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Environmentally Benign Synthesis of Tetra-Substituted Imidazoles through 4-Components Strategy Mediated by (*S*)-3-Methyl-1, 1-Diphenylbutane-1, 2-Diamine

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Abstract

(*S*)-3-Methyl-1, 1-diphenylbutane-1, 2-diamine has been found to be a mild and effective organocatalyst for one-pot 4-components synthesis of 1, 2, 4, 5-tetra-substituted imidazoles. The key benefits of this protocol is high yielding, cost effectiveness, easy purification and above all, environmentally benign.

Keywords: Multicomponent reaction; One-pot synthesis; (S)-3-methyl-1, 1-diphenylbutane-1, 2-diamine; Organocatalyst.

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1. Introduction

Substituted imidazoles are the most abundant heterocycles found in bioactive natural products and are pharmacologically attractive compounds. Importantly imidazole core units have myriad pharmacological impacts and play an important role in different biochemical processes. Modern research has revealed that, highly substituted imidazole derivatives possess a good photo-physical properties and are extensively used as antimicrobial [1], antimalarial [2], anticancer [3], antitumor [4], antibacterial [5], antifungal [6], antidepressant [7], antipyretic [8], anti-inflammatory [9] and antioxidant agents [10]. Because of the importance of imidazoles, there is a continued strong demand for new methods of construction of these heterocycles.

A lot of synthetic routes are available in the literatures to synthesize imidazoles [11] analogues including annulations of amidines and β-keto esters [12], SBA-Pr-SO_3H [13], nanocrystalline $MgAl_2O_4$ [14], Cellulose/ɤ-Fe_2O_3/Ag [15], urea/hydrogen peroxide (UHP) [16], N-acetyl glycine (NAG) [17], ($NiFe_2O_4$@SiO_2–$H_3PMo_{12}O_{40}$) [18], [Hmim]TFA

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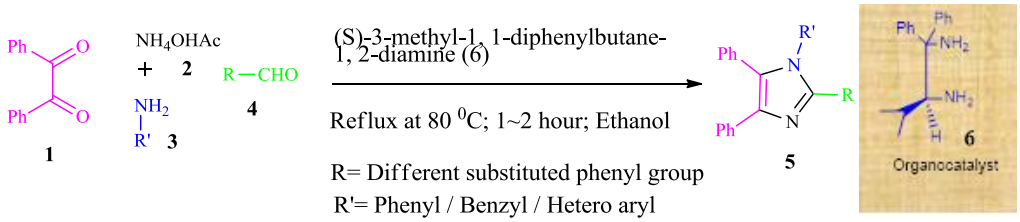
[19], pivalic acid [20], $[Et_3NH][HSO_4]$ [21], ZnO-nano tubes [22], nano-$TiCl_4.SiO_2$ [23], MoO_3/SiO_2 [24], ammonium metavanadate [25], $[CH_2]_4SO_3HMIM][HSO_4]$ [26], KH_2PO_4 [27], 2-cyanopyridine [28], *p*-TsOH [29], cellulose sulfuric acid [30], ceric (iv) ammonium nitrate (CAN) [31], boric acid [32], PEG-400 [33], $InCl_3.3H_2O$ [34], $BF_3.SiO_2$ [35], potassium-aluminum sulfate [36], sodium bisulfite [37], $ZrOCl_2.8H_2O$ [38], microwave irradiation [39], $NiCl_2.6H_2O$ [40], $K_5CoW_{12}O_{40}.3H_2O$ [41], [Bmim]Br [42], NH_4OAc ([Hbim]BF_4) [43], eutectic solvents [44], iodine [45], $Yb(OTf)_3$ [46], $ZrCl_4$ [47], AcOH [48], disubstituted alkynes [49] are noteworthy. However, almost all of these methods suffer from one or more constraints such as lower yield, harsh reaction conditions, tiresome isolation technique, uses of expensive toxic metal catalysts, limited examples etc. Despite that, very few examples of multicomponent reactions mediated by organocatalysts towards imidazole synthesis are available in the literature [33]. Hence development of a mild, economical and complementary approach for 1, 2, 4, 5-tetra-substituted imidazole derivatives are highly demandable.

