



The Use of Electro catalysis as a Tool for Organic Synthesis

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Abstract: Recently, electrochemistry has drawn more attention as a flexible method for completing difficult transformations at the cutting edge of synthetic organic chemistry. Several new electrochemical techniques for the creation of useful chemical motifs have been developed as a result of electrochemistry's exceptional capacity to produce extremely reactive radical & radical ion intermediates in a regulated manner under benign conditions. To further improve control over the selective production and downstream sensitivity of these reactive intermediates, recent innovations in electro synthesis have highlighted a greater usage of redox-active electro catalysts. Moreover, electrocatalytic mediators allow for the subversion of the thermodynamic and kinetic barriers encountered in conventional organic synthesis, allowing for the progression of synthetic transformations in a mechanistically distinct manner from simply chemical techniques. The main breakthroughs in synthetic electrocatalysis over the last ten years are highlighted in this overview, with an emphasis on the processes and fundamentals of catalyst design that underlie these developments. The nature of the electrocatalyst and the categorization of the synthetic transformation are used to categorize a wide range of both reductive and oxidative electrocatalytic techniques.

Keywords: Electrochemistry, Anodic oxidation, Bio electrochemistry, Organic synthesis

Introduction:

Electrochemistry has developed since its inception in the 19th century to serve a number of applications including energy storage, metallurgy, and chemical synthesis. Electrochemistry has been successful in a wide variety of settings because of the special advantages it offers. An externally applied potential is a powerful tool for driving processes away from their thermodynamic equilibrium states, which opens up previously inaccessible reaction paths and chemical intermediates [1]. The moderate conditions and scalability options provided by electrochemical systems lend credence to its application in industrial synthesis of bulk and fine compounds. Electrochemistry is a promising strategy for enhancing the efficacy and durability of synthetic chemistry since it uses electricity instead of ecologically damaging chemical reagents like stoichiometric oxidants and reductants.

There has been a fascinating and continuous rebirth of electrochemistry in the field of organic synthesis in recent years. The aforementioned advantages of electrochemistry have contributed to the recent explosion in the number of electrochemical synthesis procedures, some of which enable reactions that are just not possible using more conventional chemical approaches [2].

Electrochemistry, in particular, lends itself to the inventive use of radical intermediates in order to accomplish synthetic transformations, as it facilitates single-electron oxidation &

reduction processes. Electrochemistry emerges as a potent technique for tackling current trends in organic synthesis when the production and downstream sensitivity of these intermediates are put under the command of catalytic redox mediators [3].

The improved capabilities provided by redox mediators in electro synthesis are readily apparent upon conceptual study of an electrochemical reaction. Substrate activation at the electrode surface (i.e., straightforward electrolysis) is facilitated by electrochemistry, but this mode of activation frequently necessitates substrate design to regulate reactivity and can forego control over the selectivity of chemical steps subsequent to electron transfer [4]. Electrochemistry's potential becomes more apparent when it is used to build and modify complex organic molecules, but this can also highlight the limitations of direct electrolysis. Redox-active catalysts, which promote effective electron transfer events and provide more selectivity compared to non-mediated reactions, can, however, be used to circumvent such limitations.

Homogeneous or molecular electro catalysis makes use of catalytic species that are molecules themselves, such as complexes of transition metals or redox-active organic compounds. The latter scenario, in which a catalytic electrode is involved, is typically referred to as "electro catalysis," while systems with molecular mediators are called "molecular catalysis for electrochemical processes." Given that the characteristic feature of an electro catalytic system is the utilization of a redox-active species that can facilitate a catalytic cycle & accelerate a specific reaction pathway, this study defines electro catalysis to include both homogeneous and heterogeneous examples. Many of the exhibited reactivities are made possible by electro catalysis and are unavailable in the absence of an electrochemical catalyst.

The development of electro catalyst systems for energy-related purposes (such as water splitting or CO₂ reduction) as well as, more recently, synthetic chemistry, have contributed significantly to the breadth of electro catalysis in the chemical literature. There has been an enormous uptick in interest in electro catalytic techniques within the field of organic synthesis as a result of the growing need for more effective and environmentally friendly synthetic procedures.

This article summarizes the most significant developments in the field of synthetic electro catalysis during the past decade (since around 2010). We hope to demonstrate the adaptability of these electro catalytic systems and to provide examples of the design ideas underlying the creation of the electro catalytic mediators that provide their driving force. Based on how they facilitate electrochemical reactions, the electro catalysts mentioned here fall into one of two broad categories. Electro catalysts can be broken down into two broad classes: those that accelerate redox processes and those whose primary goal is to facilitate electron transfer events among electrodes and substrates (redox mediators) [5]. The second type of electro catalysts we cover are those that transmit not only electrons but also chemical data to substrates, putting the catalyst's reactivity and selectivity in the hands of the designer. Electro catalysts are largely studied for their use in sub-stoichiometric reactions, in keeping with the standard definition of a catalyst [6]. However, in order to provide a comprehensive description of the reactivities made possible by electrochemical processes, we include some illustrations in which a greater loading of an electrochemical mediator is employed.

