups) did not influence the reaction efficiency. Nonetheless, the highly electron-rich 4-methoxylthiolphenol demonstrated reduced reaction efficiency in this transition. The presence of 2—arylimidazo[1,2—*a*]pyridine did not affect the efficiency of the electrochemical oxidative C—S bond-forming reaction. Surprisingly, the C—2 substituent significantly influenced the reaction efficiency of oxidative C—H sulfenylation (Scheme 44).



R^1 = H, 6–Me, 6–F, 8–Me, 7–F;
R^2 = H, 4–FC_6H_4, 4–ClC_6H_4, 4–$CF_3C_6H_4$, 4–CNC_6H_4, 4–MeC_6H_4, 4–$OMeC_6H_4$, 2–napthyl;
R^3 = 4–ClC_6H_4, 3–ClC_6H_4, 2–ClC_6H_4, 4–FC_6H_4, 4–BrC_6H_4, 4–$CF_3C_6H_4$, 4–MeC_6H_4, 4–*i*–PrC_6H_4, 4–*t*–BuC_6H_4, 4–$OMeC_6H_4$, $CH_2C_6H_5$, $(CH_2)_2C_6H_5$, cyclopentyl, Et, dodecyl

Scheme 44. Synthesis of imidazopyridines through C-H sulfenylation.

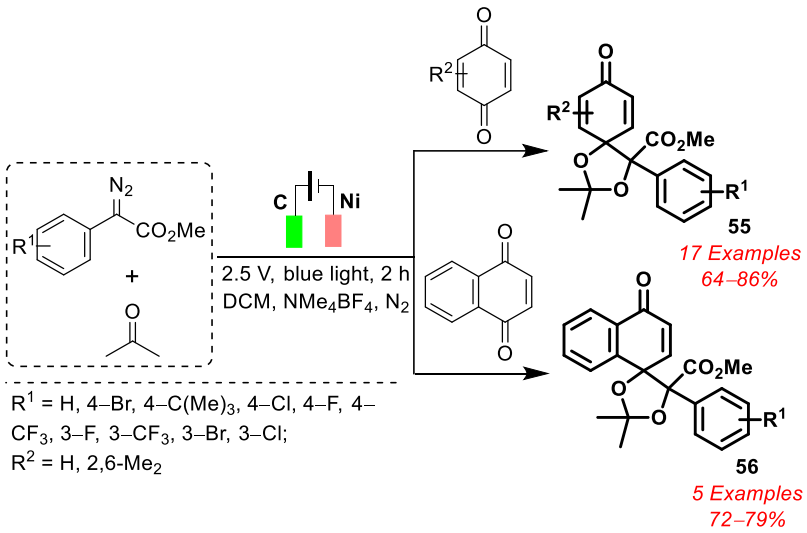
In 2023, Hu et al. [91] reported the first example of a nickel-catalyzed enantioselective electrochemical reductive cross-coupling. Chiral beta—aryl homoallylic amines **54** were prepared in 42–94% yields via the reaction of aryl aziridines with alkenyl bromides in the presence of triethylamine as a terminal reductant. The electroreductive approach, in the absence of heterogeneous metal reductants and sacrificial anodes, employs constant current electrolysis in an undivided cell (C—Ni electrode) with triethylamine as the terminal reductant. Pleasantly, the Cbz—protected aziridines showed good reactivity toward *β*-bromostyrene to generate the product in 76% yield. The transformation showed a broad substrate scope (33 examples) and exceptional tolerance of functional groups, high enantioselectivities, and excellent *E*—stereoselectivity (Scheme 45).



R^1 = C_6H_5, 4–MeC_6H_4, 4–*t*–BuC_6H_4, 4–$OAcC_6H_4$, 4–FC_6H_4, 4–ClC_6H_4, 4–BrC_6H_4, 4–$CF_3C_6H_4$, 4–$CO_2MeC_6H_4$, 4–CNC_6H_4, 2–MeC_6H_4, 3–MeC_6H_4, 2–napthyl, 5–(*tert*–butyl)–1*H*–indole–1–carboxylate);
R^2 = Ts, Cbz;
R^3 = C_6H_5, 4–MeC_6H_4, 4–$OMeC_6H_4$, 4–$OTBSC_6H_4$, 4–$OAcC_6H_4$, 4–$OCF_3C_6H_4$, 4–FC_6H_4, 4–ClC_6H_4, 4–$CF_3C_6H_4$, 4–(Bis(pinacolato)diboron)C_6H_4, 2–FC_6H_4, 3–FC_6H_4, 2–napthyl, 5–(3–OMe)pyridyl, 5–(2–OMe)pyrimidyl, cyclohexyl, $CH_2CH_2CO_2Ph$, TMS, CH=CH(Ph)

Scheme 45. Scope of nickel-catalyzed cross-electrophile couplings.

Recently (in 2024), the research group of Maiti [92] introduced C—Ni electrode-based electrolysis combined with photocatalysis. A three-component reaction assembled aryl diazoesters, acetone and 1,4—quinones/benzoquinone to afford ketal-functionalized quinones **55-56**. Ketal-functionalized quinones **55-56** were achieved in 64–86% and 72–79% yields, respectively. The electrolytic strategy configuration comprised two pairs of cathode-anode, in contrast to the traditional design, which includes a single pair. This has sustained a constant potential, generated maximum current and enhanced mass transport, thereby improving the overall efficiency of the reaction. Notably, the electron-rich singlet carbenes, generated by irradiating with a blue LED, may have rendered the vacant p-orbital less electrophilic, leading to a decreased yield (Scheme 46).

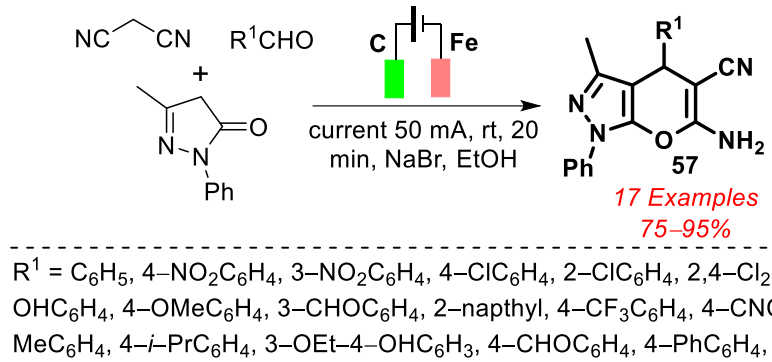


Scheme 46. Electrosynthesis of quinone via mono-ketal functionalization.

7.5. C—Fe-Based Electrode

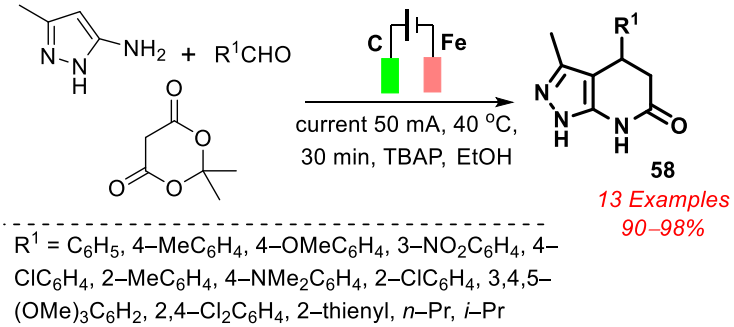
This section highlights the usefulness of C—Fe electrodes as a practical, sustainable and low-cost tool for the synthesis of various organic compounds. These systems are especially attractive for green chemistry as they employ abundant and cheap iron. Also, C—Fe electrodes are applicable to scalable methods. C—Fe electrodes worked well for one-pot multicomponent reactions that proceed rapidly under mild conditions with excellent yields.

In this direction, the research group of Vafajoo [93] in 2015 disclosed a multicomponent reaction introducing a C-—Fe electrode-based electrochemical cell. The synthesis of 1,4—dihydropyrano[2,3—*c*]pyrazole **57** was conducted via a one-pot reaction with malononitrile, aromatic aldehydes, and 3—methyl—1—phenyl—1*H*—pyrazol—5(4*H*)—one (Scheme 47). At room temperature and a steady current, the reaction took place in a single cell with an iron electrode as the cathode and a graphite electrode as the anode. The current method was a significant addition to the available conventional methods because it has a very rapid reaction time (20 min), a broad substrate scope (17 examples), high yields (75–95%), and an easy workup for the non-chromatographic purification of products. It is exciting to share that electron-withdrawing and electron-donating groups decorated starting substrates that reacted well without compromising the optimized reaction conditions.



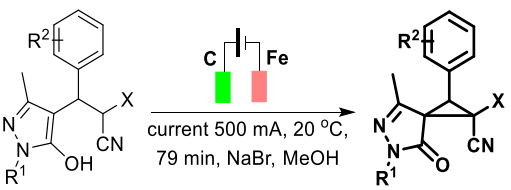
Scheme 47. Formation of dihydropyrano[2,3—*c*]pyrazoles.

Later, the research group of Jahangard [94] developed an electrochemical approach for the synthesis of tetrahydropyrazolo[3,4—*b*]pyridin-6-ones **58** (Scheme 48). One-pot three-component condensation of 5—methylpyrazol—3—amine, aromatic aldehyde and meldrum's acid in ethanol in an undivided cell (C—Fe) in the presence of tetrabutylammonium perchlorate as electrolyte. The final products (**58**) were obtained in 90–98% yields in about 30 min. It is interesting to note that aldehydes with electron-withdrawing and electron-donating substituents gave the products in high yields showing no electronic effects were observed in the reactions. Aliphatic aldehydes also gave good yields.



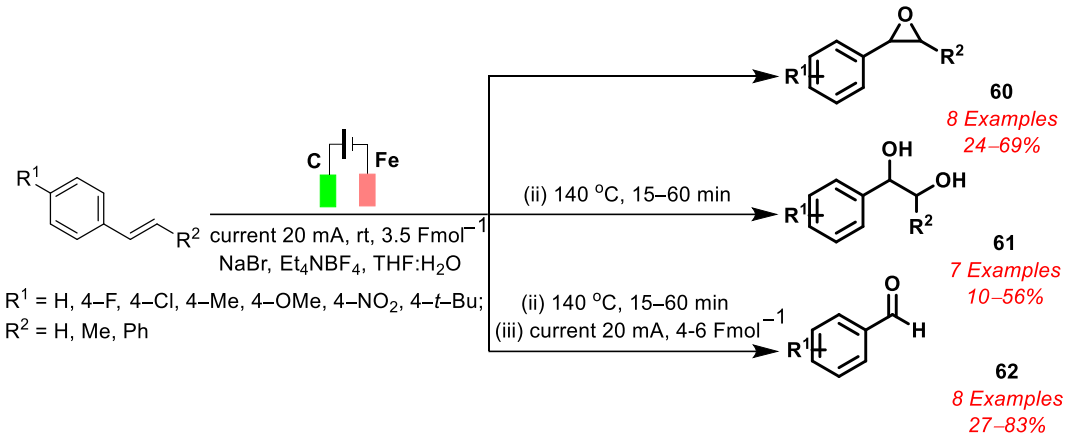
Scheme 48. Synthesis of tetrahydropyrazolo[3,4—*b*]pyridin—6—ones using C—Fe electrode.

In 2015, Vereshchagin et al. [95] reported a simple electrocatalytic system for one-pot cyclization of 3—(5—hydroxy—3—methylpyrazol—4—yl)—3—arylpropionitriles into substituted spiro—cyclopropyl-pyrazolones **59**. The electrocatalytic method allowed for a quick and easy one-step creation of substituted spiro-cyclo-propylpyrazolones with yields between 60% and 91%. The substrate scope was tested employing diversified spiro-cyclopropyl-pyrazolones to check the versatility of the protocol. In the electrocatalytic process, the stoichiometric amount of Br was substituted with catalytic amounts of sodium bromide as a mediator and the source of bromine generated at the anodes (Scheme 49).



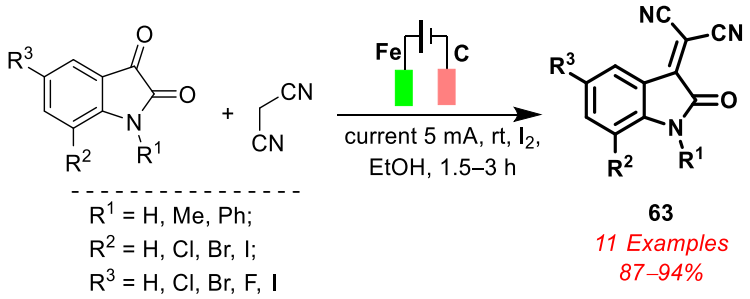
Scheme 49. Electrochemical cyclization to access spiro—cyclopropyl-pyrazolones.

In 2021, Cantillo and coworkers [96] developed a flexible electrochemical method for turning alkenes into epoxides, glycols, and aldehydes **60-62** through oxidation. NaBr accomplishes diol C—C bond breakage, ring opening and electrochemical epoxidation all in one pot. The epoxidation was carried out with water as the oxygen source and NaBr as a redox mediator. Additionally, the substrate scope was investigated in each transformation performed by the authors. The approach has been explored on a variety of functionalized terminal and non-terminal olefins (Scheme 50).



