

1.1 Introduction

Heterocyclic compounds have a major function in organic chemistry nowadays. They can be found in various foods, medicines, and agricultural supplies [1]. In addition, they can have many biological effects, including those of antibacterial, anti-inflammatory, antioxidant, anticancer, and other types [2]. *N*-heterocycles are in the lead, and not just because they are abundant; they also play an essential role in chemistry, biology, and technology [3]. These ring structures serve as a critical structural feature in many cellular components (vitamins, hormones, hemoglobin, DNA, and RNA[4].

N-heterocycles are found in various natural products, colors, pharmaceuticals, organic materials, and molecules with significant pharmacological promise [5]. Triazines [6], indoles [7], oxadiazoles [8], benzimidazoles [9], benzothiazoles [10], imidazoles [11], triazoles [12], quinolines [13], pyrimidines [14], pyrazoles [15], and quinazolines have all received considerable attention in recent years [16].

Natural products, medicinal molecules, and organic functional materials are rich sources of nitrogen-containing heterocyclic compounds. Advancements in this area are anticipated to pave the way for novel methods of synthesizing heterocyclic compounds containing nitrogen in a way that is gentle, eco-friendly, inexpensive, and easy to operate[17]. In this area, it is anticipated that new avenues will be opened for the synthesis of nitrogen-containing heterocyclic compounds under settings that are mild, friendly to the environment, inexpensive, and straightforward to operate [18].

Because of the wide-ranging applications of *N*-containing heterocycles (especially pyrazole and indolin-2,3-dione derivatives), a study was conducted through Ugi to synthesize hybrid molecules with these two structural pharmacophores [19].

1.2 Nitrogen-containing Organic Compound

Nitrogen is present in vitamins, hormones, amino acids, and nucleic acids; it serves as the basic building block of all life. This means that their structural characteristics span a wide range, from simple to complex, high to low substitution, and cyclic to acyclic. Nitrogen is a key skeleton or component of many biologically, pharmaceutically, and synthetically active compounds. *N*-heterocyclic organic molecules are crucial in many spheres of human life, not just industry and biology.

Consequently, due to the significant number of nitrogen-containing compounds, the enormous diversity of their structure, and their extremely distinct fields of use, this chapter has examined some important types of heterocyclic compounds. Heterocycles with five-membered nitrogen atoms include pyrrole, pyrazole, and triazole; pyridine has six-membered nitrogen atoms; and benzimidazole, indole, and indolin-2,3-dione are all examples of fused heterocycles [20]. Pyrrole, pyrazole, and triazole are examples of nitrogen-containing five-membered heterocycles ring; pyridine contains nitrogen-containing six-membered heterocyclic; and fused heterocycles include benzimidazole, indole, and indolin-2,3-dione.

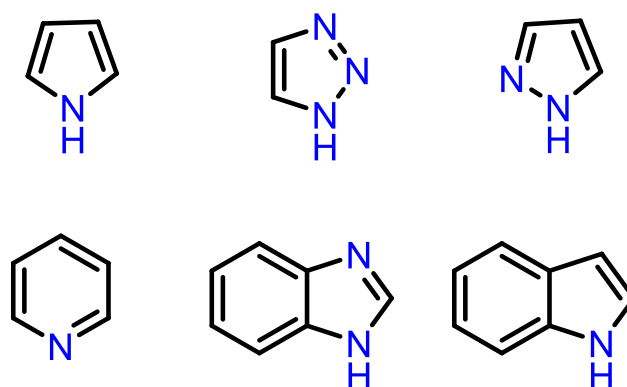


Figure 1.1 Some important group of organic compounds having *N*-heterocyclic.

1.2.1 Five-membered cyclic nitrogen-containing compounds

1.2.1.1 Pyrrole

Pyrrole is categorized as a heterocyclic aromatic molecule with five ring members in chemistry. The air quickly turns this colourless, volatile liquid black. Larger macromolecules, such as heme porphyrins, chlorophyll, chlorins, etc., all rely on pyrrole as a building block. It was initially spotted by F.F. Runge in 1834, when he was analysing coal tar. It was first extracted in 1857 from bone pyrolysate. The reaction used to detect it a red colour that it imparts to wood when moistened with hydrochloric acid gave rise to its name, which comes from the Greek word pyrroles (fiery) [21].

As components of cofactors and natural products, pyrrole derivatives are ubiquitous in biology. Many naturally occurring compounds include pyrrole, including vitamin B12, bile pigments including bilirubin and biliverdin, heme porphyrins, chlorophyll, chlorins, bacteriochlorins, and porphyrinogens. In 1929, E. Fischer created haemin, one of the earliest examples of a molecule containing the pyrrole [22]. Polymerization catalysts, corrosion inhibitors, preservatives, resin solvents, and terpenes are some of the many ways pyrroles can be used.

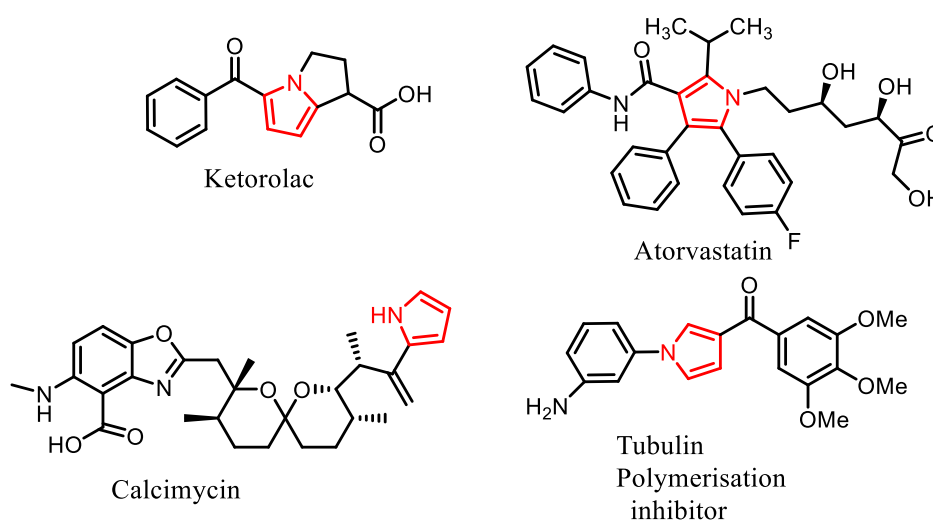
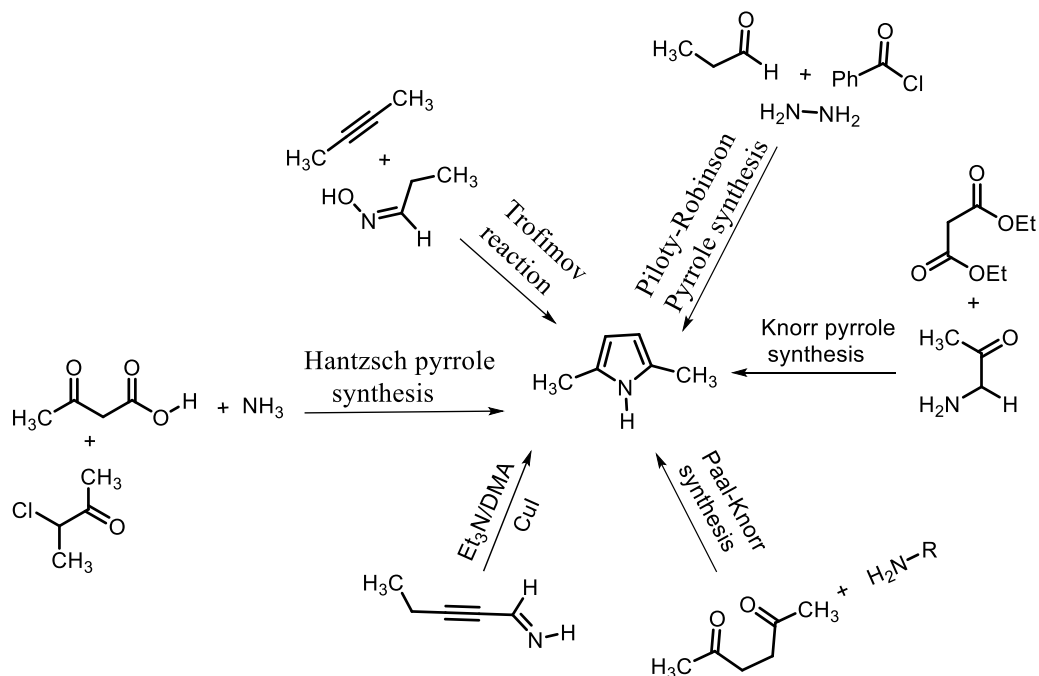


Figure 1.2 Selected biological active molecules *N*-containing pyrrole scaffold.

They are used as catalysts for uniform polymerization in transition metal complexes, luminescence chemistry, spectrochemical analysis, and metallurgical processes, among other applications. Further, certain chemicals are crucial intermediates in synthesizing physiologically significant naturally occurring alkaloids and synthetic heterocyclic derivatives [23].

Pyrroles can be produced using a variety of methods, such as the Knorr pyrrole synthesis, which involves reacting an α -amino-ketone with ethyl acetoacetate, ketones or secondary alcohols, and β -amino alcohols. Another method is the Paal-Knorr Synthesis, which involves reacting a 1,4-dicarbonyl compound with ammonia or aromatic/aliphatic amines. The three-component condensation method of benzoyl chloride, hydrazine hydrate, and aldehyde yields substituted pyrroles known as "Hantzsch pyrrole synthesis." Another method, known as the Trofimov reaction, involves reacting oxime with alkynes [24] in

Scheme 1.1.



Scheme 1.1 Pyrrole and its derivative synthesis.

1.2.1.2 Triazoles

Research into triazole heterocyclic compounds is particularly appealing because of their potential uses as pharmaceuticals, agrochemicals, synthetic materials, artificial acceptors, supramolecular ligands, biomimetic catalysts, etc. The triazole ring is a three-nitrogen-atom-containing, five-membered heterocycle that is both aromatic and electron-rich. This one-of-a-kind design provides triazole derivatives that can bind to several different enzymes and receptors in a biological system through relatively modest interactions like coordination chemistry, hydrogen bonding, ionic and cation dipoles, and so on factors such as stacking, hydrophobic effect, van der Waals force, etc. This means they are capable of various biological processes [25].

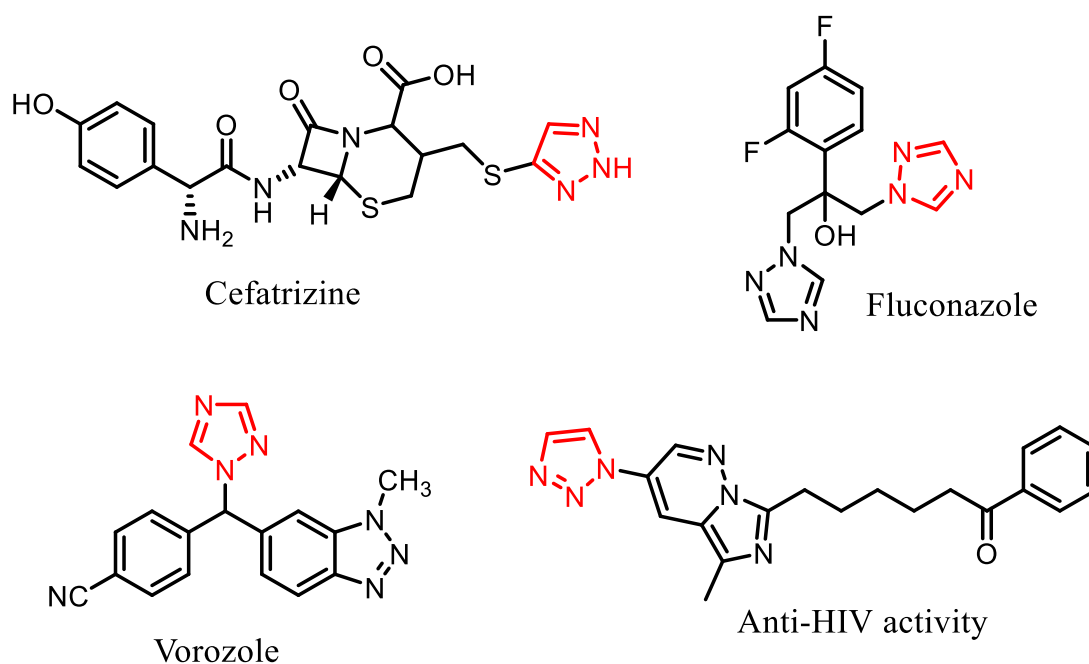


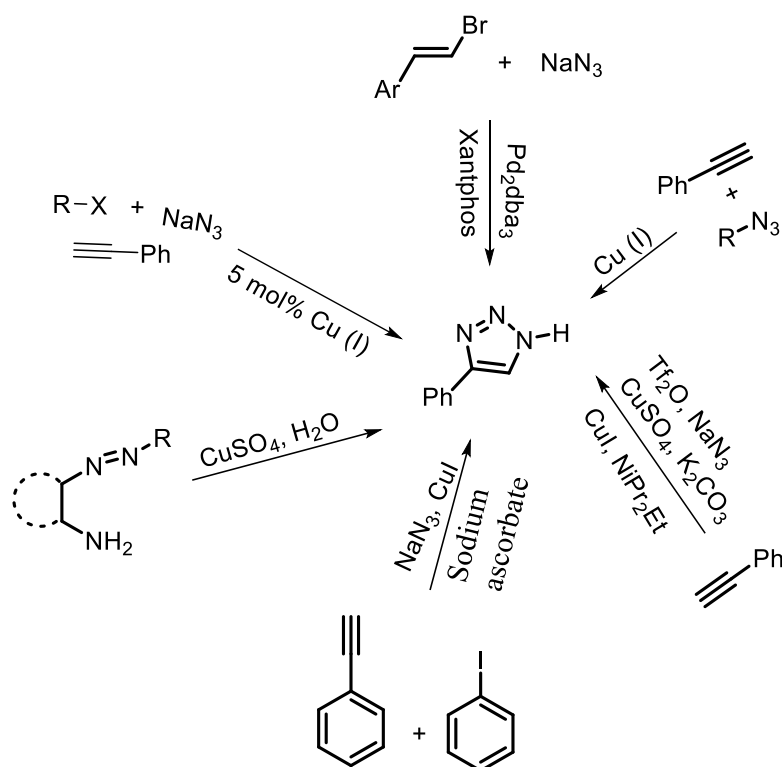
Figure 1.3 Selected biological active molecules *N*-containing triazole scaffold.

