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Microwave-assisted heteropolyanion-based ionic liquids catalyzed transamidation of non-activated carboxamides with amines under solvent-free conditions

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ABSTRACT

An environmentally benign and highly efficient protocol for the transamidation of non-activated carboxamides with amines using heteropolyanion-based ionic liquids as catalysts under microwave-assisted and solvent-free conditions has been developed. As evaluated by the reactions of a structurally diverse set of amides and amines, the scope and utility of the transamidation proved to be quite general. Operational simplicity, solvent-free media, the potential reusability of catalysts and wide functional group tolerance are attractive features. This method provides a much improved protocol over the existing methods.

1. Introduction

Amide bond is a widely prevalent linkage in numerous bioactive products,[1] as well as biological and synthetic polymers (i.e., proteins and nylons).[2] Amides also act as fundamental building blocks for the synthesis of pharmaceuticals, agrochemicals, polymers and materials.[3] Traditional amide formation relies on activation of a carboxylic acid derivative (acyl halide, anhydride, ester or acid) using a coupling reagent and subsequent coupling of the activated species with an amine, but there are limitations, such as using stoichiometric amounts of coupling reagents, poor atom efficiency, large quantities of potentially hazardous waste and difficulties in purification.[4] These drawbacks have promoted the development of numerous alternative amide formation methods in recent years.[5] Amongst various catalytic methods, transamidation of amide with amine is potentially an attractive alternative tool for the direct amide bond formation. However, due to the relatively high inertness of the amide bond in contrast with other acyl donors, transamidation is hindered under thermal and noncatalytic conditions.[6] Although lipase-catalyzed transformation has been reported, this protocol requires high substrate specificity, highly evolved enzymes and long reaction time.[7] Moreover, great advances have recently been made to develop more convenient procedures to achieve transformation at relatively lower temperatures by using activating reagents or catalysts.[8,9] Several metal species have been reported to promote transamidation reactions in the last two decades, including $AlCl_3$,[8a] $Sc(OTf)_3$,[8b] $HfCl_4$,[8c] lanthanide catalysts,[8d] $Ti(NMe_2)_4$,[8e] $Cu(OAc)_2$,[8f] CeO_2,[8g] Cp_2ZrCl_2,[8h] sulfated tungstate,[8i] Nb_2O_5,[8j] Mn(II) complex,[8k] and Fe(III)-salt.[8l,m] In addition, very recently some metal-free catalysts, such as *N*,*N*-dialkylformamide dimethyl acetals,[9a] imidazole,[9b] hydroxylamine hydrochloride,[9c] boric acid,[9d] ammonium-salt,[9e] borate esters,[9f,9g] L-proline,[9h] hypervalent iodine[9i] and benzotriazole[9j] as well as microwave irradiation[9k,l] have come to the forefront offering enough impetus to improvement. Although the existing methods have their own advantages, the resulting transamidations suffer from certain demerits, such as stoichiometric amount of the catalysts, harsh reaction conditions, long reaction times, low selectivities and limited substrate scopes. Moreover, some of the catalysts have the difficulties in separation from the reaction mass and recycling. Therefore there is a clear need for more efficient and greener methods for transamidation with a broad substrate suitability.

Over the past decades, ionic liquids (ILs) have attracted much interest as efficient and eco-friendly reaction media and/or catalysts due to their excellent properties such as negligible vapour

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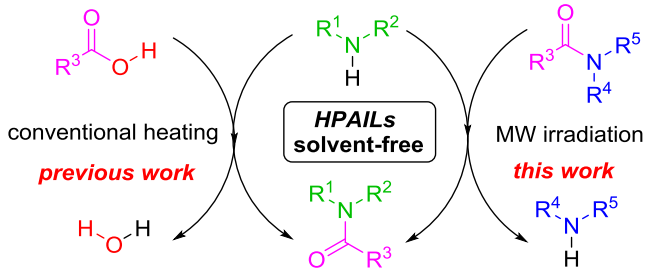
pressure, ease of recovery and reuse.[10] Amongst them a series of heteropolyanion-based ILs (HPAILs) have been recently prepared as hybrid materials by combining Keggin heteropolyanions with 'task-specific' ILs (TSILs) cations containing special functional groups.[11] HPAILs usually have high melting points, thermal stability and chemical stability owing to the large volume and high valence of heteropolyanions and hydrogen bonding net-works existing in the compounds, and that is consistent with the requirements of a solid acid catalyst. So far, HPAILs have turned out to be an eco-benign, high-efficient and recyclable catalyst for acid-catalyzed[12] or oxidative organic transformations[13] due to their advantages, such as operationally simplicity, no toxicity, easily isolation and reusability.

On the other hand, 'non-classical' methods have been developed in organic synthesis to improve yields, selectivity and experimental conditions during the last few years.[14] Amongst them microwave (MW)-assisted technology has blossomed into a useful tool for a variety of applications in organic synthesis due to their unique advantages, such as the significant rate enhancements, yield and selectivity improvements, very simplified ease of manipulation and work-up as well as less environmental polluting processes.[15] Although MW-assisted reactions in organic solvents have developed rapidly, the focus is now shifted to environmentally friendlier methods, which explore the using of MW irradiation in conjunction with solvent-free conditions or benign reaction media.[16]

Recently, our group has introduced HPAILs as eco-benign and highly efficient catalysts for condensation between carboxylic acids and amines to obtain amides.[17] We envisioned that HPAILs could potentially catalyze the transamidation reaction. To the best of our knowledge, ILs promoted transamidation has not been reported to date. In our continuing efforts in developing efficient and green protocols for catalytic methods for amide bonds formation, we wish to describe herein the first MW-assisted HPAILs catalyzed transamidation of non-activated carboxamides with amines under solvent-free conditions (Scheme 1).



Scheme 1. HPAILs catalyzed amidation reactions developed by our group.

2. Results and discussions

Our study is mainly focused on *N*-substituted imidazole, pyridine and triethylamine based HPAILs, which have already been used as catalysts for many different organic transformations.[12,13,17] Thus, six structurally related HPAILs (Fig. 1) were prepared according to published procedure.[12a] The obtained HPAILs were characterized and the results were compared with the literature data.

Initially, transamidation of acetamide with aniline was chosen as a model system to optimize the catalytic parameters (Table 1). Firstly, a control experiment was performed with conventional heating at 120 °C in the absence of any catalyst and additional solvent and, as expected, the complete lack of reactivity was observed even after a prolonged reaction time of 24 h (Table 1, entry 1). Whereas addition of 2 mol % amount of $[MIMPS]_3PW_{12}O_{40}$ to the reaction mixture resulted in the desired transformation product in 71% yield (Table 1, entry 2). The results revealed that the HPAILs

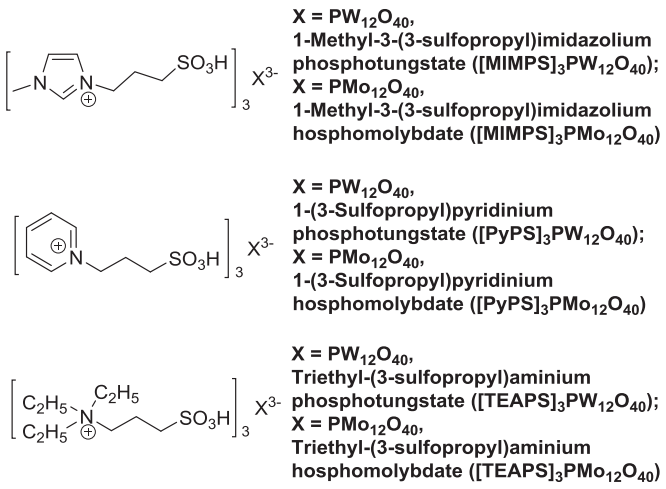
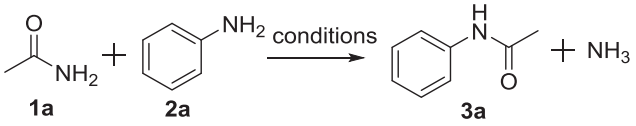


Fig. 1. *N*-Substituted imidazole, pyridine and triethylamine based HPAILs.

