**VIVEKANAND COLLEGE, KOLHAPUR (Empowered Autonomous)**

**SEED MONEY SCHEME FOR RESEARCH**

**Annual/Final Report of the work done on the Research project**

(Report to be submitted within 2 months after dompletion of each year)

1) Project report No. 1st/ 2nd (final): First report
2) University Reference No.
3) Period of report: From 01st November 2022 to 31st October 2023
4) Title of research project: **Sustainable development in organic synthesis by utilizing Task Specific Ionic Liquids (TSILs)**
5) (a) Name of the Principal Investigator: Dr. D. S. Gaikwad

   (b) Department and College where work has progressed: **Department of Chemistry, Vivekanand College, Kolhapur (Empowered Autonomous)**
6) Effective date of starting of the project: **01st November 2022**
7) Grant approved and expenditure incurred during the period of the report:

   a. Total amount approved Rs. 1,40,000/-

   b. Total expenditure Rs. 27,564/-

   c. Report of the work done:

1) Brief objective of the project:

   (a) Evaluate the environmental impact of traditional organic synthesis methods and identify key areas where sustainable alternatives, such as TSILs, can be implemented.

   (b) Investigate the properties and behaviors of Task Specific Ionic Liquids (TSILs) to understand their potential as green solvents in organic synthesis processes.

   (c) Develop new methodologies for organic synthesis utilizing TSILs as reaction media, aiming to reduce or eliminate the use of volatile organic solvents and hazardous reagents.

   (d) Assess the efficiency and selectivity of TSIL-mediated reactions compared to conventional methods, focusing on improving yields and minimizing waste generation.

   (e) Explore the recyclability and reusability of TSILs in organic synthesis to establish their long-term sustainability and economic viability.

   (f) Investigate the scalability of TSIL-based organic synthesis processes, considering factors such as cost-effectiveness and feasibility for industrial applications.

   (g) Educate the scientific community and industry stakeholders about the benefits and

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challenges of implementing TSILs in organic synthesis, promoting awareness and knowledge exchange.

(h) Contribute to the development of regulatory frameworks and standards for the safe and responsible use of TSILs in organic synthesis, ensuring compliance with environmental regulations.

(i) Continuously evaluate and optimize TSIL-based organic synthesis approaches through ongoing research and innovation, aiming for continuous improvement in sustainability metrics.

2) Work done so far and results achieved and publications, if any, resulting from the work (Give details of papers and names of the journals in which it has been published or accepted for publicaion)

- Comunicated two research paper in internationally reputed journals.

3) Has the progress been according to original plan of work and towards achieving the objective. if not, state reasons.

- Yes, the progress has been according to original plan of work towards achieving the objective of the project.

4) Please indicate the difficulties, if any experienced in implementing the project.

- Nil

5) If project has not been completed, please indicate the approximate time by which it is likely to be completed. A summary of the work done for the period (Annual basis) may please be sent to the university on a separate sheet.

- Half of the project has been successfully completed, and two research papers have been submitted to research journals. The project will be completed within the stipulated time frame. (Attached a separate sheet for summary of the work done)

6) If the project has been completed, please enclose a summary of the findings of the study. Two bound copies of the final report of work done may also be sent to the Univeristy.

- Nil

7) Any other information which would help in evaluaion of work done on the project. At the completion of the project, the first report should indicate the output, such as (a) Manpowe trained (b) Ph.D. Awarded (c) Publication of results (d) other impact, if any.

- Research papers communicated in the internationally reputed journals.

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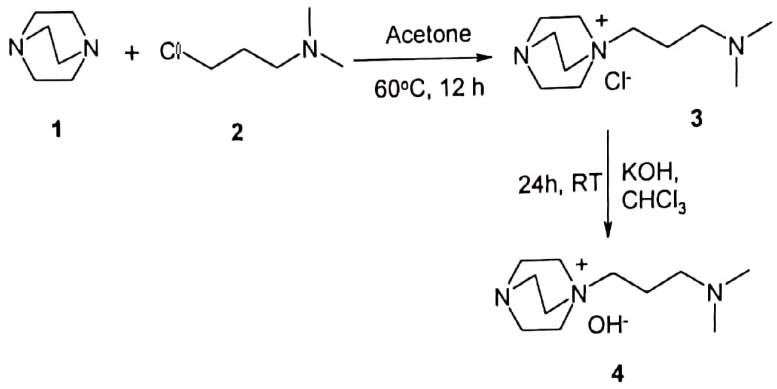
## Summary of the work done