It is vital to consider the triazole ring as an isostere of the imidazole, oxazole, pyrazole, thiazole, amide moiety, etc., while developing new pharmaceuticals. As one of the most active areas in drug discovery research, triazole-based derivatives have been the subject of significant preparation and investigation for their biological properties. In particular,

triazole compounds as antifungal medications have been playing an essential role in clinical therapy and are typically the treatments of choice for the fungal infections.

Triazole compounds are widely used clinically because of their potent therapeutic benefits, low toxicity, and low incidence of side effects. Because of the extensive studies done on them and the fact that this is currently the most active area in the development of antifungal drugs, many triazole antifungal drugs are commonly used in the clinic and play a crucial role in the treatment of fungal infections [26]. presence of copper immobilized on 3-aminopropyl functionalized silica gel and ethanol [27].

One-pot synthesis of triazole-linked glycoconjugates using 1,3-dipolar cycloaddition with Cu(I) acting as a catalyst has been found to be highly efficient. Primary aliphatic amines can undergo diazo transfer to produce azides, which can then be converted into triazoles under the proper reaction circumstances. Condensed triazoles can be produced [28] by oxidizing aryl azo heterocycles with an amino group in the ortho position, in **Scheme 1.2**.



Scheme 1.2 Triazole synthesis and its derivatives.

1.2.1.3 Pyrazole

Heterocyclic compounds with nitrogen and their derivatives have been widely used as medicinal medicines throughout medical history. Pyrazole, a five-membered ring with two nitrogen atoms, is aromatic and offers various functionalities and stereochemical complexity. Over the past decade, a wealth of information has been reported on the various pyrazole derivatives and their numerous physiological and pharmacological properties. Moreover half of all known organic compounds are heterocycles, making this group of substances exceedingly important and singular. Heterocycles also exhibit a wide range of physical, chemical, and biological properties, including reactivity and stability [29].

Natural products such as vitamins, hormones, antibiotics, and alkaloids, as well as medicines, agrochemicals, and colours, all contain heterocyclic building blocks that play an important role in metabolism [30]. Important physiological and pharmacological effects are also recognized for a wide variety of synthesized heterocyclic compounds [48]. Due to their widespread application, pyrazoles are the subject of interest of chemist, which focuses on the synthesis and biological activity of several pyrazole derivatives [31].

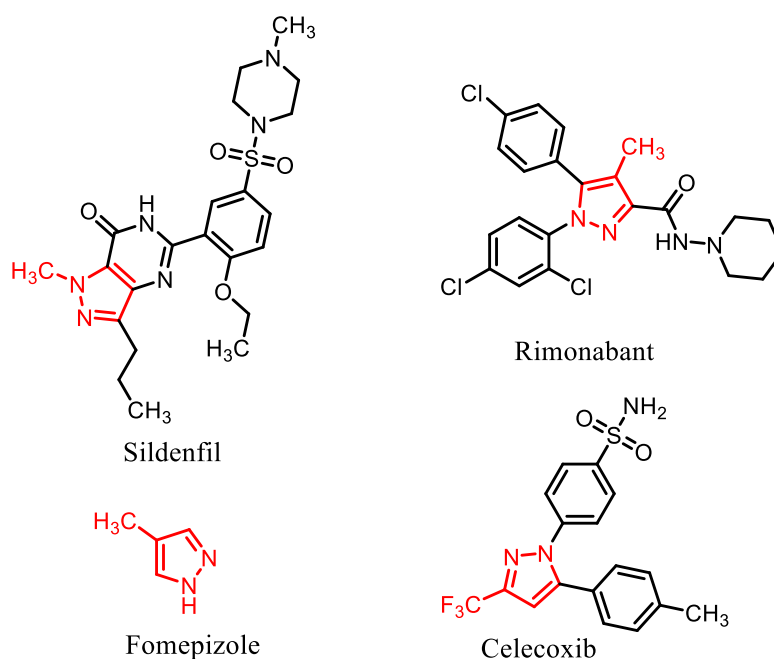
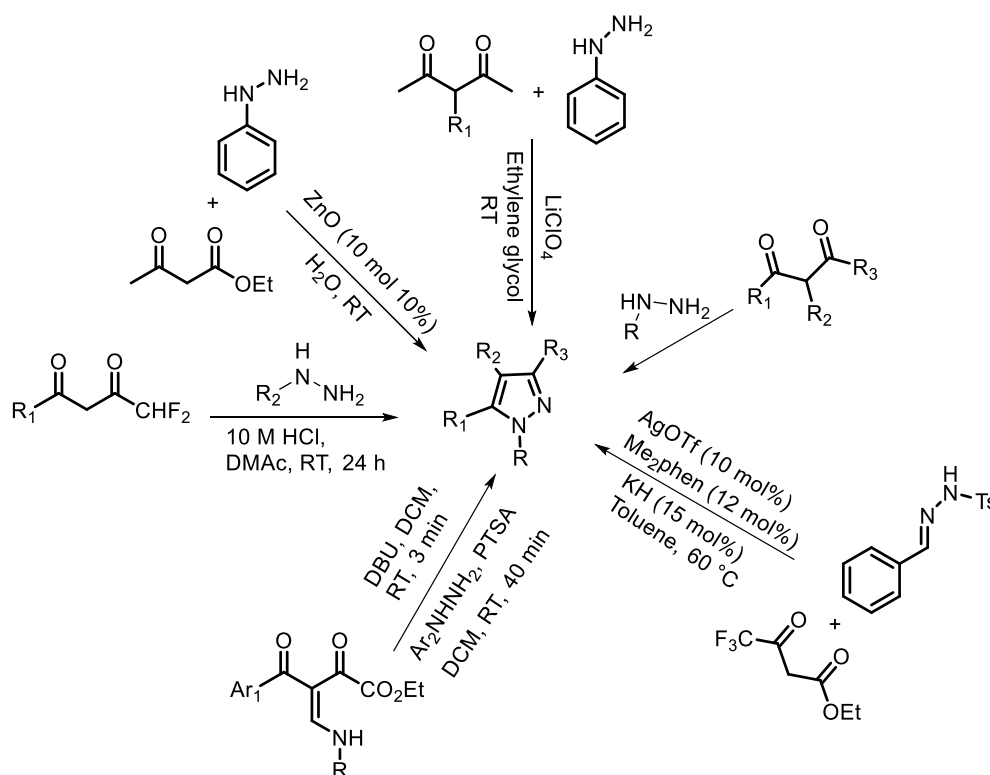


Figure 1.4 Selected biological active molecules containing pyrazole scaffold.

Recently, a number of medications based on pyrazole derivatives have been created. Among these are the cyclooxygenase-2 (COX-2) inhibitor celecoxib, the cannabinoid receptor agonist rimonabant, the alcohol dehydrogenase inhibitor fomepizole, and the phosphodiesterase inhibitor sildenafil [32].

Many well-known natural compounds exhibit various physiological, pharmacological, and poisonous properties because they contain pyrazole moieties. Pyrazoles can be easily and quickly prepared by cyclocondensation of hydrazine derivatives with 1,3-dicarbonyl compounds. The first synthesis of substituted pyrazoles was completed in 1883 by Knorr and his associates. A sustainable method of producing pyrazole derivatives was developed by Konwar *et al.*, using lithium perchlorate as a Lewis acid catalyst [33, 34] in **Scheme 1.3**.



Scheme 1.3 Pyrazole synthesis and its derivatives.

1.2.2 Nitrogen-containing six-membered cyclic compounds

1.2.2.1 Pyridine

Moreover, half of organic chemistry is devoted to nitrogen-containing heterocyclic chemistry. Due to various vital reasons, pyridine is recognized as a heteroaromatic chemical with numerous powerful biological characteristics. Coal tar, a rich natural supply of pyridine, was distilled to produce large quantities of the chemical [35]. Pyridine is a component of many other significant substances, including pyridoxine (vitamin B6), niacin (vitamin B3), and several alkaloids (quinine, nicotine, etc.) [42]. Pyridine, a heterocyclic molecule with six carbon atoms, has many uses and is widely used as a solvent, reagent, pharmaceutical and agrochemical precursor. Due to the growth in drug resistance, there is a clear medical need to create new antiviral medicines. Many pyridine scaffold derivatives have impressive biological activity [36].

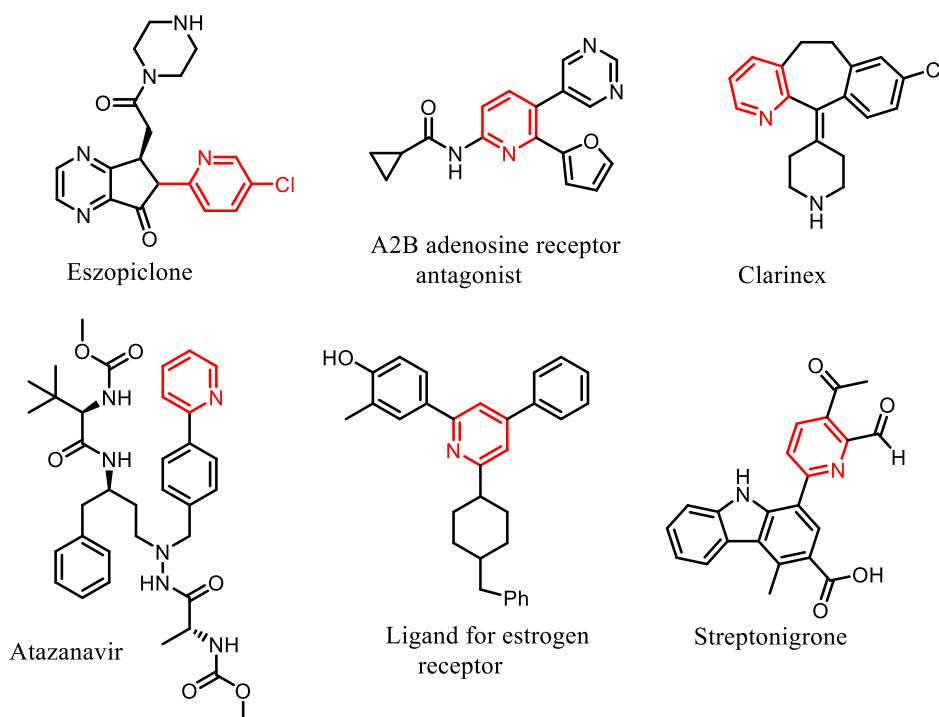
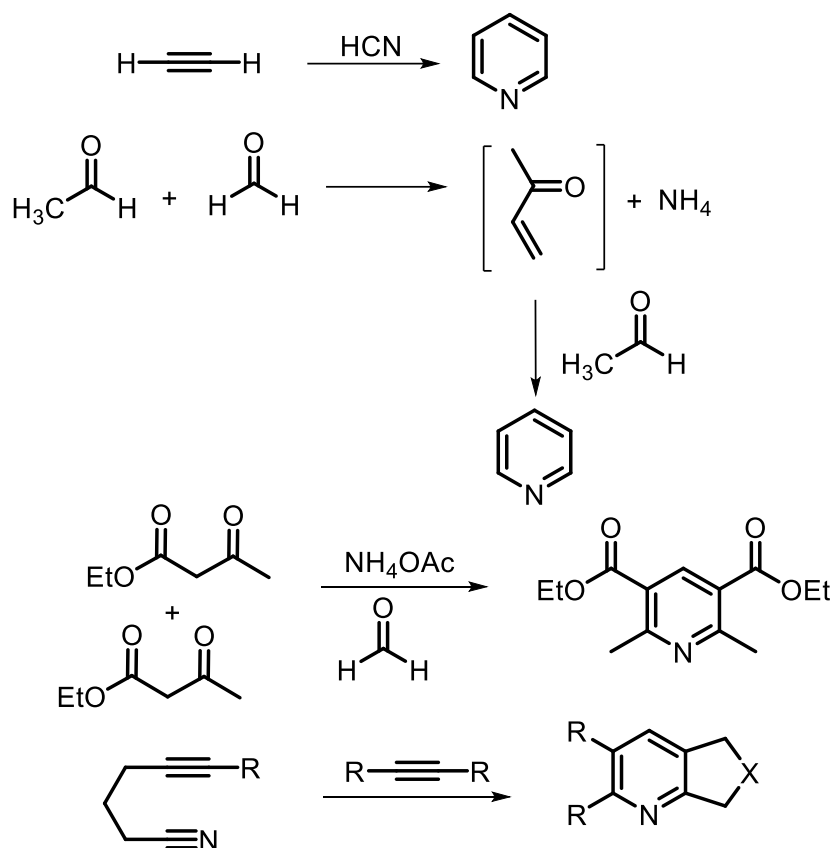


Figure 1.5 Selected biological active molecules *N*-containing pyridine scaffold.

