

## Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O: An Efficient and Green Catalyst for Synthesis of 1,5-Benzodiazepines and β-Amino Carbonyl Compounds

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Bismuth nitrate pentahydrate is found to be an efficient catalyst for the synthesis of 1,5-benzodiazepines and β-amino carbonyl compounds using condensation of *o*-phenylenediamine with ketones and one pot three component Mannich reaction, respectively under solvent free conditions at room temperature. This method offers a simple, solvent free, room temperature, environment friendly synthesis of 1,5-benzodiazepines and β-amino carbonyl compounds. The products are isolated by column chromatography and recrystallization, respectively and were characterized by their melting point, IR and <sup>1</sup>H NMR spectroscopy.

**Key Words:** 1,5-Benzodiazepines, β-Amino carbonyl compounds, Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O, Solvent free conditions.

### INTRODUCTION

The development of solvent free organic synthesis has become important research area, aiming to make the synthesis simpler, to prevent hazardous solvent waste, to save energy and toxicity in chemical processes. Many organic solvents are ecologically harmful and their use should be minimized as far as possible or should be avoided altogether<sup>1,2</sup>.

Benzodiazepines are very important biologically active compounds as these are used as anticonvulsant, antianxiety, antiinflammatory, analgesic, hypnotic, antidepressive and sedative agents<sup>3</sup>. In spite of their biological importance, benzodiazepines have application in industries such as dyes for acrylic fibres in photography<sup>4</sup>. Additionally, 1,5-benzodiazepines are useful synthons for the preparation of other fused ring compounds such as triazolo-<sup>5-7</sup>, oxadiazolo-<sup>8-10</sup>, oxazino<sup>11</sup> and furano-benzodiazepines<sup>12</sup>. Mannich reaction is important in carbon-carbon bond forming reaction in organic synthesis<sup>13-15</sup>, as it provides β-amino carbonyl compounds which are important synthetic intermediates for various nitrogen containing natural products and pharmaceutical<sup>16,17</sup>. The increasing popularity of Mannich reaction has been fuelled by the ubiquitous nature of nitrogen-containing compounds in drugs and natural products<sup>18</sup>.

1,5-Benzodiazepines are prepared by condensation reaction of *o*-phenylenediamine with α,β-unsaturated carbonyl compounds<sup>19</sup>, β-haloketones<sup>20</sup>, β-diketones<sup>21</sup>, chalcones<sup>22</sup> or ketones using variety of catalysts such as BF<sub>3</sub>·OEt<sub>2</sub><sup>23</sup>, NaBH<sub>4</sub><sup>24</sup>, Al<sub>2</sub>O<sub>3</sub>-P<sub>2</sub>O<sub>5</sub> under microwave<sup>25</sup>, HOAc microwave<sup>26</sup>, InBr<sub>3</sub><sup>27</sup>,

FeCl<sub>3</sub><sup>28</sup>, zeolite<sup>29</sup>, Sc(OTf)<sub>3</sub><sup>30</sup>, ceric ammonium nitrate<sup>31</sup>, ionic liquid<sup>32</sup>, kaolin<sup>33</sup>, NbCl<sub>5</sub><sup>34</sup>, keggin heteropolyacids<sup>35</sup>. Whereas the synthesis of β-amino carbonyl compounds involves the three-component one pot reaction using aromatic aldehydes, aromatic ketones and aromatic amines. Several methods have been reported in the literature and few catalysts, including conc. HCl<sup>36</sup>, NbCl<sub>5</sub><sup>37</sup>, Al(CH<sub>3</sub>SO<sub>3</sub>)<sub>3</sub>·4H<sub>2</sub>O<sup>38</sup>, dodecylbenzenesulfonic acid<sup>39</sup>, SnCl<sub>2</sub><sup>40</sup>, ionic liquid<sup>41</sup>, InCl<sub>3</sub><sup>42</sup> and polymer-support sulphonic acid<sup>43</sup>, GuHCl<sup>44</sup>, Me<sub>2</sub>SBr<sub>2</sub><sup>45</sup> etc., have been developed. However, most of these methods suffer from disadvantages such as strong acids or expensive catalysts, longer reaction time, elevated reaction temperature, toxic reagents, low yield. Hence there is an increasing interest in developing environmentally benign reactions and atom-economic catalytic processes for the synthesis of 1,5-benzodiazepines and β-amino carbonyl compounds.