Scheme 50. Electrochemical strategy for the functionalization of alkenes.

In 2023, dicyano-isatylidene malononitrile derivatives **63** were prepared by a molecular-iodine-catalyzed electro-organic production (Sagar et al. [97]). The dicyano—2—(2—oxoindolin—3—ylidene)malononitrile compounds were produced in 87–94% yields at room temperature (Scheme 51). This method was performed with a fast reaction time and was tolerant of a variety of EWG and EDG in the low redox potential region of the Fe—C electrode. C=C bonds were formed at ambient temperature with high atom economy. Interestingly, the electron-donating nature of the methyl group reduced the reduction potential and decreased the product formation time by approximately 50 mV and 25 min. This may result from a reduction in electron density attributable to the pronounced electron-withdrawing nature of the phenyl group. This modulation influences the electrophilicity of C—3, resulting in a reduced reduction potential and an observed acceleration in the reaction rate. Figure 14 depicts the possible reaction mechanism pathway.



Scheme 51. Development of dicyano—2—(2—oxoindolin—3—ylidene) malononitriles using C—Fe electrode.

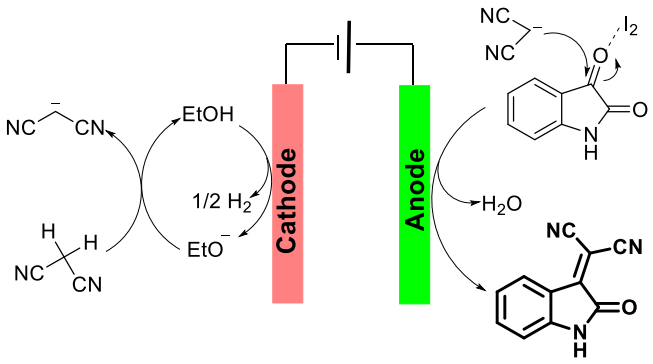
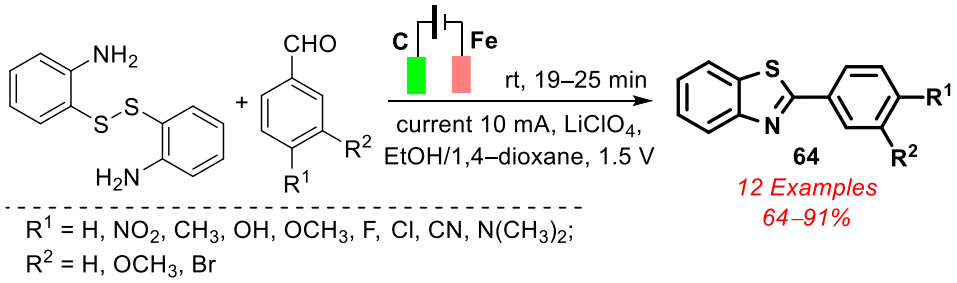


Figure 14. Plausible mechanistic study.

After investigating C—Pt electrode role, Shivhare's research group [98] reported the use of C—Fe electrode in the synthesis of benzothiazole derivative **64** (Scheme 52). The synthesis of 2—substituted benzothiazoles **64** in 64–91% yields were accomplished by combining bis(2—aminophenyl)disulfides with aromatic aldehydes in the presence of electrodes. *p*-—Nitro benzaldehyde was introduced as an aldehyde derivative to see the influence of the electron-withdrawing group on our reaction conditions; nevertheless, the yield was rather comparable to that of benzaldehyde. Conversely, aldehyde derivatives possessing electron-donating substituents at the para or meta positions relative to the aldehydic functional group exhibited a reduction in product yield. This reduction in product yield could be attributed to increased steric hindrance effects of the substituents. Lithium perchlorate is a good electrolyte for the current approach since it can handle a wide range of functional groups and works very well.



Scheme 52. Synthesis of 2-phenyl benzothiazole derivatives.

Initially, 2-aminothiophenol (**II**) is generated through the reduction in bis(2—aminophenyl)disulfide (**I**) using H^+ ions. The resultant compound (**II**), formed by the cleavage of S—S bonds in bis(2—aminophenyl)disulfide (**I**), subsequently reacts with benzaldehyde to yield an intermediate (**III**). Subsequently, intermediate (**III**) exhibited intramolecular heterocyclic ring formation, yielding 2—phenylbenzothiazoline (**IV**). The complete electrolysis process transpired in an open configuration in the presence of air. 2—phenylbenzothiazoline (**IV**) was subsequently oxidized in air to provide our stable aromatic heterocyclic product (Figure 15).

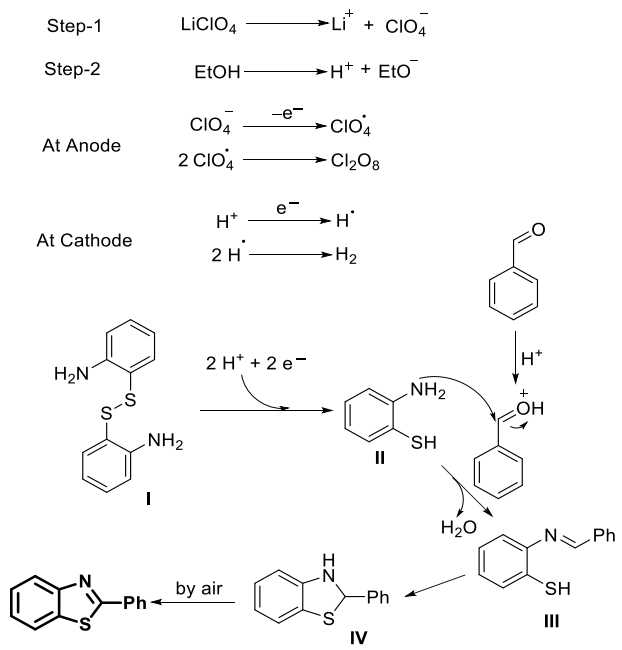
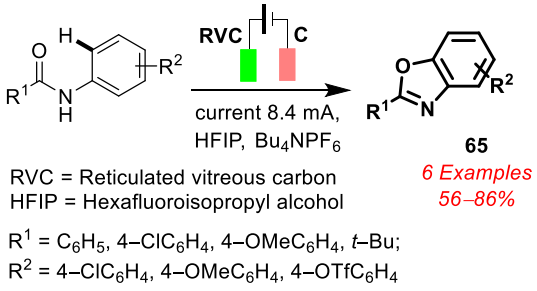


Figure 15. Tentative reaction mechanism.

7.6. C—Hybrid Material-Based Electrode

Under this section, the advanced C—hybrid material-based electrodes are highlighted. The combination of carbon supports with functional modifiers such as metals (Co), nanoparticles (Cu), and chiral organic molecules (L—proline, L—Hypip) gives rise to enhanced activity, selectivity, and reusability of these systems. Furthermore, the high yields of products, scalability and excellent stereochemical control demonstrate that C—hybrid electrodes are leading the way in green synthesis, despite the possibility of long-term stability issues. Due to their various features, several research groups explored these electrodes and successfully used them in complex reactions, like [4+2] annulations for the synthesis of sultams, and enantioselective Henry reactions and cyanations for the preparation of chiral alcohols and nitriles.

In the next year, Waldvogel et al. [99] devised a sustainable and simple electrochemical synthesis that enables the direct synthesis of benzoxazoles **65** from readily available anilides. The cyclized benzoxazoles **65** were achieved in 56–86% yields in six examples. The reaction tolerated a diverse array of functional groups, which are well-positioned to facilitate additional synthetic manipulation of the products **65**. This approach offered a green alternative to traditional methods by using ordinary electrode materials and a simple constant current protocol (Scheme 53).



Scheme 53. Electrochemical synthesis of benzoxazoles.

Deprotonation of the amide (**I**) by in situ formed alcoholate anions (**II**) generated by HFIP at the cathode can facilitate the first oxidation step. An interaction is made be-

tween the ortho carbon of the aromatic ring and the amidic oxygen to generate a heterocyclic radical (**III**). Further oxidation, coupled to proton loss, completes product formation. The process may include a secondary oxidation before cyclization and then an electrocyclic oxa—Nazarov—type approach (Figure 16).

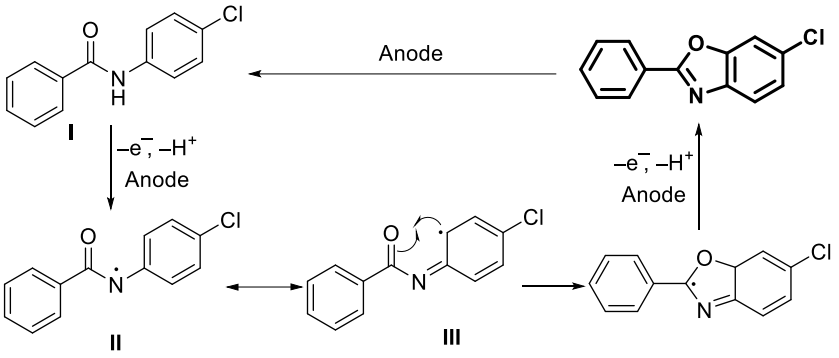
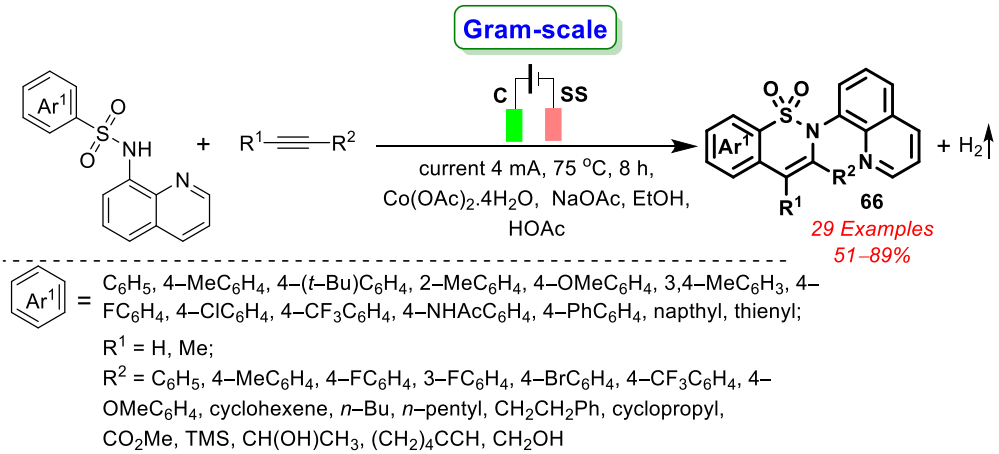


Figure 16. Proposed reaction mechanism.

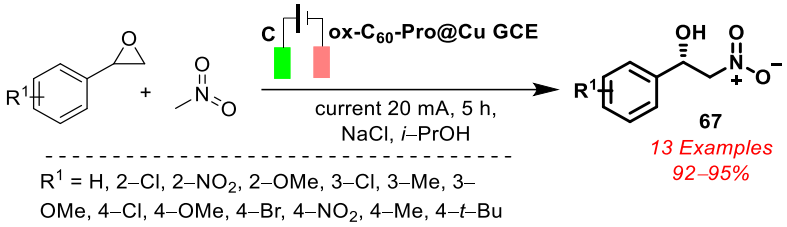
In 2020, Lei and their coworkers [100] focused on a cobalt-catalyzed electrochemical [4+2] annulation consisting of sulfonamides and alkynes (Scheme 54). Sultams **66** as final products were afforded by C—SS electrode-based electrolytic methodology through the evolution of hydrogen gas. The recovery of the cobalt catalyst was achieved by employing anodic oxidation in an undivided cell. The phenylacetylenes yielded the respective annulation products in 73–80% yields. Phenylacetylenes containing electron-withdrawing substituents (4—F, 3—F, 4—Br, and 4—CF_3) were effective substrates, yielding the corresponding [4+2] annulation products with yields ranging from 73 to 81%. It is important to highlight that reactive functional groups including C—C double bonds, C—C triple bonds, cyclopropyl moieties, esters, TMS, and hydroxyl groups were all well tolerated in the reaction.



Scheme 54. Scope of sultams synthesis through electrochemical [4+2] annulation.

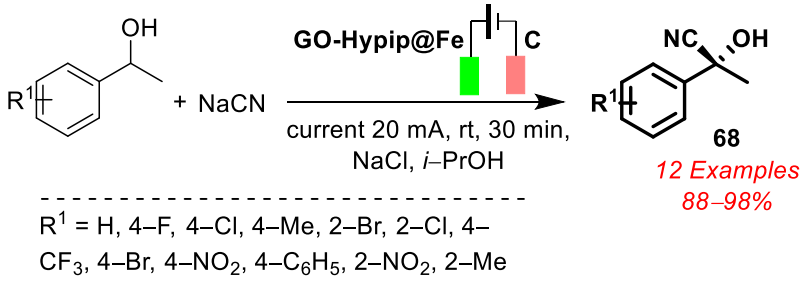
Recently (in 2024), Almehizia et al. [101] employed C—hybrid material-based electrode in chiral electro-organic synthesis of (*S*)—2-nitro—1—phenylethanol derivatives **67**. The electrode was fabricated from fullerene coated with copper nanoparticles and modified with L—proline (ox-C_{60}-Pro@Cu). The formation of final products **67** were achieved by the reaction of substituted 2—phenyloxiranes and nitromethane. Henry reaction delivered 13 examples (**67**) in 92–95% yields to prove the versatility of the protocol. The use of NaCl as an electrolyte significantly strengthens the present electrochemi-

cal process. It is interesting to share that the electrode proved good repeatability for many cycles, however corrosion properties were noticed after long time use (Scheme 55).