In 1876, acetylene and hydrogen cyanide were used to create pyridine for the first time [43]. The Chichibabin synthesis is still in use because it can produce pyridine. Through the Knoevenagel condensation process, aldehyde and formaldehyde conduct a reaction that releases acrolein. Following condensation with acetaldehyde and ammonia,

dihydropyridine is formed from acrolein[37]. The dihydropyridine is oxidized with the use of a solid-state catalyst, which ultimately results in the creation of pyridine. An aldehyde, β -keto ester (2 equivalents), and a nitrogen donor (ammonium acetate or ammonia) are required for the Hantzsch pyridine synthesis, which is a multi-component organic reaction [38]. By combining alkyl nitriles and alkynes, pyridine is produced in **Scheme 1.4**.



Scheme 1.4 Pyridine synthesis and its derivatives.

1.2.3 Nitrogen-containing fused heterocyclic compounds

1.2.3.1 Benzimidazole

Benzimidazole is a nitrogen-containing heterocyclic structure made up of a six-membered benzene ring fused with a five-membered imidazole ring. Ansari et al. (2009), Kazmierczuk et al. (2002), and Grocer et al. (2002) all found that benzimidazole derivatives were remarkably effective compounds for their inhibitory activity and their favorable selectivity ratio. Due to importance of benzimidazole and oxadiazole nucleus, it

was felt that it would be helpful to design and synthesize some new benzimidazole derivatives containing oxadiazole moiety and screen them for possible biological activities [39].

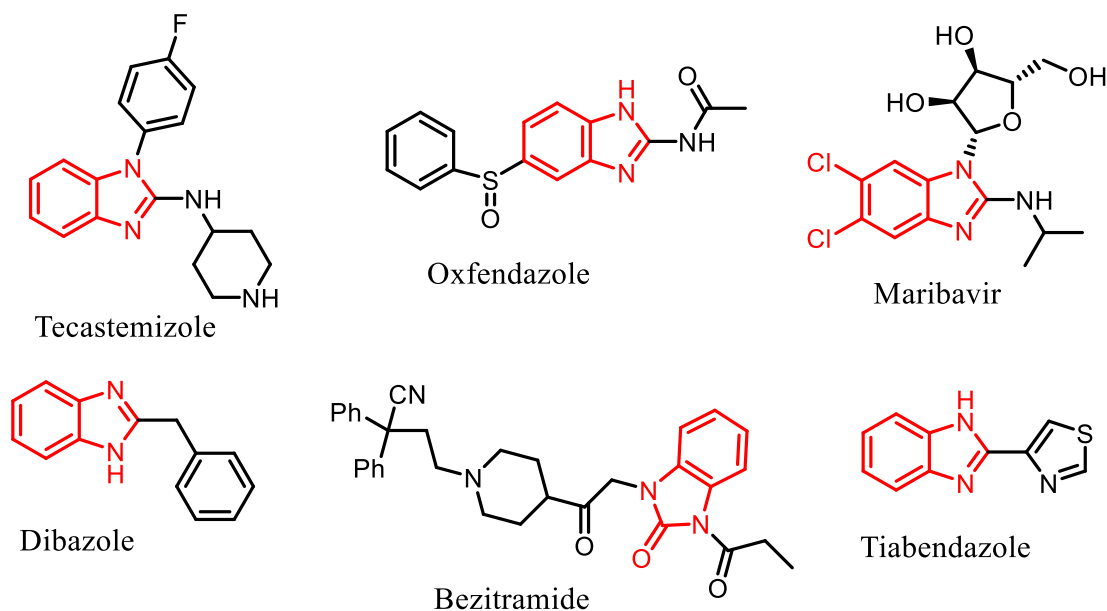
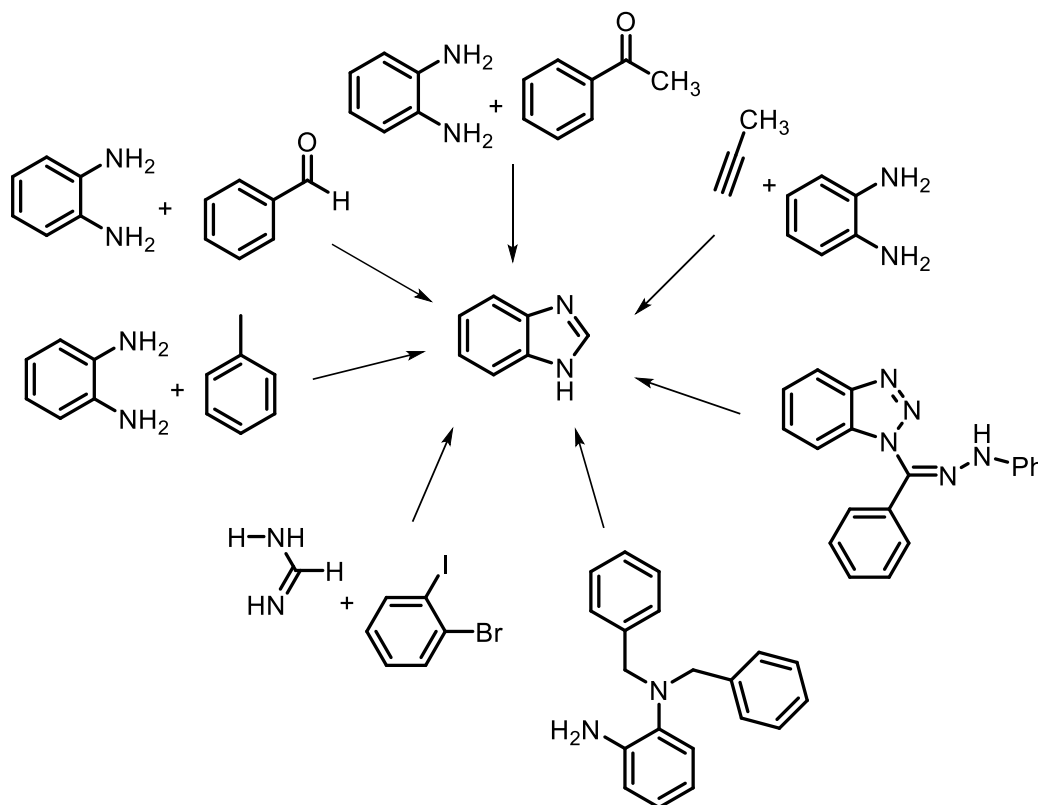


Figure 1.6 Selected biological active molecules *N*-containing benzimidazole scaffold.

The imidazole moiety is at the center of many pharmaceutically and biologically active chemicals and natural products. Numerous biologically active chemicals rely on benzimidazoles and their derivatives. Important biological targets, including DNA minor grooves, histamine receptors, β -tubulin, and serotonin receptors, are interacted with by benzimidazole derivatives [40] in **Scheme 1.5**.



Scheme 1.5 Imidazole synthesis and its derivatives.

1.2.3.2 Indole

A wide variety of natural compounds contain indole derivatives because of their vast spectrum of biological activity. Parallel to the study of indigo dye, indole chemistry came into focus. One proposed route involves the breakdown of indigo into isatin and the subsequent synthesis of an oxindole derivative. Numerous biological effects, such as those on inflammation, convulsions, the cardiovascular system, and bacteria, have been attributed to indole derivatives. Since indole and its derivatives have many useful biological properties, they are actively being used in drug discovery [41].

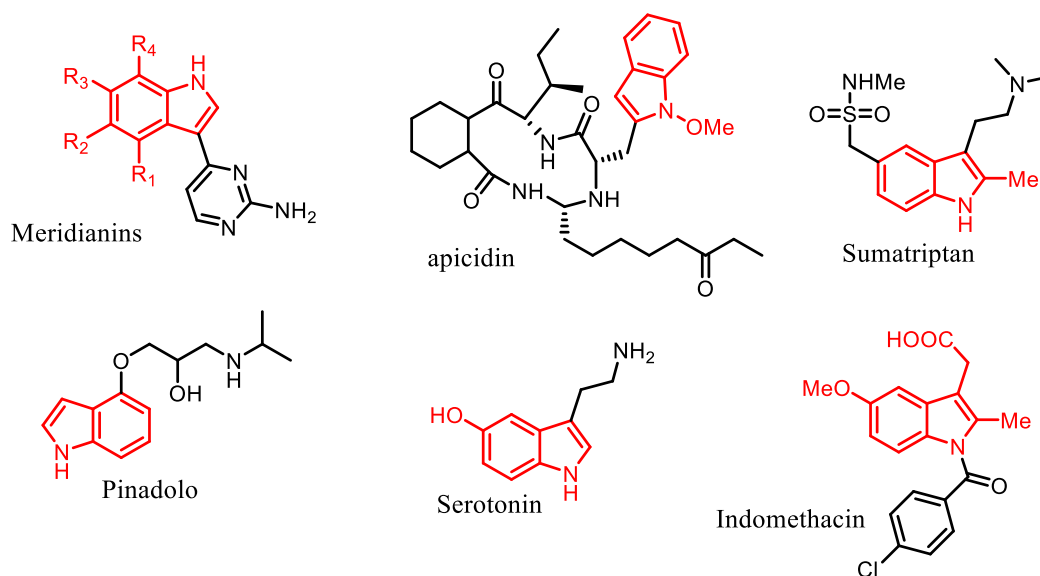
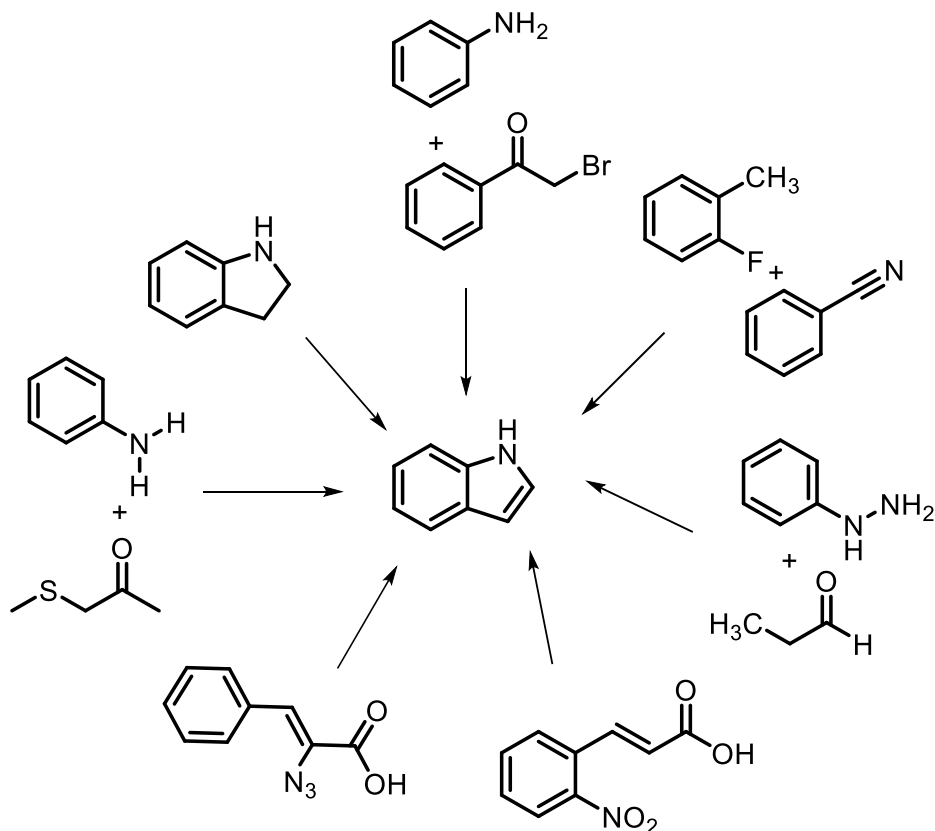


Figure 1.7 Selected biological active molecules containing indole scaffold.

Natural products containing indole derivatives are abundant, in a wide range of plant, animal, and marine species. Scientists have studied the indole core for decades because it is one of the most promising scaffolds for developing new drugs. Heterocyclic nitrogen compounds are abundant in bioactive molecules and are, therefore, excellent drug candidates [42]. Therefore, throughout the past few decades, a great deal of effort has been put into synthesizing and exploring the diverse medicinal potential of this moiety.

There are a number of ways to synthesize indole and its derivatives. Among these techniques is the so-called "Fischer indole synthesis" which calls for the acidic interaction of phenylhydrazine with carbonyls (aldehyde or ketone) [43]. The "Bischler-Mohrlau indole synthesis" utilizes alpha-bromo-acetophenone and excess aniline to create 2-aryl-indole. In addition to the "Gassman indole synthesis," which involves the addition of aniline and a ketone bearing a thioether substituent, indole synthesis also makes use of the dehydrogenation of indolin stimulated by potassium tertiary butoxide [44] in **Scheme 1.6**.



Scheme 1.6 Indole synthesis and its derivatives.

1.2.3.3 Pyrazolo[1,5-a] pyridines

One of the traditional classes in synthetic chemistry still developing is heterocycles. Heterocyclic science is the field in which most natural themes are found. In both natural and artificial contexts, heterocyclic motifs are discovered to be extremely significant. Under biological action, heterocycles are innate in the majority of natural products. Excellent biological activity is exhibited by both synthetic and natural heterocycles, which include herbicide, fungicide, insecticide, anti-tumor, anti-toxin, antidepressant, anti-HIV, microbial, antibacterial, and antiviral properties [45].