Based on present demands of tetra-substituted imidazoles for many areas, to explore a new but known [50] and efficient organocatalyst and to obviate the difficulties of the avilable methods, herein, we report a multicomponent strategy *(*Scheme 1*)* to condense benzil/benzoin, different aryl aldehydes, ammonium acetate, benzylamine, aniline or aniline derivatives by (S)-3-methyl-1, 1-diphenylbutane-1, 2-diamine **6**, which is not used as catalyst in earlier for such type of reactions.



Scheme 1

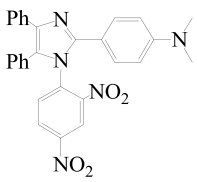
2. Experimental

The melting points were determined on a capillary melting point apparatus and are uncorrected. Infrared spectra were recorded using KBr pellets for solids and neat for liquids on FT-IR 8400 Perkin-Elmer 883 grating spectrometer. ^1H NMR spectra were taken on AC-Bruker 500 MHz spectrometer in D_6-DMSO or $CDCl_3$, containing TMS as internal standard. All *J* values are given in *Hz*, chemical shifts are in δ-units. Reactions were monitored by TLC and column chromatography was carried out on 60-120 mesh E. Merck silica gel. 2-Methylfuroic acids were purchased from Fluka Chemicals.

2.1. *General methods for the preparation of different tetra-substituted imidazoles*

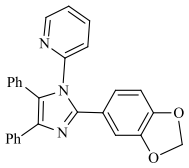
A mixture of benzil or benzoin (2 mmol), aldehyde (2 mmol), ammonium acetate (5 mmol), aryl amine (2 mmol) and (S)-3-methyl-1, 1-diphenylbutane-1, 2-diamine (10 mol %) in ethanol (2 mL) was stirred at reflux temperature for 1~2 hours. The progress of the reaction was monitored by TLC. After completion of reaction, the mixture was cooled to room temperature, diluted with water and poured on crushed ice. The obtained crude solid product was filtered, dried and finally recrystallized from ethanol to obtain sufficiently pure product **5**.

2.2. *Spectral data*



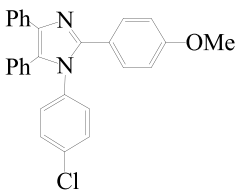
Compound 1

M. p: 218-219 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ = 9.10 (d, *J* = 2.6 Hz, 1H, Ar-H), 8.12 (dd, *J* = 2.6, 9.3 Hz, 1H, Ar-H), 7.77 (dd, *J* = 1.9, 7.0 Hz, 2H, Ar-H), 7.55 (d, *J* = 7.3 Hz, 4H, Ar-H), 7.33 (t, *J* = 7.6 Hz, 4H, Ar-H), 7.27 (t, *J* = 7.7 Hz, 2H, Ar-H), 6.82 (d, *J* = 9.3 Hz, 1H, Ar-H), 6.76 (d, *J* = 8.9 Hz, 2H, Ar-H), 3.30 (s, 6H, 2× CH_3); **^{13}C NMR** (300 MHz, $CDCl_3$): 150.8, 148.5, 147.0, 129.6, 128.6, 127.8, 127.2, 126.4, 123.9, 119.0, 117.8, 112.1, 40.3 *ppm*; HRMS: calcd. for $C_{29}H_{23}N_5O_4$, 505.5272; found: 505.5275.



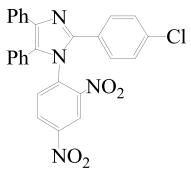
Compound 2

M. p: 180-181 °C; **^{1}H NMR** (500 MHz, $CDCl_3$ with few drops of DMSO): δ = 7.45 (dd, *J* = 7.9, 8.2 Hz, 2H, Ar-H), 6.87-7.29 (m, 12H, Ar-H), 6.86 (dd, *J* = 5.15, 5.35 Hz, 2H, Ar-H), 6.69 (dd, *J* = 8.60, 8.75 Hz, 2H, Ar-H), 3.67 (s, 3H, -OCH_3); **^{13}C NMR** (300 MHz, $CDCl_3$ with few drops of DMSO): 159.6, 146.8, 135.6, 131.0, 130.3, 130.2, 129.6, 129.2, 128.4, 128.1, 128.0, 127.1, 126.5, 122.7, 113.6, 55.1 *ppm*; HRMS: calcd. for $C_{28}H_{21}ClN_2O$, 436.9346; found: 436.9349.



