New conditions for the effective synthesis of tri and tetrasubstituted imidazoles catalysed by recyclable indium (III) triflate and magnesium sulfate heptahydrate

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A one-pot three or four component reaction of wide range of aromatic aldehydes, benzil, aliphatic and aromatic primary amines and ammonium acetate is reported for the synthesis of tri/tetra-substituted imidazoles under solvent-free conditions. Indium (III) trifluoromethanesulfonate and magnesium sulfate heptahydrate as two highly efficient and recyclable catalysts perform a key factor to synthesis of imidazole derivatives with high yields.

Keywords: catalyst, solvent-free, recyclable, cost effective, imidazole synthesis

Imidazole derivatives have received significant attentions in recent years due to their pharmaceutical properties.[1,2] Therefore, highly efficient synthesis of these type of compounds in green procedures have attractive interest for chemists. In this research, an effective and eco-friendly procedure to synthesise polysubstituted imidazoles was obtained by using indium (III) trifluoromethanesulfonate and magnesium sulfate heptahydrate as a highly efficient and recyclable catalysts under solvent free conditions. The procedure not only originates the products in excellent yields with shorter reaction times but also avoids some problems such as catalyst cost, pollution, handling, and safety.

Results and discussion

In connection with our studies on new catalysed organic reactions,[3–5] indium (III) trifluoromethanesulfonate and magnesium sulfate heptahydrate were found to be powerful, safe and recyclable catalysts for the one-pot condensation reaction to tri/tetrasubstituted imidazole derivatives. Especially, $MgSO_4.7H_2O$ is quite environmentally safe and has medicinal properties when used both externally and internally.

Firstly, different methods for the synthesis of imidazoles (compound **5a** for example) were compared (Table 1). This method not only gives the products in excellent yields with short reaction times from a simple and safe procedure but also avoids the problems associated with solvent use, pollution, catalyst cost and handling.

To obtain optimum conditions for the synthesis of the desired products, compound **5a** was chosen as a model reaction. In the presence of catalytic amounts of $In(OTf)_3$, the mixture of benzil (1 mmol), benzaldehyde (1 mmol), ammonium acetate (1 mmol), benzylamine (1 mmol) were reacted in different solvents such as water, ethanol, methanol, chloroform, acetonitrile and solvent-free conditions. From these experiments, it was clearly demonstrated that solvent-free is the best condition to accomplish this reaction (Table 2). The same results were obtained when $MgSO_4.7H_2O$ was used as catalyst.

In the absence of catalyst (up to 24 h stirring) with the same conditions gave the desired product in trace yield (12%). In catalyst-free conditions, and increasing the reaction

Table 1 Comparison of the results for the synthesis of 1-benzyl-2,4,5-triphenylimidazole (compound **5a**) with other catalysts

Catalyst	Mol/%	Solvent/temp. /°C	Time/yield min/%	Ref.
$In(OTf)_3$	5	Solvent free/120	25/96	Present work
$MgSO_4.7H_2O$	15	Solvent free/120	50/94	Present work
$NH_4H_2PO_4/Al_2O_3$	0.15 g	Solvent free/130	120/91	6
$K_5CoW_{12}O_{40}.3H_2O$[a]	10	Solvent free/140	120/90	7
$[(CH_2)_4SO_3HMIM][HSO_4]$[b]	15	Solvent free/140	120/90	8
$InCl_3.3H_2O$	10	MeOH/r.t.	444/79	9
$BF_3.SiO_2$[c]	21	Solvent free/140	120/80	10
L-Proline	15	MeOH/60	510/86	11
$AlPO_4$	1	Solvent free/140	120/85	12
$ZrCl_4$	20	CH_3CN/r.t.	60/86	13
SiO_2[d]	2 g	Solvent free, MW	8/87	14
SiO_2[d]	0.1	CH_2Cl_2,Solar heat	120/80	15
TFA[e]	20	Solvent free, MW	4/92	16
I_2	10	Solvent free/100	60/85	17

[a]Potassium dodecatugstocobaltate trihydrate.
[b]3-Methyl-1-(4-sulfonic acid)-butylimidazolium hydrogen sulfate.
[c]Silica-supported boron trifluoride.
[d]Silica gel as acidic support.
[e]Trifluoroacetic acid.