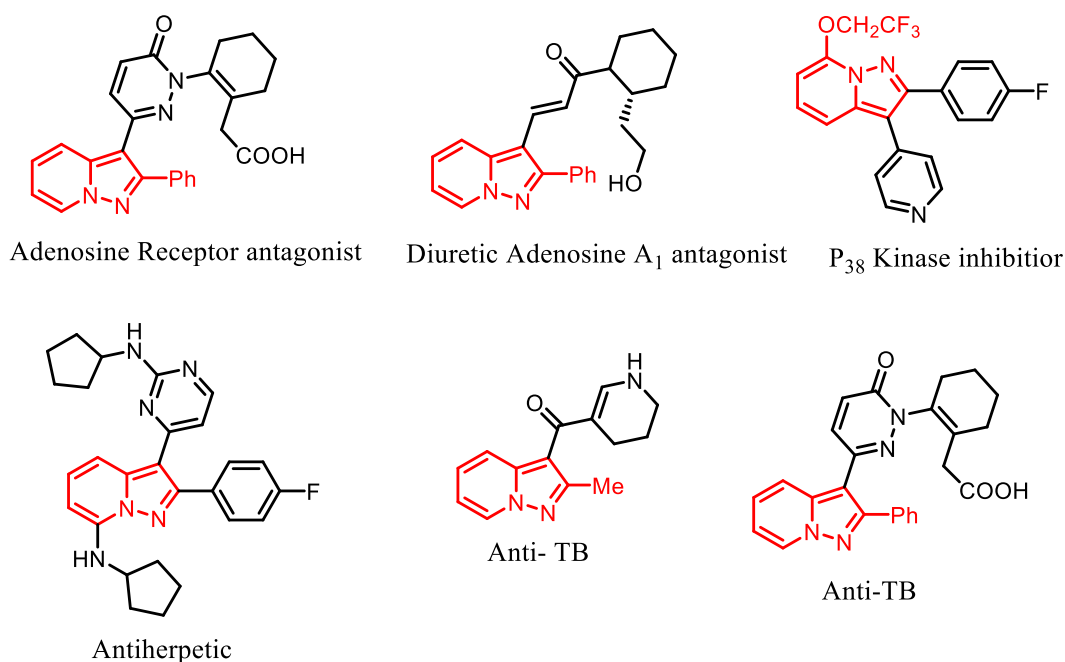
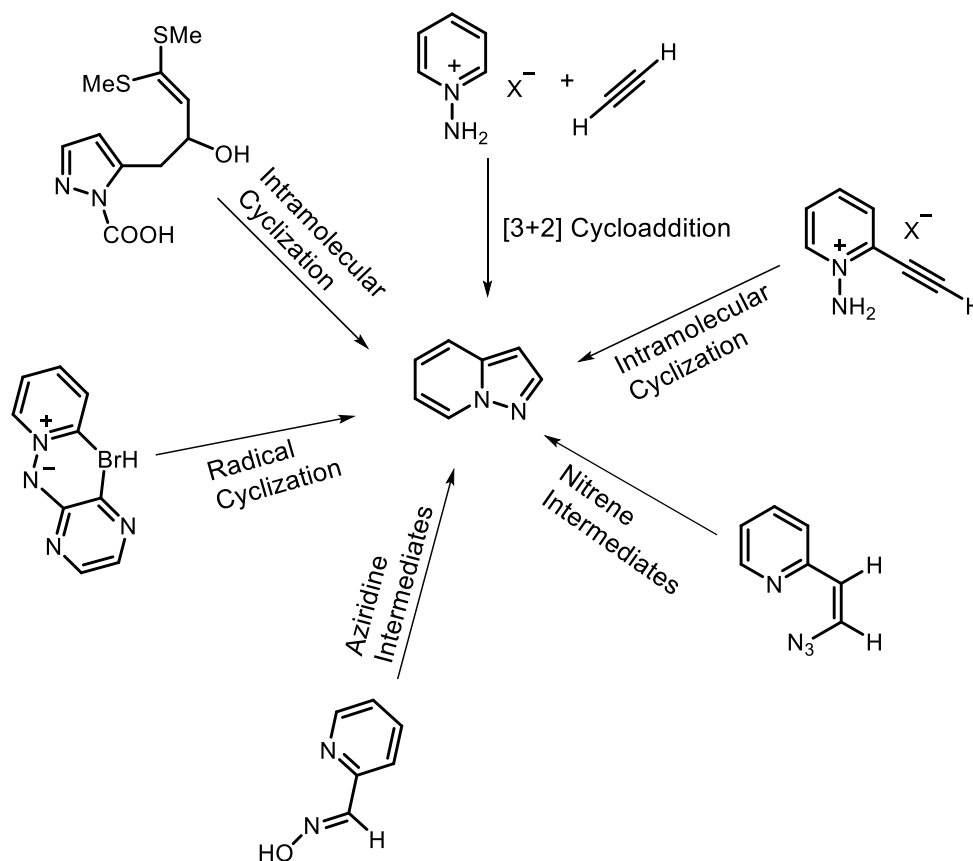


Figure 1.8 Selected biological active molecules containing pyrazolo[1,5-a] pyridines scaffold.

Pyrazolo[1,5-a] pyridines were made with copper catalysts in a single pot. Benzonitriles and pyridyl acetate were used in the [3 + 2] cyclization approach that drove the reaction forward, DMSO used as a solvent in an argon atmosphere. Cheung and colleagues synthesized a novel pyrazolo[1,5-a] pyridine motif with a ring junction. It has also been shown to be a powerful and new p38 kinase inhibitor. Compound 5-methyl-3-(4-fluorophenyl)-2-propynoate was obtained by cyclization reaction with appropriately substituted *N*-aminopyridines, yielding pyrazolo pyridine [46].

Hoashi et al., used thermal intermolecular [3 + 2] cyclization to prepare pyrazolo[1,5-a] pyridines with alkyne and *N*-aminopyridine analogy. Regioselective isomers were produced as a byproduct. Compound first produces a low-yielding product. Numerous studies offer the ideal reaction conditions that produced a high yield. The ideal conditions for obtaining pyrazolo[1,5-a] pyridines are thought to be refluxing at 80 °C in an acetic acid medium [47] in **Scheme 1.7**.



Scheme 1.7 Synthesis of Pyrazolo [1,5-a] pyridine.

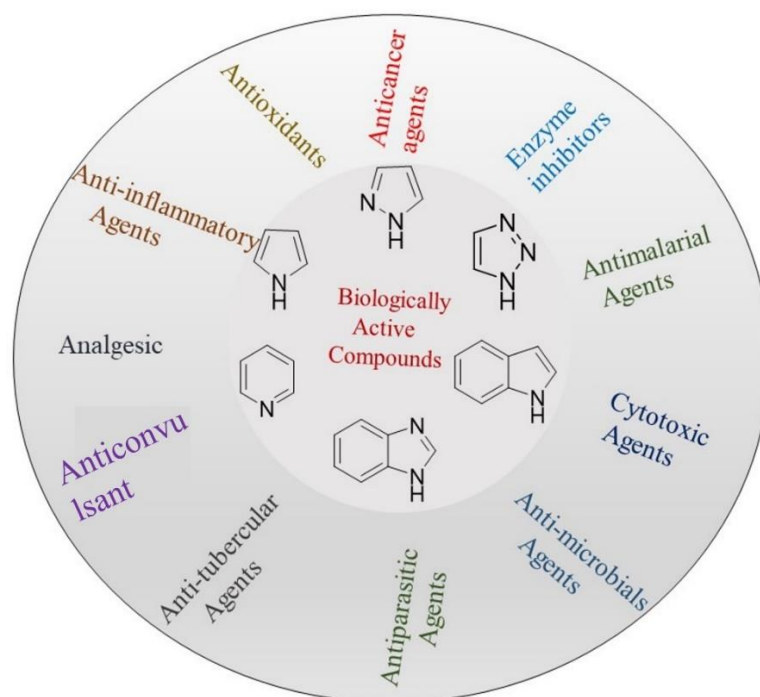


Figure 1.9 These are *N*-containing heterocyclic compounds show biological activity.

1.4 Different synthetic route -

1.4.1 Conventional Method

1.4.2 Photochemical Synthesis

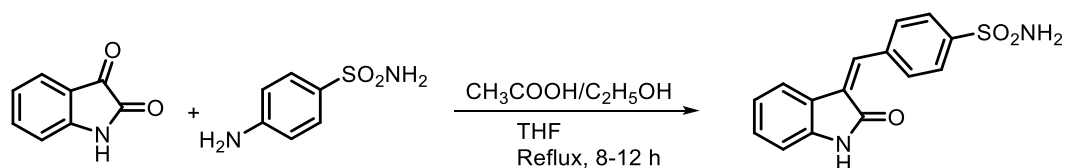
1.4.3 Microwave-Assisted Reaction

1.4.4 Electrochemical method

1.4.1 Conventional Method

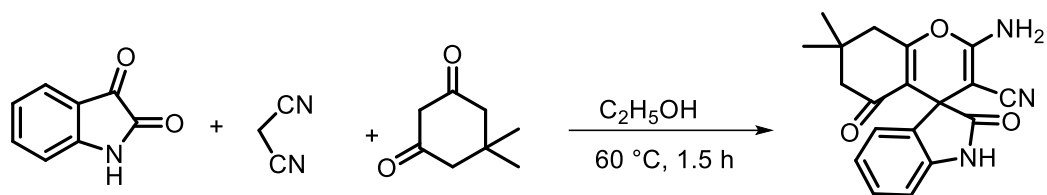
There has been a surge of interest in recent years in the fast synthesis made possible by "conventional sources of energy" refers to those that can only be used once before being depleted. These are the primary options for using conventional energy. Most substrates and reaction types can be used with conventional procedures. This adaptability helps scientists deal with a wide range of compounds and makes them useful for a number of synthetic applications. Traditional approaches are still relevant because of their proven track record, adaptability, low cost, cutting-edge methods complement one another, giving synthetic chemists more options .

Under the influence of conventional irradiation, Mohan et al., devised a rapid methodology to synthesize carbamimidoyl-3-(2-oxoindolin-3-ylidene amino) benzene sulfonamide [50].



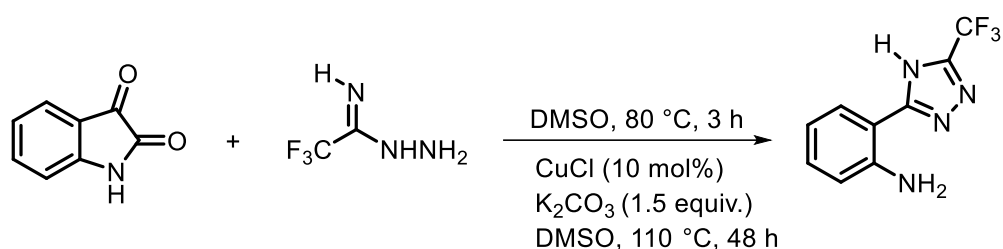
Scheme 1.8 Synthesis of carbamimidoyl-3-(2-oxoindolin-3-ylidene amino) benzene sulfonamide.

Under the influence of the conventional method in EtOH, Nath and colleagues introduced a novel class of functionally diverse spiroindoles utilizing isatin, malononitrile, and dimedone [51].



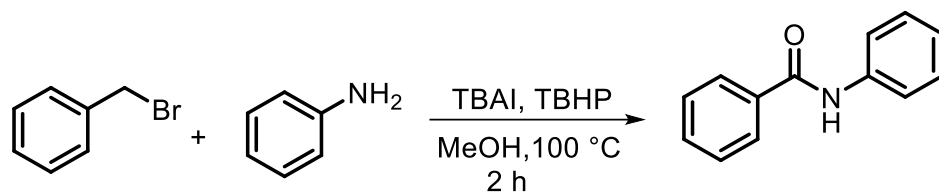
Scheme 1.9 Synthesis of spiroxindoles.

Under the influence of the conventional method in DMSO, Xia, Wu and colleagues developed a new method for synthesizing trifluoroacetimidohydrazides [52].



Scheme 1.10 Synthesis of 2-heterocyclyl-anilines.

Sundaram *et al.*, 2022 describe that by oxidatively coupling benzyl bromides with amines, TBAI-catalyzed C–N bond formation opens up a novel path for the synthesis of amides on the chemical level.



Scheme 1.11 Synthesis of benzamide.