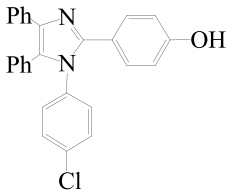
Compound 3

M. p: 252-253 °C; **^{1}H NMR** (500 MHz, $CDCl_3$ with few drops of DMSO): δ = 7.57-7.65 (m, 6H, Ar-H), 7.26-7.40 (m, 6H, Ar-H), 6.81-6.87 (m, 1H, Ar-H), 5.98 (t, *J* = 4.7 Hz, 2H, -CH_2-); **^{13}C NMR** (300 MHz, $CDCl_3$ with few drops of DMSO): 147.8, 147.6, 146.1, 128.2, 128.0, 126.9, 124.9, 119.6, 108.3, 106.3, 101.0 *ppm*; HRMS: calcd. for $C_{27}H_{19}N_3O_2$, 417.4634; found: 417.4632.



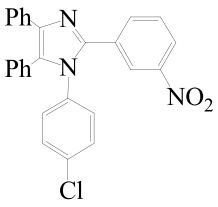
Compound 4

M. p: 190-191 °C; **^{1}H NMR** (500 MHz, $CDCl_3$ with few drops of DMSO): δ = 8.18 (s, 3H, Ar-H), 7.31-7.33 (m, 10H, Ar-H), 6.36 (t, *J* = 9.1 Hz, 4H, Ar-H); **^{13}C NMR** (300 MHz, $CDCl_3$ with few drops of DMSO): 154.7, 140.7, 133.9, 128.4, 128.3, 124.5 *ppm*; HRMS: calcd. for $C_{27}H_{17}ClN_4O_4$, 496.9073; found: 496.9070.



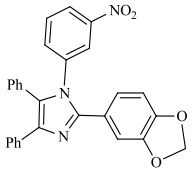
Compound 5

M. p: 292-293 °C; **^{1}H NMR** (500 MHz, $CDCl_3$ with few drops of DMSO): δ = 9.25 (s, 1H, -OH), 7.80 (d, *J* = 8.5Hz, 2H, Ar-H), 7.11-7.30 (m, 12H, Ar-H), 6.90 (d, *J* = 8.5 Hz, 2H, Ar-H), 6.72 (d, *J* = 8.2 Hz, 2H, Ar-H); **^{13}C NMR** (300 MHz, $CDCl_3$): 157.8, 147.2, 130.9, 130.3, 129.6, 129.1, 128.0, 128.9, 127.9, 127.1, 126.4, 121.0, 115.3 *ppm*; HRMS: calcd. for $C_{27}H_{19}ClN_2O$, 422.9165; found: 422.9167.



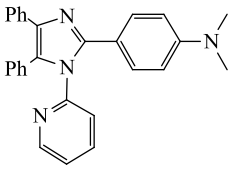
Compound 6

M. p: >300 °C; **^{1}H NMR** (500 MHz, $CDCl_3$ with few drops of DMSO): δ = 9.02-9.08 (m, 1H, Ar-H), 8.55 (d, *J* = 7.55 Hz, 1H, Ar-H), 8.16 (d, *J* = 8.00 Hz, 1H, Ar-H), 7.54-7.66 (m, 5H, Ar-H), 7.23-7.41(m, 6H, Ar-H); **^{13}C NMR** (300 MHz, $CDCl_3$): 205.0, 153.4, 143.0, 139.8, 137.3, 137.2, 136.1, 135.9, 134.5, 134.1, 133.4, 133.0, 132.3, 131.5, 127.1, 124.7 *ppm*; HRMS: calcd. for $C_{27}H_{18}ClN_3O_2$, 451.9034; found: 451.9033.