Recently, bismuth(III) salts are used as Lewis acid catalysts<sup>46-49</sup> in organic synthesis because of low cost, easy handling and friendly ecological behaviour. In continuation to our work on green synthesis<sup>50</sup> we report a rapid synthesis of 1,5-benzodiazepines and β-amino carbonyl compounds in presence of Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O because it is non-toxic, cheaper, easily available and stable in air.

### EXPERIMENTAL

Melting points were recorded on gallien kamp apparatus and are uncorrected. <sup>1</sup>H NMR (400 MHz) spectra were determined with a Bruker Advance 400 spectrometer (CDCl<sub>3</sub>) using tetramethylsilane (TMS) as internal standard. Infrared spectra

( $\text{cm}^{-1}$ ) were measured with Perkin Elmer spectrometer. All reactions were carried out using reagent-grade solvents and the reagents were purchased from local suppliers.

#### General procedure for synthesis of 1,5-benzodiazepines:

To a mixture of *o*-phenylenediamine (1 mmol) and ketone (2.2 mmol), catalyst  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  (0.05 mmol or 5 mol %) was added and the mixture was stirred at room temperature. The progress of the reaction was monitored by TLC. After the completion of reaction catalyst was removed by filtration and then reaction mixture was diluted with ethyl acetate and washed with water. The organic layer was dried over anhydrous sodium sulphate and concentrated under reduced pressure to give crude product. Then crude product was purified by silica gel column chromatography using hexane:ethyl acetate as an eluent to afford the desired compound in pure form.

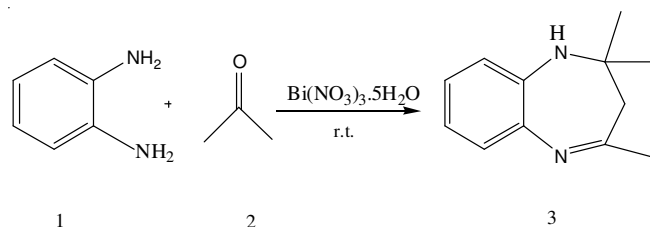
**General procedure for synthesis of  $\beta$ -amino carbonyl compounds:** To the mixture of acetophenone (1 mmol), aldehyde (1 mmol) and aniline (1 mmol) catalyst  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  (0.1 mmol or 10 mol %) was added and the mixture was stirred at room temperature until the completion of reaction as indicated by TLC. The catalyst was removed by filtration. Then the resulting mixture was washed with water. The crude product was purified by recrystallization from ethanol to give corresponding pure compound.

**2,2,4-Trimethyl-2,3-dihydro-1*H*-1,5-benzodiazepines (3a):** m.p. 135-137 °C, IR (KBr,  $\nu_{\text{max}}$ ,  $\text{cm}^{-1}$ ): 3293.3, 1632.3,  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ , ppm (*J*, Hz): 1.34 (6H, s); 2.25 (2H, s); 2.36 (3H, s); 2.95 (1H, NH, br, s); 6.71-7.14 (4H, m).

**1,3-Diphenyl-3-(phenylamino) propan-1-one (4a):** m.p. 142-143 °C, IR (KBr,  $\nu_{\text{max}}$ ,  $\text{cm}^{-1}$ ): 3384.8, 2917.8, 1671, 1291.5.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : ppm (*J*, Hz): 357-3.58 (d, 2H), 5.01- 5.042 (m, 1H), 6.64-6.66 (d, 2H), 6.74 (t, 1H), 7.10-7.60 (m, 10H), 7.91-7.92 (d, 2H).

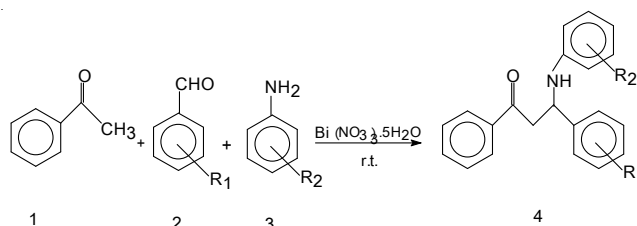
## RESULTS AND DISCUSSION

In this paper we report a facile, efficient and practical method for the synthesis of 1,5-benzodiazepines by condensation of *o*-phenylenediamine with ketones and  $\beta$ -amino carbonyl compounds by one pot three component reaction of aromatic aldehydes, aromatic ketones and aromatic amines using  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  as catalyst (Schemes I and II).



