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EXPERIMENTAL PAPER

Solvent-free, Efficient Transamidation of Carboxamides with Amines Catalyzed by Recyclable Sulfated Polyborate Catalyst

Anil S. Mali, Krishna Indalkar, and Ganesh U. Chaturbhuj

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Transamidation is a significant reaction in organic and medicinal chemistry. The amide is one of the most important functional groups used in organic transformations, and it is found in a wide variety of dyes, polymers, peptide dendrimers, agrochemicals, pharmaceuticals, and biologically active compounds.[1–5] In 2007, the American Chemical Society Green Chemistry Institute Pharmaceutical Roundtable (ACS GCIPR) designated amidation as a challenging goal in organic chemistry for which green, efficient, and sustainable alternative methods are required.[6] The amide link forms the backbone of proteins and peptides.[7] It is an inherent part of many natural products such as capsaicin, piperine, *N*-acetyl anthranilic acid, taxol, and penicillin-G and drugs such as mepivacaine, lidocaine, articaine, amoxicillin, acetazolamide, valsartan, atorvastatin, protirelin, captopril, enalapril, chloramphenicol, methyprylon, benzipram, zolpidem, and many others. Fatty acid amides exhibit excellent antimicrobial, anti-inflammatory, antiproliferative, and antitubercular activities.[5] Amides are useful intermediates for the synthesis of pharmacologically important heterocycles containing nitrogen and oxygen.[9] It has been estimated that nearly 25% of active pharmaceutical ingredients contain amide functional groups.[3]

Transamidation involves the cleavage of the C-N bond in an amide reactant and the formation of a new C-N bond in the product.[10] The exchange of the amine moiety of an amide is a conceptually simple but rare organic transformation, due to the modest reactivity of amides. Traditionally, amide synthesis involves the reaction of amines with carboxylic acid derivatives (chlorides, anhydrides, or esters), aldehydes, alcohols, or hydration of nitriles and hydroamination of alkynes.[11,12] These procedures are often limited by harsh reagents, high temperature, tedious isolation, or the generation of waste. In this vein, a number of traditional name reactions, including the Schmidt,[13] Ritter,[14] Beckmann,[15] and Ugi[16] reactions have been reported for amide synthesis.

Up to the present, the literature for transamidation includes the use of Bronsted acid ionic liquid,[17] KO^{t}Bu,[18,19] polymer-bound $HfCl_4$,[20] $AlCl_3$,[21] $Eu(OTf)_3$,[22] $ZrCl_4$,[23] $Ti(NMe_2)_4$,[24] $Sc(OTf)_3$,[25] CeO_2,[26] binuclear Mn(II) complexes,[27] Cp_2ZrCl_2,[28] Fe(III),[11] Rh(II) NHC complexes,[29] benzoic acid,[30] boric acid,[31] *L*-proline,[12] hypervalent iodine,[32] copper acetate,[33] MnO_2,[34] H_2SO_4-SiO_2,[35] H-β Zeolite,[9] Chitosan,[36] $B(OCH_2CF_3)_3$,[37]

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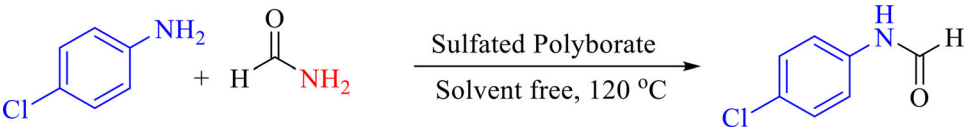


Scheme 1. Transamidation reaction of formamide and 4-chloroaniline to form *N*-(4-chlorophenyl)formamide.