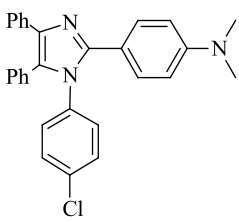
Compound 7

M. p: 247-248 °C; **^{1}H NMR** (500 MHz, $CDCl_3$ with few drops of DMSO): δ = 11.81 (s, 1H, Ar-H), 7.15-7.78 (m, 12H, Ar-H), 5.87 (t, *J* = 6.30 Hz, 2H, -CH_2-); **^{13}C NMR** (300 MHz, $CDCl_3$): 200.9, 147.8, 147.6, 147.5, 146.2, 137.6, 131.6, 129.7, 128.4, 128.0, 127.6, 127.3, 126.4, 125.0, 124.9, 119.6, 108.3, 106.3, 101.0 *ppm*; HRMS: calcd. for $C_{28}H_{19}N_3O_4$, 461.4734; found: 461.4733.



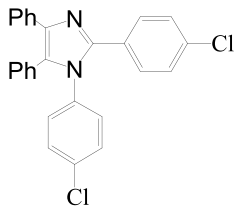
Compound 8

M. p: 254-256 °C. **^{1}H NMR** ($CDCl_3$, 500MHz): δ = 7.81 (d, *J* = 8.6 Hz, 2H, Ar-H), 7.57 (d, *J* = 6.7 Hz, 4H, Ar-H), 7.37 (t, *J* = 7.2 Hz,4H, Ar-H), 7.28-7.27 (m, 3H, Ar-H), 6.77 (d, *J* = 8.6 Hz, 2H, Ar-H), 3.04 (s, 6H, 2×CH_3); **^{13}C NMR** (300 MHz, $CDCl_3$): 150.8, 146.9, 128.5, 127.8, 127.2, 126.4, 117.8, 112.1, 40.3 *ppm*; HRMS: calcd. for $C_{28}H_{24}N_4$, 416.5254; found: 416.5251.



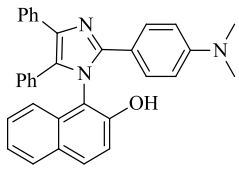
Compound 9

M. p. 168-170 °C; **^{1}H NMR** ($CDCl_3$, 500 MHz): δ = 7.61 (d, *J* = 8.2 Hz, 2H, Ar-H), 7.28-7.20 (m, 10H, Ar-H), 7.12 (m, 2H, Ar-H), 7.00 (dd, *J* = 2, 6.6 Hz, 2H, Ar-H), 6.61 (d, *J* = 8.9 *Hz*, 2H, Ar-H), 2.97 (s, 6H, 2×CH_3); **^{13}C NMR** (300 MHz, $CDCl_3$): 150.2, 147.7, 138.1, 136.1, 134.5, 133.8, 129.8, 128.4, 127.9, 126.5, 117.9, 111.6, 40.2 *ppm*; HRMS: calcd. for $C_{29}H_{24}N_3Cl$, 449.9763, found: 449.9761.



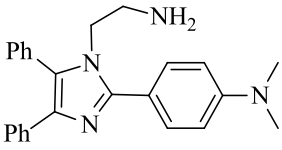
Compound 10

M. p: 200-201 °C;**^{1}H NMR** (DMSO, 500MHz): δ = 7.61 (d,*J* = 1.4 Hz,2H, Ar-H), 7.39 (q, *J* = 6.75 Hz,2H, Ar-H), 7.14-7.34 (m, 10H, Ar-H), 7.06 (dd, *J* = 2.15, 4.50 Hz, 2H, Ar-H), 6.98 (d, *J* = 1.8 Hz,2H, Ar-H); **^{13}C NMR** (300 MHz, $CDCl_3$): 145.8, 138.7, 135.4, 134.4, 134.0, 131.1, 130.9, 130.1, 129.5, 128.7, 128.5, 128.2, 127.3, 126.9 *ppm*; HRMS: calcd. for $C_{27}H_{18}Cl_2N_2$, 441.3523, found: 441.3525.