**Scheme-I:** Synthesis of 1,5-benzodiazepines using  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  as a catalyst at room temperature under solvent free conditions

Initially, the optimized reaction conditions were screened by conducting the reaction with different catalyst amount at room temperature for the preparation of 1,5-benzodiazepines (Table-1, Fig. 1). At first stage, reaction was investigated with *o*-phenylenediamine, acetone and  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  (5 mol %) under solvent free conditions at room temperature. The reaction occurred in 15 min and product was obtained in 85 % yield.



**Scheme-II:** Synthesis of  $\beta$ -amino carbonyl compounds using  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  as a catalyst at room temperature under solvent free conditions

**TABLE-1**  
EFFECT OF THE AMOUNT OF THE CATALYST ON THE YIELD OF 1,5-BENZODIAZEPINES AND  $\beta$ -AMINO CARBONYL COMPOUNDS

| Entry | Amount of catalyst (mmol or mol %) | Yield (%)            |                                   |
|-------|------------------------------------|----------------------|-----------------------------------|
|       |                                    | 1, 5-Benzodiazepines | $\beta$ -Amino carbonyl compounds |
| 1     | 2                                  | 65                   | 65                                |
| 2     | 5                                  | 92                   | 75                                |
| 3     | 7                                  | 85                   | 80                                |
| 4     | 10                                 | 83                   | 85                                |
| 5     | 12                                 | 81                   | 79                                |

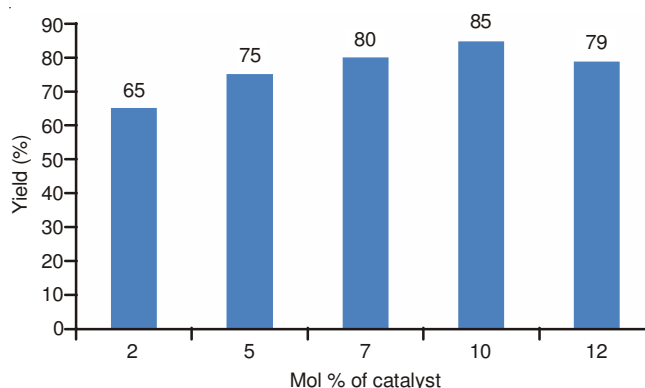


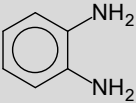
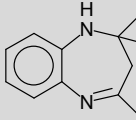
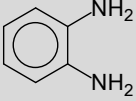
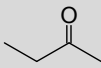
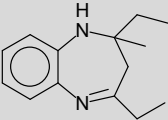
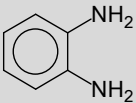
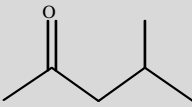
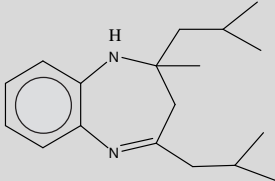
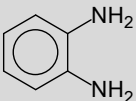
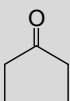
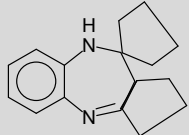
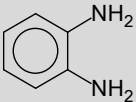
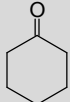
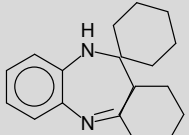
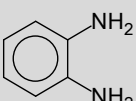
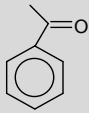
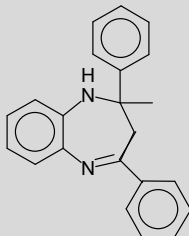
Fig. 1. Effect of amount of catalyst on the yield of 1,5-benzodiazepines

Further the optimized amount of catalyst was screened by taking *o*-phenylenediamine and acetone as model reactants. Using less amount of catalyst *i.e.*, 2 and 4 mol % resulted in the decrease of yield and by increasing the amount of catalyst up to 12 mol % did not improve the yield. However in the absence of catalyst the reaction did not proceed. It was found that reaction occurs in short time producing high yields of products. Further it was screened that 5 mol % of catalyst is sufficient for carrying out the synthesis of 1,5-benzodiazepines. An increase in the amount of catalyst did not improve the yield.