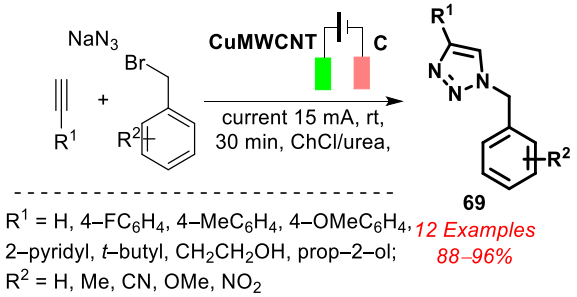
Scheme 55. Scope of electro-organic synthesis of (*S*)—2—nitro—phenylethanols.

More recently (in 2025), Alqaraguly's research team [102] investigated another C-hybrid electrode-based cell for enantioselective electro-organic synthesis of modified derivatives of chiral alcohols as (*R*)—2—hydroxy—2—phenylpropanenitriles **68**. GO-Hypip@Fe electrode was composed of graphene oxide (GO) that has been modified with (2*S*, 3*R*)—3—hydroxypiperidine-2-carboxylic acid (L—Hypip) as a cost-effective enantioselective organic linker. The reaction of substituted benzyl alcohols with sodium cyanide yielded (*R*)—2—hydroxy—2—phenylpropanenitriles **68** in 88–98% yields within 30 min only. Pleasingly, the electrochemical reaction rate was increased using NaCl that is both readily accessible and cost-effective (Scheme 56).



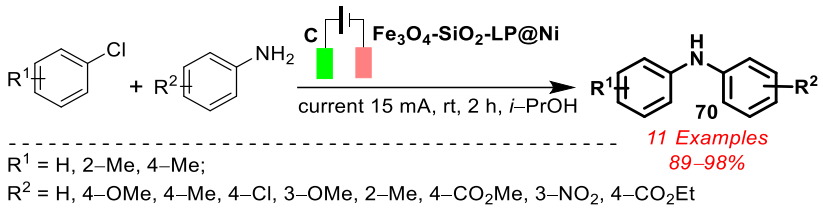
Scheme 56. Construction of (*R*)-2-hydroxy-2-phenylpropanenitriles.

In the same year, Almehizia et al. [103] reported a copper-doped electrode on MWCNTs. After that this electrode was disclosed as a catalyst at the electrode surface in the reaction of substituted phenylacetylenes, various benzyl bromides and sodium azide. Accordingly, 1,2,3—triazoles **69** were achieved in 88–96% yields at room temperature within 30 min only. The reusability of the Cu@ox-MWCNT-NH_2 SPE cathode in the reaction was evaluated over 14 cycles. The synthesized electrode had remarkable efficiency over the initial 13 cycles; however, a substantial decline was noted in the 14th cycle, resulting in an 85% reduction in reaction efficiency (Scheme 57).



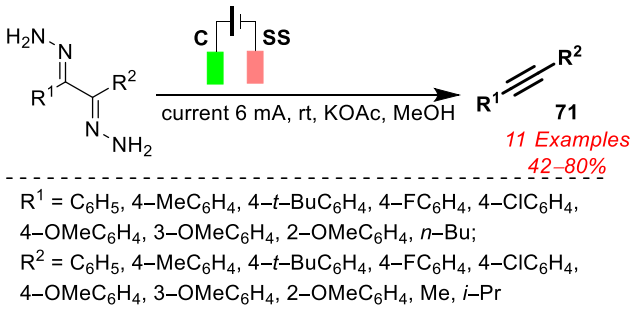
Scheme 57. Synthesis of 1,2,3-triazole derivatives using electro-organic synthesis.

Herein, Mustafa et al. [104] designed a novel magnetic electrode that incorporates Fe_3O_4-SiO_2 coated with nickel (Ni) nanoparticles (NPs) and modified with L—proline. Buchwald–Hartwig amination reactions assembled anilines and chlorobenzenes to access diphenylamine derivatives **70**. The C—N bond-forming methodology explored *i*—PrOH as the solvent, over a duration of 2 h at a steady current of 15 mA. The catalyst's reusability was assessed via a simulated reaction between aniline and chlorobenzene. Following 12 consecutive runs, the reaction yield diminished from 96% to 90%, signifying a marginal decline in efficiency. This minor decrease highlighted the resilience of the engineered electrode, which consistently exhibited excellent performance despite numerous cycles. Surprisingly, the findings demonstrate that electron-donating groups yielded superior results compared to electron-withdrawing groups (Scheme 58).



Scheme 58. Electro—organic reaction towards the formation of diphenylamines.

Later on, Waldvogel and coworkers [105] disclosed a sustainable and scalable electrosynthetic protocol for the development of alkynes **71**. A one-component reaction was accomplished using dihydrazones as the starting materials in the presence of KOAc as a supporting electrolyte. The protocol functioned under galvanostatic conditions in a commercially available undivided cell (C—SS cell), employing reusable carbon-based electrodes. Disubstituted alkynes **71** were afforded in 42–80% yields across 11 examples at room temperature. Surprisingly, electron-donating substituents on either the anilide or amide part of the substrate had a valuable consequence for the yield of the product. Furthermore, electrode reusability tests and scalability highlight the robustness and practical applicability of the protocol (Scheme 59).



Scheme 59. Electrochemical conversion of dihydrazones to alkynes.

Initially, oxidation of intermediate (**I**) resulted in the removal of two hydrogens. Intermediate (**II**), an alkene-type diradical, is observed at the surface of anode. Thereafter, the oxidation of the second N—H bond in intermediate (**II**) led to the creation of intermediate (**III**). The subsequent steps involve N_2 extrusion and radical–radical recombination to yield the alkyne. At the cathodic location, hydrogen gas is generated as a non-invasive and effective counter-reaction (Figure 17).

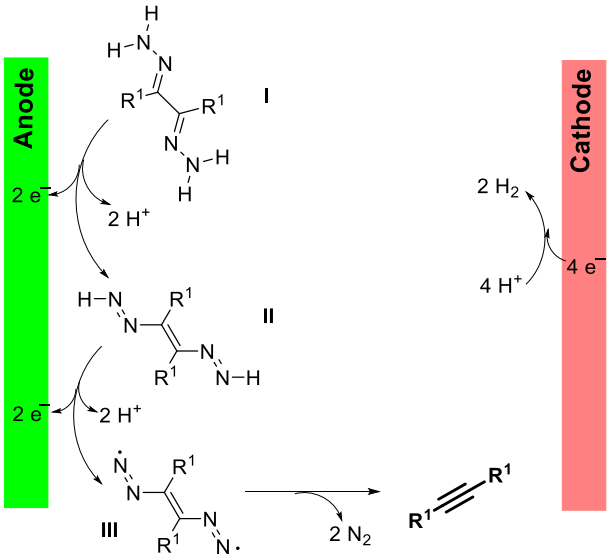
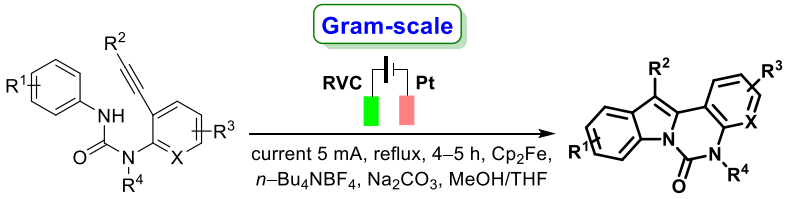


Figure 17. Tentative catalytic cycle.

7.7. RVC—Pt-Based Electrode

This section deals with the application of reticulated vitreous carbon and platinum electrodes (RVC—Pt) as a very effective and versatile platform for a variety of electro-organic syntheses. The special porous 3D structure of RVC enhances mass transfer and provides a large surface area, leading to highly efficient electrodes. Additionally, the RVC—Pt electrode system exhibits high chemo- and regioselectivity, broad substrate scope and sustainability making it a powerful tool for the construction of complex molecular architectures. The reports highlight their utility in a broad range of complex transformations such as redox-relay couplings for indole formation, C—N bond forming cyclizations for phenanthridines, cascade radical cyclizations for N—doped polyaromatics, and various C—H functionalization/annulation reactions.

In this context, in 2016, Xu's research team [106] developed an electrochemical approach that enabled the effective coupling of aryl/hetero-arylamines with tethered alkynes, allowing the highly chemo- and regioselective synthesis of indoles and azaindoles **72**. This strategy offers a new electrochemical method for the coupling of (hetero)arylamines with tethered alkynes to produce highly functionalized indoles and azaindoles **72**, without the use of precious metals or oxidants. The method utilized the cost-effective [Cp_2Fe] as the redox relay reagent, thus enabling H_2 liberation and the removal of noble metal catalysts and external oxidants (Scheme 60).

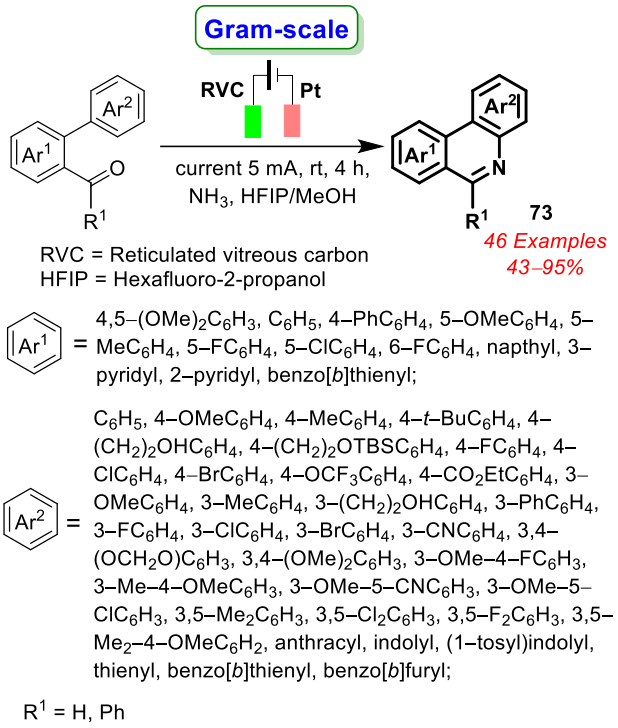


X = CH, N;
R^1 = H, 4–Me, 4–OMe, 4–F, 4–Cl, 4–Br, 4–I, 4–CN, 4–CF_3, 4–COMe, 6–OMe, 3–OMe, 4–F–6–Me, 3,6–$(OMe)_2$–4–Cl, 3,5–Cl_2–4,6–F_2;
R^2 = C_6H_5, 4–$OMeC_6H_4$, 4–BrC_6H_4, 4–$CHOC_6H_4$, 3–FC_6H_4, 5–(2–CO_2Me)furyl, cyclohexenyl, cyclohexyl, *n*–Bu, H, CH_2CH_2OH, CH_2CH_2NHTs, $CH_2(CH_2)_2OCONHPh$, $CH_2CH(NHBoc)CO_2Me$, $CH_2CH_2OCO(t–Bu)$, $CH_2CH(NHCOCH(NHBoc)i–Pr)CO_2Me$;
R^3 = H, 4–Me, 4–F, 4–Br;
R^4 = Me, Bn, *n*–Bu

72
35 Examples
43–94%

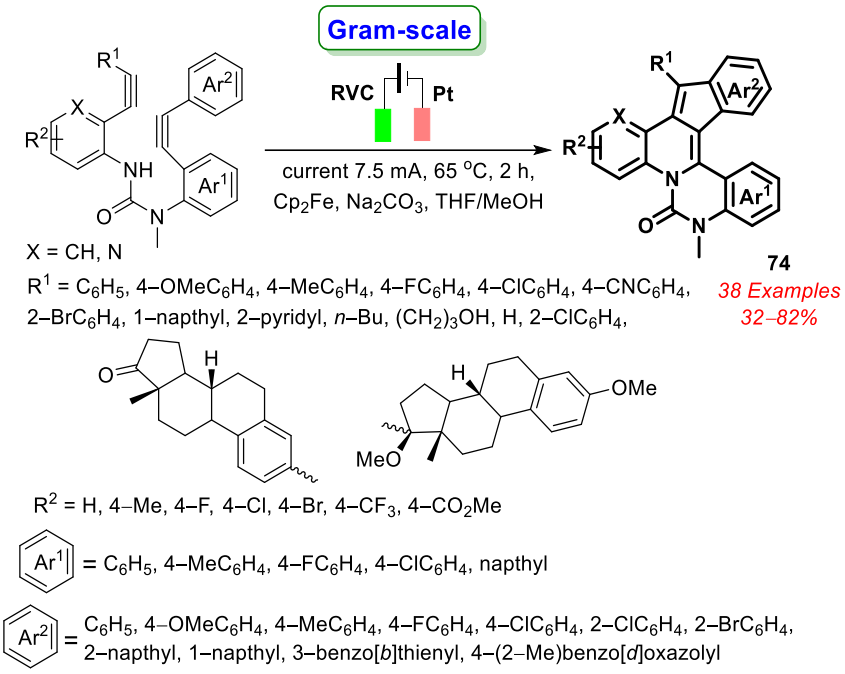
Scheme 60. Scope of indole synthesis.