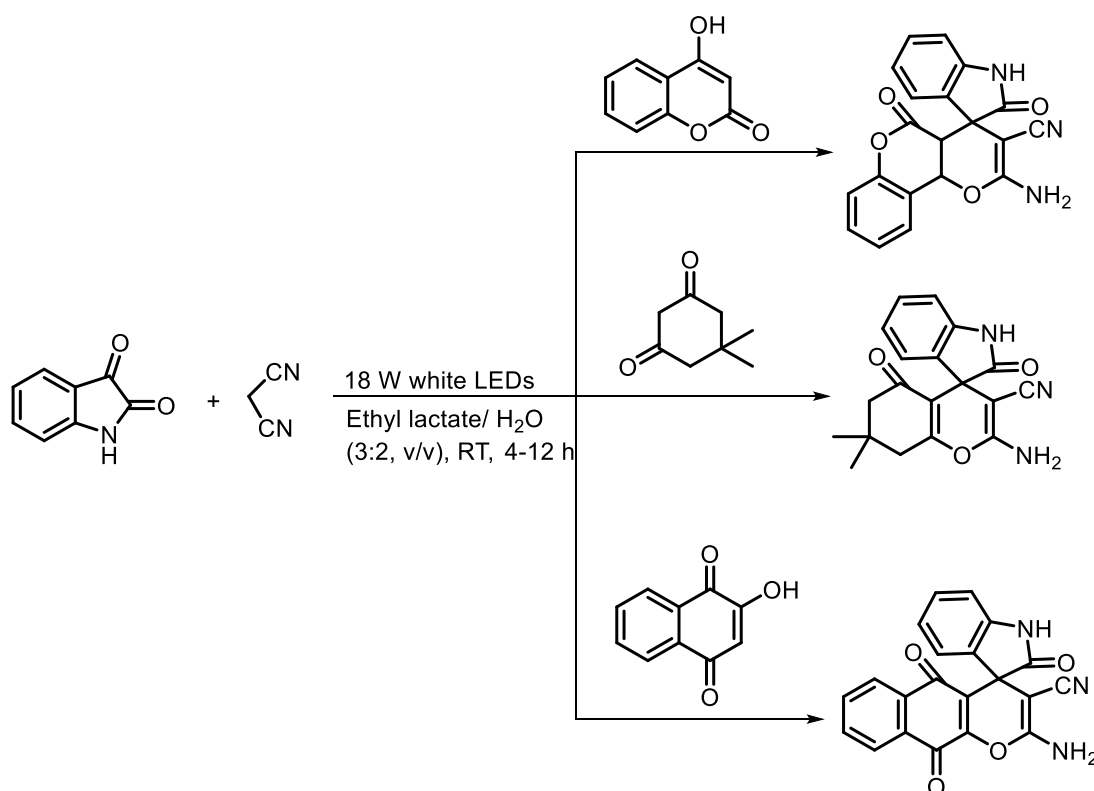
1.4.2 Photochemical Synthesis

As scientists become more aware of the pollution and waste generated by chemical processes in industrial and laboratory settings, the importance of green chemistry becomes clearer. As a result, they are increasingly interested in finding greener, more cost-effective, and less harmful alternatives to traditional methods, such as green catalysts, ecologically

friendly solvents, and conditions that render the use of solvents and catalysts excessive [53].

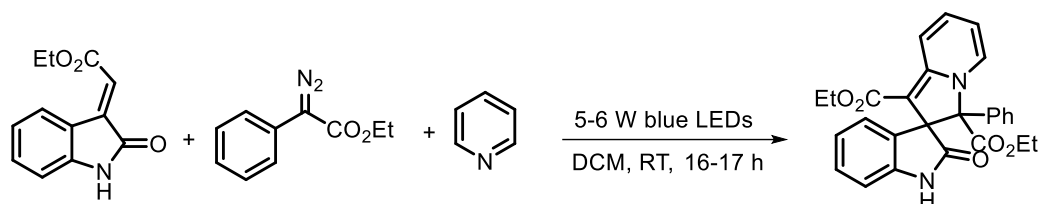
The creation of desired products in most chemical processes requires the use of an appropriate catalyst [54]. The absence of catalysts is very appealing, especially in industry and pharmaceuticals, and while the use of stimuli can mitigate some disadvantages of chemical processes, this is not the case for the absence of catalysts. However, more efficient and cost-effective options must be investigated if we are to achieve reaction outputs without catalysts [55].

Under the influence of using visible-light-initiated catalyst-free conditions, Wu, Zhang, and colleagues established a very successful three-component method for the construction of spirooxindole-pyran derivatives [56, 57].



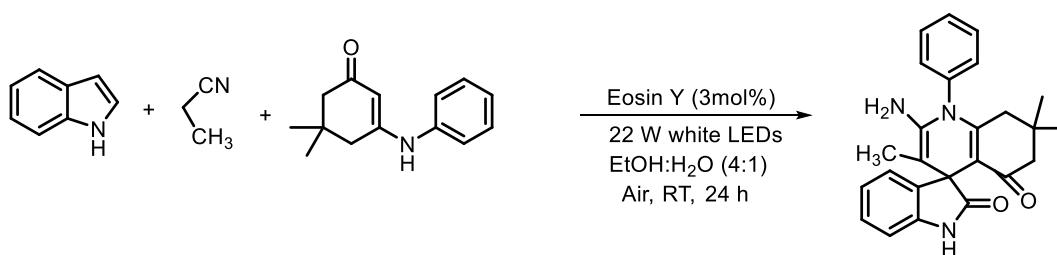
Scheme 1.12 Synthesis of spirooxindole-pyran.

Sen and colleagues found a metal-free, visible-light-promoted, three-component synthesis involving 3-alkenyloxindoles, aryl diazoesters, and pyridine that yields moderate-to-good amounts of spirocyclic dihydroindolizine-oxindoles in 2021 [58].



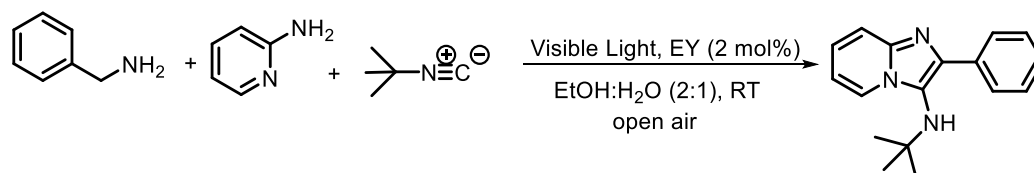
Scheme 1.13 Synthesis of spirocyclic dihydroindolizine-oxindoles.

Recently, Sundaram and co-workers discovered an effective approach for the synthesis of spiro-[indoline-3,4-quinolines] via multicomponent reaction, exploiting visible light organo photoredox catalysis [59].



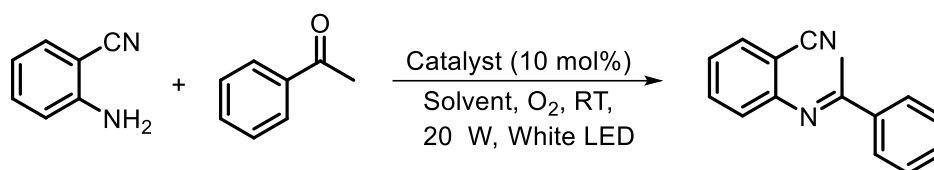
Scheme 1.14 Synthesis of spiro-[indoline-3,4-quinolines].

Sundaram and co-workers demonstrated in 2020 that 3-aminoimidazo[1,2-a]pyridines are synthesized via the HAT process under visible light by forming the C–N bond. Eosin Y is the catalyst [60].



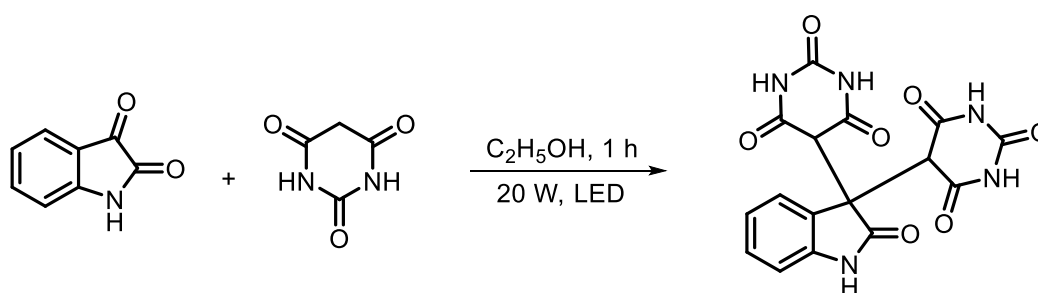
Scheme 1.15 Synthesis of 3-aminoimidazo[1,2-a]pyridines.

Sundaram and co-workers suggested in 2021 the Schiff's base of 2-amino Benzonitrile Derivatives and acetophenones: a Cu-catalyzed synthesis induced by visible light [61].



Scheme 1.16 Synthesis of azomethine chromophores.

Sundaram and co-workers demonstrated in 2022 that photocatalyst-free condensation of barbituric acid with carbonyl compounds mediated by visible light [62].



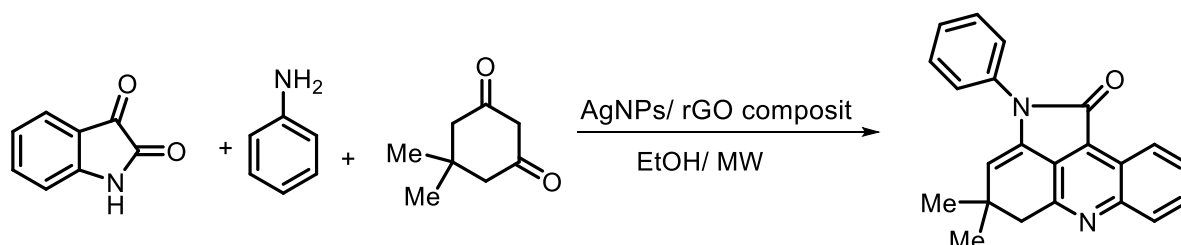
Scheme 1.17 Synthesis of dibarbiturates of oxindole.

1.4.3 Microwave-Assisted Reaction

Microwave irradiation has become an increasingly popular and valuable tool in the field of organic chemistry because it provides a novel energy source for initiating reactions. The excellent heating capabilities of microwaves typically result in a considerable improvement of reaction rates and a substantial reduction in reaction time. Recent studies of chemical synthesis utilizing microwave irradiation have shown that these effects can be largely due to microwaves' dielectric heating characteristics. At the same time, some research suggests that the non-thermal effects of microwaves can change reaction dynamics and reduce the activation energy of organic reactions [63].

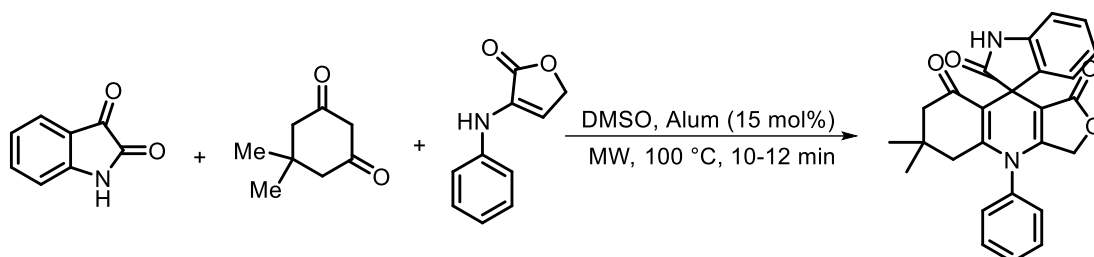
Under the influence of microwave irradiation, Amit Sharma and co-workers the synthesis of highly stable and recyclable AgNPs/rGO composite and its catalytic applications for the

chemo selective synthesis of pyrrolo[2,3,4-*kl*]acridin-1-ones by one pot reaction of dimedone, various anilines and isatins *via* ring opening sequence and intramolecular cyclization under microwave irradiation [64].



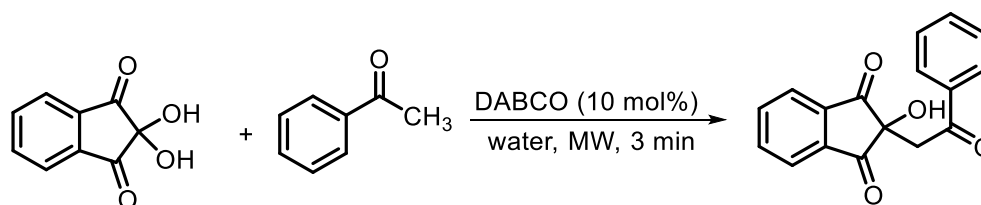
Scheme 1.18 Synthesis of pyrrolo[2,3,4-*kl*]acridin-1-ones.

Naeimi and co-workers demonstrated the derivatives of 6,6-dimethyl-4-phenyl-6,7-dihydro-1*H*-spiro[furo[3,4-*b*]quinoline-9,3'-indoline] 1,2,8(3*H*,4*H*,5*H*)-trione were prepared from the one-pot reaction of isatins, dimedone, and anilino lactones [65].



Scheme 1.19 Synthesis of 6,6-dimethyl-4-phenyl-6,7-dihydro-1*H*-spiro[furo[3,4-*b*]quinoline-9,3'-indoline] 1,2,8(3*H*,4*H*,5*H*)-trione.

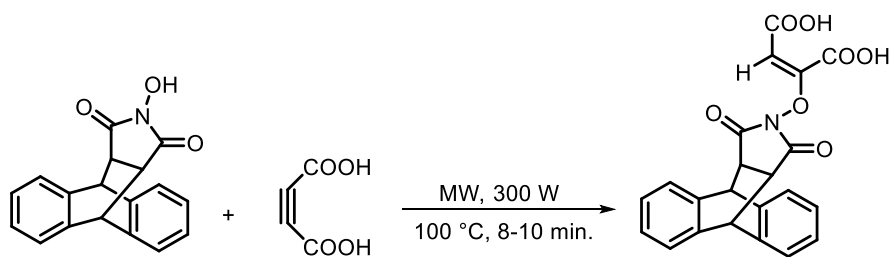
Srivastava *et al.*, suggested in 2019 that microwave-catalyzed DABCO-derivative β -hydroxy ketones from α -methyl ketones and ninhydrin [66].



Scheme 1.20 Synthesis of 2-substituted-2-hydroxy-indan-1,3-diones.

Srivastava *et al.*, approach in 2018 the traditional technique, which produced a combination of syn-(*E*) and anti-(*Z*) isomers, a novel and efficient microwave induced stereoselective

synthesis of *O*-vinyl oximes was devised. This method involved the Michael addition of an *N*-hydroxysuccinimidyl moiety to acetylenic esters in a catalyst-free environment [67].