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temperature (up to 200 °C), there was no improvement in yield. Therefore, we found that the presence of the catalytic amount of indium (III) trifluoromethanesulfonate (or magnesium sulfate heptahydrate) and solvent-free condition are the best conditions for this synthesis.

In another investigation, we evaluated the quantity of catalyst required for the synthesis of tetrasubstituted imidazole derivatives (compound **5a**). From this experiment, maximum yield of product was obtained when 5 mol% of $In(OTf)_3$ or 15 mol% of $MgSO_4.7H_2O$ were used. While for trisubstituted imidazoles (**6a** was chosen as model reaction), the optimum amount of catalyst is 5 mol% of $In(OTf)_3$ or 10 mol% of $MgSO_4.7H_2O$ (Table 3).

Progress of the reaction was examined at various temperatures in the presence of optimised amounts of catalysts to give the best yield of product with short reaction times (Table 4). From Table 4, 120 °C was chosen as optimal temperature in the presence of $In(OTf)_3$ or $MgSO_4.7H_2O$ for tri and tetrasubstituted imidazoles synthesis.

The reaction under optimal conditions with aromatic aldehydes **1**, ammonium acetate **(2)**, benzil **(3)**, aromatic and aliphatic amine **4**, and catalytic amount of $In(OTf)_3$ or $MgSO_4.7H_2O$ and gave the tetrasubstituted imidazoles **5**, whereas in the absence of aromatic and aliphatic amine **4**, trisubstituted imidazoles **6** were obtained (Scheme 1.).

All products obtained (Table 5) were characterised by IR, ^1H, ^{13}C NMR, MS analyses for novel products (Figs S1–S48, ESI), and known products were compared with their published physical properties.

After determination of the suitable reaction conditions, the scope of this MCR was examined using various starting materials under standard conditions. It was found that both electron rich and electron deficient aldehydes reacted well (Table 5).

Table 2 The effect of solvents in synthesis of 1-benzyl-2,4,5-triphenylimidazole (compound **5a**)

Entry	Solvent	Time/min	Yield/%
1	Water	90	46
2	Ethanol	35	75
3	Methanol	40	78
4	Chloroform	120	49
5	Acetonitrile	80	73
6	Solvent-free	25	96

Table 3 Optimisation of molar ratio of the catalysts in synthesis of tri and tetrasubstituted imidazoles

$In(OTf)_3$ /mol%	Compound **6a** Time/yield min/%	Compound **5a** Time/yield min/%	$MgSO_4.7H_2O$ /mol%	Compound **6a** Time/yield min/%	Compound **5a** Time/yield min/%
1	60/55	60/48	2	60/42	60/45
2	60/80	60/75	5	60/75	60/65
5	15/95	25/96	10	60/90	60/80
10	30/90	25/94	15	60/90	50/94
15	30/90	30/92	20	60/85	50/92
20	30/88	30/90	25	70/82	60/88

Table 4 Optimisation of temperature for model reaction

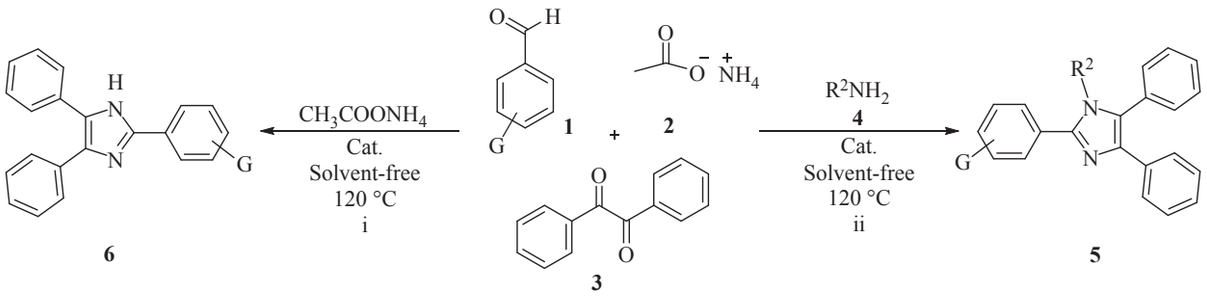
Temp. /°C[a]	Compound **6a**[a] Time/yield min/%	Compound **5a**[a] Time/yield min/%	Temp./°C[b]	Compound **6a**[b] Time/yield min/%	Compound **5a**[b] Time/yield min/%
80	20/68	30/65	80	60/70	60/62
100	25/90	30/90	100	60/80	60/70
120	15/95	25/96	120	60/90	50/94
140	25/85	30/90	140	60/88	60/92
160	25/80	30/78	160	60/75	60/86

[a]$In(OTf)_3$ was used as catalyst.
[b]$MgSO_4.7H_2O$ was used as catalyst.

