

# SYNTHETIC ACCESS TO 2-PYRIDONE SCAFFOLDS

## Abstract

In last few decades synthetic organic chemists shows immense interest on the synthesis and functionalization of the 2-pyridone moiety, which is found in many natural compounds. 2-pyridone-containing compounds have shown anticancer and antiviral action. 2-pyridone derivatives are also important building blocks in both the pharmaceutical and material sciences industries. Although several techniques for synthesis of 2-pyridones are known, new methods for more efficient synthesis of 2-pyridones are being developed. Several methods involving Lewis acids or Bronsted acids as catalysts have been developed in recent decades, and annulation of pyridine derivatives with electrophiles has emerged as a valuable strategy to access a diverse array of 2-pyridone derivatives enabling the incorporation of diverse functional groups and stereochemistry. Furthermore, transition metal-catalysed reactions, including C-H activation and cross-coupling activities, have been used to synthesise 2-pyridone scaffolds. These transitions enable late-stage diversification of existing compounds, simplifying synthesis and allowing the production of complex 2-pyridone derivatives. Overall, the development of efficient and diverse synthetic techniques has facilitated quick access to 2-pyridone scaffolds, allowing for their inclusion into drug discovery efforts and usefulness in a variety of organic synthesis applications. Continued progress in this sector bodes well for the identification of new bioactive chemicals and the extension of chemical space.

**Keywords:** 2-Pyridone, Synthetic.

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## I. STRUCTURE OF 2-PYRIDONE

2-pyridone (**1**) is a tautomeric derivative of 2-hydroxy pyridine (**2**) (see figure 1).<sup>1</sup> The tautomeric form is determined by the physical condition of the substrate. In the solid phase, it is completely 2-pyridone (**1**), but in the dilute gas phase it is 2-hydroxy pyridine (**2**). However, the polarity of the medium completely determines the tautomeric form in solution. It likes to be in the 2-pyridone form (**1**) in polar solvents (such as THF, DMSO, and H<sub>2</sub>O) and the 2-hydroxypyridine form (**2**) in non-polar solvents (such as CCl<sub>4</sub>). This scaffold has four C-H bonds of varying electrical type. Its different resonance structures reveal that C3 and C5-centers are more electron-rich than the other centres (fig 2). As a result, such locations are more reactive to various electrophiles. C4 and C6-centers, on the other hand, are very electron-deficient and reactive to various nucleophiles.



Figure 1: Tautomeric form of 2-pyridone and 2-hydroxy pyridine

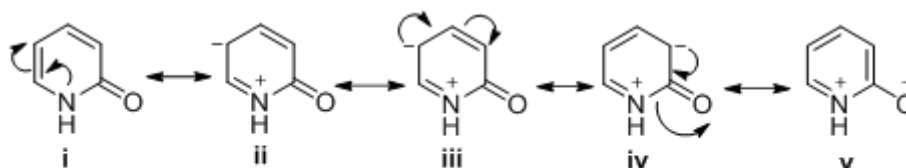
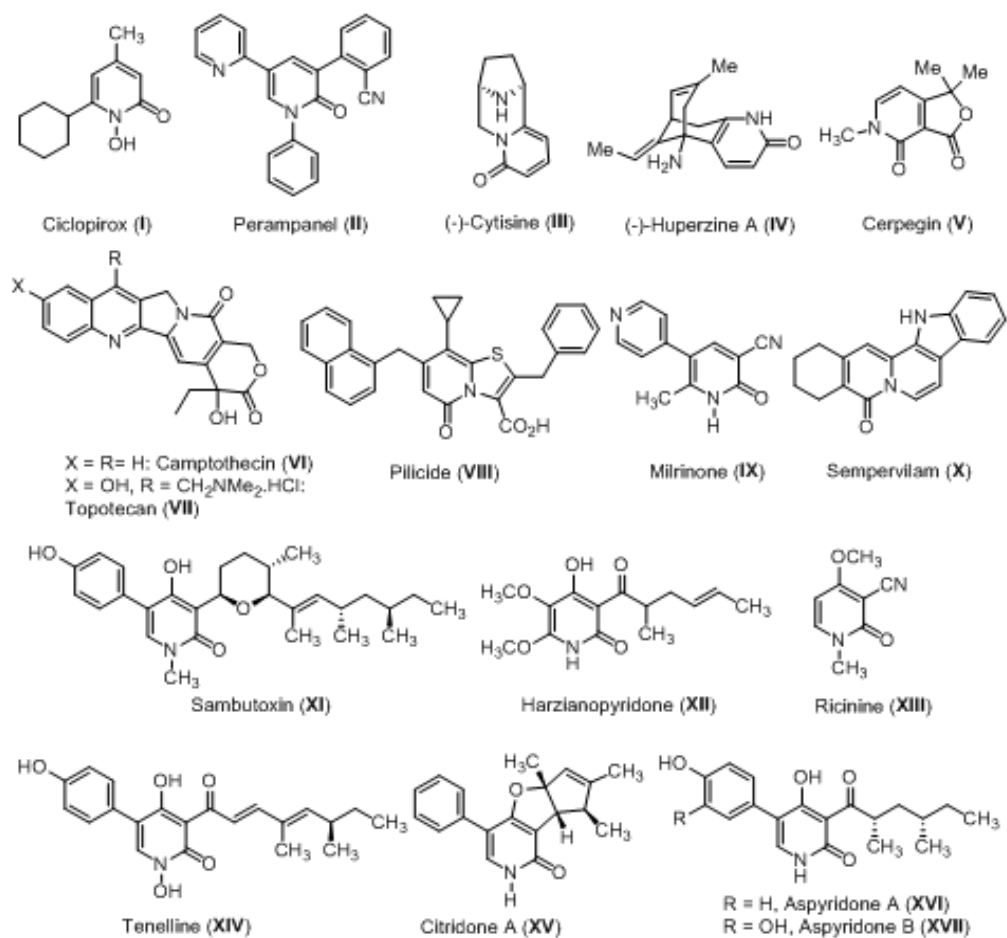


