

RAPID COMMUNICATION

## Sulfated polyborate: A mild, efficient catalyst for synthesis of *N*-*tert*-butyl/*N*-trityl protected amides via Ritter reaction

KRISHNA S INDALKAR, CHETAN K KHATRI and GANESH U CHATURBHUIJ\*

Department of Pharmaceutical Sciences and Technology, Institute of Chemical Technology,  
Mumbai, Maharashtra 400 019, India  
Email: gu.chaturbhuj@gmail.com

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**Abstract.** Rapid, efficient and inexpensive method for synthesis of *N*-*tert*-butyl/*N*-trityl protected amides via Ritter reaction of nitriles with tertiary alcohols in the presence of a sulfated polyborate catalyst under solvent-free conditions is described. The catalyst has the advantage of Lewis as well as Bronsted acidity and recyclability without significant loss in catalytic activity along with easy work-up, short reaction time and good to excellent yields.

**Keywords.** Ritter reaction; sulfated polyborate; *N*-*tert*-butyl amide; solvent-free condition.

### 1. Introduction

The Ritter reaction is the most important carbon-nitrogen (C–N) bond-forming reaction. It is an efficient and broadly used method for amide bond formation from a nitrile and a carbocation precursor like tertiary alcohol in a strongly ionizing medium, usually sulfuric acid which limits its wider applications.<sup>1,2</sup> The conversion of nitrile to corresponding *N*-*tert*-butyl amide is an important organic transformation which finds a wide range of applications in the synthesis of heterocycles,<sup>3</sup> natural products, and drugs such as Indinavir, an HIV protease inhibitor (Figure 1).<sup>4</sup>

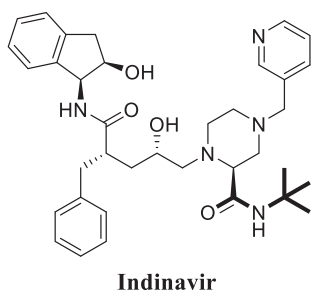
Till date, various alternative methodologies have been reported to achieve chemo-selectivity with higher yields under milder conditions to improve Ritter reaction. These include a variety of Lewis and Bronsted acid/reagents such as sulfuric acid,<sup>5,6</sup> Mg(HSO<sub>4</sub>)<sub>2</sub>,<sup>7</sup> silica-supported Fe(ClO<sub>4</sub>)<sub>3</sub>,<sup>8</sup> trifluoromethane sulfonic acid,<sup>9</sup> trifluoromethane sulfonic anhydride,<sup>10</sup> zeolites,<sup>11</sup> (BF<sub>3</sub>·OEt<sub>2</sub>),<sup>12</sup> MnO<sub>2</sub>·SiO<sub>2</sub>,<sup>13</sup> bismuth Triflate,<sup>14</sup> DNBSA,<sup>15</sup> Ca(HSO<sub>4</sub>)<sub>2</sub>,<sup>16</sup> TiCl<sub>4</sub>,<sup>17</sup> P<sub>2</sub>O<sub>5</sub>·SiO<sub>2</sub>,<sup>18</sup> Fe<sup>3+</sup>-K10 Montmorillonite,<sup>19</sup> PMA·SiO<sub>2</sub>,<sup>20</sup> Nafion-H,<sup>21</sup> NaHSO<sub>4</sub>/SiO<sub>2</sub>,<sup>22</sup> and H<sub>2</sub>PW<sub>12</sub>O<sub>40</sub>.<sup>23</sup> In addition to this, various catalytic systems are also reported which include silica sulfuric acid,<sup>24</sup> silica supported sulfonic acid,<sup>25</sup> silica-bonded *N*-propyl sulphamic acid (SBNPSA),<sup>26</sup> silica boron–sulfuric acid nanoparticles (SBSANs),<sup>27</sup> HClO<sub>4</sub>-functionalized silica-coated magnetic nanoparticles,<sup>28</sup> sulfated tungstate,<sup>29</sup>

