

# Deep Eutectic Solvent a Highly Efficient Medium for the Synthesis of Imidazo [1, 2-a] Pyridines Having Green Chemistry Approach

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## To cite this article:

Majid Shaikh, Sayyad Sultan Kasim. Deep Eutectic Solvent a Highly Efficient Medium for the Synthesis of Imidazo [1, 2-a] Pyridines Having Green Chemistry Approach. *American Journal of Heterocyclic Chemistry*. Vol. 8, No. 1, 2022, pp. 7-11.

doi: 10.11648/j.ajhc.20220801.12

Received: August 30, 2022; Accepted: September 27, 2022; Published: October 28, 2022

**Abstract:** The imidazo [1, 2-a] pyridine is valuable structural unit in the area of natural products and pharmaceuticals. Extremely effective one pot method developed for the production of imidazo [1, 2-a] pyridines. The reaction of N-bromosuccinimide, acetophenones and 2-aminopyridines in deep eutectic solvent and reaction completed within a minute. The most remarkable features of such reaction is lowest minimum time, high atom, mild reaction condition and step economy. Methods The mixture of substituted acetophenones, N-bromosuccinimide in deep eutectic solvent as a green medium and 2-aminopyridines. The optimization of the reaction conditions with regard to their chemo selectivity of deep eutectic solvent. An imidazopyridine is a nitrogen containing heterocycle which plays crucial role in medicinal and pharmacological chemistry. Results To synthesize the imidazo [1, 2-a] pyridines, In the deep eutectic solvents add N-bromosuccinimide, acetophenones at room temperature immediately reaction completed within a minute, TLC Shows single spot which indicate that formation of  $\alpha$ -bromoketones. On formation of  $\alpha$ - bromoketones; 2-aminopyridine was added in the reaction mass after completion of reaction, the reaction mixture was poured in ice-cold water; the solid product obtained was filtered. **Conclusion** The main remarkable characteristics of this protocol such as no need to isolate lachrymatory  $\alpha$ -bromoketones, clean reaction profile, mild reaction condition, require minimum reaction time, inexpensive and green aspects such as avoid hazardous solvents, poisonous catalyst, higher yield and ease of work-up.

**Keywords:** Deep Eutectic Solvent, Imidazol-Pyridine, Acetophenones, 2-Aminopyridines

## 1. Introduction

The imidazo [1, 2-a] pyridine are typically known for their pharmacological activity as nonsteroidal, anti-inflammatory drugs and it is valuable structural unit present in the natural products.

The imidazo [1, 2-a] pyridine synthesis focuses on the interpretation of synthetic processes by the application of one-pot reaction. The optimization of the reaction conditions with regard to their chemo selectivity of deep eutectic solvent. An imidazopyridine is a nitrogen containing heterocycle which plays crucial role in pharmacological and medicinal chemistry [1-7]. Imidazo [1, 2-a] pyridines are enormously accepted among researcher due to their remarkable pharmacological and

biological activities such as antibacterial [8], antifungal [9], antiviral [10], anti-osteoporotic [11], antiparasitic [12], and antihypertensive agents [13], anti-inflammatory [14], antisthmatic [15], anticancer [16], anticoccidial agents [17], In the imidazo [1, 2-a] pyridine family, several products have proceeded to market, including alpidem (anxiolytic) [18] and HIF-1 $\alpha$  prolyl hydroxylase inhibitory [19]. antiulcer [20], saripidem (anxiolytic) [21], necopidem (anxiolytic) [22], zolpidem (insomnia) [23], minodronic acid (bisphosphonate) [24], olprinone (cardiotonic) [25], zolimidine and divalpon (anxiolytic) [26].

Functionalized imidazo [1, 2-a] pyridines and other imidazo-fused heterocycles are prevalent structural motifs in pharmacological active and biologically important compounds

[8-26]. Currently, multi-component reactions at moderate temperature have acknowledged much attention in the field of synthetic medicinal chemistry and organic chemistry. N-bromosuccinimide (NBS) has emerged as a selective brominating agent, gaining popularity in organic synthesis because it is easy to handle and user friendly [27]. The remarkable advantages of present protocol over other fundamental methods such as it requires shorter reaction time, environmentally benign and products are achieved with good to excellent yield of product [28]. The deep eutectic solvent usage offer a convenient, readily available, non-toxic, safe, and cheap reaction medium for the environ-economic synthesis [29]. The literature survey reveals that diverse types of methods have been described for the synthesis of this important structure, the most exploited one involves 2-aminopyridine as starting material. The main part of the

process involves primary combination reactions between endocyclic nitrogen of 2-aminopyridine and several reagents, such as  $\alpha$ -halo carbonyl compounds [30], oxothioamide [31], 1, 2-diols [32], diazoketones [33] and imidazo [1, 2-a] pyridines. Still, maximum of these methods have suffered with several disadvantages such as use of lachrymatory  $\alpha$ -halo-ketones, monotonous workup procedures, severe heating condition, difficult-to-achieve starting materials, lowering yields, and toxic solvents and catalysts. Herein, we have developed a versatile, environmentally benign, convenient protocol for the one-pot synthesis of different derivatives of imidazo [1, 2-a] pyridines from cyclocondensation of in-situ-generated  $\alpha$ -bromoacetophenones and 2-aminopyridines in deep eutectic solvent under stirring at 75°C afforded imidazo [1, 2-a] pyridine derivatives with high yield [34-42].