In 2017, Xu et al. [107] also disclosed the synthesis of *N*—heteroaromatics from NH_3 and biaryl aldehydes via a reagent-free N—H/C—H cross-coupling. The electrosynthesis introduced NH_3 as a cost-effective and atom-efficient nitrogen donor and did not necessitate oxidizing agents. The current approach facilitated the regioselective and efficient access to a diverse array of phenanthridines **73**. Biaryl ketones exhibited a lack of reactivity in our electrochemical process, perhaps due to their diminished tendency for imine production relative to aldehydes (Scheme 61).



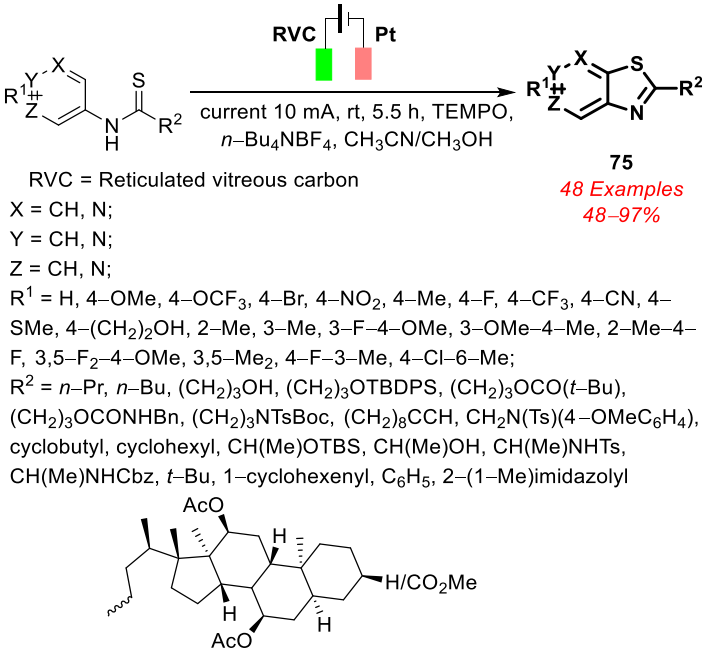
Scheme 61. Synthesis of phenanthridines through C—N bond forming cyclization.

In the same year, Xu and co-workers [108] also described an electrochemical cascade cyclization procedure that allows for the facile synthesis of nitrogen-doped polyaromatic compounds **74** from various substituted diamines. The use of Cp_2Fe as a mild and selective catalyst provides efficient cyclization products of polyaromatic hydrocarbons without overoxidation. Moreover, the introduction of ferrocene as a mild and selective redox catalyst facilitated the synthesis of a diverse array of electron-rich polycyclic aromatic hydrocarbons (helicene-like structures) **74**. The electrosynthetic method encompassed a remarkable amidyl radical cascade cyclisation process, enabling the formation of three rings in a singular operation. Conversely, the electrolysis reaction on a Pt- or graphite-based anode at various current densities led to a considerable reduction in the yield. The phenyl ring was compatible with the functional groups, including different electron—donating methyl groups, halogens and electron-withdrawing groups such as CF_3 and an ester. Likewise, the substitution of the alkyne with phenyl rings having different electronic properties, such as naphthyl, a pyridyl ring, alkyl groups, or even a hydrogen atom, did not indicate a significant effect on the product formation. The terminal phenyl ring C can be substituted by OMe, Me, F and Cl but not by the strongly electron-withdrawing CN, which led to monocyclization in the base-promoted alkyne hydroamidation. Simultaneously, *o*— or *p*—substituted phenyl rings were also possible to be added to both alkyne units (Scheme 62).



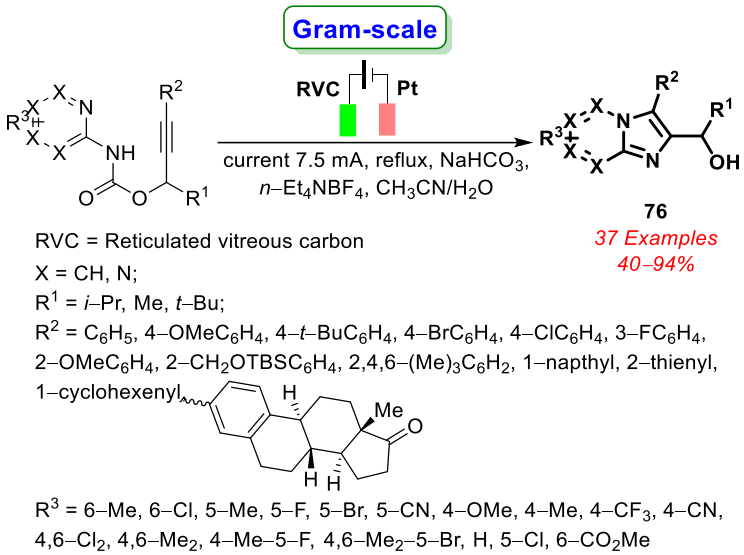
Scheme 62. Electrochemical cyclization leading to *N*—doped polycyclic aromatic hydrocarbons.

In the same year, Xu et al. [109] also synthesized benzothiazoles/thiazolopyridines **75** utilizing similar cell. It utilizes an electrolytic 2,2,6,6—tetramethylpiperidine—*N*—oxyl radical (TEMPO) to enable thiolation of the C—H bond. Final products **75** were synthesized with 48–97% yields. It is particularly important to retain the hydroxyl group, as TEMPO is a well-known redox catalyst for the electrochemical and chemical oxidation of alcohols. All electron-withdrawing (CN) and electron-donating (SMe, OMe and Me) substituents, halogens (F/Br) and Fluorine containing functions (OCF_3 and CF_3) were placed at para position and reacted easily. However, the highly electron-withdrawing group (nitro) caused the substrate to decompose and no product formation was detected (Scheme 63).



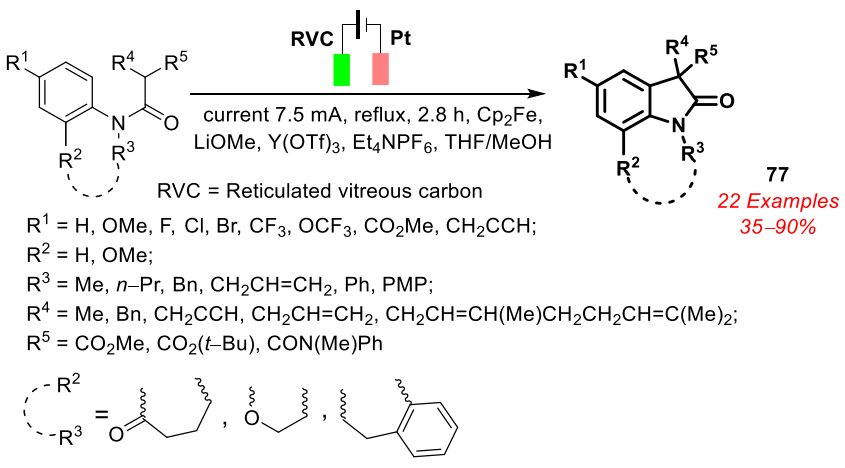
Scheme 63. C—H Thiolation via electro—organic method.

In the next year, Xu and coworkers [110] also developed an alternative way for the synthesis of several imidazo—fused *N*—heteroaromatic compounds **76**. Electrochemical [3+2] annulation reaction of tethered internal alkynes with heteroarylamines in a regioselective manner. The reaction was demonstrated to be compatible with *tert*—butyl/methyl at the propargylic position. Moreover, the substitution of the 2—aminopyridine moiety with other aminodiazine structures was demonstrated to be feasible. The electrosynthesis introduces a new tetraarylhydrazine as the catalyst, features eliminate the necessity for transition-metal catalysts and oxidizing agents and wide substrate range (Scheme 64).



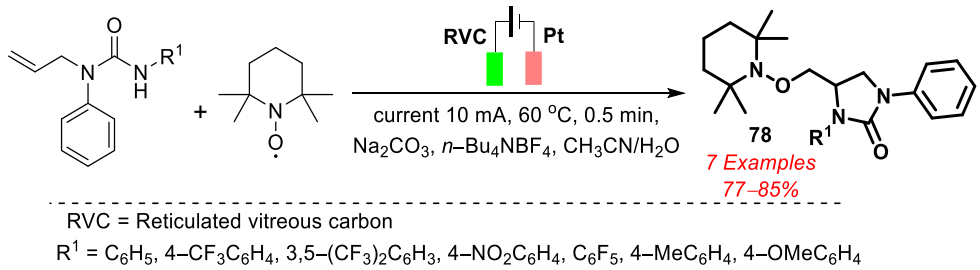
Scheme 64. Synthesis of imidazo—fused *N*—heteroaromatic derivatives.

In 2018, Wu's research team [111] performed an intramolecular C(sp^2)-H/C(sp^3)-H cross-coupling of carbonyl compounds via Cp_2Fe—catalyzed electrochemical oxidation. The dehydrogenative cyclization reactions occurred through the activation of the acidic *α*—C—H bond of the 1,3—dicarbonyl molecule to obtain a carbon radical. The synthesis of oxindole derivatives **77** were achieved in 35–90% yields within 2.8 h. Moreover, the substrate scope was also examined by the authors and it was proved that the protocol was well tolerated in the formation of 22 examples of oxindoles **77**. It was noted the diminished reaction yields for the electron-withdrawing substrates were likely attributable to the challenge of cyclizing the electrophilic C—radical onto the *N*—phenyl ring (Scheme 65).



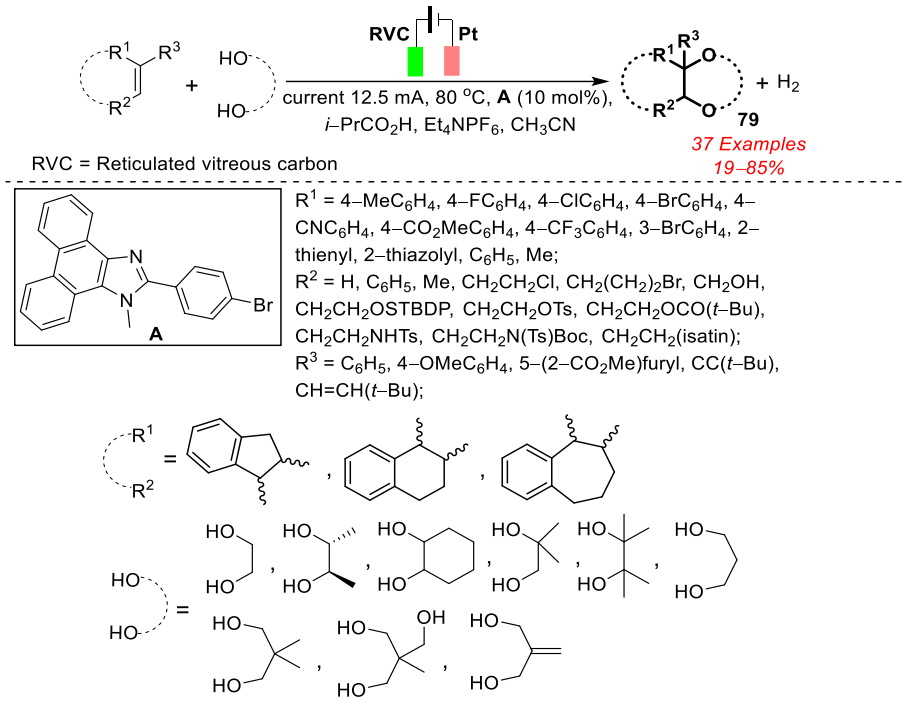
Scheme 65. Scope of oxindole synthesis.

In 2018, Khatoon et al. [112] devised a straightforward intramolecular amination of inactivated alkenes. Electrochemical-assisted difunctionalization of substituted alkenes provided access to a variety of imidazoline-based molecular hybrids **78**. During electrolysis, a nitrogen radical cyclized with the alkene, producing a terminal carbon radical that subsequently bonded with the 2,2,6,6—tetramethylpiperidine—*N*—oxyl radical (TEMPO). Surprisingly, TEMPO acts in a dual role as a redox facilitator and radical trap successfully. It is worth noting that the incorporation of tosyl and naphthyl groups, however, resulted in unsatisfactory outcomes, likely attributable to the decomposition of the product during electrolysis or the steric hindrance posed by the bulky tosyl and naphthyl groups (Scheme 66).



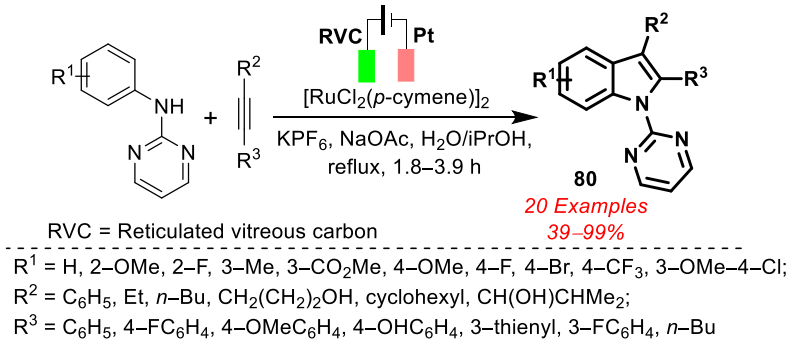
Scheme 66. Electrochemical difunctionalisation of alkenes.