Compound 11

M. p: 247-249 °C; **^{1}H NMR** (DMSO, 500MHz): δ = 9.68 (s, 1H, Ar-H), 8.46 (s, 1H, Ar-H), 7.78 (d, *J* = 8.9 Hz, 2H, Ar-H), 7.69 (m, 2H, Ar-H), 7.57 (dd, *J* = 1.9, 7.05, Hz, 1H, Ar-H),7.48 (m, 2H, Ar-H), 7.29- 7.09 (m, 7H, Ar-H), 7.02 (dd, *J* = 2.2, 8.9, Hz, 1H, Ar-H), 6.80 (d, *J* = 8.8 Hz, 1H, Ar-H), 6.46 (d, *J* =9.1 Hz, 2H, Ar-H), 2.81 (s, 6H, 2×CH_3); **^{13}C NMR**(300 MHz, DMSO): 150.4, 147.6, 146.9, 131, 130.4, 129, 128.6, 128.5, 127.5, 126.7, 122.2, 118.3, 112.4, 112, 111.7, 111.5, 103.8, 40.1 *ppm*; HRMS: calcd. for $C_{33}H_{27}N_3O$, 481.5946; found: 481.5943.



Compound 12

Mp: 233-235 °C;**^{1}H NMR** ($CDCl_3$, 500MHz): δ = 8.16 (d, *J* = 8.9 Hz, 1H, Ar-H), 7.59(d, *J* = 8.9 Hz, 2H, Ar-H), 7.38-7.51(m, 4H, Ar-H), 7.28-7.37(m, 3H, Ar-H), 7.18-7.28(m, 4H, Ar-H), 6.69 (d, *J* = 8.7 Hz, 2H, Ar-H), 4.47 (t, *J* = 8.5 Hz, 2H, -CH_2-), 3.19 (t, *J* = 8.6 Hz, 2H, -CH_2-) 3.02 (s, 6H, 2×CH_3);**^{13}C NMR**(300 MHz, $CDCl_3$):162.4, 160.3, 152, 137.8, 128.1, 127.9, 127.8, 124.5, 111.6, 111.0, 62.1, 45.8, 40.2, 39.0 *ppm*; HRMS: calcd. for $C_{25}H_{26}N_4$, 382.5066, found 382.5064.

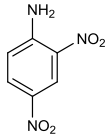
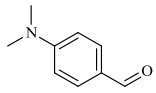
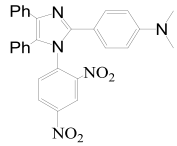
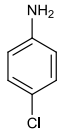
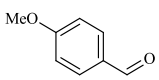
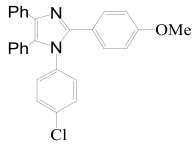
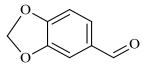
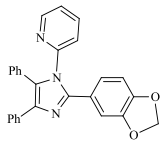
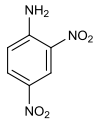
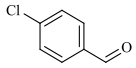
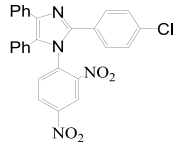
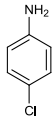
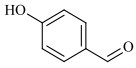
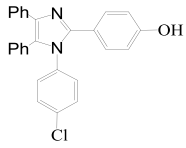
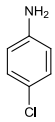
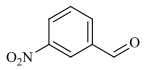
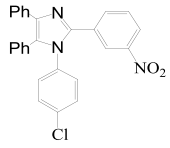
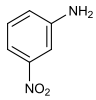
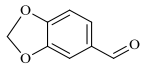
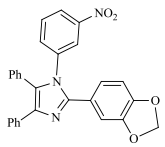
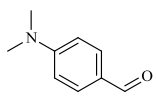
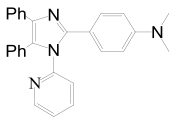
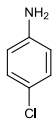
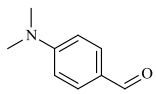
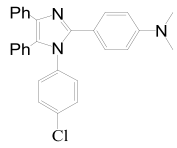
3. Results and Discussion