The state-of-the-art focuses on the mechanistic specifics of various electro catalytic reactions, both oxidative and reductive. Our discussion begins with recent developments in the well-established field of oxidative electro catalysis, and continues with new findings in the less investigated fields of electro reductive conversions, paired electrolysis, or heterogeneous electro catalysis. Lastly, we investigate bio electro catalysis and electro photo catalysis to show how electro catalytic techniques can be applied in a variety of different fields. These advances highlight the potential of electrochemistry to facilitate increasingly complex reactions and exemplify many of the design ideas utilized to achieve a variety of electrocatalytic techniques.

Anodic oxidation:

The ability to efficiently build C-C bonds and incorporate heteroatoms into molecular structures is a major benefit of oxidative processes in the synthesis of complex compounds. Stoichiometric concentrations of oxidizing agents, such as high-valent chromium & manganese complexes and hypervalent iodine species, are often used in conventional chemical procedures for oxidative processes, which can be dangerous and result in significant waste. New techniques have allowed for more gentle reaction systems that make use of more desirable oxidants like oxygen and hydrogen peroxide [7]. Yet, the ACS Green Computing Institute Pharma Roundtable still lists "alternatives for oxidations" as one of the top green chemistry research areas. The use of a catalyst that can be recycled through anodic oxidation is preferable from a green chemistry standpoint because it drastically cuts down on waste. Indeed, the primary byproduct of electrochemical oxidations is H₂ gas, which is produced by coupling the oxidation process with a hydrogen evolution event.

Anodic oxidation has been utilized in the design of electrocatalyst devices to perform a wide variety of chemical changes beyond its value in recycling conventional chemical oxidants. Chemical mediators for such oxidative systems cover a broad spectrum. Examples of radical cations used for electron transfer include triaryl amines & ferrocenium ion (Fc⁺). Hydrogen atom transfer (HAT) agents include the phthalimide N-oxyl (PINO) radical, the radical cation from quinuclidine, chlorinated radicals, and nitrate radicals [8]. For the purpose of formal hydride transfer, oxoammonium cations have been used. Reductive elimination, as seen frequently in C-H activation processes, can be facilitated by the anodic oxidation of metals ions to high valent states. Recently, a wide variety of extreme transformations have been investigated using Earth's plentiful 3D metals. In the sections that follow, we'll talk about the various types of electro oxidative reactions and classify them according to the type of redox mediator they use.

Hypervalent iodine and other halogen-based mediators:

Because their low cost and lack of toxicity, halide salts are one of the most used types of halogen-based mediators. Hypervalent iodine compounds and iodates/periodates are two other halogen-based mediators that have received less attention. Reactive halogen compounds (such Cl₂, Br₂, and I₂) are produced during anodic oxidation of halides, and hypohalites can be produced during electrolysis in water.

Oxidized halides have been used to investigate conventional reactivities such as nucleophilic substitution & base-promoted elimination. One underappreciated yet potent strategy for creating carbon-heteroatom connections is the generation of radicals by the homolytic breakage of weak heteroatom-halogen bonds [9]. To achieve high reaction yields, halogen-based mediators are sometimes utilized in stoichiometric concentrations despite the fact that they are theoretically regenerable in electrochemical systems.

- The use of halogens as an activator for alkenes

Using an electrochemical amino oxygenation process mediated by iodide, Zeng, Sun, and colleagues produced 3-methoxyindolines. (Fig. 1). This reaction involves the intramolecular S_N2 reaction of an iodine ion created by an anode, followed by an additional substitution with a methoxide ion created by a cathode. Since the iodide source also acts as an electrolyte, LiClO₄ can be withdrawn from the system without impacting the yield.

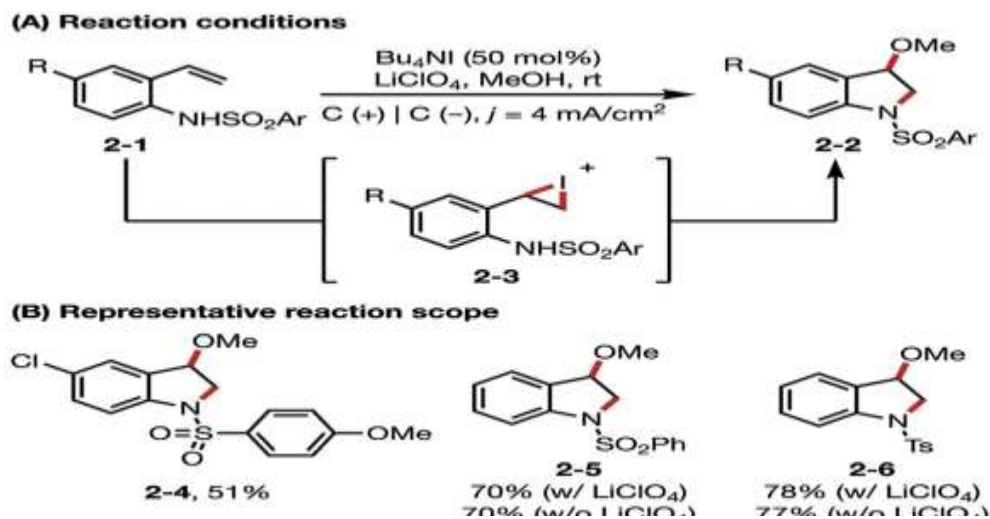


Fig 1: The alkene amino oxygenation mediated by iodide.