Table 1. Effect of catalyst loading and temperature for the transamidation of formamide.[a]

Entry	Catalyst (Wt. %)	Temperature (°C)	Time (h)	Yield (%)[b]
1	0	120	12	NR[c]
2	2.5	120	12	72
3	5.0	120	12	81
4	7.5	120	12	88
5	10	120	1	97
6	15	120	1	97
7	10	90	12	68
8	10	60	12	24
9	10	Rt	12	NR[c]

[a]Reaction condition: 4-Chloroaniline (1 mmol), formamide (1.2 mmol) and sulfated polyborate catalyst.
[b]Isolated yield.
[c]No reaction.

nano-magnetic $Fe(OH)_3@Fe_3O_4$,[38] nano-magnetic sulfonic acid,[5] OSU-6,[39] Fe^{3+}-exchanged clay,[40] $K_2S_2O_8$,[8] the use of microwave technology[41] and conventional heating without catalyst with prolonged time.[42] As the field has developed in a stepwise manner over many years, each of these methods has its own merits and drawbacks, and there are still opportunities for improvement.

In keeping with our previous experiments in the development of sulfated polyborate[43] and in catalysis for *N*-formylation,[44] direct amidation[45] quinoline synthesis,[46] reductive amination,[47] iodonation of arenes[48] and the present study was structured to investigate the suitability of sulfated polyborate as a catalyst for the transamidation of carboxamides focusing on convenience, high yields and recyclability of the catalyst. For the preliminary experiments, 4-chloroaniline (1 mmol) and formamide (1.2 mmol) were used in a model reaction to afford *N*-(4-chlorophenyl)formamide (Scheme 1, Tables 1 and 2).

The reaction did not proceed in the absence of a catalyst at 120 °C (Table 1, entry 1). An increase of the catalyst loading increased the product yield with a reduction in reaction time (Table 1, entries 2-5). However, catalyst loading beyond 10 wt. % was not advantageous (Table 1, entry 6), so 10 wt. % catalyst loading was chosen for further work. Temperature also played an important role in the model reaction. The best results were obtained at 120 °C resulting in increased product yield in shorter reaction times (Table 1, entry 5). We settled on this as the optimum temperature for performing the reaction.

The effect of several solvents on time and yield of the reaction was ascertained (Table 2). None of the solvents had any advantage of time and yield over the solvent-free condition. Hence, the solvent-free protocol was regarded as the best for cost and environmental acceptability.

The reusability of the catalyst in the model reaction under solvent-free condition at 120 °C was evaluated. The recovered catalyst could be recycled as many as four times

Table 2. The effect of solvents on our transamidation of formamide.[a]

Entry	Solvent	Temperature (°C)	Time (h)	Yield (%)[b]
1	Solvent-free	120	1	97
2	EtOH	Reflux	12	67
3	MeCN	Reflux	12	51
4	THF	Reflux	12	40
5	Water	Reflux	12	NR[c]
6	Toluene	reflux	12	Traces
7	DMSO	120	12	32

[a]Reaction condition: 4-Chloroaniline(1 mmol), and formamide (1.2 mmol) and sulfated polyborate catalyst.
[b]Isolated yield.
[c]No reaction.

Table 3. The efficiency of sulfated polyborate in comparison with literature catalysts.

Entry	Catalyst	Reaction condition	Time (h)	Yield (%)	Ref.
1	Sulfated Polyborate	Solvent-free/120 °C	1	97	This work
2	Nano Fe_3O_4 supported	Solvent-free/120 °C	2	91	5
3	H_2SO_4-SiO_2	Solvent-free/70 °C	6	90	35
4	$[RuHCl(CO)(PPh_3)_3]$	Toluene/110 °C Sealed Tube	8	99	29
5	Nano-magnetic $Fe(OH)_3$@Fe_3O_4	*p*-Xylene/138 °C	10	81	38
6	Boric acid	Water/150 °C	17	92	31
7	Binuclear Mn(II) complex	Solvent-free/120 °C	24	87	27
8	Pd(OAc)/Bpy/PivOH/BF_3·Et_2O	Toluene/120 °C	24	69	39
9	*L*-proline	Solvent-free/150 °C	36	74	12
10	Chitosan	Solvent-free/150 °C/ sealed tube	36	81	36