alumina–methanesulfonic acid (AMA),<sup>30</sup> nanocat-Fe–OSO<sub>3</sub>H,<sup>31</sup> FeCl<sub>3</sub>·6H<sub>2</sub>O,<sup>32</sup> Amberlyst-15,<sup>33</sup> KAl(SO<sub>4</sub>)<sub>2</sub>·12H<sub>2</sub>O,<sup>34</sup> CoFe<sub>2</sub>O<sub>4</sub>-SiO<sub>2</sub>-DASA,<sup>35</sup> Al(HSO<sub>4</sub>)<sub>3</sub>,<sup>2</sup> ionic liquids,<sup>36</sup> iodine,<sup>37</sup> and polyvinylpyrrolidone-supported boron trifluoride.<sup>38</sup>

However, these reagents and catalytic systems suffer from one of the following limitations such as the use of expensive and toxic reagents/catalysts, limited availability of reagent, hygroscopicity as well as instability of the reagents, use of strong protic media, harsh reaction condition, long reaction time, low yield and tedious workup procedure. Additionally, the main disadvantage of almost all the reported methods is the use of water-sensitive, non-recoverable/reusable catalysts. Due to wide applicability of amides as building blocks in medicinal as well as organic chemistry and to address several drawbacks, there is a need for the development of a mild, safe, more efficient, cost-effective and recyclable catalyst.

A literature search revealed that boric acid catalyzes many important organic transformations at a temperature above 100°C.<sup>39–41</sup> Boric acid dehydrates above 100°C and converts to its polymeric form, which presumably is the active species catalyzing the reaction.<sup>42,43</sup> Dehydrative polymerization liberates water molecules which may hamper the progress of the reactions. This inspired us to develop a polymeric boric acid catalyst with mild Bronsted acidity. To accomplish this boric acid was dehydrated at 200°C to convert it into its polymeric Lewis acid form and then sulfonated by using chlorosulphonic acid to introduce the mild Bronsted acid character. Boron is an electron deficient

\*For correspondence



**Figure 1.** Pharmaceutically active molecule.

element and electron withdrawing effect of adjacent sulfate enhances its Lewis acidity; and hence it has both Lewis as well as Bronsted acid characters (Scheme 1).

The development of a novel methodology with an objective of mild, efficient and eco-friendly protocol, the use of recyclable catalysts for organic synthesis to maximize efficiency and minimize waste is currently in demand. To achieve these objectives, herein we report sulfated polyborate as a mild, efficient and eco-friendly catalyst for the synthesis of *N*-*tert*-butyl/*N*-trityl protected amides under a solvent-free condition with high yields and short reaction times. The catalyst was prepared from readily available boric acid, which is economical and non-toxic starting material. This is the first report on the use of sulfated polyborate for the synthesis of *N*-*tert*-butyl/*N*-trityl protected amides (Scheme 1). This catalyst is environmentally benign due to its mild acidity and non-toxic nature.

## 2. Experimental

### 2.1 Materials and methods

Melting points of all the compounds were recorded by Analab ThermoCal melting point apparatus in the open capillary tube and are uncorrected. The FTIR spectra (KBr disk) were recorded on Shimadzu FTIR Affinity-1 Fourier Transform Infrared spectrophotometer.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on MR400 Agilent Technology NMR spectrometer using tetramethylsilane (TMS) as an internal

standard and  $\text{DMSO-d}_6$  or  $\text{CDCl}_3$  as the solvent. Chemicals and solvents used were of LR grade and purchased from SD fine, Avra Synthesis, and Spectrochem and used without purification. The determination of purity of the starting materials and monitoring of reaction were accomplished by thin-layer chromatography (TLC) on Merck silica gel G F<sub>254</sub> plates.