**(Progress report period 1st Nov 2022- 31st Oct 2023)**

### 1. Efficient Synthesis of 2-Pyridones via Multi-Component Reactions Using Novel Dual Basic Ionic Liquid

2-Pyridones exhibit diverse and significant pharmacological properties, making them an essential focus in medicinal chemistry and drug discovery. They have shown their importance as a pharmacophore in anti-tumoral, anti-Alzheimer, anti-heart failure, anti-fibrosis, anti-malarial, analgesic, and anti-HIV activities.1 Researchers have harnessed the versatility of the 2-pyridone scaffold to develop molecules with specific biological activities, often by incorporating additional functional groups or altering the substitution pattern on the pyridine ring. In addition to this 3-cyano-2-pyridone frameworks have found in the structural basis of the alkaloid Ricinine, the first known alkaloid containing a cyano group. The Milrinone is a 3-cyano-2-pyridone derivative that has been used for the treatment of congestive heart failure.[2] Another derivative 3-cyano-2-pyridone has shown anticancer activity which might be due to the interference of molecule with survinin protein.[3] Due to great important of this skeleton, the development of efficient and environmentally begins methodologies for the synthesis of diverse functionalized 2- pyridines is still highly desired.

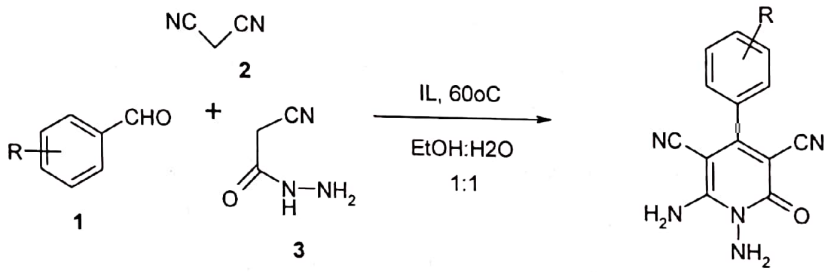
In recent times, the notion of task-specific ionic liquids (TSIL) has gained significant atraction within the realm of synthetic organic chemistry. Ionic liquids (ILs) are designed with specific functional groups, either as cations or anions, with the purpose of enabling one of these components to act as a catalyst in chemical reactions.[4-10] Therefore, an IL can prove valuable if the cations or anions within it serve as a catalyst, catalyst activator, or co-catalyst in a reaction. Certain literature explicitly indicates that ILs are intentionally crafted to have one of their ions function as the catalyst for a reaction. An IL, essentially a salt in its liquid state, primarily comprises ions and short-lived ion pairs. These substances are commonly referred to as liquid electrolytes and are often heralded as "solvents of the future" or designer solvents. ILs are renowned for their diverse potential applications, serving as potent solvents and electrically conducting fluids. Liquid salts, particularly those with low vapor pressure near ambient temperatures, hold significance in electric battery applications and have been contemplated for use as sealants.[11-15] The conventional use of ILs as solvents, owing to their low melting points below 100°C, has evolved, expanding their applications across various domains of organic synthesis. ILs have proven to be exceptionally beneficial in a multitude of analytical applications as well.




1 2 3 4

**Scheme 1: Synthesis of Novel dual basic ionic liquid**

Considering the diverse nature of 2-pyridones and their derivatives, we undertook the challenge of synthesizing them. Therefore, we endeavored to create a system that would not only address this diversity but also contribute to green chemistry. In this context, we synthesized 2-pyridone derivatives using ionic liquid (IL) as a catalyst. The utility of ILs is also expounded upon in the current project.




**Scheme 2: Synthesis of 2-Pyridones**

A convenient and efficient method has been developed for synthesizing diamino-4-phenyl-3,5-dicyano-2-pyridone through a one-pot reaction involving aryl aldehydes, malonitrile, and cyanoacetic acid hydrazide. This reaction is conducted at room temperature using a new dual basic ionic liquid as a recyclable catalyst. The reaction involves an initial Knoevenagel condensation between the aldehyde and malonitrile, followed by a Michael addition reaction with cyanoacetic acid hydrazide, and intramolecular cyclization, leading to the formation of the N-amino-2-pyridone product in good to excellent yields. The chemical structures of the synthesized compounds were confirmed using spectral techniques such as IR, $^{1}H$ NMR, and $^{13}C$ NMR.