Table 1
Optimization of the reaction conditions for transamidation of acetamide with aniline[a]



Entry	Catalyst	Temp (°C)	Time (min)	Yield[b] (%)
1[c]	—	120	1440	0
2[c]	$[MIMPS]_3PW_{12}O_{40}$, 2 mol %	120	1440	71
3	$[MIMPS]_3PW_{12}O_{40}$, 2 mol %	120	50	80
4	$[MIMPS]_3PW_{12}O_{40}$, 2 mol %	100	50	65
5	$[MIMPS]_3PW_{12}O_{40}$, 2 mol %	140	50	78
6	$[MIMPS]_3PW_{12}O_{40}$, 3 mol %	120	50	80
7	$[MIMPS]_3PW_{12}O_{40}$, 1 mol %	120	50	72
8[d]	$[MIMPS]_3PW_{12}O_{40}$, 2 mol %	120	50	47
9[e]	$[MIMPS]_3PW_{12}O_{40}$, 2 mol %	120	50	45
10[f]	$[MIMPS]_3PW_{12}O_{40}$, 2 mol %	120	50	38
11[g]	$[MIMPS]_3PW_{12}O_{40}$, 2 mol %	120	50	25
12[h]	$[MIMPS]_3PW_{12}O_{40}$, 2 mol %	100	50	12
13[i]	$[MIMPS]_3PW_{12}O_{40}$, 2 mol %	120	50	<5
14	$[MIMPS]_3PMo_{12}O_{40}$, 2 mol %	120	50	75
15	$[PyPS]_3PW_{12}O_{40}$, 2 mol %	120	50	83
16	$[PyPS]_3PMo_{12}O_{40}$, 2 mol %	120	50	78
17	$[TEAPS]_3PW_{12}O_{40}$, 2 mol %	120	50	72
18	$[TEAPS]_3PMo_{12}O_{40}$, 2 mol %	120	50	65
19	$H_3PMo_{12}O_{40}$, 2 mol %	120	50	75

[a] Unless otherwise noted, all reactions were carried out with acetamide (2 mmol), aniline (2.6 mmol) and related catalyst under MW (700 W) and solvent-free conditions.
[b] Isolated yields.
[c] Conventional heating.
[d] Toluene (1.0 mL) was used as solvent.
[e] Xylene (1.0 mL) was used as solvent.
[f] Mesitylene (1.0 mL) was used as solvent.
[g] DMSO (1.0 mL) was used as solvent.
[h] H_2O (1.0 mL) was used as solvent.
[i] *n*-C_5H_9OH (1.0 mL) was used as solvent.

should be absolutely necessary for the catalyzed transamidation. To our delight, it was shown that the rate and yield of the reaction both increased dramatically when MW-assisted heating at 120 °C was utilized in the catalyzed transamidation (Table 1, entry 3). In addition, lower or higher temperature (100 °C or 140 °C) was harmful to the transamidation product (Table 1, entries 4 and 5). Further, no improvement was observed in the present of more amount of catalyst (Table 1, entry 6), but a decrease in the catalyst diminished the yield of the product considerably (Table 1, entry 7). In order to study the effect of the medium, reactions were carried

out in various solvents. It was observed that aromatic hydrocarbon solvents such as toluene, xylene and mesitylene (Table 1, entries 8–10) were found to be better reaction media than polar aprotic (DMSO, Table 1, entry 11) or protic solvents (H_2O, *n*-C_5H_9OH, Table 1, entries 12 and 13), but a decrease was found in the yield with any solvent. Afterwards catalytic activities of other related catalysts prepared before were screened under the same reaction condition (Table 1, entry 3). It was shown that the catalytic activities of $[MIMPS]_3PW_{12}O_{40}$ and $[TEAPS]_3PW_{12}O_{40}$ were slightly lower than that of $[PyPS]_3PW_{12}O_{40}$ (Table 1, entries 3, 15, 17). In the cases of catalysts combining with different heteropolyanions, the results demonstrated that $PW_{12}O_{40}$ was more active than $PMo_{12}O_{40}$ HPAILs (Table 1, entries 3, 14–18). Although pure HPA catalyst $H_3PW_{12}O_{40}$ gave a yield of 75%, its good solubility throughout organic solvents and water made its isolation from the reaction mixture difficult (Table 1, entry 19). Finally, optimum result was obtained when the reaction was performed using 2 mol % of $[PyPS]_3PW_{12}O_{40}$ under MW (700 W) and solvent-free condition at 120 °C for 50 min affording *N*-phenylacetamide in 83% yield (Table 1, entry 15).

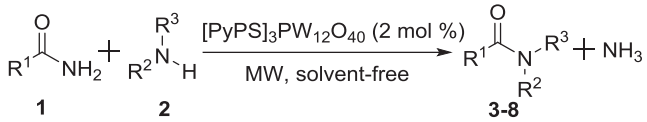
With the optimized conditions in hand, the substrate scope of this HPAILs catalyzed transamidation reaction was explored. As shown in Table 2, reactions of aliphatic or aromatic primary amides or formamide with a series of aromatic and aliphatic primary or secondary amines proceeded smoothly and afforded the desired products in reasonable to excellent yields. In general, aromatic amines were less reactive than aliphatic amines and afforded longer reaction times and lower yields (Table 2, **3a**–**3f** vs **3g**, **4a**,**4b** vs **4c**–**4e**, **5a**,**5b** vs **5c**, **6a**,**6b** vs **6c**,**6d**, **7a** vs **7b**, **8a**–**8g** vs **8h**–**8j**). Aromatic amines bearing substitution with an electron-donating group (methyl or methoxyl) exhibited higher reactivity as compared to an electron-withdrawing group (chloro or nitro, Table 2, **3b**,**3c** vs **3d**,**3e**, **8b**,**8c** vs **8d**,**8e**). Notably, heterocyclic aromatic amines (Table 2, **3f**, **8g**) reacted well and gave the corresponding product in good yields. Moreover, steric hindrance appeared to play an important role in the results of the reactions. The good conversions of benzylic amines (Table 2, **3g**, **4c**,**4d**, **6c**, **8h**,**8i**) or linear chain amines (Table 2, **4e**, **5c**, **6d**, **7b**) to their corresponding amides were observed, but in the case of hindered primary aliphatic amines (Table 2, **3h**, **4f**, **6e**,**6f**, **8j**,**8k**) and secondary amines (Table 2, **3i**,**3j**, **4g**,**4h**, **6g**, **8l**–**8n**), which are very poor nucleophiles, longer reaction times and lower yields were achieved.

With regards to the reactivities of the amides, aliphatic amides (acetamide, Table 2, **3a**–**3j**, and phenylacetamide, Table 2, **4a**–**4h**), even the hindered aliphatic amides (isobutyramide, Table 2, **5a**–**5c**), were more reactive than aromatic amides (benzamide, Table 2, **6a**–**6g**) and heteroaromatic amides (nicotinamide, Table 2, **7a**,**7b**) in this procedure. Moreover, it was worth mentioning that the formamide showed high reactivities in this catalyzed transamidation (Table 2, **8a**–**8n**), which may be due to the high electrophilicity of formamide.

