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Nickel Borate Mediated Synthesis of Imidazole-Based Heterocycles

Vishwa Deepak Tripathi*

Department of Chemistry, C. M. Science College, Darbhanga (A Constituent unit of L. N. Mithila University), Darbhanga, Bihar, India

ABSTRACT

The synthesis of 2,4,5-triaryl-1H-imidazole derivatives has been achieved efficiently through a straightforward and versatile multicomponent reaction strategy. In this method, Nickel (II) borate acts as a highly effective catalyst in an acetone medium, promoting the one-pot condensation of a 1,2-dicarbonyl compound, an aromatic aldehyde, and ammonium acetate, which serves as the *in situ* ammonia donor. This catalytic system provides a convenient and environmentally benign alternative to conventional protocols, minimizing the need for harsh reaction conditions or complex purification steps. The reaction proceeds smoothly with good compatibility toward a variety of aromatic aldehydes, thereby furnishing the desired triaryl-imidazole scaffolds in moderate to excellent yields within a relatively short reaction time. The advantages of this methodology include operational simplicity, mild conditions, low cost of catalyst, and reproducibility, making it a practical approach for the synthesis of imidazole-based heterocycles. Such compounds are of significant interest due to their broad applications in medicinal chemistry and materials science.

Key words: 2,4,5-Triaryl-1H-imidazole, Multicomponent reaction, Nickel (II) borate, Satisfactory product yields, Simple workup.

1. INTRODUCTION

Heterocyclic compounds are cyclic organic compounds showing the presence of one or more atoms of elements other than atoms of the basic element that is carbon. Most of the Heterocyclic compounds contain Nitrogen, Oxygen, or Sulfur as part of the ring system [1]. The research has established that natural and synthetic Heterocyclic compounds possess medicinal properties [2]. This has been proved to be an encouraging factor to the ongoing research in the field of Heterocyclic compounds.

Even if several Heterocyclic compounds exist, those with nitrogen as a heteroatom held the prime place [3]. Due to the diverse biological activities, the development of the systematic synthesis of nitrogen-containing heterocyclic compounds has been the subject of interest for researchers all over the world [4].

Among the several Heterocyclic compounds, Triarylimidazoles are found to be unique one as these structural units provide biological activities to many natural as well as synthetic organic compounds [5]. Further, the herbicidal [6], fungicidal [7], anti-cancer [8], antithrombotic nature [9], antiwear [10], and many other biological activities of Triaryl imidazole derivatives [11] have been well explained in the literature. Due to this, Triarylimidazoles have attracted the attention of researchers, resulting in the development of several methodologies for their synthesis.

Triarylimidazoles are generally prepared using a multicomponent pathway [12]. Metal salts in different forms have been used; for example, Nickel Chloride, Ceric ammonium nitrate, Zirconyl Chloride, Uranyl Nitrate, MnO_2-$FeSO_4$, Mn^{2+} Complex of [7-Hydroxy-4-methyl-8-coumarinyl] Glycine [13]. Silica-based catalysts, such as Silica Chloride and Sulfuric acid immobilized on silica gel, are also reported for the synthesis of Triarylimidazoles [14]. Ionic liquid-based catalyst systems are found to be effective [15]. The other protocols include PEG-400, Fe_3O_4@ SiO_2-ABMA-$MnCl_2$ nanocomposite, Co (ii)–Cu (ii) mixed-oxide, Sulfated tin oxide, and molecular sieve-supported titanium catalyst [16]. Some green protocols used glutamic acid, lemon juice, and nano ZnS-$ZnFe_2O_4$ [17].

In the field of Organic synthesis, nickel compounds occupy a prime position due to their catalytic efficacy, which is consistently highlighted throughout the relevant literature [18]. The present research work would be communicating the catalytic application of Nickel (II) borate [$Ni_3(BO_3)_2$] in a synthetic protocol to prepare 2,4,5-Triaryl-*1H*-Imidazoles [Scheme 1].