Figure 2: Resonance structures of 2-pyridone

## II. IMPORTANCE OF 2-PYRIDONE DERIVATIVES

2-Pyridone is a vital and versatile chemical with several uses in a variety of industries. Its relevance stems from its unusual chemical characteristics, which make it an useful building block in organic synthesis, a crucial component in pharmaceutical research, an essential solvent, a major player in coordination chemistry, and a key player in many other important domains.<sup>2</sup>

- 1. Chemical Building Blocks:** 2-pyridone is an important starting material in the synthesis of several chemical compounds. Because of its capacity to undergo a variety of chemical reactions, scientists may produce complex compounds that are employed in medicines, agrochemicals, and functional materials. Because of its adaptability, it is a vital tool for researchers seeking to produce unique goods and solutions.
- 2. Pharmaceutical Application:**<sup>3</sup> 2-pyridone and its derivatives are significant for its biological applications (fig 3). Because of its structural closeness to nucleic acid bases (like, cytosine), it is useful in drug design and medicinal chemistry. Scientists have investigated 2-pyridone derivatives for their antibacterial, antiviral, anti-inflammatory, and anticancer properties. These molecules provide up new avenues for the creation of life-saving drugs and treatments, tackling some of humanity's most difficult ailments.



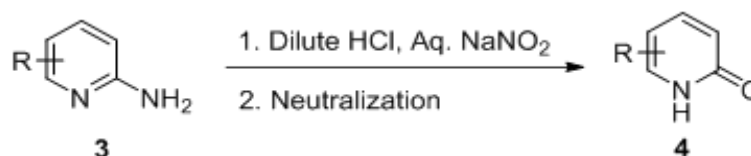
**Figure 3:** Few Biologically Important 2-Pyridone Derivatives

- Solvent Properties:** The solvent characteristics of 2-pyridone are also important. It is a polar aprotic solvent with strong solvating power, making it useful in a variety of chemical reactions and procedures. The capacity to dissolve a broad variety of chemicals allows researchers to carry out reactions more effectively, allowing for the discovery of novel chemical routes and the creation of diverse molecules.
- Application in Coordination Chemistry:** Furthermore, 2-pyridone has remarkable coordination chemistry, functioning as a ligand in the formation of complexes with metal ions. These complexes have important applications in catalysis and materials research. Furthermore, the coordination properties of 2-pyridone aid in the design and development of metal-organic frameworks (MOFs) and other functional materials with applications in gas storage, separation, and sensing.
- Application in Biochemical Research:** 2-pyridone is used in biochemical research in addition to its chemical and medicinal applications. Its metabolites have been used to study enzyme mechanisms, receptor interactions, and other biological processes. This has resulted in a greater knowledge of diverse biochemical processes, which has aided in the creation of tailored medicines and diagnostic tools.

- 6. Application in industrial processes:** Furthermore, 2-pyridone is used in a variety of industrial operations. Because of its solubility and adaptability as a chemical intermediary it is useful in the creation of plastics, polymers, and specialised chemicals, hence contributing to the manufacturing sector.

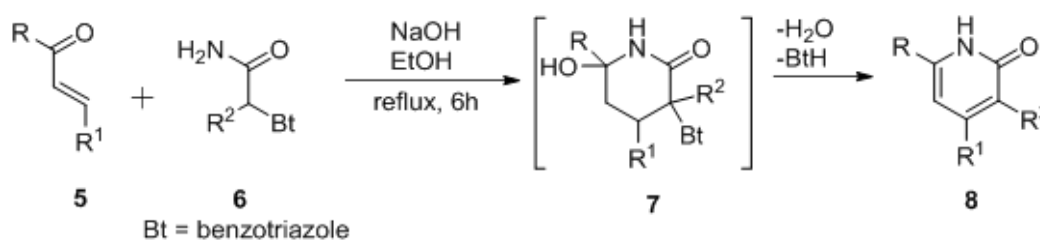
### III. SYNTHESIS OF 2-PYRIDONE SCAFFOLDS

- 1. Synthesis without Involvement of Transition Metal Catalysts:** Diazotization of 2-amino aniline derivatives converts it to corresponding phenol derivatives. It occurs in a stepwise manner. In the 1<sup>st</sup> step diazonium salt was formed in presence of dilute HCl and aqueous sodium nitrite solution. Then by means of time water present in the solution attacks the diazonium salt and converts it to corresponding phenol derivative with the liberation of environment benign molecular nitrogen. Similarly, 2-Amino pyridine derivatives (**3**) can be easily converted to 2-pyridone derivatives (**4**) by diazotization method in presence of dilute HCl and aqueous sodium nitrite solution (scheme 1).