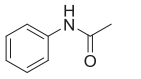
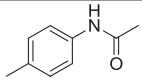
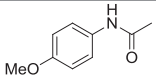
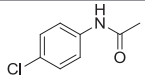
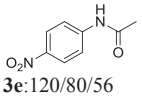
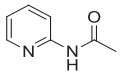
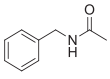
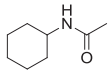
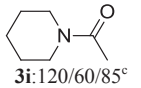
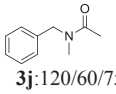
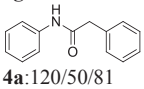
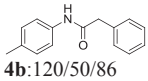
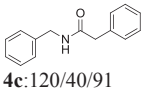
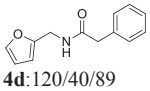
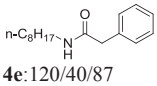
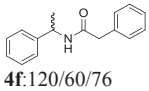
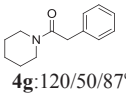
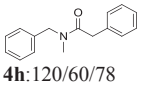
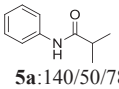
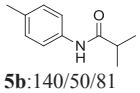
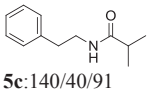
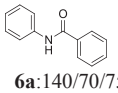
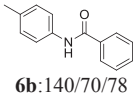
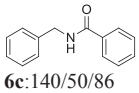
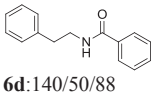
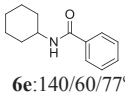
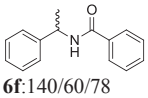
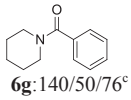
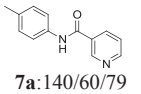
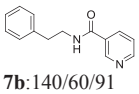
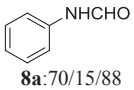
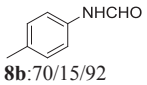
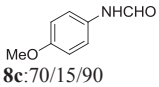
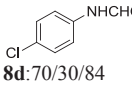
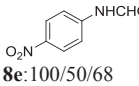
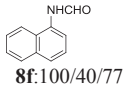
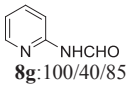
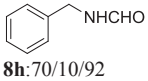
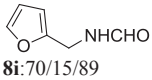
In addition, in contrast to most other transamidation methods, which are limited to primary amides, this HPAILs catalyzed transamidation is applicable to substrate primary, secondary, and even tertiary amides. As shown in Table 3, transamidation of the secondary (**9a**–**9c**) and tertiary (**9d**–**9e**) amides.

In order to further expand the substrate scope, the transamidation of phthalimide was examined with various primary amines (Table 4). We were delighted to see that all reactions reacted smoothly to provide the corresponding *N*-substituted phthalimides in good to excellent yields. In the cases of aliphatic amines the reaction could be carried out at 120 °C (Table 4, entries 3 and 4). For aromatic amine (Table 4, entries 1 and 2) or more hindered aliphatic amines (Table 4, entry 5) higher temperature and longer reaction times were required.

Table 2
HPAIL catalyzed transamidation of 1° amides with amines[a]



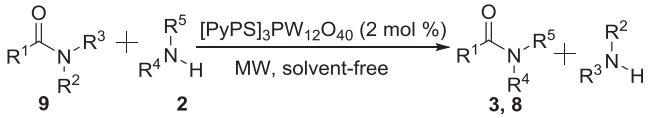
1 2 3-8

Product:temp (oC)/time (min)/yield (%)[b]			
 **3a**:120/50/83	 **3b**:120/50/85	 **3c**:120/50/90	 **3d**:120/60/81
 **3e**:120/80/56	 **3f**:120/60/78	 **3g**:120/40/92	 **3h**:120/60/78[c]
 **3i**:120/60/85[c]	 **3j**:120/60/75	 **4a**:120/50/81	 **4b**:120/50/86
 **4c**:120/40/91	 **4d**:120/40/89	 **4e**:120/40/87	 **4f**:120/60/76
 **4g**:120/50/87[c]	 **4h**:120/60/78	 **5a**:140/50/78	 **5b**:140/50/81
 **5c**:140/40/91	 **6a**:140/70/75	 **6b**:140/70/78	 **6c**:140/50/86
 **6d**:140/50/88	 **6e**:140/60/77[c]	 **6f**:140/60/78	 **6g**:140/50/76[c]
 **7a**:140/60/79	 **7b**:140/60/91	 **8a**:70/15/88	 **8b**:70/15/92
 **8c**:70/15/90	 **8d**:70/30/84	 **8e**:100/50/68	 **8f**:100/40/77
 **8g**:100/40/85	 **8h**:70/10/92	 **8i**:70/15/89	**8j**:100/90/80
8k:100/45/82[c]	**8l**:100/90/75	**8m**:100/45/72	**8n**:100/45/78[c]

[a] Reaction condition : amide (2 mmol), amine (2.6 mmol) and $[PyPS]_3PW_{12}O_{40}$ (2 mol %) under MW (700 W) and solvent-free condition.
[b] Isolated yields.
[c] Sealed tube.

A further advantage of this HPAILs catalytic system is its reusability. Thus, the recycling of the catalyst was examined based on the optimal conditions for the transamidation of acetamide with benzylamine (Table 2, **3g**). The outstanding feature of the catalyst is its excellent solubility in water or strong polar solvents but nonmiscibility with apolar esters. After completion of the reaction (monitored by TLC), EtOAc was added to the reaction mixture. After vigorous stirring the catalyst can be easily retrieved from the reaction mixture by simple centrifugation or filtration and drying at room temperature under reduced pressure. After removal of the solvent, the pure product was obtained by recrystallization or

Table 3
HPAIL catalyzed transamidation of 2° and 3° amides with amines[a]

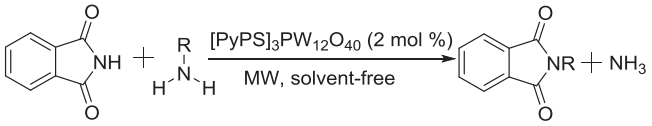


9 2 3, 8

Entry	Amide	Temp (°C)	Time (min)	Yield[b] (%)
1	HCONHMe, **9a**	120	80	**8a**, 78
2	HCONHMe, **9a**	120	80	**8c**, 80
3	HCONHMe, **9a**	120	70	**8h**, 86
4	$CH_3CONHMe$, **9b**	140	80	**3g**, 77
5	$CH_3CONHPh$, **9c**	140	80	**3g**, 67
6	$HCONMe_2$, **9d**	140	90	**8h**, 75
7	CH_3CONMe_2, **9e**	140	90	**3g**, 71

[a] Reaction condition: amide (2 mmol), amine (2.6 mmol) and $[PyPS]_3PW_{12}O_{40}$ (2 mol %) under MW (700 W) and solvent-free condition.
[b] Isolated yields.

Table 4
HPAIL catalyzed transamidation of phthalimide with amines[a]



10 2 11

Entry	Amine	Temp (°C)	Time (min)	Yield[b] (%)
1	$PhNH_2$	140	80	**11a**, 82
2	*p*-$MeOC_6H_4NH_2$	140	80	**11b**, 83
3	$BnNH_2$	120	50	**11c**, 93
4	$BnCH_2NH_2$	120	50	**11d**, 91
5	1-Phenethylamine	140	80	**11e**, 71

[a] Reaction condition: formamide (2 mmol), amine (2.6 mmol) and $[PyPS]_3PW_{12}O_{40}$ (2 mol %) under MW (700 W) and solvent-free condition.
[b] Isolated yields.

chromatography. After this treatment, the recovered catalyst was reused at least five times with a little loss of catalytic efficiency (Table 5), demonstrating the robustness of the catalyst and its reusability.