### 2.2 Preparation of sulfated polyborate

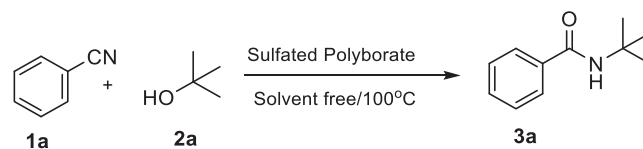
The sulfated polyborate catalyst was prepared from boric acid and characterized as reported in the literature.<sup>44</sup>

### 2.3 General procedure for the synthesis of *N*-*tert*-butyl/*N*-trityl protected amides

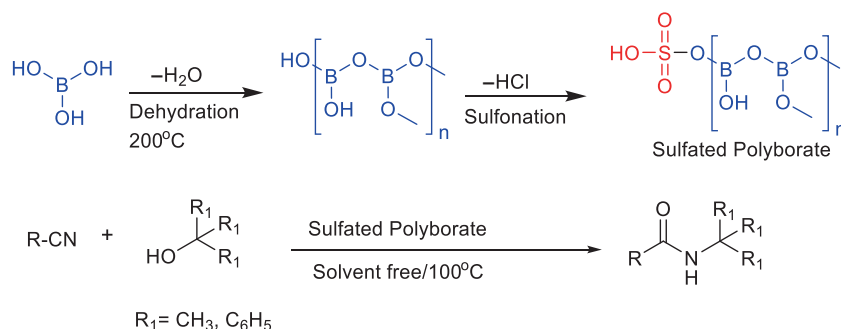
To a mixture of nitrile (2.0 mmol) and *tert*-butanol or trityl alcohol (2.0 mmol), sulfated polyborate (10 wt%) was added. The reaction mixture was stirred at 100°C in an oil bath and was monitored by thin layer chromatography (TLC). After completion of the reaction, the mixture was cooled to room temperature and quenched with water. The resultant amide was filtered/extracted with EtOAc to get the crude products. Crude products were either recrystallized from ethanol or purified by column chromatography using silica as a stationary phase and EtOAc: petroleum ether as the mobile phase. The products obtained were known compounds and were identified by melting point, FTIR,  $^1\text{H}$ , and  $^{13}\text{C}$  NMR spectroscopy. The spectral data were compared with the literature values.

## 3. Results and Discussion

The catalyst was prepared and characterized by various analytical techniques such as potentiometric analysis, Fourier transform infrared spectroscopy (FTIR), X-ray



**Scheme 2.** Synthesis of *N*-*tert*-butyl benzamide.



**Scheme 1.** Schematic representation of sulfated polyborate catalyzed Ritter reaction of nitrile and tertiary alcohol.

diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDAX).<sup>44</sup>

Recently, we have reported an efficient sulfated polyborate as a catalyst for one-pot multicomponent synthesis of 3,4-dihydropyrimidin-2(1*H*)-ones/thiones via Biginelli reaction,<sup>44</sup> synthesis of  $\alpha$ -aminophosphinates via Kabachnik-Fields reaction,<sup>45</sup> synthesis of quinoxalines,<sup>46</sup> Hantzsch 1,4-dihydropyridines,<sup>47</sup> and Kindler reaction<sup>48</sup> all under solvent-free conditions. Its ease of preparation, high stability, mild acidity, and reusability

prompted us to explore its potential to catalyze other useful reaction transformations.

Herein, we report sulfated polyborate as a catalyst for synthesis of *N*-*tert*-butyl/*N*-trityl protected amides from nitriles and tertiary alcohols via Ritter reaction with good to excellent yields (Scheme 1). Initially, we designed our study to investigate the suitability of sulfated polyborate as a catalyst at different reaction conditions for the synthesis of the *N*-*tert*-butyl amide. An equimolar mixture of benzonitrile as representative substrate and *tert*-butanol were used (Scheme 2). The effect of the catalyst loading on time and yields of the reaction was assessed (Table 1, entries 2–6). In the absence of a catalyst, the reaction does not proceed at 100°C (Table 1, entry 1), while increased catalyst loading resulted in an increased product yield (Table 1, entries 2–5). The catalyst loading beyond 10 wt% was not advantageous (Table 1, entry 6). Hence, a 10 wt% catalyst loading was considered optimum for further study.