- Nitriles and carbonyls can be α -functionalized

Recently, there has been a lot of research into the possibility of using halides as electrochemical mediators for the α -functionalization of nitriles and carbonyls. In order for these functional groups to react with Br_2 or I_2 , they must first undergo α -deprotonation, which results in stabilized anions (see intermediate 4-3, Fig. 2). In an $\text{S}_{\text{N}}2$ way, many different nucleophiles can readily displace this newly installed halogen group. Within the same molecule, cyclic motifs including cyclopropanes, cyclobutanes, cyclopentanes, α -lactams, and dihydrofurans can be generated by appending a nucleophile (Fig. 2). Nitrile anions, enolates, imidazole, sulfonates, and carbamodithioates are just few of the carbon, nitrogen, & sulfur nucleophiles that have been investigated at the intermolecular level. Although halide salts are very cheap, they are used in high loadings or even stoichiometric levels in several of these procedures (see example 4-6, Fig. 2). Baran and Sabatini created compound 4-5 to test the scalability of their procedures on a 100-gram scale.

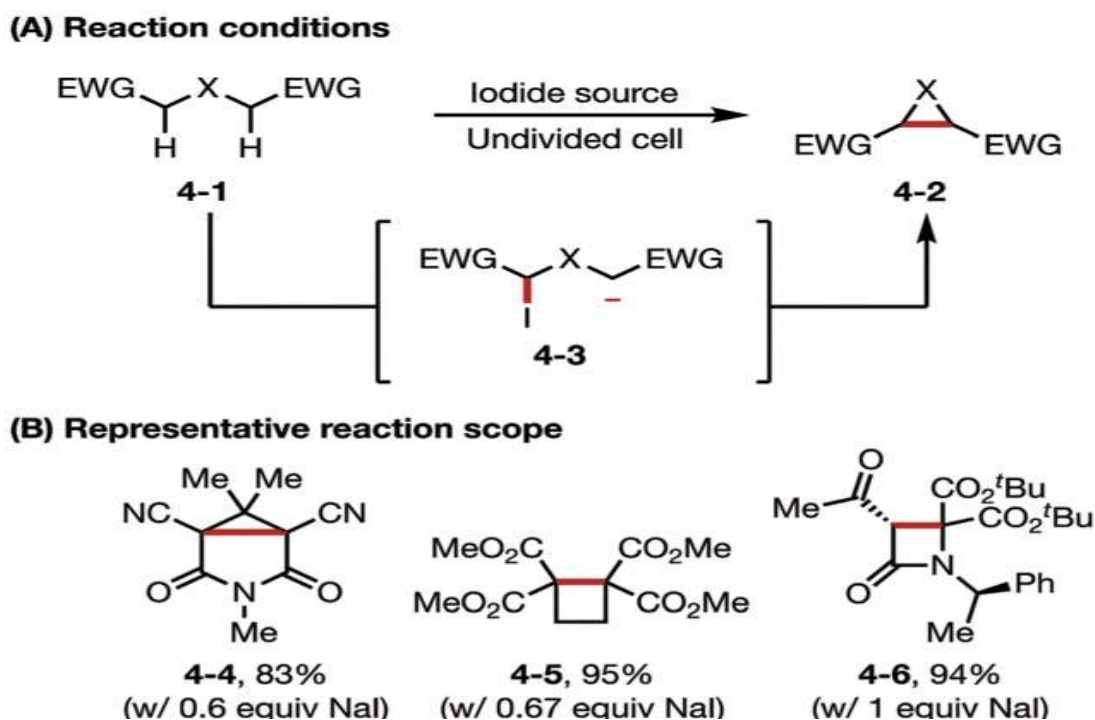


Fig 2: Cyclization facilitated by iodide.

- A Mediator Role for Periodate

It is common practice to use the periodate anion, a versatile oxidant, for oxidative cleavage of diols or in situ regeneration of catalysts like osmium tetroxide or organometallic tetroxide. Anodically produced periodate was used in a study by Pillai and colleagues to examine the oxidative breakdown of vicinal diols, problems aminoalcohols, & -hydroxyketones. To avoid inefficient cathodic reduction during manufacture of this hypervalent iodine reagent, a divided cell is used. In addition to its application in the electrolytic oxidative cleavage of alkenes, this mediator has also been exploited by Schäfer and coworkers to stimulate renewal of ruthenium tetroxide.

Nitroxyl mediators:

Since their discovery in the 1960s, stable nitroxyl radicals have piqued the interest of synthetic chemists. Electrochemical oxidation of these radicals yields oxoammonium cations, which have become the subject of much study for their ability to oxidize alcohols and amines. Using a combination of experimental and computational methods, Minter, Sigman, and colleagues (2015) classified nitroxyl mediators into four classes based on their catalytic activity for oxidizing alcohol. This study's upper left quadrant of the resulting graph features highly active nitroxyls that can be synthesized at low potential, making them attractive for use in energy-related contexts. Poorly active mediators with large oxidation potentials that are nonetheless useful as EPR labels are located in the bottom right corner. The most widely used nitroxyl catalyst and EPR label, TEMPO, is not the optimal choice for either application, while its widespread adoption is understandable given its accessibility. Electrocatalytic processes involving nitroxyl radicals were examined in depth by Stahl, Rafiee, and coworkers.

