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DBUHI3-catalyzed efficient synthesis 2,4,5-triaryl substituted imidazoles

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The new organocatalyst amine-iodine-iodide complex ($DBUHI_3$) was prepared and used as a catalyst for the synthesis of 2,4,5-trisubstituted imidazoles in moderate to good yields from inexpensive, commercially available and eco-friendly ammonium acetate as a nitrogen source, 1,2-diketones and substituted aromatic aldehydes. The direct one-step multicomponent efficient synthesis was achieved with remarkable green advantages offered by this protocol such as short reaction time, the broad scope of a substrate, and simple experimental procedure.

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1. Introduction

Heterocyclic compounds have attracted a lot of study interest because of their many crucial biological and therapeutic uses.[1-5] They bridge the gap between chemistry and biology, where much of the scientific progress and application occurs, and are found in more than 90% of novel drugs. The most common heteroatoms are nitrogen, oxygen, and sulfur, although heterocyclic rings with other heteroatoms like phosphorus, iron, magnesium, selenium, etc., are also often encountered. Heterocyclic nitrogen-containing molecules are vital in medical chemistry due to their many applications.[6-9]

A series of reactions that have been reported in the literature were triggered by the first multicomponent reaction, which is attributed to Strecker and dates to 1850.[10] A special type of synthetically valuable organic reactions known as multicomponent reactions (MCRs) involves combining one or more diverse starting materials in a single pot to create a final product. In a variety of synthetic transformations, where conventional methods usually need a drawn-out procedure with many stages, these reactions have been carefully employed. The MCR approach is very helpful in the drug development process because it offers several advantages, including high yields, atom-/step economy, reduced reaction durations, environmental friendliness, and a practical tool for creating a library of new chemical entities (NCEs).[11–17] Since heterocyclic scaffolds often comprise more than two building units, the production of highly convergent syntheses has been a constant in heterocyclic chemistry.[18-19] Since the beginning of organic chemistry, the successful development of heterocycles into functional compounds has been intimately associated with their synthesis by multicomponent reactions (MCRs).

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Imidazoles are a class of heterocycles that have garnered a lot of attention due to their diverse structural functions and biological activity (**Fig. 1**). It is a nitrogen-containing aromatic heterocyclic molecule with five members that is found in many natural products. The therapeutic index of imidazole pharmacophore may be attributed to its metal affinity and hydrogen bond donor-acceptor capacity. Imidazole compounds are a significant heterocyclic scaffold that have gained interest due to its numerous industrial uses in pharmacology and agrochemistry.[20-24] Furthermore, imidazole derivatives are employed as green organocatalysts, ionic liquids, pigments, and functional materials.[25-26]

