

# Using a Polymer-Supported Azide Ion in [2+3] Cycloaddition Reaction of Azide Ion with Nitriles

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**ABSTRACT:** The [2+3] cycloaddition between various nitriles and crosslinked poly(4-vinylpyridine) supported azide ion proceeds smoothly in the presence of ammonium bromide or ammonium chloride in *N,N*-dimethyl formamide, to give the corresponding 5-substituted-1H-tetrazoles in good to high yields. Conventional heating was used to promote reaction. It was found that using nitriles with electron-withdrawing groups result in higher

yields and lower reaction times. The present procedure offers advantages, such as shorter reaction time, and simple workup. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 123: 1977–1982, 2012

**Key words:** nitrile; polymeric reagent; polymer-supported reagent; alkyl azide; 5-substituted 1-H tetrazole; [2+3] cycloaddition

## INTRODUCTION

Tetrazoles are a class of heterocycles with wide range of applications. The growth of tetrazole chemistry over the last 25 years has been significant, mainly as a result of the roles played by tetrazoles in coordination chemistry as ligands, in medicinal chemistry as stable surrogates for carboxylic acids and in material chemistry as including explosives, rocket propellants, and agriculture.<sup>1–11</sup> Tetrazoles can be used as isosteric replacements for carboxylic acids in drug design.<sup>2</sup> An advantage of tetrazolic acids over carboxylic acids is that, they are resistant to many biological metabolic degradation pathways.<sup>3–5</sup> Another important application of tetrazoles is in the preparation of imidoylazides.<sup>12,13</sup> Also tetrazoles have found use in pharmaceuticals,<sup>14</sup> specialty explosives,<sup>15</sup> photography, and information recording systems,<sup>16</sup> not to mention as precursor to a variety of nitrogen containing heterocycles.<sup>17,18</sup>

It has long been known that simple heating of azide salt with nitrile in a solution produces the corresponding tetrazole.<sup>19–24</sup> Recently, Sharpless and coworkers<sup>25–28</sup> reported an innovative and safe procedure for the preparation of 5-substituted 1H-tetrazole from the corresponding nitriles and  $\text{NaN}_3$  in the presence of a stoichiometric amount of 50 mol % of Zn (II) salts. Later, Pizzo and coworkers reported an efficient method for the synthesis of tetrazoles by

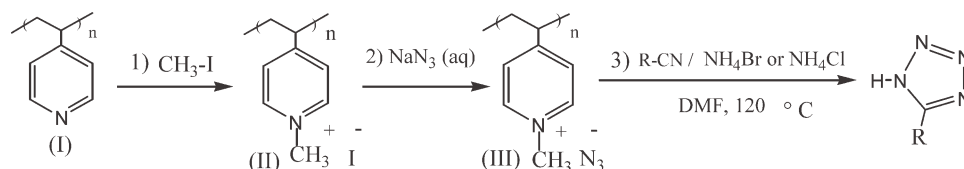
the reaction of nitriles with trimethylsilyl azide ( $\text{TMSN}_3$ ) using 50 mol % of tetrabutylammonium fluoride as catalyst.<sup>29</sup> Also several methods for synthesis of 5-substituted 1H-tetrazoles have been reported through the [2+3] cycloaddition of nitriles using  $\text{NaN}_3$  and  $\text{TMSN}_3$  in the presence of catalysts such as nanocrystalline  $\text{ZnO}$ <sup>30</sup> or zinc hydroxyapatite ( $\text{ZnHAP}$ ),<sup>31</sup> Zn/Al hydrotalcite,<sup>32</sup>  $\text{AlCl}_3$ ,<sup>33</sup>  $\text{BF}_3 \cdot \text{OEt}_2$ ,<sup>34</sup> Pd ( $\text{PPh}_3$ )<sub>4</sub>,<sup>35</sup>  $\text{Cu}_2\text{O}$ ,<sup>36</sup> and  $\text{FeCl}_3 \cdot \text{SiO}_2$ .<sup>37</sup>

Although many synthetic protocols for 5-substituted 1H-tetrazoles have been reported since the last middle century, there is still a need for the generation of more efficient processes for the synthesis of 5-substituted 1H-tetrazoles.