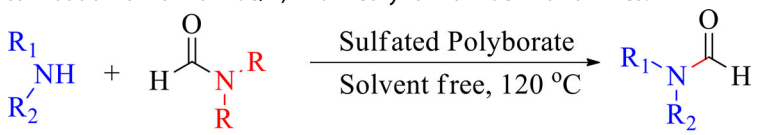
with no significant loss in catalytic activity; the catalyst could be used in the same way in the fourth run as it had been used in the first (fourth run 89% yield).

Table 3 shows a comparison of catalysts reported in the literature for the transamidation of formamide; the present method ranks among the best in terms of reaction conditions, workup procedure, time, and yields.

To study the synthetic utility and scope, the optimized reaction condition was applied to substituted amines using formamide and DMF as a formylating agent, with acetamide and dimethylacetamide (DMA) as an acetylating agent (Tables 4 and 5). The sulfated polyborate catalyzed solvent-free transamidation of DMF, formamide, acetamide, and DMA with substituted amines was effective. Reactions proceeded smoothly and afforded the desired products in good to excellent yields. Several electron-donating or electron-withdrawing substituents at the *para* position of aniline were examined and resulted in good to excellent yields (Table 4, entries 1a-e). This protocol was also extended to benzylamine and cyclohexylamine with good yields (Table 4, entries **1f** and **1g**). Alicyclic secondary amines reacted smoothly and gave good yields (Table 5, entry **1h**). Formylation reactions using DMF were slightly less effective in terms of reaction time and yield compared to formamide.

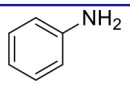
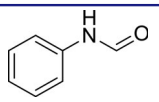
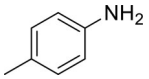
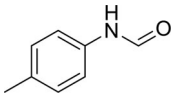
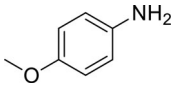
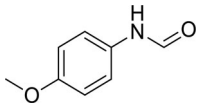
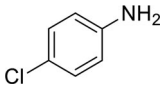
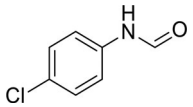
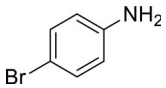
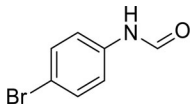
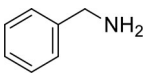
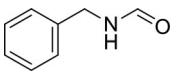
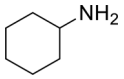
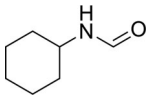
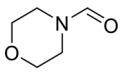
Encouraged by these promising results, our attention turned to *N*-acetylation of amines through transamidation using acetamide and DMA. The transamidation of acetamide and DMA with various amines was demonstrated (Table 5, entries **2a**-**2e**). This protocol was extended to aniline and substituted anilines, benzylic, and alicyclic

Table 4. Transamidation of formamide/*N, N*-dimethylformamide with amines.



A: R = H
B: R = CH_3

1a-h

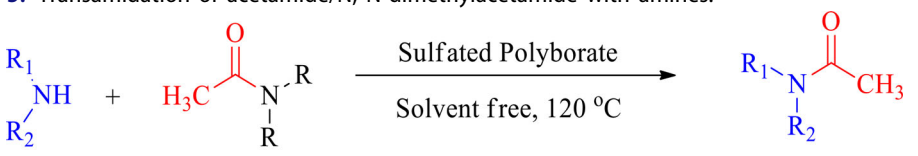
Entry	Amines	Products	Carboxamide	Time (h)	Yield (%)
1a			A B	2 6	84 79
1b			A B	1 5	97 82
1c			A B	1 5	94 88
1d			A B	1 5	97 89
1e			A B	1 5	86 80
1f			A B	1 5	94 91
1g			A B	3 6	88 83
1h			A B	2 6	85 87

secondary amines which reacted smoothly and gave good yields. Acetylation reactions using acetamide were relatively more efficient compared to DMA.