2. MATERIALS AND EXPERIMENTAL DETAILS

Chemicals procured from Sigma-Aldrich and Merck were used without further purification. Melting points were determined using the open capillary method with a Thiele's tube. Thin-layer chromatography (TLC) was employed to assess the reaction completion. The system used consisted of a mixture of Pet ether and Ethyl acetate in required proportions. The crude products were purified using recrystallization with a suitable solvent. The formation of the 2,4,5-triaryl-1H-

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***Corresponding Author:**
Vishwa Deepak Tripathi
E-mail: vdtmkclnmu@gmail.com

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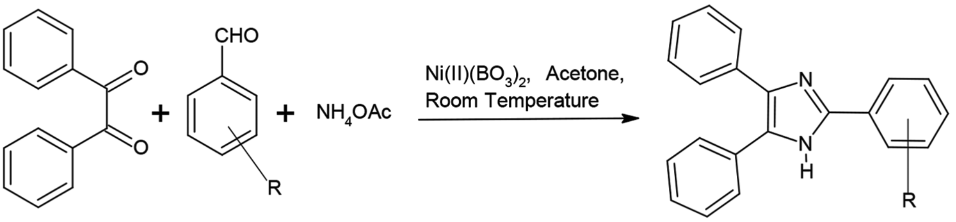
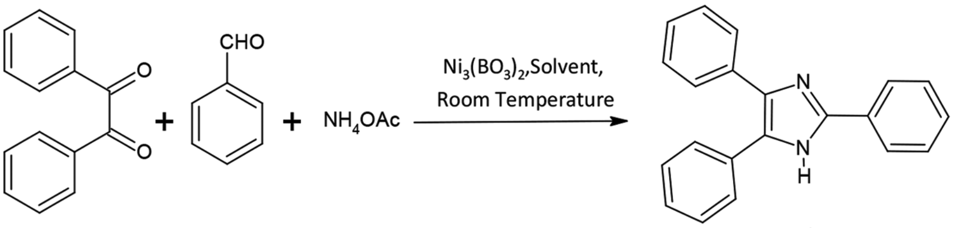


1 a-j

Scheme 1: 2,4,5-Triaryl-1H-Imidazoles.



1 a

Scheme 2: Synthesis of 2,4,5-Triaryl-1H-imidazole derivatives from Benzil, Benzaldehyde, and Ammonium acetate.

imidazoles was proved by the agreement between their melting points and previously reported values in the literature.

2.1. General Procedure

A reaction mixture comprised of Benzil (0.210 g, 1 mmol), an aromatic aldehyde (1 mmol), and ammonium acetate (0.154 g, 2 mmol) in Acetone as solvent (5 mL) was prepared. Nickel (II) borate (0.2 mmol) was then added to serve as a catalyst in the reaction. Thorough mixing of the reaction contents was ensured by employing a magnetic stirrer equipped with a magnetic needle at room temperature. Upon the completion of the reaction, as confirmed using TLC, the reaction mixture was filtered to remove the heterogeneous catalyst. The collected filtrate was then carefully transferred into a beaker of cold water (10 ml) for solidification of the product. Washing with cold water followed by recrystallization from hot ethyl alcohol gave the purified product. The prepared 2,4,5-triaryl-1H-imidazoles were characterized by melting point data.

The optimal experimental parameters for the synthesis of 2,4,5-Triaryl-1H-imidazole derivatives were elucidated by selecting a reaction involving Benzil, Benzaldehyde, and Ammonium acetate as a model reaction [Scheme 2].

The selection of a suitable solvent for the reaction was done by performing the model reaction in a series of solvents and analysing its effect on reaction time and product yield. Initially, the model reaction was performed in non-polar solvents, such as CCl_4, CH_2Cl_2, and $CHCl_3$. Further, it was performed with polar solvents, such as CH_3CN, C_2H_5OH, Acetone, DMF, DMSO, and H_2O. On the basis of analysis of data for reaction time and product yield received with different solvents, Acetone was found to be the most appropriate solvent for the reaction [Table 1, Entry 9].