Scheme 1.21 Synthesis of *O*-vinyl oximes.

1.4.4 Electrochemical method

Electro-organic synthesis is seeing a rebirth in both academic and industrial synthesis as part of the development of more environmentally friendly methods for the creation of organic molecules [68]. Using electricity as a reagent can often eliminate the need for poisonous and/or hazardous oxidants or reducing agents. The result is a significant reduction in discarded reagents. As a result, sustainable chemistry may now be pursued, as electricity can be generated from renewable resources [69].

Electricity is used to drive a chemical and thermal reaction within an electro-organic synthesis [70]. Normally non-occurring organic redox reactions can be compelled to take place. However, for a successful procedure, electrolytic conversion must occur without incident at both the cathode (reduction) and the anode (oxidation) [71]. Since the Gibbs free energy can often be described in terms of the difference in the electrode potentials, this favors the oxidation reaction over the reduction reaction, which has a higher Gibbs free energy ΔG [72].

$$\Delta G = -nF (E_{\text{anode}} - E_{\text{cathode}})$$

An electrochemically activated electrode surface, an electrochemical redox mediator, or a single-electron transfer (SET) can all trigger the transformation. Direct current (DC) or alternating current (AC) can be used to achieve either constant potential (CPE) or constant

current (CCE) conditions, respectively, for reaction control [73]. There has been a rise in interest in electrochemical organic synthesis due to its potential as a green and flexible synthetic platform. Analytical tools that can effectively guide the future design of electro-organic reactions can be developed by performing quantitative assessments of reaction thermodynamics, electro-kinetics, and associated chemical processes [74]. Electrochemistry's potential as a useful tool for quantitative analysis and mechanistic exploration in electro-organic research has been well recognized. For complex electro-organic systems, for instance, a thorough understanding of electro-kinetics, including mass transportation, interfacial electron transfer, and coupled chemical reaction, can be acquired with mature theories and experimental practice of cyclic voltammetry (CV) [75].

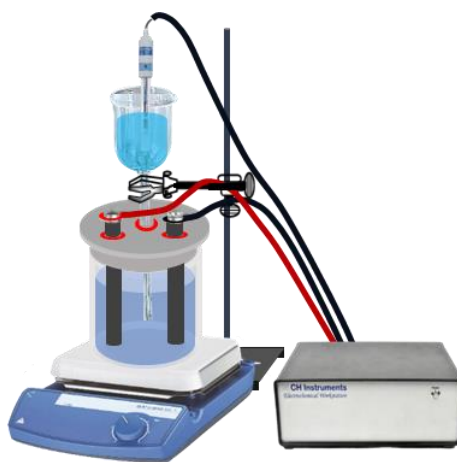
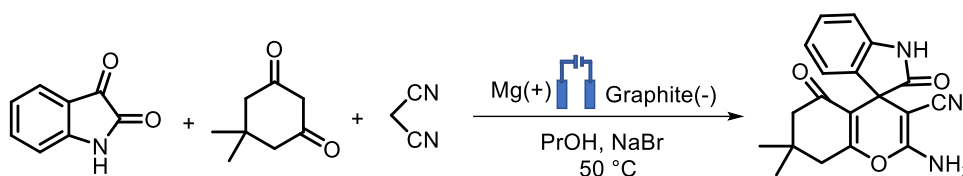


Figure 1.10. Electroorganic synthesis setup.

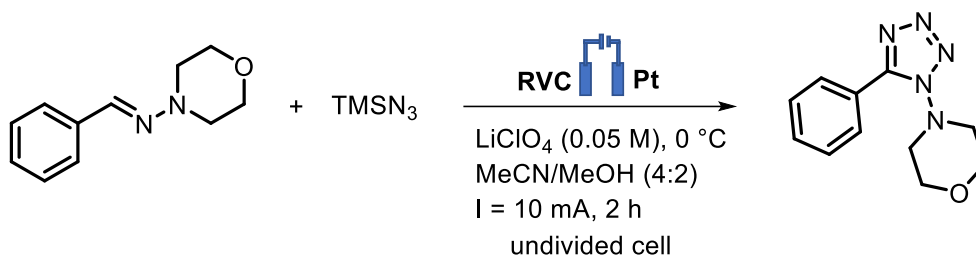
Tabakovic reviewed the synthesis of heterocyclic compounds by anodic oxidation [76], while Lund reviewed the synthesis and functionalization of heterocyclic compounds [77]. Francke compiled literature on the synthesis of heterocycles until 2014 [78], while Baran meticulously documented many electro-organic transformations until 2017 [79].

Most recently, In 2016 Makarem and co-workers developed electrochemical multicomponent cyclization for spiro oxindole derivative [80].



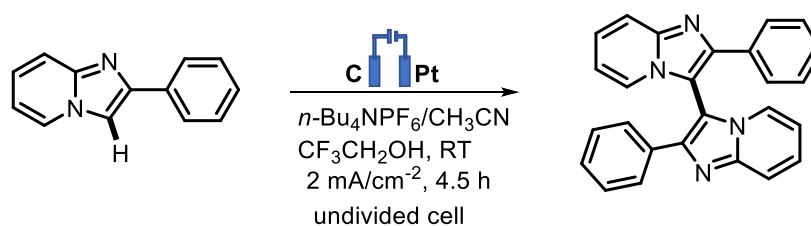
Scheme 1.22 Synthesis of spiro oxindole.

In 2018, Zhang and co-workers reported the Metal- and oxidant-free [3 + 2] cycloaddition of azides with hydrazones for the electrochemical synthesis of tetrazoles. This eco-friendly approach is simple enough to be performed on a gram scale or in a single pot, and it is compatible with a wide range of functional groups [81].



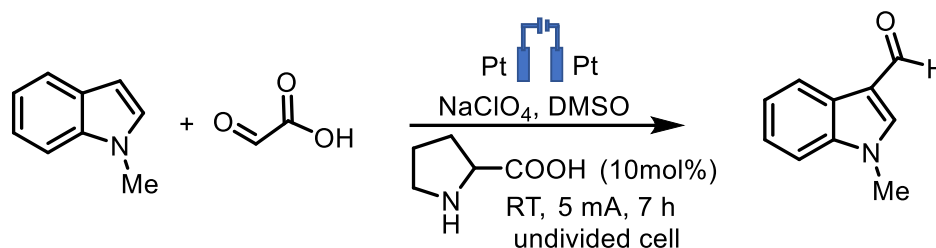
Scheme 1.23 Synthesis of synthesis of tetrazoles.

In 2018, Han and co-workers approach was easy to set up, reliable, and require no expensive catalyst; oxidant-free imidazopyridine heterocycles undergo an electrochemical oxidative homo-coupling process to biheteroaryls. Starting with the reaction described as a regioselective homo-coupling reaction of imidazo-[1,2-a] pyridine in dimethyl sulfoxide (DMSO) at 120 °C, copper is suggested to be the catalyst and 2,2'-bipyridine to be the ligand in this proposed chemistry[82].



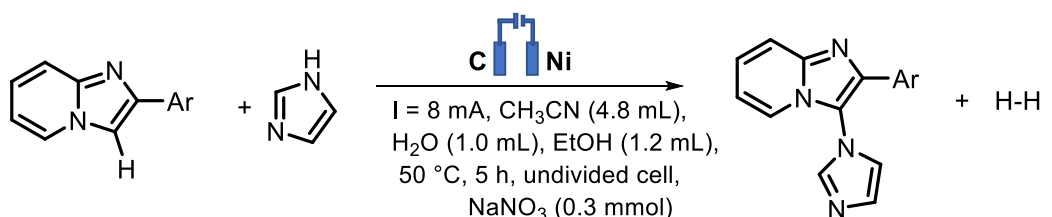
Scheme 1.24 Synthesis of 2,2'-bipyridine.

In 2019, Huang and co-workers reported the synthesis of 3-formylindoles by electrochemical decarboxylation of glyoxylic acid with an amine as a dual function Organocatalyst. Using amine as a dual-purpose organocatalyst and electrochemical decarboxylation of glyoxylic acid, a novel technique for 3-formylation of indoles has been devised [83].



Scheme 1.25 Synthesis of 3-formylindoles.

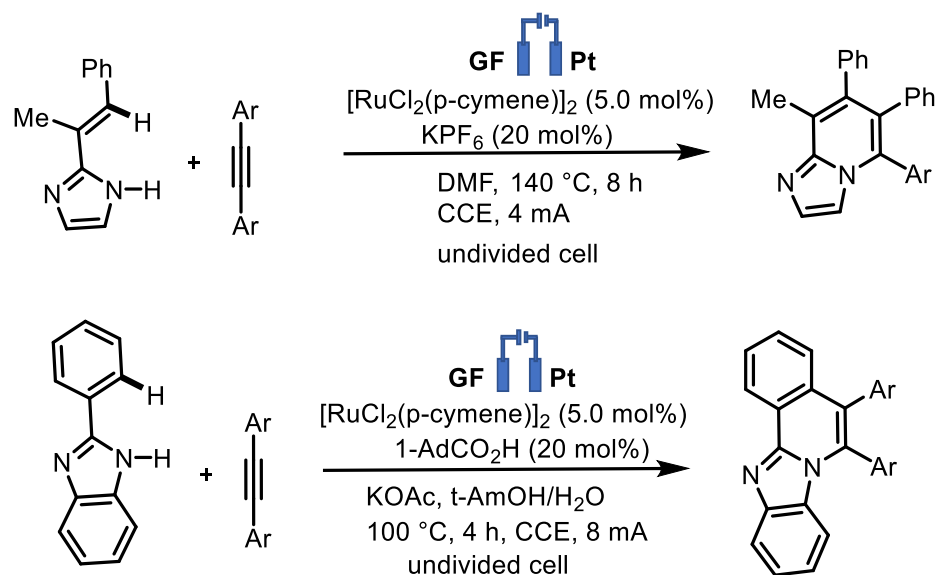
In 2019, Lei and co-workers demonstrated that it start from the reaction of electrochemical oxidative C–H/N–H cross-coupling for the production of C–N bonds and the development of hydrogen. One of the most significant structural components of many physiologically active compounds, especially those that are sold commercially, is an imidazo [1,2-a] pyridine core. An effective and green synthetic technique, electrochemical synthesis, may create C–X bonds by electrochemical oxidative C–H/X–H cross-coupling with hydrogen evolution in the absence of metal catalysts and exogenous oxygenants [84].



Scheme 1.26 Synthesis of imidazo [1,2-a] pyridine.

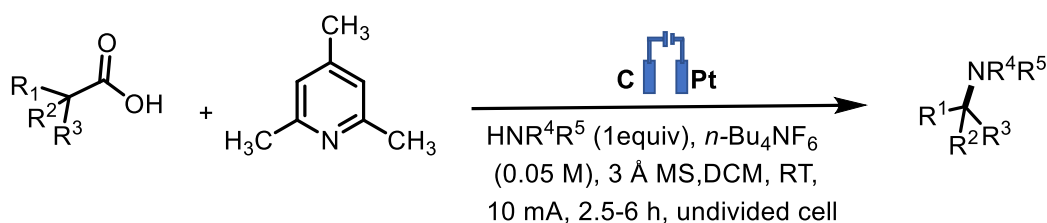
In 2020, Ackermann and co-workers proposed It has been possible to construct several bridgehead *N*-fused via ruthenium-catalyzed electrochemical dehydrogenative annulation reaction of imidazoles with alkynes. by using regioselective electrochemical C–H/N–H

annulation without the use of chemical metal oxidants, bicyclic heteroarenes. We investigated several reaction conditions for the planned electro-oxidative C-H/N-H activation of ruthenium-catalyzed alkenyl imidazole with alkyne in an operationally straightforward undivided cell configuration with a Pt cathode and a GF (graphite felt) anode [85].



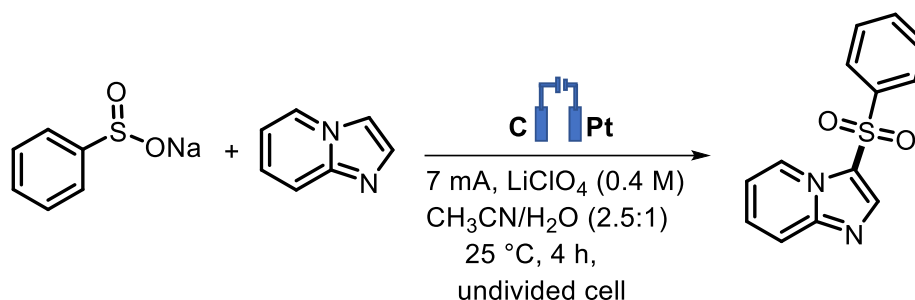
Scheme 1.27 Synthesis of bicyclic heteroarenes.

In 2020, Baran and co-workers demonstrated a straightforward technique for using nonactivated carboxylic acids as alkylating agents in the anodic decarboxylative process which is powered by electrochemistry is described for the *N*-alkylation of heterocycles. There are several ways to do this, but the most widely used ones are modifications of the S_N1 or S_N2 reaction (like Mitsunobu) [86].