In recent years, there has been increasing emphasis on the use and design of environmentally friendly polymer supported reagents. Polymer-supported reagents and catalysts have been widely applied in organic transformations. Immobilization of reactive species on a polymer support could provide many important advantages over analogous homogeneous systems; for example, the separation of the support from the reaction mixture can be achieved by simple filtration-aiding isolation and purification procedures and excess of a polymeric reagent can be readily used without incurring complication in workup.

Although there are numerous applications of solid supported reagents and scavengers in literature,<sup>38–59</sup> but there are only two reports in the literature based on polymer-supported azide ion, and there is no report on the synthesis of tetrazoles based on polymeric reagents. Hassner and coworkers have reported<sup>60</sup> the use of the polymeric quaternary ammonium azide for synthesis of alkyl azides from

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**Scheme 1** Synthesis of 5-substituted 1H-tetrazoles from aromatic nitriles by using  $[P_4\text{-VP}]N_3$ .

alkyl halides or esters. Previously, we also reported the synthesis and application of crosslinked poly(4-vinylpyridine)-supported azide ion ( $[P_4\text{-VP}]N_3$ ) for synthesis of alkyl azides from alkyl halides.<sup>54</sup> In connection with our previous study on crosslinked  $[P_4\text{-VP}]N_3$ ,<sup>54</sup> now, we report an efficient and easy method for preparation of 5-substituted 1-H tetrazoles by using a polymer-supported azide ion,  $[P_4\text{-VP}]N_3$ , with nitriles in dimethylformamide (DMF) and in the presence of ammonium bromide, or ammonium chloride under heterogeneous conditions.

## EXPERIMENTAL

### General

Chemicals were either prepared in our laboratory or were purchased from Fluka (Buchs, Switzerland), Aldrich (Milwaukee, WI), and Merck chemical companies. Poly(4-vinylpyridine) crosslinked with 2% divinyl benzene (DVB), was purchased from Fluka (Buchs, Switzerland). Crosslinked poly(*N*-methyl-4-vinylpyridinium) iodide,  $[P_4\text{-VP}]I$ , and crosslinked poly(4-vinylpyridine)-supported sodium nitrite,  $[P_4\text{-VP}]NO_2$ , were synthesized according to our reported procedures<sup>47–49,54</sup> (Scheme 1). Progress of the reaction was followed by thin layer chromatography (TLC) using silica gel Poly Gram SIL G/UV 254 plates (Fluka). All products were characterized by comparison of their melting point, FTIR, and <sup>1</sup>H-NMR spectral data, with those of known samples and all yields refer to the isolated pure products. Melting points were determined with a Buchi melting point B-540 B.V. CHI apparatus. FTIR spectra were obtained by using a Bruker, Equinox (model 55), and NMR spectra were recorded on a Bruker AC 400, Aveance DPX spectrophotometer at 400 MHz in acetone-*d*<sub>6</sub>.

### Preparation of $[P_4\text{-VP}]N_3$

Poly(4-vinylpyridine) crosslinked with 2% DVB (white powder, 100–200 mesh; 1.0 g) was added to a solution of methyl iodide (20 mmol, 3.24 g) in acetonitrile (10 mL) and slowly stirred for 24 h at room temperature. The quaternized yellow polymer,  $[P_4\text{-VP}]I$ , (II), was filtered and washed with acetonitrile

(3 × 10 mL). It was then, vacuum dried overnight at 40°C, over P<sub>2</sub>O<sub>5</sub> (Scheme 1, Step 1).

The obtained  $[P_4\text{-VP}]I$  was added to 40 mL of a 3M aqueous solution of sodium azide and slowly stirred for 24 h. The prepared resin,  $[P_4\text{-VP}]N_3$ , (III), was filtered off and washed rapidly with distilled water (3 × 8 mL). It was then washed with diethyl ether and then vacuum dried overnight at 40°C, over P<sub>2</sub>O<sub>5</sub> (Scheme 1, Step 2). The polymer contained 3.4 mmol/g of azide group as determined by potentiometric titration with a 0.1N aqueous solution of silver nitrate.