To further expand the application of the protocol, the transamidation of phthalimide with primary aromatic, benzylic, and alicyclic amines was investigated. In all cases, the reaction proceeded smoothly giving the corresponding *N*-substituted phthalimides in high yields (Table 7, entries **3a**-**3e**). Significantly, this method can now be used as an alternative for the phthaloyl protection of primary amines.

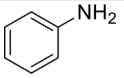
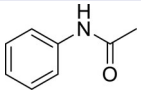
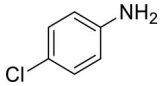
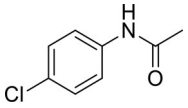
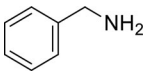
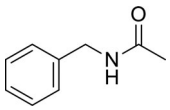
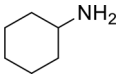
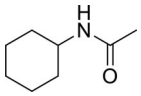
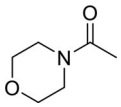
The ^{1}HNMR data of the products in Table 4, entries **1a**-**g** revealed the presence of a mixture of rotamers,[49] well depicted in the spectral data of the compounds. Table 7 provides the characterization data with literature references.

Table 5. Transamidation of acetamide/N, N-dimethylacetamide with amines.



A: R = H
B: R = CH_3

2a-e

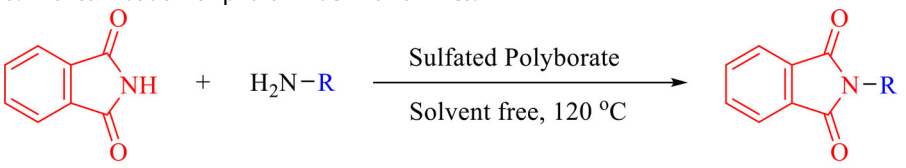
Entry	Amines	Products	Carboxamide	Time (h)	Yield (%)
2a			A B	5 6	85 75
2b			A B	4 6	92 84
2c			A B	4 6	94 86
2d			A B	5 8	88 76
2e			A B	5 8	85 78

In summary, we have demonstrated a widely applicable and simple method for transamidation of carboxamides with amines using sulfated polyborate as a catalyst. The present protocol is applicable for transamidation of DMF, formamide, acetamide, DMA, and phthalimide with substituted anilines, benzylic, and alicyclic primary and secondary amines. High yields, simple procedure, easy workup, shorter reaction time, recyclability of the catalyst, and environmentally benign reaction condition are the key features of this procedure. Moreover, this protocol has the ability to tolerate a wide variety of substituents.

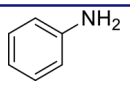
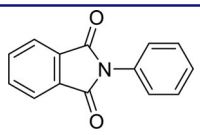
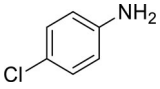
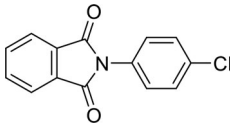
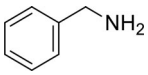
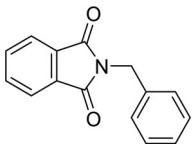
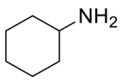
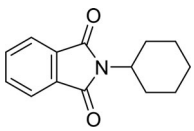
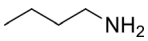
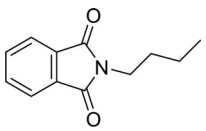
Experimental section

Melting points of all the compounds were recorded on an Analab ThermoCal melting point apparatus in an open capillary tube and are uncorrected. Chemicals and solvents used were of LR grade and purchased from Avra synthesis, SD fine, and Spectrochem and were used without purification. The preparation and characterization of sulfated polyborate have been previously described.[43] The purity determination of the starting materials and reaction monitoring was accomplished by thin-layer chromatography (TLC) on Merck silica gel G F_{254} plates. ^{1}H NMR spectra were recorded on an MR400

Table 6. Transamidation of phthalimide with amines.