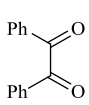
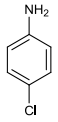
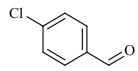
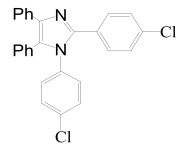
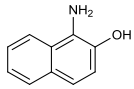
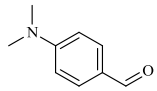
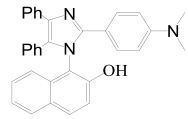
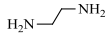
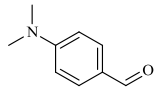
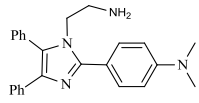
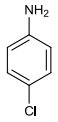
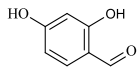
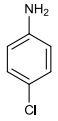
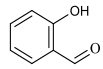
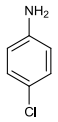
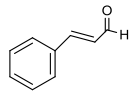
At the outset, we attempted to synthesize a tetra-substituted imidazoles from benzil, benzaldehyde, amine and ammonium acetate catalysed by *o*-aminophenol, a small entity in the realm of organic compounds through multicomponent strategy in ethanol. Reaction

mixture with *o*-aminophenol (5~20 mol%) was gently heated over an oil bath at different temperatures and the progress of the reaction were monitored by TLC. But, TLC monitoring showed a little conversion of the starting materials to products. Changing of different solvents, uses of co-solvent and the extension of reaction hour did not improve the yield. So our apprehension, *o*-aminophenol is not working as a suitable catalyst to this multicomponent reaction. By the same way as a test case, *p*-amino phenol, *p*-amino benzoic acid and *o*-amino benzoic acid were used to the same reaction. Unfortunately, all these molecules are proved to be futile for this reaction. But for *p*-amino benzoic acid, a little conversion was observed in TLC. For *o*-amino benzoic acid, presence of intramolecular hydrogen bonding may make it inactive towards this reaction. After a series of failures, we wanted to take advantages from α-amino acids and thereby we used aspartic acid and glutathione to this reaction. But neither of these molecules was successful to deliver a good conversion of the starting materials. The possible reason of failure may be the Zwitter ionic state of amino acid which inhibits it to donate proton to benzil **1**. Again, literature survey envisaged us to use vitamin-C a mild proton donor, to this four component reaction. But vitamin-C, failed to catalyze this multicomponent reaction albeit the same reaction conditions were imposed. Later, we wished to use amino alcohol (Entry-VII; Table 1) because of its versatile uses in different organic transformations as organocatalyst. But this catalyst also failed to convert fully the starting material into product. To complete the reaction, catalyst loadings, reaction hour and solvents were changed very cautiously. But in each trial, TLC report indicated only 50% conversion of the starting materials. Possibly in the presence of this catalyst the reaction suffers from a thermodynamic equilibrium. Meanwhile, we have synthesized the organocatalyst (*S*)-3-methyl-1, 1-diphenylbutane-1, 2-diamine **6** [50] by a known method.

With the exploitation of catalyst **6**, we have executed the same 4-component reaction. Fortunately, by the catalyst **6**, at 80°C in ethanol, the starting materials underwent a clean conversion to the desired product. After removal of ethanol, a white color solid was isolated from which the pure product **5** was obtained by recrystallization. Structure of the product **5** was proved by ^{1}H and ^{13}C NMR spectroscopic analyses. With the optimized conditions, we have studied the scope and limitations for benzil or benzoin, ammonium acetate or ammonium formate, aryl amines and different benzaldehydes to generalize the method. It was noticed that ammonium acetate has afforded better yields than ammonium formate in delivering NH_3. A concise Table is given below just to give an impression about the reaction products, yields etc. (Table 1).

Table 1. Synthesis of different tetra- substituted imidazoles.