To show the feasibility of reaction method the reaction was explored by carrying out the reaction using various substrates (Table-2) with optimized amount of catalyst at room temperature under solvent free conditions. Both the acyclic and cyclic ketones gave excellent yields of products. It was observed that both primary and secondary ketones such as methyl ethyl ketones and methyl *iso*-butyl ketones were reacted and less hindered primary ketone is involved in cyclisation to give the product. The products are formed successfully under ambient condition within 10-30 min.

The efficiency of the present method and catalytic activity of catalyst was compared with the reported procedures and

TABLE-2  
Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O CATALYZED SYNTHESIS OF 1,5-BENZODIAZEPINES USING  
*o*-PHENYLENEDIAMINE AND VARIOUS KETONES AT ROOM TEMPERATURE

| S. No. | Diamine                                                                             | Ketone                                                                              | Product                                                                             | Time (min) | Yield (%) | m.p. (°C) observed | m.p. (°C) reported |
|--------|-------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|------------|-----------|--------------------|--------------------|
| a      |    |    |    | 15         | 92        | 134-136            | 137-139            |
| b      |    |    |    | 15         | 80        | 135-137            | 137-139            |
| c      |    |    |    | 25         | 79        | 117-119            | 118-120            |
| d      |    |    |    | 14         | 83        | 135-138            | 137-139            |
| e      |   |   |   | 5          | 87        | 134-136            | 138-139            |
| f      |  |  |  | 25         | 90        | 110-111            | 107-109            |

catalysts. Condensation of *o*-phenylenediamine with acetone afforded 96 % yield after 3 h at 50 °C in hexane in the presence of NbCl<sub>5</sub> (5 mol %)<sup>34</sup>. Kaolin<sup>33</sup> catalysed condensation of *o*-phenylenediamine with acetone gave 90 % yield in dichloroethane after 3 h. While in case of Sc(OTf)<sub>3</sub> (5 mol %)<sup>30</sup>, reaction proceeded in 3 h at room temperature afforded 96 % yield. The reaction in presence of InBr<sub>3</sub><sup>27</sup> provided the 96 % yield in 0.5 h. Compared to above methods and catalysts Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O catalyzed condensation of *o*-phenylenediamine with acetone gave 92 % yield in 15 min at room temperature under solvent free conditions.

After the successful application of Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O as catalyst in the synthesis of 1,5-benzodiazepines, we tried to explore its activity in synthesis of β-amino carbonyl compounds using various aromatic aldehydes, aromatic ketones and aromatic amines at room temperature under solvent free conditions.

In this reaction, optimization of amount of catalyst was also screened (Table-1, Fig. 2). The yield of the product was poor with low amount of catalyst and there was gradual

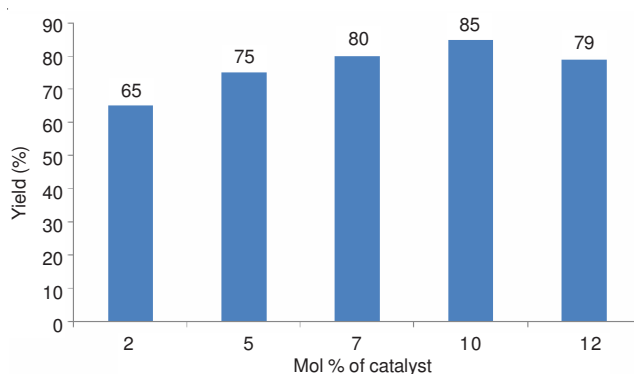


Fig. 2. Effect of amount of catalyst on the yield of β-amino carbonyl compounds

increase in the yield of product with increase in amount of catalyst. Maximum yield was obtained when the 10 mol % of catalyst was used. Further increase of catalyst did not improve the yield.

TABLE-3  
Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O CATALYZED SYNTHESIS OF β-AMINO CARBONYL COMPOUNDS USING AROMATIC KETONE, SUBSTITUTED AROMATIC ALDEHYDES AND SUBSTITUTED AROMATIC AMINES AT ROOM TEMPERATURE