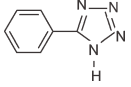
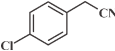
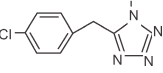

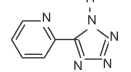

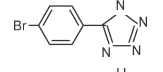
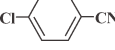
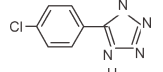

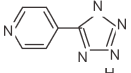
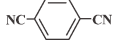
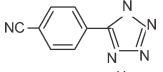
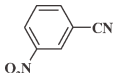
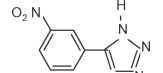

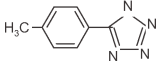
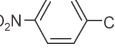
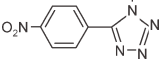
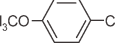
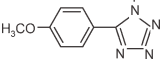
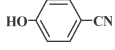
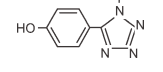
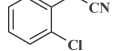
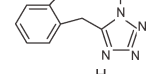
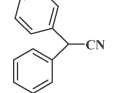
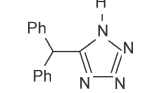
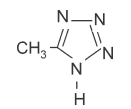
### General procedure for synthesis of 5-substituted 1-H tetrazoles from nitriles by using $[P_4\text{-VP}]N_3$ , in the presence of $NH_4Br$ or $NH_4Cl$ in DMF at 120°C

To a mixture of a nitrile compound (1 mmol),  $NH_4Br$  (294 mg, 3 mmol) or  $NH_4Cl$  (160 mg, 3 mmol) in DMF (5 mL), 0.9 g (3 mmol)  $[P_4\text{-VP}]N_3$  was added, and the mixture was slowly stirred for the appropriate time as indicated in Table I (0.5–3 h), at 120°C. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was washed with ethyl acetate (35 mL) and then with a 4N aqueous solution of HCl (20 mL). The organic layer was dried with anhydrous  $Na_2SO_4$ , and the solvent was evaporated to obtain the pure solid, 5-substituted 1-H tetrazole products with good to high yields (65–95%). All the products are known compounds,<sup>25,29,30,32,36,37</sup> and were characterized by FTIR, <sup>1</sup>H-NMR, and <sup>13</sup>C-NMR spectral data and physical properties were compared with known compounds.

## RESULTS AND DISCUSSION

During our investigation of multiple phase techniques in organic synthesis, we observed that crosslinked  $[P_4\text{-VP}]N_3$  can be easily prepared and used as a mild and efficient polymeric reagent for conversion of aromatic nitriles to the corresponding 5-substituted 1-H tetrazoles (Scheme 1). Crosslinked poly(4-vinylpyridine)-supported azide ion,  $[P_4\text{-VP}]N_3$ , was prepared according to our previous reported procedure.<sup>54</sup> It is important to note that this polymeric

TABLE I  
Synthesis of 5-Substituted 1-H Tetrazoles

Entry	Substrate	Product	Time	Yield (%) <sup>a</sup>	M.p. (°C)	Liter. M.p. (°C)/Ref.
1			3h	75	214–216	215–216/(25)
2			3h	65	214–216	–/(32)
3			1h	84	212–213	211/(25)
4			2h	93	268–270	–
5			2h	94	258–260	–/(32)
6			30 min	92	254–256	–
7			30 min	95	210–211	–/(32)
8			2h	80	154–155	–
9			2h	70	248–250	248–249/(36)
10			30 min	95	218–219	220/(25)
11			3h	trace	–	231–232/(25)
12			3h	trace	–	234–236/(25)
13			3h	No reaction	–	–
14			3h	No reaction	–	–
15	CH <sub>3</sub> –CN		3h	No reaction	–	–

<sup>a</sup> isolated Yields.

reagent is stable and can be stored for long times, without significant loss of their activity and can be readily used for synthesis of different 5-substituted 1-H tetrazoles from nitriles.