- Oxidation of Alcohol.

Cooperative electrochemical able to transmit and receive bromide and nitroxyl radicals were reported in the 2000s by the research groups of Tanaka, Onomura, & Frontana-Urbe; these cations were able to oxidize primary and secondary alcohols to their corresponding carbonyl compounds. Alcohols can be converted to carboxylic acids, aldehydes, and ketones via direct oxidation of TEMPO, as described by Tanaka and Brown.

- Amines and carbamates oxidize:

Electrochemical oxoammonium cation production has been used to oxidize amines and carbamates, resulting in the formation of amides and imides. Little used a bromide/TEMPO cooperative system; Kashiwagi and Stahl used modified TEMPO to achieve the same result (Fig. 3). The hypothesized mechanism begins with the generation of a stabilized iminium species 16-3 via a two-electron-one-proton transfer, which is then trapped by water to create a hemiaminal 16-4. The carbonyl group is formed by transferring two electrons and a proton from hemiaminal 16-4 in a second step.

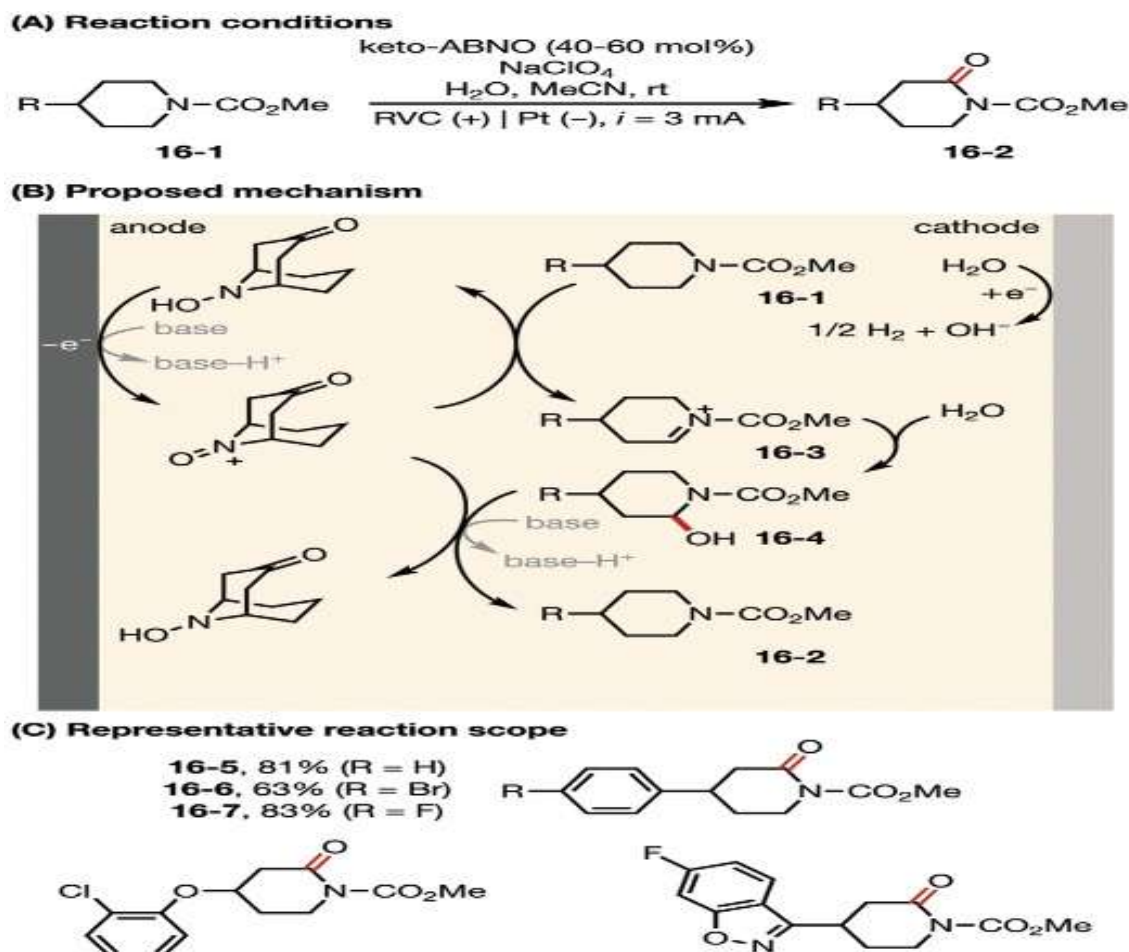


Fig 3: Carboxylate oxidation mediated by nitroxyl.

- Radical formation around nitrogen:

Nitroxyl radicals and their associated oxoammonium cations have recently been found to have novel reactivities. Under basic conditions, Xu and coworkers reported in 2014 that nitrogen-centered radicals were formed via the oxidation of amides by TEMPO⁺. It has been hypothesized through mechanistic research that this mechanism involves the transfer of a single electron from an anion with a nitrogen center to a cation with an oxoammonium. To get the bicyclic products, the nitrogenated radical was used in an intramolecular reaction with an olefin. Stoichiometric levels of TEMPO are used because it is also used as a radical trap in this reaction.