Temperature played a significant role in the synthesis of *N*-*tert*-butyl benzamide (Table 1, entries 6–8). The temperature effect was observed at ambient, 70°C and 100°C in presence of sulfated polyborate. The reaction does not proceed at room temperature and 70°C (Table 1, entries 7, 8), while it proceeded at 100°C with increased product yield in a shorter reaction time (Table 1, entry 5). Therefore, 100°C was chosen as optimum temperature for the reaction.

The effect of different solvents on time and yield of the reaction was assessed (Table 2, entries 1–6). None of the solvents presented an advantage over solvent-free condition. Therefore, the solvent-free condition was regarded as the best for the cost and ecological benefits.

There are various acid catalysts reported for Ritter reaction (Table 3, entries 2–12). Herein, the reported acid catalysts were compared with the present study,

**Table 1.** Catalyst loading and temperature optimization study.

Entry	Catalyst (wt%)	Solvent	Temp. (°C)	Time (h)	Yield <sup>a</sup> (%)
1	0	Solvent-free	100	6	NR <sup>b</sup>
2	2.5	Solvent-free	100	1	72
3	5.0	Solvent-free	100	1	81
4	7.5	Solvent-free	100	1	92
5	10	Solvent-free	100	1	96
6	15	Solvent-free	100	1	97
7	10	Solvent-free	70	6	NR <sup>b</sup>
8	10	Solvent-free	rt	6	NR <sup>b</sup>

<sup>a</sup>Isolated yield; <sup>b</sup>No reaction.

**Table 2.** Solvent optimization studies.

Entry	Catalyst (wt%)	Solvent	Temp. (°C)	Time (h)	Yield <sup>a</sup> (%)
1	10	Water	Reflux	6	NR <sup>b</sup>
2	10	MeCN	Reflux	6	Traces
3	10	THF	Reflux	6	Traces
4	10	EDC	Reflux	6	30
5	10	Toluene	Reflux	6	70
6	10	DMSO	100	6	Traces

<sup>a</sup>Isolated yield; <sup>b</sup>No reaction.

**Table 3.** Comparison of sulfated polyborate with different acid catalysts reported for synthesis of *N*-*tert*-butyl benzamide via Ritter reaction<sup>a</sup>

Entry	Catalyst	Reaction condition	Time (h)	Yield <sup>b</sup> (%)	Ref.
1	Sulfated polyborate	Solvent-free/100°C	1	96	This work
2	Amberlyst-15	Solvent-free/70°C	2	90	33
3	KAl(SO <sub>4</sub> ) <sub>2</sub>	Solvent-free/100°C	2	96	34
4	Mg(HSO <sub>4</sub> ) <sub>2</sub>	Solvent-free/20°C	2	83	7
5	Sulfated tungstate	Solvent-free/100°C	3	95	29
6	Silica supported sulfonic acid	Solvent-free/80°C	3	86	25
7	Al(HSO <sub>4</sub> ) <sub>3</sub>	CH <sub>2</sub> NO <sub>2</sub> /100°C	3	90	2
8	Polyvinylpolypyrrolidone supported-BF <sub>3</sub>	EDC/reflux	4	45	38
9	Alumina methane sulfonic acid	Solvent-free/60°C	7	86	30
10	Bismuth triflate	Water/100°C	17	85	14
11	Ionic Liquid	Solvent-free/80°C	18	92	36
12	Fe <sup>3+</sup> montmorillonite K10	Solvent-free/70°C	48	40	19

<sup>a</sup>Reaction conditions: benzonitrile and *tert*-butanol. <sup>b</sup>Isolated yield.