Table 5 Catalytic synthesis of tri/tetra-substituted imidazoles under solvent-free conditions

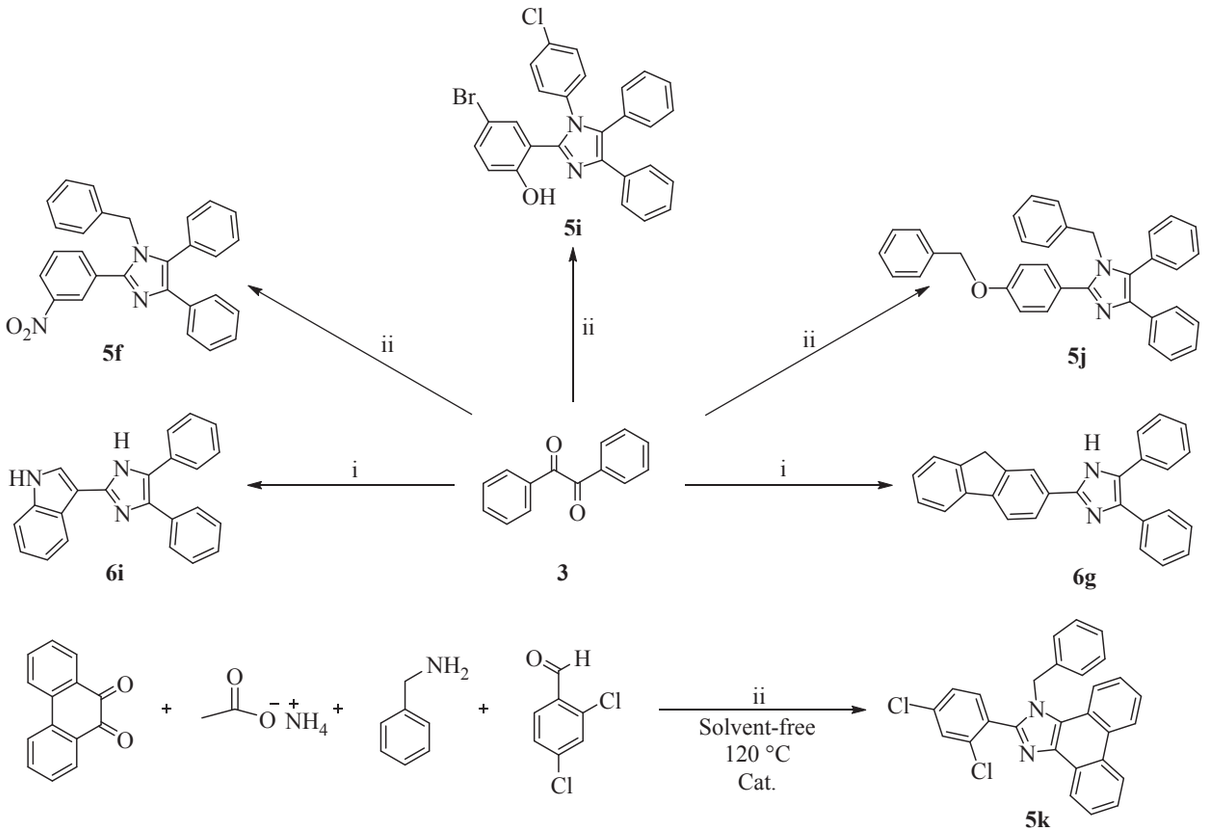
Compound	Aldehyde	Amine	$MgSO_4.7H_2O$ Time/yield[a] min/%	$In(OTf)_3$ Time/yield[a] min/%	M.p./°C[lit.]
5a	C_6H_5CHO	$C_6H_5CH_2NH_2$	50/94	25/96	162–164 (163–165)[18]
5b	4-BrC_6H_4CHO	$C_6H_5CH_2NH_2$	30/92	20/90	169–170 (170–172)[12]
5c	4-MeC_6H_4CHO	$C_6H_5CH_2NH_2$	80/94	50/92	156–158 (156–157)[19]
5d	4-ClC_6H_4CHO	$C_6H_5CH_2NH_2$	60/90	25/92	159–160 (157–158)[19]
5e	2-ClC_6H_4CHO	$C_6H_5CH_2NH_2$	30/88	20/92	139–140 (140–142)[20]
5f	3-$NO_2C_6H_4CHO$	$C_6H_5CH_2NH_2$	45/85	30/90	150–152[b]
5g	4-MeC_6H_4CHO	$C_6H_{11}NH_2$	45/75	25/88	160–161 (162–164)[21]
5h	4-$OMeC_6H_4CHO$	$C_6H_5CH_2NH_2$	60/86	40/90	163–165 (164–165)[22]