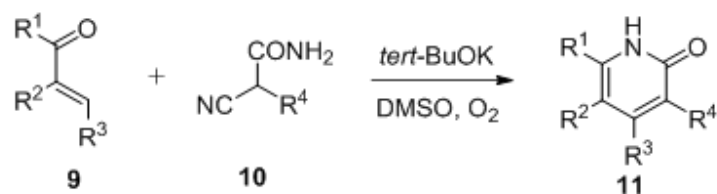
**Scheme 1:** Diazotization of 2-Amino Pyridine Derivatives

Katritzky et. al. demonstrated a base-promoted tandem cyclization reaction involving 1,3-disubstituted prop-2-en-1-one derivatives (**5**) and benzotriazole-containing amide derivative (**6**), followed by dehydration and benzotriazole loss to obtain the required pyrid-2-one derivative **8** (scheme 2).<sup>4</sup> Tandem [3 + 3] annulations comprising a Michael addition followed by cyclization provide the 2-pyridone rings.



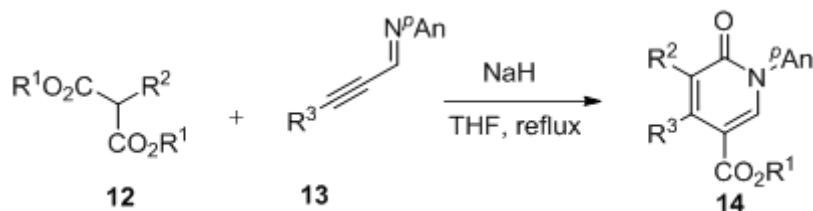
**Scheme 2:** Base-Promoted Tandem Cyclization between 1,3-Disubstituted Prop-2-En-1-Ones and Benzotriazole-Containing Amides

Condensation between  $\alpha,\beta$ -unsaturated ketone (enone derivative, **9**) and cyanoacetamide derivative (**10**) in presence of *tert*-BuOK under oxygen atmosphere gave substituted pyridine (**11**) (scheme 3).<sup>5</sup>



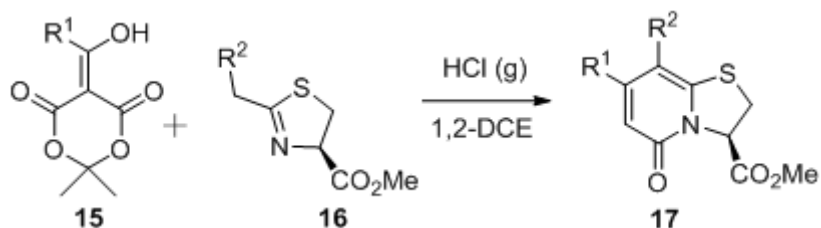
**Scheme 3:** Tert-Butoxide Mediated 2-Pyridone Derivative Synthesis from Enone and Cyanoacetamides

Another novel strategy developed by Shimizu et al. showed the synthesis of highly functionalized 2-pyridone (14) from dimethyl malonate derivative (12) and alkynyl imine derivative (13) with reaction of sodium hydride. The developed method underwent *via* nucleophilic addition of malonate anion to alkynyl imines (13) in good to excellent yield (Scheme 4).<sup>6</sup>



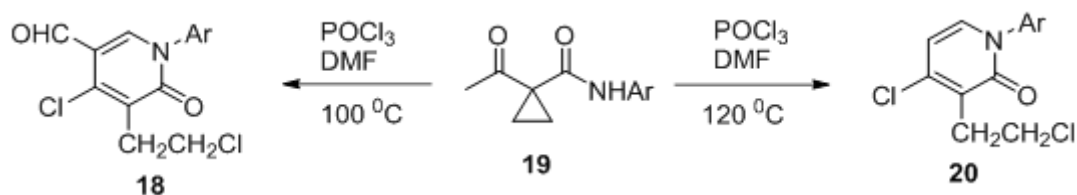
**Scheme 4:** Synthesis of 2-Pyridones from Dimethyl Malonate Derivative and Alkynyl Imine Derivative

In another work, Pemberton et. al. observed that when acyl ketene produced from acid derivative of meldrum (15) was combined with thiazolines (16) produced 2-pyridone (17) in excellent quantities (scheme 5).<sup>7</sup>