| Entry | R <sub>1</sub>     | R <sub>2</sub>     | Product   | Time (min) | Yield (%) | m.p. (°C) (observed) | Ref. m.p. (°C) (reported) |
|-------|--------------------|--------------------|-----------|------------|-----------|----------------------|---------------------------|
| 1     | H                  | H                  | <b>4a</b> | 30         | 85        | 142-144              | 143-144                   |
| 2     | H                  | 4-NO <sub>2</sub>  | <b>4b</b> | 60         | 61        | 180-182              | 184-186                   |
| 3     | H                  | 4-Cl               | <b>4c</b> | 45         | 78        | 168-170              | 170-171                   |
| 4     | H                  | 4-OCH <sub>3</sub> | <b>4d</b> | 90         | 74        | 119-121              | 124-125                   |
| 5     | H                  | 4-F                | <b>4e</b> | 20         | 64        | 158-160              | 162-163                   |
| 6     | H                  | 2-CH <sub>3</sub>  | <b>4f</b> | 360        | Trace     | —                    | —                         |
| 7     | H                  | 3-CH <sub>3</sub>  | <b>4g</b> | 30         | 76        | 129-130              | 131-132                   |
| 8     | H                  | 2-OCH <sub>3</sub> | <b>4h</b> | 300        | Trace     | —                    | —                         |
| 9     | H                  | 2-Cl               | <b>4i</b> | 120        | 43        | 112-114              | —                         |
| 10    | H                  | 2-NO <sub>2</sub>  | <b>4j</b> | 180        | 48        | 101-103              | —                         |
| 11    | 4-OCH <sub>3</sub> | H                  | <b>4k</b> | 105        | 72        | 142-145              | 147-149                   |
| 12    | 4-OCH <sub>3</sub> | 4-Cl               | <b>4l</b> | 25         | 62        | 155-156              | 158-160                   |
| 13    | 4-OCH <sub>3</sub> | 4-OCH <sub>3</sub> | <b>4m</b> | 10         | 62        | 145-146              | —                         |
| 15    | 4-OCH <sub>3</sub> | 2-Cl               | <b>4o</b> | 360        | Trace     | —                    | —                         |
| 16    | 4-OCH <sub>3</sub> | 2-NO <sub>2</sub>  | <b>4p</b> | 420        | Trace     | —                    | —                         |
| 17    | 4-OCH <sub>3</sub> | 2-CH <sub>3</sub>  | <b>4q</b> | 450        | Trace     | —                    | —                         |
| 18    | 4-Cl               | H                  | <b>4r</b> | 60         | 64        | 110-111              | 114-115                   |
| 19    | 4-Cl               | 4-Cl               | <b>4s</b> | 90         | 69        | 115-116              | 118-119                   |

A series of β-amino carbonyl compounds were prepared from various aromatic aldehydes, aromatic ketones and aromatic amines (Table-3). The scope of the reaction was examined by reacting various substituted aldehydes and substituted amines. All the reactions proceeded well in the presence of Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O (10 mol %) at room temperature to give corresponding products in moderate to good yields. It was observed that the substituted aldehydes with electron-withdrawing (-Cl) and electron-donating (-OCH<sub>3</sub>) groups gave good results. Further it was seen that the substituted amines with both electron-withdrawing and electron-donating groups at the *meta* and *para* positions gave the product in good yields, but the *ortho* substituted amines gave very low or trace amount of yield.

To check the efficiency of the present method it was compared with the reported methods. The Mannich reaction of benzaldehyde, acetophenone and aniline afforded 83 % yield after 12 h at 25 °C in the presence of ionic liquid (2.5 g)<sup>41</sup> and 86 % yield after 8 h in ethanol using Al(CH<sub>3</sub>SO<sub>3</sub>)<sub>3</sub>·4H<sub>2</sub>O (5 mol %)<sup>38</sup> SnCl<sub>2</sub><sup>40</sup> catalyzed reaction (93 % yield in ethanol in 10 h), GuHCl, (10 mol %)<sup>44</sup> as catalyst (80 % yield in 4 h) and Me<sub>2</sub>SBr<sub>2</sub><sup>45</sup> (80 % yield in 3 h). But by using Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O, β-amino carbonyl compounds was obtained in 87 % yield after 0.5 h at room temperature in solvent free conditions.

## Conclusion

We have demonstrated the simple, an efficient and green synthesis of biologically active 1,5-benzodiazepines and β-amino carbonyl compounds using an efficient catalyst Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O at room temperature in solvent free conditions. The present protocol has attractive features such as: (a) simple procedure; (b) cheap, non-toxic, oxygen and moisture tolerant catalyst; (c) solvent free synthesis; (d) shorter reaction time; (e) high yielding of products; (f) environment friendly method. The 1,5-benzodiazepines and β-amino carbonyl compounds are smoothly obtained in mild conditions.

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