Table 5
Reusability studies of catalyst for the HPAILs catalyzed transamidation[a]

Number of cycles	1	2	3	4	5
Yield[b] (%)	92	91	89	87	85

[a] Reaction condition: acetamide (2 mmol), benzylamine (2.6 mmol) and $[PyPS]_3PW_{12}O_{40}$ (2 mol %) under MW (700 W) and solvent-free condition at 120 °C.
[b] Isolated yields.

Although the reaction mechanism has not yet been elucidated, the nucleophilicities of amines appeared to play an important role in the transamidation procedure based on the above discussions. Thus a possible reaction mechanism of HPAILs catalyzed transamidation is shown in Fig. 2. The catalytic cycle starts with the activation of carbonyl of amide by the coordination with HPAIL. Amide could be much more active when it is attacked by the nucleophilic amine. The proton-exchange and subsequent deamination could produce amide and regenerate the catalyst.

3. Conclusion

In summary, we have developed a novel catalytic system for the transamidation of non-activated carboxamides with amines using catalytic amounts of HPAILs under MW-assisted and solvent-free conditions. The substrate scope of the methodology has been demonstrated by the reactions of primary, secondary, tertiary amides and phthalimide with aromatic, aliphatic, cyclic, acyclic, primary and secondary amines. Considering the economic attractiveness, operational simplicity, solvent-free media, reusable catalysts and wide functional group tolerance makes the present protocol a green and efficient alternative for the existing reports on the synthesis of amides.

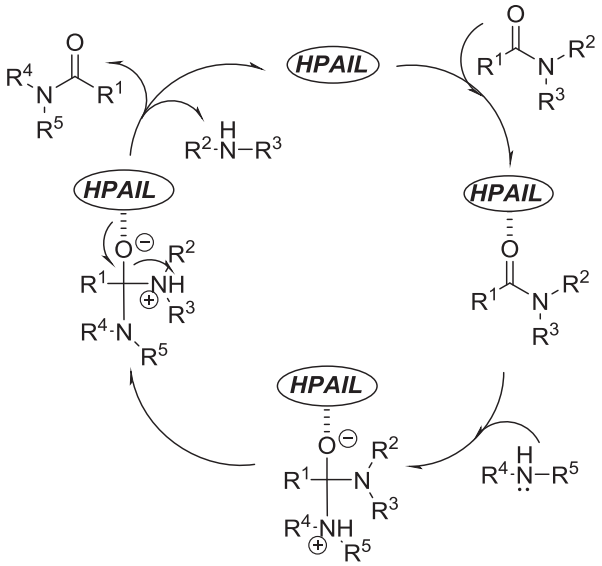


Fig. 2. Plausible mechanism of HPAIL promoted transamidation.

4. Experimental section

4.1. General

Reagent grade solvents were used for extraction, recrystallization and flash chromatography. All other commercial reagents were used as received without additional purification. The progress of reactions was checked by analytical thin-layer chromatography (TLC, silica gel 60 F-254 plates). The plates were visualized first with UV illumination followed by iodine or phosphomolybdic acid hydrate. Column chromatography was performed using silica gel (200–300 mesh). NMR spectra were obtained using BRUKER AVANCE III instrument. ^1H NMR spectra were recorded at 300 MHz and are reported in parts per million (ppm) on the δ scale relative to tetramethylsilane (TMS) as an internal standard. ^{13}C NMR spectra were recorded at 75 MHz and are reported in parts per million (ppm) on the δ scale relative to $CDCl_3$ (δ 77.16) and DMSO-d_6 (δ 39.52). Multiplicities are indicated as the following: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; dd, doubled doublet; td, tripled doublet; br, broad. Coupling constants (J values) where noted are quoted in hertz (Hz). ESI high-resolution mass spectra were obtained using Agilent 1260-6210 (TOF LC/MS) instrument. The melting point was uncorrected.

4.2. General procedure for the synthesis of HPAILs catalysts[12a]

Methylimidazole (0.11 mol) and 1,3-propane sulfone (0.10 mol) were dissolved in toluene (30 mL) and stirred for 24 h at 50 °C under a nitrogen atmosphere. A white precipitate 3-(1-methyl)imidazoliumpropanesulfonate (MIMPS) formed, which was filtered, washed with diethyl ether three times, then dried in a vacuum. MIMPS (0.09 mol) was added to an aqueous solution of $H_3PW_{12}O_{40}$ (0.03 mol), and then the mixture was stirred at room temperature for 24 h. Water was removed in vacuum to give the product $[MIMPS]_3PW_{12}O_{40}$ as a solid. Thus $[MIMPS]_3PMo_{12}O_{40}$, $[PyPS]_3PW_{12}O_{40}$,

$[PyPS]_3PMo_{12}O_{40}$, $[TEAPS]_3PW_{12}O_{40}$ and $[TEAPS]_3PMo_{12}O_{40}$ were prepared using according starting materials.

4.3. General procedure for the synthesis of amides

To a mixture of amide (2 mmol) and amine (2.6 mmol) in a 10 mL round bottomed flask was added $[PyPS]_3PW_{12}O_{40}$ (140 mg, 0.04 mmol). The reaction mixture was stirred at the corresponding temperature under MW (700 W). The progress of the reaction was monitored by TLC. On completion, the mixture was diluted with ethyl acetate (20 mL) with stirring for 30 min. The insoluble catalyst was recovered by filtration or centrifugation. The filtrate was evaporated and the residue in almost pure form. Recrystallization or column chromatography could be used for further purification.

*4.3.1. N-Phenylacetamide (**3a**).* Yellow solid. Mp: 112.8–114.1 °C; IR (KBr): 3294, 3261, 3196, 3137, 1665, 1600, 1558, 1500, 1491, 1435, 1369, 1324, 1266, 768, 760, 693, 534, 511 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.83 (br s, 1H), 7.51–7.49 (m, 2H), 7.32–7.26 (m, 2H), 7.11–7.06 (m, 1H), 2.15 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.9, 138.1, 129.0, 124.4, 120.2, 24.6; HRMS calcd for $C_8H_{10}NO$ ($M+H^+$): 136.0757; found: 136.0752.

*4.3.2. N-(p-Tolyl)acetamide (**3b**).* Yellow solid. Mp: 149.4–152.0 °C; IR (KBr): 3421, 1664, 1606, 1550, 1540, 1380, 768 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.68 (br s, 1H), 7.37 (d, J=8.4 Hz, 2H), 7.09 (d, J=8.1 Hz, 2H), 2.30 (s, 3H), 2.13 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.7, 135.5, 134.0, 129.5, 120.3, 24.5, 21.0; HRMS calcd for $C_9H_{12}NO$ ($M+H^+$): 150.0913; found: 150.0910.

*4.3.3. N-(4-Methoxyphenyl)acetamide (**3c**).* Yellow solid. Mp: 126.5–128.9 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.68 (br s, 1H), 7.41–7.36 (m, 2H), 6.85–6.80 (m, 2H), 3.77 (s, 3H), 2.12 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.7, 156.5, 131.2, 122.1, 114.2, 55.6, 24.3; HRMS calcd for $C_9H_{12}NO_2$ ($M+H^+$): 166.0863; found: 166.0866.