3a-3e

Entry	Amines	Products	Time (h)	Yield (%)
3a			3	95
3b			3	97
3c			3	96
3d			3	91
3e			3	89

Agilent Technology NMR spectrometer using tetramethylsilane (TMS) as an internal standard and $CDCl_3$ as a solvent. All the products are known compounds and were characterized by ^1H NMR spectroscopy for structural identification. Copies of the original spectra were submitted for review and are available as supporting information in the online version of this article or from the corresponding author upon request. Physical constants matched the literature values of the references shown in Table 7. Representative spectral data are shown below.

Representative procedure for transamidation of carboxamides

A mixture of amine (2.0 mmol), carboxamide (2.4 mmol) and sulfated polyborate (10 wt. %) was heated at 120 °C. The reaction was monitored by thin-layer chromatography. After completion of the reaction, the mixture was cooled to room temperature and quenched with water; the precipitated solid was filtered at the vacuum pump, washed

Table 7. Physical constants of all compounds.

Entry	Observed M. P. (°C)	Literature M. P. (°C)	Entry	Observed M. P. (°C)	Literature M. P. (°C)
1a	44-46	45 – 48[50]	**2b**	170-172	172-174[57]
1b	50-52	50 – 53[50]	**2c**	57-59	60-62[58]
1c	78-80	79-81[51]	**2d**	105-107	104-105[59]
1d	99-101	102 – 105[50]	**2e**	239-241[a]	242-243[a,60]
1e	116-119	117-121[52]	**3a**	206-208	208-210[61]
1f	61-63	60-62[53]	**3b**	198-200	200-201[62]
1g	252-255[a]	260[a,54]	**3c**	115-117	113-115[63]
1h	229-232	234 – 238[55]	**3d**	167-170	169-172[40]
2a	112-115	114-116[56]	**3e**	310-312[a]	311.8[a,64]

[a]Boiling point.

with water (3 x 5 mL), dried under vacuum and recrystallized from ethanol to afford the pure organic product. For liquid products (**1g**, **2e**, and **3e**), the reaction mixture was diluted with water and extracted with ethyl acetate (3 X 5 mL). The combined organic layers were washed with water, dried over sodium sulfate, filtered and evaporated under reduced pressure to get the crude products, which were purified by column chromatography using silica as the stationary phase and ethyl acetate/petroleum ether as the mobile phase. To recycle the catalyst, the aqueous quench and washes were saved and evaporated, then dried under vacuum. The solid thus obtained was treated as previously described[43] and used in subsequent runs without significant loss of yield.

N-Phenylformamide (Table 4, entry 1a)

White solid; m.p. 44-46 °C (lit. 42-44 °C); ^{1}H NMR (400 MHz, $CDCl_3$) the product was mixture of two rotamers (ratio 1:1): δ 8.68 (d, $J = 11.4$ Hz, 1H), 8.36 (s, 1H), 8.29 (s, 1H), 7.53 (d, $J = 7.8$ Hz, 2H), 7.47 (s, 1H), 7.37 – 7.30 (m, 4H), 7.20 – 7.07 (m, 4H).

N-Phenylacetamide (Table 5, entry 2a)

White solid; m.p. 112-115 °C (lit. 114-116 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, $J = 7.8$ Hz, 2H), 7.30 (t, $J = 7.3$ Hz, 2H), 7.20 (s, 1 H), 7.08 (t, $J = 7.2$ Hz, 1H), 2.16 (s, 3H).

2-Benzylisoindoline-1,3-dione (Table 6, entry 3c)

White solid; m.p. 115-117 °C (lit. 113-115 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 - 7.81 (m, 2H), 7.69 - 7.67 (m, 2H), 7.41 (d, $J = 7.1$ Hz, 2H), 7.29 - 7.25 (m, 3H), 4.82 (s, 2H).

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