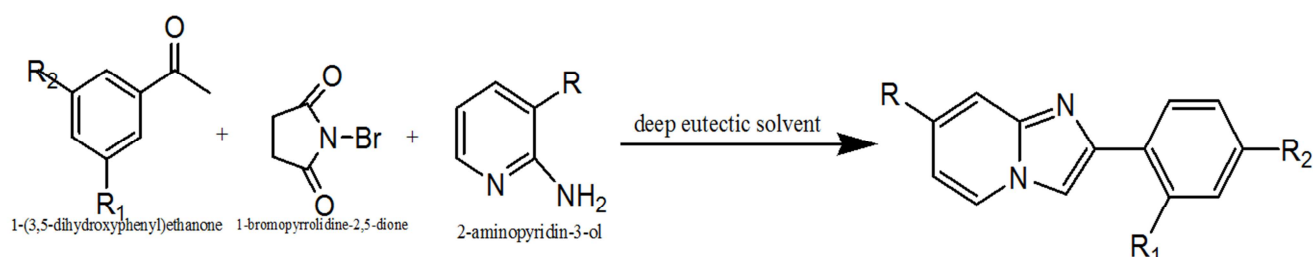


Figure 1. Scheme. One-pot synthesis of imidazo [1, 2-a] pyridines.

## 2. Result and Discussion

To synthesize the imidazo [1, 2-a] pyridines substituted acetophenones, N-bromosuccinimide (NBS) were added in deep eutectic solvents and mixture was stirring at room temperature immediately reaction completed within a minute. This mixture immediately produces single spot in Thin Layer

Chromatography (TLC) which indicate that formation of  $\alpha$ -bromoketones. On formation of  $\alpha$ -bromoketones; 2-aminopyridine was added in the reaction mass and reaction mass was heated at 75°C temperature as soon as single spot obtained in Thin Layer Chromatography (TLC). Once completion of the reaction, the reaction mixture was poured in ice-cold water; the solid product obtained was filtered. The results were reported in Table 1.

Table 1. Synthesis of imidazo [1, 2] pyridines 3 Cu<sub>2</sub>O NPs 5 mol% r.t. Methanol 70.

Product	R	R1	R2	Reaction time (min) b	Yield (%) a
4a	H	H	H	6	93
4b	6-Cl	H	4-Cl	5	93
4c	6-CH <sub>3</sub>	H	4-Cl	6	93
4d	6-CH <sub>3</sub>	2-Cl	4-Cl	5	94
4e	H	H	4-Cl	5	95
4f	H	H	4-Br	5	94
4g	6-CH <sub>3</sub>	H	4-OCH <sub>3</sub>	6	92
4h	H	H	4-OCH <sub>3</sub>	7	92
4i	H	H	4-F	6	94
4j	H	H	4-CH <sub>3</sub>	6	93
4k	6-Cl	H	4-OCH <sub>3</sub>	7	93
4L	H	2-Cl	4-Cl	7	94
4m	6-CH <sub>3</sub>	H	H	6	92

Isolated yield. High yields and shorter reaction time in green medium are the remarkable features.

## 3. Experimental Details

### 3.1. Material and Methods

All the solvents and chemicals were used without further

purification. Melting points were determined in open capillary tubes and are uncorrected. Thin Layer Chromatography (TLC) was used to monitor the reaction. Shimadzu FT-IR-8400 instrument was used for Infrared (IR) analysis. Mass spectra were recorded on Shimadzu GC-MS-QP-2010 model using the direct inlet probe technique. <sup>1</sup>H

NMR and  $^{13}\text{C}$  NMR were recorded in  $\text{CDCl}_3$  solvent on a Bruker AC 300-MHz spectrometer.

Synthesis of (DES) a choline chloride-based deep eutectic solvent:-

Thiourea, urea and choline chloride were heated in the ratio of 1:1:1 at 85-90°C with stirring for 5-10 minutes a clear and homogeneous liquid observed. Then this is used as deep eutectic solvent for the reaction. This deep eutectic solvent was used without further purification. The deep eutectic solvent was characterized which shown below.

$^1\text{H}$  NMR (for DES):-  $\delta$  ppm = 3.09 ppm (s, 9-H); 3.51 ppm (t, 2-H), 3.79 ppm (t, 2-H), 5.61 ppm (t, O-H), 5.72 ppm (N-H), 7.31 ppm (s, N-H).

IR (for DES): -  $\text{cm}^{-1}$  = 3411, 3204, 1676, 1622, 1481.