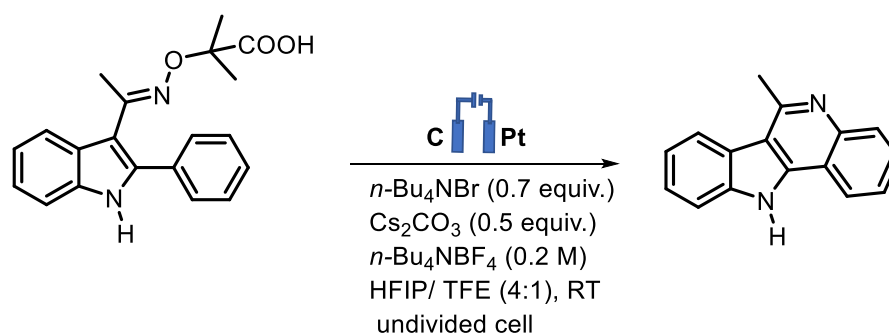
Scheme 1.28 Synthesis of *N*-alkylation of heterocycles.

In 2021, Oh and co-workers reported methods of 3-sulfonylated imidazopyridines and indolizines by electrochemical radical-radical cross-coupling guided by oxidation Potential. The sulfonyl radical addition to imidazopyridines has been achieved through the iodine-mediated conversion of sodium sulfinates to the corresponding sulfonyl iodides [87].



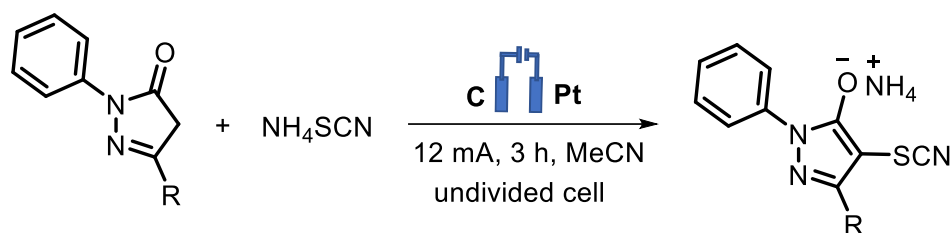
Scheme 1.29 Synthesis of 3-sulfonylated imidazopyridines.

In 2021, Huang and co-workers demonstrated that indole-fused polycyclics are constructed by electrochemically decarboxylating α -imino-oxy acids to produce indole-radicals. Iminyl radicals can be produced via homolytic breaking of the relatively weak N–O bonds in oxime derivatives [88].



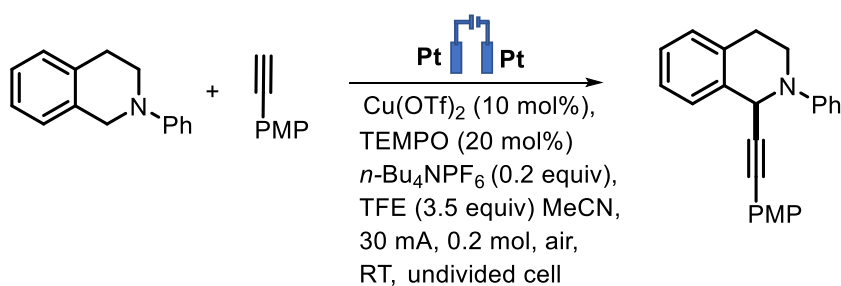
Scheme 1.30 Synthesis of indole-fused polycyclics.

In 2021, Lei group demonstrated a novel series of cross-coupling products under metal catalyst-, exogenous-oxidant-, and exogenous-electrolyte-free conditions was produced by an electrochemical oxidative cross-coupling reaction involving 2-5 substituted pyrazolin-5-ones and ammonium thiocyanate [89].



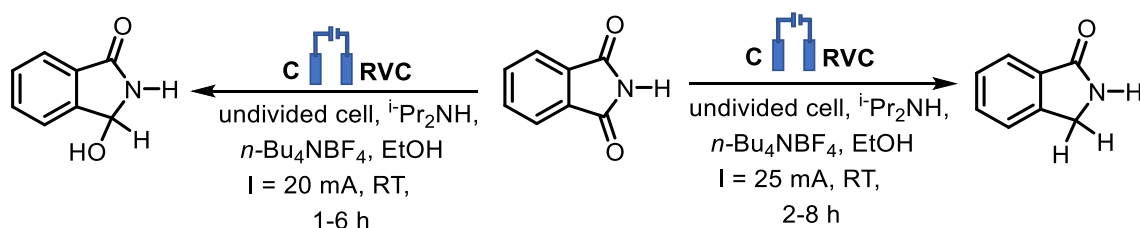
Scheme 1.31 Synthesis of ammonium 3-methyl-1-phenyl-4-thiocyanato-1H-pyrazol-5-olate.

In 2021, Xu and Guo demonstrated that cross-coupling between C(sp³) and H/C(sp) in an electrocatalytic continuous flow via TEMPO/copper relay catalysis. Two Pt electrodes were used as the anode and cathode in a microreactor that ran on a steady current during the electrosynthesis process[90].



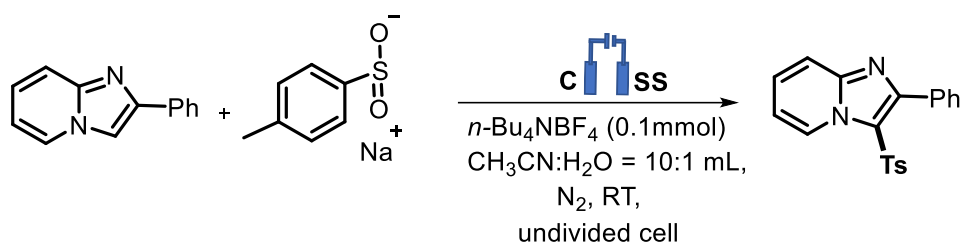
Scheme 1.32 C(sp³)-H alkylation of tetrahydroisoquinolines.

In 2021, Xiang group reported the cyclic imides are reduced to hydroxylactams and lactams in an electroselective and controlled manner. A straightforward undivided cell with carbon electrodes operating at room temperature has been used to provide an effective and useful electrochemical technique for the selective reduction of cyclic imides [91].



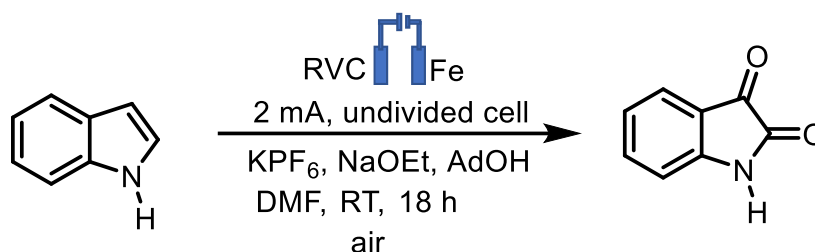
Scheme 1.33 Substrate Scope synthesis of hydroxylactams and lactams.

In 2021, Lei group reported an imidazo[1,2-a]pyridine C3-sulfonylation without the use of metals. The synthesis of numerous valuable chemicals can be achieved by environmentally friendly and atom-efficient methods such as electrochemical oxidative cross-coupling. This work devised an electrochemical method for C3-sulfonylation of imidazo[1,2-a]pyridines using sodium benzenesulfonates as sulfonylation reagents [92].



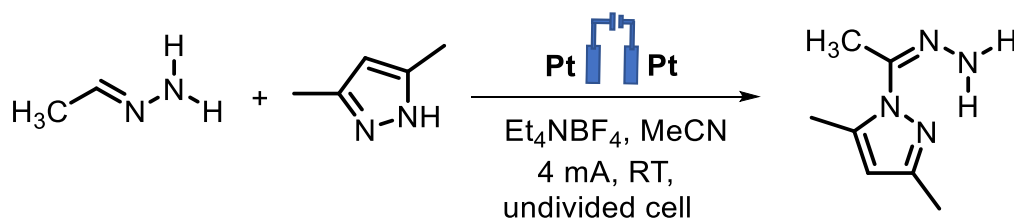
Scheme 1.34 The electrochemical C3-sulfonylation of various sodium benzenesulfinate.

Under the influence of Scalable electrochemical aerobic respiration of indoles to isatins without electron transfer mediators was demonstrated by Qiu Feng Huang and colleagues in 2022 [93].



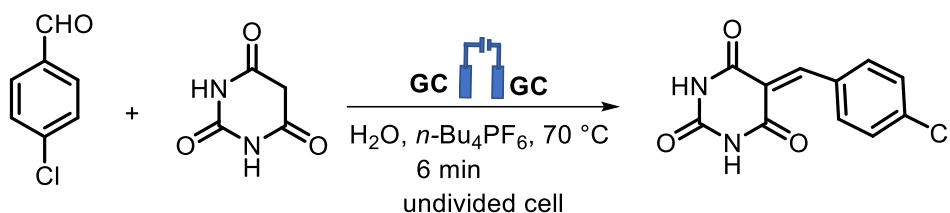
Scheme 1.35 Synthesis of isatin (indole-2,3-dione).

In 2022, Huang and co-workers demonstrated that electrochemical C(sp²)-H amination of aldehyde hydrazones with azoles has been developed as a generic and extremely efficient technique. This process may produce aminated hydrazone derivatives in high yields without the need of any foreign metals, catalysts, or oxidants. This tactic works with a wide variety of functional groups and may be used with either aromatic or aliphatic aldehyde hydrazones [94].



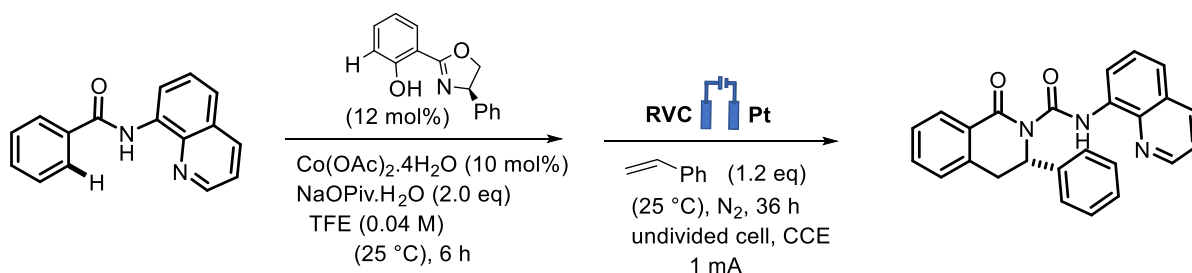
Scheme 1.36 Synthesis of aminated hydrazone.

In 2023, El-Nassan and co-workers demonstrated the use of 5-Benzylidenebarbiturate derivatives as colorimetric cyanide probes and their electrochemical synthesis. Barbituric acid and aldehydes were therefore able to be synthesized in six minutes under electrochemical circumstances using water as a green solvent, resulting in the formation of very pure molecules [95].



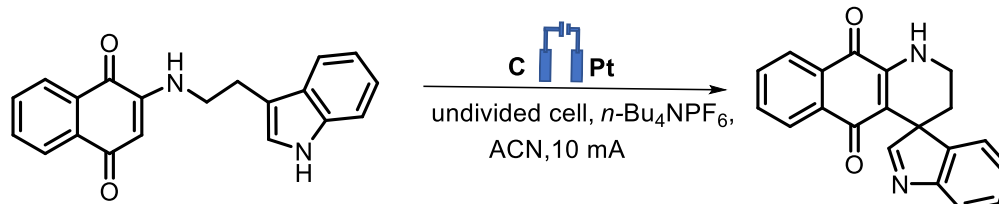
Scheme 1.37 Synthesis of 5-(4-chlorobenzylidene) barbiturate under electrochemical conditions.

In 2023, Shi and co-workers demonstrated the cobalt catalyzed enantioselective and regioselective electro-oxidative reactions. Recently, a potent and sustainable method for organic synthesis has been developed by combining electrosynthesis with 3d metal-catalyzed C-H activation [96].



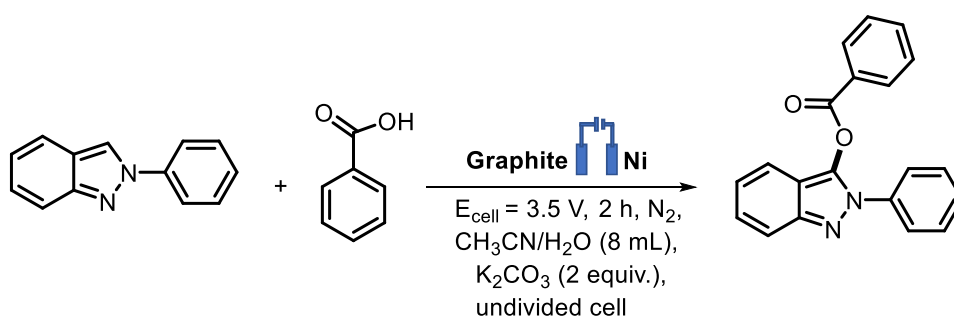
Scheme 1.38 Synthesis of dihydroquinolones by oxidative Co(II) catalyzed C-H/N-H annulation with olefins.

In 2023, Guo and co-workers demonstrated that dearomative arylation of indoles with strong functional group compatibility allowed for the electro-synthesis of spiro-indolenines in both batch and continuous flow. Easy-to-use undivided cells were employed without the need of oxidants or catalysts [97].



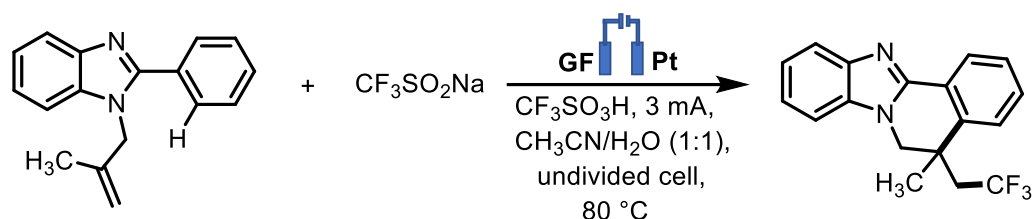
Scheme 1.39 Substrate Scope of Electro-synthesis of Spiro- indolenines with Substituent at C2 Position of Indoles.