Mediating effects of transition metal complexes:

When compared to employing stoichiometric chemical oxidants to drive catalyst turnover, the combination of trivalent catalysis & electrochemistry provides superior atom efficiency, softer reaction conditions, with safer protocols. Electrochemistry has also been found to increase previously difficult or unknown reactivities by activating metal catalysts or critical reactive intermediates produced from these catalysts.

There are two types of metal-electrochemical reactions. To begin, complexes of transition metals can act as intermediaries for the direct transmission of electrons between electrodes or target substrates. In particular, the group transfer to the substrates is more selective and efficient due to the lower oxidation potentials of transition metal-bound substrate adducts and the persistent radical properties of the oxidized open-shell species. Several previously infeasible or difficult-to-accomplish transformations have been made achievable by these metall-electrochemical techniques. Recent literature provides a more in-depth analysis of these techniques.

Second, classic transition-metal catalysed C-H activations are supported by electrochemistry, which results in a wide range of transformations that forge carbon-carbon and carbon-halogen bonds. Regenerating the active transition metal catalyst and oxidizing the transition metal catalyst intermediary to a high-valent species to cause future reductive elimination are two common functions of anodic oxidation in a transition-metal-catalysed C-H activation. Recent reviews provide in-depth treatment of transition-metal catalysed C-activation processes. Using electricity as the oxidizing equivalent in C-H activation processes is a significant benefit of metallic-electrochemistry since it eliminates the need for additional chemical oxidants and the production of unwanted by-products.

Palladium, rhodium, iridium, and rhodium-based catalysts have long dominated anodic C-H activation, although 3d earth-abundant metals like nickel and cobalt are increasingly being used in this process. Here, we'll go over the most current findings in metall-electrochemical reactions, organized by metal mediator.

Iron:

Because of its low oxidation potential, ferrocene is a functional group-tolerant, selective redox catalyst. In a number of chemical reactions, ferrocene is used to activate N-H bonds, leading to the generation of extremely reactive amidyl radicals. Electrochemical C-H activation processes are just one more use for other iron complexes.

- Cycles of radicals:

Using $\text{CF}_2\text{HSO}_2\text{NHNHBoc}$ as a CF_2H radical precursor, the Xu group describes aspects electrochemical alkyne functionalization to obtain fluorinated dibenzazepines (Fig. 4). First, Cp_2Fe is anodically oxidized to Cp_2Fe^+ , which kicks off the electrolytic process. Simultaneously, H_2 and MeO are produced when methanol is reduced at the cathode. $\text{CF}_2\text{HSO}_2\text{NHNHBoc}$ (38-2) undergoes deprotonation and oxidation to form the intermediate 38-5, which undergoes further electron and proton loss to produce the diazene 38-6. A CF_2H radical is produced upon further breakdown of 38-6, which can react with an alkyne group to provide the vinyl radical 38-8. A 7-member ring structure is obtained when this vinyl radical cyclizes into phenyl rings (reactions 38-9). Finally, the cyclized intermediate is converted into the dibenzoazepine product 38-3 through oxidative aromatization.