5i	2-OH-5-BrC_6H_3CHO	4-$ClC_6H_4NH_2$	33/92	25/98	156–158[b]
5j	4-Benzyloxy C_6H_4CHO	$C_6H_5CH_2NH_2$	80/82	60/96	138–139[b]
5k	2,4-diClC_6H_3CHO	$C_6H_5CH_2NH_2$	85/80	60/90	216–219[b]
5l	4-MeC_6H_4CHO	$C_6H_5NH_2$	60/88	30/90	182–184 (185–188)[11]
6a	C_6H_5CHO	–	60/90	15/95	275–277 (276–278)[18]
6b	3-BrC_6H_4CHO	–	35/85	10/88	120–122 (118–122)[23]
6c	2-OHC_6H_4CHO	–	60/86	15/90	209–211 (209–210)[24]
6d	2-$OMeC_6H_4CHO$	–	60/88	25/92	207–209 (210–211)[11]
6e	4-$OMeC_6H_4CHO$	–	50/85	15/88	227–230 (228–230)[18]
6f	4-Benzyloxy C_6H_4CHO	–	45/75	20/80	235–236[c] [25]
6g	Fluorene-2-carboxaldehyde	–	55/90	25/97	283–286[b]
6h	Indole-3-carboxaldehyde	–	60/80	25/88	311–313[b]
6i	4-ClC_6H_4CHO	–	45/90	10/94	257–259 (257–260)[26]
6j	3-$NO_2C_6H_4CHO$	–	50/90	10/92	308–309 (>295)[27]