Next (in 2018), the research group of Xu [113] investigated an electrochemical annulation procedure that allows alkenes and diols to be converted into 1,4—dioxane/1,4—dioxepane scaffolds **79** (Scheme 14). For this transformation, an RVC-Pt-based electrode worked well and released hydrogen gas. The methodology was accomplished by exploring an organic catalyst in combination with electrochemistry and redox catalysis that enabled electro-organic synthesis in the absence of transition metals and oxidizing reagents. Interestingly, functionalized O—heterocycles with tetrasubstituted carbon centers are easily accessible due to the reaction's high tolerance for di- and trisubstituted alkenes and compatibility with a broad range of starting substrates. Additionally, the reaction demonstrated extensive compatibility with several 1,1—diphenyl alkenes possessing substituents with variable electronic characteristics at the para- or meta-position of the benzene ring (Scheme 67).



Scheme 67. One—pot annulation reaction towards the formation of O—heterocycles.

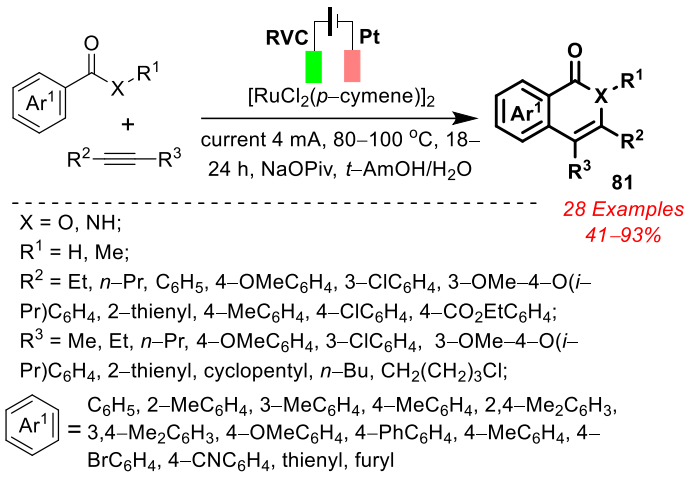
In the same year, an alternate approach was developed by Xu and coworkers [114] towards the formation of substituted indole **80** as a fused product. RVC—Pt electrode-based ruthenium-catalyzed electrochemical dehydrogenative annulation reaction of alkynes and anilines was performed by the authors (Scheme 68). As a result, the C-H activation reaction delivered final products **80** in high yields (39–99%). Interestingly, the active ruthenium-based catalyst was recycled, and the evolution of H_2 is facilitated by the use of electric current. The approach was completely compatible with a basic undivided RVC—Pt cell and may be introduced to prepare a wide variety of indole derivatives from diversified alkynes and aniline derivatives.



Scheme 68. Ru-catalyzed annulation by using RVC-Pt electrode.

In the following year, Ackermann's research team [115] discovered the first electrocatalytic C—H activation via weak O-—coordination (Scheme 69). Under this work, a multifunctional ruthenium(II) carboxylate catalyst was explored to facilitate electro-oxidative C—H/O—H functionalization for alkyne annulations without metal oxidants. This green strategy led to the formation of fused cyclic amide and cyclic lactone derivatives **81** in 41–93% yields. It is exciting to note that hydrogen was the only by-product of the C—H activation that used electricity as an oxidant. Consequently, the adaptable ruthenium catalysis system was suitable for both electron-deficient and elec-

tron-rich arenes. Electrophilic functional groups like bromo, cyano, and heterocyclic scaffolds were completely tolerated.



Scheme 69. C—H/O—H Alkyne annulation assisted by electrochemical methods.

Mechanistic investigations indicate a plausible catalytic cycle starting from simple organometallic C—H activation. Thus, ruthena(II)cycle **I** is formed with loss of two equivalents of carboxylic acid. Subsequently, migratory insertion of the alkyne generates the seven-membered ruthena(II)cycle **II**, which quickly undergoes reductive elimination to provide the ruthenium(0) sandwich complex **III**. The essential reoxidation of the ruthenium(0) complex is finally achieved via anodic oxidation (Figure 18).

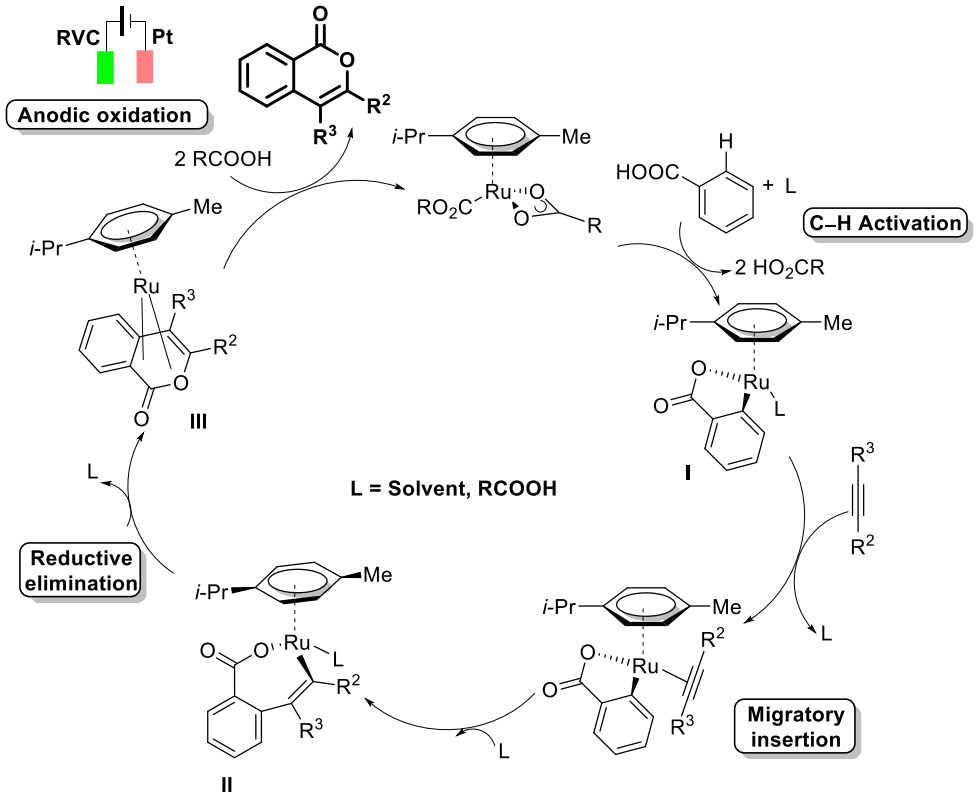
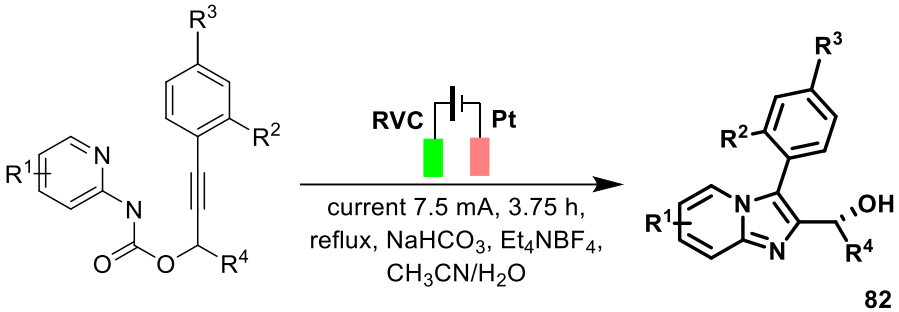


Figure 18. Proposed catalytic cycle.

In 2019, Xu et al. [116] established a diastereoselective strategy for synthesizing chiral biaryl—imidazopyridine conjugates **82**. The methodology occured via a radical

cyclization cascade to form biaryls with effective axial chirality transfer. The processes introduced readily combinable precursors and gave imidazopyridine based biaryls **82** with good to excellent yields (43–96%) and diastereoselectivity. Delightedly, the electro-organic synthesis was suitable for many functional groups, together with cyclic ketal/aryltrimethylsilane/silyl ether/ester, and further, all diastereomers were separated via flash column chromatography (Scheme 70).



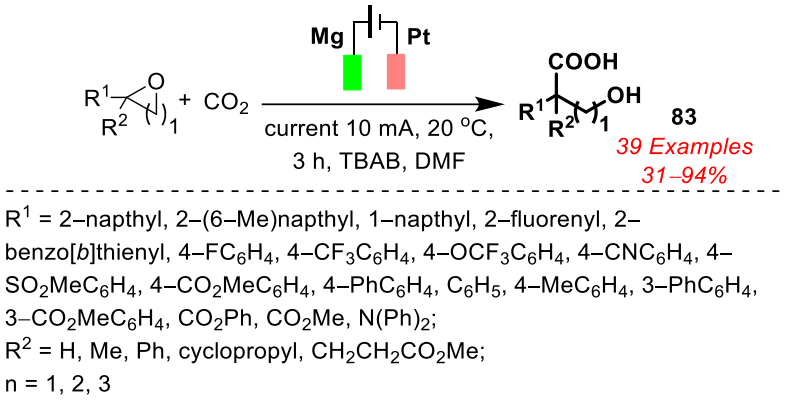
R^1 = 4–OMe, 4–Br, 4–CF_3, 5–CN, 6–Cl, 6–Me, 4,6–Cl_2, 4–Me–5–F;
R^2 = *t*–Bu, TMS, $C(Me)_2OTMS$, $C(Ph)_2OTMS$, CH(Cy)OTMS, $C(Me)_2CO_2Me$, CHMe(1,3–dioxolane);
R^3 = H, Me, OMe;
R^4 = *t*-Bu, $C(Me)_2Bn$, CHMe(Cy)

Scheme 70. Electrochemical synthesis of axially chiral biaryls.

7.8. Mg-Pt Based Electrode

This section is devoted to the unique and very useful application of Mg—Pt electrodes, especially when dealing with electrocarboxylation reactions. Although less explored, these systems show extraordinary efficiency in the reductive ring-opening and carboxylation of epoxides with carbon dioxide (CO_2). In addition to this, the broad substrate scope and scalability of this method point to its potential as a sustainable and efficient tool for CO_2 utilization in organic synthesis processes. It is important that a sacrificial magnesium anode be used. The summarized reports demonstrate the capability of this method to synthesize valuable β—hydroxy acids in excellent yields and regioselectivity under mild conditions.

In 2022, Zhang's research team [117] reported an electroreductive ring opening carboxylation of epoxides with CO_2 for preparing valuable β—hydroxy acids **83**. This reaction demonstrated a wide range of substrate applicability from styrene oxides to the practical and scalable access of β—hydroxy acids **83**. The Mg—Pt electrode-based electrochemical protocol efficiently worked in TBAB and DMF to afford products **83** with 31–94% yields. Epoxides with aryl substituents (benzothiophenyl/phenyl/fluorenyl/naphthalenyl) were appropriate substrates for this reaction, delivering comparable α—carboxylation products with exceptional regioselectivity. Disubstituted aryl epoxides with diverse electron-withdrawing groups functional groups (phenyl/halogen/esters/cyano//sulfones groups) could effectively engage in this reaction, yielding products **83** in good to excellent yields (Scheme 71).



Scheme 71. Scope of electrocarboxylation.

The ring-opening reaction was started by the single electron transfer from the cathode to the epoxides and the α—radical anion intermediate (**I**) was formed. Finally, the radical anion intermediate (**I**) was treated with CO_2 to give the *O*—carboxylated radical anion intermediate (**II**), which was rapidly transferred to a further single electron at the cathode to yield the α—carbanion intermediate (**III**). Subsequently, C-carboxylation with CO_2, either intermolecularly or by intramolecular attack of the carbanion on the carbonate moiety, would give the dicarboxylated intermediate (**IV**). At the anode, magnesium was oxidatively dissolved, producing magnesium ions, which acted as counterions to stabilize the carbonate and carboxylate intermediates. Finally, the hydroxy acid product was obtained by an acidic work—up (Figure 19).