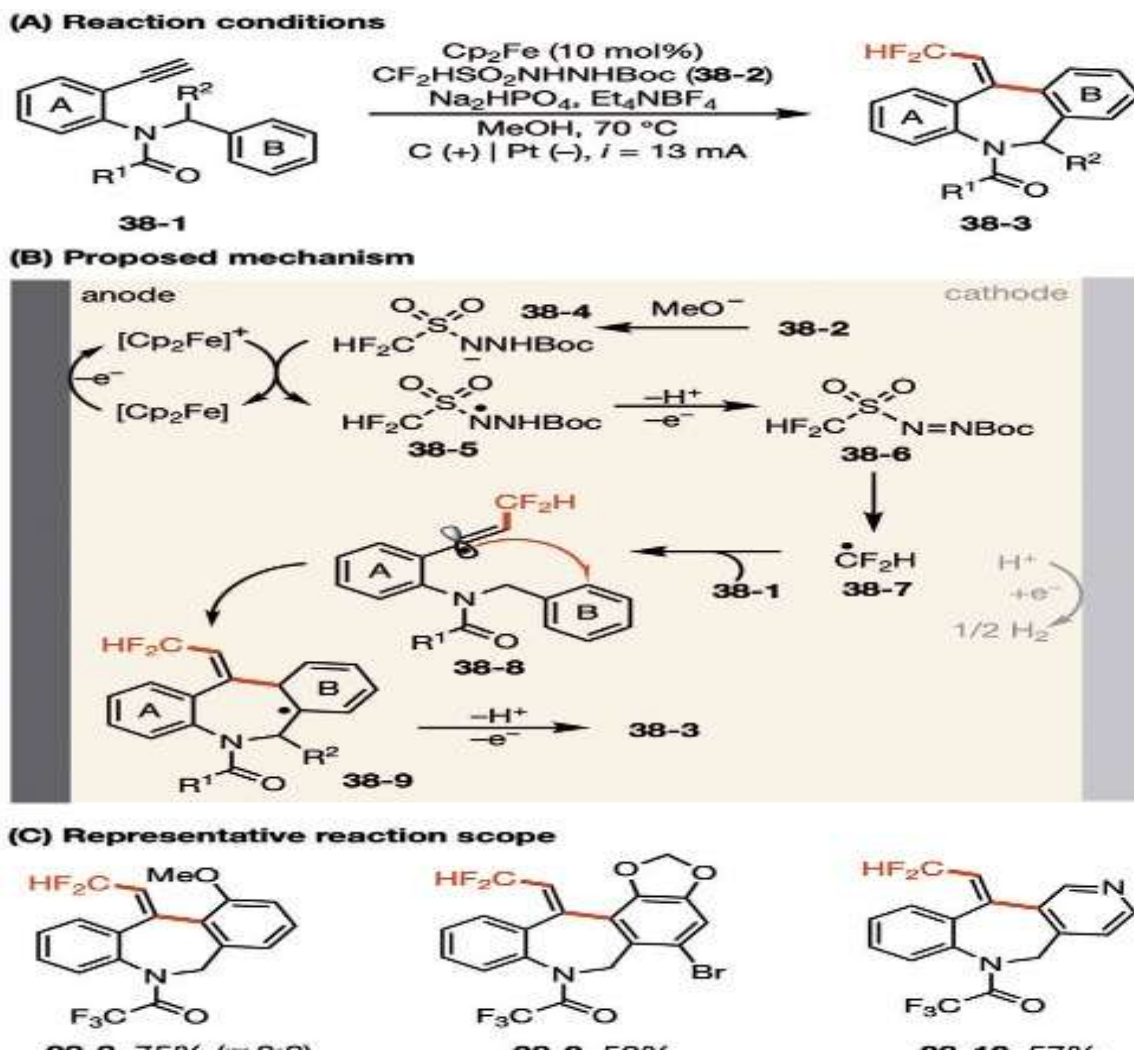


Fig 4: Difluoromethylation of alkynes via electrochemistry.

Copper:

2.9.1. Cyanation of alkenes.

Cu-catalyzed stereoselective electrocatalyst cyanophosphinoxylation of vinylarenes. As CuII-CN is oxidized to CuIII-CN, a secondary associated with taking is oxidized to produce a P-centered transient radical. Product These two open-shell intermediates react with the alkene in sequence via the persistent radical action. To stop the Cu metal from plating out during the cathodic reduction of copper ions, CN and bisoxazoline (BOX) ligands are essential. The architecture of the ester bonds on the sBOX ligand was linked to the remarkable enantioselectivity of this transition.

A dual electrocatalytic strategy for the asymmetric hydrocyanation of conjugated alkenes. CoIII-H and CuIII-CN complexes are jointly produced during anodic oxidation. Hydrogen atom transfer from the CoIII-H species to the alkene substrate is made possible, and the CuIII-CN species provides a cyanide radical that is comparable to the product alkyl radical. Again, chiral Cu(sBOX) catalysts were used to promote enantioselectivity in the hydrocyanation reaction. Electrochemistry recycles both catalysts, eliminating the need for an external oxidant. Notably, the efficiency and enantioselectivities achieved by executing the same hydrocyanation reaction with conventional chemical oxidants with electrical input have not been found to be equivalent to the electrocatalytic methodology. Conjugated alkenes such as alkenylarenes, dienes, enynes, and allenes are all amenable to functionalization via this

reaction. According to DFT calculations, the pendant ester group significantly contributed to the high enantioselectivity of this reaction by interacting with the aryl group of the alkene substrate, revealing the mechanism of enantioinduction enabled by the Cu(sBOX) catalyst.

Ruthenium:

Nitrogen-based directing groups are commonly used as a means of promoting C-H activation. Ru-catalyzed electrocatalytic C-H activation of aromatic carbamates & phenols was reported by the Ackermann group (Fig. 5). Mechanistic analyses supported the authors' proposal that aromatic carbamate coordination precedes C-H activation in the catalytic cycle, leading to intermediate 56-5 in the ruthena(II) cycle. To obtain the seven-membered ruthena(II) cycle 56-7, an alkyne is subsequently coordinated, and then migratory insertion occurs. Reductive elimination is then performed on this species to get the final product with Ru⁰ intermediate 56-8. The catalytic cycle concludes with the reversible anodic oxidation of the intermediate 56-8 back to the active Ru^{II} species 56-4.

Later, a Ru-catalyzed C-H activation was reported by the Ackermann group to allow annulations among alkynes & benzoic acids. The proposed mechanism is analogous to the preceding instance. Using a Ru^{II} catalyst, the team demonstrated in how to electrochemically assemble three separate isoquinolines.