[a]Refers to isolated yields.
[b]Novel compound.
[c]Melting point of this compound was not reported by author.



Cat.: $In(OTf)_3$, i: 5 mol%; ii: 5 mol%, or $MgSO_4.7H_2O$, i: 10 mol%; ii: 15 mol%

Scheme 1 Catalytic-assisted synthesis of tri/tetra-substituted imidazole derivatives under solvent-free conditions.



i: Solvent-free, 120 °C, Respective aldehyde, CH_3COONH_4, 5 mol% $In(OTf)_3$ or 10 mol% $MgSO_4.7H_2O$
ii: Solvent-free, 120 °C, Respective aldehyde, Respective primary amine, CH_3COONH_4, 5 mol% $In(OTf)_3$ or 15 mol% $MgSO_4.7H_2O$

Scheme 2 Catalytic synthesis of novel tri and tetra-substituted imidazole derivatives.

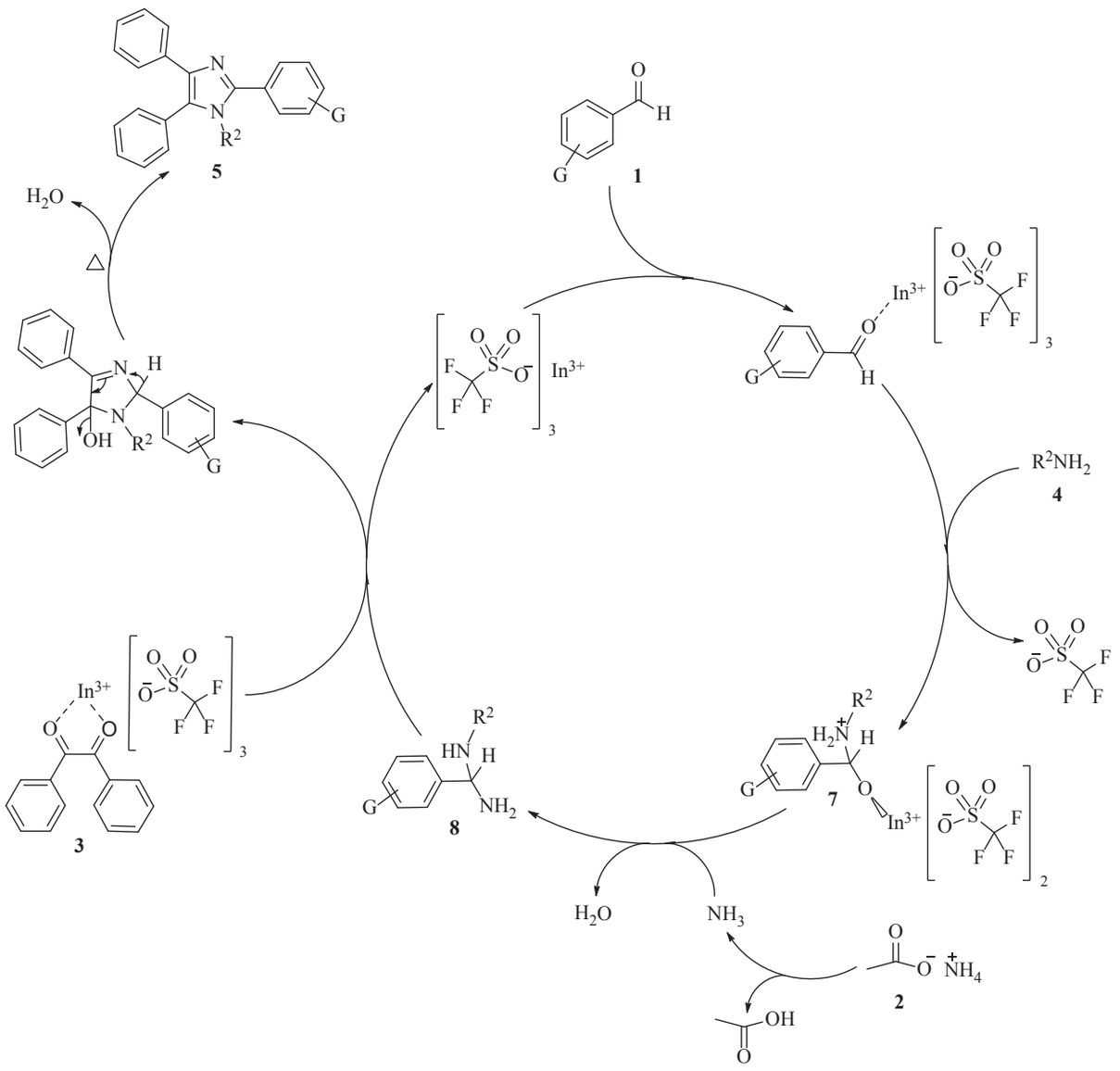
Note that in this work, the six new compounds **5(f**, **i**, **j** and **k)** and **6(g** and **h)** (Scheme 2) were synthesised and characterised by FT-IR, ^1H, ^{13}C NMR, MS analyses, and elemental analysis.

Recyclability of catalysts

After completion of the reaction, the catalyst, was separated from the reaction mixture by filtering, washed with diethyl ether, dried at 120 °C for 1 h, and reused in another reaction. We found that indium (III) trifluoromethanesulfonate and magnesium sulfate heptahydrate can be reused several times (up to five times) without significant loss of activity. The results of these observations are shown in Table 6 for a model reaction.

Scheme 3 shows a probable mechanism for the synthesis of imidazole derivatives (tetrasubstituted imidazole for example) may be postulated as shown below which recyclability of catalyst is observed.