*4.3.4. N-(4-Chlorophenyl)acetamide (**3d**).* Yellow solid. Mp: 133.5–135.1 °C; ^1H NMR (300 MHz, DMSO-d_6) δ 10.06 (s, 1H), 7.63–7.58 (m, 2H), 7.35–7.30 (m, 2H), 2.104 (s, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 168.4, 138.3, 128.6, 126.5, 120.5, 24.0; HRMS calcd for C_8H_9ClNO ($M+H^+$): 170.0367; found: 170.0371.

*4.3.5. N-(4-Nitrophenyl)acetamide (**3e**).* Yellow solid. Mp: 207.8–208.9 °C; IR (KBr): 3303, 1698, 1681, 1620, 1590, 1506, 1346, 1333, 1312, 1301 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 10.57 (br s, 1H), 8.23–8.18 (m, 2H), 7.84–7.79 (m, 2H), 2.11 (s, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 169.4, 145.5, 142.0, 125.0, 118.6, 24.3; HRMS calcd for $C_8H_9N_2O_3$ ($M+H^+$): 181.0608; found: 181.0603.

*4.3.6. N-(Pyridin-2-yl)acetamide (**3f**).* White solid. Mp: 95.4–97.1 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 9.48 (s, 1H), 8.28–8.24 (m, 2H), 7.73 (td, J=8.0, 1.8 Hz, 1H), 7.08–7.03 (m, 1H), 2.21 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.1, 151.9, 147.5, 138.7, 119.7, 114.6, 24.7; HRMS calcd for $C_7H_9N_2O$ ($M+H^+$): 137.0709; found: 137.0711.

*4.3.7. N-Benzylacetamide (**3g**).* Yellow solid. Mp: 55.4–57.1 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.35–7.25 (m, 5H), 6.05 (br s, 1H), 4.40 (d, J=5.4 Hz, 2H), 1.99 (d, J=0.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.1, 138.3, 128.8, 127.9, 127.6, 43.8, 23.3; HRMS calcd for $C_9H_{12}NO$ ($M+H^+$): 150.0913; found: 150.0910.

*4.3.8. N-Cyclohexylacetamide (**3h**).* Yellow oil. ^1H NMR (300 MHz, $CDCl_3$) δ 5.83 (br s, 1H), 3.76–3.65 (m, 1H), 1.93 (s, 3H), 1.89–1.86 (m, 2H), 1.69–1.66 (m, 2H), 1.60–1.57 (m, 1H), 1.37–1.23 (m, 2H), 1.16–1.05 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.3, 48.3, 33.2, 25.6, 25.0, 23.5; HRMS calcd for $C_8H_{16}NO$ ($M+H^+$): 142.1226; found: 142.1229.

*4.3.9. 1-(Piperidin-1-yl)ethanone (**3i**).* Yellow oil. ^1H NMR (300 MHz, $CDCl_3$) δ 3.55 (br s, 2H), 3.39 (br s, 2H), 2.08 (s, 3H), 1.64 (br s, 2H), 1.55 (br s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.9, 47.6, 42.6, 26.5, 25.6, 24.6, 21.6; HRMS calcd for $C_7H_{14}NO$ ($M+H^+$): 128.1070; found: 128.1074.

*4.3.10. N-Benzyl-N-methylacetamide (**3j**).* The presence of two rotamers (ratio 1:1.3) was observed in the NMR spectra. Yellow oil. ^1H NMR (300 MHz, $CDCl_3$) δ 7.40–7.23 (m, 8H), 7.18–7.16 (m, 2H), 4.59 (s, 2H), 4.53 (s, 2H), 2.94 (s, 3H), 2.92 (s, 3H), 2.16 (s, 3H), 2.15 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.0, 170.7, 137.4, 136.6, 129.0, 128.6, 128.0, 127.7, 127.4, 126.3, 54.3, 50.6, 35.5, 33.7, 21.9, 21.5; HRMS calcd for $C_{10}H_{14}NO$ ($M+H^+$): 164.1070; found: 164.1075.

*4.3.11. N-Phenyl-2-phenylacetamide (**4a**).* Yellow solid. Mp: 110.5–112.8 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.43–7.24 (m, 10H), 7.07 (t, J=7.5 Hz, 1H), 3.72 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.3, 137.7, 134.6, 129.6, 129.3, 129.1, 127.8, 124.6, 120.0, 44.9; HRMS calcd for $C_{14}H_{14}NO$ ($M+H^+$): 212.1070; found: 212.1074.

*4.3.12. N-(p-Tolyl)-2-phenylacetamide (**4b**).* Yellow solid. Mp: 124.5–126.0 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.41–7.25 (m, 7H), 7.15 (br s, 1H), 7.07 (d, J=8.1 Hz, 2H), 3.71 (s, 2H), 2.28 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.1, 135.2, 134.7, 134.2, 129.7, 129.5, 129.3, 127.7, 120.1, 44.9, 21.0; HRMS calcd for $C_{15}H_{16}NO$ ($M+H^+$): 226.1226; found: 226.1229.

*4.3.13. N-Benzyl-2-phenylacetamide (**4c**).* Yellow solid. Mp: 101.4–103.4 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.29–7.14 (m, 8H), 7.11–7.08 (m, 2H), 5.74 (br s, 1H), 4.32 (d, J=5.7 Hz, 2H), 3.54 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.0, 138.2, 134.9, 129.7, 129.3, 128.9, 128.5, 127.8, 127.4, 43.9, 43.7; HRMS calcd for $C_{15}H_{16}NO$ ($M+H^+$): 226.1226; found: 226.1230.

*4.3.14. N-(Furan-2-ylmethyl)-2-phenylacetamide (**4d**).* Yellow solid. Mp: 123.4–124.5 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.39–7.25 (m, 6H), 6.29 (dd, J=3.0, 1.8 Hz, 1H), 6.14 (dd, J=3.3, 0.6 Hz, 1H), 5.72 (br s, 1H), 4.40 (d, J=5.7 Hz, 2H), 3.61 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 151.3, 142.2, 134.8, 129.5, 129.0, 127.4, 110.5, 107.3, 43.7, 36.7; HRMS calcd for $C_{13}H_{14}NO_2$ ($M+H^+$): 216.1019; found: 216.1014.

*4.3.15. N-Octyl-2-phenylacetamide (**4e**).* Yellow solid. Mp: 117.5–119.9 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.39–7.24 (m, 5H), 5.43 (br s, 1H), 3.57 (s, 2H), 3.19 (q, J=6.9 Hz, 2H), 1.45–1.36 (m, 2H), 1.23 (m, 10H), 0.87 (t, J=6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.0, 135.2, 129.5, 129.0, 127.3, 44.0, 39.8, 31.8, 29.5, 29.3, 29.2, 26.9, 22.7, 14.1; HRMS calcd for $C_{16}H_{26}NO$ ($M+H^+$): 248.2009; found: 248.2010.

*4.3.16. N-(1-Phenylethyl)-2-phenylacetamide (**4f**).* Yellow solid. Mp: 97.3–99.3 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.38–7.15 (m, 10H), 5.62 (br s, 1H), 5.17–5.07 (m, 1H), 3.58 (s, 2H), 1.39 (d, J=6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.4, 143.1, 135.0, 129.5, 129.1, 128.7, 128.6, 127.4, 126.1, 48.9, 43.9, 21.9; HRMS calcd for $C_{16}H_{18}NO$ ($M+H^+$): 240.1383; found: 240.1387.