Entry	Benzil	Amine	Aldehyde	Product	Time (h)	Yield (%)[a]	M.P (°C)
1					1: 55	90	218-219
2					2:00	93	180-181
3					1:50	95	252-253
4					2:10	92	190-191
5					2:15	92	292-293
6					2:05	89	>300
7					2:05	90	247-248
8					2:00	88	254-255
9					2:10	90	168-169

10					2:05	92	200-201
11					2:08	90	247-248
12					1: 55	89	233-234
13					-	~24	no	-
14					-	~24	no	-
15					-	~24	poor	-

[b]*Isolated yield*

It was noticed that electronic and structural motifs of the aldehydes are playing a crucial role and in fact, no condensation or a few percentages of condensation were occurred for the substrates **13, 14** and **15**. The failure may come from the negative charge accumulation onto the aldehyde carbon which deterred nucleophilic attacks; or strong hydrogen bond formation between the substrates and the catalyst may make the nucleophilic attack almost impossible. Reaction mechanism may be in the same pattern as mentioned in the literature [11], but in our case, reaction mechanism may be in the following manner:

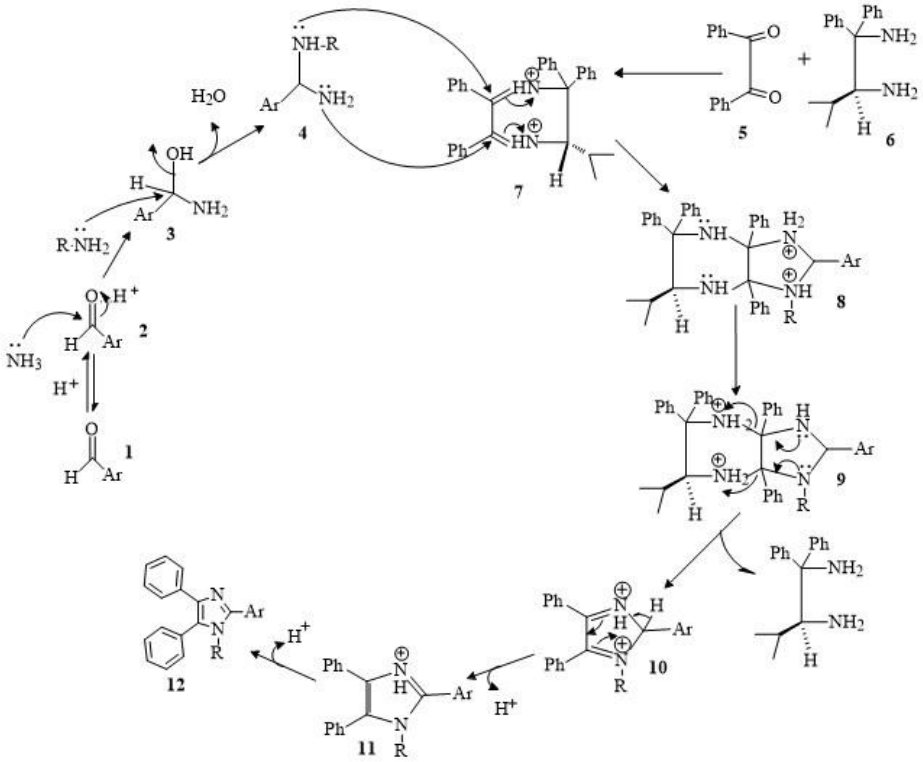


Fig. 1. Plausible reaction mechanism for imidazoles formation.

By this optimized method twelve different heterocycles were prepared with a great ease, and the yields of the isolated imidazoles are above 80% (Table 1).

4. Conclusion

In conclusion, by this newly developed one-pot, four component strategy, we have synthesized some highly substituted imidazoles with high yields without any cumbersome purification techniques. The novelty of this method lies in choosing (*S*)-3-methyl-1,1-diphenylbutane-1, 2-diamineas an organocatalyst for the first time to such type of multicomponent reaction. Moreover, metal-free, shorter reaction time, lower reaction temperature, operational simplicity, easy workup procedures, high atom economy, facile substituent variation are all notable aspects of this methodology. Although it is a tiny contribution to the vast sea of organic compounds, but we believe this method would be a contending one over other existing methodologies.

Acknowledgment

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