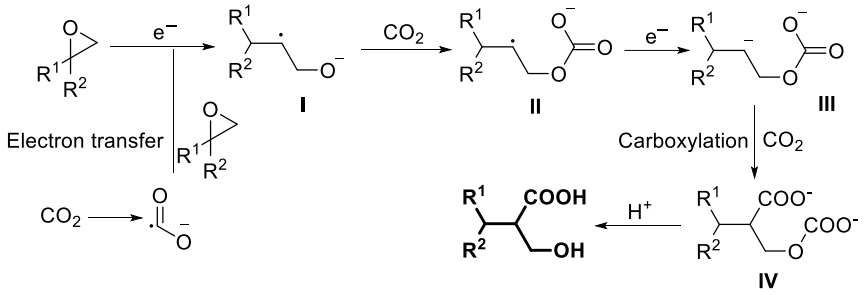
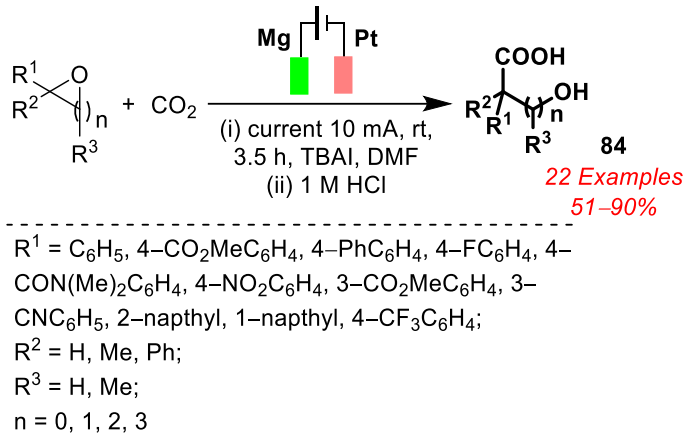


Figure 19. Possible reaction mechanism.

In order to explore the chemistry of β-—hydroxy acids, in the same year, Qiu et al. [118] also outlined an electrocarboxylation of aryl epoxides with CO_2 to afford valuable β—hydroxy acids **84**. This transformation proceeded smoothly using an Mg-Pt electrode under mild conditions, and had a broad substrate scope, as demonstrated by 22 examples in 51–90% yields. Aryl epoxides with *para*- or *meta*-substituents, along with simple styrene oxide, all functioned effectively to yield the intended products. Significantly, functional groups commonly susceptible to electroreductive environments, such as ester, nitro, cyano, and amide, were preserved, indicating a high degree of functional group compatibility. Moreover, β—hydroxy acids featuring all-carbon quaternary stereocenters were readily synthesized from highly hindered 1,1—disubstituted aryl epoxides, achieving significant chemo- and regioselectivity. Remarkably, this technique was not confined to styrene epoxides, as additional aryl-substituted cyclic ethers also performed effectively under the electroreductive conditions. Interestingly, aryl epoxides with para/meta-substituents, along with styrene oxide, all proceeded effectively to give the required products **84** (Scheme 72).

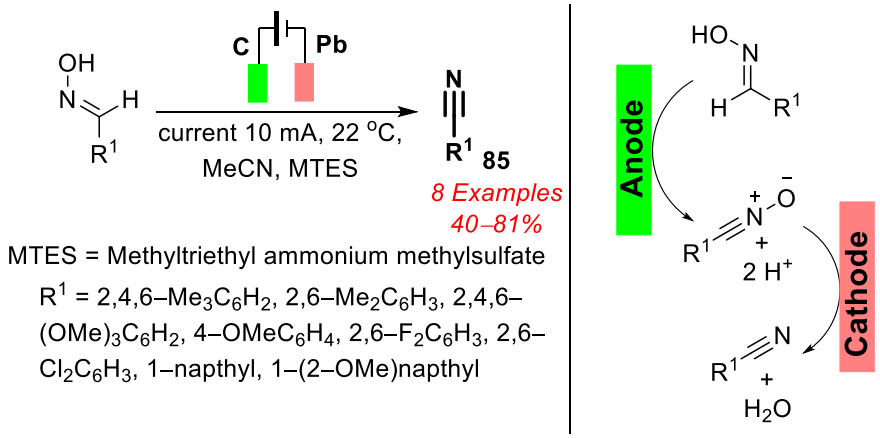


Scheme 72. Electro—organic reaction of epoxides.

7.9. Miscellaneous

In this last section, we illustrate the wide adaptability of electro—organic synthesis by summarizing the transformative reactions performed with a variety of other electrode systems. These reports demonstrate the versatility of electrochemistry with inexpensive materials such as Pb and GF as well as with combined systems such as Pt—Fe. The above-mentioned electrodes reflect a common theme that electricity can be used as a green oxidant under mild conditions to achieve moderate to high yields over a broad range of substrates, further establishing electro—synthesis as a powerful and sustainable methodology in modern chemistry. Diverse transformations are encountered, ranging from halogen—free conversion of oximes to nitriles, multicomponent cyclizations to xanthenes and phthalazines, to selective C—H oxidation.

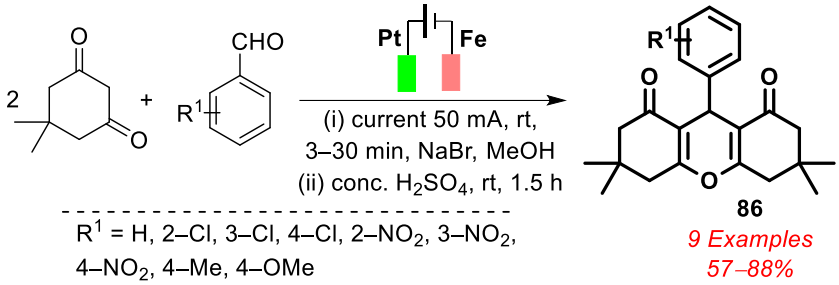
In 2015, the research group of Waldvogel [119] established a straightforward, halogen-free methodology for synthesizing nitriles **85** from oximes, achieving yields of up to 81% through the utilization of cost-effective and easily accessible electrode materials like lead and graphite. This protocol was relevant for various starting substrates without compromising the reaction conditions. The current organic transformations delivered eight examples of substituted nitrile derivatives in 40–81% yields. Also, the mechanistic insights are depicted in Scheme 73.



Scheme 73. Conversion of aldoxime to nitrile and its mechanistic insights.

In 2015, Kefayati et al. [120] delineated a two-step electro—organic synthesis by the application of a Pt—Fe-based electrochemical system (Scheme 74). At the beginning, the generation of 2,2—arylmethylene bis(3—hydroxy—5,5—dimethyl—2—cyclohexene—1—one) was observed by the reac-

tion of aromatic aldehydes and dimedone using electrogenerated base (EGB) and sodium bromide as an electrolyte. Next, the cyclization was facilitated at room temperature in the presence of sulfuric acid to access 1,8—dioxo—octahydroxanthene scaffolds **86**. Interestingly, aromatic aldehydes having electron-withdrawing groups reacted very well compared with aromatic aldehydes having electron-releasing groups. Additionally, this method has several additional advantages, like the one-pot reaction with good to excellent yields, use of inexpensive reagents and easy setup, (57–88%), and in situ base generation without the use of hazardous chemicals.



Scheme 74. Formation of 1,8—dioxo—octahydroxantheness by using Pt—Fe electrode.

In the initial step of a direct or indirect cathodic process, the methoxide anion is formed by deprotonation of methanol at the cathode. The methoxide anion then reacts with dimedone in solution to give the dimedone anion. Then, the step involves the Knoevenagel condensation of the dimedone anion with the aromatic aldehyde involving the elimination of a hydroxide anion and the production of the Knoevenagel adduct **I**. This is followed by the Michael addition of the second dimedone anion to the Knoevenagel adduct I to give intermediate **II**. The synthesis of the corresponding 2,2—aryl—methylene bis(3—hydroxy—5,5—dimethyl—2—cyclohexene—1—one) is cyclized and converted into products. Intermediate **III** is subsequently cyclized using few drops of concentrated H_2SO_4 at room temperature to provide the expected 1,8—dioxo—octahydroxanthene (Figure 20).

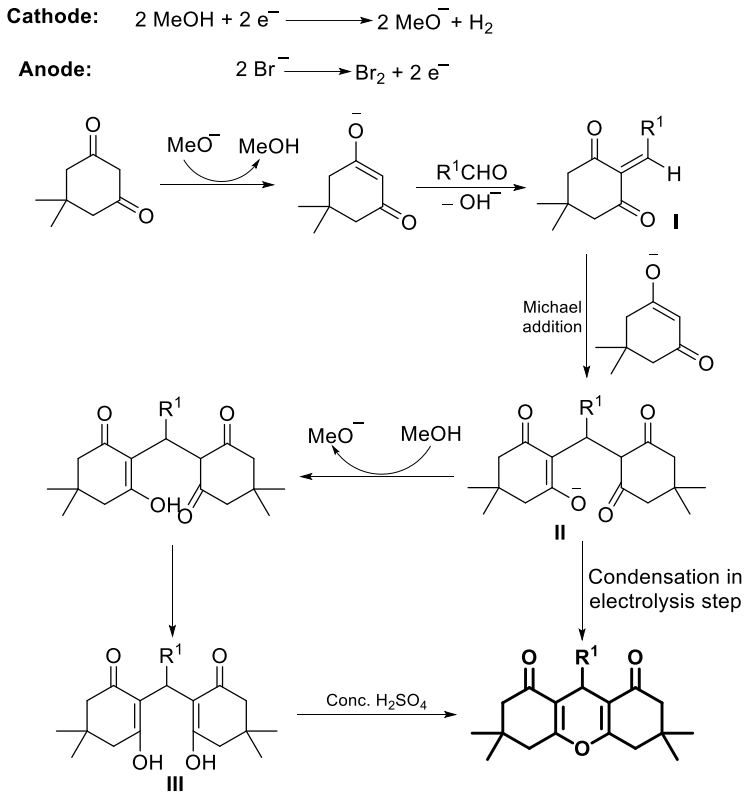
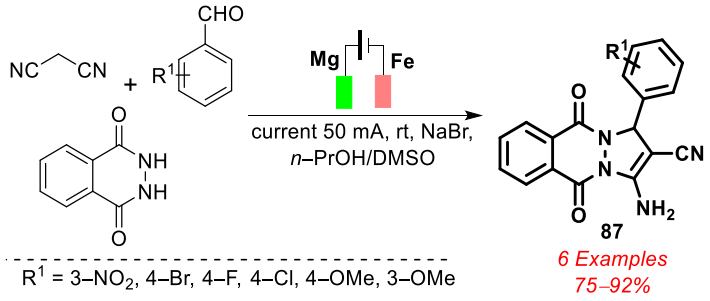


Figure 20. Possible reaction mechanism.

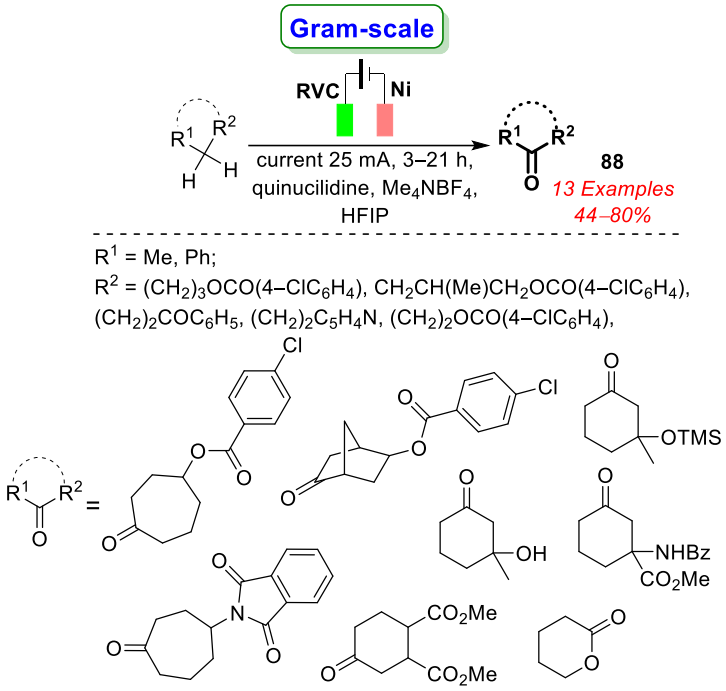
In the same year, Mohammadi and coworkers [121] developed a multicomponent method towards the synthesis of nanosized phthalazine particles (Scheme 75). The method was based on a one-pot multicomponent reaction (MCR) of aldehydes, phthalhydrazide and malononitrile in propanol, with undivided Mg—Fe cells and NaBr as an electrolyte. A total of six examples **87** were obtained in 75–92% yields to check the substrate compatibility employing electron-withdrawing and electron-donating-group-decorated starting substrates.



Scheme 75. Multicomponent formation of phthalazines by using Mg—Fe electrode.

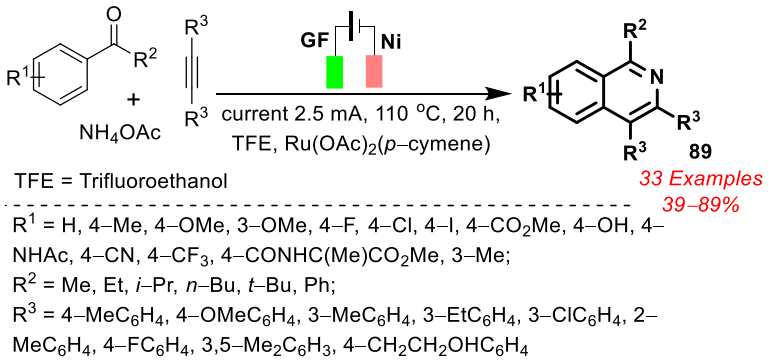
In 2017, Baran et al. [122] introduced an effective RVC—Ni electrode-based electrochemical strategy for the oxidation of inactivated C—H bonds. The present reaction employed a straightforward redox mediator, quinuclidine, alongside cost-effective carbon and nickel electrodes to achieve the selective functionalization of methylene moieties. The products, as ketones **88,** were afforded in 44–80% yields within 3–21 h. It is delight-

ful to share that diversified functional groups were revealed to be well-suited to the chemoselective process. Moreover, the procedure demonstrates gram-scale compatibility for industrial practicality (Scheme 76).