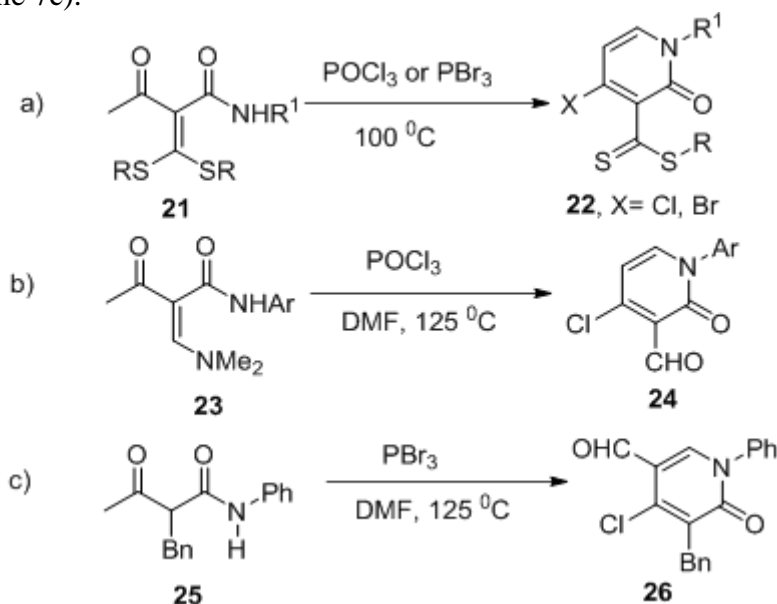
**Scheme 5:** 2-Pyridone Derivative Synthesis from Meldrum Derivative and Thiazolines

Using the Vilsmeier-Haack reaction, halogenated Pyridin-2(1H)-ones were directly synthesised from an acyclic substrate. By using the Vilsmeier Haack reaction to create highly functionalized pyridine-2-(1H)-ones (20) from 1-acetyl-1-carbamoyl cyclopropanes (19) reported by Liu and co-workers. They noticed that reactions of compound 19 with POCl<sub>3</sub> in DMF at 100°C and 120°C produced the 2-pyridone derivatives 18 and 20, respectively (scheme 6).<sup>8</sup>



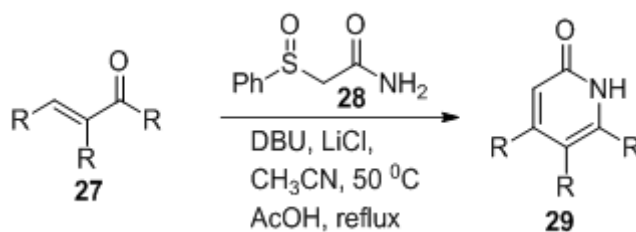
**Scheme 6:** Vilsmeier-Haack Reaction of 1-Acetyl-1-Carbamoyl Cyclopropanes

Chen et al. reported that Vilsmeier Haack reaction of  $\alpha$ -acetyl- $\alpha$ -carbamoyl ketene derivatives (**21**) furnished 4-halogenated 2(1*H*)-pyridones (**22**) in one pot in efficient way (scheme 7a).<sup>9a</sup> Dong and co-workers showed that Vilsmeier reaction of enaminones (**23**) in one pot was used for the synthesis of halogenated 2-pyridones (**24**) with moderate to good yield (scheme 7b).<sup>9b</sup> They also showed that Vilsmeier cyclization of 2-benzyl-3-oxo-*N*-phenyl butanamide (**25**) produced polysubstituted pyridines-2-ones (**26**) in good yield (Scheme 7c).<sup>9c</sup>



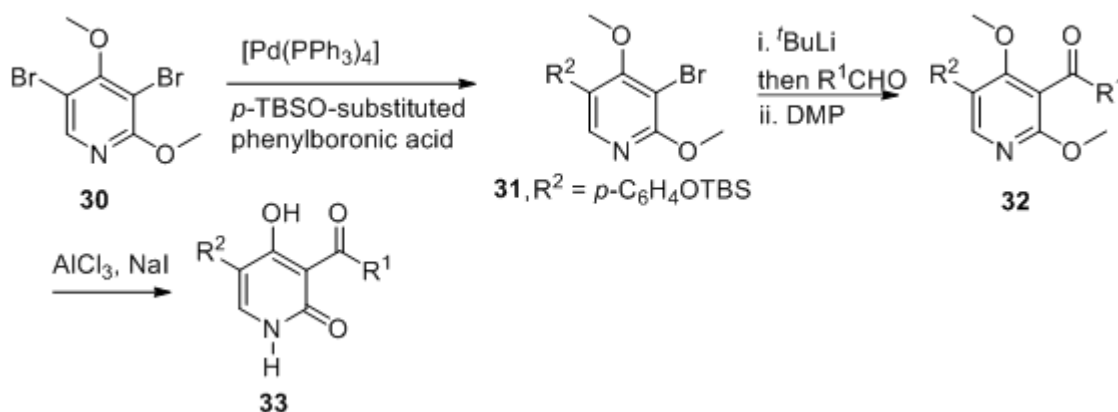
**Scheme 7:** Vilsmeier Haack Reaction of *A*-Acetyl-*A*-Carbamoyl Ketene Derivatives to Synthesize 2-Pyridone Derivatives

In another procedure, substituted 2-pyridones **29** were produced by 1,4-addition of 2-(phenylsulfinyl)-acetamide (**28**) to unsaturated ketones **27**, followed by cyclization (scheme 8).<sup>10</sup>