In 2023, Sharma and co-workers reported the structural changes in *2H*-indazole can be induced using a straightforward and effective electrochemical process that employs cell voltage manipulation. The method of organic synthesis has been completely transformed by electro-organic synthesis, which uses electrical energy to propel chemical processes and is therefore more environmentally friendly and sustainable [98].



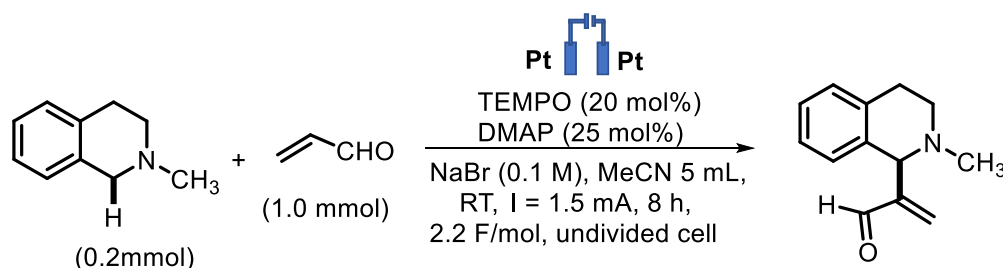
Scheme 1.40 Electrochemical *N*-1 migratory acylation of *2H*- indazole.

In 2023, Li and co-workers reported to get 5-(2,2,2-trifluoro)ethyl dihydrobenzimidazo [2,1-*a*]isoquinoline derivatives directly from benzimidazole-tethered pendent unactivated alkenes in an undivided cell, an electrochemical radical relay approach is proposed. Alkyl-, OMe, halo, and -CF₃ are a few substituents that might do well in this electrochemistry [99].



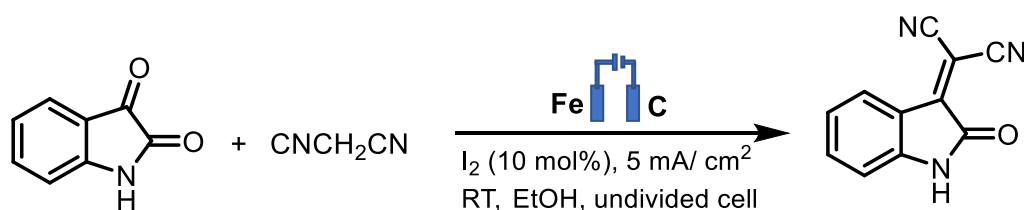
Scheme 1.41 Electrochemical access to CF_3 -containing heterocycles via radical relay of pendent alkenes.

In 2023, Mei and co-workers have reported that highly efficient electrochemical generation using TEMPO/NaBr as an electrocatalyst and 4-dimethylaminopyridine as an organic catalyst, the Morita-Baylis-Hillman reaction and Shono oxidation are combined to create the $\text{C}(\text{sp}^3)\text{-H}$. As a green oxidant, electricity allows the chemical oxidants to be omitted from this reaction [100].



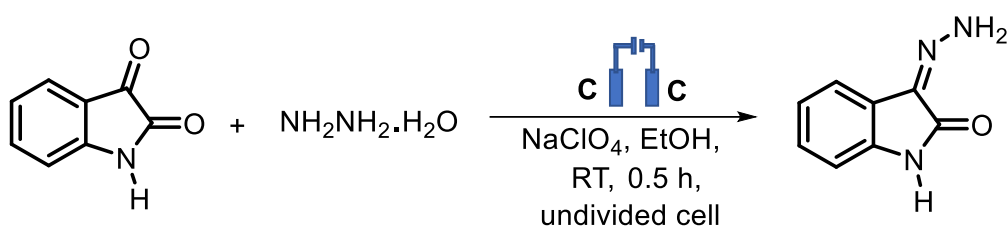
Scheme 1.42 $\text{C}(\text{sp}^3)\text{-H}$ alkenylation of tetrahydroisoquinolines.

In 2023, Malviya research group synthesized a protocol using easily accessible isatin derivatives, malononitrile, and iodine, the first electrochemical molecular iodine promoted, domino reactions for the green synthesis of physiologically relevant dicyano 2-(2-oxoindolin-3-ylidene) malononitriles have been obtained [101].



Scheme 1.43 Synthesis of dicyano 2-(2-oxoindolin-3-ylidene) malononitriles.

In 2023, Malviya research group synthesized electrochemical synthesis of isatins and hydrazones via C-N cross-coupling and C(sp²)-H/C(sp³)-H functionalization. Using C-N cross-coupling and C(sp²)-H/C(sp³)-H functionalization with the aid of I₂-DMSO, we present an efficient and innovative technique for the electrochemical synthesis of isatin (indole-2,3-dione) from 2-aminoacetophenone. A vast variety of substituted 2-aminoacetophenone substrates may be generated utilizing this synthetic approach [102].



Scheme 1.44 Synthesis of 3-hydrazineylideneindolin-2-one.

1.4.4.1 Techniques

Various technique can be used determine the electrochemical behaviour of organic compounds. We have used two type of technique in electro-organic synthesis.

1.4.4.1.1 Chrono-amperometry

In electrochemistry, a process known as chrono-amperometry is applying a constant potential to an electrochemical cell and tracking the current that results over time. Chronoamperometry is important in electro-organic synthesis for a number of reasons [103].

1.4.4.1.1.1 Kinetic Information: By tracking the current response over time, this method provides comprehensive information regarding reaction kinetics. Understanding the pace of the electrochemical reactions involved in organic synthesis can benefit from this [104].

1.4.4.1.1.2 Reaction mechanism elucidation: The dependent of current allows for the better understanding of reaction mechanisms. The overall current response may be influenced by several intermediates and reaction steps, which enables scientists to deduce

the mechanism of the electro-organic synthesis [105].

1.4.4.1.1.3 Optimization of reaction conditions: Facilitates the optimization of reaction conditions by investigating the impact of multiple parameters, including temperature, concentration, and voltage, on the electro-organic synthesis procedure. Improving the yield and selectivity of the intended products requires this.

1.4.4.1.1.4 Control of Electrode Processes: Ensures that the electrochemical reactions happen as intended by enabling fine control over electrode operations. In electro-organic synthesis, attaining great efficiency and selectivity requires this control [106].

1.4.4.1.1.5 Real-time monitoring: Provides real-time monitoring of the electrochemical process, enabling researchers to modify reaction conditions at any time. Maintaining ideal settings and preventing adverse reactions or undesirable byproducts are two reasons this is especially helpful.

1.4.4.1.1.6 Quantitative analysis: The foundation for quantitative analysis of reaction kinetics is provided by quantitative analysis, which enables researchers to ascertain reaction rates, rate constants, and other crucial characteristics. Having this knowledge is essential for creating effective electro-organic synthesis procedures.

1.4.4.1.1.7 Scale-up considerations: The scaling up of electro-organic synthesis processes can be guided by the insights obtained from chronoamperometry experiments. A thorough understanding of laboratory-scale kinetics and reaction processes is necessary for an effective scale-up to industrial levels [107].

1.4.4.1.2 Chronopotentiometry

Chronoamperometry is particularly valuable in the context of electro-organic synthesis because it can offer comprehensive insights into the electrochemical processes that are involved in the creation and modification of organic molecules. The importance of chronoamperometry in electro-organic synthesis is demonstrated by the following numero-

-us reasons [108].

1.4.4.1.2.1 Reaction kinetics understanding: Chronoamperometry is a useful tool for studying reaction kinetics since it allows you to track current changes over time. This data is essential for comprehending how quickly electrochemical reactions occur and aids in the optimization of reaction conditions for increased effectiveness [109].

1.4.4.1.2.2 Optimization of electrode material: Electrode potential can be optimized, which aids researchers in determining the ideal circumstances for accomplishing the intended electro-organic processes. Maintaining control over selectivity and yield requires this optimization [110].

1.4.4.1.2.3 Selective Functionalization: Chronoamperometry plays a role in the selective functionalization of organic molecules. By controlling the electrochemical parameters, researchers can guide the reaction towards specific functional groups, leading to tailored synthesis pathways [111].

1.4.4.1.2.4 Green chemistry applications: Electro-organic synthesis, assisted by chronoamperometry, conforms to green chemistry principles by reducing the quantity of conventional reagents used and providing softer reaction conditions. This has the potential to lead to synthetic pathways that are more ecologically friendly.

1.4.4.1.2.5 Complex Molecule Synthesis: especially helpful for the synthesis of complicated organic compounds, which might be difficult to achieve using conventional chemical techniques. Novel pathways to molecules of interest can be achieved using electro-organic synthesis led by chronoamperometry [112].

To chemical product was determined by OCP, CV, EIS.

1.4.4.1.3 Open Circuit potential

while discussing electro-organic synthesis, the term "open circuit potential" (OCP) is used to describe the voltage or potential difference across an electrochemical cell while no

current is passing through it. It is the potential of the electrochemical system in the absence of or in the presence of a very small external current. The OCP is a critical parameter that can affect the results of electrochemical reactions in the context of electro-organic synthesis. The OCP is useful for determining the viability of an electro-organic transition since it offers information about the thermodynamics of the electrochemical system [113]. Before applying an external current or voltage to start the desired electrochemical reaction, the OCP is commonly measured during electro-organic synthesis. Electrode type, electrolyte composition, temperature, and the presence of reactants and products all play a role in determining the OCP. An OCP analysis can help determine the best electrode materials and reaction conditions for an electro-organic synthesis [114].

OCP measurements are commonly used by scientists to learn more about the redox reactions happening at the electrode-electrolyte interface. The electrochemical window of the system, potential side reactions, and effective electro-organic synthesis techniques can all be determined by examining the OCP under varying conditions [115].

1.4.4.1.4 Cyclic Voltammetry

Electro-organic synthesis relies heavily on cyclic voltammetry (CV), a strong electrochemical method. Here are a few examples of why cyclic voltammetry is so crucial here:

1.4.4.1.4.1 Redox Potential determination: The redox potentials of organic molecules can be calculated by using cyclic voltammetry. Because it sheds light on the practicability and thermodynamics of electron transfer processes, knowing the redox behaviour is crucial for designing electro-organic synthesis procedures [116].

1.4.4.1.4.2 Reaction Mechanism Elucidation: The electrochemical behaviour of intermediates and reactive species in electro-organic reactions can be studied with CV, allowing for a better understanding of the underlying reaction mechanism. This aids in the

elucidation of the reaction mechanisms and yields useful data for improving reaction conditions and developing more effective synthetic routes [117].

1.4.4.1.4.3 Optimization of reaction condition: Cyclic voltammetry is useful for optimizing reaction conditions since it reveals the potential range, scan rate, and electrolyte composition that work best for a specific electro-organic synthesis. The electrochemical behavior under these conditions can be optimized to improve reaction efficiency, selectivity, and overall yield.

1.4.4.1.4.4 Exploration of new Synthetic routes: cyclic voltammetry can be used as a starting point for investigating potential novel synthesis pathways and methods. Researchers can create novel ways for the synthesis of complex molecules by taking advantage of the electrochemical reactivity of organic compounds, which can be difficult to access using more conventional chemical techniques [118].

1.4.4.1.4.5 Scale-up Consideration: Scale-up cyclic voltammetry research can provide important insights for optimizing large-scale electro-organic synthesis. Learning about electrochemical activity at the molecular level is useful for planning and optimizing reactions at the industrial scale.

1.4.4.1.5 Electrochemical Impedance Spectroscopy:

The electrical impedance of an electrochemical system can be measured as a function of frequency using electrochemical impedance spectroscopy (EIS). There are many reasons why EIS is helpful in the field of electro-organic synthesis:

1.4.4.1.5.1 Characterization of electrode interfaces: EIS can be used to characterize electrode interfaces because it reveals information on the resistance and capacitance of the electrode-electrolyte interface. In order to optimize conditions for optimal electron transfer, this knowledge of the electrode surface behaviour is critical during electro-organic synthesis [119].

1.4.4.1.5.2 Detection of electron transfer processes: EIS is sensitive to variations in the electrochemical processes taking place at the electrode interface, allowing it to detect electron transfer processes. It can be used to detect and measure electron transfer reactions, offering insights into the kinetics of redox processes involved in electro-organic synthesis.

1.4.4.1.5.3 Understanding mass transport process: During electro-organic synthesis, EIS aids in the elucidation of mass transport processes. Diffusion of reactants and products to and from the electrode surface is characterized, allowing for more effective mass transport and faster reaction rates to be designed into reaction setups.

1.4.4.1.5.4 Identification of Reaction Intermediates: EIS can be used to detect and characterize intermediates generated during electro-organic synthesis, allowing for more accurate reaction tracking and control. To better understand reaction pathways, scientists can analyze impedance changes at several frequencies to learn about the creation and behaviour of reactive species [120].

In the view of the importance of nitrogen-containing organic compounds and electrochemical synthesis as a renewable, sustainable, and benign source of energy, our interest is to explore the chemistry (synthesis and structural characterization) of Schiff's base (3-hydrazineylideneindolin-2-one) and dicyano 2-(2-oxoindolin-3-ylidene) malonitriles, pyrazolo[3,4-b] pyridine ring, 1,2,3-triazole glycosides. The studies have been described in subsequent **Chapter 2-5**.

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