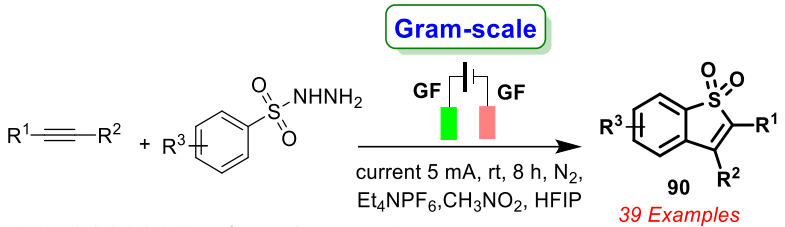
Scheme 76. Electrochemical quinuclidine-mediated C—H oxidation.

Next (in 2021), Ackermann and coworkers [123] outlined a three-component electrochemical domino-assembly of isoquinolines **89** using a new Gf—Ni-based electrode**.** The methodology generated electricity as the exclusive oxidant and produced solely H_2 as a by-product (Scheme 77). A ruthenaelectro-catalyzed C—H/N—H functionalization approach was performed between aromatic ketones, substituted alkynes, and ammonium acetate in the presence of a Gf—Ni electrode. Significantly, a variety of functional groups, such as halogens, ester, amide, acetoxyl, and cyano groups, were accommodated underscoring a considerable potential for subsequent late-stage diversification. The site—selectivity for meta-substituted ketones was influenced by steric hindrance, whereas meta-methoxy—substituted acetophenone yielded the less hindered derivative preferentially. Electron-rich or electron-poor alkynes effectively yielded the intended compounds, but electron-rich alkynes demonstrated a somewhat elevated inherent reactivity.



Scheme 77. Ru—catalyzed three-component construction of isoquinolines.

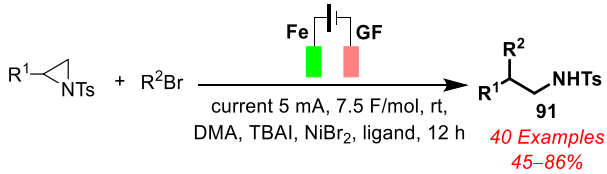
Further (in 2022), Zhang and coworkers [124] developed a Gf—Gf electrode-based electrochemical strategy towards the construction of benzo[*b*]thiophene—1,1—dioxides **90** (Scheme 78). Initially, the generation of a quaternary spirocyclization intermediate takes place through selective *ipso*—addition rather than an *ortho*—attack to facilitate sulfur migration that next leads to the formation of final products **90**. Additionally, the reaction was accomplished at room temperature in undivided cells via constant current electrolysis. The protocol eliminated the need for transition-metal catalysts or stoichiometric quantities of oxidants while accommodating various functional groups.



R^1 = Me, Et, *n*–Pr, *n*–Bu, pentyl, hexyl, hepthyl, octyl, nonyl, decyl, dodecyl, *i*–Pr, *i*–Bu, *t*–Bu, cyclopropyl, CH_2CH_2OTs, $CH_2(CH_2)_3OTs$, $CH_2CH_2CH_2$(1–isoindoline–1,3–dione), $CH_2CH_2CH_2OCOC_6H_5$, C_6H_5, 4–MeC_6H_4, 4–EtC_6H_4, 4–*t*–BuC_6H_4, 4–FC_6H_4, 4–ClC_6H_4, 4–BrC_6H_4, 3–MeC_6H_4;
R^2 = C_6H_5, 4–MeC_6H_4, 4–EtC_6H_4, 4–*t*–BuC_6H_4, 4–FC_6H_4, 4–ClC_6H_4, 4–BrC_6H_4, 3–MeC_6H_4;
R^3 = 3–Et, 3–(*n*–Pr), 3–cyclopropyl, 3–(*t*–Bu), 2–Me, 3–OMe, 4–Me

Scheme 78. An ipso-attack pathway for the synthesis of benzo[*b*]thiophene—1,1—dioxides.

In 2023, Qiu's research team [125] reported a nickel-electrocatalyzed cross-electrophile coupling of readily available aryl bromides and aryl aziridines under mild electrochemical conditions to access synthetically useful *β*—arylethylamines **91**. By using an Fe—Gf electrode, *β*—arylethylamines were obtained in 45–86% yields at room temperature. This strategy is featured by its exquisite chemo- and regioselectivity, broad substrate scope (40 examples) and good functional group compatibility. This strategy could also enable the electroreductive ring-opening cross-coupling of aryl aziridines with CO_2 to access *β*—amino acids. A range of functional groups, comprising electron-donating and electron-withdrawing groups at the meta or para positions of the benzene ring, were effectively accommodated, yielding the necessary cross-coupling products in moderate to good yields (47–86%). Subsequently, many difunctional *N*—tosyl aziridines were evaluated and demonstrated efficacy in this transition (Scheme 79).



R^1 = 4–$CF_3C_6H_4$, 4–$CO_2MeC_6H_4$, 4–FC_6H_4, 4–ClC_6H_4, 4–BrC_6H_4, 4–PhC_6H_4, 4–MeC_6H_4, 4–*t*–BuC_6H_4, 3–MeC_6H_4, 3–FC_6H_4, 3–ClC_6H_4, 3,4–$Me_2C_6H_3$, 3–F–4–ClC_6H_3, 2,4,5–$Me_3C_6H_2$, 1–napthyl, 2–napthyl;
R^2 = 4–$CO_2MeC_6H_4$, 4–$CO_2AcC_6H_4$, 4–CNC_6H_4, 4–$COMeC_6H_4$, 4–$CF_3C_6H_4$, 4–PhC_6H_4, 4–FC_6H_4, 4–ClC_6H_4, 4–$CHOC_6H_4$, 4–$OMeC_6H_4$, 4–$NMe_2C_6H_4$, 4–*t*–BuC_6H_4, C_6H_5, 3–$COMeC_6H_4$, 1–napthyl, 2–napthyl, 6–benzo[*b*]thienyl, 6–benzo[*b*]furyl, 6–(2,3–dihydrobenzofuryl), 5–(2–CF_3)pyridyl, 2–dibenzofuryl, 2–dibenzothienyl, 4–(9–carbazole)C_6H_5, 4–$(COC_4H_8NO)C_6H_5$

Scheme 79. Electro reductively driven cross—electrophile coupling.

Initially, Fe(0) undergoes oxidation to become Fe(II) at the anode. At the cathode, the oxidative addition of Ni(0)L (**I**) to aryl bromide yielded the Ar—Ni(II)L—Br intermediate (**II**). Further, a SET reduction in the aziridines at the cathode generated the benzyl radical, which was swiftly captured by intermediate (**II**), leading to the formation of intermediate (**III**). Thereafter, reductive elimination of intermediate (**IV**) afforded the cross—coupling product (Figure 21).

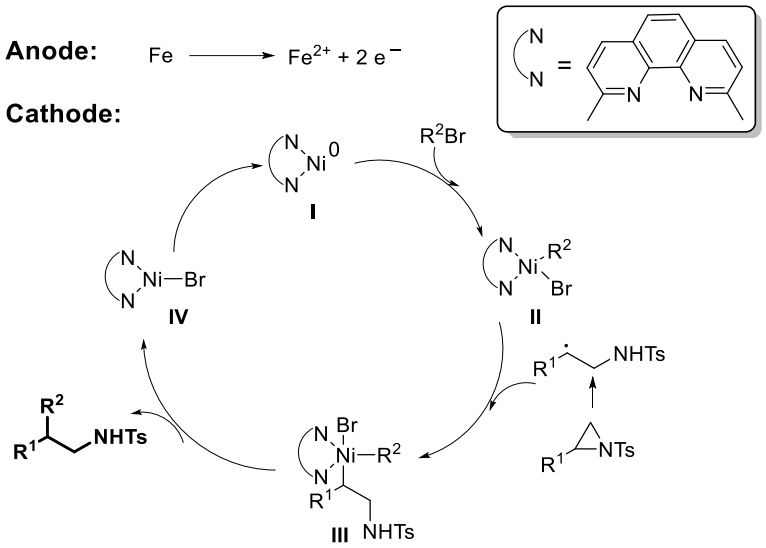
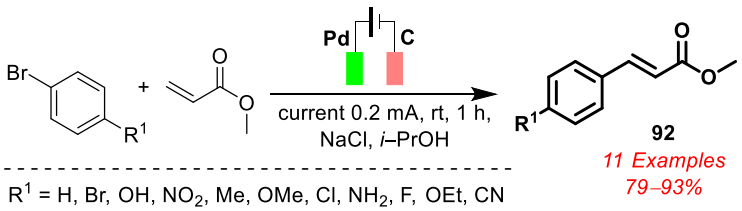


Figure 21. Proposed mechanistic cycle.

Hadi et al. [126] in 2024 devised a novel and efficient electro-organic approach for the synthesis of methyl cinnamate derivatives **92** via the Heck reaction. NaBr and NaCl had no significant effect on the reaction yield, while isopropyl alcohol as the solvent had the highest reaction yield of 93%. A remarkable trend was observed in the effect of electron-withdrawing and electron-donating functional groups on reaction yield. The electron-rich groups gave better yields than the electron-withdrawing groups, which are characterized by high electron density and the ability to contribute electrons to the chemical system. Furthermore, electron-donating functional groups (alkyl/methoxy/amino) increase electron availability and help in the formation of reactive intermediates (Scheme 80).



Scheme 80. Scope of Heck reaction.

8. Comparative Analysis of Electrode Systems

The selection of the right electrode material is an important consideration in the design of electrosynthetic routes where each system has its own trade-off between cost, efficiency and applicability. Table 1 lists a high-level comparative summary of the main families of electrodes discussed in this review, their physical advantages and disadvantages, yields and chemical niches. The most economical and environmentally friendly option is to use graphite and glassy carbon (all carbon systems). Their main advantages are that they are metal-free, following the principles of green chemistry, and

have a high overpotential for the oxygen evolution reaction (OER), widening the potential window for anodic oxidations. Reticulated vitreous carbon (RVC) also provides a large surface area, which increases the efficiency of the reaction. However, these materials are not perfect, as they are prone to surface fouling (passivation) and lack the specific active coordination sites required for many catalytic transformations. Therefore, their applications are mostly limited to radical–radical couplings and direct C—H functionalizations, giving moderate to good yields (40–85%). Conversely, noble metal electrodes (Pt, Au) have best performance in electro-organic synthesis. They exhibit excellent chemical stability and superior electrocatalytic kinetics, especially for the hydrogen evolution reaction (HER) that is important for cathodic reductions. This allows for a wide range of challenging transformations such as dehydrogenative cyclizations and intramolecular annulations, with high yields. These electrodes are the method of choice when precise control of potential is required in the synthesis of complex molecules such as indoles and oxazoles. The major drawbacks, however, are the high cost and the susceptibility to poisoning by typical organic functional groups such as sulfur and amines. On the other hand, noble/carbon mixed systems (C-Pt, C-Au) aim at a compromise between the high performance of noble metals and the low cost of carbon supports. This combination can also enable asymmetric cell configurations that reduce the total energy consumption. The main problem is to carefully match the anodic and cathodic kinetics in an effort to avoid unwanted side reactions. These mixed systems are well suited for continuous-flow electrosynthesis and enable metal-free C—N/C—S bond formations to give benzothiazines. Moreover, first-row transition metals (Ni, Fe, Cu) are increasingly being seen as a more sustainable alternative to precious metals. Their main advantages are their earth abundance and the possibility of d—orbital redox cycles, which make them able to directly act as molecular co-catalysts in different reactions. They are useful for key transformations such as cross-couplings and multicomponent reactions, giving biaryls and functionalized amines. However, they are often susceptible to anodic dissolution, and thus are used as sacrificial anodes, and have a narrow pH stability window, which limits the reaction conditions. Lastly, hybrid and modified surface electrodes are the vanguard of electrode design, with the ambition of unprecedented control over reaction outcomes. These systems can offer exquisite stereochemical control for reactions such as enantioselective C—C couplings by immobilizing chiral modifiers such as L—proline on a carbon surface. This method enables the recovery of heterogeneous catalysts and the generation of active sites that emulate enzymatic pockets for the synthesis of complex chiral alcohols and amines.

To summarize, the choice of electrode material is a compromise. All-carbon systems are best suited for simple, green and cost-effective syntheses. Noble metals provide excellent performance for complex reactions but at high cost. Mixed systems and first-row transition metals give reasonable compromises in cost and performance. Modified surfaces, in contrast, provide the greatest degree of control, especially in regard to stereoselectivity, but suffer from severe fabrication and stability issues.

Table 1. Comparison of various electrode systems for electro-organic synthesis.