The further investigation involved finding out the optimal catalyst load [Table 2, Entry 1-6]. The data mentioned in Table 2 demonstrate that 0.2 mmol of the catalyst could gave maximum yield of 2,4,5-Triaryl-1H-imidazole (1a) in 52 min. An increase in the yield of product was not observed with further increment in catalyst load.

Table 1: Section of solvent for the synthesis of 2, 4, 5-Triaryl-1H-imidazole (1a)

Entry	Catalyst	Solvent	Reaction time (Min)	Yield^a (%)
1	$Ni_3(BO_3)_2$	CCl_4	180	<5
2	$Ni_3(BO_3)_2$	CH_2Cl_2	120	20
3	$Ni_3(BO_3)_2$	$CHCl_3$	240	20
4	$Ni_3(BO_3)_2$	CH_3CN	150	40
5	$Ni_3(BO_3)_2$	DMF	110	39
6	$Ni_3(BO_3)_2$	DMSO	120	55
7	$Ni_3(BO_3)_2$	C_2H_5OH	80	45
8	$Ni_3(BO_3)_2$	Acetone	50	88
9	$Ni_3(BO_3)_2$	H_2O	70	35

^{a}Isolated yields

Table 2: Optimal catalyst load for the synthesis of 2, 4, 5-Triaryl-1H-imidazole (1a)

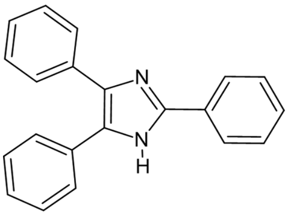
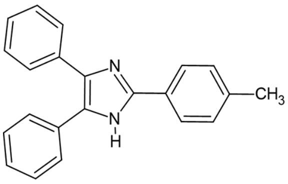
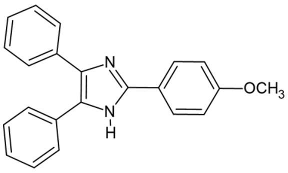
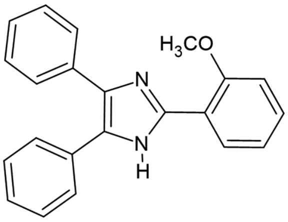
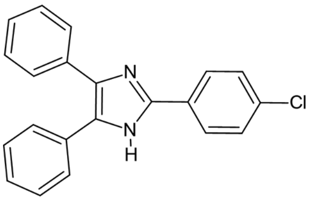
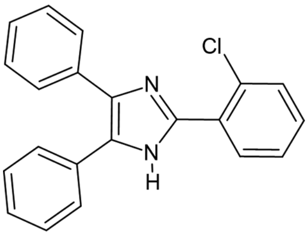
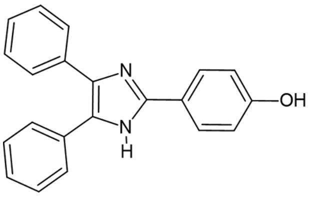
Entry	Catalyst (mmol)	Solvent	Reaction time (min)	Yield^a (%)
1	0.01	Acetone	120	49
2	0.05	Acetone	90	65
3	0.1	Acetone	86	69
4	0.2	Acetone	52	89
5	0.3	Acetone	54	88
6	0.4	Acetone	52	89