As can be seen from Scheme 3, the catalyst ($In(OTf)_3$ for example) activates the carbonyl group of aldehyde **1** to the nucleophilic attack of amine **4** which increases the interaction between aldehyde and amine with a decrease in the energy of the transition state. From this interaction, intermediate **7** was formed and was stabilised by indium (III) trifluoromethanesulfonate. Following this, the nucleophilic attack of ammonia which *in situ* was generated



Scheme 3 The suggested mechanism for the synthesis of tetrasubstituted imidazoles.

from ammonium acetate **(2)** to the intermediate **7**, gave the intermediate **8**. Condensation of intermediate **8** with activated benzil (**3**) by indium (III) trifluoromethanesulfonate and following dehydration, gave the corresponding imidazoles **5**.

Experimental

^{1}H, ^{13}C NMR spectra were recorded on a FT-NMR Bruker Avance ultra shield spectrometer (frequency line, 400.13 MHz for ^{1}H NMR and 100.62 MHz for ^{13}C NMR.; solvents, $CDCl_3$ and DMSO-d_6). FT-IR spectra were recorded in the matrix of KBr with JASCO FT-IR-680 plus spectrometer. Elemental analyses (C, H, N) were performed with a Heraeus CHN-O-Rapid analyser. Mass spectra of the products were obtained with a HP (Agilent technologies) 5937 Mass Selective Detector. Melting points were measured on an electrothermal KSB1N apparatus. TLC was performed on TLC-Grade silica gel-G/UV 254 nm plates (*n*-hexane, ethyl acetate). Chemicals were purchased from Aldrich, Fluka and Merck chemical companies. Known compounds were characterised by comparison with authentic samples and combustion analyses.

Tri/tetra-substituted imidazoles synthesis using In(OTf)$_3$ and MgSO$_4$.7H$_2$O; general procedure

For tetrasubstituted imidazoles synthesis, In(OTf)$_3$ (5 mol%)/ $MgSO_4.7H_2O$ (15 mol%) was added to stirred mixture of aldehyde (1 mmol), benzil (1 mmol), primary amine (1 mmol), ammonium acetate (1 mmol) and heated at 120 °C for the time indicated in Table 5. The progress of reaction was monitored by TLC on silica gel (SILG/UV 254) plates (*n*-hexane, ethyl acetate 10 : 3). After completion of reaction, the mixture was cooled to room temperature and washed with water (50 mL) then was filtered to remove the catalyst and the filtrate was concentrated in vacuum to afford the crude product which was recrystallised from boiling EtOAc to afford the crystalline pure product. For trisubstituted imidazoles synthesis primary amine was replaced with ammonium acetate and progress of reaction was monitored by TLC on silica gel (SILG/UV 254) plates (*n*-hexane, ethyl acetate 5 : 1).

5f: IR, ν/cm^{-1}: 3061, 3026, 2308, 1601, 1521, 1497, 1350, 810, 730, 696. ^{1}H NMR (400.13 MHz, DMSO-d_6, δ): 5.19 (s, 2H), 6.89 (d, J = 6.1 Hz, 2H), 7.21–7.63 (m, 14H), 8.04 (d, J = 7.8 Hz, 1H), 8.23 (d, J = 7.8 Hz, 1H),

8.57 (s, 1H). ^{13}C NMR (100.62 MHz, DMSO-d_6, δ): 47.4, 122.3, 122.6, 124.7, 125.7, 125.7, 126.7, 127.1, 127.8, 128.0, 128.6, 129.4, 129.93, 130.2, 131.6, 133.0, 133.5, 135.8, 144.2, 147.2. Anal. calcd for $C_{28}H_{21}N_3O_2$: C, 77.94; H, 4.91; N, 9.74; found: C, 77.87; H, 4.83; N, 9.62%. MS, *m/z*: 431 M^+, 386, 340, 295, 190, 165, 134, 91, 57.

5i: IR, ν/cm^{-1}: 3458, 3063, 1659, 1593, 1578, 1211, 1174, 1096. ^{1}H NMR (400.13 MHz, DMSO-d_6, δ): 6.92–8.53 (m, 17H), 13.06 (s, 1H). ^{13}C NMR (100.62 MHz, DMSO-d_6, δ): 115.2, 123.9, 125.5, 127.6, 134.3, 137.3, 139.3, 140.5, 151.3, 164.7, 166.9. Anal. calcd for $C_{27}H_{18}BrClN_2O$: C, 64.62; H, 3.62; N, 5.58; found: C, 64.51; H, 3.53; N, 5.49%. MS, *m/z*: 502 M^+, 267, 214, 193, 165, 75, 57.