**Reusability of Ionic Liquid:**

The main advantage of using IL is that it can be recycled. After completion of the reaction as confirmed by TLC, the solid product formed was filtered from the reaction mixture.

The remaining filterate was evaporated and washed with ethyl acetate to get the IL back. The recovered IL was dried at $60^0$c and used for subsequent reaction. It was found that IL can be used effectively at least six times without any loss in yield of product.

**Conclusion:**

In conclusion, we have synthesized 2-pyridone derivative from aldehydes, malononitrile and cyanoacetohydrazide using ethanol:water (1:1) system at room temperature. This reaction provided an excellent yield, within short time. Advantage of this procedure is it gives high yield, the reaction is completed in short time and also the procedure is simple and catalyst can be reusable.

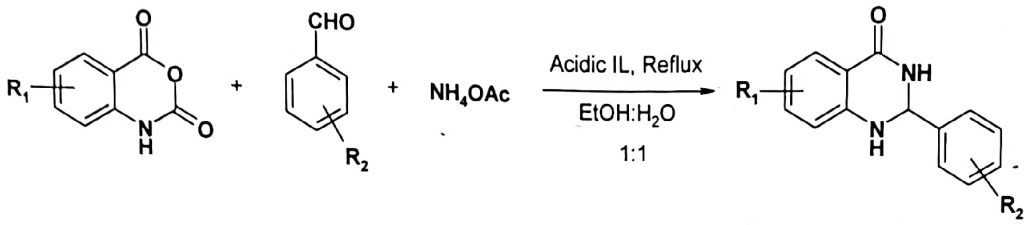
- This work has been communicated in journal, "Research on Chemical Intermediates"

## 2. Acidic ionic liquid catalyzed synthesis of Quinazoline-4-one

The Quinazoline-4-one is an important building block in the synthesis of numerous pharmaceuticals and biologically active compounds. [1] This ring system is studied for its anti-tumor, anti-inflammatory, anti-microbial and analgesic properties, making it a valuable starting material in the development of drugs for various diseases. Additionally, the ketone functional group in quinazoline-4-one can be easily modified to form new compounds with varied biological activities. In recent years, there has been a growing interest in the development of new quinazoline-4-one derivatives for use as therapeutic agents, making it an important area of research in the field of medicinal chemistry. Looking these properties of Quinazoline-4-one are emerged as a versatile agent.

Acidic ionic liquids (AILs) are a class of ionic liquids that have a pH below 7 and contain a significant concentration of hydrogen ions (H+). They are characterized by their unique combination of properties, including high thermal stability, low volatility, high electrical conductivity, and good solubility for both polar and nonpolar compounds. AILs have been attracting increasing attention in various fields due to their potential applications as catalysts, solvents, and electrolytes. In catalytic applications, AILs have been shown to have high catalytic activity and selectivity in various chemical reactions, including esterification, condensation, and oxidation reactions. In addition, AILs can act as effective solvents for polar and nonpolar compounds, making them useful in the extraction and purification of valuable chemicals. AILs also have potential applications in energy storage and conversion, particularly in batteries and supercapacitors. In these applications, AILs can serve as electrolytes, facilitating the flow of ions and electrons in the devices. However, the acidic nature of AILs can also pose some challenges, such as the corrosion of metal components and the degradation of organic compounds.

Therefore, the development of new AILs with improved properties and stability is an active area of research. In conclusion, acidic ionic liquids are a class of versatile materials with a range of potential applications, from catalysts and solvents to energy storage and conversion. Further research is needed to fully realize their potential and address the challenges associated with their acidic nature.




**Scheme 2: Synthesis of Quinozaline derivatives**

**Conclusion:**

A novel green synthetic route was developed for the facile synthesis of quinazoline-4-one derivatives using acidic ionic liquid as a catalyst. The present methodology has a high eco-scale and a low E-factor, which indicates greenness of the reaction. The ease of purification, solvent-free conditions, recyclability of the catalyst and scalability to a multigram scale make the reaction an economical and environmen-tally benign route for the selective synthesis of quinazoline-4-one on the industrial scale.

- This work has been communicated in journal, "Research on Chemical Intermediates"

**Signature of the Principal Investigator:** Dipa

(Dr. D.S. Gaikwad)

**Signature of the Principal:**

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