**Table 4.** Sulfated polyborate catalyzed Ritter reaction under solvent-free condition.

Entry	Nitriles (R)	Alcohol (R <sub>1</sub> )	Time (h)	Yield <sup>b</sup> (%)	Melting point (°C)	
					Obtained	Literature
1	C <sub>6</sub> H <sub>5</sub>	CH <sub>3</sub>	1	96	132–135	134–136 <sup>49</sup>
2	2-Cl-C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	1	85	104–106	106–107 <sup>29</sup>
3	3-Cl-C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	1	89	100–103	102–104 <sup>49</sup>
4	4-Cl-C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	1	90	136–138	138–140 <sup>49</sup>
5	4-Br-C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	1	86	108–110	110–112 <sup>50</sup>
6	4-CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	1.5	90	113–115	112–114 <sup>49</sup>
7	4-O <sub>2</sub> N-C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	3	88	156–159	156–157 <sup>50</sup>
8	4-CH <sub>3</sub> CO-C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	3	80	134–138	136–138 <sup>51</sup>
9	4-CH <sub>3</sub> O-C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	3	87	117–119	116–118 <sup>49</sup>
10	3-HO-C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	3	86	130–372	NA <sup>b</sup>
11	4-HO-C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	3	87	190–192	188–190 <sup>49</sup>
12	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub>	CH <sub>3</sub>	1	89	110–112	113–115 <sup>29</sup>
13	n-C <sub>4</sub> H <sub>9</sub>	CH <sub>3</sub>	1	93	47–49	48.8–49.8 <sup>52</sup>
14	CH <sub>3</sub>	CH <sub>3</sub>	1.5	88	95–97	95–96 <sup>50</sup>
15	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>	1.5	88	159–160	158–160 <sup>53</sup>
16	2-Cl-C <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	1.5	81	158–161	NA <sup>b</sup>
17	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub>	C <sub>6</sub> H <sub>5</sub>	1.5	84	186–188	187–188 <sup>2</sup>

<sup>a</sup>Isolated yield, <sup>b</sup>Not Available in literature.

which revealed that sulfated polyborate showed advantages with respect to work up, reaction condition, time and yields (Table 3, entries 2–12).

To study the scope and generality, the optimized reaction conditions were applied to various nitriles. A variety of *N-tert*-butyl/trityl amides were synthesized from *tert*-butanol/trityl alcohol and a substituted aromatic and aliphatic nitriles in the presence of a sulfated polyborate catalyst in good yields in shorter reaction time (Table 4, entries 1–17). Among the aromatic nitriles bearing no substituents and electron donating substituents, aliphatic and benzylic nitriles showed better yields (Table 4, entries 1–6, 12–14). On the other hand, those with electron withdrawing substituents showed comparable yield at longer reaction times (Table 4, entries 7–11). This protocol was also applied to the reaction of nitriles with trityl alcohol which resulted in similar yields in comparable reaction times (Table 4, entries 15–17). The optimized reaction protocol was also applied for the reaction of nitriles with primary and secondary alcohols with no success.

The reusability of the catalyst in the model reaction under solvent-free condition at 100°C was evaluated. After completion of each cycle, the reaction was quenched in water and the product was filtered. The filtrate was evaporated under reduced pressure to recover the catalyst quantitatively. The recovered catalyst was reused four times with no significant loss in a catalytic

activity. The yields were in the range of 90–95% upto 5 cycles.

#### 4. Conclusions

In conclusion, the present procedure is a rapid, efficient and eco-friendly for the synthesis *N-tert*-butyl/trityl amides *via* Ritter reaction of various aromatic/aliphatic nitriles and *tert*-butanol/trityl alcohol under optimal conditions. Mild reaction conditions, shorter reaction times, higher yields, ease of workup and recyclability of the catalyst are the key features of the present protocol. Moreover, the present method has the ability to tolerate a wide variety of substituents along with enhanced product purity which promises economical and ecological rewards.

#### Supplementary Information (SI)

Full experimental details, FTIR, <sup>1</sup>H and <sup>13</sup>C NMR spectra of compounds are presented in Supplementary Information, which is available at [www.ias.ac.in/chemsci](http://www.ias.ac.in/chemsci).

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