^{a}Isolated yields

3. RESULTS AND DISCUSSION

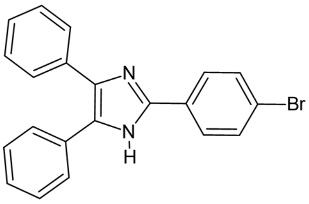
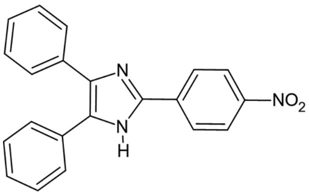
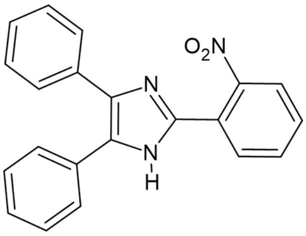
The applicability of the reaction for the synthesis of 2,4,5-Triaryl-1H-imidazoles was evaluated through the investigation of the reaction of Benzil with a range of aromatic aldehydes and ammonium acetate under the previously optimized conditions [Table 3, 1a-j]

Table 3: Synthesis of 2,4,5-Triaryl-1H-imidazoles (1a-j)

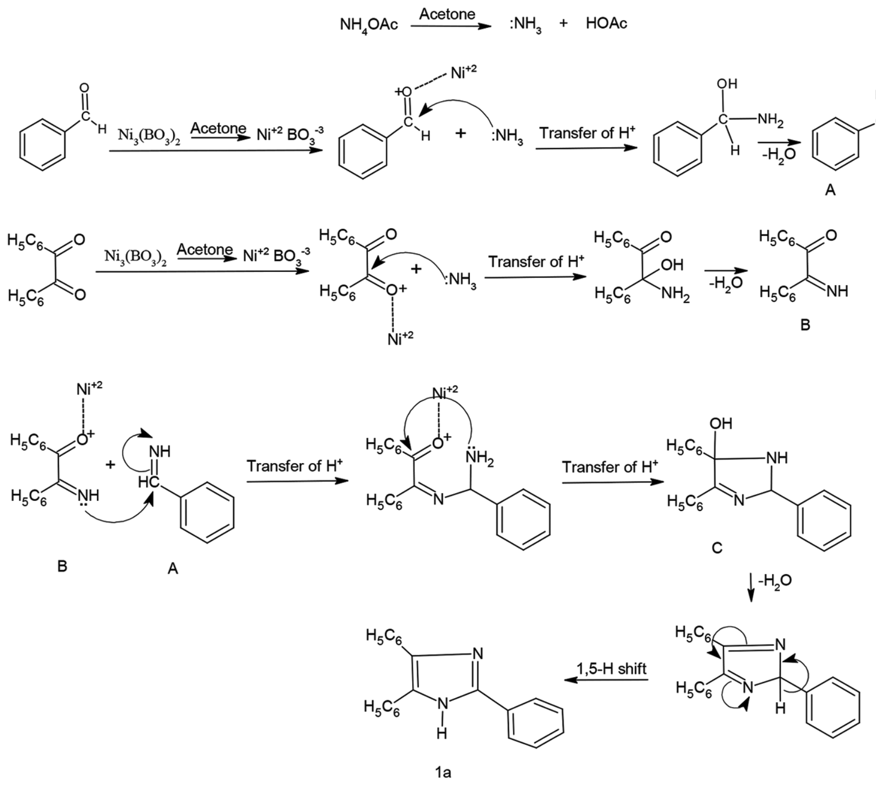
Entry	R-	Product	Time (min)	Yield^a (%)	Melting point (°C) (Literature value)
1	H	 [2,4,5-Triphenyl-1H-imidazole] (**1a**)	52	88–89	266–267 (267–269) [19]
2	4-CH_3	 [2-(p-tolyl)-4, 5-diphenyl-1H-imidazole] (**1b**)	60	77	228 (227–229) [20]
3	4-OCH_3	 [2-(p-methoxyphenyl)-4, 5-diphenyl-1H-imidazole] (**1c**)	70	72	221–222 (220–223) [19]
4	2-OCH_3	 [2-(o-methoxyphenyl)-4, 5-diphenyl-1H-imidazole] (**1d**)	80	65	209–210 (210–211) [13]
5	4-Cl	 [2-(p-chlorophenyl)-4, 5-diphenyl-1H-imidazole] (**1e**)	48	88	263 (262–264) [19]
6	2-Cl	 [2-(o-chlorophenyl)-4, 5-diphenyl-1H-imidazole] (**1f**)	44	85	187–188 (186–190) [20]
7	4-OH	 [2-(p-hydroxyphenyl)-4, 5-diphenyl-1H-imidazole] (**1g**)	42	90	231 (232–233) [19]