*4.3.17. 2-Phenyl-1-(piperidin-1-yl)ethanone (**4g**).* Yellow oil. ^1H NMR (300 MHz, $CDCl_3$) δ 7.34–7.20 (m, 5H), 3.73 (s, 2H), 3.59–3.55 (m, 2H), 3.39–3.35 (m, 2H), 1.62–1.48 (m, 4H), 1.38–1.30 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.3, 135.5, 128.7, 128.6, 126.7, 47.3, 42.9, 41.2, 26.2, 25.5, 24.5; HRMS calcd for $C_{13}H_{18}NO$ ($M+H^+$): 204.1383; found: 204.1387.

*4.3.18. N-Benzyl-N-methyl-2-phenylacetamide (**4h**).* The presence of two rotamers (ratio 1:1.3) was observed in the NMR spectra. Oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48–7.28 (m, 18H), 7.11–7.08 (m, 2H), 4.61 (s, 2H), 4.53 (s, 2H), 3.79 (s, 2H), 3.76 (s, 2H), 2.95 (s, 3H), 2.90 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.5, 171.2, 137.3, 136.5, 135.1, 135.0, 129.0, 128.9, 128.8, 128.7, 128.6, 128.5, 128.1, 127.7, 127.4, 126.9, 126.8, 126.4, 53.7, 51.0, 41.2, 41.0, 35.3, 34.1; HRMS calcd for $C_{16}H_{18}NO$ ($M+H^+$): 240.1383; found: 240.1385.

*4.3.19. N-Phenylisobutyramide (**5a**).* Yellow solid. Mp: 115.3–117.5 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.55–7.53 (m, 2H), 7.47 (br s, 1H), 7.33–7.27 (m, 2H), 7.11–7.06 (m, 1H), 7.59–7.45 (m, 1H), 1.24 (d, J=6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.5, 137.2, 128.0, 123.2, 119.0, 35.8, 18.7; HRMS calcd for $C_{10}H_{14}NO$ ($M+H^+$): 164.1070; found: 164.1073.

*4.3.20. N-(p-Tolyl)isobutyramide (**5b**).* Yellow solid. Mp: 109.3–110.1 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, J=8.4 Hz, 2H), 7.26 (br s, 1H), 7.11 (d, J=8.1 Hz, 2H), 2.56–2.43 (m, 1H), 2.31 (s, 3H), 1.24 (d, J=6.6 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.3, 135.6, 133.8, 129.5, 120.0, 36.7, 21.0, 19.8; HRMS calcd for $C_{11}H_{16}NO$ ($M+H^+$): 178.1226; found: 178.1231.

*4.3.21. N-Phenethylisobutyramide (**5c**).* Yellow solid. Mp: 88.9–90.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34–7.17 (m, 5H), 5.62 (br s, 1H), 5.27 (q, J=6.9 Hz, 2H), 2.81 (t, J=6.9 Hz, 2H), 2.36–2.22 (m, 1H), 1.11 (d, J=6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 177.0, 139.1, 128.9, 128.7, 126.6, 40.5, 35.8, 35.7, 19.7; HRMS calcd for $C_{12}H_{18}NO$ ($M+H^+$): 192.1383; found: 192.1384.

*4.3.22. N-Phenylbenzamide (**6a**).* Yellow solid. Mp: 164.2–166.6 °C; IR (KBr): 3346, 1657, 1601, 1579, 1536, 1493, 1449, 1440, 1329, 1301, 1262, 760, 716, 692, 683 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 (br s, 1H), 7.88–7.84 (m, 2H), 7.66–7.62 (m, 2H), 7.56–7.43 (m, 3H), 7.39–7.33 (m, 2H), 7.18–7.12 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.9, 138.0, 135.1, 132.0, 129.2, 128.9, 127.2, 124.7, 120.4; HRMS calcd for $C_{13}H_{12}NO$ ($M+H^+$): 198.0913; found: 198.0916.

*4.3.23. N-(p-Tolyl)benzamide (**6b**).* Yellow solid. Mp: 152.2–153.5 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 (br s, 1H), 7.87–7.82 (m, 2H), 7.55–7.42 (m, 5H), 7.15 (d, J=8.4 Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.8, 135.5, 135.2, 134.3, 131.8, 129.7, 128.8, 127.1, 120.5, 21.0; HRMS calcd for $C_{14}H_{14}NO$ ($M+H^+$): 212.1070; found: 212.1075.

*4.3.24. N-Benzylbenzamide (**6c**).* Yellow solid. Mp: 120.2–122.5 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82–7.77 (m, 2H), 7.52–7.29 (m, 8H), 6.53 (br s, 1H), 4.66–4.61 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 138.3, 134.5, 131.7, 128.9, 128.7, 128.0, 127.7, 127.1, 44.2; HRMS calcd for $C_{14}H_{14}NO$ ($M+H^+$): 212.1070; found: 212.1074.

*4.3.25. N-Phenethylbenzamide (**6d**).* Yellow solid. Mp: 133.4–136.1 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 (d, J=7.2 Hz, 2H), 7.50–7.22 (m, 8H), 6.27 (br s, 1H), 3.71 (q, J=6.9 Hz, 2H), 2.93 (t, J=6.9 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.6, 139.0, 134.8, 131.5, 128.9, 128.8, 128.6, 126.9, 126.7, 41.3, 35.8; HRMS calcd for $C_{15}H_{16}NO$ ($M+H^+$): 226.1226; found: 226.1229.

*4.3.26. N-Cyclohexylbenzamide (**6e**).* Yellow solid. Mp: 150.1–153.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, J=6.9 Hz, 2H), 7.48–7.37 (m, 3H), 6.13 (br s, 1H), 4.02–3.91 (m, 1H), 2.02–1.98 (m, 2H), 1.76–1.71 (m, 2H), 1.67–1.61 (m, 1H), 1.49–1.34 (m, 2H), 1.27–1.16 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7, 135.2, 131.3, 128.6, 126.9, 48.8, 33.3, 25.7, 25.0; HRMS calcd for $C_{13}H_{18}NO$ ($M+H^+$): 204.1383; found: 204.1386.

*4.3.27. N-(1-Phenylethyl)benzamide (**6f**).* Yellow solid. Mp: 124.5–126.3 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, J=6.9 Hz, 2H), 7.52–7.32 (m, 7H), 7.31–7.26 (m, 1H), 6.38 (br s, 1H), 5.39–5.29 (m, 1H), 1.61 (d, J=6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7, 143.3, 134.8, 131.6, 128.9, 128.7, 127.6, 127.1, 126.4, 49.4, 21.9; HRMS calcd for $C_{15}H_{16}NO$ ($M+H^+$): 226.1226; found: 226.1229.

*4.3.28. Phenyl(piperidin-1-yl)methanone (**6g**).* Yellow solid. Mp: 108.3–109.5 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (br s, 5H), 3.71 (br s, 2H), 3.34 (br s, 2H), 1.67 (br s, 4H), 1.51 (br s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3, 136.5, 129.3, 128.4, 126.8, 48.7, 43.1, 26.5, 25.6, 24.6; HRMS calcd for $C_{12}H_{16}NO$ ($M+H^+$): 190.1226; found: 190.1230.

*4.3.29. N-(p-Tolyl)nicotinamide (**7a**).* Yellow solid. Mp: 164.3–166.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.08 (d, J=1.8 Hz, 1H), 8.77 (dd, J=4.8, 1.5 Hz, 1H), 8.21 (dt, J=7.8, 1.8 Hz, 1H), 7.89 (br s, 1H), 7.52 (d, J=8.4 Hz, 2H), 7.44 (dd, J=7.8, 0.9 Hz, 1H), 7.19 (d, J=8.4 Hz, 2H), 2.35 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.2, 152.2, 148.1, 135.5, 135.1, 134.8, 131.0, 129.7, 123.6, 120.9, 21.0; HRMS calcd for $C_{13}H_{13}N_2O$ ($M+H^+$): 213.1022; found: 213.1026.