**Scheme 8:** 2-Pyridone Derivative Synthesis from 2-(Phenylsulfinyl)-Acetamide and Unsaturated Ketones

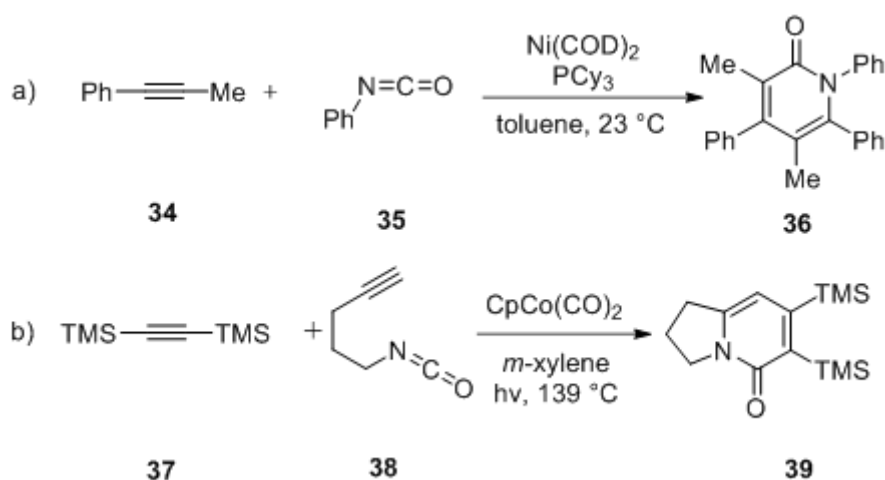
Many physiologically active natural compounds had a 4-hydroxy-2-pyridone scaffold substituted at the C3 and C5 positions. Waldmann and colleagues noted that this scaffold **33** was straightforward to reach. The presence of a Pd(0)-catalyzed Suzuki coupling between dibromopyridine and *p*-OTBS substituted phenyl boronic acid yielded monobromopyridine (**31**). The resulting monobromopyridine (**31**) was lithiated in the presence of *tert*-BuLi, and further aldehyde treatment yielded secondary alcohol, which was oxidised to keto derivative (**32**). In the presence of AlCl<sub>3</sub> and NaI, simultaneous demethylation and desilylation were performed to get 2-pyridone derivative **33** (scheme 9).<sup>11</sup>



**Scheme 9:** Pd(0)-Catalysed 2-Pyridone Derivative Synthesis from Pyridine Derivative

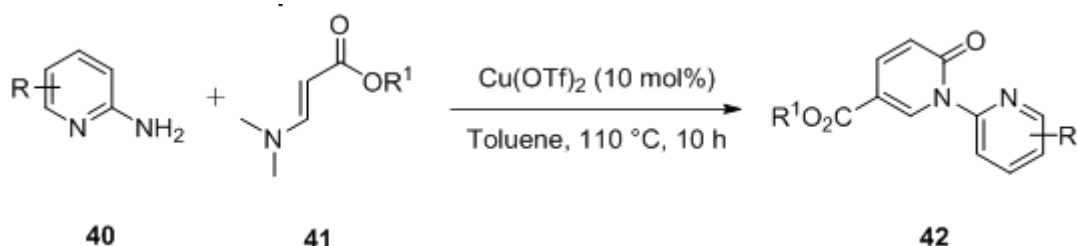
- 2. Synthesis Via Involvement of Transition Metal Catalysts:** The synthesis of 2-pyridones using transition metal catalysis is an important area in organic chemistry. Transition metal catalysis plays a crucial role in efficiently forming these compounds through various step-economic and atom-economic pathways. One common method for the synthesis of 2-pyridones involves the reaction of alkynes with nitriles in the presence of a transition metal catalyst. One of the well-known transition metal catalyzed reactions for 2-pyridone synthesis is the Povarov reaction, which typically involves the condensation of an alkyne, an imine, and a nitrile in the presence of a Lewis acid catalyst. In this case, the transition metal catalyst can act as the Lewis acid, facilitating the formation of the 2-pyridone ring. Common transition metals used for this reaction include palladium, platinum, and gold.

Hoberg's group developed intermolecular [2+2+2] cycloaddition reaction between alkynes (**34**) and isocyanates (**35**) in the presence of Ni(0)-catalyst to obtain 2-pyridone derivatives **36** (Scheme 10a).<sup>12</sup> Vollhardt and co-workers also reported the synthesis of 2-pyridone derivative **39** by treating alkyne **37** with isocyanate **38** under Co-catalysis (Scheme 10b).<sup>13</sup>