### 3.2. Importance of Deep Eutectic Solvent

The deep eutectic solvent mediated synthesis of imidazole reaction immediate at room temperature under stirring. The deep eutectic solvent reduces reaction time and reaction completed within 2 minutes. The DES is environmentally benign, low volatile, low toxic, high availability, high recyclability etc.

### 3.3. General Procedure for the Synthesis of 2-Arylimidazo [1, 2-a] Pyridines

In the deep eutectic solvent (10 ml) aromatic acetophenones (0.010 Mol), N-bromosuccinimide (NBS) (0.010 Mol) was added stirring dens fumes observed under stirring within two minute reaction completed. The reaction produces the  $\alpha$ -bromoketones intermediated which was examined by Thin Layer Chromatography (TLC). As soon as single spot observed in Thin Layer Chromatography (TLC) 2-aminopyridines (0.01 Mol) was added in to the reaction mixture and raise temperature up to 75°C. After completion of the reaction, the reaction mixture was poured in ice-cold water; the solid product obtained was filtered, dried and crystallized with aqueous ethanol.

The imidazo [1, 2-a] Pyridines [4a-4m] data characterization.

### 3.4. 2-Phenylimidazo [1, 2-a] Pyridine (4a)

Melting Point = 135-137°C;

IR (KBr):-  $\text{cm}^{-1}$  1636, 768;

$^1\text{H}$  NMR:- (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.47-8.55 (m, 1H), 8.41 (s, 1H), 7.92-7.87 (m, 2H), 7.58 (d, J = 6.9 Hz, 1H), 7.45-7.41 (m, 2H), 7.33-7.28 (m, 1H), 7.22-7.13 (m, 1H), 6.81-6.86 (m, 1H);

$^{13}\text{C}$  NMR:- (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  143.98, 134.10, 133.5, 130.51, 129.94, 129.72, 128.11, 127.47, 114.99, 113.13, 107.35;

### 3.5. 2-(4-Chlorophenyl)-6-Methyl-Imidazo [1, 2-a] Pyridine (4c)

Melting Point = 235-238°C;

$^1\text{H}$  NMR:- (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.87 (d, J = 2.82 Hz, 2H), 7.86 (s, 1H), 7.74 (s, 1H), 7.55 (t, J = 6.05 Hz, 1H), 7.38 (d, J = 8.53 Hz, 2H), 7.07 (d, J = 9.34 Hz, 1H), 2.33 (s, 3H, CH<sub>3</sub>);

$^{13}\text{C}$  NMR:- (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  144.8, 144.4, 133.8,

132.5, 128.9, 128.2, 127.3, 123.3, 122.6, 116.7, 107.9, 18.1;

### 3.6. 2-(4-Bromophenyl)-Imidazo [1, 2-a] Pyridine (4f)

Melting Point = 216-218°C;

IR (KBr):- 1635  $\text{cm}^{-1}$ , 762  $\text{cm}^{-1}$ ;

$^1\text{H}$  NMR  $\delta$  ppm:- 8.50ppm (d, J = 6.4 Hz, 1H), 8.41ppm (s, 1H), 7.91 ppm (d, J = 8.0 Hz, 2H), 7.64ppm (d, J = 8.0 Hz, 2H), 7.56ppm (d, J = 8.8 Hz, 1H), 7.27-7.26ppm (m, 1H), 6.90-6.86ppm (m, 1H).

$^{13}\text{C}$  NMR:-  $\delta$ :- 146.45, 143.16, 135.53, 135.14, 133.22, 130.12, 127.65, 126.02, 116.41, 112.53, 108.53.

### 3.7. 2-(2, 4-Dichlorophenyl)-Imidazo [1, 2-a] Pyridine (4L)

Melting Point = 184-186°C;

$^1\text{H}$  NMR:- (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.28-8.25 (m, 2H), 8.14 (d, J = 6.78 Hz, 1H), 7.61 (d, J = 9.12 Hz, 1H), 7.49-7.46 (m, 1H), 7.35 (dd, J = 2.01 Hz, J = 1.99 Hz, 1H), 7.22-7.18 (m, 1H), 6.83 (t, J = 6.76 Hz, 1H);

$^{13}\text{C}$  NMR:- (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  143.4, 141.7, 134.6, 132.6, 131.6, 130.8, 130.1, 127.6, 125.8, 125.1, 116.9, 112.6, 112.4;

## 4. Conclusion

In the current search, we have developed an naturally benign, facile and extremely competent one-pot protocol for the synthesis of 2-phenylimidazo [1, 2-a] pyridines from the reaction of aromatic ketones, N-bromosuccinimide and 2-aminopyridines in deep eutectic solvent. The main remarkable characteristics of this protocol such as no need to isolate lachrymatory  $\alpha$ -bromoketones, clean reaction profile, mild reaction condition, require minimum reaction time, inexpensive and green aspects such as avoid hazardous solvents, poisonous catalyst, higher yield and ease of work-up.

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