Electrode	Key Materials	Advantages	Drawbacks	Reaction Types	Yields	Representative Compounds/Features
All—carbon	Graphite, RVC, glassy carbon	Low cost, metal—free, high OER overpotential, massive surface area (RVC)	Prone to surface fouling (passivation), lack of active coordination sites	Radical–radical couplings, simple direct oxidations, C—H activation	40–85%	Spirooxindoles, pyrrolidines; highly compatible with green solvents (DES)
Noble metals	Pt, Au	High chemical stability, low HER overpotential, excellent electrocatalytic kinetics	High cost, potential leaching, prone to poisoning by sulfur/amines	Dehydrogenative cyclizations, intramolecular annulations, challenging reductions	50–98%	Indoles, oxazoles

Noble/carbon mixed	C—Pt, C—Au	Balance of cost and performance; asymmetric cell dynamics reduce energy loss	Requires careful matching of anodic/cathodic kinetics to avoid side reactions	Metal-free C—N/C—S bond formations, continuous-flow electrosynthesis	40–95%	Benzothiazines, indolines, xanax analogs; excellent for electrolyte-free
First-row transition metals	Ni, Fe, Cu	Earth-abundant, active coordination, able to undergo electrochemical d-orbital redox cycles	Susceptible to anodic dissolution (sacrificial anodes), narrow pH stability window	Cross—couplings (reductive/oxidative), multicomponent reactions, C—heteroatom bonds	45–96%	Biaryls, functionalized amines
Hybrid & modified surface	L—Pro@Cu, Chiral-modified carbon	Exquisite stereochemical control; heterogeneous catalyst recovery	Challenging fabrication, limited chemical/thermal stability of organic modifiers	Enantioselective C—C couplings, asymmetric additions (Henry reaction)	60–97%	Chiral alcohols and amines

9. Highlighting the Green Credentials of Electrosynthesis

As shown in Table 2, many modern electrochemical techniques have real green credentials, besides the mere replacement of reagents. The development of reusable electrodes, operation in electrolyte-free or novel solvent systems and demonstrated scalability all indicate the practical potential of this technology. However, it should also be acknowledged that the reporting of all green metrics (exact energy consumption in kWh/mol or complete life-cycle analysis) is not yet common practice in all reports. This is an important area for future development. To facilitate rigorous comparison of methodologies and to accelerate industrial uptake of electro-organic syntheses, it will be important to encourage consistent reporting of Faradaic efficiency, energy consumption and catalyst/electrode stability.

Table 2. Few green metrics from illustrative reports (2015–2025).

Scheme	Electrode	Products/Reactions Types	Key Green Metrics
7	C—C	Spirooxindole	Novel electrolyte: Utilizes a recyclable deep eutectic solvent (DES) instead of traditional organic solvents
17	Pt—Pt	Oxazoles	High efficiency: Requires only 2 F/mol of charge, indicating high Faradaic efficiency
30	C/PVDF—Pt	Benzothiazines	Electrolyte-free & flow: Works in a continuous-flow cell without auxiliary salt, enhancing scalability and safety
33	C—Pt	Indolines	Electrolyte-free & flow: Demonstrates efficient synthesis in an electrolyte-free flow environment
40	C—Pt	Thiophene chlorination	Gram-scale: The protocol's success on a gram-scale demonstrates its practical applicability for larger-scale synthesis
55	C—hybrid	Henry reaction	Electrode reusability: The fabricated ox—C_{60}—Pro@Cu electrode showed good repeatability over multiple cycles
58	Fe_3O_4—SiO_2—LP@Ni	Buchwald–Hartwig Amination	High reusability: The magnetic electrode was reused for 12 consecutive runs with only a minor drop in yield (96–90%)
71	Mg—Pt	β-—Hydroxy acids	CO_2 utilization: Employs CO_2 as a C1 building block, showcasing carbon capture and utilization (CCU)

10. Challenges, Limitations, and Future Opportunities

Electro-organic synthesis has arisen as a potent and sustainable method in contemporary synthetic chemistry; nonetheless, it encounters numerous problems and limits that hinder its wider implementation (Figure 22). A significant challenge is the restricted substrate scope, as not all organic compounds are compatible with electrochemical conditions, especially those susceptible to oxidation or reduction. The selection of supporting electrolytes and solvents presents constraints, as numerous frequently utilized ionic liquids or salts are expensive, poisonous, or challenging to eliminate from the end result. The scalability of electro-organic synthesis remains in progress; transitioning laboratory-scale reactions to industrial-scale processes necessitates specialized equipment, optimization of current density, and meticulous regulation of heat and mass transfer. Confronting these problems is essential for integrating electro-organic synthesis into conventional organic chemistry.

Researchers expect that electro-organic synthesis will facilitate the discovery of novel approaches and address critical difficulties in modern organic synthesis. Notwithstanding considerable efforts and numerous advancements, there is still much to be done to achieve benign and sustainable synthetic methods. Researchers are approaching a future where green procedures will replace all current conventional methods in organic synthesis (Figure 22). Furthermore, the amalgamation of electro-organic chemistry with diverse disciplines, including materials science, data science, and medicinal chemistry, via collaborations between academia and industry, will establish a robust foundation for the application and progression of electrosynthesis in contemporary synthesis. This review aims to significantly benefit practicing chemists by integrating electrochemistry with organic synthesis, leading to groundbreaking and revolutionary advancements. Simultaneously, the requirement for substantial quantities of supporting electrolyte and costly electrode materials poses a considerable constraint for large-scale electro-organic production.

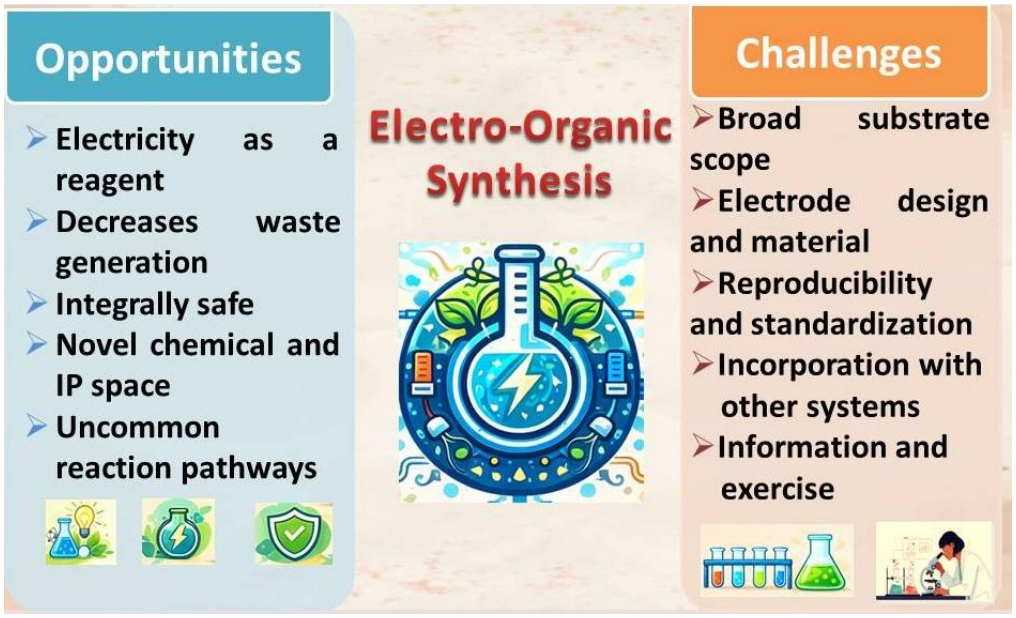


Figure 22. Challenges and opportunities associated with electro-organic synthesis.

11. Concluding Remarks

In the past years, numerous conventional and non-conventional methodologies have been employed for the synthesis of organic compounds. In this context, electro-organic synthesis is one of the greenest and most promising approaches that has

been successfully employed in the synthesis of organic compounds. Numerous types of electrodes, such as C-−C, Pt−Pt, C−Pt, C−Ni, C−Fe, C−hybrid material, RVC−Pt, Mg−Pt and miscellaneous, have been explored to perform the electro-organic transformations. Carbon-based electrodes, such as graphite and RVC, are inexpensive, readily available materials desired for their high oxygen evolution reaction (OER) overpotential that inhibits water oxidation but are prone to fouling. Platinum electrodes exhibit excellent chemical stability and catalytic activity (particularly for the hydrogen evolution reaction) but are constrained by their high costs. First-row transition metals (e.g., Ni, Fe, and Cu) offer low-cost and low-toxicity options that participate directly in catalytic redox cycles, such as cross-coupling processes. On the other hand, hybrid and modified electrodes combine conductive supports with chiral or catalytically active modifiers (such as L−proline) to behave as highly selective active systems able to drive precise enantioselective transformations.

A wide range of methodologies like sulfur insertion, Henry reaction, Buchwald–Hartwig amination, transition-metal-catalyzed, Heck reaction dehydrogenative C−N/C−O/N−N/C−P functionalization, N−H/C−H functionalization, C−N/C−S cross-coupling, ring expansion, oxidation dearomatization/annulation, C−S cyclization, C−H thiolation, dehydrogenative C(sp^2)−H amination, and C(sp^2)-H/C(sp^3) −H functionalization have shown their potential to generate a diversified library of valuable organic compounds. Specifically, phthalazin−1,4−diones, *N*−oxides, 1,3−benzothiazines, lactams, spiropyrrolidines, substituted indoles, 1,3−oxazoles, thienoacenes, diarylphosphole oxides, sultones, benzothiazoles, thiazolopyridines, imidazo−fused *N*−heteroaromatics, oxindoles, carbazoles, isatins, spirocyclo−propylpyrazolones, nitriles, benzoxazoles, epoxides, glycols, aldehydes, substituted pyrrolidines, 2−(4,5−diphenyl−1*H*−imidazol−2−yl)phenol, benzothiazoles, fused indole-based frameworks, 1,2,4−triazole, *o*−benzoquinone, 2−alkoxythiazoles, 1,2,4−thiadiazole, tetrahydroquinolines, chromeno[2,3-*d*]pyrimidine, α−pyrones, cyclic amides/lactones, phthalazine, 1,4−dihydropyrano[2,3−*c*]pyrazole, α-amino ketones, C−5 sulfenylated amino uracils, 4*H*−pyrans, (*S*)−2−nitro−1−phenylethanols, (*R*)−2−hydroxy−2−phenylpropanenitriles, diphenylamines, disubstituted alkynes, β−alkoxy and β−amino sulfides, vicinal α−azidoketones, benzyl alcohols, chiral substituted secondary amines, acetate oximes, α−fluorinated heterocycles, β−hydroxy acids, dicyano−2−(2−oxoindolin−3−ylidene) malononitriles, β−arylethylamines, and methyl cinnamate were developed during the recent decade by the application of electrochemical methods.

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Abbreviations

FDA	Food And Drug Administration
CNS	Central Nervous System
OLED	Organic Light-Emitting Diode
NHCs	*N*-Heterocyclic Carbenes
LUMO	Lowest Unoccupied Molecular Orbital
HOMO	Highest Occupied Molecular Orbital
RVC	Reticulated Vitreous Carbon
DES	Deep Eutectic Solvent
SET	Single Electron Transfer
DMSO	Dimethyl Sulfoxide
EDG	Electron-Donating Group
EWG	Electron-Withdrawing Group
HNFM	Hydroxymethyl-Nitro-Furan-Methanol
Bu_4NOAc	Tetrabutylammonium Acetate
PAHs	Polycyclic Aromatic Hydrocarbons
Cp_2Fe	Bis(Cyclopentadienyl)Iron(II)
AcOH	Acetic Acid
TBAI	Tetra *N*-Butyl Ammonium Iodide
NMR	Nuclear Magnetic Resonance
DABCO	1,4—Diazabicyclooctane
DMA	Dimethylacetamide
DFT	Density Functional Theory
MeOH	Methanol
HFIP	Hexafluoroisopropanol
TEMPO	2,2,6,6—Tetramethylpiperidine—1—Oxyl
PVDF	Polyvinylidene Fluoride
TfOH	Trifluoromethanesulfonic Acid
Mes-Acr^+	9—Mesityl—10—Methylacridinium Cation
DDQ	2,3—Dichloro—5,6—Dicyano—1,4—Benzoquinone
pH	Potential Of Hydrogen
mA	Milliampere
MeCN	Acetonitrile
Cbz	Benzyloxycarbonyl
LED	Light-Emitting Diode
mV	Millivolt
GO—Hypip	Graphene Oxide (2S,3R)-3-Hydroxypiperidine-2-Carboxylic Acid
MWCNTs	Multiwalled Carbon Nanotubes
ox	Oxalate
SPE	Solid Polymer Electrolyte
NPs	Nanoparticles
i—PrOH	Isopropyl Alcohol
tert	Tertiary
DMF	N,N—Dimethylformamide
TBAB	Tetrabutylammonium Bromide
EGB	Electrogenerated Base
MCR	Multicomponent Reaction
GF	Graphite Felt
CCE	Constant Current Electrolysis
rt	Room Temperature
h	Hour
TMS	Tetramethylsilane
Et	Ethyl

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min	Minute
OAc	Acetate
Me	Methyl
DCE	1,2—Dichloroethane
i—Pr	Isopropyl
n—Bu	Normal Butane
PivONa	Poly(Vinyl Alcohol)–Sodium Acetate
PivOH	Poly(Vinyl Alcohol)
t—AmOH	Tert—Amyl Alcohol
M	Molar
MTES	Methyltriethoxysilane
Conc.	Concentrate
n—PrOH	N—Propanol
TFE	Tetrafluoroethylene

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