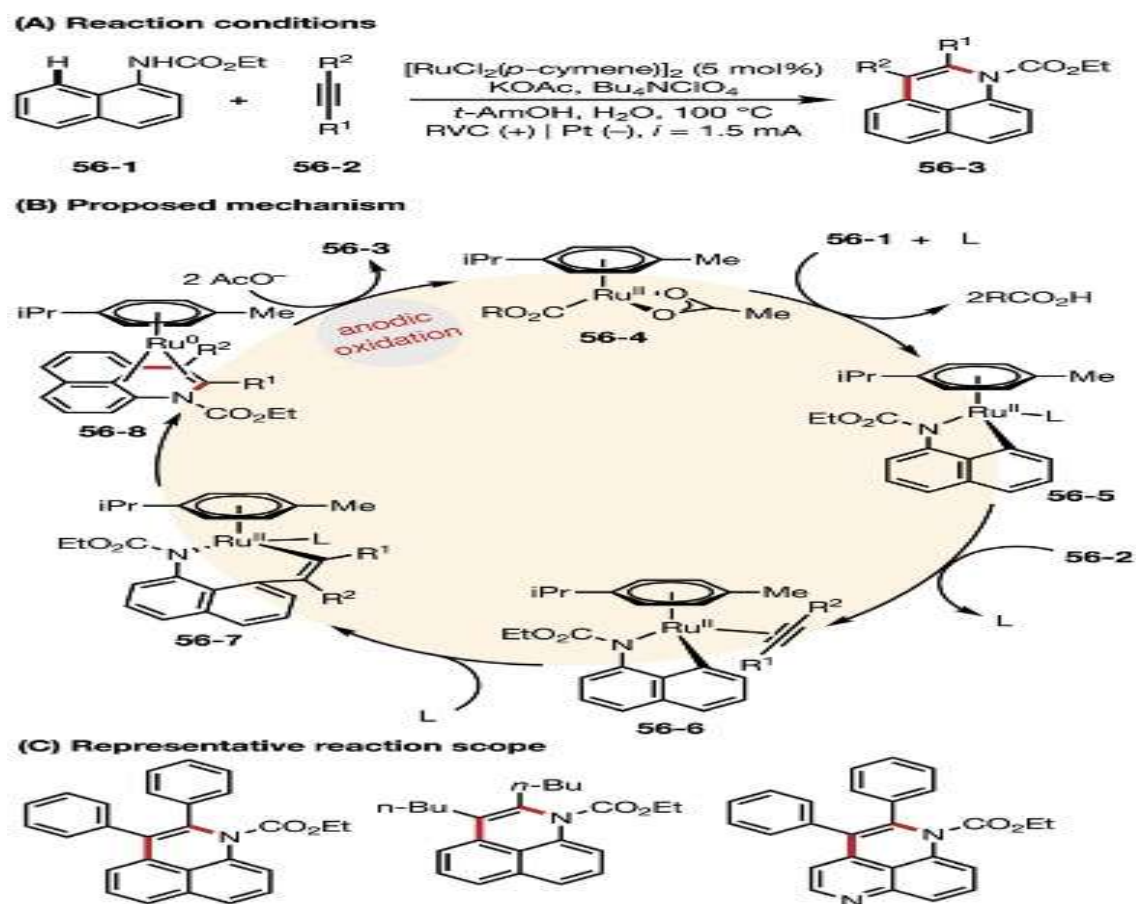


Fig 5: Electrochemical aryl carbamate alkyne annulations catalysed by ruthenium.

Electrolysis in pairs:

The capacity of paired electrolysis to couple two desirable electron transfer inside the same reaction system has garnered a lot of attention in recent years. Industrial chemical & energy conversion processes have benefited from this method, which is both effective and atom-economical. Paired electrolysis is increasingly useful, but it is still rarely used strategically in

the context of organic synthesis. Ingenious reaction design and novel solutions to synthetic issues are common outcomes of sequential and convergent pair electrolysis, in which both cathode and anode are used to promote the same transformation. However, difficulties may develop due to interference between the planned degrading routes of electrogenerated reactive intermediates and their interelectrode mass movement. Furthermore, in many instances, it can be critical to the reaction selectivity to balance the rates of both anodic and cathodic events. Recent advances in coupled electrolysis have used electrochemical mediators to improve reaction efficiency & selectivity in light of these obstacles. Earlier, we went over a few scenarios where it was suggested that paired electrolysis would play a role in the total change. Recent advances in which paired electrolysis is being used deliberately to address synthetic challenges will be the primary focus of this section.

Nickel catalysis:

The Hu group claimed that they were able to directly arylate benzylic C-H bonds using a mixture of 4,4'-dimethoxy-2,2'-bipyridine (L1) and (DME)NiBr₂ catalysts, THF/CH₃CN co-solvent, fluoride ions tin oxide (FTO) coated glass anode with carbon fiber cathode. Ketones, esters, sulfoxide, and amides are all compatible with the reaction. Toluene derivative 116-1 is oxidized to benzylic radical 116-7 at the anode to kick off the process (Fig.6). Meanwhile, NiII species 116-8 are synthesized from LNiIIBr (116-10) via cathodic reduction & oxidative addition with ArBr (116-2). NiIII complex 116-9 is formed when radical 116-7 acts as an interceptor on this intermediate. The required result, 116-3, is obtained using reductive elimination.