(*Contd...*)

Table 3: *(Continued)*

Entry	R-	Product	Time (min)	Yield^a (%)	Melting point (°C) (Literature value)
8	4-Br	 [2-(p-bromophenyl)-4, 5-diphenyl-1H-imidazole] (**1h**)	45	88	249–250 (248–249) [21]
9	4-NO_2	 [2-(p-nitrophenyl)-4, 5-diphenyl-1H-imidazole] (**1i**)	40	90	235–236 (235–238) [22]
10	3-NO_2	 [2-(o-nitrophenyl)-4, 5-diphenyl-1H-imidazole] (**1j**)	45	87	231 (230–231) [19]

*Reaction condition: Benzil (1.0 mmol), aldehyde (1.0 mmol), ammonium acetate (2 mmol), $Ni_3(BO_3)_2$ (0.2 mmol) in Aetone (5.0 ml) at Room Temperature, ^{a}Isolated Yields



Scheme 3: Mechanism.

The catalytic efficacy of $Ni_3(BO_3)_2$ in Acetone has been successfully proved as it smoothly facilitated the reaction between Benzil, Aldehyde, and Ammonium acetate to form 2,4,5-Triaryl-1H-Imidazoles [Table 3]. The reaction has occurred under mild conditions with moderate to good yields of products. As a Lewis acid, $Ni_3(BO_3)_2$ proved to be an efficient catalyst under mild conditions with moderate to good yields. $Ni_3(BO_3)_2$ as a Lewis acid was found to be reacting well with various aromatic aldehydes irrespective of their electronic nature or substitution pattern [Table 3].

Reaction rate and yield of products were significantly influenced by the nature and position of substituents on Aromatic aldehydes. Electron-withdrawing substituents must be stabilizing the transition state, due to which Aromatic aldehydes bearing such groups consistently afforded high yields within the shortest reaction duration [Table 3, Entries 1e,1g-i]. In contrast, the presence of electron-donating substituents led to a decrease in product yield and an increase of the reaction rate [Table 3, Entries 1b,1c]. The reduced reactivity of aromatic aldehydes with ortho-substituent suggests a significant steric hindrance effect near to reaction site [Table 3, Entries 1d,1j]. Notably, aliphatic aldehydes were completely unreactive under the employed conditions, highlighting the specific requirements for an aromatic system in this transformation.

On the basis of findings, the plausible mechanistic pathway for the reaction is illustrated in the scheme 3, it seems that the catalyst must have activated both an aromatic aldehyde and Benzil via coordinating with the oxygen atom of the carbonyl group, which was followed by nucleophilic attack of the ammonia molecule to form Imine intermediates (A and B). Further, Imine intermediate (B), activated by the catalyst, reacted with Imine intermediate (A) and finally gave the desired product (1a-j).

4. CONCLUSION

The use of $Ni_3(BO_3)_2$ as a catalyst in Acetone presents a highly advantageous protocol for synthesizing 2,4,5-Triaryl-1H-imidazoles via a one-pot, three-component reaction. This catalyst offers good performance along with other benefits, including easy separation due to its heterogeneous nature, cost-effectiveness, and ready availability. The use of $Ni_3(BO_3)_2$ as a catalyst facilitates smooth reactions with various aromatic aldehydes, achieving good yields of the target imidazole derivatives in a short duration. Further, the catalyst was effective in small quantity (0.2 mmol) to carry out transformations.

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