*4.3.30. N-Phenethylnicotinamide (**7b**).* Yellow solid. Mp: 132.5–134.1 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.86 (d, J=1.8 Hz, 1H), 8.65 (dd, J=4.8, 1.5 Hz, 1H), 8.07 (dt, J=8.1, 1.8 Hz, 1H), 7.38–7.21 (m, 6H), 6.62 (br s, 1H), 3.72 (q, J=6.9 Hz, 2H), 2.94 (t, J=6.9 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.7, 152.0, 147.7, 138.7, 135.4, 130.5, 128.9, 128.8, 126.8, 123.6, 41.4, 35.6; HRMS calcd for $C_{14}H_{15}N_2O$ ($M+H^+$): 227.1179; found: 227.1181.

*4.3.31. N-Phenylformamide (**8a**).* The presence of two rotamers (ratio 1:1.1) was observed in the NMR spectra. White solid. Mp: 48.1–50.6 °C; IR (KBr): 3300, 2877, 1650, 1540, 1438, 1403, 1290 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.69 (br s, 2H), 8.36 (d, J=1.5 Hz, 1H), 7.79 (br s, 1H), 7.55 (d, J=7.5 Hz, 2H), 7.38–7.30 (m, 4H), 7.21–7.09 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.0, 159.4, 137.0, 136.9, 129.8, 129.2, 125.4, 124.9, 120.2, 118.9; HRMS calcd for C_7H_8NO ($M+H^+$): 122.0600; found: 122.0603.

*4.3.32. N-p-Tolylformamide (**8b**).* The presence of two rotamers (ratio 1:1) was observed in the NMR spectra. White solid. Mp: 51.5–53.3 °C; IR (KBr): 3030, 1688, 1615, 1519, 1303 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.63 (d, J=11.1 Hz, 1H), 8.52 (br s, 1H), 8.33 (d, J=1.5 Hz, 1H), 7.65 (br s, 1H), 7.42 (d, J=8.4 Hz, 2H), 7.14 (t, J=8.4 Hz, 4H), 6.99 (d, J=8.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.0, 159.2, 135.2, 134.6, 134.5, 134.2, 130.3, 129.7, 120.2, 119.2, 21.0, 20.9; HRMS calcd for $C_8H_{10}NO$ ($M+H^+$): 136.0757; found: 136.0761.

*4.3.33. N-(4-Methoxyphenyl)formamide (**8c**).* The presence of two rotamers (ratio 1:1) was observed in the NMR spectra. Brown solid. Mp: 76.5–79.4 °C; IR (KBr): 3246, 3052, 2890, 2939, 2363, 1660, 1511, 1238 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.51 (d, J=11.4 Hz, 1H), 8.31 (d, J=1.8 Hz, 1H), 8.21 (br d, J=10.5 Hz, 1H), 7.53 (br s 1H), 7.47–7.42 (m, 2H), 7.07–7.02 (m, 2H), 6.91–6.83 (m, 4H), 3.80 (s, 3H), 3.79 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.3, 159.1, 157.8, 156.8, 130.1, 129.7, 121.9, 121.8, 115.0, 114.3, 55.7, 55.6; HRMS calcd for $C_8H_{10}NO_2$ ($M+H^+$): 152.0706; found: 152.0709.

*4.3.34. N-(4-Chlorophenyl)formamide (**8d**).* The presence of two rotamers (ratio 1:1.3) was observed in the NMR spectra. Yellow solid. Mp: 104.3–106.4 °C; IR (KBr): 3258, 1680, 1671, 1607, 1543 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.78 (br s, 1H), 8.66 (d, J=11.4 Hz, 1H), 8.36 (br s, 1H), 7.89 (br s, 1H), 7.59 (br s, 1H), 7.50 (d, J=8.7 Hz, 2H), 7.34–7.26 (m, 4H), 7.05 (d, J=8.7 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.8, 159.3, 135.5, 135.4, 130.8, 130.0, 129.2, 126.0, 121.4, 120.2; HRMS calcd for C_7H_7ClNO ($M+H^+$): 156.0211; found: 156.0215.

*4.3.35. N-(4-Nitrophenyl)formamide (**8e**).* The presence of two rotamers (ratio 3.3:1) was observed in the NMR spectra. Yellow solid.

Mp: 193.5–195.8 °C; IR (KBr): 3248, 3050, 2941, 2367, 1660, 1541, 1235 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 10.82 (br s, 1H), 10.71 (d, J=10.8 Hz, 1H), 9.05 (d, J=10.5 Hz, 1H), 8.40 (br s, 1H), 8.23–8.17 (m, 2.5H), 7.93 (d, J=8.7 Hz, 0.5H), 7.81 (d, J=8.7 Hz, 2H), 7.41 (d, J=8.7 Hz, 2H), 6.73 (br s, 0.5H), 6.59 (d, J=9.0 Hz, 0.5H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 162.8, 160.5, 144.2, 142.5, 125.5, 125.1, 119.0, 116.6; HRMS calcd for $C_7H_7N_2O_3$ ($M+H^+$): 167.0451; found: 167.0455.

*4.3.36. N-(Naphthalen-1-yl)formamide (**8f**).* The presence of two rotamers (ratio 5:2) was observed in the NMR spectra. White solid. Mp: 125.8–127.3 °C; IR (KBr): 3226, 1578, 1538, 1504, 1396, 1270, 790 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 9.10 (d, J=9.6 Hz, 1H), 8.64 (d, J=11.1 Hz, 1H), 8.60 (br s, 1H), 8.05 (d, J=7.8 Hz, 1H), 7.99 (d, J=7.5 Hz, 1H), 7.91–7.85 (m, 2H), 7.79 (d, J=8.4 Hz, 1H), 7.71 (d, J=8.1 Hz, 1H), 7.62–7.43 (m, 3H), 7.31 (d, J=7.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.5, 160.0, 134.3, 134.1, 132.4, 131.1, 128.9, 128.6, 127.8, 127.1, 127.0, 126.9, 126.9, 126.3, 126.2, 125.8, 125.6, 121.5, 121.0, 120.6, 119.0; HRMS calcd for $C_{11}H_{10}NO$ ($M+H^+$): 172.0757; found: 172.0759.

*4.3.37. N-(Pyridin-2-yl)formamide (**8g**).* The presence of two rotamers (ratio 1.2:1) was observed in the NMR spectra. Yellow solid. Mp: 34.1–36.5 °C; IR (KBr): 3197, 2852, 1691, 1595, 1433, 1303, 778, 517 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 10.40 (br s, 1H), 10.21 (br s, 1H), 9.34 (d, J=10.8 Hz, 1H), 8.32 (br s, 1H), 8.55 (s, 1H), 8.36–8.33 (m, 1H), 8.28 (d, J=8.4 Hz, 1H), 7.76 (td, J=8.0, 1.8 Hz, 1H), 7.69 (td, J=7.8, 1.8 Hz, 1H), 7.137–7.06 (m, 2H), 6.96 (d, J=8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.2, 159.7, 151.2, 151.1, 148.6, 147.5, 139.0, 138.8, 120.3, 119.9, 115.3, 110.6; HRMS calcd for $C_6H_7N_2O$ ($M+H^+$): 123.0553; found: 123.0556.