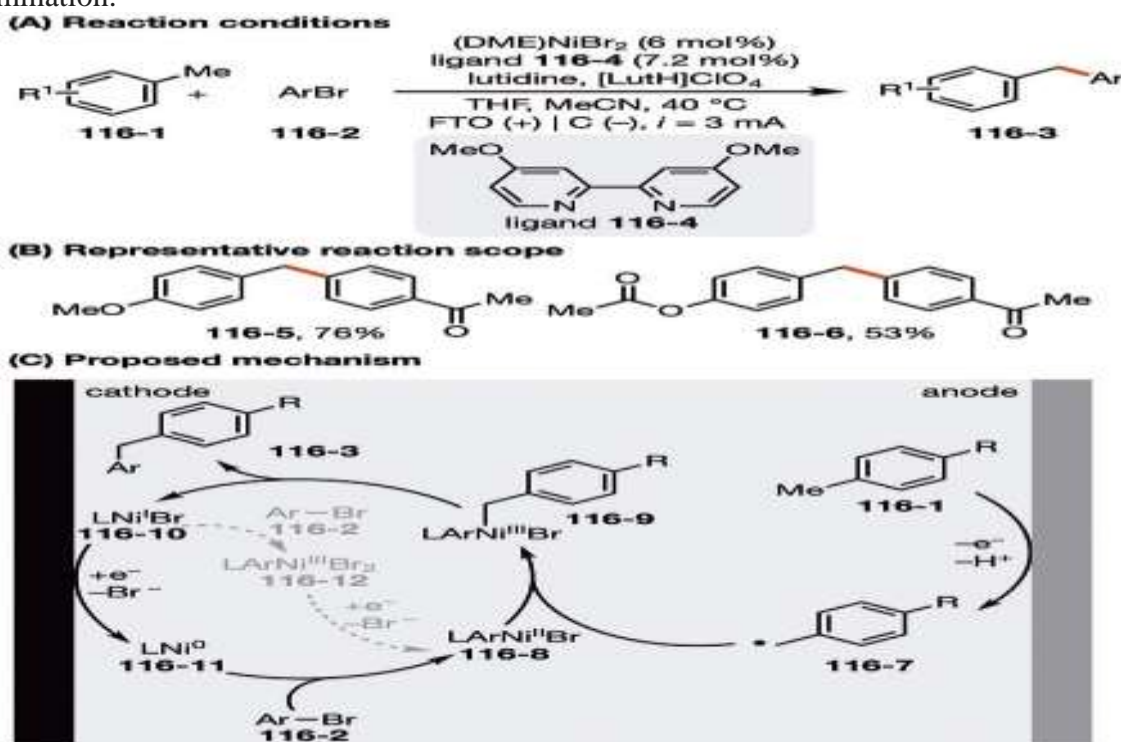


Fig 6: Direct arylation of benzylic C-H bonds using nickel catalysis in an electrochemical process.

Distinct types of electrocatalysis:

During electron transfer events, electrodes can interact closely with substrates and potentially display catalytic capabilities. Because an electrode's surface is not a small-molecule mediator like those detailed in the preceding sections, it might be difficult to discern the electrode's role in enabling a given transformation. Substrate and intermediate interactions (adsorption and desorption) with the electrode surface (electrostatic forces, -interactions, chemical bonds)

are all included in the catalytic character of the electrode when utilized as a heterogeneous catalyst.

There has been notably less research on the catalytic potential of electrodes. Electrode characteristics such as enhanced time-of-flight (TOF) or product selectivity relative to alternate electrodes were among those defined by the ACS Catalysis editorial team as defining characteristics of electrocatalysts. Some of the examples in this section contrast the characteristics of carbon with platinum electrodes, while others show how nanoparticles can be included into electrodes to improve their performance. This section is not meant to be an all-inclusive history of heterogeneous electrocatalysis; rather, it is meant to draw attention to this developing field as it pertains to synthetic organic chemistry.

Bio electrochemistry:

To facilitate chemical synthesis, bioelectrocatalysis is a diverse discipline that seeks to integrate the best features of biocatalysis with electrocatalysis. Redox reactions are made possible by bioelectrocatalytic processes, which employ redox-active enzymes or electroactive microbes as catalysts. Bioelectrocatalysis has many benefits, including its high activity, selectivity, atom efficiency, and moderate reaction conditions. Clean biofuels, biodegradable materials, and high-value chemicals are just a few examples of where bioelectrocatalysis has been put to use in recent years.

Isolated oxidoreductases & electroactive bacterial cells are two examples of common bioelectrocatalysts. Often utilized in biosynthesis are isolated oxidoreductases, whose reactivity has been further optimized by chemists thanks to advances in protein engineering methodologies like directed evolution. Electroactive microbes are utilized in bioelectrochemical systems because of their ability to transmit electrons while still being alive. Microbial cells can catalyze various chemical processes and generate many different products due to their complex metabolic pathways, which are essential for biocatalysis based on microbial cells.

Electrode-to-bioelectrocatalyst electron transfer can occur either via direct electron transfer (DET) or via a mediator (MET). Synthesis of fine compounds and chiral organic products will be the primary emphasis of the ensuing bioelectrocatalysis discussion. Recent publications have increased the length of their overviews of bioelectrocatalysis.

Conclusions:

Electrocatalysis has seen rapid development and increased interest in organic synthesis applications over the past decade. The capacity of electrochemistry to use electricity as a "imaginary" reagent to catalyze a wide variety of one-electron oxidation and reduction reactions has encouraged these recent developments. This study highlights recent advancements that show how the use of electrocatalysts, which boost the selectivity & efficiency of electrochemically driven techniques, further enhances this capacity. Accessing extremely reactive radical intermediates from available starting materials and controlling their downstream reactivity to permit complicated transformations in a single step have both been demonstrated to be possible using electrocatalysis. Electrocatalysis has thus far broadened the scope of numerous staple reactivities, frequently under benign conditions, including C-C and C-X bond creation, alkene functionalization, and C-H activation.

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