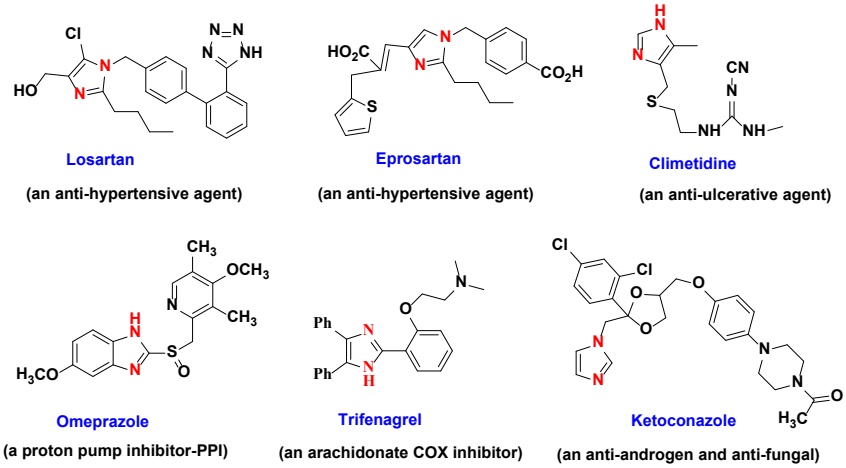


Fig. 1. Drug molecules containing an imidazole ring system

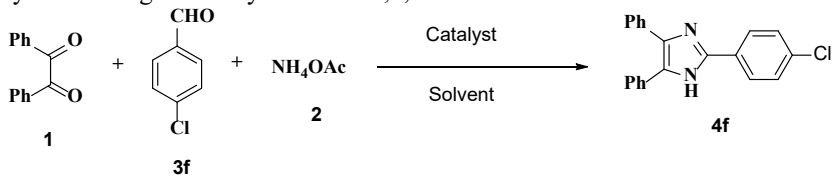
Trisubstituted imidazoles are a significant family of heterocycles that are frequently used as fine-chemical, fungicide, and medicinal intermediates.[27-28] As a result, the creation of effective processes for the synthesis of this class of imidazoles has drawn increasing attention. One of the most well-known common processes among them is the multicomponent condensation of diketones, aldehydes, and a nitrogen source to create 2,4,5-trisubstituted imidazoles. For this function, nitrogen is often obtained *via* ammonium salts, hexamethyldisilazane, amines, amidines, imidines, azides, nitriles, or ^{t}BuONO/nitrones.[29-35] In terms of reactivity, mildness, cost-effectiveness, and the ability to create NH_3 *in situ*, NH_4OAc is the best nitrogen source among ammonium salts when compared to NH_4Cl, $HCOONH_4$, $(NH_4)_2CO_3$, NH_4Cl, and urea. In general, protic and Lewis acids can act as catalysts to facilitate one-pot condensation. Each synthetic process has advantages and disadvantages, according to the synthesis method assessment of 2,4,5-trisubstituted imidazoles.[36-52] Long reaction times, costly reagents, strict reaction conditions, low selectivity, laborious stages (making a nanocatalyst), environmental contamination, and unique circumstances (grinding, high temperatures, ultrasound, irradiation) are the drawbacks of many earlier techniques. Therefore, there is plenty of room to create a novel, effective pyrazole synthesis process. The creation of effective catalytic systems for the synthesis of 2,4,5-trisubstituted imidazoles has received a lot of attention in this area. In recent years, our laboratory prepared $DBUHI_3$ for the first time and used it as a new organocatalyst to synthesize several heterocyclic scaffolds, including benzothiazoles, pyrazoles, and benzimidazoles.[53-54] Although molecular iodine has disadvantages including sublimation and moisture sensitivity, iodine catalysis, which has been around for more than a century, has significant benefits over transition metal base catalysts. To get around this problem, use DBU as a precursor to change molecular iodine into amine-iodine-iodide complexes. In keeping with these advancements, we put forth our initial results for the synthesis of 2,4,5-trisubstituted imidazoles employing $DBUHI_3$ as an effective organocatalyst. As far as we are aware, there is no report on the use of the amine-iodine-iodide complex as a catalyst in the production of substituted imidazoles. With its good substrate and functional group compatibility, low cost, mild conditions, easy and rapid product separation, and use of an organocatalyst, the current technique provides a sustainable and environmentally beneficial method.

2. Results and Discussion

For the synthesis of corresponding imidazole derivatives, we have first selected *p*-chlorobenzaldehyde (**3f**, 1 mmol), benzil (**1**, 1 mmol), and ammonium acetate (**2**, 2 mmol) as model reaction under reflux conditions to assess under different reaction conditions in the presence of amineH-I_3 complexes as catalysts. From **Table 1**, it is very clear that morpholineH-I_3, urotropineH-I_3, piperazineH-I_3, N-Me piperazineH-I_3 resulted in trace amounts of desired product (entries 2-5, Table 1). Molecular iodine proved to be reasonably good in terms of yield (entry 6, Table 1). $DBUHI_3$ proved to be the best among tested catalysts (entry 7, Table 1).

Table 2 displays the results of studies on solvent and catalyst loadings. According to **Table 2**, acetic acid was the best solvent among those examined, and the ideal reaction conditions for assessing the following set of divergent aldehydes were found to be 15 mol% $DBUHI_3$.

Table 1. Study of catalyst screening for the synthesis of 2,4,5-trisubstituted imidazoles



Entry	AmineH-I_3 complexes	Acetic acid (Yield %)
1	No catalyst	25
2	morpholineH-I_3	78
3	urotropineH-I_3	trace
4	piperazineH-I_3	68
5	N-Me piperazineH-I_3	72
6	I_2	80
7	**DBUH-I_3**	**87**

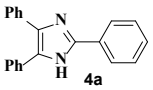
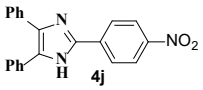
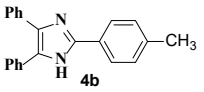
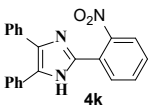
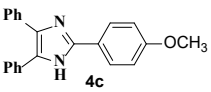
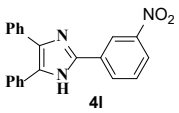
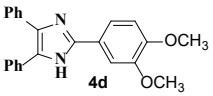
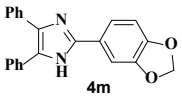
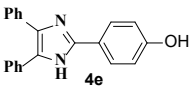
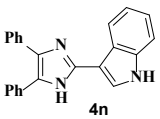
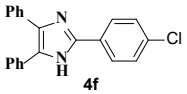
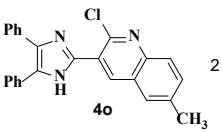
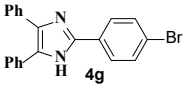
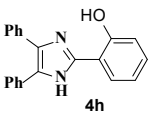
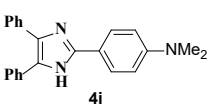
[a]Isolated yields