**Scheme 10:** Synthesize 2-Pyridone Core by Cyclo-Addition Reaction with Alkynes and Isocyanates

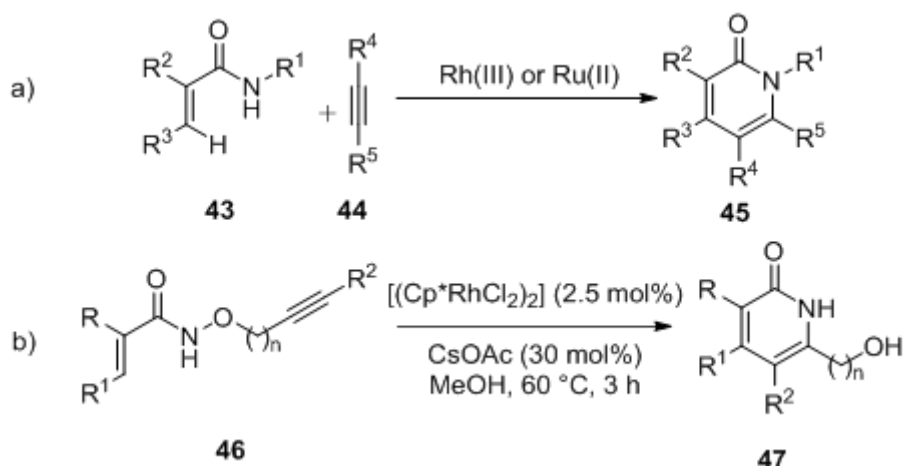
Lee group developed one-pot synthesis of *N*-heteroaryl 2-pyridone derivative **42** under Cu(II)-catalysis via [3+2+1] annulation between  $\beta$ -enamino esters (**41**) and 2-aminopyridine derivatives (**40**) (Scheme 11).<sup>14</sup>



**Scheme 11:** Cu(II)-Catalyzed Synthesis of *N*-Heteroaryl 2-Pyridone Core

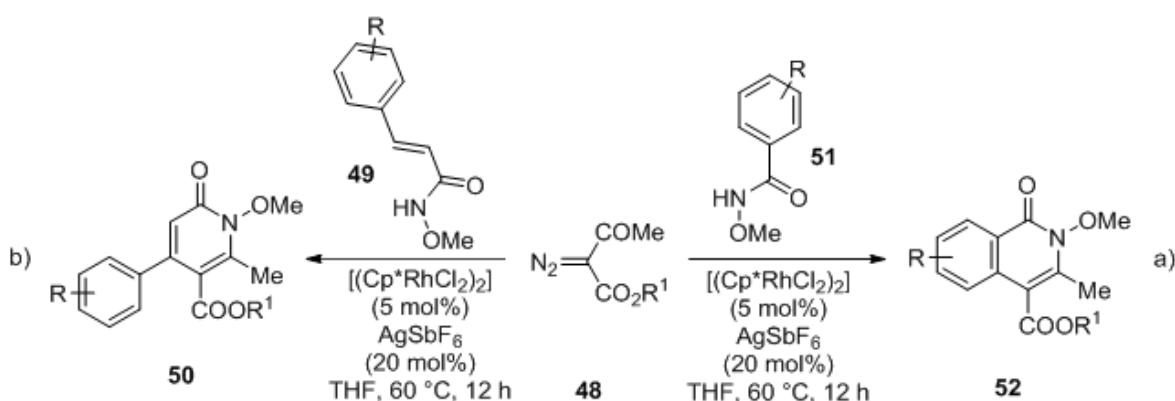
However, all these methods were associated with the cycloaddition strategy. Recently, transition metal catalyzed C-H bond functionalization strategies became significant for the synthesis of these scaffolds. Li and co-workers developed the annulation reaction between acylamides (**43**) and di-substituted alkynes (**44**) in the presence of ruthenium catalyst to provide substituted 2-pyridone derivatives (**45**) (Scheme 12a).<sup>15a</sup> They also established the regioselective coupling reaction between methylmethacramides and diphenyl acetylenes to generate pyridones in moderate yields.<sup>15b</sup> Similarly, Rovis group also explored the Rh(III)-catalyzed functionalized 2-pyridone derivative (**45**) synthesis using same coupling agents (Scheme 12a).<sup>15c</sup> Other than these reactions, there were also few reports of intramolecular coupling to form highly substituted 2-pyridone scaffolds under transition metal catalysis. Park and co-workers developed Rh(III)-catalyzed intramolecular annulation of  $\alpha$ ,  $\beta$ -unsaturated amides (**46**) and alkyne groups linked by ether linkages, which finally produced NH-free 2-pyridone derivatives (**47**) in an intramolecular fashion (Scheme 12b).<sup>16</sup>





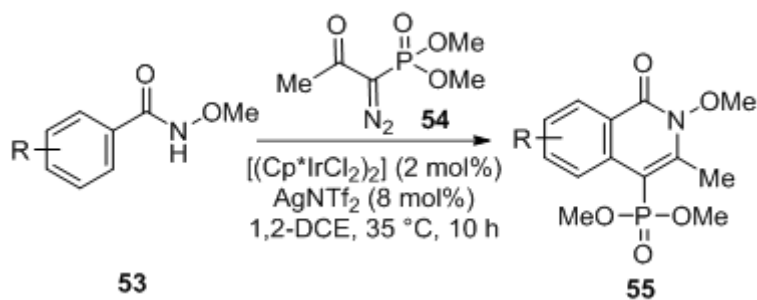
**Scheme 12:** Transition Metal Catalysed Synthesis 2-Pyridone *Via* C-H Bond Activation Followed by Annulations