A good range of available aromatic nitriles were also subjected to [2+3] cycloaddition reaction with [P<sub>4</sub>-VP]N<sub>3</sub>, in the presence of ammonium bromide or ammonium chloride in DMF, under hetero-

TABLE II  
Optimization of the Reaction Conditions by using *p*-Nitrobenzonitrile (1 mmol) and [P<sub>4</sub>-VP]N<sub>3</sub>, After 3 H

Entries	Solvent	Temperature	[PVP]N <sub>3</sub> (mmol)	NH <sub>4</sub> Br (mmol)	NH <sub>4</sub> Cl (mmol)	Isolated yield (%)
1	Water	r.t	3	3	—	0.0
2	Water	r.t	3	—	3	0.0
3	Water	100	3	—	3	10
4	DMSO	120	3	3	—	52
5	DMSO	120	3	—	3	50
6	DMF	120	2	2	—	45
7	DMF	120	2	3	—	76
8	DMF	120	3	2	—	75
9	DMF	120	3	4	—	97
10	DMF	120	4	3	—	98
11	DMF	120	3	3	—	98
12	DMF	120	3	—	3	97
13	DMF	120	2	—	3	75
14	DMF	120	3	—	2	74
15	DMF	100	3	3	—	60
16	DMF	80	3	3	—	35

geneous conditions (Scheme 1). When the ammonium chloride (instead of ammonium bromide), is used, the same result is observed.

To increase the yield of tetrazole compounds, optimization of the reaction conditions was accomplished. 4-Nitrobenzonitrile (1 mmol; Table I, Entry 10 and Table II) was chosen as a model substrate and was treated with different molar ratio of [P<sub>4</sub>-VP]N<sub>3</sub>/nitrile/NH<sub>4</sub>Br or NH<sub>4</sub>Cl, in different solvents, and at different temperature, and the results are given in Table II. It was observed that, the 3/1/3 molar ratio of [P<sub>4</sub>-VP]N<sub>3</sub>/nitrile/NH<sub>4</sub>Br or NH<sub>4</sub>Cl in DMF at 120°C were the best molar ratio to achieve the highest yield of the product. With increasing the temperature and also with increasing the amounts of [P<sub>4</sub>-VP]N<sub>3</sub>/NH<sub>4</sub>Br or [P<sub>4</sub>-VP]N<sub>3</sub>/NH<sub>4</sub>Cl, result in about higher yields and lower reaction times (Table II).

This method represents an extremely convenient procedure for obtaining a wide variety of 5-substituted 1-H tetrazoles in high yields and sufficiently pure (Table I). The inspection of Table I reveals that both steric hindrance and electronic effects are pronounced effect in these reactions (Table I, Entry 2 vs. Entry 13 and Entries 4–7 vs. Entries 9, 11, and 12) and in which 4-substituted benzonitrile reacted faster than 2-substituted isomer (Table I, Entry 2 vs. Entry 13). Also electron-withdrawing group such as CN, Cl, and Br groups (Table I, Entries 4, 5, and 7), on 4-position of benzonitrile, accelerated their reactions with [P<sub>4</sub>-VP]N<sub>3</sub>, and vice versa; the electron-donating group such as CH<sub>3</sub>, OCH<sub>3</sub>, and OH groups decrease the reaction rate (Table I, Entries 9, 11, and 12). Also as shown in Table I, aromatic nitriles such as, 4-cyanobenzonitrile were reacted faster than aliphatic nitriles such as 4-cyanophenylacetone (Table I, Entry 7 vs. Entry 2).

We have also successfully applied this new method on a large scale. For example, up to 15 mmol of *p*-nitrobenzonitrile (Table I, Entry 10) could be converted into 5-(4-nitrophenyl) 1-H tetrazole without any loss of efficiency. In this method, the reaction time will be developed which is almost, shorter than previously reported methods.<sup>24,25,29,30,32,34,36,37</sup> This can probably be attributed to the local concentration of azide ion species inside the pores.