Table 2. Solvent effect

Entry	Solvent	Yield (%)[a]
1	**Acetic acid**	**87[b]**
2	DMSO	81
3	Dioxane	68
4	Methoxy ethanol	63
5	Ethanol	62
6	Acetonitrile	50
7	DMF	64
8	Isopropanol	49

[a]Isolated yields. [b]67% (5 mol% $DBUHI_3$), 78% (10 mol% $DBUHI_3$)

Table 3. DBUH-I_3 catalyzed synthesis of divergent 2,4,5-trisubstituted imidazoles

Product (4)	Time (h)	Yield (%)[a]	Product (3)	Time (h)	Yield (%)[a]
 4a	1,5	94	 4j	1.5	90
 4b	2	64	 4k	2	86
 4c	2	68	 4l	2	81
 4d	2.5	45	 4m	2	87
 4e	2	44	 4n	2	83
 4f	1	87	 4o	2	61
 4g	1.5	85			
 4h	1.5	83			
 4i	1.5	85			

[a]Isolated yields

The best conditions for making 2,4,5-trisubstituted imidazoles were identified, and then a number of benzaldehydes were used to produce the appropriate products. **Table 3** presents the results that were achieved. Good to high yields of the matching imidazoles were obtained from a range of benzaldehydes that contained electron-donating and electron-withdrawing groups. In contrast to benzaldehydes with electron-donating functional groups (**4b-4e**, and **4h**), the presence of electron-withdrawing groups (**4f. 4g. 4i, 4j-l**) on the benzaldehyde produced greater yields of the desired imidazoles. Steric factors have a significant impact in influencing product yields, as demonstrated by the presence of a -NO_2 group at the *ortho*-, *meta*-, or *para*-position of benzaldehyde (**4j-l**, **Table 3**). Furthermore, we assessed the range of substrates for many additional heterocyclic aldehydes, and the corresponding products were produced in moderate yields (**4n** and **4o**, respectively). Only aromatic aldehydes were studied since no desirable product was produced when aliphatic aldehydes like acetaldehyde and isobutyraldehyde interacted under ideal reaction conditions. Instead of using benzil (1,2-diketones), we used benzoin and the yields from control tests on *p*-ClPhCHO (68%) were lower than those from benzil (87%).

3. Conclusions

We have shown an alternative protocol for the synthesis of 2,4,5-trisubstituted imidazole using $DBUHI_3$ as a novel catalyst and acetic acid as solvent. The reaction condition is mild and the reagent is readily available. This method provided better performance and higher product yield for various aromatic aldehydes containing electron-donating and electron-withdrawing groups. Aliphatic aldehydes were found unsuitable for this methodology.

4. Experimental

General experimental procedure*: In 10 mL round bottom flask aromatic aldehyde (1 mmol), benzil (1 mmol) and ammonium acetate (2.5 mmol) in acetic acid (2 mL) were added. To this reaction mixture, 15 mol% of DBUH-I_3 was added as catalyst and then reaction mixture was heated at reflux for an appropriate time (2 h). The progress of reaction was monitored with the help of TLC (hexane: ethyl acetate 8:2 v/v). After completion of reaction, the mixture was poured over crushed ice (100 g) and neutralized by saturated solution of sodium thiosulfate. The solid obtained was filtered and purified by recrystallization in ethanol. Please find supporting inform for more details.*

2,4,5-Triphenyl-1H-imidazole (Table 3, 4a)

White solid; mp: 276–277 °C. IR (KBr, cm^{-1}): 3400 (N–H), 1605 (C=N), 1498, 1445 (C=C aromatic), 758, 695 (C–H bending). ^{1}H NMR (500 MHz, $CDCl_3$): δ 12.69 (s, 1H, NH), 8.09 (t, J = 8.1 Hz, 3H, Ar–H), 7.52 (d, J = 7.5 Hz, 5H, Ph-H), 7.48 (q, J = 7.5 Hz, 3H, Ar–H), 7.36–7.39 (m, 4H, Ar–H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.2 (C=N), 146.0 (C–N), 133.5, 130.7, 129.2, 128.9, 128.8, 128.2, 127.6, 125.7 (aromatic C). HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{16}N_2$: 296.1336; found: 297.1340

Supporting information is available

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