The development of metal-carbenoid based migratory insertion techniques for the mild synthesis of various heterocycles during the past few decades is an intriguing topic in organic synthesis. Using such method, Yu's group skillfully demonstrated site-selective alkylation. Later, this policy was used by numerous other groups to advance. This allows for the synthesis of various heterocycles *via* either cyclization followed by dehydration or cyclo-condensation after alkylation. Isoquinolone derivatives are also an extended version of 2-pyridone derivatives. There are several reports of isoquinolone derivative synthesis using metal carbenoid strategy. In order to create *N*-methoxy isoquinolone scaffolds, Wang's group described a Rh(III)-catalyzed cascade cyclization of *N*-protected benzamides (**51**) using diazo compounds (**48**) to furnish isoquinolone derivatives (**52**) (scheme 13a).<sup>17</sup> They improved the process for making C4-arylated 2-pyridone scaffolds. They used acryl amide derivatives (**49**) and diazo derivatives (**48**) to do successful C-H bond activation followed by annulation to produce C4-arylated 2-pyridone derivatives (**50**) under Rh(III)-catalysis (scheme 13b).<sup>17</sup>



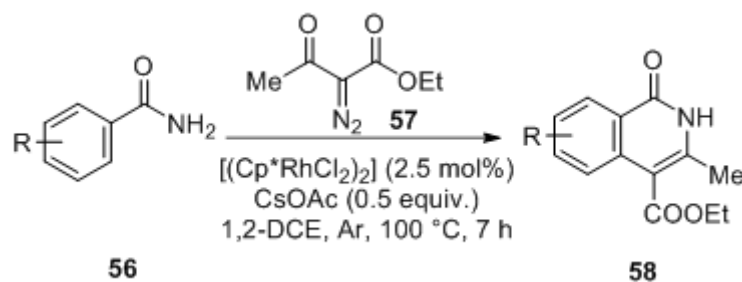
**Scheme 13:** Rh(III)-Catalysed Synthesis of Isoquinolone and 2-Pyridone Derivatives

Ramana group also produced a relatively cheaper Ir(III)-catalysed synthesis of *N*-methoxy isoquinolone derivatives (**55**) employing benzamide derivatives (**53**) with phosphonate azo (**54**) (scheme 14).<sup>18</sup>



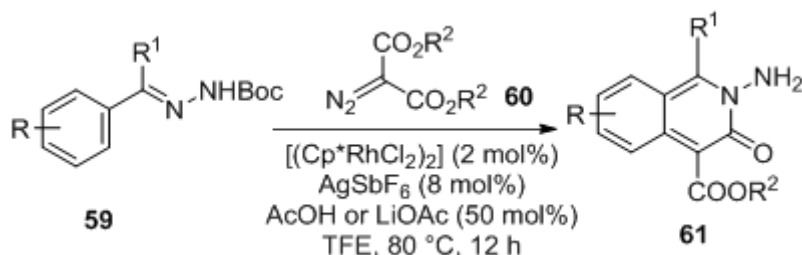
**Scheme 14:** Ir(III)-Catalyzed Synthesis of Isoquinolones using Metal-Carbenoid

In a related advance, Lin group created isoquinolone derivatives (**58**) under Rh(III)-catalysis from parent benzamide derivatives (**56**) taking diazo derivatives (**57**) as the annulating agent (scheme 15).<sup>19</sup> It provided immediate access to derivatives of NH-free isoquinolone derivatives.



**Scheme 15:** Rh(III)-Catalyzed Synthesis of NH-Free Isoquinolones from Benzamide Derivatives

The production of N-amino isoquinolin-3-ones (**61**) using *N*-Boc hydrazones (**59**) and diazomalonates (**60**) under Rh(III)-catalysis was also reported by Zhu's lab (scheme 16).<sup>20</sup> The employment of a traceless Boc-protecting group to produce a free amine derivative was the reaction's principal benefit.



**Scheme 16:** Rh(III)-Catalyzed Synthesis of 3-Isoquinolone from Hydrazones

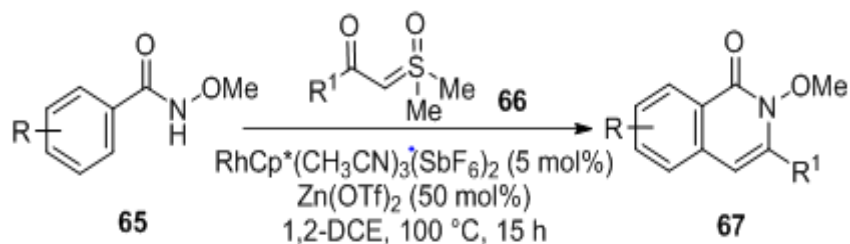
Lewis acid/cobalt Co-catalysed NH-free isoquinolone derivative (**64**) synthesis from ketimines (**62**) was also demonstrated by Glorius and colleagues (Scheme 17).<sup>21</sup> For improved catalytic efficacy,  $\text{B}(\text{C}_6\text{F}_5)_3$  was interestingly employed as the Lewis acid.

Ketimines that are free are very unstable. As a result, the Lewis assisted it act as a guiding group while also increasing its stability by binding to it.