Although the spent polymeric reagent that used for synthesis of alkyl azides in our previous work<sup>54</sup> was easily regenerated by treatment with an aqueous solution of sodium azide and reused for several times but, in this method, our effort for regeneration of the polymer was not successful. Probably because, in the synthesis of alkyl azides, the reactions took place at room temperature while, in this method, the reactions take place at 120°C that, degradation of polymer may be occurred.


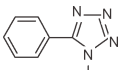

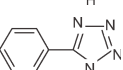

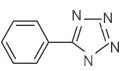

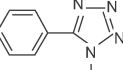

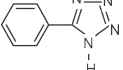

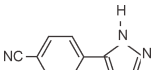

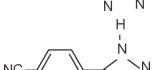

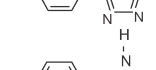

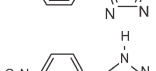
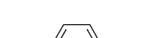
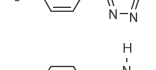

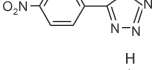

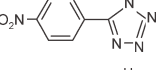

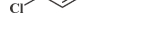
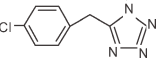
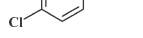
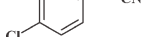
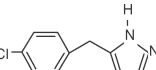
In Table III, other reported methods for preparation of 5-substituted 1-H tetrazoles are compared with this method. As it is demonstrated, the reaction time will be developed, which is shorter than previously reported methods.

The advantages of this method over conventional classical methods are separation of the supports from the reaction mixture by simple filtration, low reaction time, and excess of a polymeric reagent can be readily used without incurring complication in work-up. In addition, there is current research and general interest in heterogeneous systems because of the importance, such systems have in industry and in developing technologies.<sup>61</sup>

## CONCLUSIONS

Crosslinked poly(4-vinylpyridine)-supported sodium azide has been introduced as an efficient polymeric

**TABLE III**  
**Comparison of Different Methods for Synthesis of Various 5-Substituted 1-H Tetrazoles**

Entry	Substrate	Product	Time (h)	Isolated yield (%)	Ref.
1			12	79	37 <sup>a</sup>
2			18	86	29 <sup>b</sup>
3			14	72	30 <sup>c</sup>
4			12	84	32 <sup>d</sup>
5			3	75	Table I, Entry 1
6			20	80	37 <sup>a</sup>
7			24	78	32 <sup>d</sup>
8			0.5	95	Table I, Entry 7
9			12	81	37 <sup>a</sup>
10			3	95	29 <sup>b</sup>
11			12	96	36 <sup>e</sup>
12			0.5	95	Table I, Entry 10
13			24	69	37 <sup>a</sup>
14			24	71	30 <sup>c</sup>
15			24	65	32 <sup>d</sup>
16			3	65	Table I, entry 2

<sup>a</sup> In this procedure, the reaction was carried out in the presence of NaN<sub>3</sub>, FeCl<sub>3</sub>-SiO<sub>2</sub> in DMF at 120°C.

<sup>b</sup> In this procedure, the reaction was carried out in the presence of TMSN<sub>3</sub>, NH<sub>4</sub>F in water at 85°C.

<sup>c</sup> In this procedure, the reaction was carried out in the presence of NaN<sub>3</sub>, nano ZnO in DMF at 120–130°C.

<sup>d</sup> In this procedure, the reaction was carried out in the presence of NaN<sub>3</sub>, Zn/Al hydrotalcite in DMF at 120–130°C.

<sup>e</sup> In this procedure, the reaction was carried out in the presence of TMSN<sub>3</sub>, 2.5 mol % Cu<sub>2</sub>O in DMF/MeOH at 80°C.

reagent for synthesis of 5-substituted 1-H tetrazoles from various nitriles in high to excellent yields. It was found that using nitriles with electron-withdrawing groups result in bout higher yields and lower reaction times. The present method has the advantages of operational simplicity, mild reaction conditions, ready availability, fast reaction rates, and simple reaction workup.

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