*4.3.38. N-Benzylformamide (**8h**).* The presence of two rotamers (ratio 5.5:1) was observed in the NMR spectra. White solid. Mp: 60.4–62.8 °C; IR (KBr): 1650 (C=O) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.19 (s, 1H), 8.11 (d, J=12.0 Hz, 1H), 7.39–7.22 (m, 10H), 6.34 (br s, 2H), 4.43 (d, J=6.0 Hz, 2H), 4.37 (d, J=6.3 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.8, 161.2, 137.7, 129.0, 128.8, 128.0, 127.8, 127.7, 127.0, 45.7, 42.2; HRMS calcd for $C_8H_{10}NO$ ($M+H^+$): 136.0757; found: 136.0762.

*4.3.39. N-(Furan-2-ylmethyl)formamide (**8i**).* The presence of two rotamers (ratio 5:1) was observed in the NMR spectra. Yellow oil. ^1H NMR (300 MHz, $CDCl_3$) δ 8.20 (s, 1H), 8.15 (d, J=12.0 Hz, 1H), 7.39 (dd, J=1.8, 0.6 Hz, 1H), 7.36 (dd, J=1.8, 0.6 Hz, 1H), 6.45–6.32 (m, 4H), 6.25–6.24 (m, 2H), 4.47 (d, J=6.0 Hz, 2H), 4.37 (d, J=6.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.8, 161.1, 150.9, 150.8, 142.9, 142.4, 110.6, 110.5, 107.7, 107.6, 39.0, 35.1; HRMS calcd for $C_6H_8NO_2$ ($M+H^+$): 126.0550; found: 126.0554.

*4.3.40. N-(1-Phenylethyl)formamide (**8j**).* The presence of two rotamers (ratio 5:1) was observed in the NMR spectra. Yellow oil. ^1H NMR (300 MHz, $CDCl_3$) δ 8.13 (d, J=12.0 Hz, 1H), 8.12 (s, 1H), 7.39–7.24 (m, 10H), 6.39 (br s, 1H), 6.17 (br s, 1H), 5.24–5.14 (m, 1H), 4.72–4.62 (m, 1H), 1.55 (d, J=6.9 Hz, 3H), 1.50 (d, J=6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.3, 160.4, 142.9, 142.7, 129.0, 128.8, 127.9, 127.6, 126.2, 125.9, 51.8, 47.7, 29.8, 21.8; HRMS calcd for $C_9H_{12}NO$ ($M+H^+$): 150.0913; found: 150.0918.

*4.3.41. N-tert-Butylformamide (**8k**).* The presence of two rotamers (ratio 1:1) was observed in the NMR spectra. Yellow oil. ^1H NMR (300 MHz, $CDCl_3$) δ 8.22 (d, J=12.3 Hz, 1H), 7.98 (d, J=1.5 Hz, 1H), 6.51 (br s, 1H), 5.62 (br s, 1H), 1.34 (s, 9H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.2, 160.7, 51.4, 50.4, 30.0, 29.0; HRMS calcd for $C_5H_{12}NO$ ($M+H^+$): 102.0913; found: 102.0918.

*4.3.42. N-Methyl-N-phenylformamide (**8l**).* Yellow oil. ^1H NMR (300 MHz, $CDCl_3$) δ 8.49 (s, 1H), 7.46–7.39 (m, 2H), 7.31–7.26 (m, 1H), 7.20–7.16 (m, 2H), 3.33 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.5, 142.3, 129.7, 126.5, 122.5, 32.2; HRMS calcd for $C_8H_{10}NO$ ($M+H^+$): 136.0757; found: 136.0755.

*4.3.43. N-Benzyl-N-methylformamide (**8m**).* The presence of two rotamers (ratio 4:3) was observed in the NMR spectra. Yellow oil. ^1H NMR (300 MHz, $CDCl_3$) δ 8.29 (s, 1H), 8.16 (s, 1H), 7.40–7.19 (m, 10H), 4.52 (s, 2H), 4.39 (s, 2H), 2.84 (s, 3H), 2.78 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.8, 162.6, 136.1, 135.9, 129.0, 128.8, 128.3, 128.2, 127.7, 127.5, 53.5, 47.8, 34.1, 29.5; HRMS calcd for $C_9H_{12}NO$ ($M+H^+$): 150.0913; found: 150.0915.

*4.3.44. Piperidine-1-carbaldehyde (**8n**).* Colourless oil. IR (KBr): 2940, 2860, 1588, 1441, 1400, 1369, 1348, 1328, 1280, 1118 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.00 (br s, 1H), 3.48 (t, J=5.7 Hz, 2H), 3.32 (t, J=5.7 Hz, 2H), 1.74–1.66 (m, 2H), 1.62–1.51 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.8, 46.8, 40.7, 26.6, 25.1, 24.8; HRMS calcd for $C_6H_{12}NO$ ($M+H^+$): 114.0913; found: 114.0916.

*4.3.45. 2-Phenylisoindoline-1,3-dione (**11a**).* Yellow solid. Mp: 165.5–167.3 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.97–7.94 (m, 2H), 7.80–7.77 (m, 2H), 7.54–7.45 (m, 2H), 7.43–7.38 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.4, 134.5, 131.9, 131.8, 129.2, 128.2, 126.7, 123.9; HRMS calcd for $C_{14}H_{10}NO_2$ ($M+H^+$): 224.0706; found: 224.0708.

*4.3.46. 2-(4-Methoxyphenyl)isoindoline-1,3-dione (**11b**).* Yellow solid. Mp: 156.5–158.3 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.96–7.93 (m, 2H), 7.80–7.77 (m, 2H), 7.36–7.31 (m, 2H), 7.05–7.00 (m, 2H), 3.85 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.7, 159.4, 134.5, 132.0, 128.1, 124.4, 123.8, 114.6, 55.7; HRMS calcd for $C_{15}H_{12}NO_3$ ($M+H^+$): 254.0812; found: 254.0814.

*4.3.47. 2-Benzylisoindoline-1,3-dione (**11c**).* Yellow solid. Mp: 138.4–139.9 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.87–7.81 (m, 2H), 7.72–7.67 (m, 2H), 7.45–7.42 (m, 2H), 7.34–7.23 (m, 3H), 4.85 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 136.5, 134.1, 132.2, 128.8, 128.7, 127.9, 123.5, 41.7; HRMS calcd for $C_{15}H_{12}NO_2$ ($M+H^+$): 238.0863; found: 238.0867.

*4.3.48. 2-Phenethylisoindoline-1,3-dione (**11d**).* Yellow solid. Mp: 126.4–128.6 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.84–7.81 (m, 2H), 7.71–7.68 (m, 2H), 7.31–7.19 (m, 5H), 3.92 (t, J=7.8 Hz, 2H), 2.99 (t, J=7.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 138.1, 134.0, 132.2, 129.0, 128.7, 126.8, 123.3, 39.4, 34.7; HRMS calcd for $C_{16}H_{14}NO_2$ ($M+H^+$): 252.1019; found: 252.1022.

*4.3.49. 2-(1-Phenylethyl)isoindoline-1,3-dione (**11e**).* Yellow solid. Mp: 85.8–88.9 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.82–7.77 (m, 2H), 7.69–7.65 (m, 2H), 7.52–7.50 (m, 2H), 7.36–7.23 (m, 3H), 5.57 (q, J=7.5 Hz, 1H), 1.93 (d, J=7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 140.4, 134.0, 132.1, 128.6, 127.8, 127.5, 123.2, 49.7, 17.6; HRMS calcd for $C_{16}H_{14}NO_2$ ($M+H^+$): 252.1019; found: 252.1023.

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Supplementary data

Characterization data and copies of the ^{1}H NMR and ^{13}C NMR spectra for all final products can be found. Supplementary data associated with this article can be found in the online version, at http://dx.doi.org/10.1016/j.tet.2014.10.066.

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