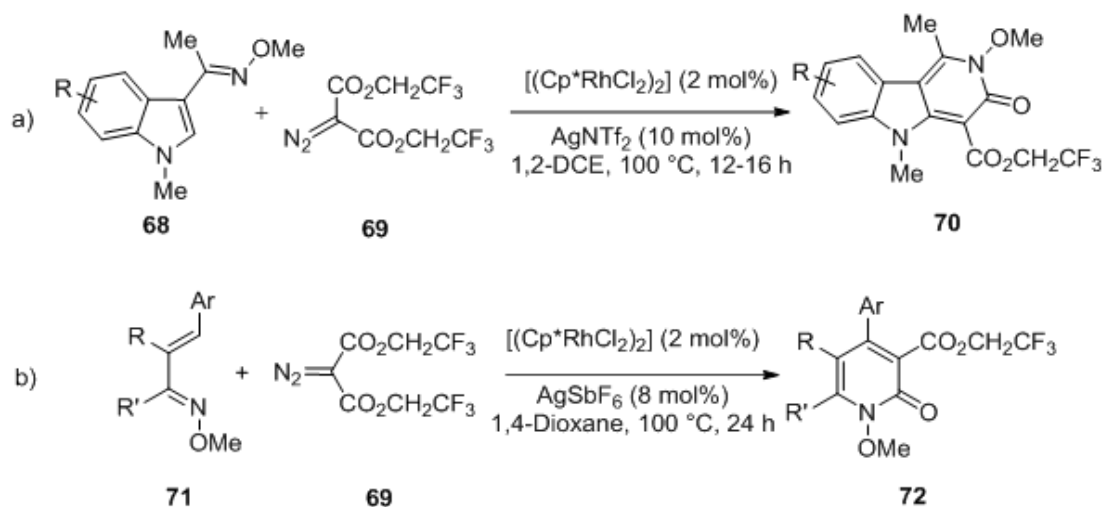
**Scheme 17:** Cobalt-Catalyzed Synthesis of 3-Isoquinolone from Ketimines

Sulfoxonium ylide has recently been employed as a carbene synthon. Dimethyl sulfoxide was released at this location and created the metal-carbenoid species. Li group published a study on the annulation of benzamide derivative (**65**) and sulfoxonium ylide (**66**) to make isoquinolone derivatives (**67**) (scheme 18).<sup>22</sup>



**Scheme 18:** Rh(III)-Catalyzed Synthesis of Isoquinolone using Sulfoxonium Ylides

Among the diazo compounds, recently using bis(2,2,2-trifluoroethyl) diazomalonate (**69**) C-H bond activation followed by annulation is new interest for researchers. Due to presence of highly electron withdrawing CF<sub>3</sub>-group, this diazo compound plays some special role in spontaneous annulation. Samanta group also synthesized indolopyridone scaffolds (**70**) *via* C-H bond alkylation of indole derivatives (**68**) with bis(2,2,2-trifluoroethyl) diazomalonate (**69**) under Rh(III)-catalysis (scheme 19a).<sup>23</sup> In another method, the quick synthesis of highly substituted 2-pyridone scaffolds (**72**) utilizing fluorinated diazomalonate (**69**) and  $\alpha$ ,  $\beta$ -unsaturated oximes (**71**) was accomplished using a Rh(III)-catalyzed method by Samanta group (scheme 19b).<sup>24</sup> Direct, site-selective alkylation based on migratory insertion and subsequent cyclocondensation are used to carry out the reaction. Different functional groups were investigated on a broad substrate scope. For this transition, the necessity of fluorinated diazomalonate was investigated. The production of the bioactive chemical added more depth to the methodology that had already been devised.



**Scheme 19:** Rh(III)-Catalyzed Synthesis of Indolopyridones and Highly Substituted 2-Pyridones

#### IV. CONCLUSION

Finally, synthetic access to 2-pyridone scaffolds offers great potential in a variety of domains such as pharmaceutical chemistry, materials science, and catalysis. The development of efficient and adaptable synthetic approaches for building these scaffolds has broadened their uses and possibilities for innovation. Researchers were able to obtain a varied spectrum of 2-pyridone derivatives with customised characteristics by carefully designing and optimising synthetic pathways. These chemicals have extraordinary biological properties, such as anticancer, antibacterial, and anti-inflammatory actions, making them promising therapeutic candidates. Furthermore, incorporating 2-pyridone motifs into functional materials has resulted in improved performance in applications such as sensors, polymers, and organic electronics. The difficulties in synthesising 2-pyridone scaffolds, such as regioselectivity, stereochemistry, and scalability, have prompted the investigation of novel reaction mechanisms and novel catalytic methods. As a result, synthetic chemists have addressed some of these issues, paving the path for more efficient and sustainable ways to these important chemicals. Future research into synthetic techniques for 2-pyridone scaffolds is expected to produce even more efficient and diversified solutions. Further research into these compounds' biological activities and mechanisms of action may lead to the creation of innovative medicinal medicines. Furthermore, the incorporation of 2-pyridone motifs into modern materials has the potential to revolutionise a number of sectors.

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