5j: IR, ν/cm^{-1}: 2857, 1601, 1575, 1526, 1289, 1247, 1177. ^{1}H NMR (400.13 MHz, DMSO-d_6, δ): 2.18 (s, 2H), 5.06 (s, 2H), 6.83–7.82 (m, 24H). ^{13}C NMR (100.62 MHz, DMSO-d_6, δ): 48.3, 70.0, 115.0, 123.8, 126.0, 126.4, 126.8, 127.4, 127.5, 128.1, 128.2, 128.7, 128.9, 129.9, 130.5, 131.1, 131.2, 134.7, 136.8, 137.7, 137.9, 148.0, 159.3. Anal. calcd for $C_{35}H_{28}N_2O$: C, 85.34; H, 5.73; N, 5.69; found: C, 85.18; H, 5.61; N, 5.54%. MS, *m/z*: 492 M^+, 402, 311, 283, 165, 91, 65.

5k: IR, ν/cm^{-1}: 3069, 2924, 1557, 1523, 1459, 1092. ^{1}H NMR (400.13 MHz, DMSO-d_6, δ): 2.18 (s, 2H), 7.45–8.41 (m, 13H), 8.64 (d, J = 7.6 Hz, 1H), 8.77 (t, J = 8.4 Hz, 2H). ^{13}C NMR (100.62 MHz, DMSO-d_6, δ): 120.9, 121.1, 123.0, 123.5, 123.8, 125.00, 126.0, 126.8, 127.2, 127.4, 127.4, 127.6, 129.0, 129.6, 131.3, 132.3, 133.8, 135.2, 136.9, 145.2. Anal. calcd for $C_{28}H_{20}Cl_2N_2$: C, 73.85; H, 4.43; N, 6.15; found: C, 73.71; H, 4.36; N, 6.09%. MS, *m/z*: 452 M^+, 363, 295, 190, 164, 91.

6g: IR, ν/cm^{-1}: 3350, 3054, 2950, 1601, 1532, 1500. ^{1}H NMR (400.13 MHz, DMSO-d_6, δ): 4.01 (s, 2H), 7.33–8.00 (m, 15H), 8.13 (d, J = 7.6 Hz, 1H), 8.32 (s, 1H), 12.73 (s, 1H). ^{13}C NMR (100.62 MHz, DMSO-d_6, δ): 39.9, 120.4, 120.7, 122.4, 124.6, 125.6, 126.7, 127.4, 128.3, 128.9, 129.4, 141.2, 141.6, 143.9, 143.9, 146.4. Anal. calcd for $C_{28}H_{20}N_2$: C, 87.47; H, 5.24; N, 7.29; found: C, 87.41; H, 5.29; N, 7.22%. MS, *m/z*: 384 M^+, 340, 190, 165, 134, 91, 65.

6h: IR, ν/cm^{-1}: 3413, 3055, 1598, 1490, 1451. ^{1}H NMR (400.13 MHz, DMSO-d_6, δ): 7.13–7.59 (m, 13H), 8.01 (d, J = 2.4 Hz, 1H), 8.46 (d, J = 7.2 Hz, 1H), 11.40 (s, 1H), 12.4 (s, 1H). ^{13}C NMR (100.62 MHz, DMSO-d_6, δ): 106.9, 112.1, 120.2, 121.9, 122.4, 124.5, 125.5, 127.4, 128.0, 128.9, 136.7, 144.1. Anal. calcd for $C_{23}H_{17}N_3$: C, 82.36; H, 5.11; N, 12.53; found: C, 82.29; H, 5.21; N, 12.40%. MS, *m/z*: 335 M^+, 165, 142, 115, 77, 55.

Conclusions

New efficient method for the synthesis of tri/and tetrasubstituted imidazoles is reported *via* three- or four-component reaction in the presence of $In(OTf)_3$/and $MgSO_4.7H_2O$ catalysts in solid phase conditions. Numerous tri/and tetrasubstituted imidazole derivatives with good to excellent yields were obtained.

Electronic Supplementary Information

Spectral data have been deposited in the ESI available through: stl.publisher.ingentaconnect.com/content/stl/jcr/supp-data.

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